Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

N,*N*′-Bis[(*E*)-1-(thiophen-3-yl)ethylidene]ethane-1,2-diamine

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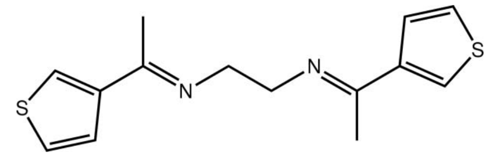
Received 5 March 2012; accepted 5 March 2012

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.039; wR factor = 0.105; data-to-parameter ratio = 18.6.

The complete molecule of the title compound, $C_{14}H_{16}N_2S_2$, is generated by a crystallographic inversion centre. The thiophene residue is close to being coplanar with the imine group [C—C—C—N torsion angle = 6.5 (2)°], and the conformation about the imine C═N bond [1.281 (2) Å] is *E*. In the crystal, the three-dimensional architecture is consolidated by C—H⋯N, C—H⋯π and S⋯S [3.3932 (7) Å] interactions.

Related literature

For background to 2-substituted thiophenes, see: Kleemann *et al.* (2006). For related structures, see: Prasath *et al.* (2010*a*,*b*).



Experimental

Crystal data

$C_{14}H_{16}N_2S_2$
M_r = 276.41
Monoclinic, $P2_1/c$
a = 7.5231 (6) Å
b = 11.2338 (6) Å
c = 8.5967 (6) Å
β = 112.894 (9)°
V = 669.30 (8) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.38 mm^{-1}
T = 100 K
0.20 × 0.15 × 0.10 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2011)
$T_{\min}$ = 0.928, $T_{\max}$ = 0.963
2789 measured reflections
1542 independent reflections
1339 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.033

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.105
S = 1.06
1542 reflections
83 parameters
H-atom parameters constrained
$\Delta\rho_{\max}$ = 0.45 e Å^{-3}
$\Delta\rho_{\min}$ = −0.41 e Å^{-3}

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

*Cg*1 is the centroid of the S1,C1–C4 ring.

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C1—H1⋯N1$^{\rm i}$	0.95	2.51	3.454 (2)	172
C6—H6*C*⋯*Cg*1$^{\rm ii}$	0.98	2.74	3.624 (2)	150

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

Data collection: *CrysAlis PRO* (Agilent, 2011); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

The authors are grateful to the Center of Excellence for Advanced Materials Research and the Chemistry Department at King Abdulaziz University for providing the research facilities. We also thank the Ministry of Higher Education (Malaysia) for funding structural studies through the High-Impact Research scheme (UM.C/HIR/MOHE/SC/12).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6669).

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supplementary materials

Acta Cryst. (2012). E**68**, o1026 [doi:10.1107/S1600536812009798]

N,*N*′-Bis[(*E*)-1-(thiophen-3-yl)ethylidene]ethane-1,2-diamine

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Comment

Thiophenes attract attention for their biological activity amongst other properties (Kleemann *et al.*, 2006). In continuation of structural studies of thienyl derivatives (Prasath *et al.*, 2010*a*; Prasath *et al.*, 2010*b*), herein the title compound, bis-[1-(thiophen-3-yl)ethylidene]ethane-1,2-diamine (I), is described.

The asymmetric unit in (I), Fig. 1, comprises half a molecule with the full molecule generated by a crystallographic centre of inversion. The thiophene residue is co-planar with the imine group as seen in the value of the C2—C3—C5—N1 torsion angle of 6.5 (2) °. In fact the entire molecule is planar with the r.m.s. deviation for the 18 non-hydrogen atoms being 0.068 Å; the maximum deviations are found for the S1 [0.092 (1) Å] and C2 [-0.099 (2) Å] atoms. The conformation about the imine N1—C5 bond [1.281 (2) Å] is *E*.

In the crystal packing the molecules associate *via* C—H···N, C—H···*π*, [Table 1] and S···S [S1···S1^i = 3.3932 (7) Å for *i*: 2 - *x*, 1 - *y*, 2 - *z*] interactions to form a three-dimensional architecture, Fig. 2.

Experimental

A mixture of ethylenediamine (0.6 g, 0.01 *M*) and 2-acetyl thiophene (0.7 g, 0.01 *M*) in dry benzene (50 ml) was refluxed using a Dean-Stark trap until no more water was collected (2 h). The benzene was then removed under reduced pressure and the residue treated with methanol. The solid that separated out was recrystallized from ethanol as colourless prisms. Yield: 72%. *M*.pt: 405–407 K.

