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Key indicators

Single-crystal X-ray study
 $T = 294$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.031
 wR factor = 0.074
Data-to-parameter ratio = 8.2For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.3,3'-Dihydroxy-4,4'-(propane-1,3-diyl)di-
benzaldehyde

The title compound, $\text{C}_{19}\text{H}_{20}\text{O}_6$, is composed of two individual vanillin subunits bonded to a central chain of C atoms. The molecule lies on a twofold rotation axis. The two aromatic rings are approximately perpendicular to each other and both make a dihedral angle of 53.36 (4) $^\circ$ with the backbone chain. A weak $\text{C}-\text{H} \cdots \pi$ interaction consolidates the crystal packing.

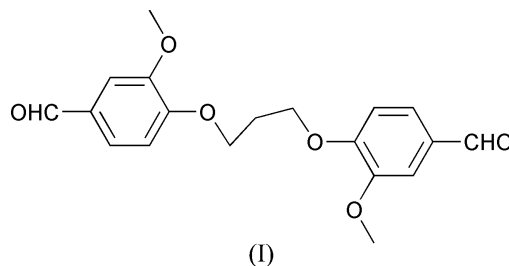
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Comment

Since the first syntheses of macrocyclic crown ethers that are capable of forming stable and selective complexes with alkali and alkaline earth metal ions were performed by Pedersen (1967), much effort has been devoted to the understanding of the factors that control the thermodynamic and kinetic stability and selectivity of the resulting complexes (Kim *et al.*, 1999). We are interested in the molecular and ionic recognition of crown ethers. As part of this study, we report the synthesis and structure of the title compound, (I).



A view of the molecule of (I) is shown in Fig. 1. The molecule lies on a twofold rotation axis. Each vanillin group ($\text{C}1-\text{C}6/\text{C}8/\text{O}1/\text{O}2/\text{O}3$) is planar, with an r.m.s. deviation for fitted atoms of 0.0093 Å. The two vanillin planes make a dihedral

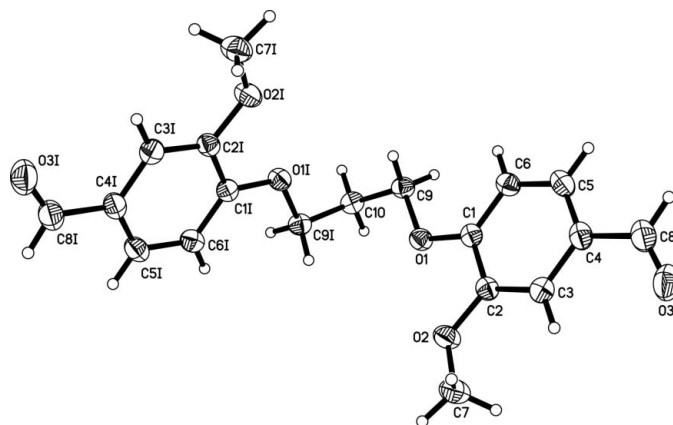


Figure 1

The structure of (I), with displacement ellipsoids for non-H atoms drawn at the 30% probability level. [Symmetry code: (I) $-x + 1, y, z$.]

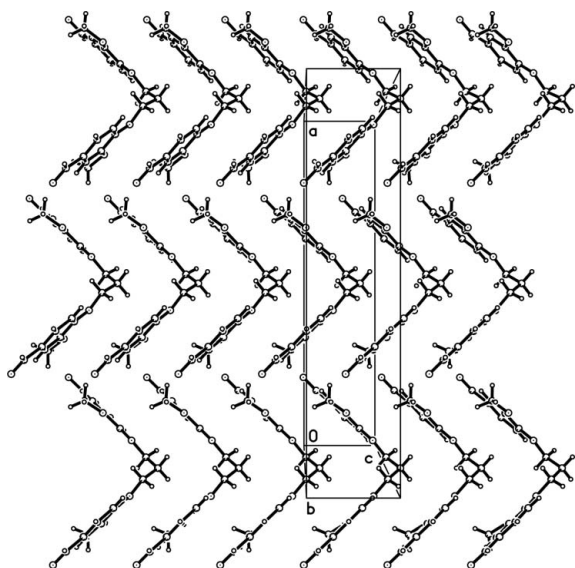


Figure 2
A view down the *b* axis of the packing arrangement in the crystal structure of (I).

