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2-Methyl-3-thiosemicarbazide

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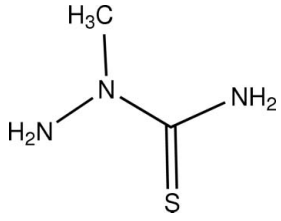
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(N–C) = 0.003 Å; R factor = 0.037; wR factor = 0.088; data-to-parameter ratio = 13.4.

The title compound, $C_2H_7N_3S$, exhibits an *anti* arrangement between the thione S atom and the hydrazine N atom. The thiosemicarbazide molecules are interconnected into a three-dimensional hydrogen-bonded network.

Related literature

For general background, see: Allen *et al.* (1997); Casas *et al.* (2000).

For related structures, see: Rapheal *et al.* (2005); West *et al.*, (2001); Castineiras *et al.* (2000); Lynch & McClenaghan (2000); Valente *et al.* (1998); Chattopadhyay *et al.* (1987, 1991); Andreetti *et al.* (1970).



Experimental

Crystal data

$C_2H_7N_3S$
M_r = 105.17
Monoclinic, $P2_1/n$
a = 8.606 (2) Å
b = 5.940 (1) Å
c = 9.843 (2) Å
β = 98.641 (3)°
V = 497.46 (17) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.50 mm^{-1}
T = 298 (2) K
0.45 × 0.33 × 0.19 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: analytical (*XPREP* in *SHELXTL*; Sheldrick, 2000)
T_{min} = 0.814, T_{max} = 0.915
3668 measured reflections
912 independent reflections
805 reflections with $I > 2\sigma(I)$
R_{int} = 0.080

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.088
S = 1.07
912 reflections
68 parameters
6 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···S1^{i}	0.88 (2)	2.90 (2)	3.636 (2)	142 (2)
N1—H1B···S1^{ii}	0.87 (2)	2.78 (2)	3.625 (2)	166 (3)
N3—H3A···N1	0.87 (2)	2.17 (3)	2.576 (3)	108 (2)
N3—H3A···S1^{iii}	0.87 (2)	2.88 (2)	3.430 (2)	123 (2)
N3—H3B···S1^{iv}	0.86 (2)	2.62 (2)	3.460 (2)	165 (2)

Symmetry codes: (i) $x+\frac{1}{2}, -y+\frac{3}{2}, z+\frac{1}{2}$; (ii) $-x+\frac{5}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$; (iii) $x, y+1, z$; (iv) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2007).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2229).

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supplementary materials

Acta Cryst. (2007). E**63**, o4082 [**doi:10.1107/S1600536807045102**]

2-Methyl-3-thiosemicarbazide

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Comment

As part of a study on the supramolecular structure of thiosemicarbazones and its metal complexes we found necessary for comparisons to know the crystal structure of the 2-methyl-3-thiosemicarbazide, (I).

The molecule (I) presents an anti arrangement between the S atom and the hydrazinic N atom with respect to the N2–C1 bond, as shown in Fig. 1. The molecule is nearly planar with the C2 [0.057 (2) Å] and N3 [0.053 (2) Å] showing the greater deviation from the mean plane of the molecule. The anti conformation is reinforced by an intramolecular N3–H3A ··· N1.

The two H atoms on N3 form N–H···S hydrogen bonds generating tapes (Fig. 2). These tapes are interconected into a 3-dimmensional H-bonded structure through N1–HA···S and N1–HB···S H-bonds (Fig. 3).

Experimental

Crystals of I were obtained directly from the botle of the commercial product (Aldrich).

Refinement

C-bound H atoms were placed in geometrically idealized positions and refined using the riding model, with C–H = 0.96 Å. H atoms on N atoms were located in a difference map and their positional parameters were refined. The U_{iso}(H) values were set at 1.5 E_{eq} for methyl H atoms and 1.2 U_{eq}(N) for N–H atoms.

Figures

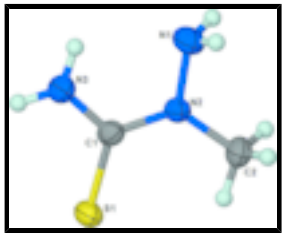

Fig. 1. Molecular structure of I, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radius.

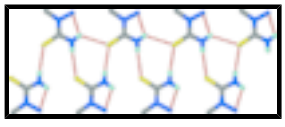

Fig. 2. Hydrogen bonded tapes observed in the crystal structure of (I). Hydrogen bonds are shown as dashed lines.

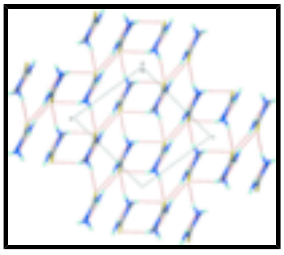

Fig. 3. The crystal packing of (I). Hydrogen bonds are shown as dashed lines.

