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N-[(5-Chlorothiophen-2-yl)methylene]-5-methylthiazol-2-amine

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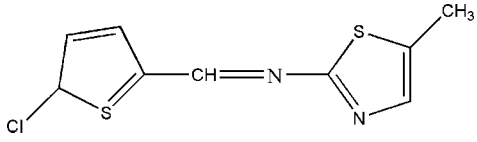
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.022; wR factor = 0.060; data-to-parameter ratio = 14.4.

The molecule of the title compound, a Schiff base, $C_9H_7ClN_2S_2$, is roughly planar, with the two rings twisted by only 8.8°. Molecules are interconnected by weak C—H···S interactions leading to the formation of chains parallel to the c axis. Weak slipped π–π stacking between the thiophene rings may help in further stabilizing the packing (centroid-to-centroid distance = 3.947 Å, interplanar distance = 3.651 Å and offset angle = 22.3°).

Related literature

For related literature, see: Alemi & Shaabani (2000); Alizadeh *et al.* (1999); Johnson *et al.* (1996); Kim & Shin (1999); Wang & Zheng (2007).



Experimental

Crystal data

$C_9H_7ClN_2S_2$
M_r = 242.74
Monoclinic, $P2_1$
a = 3.9472 (4) Å
b = 23.281 (2) Å
c = 6.0379 (6) Å
β = 104.214 (1)°
V = 537.87 (9) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.70 mm^{-1}
T = 298 (2) K
0.31 × 0.25 × 0.19 mm

Data collection

Bruker APEX area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.812, T_{max} = 0.878
3269 measured reflections
1839 independent reflections
1766 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.022
$wR(F^2)$ = 0.060
S = 1.06
1839 reflections
128 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}
Absolute structure: Flack (1983), with 836 Friedel pairs
Flack parameter: 0.04 (6)

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

D—H···A	D—H	H···A	D···A	D—H···A
C5—H5···S2^{i}	0.93	2.97	3.834 (2)	155

Symmetry code: (i) $x, y, z-1$.

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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2206).

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supplementary materials

Acta Cryst. (2007). E**63**, o3310 [**doi:10.1107/S1600536807030255**]

N-[(5-Chlorothiophen-2-yl)methylene]-5-methylthiazol-2-amine

C.-N. Zhang and Y.-F. Zheng

Comment

Schiff base ligands have significant importance in chemistry, especially in the development of Schiff base complexes, (Johnson *et al.*, 1996; Alizadeh *et al.*,1999; Wang & Zheng, 2007). Schiff bases that have solvent-dependent UV/vis spectra (solvatochromicity) can be suitable NLO (nonlinear optically active) materials (Alemi & Shaabani, 2000). They are also useful in the asymmetric oxidation of methyl phenyl sulfide and are enantioselective (Kim & Shin, 1999). In this paper, we report the synthesis and crystal structure of the title compound, (I).

The title compound is roughly planar with the two thiophene rings twisted by only (Fig. 1). The bond lengths and bond angles are usual for such compounds. The crystal packing is governed by weak C—H···S interactions (Table 1) forming chains parallel to the *c* axis and very weak slipped π–π stacking between the thiophene rings with centroid to centroid distance of 3.947 Å and interplanar distance of 3.651 Å resulting in an offset angle of 22.3°.

Experimental

Under nitrogen, a mixture of 5-chlorothiophene-2-carbaldehyde (1.67 g,10 mmol), Na_2SO_4 (3.0 g) and 5-methylthiazol-2-amine (1.58 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 *x* 15 ml) and brine (8 ml). After drying over Na_2SO_4, the solvent was removed under vacuum, and a yellow solid was isolated in 92% yield (3.1 g). Colourless single crystals of the Schiff base, (I), suitable for X-ray analysis were grown from CH_2Cl_2 and absolute ethanol (4:1) by slow evaporation of the solvents at room temperature over a period of about one week.

Refinement

All H atoms were fixed geometrically and treated as riding with C—H = 0.93 Å (aromatic) or 0.98 Å (methyl) with U_{iso}(H) = xU_{eq}(C) where *x* = 1.2 for aromatic H and 1.5 for methyl H.

