

2,6-Bis(hydroxymethyl)-4-methylphenol benzylidene 1,2-acetal

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Key indicators

Single-crystal X-ray study

$T = 298\text{ K}$

Mean $\sigma(\text{C}-\text{C}) = 0.004\text{ \AA}$

R factor = 0.063

wR factor = 0.176

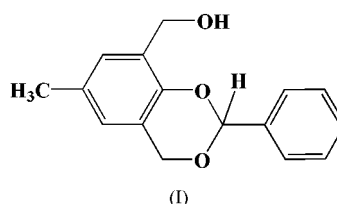
Data-to-parameter ratio = 13.2

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The title compound, $\text{C}_{16}\text{H}_{16}\text{O}_3$, is an intermediate compound in the synthesis of polypodal binucleating ligands which are suited for the preparation of dinuclear complexes, employing two non-equivalent complexation sites. There are two weak intramolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds generating a ring of pattern $S(5)$, and an intermolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bond.

Comment

The title compound, (I), is an intermediate compound in the synthesis of polypodal binucleating ligands which are suited for the preparation of dinuclear complexes employing two non-equivalent complexation sites (Belle *et al.*, 1994).



Selected geometric parameters of (I) are listed in Table 1. The molecular conformation and crystal packing are illustrated in Figs. 1 and 2. The molecule has two weak intramolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds (Pálinkó, 1999), with $\text{C}\cdots\text{O}$ distances of 2.777 (4) and 2.709 (3) \AA , generating an $S(5)$ pattern, with the hydroxy O atoms as two acceptors and two aromatic CH groups acting as donors (Bernstein *et al.*,

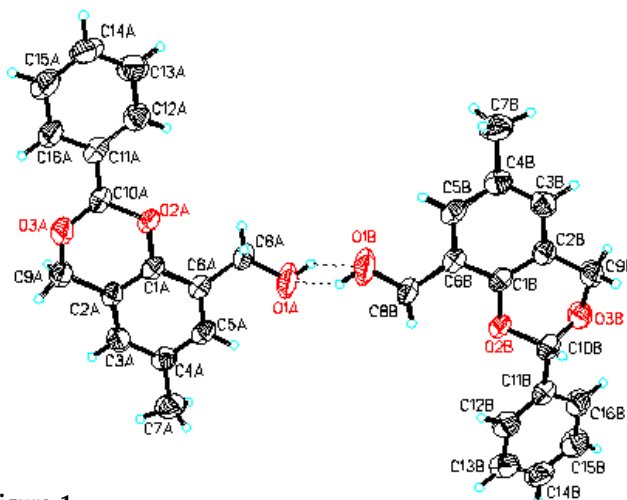


Figure 1

A view of the molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. Two molecules are shown, with the pair of hydrogen bonds (dashed lines) linking them across an inversion centre.

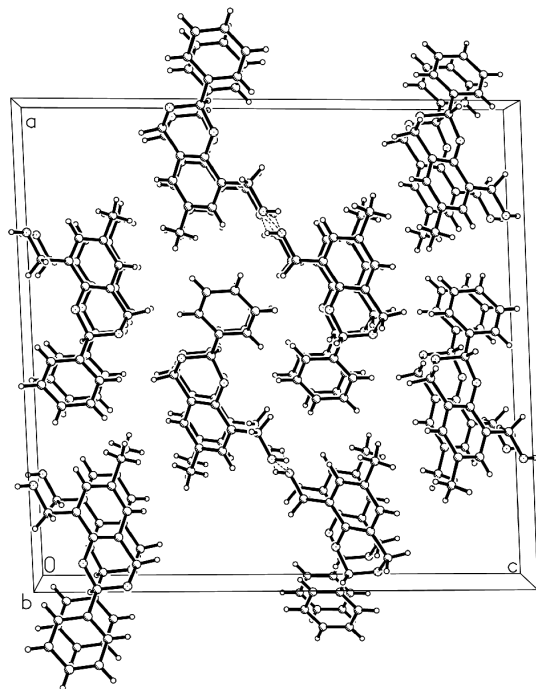


Figure 2
The packing of the title compound, viewed down the *b* axis.

