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1,3-Dimethylbenzo[*b*]dibenzothiophene

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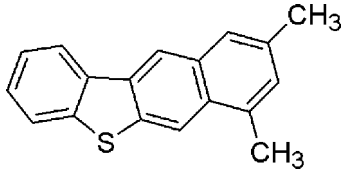
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.003 Å; R factor = 0.046; wR factor = 0.137; data-to-parameter ratio = 17.4.

The molecule of the title compound, $C_{18}H_{14}S$, is approximately planar (r.m.s. deviation = 0.029 Å). The crystal packing is stabilized by weak intermolecular C—H$\cdots\pi$ interactions.

Related literature

For the pharmacological activities of thiophen derivatives, see: Dzhurayev *et al.* (1992); El-Maghraby *et al.* (1984); Gewald *et al.* (1996). For related structures, see: Harrison *et al.* (2006); Palani *et al.* (2006). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{18}H_{14}S$
M_r = 262.35
Monoclinic, $P2_1/n$
a = 10.0219 (3) Å
b = 5.8692 (5) Å
c = 22.8554 (5) Å
β = 99.787 (1)°
V = 1324.80 (12) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 295 (2) K
0.26 × 0.20 × 0.18 mm

Data collection

Bruker Kappa APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.944, T_{max} = 0.961
27929 measured reflections
3030 independent reflections
2574 reflections with $I > 2\sigma(I)$
R_{int} = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.137
S = 1.08
3030 reflections
174 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

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

*Cg*1 is the centroid of the C7–C9/C14–C16 ring and *Cg*2 is the centroid of the C1–C6 ring.

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C17—H17*C*$\cdots$*Cg*1^{i}	0.96	2.68	3.486 (2)	142
C18—H18*A*$\cdots$*Cg*2^{ii}	0.96	2.75	3.649 (3)	155

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

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: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2861).

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supplementary materials

Acta Cryst. (2009). E**65**, o440 [**doi:10.1107/S1600536809003456**]

1,3-Dimethylbenzo[*b*]dibenzothiophene

P. R. Umarani, A. Marx, V. Dhayalan, A. K. Mohanakrishnan and V. Manivannan

Comment

Thiophen derivatives possess pharmacological activities such as anti-bacterial, anti-cancer, anti-inflammatory (El-Maghraby *et al.*, 1984; Dzhurayev *et al.*, 1992) and anti-toxic properties (Gewald *et al.*, 1996).

The geometric parameters of the title molecule (Fig. 1) agree well with related structures (Harrison *et al.*, 2006; Palani *et al.*, 2006) and literature values (Allen *et al.*, 1987). All non-H atoms lie in a common plane (r.m.s. deviation 0.029Å) .

The crystal packing is stabilized by weak intermolecular C - H···π [C17—H17C···*Cg*1 (1 - *x*, -*y*, 1 - *z*), H17C···*Cg*1 = 2.68 Å, C18—H18A··· *Cg*2 (1 + *x*, *y*, *z*), H18A···*Cg*1 = 2.75 Å; *Cg*1 and *Cg*2 are the centroid of rings defined by atoms C7/C8/C9/C14/C15/C16 and C1—C6, respectively) interactions. No significant intra- and intermolecular hydrogen bonds are observed.

Experimental

To a solution of diethyl 2-((2-(bromomethyl)banzo[*b*]thiophen-3-yl) methylene)malonate (0.35 g, 0.88 mmol) in dry 1,2-DCE (15 ml), $ZnBr_2$ (0.39 g, 1.73 mmol) and *m*-xylene (0.13 ml, 1.03 mmol), were added. The reaction mixture was then refluxed for 2 h under N_2 atmosphere. It was then poured over ice-water (50 ml) containing 2 ml of conc.HCl, extracted with chloroform (3 *X* 10 ml) and dried (Na_2SO_4). The removal of solvent followed by flash column chromatographic purification (silica gel, 230–420 mesh, n-hexane/ethyl acetate 99:1) afforded 1,3-dimethylbenzo[2,3-*b*] dibenzothiophene as a colourless crystal.

Refinement

H atoms were positioned geometrically and refined using a riding model with C—H = 0.93 Å and U_{iso}(H) = 1.2Ueq(C) for aromatic H atoms and C—H = 0.96 Å and U_{iso}(H) = 1.5Ueq(C) for methyl H atoms.

