Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

N-[*N*-(Furan-2-carbonyl)hydrazinothiocarbonyl]benzamide

Bohari M. Yamin* and M. Sukeri M. Yusof

School of Chemical Sciences and Food Technology, Universiti Kebangsaan Malaysia, 43600 Bangi, Selangor, Malaysia

Correspondence e-mail:
bohari@pkrisc.cc.ukm.my

Received 18 November 2002
Accepted 23 December 2002
Online 10 January 2003

Key indicators

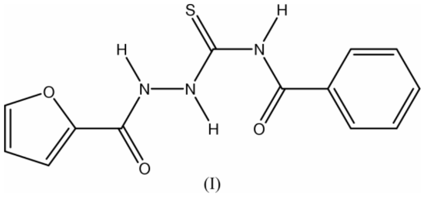
Single-crystal X-ray study
T = 299 K
Mean σ(C–C) = 0.003 Å
R factor = 0.047
wR factor = 0.141
Data-to-parameter ratio = 16.9

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

The title compound, $C_{13}H_{11}N_3O_3S$, adopts a *cis–trans* conformation, where the furoyl moiety and the benzoyl group lie respectively *cis* and *trans* relative to the S atom across the thiourea C—N bonds. Both H atoms belonging to the hydrazine moiety participate in intramolecular hydrogen bonds; one of them forms an N—H···O bond and closes the six-membered pseudo-ring, whereas the other one participates in N—H···O and N—H···S bonds, forming two five-membered pseudo-rings. The molecules in the crystal are linked by N—H···O and C—H···O intermolecular hydrogen bonds involving the amide and phenyl H atoms and the O atoms of the furoyl and benzoyl groups. The intermolecular hydrogen bonds give rise to infinite chains running along the *c* axis of the crystal.

Comment

Thiourea and dithio compounds, such as dithiocarbazates and dithiocarbamates, are potential biologically active compounds. Some thiourea derivatives, such as *N*-[2-(2-chlorophenethyl)]-*N*′-[2-(5-bromopyridyl)]thiourea, have been reported as potent non-nucleoside inhibitors of HIV reverse transcriptase at nanomolar concentrations (Vig *et al.*, 1998; Vankatachalam *et al.*, 2001). Therefore, new thiourea derivatives and their structures are presently the focus of the studies of several research groups. Within the framework of such studies carried out in our group, suitable crystals of the title compound, (I), were obtained and its crystal structure was determined.



(I)

As in the majority of benzoylthiourea derivatives, the title compound, (I) (Fig. 1), adopts a *cis–trans* conformation where the N3/C9/O2/C10/C11/C12/C13/O3 furoyl moiety and the benzoyl group lie respectively *cis* and *trans* relative to the S atom across the thiourea C—N bonds (Usman *et al.*, 2002; Shanmuga Sundara Raj *et al.*, 1999). The bond lengths and angles in (I) (Table 1) are within normal ranges (Allen *et al.*, 1987). However, the N1—C8 and N2—C8 bond lengths of 1.387 (2) and 1.318 (2) Å, respectively, are slightly shorter than the analogous bonds in *N*-benzoyl-*N*′-(2,6-dimethylphenyl)thiourea with a bulky dimethylphenyl substituent [1.395 (2) and 1.331 (3) Å, respectively (Usman *et al.*, 2002)].

The S1/C8/N1/N2/C7/O1 and the O2/O3/C9/C10/C11/C12/C13 furoyl fragments are almost planar [maximum deviations

DOI: 10.1107/S1600536802023620

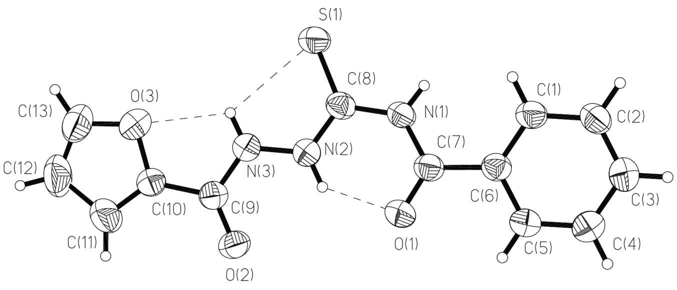


Figure 1
The molecular structure of (I), with ellipsoids at the 50% probability level. The dashed lines denote the intramolecular N—H···O and N—H···S hydrogen bonds.

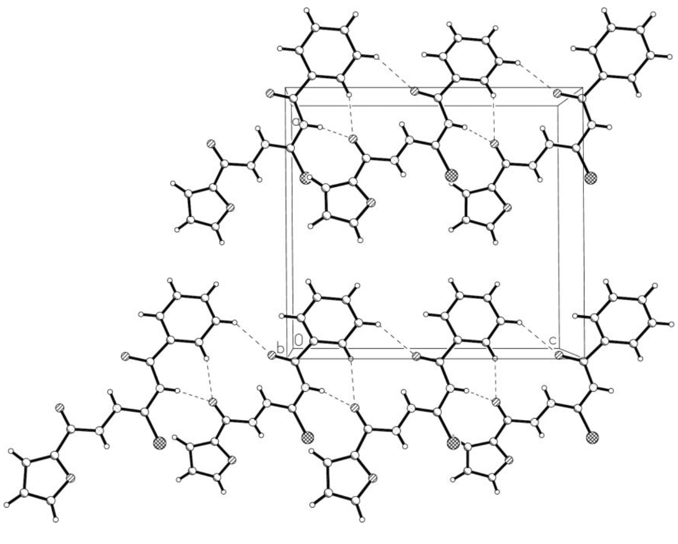


Figure 2
Packing diagram of the title complex, viewed down the *b* axis. The dashed lines denote the intermolecular N—H···O and C—H···O hydrogen bonds.

