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The 2:1 adducts of (benzoylmethylene)triphenylphosphorane with fumaric and terephthalic acids

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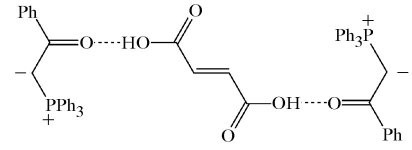
Co-crystals of the ylide (benzoylmethylene)triphenylphosphorane (BPPY) with either fumaric acid, *viz.* (benzoylmethylene)triphenylphosphorane–fumaric acid (2/1), $C_{26}H_{21}OP\cdot 0.5C_4H_4O_4$, or terephthalic acid, *viz.* (benzoylmethylene)triphenylphosphorane–terephthalic acid (2/1), $C_{26}H_{21}OP\cdot 0.5C_8H_6O_4$, have a stoichiometric ratio of 2:1 between the ylide and the corresponding dicarboxylic acid. In both adducts, the acid component lies across a centre of inversion. In neither case is the ylide protonated by the organic acid; instead the H atoms of the non-ionized dicarboxylic acid molecules participate in the formation of strong O—H···O hydrogen bonds with the benzoyl O atom of the ylide species. These structures are the first reported examples of co-crystals containing non-protonated BPPY.

Comment

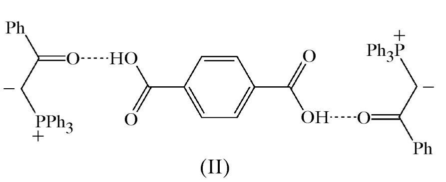
Resonance-stabilized phosphorus ylides are a class of compounds that have attracted considerable interest in the field of synthetic organometallic chemistry. Their popularity arises from their high stability, their reactivity towards a diverse range of metal salts and their ability to be tailored chemically to allow a variety of coordination modes to be accessed (Falvello *et al.*, 1996, 1997, 1998; Kalyanasundari *et al.*, 1995, 2004; Vicente *et al.*, 1988).

The manner of protonation of the resonance-stabilized ylide (benzoylmethylene)triphenylphosphorane (BPPY) has been the focus of our most recent studies. A search of the Cambridge Structural Database (Version 5.25; Allen, 2002) for the BPPY moiety yielded six cases of protonated BPPY (only structures in which BPPY featured as a discrete molecular entity, *i.e.* uncomplexed, were considered). All six structures exhibited C-protonation (Antipin & Struchkov, 1984; Baby Mariyatra *et al.*, 2002*a*,*b*, 2003; Albanese *et al.*, 1989); no examples of O-protonated BPPY were found. These results are surprising, as PM3 calculations of the proton affinities for the ylide C and the benzoyl O atoms give values that differ by only 13 kJ mol^{-1} (Laavanya, 2002), implying that although C-protonation is energetically more favourable both O- and C-protonation of BPPY are feasible.

In our previous work, we have observed that the C-protonated cation of BPPY is produced by the action of picric and maleic acids (Baby Mariyatra *et al.*, 2004*a*,*b*). In order to investigate the influence of organic dicarboxylic acids on the mode of protonation of BPPY, the reactions of this ylide with fumaric and terephthalic acid, yielding compounds (I) and (II), respectively, have been undertaken. The first-step pK_a value in aqueous solution is 3.03 for fumaric acid and 3.51 for terephthalic acid, and the second-step values are 4.44 and 4.82, respectively (Lide, 1994). These figures suggest that both of these acids are sufficiently strong to protonate BPPY (pK_a of 6.0; Speziale & Ratts, 1963).



(I)



(II)

Figs. 1 and 2 display the molecular structures of (I) and (II), respectively. In both cases, the dicarboxylic acid molecule resides on a site of inversion symmetry, and consequently each of the asymmetric units of (I) and (II) comprises a single BPPY molecule and half an acid molecule.

Tables 1 and 3 list selected geometries for (I) and (II), respectively. The inequality of the O2—C27 and O3—C27 bond lengths in (I), and the O2—C30 and O3—C30 bond lengths in (II), is indicative of the dicarboxylic acid molecules in both co-crystals existing in the un-ionized form.

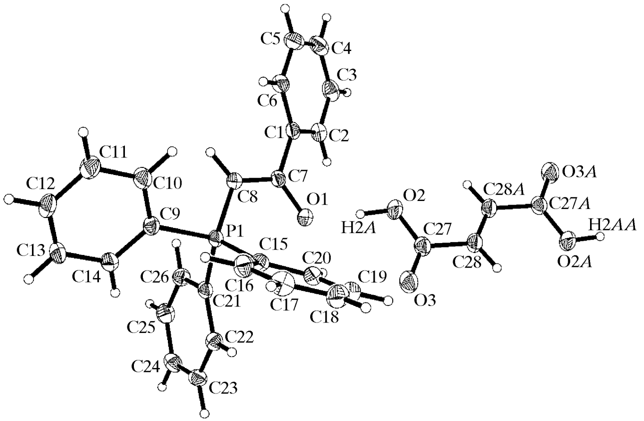


Figure 1
The molecular structure of the co-crystal, (I), of BPPY with fumaric acid. Displacement ellipsoids are drawn at the 50% probability level. The symmetry-generated atoms C28*A*, C27*A*, O2*A*, O3*A* and H2*AA* have been included for completeness [symmetry code: (*A*) $1-x, 1-y, 1-z$].

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The O1—C7 bond lengths are longer than the value of 1.210 Å expected for ketones, and the C7—C8 distances are greater than the expected C=C distance (1.331 Å; Wilson, 1992). These facts are strongly suggestive of resonance delocalization within the ylide molecules. The torsion angles surrounding atom C8 in both structures signify that the environment about this carbanion is distorted trigonal planar. These bond lengths and angles provide conclusive evidence of the presence of unprotonated BPPY in the structures of (I) and (II). Corroborating evidence for the absence of the phosphonium cation has been provided by the ^{1}H NMR spectra of (I) and (II).

In both cases, the P1—C8 and O1—C7 bonds are slightly elongated with respect to the equivalent bonds in the parent ylide, where the P—C bond lengths are 1.716 (5) and 1.725 (4) Å, and the O—C bond lengths are 1.265 (7) and 1.247 (7) Å (two ylide molecules in the asymmetric unit; Kalyanasundari & Panchanatheswaran, 1994). The presence of an exceptionally strong hydrogen bond between the O atoms of the benzoyl groups and an acid H atom of the relevant acid molecule in (I) and (II) (Tables 2 and 4) may account for this disparity.

The non-bonded P1···O1 distances [2.991 (1) Å in (I) and 2.907 (1) Å in (II)] are considerably shorter than the sum of the van der Waals radii of phosphorus and oxygen (3.3 Å; Dunitz, 1979), indicating the presence of strong intramolecular interactions between the charged P$^+$ and O$^-$ centres of the ylide molecules; these interactions explain the observed *cis* orientation about the partial C=C double bond in (I) and (II).

A strong hydrogen bond exists between the O2—H2*A* donor group of the fumaric acid molecule and atom O1 of the ylide molecule (see Table 2). This bond and its symmetry equivalent at ($1 - x$, $1 - y$, $1 - z$) link the fumaric acid and ylide molecules, as shown in Fig. 3.

The secondary interactions for (I) include several C—H···π contacts (Table 2). *Cg*1, *Cg*2 and *Cg*3 are the centroids of the rings defined by atoms C21–C26, C15–C20 and C1–C6. Two π–π interactions complete the complex network of secondary interactions present in the crystal packing of (I) (Fig. 4). Both interactions are 3.99 Å in length, and the β angles are 28 and 23° for the $Cg3\cdots Cg2^{iv}$ and $Cg2\cdots Cg3^{v}$ interactions, respectively [symmetry codes: (iv) $1 - x, -\frac{1}{2} + y, \frac{1}{2} - z$; (v) $1 - x, \frac{1}{2} + y, \frac{1}{2} - z$].

