

Dimethyl (2-hydroxy-4-phenylbut-3-en-2-yl)phosphonate

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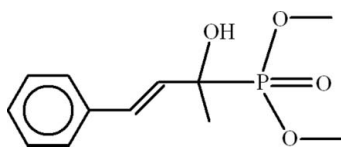
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; disorder in main residue; R factor = 0.061; wR factor = 0.175; data-to-parameter ratio = 13.6.

In the title compound, $\text{C}_{12}\text{H}_{17}\text{O}_4\text{P}$, the phenylbutenyl group is disordered over two sets of sites with an occupancy ratio of 0.755 (12):0.245 (12). In the crystal, inversion dimers linked by pairs of $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds occur, forming $R_2^2(10)$ ring motifs. The packing is consolidated by weak $\text{C}-\text{H}\cdots\pi$ interactions.

Related literature

For related structures, see: Acar *et al.* (2009); Tahir *et al.* (2007, 2009*a,b*). For graph-set theory, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$\text{C}_{12}\text{H}_{17}\text{O}_4\text{P}$
 $M_r = 256.23$
 Monoclinic, $C2/c$
 $a = 17.1522$ (12) Å
 $b = 8.1571$ (13) Å
 $c = 19.5230$ (12) Å
 $\beta = 103.771$ (10)°

$V = 2653.0$ (5) Å³
 $Z = 8$
 Mo $K\alpha$ radiation
 $\mu = 0.21$ mm⁻¹
 $T = 296$ K
 $0.25 \times 0.14 \times 0.12$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer
 Absorption correction: ψ scan (*MolEN*; Fair, 1990)
 $T_{\min} = 0.885$, $T_{\max} = 0.954$
 2519 measured reflections

2415 independent reflections
 1684 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.095$
 3 standard reflections
 frequency: 120 min
 intensity decay: 0.6%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.061$
 $wR(F^2) = 0.175$
 $S = 1.07$
 2415 reflections

177 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.53$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.42$ e Å⁻³

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

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---|-------|-------------|-------------|---------------|
| $\text{O1}-\text{H1}\cdots\text{O2}^{\text{i}}$ | 0.82 | 1.90 | 2.721 (3) | 176 |
| $\text{C6B}-\text{H6B}\cdots\text{Cg1}^{\text{ii}}$ | 0.93 | 2.83 | 3.568 (17) | 137 |
| $\text{C6B}-\text{H6B}\cdots\text{Cg2}^{\text{ii}}$ | 0.93 | 2.94 | 3.652 (17) | 134 |

Symmetry codes: (i) $-x + 1, -y + 1, -z$; (ii) $-x + \frac{1}{2}, y + \frac{1}{2}, -z + \frac{1}{2}$. Cg1 and Cg2 are the centroids of the C1A–C6A and C1B–C6B rings, respectively.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1993); cell refinement: *CAD-4 EXPRESS*; data reduction: *MolEN* (Fair, 1990); program(s) used to solve structure: *WinGX* (Farrugia, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5172).

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supplementary materials

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Dimethyl (2-hydroxy-4-phenylbut-3-en-2-yl)phosphonate

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Comment

We have reported the crystal structures which contains α -hydroxy phosphonate such as (II) Dimethyl (1-hydroxy-1,2-diphenylethyl)phosphonate (Tahir *et al.*, 2009*a*), (III) Dimethyl [hydroxy(2-nitrophenyl)methyl]phosphonate (Tahir *et al.*, 2009*b*), (IV) (*R*)-Dimethyl [(2-chlorophenyl)hydroxymethyl]phosphonate (Tahir *et al.*, 2007) and Diethyl (1-hydroxy-1,2-diphenylethyl)phosphonate (Acar *et al.*, 2009). The title compound (I, Fig. 1) is in continuation of synthesizing various α -hydroxy phosphonates.

In the crystal structure of title compound phenylbutan is disordered over two possible sites with occupancy ratio of 0.755 (12):0.245 (12). The benzene rings of disordered moieties A (C1A—C6A) and B (C1B—C6B) are nearly planar to one another as the dihedral angle between A/B is 2.76 (1.35)°. The molecules of title compound are dimerized due to O—H \cdots O type of intermolecular H-bondings (Table 1, Fig. 3) forming $R_2^2(10)$ ring motif (Bernstein *et al.*, 1995). The molecules are stabilized due to C—H \cdots π interactions (Table 1).

