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(4*S*,5*S*)-4-Benzyl-2-oxooxazolidine-5-carboxylic acid

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Key indicators

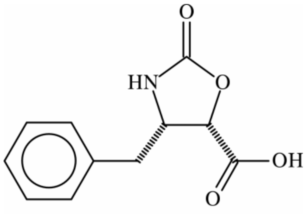
Single-crystal X-ray study
T = 160 K
Mean *σ*(C–C) = 0.002 Å
R factor = 0.027
wR factor = 0.074
Data-to-parameter ratio = 12.3

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

In the title compound, $C_{11}H_{11}NO_4$, the five-membered ring has an envelope conformation. N—H···O and O—H···O hydrogen bonds link the molecules into chains.

Comment

The title compound, (I), was synthesized as part of a study of the stereocontrolled synthesis of *anti*-α-hydroxy-β-amino acid derivatives (Ambroise *et al.*, 2002). It was prepared from the corresponding compound with a 4-tolylthiocarbonyl group instead of the carboxylic acid group. We have also determined the structure of that intermediate product (Clegg & Elsegood, 2002) in a racemic form. Compound (I), however, was obtained from an enantiomerically pure sample, demonstrating the stereoselectivity of the reaction.



(I)

The crystal structure of (I) was determined in order to confirm the relative stereochemistry of the substituents on the heterocyclic ring; this was found to be *cis*. It also demonstrates that the hydrolysis of the precursor proceeds without epimerization at the stereocentre adjacent to the thioester group, which is replaced by the carboxylic acid group in (I).

The bond lengths and angles of the heterocyclic ring are typical of those found in 39 related compounds in the April 2002 release of the Cambridge Structural Database (Allen & Kennard, 1993). The ring has a conformation close to an envelope, with C3 as the flap atom (lying 0.427 Å from the mean plane of the other four atoms, which have a root-mean-square deviation of 0.039 Å from this plane); this is seen also in the torsion angles in Table 1.

Since the structure is not centrosymmetric, the common motif of centrosymmetric pairs of hydrogen-bonded carboxylic acids is not found here. Instead N—H acts as a hydrogen-bond donor to the carboxyl C=O group, and O—H acts as a donor to the ring carbonyl group (Table 2). These interactions generate chains of molecules, as shown in Fig. 2.

Experimental

The synthesis of the title compound is described by Ambroise *et al.* (2002).

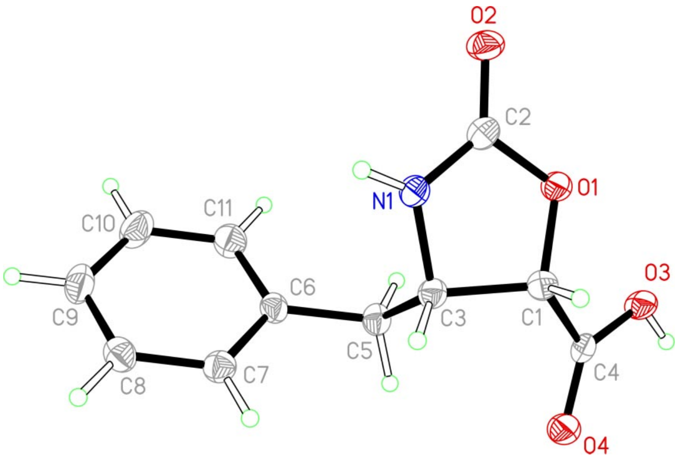


Figure 1
The molecular structure of (I), with atom labels and 50% probability ellipsoids for non-H atoms.

