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N-[(*E*)-(5-Methylthiophen-2-yl)methylidene]-1*H*-1,2,4-triazol-3-amine

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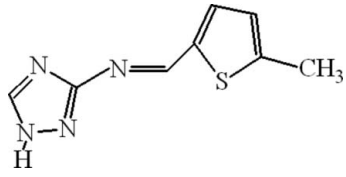
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.034; wR factor = 0.097; data-to-parameter ratio = 16.5.

In the title Schiff base, $C_8H_8N_4S$, a condensation product of 5-methylthiophene-2-carboxaldehyde and 3-amino-1,2,4-triazole, the dihedral angle between the triazolyl and thienyl rings is 6.44 (14)°. The compound exists as a polymeric chain arising from intermolecular N—H···N bonding.

Related literature

For a related comound, see: Chohan *et al.* (2009). For the biological properties of such compounds, see: Foroumadi *et al.* (2003); Manfredini *et al.* (2000).



Experimental

Crystal data

$C_8H_8N_4S$
M_r = 192.24
Orthorhombic, $P2_12_12_1$
a = 7.2570 (7) Å
b = 8.9522 (8) Å
c = 14.2930 (15) Å
V = 928.56 (16) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.31 mm^{-1}
T = 296 (2) K
0.24 × 0.16 × 0.14 mm

Data collection

Bruker KAPPA APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.928, T_{max} = 0.956
5793 measured reflections
2206 independent reflections
1859 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.097
S = 1.05
2206 reflections
134 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}
Absolute structure: Flack (1983), 854 Friedel pairs
Flack parameter: −0.02 (10)

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3n···N1^{i}	0.85 (3)	2.12 (3)	2.963 (2)	172 (2)

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *WinGX* publication routines (Farrugia, 1999) and *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2524).

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supporting information

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N-[(*E*)-(5-Methylthiophen-2-yl)methylidene]-1*H*-1,2,4-triazol-3-amine

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S1. Comment

Compounds derived from triazole possess antimicrobial, analgesic, anti-inflammatory, local anesthetic, antineoplastic and antimalarial properties (Foroumadi *et al.*, 2003). Some triazole Schiff bases also exhibited antiproliferative and anticancer activity (Manfredini *et al.*, 2000). Due to their significant biological applications they have gained much attention in bioinorganic and metal-based drug discovery. In view of its structural and biological importance, we have synthesized (Chohan *et al.*, 2009), series of triazole derived Schiff bases along with the title compound (I). We report herein, its preperation and crystal structure.

In the molecule of title compound, (Fig 1), the bond lengths and angles are within normal ranges. In this molecule 5-methylthiophen ring is attached to 5-membered ring of triazole moiety through the Schiff bond C=N. The dihedral angle between ring A(S1/C1—C4) and B(C7/N2/N3/C8/N4) is 6.44 (14)°. There exist intramolecular as well as an intermolecular H-bonds as given in Table 1. The molecules are connected to each other through intermolecular H-bonds of N–H···N type in a helical way (Fig 2).

S2. Experimental

A mixture of 5-methylthiophene-2-carboxaldehyde (1.09 ml, 0.01 *M*) and 3-amino-1,2,4-triazole (0.84 g, 0.01 *M*) in 1:1 molar proportions in methanol (40 ml) was boiled under reflux for 5 h by monitoring through TLC. The reaction mixture was cooled at room temperature and filtered; within an hour a light brown solid product separated from the clear solution. It was filtered, washed with methanol, dried and recrystallized from a mixture of ethanol:methanol (1:1).

S3. Refinement

H-atoms were positioned geometrically, with C—H = 0.96 Å for methyl carbon of thiophene ring and constrained to ride on the parent atom. The coordinates of all other H-atoms were refined. The U_{iso}(H) = xU_{eq}(C, N), where x = 1.5 for methyl H and x = 1.2 for all other H atoms.

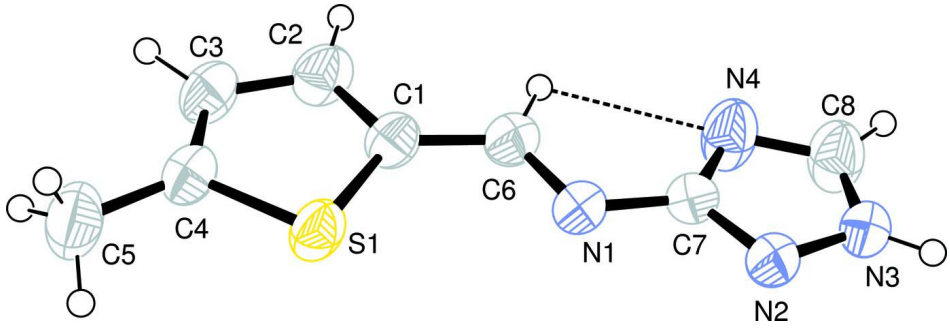

Figure 1

ORTEP-3 for Windows (Farrugia, 1997) drawing of the title compound, $C_8H_8N_4S$, with the atom numbering scheme. The thermal ellipsoids are drawn at the 50% probability level. H-atoms are shown by small circles of arbitrary radii. The intramolecular H-bonding is shown by dashed lines.

