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N′-[(Thiophen-3-yl)methylene]-isonicotinohydrazide

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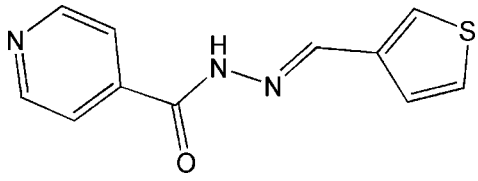
Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.002 Å; R factor = 0.039; wR factor = 0.098; data-to-parameter ratio = 16.4.

In the title compound, $C_{11}H_9N_3OS$, the dihedral angle between the thiophene and pyridine planes is 24.06 (9)°. Intermolecular N—H···O, C—H···S and C—H···N hydrogen bonds link the molecules into a two-dimensional network parallel to the (100) 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

$C_{11}H_9N_3OS$
M_r = 231.27
Monoclinic, $P2_1/c$
a = 19.861 (4) Å
b = 5.1856 (10) Å
c = 10.103 (2) Å
β = 99.55 (3)°
V = 1026.1 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.29 mm^{-1}
T = 113 (2) K
0.10 × 0.08 × 0.04 mm

Data collection

Rigaku Saturn diffractometer
Absorption correction: multi-scan *CrystalClear* (Rigaku/MSC, 2005)
T_{min} = 0.964, T_{max} = 0.988
11875 measured reflections
2440 independent reflections
2195 reflections with $I > 2\sigma(I)$
R_{int} = 0.045

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.098
S = 1.05
2440 reflections
149 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O1^{i}	0.88 (2)	2.10 (2)	2.9634 (17)	167 (2)
C1—H1···S1^{ii}	0.95	2.82	3.4374 (16)	123
C4—H4···N1^{iii}	0.95	2.59	3.535 (2)	171
C10—H10···N3^{iv}	0.95	2.49	3.409 (2)	162

Symmetry codes: (i) $x, -y+\frac{1}{2}, z+\frac{1}{2}$; (ii) $x, -y+\frac{5}{2}, z-\frac{1}{2}$; (iii) $x, -y+\frac{3}{2}, z+\frac{1}{2}$; (iv) $-x, y+\frac{1}{2}, -z+\frac{3}{2}$.

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: CI2456).

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supplementary materials

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N'-[(Thiophen-3-yl)methylene]isonicotinohydrazide

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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 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. The thiophene ring (C1—C4/S1) is essentially planar, with a maximum deviation from the mean plane of 0.005 (1) Å for atom C4. The pyridine ring (C7—C11/N3) is planar within ±0.009 (1) Å. The dihedral angle between the thiophene and pyridine planes is 24.06 (9)°. The O1/N1/N2/C5/C6 plane makes dihedral angles of 28.87 (9) and 4.89 (12)° with the pyridine and thiophene rings, respectively.

Intermolecular N—H···O hydrogen bonds (Table 1) link the molecules into a chain along the *c* axis (Fig. 2). The chains are cross-linked by C—H···S and C—H···N hydrogen bonds, forming a two-dimensional network parallel to the (1 0 0) plane.

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 isonicotinohydrazide (1.37 g, 10 mmol) and the mixture was stirred at 350 K for 6 h under N_2, whereupon a yellow precipitate appeared. The product was isolated, recrystallized from anhydrous ethanol and then dried *in vacuo* to give pure compound (I) in 91% yield. Yellow 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 its positional parameters were refined, with U_{iso}(H) = 1.2U_{eq}(N). C-bound H atoms were included in calculated positions [C—H = 0.95 Å] and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C).

Figures

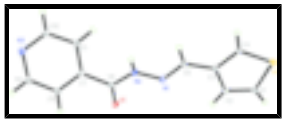

Fig. 1. The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level.

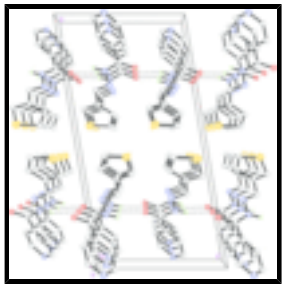

Fig. 2. The crystal packing of (I), viwed down the *b* axis, showing N—H···O hydrogen bonded (dashed lines) chains.

