

4-hydroxymethyl-1-(2',3',4',6'-tetra-O-acetyl- β -D-glucopyranosyl)-1,2,3-triazole.

Author

Wilkinson, Brendan L, Bornaghi, Laurent F, Houston, Todd A, Poulsen, Sally-Ann, White, Alan R

Published

2006

Journal Title

Acta crystallographica. Section E, Structure reports online

DOI

[10.1107/S1600536806040864](https://doi.org/10.1107/S1600536806040864)

Rights statement

© The Author(s) 2006. For information about this journal please refer to the journal's website. All articles published in Acta Crystallographica Section E are open access and distributed under the Creative Commons Attribution License, which permits unrestricted use, distribution, and reproduction in any medium, provided the original work is properly cited. See <http://creativecommons.org/licenses/by/2.0/uk/legalcode>

Downloaded from

<http://hdl.handle.net/10072/14355>

Link to published version

<http://journals.iucr.org/e/journalhomepage.html>

Griffith Research Online

<https://research-repository.griffith.edu.au>

Na Zuo and Hong-Wu He*

Key Laboratory of Pesticides and Chemical
Biology, College of Chemistry, Central China
Normal University, Wuhan 430079, People's
Republic of China.Correspondence e-mail:
he1208@mail.ccnu.edu.cn

Key indicators

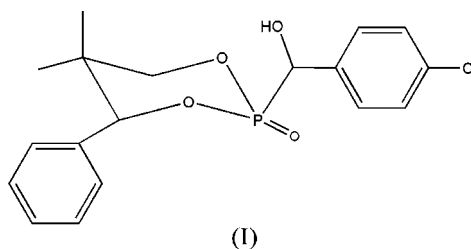
Single-crystal X-ray study
 $T = 292$ K
Mean $\sigma(\text{C}-\text{C}) = 0.006$ Å
 R factor = 0.054
 wR factor = 0.112
Data-to-parameter ratio = 17.3For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.2-[(4-Chlorophenyl)hydroxymethyl]-5,5-dimethyl-
4-phenyl-1,3,2-dioxaphosphorinan-2-one

In the title compound, $\text{C}_{18}\text{H}_{20}\text{ClO}_4\text{P}$, the P atom is in a distorted tetrahedral configuration. In the crystal structure, molecules are linked by intermolecular $\text{C}-\text{H}\cdots\text{O}$ and $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds.

Received 15 September 2006
Accepted 28 September 2006

Comment

Some hydroxyphosphonates have shown good biological and pharmaceutical activities (Chen *et al.*, 1995). In continuation of our work on the synthesis of phosphonic acid derivatives with good biological activities (He *et al.*, 2005), a number of hydroxyphosphonates have been synthesized, and we report here the crystal structure of the title compound, (I).



The dioxaphosphorinane ring in the molecule adopts a chair conformation (Fig. 1). Selected bond lengths and angles are listed in Table 1. The $\text{P1}=\text{O3}$, $\text{P1}-\text{O1}$ and $\text{P1}-\text{O2}$ bond lengths are similar to those observed in a related structure, *viz.* 2-benzylamino-5,5-dimethyl-4-phenyl-1,3,2-dioxaphosphorinane 2-oxide [1.4650 (13), 1.5814 (11) and 1.5888 (12) Å, respectively; Wan *et al.*, 2005]. The bond angles around P range from 106.1 (1) to 112.3 (1)°, indicating a distorted tetrahedral configuration for the P atom.

The crystal packing is stabilized by $\text{C}-\text{H}\cdots\text{O}$ and $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds (Table 2 and Fig. 2).

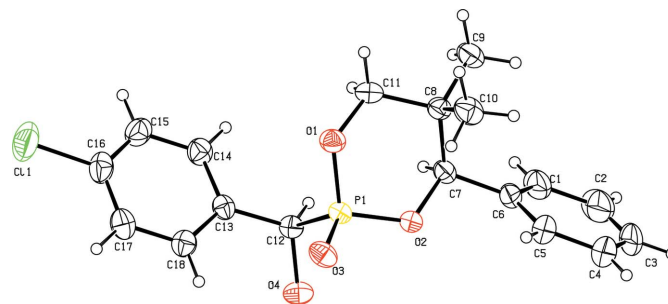


Figure 1
View of (I), showing the labeling scheme with 50% probability displacement ellipsoids.

Experimental

5,5-Dimethyl-4-phenyl-1,3,2-dioxaphosphorinan-2-one was prepared according to a literature procedure (Ten Hoeve & Wynberg, 1985). To a mixture of 5,5-dimethyl-4-phenyl-1,3,2-dioxaphosphorinan-2-one (5 mmol), 4-chlorobenzaldehyde (5 mmol) and toluene (10 ml) was added triethylamine (5 mmol). The flask was cooled in an ice-bath, and the mixture was stirred for 1 h at room temperature. The white solid was filtered, washed with toluene (5 ml) and recrystallized ($\text{CH}_2\text{Cl}_2/\text{toluene}$, 1:1) to give the title compound. Suitable crystals were obtained by vapor diffusion of ethanol into a dimethylformamide solution at room temperature (m.p. 499 K).

