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2-Acetylpyrazine 4-methylthiosemicarbazone

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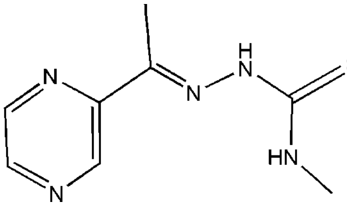
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.036; wR factor = 0.100; data-to-parameter ratio = 14.9.

The title compound, $C_8H_{11}N_5S$, has been prepared by the reaction of 2-acetylpyrazine with 4-methyl-3-thiosemicarbazide. It exists in the thione form and adopts the *E* configuration. The molecules are connected by the intermolecular N—H···N and N—H···S interactions.

Related literature

For related literature, see: Hong *et al.* (2004); Latheef *et al.* (2006); Liberta & West (1992); Mendes *et al.* (2001); Padhye & Kauffman (1985).



Experimental

Crystal data

$C_8H_{11}N_5S$
M_r = 209.28
Monoclinic, $P2_1/c$
a = 9.870 (8) Å
b = 5.976 (5) Å
c = 17.517 (14) Å
β = 91.251 (9)°
V = 1032.8 (14) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.28 mm^{-1}
T = 296 (2) K
0.20 × 0.18 × 0.16 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: none
9944 measured reflections
1919 independent reflections
1595 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.100
S = 1.05
1919 reflections
129 parameters
H-atom parameters constrained
$\Delta\rho_{\rm max}$ = 0.22 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.19 e Å^{-3}

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*D*···N4$^{\rm i}$	0.86	2.42	3.137 (3)	141
N2—H2*A*···S1$^{\rm ii}$	0.86	2.77	3.588 (3)	161

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2513).

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supplementary materials

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2-Acetylpyrazine 4-methylthiosemicarbazone

J. Zhou, Y.-X. Wang, C.-L. Chen and **M.-X. Li**

Comment

Thiosemicarbazone and its derivatives have attracted interest in recent years due to their beneficial biological applications (Padhye & Kauffman, 1985). The presence of alkyl groups at the terminal N(4) position can increase the biological activity (Liberta & West, 1992). So we report here the crystal structure of *N*(4)-methyl thiosemicarbazones derived from 2-acetylpyrazine.

The geometry of the title compound (I) is well planar (Fig. 1). The molecular exists in the E conformation about the C3—N3 bond as confirmed by the C5—C3—N3—N2 torsion angle of 179.6 °. The C—S bond distance of 1.679 (2) Å, which is much short than C—S single bond (Latheef *et al.*, 2006), shows that the title compound adopts the thione form. The bond length of C3—N3 is 1.283 (2) Å, which is within the range of typical bond length of C═N double bond. The bond length of N2—N3 is 1.368 (2) Å, accepted as typical for a single N—N bond, and in accordance with those of other thiosemicarbazone (Mendes *et al.*, 2001; Hong *et al.*, 2004).

In the crystal packing, the molecules are connected through an extended network of intermolecular hydrogen bonds involving the nitrogen atoms N1, N2, N4 and sulfur atom S1.

Experimental

All reagents were commercially available and of analytical grade. 2-Acetylpyrazine (0.24 g, 2 mmol) and 4-methyl-3-thiosemicarbazide (0.21 g, 2 mmol) were mixed in ethanol (30 ml). Eight drops of acetic acid were added and the solution was refluxed for 4 h. Crystals of (I) suitable for X-ray diffraction analysis were obtained from the filtrate by slow evaporation at room temperature.

Refinement

All H atoms were positioned geometrically and refined as riding with C—H = 0.96 Å (methyl) or 0.93 Å (aromatic), N—H = 0.86 Å and with $U_{iso}(H) = 1.2U_{eq}(C, N)$ or $1.5U_{eq}(C)$ for methyl groups.

Figures

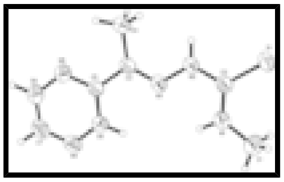

Fig. 1. The molecular structure of (I), showing atom displacement ellipsoids drawn at the 50% probability level.

