Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2,2′-[Ethane-1,2-diylbis(oxy)]dibenzaldehyde

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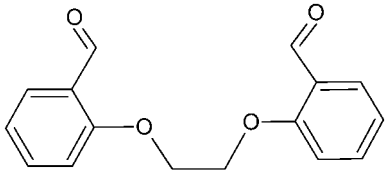
Received 9 July 2013; accepted 10 July 2013

Key indicators: single-crystal X-ray study; T = 120 K; mean σ(C–C) = 0.002 Å; R factor = 0.039; wR factor = 0.101; data-to-parameter ratio = 16.3.

In the title compound, $C_{16}H_{14}O_4$, the benzene rings are inclined at a dihedral angle of 75.14 (9)°. The torsion angle of the bridging O—C—C—O group is −76.50 (11)°. In the crystal, molecules are linked by C—H···O hydrogen bonds, forming $C(6)$ chains along [100]. Furthermore, C—H···π interactions and π–π stacking interactions [centroid–centroid distances = 3.6957 (7) and 3.6735 (8) Å] contribute to the stability of the crystal packing.

Related literature

For the synthesis and utlization of bis-funtionalized compounds, see: Holland *et al.* (2007); Pedras *et al.* (2010); Mabkhot *et al.* (2012); Gavrilova & Bosnich (2004). For bond-length data, see: Allen *et al.* (1987). For graph-set theory, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_{16}H_{14}O_4$
M_r = 270.27
Triclinic, $P\overline{1}$
a = 7.7571 (1) Å
b = 8.3277 (1) Å
c = 11.2965 (1) Å
α = 82.283 (7)°
β = 75.839 (7)°
γ = 66.823 (6)°
V = 649.87 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 120 K
0.62 × 0.44 × 0.22 mm

Data collection

Rigaku R-AXIS conversion diffractometer
Absorption correction: multi-scan (*CrystalClear-SM Expert*; Rigaku, 2012)
T_{min} = 0.878, T_{max} = 1.000
9715 measured reflections
2969 independent reflections
2862 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.101
S = 1.07
2969 reflections
182 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

*Cg*1 and *Cg*2 are the centroids of the C3–C8 and C10–C15 benzene rings, respectively.

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8···O3^{i}	0.95	2.44	3.2508 (17)	144
C2—H2A···*Cg*1^{ii}	0.99	2.68	3.4220 (12)	132
C2—H2B···*Cg*2^{iii}	0.99	2.70	3.5964 (14)	151

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

Data collection: *CrystalClear-SM Expert* (Rigaku, 2012); cell refinement: *CrystalClear-SM Expert*; data reduction: *CrystalClear-SM Expert*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *WinGX* (Farrugia, 2012) and *PLATON* (Spek, 2009).

Manchester Metropolitan University and Erciyes University are gratefully acknowledged for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT6920).

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supporting information

Acta Cryst. (2013). E**69**, o1260 [doi:10.1107/S1600536813019156]

2,2′-[Ethane-1,2-diylbis(oxy)]dibenzaldehyde

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S1. Comment

The synthesis of bis functionalized compounds has attracted the interest of chemists in chemical industry. Such compounds are considered as significant precursores for building blocks of vital molecules in different studies such as supramolecular chemistry and nanoscience (Holland *et al.*, 2007), bioactive bis heterocyclic compounds (Pedras *et al.*, 2010; Mabkhot *et al.*, 2012), and binucleating ligand designs (Gavrilova & Bosnich, 2004). In this concept the title compound has been synthesized among several derivatives of bis functionalized compounds as precursers for the synthesis of a series of macromolecular compounds.

The dihedral angle between the two benzene rings (C3–C8 and C10–C15) of the title compound (Fig. 1) is 75.14 (9)°. The torsion angle of the bridge O–C–C–O group is -76.50 (11)°. The C3–C4–C9–O3 and C10–C11–C16–O4 torsion angles of the two benzaldehyde groups are 175.10 (11) and -175.87 (13)°, respectively. Thus, they are almost coplanar with the rings to which they are attached. The bond lengths are normal (Allen *et al.*, 1987).

