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3-[4-(5-Formyl-2-methoxyphenoxy)butoxy]-4-methoxybenzaldehyde

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.004 Å
R factor = 0.049
wR factor = 0.131
Data-to-parameter ratio = 15.9

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

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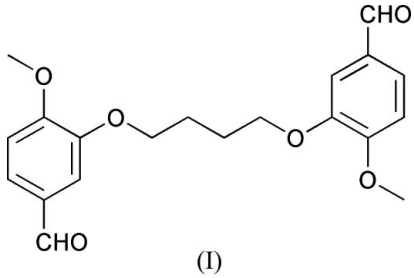
The molecule of the title compound, $C_{20}H_{22}O_6$, lies on crystallographic center of symmetry. The isovanillin group makes a dihedral angle of 2.8 (5)° with the four C atoms of the central chain. A weak intermolecular C—H···O hydrogen bond links molecules into extended one-dimensional chains.

Comment

Since the early work on macrocyclic crown ethers was carried out by Pedersen (1967), considerable effort has been devoted to the study of these species (Kim *et al.*, 1999). Crown ethers are capable of forming stable and selective complexes with metal cations, halide anions and small organic molecules. We are interested in the molecular and ionic recognition of these crown ethers. As part of this study, we report the synthesis and structure of the title compound, (I), used as a precursor in their preparation.



(I)

In (I) (Fig. 1), a crystallographic center of symmetry is located at the mid-point of the central C—C bond. The bond lengths and angles are as expected. The isovanillin group (C1–C7/O1/O2/O3) is essentially planar, with an r.m.s. deviation for the fitted atoms of 0.0261 Å. The torsion angle of 176.8 (2)° for C6—C1—O1—C9, in conjunction with the value of 2.8 (5)° for the dihedral angle between the central chain of C atoms and the isovanillin group, confirm the nearly planar conformation of the molecule. The geometry is similar to that in 4-[6-(4-formyl-2-methoxyphenoxy)hexyloxy]-3-methoxybenzaldehyde (Diao *et al.*, 2005), in which the dihedral angle between the central C-atom chain and the atoms of the isovanillin group is 3.0 (3)°

A weak intermolecular C—H···O hydrogen bond (Table 1) links molecules into extended one-dimensional chains (Fig. 2).

Experimental

To a solution of 3-hydroxy-4-methoxybenzaldehyde (15.2 g, 100 mmol) and potassium carbonate (13.8 g, 100 mmol) in acetonitrile (500 ml), 1,4-dibromobutane (10.8 g, 50 mmol) was added dropwise over a period of 30 min, and the mixture refluxed for 24 h under nitrogen. The solvent was removed and the resultant mixture poured into ice-water (500 ml). The white precipitate was then

isolated and recrystallized from ethanol to give the pure compound in 51% yield. Colorless single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an acetonitrile solution.

Crystal data

$C_{20}H_{22}O_6$	D_x = 1.277 Mg m^{-3}
M_r = 358.38	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 1180 reflections
a = 7.828 (2) Å	θ = 3.0–24.3°
b = 7.261 (2) Å	μ = 0.09 mm^{-1}
c = 16.445 (4) Å	T = 294 (2) K
β = 94.499 (5)°	Block, colorless
V = 931.9 (4) Å^3	0.22 × 0.20 × 0.14 mm
Z = 2	

Data collection

Bruker SMART-CCD area-detector diffractometer	1907 independent reflections
	1109 reflections with $I > 2\sigma(I)$
φ and ω scans	R_{int} = 0.039
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	θ_{max} = 26.4°
	$h = -9 \rightarrow 9$
T_{min} = 0.972, T_{max} = 0.987	$k = -8 \rightarrow 9$
5085 measured reflections	$l = -20 \rightarrow 12$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0515P)^2 + 0.0565P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.049	
$wR(F^2)$ = 0.131	
S = 1.14	$(\Delta/\sigma)_{max}$ = 0.005
1907 reflections	$\Delta\rho_{max}$ = 0.39 e Å^{-3}
120 parameters	$\Delta\rho_{min}$ = −0.24 e Å^{-3}
H-atom parameters constrained	

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

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8B···O1^{i}	0.96	2.57	3.512 (4)	167

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

H atoms were included in calculated positions and refined using a riding-model approximation, with C—H bond lengths and isotropic U parameters as follows: 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C) for aromatic CH; 0.97 Å and U_{iso}(H) = 1.2U_{eq}(C) for methylene CH_2; 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C) for methyl CH_3.

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

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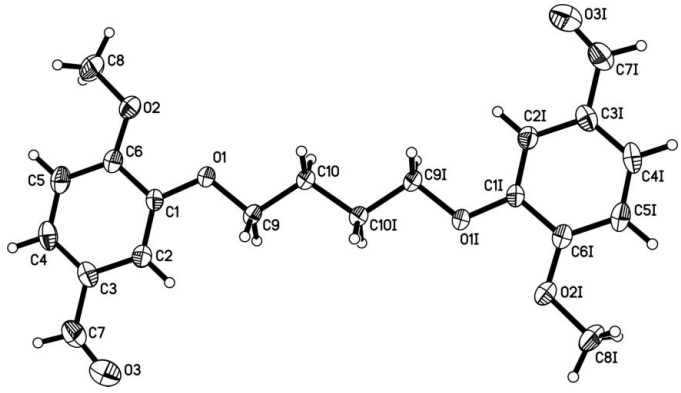

Figure 1
The structure of (I), with displacement ellipsoids drawn at the 30% probability level. H atoms are drawn as spheres of arbitrary radii [symmetry code: (I) $-x, 2-y, 2-z$].

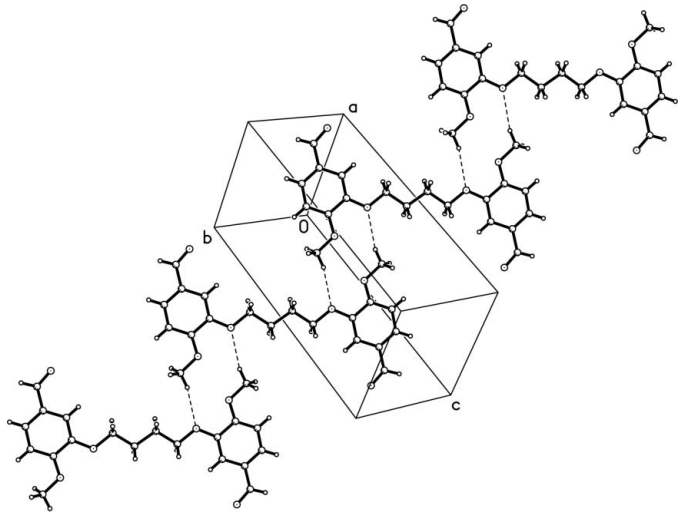

Figure 2
Partial packing diagram, showing hydrogen-bonding interactions as dashed lines.

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