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Crystal structure of (3-carboxypropyl)-triphenylphosphonium hexafluorido-phosphate

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In the title molecular salt, $C_{22}H_{22}O_2P^+ \cdot PF_6^-$, the side chain of the cation adopts an *anti–gauche* conformation [P—C—C—C and C—C—C—C torsion angles = −179.11 (10) and −77.18 (16)°, respectively]. In the crystal, the cations are linked into carboxylic acid inversion dimers by pairs of O—H⋯O hydrogen bonds. Weak C—H⋯F and C—H⋯(F,F) hydrogen bonds connect the components into a three-dimensional network, but there are no aromatic π–π stacking interactions.

Keywords: crystal structure; phosphonium salt; hydrogen bonding.

CCDC reference: 1030392

1. Related literature

For structures of related compounds, see: Li & Mak (1996); Wu *et al.* (2007). For compounds containing related metallated structures, see: Li & Mak (1997); Sabounchei *et al.* (2011). For the use of phosphonium compounds as Wittig reagents, see: Hoffman (2001), as biocodal agents, see: Kanazawa *et al.* (1993) and as phase transfer agents, see: Starks (1971).
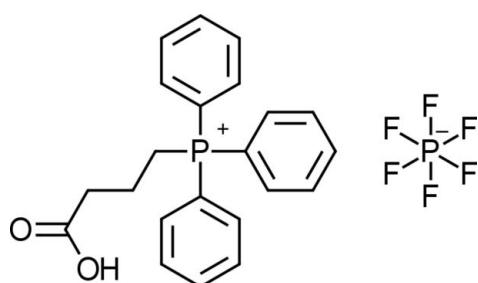
2. Experimental

2.1. Crystal data

$C_{22}H_{22}O_2P^+ \cdot PF_6^-$	$\gamma = 65.495\,(1)°$
$M_r = 494.33$	$V = 1107.46\,(3)$ Å³
Triclinic, $P\bar{1}$	$Z = 2$
$a = 9.3307\,(1)$ Å	Mo $K\alpha$ radiation
$b = 10.6773\,(2)$ Å	$\mu = 0.26$ mm⁻¹
$c = 12.8129\,(2)$ Å	$T = 100$ K
$\alpha = 72.460\,(1)°$	$0.29 \times 0.16 \times 0.07$ mm
$\beta = 82.307\,(1)°$	

2.2. Data collection

Bruker APEXII CCD diffractometer	37843 measured reflections
Absorption correction: multi-scan (*SADABS*; Bruker, 2014)	5269 independent reflections
$T_{min} = 0.865$, $T_{max} = 0.947$	4426 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.035$

2.3. Refinement

$R[F^2 > 2\sigma(F^2)] = 0.034$	290 parameters
$wR(F^2) = 0.087$	H-atom parameters constrained
$S = 1.06$	$\Delta\rho_{max} = 0.42$ e Å⁻³
5269 reflections	$\Delta\rho_{min} = -0.36$ e Å⁻³

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O1—H1⋯O2ⁱ	0.84	1.80	2.6285 (15)	171
C1—H1A⋯F2	0.99	2.48	3.455 (2)	168
C1—H1A⋯F3	0.99	2.50	3.1656 (19)	124
C22—H22⋯F4ⁱⁱ	0.95	2.51	3.3924 (18)	155

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

Data collection: *APEX2* (Bruker, 2014); cell refinement: *SAINT* (Bruker, 2013); data reduction: *SAINT*; program(s) used to solve structure: *SHELXT* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2008); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2*.

