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1,2-Bis(4-methoxyphenoxy)ethane

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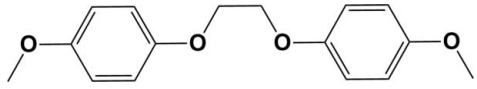
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.046; wR factor = 0.224; data-to-parameter ratio = 13.7.

The whole molecule of the title compound, $C_{16}H_{18}O_4$, is generated by twofold rotational symmetry; the twofold axis bisects the central C—C bond. The O—C—C—O torsion angle about the central C—C bond is 69.45 (16)°. Symmetry-related benzene rings are inclined to one another by 64.91 (8)°. In the crystal, molecules are connected by C—H···O hydrogen bonds, forming a three-dimensional network.

Related literature

For the synthesis, uses and properties of the title compound, see: Saito *et al.* (1988). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{16}H_{18}O_4$
M_r = 274.32
Monoclinic, $C2/c$
a = 26.073 (4) Å
b = 5.5538 (8) Å
c = 9.7591 (14) Å
β = 102.211 (3)°
V = 1381.2 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 K
0.20 × 0.18 × 0.15 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.981, T_{max} = 0.986
3780 measured reflections
1277 independent reflections
976 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.224
S = 1.10
1277 reflections
93 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1B···O1^{i}	0.96	2.60	3.453 (2)	148
C6—H6···O2^{ii}	0.93	2.59	3.490 (2)	163

Symmetry codes: (i) $-x+\frac{1}{2}, -y+\frac{3}{2}, -z+1$; (ii) $-x, -y+2, -z$.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1985); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo,1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXTL*.

The authors thank the Center of Testing and Analysis, Nanjing University, for the data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SU2572).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Enraf–Nonius (1985). *CAD-4 Software*. Enraf–Nonius, Delft, The Netherlands.
Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Saito, T., Kitani, M. & Ishibashi, T. (1988). Jpn Patent No. JP 63156731.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2013). E**69**, o547 [doi:10.1107/S1600536813007009]

1,2-Bis(4-methoxyphenoxy)ethane

Jun Ji, XiuQin Zhang, Kai Wang, ChaoFan Ju and Qiang Chen

S1. Comment

The tittle compound is used as an important organic synthesis intermediate. It is used as a sensitizer for thermal recording materials and materials for polyester-resin monomers and fire-resistant materials (Saito *et al.*,1988). We report herein on the crystal structure of the title compound.

The molecular structure of the title molecule is shown in Fig. 1. The bond lengths (Allen *et al.*, 1987) and angles are within normal ranges. The molecule has two-fold rotational symmetry, the 2-fold axis bisecting bond C8-C8A. The central O2-C8-C8A-O2A torsion angles is 69.45 (16) °. The symmetry related benzene rings [C2-C7 and C2A-C7A; symmetry code (A) -x, y, -z-1/2], are inclined to one another by 64.91 (8) °.

In the crystal, molecules are linked via C—H···O hydrogen bonds forming a three-dimensional network (Fig. 2 and Table 1).

S2. Experimental

The title compound was prepared by a method reported in literature (Saito *et al.*, 1988). To a solution of 1,2-dibromo-ethane (2.3 g, 12 mmol) and 4-methoxyphenol (3.1 g, 25 mmol) in acetonitrile (100 ml) was added anhydrous potassium carbonate (6.2 g, 45 mmol), and the mixture was stirred overnight at 338 K. The reaction mixture was filtered and the filtrate evaporated under reduced pressure. The residue was subjected to flash chromatography on silica gel, eluting with (10:1/ petroleum ether:ethyl acetate) to give title compound (Yield 2.13 g). Colourless block-like crystals of the title compound were obtained by slow evaporation of a solution in ethanol (20 ml) after about 7 d.

S3. Refinement

All H atoms were positioned geometrically and constrained to ride on their parent atom: C—H = 0.93 Å (aromatic H), 0.97 Å (CH_2), and 0.96 Å (CH_3) with $U_{iso}(H) = k \times U_{eq}(C)$, where k = 1.5 for CH_3 H atoms, and = 1.2 for other H atoms.

