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2-Bromo-1-(1-hydroxynaphthalen-2-yl)ethanone

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In the structure of the title compound, $C_{12}H_9BrO_2$, all bond lengths and angles are normal. There is an O—H···O intramolecular hydrogen bond and a weak intermolecular C—H···O hydrogen bond.

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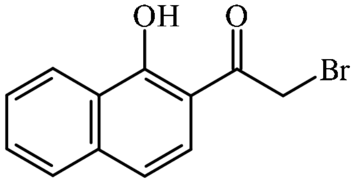
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.009 Å
R factor = 0.052
wR factor = 0.135
Data-to-parameter ratio = 14.8

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

Comment

Because of our interest in acetylnaphthalene derivatives we synthesized the title compound, (I), as a starting compound for producing some potentially pharmacologically active compounds (Walker *et al.*, 1981; Çalış *et al.*, 1988; Karakurt *et al.*, 2001). According to the literature and our own experience, a hydroxy group on the ring affects the reactions of the acetyl group, especially some of the reactions of the carbonyl group and the bromination of the α-methyl group, probably because of the hydrogen bonding between the carbonyl and hydroxy groups (Chang & Yang, 1954; Bouzard *et al.*, 1972).



(I)

In this study we aimed to investigate the intra- and/or intermolecular hydrogen bonding and the conformation of the title compound by X-ray crystallography. Because the crystal was a very weak scatterer, the structure was also confirmed by IR and NMR spectroscopic data.

As is apparent from Figs. 1 and 2, the molecule as a whole is planar. The maximum deviation of the non-H atoms from their mean plane is 0.015 (7) Å for C4.

The title compound is stabilized by hydrogen bonds, namely C12—H12*B*···O1($-x-1$, $y+\frac{1}{2}$, $\frac{1}{2}-z$) and O2—H2O···O1

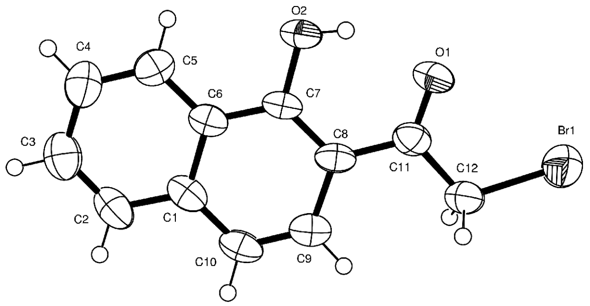


Figure 1
The structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

(Table 2). Hydroxy atom O2 acts as donor to atom O1 in an intramolecular hydrogen bond. The intramolecular hydrogen-bond parameters are similar to those for coumarin-3-carboxylic acid (Dobson & Gerkin, 1996): donor–acceptor distance 2.559 (6) Å, H atom–acceptor distance slightly greater than 1.72 Å, (1.83 Å), donor–H-atom–acceptor angle 150°. In addition, there is a weak C—H···O intermolecular hydrogen bond that links the molecules along the *b* axis. There are only van der Waals interactions along the *a* and *c* axis directions.

There are also π–π stacking interactions of the aromatic rings. The perpendicular distance between the C1–C6 ring and the C1/C6–C10 ring in a neighboring molecule is 3.46 Å.

