

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

1,6-Dibromonaphthalen-2-ol methanol monosolvate

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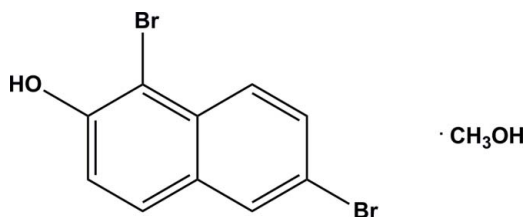
Received 6 May 2013; accepted 12 June 2013

 Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.036; wR factor = 0.074; data-to-parameter ratio = 19.5.

The naphthol-containing molecule of the title compound, $\text{C}_{10}\text{H}_6\text{Br}_2\text{O}\cdot\text{CH}_3\text{OH}$, crystallized as a methanol monosolvate and is planar to within 0.069 (1) Å for all non-H atoms. In the crystal, molecules are linked by two pairs of $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds, involving the methanol molecule, forming dimer-like arrangements. The crystal structure is further stabilized by $\pi-\pi$ stacking [centroid-centroid distance = 3.676 (2) Å] and $\text{Br}\cdots\text{Br}$ interactions [3.480 (4) and 3.786 (1) Å], forming a three-dimensional structure.

Related literature

For information on applications of 1,6-dibromo-2-naphthol, see: Costa *et al.* (2012); Takeuchi *et al.* (2000); Kalra & Kumar (2005). For related structures, see: Rozycka-Sokolowska & Marciniak (2009). For halogen-halogen interactions, see: Zordan & Brammer (2006); Schlueter *et al.* (2012); Desiraju & Parthasarathy (1989).



Experimental

Crystal data

$\text{C}_{10}\text{H}_6\text{Br}_2\text{O}\cdot\text{CH}_4\text{O}$
 $M_r = 334.01$
 Monoclinic, $P2_1/n$
 $a = 3.9971$ (4) Å
 $b = 12.4705$ (12) Å

$c = 22.462$ (2) Å
 $\beta = 92.442$ (1)°
 $V = 1118.62$ (19) Å³
 $Z = 4$
 Mo $K\alpha$ radiation

$\mu = 7.22$ mm⁻¹
 $T = 100$ K

0.26 × 0.11 × 0.01 mm

Data collection

Bruker APEXII CCD diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2011)
 $T_{\min} = 0.521$, $T_{\max} = 0.746$

12658 measured reflections
 2690 independent reflections
 2082 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.057$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.036$
 $wR(F^2) = 0.074$
 $S = 1.02$
 2690 reflections

138 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 1.31$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.60$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O1}-\text{H1}\cdots\text{O2}^{\text{i}}$	0.84	1.80	2.632 (4)	171
$\text{O2}-\text{H2}\cdots\text{O1}^{\text{ii}}$	0.84	2.01	2.809 (4)	159

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

Data collection: APEX2 (Bruker, 2011); cell refinement: SAINT (Bruker, 2011); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: CrystalMaker (CrystalMaker Software, 2009); software used to prepare material for publication: enCIFer (Allen *et al.*, 2004).

The authors gratefully acknowledge The College of New Jersey's School of Science for research funding and the National Science Foundation for major research instrumentation grant (NSF-0922931) for diffractometer acquisition.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SU2599).

References

- Allen, F. H., Johnson, O., Shields, G. P., Smith, B. R. & Towler, M. (2004). *J. Appl. Cryst.* **37**, 335–338.
- Bruker (2011). APEX2, SAINT and SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.
- Costa, B., Irvine, M., Fang, G., Eaves, R., Mayo-Martin, M., Laube, B., Jane, D. & Monaghan, D. (2012). *Neuropharmacology*, **62**, 1730–1736.
- CrystalMaker Software (2009). *CrystalMaker for Windows*. CrystalMaker Software Ltd, Oxford, England.
- Desiraju, G. & Parthasarathy, R. (1989). *J. Am. Chem. Soc.* **111**, 8725–8726.
- Kalra, B. & Kumar, A. (2005). US Patent Appl. Publ. 14.
- Rozycka-Sokolowska, E. & Marciniak, B. (2009). *Acta Cryst.* **C65**, o207–o210.
- Schlueter, J., Park, H., Halder, G., Armand, W., Dunmars, C., Chapman, K., Manson, J., Singleton, J., McDonald, R., Plonczak, A., Kang, J., Lee, C., Whangbo, M., Lancaster, T., Steele, A., Franke, I., Wright, J., Blundell, S., Pratt, F., deGeorge, J., Turnbull, M. & Landee, C. (2012). *Inorg. Chem.* **51**, 2121–2129.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Takeuchi, Y., Shibata, T. & Shirakami, T. (2000). *Jpn. Kokai Tokkyo Koho*, **9**, 99–105.
- Zordan, F. & Brammer, L. (2006). *Cryst. Growth Des.* **6**, 1374–1379.

