Acta Cryst E CRYSTALLOGRAPHIC COMMUNICATIONS

ISSN 2056-9890

Crystal structure of 3-(triphenylphosphoranylidene)-2,5-dihydrofuran-2,5-dione tetrahydrofuran monosolvate

Almaz Zagidullin,[a]* Daut Islamov,[b] Elena Oshchepkova,[a] Peter Lonnecke[c] and Vasili Miluykov[a]

[a]Arbuzov Institute of Organic and Physical Chemistry, FRC Kazan Scientific Center of RAS, Arbuzov Str. 8, 420088 Kazan, Russian Federation, [b]Department of Chemistry, Kazan State University, Kremlevskaya St. 18, 420008, Kazan, Russian Federation, and [c]University of Leipzig, Faculty of Chemistry and Mineralogy, Institute of Inorganic Chemistry, Johannisallee 29, Leipzig, Germany. *Correspondence e-mail: Almaz_zagidullin@mail.ru

Received 8 May 2018
Accepted 20 August 2018

Edited by C. Rizzoli, Universita degli Studi di Parma, Italy

Keywords: crystal structure; tetrahydrofuran solvate; pseudopolymorph; ylid.

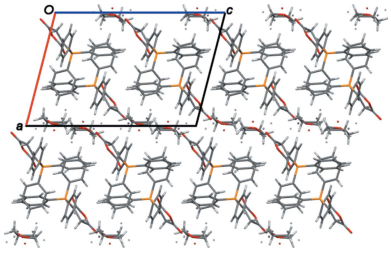
CCDC reference: 1862873

Supporting information: this article has supporting information at journals.iucr.org/e

The title pseudo-polymorph of 3-(triphenylphosphoranylidene)-2,5-dihydrofuran-2,5-dione crystallizes with a tetrahydrofuran solvent molecule, *viz*. $C_{22}H_{17}O_3P{\cdot}C_4H_8O$. The succinic anhydride ring is approximately planar (r.m.s. deviation = 0.032 Å). The tetrahydrofuran molecule is disordered over two orientations about a pseudo-twofold axis with refined occupancy ratio 0.718 (4):0.282 (4). In the crystal, C—H···O hydrogen bonds link molecules of the dihydrofuran-2,5-dione derivative into chains parallel to the *b* axis and arranged into layers stacked along [100] alternating with hydrogen-bonded tetrahydrofuran layers.

1. Chemical context

Pseudopolymorphs are solvated forms of a compound that have different crystal structures and/or differ in the nature of the included solvent (Kumar *et al.*, 1999). The investigation of this phenomenon plays an important role for both fundamental and applied reasons. Phosphorus ylides are useful intermediates, which have been used in many reactions and are involved in the synthesis of organic compounds (Selva *et al.*, 2014; Kolodiazhnyi, 1999; Balema *et al.*, 2002). In this paper, the structure of the pseudopolymorph of 3-(triphenylphosphoranylidene)-2,5-dihydrofuran-2,5-dione (Geoffroy *et al.*, 1993), crystallized with a THF solvent molecule, is described.



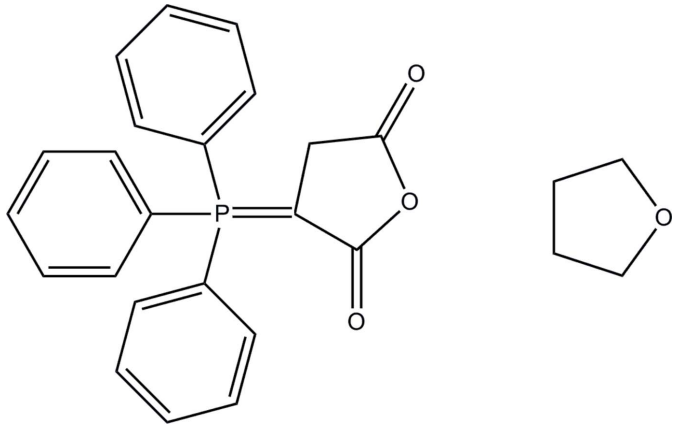


Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C9—H9···O4F^{i}	0.92 (2)	2.59 (2)	3.253 (8)	129.4 (18)
C22—H22···O2^{ii}	0.95 (2)	2.55 (2)	3.386 (2)	148.2 (16)
C20—H20···O4	1.00 (3)	2.58 (2)	3.452 (4)	145.8 (19)
C21—H21···O4F^{iii}	0.94 (2)	2.43 (2)	3.283 (6)	151 (2)