Figures

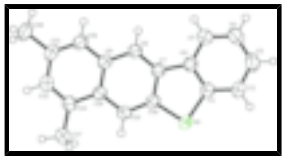

Fig. 1. The molecular structure of (I), with atom labels and 50% probability displacement ellipsoids for non-H atoms.

1,3-Dimethylbenzo[*b*]dibenzothiophene

Crystal data

$C_{18}H_{14}S$	F_{000} = 552
M_r = 262.35	D_x = 1.315 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 6370 reflections
a = 10.0219 (3) Å	θ = 2.1–27.4º
b = 5.8692 (5) Å	μ = 0.23 mm^{-1}
c = 22.8554 (5) Å	T = 295 (2) K
β = 99.787 (1)º	Block, colourless
V = 1324.80 (12) Å^3	0.26 × 0.20 × 0.18 mm
Z = 4	

Data collection

Bruker Kappa APEXII diffractometer	3030 independent reflections
Radiation source: fine-focus sealed tube	2574 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.029
T = 295(2) K	θ_{max} = 27.5º
ω and φ scans	θ_{min} = 1.8º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −13→13
T_{min} = 0.944, T_{max} = 0.961	k = −7→7
27929 measured reflections	l = −29→29

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.046	H-atom parameters constrained
$wR(F^2)$ = 0.137	$w = 1/[\sigma^2(F_o^2) + (0.0636P)^2 + 0.5697P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.08	$(\Delta/\sigma)_{max} < 0.001$
3030 reflections	$\Delta\rho_{max}$ = 0.26 e Å^{-3}
174 parameters	$\Delta\rho_{min}$ = −0.29 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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

	x	*y*	*z*	U_{iso}*/U_{eq}

C1	0.07838 (18)	−0.0783 (3)	0.33166 (8)	0.0456 (4)
C2	−0.0587 (2)	−0.0829 (4)	0.30694 (10)	0.0567 (5)
H2	−0.1153	−0.1953	0.3177	0.068*
C3	−0.1081 (2)	0.0813 (4)	0.26653 (9)	0.0603 (6)
H3	−0.1993	0.0801	0.2496	0.072*
C4	−0.0245 (2)	0.2490 (4)	0.25047 (9)	0.0579 (5)
H4	−0.0599	0.3589	0.2228	0.069*
C5	0.1110 (2)	0.2546 (3)	0.27511 (8)	0.0494 (5)
H5	0.1667	0.3678	0.2641	0.059*
C6	0.16390 (17)	0.0909 (3)	0.31632 (7)	0.0403 (4)
C7	0.30153 (17)	0.0662 (3)	0.34821 (7)	0.0378 (4)
C8	0.41378 (18)	0.1971 (3)	0.34603 (7)	0.0408 (4)
H8	0.4071	0.3228	0.3209	0.049*
C9	0.53930 (17)	0.1436 (3)	0.38139 (7)	0.0390 (4)
C10	0.65545 (19)	0.2770 (3)	0.37964 (8)	0.0472 (4)
H10	0.6490	0.4027	0.3545	0.057*
C11	0.77693 (19)	0.2268 (3)	0.41376 (9)	0.0480 (4)
C12	0.78456 (18)	0.0376 (3)	0.45225 (8)	0.0477 (4)
H12	0.8674	0.0037	0.4757	0.057*
C13	0.67622 (18)	−0.0979 (3)	0.45659 (8)	0.0419 (4)
C14	0.54908 (17)	−0.0488 (3)	0.41992 (7)	0.0381 (4)
C15	0.43329 (18)	−0.1822 (3)	0.42159 (8)	0.0437 (4)
H15	0.4381	−0.3086	0.4464	0.052*
C16	0.31376 (18)	−0.1253 (3)	0.38656 (8)	0.0418 (4)
C17	0.6892 (2)	−0.2914 (4)	0.49988 (9)	0.0523 (5)
H17A	0.7795	−0.2944	0.5222	0.078*
H17B	0.6709	−0.4324	0.4787	0.078*
H17C	0.6256	−0.2714	0.5265	0.078*
C18	0.9007 (2)	0.3703 (4)	0.41279 (11)	0.0648 (6)
H18A	0.9598	0.2941	0.3902	0.097*
H18B	0.9471	0.3935	0.4527	0.097*
H18C	0.8742	0.5149	0.3949	0.097*
S1	0.16072 (5)	−0.26890 (9)	0.38419 (3)	0.0592 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0419 (9)	0.0449 (10)	0.0474 (9)	−0.0078 (8)	0.0001 (7)	−0.0003 (8)
C2	0.0429 (10)	0.0597 (12)	0.0632 (12)	−0.0153 (9)	−0.0030 (9)	0.0030 (10)
C3	0.0429 (10)	0.0772 (15)	0.0557 (11)	−0.0015 (10)	−0.0060 (8)	−0.0008 (10)
C4	0.0542 (11)	0.0668 (14)	0.0491 (11)	0.0049 (10)	−0.0018 (9)	0.0105 (9)
C5	0.0511 (10)	0.0519 (11)	0.0442 (9)	−0.0012 (8)	0.0053 (8)	0.0069 (8)
C6	0.0406 (9)	0.0429 (9)	0.0367 (8)	−0.0034 (7)	0.0042 (7)	−0.0033 (7)
C7	0.0391 (8)	0.0378 (8)	0.0361 (8)	−0.0034 (7)	0.0054 (6)	−0.0014 (6)
C8	0.0425 (9)	0.0404 (9)	0.0393 (8)	−0.0059 (7)	0.0068 (7)	0.0049 (7)
C9	0.0388 (8)	0.0408 (9)	0.0383 (8)	−0.0050 (7)	0.0089 (6)	−0.0013 (7)
C10	0.0447 (10)	0.0488 (10)	0.0494 (10)	−0.0102 (8)	0.0120 (8)	0.0035 (8)
C11	0.0391 (9)	0.0550 (11)	0.0513 (10)	−0.0108 (8)	0.0116 (8)	−0.0053 (8)