supporting information

Acta Cryst. (2013). E69, o1149 [https://doi.org/10.1107/S1600536813016371]

1,6-Dibromonaphthalen-2-ol methanol monosolvate

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S1. Comment

Naphthol-containing compounds have gained popularity recently in the pharmaceutical industry as they have potential applications in the synthesis of antipsychotic medications (Costa *et al.*, 2012). The title compound has unique applications as a peroxidase enhancer in peroxidase-catalyzed oxidation reactions (Takeuchi *et al.*, 2000). It is also used to stabilize the two-component system for chemiluminescent assay in immunodiagnosics (Kalra & Kumar, 2005).

The molecule of the title compound is planar to within 0.069 (1) Å for all non-H atoms (Fig. 1).

In the crystal, molecules are linked by two pairs of O—H \cdots O hydrogen bonds, involving the methanol molecule, forming dimer-like arrangements (Table 1 and Fig. 2).

The crystal network is further stabilized by π stacking of the naphthol rings with a Cg1 \cdots Cg2ⁱ centroid-centroid distance of 3.676 (2) Å [Cg1 and Cg2 are the centroids of rings C1—C4/C7/C8 and C3—C6/C9/C10, respectively; symmetry code: (i) $x - 1, y, z$] (see Fig. 2). The crystal structure is also composed of a tetramer of Br \cdots Br contacts, which measure 3.480 (1) Å [Br1 \cdots Br1ⁱⁱ; symmetry code: (ii) $-x, -y + 1, -z + 1$] and 3.786 (1) Å [Br2 \cdots Br1ⁱⁱⁱ; symmetry code: (iii) $-x + 1/2, y + 1/2, -z + 3/2$]. These contacts are within the normal range of Br \cdots Br interactions, which are typically 3.05 Å to 3.80 Å (Zordan & Brammer, 2006; Schlueter *et al.*, 2012; Desiraju & Parthasarathy, 1989).

S2. Experimental

Approximately 100 mg of 1,6-dibromo-2-naphthol (Sigma-Aldrich) was dissolved in a 2 ml 50% methanol: 50% hexanes solution. On slow evaporation over the course of two weeks colourless plate-like crystals were obtained. The crystals decomposed rapidly when removed from the mother liquor.

S3. Refinement

The OH and C-bound H atoms were included in calculate positions and treated as riding atoms: O—H = 0.84 Å, C—H = 0.95 and 0.98 Å for CH and CH₃ H atoms, respectively, with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O}, \text{C-methyl})$ and $= 1.2U_{\text{eq}}(\text{C})$ for other H atoms. A residual density peak of 1.31 e/Å³ was located near atom C10. Twinning was not found and no disorder could be modeled.

