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Bis(2-bromo-5-methylphenoxy)methane

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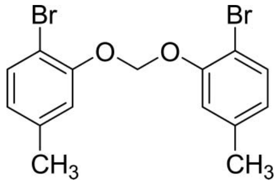
Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.012 Å; R factor = 0.055; wR factor = 0.123; data-to-parameter ratio = 11.6.

The complete molecule of the title compund, $C_{15}H_{14}Br_2O_2$, is generated by the application of crystallographic twofold symmetry, with the central C atom lying on the rotation axis. The dihedral angle between the benzene rings is 62.4 (3)°. In the crystal, short Br···Br contacts [3.4885 (16) Å] occur.

Related literature

For background to bromoaromatic compounds, see: Butler & Walker (1993); Seevers & Counsell (1982). For a related structure, see: Zheng *et al.* (2004).



Experimental

Crystal data

$C_{15}H_{14}Br_2O_2$
M_r = 386.08
Orthorhombic, $P2_12_12$
a = 10.7752 (11) Å
b = 15.8690 (17) Å
c = 4.3272 (10) Å
V = 739.9 (2) Å^3
Z = 2
Cu $K\alpha$ radiation
μ = 6.91 mm^{-1}
T = 291 K
0.35 × 0.30 × 0.30 mm

Data collection

Agilent Xcalibur Eos Gemini diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2011) $T_{\min}$ = 0.196, $T_{\max}$ = 0.231
1505 measured reflections
1022 independent reflections
754 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.044

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.123
S = 1.02
1022 reflections
88 parameters
H-atom parameters constrained
$\Delta\rho_{\max}$ = 0.36 e Å^{-3}
$\Delta\rho_{\min}$ = −0.41 e Å^{-3}
Absolute structure: Flack (1983), 212 Friedel pairs
Flack parameter: −0.11 (9)

Data collection: *CrysAlis PRO* (Agilent, 2011); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6383).

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supporting information

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S1. Comment

Bromoaromatic compounds have proven to be an important class of molecules in synthetic organic chemistry. They have been used as key intermediates in the preparation of oganometallic reagents and play vital roles in transition metal mediated coupling reactions (Butler *et al.*, 1993; Seevers *et al.*, 1982). In this paper, we synthesized the title compound and reported its crystal structure here. The title compound was synthesized by the reaction of 2-bromo-4-methylphenol, dibromomethane with potassium carbonate. The C—C—C angles within the aromatic moiety cover a range 117.7 (8) - 122.5 (8) °, and the two benzene rings make a dihedral angle of 62.5° (Fig. 1). The O and Br atoms are essentially coplanar with the benzene ring to which they are attached, with the deviation of 0.0074 Å. In addition, the benzene rings between the adjacent molecules are stacked in a face-to-face orientation with the distance of 3.701 Å, a distance longer than the π–π stacking distances of 3.33 - 3.53 Å reported elsewhere (Zheng *et al.*, 2004), indicating no π–π stacking is observed for this compound.

S2. Experimental

A mixture of 2-bromo-4-methylphenol (188 mg, 1 mmol), potassium carbonate (691 mg, 5 mmol) and dibromomethane (0.75 mmol) in acetone (5 ml) was heated to reflux for 12 h. The product was isolated and recrystallized from dicholomethane/hexane, colorless prisms of the title compound were obtained.

S3. Refinement

H atoms were generated geometrically and refined as riding atoms with C-H = 0.93Å and Uiso(H) = 1.2 times Ueq(C).

