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2,5-Dibromoindan-1-ol

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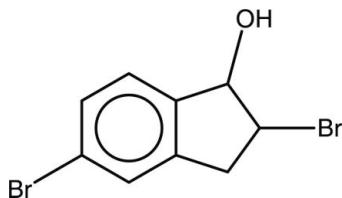
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Key indicators: single-crystal X-ray study; $T = 299$ K; mean $\sigma(\text{C}-\text{C}) = 0.020$ Å; R factor = 0.082; wR factor = 0.228; data-to-parameter ratio = 22.5.

In the title compound, $\text{C}_9\text{H}_8\text{Br}_2\text{O}$, the cyclopentene ring adopts an envelope conformation with the brominated C atom as the flap. In the crystal, molecules are linked by strong $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds into zigzag $C(4)$ chains along [010]. In addition, a $\text{C}-\text{H}\cdots\pi$ interaction involving the benzene ring and the H atom attached to the hydroxylated C atom is observed.

Related literature

For bromination of hydrocarbons, see: Cakmak *et al.* (2006); Erenler & Cakmak (2004); Erenler *et al.* (2006). For the pharmacological and medicinal properties of indanes, see: Mitrochkine *et al.* (1995); Catto *et al.* (2010); Wu (2006); McClure *et al.* (2011) and for their use in natural product chemistry, see: Snyder & Brill (2011). For a similar structure, see: Çelik *et al.* (2012). For puckering parameters, see: Cremer & Pople (1975). For hydrogen-bond motifs, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$\text{C}_9\text{H}_8\text{Br}_2\text{O}$ $a = 9.5137$ (10) Å
 $M_r = 291.95$ $b = 4.8991$ (7) Å
 Monoclinic, $P2_1/c$ $c = 20.249$ (3) Å

$\beta = 94.165$ (10)°
 $V = 941.3$ (2) Å³
 $Z = 4$
 Cu $K\alpha$ radiation

$\mu = 10.50$ mm⁻¹
 $T = 299$ K
 $0.17 \times 0.01 \times 0.01$ mm

Data collection

Agilent Xcalibur Ruby Gemini diffractometer 1777 measured reflections
 Absorption correction: refined from 1777 independent reflections
 ΔF (XABS2; Parkin *et al.*, 1995) 733 reflections with $I > 2\sigma(I)$
 $T_{\min} = 0.882$, $T_{\max} = 0.900$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.082$ 79 parameters
 $wR(F^2) = 0.228$ H-atom parameters constrained
 $S = 1.00$ $\Delta\rho_{\max} = 0.68$ e Å⁻³
 1777 reflections $\Delta\rho_{\min} = -0.78$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

 $\text{Cg}2$ is the centroid of the C1–C6 benzene ring.

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O}1-\text{H}1\cdots\text{O}1^i$	0.82	1.91	2.713 (14)	165
$\text{C}9-\text{H}9\cdots\text{Cg}2^{ii}$	0.98	2.67	3.629 (16)	166

Symmetry codes: (i) $-x + 2, y - \frac{1}{2}, -z + \frac{3}{2}$; (ii) $x, y - 1, z$.

Data collection: *CrysAlis PRO* (Agilent, 2011); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1999); software used to prepare material for publication: *WinGX* (Farrugia, 1997) and *PLATON* (Spek, 2009).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PK2441).

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supplementary materials

Acta Cryst. (2012). E68, o2795–o2796 [doi:10.1107/S1600536812035829]

2,5-Dibromoindan-1-ol

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Comment

Bromination of hydrocarbons are important processes in synthetic chemistry (Cakmak *et al.*, 2006; Erenler *et al.*, 2006; Erenler & Cakmak, 2004). Indanes are an important class of molecules due to their pharmacological and medicinal properties (Mitrochkine *et al.*, 1995; Catto *et al.*, 2010; Wu, 2006; McClure *et al.*, 2011) as well as in natural product chemistry (Snyder & Brill, 2011).

In the title compound (I), (Fig. 1), the five-membered C1/C6–C9 cyclopentene ring exhibits an envelope conformation with C8 at the tip of the envelope [the puckering parameters (Cremer & Pople, 1975) are $Q(2) = 0.289(17)$ Å and $\varphi(2) = 290(3)^\circ$]. All bond lengths and bond angles in (I) are in the normal range and are in good agreement with those reported in a similar structure (Çelik *et al.*, 2012).

In the crystal, pairs of strong O—H \cdots O hydrogen bonds connect the molecules, forming zigzag C(4) chains propagating along the *b* axis (Bernstein *et al.*, 1995; Table 1, Fig. 2). In addition, a C—H \cdots π interaction with the benzene ring is also found (Table 1).

