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(3,6-Dibromo-*o*-phenylene)dimethanol

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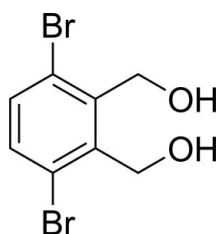
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.007$ Å; R factor = 0.043; wR factor = 0.111; data-to-parameter ratio = 20.0.

The title compound, $\text{C}_8\text{H}_8\text{Br}_2\text{O}_2$, was synthesized from the hydrolysis of 1,4-dibromo-2,3-bis(bromomethyl)benzene. One intramolecular $\text{O}-\text{H}\cdots\text{O}$ and two intramolecular $\text{C}-\text{H}\cdots\text{Br}$ interactions occur. In the crystal, molecules are linked into a chain running parallel to [010]. Adjacent chains are linked into a two-dimensional layer by a combination of intermolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds and $\text{C}-\text{H}\cdots\pi$ interactions.

Related literature

For the preparation of the title compound, see: Abad (2005); Lai & Yap, (1993).



Experimental

Crystal data

$\text{C}_8\text{H}_8\text{Br}_2\text{O}_2$
 $M_r = 295.96$
 Orthorhombic, $P2_12_12_1$
 $a = 4.482$ (2) Å
 $b = 9.123$ (5) Å
 $c = 22.819$ (11) Å

$V = 933.0$ (8) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 8.64$ mm⁻¹
 $T = 298$ K
 $0.20 \times 0.10 \times 0.10$ mm

Data collection

Bruker SMART APEX
 diffractometer
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 2008)
 $T_{\min} = 0.277$, $T_{\max} = 0.479$

11471 measured reflections
 2300 independent reflections
 1795 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.040$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.111$
 $S = 1.05$
 2300 reflections
 115 parameters
 2 restraints

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 1.17$ e Å⁻³
 $\Delta\rho_{\min} = -0.60$ e Å⁻³
 Absolute structure: Flack (1983),
 918 Friedel pairs
 Flack parameter: 0.03 (2)

Table 1

Hydrogen-bond geometry (Å, °).

Cg1 is the centroid of the C1–C6 ring.

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C8}-\text{H8A}\cdots\text{Br2}$	0.97	2.61	3.192 (6)	119
$\text{C7}-\text{H7B}\cdots\text{Br1}$	0.97	2.66	3.169 (5)	114
$\text{O2}-\text{H2}\cdots\text{O1}^{\text{i}}$	0.82 (6)	1.92 (3)	2.703 (6)	160 (7)
$\text{O1}-\text{H1}\cdots\text{O2}$	0.81 (6)	1.95 (4)	2.703 (6)	152 (7)
$\text{C8}-\text{H8B}\cdots\text{Cg1}^{\text{ii}}$	0.97	3.01	3.63(5)	123

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

Data collection: SMART (Bruker, 1997); cell refinement: SAINT (Bruker, 1999); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXL97.

The authors are grateful to Xianggao Meng for the data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: OM2391).

References

- Abad, A. (2005). *Synthesis*, **19**, 3355–3361.
 Bruker (1997). SMART. Bruker AXS Inc., Madison, Wisconsin, USA.
 Bruker (1999). SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.
 Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
 Lai, Y.-H. & Yap, A. H.-T. (1993). *J. Chem. Soc. Perkin Trans. 2*, pp. 1373–1377.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

supporting information

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(3,6-Dibromo-*o*-phenylene)dimethanol

