organic compounds

# 4-Methyl-1-(4-methylbenzylidene)thio-semicarbazide

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The title compound, $C_{10}H_{13}N_3S$, prepared by the reaction of 4-methylbenzaldehyde and 4-methylthiosemicarbazide, is approximately planar (r.m.s. deviation for the non-H atoms = 0.032 Å). Its conformation is stabilized by an intramolecular N—H···N hydrogen bond, generating an $S(5)$ ring. In the crystal, inversion dimers linked by pairs of N—H···S hydrogen bonds occur. Further weak N—H···S links connect the dimers into (100) sheets.

Related literature

For related structures, see: Li & Jian (2010); Li *et al.* (2009).
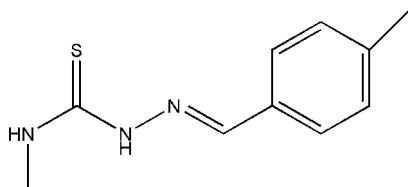
Experimental

Crystal data

$C_{10}H_{13}N_3S$	$V = 1137.1\ (4)$ Å^3
$M_r = 207.29$	$Z = 4$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 9.1139\ (18)$ Å	$\mu = 0.25$ mm^{-1}
$b = 13.689\ (3)$ Å	$T = 293$ K
$c = 9.1195\ (18)$ Å	$0.25 \times 0.23 \times 0.20$ mm
$\beta = 91.92\ (3)°$	

Data collection

Bruker SMART CCD diffractometer	2585 independent reflections
	1739 reflections with $I > 2\sigma(I)$
10620 measured reflections	$R_{int} = 0.030$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.044$	127 parameters
$wR(F^2) = 0.146$	H-atom parameters constrained
$S = 1.14$	$\Delta\rho_{max} = 0.28$ e Å^{-3}
2585 reflections	$\Delta\rho_{min} = -0.19$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···N3	0.86	2.27	2.644 (2)	107
N1—H1A···S1^i	0.86	2.89	3.4869 (16)	128
N2—H2A···S1^{ii}	0.86	2.57	3.4205 (18)	171

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5666).

References

Bruker (1997). *SMART* and *SAINT*. Bruker AXS, Inc., Madison, Wisconsin, USA.
Li, Y.-F. & Jian, F.-F. (2010). *Acta Cryst.* E**66**, o1397.
Li, Y.-F., Liu, H.-X. & Jian, F.-F. (2009). *Acta Cryst.* E**65**, o2959.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

4-Methyl-1-(4-methylbenzylidene)thiosemicarbazide

Yu-Feng Li

S1. Experimental

A mixture of the 4-methylbenzaldehyde (0.10 mol) and 4-methylthiosemicarbazide (0.10 mol) was stirred in refluxing ethanol (10 mL) for 4 h to afford the title compound (0.078 mol, yield 78%). Colourless bars of (I) were obtained by recrystallization from ethanol at room temperature.

S2. Refinement

H atoms were fixed geometrically and allowed to ride on their attached atoms, with C—H distances = 0.93–0.97 Å; N—H = 0.86Å and with $U_{iso}(H) = 1.2U_{eq}(C,N)$ or $1.5U_{eq}(C_{methyl})$.
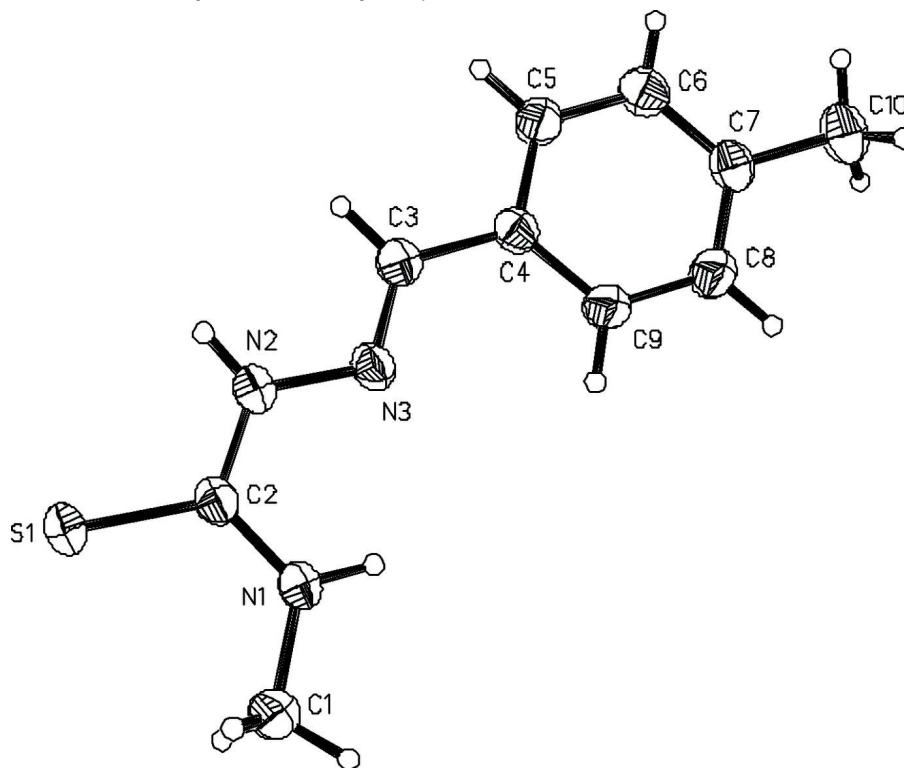
Figure 1

