Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# ($R_P$)-Menthyl (1-hydroxycyclohexyl)-phenylphosphinate

**Baoci Fu and Chang-Qiu Zhao***

College of Chemistry and Chemical Engineering, Liaocheng University, Shandong 252059, People's Republic of China
Correspondence e-mail: literabc@hotmail.com

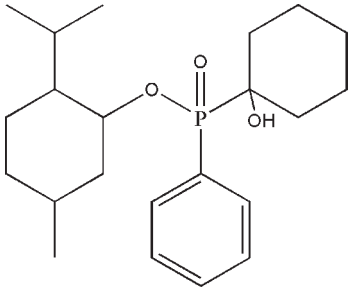
Received 24 December 2009; accepted 11 March 2010

Key indicators: single-crystal X-ray study; $T$ = 298 K; mean $\sigma$(C–C) = 0.004 Å; disorder in main residue; $R$ factor = 0.039; $wR$ factor = 0.096; data-to-parameter ratio = 14.4.

The title compound, $C_{22}H_{35}O_3P$, features a tetrahedral P atom bonded to a phenyl ring, a hydroxycyclohexyl unit and the O atom of a menthyl group. The axial chirality at phosphorus is $R_P$. In the crystal, molecules are connected through O—H···O hydrogen bonds involving the hydroxy and P═O groups, forming chains along the $2_1$ screw axis. The methyl groups of the isopropyl fragment in the menthyl unit are disordered over two sites of equal occupancy.

## Related literature

For general background to $\alpha$-hydroxy alkylphosphonates, see: Kim & Wiemer (2003). For the structures of related phenylphosphinates, see: Sheldrick *et al.* (1981); Chaloner *et al.* (1991); Grice *et al.* (2004).




## Experimental

### Crystal data

$C_{22}H_{35}O_3P$
$M_r$ = 378.47
Monoclinic, $P2_1$
$a$ = 10.1808 (11) Å
$b$ = 11.0611 (13) Å
$c$ = 10.4207 (12) Å
$\beta$ = 106.201 (1)°
$V$ = 1126.9 (2) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.14 mm$^{-1}$
$T$ = 298 K
0.42 × 0.32 × 0.26 mm

### Data collection

Siemens SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.944, $T_{max}$ = 0.965
5667 measured reflections
3787 independent reflections
3248 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.019

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.096
$S$ = 1.06
3787 reflections
263 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.16 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.24 e Å$^{-3}$
Absolute structure: Flack (1983), 1685 Friedel pairs
Flack parameter: 0.14 (10)

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O3—H3···O2$^{i}$ | 0.76 (3) | 1.94 (3) | 2.695 (3) | 170 (3) |

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

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

We acknowledge the financial support of the Natural Science Foundation of China (No. 20772055).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BH2267).

## References

Chaloner, P. A., Harrison, R. M. & Hitchcock, P. B. (1991). *Acta Cryst.* C**47**, 2241–2242.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Grice, I. D., Jenkins, I. D., Busfield, W. K., Byriel, K. A. & Kennard, C. H. L. (2004). *Acta Cryst.* E**60**, o2384–o2385.
Kim, D. Y. & Wiemer, D. F. (2003). *Tetrahedron Lett.* **44**, 2803–2805.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, W. S., Hagele, G. & Kuckelhaus, W. (1981). *J. Mol. Struct.* **74**, 331–341.
Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

# supplementary materials

*Acta Cryst.* (2010). E**66**, o859 [ **doi:10.1107/S1600536810009219** ]

# (*R*P)-Menthyl (1-hydroxycyclohexyl)phenylphosphinate

**B. Fu and C.-Q. Zhao**

## Comment

α-Hydroxy alkylphosphonates have received attention both as substrates for the preparation of other α-substituted phosphonates, and because of their potential biological activity. For example, representatives of this class act as inhibitors of farnesyl protein transferase (FPTase), renin, and HIV protease (Kim & Wiemer, 2003).

