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2-(3-Methylsulfanyl-5-phenyl-1-benzofuran-2-yl)acetic acid

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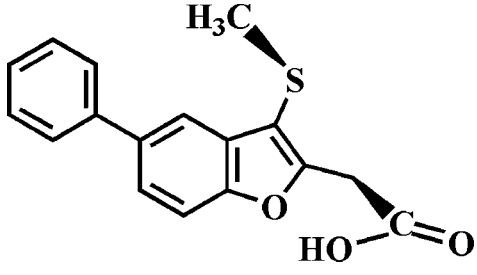
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.005 Å; R factor = 0.060; wR factor = 0.152; data-to-parameter ratio = 12.3.

The title compound, $C_{17}H_{14}O_3S$, was prepared by alkaline hydrolysis of ethyl 2-(3-methylsulfanyl-5-phenyl-1-benzofuran-2-yl)acetate. The phenyl ring is rotated out of the benzofuran plane with a dihedral angle of 44.1 (1)°. The methyl group of the methylsulfanyl substituent is almost perpendicular to the plane of the benzofuran fragment [100.4 (2)°] and is slightly tilted towards it. The crystal structure is stabilized by aromatic π–π interactions, with a centroid-to-centroid distance of 3.566 (5) Å between furan rings of neighboring molecules, and by inversion-related intermolecular O—H···O hydrogen bonds between the carboxyl groups.

Related literature

For the crystal structures of isomers of the title compound, see: Choi *et al.* (2006) and Seo *et al.* (2007).



Experimental

Crystal data

$C_{17}H_{14}O_3S$
M_r = 298.34
Monoclinic, $P2_1/c$
a = 10.857 (2) Å
b = 11.274 (2) Å
c = 11.978 (2) Å
α = 90.00°
β = 90.777 (4)°
γ = 90.00°
V = 1466.0 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 173 (2) K
0.40 × 0.10 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: none
5235 measured reflections
2390 independent reflections
1573 reflections with $I > 2\sigma(I)$
R_{int} = 0.076

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.152
S = 1.02
2390 reflections
195 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H1···O2^{i}	0.97 (6)	1.66 (6)	2.630 (4)	174 (6)

Symmetry code: (i) $-x, -y+1, -z+2$.

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*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CF2113).

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supplementary materials

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2-(3-Methylsulfanyl-5-phenyl-1-benzofuran-2-yl)acetic acid

H. D. Choi, P. J. Seo, B. W. Son and U. Lee

Comment

This work is related to our previous communications on the synthesis and structure of 2-(3-methylsulfanyl-1-benzofuran-2-yl)acetic acid analogues (Choi *et al.*, 2006; Seo *et al.*, 2007). Here we report the crystal structure of the title compound (Fig. 1).

The benzofuran unit is essentially planar, with a mean deviation of 0.012 Å from the least-squares plane defined by the nine constituent atoms. In the title compound, the dihedral angle formed by the plane of the benzofuran unit and the plane of the phenyl ring is 44.1 (1)°. The molecular packing (Fig. 2) is stabilized by π···π stacking interactions between furan rings of adjacent benzofuran systems, with a $Cg\cdots Cg^{i}$ distance of 3.566 (5) Å (*Cg* is the centroid of the O1/C8/C1/C2/C7 ring; symmetry code as in Fig. 2). Classical inversion-related O3—H1···$O2^{i}$ hydrogen bonds link the carboxyl groups of adjacent molecules (Table 1 and Fig. 2).

Experimental

Ethyl 2-(3-methylsulfanyl-5-phenyl-1-benzofuran-2-yl)acetate (652 mg, 2.0 mmol) was added to a solution of potassium hydroxide (561 mg, 10.0 mmol) in water (10 ml) and methanol (10 ml), and the mixture was heated at 333 K for 4hrs, then cooled. Water was added, and the solution was washed with dichloromethane. The aqueous layer was acidified to pH 1 with concentrated hydrochloric acid and then extracted with chloroform, 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 87%, m.p. 460–461 K; R_f = 0.64 (ethyl acetate)]. Single crystals suitable for X-ray diffraction were prepared by slow evaporation of a dilute solution of the title compound in acetone at room temperature.

Refinement

Atom H1 of the hydroxy group was found in a difference Fourier map and refined with O—H restrained to 0.9 (1) Å. The other H atoms were positioned geometrically and refined using a riding model, with C—H = 0.95 Å for aromatic H atoms, 0.98 Å for methyl H atoms and 0.99 Å for methylene H atoms, and with U_{iso}(H) = 1.2U_{eq}(C) for aromatic and methylene H atoms and U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms.