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

2. Structural commentary

In the title compound (Fig. 1), the succinic anhydride ring is almost planar (r.m.s. deviation = 0.032 Å), with the C4 methylenic carbon atom displaced by only 0.118 (2) Å out of the least-squares mean plane through atoms C1, C2, C3 and O1 [maximum deviation of 0.007 (2) Å for C2]. The phosphorus atom deviates from the least-squares mean plane of the succinic anhydride ring by 0.1855 (4) Å. The arrangement of the phenyl rings is propeller-wise, which is common arrangement for Ph_3P-*X* fragments. The THF solvent molecule is disordered over two orientations related by a pseudo-twofold axis. As recently reported by Islamov *et al.* (2017), molecules located in general positions rotate more easily than those located on symmetry elements, and the presence of disorder increases the number of minima on the profile of the rotational barrier, making the barrier even lower (Karlen *et al.*, 2010). However, since the quality of the anisotropic displacement parameters of the THF atoms is low, an attempt to determine the height of the rotational barrier using TLS analysis (Dunitz *et al.*, 1988) was unsuccessful.

3. Supramolecular features

In the crystal, 3-(triphenylphosphoranylidene)-2,5-dihydrofuran-2,5-dione molecules interact through C—H···O hydrogen bonds (Table 1), forming chains running parallel to the *b* axis. Alternating layers of chains and THF molecules are stacked parallel to the *bc* plane (Fig. 2) and connected by C—H···O hydrogen bonds (Table 1).

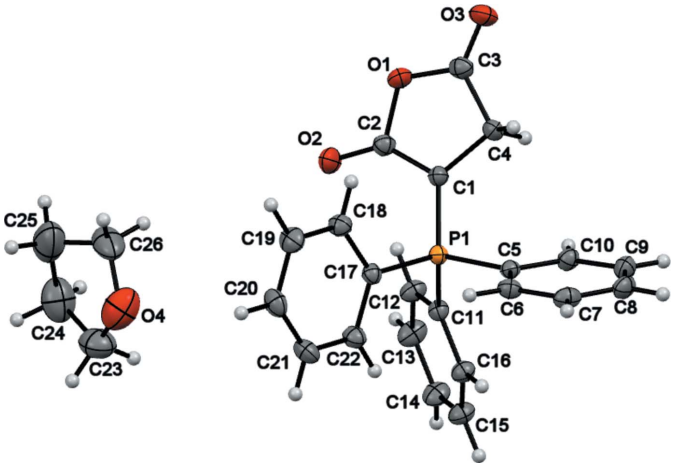


Figure 1
The molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level. Only the major component of the disordered THF molecule is shown.

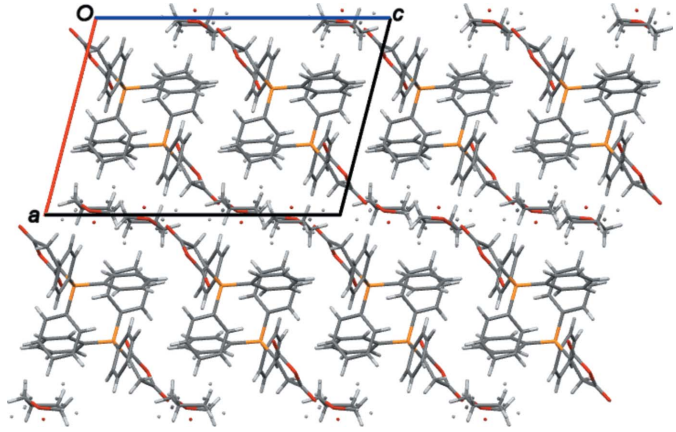


Figure 2
Crystal packing of the title compound viewed along the *b* axis.

4. Database survey

A search of the Cambridge Structural Database (Version 5.39, update February 2018; Groom *et al.*, 2016) revealed 426 structures containing the $Ph_3P{=}C$ fragment. The distribution histogram of the P=C distance [with a mean value of 1.729 Å and a standard deviation of 0.030 Å] is shown in Fig. 3. The P=C distance in the title compound is 1.717 (2) Å, which in good agreement with that of the dichloromethane pseudopolymorph [1.717 (6) Å; Geoffroy *et al.*, 1993]. In spite of the differences in the crystal packing, the conformation of the molecule is very similar to that of the CH_2Cl_2 solvate (r.m.s. deviation = 0.032 Å; Fig. 4).

