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1,2-Bis(2-methoxy-6-formylphenoxy)-ethane

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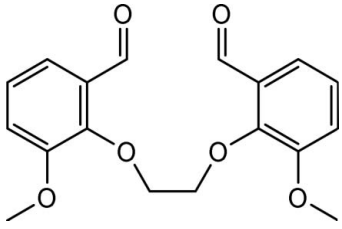
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.005 Å; R factor = 0.060; wR factor = 0.141; data-to-parameter ratio = 12.8.

In the title compound [systematic name: 3,3′-dimethoxy-2,2′-(ethane-1,2-diyldioxy)dibenzaldehyde], $C_{18}H_{18}O_6$, prepared from 1,2-dibromoethane and *ortho*-vanillin in the presence of sodium carbonate, the two vanillin units are linked *via* a CH_2–CH_2 bridge. The two benzene rings are inclined at a dihedral angle of 41.6 (5)°.

Related literature

For the use of open chain-ionophores, including polyethylene glycols, as microbiological agents and in ion binding, see: Valeur *et al.* (1992); Tuncer & Erk (2000). For the synthesis, see: Tuncer & Erk (2000). For related structures, see: Higham *et al.* (2010).



Experimental

Crystal data

$C_{18}H_{18}O_6$
M_r = 330.32
Monoclinic, $P2_1/n$
a = 4.161 (3) Å
b = 30.155 (18) Å
c = 12.934 (8) Å
β = 96.817 (7)°
V = 1611.6 (17) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 296 K
0.12 × 0.10 × 0.08 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
T_{min} = 0.988, T_{max} = 0.992
14774 measured reflections
2815 independent reflections
1519 reflections with $I > 2\sigma(I)$
R_{int} = 0.096

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.141
S = 1.00
2815 reflections
220 parameters
H-atom parameters not refined
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT-Plus* (Bruker, 2004); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ5080).

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Tuncer, H. & Erk, C. (2000). *Dyes Pigments*, **44**, 81–86.
Valeur, B., Pouget, J., Bourson, J., Kaschke, M. & Ernsting, N. P. (1992). *J. Phys. Chem.* **96**, 6545–6549.

supporting information

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1,2-Bis(2-methoxy-6-formylphenoxy)ethane

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S1. Comment

Open chain ionophores including polyethylene glycols have proved to be extremely interesting compounds due to their versatility as microbiological agents and in ion binding (Valeur *et al.*, 1992). Their extraordinary capacity for ion binding has attracted much attention in view of their acyclic and bulky structures. For example, aromatic carbonyl derivatives of glycols such as 1,2-bis(2-methoxy-6- formylphenoxy)ethane were investigated to determine the role of sodium ions using steady state fluorescence spectroscopy (Tuncer & Erk, 2000). 1,2-Bis(2-methoxy-6-formylphenoxy)ethane and its analogues have also been used in the synthesis of dienone-ether macrocycles displaying molecular and supramolecular diversity (Higham *et al.*, 2010). Herein we present the single-crystal structure of the title compound.

S2. Experimental

The title compound was prepared as reported in the literature (Tuncer & Erk, 2000). Single crystals suitable for X-ray diffraction measurement was obtained by slow evaporation of the solution in acetone [m.p. 391–393 K; literature value: 392 K (Tuncer & Erk, 2000)].

S3. Refinement

All H atoms were placed at calculated positions and refined using a riding model approximation, with C—H = 0.93–0.97 Å and with U_{iso}(H) = 1.2U_{eq}(C) for aromatic and CH_2 groups and = 1.5U_{eq}(C) for methyl groups.

