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Bis(2-thienyl)acetylene

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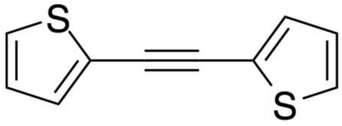
Key indicators: single-crystal X-ray study; T = 120 K; mean σ(C–C) = 0.006 Å; disorder in main residue; R factor = 0.073; wR factor = 0.173; data-to-parameter ratio = 14.6.

The planar [maximum deviation 0.0066 (4) Å] symmetrical molecule of the title compound, $C_{10}H_6S_2$, lies across a crystallographic inversion centre. The thiophene rings are rotationally disordered about the acetylene bond, with the two pseudo inversion-related S atoms in 0.80:0.20 occupancy sites. The C≡C bond distance is 1.195 (9) Å.

Related literature

For the preparation of the title compound, related diarylacetylenes and cobalt-containing metallocenes derived from these materials, see: Harrison *et al.* (1997); Harcourt *et al.* (2008). For recent synthetic organic uses, see: Yu & Rovis (2006); Geyer *et al.* (2008). The metal center employed in an acetylene cyclooligomerization may also remain as an integral component of the product, or products, see: Rausch & Genetti (1970). For spectroscopic data, see: Mio *et al.* (2002).



Experimental

Crystal data

$C_{10}H_6S_2$	a = 10.6325 (15) Å
M_r = 190.29	b = 10.8713 (15) Å
Orthorhombic, $Pbcn$	c = 7.5600 (5) Å
V = 873.85 (18) Å^3	μ = 0.54 mm^{-1}
Z = 4	T = 120 K
Mo $K\alpha$ radiation	0.55 × 0.05 × 0.03 mm

Data collection

Nonius KappaCCD diffractometer	3812 measured reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)	849 independent reflections
$T_{\min}$ = 0.755, $T_{\max}$ = 0.984	493 reflections with $I > 2\sigma(I)$
	$R_{\rm int}$ = 0.129

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.073	58 parameters
$wR(F^2)$ = 0.173	H-atom parameters constrained
S = 1.08	$\Delta\rho_{\max}$ = 0.41 e Å^{-3}
849 reflections	$\Delta\rho_{\min}$ = −0.41 e Å^{-3}

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZS2008).

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supplementary materials

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Bis(2-thienyl)acetylene

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Comment

Diarylacetylenes are versatile components of metal-mediated cycloaddition reactions. Their relative ease of preparation from palladium catalyzed coupling of aryl iodides to acetylene has ensured their continued use in the development of new synthetic routes, for example, nitrogen containing heterocycles (Yu & Rovis, 2006), and new catalytic reaction methodologies such as alkyne–nitrile cross metathesis (Geyer *et al.*, 2008). The metal center employed in an acetylene cyclooligomerization may also remain as an integral component of the product, or products, as described in the seminal work of Rausch & Genetti (1970). The title compound bis(2-thienyl)acetylene (I) is found to have inversion symmetry coincident with crystallographic symmetry (Fig. 1). However, the two 2-thiophene residues are rotationally disordered about the acetylene bond with the two pseudo-inversion related S atoms having 80/20% occupancy. The C—C triple bond distance is 1.195 (9) Å.

Experimental

The title compound was prepared by Sonogashira coupling of two equivalents of 2-iodothiophene to acetylene under standard conditions (Harrison *et al.*, 1997). Full experimental details (Harcourt *et al.*, 2008) and spectroscopic data (Mio *et al.*, 2002) have been previously published.

Refinement

All H atoms were included in the refinement at calculated positions, in the riding-model approximation, with C—H distances of 0.95 Å. The isotropic displacement parameters for all H atoms were set equal to $1.25U_{eq}$ of the carrier atom. The refined site occupancy factors for the disordered atoms (S1, C3, H3) and (S3, C1, H1) of the pseudo-centrosymmetrically related thiophene rings were 0.80 (1), and 0.20 (1) respectively. Structure factor file checks indicate that there is only one listed reflection that is likely to have been affected by the beamstop.

Figures

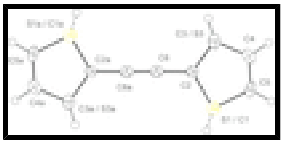

Fig. 1. Molecular configuration and atom-numbering scheme for (I) showing inversion symmetry [symmetry code: (a) -*x*, -*y* + 1, -*z*]. Rotationally disordered thiophene S/C atom pairs are S1, C3 (S.O.F. 0.80) and S3, C1 (S.O.F. 0.20). Displacement ellipsoids are drawn at the 50% probability level.

