

5-(Furan-2-yl)-1,3,4-oxadiazole-2(3H)-thione

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Key indicators

Single-crystal X-ray study

$T = 100\text{ K}$

Mean $\sigma(\text{C}-\text{C}) = 0.002\text{ \AA}$

R factor = 0.030

wR factor = 0.107

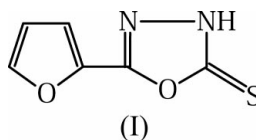
Data-to-parameter ratio = 18.5

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

The molecule of the title compound, $\text{C}_6\text{H}_4\text{N}_2\text{O}_2\text{S}$, is planar. An intermolecular $\text{N}-\text{H}\cdots\text{S}$ hydrogen bond is formed between the oxadiazole group and the S atom of a neighbouring molecule; the pair of molecules involve a twofold screw axis along the [001] direction.

Comment

Some studies have been published on the electronic structures and thiol–thione tautomeric equilibrium of 1,3,4-oxadiazole-2-thione derivatives (Aydoğan *et al.*, 2002; Charistos *et al.*, 1994; Tsoleridis *et al.*, 1997). It is known that 1,3,4-oxadiazole-2-thione derivatives show a broad spectrum of biological activities (Ram & Vlietinck, 1988; Boschelli *et al.*, 1993; Bahadur & Pandey, 1980).



The title compound, (I), has been newly synthesized by the ring-closure reaction of furan-2-carboxylic acid hydrazide with carbon disulfide. The present study was undertaken to determine the molecular and crystal structure of (I).

A view of the molecule is shown in Fig. 1. The $\text{Csp}^2=\text{Csp}^2$ and Csp^2-O bond distances within the furan ring are in agreement with the literature data (Allen *et al.*, 1987). The $\text{C}=\text{S}$ bond length, 1.659 (2) Å, is similar to that in *N*-(2-hydroxyethyl)-2-thiofuramide (Galešić *et al.*, 1987). The $\text{N1}-\text{N2}$ distance [1.381 (2) Å] in the oxadiazole moiety corresponds to the single $\text{N}-\text{N}$ bond length value. To the best of our knowledge, only one other 1,3,4-oxadiazole-2-thione crystal structure has been published (Ziyaev *et al.*, 1992) and the geometry of the ring agrees well with that in the title compound.

The molecular structure of (I) is planar, the maximum deviation of the non-H atoms being 0.005 (1) Å. The dihedral angle between the mean planes of the furan and oxadiazole

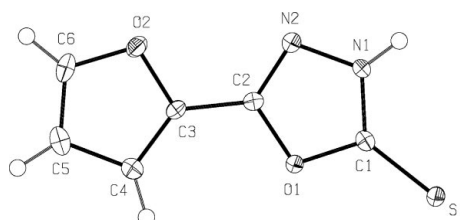


Figure 1

A view of (I) with the atom-numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level.

rings is $2.3(1)^\circ$ (Nardelli, 1995). In the crystal structure, the molecules are linked by $N1-H1 \cdots S1^i$ [symmetry code: (i) $1-x, 1-y, 1-z$] hydrogen bonds.

Experimental

A mixture of furan-2-carboxylic acid hydrazide (0.01 mol, 1.26 g), sodium hydroxide (0.01 mol, 0.4 g) carbon disulfide (0.02 mol, 1.2 ml) and absolute ethanol (100 ml) was heated under reflux for 12 h. The excess solvent was removed by vacuum evaporation, and the residue was dissolved in water and acidified with acetic acid. The product was recrystallized from water–ethanol (60:40) (yield 55%, m.p. 408–410 K). IR (cm^{-1}): 3356 (NH), 1642 (C=N), 1255 (C=S); ^1H NMR (CDCl_3): δ 6.56–7.65 (*m*, 3H, furyl), 13.70 (*s*, 1H, NH or SH).

