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1,3,5-Tris(5-iodothiophen-2-yl)benzene

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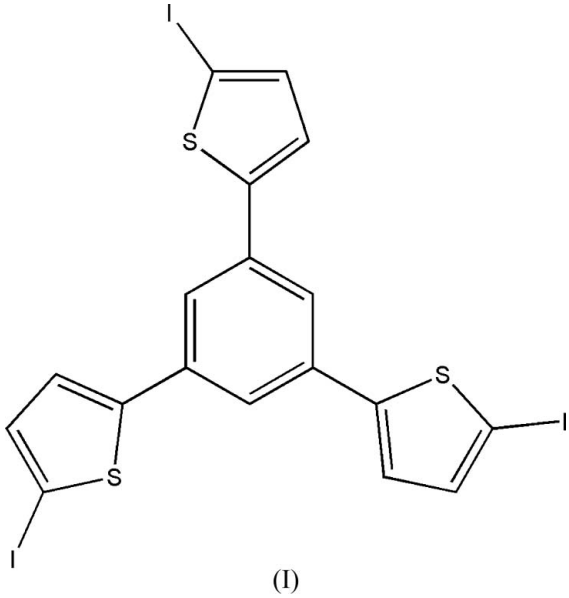
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.022 Å; R factor = 0.054; wR factor = 0.129; data-to-parameter ratio = 16.1.

The molecule of the title compound, $C_{18}H_9I_3S_3$, is nearly planar. The central benzene ring makes dihedral angles of 8.7 (1), 2.5 (6) and 11.0 (2)° with the three thiophene rings. The I···S separations of 3.5234 (4) and 3.5874 (3) Å show the short contacts between adjacent molecules.

Related literature

For general background, see: Perepichka *et al.* (2005); Hotta *et al.* (2004).



(I)

Experimental

Crystal data

$C_{18}H_9I_3S_3$
M_r = 702.13
Trigonal, $P3_221$
a = 12.969 (7) Å
c = 20.70 (2) Å
V = 3015 (4) Å^3
Z = 6
Mo $K\alpha$ radiation
μ = 4.97 mm^{-1}
T = 298 (2) K
0.41 × 0.27 × 0.23 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.188, T_{max} = 0.315
15743 measured reflections
3504 independent reflections
2631 reflections with $I > 2\sigma(I)$
R_{int} = 0.059

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.129
S = 1.04
3504 reflections
217 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.68 e Å^{-3}
$\Delta\rho_{min}$ = −0.70 e Å^{-3}
Absolute structure: Flack (1983), with 1477 Friedel pairs
Flack parameter: 0.02 (6)

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*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: XU2243).

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supplementary materials

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1,3,5-Tris(5-iodothiophen-2-yl)benzene

C.-H. Zhu, W.-J. Li, L.-F. Lv and L. Kong

Comment

Thiophene derivatives constitute a class of materials with various applications in conducting films, electrochromics and field-effect transistors (FETs) (Perepichka *et al.*, 2005). These materials are characterized by good physical properties, such as high carrier mobility and high luminescent quantum efficiencies (Hotta *et al.*, 2004). In this paper, the synthesis and crystal structure of the title thiophene derivative, (I), are reported.

The molecular structure of (I) is shown in Fig. 1, and the two-dimensional network of the compound is given in Fig. 2. The central benzene ring makes dihedral angles of 8.7 (1), 2.5 (6) and 11.0 (2)°, respectively, with the thiophene rings S1/C7–C10 (A), S2/C13–C16 (B) and S3/C19–C22 (C). The dihedral angles A/B, A/C and B/C are 7.8 (2), 12.3 (5) and 13.0 (1)°, respectively.

The I1···S2^{i} separation of 3.5234 (4) Å [symmetry code: (i) x, y+1, z] and I2···S3^{ii} separation of 3.5874 (3) Å [symmetry code: (ii) x+1, y, z] show the shorter contact between adjacent molecules.

