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

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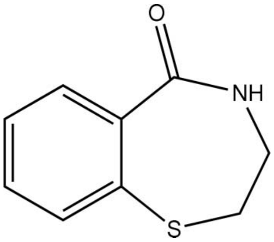
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.006 Å; R factor = 0.062; wR factor = 0.166; data-to-parameter ratio = 15.6.

In the molecule of the title compound, C_9H_9NOS, the seven-membered ring has a twist conformation. In the crystal structure, intermolecular N—H⋯O hydrogen bonds link the molecules into centrosymmetric dimers.

Related literature

For general background, see: Arya *et al.* (1977). For related literature, see: Ishibashi *et al.* (2001). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

C_9H_9NOS
M_r = 179.23
Orthorhombic, *Pbca*
a = 8.0510 (16) Å
b = 8.9580 (18) Å
c = 24.220 (5) Å
V = 1746.8 (6) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.32 mm^{-1}
T = 294 (2) K
0.20 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.939, T_{max} = 0.969
1704 measured reflections
1704 independent reflections
1089 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
3 standard reflections
frequency: 120 min
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.062
$wR(F^2)$ = 0.166
S = 1.02
1704 reflections
109 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N—H0A⋯O^{i}	0.86	2.05	2.824 (4)	149

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

Data collection: *CAD-4 Software* (Enraf–Nonius,1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo,1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Siemens,1996); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2393).

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supplementary materials

Acta Cryst. (2008). E**64**, o113 [**doi:10.1107/S1600536807062046**]

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

Z.-L. Chen, F. Hong and S.-Y. Zhao

Comment

The title compound, (I), is an important intermediate used in the synthesis of dipeptidyl peptidase-IV inhibitors, cysteine proteases inhibitors and antihypertensive agent (Arya *et al.*, 1977). As part of our ongoing studies in this area, we report herein its synthesis and crystal structure.

In the molecule of (I), (Fig. 1) the bond lengths and angles are within normal ranges (Allen *et al.*, 1987). Ring A (C3—C8) is, of course, planar, while ring B (S/N/C1—C3/C8/C9) is not planar and has a twisted conformation.

In the crystal structure, intermolecular N—H0A···O^{i} hydrogen bonds [H0A···O 2.05 Å, N···O 2.824 (3) Å and N—H0A···O 149.4°] [symmetry code: (i) $x + 1/2$, $1/2 - y$, $-z$] link the molecules into centrosymmetric dimers (Fig. 2), in which they seem to be effective in the stabilization of the structure.

Experimental

The title compound, (I), was prepared by the literature method with a minor change (Ishibashi *et al.*, 2001). 2-Mercaptobenzoic acid methyl ester (3.3 g, 19.6 mmol) was added to the solution of sodium (0.5 g, 22.0 mmol) in ethanol (20 ml). The mixture was stirred at room temperature for 10 min, and then 2-oxazolidinone (1.7 g, 19.8 mmol) was added. The mixture was heated under reflux for 6 h. The solvent was evaporated off, water (15 ml) was added to the residue, and the whole mixture was extracted with ethyl acetate (15 ml×3). The combined ester layer was dried with sodium sulfate and evaporated. The residue was recrystallized from ethanol and dried in vacuum at 323 K to give the title compound as a white solid (yield; 60%, m.p. 466–468 K) (Ishibashi *et al.*, 2001, m.p. 465–466 K). Crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an ethanol solution.

Refinement

H atoms were positioned geometrically, with N—H = 0.86 Å (for NH) and C—H = 0.93 and 0.97 Å for aromatic and methylene H, respectively, and constrained to ride on their parent atoms, with $U_{iso}(H) = 1.2U_{eq}(C,N)$.

Figures

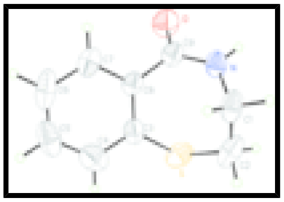

Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

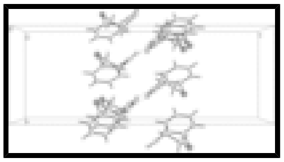

Fig. 2. A packing diagram of (I). Hydrogen bonds are shown as dashed lines.