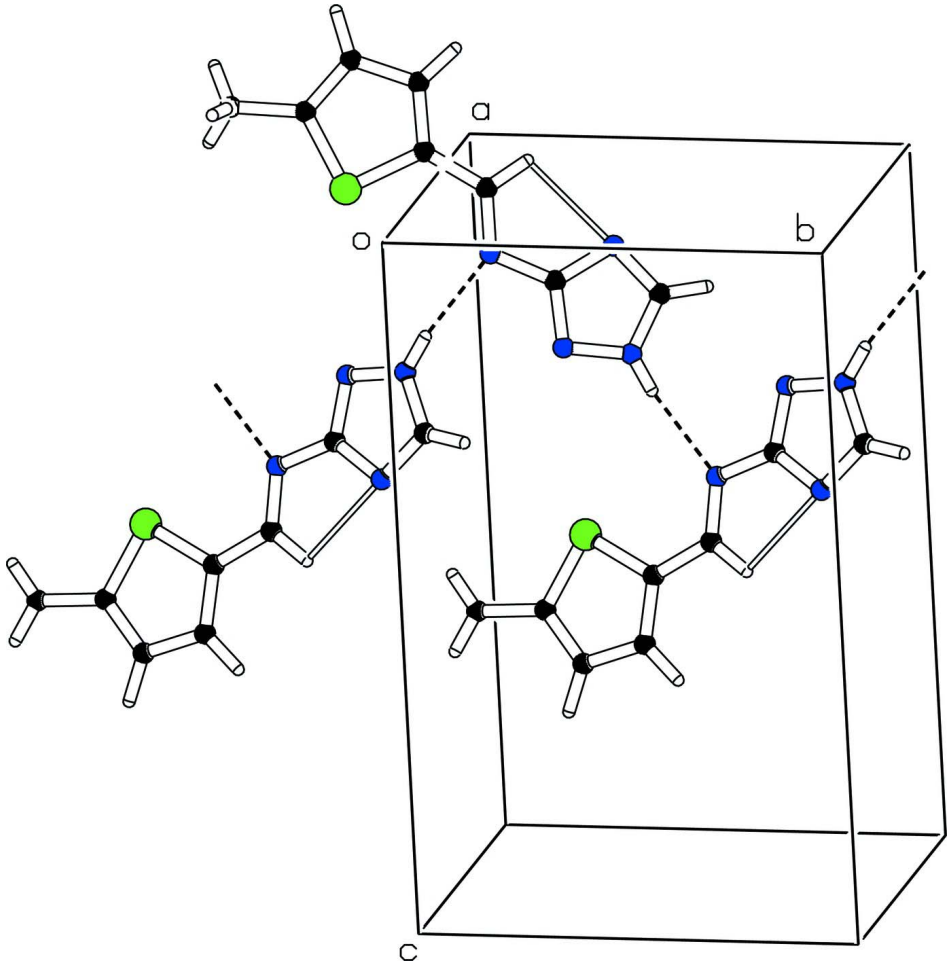

Figure 2

The partial unit cell packing of (I) (Spek, 2003) showing the interamolecular and intermolecular hydrogen bonding showing that polymeric sheets are formed.

(I)

Crystal data

$C_8H_8N_4S$
$M_r = 192.24$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 7.2570$ (7) Å
$b = 8.9522$ (8) Å
$c = 14.2930$ (15) Å
$V = 928.56$ (16) Å^3
$Z = 4$
$F(000) = 400$
$D_x = 1.375$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2206 reflections
$\theta = 2.7$–$28.3°$
$\mu = 0.31$ mm^{-1}
$T = 296$ K
Prismatic, light brown
0.24 × 0.16 × 0.14 mm

Data collection

Bruker KAPPA APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 7.4 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min} = 0.928$, $T_{max} = 0.956$
5793 measured reflections
2206 independent reflections
1859 reflections with $I > 2\sigma(I)$
$R_{int} = 0.034$
$\theta_{max} = 28.3°$, $\theta_{min} = 2.7°$
$h = -9 \rightarrow 9$
$k = -11 \rightarrow 11$
$l = -17 \rightarrow 19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.097$
$S = 1.05$
2206 reflections
134 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0562P)^2 + 0.0383P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}
Absolute structure: Flack (1983), 854 Friedel pairs
Absolute structure parameter: −0.02 (10)

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 e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

