Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N-(2-Thienylmethylene)-2-(2-{[2-(2-thienylmethyleneamino)phenyl]-sulfanyl}ethylsulfanyl)aniline

Ali Kakanejadifard[a]* and Vahid Amani[b]

[a]Department of Chemistry, Faculty of Science, Lorestan University, Khorramabad, Iran, and [b]Department of Chemistry, Islamic Azad University, Shahr-e-Rey Branch, Tehran, Iran
Correspondence e-mail: alikakanejadifard@yahoo.com

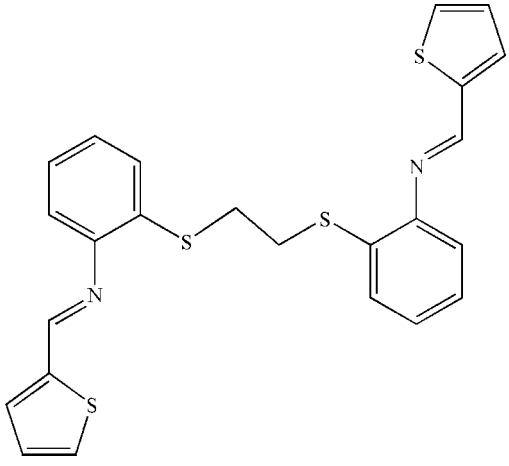
Received 18 June 2009; accepted 24 June 2009

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.029; wR factor = 0.079; data-to-parameter ratio = 21.3.

The asymmetric unit of the title compound, $C_{24}H_{20}N_2S_4$, contains one half-molecule: a crystallographic centre of inversion is located at the mid-point of the two central C atoms. The thiophene ring is oriented at a dihedral angle of 60.64 (3)° with respect to the benzene ring. In the crystal structure, π–π contacts between thiophene rings [centroid–centroid distance = 3.581 (1) Å] may stabilize the structure. A weak C—H$\cdots\pi$ interaction is also present.

Related literature

For related structures, see: Dharaa *et al.* (2005); Gok & Demirbas (1989); Kakanejadifard *et al.* (2007); Kakanejadifard & Amani (2008); Morshedi *et al.* (2009); Rajsekhar *et al.* (2002, 2004); Taylor *et al.* (2008). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{24}H_{20}N_2S_4$
M_r = 464.66
Monoclinic, $P2_1/c$
a = 11.179 (5) Å
b = 7.730 (4) Å
c = 12.608 (6) Å
β = 91.899 (12)°
V = 1088.9 (9) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.45 mm^{-1}
T = 100 K
0.30 × 0.20 × 0.15 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.895, T_{max} = 0.930
12880 measured reflections
2899 independent reflections
2569 reflections with $I > 2/s(I)$
R_{int} = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.079
S = 1.00
2899 reflections
136 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C10—H10$A\cdots Cg1^{i}$	0.95	2.80	3.740 (3)	171

Symmetry code: (i) $x, -y-\frac{1}{2}, z-\frac{1}{2}$. $Cg1$ is centroid of the ring C2–C7 ring.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL* software used to prepare material for publication: *SHELXTL*.

We are grateful to the Research Grant Council of Lorestan University for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2716).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bruker (2005). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Dharaa, P. K., Dasa, B., Lo, J. M. & Chattopadhyay, P. (2005). *Appl. Radiat. Isot.* **62**, 729–735.
Gok, Y. & Demirbas, A. (1989). *Synth. React. Inorg. Met.-Org. Chem.* **19**, 681–698.
Kakanejadifard, A. & Amani, V. (2008). *Acta Cryst.* E**64**, o1512.
Kakanejadifard, A., Sharifi, S., Delfani, F., Ranjbar, B. & Naderimanesh, H. (2007). *Iran. J. Chem. Chem. Eng.* **26**, 63–67.
Morshedi, M., Amirnasr, M., Slawin, A. M. Z., Woollins, J. D. & Dehno Khalaji, A. K. (2009). *Polyhedron*, **28**, 167–171.
Rajsekhar, G., Rao, C. P., Saarenketo, P. K., Kolehmainen, E. & Rissanen, K. (2002). *Inorg. Chem. Commun.* **5**, 649–652.
Rajsekhar, G., Rao, C. P., Saarenketo, P., Nattinen, K. & Rissanen, K. (2004). *New J. Chem.* **28**, 75–84.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Taylor, M. K., Trotter, K. D., Reglinski, J., Berlouis, L. E. A., Kennedy, A. R., Spickett, C. M. & Sowden, R. J. (2008). *Inorg. Chim. Acta*, **361**, 2851–2862.