Refinement

Carbon-bound H-atoms were placed in calculated positions [C—H = 0.95 to 0.99 Å, U_{iso}(H) = 1.2 to 1.5U_{eq}(C)] and were included in the refinement in the riding model approximation.

Computing details

Data collection: *CrysAlis PRO* (Agilent, 2011); cell refinement: *CrysAlis PRO* (Agilent, 2011); data reduction: *CrysAlis PRO* (Agilent, 2011); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

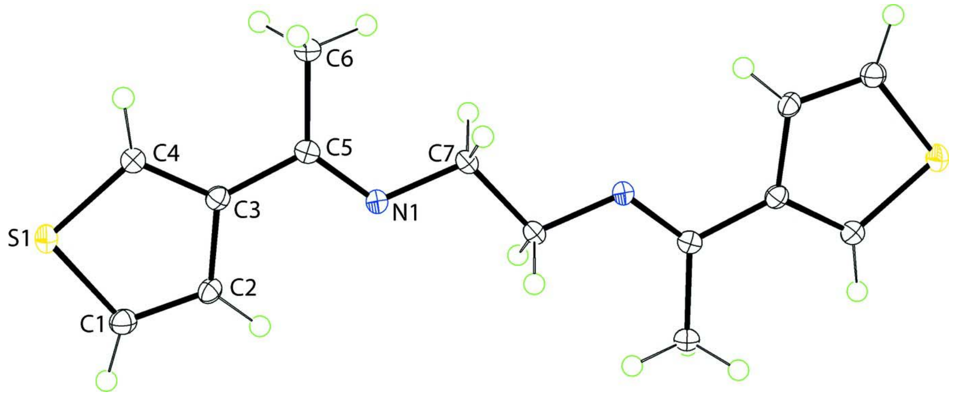

Figure 1

The molecular structure of (I) showing displacement ellipsoids at the 50% probability level. The unlabelled atoms are related by the symmetry operation (1-*x*, 1-*y*, -*z*).