N'-[(Thiophen-3-yl)methylene]isonicotinohydrazide

Crystal data

$C_{11}H_9N_3OS$	F_{000} = 480
M_r = 231.27	D_x = 1.497 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 6540 reflections
a = 19.861 (4) Å	θ = 2.3–22.5º
b = 5.1856 (10) Å	μ = 0.29 mm^{-1}
c = 10.103 (2) Å	T = 113 (2) K
β = 99.55 (3)º	Block, yellow
V = 1026.1 (4) Å^3	0.10 × 0.08 × 0.04 mm
Z = 4	

Data collection

Rigaku Saturn diffractometer	2440 independent reflections
Radiation source: rotating anode	2195 reflections with $I > 2\sigma(I)$
Monochromator: confocal	R_{int} = 0.045
T = 113(2) K	θ_{max} = 27.9º
ω and φ scans	θ_{min} = 2.1º
Absorption correction: multi-scan CrystalClear (Rigaku/MSC, 2005)	h = −26→26
T_{min} = 0.964, T_{max} = 0.988	k = −6→6
11875 measured reflections	l = −13→13

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.039	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.098	$w = 1/[\sigma^2(F_o^2) + (0.0424P)^2 + 0.7091P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.05	$(\Delta/\sigma)_{max}$ = 0.001

2440 reflections	$\Delta\rho_{max} = 0.34$ e Å^{-3}
149 parameters	$\Delta\rho_{min} = -0.40$ 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 $F^2 > 2\text{sigma}(F^2)$ 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	0.43768 (2)	1.25594 (7)	0.87336 (4)	0.01810 (13)
O1	0.22717 (6)	0.2606 (2)	0.45121 (11)	0.0203 (3)
N1	0.28541 (6)	0.5670 (2)	0.66051 (12)	0.0158 (3)
N2	0.23603 (6)	0.3826 (3)	0.67066 (13)	0.0158 (3)
N3	0.03908 (7)	−0.2485 (2)	0.60415 (13)	0.0183 (3)
C1	0.44033 (8)	1.1592 (3)	0.71162 (14)	0.0162 (3)
H1	0.4699	1.2304	0.6563	0.019*
C2	0.39400 (7)	0.9650 (3)	0.67280 (15)	0.0170 (3)
H2	0.3879	0.8854	0.5869	0.020*
C3	0.35604 (7)	0.8963 (3)	0.77658 (14)	0.0150 (3)
C4	0.37454 (7)	1.0421 (3)	0.89057 (15)	0.0165 (3)
H4	0.3546	1.0255	0.9693	0.020*
C5	0.30414 (8)	0.6968 (3)	0.76887 (15)	0.0165 (3)
H5	0.2836	0.6615	0.8454	0.020*
C6	0.20863 (7)	0.2435 (3)	0.56109 (15)	0.0145 (3)
C7	0.15088 (7)	0.0699 (3)	0.58219 (14)	0.0142 (3)
C8	0.13685 (8)	−0.1481 (3)	0.50177 (15)	0.0165 (3)
H8	0.1648	−0.1912	0.4372	0.020*
C9	0.08158 (8)	−0.3008 (3)	0.51736 (16)	0.0187 (3)
H9	0.0732	−0.4513	0.4635	0.022*
C10	0.05238 (8)	−0.0364 (3)	0.67959 (15)	0.0180 (3)
H10	0.0224	0.0056	0.7405	0.022*
C11	0.10758 (8)	0.1260 (3)	0.67384 (14)	0.0163 (3)
H11	0.1156	0.2719	0.7311	0.020*
H2A	0.2262 (11)	0.345 (4)	0.750 (2)	0.031 (5)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0206 (2)	0.0185 (2)	0.0154 (2)	−0.00303 (14)	0.00348 (15)	−0.00190 (13)
O1	0.0216 (6)	0.0261 (6)	0.0145 (5)	−0.0057 (5)	0.0065 (4)	−0.0030 (4)
N1	0.0140 (6)	0.0170 (6)	0.0162 (6)	−0.0020 (5)	0.0018 (4)	0.0011 (5)
N2	0.0159 (6)	0.0197 (7)	0.0123 (6)	−0.0042 (5)	0.0034 (5)	0.0001 (5)
N3	0.0191 (6)	0.0189 (7)	0.0165 (6)	−0.0031 (5)	0.0018 (5)	0.0021 (5)
C1	0.0177 (7)	0.0196 (8)	0.0116 (7)	0.0012 (6)	0.0035 (5)	−0.0001 (6)
C2	0.0181 (7)	0.0197 (8)	0.0134 (7)	−0.0006 (6)	0.0029 (5)	−0.0014 (6)
C3	0.0136 (6)	0.0169 (7)	0.0142 (7)	0.0006 (5)	0.0014 (5)	0.0011 (5)
C4	0.0171 (7)	0.0187 (7)	0.0144 (7)	0.0001 (6)	0.0048 (5)	0.0006 (6)
C5	0.0157 (7)	0.0196 (7)	0.0147 (7)	0.0000 (6)	0.0039 (5)	0.0009 (6)
C6	0.0139 (7)	0.0151 (7)	0.0144 (7)	0.0013 (5)	0.0022 (5)	0.0000 (5)
C7	0.0141 (6)	0.0157 (7)	0.0122 (6)	0.0007 (5)	0.0009 (5)	0.0018 (5)
C8	0.0185 (7)	0.0170 (7)	0.0145 (7)	0.0008 (6)	0.0043 (5)	0.0001 (6)
C9	0.0222 (8)	0.0176 (7)	0.0157 (7)	−0.0014 (6)	0.0013 (6)	−0.0009 (6)
C10	0.0166 (7)	0.0222 (8)	0.0156 (7)	0.0008 (6)	0.0038 (5)	0.0014 (6)
C11	0.0191 (7)	0.0164 (7)	0.0134 (7)	−0.0001 (6)	0.0029 (5)	−0.0011 (6)

