organic compounds

1,4-Bis(3-chloropropoxy)benzene

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The molecule of the title compound, $C_{12}H_{16}Cl_2O_2$, has a center of inversion at the centroid of the benzene ring and the asymmetric unit contains one half-molecule. Intermolecular C—H···π interactions stabilize the crystal structure.

Related literature

For general background to the use of alkoxybenzene derivatives as intermediates in organic synthesis, see: Dudones & Pearson *et al.* (2000); Chen & Chao (1996); Jin *et al.* (2010; Rabindranath *et al.* (2006); Zhang & Tieke (2008); Zhu *et al.* (2007). For bond-length data, see: Allen *et al.* (1987).
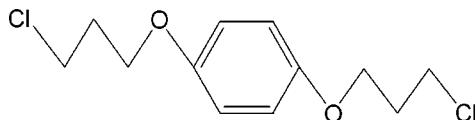
Experimental

Crystal data

$C_{12}H_{16}Cl_2O_2$
$M_r = 263.15$
Monoclinic, $P2_1/c$
$a = 4.9813 (8)$ Å
$b = 8.3200 (14)$ Å
$c = 15.273 (2)$ Å
$\beta = 93.156 (6)°$

$V = 632.02 (17)$ Å^3
$Z = 2$
Mo $K\alpha$ radiation
$\mu = 0.50$ mm^{-1}
$T = 113$ K
$0.22 \times 0.20 \times 0.18$ mm

Data collection

Rigaku Saturn diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2000)
$T_{min} = 0.899$, $T_{max} = 0.916$

5847 measured reflections
1502 independent reflections
1273 reflections with $I > 2\sigma(I)$
$R_{int} = 0.033$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.078$
$S = 1.07$
1502 reflections

73 parameters
H-atom parameters constrained
$\Delta\rho_{max} = 0.37$ e Å^{-3}
$\Delta\rho_{min} = -0.16$ e Å^{-3}

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

$Cg1$ is the centroid of the phenyl ring.

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4A···$Cg1^i$	0.99	2.74	3.577 (2)	143

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

Data collection: *CrystalClear* (Rigaku, 2000); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *CrystalStructure* (Rigaku, 2000); software used to prepare material for publication: *CrystalStructure*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BQ2255).

References

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supporting information

1,4-Bis(3-chloropropoxy)benzene

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S1. Comment

Alkoxybenzene derivatives are useful intermediates in organic synthesis (Dudones & Pearson, 2000; Chen *et al.*, 1996). Especially, halogenoalkoxybenzenes are used to synthesize diketopyrrolopyrrole derivatives which are a class of strongly fluorescent heterocyclic pigments and their structures could be easily optimized through variations of substituents at the 2,5- and 3,6-positions (Jin *et al.*, 2010; Rabindranath *et al.*, 2006; Zhang *et al.*, 2008; Zhu *et al.*, 2007). In this paper, the structure of the title compound synthesized, (I), and we report herein its crystal structure. In the molecule of (I) (Fig. 1) the bond lengths and angles are within normal ranges (Allen *et al.*, 1987). The asymmetric unit of the title compound contains a half of the molecule situated on a two-fold rotational axis. Intermolecular C4-H4···Cg1 interactions (Cg1 is the centroid of the phenyl ring ring) stabilize the crystal structure.

S2. Experimental

For the preparation of the title compound, p-dihydroxybenzene (11.0 g, 0.1 mol) was dissolved in dry acetone (100 ml). 1-Bromo-3-chloropropane (31.5 g, 0.2 mol) and potassium carbonate (138 g, 1mol) were added to this solution, the reaction was stirred under reflux for 11 h, The reaction mixture was filtered, the filtrate was concentrated, then washed with sodium hydroxide solution and extracted with ethyl acetate. After concentration, the residue was purified by recrystallization from chloroform (yield; 20.1 g, 76%, m.p. 339 K). Spectroscopic analysis: IR (KBr, v, cm^{-1}): 3085, 2957, 1541, 1351, 1032, 814. ^{1}HNMR (400 MHz, CDCl$_3$, ppm): 2.21 (m, 4H), 3.75 (t, 4H), 4.06 (t, 4H), 6.84 (d, 4H).