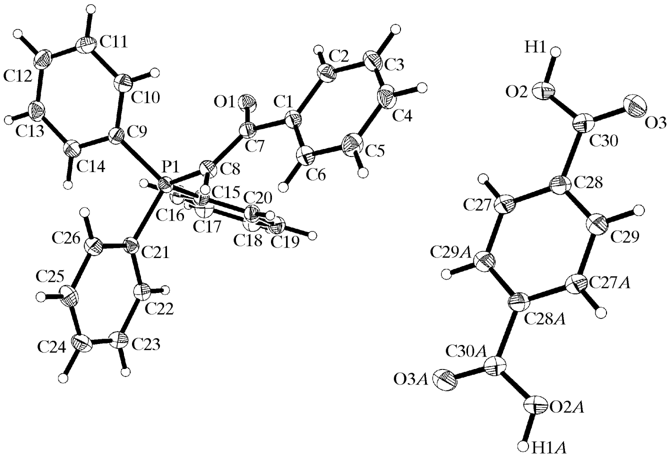

Figure 2
The molecular structure of the co-crystal, (II), of BPPY with terephthalic acid. Displacement ellipsoids are drawn at the 50% probability level. The symmetry-generated atoms C28*A*, C27*A*, C29*A*, C30*A*, O2*A*, O3*A* and H1*A* have been included for completeness [symmetry code: (*A*) $1 - x$, $1 - y$, $1 - z$].

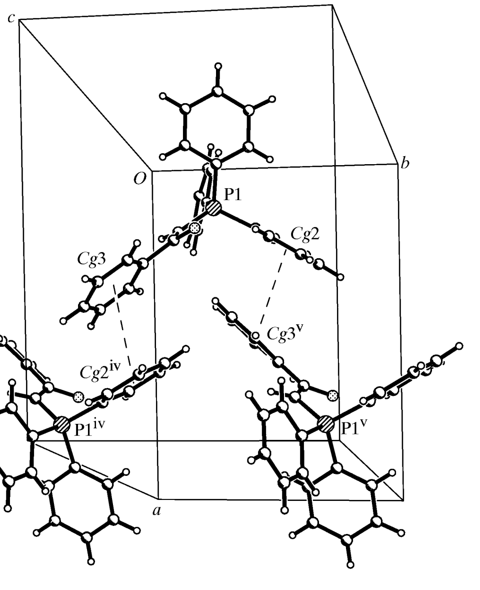

Figure 4
The π–π interactions present in the crystal structure of (I) [symmetry codes: (iv) $1 - x, -\frac{1}{2} + y, \frac{1}{2} - z$; (v) $1 - x, \frac{1}{2} + y, \frac{1}{2} - z$].

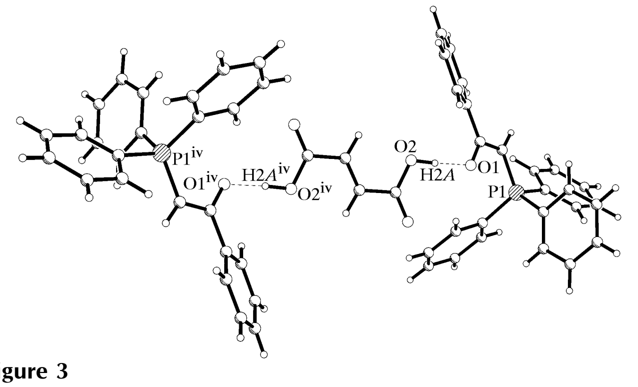

Figure 3
The unit formed by the O2—H2*A*···O1 hydrogen bond in (I) and its symmetry equivalent at ($1 - x$, $1 - y$, $1 - z$) (*viz*. symmetry code iv).

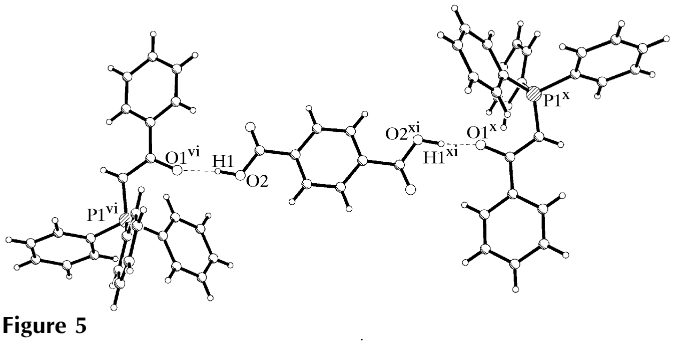

Figure 5
The unit formed by the $O2—H1\cdots O1^{vi}$ hydrogen bond and its symmetry equivalent $O2^{xi}—H1^{xi}\cdots O1^{x}$ [symmetry codes: (vi) $\frac{1}{2} + x, \frac{1}{2} - y, z - \frac{1}{2}$; (x) $\frac{1}{2} - x, -\frac{1}{2} + y, \frac{1}{2} - z$; (xi) $1 - x, -y, -z$].

Table 4 provides details of all the secondary interactions observed in the crystal structure of (II). The strong O2—H1$\cdots$O1^{vi} hydrogen bond and its symmetry equivalent O2^{xi}—H1$^{xi}\cdots$O1^{x} generate a unit comprising a single terephthalic acid molecule and two BPPY molecules (symmetry codes as in Fig. 5). The similarity of this unit to that observed in (I) (Fig. 3) is immediately apparent.

The structure of (II) is further stabilized by several C—H$\cdots\pi$ interactions (Table 4).

In conclusion, the co-crystals (I) and (II) of BPPY with fumaric acid and terephthalic acid, respectively, are the first reported examples in which BPPY remains unprotonated. We attribute this phenomenon to the preferential formation of a strong O—H$\cdots$O hydrogen bond between the benzoyl O atom of the ylide molecule and the acid H atom of the relevant un-ionized dicarboxylic acid group. These short strong hydrogen bonds result in the formation of units with a 2:1 stoichiometric ratio of BPPY to dicarboxylic acid.

Experimental

Crystals of (I) were prepared by stirring BPPY and fumaric acid together in a 1:2 molar ratio in 95% ethanol. Colourless diffraction-quality crystals were obtained on allowing the solution to stand for a week (m.p. 423–425 K). Crystals of (II) were prepared by refluxing BPPY in 95% ethanol with terephthalic acid in a 1:2 molar ratio for 20 h. On cooling the solution to room temperature, colourless crystals of diffraction quality were obtained (m.p. 498–500 K).

Compound (I)

Crystal data

$C_{26}H_{21}OP\cdot0.5C_4H_4O_4$
$M_r = 438.43$
Monoclinic, $P2_1/c$
$a = 13.0883\ (6)$ Å
$b = 9.7883\ (5)$ Å
$c = 17.9970\ (9)$ Å
$\beta = 102.500\ (2)^\circ$
$V = 2250.98\ (19)$ Å^3
$Z = 4$
$D_x = 1.294$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 984 reflections
$\theta = 2.4$–27.5°
$\mu = 0.15$ mm^{-1}
$T = 120\ (2)$ K
Block, colourless
0.39 × 0.27 × 0.22 mm

Data collection

Bruker SMART CCD 6K area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1998)
$T_{min} = 0.756$, $T_{max} = 0.968$
17 181 measured reflections
5164 independent reflections
4281 reflections with $I > 2\sigma(I)$
$R_{int} = 0.033$
$\theta_{max} = 27.5^\circ$
$h = -16 \rightarrow 16$
$k = -11 \rightarrow 12$
$l = -23 \rightarrow 23$

Table 1
Selected geometric parameters (Å, °) for (I).

P1—C8	1.739 (1)	O1—C7	1.280 (2)
P1—C21	1.803 (1)	O2—C27	1.311 (2)
P1—C9	1.805 (1)	O3—C27	1.214 (2)
P1—C15	1.807 (1)	C7—C8	1.386 (2)
C8—P1—C21	114.40 (7)	C7—C8—P1	121.1 (1)
C8—P1—C9	105.92 (6)	C7—C8—H8	124 (1)
C8—P1—C15	114.83 (6)	P1—C8—H8	115 (1)
O1—C7—C8	122.0 (1)		

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.104$
$S = 1.02$
5164 reflections
381 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0512P)^2 + 0.8208P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.41$ e Å^{-3}
$\Delta\rho_{min} = -0.36$ e Å^{-3}

Table 2
Hydrogen-bonding geometry (Å, °) for (I).

