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(*E*)-*N*′-[(5-Methyl-2-thienyl)methylene]-furan-2-carbohydrazide monohydrate

Zuo-Liang Jing,* Ming Yu and Xin Chen

College of Sciences, Tianjin University of Science and Technology, Tianjin 300457, People's Republic of China
Correspondence e-mail: jzl74@tust.edu.cn

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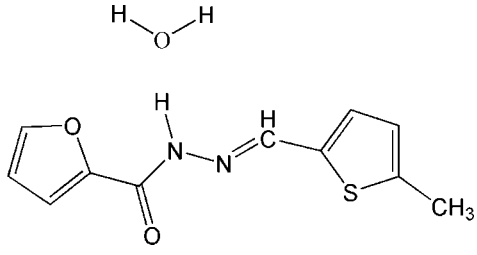
Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.003 Å; R factor = 0.044; wR factor = 0.091; data-to-parameter ratio = 16.5.

In the title compound, $C_{11}H_{10}N_2O_2S\cdot H_2O$, the dihedral angle between the planes of the thiophene and furan rings is 35.20 (2)°. Molecules are linked *via* weak intermolecular N—H···O and O—H···O hydrogen bonds to form a zigzag packing arrangement.

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

$C_{11}H_{10}N_2O_2S\cdot H_2O$
M_r = 252.29
Orthorhombic, $Pna2_1$
a = 19.789 (8) Å
b = 4.809 (2) Å
c = 12.106 (6) Å
V = 1152.1 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.28 mm^{-1}
T = 113 (2) K
0.14 × 0.10 × 0.08 mm

Data collection

Rigaku Saturn diffractometer
Absorption correction: multi-scan (Jacobson, 1998)
T_{min} = 0.962, T_{max} = 0.978
13209 measured reflections
2710 independent reflections
2591 reflections with $I > 2\sigma(I)$
R_{int} = 0.047

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.092
S = 1.06
2710 reflections
164 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}
Absolute structure: Flack (1983), with 1289 Freidel pairs
Flack parameter: 0.10 (8)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O3	0.88 (3)	1.98 (3)	2.829 (3)	163 (2)
O3—H3B···O1^{i}	0.76 (3)	2.09 (3)	2.843 (2)	174 (3)
O3—H3A···O1^{ii}	0.87 (3)	2.01 (3)	2.835 (3)	158 (3)

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

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *CrystalStructure* (Rigaku/MSC, 2005); software used to prepare material for publication: *CrystalStructure*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2385).

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supplementary materials

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(*E*)-*N*'-[(5-Methyl-2-thienyl)methylene]furan-2-carbohydrazide monohydrate

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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 Shiff 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. One molecules of the unit, the thiophen ring (C7–C10/S1) is approximately planar, with a maximum deviation from the mean plane of 0.0017 (3) Å for atom S1, as the furan group (C2–C5/O2) is approximately planar, with a maximum deviation from the mean plane of 0.0365 (5) Å for atom O2. The dihedral angle between these two planes is 35.20 (2)°. The crystal structure shows that the title compound includes a water molecule,which are linked *via* weak intermolecular N—H···O and O—H···O hydrogen bonds (Table 1), to form a zigzag packing arrangement, as illustrated in Fig.2.

Experimental

An anhydrous ethanol solution (50 ml) of 5-methylthiophene-2-carbaldehyde (1.26 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 81% yield. Colorless 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 and the H atoms of the water molecule were located in a difference Fourier map and refined freely. C-bound H atoms were included in calculated positions, with C—H = 0.93–0.96 Å, and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C).

Figures

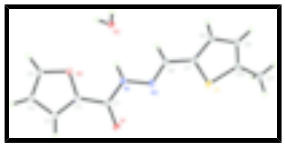

Fig. 1. The structure of the title molecule (I). Displacement ellipsoids are drawn at the 30% probability level.

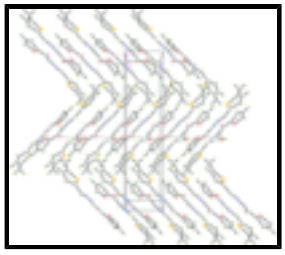

Fig. 2. The crystal packing of (I), viwed down the *a* axis. Hydrogen bonds are indicated by dashed lines.

