

Acta Crystallographica Section E

## Structure Reports

Online

ISSN 1600-5368

## 5,5-Bis(hydroxymethyl)-2-phenyl-1,3-dioxane

Yi-Ming Luo,<sup>a</sup> Xing-Ming Liu,<sup>a</sup> Xian-You Yuan,<sup>b</sup> Min Zhang<sup>b</sup> and Seik Weng Ng<sup>c\*</sup>

<sup>a</sup>School of Chemistry and Chemical Engineering, Central South University, Changsha 410083, People's Republic of China, <sup>b</sup>Department of Biology and Chemistry, Hunan University of Science and Engineering, Yongzhou 425100, People's Republic of China, and <sup>c</sup>Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia

Correspondence e-mail: seikweng@um.edu.my

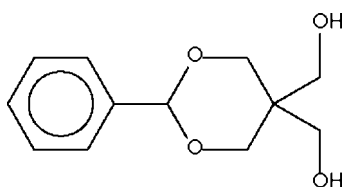
Received 10 July 2008; accepted 15 July 2008

Key indicators: single-crystal X-ray study;  $T = 173$  K; mean  $\sigma(\text{C}-\text{C}) = 0.007$  Å;  $R$  factor = 0.067;  $wR$  factor = 0.185; data-to-parameter ratio = 9.7.

In the title compound,  $\text{C}_{12}\text{H}_{16}\text{O}_4$ , the 1,3-dioxane ring adopts a chair conformation; the 2-phenyl substituent occupies an equatorial position. Adjacent molecules are linked by  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonds into a chain.

## Related literature

For the crystal structures of similar 5-aryl-1,3-dioxanes, see: Al-Mughaid *et al.* (2003); Grosu *et al.* (1997, 1998). For applications of this class of compounds, see: Wang *et al.* (1994); Yuan *et al.* (2005).



## Experimental

## Crystal data

 $\text{C}_{12}\text{H}_{16}\text{O}_4$  $M_r = 224.25$ Orthorhombic,  $C22_1$  $a = 6.2654$  (4) Å $b = 10.4593$  (6) Å $c = 34.5285$  (19) Å $V = 2262.7$  (2) Å<sup>3</sup> $Z = 8$ Mo  $K\alpha$  radiation  
 $\mu = 0.10$  mm<sup>-1</sup> $T = 173$  (2) K  
 $0.46 \times 0.42 \times 0.21$  mm

## Data collection

Bruker SMART 1000  
diffractometer  
Absorption correction: none  
5873 measured reflections1403 independent reflections  
1340 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.031$ 

## Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.066$   
 $wR(F^2) = 0.184$   
 $S = 1.14$   
1403 reflections145 parameters  
H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.25$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.41$  e Å<sup>-3</sup>

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O4}-\text{H4O}\cdots\text{O3}^i$	0.84	2.19	2.644 (6)	114

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINTE* (Bruker, 2003); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *pubCIF* (Westrip, 2008).

We thank the Key Subject Construction Project of Hunan Province (No. 2006-180), the Key Scientific Research Project of Hunan Provincial Education Department (No. 07 A023) and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WN2272).

## References

- Al-Mughaid, H., Grindley, T. B., Robertson, K. N. & Cameron, T. S. (2003). *Can. J. Chem.* **81**, 505–516.
- Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
- Bruker (1997). *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Bruker (2003). *SAINTE*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Grosu, I., Mager, S., Ple, G., Ple, N., Toscano, A., Mesaros, E. & Martinez, R. (1997). *Liebigs Ann.* pp. 2371–2377.
- Grosu, I., Mager, S., Toupet, L., Ple, G., Mesaros, E. & Mihis, A. (1998). *Acta Chem. Scand.* **52**, 366–371.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Wang, G., Yuan, X.-Y., Liu, Y.-C. & Lei, X.-G. (1994). *J. Am. Oil Chem. Soc.* **74**, 727–730.
- Westrip, S. P. (2008). *pubCIF*. In preparation.
- Yuan, X.-Y., Yang, N.-F., Luo, H.-A. & Liu, Y.-J. (2005). *Chin. J. Org. Chem.* **25**, 1049–1052.

