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Key indicators

Single-crystal X-ray study
 $T = 298\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.005\text{ \AA}$
 R factor = 0.064
 wR factor = 0.155
Data-to-parameter ratio = 15.9For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

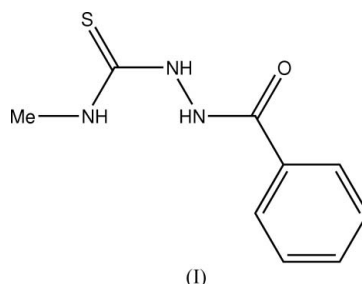
1-Benzoyl-4-methylthiosemicarbazide

In the title compound, $\text{C}_9\text{H}_{11}\text{N}_3\text{OS}$, the OCN group makes dihedral angles of $30.48(16)$ and $74.41(14)^\circ$, respectively, with the phenyl and methylthiourea groups. The crystal structure is stabilized by weak intermolecular $\text{N}-\text{H}\cdots\text{S}$, $\text{N}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds, to form a two-dimensional network.

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Comment

The continuing efforts to synthesize new thiosemicarbazide derivatives are driven by their ability to form complexes with metal ions and also their biological activities. As an example, 4-(2-methylprop-2-enyl)-1-[3-(trifluoromethyl)phenyl]thiosemicarbazide has been found to exhibit anti-implantation activity (Nagarajan *et al.*, 1984).



The title molecule, (I), adopts a *cis-trans* configuration with respect to the position of the methyl and benzoylamine

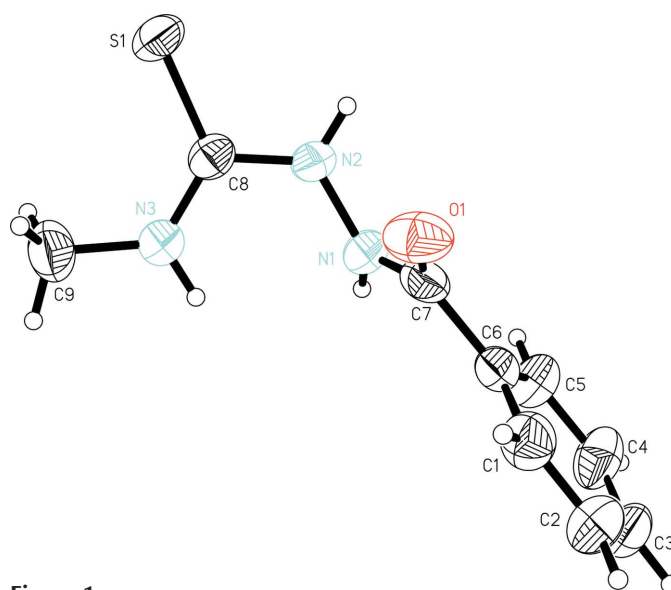


Figure 1
Molecular structure of (I), with 50% probability displacement ellipsoids.

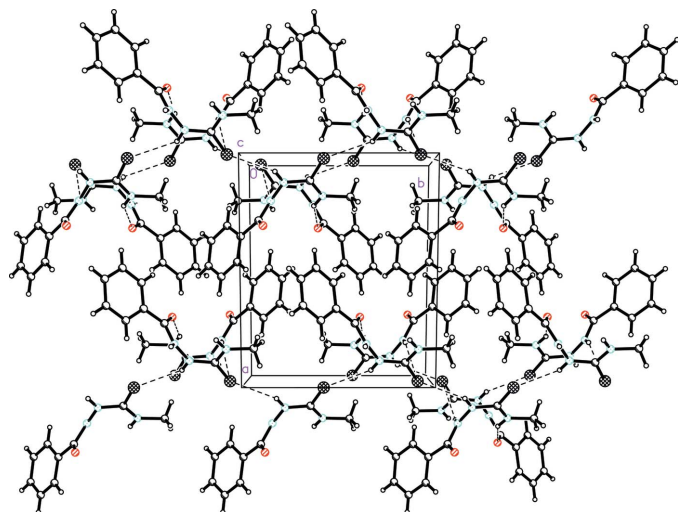


Figure 2

Packing diagram of (I), viewed down the *c* axis. The dashed lines denote the N—H···O and N—H···S hydrogen bonds.

groups, respectively, relative to the S atom across the N3—C8 and N2—C8 bonds (Fig. 1), as observed in the related compounds 1-methyl-4-salicyloylthiosemicarbazide (Gors *et al.*, 1979) and 4-phenyl-1-(propan-2-ylidene)thiosemicarbazide (Jian *et al.*, 2005). The bond lengths and angles in (I) are in normal ranges (Allen *et al.*, 1987) and comparable to those in the above-cited compounds.

