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

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.002 Å
R factor = 0.041
wR factor = 0.114
Data-to-parameter ratio = 14.9

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

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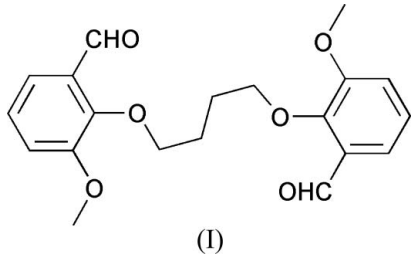
The title compound, $C_{20}H_{22}O_6$, comprises two *o*-vanillin subunits covalently linked to a central butyl chain; the molecule lies on an inversion center. The dihedral angle between the bridging butyl group and the plane through the aromatic ring is 7.13 (14)°, indicating the overall planarity of the molecule. The molecules are linked by weak C—H···O hydrogen bonds.

Comment

The ability of crown ethers to act as effective complexing agents for cations is well established (Pedersen, 1967) and represents a field of great chemical and biological significance. Consequently, significant effort has been devoted to the creation of derivatives that exhibit special affinity for specific guests (Katritzky *et al.*, 1996). As part of our interest in the molecular and ionic recognition of crown ethers, we report the synthesis and structure of the title compound, (I), used as a precursor in the preparation of crown ethers.



(I)

Molecule (I) (Fig. 1 and Table 1) is disposed about a crystallographic center of symmetry located at the mid-point of the C10—C10^{i} bond [symmetry code: (i) $-x, 2-y, -z$]. Each *o*-vanillin group (C1–C7/O1/O2) is planar, with an r.m.s. deviation of 0.019 Å and, from symmetry, the aromatic rings are parallel. Overall, the molecule is effectively planar as seen in the dihedral angle formed between the linking group and the *o*-vanillin residue of 7.13 (14)°. This observation is in contrast to the value of 55.80 (17)° reported recently for the closely related species 3-ethoxy-4-[4-(2-ethoxy-4-formyl-phenoxy)-butoxy]benzaldehyde (Han & Zhen, 2005).

A weak intermolecular C—H···O hydrogen bond is noted in the structure [C3—H3···O3: H3···O3 = 2.55 Å, C3···O3 = 3.427 (3) Å and C3—H3···O3 = 157°; symmetry code: $1+x, \frac{3}{2}-y, \frac{1}{2}+z$] which helps to consolidate the crystal packing (Fig. 2).

Experimental

To a solution of 2-hydroxy-3-methoxybenzaldehyde (15.2 g, 100 mmol) and potassium carbonate (13.8 g, 100 mmol) in aceto-

nitrile (500 ml), 1,4-dibromobutane (10.8 g, 50 mmol) was added dropwise over 30 min. The mixture was refluxed for 24 h under nitrogen. The solvent was removed and the resultant mixture poured into ice–water (500 ml). The white precipitate was isolated and recrystallized from ethanol solution to give the pure compound in 52% yield. Colorless single crystals were obtained by slow evaporation of an acetonitrile solution of (I).

Crystal data

$C_{20}H_{22}O_6$	D_x = 1.355 Mg m^{-3}
M_r = 358.38	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 1571 reflections
a = 7.068 (4) Å	θ = 2.5–24.7°
b = 16.068 (9) Å	μ = 0.10 mm^{-1}
c = 7.745 (4) Å	T = 294 (2) K
β = 93.171 (10)°	Block, colorless
V = 878.1 (8) Å^3	0.22 × 0.16 × 0.12 mm
Z = 2	

