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Green synthesis and crystal structure of 3-(benzothiazol-2-yl)thiophene

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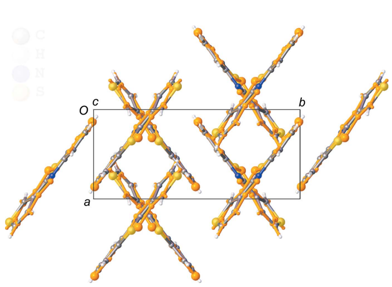
The title compound, $C_{11}H_7NS_2$, was prepared in high yield (87%) using a solvent-free microwave-assisted synthesis. The structure shows whole-molecule disorder with occupancies for two orientations (A and B) of 0.4884 (10) and 0.5116 (10), respectively. The thiophene and benzothiazole rings are almost planar and make dihedral angles of 10.02 (18) and 12.54 (19)° for orientations A and B, respectively. Slipped π–π stacking between the aromatic rings, together with C—H$\cdots\pi$, C—H$\cdots$S and C—H$\cdots$N interactions, result in a herringbone motif in the crystal packing.



1. Chemical context

Thiophene-containing heterocycles have many applications in pharmacology, such as anti-inflammatory and analgesic agents (Issa *et al.*, 2009), electrochromic and electronic devices (Elbing *et al.*, 2008), and polyelectrolytes-based water-soluble sensing agents for the detection of DNA, proteins and small bioanalytes (Ho *et al.*, 2008; Feng *et al.*, 2008). Benzothiazole-based compounds have attracted much attention in recent times due to their wide-ranging biological activities, such as anticancer, antifungal and antibacterial activities (Aiello *et al.*, 2008; Cho *et al.*, 2008). In addition, some other 2-aminobenzothiazole derivatives showed antibacterial, anti-inflammatory and analgesic properties (Bhoi *et al.*, 2014). A novel poly 3-(benzothiazol-2-yl)thiophene-based conductive polymer has been synthesized by chemical and electrochemical polymerization (Radhakrishnan *et al.*, 2006; Radhakrishnan & Somanathan, 2006). These polymers were studied for their photoabsorption and photoluminescence characteristics and were investigated in polymeric light-emitting diodes. Some synthetic methods developed for preparing 3-(benzothiazol-2-yl)thiophene are available using a mixture of thiophene-3-carbaldehyde and *o*-aminothiophenol refluxed in ethanol (Esashika *et al.*, 2009) or a mixture of 3-bromothiophene, magnesium turnings and 2-chlorobenzothiazole (Radhakrishnan *et al.*, 2003). 2-Substituted benzothiazoles have been synthesized through condensation of bis(2-aminophenyl) disulfides with arylaldehydes catalyzed by NaSH under microwave irradiation (Liu *et al.*, 2017). X-ray single-crystal structure determinations of two (1,3-benzothiazol-2-yl)thiophene derivatives synthesized from phenyl isothiocyanate (Fun *et al.*, 2012) and benzothiazole (Cheng *et al.*, 2016) have been reported, as well as of 4-(1,3-benzothiazol-2-yl)thiophene-2-sulfonamide complexed with cyclin-dependent kinase

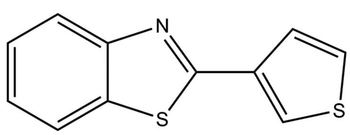
Table 1
Selected π–π interactions.

*Cg*1 is the centroid of the S15/C16–C19 plane, *Cg*2 that of the C20/S21/C22/C27/N28 plane, *Cg*3 that of the C22–C27 plane, *Cg*4 that of the S1/C2–C5 plane, *Cg*5 that of the C6/S7/C8/C13/N14 plane and *Cg*6 that of the C8–C13 plane.

CgI	*CgJ*	*Cg*–*Cg* (Å)	α (°)	*CgI*_Perp (Å)	*CgJ*_Perp (Å)
*Cg*1	*Cg*2^{i}	3.888 (3)	12.0 (2)	3.761 (2)	−3.7335 (17)
*Cg*1	*Cg*3^{i}	3.962 (3)	13.0 (2)	3.774 (2)	−3.614 (2)
*Cg*2	*Cg*1^{ii}	3.888 (3)	12.0 (2)	−3.7335 (17)	3.761 (2)
*Cg*2	*Cg*6^{ii}	3.973 (3)	9.4 (2)	−3.6796 (17)	3.708 (2)
*Cg*3	*Cg*1^{ii}	3.962 (3)	13.0 (2)	−3.614 (2)	3.774 (2)
*Cg*3	*Cg*6^{ii}	3.799 (3)	10.4 (2)	−3.631 (2)	3.720 (2)
*Cg*4	*Cg*5^{ii}	3.859 (3)	9.6 (2)	−3.5981 (19)	3.7215 (17)
*Cg*4	*Cg*6^{ii}	3.882 (3)	10.4 (2)	−3.5850 (19)	3.674 (2)
*Cg*5	*Cg*4^{i}	3.859 (3)	9.6 (2)	3.7215 (17)	−3.5981 (19)
*Cg*6	*Cg*2^{i}	3.972 (3)	9.4 (2)	3.708 (2)	−3.6796 (17)
*Cg*6	*Cg*3^{i}	3.798 (3)	10.4 (2)	3.719 (2)	−3.631 (2)
*Cg*6	*Cg*4^{i}	3.882 (3)	10.4 (2)	3.673 (2)	−3.5851 (19)

Notes: *CgI*(*J*) = plane number *I*(*J*); *Cg*–*Cg* = distance between ring centroids; *CgI*_Perp = perpendicular distance of *CgI* on ring *J*; *CgJ*_Perp = perpendicular distance of *CgJ* on ring *I*. Symmetry codes: (i) $x + 1, y, z$; (ii) $x - 1, y, z$.

