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2,2′-Bi-1,3-thiazolidine

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.036
wR factor = 0.091
Data-to-parameter ratio = 12.5

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

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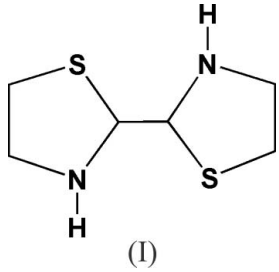
The title compound, $C_6H_{12}N_2S_2$, possesses a centre of symmetry with one-half of the molecule in the asymmetric unit. The crystal structure has intermolecular N—H···N and C—H···S contacts and intramolecular S···N interactions.

Comment

The condensation of glyoxal with α-amino-β-thiols results in the formation of 2,2′-bisthiazolidine (Jadamus *et al.*, 1964). Thiazolidines, compounds containing N and S in a five-membered ring, are of great interest because of the presence of this ring in the important antibiotic penicillin (Fife *et al.*, 1991). In addition, there are few X-ray diffraction studies for sulfur–nitrogen-containing ligands. These types of derivatives have been used for the preparation of nickel(II) chelates (Jadamus *et al.*, 1964), and *N*,*N*′-methylene-bisthiazolidine analogues are also potentially useful as chiral auxiliaries and ligands for asymmetric synthesis (Díaz *et al.*, 2001). Against this background, we present here the crystal structure of the title compound, (I).



(I)

The five-membered ring in compound (I) shows an envelope conformation, with a puckering parameter (Cremer & Pople, 1975) $\varphi = 67.0\ (3)^\circ$, similar to the value reported for the heterocyclic ring in bi(benzothiazolyl) ($\varphi = 37.2^\circ$) (Farfán *et al.*, 1994). The similarities in the conformation can be attributed to the fact that, in both cases, the heterocyclic rings are not substituted. In contrast, the substituted bis(dimethyl-2,3 benzothiazoline) shows a twisted conformation ($\varphi = 27.0^\circ$) for the five-membered ring (Miler-Srenger *et al.*, 1973).

Owing to the presence of an inversion centre in (I), the N [N3—C2—C2^i—N3^i = 180°] and S atoms [S1—C2—C2^i—S1^i = −180°; symmetry code as in Table 1] are antiperiplanar. The C2—C2^i, C4—N3 and C2—N3 bond distances (Table 1) are similar to the average values reported for Csp^3—Csp^3 in cyclopentane and (Csp^3)$_2$—NH (Nsp^3: pyramidal) (1.543 and 1.469 Å). It is worth mentioning that the C2—S1 bond distance is abnormally large compared with the standard Csp^3—S bond distance (1.817 Å; Allen *et al.*, 1987). The

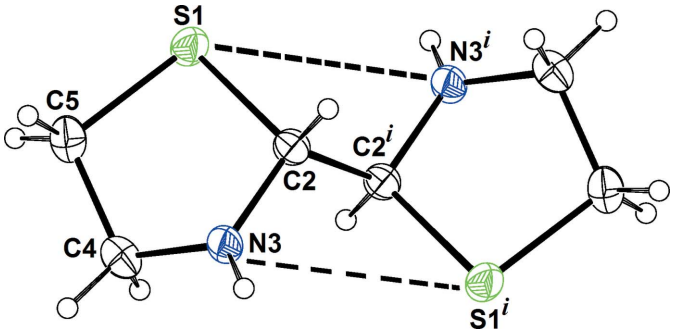


Figure 1
View of (I), with the atomic numbering scheme. Displacement ellipsoids are drawn at 30% probability level. Dashed lines indicate intramolecular N···S interactions [symmetry code: (i) $-x, 1-y, -z$].

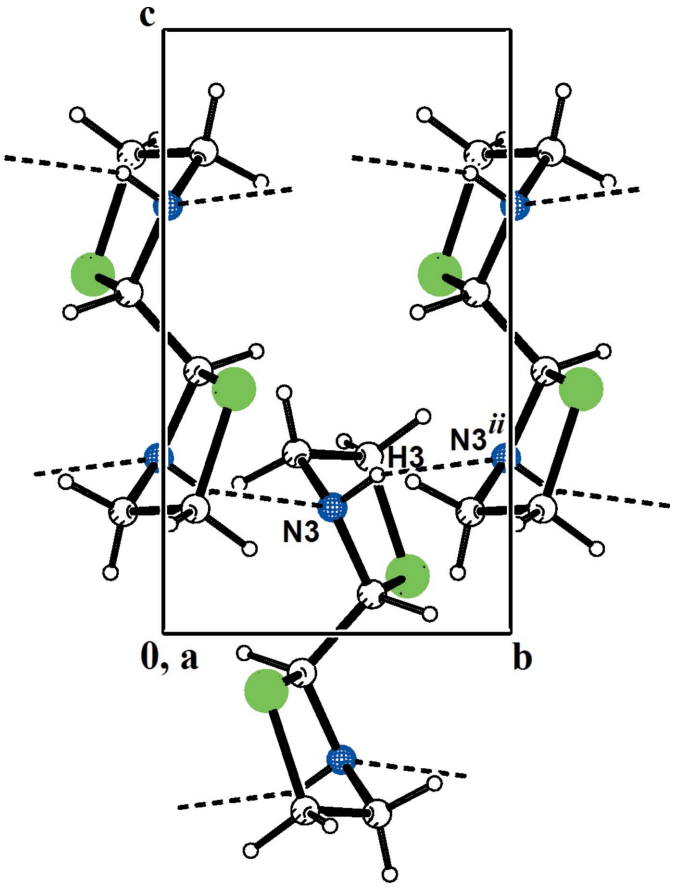


Figure 2
Intermolecular N3–H3···N3^{ii} interactions (dashed lines) in (I) [symmetry code: (ii) $\frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$].

