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# 2-(Thiophen-2-yl)-*N*-(4-{(*E*)-[2-(thiophen-2-yl)ethyl]iminomethyl}benzylidene)-ethanamine

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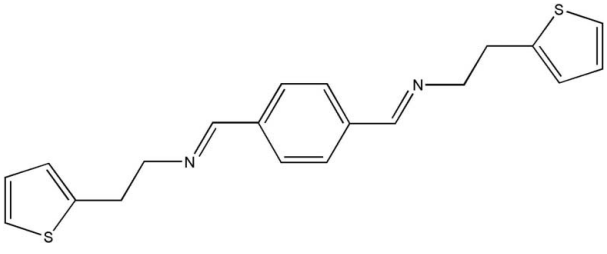
Key indicators: single-crystal X-ray study; $T$ = 173 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.057; $wR$ factor = 0.181; data-to-parameter ratio = 20.5.

In the crystal of the centrosymmetric title compound, $C_{20}H_{20}N_2S_2$, molecules are linked by head-to-tail C—H···N hydrogen bonds, resulting in chains extending along the *a* axis. Three additional C—H···$\pi$ intermolecular interactions give rise to a herringbone packing motif which extends along the *c* axis. The C—H···N interactions provide links between the sheets.

## Related literature

For related literature on bidendate Schiff base ligands, see: Chakraborty *et al.* (1999); Haga & Koizumi (1985).



## Experimental

### Crystal data

$C_{20}H_{20}N_2S_2$
$M_r$ = 352.52
Monoclinic, $C2/c$
$a$ = 9.8592 (10) Å
$b$ = 7.1533 (6) Å
$c$ = 25.678 (2) Å
$\beta$ = 96.646 (5)°
$V$ = 1798.8 (3) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.30 mm$^{-1}$
$T$ = 173 K
0.22 × 0.2 × 0.04 mm

### Data collection

Nonius Kappa CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2007)
$T_{min}$ = 0.925, $T_{max}$ = 0.988
16248 measured reflections
2230 independent reflections
1679 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.045

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.181
$S$ = 1.08
2230 reflections
109 parameters
14 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.80 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.42 e Å$^{-3}$

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

*Cg*1 andCg2 are the centroids of the thiophene and benzene rings, respectively.

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C4—H4···N8$^{i}$ | 0.95 | 2.61 | 3.514 (3) | 159 |
| C2—H2···*Cg*1$^{ii}$ | 0.95 | 2.79 | 3.702 (3) | 161 |
| C6—H6*A*···*Cg*2$^{iii}$ | 0.99 | 2.72 | 3.515 (3) | 137 |
| C6—H6*A*···*Cg*2$^{iv}$ | 0.99 | 2.72 | 3.515 (3) | 137 |

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

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2005) and *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GO2007).

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# supplementary materials

*Acta Cryst.* (2011). E**67**, o922 [ **doi:10.1107/S1600536811009810** ]

# 2-(Thiophen-2-yl)-*N*-(4-{(*E*)-[2-(thiophen-2-yl)ethyl]iminomethyl}benzylidene)ethanamine

**H. Chiririwa, R. Meijboom and B. Omondi**

## Comment

The title compound belongs to a class of tetradentate ligands. To the best of our knowledge, this is the first example of a neutral thiophenyldimine-based bridging ligand. This compound is a potential tetra-coordinate ligand but on complexation the compound will probably behave as a bidentate ligand as the sulfur, on the thiophene, has weak donor capacity towards co-ordination for majority of metal ions. Besides its use as a ligand, it is interesting from the crystal engineering point of view for the analysis of the packing mode of (I).

Compound (I) crystallizes with half a molecule in the asymmetric unit, with the other half generated through symmetry located in the center of the phenyl ring (Fig. 1). The phenyl ring together with the atoms C7—N8—C9 and the thiophene ring together with the atom C6 are planar with N8 and C5 deviating the most from the planes by 0.018 (2) Å and 0.010 (2) Å respectively. The two planes are close to parallel, the angle between them being 9.3 (1)°. Bond distances and angles in (I) are as expected from the chemical bonding.