5. Synthesis and crystallization

To a stirred solution maleic anhydride (0.17 g, 1.72 mmol) in tetrahydrofuran THF (5 mL) was added triphenylphosphine

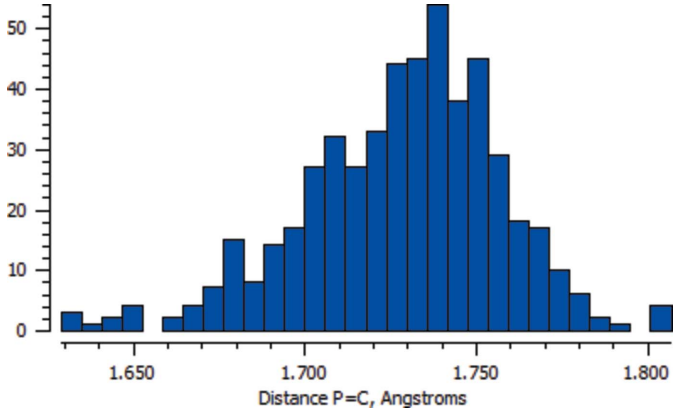


Figure 3
Distribution histogram of the P=C distance in $Ph_3P{=}C$ fragments.

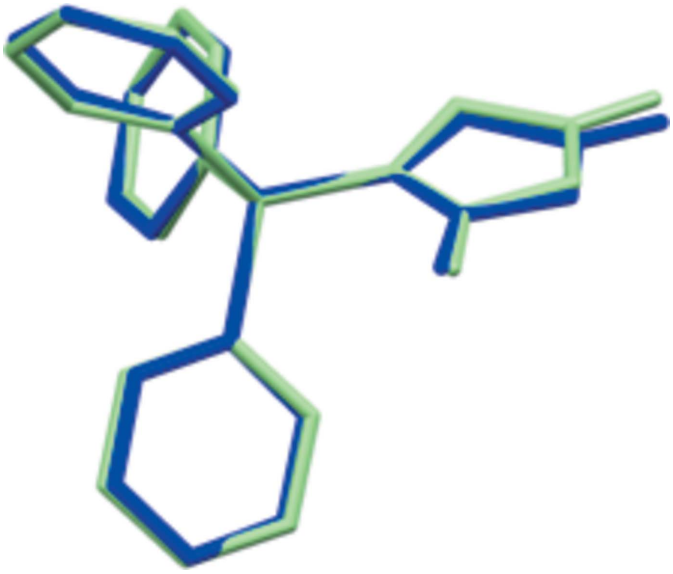


Figure 4
Structure overlay of THF solvate (green) and CH_2Cl_2 solvate (blue; Geoffroy *et al.*, 1993).

(0.45 g, 1.72 mmol) at room temperature. The reaction mixture was stirred at room temperature for 24 h, then the solution was filtered and concentrated under reduced pressure. The reaction mixture was allowed to cool in the freezer (243 K, three days) and yellowish crystals precipitated. The crystals were separated from solvent and dried to give 0.56 g (90%) of the title compound. ^{1}H NMR ($CDCl_3$, δ, ppm, J, Hz): 1.78 (*m*, CH_2 from THF), 3.14 (s, 2H, CH_2), 3.67 (*m*, OCH_2 from THF), 7.44–7.72 (*m*, 15H, Ph). ^{31}P{^{1}H} NMR ($CDCl_3$, δ, ppm, J, Hz): +13.6 (s).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The THF molecule is disordered over two sites with an occupancy ratio of 0.718 (4):0.282 (4). EADP and SAME restraints were used to model this disordered molecule. The H atoms of the 3-(triphenylphosphoranylidene) dihydrofuran-2,5-dione molecule were located in difference-Fourier maps and refined freely. The THF H atoms were placed geometrically and refined using a riding-model approximation with C—H = 0.99 Å, and with U_{iso}(H) = 1.2U_{eq}(C).

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{22}H_{17}O_3P{\cdot}C_4H_8O$
M_r	432.43
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	130
a, b, c (Å)	12.1287 (5), 10.5530 (4), 17.5838 (8)
β (°)	104.435 (4)
V (Å^3)	2179.57 (16)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.16
Crystal size (mm)	0.20 × 0.15 × 0.05
Data collection	
Diffractometer	Agilent Xcalibur Sapphire3 CCD
Absorption correction	Multi-scan (*CrysAlis PRO*; Agilent, 2014)
T_{min}, T_{max}	0.995, 1
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	30663, 7381, 5123
R_{int}	0.061
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.758
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.062, 0.130, 1.03
No. of reflections	7381
No. of parameters	370
No. of restraints	10
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.55, −0.39

Computer programs: *CrysAlis PRO* (Agilent, 2014), *SHELXS97* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *Mercury* (Macrae *et al.*, 2008), *publCIF* (Westrip, 2010).

References

Agilent (2014). *CrysAlis PRO*. Agilent Technologies Ltd, Yarnton, England.

Balema, V. P., Wiench, J. W., Pruski, M. & Pecharsky, V. K. (2002). *J. Am. Chem. Soc.* **124**, 6244–6245.