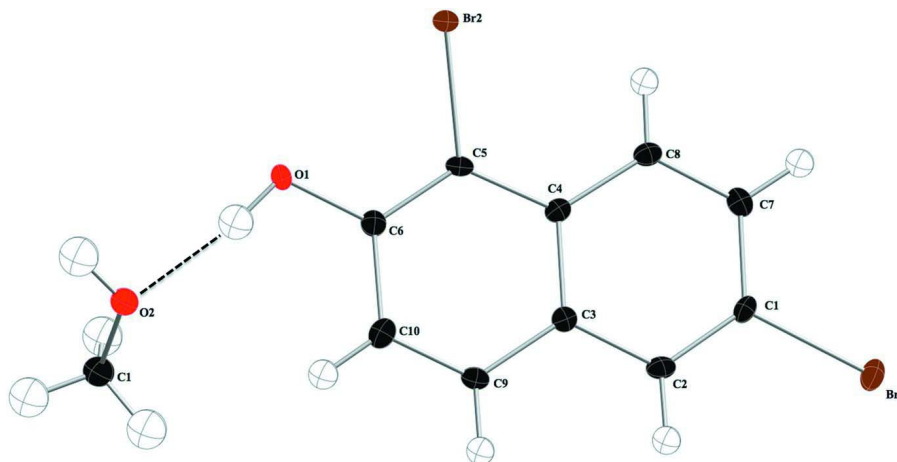


Figure 1

The molecular structure of the title compound, with atom labelling. Displacement ellipsoids are drawn at the 50% probability level. The intermolecular O—H...O hydrogen bond is shown as a dashed line.

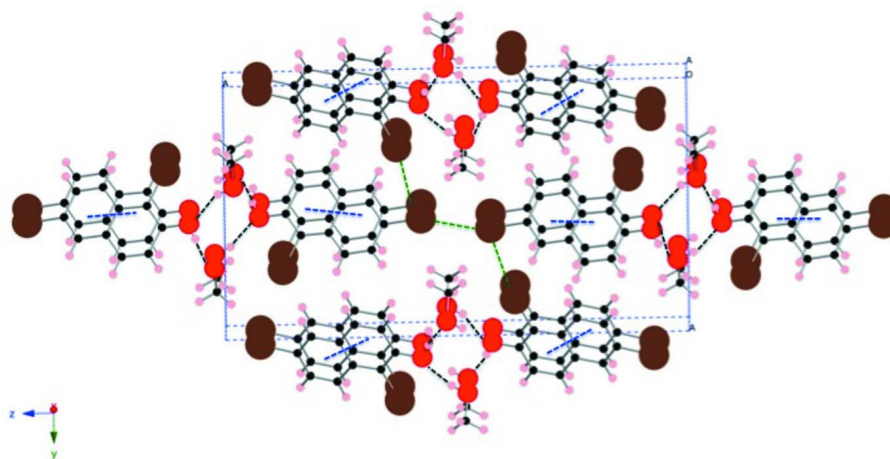


Figure 2

A view along the *a* axis of the crystal packing of the title compound. The tetramer of Br...Br contacts, the π stacking of the naphthol rings, and the O—H...O hydrogen bonds (Table 1) are shown as green, blue and black dashed lines, respectively.

1,6-Dibromonaphthalen-2-yl methanol monosolvate

Crystal data

$C_{10}H_6Br_2O \cdot CH_4O$
 $M_r = 334.01$
 Monoclinic, $P2_1/n$
 Hall symbol: $P 2_1/n$
 $a = 3.9971(4) \text{ \AA}$
 $b = 12.4705(12) \text{ \AA}$
 $c = 22.462(2) \text{ \AA}$
 $\beta = 92.442(1)^\circ$
 $V = 1118.62(19) \text{ \AA}^3$
 $Z = 4$

$F(000) = 648$
 $D_x = 1.983 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
 Cell parameters from 125 reflections
 $\theta = 5.5\text{--}28.9^\circ$
 $\mu = 7.22 \text{ mm}^{-1}$
 $T = 100 \text{ K}$
 Plate, colourless
 $0.26 \times 0.11 \times 0.01 \text{ mm}$

Data collection

Bruker APEXII CCD
diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8.3333 pixels mm⁻¹
 ω and φ scans
Absorption correction: multi-scan
(*SADABS*; Bruker, 2011)
 $T_{\min} = 0.521$, $T_{\max} = 0.746$

12658 measured reflections
2690 independent reflections
2082 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.057$
 $\theta_{\max} = 28.5^\circ$, $\theta_{\min} = 1.8^\circ$
 $h = -5 \rightarrow 5$
 $k = -16 \rightarrow 15$
 $l = -29 \rightarrow 29$

