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tert-Butyl (*S*)-*N*-[(*S*)-1-(oxiranyl)ethyl]carbamate

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The investigation of the title compound, $C_9H_{17}NO_3$, which has two independent but virtually identical molecules in the asymmetric unit, allowed the determination of the relative configurations of the chiral atoms as *S*.

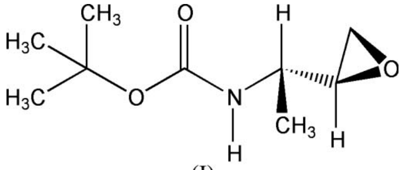
Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.008 Å
R factor = 0.055
wR factor = 0.128
Data-to-parameter ratio = 10.7

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

Comment

The title compound, (I) [alternative name 3-(*tert*-butoxycarboxamido)-1,2-epoxybutane], was prepared as a model for the synthesis of the *syn* and *anti* stereoisomers of *N*-Boc-statine (Yuste *et al.*, 2003). It is also one of the diastereoisomeric products obtained following the synthetic sequence involving reduction of the parent sulfoxide, methylation of the sulfenyl group and intramolecular nucleophilic substitution (S_Ni) of the sulfonium salt by the alkoxide generated by treatment with base (Solladié *et al.*, 1985).



(I)

Compound (I) crystallizes with two chemically identical but crystallographically independent molecules (*A* and *B*) (Fig. 1). The main difference between *A* and *B* is found in the torsion angles around the N1—C5 peptide bond, which displays an *s*–*cis* conformation (Table 1). The sum of the angles around N1 is close to 360° [354° for molecule *A* and 359° for molecule *B*], suggesting a considerable degree of sp^2 hybridization for this atom. The methyl and oxiranyl substituents at atom C3 are oriented in such a way that the 1,3-interactions are minimized.

In the crystal structure, molecules are arranged so that the amide group of each independent molecule alternates to allow for the formation of a rather long hydrogen bond with the carbonyl O atom, as detailed in Table 2. As illustrated in Fig. 2, this gives rise to a two-stranded antiparallel sheet.

Experimental

To a stirred solution of ($2S,3S,R_S$)-*N*-(*tert*-butoxycarbonyl)-3-amino-1-[(4-methylphenyl)sulfinyl]-2-butanol (Yuste *et al.*, 2000) (0.67 g, 2.05 mmol, 1 equivalent) in EtOH (6 ml), a 15% wt solution of $TiCl_3$ (2 equivalents) in 20–30% aqueous HCl was added at room temperature under an argon atmosphere. After 45 min, the solution was treated with water (20 ml) and extracted with CH_2Cl_2 (2 × 20 ml). The organic phase was washed with brine, dried and evaporated. The product was purified by column chromatography (hexane–ethyl acetate, 7:3 *v*/*v*) to give (2*S*,3*S*)-*N*-(*tert*-butoxy-

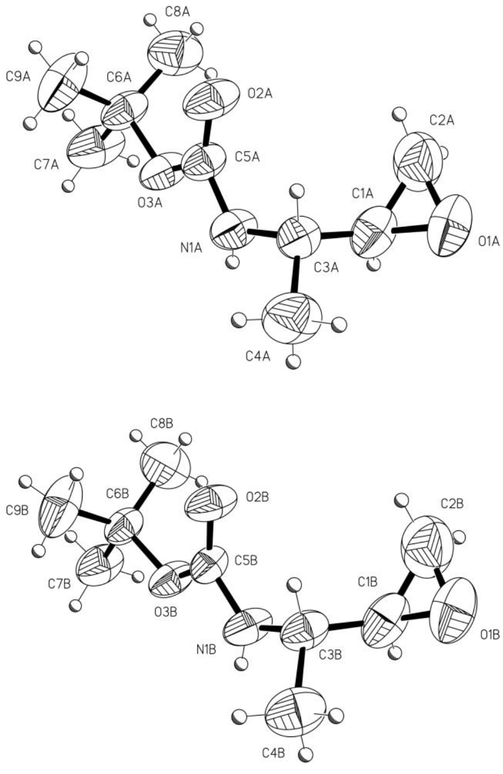


Figure 1
View of the asymmetric unit of (I), showing the atom-labelling scheme and displacement ellipsoids drawn at the 50% probability level.

