

Acta Crystallographica Section E

## Structure Reports

Online

ISSN 1600-5368

## (Cyclopentane-1,1-diyl)dimethanol

Richard Betz, Peter Klüfers\* and Peter Mayer

 Ludwig-Maximilians Universität, Department Chemie und Biochemie,  
 Butenandtstrasse 5–13 (Haus D), 81377 München, Germany  
 Correspondence e-mail: kluef@cup.uni-muenchen.de

Received 17 December 2008; accepted 12 January 2009

 Key indicators: single-crystal X-ray study;  $T = 200$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  
 $R$  factor = 0.061;  $wR$  factor = 0.149; data-to-parameter ratio = 19.9.

In the title compound,  $\text{C}_7\text{H}_{14}\text{O}_2$ , co-operative eight-membered homodromic rings of  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonds connect the molecules into strands along [100]. According to graph-set analysis, the descriptor of these cycles is  $R_4^4(8)$ . The cyclopentane-ring adopts an envelope conformation ( ${}^4C_1E$ ).

## Related literature

The compound was synthesized according to a published procedure (Domin *et al.*, 2005). For the influence of chelation to (semi-)metals on the geometry of bifunctional alcohols, see: Klüfers & Vogler (2007). For the structure of a related compound, see Wender *et al.* (1999). For details on graph-set analysis of hydrogen bonds, see Etter *et al.* (1990); Bernstein *et al.* (1995). For details of puckering analysis, see Cremer & Pople (1975).



## Experimental

## Crystal data

 $\text{C}_7\text{H}_{14}\text{O}_2$   
 $M_r = 130.18$   
 Monoclinic,  $P2_1/n$   
 $a = 5.8614$  (16) Å

 $b = 10.631$  (3) Å  
 $c = 11.917$  (3) Å  
 $\beta = 98.33$  (2)°  
 $V = 734.7$  (3) Å<sup>3</sup>
 $Z = 4$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.08$  mm<sup>-1</sup>
 $T = 200$  (2) K  
 $0.20 \times 0.17 \times 0.06$  mm

## Data collection

 Oxford Diffraction Xcalibur  
 diffractometer  
 Absorption correction: none  
 4224 measured reflections

 1692 independent reflections  
 924 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.067$ 

## Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.061$   
 $wR(F^2) = 0.149$   
 $S = 1.01$   
 1692 reflections

 85 parameters  
 H-atom parameters constrained  
 $\Delta\rho_{\text{max}} = 0.19$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.17$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O1}-\text{H1}\cdots\text{O2}^{\text{i}}$	0.84	1.91	2.720 (2)	163
$\text{O2}-\text{H2}\cdots\text{O1}^{\text{ii}}$	0.84	1.88	2.691 (2)	161

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2005); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2005); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

The authors thank Professor Klapötke for generous allocation of diffractometer time and Sandra Albrecht for professional support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZL2170).

## References

- Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.  
 Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.  
 Domin, D., Benito-Garagorri, D., Mereiter, K., Froehlich, J. & Kirchner, K. (2005). *Organometallics*, **24**, 3957–3965.  
 Etter, M. C., MacDonald, J. C. & Bernstein, J. (1990). *Acta Cryst.* **B46**, 256–262.  
 Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.  
 Klüfers, P. & Vogler, C. (2007). *Z. Anorg. Allg. Chem.* **633**, 908–912.  
 Oxford Diffraction (2005). *CrysAlis CCD* and *CrysAlis RED*. Oxford Diffraction Ltd, Abingdon, England.  
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.  
 Wender, P. A., Glorius, F., Husfeld, C. O., Langkopf, E. & Love, J. A. (1999). *J. Am. Chem. Soc.* **121**, 5348–5349.

## supporting information

*Acta Cryst.* (2009). E65, o479 [doi:10.1107/S1600536809001330]

**(Cyclopentane-1,1-diyl)dimethanol**

**Richard Betz, Peter Klüfers and Peter Mayer**

**S1. Comment**

In a program focused on the influence of chelation to (semi-)metals on the geometry of bifunctional alcohols (Klüfers & Vogler, 2007), the structure of 1,1-bis(hydroxymethyl)cyclopentane was elucidated.

Neglecting the hydrogen atoms of the hydroxy groups, the molecule would show non-crystallographic  $C_2$  symmetry (Fig. 1).

