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N-[(Pyridin-2-yl)methyl]thiophene-2-carboxamide

M. Mohanbabu,[a] P. N. Sathishkumar,[b] N. S. P. Bhuvanesh,[c] R. Karvembu[b] and S. Aravindhan[d]*

[a]Department of Physics, Sri Malolan College of Arts & Science, Madhurantakam, Kanchipuram - 603 306, India, [b]Department of Chemistry, National Institute of Technology, Tiruchirappalli - 620 015, India, [c]Department of Chemistry, Texas A & M University, College Station, TX, 77842, USA, and [d]Department of Physics, Presidency College (Autonomous), Chennai - 600 005, India. *Correspondence e-mail: saravindhanpresidency@gmail.com

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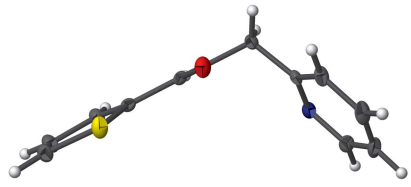
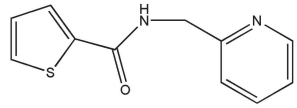
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{11}H_{10}N_2OS$, the dihedral angle between the thiophene and pyridine rings is 77.79 (8)°. In the crystal, inversion dimers linked by pairs of N—H···N hydrogen bonds generate $R_2^2(10)$ loops. The dimers are reinforced by pairs of C—H···N interactions and C—H···O interactions link the dimers into [010] chains.

3D view



Chemical scheme



Structure description

Thiophene and its derivatives have various biological properties including anti-microbial (Russell *et al.*, 1988), analgesic and anti-inflammatory (Chen *et al.*, 2008), anti-hypertensive (Monge Vega *et al.*, 1980), anti-diabetes mellitus (Abdelhamid *et al.*, 2009) and gonadotropin releasing hormone antagonist (Sabins *et al.*, 1944) activities. As part of our studies of potential active pharmaceutical ingredients (APIs) based on thiophenes, we report here the synthesis and crystal structure of the title compound (Fig. 1).

The key torsion angle of the molecule, S1—C8—C7—O1 and C9—C8—C7—N2 with (−)*syn*-periplanar conformations and N1—C1—C6—N2 and C1—C6—N2—C7 with (+)*syn*-clinal conformations are −5.13 (19), −6.4 (2), 79.64 (16) and 73.47 (17)°, respectively. The dihedral angle between the thiophene ring and the pyridine ring is 77.79 (8)°.

In the crystal, the molecules are linked *via* pairs of N2—H2···N1 hydrogen bonds, forming inversion dimers with an $R_2^2(10)$ ring motif; the dimers are reinforced by a pair of C9—H9···N1 interactions. The dimers are linked into [010] chains by C5—H5···O1 interactions (Table 1 and Figs. 2 and 3).

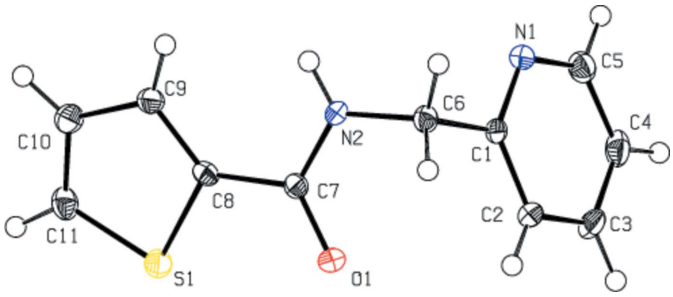


Figure 1
The molecular structure of the title compound, with displacement ellipsoids drawn at the 30% probability level.