Dunitz, J., Schomaker, V. & Trueblood, K. N. (1988). *J. Phys. Chem.* **92**, 856–867.

Geoffroy, M., Rao, G., Tancic, Z. & Bernardinelli, G. (1993). *Faraday Trans.* **89**, 2391–2396.

Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* B**72**, 171–179.

Islamov, D. R., Shtyrlin, V. G., Serov, N. Y., Fedyanin, I. V. & Lyssenko, K. A. (2017). *Cryst. Growth Des.* **17**, 4703–4709.

Karlen, S. D., Reyes, H., Taylor, R. E., Khan, S. I., Hawthorne, M. F. & Garcia-Garibay, M. A. (2010). *Proc. Nat. Acad. Sci. USA*, **107**, 14973–14977.

Kolodiazhnyi, O. I. (1999). *Organic Synthesis*, pp. 478–494. Weinheim: Wiley-VCH.

Kumar, V. S. S., Kuduva, S. S. & Desiraju, G. R. (1999). *J. Chem. Soc. Perkin Trans. 2*, pp. 1069–1074.

Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.

Selva, M., Perosa, A. & Noè, M. (2014). *Organophosphorus Chemistry*, Vol. 43, pp. 85–116. UK: Royal Society of Chemistry.

Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.

Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.

Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

supporting information

Acta Cryst. (2018). E**74**, 1336-1338 [https://doi.org/10.1107/S2056989018011775]

Crystal structure of 3-(triphenylphosphoranylidene)-2,5-dihydrofuran-2,5-dione tetrahydrofuran monosolvate

Almaz Zagidullin, Daut Islamov, Elena Oshchepkova, Peter Lonnecke and Vasili Miluykov

Computing details

Data collection: *CrysAlis PRO* (Agilent, 2014); cell refinement: *CrysAlis PRO* (Agilent, 2014); data reduction: *CrysAlis PRO* (Agilent, 2014); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015b); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *publCIF* (Westrip, 2010).

3-(Triphenylphosphoranylidene)-2,5-dihydrofuran-2,5-dione tetrahydrofuran monosolvate

Crystal data

$C_{22}H_{17}O_3P·C_4H_8O$
$M_r = 432.43$
Monoclinic, $P2_1/c$
$a = 12.1287$ (5) Å
$b = 10.5530$ (4) Å
$c = 17.5838$ (8) Å
$\beta = 104.435$ (4)°
$V = 2179.57$ (16) Å^3
$Z = 4$
$F(000) = 912$
$D_x = 1.318$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5863 reflections
$\theta = 3.0$–$32.6°$
$\mu = 0.16$ mm^{-1}
$T = 130$ K
Needles, pale yellow
0.20 × 0.15 × 0.05 mm

