Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*E*)-*N*′-[(5-Methylthiophen-2-yl)-methylene]isonicotinohydrazide

Chang-Lu Wang,[a] Zhen-Heng Zhang[a] and Zuo-Liang Jing[b]*

[a]College of Food Engineering and Biotechnology, Tianjin University of Science and Technology, Tianjin 300457, People's Republic of China, and [b]College of Sciences, Tianjin University of Science and Technology, Tianjin 300457, People's Republic of China
Correspondence e-mail: jzl74@tust.edu.cn

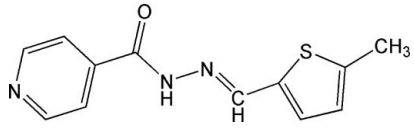
Received 12 November 2007; accepted 15 November 2007

Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.004 Å; R factor = 0.043; wR factor = 0.115; data-to-parameter ratio = 13.0.

In the title compound, $C_{12}H_{11}N_3OS$, the dihedral angle between the thiophene and pyridine planes is 12.30 (2)°. The molecules are linked *via* weak intermolecular N—H···O hydrogen bonding, forming a chain supramolecular arrangement along the *c* axis.

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

$C_{12}H_{11}N_3OS$
M_r = 245.30
Monoclinic, $P2_1/c$
a = 19.172 (7) Å
b = 5.884 (2) Å
c = 10.273 (4) Å
β = 97.309 (7)°
V = 1149.4 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.27 mm^{-1}
T = 294 (2) K
0.24 × 0.22 × 0.16 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.939, T_{max} = 0.959
5666 measured reflections
2018 independent reflections
1361 reflections with $I > 2\sigma(I)$
R_{int} = 0.053

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.115
S = 1.04
2018 reflections
155 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O1^{i}	0.86	2.20	3.031 (3)	163

Symmetry code: (i) $x, -y+\frac{3}{2}, z-\frac{1}{2}$.

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: AT2489).

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 doi:10.1107/S1600536807059363

supplementary materials

Acta Cryst. (2007). E**63**, o4825 [**doi:10.1107/S1600536807059363**]

(*E*)-*N*'-[(5-Methylthiophen-2-yl)methylene]isonicotinohydrazide

C.-L. Wang, Z.-H. Zhang and Z.-L. Jing

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 (C2–C5/S1) is approximately planar, with a maximum deviation from the mean plane of 0.0048 (2) Å for atom S1,as are the pyridine group (C8—C11/N3) is approximately planar, with a maximum deviation from the mean plane of 0.0068 (2) Å for atom N3.The dihedral angle between these two planes is 12.30 (2)°·The molecules are linked *via* weak intermolecular N—H···O hydrogen bond(Table 1), Forming a chain supramolecular arrangement along the *c* axis.,as illustraed 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 isonicotinohydrazide (1.37 g, 10 mmol), and the mixture was stirred at 350 K for 6 h under N_2, whereupon a yelllow precipitate appeared. The product was isolated, recrystallized from anhydrous ethanol and then dried *in vacuo* to give pure compound (I) in 81% yield. Yellow single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an anhydrous ethanol solution.

Refinement

All H atoms were included in calculated positions, with N—H = 0.86 (amine), C—H = 0.93 (aromatic) or 0.96 Å (methyl), and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C,*N*) for aromatic and amine H atoms and 1.5U_{eq}(C) for methyl H atoms.

Figures

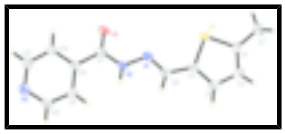

Fig. 1. The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level.

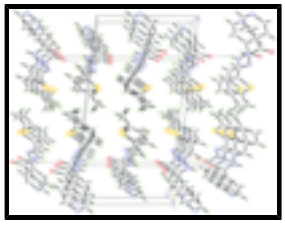

Fig. 2. The crystal packing of (I), viwed down the *a* axis. Hydrogen bonds are indicated by dashed lines.