The structure of (I) showing 30% probability displacement ellipsoids.

4-Methyl-1-(4-methylbenzylidene)thiosemicarbazide

Crystal data

$C_{10}H_{13}N_3S$	$F(000) = 440$
$M_r = 207.29$	$D_x = 1.211$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 1840 reflections
$a = 9.1139$ (18) Å	$\theta = 3.3–25.2°$
$b = 13.689$ (3) Å	$\mu = 0.25$ mm^{-1}
$c = 9.1195$ (18) Å	$T = 293$ K
$\beta = 91.92$ (3)°	Bar, colorless
$V = 1137.1$ (4) Å^3	$0.25 \times 0.23 \times 0.20$ mm
$Z = 4$	

Data collection

Bruker SMART CCD diffractometer	1739 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.030$
Graphite monochromator	$\theta_{max} = 27.5°$, $\theta_{min} = 3.5°$
phi and ω scans	$h = -11 \rightarrow 11$
10620 measured reflections	$k = -16 \rightarrow 17$
2585 independent reflections	$l = -11 \rightarrow 11$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.044$	H-atom parameters constrained
$wR(F^2) = 0.146$	$w = 1/[\sigma^2(F_o^2) + (0.0782P)^2 + 0.0376P]$
$S = 1.14$	where $P = (F_o^2 + 2F_c^2)/3$
2585 reflections	$(\Delta/\sigma)_{max} = 0.001$
127 parameters	$\Delta\rho_{max} = 0.28$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.19$ e Å^{-3}
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 $F^2 > \sigma(F^2)$ 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	0.32325 (7)	0.10479 (4)	1.03876 (5)	0.0635 (2)
N3	0.52576 (18)	0.10902 (11)	0.66835 (16)	0.0500 (4)
N2	0.48287 (19)	0.08528 (11)	0.80716 (16)	0.0546 (4)
H2A	0.5276	0.0396	0.8555	0.065*
N1	0.30549 (18)	0.20245 (12)	0.78748 (16)	0.0577 (4)