2-Methyl-3-thiosemicarbazide

Crystal data

$C_2H_7N_3S$	F_{000} = 224
M_r = 105.17	D_x = 1.404 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 2596 reflections
a = 8.606 (2) Å	θ = 2.9–25.4º
b = 5.940 (1) Å	μ = 0.50 mm^{-1}
c = 9.843 (2) Å	T = 298 (2) K
β = 98.641 (3)º	Prism, clear colourless
V = 497.46 (17) Å^3	0.45 × 0.33 × 0.19 mm
Z = 4	

Data collection

Bruker SMART APEX CCD diffractometer	912 independent reflections
Radiation source: fine-focus sealed tube	805 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.080
Detector resolution: 0.661 pixels mm^{-1}	θ_{max} = 25.4º
T = 298(2) K	θ_{min} = 2.9º
ω–scans	h = −10→10
Absorption correction: analytical (XPREP in SHELXTL; Sheldrick, 2000)	k = −7→7
T_{min} = 0.814, T_{max} = 0.915	l = −11→11
3668 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.037	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.088	$w = 1/[\sigma^2(F_o^2) + (0.0392P)^2 + 0.0728P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.07	$(\Delta/\sigma)_{max} < 0.001$

912 reflections	$\Delta\rho_{max}$ = 0.25 e Å^{-3}
68 parameters	$\Delta\rho_{min}$ = −0.20 e Å^{-3}
6 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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}
S1	0.94085 (5)	0.40767 (8)	0.26511 (6)	0.0435 (2)
N1	1.2493 (2)	0.8483 (3)	0.4444 (2)	0.0470 (5)
H1A	1.279 (3)	0.839 (4)	0.5340 (19)	0.070*
H1B	1.335 (3)	0.868 (4)	0.409 (3)	0.070*
N2	1.18369 (17)	0.6403 (2)	0.39541 (17)	0.0360 (4)
N3	0.9600 (2)	0.8401 (3)	0.3293 (2)	0.0454 (5)
H3A	1.014 (3)	0.958 (4)	0.363 (3)	0.068*
H3B	0.863 (2)	0.845 (4)	0.291 (3)	0.068*
C1	1.0337 (2)	0.6427 (3)	0.33555 (19)	0.0332 (4)
C2	1.2784 (2)	0.4402 (3)	0.4250 (2)	0.0452 (5)
H2A	1.2199	0.3108	0.3880	0.068*
H2B	1.3059	0.4235	0.5227	0.068*
H2C	1.3724	0.4533	0.3840	0.068*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0331 (3)	0.0333 (3)	0.0622 (4)	−0.00350 (19)	0.0011 (2)	−0.0053 (2)
N1	0.0418 (10)	0.0371 (10)	0.0599 (12)	−0.0072 (7)	0.0007 (9)	−0.0092 (8)
N2	0.0307 (8)	0.0276 (8)	0.0486 (10)	−0.0008 (6)	0.0029 (7)	−0.0021 (7)
N3	0.0337 (9)	0.0314 (9)	0.0694 (13)	0.0038 (7)	0.0021 (8)	−0.0028 (8)
C1	0.0306 (10)	0.0299 (10)	0.0405 (10)	0.0002 (7)	0.0102 (8)	0.0023 (7)
C2	0.0354 (10)	0.0385 (12)	0.0596 (14)	0.0067 (8)	−0.0003 (9)	0.0009 (9)

Geometric parameters (Å, °)

S1—C1	1.704 (3)	N3—C1	1.330 (2)

N1—N2	1.413 (2)	N3—H3A	0.87 (2)
N1—H1A	0.88 (2)	N3—H3B	0.86 (2)
N1—H1B	0.87 (2)	C2—H2A	0.9600
N2—C1	1.336 (2)	C2—H2B	0.9600
N2—C2	1.446 (2)	C2—H2C	0.9600
N2—N1—H1A	109 (2)	N3—C1—N2	116.9 (2)
N2—N1—H1B	107 (2)	N3—C1—S1	120.8 (2)
H1A—N1—H1B	106 (3)	N2—C1—S1	122.2 (1)
C1—N2—N1	116.8 (1)	N2—C2—H2A	109.5
C1—N2—C2	125.2 (2)	N2—C2—H2B	109.5
N1—N2—C2	117.8 (2)	H2A—C2—H2B	109.5
C1—N3—H3A	117 (2)	N2—C2—H2C	109.5
C1—N3—H3B	118 (2)	H2A—C2—H2C	109.5
H3A—N3—H3B	124 (2)	H2B—C2—H2C	109.5

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1A···S1^{i}	0.88 (2)	2.90 (2)	3.636 (2)	142 (2)
N1—H1B···S1^{ii}	0.87 (2)	2.78 (2)	3.625 (2)	166 (3)
N3—H3A···N1	0.87 (2)	2.17 (3)	2.576 (3)	108 (2)
N3—H3A···S1^{iii}	0.87 (2)	2.88 (2)	3.430 (2)	123 (2)
N3—H3B···S1^{iv}	0.86 (2)	2.62 (2)	3.460 (2)	165 (2)

Symmetry codes: (i) *x*+1/2, −*y*+3/2, *z*+1/2; (ii) −*x*+5/2, *y*+1/2, −*z*+1/2; (iii) *x*, *y*+1, *z*; (iv) −*x*+3/2, *y*+1/2, −*z*+1/2.