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

Crystal data

C_9H_9NOS

$M_r = 179.23$

Orthorhombic, *Pbca*

Hall symbol: -P 2ac 2ab

$a = 8.0510\ (16)$ Å

$b = 8.9580\ (18)$ Å

$c = 24.220\ (5)$ Å

$V = 1746.8\ (6)$ Å^3

$Z = 8$

$F_{000} = 752$

$D_x = 1.363$ Mg m^{-3}

Mo *K*α radiation
λ = 0.71073 Å

Cell parameters from 25 reflections

θ = 9–13°

μ = 0.32 mm^{-1}

$T = 294\ (2)$ K

Block, colorless

0.20 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 294(2)$ K

ω/2θ scans

Absorption correction: ψ scan (North *et al.*, 1968)

$T_{min} = 0.939$, $T_{max} = 0.969$

1704 measured reflections

1704 independent reflections

1089 reflections with $I > 2\sigma(I)$

$R_{int} = 0.022$

$\theta_{max} = 26.0°$

$\theta_{min} = 1.7°$

$h = 0\rightarrow9$

$k = 0\rightarrow10$

$l = 0\rightarrow29$

3 standard reflections

every 120 min

intensity decay: none

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.062$

$wR(F^2) = 0.166$

$S = 1.02$

1704 reflections

109 parameters

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.060P)^2 + 2.7P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.23$ e Å^{-3}

$\Delta\rho_{min} = -0.22$ e Å^{-3}

Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S	0.20239 (14)	0.28682 (12)	0.17431 (4)	0.0620 (4)
O	−0.0306 (4)	0.2669 (4)	0.02305 (13)	0.0753 (10)
N	0.2218 (4)	0.3438 (3)	0.04867 (12)	0.0452 (8)
H0A	0.2651	0.2918	0.0225	0.054*
C1	0.3328 (4)	0.4320 (4)	0.08290 (17)	0.0507 (10)
H1A	0.2801	0.5269	0.0911	0.061*
H1B	0.4336	0.4525	0.0623	0.061*
C2	0.3779 (5)	0.3569 (5)	0.1361 (2)	0.0674 (13)
H2A	0.4376	0.4275	0.1591	0.081*
H2B	0.4524	0.2746	0.1282	0.081*
C3	0.0396 (4)	0.4103 (4)	0.15575 (16)	0.0439 (9)
C4	−0.0452 (5)	0.4849 (5)	0.19751 (19)	0.0633 (12)
H4A	−0.0085	0.4768	0.2338	0.076*
C5	−0.1832 (6)	0.5708 (5)	0.1857 (2)	0.0695 (13)
H5A	−0.2375	0.6212	0.2140	0.083*
C6	−0.2398 (5)	0.5822 (5)	0.1333 (2)	0.0710 (14)
H6A	−0.3327	0.6402	0.1255	0.085*
C7	−0.1587 (4)	0.5068 (4)	0.09133 (18)	0.0518 (10)
H7A	−0.1988	0.5141	0.0554	0.062*
C8	−0.0196 (4)	0.4211 (4)	0.10152 (14)	0.0377 (8)
C9	0.0579 (4)	0.3377 (4)	0.05503 (16)	0.0449 (9)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S	0.0642 (7)	0.0529 (7)	0.0691 (7)	0.0009 (6)	−0.0161 (5)	0.0134 (5)
O	0.0568 (18)	0.088 (2)	0.081 (2)	−0.0102 (17)	−0.0143 (16)	−0.0395 (18)
N	0.0378 (18)	0.0425 (17)	0.0553 (17)	0.0002 (15)	0.0038 (14)	−0.0121 (14)
C1	0.0335 (19)	0.040 (2)	0.079 (3)	−0.0057 (18)	0.0068 (18)	−0.014 (2)
C2	0.038 (2)	0.059 (3)	0.106 (4)	0.001 (2)	−0.013 (2)	0.000 (3)
C3	0.0356 (19)	0.0351 (19)	0.061 (2)	−0.0092 (17)	0.0057 (17)	−0.0101 (17)

C4	0.061 (3)	0.062 (3)	0.067 (3)	−0.024 (2)	0.010 (2)	−0.012 (2)
C5	0.052 (3)	0.060 (3)	0.097 (4)	−0.008 (2)	0.030 (3)	−0.024 (3)
C6	0.037 (2)	0.041 (2)	0.135 (4)	0.007 (2)	0.012 (3)	−0.007 (3)
C7	0.038 (2)	0.047 (2)	0.071 (2)	0.0017 (19)	0.0021 (19)	0.010 (2)
C8	0.0312 (17)	0.0319 (18)	0.050 (2)	−0.0033 (16)	−0.0032 (15)	−0.0037 (15)
C9	0.040 (2)	0.040 (2)	0.055 (2)	0.0005 (18)	−0.0045 (17)	−0.0031 (17)

Geometric parameters (Å, °)

