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N,2-Dimethyl-3-furanthiocarboxanilide

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.053
wR factor = 0.167
Data-to-parameter ratio = 15.4

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

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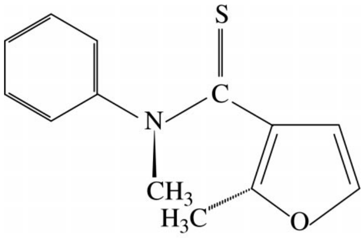
The title compound, $C_{13}H_{13}NOS$, was prepared and structurally characterized. The molecule is not planar; the furan ring is inclined at an angle of 26.5° to the phenyl ring. The crystal structure is dominated by van der Waals forces.

Comment

The biological activity of *N*-arylfuranthiocarboxamides has been well known for years (Landquist, 1984). *N*-Aryl-3-furanthiocarboxamides were linvestigated to a lerrer extent due to difficulties in their synthesis and unrecognized pharmaceutical importance (Dodd *et al.*, 1970). Fungicidal and insecticidal activity of the derivatives of 3-furancarboxylic acid has encouraged the synthesis of new *N*-aryl-3-furanthiocarboxamide derivatives.



(I)

In order to get more information on the structural criteria that an organic molecule must fulfill to exhibit such activity, we investigated the structure of the title compound, (I) (Fig. 1).

Its antibacterial activity is currently under preliminary investigation. A survey of the Cambridge Structural Database (Allen & Kennard, 1993) revealed only two structures involving furanthioamide, *viz*. *N*-(2-hydroxyethyl)-2-thiofuramide (FELYIE; Galešić *et al.*, 1987) and 5-nitro–*N*-phenyl-2-thiofuramide (KOPGUR; Pavlović *et al.*, 2000). The $Csp^2{=}Csp^2$ and $Csp^2{-}O$ bond distances within the furan ring are in agreement with literature data (Allen *et al.*, 1987). The $Csp^2{-}Csp^2$ bond distance value is slightly longer than the average value of 1.432 Å (Allen *et al.*, 1987). The C=S bond length is consistent with the average value found in the fragment $X_2C{=}S$ (X = C, N, O, S) of 1.671 Å, as well as in *N*-(2-hydroxyethyl)-2-thiofuramide (Galešić *et al.*, 1987). The C—N distances of the thioamide moiety correspond to single C—N bond values, while N1—C6 is quite shorter as a result of π-electron delocalization. In the structure of *N*-(2-hydroxyethyl)-2-thiofuramide (Galešić *et al.*, 1987), the analogous bond length is even shorter, 1.317 (4) Å. The sum of the angle

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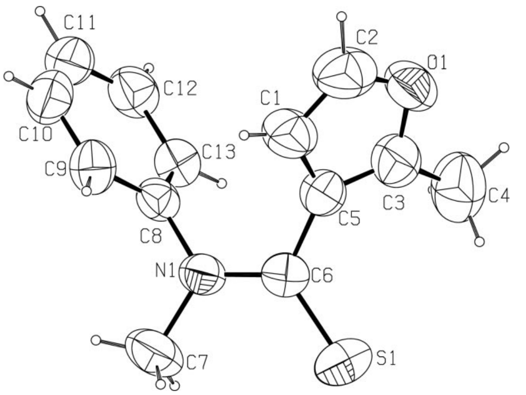

Figure 1
PLATON drawing (Spek, 1998) with the atom-numbering scheme. The displacement ellipsoids are at the 50% probability level for the non-H atoms. H atoms are shown as spheres of arbitrary radii.

values around the thioamido N atom is 358.7 (2)°, confirming sp^2-hybridization. The title molecule is not planar, exibiting twisting around the single Nsp^2—Csp^2 bond (N1—C6). The measure of twisting is described by the torsion angle C3—C5—C6—N1 of −140.9 (2)°.

Experimental

The title compound was prepared by thionation of the corresponding amide with phosphorus pentasulfide, according to the reported procedure (Hahn *et al.*, 1970). By recrystallization from ethanol prismatic crystals of good diffraction quality were obtained.

Crystal data

$C_{13}H_{13}NOS$
$M_r = 231.30$
Orthorhombic, *Pbca*
$a = 9.577$ (3) Å
$b = 16.039$ (3) Å
$c = 16.127$ (2) Å
$V = 2477.2$ (10) Å^3
$Z = 8$
$D_x = 1.240$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 33 reflections
$\theta = 11.9$–16.4°
$\mu = 0.24$ mm^{-1}
$T = 293$ (2) K
Prism, yellow
0.34 × 0.26 × 0.11 mm

Data collection

Philips PW1100 diffractometer updated by Stoe
$\theta/2\theta$ scans
2671 measured reflections
2671 independent reflections
1280 reflections with $I > 2\sigma(I)$
$\theta_{max} = 27.0°$
$h = 0 \rightarrow 12$
$k = 0 \rightarrow 20$
$l = 0 \rightarrow 20$
4 standard reflections
frequency: 90 min
intensity decay: 2.4%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.167$
$S = 0.97$
2671 reflections
174 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0849P)^2 + 0.4315P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.35$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.015 (2)

Table 1
Selected geometric parameters (Å, °).

C5—C6	1.483 (4)	N1—C8	1.443 (3)
C6—N1	1.335 (3)	N1—C7	1.468 (3)
C6—S1	1.667 (3)		
N1—C6—C5	115.5 (2)	C6—N1—C8	121.9 (2)
N1—C6—S1	123.2 (2)	C6—N1—C7	121.3 (2)
C5—C6—S1	121.1 (2)	C8—N1—C7	115.4 (2)

The positions of the H atoms attached to the C atoms of the furan and phenyl ring were generated geometrically and refined using the riding model. Those belonging to the methyl groups were located in a difference Fourier map.

Data collection: *STADI*4 (Stoe & Cie, 1995); cell refinement: *STADI*4; data reduction: *X-RED* (Stoe & Cie, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 1998); software used to prepare material for publication: *SHELXL*97.

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References

Allen, F. H. & Kennard, O. (1993). *Chem. Des. Autom. News*, **8**, 1, 31–37.
Allen, F. H., Kennard, O., Watson, D. G., Brammer, L. & Orpen, A. G. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–19.
Dodd, M. C., Stillman, W. B., Roys, M. & Crosby, C. (1970). *J. Pharmacol. Exp. Ther.* **82**, 11–18.
Galešić, N., Vlahov, A. & Galešić, M. (1987). *Acta Cryst.* C**43**, 479–482.
Hahn, V., Zupanc, S. & Jakopčić, K. (1970). *Croat. Chem. Acta*, **42**, 585–588.
Landquist, J. K. (1984). *Application as Pharmaceuticals*, in *Comprehensive Heterocyclic Chemistry*, Vol. 1/1.06, edited by A. R. Katritzky & C. W. Rees, pp. 531–597. Oxford: Pergamon Press.
Pavlović, G. Mance, A. D. & Jakopčić, K. (2000). *Acta Cryst.* C**56**, 604–606.
Sheldrick, G. M. (1997). *SHELXL*97 and *SHELXS*97. University of Göttingen, Germany.
Stoe & Cie (1995). *STADI*4 and *X-RED*. Stoe & Cie, Darmstadt, Germany.
Spek, L. (1998). *PLATON*98 *for Windows*. University of Utrecht, The Netherlands.