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2-Amino-7-oxo-4,5,6,7-tetrahydro-1-benzothiophene-3-carbonitrile

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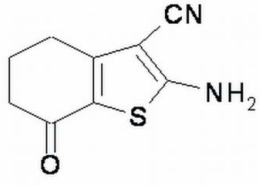
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.043; wR factor = 0.126; data-to-parameter ratio = 17.4.

In the title compound, $C_9H_8N_2OS$, the benzothiophene ring is substituted with amino, oxo and carbonitrile groups. The thiophene ring is essentially planar (r.m.s. deviation = 0.0003 Å), while the cyclohexene ring is in a half-chair conformation. In the crystal, N—H···O hydrogen bonds generate chains of molecules in a zigzag pattern along the b axis. Pairs of N—H···N hydrogen bonds form centrosymmetric head-to-head dimers about inversion centres, corresponding to an $R_2^2(12)$ graph-set motif. In addition, rather weak N—H···S interactions are also present in the structure and the supramolecular assembly is further consolidated by π–π stacking interactions between the benzothiophene rings, disposed at a distance of 3.742 (3) Å.

Related literature

For the preparation of the title compound, see: Shetty *et al.* (2009). For general background, see: Jordan (2003); Russell & Press (1996); Mery *et al.* (2002). For related structures, see: Akkurt *et al.* (2008); Harrison *et al.* (2006); Vasu *et al.* (2004). For Cremer–Pople puckering parameters, see: Cremer & Pople (1975). For hydrogen-bond graph-set nomenclature, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_9H_8N_2OS$
M_r = 192.24
Monoclinic, $P2_1/n$
a = 7.2986 (3) Å
b = 8.7555 (3) Å
c = 14.7307 (6) Å
β = 94.151 (1)°
V = 938.87 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.30 mm^{-1}
T = 296 K
0.20 × 0.18 × 0.18 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.942, T_{max} = 0.947
6202 measured reflections
2058 independent reflections
1671 reflections with $I > 2\sigma(I)$
R_{int} = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.126
S = 1.02
2058 reflections
118 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.70 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···N2^{i}	0.86	2.19	3.038 (3)	169
N1—H1B···O1^{ii}	0.86	2.10	2.903 (3)	156
N1—H1B···S1^{ii}	0.86	3.02	3.482 (2)	116

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT-Plus* (Bruker, 1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008) and *PARST* (Nardelli, 1983); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *CAMERON* (Watkin *et al.*, 1996); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

NSB is grateful to the University Grants Commission (UGC), India, for financial assistance, and the Department of Science and Technology (DST), India, for the data-collection facility under the IRHPA–DST programme.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2326).

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supporting information

Acta Cryst. (2010). E**66**, o2616–o2617 [doi:10.1107/S160053681003730X]

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S1. Comment

Benzothiophenes are important biologically active molecules. One of the most important drugs based on the benzothiophene system is Raloxifene, used for the prevention and treatment of osteoporosis in postmenopausal women (Jordan, 2003). Benzothiophenes are also luminescent components used in organic materials (Russell & Press, 1996). In addition, they are regarded as important units in liquid crystal research (Mery *et al.*, 2002). In this article, we report the structure of the title compound which has been synthesized in our laboratory.

In the title compound (Fig. 1), the thiophene ring is essentially planar while the cyclohexene ring is in a half-chair conformation; the atoms C5 and C6 deviate from the mean plane C1/C7/C4/C8 by 0.341 (3)and -0.233 (2) Å, respectively. The puckering parameters (Cremer & Pople, 1975) for the cyclohexene ring are: $Q(2)$ = 0.3175 (3) Å, $\varphi(2)$ = -17.96 (8)° and θ = 129.68 (7)°. In several benzothiophene derivatives the cyclohexyl ring adopts half-chair conformation, *e.g.*, (Akkurt *et al.*, 2008; Harrison *et al.*, 2006; Vasu *et al.*, 2004).