C12	0.0352 (8)	0.0562 (11)	0.0507 (10)	0.0011 (8)	0.0042 (7)	−0.0036 (8)
C13	0.0401 (9)	0.0432 (9)	0.0422 (9)	0.0020 (7)	0.0066 (7)	−0.0026 (7)
C14	0.0387 (8)	0.0371 (9)	0.0386 (8)	−0.0022 (7)	0.0068 (6)	−0.0023 (7)
C15	0.0445 (9)	0.0353 (9)	0.0497 (10)	−0.0051 (7)	0.0036 (7)	0.0050 (7)
C16	0.0417 (9)	0.0364 (9)	0.0461 (9)	−0.0101 (7)	0.0036 (7)	0.0007 (7)
C17	0.0486 (11)	0.0520 (11)	0.0544 (11)	0.0057 (9)	0.0033 (8)	0.0060 (9)
C18	0.0422 (10)	0.0745 (15)	0.0786 (15)	−0.0188 (10)	0.0127 (10)	−0.0002 (12)
S1	0.0460 (3)	0.0503 (3)	0.0749 (4)	−0.0193 (2)	−0.0080 (2)	0.0180 (2)

Geometric parameters (Å, °)

C1—C2	1.394 (3)	C10—C11	1.362 (3)
C1—C6	1.395 (3)	C10—H10	0.9300
C1—S1	1.7425 (19)	C11—C12	1.411 (3)
C2—C3	1.368 (3)	C11—C18	1.502 (3)
C2—H2	0.9300	C12—C13	1.363 (3)
C3—C4	1.382 (3)	C12—H12	0.9300
C3—H3	0.9300	C13—C14	1.430 (2)
C4—C5	1.379 (3)	C13—C17	1.497 (3)
C4—H4	0.9300	C14—C15	1.406 (2)
C5—C6	1.386 (3)	C15—C16	1.364 (2)
C5—H5	0.9300	C15—H15	0.9300
C6—C7	1.454 (2)	C16—S1	1.7428 (17)
C7—C8	1.370 (2)	C17—H17A	0.9600
C7—C16	1.418 (2)	C17—H17B	0.9600
C8—C9	1.410 (2)	C17—H17C	0.9600
C8—H8	0.9300	C18—H18A	0.9600
C9—C10	1.409 (2)	C18—H18B	0.9600
C9—C14	1.425 (2)	C18—H18C	0.9600
C2—C1—C6	121.14 (18)	C10—C11—C18	122.00 (19)
C2—C1—S1	125.84 (16)	C12—C11—C18	119.55 (19)
C6—C1—S1	113.01 (13)	C13—C12—C11	123.04 (17)
C3—C2—C1	118.62 (19)	C13—C12—H12	118.5
C3—C2—H2	120.7	C11—C12—H12	118.5
C1—C2—H2	120.7	C12—C13—C14	118.76 (16)
C2—C3—C4	120.96 (19)	C12—C13—C17	120.64 (17)
C2—C3—H3	119.5	C14—C13—C17	120.59 (16)
C4—C3—H3	119.5	C15—C14—C9	119.19 (16)
C5—C4—C3	120.53 (19)	C15—C14—C13	121.95 (16)
C5—C4—H4	119.7	C9—C14—C13	118.85 (15)
C3—C4—H4	119.7	C16—C15—C14	119.62 (16)
C4—C5—C6	119.81 (19)	C16—C15—H15	120.2
C4—C5—H5	120.1	C14—C15—H15	120.2
C6—C5—H5	120.1	C15—C16—C7	122.13 (16)
C5—C6—C1	118.93 (16)	C15—C16—S1	125.46 (14)
C5—C6—C7	129.09 (17)	C7—C16—S1	112.39 (13)
C1—C6—C7	111.98 (15)	C13—C17—H17A	109.5
C8—C7—C16	118.80 (15)	C13—C17—H17B	109.5
C8—C7—C6	129.86 (16)	H17A—C17—H17B	109.5