The P-chiral title compound, which can be synthesized by addition of (*R*P)-menthyl-phenylphosphinate to cyclohexanone (see *Experimental*), is comprised of fully extended substituents: phenyl, menthyl and α-hydroxycyclohexyl. The configuration of the central P atom is *R* and the four groups around the P atom form an irregular tetrahedron as found in *tert*-butyl diphenylphosphinate (Grice *et al.*, 2004). The bond angles are C1—P—C17 = 107.13 (11)°, O1—P—C1 = 107.06 (10)°, O1—P—C17 = 102.87 (9)°, O2—P—O1 = 113.87 (9)°, O2—P—C17 = 113.18 (11)° and O2—P—C1 = 112.05 (11)°, which compare with angles observed in related phenylphosphinate derivatives bearing a menthyl group (Chaloner *et al.*, 1991; Sheldrick *et al.*, 1981). Part of methyl groups (C14 and C15) were found to be disordered over two sites with equal occupancies.

Intramolecular O3—H3···O2 hydrogen bonds are found in the crystal structure. The crystal packing is further stabilized by van der Waals interactions.

## Experimental

Cyclohexanone was added to a stirred DMF solution of (*R*P)-menthyl-phenylphosphinate in a flask and the mixture was stirred for 48 h at room temperature. After washing with water, the resulting solid was dried, and recrystallized from diethyl ether, to afford the pure title product.

## Refinement

Atoms C14 and C15 were found to be disordered over two sites, and the ratio of the occupancy factors was fixed to 0.50:0.50 and 0.50:0.50 for atoms C14:C14'and C15:C15', respectively. All H atoms attached to C atoms were fixed geometrically and treated as riding with C—H = 0.93–0.98 Å, and with $U_{iso}$(H) = 1.2 $U_{eq}$(C) or 1.5 $U_{eq}$(C) for the methyl groups. Atom H3 was found in a difference map and refined with free coordinates, converging to O—H = 0.76 (3) Å. Assignment of absolute configuration is based on measurement of 1685 Friedel pairs.

## Figures

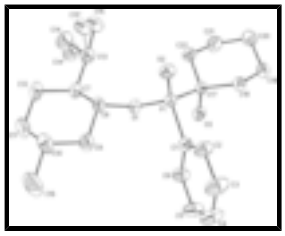


Fig. 1. The molecular structure of the title compound. H atoms have been omitted for clarity. Primed atoms C14' and C15' are disordered with C14 and C15, respectively.

## (*R*P)-Menthyl (1-hydroxycyclohexyl)phenylphosphinate

*Crystal data*

| | |
|---|---|
| $C_{22}H_{35}O_3P$ | $F(000) = 412$ |
| $M_r = 378.47$ | $D_x = 1.115$ Mg m$^{-3}$ |
| Monoclinic, $P2_1$ | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| Hall symbol: P 2yb | Cell parameters from 2598 reflections |
| $a = 10.1808$ (11) Å | $\theta = 2.5$–$24.8°$ |
| $b = 11.0611$ (13) Å | $\mu = 0.14$ mm$^{-1}$ |
| $c = 10.4207$ (12) Å | $T = 298$ K |
| $\beta = 106.201$ (1)° | Block, colorless |
| $V = 1126.9$ (2) Å$^3$ | 0.42 × 0.32 × 0.26 mm |
| $Z = 2$ | |

*Data collection*

| | |
|---|---|
| Siemens SMART CCD area-detector diffractometer | 3787 independent reflections |
| Radiation source: fine-focus sealed tube | 3248 reflections with $I > 2\sigma(I)$ |
| graphite | $R_{int} = 0.019$ |
| φ and ω scans | $\theta_{max} = 25.0°$, $\theta_{min} = 2.0°$ |
| Absorption correction: multi-scan (SADABS; Sheldrick, 1996) | $h = -8 \rightarrow 12$ |
| $T_{min} = 0.944$, $T_{max} = 0.965$ | $k = -13 \rightarrow 13$ |
| 5667 measured reflections | $l = -12 \rightarrow 10$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.039$ | H atoms treated by a mixture of independent and constrained refinement |
| $wR(F^2) = 0.096$ | $w = 1/[\sigma^2(F_o^2) + (0.0437P)^2 + 0.1698P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 1.06$ | $(\Delta/\sigma)_{max} = 0.001$ |