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S1. Comment

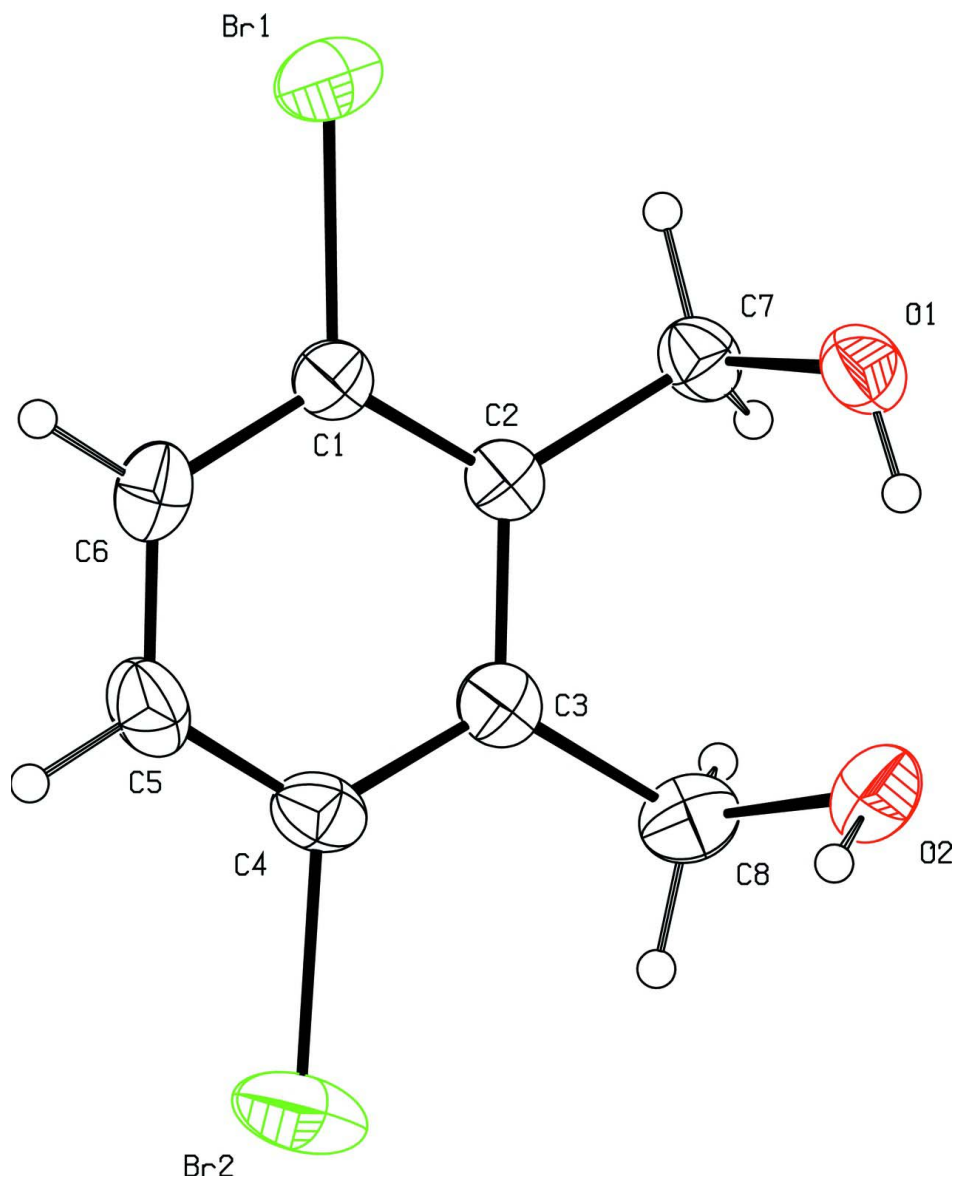
Benzyl alcohol derivatives are important compounds as preservatives, dyes, fibers, and nylons. In our work, the hydrolysis of 1,4-dibromo-2,3-bis(bromomethyl)benzene gave rise to the dimethanol product reported here. The molecules of (3,6-dibromo-1,2-phenylene)dimethanol (Figure 1) are linked into a one-dimensional chain running parallel to the [010] direction by intra- and intermolecular O—H \cdots O hydrogen bond interactions. The adjacent chains are linked into a two-dimensional layer by the combination of the O2—H2 \cdots O1(1 - *x*, *y* - 1/2, 1/2 - *z*) (Table 1) hydrogen bonds and C—H \cdots π interaction (H8B (1+*x*,*y*,*z*) \cdots Cg1 = 3.01 (1) Å, C8—H8B \cdots Cg1 = 122.9°, Cg1 is the centroid defined by atoms C1—C6.)

S2. Experimental

The title compound was synthesized according to the reported literature (Abad *et al.*, 2005 and Lai *et al.*, 1993). The reaction of 1,4-dibromo-2,3-dimethylbenzene with N-bromosuccinimide in carbon tetrachloride in the presence of benzoyl peroxide under illumination and reflux gave 1,4-dibromo-2,3-bis(bromomethyl)benzene. Then 1,4-dibromo-2,3-bis(bromomethyl)benzene with NaOH in water at room temperature gave rise to (3,6-dibromo-1,2-phenylene)dimethanol. Crystals suitable for X-ray diffraction were grown by slow evaporation of a chloroform-methanol (5:1) solution of the title compound at room temperature.

S3. Refinement

All carbon H atoms were placed at their idealized positions with C—H = 0.93 Å (aromatic), 0.97 Å (methylene)) and $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$ (aromatic and methylene). Hydrogen atoms bonded oxygen atoms were located from the Fourier difference maps and refined with the restraints of O—H = 0.82 (1) Å and $U_{\text{iso}}(\text{H}) = 1.5 U_{\text{eq}}(\text{O})$.