*Cg*1, *Cg*2 and *Cg*3 are the centroids of the rings defined by atoms C21–C26, C15–C20 and C1–C6, respectively.

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O2—H2*A*$\cdots$O1	0.99 (3)	1.52 (3)	2.509 (2)	176 (3)
C14—H14$\cdots$*Cg*1	0.92 (2)	3.00	3.72	136
C12—H12$\cdots$*Cg*1^{i}	0.98 (2)	2.61	3.50	152
C13—H13$\cdots$*Cg*2^{ii}	0.98 (2)	2.96	3.69	133
C24—H24$\cdots$*Cg*3^{iii}	0.96 (2)	2.98	3.87	155

Symmetry codes: (i) $x, \frac{1}{2} - y, z - \frac{1}{2}$; (ii) $-x, \frac{3}{2} + y, \frac{1}{2} - z$; (iii) $-x, -y, -z$.

Compound (II)

Crystal data

$C_{26}H_{21}OP\cdot0.5C_8H_6O_4$
$M_r = 463.46$
Monoclinic, $P2_1/n$
$a = 10.0900\ (6)$ Å
$b = 17.9737\ (9)$ Å
$c = 13.1613\ (7)$ Å
$\beta = 92.048\ (2)^\circ$
$V = 2385.3\ (2)$ Å^3
$Z = 4$
$D_x = 1.291$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 948 reflections
$\theta = 2.8$–27.3°
$\mu = 0.15$ mm^{-1}
$T = 120\ (2)$ K
Block, colourless
0.30 × 0.25 × 0.12 mm

Data collection

Bruker SMART CCD 6K area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1998)
$T_{min} = 0.940$, $T_{max} = 0.983$
18 211 measured reflections
5463 independent reflections
4288 reflections with $I > 2\sigma(I)$
$R_{int} = 0.039$
$\theta_{max} = 27.5^\circ$
$h = -13 \rightarrow 10$
$k = -23 \rightarrow 21$
$l = -17 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.111$
$S = 1.03$
5463 reflections
403 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0387P)^2 + 1.0579P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.44$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}

Table 3
Selected geometric parameters (Å, °) for (II).

P1—C8	1.729 (2)	C7—O1	1.277 (2)
P1—C9	1.806 (2)	C7—C8	1.388 (2)
P1—C15	1.810 (2)	C30—O3	1.205 (2)
P1—C21	1.810 (2)	C30—O2	1.319 (2)
C8—P1—C9	114.72 (8)	C7—C8—P1	118.4 (1)
C8—P1—C15	112.28 (7)	C7—C8—H8	124 (1)
C8—P1—C21	106.63 (7)	P1—C8—H8	118 (1)
O1—C7—C8	121.1 (1)		

Table 4
Hydrogen-bonding geometry (Å, °) for (II).

*Cg*1, *Cg*2 and *Cg*3 are the centroids of the rings defined by atoms C9–C14, C21–C26 and C1–C6, respectively.

D—H···A	D—H	H···A	D···A	D—H···A
O2—H1···O1^{vi}	0.92 (3)	1.64 (3)	2.526 (2)	161 (3)
C3—H3···$Cg1^{vi}$	0.96 (2)	2.84	3.59	135
C19—H19···$Cg2^{vii}$	0.99 (2)	3.00	3.79	137
C13—H13···$Cg2^{viii}$	0.93 (2)	2.73	3.59	156
C18—H18···$Cg3^{ix}$	0.94 (2)	2.71	3.53	146

Symmetry codes: (vi) $\frac{1}{2}+x, \frac{1}{2}-y, z-\frac{1}{2}$; (vii) $-x, -y, 1-z$; (viii) $-x, -y, 2-z$; (ix) $x-1, y, z$.

All H atoms were located in difference Fourier maps, and their positional and $U_{\rm iso}$ parameters were refined. The refined C—H distances for (I) are in the range 0.920 (17)–0.995 (19) Å; for (II), the range is 0.92 (2)–0.99 (2) Å. The secondary interaction analysis was performed using a combination of *MERCURY* (Bruno *et al.*, 2002) and *PLATON* (Spek, 2003).

For both compounds, data collection: *SMART-NT* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1998); program(s) used to solve structure: *SHELXTL* (Bruker, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: GD1343). Services for accessing these data are described at the back of the journal.

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supporting information

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The 2:1 adducts of (benzoylmethylene)triphenylphosphorane with fumaric and terephthalic acids

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Computing details

For both compounds, data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1998); program(s) used to solve structure: *SHELXTL* (Bruker, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

(I) (benzoylmethylene)triphenylphosphorane–fumaric acid (2/1)

Crystal data

$C_{26}H_{21}OP·0.5C_4H_4O_4$
M_r = 438.43
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 13.0883 (6) Å
b = 9.7883 (5) Å
c = 17.9970 (9) Å
β = 102.500 (2)°
V = 2250.98 (19) Å^3
Z = 4
$F(000)$ = 920
D_x = 1.294 Mg m^{-3}
Melting point: 424 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 984 reflections
θ = 2.4–27.5°
μ = 0.15 mm^{-1}
T = 120 K
Block, colourless
0.39 × 0.27 × 0.22 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: multi-scan (*SADABS*; Sheldrick, 1998)
T_{min} = 0.756, T_{max} = 0.968
17181 measured reflections
5164 independent reflections
4281 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
θ_{max} = 27.5°, θ_{min} = 1.6°
h = −16→16
k = −11→12
l = −23→23

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.104
S = 1.02
5164 reflections
381 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0512P)^2 + 0.8208P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ < 0.001
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

Special details

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	0.15578 (3)	0.29489 (4)	0.116942 (19)	0.01919 (10)
O1	0.26748 (8)	0.28033 (10)	0.28037 (5)	0.0240 (2)
O2	0.39840 (9)	0.36099 (11)	0.39512 (6)	0.0321 (3)
H2A	0.345 (2)	0.332 (3)	0.3504 (17)	0.079 (8)*
O3	0.33980 (9)	0.57307 (12)	0.36341 (6)	0.0346 (3)
C1	0.36392 (10)	0.07388 (13)	0.27647 (8)	0.0207 (3)
C2	0.37744 (11)	0.04920 (15)	0.35455 (8)	0.0249 (3)
H2	0.3391 (13)	0.0998 (18)	0.3831 (10)	0.027 (4)*
C3	0.44652 (12)	−0.05154 (16)	0.38948 (9)	0.0303 (3)
H3	0.4539 (14)	−0.0720 (19)	0.4445 (11)	0.038 (5)*
C4	0.50331 (12)	−0.12699 (16)	0.34732 (10)	0.0319 (3)
H4	0.5498 (16)	−0.198 (2)	0.3708 (12)	0.041 (5)*
C5	0.49199 (11)	−0.10129 (16)	0.27023 (10)	0.0307 (3)
H5	0.5312 (16)	−0.153 (2)	0.2408 (11)	0.042 (5)*
C6	0.42279 (11)	−0.00159 (15)	0.23484 (9)	0.0247 (3)
H6	0.4166 (13)	0.0192 (18)	0.1821 (11)	0.030 (4)*
C7	0.28756 (10)	0.18112 (14)	0.23947 (8)	0.0198 (3)
C8	0.23982 (10)	0.16899 (14)	0.16312 (8)	0.0219 (3)
H8	0.2484 (13)	0.0898 (18)	0.1320 (10)	0.028 (4)*
C9	0.10647 (11)	0.23261 (14)	0.02154 (7)	0.0215 (3)
C10	0.17593 (12)	0.17661 (17)	−0.01932 (8)	0.0285 (3)
H10	0.2505 (16)	0.167 (2)	0.0042 (11)	0.040 (5)*
C11	0.13933 (12)	0.13232 (17)	−0.09370 (9)	0.0301 (3)
H11	0.1874 (14)	0.0918 (19)	−0.1210 (11)	0.036 (5)*
C12	0.03405 (12)	0.14494 (15)	−0.12742 (8)	0.0275 (3)
H12	0.0073 (14)	0.1153 (19)	−0.1801 (11)	0.036 (5)*
C13	−0.03549 (12)	0.19860 (15)	−0.08729 (9)	0.0285 (3)
H13	−0.1103 (15)	0.2057 (18)	−0.1094 (11)	0.035 (5)*
C14	0.00079 (11)	0.24256 (15)	−0.01250 (8)	0.0247 (3)
H14	−0.0461 (13)	0.2814 (17)	0.0127 (10)	0.024 (4)*
C15	0.21684 (10)	0.45832 (14)	0.10913 (8)	0.0214 (3)
C16	0.22401 (11)	0.51085 (16)	0.03789 (8)	0.0269 (3)
H16	0.1928 (13)	0.4625 (17)	−0.0081 (10)	0.027 (4)*
C17	0.27617 (12)	0.63307 (17)	0.03339 (9)	0.0314 (3)
H17	0.2823 (14)	0.6684 (19)	−0.0157 (11)	0.034 (5)*

