gram(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *TEXSAN*. Software used to prepare material for publication: *SHELXL*93.

We thank the EPSRC and Zeneca Specialities for funding and Dr Neil Feeder for his assistance.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: HA1205). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1998). C**54**, 240–242

Cyclothreonine at 122 K

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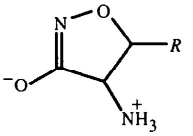
(*Received 24 April 1997; accepted 17 October 1997*)

Abstract

Spectroscopic and crystallographic data for the title compound (4*S*,5*R*)-4-amino-5-methyl-3-isoxazolidinone are reported. The title compound crystallizes with one half equivalent of water in a zwitterionic structure, $C_4H_8N_2O_2.0.5H_2O$, stabilized by intermolecular hydrogen bonds.

Comment

Cyclothreonine has been characterized as part of a study of the siderophore pseudomonine, isolated from iron-deficient cultures of *Pseudomonas fluorescens* (Anthoni *et al.*, 1995). The stereochemistry of cyclothreonine determined from ^{1}H NMR spectra (D_2O) was discussed by Turchin (1971), who stated that the coupling constant between the two H atoms connected to the five-membered ring (H4 and H5) would be 7.0 Hz in a *cis* conformation and 8.7 Hz in a *trans* conformation. However, this has not been confirmed by independent methods, so an investigation using X-ray diffraction analysis was initiated. The structure of an analogous compound, cycloserine (4-amino-3-isoxazolidinone), has been examined by IR spectroscopy (Preti & Tosi, 1979), derivatization and potentiometric studies (Hidy *et al.*, 1955). It was deduced that cycloserine exists as a zwitterion in aqueous solution and in the solid state. An early study of the crystal structure of cycloserine hydrochloride has been reported (Turley & Pepinsky, 1957).



R = H, cycloserine
R = CH_3, cyclothreonine

From Fig. 1 and the geometric parameters in Table 2, is can be seen that the methyl and amino groups are *cis* with respect to each other confirming the suggestions of Turchin (1971). The amino group is protonated giving rise to a zwitterionic structure, as in cycloserine. The isoxazolidinone ring adopts an envelope conformation, the O1, N2, C3, O6 and C4 atoms being coplanar [r.m.s. deviation of the least-squares plane is 0.01 Å and

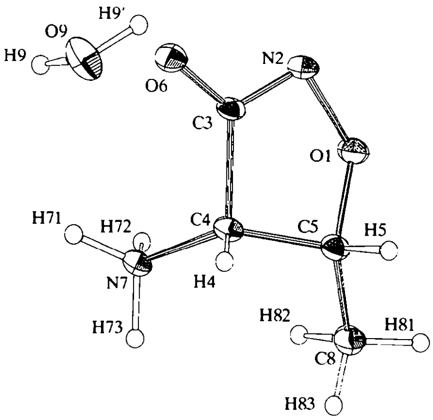


Fig. 1. View of cyclothreonine showing the labelling of the atoms. Displacement ellipsoids are shown at the 50% probability level. H atoms are drawn as spheres of fixed radii.

largest deviation from the plane is 0.015 (1) Å for N2]. The interplanar angle between this plane and the plane defined by atoms C5, O1 and N2 is 20.89 (11)°. The N2—C3 and C3—O6 distances are consistent with an enolized lactam having a double bond between N2 and C3.

The five potential H-atom donors participate in hydrogen bonds (Table 3 and Fig. 2). The water molecule plays an important role in this system. It acts as a double acceptor and a double donor of hydrogen bonds and thereby links molecules related through the symmetry of the twofold axis. The H72 atom is involved in a bifurcated hydrogen bond to O6 and O9. The imino N atom, N2, accepts another proton, H73, from the ammonium group. The deprotonized enol O atom, O6, accepts three hydrogen bonds.