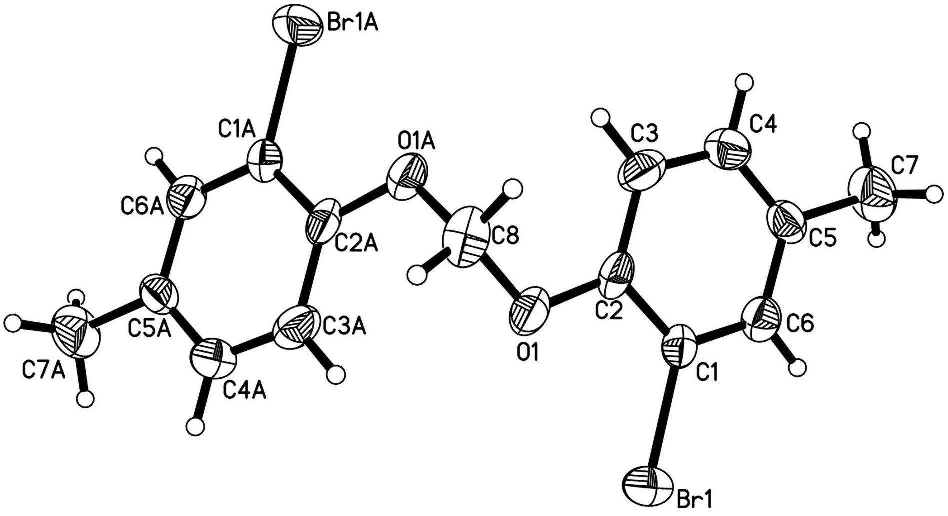

Figure 1

View of the title compound, showing 30% probability ellipsolids. Atoms with suffix A are generated by (1–x, 2–y, z0.

