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Methyl 2-(5-methyl-3-methylsulfinyl-1-benzofuran-2-yl)acetate

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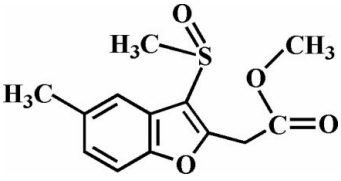
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.046; wR factor = 0.124; data-to-parameter ratio = 13.5.

The title compound, $C_{13}H_{14}O_4S$, was prepared by oxidation of methyl 2-(5-methyl-3-methylsulfanyl-1-benzofuran-2-yl)acetate with 3-chloroperoxybenzoic acid. The O atom and methyl group of the methylsulfinyl substituent lie on opposite sides of the plane of the benzofuran system. The crystal structure is stabilized by intermolecular aromatic π–π interactions between the benzene rings of neighbouring molecules, with a centroid–centroid separation of 3.841 (3) Å.

Related literature

For the crystal structures of similar ethyl 2-(3-methylsulfinyl-1-benzofuran-2-yl)acetate derivatives, see: Choi *et al.* (2007*a*,*b*).



Experimental

Crystal data

$C_{13}H_{14}O_4S$
M_r = 266.30
Triclinic, $P\overline{1}$
a = 7.9331 (6) Å
b = 8.1097 (6) Å
c = 10.7017 (8) Å
α = 71.601 (1)°
β = 81.107 (1)°
γ = 84.303 (1)°
V = 644.51 (8) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.26 mm^{-1}
T = 298 (2) K
0.40 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: none
3414 measured reflections
2237 independent reflections
1788 reflections with $I > 2\sigma(I)$
R_{int} = 0.052

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.123
S = 1.03
2237 reflections
166 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *DIAMOND* (Brandenburg, 1998); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BH2186).

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Choi, H. D., Seo, P. J., Son, B. W. & Lee, U. (2007*b*). *Acta Cryst.* E**63**, o3839.
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supporting information

Acta Cryst. (2008). E**64**, o1711 [doi:10.1107/S1600536808024689]

Methyl 2-(5-methyl-3-methylsulfinyl-1-benzofuran-2-yl)acetate

Hong Dae Choi, Pil Ja Seo, Byeng Wha Son and Uk Lee

S1. Comment

This work is related to our previous communications on the synthesis and structures of ethyl 2-(3-methylsulfinyl-1-benzofuran-2-yl)acetate analogues, *viz*. ethyl 2-(5-chloro-3-methylsulfinyl-1-benzofuran-2-yl)acetate (Choi *et al.*, 2007*a*) and ethyl 2-(5-methyl-3-methylsulfinyl-1-benzofuran-2-yl)acetate (Choi *et al.*, 2007*b*). Here we report the crystal structure of the title compound, methyl 2-(5-methyl-3-methylsulfinyl-1-benzofuran-2-yl)acetate (Fig. 1).

The benzofuran unit is essentially planar, with a mean deviation of 0.009 (2) Å from the least-squares plane defined by the nine constituent atoms. The packing structure is stabilized by aromatic π–π stacking interactions between adjacent benzene units, with a $Cg\cdots Cg^{i}$ distance is 3.841 (3) Å (Fig. 2).

S2. Experimental

77% 3-Chloroperoxybenzoic acid (359 mg, 1.6 mmol) was added in small portions to a stirred solution of methyl 2-(5-methyl-3-methylsulfanyl-1-benzofuran-2-yl)acetate (375 mg, 1.5 mmol) in dichloromethane (30 ml) at 273 K. After being stirred for 3 h at room temperature, the mixture was washed with saturated sodium bicarbonate solution and the organic layer was separated, dried over magnesium sulfate, filtered and concentrated under vacuum. The residue was purified by column chromatography (ethyl acetate) to afford the title compound as a colorless solid [yield 79%, m.p. 380–381 K; R_f = 0.58 (ethyl acetate)]. Single crystals suitable for X-ray diffraction were prepared by evaporation of a solution of the title compound in ethyl acetate at room temperature. Spectroscopic analysis: ^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.45 (s, 3H), 3.07 (s, 3H), 3.74 (s, 3H), 4.04 (s, 2H), 7.17 (dd, J = 8.44 Hz and J = 1.08 Hz, 1H), 7.38 (d, J = 8.40 Hz, 1H), 7.71 (s, 1H); EI–MS 266 [M^+].

