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# 6-(4-Aminophenyl)-2-ethoxy-4-(2-thienyl)nicotinonitrile

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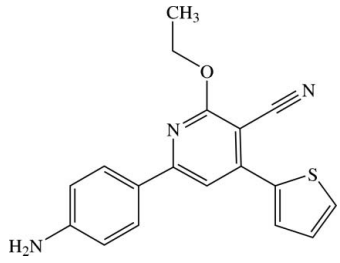
Key indicators: single-crystal X-ray study; $T$ = 100 K; mean $\sigma$(C–C) = 0.003 Å; disorder in main residue; $R$ factor = 0.038; $wR$ factor = 0.085; data-to-parameter ratio = 13.1.

In the title nicotinonitrile derivative, $C_{18}H_{15}N_3OS$, the central pyridyl ring makes dihedral angles of 25.22 (10) and 24.80 (16)° with the 4-aminophenyl and thiophene rings, respectively. The thiophene ring is disordered over two orientations by rotation around the C(thiophene)—C(pyridine) bond; the occupancies are 0.858 (2) and 0.142 (2). The ethoxy group is slightly twisted from the attached pyridyl ring [C—O—C—C torsion angle = 171.13 (16)°]. In the crystal structure, molecules are linked by N—H···N hydrogen bonds into chains along [010]. These chains are stacked along the $a$ axis. C—H···$\pi$ weak interactions involving the thiophene ring are observed.

## Related literature

For reference bond-length data, see: Allen *et al.* (1987). For the synthesis and applications of nicotinonitrile derivatives, see: Amr & Abdulla (2006); Borgna *et al.* (1993); Fun *et al.* (2009); Goda *et al.* (2004); Kamal *et al.* (2007); Malinka *et al.* (1998). For related structures, see: Chantrapromma *et al.* (2009, 2010); Fun *et al.* (2009). For the stability of the temperature controller used in the data collection, see Cosier & Glazer (1986).



## Experimental

### Crystal data

$C_{18}H_{15}N_3OS$
$M_r$ = 321.38
Orthorhombic, *Pbca*
$a$ = 7.0751 (12) Å
$b$ = 20.843 (4) Å
$c$ = 20.983 (4) Å
$V$ = 3094.3 (9) Å$^3$
$Z$ = 8
Mo $K\alpha$ radiation
$\mu$ = 0.22 mm$^{-1}$
$T$ = 100 K
0.35 × 0.11 × 0.04 mm

### Data collection

Bruker APEXII DUO CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
$T_{min}$ = 0.928, $T_{max}$ = 0.992
34805 measured reflections
3045 independent reflections
2188 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.092

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.085
$S$ = 1.05
3045 reflections
233 parameters
88 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.21 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.32 e Å$^{-3}$

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

*Cg*1 is the centroid of the major disorder component of the thiophene ring.

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N2—H1*N2*···N3$^{i}$ | 0.92 (2) | 2.29 (2) | 3.197 (3) | 168.2 (19) |
| C3—H3*A*···*Cg*1$^{ii}$ | 0.93 | 2.93 | 3.566 (6) | 127 |
| C12—H12*A*···*Cg*1$^{iii}$ | 0.93 | 2.78 | 3.430 (3) | 128 |

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; 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* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WN2394).

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# supporting information

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## 6-(4-Aminophenyl)-2-ethoxy-4-(2-thienyl)nicotinonitrile

**Hoong-Kun Fun, Suchada Chantrapromma, Thawanrat Kobkeatthawin, Mahesh Padaki and Arun M. Isloor**

### S1. Comment

Heterocyclic compounds containing the pyridine ring are reported to possess a diverse range of biological activities such as antimicrobial, antitumor and anti-inflammatory (Amr & Abdulla, 2006; Borgna *et al.*, 1993; Goda *et al.*, 2004; Kamal *et al.*, 2007; Malinka *et al.*, 1998) properties. Our research is aimed at the synthesis and preliminary biological (*in vitro*) and pharmacological (*in vivo*) screening, together with enzyme inhibitory activity, of the nicotinonitrile derivatives. The title compound, which is a substituted pyridine compound, was synthesized by cyclization of our previous chalcone derivative (Fun *et al.*, 2009) and malononitrile.

