Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N,*N*′-Bis(2-thienylmethylene)ethane-1,2-diamine

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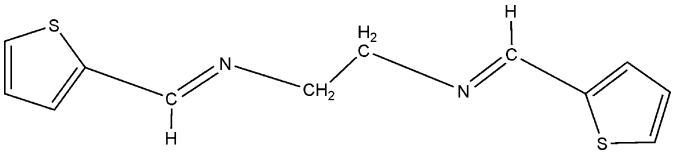
Received 9 November 2007; accepted 18 November 2007

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.007 Å; R factor = 0.044; wR factor = 0.081; data-to-parameter ratio = 11.5.

The title compound, $C_{12}H_{12}N_2S_2$, was prepared from a condensation reaction of thiophene-2-carboxaldehyde with ethylenediamine in refluxing ethanol. The molecule adopts a *Z*-shaped conformation, with the two thiophene rings lying on either side of the Csp^3—Csp^3 bond. The two thiophene rings form a dihedral angle of 11.2 (2)°. The crystal packing is mainly stabilized by van der Waals forces.

Related literature

For general background, see: Ittle *et al.* (2000).



Experimental

Crystal data

$C_{12}H_{12}N_2S_2$
M_r = 248.36
Monoclinic, $P2_1$
a = 6.4822 (8) Å
b = 7.4181 (10) Å
c = 13.1433 (15) Å
β = 101.425 (2)°
V = 619.48 (13) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.40 mm^{-1}
T = 298 (2) K
0.36 × 0.13 × 0.08 mm

Data collection

Siemens SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.869, T_{max} = 0.969
3150 measured reflections
1663 independent reflections
1136 reflections with $I > 2\sigma(I)$
R_{int} = 0.046

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.081
S = 1.03
1663 reflections
145 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}
Absolute structure: Flack (1983); 481 Friedel pairs
Flack parameter: −0.03 (12)

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

The authors thank Shandong Province Science Foundation, the State Key Laboratory of Crystalline Materials and Shandong University for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2514).

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 doi:10.1107/S1600536807060308

supplementary materials

Acta Cryst. (2007). E**63**, o4865 [**doi:10.1107/S1600536807060308**]

N,*N*'-Bis(2-thienylmethylene)ethane-1,2-diamine

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Comment

Multi-dentate complexes of iron and nickel show high activities of ethylene oligomerization and polymerization (Ittle *et al.*, 2000). We report here the crystal structure of the title compound, a new multidentate Schiff base compound.

The molecular structure of the title compound is shown in Fig.1. The molecule adopts a *Z*-shaped conformation, with the two thiophene rings lying on either side of the C11—C12 bond. The torsion angles C11—N1—C1—C2 [−177.9 (4)°], N1—C11—C12—N2 [178.9 (4)°] and C12—N2—C6—C7 [−176.2 (4)°] describe the overall conformation of the molecule. The two thiophene rings form a dihedral angle of 11.2 (2)°. The crystal packing is mainly stabilized by van der Waals forces.

Experimental

A absolute ethanol mixture of thiophene-2-carboxaldehyde (4 mmol) and ethyldiamine (2 mmol) was heated under reflux with stirring for 1.5 h and then filtered to obtain a clear pale yellow solution. Single crystals of the title compound suitable for X-ray diffraction analysis were obtained by vapour diffusion of diethyl ether into the yellow solution.

Refinement

H atoms were positioned geometrically [C—H = 0.93 (aromatic) or 0.97 Å (methylene)] and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C).

Figures

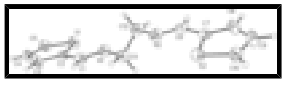

Fig. 1. The molecular structure of the title compound, showing 30% probability displacement ellipsoids and the atom-numbering scheme.

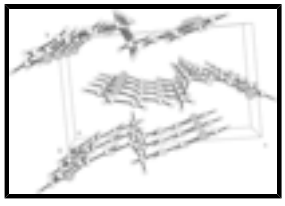

Fig. 2. The crystal packing of the title compound, viewed approximately along the *a* axis.

N,*N*'-Bis(2-thienylmethylene)ethane-1,2-diamine

Crystal data

$C_{12}H_{12}N_2S_2$	F_{000} = 260
M_r = 248.36	D_x = 1.331 Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation

	λ = 0.71073 Å
Hall symbol: P 2yb	Cell parameters from 1008 reflections
a = 6.4822 (8) Å	θ = 3.2–27.4º
b = 7.4181 (10) Å	μ = 0.40 mm^{-1}
c = 13.1433 (15) Å	T = 298 (2) K
β = 101.425 (2)º	Block, yellow
V = 619.48 (13) $Å^3$	0.36 × 0.13 × 0.08 mm
Z = 2	