5 (Malmström *et al.*, 2012). However, 3-(benzothiazol-2-yl)thiophene itself has not been studied by crystallographic methods. In this study, we present a solvent-free microwave-assisted synthesis of 3-(benzothiazol-2-yl)thiophene, starting from thiophene-3-carbaldehyde and *o*-aminothiophenol, together with its crystal structure determination. The reaction was performed in a short time, without solvent and catalyst, leading to a simple purification protocol and a high yield (87%).



2. Structural commentary

The title compound crystallizes in the monoclinic space group $P2_1/c$ with four molecules in the unit cell. The structure exhibits whole-molecule disorder by a rotation of approximately 180° around an axis running close to the S and N atoms of the benzothiazole ring, resulting in two orientations (A and B) of about the same shape (Fig. 1). In addition, orientations A and B both have similar occupancies of 0.4884 (10) and 0.5116 (10), respectively. All the heterocyclic rings are almost planar, with r.m.s. deviations of 0.017 (thiophene ring S1–C5), 0.004 (thiophene ring S15–C19), 0.010 (benzothiazole ring C6–N14) and 0.021 Å (benzothiazole ring C20–N28). For orientation A, the angle between the best planes through the thiophene and benzothiazole rings is 10.02 (18)°. In orientation B, this angle is 12.54 (19)°. The relatively planar structure of the compound results in intramolecular S⋯H contact distances shorter than the sum of the van der Waals radii of S and H (S7⋯H2 = 2.849 Å and S21⋯H16 = 2.824 Å).

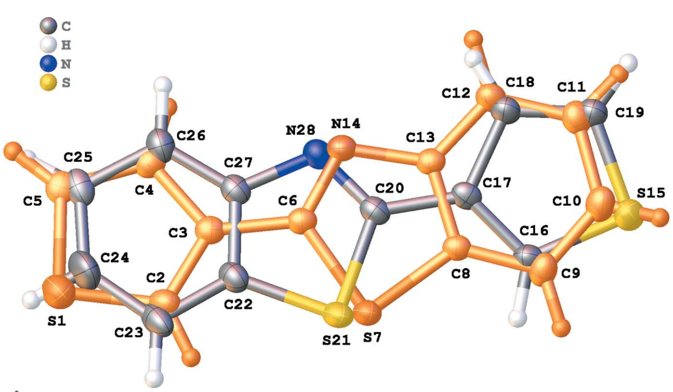


Figure 1
View of the asymmetric unit of the title compound, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are shown as small circles of arbitrary radii. Orientation A of the disordered compound (occupancy factor 0.488) is shown in orange.

Table 2
Hydrogen-bond geometry (Å, °).

*Cg*1 is the centroid of the S15/C16–C19 plane, *Cg*3 that of the C22–C27 plane, *Cg*4 that of the S1/C2–C5 plane and *Cg*6 that of the C8–C13 plane.

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
C9—H9⋯N14^{i}	0.95	2.54	3.355 (6)	144
C26—H26⋯S15^{ii}	0.95	2.87	3.522 (5)	126
C5—H5⋯*Cg*1^{iii}	0.95	2.86	3.496 (5)	125
C5—H5⋯*Cg*6^{iii}	0.95	2.93	3.532 (5)	123
C11—H11⋯*Cg*3^{iv}	0.95	2.90	3.670 (6)	139
C11—H11⋯*Cg*4^{iv}	0.95	2.90	3.705 (6)	143
C19—H19⋯*Cg*3^{iv}	0.95	2.74	3.418 (6)	129
C19—H19⋯*Cg*4^{iv}	0.95	2.73	3.447 (6)	133

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

3. Supramolecular features

The crystal packing of the title compound shows a herringbone motif (Fig. 2). This motif is built up by slipped π–π stacking between the aromatic rings and C—H⋯π interactions. The shortest centroid–centroid distances (*Cg*⋯*Cg*)

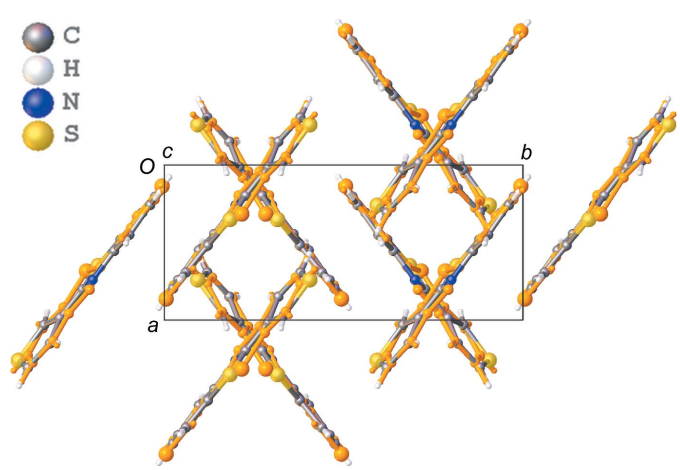


Figure 2
Crystal packing of the title compound shown in projection down the *c* axis. Orientation A of the disordered compound (occupancy factor 0.488) is shown in orange.