The molecule of the title compound, $C_{18}H_{15}N_3OS$, is not planar (Fig. 1). The central pyridyl ring is inclined to the 4-aminophenyl and thiophene rings with dihedral angles of 25.22 (10)° and 24.80 (16)°, respectively. The thiophene ring is disordered over two orientations by rotation around the C4—C5 bond, with occupancies of 0.858 (2) and 0.142 (1). The ethoxy group is twisted slightly from the attached pyridyl ring, as indicated by the torsion angles C14—O1—C16—C17 = 171.13 (16)° and C16—O1—C14—C15 = 179.01 (16) °. The bond distances agree with the literature values (Allen *et al.*, 1987) and are comparable with those for related structures (Chantrapromma *et al.*, 2009; 2010).

In the crystal structure, (Fig. 2), the molecules are linked by weak intermolecular N2—H1N2···N3 hydrogen bond (Table 1) into chains along [010]. These chains are stacked along the *a* axis. The crystal structure is further stabilized by C—H···$\pi$ interactions (Table 1); $Cg_1$ is the centroid of the C1–C4/S1 ring (major disorder component).

### S2. Experimental

(2*E*)-1-(4-Aminophenyl)-3-(2-thienyl)prop-2-en-1-one (0.34 g, 0.0015 mole) which was synthesized according to a previous procedure (Fun *et al.*, 2009) was added with continuous stirring to a freshly prepared sodium alkoxide solution (0.0014 mole of sodium in 100 ml of ethanol). Malononitrile (1.30 g, 0.02 mol) was then added with continuous stirring at room temperature until the precipitate separated out. The resulting solid was filtered (yield 68%). Yellow plate-shaped single crystals of the title compound suitable for *X*-ray structure determination were recrystalized from ethanol by the slow evaporation of the solvent at room temperature over several days. Mp. 470–471 K.

### S3. Refinement

The amino H atoms were located in difference maps and refined isotropically. The remaining H atoms were positioned geometrically and allowed to ride on their parent atoms, with d(C—H) = 0.93 Å for aromatic, 0.97 for $CH_2$ and 0.96 Å for $CH_3$ atoms. The $U_{iso}$ values were constrained to be $1.5U_{eq}$ of the carrier atom for methyl H atoms and $1.2U_{eq}$ for the remaining carbon-bound H atoms. A rotating group model was used for the methyl groups. Atoms S1, C1, C2, C3 of the thiophene ring are disordered over two positions by rotation about the C4—C5 bond; the occupancies are 0.858 (2) and 0.142 (2).