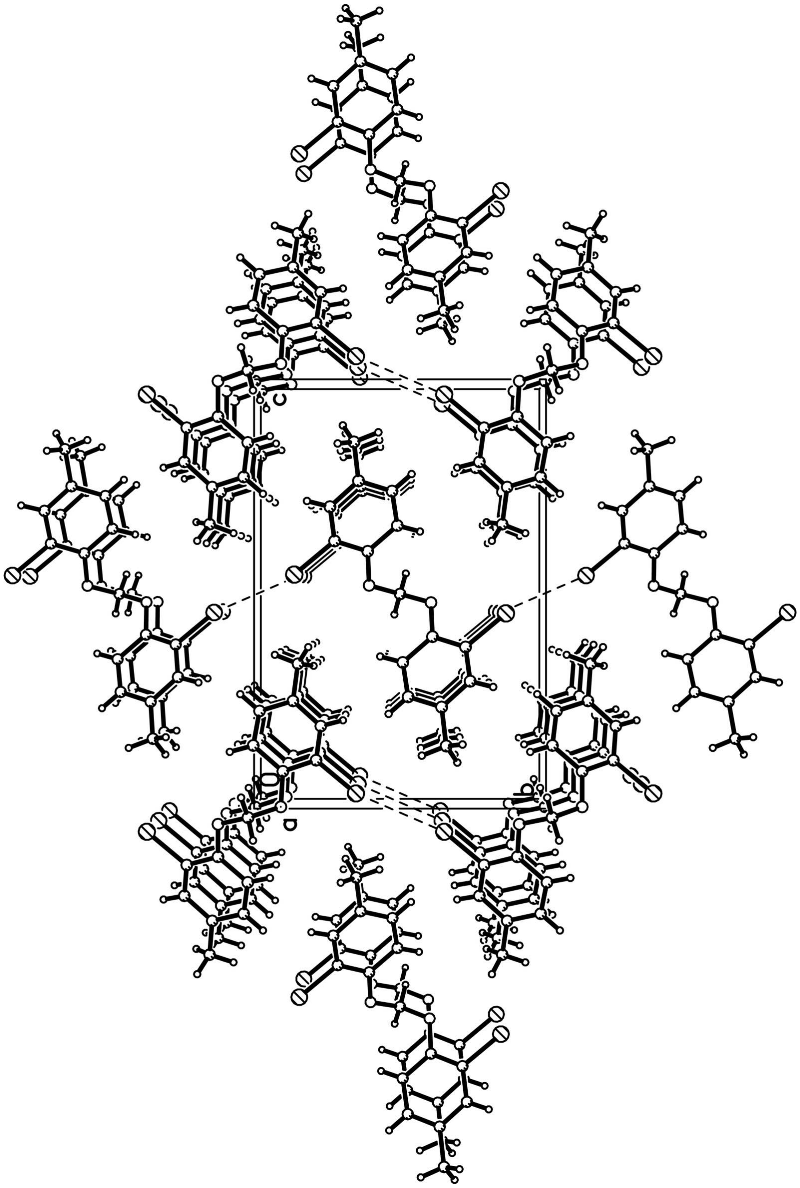

Figure 2

A view of the crystal packing along the *c* axis, with short Br···Br contacts indicated by dashed lines.

Bis(2-bromo-5-methylphenoxy)methane

Crystal data

$C_{15}H_{14}Br_2O_2$	$D_x = 1.733$ Mg m^{-3}
$M_r = 386.08$	Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å
Orthorhombic, $P2_12_12$	Cell parameters from 443 reflections
$a = 10.7752$ (11) Å	$\theta = 4.1$–$69.9°$
$b = 15.8690$ (17) Å	$\mu = 6.91$ mm^{-1}
$c = 4.3272$ (10) Å	$T = 291$ K
$V = 739.9$ (2) Å^3	Prism, colorless
$Z = 2$	0.35 × 0.30 × 0.30 mm
$F(000) = 380$	

Data collection

Agilent Xcalibur Eos Gemini diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 16.2312 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2011)
$T_{\min} = 0.196$, $T_{\max} = 0.231$
1505 measured reflections
1022 independent reflections
754 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.044$
$\theta_{\max} = 66.9°$, $\theta_{\min} = 5.0°$
$h = -12 \rightarrow 9$
$k = -18 \rightarrow 14$
$l = -3 \rightarrow 4$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.123$
$S = 1.02$
1022 reflections
88 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
$w = 1/[\sigma^2(F_o^2) + (0.040P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.36$ e Å^{-3}
$\Delta\rho_{\min} = -0.41$ e Å^{-3}
Absolute structure: Flack (1983), 212 Friedel pairs
Absolute structure parameter: −0.11 (9)

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}	Occ. (<1)
Br1	0.85154 (9)	0.95619 (6)	0.6233 (4)	0.0946 (6)	
O1	0.6042 (5)	0.9803 (3)	0.3521 (17)	0.0668 (18)	
C1	0.7175 (7)	0.8802 (4)	0.624 (3)	0.054 (2)	
C2	0.6083 (7)	0.9029 (5)	0.489 (2)	0.056 (3)	
C3	0.5091 (8)	0.8444 (5)	0.500 (2)	0.066 (3)	
H3	0.4330	0.8573	0.4098	0.079*	
C4	0.5267 (8)	0.7679 (5)	0.646 (3)	0.070 (3)	
H4	0.4617	0.7294	0.6501	0.084*	
C5	0.6369 (8)	0.7465 (4)	0.787 (2)	0.060 (3)	
C6	0.7312 (8)	0.8044 (5)	0.779 (2)	0.058 (3)	
H6	0.8056	0.7925	0.8792	0.069*	
C7	0.6509 (9)	0.6621 (5)	0.946 (2)	0.090 (3)	
H7A	0.7023	0.6684	1.1261	0.135*	
H7B	0.6886	0.6226	0.8070	0.135*	

H7C	0.5706	0.6417	1.0072	0.135*	
C8	0.5000	1.0000	0.168 (4)	0.080 (5)	
H8A	0.4805	0.9524	0.0361	0.096*	0.50
H8B	0.5195	1.0476	0.0361	0.096*	0.50