supporting information

Acta Cryst. (2009). E**65**, o1712 [doi:10.1107/S1600536809024404]

N-(2-Thienylmethylene)-2-(2-{[2-(2-thienylmethyleneamino)phenyl]-sulfanyl}ethylsulfanyl)aniline

Ali Kakanejadifard and Vahid Amani

S1. Comment

There are several examples of N_2S_2 Schiff bases type of adducts which exist as anti configuration. For 2-[2-(2-amino-phenylthio)benzeneamine] adduct see: (Gok & Demirbas, 1989; Dharaa *et al.*, 2005; Kakanejadifard *et al.*, 2007; Kakanejadifard & Amani, 2008). For N_2S_2 Schiff bases adduct see: (Rajsekhar *et al.*, 2002; Taylor *et al.*, 2008; Morshedi *et al.*, 2009). For $N_2O_2S_2$ Schiff bases adduct see: (Rajsekhar *et al.*, 2004). We report herein the synthesis and crystal structure of the title compound.

The asymmetric unit of the title compound, (Fig. 1), contains one-half molecule. A crystallographic centre of inversion is located at the midpoint between the two central C atoms. The bond lengths (Allen *et al.*, 1987) and angles are within normal ranges. Rings A (C2-C7) and B (S2/C9-C12) are, of course, planar and they are oriented at a dihedral angle of 60.64 (3)°.

In the crystal structure, the π–π contact between the thiophene rings, Cg2—Cg2^{i}, [symmetry code: (i) -x, -y, 1 - z, where Cg2 is centroid of the ring B (S2/C9-C12)] may stabilize the structure, with centroid-centroid distance of 3.581 (1) Å. There also exits a weak C—H···π interaction (Table 1).

S2. Experimental

For the preparation of the title compound, a solution of thiophencarbaldehyde (20 mmol) was added dropwise to a solution of 2-[2-(2-aminophenylthio)benzeneamine] (2.76 g, 10 mmol) in absolute ethanol (25 ml) with stirring in 10 min at room temperature. The mixture was stirred and heated to reflux for 5 h. The product was filtered and crystallized from CH_3CN (yield; 45%, m.p. 398-399 K).

S3. Refinement

H atoms were positioned geometrically with C-H = 0.95 and 0.99 Å for aromatic and methylene H atoms, respectively, and constrained to ride on their parent atoms, with U_{iso}(H) = *1.2*U_{eq}(C).

