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# (±)-*exo*-2-Hydroxy-5-oxo-4,8-dioxatricyclo-[4.2.1.0$^{3,7}$]nonane-9-*exo*-carboxylic acid

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**Key indicators**

Single-crystal X-ray study
*T* = 150 K
Mean σ(C–C) = 0.002 Å
*R* factor = 0.036
*wR* factor = 0.096
Data-to-parameter ratio = 12.5

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



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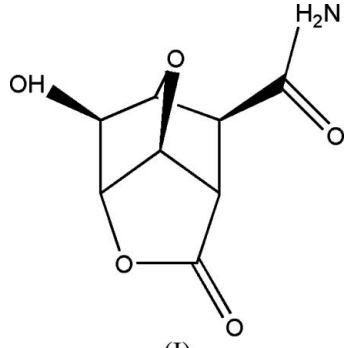
The title compound, $C_8H_9NO_5$, was prepared as a by-product in synthetic efforts to prepare a carbasugar analogue of a putative intermediate, *viz*. (±)-6-hydroxymethyl-7-oxabicyclo[2.2.1]hept-2-*exo*-3-*endo*-diol, in the uridine diphosphate–galactopyranose mutase-catalysed reaction. The structure shows extensive hydrogen bonding involving N—H···O and O—H···O as well as C—H···O interactions.

## Comment

The title compound, (I), was prepared as a by-product in synthetic efforts to prepare a carbasugar analogue of a putative intermediate, *viz*. (±)-6-hydroxymethyl-7-oxabicyclo-[2.2.1]hept-2-*exo*-3-*endo*-diol in the uridine diphosphate–galactopyranose mutase-catalysed reaction, and was synthesized from racemic *exo*-5,6-epoxy-7-oxabicyclo[2.2.1]heptan-*trans*-2,3-dicarboxylic acid dimethyl ester (Sadeghi-Khomami *et al.*, 2005) through treatment with concentrated ammonia solution (30% *w*/*v*).




(I)

The molecular structure of (I) is shown in Fig. 1. There is extensive hydrogen bonding in the structure (see Table 1). N—H···O interactions form a ribbon structure (Fig. 2), which lies parallel to the *ac* plane and propagates along the *c*-axis direction. These ribbons can be considered to be linked by O—H···O interactions, forming a two-dimensional layer parallel to the *bc* plane (Fig. 3). In addition, there are C—H···O interactions in the structure (Table 1) which conform to the geometric conditions for the weak hydrogen bonds given by Desiraju & Steiner (1999).

## Experimental

Formation of the title compound occurred *via* hydrolysis of the *endo*-methyl carboxylate, followed by a 5-*exo*-Tet lactonization on to the *exo*-epoxide. Concurrently, the *exo*-methyl carboxylate is hydrolysed

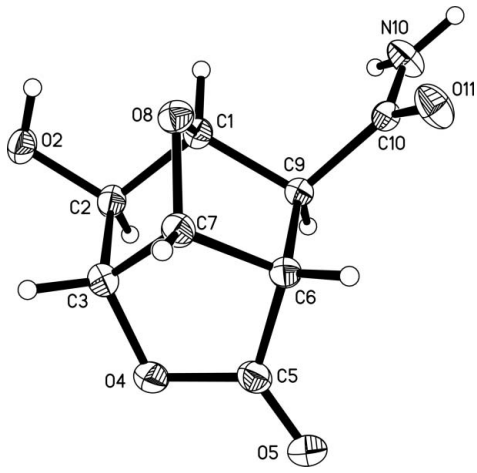



**Figure 1**
View showing the molecular structure and atom-labelling scheme of the title compound. Displacement ellipsoids are drawn at the 50% probability level.

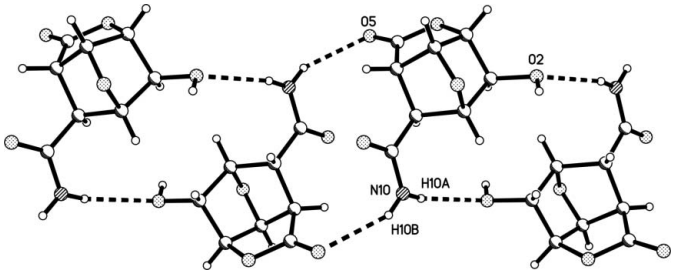



**Figure 2**
View showing N—H···O hydrogen-bonding interactions (dashed lines), leading to a ribbon structure parallel to the *ac* plane and propagating parallel to the *c* axis.