The crystal structure of (I) is composed of head-to-tail C—H···N hydrogen bonded chains (Table 1) that extend in the crystallographic *a* axis (Fig. 2). Additionally, the phenyl and thiophen rings are involved in C—H···π intermolecular interactions that result in a herringbone motif that spreads along the crystallographic *c* axis (Fig. 3). The C—H···N interactions are found to connect these herringbone sheets along the *a* axis.,

## Experimental

A solution of benzene 1,4-dicarboxaldehyde (0.50 g, 3.73 mmol) in methanol (10 ml) was added dropwise to a stirred solution of 2-thiophenylethylamine (0.95 g, 7.42 mmol) in methanol (10 ml). The mixture was stirred at room temperature for *ca* 16 h. The precipitate was filtered off and washed with diethylether and dried under vacuum for 4 h affording a fine shiny white powder in 80% yield. *M*.p.: 240–242 °C. Recrystallization was done by slow diffusion of $Et_2O$ into a concentrated $CH_2Cl_2$ solution of the white powder to give colorless crystals fo (I).

## Refinement

The methine and aromatic H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H = 0.95 Å and $U_{iso}$(H) = 1.2$U_{eq}$(C) for aromatic, C—H = 0.99 Å and $U_{iso}$(H) = 1.2$U_{eq}$(C) for $CH_2$ C—H = 0.95 Å and $U_{iso}$(H) = 1.2$U_{eq}$(C) for CH.

## Figures

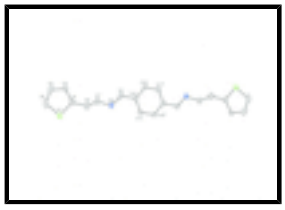

Fig. 1. View of (I) (50% probability displacement ellipsoids) with H atoms presented as small spheres of arbitrary radii.

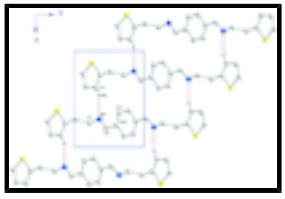

Fig. 2. C—H···N hydrogen bond interactions in the crystal structure of (I). [Symmetry operators: (i) = -1/2 + *x*, 1/2 + *y*, *z*]

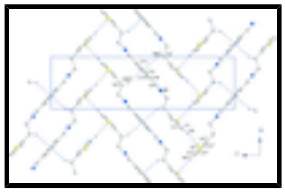

Fig. 3. Sheets of C—H···π intermolecular interactions between molecules alligned along the *bc* face.

## 2-(Thiophen-2-yl)-*N*-(4-{(*E*)-[2-(thiophen-2- yl)ethyl]iminomethyl}benzylidene)ethanamine

*Crystal data*

| | |
|---|---|
| $C_{20}H_{20}N_2S_2$ | $F(000) = 744$ |
| $M_r = 352.52$ | $D_x = 1.302$ Mg m$^{-3}$ |
| Monoclinic, *C*2/*c* | Mo *K*α radiation, λ = 0.71073 Å |
| Hall symbol: -C 2yc | Cell parameters from 34223 reflections |
| *a* = 9.8592 (10) Å | θ = 3.2–28.3° |
| *b* = 7.1533 (6) Å | μ = 0.30 mm$^{-1}$ |
| *c* = 25.678 (2) Å | *T* = 173 K |
| β = 96.646 (5)° | Plate, colourless |
| *V* = 1798.8 (3) Å$^3$ | 0.22 × 0.2 × 0.04 mm |
| *Z* = 4 | |

*Data collection*

| | |
|---|---|
| Nonius Kappa CCD diffractometer | $R_{int} = 0.045$ |
| graphite | $\theta_{max} = 28.3°$, $\theta_{min} = 3.2°$ |
| 1.0° ω scans, 60s | *h* = −13→13 |
| 16248 measured reflections | *k* = −9→9 |
| 2230 independent reflections | *l* = −34→34 |
| 1679 reflections with $I > 2\sigma(I)$ | |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Primary atom site location: structure-invariant direct methods |
| Least-squares matrix: full | Secondary atom site location: difference Fourier map |
| $R[F^2 > 2\sigma(F^2)] = 0.057$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.181$ | H-atom parameters constrained |
| $S = 1.08$ | $w = 1/[\sigma^2(F_o^2) + (0.0971P)^2 + 3.1807P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| 2230 reflections | $(\Delta/\sigma)_{max} < 0.001$ |
| 109 parameters | $\Delta\rho_{max} = 0.80$ e Å$^{-3}$ |
| 14 restraints | $\Delta\rho_{min} = -0.42$ e Å$^{-3}$ |