S3. Refinement

All H atoms were geometrically positioned and refined using a riding model, with C—H = 0.93 Å for aromatic H atoms, 0.96 Å for methyl H atoms and 0.97 Å for methylene H atoms, respectively, and with U_{iso}(H) = 1.2Ueq(C) for aromatic and methylene H atoms and 1.5Ueq(C) for methyl H atoms.

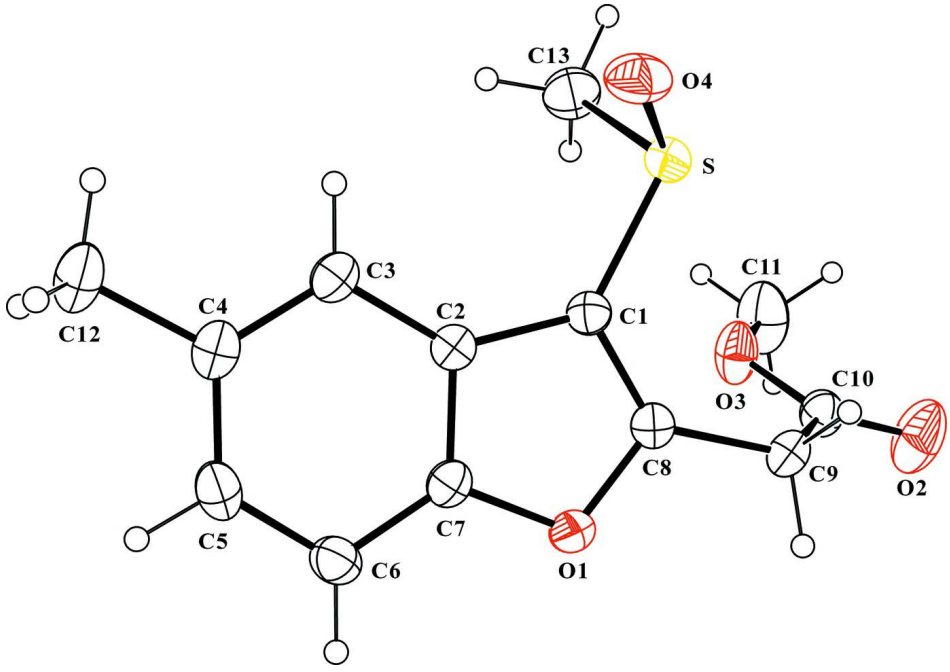

Figure 1

The molecular structure of the title compound, showing displacement ellipsoids drawn at the 30% probability level.