Data collection

Siemens SMART CCD area-detector diffractometer	1663 independent reflections
Radiation source: fine-focus sealed tube	1136 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.046
T = 298(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 1.6º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -7 \rightarrow 7$
T_{min} = 0.869, T_{max} = 0.969	$k = -8 \rightarrow 5$
3150 measured reflections	$l = -13 \rightarrow 15$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.044	$w = 1/[\sigma^2(F_o^2) + (0.0294P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.081	$(\Delta/\sigma)_{max}$ = 0.001
S = 1.03	$\Delta\rho_{max}$ = 0.19 e $Å^{-3}$
1663 reflections	$\Delta\rho_{min}$ = −0.26 e $Å^{-3}$
145 parameters	Extinction correction: none
1 restraint	Absolute structure: Flack (1983); 481 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: −0.03 (12)
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 calculat-

ing *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	0.08972 (17)	0.55346 (16)	0.29575 (9)	0.0620 (4)
S2	0.98557 (19)	0.38242 (15)	0.95907 (9)	0.0589 (4)
N1	0.3457 (6)	0.4695 (4)	0.5119 (3)	0.0544 (10)
N2	0.7400 (6)	0.5301 (5)	0.7551 (3)	0.0562 (10)
C1	0.1512 (8)	0.4405 (6)	0.4996 (3)	0.0543 (14)
H1	0.0983	0.3942	0.5550	0.065*
C2	0.0056 (7)	0.4765 (5)	0.4024 (3)	0.0473 (12)
C3	−0.2071 (7)	0.4550 (6)	0.3820 (4)	0.0607 (14)
H3	−0.2828	0.4144	0.4307	0.073*
C4	−0.3001 (8)	0.5010 (7)	0.2791 (4)	0.0668 (15)
H4	−0.4439	0.4944	0.2524	0.080*
C5	−0.1594 (7)	0.5547 (8)	0.2245 (4)	0.0625 (13)
H5	−0.1935	0.5891	0.1552	0.075*
C6	0.9347 (7)	0.5520 (7)	0.7699 (3)	0.0514 (12)
H6	0.9913	0.6062	0.7177	0.062*
C7	1.0773 (7)	0.4971 (5)	0.8644 (3)	0.0460 (11)
C8	1.2899 (7)	0.5175 (7)	0.8889 (3)	0.0634 (14)
H8	1.3693	0.5765	0.8474	0.076*
C9	1.3748 (8)	0.4369 (7)	0.9864 (4)	0.0738 (17)
H9	1.5177	0.4373	1.0156	0.089*
C10	1.2317 (8)	0.3615 (7)	1.0316 (4)	0.0616 (14)
H10	1.2623	0.3037	1.0956	0.074*
C11	0.4766 (7)	0.4224 (6)	0.6113 (3)	0.0619 (14)
H11A	0.5646	0.3200	0.6027	0.074*
H11B	0.3880	0.3884	0.6596	0.074*
C12	0.6137 (7)	0.5800 (6)	0.6546 (3)	0.0627 (14)
H12A	0.7049	0.6127	0.6073	0.075*
H12B	0.5264	0.6833	0.6625	0.075*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0541 (8)	0.0700 (9)	0.0616 (8)	−0.0122 (7)	0.0105 (6)	0.0102 (7)
S2	0.0584 (9)	0.0623 (8)	0.0572 (8)	−0.0005 (7)	0.0144 (7)	0.0098 (6)
N1	0.055 (2)	0.061 (3)	0.045 (2)	0.000 (2)	0.004 (2)	0.0015 (17)
N2	0.062 (3)	0.055 (3)	0.047 (2)	0.007 (2)	0.002 (2)	0.001 (2)
C1	0.073 (4)	0.048 (3)	0.043 (3)	−0.005 (3)	0.015 (3)	−0.005 (2)
C2	0.050 (3)	0.043 (3)	0.050 (3)	−0.006 (2)	0.011 (2)	−0.007 (2)
C3	0.052 (3)	0.070 (4)	0.066 (4)	−0.018 (3)	0.026 (3)	−0.015 (3)
C4	0.048 (3)	0.070 (4)	0.080 (4)	−0.006 (3)	0.006 (3)	−0.009 (3)
C5	0.061 (3)	0.059 (3)	0.060 (3)	−0.006 (3)	−0.006 (3)	0.005 (3)
C6	0.072 (3)	0.040 (3)	0.043 (3)	−0.001 (3)	0.012 (3)	0.000 (2)

C7	0.055 (3)	0.041 (3)	0.043 (3)	0.000 (2)	0.014 (2)	−0.001 (2)
C8	0.056 (3)	0.076 (4)	0.060 (3)	−0.017 (3)	0.017 (3)	−0.001 (3)
C9	0.053 (3)	0.094 (4)	0.066 (4)	−0.008 (3)	−0.009 (3)	−0.005 (3)
C10	0.060 (3)	0.065 (4)	0.054 (3)	−0.001 (3)	−0.001 (3)	0.009 (3)
C11	0.072 (3)	0.063 (4)	0.047 (3)	0.002 (3)	0.002 (3)	0.007 (2)
C12	0.076 (3)	0.059 (3)	0.047 (3)	0.005 (3)	−0.001 (3)	0.006 (2)

