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2,5-Bis(5-bromo-2-thienyl)thiophene

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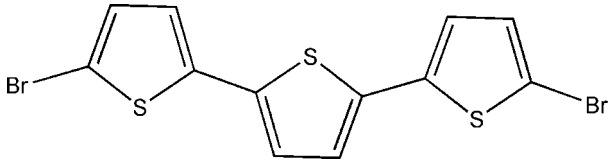
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.008 Å; R factor = 0.053; wR factor = 0.114; data-to-parameter ratio = 19.6.

In the crystal structure of the title compound, $C_{12}H_6Br_2S_3$, the molecules are planar (r.m.s. deviation = 0.06 Å). Consecutive molecules do not stack in a planar fashion. There is an angle of 81.7 (12)° between the planes of the closest molecules.

Related literature

For related structures, see: Pyrka *et al.* (1988). For literature related to synthesis, see: Hoffmann & Carlsen (1999); Mei *et al.* (2009). For a recent review of oligothiophenes, see: Mishra *et al.* (2009).



Experimental

Crystal data

$C_{12}H_6Br_2S_3$
M_r = 406.17
Orthorhombic, $Pcc2$
a = 7.6216 (16) Å
b = 30.003 (6) Å
c = 5.8841 (13) Å
V = 1345.5 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 6.46 mm^{-1}
T = 173 K
0.37 × 0.24 × 0.10 mm

Data collection

Siemens SMART Platform CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2008*a*)
T_{min} = 0.184, T_{max} = 0.524
9565 measured reflections
3045 independent reflections
2818 reflections with $I > 2\sigma(I)$
R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.114
S = 1.25
3045 reflections
155 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.62 e Å^{-3}
Absolute structure: Flack (1983), 1341 Friedel pairs
Flack parameter: 0.00 (7)

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008*b*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008*b*); molecular graphics: *SHELXTL* (Sheldrick, 2008*b*); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2619).

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supporting information

Acta Cryst. (2009). E**65**, o2119 [doi:10.1107/S1600536809030864]

2,5-Bis(5-bromo-2-thienyl)thiophene

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S1. Comment

Dibromothiophenes are important building blocks in materials chemistry. They are mainly used in the prepartaion of various thiophene oligomers and polymers utilizing coupling reactions such as Stille and Suzuki couplings.

For literature related to the synthesis see: Hoffman & Carlsen (1999) and Mei (2009). For a recent review on synthesis and applications of oligothiophenes,see: Mishra (2009).

S2. Experimental

Synthesis was carried out following literature procedures (Hoffman) as follows: to a a solution of terthiophene dissolved in chloroform was added 2 equivalents of *N*-bromosuccinimde and the reaction mixture was stirred at room temperature for 2 h. The reaction mixture was then extracted with water and product obtained by evaporation of chloroform and recrystallized twice from hexanes. The crystals were very thin, hence the large number in the second weighting scheme.

S3. Refinement

The structure was solved using *SHELXS97* and refined using *SHELXL97* (Sheldrick, 2008). The space group Pcc2 was determined based on systematic absences and intensity statistics. A direct-methods solution was calculated which provided most non-hydrogen atoms from the E-map. Full-matrix least squares / difference Fourier cycles were performed which located the remaining non-hydrogen atoms. All non-hydrogen atoms were refined with anisotropic displacement parameters. All hydrogen atoms were placed in ideal positions and refined as riding atoms with relative isotropic displacement parameters. The final full matrix least squares refinement converged to R1 = 0.0527 and wR2 = 0.1169 (F2, all data).

