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1,3-Bis(4-bromobutoxy)benzene

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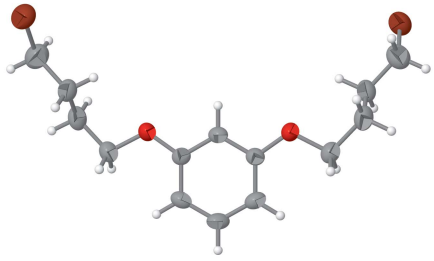
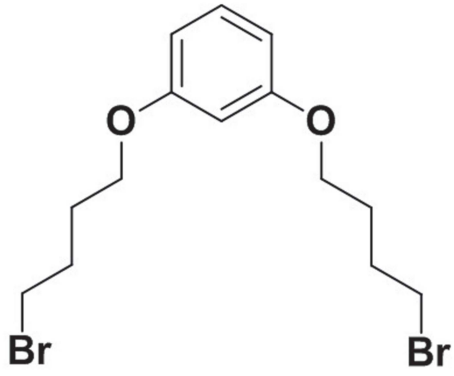
Structural data: full structural data are available from iucrdata.iucr.org

The whole molecule of the title compound, $C_{14}H_{20}Br_2O_2$, is generated by twofold rotational symmetry, with the twofold axis bisecting the benzene ring. The packing of the molecules features C—H$\cdots\pi$ interactions, which link the molecules to form chains along [100].

3D view



Chemical scheme



Structure description

Alkoxy-substituted benzenes are useful precursors in the synthesis of monodisperse aromatic oligomers (Lightowler & Hird, 2005). The *tert*-butoxy radicals plays an active role in initiating polymerization (Rizzardo & Solomon, 1979).

In the title compound (Fig. 1), the bromobutoxy side chains are attached to the benzene ring in positions 1 and 3. The asymmetric unit contains one-half of the molecule, the whole molecule being generated by twofold rotational symmetry. This twofold axis bisects the benzene ring at atoms C5 and C8. The dihedral angle between the benzene ring and the mean plane which best fits the atoms of the bromobutoxy side chain is 40.75°. The angle between the bonds [O1—C7 and C7a—O1a; symmetry code: (a) $-x+1, y, -z+\frac{1}{2}$] connecting the bromobutoxy side chains with the benzene ring is 69.9 (2)°. In the crystal, molecules are linked by C—H$\cdots\pi$ interactions, forming chains along the *a*-axis direction (Fig. 2 and Table 1).

Synthesis and crystallization

A mixture of resorcinol/hydroquinol (1.0 equivalent) and potassium carbonate (2.0 equivalents) in acetone (50 ml) was stirred for 15 min at 333 K. 1,3-Dibromobutane (2.1 equivalents) was added to the reaction mixture and stirred at 333 K for 7 h. After completion of the reaction (monitored by thin-layer chromatography), the solvent was

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

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4*B*···*Cg*1^{i}	0.97	2.81	3.664 (4)	147

Symmetry code: (i) $x-1, y, z$.

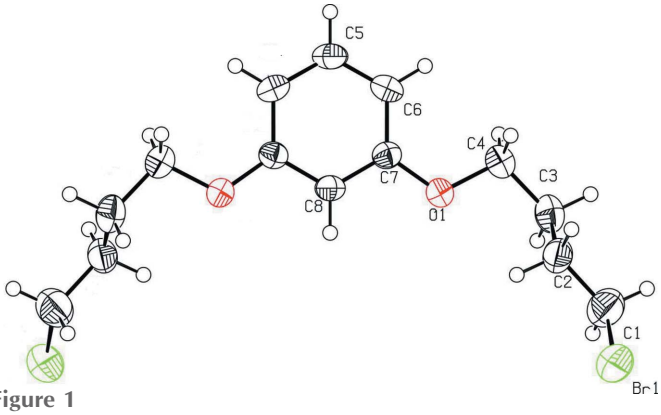


Figure 1
The molecular structure of the title compound, with the atom labelling. Displacement ellipsoids are drawn at the 50% probability level. Unlabelled atoms are related to the labelled atoms by the twofold rotation axis (symmetry code: $-x+1, y, -z+\frac{1}{2}$) that bisects atoms C5 and C8.