The methylthiourea (S1/N2/N3/C8/C9), phenyl (C1—C6) and O1/C7/N1 fragments are each planar. The maximum deviation is 0.011 (3) Å for atom C1 in the phenyl group. The O1/C7/N1 fragment makes dihedral angles of 30.48 (16) and 74.41 (14)° with the phenyl and methylthiourea fragments, respectively. The phenyl and methylthiourea fragments are inclined to each other by 75.12 (14)°. In the crystal structure, the molecules are linked by weak intermolecular N—H···S, N—H···O and C—H···O hydrogen bonds (Table 2) to form a two-dimensional network (Fig. 2).

Experimental

A solution of 4-methylthiosemicarbazide (1.05 g, 0.01 mol) in acetone (50 ml) was added dropwise into an acetone solution (50 ml) containing an equimolar amount of benzoylchloride and ammonium thiocyanate in a two-necked round-bottomed flask. The mixture was refluxed for about 2 h. The light-yellow solution was filtered off and colourless crystals were obtained after five days of evaporation (yield 85%, m.p. 185.3–188.8 K).

Crystal data

C₉H₁₁N₃OS
M_r = 209.27
 Monoclinic, *P*2₁/*c*
a = 12.110 (14) Å
b = 9.958 (11) Å
c = 8.758 (10) Å
 β = 102.69 (2)°
V = 1030 (2) Å³
Z = 4

D_x = 1.349 Mg m⁻³
 Mo *K*α radiation
 Cell parameters from 2263 reflections
 θ = 1.7–26.0°
 μ = 0.28 mm⁻¹
T = 298 (2) K
 Block, colourless
 0.49 × 0.20 × 0.19 mm

Data collection

Bruker SMART CCD area-detector diffractometer
 ω scans
 Absorption correction: multi-scan (SADABS; Bruker, 2000)
 T_{\min} = 0.873, T_{\max} = 0.947
 5555 measured reflections

2014 independent reflections
 1807 reflections with $I > 2\sigma(I)$
 R_{int} = 0.050
 θ_{max} = 26.0°
 h = -13 → 14
 k = -12 → 11
 l = -10 → 10

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)]$ = 0.064
 $wR(F^2)$ = 0.155
 S = 1.23
 2014 reflections
 127 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0526P)^2 + 0.5802P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.29 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.22 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (Å, °).

S1—C8	1.694 (3)	N2—C8	1.347 (4)
O1—C7	1.220 (4)	N3—C8	1.320 (4)
N1—C7	1.361 (4)	N3—C9	1.448 (4)
N1—N2	1.387 (3)		
C7—N1—N2	117.8 (3)	N3—C8—N2	117.9 (2)
C8—N2—N1	122.7 (2)	N3—C8—S1	125.4 (2)
C8—N3—C9	124.7 (3)	N2—C8—S1	116.7 (2)

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
N1—H1A···S1 ⁱ	0.86	2.66	3.334 (5)	136
N2—H2A···S1 ⁱⁱ	0.86	2.66	3.248 (4)	127
N3—H3A···O1 ⁱ	0.86	2.22	2.936 (5)	140
C3—H3B···O1 ⁱⁱⁱ	0.93	2.60	3.321 (6)	135

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

All H atoms were placed in idealized positions and allowed to ride on their parent C and N atoms with distances constrained to 0.93 (aromatic C—H), 0.96 (methyl C—H) or 0.86 Å (N—H). $U_{\text{iso}}(\text{H})$ values were set at $1.2U_{\text{eq}}(\text{carrier atom})$ for aromatic CH and NH groups, and $1.5U_{\text{eq}}(\text{carrier atom})$ for the CH₃ group.

Data collection: SMART (Bruker, 2000); cell refinement: SAINT (Bruker, 2000); 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, 2003).

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