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Key indicators

Single-crystal X-ray study

T = 294 K

Mean $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$

R factor = 0.036

wR factor = 0.104

Data-to-parameter ratio = 12.6

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

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

The title compound, $\text{C}_{18}\text{H}_{18}\text{O}_6$, is composed of two individual isovanillin subunits covalently appended to a central C-atom chain. The molecules are linked into a three-dimensional framework by weak intermolecular C—H...O hydrogen bonds and π – π interactions.

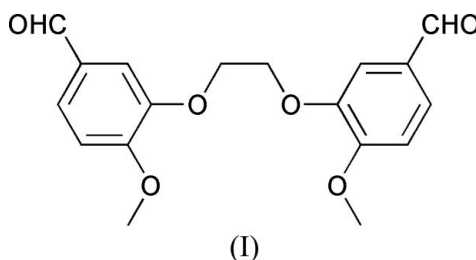
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Comment

Macrocyclic ionophores based on the discovery of the crown ethers by Pedersen (1967) have been intensively studied for applications in many areas due to their selective affinity for cations (Zhang & Buchwald, 2000).



We are interested in the molecular and ionic recognition of crown ethers. As a 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. Each individual isovanillin group is planar, with r.m.s. deviations for fitted atoms of 0.0149 (18) \AA (C1–C8/O2/O3) and 0.0309 (17) \AA (C11–C18/O4/O5), respectively. The dihedral angle between the two isovanillin planes is 58.94 (3)°. All bond lengths and angles (Table 1) are within normal ranges (Allen *et al.*, 1987).

The short distance [3.491 (2) \AA] between the centroids of benzene rings of neighboring molecules may indicate a π – π interaction, forming centrosymmetric dimers (Fig. 2). The

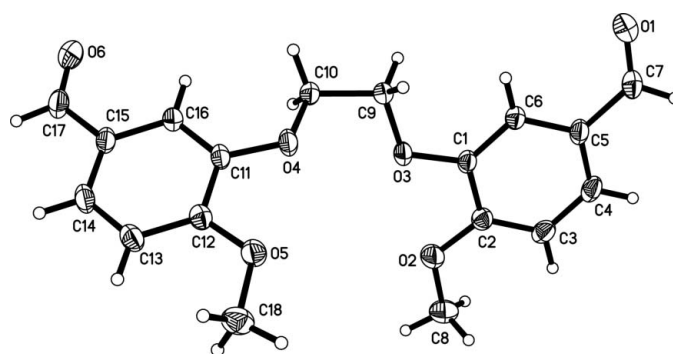


Figure 1

The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

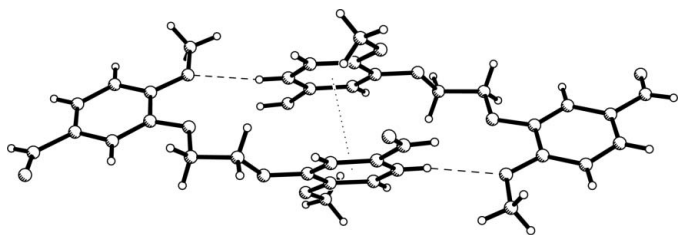


Figure 2
The centrosymmetric dimer formed by intermolecular hydrogen bonds (dashed lines) and π - π interaction (dotted line).

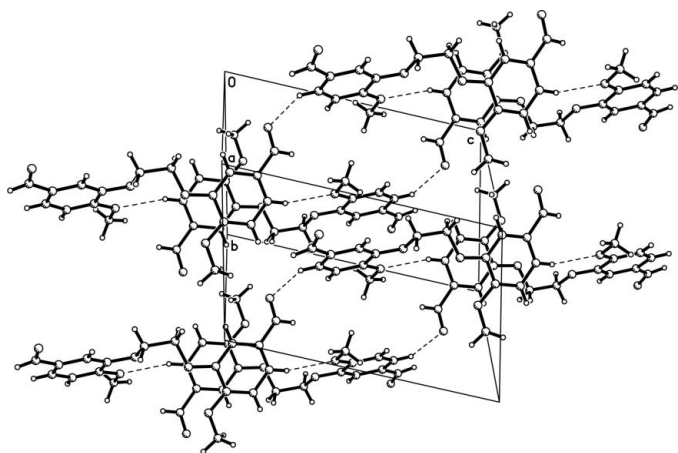


Figure 3
Packing diagram of (I); C—H...O hydrogen bonds are shown as dashed lines.

molecules are linked into a three-dimensional framework (Fig. 3) by weak intermolecular C—H...O hydrogen bonds (Table 2).