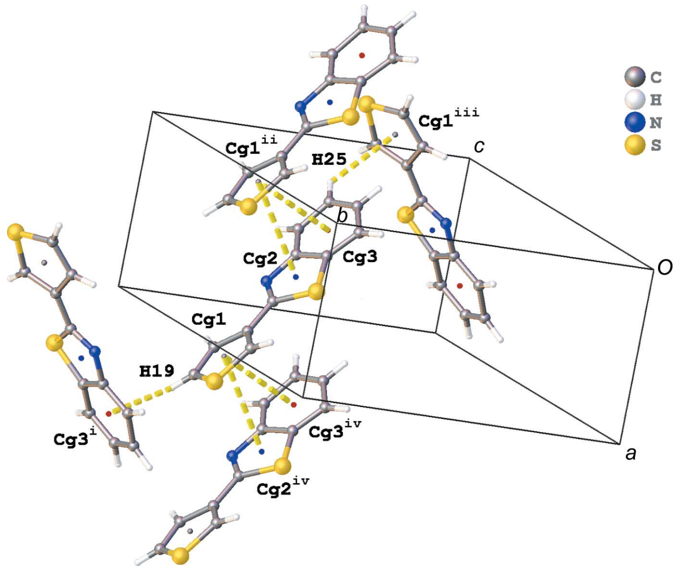


Figure 3
Slipped π–π stacking between the aromatic rings and C—H$\cdots\pi$ interactions for orientation B. [Symmetry codes: (i) $-x + 2, y + \frac{1}{2}, -z + \frac{3}{2}$; (ii) $x - 1, y, z$; (iii) $-x + 1, y - \frac{1}{2}, -z + \frac{3}{2}$; (iv) $x + 1, y, z$.]

observed in the π–π stacking for orientation B are shown in Fig. 3 and are listed in Table 1 for both orientations. The stacking molecules interact further with neighbouring molecules through C—H$\cdots\pi$ interactions (Fig. 3 and Table 2). In addition, infinite chains running in the [201] direction are formed through C—H$\cdots$N and C—H$\cdots$S interactions (Fig. 4 and Table 2). The crystal packing contains no voids. Whole-molecule disorder is usually caused by a packing which is determined by van der Waals interactions only or by a lack of directional interactions in the packing. However, the crystal packing of the title compound shows several directional interactions, and hence the whole-molecule disorder is the consequence of the very similar interations with neighbouring molecules for the two orientations.

Additional insight into the intermolecular interactions was obtained from an analysis of the Hirshfeld surface and two-dimensional fingerprint plots using *CrystalExplorer* (McKinnon *et al.*, 2007; Spackman & Jayatilaka, 2009). Fig. 5 illustrates the Hirshfeld surfaces mapped over d_{norm} for both orientations. The bright-red spots near atoms H9 and N14 for orientation A and near atoms H26 and S15 for orientation B are indicative for the hydrogen bonds given in Table 2. For

Table 3
Percentage contributions of interatomic contacts to the Hirshfeld surfaces.

Contact	Orientation A	Orientation B
H$\cdots$H	35.8	30.1
S$\cdots$H/H$\cdots$S	15.9	25.4
C$\cdots$H/H$\cdots$C	20.2	21.8
N$\cdots$H/H$\cdots$N	6.4	7.7
C$\cdots$C	8.0	8.9
C$\cdots$S/S$\cdots$C	6.1	3.0
S$\cdots$S	4.2	0.9
S$\cdots$N/N$\cdots$S	2.3	1.1
C$\cdots$N/N$\cdots$C	1.0	1.1

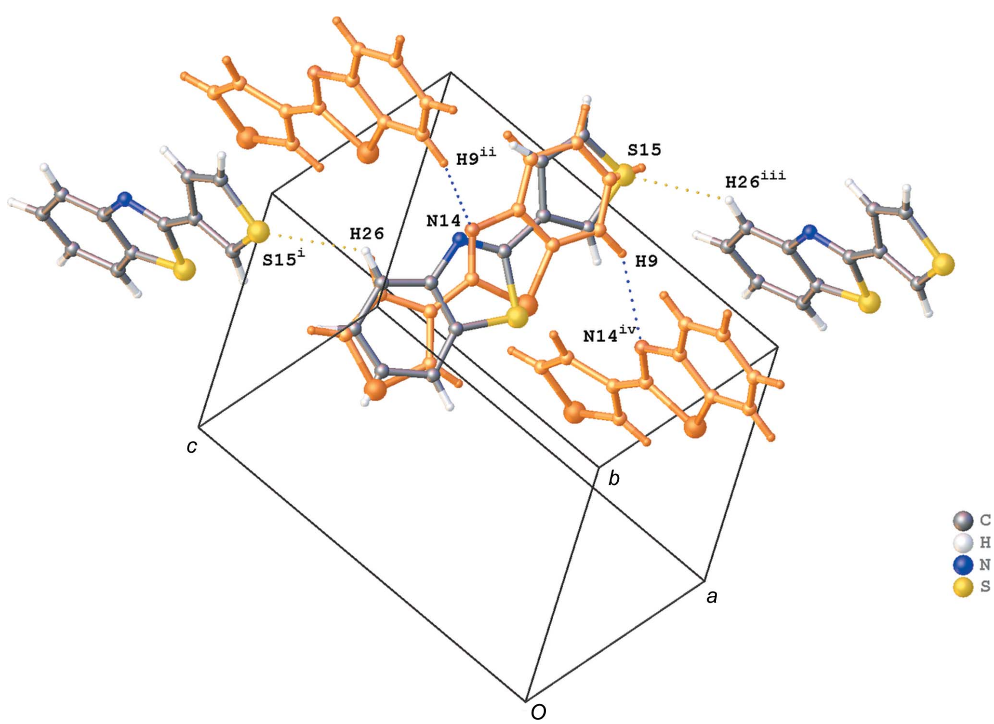


Figure 4
Infinite chain formation through C—H$\cdots$N (blue dashed lines) and C—H$\cdots$S (yellow dashed lines) interactions in the crystal packing of the title compound. Orientation A of the disordered compound (occupancy factor 0.488) is shown in orange. [Symmetry codes: (i) $x - 1, -y + \frac{3}{2}, z + \frac{1}{2}$; (ii) $x, -y + \frac{3}{2}, z + \frac{1}{2}$; (iii) $x + 1, -y + \frac{3}{2}, z - \frac{1}{2}$; (iv) $x, -y + \frac{3}{2}, z - \frac{1}{2}$.]

