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Bis(triphenylphosphine)iminium tetrafluoroborate chloroform monosolvate

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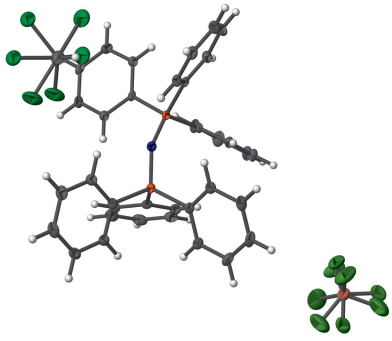
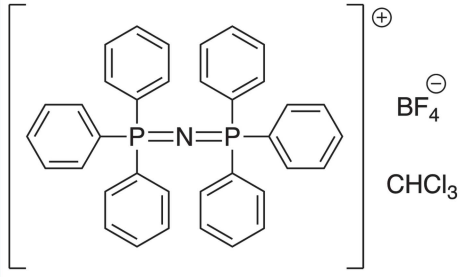
In the title compound, $C_{36}H_{30}NP_2^+\cdot BF_4^-\cdot CHCl_3$ or $[PPN]BF_4\cdot CHCl_3$, where $[PPN] = [(Ph_3P)_2N]^+$, two triphenylphosphine units are attached to a central N atom. The P—N—P bond angle is 137.69 (11)°. The two P—N bonds are nearly equivalent, with lengths of 1.5834 (18) and 1.5798 (17) Å. Both the BF_4^- anion and the chloroform solvent molecule are disordered over two positions, with occupancy ratios of 0.872 (3):0.128 (3) and 0.9628 (9):0.0372 (9), respectively. In the crystal, C—H···F and C—H···Cl hydrogen bonds link the $[PPN]^+$ cations, the BF_4^- anions, and the chloroform solvent molecules into an array which extends along the *b*-axis direction.

3D view



Chemical scheme



Structure description

The bis(triphenylphosphine)iminium cation, $[PPN]^+$ is a large cation commonly used by synthetic chemists to isolate reactive anions. $[PPN]^+$ salts such as the commercially available [PPN]Cl are a common source of the cation. The utility of $[PPN]^+$ is demonstrated in part by the over 4600 substances containing the cation listed in SciFinder® (SciFinder, 2018). Indeed $[PPN]^+$ has been used in many diverse applications. For example, some $[PPN]^+$ salts have been shown to have *in vitro* anticancer activity (Folda *et al.*, 2015) while $[PPN]NO_2$ is an often used nitrosylating reagent in chemical synthesis (Stevens *et al.*, 1981). $[PPN]^+$-(bipyridyl)tetracyanidoruthenate has been used as a humidity sensor (Evju & Mann, 1999), and the $[PPN]^+$ has been used to construct a nitrate-selective electrode (Werner *et al.*, 1989). A variety of $[PPN]^+$ salts are co-catalysts for the copolymerization of cyclohexene oxide and CO_2 (Darensbourg & Mackiewicz, 2005).

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

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8···F2^{i}	0.95	2.57	3.394 (3)	145
C8—H8···F2′^{i}	0.95	2.32	2.903 (8)	119
C14—H14···F2^{i}	0.95	2.44	3.333 (3)	157
C18—H18···Cl3′	0.95	2.78	3.481 (18)	131
C20—H20···F3′^{ii}	0.95	2.61	3.308 (15)	131
C26—H26···F4′^{iii}	0.95	2.52	3.427 (14)	160
C1*S*—H1*S*···F2^{ii}	1.00	2.15	3.132 (3)	166

Symmetry codes: (i) $-x+2, -y+2, -z+1$; (ii) $-x+2, -y+1, -z+1$; (iii) $-x+1, -y+1, -z+1$.

To the best of our knowlege, only four reports of $[PPN]BF_4$ structures are known: solvent-free $[PPN]BF_4$ (Bertocco *et al.*, 2016; Denny & Darensbourg, 2016; Folda *et al.*, 2015) and the solvate $[PPN]BF_4{\cdot}CH_2Cl_2$ (Liau *et al.*, 2002).

The structure of the title compound is shown in Fig. 1. There is one formula unit per asymmetric unit that consists of one $[PPN]^+$ cation, a tetrafluoroborate (BF_4^-) anion, and one chloroform solvate molecule. The anion and solvate molecule are each disordered over two sites. The central P—N—P bond angle of 137.69 (11)° is similar to the angles observed in the solvent free $[PPN]BF_4$, 139.42 (10)°, and its dichloromethane solvate, 138.54 (14)°. The extensive C—H···F and C—H···Cl hydrogen-bonding interactions, summarized in Table 1, help to stack an array of $[PPN]^+$ cations, BF_4^- anions and $CHCl_3$ molecules along the *b*-axis direction as shown in Fig. 2.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{36}H_{30}NP_2^+{\cdot}BF_4^-{\cdot}CHCl_3$
M_r	744.73
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	100
a, b, c (Å)	9.6306 (7), 10.8130 (8), 17.0381 (14)
α, β, γ (°)	91.826 (5), 95.619 (4), 90.734 (4)
V (Å^3)	1764.6 (2)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.40
Crystal size (mm)	0.57 × 0.14 × 0.11
Data collection	
Diffractometer	Bruker SMART APEX CCD area detector
Absorption correction	Multi-scan (*SADABS*; Bruker, 2016)
T_{min}, T_{max}	0.769, 0.838
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	32342, 11718, 7247
R_{int}	0.056
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.736
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.053, 0.130, 1.03
No. of reflections	11718
No. of parameters	474
No. of restraints	109
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.47, −0.90

Computer programs: *APEX2* and *SAINT* (Bruker, 2013), *SHELXT2013* (Sheldrich, 2015*a*), *SHELXL2016* (Sheldrich, 2015*b*), *Mercury* (Macrae *et al.*, 2006) and *SHELXTL* (Sheldrich, 2008).

