

Conformations of diester triphenylphosphonium ylides with an ylidic ester or keto and ester ylidic groups

Fernando Castañeda,^{a*} Paul Silva,^a Clifford A. Bunton,^b María Teresa Garland^c and Ricardo Baggio^d

^aDepartamento de Química Orgánica y Físicoquímica, Facultad de Ciencias Químicas y Farmacéuticas, Universidad de Chile, Casilla 233, Santiago, Chile,

^bDepartment of Chemistry and Biochemistry, University of California, Santa Barbara, CA 93106, USA, ^cDepartamento de Física, Facultad de Ciencias Físicas y

Matemáticas, Universidad de Chile, Casilla 487-3, Santiago de Chile, Chile, and

^dDepartamento de Física, Comisión Nacional de Energía Atómica, Avenida Gral Paz 1499, 1650 Buenos Aires, Argentina

Correspondence e-mail: fcastane@ciq.uchile.cl

Received 12 January 2011

Accepted 8 July 2011

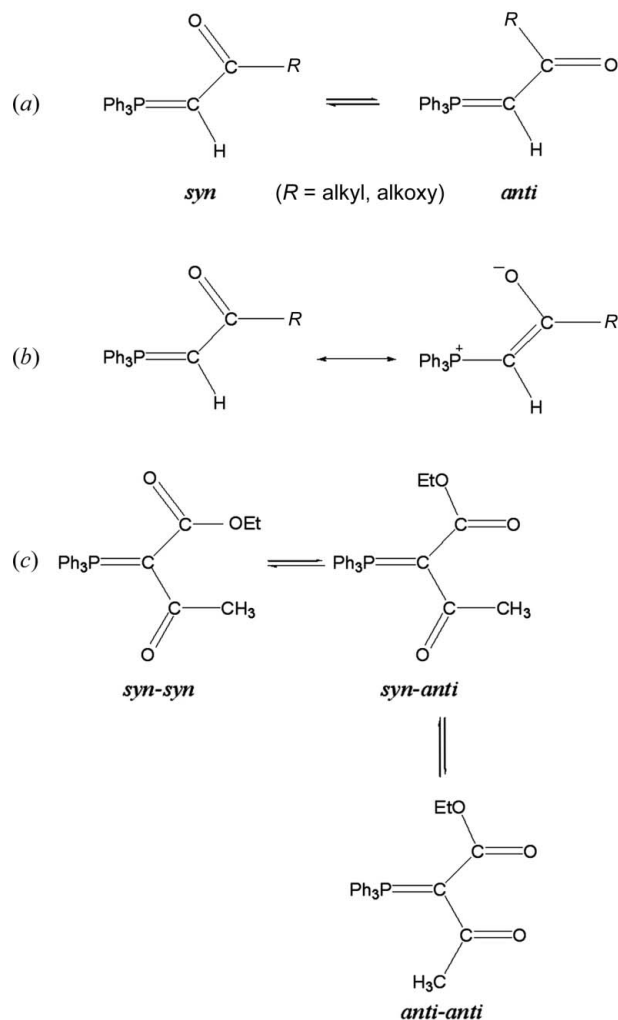
Online 22 July 2011

The structures of three related keto diester and diester ylides, namely diethyl 3-oxo-2-(triphenylphosphoranylidene)glutarate, $C_{27}H_{27}O_5P$, (I), diethyl 3-oxo-2-(triphenylphosphoranylidene)glutarate acetic acid monosolvate, $C_{27}H_{27}O_5P \cdot C_2H_4O_2$, (II), and diethyl 2-(triphenylphosphoranylidene)succinate, $C_{26}H_{27}O_4P$, (III), are presented. The *syn*-keto *anti*-ester conformations in the crystalline keto diesters are governed by electronic delocalization between the P—C and ylidic bonds and an acyl group, and by intra- and intermolecular interactions of different types (C—H \cdots O and C—H $\cdots\pi$) controlling the molecular conformations. The monoylidic diester (III) has an *anti*-ester conformation, while those for (I) and (II) are related to pyrolytic formation of acetylene derivatives. The terminal nonylidic ester group in (I) was disordered over two sets of almost equally populated positions.