Geometric parameters (Å, °)

S1—C4	1.7044 (16)	C3—C4	1.375 (2)
S1—C1	1.7185 (15)	C3—C5	1.453 (2)
O1—C6	1.2295 (19)	C4—H4	0.95
N1—C5	1.287 (2)	C5—H5	0.95
N1—N2	1.3856 (18)	C6—C7	1.501 (2)
N2—C6	1.3572 (19)	C7—C8	1.393 (2)
N2—H2A	0.88 (2)	C7—C11	1.395 (2)
N3—C10	1.339 (2)	C8—C9	1.384 (2)
N3—C9	1.342 (2)	C8—H8	0.95
C1—C2	1.377 (2)	C9—H9	0.95
C1—H1	0.95	C10—C11	1.391 (2)
C2—C3	1.434 (2)	C10—H10	0.95
C2—H2	0.95	C11—H11	0.95
C4—S1—C1	92.64 (8)	C3—C5—H5	119.3
C5—N1—N2	113.98 (13)	O1—C6—N2	123.95 (14)
C6—N2—N1	119.96 (13)	O1—C6—C7	121.46 (13)
C6—N2—H2A	120.4 (14)	N2—C6—C7	114.55 (13)
N1—N2—H2A	119.3 (14)	C8—C7—C11	118.07 (14)
C10—N3—C9	116.86 (13)	C8—C7—C6	119.18 (13)
C2—C1—S1	111.10 (11)	C11—C7—C6	122.62 (13)
C2—C1—H1	124.4	C9—C8—C7	118.93 (14)
S1—C1—H1	124.4	C9—C8—H8	120.5
C1—C2—C3	112.38 (13)	C7—C8—H8	120.5
C1—C2—H2	123.8	N3—C9—C8	123.76 (15)
C3—C2—H2	123.8	N3—C9—H9	118.1