The N—H···O hydrogen bonds generate chains of molecules in a zigzag pattern along the *b*-axis. While the N—H···N hydrogen bonds form centrosymmetric, head-to-head dimers about inversion centers corresponding to graph set $R^2_2(12)$ motif. In addition, rather weak N—H···S interactions are also present in the structure and the supramolecular assembly is further consolidated by π–π-stacking interactions between the benzothiophene rings; C—C disposed at a distance of 3.742 (3) Å.

The intermolecular interactions of the type N—H···O, N—H···N and N—H···S stabilize the crystal structure (Table 1). The N1—H1A···O1 hydrogen bonds generate chains of molecules in a zigzag pattern along the *b*-axis (Fig. 2). The N1—H1B···N2 hydrogen bonds on the other hand, form centrosymmetric, head-to-head dimers about inversion centers corresponding to graph set $R^2_2(12)$ motif (Bernstein *et al.*, 1995) (Fig. 2). In addition, rather weak N1—H1B···S1 interactions are also present in the structure and the supramolecular assembly is further consolidated by π–π-stacking interactions between the benzothiophene rings; C—C disposed at a distance of 3.742 (3) Å.

S2. Experimental

The title compound was synthesized by following the procedure reported earlier (Shetty *et al.*, 2009).

S3. Refinement

The H atoms were placed at calculated positions in the riding model approximation with N—H = 0.86 and C—H = 0.97 Å, and U_{iso}(H) = 1.2U_{eq}(N/C).

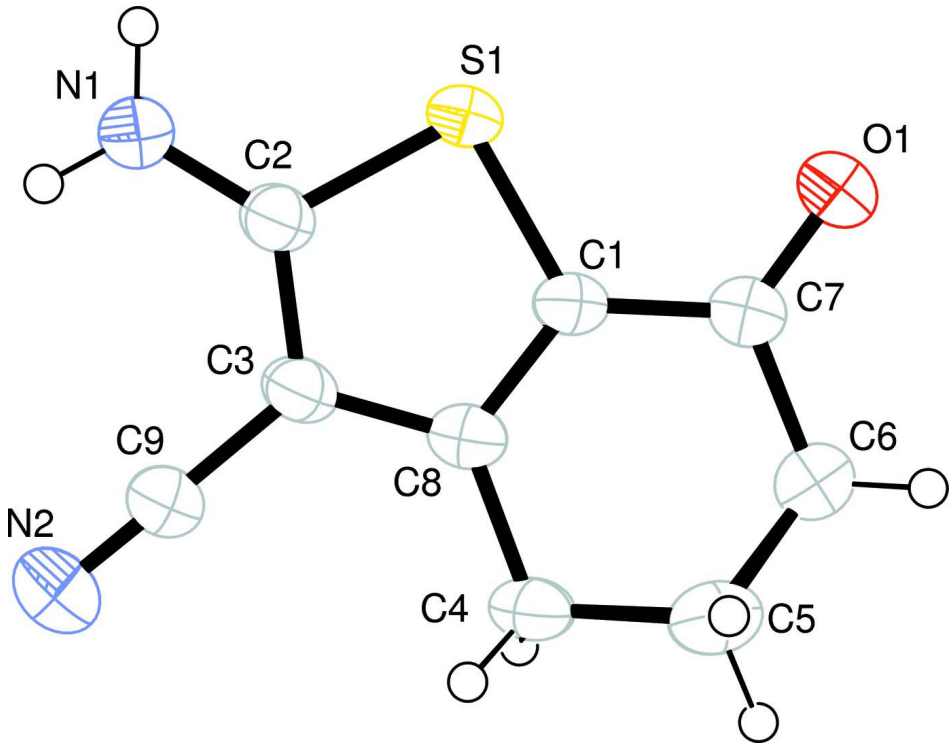

Figure 1

ORTEP (Farrugia, 1997) view of the title compound, showing 50% probability ellipsoids and the atom numbering scheme.

