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Key indicators

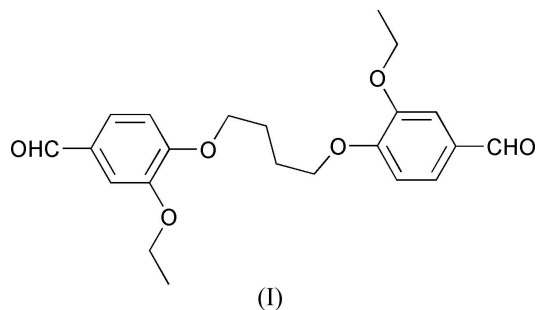
Single-crystal X-ray study
 $T = 294$ K
Mean $\sigma(\text{C}-\text{C}) = 0.002$ Å
 R factor = 0.041
 wR factor = 0.117
Data-to-parameter ratio = 16.5For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.3-Ethoxy-4-[4-(2-ethoxy-4-formylphenoxy)-
butoxy]benzaldehyde

The title compound, $\text{C}_{22}\text{H}_{26}\text{O}_6$, has a crystallographic centre of inversion at the mid-point of the central C—C bond of the aliphatic chain. The four C atoms linking the two aromatic rings are coplanar, and this plane makes a dihedral angle of $55.80(17)^\circ$ with the ethylvanillin plane. The two aromatic rings are exactly parallel by symmetry.

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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 metal cations, halide anions and small organic molecules. Much effort has been devoted to the investigation of these species (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), used as a precursor in their preparation.



A view of the molecule of (I) is shown in Fig. 1. A crystallographic centre of symmetry is located at the mid-point of the C1—C1ⁱ bond [symmetry code: (i) $-x, -y, -z$]. Each ethylvanillin group (C3—C8/C11/O1/O2) is planar, with an r.m.s. deviation for fitted atoms of 0.0170 Å. The chain of atoms C2—C1—C1ⁱ—C2ⁱ linking the two vanillin systems is exactly planar by symmetry. The results are similar to those reported for 4-[4-(4-formyl-2-methoxyphenoxy)butoxy]-3-methoxybenzaldehyde (Duan & Zhang, 2005). However, the dihedral angle between the central plane (C2—C1—C1ⁱ—C2ⁱ) and each ethylvanillin plane is $55.80(17)^\circ$, compared with $5.2(2)^\circ$ in 4-[4-(4-formyl-2-methoxyphenoxy)butoxy]-3-methoxybenzaldehyde.

Experimental

To a solution of 3-ethoxy-4-hydroxybenzaldehyde (16.6 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). A white precipitate was isolated and recrystallized from ethanol to give a pure compound in 57% yield. Colourless 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, $P2_1/n$
 $a = 4.8573$ (9) Å
 $b = 8.5362$ (16) Å
 $c = 25.368$ (5) Å
 $\beta = 95.336$ (3)°
 $V = 1047.3$ (3) Å³
 $Z = 2$

$D_x = 1.225$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 2228 reflections
 $\theta = 2.5$ – 26.2 °
 $\mu = 0.09$ mm⁻¹
 $T = 294$ (2) K
 Prism, colourless
 $0.38 \times 0.32 \times 0.22$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.960$, $T_{\max} = 0.981$
 5682 measured reflections

2131 independent reflections
 1532 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.025$
 $\theta_{\text{max}} = 26.4$ °
 $h = -5 \rightarrow 6$
 $k = -6 \rightarrow 10$
 $l = -27 \rightarrow 31$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.042$
 $wR(F^2) = 0.118$
 $S = 1.06$
 2131 reflections
 129 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0556P)^2 + 0.1434P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.17$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.16$ e Å⁻³
 Extinction correction: SHELXL97
 Extinction coefficient: 0.042 (5)

H atoms were included in calculated positions and refined using a riding-model approximation. Constrained C–H bond lengths and isotropic U parameters were as follows: aromatic C–H = 0.93 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$; methyl C–H = 0.96 Å and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$.

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.

References

Bruker (1999). SMART (Version 5.0) and SAINT (Version 4.0) for Windows NT. Bruker AXS Inc., Madison, Wisconsin, USA.

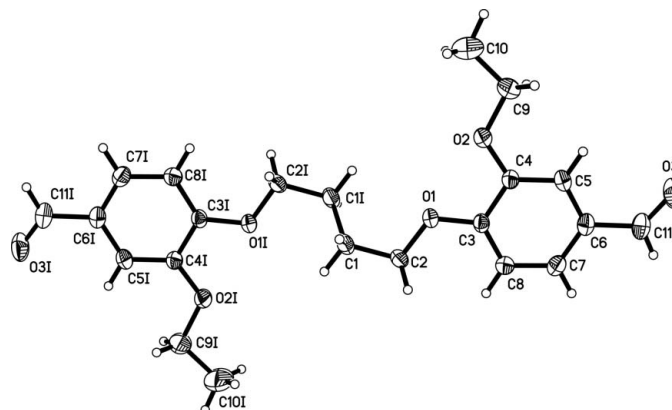


Figure 1

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

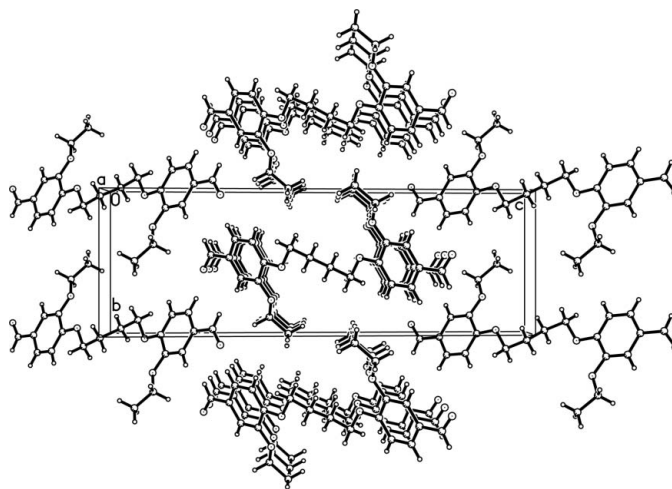


Figure 2

A view down the a axis of the packing arrangement in the crystal structure of (I).

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