(*E*)-*N*'-[(5-Methylthiophen-2-yl)methylene]isonicotinohydrazide

Crystal data

$C_{12}H_{11}N_3OS$	F_{000} = 512
M_r = 245.30	D_x = 1.418 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 1387 reflections
a = 19.172 (7) Å	θ = 3.6–24.6º
b = 5.884 (2) Å	μ = 0.27 mm^{-1}
c = 10.273 (4) Å	T = 294 (2) K
β = 97.309 (7)º	Block, yellow
V = 1149.4 (8) Å^3	0.24 × 0.22 × 0.16 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	2018 independent reflections
Radiation source: fine-focus sealed tube	1361 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.053
T = 294(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 1.1º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −16→22
T_{min} = 0.939, T_{max} = 0.959	k = −7→6
5666 measured reflections	l = −11→12

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.043	H-atom parameters constrained
$wR(F^2)$ = 0.115	$w = 1/[\sigma^2(F_o^2) + (0.0602P)^2 + 0.0201P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.04	$(\Delta/\sigma)_{max}$ = 0.004
2018 reflections	$\Delta\rho_{max}$ = 0.23 e Å^{-3}
155 parameters	$\Delta\rho_{min}$ = −0.26 e Å^{-3}

Primary atom site location: structure-invariant direct methods

Extinction correction: none

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 $F^2 > 2\mathrm{sigma}(F^2)$ 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	0.38071 (4)	1.38538 (11)	0.10139 (6)	0.0449 (2)
O1	0.20976 (10)	0.7241 (3)	0.18993 (17)	0.0563 (5)
N1	0.26629 (10)	1.0178 (3)	0.02564 (19)	0.0406 (5)
N2	0.21957 (11)	0.8469 (3)	−0.01604 (19)	0.0426 (6)
H2	0.2071	0.8268	−0.0987	0.051*
N3	0.03831 (13)	0.2277 (4)	−0.0882 (2)	0.0545 (6)
C1	0.47021 (16)	1.7636 (5)	0.1251 (3)	0.0602 (8)
H1A	0.5095	1.7888	0.0776	0.090*
H1B	0.4856	1.6820	0.2045	0.090*
H1C	0.4507	1.9072	0.1464	0.090*
C2	0.41553 (13)	1.6283 (4)	0.0427 (3)	0.0414 (6)
C3	0.38671 (15)	1.6675 (5)	−0.0821 (3)	0.0498 (7)
H3	0.3982	1.7932	−0.1299	0.060*
C4	0.33807 (15)	1.5035 (5)	−0.1333 (3)	0.0493 (7)
H4	0.3147	1.5076	−0.2183	0.059*
C5	0.32862 (13)	1.3368 (4)	−0.0450 (2)	0.0395 (6)
C6	0.28111 (13)	1.1477 (4)	−0.0664 (3)	0.0430 (7)
H6	0.2601	1.1187	−0.1514	0.052*
C7	0.19310 (13)	0.7107 (4)	0.0714 (2)	0.0401 (6)
C8	0.13952 (13)	0.5436 (4)	0.0116 (2)	0.0369 (6)
C9	0.09620 (14)	0.5838 (5)	−0.1038 (2)	0.0457 (7)
H9	0.1004	0.7173	−0.1507	0.055*
C10	0.04682 (15)	0.4237 (5)	−0.1483 (3)	0.0553 (8)
H10	0.0175	0.4544	−0.2255	0.066*
C11	0.08050 (15)	0.1924 (5)	0.0227 (3)	0.0531 (8)
H11	0.0759	0.0560	0.0666	0.064*
C12	0.13018 (14)	0.3430 (5)	0.0768 (3)	0.0478 (7)
H12	0.1573	0.3108	0.1563	0.057*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0546 (5)	0.0465 (4)	0.0323 (4)	−0.0060 (3)	0.0004 (3)	0.0045 (3)
O1	0.0644 (13)	0.0732 (13)	0.0284 (11)	−0.0133 (11)	−0.0055 (9)	−0.0013 (9)
N1	0.0410 (13)	0.0488 (13)	0.0311 (12)	−0.0031 (11)	0.0010 (10)	−0.0061 (10)
N2	0.0462 (14)	0.0548 (14)	0.0254 (11)	−0.0072 (11)	−0.0014 (9)	−0.0073 (10)
N3	0.0580 (16)	0.0593 (16)	0.0455 (15)	−0.0120 (13)	0.0040 (12)	−0.0078 (12)
C1	0.071 (2)	0.0535 (18)	0.059 (2)	−0.0150 (16)	0.0179 (17)	−0.0079 (15)
C2	0.0477 (16)	0.0377 (14)	0.0411 (16)	0.0024 (13)	0.0146 (13)	−0.0001 (12)
C3	0.064 (2)	0.0433 (16)	0.0447 (17)	0.0023 (15)	0.0180 (14)	0.0085 (13)
C4	0.0543 (18)	0.0615 (18)	0.0316 (15)	0.0122 (15)	0.0041 (13)	0.0087 (14)
C5	0.0394 (15)	0.0480 (16)	0.0302 (14)	0.0054 (13)	0.0013 (11)	−0.0005 (12)
C6	0.0415 (16)	0.0551 (17)	0.0311 (15)	0.0039 (13)	−0.0002 (12)	−0.0060 (13)
C7	0.0398 (16)	0.0491 (15)	0.0304 (15)	0.0055 (13)	0.0008 (12)	−0.0043 (12)
C8	0.0371 (15)	0.0435 (15)	0.0304 (14)	0.0016 (12)	0.0048 (11)	−0.0029 (11)
C9	0.0511 (17)	0.0485 (17)	0.0358 (16)	−0.0054 (13)	−0.0020 (13)	0.0021 (12)
C10	0.0577 (19)	0.067 (2)	0.0387 (17)	−0.0083 (16)	−0.0043 (14)	−0.0012 (14)
C11	0.060 (2)	0.0494 (17)	0.0508 (19)	−0.0045 (15)	0.0089 (15)	0.0009 (14)
C12	0.0501 (18)	0.0549 (18)	0.0369 (16)	0.0038 (14)	0.0001 (13)	0.0015 (13)