C4—C3—C2	112.09 (13)	C8—C9—H9	118.1
C4—C3—C5	121.64 (14)	N3—C10—C11	123.76 (14)
C2—C3—C5	126.27 (14)	N3—C10—H10	118.1
C3—C4—S1	111.78 (11)	C11—C10—H10	118.1
C3—C4—H4	124.1	C10—C11—C7	118.59 (14)
S1—C4—H4	124.1	C10—C11—H11	120.7
N1—C5—C3	121.37 (14)	C7—C11—H11	120.7
N1—C5—H5	119.3		
C5—N1—N2—C6	175.80 (13)	O1—C6—C7—C8	27.3 (2)
C4—S1—C1—C2	−0.55 (12)	N2—C6—C7—C8	−154.84 (13)
S1—C1—C2—C3	0.19 (17)	O1—C6—C7—C11	−148.57 (15)
C1—C2—C3—C4	0.39 (19)	N2—C6—C7—C11	29.3 (2)
C1—C2—C3—C5	−178.99 (14)	C11—C7—C8—C9	−0.9 (2)
C2—C3—C4—S1	−0.80 (17)	C6—C7—C8—C9	−176.99 (13)
C5—C3—C4—S1	178.62 (11)	C10—N3—C9—C8	−0.6 (2)
C1—S1—C4—C3	0.78 (12)	C7—C8—C9—N3	1.6 (2)
N2—N1—C5—C3	179.34 (13)	C9—N3—C10—C11	−1.1 (2)
C4—C3—C5—N1	177.64 (14)	N3—C10—C11—C7	1.7 (2)
C2—C3—C5—N1	−3.0 (2)	C8—C7—C11—C10	−0.6 (2)
N1—N2—C6—O1	3.7 (2)	C6—C7—C11—C10	175.34 (13)
N1—N2—C6—C7	−174.11 (12)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2A···O1^{i}	0.88 (2)	2.10 (2)	2.9634 (17)	167 (2)
C1—H1···S1^{ii}	0.95	2.82	3.4374 (16)	123
C4—H4···N1^{iii}	0.95	2.59	3.535 (2)	171
C10—H10···N3^{iv}	0.95	2.49	3.409 (2)	162

Symmetry codes: (i) *x*, −*y*+1/2, *z*+1/2; (ii) *x*, −*y*+5/2, *z*−1/2; (iii) *x*, −*y*+3/2, *z*+1/2; (iv) −*x*, *y*+1/2, −*z*+3/2.

Fig. 1

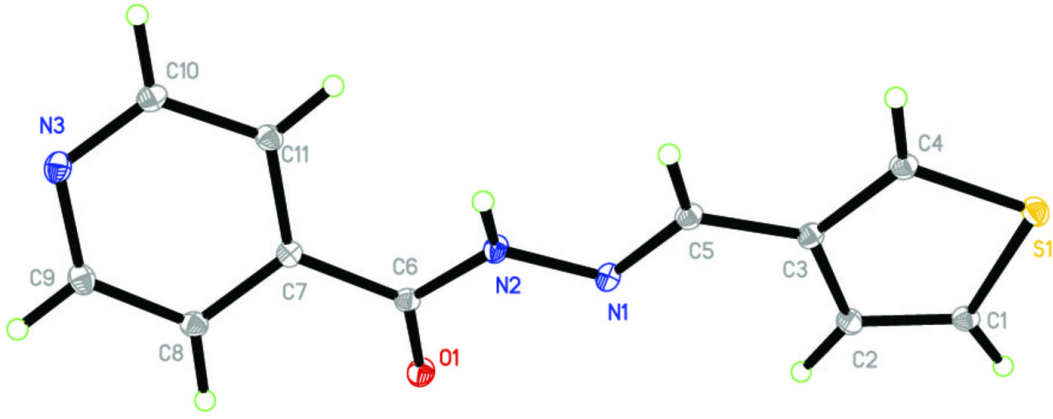

Fig. 2