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(1*S*,2*R*,4*R*,5*R*)-*tert*-Butyl *N*-(4-hydroxymethyl-6-oxabicyclo[3.1.0]hex-2-yl)carbamate

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Key indicators

Single-crystal X-ray study
T = 193 K
Mean σ(C–C) = 0.003 Å
R factor = 0.032
wR factor = 0.087
Data-to-parameter ratio = 9.1

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

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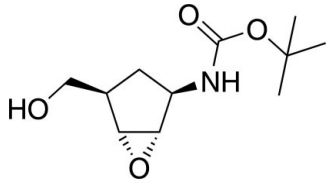
The title compound, $C_{11}H_{19}NO_4$, is a precursor for the preparation of (1*S*,2*R*,4*R*)-4-amino-2-(hydroxymethyl)cyclopentanol, which is an important carbocyclic analogue of β-2-deoxyribosylamine. The crystal packing of the title compound is stabilized by intermolecular O—H···O hydrogen bonds.

Comment

Carbocyclic analogues of 2′-deoxyribonucleotides [such as the antiviral compounds carbovir (Vince & Brownell, 1990) and 1592U89 (Daluge *et al.*, 1997)] are commonly used as drugs. Their therapeutic mode of action can be rationalized by the stabilized linkage between the sugar moiety and the heterocycle.

An easy synthesis of (1*S*,2*R*,4*R*)-4-amino-2-hydroxymethylcyclopentanol from 2-azabicyclo[2.2.1]hept-5-en-3-one has been developed (Dominguez & Cullis, 1999). In this context, the crystal structure of *tert*-butyl *N*-[(1*R*,3*S*,4*R*)-3-hydroxy-4-(hydroxymethyl)cyclopentyl]carbamate, (II), has been determined recently (Ober *et al.*, 2004). We report here the crystal structure of the [(1*S*,2*R*,4*R*,5*R*)-4-hydroxymethyl-6-oxabicyclo[3.1.0]hex-2-yl]carbamic acid *tert*-butyl ester, (I) (Fig. 1), which can be transformed into (II) using a regiospecific opening of the epoxide in two steps. The deprotection of the amine leads to a carbocyclic analogue of the 2′-deoxyribofuranose as the end product of the reaction sequence.



(I)

Experimental

The title compound was prepared from (1*R*,2*R*,4*S*,5*R*)-7-oxo-3-oxa-6-azatricyclo[3.2.1.0^{2,4}]octane-6-carboxylic acid *tert*-butyl ester (3.75 g, 16.6 mmol) by treatment with $NaBH_4$ (2.85 g, 75.3 mmol) in dry methanol for 1 h at 273 K. Colourless crystals were obtained by recrystallization from methanol.

Crystal data

$C_{11}H_{19}NO_4$
M_r = 229.27
Orthorhombic, $P2_12_12_1$
a = 9.4088 (6) Å
b = 10.2617 (8) Å
c = 12.6689 (8) Å
V = 1223.19 (15) Å^3
Z = 4
D_x = 1.245 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 15 158 reflections
θ = 2–26°
μ = 0.09 mm^{-1}
T = 193 (2) K
Prism, colourless
0.35 × 0.30 × 0.28 mm

Data collection

Stoe IPDS-II diffractometer	R_{int} = 0.056
ω scans	θ_{max} = 26.2°
Absorption correction: none	$h = -11 \rightarrow 11$
17 917 measured reflections	$k = -12 \rightarrow 12$
1428 independent reflections	$l = -15 \rightarrow 15$
1256 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0613P)^2 + 0.0043P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.032	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.087	$\Delta\rho_{max}$ = 0.17 e Å^{-3}
S = 1.06	$\Delta\rho_{min}$ = −0.14 e Å^{-3}
1428 reflections	Extinction correction: *SHELXL*97
157 parameters	Extinction coefficient: 0.051 (8)
H atoms treated by a mixture of independent and constrained refinement	

Table 1
Selected geometric parameters (Å, °).

N1—C7	1.338 (2)	O2—C6	1.415 (2)
N1—C2	1.450 (2)	C2—C3	1.534 (3)
O1—C1	1.443 (2)	C3—C4	1.542 (3)
O1—C5	1.445 (2)	C4—C5	1.493 (3)
C1—C5	1.456 (3)	C4—C6	1.520 (3)
C1—C2	1.498 (3)		
C7—N1—C2	124.17 (15)	C2—C3—C4	107.07 (15)
C1—O1—C5	60.57 (14)	C5—C4—C6	111.69 (18)
O1—C1—C5	59.78 (13)	C5—C4—C3	103.12 (15)
O1—C1—C2	112.39 (17)	C6—C4—C3	114.72 (16)
C5—C1—C2	109.57 (17)	O1—C5—C1	59.64 (13)
N1—C2—C1	110.06 (16)	O1—C5—C4	112.69 (18)
N1—C2—C3	112.20 (15)	C1—C5—C4	110.20 (17)
C1—C2—C3	103.44 (16)	O2—C6—C4	109.26 (16)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H10···O2	0.85 (2)	2.10 (2)	2.805 (2)	141 (2)
O2—H20···O3^{i}	0.83 (3)	1.90 (3)	2.7050 (18)	166 (3)

Symmetry code: (i) $\frac{3}{2} - x, 2 - y, z - \frac{1}{2}$.

H atoms were initially refined independently, but in the final stage of refinement the methine and methylene H atoms were constrained in the riding-model approximation [U_{iso}(H) = 1.2U_{eq}(C)], with the C—H distances obtained from the refinement; these are in the range 0.95–1.09 Å. The methyl groups were refined with idealized C—H distances (0.98 Å) and H—C—H angles, and U_{iso}(H) = 1.5U_{eq}(C). The N- and O-bonded H atoms are involved in hydrogen bonds (see Table 2) and were refined freely in the final stage of the refinement. In the absence of anomalous dispersion effects, 1027 Friedel pairs were merged, and the absolute configuration was assumed from the synthesis.

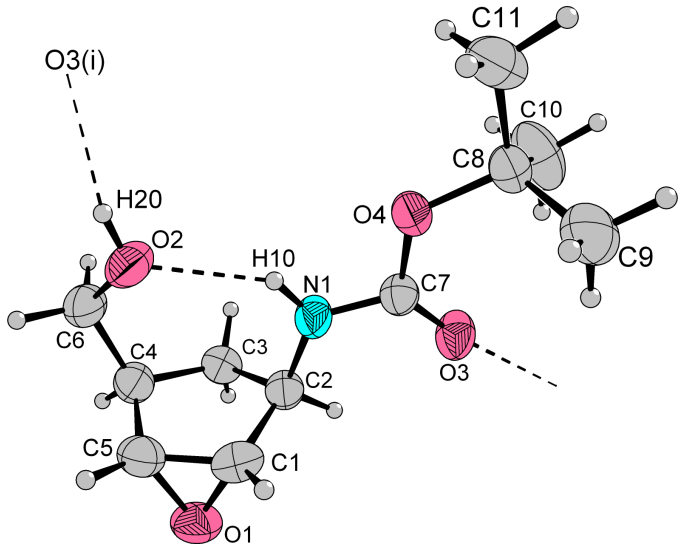


Figure 1
A view of (I), showing the hydrogen-bond interactions (dashed lines). Displacement ellipsoids are drawn at the 50% probability level. [Symmetry code (i) as in Table 2].

Data collection: *X-AREA* (Stoe & Cie, 2003); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 2001); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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