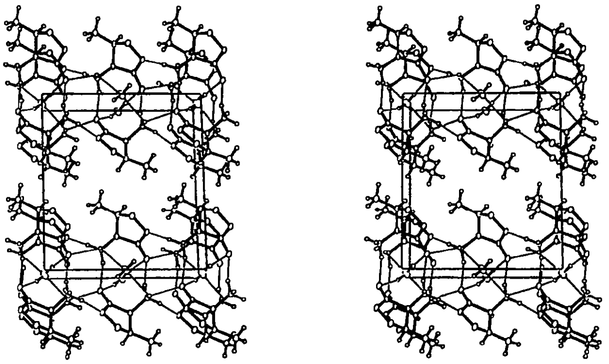


Fig. 2. Stereoscopic view of the crystal packing viewed along the *b* axis. The *c* axis is vertical. The hydrogen bonds are draw as thin lines.

Furthermore, molecules are related by translational symmetry along the *b* axis interacting through C—H···O hydrogen bonds (Table 3) which connect the molecules into infinite chains.

The molecules pack into a layer structure in which layers of hydrogen-bonded molecules in the *ab* plane interact though weaker interactions along the *c* axis.

Experimental

(4*S*,5*R*)-4-Amino-5-methyl-3-isoxazolidinone (*cis*-cyclothreonine) was prepared from L-threonine according to the procedure given for DL-*cis*-5-methyl-4-amino-3-isoxazolidinone (Platner *et al.*, 1957). Crystals were prepared by recrystallization from an ethanol–water (90/10) mixture containing 0.01% picric acid. Yield: 36%, yellowish crystals; m.p. 438–449 K. Analysis calculated for $C_4H_8N_2O_2.0.5H_2O$: C 38.40, H 7.25, N 22.39%; found: C 38.90, H 6.94, N 22.67%. FABMS: *m/z* 117 (M^+H). ^{1}H NMR (D_2O): 1.27 (*d*, CH_3 , J = 6.7 Hz), 3.99 (*d*, H4, J = 7.0 Hz), 4.61 (*dq*, H5, J = 6.7, 7.0 Hz) p.p.m. ^{13}C NMR (D_2O): 174.6 (C═O), 79.9 (C5), 59.6 (C4), 14.8 (CH_3) p.p.m.

Crystal data

$C_4H_8N_2O_2.0.5H_2O$
$M_r = 125.13$
Monoclinic
*C*2
$a = 10.301\ (3)$ Å
$b = 4.8869\ (12)$ Å
$c = 11.510\ (2)$ Å
$\beta = 91.102\ (19)°$
$V = 579.3\ (2)$ Å^3
$Z = 4$
$D_x = 1.435$ Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
$\lambda = 1.54184$ Å
Cell parameters from 18 reflections
$\theta = 36.62$–$42.02°$
$\mu = 1.015$ mm^{-1}
$T = 122\ (2)$ K
Parallelepiped
0.45 × 0.25 × 0.10 mm
Translucent light yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
Profile data from ω–2θ scans
Absorption correction: none
2533 measured reflections
1190 independent reflections
1184 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.023$
$\theta_{\rm max} = 75°$
$h = -12 \rightarrow 12$
$k = -6 \rightarrow 6$
$l = -14 \rightarrow 14$
5 standard reflections
frequency: 166.7 min
intensity decay: 5.6%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.025$
$wR(F^2) = 0.066$
$S = 1.084$
1190 reflections
115 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0452P)^2 + 0.1577P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.27\ (4)$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.19\ (4)$ e Å^{-3}

Extinction correction: *SHELXL*96
Extinction coefficient: 0.0094 (12)
Scattering factors from *International Tables for Crystallography* (Vol. C)
Absolute structure: Flack (1983)
Flack parameter = −0.05 (19)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U^{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	$U_{\rm eq}$
O1	0.44674 (8)	0.18647 (17)	0.68695 (7)	0.0179 (2)
N2	0.35426 (9)	0.2789 (2)	0.77292 (9)	0.0170 (2)
C3	0.40400 (10)	0.4882 (2)	0.82773 (10)	0.0139 (2)
C4	0.53660 (10)	0.5668 (2)	0.78026 (10)	0.0134 (2)
C5	0.53035 (11)	0.4184 (2)	0.66448 (10)	0.0150 (2)
O6	0.35002 (7)	0.61767 (18)	0.91014 (7)	0.0175 (2)
N7	0.64313 (9)	0.4720 (2)	0.85975 (9)	0.0145 (2)
C8	0.65460 (12)	0.3134 (3)	0.61227 (11)	0.0216 (3)
O9	1/2	0.0553 (3)	1	0.0252 (3)