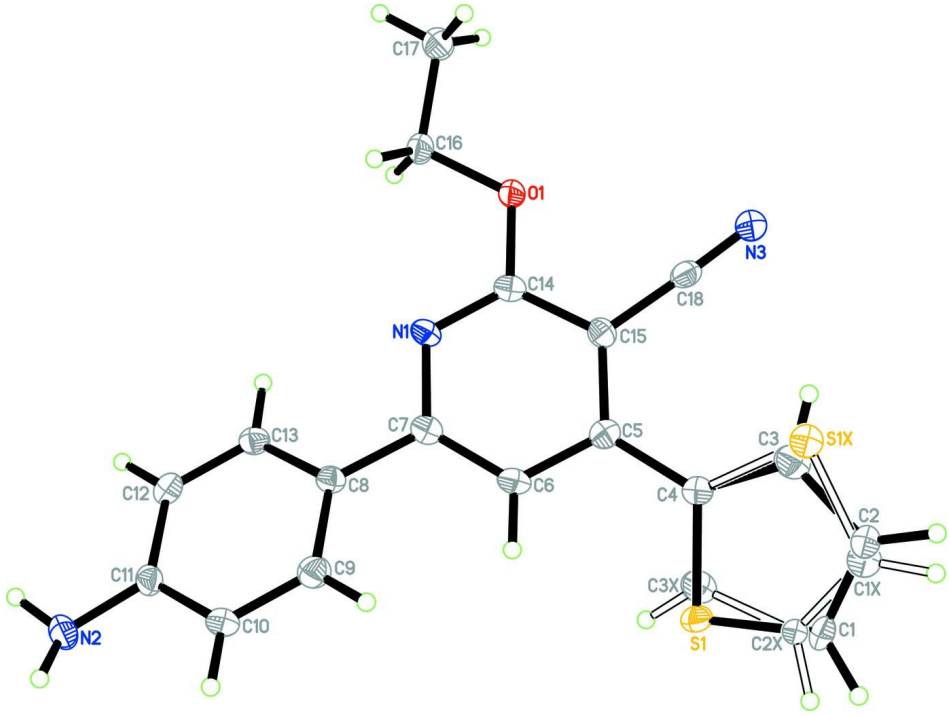


**Figure 1**

The molecular structure of the title compound, showing 50% probability displacement ellipsoids and the atom-numbering scheme. Hydrogen atoms are shown as spheres of arbitrary radius. The major and minor components of the disorder are shown by shaded and open bonds, respectively.

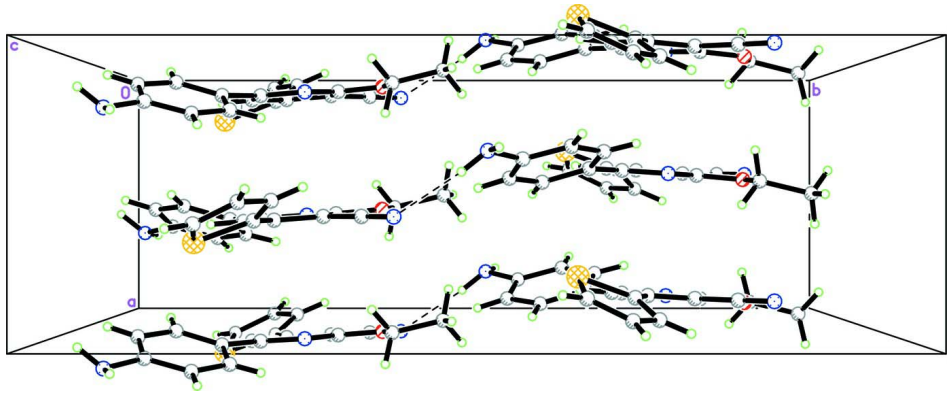


**Figure 2**

The crystal packing of the title compound, viewed approximately along the *c* axis, showing chains along [010]. Hydrogen bonds are shown as dashed lines. Only the major disorder components are shown.

### 6-(4-Aminophenyl)-2-ethoxy-4-(2-thienyl)pyridine-3-carbonitrile

*Crystal data*

$C_{18}H_{15}N_3OS$
$M_r = 321.38$
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab

$a = 7.0751\ (12)$ Å
$b = 20.843\ (4)$ Å
$c = 20.983\ (4)$ Å
$V = 3094.3\ (9)$ Å$^3$

$Z = 8$
$F(000) = 1344$
$D_x = 1.380$ Mg m$^{-3}$
Melting point = 470–471 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3045 reflections

$\theta = 1.9$–$26.0°$
$\mu = 0.22$ mm$^{-1}$
$T = 100$ K
Plate, yellow
0.35 × 0.11 × 0.04 mm

*Data collection*

Bruker APEXII DUO CCD area-detector diffractometer
Radiation source: sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
$T_{min} = 0.928$, $T_{max} = 0.992$