| | |
|---|---|
| 3787 reflections | $\Delta\rho_{max}$ = 0.16 e Å$^{-3}$ |
| 263 parameters | $\Delta\rho_{min}$ = −0.24 e Å$^{-3}$ |
| 1 restraint | Absolute structure: Flack (1983), 1685 Friedel pairs |
| 0 constraints | Flack parameter: 0.14 (10) |
| Primary atom site location: structure-invariant direct methods | |

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| O1 | 0.95710 (16) | 0.72151 (14) | 0.74639 (14) | 0.0404 (4) | |
| O2 | 0.97738 (18) | 0.87366 (15) | 0.56708 (17) | 0.0462 (4) | |
| O3 | 0.8831 (2) | 0.52789 (16) | 0.54628 (18) | 0.0454 (5) | |
| H3 | 0.930 (3) | 0.488 (3) | 0.520 (3) | 0.056 (10)* | |
| P1 | 0.99001 (6) | 0.74570 (6) | 0.60864 (6) | 0.03512 (16) | |
| C1 | 1.1586 (3) | 0.6871 (2) | 0.6245 (2) | 0.0410 (6) | |
| C2 | 1.2450 (3) | 0.7469 (3) | 0.5653 (3) | 0.0561 (7) | |
| H2 | 1.2159 | 0.8181 | 0.5184 | 0.067* | |
| C3 | 1.3736 (4) | 0.7031 (3) | 0.5743 (4) | 0.0805 (11) | |
| H3A | 1.4317 | 0.7456 | 0.5359 | 0.097* | |
| C4 | 1.4161 (3) | 0.5965 (4) | 0.6402 (4) | 0.0815 (11) | |
| H4 | 1.5020 | 0.5657 | 0.6438 | 0.098* | |
| C5 | 1.3327 (3) | 0.5355 (3) | 0.7005 (4) | 0.0720 (9) | |
| H5 | 1.3626 | 0.4641 | 0.7465 | 0.086* | |
| C6 | 1.2035 (3) | 0.5802 (3) | 0.6929 (3) | 0.0551 (7) | |
| H6 | 1.1466 | 0.5386 | 0.7336 | 0.066* | |
| C7 | 0.8846 (3) | 0.8021 (3) | 0.9295 (3) | 0.0561 (7) | |
| H7 | 0.8856 | 0.7192 | 0.9628 | 0.067* | |
| C8 | 0.9937 (3) | 0.8099 (3) | 0.8559 (2) | 0.0452 (6) | |
| H8 | 0.9926 | 0.8912 | 0.8181 | 0.054* | |
| C9 | 1.1352 (3) | 0.7835 (3) | 0.9449 (3) | 0.0571 (8) | |
| H9A | 1.2009 | 0.7927 | 0.8937 | 0.068* | |
| H9B | 1.1391 | 0.7004 | 0.9752 | 0.068* | |
| C10 | 1.1745 (3) | 0.8673 (3) | 1.0659 (3) | 0.0697 (9) | |
| H10 | 1.1755 | 0.9504 | 1.0336 | 0.084* | |
| C11 | 1.0682 (4) | 0.8595 (4) | 1.1413 (3) | 0.0788 (10) | |
| H11A | 1.0902 | 0.9170 | 1.2145 | 0.095* | |
| H11B | 1.0699 | 0.7791 | 1.1791 | 0.095* | |
| C12 | 0.9261 (4) | 0.8856 (4) | 1.0522 (3) | 0.0797 (11) | |
| H12A | 0.9222 | 0.9689 | 1.0222 | 0.096* | |
| H12B | 0.8608 | 0.8763 | 1.1037 | 0.096* | |
| C13 | 0.7400 (3) | 0.8263 (4) | 0.8383 (3) | 0.0776 (11) | |
| H13 | 0.7166 | 0.7564 | 0.7786 | 0.093* | |
| C14 | 0.627 (3) | 0.8350 (16) | 0.911 (3) | 0.095 (5) | 0.50 |
| H14A | 0.6335 | 0.9114 | 0.9559 | 0.143* | 0.50 |
| H14B | 0.6381 | 0.7707 | 0.9749 | 0.143* | 0.50 |
| H14C | 0.5388 | 0.8282 | 0.8467 | 0.143* | 0.50 |
| C15 | 0.714 (4) | 0.938 (4) | 0.748 (3) | 0.108 (6) | 0.50 |

