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(4-Carboxybenzyl)triphenylphosphonium hexafluoridophosphate tetrahydrofuran monosolvate

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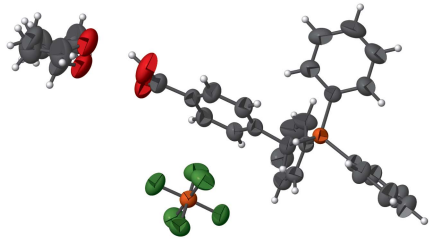
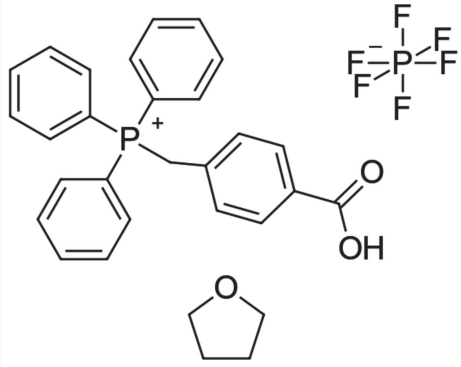
The title compound, $C_{26}H_{22}O_2P^+\cdot PF_6^-\cdot C_4H_7O$, crystallizes as a cation-anion pair with a single solvent molecule in the asymmetric unit. Hydrogen bonding occurs between the carboxylic acid group on the cation and the oxygen atom of the solvent molecule. Longer hydrogen-bonding interactions are observed between fluorine atoms of the anion and H atoms on the phenyl rings of the cation.

3D view



Chemical scheme



Structure description

The title compound (Fig. 1) is a single cation–anion pair with a disordered solvent tetrahydrofuran molecule in the asymmetric unit. There are intermolecular interactions between the hydrogen atom of the carboxylic acid moiety and the tetrahydrofuran solvent. However, the solvent molecule is disordered over two positions making arguments regarding the importance of this interaction difficult. Further, there are weak interactions between phenyl H atoms and fluorine atoms, on the cation and anion respectively, in the crystal, forming an extended network (Table 1). There are no observed π–π interactions, likely due to the torsion angles of the phenyl rings preventing any direct interactions because of steric hindrance.

For structures of similar triphenylphosphonium compounds, see: Bonnet & Kariuki (2006) and Ibrahim *et al.* (2011). For uses of triphenylphosphonium compounds in synthesis, see: Liang (2015).

Synthesis and crystallization

The title compound was synthesized by dissolving 1.0 g (2.09 mmol) of (4-carboxybenzyl)triphenylphosphonium bromide and 0.45 g (2.45 mmol) of potassium hexafluoridophosphate into 20 ml of water. The resultant mixture was stirred overnight, during which time a powder precipitated from solution. The white powder was filtered,

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O3	0.80	1.91	2.654 (4)	154
O1—H1···O3′	0.80	2.42	2.997 (10)	129
C3—H3···F4^{i}	0.93	2.82	3.467 (3)	128
C3—H3···F6^{i}	0.93	2.54	3.468 (3)	172
C4—H4···F2^{ii}	0.93	2.80	3.497 (3)	133
C6—H6···F1^{iii}	0.93	2.62	3.360 (3)	137
C10—H10···F1^{iv}	0.93	2.57	3.410 (4)	150
C11—H11···F6^{iv}	0.93	2.83	3.479 (3)	128
C15—H15···F4	0.93	2.56	3.219 (3)	129
C17—H17···O1^{v}	0.93	2.71	3.402 (4)	131
C19—H19B···F3^{iii}	0.97	2.42	3.380 (3)	172
C19—H19B···F5^{iii}	0.97	2.54	3.275 (3)	132
C30—H30A···O2	0.97	2.66	3.323 (8)	126
C28′—H28C···F3^{vi}	0.97	2.59	3.385 (18)	139

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

washed with water (3 × 25 ml), and dried under high vacuum yielding (4-carboxybenzyl)triphenylphosphonium hexafluoridophosphate. Single crystals suitable for diffraction were grown by slow diffusion of hexane into a tetrahydrofuran solution.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The tetrahydrofuran solvent molecule within the structure is disordered over two positions, of which one part was found to be approximately 67% of the occupancy with the remaining occupancy arising from the disorder. The disorder itself appears to arise from a ring inversion as has been observed in many other structures. The secondary portion of the disorder was restrained to allow for proper modeling.