*Special details*

**Experimental**. The intensity data was collected on a Nonius Kappa CCD diffractometer using an exposure time of 60 sec/per frame. Analytical data: IR (KBr): 1613?cm-1 (C=N, imine); 1H NMR: (CDCl3) δ H 8.23 (d, 2H) 7.76 (s, 2H) 7.13 (dd, 2H) 6.92 (dd, 2H) 6.84 (dd, 4H) 3.91 (dt, 4H) 3.25 (t, 4H); Anal. calcd. for C20H20N2S2: C, 68.14%; H, 5.72%; N, 7.95%; S, 18.19; Found: C, 68.19%; H, 5.52%; N, 7.72%; S, 18.44; EI—MS: m/z 351.76 [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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| S1 | 0.62222 (7) | −0.24504 (10) | 0.31079 (3) | 0.0357 (3) |
| C2 | 0.7356 (3) | −0.3763 (4) | 0.28220 (10) | 0.0400 (7) |
| H2 | 0.711 | −0.4684 | 0.2562 | 0.048* |
| C3 | 0.8651 (3) | −0.3341 (4) | 0.30095 (10) | 0.0355 (6) |
| H3 | 0.9419 | −0.3955 | 0.2896 | 0.043* |
| C4 | 0.8766 (2) | −0.1915 (3) | 0.33866 (8) | 0.0194 (4) |
| H4 | 0.9607 | −0.1441 | 0.3552 | 0.023* |
| C5 | 0.7453 (2) | −0.1270 (3) | 0.34886 (9) | 0.0230 (5) |
| C6 | 0.7117 (3) | 0.0246 (3) | 0.38573 (10) | 0.0286 (5) |
| H6A | 0.7616 | 0.0005 | 0.4208 | 0.034* |
| H6B | 0.6128 | 0.0204 | 0.3891 | 0.034* |
| C7 | 0.7483 (3) | 0.2186 (3) | 0.36770 (10) | 0.0266 (5) |
| H7A | 0.8479 | 0.2266 | 0.366 | 0.032* |
| H7B | 0.701 | 0.2431 | 0.3322 | 0.032* |

