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Absolute configuration of *N*-[(*R*)-3-methyl-2-butyl]-(*S*)-2-(6-methoxy-2-naphthyl)propionamide

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.053
wR factor = 0.149
Data-to-parameter ratio = 13.2

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

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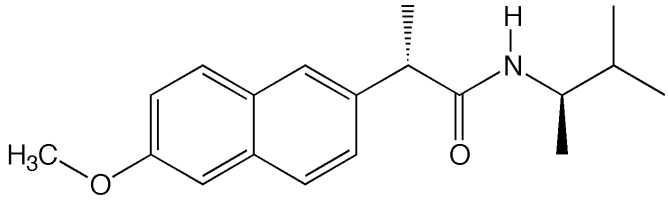
The title compound, $C_{19}H_{25}NO_2$, was made in order to confirm the absolute configuration of (*R*)-3-methyl-2-butylamine obtained from commercially available (*R*/*S*)-3-methyl-2-butylamine (racemate) after fractional crystallization of its ammonium salt with the optically pure carboxylic acid derivative, (*S*)-naproxene.

Comment

(*R*)-3-Methyl-2-butylamine reacted with succinimidyl (*S*)-(6-methoxy-2-naphthyl)propionate to form the corresponding amide, *i.e.* the title compound, (I). The Flack (1983) parameter −0.3 (12) calculated after the crystal structure refinement does not allow us to determine reliably the absolute configuration. Nevertheless, as the product was prepared from (*S*)-naproxene, the C12*A* and C12*B* atoms have an *S* configuration and the chirality of C15*A* and C15*B*, deduced from the torsional angles, is *R*. The stacking of the molecules is the result of van der Waals contacts, and of N1*A*—H···O2*B* and N1*B*—H···O2*A*^{i} [symmetry code: (i) 1 + *x*, *y*, 1 + *z*] hydrogen bonds. Molecules *A* and *B* form infinite *ABABAB*... chains in a direction parallel to the diagonal of the *a* and *c* axes.



(I)

Experimental

The preparation of the title compound is described by Khelili *et al.* (1999). Crystals were obtained by slow evaporation of a methanol solution.

Crystal data

$C_{19}H_{25}NO_2$
M_r = 299.40
Monoclinic, $P2_1$
a = 6.879 (2) Å
b = 29.563 (9) Å
c = 8.511 (3) Å
β = 99.11 (2)°
V = 1709.0 (9) Å^3
Z = 4
D_x = 1.164 Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 133 reflections
θ = 2.5–24.5°
μ = 0.08 mm^{-1}
T = 293 (2) K
Prism, colourless
0.34 × 0.23 × 0.23 mm