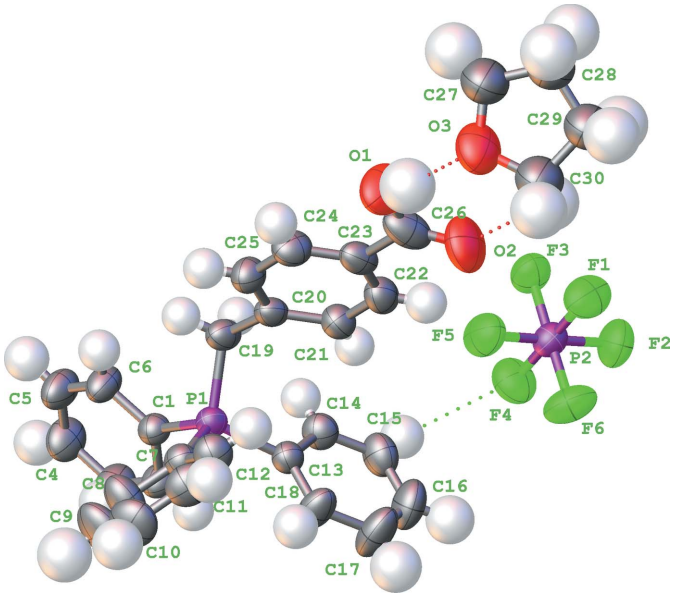


Figure 1
Structure of title compounds with 50% probability ellipsoids. The disordered portion of the solvent molecule is omitted for clarity.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{26}H_{22}O_2P^+·PF_6^-·C_4H_7O$
M_r	613.47
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	170
a, b, c (Å)	10.3724 (4), 11.1647 (5), 14.1562 (5)
α, β, γ (°)	85.454 (3), 71.125 (4), 74.999 (4)
V (Å^3)	1498.32 (11)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.21
Crystal size (mm)	0.46 × 0.42 × 0.26
Data collection	
Diffractometer	Rigaku XtaLAB Mini
Absorption correction	Multi-scan (*REQAB*; Rigaku, 1998)
T_{min}, T_{max}	0.979, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	36497, 10830, 5318
R_{int}	0.042
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.769
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.064, 0.212, 1.03
No. of reflections	10830
No. of parameters	376
No. of restraints	355
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.35, −0.34

Computer programs: *CrysAlis PRO* (Rigaku OD, 2018), *SHELXT* (Sheldrick, 2015*b*), *SHELXL* (Sheldrick, 2015*a*), *OLEX2* (Dolomanov *et al.*, 2009).

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full crystallographic data

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Crystal data

$C_{26}H_{22}O_2P^+ \cdot PF_6^- \cdot C_4H_7O$
M_r = 613.47
Triclinic, $P\overline{1}$
a = 10.3724 (4) Å
b = 11.1647 (5) Å
c = 14.1562 (5) Å
α = 85.454 (3)°
β = 71.125 (4)°
γ = 74.999 (4)°
V = 1498.32 (11) Å^3
Z = 2
$F(000)$ = 634
D_x = 1.360 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5598 reflections
θ = 2.4–23.2°
μ = 0.21 mm^{-1}
T = 170 K
Block, colorless
0.46 × 0.42 × 0.26 mm

