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Bis(2-bromobenzyl) ether

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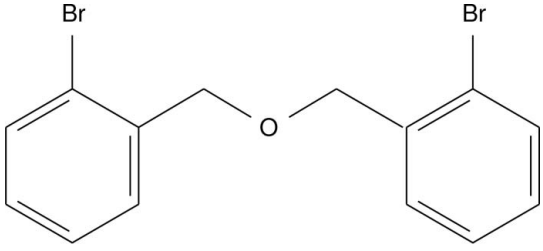
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.009 Å; R factor = 0.075; wR factor = 0.204; data-to-parameter ratio = 14.1.

In the title compound, $C_{14}H_{12}Br_2O$, the dihedral angle between the aromatic rings is 2.7 (3)° and the Br atoms lie on the same side of the molecule. No intermolecular interactions occur in the crystal beyond van der Waals contacts.

Related literature

For the use of benzyl groups in organic synthesis, see; Rao & Kumar (2001); Tareque *et al.* (2006).



Experimental

Crystal data

$C_{14}H_{12}Br_2O$
M_r = 356.04
Monoclinic, $P2_1/n$
a = 11.6022 (6) Å
b = 10.1590 (5) Å
c = 12.2368 (6) Å
β = 112.853 (2)°
V = 1329.10 (12) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 7.58 mm^{-1}
T = 296 K
0.23 × 0.22 × 0.21 mm

Data collection

Bruker X8 Proteum diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2013)
T_{min} = 0.275, T_{max} = 0.299
10361 measured reflections
2185 independent reflections
1957 reflections with $I > 2\sigma(I)$
R_{int} = 0.054

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.073
$wR(F^2)$ = 0.192
S = 1.07
2185 reflections
155 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.26 e Å^{-3}
$\Delta\rho_{min}$ = −1.61 e Å^{-3}

Data collection: *APEX2* (Bruker, 2013); cell refinement: *SAINT* (Bruker, 2013); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *Mercury*.

We are grateful to the IOE, University of Mysore, for providing the single-crystal X-ray diffraction facility. PN thanks the BET Academy of Higher Education for the facilities.

Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7230).

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supporting information

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S1. Comment

Benzyl groups are commonly used for the protection of alcohol and phenol moieties for synthesis. The benzyl alcohol used in the benzylation of phenol (Tareque, *et al.*,, 2006). The benzyl ethers are used as intermediates in sigmatropic rearrangement reactions such as Claisen and the Cope rearrangements (Rao and Kumar, 2001).

In the title compound, $C_{14}H_{10}Br_2O$, (Fig. 1), the dihedral angle between the aromatic rings is 2.7 (3)° and the Br atoms lie on the same side of the molecule. No intermolecular interactions occur in the crystal beyond van der Waals′ contacts.

S2. Experimental

2-Bromobenzyl alcohol (1.87 g, 0.01 mol), sodium hydride 0.24 g, 0.01 mol) and 2-bromobenzyl bromide (2.52 g, 0.01 mol) were ground well and mixed in 25 ml of THF. The mixture were stirred in a beaker at 60 °C for one hour. The mixture was kept aside for five days at room temperature in a vaccum desiccator over phosphorous pentoxide. The colourless crystals were obtained by slow evaporation (*M*. P. 374 - 376 K). Colourless blocks were obtained from slow evaporation of a solution of ethylacetate.

S3. Refinement

The hydrogen atom were fixed geometrically (C—H=0.93–0.96 Å) and allowed to ride on their parent atoms with U_{iso}(H) = 1.2U_{eq}(C).

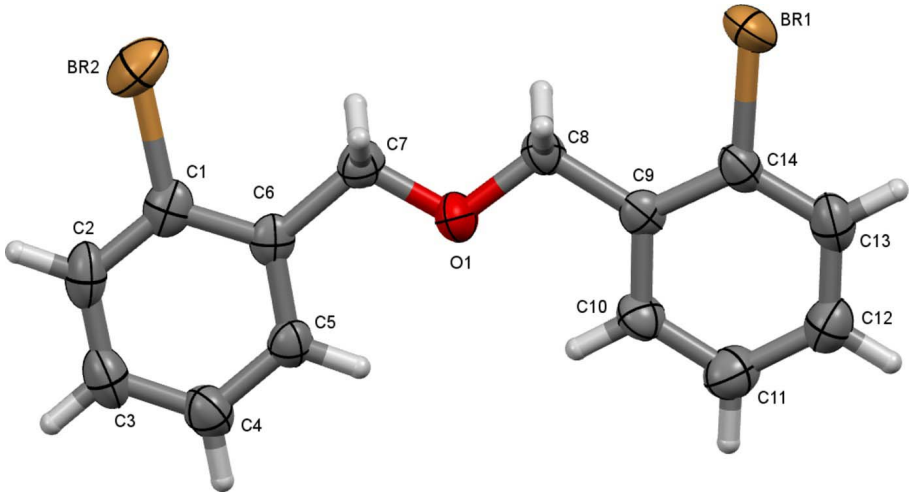

Figure 1

A view of the title molecule, with displacement ellipsoids drawn at the 50% probability level.