Experimental

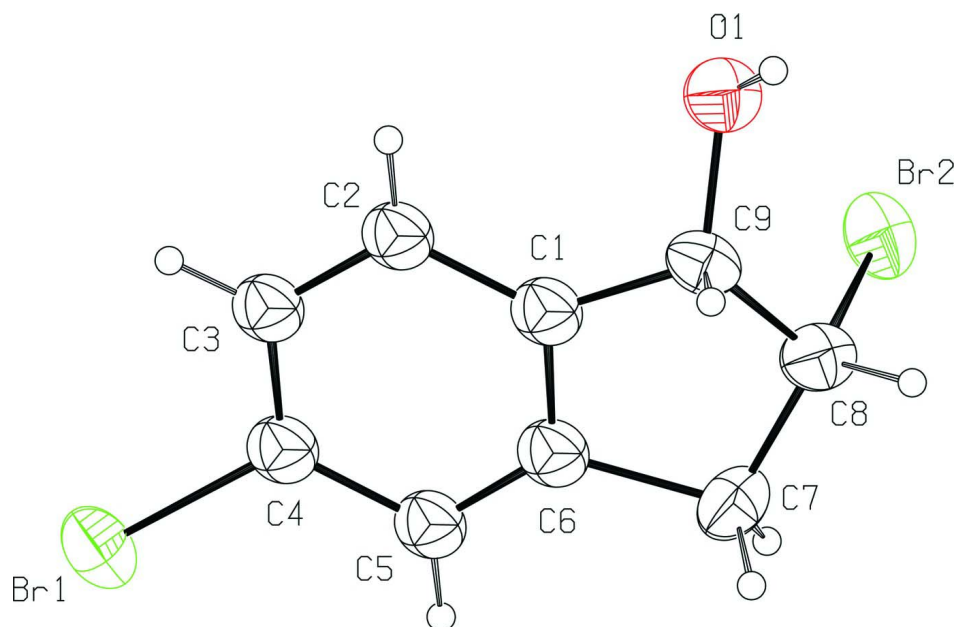
To a stirred solution of tribromide (1.0 g, 2.8 mmol) in THF (10 ml) was added a solution of AgClO₄·H₂O (0.82 g, 3.64 mmol) in aqueous THF (5 ml THF / 2 ml H₂O). The resulting mixture was stirred at room temperature for 6 h. The precipitated AgBr was removed by filtration and then the solution was dried over calcium chloride. After removal of the solvent, the residue was purified by silica gel column chromatography. Elution with hexane/ethyl acetate (4:1) afforded the 2,5-dibromo-1-hydroxyindane (0.59 g, 72%).

Refinement

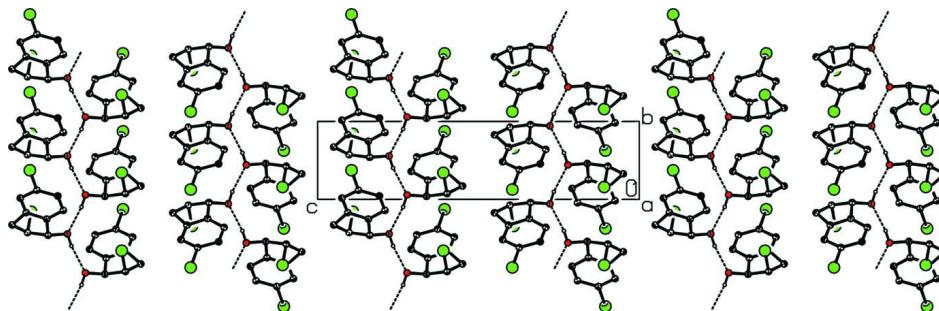
H-atoms were positioned geometrically and refined using a riding model with O—H = 0.82 Å, C—H = 0.93–0.98 Å, and with $U_{\text{iso}}(\text{H}) = 1.2$ or $1.5U_{\text{eq}}(\text{C}, \text{O})$.

Computing details

Data collection: *CrysAlis PRO* (Agilent, 2011); cell refinement: *CrysAlis PRO* (Agilent, 2011); data reduction: *CrysAlis PRO* (Agilent, 2011); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1999); software used to prepare material for publication: *WinGX* (Farrugia, 1997) and *PLATON* (Spek, 2009).


Figure 1

An ORTEP plot of (I) with the atom numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 30% probability level.


Figure 2

View of the packing and hydrogen bonding of (I), along the *a* axis. H atoms not involved in hydrogen bonding are omitted for the sake of clarity.

