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10,11-Dihydrodiindeno[1,2-*b*:2′,1′-*d*]-thiophene

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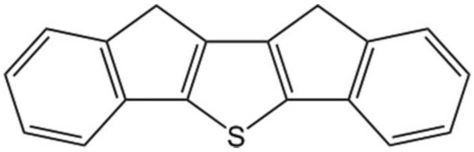
Key indicators: single-crystal X-ray study; T = 120 K; mean σ(C–C) = 0.004 Å; R factor = 0.063; wR factor = 0.159; data-to-parameter ratio = 16.6.

The title compound, $C_{18}H_{12}S$, comprises five fused rings forming an essentially planar molecule, with a total puckering amplitude (Q) of 0.032 Å and a maximum deviation from the mean plane of 0.014 (4) Å for the C atoms of the methylene groups. A crystallographic mirror plane orthogonal to the molecular plane passes through the S atom and the midpoint of the opposite C—C bond within the central five-membered ring. The molecules lie in layers, forming edge-to-face C—H$\cdots\pi$ interactions, with a separation of 2.66 Å between one H atom of the methylene group and the centroid of an adjacent indene ring.

Related literature

For related synthetic chemistry and properties, see: Boberg *et al.* (1983, 1994); Baierweck *et al.* (1988). For related structures, see: Klebl *et al.* (2002); Wong *et al.* (2006). The puckering amplitude (Q) is defined by Cremer & Pople (1975).



Experimental

Crystal data

$C_{18}H_{12}S$
M_r = 260.34
Orthorhombic, *Pnma*
a = 8.3358 (6) Å
b = 26.3096 (17) Å
c = 5.6923 (3) Å
V = 1248.39 (14) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 120 (2) K
0.26 × 0.15 × 0.01 mm

Data collection

Bruker–Nonius APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2007)
T_{min} = 0.940, T_{max} = 0.998
7885 measured reflections
1461 independent reflections
1201 reflections with $I > 2\sigma(I)$
R_{int} = 0.064

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.063
$wR(F^2)$ = 0.159
S = 1.07
1461 reflections
88 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.91 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 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, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *publCIF* (Westrip, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BI2268).

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supporting information

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10,11-Dihydrodiindeno[1,2-*b*:2′,1′-*d*]thiophene

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S1. Comment

The title compound forms an essentially planar molecule with just the two methylene groups deviating significantly from this plane. The maximum deviation from the mean plane of the molecule is 0.014 (4)Å for the methylene atoms and the fused ring system has a total puckering amplitude (Cremer & Pople, 1975) Q = 0.032, providing a further indicator of planarity. There are just two similar structures in the CSD: MUDYIT (Klebl *et al.*, 2002) and SERTUF (Wong *et al.*, 2006). However, these are unsuitable for comparison as they are not planar due to substitution with bulky functional groups at the methylene position.

The molecules lack hydrogen bonding functionality and pack in layers parallel to the (010) planes. Within these layers, one $C—H_{(methylene)}\cdots\pi_{(indene)}$ contact has a reasonably close separation of 2.66 Å between the H atom and the ring centroid.

S2. Experimental

The compound was prepared by following a similar methodology to that previously reported (Boberg *et al.*, 1994). Purity of the compound was confirmed by comparison of ^{1}H NMR spectroscopy with that already reported in the literature (Boberg *et al.*, 1983). ^{1}H-NMR ($CDC1_3$, δ, p.p.m.): 7.49 (dm, 4H, $3J$ = 8.4 Hz), 7.33 (t, 2H, $3J$ = 7.6 Hz), 7.19 (td, 2H, $3J$ = 7.6 Hz, $4J$ = 1.2 Hz), 3.73 (s; 4H). *M*.p.: 559–561 K (lit. 561 K; Baierweck *et al.* (1988)).

S3. Refinement

H atoms were fixed in idealized positions [C—H = 0.99 (CH_2) or 0.95 Å (CH)] and refined using the riding model with $U_{iso}(H) = 1.2U_{eq}(C)$.