Acknowledgements

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supporting information

Crystal structure of (3-carboxypropyl)triphenylphosphonium hexafluoridophosphate

Patrick C. Hillesheim, Kent A. Scipione and Sean L. Stokes

S1. Synthesis and crystallization

A 1.0g (2.3mmol) sample of 3-carboxypropyltriphenylphosphonium chloride and 0.4g (2.4mmol) of sodium hexafluoro-phosphate were dissolved in 40mL of water. A white precipitate immediately formed and the slurry was stirred for 1 hour. The mixture was filtered, the solid was washed with water (3 x 25mL), and dried under high vacuum to yield a white solid. Yield: 0.65g (80.8%). Single crystals suitable for X-ray diffraction were grown from slow evaporation of dichloro-methane. ^{1}H NMR (CHLOROFORM-*d* ,300MHz): δ = 7.90–8.02 (*m*, 9H), 7.77–7.89 (*m*, 6H), 3.54–3.72 (*m*, 2H), 2.66 (*t*, *J* = 6.6 Hz, 2H), 2.04–2.07 p.p.m. (*m*, 2H). ^{13}C NMR (CHLOROFORM-*d* ,75MHz): δ = 174.0, 136.4, 134.9, 131.6, 120.3, 119.1, 34.2, 34.0, 22.0, 19.2 p.p.m. HRMS (ESI–TOF) *m*/*z*: [*M*$^+$] Calcd for $C_{22}H_{22}O_2P^+$ 349.381; found 349.1355. [*M*$^-$] Calcd for PF_6 144.965; found 144.9632.

S2. Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H = 0.95 to 0.99 Å, O—H = 0.84 Å) and were included in the refinement in the riding model approximation with $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}(O)$.

S3. Comment

Organic phosphonium cations have been used as phase transfer catalysts (Starks, 1971), biocidal agents (Kanazawa *et al.* 1993), and as reagents for Wittig reactions (Hoffman, 2001). There are few examples in the crystallographic literature, however, of triphenylphosphonium cations bearing a carboxylic acid functional group.

   In the title compound, 3-carboxypropyltriphenylphosphonium hexafluorophosphate (Fig. 1), crystallizes in a triclinic unit cell with a single cation-anion pair in the asymmetric unit. The dominant intermolecular interactions is hydrogen bonding from the carboxylic acid moiety on the cation (Table 1). The alkyl chain attached to the phosphorous deviates from the expected staggered conformation, showing a rotation at the C1—C2 carbons. This twist in the carbons is likely the cause of the unusual torsion angles observed in the three phenyl rings (Table 2). The phenyl ring that is located under the C2 hydrogens is nearly perpendicular when compared to the other two rings. It is suspected that this perpendicular arrangement of the phenyl ring is assumed to minimize potential steric interactions with the bent portion of the alkyl chain. Interestingly, there are no observed π–π interactions from any of the phenyl rings and there are no weak C—H···F interactions.
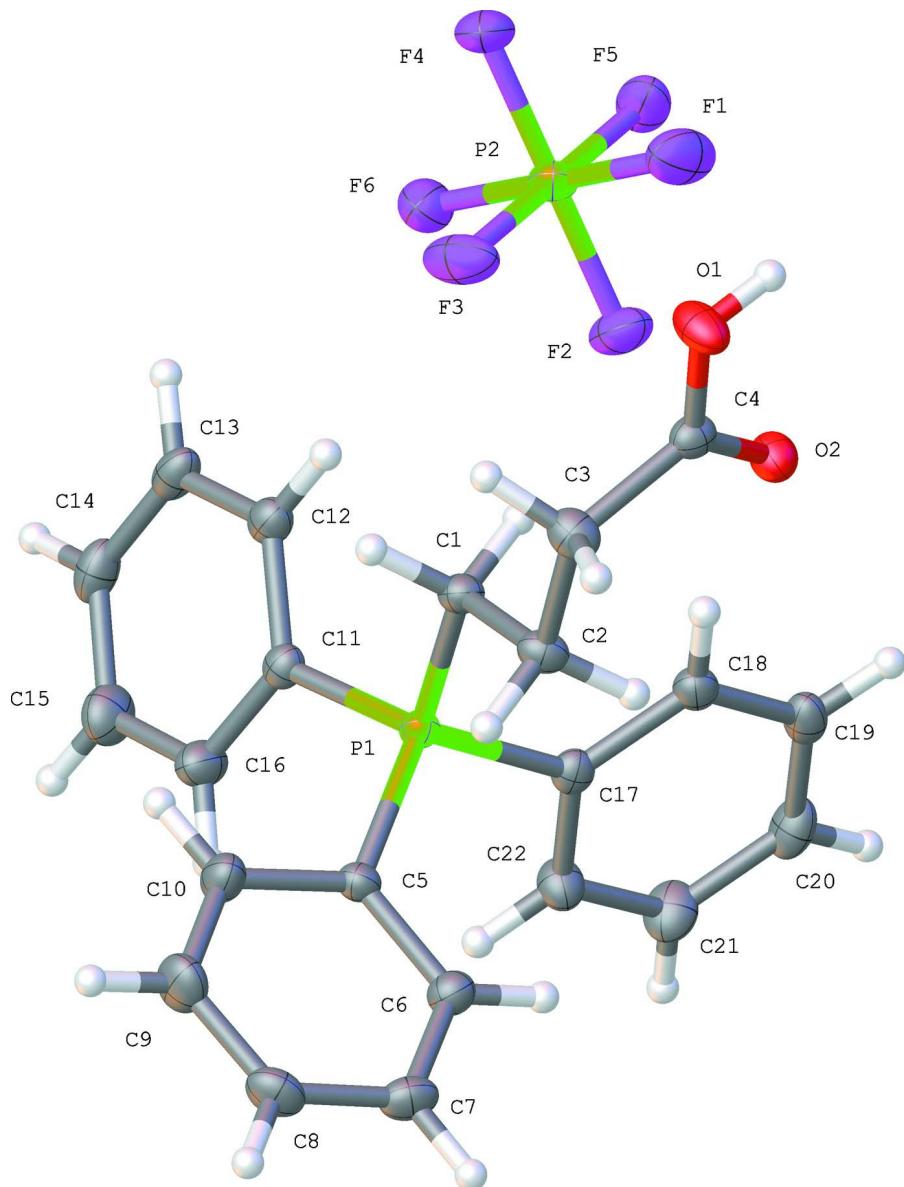
Figure 1