Crystal data

$\text{C}_{18}\text{H}_{20}\text{ClO}_4\text{P}$	$Z = 2$
$M_r = 366.76$	$D_x = 1.353 \text{ Mg m}^{-3}$
Monoclinic, $P2_1$	Mo $K\alpha$ radiation
$a = 10.5609 (14) \text{ \AA}$	$\mu = 0.32 \text{ mm}^{-1}$
$b = 6.4450 (9) \text{ \AA}$	$T = 292 (2) \text{ K}$
$c = 14.1161 (19) \text{ \AA}$	Needle, colorless
$\beta = 110.475 (2)^\circ$	$0.30 \times 0.10 \times 0.05 \text{ mm}$
$V = 900.1 (2) \text{ \AA}^3$	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	3837 independent reflections
φ and ω scans	2940 reflections with $I > 2\sigma(I)$
Absorption correction: none	$R_{\text{int}} = 0.032$
6763 measured reflections	$\theta_{\text{max}} = 27.0^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0477P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.054$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.112$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 0.99$	$\Delta\rho_{\text{max}} = 0.30 \text{ e \AA}^{-3}$
3837 reflections	$\Delta\rho_{\text{min}} = -0.23 \text{ e \AA}^{-3}$
222 parameters	Absolute structure: Flack (1983),
H atoms treated by a mixture of independent and constrained refinement	1711 Friedel pairs
	Flack parameter: 0.11 (9)

Table 1

Selected geometric parameters (\AA , $^\circ$).

C12—P1	1.817 (3)	O2—P1	1.568 (2)
O1—P1	1.564 (2)	O3—P1	1.466 (2)
O3—P1—O1	111.83 (13)	O3—P1—C12	112.29 (14)
O3—P1—O2	111.64 (13)	O1—P1—C12	107.05 (13)
O1—P1—O2	106.09 (11)	O2—P1—C12	107.60 (13)

Table 2

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

$D\cdots A$	$D\cdots H$	$H\cdots A$	$D\cdots A$	$D\cdots H\cdots A$
C11—H11A \cdots O1 ⁱ	0.97	2.57	3.290 (4)	131
C12—H12 \cdots O4 ⁱⁱ	0.98	2.29	3.126 (4)	143
C9—H9A \cdots O3 ⁱⁱⁱ	0.96	2.52	3.439 (4)	159
O4—H4A \cdots O3 ⁱⁱ	0.814 (10)	1.913 (16)	2.696 (3)	161 (4)

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

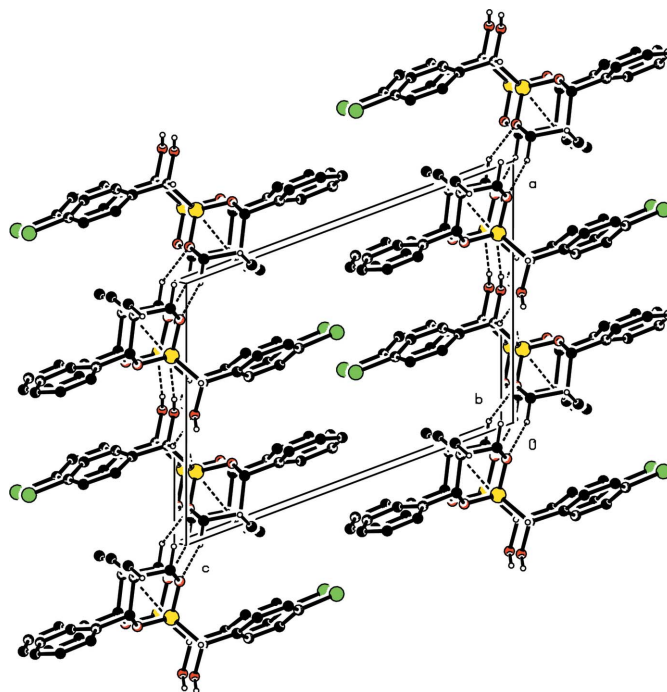


Figure 2

The molecular structure of (I), showing the C—H \cdots O and O—H \cdots O hydrogen bonds (dashed lines). H atoms not involved in these interactions have been omitted.

The hydroxyl H atom was located in a difference map and refined with the restraint O—H = 0.82 (1) \AA ; $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$. All the carbon-bound H atoms were positioned geometrically and treated as riding on their parent atoms; C—H = 0.93–0.98 \AA , $U_{\text{iso}}(\text{H}) = xU_{\text{eq}}(\text{C})$, where $x = 1.5$ for methyl H atoms and 1.2 for the remaining H atoms.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

We gratefully acknowledge the financial support of this work by the National Basic Research Program of China (2003CB114400) and the National Natural Science Foundation of China (No. 20372023).

References

- Bruker (1997). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
- Bruker (2000). *SMART* (Version 5.059) and *SAINT* (Version 6.01). Bruker AXS Inc., Madison, Wisconsin, USA.
- Chen, R. Y., Mao, L. J. & Zhang, M. (1995). *Sci. China B*, **25**, 240–246.
- Flack, H. D. (1983). *Acta Cryst. A* **39**, 876–881.
- He, H. W., Wang, T. & Yuan, J. L. (2005). *J. Organomet. Chem.* **690**, 2608–2613.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Ten Hoeve, W. & Wynberg, H. (1985). *J. Org. Chem.* **50**, 4508–4514.
- Wan, S.-G., Liu, C., Yu, Y. & Yang, X.-Y. (2005). *Acta Cryst. E* **61**, o2117–o2119.