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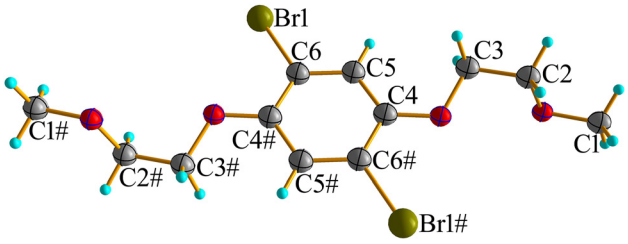
Crystal structure of 1,4-dibromo-2,5-bis(2-methoxyethoxy)benzene-1,4-diol, $C_{12}H_{16}Br_2O_4$



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Abstract

$C_{12}H_{16}Br_2O_4$, monoclinic, $P2_1/n$ (no. 14), a = 12.594(5) Å, b = 4.4613(16) Å, c = 12.878(4) Å, β = 91.209(12)°, V = 723.4(4) Å^3, Z = 2, $R_{gt}(F)$ = 0.0450, $wR_{ref}(F^2)$ = 0.1103, T = 293 (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.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.16 × 0.14 × 0.13 mm
Wavelength:	MoKα radiation (0.71073 Å)
μ:	5.61 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	25.0°, 99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	4000, 1256, 0.066
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 883
$N(param)_{refined}$:	83
Programs:	Olex2 [1], Bruker [2], SHELX [3], Diamond [4]

1 Source of materials

Weigh 2,5-dibromobenzene-1,4-diol (1.33 g, 5 mmol) and 2-bromoethyl methyl ether (3.48 g, 25 mmol) into a 100 mL circular bottom flask, add 60 mL of N, N-dimethylformamide, react at 100 °C for 72 h, cool to room temperature, filter, remove solvent under reduced pressure, separate by column chromatography, and use dichloromethane as eluent to obtain a white solid. Take a small amount of the solid, dissolve it in ethanol, let it evaporate at room temperature for 7 days, and grow crystals.

2 Experimental details

Using Olex2 [1], the structure was solved using Charge Flipping and refined with the ShelXL [3] refinement. All hydrogen atoms were positioned geometrically, with the d(C–H) = 0.97–0.99 Å, U_{iso}(H) = 1.2 times U_{eq}(C) and U_{iso}(H) = 1.5 times U_{eq}(O).

3 Comment

The bromine atom of dibromobenzene is prone to react with vinyl pyridine to form compounds with large conjugated structures, which have excellent luminescent properties and have attracted the attention of researchers [5]. Modifying the alkyl chain on the benzene ring can regulate the hydrophobicity of the compound [6] and may be useful as an intermediate for synthesizing polymer materials [7]. The asymmetric unit of the title compound, $C_{12}H_{16}Br_2O_4$, contains one half molecule located around an inversion center. The bond lengths and bond angles are within the normal range, and literature reports that alkyl chains with similar structures have a serrated structure [8]. Due to the presence of oxygen atoms in the alkyl chain of this target compound, it is not a serrated structure. The molecules contain atypical C1–H1B···Br1 hydrogen bonds.

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Br1	1.19091 (5)	0.22701 (13)	0.64583 (4)	0.0550 (3)
O2	0.7896 (3)	0.4046 (8)	0.5450 (3)	0.0479 (10)
O1	0.5907 (3)	0.3804 (9)	0.6528 (3)	0.0630 (11)
C1	0.4789 (5)	0.3470 (15)	0.6446 (6)	0.080 (2)
H1A	0.4577	0.3365	0.5726	0.119*
H1B	0.4452	0.5156	0.6764	0.119*
H1C	0.4582	0.1662	0.6791	0.119*
C2	0.6449 (5)	0.1354 (12)	0.6104 (4)	0.0488 (15)
H2A	0.6238	0.1116	0.5380	0.059*
H2B	0.6257	−0.0462	0.6470	0.059*
C3	0.7619 (5)	0.1801 (11)	0.6187 (4)	0.0443 (14)
H3A	0.7820	0.2440	0.6884	0.053*
H3B	0.7985	−0.0059	0.6039	0.053*
C4	0.8939 (4)	0.4446 (11)	0.5251 (4)	0.0364 (12)
C6	1.0814 (4)	0.3853 (11)	0.5603 (4)	0.0371 (12)
C5	0.9769 (4)	0.3302 (10)	0.5863 (4)	0.0393 (13)
H5	0.9622	0.2164	0.6447	0.047*

Author contribution: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Competing interest: The authors declare no conflicts of interest regarding this article.

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