organic compounds

(E)-N′-(3-Thienylmethylene)furan-2-carbohydrazide

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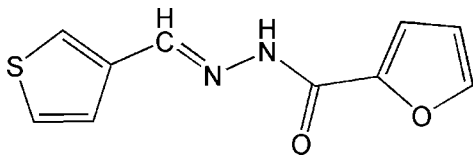
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In the title compound, $C_{10}H_8N_2O_2S$, the dihedral angle between the thiophene and furan planes is 12.3 (1)°. The molecules are linked *via* weak intermolecular N—H···O, C—H···S and C—H···O hydrogen bonds, forming a two-dimensional network parallel to the *ab* plane.

Related literature

For general background, see: Belloni *et al.* (2005); Kahwa *et al.* (1986); Parashar *et al.* (1988); Santos *et al.* (2001); Tynan *et al.* (2005).

Experimental

Crystal data

$C_{10}H_8N_2O_2S$	V = 972.0 (2) Å³
M_r = 220.24	Z = 4
Orthorhombic, $Pca2_1$	Mo $K\alpha$ radiation
a = 10.1150 (15) Å	μ = 0.31 mm⁻¹
b = 4.7969 (7) Å	T = 294 (2) K
c = 20.033 (3) Å	0.24 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer	Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996) T_{min} = 0.929, T_{max} = 0.946

4486 measured reflections
1694 independent reflections
1616 reflections with $I > 2\sigma(I)$
R_{int} = 0.018

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.023	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.065	$\Delta\rho_{max}$ = 0.14 e Å⁻³
S = 1.11	$\Delta\rho_{min}$ = −0.14 e Å⁻³
1694 reflections	Absolute structure: Flack (1983), with 811 Friedel pairs
140 parameters	Flack parameter: 0.04 (7)
2 restraints	

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O1ⁱ	0.891 (9)	2.163 (12)	3.010 (2)	158 (2)
C1—H1···S1ⁱⁱ	0.93	2.86	3.438 (2)	122
C8—H8···O1ⁱ	0.93	2.50	3.328 (3)	149

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

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2444).

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supplementary materials

(*E*)-*N'*-(3-Thienylmethylene)furan-2-carbohydrazide

Z.-L. Jing, M. Yu and X. Chen

Comment

In order to establish control over the preparation of crystalline solid materials so that their architecture and properties are predictable (Belloni *et al.*, 2005; Tynan *et al.*, 2005; Parashar *et al.*, 1988), the synthesis of new and designed crystal structures has become a major strand of modern chemistry. Metal complexes based on Schiff bases have attracted much attention because they can be utilized as model compounds of the active centres in various proteins and enzymes (Kahwa *et al.*, 1986; Santos *et al.*, 2001). As part of an investigation of the coordination properties of Schiff bases functioning as ligands, we report the synthesis and crystal structure of the title compound, (I).

In the molecular structure of the compound (I) (Fig. 1), the geometric parameters are normal. The thiophene ring (C1–C4/ S1) is planar within ±0.002 (1) Å. The furan ring (C7–C10/O2) is essentially planar, with a maximum deviation from the mean plane of 0.004 (1) Å for atom C10. The dihedral angle between these planes is 12.3 (1)°.

The molecules are linked *via* weak intermolecular N—H···O, C—H···S and C—H···O type hydrogen bonds (Table 1), to form a two-dimensional network parallel to the *ab* plane, as illustraed in Fig.2.

Experimental

An anhydrous ethanol solution (50 ml) of thiophene-3-carbaldehyde (1.12 g, 10 mmol) was added to an anhydrous ethanol solution (50 ml) of furan-2-carbohydrazide (1.26 g, 10 mmol), and the mixture was stirred at 350 K for 6 h under N_2, whereupon a colourless precipitate appeared. The product was isolated, recrystallized from anhydrous ethanol and then dried *in vacuo* to give pure compound (I) in 79% yield. Colourless single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an anhydrous ethanol solution.

Refinement

The N-bound H atom was located in a difference Fourier map and refined with a N—H distance restraint of 0.90 (1) Å. C-bound H atoms were included in calculated positions [C—H = 0.93 Å] and refined using a riding model, with $U_{iso}(H) = 1.2U_{eq}(C)$.
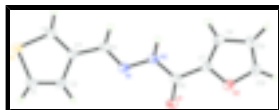
Figures

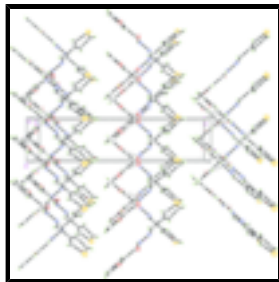Fig. 1. The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level.

Fig. 2. The crystal packing of (I), viwed down the *a* axis. Hydrogen bonds are indicated by dashed lines.