Data collection

Rigaku XtaLAB Mini diffractometer
Radiation source: Sealed Tube
Graphite Monochromator monochromator
Detector resolution: 13.6612 pixels mm^{-1}
profile data from ω–scans
Absorption correction: multi-scan (*REQAB*; Rigaku, 1998)
T_{min} = 0.979, T_{max} = 1.000
36497 measured reflections
10830 independent reflections
5318 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
θ_{max} = 33.1°, θ_{min} = 1.9°
h = −15→15
k = −16→16
l = −21→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.064
$wR(F^2)$ = 0.212
S = 1.03
10830 reflections
376 parameters
355 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0843P)^2 + 0.3868P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 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)
P1	0.74066 (6)	0.63718 (5)	0.30298 (4)	0.04533 (15)	
O1	0.8721 (4)	0.0549 (2)	0.6234 (2)	0.1160 (9)	
H1	0.918 (4)	0.0158 (11)	0.657 (2)	0.139*	
O2	0.8305 (3)	0.2077 (2)	0.72578 (18)	0.1091 (8)	
C1	0.6458 (2)	0.7187 (2)	0.22186 (16)	0.0484 (5)	
C2	0.6196 (3)	0.8464 (2)	0.21474 (19)	0.0624 (6)	
H2	0.651620	0.890564	0.251697	0.075*	
C3	0.5452 (3)	0.9086 (3)	0.1522 (2)	0.0772 (8)	
H3	0.527528	0.994509	0.147005	0.093*	
C4	0.4976 (3)	0.8430 (3)	0.0978 (2)	0.0775 (8)	
H4	0.447278	0.884980	0.056302	0.093*	
C5	0.5236 (3)	0.7165 (3)	0.1043 (2)	0.0756 (8)	
H5	0.490899	0.672951	0.067442	0.091*	
C6	0.5985 (3)	0.6535 (2)	0.16561 (19)	0.0647 (6)	
H6	0.617192	0.567505	0.169297	0.078*	
C7	0.9235 (2)	0.5847 (2)	0.23621 (17)	0.0518 (5)	
C8	0.9759 (3)	0.6236 (3)	0.1395 (2)	0.0841 (9)	
H8	0.915867	0.675518	0.108535	0.101*	
C9	1.1180 (4)	0.5851 (4)	0.0888 (3)	0.1118 (13)	
H9	1.153053	0.612248	0.023932	0.134*	
C10	1.2067 (3)	0.5088 (4)	0.1319 (3)	0.0961 (10)	
H10	1.302136	0.484259	0.096759	0.115*	
C11	1.1568 (3)	0.4673 (3)	0.2271 (3)	0.0790 (8)	
H11	1.218125	0.413546	0.256036	0.095*	
C12	1.0149 (3)	0.5054 (3)	0.2806 (2)	0.0664 (6)	
H12	0.981129	0.478122	0.345627	0.080*	
C13	0.7139 (2)	0.7373 (2)	0.40360 (16)	0.0487 (5)	
C14	0.5770 (2)	0.7835 (2)	0.46638 (18)	0.0592 (6)	
H14	0.501928	0.765693	0.452887	0.071*	
C15	0.5525 (3)	0.8547 (3)	0.54755 (19)	0.0716 (7)	
H15	0.461258	0.884205	0.589679	0.086*	
C16	0.6626 (4)	0.8826 (3)	0.5666 (2)	0.0885 (9)	
H16	0.645946	0.929729	0.622600	0.106*	
C17	0.7963 (4)	0.8418 (4)	0.5044 (3)	0.0989 (11)	
H17	0.869711	0.864235	0.516797	0.119*	
C18	0.8241 (3)	0.7675 (3)	0.4231 (2)	0.0741 (8)	
H18	0.915927	0.738160	0.381855	0.089*	
C19	0.6635 (3)	0.5082 (2)	0.35274 (18)	0.0555 (5)	
H19A	0.563440	0.541874	0.383806	0.067*	
H19B	0.674226	0.457737	0.296686	0.067*	
C20	0.7174 (2)	0.4229 (2)	0.42731 (17)	0.0509 (5)	
C21	0.6971 (3)	0.4625 (2)	0.52283 (17)	0.0586 (6)	
H21	0.656054	0.545436	0.540365	0.070*	
C22	0.7372 (3)	0.3796 (3)	0.59205 (19)	0.0637 (6)	
H22	0.724507	0.407528	0.655365	0.076*	