Synthesis and crystallization

The title compound was obtained during our attempt to crystallize the mono-substituted VDPP derivative of $Fe(NO)_2(CO)_2$ (VDPP = 1,1-bis(diphenylphosphino)ethylene), which had been prepared in THF by reaction of $[PPN][Fe(CO)_3(NO)]$ and $[NO]BF_4$. The resulting solution of $Fe(NO)_2(CO)_2$ was ostensibly isolated from the solid $[PPN]BF_4$ byproduct by filter cannulation. Subsequent reaction of $Fe(NO)_2(CO)_2$ with VDPP produced $Fe(NO)_2$-(CO)(vdpp) which was isolated as a solid *in vacuo*. A chloroform solution of the filtered $Fe(NO)_2(CO)$(vdpp) was

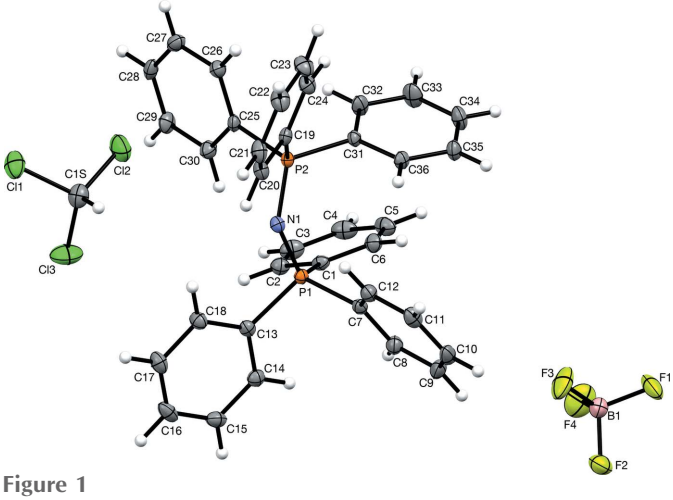


Figure 1
A view of the asymmetric unit of $[PPN]BF_4{\cdot}CHCl_3$ with displacement ellipsoids drawn at the 50% probability level. For clarity only the major disorder components of the BF_4^- anion and $CHCl_3$ solvent molecule are shown.

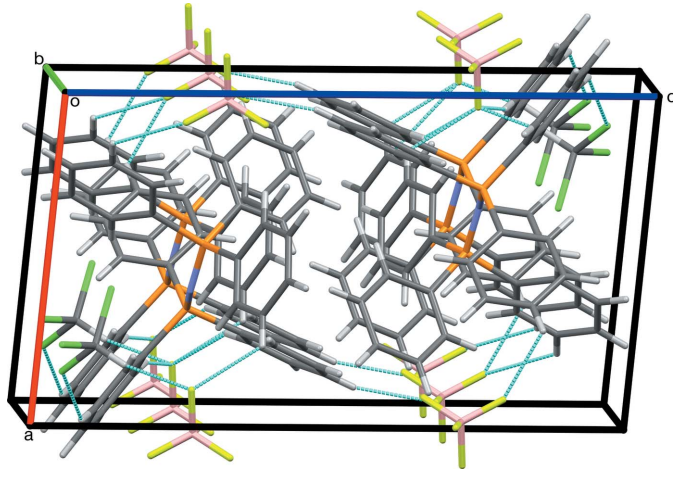


Figure 2
Overall packing viewed along the *b*-axis showing representative C—H···F and C—H···Cl hydrogen bonding contacts as dotted lines. Only the major disorder components are shown.

layered with pentane and allowed to evaporate slowly at room temperature under argon. After one week crystals of [PPN]BF_4 suitable for single-crystal structure determination were serendipitously obtained.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. Both the BF_4^- anion and the chloroform molecules were found to be disordered over two positions. Their occupancies were separately refined to sum to unity and the occupancy ratios converged to BF_4^- [0.872 (3):0.128 (3)] and $CHCl_3$ [0.9628 (9):0.0372 (9)], respectively. The disorder models were refined with geometrical constraints (SADI). The solvent Cl atoms were refined with displacement parameter constraints (EADP).