## supporting information

*Acta Cryst.* (2008). E64, o1536 [doi:10.1107/S1600536808022046]

**5,5-Bis(hydroxymethyl)-2-phenyl-1,3-dioxane**

**Yi-Ming Luo, Xing-Ming Liu, Xian-You Yuan, Min Zhang and Seik Weng Ng**

**S1. Comment**

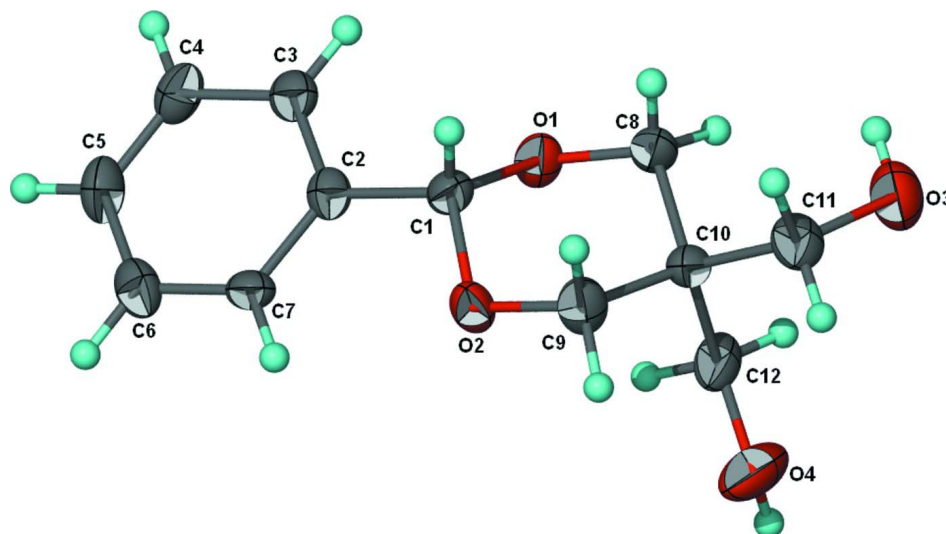
The title compound was synthesized to be used as a precursor in organic syntheses. This class of compounds has useful insecticidal as well as anti-foaming properties (Yuan *et al.*, 2005; Wang *et al.*, 1994). The 1,3-dioxane ring adopts a chair conformation; the phenyl substituent in the 2-position occupies an equatorial position (Fig. 1). Adjacent molecules are linked by O—H $\cdots$ O hydrogen bonds into a chain. The crystal structures of some similar 5-aryl-1,3-dioxanes have been reported (Al-Mughaid *et al.*, 2003; Grosu *et al.* 1997; 1998).

**S2. Experimental**

2,2-Bis(hydroxymethyl)-1,3-propanediol (4.0 g, 30 mmol) and DMF (20 ml) were heated to 353 K. Iodine (0.5 g in an active carbon load of 23.6% by mass) and benzaldehyde (30 ml) were added. The clear solution was heated at 353–363 K for 5 h. The solution was filtered hot and the solvent removed by evaporation. The residue was dissolved in diethyl ether (50 ml) and the solution washed with 5% aqueous sodium bicarbonate. The diethyl ether solution was dried over sodium sulfate. Removal of the solvent gave a solid that was recrystallized from ethyl acetate (yield 5.5 g, 80%); m.p. 408 K.

**S3. Refinement**

In the absence of significant anomalous scattering effects, Friedel pairs were merged. Carbon-bound H-atoms were placed in calculated positions (C—H 0.95 to 0.99 Å) and were included in the refinement in the riding model approximation,  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ . The H-atoms of the hydroxyl groups were placed at calculated positions and then refined as riding; O—H = 0.84 Å and  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$ . For one of the two hydroxyl groups (O3), its hydrogen atom does not form a hydrogen bond to an adjacent acceptor atom. Other possibilities for placing hydrogen atoms on the two groups led to unacceptably short H $\cdots$ H interactions of less than 2 Å.

