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(2*R*,6*S*)-6-Amino-2-(2-thienyl)-1,4-thiazepan-5-one

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.002 Å
R factor = 0.025
wR factor = 0.069
Data-to-parameter ratio = 18.9

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

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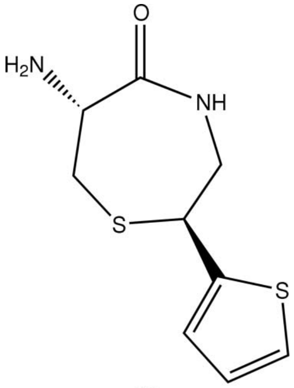
In the title compound, $C_9H_{12}N_2OS_2$, the thiazepane ring adopts a chair conformation. The molecules are linked into chains *via* N—H···O hydrogen bonds.

Comment

The title compound, (I), is a key intermediate used in the preparation of temocapril, one kind of angiotensin-converting enzyme inhibitor against glutamate-induced neurotoxicity (Ishizuka *et al.*, 1997; Yanagisawa *et al.*, 1987). All the geometrical parameters for (I) lie within their expected ranges (Allen *et al.*, 1987). The thiazepane ring adopts a chair conformation, with atoms S1, C3 and N1 having deviations of 0.823 (2), −1.118 (2) and −1.120 (2) Å, respectively, from the least-squares plane through the other four atoms (Fig. 1). The amino and carbonyl groups are involved in intramolecular and intermolecular N—H···O hydrogen bonds (Table 2). An intermolecular hydrogen bond links molecules into a ribbon motif extending along the *a* axis (Fig. 2).



(I)

Experimental

The material supplied by Zhejiang Huahai Pharmaceutical Co. Ltd was an enantiomer, the specific rotation at 293 K being +49.5° (*c* = 1.0, DMF). It was recrystallized from ethanol–ethyl acetate (4:1 *v*/*v*) by slow evaporation, giving colorless crystals of (I) suitable for X-ray diffraction.

Crystal data

$C_9H_{12}N_2OS_2$	$Z = 4$
$M_r = 228.33$	$D_x = 1.420$ Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation
$a = 6.314$ (3) Å	$\mu = 0.47$ mm^{-1}
$b = 9.785$ (3) Å	$T = 298$ (1) K
$c = 17.289$ (6) Å	Chunk, colorless
$V = 1068.2$ (7) Å^3	0.34 × 0.32 × 0.26 mm

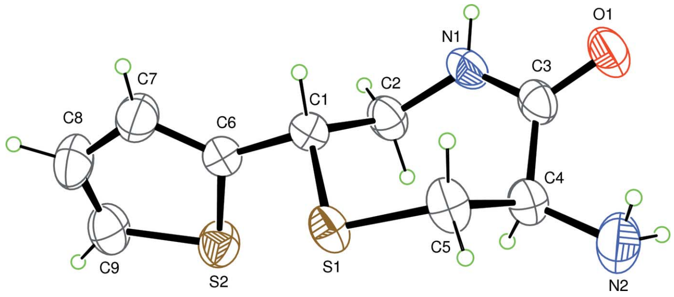


Figure 1
The molecular structure and atom-numbering scheme for (I). Displacement ellipsoids are drawn at the 50% probability level.

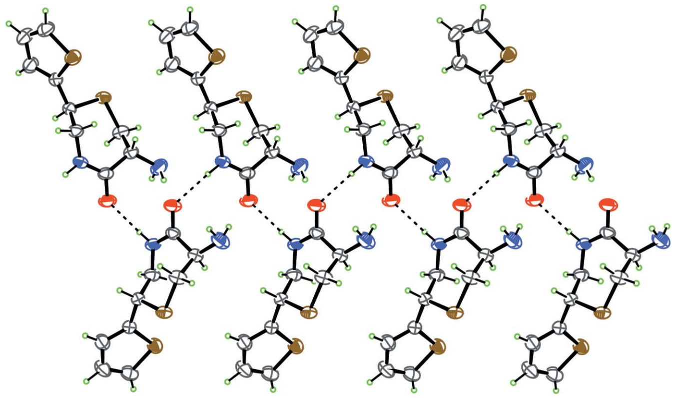


Figure 2
Molecular chain in (I). Displacement ellipsoids are drawn at the 30% probability level and dashed lines indicate hydrogen bonds.

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{\min} = 0.859$, $T_{\max} = 0.886$
10534 measured reflections
2442 independent reflections
2239 reflections with $F^2 > 2\sigma(F^2)$
$R_{\rm int} = 0.022$
$\theta_{\max} = 27.5^\circ$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.025$
$wR(F^2) = 0.069$
$S = 1.04$
2442 reflections
129 parameters
H-atom parameters constrained
$w = 1/[0.0004F_o^2 + \sigma(F_o^2)]/(4F_o^2)$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.31$ e Å^{-3}
$\Delta\rho_{\min} = -0.26$ e Å^{-3}
Extinction correction: Larson (1970)
Extinction coefficient: 45 (5)
Absolute structure: Flack (1983), 1008 Friedel pairs
Flack parameter: 0.006 (7)

Table 1
Selected bond lengths (Å).

S1—C1	1.8254 (15)	O1—C3	1.2408 (18)
S1—C5	1.7938 (17)	N1—C2	1.459 (2)
S2—C6	1.7291 (16)	N1—C3	1.336 (2)
S2—C9	1.705 (2)	N2—C4	1.458 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H101···O1$^{\rm i}$	0.86	2.09	2.945 (2)	177
N2—H201···O1$^{\rm ii}$	0.86	2.32	3.161 (2)	165
N2—H202···O1	0.87	2.48	2.784 (2)	101

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

The two H atoms of the NH_2 group were located in difference Fourier maps and refined as riding on the N atom with the as-found N—H distances and with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm N})$. All other H atoms were placed in calculated positions with N—H = 0.86 Å and C—H = 0.93–0.98 Å, and constrained to ride on their parent atoms, with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}$(carrier atom).

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure*.

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