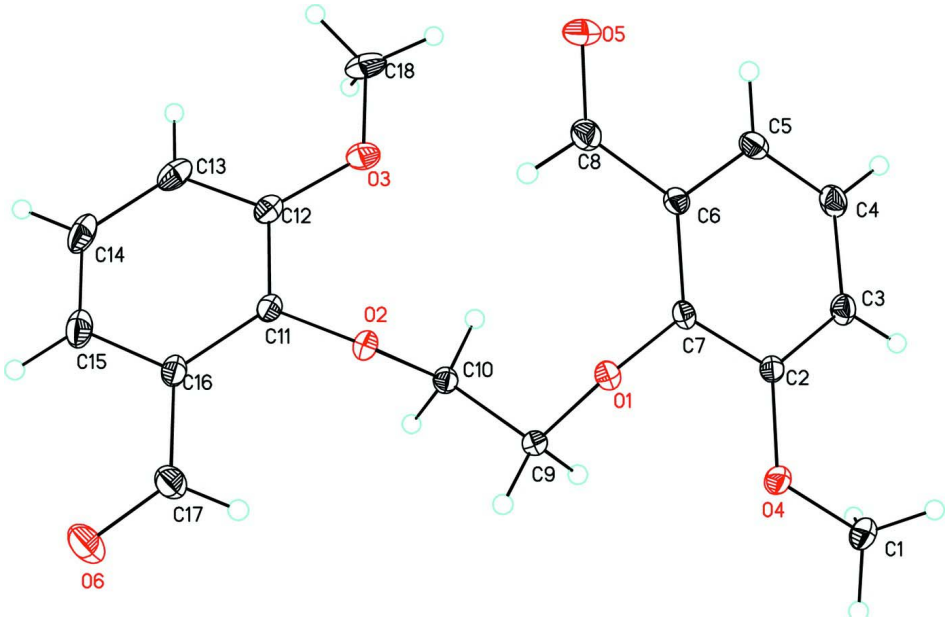

Figure 1

A view of the molecule of the title compound. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

3,3′-Dimethoxy-2,2′-(ethane-1,2-diyldioxy)dibenzaldehyde

Crystal data

$C_{18}H_{18}O_6$
$M_r = 330.32$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 4.161$ (3) Å
$b = 30.155$ (18) Å
$c = 12.934$ (8) Å
$\beta = 96.817$ (7)°
$V = 1611.6$ (17) Å^3
$Z = 4$

$F(000) = 696$
$D_x = 1.361$ Mg m^{-3}
Melting point = 391–393 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1432 reflections
$\theta = 2.6$–19.0°
$\mu = 0.10$ mm^{-1}
$T = 296$ K
Block, colorless
0.12 × 0.10 × 0.08 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
$T_{min} = 0.988$, $T_{max} = 0.992$

14774 measured reflections
2815 independent reflections
1519 reflections with $I > 2\sigma(I)$
$R_{int} = 0.096$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.6°$
$h = -4 \rightarrow 4$
$k = -35 \rightarrow 35$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.060$
$wR(F^2) = 0.141$

$S = 1.00$
2815 reflections
220 parameters
0 restraints

Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters not refined

$w = 1/[\sigma^2(F_o^2) + (0.059P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}
Extinction correction: *SHELXL*, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0130 (19)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.3389 (9)	0.03026 (11)	0.5936 (2)	0.0647 (10)
H1A	0.5683	0.0344	0.5956	0.097*
H1B	0.2895	−0.0008	0.5895	0.097*
H1C	0.2676	0.0424	0.6556	0.097*
C2	0.2236 (7)	0.09709 (10)	0.4969 (2)	0.0448 (8)
C3	0.4057 (8)	0.12260 (11)	0.5701 (2)	0.0554 (9)
H3	0.5160	0.1092	0.6286	0.066*
C4	0.4264 (8)	0.16785 (12)	0.5577 (3)	0.0622 (10)
H4	0.5543	0.1845	0.6071	0.075*
C5	0.2608 (8)	0.18851 (11)	0.4733 (3)	0.0547 (9)
H5	0.2717	0.2192	0.4666	0.066*
C6	0.0754 (7)	0.16342 (10)	0.3975 (2)	0.0453 (8)
C7	0.0586 (7)	0.11786 (10)	0.4081 (2)	0.0420 (8)
C8	−0.1070 (8)	0.18701 (12)	0.3090 (3)	0.0612 (10)
H8	−0.2209	0.1703	0.2564	0.073*
C9	0.0325 (8)	0.06410 (9)	0.2724 (2)	0.0461 (8)
H9A	−0.1130	0.0407	0.2449	0.055*
H9B	0.2118	0.0504	0.3156	0.055*
C10	0.1595 (7)	0.08728 (10)	0.1841 (2)	0.0438 (8)
H10A	0.2860	0.1129	0.2094	0.053*
H10B	0.2979	0.0675	0.1501	0.053*
C11	−0.0342 (7)	0.11450 (10)	0.0157 (2)	0.0440 (8)
C12	0.1182 (8)	0.15543 (11)	0.0050 (3)	0.0532 (9)
C13	0.1759 (9)	0.16920 (13)	−0.0933 (3)	0.0714 (11)
H13	0.2742	0.1965	−0.1016	0.086*
C14	0.0882 (11)	0.14262 (16)	−0.1793 (3)	0.0814 (13)
H14	0.1269	0.1523	−0.2450	0.098*
C15	−0.0523 (10)	0.10304 (14)	−0.1689 (3)	0.0747 (12)

