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(S)-2-[1-(4-Bromophenyl)-1-hydroxyethyl]-5,5-dimethyl-1,3,2-dioxaphosphinane 2-oxide

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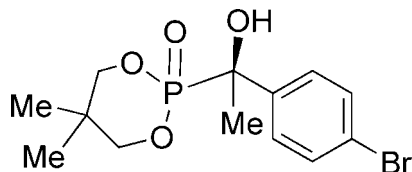
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 Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.045; wR factor = 0.105; data-to-parameter ratio = 20.6.

In the crystal structure of the title molecule, $\text{C}_{13}\text{H}_{18}\text{BrO}_4\text{P}$, the phosphonate ring adopts a chair conformation. Molecules are linked by an $\text{O}-\text{H}\cdots\text{O}$ hydrogen bond [$\text{O}\cdots\text{O} = 2.780$ (3) Å] to form chains parallel to the c axis. Two $\text{C}-\text{H}\cdots\text{O}$ interactions help to stabilize the crystal structure.

Related literature

For the synthesis and biological activity of hydroxyphosphonate derivatives, see: Peng *et al.* (2007); Liu *et al.* (2006). For the synthesis of hydroxyphosphonates, see: Zhou *et al.* (2008). For standard bond lengths, see: (Allen *et al.*, 1987).



Experimental

Crystal data

 $\text{C}_{13}\text{H}_{18}\text{BrO}_4\text{P}$
 $M_r = 349.15$

 Orthorhombic, $P2_12_12_1$
 $a = 11.0662$ (17) Å

 $b = 11.3149$ (18) Å

 $c = 11.9609$ (19) Å

 $V = 1497.7$ (4) Å³
 $Z = 4$

 Mo $K\alpha$ radiation

 $\mu = 2.86$ mm⁻¹
 $T = 298$ K

 $0.20 \times 0.12 \times 0.10$ mm

Data collection

 Bruker SMART APEX CCD area-detector diffractometer
10072 measured reflections

 3627 independent reflections
2691 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.114$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.045$
 $wR(F^2) = 0.105$
 $S = 0.93$

3627 reflections

176 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.46$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.45$ e Å⁻³

Absolute structure: Flack (1983),

with 1517 Friedel pairs

 Flack parameter: -0.011 (10)

Table 1

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|---|--------------|--------------------|-------------|----------------------|
| $\text{O1}-\text{H1}\cdots\text{O2}^i$ | 0.82 | 2.01 | 2.780 (3) | 156 |
| $\text{C9}-\text{H9B}\cdots\text{O1}$ | 0.97 | 2.58 | 3.163 (4) | 119 |
| $\text{C11}-\text{H11A}\cdots\text{O2}^i$ | 0.97 | 2.57 | 3.517 (4) | 165 |

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

Data collection: SMART (Bruker, 2001); cell refinement: SAINT-Plus (Bruker, 2001); data reduction: SAINT-Plus; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: PLATON (Spek, 2009); software used to prepare material for publication: PLATON and SHELXL97.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: QK2003).

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supporting information

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(S)-2-[1-(4-Bromophenyl)-1-hydroxyethyl]-5,5-dimethyl-1,3,2-dioxaphosphinane 2-oxide