Data collection

Agilent Xcalibur Sapphire3 CCD diffractometer
Radiation source: sealed x-ray tube
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Agilent, 2014)
$T_{min} = 0.995$, $T_{max} = 1$
30663 measured reflections
7381 independent reflections
5123 reflections with $I > 2\sigma(I)$
$R_{int} = 0.061$
$\theta_{max} = 32.6°$, $\theta_{min} = 3.0°$
$h = -18\rightarrow17$
$k = -15\rightarrow15$
$l = -26\rightarrow26$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.062$
$wR(F^2) = 0.130$
$S = 1.02$
7381 reflections
370 parameters
10 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0367P)^2 + 1.6405P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.55$ e Å^{-3}
$\Delta\rho_{min} = -0.39$ 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	0.35862 (4)	0.49261 (4)	0.15957 (2)	0.01719 (10)	
O1	0.20198 (11)	0.76108 (12)	0.01566 (8)	0.0267 (3)	
O2	0.35896 (12)	0.79120 (13)	0.11583 (8)	0.0325 (3)	
O3	0.05937 (12)	0.67737 (15)	−0.07627 (8)	0.0365 (4)	
C1	0.26851 (14)	0.58651 (16)	0.09179 (10)	0.0196 (3)	
C2	0.28856 (15)	0.71485 (17)	0.08280 (10)	0.0228 (4)	
C3	0.13225 (15)	0.66384 (19)	−0.01654 (11)	0.0252 (4)	
C4	0.16138 (15)	0.54664 (18)	0.03290 (11)	0.0223 (3)	
C5	0.32090 (14)	0.33056 (16)	0.13491 (9)	0.0184 (3)	
C6	0.39680 (15)	0.24576 (18)	0.11459 (10)	0.0223 (4)	
C7	0.36203 (17)	0.12181 (18)	0.09439 (11)	0.0263 (4)	
C8	0.25270 (17)	0.08421 (18)	0.09336 (11)	0.0272 (4)	
C9	0.17691 (17)	0.16811 (18)	0.11337 (11)	0.0259 (4)	
C10	0.21095 (15)	0.29100 (17)	0.13488 (11)	0.0224 (3)	
C11	0.34766 (14)	0.51365 (17)	0.25923 (9)	0.0191 (3)	
C12	0.30953 (17)	0.62921 (19)	0.28046 (11)	0.0259 (4)	
C13	0.30127 (18)	0.6482 (2)	0.35722 (12)	0.0302 (4)	
C14	0.33144 (17)	0.5526 (2)	0.41208 (11)	0.0293 (4)	
C15	0.36981 (17)	0.4372 (2)	0.39104 (11)	0.0278 (4)	
C16	0.37760 (15)	0.41676 (18)	0.31450 (11)	0.0237 (4)	
C17	0.50533 (14)	0.51511 (16)	0.15815 (10)	0.0194 (3)	
C18	0.53141 (16)	0.56868 (18)	0.09223 (11)	0.0241 (4)	
C19	0.64382 (17)	0.5776 (2)	0.08833 (12)	0.0304 (4)	
C20	0.73049 (16)	0.5328 (2)	0.14956 (12)	0.0303 (4)	
C21	0.70505 (16)	0.4806 (2)	0.21537 (11)	0.0283 (4)	
C22	0.59299 (15)	0.47212 (19)	0.22015 (11)	0.0247 (4)	
H4A	0.1714 (16)	0.476 (2)	0.0002 (12)	0.023 (5)*	
H4B	0.0986 (18)	0.530 (2)	0.0566 (12)	0.028 (6)*	
H6	0.4736 (18)	0.273 (2)	0.1138 (12)	0.027 (5)*	
H7	0.4134 (16)	0.064 (2)	0.0801 (12)	0.021 (5)*	
H8	0.2294 (18)	0.001 (2)	0.0810 (12)	0.028 (6)*	
H9	0.1041 (19)	0.142 (2)	0.1131 (13)	0.033 (6)*	
H10	0.1598 (18)	0.349 (2)	0.1476 (13)	0.031 (6)*	
H12	0.2872 (18)	0.696 (2)	0.2434 (13)	0.033 (6)*	
H13	0.273 (2)	0.728 (2)	0.3708 (14)	0.044 (7)*	
H14	0.3238 (19)	0.566 (2)	0.4650 (14)	0.036 (6)*	
H15	0.3900 (19)	0.370 (2)	0.4293 (14)	0.035 (6)*	
H16	0.4058 (18)	0.332 (2)	0.2995 (13)	0.033 (6)*	
H18	0.4709 (18)	0.601 (2)	0.0507 (13)	0.032 (6)*	

