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

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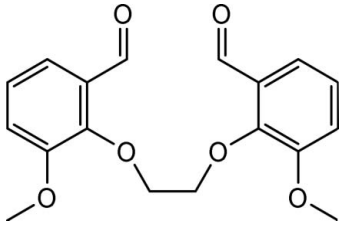
Key indicators: single-crystal X-ray study; $T$ = 296 K; mean $\sigma$(C–C) = 0.005 Å; $R$ factor = 0.060; $wR$ factor = 0.141; data-to-parameter ratio = 12.8.

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
$\beta$ = 96.817 (7)°
$V$ = 1611.6 (17) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 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).

## References

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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.

# supplementary materials

*Acta Cryst.* (2011). E**67**, o306 [ **doi:10.1107/S1600536810054085** ]

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

**H. Li, L. Cai, D. Chen, J. Li and Y. Chen**

## 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.

## 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)].

## 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) = 1.2$U_{eq}$(C) for aromatic and $CH_2$ groups and = 1.5$U_{eq}$(C) for methyl groups.

## Figures

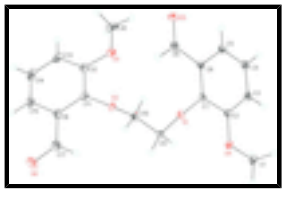


Fig. 1. A view of the molecule of the title compound. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

## 3,3'-Dimethoxy-2,2'-(ethane-1,2-diyldioxy)dibenzaldehyde

*Crystal data*

| | |
|---|---|
| $C_{18}H_{18}O_6$ | $F$(000) = 696 |
| $M_r$ = 330.32 | $D_x$ = 1.361 Mg m$^{-3}$ |
| Monoclinic, $P2_1/n$ | Melting point = 391–393 K |
| Hall symbol: -P 2yn | Mo $K\alpha$ radiation, λ = 0.71073 Å |
| $a$ = 4.161 (3) Å | Cell parameters from 1432 reflections |

| | |
|---|---|
| $b$ = 30.155 (18) Å | θ = 2.6–19.0° |
| $c$ = 12.934 (8) Å | μ = 0.10 mm$^{-1}$ |
| β = 96.817 (7)° | $T$ = 296 K |
| $V$ = 1611.6 (17) Å$^3$ | Block, colorless |
| $Z$ = 4 | 0.12 × 0.10 × 0.08 mm |

*Data collection*

| | |
|---|---|
| Bruker APEXII CCD diffractometer | 2815 independent reflections |
| Radiation source: fine-focus sealed tube | 1519 reflections with $I > 2\sigma(I)$ |
| graphite | $R_{int}$ = 0.096 |
| φ and ω scans | $\theta_{max}$ = 25.0°, $\theta_{min}$ = 2.6° |
| Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004) | $h$ = −4→4 |
| $T_{min}$ = 0.988, $T_{max}$ = 0.992 | $k$ = −35→35 |
| 14774 measured reflections | $l$ = −15→15 |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)]$ = 0.060 | H-atom parameters not refined |
| $wR(F^2)$ = 0.141 | $w = 1/[\sigma^2(F_o^2) + (0.059P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $S$ = 1.00 | $(\Delta/\sigma)_{max}$ = 0.001 |
| 2815 reflections | $\Delta\rho_{max}$ = 0.18 e Å$^{-3}$ |
| 220 parameters | $\Delta\rho_{min}$ = −0.18 e Å$^{-3}$ |
| 0 restraints | Extinction correction: *SHELXL*, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$ |
| Primary atom site location: structure-invariant direct methods | 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 (Å$^2$)*

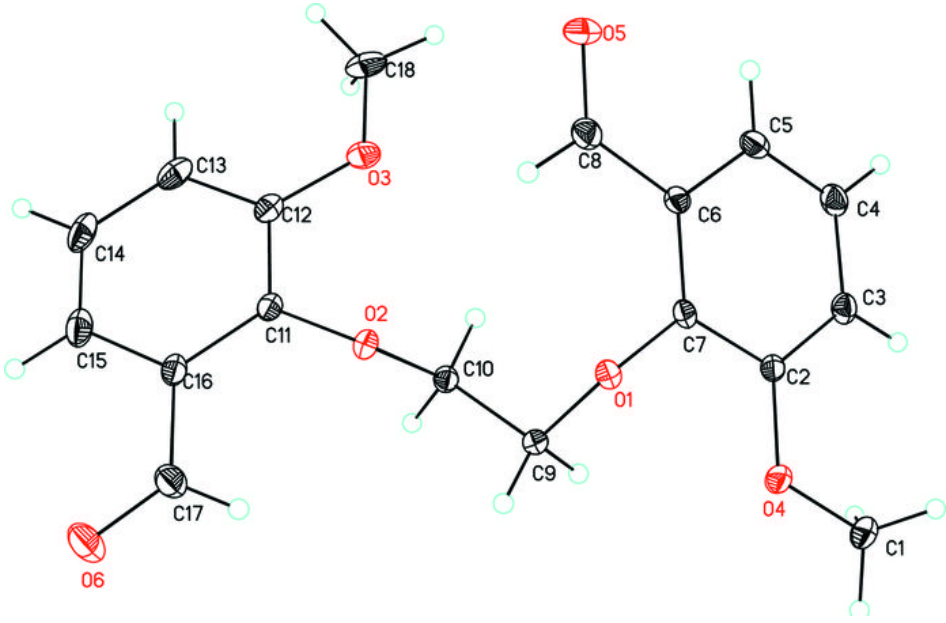
| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.080 (3) | 0.067 (2) | 0.045 (2) | 0.001 (2) | −0.0003 (19) | 0.0127 (18) |
| C2 | 0.052 (2) | 0.045 (2) | 0.0370 (18) | −0.0069 (17) | 0.0063 (15) | −0.0016 (16) |
| C3 | 0.059 (2) | 0.066 (2) | 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, °)*

| | | | |
|---|---|---|---|
| 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) |

**Fig. 1**


O5
C18
C5
C8
C13
O3
C6
C4
C12
C14
C3
O2
C11
C7
C15
C10
O1
C2
C16
C9
O4
C17
C1
O6