Chubei Wang, Hao Peng and Hongwu He

S1. Comment

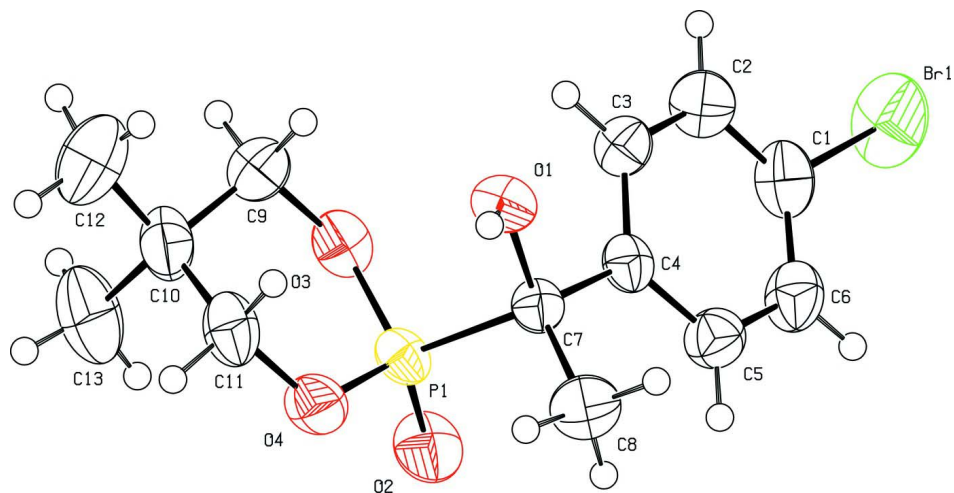
Acyclic and cyclic α -hydroxyphosphonates can be used as very viable intermediates and they are also an attractive class of biologically active compounds (Peng *et al.*, 2007; Liu *et al.*, 2006). In our research work aimed at searching for novel agrochemicals, we attempted to synthesize hydroxyphosphonates according to published literature procedures. Here we report the synthesis and crystal structure of the chiral title compound (I) (Fig. 1). The bond lengths and angles show normal values (Allen *et al.*, 1987). In the crystal structure, the cyclic phosphonate ring adopts a chair conformation.

S2. Experimental

Hydroxyphosphonate (I) was prepared according to a literature procedure (Zhou *et al.*, 2008). Diethylaluminium chloride (1 mmol) was added to a solution of (*S,E*)-2-(adamantan-1-yl)-4-(*tert*-butyl)-6(((1-hydroxy-3-methylbutan-2-yl)imino)-methyl)-phenol (1 mmol) in dichloromethane (10 ml). The mixture was stirred at room temperature for 1 h. The ketone (11 mmol) and the cyclic phosphate (10 mmol) were added and the mixture was stirred for 2 h. The reaction was quenched by diluted hydrochloric acid (15:1, *v/v*). The pure hydroxyphosphonate was afforded by column chromatography on silica gel (acetone/petroleum ether = 1:2), 71% yield, $[\alpha]_D = -64.8^\circ$ ($c = 0.56$, chloroform). Then recrystallization from acetic ester over a period of one week gave colourless crystals of (I).

S3. Refinement

C-bound H atoms were geometrically positioned (C—H = 0.93–0.98 Å) and refined as riding, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}} - 1.5U_{\text{eq}}(\text{C})$. The O-bound H atom was located from a difference Fourier map and refined as riding, with O—H = 0.81 (2) Å and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$.

**Figure 1**

Molecular structure of (I), showing the labeling scheme and 50% probability thermal ellipsoids.