increase in C—S bond distance in substituted benzothiazolidines with methyl groups on the N atom could be attributed to the electron-donating nature of the methyl group that leads to a perturbation in the C—S bond in order to decrease the repulsive energy of the electrons. However, comparison with substituted benzothiazolidine derivatives allows one to conclude that the deformation in the C—S bond is characteristic for the benzothiazoline molecule (Miler-Srenger *et al.*, 1973). In the case of compound (I), with unsubstituted N

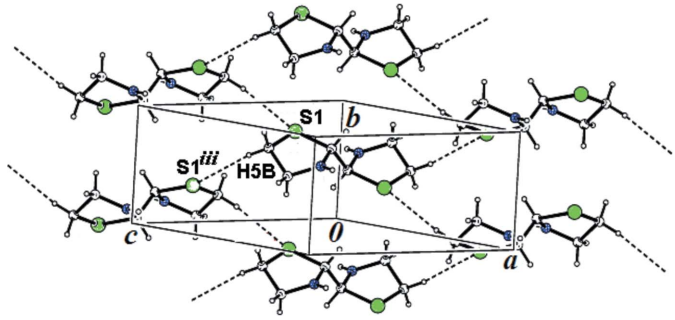


Figure 3
Intermolecular S1···H5B^{iii} interactions (dashed lines) in (I) [symmetry code: (iii) $-\frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$].

atoms, the increase in C—S bond length may be attributed to an N3···S1^{i} intramolecular interaction [3.121 (2) Å]. The bond angles at N3, S1 and C2 (Table 1) are in agreement with the observed tendencies for the bond angles in bi(benzothiazolyl) analogues, which show values of 111.5° for C—N—C, 123° for C—N—H, 90.6 for C—S—C and 104.6° for S—C—N (Farfán *et al.*, 1994).

Compound (I) shows intermolecular N—H···N and C—H···S interactions (Fig. 2 and Table 2), with H···acceptor distances which are less than the sum of the van der Waals radii (Bondi, 1964). These interactions are propagated along [$\bar{1}0\bar{1}$]. The combination of these interactions with the molecular centrosymmetry generates a two-dimensional network (Fig. 3).

Experimental

Compound (I) was prepared from glyoxal and α-amino-β-thiol (m.p. 438–440 K; literature value 452–454 K; Jadamus *et al.*, 1964). Crystals suitable for X-ray crystallography were grown from chloroform at room temperature by slow evaporation. MS, *m*/*z* (%): 177 (M^{+}, 1); ^{1}H NMR ($CDCl_3$, DMSO/D_2O, 500 MHz, δ, p.p.m.): 4.87 (1H, *s*, H2), 3.51 (1H, *ddd*, *J* = 12.4, 6.1 and 4.0 Hz, H4*B*), 3.05 (1H, *ddd*, *J* = 12.4, 8.2 and 6.2 Hz, H4*A*), 2.95 (1H, *ddd*, *J* = 10.0, 6.20 and 4.0 Hz, H5*B*), 2.87 (1H, *ddd*, *J* = 10.0, 8.2 and 6.1 Hz, H5*A*); ^{13}C NMR ($CDCl_3$, 67.93 MHz, δ, p.p.m.): 74.0 (C2), 53.0 (C4), 35.4 (C5).

Crystal data

$C_6H_{12}N_2S_2$
M_r = 176.30
Monoclinic, $P2_1/n$
a = 9.4931 (6) Å
b = 5.0070 (4) Å
c = 9.5942 (8) Å
β = 114.844 (3)°
V = 413.83 (6) Å^3
Z = 2
D_x = 1.415 Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 600 reflections
θ = 20–25°
μ = 0.57 mm^{-1}
T = 293 (2) K
Block, colourless
0.15 × 0.10 × 0.08 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ and ω scans
Absorption correction: none
1332 measured reflections
878 independent reflections
785 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 27.5°
h = −10 → 10
k = −6 → 4
l = −12 → 12

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0315P)^2 + 0.2832P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.036$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.091$	$\Delta\rho_{max} = 0.62$ e Å^{-3}
$S = 1.07$	$\Delta\rho_{min} = -0.29$ e Å^{-3}
878 reflections	
70 parameters	
All H-atom parameters refined	

Table 1
Selected geometric parameters (Å, °).

C2—C2^{i}	1.519 (3)	N3—C2	1.455 (2)
S1—C2	1.869 (2)	N3—C4	1.465 (3)
S1—C5	1.8127 (19)		
C2—S1—C5	92.59 (11)	N3—C2—C2^{i}	111.96 (17)
C2—N3—C4	110.20 (17)	N3—C4—C5	108.04 (19)
S1—C2—C2^{i}	108.27 (14)	S1—C5—C4	103.97 (14)
S1—C2—N3	107.22 (13)		

Symmetry code: (i) $-x, -y+1, -z$.

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3···N3^{ii}	0.89 (3)	2.36 (3)	3.183 (3)	153 (2)
C5—H5B···S1^{iii}	0.95 (3)	2.91 (3)	3.842 (3)	160.8 (6)

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

All H atoms were found in difference Fourier maps and refined freely [C—H = 0.94 (3)–1.06 (3) Å].

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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