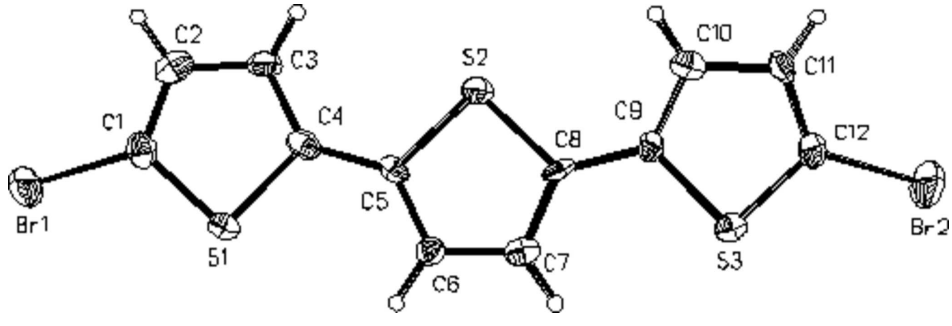

Figure 1

2,5-bis(5-bromothiophen-2-yl)thiophene.

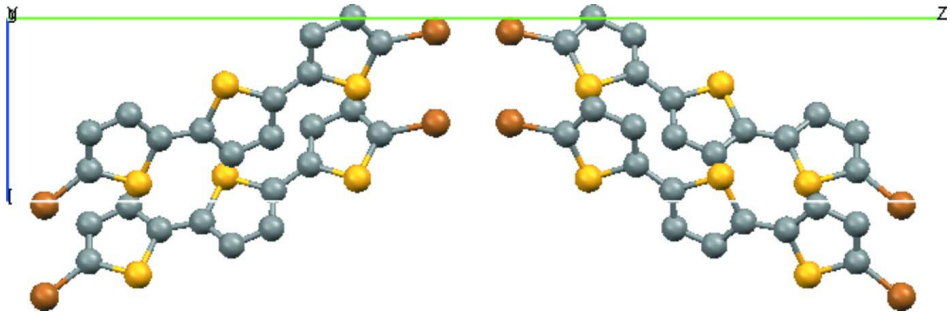

Figure 2

Crystal packing viewed along the *a* axis.

2,5-Bis(5-bromo-2-thienyl)thiophene

Crystal data

$C_{12}H_6Br_2S_3$
$M_r = 406.17$
Orthorhombic, *Pcc*2
Hall symbol: P 2 -2c
$a = 7.6216\ (16)$ Å
$b = 30.003\ (6)$ Å
$c = 5.8841\ (13)$ Å
$V = 1345.5\ (5)$ Å^3
$Z = 4$
$F(000) = 784$
$D_x = 2.005$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 903 reflections
$\theta = 2.7$–$27.5°$
$\mu = 6.46$ mm^{-1}
$T = 173$ K
Plate, pale yellow
0.37 × 0.24 × 0.10 mm

Data collection

Siemens SMART Platform CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2008a)
$T_{min} = 0.184$, $T_{max} = 0.524$
9565 measured reflections
3045 independent reflections
2818 reflections with $I > 2\sigma(I)$
$R_{int} = 0.035$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.7°$
$h = -9 \rightarrow 9$
$k = -38 \rightarrow 38$
$l = -7 \rightarrow 7$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.114$
$S = 1.25$
3045 reflections
155 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0403P)^2 + 2.9087P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.24$ e Å^{-3}
$\Delta\rho_{min} = -0.62$ e Å^{-3}
Absolute structure: Flack (1983), 1341 Friedel pairs
Absolute structure parameter: 0.00 (7)