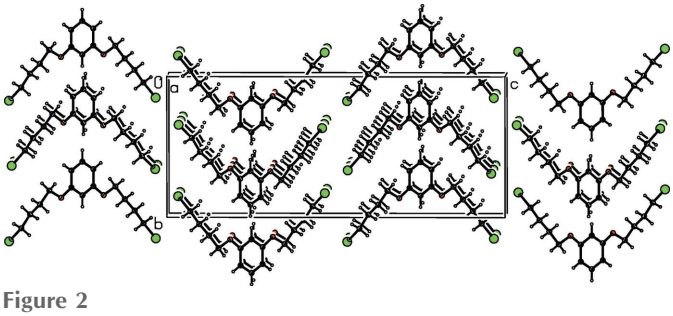


Figure 2
The crystal packing of the title compound, viewed along the *a* axis.

removed under reduced pressure and the residue was extracted with $CHCl_3$ (3 × 100 ml), then washed with water (2 × 100 ml) and brine (150 ml), and finally dried over anhydrous Na_2SO_4. The resulting solution was filtered and concentrated *in vacuo* and the residue obtained was purified by column chromatography using $CHCl_3$–hexane (1:9 *v*/*v*) as eluent. The white solid obtained was crystallized from methanol solution by slow evaporation, giving colourless block-like crystals.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{14}H_{20}Br_2O_2$
M_r	380.12
Crystal system, space group	Orthorhombic, *Pbcn*
Temperature (K)	296
a, *b*, *c* (Å)	4.8948 (3), 11.4976 (8), 27.636 (2)
V (Å^3)	1555.30 (18)
Z	4
Radiation type	Mo *Kα*
μ (mm^{-1})	5.21
Crystal size (mm)	0.35 × 0.30 × 0.25
Data collection	
Diffractometer	Bruker Kappa APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2004)
T_{min}, T_{max}	0.562, 0.745
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	17085, 1366, 974
R_{int}	0.035
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.033, 0.070, 1.08
No. of reflections	1366
No. of parameters	83
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.45, −0.39

Computer programs: *APEX2* (Bruker, 2004), *SAINT* (Bruker, 2004), *SHELXS97* (Sheldrick, 2008), *ORTEP-3 for Windows* (Farrugia, 2012), *SHELXL2016* (Sheldrick, 2015), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

Refinement

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

Acknowledgements

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full crystallographic data

IUCrData (2017). **2**, x171208 [https://doi.org/10.1107/S2414314617012081]

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Crystal data

$C_{14}H_{20}Br_2O_2$
M_r = 380.12
Orthorhombic, *Pbcn*
a = 4.8948 (3) Å
b = 11.4976 (8) Å
c = 27.636 (2) Å
V = 1555.30 (18) Å^3
Z = 4
F(000) = 760
D_x = 1.623 Mg m^{-3}
Mo *Kα* radiation, λ = 0.71073 Å
Cell parameters from 4934 reflections
θ = 5.9–48.6°
μ = 5.21 mm^{-1}
T = 296 K
Block, colourless
0.35 × 0.30 × 0.25 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Bruker axs kappa axes2 CCD Diffractometer scans
Absorption correction: multi-scan (SADABS; Bruker, 2004)
T_{min} = 0.562, T_{max} = 0.745
17085 measured reflections
1366 independent reflections
974 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
θ_{max} = 25.0°, θ_{min} = 3.0°
h = −5→5
k = −13→13
l = −32→27