Crystal structure and labeling scheme of compound (1). 50% probablility ellipsoids. Phosphorous is in green, oxygen in red, fluorine in purple, and carbon in grey.

(3-Carboxypropyl)triphenylphosphonium hexafluoridophosphate

Crystal data
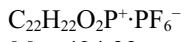
$C_{22}H_{22}O_2P^+ \cdot PF_6^-$	$\gamma = 65.495\,(1)°$
$M_r = 494.33$	$V = 1107.46\,(3)$ Å³
Triclinic, $P\bar{1}$	$Z = 2$
$a = 9.3307\,(1)$ Å	$F(000) = 508$
$b = 10.6773\,(2)$ Å	$D_x = 1.482$ Mg m⁻³
$c = 12.8129\,(2)$ Å	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$\alpha = 72.460\,(1)°$	Cell parameters from 9960 reflections
$\beta = 82.307\,(1)°$	$\theta = 2.4–27.8°$

$\mu = 0.26$ mm^{-1}
$T = 100$ K

Block, colourless
$0.29 \times 0.16 \times 0.07$ mm

Data collection

Bruker APEXII CCD diffractometer	37843 measured reflections
Radiation source: fine-focus sealed tube	5269 independent reflections
Graphite monochromator	4426 reflections with $I > 2\sigma(I)$
Detector resolution: 7.9 pixels mm^{-1}	$R_{int} = 0.035$
ω and φ scans	$\theta_{max} = 27.9°$, $\theta_{min} = 1.7°$
Absorption correction: multi-scan	$h = -12 \rightarrow 11$
(*SADABS*; Bruker, 2014)	$k = -14 \rightarrow 14$
$T_{min} = 0.865$, $T_{max} = 0.947$	$l = -16 \rightarrow 16$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.034$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.087$	
$S = 1.06$	H-atom parameters constrained
5269 reflections	$w = 1/[\sigma^2(F_o^2) + (0.0364P)^2 + 0.4848P]$
290 parameters	where $P = (F_o^2 + 2F_c^2)/3$
0 restraints	$(\Delta/\sigma)_{max} = 0.001$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{max} = 0.42$ e Å^{-3}
	$\Delta\rho_{min} = -0.36$ e Å^{-3}

Special details

Experimental. Absorption correction: SADABS-2014/2 (Bruker, 2014) was used for absorption correction. wR2(int) was 0.0583 before and 0.0488 after correction. The Ratio of minimum to maximum transmission is 0.9133. The $\lambda/2$ correction factor is 0.00150.