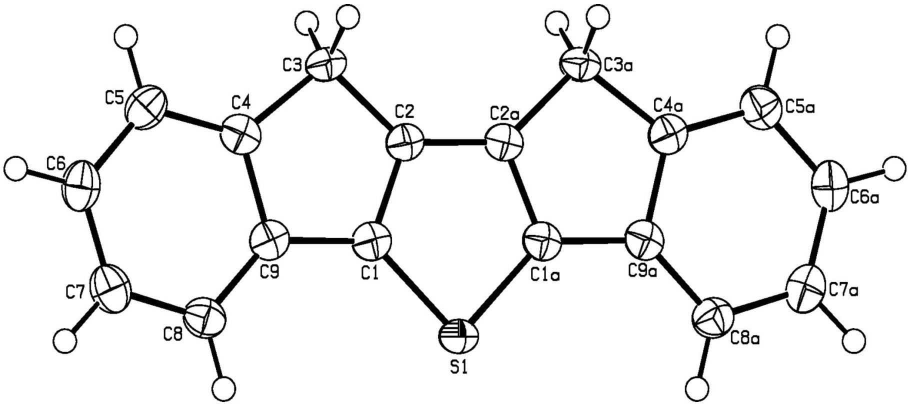

Figure 1

Molecular structure with 50% probability displacement ellipsoids for non-H atoms.

10,11-Dihydrodiindeno[1,2 - b:2′,1′-d]thiophene

Crystal data

$C_{18}H_{12}S$	$F(000) = 544$
$M_r = 260.34$	$D_x = 1.385$ Mg m^{-3}
Orthorhombic, *Pnma*	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ac 2n	Cell parameters from 9689 reflections
$a = 8.3358 (6)$ Å	$\theta = 2.9–27.5°$
$b = 26.3096 (17)$ Å	$\mu = 0.24$ mm^{-1}
$c = 5.6923 (3)$ Å	$T = 120$ K
$V = 1248.39 (14)$ Å^3	Plate, colourless
$Z = 4$	0.26 × 0.15 × 0.01 mm

Data collection

Bruker–Nonius APEXII CCD diffractometer	7885 measured reflections
Radiation source: Bruker-Nonius FR591 rotating anode	1461 independent reflections
10 cm confocal mirrors monochromator	1201 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{\text{int}} = 0.064$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2007)	$\theta_{\text{max}} = 27.5°$, $\theta_{\text{min}} = 3.1°$
$T_{\text{min}} = 0.940$, $T_{\text{max}} = 0.998$	$h = -10\rightarrow10$
	$k = -34\rightarrow33$
	$l = -7\rightarrow7$

Refinement

Refinement on F^2	0 restraints
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.063$	$w = 1/[\sigma^2(F_o^2) + (0.0523P)^2 + 2.4881P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.159$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.07$	$\Delta\rho_{\text{max}} = 0.91$ e Å^{-3}
1461 reflections	$\Delta\rho_{\text{min}} = -0.34$ e Å^{-3}
88 parameters	

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.

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

	x	y	z	U_{iso}*/U_{eq}
C1	0.3662 (3)	0.29623 (9)	0.0799 (4)	0.0255 (5)
C2	0.2804 (3)	0.27680 (9)	−0.1063 (4)	0.0242 (5)
C3	0.2105 (3)	0.31895 (9)	−0.2498 (4)	0.0263 (5)
H3A	0.0917	0.3178	−0.2492	0.032*
H3B	0.2494	0.3178	−0.414	0.032*
C4	0.2733 (3)	0.36587 (10)	−0.1208 (4)	0.0266 (6)
C5	0.2502 (3)	0.41677 (10)	−0.1715 (5)	0.0319 (6)
H5	0.1889	0.4267	−0.3045	0.038*
C6	0.3182 (4)	0.45307 (10)	−0.0244 (5)	0.0340 (7)
H6	0.3034	0.4882	−0.0571	0.041*

