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N-Benzoylthiosemicarbazide

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.006 Å
R factor = 0.042
wR factor = 0.124
Data-to-parameter ratio = 12.8

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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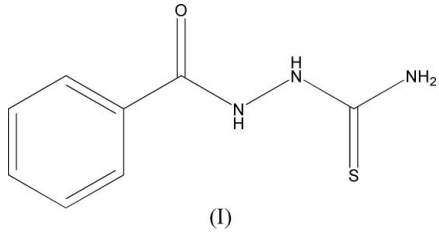
The title compound, $C_8H_9N_3OS$, crystallizes in a triclinic unit cell, with two crystallographically independent molecules in the asymmetric unit. It has a two-dimensional layer structure with intermolecular hydrogen bonding.

Comment

N-Substituted thiosemicarbazides have been widely investigated because of the large number of potentially useful biological properties, particularly antitumour activity (Castiñeiras *et al.*, 2000). Therefore, we have synthesized the novel analogous compound *N*-benzoylthiosemicarbazide, (I), and determined its crystal structure.



(I)

The crystal structure of (I) is shown in Fig. 1. The hydrogen-bonding data for (I) are listed in Table 1. The title compound crystallizes in the triclinic space group $P\overline{1}$, with two crystallographically independent molecules in the asymmetric unit. There are two planes (plane 1: thiourea group; plane 2: benzoyl group) in each molecule. The angles between the two planes in the two molecules are 56.9 (3) and 79.5 (3)°. In the two molecules, the angles at C7, C8, C15 and C16 are near 120°, indicating a trigonal planar arrangement around these C atoms. The angle between the two benzene rings is 28.2 (2)°. The C8—S1 [1.721 (2) Å] and C16—S2 [1.682 (3) Å] bond lengths indicate character intermediate between single and double bonds, being shorter than a pure single bond (1.84 Å; Liu *et al.*, 1999).

In addition, neighbouring molecules are connected by hydrogen bonds, forming an infinite two-dimensional system (Fig. 2). Both molecules form intramolecular hydrogen bonds, *viz*. N3—H3*A*···N1 and N6—H6*A*···N4 in the two molecules. The S atoms interact with a total of five H atoms from five different molecules. Atom S1 interacts with the N1—H1, N5—H5 and N6—H6*B* groups, and S2 interacts with the N2—H2 and N3—H3*B* groups.

Experimental

Benzoyl chloride (39.5 mmol) was added to a solution of sodium benzoate (39.5 mmmol) in chloroform (60 ml) at 278 K. The reaction mixture was then warmed slowly to 295 K and stirred for 2 h. Thio-

semicarbazide (32.9 mmol) was added to the mixture to give a white suspension, which was collected, rinsed with chloroform and diethyl ether, and then dried to give *N*-benzoylthiosemicarbazide. Colourless single crystals were obtained from absolute methanol.

Crystal data

$C_8H_9N_3OS$	$Z = 4$
$M_r = 195.24$	$D_x = 1.331$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 8.237$ (3) Å	Cell parameters from 905 reflections
$b = 8.438$ (3) Å	$\theta = 2.6$–$26.1°$
$c = 14.590$ (6) Å	$\mu = 0.30$ mm^{-1}
$\alpha = 83.554$ (7)°	$T = 293$ (2) K
$\beta = 75.631$ (7)°	Block, colourless
$\gamma = 84.998$ (7)°	0.20 × 0.16 × 0.14 mm
$V = 974.3$ (7) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	3408 independent reflections
φ and ω scans	2413 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)	$R_{int} = 0.020$
$T_{min} = 0.927$, $T_{max} = 0.959$	$\theta_{max} = 25.0°$
5064 measured reflections	$h = -9 \rightarrow 9$
	$k = -10 \rightarrow 8$
	$l = -17 \rightarrow 11$

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.042$	$w = 1/[\sigma^2(F_o^2) + (0.066P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.124$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.09$	$\Delta\rho_{max} = 0.33$ e Å^{-3}
3408 reflections	$\Delta\rho_{min} = -0.29$ e Å^{-3}
267 parameters	