In the crystal, molecules are connected by C–H···O hydrogen bonds, generating infinite chains with the graph-set motif C(6) (Table 1, Fig. 2; Bernstein *et al.*, 1995) along the *a* axis. In addition, C–H···π interactions (Table 1) and π-π stacking interactions [*Cg*1···*Cg*1(- *x*, 2 - *y*, 1 - *z*) = 3.6957 (7) Å and *Cg*2···*Cg*2(1 - *x*, 1 - *y*, 1 - *z*) = 3.6735 (8) Å; where *Cg*1 and *Cg*2 are the centroids of the C3–C8 and C10–C15 benzene rings, respectively] contribute to stabilize the crystal structure.

S2. Experimental

A solution of 1.22 g m (0.01 mol) salicyldehyde in hot ethanolic KOH (prepared by dissolving 560 mg (0.01 mol) KOH in 100 ml of absolute ethanol) was stirred until a clear solution was obtained, which was then evaporated under vacuum. The residue was dissolved in DMF (25 ml) and 940 mg (0.005 mol) of dibromoethane was added. The reaction mixture was refluxed for 5 minutes, during which KBr was separated out. The solvent was then removed in vacuo and the remaining solid was washed with water and crystallized from ethanol to give high quality crystals (*Mp*. 393 K) suitable for X-ray analysis in an good yield (84%).

S3. Refinement

All H atoms were found in a difference map, but placed geometrically with C—H = 0.95 Å (aromatic H) and 0.99 Å (methylene H) and were refined using a riding model with U_{iso}(H) = 1.2U_{eq}(C).

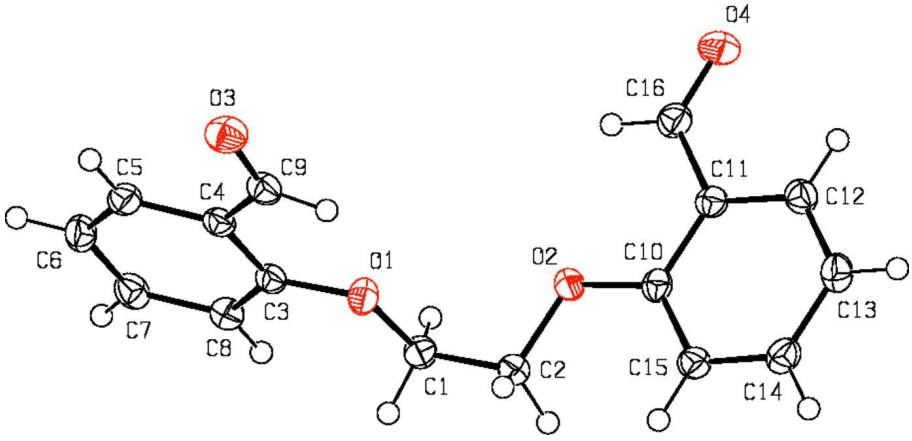

Figure 1

View of the molecular structure of the title compound with the atom numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level.

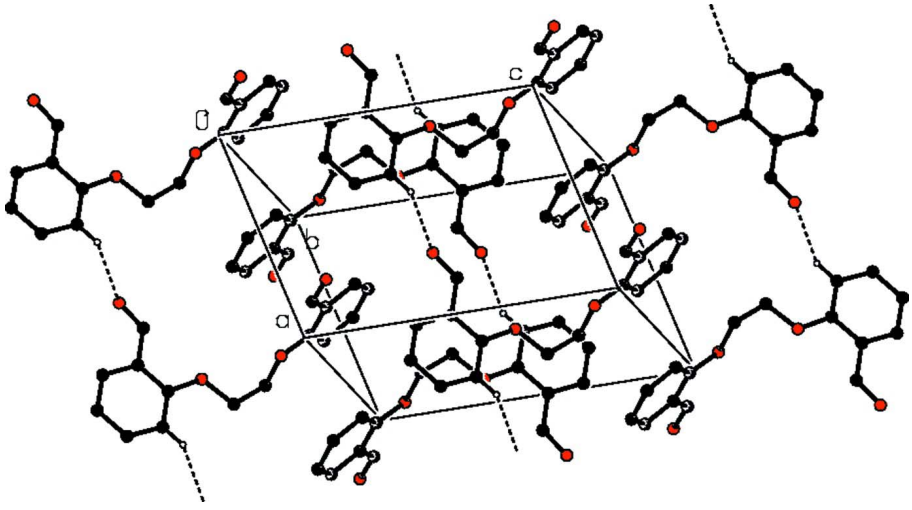

Figure 2

Partial packing diagram of the title compound showing hydrogen bonds as dashed lines. H atoms not involved in hydrogen bonds have been omitted for clarity.