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full crystallographic data

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Bis(triphenylphosphine)iminium tetrafluoroborate chloroform monosolvate

Rachana Manandhar, Nigam P. Rath and Myron W. Jones

Bis(triphenyl-λ^5-phosphanylidene)azanium tetrafluoridoborate chloroform monosolvate

Crystal data

$C_{36}H_{30}NP_2^+\cdot BF_4^-\cdot CHCl_3$
M_r = 744.73
Triclinic, $P\overline{1}$
a = 9.6306 (7) Å
b = 10.8130 (8) Å
c = 17.0381 (14) Å
α = 91.826 (5)°
β = 95.619 (4)°
γ = 90.734 (4)°
V = 1764.6 (2) Å^3
Z = 2
$F(000)$ = 764
D_x = 1.402 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 6075 reflections
θ = 2.3–29.4°
μ = 0.40 mm^{-1}
T = 100 K
Rod, yellow
0.57 × 0.14 × 0.11 mm

Data collection

Bruker SMART APEX CCD area detector diffractometer
Radiation source: sealed tube
Detector resolution: 8 pixels mm^{-1}
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2016)
T_{min} = 0.769, T_{max} = 0.838
32342 measured reflections
11718 independent reflections
7247 reflections with $I > 2\sigma(I)$
R_{int} = 0.056
θ_{max} = 31.6°, θ_{min} = 1.9°
h = −11→14
k = −15→15
l = −24→24

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.130
S = 1.02
11718 reflections
474 parameters
109 restraints
Primary atom site location: dual
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0463P)^2 + 0.7191P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.47 e Å^{-3}
$\Delta\rho_{min}$ = −0.89 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.

Refinement. All H atoms were positioned geometrically and refined using a riding model with C—H = 0.95–0.99 Å and with U_{iso}(H) = 1.2 (1.5 for methyl groups) times U_{eq}(C).

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
P1	0.69176 (5)	0.63460 (5)	0.26638 (3)	0.01319 (11)	
P2	0.48489 (5)	0.43360 (5)	0.27647 (3)	0.01301 (11)	
N1	0.62869 (16)	0.49844 (16)	0.26408 (10)	0.0148 (3)	
C1	0.57105 (19)	0.75089 (19)	0.23202 (12)	0.0157 (4)	
C2	0.5522 (2)	0.7720 (2)	0.15149 (13)	0.0196 (4)	
H2	0.610487	0.732477	0.116805	0.023*	
C3	0.4485 (2)	0.8504 (2)	0.12177 (14)	0.0252 (5)	
H3	0.436059	0.864985	0.066854	0.030*	
C4	0.3633 (2)	0.9074 (2)	0.17236 (15)	0.0262 (5)	
H4	0.291936	0.960845	0.151920	0.031*	
C5	0.3811 (2)	0.8873 (2)	0.25255 (15)	0.0245 (5)	
H5	0.322160	0.926754	0.286924	0.029*	
C6	0.4851 (2)	0.8094 (2)	0.28262 (14)	0.0205 (4)	
H6	0.497801	0.795870	0.337662	0.025*	
C7	0.76765 (19)	0.68021 (19)	0.36417 (12)	0.0158 (4)	
C8	0.8034 (2)	0.8032 (2)	0.38425 (13)	0.0202 (4)	
H8	0.784752	0.865338	0.346625	0.024*	
C9	0.8660 (2)	0.8352 (2)	0.45895 (13)	0.0248 (5)	
H9	0.889935	0.919186	0.472459	0.030*	
C10	0.8937 (2)	0.7448 (2)	0.51411 (13)	0.0249 (5)	
H10	0.935525	0.767132	0.565514	0.030*	
C11	0.8605 (2)	0.6222 (2)	0.49425 (13)	0.0229 (5)	
H11	0.880453	0.560347	0.531933	0.028*	
C12	0.7980 (2)	0.5890 (2)	0.41919 (12)	0.0185 (4)	
H12	0.776046	0.504651	0.405501	0.022*	
C13	0.83060 (19)	0.63487 (19)	0.20303 (12)	0.0153 (4)	
C14	0.9004 (2)	0.7454 (2)	0.19064 (12)	0.0181 (4)	
H14	0.870581	0.821089	0.212779	0.022*	
C15	1.0130 (2)	0.7446 (2)	0.14607 (13)	0.0215 (5)	
H15	1.060562	0.819759	0.137526	0.026*	
C16	1.0565 (2)	0.6340 (2)	0.11391 (13)	0.0237 (5)	
H16	1.134730	0.633353	0.084002	0.028*	
C17	0.9866 (2)	0.5248 (2)	0.12519 (13)	0.0236 (5)	
H17	1.015904	0.449618	0.102114	0.028*	
C18	0.8737 (2)	0.5239 (2)	0.16997 (13)	0.0189 (4)	
H18	0.826224	0.448500	0.178013	0.023*	
C19	0.5175 (2)	0.29576 (19)	0.33066 (12)	0.0152 (4)	
C20	0.6497 (2)	0.2436 (2)	0.33518 (12)	0.0182 (4)	
H20	0.722698	0.281398	0.310350	0.022*	
C21	0.6734 (2)	0.1357 (2)	0.37646 (13)	0.0227 (5)	
H21	0.763352	0.100050	0.380535	0.027*	
C22	0.5661 (2)	0.0803 (2)	0.41155 (14)	0.0247 (5)	