Crystal data

$\text{C}_6\text{H}_4\text{N}_2\text{O}_2\text{S}$	$D_x = 1.585 \text{ Mg m}^{-3}$
$M_r = 168.18$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 145 reflections
$a = 5.194(5) \text{ \AA}$	$\theta = 6\text{--}20.0^\circ$
$b = 20.102(5) \text{ \AA}$	$\mu = 0.40 \text{ mm}^{-1}$
$c = 6.914(5) \text{ \AA}$	$T = 100 \text{ K}$
$\beta = 102.573(5)^\circ$	Block, pale yellow
$V = 704.6(9) \text{ \AA}^3$	$0.33 \times 0.28 \times 0.19 \text{ mm}$
$Z = 4$	

Data collection

Nonius KappaCCD diffractometer	1618 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{\text{int}} = 0.025$
Absorption correction: multi-scan (SADABS; Sheldrick, 1997)	$\theta_{\text{max}} = 29.0^\circ$
$T_{\text{min}} = 0.879$, $T_{\text{max}} = 0.928$	$h = -6 \rightarrow 7$
15534 measured reflections	$k = -27 \rightarrow 27$
1855 independent reflections	$l = -9 \rightarrow 9$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.030$	$w = 1/[\sigma^2(F_o^2) + (0.08P)^2]$
$wR(F^2) = 0.107$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.09$	$(\Delta/\sigma)_{\text{max}} = 0.001$
1855 reflections	$\Delta\rho_{\text{max}} = 0.33 \text{ e \AA}^{-3}$
100 parameters	$\Delta\rho_{\text{min}} = -0.29 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (\AA , $^\circ$).

S1—C1	1.659 (2)	O2—C6	1.369 (2)
O1—C1	1.373 (2)	N1—N2	1.381 (2)
O1—C2	1.3797 (19)	N1—C1	1.333 (2)
O2—C3	1.368 (2)	N2—C2	1.293 (2)
C1—O1—C2	105.37 (10)	O1—C2—C3	117.43 (10)
C3—O2—C6	105.43 (10)	O1—C2—N2	113.52 (10)
N2—N1—C1	113.03 (10)	N2—C2—C3	129.04 (11)
N1—N2—C2	102.77 (10)	O2—C3—C4	111.37 (11)
S1—C1—N1	130.93 (10)	O2—C3—C2	115.95 (10)
S1—C1—O1	123.77 (11)	O2—C6—C5	111.03 (12)
O1—C1—N1	105.30 (10)		

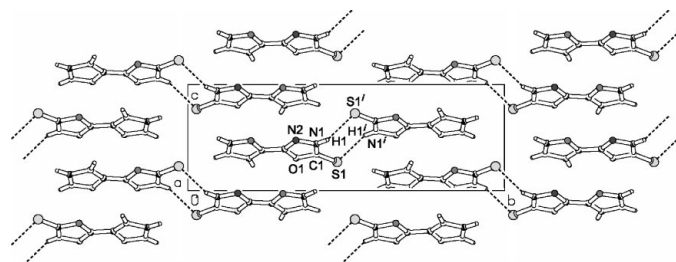


Figure 2

The packing of (I) and the intermolecular hydrogen bonds, shown as dashed lines. [Symmetry code: (i) $1-x, 1-y, 1-z$.]

Table 2

Hydrogen-bonding geometry (\AA , $^\circ$).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$N1-H1 \cdots S1^i$	0.86	2.53	3.321 (3)	154

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

The H atoms were placed in calculated positions ($C-H = 0.93 \text{ \AA}$ and $N-H = 0.86 \text{ \AA}$) and refined in the riding-model approximation; the $U_{\text{iso}}(\text{H})$ values were set equal to $1.2U_{\text{eq}}(\text{parent atom})$.

Data collection: COLLECT (Nonius, 1999); cell refinement: EVALCCD (Duisenberg *et al.*, 2003); data reduction: EVALCCD; 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); software used to prepare material for publication: WinGX publication routines (Farrugia, 1999).

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