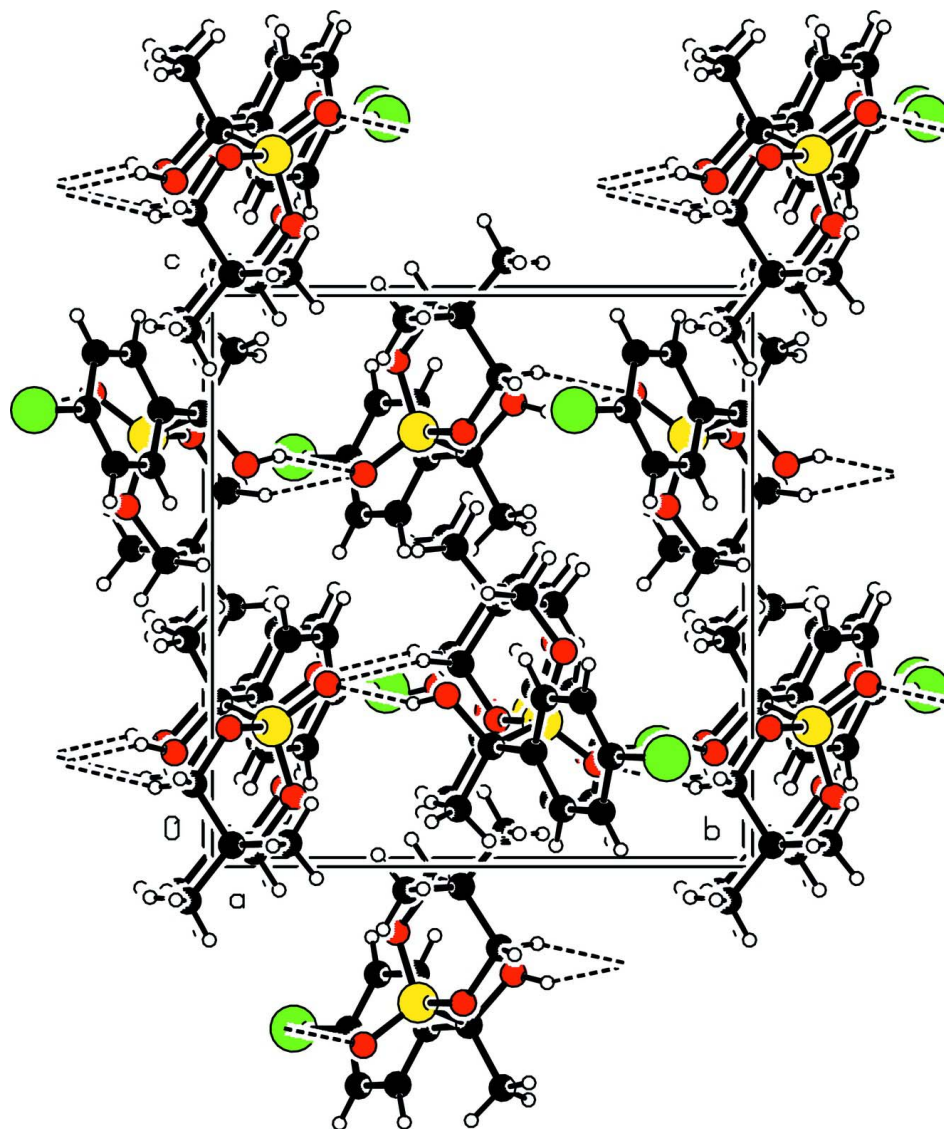


Figure 2

Part of the crystal packing, showing the intermolecular hydrogen bonds as dashed lines.

(S)-2-[1-(4-Bromophenyl)-1-hydroxyethyl]-5,5-dimethyl-1,3,2-dioxaphosphinane 2-oxide

Crystal data

$C_{13}H_{18}BrO_4P$

$M_r = 349.15$

Orthorhombic, $P2_12_12_1$

Hall symbol: P 2ac 2ab

$a = 11.0662 (17) \text{ \AA}$

$b = 11.3149 (18) \text{ \AA}$

$c = 11.9609 (19) \text{ \AA}$

$V = 1497.7 (4) \text{ \AA}^3$

$Z = 4$

$F(000) = 712$

$D_x = 1.549 \text{ Mg m}^{-3}$

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

Cell parameters from 3882 reflections

$\theta = 2.5\text{--}26.7^\circ$

$\mu = 2.86 \text{ mm}^{-1}$

$T = 298 \text{ K}$

Block, colourless

$0.20 \times 0.12 \times 0.10 \text{ mm}$

Data collection

Bruker SMART APEX CCD area-detector
diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
 φ and ω scans
10072 measured reflections
3627 independent reflections