H19	0.6601 (19)	0.614 (2)	0.0407 (14)	0.039 (6)*	
H20	0.811 (2)	0.539 (2)	0.1460 (14)	0.039 (6)*	
H21	0.7635 (19)	0.451 (2)	0.2572 (14)	0.037 (6)*	
O4	0.9797 (3)	0.6888 (3)	0.1470 (2)	0.0667 (10)	0.718 (4)
C23	0.9886 (4)	0.7506 (4)	0.2223 (2)	0.0567 (10)	0.718 (4)
H23A	0.9324	0.7145	0.2485	0.068*	0.718 (4)
H23B	1.0658	0.7385	0.2570	0.068*	0.718 (4)
C24	0.9665 (11)	0.8833 (6)	0.2065 (6)	0.0645 (14)	0.718 (4)
H24A	1.0263	0.9358	0.2408	0.077*	0.718 (4)
H24B	0.8917	0.9074	0.2153	0.077*	0.718 (4)
C25	0.9668 (12)	0.9006 (11)	0.1196 (4)	0.076 (2)	0.718 (4)
H25A	0.9144	0.9690	0.0946	0.092*	0.718 (4)
H25B	1.0442	0.9189	0.1136	0.092*	0.718 (4)
C26	0.9267 (7)	0.7765 (9)	0.0877 (3)	0.0600 (12)	0.718 (4)
H26A	0.8428	0.7709	0.0770	0.072*	0.718 (4)
H26B	0.9496	0.7601	0.0383	0.072*	0.718 (4)
O4F	1.0380 (5)	0.9389 (7)	0.1713 (4)	0.057 (2)	0.282 (4)
C23F	0.965 (3)	0.9030 (19)	0.2222 (17)	0.0645 (14)	0.282 (4)
H23C	0.8965	0.9582	0.2132	0.077*	0.282 (4)
H23D	1.0061	0.9077	0.2782	0.077*	0.282 (4)
C24F	0.9329 (11)	0.7719 (12)	0.1986 (6)	0.0567 (10)	0.282 (4)
H24C	0.8575	0.7504	0.2072	0.068*	0.282 (4)
H24D	0.9902	0.7114	0.2281	0.068*	0.282 (4)
C25F	0.930 (2)	0.771 (3)	0.1100 (8)	0.0600 (12)	0.282 (4)
H25C	0.9780	0.7024	0.0974	0.072*	0.282 (4)
H25D	0.8510	0.7605	0.0774	0.072*	0.282 (4)
C26F	0.975 (3)	0.894 (3)	0.0982 (12)	0.076 (2)	0.282 (4)
H26C	1.0239	0.8877	0.0612	0.092*	0.282 (4)
H26D	0.9116	0.9534	0.0759	0.092*	0.282 (4)
H22	0.5776 (17)	0.437 (2)	0.2658 (12)	0.026 (5)*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.01787 (19)	0.0168 (2)	0.01680 (18)	0.00042 (16)	0.00412 (14)	−0.00023 (17)
O1	0.0279 (7)	0.0222 (7)	0.0280 (7)	0.0025 (5)	0.0034 (5)	0.0065 (5)
O2	0.0379 (8)	0.0195 (7)	0.0358 (8)	−0.0044 (6)	0.0013 (6)	−0.0017 (6)
O3	0.0282 (7)	0.0449 (9)	0.0310 (7)	0.0028 (6)	−0.0029 (6)	0.0112 (7)
C1	0.0203 (8)	0.0182 (8)	0.0191 (8)	0.0000 (6)	0.0027 (6)	0.0005 (6)
C2	0.0236 (8)	0.0212 (9)	0.0231 (8)	0.0020 (7)	0.0048 (7)	0.0006 (7)
C3	0.0203 (8)	0.0301 (10)	0.0254 (9)	0.0035 (7)	0.0061 (7)	0.0043 (8)
C4	0.0182 (8)	0.0238 (9)	0.0236 (8)	−0.0002 (7)	0.0024 (6)	0.0026 (7)
C5	0.0208 (8)	0.0170 (8)	0.0165 (7)	0.0008 (6)	0.0032 (6)	0.0013 (6)
C6	0.0224 (8)	0.0235 (9)	0.0204 (8)	0.0040 (7)	0.0041 (7)	0.0007 (7)
C7	0.0340 (10)	0.0213 (9)	0.0228 (9)	0.0082 (8)	0.0056 (7)	−0.0008 (7)
C8	0.0369 (10)	0.0159 (8)	0.0269 (9)	−0.0010 (8)	0.0043 (8)	0.0005 (7)
C9	0.0270 (9)	0.0205 (9)	0.0300 (9)	−0.0020 (7)	0.0066 (7)	0.0012 (7)
C10	0.0237 (8)	0.0183 (8)	0.0257 (9)	0.0017 (7)	0.0069 (7)	0.0010 (7)

C11	0.0190 (7)	0.0204 (8)	0.0186 (7)	−0.0006 (6)	0.0060 (6)	−0.0012 (6)
C12	0.0340 (10)	0.0216 (9)	0.0236 (9)	0.0040 (8)	0.0097 (7)	0.0020 (7)
C13	0.0427 (11)	0.0242 (10)	0.0281 (9)	0.0053 (8)	0.0170 (8)	−0.0012 (8)
C14	0.0375 (11)	0.0318 (10)	0.0218 (9)	0.0002 (8)	0.0135 (8)	0.0002 (8)
C15	0.0346 (10)	0.0272 (10)	0.0228 (9)	0.0023 (8)	0.0095 (8)	0.0037 (8)
C16	0.0269 (9)	0.0234 (9)	0.0221 (8)	0.0013 (7)	0.0082 (7)	0.0012 (7)
C17	0.0195 (7)	0.0201 (8)	0.0185 (7)	−0.0013 (6)	0.0047 (6)	−0.0016 (6)
C18	0.0237 (8)	0.0275 (9)	0.0217 (8)	0.0009 (7)	0.0069 (7)	0.0009 (7)
C19	0.0286 (10)	0.0383 (12)	0.0273 (10)	−0.0018 (8)	0.0125 (8)	0.0016 (9)
C20	0.0208 (9)	0.0387 (12)	0.0322 (10)	−0.0040 (8)	0.0084 (7)	−0.0051 (9)
C21	0.0208 (8)	0.0373 (11)	0.0246 (9)	0.0006 (8)	0.0014 (7)	−0.0034 (8)
C22	0.0238 (8)	0.0293 (10)	0.0206 (8)	−0.0004 (7)	0.0048 (7)	−0.0001 (7)
O4	0.079 (2)	0.0495 (17)	0.083 (2)	0.0060 (15)	0.0406 (17)	−0.0007 (16)
C23	0.059 (3)	0.069 (3)	0.039 (2)	0.002 (2)	0.0074 (19)	0.0047 (19)
C24	0.061 (2)	0.058 (3)	0.079 (5)	−0.004 (3)	0.027 (3)	−0.022 (3)
C25	0.050 (3)	0.061 (3)	0.113 (6)	−0.007 (2)	0.012 (5)	0.018 (4)
C26	0.0586 (19)	0.092 (3)	0.029 (3)	−0.0256 (19)	0.011 (3)	−0.001 (3)
O4F	0.051 (4)	0.076 (5)	0.037 (3)	−0.041 (3)	−0.004 (3)	0.012 (3)
C23F	0.061 (2)	0.058 (3)	0.079 (5)	−0.004 (3)	0.027 (3)	−0.022 (3)
C24F	0.059 (3)	0.069 (3)	0.039 (2)	0.002 (2)	0.0074 (19)	0.0047 (19)
C25F	0.0586 (19)	0.092 (3)	0.029 (3)	−0.0256 (19)	0.011 (3)	−0.001 (3)
C26F	0.050 (3)	0.061 (3)	0.113 (6)	−0.007 (2)	0.012 (5)	0.018 (4)