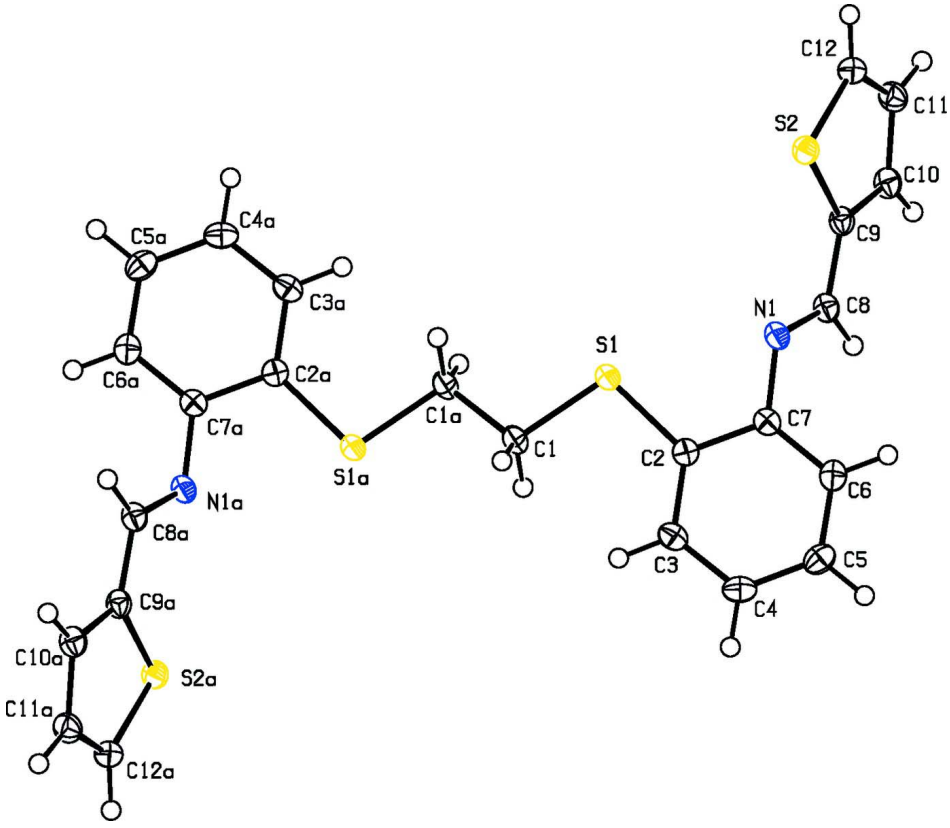


Figure 1

The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level [symmetry code: (a) 1 - x, -y, 2 -z].

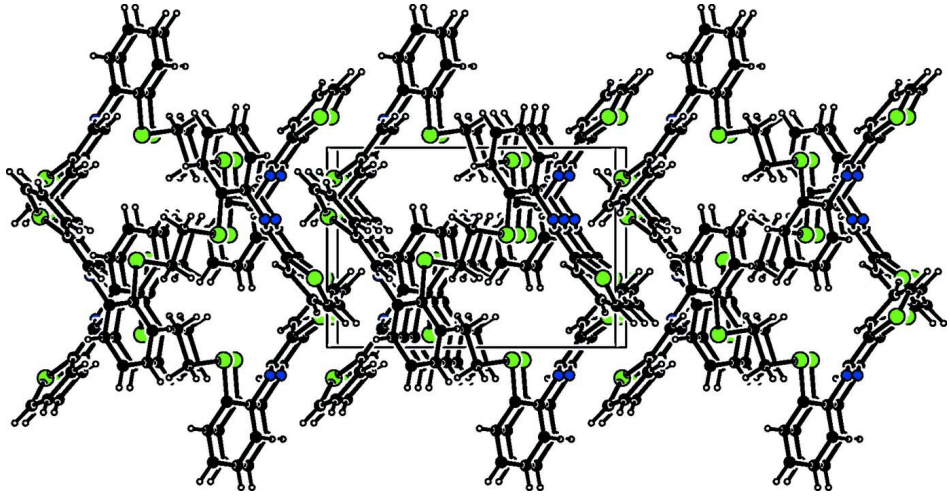


Figure 2

A partial packing diagram.

N-(2-Thienylmethylene)-2-(2-{[2-(2- thienylmethyleneamino)phenyl]sulfanyl}ethylsulfanyl)aniline

Crystal data

$C_{24}H_{20}N_2S_4$
$M_r = 464.66$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 11.179$ (5) Å
$b = 7.730$ (4) Å
$c = 12.608$ (6) Å
$\beta = 91.899$ (12)°
$V = 1088.9$ (9) Å^3
$Z = 2$
$F(000) = 484$
$D_x = 1.417$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 887 reflections
$\theta = 3$–30°
$\mu = 0.45$ mm^{-1}
$T = 100$ K
Prism, yellow
0.30 × 0.20 × 0.15 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min} = 0.895$, $T_{max} = 0.930$
12880 measured reflections
2899 independent reflections
2569 reflections with $I > 2/s(I)$
$R_{int} = 0.029$
$\theta_{max} = 29.0°$, $\theta_{min} = 1.8°$
$h = -15 \rightarrow 15$
$k = -10 \rightarrow 10$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.079$
$S = 1.00$
2899 reflections
136 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
$w = 1/[\sigma^2(F_o^2) + (0.0441P)^2 + 0.43P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.34$ e Å^{-3}
$\Delta\rho_{min} = -0.26$ e Å^{-3}

