organic compounds

3,3'-Bithiophene

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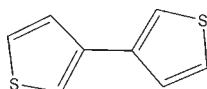Correspondence e-mail: jnlow111@googlemail.com

The title compound, $C_8H_6S_2$, is disordered [occupancy ratio = 0.839 (2):0.161 (2)] and sits across a centre of symmetry. In the crystal, the molecules are linked by a weak C—H⋯π interaction.

Related literature

For a discussion of the disorder in this compound, see: Visser *et al.* (1968). For thiophene C–S bond distances, see: Allen *et al.* (1987).

Experimental

Crystal data

$C_8H_6S_2$
M_r = 166.25
Orthorhombic, $Pccn$
a = 7.5187 (7) Å
b = 18.2181 (17) Å
c = 5.5029 (5) Å

V = 753.77 (12) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.62 mm^{-1}
T = 150 K
$0.60 \times 0.40 \times 0.04$ mm

Data collection

Bruker SMART APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
T_{min} = 0.709, T_{max} = 0.976

11635 measured reflections
1151 independent reflections
987 reflections with $I > 2\sigma(I)$
R_{int} = 0.039

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.101
S = 1.10
1151 reflections
59 parameters

6 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.48 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

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

Cg and Cg' are the centroids of the thiophene ring in the major and minor occupancy disorder components, respectively.

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C2—H2⋯Cg^i	0.95	2.86	3.6039 (17)	136
C2—H2⋯Cg'^i	0.95	2.86	3.607 (5)	136

Symmetry code: (i) $-x + \frac{1}{2}, y, z - \frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEPII* (Johnson, 1976) and *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bruker (2004). *APEXII, SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Visser, G. J., Heeres, G. J., Wolters, J. & Vos, A. (1968). *Acta Cryst.* B**24**, 467–473.

supporting information

3,3′-Bithiophene

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S1. Comment

The disorder in the title compound was discussed briefly by Visser *et al.* (1968). However, this paper gives no coordinates and the structure determination was at room temperature. This is a low temerature determination. A view of the major, 0.839 (2), site occupancy, and minor, 0.161 (2), site occupancy, components are shown in Fig. 1. There is a weak C–H···π interaction, C2–H2···Cg(thiophene) (0.5-x, y, z-0.5) in which H2···Cg is 2.86Å and C2···Cg is 3.6039 (17) Å. The angle at H2 ia 136° for the major component. The C2···Cg2 distance for the minor component is 3.607 (5) Å. The H2···Cg distance and angle at H2 are the same.

S2. Experimental

The compound was obtained commercially and re-crystallised from dichloromethane.

S3. Refinement

H atoms were treated as riding atoms with C—H(aromatic), 0.95Å. The S atom was disordered by rotation of 180° around the bond connecting the 2 thiophene rings. The C—S distances were restrained the average value quoted in Allen, *et al.*, 1987 using tight restraints. Specifically, the C2-C5a and C4-C5 bonds were restrained in SHELXL97 refinements using DFIX 1.380 0.001 and the C5-S1, C2-S1, C4-S1A and C5A-S1A bonds were restrained using DFIX 1.72 0.001. The anisotropic thermal parameters for atom C5A (minor component) were constrained to be the same as those those of atom C5 (major component) using the EADP instruction.
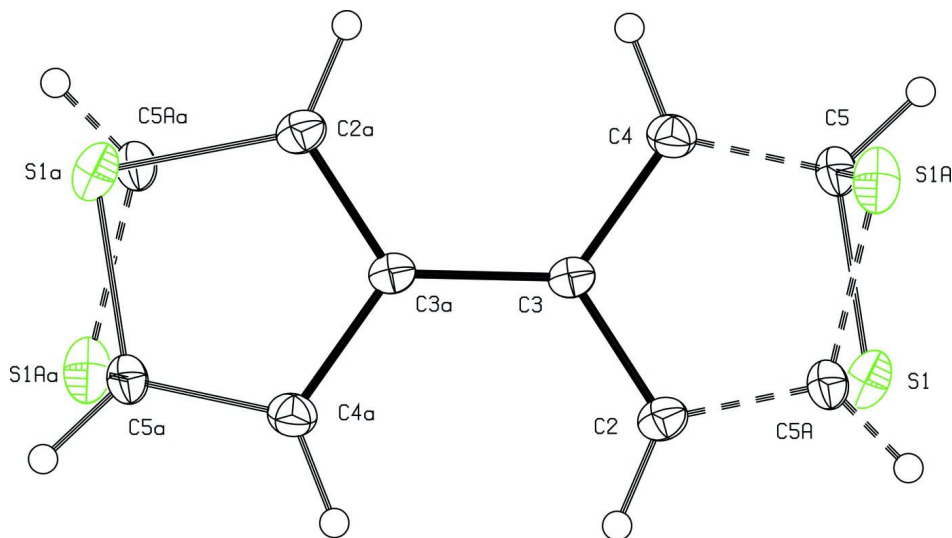
Figure 1

A view of the title compound with our numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. The molecule sits across the centre-of-symmetry at (0.5,0.5,0.5). The bonds in the minor component are marked as dotted lines.

