2,3-Dihydro-1,5-benzothiazepin-4(5H)-one

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.003 Å
R factor = 0.043
wR factor = 0.108
Data-to-parameter ratio = 14.0

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

The title compound, C_9H_9NOS, crystallizes with two independent molecules in the asymmetric unit. The bond lengths and angles in both molecules are within normal ranges. The crystal packing is stabilized by intermolecular N—H···O hydrogen bonds and van der Waals forces.

Comment

A number of 1,5-benzothiazepine derivatives have been shown to exhibit pharmacological properties as calcium antagonists (Rampe & Triggle, 1993), coronary vasodilators (Hirozumi *et al.*, 1991), antihypertensives (Hiroshi & Hirshi, 1992) and blood-platelet aggregation inhibitors (Hirozumi & Trunehiro, 1990). In this paper, we report the crystal structure of the title compound, (I).

(I)
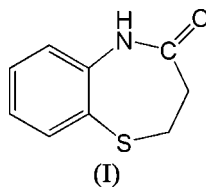
Compound (I) crystallizes with two independent molecules in the asymmetric unit (Fig. 1). The bond lengths and angles of the thiazepine ring (Table 1) compare well with those found in other structures containing this system (Schwartz *et al.*, 1992; Inoue *et al.*, 1991) extracted from the Cambridge Structural Database (Version 5.14; Allen, 2002). The thiazepinone ring system is found in the same twist-boat conformation in the two independent molecules.

Intermolecular N—H···O hydrogen bonds (Table 2) link symmetry-equivalent molecules into two independent chains extending along the *b* axis. The crystal packing (Fig. 2) is further stablized by van der Waals forces.

Experimental

Compound (I) was prepared by the reaction of 2-aminothiophenol with methyl 3-bromopropionate at 383 K, according to the method of Katritzky *et al.* (2001). Crystals suitable for X-ray diffraction were obtained by slow evaporation of a solution of the title compound in chloroform at room temperature over a period of one week.

Crystal data

C_9H_9NOS	$Z = 8$
$M_r = 179.23$	$D_x = 1.381$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 24.292$ (4) Å	$\mu = 0.32$ mm^{-1}
$b = 7.7382$ (12) Å	$T = 298$ (2) K
$c = 9.1824$ (15) Å	Block, colourless
$\beta = 92.509$ (2)°	$0.45 \times 0.31 \times 0.20$ mm
$V = 1724.4$ (5) Å^3	
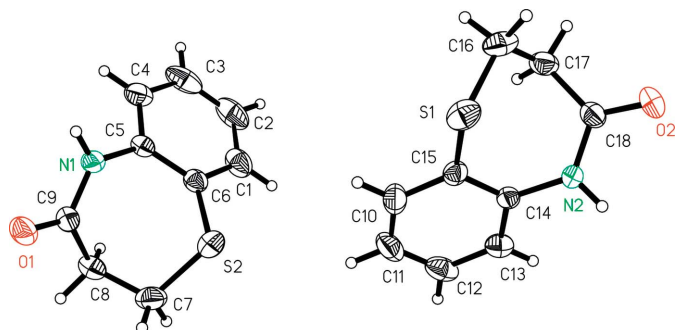
Figure 1
A view of the two independent molecules of (I), with displacement ellipsoids drawn at the 40% probability level.

Data collection

Bruker SMART CCD area-detector diffractometer	8354 measured reflections
φ and ω scans	3039 independent reflections
Absorption correction: multi-scan	2647 reflections with $I > 2\sigma(I)$
(*SADABS*; Sheldrick, 1996)	$R_{int} = 0.032$
$T_{min} = 0.869$, $T_{max} = 0.939$	$\theta_{max} = 25.0°$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0468P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.043$	$+ 0.6434P]$
$wR(F^2) = 0.108$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.08$	$(\Delta/\sigma)_{max} = 0.001$
3039 reflections	$\Delta\rho_{max} = 0.38$ e Å^{-3}
217 parameters	$\Delta\rho_{min} = -0.29$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