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.63766 (3)	−0.06436 (4)	0.88963 (2)	0.01981 (9)
S2	0.96427 (3)	0.15452 (4)	0.67870 (2)	0.01990 (9)
N1	0.79244 (9)	−0.14058 (14)	0.72259 (8)	0.0188 (2)
C1	0.51924 (11)	−0.09011 (16)	0.98300 (10)	0.0199 (2)

H1A	0.4507	−0.1525	0.9492	0.024*
H1B	0.5485	−0.1574	1.0454	0.024*
C2	0.64682 (11)	−0.27273 (16)	0.83345 (10)	0.0178 (2)
C3	0.58160 (11)	−0.41715 (17)	0.86395 (10)	0.0212 (3)
H3A	0.5258	−0.4071	0.9188	0.025*
C4	0.59782 (12)	−0.57566 (17)	0.81445 (11)	0.0232 (3)
H4A	0.5528	−0.6732	0.8357	0.028*
C5	0.67894 (12)	−0.59283 (18)	0.73454 (11)	0.0238 (3)
H5A	0.6912	−0.7024	0.7025	0.029*
C6	0.74233 (12)	−0.44926 (17)	0.70128 (10)	0.0216 (3)
H6A	0.7970	−0.4605	0.6456	0.026*
C7	0.72613 (10)	−0.28888 (16)	0.74913 (9)	0.0180 (2)
C8	0.79459 (11)	−0.09360 (17)	0.62510 (10)	0.0196 (2)
H8A	0.7488	−0.1565	0.5733	0.024*
C9	0.86458 (11)	0.05200 (17)	0.59218 (10)	0.0188 (2)
C10	0.86686 (12)	0.12347 (18)	0.49235 (10)	0.0213 (3)
H10A	0.8185	0.0843	0.4338	0.026*
C11	0.94924 (12)	0.26160 (18)	0.48653 (10)	0.0234 (3)
H11A	0.9619	0.3261	0.4237	0.028*
C12	1.00852 (12)	0.29220 (17)	0.58100 (10)	0.0223 (3)
H12B	1.0675	0.3797	0.5914	0.027*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.01936 (16)	0.01889 (16)	0.02154 (16)	−0.00077 (11)	0.00610 (11)	−0.00011 (11)
S2	0.02172 (16)	0.02322 (17)	0.01469 (15)	−0.00207 (11)	−0.00047 (11)	−0.00144 (11)
N1	0.0163 (5)	0.0226 (5)	0.0174 (5)	−0.0002 (4)	0.0025 (4)	−0.0003 (4)
C1	0.0180 (6)	0.0221 (6)	0.0199 (6)	0.0015 (5)	0.0049 (4)	0.0027 (5)
C2	0.0168 (5)	0.0188 (6)	0.0178 (5)	0.0017 (4)	−0.0008 (4)	0.0010 (4)
C3	0.0190 (6)	0.0226 (6)	0.0221 (6)	−0.0004 (5)	0.0013 (5)	0.0045 (5)
C4	0.0226 (6)	0.0196 (6)	0.0270 (6)	−0.0027 (5)	−0.0036 (5)	0.0044 (5)
C5	0.0239 (6)	0.0210 (6)	0.0261 (6)	0.0016 (5)	−0.0053 (5)	−0.0026 (5)
C6	0.0196 (6)	0.0251 (6)	0.0200 (6)	0.0021 (5)	−0.0005 (5)	−0.0024 (5)
C7	0.0160 (5)	0.0210 (6)	0.0170 (5)	−0.0001 (4)	−0.0013 (4)	0.0014 (4)
C8	0.0173 (5)	0.0240 (6)	0.0176 (6)	0.0002 (5)	0.0007 (4)	−0.0011 (5)
C9	0.0173 (5)	0.0229 (6)	0.0163 (5)	0.0004 (4)	0.0001 (4)	−0.0020 (5)
C10	0.0220 (6)	0.0259 (6)	0.0159 (6)	0.0006 (5)	−0.0010 (4)	0.0000 (5)
C11	0.0277 (6)	0.0230 (6)	0.0198 (6)	0.0008 (5)	0.0034 (5)	0.0037 (5)
C12	0.0247 (6)	0.0193 (6)	0.0232 (6)	−0.0022 (5)	0.0032 (5)	0.0002 (5)

