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Key indicators

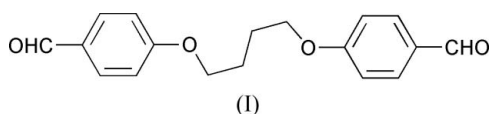
Single-crystal X-ray study
 $T = 294$ K
Mean $\sigma(\text{C}-\text{C}) = 0.002$ Å
 R factor = 0.039
 wR factor = 0.118
Data-to-parameter ratio = 15.3For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

4-[4-(4-Formylphenoxy)butoxy]benzaldehyde

The molecule of the title compound, $\text{C}_{18}\text{H}_{18}\text{O}_4$, lies on crystallographic center of symmetry. The benzaldehyde group makes a dihedral angle of $62.82(16)^\circ$ with the four C atoms in the central chain. The two aromatic ring are parallel to each other by symmetry.

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. Consequently, these species have been used to study their molecular recognition for special guest molecules and cations (Habata *et al.*, 1996; Zhang & Buchwald, 2000). 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.



In (I) (Fig. 1), a crystallographic center of symmetry is located at the mid-point of the central $\text{C9}-\text{C9}^i$ bond [symmetry code: (i) $-x, -y, -z$]. The benzaldehyde group ($\text{C1}-\text{C7}/\text{O1}/\text{O2}$) is planar, with an r.m.s. deviation for fitted atoms of 0.0353 Å. The chain of atoms $\text{C8}-\text{C9}-\text{C9}^i-\text{C8}^i$ linking the two aromatic systems is exactly planar by symmetry. The results are similar to that reported recently for the closely related species 4-[6-(4-formyl-2-methoxyphenoxy)hexyloxy]-3-methoxybenzaldehyde (Diao *et al.*, 2005) and 4-[4-(4-formyl-2-methoxyphenoxy)butoxy]-3-methoxybenzaldehyde (Duan & Zhang, 2005). However, the dihedral angle between the bridge plane ($\text{C8}/\text{C9}/\text{C9}^i/\text{C8}^i$) and the benzaldehyde group plane is $62.82(16)^\circ$, in comparison with $3.0(3)^\circ$ and $55.80(17)^\circ$ in 4-[6-(4-formyl-2-methoxyphenoxy)hexyloxy]-3-methoxybenzaldehyde and 4-[4-(4-formyl-2-methoxy-

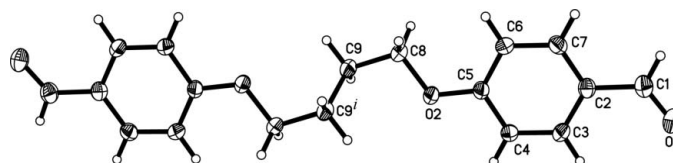


Figure 1

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

phenoxy)butoxy]-3-methoxybenzaldehyde, respectively. All bond lengths and angles (Table 1) are within normal ranges (Allen *et al.*, 1987). The crystal packing arrangement, a zigzag pattern, is shown in Fig. 2.

Experimental

To a solution of 4-hydroxybenzaldehyde (12.2 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). The white precipitate was isolated and recrystallized from ethanol to give the pure compound in 46% yield. Colorless single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an acetonitrile solution.

Crystal data

$C_{18}H_{18}O_4$	$D_x = 1.313 \text{ Mg m}^{-3}$
$M_r = 298.32$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 1332 reflections
$a = 7.988$ (2) Å	$\theta = 2.9\text{--}26.5^\circ$
$b = 6.6635$ (16) Å	$\mu = 0.09 \text{ mm}^{-1}$
$c = 14.260$ (4) Å	$T = 294$ (2) K
$\beta = 96.354$ (4)°	Block, colorless
$V = 754.4$ (3) Å ³	$0.30 \times 0.26 \times 0.18 \text{ mm}$
$Z = 2$	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	1547 independent reflections
φ and ω scans	1034 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$R_{\text{int}} = 0.027$
$T_{\text{min}} = 0.960$, $T_{\text{max}} = 0.984$	$\theta_{\text{max}} = 26.5^\circ$
4120 measured reflections	$h = -6 \rightarrow 10$
	$k = -8 \rightarrow 8$
	$l = -17 \rightarrow 16$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0611P)^2 + 0.0823P]$
$R[F^2 > 2\sigma(F^2)] = 0.039$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.118$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.00$	$\Delta\rho_{\text{max}} = 0.16 \text{ e } \text{Å}^{-3}$
1547 reflections	$\Delta\rho_{\text{min}} = -0.14 \text{ e } \text{Å}^{-3}$
101 parameters	Extinction correction: SHELXL97
H-atom parameters constrained	Extinction coefficient: 0.053 (7)

Table 1

Selected geometric parameters (Å, °).

O1—C1	1.2110 (19)	O2—C8	1.4346 (17)
O2—C5	1.3582 (17)		
C5—O2—C8	118.84 (11)	O2—C5—C4	115.31 (13)
O1—C1—C2	125.16 (17)	O2—C8—C9	107.58 (12)
O2—C5—C6	124.70 (13)		

The H atoms were included in calculated positions and refined using a riding model approximation, with C—H = 0.93 Å and $U_{\text{iso}}(\text{H})$

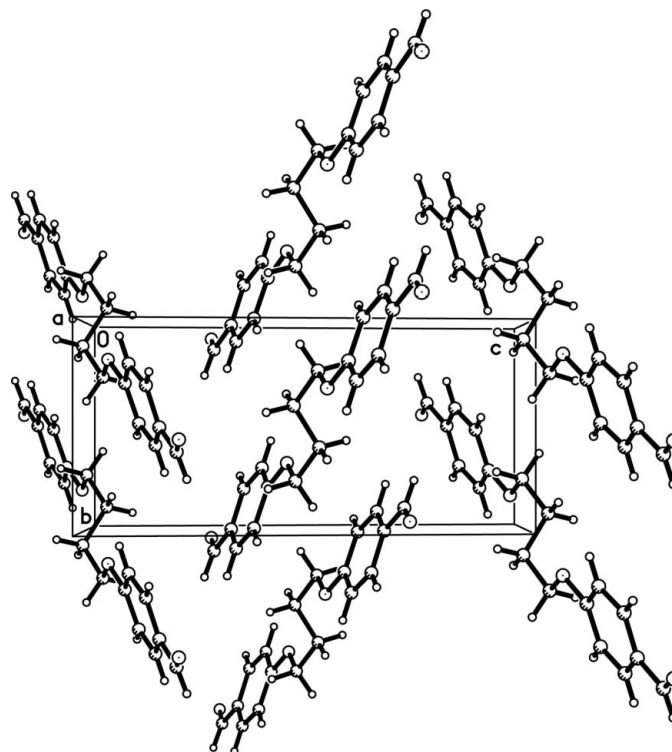


Figure 2

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

$= 1.2U_{\text{eq}}(\text{C})$ for aromatic CH; C—H = 0.97 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for methylene CH₂.

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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