

***N*-(Benzylaminothiocarbonyl)benzamide**Jose R. Sabino,^{a*} Silvio Cunha,^b
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Key indicatorsSingle-crystal X-ray study
T = 297 K
Mean σ (C–C) = 0.005 Å
R factor = 0.052
wR factor = 0.199
Data-to-parameter ratio = 14.1For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

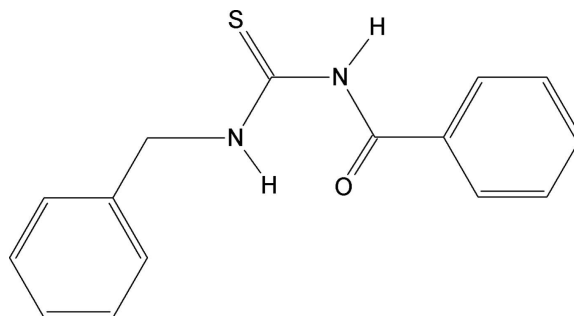
In the title compound $C_{15}H_{14}N_2OS$, centrosymmetric dimers are formed *via* intermolecular N–H···S hydrogen bonds. The dimeric units are linked by a weak intermolecular C–H···S hydrogen bond, forming infinite chains along [010]. Additionally, unprecedented C–H··· π interactions, involving the benzoyl group of the thiourea group as donor–acceptor, connect parallel chains.

Received 11 August 2006

Accepted 12 August 2006

Comment

In recent years, thiourea derivatives have attracted the attention of several research groups owing to their potential in medicinal chemistry (Sun *et al.*, 2006; Coppola *et al.*, 2005) and their use as organocatalysts in diverse organic transformations (Takemoto, 2005). A broad spectrum of thiourea derivatives and their metal complexes have been investigated for their bioactivities such as antifungal (Phuong *et al.*, 2004) and antimalarial (Egan *et al.*, 2004). Thiourea-based non-nucleoside inhibitors of HIV reverse transcriptase have also been described (Venkatachalam *et al.*, 2004). As part of our ongoing synthesis and structural study of potential bioactive compounds (Cunha *et al.*, 2005, 2006; Vencato *et al.*, 2006; Vencato, Cunha, Ferrari *et al.*, 2004; Vencato, Cunha, Rocha *et al.*, 2004), we describe here the X-ray crystal structure of the title disubstituted thiourea derivative, (I).



Despite the fact that four planar conformations are possible for N^1,N^2 -disubstituted thioureas (Sudha & Sathyanarayana, 1984), only the *cis-trans* conformation was observed for (I) (Fig. 1). The C2=N1 and C4=O6 bond lengths indicate that these are double bonds (Table 1). The lengthening of the C7–C4 bond relative to the formal single bond by 0.038 Å is due to the lack of π -orbital overlap between these atoms, as indicated by the torsion angle N3–C4–C7–C8 of -31.0 (4)°. The conformation is stabilized by an intramolecular hydrogen bond (N1–H1···O6; Table 2), responsible for the torsion angle O6–C4–N3–C2 of -7.4 (4)°, and by a short contact

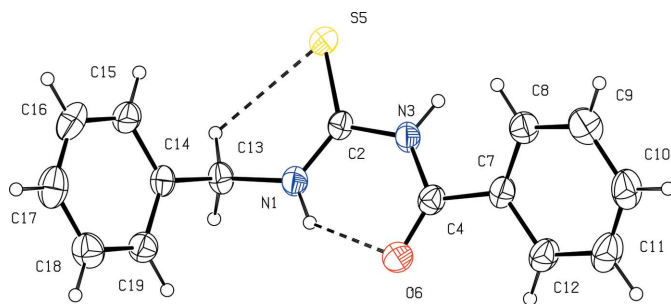


Figure 1
View of (I) with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii. Hydrogen bonds are shown as dashed lines.

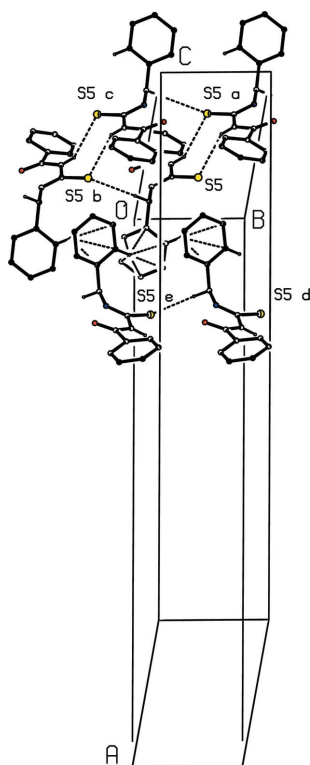


Figure 2
Packing diagram showing the infinite chain along the *b* axis. Intermolecular hydrogen interactions are shown as dashed lines. Only the H atoms involved in hydrogen bonds are shown. [Symmetry codes: (a) $-x, 1 - y, 1 - z$; (b) $x, y - 1, z + 1$; (c) $-x, -y, 1 - z$; (d) $\frac{1}{2} - x, \frac{1}{2} + y, \frac{3}{2} - z$; (e) $\frac{1}{2} - x, y - \frac{1}{2}, \frac{3}{2} - z$.]

C13—H13A \cdots S5 that favors the torsion angle C2—N1—C13—C14 of 107.0 (3)°.

