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ISSN 2414-3146

2-Amino-*N*-(furan-2-ylmethyl)-5,6-dihydro-4*H*-cyclopenta[*b*]thiophene-3-carboxamide

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Received 19 August 2017
Accepted 22 August 2017

Edited by W. T. A. Harrison, University of Aberdeen, Scotland

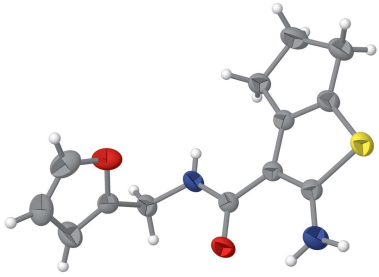
Keywords: crystal structure; thiophene derivative; hydrogen bonding; biological activities.

CCDC reference: 1570199

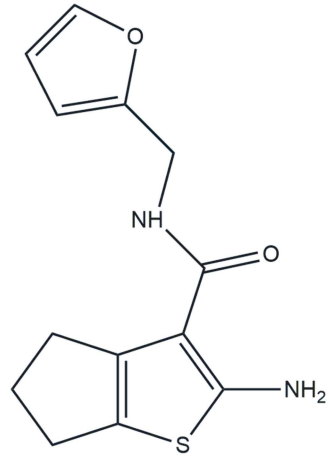
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{13}H_{14}N_2O_2S$, the dihedral angle between the furan and cyclopentathiophene groups is 89.88 (14)°. The carboximidamide unit is in an anticlinal conformation with respect to the cycloheptathiophene moiety and an intramolecular N—H···O hydrogen bond closes an *S*(6) ring. In the crystal, N—H···O hydrogen bonds link the molecules into [010] *C*(6) chains and a very weak C—H···O interaction is also observed.

3D view



Chemical scheme



Structure description

Tetrahydrothieno derivatives are studied extensively in medicinal chemistry due to their various biological activities (Lopez-Rodriguez *et al.*, 2001). As part of our interest in these compounds, we have synthesized the title compound (Fig. 1) and determined its crystal structure.

The mean plane of the furan moiety (O1/C10–C13) is oriented at a dihedral angle of 89.88 (14)° with respect to the plane of the almost planar (r.m.s. deviation = 0.014 Å) cyclopentathiophene ring (S1–C7). The carboximidamide unit is in an antiperiplanar conformation with respect to the cycloheptathiophene moiety, as indicated by the torsion angle of 177.07 (18)° for N2—C8—C7—C6. An intramolecular N1—H1*A*···O2 hydrogen bond (Table 1) closes an *S*(6) ring.

In the crystal, N1—H1*B*···O2^{i} hydrogen bonds link the molecules into *C*(6) chains propagating in the [010] direction (Fig. 2). A very weak C—H···O interaction is also observed. The amide N2—H1N group does not participate in hydrogen bonding, perhaps due to steric crowding.

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O2	0.86	2.15	2.738 (3)	125
N1—H1B···O2^{i}	0.86	2.06	2.902 (3)	167
C9—H9B···O1^{ii}	0.97	2.59	3.224 (4)	123

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

Synthesis and crystallization

Cyclopentanone (0.8 equivalents), diethylamine (0.9 equivalents) and *N*-(2-methoxyphenyl)acetamide (1.2 equivalents) were taken in 15 ml ethanol and mixed thoroughly in a microwave tube. The tube was sealed and irradiated at 325 K for 15 min. After cooling, ethyl acetate was added to the reaction mixture and the solid residue was removed by filtration. The reaction mixture was poured into ice-cold water, and the separated solid was filtered off and recrystallized from ethyl alcohol solution to give pale-yellow blocks of the title compound.

Refinement

The H atoms were positioned geometrically and allowed to ride on their parent atom, with N—H = 0.86 Å and C—H = 0.93–0.97 Å, and with U_{iso}(H) = 1.2–1.5U_{eq}(carrier) for all H atoms.