| | | | | | |
|---|---|---|---|---|---|
| H15A | 0.7745 | 0.9366 | 0.6920 | 0.162* | 0.50 |
| H15B | 0.7302 | 1.0094 | 0.8022 | 0.162* | 0.50 |
| H15C | 0.6209 | 0.9371 | 0.6934 | 0.162* | 0.50 |
| C14' | 0.635 (3) | 0.7783 (17) | 0.912 (3) | 0.095 (5) | 0.50 |
| H14D | 0.6474 | 0.8216 | 0.9944 | 0.143* | 0.50 |
| H14E | 0.6507 | 0.6936 | 0.9306 | 0.143* | 0.50 |
| H14F | 0.5438 | 0.7903 | 0.8562 | 0.143* | 0.50 |
| C15' | 0.735 (4) | 0.959 (4) | 0.789 (3) | 0.108 (6) | 0.50 |
| H15D | 0.7474 | 1.0123 | 0.8642 | 0.162* | 0.50 |
| H15E | 0.6473 | 0.9740 | 0.7266 | 0.162* | 0.50 |
| H15R | 0.8057 | 0.9717 | 0.7467 | 0.162* | 0.50 |
| C16 | 1.3180 (4) | 0.8374 (5) | 1.1542 (4) | 0.1122 (16) | |
| H16A | 1.3206 | 0.7549 | 1.1833 | 0.168* | |
| H16B | 1.3403 | 0.8899 | 1.2306 | 0.168* | |
| H16C | 1.3831 | 0.8487 | 1.1040 | 0.168* | |
| C17 | 0.8687 (2) | 0.6464 (2) | 0.4925 (2) | 0.0352 (5) | |
| C18 | 0.8980 (3) | 0.6531 (2) | 0.3562 (2) | 0.0448 (6) | |
| H18A | 0.8993 | 0.7372 | 0.3301 | 0.054* | |
| H18B | 0.9877 | 0.6192 | 0.3638 | 0.054* | |
| C19 | 0.7919 (3) | 0.5853 (3) | 0.2483 (3) | 0.0588 (8) | |
| H19A | 0.8104 | 0.5973 | 0.1628 | 0.071* | |
| H19B | 0.7987 | 0.4995 | 0.2681 | 0.071* | |
| C20 | 0.6479 (3) | 0.6285 (3) | 0.2392 (3) | 0.0709 (9) | |
| H20A | 0.6380 | 0.7123 | 0.2105 | 0.085* | |
| H20B | 0.5823 | 0.5807 | 0.1733 | 0.085* | |
| C21 | 0.6187 (3) | 0.6171 (3) | 0.3728 (3) | 0.0657 (9) | |
| H21A | 0.6223 | 0.5326 | 0.3983 | 0.079* | |
| H21B | 0.5272 | 0.6467 | 0.3654 | 0.079* | |
| C22 | 0.7219 (3) | 0.6887 (3) | 0.4808 (3) | 0.0492 (7) | |
| H22A | 0.7022 | 0.6777 | 0.5660 | 0.059* | |
| H22B | 0.7136 | 0.7741 | 0.4590 | 0.059* | |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0456 (9) | 0.0393 (11) | 0.0388 (8) | −0.0032 (8) | 0.0161 (7) | −0.0047 (7) |
| O2 | 0.0508 (11) | 0.0335 (10) | 0.0567 (10) | −0.0007 (8) | 0.0190 (8) | 0.0038 (8) |