Fig. 1

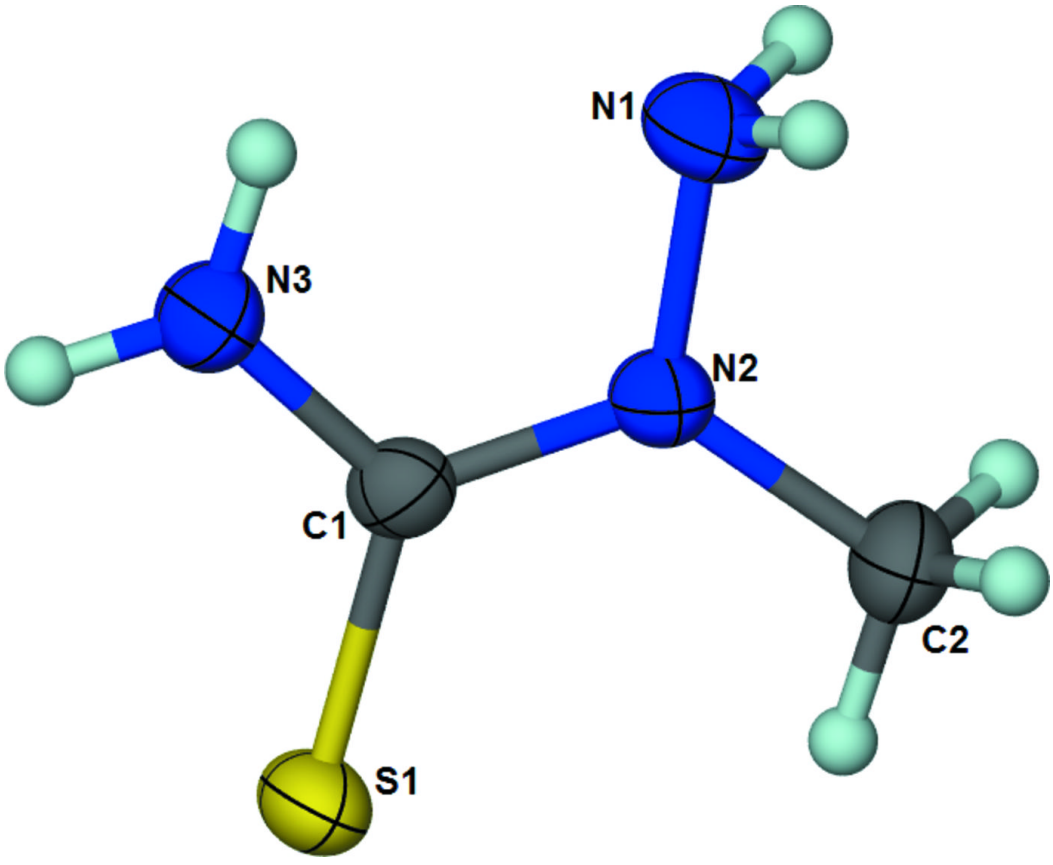

Fig. 2

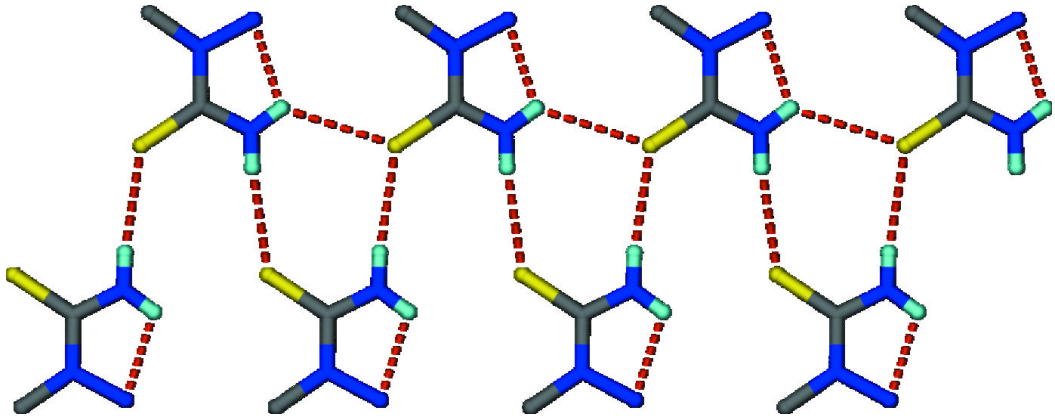

Fig. 3