| | | | | |
|---|---|---|---|---|
| N8 | 0.7077 (2) | 0.3574 (3) | 0.40432 (8) | 0.0257 (5) |
| C9 | 0.8002 (2) | 0.4632 (3) | 0.42630 (9) | 0.0232 (5) |
| H9 | 0.891 | 0.4474 | 0.4181 | 0.028* |
| C10 | 0.7729 (2) | 0.6096 (3) | 0.46404 (9) | 0.0220 (5) |
| C11 | 0.6408 (2) | 0.6450 (3) | 0.47609 (9) | 0.0235 (5) |
| H11 | 0.5661 | 0.5741 | 0.4598 | 0.028* |
| C12 | 0.8813 (2) | 0.7162 (3) | 0.48807 (9) | 0.0234 (5) |
| H12 | 0.9713 | 0.6935 | 0.4798 | 0.028* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| S1 | 0.0339 (4) | 0.0352 (4) | 0.0373 (4) | −0.0022 (3) | 0.0007 (3) | −0.0044 (3) |
| C2 | 0.073 (2) | 0.0227 (13) | 0.0245 (12) | −0.0002 (13) | 0.0045 (13) | −0.0058 (10) |
| C3 | 0.0487 (16) | 0.0284 (13) | 0.0314 (13) | 0.0127 (12) | 0.0133 (12) | −0.0009 (9) |
| C4 | 0.0167 (9) | 0.0190 (10) | 0.0217 (10) | 0.0007 (8) | −0.0012 (8) | 0.0046 (7) |
| C5 | 0.0288 (11) | 0.0183 (11) | 0.0230 (11) | 0.0037 (9) | 0.0073 (9) | 0.0019 (9) |
| C6 | 0.0396 (14) | 0.0218 (12) | 0.0262 (12) | 0.0020 (10) | 0.0118 (10) | −0.0013 (9) |
| C7 | 0.0309 (13) | 0.0223 (12) | 0.0282 (12) | −0.0010 (9) | 0.0096 (10) | −0.0063 (9) |
| N8 | 0.0293 (11) | 0.0214 (10) | 0.0269 (10) | 0.0010 (8) | 0.0055 (8) | −0.0061 (8) |
| C9 | 0.0260 (11) | 0.0207 (11) | 0.0238 (11) | 0.0007 (9) | 0.0073 (9) | −0.0010 (9) |
| C10 | 0.0269 (12) | 0.0181 (11) | 0.0211 (10) | −0.0002 (9) | 0.0028 (8) | −0.0003 (9) |
| C11 | 0.0236 (11) | 0.0220 (11) | 0.0248 (11) | −0.0024 (9) | 0.0026 (9) | −0.0030 (9) |
| C12 | 0.0200 (11) | 0.0245 (12) | 0.0263 (11) | 0.0009 (9) | 0.0048 (9) | −0.0015 (9) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| S1—C2 | 1.691 (3) | C7—N8 | 1.455 (3) |
| S1—C5 | 1.693 (2) | C7—H7A | 0.99 |
| C2—C3 | 1.345 (4) | C7—H7B | 0.99 |
| C2—H2 | 0.95 | N8—C9 | 1.266 (3) |
| C3—C4 | 1.402 (4) | C9—C10 | 1.472 (3) |
| C3—H3 | 0.95 | C9—H9 | 0.95 |
| C4—C5 | 1.427 (3) | C10—C11 | 1.397 (3) |
| C4—H4 | 0.95 | C10—C12 | 1.397 (3) |
| C5—C6 | 1.501 (3) | C11—C12$^{i}$ | 1.388 (3) |
| C6—C7 | 1.520 (3) | C11—H11 | 0.95 |
| C6—H6A | 0.99 | C12—C11$^{i}$ | 1.388 (3) |
| C6—H6B | 0.99 | C12—H12 | 0.95 |
| C2—S1—C5 | 93.55 (13) | N8—C7—C6 | 109.48 (19) |
| C3—C2—S1 | 111.6 (2) | N8—C7—H7A | 109.8 |
| C3—C2—H2 | 124.2 | C6—C7—H7A | 109.8 |
| S1—C2—H2 | 124.2 | N8—C7—H7B | 109.8 |
| C2—C3—C4 | 114.1 (2) | C6—C7—H7B | 109.8 |
| C2—C3—H3 | 122.9 | H7A—C7—H7B | 108.2 |
| C4—C3—H3 | 122.9 | C9—N8—C7 | 117.3 (2) |
| C3—C4—C5 | 111.0 (2) | N8—C9—C10 | 122.8 (2) |
| C3—C4—H4 | 124.5 | N8—C9—H9 | 118.6 |

| | | | |
|---|---|---|---|
| C5—C4—H4 | 124.5 | C10—C9—H9 | 118.6 |
| C4—C5—C6 | 128.3 (2) | C11—C10—C12 | 119.2 (2) |
| C4—C5—S1 | 109.73 (17) | C11—C10—C9 | 121.4 (2) |
| C6—C5—S1 | 121.93 (18) | C12—C10—C9 | 119.4 (2) |
| C5—C6—C7 | 113.0 (2) | C12$^{i}$—C11—C10 | 119.9 (2) |
| C5—C6—H6A | 109 | C12$^{i}$—C11—H11 | 120 |
| C7—C6—H6A | 109 | C10—C11—H11 | 120 |
| C5—C6—H6B | 109 | C11$^{i}$—C12—C10 | 120.8 (2) |
| C7—C6—H6B | 109 | C11$^{i}$—C12—H12 | 119.6 |
| H6A—C6—H6B | 107.8 | C10—C12—H12 | 119.6 |
| C5—S1—C2—C3 | 0.4 (2) | C5—C6—C7—N8 | 177.7 (2) |
| S1—C2—C3—C4 | −0.9 (3) | C6—C7—N8—C9 | 121.8 (2) |
| C2—C3—C4—C5 | 1.1 (3) | C7—N8—C9—C10 | 179.9 (2) |
| C3—C4—C5—C6 | −179.2 (2) | N8—C9—C10—C11 | −2.7 (4) |
| C3—C4—C5—S1 | −0.8 (2) | N8—C9—C10—C12 | 177.7 (2) |
| C2—S1—C5—C4 | 0.25 (18) | C12—C10—C11—C12$^{i}$ | −0.5 (4) |
| C2—S1—C5—C6 | 178.7 (2) | C9—C10—C11—C12$^{i}$ | 179.9 (2) |
| C4—C5—C6—C7 | 69.3 (3) | C11—C10—C12—C11$^{i}$ | 0.6 (4) |
| S1—C5—C6—C7 | −108.9 (2) | C9—C10—C12—C11$^{i}$ | −179.9 (2) |