Comment

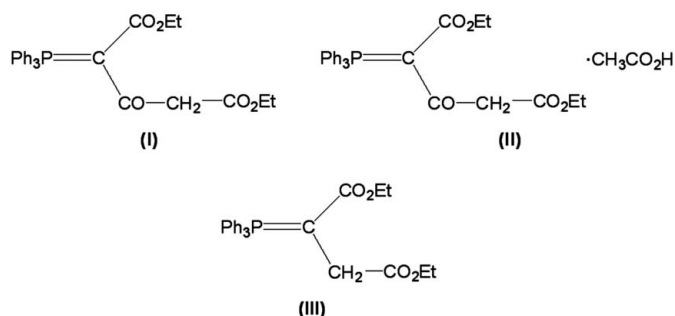
The conformations of triphenylphosphonium ylides with a single keto or ester group conjugated with the ylidic double bond are best established by X-ray crystallography, provided that suitable crystals can be isolated, or else are inferred from NMR or IR spectroscopy (Bachrach & Nitsche, 1994). The ylidic residue is typically close to planar, with electronic delocalization involving the P atom, the ylidic C atom and the associated acyl group. The conformations are designated as *syn* or *anti*, depending on the orientation of the acyl group, *viz.* either towards or away from the P atom (Aitken *et al.*, 2000) [part (a) of Scheme 1]. However, these classical representa-

tions of structures with a C=P double bond are inadequate in that they neglect electronic delocalization. The existence of zwitterionic structures with extensive delocalization [part (b)



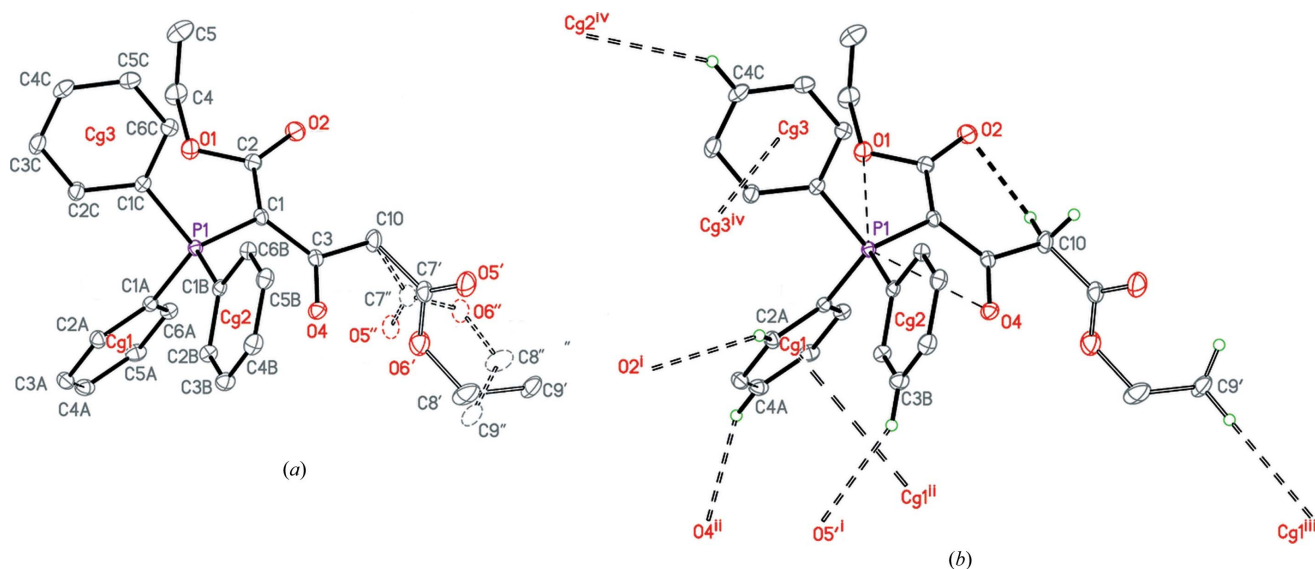
Scheme 1

of Scheme 1] provides due evidence of this fact (Bachrach, 1992). In addition, conformations with two acyl groups could be *syn*-*syn*, *syn*-*anti* or *anti*-*anti* (Aitken *et al.* 2000) [part (c) of Scheme 1].

