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(*R*)-1,3-Thiazolidin-3-ium-4-carboxylate

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The title compound, synthetic (*R*)-thioproline, $C_4H_7NO_2S$, crystallizes from water as a zwitterion. The N—H···O hydrogen-bonding scheme involves both intra- and intermolecular interactions.

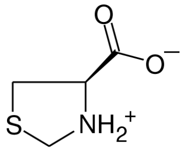
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.029
wR factor = 0.079
Data-to-parameter ratio = 22.1

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

Comment

(*R*)-1,3-Thiazolidin-3-ium-4-carboxylate, (I), was prepared as part of our studies into synthetic routes to derivatives of the potent cytotoxic agent thiocoraline (Boger *et al.*, 2001).



(I)

The non-H-atom geometrical parameters are normal and are determined to higher precision than those arising from the earlier studies on the natural *S*-form of thioproline (Loscalzo *et al.*, 1973; Chacko, 1974). There was some ambiguity in these studies as to the location of the transferrable (carboxyl/amino) proton. The small C—S—C bond angle is comparable to that seen in other thiazolidine derivatives (Gainsford *et al.*, 1980). The absolute structure (*R* chirality of C2) of the title compound is consistent with that of the L-cysteine starting material.

This work unambiguously shows that a zwitterion is present in the crystal. The two N—H entities interact with both carboxyl oxygen species (Table 2) resulting in a one-dimensional network of hydrogen bonds propagating in the [100] direction (Fig. 3). The N1—H1*B* moiety is involved in bifurcated hydrogen bonding, one link being intramolecular, the other intermolecular. There are no significant sulfur–sulfur interactions [shortest $S\cdots S^{i}$ contact = 3.981 (1) Å; symmetry code: (i) $\frac{1}{2}+x, \frac{1}{2}-y, 1-z$].

Experimental

L-Cysteine (0.363 g, 0.003 mol) was dissolved in 2 ml of water to which 1.5 ml (0.003 mol) of 2 *M* HCl solution was added. 0.4 ml (0.003 mol) of 37% (*w*/*w*) formaldehyde was added. The mixture was left to react for 15 h at room temperature. Then 0.5 ml of pyridine was added. After 30 min, a white solid began to separate from the solu-

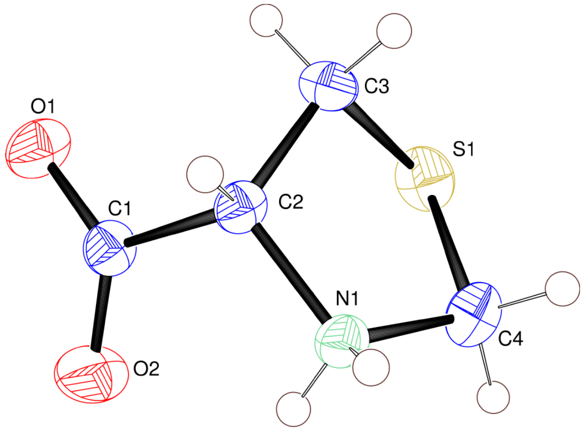


Figure 1
The structure of (I) (50% displacement ellipsoids, arbitrary spheres for the H atoms).

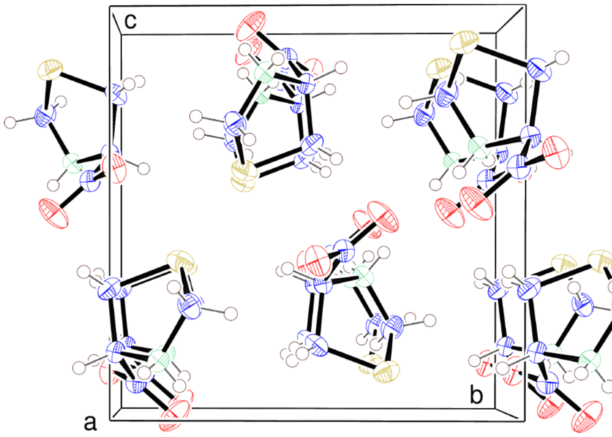


Figure 2
Packing diagram for (I).

tion. Ethanol (1 ml) was added and the solution was left to stand. Prismatic crystals separated from the solution by vacuum filtration. The crude product was recrystallized from hot water.

