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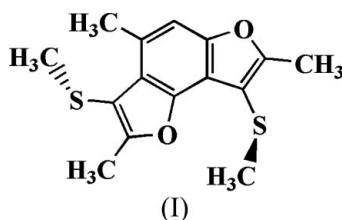
Key indicators

Single-crystal X-ray study
 $T = 298$ K
Mean $\sigma(\text{C}-\text{C}) = 0.005$ Å
 R factor = 0.067
 wR factor = 0.167
Data-to-parameter ratio = 18.7For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.2,4,7-Trimethyl-3,8-bis(methylsulfanyl)-benzo[1,2-*b*:5,6-*b'*]difuran

The title compound, $\text{C}_{15}\text{H}_{16}\text{O}_2\text{S}_2$, was prepared by the Lewis acid-catalysed reaction of 5-methylresorcinol with α -chloro- α -(methylsulfanyl)acetone. In the crystal structure, aromatic π - π stacking interactions are not observed between adjacent molecules, because of steric hindrance induced by the five methyl groups in the molecule.

Comment

Benzodifuran ring systems have attracted interest as compounds with biological activity and have potential applications as photosensitizers and fluorescent probes (Bilger *et al.*, 1987; Chambers *et al.*, 2001; Rene *et al.*, 1978). We have shown great interest in the synthesis and structural analysis of a series of benzodifuran derivatives (Choi *et al.*, 2006; Kang *et al.*, 2004; Takahashi & Kobayashi, 2000). The one-pot reaction of 5-methylresorcinol with an excess of α -chloro- α -(methylsulfanyl)acetone in the presence of zinc chloride gave the title compound, (I).



The molecule lies on a general position (Fig. 1). The bond lengths and angles are within normal ranges (Allen *et al.*, 1987; Choi *et al.*, 2006). The benzo[1,2-*b*:5,6-*b'*]difuran unit is almost planar, with a mean deviation from the least-squares plane of 0.013 Å. The dihedral angles between the central benzene ring and the furan rings are 0.4 (2)° (C1-C3/C10/O1) and 1.6 (2)° (C6-C9/O2). The methyl groups of the methylsulfanyl substituents point in opposite directions with respect to the plane of the ring system. Atoms S1 and S2 lie 0.068 (5) and 0.001 (6) Å out of the C1-C3/C10/O1 and C6-C9/O2 planes, respectively.

Fig. 2 shows the packing of (I) in the crystal structure. The closest approach between the benzodifuran planes (BF) is $\text{BF} \cdots \text{BF}^i = 3.725$ (8) Å, but the nearest ring centroid separation, $\text{Cg} \cdots \text{Cg}^i$, is 4.139 (5) Å [Cg is the centroid of the C6-C9/O2 ring; symmetry code: (i) $1 - x, 1 - y, 1 - z$]. The angle $\text{C9} \cdots \text{Cg} \cdots \text{Cg}^i$ is 115.8 (3)°. These data clearly indicate the absence of significant π - π stacking interactions. It thus seems that the skewed conformation of the rings of (I) is not efficient for the formation of a π - π stacked crystal structure. This feature may also be related to the steric hindrance induced by the five methyl groups in the molecule.

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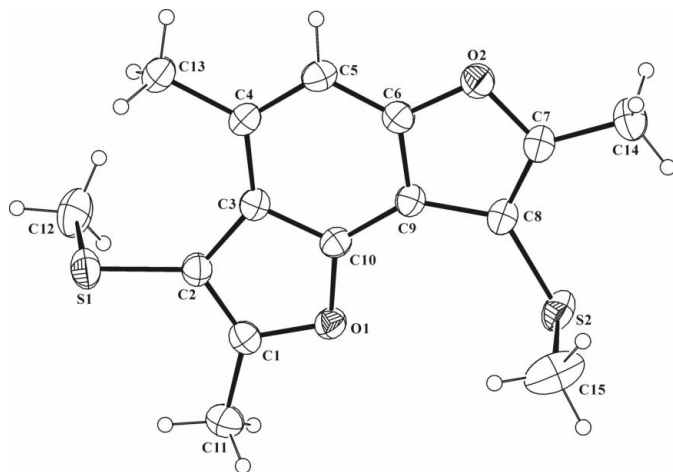


Figure 1
A view of the structure of (I), with 30% probability displacement ellipsoids.

