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N,*N*-Dimethyl-2-(2-naphthyloxy)acetamide monohydrate

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Key indicators

Single-crystal X-ray study
T = 160 K
Mean σ(C–C) = 0.003 Å
H-atom completeness 89%
Disorder in main residue
R factor = 0.066
wR factor = 0.213
Data-to-parameter ratio = 16.5

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

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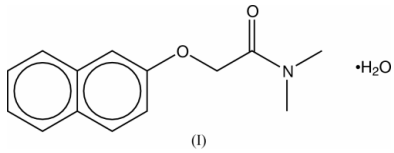
2-(2-Naphthyloxy)acetate derivatives. Part III.

The title compound, $C_{14}H_{15}NO_2\cdot H_2O$, is a potential anti-amnesic agent. One methyl group of the dimethyl-substituted N atom appears to be disordered over two sites. In the crystal structure, the water molecule is involved in intermolecular hydrogen bonds with two neighbouring water molecules, as well as with the O atom of the amide group. The water···water interactions form extended chains which run parallel to the *c* axis. Intermolecular C—H···O and C—H···π interactions are also observed.

Comment

The conformation of molecules with antiamnesic activity has attracted considerable interest (Amato *et al.*, 1991), and the structure determination presented here, namely that of *N*,*N*-dimethyl-2-(2-naphthyloxy)acetamide monohydrate, (I), is part of our research programme on biologically active 2-(2-naphthyloxy)acetate derivatives. Compound (I) is important in the treatment of human cognitive disorders (Thamotharan *et al.*, 2003, and references therein; Thamotharan *et al.*, 2003*a*,*b*).



(I)

Fig. 1 shows the asymmetric unit of (I) with the atomic numbering scheme. One methyl group of the dimethyl-substituted N atom appears to be disordered over two sites with the major conformation occurring in approximately 67% of the molecules. The bond lengths and angles in (I) are comparable with those in the related structures *N*-(2-naphthyloxymethylcarbonyl)piperidine and 3-methyl-*N*-(2-naphthyloxymethylcarbonyl)piperidine (Thamotharan *et al.*, 2003), as well as 4-(2-naphthyloxymethylcarbonyl)morpholine and 1-(2-naphthyloxymethylcarbonyl)-4-methylpiperazine (Thamotharan *et al.*, 2003*a*). In (I), the C2—O11—C12—C13 [178.56 (16)°] and O11—C12—C13—N14 [179.19 (18)°] torsion angles show that the central unit has an antiperiplanar conformation. The exocyclic bond angle C1—C2—O11 [125.04 (18)°] deviates significantly from the normal value of 120°; this may be due to steric repulsion between the H atoms on C1 and C12 (H1···H121 = 2.29 Å and H1···H122 = 2.25 Å).

Although it was not possible to locate the H atoms of the water molecule, an analysis of the O···O distances still allows conclusions to be drawn concerning the hydrogen-bonding network. In the crystal structure, the water molecule is

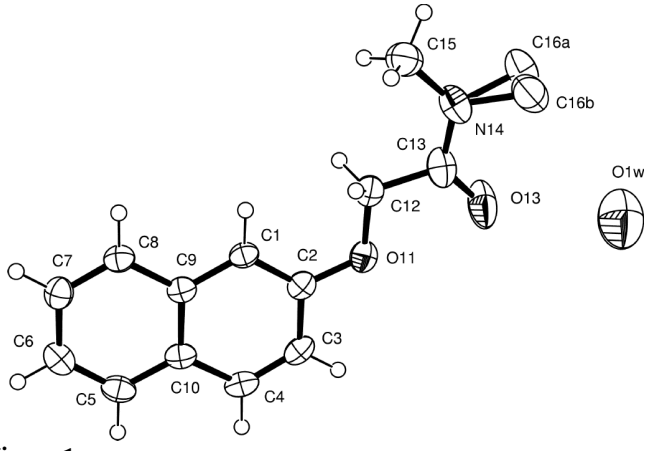


Figure 1
View of the molecule of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by circles of arbitrary radii and H atoms bonded to the disordered methyl group have been omitted for clarity.