**Figure 1**

The molecular structure of the title compound, with displacement ellipsoids drawn at the 70% level. Hydrogen atoms are drawn as spheres of arbitrary radius.

### 5,5-Bis(hydroxymethyl)-2-phenyl-1,3-dioxane

#### Crystal data

$C_{12}H_{16}O_4$

$M_r = 224.25$

Orthorhombic,  $C222_1$

Hall symbol:  $C\ 2c\ 2$

$a = 6.2654\ (4)\ \text{\AA}$

$b = 10.4593\ (6)\ \text{\AA}$

$c = 34.5285\ (19)\ \text{\AA}$

$V = 2262.7\ (2)\ \text{\AA}^3$

$Z = 8$

$F(000) = 960$

$D_x = 1.317\ \text{Mg m}^{-3}$

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

Cell parameters from 4610 reflections

$\theta = 2.4\text{--}27.0^\circ$

$\mu = 0.10\ \text{mm}^{-1}$

$T = 173\ \text{K}$

Block, colorless

$0.46 \times 0.42 \times 0.21\ \text{mm}$

#### Data collection

Bruker SMART 1000  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\varphi$  and  $\omega$  scans

5873 measured reflections

1403 independent reflections

1340 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.031$

$\theta_{\text{max}} = 27.0^\circ$ ,  $\theta_{\text{min}} = 2.4^\circ$

$h = -7 \rightarrow 7$

$k = -13 \rightarrow 11$

$l = -24 \rightarrow 44$

#### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.066$

$wR(F^2) = 0.184$

$S = 1.14$

1403 reflections

145 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

$w = 1/[\sigma^2(F_o^2) + (0.059P)^2 + 10.1519P]$

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

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

$\Delta\rho_{\text{max}} = 0.25\ \text{e \AA}^{-3}$

$\Delta\rho_{\text{min}} = -0.41\ \text{e \AA}^{-3}$

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.8999 (6)	0.8806 (3)	0.63367 (9)	0.0263 (8)
O2	0.7606 (6)	0.6825 (3)	0.61705 (10)	0.0271 (8)
O3	0.3975 (7)	0.9308 (4)	0.71647 (11)	0.0410 (10)
H3O	0.4584	0.9956	0.7073	0.061*
O4	0.6297 (7)	0.6260 (4)	0.71881 (12)	0.0509 (12)
H4O	0.7073	0.5941	0.7361	0.076*
C1	0.8416 (8)	0.7994 (4)	0.60313 (13)	0.0223 (10)
H1	0.7304	0.8424	0.5869	0.027*
C2	1.0363 (7)	0.7728 (4)	0.57852 (12)	0.0216 (9)
C3	1.1598 (8)	0.8734 (5)	0.56544 (13)	0.0259 (10)
H3	1.1272	0.9583	0.5732	0.031*
C4	1.3332 (8)	0.8500 (5)	0.54074 (13)	0.0283 (11)
H4	1.4162	0.9193	0.5313	0.034*
C5	1.3830 (9)	0.7266 (5)	0.53020 (14)	0.0304 (11)
H5	1.5014	0.7107	0.5137	0.036*
C6	1.2612 (9)	0.6257 (5)	0.54353 (13)	0.0289 (10)
H6	1.2966	0.5406	0.5364	0.035*
C7	1.0871 (8)	0.6485 (4)	0.56740 (12)	0.0225 (10)
H7	1.0023	0.5791	0.5762	0.027*
C8	0.7140 (9)	0.9151 (4)	0.65602 (14)	0.0266 (11)
H8A	0.6150	0.9651	0.6396	0.032*
H8B	0.7578	0.9698	0.6780	0.032*
C9	0.5640 (8)	0.7038 (5)	0.63773 (14)	0.0282 (11)
H9A	0.5098	0.6214	0.6479	0.034*
H9B	0.4556	0.7391	0.6198	0.034*
C10	0.5989 (8)	0.7970 (4)	0.67141 (12)	0.0210 (9)
C11	0.3785 (8)	0.8368 (5)	0.68698 (14)	0.0287 (11)
H11A	0.2913	0.8713	0.6655	0.034*
H11B	0.3045	0.7608	0.6975	0.034*
C12	0.7350 (8)	0.7357 (5)	0.70299 (13)	0.0300 (11)
H12A	0.8744	0.7098	0.6920	0.036*
H12B	0.7618	0.7987	0.7238	0.036*

