organic compounds

N,N′-Bis(2-thienylmethylene)benzene-1,4-diamine

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The Schiff base, $C_{16}H_{12}N_2S_2$, has been synthesized by refluxing an ethanolic solution of thiophene-2-carbaldehyde and benzene-1,4-diamine. The center of the benzene ring is located on a crystallographic center of inversion. The dihedral angle between the benzene and thiophene rings is 63.6 (1)°.

Related literature

For general background to Schiff base complexes, see: Andersen *et al.* (2005); Koizumi *et al.* (2005); Boskovic *et al.* (2003); Oshio *et al.* (2005). For the synthesis, see: Kannappan *et al.* (2005).
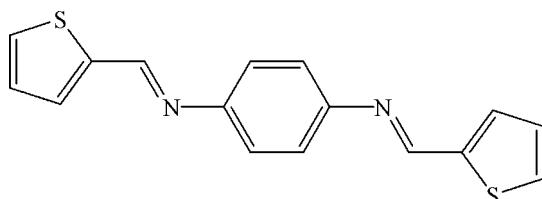
Experimental

Crystal data

$C_{16}H_{12}N_2S_2$

$M_r = 296.40$

Monoclinic, $P2_1/n$
$a = 6.1882$ (7) Å
$b = 7.2371$ (9) Å
$c = 16.375$ (2) Å
$\beta = 95.860$ (2)°
$V = 729.5$ (2) Å^3

$Z = 2$
Mo $K\alpha$ radiation
$\mu = 0.36$ mm^{-1}
$T = 294$ K
$0.15 \times 0.11 \times 0.09$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
$T_{min} = 0.949$, $T_{max} = 0.969$

3380 measured reflections
1252 independent reflections
1021 reflections with $I > 2\sigma(I)$
$R_{int} = 0.019$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.065$
$wR(F^2) = 0.196$
$S = 1.00$
1252 reflections

91 parameters
H-atom parameters not refined
$\Delta\rho_{max} = 1.07$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}

Data collection: *APEX2* (Bruker, 2002); cell refinement: *SAINT-Plus* (Bruker, 2003); data reduction: *SAINT-Plus*; 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*.

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supplementary materials

N,N'-Bis(2-thienylmethylene)benzene-1,4-diamine

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Comment

During the past decades, Schiff bases have been intensively investigated not only because of their strong coordination capability but also due to diverse biological activities, such as e.g. antibacterial and antitumor activities (Andersen *et al.*, 2005; Koizumi *et al.*, 2005; Boskovic *et al.*, 2003; Oshio *et al.*, 2005). Here we report the structure of N,N'-bis-thiophene-2-yl-methylene-1,4-benzenediamine, which could be used as another Schiff base ligand in combination with metal ions.

The molecular structure of the title compound is depicted in Figure 1. The center of the phenyl ring represents a crystallographic center of inversion. The dihedral angle between the phenyl and thiophene rings is 63.6 (1)°. As it is expected the thiophene rings also are perfectly planar with a deviation of the S atom of 0.001 Å.

Experimental

N,N'-Bis-thiophene-2-yl-methylene-1,4-benzenediamine was prepared according to the method reported in the literature (Kannappan *et al.*, 2005). Thiophene-2-carboxaldehyde (0.02 mol) was added to a stirred ethanolic solution of *p*-phenylenediamine (0.01 mol). The reaction mixture was stirred about 3 h and then the mixture was allowed to stand at room temperature for about two days. Yellow crystals suitable for X-ray diffraction analysis were collected with a yield of 60%. Anal. Calc. for $C_{16}H_{12}N_2S_2$: C 64.78, H 4.05, N 9.45%; Found: C 64.59, H 4.01, N 9.25%.

Refinement

All H atoms were placed in calculated positions with C—H = 0.93Å and refined as riding with $U_{iso}(H) = 1.2U_{eq}$(carrier).

Figures
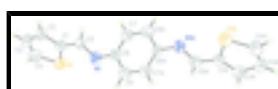
Fig. 1. View of the molecular structure of the title compound, showing the atomic numbering scheme and 50% probability displacement ellipsoids. Atoms labeled with A at the symmetry positions (-*x* + 1,-*y* + 1,-*z* + 1).

N,N'-Bis(2-thienylmethylene)benzene-1,4-diamine

Crystal data

$C_{16}H_{12}N_2S_2$	$F_{000} = 308$
$M_r = 296.40$	$D_x = 1.349$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2yn	Cell parameters from 1252 reflections
$a = 6.1882$ (7) Å	$\theta = 3.1–25.0°$
$b = 7.2371$ (9) Å	$\mu = 0.36$ mm^{-1}