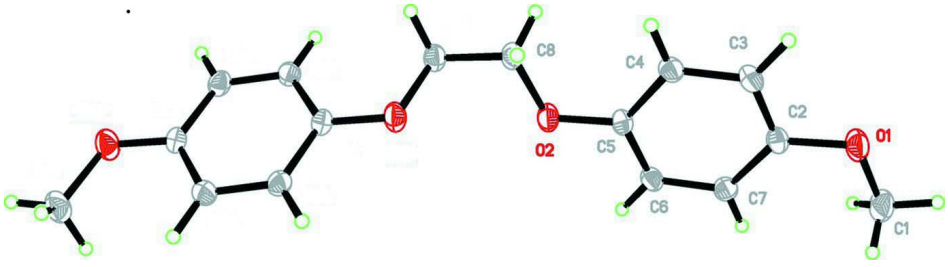


Figure 1

The molecular structure of the title molecule, with the atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

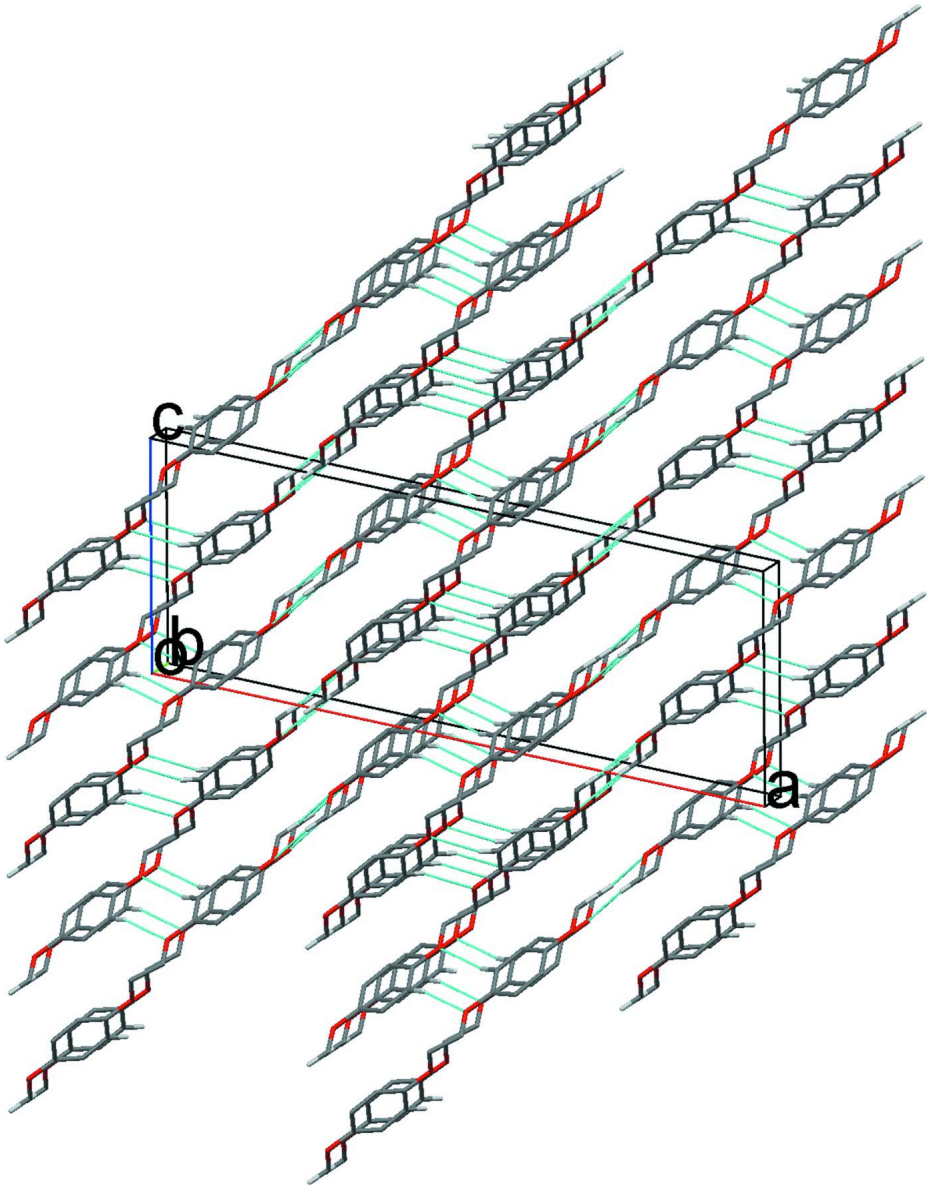


Figure 2

A view along the b axis of the crystal packing of the title compound, showing the C-H···O hydrogen bonds (dashed lines; see Table 1 for details; H atoms not involved in these interactions have been omitted for clarity).

1,2-Bis(4-methoxyphenoxy)ethane

Crystal data

$C_{16}H_{18}O_4$
$M_r = 274.32$
Monoclinic, $C2/c$
Hall symbol: -C 2yc
$a = 26.073$ (4) Å
$b = 5.5538$ (8) Å
$c = 9.7591$ (14) Å
$\beta = 102.211$ (3)°
$V = 1381.2$ (4) Å^3
$Z = 4$
$F(000) = 584$
$D_x = 1.319$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1571 reflections
$\theta = 3.2$–30.3°
$\mu = 0.09$ mm^{-1}

T = 293 K
Block, colourless

0.20 × 0.18 × 0.15 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.981, T_{max} = 0.986
3780 measured reflections

1277 independent reflections
976 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 25.5°, θ_{min} = 1.6°
h = −31→24
k = −6→5
l = −11→11
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.224
S = 1.10
1277 reflections
93 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 constrained
$w = 1/[\sigma^2(F_o^2) + (0.168P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.020 (6)

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 esds are taken into account in the estimation of distances, angles and torsion angles

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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