C16—C7—C6	111.34 (15)	C13—C17—H17C	109.5
C7—C8—C9	120.82 (16)	H17A—C17—H17C	109.5
C7—C8—H8	119.6	H17B—C17—H17C	109.5
C9—C8—H8	119.6	C11—C18—H18A	109.5
C10—C9—C8	121.39 (16)	C11—C18—H18B	109.5
C10—C9—C14	119.18 (16)	H18A—C18—H18B	109.5
C8—C9—C14	119.43 (15)	C11—C18—H18C	109.5
C11—C10—C9	121.72 (18)	H18A—C18—H18C	109.5
C11—C10—H10	119.1	H18B—C18—H18C	109.5
C9—C10—H10	119.1	C1—S1—C16	91.28 (9)
C10—C11—C12	118.42 (17)		
C6—C1—C2—C3	0.7 (3)	C18—C11—C12—C13	178.60 (19)
S1—C1—C2—C3	179.28 (17)	C11—C12—C13—C14	0.9 (3)
C1—C2—C3—C4	−0.1 (3)	C11—C12—C13—C17	−177.74 (17)
C2—C3—C4—C5	−0.2 (4)	C10—C9—C14—C15	−179.87 (16)
C3—C4—C5—C6	0.0 (3)	C8—C9—C14—C15	0.4 (3)
C4—C5—C6—C1	0.6 (3)	C10—C9—C14—C13	1.3 (2)
C4—C5—C6—C7	−179.00 (19)	C8—C9—C14—C13	−178.50 (16)
C2—C1—C6—C5	−0.9 (3)	C12—C13—C14—C15	179.42 (17)
S1—C1—C6—C5	−179.67 (14)	C17—C13—C14—C15	−1.9 (3)
C2—C1—C6—C7	178.73 (18)	C12—C13—C14—C9	−1.7 (3)
S1—C1—C6—C7	0.0 (2)	C17—C13—C14—C9	176.94 (16)
C5—C6—C7—C8	0.2 (3)	C9—C14—C15—C16	−0.2 (3)
C1—C6—C7—C8	−179.43 (18)	C13—C14—C15—C16	178.66 (17)
C5—C6—C7—C16	−179.98 (18)	C14—C15—C16—C7	−0.3 (3)
C1—C6—C7—C16	0.4 (2)	C14—C15—C16—S1	−178.84 (14)
C16—C7—C8—C9	−0.3 (3)	C8—C7—C16—C15	0.5 (3)
C6—C7—C8—C9	179.59 (17)	C6—C7—C16—C15	−179.40 (17)
C7—C8—C9—C10	−179.90 (16)	C8—C7—C16—S1	179.24 (13)
C7—C8—C9—C14	−0.1 (3)	C6—C7—C16—S1	−0.65 (19)
C8—C9—C10—C11	179.83 (18)	C2—C1—S1—C16	−179.0 (2)
C14—C9—C10—C11	0.1 (3)	C6—C1—S1—C16	−0.29 (15)
C9—C10—C11—C12	−0.9 (3)	C15—C16—S1—C1	179.24 (18)
C9—C10—C11—C18	−179.07 (19)	C7—C16—S1—C1	0.54 (14)
C10—C11—C12—C13	0.4 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C17—H17C···Cg1^{i}	0.96	2.68	3.486 (2)	142
C18—H18A···Cg2^{ii}	0.96	2.75	3.649 (3)	155

Symmetry codes: (i) −*x*+1, −*y*, −*z*+1; (ii) *x*+1, *y*, *z*.

Fig. 1