Experimental

Benzylacetone (4.45 g, 30 mmol) and dimethylphosphonate (3.30 g, 30 mmol) were dissolved in 50 ml of tetrahydrofuran. The mixture was cooled to 273 K and in it KF (1.74 g, 30 mmol) and γ -Al₂O₃ (1.74 g, 17 mmol) were added and refluxed. The precipitates obtained after 48 h were washed with hot distilled water (50 ml) and dried. The crude material was dissolved in distilled water with few drops of ethyl alcohol and colourless needles of (I) were obtained after 4 days.

Refinement

The H-atoms were positioned geometrically (O—H = 0.82 Å, C—H = 0.93-0.96 Å) and refined as riding with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{carrier})$ or $1.5U_{\text{eq}}(\text{methyl C})$.

The incorrect bond distances and higher thermal parameters of phenylbutan lead to disorder. In the disordered group the benzene rings were refined using *AFIX 66*. The benzene ring B (C2B—C6B) was refined using *EADP*.

Figures

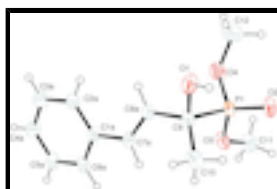


Fig. 1. View of (I) with the atom numbering scheme for atoms of greater occupancy ratio. The displacement ellipsoids are drawn at the 30% probability level. H-atoms are shown by small circles of arbitrary radii.

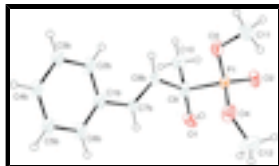


Fig. 2. View of the title compound with the atom numbering scheme for atoms of smaller occupancy ratio. The displacement ellipsoids are drawn at the 30% probability level. H-atoms are shown by small circles of arbitrary radii.

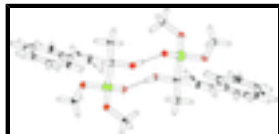


Fig. 3. The partial packing of (I), which shows that molecules form inversion dimers.

Dimethyl (2-hydroxy-4-phenylbut-3-en-2-yl)phosphonate

Crystal data

$C_{12}H_{17}O_4P$

$M_r = 256.23$

Monoclinic, $C2/c$

Hall symbol: $-C\ 2yc$

$a = 17.1522\ (12)\ \text{\AA}$

$b = 8.1571\ (13)\ \text{\AA}$

$c = 19.5230\ (12)\ \text{\AA}$

$\beta = 103.771\ (10)^\circ$

$V = 2653.0\ (5)\ \text{\AA}^3$

$Z = 8$

$F_{000} = 1088$

$D_x = 1.283\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 25 reflections

$\theta = 9.9\text{--}13.9^\circ$

$\mu = 0.21\ \text{mm}^{-1}$

$T = 296\ \text{K}$

Needle, colourless

$0.25 \times 0.14 \times 0.12\ \text{mm}$

Data collection

Enraf–Nonius CAD-4 diffractometer

$T = 296\ \text{K}$

$\omega/2\theta$ scans

Absorption correction: ψ scan
(MolEN; Fair, 1990)

$T_{\min} = 0.885$, $T_{\max} = 0.954$

2519 measured reflections

2415 independent reflections

1684 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.095$

$\theta_{\max} = 25.5^\circ$

$\theta_{\min} = 2.8^\circ$

$h = -20 \rightarrow 20$

$k = 0 \rightarrow 9$

$l = -23 \rightarrow 0$

3 standard reflections

every 120 min

intensity decay: 0.6%

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.061$

$wR(F^2) = 0.175$

$S = 1.07$

Secondary atom site location: difference Fourier map

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.114P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.53\ \text{e \AA}^{-3}$

