

N-(2-Thienylmethylene)naphthalen-1-amine

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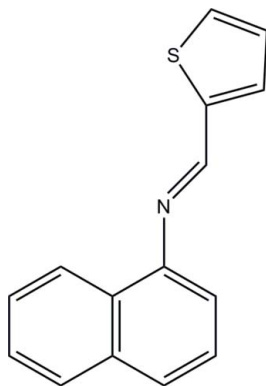
Received 20 September 2009; accepted 13 October 2009

Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.036; wR factor = 0.076; data-to-parameter ratio = 13.7.

In the title compound, $\text{C}_{15}\text{H}_{11}\text{NS}$, the dihedral angle between the thiophene and 1-naphthyl rings is $31.42(11)^\circ$. The molecule adopts a *trans* configuration about the central $\text{C}=\text{N}$ bond. In the crystal, the molecules are connected *via* weak $\text{C}-\text{H}\cdots\pi$ interactions.

Related literature

The condensation of primary amines with carbonyl compounds yields Schiff bases, see: Dey *et al.* (1981). For the chemistry and applications of Schiff bases, see: Doine (1985); Opstal *et al.* (2002).



Experimental

Crystal data

$\text{C}_{15}\text{H}_{11}\text{NS}$	$V = 2457.7(4) \text{ \AA}^3$
$M_r = 237.31$	$Z = 8$
Orthorhombic, $Aba2$	Mo $K\alpha$ radiation
$a = 10.7793(12) \text{ \AA}$	$\mu = 0.24 \text{ mm}^{-1}$
$b = 21.260(2) \text{ \AA}$	$T = 298 \text{ K}$
$c = 10.7244(10) \text{ \AA}$	$0.40 \times 0.38 \times 0.18 \text{ mm}$

Data collection

Bruker SMART APEX CCD area-detector diffractometer	5898 measured reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	2115 independent reflections
$T_{\min} = 0.911$, $T_{\max} = 0.958$	1613 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.038$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.036$	H-atom parameters constrained
$wR(F^2) = 0.076$	$\Delta\rho_{\max} = 0.14 \text{ e \AA}^{-3}$
$S = 1.05$	$\Delta\rho_{\min} = -0.22 \text{ e \AA}^{-3}$
2115 reflections	Absolute structure: Flack (1983),
154 parameters	965 Friedel pairs
1 restraint	Flack parameter: 0.01 (9)

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C13}-\text{H13}\cdots\text{Cg1}^i$	0.93	2.87	3.783 (3)	168

Symmetry code: (i) $-x, -y, z$. Cg1 is the centroid of the S1,C2–C5 ring.

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL.

The authors acknowledge the financial support of Liaocheng University (X20090101).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BX2244).

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supplementary materials

Acta Cryst. (2009). E65, o2779 [doi:10.1107/S1600536809041774]

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Comment

The condensation of primary amines with carbonyl compounds yields Schiff bases (Dey *et al.*, 1981). In the recent years, there has been considerable interest in the chemistry of Schiff bases (Doine, 1985). This is due to the fact that Schiff bases offer opportunities for inducing substrate chirality, tuning the metal centred electronic factor, enhancing the solubility and stability of either homogeneous or heterogeneous catalysts (Opstal *et al.*, 2002). We report here the synthesis and crystal structure of (I) present a new compound, 2-(2-(naphthalen-1-yl)vinyl)thiophene schiff base, (I) in this paper.

The structure of (I) consists of 1-naphthyl ring covalently linked to a thiophene ring by an azomethine bond with more stable E isomer being observed. The mean plane of the 1-naphthyl ring is twisted by 35.4 (2)° from the azomethine bond to which is connected. The molecule adopts a trans configuration about the central C=N bond. In the crystal structure the molecules are interconnected via a C—H··· π interactions, and the molecular structure is stabilized by one intramolecular C—H···N hydrogen bond, Table 1, Fig 2.

Experimental

Naphthylamine (10 mmol), thiophene-2-carbaldehyde (20 mmol) and 20 ml ethanol were mixed in 50 ml flask. After stirring 3 h at 303 K, the resulting mixture was recrystallized from ethanol, affording the title compound as a red crystalline solid. The single crystals were obtained methylene dichloride and n-hexane solution. The Elemental analysis: calculated for C₁₅H₁₁NS: C 75.91, H 4.67, N 5.90%; found: C 75.82, H 4.54, N 9.57%.

Refinement

All H atoms were placed in geometrically idealized positions (C—H distances is 0.93 Å) and treated as riding on their parent atoms, with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$.