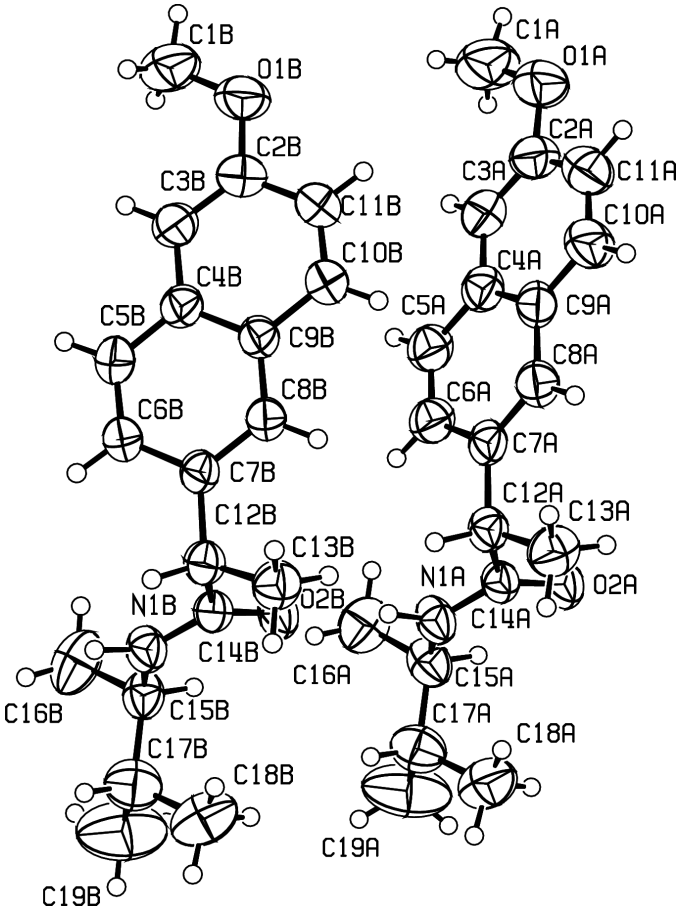


Figure 1
The molecular structure with atom-labelling schemes of molecules *A* and *B*. Displacement ellipsoids are shown at 50% probability levels and H atoms are drawn as small circles of arbitrary radii.

Table 1
Selected geometric parameters (Å, °).

C1*A*—O1*A*	1.414 (4)	C1*B*—O1*B*	1.418 (3)
C2*A*—O1*A*	1.380 (3)	C2*B*—O1B	1.369 (3)
C14*A*—O2*A*	1.228 (3)	C14*B*—O2*B*	1.229 (3)
C14*A*—N1*A*	1.332 (3)	C14*B*—N1*B*	1.337 (3)
C15*A*—N1*A*	1.475 (3)	C15*B*—N1*B*	1.463 (3)
O2*A*—C14*A*—N1*A*	124.4 (2)	O2*B*—C14*B*—N1*B*	123.3 (2)
O2*A*—C14*A*—C12*A*	121.2 (2)	O2*B*—C14*B*—C12*B*	120.9 (2)
N1*A*—C14*A*—C12*A*	114.3 (2)	N1*B*—C14*B*—C12*B*	115.8 (2)
C2*A*—O1*A*—C1*A*	118.0 (3)	C2*B*—O1*B*—C1*B*	117.2 (2)
C7*A*—C12*A*—C14*A*—O2*A*	71.6 (2)	C7*B*—C12*B*—C14*B*—O2*B*	75.6 (2)
C7*A*—C12*A*—C14*A*—N1*A*	−106.6 (2)	C7*B*—C12*B*—C14*B*—N1*B*	−103.6 (2)
C12*A*—C14*A*—N1*A*—C15*A*	171.3 (2)	C12*B*—C14*B*—N1*B*—C15*B*	178.7 (2)
C17*A*—C15*A*—N1*A*—C14*A*	114.6 (3)	C16*B*—C15*B*—N1*B*—C14*B*	−125.4 (3)
C16*A*—C15*A*—N1*A*—C14*A*	−120.5 (3)	C17*B*—C15*B*—N1*B*—C14*B*	111.4 (3)
C3*A*—C2*A*—O1*A*—C1*A*	5.5 (5)	C3*B*—C2*B*—O1*B*—C1*B*	−4.0 (4)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1*A*—H25*A*···O2*B*	0.97 (3)	1.96 (3)	2.911 (3)	166 (2)
N1*B*—H25*B*···O2*A*^{i}	0.98 (3)	2.09 (3)	3.051 (3)	166 (2)

Symmetry code: (i) $1+x, y, 1+z$.

Data collection

MAR345 image-plate diffractometer
222 images, 150 mm, ΔΦ 2° scans
19 313 measured reflections
5448 independent reflections
4896 reflections with $I > 2\sigma(I)$

$R_{int} = 0.056$
$\theta_{max} = 24.4°$
$h = -7 \rightarrow 7$
$k = -34 \rightarrow 34$
$l = -9 \rightarrow 9$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.150$
$S = 1.06$
5448 reflections
414 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.1033P)^2 + 0.0697P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.171 (12)
Absolute structure: Flack (1983); 1311 Friedel pairs
Flack parameter = −0.3 (12)

H atoms were constrained (included as riding atoms) except for HN1*A* and HN1*B* which were refined, with isotropic displacement parameters fixed at $1.2U_{eq}$ of the parent atom ($1.5U_{eq}$ for methyl atoms).

Data collection: MAR software; cell refinement: *marHKL* (Klein & Bartels, 2000); data reduction: *marHKL* (Klein & Bartels, 2000); 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: *SHELXL*97.

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