C23	0.7959 (2)	0.2560 (2)	0.56810 (19)	0.0597 (6)	
C24	0.8165 (3)	0.2157 (2)	0.4731 (2)	0.0669 (6)	
H24	0.856520	0.132446	0.456076	0.080*	
C25	0.7776 (3)	0.2990 (2)	0.40295 (19)	0.0620 (6)	
H25	0.792159	0.271259	0.339220	0.074*	
C26	0.8337 (3)	0.1693 (3)	0.6463 (3)	0.0837 (9)	
P2	0.40321 (7)	0.72795 (6)	0.84033 (5)	0.05614 (18)	
F1	0.4425 (2)	0.61011 (15)	0.90639 (15)	0.0898 (5)	
F2	0.3132 (2)	0.80537 (17)	0.93905 (12)	0.0885 (5)	
F3	0.26869 (16)	0.68270 (16)	0.84047 (12)	0.0765 (4)	
F4	0.3598 (2)	0.84541 (16)	0.77637 (13)	0.0880 (5)	
F5	0.49227 (19)	0.64899 (17)	0.74183 (13)	0.0888 (5)	
F6	0.53703 (18)	0.77003 (16)	0.84106 (16)	0.0922 (6)	
O3	0.9436 (5)	−0.0783 (4)	0.7706 (3)	0.1069 (17)	0.677 (7)
C27	0.8665 (8)	−0.1658 (6)	0.8066 (5)	0.1210 (16)	0.677 (7)
H27	0.818201	−0.197237	0.773400	0.145*	0.677 (7)
C28	0.8753 (9)	−0.1996 (7)	0.9067 (5)	0.1210 (16)	0.677 (7)
H28A	0.784141	−0.205188	0.951909	0.145*	0.677 (7)
H28B	0.941887	−0.279065	0.905110	0.145*	0.677 (7)
C29	0.9219 (12)	−0.1022 (8)	0.9390 (5)	0.1210 (16)	0.677 (7)
H29A	0.997832	−0.137682	0.966465	0.145*	0.677 (7)
H29B	0.845038	−0.050248	0.989461	0.145*	0.677 (7)
C30	0.9709 (11)	−0.0292 (7)	0.8483 (5)	0.1210 (16)	0.677 (7)
H30A	0.921060	0.057500	0.859217	0.145*	0.677 (7)
H30B	1.070759	−0.035581	0.831996	0.145*	0.677 (7)
O3′	0.8547 (11)	−0.0047 (9)	0.8366 (8)	0.115 (3)	0.323 (7)
C27′	0.8325 (17)	−0.1247 (11)	0.8378 (12)	0.115 (3)	0.323 (7)
H27A	0.859726	−0.154340	0.769843	0.138*	0.323 (7)
H27B	0.733414	−0.121126	0.869012	0.138*	0.323 (7)
C28′	0.9160 (19)	−0.2124 (13)	0.8937 (14)	0.115 (3)	0.323 (7)
H28C	1.006357	−0.256937	0.849930	0.138*	0.323 (7)
H28D	0.865293	−0.270856	0.932682	0.138*	0.323 (7)
C29′	0.931 (2)	−0.1204 (14)	0.9579 (13)	0.115 (3)	0.323 (7)
H29C	1.011503	−0.152612	0.980819	0.138*	0.323 (7)
H29D	0.846886	−0.096822	1.015150	0.138*	0.323 (7)
C30′	0.9503 (16)	−0.0117 (12)	0.8872 (11)	0.115 (3)	0.323 (7)
H30′	1.012053	0.038261	0.879819	0.138*	0.323 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.0450 (3)	0.0476 (3)	0.0460 (3)	−0.0146 (2)	−0.0158 (2)	0.0025 (2)
O1	0.176 (3)	0.0861 (16)	0.116 (2)	−0.0328 (17)	−0.094 (2)	0.0355 (15)
O2	0.140 (2)	0.1107 (19)	0.0745 (14)	−0.0115 (16)	−0.0513 (15)	0.0239 (13)
C1	0.0525 (11)	0.0457 (11)	0.0488 (11)	−0.0131 (9)	−0.0178 (9)	0.0008 (8)
C2	0.0810 (17)	0.0471 (12)	0.0692 (15)	−0.0166 (11)	−0.0371 (13)	0.0023 (11)
C3	0.107 (2)	0.0499 (14)	0.0852 (19)	−0.0116 (14)	−0.0514 (17)	0.0081 (13)
C4	0.096 (2)	0.0684 (16)	0.0803 (18)	−0.0118 (15)	−0.0529 (17)	0.0115 (14)