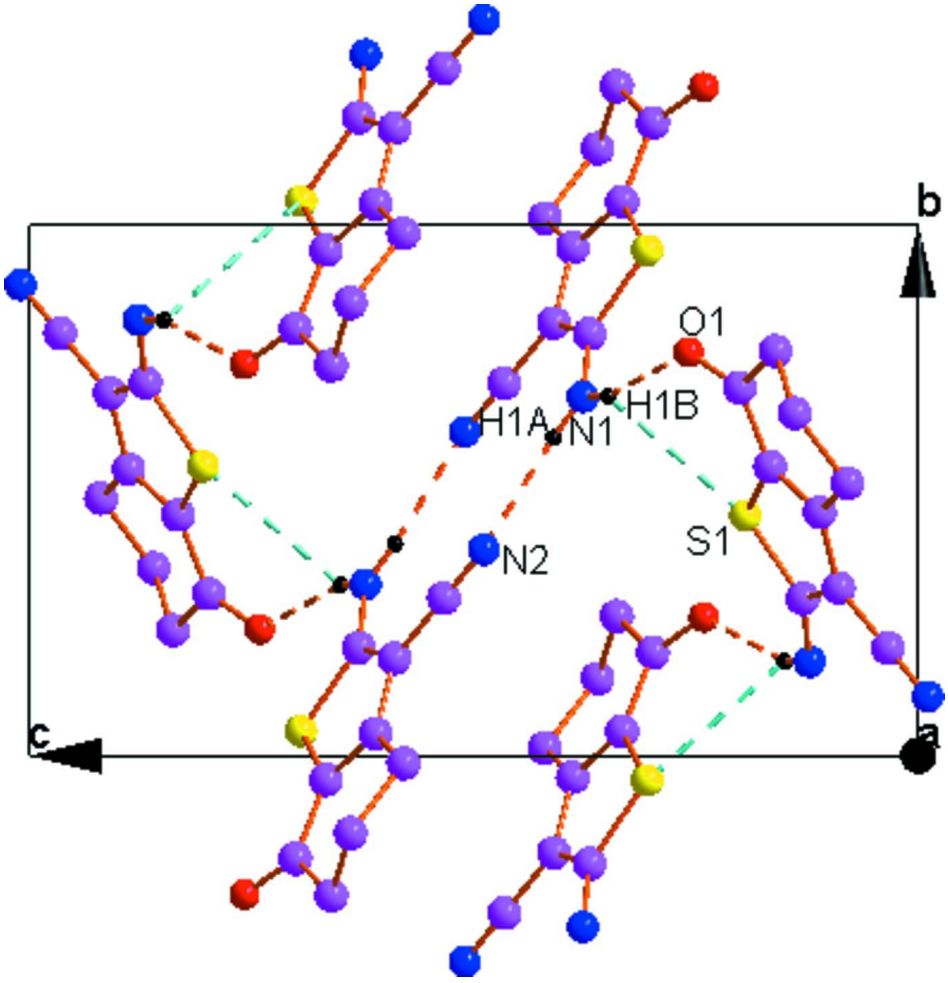

Figure 2

A unit cell packing of the title compound showing intermolecular hydrogen bonds with dotted lines. H atoms not involved in hydrogen bonding have been excluded.

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Crystal data

$C_9H_8N_2OS$
M_r = 192.24
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 7.2986 (3) Å
b = 8.7555 (3) Å
c = 14.7307 (6) Å
β = 94.151 (1)°
V = 938.87 (6) Å^3
Z = 4
F(000) = 400
D_x = 1.360 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2058 reflections
θ = 2.7–27.0°
μ = 0.30 mm^{-1}
T = 296 K
Block, yellow
0.20 × 0.18 × 0.18 mm

Data collection

Bruker SMART APEX CCD diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.942, T_{max} = 0.947