Figures

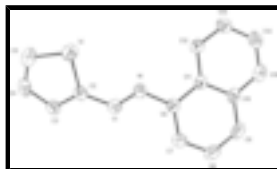


Fig. 1. The content of asymmetric unit of the title compound showing the atomic numbering scheme and 30% probability displacement ellipsoids.

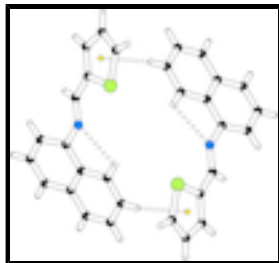


Fig. 2. Part of the crystal structure of (I) showing the hydrogen bond and C-H... π interactions.

***N*-(2-Thienylmethylene)naphthalen-1-amine**

Crystal data

$C_{15}H_{11}NS$	$D_x = 1.283 \text{ Mg m}^{-3}$
$M_r = 237.31$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
Orthorhombic, <i>Aba2</i>	Cell parameters from 2185 reflections
$a = 10.7793 (12) \text{ \AA}$	$\theta = 2.7\text{--}22.1^\circ$
$b = 21.260 (2) \text{ \AA}$	$\mu = 0.24 \text{ mm}^{-1}$
$c = 10.7244 (10) \text{ \AA}$	$T = 298 \text{ K}$
$V = 2457.7 (4) \text{ \AA}^3$	Block, red
$Z = 8$	$0.40 \times 0.38 \times 0.18 \text{ mm}$
$F_{000} = 992$	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	2115 independent reflections
Radiation source: fine-focus sealed tube	1613 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.038$
$T = 298 \text{ K}$	$\theta_{\text{max}} = 25.0^\circ$
phi and ω scans	$\theta_{\text{min}} = 1.9^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -12 \rightarrow 12$
$T_{\text{min}} = 0.911$, $T_{\text{max}} = 0.958$	$k = -25 \rightarrow 15$
5898 measured reflections	$l = -11 \rightarrow 12$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.036$	$w = 1/[\sigma^2(F_o^2) + (0.0332P)^2]$
$wR(F^2) = 0.076$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{\text{max}} < 0.001$
2115 reflections	$\Delta\rho_{\text{max}} = 0.14 \text{ e \AA}^{-3}$
154 parameters	$\Delta\rho_{\text{min}} = -0.22 \text{ e \AA}^{-3}$
	Extinction correction: none

1 restraint Absolute structure: Flack (1983), 965 Friedel pairs
 Primary atom site location: structure-invariant direct methods Flack parameter: 0.01 (9)
 Secondary atom site location: difference Fourier map

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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.21178 (7)	0.07346 (3)	0.26408 (7)	0.0623 (2)
N1	0.0437 (2)	0.14325 (8)	0.08130 (17)	0.0461 (5)
C1	0.1600 (2)	0.14895 (11)	0.0624 (2)	0.0479 (6)
H1	0.1865	0.1716	-0.0068	0.058*
C2	0.2522 (2)	0.12187 (12)	0.1433 (2)	0.0473 (6)
C3	0.3789 (2)	0.12590 (13)	0.1330 (2)	0.0590 (7)
H3	0.4188	0.1494	0.0718	0.071*
C4	0.4419 (3)	0.09107 (14)	0.2238 (3)	0.0657 (8)
H4	0.5279	0.0893	0.2306	0.079*
C5	0.3642 (3)	0.06064 (14)	0.2997 (2)	0.0684 (9)
H5	0.3903	0.0351	0.3651	0.082*
C6	-0.0400 (2)	0.16984 (11)	-0.0053 (2)	0.0443 (6)
C7	-0.0183 (3)	0.22590 (13)	-0.0651 (3)	0.0589 (7)
H7	0.0523	0.2491	-0.0462	0.071*
C8	-0.1020 (3)	0.24811 (14)	-0.1541 (3)	0.0713 (8)
H8	-0.0853	0.2857	-0.1952	0.086*
C9	-0.2072 (3)	0.21592 (14)	-0.1819 (3)	0.0645 (8)
H9	-0.2608	0.2313	-0.2426	0.077*
C10	-0.2360 (2)	0.15979 (12)	-0.1201 (2)	0.0478 (6)
C11	-0.1523 (2)	0.13664 (11)	-0.0279 (2)	0.0411 (6)
C12	-0.1834 (3)	0.08006 (11)	0.0346 (3)	0.0502 (7)
H12	-0.1291	0.0633	0.0932	0.060*
C13	-0.2930 (3)	0.04964 (15)	0.0095 (3)	0.0608 (9)
H13	-0.3135	0.0132	0.0529	0.073*
C14	-0.3738 (3)	0.07315 (14)	-0.0811 (3)	0.0637 (8)
H14	-0.4475	0.0520	-0.0978	0.076*
C15	-0.3464 (2)	0.12602 (14)	-0.1444 (2)	0.0577 (7)
H15	-0.4009	0.1406	-0.2051	0.069*

