organic compounds
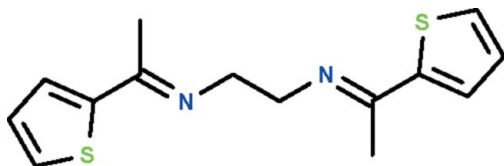
N,N′-Bis[1-(thiophen-2-yl)ethylidene]-ethane-1,2-diamine

Abdullah M. Asiri,[a,b] Abdulrahman O. Al-Youbi,[a] Hassan M. Faidallah,[a] Khalid A. Alamry[a] and Seik Weng Ng[c,a]*

[a]Chemistry Department, Faculty of Science, King Abdulaziz University, PO Box 80203 Jeddah, Saudi Arabia, [b]Center of Excellence for Advanced Materials Research, King Abdulaziz University, PO Box 80203 Jeddah, Saudi Arabia, and [c]Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia
Correspondence e-mail: seikweng@um.edu.my

Molecules of the title compound, $C_{14}H_{16}N_2S_2$, have a centre of inversion in the middle of the –CH_2–CH_2– bond; the $(C_4H_3S)(CH_3)C{=}N{-}CH_2$– moiety is almost planar (r.m.s. deviation for non-H atoms 0.027 Å).

Related literature

For a related transition metal adduct, see: Modder *et al.* (1995).

Experimental

Crystal data

$C_{14}H_{16}N_2S_2$	$V = 674.68$ (5) Å³
$M_r = 276.41$	$Z = 2$
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
$a = 5.5831$ (3) Å	$\mu = 0.38$ mm⁻¹
$b = 9.3939$ (4) Å	$T = 100$ K
$c = 12.9202$ (5) Å	$0.25 \times 0.20 \times 0.15$ mm
$\beta = 95.342$ (4)°	

Data collection

Agilent SuperNova Dual diffractometer with Atlas detector	3036 measured reflections
	1495 independent reflections
	1244 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010) $T_{min} = 0.912$, $T_{max} = 0.946$	$R_{int} = 0.028$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.035$	83 parameters
$wR(F^2) = 0.090$	H-atom parameters constrained
$S = 1.04$	$\Delta\rho_{max} = 0.44$ e Å⁻³
1495 reflections	$\Delta\rho_{min} = -0.40$ e Å⁻³

Data collection: *CrysAlis PRO* (Agilent, 2010); 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: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

References

Agilent (2010). *CrysAlis PRO*. Agilent Technologies, Yarnton, Oxfordshire, England.
Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
Modder, J. F., Leijen, R. J., Vrieze, K., Smeets, W. J. J., Spek, A. L. & van Koten, G. (1995). *J. Chem. Soc. Dalton Trans.* pp. 4021–4028.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

supplementary materials

N,N'-Bis[1-(thiophen-2-yl)ethylidene]ethane-1,2-diamine

A. M. Asiri, A. O. Al-Youbi, H. M. Faidallah, K. A. Alamry and **S. W. Ng**

Comment

A large number of transition metal adducts of Schiff bases derived by condensing ethylenediamine with a ketone have been reported; in these adducts, the ligand typically functions in a chelating mode. However, there are few studies on the title Schiff base (Scheme I), and only one crystal structure study has been reported (Modder *et al.*, 1995). The $C_{14}H_{16}N_2S_2$ molecule lies on a center-of-inversion (Fig. 1); the $(C_4H_3S)(CH_3)C= N–CH_2–$ moiety is planar, and the chain connecting the two aromatic rings adopts an extended zigzag conformation [C=N–C–C 88.1 (2)°].

Experimental

Ethylenediamine (0.6 g, 10 mmol) and 2-acetylthiophene (0.7 g, 10 mmol) in dry benzene (50 ml) were refluxed in a Dean–Stark apparatus until no more water was collected (in about 2 h). The solvent was removed and the solid that separated was collected and recystallized from ethanol.

Refinement

H-atoms were placed in calculated positions [C—H 0.95–0.98 Å, $U_{iso}(H) = 1.2$–$1.5U_{eq}(C)$] and were included in the refinement in the riding model approximation.
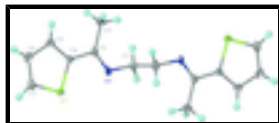
Figures

Fig. 1. Anisotropic displacement ellipsoid plot (Barbour, 2001) of $C_{14}H_{16}N_2S_2$ at the 70% probability level; H atoms are drawn as spheres of arbitrary radius. The molecule lies on a center-of-inversion.

N,N'-Bis[1-(thiophen-2-yl)ethylidene]ethane-1,2-diamine

Crystal data

$C_{14}H_{16}N_2S_2$	$F(000) = 292$
$M_r = 276.41$	$D_x = 1.361$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2yn	Cell parameters from 1562 reflections
$a = 5.5831$ (3) Å	$\theta = 2.7$–29.1°
$b = 9.3939$ (4) Å	$\mu = 0.38$ mm^{-1}
$c = 12.9202$ (5) Å	$T = 100$ K
$\beta = 95.342$ (4)°	Prism, colourless
$V = 674.68$ (5) Å^3	$0.25 \times 0.20 \times 0.15$ mm

$Z = 2$

Data collection

Agilent SuperNova Dual diffractometer with Atlas detector	1495 independent reflections
Radiation source: SuperNova (Mo) X-ray Source mirror	1244 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.028$
Detector resolution: 10.4041 pixels mm^{-1}	$\theta_{max} = 27.5°$, $\theta_{min} = 2.7°$
ω scans	$h = -5 \rightarrow 7$
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)	$k = -12 \rightarrow 9$
$T_{min} = 0.912$, $T_{max} = 0.946$	$l = -16 \rightarrow 16$
3036 measured reflections	