Experimental

1,2-Dibromoethane (9.4 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 resulting mixture was poured into ice-water (500 ml). The white precipitate was then isolated and recrystallized from ethanol to give the pure compound (yield 7.9 g, 48%, m.p. 441 K). Colorless single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an acetonitrile solution.

Crystal data

$C_{18}H_{18}O_6$	$Z = 2$
$M_r = 330.32$	$D_x = 1.381 \text{ Mg m}^{-3}$
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 8.1520$ (17) Å	Cell parameters from 2040 reflections
$b = 8.4524$ (18) Å	$\theta = 2.8$ – 26.2°
$c = 13.496$ (3) Å	$\mu = 0.10 \text{ mm}^{-1}$
$\alpha = 76.932$ (3)°	$T = 294$ (2) K
$\beta = 80.645$ (4)°	Block, colorless
$\gamma = 61.528$ (3)°	$0.46 \times 0.40 \times 0.24 \text{ mm}$
$V = 794.6$ (3) Å ³	

Data collection

Bruker SMART CCD area-detector diffractometer	2774 independent reflections
φ and ω scans	2115 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$R_{\text{int}} = 0.019$
$T_{\text{min}} = 0.940$, $T_{\text{max}} = 0.975$	$\theta_{\text{max}} = 25.0^\circ$
4037 measured reflections	$h = -8 \rightarrow 9$
	$k = -10 \rightarrow 9$
	$l = -16 \rightarrow 15$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0524P)^2 + 0.1439P]$
$R[F^2 > 2\sigma(F^2)] = 0.036$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.104$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.02$	$\Delta\rho_{\text{max}} = 0.16 \text{ e \AA}^{-3}$
2774 reflections	$\Delta\rho_{\text{min}} = -0.17 \text{ e \AA}^{-3}$
220 parameters	Extinction correction: SHELXL97
H-atom parameters constrained	Extinction coefficient: 0.209 (10)

Table 1

Selected geometric parameters (Å, °).

O1—C7	1.209 (2)	O4—C11	1.3687 (17)
O2—C2	1.3584 (19)	O4—C10	1.4251 (18)
O2—C8	1.421 (2)	O5—C12	1.3575 (15)
O3—C1	1.3680 (17)	O5—C18	1.428 (2)
O3—C9	1.4276 (18)	O6—C17	1.206 (2)
C2—O2—C8	118.46 (13)	O3—C1—C6	125.38 (14)
C1—O3—C9	116.95 (11)	O3—C1—C2	114.86 (13)
C11—O4—C10	117.10 (11)	O2—C2—C3	125.15 (15)
C12—O5—C18	117.52 (13)	O2—C2—C1	114.86 (13)

Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C4—H4...O6 ⁱ	0.93	2.57	3.314 (2)	137
C7—H7...O5 ⁱⁱ	0.93	2.53	3.405 (2)	157
C14—H14...O2 ⁱⁱⁱ	0.93	2.50	3.379 (2)	158

Symmetry codes: (i) $x, y - 1, z + 1$; (ii) $-x, -y + 1, -z + 1$; (iii) $-x + 1, -y + 1, -z$.

H atoms were positioned geometrically [0.93 (CH), 0.97 (CH₂) and 0.96 Å (CH₃)] and constrained to ride on their parent atoms with $U_{\text{iso}}(\text{H})$ values of 1.2 (1.5 for methyl) times $U_{\text{eq}}(\text{carrier atom})$.

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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