at N1 and C10 from their respective mean planes are 0.039 (1) and 0.023 (2) Å]. Unlike the bulky *N*-benzoyl-*N'*-(2,6-dimethylphenyl)thiourea, where the aromatic rings are almost perpendicular to each other, the present compound shows the dihedral angle between the phenyl and the furoyl O2/O3/C9/C10/C11/C12/C13 planes of only 1.72 (8)°. The dihedral angles between the carbonylthiourea moiety S1/C8/N1/N2/C7/O1 and the phenyl C1/C2/C3/C4/C5/C6/C7 and furoyl O2/O3/C9/C10/C11/C12/C13 fragments are 18.74 (7) and 17.60 (6)°, respectively.

There are three intramolecular hydrogen bonds in the molecule of (I) (N2—H2*A*···O1, N3—H3*A*···S1 and N3—H3A···O3; Table 2). As a result, in addition to the six-membered pseudo-ring O1—C7—N1—C8—N2—H2*A*, typical of benzoylthiourea systems, two five-membered rings (O3—C10—C9—N3—H3*A* and S1—C8—N2—N3—H3*A*) are formed. In the crystal structure, three intermolecular hydrogen bonds [N1—H1*A*···O2^{i}, C1—H1*B*···O2^{i} and C2—H2*B*···O1^{i}; Table 2] link the molecules into infinite chains parallel to the *c* axis (Fig. 2).

Experimental

A solution of furan-2-carboxylic acid hydrazide (0.16 g, 1.40 mmol) in acetone (50 ml) was added dropwise to 50 ml of an acetone solution containing an equimolar amount of benzoyl thiocyanate in a two-neck round-bottomed flask. The solution was refluxed for about one hour and then cooled on ice. The brown precipitate was filtered off and washed with ethanol-distilled water, then dried in a vacuum (yield 76%). Recrystallization from DMF yielded single crystals suitable for X-ray analysis.

Crystal data

$C_{13}H_{11}N_3O_3S$
$M_r = 289.31$
Monoclinic, $P2_1/c$
$a = 12.7902$ (14) Å
$b = 7.5505$ (8) Å
$c = 13.9952$ (15) Å
$\beta = 90.375$ (2)°
$V = 1351.5$ (3) Å^3
$Z = 4$
$D_x = 1.422$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3018 reflections
$\theta = 1.6$–27.5°
$\mu = 0.25$ mm^{-1}
$T = 299$ (2) K
Block, brown
0.61 × 0.39 × 0.30 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.862$, $T_{max} = 0.929$
7854 measured reflections
3065 independent reflections
2548 reflections with $I > 2\sigma(I)$
$R_{int} = 0.018$
$\theta_{max} = 27.5°$
$h = -16 \rightarrow 16$
$k = -9 \rightarrow 7$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.141$
$S = 1.04$
3065 reflections
181 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0818P)^2 + 0.2233P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.26$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—C8	1.6631 (18)	N1—C7	1.374 (2)
O1—C7	1.219 (2)	N1—C8	1.387 (2)
O2—C9	1.220 (2)	N2—N3	1.371 (2)
O3—C10	1.368 (2)	N2—C8	1.318 (2)
O3—C13	1.351 (3)	N3—C9	1.342 (2)
C13—O3—C10	105.60 (18)	O1—C7—N1	121.75 (16)
C7—N1—C8	126.13 (14)	N1—C7—C6	117.05 (14)
C8—N2—N3	119.77 (14)	N2—C8—N1	115.66 (14)
C9—N3—N2	120.29 (14)	O2—C9—N3	122.36 (15)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O2^{i}	0.86	2.17	2.988 (2)	158
N2—H2*A*···O1	0.86	1.86	2.5395 (19)	134
N3—H3*A*···S1	0.86	2.56	2.9148 (16)	105
N3—H3*A*···O3	0.86	2.21	2.606 (2)	108
C1—H1*B*···O2^{i}	0.93	2.57	3.194 (2)	124
C2—H2*B*···O1^{i}	0.93	2.49	3.284 (2)	143

Symmetry codes: (i) $x, \frac{1}{2} - y, z - \frac{1}{2}$.

After checking their presence in a difference map, all H atoms were included in the refinement in the riding-motion approximation, and allowed to ride on the parent C or N atoms, with C—H = 0.97 Å and N—H = 0.89 Å.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); 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, 1990).

The authors thank the Malaysian Government and Universiti Kebangsaan Malaysia for the research grants IRPA (No. 09-02-02-0163).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–19.

Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.

Shanmuga Sundara Raj, S., Puviarasan, K., Velmurugan, D., Jayanthi, G. & Fun, H.-K. (1999). *Acta Cryst.* C**55**, 1318–1320.

Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.

Sheldrick, G. M. (1997). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.

Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Spek, A. L. (1990). *Acta Cryst.* A**46**, C-34.

Usman, A., Razak, I. A., Satar, S., Kadir, M. A., Yamin, B. M & Fun, H.-K. (2002). *Acta Cryst.* E**58**, o656–o658.

Vankatachalam, T. K., Sudbeck, E. A., Mao, C. & Uckun, F. M. (2001). *Bioorg. Med. Chem. Lett.* **11**, 523–528.

Vig, R., Mao, C., Vankatachalam, T. K., Tuel-Ahlgren, L., Sudbeck, E. A. & Uckun, F. M. (1998). *Bioorg. Med. Chem.* **6**, 1789–1797.