supplementary materials

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0579 (4)	0.0738 (5)	0.0552 (4)	0.0094 (4)	0.0061 (4)	0.0063 (4)
N1	0.0454 (14)	0.0480 (12)	0.0448 (11)	0.0009 (10)	-0.0020 (10)	-0.0021 (10)
C1	0.0512 (18)	0.0478 (15)	0.0447 (14)	0.0005 (13)	0.0007 (14)	-0.0021 (11)
C2	0.0463 (15)	0.0472 (14)	0.0485 (14)	-0.0001 (12)	-0.0004 (12)	-0.0075 (12)
C3	0.0502 (18)	0.0651 (17)	0.0617 (16)	-0.0062 (14)	-0.0018 (14)	0.0004 (15)
C4	0.0409 (17)	0.086 (2)	0.0697 (18)	0.0033 (15)	-0.0091 (15)	-0.0108 (17)
C5	0.068 (2)	0.081 (2)	0.0557 (19)	0.0227 (17)	-0.0098 (15)	-0.0066 (15)
C6	0.0417 (16)	0.0465 (15)	0.0445 (14)	0.0047 (13)	0.0049 (12)	-0.0015 (13)
C7	0.0542 (18)	0.0503 (17)	0.0724 (17)	-0.0043 (14)	0.0003 (14)	0.0104 (15)
C8	0.067 (2)	0.0600 (18)	0.087 (2)	0.0053 (17)	0.0011 (18)	0.0265 (17)
C9	0.0610 (19)	0.0690 (19)	0.0634 (16)	0.0200 (17)	-0.0064 (15)	0.0175 (16)
C10	0.0453 (16)	0.0531 (15)	0.0450 (13)	0.0116 (13)	0.0026 (12)	-0.0004 (13)
C11	0.0414 (15)	0.0440 (14)	0.0380 (12)	0.0053 (12)	0.0009 (11)	-0.0032 (12)
C12	0.055 (2)	0.0485 (16)	0.0471 (14)	0.0007 (14)	-0.0011 (12)	0.0038 (12)
C13	0.060 (2)	0.0553 (16)	0.0668 (18)	-0.0102 (16)	-0.0012 (15)	0.0014 (15)
C14	0.0516 (19)	0.060 (2)	0.080 (2)	-0.0047 (15)	-0.0107 (16)	-0.0131 (17)
C15	0.0547 (18)	0.0625 (19)	0.0558 (15)	0.0154 (15)	-0.0111 (13)	-0.0086 (15)

Geometric parameters (\AA , $^\circ$)

S1—C5	1.709 (3)	C7—H7	0.9300
S1—C2	1.711 (3)	C8—C9	1.358 (4)
N1—C1	1.276 (3)	C8—H8	0.9300
N1—C6	1.413 (3)	C9—C10	1.400 (4)
C1—C2	1.440 (3)	C9—H9	0.9300
C1—H1	0.9300	C10—C15	1.414 (4)
C2—C3	1.372 (4)	C10—C11	1.427 (3)
C3—C4	1.400 (4)	C11—C12	1.417 (3)
C3—H3	0.9300	C12—C13	1.374 (4)
C4—C5	1.335 (4)	C12—H12	0.9300
C4—H4	0.9300	C13—C14	1.396 (4)
C5—H5	0.9300	C13—H13	0.9300
C6—C7	1.373 (3)	C14—C15	1.346 (4)
C6—C11	1.422 (3)	C14—H14	0.9300
C7—C8	1.396 (4)	C15—H15	0.9300
C5—S1—C2	91.16 (14)	C9—C8—H8	119.3
C1—N1—C6	119.0 (2)	C7—C8—H8	119.3
N1—C1—C2	123.0 (2)	C8—C9—C10	120.7 (3)
N1—C1—H1	118.5	C8—C9—H9	119.7
C2—C1—H1	118.5	C10—C9—H9	119.7
C3—C2—C1	127.8 (2)	C9—C10—C15	122.1 (2)
C3—C2—S1	110.6 (2)	C9—C10—C11	118.8 (2)
C1—C2—S1	121.4 (2)	C15—C10—C11	119.0 (2)
C2—C3—C4	113.2 (3)	C12—C11—C6	122.9 (2)