| O3 | 0.0580 (12) | 0.0339 (10) | 0.0494 (11) | −0.0026 (9) | 0.0233 (9) | −0.0016 (8) |
| P1 | 0.0363 (3) | 0.0318 (3) | 0.0396 (3) | −0.0007 (3) | 0.0144 (2) | 0.0000 (3) |
| C1 | 0.0396 (14) | 0.0410 (14) | 0.0434 (14) | 0.0005 (11) | 0.0132 (11) | −0.0020 (12) |
| C2 | 0.0513 (15) | 0.0501 (15) | 0.0746 (17) | 0.0069 (17) | 0.0305 (13) | 0.0119 (19) |
| C3 | 0.058 (2) | 0.078 (3) | 0.119 (3) | 0.0097 (17) | 0.048 (2) | 0.018 (2) |
| C4 | 0.0510 (19) | 0.082 (3) | 0.119 (3) | 0.0225 (19) | 0.035 (2) | 0.012 (2) |
| C5 | 0.059 (2) | 0.063 (2) | 0.093 (2) | 0.0203 (17) | 0.0181 (18) | 0.0197 (18) |
| C6 | 0.0487 (17) | 0.0538 (18) | 0.0643 (18) | 0.0038 (14) | 0.0184 (14) | 0.0140 (15) |
| C7 | 0.0615 (18) | 0.0665 (18) | 0.0429 (15) | 0.0104 (15) | 0.0191 (14) | −0.0072 (13) |
| C8 | 0.0591 (17) | 0.0389 (14) | 0.0371 (14) | −0.0023 (14) | 0.0128 (13) | −0.0048 (12) |
| C9 | 0.0543 (17) | 0.065 (2) | 0.0514 (16) | −0.0048 (13) | 0.0134 (13) | −0.0063 (13) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C10 | 0.082 (2) | 0.069 (2) | 0.0504 (17) | −0.0200 (18) | 0.0051 (16) | −0.0067 (16) |
| C11 | 0.094 (3) | 0.092 (3) | 0.0444 (17) | −0.004 (2) | 0.0104 (18) | −0.0162 (18) |
| C12 | 0.095 (3) | 0.096 (3) | 0.0519 (18) | 0.014 (2) | 0.0266 (18) | −0.0245 (19) |
| C13 | 0.062 (2) | 0.114 (3) | 0.0585 (19) | 0.029 (2) | 0.0188 (17) | −0.015 (2) |
| C14 | 0.067 (7) | 0.137 (16) | 0.090 (7) | 0.030 (11) | 0.034 (5) | −0.024 (13) |
| C15 | 0.099 (12) | 0.131 (17) | 0.072 (14) | 0.055 (11) | −0.012 (11) | −0.015 (12) |
| C14' | 0.067 (6) | 0.137 (16) | 0.089 (7) | 0.030 (12) | 0.034 (5) | −0.024 (13) |
| C15' | 0.099 (12) | 0.130 (17) | 0.072 (14) | 0.055 (11) | −0.012 (11) | −0.014 (12) |
| C16 | 0.089 (3) | 0.148 (4) | 0.078 (3) | −0.027 (3) | −0.011 (2) | −0.026 (3) |
