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1,4-Bis(4-bromobutoxy)benzene

Gunasekaran Maragatham,[a] Sivasamy Selvarani,[b] Perumal Rajakumar[b] and Srinivasakannan Lakshmi[a]*

[a]Department of Physics, S.D.N.B. Vaishnav College for Women, Chromepet, Chennai 600 044, India, and [b]Department of Organic Chemistry, University of Madras, Guindy Campus, Chennai 600 025, India. *Correspondence e-mail: lakssdnbvc@gmail.com

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Keywords: crystal structure; bromobutoxy; C—H$\cdots\pi$ interactions.

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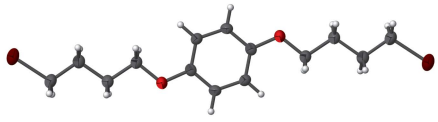
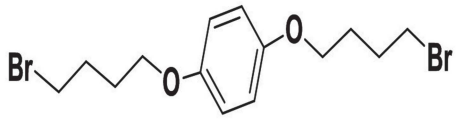
Structural data: full structural data are available from iucrdata.iucr.org

The complete molecule of the title compound, $C_{14}H_{20}Br_2O_2$, is generated by crystallographic inversion symmetry and the 4-bromobutoxy side chain adopts an extended conformation. In the crystal, weak C—H$\cdots\pi$ interactions are observed, which help to consolidate a herringbone packing motif.

3D view



Chemical scheme



Structure description

Compounds with alkyloxy substituents act as intermediates to engineer soluble electroluminescent oligomers and polymers for LED applications (Huang *et al.*, 2007). As part of our studies in this area, we now describe the synthesis and structure of the title compound.

The asymmetric unit, contains one-half of the molecule, while the other half is generated through crystallographic inversion symmetry [symmetry code: (i) $-x$, $1-y$, $-z$] (Fig. 1). The bromoalkoxyl tail is roughly co-planar with the attached benzene ring with a C6—C5—O1—C4 torsion angle of −2.2 (3)°. The bromoalkoxyl tail adopts an extended conformation as shown by the C5—O1—C4—C3, O1—C4—C3—C2, C4—C3—C2—C1 and C3—C2—C1—Br1 torsion angles of −179.55 (19), −176.29 (18), 177.5 (2) and 179.19 (17)°, respectively. The packing of the molecules features weak C—H$\cdots\pi$ interactions (Table 1), which lead to a herringbone arrangement when viewed along [100] (Fig. 2).

Synthesis and crystallization

A mixture of (1.0 equiv.) of resorcinol and potassium carbonate (2.0 equiv.) in acetone (50 ml) was stirred for 15 minutes at 60° C. 1,4-Dibromobutane (2.1 equiv.) was added to the reaction mixture and stirred at 60° C for 7 h. After completion of the reaction, the

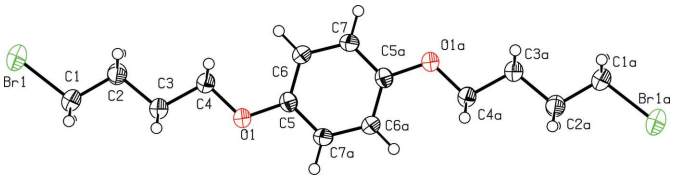


Figure 1
The molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level [symmetry code: (a) $-x$, $1-y$, $-z$].

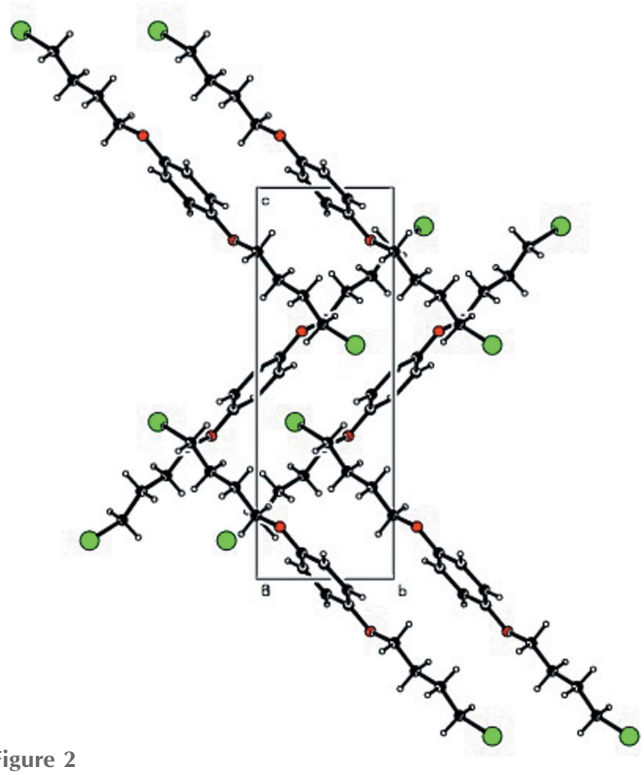


Figure 2
The packing of the molecules viewed along the *a*-axis direction.