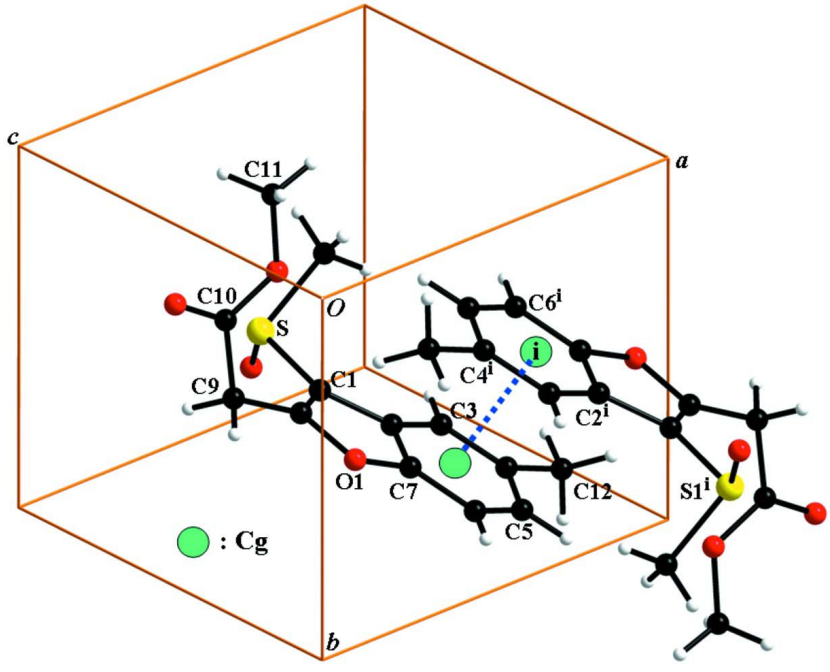

Figure 2

Intermolecular π–π interactions (dotted lines) in the title compound. *Cg* denotes ring centroid. Symmetry code: (i) 1-*x*, 2-*y*, -*z*.

Methyl 2-(5-methyl-3-methylsulfinyl-1-benzofuran-2-yl)acetate

Crystal data

$C_{13}H_{14}O_4S$
$M_r = 266.30$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 7.9331\ (6)$ Å
$b = 8.1097\ (6)$ Å
$c = 10.7017\ (8)$ Å
$\alpha = 71.601\ (1)°$
$\beta = 81.107\ (1)°$
$\gamma = 84.303\ (1)°$
$V = 644.51\ (8)$ Å^3
$Z = 2$
$F(000) = 280$
$D_x = 1.372$ Mg m^{-3}
Melting point = 380–381 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1817 reflections
$\theta = 2.6–27.3°$
$\mu = 0.26$ mm^{-1}
$T = 298$ K
Block, colorless
0.40 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 10.0 pixels mm^{-1}
φ and ω scans
3414 measured reflections
2237 independent reflections
1788 reflections with $I > 2\sigma(I)$
$R_{int} = 0.052$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.0°$
$h = -9\rightarrow9$
$k = -9\rightarrow9$
$l = -9\rightarrow12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.123$
$S = 1.04$
2237 reflections
166 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0647P)^2 + 0.1924P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.34$ e Å^{-3}
$\Delta\rho_{min} = -0.36$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
S	0.22227 (8)	0.36975 (8)	0.45695 (6)	0.0430 (2)
O1	0.16435 (19)	0.5432 (2)	0.07927 (16)	0.0392 (4)
O2	−0.1560 (3)	0.0853 (3)	0.3071 (3)	0.0850 (8)
O3	0.1251 (2)	0.0969 (2)	0.2923 (2)	0.0565 (5)
O4	0.2489 (3)	0.5059 (3)	0.51807 (19)	0.0597 (5)
C1	0.2418 (3)	0.4676 (3)	0.2838 (2)	0.0348 (5)
C2	0.3713 (3)	0.5757 (3)	0.1944 (2)	0.0342 (5)
C3	0.5246 (3)	0.6396 (3)	0.2059 (3)	0.0410 (6)
H3	0.5647	0.6111	0.2877	0.049*
C4	0.6143 (3)	0.7457 (3)	0.0930 (3)	0.0445 (6)
C5	0.5523 (3)	0.7870 (3)	−0.0291 (3)	0.0486 (7)
H5	0.6147	0.8592	−0.1038	0.058*
C6	0.4028 (3)	0.7256 (3)	−0.0442 (3)	0.0460 (6)

