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trans,trans,trans-2,3,5,8-Tetrabromo-1,4-dihydroxy-1,2,3,4-tetrahydronaphthalene

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
R factor = 0.042
wR factor = 0.082
Data-to-parameter ratio = 21.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The structure of the title compound, $C_{10}H_8Br_4O_2$, has been determined at room temperature. The ten-membered tetrahydronaphthalene ring system is nearly planar, with a total puckering amplitude Q_T of 0.386 (5) Å. The crystal structure is stabilized by short inter- and intramolecular hydrogen-bond contacts.

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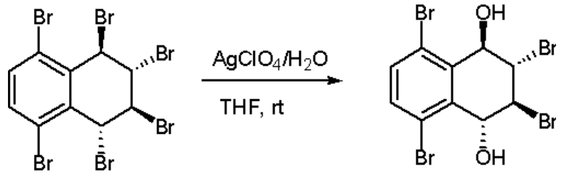
Comment

Naphthalenes, playing an important role as structural units or key intermediates of naturally occurring alkaloids, attract the interest of synthetic and natural product chemists for their biological activities (Huang & Wang, 2001). Halogenated aromatic compounds are an important class of molecules in synthetic organic chemistry. They are key intermediates in the preparation of organometallic reagents (Wakefield, 1976; Davis, 1982; Cannon & Krow, 1996) and play vital roles in transition-metal-mediated coupling reactions (Still, 1985; Miyaura & Suzuki, 1995; Beletskaya & Cheprakov, 2000; Cabri & Candiani, 1995; Meijere & Meyer, 1994; Sonogashira, 1991). Numerous industrially valuable products such as pesticides, insecticides, herbicides, pharmaceutically and medicinally active compounds, fire retardants, and other new materials carry halogen functionality. Hydroxy derivatives of naphthalene have importance for natural products (Kawazoe *et al.*, 1999), as well as for their pharmacological properties (Orsini *et al.*, 2002), and they are useful precursors of other valuable naphthalene derivatives (Ogata *et al.*, 2004). In this study, the crystal structure of the title compound, (II), was determined by X-ray analysis and the molecular structure and the numbering scheme are shown in Fig. 1. Selected geometric parameters are given in Table 1 and their values are normal.



(I)

(II)

The geometry of (II) compares well with similar structures reported in the literature (Allen *et al.*, 1987). The mean O—C and Br—C bond distances are 1.422 (6) and 1.938 (5) Å, respectively, with Br—C—C bond angles in the range 107.0 (3)–121.6 (4)° [average values for related compounds: 112.6 (7) (Hökelek *et al.*, 2001), 113.9 (7) (Hökelek *et al.*, 1998) and 115.1 (6)° (Hökelek *et al.*, 1991)]. The ten-membered tetrahydronaphthalene ring system of (II) has the

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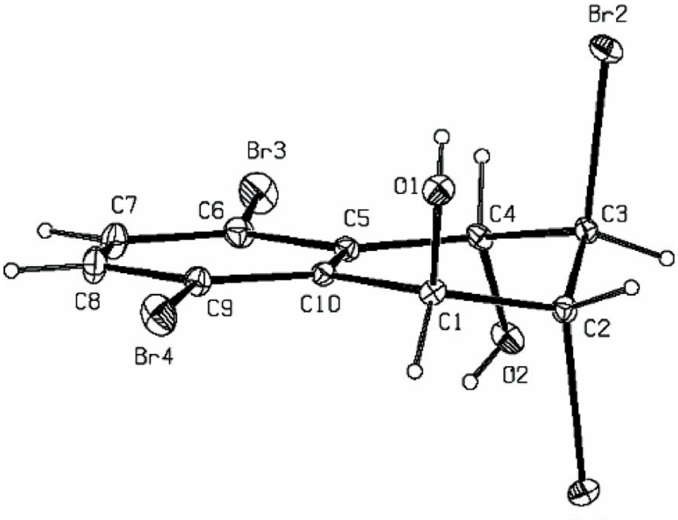

Figure 1
A view of the title compound with the atom-numbering scheme. Displacement ellipsoids are drawn at the 10% probability level and H atoms are shown as small spheres of arbitrary radii.