Geometric parameters (Å, º)

P1—C1	1.7168 (17)	C17—C22	1.395 (2)
P1—C5	1.7952 (18)	C18—C19	1.385 (3)
P1—C17	1.8016 (17)	C18—H18	0.96 (2)
P1—C11	1.8039 (17)	C19—C20	1.387 (3)
O1—C3	1.360 (2)	C19—H19	0.98 (2)
O1—C2	1.455 (2)	C20—C21	1.384 (3)
O2—C2	1.212 (2)	C20—H20	1.00 (2)
O3—C3	1.201 (2)	C21—C22	1.385 (3)
C1—C2	1.392 (2)	C21—H21	0.94 (2)
C1—C4	1.506 (2)	C22—H22	0.95 (2)
C3—C4	1.502 (3)	O4—C26	1.423 (8)
C4—H4A	0.97 (2)	O4—C23	1.455 (5)
C4—H4B	0.97 (2)	C23—C24	1.440 (9)
C5—C6	1.393 (2)	C23—H23A	0.9900
C5—C10	1.397 (2)	C23—H23B	0.9900
C6—C7	1.393 (3)	C24—C25	1.540 (10)
C6—H6	0.98 (2)	C24—H24A	0.9900
C7—C8	1.380 (3)	C24—H24B	0.9900
C7—H7	0.95 (2)	C25—C26	1.460 (7)
C8—C9	1.383 (3)	C25—H25A	0.9900
C8—H8	0.94 (2)	C25—H25B	0.9900
C9—C10	1.385 (3)	C26—H26A	0.9900
C9—H9	0.93 (2)	C26—H26B	0.9900

C10—H10	0.94 (2)	O4F—C26F	1.404 (19)
C11—C12	1.388 (3)	O4F—C23F	1.461 (17)
C11—C16	1.395 (2)	C23F—C24F	1.468 (15)
C12—C13	1.393 (3)	C23F—H23C	0.9900
C12—H12	0.95 (2)	C23F—H23D	0.9900
C13—C14	1.381 (3)	C24F—C25F	1.549 (14)
C13—H13	0.96 (3)	C24F—H24C	0.9900
C14—C15	1.386 (3)	C24F—H24D	0.9900
C14—H14	0.97 (2)	C25F—C26F	1.445 (15)
C15—C16	1.389 (3)	C25F—H25C	0.9900
C15—H15	0.97 (2)	C25F—H25D	0.9900
C16—H16	1.02 (2)	C26F—H26C	0.9900
C17—C18	1.395 (2)	C26F—H26D	0.9900