2415 reflections

$$\Delta\rho_{\min} = -0.42 \text{ e } \text{\AA}^{-3}$$

177 parameters

Extinction correction: none

Primary atom site location: structure-invariant direct methods

Special details

Geometry. Bond distances, angles *etc.* have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ | Occ. (<1) |
|------|--------------|-------------|---------------|----------------------------------|------------|
| P1 | 0.43961 (4) | 0.29251 (9) | 0.06739 (4) | 0.0399 (3) | |
| O1 | 0.43699 (13) | 0.6068 (3) | 0.08493 (11) | 0.0548 (8) | |
| O2 | 0.49603 (12) | 0.2946 (3) | 0.02130 (12) | 0.0528 (8) | |
| O3 | 0.37510 (12) | 0.1526 (2) | 0.05085 (11) | 0.0500 (7) | |
| O4 | 0.48115 (13) | 0.2636 (3) | 0.14713 (12) | 0.0597 (8) | |
| C1A | 0.2124 (3) | 0.4228 (6) | 0.1674 (2) | 0.0453 (16) | 0.755 (12) |
| C2A | 0.23963 (19) | 0.4970 (7) | 0.2329 (3) | 0.058 (2) | 0.755 (12) |
| C3A | 0.1939 (3) | 0.4884 (9) | 0.28271 (18) | 0.078 (2) | 0.755 (12) |
| C4A | 0.1209 (3) | 0.4058 (9) | 0.2671 (3) | 0.072 (2) | 0.755 (12) |
| C5A | 0.0937 (2) | 0.3316 (6) | 0.2017 (3) | 0.062 (2) | 0.755 (12) |
| C6A | 0.1395 (3) | 0.3401 (6) | 0.15183 (15) | 0.0524 (17) | 0.755 (12) |
| C7A | 0.2601 (3) | 0.4283 (6) | 0.1139 (2) | 0.0451 (16) | 0.755 (12) |
| C8A | 0.3344 (4) | 0.4814 (7) | 0.1215 (3) | 0.0414 (19) | 0.755 (12) |
| C9 | 0.37966 (18) | 0.4799 (3) | 0.06310 (16) | 0.0440 (10) | |
| C10 | 0.3323 (2) | 0.5090 (4) | -0.01138 (18) | 0.0584 (11) | |
| C11 | 0.4001 (2) | -0.0152 (4) | 0.0447 (2) | 0.0644 (13) | |
| C12 | 0.5626 (2) | 0.3125 (6) | 0.1775 (2) | 0.0896 (19) | |
| C6B | 0.1066 (9) | 0.341 (2) | 0.1847 (9) | 0.061 (3) | 0.245 (12) |
| C7B | 0.3065 (12) | 0.441 (2) | 0.0916 (13) | 0.046 (6) | 0.245 (12) |
| C1B | 0.1670 (11) | 0.380 (2) | 0.1509 (5) | 0.061 (3) | 0.245 (12) |
| C2B | 0.2328 (8) | 0.471 (2) | 0.1857 (9) | 0.061 (3) | 0.245 (12) |
| C3B | 0.2381 (8) | 0.525 (2) | 0.2543 (9) | 0.061 (3) | 0.245 (12) |
| C4B | 0.1777 (12) | 0.486 (3) | 0.2880 (7) | 0.061 (3) | 0.245 (12) |
| C5B | 0.1119 (10) | 0.395 (3) | 0.2532 (9) | 0.061 (3) | 0.245 (12) |
| C8B | 0.2981 (10) | 0.5086 (19) | 0.1470 (10) | 0.057 (6) | 0.245 (12) |
| H1 | 0.45706 | 0.63143 | 0.05218 | 0.0821* | |
| H2A | 0.28844 | 0.55226 | 0.24331 | 0.0697* | 0.755 (12) |
| H10C | 0.36812 | 0.51292 | -0.04233 | 0.0875* | |
| H11A | 0.35594 | -0.08762 | 0.04446 | 0.0966* | |
| H11B | 0.41720 | -0.02795 | 0.00165 | 0.0966* | |
| H11C | 0.44381 | -0.04117 | 0.08403 | 0.0966* | |
| H12A | 0.59764 | 0.26187 | 0.15202 | 0.1344* | |
| H12B | 0.56693 | 0.42950 | 0.17485 | 0.1344* | |
| H12C | 0.57762 | 0.27862 | 0.22598 | 0.1344* | |

