

Naphthalene-1,3-diol

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Received 22 November 2005

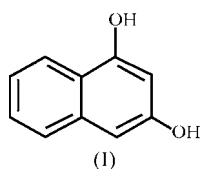
Accepted 6 December 2005

Online 14 January 2006

The molecules of the title compound, $C_{10}H_8O_2$, are linked by two $O-H \cdots O$ hydrogen bonds, which form infinite chains with a graph-set descriptor of $C(6)$. These chains are linked into puckered (100) sheets of $R_4^4(8)$ and $R_4^4(24)$ rings. Adjacent sheets are connected by weak $C-H \cdots \pi$ and $\pi-\pi$ interactions into a continuous three-dimensional network.

Comment

In previous papers, we have reported the structures of some mono- and dihydroxynaphthalenes, such as 1-hydroxynaphthalene (1-naphthol; Rozycka-Sokolowska *et al.*, 2004), 2-hydroxynaphthalene (2-naphthol; Marciniak *et al.*, 2003) and 2,7-dihydroxynaphthalene (2,7-naphthalenediol; Rozycka-Sokolowska *et al.*, 2005). Continuing this research, the results of structural studies of naphthalene-1,3-diol, commonly known as naphthoresorcinol, (I) (Fig. 1), are presented here.



The molecule of (I) contains a naphthalene ring with two hydroxyl groups attached to it on atoms C1 and C3. The presence of hydroxyl substituents at positions 1 and 3 modifies the geometric parameters within the aromatic rings. The C—C bond distances (Table 1) vary from 1.354 (3) to 1.410 (2) Å; bonds C1—C2, C3—C4, C6—C7 and C8—C9 are shorter than the typical aromatic bond length of 1.384 (13) Å (Allen *et al.*, 1987), whereas all the other bonds in the aromatic rings are longer. The values of the bond angles within the aromatic rings vary from 117.9 (2) to 121.7 (2)°. Despite these variations in bond lengths and angles, the naphthalene ring remains planar, with an average out-of-plane deviation of 0.006 (2) Å. Atoms O11 and O12 attached to the naphthalene ring deviate from its plane by only -0.034 (1) and 0.033 (1) Å, respectively.

The unit cell of (I) consists of four molecules, which occupy one non-equivalent set of general positions. The molecules are

linked together by two strong $O11-H11 \cdots O12$ (A in Fig. 2) and $O12-H12 \cdots O11$ (B in Fig. 2) hydrogen bonds, forming infinite chains which run parallel to the *b* and *c* axes, respectively, and which both have a graph-set motif of $C(6)$ (Bernstein *et al.*, 1995). Together, these hydrogen bonds produce a deeply puckered sheet parallel to (100) containing $R_4^4(8)$ and $R_4^4(24)$ rings, which are arranged alternately in a chessboard fashion (Fig. 2). The non-H atoms belonging to the (100) sheets lie in domains $(-0.28 + x) < a < (x + 1.28)$ (*x* is zero or an integer). Each sheet is interwoven with two neighbouring sheets and is linked to them by weak $C-H \cdots \pi$ and $\pi-\pi$ interactions to form a continuous three-dimensional network (Fig. 3).

Atom C9 in the molecule at (*x*, *y*, *z*), belonging to the (100) sheet in domain $-0.28 < x < 1.28$ acts as a hydrogen-bond donor, *via* atom H9, to the C5—C10 benzene ring (centroid *Cg*2) of the molecule at $(-x, \frac{1}{2} + y, \frac{1}{2} - z)$, which belongs to the adjacent sheet lying in domain $-1.28 < a < 0.28$ (Fig. 3 and Table 2). As mentioned above, in the structure of (I) there are also $\pi-\pi$ stacking interactions involving the C1—C5/C10 (centroid *Cg*1) and C5—C10 (centroid *Cg*2) rings (Fig. 3). The

