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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ (C–C) = 0.003 Å
R factor = 0.043
wR factor = 0.123
Data-to-parameter ratio = 16.2For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

Acetone 4-phenylthiosemicarbazone

In the title compound, C₁₀H₁₃N₃S, the isopropylideneamine and phenyl groups are *trans* and *cis*, respectively, with respect to the thione group across their C–N bonds. The molecule is stabilized by intra- and intermolecular hydrogen bonds to form dimers parallel to (010).

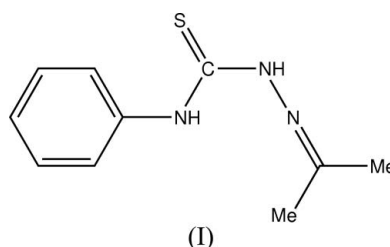
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Comment

In the acetone-*N*(4)-phenylthiosemicarbazone–SnMe₂Cl complex, C₁₂H₁₆ClN₃SSn, the ligand is chelated to the Sn atom in a bidentate manner *via* the thiono S and azomethine N atoms (de Sousa *et al.*, 2004). The hydrazinic N atom is *cis* to the thiono S atom across the C–N bond. However, in the free ligand, (I), the azomethine atom N3 is *trans* to the thiono atom S1 (Fig. 1). Similar configurations have been observed in many thiosemicarbazones, such as acetone-4-methylthiocarbazone (Parsons *et al.*, 2000). Thus, a change of configuration may have taken place during the complexation that led to the bidentate chelation.



The bond lengths and angles in (I) are in normal ranges (Allen *et al.*, 1987), although the C7–N1 and C7–N2 bonds show slight variations compared with the values observed in acetone-4-methylthiosemicarbazone [1.325 (3) and 1.361 (2) Å, respectively; Parsons *et al.*, 2000]. In contrast, the

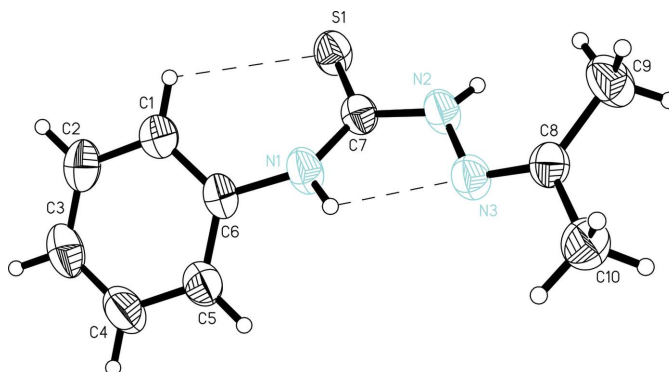
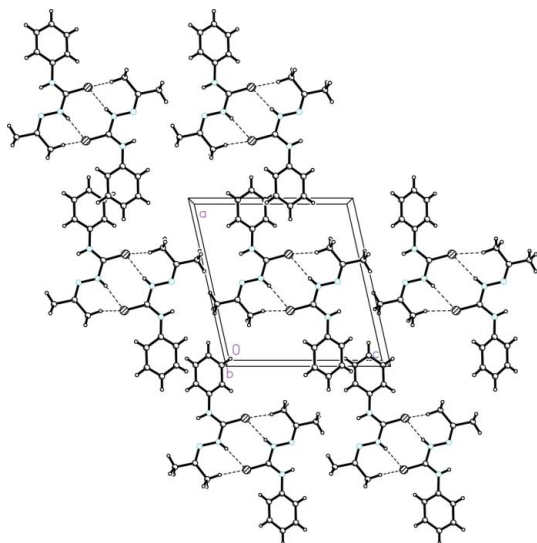


Figure 1

The molecular structure of (I), with 50% probability displacement ellipsoids. Dashed lines indicate the intramolecular hydrogen bonds.