O1	0.20123 (6)	0.7928 (2)	0.33002 (13)	0.0496 (5)
O2	0.02888 (5)	0.7020 (2)	−0.10672 (11)	0.0412 (5)
C1	0.20477 (9)	0.9943 (3)	0.42124 (19)	0.0545 (8)
C2	0.15790 (7)	0.7773 (3)	0.22314 (17)	0.0332 (6)
C3	0.15573 (7)	0.5837 (3)	0.13368 (16)	0.0364 (6)
C4	0.11377 (7)	0.5526 (3)	0.02195 (16)	0.0343 (6)
C5	0.07272 (7)	0.7183 (3)	−0.00101 (16)	0.0316 (5)
C6	0.07499 (7)	0.9113 (3)	0.08874 (15)	0.0352 (6)
C7	0.11697 (7)	0.9435 (3)	0.20009 (16)	0.0361 (6)
C8	0.02601 (7)	0.5040 (3)	−0.20015 (15)	0.0369 (6)
H1A	0.20640	1.13950	0.36900	0.0820*
H1B	0.23590	0.98070	0.49380	0.0820*

H1C	0.17450	0.99880	0.46250	0.0820*
H3	0.18300	0.47210	0.14870	0.0440*
H4	0.11300	0.42150	−0.03770	0.0410*
H6	0.04770	1.02230	0.07400	0.0420*
H7	0.11790	1.07540	0.25930	0.0430*
H8A	0.03060	0.35460	−0.14740	0.0440*
H8B	0.05390	0.51580	−0.25180	0.0440*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0415 (9)	0.0576 (10)	0.0417 (9)	0.0100 (6)	−0.0089 (6)	−0.0100 (6)
O2	0.0360 (9)	0.0484 (9)	0.0342 (8)	0.0108 (5)	−0.0037 (6)	−0.0086 (5)
C1	0.0498 (13)	0.0594 (14)	0.0457 (12)	0.0018 (9)	−0.0094 (9)	−0.0141 (8)
C2	0.0324 (10)	0.0394 (11)	0.0261 (9)	0.0017 (7)	0.0024 (7)	0.0032 (6)
C3	0.0336 (11)	0.0408 (11)	0.0340 (10)	0.0103 (7)	0.0052 (7)	0.0028 (7)
C4	0.0373 (11)	0.0348 (10)	0.0307 (9)	0.0061 (7)	0.0067 (7)	−0.0021 (6)
C5	0.0302 (10)	0.0388 (10)	0.0250 (8)	0.0028 (6)	0.0041 (7)	0.0024 (6)
C6	0.0339 (10)	0.0355 (10)	0.0345 (10)	0.0087 (7)	0.0037 (8)	0.0003 (7)
C7	0.0393 (11)	0.0346 (10)	0.0335 (9)	0.0039 (7)	0.0054 (8)	−0.0039 (6)
C8	0.0375 (11)	0.0425 (11)	0.0286 (9)	0.0043 (6)	0.0024 (8)	−0.0033 (6)