H22	0.582976	0.006646	0.439609	0.030*	
C23	0.4346 (2)	0.1308 (2)	0.40634 (14)	0.0245 (5)	
H23	0.361194	0.091130	0.429795	0.029*	
C24	0.4104 (2)	0.2395 (2)	0.36678 (12)	0.0195 (4)	
H24	0.320791	0.275719	0.364244	0.023*	
C25	0.39051 (19)	0.39030 (19)	0.18288 (11)	0.0147 (4)	
C26	0.2980 (2)	0.2889 (2)	0.17448 (13)	0.0184 (4)	
H26	0.282939	0.241214	0.218841	0.022*	
C27	0.2284 (2)	0.2582 (2)	0.10121 (13)	0.0208 (4)	
H27	0.166375	0.188757	0.095418	0.025*	
C28	0.2484 (2)	0.3278 (2)	0.03660 (13)	0.0204 (4)	
H28	0.200121	0.306118	−0.013360	0.024*	
C29	0.3386 (2)	0.4291 (2)	0.04445 (12)	0.0214 (4)	
H29	0.351457	0.477553	0.000110	0.026*	
C30	0.4104 (2)	0.4597 (2)	0.11750 (12)	0.0201 (4)	
H30	0.473351	0.528467	0.122725	0.024*	
C31	0.37409 (19)	0.52727 (19)	0.33263 (11)	0.0141 (4)	
C32	0.2454 (2)	0.5688 (2)	0.30098 (13)	0.0223 (5)	
H32	0.208129	0.541282	0.249618	0.027*	
C33	0.1714 (2)	0.6509 (2)	0.34489 (14)	0.0286 (5)	
H33	0.083476	0.679621	0.323240	0.034*	
C34	0.2246 (2)	0.6910 (2)	0.41956 (13)	0.0247 (5)	
H34	0.173998	0.748083	0.448792	0.030*	
C35	0.3519 (2)	0.6480 (2)	0.45208 (13)	0.0203 (4)	
H35	0.388190	0.674706	0.503774	0.024*	
C36	0.4257 (2)	0.56592 (19)	0.40883 (12)	0.0161 (4)	
H36	0.512425	0.535668	0.431262	0.019*	
B1	0.9734 (2)	0.9172 (2)	0.72177 (15)	0.0247 (5)	
F1	0.9085 (2)	0.9440 (2)	0.78950 (13)	0.0427 (6)	0.872 (3)
F2	1.10792 (15)	0.96714 (16)	0.73349 (10)	0.0332 (5)	0.872 (3)
F3	0.9817 (3)	0.79082 (18)	0.70909 (14)	0.0496 (7)	0.872 (3)
F4	0.9052 (2)	0.9718 (2)	0.65640 (11)	0.0535 (7)	0.872 (3)
F1′	0.9596 (18)	0.9723 (15)	0.7953 (6)	0.067 (5)	0.128 (3)
F2′	1.0385 (15)	0.9971 (11)	0.6767 (8)	0.067 (5)	0.128 (3)
F3′	1.0466 (14)	0.8091 (9)	0.7339 (10)	0.056 (5)	0.128 (3)
F4′	0.8399 (7)	0.8879 (10)	0.6880 (7)	0.040 (4)	0.128 (3)
C1S	0.7670 (2)	0.1105 (2)	0.09896 (14)	0.0279 (5)	
H1S	0.821917	0.092867	0.150168	0.033*	
Cl1	0.75419 (7)	−0.02595 (6)	0.03878 (4)	0.03460 (16)	0.9628 (9)
Cl2	0.60017 (7)	0.15976 (7)	0.11801 (5)	0.04246 (19)	0.9628 (9)
Cl3	0.85322 (8)	0.22581 (7)	0.05260 (5)	0.0483 (2)	0.9628 (9)
Cl1′	0.8811 (16)	0.0694 (15)	0.0272 (8)	0.03460 (16)	0.0372 (9)
Cl3′	0.7185 (19)	0.2620 (10)	0.0726 (13)	0.0483 (2)	0.0372 (9)
Cl2′	0.6530 (16)	0.0073 (13)	0.1361 (11)	0.04246 (19)	0.0372 (9)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.0129 (2)	0.0141 (3)	0.0125 (2)	0.00158 (18)	0.00078 (18)	−0.00049 (19)
P2	0.0146 (2)	0.0142 (3)	0.0102 (2)	0.00071 (18)	0.00114 (18)	−0.00015 (19)
N1	0.0149 (7)	0.0151 (8)	0.0148 (8)	0.0016 (6)	0.0026 (6)	0.0002 (7)
C1	0.0133 (8)	0.0140 (10)	0.0194 (11)	0.0006 (7)	−0.0007 (7)	0.0004 (8)
C2	0.0182 (9)	0.0204 (11)	0.0201 (11)	0.0006 (8)	0.0007 (8)	0.0018 (9)
C3	0.0227 (10)	0.0260 (12)	0.0257 (12)	0.0006 (9)	−0.0064 (9)	0.0084 (10)
C4	0.0182 (10)	0.0211 (12)	0.0387 (14)	0.0025 (9)	−0.0028 (9)	0.0087 (10)
C5	0.0181 (10)	0.0179 (11)	0.0384 (14)	0.0041 (8)	0.0069 (9)	−0.0001 (10)
C6	0.0209 (10)	0.0182 (11)	0.0224 (11)	0.0019 (8)	0.0027 (8)	−0.0006 (9)
C7	0.0125 (8)	0.0197 (10)	0.0151 (10)	0.0011 (7)	0.0023 (7)	−0.0009 (8)
C8	0.0212 (10)	0.0212 (11)	0.0182 (11)	0.0010 (8)	0.0025 (8)	−0.0019 (9)
C9	0.0232 (11)	0.0283 (13)	0.0219 (12)	−0.0013 (9)	0.0007 (9)	−0.0104 (10)
C10	0.0174 (10)	0.0414 (15)	0.0151 (11)	0.0000 (9)	0.0000 (8)	−0.0070 (10)
C11	0.0187 (10)	0.0347 (13)	0.0154 (11)	0.0042 (9)	0.0001 (8)	0.0040 (9)
C12	0.0171 (9)	0.0217 (11)	0.0169 (10)	0.0017 (8)	0.0022 (8)	0.0008 (8)
C13	0.0129 (8)	0.0199 (10)	0.0131 (10)	0.0024 (7)	−0.0003 (7)	0.0018 (8)
C14	0.0180 (9)	0.0195 (11)	0.0163 (10)	−0.0001 (8)	−0.0006 (8)	0.0008 (8)
C15	0.0174 (9)	0.0268 (12)	0.0198 (11)	−0.0058 (8)	−0.0007 (8)	0.0041 (9)
C16	0.0146 (9)	0.0369 (14)	0.0204 (11)	0.0055 (9)	0.0043 (8)	0.0057 (10)
C17	0.0223 (10)	0.0263 (12)	0.0232 (12)	0.0078 (9)	0.0069 (9)	0.0016 (9)
C18	0.0181 (9)	0.0186 (11)	0.0205 (11)	0.0020 (8)	0.0036 (8)	0.0014 (8)
C19	0.0192 (9)	0.0155 (10)	0.0107 (9)	0.0012 (7)	0.0008 (7)	−0.0020 (8)
C20	0.0204 (9)	0.0194 (11)	0.0156 (10)	0.0033 (8)	0.0049 (8)	0.0003 (8)
C21	0.0298 (11)	0.0196 (11)	0.0195 (11)	0.0076 (9)	0.0052 (9)	0.0001 (9)
C22	0.0352 (12)	0.0183 (11)	0.0218 (12)	0.0060 (9)	0.0070 (9)	0.0049 (9)
C23	0.0284 (11)	0.0243 (12)	0.0224 (12)	−0.0003 (9)	0.0085 (9)	0.0064 (9)
C24	0.0206 (10)	0.0220 (11)	0.0162 (11)	0.0022 (8)	0.0026 (8)	0.0035 (9)
C25	0.0161 (9)	0.0177 (10)	0.0102 (9)	0.0020 (7)	0.0014 (7)	−0.0026 (8)
C26	0.0190 (9)	0.0199 (11)	0.0164 (10)	−0.0014 (8)	0.0024 (8)	0.0004 (8)
C27	0.0175 (9)	0.0218 (11)	0.0223 (11)	−0.0019 (8)	−0.0015 (8)	−0.0032 (9)
C28	0.0196 (10)	0.0251 (12)	0.0152 (10)	0.0045 (8)	−0.0028 (8)	−0.0070 (9)
C29	0.0277 (11)	0.0246 (12)	0.0117 (10)	0.0011 (9)	0.0001 (8)	0.0024 (9)
C30	0.0243 (10)	0.0197 (11)	0.0162 (11)	−0.0031 (8)	0.0023 (8)	0.0004 (8)
C31	0.0152 (8)	0.0170 (10)	0.0104 (9)	0.0009 (7)	0.0021 (7)	0.0000 (7)
C32	0.0178 (9)	0.0334 (13)	0.0150 (11)	0.0042 (9)	−0.0021 (8)	−0.0029 (9)
C33	0.0198 (10)	0.0398 (15)	0.0256 (13)	0.0129 (10)	−0.0008 (9)	−0.0019 (11)
C34	0.0246 (11)	0.0302 (13)	0.0203 (12)	0.0101 (9)	0.0067 (9)	−0.0012 (10)
C35	0.0220 (10)	0.0249 (12)	0.0139 (10)	0.0037 (8)	0.0023 (8)	−0.0029 (9)
C36	0.0164 (9)	0.0197 (10)	0.0119 (10)	0.0026 (8)	−0.0009 (7)	0.0008 (8)
B1	0.0252 (12)	0.0247 (14)	0.0241 (14)	−0.0004 (10)	0.0032 (10)	−0.0035 (11)
F1	0.0458 (12)	0.0362 (12)	0.0516 (13)	0.0010 (9)	0.0338 (10)	−0.0018 (9)
F2	0.0253 (8)	0.0431 (11)	0.0322 (10)	−0.0042 (7)	0.0082 (7)	0.0032 (8)
F3	0.0881 (19)	0.0230 (11)	0.0372 (14)	−0.0085 (11)	0.0084 (12)	−0.0088 (9)
F4	0.0584 (14)	0.0584 (15)	0.0396 (12)	0.0099 (11)	−0.0190 (10)	0.0091 (10)
F1′	0.087 (11)	0.052 (8)	0.069 (9)	−0.009 (7)	0.039 (8)	0.017 (6)