34805 measured reflections
3045 independent reflections
2188 reflections with $I > 2\sigma(I)$
$R_{int} = 0.092$
$\theta_{max} = 26.0°$, $\theta_{min} = 1.9°$
$h = -8 \rightarrow 8$
$k = -25 \rightarrow 24$
$l = -25 \rightarrow 25$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.085$
$S = 1.05$
3045 reflections
233 parameters
88 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0278P)^2 + 1.8001P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.21$ e Å$^{-3}$
$\Delta\rho_{min} = -0.32$ e Å$^{-3}$

*Special details*

**Experimental**. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

**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$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| O1 | 0.56053 (19) | 0.37231 (6) | 0.25979 (6) | 0.0190 (3) | |
| N1 | 0.5847 (2) | 0.26630 (7) | 0.22980 (7) | 0.0163 (4) | |
| N2 | 0.6671 (3) | 0.01670 (9) | 0.05279 (9) | 0.0245 (4) | |
| N3 | 0.5866 (2) | 0.39328 (8) | 0.41791 (8) | 0.0245 (4) | |
| C4 | 0.6016 (3) | 0.21000 (9) | 0.42677 (9) | 0.0167 (4) | |
| S1 | 0.68410 (11) | 0.13404 (3) | 0.44617 (3) | 0.01858 (19) | 0.8583 (19) |
| C1 | 0.6115 (5) | 0.14171 (15) | 0.52389 (16) | 0.0192 (7) | 0.8583 (19) |
| H1A | 0.6270 | 0.1101 | 0.5547 | 0.023* | 0.8583 (19) |

