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

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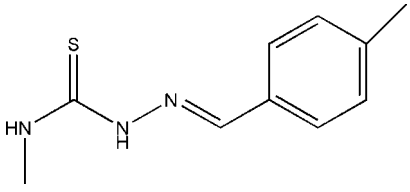
Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.044; $wR$ factor = 0.146; data-to-parameter ratio = 20.4.

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

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

### Data collection

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

### Refinement

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

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—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) $x, -y+\frac{1}{2}, z-\frac{1}{2}$; (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.
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Li, Y.-F., Liu, H.-X. & Jian, F.-F. (2009). *Acta Cryst.* E**65**, o2959.
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# supplementary materials

*Acta Cryst.* (2010). E**66**, o2853 [ **doi:10.1107/S1600536810040663** ]

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

**Y.-F. Li**

## 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.

## 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) = 1.2$U_{eq}$(C,*N*) or 1.5$U_{eq}$($C_{methyl}$).

## Figures

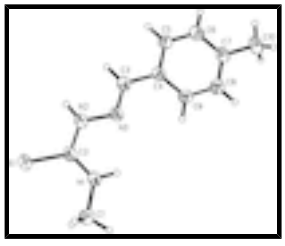

Fig. 1. The structure of (I) showing 30% probability displacement ellipsoids.

## 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, λ = 0.71073 Å |
| Hall symbol: -P 2ybc | Cell parameters from 1840 reflections |
| $a$ = 9.1139 (18) Å | θ = 3.3–25.2° |
| $b$ = 13.689 (3) Å | μ = 0.25 mm$^{-1}$ |
| $c$ = 9.1195 (18) Å | $T$ = 293 K |
| β = 91.92 (3)° | Bar, colorless |
| $V$ = 1137.1 (4) Å$^3$ | 0.25 × 0.23 × 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 | $\theta_{max} = 27.5°$, $\theta_{min} = 3.5°$ |
| 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 $F^2$ | Primary atom site location: structure-invariant direct methods |
| Least-squares matrix: full | Secondary atom site location: difference Fourier map |
| $R[F^2 > 2\sigma(F^2)] = 0.044$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.146$ | H-atom parameters constrained |
| $S = 1.14$ | $w = 1/[\sigma^2(F_o^2) + (0.0782P)^2 + 0.0376P]$ 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$^{-3}$ |

*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 (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| 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, °)*

| | | | |
|---|---|---|---|
| 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, °)*

| *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*+1/2, *z*−1/2; (ii) −*x*+1, −*y*, −*z*+2.

**Fig. 1**