N-Benzyl-2-methylfuran-3-thiocarboxanilide

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The molecule of the title compound, $C_{13}H_{13}NOS$, is non-planar [the dihedral angle between the least-squares planes defined by the phenyl C atoms and furan ring atoms is 71.7 (2)°]. The *anti* conformation of the amide and thio groups in the thioamide fragment is consistent with infinite $C(4)$ chain formation along the b axis *via* N—H···S intermolecular hydrogen bonds [N···S = 3.444 (3) Å] between the amide group and thioamide S atom.

Comment

The crystal structure determination of the title compound, (I), is part of our investigation of aryl-substituted benzothiazole derivatives and their group 12 metal complexes (Tralić-Kulenović *et al.*, 1993; Davidović *et al.*, 1999; Racané *et al.*, 2001; Popović *et al.*, 2003). Thiofuramides are precursors in the synthesis of aryl-substituted benzothiazoles (Fišer-Jakić *et al.*, 1980). A survey of the Cambridge Structural Database (Version 5.25 of November 2003; Allen, 2002) reveals only two thiofuramides, *viz*. *N*-(2-hydroxyethyl)-2-thiofuramide (Galešić *et al.*, 1987) and 5-nitro-*N*-phenyl-2-thiofuramide (Pavlović *et al.*, 2000), and only one 2-methyl derivative of thiofuramides (Popović *et al.*, 2001).

(I)
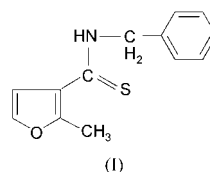
The molecule of (I) is non-planar (Fig. 1). The dihedral angle between the least-squares planes defined by the phenyl C atoms and furan ring atoms is 71.7 (2)°. The analogous dihedral angles are 26.5° in *N*,2-dimethyl-3-thiofuramide (Popović *et al.*, 2001) and 46.3 (1)° [47.0 (1)° for the second molecule] in 5-nitro-*N*-phenyl-2-thiofuramide (Pavlović *et al.*, 2000). The planarity of the thiofuramide moiety is not preserved in (I) [the dihedral angle between planes defined by the furan ring atoms and thioamide atoms N, S1 and C5 is 19.1 (2)°]. This structure contrasts with those of 5-nitro-*N*-phenyl-2-thiofuramide (Pavlović *et al.*, 2000) and *N*-phenyl-2-furamide (Pavlović *et al.*, 2004), where planarity is maintained by the strong N—H···O $S(5)$ intramolecular hydrogen bonds between the amide N atom and the furan O atom.

The value of the S1—C5 bond distance [1.676 (3) Å; Table 1] is in agreement with that found in the X_2—C=S (X = C, N, O and S) structural fragment (1.671 Å; Allen *et al.*, 1987) and with those found in *N*,2-dimethyl-3-thiofuramide [1.667 (3) Å; Popović *et al.*, 2001] and *N*-(2-hydroxyethyl)-2-thiofuramide

[1.673 (3) Å; Galešić *et al.*, 1987]. The corresponding bond in the structure of 5-nitro-*N*-phenyl-2-thiofuramide (Pavlović *et al.*, 2000) is shorter [1.655 (2) Å in both symmetrically independent molecules] because of the presence of the nitro group. The thiofuramide N—C5 bond distance [1.327 (4) Å] possesses significant double-bond character [1.335 (3) Å in *N*,2-dimethyl-3-thiofuramide; 1.344 (3) and 1.348 (3) Å in 5-nitro-*N*-phenyl-2-thiofuramide; 1.317 (4) Å in *N*-(2-hydroxy-ethyl)-2-thiofuramide], in contrast to the N—C6 bond distance [1.456 (4) Å in (I); 1.443 (3) Å in *N*,2-dimethyl-3-thiofuramide; 1.419 (3) and 1.422 (3) Å in 5-nitro-*N*-phenyl-2-thiofuramide; 1.460 (3) Å in *N*-(2-hydroxyethyl)-2-thiofuramide], which is considered as a single C—N bond. The shorter bond distances of 1.419 (3) and 1.422 (3) Å in 5-nitro-*N*-phenyl-2-thiofuramide [compared with the values in other structures) are accompanied by a significantly pronounced π-electron delocalization. The pattern of one shorter and one longer O—Csp^2 furan bond distances found in the 5-nitro-2-furyl fragments (Allen *et al.*, 1987) is not observed in (I) [the O—C3 and O—C4 bonds are 1.376 (6) and 1.366 (5) Å; similar values are found in *N*-(2-hydroxyethyl)-2-thiofuramide, 1.368 (4) and 1.372 (3) Å].

The molecules are connected into infinite *C*(4) chains along the *b* axis (Fig. 2) by N—H⋯S intermolecular hydrogen bonds between the amide group and thioamide S atom (Table 2).