Figures

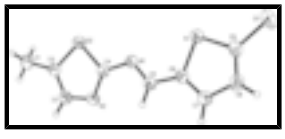

Fig. 1. The molecular structure of (I), showing the atomic numbering scheme. Probability displacement ellipsoids are drawn at the 30% level.

N-[(5-Chlorothiophen-2-yl)methylene]-5-methylthiazol-2-amine

Crystal data

$C_9H_7ClN_2S_2$	F_{000} = 248
M_r = 242.74	D_x = 1.499 Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2yb	Cell parameters from 1839 reflections
a = 3.9472 (4) Å	θ = 3.5–25.2º
b = 23.281 (2) Å	μ = 0.70 mm^{-1}
c = 6.0379 (6) Å	T = 298 (2) K
β = 104.214 (1)º	Block, colourless
V = 537.87 (9) Å^3	0.31 × 0.25 × 0.19 mm
Z = 2	

Data collection

Bruker APEX area-detector diffractometer	1839 independent reflections
Radiation source: fine-focus sealed tube	1766 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.027
T = 298(2) K	θ_{max} = 25.2º
φ and ω scans	θ_{min} = 3.5º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −4→4
T_{min} = 0.812, T_{max} = 0.878	k = −24→27
3269 measured reflections	l = −7→7

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.022	$w = 1/[\sigma^2(F_o^2) + (0.0362P)^2 + 0.0225P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.060	$(\Delta/\sigma)_{max}$ = 0.001
S = 1.06	$\Delta\rho_{max}$ = 0.18 e Å^{-3}
1839 reflections	$\Delta\rho_{min}$ = −0.12 e Å^{-3}
128 parameters	Extinction correction: none
1 restraint	Absolute structure: Flack (1983), with 836 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.04 (6)
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 $F^2 > 2\text{sigma}(F^2)$ 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}
C1	1.4257 (6)	0.94947 (11)	1.1100 (4)	0.0662 (6)
H1A	1.5326	0.9797	1.0429	0.099*
H1B	1.5992	0.9311	1.2282	0.099*
H1C	1.2463	0.9653	1.1742	0.099*
C2	1.2681 (5)	0.90636 (10)	0.9303 (4)	0.0501 (4)
C3	1.2361 (6)	0.90871 (10)	0.7032 (4)	0.0564 (5)
H3	1.3129	0.9405	0.6359	0.068*
C4	1.0034 (5)	0.82366 (9)	0.7061 (3)	0.0469 (4)
C5	0.7404 (5)	0.75947 (9)	0.4337 (4)	0.0491 (4)
H5	0.7667	0.7873	0.3286	0.059*
C6	0.5772 (5)	0.70562 (9)	0.3507 (4)	0.0486 (5)
C7	0.4437 (7)	0.68890 (11)	0.1310 (4)	0.0634 (6)
H7	0.4465	0.7120	0.0059	0.076*
C8	0.3010 (6)	0.63335 (11)	0.1111 (4)	0.0657 (6)
H8	0.1995	0.6156	−0.0274	0.079*
C9	0.3287 (5)	0.60889 (9)	0.3165 (4)	0.0542 (5)
Cl1	0.18698 (18)	0.54193 (3)	0.36876 (14)	0.0784 (2)
N1	0.8487 (4)	0.77019 (7)	0.6444 (3)	0.0496 (4)
N2	1.0867 (5)	0.86277 (8)	0.5751 (3)	0.0559 (4)
S1	0.52770 (13)	0.65240 (2)	0.53993 (8)	0.05352 (14)
S2	1.10583 (13)	0.84148 (2)	0.99287 (8)	0.05410 (15)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0655 (13)	0.0622 (16)	0.0671 (14)	−0.0105 (11)	0.0090 (11)	−0.0118 (11)
C2	0.0461 (9)	0.0458 (11)	0.0565 (11)	0.0005 (9)	0.0092 (8)	−0.0018 (9)
C3	0.0657 (12)	0.0436 (11)	0.0586 (12)	−0.0054 (10)	0.0127 (10)	0.0040 (9)
C4	0.0496 (10)	0.0436 (11)	0.0476 (10)	0.0025 (8)	0.0120 (8)	0.0017 (8)
C5	0.0520 (10)	0.0441 (11)	0.0520 (11)	0.0007 (8)	0.0139 (8)	0.0065 (8)
C6	0.0501 (10)	0.0447 (12)	0.0503 (11)	0.0024 (8)	0.0113 (8)	0.0041 (8)
C7	0.0821 (14)	0.0542 (14)	0.0505 (12)	−0.0005 (11)	0.0095 (11)	0.0023 (10)