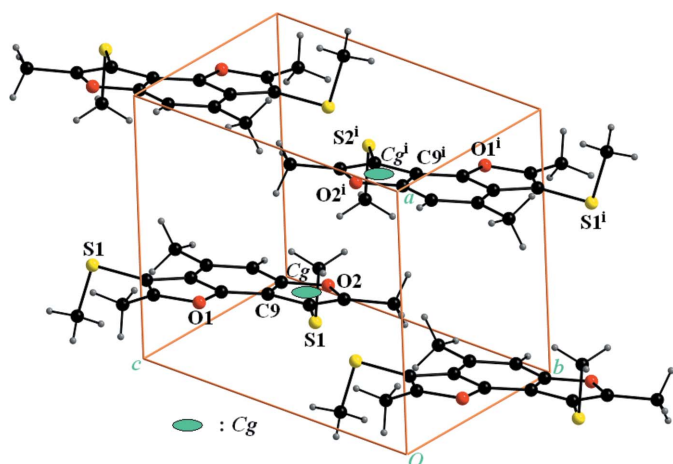


Figure 2
A packing diagram for (I) [symmetry code: (i) $1 - x, 1 - y, 1 - z$].

Experimental

The title compound was prepared according to a general method for benzo[1,2-*b*:4,3-*b'*]difuran derivatives (Choi *et al.*, 2006). Zinc chloride (1.50 g, 11.0 mmol) was added at room temperature to a stirred solution of 5-methylresorcinol (623 mg, 5.0 mmol) and α -chloro- α -(methylsulfanyl)acetone (1.53 g, 11.0 mmol) in dichloromethane (30 ml) and tetrahydrofuran (5 ml), and the mixture was stirred for 40 min. The reaction was quenched with water and the organic layer separated, dried over magnesium sulfate, filtered and concentrated in vacuum. The residue was purified by column chromatography (CCl_4) to afford (I) as a white solid. Crystals suitable for X-ray analysis were grown by slow evaporation of a benzene solution [yield 24%, m.p. 375–377 K; $R_f = 0.66$ (CCl_4)].

Crystal data

$\text{C}_{15}\text{H}_{16}\text{O}_2\text{S}_2$
 $M_r = 292.40$
Triclinic, $P\bar{1}$
 $a = 7.7512$ (6) Å
 $b = 9.8886$ (8) Å
 $c = 10.6471$ (9) Å
 $\alpha = 72.280$ (8)°
 $\beta = 71.435$ (8)°
 $\gamma = 74.554$ (7)°

$V = 723.8$ (1) Å³
 $Z = 2$
 $D_x = 1.342$ Mg m⁻³
Mo $K\alpha$ radiation
 $\mu = 0.36$ mm⁻¹
 $T = 298$ (2) K
Block, colourless
 $0.32 \times 0.25 \times 0.23$ mm

Data collection

Stoe Stadi-4 four-circle diffractometer
 $\omega/2\theta$ scans
Absorption correction: none
3302 measured reflections
3302 independent reflections

2226 reflections with $I > 2\sigma(I)$
 $\theta_{\text{max}} = 27.5^\circ$
3 standard reflections
frequency: 60 min
intensity decay: 2.4%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.067$
 $wR(F^2) = 0.167$
 $S = 1.11$
3302 reflections
177 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0543P)^2 + 0.6399P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.25$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.26$ e Å⁻³

All H atoms were positioned geometrically and refined using a riding model, with C–H = 0.93 Å for aromatic and 0.96 Å for methyl H atoms, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for aromatic H atoms and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for methyl H atoms.

Data collection: *STADIA* (Stoe & Cie, 1996); cell refinement: *STADIA*; data reduction: *X-RED* (Stoe & Cie, 1996); 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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