Acta Crystallographica Section C
Crystal Structure Communications
ISSN 0108-2701

O-Isopropyl *N*-(2-furoyl)thiocarbamate

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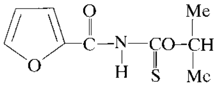
Received 8 May 2000
Accepted 22 May 2000

The title compound, $C_9H_{11}NO_3S$, has crystallographic mirror symmetry, occurs in the thiocarbamate form and is stabilized in an *s-cisoid,s-transoid* conformation with respect to the C—N—C group. There are two intramolecular hydrogen bonds, one between the H atom of the N—H group and the O atom of the furan ring, and the other between the H atom of the secondary carbon of the isopropyl group and the S atom. The packing of the molecules is assumed to be dictated by van der Waals interactions.

Comment

The chemistry of organic sulfur compounds, such as thiocarbamates and their *S*-alkylated products, has been investigated with increasing interest due to their biological activity (Schröpl & Pohloudek-Fabini, 1968). In recent years, *O*-alkyl *N*-acylthiocarbamates have been proposed as intermediates for regio- and chemoselective deoxygenation of primary and secondary aliphatic alcohols (Oba & Nishiyama, 1994). Structural studies of *O*-alkyl *N*-benzoylthiocarbamate showed the C—N bonds to have partial double-bond character, thus giving rise to hindered rotation (Schröder *et al.*, 1995). The present paper reports the crystal structure of *O*-isopropyl *N*-(2-furoyl)thiocarbamate, (I), in order to provide a better understanding of the above-mentioned properties.



(I)

The title compound crystallizes in the thiocarbamate form. A view of the molecule with the atom-numbering scheme is shown in Fig. 1. The asymmetry of the C3—C4—C5 and O1—C4—C5 angles [131.8 (5) and 118.9 (4)°, respectively] is probably caused by repulsion in the C3—H3···O2 system [C3···O2 = 3.006 (6) and H3···O2 = 2.96 Å] and attraction in the N—H···O1 system (see Table 2). The molecule is stabilized in an *s-cisoid,s-transoid* conformation with respect to the C5—N1—C6 group. The C6—O3 bond distance indicates double-bond character, while the C5—N1 and C6—N1 distances indicate Csp^2—Nsp^2 single-bond character. This indicates that there is π–π conjugation along S1—C6—O3, but not along O2—C5—N1 and N1—C6—S1, as reported for *O*-alkyl *N*-benzoylthiocarbamate (Schröder *et al.*, 1995).

There are two intramolecular hydrogen bonds, one between the N—H group and the O atom of the furan ring, and the other between the H atom of the secondary carbon of the isopropyl group and the S atom. The packing of the molecules is assumed to be dictated by short contacts and van der Waals interactions.

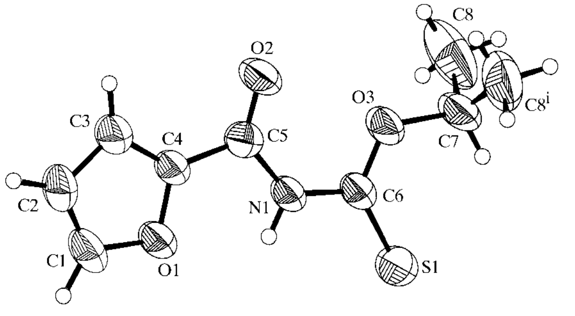


Figure 1
Plot of (I) showing the atomic numbering scheme. Displacement ellipsoids are drawn at 50% probability level for non-H atoms. [Symmetry code: (i) $x, \frac{1}{2} - y, z$.]

Experimental

The title compound was synthesized by the reaction of furoyl isothiocyanate with isopropyl alcohol using acetone as solvent (Schröder *et al.*, 1995). Recrystallization from methanol gave crystals suitable for X-ray analysis.

Crystal data

$C_9H_{11}NO_3S$
$M_r = 213.25$
Monoclinic, $P2_1/m$
$a = 7.679$ (4) Å
$b = 7.004$ (5) Å
$c = 10.372$ (5) Å
$\beta = 107.43$ (4)°
$V = 532.2$ (5) Å^3
$Z = 2$

$D_x = 1.331$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 10$–$15°$
$\mu = 0.285$ mm^{-1}
$T = 293$ (2) K
Prism, colourless
0.6 × 0.2 × 0.1 mm

Data collection

Stoe Stadi-4 four-circle diffractometer
ω scans
1980 measured reflections
1019 independent reflections
457 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.058$

$\theta_{\max} = 24.99°$
$h = -2 \rightarrow 9$
$k = -2 \rightarrow 8$
$l = -12 \rightarrow 12$
2 standard reflections
frequency: 60 min
intensity decay: <2.0%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.134$
$S = 0.979$
1019 reflections
84 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0545P)^2 + 0.0084P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.19$ e Å^{-3}
$\Delta\rho_{\min} = -0.19$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.038 (7)

Table 1
Selected geometric parameters (Å, °).

S1—C6	1.636 (5)	O3—C6	1.290 (6)
O1—C4	1.356 (6)	O3—C7	1.477 (6)
O1—C1	1.375 (6)	N1—C6	1.379 (6)
O2—C5	1.194 (6)	N1—C5	1.387 (6)
C6—O3—C7	120.1 (4)	N1—C5—C4	112.6 (5)
C6—N1—C5	131.4 (4)	O3—C6—N1	111.9 (4)
O2—C5—N1	124.8 (5)	O3—C6—S1	128.4 (4)
O2—C5—C4	122.5 (5)	N1—C6—S1	119.7 (4)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1	0.86	2.19	2.654 (6)	114
C1—H1A···O2^{i}	0.93	2.40	3.322 (7)	172
C7—H7···S1	0.98	2.55	3.048 (7)	111

Symmetry code: (i) $x - 1, y, z$.

H atoms were calculated geometrically and included in the refinement, but were restrained to ride on their parent atoms. The isotropic displacement parameters of the H atoms were fixed at $1.3U_{\rm eq}$ of their parent atoms.

Data collection: *DIF*4 (Stoe & Cie, 1992); cell refinement: *DIF*4; data reduction: *REDU*4 (Stoe & Cie, 1992); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *DIAMOND* (Bergerhoff, 1996); software used to prepare material for publication: *SHELXL*97 and *PARST* (Nardelli, 1983, 1995).

HNdeA thanks KU Leuven (Belgium) for support through IRO scholarships.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1477). Services for accessing these data are described at the back of the journal.

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