6202 measured reflections
2058 independent reflections
1671 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 27.0°, θ_{min} = 2.7°
$h = -9 \rightarrow 5$
$k = -11 \rightarrow 10$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.126
S = 1.02
2058 reflections
118 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0675P)^2 + 0.447P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.70 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

Special details

Experimental. The compound was synthesized by following the procedure given in Shetty *et al.*, (2009)

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 $F^2 > \sigma(F^2)$ 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}
C1	−0.0414 (3)	1.0457 (2)	0.33340 (13)	0.0378 (4)
C2	0.1087 (3)	0.7962 (2)	0.37011 (13)	0.0386 (4)
C3	−0.0580 (3)	0.8166 (2)	0.41027 (13)	0.0391 (4)
C4	−0.3211 (3)	1.0145 (3)	0.42024 (14)	0.0430 (5)
H4A	−0.3036	1.0483	0.4830	0.052*
H4B	−0.4087	0.9310	0.4176	0.052*
C5	−0.3979 (3)	1.1460 (3)	0.3610 (2)	0.0664 (7)
H5A	−0.4533	1.1044	0.3045	0.080*
H5B	−0.4942	1.1960	0.3921	0.080*
C6	−0.2579 (3)	1.2634 (3)	0.33910 (19)	0.0588 (6)
H6A	−0.2179	1.3181	0.3943	0.071*
H6B	−0.3147	1.3366	0.2964	0.071*
C7	−0.0921 (3)	1.1943 (2)	0.29875 (14)	0.0444 (5)
C8	−0.1417 (3)	0.9590 (2)	0.38916 (13)	0.0371 (4)
C9	−0.1327 (3)	0.7027 (3)	0.46592 (16)	0.0494 (5)

N1	0.2242 (3)	0.6773 (2)	0.37766 (13)	0.0539 (5)
H1A	0.1994	0.6002	0.4108	0.065*
H1B	0.3233	0.6781	0.3494	0.065*
N2	−0.1907 (3)	0.6114 (3)	0.51076 (18)	0.0781 (7)
O1	−0.0078 (2)	1.2613 (2)	0.24138 (12)	0.0622 (5)
S1	0.15957 (6)	0.95344 (6)	0.30522 (3)	0.04133 (19)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0312 (9)	0.0402 (10)	0.0431 (10)	−0.0005 (8)	0.0107 (8)	0.0018 (8)
C2	0.0401 (10)	0.0374 (10)	0.0395 (10)	−0.0024 (8)	0.0107 (8)	0.0004 (8)
C3	0.0396 (10)	0.0402 (10)	0.0391 (10)	−0.0035 (8)	0.0126 (8)	0.0025 (8)
C4	0.0363 (10)	0.0507 (12)	0.0437 (10)	−0.0021 (9)	0.0147 (8)	−0.0008 (9)
C5	0.0440 (12)	0.0647 (16)	0.0931 (19)	0.0074 (12)	0.0246 (12)	0.0112 (14)
C6	0.0500 (13)	0.0545 (13)	0.0741 (16)	0.0128 (11)	0.0200 (12)	0.0146 (12)
C7	0.0387 (10)	0.0458 (12)	0.0499 (11)	0.0007 (9)	0.0107 (9)	0.0069 (9)
C8	0.0340 (9)	0.0421 (11)	0.0359 (9)	−0.0037 (8)	0.0080 (7)	−0.0024 (8)
C9	0.0490 (12)	0.0433 (12)	0.0585 (13)	0.0020 (9)	0.0211 (10)	0.0079 (10)
N1	0.0563 (11)	0.0431 (10)	0.0659 (12)	0.0097 (8)	0.0285 (9)	0.0104 (9)
N2	0.0778 (16)	0.0632 (14)	0.0985 (18)	0.0055 (13)	0.0416 (14)	0.0298 (14)
O1	0.0572 (10)	0.0555 (10)	0.0773 (11)	0.0072 (8)	0.0295 (8)	0.0249 (8)
S1	0.0344 (3)	0.0419 (3)	0.0496 (3)	−0.0004 (2)	0.0160 (2)	0.0072 (2)