solvent was removed under reduced pressure and the residue was extracted with $CHCl_3$ (3 × 100 ml), washed with water (2 × 100 ml), brine (150 ml) and dried over anhydrous Na_2SO_4, filtered and concentrated *in vacuo*. The residue obtained was purified by column chromatography using $CHCl_3$:hexane (1:9) as eluent to afford the title compound as a white solid, which was recrystallized from methanol solution to yield colourless blocks.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

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

*Cg*1 is the centroid of the benzene ring.

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2B···$Cg1^{i}$	0.97	2.84	3.664 (3)	144

Symmetry code: (i) $x-\frac{1}{2}, -y-\frac{1}{2}, z-\frac{3}{2}$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{14}H_{20}Br_2O_2$
M_r	380.12
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	296
a, b, c (Å)	9.0845 (10), 5.3436 (5), 15.3509 (15)
β (°)	95.567 (4)
V (Å^3)	741.68 (13)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	5.46
Crystal size (mm)	0.35 × 0.25 × 0.20
Data collection	
Diffractometer	Bruker Kappa APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2004)
T_{min}, T_{max}	0.525, 0.745
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	7040, 1310, 1126
R_{int}	0.027
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.024, 0.052, 1.06
No. of reflections	1310
No. of parameters	82
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.32, −0.33

Computer programs: *APEX2* and *SAINT* (Bruker, 2004), *SIR92* (Altomare *et al.*, 1993), *SHELXL2014* (Sheldrick, 2015), *ORTEP-3 for Windows* (Farrugia, 2012), *Mercury* (Bruno *et al.*, 2002) and *publCIF* (Westrip, 2010).

Acknowledgements

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full crystallographic data

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1,4-Bis(4-bromobutoxy)benzene

Crystal data

$C_{14}H_{20}Br_2O_2$
M_r = 380.12
Monoclinic, $P2_1/n$
a = 9.0845 (10) Å
b = 5.3436 (5) Å
c = 15.3509 (15) Å
β = 95.567 (4)°
V = 741.68 (13) Å^3
Z = 2
$F(000)$ = 380
D_x = 1.702 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3047 reflections
θ = 2.7–24.9°
μ = 5.46 mm^{-1}
T = 296 K
Block, colourless
0.35 × 0.25 × 0.20 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scan
Absorption correction: multi-scan (SADABS; Bruker, 2004)
T_{min} = 0.525, T_{max} = 0.745
7040 measured reflections
1310 independent reflections
1126 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 25.0°, θ_{min} = 2.5°
h = −10→10
k = −6→6
l = −18→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.024
$wR(F^2)$ = 0.052
S = 1.06
1310 reflections
82 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0214P)^2 + 0.4039P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