Crystal data

$C_4H_7NO_2S$
$M_r = 133.18$
Orthorhombic, $P2_12_12_1$
$a = 5.6733$ (3) Å
$b = 9.9375$ (6) Å
$c = 9.9407$ (6) Å
$V = 560.44$ (6) Å^3
$Z = 4$
$D_x = 1.578$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2816 reflections
$\theta = 2.9$–$30.0°$
$\mu = 0.48$ mm^{-1}
$T = 293$ (2) K
Block, colourless
0.52 × 0.20 × 0.20 mm

Data collection

Bruker SMART1000 CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*, Bruker, 1999)
$T_{\min} = 0.875$, $T_{\max} = 0.920$
4858 measured reflections
1611 independent reflections
1483 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.027$
$\theta_{\max} = 30.0°$
$h = -7 \rightarrow 5$
$k = -12 \rightarrow 13$
$l = -13 \rightarrow 13$
Intensity decay: none

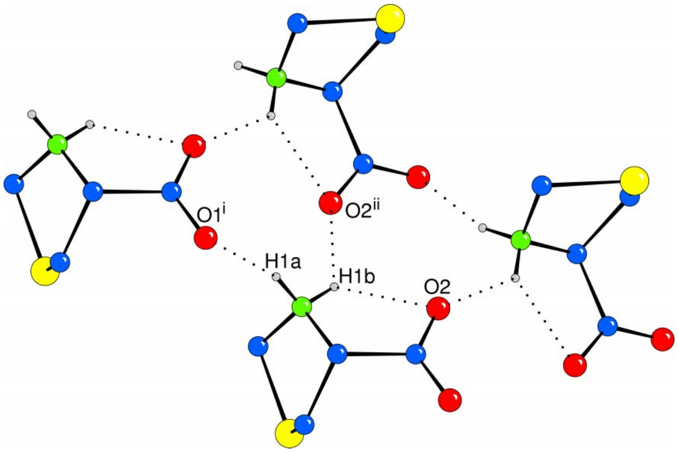


Figure 3
Hydrogen-bonding scheme in (I). Symmetry codes are as in Table 2.

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.079$
$S = 1.03$
1611 reflections
73 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.053P)^2P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.28$ e Å^{-3}
$\Delta\rho_{\min} = -0.23$ e Å^{-3}
Absolute structure: (Flack, 1983)
Flack parameter = 0.07 (8), 654 Friedel pairs

Table 1
Selected geometric parameters (Å, °).

S1—C4	1.7945 (16)	N1—C2	1.4978 (17)
S1—C3	1.8097 (17)	O2—C1	1.2386 (18)
O1—C1	1.2509 (18)	C1—C2	1.5457 (18)
N1—C4	1.4942 (18)	C2—C3	1.5217 (19)
C4—S1—C3	88.62 (7)	N1—C2—C3	107.25 (11)
C4—N1—C2	111.56 (10)	N1—C2—C1	109.28 (10)
O2—C1—O1	127.34 (13)	C3—C2—C1	113.02 (12)
O2—C1—C2	117.08 (12)	C2—C3—S1	103.92 (9)
O1—C1—C2	115.56 (12)	N1—C4—S1	106.49 (9)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1^{i}	0.79	1.89	2.6556 (16)	163
N1—H1B···O2	0.81	2.12	2.6389 (16)	122
N1—H1B···O2^{ii}	0.81	2.17	2.7819 (16)	133

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

Data collection: *SMART* (Bruker, 1999); cell refinement: *SMART*; data reduction: *SMART*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97.

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