Geometric parameters (Å, º)

O1—C1	1.421 (2)	C8—C8^{i}	1.493 (2)
O1—C2	1.369 (2)	C1—H1A	0.9600
O2—C5	1.371 (2)	C1—H1B	0.9600
O2—C8	1.4203 (19)	C1—H1C	0.9600
C2—C3	1.379 (2)	C3—H3	0.9300
C2—C7	1.393 (3)	C4—H4	0.9300
C3—C4	1.382 (2)	C6—H6	0.9300
C4—C5	1.393 (3)	C7—H7	0.9300
C5—C6	1.378 (2)	C8—H8A	0.9700
C6—C7	1.381 (2)	C8—H8B	0.9700
C1—O1—C2	117.44 (15)	H1A—C1—H1B	109.00
C5—O2—C8	117.16 (13)	H1A—C1—H1C	110.00
O1—C2—C3	116.64 (15)	H1B—C1—H1C	110.00
O1—C2—C7	124.21 (15)	C2—C3—H3	119.00
C3—C2—C7	119.16 (16)	C4—C3—H3	119.00
C2—C3—C4	121.10 (16)	C3—C4—H4	120.00
C3—C4—C5	119.81 (15)	C5—C4—H4	120.00
O2—C5—C4	124.55 (14)	C5—C6—H6	119.00
O2—C5—C6	116.53 (15)	C7—C6—H6	119.00
C4—C5—C6	118.91 (15)	C2—C7—H7	120.00
C5—C6—C7	121.42 (16)	C6—C7—H7	120.00
C2—C7—C6	119.60 (15)	O2—C8—H8A	110.00
O2—C8—C8^{i}	109.61 (14)	O2—C8—H8B	110.00

O1—C1—H1A	109.00	H8A—C8—H8B	108.00
O1—C1—H1B	109.00	C8^{i}—C8—H8A	110.00
O1—C1—H1C	109.00	C8^{i}—C8—H8B	110.00
C1—O1—C2—C3	178.49 (16)	C3—C2—C7—C6	0.2 (3)
C1—O1—C2—C7	−1.2 (2)	C2—C3—C4—C5	−0.3 (3)
C8—O2—C5—C4	−1.2 (2)	C3—C4—C5—O2	−178.66 (16)
C8—O2—C5—C6	179.96 (15)	C3—C4—C5—C6	0.2 (3)
C5—O2—C8—C8^{i}	175.16 (13)	O2—C5—C6—C7	179.05 (15)
O1—C2—C3—C4	−179.60 (16)	C4—C5—C6—C7	0.1 (2)
C7—C2—C3—C4	0.1 (3)	C5—C6—C7—C2	−0.3 (3)
O1—C2—C7—C6	179.87 (16)	O2—C8—C8^{i}—O2^{i}	69.45 (16)

Symmetry code: (i) −*x*, *y*, −*z*−1/2.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1*B*···O1^{ii}	0.96	2.60	3.453 (2)	148
C6—H6···O2^{iii}	0.93	2.59	3.490 (2)	163

Symmetry codes: (ii) −*x*+1/2, −*y*+3/2, −*z*+1; (iii) −*x*, −*y*+2, −*z*.