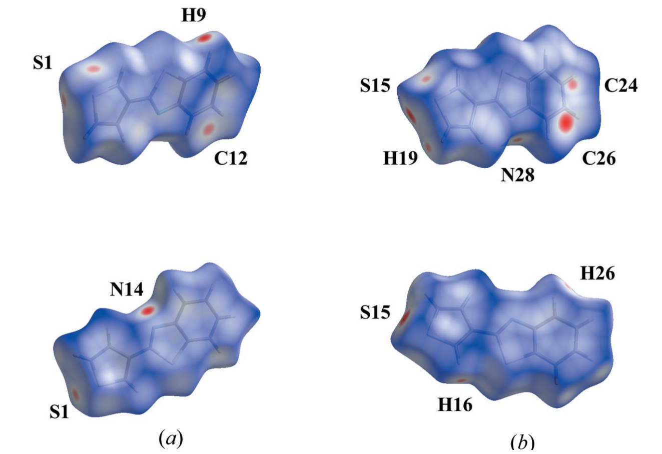


Figure 5
Two views of the Hirshfeld surfaces mapped over d_{norm} for (*a*) orientation A in the range −0.151 to 1.099 a.u. and (*b*) orientation B in the range −0.134 to 0.936 a.u.

orientation A, the red spots near atoms S1 and C12 refer to short C⋯S/S⋯C contacts and in the case of S1 also S⋯S contacts. The red spots for orientation B near atoms N28 and H16 characterize short N⋯H/H⋯N contacts, and near atoms H19 and C24 indicate short H⋯C/C⋯H contacts. The relative distributions from the different interatomic contacts to the Hirshfeld surfaces are summarized in Table 3. The largest contributions are contacts in which H atoms are involved. The largest differences between both orientations are observed for H⋯S/S⋯H (9.5%), H⋯H (5.7%), S⋯S (3.3%) and C⋯S/S⋯C (3.1%) contacts, and are caused by the presence of the C26—H26⋯S15^{ii} hydrogen bond in orientation B.

4. Database survey

A search of the Cambridge Structral Database (CSD, Version 3.38, last update May 2017; Groom *et al.*, 2016) for 3-(benzothiazol-2-yl)thiophene derivatives gives two hits: 2-anilino-4-(1,3-benzothiazol-2-yl)-5-(4-chlorobenzoyl)thiophene-3-carbonitrile (refcode LEGHOW; Fun *et al.*, 2012) and 3-(1,3-benzothiazol-2-yl)-*N*-(quinolin-8-yl)thiophene-2-carboxamide (refcode UVUGOJ; Cheng *et al.*, 2016). The substitution of the thiophene ring in these two compounds has an influence on the angle between the best planes through the thiophene and benzothiazole rings. In the monosubstituted derivative UVUGOJ, an intramolecular N—H⋯S hydrogen bond lowers the angle to 5.95°. For the trisubstituted derivative LEGHOW, the angle increases to 46.77°.

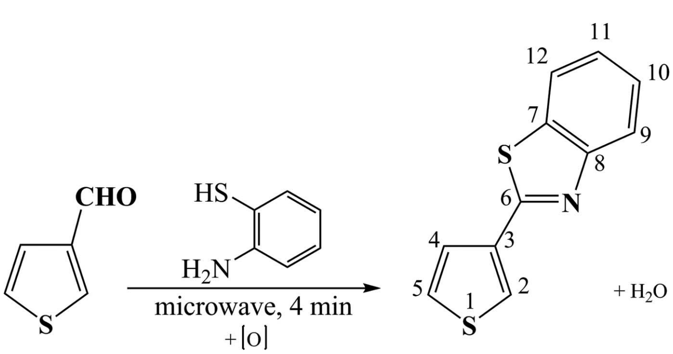


Figure 6
Reaction scheme for the title compound.

Table 4
Experimental details.

Crystal data	
Chemical formula	$C_{11}H_7NS_2$
M_r	217.30
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	100
a, *b*, *c* (Å)	6.1368 (4), 13.9799 (9), 11.4609 (7)
β (°)	100.193 (2)
V (Å^3)	967.73 (11)
Z	4
Radiation type	Mo *Kα*
μ (mm^{-1})	0.50
Crystal size (mm)	0.44 × 0.36 × 0.31
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2014)
T_{min}, T_{max}	0.703, 0.747
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	19256, 2385, 2255
R_{int}	0.034
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.667
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.076, 0.172, 1.22
No. of reflections	2385
No. of parameters	254
No. of restraints	228
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.61, −0.52

Computer programs: *APEX2* (Bruker, 2014), *SAINT* (Bruker, 2013), *SHELXT2016* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*) and *OLEX2* (Dolomanov *et al.*, 2009).

5. Synthesis and crystallization

The reaction scheme to synthesize the title compound is given in Fig. 6. The reaction mechanism is similar to that described by Mukhopadhyay & Datta (2007) for the synthesis of 2-arylbenzothiazoles.