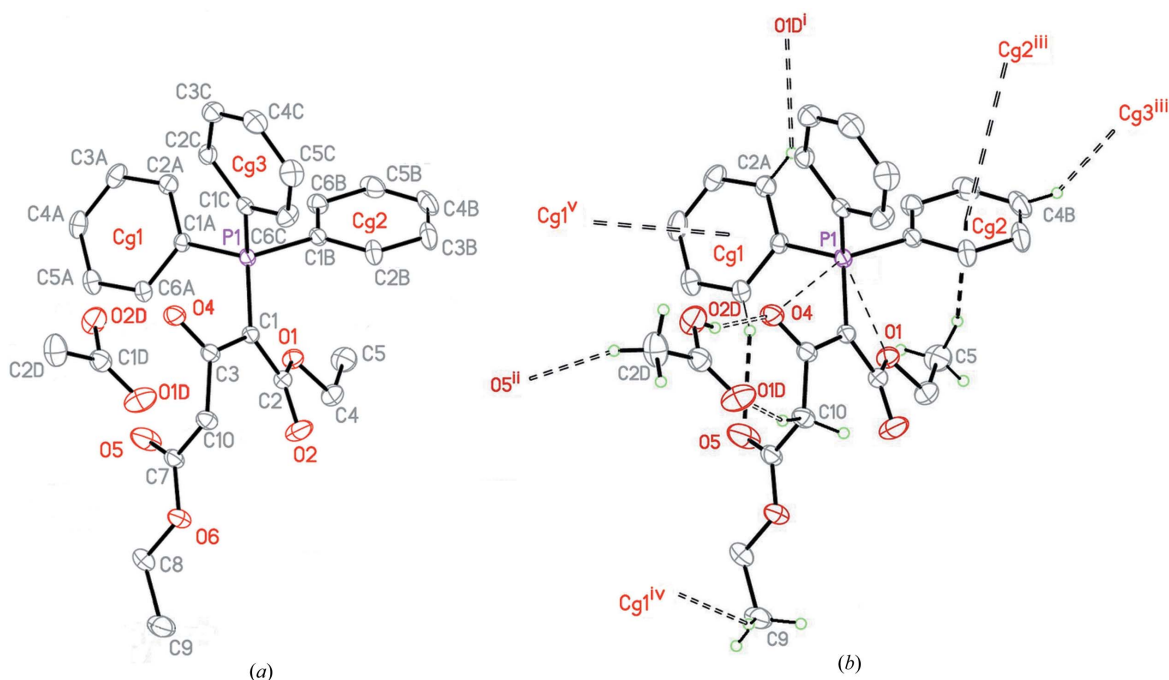


Scheme 2

Only *syn*-*anti* and *anti*-*anti* conformers of aliphatic diacyl derivatives have been observed to date and the latter only with a few diesters (Castañeda *et al.*, 2001, 2005). The situation