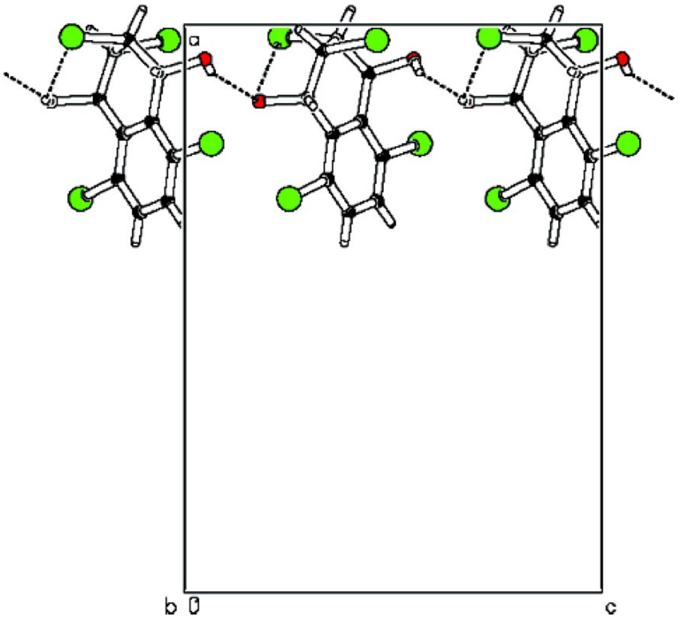

Figure 2
A view of part of the molecular packing and hydrogen bonding in the unit cell of (II).

hydroxy O, Br and H atoms in a *trans,trans,trans* configuration. The maximum deviations from the mean plane of the ring system are −0.058 (5), 0.263 (5) and −0.255 (5) Å for atoms C1, C2 and C3, respectively. The total puckering amplitude Q_T is 0.386 (5) Å (Cremer & Pople, 1975). The crystal structure is stabilized by inter- and intramolecular hydrogen-bonding contacts, involving both hydroxy groups, leading to the formation of chains along the *c* axis (Fig. 2 and Table 2).

Experimental

(1*R*,2*S*,3*S*,4*R*)-1,2,3,4,5,8-Hexabromo-1,2,3,4-tetrahydronaphthalene, (I), was treated with a solution of $AgCl_4 \cdot nH_2O$ (2.45 g, 10.9 mmol) in aqueous THF (7 ml THF/3 ml H_2O) (Erenler & Çakmak, 2004) to form the title diol (II) (m.p. 461–462 K). This was isolated by column chromatography and purified by crystallization from acetone–hexane.

Crystal data

$C_{10}H_8Br_4O_2$
$M_r = 479.76$
Orthorhombic, *Pbca*
$a = 16.8495$ (9) Å
$b = 12.1086$ (9) Å
$c = 12.3570$ (7) Å
$V = 2521.1$ (3) Å^3
$Z = 8$
$D_x = 2.528$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 3257 reflections
$\theta = 1.7$–28.9°
$\mu = 12.75$ mm^{-1}
$T = 293$ K
Plate, colorless
0.64 × 0.41 × 0.12 mm

Data collection

Stoe IPDS-II diffractometer
ω scans
Absorption correction: by integration (*X-RED*32; Stoe & Cie, 2002)
$T_{min} = 0.045$, $T_{max} = 0.310$
15995 measured reflections

3257 independent reflections
2283 reflections with $I > 2\sigma(I)$
$R_{int} = 0.089$
$\theta_{max} = 28.8°$
$h = -22 \rightarrow 22$
$k = -16 \rightarrow 15$
$l = -13 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.082$
$S = 1.02$
3257 reflections
153 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0198P)^2 + 5.027P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.91$ e Å^{-3}
$\Delta\rho_{min} = -0.73$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Br1—C2	1.978 (5)	Br4—C9	1.897 (5)
Br2—C3	1.982 (5)	O1—C1	1.429 (6)
Br3—C6	1.895 (5)	O2—C4	1.415 (6)
O1—C1—C2	109.4 (4)	O2—C4—C5	112.5 (4)
O1—C1—C10	111.8 (4)	O2—C4—C3	108.6 (4)
Br1—C2—C1	108.1 (3)	Br3—C6—C5	121.4 (4)
Br1—C2—C3	107.0 (3)	Br3—C6—C7	116.2 (4)
Br2—C3—C4	108.3 (3)	Br4—C9—C10	121.6 (4)
Br2—C3—C2	107.0 (3)	Br4—C9—C8	116.4 (4)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—HO1···Br2	0.62 (7)	2.81 (9)	3.232 (5)	130 (10)
O2—HO2···O1^i	0.64 (6)	2.25 (6)	2.840 (6)	155 (7)

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

The hydroxyl H atoms were found in a difference Fourier map and refined isotropically. Other H atoms were positioned geometrically, with C—H distances of 0.93 Å for aromatic and 0.98 Å for methylene H atoms, and allowed to ride with, $U_{iso}(H) = 1.2U_{eq}$(carrier atom).

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED*32 (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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