(*E*)—*N*'-[(5-Methyl-2-thienyl)methylene]furan-2-carbohydrazide monohydrate

Crystal data

$C_{11}H_{10}N_2O_2S·H_2O$	F_{000} = 528
M_r = 252.29	D_x = 1.455 Mg m^{-3}
Orthorhombic, $Pna2_1$	Mo $K\alpha$ radiation λ = 0.71070 Å
Hall symbol: P 2c -2n	Cell parameters from 3302 reflections
a = 19.789 (8) Å	θ = 2.7–25.0º
b = 4.809 (2) Å	μ = 0.28 mm^{-1}
c = 12.106 (6) Å	T = 113 (2) K
V = 1152.1 (9) Å^3	Prism, colourless
Z = 4	0.14 × 0.10 × 0.08 mm

Data collection

Rigaku Saturn diffractometer	2591 reflections with $I > 2\sigma(I)$
Radiation source: rotating anode	R_{int} = 0.047
Monochromator: confocal	θ_{max} = 27.8º
T = 113(2) K	θ_{min} = 2.1º
ω scans	h = −25→25
Absorption correction: multi-scan (Jacobson, 1998)	k = −6→6
T_{min} = 0.962, T_{max} = 0.978	l = −15→15
13209 measured reflections	Standard reflections: ?
2710 independent reflections	

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
$R[F^2 > 2\sigma(F^2)]$ = 0.044	$w = 1/[\sigma^2(F_o^2) + (0.0422P)^2 + 0.2449P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.092	$(\Delta/\sigma)_{max} < 0.001$
S = 1.06	$\Delta\rho_{max}$ = 0.29 e Å^{-3}

2710 reflections	$\Delta\rho_{min} = -0.30$ e Å^{-3}
164 parameters	Extinction correction: none
1 restraint	Absolute structure: Flack (1983), 1289 Freidel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.10 (8)
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 $F^2 > 2\text{sigma}(F^2)$ 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	0.32329 (3)	0.81744 (11)	0.57447 (5)	0.02311 (15)
O1	0.53841 (8)	0.1879 (3)	0.59068 (13)	0.0212 (4)
O2	0.54514 (7)	−0.0756 (3)	0.86546 (13)	0.0199 (3)
O3	0.44096 (9)	0.3131 (4)	0.97064 (14)	0.0240 (4)
H3A	0.4516 (13)	0.436 (6)	1.020 (3)	0.036*
H3B	0.4473 (15)	0.186 (6)	1.006 (3)	0.036*
N1	0.48345 (10)	0.3262 (3)	0.74758 (16)	0.0180 (4)
H1	0.4770 (13)	0.299 (5)	0.818 (2)	0.022*
N2	0.43996 (9)	0.5000 (4)	0.68901 (16)	0.0191 (4)
C1	0.52804 (11)	0.1685 (4)	0.69145 (18)	0.0182 (5)
C2	0.56544 (10)	−0.0345 (4)	0.75791 (19)	0.0184 (4)
C3	0.61816 (11)	−0.2052 (4)	0.7334 (2)	0.0206 (5)
H3	0.6412	−0.2185	0.6648	0.025*
C4	0.63178 (12)	−0.3592 (5)	0.8308 (2)	0.0215 (5)
H4	0.6661	−0.4953	0.8401	0.026*
C5	0.58673 (12)	−0.2760 (5)	0.9079 (2)	0.0216 (5)
H5	0.5843	−0.3466	0.9810	0.026*
C6	0.40692 (11)	0.6696 (4)	0.75078 (19)	0.0187 (5)
H6	0.4172	0.6780	0.8274	0.022*
C7	0.35484 (12)	0.8472 (4)	0.70708 (18)	0.0199 (5)
C8	0.32160 (11)	1.0548 (5)	0.7638 (2)	0.0213 (5)
H8	0.3317	1.1048	0.8379	0.026*
C9	0.27098 (12)	1.1854 (5)	0.7001 (2)	0.0229 (5)
H9	0.2434	1.3318	0.7275	0.027*
C10	0.26523 (11)	1.0817 (5)	0.5956 (2)	0.0230 (5)
C11	0.21736 (13)	1.1647 (5)	0.5057 (2)	0.0303 (6)
H11A	0.1905	1.3246	0.5301	0.045*

