Acta Cryst. (1998). C**54**, IUC9800033 [**doi:10.1107/S010827019809951X**]

(2*S*,3*R*,4*R*,5*R*)-2-Benzyl-*N*-(*tert*-butoxycarbonyl)-3-hydroxy-4,5-dimethylpyrrolidine at 133 K

J. W. Bats, T. Heinrich and M. Reggelin

Abstract

The structure determination of the title compound $C_{18}H_{27}NO_3$ was undertaken to establish the relative configuration of the chiral centers. The pyrrolidine ring has a twist conformation. The crystal packing is stabilized by intermolecular hydrogen bonding.

Comment

The synthesis of the title compound and a number of isomers has been reported by Reggelin and Heinrich (1998). A structure determination was undertaken to confirm the expected configurations of the chiral centers. The pyrrolidine ring has a twist conformation with a pseudo-twofold axis passing through N and the midpoint of the C2—C3 bond. The ring puckering parameters defined by Cremer and Pople (1975) are q = 0.393 Å and φ = 267 °. A stereoisomer of the title compound with (2*S*,3S,4*S*,5S)- configuration reported by Bolte (1997) has an inverted ring conformation (φ = 84°). The hydroxyl group is in a pseudo-axial position, methyl group C12 is in a pseudo-equatorial position with respect the the five- membered ring. The N atom shows a small deviation from planarity (sum of bond angles about the N atom: 357.3°). The hydroxyl group is involved in an intermolecular hydrogen bond to atom O2 of a symmetry-related molecule (symmetry code: $-x$, $y - 1/2$, $1.5 - z$). The dimensions of the hydrogen bond are: O1 - H01: 0.83 (2) Å, H01 ··· O2: 1.94 (2) Å, O1 ··· O2: 2.763 (1) Å and angle O1 - H01 - O2: 169 (1) °. The hydrogen bonds connect the molecules to zigzag chains in the crystallographic b - direction. There are no other significant inter- molecular contacts.

Experimental

Crystals were grown from a solution of the title compound in ether/ n-hexane.

Computing details

Data collection: *SMART* (Siemens, 1995); cell refinement: *SMART* (Siemens, 1995); data reduction: *SAINT* (Siemens, 1995); program(s) used to solve structure: Direct methods (*SHELXS*, Sheldrick, 1996); program(s) used to refine structure: *LSFM MolEN* (Fair, 1990); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 1996); software used to prepare material for publication: *BTABLE PTABLE* CIF in *MolEN* (Fair, 1990).

(2*S*,3*R*,4*R*,5*R*)-2-benzyl-*N*-(*tert*-butyloxycarbonyl)-3-hydroxy- 4,5-dimethyl-pyrrolidine

Crystal data

$C_{18}H_{27}NO_3$ $V = 1761.6$ (4) Å^3

CIF access

M_r = 305.42	Z = 4
Orthorhombic, $P2_12_12_1$	Mo- $K\alpha$
a = 11.2771 (10) Å	μ = 0.07 mm^{-1}
b = 11.3895 (11) Å	T = 133 K
c = 13.715 (2) Å	0.65 × 0.46 × 0.30 mm

Data collection

SIEMENS SMART diffractometer	2672 independent reflections
Absorption correction: none	2663 reflections with $I > 0.0\sigma(I)$
26770 measured reflections	R_{int} = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033	$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$wR(F^2)$ = 0.037	$\Delta\rho_{min}$ = −0.12 e Å^{-3}
2663 reflections	Absolute structure: The absolute configuration of the compound has been established by the known 2S-configuration.
308 parameters	Flack parameter: −0.1 (6)
H atoms refined isotropically	

References

+qa0017+Bolte, M. (1997). *Acta Cryst.* C53 IUC9700028.

Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.

Fair, C. K. (1990). *MolEN*, An Interactive Intelligent System for Crystal Structure Analysis, User Manual, Enraf - Nonius, Delft, The Netherlands.

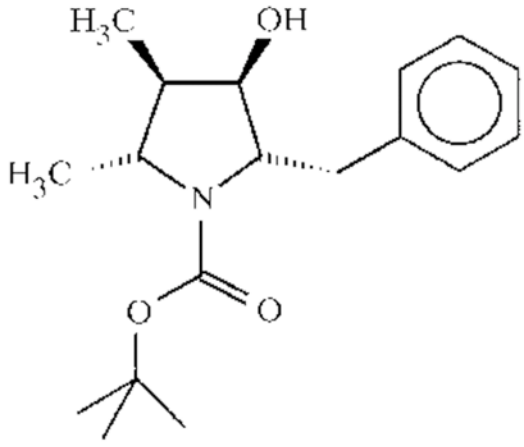
Reggelin, M. & Heinrich, T. (1998). *Angew. Chem.* in preparation.