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.035$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.090$	H-atom parameters constrained
$S = 1.04$	$w = 1/[\sigma^2(F_o^2) + (0.0323P)^2 + 0.5515P]$ where $P = (F_o^2 + 2F_c^2)/3$
1495 reflections	$(\Delta/\sigma)_{max} = 0.001$
83 parameters	$\Delta\rho_{max} = 0.44$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.40$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	$U_{iso}*/U_{eq}$
S1	0.66665 (8)	0.89024 (5)	0.29153 (3)	0.01547 (15)
N1	0.5827 (3)	0.68042 (16)	0.45253 (11)	0.0141 (3)
C1	0.5756 (4)	0.56063 (19)	0.52477 (14)	0.0163 (4)
H1A	0.5065	0.5926	0.5887	0.020*
H1B	0.7412	0.5264	0.5446	0.020*
C2	0.4204 (3)	0.77676 (19)	0.44907 (13)	0.0122 (4)
C3	0.2062 (3)	0.7852 (2)	0.51239 (14)	0.0168 (4)
H3A	0.0574	0.7795	0.4659	0.025*
H3B	0.2102	0.8756	0.5503	0.025*
H3C	0.2120	0.7060	0.5619	0.025*
C4	0.4397 (3)	0.89283 (18)	0.37376 (13)	0.0113 (4)
C5	0.2995 (3)	1.0123 (2)	0.35677 (14)	0.0144 (4)
H5	0.1648	1.0330	0.3940	0.017*
C6	0.3775 (3)	1.1021 (2)	0.27724 (14)	0.0163 (4)
H6	0.3017	1.1893	0.2561	0.020*
C7	0.5731 (4)	1.0482 (2)	0.23535 (13)	0.0171 (4)

H7	0.6491	1.0930	0.1812	0.021*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0182 (3)	0.0134 (3)	0.0156 (2)	0.00002 (19)	0.00604 (18)	0.00080 (18)
N1	0.0170 (8)	0.0116 (7)	0.0135 (7)	−0.0028 (7)	0.0004 (6)	0.0025 (6)
C1	0.0195 (10)	0.0134 (9)	0.0154 (8)	−0.0006 (8)	−0.0007 (7)	0.0034 (8)
C2	0.0132 (9)	0.0120 (9)	0.0111 (8)	−0.0033 (7)	−0.0008 (7)	−0.0025 (7)
C3	0.0148 (9)	0.0213 (10)	0.0147 (8)	−0.0010 (8)	0.0030 (7)	0.0010 (8)
C4	0.0118 (8)	0.0118 (9)	0.0102 (8)	−0.0018 (7)	0.0006 (6)	−0.0013 (7)
C5	0.0131 (9)	0.0150 (9)	0.0151 (8)	0.0000 (8)	0.0007 (7)	−0.0015 (7)
C6	0.0185 (10)	0.0134 (9)	0.0156 (8)	0.0012 (8)	−0.0055 (7)	0.0013 (7)
C7	0.0239 (10)	0.0146 (9)	0.0123 (8)	−0.0052 (8)	−0.0006 (7)	0.0019 (7)

Geometric parameters (Å, °)

S1—C7	1.712 (2)	C3—H3A	0.9800
S1—C4	1.7278 (17)	C3—H3B	0.9800
N1—C2	1.279 (2)	C3—H3C	0.9800
N1—C1	1.465 (2)	C4—C5	1.375 (2)
C1—C1^i	1.523 (4)	C5—C6	1.428 (3)
C1—H1A	0.9900	C5—H5	0.9500
C1—H1B	0.9900	C6—C7	1.361 (3)
C2—C4	1.472 (2)	C6—H6	0.9500
C2—C3	1.513 (2)	C7—H7	0.9500
C7—S1—C4	92.09 (9)	H3A—C3—H3C	109.5
C2—N1—C1	120.31 (15)	H3B—C3—H3C	109.5
N1—C1—C1^i	110.72 (18)	C5—C4—C2	129.41 (16)
N1—C1—H1A	109.5	C5—C4—S1	110.65 (13)
C1^i—C1—H1A	109.5	C2—C4—S1	119.94 (13)
N1—C1—H1B	109.5	C4—C5—C6	112.95 (16)
C1^i—C1—H1B	109.5	C4—C5—H5	123.5
H1A—C1—H1B	108.1	C6—C5—H5	123.5
N1—C2—C4	116.88 (15)	C7—C6—C5	112.08 (17)
N1—C2—C3	127.72 (16)	C7—C6—H6	124.0
C4—C2—C3	115.39 (16)	C5—C6—H6	124.0
C2—C3—H3A	109.5	C6—C7—S1	112.24 (14)
C2—C3—H3B	109.5	C6—C7—H7	123.9
H3A—C3—H3B	109.5	S1—C7—H7	123.9
C2—C3—H3C	109.5		
C2—N1—C1—C1^i	88.1 (2)	C7—S1—C4—C5	0.05 (14)
C1—N1—C2—C4	−179.53 (15)	C7—S1—C4—C2	−179.44 (14)
C1—N1—C2—C3	−0.6 (3)	C2—C4—C5—C6	179.10 (17)
N1—C2—C4—C5	−176.62 (18)	S1—C4—C5—C6	−0.3 (2)
C3—C2—C4—C5	4.3 (3)	C4—C5—C6—C7	0.5 (2)
N1—C2—C4—S1	2.8 (2)	C5—C6—C7—S1	−0.5 (2)

| C3—C2—C4—S1 | −176.34 (13) | C4—S1—C7—C6 | 0.25 (15) |

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

Fig. 1