2-Acetylpyrazine 4-methylthiosemicarbazone

Crystal data

$C_8H_{11}N_5S$	$F_{000} = 440$
$M_r = 209.28$	$D_x = 1.346$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 3140 reflections
$a = 9.870$ (8) Å	$\theta = 2.3$–26.0°
$b = 5.976$ (5) Å	$\mu = 0.28$ mm^{-1}
$c = 17.517$ (14) Å	$T = 296$ (2) K
$\beta = 91.251$ (9)°	Block, colourless
$V = 1032.8$ (14) Å^3	0.20 × 0.18 × 0.16 mm
$Z = 4$	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	1595 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.028$
Monochromator: graphite	$\theta_{max} = 25.5$°
$T = 296(2)$ K	$\theta_{min} = 2.1$°
0.3° wide ω scans	$h = -11 \rightarrow 11$
Absorption correction: none	$k = -7 \rightarrow 7$
9944 measured reflections	$l = -21 \rightarrow 21$
1919 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.036$	H-atom parameters constrained
$wR(F^2) = 0.100$	$w = 1/[\sigma^2(F_o^2) + (0.0519P)^2 + 0.2669P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{max} < 0.001$
1919 reflections	$\Delta\rho_{max} = 0.22$ e Å^{-3}
129 parameters	$\Delta\rho_{min} = -0.19$ 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 > 2sigma(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	0.53153 (5)	0.14546 (9)	0.39197 (3)	0.0578 (2)
C1	0.3602 (2)	0.1901 (4)	0.24436 (12)	0.0638 (6)
H1A	0.2945	0.1858	0.2031	0.096*
H1B	0.4483	0.1567	0.2252	0.096*
H1C	0.3610	0.3365	0.2669	0.096*
C2	0.39403 (17)	−0.0043 (3)	0.36625 (9)	0.0403 (4)
C3	0.20657 (16)	−0.4638 (3)	0.43186 (9)	0.0381 (4)
C4	0.27288 (19)	−0.5396 (3)	0.50496 (10)	0.0517 (5)
H4A	0.3690	−0.5166	0.5026	0.078*
H4B	0.2547	−0.6958	0.5125	0.078*
H4C	0.2376	−0.4552	0.5467	0.078*
C5	0.08835 (16)	−0.5912 (3)	0.40117 (9)	0.0373 (4)
C6	0.01556 (17)	−0.5220 (3)	0.33622 (9)	0.0457 (4)
H6A	0.0401	−0.3887	0.3129	0.055*
C7	−0.1190 (2)	−0.8254 (3)	0.34285 (11)	0.0549 (5)
H7A	−0.1901	−0.9129	0.3240	0.066*
C8	−0.0499 (2)	−0.8930 (4)	0.40744 (12)	0.0626 (6)
H8A	−0.0764	−1.0244	0.4313	0.075*
N1	0.32507 (15)	0.0258 (3)	0.30149 (8)	0.0480 (4)
H1D	0.2552	−0.0570	0.2928	0.058*
N2	0.35053 (14)	−0.1701 (2)	0.41299 (8)	0.0453 (4)
H2A	0.3907	−0.1941	0.4563	0.054*
N3	0.24209 (14)	−0.2982 (2)	0.39019 (8)	0.0416 (4)
N4	−0.08728 (15)	−0.6382 (3)	0.30628 (9)	0.0517 (4)
N5	0.05389 (16)	−0.7775 (3)	0.43754 (9)	0.0536 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0550 (3)	0.0710 (4)	0.0472 (3)	−0.0316 (3)	−0.0039 (2)	−0.0010 (2)
C1	0.0728 (14)	0.0604 (13)	0.0579 (12)	−0.0125 (11)	−0.0085 (10)	0.0176 (10)
C2	0.0397 (9)	0.0426 (10)	0.0385 (9)	−0.0058 (7)	0.0000 (7)	−0.0041 (7)
C3	0.0383 (9)	0.0405 (9)	0.0352 (8)	−0.0057 (7)	−0.0026 (7)	−0.0030 (7)
C4	0.0545 (11)	0.0562 (12)	0.0439 (10)	−0.0144 (9)	−0.0136 (8)	0.0051 (9)
C5	0.0384 (9)	0.0394 (9)	0.0339 (8)	−0.0053 (7)	−0.0001 (7)	−0.0016 (7)
C6	0.0470 (10)	0.0499 (11)	0.0397 (9)	−0.0115 (8)	−0.0073 (8)	0.0044 (8)
C7	0.0489 (11)	0.0626 (13)	0.0532 (11)	−0.0211 (9)	−0.0035 (9)	−0.0084 (10)