involved in intermolecular hydrogen bonds with two neighbouring water molecules, as well as with amide atom O13. The water···water interactions form extended chains which run parallel to the *c* axis. The interactions are: O1*W*···O13 = 2.769 (3) Å, O1*W*···O1*W*^{iv} = 2.752 (4) Å and O1*W*···O1*W*^{v} = 2.740 (5) Å [symmetry codes: (iv) $-x, y, \frac{1}{2}-z$; (v) $-x, 1-y, 1-z$]. In addition, atom C15 has a weak intermolecular C—H···O interaction with atom O13 of an adjacent molecule (Table 1). This interaction links the molecules into chains, which run parallel to the *c* axis and have a graph-set motif of *C*(5) (Bernstein *et al.*, 1995). The disordered methyl atom C16*A* is involved in an intermolecular C—H···O interaction with each of two neighbouring water molecules, while the alternative position for this methyl group (C16*B*) has a similar interaction with just one water molecule. Atoms C8 (*via* H8) and C4 (*via* H4) act as donors for weak intermolecular C—H···π interactions. The former interaction is with the centroid (*Cg*1) of the amide-substituted six-membered ring of the naphthyl moiety in the molecule at $(x, -y, \frac{1}{2}+z)$, while the latter is with the centroid (*Cg*2) of the unsubstituted six-membered ring of the naphthyl moiety in the molecule at $(x, 1-y, z-\frac{1}{2})$ (Table 1).

Experimental

Methyl 2-(2-naphthyloxy)acetate (0.5 g) was reacted with *N,N*-dimethylamine. The resulting oily product obtained was treated with water. The precipitate which formed was filtered off, dried and crystallized from acetone to afford crystals of (I) (0.245 g, 46.21%, m.p. 345–349 K).

Crystal data

$C_{14}H_{15}NO_2\cdot H_2O$
M_r = 247.29
Monoclinic, *C*2/*c*
a = 51.8464 (6) Å
b = 5.9134 (1) Å
c = 8.3112 (1) Å
β = 97.4569 (5)°
V = 2526.57 (6) Å^3
Z = 8
D_x = 1.300 Mg m^{-3}
Mo Kα radiation
Cell parameters from 3163 reflections
θ = 2.0–27.5°
μ = 0.09 mm^{-1}
T = 160 (2) K
Plate, colourless
0.25 × 0.23 × 0.05 mm

Data collection

Nonius KappaCCD diffractometer
φ and ω scans with κ offsets
26 182 measured reflections
2900 independent reflections
2033 reflections with $I > 2\sigma(I)$
R_{int} = 0.056
θ_{max} = 27.5°
$h = 0 \rightarrow 66$
$k = 0 \rightarrow 7$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.066
$wR(F^2)$ = 0.213
S = 1.07
2900 reflections
176 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1228P)^2 + 1.8212P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.51 e Å^{-3}

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

*Cg*1 is the centroid of the amide-substituted six-membered ring and *Cg*2 is the centroid of the unsubstituted six-membered ring of the naphthyl moiety.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C15—H153···O13^{i}	0.98	2.49	3.302 (3)	140
C16*A*—H161···O1*W*^{i}	0.98	2.57	3.414 (12)	144
C16*A*—H162···O1*W*^{ii}	0.98	2.43	3.283 (12)	145
C4—H4···*Cg*2^{iii}	0.95	2.75	3.519 (2)	139
C8—H8···*Cg*1^{i}	0.95	2.70	3.440 (2)	136

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

The available crystals of the title compound were thin plates of poor quality; this has impacted on the quality of the results. The asymmetric unit contains one molecule of the amide plus one water molecule. One methyl group of the dimethyl-substituted N atom appears to be disordered over two sites, although this could be an artefact of the data quality. Two positions were defined for this group and constrained refinement of the site-occupation factors led to a value of 0.67 (3) for the major conformation. The disordered and ordered N—C(methyl) bond lengths were restrained to be similar, as were the anisotropic displacement parameters of the disordered C atoms. The H atoms of the water molecule could not be located and were omitted from the model. The methyl H atoms were constrained to an ideal geometry (C—H = 0.98 Å) with U_{iso}(H) = 1.5U_{eq}(C), but were allowed to rotate freely about the C—C bonds. All remaining H atoms were placed in geometrically idealized positions (C—H = 0.95–0.99 Å) and constrained to ride on their parent atoms with U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Version 1.07; Farrugia, 1997); software used to prepare material for publication: *SHELXL*97 and *PLATON* (Spek, 2003).

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