supplementary materials

| | | | | | |
|------|---------|---------|----------|---------|------------|
| H3A | 0.21203 | 0.53806 | 0.32650 | 0.0928* | 0.755 (12) |
| H4A | 0.09026 | 0.40008 | 0.30047 | 0.0865* | 0.755 (12) |
| H5A | 0.04489 | 0.27630 | 0.19124 | 0.0741* | 0.755 (12) |
| H6A | 0.12130 | 0.29050 | 0.10804 | 0.0629* | 0.755 (12) |
| H7A | 0.23485 | 0.38956 | 0.06932 | 0.0541* | 0.755 (12) |
| H8A | 0.36086 | 0.52242 | 0.16532 | 0.0500* | 0.755 (12) |
| H10A | 0.29444 | 0.42147 | -0.02555 | 0.0875* | |
| H10B | 0.30398 | 0.61121 | -0.01371 | 0.0875* | |
| H1B | 0.16342 | 0.34369 | 0.10503 | 0.0729* | 0.245 (12) |
| H3B | 0.28213 | 0.58635 | 0.27758 | 0.0729* | 0.245 (12) |
| H4B | 0.18126 | 0.52218 | 0.33391 | 0.0729* | 0.245 (12) |
| H5B | 0.07147 | 0.36876 | 0.27580 | 0.0729* | 0.245 (12) |
| H6B | 0.06255 | 0.27951 | 0.16136 | 0.0729* | 0.245 (12) |
| H7B | 0.26815 | 0.36654 | 0.06805 | 0.0543* | 0.245 (12) |
| H8B | 0.33514 | 0.58922 | 0.16626 | 0.0680* | 0.245 (12) |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| P1 | 0.0354 (4) | 0.0436 (5) | 0.0463 (5) | 0.0019 (3) | 0.0206 (3) | 0.0024 (3) |
| O1 | 0.0676 (15) | 0.0482 (12) | 0.0568 (13) | -0.0064 (11) | 0.0312 (12) | -0.0074 (10) |
| O2 | 0.0520 (13) | 0.0542 (13) | 0.0644 (14) | 0.0030 (10) | 0.0382 (11) | 0.0013 (10) |
| O3 | 0.0399 (12) | 0.0423 (11) | 0.0723 (15) | 0.0011 (9) | 0.0225 (10) | -0.0003 (10) |
| O4 | 0.0499 (13) | 0.0785 (15) | 0.0530 (14) | 0.0069 (12) | 0.0167 (11) | 0.0118 (11) |
| C1A | 0.037 (3) | 0.064 (3) | 0.036 (2) | 0.007 (2) | 0.011 (2) | -0.005 (2) |
| C2A | 0.043 (3) | 0.093 (4) | 0.045 (4) | -0.002 (2) | 0.022 (2) | -0.011 (3) |
| C3A | 0.055 (4) | 0.140 (5) | 0.047 (3) | -0.025 (4) | 0.031 (3) | -0.022 (3) |
| C4A | 0.053 (4) | 0.121 (5) | 0.051 (3) | -0.013 (3) | 0.029 (3) | -0.020 (3) |
| C5A | 0.046 (3) | 0.096 (4) | 0.054 (4) | 0.000 (2) | 0.033 (2) | -0.008 (3) |
| C6A | 0.035 (3) | 0.078 (3) | 0.051 (3) | -0.002 (2) | 0.024 (2) | -0.007 (2) |
| C7A | 0.040 (3) | 0.061 (3) | 0.040 (2) | -0.007 (2) | 0.021 (2) | -0.011 (2) |
| C8A | 0.038 (4) | 0.047 (3) | 0.040 (3) | 0.002 (2) | 0.011 (3) | -0.005 (2) |
| C9 | 0.0388 (16) | 0.0440 (16) | 0.0560 (18) | 0.0029 (13) | 0.0245 (14) | 0.0051 (13) |
| C10 | 0.051 (2) | 0.0560 (19) | 0.069 (2) | 0.0017 (15) | 0.0160 (17) | 0.0091 (16) |
| C11 | 0.066 (2) | 0.0462 (18) | 0.085 (3) | 0.0049 (16) | 0.026 (2) | -0.0011 (16) |
| C12 | 0.065 (3) | 0.119 (4) | 0.074 (3) | -0.015 (2) | -0.005 (2) | -0.003 (2) |
| C6B | 0.037 (4) | 0.112 (7) | 0.040 (5) | -0.002 (4) | 0.024 (3) | -0.003 (4) |
| C7B | 0.034 (10) | 0.047 (9) | 0.058 (13) | 0.003 (7) | 0.015 (9) | 0.002 (7) |
| C1B | 0.037 (4) | 0.112 (7) | 0.040 (5) | -0.002 (4) | 0.024 (3) | -0.003 (4) |
| C2B | 0.037 (4) | 0.112 (7) | 0.040 (5) | -0.002 (4) | 0.024 (3) | -0.003 (4) |
| C3B | 0.037 (4) | 0.112 (7) | 0.040 (5) | -0.002 (4) | 0.024 (3) | -0.003 (4) |
| C4B | 0.037 (4) | 0.112 (7) | 0.040 (5) | -0.002 (4) | 0.024 (3) | -0.003 (4) |
| C5B | 0.037 (4) | 0.112 (7) | 0.040 (5) | -0.002 (4) | 0.024 (3) | -0.003 (4) |
| C8B | 0.036 (8) | 0.056 (9) | 0.083 (12) | -0.010 (7) | 0.023 (9) | -0.028 (8) |