Acknowledgements

The authors thank HKBK College of Engineering, Bengaluru, R. V. College of Engineering, Bengaluru, and The National Institute of Engineering (NIE), Mysuru, for support.

References

Bruker (2009). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Lopez-Rodriguez, M. L., Murcia, M., Benhamu, B., Viso, A., Campillo, M. & Pardo, L. (2001). *Bioorg. Med. Chem. Lett.* **11**, 2807–2811.

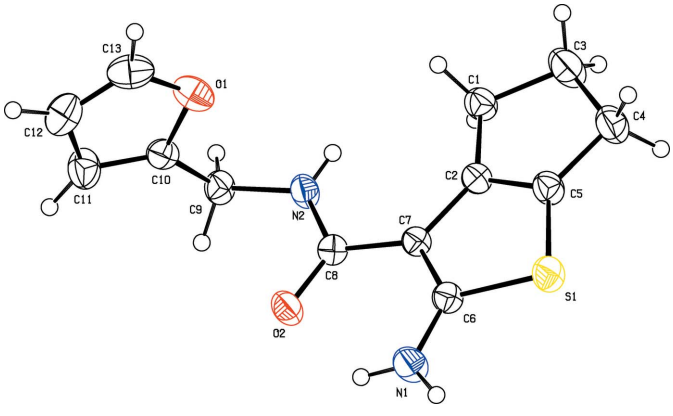


Figure 1
The molecular structure of (I), shown with 50% probability displacement ellipsoids.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{13}H_{14}N_2O_2S$
M_r	262.32
Crystal system, space group	Monoclinic, *C*2/*c*
Temperature (K)	273
a, *b*, *c* (Å)	21.694 (2), 9.8547 (11), 15.1162 (16)
β (°)	131.661 (2)
V (Å^3)	2414.3 (4)
Z	8
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.26
Crystal size (mm)	0.28 × 0.25 × 0.22
Data collection	
Diffractometer	Bruker APEXII CCD
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	9118, 2375, 1950
R_{int}	0.026
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.617
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.043, 0.120, 0.94
No. of reflections	2375
No. of parameters	167
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.25, −0.27

Computer programs: *APEX2* (Bruker, 2009), *SAINT* (Bruker, 2009), *SHELXS97* (Sheldrick, 2008), *SHELXL97* (Sheldrick, 2008) and *PLATON* (Spek, 2009).

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

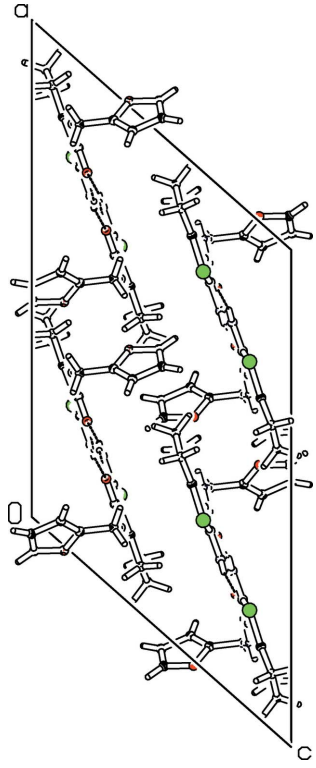


Figure 2
Packing diagram viewed down [010].

full crystallographic data

IUCrData (2017). **2**, x171211 [https://doi.org/10.1107/S2414314617012111]

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Crystal data

$C_{13}H_{14}N_2O_2S$
M_r = 262.32
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
a = 21.694 (2) Å
b = 9.8547 (11) Å
c = 15.1162 (16) Å
β = 131.661 (2)°
V = 2414.3 (4) Å^3
Z = 8
F(000) = 1104
D_x = 1.443 Mg m^{-3}
Mo *K*α radiation, λ = 0.71073 Å
Cell parameters from 2375 reflections
θ = 2.4–26.0°
μ = 0.26 mm^{-1}
T = 273 K
Block, pale yellow
0.28 × 0.25 × 0.22 mm

