Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

(*E*)-*N*-[(5-Bromothiophen-2-yl)methylene]-5-methylthiazol-2-amine

Yong-Bing Gu, Chun-Niu Zhang and Ming-Hua Yang*

Department of Chemistry, Lishui University, 323000 Lishui, Zhejiang, People's Republic of China
Correspondence e-mail: zjlsxyhx@126.com

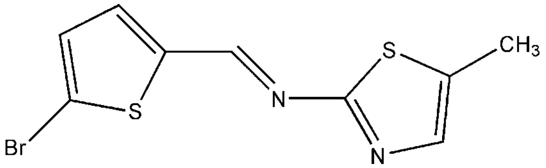
Received 20 April 2007; accepted 23 April 2007

Key indicators: single-crystal X-ray study; *T* = 298 K; mean σ(C–C) = 0.004 Å; *R* factor = 0.034; *wR* factor = 0.084; data-to-parameter ratio = 16.4.

The title compound, $C_9H_7BrN_2S_2$, is a Schiff base. There is an intramolecular hydrogen bond which stabilizes the structure. The molecules are further assembled into a supramolecular network *via* weak C—H···N intermolecular hydrogen bonds and π–π interactions with a distance of 3.15 Å between the thiophene rings.

Related literature

For general background, see: Johnson *et al.* (1996); Alizadeh *et al.* (1999); Alemi *et al.* (2000); Kim *et al.* (1999).



Experimental

Crystal data

$C_9H_7BrN_2S_2$	V = 1088.03 (19) Å^3
M_r = 287.20	Z = 4
Monoclinic, $P2_1/c$	Mo *Kα* radiation
a = 9.5515 (10) Å	μ = 4.12 mm^{-1}
b = 10.9177 (11) Å	T = 298 (2) K
c = 10.9141 (11) Å	0.36 × 0.22 × 0.22 mm
β = 107.06 (10)°	

Data collection

Bruker APEXII area-detector diffractometer	6883 measured reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996) T_{min} = 0.922, T_{max} = 0.938 (expected range = 0.397–0.404)	2096 independent reflections
	1622 reflections with $I > 2\sigma(I)$
	R_{int} = 0.067

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034	128 parameters
$wR(F^2)$ = 0.084	H-atom parameters constrained
S = 0.94	$\Delta\rho_{max}$ = 0.73 e Å^{-3}
2096 reflections	$\Delta\rho_{min}$ = −0.53 e Å^{-3}

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5···S2	0.93	2.57	3.034 (3)	112
C5—H5···N2^{i}	0.93	2.52	3.398 (4)	157

Symmetry code: (i) $x, -y+\frac{3}{2}, z-\frac{1}{2}$.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

The authors are grateful to the Natural Science Foundation of Zhejiang Province (No. M203052) and the Research Foundation of Lishui University (No. FC06002) for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2165).

References

Alemi, A. A. & Shaabani, B. (2000). *Acta Chim. Slov.* **47**, 363–369.
Alizadeh, N., Ershad, S., Naeimi, H., Sharghi, H. & Shamsipur, M. (1999). *Pol. J. Chem.* **73**, 915–925.
Bruker (1998). *SMART* (Version 5.0) and *SHELXTL* (Version 5.1). Bruker AXS Inc, Madison, Wisconsin, USA.
Bruker (1999). *SAINT*. Version 6.0. Bruker AXS Inc., Madison, Wisconsis, USA.
Johnson, C. P., Atwood, J. L., Steed, J. W., Bauer, C. B. & Rogers, R. D. (1996). *Inorg. Chem.* **35**, 2602–2610.
Kim, G. J. & Shin, J. W. (1999). *Catal. Lett.* **63**, 83–89.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

doi:10.1107/S1600536807020211

supplementary materials

Acta Cryst. (2007). E**63**, o2708 [**doi:10.1107/S1600536807020211**]

(*E*)-*N*-[(5-Bromothiophen-2-yl)methylene]-5-methylthiazol-2-amine

Y.-B. Gu, C.-N. Zhang and **M.-H. Yang**

Comment

Schiff base ligands have significant importance in chemistry, specially in the development of Schiff base complexes, because Schiff base ligands are potentially capable of forming stable complexes with metal ions (Johnson *et al.*, 1996; Alizadeh *et al.*, 1999). Schiff bases that have solvent dependent UV/vis spectra (solvatochromicity) can be suitable NLO (nonlinear optical active) materials (Alemi *et al.*, 2000). They are also useful in asymmetric oxidation of methyl phenyl sulfide and enantioselective (Kim *et al.*, 1999). In this paper, we report the synthesis and crystal structure of the title compound (I).