Experimental

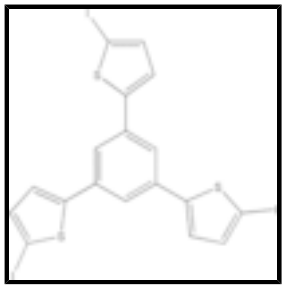
For the preparation of 1,3,5-tris(2-thienyl)benzene, a three-necked flask was charged with a mixture of 2-acetylthiophene (1 ml, 9.3 mmol) and dry ethanol (50 m) in ice–water, stirred vigorously. Tetrachlorosilane (10 ml, 88 mmol) was added to the solution. Stirring continued under nitrogen for 18 h. This mixture was poured in to water and saturated with ammonium chloride (100 ml), stirred vigorously and extracted with dichloromethane (4 × 100 ml). The organic layer was dried over anhydrous magnesium sulfate and removed under reduced pressure. The filtrate was purified by column chromatography with light

petroleum as the eluent, to give the white solid product (0.62 g, yield 61.73%). For the preparation of 1,3,5-tris(5-iodothiophen-2-yl)benzene, 1,3,5-tris(2-thienyl)benzene (0.9 g, 2.8 mmol) and benzene (15 ml) were added to a three-necked flask equipped with a magnetic stirrer, a reflux condenser and an isobaric dropping funnel. ICl (4.2 g, 25.8 mmol)/ethanol (15 ml) was added to the mixture at 353 K. The reaction mixture was refluxed for 2 h and then cooled to room temperature. The resulting grey solid was filtered off and air-dried after washing with dry ethanol three times, to give 1.68 g of the product (yield 86%). Single crystals of (I) were obtained by slow evaporation of a benzene solution at room temperature.

Refinement

H atoms were placed in geometrically idealized positions, with C—H = 0.93 Å, and refined in riding mode, with U_{iso}(H) = 1.2U_{eq}(C). Due to the low quality of the crystalline sample, the precision of the determination is poor.

Figures



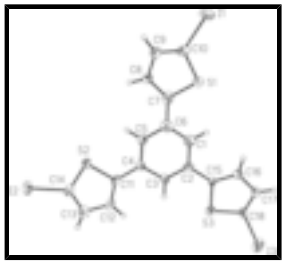


Fig. 1. The molecular structure of (I), showing 30% probability displacement ellipsoids.

1,3,5-Tris(5-iodothiophen-2-yl)benzene

Crystal data

$C_{18}H_9I_3S_3$	$Z = 6$
$M_r = 702.13$	$F_{000} = 1944$
Trigonal, $P3_221$	$D_x = 2.320$ Mg m^{-3}
Hall symbol: P 32 2"	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 12.969$ (7) Å	Cell parameters from 4393 reflections
$b = 12.969$ (7) Å	$\theta = 2.6$–22.7º
$c = 20.70$ (2) Å	$\mu = 4.98$ mm^{-1}
$\alpha = 90$º	$T = 298$ (2) K
$\beta = 90$º	Block, yellow
$\gamma = 120$º	0.41 × 0.27 × 0.23 mm
$V = 3015$ (4) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	3504 independent reflections
Radiation source: fine-focus sealed tube	2631 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.059$
$T = 298$(2) K	$\theta_{max} = 25.0$º
φ and ω scans	$\theta_{min} = 1.8$º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -9 \rightarrow 15$
$T_{min} = 0.188$, $T_{max} = 0.315$	$k = -15 \rightarrow 14$
15743 measured reflections	$l = -24 \rightarrow 23$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.054$	$w = 1/[\sigma^2(F_o^2) + (0.0615P)^2 + 25.2524P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.129$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.04$	$\Delta\rho_{max} = 0.68$ e Å^{-3}
3504 reflections	$\Delta\rho_{min} = -0.70$ e Å^{-3}
217 parameters	Extinction correction: none
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), with 1477 Friedel pairs
Secondary atom site location: difference Fourier map	Flack parameter: 0.02 (6)

