

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

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Key indicators

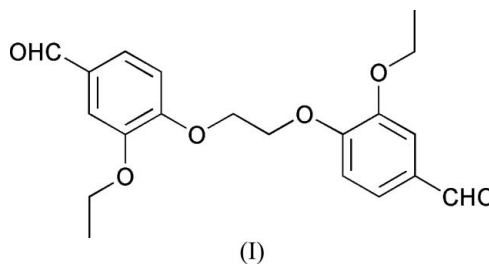
Single-crystal X-ray study
 $T = 294$ K
Mean $\sigma(\text{C}-\text{C}) = 0.002$ Å
 R factor = 0.042
 wR factor = 0.121
Data-to-parameter ratio = 15.0For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The title compound, $\text{C}_{20}\text{H}_{22}\text{O}_6$, consists of two ethylvanillin subunits covalently linked to a central C-atom chain. A crystallographic center of symmetry is located at the mid-point of the central C—C bond. The two aromatic rings are parallel to each other.

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Comment

Following the discovery of the strong affinity crown ethers have for alkali and alkaline earth metal ions (Pedersen, 1967), many studies have been carried out to understand the factors that control the thermodynamic and kinetic stability and selectivity of the resulting complexes (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).



A view of the molecule is shown in Fig. 1. A crystallographic center of symmetry is located at the mid-point of the $\text{C}7-\text{C}7^i$ bond [symmetry code: (i) $-x + 2, -y + 1, -z + 1$]. Each ethylvanillin unit ($\text{C}1-\text{C}10/\text{O}1/\text{O}3$) is planar, each with an r.m.s. deviation for fitted atoms of 0.0415 Å. The two aromatic rings in the molecule are parallel to one another. The results are similar to those for 4-[4-(4-formyl-2-methoxyphenoxy)butoxy]-3-methoxybenzaldehyde (Duan & Zhang, 2005). The bond lengths and angles are unexceptional.

Experimental

To a solution of 4-hydroxy-3-ethoxybenzaldehyde (16.6 g, 100 mmol) and potassium carbonate (13.8 g, 100 mmol) in acetonitrile (500 ml), 1,2-dibromoethane (9.4 g, 50 mmol) was added dropwise over a period of 30 min, and 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 then isolated and recrystallized from acetonitrile to give the pure compound in 57% yield. Colorless single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an acetonitrile solution.

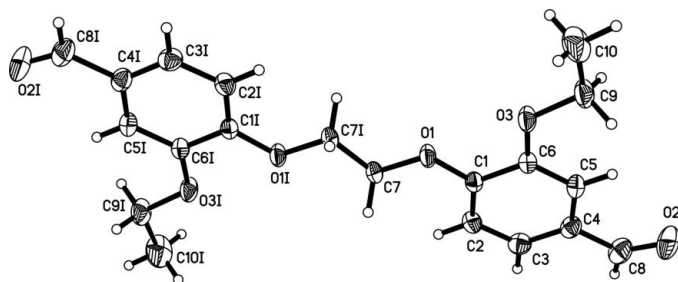


Figure 1
The structure of (I), with displacement ellipsoids for non-H atoms drawn at the 30% probability level.

Crystal data

$C_{20}H_{22}O_6$
 $M_r = 358.38$
 Monoclinic, $P2_1/c$
 $a = 4.7746$ (8) Å
 $b = 8.6976$ (16) Å
 $c = 21.557$ (4) Å
 $\beta = 96.041$ (3)°
 $V = 890.2$ (3) Å³
 $Z = 2$

$D_x = 1.337$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 2290 reflections
 $\theta = 2.3$ – 26.2 °
 $\mu = 0.10$ mm⁻¹
 $T = 294$ (2) K
 Block *needle?*, colorless
 $0.40 \times 0.14 \times 0.12$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.952$, $T_{\max} = 0.988$
 4811 measured reflections

1795 independent reflections
 1398 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.023$
 $\theta_{\text{max}} = 26.3$ °
 $h = -5 \rightarrow 3$
 $k = -10 \rightarrow 10$
 $l = -25 \rightarrow 26$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.042$
 $wR(F^2) = 0.121$
 $S = 1.04$
 1795 reflections
 120 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0537P)^2 + 0.2574P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.23$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.15$ e Å⁻³
 Extinction correction: *SHELXL97*
 Extinction coefficient: 0.032 (4)

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

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

References

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