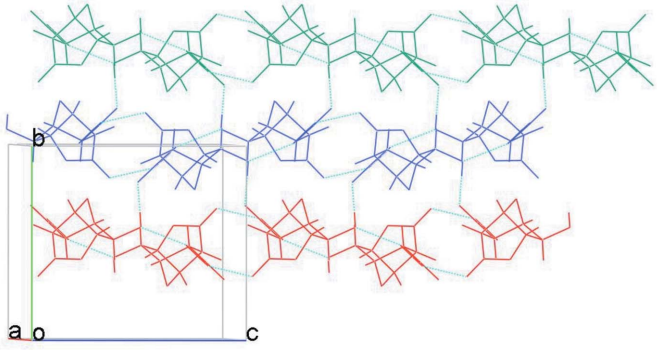
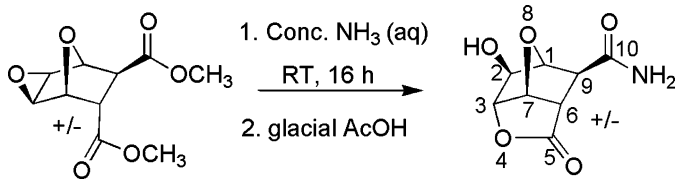



**Figure 3**
View showing linkage of the N—H···O ribbons (each shown as a single colour) by O—H···O interactions (dashed lines), forming a sheet in the *bc* plane.

and, somewhat surprisingly, forms the carboxamide rather than the expected ammonium salt of the carboxylic acid. The resulting solution was neutralized to pH 7.0 after 16 h at room temperature by dropwise addition of glacial acetic acid and the solvent removed by lyophilization (see scheme). This procedure gave the amide-lactone product ($R_F$ = 0.5, 2-propanol/MeOH 2:1), which crystallized from methanol as colourless blocks. The IR spectrum of the title compound clearly revealed carbonyl bands for the lactone (1780 cm$^{-1}$) and carboxamide functional groups (1670 cm$^{-1}$).




*Crystal data*

$C_8H_9NO_5$
$M_r$ = 199.16
Monoclinic, $P2_1/c$
$a$ = 8.3843 (6) Å
$b$ = 9.1844 (6) Å
$c$ = 10.1638 (7) Å
$\beta$ = 97.525 (1)°
$V$ = 775.92 (9) Å$^3$
$Z$ = 4
$D_x$ = 1.705 Mg m$^{-3}$
Mo $K\alpha$ radiation
$\mu$ = 0.14 mm$^{-1}$
$T$ = 150 (2) K
Block, colourless
0.67 × 0.49 × 0.31 mm

*Data collection*

Bruker SMART1000 CCD area-detector diffractometer
$\omega$ scans
Absorption correction: none
6671 measured reflections
1744 independent reflections
1665 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.051
$\theta_{max}$ = 27.5°

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.096
$S$ = 1.03
1744 reflections
140 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0546P)^2 + 0.3811P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.40 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.24 e Å$^{-3}$
Extinction correction: *SHELXL97*
Extinction coefficient: 0.025 (4)

**Table 1**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N10—H10$A$···O2$^{i}$ | 0.84 (2) | 2.30 (2) | 3.0871 (15) | 156.1 (17) |
| N10—H10$B$···O5$^{ii}$ | 0.88 (2) | 2.39 (2) | 3.1743 (15) | 149.8 (16) |
| O2—H2···O11$^{iii}$ | 0.84 (2) | 2.06 (2) | 2.8841 (13) | 167 (2) |
| C2—H2$A$···O11$^{iv}$ | 1.00 | 2.55 | 3.4779 (15) | 154 |
| C1—H1$A$···O11$^{iii}$ | 1.00 | 2.38 | 2.9723 (14) | 117 |
| C7—H7$A$···O4$^{v}$ | 1.00 | 2.43 | 3.2000 (14) | 133 |
| C7—H7$A$···O5$^{vi}$ | 1.00 | 2.60 | 3.2862 (15) | 126 |

Symmetry codes: (i) $-x+1, -y+2, -z+2$; (ii) $-x+1, -y+2, -z+1$; (iii) $x, -y+\frac{3}{2}, z+\frac{1}{2}$; (iv) $-x+1, y+\frac{1}{2}, -z+\frac{3}{2}$; (v) $-x, y-\frac{1}{2}, -z+\frac{3}{2}$; (vi) $-x, -y+2, -z+1$.

All H atoms could be located in a difference Fourier map. However, the H atoms bound to carbon were subsequently placed in idealized positions and included as part of a riding model, with C—H = 1.00 Å and $U_{iso}$(H) = 1.2$U_{eq}$(C). Positional and $U_{iso}$ parameters were refined for H atoms bound to nitrogen and oxygen.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT* and *SHELXTL* (Bruker, 2001); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *MERCURY* (Version 1.4.1; Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

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