| | | | | | |
|---|---|---|---|---|---|
| C2 | 0.5284 (8) | 0.1996 (2) | 0.53499 (16) | 0.0168 (8) | 0.8583 (19) |
| H2A | 0.4807 | 0.2124 | 0.5743 | 0.020* | 0.8583 (19) |
| C3 | 0.5240 (8) | 0.2374 (2) | 0.4797 (2) | 0.0231 (10) | 0.8583 (19) |
| H3A | 0.4718 | 0.2783 | 0.4791 | 0.028* | 0.8583 (19) |
| S1X | 0.5077 (14) | 0.2515 (4) | 0.4850 (4) | 0.0200 (18)* | 0.1417 (19) |
| C1X | 0.560 (5) | 0.1904 (12) | 0.5376 (10) | 0.021 (8)* | 0.1417 (19) |
| H1XA | 0.5349 | 0.1919 | 0.5810 | 0.025* | 0.1417 (19) |
| C2X | 0.643 (3) | 0.1399 (10) | 0.5079 (8) | 0.012 (5)* | 0.1417 (19) |
| H2XA | 0.6719 | 0.1011 | 0.5274 | 0.014* | 0.1417 (19) |
| C3X | 0.679 (3) | 0.1544 (8) | 0.4425 (9) | 0.0231 (10) | 0.14 |
| H3XA | 0.7470 | 0.1284 | 0.4149 | 0.028* | 0.1417 (19) |
| C5 | 0.6017 (3) | 0.23136 (9) | 0.36009 (9) | 0.0163 (4) | |
| C6 | 0.6082 (3) | 0.18612 (9) | 0.31136 (9) | 0.0176 (4) | |
| H6A | 0.6197 | 0.1429 | 0.3217 | 0.021* | |
| C7 | 0.5978 (3) | 0.20381 (9) | 0.24772 (9) | 0.0164 (4) | |
| C8 | 0.6032 (3) | 0.15594 (9) | 0.19636 (9) | 0.0158 (4) | |
| C9 | 0.5488 (3) | 0.09205 (9) | 0.20666 (9) | 0.0203 (4) | |
| H9A | 0.5016 | 0.0800 | 0.2463 | 0.024* | |
| C10 | 0.5641 (3) | 0.04666 (9) | 0.15893 (9) | 0.0201 (4) | |
| H10A | 0.5253 | 0.0047 | 0.1666 | 0.024* | |
| C11 | 0.6372 (3) | 0.06310 (9) | 0.09920 (9) | 0.0178 (4) | |
| C12 | 0.6864 (3) | 0.12704 (9) | 0.08808 (9) | 0.0181 (4) | |
| H12A | 0.7319 | 0.1392 | 0.0483 | 0.022* | |
| C13 | 0.6680 (3) | 0.17231 (9) | 0.13553 (9) | 0.0171 (4) | |
| H13A | 0.6995 | 0.2147 | 0.1269 | 0.021* | |
| C14 | 0.5779 (3) | 0.30963 (9) | 0.27536 (9) | 0.0168 (4) | |
| C15 | 0.5874 (3) | 0.29610 (9) | 0.34132 (9) | 0.0163 (4) | |
| C16 | 0.5537 (3) | 0.38737 (9) | 0.19226 (9) | 0.0197 (4) | |
| H16A | 0.4594 | 0.3611 | 0.1712 | 0.024* | |
| H16B | 0.6754 | 0.3791 | 0.1727 | 0.024* | |
| C17 | 0.5035 (3) | 0.45707 (9) | 0.18628 (10) | 0.0248 (5) | |
| H17A | 0.5012 | 0.4689 | 0.1421 | 0.037* | |
| H17B | 0.5961 | 0.4825 | 0.2082 | 0.037* | |
| H17C | 0.3813 | 0.4644 | 0.2047 | 0.037* | |
| C18 | 0.5861 (3) | 0.34927 (9) | 0.38470 (9) | 0.0181 (4) | |
| H1N2 | 0.586 (3) | −0.0177 (12) | 0.0555 (11) | 0.039 (7)* | |
| H2N2 | 0.678 (3) | 0.0308 (10) | 0.0137 (11) | 0.029 (6)* | |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0293 (7) | 0.0135 (7) | 0.0143 (7) | 0.0002 (6) | 0.0006 (6) | 0.0000 (6) |
| N1 | 0.0175 (8) | 0.0123 (9) | 0.0191 (9) | −0.0004 (7) | 0.0020 (7) | −0.0014 (7) |
| N2 | 0.0355 (10) | 0.0196 (10) | 0.0184 (9) | −0.0040 (8) | −0.0002 (9) | −0.0040 (8) |
| N3 | 0.0352 (10) | 0.0189 (10) | 0.0195 (9) | 0.0024 (8) | 0.0004 (8) | 0.0006 (8) |
| C4 | 0.0162 (9) | 0.0156 (10) | 0.0182 (10) | 0.0001 (8) | −0.0005 (8) | 0.0003 (8) |
| S1 | 0.0220 (3) | 0.0159 (4) | 0.0179 (3) | 0.0049 (3) | 0.0012 (2) | 0.0032 (3) |