According to a conformational analysis (Cremer & Pople, 1975), the cyclopentane-moiety adopts an *envelope* conformation  ${}^cE$  ( $Q_2 = 0.404$  (3) Å), which is slightly distorted towards a *twist* conformation  ${}^cT_{C_3}$  ( $\varphi_2 = 280$  (4)°).

In the crystals structure, hydrogen bonds furnish the formation of cooperative eight-membered homodromic rings (Fig. 2). These connect the molecules to strands along [1 0 0]. In terms of graph-set analysis (Etter *et al.*, 1990; Bernstein *et al.*, 1995), the descriptor for this pattern is  $R_4^4(8)$ .

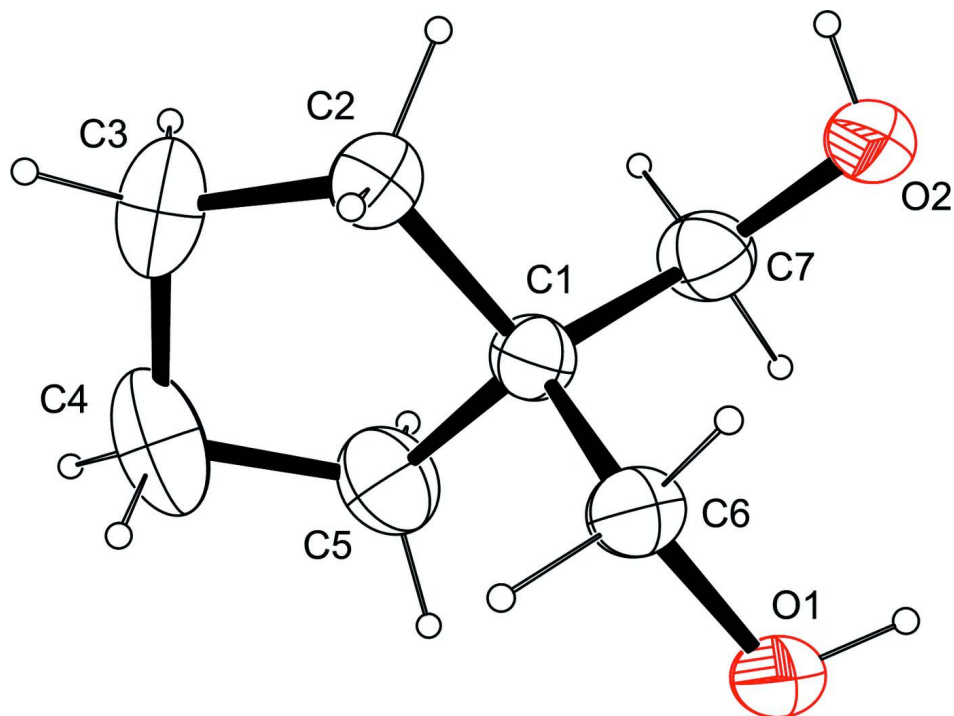
The molecular packing of the compound is shown in Figure 3.

**S2. Experimental**

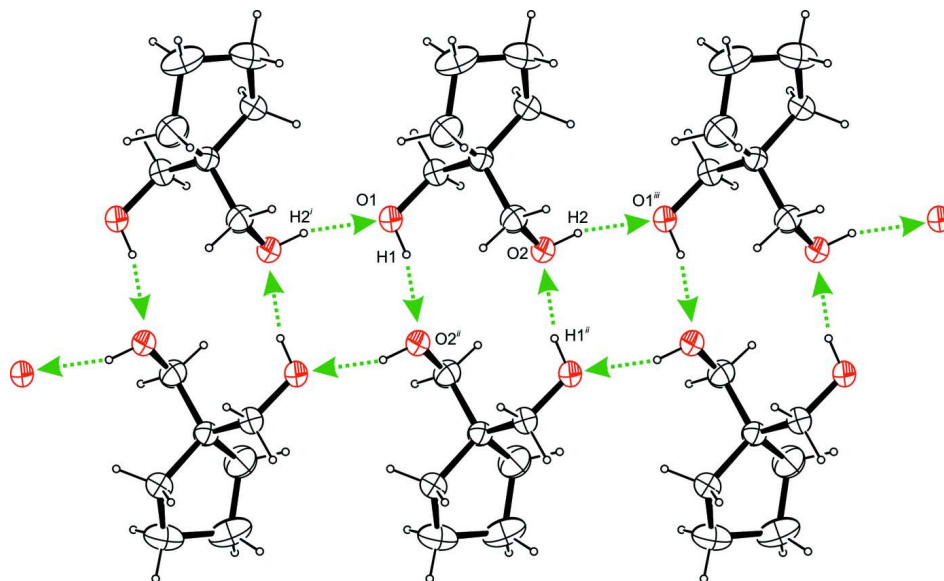
The compound was prepared upon reacting 1,4-dibromobutane with malonic acid diethylester under basic conditions according to a published procedure (Domin *et al.*, 2005). Crystals suitable for X-ray analysis were obtained upon recrystallization of the crude reaction product from a boiling mixture of ethyl acetate - light petrol ether (1:1).