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

*Hydrogen-bond geometry (Å, °)*

Cg1 andCg2 are the centroids of the thiophene and benzene rings, respectively.

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C4—H4···N8$^{ii}$ | 0.95 | 2.61 | 3.514 (3) | 159 |
| C2—H2···Cg1$^{iii}$ | 0.95 | 2.79 | 3.702 (3) | 161 |
| C6—H6A···Cg2$^{iv}$ | 0.99 | 2.72 | 3.515 (3) | 137 |
| C6—H6A···Cg2$^{v}$ | 0.99 | 2.72 | 3.515 (3) | 137 |

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

**Fig. 1**

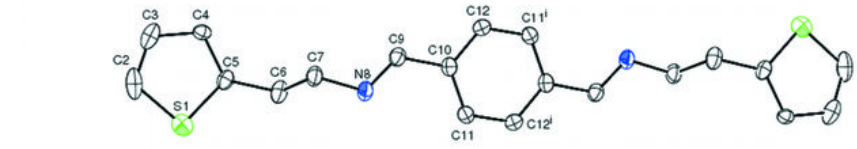

**Fig. 2**

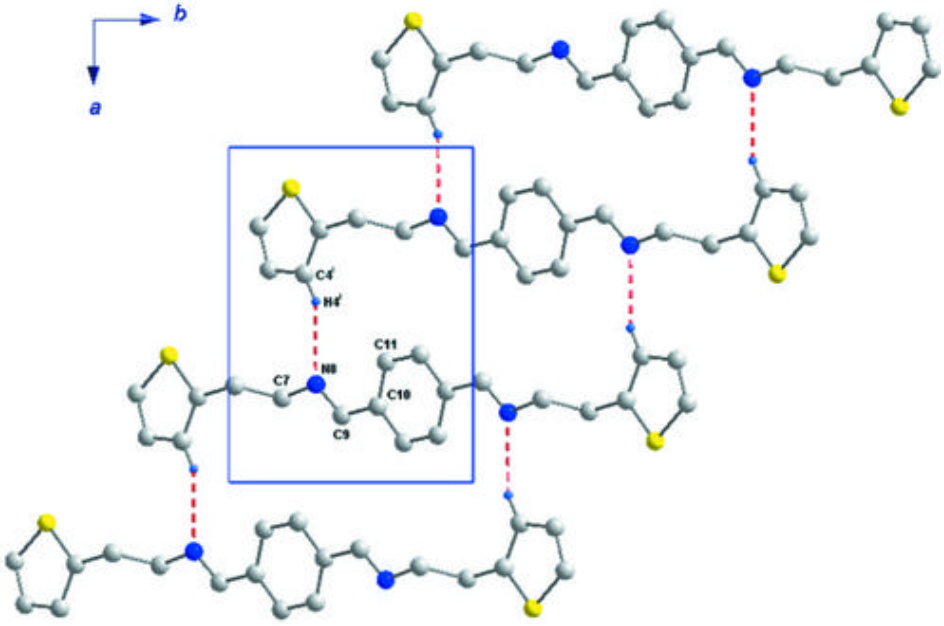

**Fig. 3**