| C1 | 0.0239 (16) | 0.0225 (16) | 0.0113 (15) | 0.0003 (12) | 0.0022 (13) | 0.0024 (13) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C2 | 0.015 (2) | 0.0202 (19) | 0.0150 (15) | 0.0005 (13) | 0.0000 (11) | 0.0003 (10) |
| C3 | 0.0245 (19) | 0.019 (2) | 0.025 (2) | −0.0002 (17) | 0.0011 (13) | −0.0016 (17) |
| C3X | 0.0245 (19) | 0.019 (2) | 0.025 (2) | −0.0002 (17) | 0.0011 (13) | −0.0016 (17) |
| C5 | 0.0132 (9) | 0.0181 (10) | 0.0177 (10) | 0.0004 (8) | 0.0000 (8) | 0.0014 (8) |
| C6 | 0.0178 (9) | 0.0123 (10) | 0.0227 (11) | 0.0007 (8) | 0.0019 (8) | 0.0005 (8) |
| C7 | 0.0136 (9) | 0.0154 (10) | 0.0202 (10) | −0.0013 (8) | 0.0019 (8) | −0.0003 (9) |
| C8 | 0.0157 (9) | 0.0144 (10) | 0.0172 (10) | 0.0012 (8) | 0.0009 (8) | 0.0002 (8) |
| C9 | 0.0222 (10) | 0.0195 (11) | 0.0192 (10) | −0.0001 (9) | 0.0026 (8) | 0.0016 (9) |
| C10 | 0.0247 (10) | 0.0138 (10) | 0.0217 (11) | −0.0015 (8) | −0.0007 (9) | 0.0009 (8) |
| C11 | 0.0179 (10) | 0.0191 (10) | 0.0163 (10) | 0.0024 (8) | −0.0038 (8) | −0.0024 (8) |
| C12 | 0.0171 (9) | 0.0217 (11) | 0.0156 (10) | −0.0017 (9) | −0.0013 (8) | 0.0029 (8) |
| C13 | 0.0161 (9) | 0.0160 (10) | 0.0193 (10) | −0.0005 (8) | −0.0008 (8) | 0.0015 (8) |
| C14 | 0.0152 (9) | 0.0145 (11) | 0.0207 (10) | 0.0007 (8) | 0.0010 (8) | 0.0009 (8) |
| C15 | 0.0146 (9) | 0.0169 (10) | 0.0174 (10) | 0.0003 (8) | 0.0020 (8) | −0.0015 (8) |
| C16 | 0.0265 (10) | 0.0186 (11) | 0.0141 (10) | −0.0010 (9) | 0.0004 (8) | 0.0002 (8) |
| C17 | 0.0347 (12) | 0.0198 (11) | 0.0200 (11) | 0.0012 (9) | 0.0033 (9) | 0.0007 (9) |
| C18 | 0.0197 (10) | 0.0173 (11) | 0.0173 (10) | 0.0019 (8) | 0.0009 (8) | 0.0048 (9) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C14 | 1.352 (2) | C3X—H3XA | 0.9300 |
| O1—C16 | 1.452 (2) | C5—C6 | 1.392 (3) |
| N1—C14 | 1.316 (2) | C5—C15 | 1.409 (3) |
| N1—C7 | 1.359 (2) | C6—C7 | 1.387 (3) |
| N2—C11 | 1.389 (2) | C6—H6A | 0.9300 |
| N2—H1N2 | 0.92 (2) | C7—C8 | 1.469 (3) |
| N2—H2N2 | 0.87 (2) | C8—C13 | 1.398 (3) |
| N3—C18 | 1.152 (2) | C8—C9 | 1.403 (3) |
| C4—C3X | 1.323 (14) | C9—C10 | 1.382 (3) |
| C4—C3 | 1.364 (5) | C9—H9A | 0.9300 |
| C4—C5 | 1.468 (3) | C10—C11 | 1.399 (3) |
| C4—S1X | 1.638 (8) | C10—H10A | 0.9300 |
| C4—S1 | 1.736 (2) | C11—C12 | 1.397 (3) |
| S1—C1 | 1.717 (3) | C12—C13 | 1.378 (3) |
| C1—C2 | 1.361 (4) | C12—H12A | 0.9300 |
| C1—H1A | 0.9300 | C13—H13A | 0.9300 |
| C2—C3 | 1.404 (6) | C14—C15 | 1.414 (3) |
| C2—H2A | 0.9300 | C15—C18 | 1.434 (3) |
| C3—H3A | 0.9300 | C16—C17 | 1.501 (3) |
| S1X—C1X | 1.725 (17) | C16—H16A | 0.9700 |
| C1X—C2X | 1.355 (16) | C16—H16B | 0.9700 |
| C1X—H1XA | 0.9300 | C17—H17A | 0.9600 |
| C2X—C3X | 1.427 (17) | C17—H17B | 0.9600 |
| C2X—H2XA | 0.9300 | C17—H17C | 0.9600 |
| | | | |
| C14—O1—C16 | 116.59 (14) | N1—C7—C8 | 116.73 (17) |
| C14—N1—C7 | 117.34 (16) | C6—C7—C8 | 121.62 (17) |
| C11—N2—H1N2 | 113.9 (15) | C13—C8—C9 | 117.52 (17) |