H15	−0.1064	0.0854	−0.2274	0.090*
C16	−0.1190 (8)	0.08786 (11)	−0.0705 (2)	0.0526 (9)
C17	−0.2777 (9)	0.04495 (12)	−0.0610 (3)	0.0703 (11)
H17	−0.3183	0.0360	0.0050	0.084*
C18	0.3642 (9)	0.21970 (10)	0.0892 (3)	0.0809 (12)
H18A	0.5724	0.2134	0.0677	0.121*
H18B	0.3920	0.2335	0.1565	0.121*
H18C	0.2469	0.2393	0.0398	0.121*
O1	−0.1379 (5)	0.09317 (6)	0.33650 (14)	0.0448 (6)
O2	−0.1126 (5)	0.10109 (6)	0.11105 (14)	0.0446 (6)
O3	0.1872 (6)	0.17924 (7)	0.09468 (19)	0.0640 (7)
O4	0.1771 (5)	0.05236 (7)	0.50439 (15)	0.0560 (6)
O5	−0.1154 (7)	0.22701 (8)	0.30153 (19)	0.0849 (9)
O6	−0.3593 (8)	0.02042 (9)	−0.1333 (2)	0.1053 (11)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.080 (3)	0.067 (2)	0.045 (2)	0.001 (2)	−0.0003 (19)	0.0127 (18)
C2	0.052 (2)	0.045 (2)	0.0370 (18)	−0.0069 (17)	0.0063 (15)	−0.0016 (16)
C3	0.059 (2)	0.066 (2)	0.039 (2)	−0.0036 (19)	−0.0018 (17)	−0.0019 (18)
C4	0.069 (3)	0.067 (3)	0.050 (2)	−0.018 (2)	0.0009 (19)	−0.0127 (19)
C5	0.060 (2)	0.047 (2)	0.058 (2)	−0.0068 (17)	0.0130 (19)	−0.0113 (18)
C6	0.050 (2)	0.044 (2)	0.043 (2)	0.0017 (16)	0.0124 (16)	−0.0041 (16)
C7	0.0433 (19)	0.050 (2)	0.0340 (18)	−0.0050 (16)	0.0084 (15)	−0.0083 (16)
C8	0.068 (2)	0.061 (3)	0.056 (2)	0.013 (2)	0.0077 (19)	−0.0067 (19)
C9	0.061 (2)	0.0381 (18)	0.0395 (18)	0.0023 (16)	0.0051 (16)	0.0010 (14)
C10	0.0451 (19)	0.0461 (19)	0.0399 (18)	0.0039 (15)	0.0038 (15)	0.0001 (15)
C11	0.0456 (19)	0.049 (2)	0.0382 (19)	0.0093 (16)	0.0081 (15)	0.0059 (16)
C12	0.053 (2)	0.055 (2)	0.053 (2)	0.0084 (18)	0.0105 (18)	0.0120 (18)
C13	0.070 (3)	0.072 (3)	0.076 (3)	0.010 (2)	0.024 (2)	0.032 (2)
C14	0.093 (3)	0.108 (4)	0.046 (3)	0.025 (3)	0.023 (2)	0.027 (3)
C15	0.088 (3)	0.094 (3)	0.042 (2)	0.026 (3)	0.006 (2)	0.001 (2)
C16	0.057 (2)	0.061 (2)	0.0390 (19)	0.0138 (18)	0.0035 (16)	0.0029 (18)
C17	0.082 (3)	0.062 (3)	0.064 (3)	0.008 (2)	−0.002 (2)	−0.014 (2)
C18	0.069 (3)	0.046 (2)	0.123 (3)	−0.009 (2)	−0.006 (2)	0.024 (2)
O1	0.0467 (13)	0.0490 (13)	0.0387 (12)	−0.0020 (11)	0.0051 (10)	−0.0055 (10)
O2	0.0433 (12)	0.0535 (14)	0.0371 (12)	0.0007 (10)	0.0055 (10)	0.0064 (10)
O3	0.0704 (16)	0.0468 (14)	0.0756 (18)	−0.0126 (12)	0.0120 (13)	0.0075 (13)
O4	0.0708 (16)	0.0526 (15)	0.0419 (13)	−0.0078 (12)	−0.0044 (11)	0.0059 (10)
O5	0.122 (2)	0.0432 (16)	0.086 (2)	0.0179 (15)	−0.0016 (16)	−0.0016 (13)
O6	0.138 (3)	0.083 (2)	0.089 (2)	0.0013 (18)	−0.012 (2)	−0.0349 (17)