Special details

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. The structure refined as a merohedral inversion twin, whose mass ratio converged to 61:39.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.69877 (9)	0.96042 (2)	1.02236 (11)	0.0374 (2)
Br2	0.71126 (13)	0.54014 (2)	0.07093 (13)	0.0532 (3)
S1	0.6713 (2)	0.86007 (5)	0.9031 (3)	0.0271 (3)
S2	0.81817 (19)	0.76622 (5)	0.3646 (3)	0.0232 (3)
S3	0.6749 (2)	0.62433 (5)	0.3757 (3)	0.0279 (3)
C1	0.7418 (8)	0.9124 (2)	0.8288 (11)	0.0253 (13)
C2	0.8278 (8)	0.9124 (2)	0.6263 (12)	0.0287 (14)
H2	0.8761	0.9384	0.5584	0.034*
C3	0.8376 (7)	0.86976 (19)	0.5284 (11)	0.0238 (12)
H3	0.8933	0.8642	0.3868	0.029*
C4	0.7597 (7)	0.8370 (2)	0.6553 (10)	0.0215 (12)
C5	0.7360 (7)	0.7908 (2)	0.6122 (10)	0.0172 (12)
C6	0.6539 (7)	0.75937 (18)	0.7416 (10)	0.0197 (12)
H6	0.6009	0.7660	0.8838	0.024*
C7	0.6546 (7)	0.71661 (19)	0.6471 (10)	0.0201 (12)
H7	0.6016	0.6916	0.7182	0.024*
C8	0.7397 (8)	0.71439 (19)	0.4407 (12)	0.0190 (12)
C9	0.7621 (7)	0.67576 (19)	0.2972 (10)	0.0175 (11)
C10	0.8467 (8)	0.6726 (2)	0.0932 (10)	0.0233 (12)
H10	0.9036	0.6972	0.0231	0.028*
C11	0.8421 (8)	0.62914 (19)	−0.0055 (11)	0.0249 (12)
H11	0.8927	0.6216	−0.1479	0.030*
C12	0.7553 (9)	0.5999 (2)	0.1323 (12)	0.0269 (13)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0447 (4)	0.0317 (4)	0.0359 (4)	0.0053 (3)	0.0051 (4)	−0.0089 (3)
Br2	0.0814 (7)	0.0272 (4)	0.0512 (7)	−0.0077 (4)	0.0045 (5)	−0.0099 (4)
S1	0.0315 (8)	0.0282 (7)	0.0217 (8)	−0.0025 (6)	0.0079 (6)	−0.0021 (6)
S2	0.0252 (7)	0.0259 (7)	0.0185 (7)	−0.0031 (6)	0.0045 (6)	0.0006 (6)
S3	0.0338 (8)	0.0234 (7)	0.0263 (8)	−0.0046 (6)	0.0066 (7)	0.0012 (7)
C1	0.028 (3)	0.025 (3)	0.023 (3)	0.002 (2)	−0.005 (3)	−0.003 (3)

C2	0.023 (3)	0.029 (3)	0.035 (3)	−0.002 (2)	−0.003 (3)	0.009 (3)
C3	0.026 (3)	0.024 (3)	0.021 (3)	0.000 (2)	0.006 (3)	0.004 (3)
C4	0.018 (3)	0.030 (3)	0.017 (3)	0.003 (2)	0.000 (2)	0.000 (2)
C5	0.012 (3)	0.026 (3)	0.013 (3)	−0.001 (2)	−0.003 (2)	−0.004 (2)
C6	0.019 (3)	0.023 (3)	0.017 (3)	−0.001 (2)	−0.001 (2)	0.001 (2)
C7	0.012 (3)	0.026 (3)	0.022 (3)	0.000 (2)	−0.001 (2)	0.005 (2)
C8	0.014 (2)	0.017 (3)	0.026 (3)	−0.002 (2)	0.004 (2)	0.010 (2)
C9	0.016 (3)	0.015 (3)	0.021 (3)	0.002 (2)	−0.001 (2)	0.000 (2)
C10	0.020 (3)	0.029 (3)	0.021 (3)	0.002 (2)	−0.001 (2)	0.003 (2)
C11	0.028 (3)	0.023 (3)	0.024 (3)	0.005 (2)	0.004 (3)	−0.006 (2)
C12	0.035 (3)	0.022 (3)	0.024 (3)	−0.004 (3)	0.000 (3)	−0.006 (3)

Geometric parameters (Å, º)

