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1-Morpholinomethyl-2-naphthol

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.046
wR factor = 0.132
Data-to-parameter ratio = 18.3

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

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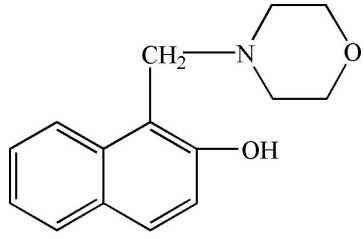
The title compound, $C_{15}H_{17}NO_2$, is a product of a Mannich reaction involving 2-naphthol, formaldehyde and morpholine. The morpholine ring has a chair conformation and the crystal structure is stabilized by an intramolecular O—H···N hydrogen bond linking the naphthol OH group and the morpholine N atom.

Comment

The Mannich reaction (Arend *et al.*, 1998; Bur & Martin, 2001) is an important C—C bond formation reaction widely used in the synthesis of secondary and tertiary amine derivatives, and is a key step in the synthesis of many bioactive molecules and complex natural products (Ito *et al.*, 2001; Liras *et al.*, 2001). We have prepared the title compound, (I), by the Mannich reaction (Shriner *et al.*, 1946) and the crystal structure of (I) is reported in this paper.



(I)

The molecular structure of (I) is shown in Fig. 1. In the structure, the morpholine ring has the usual chair conformation. The naphthol ring system is almost planar, with atoms C5 and C6 deviating by 0.0200 (2) and 0.0174 (2) Å, respectively, from the mean plane. An intramolecular hydrogen bond between the hydroxy group and the N atom stabilizes the crystal structure.

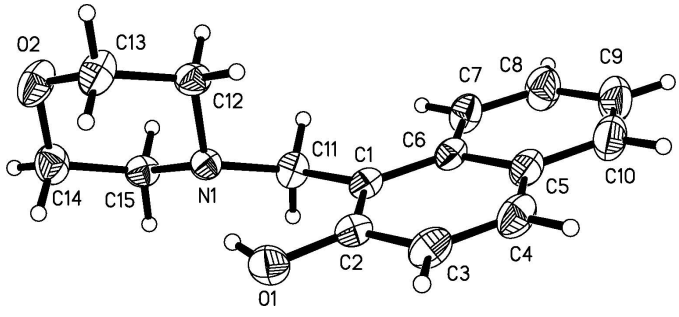


Figure 1
View of the molecule of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 35% probability level.

Experimental

A sample of 2-naphthol (68 g) was dissolved in 95% alcohol (450 ml) and cooled at 278 K. Morpholine (49 g) was added very slowly to formalin (38 g) cooled to 278 K. The 2-naphthol solution was added in three portions with stirring. A flaky crystalline precipitate was obtained. This was filtered, dried and recrystallized from 95% alcohol. Colorless plates were formed in 75% yield. M.p. 388–389 K. IR (KBr, cm^{-1}): ν 3433, 2974, 2851, 1458, 1116; ^1H NMR ($CDCl_3$, p.p.m.): δ 7.095–8.367 (*m*, 6H), 4.181 (*s*, 2H), 3.713–3.821 (*m*, 4H), 2.685–2.722 (*m*, 4H).

Crystal data

$C_{15}H_{17}NO_2$
M_r = 243.30
Monoclinic, $P2_1/c$
a = 22.162 (6) Å
b = 5.8941 (17) Å
c = 9.743 (3) Å
β = 91.120 (4)°
V = 1272.5 (6) Å^3
Z = 4
D_x = 1.270 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1509 reflections
θ = 2.8–22.9°
μ = 0.08 mm^{-1}
T = 293 (2) K
Block, colourless
0.26 × 0.24 × 0.22 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.978, T_{max} = 0.982
7978 measured reflections
3052 independent reflections
1908 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 28.0°
$h = -24 \rightarrow 29$
$k = -7 \rightarrow 7$
$l = -9 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.132
S = 1.08
3052 reflections
167 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.047P)^2 + 0.1845P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

Table 1
Selected bond lengths (Å).

N1—C15	1.462 (2)	N1—C11	1.472 (2)
N1—C12	1.465 (2)	O1—C2	1.364 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1	0.87 (1)	1.80 (1)	2.597 (2)	153 (2)

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 atom, with U_{iso}(H) values equal to $1.2U_{eq}$(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*.

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