Data collection

Bruker APEXII CCD diffractometer
Detector resolution: 18.4 pixels mm^{-1}
ω and φ scans
9118 measured reflections
2375 independent reflections
1950 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 26.0°, θ_{min} = 2.4°
h = −25→26
k = −11→12
l = −18→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.120
S = 0.94
2375 reflections
167 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$W = 1/[\Sigma^2(FO^2) + (0.0768P)^2 + 1.5332P]$ WHERE $P = (FO^2 + 2FC^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Special details

Geometry. Bond distances, angles etc. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell esds are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted R-factors wR and all goodnesses 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 observed criterion of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating -R-factor-obs 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.20078 (3)	0.46023 (5)	0.33918 (5)	0.0417 (2)
O1	−0.01010 (9)	1.03238 (16)	0.12373 (13)	0.0494 (5)
O2	0.20894 (9)	0.91283 (14)	0.28657 (13)	0.0419 (5)
N1	0.24893 (11)	0.65497 (18)	0.27274 (16)	0.0460 (6)
N2	0.13634 (11)	0.94561 (16)	0.34213 (16)	0.0369 (6)
C1	0.09466 (13)	0.6855 (2)	0.42005 (18)	0.0387 (6)
C2	0.13894 (11)	0.64691 (19)	0.37874 (15)	0.0315 (5)
C3	0.07541 (17)	0.5480 (2)	0.4461 (3)	0.0579 (9)
C4	0.11271 (15)	0.4340 (2)	0.4249 (2)	0.0481 (8)
C5	0.14820 (12)	0.5112 (2)	0.38300 (17)	0.0374 (6)
C6	0.20996 (11)	0.62804 (19)	0.31246 (16)	0.0323 (6)
C7	0.17454 (11)	0.71866 (18)	0.33821 (15)	0.0296 (5)
C8	0.17492 (11)	0.86383 (19)	0.32061 (15)	0.0307 (5)
C9	0.12394 (12)	1.08935 (19)	0.31397 (17)	0.0357 (6)
C10	0.05629 (11)	1.11794 (19)	0.18473 (17)	0.0341 (6)
C11	0.04371 (14)	1.2137 (2)	0.11172 (19)	0.0488 (7)
C12	−0.03482 (14)	1.1888 (2)	−0.00186 (19)	0.0520 (8)
C13	−0.06404 (14)	1.0795 (3)	0.0094 (2)	0.0530 (8)
H1A	0.25426	0.73747	0.26020	0.0550*
H1B	0.26842	0.58963	0.25995	0.0550*
H1D	0.04431	0.73478	0.35898	0.0460*
H1E	0.12945	0.74105	0.49084	0.0460*
H1N	0.1123 (15)	0.914 (2)	0.359 (2)	0.047 (7)*
H3A	0.09891	0.54570	0.52755	0.0700*
H3B	0.01619	0.53591	0.39458	0.0700*
H4A	0.15513	0.38489	0.49735	0.0580*
H4B	0.07068	0.37072	0.36516	0.0580*
H9A	0.17493	1.12798	0.34037	0.0430*
H9B	0.11115	1.13391	0.35731	0.0430*
H11	0.07968	1.28374	0.13150	0.0590*
H12	−0.06040	1.23942	−0.07033	0.0620*
H13	−0.11455	1.03962	−0.05200	0.0640*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0525 (3)	0.0264 (3)	0.0565 (4)	−0.0005 (2)	0.0405 (3)	−0.0038 (2)
O1	0.0473 (9)	0.0544 (10)	0.0507 (9)	−0.0160 (7)	0.0344 (8)	−0.0103 (7)
O2	0.0565 (9)	0.0323 (7)	0.0596 (9)	−0.0007 (6)	0.0481 (8)	0.0022 (6)