The molecular structure of the title compound (Fig. 1) contains one intramolecular hydrogen bonds [C5—H5\···S2](Table 1). The C5—N1 bond lengths is 1.277 (3) Å, indicative of standard C=N double bond. The other C—N, C—S and C—C distances show no remarkable features. A supramolecular network is formed by C—H···N intermolecular hydrogen bonding and weak π-π interactions. The centroid-to-centroid and interplanar distances between adjacent rings (Symmetry code: 1 - *x*, 2 - *y*, -*z*) are 3.712 (4) and 3.556 (3) Å, respectively.

Experimental

Under nitrogen, a mixture of 5-methylthiazol-2-amine (1.63 g,8 mmol), Na_2SO_4 (3.0 g) and 5-bromo-2-thiophenecarboxaldehyde(1.52 g,10 mmol) in absolute ethanol (20 ml) was refluxed for about 12 h to yield a yellow precipitate. The product was collected by vacuum filtration and washed with ethanol. The crude solid was redissolved in CH_2Cl_2 (100 ml) and washed with water (2*10 ml)and brine(10 ml). After dried over Na_2SO_4, the solvent was removed under vacuum, and yellow solid was isolated in yield 92% (3.1 g). Yellow single crystals of the compound suitable for X-ray analysis were grown from CH_2Cl_2 and absolute ethanol(4:1) by slow evaporation of the solvent at room temperature over a period of about a week.

Refinement

All H atoms were placed in calculated positions, C—H = 0.93Å ($C_{aromatic}$) or 0.96 Å (CH_3) and treated as riding on their parent atoms, with $U_{iso}(H) = 1.2U_{eq}(C_{aromatic})$ and $U_{iso}(H) = 1.5U_{eq}(CH_3)$.

Figures

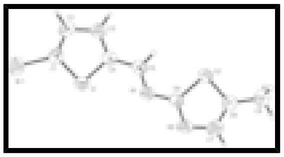


Fig. 1. The structure of (I), showing the atomic numbering scheme. Non-H atoms are shown as 50% probability displacement ellipsoids.

(*E*)-*N*-[(5-Bromothiophen-2-yl)methylene]-5-methylthiazol-2-amine

Crystal data

$C_9H_7BrN_2S_2$	F_{000} = 568
M_r = 287.20	D_x = 1.753 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 2100 reflections
a = 9.5515 (10) Å	θ = 1.7–28.0º
b = 10.9177 (11) Å	μ = 4.12 mm^{-1}
c = 10.9141 (11) Å	*T* = 298 (2) K
β = 107.06 (10)º	Block, colourless
V = 1088.03 (19) Å^3	0.36 × 0.22 × 0.22 mm
Z = 4	

Data collection

Bruker APEXII area-detector diffractometer	2096 independent reflections
Radiation source: fine-focus sealed tube	1622 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.067
T = 298(2) K	θ_{max} = 26.0º
φ and ω scans	θ_{min} = 2.2º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -11 \rightarrow 11$
T_{min} = 0.922, T_{max} = 0.938	$k = -12 \rightarrow 13$
6883 measured reflections	$l = -13 \rightarrow 13$

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0459P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.034	$(\Delta/\sigma)_{max}$ < 0.001
$wR(F^2)$ = 0.084	$\Delta\rho_{max}$ = 0.73 e Å^{-3}
S = 0.94	$\Delta\rho_{min}$ = −0.53 e Å^{-3}
2096 reflections	Extinction correction: none
128 parameters	
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	
Hydrogen site location: inferred from neighbouring sites	

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.