C18	0.32162 (12)	0.70341 (16)	0.09927 (10)	0.0316 (3)
H18	0.3594 (16)	0.787 (2)	0.0937 (11)	0.043 (5)*
C19	0.31347 (11)	0.65289 (15)	0.17001 (9)	0.0280 (3)
H19	0.3450 (15)	0.7014 (19)	0.2178 (11)	0.037 (5)*
C20	0.26091 (11)	0.53168 (15)	0.17519 (8)	0.0242 (3)
H20	0.2561 (13)	0.4959 (18)	0.2240 (10)	0.028 (4)*
C21	0.04178 (10)	0.32295 (14)	0.15598 (7)	0.0202 (3)
C22	−0.00575 (11)	0.45062 (15)	0.15439 (8)	0.0222 (3)
H22	0.0272 (13)	0.5299 (18)	0.1397 (10)	0.026 (4)*
C23	−0.10330 (11)	0.46180 (16)	0.17353 (8)	0.0250 (3)
H23	−0.1343 (13)	0.5530 (18)	0.1714 (9)	0.028 (4)*
C24	−0.15317 (11)	0.34601 (16)	0.19273 (8)	0.0269 (3)
H24	−0.2207 (14)	0.3555 (18)	0.2041 (10)	0.030 (4)*
C25	−0.10496 (12)	0.21935 (16)	0.19535 (9)	0.0281 (3)
H25	−0.1388 (15)	0.137 (2)	0.2096 (11)	0.038 (5)*
C26	−0.00729 (11)	0.20790 (15)	0.17754 (8)	0.0241 (3)
H26	0.0256 (13)	0.1199 (19)	0.1801 (10)	0.030 (4)*
C27	0.39633 (11)	0.49353 (15)	0.40497 (8)	0.0257 (3)
C28	0.47073 (11)	0.54169 (16)	0.47523 (8)	0.0255 (3)
H28	0.4751 (13)	0.6381 (18)	0.4814 (10)	0.027 (4)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.01824 (17)	0.02249 (18)	0.01583 (17)	0.00028 (12)	0.00146 (13)	−0.00248 (12)
O1	0.0236 (5)	0.0254 (5)	0.0207 (5)	0.0016 (4)	0.0001 (4)	−0.0053 (4)
O2	0.0333 (6)	0.0298 (6)	0.0273 (5)	0.0021 (4)	−0.0068 (5)	−0.0083 (4)
O3	0.0350 (6)	0.0322 (6)	0.0304 (6)	−0.0001 (5)	−0.0065 (5)	0.0003 (5)
C1	0.0173 (6)	0.0205 (6)	0.0225 (6)	−0.0035 (5)	0.0006 (5)	−0.0007 (5)
C2	0.0227 (6)	0.0274 (7)	0.0232 (7)	−0.0024 (5)	0.0020 (6)	−0.0009 (5)
C3	0.0279 (7)	0.0303 (8)	0.0291 (8)	−0.0051 (6)	−0.0016 (6)	0.0070 (6)
C4	0.0228 (7)	0.0273 (8)	0.0423 (9)	0.0006 (6)	−0.0004 (6)	0.0079 (6)
C5	0.0229 (7)	0.0273 (8)	0.0418 (9)	0.0020 (6)	0.0072 (6)	0.0007 (7)
C6	0.0213 (6)	0.0254 (7)	0.0268 (7)	−0.0014 (5)	0.0042 (6)	−0.0004 (5)
C7	0.0164 (6)	0.0223 (7)	0.0202 (6)	−0.0021 (5)	0.0029 (5)	−0.0013 (5)
C8	0.0204 (6)	0.0236 (7)	0.0202 (6)	0.0015 (5)	0.0014 (5)	−0.0038 (5)
C9	0.0233 (6)	0.0232 (7)	0.0167 (6)	−0.0001 (5)	0.0017 (5)	−0.0009 (5)
C10	0.0232 (7)	0.0397 (8)	0.0222 (7)	0.0001 (6)	0.0042 (6)	−0.0057 (6)
C11	0.0332 (8)	0.0360 (9)	0.0219 (7)	−0.0004 (6)	0.0078 (6)	−0.0055 (6)
C12	0.0381 (8)	0.0240 (7)	0.0178 (7)	−0.0032 (6)	0.0005 (6)	−0.0018 (5)
C13	0.0270 (7)	0.0301 (8)	0.0232 (7)	0.0011 (6)	−0.0057 (6)	−0.0023 (6)
C14	0.0231 (7)	0.0283 (7)	0.0213 (7)	0.0031 (5)	0.0018 (6)	−0.0026 (6)
C15	0.0174 (6)	0.0243 (7)	0.0222 (7)	0.0012 (5)	0.0035 (5)	−0.0009 (5)
C16	0.0272 (7)	0.0309 (8)	0.0221 (7)	−0.0014 (6)	0.0047 (6)	0.0001 (6)
C17	0.0332 (8)	0.0329 (8)	0.0298 (8)	−0.0016 (6)	0.0105 (6)	0.0038 (6)
C18	0.0293 (8)	0.0269 (8)	0.0399 (9)	−0.0047 (6)	0.0102 (7)	−0.0010 (6)
C19	0.0247 (7)	0.0266 (7)	0.0314 (8)	−0.0008 (6)	0.0031 (6)	−0.0035 (6)
C20	0.0220 (6)	0.0258 (7)	0.0238 (7)	0.0011 (5)	0.0029 (5)	−0.0015 (5)

C21	0.0185 (6)	0.0252 (7)	0.0158 (6)	0.0003 (5)	0.0014 (5)	−0.0024 (5)
C22	0.0241 (6)	0.0234 (7)	0.0183 (6)	0.0000 (5)	0.0033 (5)	0.0005 (5)
C23	0.0252 (7)	0.0279 (7)	0.0212 (7)	0.0062 (6)	0.0032 (5)	0.0007 (5)
C24	0.0207 (7)	0.0378 (8)	0.0222 (7)	0.0020 (6)	0.0046 (5)	0.0002 (6)
C25	0.0261 (7)	0.0294 (8)	0.0287 (7)	−0.0040 (6)	0.0060 (6)	0.0018 (6)
C26	0.0253 (7)	0.0227 (7)	0.0241 (7)	0.0004 (5)	0.0046 (6)	0.0007 (5)
C27	0.0238 (7)	0.0303 (8)	0.0219 (7)	−0.0012 (5)	0.0024 (6)	−0.0032 (6)
C28	0.0247 (7)	0.0274 (8)	0.0232 (7)	−0.0030 (5)	0.0024 (6)	−0.0048 (6)

Geometric parameters (Å, º)