C5	0.103 (2)	0.0677 (16)	0.0780 (18)	−0.0260 (15)	−0.0560 (17)	0.0082 (13)
C6	0.0907 (18)	0.0499 (13)	0.0684 (15)	−0.0197 (12)	−0.0441 (14)	0.0037 (11)
C7	0.0500 (11)	0.0530 (12)	0.0510 (11)	−0.0125 (9)	−0.0133 (9)	−0.0038 (9)
C8	0.0652 (16)	0.104 (2)	0.0641 (16)	−0.0110 (16)	−0.0066 (13)	0.0148 (15)
C9	0.070 (2)	0.147 (4)	0.083 (2)	−0.015 (2)	0.0101 (17)	0.017 (2)
C10	0.0522 (16)	0.119 (3)	0.097 (2)	−0.0114 (17)	0.0008 (15)	−0.016 (2)
C11	0.0547 (15)	0.0779 (19)	0.101 (2)	−0.0042 (13)	−0.0273 (15)	−0.0097 (16)
C12	0.0552 (13)	0.0718 (16)	0.0704 (16)	−0.0115 (12)	−0.0209 (12)	0.0009 (13)
C13	0.0493 (11)	0.0510 (12)	0.0467 (11)	−0.0141 (9)	−0.0154 (9)	0.0015 (9)
C14	0.0520 (12)	0.0628 (14)	0.0592 (13)	−0.0119 (11)	−0.0141 (10)	−0.0016 (11)
C15	0.0709 (17)	0.0754 (17)	0.0544 (14)	−0.0067 (14)	−0.0084 (12)	−0.0059 (12)
C16	0.098 (2)	0.098 (2)	0.0699 (18)	−0.0111 (18)	−0.0294 (17)	−0.0303 (16)
C17	0.082 (2)	0.124 (3)	0.104 (2)	−0.0247 (19)	−0.0369 (19)	−0.047 (2)
C18	0.0538 (14)	0.094 (2)	0.0807 (18)	−0.0222 (13)	−0.0207 (13)	−0.0268 (15)
C19	0.0613 (13)	0.0573 (13)	0.0598 (13)	−0.0253 (11)	−0.0293 (11)	0.0110 (10)
C20	0.0518 (12)	0.0556 (12)	0.0537 (12)	−0.0248 (10)	−0.0209 (10)	0.0097 (9)
C21	0.0630 (14)	0.0603 (14)	0.0540 (13)	−0.0191 (11)	−0.0186 (11)	0.0046 (10)
C22	0.0675 (15)	0.0759 (16)	0.0548 (13)	−0.0245 (13)	−0.0256 (12)	0.0103 (11)
C23	0.0539 (13)	0.0700 (15)	0.0641 (14)	−0.0253 (11)	−0.0274 (11)	0.0212 (11)
C24	0.0763 (17)	0.0541 (14)	0.0764 (16)	−0.0173 (12)	−0.0341 (14)	0.0121 (12)
C25	0.0744 (16)	0.0598 (14)	0.0597 (14)	−0.0200 (12)	−0.0299 (12)	0.0048 (11)
C26	0.0672 (17)	0.093 (2)	0.093 (2)	−0.0255 (16)	−0.0346 (16)	0.0434 (18)
P2	0.0597 (4)	0.0537 (4)	0.0591 (4)	−0.0149 (3)	−0.0224 (3)	−0.0037 (3)
F1	0.1094 (14)	0.0694 (10)	0.1083 (13)	−0.0222 (9)	−0.0621 (11)	0.0190 (9)
F2	0.1015 (12)	0.0924 (12)	0.0683 (10)	−0.0162 (10)	−0.0227 (9)	−0.0241 (9)
F3	0.0670 (9)	0.0961 (12)	0.0764 (10)	−0.0301 (8)	−0.0268 (8)	−0.0057 (8)
F4	0.1001 (13)	0.0765 (11)	0.0825 (11)	−0.0139 (9)	−0.0327 (9)	0.0193 (9)
F5	0.0819 (11)	0.0928 (12)	0.0821 (11)	−0.0240 (9)	−0.0039 (9)	−0.0300 (9)
F6	0.0745 (10)	0.0766 (11)	0.1397 (16)	−0.0268 (9)	−0.0427 (11)	−0.0143 (10)
O3	0.146 (4)	0.086 (3)	0.093 (3)	−0.018 (3)	−0.056 (3)	0.021 (2)
C27	0.156 (4)	0.117 (3)	0.105 (2)	−0.061 (3)	−0.045 (2)	0.018 (2)
C28	0.156 (4)	0.117 (3)	0.105 (2)	−0.061 (3)	−0.045 (2)	0.018 (2)
C29	0.156 (4)	0.117 (3)	0.105 (2)	−0.061 (3)	−0.045 (2)	0.018 (2)
C30	0.156 (4)	0.117 (3)	0.105 (2)	−0.061 (3)	−0.045 (2)	0.018 (2)
O3′	0.125 (5)	0.098 (4)	0.123 (5)	−0.012 (4)	−0.062 (4)	0.043 (4)
C27′	0.125 (5)	0.098 (4)	0.123 (5)	−0.012 (4)	−0.062 (4)	0.043 (4)
C28′	0.125 (5)	0.098 (4)	0.123 (5)	−0.012 (4)	−0.062 (4)	0.043 (4)
C29′	0.125 (5)	0.098 (4)	0.123 (5)	−0.012 (4)	−0.062 (4)	0.043 (4)
C30′	0.125 (5)	0.098 (4)	0.123 (5)	−0.012 (4)	−0.062 (4)	0.043 (4)