C8	0.0766 (15)	0.0574 (15)	0.0548 (12)	−0.0005 (10)	0.0001 (10)	−0.0081 (10)
C9	0.0478 (10)	0.0431 (11)	0.0702 (13)	0.0012 (8)	0.0118 (9)	−0.0054 (9)
Cl1	0.0752 (4)	0.0445 (3)	0.1166 (5)	−0.0065 (3)	0.0253 (3)	0.0009 (3)
N1	0.0539 (9)	0.0418 (10)	0.0522 (10)	−0.0004 (7)	0.0114 (7)	0.0020 (7)
N2	0.0713 (11)	0.0466 (11)	0.0492 (9)	−0.0048 (8)	0.0134 (8)	0.0021 (7)
S1	0.0612 (3)	0.0480 (3)	0.0502 (3)	−0.0033 (2)	0.0115 (2)	0.0039 (2)
S2	0.0625 (3)	0.0525 (3)	0.0464 (2)	−0.0058 (2)	0.0117 (2)	0.0024 (2)

Geometric parameters (Å, °)

C1—C2	1.496 (3)	C5—N1	1.264 (3)
C1—H1A	0.9600	C5—C6	1.442 (3)
C1—H1B	0.9600	C5—H5	0.9300
C1—H1C	0.9600	C6—C7	1.358 (3)
C2—C3	1.347 (3)	C6—S1	1.729 (2)
C2—S2	1.719 (2)	C7—C8	1.404 (4)
C3—N2	1.366 (3)	C7—H7	0.9300
C3—H3	0.9300	C8—C9	1.345 (3)
C4—N2	1.300 (3)	C8—H8	0.9300
C4—N1	1.396 (3)	C9—Cl1	1.712 (2)
C4—S2	1.729 (2)	C9—S1	1.715 (2)
C2—C1—H1A	109.5	C6—C5—H5	118.9
C2—C1—H1B	109.5	C7—C6—C5	128.4 (2)
H1A—C1—H1B	109.5	C7—C6—S1	111.11 (17)
C2—C1—H1C	109.5	C5—C6—S1	120.45 (16)
H1A—C1—H1C	109.5	C6—C7—C8	113.5 (2)
H1B—C1—H1C	109.5	C6—C7—H7	123.2
C3—C2—C1	129.0 (2)	C8—C7—H7	123.2
C3—C2—S2	108.21 (16)	C9—C8—C7	111.8 (2)
C1—C2—S2	122.77 (17)	C9—C8—H8	124.1
C2—C3—N2	117.6 (2)	C7—C8—H8	124.1
C2—C3—H3	121.2	C8—C9—Cl1	126.84 (18)
N2—C3—H3	121.2	C8—C9—S1	113.16 (17)
N2—C4—N1	128.43 (19)	Cl1—C9—S1	120.00 (14)
N2—C4—S2	114.16 (15)	C5—N1—C4	117.54 (17)
N1—C4—S2	117.41 (14)	C4—N2—C3	110.04 (18)
N1—C5—C6	122.24 (18)	C9—S1—C6	90.44 (11)
N1—C5—H5	118.9	C2—S2—C4	89.94 (10)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5···S2^{i}	0.93	2.97	3.834 (2)	155

Symmetry codes: (i) *x*, *y*, *z*−1.

Fig. 1