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1,6-Bis[3-(hydroxymethyl)phenoxy]hexane

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Key indicators

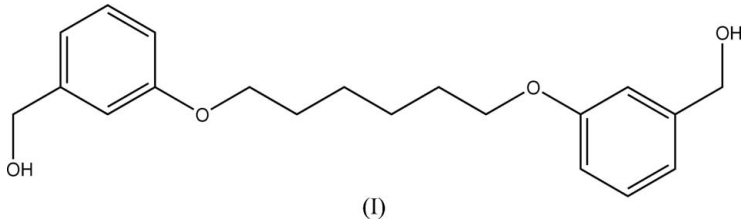
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.060
wR factor = 0.178
Data-to-parameter ratio = 16.1

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

The molecule of the title compound, $C_{20}H_{26}O_4$, has a center of symmetry. Molecules are linked into a zigzag chain *via* an O—H···O hydrogen bond.

Comment

Diethers with terminal hydroxyl radicals can be used to synthesize linear or cyclic ethers in a highly efficient and convergent manner (Davies *et al.*, 2000). The method permits a high level of control, affording non-symmetrical compounds. In addition, the procedure can be used for the stereo-controlled synthesis of cyclic ethers (Diaz *et al.*, 2001). Several kinetic studies of the OH-initiated degradation of diethers have been reported by Moriarty *et al.* (2003). As part of a study on the reactivity of ethers, we have determined the structure of the title compound, (I).



(I)

The title molecule has a crystallographically imposed center of symmetry. All bond lengths and angles in (I) have normal values (Allen *et al.*, 1987). In the crystal structure, molecules are linked into a zigzag chain along the [201] direction by a weak O—H···O hydrogen bond (Fig. 2 and Table 2).

Experimental

The title compound was obtained according to the literature method (Fekner *et al.*, 2004). To a solution of 3-hydroxymethylphenol (1.5 g,

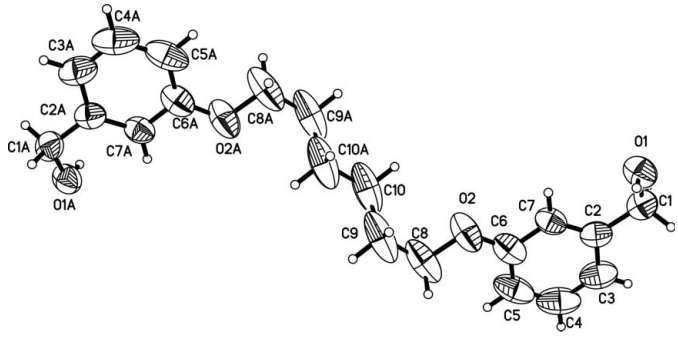


Figure 1
The structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. The suffix A corresponds to symmetry code $(1-x, 2-y, 1-z)$.

12 mmol) in EtOH (90 ml) was added 10 mol L^{-1} NaOH (1.2 ml, 12 mmol), followed by 1,6-dibromohexane (1.5 g, 6 mmol). The reaction was refluxed for 26 h, cooled to room temperature, and diluted with water (30 ml). The brown mixture was subsequently extracted with CH_2Cl_2, and the combined extracts were dried ($MgSO_4$) and concentrated *in vacuo* to give an off-white solid. Purification by flash chromatography (CH_2Cl_2/Me_2CO 4:1, *v*/*v*) gave the title compound as a slightly pink solid. Colourless single crystals were obtained by slow evaporation of an EtOH solution at room temperature over a period of two weeks.

Crystal data

$C_{20}H_{26}O_4$
M_r = 330.41
Monoclinic, $P2_1/c$
a = 14.7385 (17) Å
b = 4.9176 (6) Å
c = 12.7760 (14) Å
β = 103.749 (2)°
V = 899.45 (18) Å^3
Z = 2
D_x = 1.220 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1296 reflections
θ = 2.8–23.5°
μ = 0.08 mm^{-1}
T = 293 (2) K
Plate, colourless
0.34 × 0.23 × 0.06 mm

Data collection

Siemens SMART 1000 CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.972, T_{max} = 0.995
4749 measured reflections
1756 independent reflections
1281 reflections with $I > 2\sigma(I)$
R_{int} = 0.017
θ_{max} = 26.0°
$h = -18 \rightarrow 9$
$k = -5 \rightarrow 6$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.178
S = 1.06
1756 reflections
109 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0794P)^2 + 0.2564P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Table 1
Selected bond lengths (Å).

O1—C1	1.400 (2)	C8—C9	1.493 (5)
O2—C6	1.369 (3)	C9—C10	1.523 (5)
O2—C8	1.431 (3)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···O1^{i}	0.82	1.96	2.775 (2)	170

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

All H atoms were located in a difference Fourier map and constrained to ride on their parent atoms, with O—H = 0.82 Å and C—H = 0.93–0.97 Å, and with U_{iso}(H) = 1.5U_{eq}(O) and 1.2 U_{eq}(C).

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

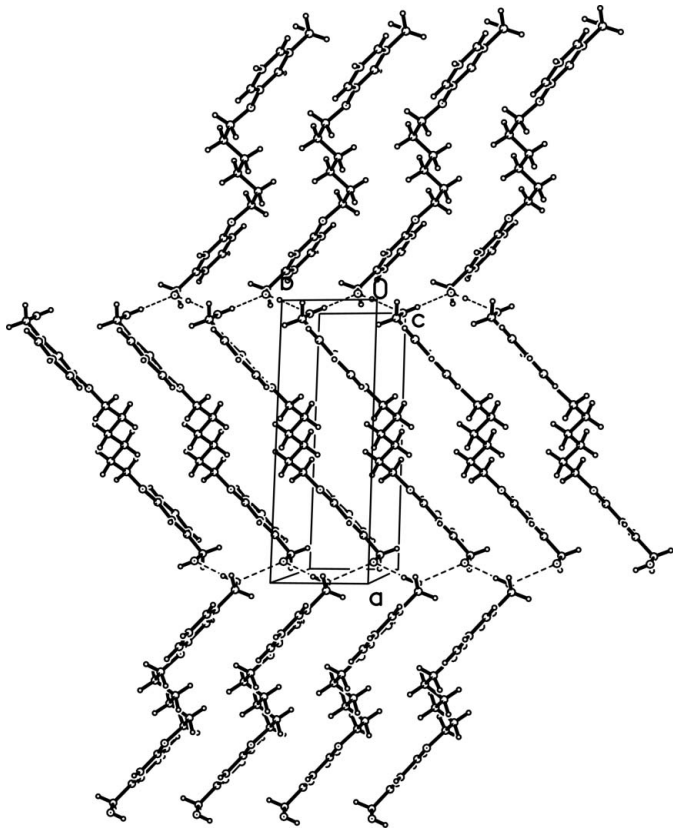


Figure 2
The packing, viewed along the *c* axis. Hydrogen bonds are indicated by dashed lines.

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