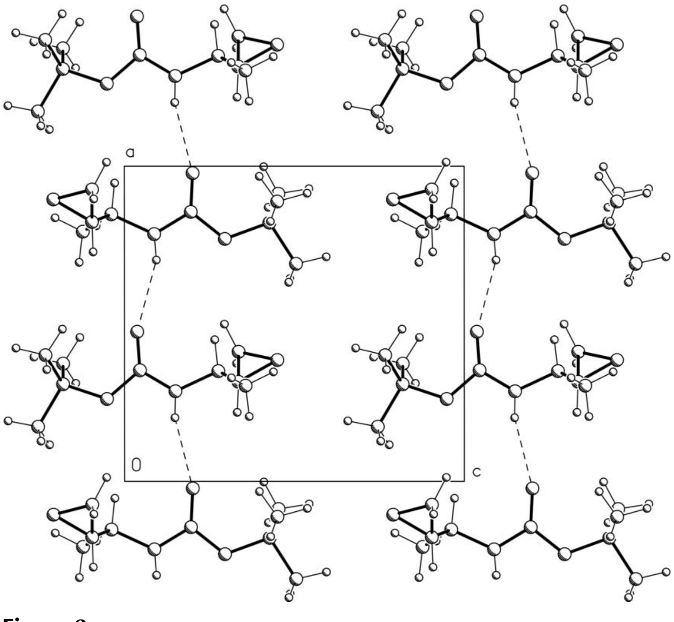


Figure 2
The molecular packing in (I), viewed along the *b* axis. Dashed lines indicate the hydrogen-bonding interactions.

carbonyl)-3-amino-1-[(4-methylphenyl)sulfenyl]-2-butanol in 65% yield as white crystals [CH_2Cl_2–hexane; m.p. 351–353 K; $[\alpha]_D$ +11.4 (*c* 1.0, $CHCl_3$)]. To a solution of the sulfenyl derivative (0.24 g, 0.77 mmol, 1 equivalent) in dry CH_2Cl_2 (3 ml) trimethyloxonium tetrafluoroborate (0.128 g, 0.85 mmol, 1 equivalent) was added at room temperature under an argon atmosphere. After 20 h, a solution of K_2CO_3 (1.54 mmol) in water (6 ml) was added and the resulting mixture was vigorously stirred for 51 h. The organic phase was separated and the aqueous phase was extracted with CH_2Cl_2 (3 × 5 ml). The combined organic layers were washed with brine, dried and evaporated. The residue was purified by column chromatography (hexane–ethyl acetate, 7:3 *v*/*v*) to give (*S*)-1-[(*S*)-1-(*tert*-butoxycarbonylamino)ethyl]oxirane (hexane–ethyl acetate, 7:3 *v*/*v*) in 45% yield as colourless crystals [CH_2Cl_2–pentane; m.p. 321–323 K; $[\alpha]_D$ −15.0 (*c* 1.0, $CHCl_3$)].

Crystal data

$C_9H_{17}NO_3$
$M_r = 187.24$
Monoclinic, $P2_1$
$a = 9.741$ (2) Å
$b = 10.882$ (2) Å
$c = 10.492$ (1) Å
$\beta = 90.04$ (1)°
$V = 1112.2$ (3) Å^3
$Z = 4$
$D_x = 1.118$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 37 reflections
$\theta = 5.0$–12.5°
$\mu = 0.08$ mm^{-1}
$T = 294$ (2) K
Block, colourless
0.50 × 0.40 × 0.34 mm