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}
Br1	0.59314 (4)	1.23977 (3)	0.17687 (4)	0.06274 (16)
C1	0.4366 (3)	1.1433 (3)	0.0801 (3)	0.0458 (7)
C2	0.3745 (3)	1.1446 (3)	−0.0481 (3)	0.0527 (8)
H2	0.4021	1.1968	−0.1042	0.063*
C3	0.2627 (3)	1.0568 (3)	−0.0855 (3)	0.0520 (7)
H3	0.2081	1.0445	−0.1704	0.062*
C4	0.2416 (3)	0.9918 (3)	0.0131 (3)	0.0432 (7)
C5	0.1400 (3)	0.8926 (3)	0.0063 (3)	0.0453 (7)
H5	0.0812	0.8673	−0.0737	0.054*
C6	0.0274 (3)	0.7426 (2)	0.0921 (3)	0.0401 (6)
C7	−0.0887 (3)	0.5910 (3)	0.1508 (3)	0.0568 (8)
H7	−0.1098	0.5375	0.2094	0.068*
C8	−0.1607 (3)	0.5841 (3)	0.0254 (3)	0.0462 (7)
C9	−0.2806 (4)	0.4988 (3)	−0.0419 (3)	0.0684 (9)
H9A	−0.2957	0.4395	0.0179	0.103*
H9B	−0.2541	0.4577	−0.1096	0.103*
H9C	−0.3692	0.5444	−0.0773	0.103*
N1	0.1279 (2)	0.8383 (2)	0.1063 (2)	0.0426 (5)
N2	0.0168 (3)	0.6793 (3)	0.1898 (2)	0.0547 (7)
S1	0.36114 (8)	1.03825 (6)	0.15710 (7)	0.0455 (2)
S2	−0.09293 (8)	0.69442 (7)	−0.05398 (7)	0.0467 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0652 (3)	0.0450 (2)	0.0753 (3)	−0.01064 (14)	0.0164 (2)	−0.00415 (16)
C1	0.0477 (17)	0.0379 (16)	0.0550 (19)	0.0027 (12)	0.0202 (15)	0.0012 (13)
C2	0.064 (2)	0.0484 (18)	0.0535 (19)	0.0008 (15)	0.0297 (17)	0.0066 (15)
C3	0.0604 (19)	0.0592 (19)	0.0378 (16)	−0.0002 (15)	0.0167 (14)	−0.0006 (14)
C4	0.0431 (16)	0.0445 (16)	0.0416 (15)	0.0041 (12)	0.0118 (13)	−0.0020 (13)
C5	0.0427 (16)	0.0500 (18)	0.0411 (17)	0.0009 (13)	0.0093 (14)	−0.0054 (14)
C6	0.0403 (15)	0.0451 (17)	0.0348 (15)	0.0055 (12)	0.0107 (12)	−0.0012 (12)

C7	0.0583 (19)	0.066 (2)	0.052 (2)	−0.0056 (16)	0.0244 (16)	0.0121 (16)
C8	0.0475 (16)	0.0448 (17)	0.0487 (18)	0.0023 (13)	0.0177 (15)	−0.0004 (13)
C9	0.068 (2)	0.062 (2)	0.074 (2)	−0.0127 (17)	0.0188 (19)	−0.0009 (18)
N1	0.0442 (13)	0.0475 (14)	0.0360 (13)	−0.0008 (10)	0.0115 (11)	−0.0016 (11)
N2	0.0578 (16)	0.0718 (18)	0.0350 (14)	−0.0071 (14)	0.0144 (12)	0.0068 (13)
S1	0.0506 (4)	0.0430 (4)	0.0410 (4)	−0.0008 (3)	0.0105 (3)	0.0022 (3)
S2	0.0559 (5)	0.0444 (4)	0.0360 (4)	−0.0034 (3)	0.0074 (3)	0.0011 (3)

Geometric parameters (Å, °)

Br1—C1	1.880 (3)	C6—N2	1.299 (4)
C1—C2	1.350 (4)	C6—N1	1.397 (3)
C1—S1	1.702 (3)	C6—S2	1.750 (3)
C2—C3	1.403 (4)	C7—C8	1.342 (4)
C2—H2	0.9300	C7—N2	1.370 (4)
C3—C4	1.353 (4)	C7—H7	0.9300
C3—H3	0.9300	C8—C9	1.491 (4)
C4—C5	1.442 (4)	C8—S2	1.718 (3)
C4—S1	1.725 (3)	C9—H9A	0.9600
C5—N1	1.277 (3)	C9—H9B	0.9600
C5—H5	0.9300	C9—H9C	0.9600
C2—C1—S1	113.4 (2)	N1—C6—S2	124.9 (2)
C2—C1—Br1	127.7 (2)	C8—C7—N2	117.8 (3)
S1—C1—Br1	118.84 (17)	C8—C7—H7	121.1
C1—C2—C3	111.1 (3)	N2—C7—H7	121.1
C1—C2—H2	124.5	C7—C8—C9	128.8 (3)
C3—C2—H2	124.5	C7—C8—S2	108.6 (2)
C4—C3—C2	113.9 (3)	C9—C8—S2	122.6 (2)
C4—C3—H3	123.1	C8—C9—H9A	109.5
C2—C3—H3	123.1	C8—C9—H9B	109.5
C3—C4—C5	127.5 (3)	H9A—C9—H9B	109.5
C3—C4—S1	110.9 (2)	C8—C9—H9C	109.5
C5—C4—S1	121.6 (2)	H9A—C9—H9C	109.5
N1—C5—C4	122.2 (3)	H9B—C9—H9C	109.5
N1—C5—H5	118.9	C5—N1—C6	119.0 (3)
C4—C5—H5	118.9	C6—N2—C7	110.3 (3)
N2—C6—N1	121.6 (3)	C1—S1—C4	90.63 (14)
N2—C6—S2	113.6 (2)	C8—S2—C6	89.72 (14)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5···S2	0.93	2.57	3.034 (3)	112
C5—H5···N2$^{\mathrm{i}}$	0.93	2.52	3.398 (4)	157

Symmetry codes: (i) *x*, −*y*+3/2, *z*−1/2.

Fig. 1