Br1—C1	1.864 (6)	C4—C5	1.419 (8)
Br2—C12	1.858 (6)	C5—C6	1.365 (8)
S1—C1	1.717 (7)	C6—C7	1.398 (8)
S1—C4	1.749 (6)	C6—H6	0.9500
S2—C8	1.725 (6)	C7—C8	1.379 (9)
S2—C5	1.750 (6)	C7—H7	0.9500
S3—C12	1.722 (7)	C8—C9	1.444 (8)
S3—C9	1.742 (6)	C9—C10	1.366 (8)
C1—C2	1.360 (10)	C10—C11	1.428 (8)
C2—C3	1.404 (9)	C10—H10	0.9500
C2—H2	0.9500	C11—C12	1.367 (9)
C3—C4	1.370 (8)	C11—H11	0.9500
C3—H3	0.9500		
C1—S1—C4	91.7 (3)	C7—C6—H6	122.9
C8—S2—C5	92.3 (3)	C8—C7—C6	113.4 (5)
C12—S3—C9	91.2 (3)	C8—C7—H7	123.3
C2—C1—S1	111.9 (5)	C6—C7—H7	123.3
C2—C1—Br1	128.4 (5)	C7—C8—C9	127.6 (5)
S1—C1—Br1	119.8 (4)	C7—C8—S2	110.4 (5)
C1—C2—C3	112.7 (6)	C9—C8—S2	122.1 (5)
C1—C2—H2	123.6	C10—C9—C8	128.7 (5)
C3—C2—H2	123.6	C10—C9—S3	110.6 (4)
C4—C3—C2	114.0 (6)	C8—C9—S3	120.7 (4)
C4—C3—H3	123.0	C9—C10—C11	114.2 (6)
C2—C3—H3	123.0	C9—C10—H10	122.9
C3—C4—C5	131.2 (5)	C11—C10—H10	122.9
C3—C4—S1	109.7 (5)	C12—C11—C10	110.9 (5)
C5—C4—S1	119.1 (4)	C12—C11—H11	124.5
C6—C5—C4	129.3 (5)	C10—C11—H11	124.5
C6—C5—S2	109.7 (4)	C11—C12—S3	113.0 (5)
C4—C5—S2	121.0 (4)	C11—C12—Br2	126.3 (5)
C5—C6—C7	114.3 (5)	S3—C12—Br2	120.6 (4)
C5—C6—H6	122.9		

C4—S1—C1—C2	0.0 (5)	C6—C7—C8—C9	−179.1 (6)
C4—S1—C1—Br1	−179.1 (4)	C6—C7—C8—S2	−0.3 (6)
S1—C1—C2—C3	0.1 (7)	C5—S2—C8—C7	0.0 (5)
Br1—C1—C2—C3	179.1 (5)	C5—S2—C8—C9	179.0 (5)
C1—C2—C3—C4	−0.2 (8)	C7—C8—C9—C10	−179.5 (6)
C2—C3—C4—C5	177.2 (6)	S2—C8—C9—C10	1.8 (9)
C2—C3—C4—S1	0.2 (7)	C7—C8—C9—S3	0.8 (9)
C1—S1—C4—C3	−0.1 (5)	S2—C8—C9—S3	−178.0 (3)
C1—S1—C4—C5	−177.6 (5)	C12—S3—C9—C10	0.2 (5)
C3—C4—C5—C6	−178.4 (7)	C12—S3—C9—C8	180.0 (5)
S1—C4—C5—C6	−1.6 (9)	C8—C9—C10—C11	−179.2 (6)
C3—C4—C5—S2	2.0 (9)	S3—C9—C10—C11	0.6 (7)
S1—C4—C5—S2	178.8 (3)	C9—C10—C11—C12	−1.3 (8)
C8—S2—C5—C6	0.2 (5)	C10—C11—C12—S3	1.4 (7)
C8—S2—C5—C4	179.8 (5)	C10—C11—C12—Br2	178.2 (5)
C4—C5—C6—C7	−180.0 (5)	C9—S3—C12—C11	−1.0 (5)
S2—C5—C6—C7	−0.4 (7)	C9—S3—C12—Br2	−177.9 (4)
C5—C6—C7—C8	0.4 (7)		