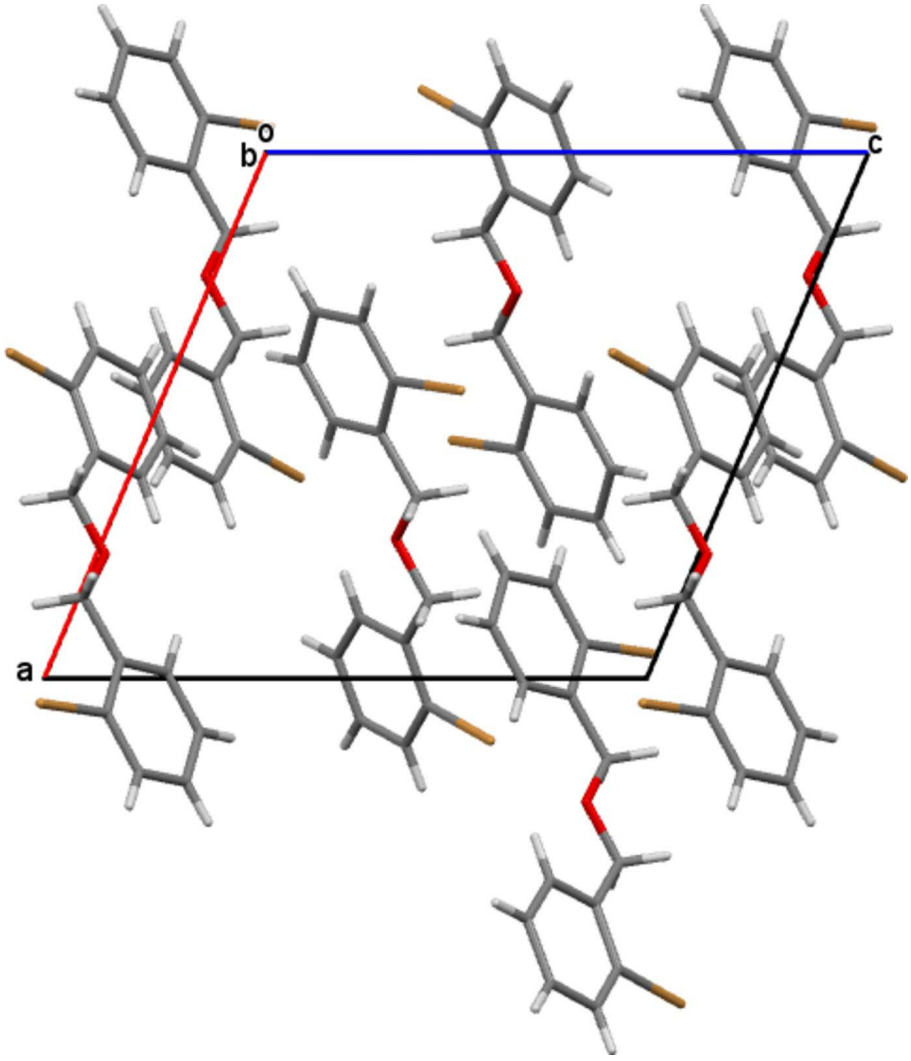

Figure 2

A viewed along the *b* axis of the crystal packing of the title compound.

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Crystal data

$C_{14}H_{12}Br_2O$
$M_r = 356.04$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 11.6022\ (6)$ Å
$b = 10.1590\ (5)$ Å
$c = 12.2368\ (6)$ Å
$\beta = 112.853\ (2)°$
$V = 1329.10\ (12)$ Å^3
$Z = 4$
$F(000) = 688$
$D_x = 1.769$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å
Cell parameters from 2185 reflections
$\theta = 4.5–64.7°$
$\mu = 7.58$ mm^{-1}
$T = 296$ K
Block, colourless
0.23 × 0.22 × 0.21 mm

Data collection

Bruker X8 Proteum diffractometer
Radiation source: Bruker MicroStar microfocus rotating anode
Helios multilayer optics monochromator
Detector resolution: 10.7 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2013)
T_{min} = 0.275, T_{max} = 0.299
10361 measured reflections
2185 independent reflections
1957 reflections with $I > 2\sigma(I)$
R_{int} = 0.054
θ_{max} = 64.7°, θ_{min} = 4.5°
h = −5→13
k = −11→11
l = −14→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.073
$wR(F^2)$ = 0.192
S = 1.07
2185 reflections
155 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
$w = 1/[\sigma^2(F_o^2) + (0.137P)^2 + 1.9645P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 1.26 e Å^{-3}
$\Delta\rho_{min}$ = −1.61 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), FC*=KFC[1+0.001XFC^{2}Λ^3/SIN(2Θ)]$^{-1/4}$
Extinction coefficient: 0.0219 (17)

