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Key indicators

Single-crystal X-ray study

$T = 293\text{ K}$

Mean $\sigma(\text{C}-\text{C}) = 0.004\text{ Å}$

R factor = 0.053

wR factor = 0.149

Data-to-parameter ratio = 13.2

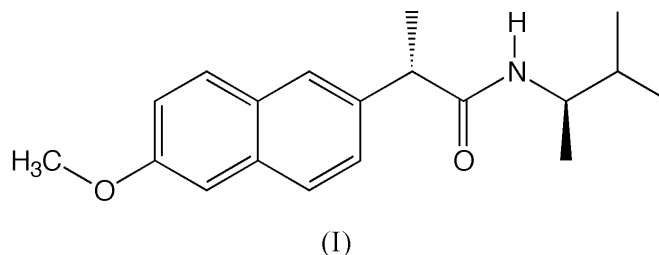
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

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

The title compound, $\text{C}_{19}\text{H}_{25}\text{NO}_2$, 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 $\text{N1A}-\text{H}\cdots\text{O2B}$ and $\text{N1B}-\text{H}\cdots\text{O2A}^i$ [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.



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

$\text{C}_{19}\text{H}_{25}\text{NO}_2$
 $M_r = 299.40$
Monoclinic, $P2_1$
 $a = 6.879(2)\text{ Å}$
 $b = 29.563(9)\text{ Å}$
 $c = 8.511(3)\text{ Å}$
 $\beta = 99.11(2)^\circ$
 $V = 1709.0(9)\text{ Å}^3$
 $Z = 4$

$D_x = 1.164\text{ Mg m}^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 133 reflections
 $\theta = 2.5\text{--}24.5^\circ$
 $\mu = 0.08\text{ mm}^{-1}$
 $T = 293(2)\text{ K}$
Prism, colourless
 $0.34 \times 0.23 \times 0.23\text{ mm}$

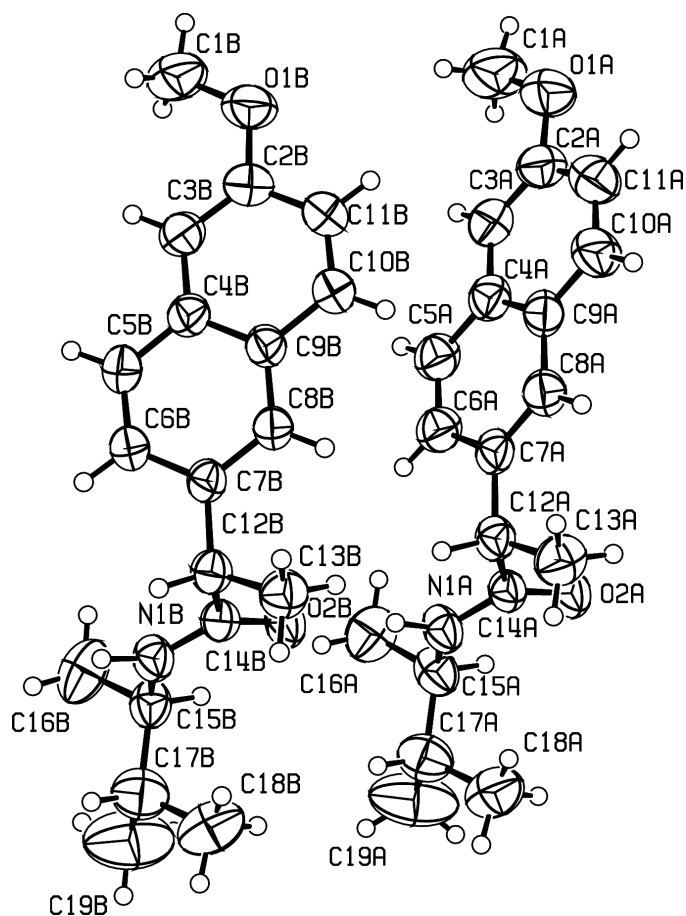


Figure 1

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.

Data collection

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

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

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 $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.23 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.20 \text{ e } \text{\AA}^{-3}$
Extinction correction: *SHELXL97*
Extinction coefficient: 0.171 (12)
Absolute structure: Flack (1983);
1311 Friedel pairs
Flack parameter = -0.3 (12)

Table 1

Selected geometric parameters ($\text{\AA}$, $^\circ$).

C1A—O1A	1.414 (4)	C1B—O1B	1.418 (3)
C2A—O1A	1.380 (3)	C2B—O1B	1.369 (3)
C14A—O2A	1.228 (3)	C14B—O2B	1.229 (3)
C14A—N1A	1.332 (3)	C14B—N1B	1.337 (3)
C15A—N1A	1.475 (3)	C15B—N1B	1.463 (3)
O2A—C14A—N1A	124.4 (2)	O2B—C14B—N1B	123.3 (2)
O2A—C14A—C12A	121.2 (2)	O2B—C14B—C12B	120.9 (2)
N1A—C14A—C12A	114.3 (2)	N1B—C14B—C12B	115.8 (2)
C2A—O1A—C1A	118.0 (3)	C2B—O1B—C1B	117.2 (2)
C7A—C12A—C14A—O2A	71.6 (2)	C7B—C12B—C14B—O2B	75.6 (2)
C7A—C12A—C14A—N1A	−106.6 (2)	C7B—C12B—C14B—N1B	−103.6 (2)
C12A—C14A—N1A—C15A	171.3 (2)	C12B—C14B—N1B—C15B	178.7 (2)
C17A—C15A—N1A—C14A	114.6 (3)	C16B—C15B—N1B—C14B	−125.4 (3)
C16A—C15A—N1A—C14A	−120.5 (3)	C17B—C15B—N1B—C14B	111.4 (3)
C3A—C2A—O1A—C1A	5.5 (5)	C3B—C2B—O1B—C1B	−4.0 (4)

Table 2

Hydrogen-bonding geometry ($\text{\AA}$, $^\circ$).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1A—H25A...O2B	0.97 (3)	1.96 (3)	2.911 (3)	166 (2)
N1B—H25B...O2A ⁱ	0.98 (3)	2.09 (3)	3.051 (3)	166 (2)

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

H atoms were constrained (included as riding atoms) except for HN1A and HN1B which were refined, with isotropic displacement parameters fixed at $1.2U_{\text{eq}}$ of the parent atom ($1.5U_{\text{eq}}$ 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: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP*III (Burnett & Johnson, 1996); software used to prepare material for publication: *SHELXL97*.

References

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