Data collection

Bruker SMART CCD area-detector diffractometer	1774 independent reflections
	1179 reflections with $I > 2\sigma(I)$
φ and ω scans	R_{int} = 0.033
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	θ_{max} = 26.3°
	$h = -8 \rightarrow 8$
T_{min} = 0.967, T_{max} = 0.988	$k = -20 \rightarrow 19$
4812 measured reflections	$l = -9 \rightarrow 6$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0497P)^2 + 0.2229P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.041	
$wR(F^2)$ = 0.114	
S = 1.02	$(\Delta/\sigma)_{max}$ = 0.001
1774 reflections	$\Delta\rho_{max}$ = 0.20 e Å^{-3}
119 parameters	$\Delta\rho_{min}$ = −0.17 e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

O1—C2	1.367 (2)	O2—C9	1.443 (2)
O1—C8	1.432 (2)	O3—C7	1.204 (2)
O2—C1	1.3793 (19)		
C2—O1—C8	117.13 (13)	O1—C2—C3	123.99 (15)
C1—O2—C9	115.68 (13)	O3—C7—C6	124.50 (18)
O2—C1—C2	121.75 (15)	O2—C9—C10	108.73 (14)
O2—C1—C6	118.46 (15)	C9—C10—C10^{i}	114.20 (18)
O1—C2—C1	116.88 (14)		

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

H atoms were included in the riding-model approximation, with C—H = 0.93 (aromatic C), 0.97 (methylene C) and 0.96 Å (methyl H), and U_{iso}(H) = 1.2U_{eq}(aromatic and methylene C) and 1.5U_{eq}(methyl 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*.

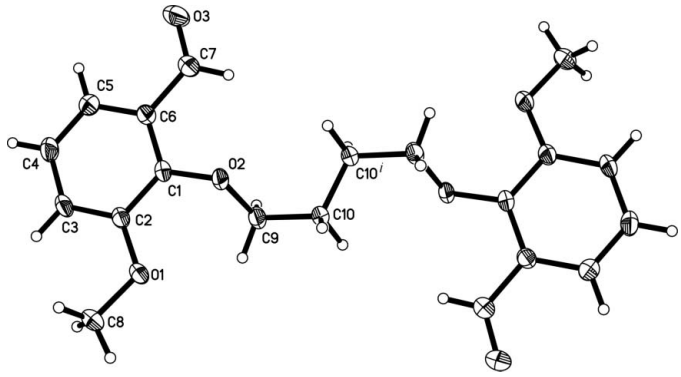


Figure 1
The structure of (I) with displacement ellipsoids for non-H atoms drawn at the 30% probability level. [Symmetry code (i): $-x, 2-y, -z$.]

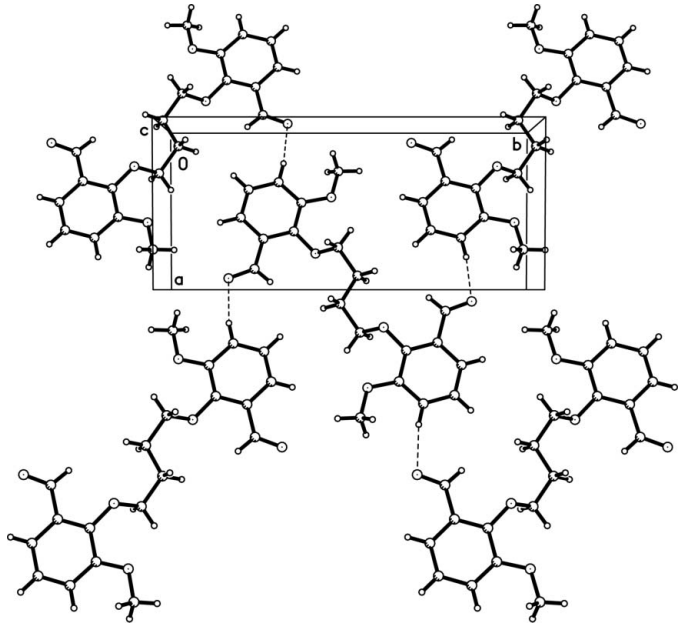


Figure 2
Intermolecular hydrogen-bonding interactions (dashed lines) in (I).

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