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.036$
 $wR(F^2) = 0.074$
 $S = 1.02$
2690 reflections
138 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
 $w = 1/[\sigma^2(F_o^2) + (0.0264P)^2 + 1.7105P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.002$
 $\Delta\rho_{\max} = 1.31 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.60 \text{ e } \text{\AA}^{-3}$

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 e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.11971 (11)	0.48530 (3)	0.57493 (2)	0.0238 (1)
Br2	0.46378 (10)	0.69738 (3)	0.87432 (2)	0.0191 (1)
O1	0.8338 (7)	0.50830 (19)	0.92461 (11)	0.0218 (9)
C1	0.2711 (9)	0.5134 (3)	0.65501 (16)	0.0160 (11)
C2	0.4589 (9)	0.4385 (3)	0.68511 (17)	0.0181 (11)
C3	0.5579 (9)	0.4567 (3)	0.74594 (17)	0.0154 (11)
C4	0.4629 (9)	0.5538 (3)	0.77388 (16)	0.0158 (10)
C5	0.5664 (9)	0.5675 (3)	0.83471 (17)	0.0155 (10)
C6	0.7448 (9)	0.4917 (3)	0.86645 (17)	0.0176 (11)
C7	0.1771 (9)	0.6095 (3)	0.68189 (17)	0.0185 (11)
C8	0.2723 (9)	0.6286 (3)	0.74024 (17)	0.0178 (11)
C9	0.7470 (9)	0.3789 (3)	0.77869 (17)	0.0175 (11)
C10	0.8383 (9)	0.3942 (3)	0.83702 (17)	0.0197 (11)
O2	0.1551 (8)	0.3491 (2)	0.97853 (13)	0.0287 (9)
C11	-0.0041 (11)	0.2471 (3)	0.98332 (19)	0.0286 (14)
H1	0.94170	0.45510	0.93800	0.0330*
H2A	0.52360	0.37460	0.66570	0.0220*

H7	0.04810	0.66100	0.65980	0.0220*
H8	0.20790	0.69390	0.75830	0.0210*
H9	0.81130	0.31480	0.75950	0.0210*
H10	0.96350	0.34080	0.85830	0.0240*
H2	0.17580	0.37740	1.01240	0.0430*
H11A	0.11630	0.20400	1.01380	0.0430*
H11B	-0.00170	0.20990	0.94490	0.0430*
H11C	-0.23620	0.25730	0.99460	0.0430*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0299 (2)	0.0256 (2)	0.0156 (2)	-0.0058 (2)	-0.0031 (2)	0.0001 (2)
Br2	0.0230 (2)	0.0152 (2)	0.0191 (2)	0.0037 (2)	-0.0005 (2)	-0.0024 (2)
O1	0.0330 (17)	0.0166 (13)	0.0152 (14)	0.0041 (11)	-0.0052 (12)	0.0022 (11)
C1	0.0176 (19)	0.0182 (18)	0.0122 (18)	-0.0078 (15)	0.0007 (15)	-0.0009 (14)
C2	0.019 (2)	0.0136 (17)	0.022 (2)	-0.0052 (15)	0.0031 (16)	-0.0035 (15)
C3	0.0129 (18)	0.0165 (17)	0.017 (2)	-0.0035 (14)	0.0041 (15)	0.0011 (14)
C4	0.0148 (18)	0.0159 (17)	0.0168 (19)	-0.0043 (14)	0.0028 (15)	-0.0015 (15)
C5	0.0137 (18)	0.0121 (16)	0.021 (2)	0.0005 (14)	0.0032 (15)	-0.0023 (14)
C6	0.0181 (19)	0.0160 (17)	0.0187 (19)	-0.0041 (15)	-0.0001 (15)	0.0011 (15)
C7	0.0165 (19)	0.0158 (18)	0.023 (2)	-0.0040 (15)	0.0002 (16)	0.0050 (15)
C8	0.018 (2)	0.0156 (17)	0.020 (2)	-0.0036 (14)	0.0039 (16)	-0.0027 (15)
C9	0.0171 (19)	0.0135 (17)	0.022 (2)	0.0002 (14)	0.0029 (16)	-0.0008 (15)
C10	0.0159 (19)	0.024 (2)	0.019 (2)	-0.0074 (15)	-0.0001 (16)	-0.0015 (16)
O2	0.0408 (18)	0.0207 (14)	0.0239 (16)	0.0050 (13)	-0.0063 (14)	-0.0016 (12)
C11	0.034 (3)	0.025 (2)	0.027 (2)	0.0026 (18)	0.0030 (19)	0.0009 (18)