N1	0.0646 (12)	0.0324 (9)	0.0711 (12)	0.0011 (8)	0.0578 (11)	−0.0032 (8)
N2	0.0517 (10)	0.0271 (9)	0.0492 (10)	0.0024 (7)	0.0408 (9)	0.0029 (7)
C1	0.0446 (11)	0.0370 (11)	0.0448 (11)	−0.0010 (9)	0.0340 (10)	−0.0001 (9)
C2	0.0326 (9)	0.0309 (10)	0.0309 (9)	−0.0025 (7)	0.0211 (8)	−0.0023 (7)
C3	0.0780 (17)	0.0436 (14)	0.0860 (18)	−0.0057 (11)	0.0687 (16)	0.0008 (12)
C4	0.0580 (13)	0.0365 (12)	0.0608 (14)	−0.0056 (10)	0.0441 (12)	0.0013 (10)
C5	0.0424 (11)	0.0311 (10)	0.0430 (11)	−0.0037 (8)	0.0302 (10)	−0.0023 (8)
C6	0.0346 (10)	0.0287 (10)	0.0345 (9)	−0.0025 (7)	0.0234 (8)	−0.0038 (7)
C7	0.0314 (9)	0.0283 (10)	0.0306 (9)	−0.0017 (7)	0.0213 (8)	−0.0022 (7)
C8	0.0324 (9)	0.0300 (10)	0.0297 (9)	−0.0003 (7)	0.0207 (8)	−0.0014 (7)
C9	0.0449 (11)	0.0256 (10)	0.0430 (11)	0.0003 (8)	0.0319 (10)	−0.0029 (8)
C10	0.0374 (10)	0.0280 (9)	0.0438 (11)	−0.0017 (8)	0.0299 (9)	−0.0061 (8)
C11	0.0503 (13)	0.0365 (12)	0.0469 (12)	−0.0049 (10)	0.0270 (11)	0.0013 (9)
C12	0.0537 (14)	0.0502 (14)	0.0418 (12)	0.0107 (11)	0.0274 (11)	0.0032 (10)
C13	0.0396 (12)	0.0718 (17)	0.0449 (13)	−0.0038 (11)	0.0269 (11)	−0.0131 (11)

Geometric parameters (Å, º)

S1—C5	1.731 (3)	C6—C7	1.392 (3)
S1—C6	1.745 (2)	C7—C8	1.456 (3)
O1—C10	1.368 (3)	C9—C10	1.496 (3)
O1—C13	1.372 (3)	C10—C11	1.335 (3)
O2—C8	1.244 (3)	C11—C12	1.422 (3)
N1—C6	1.349 (4)	C12—C13	1.317 (4)
N2—C8	1.348 (4)	C1—H1D	0.9700
N2—C9	1.452 (2)	C1—H1E	0.9700
C1—C2	1.502 (4)	C3—H3A	0.9700
C1—C3	1.544 (4)	C3—H3B	0.9700
N1—H1A	0.8600	C4—H4A	0.9700
N1—H1B	0.8600	C4—H4B	0.9700
N2—H1N	0.78 (4)	C9—H9A	0.9700
C2—C5	1.348 (3)	C9—H9B	0.9700
C2—C7	1.449 (4)	C11—H11	0.9300
C3—C4	1.538 (5)	C12—H12	0.9300
C4—C5	1.490 (4)	C13—H13	0.9300
C5—S1—C6	91.10 (12)	C10—C11—C12	107.4 (2)
C10—O1—C13	106.0 (2)	C11—C12—C13	106.2 (2)
C8—N2—C9	122.4 (2)	O1—C13—C12	111.0 (2)
C2—C1—C3	103.9 (2)	C2—C1—H1D	111.00
C6—N1—H1B	120.00	C2—C1—H1E	111.00
H1A—N1—H1B	120.00	C3—C1—H1D	111.00
C6—N1—H1A	120.00	C3—C1—H1E	111.00
C1—C2—C5	110.4 (2)	H1D—C1—H1E	109.00
C1—C2—C7	136.07 (18)	C1—C3—H3A	110.00
C8—N2—H1N	119.8 (18)	C1—C3—H3B	110.00
C9—N2—H1N	116.8 (19)	C4—C3—H3A	110.00
C5—C2—C7	113.6 (2)	C4—C3—H3B	110.00