**Figure 1**

A view of the title compound, showing the atom labelling scheme, with displacement ellipsoids drawn at the 30% probability level. H atoms are omitted for clarity.

(3,6-Dibromo-*o*-phenylene)dimethanol

Crystal data

$C_8H_8Br_2O_2$

$M_r = 295.96$

Orthorhombic, $P2_12_12_1$

Hall symbol: P 2ac 2ab

$a = 4.482(2) \text{ \AA}$

$b = 9.123(5) \text{ \AA}$

$c = 22.819(11) \text{ \AA}$

$V = 933.0(8) \text{ \AA}^3$

$Z = 4$

$F(000) = 568$

$D_x = 2.107 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 3167 reflections

$\theta = 2.4\text{--}24.1^\circ$

$\mu = 8.64 \text{ mm}^{-1}$

$T = 298 \text{ K}$

Needle, colorless

$0.20 \times 0.10 \times 0.10 \text{ mm}$

Data collection

Bruker SMART APEX diffractometer	11471 measured reflections
Radiation source: fine-focus sealed tube	2300 independent reflections
Graphite monochromator	1795 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{\text{int}} = 0.040$
Absorption correction: multi-scan (SADABS; Sheldrick, 2008)	$\theta_{\text{max}} = 28.3^\circ$, $\theta_{\text{min}} = 2.4^\circ$
$T_{\text{min}} = 0.277$, $T_{\text{max}} = 0.479$	$h = -5 \rightarrow 5$
	$k = -12 \rightarrow 12$
	$l = -30 \rightarrow 30$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.043$	$w = 1/[\sigma^2(F_o^2) + (0.0525P)^2 + 0.6273P]$
$wR(F^2) = 0.111$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{\text{max}} = 0.004$
2300 reflections	$\Delta\rho_{\text{max}} = 1.17 \text{ e } \text{\AA}^{-3}$
115 parameters	$\Delta\rho_{\text{min}} = -0.60 \text{ e } \text{\AA}^{-3}$
2 restraints	Absolute structure: Flack (1983), 918 Friedel pairs
Primary atom site location: structure-invariant direct methods	Absolute structure parameter: 0.03 (2)
Secondary atom site location: difference Fourier map	

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.30061 (15)	0.81127 (6)	0.06905 (3)	0.0657 (2)
Br2	0.8120 (2)	0.15549 (7)	0.12029 (4)	0.0930 (3)
C1	0.4665 (11)	0.6246 (5)	0.0868 (2)	0.0422 (11)
C2	0.6474 (10)	0.6045 (5)	0.13420 (19)	0.0380 (10)
C3	0.7599 (11)	0.4622 (5)	0.14561 (19)	0.0443 (11)
C4	0.6766 (13)	0.3496 (5)	0.1078 (2)	0.0518 (12)
C5	0.4952 (14)	0.3730 (7)	0.0605 (2)	0.0593 (14)
H5	0.4439	0.2955	0.0360	0.071*
C6	0.3902 (13)	0.5100 (7)	0.0495 (2)	0.0532 (13)
H6	0.2683	0.5270	0.0172	0.064*
C7	0.7251 (12)	0.7281 (5)	0.17504 (19)	0.0466 (11)
H7A	0.9324	0.7197	0.1868	0.056*
H7B	0.7003	0.8209	0.1549	0.056*
C8	0.9546 (13)	0.4374 (7)	0.1983 (2)	0.0566 (13)
H8A	1.0251	0.3369	0.1982	0.068*

H8B	1.1273	0.5013	0.1960	0.068*
O1	0.5374 (9)	0.7250 (4)	0.22612 (15)	0.0555 (9)
H1	0.582 (16)	0.651 (5)	0.244 (3)	0.083*
O2	0.7969 (9)	0.4656 (4)	0.25204 (16)	0.0610 (9)
H2	0.680 (14)	0.397 (6)	0.250 (3)	0.091*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0672 (3)	0.0603 (3)	0.0697 (3)	0.0117 (3)	0.0053 (3)	0.0190 (3)
Br2	0.1119 (6)	0.0401 (3)	0.1269 (6)	0.0091 (4)	0.0183 (5)	0.0023 (3)
C1	0.042 (3)	0.042 (3)	0.043 (2)	-0.004 (2)	0.006 (2)	0.0020 (19)
C2	0.031 (2)	0.040 (2)	0.042 (2)	-0.0079 (18)	0.0103 (19)	-0.0006 (18)
C3	0.041 (3)	0.046 (2)	0.046 (2)	-0.006 (2)	0.013 (2)	0.0019 (19)
C4	0.054 (3)	0.039 (2)	0.062 (3)	-0.005 (2)	0.010 (3)	0.002 (2)
C5	0.070 (4)	0.053 (3)	0.056 (3)	-0.014 (3)	-0.001 (3)	-0.015 (2)
C6	0.052 (3)	0.067 (3)	0.041 (2)	-0.010 (2)	-0.004 (2)	0.001 (2)
C7	0.047 (3)	0.043 (2)	0.050 (2)	-0.006 (2)	0.004 (2)	-0.0046 (19)
C8	0.045 (3)	0.060 (3)	0.065 (3)	0.002 (3)	0.000 (3)	0.009 (3)
O1	0.066 (2)	0.049 (2)	0.052 (2)	-0.0007 (19)	0.0113 (19)	-0.0068 (16)
O2	0.062 (2)	0.069 (2)	0.0515 (18)	-0.007 (2)	-0.004 (2)	0.0116 (18)