angle of $88.20(2)^\circ$, indicating that they are approximately perpendicular to each other. However, the dihedral angle between the $C9-C10-C9^i$ plane [symmetry code: (i) $-x + 1, -y, z$] and the vanillin plane is $53.36(4)^\circ$, in contrast with the value of $3.0(3)^\circ$ in 4-[6-(4-formyl-2-methoxyphenoxy)hexyloxy]-3-methoxybenzaldehyde (Diao *et al.*, 2005). The difference may be attributed to the shorter C-atom chain in (I). All bond lengths and angles for (I) (Table 1) are within normal ranges. Furthermore, the $C9-H9A \cdots Cg^{ii}$ [symmetry code: (ii) $x, y, z + 1$; Cg is the centroid of the C1–C6 ring] distance is 3.631 Å, suggesting the existence of a weak C–H $\cdots\pi$ interaction.

Experimental

To a solution of 4-hydroxy-3-methoxybenzaldehyde (15.2 g, 100 mmol) and potassium carbonate (13.8 g, 100 mmol) in acetonitrile (500 ml), 1,3-dibromohexane (10.1 g, 50 mmol) was added over a period of 30 min, and the mixture refluxed for 24 h under nitrogen. The solvent was removed and the resultant oil poured into ice–water (500 ml). The white precipitate was then isolated and recrystallized from ethanol to give the pure compound in 55% yield. Colourless single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an acetonitrile solution.

Crystal data

$C_{19}H_{20}O_6$
 $M_r = 344.35$
 Orthorhombic, *Fdd2*
 $a = 22.247(5)$ Å
 $b = 31.175(6)$ Å
 $c = 4.8562(10)$ Å
 $V = 3368.1(12)$ Å³
 $Z = 8$
 $D_x = 1.358$ Mg m⁻³

Mo $K\alpha$ radiation
 Cell parameters from 1884 reflections
 $\theta = 2.3$ – 26.1°
 $\mu = 0.10$ mm⁻¹
 $T = 294(2)$ K
 Block, colourless
 $0.26 \times 0.20 \times 0.18$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.964$, $T_{\max} = 0.982$
 4635 measured reflections

950 independent reflections
 781 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.029$
 $\theta_{\max} = 26.2^\circ$
 $h = -20 \rightarrow 27$
 $k = -38 \rightarrow 37$
 $l = -6 \rightarrow 4$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.031$
 $wR(F^2) = 0.074$
 $S = 1.10$
 950 reflections
 116 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0299P)^2 + 1.8179P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.13$ e Å⁻³
 $\Delta\rho_{\min} = -0.12$ e Å⁻³
 Extinction correction: SHELXL97
 Extinction coefficient: 0.0028 (3)

Table 1

Selected geometric parameters (Å, °).

O1–C1	1.355 (2)	O2–C7	1.423 (3)
O1–C9	1.439 (2)	O3–C8	1.204 (3)
O2–C2	1.371 (2)		
C1–O1–C9	116.87 (15)	O2–C2–C3	124.5 (2)
C2–O2–C7	116.59 (18)	O2–C2–C1	115.52 (18)
O1–C1–C6	124.5 (2)	O3–C8–C4	125.8 (2)
O1–C1–C2	115.80 (17)	O1–C9–C10	108.09 (15)

H atoms were included in calculated positions and refined using a riding-model approximation. Constrained C–H bond lengths are within the range 0.93–0.97 Å. $U_{\text{iso}}(\text{H})$ values were set equal to $1.2U_{\text{eq}}(\text{C})$ for aromatic and methylene H atoms, and $1.5U_{\text{eq}}(\text{C})$ for methyl H atoms. In the absence of significant anomalous scattering effects, Friedel pairs were merged.

Data collection: SMART (Bruker, 1999); cell refinement: SAINT (Bruker, 1999); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997a); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997a); molecular graphics: SHELXTL (Sheldrick, 1997b); software used to prepare material for publication: SHELXTL.

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