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N^1,N^4-Bis(2-thienylmethylene)cyclohexane-1,4-diamine

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Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.002 Å; R factor = 0.032; wR factor = 0.089; data-to-parameter ratio = 14.9.

The title compound, $C_{16}H_{18}N_2S_2$, lies about an inversion center with only half of the molecule in the asymmetric unit. The cyclohexane ring adopts a chair conformation, and the terminal thiophene rings are in a *transoid* orientation, with an S$\cdots$S separation between the two terminal 2-thiophene rings of 11.6733 (9) Å.

Related literature

For a general introduction to coordination polymers, see: Batten *et al.* (2009); Perry *et al.* (2009); Robin & Fromm (2006). For structurally related compounds, see: Yun *et al.* (2009). For related linking ligands containing terminal thiophene rings, see: Lee & Lee (2007); Huh *et al.* (2008); Kim & Lee (2008).

Experimental

Crystal data

$C_{16}H_{18}N_2S_2$	M_r = 302.44
Monoclinic, $P2_1/n$	Z = 2
a = 6.2173 (4) Å	Mo $K\alpha$ radiation
b = 7.4999 (5) Å	μ = 0.33 mm^{-1}
c = 17.1289 (12) Å	T = 296 K
β = 97.047 (3)°	0.46 × 0.24 × 0.20 mm
V = 792.67 (9) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	9045 measured reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	1889 independent reflections
T_{min} = 0.864, T_{max} = 0.937	1590 reflections with $I > 2\sigma(I)$
	R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032	127 parameters
$wR(F^2)$ = 0.089	All H-atom parameters refined
S = 1.02	$\Delta\rho_{max}$ = 0.19 e Å^{-3}
1889 reflections	$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2205).

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supplementary materials

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N^1,*N*^{4}-Bis(2-thienylmethylene)cyclohexane-1,4-diamine

K.-E. Lee and S. W. Lee

Comment

Design and construction of coordination polymers (or metal–organic frameworks, MOFs) are currently under intensive study due to their desirable applications in catalysis, nonlinear optical activity, spin crossover, luminescence, long-range magnetism, adsorption–desorption, and gas storage (Batten *et al.*, 2009; Perry IV *et al.*, 2009; Robin & Fromm, 2006). In preparing such polymers, appropriate linking ligands play a fundamental role. We have continually reported long bis(pyridine)-, bis(furan)-, bis(thiophene)-, and (pyridine–amine)-type linking ligands and their coordination polymers (Yun *et al.* 2009). As an extension to our ongoing study of novel linking ligands and their coordination polymers, we have prepared a long, potential linking ligand containing an intervening cyclohexane ring with two terminal thiophene rings.

The molecular structure of the title compound (Fig. 1) contains an intervening cyclohexane ring between two imine-thiophene (—*N*═CH—2-thiophene) fragments. The cyclohexane ring fragment adopts a chair conformation. The imine fragments occupy the equatorial sites of the cyclohexane ring and are *trans* with respect to each other. The terminal thiophene rings also adopt an overall *transoid* conformation. The S···S separation between the two terminal 2-thiophene rings is 11.6733 (9) Å. Several related linking ligands containing terminal thiophene rings were previously employed to obtain coordination networks: (2-thiophene)—CH═*N*—*N*═CH—(2-thiophene) (Lee & Lee, 2007; Huh *et al.*, 2008) and (3-thiophene)—CH═*N*—*N*═CH—(3-thiophene) (Kim & Lee, 2008).

Experimental

At room temperature, *trans*-1,4-diaminocyclohexane (1.0 g, 8.76 mmol) was added to 2-thiophene carboxaldehyde (1.72 ml, 18.72 mmol) in 80 ml methanol. After adding dichloromethane (50 ml) and three drops of formic acid, the mixture was stirred for 15 h, and then the solvent was removed under vacuum. The resulting solid was extracted with dichloromethane (150 ml) and washed with water (30 ml × 3). The organic phase was dried over $MgSO_4$ and then filtered. All the solvent was removed to give white crude solid, which was recrystallized from dichloromethane/hexane to give colorless crystals of the title compound suitable for X-ray crystallographic study (2.05 g, 6.78 mmol, 77%). mp: 511–513 K.

Refinement

All H atoms were located from difference maps and refined isotropically.

Figures

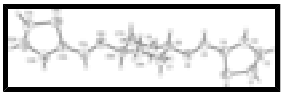


Fig. 1. Molecular structure of the title compound showing 50% probability displacement ellipsoids. Symmetry code for the atoms with A in their labels: -*x*+2, -*y*+2, -*z*+2.