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}
S1	0.48892 (7)	−0.18020 (5)	0.00175 (3)	0.0512 (2)
N1	0.5269 (2)	0.13362 (16)	0.09627 (10)	0.0456 (4)
N2	0.4898 (2)	0.29617 (16)	0.22417 (10)	0.0498 (5)

N3	0.5397 (2)	0.43946 (17)	0.23930 (11)	0.0502 (5)
N4	0.6332 (3)	0.39225 (19)	0.09806 (13)	0.0659 (7)
C1	0.5665 (3)	−0.0136 (2)	−0.04286 (12)	0.0460 (5)
C2	0.6222 (3)	−0.0303 (3)	−0.13344 (14)	0.0565 (7)
C3	0.6008 (3)	−0.1767 (3)	−0.16735 (14)	0.0562 (7)
C4	0.5306 (3)	−0.2720 (2)	−0.10199 (12)	0.0497 (5)
C5	0.4937 (4)	−0.4350 (3)	−0.11161 (16)	0.0737 (9)
C6	0.5724 (3)	0.1226 (2)	0.00957 (13)	0.0473 (5)
C7	0.5491 (3)	0.27297 (19)	0.13780 (12)	0.0432 (5)
C8	0.6228 (3)	0.4940 (3)	0.16478 (17)	0.0654 (8)
H2	0.663 (3)	0.048 (2)	−0.1784 (19)	0.0678*
H3	0.629 (3)	−0.210 (3)	−0.2327 (18)	0.0675*
H3N	0.515 (3)	0.488 (3)	0.2891 (17)	0.0602*
H5A	0.52067	−0.48427	−0.05354	0.1103*
H5B	0.57023	−0.47560	−0.16015	0.1103*
H5C	0.36648	−0.45026	−0.12734	0.1103*
H6	0.621 (3)	0.209 (2)	−0.0237 (15)	0.0567*
H8	0.671 (4)	0.595 (2)	0.1599 (17)	0.0785*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0642 (3)	0.0540 (3)	0.0354 (2)	−0.0070 (2)	0.0078 (2)	−0.0047 (2)
N1	0.0516 (8)	0.0465 (7)	0.0386 (7)	0.0015 (7)	−0.0023 (7)	−0.0011 (6)
N2	0.0668 (10)	0.0434 (7)	0.0392 (7)	0.0024 (7)	0.0000 (8)	0.0002 (5)
N3	0.0644 (10)	0.0439 (8)	0.0422 (8)	0.0024 (7)	−0.0020 (8)	−0.0053 (6)
N4	0.0895 (14)	0.0554 (10)	0.0529 (10)	−0.0181 (9)	0.0184 (10)	−0.0072 (8)
C1	0.0475 (9)	0.0518 (10)	0.0388 (8)	−0.0018 (7)	0.0027 (8)	−0.0017 (7)
C2	0.0641 (13)	0.0637 (13)	0.0418 (10)	−0.0064 (9)	0.0110 (9)	0.0012 (9)
C3	0.0605 (12)	0.0706 (13)	0.0376 (9)	0.0014 (10)	0.0098 (9)	−0.0096 (9)
C4	0.0515 (10)	0.0573 (10)	0.0404 (8)	−0.0008 (8)	0.0011 (8)	−0.0092 (7)
C5	0.100 (2)	0.0629 (12)	0.0582 (12)	−0.0099 (13)	0.0055 (13)	−0.0160 (10)
C6	0.0521 (9)	0.0499 (9)	0.0398 (8)	−0.0004 (8)	−0.0003 (8)	0.0008 (7)
C7	0.0475 (9)	0.0431 (8)	0.0391 (8)	0.0031 (7)	−0.0022 (8)	0.0003 (7)
C8	0.0827 (16)	0.0504 (11)	0.0630 (13)	−0.0152 (11)	0.0126 (12)	−0.0079 (10)

Geometric parameters (Å, º)

S1—C1	1.7169 (19)	C1—C6	1.432 (3)
S1—C4	1.7220 (18)	C2—C3	1.406 (4)
N1—C6	1.286 (2)	C3—C4	1.364 (3)
N1—C7	1.391 (2)	C4—C5	1.490 (3)
N2—N3	1.350 (2)	C2—H2	1.00 (2)
N2—C7	1.324 (2)	C3—H3	1.00 (3)
N3—C8	1.318 (3)	C5—H5A	0.9600
N4—C7	1.355 (3)	C5—H5B	0.9600
N4—C8	1.321 (3)	C5—H5C	0.9600
N3—H3N	0.85 (3)	C6—H6	0.974 (19)