2691 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.114$
 $\theta_{\text{max}} = 28.3^\circ$, $\theta_{\text{min}} = 2.5^\circ$
 $h = -14 \rightarrow 14$
 $k = -14 \rightarrow 14$
 $l = -15 \rightarrow 12$

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.045$
 $wR(F^2) = 0.105$
 $S = 0.93$
3627 reflections
176 parameters
0 restraints
Primary atom site location: structure-invariant
direct methods
Secondary atom site location: difference Fourier
map

Hydrogen site location: inferred from
neighbouring sites
H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0423P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.46 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.45 \text{ e } \text{\AA}^{-3}$
Absolute structure: Flack (1983), with 1517
Friedel pairs
Absolute structure parameter: -0.011 (10)

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 (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|-------------|-------------|----------------------------------|
| Br1 | -0.07457 (4) | 0.16872 (4) | 0.69930 (5) | 0.0869 (2) |
| C1 | 0.0650 (3) | 0.2689 (3) | 0.7001 (3) | 0.0535 (8) |
| C2 | 0.0982 (3) | 0.3216 (3) | 0.7973 (3) | 0.0533 (8) |
| H2 | 0.0520 | 0.3113 | 0.8615 | 0.064* |
| C3 | 0.2007 (3) | 0.3905 (3) | 0.8000 (3) | 0.0429 (7) |
| H3 | 0.2244 | 0.4250 | 0.8671 | 0.052* |
| C4 | 0.2690 (2) | 0.4094 (2) | 0.7050 (3) | 0.0373 (6) |
| C5 | 0.2317 (3) | 0.3561 (3) | 0.6058 (3) | 0.0498 (8) |
| H5 | 0.2762 | 0.3679 | 0.5408 | 0.060* |
| C6 | 0.1288 (3) | 0.2857 (3) | 0.6026 (3) | 0.0542 (9) |
| H6 | 0.1037 | 0.2507 | 0.5362 | 0.065* |
| C7 | 0.3824 (3) | 0.4854 (2) | 0.7128 (3) | 0.0364 (6) |
| C8 | 0.4129 (4) | 0.5472 (3) | 0.6033 (3) | 0.0557 (9) |
| H8A | 0.3464 | 0.5965 | 0.5813 | 0.084* |
| H8B | 0.4277 | 0.4892 | 0.5464 | 0.084* |

| | | | | |
|------|--------------|--------------|--------------|--------------|
| H8C | 0.4838 | 0.5949 | 0.6133 | 0.084* |
| C9 | 0.5250 (3) | 0.4228 (4) | 0.9724 (3) | 0.0608 (10) |
| H9A | 0.5186 | 0.3769 | 1.0406 | 0.073* |
| H9B | 0.4712 | 0.4902 | 0.9791 | 0.073* |
| C10 | 0.6530 (3) | 0.4663 (4) | 0.9596 (3) | 0.0598 (10) |
| C11 | 0.6616 (3) | 0.5380 (3) | 0.8524 (3) | 0.0544 (9) |
| H11A | 0.6113 | 0.6079 | 0.8592 | 0.065* |
| H11B | 0.7444 | 0.5638 | 0.8419 | 0.065* |
| C12 | 0.6801 (5) | 0.5486 (6) | 1.0566 (4) | 0.1081 (19) |
| H12A | 0.6297 | 0.6174 | 1.0514 | 0.162* |
| H12B | 0.7635 | 0.5720 | 1.0540 | 0.162* |
| H12C | 0.6644 | 0.5085 | 1.1258 | 0.162* |
| C13 | 0.7422 (4) | 0.3633 (4) | 0.9547 (5) | 0.0885 (15) |
| H13A | 0.7281 | 0.3112 | 1.0167 | 0.133* |
| H13B | 0.8233 | 0.3932 | 0.9585 | 0.133* |
| H13C | 0.7314 | 0.3208 | 0.8860 | 0.133* |
| O1 | 0.36447 (19) | 0.56809 (16) | 0.80211 (19) | 0.0426 (5) |
| H1 | 0.3995 | 0.6301 | 0.7874 | 0.064* |
| O2 | 0.5245 (2) | 0.28766 (19) | 0.6800 (2) | 0.0596 (6) |
| O3 | 0.48668 (19) | 0.34977 (18) | 0.8773 (2) | 0.0497 (5) |
| O4 | 0.6236 (2) | 0.4709 (2) | 0.7554 (2) | 0.0520 (6) |
| P1 | 0.50921 (6) | 0.38934 (6) | 0.75398 (7) | 0.03831 (18) |