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

D—H···A	D—H	H···A	D···A	D—H···A
N6—H6B···S1^{i}	0.87 (1)	2.584 (12)	3.439 (3)	169 (3)
N5—H5···S1^{ii}	0.85 (1)	2.650 (11)	3.495 (3)	172 (2)
N4—H4···S1^{iii}	0.85 (1)	2.99 (3)	3.503 (3)	120 (3)
N4—H4···S2^{iv}	0.85 (1)	2.90 (2)	3.584 (3)	138 (3)
N3—H3B···S2^{v}	0.85 (1)	2.635 (12)	3.475 (3)	167 (3)
N3—H3A···O2^{vi}	0.86 (1)	2.069 (13)	2.907 (3)	164 (3)
N2—H2···S2^{vii}	0.86 (1)	2.431 (13)	3.268 (3)	165 (3)
N1—H1···S1^{viii}	0.86 (1)	2.530 (13)	3.358 (3)	162 (2)

Symmetry codes: (i) $1+x, 1+y, z-1$; (ii) $1+x, y, z-1$; (iii) $1-x, 1-y, 1-z$; (iv) $2-x, 2-y, -z$; (v) $x-1, y-1, 1+z$; (vi) $2-x, 1-y, 1-z$; (vii) $x-1, y, 1+z$; (viii) $1-x, 1-y, 2-z$.

The H atoms attached to N were located in a difference Fourier map and their coordinates and isotropic displacement parameters were refined [N—H = 0.85 (1)–0.87 (1) Å]. All other H atoms were placed in idealized positions and were refined using a riding model [C—H = 0.93 Å; U_{iso}(H) = 1.2U_{eq}(C)].

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

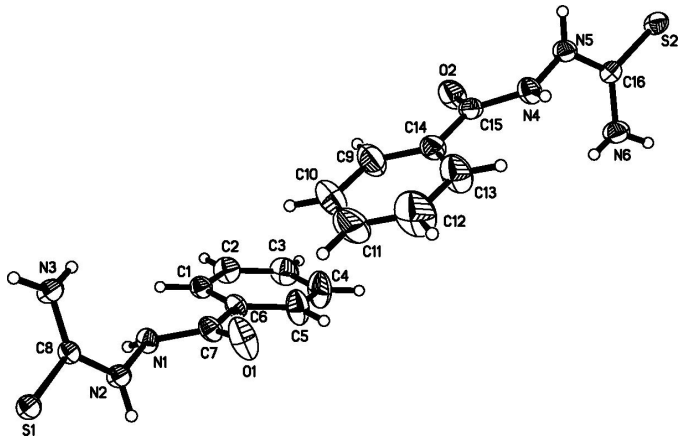


Figure 1
A view of the asymmetric unit of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% level.

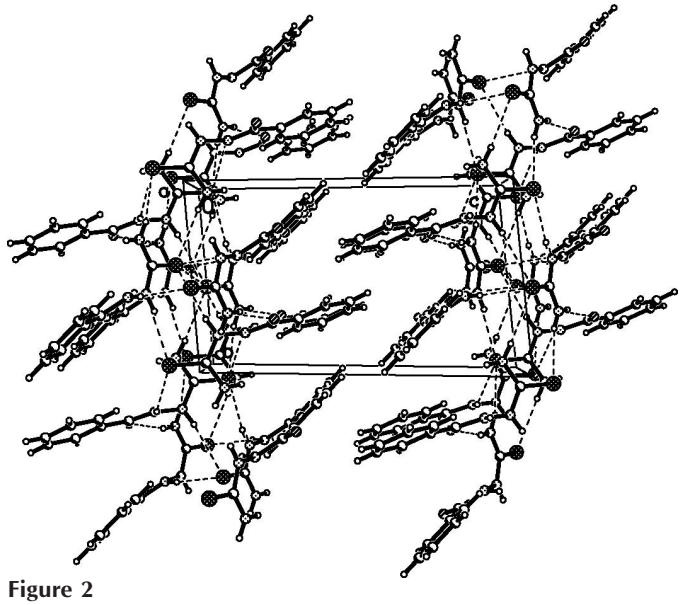


Figure 2
The two-dimensional network of hydrogen bonds (shown as dashed lines) in (I).

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References

Bruker (1997). *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (1998). *SMART* (Version 5.0) and *SAINT* (Version 4.0). Bruker AXS Inc., Madison, Wisconsin, USA.
Castiñeiras, A., Bermejo, E., Valdes-Martínez J., Espinosa-Pérez, G. & West, D. X. (2000). *J. Mol. Struct.* **522**, 271–278.
Liu, B., Hu, R. X. & Liang, H. (1999). *Chin. J. Struct. Chem.* **18**, 414–417.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.