C2—C3—H3	123.4	C12—C11—C10	118.1 (2)
C4—C3—H3	123.4	C6—C11—C10	119.0 (2)
C5—C4—C3	112.1 (3)	C13—C12—C11	120.7 (3)
C5—C4—H4	124.0	C13—C12—H12	119.6
C3—C4—H4	124.0	C11—C12—H12	119.6
C4—C5—S1	112.9 (2)	C12—C13—C14	120.3 (3)
C4—C5—H5	123.5	C12—C13—H13	119.9
S1—C5—H5	123.5	C14—C13—H13	119.9
C7—C6—N1	123.1 (2)	C15—C14—C13	120.9 (3)
C7—C6—C11	119.8 (2)	C15—C14—H14	119.6
N1—C6—C11	117.2 (2)	C13—C14—H14	119.6
C6—C7—C8	120.2 (3)	C14—C15—C10	121.0 (3)
C6—C7—H7	119.9	C14—C15—H15	119.5
C8—C7—H7	119.9	C10—C15—H15	119.5
C9—C8—C7	121.3 (3)		
C6—N1—C1—C2	-178.1 (2)	C8—C9—C10—C11	-0.5 (4)
N1—C1—C2—C3	-178.9 (2)	C7—C6—C11—C12	-177.1 (2)
N1—C1—C2—S1	6.2 (3)	N1—C6—C11—C12	1.6 (3)
C5—S1—C2—C3	0.9 (2)	C7—C6—C11—C10	5.0 (3)
C5—S1—C2—C1	176.7 (2)	N1—C6—C11—C10	-176.3 (2)
C1—C2—C3—C4	-176.7 (2)	C9—C10—C11—C12	179.4 (2)
S1—C2—C3—C4	-1.3 (3)	C15—C10—C11—C12	0.6 (3)
C2—C3—C4—C5	1.0 (3)	C9—C10—C11—C6	-2.6 (3)
C3—C4—C5—S1	-0.3 (3)	C15—C10—C11—C6	178.6 (2)
C2—S1—C5—C4	-0.4 (2)	C6—C11—C12—C13	-179.9 (2)
C1—N1—C6—C7	-36.6 (3)	C10—C11—C12—C13	-2.0 (4)
C1—N1—C6—C11	144.7 (2)	C11—C12—C13—C14	2.0 (4)
N1—C6—C7—C8	176.9 (2)	C12—C13—C14—C15	-0.5 (4)
C11—C6—C7—C8	-4.4 (4)	C13—C14—C15—C10	-0.9 (4)
C6—C7—C8—C9	1.4 (4)	C9—C10—C15—C14	-177.9 (3)
C7—C8—C9—C10	1.1 (5)	C11—C10—C15—C14	0.8 (4)
C8—C9—C10—C15	178.3 (3)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C12—H12...N1	0.93	2.52	2.837 (4)	100
C13—H13...Cg1 ⁱ	0.93	2.87	3.783 (3)	168

Symmetry codes: (i) $-x, -y, z$.

Fig. 1

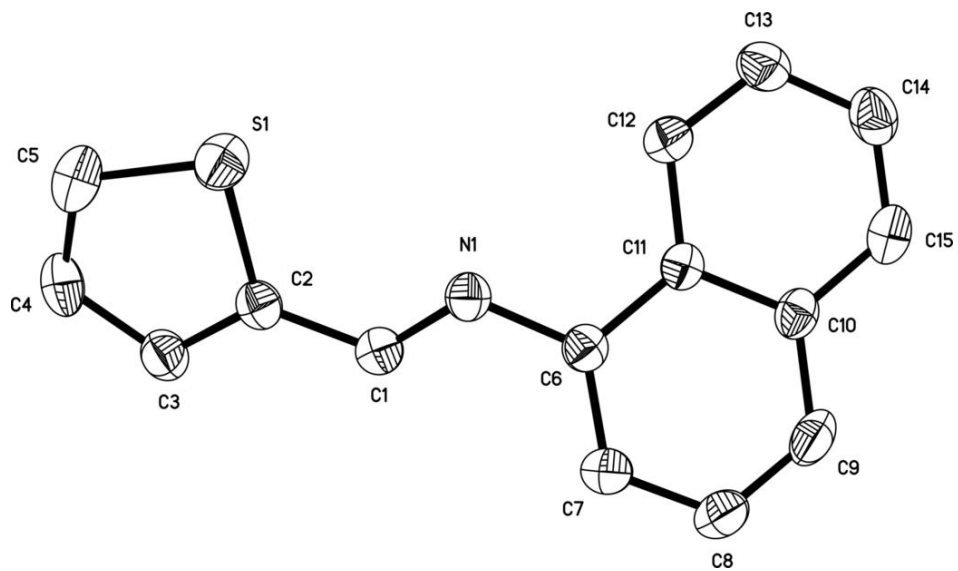


Fig. 2

