1-(Piperidin-1-ylmethyl)-2-naphthol

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Key indicators

Single-crystal X-ray study
$T = 294$ K
Mean σ(C–C) = 0.004 Å
R factor = 0.051
wR factor = 0.159
Data-to-parameter ratio = 16.1

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

The title compound, $C_{16}H_{19}NO$, is a product of a Mannich reaction involving 2-naphthol, formaldehyde and piperidine. The piperidine ring has a chair conformation. The molecular structure is stabilized by an intramolecular O—H···N hydrogen bond, linking the naphthol OH group and the piperidine N atom.

Comment

The Mannich reaction (Arend *et al.*, 1998; Bur & Martin, 2001) is an important carbon–carbon bond-formation reaction widely used in the synthesis of secondary and tertiary amine derivatives and applied as a key step in the synthesis of many bioactive molecules and complex natural products (Ito *et al.*, 2001; Liras *et al.*, 2001). Recently, 1-piperidinomethyl-2-naphthol, (I), has been prepared by the Mannich reaction (Shriner *et al.*, 1946). The crystal structure of (I) is reported in this paper.

(I)
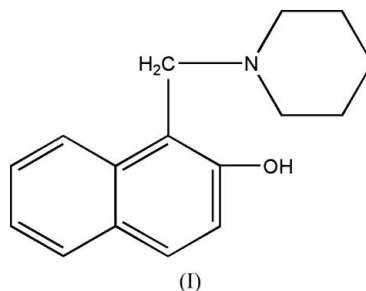
The molecular structure of (I) is shown in Fig. 1. In the crystal structure, the piperidine ring has the usual chair conformation. The naphthol ring system is almost planar. The C1/C10/C11/H1/O1 plane is almost coplanar with the naphthol plane, deviating from it by 0.0496 (2) Å; the N atom deviates from this latter plane by 0.449 (2) Å. An intramolecular hydrogen bond is formed between the hydroxyl group and the N atom, stabilizing the molecular structure.

Experimental

A mixture of piperidine (85 g) and formalin (75 g) was chilled to 278 K. A solution of 115 g of 2-naphthol in 450 ml of 95% ethanol was chilled to 278 K, and then added in three portions to the above mixture with mechanical stirring. The product crystallized in a short time and was collected by filtration and washed with a small amount of ethanol. Crystallization from 95% ethanol gave colorless crystals which melted at 367.5–368.5 K. The yield amounted to 198 g (82%). Spectroscopic analysis: IR (KBr, ν cm^{-1}): 3430, 2970, 2847, 1517, 1475, 1236; ^{1}H NMR (CDCl$_3$, δ, p.p.m.): 9.83 (*m*, 1H), 6.84–8.13 (*m*, 6H), 4.06 (*s*, 2H), 2.24 (*m*, 4H), 1.50 (*m*, 6H).

Crystal data

$C_{16}H_{19}NO$
$M_r = 241.32$
Monoclinic, $P2_1/c$
$a = 6.3262\ (19)$ Å
$b = 9.360\ (3)$ Å
$c = 22.629\ (7)$ Å
$\beta = 96.924\ (5)°$
$V = 1330.2\ (7)$ Å^3
$Z = 4$

$D_x = 1.205$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1065
    reflections
$\theta = 2.4–21.2°$
$\mu = 0.08$ mm^{-1}
$T = 294\ (2)$ K
Block, colorless
$0.26 \times 0.22 \times 0.20$ mm

Data collection

Bruker SMART CCD area-detector
    diffractometer
φ and ω scans
Absorption correction: multi-scan
    (*SADABS*; Sheldrick, 1996)
    $T_{min} = 0.981$, $T_{max} = 0.985$
7229 measured reflections

2702 independent reflections
1152 reflections with $I > 2\sigma(I)$
$R_{int} = 0.062$
$\theta_{max} = 26.4°$
$h = -7 \rightarrow 6$
$k = -11 \rightarrow 9$
$l = -27 \rightarrow 28$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.159$
$S = 0.94$
2702 reflections
168 parameters
H atoms treated by a mixture of
    independent and constrained
    refinement

$w = 1/[\sigma^2(F_o^2) + (0.0743P)^2]$
    where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.024 (4)

Table 1
Selected bond lengths (Å).

O1—C1	1.360 (3)	N1—C16	1.462 (3)
N1—C12	1.460 (3)	N1—C11	1.474 (3)

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

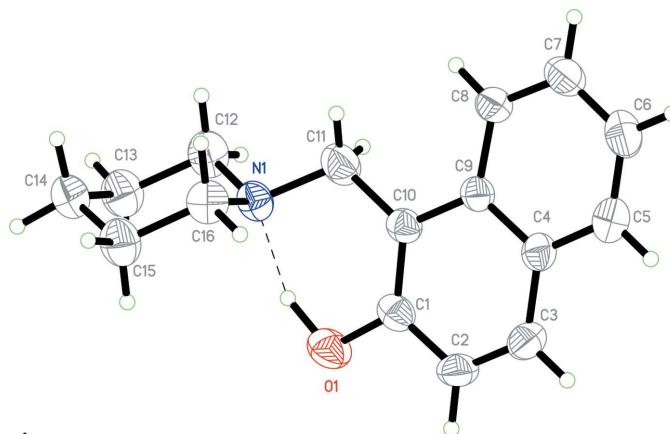| D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O1—H1$\cdots$N1	1.04 (4)	1.62 (4)	2.590 (3)	153 (3)

Figure 1
View of the molecule of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 35% probability level.

The H atom of the hydroxy group was located in a difference Fourier map and was refined isotropically. The remaining H atoms were positioned geometrically (C–H = 0.93–0.97 Å) and refined as riding on their parent atoms, with $U_{iso}(H) = 1.2U_{eq}$(carrier atom).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

References

Arend, M., Westermann, B. & Risch, N. (1998). *Angew. Chem. Int. Ed.* **37**, 1044–1070.
Bruker (1997). *SMART, SAINT and SHELXTL.* Versions 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Bur, S. K. & Martin, S. F. (2001). *Tetrahedron*, **57**, 3221–3242.
Ito, M., Clark, C. W., Mortimore, M., Goh, J. B. & Martin, S. F. (2001). *J. Am. Chem. Soc.* **123**, 8003–8010.
Liras, S., Davoren, J. E. & Bordner, J. (2001). *Org. Lett.* **3**, 703–706.
Sheldrick, G. M. (1996). *SADABS.* University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97.* University of Göttingen, Germany.
Shriner, R. L., Grillot, G. F. & Teeters, W. O. (1946). *J. Am. Chem. Soc.* **68**, 946–947.