(*E*)-*N*'-(3-Thienylmethylene)furan-2-carbohydrazide

Crystal data

$C_{10}H_8N_2O_2S$	$F_{000} = 456$
$M_r = 220.24$	$D_x = 1.505$ Mg m^{-3}
Orthorhombic, $Pca2_1$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: P 2c -2ac	Cell parameters from 3232 reflections
$a = 10.1150$ (15) Å	$\theta = 4.0–26.2°$
$b = 4.7969$ (7) Å	$\mu = 0.31$ mm^{-1}
$c = 20.033$ (3) Å	$T = 294$ (2) K
$V = 972.0$ (2) Å^3	Block, colourless
$Z = 4$	$0.24 \times 0.20 \times 0.18$ mm

Data collection

Bruker SMART CCD area-detector diffractometer	1694 independent reflections
Radiation source: fine-focus sealed tube	1616 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.018$
$T = 294(2)$ K	$\theta_{max} = 25.0°$
φ and ω scans	$\theta_{min} = 2.0°$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -10 \rightarrow 12$
$T_{min} = 0.929$, $T_{max} = 0.946$	$k = -5 \rightarrow 4$
4486 measured reflections	$l = -23 \rightarrow 23$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.023$	$w = 1/[\sigma^2(F_o^2) + (0.0391P)^2 + 0.0578P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.065$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.11$	$\Delta\rho_{max} = 0.14$ e Å^{-3}

1694 reflections	$\Delta\rho_{min} = -0.14$ e Å^{-3}
140 parameters	Extinction correction: none
2 restraints	Absolute structure: Flack (1983), with 811 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.04 (7)
Secondary atom site location: difference Fourier map	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of F$^2 > 2$sigma(F^2) is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
S1	0.22029 (5)	1.02129 (9)	0.32352 (3)	0.04200 (14)
O1	−0.15782 (12)	0.0679 (3)	0.10495 (7)	0.0473 (4)
O2	−0.11967 (13)	−0.2927 (3)	0.00357 (7)	0.0547 (4)
N1	0.04650 (14)	0.3353 (3)	0.16534 (7)	0.0352 (3)
N2	0.06307 (14)	0.1461 (3)	0.11429 (8)	0.0352 (3)
C1	0.05895 (18)	0.9185 (4)	0.32440 (11)	0.0412 (4)
H1	−0.0038	0.9854	0.3543	0.049*
C2	0.03343 (17)	0.7284 (4)	0.27648 (10)	0.0398 (4)
H2	−0.0493	0.6488	0.2696	0.048*
C3	0.14683 (17)	0.6629 (4)	0.23753 (9)	0.0324 (4)
C4	0.2551 (2)	0.8089 (4)	0.25789 (10)	0.0383 (4)
H4	0.3382	0.7940	0.2383	0.046*
C5	0.1512 (2)	0.4638 (4)	0.18335 (9)	0.0360 (4)
H5	0.2307	0.4291	0.1615	0.043*
C6	−0.04417 (19)	0.0210 (4)	0.08752 (9)	0.0341 (4)
C7	−0.01324 (16)	−0.1763 (4)	0.03386 (9)	0.0348 (4)
C8	0.0966 (2)	−0.2736 (5)	0.00599 (11)	0.0535 (6)
H8	0.1827	−0.2267	0.0177	0.064*
C9	0.0584 (2)	−0.4613 (5)	−0.04464 (12)	0.0536 (6)
H9	0.1139	−0.5610	−0.0729	0.064*
C10	−0.0714 (3)	−0.4667 (5)	−0.04373 (12)	0.0538 (5)
H10	−0.1232	−0.5756	−0.0718	0.065*
H2A	0.1439 (12)	0.100 (5)	0.1005 (10)	0.043 (6)*

supplementary materials

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0360 (2)	0.0452 (3)	0.0448 (2)	−0.00471 (19)	−0.0011 (2)	−0.0056 (2)
O1	0.0262 (7)	0.0623 (9)	0.0533 (8)	0.0012 (6)	0.0024 (6)	−0.0137 (7)
O2	0.0331 (7)	0.0682 (10)	0.0629 (9)	−0.0067 (6)	−0.0010 (7)	−0.0236 (8)
N1	0.0298 (7)	0.0381 (9)	0.0378 (8)	0.0020 (6)	−0.0006 (6)	−0.0029 (7)
N2	0.0256 (7)	0.0403 (9)	0.0397 (8)	0.0018 (7)	0.0017 (6)	−0.0063 (7)
C1	0.0333 (10)	0.0482 (10)	0.0422 (9)	0.0001 (7)	0.0064 (10)	0.0007 (11)
C2	0.0291 (9)	0.0429 (10)	0.0475 (11)	−0.0016 (8)	0.0014 (8)	−0.0022 (9)
C3	0.0268 (9)	0.0334 (9)	0.0372 (10)	0.0013 (7)	0.0002 (7)	0.0042 (8)
C4	0.0299 (9)	0.0403 (10)	0.0445 (10)	−0.0010 (8)	0.0047 (7)	−0.0001 (8)
C5	0.0293 (9)	0.0382 (10)	0.0407 (10)	0.0023 (7)	0.0016 (8)	0.0021 (8)
C6	0.0294 (12)	0.0367 (10)	0.0362 (10)	0.0005 (7)	0.0005 (7)	0.0035 (7)
C7	0.0309 (9)	0.0367 (9)	0.0367 (9)	−0.0035 (7)	−0.0036 (7)	0.0029 (8)
C8	0.0283 (10)	0.0697 (15)	0.0624 (13)	0.0015 (9)	−0.0026 (9)	−0.0237 (11)
C9	0.0418 (13)	0.0604 (13)	0.0585 (13)	0.0018 (9)	0.0065 (10)	−0.0197 (12)
C10	0.0513 (13)	0.0544 (12)	0.0558 (13)	−0.0036 (10)	−0.0070 (11)	−0.0190 (11)

