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The crystal structure of 1,2-bis(3-bromophenoxy) ethane, $C_{14}H_{12}Br_2O_2$

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Abstract

$C_{14}H_{12}Br_2O_2$, monoclinic, $C2/c$ (no. 15), $a = 27.323(3)$ Å, $b = 5.8516(6)$ Å, $c = 8.9145(10)$ Å, $\beta = 103.596(3)^\circ$, $V = 1385.4(3)$ Å³, $Z = 4$, $R_{gt}(F) = 0.0378$, $wR_{ref}(F^2) = 0.1019$, $T = 296(2)$ K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

All chemicals, reagents and solvents are of analytical grade and are commercially available. **Preparation of 1,2-bis(3-bromophenoxy) ethane:** Potassium carbonate

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	$0.22 \times 0.20 \times 0.18$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	5.84 mm ⁻¹
Diffraction, scan mode:	APEX-CCD, φ and ω
θ_{max} , completeness:	28.8° , $>99\%$
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	5705, 1782, 0.038
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 1172
$N(param)_{refined}$:	82
Programs:	Bruker [1], SHELX [2]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	U_{iso}^*/U_{eq}
Br1	0.213396(13)	1.07237(6)	0.64516(5)	0.0711(2)
C1	0.11508(11)	0.9158(5)	0.6382(3)	0.0424(7)
H1	0.1133	1.0448	0.6977	0.051*
C2	0.15822(11)	0.8665(5)	0.5889(4)	0.0449(7)
C3	0.16222(13)	0.6788(6)	0.5015(4)	0.0575(9)
H3	0.1917	0.6492	0.4702	0.069*
C4	0.12132(15)	0.5342(6)	0.4608(4)	0.0602(9)
H4	0.1234	0.4058	0.4012	0.072*
C5	0.07754(14)	0.5764(5)	0.5066(4)	0.0509(8)
H5	0.0502	0.4780	0.4777	0.061*
C6	0.07448(11)	0.7675(5)	0.5964(3)	0.0411(7)
C7	0.02557(12)	0.9876(6)	0.7337(4)	0.0504(8)
H7A	0.0309	1.1288	0.6828	0.060*
H7B	0.0511	0.9764	0.8295	0.060*
O1	0.02939(8)	0.7980(4)	0.6370(3)	0.0526(6)

(23.9 g, 0.173 mol), *m*-bromophenol (10 g, 0.058 mol) and 1,2-dibromoethane (21.8 g, 0.116 mol) were dissolved in 100 mL DMF (dimethylformamide) and the reaction solution was stirred at 60 °C. The progress of the reaction was monitored by TLC. The reaction mixture was poured into water, and then extracted with ethyl acetate. The organic phase was dehydrated with anhydrous sodium sulfate and concentrated at reduced pressure to afford a light yellow oil. Crystallisation was undertaken from hexane at room temperature to afford an analytically pure product (9.3 g, 43%).

Experimental details

All hydrogen atoms were placed in geometrically calculated positions. The U_{iso} values of the hydrogen atoms of methyl

groups were set to 1.5 $U_{eq}(C_{methyl})$ and the U_{iso} values of all other hydrogen atoms were set to 1.2 $U_{eq}(C)$.

Comment

1,2-bis(3-bromophenoxy) ethane and its derivatives can be used as an important intermediate in the synthesis of crown ether [3], macrocyclic C-glycosyl compounds as chiral [4], poly(*p*-phenylene), which conducts electricity when doped with oxidizing or reducing agents [5] and so on. The structure of the title compound was elucidated by X-ray diffraction.

There is one half of the title molecule in the asymmetric unit. The molecule is arranged around a twofold axis in the monoclinic space group no.15. Bond lengths and angles are in the expected ranges [6].

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