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5,6-Methylenedioxy-3-methylsulfinyl-2-phenylbenzofuran

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The title compound, $C_{16}H_{12}O_4S$, was prepared by oxidation of 5,6-methylenedioxy-3-methylsulfanylbenzofuran using *m*-chloroperbenzoic acid. The 5,6-methylenedioxybenzofuran ring system is approximately planar and the crystal structure involves aromatic π–π stacking.

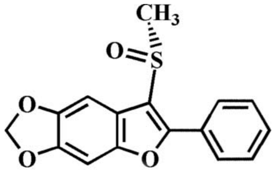
Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.003 Å
R factor = 0.049
wR factor = 0.118
Data-to-parameter ratio = 14.9

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

Comment

5,6-Methylenedioxybenzofuran ring systems are mostly found in natural products, and some of them are known to exhibit interesting physiological properties (Matsuura *et al.*, 1993; Shirataki *et al.*, 1999; Zhao *et al.*, 2003). With our ongoing interest in the synthesis and structure of 5,6-methylenedioxybenzofuran derivatives (Choi & Seo, 2000), we report here the crystal structure of the title compound, (I) (Fig. 1), which was obtained by oxidation of 5,6-methylenedioxy-3-sulfanylbenzofuran using *m*-chloroperbenzoic acid.



(I)

The bond lengths and angles in (I) are within normal ranges. The 5,6-methylenedioxybenzofuran unit is essentially planar, with a mean deviation of 0.032 Å from the least-squares plane defined by the 12 constituent atoms. The dihedral angle formed by the phenyl ring and the 5,6-methylenedioxybenzofuran group is 12.78 (5)° (Fig. 1).

In the crystal structure, aromatic π–π stacking interactions are observed between the molecules (Fig. 2), with $Cg1\cdots Cg2^{ii}$ = 3.721 (3) Å and $Cg2\cdots Cg2^{i}$ = 3.495 (3) Å [$Cg1$ and $Cg2$ are the centroids of the C2–C4/C6–C8 and C1/C2/C8/O3/C9 rings, respectively; symmetry codes: (i) $1 - x, -y, 1 - z$; (ii) $-x, -y, 1 - z$].

Experimental

m-Chloroperbenzoic acid (0.605 g, 2.70 mmol) was added in small portions to a stirred solution of 5,6-methylenedioxy-3-methylsulfanyl-1-benzofuran (0.710 g, 2.50 mmol) in dichloromethane (30 ml) at 273 K. After being stirred at room temperature for 1 h, the mixture was washed with saturated sodium bicarbonate solution and the organic layer separated, dried over magnesium sulfate, filtered and concentrated in vacuum. The residue was purified by column chromatography (hexane–EtOAc 1:1 *v*/*v*) to afford (I) as a white solid. Crystals suitable for X-ray analysis were grown by slow

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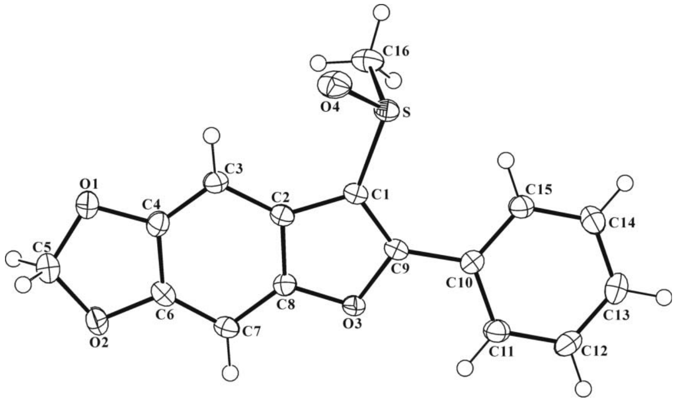

Figure 1
The molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level.

evaporation of an acetone solution [yield 85%, m.p. 481–482 K; R_f = 0.43 (hexane–EtOAc 1:1 *v*/*v*)].

Crystal data

$C_{16}H_{12}O_4S$
$M_r = 300.32$
Monoclinic, $P2_1/n$
$a = 7.4000$ (5) Å
$b = 15.777$ (1) Å
$c = 11.3155$ (8) Å
$\beta = 101.196$ (1)°
$V = 1295.94$ (15) Å^3
$Z = 4$
$D_x = 1.539$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.26$ mm^{-1}
$T = 173$ (2) K
Plate, colourless
0.51 × 0.32 × 0.09 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
7774 measured reflections
2833 independent reflections
2648 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.035$
$\theta_{\max} = 27.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.118$
$S = 1.10$
2833 reflections
190 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0445P)^2 + 1.6446P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 1.14$ e Å^{-3}
$\Delta\rho_{\min} = -0.47$ e Å^{-3}

All H atoms were geometrically located in ideal positions and refined using a riding model, with C—H = 0.93 Å for aromatic H atoms, 0.97 Å for the methylene group or 0.96 Å for the methyl group, and with $U_{\rm iso}$(H) = $1.2U_{\rm eq}$(C) for aromatic or methylene H atoms, or $1.5U_{\rm eq}$(C) for methyl H atoms. The highest peak in the residual electron density is located between S and C16, 0.99 and 0.92 Å from the two atoms, respectively.

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*.

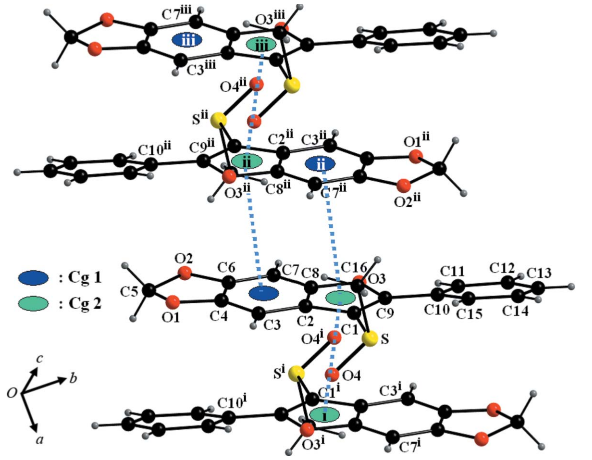

Figure 2
The π–π stacking interactions in (I) (dashed lines). *Cg*1 and *Cg*2 denote the centroids of each ring. [Symmetry codes: (i) $1 - x, -y, 1 - z$; (ii) $-x, -y, 1 - z$; (iii) $x - 1, y, z$].

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