Sheldrick, G. M. (1996). *SHELXTL*. Structure Determination Software Programs. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Siemens (1995). *SMART* and *SAINT*. Area-Detector Control and Integration Software. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Zachariasen, W. H. (1967). *Acta Cryst.* **23**, 558–564.

Scheme 1



supplementary materials

(2*S*,3*R*,4*R*,5*R*)-2-benzyl-*N*-(*tert*-butyloxycarbonyl)-3-hydroxy- 4,5-dimethyl-pyrrolidine

Crystal data

$C_{18}H_{27}NO_3$	D_x = 1.152 Mg m^{-3}
M_r = 305.42	Mo- $K\alpha$ radiation λ = 0.71073 Å
Orthorhombic, $P2_12_12_1$	Cell parameters from 333 reflections
a = 11.2771 (10) Å	θ = 3–23º
b = 11.3895 (11) Å	μ = 0.07 mm^{-1}
c = 13.715 (2) Å	T = 133 K
V = 1761.6 (4) Å^3	Block, colorless
Z = 4	0.65 × 0.46 × 0.30 mm
F_{000} = 664	

Data collection

SIEMENS SMART diffractometer	R_{int} = 0.020
ω scans	θ_{max} = 29.9º
Absorption correction: none	h = −14→15
26770 measured reflections	k = −15→15
2672 independent reflections	l = −18→18
2663 reflections with $I > 0.0\sigma(I)$	

Refinement

Refinement on F	$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$R[F^2 > 2\sigma(F^2)]$ = 0.033	$\Delta\rho_{min}$ = −0.12 e Å^{-3}
$wR(F^2)$ = 0.037	Extinction correction: isotropic (Zachariasen, 1967)
2663 reflections	Extinction coefficient: 0.0000017 (5)
308 parameters	Absolute structure: The absolute configuration of the compound has been established by the known 2S-configuration.
H atoms refined isotropically	Flack parameter: −0.1 (6)
$4F_o^2/(\sigma^2(F_o^2) + 0.0009\,F_o^4)$?	

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
O1	−0.20087 (8)	0.54522 (7)	0.75808 (7)	0.0250 (4)
O2	0.08698 (8)	0.83851 (7)	0.79032 (7)	0.0250 (4)
O3	0.16754 (7)	0.65792 (8)	0.82047 (7)	0.0236 (4)
N	−0.02055 (8)	0.69245 (8)	0.85899 (8)	0.0186 (4)
C1	−0.0394 (1)	0.56532 (9)	0.87478 (9)	0.0174 (5)
C2	−0.1736 (1)	0.5531 (1)	0.85918 (9)	0.0191 (5)
C3	−0.2225 (1)	0.6698 (1)	0.89675 (9)	0.0199 (5)