Figure 1

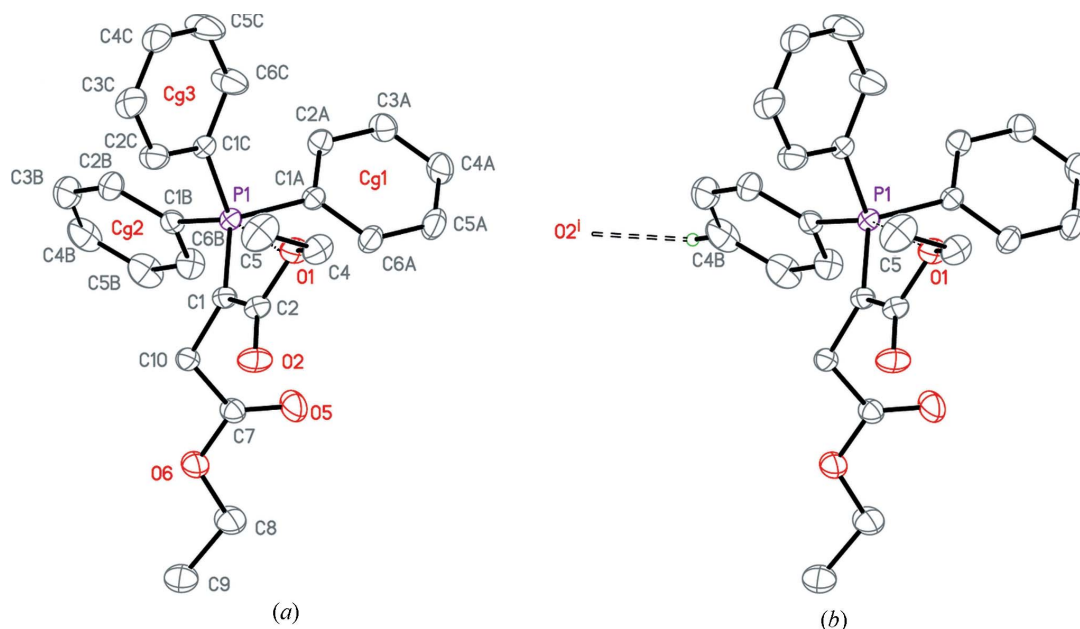
Molecular diagrams for (I), showing (a) the atomic numbering scheme (singly and doubly primed atoms correspond to the disordered part of the molecule) and (b) the nonbonding interactions. Narrow broken lines denote P...O contacts, double broken lines intermolecular interactions and thick broken lines intramolecular bonds. Displacement ellipsoids are drawn at the 40% probability level. [Symmetry codes: (i) $x - 1, y, z$; (ii) $-x + 1, -y + 2, -z + 1$; (iii) $x + 1, y + 1, z$; (iv) $-x, -y + 1, -z$.]


Figure 2

Molecular diagrams for (II), showing (a) the atomic numbering scheme and (b) the nonbonding interactions. Narrow broken lines denote P...O contacts, double broken lines intermolecular interactions and thick broken lines intramolecular bonds. Displacement ellipsoids are drawn at the 40% probability level. [Symmetry codes: (i) $x - 1, y, z$; (ii) $-x + 1, -y + 1, -z + 1$; (iii) $-x, -y + 1, -z$; (iv) $x + 1, y, z$; (v) $-x, 1 - y, 1 - z$.]

is more complicated with different acyl groups, but generally in aliphatic keto esters the ester acyl group is *anti* and the keto acyl group *syn* (Castañeda *et al.*, 2001, 2003). In mixed aliphatic diesters, the smaller ester group is usually *syn* and the large group *anti* with respect to P (Castañeda *et al.*, 2009a,b). These generalizations do not apply when aryl groups are present in the ylidic residue, where electronic delocalization and steric effects have to be considered, nor to some diylides with bulky structures (Aitken *et al.*, 2000).

The present work involves three triphenyl phosphonium ylides: two keto diesters, diethyl 3-oxo-2-(triphenylphosphoranyliden)glutarate, (I), and diethyl 3-oxo-2-(triphenylphosphoranyliden)glutarate acetic acid monosolvate, (II), and one diester, diethyl 2-(triphenylphosphoranyliden)succinate, (III), with one ylidic and one nonylidic ester group [see Scheme 2]. These ylides were prepared in order to examine whether nonylidic external carboxylic ester groups affect the conformations of the ylidic moieties in the crystal structures.

**Figure 3**

Molecular diagrams for (III), showing (a) the atomic numbering scheme and (b) the nonbonding interactions. Narrow broken lines (partially eclipsed by atom C5) denote $P \cdots O$ contacts and double broken lines intermolecular $C-H \cdots O$ contacts. Displacement ellipsoids are drawn at the 40% probability level. [Symmetry code: (i) $-x + \frac{3}{2}, y - \frac{1}{2}, z$.]

Figs. 1(a), 2(a) and 3(a) show the corresponding molecular views and atomic labelling schemes used for (I), (II) and (III), respectively. These three ylides share common features, in particular a slightly distorted tetrahedral arrangement around the P atom, with the phenyl groups in a propeller-like disposition, as observed for stabilized keto ester ylides (Castañeda *et al.*, 2001). The sums of the angles about ylidic atom C1 are very near the ideal value of 360° , consistent with sp^2 hybridization [358.0 (4°) in (I), 358.6 (5°) in (II) and 359.9 (5°) in (III)] in a near trigonal-planar geometry.

