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Methyl *N*-[(2-hydroxynaphthalen-1-yl)-(phenyl)methyl]carbamate

Ayoob Bazgir,[a] Vahid Amani[b] and Hamid Reza Khavasi[a]*

[a]Department of Chemistry, Shahid Beheshti University, Evin, Tehran 1983963113, Iran, and [b]Tehran Suburb Education Organization, Chahardangeh region, Tehran, Iran

Correspondence e-mail: h-khavasi@sbu.ac.ir

Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.005 Å
R factor = 0.070
wR factor = 0.133
Data-to-parameter ratio = 12.5

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

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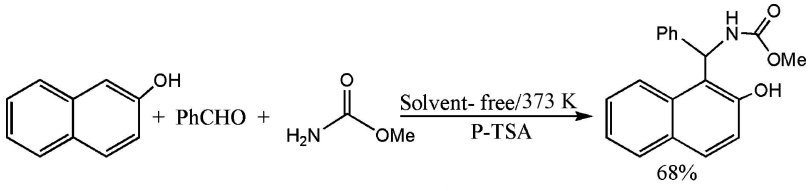
Intermolecular O—H···O and intramolecular N—H···O hydrogen bonds are effective in the stabilization of the crystal structure of the title compound, $C_{19}H_{17}NO_3$.

Comment

Multi-step syntheses produce considerable amounts of waste mainly due to a series of complex isolation procedures often involving environmentally unfavorable solvents after each step. Thus, the combination of steps into a multi-step, one-pot reaction sequence under solvent-free conditions can be economically and environmentally very advantageous as long as the overall yield and efficiency are not adversely affected (Domling & Ugi, 2000; Heck & Domling, 2000).

The carbamate function is widely encountered in the structure of biologically active compounds. These compounds are generally prepared from phosegene (Nowick *et al.* 1992), phosgene derivatives (Majer & Randad, 1994; Batey *et al.* 1998) or isocyanates (Ozaki, 1972), in reaction with alcohols. Nevertheless, none of these methods are environmentally benign. In light of the above, we have synthesized the title compound, (I), under solvent-free conditions and characterized its structure.



(I)

In the molecule of the title compound, (I) (Fig. 1), the bond lengths and angles are within normal ranges (Allen *et al.*, 1987).

The rings *A* (C4–C9), *B* (C10–C14/C19) and *C* (C14–C19) are, of course, planar and the dihedral angles between them are *A*/*B* = 81.54 (4)°, *A*/*C* = 83.98 (3)° and *B*/*C* = 2.51 (4)°.

As can be seen from the packing diagram (Fig. 2), the intermolecular O—H···O hydrogen bonds (Table 1) link the molecules, to form infinite chains along the *c* axis. The intermolecular O—H···O and intramolecular N—H···O hydrogen bonds seem to be effective in the stabilization of the crystal structure. Dipole–dipole and van der Waals interactions are effective in the molecular packing.

Experimental

2-Naphthol (1 mmol), benzaldehyde (1 mmol), methyl carbamate (1.5 mmol) and *p*-toluenesulfonic acid (0.1 g) were mixed and the

reaction mixture was placed in a screw-capped vial and heated at 373 K for 3 h. After cooling, the reaction mixture was washed with water and then recrystallized from EtOAc/hexane (1:3) to afford the pure product (yield 68%).

Crystal data

$C_{19}H_{17}NO_3$
M_r = 307.34
Monoclinic, $P2_1/c$
a = 9.217 (3) Å
b = 18.106 (5) Å
c = 11.305 (4) Å
β = 121.11 (2)°
V = 1615.4 (10) Å^3
Z = 4
D_x = 1.264 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 294 (2) K
Prism, yellow
0.3 × 0.15 × 0.1 mm