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0546 (6)	0.0668 (6)	0.1623 (14)	−0.0129 (5)	−0.0131 (8)	0.0130 (8)
O1	0.049 (3)	0.062 (3)	0.089 (5)	0.017 (3)	0.004 (4)	0.012 (4)
C1	0.052 (4)	0.042 (4)	0.066 (7)	0.009 (3)	0.001 (5)	0.004 (5)
C2	0.048 (4)	0.056 (5)	0.062 (8)	0.020 (4)	0.010 (5)	−0.001 (5)
C3	0.043 (4)	0.081 (6)	0.074 (8)	0.006 (5)	−0.009 (5)	−0.012 (6)
C4	0.058 (5)	0.061 (5)	0.090 (9)	−0.011 (4)	0.009 (7)	−0.008 (7)
C5	0.064 (5)	0.041 (4)	0.074 (8)	−0.006 (4)	0.020 (5)	−0.001 (5)
C6	0.058 (5)	0.057 (5)	0.058 (7)	0.008 (4)	0.007 (5)	0.002 (5)
C7	0.110 (8)	0.062 (5)	0.098 (9)	−0.002 (6)	0.019 (9)	0.026 (6)
C8	0.082 (10)	0.077 (9)	0.081 (12)	0.023 (8)	0.000	0.000

Geometric parameters (Å, º)

Br1—C1	1.882 (7)	C5—C6	1.370 (10)
O1—C2	1.364 (9)	C5—C7	1.515 (10)
O1—C8	1.412 (10)	C6—H6	0.9300
C1—C2	1.363 (11)	C7—H7A	0.9600
C1—C6	1.385 (10)	C7—H7B	0.9600
C2—C3	1.417 (11)	C7—H7C	0.9600
C3—C4	1.383 (11)	C8—O1^{i}	1.412 (10)
C3—H3	0.9300	C8—H8A	0.9700
C4—C5	1.376 (12)	C8—H8B	0.9700
C4—H4	0.9300		

C2—O1—C8	118.0 (6)	C5—C6—C1	121.0 (8)
C2—C1—C6	122.1 (7)	C5—C6—H6	119.5
C2—C1—Br1	119.4 (6)	C1—C6—H6	119.5
C6—C1—Br1	118.4 (6)	C5—C7—H7A	109.5
C1—C2—O1	117.0 (7)	C5—C7—H7B	109.5
C1—C2—C3	117.6 (8)	H7A—C7—H7B	109.5
O1—C2—C3	125.5 (8)	C5—C7—H7C	109.5
C4—C3—C2	119.2 (8)	H7A—C7—H7C	109.5
C4—C3—H3	120.4	H7B—C7—H7C	109.5
C2—C3—H3	120.4	O1—C8—O1^{i}	111.2 (12)
C5—C4—C3	122.5 (8)	O1—C8—H8A	109.4
C5—C4—H4	118.7	O1^{i}—C8—H8A	109.4
C3—C4—H4	118.7	O1—C8—H8B	109.4
C6—C5—C4	117.6 (8)	O1^{i}—C8—H8B	109.4
C6—C5—C7	122.0 (9)	H8A—C8—H8B	108.0
C4—C5—C7	120.3 (8)		

C6—C1—C2—O1	177.7 (8)	C2—C3—C4—C5	1.0 (16)
Br1—C1—C2—O1	1.3 (13)	C3—C4—C5—C6	0.2 (16)
C6—C1—C2—C3	−2.4 (15)	C3—C4—C5—C7	179.6 (9)
Br1—C1—C2—C3	−178.8 (7)	C4—C5—C6—C1	−2.5 (15)
C8—O1—C2—C1	170.0 (9)	C7—C5—C6—C1	178.1 (8)
C8—O1—C2—C3	−9.8 (14)	C2—C1—C6—C5	3.7 (15)
C1—C2—C3—C4	0.1 (15)	Br1—C1—C6—C5	−179.8 (7)
O1—C2—C3—C4	179.9 (9)	C2—O1—C8—O1^{i}	76.7 (6)

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