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| C11—N2—H2N2 | 116.0 (14) | C13—C8—C7 | 120.83 (17) |
| H1N2—N2—H2N2 | 112 (2) | C9—C8—C7 | 121.63 (17) |
| C3X—C4—C3 | 109.2 (9) | C10—C9—C8 | 121.11 (18) |
| C3X—C4—C5 | 120.2 (8) | C10—C9—H9A | 119.4 |
| C3—C4—C5 | 130.5 (3) | C8—C9—H9A | 119.4 |
| C3X—C4—S1X | 116.3 (8) | C9—C10—C11 | 120.71 (18) |
| C5—C4—S1X | 123.5 (3) | C9—C10—H10A | 119.6 |
| C3—C4—S1 | 109.1 (2) | C11—C10—H10A | 119.6 |
| C5—C4—S1 | 119.99 (14) | N2—C11—C12 | 120.63 (18) |
| S1X—C4—S1 | 116.3 (3) | N2—C11—C10 | 120.94 (18) |
| C1—S1—C4 | 92.13 (12) | C12—C11—C10 | 118.39 (17) |
| C2—C1—S1 | 112.0 (3) | C13—C12—C11 | 120.61 (18) |
| C2—C1—H1A | 124.0 | C13—C12—H12A | 119.7 |
| S1—C1—H1A | 124.0 | C11—C12—H12A | 119.7 |
| C1—C2—C3 | 111.5 (3) | C12—C13—C8 | 121.57 (18) |
| C1—C2—H2A | 124.3 | C12—C13—H13A | 119.2 |
| C3—C2—H2A | 124.3 | C8—C13—H13A | 119.2 |
| C4—C3—C2 | 115.4 (4) | N1—C14—O1 | 119.39 (17) |
| C4—C3—H3A | 122.3 | N1—C14—C15 | 124.92 (17) |
| C2—C3—H3A | 122.3 | O1—C14—C15 | 115.69 (16) |
| C4—S1X—C1X | 90.0 (8) | C5—C15—C14 | 117.90 (17) |
| C2X—C1X—S1X | 111.9 (15) | C5—C15—C18 | 124.26 (17) |
| C2X—C1X—H1XA | 124.1 | C14—C15—C18 | 117.83 (17) |
| S1X—C1X—H1XA | 124.1 | O1—C16—C17 | 107.39 (15) |
| C1X—C2X—C3X | 110.7 (16) | O1—C16—H16A | 110.2 |
| C1X—C2X—H2XA | 124.6 | C17—C16—H16A | 110.2 |
| C3X—C2X—H2XA | 124.6 | O1—C16—H16B | 110.2 |
| C4—C3X—C2X | 110.6 (14) | C17—C16—H16B | 110.2 |
| C4—C3X—H3XA | 124.7 | H16A—C16—H16B | 108.5 |
| C2X—C3X—H3XA | 124.7 | C16—C17—H17A | 109.5 |
| C6—C5—C15 | 116.48 (17) | C16—C17—H17B | 109.5 |
| C6—C5—C4 | 119.64 (17) | H17A—C17—H17B | 109.5 |
| C15—C5—C4 | 123.82 (17) | C16—C17—H17C | 109.5 |
| C7—C6—C5 | 121.69 (18) | H17A—C17—H17C | 109.5 |
| C7—C6—H6A | 119.2 | H17B—C17—H17C | 109.5 |
| C5—C6—H6A | 119.2 | N3—C18—C15 | 177.8 (2) |
| N1—C7—C6 | 121.64 (17) | | |