F2′	0.087 (11)	0.052 (8)	0.069 (9)	−0.009 (7)	0.039 (8)	0.017 (6)
F3′	0.061 (11)	0.040 (9)	0.065 (13)	0.006 (8)	0.004 (8)	−0.025 (8)
F4′	0.021 (6)	0.034 (8)	0.062 (9)	0.005 (5)	−0.012 (5)	−0.016 (6)
C1S	0.0330 (12)	0.0253 (13)	0.0251 (13)	0.0042 (10)	0.0012 (10)	0.0009 (10)
Cl1	0.0473 (4)	0.0227 (3)	0.0350 (4)	0.0078 (3)	0.0097 (3)	−0.0010 (3)
Cl2	0.0410 (4)	0.0408 (4)	0.0481 (5)	0.0076 (3)	0.0192 (3)	−0.0086 (3)
Cl3	0.0442 (4)	0.0402 (4)	0.0609 (5)	−0.0183 (3)	0.0070 (4)	0.0095 (4)
Cl1′	0.0473 (4)	0.0227 (3)	0.0350 (4)	0.0078 (3)	0.0097 (3)	−0.0010 (3)
Cl3′	0.0442 (4)	0.0402 (4)	0.0609 (5)	−0.0183 (3)	0.0070 (4)	0.0095 (4)
Cl2′	0.0410 (4)	0.0408 (4)	0.0481 (5)	0.0076 (3)	0.0192 (3)	−0.0086 (3)