A reaction mixture of thiophene-3-carbaldehyde (2 mmol) and *o*-aminothiophenol (2 mmol) was heated for 4 min in a domestic microwave (Sanyo EM-S1065, 800 W) at medium power level (400 W). The progress of the reaction was monitored with thin-layer chromatography (TLC) every minute. The mixture was cooled to room temperature and then dissolved in an *n*-hexane–ethyl acetate mixture (5:1 *v*/*v*) to obtain a solid product, which was further crystallized in the same solvent to give 0.38 g (yield 87%) of the title product as pale-yellow crystals (m.p. 386 K). IR (Nicolet Impact 410 FT–IR, KBr, cm^{-1}): 3067 (ν_{CH}), 1581 ($\nu_{C=C}$), 1634 ($\nu_{C=N}$). ^{1}H NMR [Bruker XL-500, 500 MHz, d_6-DMSO, δ (ppm), *J* (Hz)]: 8.36 (*dd*, 1H, ^4J = 1.0, ^5J = 2.5, H^2), 7.72 (*dd*, 1H, ^2J = 1.0, ^5J = 5.0, H^4), 7.77 (*dd*, 1H, ^2J = 2.5, ^4J = 5.0, H^5), 8.02 (*dd*, 1H, ^{11}J = 1.0, ^{10}J = 8.0, H^9), 7.52 (*td*, 1H, ^{12}J = 1.0, ^{11}J = 7.5, ^9J = 8.0, H^{10}), 7.44 (*td*, 1H, ^9J = 1.0, ^{10}J = 7.5, ^{12}J = 8.0, H^{11}), 8.11 (*dd*, 1H, ^{10}J =

1.0, ^{11}J = 8.0, H^{12}). ^{13}C NMR [Bruker XL-500, 125 MHz, d_6-DMSO, δ (ppm)]: 127.54 (C^2), 135.17 (C^3),126.17 (C^4), 128.38 (C^5), 162.17 (C^6), 134.17 (C^7), 153.30 (C^8), 122.57 (C^9), 126.53 (C^{10}), 125.30 (C^{11}), 122.22 (C^{12}). Calculation for $C_{11}H_7NS_2$: M = 217 a.u.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 4. The molecule is disordered over two positions (*A* and *B*) by a rotation of approximately 180°. The final occupancy factors are 0.4884 (10) for molecule *A* and 0.5116 (10) for molecule *B*. Enhanced rigid-body restraints (RIGU) were applied for all atoms. The H atoms were placed in idealized positions and refined in riding mode, with U_{iso}(H) values assigned as $1.2U_{eq}$ of the parent atoms, with a C—H distance of 0.95 Å. In the final cycles of refinement, 17 outliers were omitted.

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supporting information

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Green synthesis and crystal structure of 3-(benzothiazol-2-yl)thiophene

Linh Nguyen Ngoc, Trung Vu Quoc, Hoan Duong Quoc, Manh Vu Quoc, Luong Truong Minh, Chien Thang Pham and Luc Van Meervelt

Computing details

Data collection: *APEX2* (Bruker, 2014); cell refinement: *SAINT* (Bruker, 2013); data reduction: *SAINT* (Bruker, 2013); program(s) used to solve structure: SHELXT2016 (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015b); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009).

3-(Benzothiazol-2-yl)thiophene

Crystal data

$C_{11}H_7NS_2$
M_r = 217.30
Monoclinic, $P2_1/c$
a = 6.1368 (4) Å
b = 13.9799 (9) Å
c = 11.4609 (7) Å
β = 100.193 (2)°
V = 967.73 (11) Å^3
Z = 4
$F(000)$ = 448
D_x = 1.491 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9904 reflections
θ = 2.9–32.6°
μ = 0.50 mm^{-1}
T = 100 K
Block, colorless
0.44 × 0.36 × 0.31 mm