Atomic displacement parameters (Å²)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| Br1 | 0.0855 (3) | 0.0813 (3) | 0.0938 (3) | -0.0491 (2) | -0.0168 (3) | 0.0076 (2) |
| C1 | 0.0510 (16) | 0.0373 (14) | 0.072 (2) | -0.0124 (14) | -0.013 (2) | 0.0034 (18) |
| C2 | 0.0555 (18) | 0.0470 (16) | 0.057 (2) | -0.0092 (15) | 0.0010 (18) | 0.0093 (18) |
| C3 | 0.0469 (15) | 0.0378 (14) | 0.0441 (16) | -0.0061 (13) | -0.0027 (15) | -0.0018 (16) |
| C4 | 0.0366 (13) | 0.0287 (12) | 0.0464 (16) | 0.0067 (11) | -0.0042 (14) | -0.0006 (13) |
| C5 | 0.0528 (17) | 0.0502 (19) | 0.0464 (18) | 0.0022 (17) | -0.0007 (15) | -0.0032 (16) |
| C6 | 0.0547 (18) | 0.0492 (18) | 0.059 (2) | -0.0037 (18) | -0.0127 (18) | -0.0109 (17) |
| C7 | 0.0373 (13) | 0.0317 (12) | 0.0400 (15) | 0.0021 (11) | -0.0015 (13) | -0.0030 (12) |
| C8 | 0.064 (2) | 0.0477 (17) | 0.055 (2) | -0.0051 (19) | 0.0061 (18) | 0.0061 (16) |
| C9 | 0.0599 (19) | 0.077 (2) | 0.0454 (19) | 0.006 (2) | -0.0014 (17) | 0.0083 (18) |
| C10 | 0.0461 (17) | 0.067 (2) | 0.067 (2) | 0.0070 (18) | -0.0158 (18) | -0.002 (2) |
| C11 | 0.0437 (16) | 0.0476 (19) | 0.072 (2) | -0.0055 (15) | -0.0129 (17) | -0.0052 (18) |
| C12 | 0.104 (4) | 0.146 (5) | 0.074 (3) | -0.010 (4) | -0.031 (3) | -0.029 (4) |
| C13 | 0.063 (2) | 0.079 (3) | 0.124 (4) | 0.015 (2) | -0.022 (3) | 0.020 (3) |
| O1 | 0.0476 (11) | 0.0263 (9) | 0.0540 (13) | -0.0040 (8) | 0.0070 (11) | -0.0088 (10) |
| O2 | 0.0618 (13) | 0.0404 (11) | 0.0768 (17) | 0.0148 (12) | -0.0035 (13) | -0.0178 (11) |
| O3 | 0.0441 (10) | 0.0430 (11) | 0.0619 (13) | -0.0006 (11) | -0.0005 (11) | 0.0105 (10) |
| O4 | 0.0416 (10) | 0.0596 (13) | 0.0548 (13) | -0.0138 (11) | 0.0053 (11) | -0.0026 (12) |
| P1 | 0.0339 (3) | 0.0299 (3) | 0.0511 (4) | 0.0027 (3) | 0.0014 (3) | -0.0047 (3) |