Geometric parameters (Å, °)

S1—C2	1.719 (2)	C3—H3	0.9300
S1—C5	1.720 (3)	C4—C5	1.363 (3)
O1—C7	1.222 (3)	C4—H4	0.9300
N1—C6	1.275 (3)	C5—C6	1.437 (3)
N1—N2	1.378 (3)	C6—H6	0.9300
N2—C7	1.350 (3)	C7—C8	1.496 (3)
N2—H2	0.8600	C8—C9	1.379 (3)
N3—C11	1.327 (4)	C8—C12	1.380 (3)
N3—C10	1.328 (4)	C9—C10	1.372 (4)
C1—C2	1.492 (4)	C9—H9	0.9300
C1—H1A	0.9600	C10—H10	0.9300
C1—H1B	0.9600	C11—C12	1.366 (4)
C1—H1C	0.9600	C11—H11	0.9300
C2—C3	1.349 (4)	C12—H12	0.9300
C3—C4	1.398 (4)		
C2—S1—C5	92.26 (13)	C6—C5—S1	123.27 (19)
C6—N1—N2	113.8 (2)	N1—C6—C5	123.2 (2)
C7—N2—N1	120.7 (2)	N1—C6—H6	118.4
C7—N2—H2	119.6	C5—C6—H6	118.4
N1—N2—H2	119.6	O1—C7—N2	123.5 (2)
C11—N3—C10	116.0 (3)	O1—C7—C8	122.0 (2)
C2—C1—H1A	109.5	N2—C7—C8	114.4 (2)
C2—C1—H1B	109.5	C9—C8—C12	117.6 (2)
H1A—C1—H1B	109.5	C9—C8—C7	123.2 (2)