The $P1-C1$ bond lengths [1.7196 (18)– 1.758 (4) Å] lie between accepted values for single and double bonds (1.80 – 1.83 Å and 1.66 Å, respectively; Howells *et al.*, 1973) due to electronic delocalization which shortens $C1-C2$ (in all three structures) and $C1-C3$ [in (I) and (II)]. The ylidic keto carbonyl bonds are longer than the typical value of 1.21 Å [$C3-O4 = 1.239$ (2)– 1.254 (2) Å] for keto-ester, diester and diketo ylides (Castañeda *et al.*, 2001, 2005).

In the crystalline stabilized ylides (I) and (II), the keto O atom and the ylidic alkoxy groups are oriented towards P with *syn*-keto and *anti*-ester conformations, respectively (Figs. 1 and 2). A *syn*-keto conformation is also established by pyrolysis of diketo or keto ester-ylides, *i.e.* (I), where *syn*-keto conformations are required to form keto or ester acetylenes, respectively (Gough & Trippett, 1962; Chopard *et al.* 1965).

Coplanarity between the ylidic keto carbonyl and ester carbonyl units in (I) and (II) is indicated by their torsion angles. The $P-C-C-O$ torsion angles are close to -2.5° for the keto group and near to 167° for the ester carbonyl, showing stronger delocalization of the keto group. The keto and ester carbonyl groups in (I) and (II) have opposite orientations, which reduces dipole–dipole repulsions, and the keto O atoms are within 2.88 – 2.91 Å of P, *i.e.* within the sum of

the van der Waals radii (Bondi, 1964), and favourable interactions should stabilize the conformer. The ester groups in (I)–(III) have the typical *Z* conformation (Eliel & Wilen, 1994) and are approximately in the ylidic plane.

The keto diester ylide solvate, (II), was prepared because acetic acid can promote the formation of good single crystals (Abel *et al.* 1989) and new intermolecular interactions could modify the *syn*-keto *anti*-ester conformation of ylide (I). However, (II) is a 1:1 ylide solvate with intermolecular hydrogen-bond interactions and *syn*-keto *anti*-ester conformations, as in (I).

The *anti*-ester conformations in crystalline ylides (I)–(III) (Figs. 1–3) do not depend on the presence of a *syn*-keto group and could be favoured by alkoxy $O \cdots P$ interactions, an attractive $C-H \cdots \pi$ effect (Nishio *et al.*, 1995) or the absence of nonbonded steric repulsion between alkoxy ylidic and nonylidic ester groups. In the crystal structure and in solution, the structures present one alkoxy group *syn* to P directed towards the face of a phenyl group which is approximately orthogonal to the ylidic $C-P$ bond with a modestly stabilizing $C-H \cdots \pi$ interaction (Nishio *et al.*, 1995; Nishio & Hirota, 1989). The crystal structure of mono-ylidic diester (III) shows an *anti*-ester conformation with a contact distance of 2.8177 (15) Å between atoms P1 and O1.

The nonbonding interactions in (I), (II) and (III) are quite different in all three structures. Compound (III) presents only one close intramolecular $P \cdots O$ contact [$P1 \cdots O1 = 2.818$ (2) Å] and one moderate $C-H \cdots O$ intermolecular contact (Table 8), the remaining interactions being mostly van der Waals. By contrast, (I) and (II) display a large number of nonbonding contacts of diverse nature and strength. There are two short intramolecular $P \cdots O$ contacts in each [$P1 \cdots O1 = 2.877$ (2) Å and $P1 \cdots O4 = 2.879$ (2) Å in (I); $P1 \cdots O1 =$

2.888 (2) Å and P1···O4 = 2.915 (2) Å in (II)], many conventional and nonconventional hydrogen bonds and C—H··· π contacts (both intra- and intermolecular) (Tables 2 and 5), and some π – π stacking interactions between aromatic rings (Tables 3 and 6). All these contacts are presented schematically in Figs. 1(b), 2(b) and 3(b), and their inspection confirms a final three-dimensional packing structure for (I) and (II), but a much simpler one-dimensional (chain-like) structure for (III).