N^1,*N*^{4}-Bis(2-thienylmethylene)cyclohexane-1,4-diamine

Crystal data

$C_{16}H_{18}N_2S_2$

$M_r = 302.44$

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

$a = 6.2173$ (4) Å

$b = 7.4999$ (5) Å

$c = 17.1289$ (12) Å

$\beta = 97.047$ (3)°

$V = 792.67$ (9) Å^3

$Z = 2$

$F_{000} = 320$

$D_x = 1.267$ Mg m^{-3}

Mo *K*α radiation, λ = 0.71073 Å

Cell parameters from 5365 reflections

θ = 2.4–28.2°

$\mu = 0.33$ mm^{-1}

$T = 296$ K

Block, colourless

0.46 × 0.24 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer

Radiation source: sealed tube

Monochromator: graphite

$T = 296$ K

φ and ω scans

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

$T_{min} = 0.864$, $T_{max} = 0.937$

9045 measured reflections

1889 independent reflections

1590 reflections with $I > 2\sigma(I)$

$R_{int} = 0.022$

$\theta_{max} = 28.3$°

$\theta_{min} = 2.4$°

$h = -8 \rightarrow 8$

$k = -8 \rightarrow 9$

$l = -22 \rightarrow 19$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.032$

$wR(F^2) = 0.089$

$S = 1.02$

1889 reflections

127 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0484P)^2 + 0.1406P]$
where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{max} = 0.19$ e Å^{-3}

$\Delta\rho_{min} = -0.18$ e Å^{-3}

Extinction correction: none

Special details

Experimental. IR (KBr, cm^{-1}): 3443 (w), 3103 (w), 2923 (*m*), 2852 (w), 1626 (*s*), 1430 (*m*), 1306 (w), 1210 (*m*), 1084 (*m*), 944 (*m*), 847 (*m*), 730 (*s*), 498 (*m*).

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	$U_{\rm iso}$*/$U_{\rm eq}$
S1	0.65706 (5)	0.42717 (5)	0.78995 (2)	0.04947 (14)
N1	0.78346 (18)	0.71526 (15)	0.91441 (7)	0.0458 (3)
C1	0.4495 (3)	0.2897 (2)	0.75469 (10)	0.0554 (4)
C2	0.2791 (3)	0.3032 (2)	0.79591 (10)	0.0575 (4)
C3	0.3138 (2)	0.42688 (19)	0.85814 (9)	0.0488 (3)
C4	0.51420 (19)	0.50601 (18)	0.86246 (7)	0.0396 (3)
C5	0.6024 (2)	0.64140 (18)	0.91770 (7)	0.0412 (3)
C6	0.8511 (2)	0.85271 (18)	0.97279 (8)	0.0430 (3)
C7	0.8308 (3)	1.0366 (2)	0.93470 (10)	0.0527 (4)
C8	1.0844 (2)	0.8189 (2)	1.00708 (10)	0.0516 (3)
H1	0.465 (3)	0.218 (2)	0.7107 (11)	0.069 (5)*
H2	0.157 (3)	0.239 (3)	0.7840 (10)	0.070 (5)*
H3	0.218 (2)	0.4550 (19)	0.8910 (10)	0.051 (4)*
H5	0.518 (2)	0.6691 (19)	0.9570 (9)	0.050 (4)*
H6	0.761 (2)	0.847 (2)	1.0152 (9)	0.053 (4)*
H7A	0.687 (3)	1.058 (2)	0.9140 (12)	0.074 (6)*
H7B	0.909 (3)	1.037 (2)	0.8896 (11)	0.061 (5)*
H8B	1.176 (3)	0.821 (2)	0.9662 (10)	0.056 (4)*
H8A	1.104 (3)	0.705 (2)	1.0311 (10)	0.059 (4)*

Atomic displacement parameters (Å^2)