P1—C8	1.739 (1)	C12—H12	0.980 (19)
P1—C21	1.803 (1)	C13—C14	1.395 (2)
P1—C9	1.805 (1)	C13—H13	0.976 (19)
P1—C15	1.807 (1)	C14—H14	0.920 (17)
O1—C7	1.280 (2)	C15—C20	1.4018 (19)
O2—C27	1.311 (2)	C15—C16	1.403 (2)
O2—H2A	0.99 (3)	C16—C17	1.388 (2)
O3—C27	1.214 (2)	C16—H16	0.963 (18)
C1—C6	1.397 (2)	C17—C18	1.389 (2)
C1—C2	1.3988 (19)	C17—H17	0.968 (19)
C1—C7	1.5020 (18)	C18—C19	1.391 (2)
C2—C3	1.392 (2)	C18—H18	0.97 (2)
C2—H2	0.934 (18)	C19—C20	1.385 (2)
C3—C4	1.385 (2)	C19—H19	0.991 (19)
C3—H3	0.995 (19)	C20—H20	0.960 (18)
C4—C5	1.386 (2)	C21—C26	1.393 (2)
C4—H4	0.96 (2)	C21—C22	1.3934 (19)
C5—C6	1.389 (2)	C22—C23	1.397 (2)
C5—H5	0.96 (2)	C22—H22	0.952 (18)
C6—H6	0.957 (18)	C23—C24	1.388 (2)
C7—C8	1.386 (2)	C23—H23	0.977 (17)
C8—H8	0.977 (18)	C24—C25	1.387 (2)
C9—C14	1.3897 (19)	C24—H24	0.955 (18)
C9—C10	1.399 (2)	C25—C26	1.388 (2)
C10—C11	1.390 (2)	C25—H25	0.98 (2)
C10—H10	0.98 (2)	C26—H26	0.959 (18)
C11—C12	1.386 (2)	C27—C28	1.4953 (19)
C11—H11	0.963 (19)	C28—C28^{i}	1.324 (3)
C12—C13	1.382 (2)	C28—H28	0.951 (18)
C8—P1—C21	114.40 (7)	C9—C14—C13	120.14 (14)
C8—P1—C9	105.92 (6)	C9—C14—H14	121.2 (10)
C21—P1—C9	105.46 (6)	C13—C14—H14	118.6 (10)
C8—P1—C15	114.83 (6)	C20—C15—C16	119.38 (13)
C21—P1—C15	108.23 (6)	C20—C15—P1	119.68 (11)
C9—P1—C15	107.34 (6)	C16—C15—P1	120.90 (11)
C27—O2—H2A	111.3 (16)	C17—C16—C15	120.00 (14)

C6—C1—C2	118.89 (13)	C17—C16—H16	119.7 (10)
C6—C1—C7	121.55 (12)	C15—C16—H16	120.3 (10)
C2—C1—C7	119.56 (12)	C16—C17—C18	120.17 (15)
C3—C2—C1	120.29 (14)	C16—C17—H17	120.0 (11)
C3—C2—H2	120.0 (10)	C18—C17—H17	119.8 (11)
C1—C2—H2	119.7 (10)	C17—C18—C19	120.11 (15)
C4—C3—C2	120.22 (15)	C17—C18—H18	117.6 (12)
C4—C3—H3	119.4 (11)	C19—C18—H18	122.3 (12)
C2—C3—H3	120.4 (11)	C20—C19—C18	120.25 (14)
C3—C4—C5	119.86 (14)	C20—C19—H19	118.1 (11)
C3—C4—H4	120.6 (12)	C18—C19—H19	121.6 (11)
C5—C4—H4	119.5 (12)	C19—C20—C15	120.07 (14)
C4—C5—C6	120.30 (15)	C19—C20—H20	120.4 (10)
C4—C5—H5	120.3 (12)	C15—C20—H20	119.5 (10)
C6—C5—H5	119.4 (12)	C26—C21—C22	120.01 (13)
C5—C6—C1	120.42 (14)	C26—C21—P1	117.13 (10)
C5—C6—H6	120.7 (11)	C22—C21—P1	122.16 (11)
C1—C6—H6	118.8 (11)	C21—C22—C23	119.54 (13)
O1—C7—C8	122.0 (1)	C21—C22—H22	120.6 (10)
O1—C7—C1	118.37 (12)	C23—C22—H22	119.8 (10)
C8—C7—C1	119.58 (12)	C24—C23—C22	120.09 (14)
C7—C8—P1	121.1 (1)	C24—C23—H23	122.8 (10)
C7—C8—H8	124 (1)	C22—C23—H23	117.1 (10)
P1—C8—H8	115 (1)	C25—C24—C23	120.21 (14)
C14—C9—C10	119.59 (13)	C25—C24—H24	121.0 (11)
C14—C9—P1	120.84 (11)	C23—C24—H24	118.8 (10)
C10—C9—P1	119.55 (10)	C24—C25—C26	119.94 (14)
C11—C10—C9	120.05 (14)	C24—C25—H25	120.9 (11)
C11—C10—H10	119.4 (12)	C26—C25—H25	119.2 (11)
C9—C10—H10	120.5 (12)	C25—C26—C21	120.16 (13)
C12—C11—C10	119.78 (14)	C25—C26—H26	119.1 (10)
C12—C11—H11	120.7 (11)	C21—C26—H26	120.7 (10)
C10—C11—H11	119.5 (11)	O3—C27—O2	125.40 (13)
C13—C12—C11	120.66 (13)	O3—C27—C28	121.16 (14)
C13—C12—H12	118.7 (11)	O2—C27—C28	113.43 (13)
C11—C12—H12	120.7 (11)	C28^{i}—C28—C27	123.46 (18)
C12—C13—C14	119.76 (14)	C28^{i}—C28—H28	121.4 (10)
C12—C13—H13	122.0 (11)	C27—C28—H28	115.1 (10)
C14—C13—H13	118.3 (11)		

C6—C1—C2—C3	−1.7 (2)	C8—P1—C15—C20	−63.51 (13)
C7—C1—C2—C3	178.84 (12)	C21—P1—C15—C20	65.66 (12)
C1—C2—C3—C4	0.8 (2)	C9—P1—C15—C20	179.04 (11)
C2—C3—C4—C5	0.6 (2)	C8—P1—C15—C16	114.28 (12)
C3—C4—C5—C6	−1.0 (2)	C21—P1—C15—C16	−116.55 (12)
C4—C5—C6—C1	0.0 (2)	C9—P1—C15—C16	−3.18 (13)
C2—C1—C6—C5	1.3 (2)	C20—C15—C16—C17	1.3 (2)
C7—C1—C6—C5	−179.25 (13)	P1—C15—C16—C17	−176.54 (11)

C6—C1—C7—O1	−152.84 (13)	C15—C16—C17—C18	0.2 (2)
C2—C1—C7—O1	26.64 (18)	C16—C17—C18—C19	−1.1 (2)
C6—C1—C7—C8	28.28 (19)	C17—C18—C19—C20	0.6 (2)
C2—C1—C7—C8	−152.25 (13)	C18—C19—C20—C15	0.9 (2)
O1—C7—C8—P1	4.27 (19)	C16—C15—C20—C19	−1.8 (2)
C1—C7—C8—P1	−176.89 (10)	P1—C15—C20—C19	176.03 (11)
C21—P1—C8—C7	−60.94 (13)	C8—P1—C21—C26	−40.91 (13)
C9—P1—C8—C7	−176.64 (11)	C9—P1—C21—C26	75.07 (12)
C15—P1—C8—C7	65.10 (13)	C15—P1—C21—C26	−170.31 (10)
C8—P1—C9—C14	135.22 (12)	C8—P1—C21—C22	148.72 (11)
C21—P1—C9—C14	13.58 (14)	C9—P1—C21—C22	−95.31 (12)
C15—P1—C9—C14	−101.66 (13)	C15—P1—C21—C22	19.31 (13)
C8—P1—C9—C10	−46.33 (14)	C26—C21—C22—C23	−0.9 (2)
C21—P1—C9—C10	−167.97 (12)	P1—C21—C22—C23	169.22 (10)
C15—P1—C9—C10	76.79 (13)	C21—C22—C23—C24	−1.1 (2)
C14—C9—C10—C11	0.5 (2)	C22—C23—C24—C25	2.0 (2)
P1—C9—C10—C11	−177.95 (12)	C23—C24—C25—C26	−1.0 (2)
C9—C10—C11—C12	0.6 (2)	C24—C25—C26—C21	−0.9 (2)
C10—C11—C12—C13	−1.3 (2)	C22—C21—C26—C25	1.9 (2)
C11—C12—C13—C14	1.0 (2)	P1—C21—C26—C25	−168.70 (11)
C10—C9—C14—C13	−0.8 (2)	O3—C27—C28—C28^{i}	175.71 (19)
P1—C9—C14—C13	177.61 (12)	O2—C27—C28—C28^{i}	−3.2 (3)
C12—C13—C14—C9	0.1 (2)		

