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3-[6-(5-Formyl-2-methoxyphenoxy)hexyloxy]-4-methoxybenzaldehyde

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Key indicators

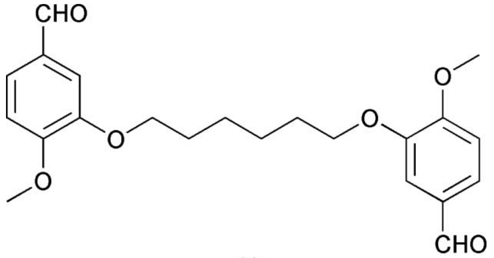
Single-crystal X-ray study
T = 294 K
Mean *σ*(C–C) = 0.005 Å
Disorder in main residue
R factor = 0.068
wR factor = 0.141
Data-to-parameter ratio = 12.9

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

The title compound, $C_{18}H_{18}O_4$, lies on crystallographic center of symmetry. Each isovanillin group makes a dihedral angle of 8.0 (4)° with the six C atoms in the central chain. The two aromatic rings are parallel to each other by symmetry.

Comment

The pioneering work of Pedersen (1967) on the synthesis of macrocyclic crown ethers was a milestone in this field of chemistry. These compounds are capable of forming stable and selective complexes with alkali and alkaline earth metal ions. Consequently, a significant effort has been devoted to the synthesis of derivatives and these species have been used to study their molecular recognition of special guest molecules and cations (Lee *et al.*, 2000; Zhang & Buchwald, 2000). As part of our interest in the molecular and ionic recognition properties of crown ethers, we investigated the title compound, (I), which is used as a precursor in the preparation of crown ethers.



(I)

In (I) (Fig. 1), a crystallographic center of symmetry is located at the mid-point of the central C11—C11^{i} bond [symmetry code: (i) $-x + 1$, $-y - 1$, $-z$]. Each isovanillin group (C1–C6/C8/O1/O2) is essentially planar. The chain of atoms C9–C11/C11^{i}–C9^{i} linking the two isovanillin groups is planar. The two aromatic rings in the molecule are exactly parallel to each other by symmetry. The C9—O2—C1—C2 torsion angle of 174.1 (3)° confirms the nearly planar conformation of the molecule. The geometry is similar to that reported recently for the closely related species 4-[6-(4-formyl-2-methoxyphenoxy)hexyloxy]-3-methoxybenzaldehyde (Diao *et al.*, 2005). However, the dihedral angle between the bridge plane (C9–C11/C11^{i}–C9^{i}) and the isovanillin group plane is 8.0 (4)°, in comparison with 3.0 (3)° in 4-[6-(4-formyl-2-methoxyphenoxy)hexyloxy]-3-methoxybenzaldehyde. One of the O atoms is disordered over two positions, O3 and O3′ [occupancies 0.854 99)/0.146 (9)]. Otherwise, the bond lengths and angles (Table 1) are within normal ranges (Allen *et al.*, 1987). The crystal packing arrangement is shown in Fig. 2.

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Experimental

1,6-Dibromohexane (12.2 g, 50 mmol) was added dropwise 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) over a period of 30 min, and the mixture was refluxed for 24 h under a nitrogen atmosphere. The solvent was removed and the resultant mixture was poured into ice–water (500 ml). The white precipitate was then isolated and recrystallized from ethanol to give the pure compound in 45% yield. Colorless single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an acetonitrile solution.

