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2,6,8-Trimethyl-3,5-bis(methylsulfanyl)benzo[1,2-*b*:5,4-*b*′]difuran

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.003 Å
R factor = 0.046
wR factor = 0.135
Data-to-parameter ratio = 17.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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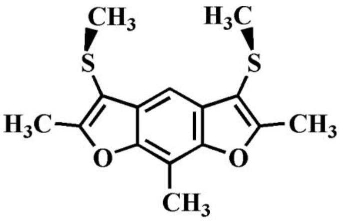
The title compound, $C_{15}H_{16}O_2S_2$, was prepared by the Lewis acid-catalysed reaction of 2-methylresorcinol with α-chloro-α-(methylsulfanyl)acetone. The benzo[1,2-*b*:5,4-*b*′]difuran group is almost planar. The crystal structure is stabilized by aromatic π–π stacking and $—CH_2—H\cdots\pi$ interactions.

Comment

This work follows on from our previous reports on the benzodifuran analogues, 2,5-dimethyl-3,4-bis(methylsulfanyl)benzo[1,2-*b*:4,3-*b*′]difuran (Choi, Jang *et al.*, 2006) and 2,4,7-trimethyl-3,8-bis(methylsulfanyl)benzo[1,2-*b*:5,6-*b*′]difuran (Choi, Seo *et al.*, 2006). The isomeric title compound, (I) (Fig. 1), was obtained by the one-pot reaction of 2-methylresorcinol with an excess of α-chloro-α-(methylsulfanyl)acetone in the presence of zinc chloride.



(I)

The bond lengths and angles in (I) are within normal ranges (Allen *et al.*, 1987; Choi, Jang *et al.*, 2006; Choi, Seo *et al.*, 2006). The benzo[1,2-*b*:5,4-*b*′]difuran unit is nearly planar,

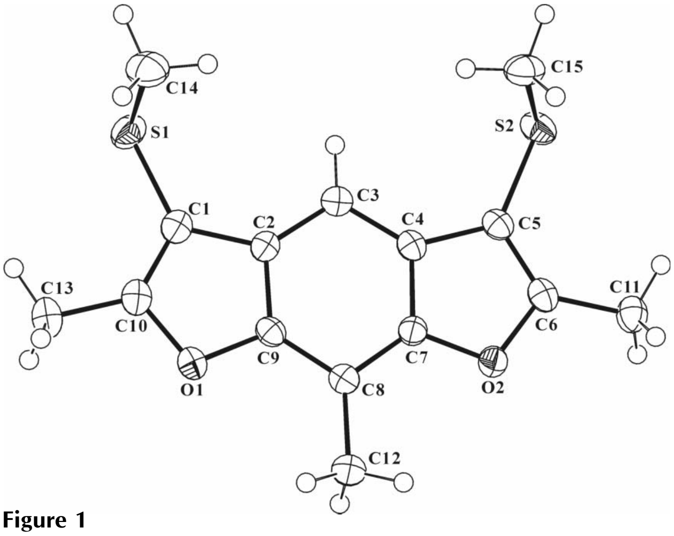


Figure 1
The molecular structure of (I), with 50% probability displacement ellipsoids for non-H atoms.

with a mean deviation of 0.014 Å from the least-squares plane defined by the 12 constituent C and O atoms. The methyl groups of the methylsulfanyl substituents point in the same direction with respect to the plane of the ring system. Atoms S1 and S2 lie 0.022 (2) and 0.088 (3) Å, respectively, out of the C1/C2/C9/O1/C10 and C4–C6/O2/C7 planes.

The crystal structure is stabilized by aromatic π–π stacking and $-CH_2-H\cdots\pi$ interactions (Fig. 2). The closest approach between the benzodifuran planes (BF) is $BF\cdots BF^{iii}$ = 3.556 (5) Å, while the nearest ring-centroid separation, $Cg2\cdots Cg3^{iii}$, is 3.894 (4) Å ($Cg2$ and $Cg3$ are the centroids of the C2–C4/C7–C9 and C4–C6/O2/C7 rings, respectively; symmetry code as in Fig. 2). These data indicate the existence of a weak π–π stacking interaction. The packing of the molecules is additionally stabilized by the $-CH_2-H\cdots\pi$ interactions between adjacent furan rings (Table 1).

Experimental

The title compound was prepared according to the method described for the previously reported isomer (Choi, Seo *et al.*, 2006), using 2-methylresorcinol instead of 5-methylresorcinol. Single crystals suitable for X-ray analysis were obtained by slow evaporation of a tetrahydrofuran solution [yield 53%; m.p. 403–404 K; R_F = 0.67 (CCl_4)].

Crystal data

$C_{15}H_{16}O_2S_2$
M_r = 292.40
Monoclinic, $P2_1/c$
a = 7.895 (3) Å
b = 13.008 (5) Å
c = 14.498 (6) Å
β = 104.479 (7)°
V = 1441.6 (10) Å^3
Z = 4
D_x = 1.347 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.36 mm^{-1}
T = 173 (2) K
Block, colorless
0.65 × 0.58 × 0.54 mm

Data collection

Bruker SMART CCD diffractometer
φ and ω scans
Absorption correction: none
8031 measured reflections
3097 independent reflections
2702 reflections with $I > 2\sigma(I)$
R_{int} = 0.070
θ_{max} = 27.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.135
S = 1.05
3097 reflections
173 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0664P)^2 + 1.1009P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

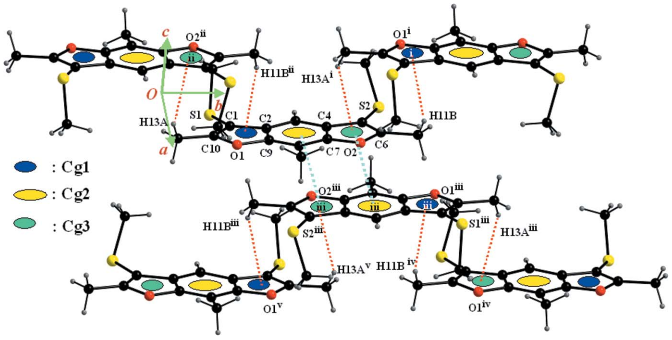


Figure 2
The C—H···π and π–π interactions (dotted lines) in (I). *Cgn* denotes the centroid of each ring. [Symmetry codes: (i) $1 - x, \frac{1}{2} + y, \frac{1}{2} - z$; (ii) $1 - x, y - \frac{1}{2}, \frac{1}{2} - z$; (iii) $1 - x, 1 - y, -z$; (iv) $x, \frac{3}{2} - y, z - \frac{1}{2}$; (v) $x, \frac{1}{2} - y, z - \frac{1}{2}$.]

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

D—H···A	D—H	H···A	D···A	D—H···A
C11—H11B···$Cg1^i$	0.96	2.75	3.480 (3)	133
C13—H13A···$Cg3^{ii}$	0.96	2.82	3.586 (3)	138

Symmetry codes: (i) $-x + 1, y + \frac{1}{2}, -z + \frac{1}{2}$; (ii) $-x + 1, y - \frac{1}{2}, -z + \frac{1}{2}$. $Cg1$ is the centroid of the C1/C2/C9/O1/C10 ring and $Cg3$ is the centroid of the C4–C6/O2/C7 ring.

All H atoms were positioned geometrically (C—H = 0.93–0.96 Å) and refined as riding, with $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}$(methyl C).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 1998); software used to prepare material for publication: *SHELXL97*.

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