Geometric parameters (Å, º)

C1—O4	1.430 (3)	C10—O2	1.447 (3)
C1—H1A	0.9600	C10—H10A	0.9700
C1—H1B	0.9600	C10—H10B	0.9700

C1—H1C	0.9600	C11—O2	1.373 (3)
C2—O4	1.368 (4)	C11—C16	1.385 (4)
C2—C3	1.376 (4)	C11—C12	1.402 (4)
C2—C7	1.413 (4)	C12—O3	1.366 (4)
C3—C4	1.378 (4)	C12—C13	1.385 (4)
C3—H3	0.9300	C13—C14	1.384 (5)
C4—C5	1.369 (4)	C13—H13	0.9300
C4—H4	0.9300	C14—C15	1.343 (5)
C5—C6	1.396 (4)	C14—H14	0.9300
C5—H5	0.9300	C15—C16	1.412 (4)
C6—C7	1.383 (4)	C15—H15	0.9300
C6—C8	1.479 (4)	C16—C17	1.464 (5)
C7—O1	1.378 (3)	C17—O6	1.209 (4)
C8—O5	1.210 (4)	C17—H17	0.9300
C8—H8	0.9300	C18—O3	1.431 (3)
C9—O1	1.449 (3)	C18—H18A	0.9600
C9—C10	1.489 (4)	C18—H18B	0.9600
C9—H9A	0.9700	C18—H18C	0.9600
C9—H9B	0.9700		

O4—C1—H1A	109.5	C9—C10—H10A	110.0
O4—C1—H1B	109.5	O2—C10—H10B	110.0
H1A—C1—H1B	109.5	C9—C10—H10B	110.0
O4—C1—H1C	109.5	H10A—C10—H10B	108.4
H1A—C1—H1C	109.5	O2—C11—C16	119.1 (3)
H1B—C1—H1C	109.5	O2—C11—C12	120.4 (3)
O4—C2—C3	125.0 (3)	C16—C11—C12	120.4 (3)
O4—C2—C7	115.8 (3)	O3—C12—C13	125.5 (3)
C3—C2—C7	119.1 (3)	O3—C12—C11	115.5 (3)
C2—C3—C4	120.7 (3)	C13—C12—C11	119.0 (3)
C2—C3—H3	119.7	C14—C13—C12	120.4 (4)
C4—C3—H3	119.7	C14—C13—H13	119.8
C5—C4—C3	120.7 (3)	C12—C13—H13	119.8
C5—C4—H4	119.6	C15—C14—C13	120.7 (4)
C3—C4—H4	119.6	C15—C14—H14	119.6
C4—C5—C6	119.8 (3)	C13—C14—H14	119.6
C4—C5—H5	120.1	C14—C15—C16	120.9 (4)
C6—C5—H5	120.1	C14—C15—H15	119.6
C7—C6—C5	120.0 (3)	C16—C15—H15	119.6
C7—C6—C8	121.8 (3)	C11—C16—C15	118.6 (3)
C5—C6—C8	118.2 (3)	C11—C16—C17	121.3 (3)
O1—C7—C6	120.2 (3)	C15—C16—C17	120.1 (3)
O1—C7—C2	119.9 (3)	O6—C17—C16	124.3 (4)
C6—C7—C2	119.7 (3)	O6—C17—H17	117.8
O5—C8—C6	123.2 (3)	C16—C17—H17	117.8
O5—C8—H8	118.4	O3—C18—H18A	109.5
C6—C8—H8	118.4	O3—C18—H18B	109.5
O1—C9—C10	113.4 (2)	H18A—C18—H18B	109.5

O1—C9—H9A	108.9	O3—C18—H18C	109.5
C10—C9—H9A	108.9	H18A—C18—H18C	109.5
O1—C9—H9B	108.9	H18B—C18—H18C	109.5
C10—C9—H9B	108.9	C7—O1—C9	114.8 (2)
H9A—C9—H9B	107.7	C11—O2—C10	114.8 (2)
O2—C10—C9	108.3 (2)	C12—O3—C18	117.6 (3)
O2—C10—H10A	110.0	C2—O4—C1	117.4 (2)