1995). The pairs of enantiomers related by the inversion centre are linked by two O—H...O hydrogen bonds between their OH groups (Fig. 1, Table 2). Specifically, the O—H...O($\frac{1}{2} - x, \frac{3}{2} - y, -z$) hydrogen bond, with a distance of 2.721 (4) Å (Table 2), is formed. This generates a ring with motif $R_1^1(4)$ (Bernstein *et al.*, 1995). The lamellar structure is dominated by van der Waals interactions in the packing of the molecules. Within the molecule, atom C10 deviates by 0.0853 (4) Å from the C11/C12/C13/C14/C15/C16 plane. Atoms O2 and O3 deviate by 0.7762 (4) and 0.2811 (5), respectively, from the C11/C12/C13/C14/C15/C16 plane; the offsets are 0.0116 (3) and 0.4233 (4), respectively, from the C1/C2/C3/C4/C5/C6 plane.

Experimental

The title compound was synthesized according to a literature method (Belle *et al.*, 1994). The compound (100 mg) was dissolved in hexane/ethyl acetate (8:1, 10 ml) and the solution was allowed to evaporate slowly at room temperature over a period of several months. Colourless crystals, suitable for X-ray crystallography studies, were formed. $^1\text{H NMR}$ (CDCl_3 , δ , p.p.m.): 2.26 (s, 3H, CH_3), 4.65–5.20 (dd, 4H, CH_2), 6.80–7.90 (m, 7H, ArH), 6.01 (s, 1H, OH); m.p. 368 K.

Crystal data

$\text{C}_{16}\text{H}_{16}\text{O}_3$
 $M_r = 256.29$
Monoclinic, $C2/c$
 $a = 22.782$ (11) Å
 $b = 4.884$ (2) Å
 $c = 23.432$ (11) Å
 $\beta = 92.461$ (7)°
 $V = 2605$ (2) Å³
 $Z = 8$

$D_x = 1.307$ Mg m⁻³
Mo $K\alpha$ radiation
Cell parameters from 14967 reflections
 $\theta = 2.4$ – 22.6°
 $\mu = 0.09$ mm⁻¹
 $T = 298$ (2) K
Rod, colourless
 $0.60 \times 0.30 \times 0.10$ mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer
 ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.948$, $T_{\max} = 0.991$
5974 measured reflections

2287 independent reflections
1542 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.087$
 $\theta_{\text{max}} = 25.0^\circ$
 $h = -21 \rightarrow 27$
 $k = -5 \rightarrow 5$
 $l = -27 \rightarrow 24$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.063$
 $wR(F^2) = 0.177$
 $S = 1.04$
2287 reflections
173 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0889P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.25$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.22$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

O2—C1	1.392 (3)	C2—C9	1.505 (3)
O2—C10	1.433 (3)	C10—C11	1.502 (3)
O3—C10	1.379 (3)	C4—C7	1.502 (4)
O3—C9	1.431 (3)	C8—O1	1.418 (3)
C6—C8	1.500 (4)		
C1—O2—C10	112.32 (18)	O3—C10—O2	111.1 (2)
C10—O3—C9	110.93 (19)	O3—C10—C11	110.0 (2)
C5—C6—C8	123.0 (2)	O2—C10—C11	108.4 (2)
C1—C6—C8	119.7 (2)	C5—C4—C7	120.7 (2)
C2—C1—O2	121.1 (2)	C3—C4—C7	121.4 (3)
O2—C1—C6	117.0 (2)	O3—C9—C2	110.3 (2)
C1—C2—C9	120.0 (2)	O1—C8—C6	111.4 (2)
C3—C2—C9	121.1 (2)		

Table 2

Hydrogen-bonding geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1...O1 ⁱ	0.82	2.11	2.721 (4)	132
C5—H5...O1	0.93	2.42	2.777 (4)	102
C16—H16...O3	0.93	2.38	2.709 (3)	101

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

All H atoms on Csp^3 atoms were treated as riding, with $\text{C—H} = 0.96$ – 0.98 Å and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ of the parent atom. The H atoms on Csp^2 atoms were treated as riding, with $\text{C—H} = 0.93$ Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The hydroxyl H atom was obtained from a difference Fourier map and was constrained to maintain a bond distance of 0.82 Å and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$.

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, 1999); software used to prepare material for publication: SHELXTL.

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