Symmetry code: (i) −*x*+1, −*y*+1, −*z*+1.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*A*···O1	0.99 (3)	1.52 (3)	2.509 (2)	176 (3)
C14—H14···*Cg*1	0.92 (2)	3.00	3.72	136
C12—H12···*Cg*1^{ii}	0.98 (2)	2.61	3.50	152
C13—H13···*Cg*2^{iii}	0.98 (2)	2.96	3.69	133
C24—H24···*Cg*3^{iv}	0.96 (2)	2.98	3.87	155

Symmetry codes: (ii) *x*, −*y*+1/2, *z*−1/2; (iii) −*x*, *y*+3/2, −*z*+1/2; (iv) −*x*, −*y*, −*z*.

(II) (benzoylmethylene)triphenylphosphorane–terephthalic acid (2/1)

Crystal data

$C_{26}H_{21}OP\cdot0.5C_8H_6O_4$	$F(000) = 972$
$M_r = 463.46$	$D_x = 1.291$ Mg m^{-3}
Monoclinic, $P2_1/n$	Melting point: 499 K
Hall symbol: -P 2yn	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 10.0900$ (6) Å	Cell parameters from 948 reflections
$b = 17.9737$ (9) Å	$\theta = 2.8$–$27.3°$
$c = 13.1613$ (7) Å	$\mu = 0.15$ mm^{-1}
$\beta = 92.048$ (2)°	$T = 120$ K
$V = 2385.3$ (2) Å^3	Block, colourless
$Z = 4$	0.30 × 0.25 × 0.12 mm

Data collection

Bruker SMART CCD 6K area-detector diffractometer	18211 measured reflections
Radiation source: fine-focus sealed tube	5463 independent reflections
Graphite monochromator	4288 reflections with $I > 2\sigma(I)$
Detector resolution: 8 pixels mm^{-1}	$R_{\text{int}} = 0.039$
ω scans	$\theta_{\max} = 27.5°$, $\theta_{\min} = 1.9°$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1998)	$h = -13 \rightarrow 10$
$T_{\min} = 0.940$, $T_{\max} = 0.983$	$k = -23 \rightarrow 21$
	$l = -17 \rightarrow 16$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.041$	All H-atom parameters refined
$wR(F^2) = 0.111$	$w = 1/[\sigma^2(F_o^2) + (0.0387P)^2 + 1.0579P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{\max} = 0.001$
5463 reflections	$\Delta\rho_{\max} = 0.44$ e Å^{-3}
403 parameters	$\Delta\rho_{\min} = -0.27$ e Å^{-3}
0 restraints	
Primary atom site location: structure-invariant direct methods	

Special details

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
P1	0.09257 (4)	0.05436 (2)	0.71012 (3)	0.01656 (11)
C1	0.33402 (15)	0.13224 (9)	0.49083 (12)	0.0189 (3)
C2	0.37333 (16)	0.20261 (9)	0.45790 (13)	0.0220 (3)
H2	0.3422 (17)	0.2460 (10)	0.4919 (13)	0.018 (4)*
C3	0.45588 (17)	0.20968 (10)	0.37613 (13)	0.0262 (4)
H3	0.4796 (19)	0.2588 (11)	0.3544 (15)	0.029 (5)*
C4	0.50052 (17)	0.14705 (11)	0.32640 (14)	0.0284 (4)
H4	0.559 (2)	0.1529 (11)	0.2686 (15)	0.029 (5)*
C5	0.46312 (18)	0.07680 (11)	0.35881 (13)	0.0276 (4)
H5	0.493 (2)	0.0322 (12)	0.3221 (16)	0.033 (5)*
C6	0.38014 (16)	0.06933 (9)	0.44016 (12)	0.0219 (3)
H6	0.3561 (19)	0.0225 (11)	0.4609 (15)	0.027 (5)*
C7	0.24066 (15)	0.12721 (8)	0.57705 (12)	0.0180 (3)
C8	0.21723 (16)	0.05936 (9)	0.62356 (12)	0.0192 (3)
H8	0.260 (2)	0.0158 (11)	0.6075 (15)	0.029 (5)*
C9	0.13250 (16)	0.09662 (8)	0.83188 (12)	0.0185 (3)

C10	0.23301 (16)	0.15013 (9)	0.83917 (13)	0.0228 (3)
H10	0.2821 (19)	0.1634 (10)	0.7795 (15)	0.023 (5)*
C11	0.26251 (18)	0.18462 (10)	0.93167 (14)	0.0257 (4)
H11	0.331 (2)	0.2200 (11)	0.9359 (15)	0.029 (5)*
C12	0.19370 (19)	0.16559 (10)	1.01722 (14)	0.0282 (4)
H12	0.214 (2)	0.1894 (12)	1.0817 (17)	0.040 (6)*
C13	0.09436 (19)	0.11226 (10)	1.01027 (13)	0.0273 (4)
H13	0.0484 (19)	0.0994 (11)	1.0672 (16)	0.027 (5)*
C14	0.06388 (17)	0.07749 (9)	0.91848 (13)	0.0238 (3)
H14	−0.007 (2)	0.0395 (12)	0.9128 (15)	0.032 (5)*
C15	−0.06206 (15)	0.09309 (8)	0.66028 (12)	0.0182 (3)
C16	−0.16209 (17)	0.11541 (9)	0.72397 (13)	0.0233 (3)
H16	−0.1490 (19)	0.1149 (10)	0.7982 (16)	0.027 (5)*
C17	−0.28252 (18)	0.14004 (10)	0.68204 (14)	0.0270 (4)
H17	−0.348 (2)	0.1548 (11)	0.7269 (16)	0.031 (5)*
C18	−0.30315 (17)	0.14351 (9)	0.57738 (14)	0.0257 (4)
H18	−0.3827 (19)	0.1626 (10)	0.5477 (14)	0.024 (5)*
C19	−0.20305 (17)	0.12233 (9)	0.51410 (13)	0.0239 (3)
H19	−0.217 (2)	0.1253 (11)	0.4392 (17)	0.035 (6)*
C20	−0.08320 (17)	0.09651 (9)	0.55514 (12)	0.0211 (3)
H20	−0.014 (2)	0.0816 (11)	0.5110 (15)	0.028 (5)*
C21	0.06361 (15)	−0.04351 (8)	0.73310 (11)	0.0178 (3)
C22	−0.05286 (17)	−0.07811 (9)	0.69852 (12)	0.0215 (3)
H22	−0.1223 (18)	−0.0492 (9)	0.6688 (13)	0.015 (4)*
C23	−0.06747 (18)	−0.15489 (9)	0.70885 (13)	0.0256 (4)
H23	−0.150 (2)	−0.1799 (12)	0.6856 (17)	0.040 (6)*
C24	0.03421 (18)	−0.19636 (9)	0.75375 (13)	0.0254 (4)
H24	0.023 (2)	−0.2502 (12)	0.7627 (15)	0.034 (5)*
C25	0.14995 (17)	−0.16218 (9)	0.78929 (14)	0.0251 (4)
H25	0.218 (2)	−0.1899 (12)	0.8219 (16)	0.036 (6)*
C26	0.16525 (16)	−0.08581 (9)	0.77951 (12)	0.0214 (3)
H26	0.246 (2)	−0.0618 (10)	0.8043 (14)	0.024 (5)*
C27	0.46622 (18)	0.05034 (10)	0.07495 (13)	0.0258 (4)
H27	0.4424 (19)	0.0853 (11)	0.1239 (15)	0.027 (5)*
C28	0.54177 (17)	0.07348 (9)	−0.00534 (13)	0.0236 (3)
C29	0.57538 (19)	0.02301 (10)	−0.08039 (14)	0.0281 (4)
H29	0.628 (2)	0.0397 (11)	−0.1369 (16)	0.032 (5)*
C30	0.58964 (18)	0.15223 (9)	−0.01359 (13)	0.0257 (4)
O1	0.18317 (11)	0.18648 (6)	0.60581 (9)	0.0221 (3)
O2	0.56381 (13)	0.19272 (7)	0.06691 (9)	0.0285 (3)
H1	0.605 (3)	0.2385 (16)	0.066 (2)	0.064 (8)*
O3	0.64555 (17)	0.17524 (7)	−0.08662 (11)	0.0431 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
P1	0.0177 (2)	0.01564 (19)	0.0164 (2)	0.00082 (15)	0.00153 (14)	0.00079 (14)
C1	0.0159 (7)	0.0225 (8)	0.0183 (8)	−0.0006 (6)	−0.0003 (6)	0.0028 (6)