S3. Refinement

All H atoms were positioned geometrically and refined as riding (C-H = 0.95-0.99Å) and allowed to ride on their parent atoms, with U_{iso}(H) =1.2U_{eq}(parent).
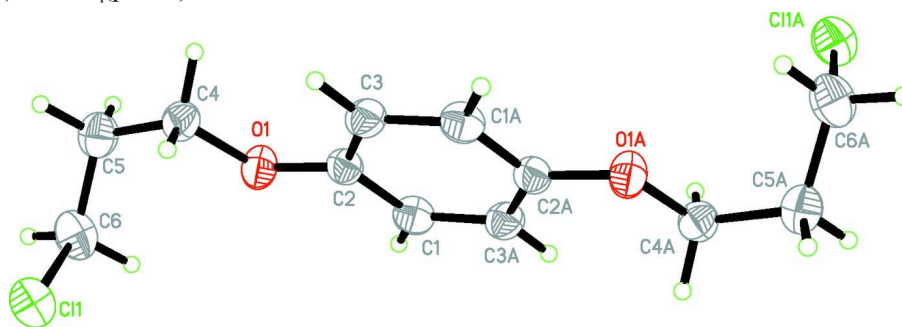

Figure 1

The molecular structure of the title molecule, with the atom-numbering scheme, symmetry code: -x+1, -y, -z+1. Displacement ellipsoids are drawn at the 75% probability level.
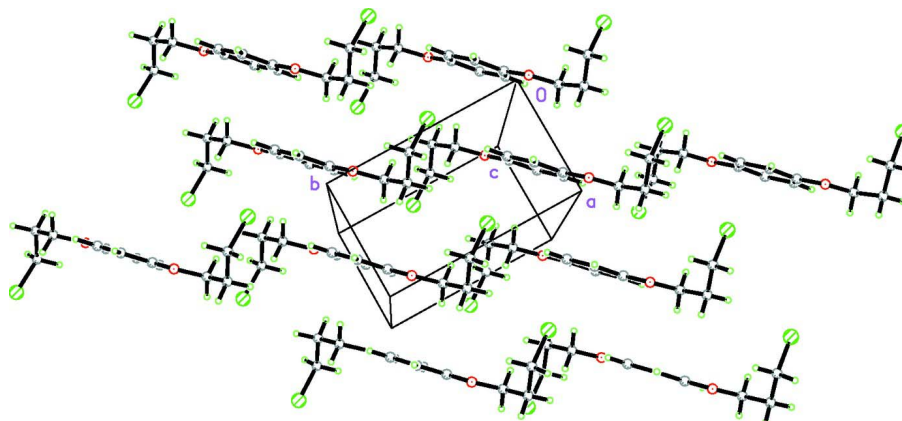
Figure 2
Part of the crystal structure of the title compound showing molecules being stacked along the c-axis.

1,4-Bis(3-chloropropoxy)benzene

Crystal data

$C_{12}H_{16}Cl_2O_2$	$F(000) = 276$
$M_r = 263.15$	$D_x = 1.383$ Mg m^{-3}
Monoclinic, $P2_1/c$	Melting point: 339 K
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation, $\lambda = 0.71075$ Å
$a = 4.9813$ (8) Å	Cell parameters from 2060 reflections
$b = 8.3200$ (14) Å	$\theta = 2.7–27.9°$
$c = 15.273$ (2) Å	$\mu = 0.50$ mm^{-1}
$\beta = 93.156$ (6)°	$T = 113$ K
$V = 632.02$ (17) Å^3	Prism, colorless
$Z = 2$	$0.22 \times 0.20 \times 0.18$ mm

Data collection

Rigaku Saturn diffractometer	5847 measured reflections
Radiation source: rotating anode	1502 independent reflections
Multilayer monochromator	1273 reflections with $I > 2\sigma(I)$
Detector resolution: 14.222 pixels mm^{-1}	$R_{int} = 0.033$
ω scans	$\theta_{max} = 27.9°$, $\theta_{min} = 2.7°$
Absorption correction: multi-scan	$h = -4 \rightarrow 6$
(*CrystalClear*; Rigaku, 2000)	$k = -10 \rightarrow 9$
$T_{min} = 0.899$, $T_{max} = 0.916$	$l = -20 \rightarrow 20$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.031$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.078$	
$S = 1.07$	H-atom parameters constrained
1502 reflections	$w = 1/[\sigma^2(F_o^2) + (0.0407P)^2 +]$
73 parameters	where $P = (F_o^2 + 2F_c^2)/3$
0 restraints	$(\Delta/\sigma)_{max} < 0.001$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{max} = 0.37$ e Å^{-3}
	$\Delta\rho_{min} = -0.16$ e Å^{-3}