Geometric parameters (Å, º)

P1—C1	1.793 (2)	C19—C20	1.510 (3)
P1—C7	1.785 (2)	C20—C21	1.389 (3)
P1—C13	1.787 (2)	C20—C25	1.382 (3)
P1—C19	1.811 (2)	C21—H21	0.9300
O1—H1	0.803 (18)	C21—C22	1.382 (3)
O1—C26	1.270 (4)	C22—H22	0.9300

O2—C26	1.223 (4)	C22—C23	1.377 (4)
C1—C2	1.382 (3)	C23—C24	1.385 (4)
C1—C6	1.391 (3)	C23—C26	1.493 (4)
C2—H2	0.9300	C24—H24	0.9300
C2—C3	1.389 (3)	C24—C25	1.390 (3)
C3—H3	0.9300	C25—H25	0.9300
C3—C4	1.377 (4)	P2—F1	1.5956 (17)
C4—H4	0.9300	P2—F2	1.5815 (17)
C4—C5	1.369 (4)	P2—F3	1.6016 (16)
C5—H5	0.9300	P2—F4	1.5849 (17)
C5—C6	1.380 (3)	P2—F5	1.5858 (17)
C6—H6	0.9300	P2—F6	1.5796 (16)
C7—C8	1.381 (4)	O3—C27	1.384 (6)
C7—C12	1.394 (3)	O3—C30	1.404 (6)
C8—H8	0.9300	C27—H27	0.9300
C8—C9	1.381 (4)	C27—C28	1.462 (7)
C9—H9	0.9300	C28—H28A	0.9700
C9—C10	1.349 (5)	C28—H28B	0.9700
C10—H10	0.9300	C28—C29	1.456 (8)
C10—C11	1.368 (5)	C29—H29A	0.9700
C11—H11	0.9300	C29—H29B	0.9700
C11—C12	1.388 (4)	C29—C30	1.482 (7)
C12—H12	0.9300	C30—H30A	0.9700
C13—C14	1.396 (3)	C30—H30B	0.9700
C13—C18	1.384 (3)	O3′—C27′	1.416 (12)
C14—H14	0.9300	O3′—C30′	1.382 (13)
C14—C15	1.367 (3)	C27′—H27A	0.9700
C15—H15	0.9300	C27′—H27B	0.9700
C15—C16	1.367 (4)	C27′—C28′	1.488 (13)
C16—H16	0.9300	C28′—H28C	0.9700
C16—C17	1.361 (5)	C28′—H28D	0.9700
C17—H17	0.9300	C28′—C29′	1.490 (14)
C17—C18	1.382 (4)	C29′—H29C	0.9700
C18—H18	0.9300	C29′—H29D	0.9700
C19—H19A	0.9700	C29′—C30′	1.523 (13)
C19—H19B	0.9700	C30′—H30′	0.9300