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O1	0.0287 (17)	0.0241 (16)	0.0261 (15)	−0.0100 (16)	0.0061 (15)	−0.0039 (13)
O2	0.0310 (18)	0.0194 (15)	0.0310 (16)	−0.0038 (16)	0.0109 (15)	−0.0052 (13)
O3	0.045 (2)	0.039 (2)	0.039 (2)	0.001 (2)	0.011 (2)	−0.0089 (17)
O4	0.046 (2)	0.057 (3)	0.049 (2)	0.018 (2)	0.018 (2)	0.031 (2)
C1	0.027 (2)	0.020 (2)	0.020 (2)	−0.002 (2)	0.0019 (18)	0.0012 (17)
C2	0.020 (2)	0.026 (2)	0.0188 (19)	0.0006 (19)	−0.0012 (17)	−0.0025 (18)
C3	0.032 (3)	0.025 (2)	0.0204 (19)	−0.001 (2)	0.000 (2)	−0.0016 (18)
C4	0.025 (2)	0.037 (3)	0.024 (2)	−0.008 (2)	0.0047 (19)	0.000 (2)
C5	0.024 (2)	0.042 (3)	0.025 (2)	0.000 (2)	0.006 (2)	−0.006 (2)

C6	0.031 (3)	0.030 (2)	0.025 (2)	0.003 (2)	0.000 (2)	-0.0081 (19)
C7	0.030 (2)	0.016 (2)	0.0219 (19)	-0.0002 (19)	0.000 (2)	0.0024 (16)
C8	0.038 (3)	0.0165 (19)	0.025 (2)	-0.006 (2)	0.007 (2)	-0.0024 (18)
C9	0.027 (3)	0.025 (2)	0.032 (2)	-0.009 (2)	0.008 (2)	-0.0056 (19)
C10	0.023 (2)	0.0186 (19)	0.0213 (19)	0.0015 (19)	0.0029 (18)	0.0017 (16)
C11	0.023 (2)	0.033 (3)	0.031 (2)	0.006 (2)	0.000 (2)	0.000 (2)
C12	0.025 (2)	0.042 (3)	0.023 (2)	0.014 (2)	0.004 (2)	0.006 (2)