Data collection

Bruker APEXII CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2014)
T_{min} = 0.703, T_{max} = 0.747
19256 measured reflections
2385 independent reflections
2255 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 28.3°, θ_{min} = 2.9°
h = −8→8
k = −18→18
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.076
$wR(F^2)$ = 0.172
S = 1.22
2385 reflections
254 parameters
228 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0293P)^2 + 2.8579P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.61 e Å^{-3}
$\Delta\rho_{min}$ = −0.52 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
S1	0.1359 (2)	0.50570 (11)	0.64696 (12)	0.0370 (3)	0.4884 (10)
C2	0.3260 (9)	0.5734 (4)	0.5993 (4)	0.0304 (8)	0.4884 (10)
H2	0.332074	0.581845	0.517697	0.036*	0.4884 (10)
C3	0.4732 (7)	0.6159 (3)	0.6887 (4)	0.0208 (6)	0.4884 (10)
C4	0.4069 (8)	0.5922 (3)	0.8006 (4)	0.0242 (7)	0.4884 (10)
H4	0.478997	0.617848	0.874094	0.029*	0.4884 (10)
C5	0.2230 (7)	0.5268 (3)	0.7915 (4)	0.0218 (7)	0.4884 (10)
H5	0.162696	0.500659	0.855346	0.026*	0.4884 (10)
C6	0.6552 (7)	0.6774 (3)	0.6747 (4)	0.0202 (6)	0.4884 (10)
S7	0.68433 (19)	0.71644 (9)	0.53194 (10)	0.0230 (2)	0.4884 (10)
C8	0.9176 (7)	0.7805 (3)	0.5945 (3)	0.0188 (6)	0.4884 (10)
C9	1.0540 (8)	0.8389 (4)	0.5407 (4)	0.0256 (7)	0.4884 (10)
H9	1.026389	0.849650	0.457569	0.031*	0.4884 (10)
C10	1.2293 (8)	0.8799 (4)	0.6130 (4)	0.0294 (8)	0.4884 (10)
H10	1.325861	0.919477	0.577890	0.035*	0.4884 (10)
C11	1.2752 (8)	0.8670 (4)	0.7362 (4)	0.0267 (8)	0.4884 (10)
H11	1.398885	0.897336	0.783337	0.032*	0.4884 (10)
C12	1.1363 (8)	0.8092 (4)	0.7873 (4)	0.0246 (7)	0.4884 (10)
H12	1.163974	0.799310	0.870616	0.029*	0.4884 (10)
C13	0.9581 (7)	0.7659 (3)	0.7183 (4)	0.0196 (6)	0.4884 (10)
N14	0.8042 (6)	0.7076 (3)	0.7618 (3)	0.0213 (6)	0.4884 (10)
S15	1.2597 (2)	0.90620 (10)	0.59226 (10)	0.0325 (3)	0.5116 (10)
C16	1.0318 (8)	0.8344 (4)	0.5615 (4)	0.0260 (8)	0.5116 (10)
H16	0.946788	0.825702	0.484562	0.031*	0.5116 (10)
C17	0.9864 (6)	0.7905 (3)	0.6624 (3)	0.0204 (6)	0.5116 (10)
C18	1.1510 (8)	0.8185 (3)	0.7658 (4)	0.0247 (7)	0.5116 (10)
H18	1.149066	0.795829	0.843803	0.030*	0.5116 (10)
C19	1.3063 (8)	0.8798 (4)	0.7391 (4)	0.0255 (8)	0.5116 (10)
H19	1.425444	0.904712	0.795189	0.031*	0.5116 (10)
C20	0.8024 (7)	0.7272 (3)	0.6680 (4)	0.0216 (6)	0.5116 (10)
S21	0.6485 (2)	0.68370 (9)	0.53382 (10)	0.0282 (3)	0.5116 (10)
C22	0.4817 (7)	0.6220 (3)	0.6160 (4)	0.0255 (6)	0.5116 (10)
C23	0.3070 (8)	0.5607 (4)	0.5775 (5)	0.0319 (8)	0.5116 (10)
H23	0.260774	0.544164	0.496484	0.038*	0.5116 (10)
C24	0.2039 (10)	0.5250 (4)	0.6684 (5)	0.0466 (10)	0.5116 (10)
H24	0.078484	0.485473	0.643384	0.056*	0.5116 (10)
C25	0.2579 (7)	0.5390 (3)	0.7860 (5)	0.0269 (7)	0.5116 (10)
H25	0.175882	0.512781	0.841294	0.032*	0.5116 (10)
C26	0.4498 (7)	0.5970 (4)	0.8198 (4)	0.0276 (7)	0.5116 (10)

H26	0.506396	0.606303	0.901659	0.033*	0.5116 (10)
C27	0.5547 (7)	0.6395 (3)	0.7374 (4)	0.0227 (6)	0.5116 (10)
N28	0.7402 (6)	0.6993 (3)	0.7626 (3)	0.0235 (6)	0.5116 (10)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0362 (6)	0.0388 (7)	0.0338 (6)	−0.0082 (5)	0.0007 (5)	0.0022 (5)
C2	0.0364 (15)	0.0304 (19)	0.0229 (11)	−0.0075 (11)	0.0016 (10)	−0.0026 (12)
C3	0.0204 (10)	0.0211 (14)	0.0209 (10)	0.0033 (8)	0.0036 (8)	−0.0010 (10)
C4	0.0273 (13)	0.0233 (16)	0.0227 (10)	0.0023 (10)	0.0062 (9)	−0.0004 (11)
C5	0.0224 (13)	0.0164 (15)	0.0276 (11)	0.0063 (10)	0.0069 (11)	−0.0006 (12)
C6	0.0221 (10)	0.0205 (14)	0.0180 (10)	0.0019 (8)	0.0038 (8)	−0.0019 (10)
S7	0.0251 (5)	0.0246 (5)	0.0180 (4)	−0.0042 (4)	0.0004 (4)	0.0005 (4)
C8	0.0206 (11)	0.0185 (14)	0.0172 (9)	0.0014 (8)	0.0033 (8)	−0.0023 (9)
C9	0.0310 (13)	0.0225 (16)	0.0257 (12)	−0.0041 (10)	0.0115 (9)	−0.0031 (11)
C10	0.0341 (15)	0.0267 (19)	0.0296 (10)	−0.0065 (12)	0.0116 (10)	−0.0033 (12)
C11	0.0245 (14)	0.0281 (18)	0.0286 (10)	−0.0022 (11)	0.0076 (10)	−0.0038 (12)
C12	0.0242 (12)	0.0293 (16)	0.0205 (12)	−0.0030 (9)	0.0048 (9)	−0.0046 (11)
C13	0.0221 (10)	0.0188 (14)	0.0178 (9)	0.0022 (8)	0.0029 (8)	−0.0004 (9)
N14	0.0224 (10)	0.0225 (14)	0.0185 (10)	0.0005 (9)	0.0020 (8)	0.0000 (10)
S15	0.0324 (5)	0.0418 (6)	0.0241 (4)	−0.0105 (5)	0.0072 (4)	−0.0022 (5)
C16	0.0259 (14)	0.0317 (17)	0.0198 (10)	−0.0032 (11)	0.0026 (10)	−0.0032 (11)
C17	0.0209 (10)	0.0203 (13)	0.0200 (10)	0.0019 (8)	0.0037 (8)	−0.0057 (9)
C18	0.0268 (12)	0.0271 (16)	0.0198 (11)	−0.0038 (10)	0.0030 (9)	−0.0034 (11)
C19	0.0267 (13)	0.0279 (16)	0.0215 (11)	−0.0044 (10)	0.0033 (11)	−0.0019 (12)
C20	0.0200 (10)	0.0206 (13)	0.0233 (10)	0.0012 (8)	0.0009 (8)	−0.0030 (9)
S21	0.0306 (5)	0.0311 (6)	0.0203 (4)	−0.0078 (4)	−0.0028 (4)	0.0016 (4)
C22	0.0258 (12)	0.0208 (14)	0.0279 (10)	−0.0029 (9)	−0.0006 (9)	−0.0009 (10)
C23	0.0302 (14)	0.0231 (16)	0.0374 (13)	−0.0065 (10)	−0.0075 (10)	0.0021 (12)
C24	0.0482 (19)	0.044 (2)	0.0448 (11)	−0.0254 (15)	0.0000 (10)	−0.0028 (12)
C25	0.0213 (13)	0.0174 (16)	0.0412 (11)	0.0018 (10)	0.0029 (11)	0.0000 (13)
C26	0.0242 (12)	0.0260 (16)	0.0327 (12)	−0.0040 (10)	0.0054 (9)	−0.0013 (11)
C27	0.0204 (11)	0.0207 (14)	0.0261 (9)	0.0013 (8)	0.0017 (8)	−0.0018 (9)
N28	0.0234 (11)	0.0225 (13)	0.0242 (9)	−0.0012 (9)	0.0032 (8)	−0.0032 (9)