S—C3	1.773 (4)	C3—C4	1.391 (6)
S—C2	1.802 (5)	C3—C8	1.401 (5)
N—C9	1.330 (4)	C4—C5	1.382 (6)
N—C1	1.453 (4)	C4—H4A	0.9300
N—H0A	0.8600	C5—C6	1.354 (7)
O—C9	1.229 (4)	C5—H5A	0.9300
C1—C2	1.499 (6)	C6—C7	1.384 (6)
C1—H1A	0.9700	C6—H6A	0.9300
C1—H1B	0.9700	C7—C8	1.380 (5)
C2—H2A	0.9700	C7—H7A	0.9300
C2—H2B	0.9700	C8—C9	1.488 (5)
C3—S—C2	103.42 (19)	C5—C4—C3	120.8 (4)
C9—N—C1	124.5 (3)	C5—C4—H4A	119.6
C9—N—H0A	117.8	C3—C4—H4A	119.6
C1—N—H0A	117.8	C6—C5—C4	120.5 (4)
N—C1—C2	113.3 (3)	C6—C5—H5A	119.8
N—C1—H1A	108.9	C4—C5—H5A	119.8
C2—C1—H1A	108.9	C5—C6—C7	119.5 (4)
N—C1—H1B	108.9	C5—C6—H6A	120.2
C2—C1—H1B	108.9	C7—C6—H6A	120.2
H1A—C1—H1B	107.7	C8—C7—C6	121.6 (4)
C1—C2—S	114.1 (3)	C8—C7—H7A	119.2
C1—C2—H2A	108.7	C6—C7—H7A	119.2
S—C2—H2A	108.7	C7—C8—C3	118.8 (3)
C1—C2—H2B	108.7	C7—C8—C9	119.0 (3)
S—C2—H2B	108.7	C3—C8—C9	122.2 (3)
H2A—C2—H2B	107.6	O—C9—N	121.5 (4)
C4—C3—C8	118.8 (4)	O—C9—C8	119.5 (3)
C4—C3—S	118.6 (3)	N—C9—C8	118.9 (3)
C8—C3—S	122.1 (3)		
C9—N—C1—C2	82.3 (5)	C6—C7—C8—C9	177.5 (4)
N—C1—C2—S	−49.9 (4)	C4—C3—C8—C7	0.8 (5)
C3—S—C2—C1	−29.6 (4)	S—C3—C8—C7	172.7 (3)
C2—S—C3—C4	−124.1 (3)	C4—C3—C8—C9	−176.4 (3)
C2—S—C3—C8	63.9 (3)	S—C3—C8—C9	−4.4 (5)
C8—C3—C4—C5	−1.4 (6)	C1—N—C9—O	176.3 (4)
S—C3—C4—C5	−173.6 (3)	C1—N—C9—C8	−2.7 (6)
C3—C4—C5—C6	1.0 (6)	C7—C8—C9—O	−45.4 (5)
C4—C5—C6—C7	0.1 (7)	C3—C8—C9—O	131.8 (4)
C5—C6—C7—C8	−0.7 (6)	C7—C8—C9—N	133.6 (4)

C6—C7—C8—C3	0.3 (6)	C3—C8—C9—N	−49.3 (5)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N—H0A···O^{i}	0.86	2.05	2.824 (4)	149

Symmetry codes: (i) −*x*, *y*+1/2, −*z*+1/2.

Fig. 1

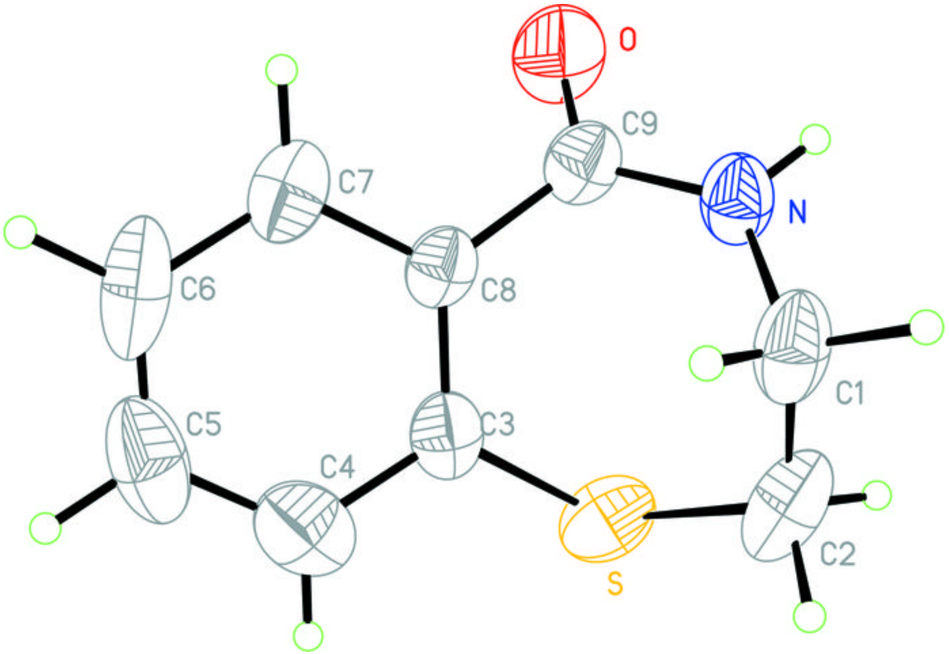

Fig. 2