Crystal data

$C_{11}H_{11}NO_4$
$M_r = 221.21$
Monoclinic, $P2_1$
$a = 7.244$ (3) Å
$b = 6.3067$ (19) Å
$c = 11.554$ (3) Å
$\beta = 99.41$ (3)°
$V = 520.7$ (3) Å^3
$Z = 2$

$D_x = 1.411$ Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 39 reflections
$\theta = 20.6$–24.9°
$\mu = 0.92$ mm^{-1}
$T = 160$ (2) K
Block, colourless
0.61 × 0.40 × 0.36 mm

Data collection

Stoe–Siemens four-circle diffractometer
ω/θ scans with on-line profile fitting (Clegg, 1981)
Absorption correction: ψ scan (*XPREP* in *SHELXTL*; Sheldrick, 1994)
$T_{\min} = 0.605$, $T_{\max} = 0.720$
2697 measured reflections
1851 independent reflections

1841 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.028$
$\theta_{\max} = 67.3°$
$h = -8 \rightarrow 8$
$k = -7 \rightarrow 7$
$l = -13 \rightarrow 13$
5 standard reflections
frequency: 60 min
intensity decay: none

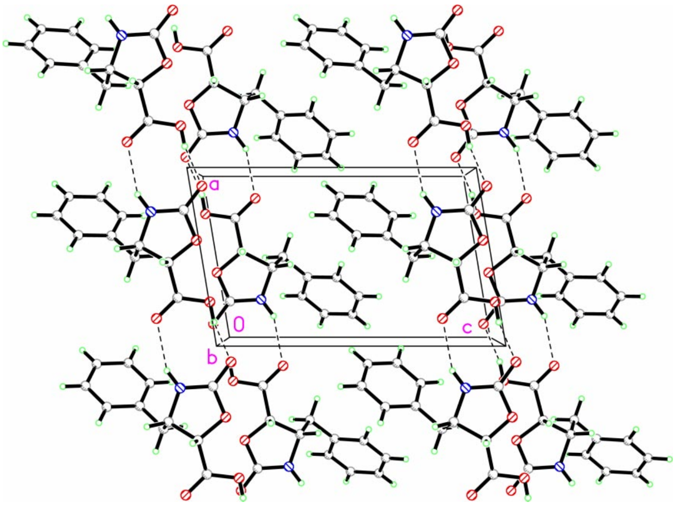


Figure 2
A packing diagram, viewed down the *b* axis, showing the hydrogen bonding as dashed lines.

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.027$
$wR(F^2) = 0.074$
$S = 1.06$
1851 reflections
150 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0421P)^2 + 0.1206P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.19$ e Å^{-3}
$\Delta\rho_{\min} = -0.14$ e Å^{-3}
Extinction correction: *SHELXTL*
Extinction coefficient: 0.044 (2)
Absolute structure: Flack (1983), 816 Friedel pairs
Flack parameter = −0.10 (18)

Table 1
Selected geometric parameters (Å, °).

C1—O1	1.4481 (17)	C2—N1	1.3293 (19)
C1—C3	1.5420 (19)	C2—O2	1.2191 (17)
O1—C2	1.3578 (16)	N1—C3	1.4655 (18)
O1—C1—C3	104.71 (11)	N1—C2—O2	129.73 (13)
C1—O1—C2	107.41 (10)	C2—N1—C3	112.62 (12)
O1—C2—N1	109.93 (11)	C1—C3—N1	97.33 (11)
O1—C2—O2	120.33 (12)		
C3—C1—O1—C2	23.86 (15)	C2—N1—C3—C1	23.13 (15)
C1—O1—C2—N1	−9.54 (16)	O1—C1—C3—N1	−26.95 (13)
O1—C2—N1—C3	−10.06 (18)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O4$^{\rm i}$	0.785 (19)	2.274 (19)	2.9806 (19)	150.1 (18)
O3—H3A···O2$^{\rm ii}$	0.84	1.76	2.5841 (16)	165

Symmetry codes: (i) $1 + x, y, z$; (ii) $x - 1, y, z$.

H atoms attached to C and O atoms were placed geometrically and refined with a riding model (including free rotation about the C—O bond), and with $U_{\rm iso}$ constrained to be 1.2 times $U_{\rm eq}$ of the carrier atom. The positional parameters of the H atom on the oxazolidine N atom were refined freely.

Data collection: *DIF*4 (Stoe & Cie, 1988); cell refinement: *DIF*4; data reduction: local programs; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1994); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and local programs.

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