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.070
S = 1.08
1366 reflections
83 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
$w = 1/[\sigma^2(F_o^2) + (0.0142P)^2 + 2.6984P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.11607 (11)	0.33877 (4)	0.03546 (2)	0.0782 (2)
O1	0.1750 (5)	0.64764 (18)	0.18910 (8)	0.0444 (6)
C8	0.500000	0.6559 (4)	0.250000	0.0356 (10)
H8	0.499996	0.575059	0.250002	0.043*
C6	0.3314 (7)	0.8365 (3)	0.21849 (12)	0.0418 (8)
H6	0.218658	0.877424	0.197420	0.050*
C7	0.3318 (6)	0.7156 (3)	0.21855 (11)	0.0346 (7)
C3	−0.1534 (8)	0.6095 (3)	0.12858 (13)	0.0534 (10)
H3A	−0.288821	0.645044	0.107659	0.064*
H3B	−0.249217	0.560942	0.151726	0.064*
C5	0.500000	0.8946 (4)	0.250000	0.0457 (12)
H5	0.499996	0.975466	0.250001	0.055*
C4	−0.0030 (7)	0.7038 (3)	0.15550 (12)	0.0463 (9)
H4A	0.101479	0.751205	0.133121	0.056*
H4B	−0.131214	0.753551	0.172471	0.056*
C1	−0.1216 (9)	0.4392 (4)	0.07311 (15)	0.0707 (12)
H1A	−0.256633	0.473929	0.051861	0.085*
H1B	−0.217731	0.392901	0.096989	0.085*
C2	0.0331 (8)	0.5336 (3)	0.09825 (13)	0.0520 (10)
H2A	0.125154	0.581386	0.074360	0.062*
H2B	0.171316	0.499101	0.118904	0.062*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.1073 (4)	0.0652 (3)	0.0622 (3)	0.0010 (3)	−0.0155 (3)	−0.0107 (2)
O1	0.0516 (15)	0.0376 (12)	0.0439 (12)	−0.0008 (12)	−0.0128 (11)	0.0034 (10)
C8	0.043 (3)	0.029 (2)	0.035 (2)	0.000	0.005 (2)	0.000
C6	0.045 (2)	0.0326 (16)	0.0474 (18)	0.0052 (18)	0.0027 (16)	0.0071 (16)
C7	0.0363 (19)	0.0331 (16)	0.0343 (16)	−0.0027 (15)	0.0045 (15)	0.0001 (14)
C3	0.045 (2)	0.066 (2)	0.050 (2)	0.001 (2)	−0.0093 (18)	0.0053 (19)
C5	0.046 (3)	0.029 (2)	0.061 (3)	0.000	0.002 (3)	0.000
C4	0.045 (2)	0.049 (2)	0.044 (2)	0.0043 (18)	−0.0043 (18)	0.0049 (17)
C1	0.076 (3)	0.068 (3)	0.068 (3)	−0.013 (3)	−0.007 (3)	−0.004 (2)
C2	0.057 (2)	0.055 (2)	0.044 (2)	−0.007 (2)	−0.0086 (19)	−0.0001 (18)

Geometric parameters (Å, º)

Br1—C1	1.942 (4)	C3—H3A	0.9700
O1—C7	1.364 (4)	C3—H3B	0.9700
O1—C4	1.428 (4)	C5—H5	0.9300
C8—C7^{i}	1.380 (4)	C4—H4A	0.9700
C8—C7	1.380 (4)	C4—H4B	0.9700
C8—H8	0.9300	C1—C2	1.494 (5)
C6—C5	1.373 (4)	C1—H1A	0.9700

C6—C7	1.390 (4)	C1—H1B	0.9700
C6—H6	0.9300	C2—H2A	0.9700
C3—C4	1.507 (5)	C2—H2B	0.9700
C3—C2	1.515 (5)		

C7—O1—C4	118.2 (2)	O1—C4—C3	107.1 (3)
C7^{i}—C8—C7	120.4 (4)	O1—C4—H4A	110.3
C7^{i}—C8—H8	119.8	C3—C4—H4A	110.3
C7—C8—H8	119.8	O1—C4—H4B	110.3
C5—C6—C7	119.0 (3)	C3—C4—H4B	110.3
C5—C6—H6	120.5	H4A—C4—H4B	108.6
C7—C6—H6	120.5	C2—C1—Br1	112.2 (3)
O1—C7—C8	115.3 (3)	C2—C1—H1A	109.2
O1—C7—C6	124.8 (3)	Br1—C1—H1A	109.2
C8—C7—C6	119.9 (3)	C2—C1—H1B	109.2
C4—C3—C2	113.2 (3)	Br1—C1—H1B	109.2
C4—C3—H3A	108.9	H1A—C1—H1B	107.9
C2—C3—H3A	108.9	C1—C2—C3	111.7 (3)
C4—C3—H3B	108.9	C1—C2—H2A	109.3
C2—C3—H3B	108.9	C3—C2—H2A	109.3
H3A—C3—H3B	107.8	C1—C2—H2B	109.3
C6^{i}—C5—C6	121.8 (4)	C3—C2—H2B	109.3
C6^{i}—C5—H5	119.1	H2A—C2—H2B	107.9
C6—C5—H5	119.1		

C4—O1—C7—C8	−179.7 (2)	C7—C6—C5—C6^{i}	0.0 (2)
C4—O1—C7—C6	0.2 (5)	C7—O1—C4—C3	−179.0 (3)
C7^{i}—C8—C7—O1	179.9 (3)	C2—C3—C4—O1	−64.1 (4)
C7^{i}—C8—C7—C6	0.0 (2)	Br1—C1—C2—C3	−178.5 (2)
C5—C6—C7—O1	−179.9 (2)	C4—C3—C2—C1	178.5 (3)
C5—C6—C7—C8	0.0 (4)		

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

Hydrogen-bond geometry (Å, º)

Cg1 is the centroid of the bezne ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4*B*···*Cg*1^{ii}	0.97	2.81	3.664 (4)	147

Symmetry code: (ii) *x*−1, *y*, *z*.