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| C3X—C4—S1—C1 | 93 (10) | C14—N1—C7—C6 | −1.5 (3) |
| C3—C4—S1—C1 | 0.1 (3) | C14—N1—C7—C8 | 179.37 (16) |
| C5—C4—S1—C1 | −172.59 (18) | C5—C6—C7—N1 | 1.4 (3) |
| S1X—C4—S1—C1 | 1.6 (4) | C5—C6—C7—C8 | −179.56 (17) |
| C4—S1—C1—C2 | −0.2 (3) | N1—C7—C8—C13 | 25.1 (3) |
| S1—C1—C2—C3 | 0.2 (5) | C6—C7—C8—C13 | −154.02 (19) |
| C3X—C4—C3—C2 | −5.3 (12) | N1—C7—C8—C9 | −156.59 (17) |
| C5—C4—C3—C2 | 171.7 (3) | C6—C7—C8—C9 | 24.3 (3) |
| S1X—C4—C3—C2 | −170 (5) | C13—C8—C9—C10 | 1.9 (3) |
| S1—C4—C3—C2 | −0.1 (5) | C7—C8—C9—C10 | −176.49 (18) |

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| C1—C2—C3—C4 | −0.1 (7) | C8—C9—C10—C11 | 1.0 (3) |
| C3X—C4—S1X—C1X | −3 (2) | C9—C10—C11—N2 | 174.78 (18) |
| C3—C4—S1X—C1X | 14 (4) | C9—C10—C11—C12 | −2.9 (3) |
| C5—C4—S1X—C1X | 176.6 (14) | N2—C11—C12—C13 | −175.82 (17) |
| S1—C4—S1X—C1X | 2.6 (15) | C10—C11—C12—C13 | 1.8 (3) |
| C4—S1X—C1X—C2X | −2 (3) | C11—C12—C13—C8 | 1.1 (3) |
| S1X—C1X—C2X—C3X | 6 (4) | C9—C8—C13—C12 | −2.9 (3) |
| C3—C4—C3X—C2X | 4 (2) | C7—C8—C13—C12 | 175.46 (17) |
| C5—C4—C3X—C2X | −173.0 (13) | C7—N1—C14—O1 | −178.52 (15) |
| S1X—C4—C3X—C2X | 6 (2) | C7—N1—C14—C15 | 1.5 (3) |
| S1—C4—C3X—C2X | −85 (10) | C16—O1—C14—N1 | −1.0 (2) |
| C1X—C2X—C3X—C4 | −8 (3) | C16—O1—C14—C15 | 179.01 (16) |
| C3X—C4—C5—C6 | 26.4 (13) | C6—C5—C15—C14 | 0.9 (3) |
| C3—C4—C5—C6 | −150.2 (4) | C4—C5—C15—C14 | −176.28 (17) |
| S1X—C4—C5—C6 | −153.1 (5) | C6—C5—C15—C18 | −177.58 (17) |
| S1—C4—C5—C6 | 20.7 (2) | C4—C5—C15—C18 | 5.2 (3) |
| C3X—C4—C5—C15 | −156.5 (12) | N1—C14—C15—C5 | −1.2 (3) |
| C3—C4—C5—C15 | 26.9 (4) | O1—C14—C15—C5 | 178.77 (16) |
| S1X—C4—C5—C15 | 24.1 (5) | N1—C14—C15—C18 | 177.37 (17) |
| S1—C4—C5—C15 | −162.13 (15) | O1—C14—C15—C18 | −2.6 (3) |
| C15—C5—C6—C7 | −1.0 (3) | C14—O1—C16—C17 | 171.13 (16) |
| C4—C5—C6—C7 | 176.29 (17) | | |

*Hydrogen-bond geometry (Å, º)*

Cg1 is the centroid of the major disorder component of the thiophene ring.

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N2—H1*N*2···N3$^{i}$ | 0.92 (2) | 2.29 (2) | 3.197 (3) | 168.2 (19) |
| C3—H3*A*···*Cg*1$^{ii}$ | 0.93 | 2.93 | 3.566 (6) | 127 |
| C12—H12*A*···*Cg*1$^{iii}$ | 0.93 | 2.78 | 3.430 (3) | 128 |

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