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 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 $F^2 > 2$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}
I1	0.19980 (12)	0.83723 (10)	0.40014 (7)	0.0808 (4)
I2	0.61606 (10)	0.15440 (10)	0.41003 (6)	0.0643 (3)
I3	−0.47717 (10)	−0.26393 (11)	0.44787 (7)	0.0858 (4)
S1	0.2048 (3)	0.5886 (3)	0.3997 (2)	0.0549 (9)
S2	0.3634 (3)	0.1496 (3)	0.41515 (18)	0.0507 (9)
S3	−0.2304 (3)	−0.0070 (3)	0.4388 (2)	0.0601 (10)
C1	0.0984 (12)	0.3438 (11)	0.4149 (7)	0.045 (3)
C2	0.2091 (11)	0.3565 (12)	0.4124 (6)	0.043 (3)
H2	0.2752	0.4327	0.4110	0.051*
C3	0.2265 (11)	0.2563 (11)	0.4118 (6)	0.040 (3)
C4	0.1253 (12)	0.1468 (12)	0.4153 (6)	0.042 (3)
H4	0.1338	0.0797	0.4148	0.050*
C5	0.0093 (13)	0.1295 (13)	0.4194 (6)	0.051 (4)
C6	0.0005 (13)	0.2307 (12)	0.4196 (7)	0.050 (4)
H6	−0.0747	0.2228	0.4230	0.060*
C7	0.0855 (12)	0.4520 (11)	0.4141 (7)	0.043 (3)
C8	−0.0123 (12)	0.4593 (12)	0.4246 (7)	0.049 (4)

H8	−0.0857	0.3930	0.4341	0.059*
C9	0.0041 (13)	0.5752 (12)	0.4201 (7)	0.054 (4)
H9	−0.0553	0.5942	0.4272	0.064*
C10	0.1159 (12)	0.6535 (12)	0.4045 (7)	0.047 (3)
C11	0.3487 (12)	0.2755 (11)	0.4132 (7)	0.042 (3)
C12	0.4549 (13)	0.3736 (13)	0.4055 (7)	0.053 (4)
H12	0.4626	0.4485	0.4008	0.063*
C13	0.5548 (16)	0.3572 (17)	0.4050 (9)	0.074 (5)
H13	0.6342	0.4168	0.4019	0.088*
C14	0.5140 (13)	0.2392 (13)	0.4098 (6)	0.049 (3)
C15	−0.0910 (15)	0.0133 (13)	0.4214 (7)	0.055 (4)
C16	−0.1060 (14)	−0.0996 (13)	0.4122 (7)	0.060 (4)
H16	−0.0421	−0.1107	0.4028	0.072*
C17	−0.2139 (13)	−0.1891 (13)	0.4175 (7)	0.052 (4)
H17	−0.2329	−0.2677	0.4114	0.063*
C18	−0.2960 (14)	−0.1576 (12)	0.4325 (6)	0.050 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.0890 (9)	0.0417 (6)	0.1176 (10)	0.0371 (6)	0.0285 (8)	0.0172 (6)
I2	0.0623 (7)	0.0708 (8)	0.0793 (7)	0.0479 (6)	0.0112 (6)	0.0136 (6)
I3	0.0496 (6)	0.0606 (7)	0.1153 (10)	0.0035 (6)	0.0048 (6)	−0.0145 (7)
S1	0.048 (2)	0.0311 (19)	0.086 (3)	0.0201 (17)	0.006 (2)	−0.0017 (19)
S2	0.045 (2)	0.0367 (18)	0.075 (3)	0.0245 (16)	0.0127 (18)	0.0110 (18)
S3	0.041 (2)	0.0383 (19)	0.096 (3)	0.0161 (16)	−0.002 (2)	−0.006 (2)
C1	0.041 (7)	0.028 (7)	0.051 (8)	0.006 (6)	0.009 (7)	−0.007 (6)
C2	0.031 (7)	0.042 (8)	0.053 (8)	0.017 (6)	0.010 (6)	−0.003 (6)
C3	0.030 (7)	0.038 (7)	0.049 (8)	0.014 (6)	0.005 (6)	−0.001 (6)
C4	0.043 (8)	0.037 (7)	0.048 (8)	0.023 (6)	0.013 (7)	0.008 (6)
C5	0.055 (9)	0.057 (9)	0.034 (7)	0.022 (8)	0.008 (6)	0.016 (7)
C6	0.051 (9)	0.037 (8)	0.060 (9)	0.020 (7)	0.006 (7)	0.023 (7)
C7	0.035 (7)	0.035 (7)	0.068 (9)	0.024 (6)	−0.022 (7)	−0.013 (6)
C8	0.038 (8)	0.032 (7)	0.073 (10)	0.015 (6)	0.010 (7)	0.006 (7)
C9	0.038 (8)	0.047 (9)	0.083 (11)	0.027 (7)	−0.010 (8)	−0.014 (8)
C10	0.037 (8)	0.038 (7)	0.074 (9)	0.024 (6)	0.009 (7)	0.008 (7)
C11	0.048 (8)	0.021 (7)	0.061 (9)	0.020 (6)	0.007 (7)	0.002 (6)
C12	0.063 (9)	0.047 (8)	0.065 (10)	0.040 (8)	−0.002 (7)	−0.014 (7)
C13	0.046 (9)	0.089 (13)	0.091 (13)	0.038 (9)	−0.012 (9)	−0.008 (10)
C14	0.071 (10)	0.039 (8)	0.051 (8)	0.038 (8)	0.006 (7)	0.008 (7)
C15	0.068 (10)	0.052 (9)	0.055 (9)	0.038 (8)	0.008 (8)	0.028 (8)
C16	0.049 (9)	0.048 (9)	0.082 (11)	0.024 (8)	−0.003 (8)	−0.029 (8)
C17	0.054 (9)	0.045 (8)	0.061 (9)	0.028 (8)	−0.011 (7)	−0.004 (7)
C18	0.065 (10)	0.037 (7)	0.037 (7)	0.017 (7)	−0.004 (7)	−0.007 (6)