*Geometric parameters (Å, °)*

O1—C1	1.403 (5)	C5—H5	0.9500
O1—C8	1.443 (6)	C6—C7	1.388 (7)
O2—C1	1.409 (5)	C6—H6	0.9500
O2—C9	1.441 (6)	C7—H7	0.9500
O3—C11	1.421 (6)	C8—C10	1.526 (6)
O3—H3O	0.8400	C8—H8A	0.9900
O4—C12	1.432 (7)	C8—H8B	0.9900
O4—H4O	0.8399	C9—C10	1.534 (6)
C1—C2	1.512 (6)	C9—H9A	0.9900
C1—H1	1.0000	C9—H9B	0.9900
C2—C3	1.382 (7)	C10—C12	1.526 (6)
C2—C7	1.392 (6)	C10—C11	1.539 (7)
C3—C4	1.403 (7)	C11—H11A	0.9900
C3—H3	0.9500	C11—H11B	0.9900
C4—C5	1.377 (7)	C12—H12A	0.9900
C4—H4	0.9500	C12—H12B	0.9900
C5—C6	1.381 (8)		
C1—O1—C8	110.1 (4)	O1—C8—H8A	109.3
C1—O2—C9	110.1 (4)	C10—C8—H8A	109.3
C11—O3—H3O	109.0	O1—C8—H8B	109.3
C12—O4—H4O	108.9	C10—C8—H8B	109.3
O1—C1—O2	111.3 (3)	H8A—C8—H8B	108.0
O1—C1—C2	108.9 (4)	O2—C9—C10	110.6 (4)
O2—C1—C2	108.8 (4)	O2—C9—H9A	109.5
O1—C1—H1	109.3	C10—C9—H9A	109.5
O2—C1—H1	109.3	O2—C9—H9B	109.5
C2—C1—H1	109.3	C10—C9—H9B	109.5
C3—C2—C7	119.5 (4)	H9A—C9—H9B	108.1
C3—C2—C1	119.7 (4)	C8—C10—C12	109.0 (4)
C7—C2—C1	120.8 (4)	C8—C10—C9	108.6 (4)
C2—C3—C4	120.0 (5)	C12—C10—C9	110.7 (4)
C2—C3—H3	120.0	C8—C10—C11	109.1 (4)
C4—C3—H3	120.0	C12—C10—C11	111.4 (4)
C5—C4—C3	120.0 (5)	C9—C10—C11	108.0 (4)
C5—C4—H4	120.0	O3—C11—C10	111.3 (4)
C3—C4—H4	120.0	O3—C11—H11A	109.4
C4—C5—C6	120.2 (5)	C10—C11—H11A	109.4

C4—C5—H5	119.9	O3—C11—H11B	109.4
C6—C5—H5	119.9	C10—C11—H11B	109.4
C5—C6—C7	120.1 (5)	H11A—C11—H11B	108.0
C5—C6—H6	120.0	O4—C12—C10	110.6 (4)
C7—C6—H6	120.0	O4—C12—H12A	109.5
C6—C7—C2	120.3 (4)	C10—C12—H12A	109.5
C6—C7—H7	119.9	O4—C12—H12B	109.5
C2—C7—H7	119.9	C10—C12—H12B	109.5
O1—C8—C10	111.4 (4)	H12A—C12—H12B	108.1
C8—O1—C1—O2	-64.2 (5)	C1—C2—C7—C6	177.4 (4)
C8—O1—C1—C2	175.9 (4)	C1—O1—C8—C10	56.9 (5)
C9—O2—C1—O1	65.2 (5)	C1—O2—C9—C10	-58.0 (5)
C9—O2—C1—C2	-174.9 (4)	O1—C8—C10—C12	70.6 (5)
O1—C1—C2—C3	-50.6 (5)	O1—C8—C10—C9	-50.1 (5)
O2—C1—C2—C3	-172.1 (4)	O1—C8—C10—C11	-167.6 (4)
O1—C1—C2—C7	132.3 (4)	O2—C9—C10—C8	50.6 (5)
O2—C1—C2—C7	10.9 (6)	O2—C9—C10—C12	-69.0 (5)
C7—C2—C3—C4	0.8 (7)	O2—C9—C10—C11	168.7 (4)
C1—C2—C3—C4	-176.2 (4)	C8—C10—C11—O3	-57.8 (5)
C2—C3—C4—C5	-1.4 (7)	C12—C10—C11—O3	62.5 (5)
C3—C4—C5—C6	0.7 (8)	C9—C10—C11—O3	-175.6 (4)
C4—C5—C6—C7	0.5 (8)	C8—C10—C12—O4	178.6 (4)
C5—C6—C7—C2	-1.0 (7)	C9—C10—C12—O4	-62.0 (5)
C3—C2—C7—C6	0.3 (7)	C11—C10—C12—O4	58.2 (5)

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O4—H4O $\cdots$ O3 <sup>i</sup>	0.84	2.19	2.644 (6)	114

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