Bis(2-thienyl)acetylene

Crystal data

$C_{10}H_6S_2$	$F_{000} = 392$
$M_r = 190.29$	$D_x = 1.446$ Mg m^{-3}

Orthorhombic, *Pbcn*

Hall symbol: -P 2n 2ab

a = 10.6325 (15) Å

b = 10.8713 (15) Å

c = 7.5600 (5) Å

V = 873.85 (18) Å^3

Z = 4

Mo $K\alpha$ radiation, λ = 0.71073 Å

Cell parameters from 1041 reflections

θ = 1.0–27.5°

μ = 0.54 mm^{-1}

T = 120 K

Needle, colourless

0.55 × 0.05 × 0.03 mm

Data collection

Nonius KappaCCD diffractometer

Radiation source: Bruker Nonius FR591 rotating anode

Monochromator: 10 cm confocal mirrors

Detector resolution: 9.091 pixels mm^{-1}

T = 120 K

φ and ω scans

Absorption correction: multi-scan (SADABS; Sheldrick, 2003)

$T_{\min}$ = 0.755, $T_{\max}$ = 0.984

3812 measured reflections

849 independent reflections

493 reflections with $I > 2\sigma(I)$

R_{int} = 0.129

$\theta_{\max}$ = 26.0°

$\theta_{\min}$ = 2.7°

$h = -13 \rightarrow 11$

$k = -13 \rightarrow 12$

$l = -9 \rightarrow 8$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)]$ = 0.073

$wR(F^2)$ = 0.173

S = 1.08

849 reflections

58 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.050P)^2 + 3.1085P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max}$ = 0.41 e Å^{-3}

$\Delta\rho_{\min}$ = −0.41 e Å^{-3}

Extinction correction: none

Special details

Experimental. The minimum and maximum absorption values stated above are those calculated in *SHELXL97* from the given crystal dimensions. The ratio of minimum to maximum apparent transmission was determined experimentally as 0.675726.

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

	x	y	z	U_{iso}*/U_{eq}	Occ. (<1)
S1	0.17278 (13)	0.28773 (15)	0.17139 (19)	0.0303 (6)	0.80
C1	0.17278 (13)	0.28773 (15)	0.17139 (19)	0.0303 (6)	0.20
H1	0.1105	0.2380	0.2263	0.038*	0.20

C2	0.1671 (4)	0.4188 (5)	0.0513 (6)	0.0220 (12)	
C3	0.2949 (3)	0.4642 (3)	−0.0126 (4)	0.0291 (9)	0.80
H3	0.3142	0.5342	−0.0827	0.036*	0.80
S3	0.2949 (3)	0.4642 (3)	−0.0126 (4)	0.0291 (9)	0.20
C4	0.3820 (5)	0.3703 (5)	0.0635 (6)	0.0295 (15)	
H4	0.4705	0.3749	0.0468	0.037*	
C5	0.3285 (4)	0.2786 (5)	0.1572 (6)	0.0271 (13)	
H5	0.3757	0.2144	0.2105	0.034*	
C6	0.0498 (4)	0.4768 (5)	0.0149 (6)	0.0235 (14)	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0283 (8)	0.0314 (11)	0.0311 (8)	0.0002 (8)	−0.0017 (6)	0.0030 (7)
C1	0.0283 (8)	0.0314 (11)	0.0311 (8)	0.0002 (8)	−0.0017 (6)	0.0030 (7)
C2	0.022 (2)	0.023 (3)	0.021 (2)	−0.001 (2)	−0.0019 (19)	0.000 (2)
C3	0.0264 (16)	0.029 (2)	0.0322 (16)	0.0028 (17)	−0.0049 (13)	−0.0064 (15)
S3	0.0264 (16)	0.029 (2)	0.0322 (16)	0.0028 (17)	−0.0049 (13)	−0.0064 (15)
C4	0.021 (2)	0.035 (4)	0.032 (3)	−0.002 (3)	0.003 (2)	−0.008 (3)
C5	0.030 (3)	0.027 (3)	0.025 (2)	0.012 (3)	−0.007 (2)	−0.006 (2)
C6	0.025 (2)	0.021 (4)	0.025 (2)	−0.002 (2)	−0.001 (2)	−0.001 (2)

Geometric parameters (Å, °)

S1—C2	1.691 (5)	C4—C5	1.349 (7)
C2—C6	1.424 (6)	C4—H4	0.95
C2—C3	1.525 (5)	C5—H5	0.95
C3—C4	1.493 (6)	C6—C6^{i}	1.195 (9)
C3—H3	0.95		
C6—C2—C3	125.2 (4)	C5—C4—C3	116.4 (4)
C6—C2—S1	120.5 (4)	C5—C4—H4	121.8
C3—C2—S1	114.2 (3)	C3—C4—H4	121.8
C4—C3—C2	102.1 (3)	C4—C5—H5	122.9
C4—C3—H3	128.9	C6^{i}—C6—C2	178.7 (7)
C2—C3—H3	128.9		
C6—C2—C3—C4	179.9 (4)	C2—C3—C4—C5	0.8 (5)
S1—C2—C3—C4	−1.1 (4)		

Symmetry codes: (i) $-x$, $-y+1$, $-z$.

Fig. 1