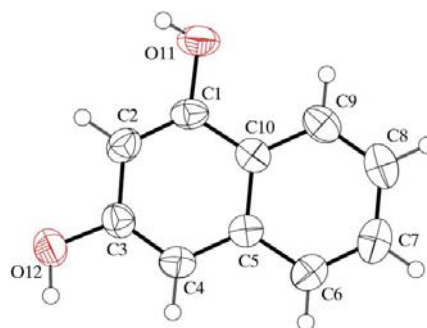


Figure 1

A view of the molecule of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

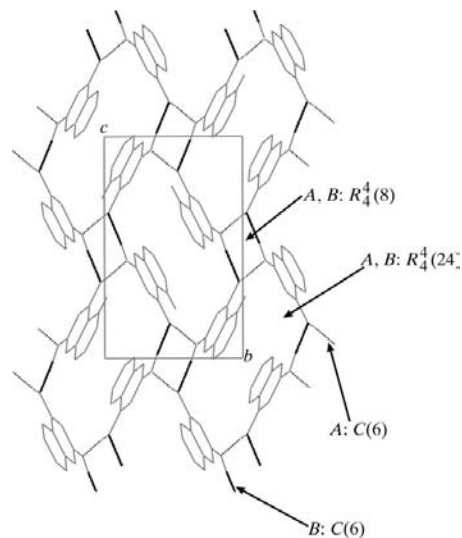


Figure 2

A unit-cell plot of (I), showing the intermolecular hydrogen bonds in the crystal structure and the assigned graph-set motifs. Hydrogen bonds are shown as dashed (motif A) and heavy (motif B) black lines. All C-bound H atoms have been omitted for clarity.

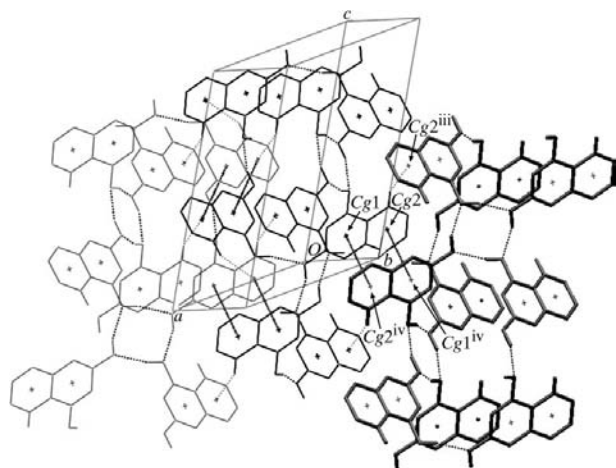


Figure 3

Part of the crystal structure of (I), showing the (100) sheets lying in domains $-1.28 < a < 0.28$ (heavy grey lines), $-0.28 < a < 1.28$ (thin black lines) and $0.72 < a < 2.28$ (thin grey lines), as well as the intermolecular O—H...O hydrogen bonds (dashed black lines), C—H... π hydrogen bonds (dashed heavy grey lines) and π — π interactions (heavy grey dotted lines). [Symmetry codes: (iii) $-x, \frac{1}{2} + y, \frac{1}{2} - z$; (iv) $-x, 2 - y, -z$.] Cg1 and Cg2 are the centroids of the C1—C5/C10 and C5—C10 benzene rings, respectively, and are denoted by small crosses. H atoms, except for atoms H9, H11 and H12, have been omitted for clarity.

perpendicular distance of the ring centroids Cg1 and Cg2 from the symmetry-related centroids at $(-x, 2 - y, -z)$, i.e. Cg2^{iv} and Cg1^{iv}, respectively, is 3.42 Å, and the centroid-centroid separation is 3.68 Å. The planes of these rings are practically parallel, making an angle of only 0.6°.

Experimental

Naphthalene-1,3-diol was obtained from Sigma (purity 99%) and used without further purification. Despite many attempts, we were initially unable to grow single crystals suitable for X-ray structure analysis. Among the seven organic solvents (ethanol, methanol, butanol, chloroform, acetone, ethyl acetate and xylene) used in the growth experiments carried out with the help of temperature-lowering and evaporation techniques, only xylene proved suitable for the growth of high-quality crystals using the latter method. Only polycrystalline material was obtained when recrystallization was attempted from the other solvents.