Synthesis and crystallization

Thiophene 2-carbonyl chloride (1 mmol) and dimethylaminopyridine (DMAP) (1.1 mmol) were dissolved in 10 ml of dry toluene and the mixture was refluxed with stirring for 1 h. The reaction mixture was cooled to room temperature and a solution of 2-aminomethylpyridine (1 mmol) in 5 ml of dry toluene was slowly added to it. The resultant solution was refluxed again for 3 h and the completion of the reaction was confirmed through TLC. The resultant solution was filtered and the filtrate volume was reduced using a rotary evaporator. The residue obtained was dissolved in dichloromethane and washed with water. The organic layer was separated and dried over sodium sulfate and kept for crystallization.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···N1^{i}	0.88	2.11	2.963 (2)	164
C5—H5···O1^{ii}	0.95	2.43	3.361 (2)	168
C9—H9···N1^{i}	0.95	2.56	3.441 (2)	155

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $x, y-1, z$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{11}H_{10}N_2OS$
M_r	218.27
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	110
a, b, c (Å)	8.681 (3), 8.088 (3), 14.875 (6)
β (°)	103.175 (4)
V (Å^3)	1016.8 (7)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.29
Crystal size (mm)	0.57 × 0.57 × 0.56
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2013)
T_{min}, T_{max}	0.539, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	11046, 2319, 2058
R_{int}	0.041
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.651
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.038, 0.098, 1.04
No. of reflections	2319
No. of parameters	136
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.29, −0.35

Computer programs: *APEX2* and *SAINT* (Bruker, 2013), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*) and *OLEX2* (Dolomanov *et al.*, 2009).

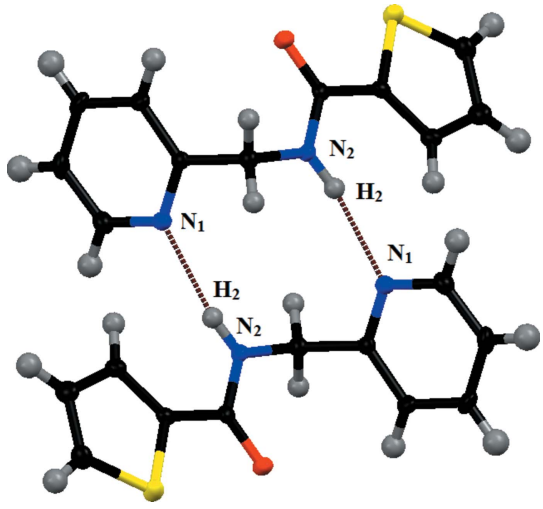


Figure 2
An inversion dimer with graph-set motif $R_2^2(10)$ formed by a pair of N—H···N hydrogen bonds. The bottom molecule is generated by the symmetry operation $-x+1, -y+1, -z+1$.

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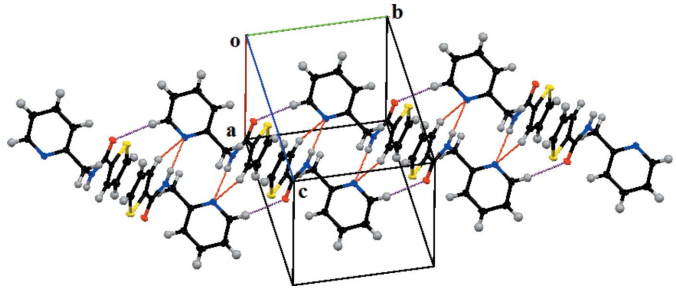

Figure 3
Packing diagram showing the formation of [010] chains linked by N—H···N, C—H···N and C—H···O hydrogen bonds.

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full crystallographic data

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Crystal data

$C_{11}H_{10}N_2OS$
M_r = 218.27
Monoclinic, $P2_1/c$
a = 8.681 (3) Å
b = 8.088 (3) Å
c = 14.875 (6) Å
β = 103.175 (4)°
V = 1016.8 (7) Å^3
Z = 4
$F(000)$ = 456
D_x = 1.426 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4931 reflections
θ = 2.4–27.5°
μ = 0.29 mm^{-1}
T = 110 K
Black, colourless
0.57 × 0.57 × 0.56 mm

Data collection

Bruker APEXII CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2013)
T_{min} = 0.539, T_{max} = 0.746
11046 measured reflections
2319 independent reflections
2058 reflections with $I > 2\sigma(I)$
R_{int} = 0.041
θ_{max} = 27.6°, θ_{min} = 2.4°
h = −11→11
k = −10→10
l = −19→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.098
S = 1.04
2319 reflections
136 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0462P)^2 + 0.5739P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