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}
P1	0.42475 (4)	0.61911 (4)	0.72374 (3)	0.01582 (9)
P2	0.21110 (5)	0.22893 (4)	0.72342 (3)	0.02490 (10)
F2	0.18704 (11)	0.33829 (11)	0.79338 (8)	0.0344 (2)
F6	0.13566 (12)	0.36150 (10)	0.61884 (8)	0.0352 (2)
F4	0.23378 (12)	0.12048 (10)	0.65303 (8)	0.0342 (2)
F5	0.03862 (12)	0.23173 (12)	0.76017 (8)	0.0387 (3)
F3	0.38172 (12)	0.22834 (11)	0.68553 (9)	0.0406 (3)
O2	0.48983 (12)	0.17127 (10)	0.96794 (8)	0.0235 (2)
F1	0.28391 (14)	0.09741 (11)	0.82789 (9)	0.0453 (3)
O1	0.66794 (13)	−0.01961 (11)	0.91753 (10)	0.0295 (3)
H1	0.6104	−0.0598	0.9557	0.044*
C5	0.58121 (16)	0.67621 (14)	0.71818 (11)	0.0172 (3)
C18	0.23325 (17)	0.57402 (16)	0.90634 (11)	0.0208 (3)
H18	0.2785	0.4755	0.9084	0.025*

C11	0.32167 (16)	0.70037 (15)	0.59658 (11)	0.0184 (3)
C17	0.28593 (16)	0.67064 (15)	0.82977 (11)	0.0182 (3)
C4	0.60945 (17)	0.11522 (15)	0.91839 (11)	0.0200 (3)
C1	0.51033 (16)	0.42771 (14)	0.74813 (12)	0.0188 (3)
H1A	0.4262	0.3910	0.7713	0.023*
H1B	0.5569	0.4039	0.6789	0.023*
C12	0.25839 (16)	0.62504 (16)	0.55808 (12)	0.0217 (3)
H12	0.2804	0.5273	0.5944	0.026*
C2	0.63811 (16)	0.35274 (15)	0.83609 (12)	0.0207 (3)
H2A	0.7222	0.3896	0.8137	0.025*
H2B	0.5918	0.3743	0.9060	0.025*
C6	0.62009 (17)	0.69945 (15)	0.80985 (12)	0.0213 (3)
H6	0.5526	0.7019	0.8723	0.026*
C19	0.11368 (17)	0.62379 (17)	0.97956 (12)	0.0243 (3)
H19	0.0781	0.5585	1.0325	0.029*
C22	0.22021 (17)	0.81526 (15)	0.82747 (12)	0.0239 (3)
H22	0.2574	0.8806	0.7762	0.029*
C13	0.16332 (17)	0.69379 (18)	0.46650 (12)	0.0255 (3)
H13	0.1208	0.6429	0.4394	0.031*
C3	0.70880 (17)	0.19153 (15)	0.85265 (13)	0.0240 (3)
H3A	0.7292	0.1726	0.7799	0.029*
H3B	0.8116	0.1506	0.8893	0.029*
C14	0.13030 (18)	0.83680 (18)	0.41448 (12)	0.0276 (3)
H14	0.0626	0.8843	0.3530	0.033*
C16	0.29145 (19)	0.84353 (16)	0.54243 (13)	0.0262 (3)
H16	0.3363	0.8942	0.5677	0.031*
C7	0.75827 (17)	0.71902 (16)	0.80929 (13)	0.0253 (3)
H7	0.7852	0.7355	0.8714	0.030*
C8	0.85698 (18)	0.71457 (17)	0.71847 (14)	0.0275 (3)
H8	0.9528	0.7254	0.7193	0.033*
C10	0.67893 (18)	0.67535 (17)	0.62542 (12)	0.0251 (3)
H10	0.6511	0.6618	0.5622	0.030*
C21	0.10050 (19)	0.86258 (17)	0.90047 (13)	0.0296 (3)
H21	0.0552	0.9609	0.8990	0.035*
C20	0.04614 (17)	0.76728 (18)	0.97598 (12)	0.0271 (3)
H20	−0.0373	0.8007	1.0251	0.033*
C9	0.81677 (19)	0.69444 (19)	0.62641 (14)	0.0316 (4)
H9	0.8839	0.6937	0.5637	0.038*
C15	0.1951 (2)	0.91087 (18)	0.45130 (13)	0.0315 (4)
H15	0.1735	1.0083	0.4141	0.038*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.01569 (17)	0.01745 (17)	0.01619 (17)	−0.00773 (13)	0.00018 (13)	−0.00564 (13)
P2	0.0302 (2)	0.0301 (2)	0.0236 (2)	−0.01993 (18)	−0.00010 (16)	−0.00870 (16)
F2	0.0362 (5)	0.0439 (6)	0.0369 (5)	−0.0220 (5)	0.0021 (4)	−0.0225 (5)
F6	0.0485 (6)	0.0327 (5)	0.0294 (5)	−0.0224 (5)	−0.0021 (4)	−0.0055 (4)