Experimental

Diethyl 3-oxo-2-(triphenylphosphoranylidene)glutarate, (I) (m.p. 379 K, yield 68%), was prepared by reaction of ethyl 2-(triphenylphosphoranylidene)acetate, Ph₃P=CH–CO₂Et, with ethyl malonyl chloride in dry benzene at room temperature. Recrystallization of (I) from ethyl acetate–hexane (1:1 v/v) and then from acetic acid gave the solvate (II) (m.p. 433 K, yield 72%). Diethyl 2-(triphenylphosphoranylidene)succinate, (III) [m.p. 370 K, recrystallized from ethyl acetate–hexane (1:1 v/v), yield 71%], was synthesized by transylidation of Ph₃P=CH–CO₂Et with ethyl 2-bromoacetate in dry ethyl acetate at 313 K for 4 h. Elemental analyses using a Fisons EA 1108 analyser agreed with the determined structures of ylides (I)–(III).

Table 1

Selected bond lengths (Å) for (I).

P1–C1	1.7546 (18)	O1–C4	1.452 (2)
P1–C1A	1.8055 (19)	O2–C2	1.220 (2)
P1–C1C	1.8106 (19)	O4–C3	1.240 (2)
P1–C1B	1.8183 (19)	C1–C3	1.432 (3)
O1–C2	1.361 (2)	C1–C2	1.453 (3)

Table 2

Hydrogen-bond geometry (Å, °) for (I).

Cg1 is the centroid of the C1A–C6A ring and Cg2 that of the C1B–C6B ring.

D–H···A	D–H	H···A	D···A	D–H···A
C10–H10A···O2	0.99	2.23	2.853 (3)	120
C2A–H2A···O2 ⁱ	0.95	2.54	3.189 (3)	126
C3B–H3B···O5 ⁱⁱ	0.95	2.48	3.327 (5)	150
C4A–H4A···O4 ⁱⁱⁱ	0.95	2.38	3.245 (3)	151
C9′–H9′A···Cg1 ⁱⁱⁱⁱ	0.98	2.74	3.712 (4)	172
C4C–H4C···Cg2 ^{iv}	0.95	2.79	3.604 (2)	144

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

Table 3

π – π contacts (Å) for (I).

CCD is the centroid–centroid distance and PCD is the (mean) centroid to opposite plane distance; for details, see Janiak (2000). Cg1 is the centroid of the C1A–C6A ring and Cg3 that of the C1C–C6C ring. The rings are parallel by symmetry.

Group 1/group 2	CCD (Å)	PCD (Å)
Cg1···Cg1 ⁱⁱ	3.8512 (12)	3.5410 (8)
Cg3···Cg3 ^{iv}	3.9326 (12)	3.6424 (8)

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

Compound (I)

Crystal data

C ₂₇ H ₂₇ O ₅ P	$\gamma = 107.304 (2)^\circ$
$M_r = 462.46$	$V = 1152.0 (2) \text{ \AA}^3$
Triclinic, $P\bar{1}$	$Z = 2$
$a = 9.0348 (10) \text{ \AA}$	Mo $K\alpha$ radiation
$b = 10.3770 (11) \text{ \AA}$	$\mu = 0.16 \text{ mm}^{-1}$
$c = 13.8951 (15) \text{ \AA}$	$T = 150 \text{ K}$
$\alpha = 95.994 (2)^\circ$	$0.46 \times 0.26 \times 0.19 \text{ mm}$
$\beta = 108.342 (2)^\circ$	

Data collection

Bruker SMART CCD area-detector diffractometer	9669 measured reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 2001)	4935 independent reflections
$T_{\min} = 0.95, T_{\max} = 0.97$	4077 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.021$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.050$	122 restraints
$wR(F^2) = 0.143$	H-atom parameters constrained
$S = 1.09$	$\Delta\rho_{\text{max}} = 1.05 \text{ e \AA}^{-3}$
4935 reflections	$\Delta\rho_{\text{min}} = -0.36 \text{ e \AA}^{-3}$
344 parameters	

Table 4

Selected bond lengths (Å) for (II).

P1–C1	1.758 (2)	O1–C4	1.450 (3)
P1–C1A	1.806 (2)	O2–C2	1.212 (2)
P1–C1B	1.812 (2)	O4–C3	1.255 (2)
P1–C1C	1.817 (2)	C1–C3	1.417 (3)
O1–C2	1.343 (3)	C1–C2	1.455 (3)

Table 5

Hydrogen-bond geometry (Å, °) for (II).