Special details

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

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.48410 (5)	1.06177 (5)	0.27924 (3)	0.02145 (13)

O1	0.27142 (13)	0.92442 (13)	0.38710 (8)	0.0201 (3)
N1	0.25034 (15)	0.41563 (15)	0.44501 (9)	0.0164 (3)
N2	0.44226 (14)	0.74541 (15)	0.47523 (9)	0.0149 (3)
H2	0.5392	0.7058	0.4897	0.018*
C1	0.20492 (17)	0.57130 (18)	0.45674 (10)	0.0138 (3)
C2	0.05897 (18)	0.63262 (19)	0.41132 (11)	0.0195 (3)
H2A	0.0290	0.7424	0.4224	0.023*
C3	−0.04294 (19)	0.5317 (2)	0.34950 (12)	0.0245 (4)
H3	−0.1432	0.5714	0.3169	0.029*
C4	0.00436 (19)	0.3724 (2)	0.33628 (12)	0.0238 (4)
H4	−0.0625	0.3006	0.2941	0.029*
C5	0.15017 (19)	0.3193 (2)	0.38527 (11)	0.0210 (3)
H5	0.1814	0.2091	0.3763	0.025*
C6	0.32272 (17)	0.68000 (18)	0.52048 (10)	0.0162 (3)
H6A	0.2661	0.7731	0.5418	0.019*
H6B	0.3754	0.6152	0.5754	0.019*
C7	0.40555 (17)	0.86572 (17)	0.41170 (10)	0.0143 (3)
C8	0.53409 (18)	0.92391 (17)	0.36918 (10)	0.0141 (3)
C9	0.69193 (18)	0.88705 (19)	0.38810 (11)	0.0176 (3)
H9	0.7421	0.8136	0.4357	0.021*
C10	0.77229 (19)	0.9712 (2)	0.32843 (11)	0.0213 (3)
H10	0.8824	0.9607	0.3317	0.026*
C11	0.6740 (2)	1.0683 (2)	0.26630 (11)	0.0208 (3)
H11	0.7072	1.1326	0.2207	0.025*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0171 (2)	0.0242 (2)	0.0209 (2)	−0.00174 (15)	−0.00019 (15)	0.00863 (15)
O1	0.0139 (5)	0.0177 (6)	0.0278 (6)	0.0023 (4)	0.0029 (5)	0.0045 (4)
N1	0.0146 (6)	0.0150 (6)	0.0199 (7)	0.0009 (5)	0.0046 (5)	0.0026 (5)
N2	0.0101 (6)	0.0151 (6)	0.0194 (6)	−0.0001 (5)	0.0029 (5)	0.0018 (5)
C1	0.0124 (7)	0.0155 (7)	0.0149 (7)	−0.0001 (5)	0.0061 (5)	0.0032 (5)
C2	0.0132 (7)	0.0178 (7)	0.0283 (9)	0.0020 (6)	0.0065 (6)	0.0048 (6)
C3	0.0112 (7)	0.0306 (9)	0.0296 (9)	−0.0014 (6)	0.0003 (6)	0.0080 (7)
C4	0.0194 (8)	0.0268 (9)	0.0236 (8)	−0.0102 (7)	0.0018 (6)	0.0008 (6)
C5	0.0223 (8)	0.0166 (7)	0.0252 (8)	−0.0024 (6)	0.0074 (7)	−0.0013 (6)
C6	0.0158 (7)	0.0168 (7)	0.0172 (7)	−0.0009 (6)	0.0063 (6)	0.0009 (6)
C7	0.0148 (7)	0.0110 (6)	0.0169 (7)	−0.0012 (5)	0.0032 (6)	−0.0025 (5)
C8	0.0175 (7)	0.0112 (6)	0.0131 (7)	−0.0001 (5)	0.0023 (5)	−0.0002 (5)
C9	0.0176 (8)	0.0168 (7)	0.0197 (8)	0.0027 (6)	0.0069 (6)	0.0033 (6)
C10	0.0197 (8)	0.0223 (8)	0.0249 (8)	0.0026 (6)	0.0110 (6)	0.0030 (6)
C11	0.0225 (8)	0.0226 (8)	0.0187 (8)	−0.0036 (6)	0.0075 (6)	0.0030 (6)