| C17 | 0.0406 (14) | 0.0327 (13) | 0.0352 (12) | −0.0009 (10) | 0.0155 (11) | −0.0018 (10) |
| C18 | 0.0491 (15) | 0.0478 (16) | 0.0400 (13) | 0.0045 (12) | 0.0163 (12) | 0.0001 (12) |
| C19 | 0.069 (2) | 0.0644 (19) | 0.0406 (15) | 0.0064 (16) | 0.0124 (14) | −0.0084 (14) |
| C20 | 0.061 (2) | 0.085 (2) | 0.0560 (18) | 0.0042 (17) | −0.0025 (15) | −0.0180 (16) |
| C21 | 0.0420 (16) | 0.081 (2) | 0.070 (2) | −0.0046 (15) | 0.0094 (15) | −0.0206 (17) |
| C22 | 0.0413 (15) | 0.0544 (17) | 0.0514 (15) | 0.0028 (13) | 0.0121 (12) | −0.0086 (13) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| O1—C8 | 1.469 (3) | C13—C15' | 1.55 (5) |
| O1—P1 | 1.5853 (16) | C13—C14 | 1.55 (3) |
| O2—P1 | 1.4752 (18) | C13—C14' | 1.57 (3) |
| O3—C17 | 1.417 (3) | C13—H13 | 0.9800 |
| O3—H3 | 0.76 (3) | C14—H14A | 0.9600 |
| P1—C1 | 1.799 (3) | C14—H14B | 0.9600 |
| P1—C17 | 1.834 (2) | C14—H14C | 0.9600 |
| C1—C2 | 1.376 (4) | C15—H15A | 0.9600 |
| C1—C6 | 1.390 (4) | C15—H15B | 0.9600 |
| C2—C3 | 1.375 (4) | C15—H15C | 0.9600 |
| C2—H2 | 0.9300 | C14'—H14D | 0.9600 |
| C3—C4 | 1.371 (5) | C14'—H14E | 0.9600 |
| C3—H3A | 0.9300 | C14'—H14F | 0.9600 |
| C4—C5 | 1.367 (5) | C15'—H15D | 0.9600 |
| C4—H4 | 0.9300 | C15'—H15E | 0.9600 |
| C5—C6 | 1.387 (4) | C15'—H15R | 0.9600 |
| C5—H5 | 0.9300 | C16—H16A | 0.9600 |
| C6—H6 | 0.9300 | C16—H16B | 0.9600 |
| C7—C8 | 1.518 (4) | C16—H16C | 0.9600 |
| C7—C13 | 1.537 (4) | C17—C18 | 1.533 (3) |
| C7—C12 | 1.537 (4) | C17—C22 | 1.538 (3) |
| C7—H7 | 0.9800 | C18—C19 | 1.522 (4) |
| C8—C9 | 1.508 (4) | C18—H18A | 0.9700 |
| C8—H8 | 0.9800 | C18—H18B | 0.9700 |
| C9—C10 | 1.526 (4) | C19—C20 | 1.520 (4) |
| C9—H9A | 0.9700 | C19—H19A | 0.9700 |
| C9—H9B | 0.9700 | C19—H19B | 0.9700 |
| C10—C11 | 1.507 (5) | C20—C21 | 1.507 (4) |
| C10—C16 | 1.529 (5) | C20—H20A | 0.9700 |
| C10—H10 | 0.9800 | C20—H20B | 0.9700 |
| C11—C12 | 1.513 (5) | C21—C22 | 1.529 (4) |