F4	0.0490 (6)	0.0336 (5)	0.0328 (5)	−0.0257 (5)	−0.0006 (4)	−0.0132 (4)
F5	0.0403 (6)	0.0640 (7)	0.0306 (5)	−0.0381 (5)	0.0065 (4)	−0.0166 (5)
F3	0.0308 (5)	0.0479 (6)	0.0599 (7)	−0.0238 (5)	0.0105 (5)	−0.0308 (5)
O2	0.0238 (5)	0.0203 (5)	0.0248 (5)	−0.0076 (4)	0.0042 (4)	−0.0074 (4)
F1	0.0651 (7)	0.0398 (6)	0.0371 (6)	−0.0275 (5)	−0.0218 (5)	−0.0001 (5)
O1	0.0294 (6)	0.0194 (5)	0.0389 (7)	−0.0107 (5)	0.0116 (5)	−0.0102 (5)
C5	0.0165 (6)	0.0172 (6)	0.0193 (7)	−0.0080 (5)	0.0001 (5)	−0.0053 (5)
C18	0.0214 (7)	0.0237 (7)	0.0199 (7)	−0.0115 (6)	−0.0009 (5)	−0.0058 (6)
C11	0.0157 (6)	0.0226 (7)	0.0173 (6)	−0.0076 (5)	0.0014 (5)	−0.0070 (5)
C17	0.0149 (6)	0.0221 (7)	0.0175 (6)	−0.0070 (5)	−0.0012 (5)	−0.0054 (5)
C4	0.0216 (7)	0.0181 (6)	0.0192 (7)	−0.0060 (5)	−0.0033 (5)	−0.0052 (5)
C1	0.0192 (7)	0.0192 (6)	0.0209 (7)	−0.0089 (5)	0.0005 (5)	−0.0077 (5)
C12	0.0193 (7)	0.0283 (7)	0.0208 (7)	−0.0119 (6)	0.0023 (5)	−0.0087 (6)
C2	0.0182 (7)	0.0185 (7)	0.0260 (7)	−0.0077 (5)	−0.0021 (5)	−0.0054 (6)
C6	0.0210 (7)	0.0230 (7)	0.0219 (7)	−0.0089 (6)	0.0002 (6)	−0.0087 (6)
C19	0.0216 (7)	0.0361 (8)	0.0194 (7)	−0.0169 (6)	0.0012 (6)	−0.0060 (6)
C22	0.0244 (7)	0.0200 (7)	0.0238 (7)	−0.0068 (6)	0.0029 (6)	−0.0053 (6)
C13	0.0201 (7)	0.0415 (9)	0.0212 (7)	−0.0151 (7)	0.0017 (6)	−0.0138 (6)
C3	0.0186 (7)	0.0208 (7)	0.0296 (8)	−0.0061 (6)	0.0011 (6)	−0.0055 (6)
C14	0.0199 (7)	0.0402 (9)	0.0183 (7)	−0.0071 (6)	−0.0011 (6)	−0.0084 (6)
C16	0.0319 (8)	0.0241 (7)	0.0248 (8)	−0.0124 (6)	−0.0042 (6)	−0.0060 (6)
C7	0.0233 (7)	0.0252 (7)	0.0307 (8)	−0.0088 (6)	−0.0061 (6)	−0.0108 (6)
C8	0.0209 (7)	0.0291 (8)	0.0361 (9)	−0.0142 (6)	−0.0014 (6)	−0.0070 (7)
C10	0.0264 (8)	0.0345 (8)	0.0204 (7)	−0.0167 (7)	0.0038 (6)	−0.0107 (6)
C21	0.0264 (8)	0.0255 (8)	0.0293 (8)	−0.0020 (6)	0.0028 (6)	−0.0102 (6)
C20	0.0175 (7)	0.0396 (9)	0.0217 (7)	−0.0071 (6)	0.0029 (6)	−0.0122 (7)
C9	0.0264 (8)	0.0440 (10)	0.0297 (8)	−0.0213 (7)	0.0081 (7)	−0.0108 (7)
C15	0.0356 (9)	0.0268 (8)	0.0251 (8)	−0.0078 (7)	−0.0054 (7)	−0.0018 (6)