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Crystal data

$C_{16}H_{14}O_4$
$M_r = 270.27$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 7.7571$ (1) Å
$b = 8.3277$ (1) Å
$c = 11.2965$ (1) Å
$\alpha = 82.283$ (7)°
$\beta = 75.839$ (7)°
$\gamma = 66.823$ (6)°
$V = 649.87$ (4) Å^3
$Z = 2$
$F(000) = 284$
$D_x = 1.381$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71075$ Å
Cell parameters from 4015 reflections
$\theta = 1.9$–27.5°
$\mu = 0.10$ mm^{-1}
$T = 120$ K
Block, colourless
0.62 × 0.44 × 0.22 mm

Data collection

Rigaku R-AXIS conversion diffractometer
Radiation source: Sealed Tube
Graphite Monochromator monochromator
Detector resolution: 10.0000 pixels mm^{-1}
profile data from ω–scans
Absorption correction: multi-scan (*CrystalClear-SM Expert*; Rigaku, 2012)
T_{min} = 0.878, T_{max} = 1.000
9715 measured reflections
2969 independent reflections
2862 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 27.5°, θ_{min} = 2.9°
h = −10→8
k = −10→10
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.101
S = 1.07
2969 reflections
182 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
W = 1/[Σ^2(FO^2) + (0.0397P)^2 + 0.2051P] WHERE P = (FO^2 + 2FC^2)/3
$(\Delta/\sigma)_{max}$ < 0.001
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), FC*=KFC[1+0.001XFC^{2}Λ^3/SIN(2Θ)]$^{-1/4}$
Extinction coefficient: 0.039 (4)

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted R-factors wR and all goodnesses 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 observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating -R-factor-obs *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}
O1	0.02681 (11)	0.67683 (11)	0.39979 (7)	0.0234 (2)
O2	0.09562 (12)	0.63138 (10)	0.13897 (7)	0.0229 (2)
O3	0.42624 (12)	0.70766 (12)	0.54585 (8)	0.0310 (3)
O4	0.29048 (14)	0.91784 (11)	−0.12808 (8)	0.0335 (3)
C1	−0.10587 (16)	0.66169 (14)	0.33728 (10)	0.0222 (3)
C2	0.00997 (16)	0.53887 (14)	0.23588 (10)	0.0221 (3)
C3	−0.04430 (16)	0.76808 (13)	0.50371 (9)	0.0198 (3)
C4	0.09320 (16)	0.78082 (14)	0.55953 (10)	0.0203 (3)
C5	0.03276 (17)	0.87486 (14)	0.66511 (10)	0.0238 (3)
C6	−0.16055 (18)	0.95186 (15)	0.71668 (10)	0.0264 (3)
C7	−0.29501 (17)	0.93499 (15)	0.66234 (10)	0.0254 (3)
C8	−0.23910 (16)	0.84489 (14)	0.55568 (10)	0.0225 (3)
C9	0.29937 (16)	0.69362 (15)	0.50767 (10)	0.0235 (3)
C10	0.20867 (15)	0.54157 (14)	0.03822 (10)	0.0201 (3)
C11	0.28537 (16)	0.63720 (14)	−0.05755 (10)	0.0205 (3)
C12	0.40808 (16)	0.55234 (15)	−0.16216 (10)	0.0237 (3)