| | | | |
|---|---|---|---|
| C11—H11A | 0.9700 | C21—H21A | 0.9700 |
| C11—H11B | 0.9700 | C21—H21B | 0.9700 |
| C12—H12A | 0.9700 | C22—H22A | 0.9700 |
| C12—H12B | 0.9700 | C22—H22B | 0.9700 |
| C13—C15 | 1.53 (5) | | |
| C8—O1—P1 | 121.25 (15) | C7—C13—C14' | 107.6 (10) |
| C17—O3—H3 | 114 (2) | C15'—C13—C14' | 121.0 (17) |
| O2—P1—O1 | 113.87 (9) | C15—C13—H13 | 106.1 |
| O2—P1—C1 | 112.05 (11) | C7—C13—H13 | 106.1 |
| O1—P1—C1 | 107.06 (10) | C14—C13—H13 | 106.1 |
| O2—P1—C17 | 113.18 (11) | C13—C14—H14A | 109.5 |
| O1—P1—C17 | 102.87 (9) | C13—C14—H14B | 109.5 |
| C1—P1—C17 | 107.13 (11) | C13—C14—H14C | 109.5 |
| C2—C1—C6 | 118.6 (2) | C13—C15—H15A | 109.5 |
| C2—C1—P1 | 119.9 (2) | C13—C15—H15B | 109.5 |
| C6—C1—P1 | 121.5 (2) | C13—C15—H15C | 109.5 |
| C3—C2—C1 | 121.0 (3) | C13—C14'—H14D | 109.5 |
| C3—C2—H2 | 119.5 | C13—C14'—H14E | 109.5 |
| C1—C2—H2 | 119.5 | H14D—C14'—H14E | 109.5 |
| C4—C3—C2 | 119.9 (3) | C13—C14'—H14F | 109.5 |
| C4—C3—H3A | 120.0 | H14D—C14'—H14F | 109.5 |
| C2—C3—H3A | 120.0 | H14E—C14'—H14F | 109.5 |
| C5—C4—C3 | 120.3 (3) | C13—C15'—H15D | 109.5 |
| C5—C4—H4 | 119.8 | C13—C15'—H15E | 109.5 |
| C3—C4—H4 | 119.8 | H15D—C15'—H15E | 109.5 |
| C4—C5—C6 | 119.8 (3) | C13—C15'—H15R | 109.5 |
| C4—C5—H5 | 120.1 | H15D—C15'—H15R | 109.5 |
| C6—C5—H5 | 120.1 | H15E—C15'—H15R | 109.5 |
| C5—C6—C1 | 120.2 (3) | C10—C16—H16A | 109.5 |
| C5—C6—H6 | 119.9 | C10—C16—H16B | 109.5 |
| C1—C6—H6 | 119.9 | H16A—C16—H16B | 109.5 |
| C8—C7—C13 | 112.8 (2) | C10—C16—H16C | 109.5 |
| C8—C7—C12 | 108.4 (3) | H16A—C16—H16C | 109.5 |
| C13—C7—C12 | 113.8 (3) | H16B—C16—H16C | 109.5 |
| C8—C7—H7 | 107.2 | O3—C17—C18 | 112.73 (19) |
| C13—C7—H7 | 107.2 | O3—C17—C22 | 107.7 (2) |
| C12—C7—H7 | 107.2 | C18—C17—C22 | 110.4 (2) |
| O1—C8—C9 | 109.9 (2) | O3—C17—P1 | 108.49 (16) |
| O1—C8—C7 | 107.0 (2) | C18—C17—P1 | 108.25 (17) |
| C9—C8—C7 | 112.7 (2) | C22—C17—P1 | 109.30 (16) |
| O1—C8—H8 | 109.1 | C19—C18—C17 | 112.3 (2) |
| C9—C8—H8 | 109.1 | C19—C18—H18A | 109.1 |
| C7—C8—H8 | 109.1 | C17—C18—H18A | 109.1 |
| C8—C9—C10 | 112.1 (2) | C19—C18—H18B | 109.1 |
| C8—C9—H9A | 109.2 | C17—C18—H18B | 109.1 |
| C10—C9—H9A | 109.2 | H18A—C18—H18B | 107.9 |
| C8—C9—H9B | 109.2 | C20—C19—C18 | 111.4 (2) |
| C10—C9—H9B | 109.2 | C20—C19—H19A | 109.3 |
| H9A—C9—H9B | 107.9 | C18—C19—H19A | 109.3 |