C4	−0.1323 (1)	0.7602 (1)	0.85962 (9)	0.0185 (5)
C5	0.0031 (1)	0.5271 (1)	0.97603 (9)	0.0209 (5)
C6	−0.0004 (1)	0.3955 (1)	0.99051 (9)	0.0200 (5)
C7	0.0702 (1)	0.3224 (1)	0.93349 (9)	0.0253 (6)
C8	0.0701 (1)	0.2018 (1)	0.9474 (1)	0.0322 (6)
C9	0.0003 (1)	0.1529 (1)	1.0191 (1)	0.0327 (6)
C10	−0.0709 (1)	0.2239 (1)	1.0759 (1)	0.0326 (6)
C11	−0.0721 (1)	0.3447 (1)	1.06167 (9)	0.0254 (6)
C12	−0.3499 (1)	0.6957 (1)	0.8669 (1)	0.0274 (6)
C13	−0.1253 (1)	0.8704 (1)	0.9224 (1)	0.0265 (6)
C14	0.0789 (1)	0.7377 (1)	0.82004 (9)	0.0199 (5)
C15	0.2748 (1)	0.6747 (1)	0.76002 (9)	0.0202 (5)
C16	0.3377 (1)	0.5574 (1)	0.7730 (1)	0.0280 (6)
C17	0.3498 (1)	0.7746 (1)	0.7994 (1)	0.0262 (6)
C18	0.2399 (1)	0.6933 (1)	0.6542 (1)	0.0285 (6)
H1	0.002 (1)	0.520 (1)	0.824 (1)	0.019 (3)*
H01	−0.167 (1)	0.486 (1)	0.736 (1)	0.042 (4)*
H2	−0.206 (1)	0.488 (1)	0.8955 (9)	0.021 (3)*
H3	−0.216 (1)	0.671 (1)	0.9694 (9)	0.015 (3)*
H4	−0.152 (1)	0.782 (1)	0.7949 (9)	0.022 (3)*
H5B	0.083 (1)	0.552 (1)	0.981 (1)	0.024 (4)*
H5A	−0.046 (1)	0.562 (1)	1.026 (1)	0.029 (4)*
H7	0.118 (1)	0.355 (1)	0.887 (1)	0.031 (4)*
H8	0.123 (1)	0.155 (1)	0.908 (1)	0.049 (5)*
H9	−0.001 (2)	0.067 (1)	1.028 (1)	0.053 (5)*
H10	−0.123 (1)	0.195 (1)	1.126 (1)	0.038 (4)*
H11	−0.120 (1)	0.395 (1)	1.102 (1)	0.033 (4)*
H12A	−0.357 (1)	0.695 (1)	0.797 (1)	0.040 (5)*
H12C	−0.405 (1)	0.638 (1)	0.895 (1)	0.054 (5)*
H12B	−0.378 (1)	0.771 (1)	0.894 (1)	0.037 (4)*
H13C	−0.070 (1)	0.935 (1)	0.893 (1)	0.039 (4)*
H13B	−0.094 (1)	0.850 (1)	0.989 (1)	0.035 (4)*
H13A	−0.206 (1)	0.907 (1)	0.927 (1)	0.047 (5)*
H16B	0.287 (1)	0.496 (1)	0.747 (1)	0.031 (4)*
H16A	0.410 (1)	0.555 (1)	0.735 (1)	0.044 (5)*
H16C	0.355 (1)	0.541 (1)	0.842 (1)	0.028 (4)*
H17A	0.367 (1)	0.757 (1)	0.871 (1)	0.051 (5)*
H17B	0.309 (1)	0.849 (1)	0.790 (1)	0.037 (4)*
H17C	0.429 (1)	0.774 (1)	0.763 (1)	0.033 (4)*
H18B	0.188 (1)	0.632 (1)	0.629 (1)	0.045 (5)*
H18C	0.196 (1)	0.766 (1)	0.647 (1)	0.029 (4)*
H18A	0.310 (1)	0.698 (1)	0.613 (1)	0.041 (5)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0269 (4)	0.0221 (4)	0.0262 (4)	0.0053 (4)	−0.0068 (4)	−0.0066 (4)
O2	0.0222 (4)	0.0197 (4)	0.0359 (5)	0.0001 (4)	0.0038 (4)	0.0077 (4)

O3	0.0169 (4)	0.0232 (4)	0.0337 (4)	0.0022 (4)	0.0056 (4)	0.0095 (4)
N	0.0171 (4)	0.0143 (4)	0.0262 (5)	0.0008 (4)	0.0016 (4)	0.0027 (4)
C1	0.0171 (5)	0.0137 (4)	0.0223 (5)	−0.0005 (4)	0.0006 (4)	0.0013 (4)
C2	0.0178 (5)	0.0176 (5)	0.0224 (5)	−0.0010 (5)	−0.0001 (5)	0.0006 (5)
C3	0.0169 (5)	0.0205 (5)	0.0227 (5)	0.0004 (5)	0.0015 (4)	−0.0007 (5)
C4	0.0182 (5)	0.0166 (5)	0.0209 (5)	0.0026 (4)	0.0008 (4)	0.0006 (4)
C5	0.0220 (5)	0.0182 (5)	0.0227 (5)	−0.0006 (5)	−0.0015 (5)	0.0001 (5)
C6	0.0220 (5)	0.0186 (5)	0.0195 (5)	−0.0005 (5)	−0.0061 (5)	0.0016 (5)
C7	0.0283 (6)	0.0259 (6)	0.0220 (5)	0.0030 (6)	−0.0028 (5)	0.0008 (5)
C8	0.0422 (7)	0.0246 (6)	0.0322 (6)	0.0095 (6)	−0.0124 (6)	−0.0060 (5)
C9	0.0499 (8)	0.0184 (5)	0.0379 (7)	−0.0046 (6)	−0.0195 (6)	0.0038 (6)
C10	0.0395 (7)	0.0293 (6)	0.0301 (6)	−0.0122 (6)	−0.0077 (6)	0.0088 (6)
C11	0.0250 (5)	0.0269 (6)	0.0244 (6)	−0.0033 (5)	−0.0025 (5)	0.0023 (5)
C12	0.0174 (5)	0.0282 (6)	0.0420 (7)	0.0025 (5)	0.0006 (6)	−0.0053 (6)
C13	0.0296 (6)	0.0189 (5)	0.0334 (6)	0.0010 (5)	0.0035 (6)	−0.0044 (5)
C14	0.0180 (5)	0.0195 (5)	0.0225 (5)	−0.0004 (5)	0.0006 (5)	0.0023 (5)
C15	0.0147 (4)	0.0233 (5)	0.0242 (5)	0.0008 (5)	0.0019 (4)	0.0034 (5)
C16	0.0238 (5)	0.0275 (6)	0.0336 (6)	0.0063 (5)	0.0015 (6)	0.0016 (6)
C17	0.0197 (5)	0.0269 (6)	0.0340 (6)	−0.0010 (5)	−0.0018 (5)	0.0011 (6)
C18	0.0271 (6)	0.0329 (7)	0.0258 (6)	0.0022 (6)	−0.0015 (5)	0.0020 (6)