Crystal data

C₁₀H₈O₂
M_r = 160.16
 Monoclinic, *P*₂₁/*c*
a = 9.061 (2) Å
b = 7.203 (1) Å
c = 13.309 (3) Å
 β = 119.96 (3)°
V = 752.5 (4) Å³
Z = 4
D_x = 1.414 Mg m⁻³

Mo *K* α radiation
 Cell parameters from 3650 reflections
 θ = 4.6–25.0°
 μ = 0.10 mm⁻¹
T = 293 (2) K
 Plate, red
 0.50 × 0.30 × 0.05 mm

Data collection

Oxford Xcalibur3 CCD area-detector diffractometer
 ω scans
 3650 measured reflections
 1312 independent reflections
 1139 reflections with *I* > 2 σ (*I*)

*R*_{int} = 0.025
 θ_{max} = 25.0°
h = -8 → 10
k = -7 → 8
l = -15 → 14

Refinement

Refinement on *F*²
R[*F*² > 2 σ (*F*²)] = 0.049
wR(*F*²) = 0.119
S = 1.17
 1312 reflections
 118 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0492P)^2 + 0.1818P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.007$
 $\Delta\rho_{\text{max}} = 0.13 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.16 \text{ e \AA}^{-3}$
 Extinction correction: SHELXL97 (Sheldrick, 1997)
 Extinction coefficient: 0.065 (9)

Table 1

Selected geometric parameters (Å, °).

C1—C2	1.353 (3)	C5—C6	1.406 (3)
C1—O11	1.368 (2)	C5—C10	1.408 (3)
C1—C10	1.406 (3)	C6—C7	1.350 (3)
C2—C3	1.396 (3)	C7—C8	1.396 (3)
C3—C4	1.342 (3)	C8—C9	1.352 (3)
C3—O12	1.369 (2)	C9—C10	1.403 (3)
C4—C5	1.404 (3)		
C2—C1—O11	122.6 (2)	C4—C5—C10	120.1 (2)
C2—C1—C10	121.1 (2)	C6—C5—C10	118.3 (2)
O11—C1—C10	116.3 (2)	C7—C6—C5	120.6 (2)
C1—C2—C3	119.7 (2)	C6—C7—C8	121.0 (2)
C4—C3—O12	122.5 (2)	C9—C8—C7	120.1 (2)
C4—C3—C2	121.7 (2)	C8—C9—C10	120.4 (2)
O12—C3—C2	115.9 (2)	C9—C10—C1	122.5 (2)
C3—C4—C5	119.5 (2)	C1—C10—C5	117.9 (2)
C4—C5—C6	121.6 (2)		

Table 2

Hydrogen-bond geometry (Å, °).

Cg2 is the centroid of the C5—C10 ring.

D—H...A	D—H	H...A	D...A	D—H...A
O11—H11...O12 ⁱ	0.84 (4)	1.89 (4)	2.709 (2)	163 (3)
O12—H12...O11 ⁱⁱ	0.90 (2)	1.93 (3)	2.766 (2)	156 (3)
C9—H9...Cg2 ⁱⁱⁱ	0.93	2.90	3.710 (2)	146

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

H atoms bonded to C atoms were allowed for in geometrically idealized positions, with C—H distances of 0.93 Å and with *U*_{iso}(H) values 1.2 times the *U*_{eq} of the parent atoms. The H atoms of the hydroxyl groups were located in difference maps and refined isotropically, giving O—H distances of 0.84 (4) and 0.90 (2) Å.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2002); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2002); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003) and *RPLUTO* (Motherwell *et al.*, 2000); software used to prepare material for publication: *SHELXL97*.

This paper was partly financed by the Financial Means for Science in 2005–2006, as project No. 3 T09A 108 29.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: GD1424). Services for accessing these data are described at the back of the journal.

References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
- Bernstein, J., Davis, R. E., Shimon, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
- Marciniak, B., Rozycka-Sokolowska, E. & Pavlyuk, V. (2003). *Acta Cryst.* **E59**, o52–o53.
- Motherwell, W. D. S., Shields, G. P. & Allen, F. H. (2000). *Acta Cryst.* **B56**, 466–473.
- Oxford Diffraction (2002). *CrysAlis CCD* and *CrysAlis RED*. Oxford Diffraction Poland, Wrocław, Poland.
- Rozycka-Sokolowska, E., Marciniak, B. & Pavlyuk, V. (2004). *Acta Cryst.* **E60**, o884–o885.
- Rozycka-Sokolowska, E., Marciniak, B. & Pavlyuk, V. (2005). *Acta Cryst.* **E61**, o114–o115.
- Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.
- Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.