Geometric parameters (Å, º)

S1—C8	1.7195 (16)	C3—C4	1.380 (3)
S1—C11	1.7031 (18)	C4—H4	0.9500

O1—C7	1.2331 (18)	C4—C5	1.377 (2)
N1—C1	1.3425 (19)	C5—H5	0.9500
N1—C5	1.342 (2)	C6—H6A	0.9900
N2—H2	0.8800	C6—H6B	0.9900
N2—C6	1.4587 (18)	C7—C8	1.479 (2)
N2—C7	1.3431 (19)	C8—C9	1.367 (2)
C1—C2	1.385 (2)	C9—H9	0.9500
C1—C6	1.509 (2)	C9—C10	1.421 (2)
C2—H2A	0.9500	C10—H10	0.9500
C2—C3	1.387 (2)	C10—C11	1.356 (2)
C3—H3	0.9500	C11—H11	0.9500

C11—S1—C8	91.66 (8)	N2—C6—H6A	109.2
C5—N1—C1	117.73 (13)	N2—C6—H6B	109.2
C6—N2—H2	119.7	C1—C6—H6A	109.2
C7—N2—H2	119.7	C1—C6—H6B	109.2
C7—N2—C6	120.51 (13)	H6A—C6—H6B	107.9
N1—C1—C2	122.39 (14)	O1—C7—N2	122.98 (13)
N1—C1—C6	116.81 (13)	O1—C7—C8	120.25 (13)
C2—C1—C6	120.77 (14)	N2—C7—C8	116.75 (13)
C1—C2—H2A	120.4	C7—C8—S1	117.25 (11)
C1—C2—C3	119.16 (15)	C9—C8—S1	111.40 (11)
C3—C2—H2A	120.4	C9—C8—C7	131.35 (14)
C2—C3—H3	120.7	C8—C9—H9	123.9
C4—C3—C2	118.57 (15)	C8—C9—C10	112.24 (14)
C4—C3—H3	120.7	C10—C9—H9	123.9
C3—C4—H4	120.6	C9—C10—H10	123.8
C5—C4—C3	118.90 (15)	C11—C10—C9	112.37 (15)
C5—C4—H4	120.6	C11—C10—H10	123.8
N1—C5—C4	123.23 (15)	S1—C11—H11	123.8
N1—C5—H5	118.4	C10—C11—S1	112.33 (12)
C4—C5—H5	118.4	C10—C11—H11	123.8
N2—C6—C1	111.90 (12)		

S1—C8—C9—C10	−0.32 (17)	C5—N1—C1—C2	1.5 (2)
O1—C7—C8—S1	−5.13 (19)	C5—N1—C1—C6	−176.95 (13)
O1—C7—C8—C9	175.25 (15)	C6—N2—C7—O1	−1.4 (2)
N1—C1—C2—C3	−1.8 (2)	C6—N2—C7—C8	−179.75 (12)
N1—C1—C6—N2	79.64 (16)	C6—C1—C2—C3	176.59 (14)
N2—C7—C8—S1	173.26 (11)	C7—N2—C6—C1	73.47 (17)
N2—C7—C8—C9	−6.4 (2)	C7—C8—C9—C10	179.31 (15)
C1—N1—C5—C4	−0.2 (2)	C8—S1—C11—C10	−0.70 (13)
C1—C2—C3—C4	0.8 (2)	C8—C9—C10—C11	−0.2 (2)
C2—C1—C6—N2	−98.81 (16)	C9—C10—C11—S1	0.64 (19)
C2—C3—C4—C5	0.4 (2)	C11—S1—C8—C7	−179.12 (12)
C3—C4—C5—N1	−0.7 (2)	C11—S1—C8—C9	0.58 (12)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···N1^{i}	0.88	2.11	2.963 (2)	164
C5—H5···O1^{ii}	0.95	2.43	3.361 (2)	168
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