Geometric parameters (Å, º)

P1—C5	1.7867 (14)	C2—H2B	0.9900
P1—C11	1.7910 (14)	C2—C3	1.5227 (19)
P1—C17	1.7930 (14)	C6—H6	0.9500
P1—C1	1.8000 (14)	C6—C7	1.388 (2)
P2—F2	1.6053 (10)	C19—H19	0.9500
P2—F6	1.6054 (10)	C19—C20	1.382 (2)
P2—F4	1.6044 (10)	C22—H22	0.9500
P2—F5	1.6058 (10)	C22—C21	1.384 (2)
P2—F3	1.5990 (10)	C13—H13	0.9500
P2—F1	1.5951 (11)	C13—C14	1.386 (2)
O2—C4	1.2216 (17)	C3—H3A	0.9900
O1—H1	0.8400	C3—H3B	0.9900
O1—C4	1.3140 (17)	C14—H14	0.9500
C5—C6	1.3919 (19)	C14—C15	1.382 (2)
C5—C10	1.3977 (19)	C16—H16	0.9500
C18—H18	0.9500	C16—C15	1.388 (2)
C18—C17	1.3964 (19)	C7—H7	0.9500

C18—C19	1.391 (2)	C7—C8	1.384 (2)
C11—C12	1.396 (2)	C8—H8	0.9500
C11—C16	1.397 (2)	C8—C9	1.384 (2)
C17—C22	1.3975 (19)	C10—H10	0.9500
C4—C3	1.496 (2)	C10—C9	1.385 (2)
C1—H1A	0.9900	C21—H21	0.9500
C1—H1B	0.9900	C21—C20	1.390 (2)
C1—C2	1.5352 (19)	C20—H20	0.9500
C12—H12	0.9500	C9—H9	0.9500
C12—C13	1.386 (2)	C15—H15	0.9500
C2—H2A	0.9900		
C5—P1—C11	110.19 (6)	C3—C2—C1	110.82 (12)
C5—P1—C17	110.68 (6)	C3—C2—H2A	109.5
C5—P1—C1	107.80 (6)	C3—C2—H2B	109.5
C11—P1—C17	107.83 (6)	C5—C6—H6	120.3
C11—P1—C1	109.51 (6)	C7—C6—C5	119.50 (13)
C17—P1—C1	110.83 (7)	C7—C6—H6	120.3
F2—P2—F6	89.92 (5)	C18—C19—H19	119.7
F2—P2—F5	89.94 (5)	C20—C19—C18	120.63 (14)
F6—P2—F5	89.47 (6)	C20—C19—H19	119.7
F4—P2—F2	179.53 (6)	C17—C22—H22	120.3
F4—P2—F6	89.72 (5)	C21—C22—C17	119.49 (14)
F4—P2—F5	89.76 (5)	C21—C22—H22	120.3
F3—P2—F2	89.89 (5)	C12—C13—H13	120.0
F3—P2—F6	89.71 (6)	C12—C13—C14	119.99 (14)
F3—P2—F4	90.41 (5)	C14—C13—H13	120.0
F3—P2—F5	179.17 (7)	C4—C3—C2	115.15 (12)
F1—P2—F2	89.99 (6)	C4—C3—H3A	108.5
F1—P2—F6	179.28 (6)	C4—C3—H3B	108.5
F1—P2—F4	90.37 (6)	C2—C3—H3A	108.5
F1—P2—F5	89.82 (6)	C2—C3—H3B	108.5
F1—P2—F3	91.00 (6)	H3A—C3—H3B	107.5
C4—O1—H1	109.5	C13—C14—H14	119.7
C6—C5—P1	120.87 (11)	C15—C14—C13	120.53 (14)
C6—C5—C10	120.30 (13)	C15—C14—H14	119.7
C10—C5—P1	118.10 (11)	C11—C16—H16	120.4
C17—C18—H18	120.4	C15—C16—C11	119.24 (15)
C19—C18—H18	120.4	C15—C16—H16	120.4
C19—C18—C17	119.17 (13)	C6—C7—H7	119.9
C12—C11—P1	119.73 (11)	C8—C7—C6	120.18 (14)
C12—C11—C16	120.37 (13)	C8—C7—H7	119.9
C16—C11—P1	119.58 (11)	C7—C8—H8	119.8
C18—C17—P1	121.95 (11)	C7—C8—C9	120.35 (14)
C18—C17—C22	120.34 (13)	C9—C8—H8	119.8
C22—C17—P1	117.45 (11)	C5—C10—H10	120.3
O2—C4—O1	124.32 (13)	C9—C10—C5	119.44 (14)
O2—C4—C3	123.95 (13)	C9—C10—H10	120.3