Geometric parameters (Å, °)

O1—C2	1.423 (2)	C4—C13	1.524 (2)
O2—C14	1.221 (1)	C5—C6	1.512 (2)
O3—C14	1.352 (1)	C6—C7	1.392 (2)
O3—C15	1.479 (1)	C6—C11	1.393 (2)
N—C1	1.479 (1)	C7—C8	1.387 (2)
N—C4	1.478 (1)	C8—C9	1.377 (2)
N—C14	1.345 (2)	C9—C10	1.381 (2)
C1—C2	1.535 (2)	C10—C11	1.389 (2)
C1—C5	1.532 (2)	C15—C16	1.523 (2)
C2—C3	1.529 (2)	C15—C17	1.517 (2)
C3—C4	1.535 (2)	C15—C18	1.519 (2)
C3—C12	1.523 (2)		
C14—O3—C15	121.03 (9)	C5—C6—C7	120.3 (1)
C1—N—C4	112.84 (9)	C5—C6—C11	121.3 (1)
C1—N—C14	123.60 (9)	C7—C6—C11	118.5 (1)
C4—N—C14	120.83 (9)	C6—C7—C8	121.0 (1)
N—C1—C2	102.14 (9)	C7—C8—C9	119.9 (1)
N—C1—C5	111.47 (9)	C8—C9—C10	119.9 (1)
C2—C1—C5	114.2 (1)	C9—C10—C11	120.4 (1)
O1—C2—C1	110.76 (9)	C6—C11—C10	120.3 (1)
O1—C2—C3	107.76 (9)	O2—C14—O3	125.3 (1)
C1—C2—C3	103.26 (9)	O2—C14—N	123.7 (1)
C2—C3—C4	103.46 (9)	O3—C14—N	110.9 (1)
C2—C3—C12	114.7 (1)	O3—C15—C16	101.66 (9)
C4—C3—C12	114.0 (1)	O3—C15—C17	110.7 (1)
N—C4—C3	102.51 (9)	O3—C15—C18	110.02 (9)

N—C4—C13	112.9 (1)	C16—C15—C17	110.8 (1)
C3—C4—C13	113.5 (1)	C16—C15—C18	110.7 (1)
C1—C5—C6	113.1 (1)	C17—C15—C18	112.4 (1)
C15—O3—C14—O2	−17.1 (2)	N—C1—C5—C6	−172.8 (1)
C15—O3—C14—N	164.2 (1)	C2—C1—C5—C6	72.1 (1)
C14—O3—C15—C16	−172.0 (1)	O1—C2—C3—C4	−76.9 (1)
C14—O3—C15—C17	70.1 (1)	O1—C2—C3—C12	47.8 (1)
C14—O3—C15—C18	−54.7 (1)	C1—C2—C3—C4	40.3 (1)
C4—N—C1—C2	14.1 (1)	C1—C2—C3—C12	165.1 (1)
C4—N—C1—C5	−108.2 (1)	C2—C3—C4—N	−30.9 (1)
C14—N—C1—C2	−147.3 (1)	C2—C3—C4—C13	−153.1 (1)
C14—N—C1—C5	90.4 (1)	C12—C3—C4—N	−156.2 (1)
C1—N—C4—C3	10.5 (1)	C12—C3—C4—C13	81.7 (1)
C1—N—C4—C13	133.0 (1)	C1—C5—C6—C7	63.2 (1)
C14—N—C4—C3	172.5 (1)	C1—C5—C6—C11	−117.9 (1)
C14—N—C4—C13	−65.0 (1)	C5—C6—C7—C8	178.5 (1)
C1—N—C14—O2	166.0 (1)	C11—C6—C7—C8	−0.4 (2)
C1—N—C14—O3	−15.3 (2)	C5—C6—C11—C10	−177.8 (1)
C4—N—C14—O2	6.0(12	C7—C6—C11—C10	1.1 (2)
C4—N—C14—O3	−175.3 (1)	C6—C7—C8—C9	−0.7 (2)
N—C1—C2—O1	82.2 (1)	C7—C8—C9—C10	1.1 (2)
N—C1—C2—C3	−33.0 (1)	C8—C9—C10—C11	−0.5 (2)
C5—C1—C2—O1	−157.4 (1)	C9—C10—C11—C6	−0.6 (2)
C5—C1—C2—C3	87.5 (1)		