Geometric parameters (Å, º)

S1a—C2	1.667 (6)	S15b—C16	1.707 (5)
C2a—H2	0.9500	C16b—H16	0.9500
C2a—C3	1.375 (6)	C16b—C17	1.380 (6)
C3a—C4	1.449 (6)	C17b—C18	1.468 (6)
C4a—H4	0.9500	C18b—H18	0.9500
S1a—C5	1.674 (5)	S15b—C19	1.697 (5)
C4a—C5	1.442 (7)	C18b—C19	1.357 (7)
C5a—H5	0.9500	C19b—H19	0.9500
C3a—C6	1.442 (6)	C17b—C20	1.444 (6)
C6a—S7	1.763 (4)	C20b—S21	1.764 (4)

S7a—C8	1.733 (4)	S21b—C22	1.738 (5)
C8a—C9	1.389 (6)	C22b—C23	1.383 (6)
C9a—H9	0.9500	C23b—H23	0.9500
C9a—C10	1.363 (7)	C23b—C24	1.403 (8)
C10a—H10	0.9500	C24b—H24	0.9500
C10a—C11	1.402 (7)	C24b—C25	1.344 (8)
C11a—H11	0.9500	C25b—H25	0.9500
C11a—C12	1.378 (7)	C25b—C26	1.426 (6)
C12a—H12	0.9500	C26b—H26	0.9500
C12a—C13	1.372 (6)	C26b—C27	1.369 (7)
C8a—C13	1.411 (5)	C22b—C27	1.406 (6)
C6a—N14	1.299 (5)	C20b—N28	1.273 (6)
C13a—N14	1.404 (6)	C27b—N28	1.401 (5)

C3a—C2a—S1	114.0 (4)	C18b—C19b—S15	111.1 (3)
C4a—C5a—S1	107.0 (3)	C17b—C16b—S15	111.6 (3)
C3a—C2a—H2	123.0	C19b—S15b—C16	93.7 (2)
S1a—C2a—H2	123.0	C17b—C16b—H16	124.2
N14a—C6a—C3	124.2 (4)	S15b—C16b—H16	124.2
C5a—C4a—C3	114.8 (4)	N28b—C20b—C17	125.4 (4)
C2a—C3a—C4	108.1 (4)	C19b—C18b—C17	113.4 (4)
C6a—C3a—C4	125.4 (4)	C20b—C17b—C18	124.0 (4)
C5a—C4a—H4	122.6	C16b—C17b—C18	110.2 (4)
C3a—C4a—H4	122.6	C19b—C18b—H18	123.3
C2a—S1a—C5	96.0 (2)	C17b—C18b—H18	123.3
C4a—C5a—H5	126.5	S15b—C19b—H19	124.5
S1a—C5a—H5	126.5	C18b—C19b—H19	124.5
C2a—C3a—C6	126.4 (4)	C16b—C17b—C20	125.8 (4)
C8a—S7a—C6	89.31 (19)	C22b—S21b—C20	88.5 (2)
C3a—C6a—S7	119.8 (3)	C17b—C20b—S21	118.4 (3)
N14a—C6a—S7	116.1 (3)	N28b—C20b—S21	116.2 (3)
C9a—C8a—S7	129.7 (3)	C23b—C22b—S21	129.2 (4)
C13a—C8a—S7	109.1 (3)	C27b—C22b—S21	109.6 (3)
C10a—C9a—C8	116.8 (4)	C26b—C27b—C22	120.0 (4)
N14a—C13a—C8	115.5 (4)	N28b—C27b—C22	114.4 (4)
C12a—C13a—C8	119.7 (4)	C25b—C24b—C23	129.0 (5)
C8a—C9a—H9	121.6	C24b—C23b—H23	122.9
C10a—C9a—H9	121.6	C22b—C23b—H23	122.9
C12a—C11a—C10	118.3 (4)	C22b—C23b—C24	114.1 (5)
C11a—C10a—H10	118.2	C23b—C24b—H24	115.5
C9a—C10a—H10	118.2	C25b—C24b—H24	115.5
C9a—C10a—C11	123.6 (5)	C27b—C26b—C25	121.7 (4)
C13a—C12a—C11	120.3 (4)	C26b—C25b—H25	123.2
C12a—C11a—H11	120.8	C24b—C25b—H25	123.2
C10a—C11a—H11	120.8	C24b—C25b—C26	113.6 (5)
C11a—C12a—H12	119.9	C25b—C26b—H26	119.1
C13a—C12a—H12	119.9	C27b—C26b—H26	119.1
C9a—C8a—C13	121.2 (4)	C20b—N28b—C27	111.3 (4)