C1—C3—C4	108.6 (3)	H3A—C3—H3B	108.00
C3—C4—C5	102.0 (2)	C3—C4—H4A	111.00
C2—C5—C4	115.1 (2)	C3—C4—H4B	111.00
S1—C5—C2	112.6 (2)	C5—C4—H4A	111.00
S1—C5—C4	132.26 (17)	C5—C4—H4B	111.00
S1—C6—N1	119.41 (17)	H4A—C4—H4B	109.00
S1—C6—C7	111.98 (19)	N2—C9—H9A	109.00
N1—C6—C7	128.61 (19)	N2—C9—H9B	109.00
C2—C7—C6	110.71 (17)	C10—C9—H9A	109.00
C6—C7—C8	120.5 (2)	C10—C9—H9B	109.00
C2—C7—C8	128.7 (2)	H9A—C9—H9B	108.00
O2—C8—C7	122.3 (2)	C10—C11—H11	126.00
N2—C8—C7	117.6 (2)	C12—C11—H11	126.00
O2—C8—N2	120.13 (18)	C11—C12—H12	127.00
N2—C9—C10	113.35 (16)	C13—C12—H12	127.00
O1—C10—C9	116.34 (18)	O1—C13—H13	125.00
O1—C10—C11	109.48 (19)	C12—C13—H13	124.00
C9—C10—C11	134.2 (2)		

C6—S1—C5—C2	−0.58 (17)	C1—C2—C7—C6	179.0 (2)
C6—S1—C5—C4	−179.9 (2)	C1—C2—C7—C8	−2.5 (4)
C5—S1—C6—N1	−179.91 (18)	C1—C3—C4—C5	−2.3 (3)
C5—S1—C6—C7	0.76 (16)	C3—C4—C5—S1	−179.6 (2)
C13—O1—C10—C9	178.8 (2)	C3—C4—C5—C2	1.1 (3)
C13—O1—C10—C11	0.3 (3)	S1—C6—C7—C2	−0.7 (2)
C10—O1—C13—C12	−0.8 (3)	N1—C6—C7—C8	1.4 (3)
C9—N2—C8—O2	7.5 (3)	S1—C6—C7—C8	−179.33 (14)
C9—N2—C8—C7	−172.27 (18)	N1—C6—C7—C2	−180.0 (2)
C8—N2—C9—C10	76.0 (3)	C2—C7—C8—O2	179.03 (19)
C2—C1—C3—C4	2.7 (3)	C2—C7—C8—N2	−1.2 (3)
C3—C1—C2—C5	−2.1 (3)	C6—C7—C8—O2	−2.7 (3)
C3—C1—C2—C7	179.2 (2)	C6—C7—C8—N2	177.07 (18)
C1—C2—C5—S1	−178.78 (14)	N2—C9—C10—O1	35.1 (4)
C1—C2—C5—C4	0.7 (3)	N2—C9—C10—C11	−147.0 (3)
C7—C2—C5—S1	0.3 (2)	O1—C10—C11—C12	0.2 (3)
C7—C2—C5—C4	179.70 (18)	C9—C10—C11—C12	−177.8 (3)
C5—C2—C7—C8	178.76 (19)	C10—C11—C12—C13	−0.7 (4)
C5—C2—C7—C6	0.3 (2)	C11—C12—C13—O1	0.9 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O2	0.86	2.15	2.738 (3)	125
N1—H1*B*···O2^{i}	0.86	2.06	2.902 (3)	167
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