Geometric parameters (Å, °)

Br1—C1	1.906 (4)	C5—C6	1.366 (5)
Br2—C5	1.901 (4)	C6—C10	1.441 (5)
O1—C6	1.355 (5)	C7—C8	1.370 (5)
O1—H1	0.8400	C9—C10	1.359 (5)
O2—C11	1.428 (5)	C2—H2A	0.9500
O2—H2	0.8400	C7—H7	0.9500
C1—C2	1.360 (5)	C8—H8	0.9500
C1—C7	1.400 (5)	C9—H9	0.9500
C2—C3	1.424 (5)	C10—H10	0.9500
C3—C9	1.417 (5)	C11—H11A	0.9800
C3—C4	1.423 (5)	C11—H11B	0.9800
C4—C5	1.421 (5)	C11—H11C	0.9800
C4—C8	1.404 (5)		
C6—O1—H1	110.00	C4—C8—C7	121.4 (3)
C11—O2—H2	109.00	C3—C9—C10	121.3 (3)
Br1—C1—C7	119.0 (3)	C6—C10—C9	119.8 (3)
Br1—C1—C2	119.3 (3)	C3—C2—H2A	120.00

C2—C1—C7	121.7 (3)	C1—C2—H2A	120.00
C1—C2—C3	119.5 (3)	C1—C7—H7	120.00
C2—C3—C4	119.4 (3)	C8—C7—H7	120.00
C2—C3—C9	120.6 (3)	C7—C8—H8	119.00
C4—C3—C9	120.0 (3)	C4—C8—H8	119.00
C3—C4—C8	118.5 (3)	C3—C9—H9	119.00
C3—C4—C5	117.0 (3)	C10—C9—H9	119.00
C5—C4—C8	124.5 (3)	C6—C10—H10	120.00
Br2—C5—C6	117.6 (3)	C9—C10—H10	120.00
C4—C5—C6	122.9 (3)	O2—C11—H11A	109.00
Br2—C5—C4	119.5 (3)	O2—C11—H11B	110.00
O1—C6—C10	120.6 (3)	O2—C11—H11C	109.00
C5—C6—C10	119.0 (3)	H11A—C11—H11B	109.00
O1—C6—C5	120.5 (3)	H11A—C11—H11C	109.00
C1—C7—C8	119.5 (3)	H11B—C11—H11C	110.00
Br1—C1—C2—C3	-176.7 (3)	C3—C4—C5—C6	-1.0 (5)
C7—C1—C2—C3	1.3 (6)	C8—C4—C5—Br2	-3.1 (5)
Br1—C1—C7—C8	177.2 (3)	C8—C4—C5—C6	178.3 (4)
C2—C1—C7—C8	-0.8 (6)	C3—C4—C8—C7	0.2 (5)
C1—C2—C3—C4	-1.0 (5)	C5—C4—C8—C7	-179.1 (4)
C1—C2—C3—C9	178.6 (3)	Br2—C5—C6—O1	2.1 (5)
C2—C3—C4—C5	179.6 (3)	Br2—C5—C6—C10	-177.5 (3)
C2—C3—C4—C8	0.2 (5)	C4—C5—C6—O1	-179.2 (3)
C9—C3—C4—C5	0.1 (5)	C4—C5—C6—C10	1.2 (6)
C9—C3—C4—C8	-179.3 (3)	O1—C6—C10—C9	-180.0 (3)
C2—C3—C9—C10	-178.8 (4)	C5—C6—C10—C9	-0.3 (5)
C4—C3—C9—C10	0.7 (6)	C1—C7—C8—C4	0.0 (6)
C3—C4—C5—Br2	177.6 (3)	C3—C9—C10—C6	-0.6 (6)

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1 \cdots O2 ⁱ	0.84	1.80	2.632 (4)	171
O2—H2 \cdots O1 ⁱⁱ	0.84	2.01	2.809 (4)	159

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