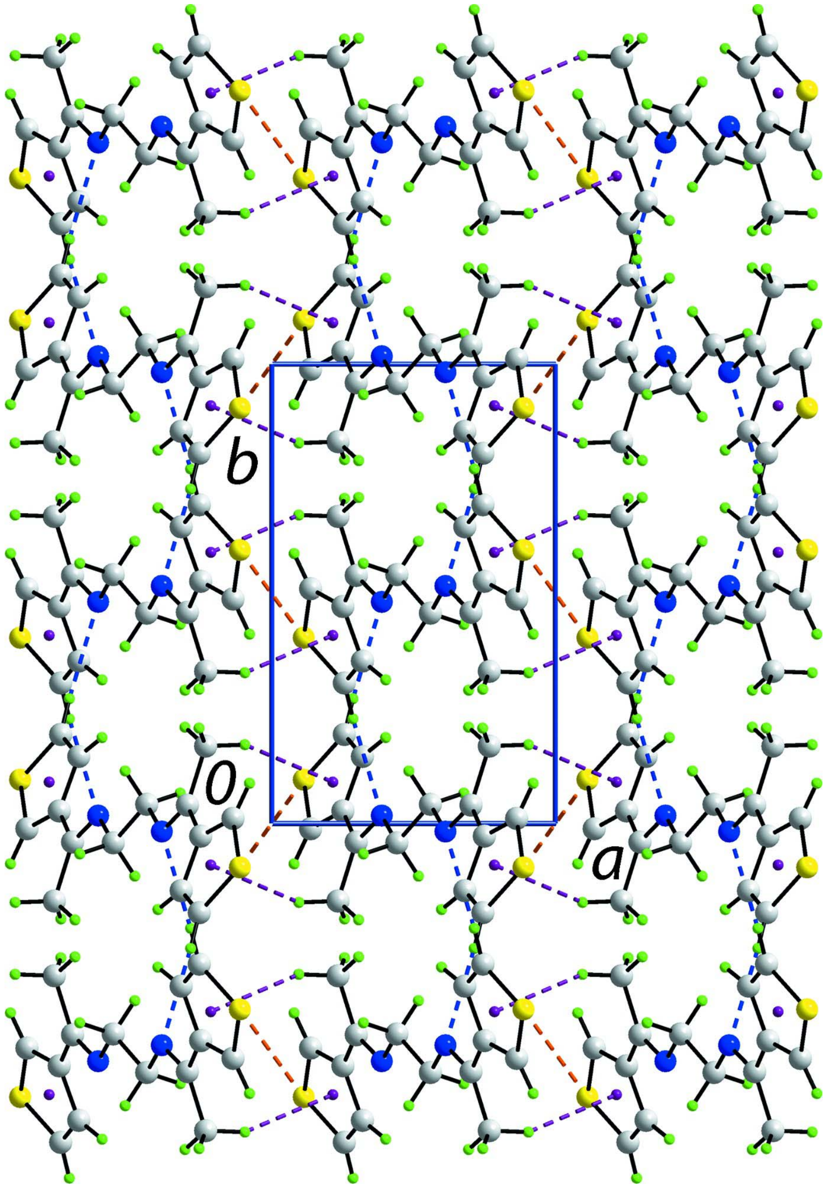

Figure 2

A view in projection down the *c* axis of the unit-cell contents of (I). The C—N···N, C—H···π and S···S interactions are shown as blue, purple and orange dashed lines, respectively.

N,*N*′-Bis[(*E*)-1-(thiophen-3-yl)ethylidene]ethane-1,2-diamine

Crystal data

$C_{14}H_{16}N_2S_2$	$V = 669.30\ (8)$ Å^3
$M_r = 276.41$	$Z = 2$
Monoclinic, $P2_1/c$	$F(000) = 292$
Hall symbol: -P 2ybc	$D_x = 1.372$ Mg m^{-3}
$a = 7.5231\ (6)$ Å	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$b = 11.2338\ (6)$ Å	Cell parameters from 1637 reflections
$c = 8.5967\ (6)$ Å	$\theta = 2.6$–$27.5°$
$\beta = 112.894\ (9)°$	$\mu = 0.38$ mm^{-1}

T = 100 K

Prism, colourless

0.20 × 0.15 × 0.10 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector

Radiation source: SuperNova (Mo) X-ray Source

Mirror monochromator

Detector resolution: 10.4041 pixels mm^{-1}

ω scan

Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2011)

$T_{\min}$ = 0.928, $T_{\max}$ = 0.963

2789 measured reflections

1542 independent reflections

1339 reflections with $I > 2\sigma(I)$

R_{int} = 0.033

$\theta_{\max}$ = 27.6°, $\theta_{\min}$ = 2.9°

$h = -9 \rightarrow 9$

$k = -14 \rightarrow 9$

$l = -11 \rightarrow 7$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)]$ = 0.039

$wR(F^2)$ = 0.105

S = 1.06

1542 reflections

83 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

$w = 1/[\sigma^2(F_o^2) + (0.0482P)^2 + 0.3751P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max}$ = 0.001

$\Delta\rho_{\max}$ = 0.45 e Å^{-3}

$\Delta\rho_{\min}$ = −0.41 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	0.88734 (7)	0.59544 (4)	0.83858 (6)	0.01735 (17)
N1	0.6122 (2)	0.51740 (13)	0.23310 (18)	0.0129 (3)
C1	0.7443 (3)	0.69397 (16)	0.6897 (2)	0.0161 (4)
H1	0.7138	0.7719	0.7143	0.019*
C2	0.6805 (3)	0.64614 (16)	0.5325 (2)	0.0138 (4)
H2	0.5985	0.6872	0.4342	0.017*
C3	0.7493 (2)	0.52741 (15)	0.5294 (2)	0.0127 (4)
C4	0.8632 (3)	0.48878 (16)	0.6889 (2)	0.0151 (4)
H4	0.9214	0.4123	0.7133	0.018*
C5	0.7023 (2)	0.45986 (15)	0.3700 (2)	0.0121 (4)
C6	0.7668 (3)	0.33137 (16)	0.3829 (2)	0.0156 (4)
H6A	0.6939	0.2903	0.2764	0.023*
H6B	0.7435	0.2923	0.4751	0.023*
H6C	0.9049	0.3283	0.4055	0.023*