Geometric parameters (Å, °)

S1—C5	1.699 (4)	C5—H5	0.93
S1—C2	1.700 (4)	C6—C7	1.452 (5)
S2—C10	1.696 (5)	C6—H6	0.93
S2—C7	1.708 (4)	C7—C8	1.360 (5)
N1—C1	1.257 (5)	C8—C9	1.422 (6)
N1—C11	1.453 (5)	C8—H8	0.93
N2—C6	1.249 (5)	C9—C10	1.320 (6)
N2—C12	1.457 (5)	C9—H9	0.93
C1—C2	1.455 (6)	C10—H10	0.93
C1—H1	0.93	C11—C12	1.510 (5)
C2—C3	1.361 (6)	C11—H11A	0.97
C3—C4	1.409 (6)	C11—H11B	0.97
C3—H3	0.93	C12—H12A	0.97
C4—C5	1.328 (6)	C12—H12B	0.97
C4—H4	0.93		
C5—S1—C2	91.8 (2)	C8—C7—S2	111.4 (3)
C10—S2—C7	91.7 (2)	C6—C7—S2	120.6 (3)
C1—N1—C11	117.8 (4)	C7—C8—C9	111.1 (4)
C6—N2—C12	118.5 (4)	C7—C8—H8	124.5
N1—C1—C2	122.8 (4)	C9—C8—H8	124.5
N1—C1—H1	118.6	C10—C9—C8	113.7 (4)
C2—C1—H1	118.6	C10—C9—H9	123.1
C3—C2—C1	127.4 (4)	C8—C9—H9	123.1
C3—C2—S1	110.8 (3)	C9—C10—S2	112.0 (4)
C1—C2—S1	121.8 (3)	C9—C10—H10	124.0
C2—C3—C4	112.6 (4)	S2—C10—H10	124.0
C2—C3—H3	123.7	N1—C11—C12	110.4 (3)
C4—C3—H3	123.7	N1—C11—H11A	109.6
C5—C4—C3	112.5 (4)	C12—C11—H11A	109.6
C5—C4—H4	123.8	N1—C11—H11B	109.6
C3—C4—H4	123.8	C12—C11—H11B	109.6
C4—C5—S1	112.3 (4)	H11A—C11—H11B	108.1
C4—C5—H5	123.8	N2—C12—C11	109.2 (4)
S1—C5—H5	123.8	N2—C12—H12A	109.8
N2—C6—C7	123.3 (4)	C11—C12—H12A	109.8
N2—C6—H6	118.4	N2—C12—H12B	109.8
C7—C6—H6	118.4	C11—C12—H12B	109.8
C8—C7—C6	127.9 (4)	H12A—C12—H12B	108.3
C11—N1—C1—C2	−177.9 (4)	N2—C6—C7—S2	4.7 (7)
N1—C1—C2—C3	−177.9 (4)	C10—S2—C7—C8	−0.1 (4)

N1—C1—C2—S1	2.4 (6)	C10—S2—C7—C6	177.0 (4)
C5—S1—C2—C3	−0.4 (4)	C6—C7—C8—C9	−176.7 (4)
C5—S1—C2—C1	179.2 (4)	S2—C7—C8—C9	0.2 (5)
C1—C2—C3—C4	−179.4 (4)	C7—C8—C9—C10	−0.2 (6)
S1—C2—C3—C4	0.2 (5)	C8—C9—C10—S2	0.2 (6)
C2—C3—C4—C5	0.2 (6)	C7—S2—C10—C9	−0.1 (4)
C3—C4—C5—S1	−0.5 (6)	C1—N1—C11—C12	−128.7 (4)
C2—S1—C5—C4	0.6 (5)	C6—N2—C12—C11	127.3 (5)
C12—N2—C6—C7	−176.2 (4)	N1—C11—C12—N2	178.9 (4)
N2—C6—C7—C8	−178.7 (5)		

Fig. 1

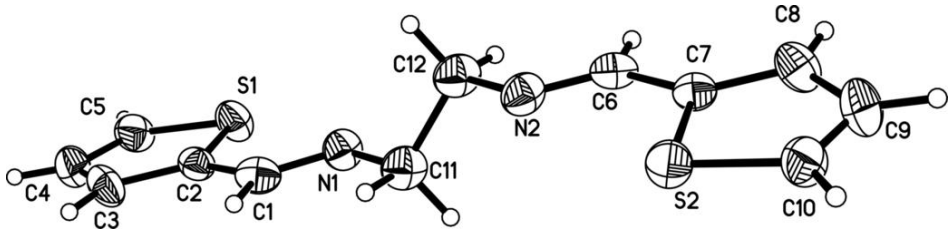

Fig. 2