Geometric parameters (Å, º)

P1—N1	1.5834 (18)	C21—C22	1.383 (3)
P1—C1	1.7962 (19)	C21—H21	0.9500
P1—C13	1.799 (2)	C22—C23	1.381 (3)
P1—C7	1.804 (2)	C22—H22	0.9500
P2—N1	1.5798 (17)	C23—C24	1.384 (3)
P2—C19	1.793 (2)	C23—H23	0.9500
P2—C31	1.802 (2)	C24—H24	0.9500
P2—C25	1.802 (2)	C25—C30	1.390 (3)
C1—C2	1.393 (3)	C25—C26	1.399 (3)
C1—C6	1.395 (3)	C26—C27	1.387 (3)
C2—C3	1.387 (3)	C26—H26	0.9500
C2—H2	0.9500	C27—C28	1.380 (3)
C3—C4	1.384 (3)	C27—H27	0.9500
C3—H3	0.9500	C28—C29	1.384 (3)
C4—C5	1.385 (3)	C28—H28	0.9500
C4—H4	0.9500	C29—C30	1.391 (3)
C5—C6	1.387 (3)	C29—H29	0.9500
C5—H5	0.9500	C30—H30	0.9500
C6—H6	0.9500	C31—C32	1.388 (3)
C7—C8	1.393 (3)	C31—C36	1.393 (3)
C7—C12	1.396 (3)	C32—C33	1.391 (3)
C8—C9	1.385 (3)	C32—H32	0.9500
C8—H8	0.9500	C33—C34	1.378 (3)
C9—C10	1.388 (3)	C33—H33	0.9500
C9—H9	0.9500	C34—C35	1.387 (3)
C10—C11	1.384 (3)	C34—H34	0.9500
C10—H10	0.9500	C35—C36	1.383 (3)
C11—C12	1.393 (3)	C35—H35	0.9500
C11—H11	0.9500	C36—H36	0.9500
C12—H12	0.9500	B1—F2′	1.361 (6)
C13—C18	1.394 (3)	B1—F3	1.381 (3)
C13—C14	1.395 (3)	B1—F3′	1.384 (6)
C14—C15	1.384 (3)	B1—F4′	1.385 (6)
C14—H14	0.9500	B1—F4	1.389 (3)
C15—C16	1.386 (3)	B1—F1′	1.389 (7)

C15—H15	0.9500	B1—F1	1.389 (3)
C16—C17	1.380 (3)	B1—F2	1.391 (3)
C16—H16	0.9500	C1S—Cl2′	1.732 (8)
C17—C18	1.388 (3)	C1S—Cl3	1.742 (2)
C17—H17	0.9500	C1S—Cl2	1.756 (2)
C18—H18	0.9500	C1S—Cl1	1.765 (2)
C19—C24	1.395 (3)	C1S—Cl3′	1.769 (8)
C19—C20	1.395 (3)	C1S—Cl1′	1.772 (7)
C20—C21	1.391 (3)	C1S—H1S	1.0000
C20—H20	0.9500		