Geometric parameters (\AA , $^\circ$)

| | | | |
|-------|-----------|--------|-----------|
| P1—O2 | 1.470 (2) | C8A—C9 | 1.525 (7) |
| P1—O3 | 1.569 (2) | C9—C10 | 1.506 (5) |

| | | | |
|-------------|-------------|-------------|--------|
| P1—O4 | 1.568 (2) | C1B—H1B | 0.9300 |
| P1—C9 | 1.833 (3) | C2A—H2A | 0.9300 |
| O1—C9 | 1.422 (4) | C3A—H3A | 0.9300 |
| O3—C11 | 1.448 (4) | C3B—H3B | 0.9300 |
| O4—C12 | 1.438 (4) | C4A—H4A | 0.9300 |
| O1—H1 | 0.8200 | C4B—H4B | 0.9300 |
| C1A—C2A | 1.391 (7) | C5A—H5A | 0.9300 |
| C1A—C7A | 1.473 (7) | C5B—H5B | 0.9300 |
| C1A—C6A | 1.389 (7) | C6A—H6A | 0.9300 |
| C1B—C2B | 1.39 (2) | C6B—H6B | 0.9300 |
| C1B—C6B | 1.39 (2) | C7A—H7A | 0.9300 |
| C2A—C3A | 1.389 (6) | C7B—H7B | 0.9300 |
| C2B—C8B | 1.52 (2) | C8A—H8A | 0.9300 |
| C2B—C3B | 1.39 (2) | C8B—H8B | 0.9300 |
| C3A—C4A | 1.390 (8) | C10—H10B | 0.9600 |
| C3B—C4B | 1.39 (2) | C10—H10A | 0.9600 |
| C4A—C5A | 1.390 (8) | C10—H10C | 0.9600 |
| C4B—C5B | 1.39 (3) | C11—H11A | 0.9600 |
| C5A—C6A | 1.391 (6) | C11—H11B | 0.9600 |
| C5B—C6B | 1.39 (2) | C11—H11C | 0.9600 |
| C7A—C8A | 1.321 (9) | C12—H12A | 0.9600 |
| C7B—C8B | 1.25 (3) | C12—H12B | 0.9600 |
| C7B—C9 | 1.52 (2) | C12—H12C | 0.9600 |
| O2—P1—O3 | 114.66 (13) | C2A—C3A—H3A | 120.00 |
| O2—P1—O4 | 113.56 (13) | C4A—C3A—H3A | 120.00 |
| O2—P1—C9 | 113.97 (14) | C2B—C3B—H3B | 120.00 |
| O3—P1—O4 | 103.09 (12) | C4B—C3B—H3B | 120.00 |
| O3—P1—C9 | 103.70 (12) | C3A—C4A—H4A | 120.00 |
| O4—P1—C9 | 106.75 (14) | C5A—C4A—H4A | 120.00 |
| P1—O3—C11 | 119.8 (2) | C3B—C4B—H4B | 120.00 |
| P1—O4—C12 | 122.3 (2) | C5B—C4B—H4B | 120.00 |
| C9—O1—H1 | 109.00 | C4A—C5A—H5A | 120.00 |
| C2A—C1A—C7A | 121.1 (4) | C6A—C5A—H5A | 120.00 |
| C6A—C1A—C7A | 118.9 (4) | C4B—C5B—H5B | 120.00 |
| C2A—C1A—C6A | 120.0 (4) | C6B—C5B—H5B | 120.00 |
| C2B—C1B—C6B | 120.1 (12) | C5A—C6A—H6A | 120.00 |
| C1A—C2A—C3A | 120.0 (4) | C1A—C6A—H6A | 120.00 |
| C1B—C2B—C3B | 120.0 (14) | C1B—C6B—H6B | 120.00 |
| C1B—C2B—C8B | 118.3 (14) | C5B—C6B—H6B | 120.00 |
| C3B—C2B—C8B | 121.7 (14) | C1A—C7A—H7A | 116.00 |
| C2A—C3A—C4A | 120.0 (5) | C8A—C7A—H7A | 116.00 |
| C2B—C3B—C4B | 119.8 (15) | C9—C7B—H7B | 120.00 |
| C3A—C4A—C5A | 120.0 (5) | C8B—C7B—H7B | 120.00 |
| C3B—C4B—C5B | 120.2 (15) | C7A—C8A—H8A | 118.00 |
| C4A—C5A—C6A | 120.0 (4) | C9—C8A—H8A | 118.00 |
| C4B—C5B—C6B | 120.0 (16) | C2B—C8B—H8B | 117.00 |
| C1A—C6A—C5A | 120.0 (4) | C7B—C8B—H8B | 117.00 |
| C1B—C6B—C5B | 119.9 (15) | C9—C10—H10C | 110.00 |
| C1A—C7A—C8A | 127.9 (4) | C9—C10—H10B | 109.00 |