C13	0.45243 (17)	0.37632 (16)	−0.17322 (11)	0.0267 (3)
C14	0.37194 (17)	0.28415 (15)	−0.07953 (11)	0.0255 (3)
C15	0.25049 (17)	0.36520 (15)	0.02579 (10)	0.0228 (3)
C16	0.23854 (17)	0.82470 (15)	−0.04713 (11)	0.0252 (3)
H1A	−0.18070	0.77750	0.30370	0.0270*
H1B	−0.19620	0.61520	0.39420	0.0270*
H2A	0.11090	0.43660	0.26590	0.0270*
H2B	−0.07370	0.49650	0.20600	0.0270*
H5	0.12500	0.88620	0.70190	0.0290*
H6	−0.20120	1.01580	0.78860	0.0320*
H7	−0.42750	0.98600	0.69880	0.0300*
H8	−0.33240	0.83580	0.51880	0.0270*
H9	0.33550	0.62190	0.44020	0.0280*
H12	0.46170	0.61600	−0.22640	0.0280*
H13	0.53720	0.31880	−0.24420	0.0320*
H14	0.40060	0.16380	−0.08780	0.0310*
H15	0.19620	0.30080	0.08900	0.0270*
H16	0.16270	0.87630	0.02780	0.0300*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0200 (4)	0.0302 (4)	0.0205 (4)	−0.0089 (3)	−0.0034 (3)	−0.0061 (3)
O2	0.0284 (4)	0.0212 (4)	0.0187 (4)	−0.0107 (3)	−0.0016 (3)	−0.0015 (3)
O3	0.0251 (5)	0.0358 (5)	0.0361 (5)	−0.0147 (4)	−0.0104 (4)	0.0025 (4)
O4	0.0444 (6)	0.0263 (4)	0.0336 (5)	−0.0190 (4)	−0.0086 (4)	0.0051 (4)
C1	0.0208 (5)	0.0257 (5)	0.0218 (5)	−0.0099 (4)	−0.0053 (4)	−0.0010 (4)
C2	0.0255 (6)	0.0231 (5)	0.0197 (5)	−0.0118 (4)	−0.0047 (4)	0.0009 (4)
C3	0.0227 (5)	0.0180 (5)	0.0180 (5)	−0.0084 (4)	−0.0031 (4)	0.0013 (4)
C4	0.0231 (5)	0.0192 (5)	0.0193 (5)	−0.0100 (4)	−0.0044 (4)	0.0035 (4)
C5	0.0310 (6)	0.0215 (5)	0.0222 (5)	−0.0123 (5)	−0.0090 (4)	0.0026 (4)
C6	0.0341 (6)	0.0218 (5)	0.0203 (5)	−0.0087 (5)	−0.0028 (5)	−0.0026 (4)
C7	0.0240 (6)	0.0220 (5)	0.0244 (5)	−0.0059 (4)	0.0002 (4)	−0.0003 (4)
C8	0.0215 (5)	0.0230 (5)	0.0228 (5)	−0.0090 (4)	−0.0043 (4)	0.0006 (4)
C9	0.0240 (6)	0.0250 (5)	0.0219 (5)	−0.0107 (4)	−0.0056 (4)	0.0035 (4)
C10	0.0199 (5)	0.0228 (5)	0.0190 (5)	−0.0077 (4)	−0.0068 (4)	−0.0013 (4)
C11	0.0210 (5)	0.0220 (5)	0.0210 (5)	−0.0093 (4)	−0.0077 (4)	0.0009 (4)
C12	0.0245 (6)	0.0262 (6)	0.0212 (5)	−0.0103 (4)	−0.0055 (4)	0.0005 (4)
C13	0.0284 (6)	0.0272 (6)	0.0217 (5)	−0.0079 (5)	−0.0029 (4)	−0.0045 (4)
C14	0.0299 (6)	0.0206 (5)	0.0264 (6)	−0.0082 (4)	−0.0079 (5)	−0.0030 (4)
C15	0.0264 (6)	0.0224 (5)	0.0219 (5)	−0.0111 (4)	−0.0071 (4)	0.0015 (4)
C16	0.0296 (6)	0.0234 (5)	0.0246 (5)	−0.0106 (5)	−0.0087 (5)	−0.0001 (4)

Geometric parameters (Å, º)

O1—C1	1.4337 (16)	C12—C13	1.3834 (17)
O1—C3	1.3611 (13)	C13—C14	1.3910 (18)
O2—C2	1.4332 (14)	C14—C15	1.3876 (17)

O2—C10	1.3593 (13)	C1—H1A	0.9900
O3—C9	1.2166 (17)	C1—H1B	0.9900
O4—C16	1.2147 (15)	C2—H2A	0.9900
C1—C2	1.5011 (15)	C2—H2B	0.9900
C3—C4	1.4081 (18)	C5—H5	0.9500
C3—C8	1.3940 (18)	C6—H6	0.9500
C4—C5	1.3942 (15)	C7—H7	0.9500
C4—C9	1.4731 (18)	C8—H8	0.9500
C5—C6	1.3839 (19)	C9—H9	0.9500
C6—C7	1.392 (2)	C12—H12	0.9500
C7—C8	1.3906 (16)	C13—H13	0.9500
C10—C11	1.4090 (16)	C14—H14	0.9500
C10—C15	1.3929 (16)	C15—H15	0.9500
C11—C12	1.3951 (16)	C16—H16	0.9500
C11—C16	1.4717 (16)		