Special details

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

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 (Å^2)

	x	y	z	$U_{iso}*/U_{eq}$
Cl1	0.22691 (7)	0.61216 (4)	0.66618 (2)	0.02618 (13)
O1	0.16835 (18)	0.17363 (11)	0.60468 (6)	0.0199 (2)
C1	0.5063 (2)	−0.01858 (15)	0.59005 (8)	0.0186 (3)
H1	0.5102	−0.0314	0.6519	0.022*
C2	0.3281 (3)	0.09041 (14)	0.54960 (8)	0.0167 (3)
C3	0.3213 (3)	0.10898 (15)	0.45872 (9)	0.0185 (3)
H3	0.1997	0.1829	0.4304	0.022*
C4	−0.0045 (3)	0.29417 (15)	0.56500 (9)	0.0194 (3)
H4A	−0.1349	0.2444	0.5218	0.023*
H4B	0.1027	0.3745	0.5342	0.023*
C5	−0.1508 (3)	0.37389 (16)	0.63743 (9)	0.0226 (3)
H5A	−0.2664	0.2930	0.6644	0.027*
H5B	−0.2697	0.4587	0.6114	0.027*
C6	0.0331 (3)	0.44827 (16)	0.70864 (9)	0.0250 (3)
H6A	0.1569	0.3651	0.7337	0.030*
H6B	−0.0759	0.4887	0.7562	0.030*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0298 (2)	0.0224 (2)	0.0265 (2)	−0.00092 (14)	0.00272 (15)	−0.00187 (14)
O1	0.0221 (5)	0.0210 (5)	0.0167 (5)	0.0060 (4)	0.0016 (4)	0.0003 (4)
C1	0.0212 (7)	0.0210 (7)	0.0135 (6)	−0.0013 (5)	0.0005 (5)	0.0011 (5)
C2	0.0159 (7)	0.0165 (6)	0.0178 (7)	−0.0012 (5)	0.0015 (5)	−0.0018 (5)
C3	0.0184 (7)	0.0174 (6)	0.0193 (7)	0.0010 (5)	−0.0021 (5)	0.0016 (5)
C4	0.0199 (7)	0.0191 (6)	0.0190 (7)	0.0029 (5)	−0.0014 (5)	0.0003 (5)
C5	0.0224 (7)	0.0224 (7)	0.0234 (8)	0.0019 (5)	0.0043 (6)	0.0004 (6)
C6	0.0318 (8)	0.0227 (7)	0.0210 (7)	−0.0005 (6)	0.0056 (6)	−0.0005 (6)

Geometric parameters (Å, º)

Cl1—C6	1.8113 (14)	C4—C5	1.5111 (17)
O1—C2	1.3762 (14)	C4—H4A	0.9900
O1—C4	1.4342 (15)	C4—H4B	0.9900
C1—C3^i	1.3887 (17)	C5—C6	1.5151 (19)
C1—C2	1.3897 (17)	C5—H5A	0.9900

C1—H1	0.9500	C5—H5B	0.9900
C2—C3	1.3951 (18)	C6—H6A	0.9900
C3—C1ⁱ	1.3886 (17)	C6—H6B	0.9900
C3—H3	0.9500		
C2—O1—C4	116.58 (10)	C5—C4—H4B	110.2
C3ⁱ—C1—C2	120.91 (12)	H4A—C4—H4B	108.5
C3ⁱ—C1—H1	119.5	C4—C5—C6	114.07 (11)
C2—C1—H1	119.5	C4—C5—H5A	108.7
O1—C2—C1	115.67 (11)	C6—C5—H5A	108.7
O1—C2—C3	124.69 (12)	C4—C5—H5B	108.7
C1—C2—C3	119.63 (11)	C6—C5—H5B	108.7
C1ⁱ—C3—C2	119.46 (12)	H5A—C5—H5B	107.6
C1ⁱ—C3—H3	120.3	C5—C6—Cl1	111.33 (9)
C2—C3—H3	120.3	C5—C6—H6A	109.4
O1—C4—C5	107.47 (11)	Cl1—C6—H6A	109.4
O1—C4—H4A	110.2	C5—C6—H6B	109.4
C5—C4—H4A	110.2	Cl1—C6—H6B	109.4
O1—C4—H4B	110.2	H6A—C6—H6B	108.0
C4—O1—C2—C1	176.07 (11)	C1—C2—C3—C1ⁱ	−0.2 (2)
C4—O1—C2—C3	−3.86 (18)	C2—O1—C4—C5	−177.69 (10)
C3ⁱ—C1—C2—O1	−179.68 (10)	O1—C4—C5—C6	57.71 (14)
C3ⁱ—C1—C2—C3	0.2 (2)	C4—C5—C6—Cl1	64.36 (13)
O1—C2—C3—C1ⁱ	179.68 (11)		

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

Hydrogen-bond geometry (Å, º)

Cg1 is the centroid of the phenyl ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4*A*···*Cg*1ⁱⁱ	0.99	2.74	3.577 (2)	143

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