Cg1 is the centroid of the C1A–C6A ring, Cg2 that of the C1B–C6B ring and Cg3 that of the C1C–C6C ring.

D–H···A	D–H	H···A	D···A	D–H···A
C6A–H6A···O5	0.95	2.57	3.498 (4)	165
C5–H5E···Cg2	0.98	2.97	3.820 (3)	146
O2D–H2D···O4	0.859 (10)	1.85 (2)	2.636 (3)	152 (4)
C10–H10B···O1D	0.99	2.47	3.430 (3)	163
C2A–H2A···O1D ⁱ	0.95	2.59	3.377 (3)	140
C2D–H2DB···O5 ⁱⁱ	0.98	2.41	3.345 (4)	160
C4B–H4B···Cg3 ⁱⁱⁱ	0.95	2.85	3.711 (4)	152
C9–H9B···Cg1 ^{iv}	0.98	2.86	3.744 (4)	150

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

Table 6

π – π contacts (Å) for (II).

CCD is the centroid–centroid distance and PCD is the (mean) centroid to opposite plane distance; for details, see Janiak (2000). Cg1 is the centroid of the C1A–C6A ring and Cg2 that of the C1B–C6B ring. The rings are parallel by symmetry.

Group 1/group 2	CCD (Å)	PCD (Å)
Cg1···Cg1 ^v	3.762 (2)	3.520 (2)
Cg2···Cg2 ⁱⁱⁱ	3.814 (2)	3.630 (2)

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

Compound (II)

Crystal data

$C_{27}H_{27}O_5P \cdot C_2H_4O_2$	$\gamma = 64.285 (6)^\circ$
$M_r = 522.51$	$V = 1340.7 (9) \text{ \AA}^3$
Triclinic, $P\bar{1}$	$Z = 2$
$a = 9.957 (4) \text{ \AA}$	Mo $K\alpha$ radiation
$b = 10.589 (4) \text{ \AA}$	$\mu = 0.15 \text{ mm}^{-1}$
$c = 14.928 (6) \text{ \AA}$	$T = 150 \text{ K}$
$\alpha = 78.191 (6)^\circ$	$0.46 \times 0.38 \times 0.32 \text{ mm}$
$\beta = 71.505 (6)^\circ$	

Data collection

Bruker SMART CCD area-detector diffractometer	11211 measured reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 2001)	5728 independent reflections
$T_{\min} = 0.93, T_{\max} = 0.95$	4037 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.112$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.058$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.171$	$\Delta\rho_{\text{max}} = 0.49 \text{ e \AA}^{-3}$
$S = 0.93$	$\Delta\rho_{\text{min}} = -0.36 \text{ e \AA}^{-3}$
5728 reflections	
341 parameters	
1 restraint	

Compound (III)

Crystal data

$C_{26}H_{27}O_4P$	$V = 4688.9 (9) \text{ \AA}^3$
$M_r = 434.45$	$Z = 8$
Orthorhombic, $Pbca$	Mo $K\alpha$ radiation
$a = 8.7357 (10) \text{ \AA}$	$\mu = 0.15 \text{ mm}^{-1}$
$b = 16.8280 (18) \text{ \AA}$	$T = 150 \text{ K}$
$c = 31.896 (4) \text{ \AA}$	$0.57 \times 0.47 \times 0.06 \text{ mm}$

Data collection

Bruker SMART CCD area-detector diffractometer	36723 measured reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 2001)	5370 independent reflections
$T_{\min} = 0.92, T_{\max} = 0.99$	3279 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.086$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.052$	280 parameters
$wR(F^2) = 0.139$	H-atom parameters constrained
$S = 0.91$	$\Delta\rho_{\text{max}} = 0.44 \text{ e \AA}^{-3}$
5370 reflections	$\Delta\rho_{\text{min}} = -0.22 \text{ e \AA}^{-3}$

All H atoms were located from difference Fourier maps but were treated differently in refinement. H atoms bonded to C atoms were re-positioned at their geometrically expected locations and allowed to ride, with C—H = 0.95–0.99 Å. The acetic acid H atom bonded to O in (II) was refined with a restrained O—H distance of 0.85 (1) Å and free U_{iso} values; in all remaining cases, $U_{\text{iso}}(\text{H}) = 1.2$ or $1.5U_{\text{eq}}(\text{host})$. The terminal nonylidic ester group in (I) appeared to be disordered over two sets of positions which were almost equally populated. They were refined with restraints on interatomic distances and displacement factors [SAME/SADI and SIMU instructions in *SHELXL97* (Sheldrick, 2008)], and refinement of the occupancies converged to 0.522 (3):0.478 (3).

