

P. G. Aravindan,<sup>a</sup> M. Yogavel,<sup>a</sup>  
M. Thirumavalavan,<sup>b</sup> P. Akilan,<sup>b</sup>  
D. Velmurugan,<sup>a\*</sup> M.  
Kandaswamy,<sup>b</sup> S. Shanmuga  
Sundara Raj<sup>c</sup> and H.-K. Fun<sup>d</sup>

<sup>a</sup>Department of Crystallography and Biophysics, University of Madras, Guindy Campus, Chennai 600 025, India, <sup>b</sup>Department of Inorganic Chemistry, University of Madras, Guindy Campus, Chennai 600 025, India, <sup>c</sup>B3121 Medical Centre North, Department of Medicine-Nephrology, Nashville, USA, and <sup>d</sup>X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia

Correspondence e-mail: d\_velu@yahoo.com

#### Key indicators

Single-crystal X-ray study  
T = 293 K  
Mean  $\sigma(\text{C}-\text{C}) = 0.005 \text{ \AA}$   
R factor = 0.086  
wR factor = 0.240  
Data-to-parameter ratio = 17.4

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

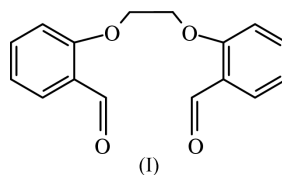
## 3,4:9,10-Dibenzo-1,12-diformyl-5,8-dioxododecane

In the title compound,  $\text{C}_{16}\text{H}_{14}\text{O}_4$ , the dihedral angle between the two aromatic rings is  $75.8 (1)^\circ$ . The torsion angle about the central C—C bond is  $77.2 (3)^\circ$ . The packing of the molecules in the solid state is stabilized by C—H $\cdots$ O, C—H $\cdots$  $\pi$  and  $\pi$ — $\pi$  intermolecular interactions.

Received 8 April 2003  
Accepted 6 May 2003  
Online 16 May 2003

#### Comment

Polyether compounds are of much interest recently due to their remarkable metal ion selectivity, ability to transport alkali metal ions through biological and artificial membranes and their utilization in organic synthetic procedures (Armstrong & Lindoy, 1975; Grimsley *et al.*, 1977).

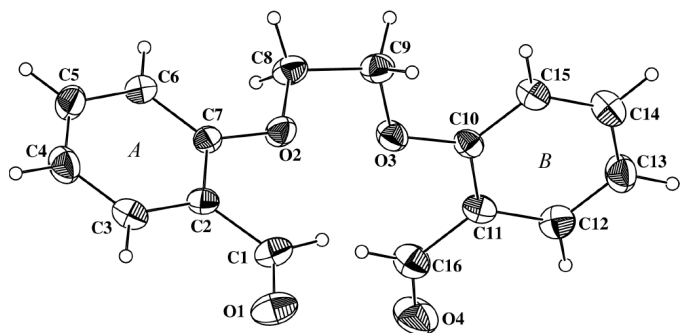


The bond lengths and angles of the title compound, (I), agree with the values reported for 2,2'-[1,2-ethanediylbis(oxy)](benzenemethanol) (Bailey *et al.*, 1989). The dihedral angle between the aromatic rings *A* (C2—C7) and *B* (C10—C15) is  $75.9 (1)^\circ$ . Atoms O1/C1/O2/C8 and atoms O4/C16/O3/C9 are almost coplanar with rings *A* and *B*, respectively [the deviations of these atoms are 0.020 (3), 0.058 (4),  $-0.010$  (2) and  $-0.040$  (4)  $\text{\AA}$ , and  $-0.101$  (4),  $-0.030$  (5), 0.050 (3) and 0.012 (4)  $\text{\AA}$ , respectively]. The ethylene group between the aromatic rings has the *gauche* form and the torsion angle about the C8—C9 bond is  $77.2 (4)^\circ$ , indicating that the molecule is twisted.

The molecules are linked by weak C—H $\cdots$ O interactions and form an infinite chain running along the *a* direction (Fig. 2). The face-to-face interactions are between *A* and *A*<sup>iv</sup>, and *B* and *B*<sup>v</sup>, which stack in the lattice along the *b* and *a* axes, respectively, with centroid—centroid distances of 3.784 (2) and 3.731 (2)  $\text{\AA}$ , respectively [symmetry codes: (iv)  $1 - x, 1 - y, -z$ ; (v)  $-x, 2 - y, 1 - z$ ]. Apart from these weak  $\pi$ — $\pi$  interactions, the packing of the molecules in the solid state is stabilized by C—H $\cdots$ O and C—H $\cdots$  $\pi$  intermolecular interactions (Table 2).

#### Experimental

1,2-Dibromoethane (9.396 g, 0.05 mol) in methanol (10 ml) was added to salicylaldehyde (9.052 g, 0.1 mol) in methanol (30 ml) and the solution was heated. Sodium hydroxide pellets (4 g, 0.1 mol) in water (15 ml) were added and the reaction mixture was refluxed under an  $\text{N}_2$  atmosphere for nearly 48 h. The resulting solution was then allowed to cool at room temperature (298 K). The resulting solid



**Figure 1**  
The molecular structure, with the atom-numbering scheme. Ellipsoids are drawn at the 35% probability level.

was then filtered off and washed with water and methanol. Crystals were obtained by recrystallization from chloroform.