H11B	0.1872	1.0087	0.4888	0.045*
H11C	0.2430	1.2151	0.4394	0.045*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0265 (3)	0.0216 (3)	0.0213 (3)	0.0020 (2)	−0.0030 (2)	0.0004 (3)
O1	0.0278 (8)	0.0208 (8)	0.0151 (8)	0.0021 (6)	0.0012 (7)	0.0005 (6)
O2	0.0214 (7)	0.0212 (8)	0.0169 (7)	0.0032 (6)	0.0004 (6)	0.0022 (7)
O3	0.0340 (10)	0.0203 (9)	0.0176 (9)	0.0050 (7)	−0.0002 (7)	−0.0011 (7)
N1	0.0191 (10)	0.0198 (9)	0.0151 (9)	0.0019 (7)	−0.0015 (7)	0.0015 (8)
N2	0.0181 (9)	0.0172 (8)	0.0218 (9)	0.0017 (7)	−0.0032 (7)	0.0035 (8)
C1	0.0202 (11)	0.0169 (10)	0.0176 (11)	−0.0012 (8)	−0.0001 (9)	−0.0029 (8)
C2	0.0208 (11)	0.0208 (10)	0.0136 (9)	−0.0022 (8)	−0.0003 (8)	0.0014 (9)
C3	0.0192 (11)	0.0237 (12)	0.0190 (11)	−0.0021 (8)	0.0027 (9)	−0.0040 (9)
C4	0.0183 (11)	0.0218 (11)	0.0244 (12)	0.0024 (9)	−0.0014 (9)	0.0010 (9)
C5	0.0248 (11)	0.0197 (11)	0.0201 (11)	0.0040 (9)	−0.0072 (9)	0.0018 (9)
C6	0.0187 (11)	0.0185 (11)	0.0188 (11)	−0.0019 (8)	−0.0002 (9)	0.0023 (9)
C7	0.0205 (12)	0.0184 (10)	0.0208 (12)	−0.0021 (8)	0.0012 (9)	0.0029 (9)
C8	0.0228 (11)	0.0217 (11)	0.0195 (10)	−0.0004 (9)	0.0028 (9)	0.0010 (10)
C9	0.0206 (11)	0.0191 (11)	0.0289 (13)	0.0035 (8)	0.0060 (9)	0.0041 (10)
C10	0.0179 (10)	0.0213 (11)	0.0297 (13)	−0.0005 (8)	0.0002 (10)	0.0065 (10)
C11	0.0247 (13)	0.0321 (14)	0.0340 (14)	0.0020 (10)	−0.0051 (11)	0.0099 (12)

Geometric parameters (Å, °)

S1—C7	1.728 (2)	C4—C5	1.351 (3)
S1—C10	1.732 (2)	C4—H4	0.9500
O1—C1	1.241 (3)	C5—H5	0.9500
O2—C5	1.367 (3)	C6—C7	1.439 (3)
O2—C2	1.377 (3)	C6—H6	0.9500
O3—H3A	0.87 (3)	C7—C8	1.379 (3)
O3—H3B	0.76 (3)	C8—C9	1.411 (3)
N1—C1	1.348 (3)	C8—H8	0.9500
N1—N2	1.393 (2)	C9—C10	1.365 (3)
N1—H1	0.88 (3)	C9—H9	0.9500
N2—C6	1.285 (3)	C10—C11	1.497 (3)
C1—C2	1.466 (3)	C11—H11A	0.9800
C2—C3	1.360 (3)	C11—H11B	0.9800
C3—C4	1.418 (3)	C11—H11C	0.9800
C3—H3	0.9500		
C7—S1—C10	92.39 (11)	N2—C6—C7	121.8 (2)
C5—O2—C2	106.28 (17)	N2—C6—H6	119.1
H3A—O3—H3B	97 (3)	C7—C6—H6	119.1
C1—N1—N2	119.04 (19)	C8—C7—C6	126.0 (2)
C1—N1—H1	120.3 (17)	C8—C7—S1	110.50 (18)
N2—N1—H1	119.9 (17)	C6—C7—S1	123.43 (17)
C6—N2—N1	113.50 (19)	C7—C8—C9	112.9 (2)