For all compounds, data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008);

Table 7

Selected bond lengths (Å) for (III).

P1—C1	1.7196 (18)	C1—C10	1.511 (3)
P1—C1B	1.8073 (19)	C2—O2	1.230 (2)
P1—C1A	1.8172 (18)	C2—O1	1.374 (2)
P1—C1C	1.8186 (18)	C4—O1	1.435 (2)
C1—C2	1.404 (3)		

Table 8

Hydrogen-bond geometry (Å, °) for (III).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$C4B-H4B \cdots O2^i$	0.95	2.46	3.389 (3)	165

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

program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

The authors acknowledge the Spanish Research Council (CSIC) for providing a free-of-charge licence to the Cambridge Structural Database (Allen, 2002).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: GT3033). Services for accessing these data are described at the back of the journal.

References

- Abel, A. D., Clark, B. M. & Robinson, W. T. (1989). *Aust. J. Chem.* **35**, 1161–1167.
- Aitken, R. A., Karodia, N. & Lighthfoot, P. (2000). *J. Chem. Soc. Perkin Trans. 2*, pp. 333–340.
- Allen, F. H. (2002). *Acta Cryst.* **B58**, 380–388.
- Bachrach, S. M. (1992). *J. Org. Chem.* **57**, 4367–4373.
- Bachrach, S. M. & Nitsche, C. I. (1994). *The Chemistry of Organophosphorus Compounds*, edited by F. R. Hartley, Vol. 3, ch. 4, pp. 273–302. Chichester: Wiley.
- Bondi, A. (1964). *J. Phys. Chem.* **68**, 441–451.
- Bruker (2001). *SMART-NT* (Version 5.624) and *SAINT-NT* (Version 6.04) for Windows NT. Bruker AXS Inc., Madison, Wisconsin, USA.
- Castañeda, F., Aliaga, C., Bunton, C. A., Garland, M. T. & Baggio, R. (2005). *Acta Cryst.* **C61**, o496–o499.
- Castañeda, F., Silva, P., Garland, M. T., Shirazi, A. & Bunton, C. A. (2009a). *Phosphorus Sulfur Silicon Relat. Elem.* **184**, 19–33.
- Castañeda, F., Silva, P., Garland, M. T., Shirazi, A. & Bunton, C. A. (2009b). *Phosphorus Sulfur Silicon Relat. Elem.* **184**, 2152–2168.
- Castañeda, F., Terraza, C. A., Bunton, C. A., Gillitt, N. D. & Garland, M. T. (2003). *Phosphorus Sulfur Silicon Relat. Elem.* **178**, 1973–1985.
- Castañeda, F., Terraza, C. A., Garland, M. T., Bunton, C. A. & Baggio, R. F. (2001). *Acta Cryst.* **C57**, 180–184.
- Chopard, P. A., Searle, R. J. G. & Devitt, F. H. (1965). *J. Org. Chem.* **30**, 1015–1019.
- Eliel, E. L. & Wilen, S. H. (1994). *Stereochemistry of Organic Compounds*, ch. 4, pp. 618–619. New York: Wiley.
- Gough, S. T. D. & Trippett, S. (1962). *J. Chem. Soc.* pp. 2333–2337.
- Howells, M. A., Howells, R. D., Baenziger, N. C. & Burton, D. J. (1973). *J. Am. Chem. Soc.* **95**, 5366–5370.
- Janiak, C. (2000). *J. Chem. Soc. Dalton Trans.* pp. 3885–3898.
- Nishio, M. & Hirota, M. (1989). *Tetrahedron.* **45**, 7201–7245.
- Nishio, M., Umezawa, Y., Hirota, M. & Takeuchi, Y. (1995). *Tetrahedron.* **51**, 8665–8701.
- Sheldrick, G. M. (2001). *SADABS*. University of Göttingen, Germany.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.