Table 2. *Selected geometric parameters (Å, °)*

O1—C5	1.4499 (14)	C3—C4	1.5299 (14)
O1—N2	1.4583 (13)	C4—N7	1.4890 (14)
N2—C3	1.3016 (16)	C4—C5	1.5175 (16)
C3—O6	1.2766 (15)	C5—C8	1.5140 (15)

C5—O1—N2	106.08 (8)	N7—C4—C3	110.78 (9)
C3—N2—O1	108.42 (9)	C5—C4—C3	99.81 (9)
O6—C3—N2	125.21 (10)	O1—C5—C8	108.37 (10)
O6—C3—C4	123.05 (10)	O1—C5—C4	103.43 (8)
N2—C3—C4	111.73 (9)	C8—C5—C4	119.25 (10)
N7—C4—C5	114.16 (9)		

Table 3. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N7—H71···O6^{i}	0.921 (17)	1.838 (17)	2.742 (1)	167 (2)
N7—H72···O6^{ii}	0.94 (2)	2.26 (2)	2.798 (1)	116 (2)
N7—H72···O9	0.94 (2)	2.48 (2)	3.003 (1)	115 (1)
N7—H73···N2^{iii}	0.954 (17)	1.929 (18)	2.839 (1)	159 (2)
O9—H9···O6^{iv}	0.87 (2)	1.99 (2)	2.823 (1)	160 (2)
C4—H4···O1^{v}	0.92 (2)	2.52 (2)	3.338 (2)	148 (1)

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

Since the molecule is optically active, the choice of a non-centrosymmetric space group is obvious. The absolute structure determined by means of the anomalous scattering contribution is in agreement with the known chirality of the molecule. At the time of data collection, an absorption correction was deemed unnecessary. Afterwards, when a water molecule was detected in the structure, increasing the absorption coefficient, the crystal had deteriorated so that corrections by ψ scans or numerical integration could not be made.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *DREADD* (Blessing, 1987). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*96 (Sheldrick, 1996). Molecular graphics: *ORTEP*II (Johnson, 1976).

We wish to thank Mr Flemming Hansen for technical assistance and Dr Sine Larsen for valuable discussions.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: AB1493). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1998). **C54**, 242–244

6α-Chloro-3α-hydroxymethyl-2,2-dimethylpenam 1,1-Dioxide

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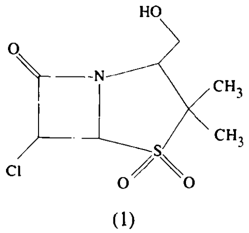
(*Received 3 June 1997; accepted 6 October 1997*)

Abstract

In the title compound (6-chloro-3-hydroxymethyl-2,2-dimethyl-5-oxo-2,3,6,6a-tetrahydro-5*H*-azeto[2,1-*b*]thiazole 1,1-dioxide, $C_8H_{12}ClNO_4S$), the 2-azetidinone ring is non-planar. The thiazolidine ring has a conformation that can be classified as *C*3, with 2α-CH_3 pseudo-equatorial, 2β-CH_3 axial and 3α-*R* axial. The N atom has a pyramidal bonding arrangement. The structure is stabilized by O—H···O hydrogen bonds along the direction of the *c* axis.

Comment

As a first step towards the study of structure–activity relationships of β-lactam derivatives as elastase inhibitors, the structure of (1), an intermediate in the synthesis of the active compounds (Boschetti, Mascaretti *et al.*, 1995; Boschetti, Mata *et al.*, 1995), has been determined.



(1)

Analysis of the molecular geometry (Table 1) shows that the 2-azetidinone (β-lactam) ring is non-planar and that the N(4) atom has a pyramidal bonding arrangement, being out of the plane defined by its substituents (Fig. 1). The value of the out-of-plane distance, 0.387 (2) Å, and the sum of the angles around N(4), 338.3°, compare well with the reported values for

Acta Crystallographica Section C
ISSN 0108-2701 © 1998