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(1*S*,2*R*,4*S*,5*R*)-*tert*-Butyl 7-oxo-3-oxa-6-aza-tricyclo[3.2.1.$0^{2,4}$]octane-6-carboxylate

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Key indicators

Single-crystal X-ray study
T = 193 K
Mean σ(C–C) = 0.003 Å
R factor = 0.031
wR factor = 0.074
Data-to-parameter ratio = 8.1

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

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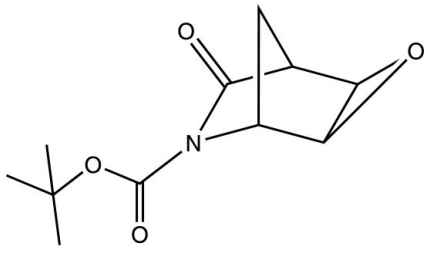
The tricyclic title compound, $C_{11}H_{15}NO_4$, is an intermediate in the synthesis of (1*S*,2*R*,4*R*)-4-amino-2-(hydroxymethyl)cyclopentanol, which is an important carbocyclic analogue of β-2-deoxyribosylamine. All bond lengths and angles in the '*exo* epoxide' are in normal ranges.

Comment

Carbocyclic analogues of 2′-deoxyribonucleotides [such as the antiviral compounds carbavir (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.



(I)

A facile synthesis of (1*S*,2*R*,4*R*)-4-amino-2-(hydroxymethyl)cyclopentanol from 2-azabicyclo[2.2.1]hept-5-en-3-one has been developed (Dominguez & Cullis, 1999). It can be used for the enantioselective synthesis of stabilized 2′-deoxyribonucleotides. In this context, the crystal structure of a protected β-D-2-deoxyribosylamine has been determined recently (Ober *et al.*, 2004). We report here the crystal structure of the second intermediate, *viz*. (1*S*,2*R*,4*S*,5*R*)-*tert*-butyl 7-oxo-3-oxa-6-azatricyclo[3.2.1.$0^{2,4}$]octane-6-carboxylate, (I), in this synthesis, confirming its relative configuration (Fig. 1 and Table 1). The epoxide group adopts the '*exo*' position. The crystal structure of the NH derivative with the epoxide group in the '*endo*' position has already been determined (Dominguez & Cullis, 1999).

Experimental

The title compound was prepared from (1*R*,4*S*)-*tert*-butyl 3-oxo-2-azabicyclo[2.2.1]hept-5-ene-2-carboxylate (3.82 g, 18.1 mmol) by treatment with 3-chlorperoxybenzoic acid (9.83 g, 42.7 mmol, 2.36 equivalents) in CH_2Cl_2 (150ml) for 16 h at room temperature. Colourless crystals were obtained by recrystalization from chloroform (yield: 3.75 g, 16.6 mmol, 92.0%).

Crystal data

$C_{11}H_{15}NO_4$
M_r = 225.24
Orthorhombic, $P2_12_12_1$
a = 5.8200 (3) Å
b = 8.0891 (5) Å
c = 23.3254 (18) Å
V = 1098.13 (12) Å^3
Z = 4
D_x = 1.362 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 7535 reflections
θ = 2.5–25.7°
μ = 0.10 mm^{-1}
T = 193 (2) K
Prism, colourless
0.30 × 0.26 × 0.20 mm

Data collection

Stoe IPDS-II diffractometer
ω scans
Absorption correction: none
5125 measured reflections
1170 independent reflections
992 reflections with $I > 2\sigma(I)$
R_{int} = 0.064
θ_{max} = 25.7°
$h = -6 \rightarrow 7$
$k = -9 \rightarrow 9$
$l = -28 \rightarrow 28$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.074
S = 1.01
1170 reflections
145 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0462P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.13 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—C8	1.523 (3)	O4—C7	1.210 (2)
C1—C7	1.530 (3)	C4—C5	1.534 (3)
C1—C2	1.541 (3)	C5—N6	1.493 (2)
C2—O3	1.444 (2)	C5—C8	1.517 (3)
C2—C4	1.446 (3)	N6—C9	1.386 (3)
O3—C4	1.435 (2)	N6—C7	1.413 (3)
C8—C1—C7	100.58 (16)	N6—C5—C8	100.15 (15)
C8—C1—C2	101.51 (15)	N6—C5—C4	103.11 (13)
C7—C1—C2	101.59 (16)	C8—C5—C4	102.57 (18)
O3—C2—C4	59.57 (13)	C9—N6—C7	130.24 (14)
O3—C2—C1	114.72 (15)	C9—N6—C5	120.55 (16)
C4—C2—C1	105.64 (17)	C7—N6—C5	107.36 (15)
C4—O3—C2	60.29 (13)	O4—C7—N6	127.19 (18)
O3—C4—C2	60.14 (13)	O4—C7—C1	128.7 (2)
O3—C4—C5	114.69 (16)	C5—C8—C1	94.34 (14)
C2—C4—C5	103.89 (17)		

The H atoms were initially refined independently, but in the final stage of refinement they were included in the riding-model approximation [U_{iso} = 1.2U_{eq}(C) for the methine and methylene H atoms and 1.5U_{eq}(C) for the methyl H atoms], with the C—H distances obtained from the refinement; these are in the range 0.91–1.03 Å. In the absence of anomalous dispersion effects, 697 Friedel pairs were merged and the absolute configuration was assumed from the synthesis.

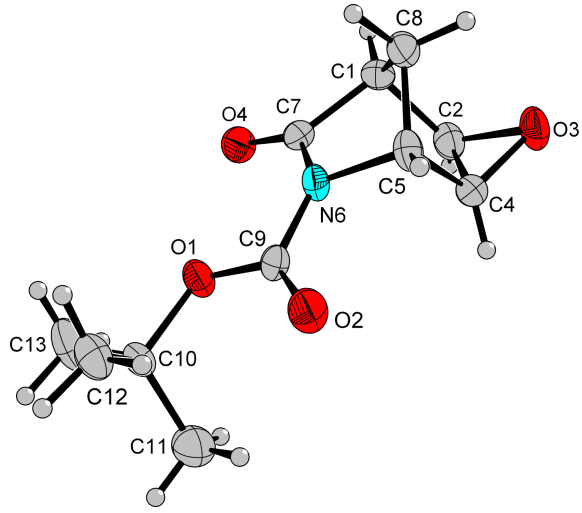


Figure 1
A view of (I). Displacement ellipsoids are drawn at the 50% probability level.

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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