N1—P1—C1	114.95 (9)	C21—C20—H20	120.4
N1—P1—C13	107.55 (9)	C19—C20—H20	120.4
C1—P1—C13	106.98 (9)	C22—C21—C20	120.0 (2)
N1—P1—C7	111.29 (10)	C22—C21—H21	120.0
C1—P1—C7	108.64 (9)	C20—C21—H21	120.0
C13—P1—C7	107.06 (9)	C23—C22—C21	120.9 (2)
N1—P2—C19	109.18 (9)	C23—C22—H22	119.6
N1—P2—C31	113.71 (9)	C21—C22—H22	119.6
C19—P2—C31	106.23 (9)	C22—C23—C24	119.7 (2)
N1—P2—C25	110.65 (9)	C22—C23—H23	120.2
C19—P2—C25	108.14 (9)	C24—C23—H23	120.2
C31—P2—C25	108.70 (9)	C23—C24—C19	120.01 (19)
P2—N1—P1	137.69 (11)	C23—C24—H24	120.0
C2—C1—C6	119.55 (18)	C19—C24—H24	120.0
C2—C1—P1	118.68 (15)	C30—C25—C26	119.34 (19)
C6—C1—P1	121.35 (16)	C30—C25—P2	118.98 (16)
C3—C2—C1	120.2 (2)	C26—C25—P2	121.68 (16)
C3—C2—H2	119.9	C27—C26—C25	119.8 (2)
C1—C2—H2	119.9	C27—C26—H26	120.1
C4—C3—C2	119.8 (2)	C25—C26—H26	120.1
C4—C3—H3	120.1	C28—C27—C26	120.5 (2)
C2—C3—H3	120.1	C28—C27—H27	119.7
C3—C4—C5	120.52 (19)	C26—C27—H27	119.7
C3—C4—H4	119.7	C27—C28—C29	120.15 (19)
C5—C4—H4	119.7	C27—C28—H28	119.9
C4—C5—C6	119.9 (2)	C29—C28—H28	119.9
C4—C5—H5	120.0	C28—C29—C30	119.8 (2)
C6—C5—H5	120.0	C28—C29—H29	120.1
C5—C6—C1	120.0 (2)	C30—C29—H29	120.1
C5—C6—H6	120.0	C25—C30—C29	120.4 (2)
C1—C6—H6	120.0	C25—C30—H30	119.8
C8—C7—C12	119.63 (19)	C29—C30—H30	119.8
C8—C7—P1	121.33 (16)	C32—C31—C36	119.51 (18)
C12—C7—P1	118.95 (16)	C32—C31—P2	122.58 (16)
C9—C8—C7	120.2 (2)	C36—C31—P2	117.74 (14)
C9—C8—H8	119.9	C31—C32—C33	119.6 (2)
C7—C8—H8	119.9	C31—C32—H32	120.2

C8—C9—C10	120.2 (2)	C33—C32—H32	120.2
C8—C9—H9	119.9	C34—C33—C32	120.55 (19)
C10—C9—H9	119.9	C34—C33—H33	119.7
C11—C10—C9	120.0 (2)	C32—C33—H33	119.7
C11—C10—H10	120.0	C33—C34—C35	120.1 (2)
C9—C10—H10	120.0	C33—C34—H34	120.0
C10—C11—C12	120.3 (2)	C35—C34—H34	120.0
C10—C11—H11	119.9	C36—C35—C34	119.6 (2)
C12—C11—H11	119.9	C36—C35—H35	120.2
C11—C12—C7	119.7 (2)	C34—C35—H35	120.2
C11—C12—H12	120.1	C35—C36—C31	120.57 (18)
C7—C12—H12	120.1	C35—C36—H36	119.7
C18—C13—C14	120.00 (19)	C31—C36—H36	119.7
C18—C13—P1	120.05 (16)	F2′—B1—F3′	112.4 (8)
C14—C13—P1	119.85 (16)	F2′—B1—F4′	111.0 (7)
C15—C14—C13	119.9 (2)	F3′—B1—F4′	109.1 (7)
C15—C14—H14	120.0	F3—B1—F4	110.8 (2)
C13—C14—H14	120.0	F2′—B1—F1′	109.8 (8)
C14—C15—C16	119.9 (2)	F3′—B1—F1′	107.5 (8)
C14—C15—H15	120.0	F4′—B1—F1′	106.8 (7)
C16—C15—H15	120.0	F3—B1—F1	110.7 (2)
C17—C16—C15	120.2 (2)	F4—B1—F1	111.3 (2)
C17—C16—H16	119.9	F3—B1—F2	108.7 (2)
C15—C16—H16	119.9	F4—B1—F2	107.8 (2)
C16—C17—C18	120.5 (2)	F1—B1—F2	107.4 (2)
C16—C17—H17	119.7	Cl3—C1S—Cl2	110.20 (13)
C18—C17—H17	119.7	Cl3—C1S—Cl1	109.71 (13)
C17—C18—C13	119.3 (2)	Cl2—C1S—Cl1	110.29 (13)
C17—C18—H18	120.3	Cl2′—C1S—Cl3′	122.6 (9)
C13—C18—H18	120.3	Cl2′—C1S—Cl1′	123.8 (8)
C24—C19—C20	120.12 (19)	Cl3′—C1S—Cl1′	102.3 (9)
C24—C19—P2	120.14 (15)	Cl3—C1S—H1S	108.9
C20—C19—P2	119.73 (16)	Cl2—C1S—H1S	108.9
C21—C20—C19	119.3 (2)	Cl1—C1S—H1S	108.9