C2—C1—H1C	109.5	C12—C8—C7	119.2 (2)
H1A—C1—H1C	109.5	C10—C9—C8	118.9 (3)
H1B—C1—H1C	109.5	C10—C9—H9	120.5
C3—C2—C1	128.4 (2)	C8—C9—H9	120.5
C3—C2—S1	110.2 (2)	N3—C10—C9	124.1 (3)
C1—C2—S1	121.4 (2)	N3—C10—H10	117.9
C2—C3—C4	114.3 (2)	C9—C10—H10	117.9
C2—C3—H3	122.8	N3—C11—C12	124.4 (3)
C4—C3—H3	122.8	N3—C11—H11	117.8
C5—C4—C3	112.6 (3)	C12—C11—H11	117.8
C5—C4—H4	123.7	C11—C12—C8	119.0 (3)
C3—C4—H4	123.7	C11—C12—H12	120.5
C4—C5—C6	126.2 (2)	C8—C12—H12	120.5
C4—C5—S1	110.5 (2)		
C6—N1—N2—C7	−173.0 (2)	N1—N2—C7—C8	176.13 (19)
C5—S1—C2—C3	1.1 (2)	O1—C7—C8—C9	149.0 (3)
C5—S1—C2—C1	−178.4 (2)	N2—C7—C8—C9	−29.4 (3)
C1—C2—C3—C4	178.0 (2)	O1—C7—C8—C12	−28.0 (4)
S1—C2—C3—C4	−1.4 (3)	N2—C7—C8—C12	153.6 (2)
C2—C3—C4—C5	1.0 (3)	C12—C8—C9—C10	−0.5 (4)
C3—C4—C5—C6	178.4 (2)	C7—C8—C9—C10	−177.5 (2)
C3—C4—C5—S1	−0.2 (3)	C11—N3—C10—C9	1.1 (4)
C2—S1—C5—C4	−0.51 (19)	C8—C9—C10—N3	−1.1 (4)
C2—S1—C5—C6	−179.1 (2)	C10—N3—C11—C12	0.5 (4)
N2—N1—C6—C5	180.0 (2)	N3—C11—C12—C8	−2.0 (4)
C4—C5—C6—N1	−168.5 (2)	C9—C8—C12—C11	1.9 (4)
S1—C5—C6—N1	9.9 (4)	C7—C8—C12—C11	179.1 (2)
N1—N2—C7—O1	−2.2 (4)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O1^{i}	0.86	2.20	3.031 (3)	163

Symmetry codes: (i) *x*, −*y*+3/2, *z*−1/2.

Fig. 1

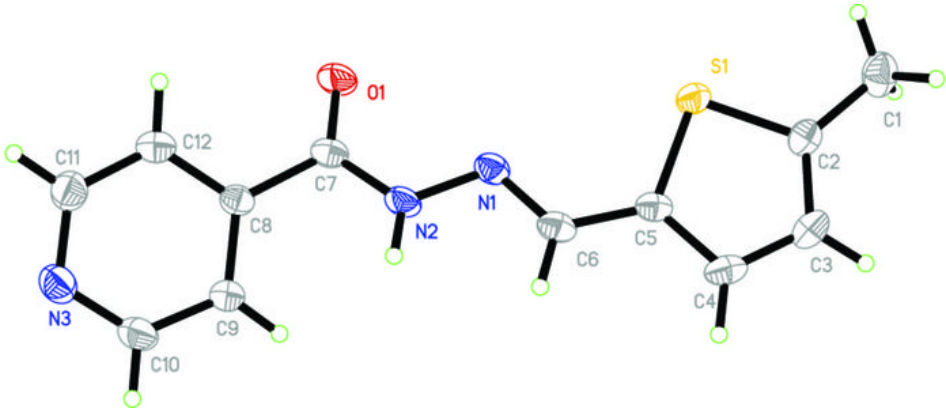

Fig. 2