C8	0.0685 (14)	0.0562 (13)	0.0627 (13)	−0.0295 (11)	−0.0104 (10)	0.0107 (10)
N1	0.0479 (9)	0.0490 (9)	0.0466 (9)	−0.0134 (7)	−0.0081 (7)	0.0070 (7)
N2	0.0457 (8)	0.0504 (9)	0.0393 (8)	−0.0180 (7)	−0.0099 (6)	0.0051 (7)
N3	0.0392 (8)	0.0452 (8)	0.0402 (8)	−0.0113 (6)	−0.0050 (6)	−0.0007 (6)
N4	0.0468 (9)	0.0641 (11)	0.0435 (8)	−0.0125 (8)	−0.0099 (7)	−0.0018 (8)
N5	0.0572 (10)	0.0522 (9)	0.0509 (9)	−0.0197 (8)	−0.0109 (7)	0.0117 (8)

Geometric parameters (Å, °)

S1—C2	1.6789 (19)	C5—N5	1.331 (2)
C1—N1	1.449 (2)	C5—C6	1.395 (2)
C1—H1A	0.9600	C6—N4	1.328 (2)
C1—H1B	0.9600	C6—H6A	0.9300
C1—H1C	0.9600	C7—N4	1.330 (3)
C2—N1	1.322 (2)	C7—C8	1.369 (3)
C2—N2	1.361 (2)	C7—H7A	0.9300
C3—N3	1.283 (2)	C8—N5	1.334 (2)
C3—C5	1.484 (2)	C8—H8A	0.9300
C3—C4	1.496 (2)	N1—H1D	0.8600
C4—H4A	0.9600	N2—N3	1.368 (2)
C4—H4B	0.9600	N2—H2A	0.8600
C4—H4C	0.9600		
N1—C1—H1A	109.5	C6—C5—C3	122.00 (15)
N1—C1—H1B	109.5	N4—C6—C5	122.84 (17)
H1A—C1—H1B	109.5	N4—C6—H6A	118.6
N1—C1—H1C	109.5	C5—C6—H6A	118.6
H1A—C1—H1C	109.5	N4—C7—C8	121.85 (17)
H1B—C1—H1C	109.5	N4—C7—H7A	119.1
N1—C2—N2	116.88 (15)	C8—C7—H7A	119.1
N1—C2—S1	123.73 (14)	N5—C8—C7	122.59 (18)
N2—C2—S1	119.38 (13)	N5—C8—H8A	118.7
N3—C3—C5	114.34 (14)	C7—C8—H8A	118.7
N3—C3—C4	126.94 (15)	C2—N1—C1	123.93 (16)
C5—C3—C4	118.71 (15)	C2—N1—H1D	118.0
C3—C4—H4A	109.5	C1—N1—H1D	118.0
C3—C4—H4B	109.5	C2—N2—N3	119.13 (14)
H4A—C4—H4B	109.5	C2—N2—H2A	120.4
C3—C4—H4C	109.5	N3—N2—H2A	120.4
H4A—C4—H4C	109.5	C3—N3—N2	119.15 (14)
H4B—C4—H4C	109.5	C6—N4—C7	115.83 (16)
N5—C5—C6	120.41 (15)	C5—N5—C8	116.45 (16)
N5—C5—C3	117.58 (15)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1D···N4$^{\rm i}$	0.86	2.42	3.137 (3)	141
N2—H2A···S1$^{\rm ii}$	0.86	2.77	3.588 (3)	161

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

Fig. 1