C1—C2	1.365 (3)	C8—H8	0.97 (2)
S1···N1	3.1295 (15)	N4···H5A^{vii}	2.5700
S1···C4^{i}	3.647 (2)	N4···H6	2.39 (2)
N1···S1	3.1295 (15)	C1···N4^{v}	3.419 (3)
N1···N3^{ii}	2.963 (2)	C4···S1^{viii}	3.647 (2)
N2···N4	2.252 (2)	C8···N2^{iii}	3.241 (3)
N2···C8^{ii}	3.241 (3)	C7···H3^{vi}	3.03 (2)
N2···N3^{ii}	3.243 (2)	C7···H3N^{ii}	2.80 (3)
N3···N1^{iii}	2.963 (2)	H2···N2^{iv}	2.83 (2)
N3···N4	2.171 (2)	H2···N3^{iv}	2.87 (2)
N3···N2^{iii}	3.243 (2)	H3···N2^{ix}	2.94 (2)
N4···C1^{iv}	3.419 (3)	H3···C7^{ix}	3.03 (2)
N4···N3	2.171 (2)	H3N···N1^{iii}	2.12 (3)
N1···H3N^{ii}	2.12 (3)	H3N···N2^{iii}	2.77 (3)
N2···H8^{ii}	2.71 (2)	H3N···C7^{iii}	2.80 (3)
N2···H2^{v}	2.83 (2)	H5A···N4^{x}	2.5700
N2···H3^{vi}	2.94 (2)	H6···N4	2.39 (2)
N2···H3N^{ii}	2.77 (3)	H8···N2^{iii}	2.71 (2)
N3···H2^{v}	2.87 (2)		
C1—S1—C4	92.13 (9)	N1—C7—N4	125.46 (16)
C6—N1—C7	116.76 (15)	N2—C7—N4	114.44 (16)
N3—N2—C7	102.19 (14)	N3—C8—N4	110.7 (2)
N2—N3—C8	110.20 (17)	C1—C2—H2	128.6 (14)
C7—N4—C8	102.42 (18)	C3—C2—H2	117.8 (14)
C8—N3—H3N	125.6 (17)	C2—C3—H3	125.2 (15)
N2—N3—H3N	124.1 (17)	C4—C3—H3	121.9 (15)
S1—C1—C6	123.72 (14)	C4—C5—H5A	109.00
C2—C1—C6	125.52 (19)	C4—C5—H5B	109.00
S1—C1—C2	110.75 (16)	C4—C5—H5C	110.00
C1—C2—C3	113.4 (2)	H5A—C5—H5B	109.00
C2—C3—C4	112.85 (18)	H5A—C5—H5C	109.00
S1—C4—C3	110.91 (15)	H5B—C5—H5C	109.00
C3—C4—C5	128.07 (19)	N1—C6—H6	120.2 (12)
S1—C4—C5	121.02 (15)	C1—C6—H6	115.6 (12)
N1—C6—C1	124.19 (17)	N3—C8—H8	124.6 (15)
N1—C7—N2	120.07 (16)	N4—C8—H8	124.7 (15)
C1—S1—C4—C3	−0.12 (18)	C8—N4—C7—N2	−0.4 (3)
C4—S1—C1—C2	−0.28 (18)	C7—N4—C8—N3	0.4 (2)
C4—S1—C1—C6	−179.69 (19)	C8—N4—C7—N1	−178.6 (2)
C1—S1—C4—C5	178.8 (2)	C2—C1—C6—N1	−176.6 (2)
C6—N1—C7—N4	−7.9 (3)	S1—C1—C2—C3	0.6 (2)
C7—N1—C6—C1	177.03 (19)	C6—C1—C2—C3	−180.0 (2)
C6—N1—C7—N2	173.91 (18)	S1—C1—C6—N1	2.7 (3)
C7—N2—N3—C8	0.1 (2)	C1—C2—C3—C4	−0.7 (3)
N3—N2—C7—N1	178.54 (17)	C2—C3—C4—S1	0.5 (2)

N3—N2—C7—N4	0.2 (2)	C2—C3—C4—C5	−178.4 (2)
N2—N3—C8—N4	−0.3 (2)		

Symmetry codes: (i) *x*−1/2, −*y*−1/2, −*z*; (ii) −*x*+1, *y*−1/2, −*z*+1/2; (iii) −*x*+1, *y*+1/2, −*z*+1/2; (iv) *x*+1/2, −*y*+1/2, −*z*; (v) *x*−1/2, −*y*+1/2, −*z*; (vi) −*x*+3/2, −*y*, *z*+1/2; (vii) *x*, *y*+1, *z*; (viii) *x*+1/2, −*y*−1/2, −*z*; (ix) −*x*+3/2, −*y*, *z*−1/2; (x) *x*, *y*−1, *z*.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3n···N1^{iii}	0.85 (3)	2.12 (3)	2.963 (2)	172 (2)
C6—H6···N4	0.974 (19)	2.39 (2)	2.761 (3)	101.7 (15)

Symmetry code: (iii) −*x*+1, *y*+1/2, −*z*+1/2.