Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

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

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.003 Å
R factor = 0.044
wR factor = 0.115
Data-to-parameter ratio = 15.3

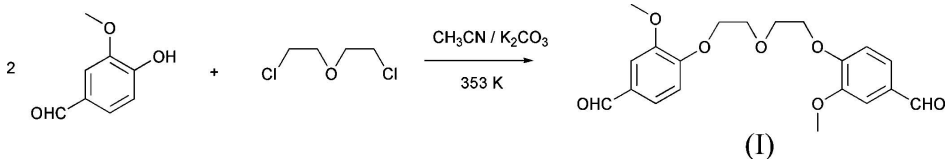
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 30 August 2005
Accepted 17 October 2005
Online 22 October 2005

The title compound, $C_{20}H_{22}O_7$, was prepared by reaction of 4-hydroxy-3-methoxybenzaldehyde and 1-chloro-2-(2-chloroethoxy)ethane. Intermolecular C—H···O hydrogen-bonding contributes to the stability of the structure in the solid state.

Comment

The synthesis of new and designed crystal structures continues to be an important area of research for chemists (Tynan *et al.*, 2005; Parashar *et al.*, 1988). One of the aims of crystal engineering is to establish control over the preparation of crystalline solid materials so that their architecture and properties are predictable. In order to investigate their crystal structures, which will provide useful information for their physical and chemical properties, in the present study we report the synthesis and molecular structure of the title compound, (I). It was synthesized by the reaction of 4-hydroxy-3-methoxybenzaldehyde with 1-chloro-2-(2-chloroethoxy)ethane.



(I)

The molecular structure of (I) is shown in Fig. 1. Some bond lengths and bond angles are listed in Table 1. Both vanillin groups are essentially planar, with an r.m.s. deviation of 0.0389 Å for C1–C7/O1–O3 and 0.0541 Å for C13–C19/O5–O7, and the dihedral angle between the two vanillin groups is 54.67 (3)°. A weak C—H···O hydrogen bond (Table 2) contributes to the stabilization of the solid-state structure.

Experimental

1-Chloro-2-(2-chloroethoxy)ethane (7.1 g, 50 mmol) was added (30 min) to a solution of 4-hydroxy-3-methoxybenzaldehyde (15.2 g, 100 mmol) and potassium carbonate (13.8 g, 100 mmol) in acetonitrile (500 ml). The mixture was refluxed for 24 h under nitrogen. The solvent was removed and the resultant oil was poured into ice–water (500 ml). The white precipitate was isolated and recrystallized from ethanol as pure (I) in 41% yield. Colorless single crystals were obtained by slow evaporation of an acetonitrile solution.

Crystal data

$C_{20}H_{22}O_7$
M_r = 374.38
Monoclinic, $P2_1/c$
a = 8.0786 (16) Å
b = 30.476 (6) Å
c = 8.4775 (17) Å
β = 117.817 (3)°
V = 1846.0 (6) Å^3
Z = 4
D_x = 1.347 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2167 reflections
θ = 2.7–25.4°
μ = 0.10 mm^{-1}
T = 294 (2) K
Block, colorless
0.34 × 0.30 × 0.26 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.956, T_{max} = 0.974
10313 measured reflections
3755 independent reflections
1993 reflections with $I > 2\sigma(I)$
R_{int} = 0.043
θ_{max} = 26.3°
$h = -10 \rightarrow 10$
$k = -30 \rightarrow 38$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.115
S = 1.00
3755 reflections
246 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0508P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C7	1.207 (3)	O4—C10	1.420 (2)
O2—C6	1.360 (2)	O5—C13	1.359 (2)
O2—C8	1.427 (2)	O5—C12	1.433 (2)
O3—C1	1.360 (2)	O6—C18	1.368 (2)
O3—C9	1.435 (2)	O6—C20	1.425 (2)
O4—C11	1.414 (2)	O7—C19	1.209 (3)
C6—O2—C8	116.75 (16)	C13—O5—C12	117.15 (15)
C1—O3—C9	117.03 (14)	C18—O6—C20	116.46 (15)
C11—O4—C10	111.58 (15)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C9—H9B···O7^{i}	0.97	2.60	3.527 (3)	160

Symmetry code: (i) $-x+1, -y, -z+2$.

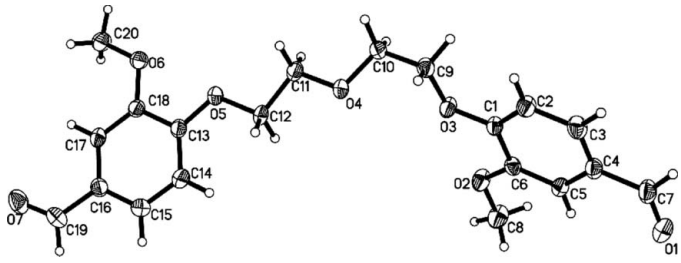


Figure 1
The structure of (I), with displacement ellipsoids for non-H atoms drawn at the 30% probability level.

H atoms bonded to C atoms were included in calculated positions [C—H = 0.93–0.97 Å] and refined using a riding-model approximation, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C_{methyl}).

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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