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Key indicators

Single-crystal X-ray study
T = 293 K
 Mean σ (C–C) = 0.005 Å
R factor = 0.053
wR factor = 0.137
 Data-to-parameter ratio = 10.1

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

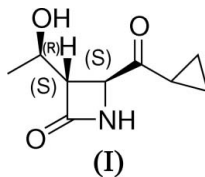
(3*S*,4*S*)-4-(Cyclopropylcarbonyl)-3-[(1*R*)-1-hydroxyethyl]azetidin-2-one

The crystal structure of the title compound, C₉H₁₃NO₃, is stabilized by intermolecular O–H···O and N–H···O hydrogen bonds, leading to a two-dimensional network extending parallel to the (001) plane. The absolute configuration has been assigned by reference to the chiral hydroxy C atom, which remains unchanged during the synthetic procedure.

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Comment

In the course of the synthesis of acetoxyazetidinone, which is used as a key structure for the preparation of carbapenems, which are a growing class of β -lactam antibiotics, the title compound, (3*S*,4*S*)-4-(cyclopropylloxomethyl)-3-[(1*R*)-1-hydroxyethyl]azetidin-2-one, (I), was prepared and obtained as single crystals suitable for X-ray structural analysis. The absolute configuration of the three stereo centres has been assigned by reference to the chiral hydroxy C atom, which remains unchanged during the synthetic procedure.



The occurrence of O–H···O and N–H···O intermolecular hydrogen bonds in the structure of (I) results in the

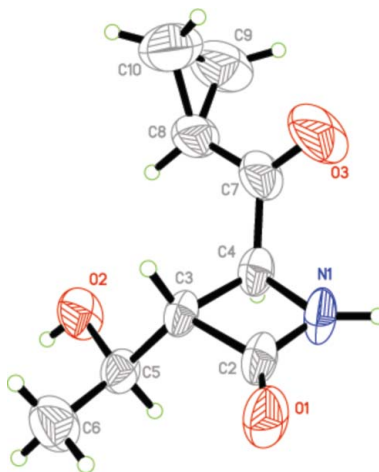


Figure 1
 A molecular view of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

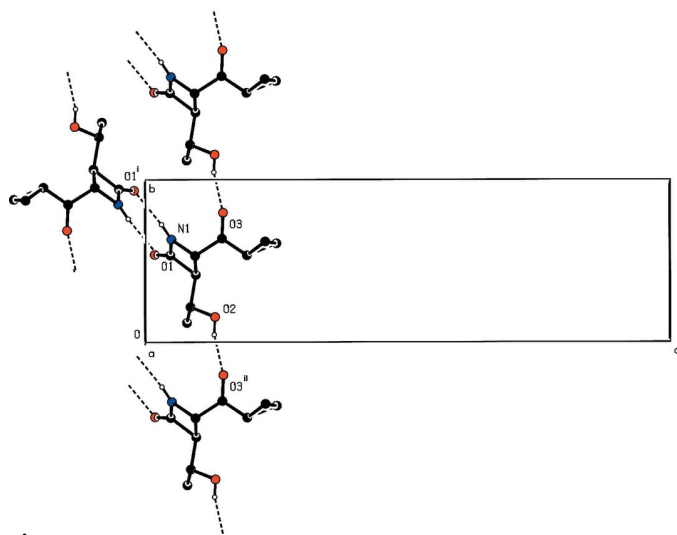


Figure 2

A partial packing view, showing the O—H...O and N—H...O hydrogen bonds (dashed lines). H atoms not involved in the hydrogen bonding have been omitted for clarity. [Symmetry codes: (i) $x - \frac{1}{2}, \frac{3}{2} - y, -z$; (ii) $x, y - 1, z$.]

formation of a two-dimensional network extending parallel to the (001) plane (Table 1, Fig. 2).

Experimental

To a solution of (3*S*,4*S*)-4-(cyclopropyloxomethyl)-*N*-diphenylmethyl-3-[(1*R*)-1-hydroxyethyl]azetidin-2-one, (II) (1.0 g, 2.9 mmol, 1.0 equivalent) in CH₂Cl₂ (40 ml) and water (25 ml) were added *N*-bromosuccinimide (0.57 g, 3.2 mmol, 1.1 equivalents) and bromine (0.023 g, 0.15 mmol, 0.05 equivalents). This two-phase system was stirred overnight at room temperature under the light of the fume hood. After dilution with CH₂Cl₂ (20 ml) and extraction with 5% Na₂SO₃ (20 ml), the organic phase was concentrated under reduced pressure to give a yellow oil. To this intermediate was added a mixture of acetone and water (1:1 v/v; 50 ml) and *p*-TsOH (0.55 g, 2.9 mmol, 1.0 equivalents). The solution was stirred overnight at room temperature in the dark. Solid Na₂CO₃ (0.154 g, 1.45 mmol, 0.5 equivalents) was then added and the mixture was concentrated under reduced pressure. The residue was extracted with petroleum ether (2 × 10 ml). The residue was then extracted with acetone (5 × 10 ml). The acetone phase was concentrated under reduced pressure to give the crude product. The product (0.32 g, 1.74 mmol, 61% yield) was obtained by recrystallization from propan-2-ol (Laurent *et al.*, 2004, 2003). Single crystals of (I) were obtained from a solution in ethyl acetate and petroleum ether (1:1 v/v).

Crystal data

C₉H₁₃NO₃

$M_r = 183.20$

Orthorhombic, $P2_12_12_1$

$a = 5.422$ (2) Å

$b = 7.436$ (3) Å

$c = 24.043$ (9) Å

$V = 969.2$ (6) Å³

$Z = 4$

$D_x = 1.255$ Mg m⁻³

Mo $K\alpha$ radiation

Cell parameters from 2179

reflections

$\theta = 2.9$ – 23.6°

$\mu = 0.09$ mm⁻¹

$T = 293$ (2) K

Block, colourless

$0.40 \times 0.22 \times 0.20$ mm

Data collection

Bruker APEX area-detector diffractometer

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2001)

$T_{\min} = 0.963$, $T_{\max} = 0.981$

5400 measured reflections

1208 independent reflections

1094 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.025$

$\theta_{\max} = 26.5^\circ$

$h = -6 \rightarrow 6$

$k = -9 \rightarrow 9$

$l = -21 \rightarrow 30$

Refinement

Refinement on F^2

$R[F^2 > 2\sigma(F^2)] = 0.053$

$wR(F^2) = 0.137$

$S = 1.09$

1208 reflections

120 parameters

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0672P)^2 + 0.2655P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.20$ e Å⁻³

$\Delta\rho_{\min} = -0.17$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1 \cdots O1 ⁱ	0.86	2.02	2.874 (3)	170
O2—H2 \cdots O3 ⁱⁱ	0.82	1.98	2.769 (3)	161

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

The H atoms were positioned geometrically and treated as riding on their parent atoms, with C—H = 0.98 (C_{methylene}), 0.97 (C_{methylene}) and 0.96 Å (C_{methyl}), O—H = 0.82 Å and N—H = 0.86 Å. $U_{\text{iso}}(\text{H})$ values were set equal to xU_{eq} of the carrier atom, where $x = 1.5$ for methyl and $x = 1.2$ for all other H atoms. In the absence of significant anomalous scattering effects, Friedel pairs were merged. The absolute configuration has been assigned by reference to the chiral hydroxy C atom, which remains unchanged during the synthetic procedure.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINTE* (Bruker, 2001); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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