Geometric parameters (Å, °)

Br1—C1	1.902 (5)	C5—H5	0.9300
Br2—C4	1.894 (5)	C6—H6	0.9300
C1—C2	1.365 (6)	C7—O1	1.438 (5)
C1—C6	1.391 (7)	C7—H7A	0.9700
C2—C3	1.417 (7)	C7—H7B	0.9700
C2—C7	1.504 (6)	C8—O2	1.439 (7)
C3—C4	1.392 (7)	C8—H8A	0.9700
C3—C8	1.502 (7)	C8—H8B	0.9700
C4—C5	1.368 (8)	O1—H1	0.81 (6)
C5—C6	1.359 (9)	O2—H2	0.82 (6)
C2—C1—C6	122.0 (5)	C5—C6—H6	120.2
C2—C1—Br1	121.4 (4)	C1—C6—H6	120.2
C6—C1—Br1	116.5 (4)	O1—C7—C2	110.6 (4)
C1—C2—C3	118.7 (4)	O1—C7—H7A	109.5
C1—C2—C7	121.9 (4)	C2—C7—H7A	109.5
C3—C2—C7	119.4 (4)	O1—C7—H7B	109.5
C4—C3—C2	117.8 (4)	C2—C7—H7B	109.5
C4—C3—C8	122.7 (4)	H7A—C7—H7B	108.1
C2—C3—C8	119.4 (4)	O2—C8—C3	111.7 (4)
C5—C4—C3	122.3 (5)	O2—C8—H8A	109.3
C5—C4—Br2	117.0 (4)	C3—C8—H8A	109.3
C3—C4—Br2	120.7 (4)	O2—C8—H8B	109.3
C6—C5—C4	119.7 (5)	C3—C8—H8B	109.3

C6—C5—H5	120.2	H8A—C8—H8B	107.9
C4—C5—H5	120.2	C7—O1—H1	107 (5)
C5—C6—C1	119.5 (5)	C8—O2—H2	98 (5)
C6—C1—C2—C3	-0.1 (7)	C8—C3—C4—Br2	-1.0 (6)
Br1—C1—C2—C3	178.9 (3)	C3—C4—C5—C6	-0.1 (8)
C6—C1—C2—C7	-178.9 (4)	Br2—C4—C5—C6	179.8 (4)
Br1—C1—C2—C7	0.1 (6)	C4—C5—C6—C1	-0.6 (8)
C1—C2—C3—C4	-0.6 (6)	C2—C1—C6—C5	0.7 (8)
C7—C2—C3—C4	178.2 (4)	Br1—C1—C6—C5	-178.4 (4)
C1—C2—C3—C8	-178.8 (4)	C1—C2—C7—O1	98.1 (5)
C7—C2—C3—C8	0.0 (6)	C3—C2—C7—O1	-80.7 (5)
C2—C3—C4—C5	0.7 (7)	C4—C3—C8—O2	-114.8 (5)
C8—C3—C4—C5	178.9 (5)	C2—C3—C8—O2	63.3 (6)
C2—C3—C4—Br2	-179.2 (3)		

Hydrogen-bond geometry (Å, °)

Cg1 is the centroid of the C1—C6 ring.

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C8—H8A...Br2	0.97	2.61	3.192 (6)	119
C7—H7B...Br1	0.97	2.66	3.169 (5)	114
O2—H2...O1 ⁱ	0.82 (6)	1.92 (3)	2.703 (6)	160 (7)
O1—H1...O2	0.81 (6)	1.95 (4)	2.703 (6)	152 (7)
C8—H8B...Cg1 ⁱⁱ	0.97	3.01	3.63 (5)	123

Symmetry codes: (i) $-x+1, y-1/2, -z+1/2$; (ii) $x+1, y, z$.