The crystal packing of (I) does not show any strong hydrogen bonds, in contrast to those of other benzoylthioureas studied by us (Cunha *et al.*, 2006). The packing (Fig. 2) is mediated by intermolecular N3—H3 \cdots S5ⁱ hydrogen bonds [symmetry code: (i) $-x, 1 - y, -z$], forming dimers, and a short contact C13—H13B \cdots S5ⁱⁱ [symmetry code: (ii) $x, 1 + y, z$], linking neighboring dimers to form infinite chains along [010]. There is an additional C—H \cdots π interaction between C15—H15 and the C15ⁱⁱⁱ ring, connecting parallel chains

[C15—H15 \cdots Cg = 130°, H15 \cdots Cg = 2.96 Å and C15 \cdots Cg = 3.627 (4) Å [Cg is the centroid of the C15ⁱⁱⁱ ring; symmetry code: (iii) $\frac{1}{2} - x, \frac{1}{2} + y, \frac{3}{2} - z$]. This is the first description of a C—H \cdots π intermolecular interaction in structural studies of benzoylthioureas, as indicated by a search in the Cambridge Structural Database (May 2006 update; Allen, 2002). This interaction should be important in the design of benzoylthioureas for organocatalysis.

Experimental

Benzoyl isothiocyanate (5 mmol) was added dropwise to a solution of benzylamine (5 mmol) in benzene (5 ml) with stirring and ice-bath cooling. The reaction mixture was left for 3 h at room temperature, after which time the solvent was evaporated and the crude solid was triturated with petroleum ether. Recrystallization with CH₂Cl₂/petroleum ether (4:1) afforded the thiourea (I).

Crystal data

C ₁₅ H ₁₄ N ₂ OS	<i>Z</i> = 8
<i>M_r</i> = 270.35	<i>D_x</i> = 1.342 Mg m ⁻³
Monoclinic, <i>C</i> 2/ <i>c</i>	Cu <i>K</i> α radiation
<i>a</i> = 30.560 (7) Å	<i>μ</i> = 2.09 mm ⁻¹
<i>b</i> = 6.112 (2) Å	<i>T</i> = 297 (2) K
<i>c</i> = 17.364 (4) Å	Prism, colorless
<i>β</i> = 124.40 (2)°	0.35 × 0.25 × 0.15 mm
<i>V</i> = 2676.1 (14) Å ³	

Data collection

Enraf–Nonius CAD-4 diffractometer	2439 independent reflections
<i>ω</i> / <i>2θ</i> scans	2125 reflections with <i>I</i> > 2σ(<i>I</i>)
Absorption correction: <i>ψ</i> scan (North <i>et al.</i> , 1968)	<i>R</i> _{int} = 0.022
<i>T</i> _{min} = 0.518, <i>T</i> _{max} = 0.730	<i>θ</i> _{max} = 67.9°
2569 measured reflections	2 standard reflections
	frequency: 120 min
	intensity decay: 1%

Refinement

Refinement on <i>F</i> ²	$w = 1/[\sigma^2(F_o^2) + (0.1204P)^2 + 2.5013P]$
$R[F^2 > 2\sigma(F^2)] = 0.052$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.199$	(Δ/σ) _{max} < 0.001
<i>S</i> = 1.15	$\Delta\rho_{\text{max}} = 0.59 \text{ e } \text{Å}^{-3}$
2439 reflections	$\Delta\rho_{\text{min}} = -0.38 \text{ e } \text{Å}^{-3}$
173 parameters	Extinction correction: <i>SHELXL97</i>
H-atom parameters constrained	Extinction coefficient: 0.0045 (5)

Table 1

Selected geometric parameters (Å, °).

N1—C2	1.316 (4)	N3—C4	1.375 (4)
N1—C13	1.453 (3)	C4—O6	1.215 (4)
C2—N3	1.393 (3)	C4—C7	1.498 (4)
C2—S5	1.677 (3)		
C2—N1—C13	124.9 (2)	N3—C2—S5	117.5 (2)
N1—C2—N3	117.2 (2)	C4—N3—C2	127.6 (2)
N1—C2—S5	125.3 (2)	O6—C4—N3	123.3 (3)
C13—N1—C2—S5	2.9 (4)	N3—C4—C7—C8	−31.0 (4)
N1—C2—N3—C4	3.8 (4)	O6—C4—C7—C12	−28.4 (4)
S5—C2—N3—C4	−176.0 (2)	C2—N1—C13—C14	107.0 (3)

Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1 \cdots O6	0.86	1.98	2.663 (3)	136
N3—H3 \cdots S5 ⁱ	0.86	2.73	3.479 (2)	146
C13—H13A \cdots S5	0.97	2.64	3.121 (3)	111
C13—H13B \cdots S5 ⁱⁱ	0.97	2.86	3.610 (3)	135

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

All H atoms were placed in calculated positions and allowed to ride on their parent atoms, with C—H distances in the range 0.93–0.97 Å and N—H distances of 0.86 Å, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N})$. The aromatic ring atoms C10, C11, C16 and C17 have very anisotropic displacement ellipsoids with the longer axis along the bonds C10—C11 and C16—C17, for which the highest s.u. values are observed.

Data collection: *CAD-4-PC* (Enraf–Nonius, 1993); cell refinement: *CAD-4-PC*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

The authors gratefully acknowledge the financial support of Conselho Nacional de Desenvolvimento Científico e Tecnológico – CNPq, Fundação de Amparo à Pesquisa do Estado da Bahia – FAPESB and Fundação de Apoio à Pesquisa – FUNAPE/UFG. We also thank CNPq for a scholarship to RMB and for research fellowships to IV and SC.

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