supplementary materials

| | | | |
|--------------|-------------|-----------------|------------|
| C8B—C7B—C9 | 119.9 (17) | C9—C10—H10A | 109.00 |
| C7A—C8A—C9 | 124.3 (5) | H10B—C10—H10C | 109.00 |
| C2B—C8B—C7B | 126.1 (17) | H10A—C10—H10B | 109.00 |
| C7B—C9—C10 | 94.8 (9) | H10A—C10—H10C | 110.00 |
| O1—C9—C10 | 110.5 (2) | H11A—C11—H11B | 110.00 |
| O1—C9—C7B | 127.9 (8) | O3—C11—H11A | 109.00 |
| C8A—C9—C10 | 117.9 (3) | O3—C11—H11B | 109.00 |
| P1—C9—O1 | 104.7 (2) | O3—C11—H11C | 109.00 |
| P1—C9—C8A | 110.6 (3) | H11A—C11—H11C | 109.00 |
| P1—C9—C10 | 110.1 (2) | H11B—C11—H11C | 109.00 |
| P1—C9—C7B | 108.2 (7) | H12B—C12—H12C | 109.00 |
| O1—C9—C8A | 102.1 (3) | O4—C12—H12A | 109.00 |
| C2B—C1B—H1B | 120.00 | O4—C12—H12B | 109.00 |
| C6B—C1B—H1B | 120.00 | O4—C12—H12C | 109.00 |
| C1A—C2A—H2A | 120.00 | H12A—C12—H12B | 110.00 |
| C3A—C2A—H2A | 120.00 | H12A—C12—H12C | 109.00 |
| O2—P1—O3—C11 | 50.8 (3) | C6A—C1A—C2A—C3A | 0.0 (8) |
| O4—P1—O3—C11 | -73.1 (2) | C7A—C1A—C2A—C3A | -179.3 (5) |
| C9—P1—O3—C11 | 175.7 (2) | C2A—C1A—C6A—C5A | 0.1 (7) |
| O2—P1—O4—C12 | 28.4 (3) | C7A—C1A—C6A—C5A | 179.3 (4) |
| O3—P1—O4—C12 | 153.1 (3) | C2A—C1A—C7A—C8A | 8.7 (8) |
| C9—P1—O4—C12 | -98.0 (3) | C6A—C1A—C7A—C8A | -170.6 (5) |
| O2—P1—C9—O1 | -60.4 (2) | C1A—C2A—C3A—C4A | -0.1 (9) |
| O2—P1—C9—C8A | -169.6 (3) | C2A—C3A—C4A—C5A | 0.1 (10) |
| O2—P1—C9—C10 | 58.4 (3) | C3A—C4A—C5A—C6A | -0.1 (9) |
| O3—P1—C9—O1 | 174.29 (18) | C4A—C5A—C6A—C1A | 0.0 (8) |
| O3—P1—C9—C8A | 65.1 (3) | C1A—C7A—C8A—C9 | 179.0 (4) |
| O3—P1—C9—C10 | -67.0 (2) | C7A—C8A—C9—P1 | -93.6 (6) |
| O4—P1—C9—O1 | 65.8 (2) | C7A—C8A—C9—O1 | 155.5 (5) |
| O4—P1—C9—C8A | -43.4 (3) | C7A—C8A—C9—C10 | 34.2 (7) |
| O4—P1—C9—C10 | -175.4 (2) | | |