2,5-Dibromoindan-1-ol

Crystal data

$C_9H_8Br_2O$

$M_r = 291.95$

Monoclinic, $P2_1/c$

Hall symbol: $-P 2_1/c$

$a = 9.5137 (10) \text{ \AA}$

$b = 4.8991 (7) \text{ \AA}$

$c = 20.249 (3) \text{ \AA}$

$\beta = 94.165 (10)^\circ$

$V = 941.3 (2) \text{ \AA}^3$

$Z = 4$

$F(000) = 560$

$D_x = 2.060 \text{ Mg m}^{-3}$

Cu $K\alpha$ radiation, $\lambda = 1.5418 \text{ \AA}$

Cell parameters from 378 reflections

$\theta = 4.4\text{--}70.4^\circ$

$\mu = 10.50 \text{ mm}^{-1}$

$T = 299 \text{ K}$

Prism, colourless

$0.17 \times 0.01 \times 0.01 \text{ mm}$

Data collection

Agilent Xcalibur Ruby Gemini diffractometer	$T_{\min} = 0.882$, $T_{\max} = 0.900$
Radiation source: Enhance (Cu) X-ray Source	1777 measured reflections
Graphite monochromator	1777 independent reflections
Detector resolution: 10.2673 pixels mm ⁻¹	733 reflections with $I > 2\sigma(I)$
ω scans	$R_{\text{int}} = 0.000$
Absorption correction: part of the refinement model (ΔF)	$\theta_{\max} = 70.6^\circ$, $\theta_{\min} = 4.4^\circ$
(XABS2; Parkin <i>et al.</i> , 1995)	$h = -11 \rightarrow 11$
	$k = 0 \rightarrow 5$
	$l = 0 \rightarrow 24$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.082$	H-atom parameters constrained
$wR(F^2) = 0.228$	$w = 1/[\sigma^2(F_o^2) + (0.0341P)^2]$
$S = 1.00$	where $P = (F_o^2 + 2F_c^2)/3$
1777 reflections	$(\Delta/\sigma)_{\max} < 0.001$
79 parameters	$\Delta\rho_{\max} = 0.68 \text{ e } \text{\AA}^{-3}$
0 restraints	$\Delta\rho_{\min} = -0.78 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	

Special details

Experimental. Absorption correction: XABS2 (Parkin *et al.*, 1995); Quadratic fit to $\sin(\theta)/\lambda$ - 18 parameters

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 e.s.d.'s 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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.4208 (2)	0.8732 (4)	0.60524 (11)	0.0799 (7)
Br2	1.11644 (19)	0.3412 (4)	0.60918 (10)	0.0766 (7)
O1	0.9808 (12)	0.057 (2)	0.7220 (5)	0.069 (4)
C1	0.7790 (16)	0.247 (3)	0.6578 (8)	0.062 (2)
C2	0.7029 (16)	0.359 (3)	0.7090 (8)	0.062 (2)
C3	0.5983 (16)	0.544 (3)	0.6919 (8)	0.062 (2)
C4	0.5687 (16)	0.618 (3)	0.6270 (7)	0.062 (2)
C5	0.6461 (16)	0.517 (3)	0.5754 (8)	0.062 (2)
C6	0.7444 (16)	0.331 (3)	0.5936 (8)	0.062 (2)
C7	0.8485 (19)	0.172 (3)	0.5499 (7)	0.067 (6)
C8	0.9613 (18)	0.074 (3)	0.5978 (7)	0.063 (6)
C9	0.8931 (16)	0.040 (3)	0.6617 (7)	0.057 (5)
H1	1.00390	-0.09700	0.73430	0.1030*
H2	0.72330	0.30880	0.75290	0.0740*
H3	0.54710	0.62080	0.72470	0.0740*

H5	0.62990	0.57520	0.53190	0.0740*
H7A	0.80100	0.02070	0.52700	0.0810*
H7B	0.88630	0.29260	0.51750	0.0810*
H8	0.99730	-0.10210	0.58330	0.0750*
H9	0.84820	-0.14010	0.66060	0.0680*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0591 (11)	0.0778 (13)	0.1011 (14)	0.0087 (10)	-0.0058 (10)	0.0030 (11)
Br2	0.0611 (11)	0.0755 (12)	0.0942 (13)	0.0033 (9)	0.0123 (9)	0.0108 (10)
O1	0.081 (8)	0.052 (6)	0.072 (7)	0.005 (6)	-0.002 (6)	0.004 (5)
C1	0.052 (4)	0.069 (4)	0.064 (4)	-0.007 (3)	0.001 (3)	-0.001 (3)
C2	0.052 (4)	0.069 (4)	0.064 (4)	-0.007 (3)	0.001 (3)	-0.001 (3)
C3	0.052 (4)	0.069 (4)	0.064 (4)	-0.007 (3)	0.001 (3)	-0.001 (3)
C4	0.052 (4)	0.069 (4)	0.064 (4)	-0.007 (3)	0.001 (3)	-0.001 (3)
C5	0.052 (4)	0.069 (4)	0.064 (4)	-0.007 (3)	0.001 (3)	-0.001 (3)
C6	0.052 (4)	0.069 (4)	0.064 (4)	-0.007 (3)	0.001 (3)	-0.001 (3)
C7	0.089 (12)	0.058 (9)	0.056 (8)	0.007 (9)	0.013 (8)	-0.005 (8)
C8	0.083 (12)	0.046 (8)	0.058 (9)	0.008 (8)	-0.005 (8)	0.004 (7)
C9	0.059 (9)	0.048 (8)	0.061 (9)	-0.003 (7)	-0.008 (8)	0.010 (7)