$c = 16.375\ (2)$ Å

$\beta = 95.860\ (2)°$

$V = 729.5\ (2)$ Å^3

$Z = 2$

$T = 294$ K

Block, yellow

$0.15 \times 0.11 \times 0.09$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer	1252 independent reflections
Radiation source: fine-focus sealed tube	1021 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.019$
$T = 294$ K	$\theta_{max} = 25.0°$
φ and ω scans	$\theta_{min} = 3.1°$
Absorption correction: multi-scan (SADABS; Sheldrick, 2004)	$h = -7 \rightarrow 7$
$T_{min} = 0.949$, $T_{max} = 0.969$	$k = -8 \rightarrow 8$
3380 measured reflections	$l = -19 \rightarrow 10$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.065$	H-atom parameters not refined
$wR(F^2) = 0.196$	$w = 1/[\sigma^2(F_o{}^2) + (0.141P)^2 + 0.3146P]$ where $P = (F_o{}^2 + 2F_c{}^2)/3$
$S = 1.00$	$(\Delta/\sigma)_{max} < 0.001$
1252 reflections	$\Delta\rho_{max} = 1.07$ e Å^{-3}
91 parameters	$\Delta\rho_{min} = -0.21$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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	0.15131 (14)	−0.10612 (13)	0.29041 (6)	0.0619 (5)

N1	0.3196 (4)	0.1917 (4)	0.41314 (16)	0.0486 (7)
C1	0.0201 (5)	0.0013 (4)	0.36497 (18)	0.0462 (7)
C2	−0.1804 (5)	−0.0755 (5)	0.3705 (2)	0.0565 (9)
H2	−0.2774	−0.0333	0.4061	0.068*
C3	−0.2234 (6)	−0.2258 (5)	0.3163 (3)	0.0664 (10)
H3	−0.3489	−0.2972	0.3136	0.080*
C4	−0.0594 (6)	−0.2534 (6)	0.2685 (3)	0.0707 (11)
H4	−0.0624	−0.3437	0.2280	0.085*
C5	0.1188 (5)	0.1553 (4)	0.41151 (18)	0.0456 (7)
H5	0.0321	0.2297	0.4411	0.055*
C6	0.4043 (5)	0.3492 (4)	0.45720 (17)	0.0422 (7)
C7	0.3093 (5)	0.5209 (5)	0.4490 (2)	0.0564 (9)
H7	0.1820	0.5361	0.4142	0.068*
C8	0.6000 (6)	0.3299 (5)	0.5086 (2)	0.0576 (9)
H8	0.6681	0.2153	0.5138	0.069*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0552 (6)	0.0657 (7)	0.0656 (7)	−0.0030 (4)	0.0105 (4)	−0.0170 (4)
N1	0.0464 (15)	0.0470 (15)	0.0525 (15)	−0.0058 (11)	0.0059 (11)	−0.0114 (12)
C1	0.0453 (16)	0.0484 (17)	0.0436 (15)	0.0007 (12)	−0.0013 (12)	−0.0027 (13)
C2	0.0483 (18)	0.062 (2)	0.059 (2)	−0.0059 (15)	0.0054 (15)	−0.0085 (16)
C3	0.0528 (19)	0.061 (2)	0.083 (3)	−0.0100 (16)	−0.0059 (18)	−0.0154 (19)
C4	0.068 (2)	0.063 (2)	0.079 (2)	−0.0017 (18)	−0.0038 (19)	−0.0248 (19)
C5	0.0500 (17)	0.0447 (16)	0.0424 (16)	0.0006 (13)	0.0052 (13)	−0.0041 (12)
C6	0.0422 (16)	0.0427 (15)	0.0417 (15)	−0.0024 (11)	0.0046 (12)	−0.0051 (12)
C7	0.0480 (17)	0.0531 (19)	0.065 (2)	0.0017 (14)	−0.0066 (15)	−0.0032 (16)
C8	0.055 (2)	0.0484 (18)	0.068 (2)	0.0073 (14)	−0.0004 (16)	−0.0039 (16)

Geometric parameters (Å, °)

S1—C4	1.694 (4)	C3—H3	0.9300
S1—C1	1.719 (3)	C4—H4	0.9300
N1—C5	1.268 (4)	C5—H5	0.9300
N1—C6	1.421 (4)	C6—C7	1.375 (4)
C1—C2	1.371 (4)	C6—C8	1.410 (4)
C1—C5	1.449 (4)	C7—C8^{i}	1.373 (5)
C2—C3	1.412 (5)	C7—H7	0.9300
C2—H2	0.9300	C8—C7^{i}	1.373 (5)
C3—C4	1.358 (6)	C8—H8	0.9300
C4—S1—C1	91.48 (17)	S1—C4—H4	123.5
C5—N1—C6	119.1 (3)	N1—C5—C1	122.0 (3)
C2—C1—C5	127.8 (3)	N1—C5—H5	119.0
C2—C1—S1	111.1 (2)	C1—C5—H5	119.0
C5—C1—S1	121.1 (2)	C7—C6—C8	118.8 (3)
C1—C2—C3	112.6 (3)	C7—C6—N1	122.9 (3)
C1—C2—H2	123.7	C8—C6—N1	118.2 (3)

C3—C2—H2	123.7	C8^i—C7—C6	120.8 (3)
C4—C3—C2	111.8 (3)	C8^i—C7—H7	119.6
C4—C3—H3	124.1	C6—C7—H7	119.6
C2—C3—H3	124.1	C7^i—C8—C6	120.4 (3)
C3—C4—S1	113.0 (3)	C7^i—C8—H8	119.8
C3—C4—H4	123.5	C6—C8—H8	119.8

Symmetry codes: (i) −x+1, −y+1, −z+1.

Fig. 1