Experimental

For the synthesis of 1-acetyloxy-2-acetylnaphthalene, (II), 1-hydroxy-2-acetylnaphthalene (1.86 g, 0.01 mol) was added to a mixture of acetic anhydride (12.5 ml) and anhydrous pyridine (1.5 ml); the mixture was shaken to dissolve the reactants and allowed to stand for 12 h at 298 K. The mixture was poured into stirred ice-water. The solid precipitate was filtered off, washed with dilute HCl and recrystallized from ethanol (yield 2.15 g, 94%; m.p 376 K). For the synthesis of 1-acetyloxy-2-bromoacetylnaphthalene, (III), compound (II) (0.57 g, 2.5 mmol) was dissolved in CCl_4 (25 ml) and was added in small portions to bromine (0.13 ml) in CCl_4 (3.87 ml), waiting for decolorization of the bromine after each addition. The solvent was evaporated, the oily residue was dissolved in benzene (4 ml) and hexane (25 ml) was added. The precipitate was filtered off, dried and recrystallized from hexane (yield 2.14 g, 69%; m.p 344–346 K). For the preparation of (I), compound (III) (0.47 g, 1.53 mmol) was dissolved in methanol (5 ml) and was added to 48% hydrobromic acid in aqueous solution. The compound crystallized as yellow plate-shaped crystals (yield 0.32 g, 79%; m.p 399–401 K). IR (KBr, cm^{-1}): 3120 (C—H aromatic), 1611 (C═O), 931 (N—O), 887, 794 (C—H naphthalene A), 769 (C—H naphthalene B). ^1H NMR ($CDCl_3$, 80 MHz): δ 4.50 (*s*, 2H, CH_2), 6.80–8.50 (*m*, 6H, aromatic rings).

Crystal data

$C_{12}H_9BrO_2$
$M_r = 265.10$
Monoclinic, $P2_1/c$
$a = 4.9207\ (5)$ Å
$b = 10.5041\ (10)$ Å
$c = 20.357\ (2)$ Å
$\beta = 100.293\ (8)°$
$V = 1035.28\ (18)$ Å^3
$Z = 4$

$D_x = 1.701$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 12 498 reflections
$\theta = 1.9$–$27.1°$
$\mu = 3.94$ mm^{-1}
$T = 293\ (2)$ K
Plate, yellow
0.50 × 0.23 × 0.02 mm

Data collection

Stoe IPDS-II diffractometer
ω scans
Absorption correction: by integration (*X-RED*32; Stoe & Cie, 2002)
$T_{min} = 0.309$, $T_{max} = 0.794$
7371 measured reflections

2007 independent reflections
842 reflections with $I > 2\sigma(I)$
$R_{int} = 0.092$
$\theta_{max} = 26.0°$
$h = -5 \rightarrow 6$
$k = -12 \rightarrow 12$
$l = -25 \rightarrow 25$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.135$
$S = 0.87$
2007 reflections
136 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0602P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.61$ e Å^{-3}
$\Delta\rho_{min} = -0.59$ e Å^{-3}

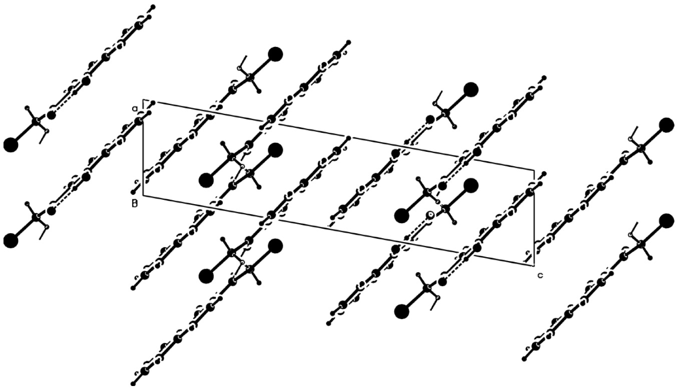


Figure 2
A view of the packing of molecules in the unit cell.

Table 1
Selected geometric parameters (Å, °).

O2—C7	1.366 (7)	O1—C11	1.224 (7)
Br1—C12	1.909 (6)		
O1—C11—C8	122.4 (5)	C8—C11—C12	117.6 (5)
O1—C11—C12	119.9 (6)		
C7—C8—C11—O1	3.4 (10)	C8—C11—C12—Br1	175.9 (5)
C9—C8—C7—O2	178.4 (6)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C12—H12*B*···O1^i	0.97	2.53	3.265 (8)	132
O2—H2O···O1	0.82	1.83	2.559 (6)	150

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

H atoms were positioned geometrically and refined using a riding model, fixing the aromatic C—H distances at 0.93 Å, the CH_2 C—H distances at 0.97 Å and the O—H distance at 0.82 Å, with $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}(C,O)$.

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*III (Burnett & Johnson, 1996); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PARST* (Nardelli, 1995).

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