C19—P2—N1—P1	−139.36 (16)	C16—C17—C18—C13	0.6 (3)
C31—P2—N1—P1	−20.9 (2)	C14—C13—C18—C17	0.3 (3)
C25—P2—N1—P1	101.73 (17)	P1—C13—C18—C17	−176.06 (16)
C1—P1—N1—P2	−34.6 (2)	N1—P2—C19—C24	163.33 (16)
C13—P1—N1—P2	−153.64 (15)	C31—P2—C19—C24	40.33 (19)
C7—P1—N1—P2	89.39 (17)	C25—P2—C19—C24	−76.21 (18)
N1—P1—C1—C2	−85.86 (18)	N1—P2—C19—C20	−17.96 (19)
C13—P1—C1—C2	33.46 (19)	C31—P2—C19—C20	−140.97 (17)
C7—P1—C1—C2	148.73 (16)	C25—P2—C19—C20	102.49 (17)
N1—P1—C1—C6	86.61 (19)	C24—C19—C20—C21	−0.4 (3)
C13—P1—C1—C6	−154.07 (17)	P2—C19—C20—C21	−179.12 (16)
C7—P1—C1—C6	−38.8 (2)	C19—C20—C21—C22	0.9 (3)
C6—C1—C2—C3	−0.1 (3)	C20—C21—C22—C23	0.0 (3)

P1—C1—C2—C3	172.51 (17)	C21—C22—C23—C24	−1.2 (4)
C1—C2—C3—C4	−0.3 (3)	C22—C23—C24—C19	1.7 (3)
C2—C3—C4—C5	0.3 (3)	C20—C19—C24—C23	−0.9 (3)
C3—C4—C5—C6	0.0 (3)	P2—C19—C24—C23	177.85 (17)
C4—C5—C6—C1	−0.4 (3)	N1—P2—C25—C30	−29.05 (18)
C2—C1—C6—C5	0.4 (3)	C19—P2—C25—C30	−148.59 (16)
P1—C1—C6—C5	−171.98 (17)	C31—P2—C25—C30	96.49 (17)
N1—P1—C7—C8	−166.62 (15)	N1—P2—C25—C26	150.98 (16)
C1—P1—C7—C8	−39.09 (19)	C19—P2—C25—C26	31.45 (19)
C13—P1—C7—C8	76.11 (18)	C31—P2—C25—C26	−83.48 (18)
N1—P1—C7—C12	16.94 (18)	C30—C25—C26—C27	0.6 (3)
C1—P1—C7—C12	144.47 (16)	P2—C25—C26—C27	−179.47 (15)
C13—P1—C7—C12	−100.32 (17)	C25—C26—C27—C28	−0.7 (3)
C12—C7—C8—C9	−1.4 (3)	C26—C27—C28—C29	0.1 (3)
P1—C7—C8—C9	−177.76 (16)	C27—C28—C29—C30	0.8 (3)
C7—C8—C9—C10	0.2 (3)	C26—C25—C30—C29	0.3 (3)
C8—C9—C10—C11	0.8 (3)	P2—C25—C30—C29	−179.69 (16)
C9—C10—C11—C12	−0.6 (3)	C28—C29—C30—C25	−0.9 (3)
C10—C11—C12—C7	−0.6 (3)	N1—P2—C31—C32	116.20 (19)
C8—C7—C12—C11	1.6 (3)	C19—P2—C31—C32	−123.70 (19)
P1—C7—C12—C11	178.05 (15)	C25—P2—C31—C32	−7.5 (2)
N1—P1—C13—C18	−6.98 (19)	N1—P2—C31—C36	−59.10 (19)
C1—P1—C13—C18	−130.97 (16)	C19—P2—C31—C36	61.01 (18)
C7—P1—C13—C18	112.72 (17)	C25—P2—C31—C36	177.17 (16)
N1—P1—C13—C14	176.65 (15)	C36—C31—C32—C33	1.7 (3)
C1—P1—C13—C14	52.66 (18)	P2—C31—C32—C33	−173.55 (19)
C7—P1—C13—C14	−63.65 (18)	C31—C32—C33—C34	−0.2 (4)
C18—C13—C14—C15	−0.5 (3)	C32—C33—C34—C35	−1.0 (4)
P1—C13—C14—C15	175.84 (15)	C33—C34—C35—C36	0.7 (4)
C13—C14—C15—C16	−0.1 (3)	C34—C35—C36—C31	0.7 (3)
C14—C15—C16—C17	1.0 (3)	C32—C31—C36—C35	−1.9 (3)
C15—C16—C17—C18	−1.2 (3)	P2—C31—C36—C35	173.51 (17)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8···F2^{i}	0.95	2.57	3.394 (3)	145
C8—H8···F2′^{i}	0.95	2.32	2.903 (8)	119
C14—H14···F2^{i}	0.95	2.44	3.333 (3)	157
C18—H18···Cl3′	0.95	2.78	3.481 (18)	131
C20—H20···F3′^{ii}	0.95	2.61	3.308 (15)	131
C26—H26···F4′^{iii}	0.95	2.52	3.427 (14)	160
C1*S*—H1*S*···F2^{ii}	1.00	2.15	3.132 (3)	166

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