Geometric parameters (\AA , $^\circ$)

Br1—C4	1.910 (15)	C6—C7	1.58 (2)
Br2—C8	1.974 (16)	C7—C8	1.47 (2)
O1—C9	1.430 (18)	C8—C9	1.50 (2)
O1—H1	0.8200	C2—H2	0.9300
C1—C6	1.38 (2)	C3—H3	0.9300
C1—C9	1.48 (2)	C5—H5	0.9300
C1—C2	1.42 (2)	C7—H7A	0.9700
C2—C3	1.37 (2)	C7—H7B	0.9700
C3—C4	1.37 (2)	C8—H8	0.9800
C4—C5	1.41 (2)	C9—H9	0.9800
C5—C6	1.34 (2)		
C9—O1—H1	109.00	O1—C9—C1	112.6 (12)
C2—C1—C6	118.3 (14)	C1—C2—H2	121.00
C6—C1—C9	112.2 (13)	C3—C2—H2	121.00
C2—C1—C9	129.5 (14)	C2—C3—H3	120.00
C1—C2—C3	118.1 (15)	C4—C3—H3	120.00
C2—C3—C4	120.7 (15)	C4—C5—H5	122.00
Br1—C4—C5	118.3 (11)	C6—C5—H5	122.00
C3—C4—C5	122.3 (14)	C6—C7—H7A	111.00
Br1—C4—C3	119.4 (11)	C6—C7—H7B	111.00
C4—C5—C6	115.3 (15)	C8—C7—H7A	111.00
C1—C6—C7	105.3 (12)	C8—C7—H7B	111.00
C5—C6—C7	129.4 (14)	H7A—C7—H7B	109.00
C1—C6—C5	125.2 (15)	Br2—C8—H8	110.00
C6—C7—C8	104.4 (12)	C7—C8—H8	110.00

Br2—C8—C9	109.9 (10)	C9—C8—H8	110.00
C7—C8—C9	105.3 (13)	O1—C9—H9	107.00
Br2—C8—C7	111.3 (10)	C1—C9—H9	107.00
O1—C9—C8	117.9 (13)	C8—C9—H9	107.00
C1—C9—C8	103.8 (12)		
C6—C1—C2—C3	0 (2)	Br1—C4—C5—C6	-177.5 (11)
C9—C1—C2—C3	-178.1 (15)	C3—C4—C5—C6	4 (2)
C2—C1—C6—C5	2 (2)	C4—C5—C6—C1	-4 (2)
C2—C1—C6—C7	179.4 (13)	C4—C5—C6—C7	179.1 (14)
C9—C1—C6—C5	-179.5 (15)	C1—C6—C7—C8	-16.7 (16)
C9—C1—C6—C7	-1.9 (17)	C5—C6—C7—C8	160.8 (16)
C2—C1—C9—O1	-33 (2)	C6—C7—C8—Br2	-90.7 (12)
C2—C1—C9—C8	-162.1 (15)	C6—C7—C8—C9	28.3 (15)
C6—C1—C9—O1	148.1 (13)	Br2—C8—C9—O1	-34.8 (16)
C6—C1—C9—C8	19.4 (17)	Br2—C8—C9—C1	90.6 (12)
C1—C2—C3—C4	0 (2)	C7—C8—C9—O1	-154.8 (12)
C2—C3—C4—Br1	179.3 (12)	C7—C8—C9—C1	-29.4 (15)
C2—C3—C4—C5	-2 (2)		

Hydrogen-bond geometry (\AA , $^\circ$)

Cg2 is the centroid of the C1–C6 benzene ring.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1 \cdots O1 ⁱ	0.82	1.91	2.713 (14)	165
C9—H9 \cdots Cg2 ⁱⁱ	0.98	2.67	3.629 (16)	166

Symmetry codes: (i) $-x+2, y-1/2, -z+3/2$; (ii) $x, y-1, z$.

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