C7	0.5591 (3)	0.45805 (16)	0.0702 (2)	0.0144 (4)
H7A	0.4831	0.3856	0.0675	0.017*
H7B	0.6771	0.4338	0.0537	0.017*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0200 (3)	0.0185 (3)	0.0112 (3)	0.00100 (18)	0.00349 (19)	−0.00088 (17)
N1	0.0144 (8)	0.0129 (7)	0.0110 (7)	−0.0001 (6)	0.0043 (6)	−0.0005 (6)
C1	0.0183 (9)	0.0150 (8)	0.0170 (9)	0.0011 (7)	0.0091 (7)	0.0012 (7)
C2	0.0147 (9)	0.0151 (9)	0.0128 (8)	0.0013 (7)	0.0065 (7)	0.0023 (7)
C3	0.0124 (8)	0.0129 (8)	0.0134 (9)	−0.0012 (7)	0.0057 (7)	0.0010 (7)
C4	0.0171 (9)	0.0137 (8)	0.0136 (9)	0.0003 (7)	0.0052 (7)	0.0001 (7)
C5	0.0101 (8)	0.0123 (8)	0.0146 (9)	−0.0011 (6)	0.0055 (7)	−0.0006 (7)
C6	0.0184 (9)	0.0125 (8)	0.0158 (9)	0.0020 (7)	0.0064 (7)	0.0004 (7)
C7	0.0177 (9)	0.0124 (8)	0.0121 (9)	0.0005 (7)	0.0047 (7)	−0.0024 (7)

Geometric parameters (Å, º)

S1—C4	1.7154 (18)	C3—C5	1.484 (2)
S1—C1	1.7174 (19)	C4—H4	0.9500
N1—C5	1.281 (2)	C5—C6	1.513 (2)
N1—C7	1.460 (2)	C6—H6A	0.9800
C1—C2	1.357 (2)	C6—H6B	0.9800
C1—H1	0.9500	C6—H6C	0.9800
C2—C3	1.435 (2)	C7—C7^{i}	1.517 (3)
C2—H2	0.9500	C7—H7A	0.9900
C3—C4	1.374 (2)	C7—H7B	0.9900
C4—S1—C1	92.19 (9)	N1—C5—C6	126.06 (15)
C5—N1—C7	120.01 (15)	C3—C5—C6	117.77 (15)
C2—C1—S1	111.31 (14)	C5—C6—H6A	109.5
C2—C1—H1	124.3	C5—C6—H6B	109.5
S1—C1—H1	124.3	H6A—C6—H6B	109.5
C1—C2—C3	113.35 (16)	C5—C6—H6C	109.5
C1—C2—H2	123.3	H6A—C6—H6C	109.5
C3—C2—H2	123.3	H6B—C6—H6C	109.5
C4—C3—C2	111.37 (16)	N1—C7—C7^{i}	109.65 (18)
C4—C3—C5	126.30 (16)	N1—C7—H7A	109.7
C2—C3—C5	122.32 (15)	C7^{i}—C7—H7A	109.7
C3—C4—S1	111.78 (14)	N1—C7—H7B	109.7
C3—C4—H4	124.1	C7^{i}—C7—H7B	109.7
S1—C4—H4	124.1	H7A—C7—H7B	108.2
N1—C5—C3	116.16 (15)		
C4—S1—C1—C2	0.34 (14)	C7—N1—C5—C3	−179.72 (15)
S1—C1—C2—C3	−0.7 (2)	C7—N1—C5—C6	1.3 (3)
C1—C2—C3—C4	0.8 (2)	C4—C3—C5—N1	−172.11 (17)
C1—C2—C3—C5	−177.97 (16)	C2—C3—C5—N1	6.5 (2)
C2—C3—C4—S1	−0.6 (2)	C4—C3—C5—C6	7.0 (3)

C5—C3—C4—S1	178.19 (14)	C2—C3—C5—C6	−174.39 (15)
C1—S1—C4—C3	0.13 (15)	C5—N1—C7—C7^{i}	175.58 (18)

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

Hydrogen-bond geometry (Å, º)

Cg1 is the centroid of the S1,C1–C4 ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1···N1^{ii}	0.95	2.51	3.454 (2)	172
C6—H6*C*···*Cg*1^{iii}	0.98	2.74	3.624 (2)	150

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