S1—C15	1.766 (2)	N1—C9	1.346 (3)
S1—C16	1.807 (2)	N1—C5	1.411 (2)
S2—C6	1.771 (2)	N2—C18	1.345 (3)
S2—C7	1.805 (2)	N2—C14	1.410 (2)
C15—S1—C16	104.07 (10)	C9—N1—C5	126.29 (17)
C6—S2—C7	103.49 (11)	C18—N2—C14	126.98 (17)

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1—H1A⋯O1^i	0.86	2.04	2.873 (2)	164
N2—H2A⋯O2^{ii}	0.86	2.01	2.834 (2)	161

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

All H atoms were placed in calculated positions, with C—H = 0.93 or 0.97 Å and N—H = 0.86 Å, and included in the final cycles of refinement using a riding model, with $U_{iso}(H) = 1.2U_{eq}$(parent atom).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve
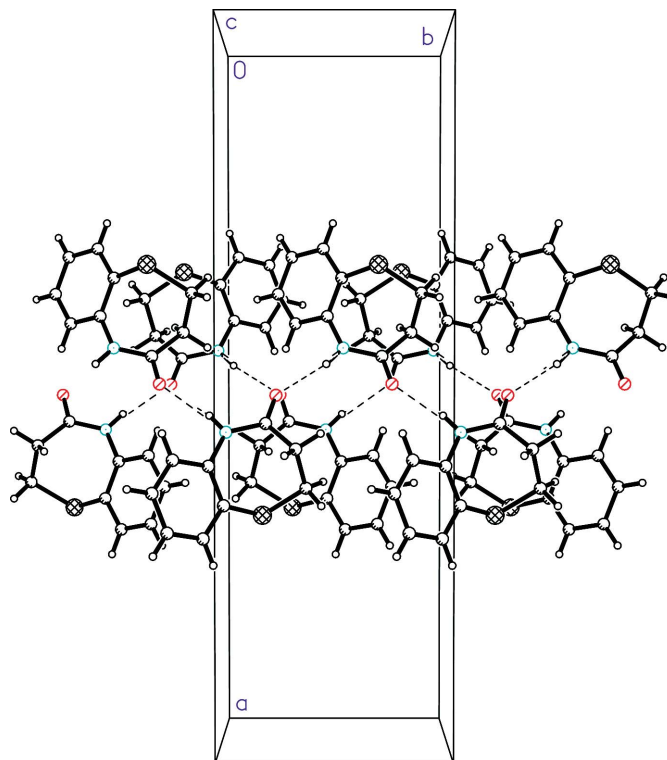
Figure 2
A packing diagram, viewed down the *c* axis. Intermolecular hydrogen bonds are shown as dashed lines.

structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Bruker (1998). *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (1999). *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Hiroshi, N. & Hirshi, N. (1992). Jpn. Kokai Tokyo Koho JP 03 258 721 (1992) [91258721] (Cl. A. 61K31/55), 19 November (1991); *Chem. Abstr.* **116**, 136275q.
Hirozumi, I., Mikihiko, K., Iomoki, H., Hisao, O. & Mikio, T. (1991). *J. Med. Chem.* **34**, 675–687.
Hirozumi, I. & Trunehiro, H. (1990). Fr. Demande Fr. 2623192 (1990) [Cl. CO7D 281/10], 19 May (1989); *Chem. Abstr.* **112**, 55929w.
Inoue, H., Konda, M., Hashiyama, T., Otsuka, H., Takahashi, K., Gaino, M., Date, T., Aoe, K., Takeda, M., Murata, S., Narita, H. & Nagao, T. (1991). *J. Med. Chem.* **34**, 675–687.
Katritzky, A. R., Odens, H. H. & Zhang, S. M. (2001). *J. Org. Chem.* **66**, 6792–6796.
Rampe, D. & Triggle, D. (1993). *J. Progr. Drug Res.* **40**, 191–205.
Schwartz, A., Madan, P. B., Mohacsi, E., O'Brien, J. P., Todaro, L. J. & Coffen, D. L. (1992). *J. Org. Chem.* **57**, 851–856.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.