C7	0.4073 (4)	0.43846 (10)	0.1690 (5)	0.0344 (7)
H7	0.4524	0.4639	0.2674	0.041*
C8	0.4327 (3)	0.38786 (10)	0.2235 (5)	0.0298 (6)
H8	0.4946	0.3783	0.3565	0.036*
C9	0.3650 (3)	0.35156 (9)	0.0778 (4)	0.0251 (5)
S1	0.45076 (12)	0.25	0.25580 (16)	0.0275 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0217 (12)	0.0301 (12)	0.0248 (12)	0.0012 (10)	0.0025 (10)	0.0004 (10)
C2	0.0216 (12)	0.0289 (13)	0.0222 (11)	0.0005 (10)	0.0020 (10)	0.0012 (10)
C3	0.0263 (13)	0.0323 (12)	0.0204 (11)	0.0013 (10)	−0.0030 (10)	0.0017 (10)
C4	0.0224 (12)	0.0317 (13)	0.0257 (12)	0.0000 (10)	0.0035 (10)	−0.0027 (10)
C5	0.0340 (15)	0.0338 (13)	0.0277 (13)	0.0035 (12)	0.0036 (11)	0.0034 (11)
C6	0.0383 (16)	0.0261 (12)	0.0377 (15)	0.0022 (12)	0.0068 (12)	0.0004 (11)
C7	0.0348 (16)	0.0309 (13)	0.0375 (15)	−0.0018 (11)	0.0047 (13)	−0.0054 (11)
C8	0.0259 (13)	0.0341 (13)	0.0293 (13)	−0.0004 (11)	0.0003 (11)	−0.0020 (11)
C9	0.0202 (12)	0.0309 (12)	0.0243 (12)	0.0017 (10)	0.0051 (10)	0.0008 (10)
S1	0.0278 (5)	0.0291 (5)	0.0254 (5)	0	−0.0065 (4)	0

Geometric parameters (Å, º)

C1—C2	1.377 (4)	C5—C6	1.391 (4)
C1—C9	1.456 (3)	C5—H5	0.950
C1—S1	1.726 (3)	C6—C7	1.383 (4)
C2—C2^{i}	1.410 (5)	C6—H6	0.950
C2—C3	1.496 (3)	C7—C8	1.383 (4)
C3—C4	1.529 (3)	C7—H7	0.950
C3—H3A	0.990	C8—C9	1.385 (4)
C3—H3B	0.990	C8—H8	0.950
C4—C5	1.383 (4)	S1—C1^{i}	1.726 (3)
C4—C9	1.415 (4)		

C2—C1—C9	111.2 (2)	C4—C5—H5	120.6
C2—C1—S1	113.39 (19)	C6—C5—H5	120.6
C9—C1—S1	135.4 (2)	C7—C6—C5	120.5 (3)
C1—C2—C2^{i}	111.79 (15)	C7—C6—H6	119.8
C1—C2—C3	110.3 (2)	C5—C6—H6	119.8
C2^{i}—C2—C3	137.85 (13)	C6—C7—C8	121.9 (3)
C2—C3—C4	101.7 (2)	C6—C7—H7	119.1
C2—C3—H3A	111.4	C8—C7—H7	119.1
C4—C3—H3A	111.4	C7—C8—C9	117.9 (3)
C2—C3—H3B	111.4	C7—C8—H8	121.1
C4—C3—H3B	111.4	C9—C8—H8	121.1
H3A—C3—H3B	109.3	C8—C9—C4	121.0 (2)
C5—C4—C9	120.0 (2)	C8—C9—C1	133.0 (2)
C5—C4—C3	129.3 (2)	C4—C9—C1	106.0 (2)

C9—C4—C3	110.7 (2)	C1$^{\mathrm{i}}$—S1—C1	89.61 (17)
C4—C5—C6	118.8 (3)		

C9—C1—C2—C2$^{\mathrm{i}}$	−179.91 (15)	C7—C8—C9—C4	0.2 (4)
S1—C1—C2—C2$^{\mathrm{i}}$	0.9 (2)	C7—C8—C9—C1	179.9 (3)
C9—C1—C2—C3	−1.0 (3)	C5—C4—C9—C8	−0.1 (4)
S1—C1—C2—C3	179.78 (18)	C3—C4—C9—C8	−179.2 (2)
C1—C2—C3—C4	1.5 (3)	C5—C4—C9—C1	−179.8 (2)
C2$^{\mathrm{i}}$—C2—C3—C4	179.98 (13)	C3—C4—C9—C1	1.0 (3)
C2—C3—C4—C5	179.4 (3)	C2—C1—C9—C8	−179.7 (3)
C2—C3—C4—C9	−1.5 (3)	S1—C1—C9—C8	−0.7 (5)
C9—C4—C5—C6	0.0 (4)	C2—C1—C9—C4	0.0 (3)
C3—C4—C5—C6	179.0 (3)	S1—C1—C9—C4	179.0 (2)
C4—C5—C6—C7	0.0 (4)	C2—C1—S1—C1$^{\mathrm{i}}$	−1.1 (3)
C5—C6—C7—C8	0.2 (4)	C9—C1—S1—C1$^{\mathrm{i}}$	179.9 (2)
C6—C7—C8—C9	−0.3 (4)		

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