Geometric parameters (Å, °)

I1—C10	2.068 (13)	C5—C6	1.37 (2)
I2—C14	2.102 (13)	C5—C15	1.42 (2)

I3—C18	2.070 (15)	C6—H6	0.9300
S1—C7	1.697 (14)	C7—C8	1.336 (18)
S1—C10	1.737 (13)	C8—C9	1.412 (18)
S2—C14	1.705 (16)	C8—H8	0.9300
S2—C11	1.736 (12)	C9—C10	1.329 (19)
S3—C18	1.701 (14)	C9—H9	0.9300
S3—C15	1.730 (17)	C11—C12	1.34 (2)
C1—C2	1.362 (18)	C12—C13	1.42 (2)
C1—C6	1.382 (18)	C12—H12	0.9300
C1—C7	1.494 (18)	C13—C14	1.35 (2)
C2—C3	1.426 (19)	C13—H13	0.9300
C2—H2	0.9300	C15—C16	1.39 (2)
C3—C4	1.372 (18)	C16—C17	1.301 (19)
C3—C11	1.476 (17)	C16—H16	0.9300
C4—C5	1.408 (19)	C17—C18	1.35 (2)
C4—H4	0.9300	C17—H17	0.9300
C7—S1—C10	90.9 (6)	C8—C9—H9	124.6
C14—S2—C11	89.1 (7)	C9—C10—S1	112.3 (10)
C18—S3—C15	92.5 (8)	C9—C10—I1	129.4 (10)
C2—C1—C6	119.0 (13)	S1—C10—I1	117.5 (7)
C2—C1—C7	119.5 (12)	C12—C11—C3	131.7 (12)
C6—C1—C7	121.5 (13)	C12—C11—S2	110.6 (10)
C1—C2—C3	121.9 (12)	C3—C11—S2	117.1 (10)
C1—C2—H2	119.1	C11—C12—C13	116.2 (14)
C3—C2—H2	119.1	C11—C12—H12	121.9
C4—C3—C2	115.9 (12)	C13—C12—H12	121.9
C4—C3—C11	124.3 (12)	C14—C13—C12	107.5 (15)
C2—C3—C11	119.5 (12)	C14—C13—H13	126.3
C3—C4—C5	124.1 (13)	C12—C13—H13	126.3
C3—C4—H4	117.9	C13—C14—S2	116.4 (11)
C5—C4—H4	117.9	C13—C14—I2	126.9 (12)
C6—C5—C4	116.2 (14)	S2—C14—I2	116.7 (7)
C6—C5—C15	123.1 (15)	C16—C15—C5	133.5 (16)
C4—C5—C15	120.7 (15)	C16—C15—S3	106.4 (12)
C5—C6—C1	122.9 (15)	C5—C15—S3	120.1 (12)
C5—C6—H6	118.6	C17—C16—C15	116.7 (15)
C1—C6—H6	118.6	C17—C16—H16	121.6
C8—C7—C1	128.3 (13)	C15—C16—H16	121.6
C8—C7—S1	111.0 (10)	C16—C17—C18	114.1 (14)
C1—C7—S1	120.7 (10)	C16—C17—H17	123.0
C7—C8—C9	114.9 (13)	C18—C17—H17	123.0
C7—C8—H8	122.5	C17—C18—S3	110.3 (11)
C9—C8—H8	122.5	C17—C18—I3	129.5 (11)
C10—C9—C8	110.8 (13)	S3—C18—I3	120.2 (9)
C10—C9—H9	124.6		