O1—C4—C3	111.72 (12)	C22—C21—H21	119.8
P1—C1—H1A	109.1	C22—C21—C20	120.45 (14)
P1—C1—H1B	109.1	C20—C21—H21	119.8
H1A—C1—H1B	107.8	C19—C20—C21	119.90 (14)
C2—C1—P1	112.67 (9)	C19—C20—H20	120.1
C2—C1—H1A	109.1	C21—C20—H20	120.1
C2—C1—H1B	109.1	C8—C9—C10	120.20 (14)
C11—C12—H12	120.2	C8—C9—H9	119.9
C13—C12—C11	119.56 (14)	C10—C9—H9	119.9
C13—C12—H12	120.2	C14—C15—C16	120.28 (15)
C1—C2—H2A	109.5	C14—C15—H15	119.9
C1—C2—H2B	109.5	C16—C15—H15	119.9
H2A—C2—H2B	108.1		
P1—C5—C6—C7	168.72 (11)	C17—P1—C11—C12	−92.86 (12)
P1—C5—C10—C9	−168.71 (12)	C17—P1—C11—C16	80.66 (13)
P1—C11—C12—C13	172.55 (11)	C17—P1—C1—C2	−77.68 (11)
P1—C11—C16—C15	−172.12 (12)	C17—C18—C19—C20	0.8 (2)
P1—C17—C22—C21	173.20 (12)	C17—C22—C21—C20	0.3 (2)
P1—C1—C2—C3	−179.11 (10)	C1—P1—C5—C6	−96.79 (12)
O2—C4—C3—C2	−9.1 (2)	C1—P1—C5—C6	−96.79 (12)
O1—C4—C3—C2	171.88 (13)	C1—P1—C5—C10	73.39 (13)
C5—P1—C11—C12	146.24 (11)	C1—P1—C11—C12	27.83 (13)
C5—P1—C11—C16	−40.23 (13)	C1—P1—C11—C12	27.83 (13)
C5—P1—C17—C18	−131.04 (12)	C1—P1—C11—C16	−158.65 (11)
C5—P1—C17—C22	54.76 (13)	C1—P1—C17—C18	−11.48 (14)
C5—P1—C1—C2	43.58 (12)	C1—P1—C17—C18	−11.48 (14)
C5—C6—C7—C8	−0.4 (2)	C1—P1—C17—C22	174.32 (11)
C5—C10—C9—C8	−0.2 (2)	C1—C2—C3—C4	−77.18 (16)
C18—C17—C22—C21	−1.1 (2)	C12—C11—C16—C15	1.4 (2)
C18—C19—C20—C21	−1.6 (2)	C12—C13—C14—C15	1.8 (2)
C11—P1—C5—C6	143.74 (11)	C6—C5—C10—C9	1.5 (2)
C11—P1—C5—C10	−46.07 (13)	C6—C7—C8—C9	1.7 (2)
C11—P1—C17—C18	108.38 (12)	C19—C18—C17—P1	−173.47 (11)
C11—P1—C17—C22	−65.82 (13)	C19—C18—C17—C22	0.6 (2)
C11—P1—C1—C2	163.47 (10)	C22—C21—C20—C19	1.1 (2)
C11—C12—C13—C14	−0.7 (2)	C13—C14—C15—C16	−1.4 (2)
C11—C16—C15—C14	−0.2 (2)	C16—C11—C12—C13	−0.9 (2)
C17—P1—C5—C6	24.57 (14)	C7—C8—C9—C10	−1.5 (3)
C17—P1—C5—C10	−165.24 (11)	C10—C5—C6—C7	−1.2 (2)

Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2[i]	0.84	1.80	2.6285 (15)	171
C1—H1A···F2	0.99	2.48	3.455 (2)	168

C1—H1*A*···F3	0.99	2.50	3.1656 (19)	124
C22—H22···F4[ii]	0.95	2.51	3.3924 (18)	155

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