C2	0.0194 (8)	0.0216 (8)	0.0251 (8)	−0.0007 (6)	−0.0009 (6)	0.0041 (7)
C3	0.0223 (8)	0.0299 (9)	0.0265 (9)	−0.0047 (7)	−0.0003 (7)	0.0090 (7)
C4	0.0222 (9)	0.0410 (10)	0.0224 (9)	−0.0040 (7)	0.0054 (7)	0.0042 (7)
C5	0.0258 (9)	0.0337 (9)	0.0238 (9)	0.0001 (7)	0.0062 (7)	−0.0035 (7)
C6	0.0223 (8)	0.0226 (8)	0.0210 (8)	−0.0002 (6)	0.0020 (6)	0.0015 (6)
C7	0.0163 (7)	0.0189 (7)	0.0188 (8)	−0.0007 (6)	−0.0004 (6)	−0.0003 (6)
C8	0.0205 (8)	0.0173 (7)	0.0201 (8)	0.0015 (6)	0.0041 (6)	0.0005 (6)
C9	0.0204 (8)	0.0173 (7)	0.0177 (7)	0.0027 (6)	−0.0007 (6)	−0.0005 (6)
C10	0.0214 (8)	0.0216 (8)	0.0253 (9)	0.0004 (6)	0.0003 (7)	0.0006 (6)
C11	0.0248 (9)	0.0224 (8)	0.0295 (9)	−0.0018 (7)	−0.0044 (7)	−0.0035 (7)
C12	0.0375 (10)	0.0247 (8)	0.0219 (9)	0.0030 (7)	−0.0052 (7)	−0.0044 (7)
C13	0.0378 (10)	0.0258 (9)	0.0185 (8)	0.0012 (7)	0.0057 (7)	0.0007 (7)
C14	0.0280 (9)	0.0213 (8)	0.0223 (8)	−0.0018 (7)	0.0018 (7)	0.0009 (6)
C15	0.0207 (8)	0.0159 (7)	0.0181 (7)	0.0004 (6)	0.0007 (6)	−0.0001 (6)
C16	0.0242 (8)	0.0253 (8)	0.0203 (8)	0.0035 (7)	0.0012 (7)	−0.0002 (6)
C17	0.0228 (9)	0.0297 (9)	0.0288 (9)	0.0070 (7)	0.0044 (7)	−0.0006 (7)
C18	0.0210 (8)	0.0236 (8)	0.0320 (9)	0.0037 (7)	−0.0045 (7)	0.0025 (7)
C19	0.0281 (9)	0.0211 (8)	0.0222 (8)	0.0007 (7)	−0.0039 (7)	0.0017 (6)
C20	0.0239 (8)	0.0207 (8)	0.0189 (8)	0.0008 (6)	0.0021 (6)	0.0006 (6)
C21	0.0208 (8)	0.0163 (7)	0.0166 (7)	0.0003 (6)	0.0036 (6)	0.0002 (6)
C22	0.0223 (8)	0.0218 (8)	0.0205 (8)	−0.0006 (6)	0.0006 (6)	0.0008 (6)
C23	0.0276 (9)	0.0227 (8)	0.0265 (9)	−0.0063 (7)	−0.0006 (7)	−0.0002 (7)
C24	0.0303 (9)	0.0173 (8)	0.0289 (9)	−0.0017 (7)	0.0056 (7)	0.0006 (6)
C25	0.0249 (9)	0.0222 (8)	0.0284 (9)	0.0044 (7)	0.0014 (7)	0.0056 (7)
C26	0.0207 (8)	0.0208 (8)	0.0228 (8)	−0.0004 (6)	0.0015 (6)	0.0011 (6)
C27	0.0317 (9)	0.0222 (8)	0.0235 (8)	−0.0040 (7)	0.0026 (7)	−0.0045 (7)
C28	0.0263 (8)	0.0207 (8)	0.0236 (8)	−0.0036 (6)	−0.0024 (7)	−0.0009 (6)
C29	0.0340 (10)	0.0254 (9)	0.0254 (9)	−0.0063 (7)	0.0062 (7)	−0.0019 (7)
C30	0.0311 (9)	0.0217 (8)	0.0242 (9)	−0.0043 (7)	−0.0009 (7)	−0.0018 (7)
O1	0.0260 (6)	0.0172 (5)	0.0233 (6)	0.0027 (4)	0.0031 (5)	0.0009 (4)
O2	0.0381 (7)	0.0196 (6)	0.0277 (7)	−0.0080 (5)	0.0023 (5)	−0.0036 (5)
O3	0.0690 (10)	0.0290 (7)	0.0321 (8)	−0.0188 (7)	0.0154 (7)	−0.0036 (6)

Geometric parameters (Å, º)

P1—C8	1.729 (2)	C15—C20	1.394 (2)
P1—C9	1.806 (2)	C16—C17	1.389 (2)
P1—C15	1.810 (2)	C16—H16	0.98 (2)
P1—C21	1.810 (2)	C17—C18	1.387 (3)
C1—C2	1.399 (2)	C17—H17	0.94 (2)
C1—C6	1.401 (2)	C18—C19	1.385 (2)
C1—C7	1.503 (2)	C18—H18	0.94 (2)
C2—C3	1.390 (2)	C19—C20	1.387 (2)
C2—H2	0.958 (18)	C19—H19	0.99 (2)
C3—C4	1.385 (3)	C20—H20	0.97 (2)
C3—H3	0.96 (2)	C21—C22	1.392 (2)
C4—C5	1.389 (3)	C21—C26	1.399 (2)
C4—H4	0.99 (2)	C22—C23	1.395 (2)

C5—C6	1.389 (2)	C22—H22	0.946 (18)
C5—H5	0.99 (2)	C23—C24	1.384 (3)
C6—H6	0.92 (2)	C23—H23	0.99 (2)
C7—O1	1.277 (2)	C24—C25	1.386 (3)
C7—C8	1.388 (2)	C24—H24	0.98 (2)
C8—H8	0.92 (2)	C25—C26	1.388 (2)
C9—C14	1.398 (2)	C25—H25	0.94 (2)
C9—C10	1.399 (2)	C26—H26	0.97 (2)
C10—C11	1.389 (2)	C27—C29^{i}	1.386 (2)
C10—H10	0.973 (19)	C27—C28	1.389 (2)
C11—C12	1.387 (3)	C27—H27	0.94 (2)
C11—H11	0.94 (2)	C28—C29	1.392 (2)
C12—C13	1.388 (3)	C28—C30	1.501 (2)
C12—H12	0.97 (2)	C29—C27^{i}	1.386 (2)
C13—C14	1.385 (2)	C29—H29	0.98 (2)
C13—H13	0.93 (2)	C30—O3	1.205 (2)
C14—H14	0.99 (2)	C30—O2	1.319 (2)
C15—C16	1.394 (2)	O2—H1	0.92 (3)