C1—P1—C5	107.60 (8)	C17—C18—H18	119.1 (13)
C1—P1—C17	111.98 (8)	C18—C19—C20	120.22 (18)
C5—P1—C17	108.38 (8)	C18—C19—H19	118.5 (13)
C1—P1—C11	114.52 (8)	C20—C19—H19	121.3 (13)
C5—P1—C11	106.01 (8)	C21—C20—C19	120.07 (18)
C17—P1—C11	108.03 (8)	C21—C20—H20	120.2 (14)
C3—O1—C2	109.44 (14)	C19—C20—H20	119.7 (14)
C2—C1—C4	109.82 (15)	C20—C21—C22	120.18 (18)
C2—C1—P1	122.78 (13)	C20—C21—H21	120.4 (14)
C4—C1—P1	127.38 (13)	C22—C21—H21	119.4 (14)
O2—C2—C1	135.62 (17)	C21—C22—C17	119.98 (17)
O2—C2—O1	116.47 (16)	C21—C22—H22	118.8 (12)
C1—C2—O1	107.90 (15)	C17—C22—H22	121.3 (12)
O3—C3—O1	121.34 (18)	C26—O4—C23	107.1 (4)
O3—C3—C4	128.29 (19)	C24—C23—O4	107.0 (5)
O1—C3—C4	110.37 (15)	C24—C23—H23A	110.3
C3—C4—C1	101.89 (15)	O4—C23—H23A	110.3
C3—C4—H4A	109.7 (12)	C24—C23—H23B	110.3
C1—C4—H4A	114.2 (12)	O4—C23—H23B	110.3
C3—C4—H4B	107.5 (13)	H23A—C23—H23B	108.6
C1—C4—H4B	113.0 (12)	C23—C24—C25	105.1 (6)
H4A—C4—H4B	110.0 (17)	C23—C24—H24A	110.7
C6—C5—C10	119.98 (16)	C25—C24—H24A	110.7
C6—C5—P1	121.94 (13)	C23—C24—H24B	110.7
C10—C5—P1	118.06 (13)	C25—C24—H24B	110.7
C5—C6—C7	119.43 (17)	H24A—C24—H24B	108.8
C5—C6—H6	120.6 (13)	C26—C25—C24	101.1 (6)
C7—C6—H6	120.0 (13)	C26—C25—H25A	111.6
C8—C7—C6	120.15 (18)	C24—C25—H25A	111.6
C8—C7—H7	120.1 (12)	C26—C25—H25B	111.6
C6—C7—H7	119.7 (12)	C24—C25—H25B	111.6
C7—C8—C9	120.65 (18)	H25A—C25—H25B	109.4
C7—C8—H8	120.5 (13)	O4—C26—C25	104.8 (7)
C9—C8—H8	118.8 (13)	O4—C26—H26A	110.8

C8—C9—C10	119.81 (18)	C25—C26—H26A	110.8
C8—C9—H9	120.0 (14)	O4—C26—H26B	110.8
C10—C9—H9	120.2 (14)	C25—C26—H26B	110.8
C9—C10—C5	119.96 (17)	H26A—C26—H26B	108.9
C9—C10—H10	120.4 (13)	C26F—O4F—C23F	101.3 (16)
C5—C10—H10	119.6 (13)	O4F—C23F—C24F	103.2 (12)
C12—C11—C16	120.19 (16)	O4F—C23F—H23C	111.1
C12—C11—P1	118.66 (14)	C24F—C23F—H23C	111.1
C16—C11—P1	121.14 (13)	O4F—C23F—H23D	111.1
C11—C12—C13	119.78 (18)	C24F—C23F—H23D	111.1
C11—C12—H12	121.6 (14)	H23C—C23F—H23D	109.1
C13—C12—H12	118.6 (14)	C23F—C24F—C25F	103.2 (14)
C14—C13—C12	120.07 (18)	C23F—C24F—H24C	111.1
C14—C13—H13	121.2 (15)	C25F—C24F—H24C	111.1
C12—C13—H13	118.7 (15)	C23F—C24F—H24D	111.1
C13—C14—C15	120.23 (18)	C25F—C24F—H24D	111.1
C13—C14—H14	119.8 (14)	H24C—C24F—H24D	109.1
C15—C14—H14	120.0 (14)	C26F—C25F—C24F	102.9 (14)
C14—C15—C16	120.24 (18)	C26F—C25F—H25C	111.2
C14—C15—H15	120.3 (14)	C24F—C25F—H25C	111.2
C16—C15—H15	119.5 (14)	C26F—C25F—H25D	111.2
C15—C16—C11	119.48 (17)	C24F—C25F—H25D	111.2
C15—C16—H16	119.8 (13)	H25C—C25F—H25D	109.1
C11—C16—H16	120.7 (13)	O4F—C26F—C25F	108.1 (17)
C18—C17—C22	119.64 (16)	O4F—C26F—H26C	110.1
C18—C17—P1	119.62 (13)	C25F—C26F—H26C	110.1
C22—C17—P1	120.61 (13)	O4F—C26F—H26D	110.1
C19—C18—C17	119.90 (17)	C25F—C26F—H26D	110.1
C19—C18—H18	121.0 (13)	H26C—C26F—H26D	108.4

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C9—H9···O4*F*^{i}	0.92 (2)	2.59 (2)	3.253 (8)	129.4 (18)
C22—H22···O2^{ii}	0.95 (2)	2.55 (2)	3.386 (2)	148.2 (16)
C20—H20···O4	1.00 (3)	2.58 (2)	3.452 (4)	145.8 (19)
C21—H21···O4*F*^{iii}	0.94 (2)	2.43 (2)	3.283 (6)	151 (2)

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