O1—C1—N1	124.2 (2)	C7—C8—H8	123.6
O1—C1—C2	120.4 (2)	C9—C8—H8	123.6
N1—C1—C2	115.4 (2)	C10—C9—C8	113.8 (2)
C3—C2—O2	110.09 (19)	C10—C9—H9	123.1
C3—C2—C1	132.0 (2)	C8—C9—H9	123.1
O2—C2—C1	117.86 (19)	C9—C10—C11	129.0 (2)
C2—C3—C4	106.2 (2)	C9—C10—S1	110.45 (17)
C2—C3—H3	126.9	C11—C10—S1	120.53 (19)
C4—C3—H3	126.9	C10—C11—H11A	109.5
C5—C4—C3	107.1 (2)	C10—C11—H11B	109.5
C5—C4—H4	126.5	H11A—C11—H11B	109.5
C3—C4—H4	126.5	C10—C11—H11C	109.5
C4—C5—O2	110.3 (2)	H11A—C11—H11C	109.5
C4—C5—H5	124.9	H11B—C11—H11C	109.5
O2—C5—H5	124.9		
C1—N1—N2—C6	−170.2 (2)	C2—O2—C5—C4	0.1 (2)
N2—N1—C1—O1	8.1 (3)	N1—N2—C6—C7	−173.95 (18)
N2—N1—C1—C2	−172.18 (17)	N2—C6—C7—C8	−172.9 (2)
C5—O2—C2—C3	0.2 (2)	N2—C6—C7—S1	9.5 (3)
C5—O2—C2—C1	178.82 (19)	C10—S1—C7—C8	−0.37 (17)
O1—C1—C2—C3	7.5 (4)	C10—S1—C7—C6	177.55 (19)
N1—C1—C2—C3	−172.2 (2)	C6—C7—C8—C9	−177.4 (2)
O1—C1—C2—O2	−170.7 (2)	S1—C7—C8—C9	0.4 (2)
N1—C1—C2—O2	9.6 (3)	C7—C8—C9—C10	−0.3 (3)
O2—C2—C3—C4	−0.4 (2)	C8—C9—C10—C11	179.3 (2)
C1—C2—C3—C4	−178.8 (2)	C8—C9—C10—S1	0.0 (3)
C2—C3—C4—C5	0.5 (3)	C7—S1—C10—C9	0.21 (18)
C3—C4—C5—O2	−0.3 (3)	C7—S1—C10—C11	−179.17 (19)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O3	0.88 (3)	1.98 (3)	2.829 (3)	163 (2)
O3—H3B···O1^{i}	0.76 (3)	2.09 (3)	2.843 (2)	174 (3)
O3—H3A···O1^{ii}	0.87 (3)	2.01 (3)	2.835 (3)	158 (3)

Symmetry codes: (i) −*x*+1, −*y*, *z*+1/2; (ii) −*x*+1, −*y*+1, *z*+1/2.

Fig. 1

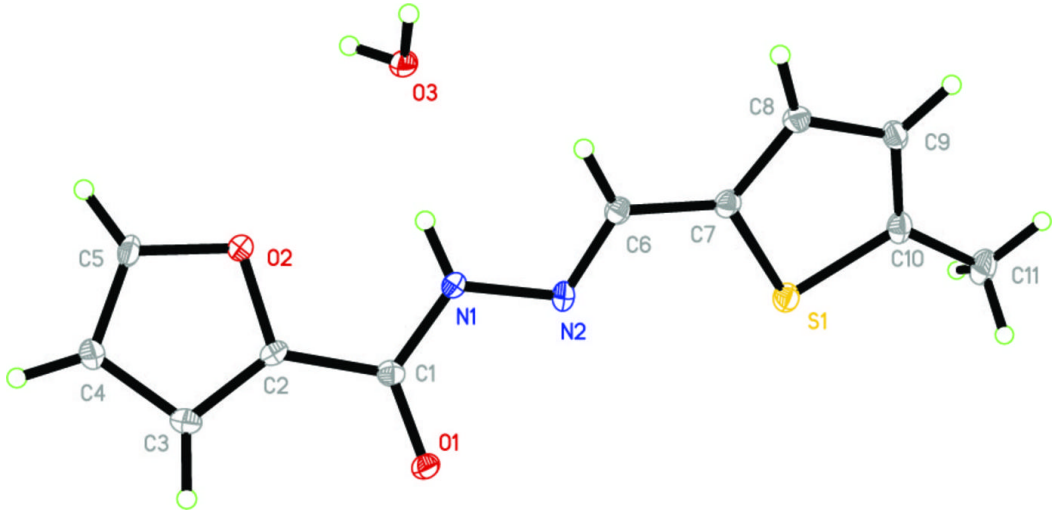

Fig. 2