**Figure 2**

A packing diagram for (I). Dashed lines denote C–H...S hydrogen bonds.

presence of a phenyl group attached to atom N1 causes the molecule to lose its overall planarity. The N1/S1/N2/C7, N3/C8/C9/C10 and phenyl (C1–C6) fragments are all planar. The maximum deviation is 0.014 (2) Å for atom N3 in the N3/C8/C9/C10 fragment. The central N1/S1/N2/C7 fragment makes dihedral angles of 30.61 (11) and 22.50 (10)° with the C1–C6 and N3/C8/C9/C10 fragments, respectively. The phenyl ring is inclined to the N3/C8/C9/C10 fragment at an angle of 43.96 (9)°.

There are two intramolecular hydrogen bonds in (I), namely N1–H1B...N3 and C1–H1A...S1, resulting in the formation of one pseudo-six-membered ring, C1–C6–N1–C7–S1...H1A, and one five-membered ring, N2–C7–N1–H1B...N3.

In the crystal structure, the molecules are linked to form dimers *via* N2–H2B...S1ⁱ and C9–H9A...S1ⁱ intermolecular hydrogen bonds (see Table 2 for symmetry code), parallel to the *ac* face (Fig. 2).

Experimental

4-Phenylthiosemicarbazide (2 mmol) was added to a refluxing mixture containing excess acetone (10 ml) in methanol (50 ml). The mixture was further heated and stirred for about 1 h, and then left at room temperature in the open air to yield colourless crystals of (I) suitable for X-ray diffraction (yield 70%, m.p. 397–399 K).

Crystal data

C₁₀H₁₃N₃S
M_r = 207.29
 Monoclinic, *P*2₁/*c*
a = 12.225 (3) Å
b = 7.618 (2) Å
c = 11.639 (3) Å
 β = 102.660 (4)°
V = 1057.6 (5) Å³
Z = 4

D_x = 1.302 Mg m⁻³
 Mo *K*α radiation
 Cell parameters from 3919 reflections
 θ = 1.7–26.5°
 μ = 0.27 mm⁻¹
T = 298 (2) K
 Block, colourless
 0.43 × 0.36 × 0.31 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
 ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
*T*_{min} = 0.893, *T*_{max} = 0.921
 5827 measured reflections

2187 independent reflections
 2014 reflections with *I* > 2σ(*I*)
*R*_{int} = 0.013
 θ _{max} = 26.5°
h = –9 → 15
k = –9 → 7
l = –13 → 14

Refinement

Refinement on *F*²
R[*F*² > 2σ(*F*²)] = 0.043
wR(*F*²) = 0.123
S = 1.06
 2187 reflections
 135 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0727P)^2 + 0.3195P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.29 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.39 \text{ e } \text{Å}^{-3}$

Table 1

Selected geometric parameters (Å, °).

S1–C7	1.6825 (17)	N2–C7	1.348 (2)
N1–C7	1.343 (2)	N2–N3	1.392 (2)
N1–C6	1.425 (2)	N3–C8	1.276 (2)
C7–N1–C6	127.96 (14)	N1–C7–S1	125.76 (12)
C7–N2–N3	118.33 (14)	N2–C7–S1	119.50 (12)
C8–N3–N2	117.73 (14)		

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–H1B...N3	0.86 (2)	2.13 (2)	2.581 (2)	112
C1–H1A...S1	0.93	2.84	3.248 (2)	108
N2–H2B...S1 ⁱ	0.83 (2)	2.68 (2)	3.4969 (18)	168
C9–H9A...S1 ⁱ	0.96	2.87	3.546 (2)	128

Symmetry codes: (ii) $-x + 1, -y, -z + 1$.

H atoms attached to N atoms were located in a difference map and refined isotropically. All other H atoms were located in a difference map and repositioned geometrically, with C–H = 0.93–0.96 Å, and allowed to ride on their parent C atoms, with *U*_{iso}(H) = 1.2*U*_{eq}(C), or 1.5*U*_{eq}(C) for CH₃.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINTE* (Bruker, 2000); data reduction: *SAINTE*; 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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