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Benzyl *N*-[1-(thiophen-2-yl)ethylidene)]-hydrazinecarbodithioate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.039
wR factor = 0.113
Data-to-parameter ratio = 19.7

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

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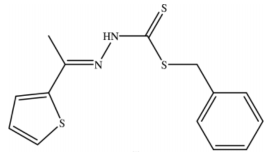
In the title compound, $C_{14}H_{14}N_2S_3$, the benzyl and 1-(thiophen-2-yl)ethylideneamine groups are *cis* and *trans* to the thiono S atom across the C—S and C—N bonds, respectively. The dithiocarbazate plane makes dihedral angles of 9.14 (6) and 85.40 (8)° with the thiophenemethyleneamine and benzyl fragments, respectively. In the crystal structure, the molecules are linked by N—H···S hydrogen bonds between the amino group and the thiono S atoms and are arranged as dimers parallel to *ab* face.

Comment

As in the majority of dithiocarbazate derivatives such as *S*-benzyldithiocarbazate (Shanmuga Sundra Raj *et al.*, 2000) and *S*-methyl *β*-*N*-(methylacetyl)methylenedithiocarbazate (Tarafder *et al.*, 2001), the title compound, (I) (Fig. 1) maintains the preferred conformation, which is *cis* for the benzyl group across the C—S bond, and *trans* for the 1-thiophen-2-yl-ethylideneamine (S3/C9/C10/C11/C12/C13/C14/N2) group across the C—N bond, both relative to the terminal thiono S atom. The C9—N2, N2—N1, N1—C8, C8—S2 and C8—S1 bond lengths [1.276 (2), 1.3765 (19), 1.342 (2), 1.6503 (17) and 1.7525 (17) Å, respectively] are typical of Schiff base compounds of the dithiocarbazate system and are within normal ranges (Allen *et al.*, 1987).



(I)

There are two intramolecular interactions C5—H5···S1 and C7—H7*B*···S2, forming two pseudo-five-membered rings S1···H5—C5—C6—C7 and S2···H7*B*—C7—S1—C8, respectively. In the crystal structure, the molecules are packed as dimers, *via* weak N1—H1*A*···S2^{i} interactions (symmetry code as in Table 2), and arranged parallel to the *ab* face. (Fig. 2).

Experimental

S-benzyldithiocarbazate (1.98 g, 0.01 mol), prepared as previously described (Shanmuga Sundra Raj *et al.*, 2000), was dissolved in absolute ethanol (35 ml). To this solution was added a solution of 2-acetylthiophene (1.26 g, 0.01 mol) in absolute ethanol (20 ml). The mixture was heated and continuously stirred for 15 min and allowed to stand until bright yellow crystals formed. The crystals were filtered, washed with ethanol and dried *in vacuo* over P_2O_5. Suitable crystals for X-ray investigation were obtained by recrystallization from ethanol. Yield: 72.6%; m.p 404–405 K; CHN found: C 54.55, H 4.54, N

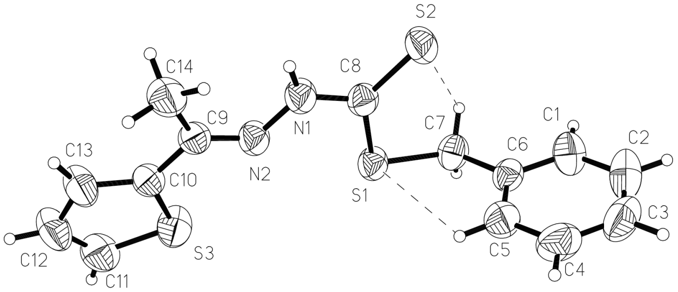


Figure 1
Molecular structure of the title compound, with 50% probability displacement ellipsoids. Intramolecular hydrogen bonds are shown as dashed lines.

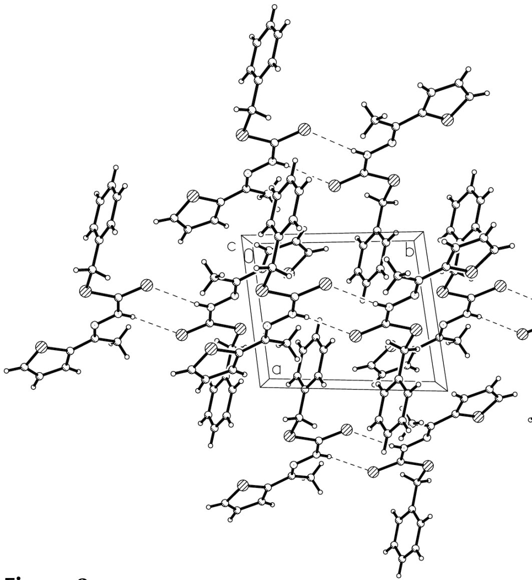


Figure 2
Packing diagram of (I), viewed down the *c* axis. Dashed lines denote the N—H···S hydrogen bonds.

9.38, S 31.28%; calculated for $C_{14}H_{15}N_2S_3$: C 54.97, H 4.60, N 9.14, S 31.39%.

Crystal data

$C_{14}H_{14}N_2S_3$
$M_r = 306.45$
Triclinic, $P\overline{1}$
$a = 7.8297$ (13) Å
$b = 9.8086$ (16) Å
$c = 11.0310$ (18) Å
$\alpha = 65.369$ (3)°
$\beta = 75.843$ (3)°
$\gamma = 77.150$ (3)°
$V = 739.7$ (2) Å^3

$Z = 2$
$D_x = 1.376$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5262 reflections
$\theta = 2.1$–27.5°
$\mu = 0.49$ mm^{-1}
$T = 293$ (2) K
Block, yellow
0.46 × 0.30 × 0.21 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.807$, $T_{\max} = 0.905$
9682 measured reflections

3405 independent reflections
3012 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.016$
$\theta_{\max} = 27.5°$
$h = -10 \rightarrow 10$
$k = -12 \rightarrow 12$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.113$
$S = 1.04$
3405 reflections
173 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.063P)^2 + 0.1918P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.31$ e Å^{-3}
$\Delta\rho_{\min} = -0.29$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—C8	1.7525 (17)	N1—C8	1.342 (2)
S1—C7	1.8009 (18)	N1—N2	1.3765 (19)
S2—C8	1.6503 (17)	N2—C9	1.276 (2)
C8—S1—C7	101.92 (8)	N1—C8—S1	112.89 (12)
C9—N2—N1	118.42 (14)	S2—C8—S1	125.60 (10)
N1—C8—S2	121.51 (13)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···S2$^{\rm i}$	0.86	2.81	3.6450 (17)	165
C5—H5···S1	0.93	2.76	3.164 (2)	107
C7—H7*B*···S2	0.97	2.73	3.114 (2)	104

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

After their location in a difference Fourier map, all H atoms were included in the refinement in geometrically determined positions, and allowed to ride on the parent C or N atoms, with C—H = 0.97 Å and N—H = 0.89 Å.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL, PARST* (Nardelli, 1995) and *PLATON* (Spek, 1990).

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