Data collection

Stoe IPDS-II diffractometer
ω scans
Absorption correction: numerical (*X-SHAPE*; Stoe & Cie, 2005)
T_{min} = 0.985, T_{max} = 0.991
12313 measured reflections
3450 independent reflections
2208 reflections with $I > 2\sigma(I)$
R_{int} = 0.058
θ_{max} = 26.8°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.070
$wR(F^2)$ = 0.133
S = 1.16
3450 reflections
276 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0376P)^2 + 0.466P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.13 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3B···O2^{i}	0.85 (3)	1.87 (4)	2.715 (4)	174.5 (3)
N1—H1D···O3	0.84 (3)	2.23 (3)	2.730 (3)	117 (2)

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

H atoms were located in a difference synthesis and refined isotropically [N—H = 0.84 (3) Å, U_{iso}(H) = 0.066 (9) Å^2; O—H = 0.85 (3) Å, U_{iso}(H) = 0.065 (9) Å^2; and C—H = 0.84 (4)–0.98 (4) Å, U_{iso}(H) = 0.052 (7)–0.20 (3) Å^2].

Data collection: *X-AREA* (Stoe & Cie, 2005); cell refinement: *X-AREA*; data reduction: *X-RED* (Stoe & Cie, 2005); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-STEP32* (Stoe & Cie, 2000); software used to prepare material for publication: *SHELXL97*.

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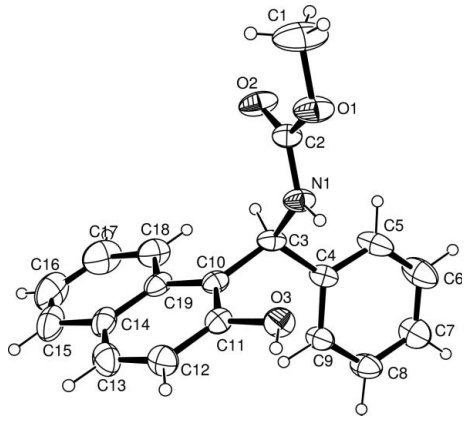

Figure 1
The molecular structure with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

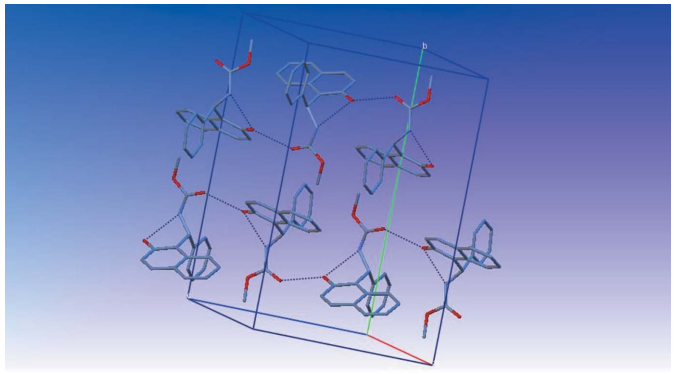

Figure 2
A packing diagram of (I). Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

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.
Batey, R. A., Santhakumar, V., Yoshina-Ishii, C. & Taylor, S. D. (1998). *Tetrahedron Lett.* **39**, 6267–6270.
Domling, A. & Ugi, I. (2000). *Angew. Chem. Int. Ed.* **39**, 3169–3210.
Heck, S. & Domling, A. (2000). *Synlett*, pp. 424–426.
Majer, P. & Randad, R. S. (1994). *J. Org. Chem.* **59**, 1937–1938.
Nowick, J. S., Powell, N. A., Nguyen, T. M. & Noronha, G. (1992). *J. Org. Chem.* **57**, 7364–7366.
Ozaki, S. (1972). *Chem. Rev.* **72**, 457–496.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Stoe & Cie (2000). *X-STEP32*. Version 1.07e. Stoe & Cie, Darmstadt, Germany.
Stoe & Cie (2005). *X-AREA* (Version 1.31), *X-RED* (Version 1.31) and *X-SHAPE* (Version 2.05). Stoe & Cie, Darmstadt, Germany.