3,3′-bithiophene

Crystal data

$C_8H_6S_2$	$D_x = 1.465$ Mg m^{-3}
$M_r = 166.25$	Melting point: 406 K
Orthorhombic, $Pccn$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 7.5187$ (7) Å	Cell parameters from 124 reflections
$b = 18.2181$ (17) Å	$\theta = 2.8–30.5°$
$c = 5.5029$ (5) Å	$\mu = 0.62$ mm^{-1}
$V = 753.77$ (12) Å^3	$T = 150$ K
$Z = 4$	Plate, yellow
$F(000) = 344$	$0.60 \times 0.40 \times 0.04$ mm

Data collection

Bruker SMART APEXII diffractometer	11635 measured reflections
	1151 independent reflections
Radiation source: fine-focus sealed tube	987 reflections with $I > 2\sigma(I)$
Graphite monochromator	$R_{int} = 0.039$
ω scans	$\theta_{max} = 30.6°$, $\theta_{min} = 4.3°$
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)	$h = -10 \rightarrow 10$
	$k = -23 \rightarrow 26$
$T_{min} = 0.709$, $T_{max} = 0.976$	$l = -7 \rightarrow 7$

Refinement

Refinement on F^2	6 restraints
Least-squares matrix: full	Primary atom site location: structure-invariant direct methods
$R[F^2 > 2\sigma(F^2)] = 0.039$	
$wR(F^2) = 0.101$	Secondary atom site location: difference Fourier map
$S = 1.10$	
1151 reflections	Hydrogen site location: inferred from neighbouring sites
59 parameters	

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0478P)^2 + 0.3736P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.48$ e Å^{-3}
$\Delta\rho_{min} = -0.33$ 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.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor w*R* 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)
S1	0.44974 (10)	0.67774 (2)	0.55925 (11)	0.03155 (18)	0.839 (2)
C5A	0.446 (2)	0.6612 (2)	0.5331 (18)	0.0246 (5)	0.161 (2)
H5A	0.4003	0.7061	0.4718	0.030*	0.161 (2)
C2	0.4207 (2)	0.59508 (6)	0.4154 (2)	0.0255 (3)	
H2	0.3574	0.5891	0.2673	0.031*	
C3	0.50025 (17)	0.53842 (7)	0.5407 (2)	0.0199 (3)	
C4	0.58337 (19)	0.56308 (7)	0.7572 (3)	0.0262 (3)	
H4	0.6439	0.5314	0.8666	0.031*	
C5	0.5674 (8)	0.63775 (10)	0.7926 (7)	0.0246 (5)	0.839 (2)
H5	0.6150	0.6636	0.9277	0.030*	0.839 (2)
S1A	0.5658 (13)	0.65626 (13)	0.7988 (13)	0.0391 (12)	0.161 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0353 (3)	0.0217 (2)	0.0377 (3)	0.0028 (2)	0.0068 (2)	0.00256 (18)
C5A	0.0243 (10)	0.0215 (10)	0.0279 (10)	−0.0028 (13)	0.0000 (8)	−0.0033 (10)
C2	0.0264 (7)	0.0274 (6)	0.0227 (6)	0.0032 (5)	0.0010 (5)	0.0039 (5)
C3	0.0152 (6)	0.0247 (6)	0.0198 (6)	0.0022 (5)	0.0025 (5)	0.0022 (5)
C4	0.0234 (7)	0.0303 (7)	0.0249 (6)	0.0026 (5)	−0.0034 (5)	−0.0015 (5)
C5	0.0243 (10)	0.0215 (10)	0.0279 (10)	−0.0028 (13)	0.0000 (8)	−0.0033 (10)
S1A	0.040 (2)	0.0276 (17)	0.049 (2)	−0.001 (2)	0.0038 (15)	−0.0074 (17)

Geometric parameters (Å, º)

S1—C2	1.7152 (9)	C3—C4	1.4181 (19)
S1—C5	1.7215 (10)	C3—C3^i	1.470 (3)
C5A—C2	1.3802 (10)	C4—C5	1.3794 (10)
C5A—S1A	1.7203 (10)	C4—S1A	1.7180 (10)
C5A—H5A	0.9500	C4—H4	0.9500
C2—C3	1.3778 (19)	C5—H5	0.9500
C2—H2	0.9500		

C2—S1—C5	92.19 (8)	C4—C3—C3ⁱ	123.98 (15)
C2—C5A—S1A	115.2 (3)	C5—C4—C3	113.15 (13)
C2—C5A—H5A	122.4	C3—C4—S1A	113.04 (17)
S1A—C5A—H5A	122.4	C5—C4—H4	123.4
C3—C2—C5A	111.1 (2)	C3—C4—H4	123.4
C3—C2—S1	111.81 (10)	S1A—C4—H4	123.5
C3—C2—H2	124.1	C4—C5—S1	110.87 (12)
C5A—C2—H2	124.9	C4—C5—H5	124.6
S1—C2—H2	124.1	S1—C5—H5	124.6
C2—C3—C4	111.98 (11)	C4—S1A—C5A	88.8 (2)
C2—C3—C3ⁱ	124.05 (15)		
S1A—C5A—C2—C3	−0.2 (15)	C3ⁱ—C3—C4—C5	179.3 (3)
C5—S1—C2—C3	−0.6 (3)	C2—C3—C4—S1A	−1.3 (5)
C5A—C2—C3—C4	1.0 (8)	C3ⁱ—C3—C4—S1A	178.5 (4)
S1—C2—C3—C4	0.73 (16)	C3—C4—C5—S1	0.1 (5)
C5A—C2—C3—C3ⁱ	−178.8 (8)	C2—S1—C5—C4	0.3 (4)
S1—C2—C3—C3ⁱ	−179.03 (14)	C3—C4—S1A—C5A	1.0 (10)
C2—C3—C4—C5	−0.5 (3)	C2—C5A—S1A—C4	−0.4 (14)

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

Hydrogen-bond geometry (Å, º)

Cg and Cg′ are the centroids of the thiophene ring in the major and minor occupancy disorder components, respectively.

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
C2—H2⋯*Cg*ⁱⁱ	0.95	2.86	3.6039 (17)	136
C2—H2⋯*Cg*′ⁱⁱ	0.95	2.86	3.607 (5)	136

Symmetry code: (ii) −*x*+1/2, *y*, *z*−1/2.