H6	0.3628	0.7534	−0.1260	0.055*
C7	0.3168 (3)	0.6199 (3)	0.0709 (2)	0.0366 (5)
C8	0.1227 (3)	0.4533 (3)	0.2103 (2)	0.0352 (5)
C9	−0.0416 (3)	0.3635 (3)	0.2452 (3)	0.0404 (6)
H9A	−0.1049	0.4071	0.1697	0.048*
H9B	−0.1081	0.3975	0.3181	0.048*
C10	−0.0307 (3)	0.1679 (3)	0.2841 (3)	0.0447 (6)
C11	0.1449 (4)	−0.0918 (4)	0.3298 (4)	0.0801 (11)
H11A	0.0761	−0.1409	0.4132	0.120*
H11B	0.2627	−0.1280	0.3382	0.120*
H11C	0.1090	−0.1312	0.2629	0.120*
C12	0.7804 (3)	0.8174 (4)	0.1000 (3)	0.0616 (8)
H12A	0.8119	0.7677	0.1878	0.092*
H12B	0.7661	0.9416	0.0797	0.092*
H12C	0.8685	0.7884	0.0369	0.092*
C13	0.4142 (4)	0.2333 (4)	0.4661 (3)	0.0573 (7)
H13A	0.5112	0.3042	0.4396	0.086*
H13B	0.4176	0.1635	0.4080	0.086*
H13C	0.4167	0.1588	0.5556	0.086*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S	0.0403 (4)	0.0487 (4)	0.0362 (4)	−0.0034 (3)	−0.0055 (3)	−0.0072 (3)
O1	0.0378 (9)	0.0428 (9)	0.0376 (9)	−0.0039 (7)	−0.0099 (7)	−0.0104 (7)
O2	0.0557 (13)	0.0589 (13)	0.135 (2)	−0.0209 (11)	−0.0173 (13)	−0.0150 (14)
O3	0.0415 (10)	0.0357 (9)	0.0832 (14)	−0.0039 (8)	0.0035 (9)	−0.0100 (9)
O4	0.0708 (13)	0.0711 (13)	0.0456 (11)	0.0066 (10)	−0.0141 (10)	−0.0297 (10)
C1	0.0363 (12)	0.0338 (12)	0.0349 (12)	−0.0017 (9)	−0.0056 (10)	−0.0110 (10)
C2	0.0354 (12)	0.0307 (11)	0.0377 (13)	0.0006 (9)	−0.0047 (10)	−0.0128 (10)
C3	0.0376 (13)	0.0413 (13)	0.0482 (15)	−0.0020 (10)	−0.0082 (11)	−0.0182 (12)
C4	0.0369 (13)	0.0363 (13)	0.0611 (17)	−0.0010 (10)	−0.0039 (12)	−0.0175 (12)
C5	0.0433 (14)	0.0415 (14)	0.0517 (16)	−0.0044 (11)	0.0054 (12)	−0.0060 (12)
C6	0.0492 (15)	0.0442 (14)	0.0397 (14)	−0.0006 (12)	−0.0058 (12)	−0.0065 (11)
C7	0.0337 (12)	0.0341 (12)	0.0435 (14)	0.0006 (9)	−0.0065 (10)	−0.0139 (10)
C8	0.0354 (12)	0.0317 (11)	0.0381 (13)	−0.0004 (9)	−0.0038 (10)	−0.0110 (10)
C9	0.0317 (12)	0.0448 (14)	0.0466 (15)	−0.0027 (10)	−0.0082 (11)	−0.0152 (11)
C10	0.0423 (14)	0.0478 (15)	0.0453 (15)	−0.0094 (11)	−0.0040 (11)	−0.0148 (12)
C11	0.070 (2)	0.0385 (16)	0.115 (3)	−0.0043 (15)	0.006 (2)	−0.0086 (17)
C12	0.0427 (15)	0.0532 (16)	0.088 (2)	−0.0115 (12)	−0.0069 (15)	−0.0182 (16)
C13	0.0571 (17)	0.0524 (16)	0.0596 (18)	0.0078 (13)	−0.0169 (14)	−0.0116 (14)

Geometric parameters (Å, º)

S—O4	1.495 (2)	C5—H5	0.9300
S—C1	1.759 (2)	C6—C7	1.381 (3)
S—C13	1.788 (3)	C6—H6	0.9300
O1—C8	1.366 (3)	C8—C9	1.495 (3)

O1—C7	1.392 (3)	C9—C10	1.505 (3)
O2—C10	1.204 (3)	C9—H9A	0.9700
O3—C10	1.315 (3)	C9—H9B	0.9700
O3—C11	1.453 (3)	C11—H11A	0.9600
C1—C8	1.354 (3)	C11—H11B	0.9600
C1—C2	1.444 (3)	C11—H11C	0.9600
C2—C7	1.382 (3)	C12—H12A	0.9600
C2—C3	1.405 (3)	C12—H12B	0.9600
C3—C4	1.381 (4)	C12—H12C	0.9600
C3—H3	0.9300	C13—H13A	0.9600
C4—C5	1.397 (4)	C13—H13B	0.9600
C4—C12	1.513 (3)	C13—H13C	0.9600
C5—C6	1.382 (4)		