Geometric parameters (Å, °)

S1—C4	1.7003 (19)	C2—H2	0.93
S1—C1	1.7050 (19)	C3—C4	1.363 (3)
O1—C6	1.222 (2)	C3—C5	1.446 (3)
O2—C10	1.354 (3)	C4—H4	0.93
O2—C7	1.356 (2)	C5—H5	0.93
N1—C5	1.277 (2)	C6—C7	1.466 (3)
N1—N2	1.378 (2)	C7—C8	1.328 (3)
N2—C6	1.351 (2)	C8—C9	1.410 (3)
N2—H2A	0.891 (9)	C8—H8	0.93
C1—C2	1.349 (3)	C9—C10	1.313 (3)
C1—H1	0.93	C9—H9	0.93
C2—C3	1.423 (2)	C10—H10	0.93
C4—S1—C1	91.90 (10)	N1—C5—C3	120.34 (18)
C10—O2—C7	106.30 (16)	N1—C5—H5	119.8
C5—N1—N2	115.26 (15)	C3—C5—H5	119.8
C6—N2—N1	119.32 (15)	O1—C6—N2	124.07 (17)
C6—N2—H2A	120.3 (15)	O1—C6—C7	121.93 (17)
N1—N2—H2A	120.3 (15)	N2—C6—C7	113.98 (16)
C2—C1—S1	111.79 (15)	C8—C7—O2	109.34 (17)
C2—C1—H1	124.1	C8—C7—C6	135.53 (17)
S1—C1—H1	124.1	O2—C7—C6	115.13 (15)
C1—C2—C3	112.67 (17)	C7—C8—C9	107.30 (18)
C1—C2—H2	123.7	C7—C8—H8	126.3
C3—C2—H2	123.7	C9—C8—H8	126.3
C4—C3—C2	111.75 (16)	C10—C9—C8	106.1 (2)
C4—C3—C5	122.64 (18)	C10—C9—H9	127.0

C2—C3—C5	125.60 (17)	C8—C9—H9	127.0
C3—C4—S1	111.89 (15)	C9—C10—O2	111.0 (2)
C3—C4—H4	124.1	C9—C10—H10	124.5
S1—C4—H4	124.1	O2—C10—H10	124.5
C5—N1—N2—C6	−173.46 (17)	N1—N2—C6—C7	−179.80 (14)
C4—S1—C1—C2	0.24 (17)	C10—O2—C7—C8	0.4 (2)
S1—C1—C2—C3	−0.1 (2)	C10—O2—C7—C6	−179.86 (17)
C1—C2—C3—C4	−0.1 (2)	O1—C6—C7—C8	−177.7 (2)
C1—C2—C3—C5	178.91 (18)	N2—C6—C7—C8	3.3 (3)
C2—C3—C4—S1	0.3 (2)	O1—C6—C7—O2	2.6 (3)
C5—C3—C4—S1	−178.77 (15)	N2—C6—C7—O2	−176.41 (15)
C1—S1—C4—C3	−0.29 (15)	O2—C7—C8—C9	0.1 (3)
N2—N1—C5—C3	−178.56 (15)	C6—C7—C8—C9	−179.7 (2)
C4—C3—C5—N1	−178.84 (17)	C7—C8—C9—C10	−0.5 (3)
C2—C3—C5—N1	2.3 (3)	C8—C9—C10—O2	0.7 (3)
N1—N2—C6—O1	1.2 (3)	C7—O2—C10—C9	−0.7 (3)

Hydrogen-bond geometry (Å, °)

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N2—H2A⋯O1[i]	0.891 (9)	2.163 (12)	3.010 (2)	158 (2)
C1—H1⋯S1[ii]	0.93	2.86	3.438 (2)	122
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Symmetry codes: (i) $x+1/2$, $−y$, z; (ii) $x−1/2$, $−y+2$, z.

Fig. 1

Fig. 2