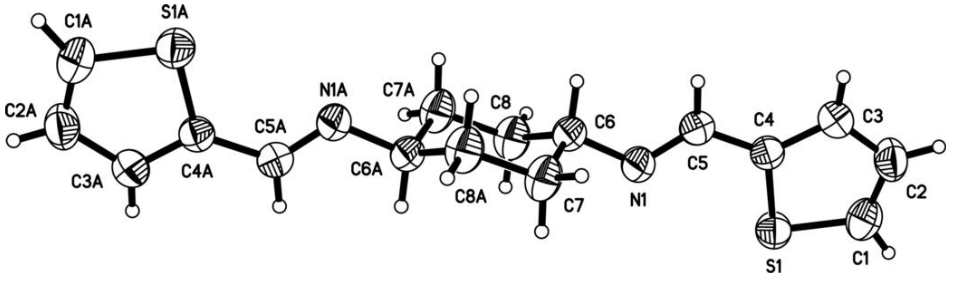
	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0449 (2)	0.0531 (2)	0.0512 (2)	−0.00126 (14)	0.00905 (15)	−0.00792 (15)
N1	0.0473 (6)	0.0465 (6)	0.0443 (6)	−0.0070 (5)	0.0087 (5)	−0.0088 (5)
C1	0.0617 (9)	0.0484 (8)	0.0545 (8)	−0.0023 (7)	0.0005 (7)	−0.0112 (7)
C2	0.0523 (8)	0.0552 (9)	0.0631 (9)	−0.0150 (7)	−0.0003 (7)	−0.0052 (7)
C3	0.0445 (7)	0.0532 (8)	0.0491 (7)	−0.0066 (6)	0.0077 (6)	−0.0008 (6)
C4	0.0400 (6)	0.0389 (6)	0.0393 (6)	0.0006 (5)	0.0035 (5)	0.0025 (5)
C5	0.0418 (6)	0.0428 (7)	0.0389 (6)	0.0016 (5)	0.0051 (5)	0.0000 (5)
C6	0.0450 (7)	0.0441 (7)	0.0407 (6)	−0.0041 (5)	0.0084 (5)	−0.0076 (6)
C7	0.0531 (8)	0.0492 (8)	0.0518 (8)	0.0023 (6)	−0.0091 (7)	−0.0035 (6)
C8	0.0544 (8)	0.0397 (8)	0.0584 (9)	0.0050 (6)	−0.0030 (7)	−0.0044 (6)

Geometric parameters (Å, °)

S1—C1	1.7038 (16)	C5—H5	0.928 (15)
S1—C4	1.7181 (13)	C6—C8	1.518 (2)
N1—C5	1.2619 (16)	C6—C7	1.524 (2)
N1—C6	1.4616 (16)	C6—H6	0.971 (16)
C1—C2	1.347 (2)	C7—C8^{i}	1.522 (2)
C1—H1	0.941 (18)	C7—H7A	0.94 (2)
C2—C3	1.410 (2)	C7—H7B	0.960 (19)
C2—H2	0.900 (18)	C8—C7^{i}	1.522 (2)
C3—C4	1.3736 (18)	C8—H8B	0.954 (16)
C3—H3	0.894 (15)	C8—H8A	0.951 (17)
C4—C5	1.4492 (19)		
C1—S1—C4	91.62 (7)	N1—C6—C7	110.09 (11)
C5—N1—C6	117.53 (11)	C8—C6—C7	109.96 (12)
C2—C1—S1	112.26 (12)	N1—C6—H6	109.6 (9)
C2—C1—H1	129.0 (11)	C8—C6—H6	108.2 (9)
S1—C1—H1	118.7 (11)	C7—C6—H6	109.8 (9)
C1—C2—C3	112.85 (14)	C8^{i}—C7—C6	111.09 (12)
C1—C2—H2	122.3 (11)	C8^{i}—C7—H7A	111.4 (11)
C3—C2—H2	124.8 (11)	C6—C7—H7A	110.0 (11)
C4—C3—C2	112.27 (13)	C8^{i}—C7—H7B	111.0 (11)
C4—C3—H3	122.4 (10)	C6—C7—H7B	108.7 (10)
C2—C3—H3	125.3 (10)	H7A—C7—H7B	104.5 (15)
C3—C4—C5	127.31 (12)	C6—C8—C7^{i}	111.86 (12)
C3—C4—S1	110.99 (11)	C6—C8—H8B	109.9 (10)
C5—C4—S1	121.69 (9)	C7^{i}—C8—H8B	106.4 (10)
N1—C5—C4	123.14 (12)	C6—C8—H8A	112.4 (10)
N1—C5—H5	121.5 (10)	C7^{i}—C8—H8A	110.0 (10)
C4—C5—H5	115.3 (10)	H8B—C8—H8A	106.0 (13)
N1—C6—C8	109.16 (11)		

Symmetry codes: (i) $-x+2$, $-y+2$, $-z+2$.

Fig. 1


C1A
S1A
C2A
C3A
C4A
C5A
N1A
C7A
C6A
C8
C8A
C6
C7
N1
C5
C4
C3
C2
S1
C1