Special details

Geometry. Bond distances, angles etc. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell esds are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted R-factors wR and all goodnesses 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 observed criterion of F^2 > 2sigma(F^2) is used only for calculating -R-factor-obs 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}
Br1	0.45265 (7)	0.18786 (6)	0.48911 (7)	0.0610 (3)
Br2	1.11871 (7)	0.50492 (7)	0.78422 (6)	0.0629 (4)
O1	0.7350 (4)	0.4954 (4)	0.4857 (4)	0.0441 (14)
C1	1.0514 (5)	0.6241 (6)	0.6552 (5)	0.0410 (17)
C2	1.1258 (5)	0.7260 (7)	0.6461 (6)	0.051 (2)
C3	1.0785 (6)	0.8126 (6)	0.5532 (6)	0.052 (2)
C4	0.9584 (6)	0.7975 (6)	0.4701 (6)	0.0488 (19)
C5	0.8845 (5)	0.6954 (5)	0.4814 (5)	0.0391 (17)
C6	0.9296 (5)	0.6058 (5)	0.5729 (5)	0.0348 (16)
C7	0.8506 (5)	0.4919 (5)	0.5835 (5)	0.0389 (17)
C8	0.6547 (5)	0.3941 (5)	0.4916 (5)	0.0384 (16)
C9	0.5324 (5)	0.4041 (5)	0.3857 (5)	0.0350 (16)
C10	0.5112 (5)	0.5001 (5)	0.3003 (5)	0.0411 (17)
C11	0.3985 (6)	0.5106 (7)	0.2048 (6)	0.053 (2)

C12	0.3028 (6)	0.4217 (6)	0.1926 (5)	0.0499 (17)
C13	0.3212 (6)	0.3256 (6)	0.2769 (6)	0.0493 (19)
C14	0.4339 (5)	0.3178 (5)	0.3721 (5)	0.0401 (16)
H2	1.20690	0.73590	0.70220	0.0610*
H3	1.12770	0.88170	0.54620	0.0630*
H4	0.92700	0.85570	0.40670	0.0590*
H5	0.80290	0.68710	0.42610	0.0460*
H7A	0.89290	0.40930	0.58440	0.0470*
H7B	0.83730	0.49890	0.65680	0.0470*
H8A	0.64000	0.40210	0.56410	0.0460*
H8B	0.69290	0.30910	0.49190	0.0460*
H10	0.57470	0.55950	0.30740	0.0490*
H11	0.38640	0.57660	0.14880	0.0630*
H12	0.22690	0.42740	0.12790	0.0600*
H13	0.25770	0.26610	0.26950	0.0590*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0563 (6)	0.0420 (5)	0.0818 (7)	−0.0090 (3)	0.0236 (4)	0.0184 (3)
Br2	0.0562 (6)	0.0755 (7)	0.0436 (6)	0.0143 (3)	0.0047 (4)	−0.0006 (3)
O1	0.031 (2)	0.047 (2)	0.054 (3)	−0.0052 (16)	0.0161 (18)	0.0079 (16)
C1	0.039 (3)	0.048 (3)	0.038 (3)	0.004 (2)	0.017 (2)	−0.014 (2)
C2	0.035 (3)	0.058 (4)	0.058 (4)	−0.009 (3)	0.015 (3)	−0.025 (3)
C3	0.046 (3)	0.047 (4)	0.068 (4)	−0.014 (3)	0.028 (3)	−0.016 (3)
C4	0.048 (3)	0.039 (3)	0.061 (4)	−0.003 (2)	0.023 (3)	−0.004 (3)
C5	0.033 (3)	0.037 (3)	0.046 (3)	0.002 (2)	0.014 (2)	−0.004 (2)
C6	0.031 (2)	0.037 (3)	0.041 (3)	0.0037 (19)	0.019 (2)	−0.011 (2)
C7	0.034 (3)	0.043 (3)	0.041 (3)	0.003 (2)	0.016 (2)	0.002 (2)
C8	0.030 (2)	0.037 (3)	0.052 (3)	−0.001 (2)	0.020 (2)	0.007 (2)
C9	0.033 (2)	0.033 (3)	0.046 (3)	0.004 (2)	0.023 (2)	−0.002 (2)
C10	0.042 (3)	0.043 (3)	0.044 (3)	−0.003 (2)	0.023 (3)	0.005 (2)
C11	0.048 (4)	0.062 (4)	0.052 (4)	0.008 (3)	0.023 (3)	0.011 (3)
C12	0.040 (3)	0.056 (3)	0.049 (3)	0.003 (3)	0.012 (2)	−0.002 (3)
C13	0.039 (3)	0.045 (3)	0.064 (4)	−0.008 (2)	0.020 (3)	−0.013 (3)
C14	0.037 (3)	0.029 (2)	0.060 (3)	−0.0028 (19)	0.025 (3)	−0.004 (2)