Hydrogen-bond geometry (\AA , $^\circ$)

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|------------------------------------|-------|-------------|-------------|---------------|
| O1—H1 \cdots O2 ⁱ | 0.82 | 1.90 | 2.721 (3) | 176 |
| C6B—H6B \cdots Cg1 ⁱⁱ | 0.93 | 2.83 | 3.568 (17) | 137 |
| C6B—H6B \cdots Cg2 ⁱⁱ | 0.93 | 2.94 | 3.652 (17) | 134 |

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

Fig. 1

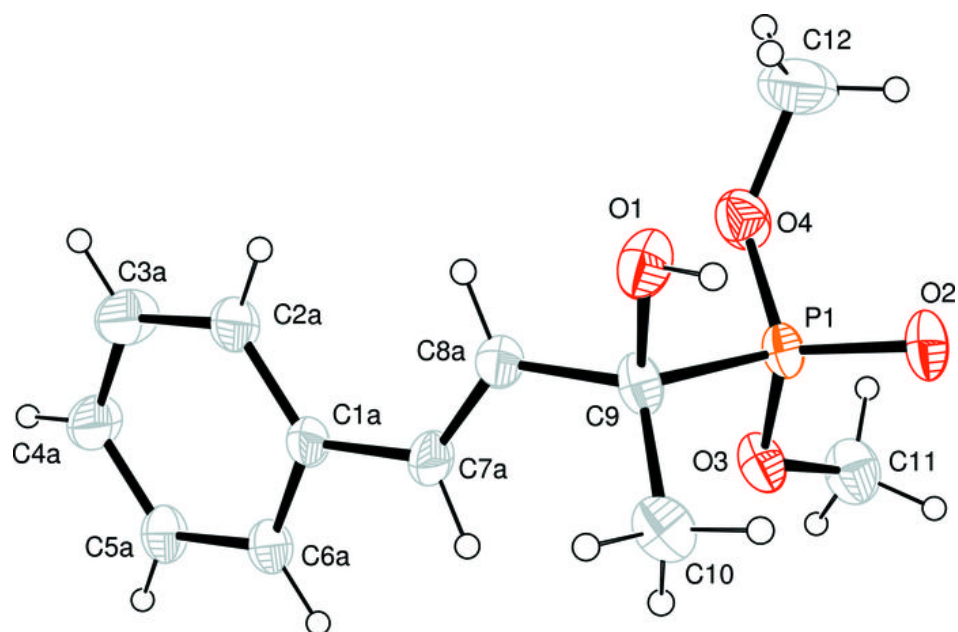


Fig. 2

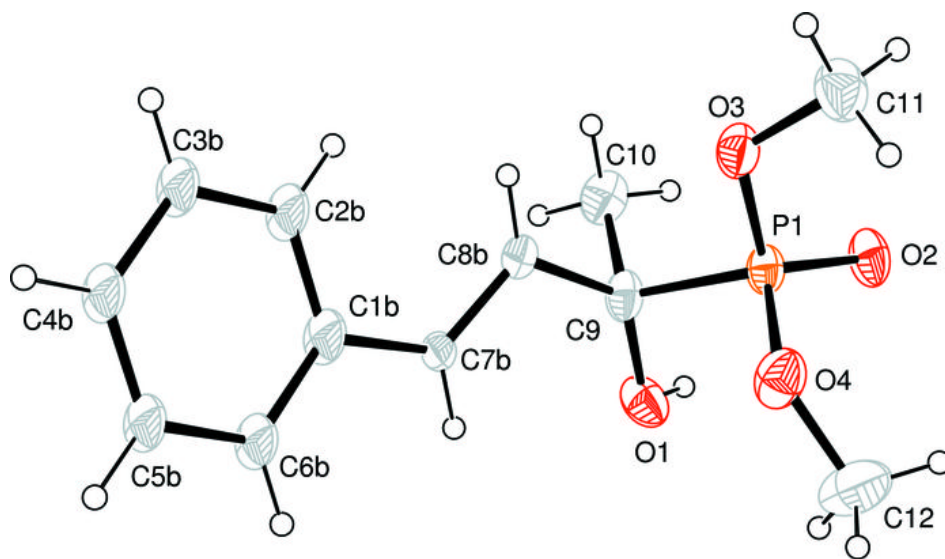


Fig. 3