**S3. Refinement**

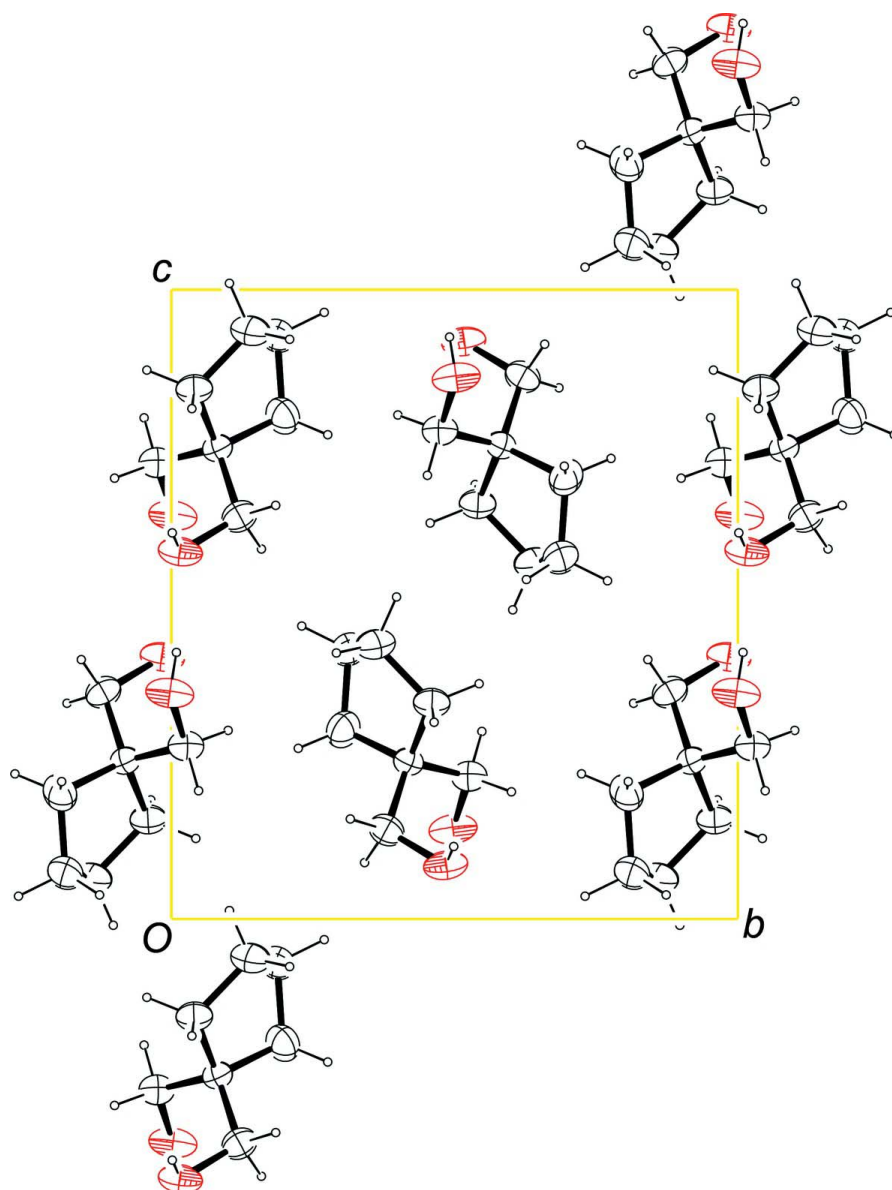
All H-atoms were placed in calculated positions (C—H 0.99 Å and O—H 0.84 Å) and were included in the refinement in the riding model approximation, with  $U(H)$  set to  $1.2U_{eq}(C)$  for methylene groups and  $U(H)$  set to  $1.5U_{eq}(O)$ . Hydroxyl H atoms were allowed to rotate with a fixed angle around the C-O bond to best fit the experimental electron density (HFIX 147 in the *SHELX* program suite (Sheldrick, 2008)).