C1—P1—C19	106.09 (10)	C23—C22—C21	120.6 (2)
C7—P1—C1	110.78 (10)	C23—C22—H22	119.7
C7—P1—C13	110.72 (10)	C22—C23—C24	119.2 (2)
C7—P1—C19	111.29 (11)	C22—C23—C26	118.7 (3)
C13—P1—C1	109.01 (10)	C24—C23—C26	122.1 (3)
C13—P1—C19	108.83 (11)	C23—C24—H24	119.8
C26—O1—H1	111.1	C23—C24—C25	120.3 (2)
C2—C1—P1	120.31 (17)	C25—C24—H24	119.8
C2—C1—C6	119.7 (2)	C20—C25—C24	120.4 (2)
C6—C1—P1	120.03 (17)	C20—C25—H25	119.8
C1—C2—H2	120.1	C24—C25—H25	119.8

C1—C2—C3	119.8 (2)	O1—C26—C23	115.5 (3)
C3—C2—H2	120.1	O2—C26—O1	123.0 (3)
C2—C3—H3	120.0	O2—C26—C23	121.4 (3)
C4—C3—C2	119.9 (2)	F1—P2—F3	88.66 (9)
C4—C3—H3	120.0	F2—P2—F1	89.58 (10)
C3—C4—H4	119.7	F2—P2—F3	90.19 (10)
C5—C4—C3	120.6 (2)	F2—P2—F4	89.45 (10)
C5—C4—H4	119.7	F2—P2—F5	179.23 (10)
C4—C5—H5	120.0	F4—P2—F1	178.46 (11)
C4—C5—C6	120.1 (2)	F4—P2—F3	90.14 (10)
C6—C5—H5	120.0	F4—P2—F5	90.96 (10)
C1—C6—H6	120.0	F5—P2—F1	89.99 (11)
C5—C6—C1	120.0 (2)	F5—P2—F3	89.16 (9)
C5—C6—H6	120.0	F6—P2—F1	90.23 (10)
C8—C7—P1	119.9 (2)	F6—P2—F2	90.03 (10)
C8—C7—C12	119.2 (2)	F6—P2—F3	178.87 (10)
C12—C7—P1	120.91 (18)	F6—P2—F4	90.97 (10)
C7—C8—H8	120.2	F6—P2—F5	90.61 (10)
C7—C8—C9	119.7 (3)	C27—O3—C30	110.7 (5)
C9—C8—H8	120.2	O3—C27—H27	126.4
C8—C9—H9	119.4	O3—C27—C28	107.2 (4)
C10—C9—C8	121.1 (3)	C28—C27—H27	126.4
C10—C9—H9	119.4	C27—C28—H28A	110.4
C9—C10—H10	119.9	C27—C28—H28B	110.4
C9—C10—C11	120.2 (3)	H28A—C28—H28B	108.6
C11—C10—H10	119.9	C29—C28—C27	106.5 (4)
C10—C11—H11	119.9	C29—C28—H28A	110.4
C10—C11—C12	120.2 (3)	C29—C28—H28B	110.4
C12—C11—H11	119.9	C28—C29—H29A	110.6
C7—C12—H12	120.2	C28—C29—H29B	110.6
C11—C12—C7	119.5 (3)	C28—C29—C30	105.7 (4)
C11—C12—H12	120.2	H29A—C29—H29B	108.7
C14—C13—P1	118.54 (17)	C30—C29—H29A	110.6
C18—C13—P1	122.38 (18)	C30—C29—H29B	110.6
C18—C13—C14	119.0 (2)	O3—C30—C29	107.1 (4)
C13—C14—H14	119.8	O3—C30—H30A	110.3
C15—C14—C13	120.5 (2)	O3—C30—H30B	110.3
C15—C14—H14	119.8	C29—C30—H30A	110.3
C14—C15—H15	120.1	C29—C30—H30B	110.3
C16—C15—C14	119.8 (3)	H30A—C30—H30B	108.5
C16—C15—H15	120.1	C30′—O3′—C27′	107.1 (9)
C15—C16—H16	119.7	O3′—C27′—H27A	109.5
C17—C16—C15	120.5 (3)	O3′—C27′—H27B	109.5
C17—C16—H16	119.7	O3′—C27′—C28′	110.7 (10)
C16—C17—H17	119.7	H27A—C27′—H27B	108.1
C16—C17—C18	120.7 (3)	C28′—C27′—H27A	109.5
C18—C17—H17	119.7	C28′—C27′—H27B	109.5
C13—C18—H18	120.3	C27′—C28′—H28C	112.1