O4—S—C1	107.63 (11)	O1—C8—C9	116.0 (2)
O4—S—C13	105.85 (13)	C8—C9—C10	117.4 (2)
C1—S—C13	98.82 (13)	C8—C9—H9A	108.0
C8—O1—C7	106.00 (17)	C10—C9—H9A	108.0
C10—O3—C11	117.3 (2)	C8—C9—H9B	108.0
C8—C1—C2	107.3 (2)	C10—C9—H9B	108.0
C8—C1—S	122.09 (18)	H9A—C9—H9B	107.2
C2—C1—S	130.59 (18)	O2—C10—O3	123.6 (2)
C7—C2—C3	119.0 (2)	O2—C10—C9	121.9 (2)
C7—C2—C1	104.91 (19)	O3—C10—C9	114.4 (2)
C3—C2—C1	136.1 (2)	O3—C11—H11A	109.5
C4—C3—C2	118.5 (2)	O3—C11—H11B	109.5
C4—C3—H3	120.8	H11A—C11—H11B	109.5
C2—C3—H3	120.8	O3—C11—H11C	109.5
C3—C4—C5	120.0 (2)	H11A—C11—H11C	109.5
C3—C4—C12	120.5 (3)	H11B—C11—H11C	109.5
C5—C4—C12	119.4 (2)	C4—C12—H12A	109.5
C6—C5—C4	123.0 (2)	C4—C12—H12B	109.5
C6—C5—H5	118.5	H12A—C12—H12B	109.5
C4—C5—H5	118.5	C4—C12—H12C	109.5
C7—C6—C5	115.3 (2)	H12A—C12—H12C	109.5
C7—C6—H6	122.4	H12B—C12—H12C	109.5
C5—C6—H6	122.4	S—C13—H13A	109.5
C6—C7—C2	124.2 (2)	S—C13—H13B	109.5
C6—C7—O1	125.2 (2)	H13A—C13—H13B	109.5
C2—C7—O1	110.58 (19)	S—C13—H13C	109.5
C1—C8—O1	111.21 (19)	H13A—C13—H13C	109.5
C1—C8—C9	132.8 (2)	H13B—C13—H13C	109.5

O4—S—C1—C8	−129.6 (2)	C1—C2—C7—C6	179.0 (2)
C13—S—C1—C8	120.6 (2)	C3—C2—C7—O1	179.36 (19)
O4—S—C1—C2	48.5 (2)	C1—C2—C7—O1	−1.1 (2)
C13—S—C1—C2	−61.4 (2)	C8—O1—C7—C6	−178.8 (2)
C8—C1—C2—C7	0.5 (2)	C8—O1—C7—C2	1.2 (2)

S—C1—C2—C7	−177.78 (18)	C2—C1—C8—O1	0.3 (3)
C8—C1—C2—C3	180.0 (3)	S—C1—C8—O1	178.70 (15)
S—C1—C2—C3	1.7 (4)	C2—C1—C8—C9	−178.3 (2)
C7—C2—C3—C4	0.5 (3)	S—C1—C8—C9	0.1 (4)
C1—C2—C3—C4	−178.9 (2)	C7—O1—C8—C1	−0.9 (2)
C2—C3—C4—C5	−0.1 (4)	C7—O1—C8—C9	177.97 (19)
C2—C3—C4—C12	−179.8 (2)	C1—C8—C9—C10	−72.1 (3)
C3—C4—C5—C6	−0.3 (4)	O1—C8—C9—C10	109.3 (2)
C12—C4—C5—C6	179.4 (2)	C11—O3—C10—O2	0.7 (4)
C4—C5—C6—C7	0.3 (4)	C11—O3—C10—C9	179.8 (3)
C5—C6—C7—C2	0.2 (4)	C8—C9—C10—O2	−176.1 (3)
C5—C6—C7—O1	−179.8 (2)	C8—C9—C10—O3	4.8 (3)
C3—C2—C7—C6	−0.6 (3)		