C8—P1—C9	114.72 (8)	C16—C15—P1	121.74 (12)
C8—P1—C15	112.28 (7)	C20—C15—P1	118.43 (12)
C9—P1—C15	108.78 (7)	C17—C16—C15	119.66 (16)
C8—P1—C21	106.63 (7)	C17—C16—H16	118.9 (11)
C9—P1—C21	107.02 (7)	C15—C16—H16	121.4 (12)
C15—P1—C21	106.98 (7)	C18—C17—C16	120.46 (16)
C2—C1—C6	118.66 (15)	C18—C17—H17	121.8 (13)
C2—C1—C7	118.72 (14)	C16—C17—H17	117.7 (13)
C6—C1—C7	122.60 (14)	C19—C18—C17	119.86 (16)
C3—C2—C1	120.46 (16)	C19—C18—H18	118.6 (12)
C3—C2—H2	120.1 (10)	C17—C18—H18	121.5 (12)
C1—C2—H2	119.4 (10)	C18—C19—C20	120.15 (16)
C4—C3—C2	120.35 (16)	C18—C19—H19	120.4 (12)
C4—C3—H3	121.1 (12)	C20—C19—H19	119.5 (12)
C2—C3—H3	118.5 (12)	C19—C20—C15	120.12 (16)
C3—C4—C5	119.80 (16)	C19—C20—H20	120.1 (12)
C3—C4—H4	119.5 (12)	C15—C20—H20	119.7 (12)
C5—C4—H4	120.7 (12)	C22—C21—C26	119.81 (15)
C6—C5—C4	120.15 (17)	C22—C21—P1	121.27 (12)
C6—C5—H5	120.1 (12)	C26—C21—P1	118.73 (12)
C4—C5—H5	119.7 (12)	C21—C22—C23	120.08 (16)
C5—C6—C1	120.58 (16)	C21—C22—H22	119.6 (10)
C5—C6—H6	119.3 (12)	C23—C22—H22	120.4 (10)
C1—C6—H6	120.1 (12)	C24—C23—C22	119.68 (16)
O1—C7—C8	121.1 (1)	C24—C23—H23	119.5 (13)
O1—C7—C1	118.50 (13)	C22—C23—H23	120.8 (13)
C8—C7—C1	120.45 (14)	C23—C24—C25	120.56 (16)
C7—C8—P1	118.4 (1)	C23—C24—H24	119.9 (12)
C7—C8—H8	124 (1)	C25—C24—H24	119.5 (12)

P1—C8—H8	118 (1)	C24—C25—C26	120.15 (16)
C14—C9—C10	119.50 (15)	C24—C25—H25	120.9 (13)
C14—C9—P1	121.26 (12)	C26—C25—H25	119.0 (13)
C10—C9—P1	119.23 (12)	C25—C26—C21	119.71 (15)
C11—C10—C9	119.95 (16)	C25—C26—H26	120.3 (11)
C11—C10—H10	119.9 (11)	C21—C26—H26	120.0 (11)
C9—C10—H10	120.2 (11)	C29^{i}—C27—C28	119.98 (16)
C12—C11—C10	120.25 (16)	C29^{i}—C27—H27	121.1 (12)
C12—C11—H11	120.5 (12)	C28—C27—H27	118.9 (12)
C10—C11—H11	119.3 (12)	C27—C28—C29	119.94 (16)
C11—C12—C13	119.90 (16)	C27—C28—C30	121.63 (15)
C11—C12—H12	120.7 (13)	C29—C28—C30	118.43 (15)
C13—C12—H12	119.4 (13)	C27^{i}—C29—C28	120.08 (16)
C14—C13—C12	120.43 (16)	C27^{i}—C29—H29	120.4 (12)
C14—C13—H13	119.5 (13)	C28—C29—H29	119.5 (12)
C12—C13—H13	120.1 (12)	O3—C30—O2	124.26 (16)
C13—C14—C9	119.96 (16)	O3—C30—C28	122.85 (16)
C13—C14—H14	121.1 (12)	O2—C30—C28	112.89 (15)
C9—C14—H14	119.0 (12)	C30—O2—H1	112.3 (16)
C16—C15—C20	119.73 (15)		

C6—C1—C2—C3	0.4 (2)	C8—P1—C15—C20	23.26 (15)
C7—C1—C2—C3	−177.77 (15)	C9—P1—C15—C20	151.33 (12)
C1—C2—C3—C4	−0.2 (3)	C21—P1—C15—C20	−93.39 (13)
C2—C3—C4—C5	−0.3 (3)	C20—C15—C16—C17	0.8 (2)
C3—C4—C5—C6	0.6 (3)	P1—C15—C16—C17	−175.59 (13)
C4—C5—C6—C1	−0.4 (3)	C15—C16—C17—C18	−0.9 (3)
C2—C1—C6—C5	−0.1 (2)	C16—C17—C18—C19	0.0 (3)
C7—C1—C6—C5	178.00 (15)	C17—C18—C19—C20	1.1 (3)
C2—C1—C7—O1	11.6 (2)	C18—C19—C20—C15	−1.2 (2)
C6—C1—C7—O1	−166.45 (15)	C16—C15—C20—C19	0.3 (2)
C2—C1—C7—C8	−168.82 (15)	P1—C15—C20—C19	176.75 (12)
C6—C1—C7—C8	13.1 (2)	C8—P1—C21—C22	−110.36 (14)
O1—C7—C8—P1	8.3 (2)	C9—P1—C21—C22	126.42 (13)
C1—C7—C8—P1	−171.30 (11)	C15—P1—C21—C22	9.96 (15)
C9—P1—C8—C7	−74.53 (14)	C8—P1—C21—C26	64.58 (14)
C15—P1—C8—C7	50.33 (15)	C9—P1—C21—C26	−58.64 (14)
C21—P1—C8—C7	167.19 (12)	C15—P1—C21—C26	−175.10 (12)
C8—P1—C9—C14	−159.29 (13)	C26—C21—C22—C23	−0.9 (2)
C15—P1—C9—C14	74.03 (15)	P1—C21—C22—C23	174.00 (13)
C21—P1—C9—C14	−41.23 (15)	C21—C22—C23—C24	0.1 (3)
C8—P1—C9—C10	21.47 (16)	C22—C23—C24—C25	0.5 (3)
C15—P1—C9—C10	−105.21 (14)	C23—C24—C25—C26	−0.4 (3)
C21—P1—C9—C10	139.54 (13)	C24—C25—C26—C21	−0.4 (2)
C14—C9—C10—C11	−1.0 (2)	C22—C21—C26—C25	1.0 (2)
P1—C9—C10—C11	178.26 (13)	P1—C21—C26—C25	−174.01 (13)
C9—C10—C11—C12	0.8 (3)	C29^{i}—C27—C28—C29	0.1 (3)
C10—C11—C12—C13	−0.4 (3)	C29^{i}—C27—C28—C30	−179.51 (17)

C11—C12—C13—C14	0.4 (3)	C27—C28—C29—C27^{i}	−0.1 (3)
C12—C13—C14—C9	−0.6 (3)	C30—C28—C29—C27^{i}	179.53 (17)
C10—C9—C14—C13	0.9 (2)	C27—C28—C30—O3	−173.53 (18)
P1—C9—C14—C13	−178.31 (13)	C29—C28—C30—O3	6.8 (3)
C8—P1—C15—C16	−160.36 (13)	C27—C28—C30—O2	6.4 (2)
C9—P1—C15—C16	−32.28 (15)	C29—C28—C30—O2	−173.26 (16)
C21—P1—C15—C16	83.00 (14)		

Symmetry code: (i) −*x*+1, −*y*, −*z*.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H1···O1^{ii}	0.92 (3)	1.64 (3)	2.526 (2)	161 (3)
C3—H3···*Cg*1^{ii}	0.96 (2)	2.84	3.59	135
C19—H19···*Cg*2^{iii}	0.99 (2)	3.00	3.79	137
C13—H13···*Cg*2^{iv}	0.93 (2)	2.73	3.59	156
C18—H18···*Cg*3^{v}	0.94 (2)	2.71	3.53	146

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