Geometric parameters (Å, º)

Br1—C14	1.897 (5)	C11—C12	1.394 (10)
Br2—C1	1.900 (6)	C12—C13	1.375 (9)
O1—C7	1.409 (8)	C13—C14	1.375 (9)
O1—C8	1.409 (7)	C2—H2	0.9300
C1—C2	1.380 (9)	C3—H3	0.9300
C1—C6	1.393 (8)	C4—H4	0.9300
C2—C3	1.372 (9)	C5—H5	0.9300
C3—C4	1.378 (10)	C7—H7A	0.9700
C4—C5	1.386 (9)	C7—H7B	0.9700

C5—C6	1.379 (8)	C8—H8A	0.9700
C6—C7	1.513 (8)	C8—H8B	0.9700
C8—C9	1.508 (8)	C10—H10	0.9300
C9—C10	1.380 (8)	C11—H11	0.9300
C9—C14	1.398 (8)	C12—H12	0.9300
C10—C11	1.378 (9)	C13—H13	0.9300

C7—O1—C8	111.6 (4)	C2—C3—H3	120.00
Br2—C1—C2	118.5 (5)	C4—C3—H3	120.00
Br2—C1—C6	119.3 (4)	C3—C4—H4	120.00
C2—C1—C6	122.2 (6)	C5—C4—H4	120.00
C1—C2—C3	119.1 (6)	C4—C5—H5	119.00
C2—C3—C4	120.3 (6)	C6—C5—H5	119.00
C3—C4—C5	119.8 (6)	O1—C7—H7A	110.00
C4—C5—C6	121.4 (6)	O1—C7—H7B	110.00
C1—C6—C5	117.2 (5)	C6—C7—H7A	110.00
C1—C6—C7	121.2 (5)	C6—C7—H7B	110.00
C5—C6—C7	121.5 (5)	H7A—C7—H7B	108.00
O1—C7—C6	108.5 (4)	O1—C8—H8A	110.00
O1—C8—C9	109.1 (4)	O1—C8—H8B	110.00
C8—C9—C10	122.0 (5)	C9—C8—H8A	110.00
C8—C9—C14	120.9 (5)	C9—C8—H8B	110.00
C10—C9—C14	117.1 (5)	H8A—C8—H8B	108.00
C9—C10—C11	121.9 (6)	C9—C10—H10	119.00
C10—C11—C12	119.7 (6)	C11—C10—H10	119.00
C11—C12—C13	119.6 (6)	C10—C11—H11	120.00
C12—C13—C14	119.7 (6)	C12—C11—H11	120.00
Br1—C14—C9	119.9 (4)	C11—C12—H12	120.00
Br1—C14—C13	118.2 (5)	C13—C12—H12	120.00
C9—C14—C13	122.0 (5)	C12—C13—H13	120.00
C1—C2—H2	120.00	C14—C13—H13	120.00
C3—C2—H2	120.00		

C8—O1—C7—C6	−178.2 (5)	C5—C6—C7—O1	2.3 (7)
C7—O1—C8—C9	179.3 (5)	O1—C8—C9—C10	0.5 (7)
Br2—C1—C2—C3	179.8 (5)	O1—C8—C9—C14	−177.6 (5)
C6—C1—C2—C3	0.2 (10)	C8—C9—C10—C11	−178.7 (6)
Br2—C1—C6—C5	179.5 (4)	C14—C9—C10—C11	−0.6 (9)
Br2—C1—C6—C7	−0.8 (8)	C8—C9—C14—Br1	1.0 (7)
C2—C1—C6—C5	−0.9 (9)	C8—C9—C14—C13	179.5 (6)
C2—C1—C6—C7	178.9 (6)	C10—C9—C14—Br1	−177.2 (4)
C1—C2—C3—C4	−0.2 (10)	C10—C9—C14—C13	1.3 (9)
C2—C3—C4—C5	0.9 (10)	C9—C10—C11—C12	−0.5 (10)
C3—C4—C5—C6	−1.7 (10)	C10—C11—C12—C13	0.9 (10)
C4—C5—C6—C1	1.6 (9)	C11—C12—C13—C14	−0.3 (10)
C4—C5—C6—C7	−178.2 (6)	C12—C13—C14—Br1	177.7 (5)
C1—C6—C7—O1	−177.4 (5)	C12—C13—C14—C9	−0.9 (10)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5···O1	0.93	2.32	2.685 (7)	103
C10—H10···O1	0.93	2.34	2.705 (8)	103