C17—C18—C13	119.4 (3)	C27′—C28′—H28D	112.1
C17—C18—H18	120.3	C27′—C28′—C29′	98.4 (10)
P1—C19—H19A	107.5	H28C—C28′—H28D	109.7
P1—C19—H19B	107.5	C29′—C28′—H28C	112.1
H19A—C19—H19B	107.0	C29′—C28′—H28D	112.1
C20—C19—P1	119.25 (15)	C28′—C29′—H29C	111.3
C20—C19—H19A	107.5	C28′—C29′—H29D	111.3
C20—C19—H19B	107.5	C28′—C29′—C30′	102.4 (10)
C21—C20—C19	122.0 (2)	H29C—C29′—H29D	109.2
C25—C20—C19	118.9 (2)	C30′—C29′—H29C	111.3
C25—C20—C21	118.8 (2)	C30′—C29′—H29D	111.3
C20—C21—H21	119.7	O3′—C30′—C29′	103.7 (10)
C22—C21—C20	120.6 (2)	O3′—C30′—H30′	128.1
C22—C21—H21	119.7	C29′—C30′—H30′	128.1
C21—C22—H22	119.7		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O3	0.80	1.91	2.654 (4)	154
O1—H1···O3′	0.80	2.42	2.997 (10)	129
C3—H3···F4^{i}	0.93	2.82	3.467 (3)	128
C3—H3···F6^{i}	0.93	2.54	3.468 (3)	172
C4—H4···F2^{ii}	0.93	2.80	3.497 (3)	133
C6—H6···F1^{iii}	0.93	2.62	3.360 (3)	137
C10—H10···F1^{iv}	0.93	2.57	3.410 (4)	150
C11—H11···F6^{iv}	0.93	2.83	3.479 (3)	128
C15—H15···F4	0.93	2.56	3.219 (3)	129
C17—H17···O1^{v}	0.93	2.71	3.402 (4)	131
C19—H19*B*···F3^{iii}	0.97	2.42	3.380 (3)	172
C19—H19*B*···F5^{iii}	0.97	2.54	3.275 (3)	132
C30—H30*A*···O2	0.97	2.66	3.323 (8)	126
C28′—H28*C*···F3^{vi}	0.97	2.59	3.385 (18)	139

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