Special details

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

Refinement. H atoms were included in the refinement at calculated positions (C—H = 0.93–0.98 Å), with U_{iso}(H) = 1.2Ueq(C) using a riding-model approximation.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	−0.0397 (3)	1.4815 (5)	−0.34901 (18)	0.0487 (7)
H1A	−0.1220	1.5707	−0.3277	0.058*
H1B	−0.0796	1.3642	−0.3934	0.058*
C2	0.0404 (3)	1.3397 (4)	−0.27508 (15)	0.0354 (6)
H2A	0.0812	1.4561	−0.2306	0.043*
H2B	0.1216	1.2472	−0.2962	0.043*
C3	−0.0642 (3)	1.1585 (4)	−0.23511 (15)	0.0343 (6)
H3A	−0.1431	1.2523	−0.2119	0.041*
H3B	−0.1085	1.0478	−0.2804	0.041*
C4	0.0151 (3)	1.0047 (4)	−0.16294 (15)	0.0323 (5)
H4A	0.0974	0.9162	−0.1845	0.039*
H4B	0.0532	1.1121	−0.1150	0.039*
C5	−0.0386 (3)	0.6709 (4)	−0.06653 (14)	0.0288 (5)
C6	0.1050 (3)	0.6624 (4)	−0.02794 (15)	0.0305 (5)
H6	0.1759	0.7708	−0.0464	0.037*
C7	0.1431 (3)	0.4919 (4)	0.03832 (14)	0.0307 (5)
H7	0.2399	0.4864	0.0642	0.037*
O1	−0.08882 (18)	0.8306 (3)	−0.13368 (11)	0.0376 (4)
Br1	0.08821 (3)	1.72008 (5)	−0.40166 (2)	0.04831 (12)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0408 (17)	0.0559 (16)	0.0491 (16)	−0.0108 (13)	0.0028 (13)	0.0206 (13)
C2	0.0386 (15)	0.0339 (12)	0.0338 (13)	−0.0018 (11)	0.0035 (11)	0.0026 (10)
C3	0.0347 (15)	0.0358 (13)	0.0324 (13)	−0.0015 (11)	0.0027 (11)	0.0056 (10)
C4	0.0335 (14)	0.0322 (12)	0.0314 (12)	−0.0024 (10)	0.0038 (11)	0.0037 (10)
C5	0.0323 (13)	0.0283 (11)	0.0256 (12)	0.0017 (10)	0.0021 (10)	0.0006 (9)
C6	0.0274 (13)	0.0322 (12)	0.0321 (12)	−0.0037 (10)	0.0035 (10)	0.0028 (10)
C7	0.0248 (13)	0.0353 (12)	0.0313 (12)	−0.0012 (10)	−0.0001 (10)	0.0015 (10)
O1	0.0323 (10)	0.0402 (9)	0.0390 (9)	−0.0043 (8)	−0.0022 (8)	0.0147 (8)
Br1	0.0566 (2)	0.04394 (17)	0.04603 (18)	−0.00749 (13)	0.01338 (13)	0.01054 (12)

Geometric parameters (Å, º)

C1—C2	1.494 (3)	C4—O1	1.428 (3)
C1—Br1	1.952 (2)	C4—H4A	0.9700
C1—H1A	0.9700	C4—H4B	0.9700
C1—H1B	0.9700	C5—C6	1.381 (3)
C2—C3	1.526 (3)	C5—O1	1.381 (3)
C2—H2A	0.9700	C5—C7^{i}	1.387 (3)
C2—H2B	0.9700	C6—C7	1.384 (3)
C3—C4	1.506 (3)	C6—H6	0.9300
C3—H3A	0.9700	C7—C5^{i}	1.387 (3)
C3—H3B	0.9700	C7—H7	0.9300

C2—C1—Br1	112.34 (19)	H3A—C3—H3B	107.9
C2—C1—H1A	109.1	O1—C4—C3	107.66 (19)
Br1—C1—H1A	109.1	O1—C4—H4A	110.2
C2—C1—H1B	109.1	C3—C4—H4A	110.2
Br1—C1—H1B	109.1	O1—C4—H4B	110.2
H1A—C1—H1B	107.9	C3—C4—H4B	110.2
C1—C2—C3	110.6 (2)	H4A—C4—H4B	108.5
C1—C2—H2A	109.5	C6—C5—O1	124.8 (2)
C3—C2—H2A	109.5	C6—C5—C7^{i}	119.5 (2)
C1—C2—H2B	109.5	O1—C5—C7^{i}	115.7 (2)
C3—C2—H2B	109.5	C5—C6—C7	119.7 (2)
H2A—C2—H2B	108.1	C5—C6—H6	120.2
C4—C3—C2	111.7 (2)	C7—C6—H6	120.2
C4—C3—H3A	109.3	C6—C7—C5^{i}	120.8 (2)
C2—C3—H3A	109.3	C6—C7—H7	119.6
C4—C3—H3B	109.3	C5^{i}—C7—H7	119.6
C2—C3—H3B	109.3	C5—O1—C4	117.18 (18)
Br1—C1—C2—C3	179.19 (17)	C5—C6—C7—C5^{i}	0.0 (4)
C1—C2—C3—C4	177.5 (2)	C6—C5—O1—C4	−2.2 (3)
C2—C3—C4—O1	−176.29 (18)	C7^{i}—C5—O1—C4	178.59 (18)
O1—C5—C6—C7	−179.2 (2)	C3—C4—O1—C5	−179.55 (19)
C7^{i}—C5—C6—C7	0.0 (4)		

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

Hydrogen-bond geometry (Å, º)

*Cg*1 is the centroid of the benzene ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2*B*···*Cg*1^{ii}	0.97	2.84	3.664 (3)	144

Symmetry code: (ii) *x*−1/2, −*y*−1/2, *z*−3/2.