**Figure 1**

The molecular structure of the title compound, with atom labels and anisotropic displacement ellipsoids (drawn at 50% probability level) for non-H atoms.

**Figure 2**

Hydrogen bonds in the crystal structure of the title compound, viewed along  $[0\ 1\ 0]$ . Symmetry operators:  $i$   $x - 1, y, z$ ;  $ii$   $-x + 1, -y + 1, -z$ ;  $iii$   $x + 1, y, z$ .

**Figure 3**

The packing of the title compound, viewed along  $[-1\ 0\ 0]$ .

### (Cyclopentane-1,1-diyl)dimethanol

#### Crystal data

$C_7H_{14}O_2$

$M_r = 130.18$

Monoclinic,  $P2_1/n$

Hall symbol:  $-P\ 2_1n$

$a = 5.8614\ (16)\ \text{\AA}$

$b = 10.631\ (3)\ \text{\AA}$

$c = 11.917\ (3)\ \text{\AA}$

$\beta = 98.33\ (2)^\circ$

$V = 734.7\ (3)\ \text{\AA}^3$

$Z = 4$

$F(000) = 288$

$D_x = 1.177\ \text{Mg m}^{-3}$

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

$\theta = 4.6\text{--}27.5^\circ$

$\mu = 0.08\ \text{mm}^{-1}$

$T = 200\ \text{K}$

Platelet, colourless

$0.20 \times 0.17 \times 0.06\ \text{mm}$

*Data collection*

Oxford Diffraction Xcalibur  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\omega$  scans

4224 measured reflections

1692 independent reflections

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

$R_{\text{int}} = 0.067$

$\theta_{\text{max}} = 27.5^\circ$ ,  $\theta_{\text{min}} = 4.6^\circ$

$h = -7 \rightarrow 4$

$k = -13 \rightarrow 13$

$l = -14 \rightarrow 15$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

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

$wR(F^2) = 0.149$

$S = 1.01$

1692 reflections

85 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

$w = 1/[\sigma^2(F_o^2) + (0.0527P)^2]$

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

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

$\Delta\rho_{\text{max}} = 0.19 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\text{min}} = -0.17 \text{ e } \text{\AA}^{-3}$

*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	0.2142 (2)	0.49721 (17)	0.14033 (13)	0.0473 (5)
H1	0.2522	0.5090	0.0758	0.071*
O2	0.7532 (3)	0.48406 (16)	0.08445 (12)	0.0431 (5)
H2	0.8893	0.4984	0.1146	0.065*
C1	0.5747 (4)	0.41846 (19)	0.24878 (17)	0.0299 (5)
C2	0.7724 (4)	0.4594 (2)	0.34277 (18)	0.0381 (6)
H21	0.9204	0.4638	0.3122	0.046*
H22	0.7393	0.5432	0.3729	0.046*
C3	0.7843 (5)	0.3602 (2)	0.4357 (2)	0.0562 (8)
H31	0.8906	0.2912	0.4223	0.067*
H32	0.8361	0.3975	0.5112	0.067*
C4	0.5393 (6)	0.3131 (3)	0.4270 (2)	0.0586 (8)
H41	0.4413	0.3733	0.4619	0.070*
H42	0.5330	0.2302	0.4641	0.070*
C5	0.4640 (4)	0.3034 (2)	0.2995 (2)	0.0475 (7)
H51	0.2939	0.3064	0.2812	0.057*
H52	0.5198	0.2240	0.2696	0.057*
C6	0.4039 (4)	0.5258 (2)	0.22489 (19)	0.0363 (6)
H61	0.3447	0.5481	0.2960	0.044*
H62	0.4853	0.6002	0.2004	0.044*
C7	0.6631 (4)	0.3799 (2)	0.14014 (19)	0.0388 (6)
H71	0.7856	0.3158	0.1581	0.047*
H72	0.5356	0.3410	0.0880	0.047*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O1	0.0243 (8)	0.0853 (13)	0.0320 (8)	0.0014 (8)	0.0036 (7)	0.0073 (9)
O2	0.0276 (8)	0.0721 (12)	0.0303 (8)	-0.0023 (9)	0.0060 (7)	0.0062 (8)
C1	0.0290 (11)	0.0332 (11)	0.0274 (11)	-0.0025 (10)	0.0043 (9)	-0.0028 (9)
C2	0.0331 (12)	0.0496 (14)	0.0308 (11)	-0.0007 (11)	0.0018 (10)	-0.0010 (10)
C3	0.074 (2)	0.0586 (17)	0.0326 (13)	0.0067 (16)	-0.0032 (14)	0.0047 (12)
C4	0.093 (2)	0.0465 (15)	0.0408 (14)	-0.0035 (16)	0.0266 (15)	0.0052 (12)
C5	0.0526 (16)	0.0417 (14)	0.0500 (15)	-0.0048 (13)	0.0139 (13)	0.0066 (12)
C6	0.0288 (11)	0.0470 (14)	0.0330 (11)	0.0021 (11)	0.0039 (10)	0.0001 (10)
C7	0.0348 (12)	0.0454 (13)	0.0370 (13)	0.0008 (11)	0.0074 (11)	-0.0068 (11)

Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

O1—C6	1.421 (3)	C3—H31	0.9900
O1—H1	0.8400	C3—H32	0.9900
O2—C7	1.431 (3)	C4—C5	1.523 (4)
O2—H2	0.8400	C4—H41	0.9900
C1—C6	1.517 (3)	C4—H42	0.9900
C1—C7	1.519 (3)	C5—H51	0.9900
C1—C5	1.548 (3)	C5—H52	0.9900
C1—C2	1.553 (3)	C6—H61	0.9900
C2—C3	1.524 (3)	C6—H62	0.9900
C2—H21	0.9900	C7—H71	0.9900
C2—H22	0.9900	C7—H72	0.9900
C3—C4	1.510 (4)		
C6—O1—H1	109.5	C5—C4—H41	111.2
C7—O2—H2	109.5	C3—C4—H42	111.2
C6—C1—C7	109.90 (18)	C5—C4—H42	111.2
C6—C1—C5	111.40 (18)	H41—C4—H42	109.1
C7—C1—C5	109.48 (18)	C4—C5—C1	104.99 (19)
C6—C1—C2	109.13 (17)	C4—C5—H51	110.7
C7—C1—C2	112.31 (17)	C1—C5—H51	110.7
C5—C1—C2	104.54 (18)	C4—C5—H52	110.7
C3—C2—C1	106.30 (19)	C1—C5—H52	110.7
C3—C2—H21	110.5	H51—C5—H52	108.8
C1—C2—H21	110.5	O1—C6—C1	113.57 (18)
C3—C2—H22	110.5	O1—C6—H61	108.9
C1—C2—H22	110.5	C1—C6—H61	108.9
H21—C2—H22	108.7	O1—C6—H62	108.9
C4—C3—C2	103.7 (2)	C1—C6—H62	108.9
C4—C3—H31	111.0	H61—C6—H62	107.7
C2—C3—H31	111.0	O2—C7—C1	112.37 (18)
C4—C3—H32	111.0	O2—C7—H71	109.1
C2—C3—H32	111.0	C1—C7—H71	109.1
H31—C3—H32	109.0	O2—C7—H72	109.1

C3—C4—C5	103.1 (2)	C1—C7—H72	109.1
C3—C4—H41	111.2	H71—C7—H72	107.9
C6—C1—C2—C3	125.3 (2)	C2—C1—C5—C4	19.5 (2)
C7—C1—C2—C3	-112.5 (2)	C7—C1—C6—O1	57.0 (2)
C5—C1—C2—C3	6.1 (2)	C5—C1—C6—O1	-64.6 (2)
C1—C2—C3—C4	-29.5 (2)	C2—C1—C6—O1	-179.48 (17)
C2—C3—C4—C5	41.6 (3)	C6—C1—C7—O2	53.2 (2)
C3—C4—C5—C1	-38.0 (3)	C5—C1—C7—O2	175.88 (19)
C6—C1—C5—C4	-98.3 (2)	C2—C1—C7—O2	-68.5 (2)
C7—C1—C5—C4	140.0 (2)		

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1 $\cdots$ O2 <sup>i</sup>	0.84	1.91	2.720 (2)	163
O2—H2 $\cdots$ O1 <sup>ii</sup>	0.84	1.88	2.691 (2)	161

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