Experimental

Compound (I) was prepared according to a literature procedure (Fišer-Jakić *et al.*, 1980). Single crystals were obtained by the liquid-diffusion crystallization method with dichloromethane as solvent and *n*-hexane as precipitant.

Crystal data

$C_{13}H_{13}NOS$	Mo $K\alpha$ radiation
$M_r = 231.30$	Cell parameters from 4482
Orthorhombic, $P2_12_12_1$	reflections
$a = 5.7601 (11)$ Å	$\theta = 10.0–30.0°$
$b = 8.9620 (19)$ Å	$\mu = 0.25$ mm^{-1}
$c = 22.983 (4)$ Å	$T = 296 (2)$ K
$V = 1186.4 (4)$ Å^3	Prism, colourless
$Z = 4$	$0.51 \times 0.49 \times 0.33$ mm
$D_x = 1.295$ Mg m^{-3}	

Data collection

Oxford Diffraction Xcalibur2 diffractometer with Sapphire 2 CCD detector	1414 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.074$
φ and ω scans	$\theta_{max} = 27.0°$
Absorption correction: none	$h = -7 \rightarrow 7$
19193 measured reflections	$k = -11 \rightarrow 11$
1501 independent reflections	$l = -29 \rightarrow 29$

Refinement

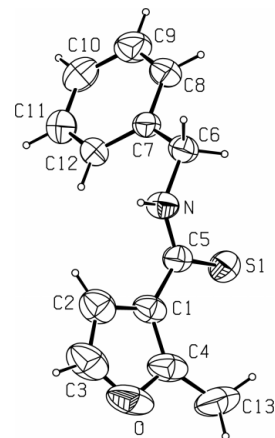|	
Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.047P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.048$	$+ 0.5024P]$
$wR(F^2) = 0.114$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.10$	$(\Delta/\sigma)_{max} < 0.001$
1501 reflections	$\Delta\rho_{max} = 0.19$ e Å^{-3}
150 parameters	$\Delta\rho_{min} = -0.19$ e Å^{-3}
H atoms treated by a mixture of independent and constrained refinement	

Figure 1
The molecular structure of (I), with the atom-labelling scheme and displacement ellipsoids at the 50% probability level.
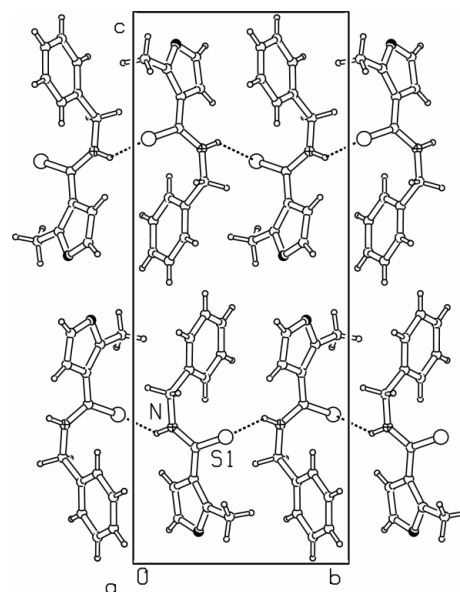

Figure 2
PLATON view of the crystal structure of (I), showing infinite *C*(4) chains extending along the *b* axis. Hydrogen bonds are indicated by dashed lines.

Table 1
Selected geometric parameters (Å, °).

S1—C5	1.676 (3)	C1—C4	1.370 (5)
N—C5	1.327 (4)	C1—C2	1.438 (5)
N—C6	1.456 (4)	C1—C5	1.470 (4)
O—C4	1.366 (5)	C2—C3	1.331 (5)
O—C3	1.376 (6)		
C5—N—C6	124.6 (3)	C4—O—C3	107.4 (3)

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N—H1N⋯S1^i	0.85 (5)	2.71 (5)	3.444 (3)	145 (4)

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

The absolute configuration of (I) could not be determined reliably. The Flack (1983) parameter is unreliable [−0.21 (12)] since the compound is a weak anomalous scatterer, especially considering the use of Mo K_α radiation at ambient temperature. The number of collected Friedel pairs was 1049 (41% of the total unique reflections). At the final stage of refinement, all equivalents, including Friedel opposites, were averaged. H atoms bonded to phenyl, furan, methyl and methylene C atoms were introduced at calculated positions and treated as riding [U_{iso}(H) = 1.5U_{eq}(C) for methyl groups and 1.2U_{eq}(C) for other atoms, and C—H = 0.93, 0.96 and 0.97 Å]. The H atom on the amide N atom was found in a difference Fourier electron-density map and refined freely.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2003); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction, 2003); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON*98 *for Windows* (Spek, 1998); software used to prepare material for publication: *SHELXL*97.

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