C1—O1—C3	118.45 (10)	C2—C1—H1A	110.00
C2—O2—C10	117.35 (8)	C2—C1—H1B	110.00
O1—C1—C2	107.09 (10)	H1A—C1—H1B	109.00
O2—C2—C1	108.23 (9)	O2—C2—H2A	110.00
O1—C3—C4	115.69 (11)	O2—C2—H2B	110.00
O1—C3—C8	124.17 (11)	C1—C2—H2A	110.00
C4—C3—C8	120.14 (10)	C1—C2—H2B	110.00
C3—C4—C5	119.49 (11)	H2A—C2—H2B	108.00
C3—C4—C9	120.26 (10)	C4—C5—H5	120.00
C5—C4—C9	120.24 (12)	C6—C5—H5	120.00
C4—C5—C6	120.50 (12)	C5—C6—H6	120.00
C5—C6—C7	119.52 (11)	C7—C6—H6	120.00
C6—C7—C8	121.24 (12)	C6—C7—H7	119.00
C3—C8—C7	119.08 (12)	C8—C7—H7	119.00
O3—C9—C4	124.12 (11)	C3—C8—H8	120.00
O2—C10—C11	116.23 (9)	C7—C8—H8	120.00
O2—C10—C15	123.99 (10)	O3—C9—H9	118.00
C11—C10—C15	119.79 (10)	C4—C9—H9	118.00
C10—C11—C12	119.48 (10)	C11—C12—H12	120.00
C10—C11—C16	120.45 (10)	C13—C12—H12	120.00
C12—C11—C16	120.07 (11)	C12—C13—H13	120.00
C11—C12—C13	120.59 (11)	C14—C13—H13	120.00
C12—C13—C14	119.48 (11)	C13—C14—H14	119.00
C13—C14—C15	121.06 (11)	C15—C14—H14	119.00
C10—C15—C14	119.57 (11)	C10—C15—H15	120.00
O4—C16—C11	124.30 (11)	C14—C15—H15	120.00
O1—C1—H1A	110.00	O4—C16—H16	118.00
O1—C1—H1B	110.00	C11—C16—H16	118.00

C3—O1—C1—C2	−173.57 (9)	C4—C5—C6—C7	−0.04 (16)
C1—O1—C3—C8	1.68 (15)	C5—C6—C7—C8	−1.24 (17)
C1—O1—C3—C4	−178.90 (9)	C6—C7—C8—C3	0.98 (17)

C10—O2—C2—C1	179.42 (10)	O2—C10—C11—C12	177.80 (11)
C2—O2—C10—C15	−2.20 (18)	O2—C10—C11—C16	−1.51 (18)
C2—O2—C10—C11	177.73 (11)	C15—C10—C11—C12	−2.27 (19)
O1—C1—C2—O2	−76.50 (11)	C15—C10—C11—C16	178.42 (12)
O1—C3—C4—C9	−1.99 (15)	O2—C10—C15—C14	−178.22 (12)
C8—C3—C4—C5	−1.78 (16)	C11—C10—C15—C14	1.9 (2)
O1—C3—C8—C7	179.93 (9)	C10—C11—C12—C13	1.0 (2)
C4—C3—C8—C7	0.54 (16)	C16—C11—C12—C13	−179.70 (13)
C8—C3—C4—C9	177.46 (10)	C10—C11—C16—O4	−175.87 (13)
O1—C3—C4—C5	178.78 (10)	C12—C11—C16—O4	4.8 (2)
C3—C4—C9—O3	175.10 (11)	C11—C12—C13—C14	0.7 (2)
C5—C4—C9—O3	−5.67 (18)	C12—C13—C14—C15	−1.1 (2)
C3—C4—C5—C6	1.53 (16)	C13—C14—C15—C10	−0.2 (2)
C9—C4—C5—C6	−177.71 (10)		

Hydrogen-bond geometry (Å, º)

Cg1 and Cg2 are the centroids of the C3–C8 and C10–C15 benzene rings, respectively.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8···O3^{i}	0.95	2.44	3.2508 (17)	144
C9—H9···O1	0.95	2.40	2.7412 (16)	101
C16—H16···O2	0.95	2.42	2.7561 (15)	101
C2—H2*A*···*Cg*1^{ii}	0.99	2.68	3.4220 (12)	132
C2—H2*B*···*Cg*2^{iii}	0.99	2.70	3.5964 (14)	151

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