Crystal data

$C_{22}H_{26}O_6$
$M_r = 386.43$
Monoclinic, $P2_1/c$
$a = 10.867$ (5) Å
$b = 5.154$ (2) Å
$c = 18.454$ (8) Å
$\beta = 91.528$ (7)°
$V = 1033.2$ (8) Å^3
$Z = 2$
$D_x = 1.242$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1065 reflections
$\theta = 2.9$–26.1°
$\mu = 0.09$ mm^{-1}
$T = 294$ (2) K
Block, colorless
0.24 × 0.16 × 0.10 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.952$, $T_{\max} = 0.991$
4602 measured reflections
1777 independent reflections
972 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.074$
$\theta_{\max} = 25.0°$
$h = -9 \rightarrow 12$
$k = -6 \rightarrow 5$
$l = -21 \rightarrow 21$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.068$
$wR(F^2) = 0.141$
$S = 1.06$
1777 reflections
138 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0386P)^2 + 0.7149P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.16$ e Å^{-3}
$\Delta\rho_{\min} = -0.18$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C2	1.363 (4)	O2—C9	1.438 (3)
O1—C7	1.431 (4)	C8—O3	1.186 (5)
O2—C1	1.360 (4)		
C2—O1—C7	117.7 (3)	O1—C2—C3	124.7 (3)
C1—O2—C9	117.0 (2)	O1—C2—C1	114.8 (3)
O2—C1—C6	125.4 (3)	O3—C8—C5	126.5 (5)
O2—C1—C2	115.7 (3)	O2—C9—C10	108.0 (3)

The H atoms were included in calculated positions and refined using a riding-model approximation. Constrained C—H bond lengths and isotropic U parameters: 0.93 Å and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$ for aromatic CH; 0.97 Å and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$ for methylene CH_2; 0.96 Å and $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$ for methyl CH_3. Atoms H8*A*/*B* were located in a difference Fourier map and constrained [$U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$].

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*.

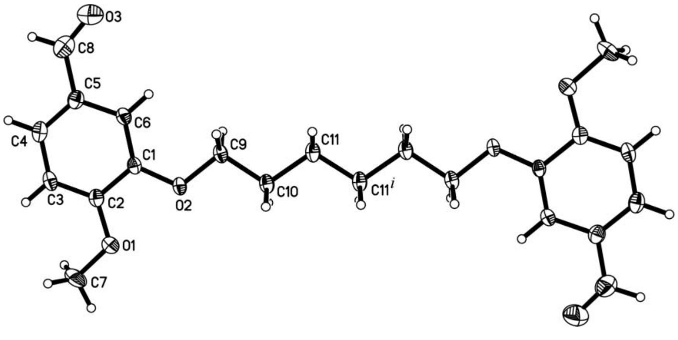

Figure 1
The structure of (I), with displacement ellipsoids for non-H atoms drawn at the 30% probability level. Only the major component of the disordered aldehyde group is shown. [Symmetry code: (i) $-x + 1, -y - 1, -z$.]

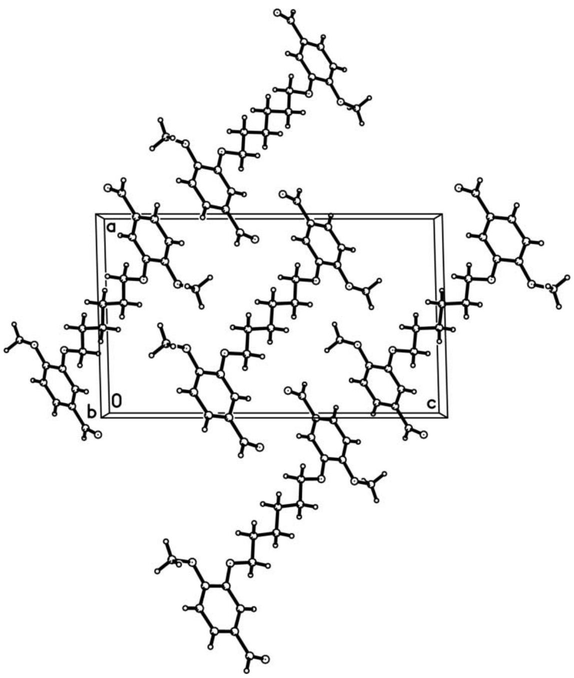

Figure 2
A view, down the *b* axis, of the packing arrangement in the crystal structure of (I). Only the major component of the disordered aldehyde group is shown.

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