Geometric parameters (Å, º)

S1—C2	1.7639 (15)	C4—H4A	0.9500
S1—C1	1.8114 (14)	C5—C6	1.389 (2)
S2—C12	1.7132 (15)	C5—H5A	0.9500
S2—C9	1.7261 (14)	C6—C7	1.3932 (19)
N1—C8	1.2827 (17)	C6—H6A	0.9500

N1—C7	1.4114 (17)	C8—C9	1.4396 (18)
C1—C1^{i}	1.524 (3)	C8—H8A	0.9500
C1—H1A	0.9900	C9—C10	1.3757 (18)
C1—H1B	0.9900	C10—C11	1.414 (2)
C2—C3	1.3944 (18)	C10—H10A	0.9500
C2—C7	1.4122 (17)	C11—C12	1.3644 (19)
C3—C4	1.3897 (19)	C11—H11A	0.9500
C3—H3A	0.9500	C12—H12B	0.9500
C4—C5	1.384 (2)		

C2—S1—C1	102.36 (6)	C5—C6—C7	120.35 (12)
C12—S2—C9	91.53 (7)	C5—C6—H6A	119.8
C8—N1—C7	118.94 (11)	C7—C6—H6A	119.8
C1^{i}—C1—S1	107.55 (11)	C6—C7—N1	122.90 (11)
C1^{i}—C1—H1A	110.2	C6—C7—C2	119.91 (12)
S1—C1—H1A	110.2	N1—C7—C2	117.03 (11)
C1^{i}—C1—H1B	110.2	N1—C8—C9	121.68 (12)
S1—C1—H1B	110.2	N1—C8—H8A	119.2
H1A—C1—H1B	108.5	C9—C8—H8A	119.2
C3—C2—C7	118.94 (12)	C10—C9—C8	127.19 (12)
C3—C2—S1	125.63 (10)	C10—C9—S2	111.26 (10)
C7—C2—S1	115.42 (9)	C8—C9—S2	121.50 (10)
C4—C3—C2	120.32 (12)	C9—C10—C11	112.50 (12)
C4—C3—H3A	119.8	C9—C10—H10A	123.7
C2—C3—H3A	119.8	C11—C10—H10A	123.7
C5—C4—C3	120.66 (12)	C12—C11—C10	112.59 (12)
C5—C4—H4A	119.7	C12—C11—H11A	123.7
C3—C4—H4A	119.7	C10—C11—H11A	123.7
C4—C5—C6	119.75 (13)	C11—C12—S2	112.12 (10)
C4—C5—H5A	120.1	C11—C12—H12B	123.9
C6—C5—H5A	120.1	S2—C12—H12B	123.9

C2—S1—C1—C1^{i}	−166.07 (12)	S1—C2—C7—C6	178.23 (9)
C1—S1—C2—C3	−4.13 (13)	C3—C2—C7—N1	−178.52 (11)
C1—S1—C2—C7	174.52 (9)	S1—C2—C7—N1	2.73 (14)
C7—C2—C3—C4	2.25 (19)	C7—N1—C8—C9	−177.31 (11)
S1—C2—C3—C4	−179.14 (10)	N1—C8—C9—C10	−174.31 (13)
C2—C3—C4—C5	0.2 (2)	N1—C8—C9—S2	8.43 (18)
C3—C4—C5—C6	−1.9 (2)	C12—S2—C9—C10	−0.04 (10)
C4—C5—C6—C7	1.1 (2)	C12—S2—C9—C8	177.62 (11)
C5—C6—C7—N1	176.60 (12)	C8—C9—C10—C11	−177.69 (12)
C5—C6—C7—C2	1.38 (19)	S2—C9—C10—C11	−0.20 (15)
C8—N1—C7—C6	53.92 (17)	C9—C10—C11—C12	0.42 (17)
C8—N1—C7—C2	−130.73 (13)	C10—C11—C12—S2	−0.44 (15)
C3—C2—C7—C6	−3.02 (18)	C9—S2—C12—C11	0.28 (11)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C10—H10*A*···*Cg*1^{ii}	0.95	2.80	3.740 (3)	171

Symmetry code: (ii) $x, -y-1/2, z-1/2$.