Fig. 1

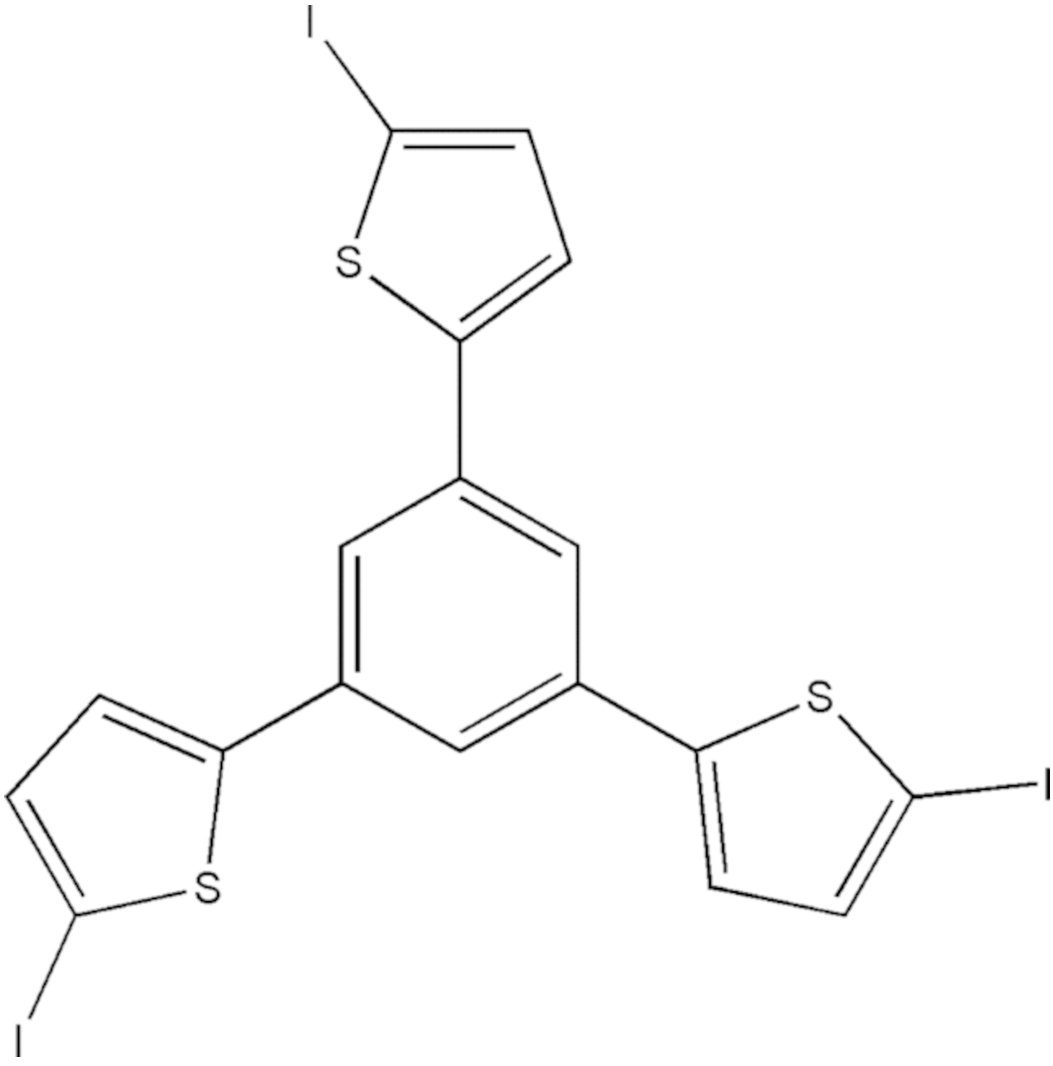
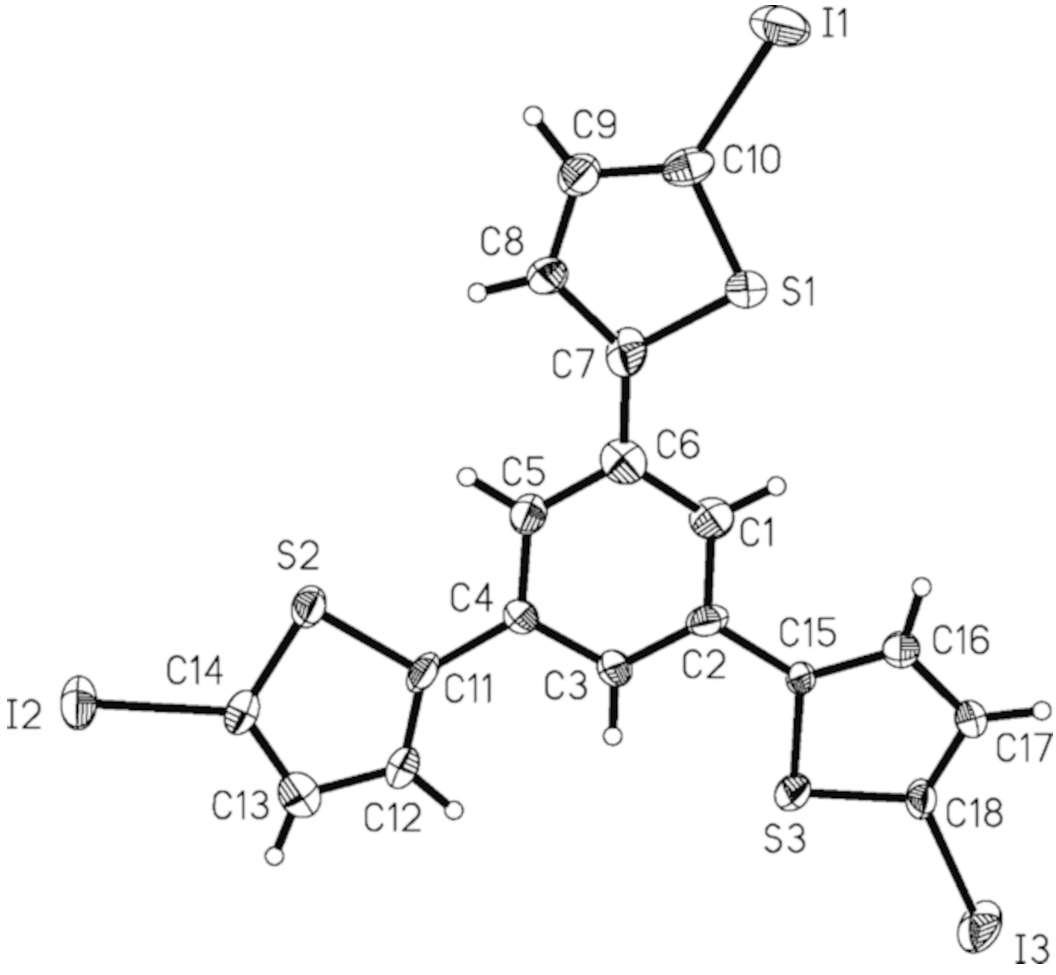


Fig. 2


I1
C9
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C15
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C3
C2
I2
C17
C13
C12
S3
C18
I3