Geometric parameters (Å, °)

| | | | |
|-----------|-------------|---------------|-----------|
| Br1—C1 | 1.915 (3) | C9—C10 | 1.508 (5) |
| C1—C2 | 1.357 (5) | C9—H9A | 0.9700 |
| C1—C6 | 1.377 (5) | C9—H9B | 0.9700 |
| C2—C3 | 1.377 (4) | C10—C12 | 1.518 (7) |
| C2—H2 | 0.9300 | C10—C11 | 1.520 (5) |
| C3—C4 | 1.382 (5) | C10—C13 | 1.528 (5) |
| C3—H3 | 0.9300 | C11—O4 | 1.449 (4) |
| C4—C5 | 1.393 (5) | C11—H11A | 0.9700 |
| C4—C7 | 1.524 (4) | C11—H11B | 0.9700 |
| C5—C6 | 1.391 (5) | C12—H12A | 0.9600 |
| C5—H5 | 0.9300 | C12—H12B | 0.9600 |
| C6—H6 | 0.9300 | C12—H12C | 0.9600 |
| C7—O1 | 1.434 (4) | C13—H13A | 0.9600 |
| C7—C8 | 1.522 (5) | C13—H13B | 0.9600 |
| C7—P1 | 1.842 (3) | C13—H13C | 0.9600 |
| C8—H8A | 0.9600 | O1—H1 | 0.8200 |
| C8—H8B | 0.9600 | O2—P1 | 1.461 (2) |
| C8—H8C | 0.9600 | O3—P1 | 1.562 (2) |
| C9—O3 | 1.469 (4) | O4—P1 | 1.566 (2) |
| | | | |
| C2—C1—C6 | 121.7 (3) | H9A—C9—H9B | 107.9 |
| C2—C1—Br1 | 118.9 (3) | C9—C10—C12 | 107.9 (4) |
| C6—C1—Br1 | 119.4 (3) | C9—C10—C11 | 108.6 (3) |
| C1—C2—C3 | 119.5 (3) | C12—C10—C11 | 107.8 (4) |
| C1—C2—H2 | 120.3 | C9—C10—C13 | 111.3 (3) |
| C3—C2—H2 | 120.3 | C12—C10—C13 | 111.7 (4) |
| C2—C3—C4 | 121.3 (3) | C11—C10—C13 | 109.6 (3) |
| C2—C3—H3 | 119.4 | O4—C11—C10 | 112.2 (3) |
| C4—C3—H3 | 119.4 | O4—C11—H11A | 109.2 |
| C3—C4—C5 | 118.1 (3) | C10—C11—H11A | 109.2 |
| C3—C4—C7 | 119.2 (3) | O4—C11—H11B | 109.2 |
| C5—C4—C7 | 122.7 (3) | C10—C11—H11B | 109.2 |
| C6—C5—C4 | 120.9 (3) | H11A—C11—H11B | 107.9 |
| C6—C5—H5 | 119.5 | C10—C12—H12A | 109.5 |
| C4—C5—H5 | 119.5 | C10—C12—H12B | 109.5 |
| C1—C6—C5 | 118.4 (3) | H12A—C12—H12B | 109.5 |
| C1—C6—H6 | 120.8 | C10—C12—H12C | 109.5 |
| C5—C6—H6 | 120.8 | H12A—C12—H12C | 109.5 |
| O1—C7—C8 | 111.8 (2) | H12B—C12—H12C | 109.5 |
| O1—C7—C4 | 107.5 (2) | C10—C13—H13A | 109.5 |
| C8—C7—C4 | 112.9 (3) | C10—C13—H13B | 109.5 |
| O1—C7—P1 | 106.92 (19) | H13A—C13—H13B | 109.5 |
| C8—C7—P1 | 109.4 (2) | C10—C13—H13C | 109.5 |
| C4—C7—P1 | 108.11 (18) | H13A—C13—H13C | 109.5 |
| C7—C8—H8A | 109.5 | H13B—C13—H13C | 109.5 |
| C7—C8—H8B | 109.5 | C7—O1—H1 | 109.5 |