C6a—N14a—C13	110.0 (4)	C23b—C22b—C27	121.1 (4)
C12a—C13a—N14	124.7 (4)	C26b—C27b—N28	125.6 (4)

C5a—S1a—C2a—C3a	1.2 (4)	C19b—S15b—C16b—C17b	0.8 (4)
S1a—C2a—C3a—C6a	179.5 (4)	S15b—C16b—C17b—C20b	178.4 (3)
S1a—C2a—C3a—C4a	−3.4 (5)	S15b—C16b—C17b—C18b	−0.7 (5)
C2a—C3a—C4a—C5a	4.7 (6)	C16b—C17b—C18b—C19b	0.1 (6)
C6a—C3a—C4a—C5a	−178.3 (4)	C20b—C17b—C18b—C19b	−178.9 (4)
C3a—C4a—C5a—S1a	−3.8 (5)	C17b—C18b—C19b—S15b	0.5 (5)
C2a—S1a—C5a—C4a	1.5 (4)	C16b—S15b—C19b—C18b	−0.7 (4)
C2a—C3a—C6a—N14a	−172.3 (5)	C16b—C17b—C20b—N28b	−167.9 (5)
C4a—C3a—C6a—N14a	11.2 (7)	C18b—C17b—C20b—N28b	11.0 (7)
C2a—C3a—C6a—S7a	8.0 (6)	C16b—C17b—C20b—S21b	12.2 (6)
C4a—C3a—C6a—S7a	−168.5 (4)	C18b—C17b—C20b—S21b	−168.9 (3)
N14a—C6a—S7a—C8a	−0.5 (4)	N28b—C20b—S21b—C22b	0.4 (4)
C3a—C6a—S7a—C8a	179.2 (4)	C17b—C20b—S21b—C22b	−179.7 (3)
C6a—S7a—C8a—C9a	−178.7 (4)	C20b—S21b—C22b—C23b	−177.2 (5)
C6a—S7a—C8a—C13a	1.2 (3)	C20b—S21b—C22b—C27b	0.3 (3)
C13a—C8a—C9a—C10a	0.8 (7)	C27b—C22b—C23b—C24b	4.4 (7)
S7a—C8a—C9a—C10a	−179.3 (4)	S21b—C22b—C23b—C24b	−178.3 (4)
C8a—C9a—C10a—C11a	−0.8 (8)	C22b—C23b—C24b—C25b	−3.2 (9)
C9a—C10a—C11a—C12a	0.4 (8)	C23b—C24b—C25b—C26b	−1.3 (9)
C10a—C11a—C12a—C13a	0.0 (8)	C24b—C25b—C26b—C27b	4.9 (7)
C11a—C12a—C13a—N14a	−178.5 (4)	C25b—C26b—C27b—N28b	178.3 (4)
C11a—C12a—C13a—C8a	0.1 (7)	C25b—C26b—C27b—C22b	−3.8 (7)
C9a—C8a—C13a—C12a	−0.5 (7)	C23b—C22b—C27b—C26b	−1.2 (7)
S7a—C8a—C13a—C12a	179.6 (4)	S21b—C22b—C27b—C26b	−179.0 (4)
C9a—C8a—C13a—N14a	178.2 (4)	C23b—C22b—C27b—N28b	176.9 (4)
S7a—C8a—C13a—N14a	−1.7 (5)	S21b—C22b—C27b—N28b	−0.9 (5)
C3a—C6a—N14a—C13a	179.9 (4)	C17b—C20b—N28b—C27b	179.1 (4)
S7a—C6a—N14a—C13a	−0.3 (5)	S21b—C20b—N28b—C27b	−1.0 (5)
C12a—C13a—N14a—C6a	180.0 (4)	C26b—C27b—N28b—C20b	179.2 (4)
C8a—C13a—N14a—C6a	1.3 (5)	C22b—C27b—N28b—C20b	1.2 (5)

Hydrogen-bond geometry (Å, º)

*Cg*1 is the centroid of the S15/C16–C19 plane, *Cg*3 that of the C22–C27 plane, *Cg*4 that of the S1/C2–C5 plane and *Cg*6 that of the C8–C13 plane.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C9—H9···N14^{i}	0.95	2.54	3.355 (6)	144
C26—H26···S15^{ii}	0.95	2.87	3.522 (5)	126
C5—H5···*Cg*1^{iii}	0.95	2.86	3.496 (5)	125
C5—H5···*Cg*6^{iii}	0.95	2.93	3.532 (5)	123
C11—H11···*Cg*3^{iv}	0.95	2.90	3.670 (6)	139
C11—H11···*Cg*4^{iv}	0.95	2.90	3.705 (6)	143
C19—H19···*Cg*3^{iv}	0.95	2.74	3.418 (6)	129
C19—H19···*Cg*4^{iv}	0.95	2.73	3.447 (6)	133

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