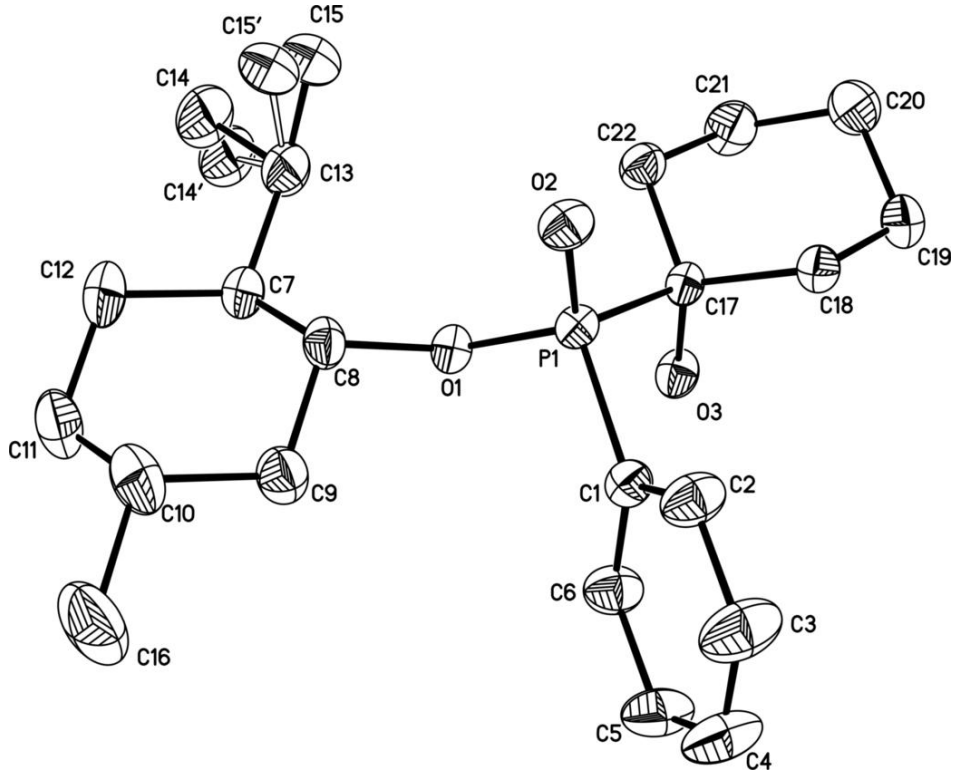
| | | | |
|---|---|---|---|
| C11—C10—C9 | 109.6 (3) | C20—C19—H19B | 109.3 |
| C11—C10—C16 | 112.2 (3) | C18—C19—H19B | 109.3 |
| C9—C10—C16 | 110.6 (3) | H19A—C19—H19B | 108.0 |
| C11—C10—H10 | 108.1 | C21—C20—C19 | 110.6 (3) |
| C9—C10—H10 | 108.1 | C21—C20—H20A | 109.5 |
| C16—C10—H10 | 108.1 | C19—C20—H20A | 109.5 |
| C10—C11—C12 | 111.8 (3) | C21—C20—H20B | 109.5 |
| C10—C11—H11A | 109.3 | C19—C20—H20B | 109.5 |
| C12—C11—H11A | 109.3 | H20A—C20—H20B | 108.1 |
| C10—C11—H11B | 109.3 | C20—C21—C22 | 111.4 (3) |
| C12—C11—H11B | 109.3 | C20—C21—H21A | 109.3 |
| H11A—C11—H11B | 107.9 | C22—C21—H21A | 109.3 |
| C11—C12—C7 | 113.0 (3) | C20—C21—H21B | 109.3 |
| C11—C12—H12A | 109.0 | C22—C21—H21B | 109.3 |
| C7—C12—H12A | 109.0 | H21A—C21—H21B | 108.0 |
| C11—C12—H12B | 109.0 | C21—C22—C17 | 110.7 (2) |
| C7—C12—H12B | 109.0 | C21—C22—H22A | 109.5 |
| H12A—C12—H12B | 107.8 | C17—C22—H22A | 109.5 |
| C15—C13—C7 | 119.6 (14) | C21—C22—H22B | 109.5 |
| C7—C13—C15' | 108.0 (13) | C17—C22—H22B | 109.5 |
| C15—C13—C14 | 103.0 (17) | H22A—C22—H22B | 108.1 |
| C7—C13—C14 | 115.1 (11) | | |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O3—H3···O2$^{i}$ | 0.76 (3) | 1.94 (3) | 2.695 (3) | 170 (3) |

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

**Fig. 1**


C15′
C15
C14
C14′
C13
C12
C7
C8
O1
O2
P1
C17
C18
C19
C20
C21
C22
O3
C11
C10
C9
C1
C2
C6
C3
C5
C4
C16