3-Ethoxy-4-[3-(2-ethoxy-4-formylphenoxy)-propoxy]benzaldehyde

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The title compound, $C_{21}H_{24}O_6$, comprises two ethylvanillin subunits covalently linked to a bridging propyl chain; the molecule has a twofold rotation axis passing through the central C atom of the propyl chain. The two aromatic rings are approximately perpendicular to each other.

Comment

Since their first synthesis by Pedersen (1967), crown ethers have been shown to be of great importance in supramolecular chemistry as they can form supramolecular structures which can be used as models for studying weak interactions. Consequently, numerous derivatives of crown ethers were subsequently developed for many applications (Kim *et al.*, 1999). As part of our interest in the molecular and ionic recognition properties of crown ethers, we investigated the title compound, (I), used as a precursor in the preparation of crown ethers.

(I)
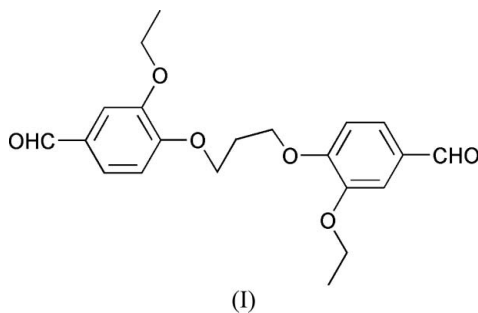
The central C11 atom of (I) (Fig. 1 and Table 1) lies on a twofold rotation axis. Atoms C1–C7 and O1–O3 of the ethylvanillin group are coplanar, with an r.m.s. deviation of 0.033 Å and this group forms a dihedral angle of 84.70 (3)° with its symmetry-related counterpart, indicating a perpendicular relationship [symmetry code: (*i*) $1 - x$, $1 - y$, z]. The dihedral angle between the plane through the C10—C11—C10^i atoms and that through the aromatic ring is 55.96 (6)°, a result in contrast to the value of 3.0 (3)° found in the closely related structure of 4-[6-(4-formyl-2-methoxyphenoxy)hexyloxy]-3-methoxybenzaldehyde (Diao *et al.*, 2005). All bond lengths and angles for (I) are within normal ranges (Table 1).

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), 1,3-dibromohexane (10.1 g, 50 mmol) was added dropwise over a period of 30 min. The mixture was refluxed for 24 h under nitrogen. The solvent was removed and the resultant mixture poured into ice–

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water (500 ml). The white precipitate was isolated and recrystallized from ethanol solution to give the pure compound in 51% yield. Colorless single crystals were obtained by slow evaporation of an acetonitrile solution of (I).

Crystal data

$C_{21}H_{24}O_6$	Mo $K\alpha$ radiation
$M_r = 372.40$	Cell parameters from 2281
Orthorhombic, $P2_12_12$	reflections
$a = 8.7778\ (16)$ Å	$\theta = 2.9–25.8°$
$b = 24.029\ (4)$ Å	$\mu = 0.09$ mm^{-1}
$c = 4.7259\ (9)$ Å	$T = 293\ (2)$ K
$V = 996.8\ (3)$ Å^3	Block, colorless
$Z = 2$	$0.24 \times 0.20 \times 0.14$ mm
$D_x = 1.241$ Mg m^{-3}	

Data collection

Bruker SMART CCD area-detector diffractometer	1227 independent reflections
	949 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.027$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$\theta_{max} = 26.4°$
	$h = -10 \rightarrow 9$
$T_{min} = 0.968$, $T_{max} = 0.987$	$k = -23 \rightarrow 30$
5593 measured reflections	$l = -5 \rightarrow 5$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0472P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.037$	$+ 0.1241P]$
$wR(F^2) = 0.100$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.09$	$(\Delta/\sigma)_{max} < 0.001$
1227 reflections	$\Delta\rho_{max} = 0.16$ e Å^{-3}
124 parameters	$\Delta\rho_{min} = -0.13$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.209 (4)	O3—C5	1.357 (3)
O2—C6	1.358 (3)	O3—C10	1.432 (3)
O2—C8	1.435 (3)		
C6—O2—C8	117.4 (2)	O2—C6—C5	114.89 (19)
C5—O3—C10	118.43 (18)	O2—C6—C7	125.2 (2)
O1—C1—C2	125.0 (3)	O2—C8—C9	108.4 (3)
O3—C5—C4	125.5 (2)	O3—C10—C11	107.59 (17)
O3—C5—C6	115.16 (19)	C10^i—C11—C10	114.5 (3)

Symmetry code: (i) $-x + 1, -y + 1, z$.

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 with $U_{iso}(H) = 1.2U_{eq}$(aromatic and methylene C) and
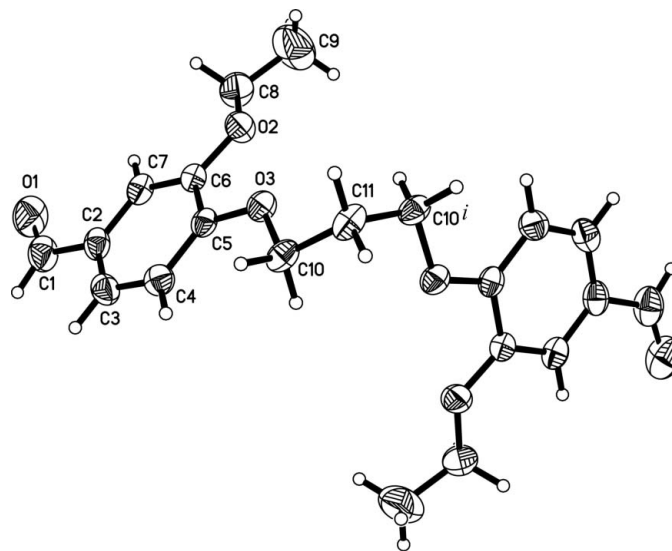

Figure 1
The molecule of (I), with displacement ellipsoids drawn at the 30% probability level [symmetry code (i): $1 - x, 1 - y, z$].

$1.5U_{eq}$(methyl C). In the absence of significant anomalous dispersion effects, Friedel pairs were averaged.

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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