H1A	0.3375	0.2139	0.7015	0.069*
C2	0.3712 (2)	0.13330 (14)	0.86754 (17)	0.0480 (4)
C4	0.6920 (2)	0.08023 (14)	0.47593 (18)	0.0484 (4)
C7	0.8095 (2)	0.11507 (15)	0.1996 (2)	0.0549 (5)
C3	0.6335 (2)	0.06052 (14)	0.6196 (2)	0.0536 (5)
H3B	0.6757	0.0113	0.6774	0.064*
C9	0.6309 (2)	0.14953 (15)	0.3821 (2)	0.0603 (5)
H9A	0.5496	0.1851	0.4103	0.072*
C5	0.8110 (2)	0.02762 (16)	0.4281 (2)	0.0612 (5)
H5A	0.8527	−0.0204	0.4883	0.073*
C6	0.8688 (2)	0.04527 (18)	0.2926 (2)	0.0656 (6)
H6A	0.9493	0.0093	0.2636	0.079*
C8	0.6893 (3)	0.16642 (16)	0.2469 (2)	0.0640 (6)
H8A	0.6467	0.2137	0.1859	0.077*
C10	0.8716 (3)	0.1343 (2)	0.0510 (2)	0.0766 (7)
H10A	0.9542	0.0921	0.0370	0.115*
H10B	0.9026	0.2012	0.0454	0.115*
H10C	0.7976	0.1217	−0.0240	0.115*
C1	0.1832 (3)	0.2597 (2)	0.8363 (3)	0.0953 (10)
H1B	0.1537	0.3053	0.7611	0.143*
H1C	0.2121	0.2946	0.9239	0.143*
H1D	0.1026	0.2171	0.8564	0.143*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0813 (4)	0.0682 (4)	0.0421 (3)	0.0139 (3)	0.0186 (2)	0.00603 (19)
N3	0.0541 (10)	0.0515 (9)	0.0452 (8)	−0.0020 (7)	0.0137 (7)	0.0010 (6)
N2	0.0613 (11)	0.0587 (9)	0.0444 (8)	0.0068 (8)	0.0135 (7)	0.0048 (6)
N1	0.0608 (11)	0.0644 (10)	0.0491 (8)	0.0117 (8)	0.0193 (7)	0.0127 (7)
C2	0.0525 (11)	0.0490 (10)	0.0430 (9)	−0.0033 (8)	0.0081 (7)	−0.0011 (7)
C4	0.0456 (11)	0.0519 (10)	0.0481 (10)	−0.0013 (8)	0.0075 (7)	−0.0004 (7)
C7	0.0503 (12)	0.0649 (12)	0.0501 (10)	−0.0083 (9)	0.0095 (8)	0.0013 (8)
C3	0.0548 (12)	0.0547 (11)	0.0517 (10)	0.0023 (9)	0.0086 (8)	0.0040 (8)
C9	0.0606 (14)	0.0608 (13)	0.0605 (12)	0.0157 (10)	0.0146 (9)	0.0079 (9)
C5	0.0540 (13)	0.0715 (13)	0.0586 (11)	0.0135 (10)	0.0102 (8)	0.0107 (9)
C6	0.0510 (13)	0.0826 (15)	0.0644 (12)	0.0124 (10)	0.0180 (9)	0.0041 (10)
C8	0.0682 (15)	0.0639 (13)	0.0603 (11)	0.0094 (10)	0.0104 (9)	0.0147 (9)
C10	0.0778 (17)	0.0945 (17)	0.0587 (13)	−0.0095 (14)	0.0226 (11)	0.0069 (11)
C1	0.096 (2)	0.108 (2)	0.0849 (16)	0.0483 (16)	0.0451 (14)	0.0415 (14)

Geometric parameters (Å, º)

S1—C2	1.6813 (17)	C3—H3B	0.9300
N3—C3	1.277 (2)	C9—C8	1.378 (3)
N3—N2	1.376 (2)	C9—H9A	0.9300
N2—C2	1.345 (2)	C5—C6	1.381 (3)
N2—H2A	0.8600	C5—H5A	0.9300

N1—C2	1.326 (2)	C6—H6A	0.9300
N1—C1	1.444 (3)	C8—H8A	0.9300
N1—H1A	0.8600	C10—H10A	0.9600
C4—C9	1.382 (3)	C10—H10B	0.9600
C4—C5	1.385 (3)	C10—H10C	0.9600
C4—C3	1.456 (2)	C1—H1B	0.9600
C7—C6	1.376 (3)	C1—H1C	0.9600
C7—C8	1.383 (3)	C1—H1D	0.9600
C7—C10	1.509 (2)		
C3—N3—N2	116.28 (16)	C6—C5—C4	121.15 (18)
C2—N2—N3	120.22 (16)	C6—C5—H5A	119.4
C2—N2—H2A	119.9	C4—C5—H5A	119.4
N3—N2—H2A	119.9	C7—C6—C5	121.32 (19)
C2—N1—C1	123.79 (16)	C7—C6—H6A	119.3
C2—N1—H1A	118.1	C5—C6—H6A	119.3
C1—N1—H1A	118.1	C9—C8—C7	121.85 (19)
N1—C2—N2	117.22 (15)	C9—C8—H8A	119.1
N1—C2—S1	123.41 (14)	C7—C8—H8A	119.1
N2—C2—S1	119.37 (14)	C7—C10—H10A	109.5
C9—C4—C5	117.71 (17)	C7—C10—H10B	109.5
C9—C4—C3	122.18 (17)	H10A—C10—H10B	109.5
C5—C4—C3	120.09 (17)	C7—C10—H10C	109.5
C6—C7—C8	117.33 (18)	H10A—C10—H10C	109.5
C6—C7—C10	121.6 (2)	H10B—C10—H10C	109.5
C8—C7—C10	121.1 (2)	N1—C1—H1B	109.5
N3—C3—C4	121.71 (18)	N1—C1—H1C	109.5
N3—C3—H3B	119.1	H1B—C1—H1C	109.5
C4—C3—H3B	119.1	N1—C1—H1D	109.5
C8—C9—C4	120.63 (18)	H1B—C1—H1D	109.5
C8—C9—H9A	119.7	H1C—C1—H1D	109.5
C4—C9—H9A	119.7		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···N3	0.86	2.27	2.644 (2)	107
N1—H1*A*···S1[i]	0.86	2.89	3.4869 (16)	128
N2—H2*A*···S1[ii]	0.86	2.57	3.4205 (18)	171

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