| | | | |
|---------------|------------|----------------|-------------|
| H8A—C8—H8B | 109.5 | C9—O3—P1 | 121.6 (2) |
| C7—C8—H8C | 109.5 | C11—O4—P1 | 123.5 (2) |
| H8A—C8—H8C | 109.5 | O2—P1—O3 | 111.41 (14) |
| H8B—C8—H8C | 109.5 | O2—P1—O4 | 112.12 (14) |
| O3—C9—C10 | 112.1 (3) | O3—P1—O4 | 106.72 (12) |
| O3—C9—H9A | 109.2 | O2—P1—C7 | 112.97 (13) |
| C10—C9—H9A | 109.2 | O3—P1—C7 | 107.47 (13) |
| O3—C9—H9B | 109.2 | O4—P1—C7 | 105.74 (13) |
| C10—C9—H9B | 109.2 | | |
| | | | |
| C6—C1—C2—C3 | -2.4 (5) | C12—C10—C11—O4 | -173.3 (4) |
| Br1—C1—C2—C3 | 177.7 (2) | C13—C10—C11—O4 | 65.0 (4) |
| C1—C2—C3—C4 | 1.6 (5) | C10—C9—O3—P1 | -47.2 (4) |
| C2—C3—C4—C5 | -0.3 (4) | C10—C11—O4—P1 | 43.1 (4) |
| C2—C3—C4—C7 | -179.4 (3) | C9—O3—P1—O2 | 150.2 (2) |
| C3—C4—C5—C6 | -0.2 (4) | C9—O3—P1—O4 | 27.5 (3) |
| C7—C4—C5—C6 | 178.8 (3) | C9—O3—P1—C7 | -85.6 (2) |
| C2—C1—C6—C5 | 1.8 (5) | C11—O4—P1—O2 | -148.2 (2) |
| Br1—C1—C6—C5 | -178.3 (2) | C11—O4—P1—O3 | -25.9 (3) |
| C4—C5—C6—C1 | -0.5 (5) | C11—O4—P1—C7 | 88.3 (3) |
| C3—C4—C7—O1 | -28.2 (3) | O1—C7—P1—O2 | 169.99 (19) |
| C5—C4—C7—O1 | 152.7 (3) | C8—C7—P1—O2 | -68.8 (2) |
| C3—C4—C7—C8 | -152.0 (3) | C4—C7—P1—O2 | 54.6 (2) |
| C5—C4—C7—C8 | 29.0 (4) | O1—C7—P1—O3 | 46.7 (2) |
| C3—C4—C7—P1 | 86.8 (3) | C8—C7—P1—O3 | 167.9 (2) |
| C5—C4—C7—P1 | -92.2 (3) | C4—C7—P1—O3 | -68.8 (2) |
| O3—C9—C10—C12 | 175.4 (4) | O1—C7—P1—O4 | -67.0 (2) |
| O3—C9—C10—C11 | 58.9 (4) | C8—C7—P1—O4 | 54.2 (2) |
| O3—C9—C10—C13 | -61.8 (4) | C4—C7—P1—O4 | 177.5 (2) |
| C9—C10—C11—O4 | -56.7 (4) | | |

Hydrogen-bond geometry (Å, °)

| <i>D</i> —H... <i>A</i> | <i>D</i> —H | H... <i>A</i> | <i>D</i> ... <i>A</i> | <i>D</i> —H... <i>A</i> |
|----------------------------|-------------|---------------|-----------------------|-------------------------|
| O1—H1...O2 ⁱ | 0.82 | 2.01 | 2.780 (3) | 156 |
| C9—H9B...O1 | 0.97 | 2.58 | 3.163 (4) | 119 |
| C11—H11A...O2 ⁱ | 0.97 | 2.57 | 3.517 (4) | 165 |

Symmetry code: (i) $-x+1, y+1/2, -z+3/2$.