Geometric parameters (Å, º)

C1—C8	1.368 (3)	C4—H4B	0.9700
C1—C7	1.437 (3)	C5—C6	1.501 (3)
C1—S1	1.7504 (19)	C5—H5A	0.9700
C2—N1	1.339 (3)	C5—H5B	0.9700
C2—C3	1.402 (3)	C6—C7	1.512 (3)
C2—S1	1.732 (2)	C6—H6A	0.9700
C3—C8	1.414 (3)	C6—H6B	0.9700
C3—C9	1.424 (3)	C7—O1	1.230 (2)
C4—C8	1.499 (3)	C9—N2	1.138 (3)
C4—C5	1.526 (3)	N1—H1A	0.8600
C4—H4A	0.9700	N1—H1B	0.8600
C8—C1—C7	125.45 (18)	C4—C5—H5B	108.7
C8—C1—S1	112.37 (15)	H5A—C5—H5B	107.6
C7—C1—S1	122.18 (15)	C5—C6—C7	112.8 (2)
N1—C2—C3	128.63 (18)	C5—C6—H6A	109.0
N1—C2—S1	120.34 (15)	C7—C6—H6A	109.0
C3—C2—S1	111.02 (15)	C5—C6—H6B	109.0
C2—C3—C8	113.20 (17)	C7—C6—H6B	109.0
C2—C3—C9	122.26 (19)	H6A—C6—H6B	107.8
C8—C3—C9	124.54 (18)	O1—C7—C1	123.17 (19)
C8—C4—C5	111.26 (17)	O1—C7—C6	122.3 (2)

C8—C4—H4A	109.4	C1—C7—C6	114.57 (18)
C5—C4—H4A	109.4	C1—C8—C3	112.31 (17)
C8—C4—H4B	109.4	C1—C8—C4	121.39 (19)
C5—C4—H4B	109.4	C3—C8—C4	126.28 (17)
H4A—C4—H4B	108.0	N2—C9—C3	179.3 (3)
C6—C5—C4	114.3 (2)	C2—N1—H1A	120.0
C6—C5—H5A	108.7	C2—N1—H1B	120.0
C4—C5—H5A	108.7	H1A—N1—H1B	120.0
C6—C5—H5B	108.7	C2—S1—C1	91.10 (9)

N1—C2—C3—C8	178.3 (2)	C7—C1—C8—C4	0.0 (3)
S1—C2—C3—C8	−0.7 (2)	S1—C1—C8—C4	−178.70 (14)
N1—C2—C3—C9	−2.1 (4)	C2—C3—C8—C1	0.4 (3)
S1—C2—C3—C9	178.95 (17)	C9—C3—C8—C1	−179.2 (2)
C8—C4—C5—C6	−43.4 (3)	C2—C3—C8—C4	179.08 (18)
C4—C5—C6—C7	52.8 (3)	C9—C3—C8—C4	−0.5 (3)
C8—C1—C7—O1	−172.2 (2)	C5—C4—C8—C1	17.3 (3)
S1—C1—C7—O1	6.4 (3)	C5—C4—C8—C3	−161.3 (2)
C8—C1—C7—C6	8.3 (3)	C2—C3—C9—N2	38 (24)
S1—C1—C7—C6	−173.14 (17)	C8—C3—C9—N2	−142 (24)
C5—C6—C7—O1	146.5 (2)	N1—C2—S1—C1	−178.50 (18)
C5—C6—C7—C1	−33.9 (3)	C3—C2—S1—C1	0.57 (16)
C7—C1—C8—C3	178.76 (19)	C8—C1—S1—C2	−0.37 (17)
S1—C1—C8—C3	0.1 (2)	C7—C1—S1—C2	−179.11 (18)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···N2^{i}	0.86	2.19	3.038 (3)	169
N1—H1*B*···O1^{ii}	0.86	2.10	2.903 (3)	156
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