#### Crystal data

$C_{16}H_{14}O_4$	$Z = 2$
$M_r = 270.27$	$D_x = 1.334 \text{ Mg m}^{-3}$
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 7.7956 (7) \text{ \AA}$	Cell parameters from 1815 reflections
$b = 8.4511 (8) \text{ \AA}$	$\theta = 1.8\text{--}28.3^\circ$
$c = 11.4697 (11) \text{ \AA}$	$\mu = 0.10 \text{ mm}^{-1}$
$\alpha = 83.054 (2)^\circ$	$T = 293 (2) \text{ K}$
$\beta = 75.008 (2)^\circ$	Block, yellow
$\gamma = 67.219 (2)^\circ$	$0.36 \times 0.24 \times 0.16 \text{ mm}$
$V = 672.80 (11) \text{ \AA}^3$	

#### Data collection

Siemens SMART CCD area-detector	1273 reflections with $I > 2\sigma(I)$
$\omega$ scans	$R_{\text{int}} = 0.064$
Absorption correction: none	$\theta_{\text{max}} = 28.3^\circ$
4829 measured reflections	$h = -10 \rightarrow 9$
3172 independent reflections	$k = -10 \rightarrow 11$
	$l = -15 \rightarrow 15$

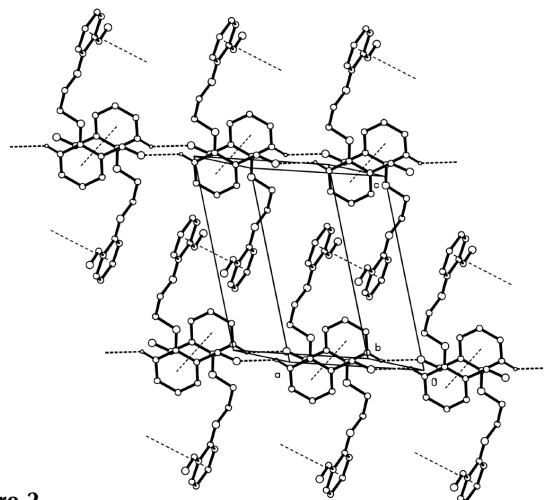
#### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.1202P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.086$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.240$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 0.83$	$\Delta\rho_{\text{max}} = 0.37 \text{ e \AA}^{-3}$
3172 reflections	$\Delta\rho_{\text{min}} = -0.38 \text{ e \AA}^{-3}$
182 parameters	Extinction correction: <i>SHELXL97</i>
H-atom parameters constrained	Extinction coefficient: 0.036 (10)

**Table 1**

Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ).

O1—C1	1.204 (4)	O4—C16	1.205 (4)
O2—C7	1.365 (3)	C1—C2	1.464 (5)
O2—C8	1.423 (3)	C8—C9	1.493 (4)
O3—C10	1.365 (4)	C11—C16	1.459 (5)
O3—C9	1.432 (3)		
C7—O2—C8	119.2 (2)	O2—C8—C9	108.0 (3)
C10—O3—C9	117.3 (2)	O3—C9—C8	108.1 (3)
O1—C1—C2	125.5 (4)	O3—C10—C15	124.7 (3)
O2—C7—C6	123.9 (3)	O3—C10—C11	115.4 (3)
O2—C7—C2	115.9 (3)	O4—C16—C11	124.7 (4)
O1—C1—C2—C3	3.4 (5)	O2—C8—C9—O3	77.2 (3)
C7—O2—C8—C9	173.3 (3)	C12—C11—C16—O4	−3.0 (6)
C10—O3—C9—C8	178.4 (2)		



**Figure 2**  
A packing diagram of the molecule, viewed down the  $a$  axis.  $\pi$ - $\pi$  and C—H...O interactions are indicated by dashed lines.

**Table 2**

Hydrogen-bonding geometry ( $\text{\AA}$ ,  $^\circ$ ).

$CgA$  and  $CgB$  are the centroids of rings  $A$  and  $B$ , respectively.

$D\text{---}H\cdots A$	$D\text{---}H$	$H\cdots A$	$D\cdots A$	$D\text{---}H\cdots A$
C1—H1...O2	0.93	2.40	2.746 (4)	102
C16—H16...O3	0.93	2.41	2.748 (4)	102
C6—H6...O1 <sup>i</sup>	0.93	2.48	3.290 (4)	146
C9—H9A...CgB <sup>ii</sup>	0.97	2.84	3.712 (2)	150
C9—H9B...CgA <sup>iii</sup>	0.97	2.76	3.473 (2)	131

Symmetry codes: (i)  $1 + x, y, z$ ; (ii)  $1 - x, 2 - y, 1 - z$ ; (iii)  $1 - x, 2 - y, -z$ .

All the H atoms were geometrically positioned and were treated as riding on their parent atoms, with C—H distances in the range 0.93–0.97  $\text{\AA}$ .

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ZORTEP* (Zsolnai, 1997) and *PLATON* (Spek, 1990); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1995).

Financial support from the Department of Science and Technology (DST) and the University Grants Commission (UGC), Government of India, are gratefully acknowledged.

#### References

- Armstrong, L. G. & Lindoy, L. F. (1975). *Inorg. Chem.* **14**, 1322–1326.  
 Bailey, N. A., Fenton, D. E., Williams, M. G. & Winter, D. J. (1989). *Acta Cryst.* **C45**, 1778–1780.  
 Grimsley, P. G., Lindoy, L. F., Lip, H. C., Smith, R. J. & Baker, J. T. (1977). *Aust. J. Chem.* **30**, 2095–2098.  
 Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.  
 Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.  
 Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.  
 Spek, A. L. (1990). *Acta Cryst.* **A46**, C-34.  
 Zsolnai, L. (1997). *ZORTEP*. University of Heidelberg, Germany.