Data collection

Siemens *P*4/PC diffractometer
ω–2θ scans
Absorption correction: ψ scan (*XPREP* in *SHELXTL*; Sheldrick, 2000)
$T_{\min} = 0.955$, $T_{\max} = 0.971$
2835 measured reflections
2685 independent reflections
1465 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.027$
$\theta_{\max} = 27.5$°
$h = 0 \rightarrow 12$
$k = 0 \rightarrow 14$
$l = -13 \rightarrow 13$
3 standard reflections every 97 reflections intensity decay: 2.6%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.128$
$S = 1.02$
2685 reflections
250 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0453P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.13$ e Å^{-3}
$\Delta\rho_{\min} = -0.12$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.063 (7)

Table 1
Selected geometric parameters (Å, °).

O1*A*—C1*A*	1.420 (6)	O1*B*—C1*B*	1.413 (6)
O1*A*—C2*A*	1.427 (7)	O1*B*—C2*B*	1.439 (8)
O2*A*—C5*A*	1.211 (4)	O2*B*—C5*B*	1.212 (4)
O3*A*—C5*A*	1.355 (4)	O3*B*—C5*B*	1.342 (4)
O3*A*—C6*A*	1.481 (5)	O3*B*—C6*B*	1.470 (4)
N1*A*—C3*A*	1.464 (5)	N1*B*—C3*B*	1.460 (4)
N1*A*—C5*A*	1.336 (5)	N1*B*—C5*B*	1.334 (5)
C1*A*—C2*A*	1.427 (8)	C1*B*—C2*B*	1.436 (9)
C1*A*—C3*A*	1.495 (7)	C1*B*—C3*B*	1.499 (8)
C3*A*—C4*A*	1.511 (7)	C3*B*—C4*B*	1.527 (7)
C6*A*—C7*A*	1.523 (6)	C6*B*—C7*B*	1.516 (5)
C6*A*—C8*A*	1.507 (8)	C6*B*—C8*B*	1.488 (8)
C6*A*—C9*A*	1.489 (7)	C6*B*—C9*B*	1.496 (8)
C3*A*—N1*A*—C5*A*	121.8 (4)	C3*B*—N1*B*—C5*B*	123.2 (3)
C2*A*—C1*A*—C3*A*—N1*A*	99.8 (6)	C2*B*—C1*B*—C3*B*—N1*B*	100.6 (6)
C2*A*—C1*A*—C3*A*—C4*A*	−137.5 (5)	C2*B*—C1*B*—C3*B*—C4*B*	−136.7 (6)
C3*A*—N1*A*—C5*A*—O2*A*	−14.4 (9)	C3*B*—N1*B*—C5*B*—O2*B*	−3.0 (9)
C3*A*—N1*A*—C5*A*—O3*A*	166.7 (4)	C3*B*—N1*B*—C5*B*—O3*B*	176.4 (5)

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

$D—H\cdots A$	$D—H$	$H\cdots A$	$D\cdots A$	$D—H\cdots A$
N1*B*—H1*B*···O2*A*	0.81 (3)	2.29 (4)	3.065 (4)	158 (5)
N1*A*—H1*A*···O2*B*^{i}	0.82 (3)	2.25 (4)	3.051 (4)	164 (4)

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

The methyl H atoms were constrained to an ideal geometry, with C—H distances of 0.98 Å and $U_{iso}(H) = 1.5U_{eq}(C)$, but each group was allowed to rotate freely about its C—C bond. The positions of the amide H atoms were refined freely, giving distances as reported in Table 2. All other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with methyl C—H = 0.96 Å, methylene C—H = 0.97 Å and methine C—H = 0.98 Å, and with $U_{iso}(H) = 1.2U_{eq}(C)$ for methylene and methine or $1.5U_{eq}(C)$ for methyl H atoms. In the absence of significant anomalous dispersion effects, Friedel pairs were averaged. The absolute configuration of atom C3 was determined on the basis of the synthesis.

Data collection: *XSCANS* (Siemens, 1993); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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