Figures

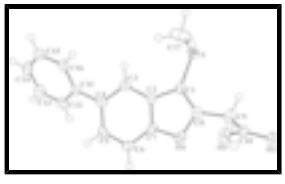

Fig. 1. The molecular structure of the title compound, showing displacement ellipsoides drawn at the 50% probability level.

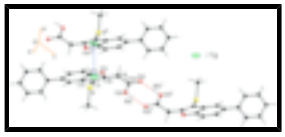

Fig. 2. π—π interactions (dotted lines) in the title compound. *Cg* denotes the ring centroid. [Symmetry code: (i) −*x*, 1 − *y*, 2 − *z*; (ii) −*x*, 1 − *y*, 1 − *z*.]

2-(3-Methylsulfanyl-5-phenyl-1-benzofuran-2-yl)acetic acid

Crystal data

$C_{17}H_{14}O_3S$	F_{000} = 624
M_r = 298.34	D_x = 1.352 Mg m^{-3}
Monoclinic, $P2_1/c$	Melting point: 460-461 K
Hall symbol: -p_2ybc	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 10.857 (2) Å	Cell parameters from 1299 reflections
b = 11.274 (2) Å	θ = 2.5–27.1º
c = 11.978 (2) Å	μ = 0.23 mm^{-1}
β = 90.777 (4)º	T = 173 (2) K
V = 1466.0 (4) Å^3	Block, colourless
Z = 4	0.40 × 0.10 × 0.10 mm

Data collection

Bruker SMART CCD area-detector diffractometer	2390 independent reflections
Radiation source: fine-focus sealed tube	1573 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.076
Detector resolution: 10.00 pixels mm^{-1}	θ_{max} = 25.0º
T = 173(2) K	θ_{min} = 2.5º
φ and ω scans	h = −12→12
Absorption correction: none	k = −11→13
5235 measured reflections	l = −14→14

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.060	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.152	$w = 1/[\sigma^2(F_o^2) + (0.0737P)^2 + 0.4676P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.03	$(\Delta/\sigma)_{max} < 0.001$
2390 reflections	$\Delta\rho_{max}$ = 0.41 e Å^{-3}
195 parameters	$\Delta\rho_{min}$ = −0.30 e Å^{-3}
1 restraint	Extinction correction: none

Primary atom site location: structure-invariant direct methods

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
S	0.14850 (10)	0.23359 (9)	0.57947 (9)	0.0309 (3)
O1	0.0811 (2)	0.5739 (2)	0.6300 (2)	0.0266 (7)
O2	−0.1048 (2)	0.4462 (2)	0.9100 (2)	0.0265 (7)
O3	0.0877 (2)	0.5047 (3)	0.8757 (2)	0.0297 (7)
H1	0.099 (6)	0.526 (6)	0.954 (5)	0.11 (2)*
C1	0.1424 (3)	0.3886 (3)	0.5843 (3)	0.0198 (9)
C2	0.2125 (3)	0.4721 (3)	0.5198 (3)	0.0189 (9)
C3	0.3023 (3)	0.4618 (3)	0.4376 (3)	0.0203 (9)
H3	0.3308	0.3859	0.4150	0.024*
C4	0.3495 (3)	0.5651 (3)	0.3894 (3)	0.0200 (9)
C5	0.3068 (3)	0.6753 (4)	0.4257 (3)	0.0232 (9)
H5	0.3402	0.7450	0.3933	0.028*
C6	0.2181 (3)	0.6875 (4)	0.5067 (3)	0.0250 (9)
H6	0.1906	0.7630	0.5313	0.030*
C7	0.1722 (3)	0.5836 (3)	0.5494 (3)	0.0205 (9)
C8	0.0665 (3)	0.4546 (4)	0.6483 (3)	0.0234 (9)
C9	−0.0320 (3)	0.4211 (4)	0.7269 (3)	0.0307 (11)
H9A	−0.0398	0.3336	0.7260	0.037*
H9B	−0.1107	0.4540	0.6979	0.037*
C10	−0.0165 (3)	0.4601 (3)	0.8469 (3)	0.0220 (9)
C11	0.4407 (3)	0.5580 (3)	0.2981 (3)	0.0198 (9)
C12	0.4281 (3)	0.6317 (4)	0.2039 (3)	0.0275 (10)
H12	0.3616	0.6863	0.1987	0.033*
C13	0.5120 (4)	0.6246 (4)	0.1194 (3)	0.0332 (11)
H13	0.5007	0.6726	0.0549	0.040*
C14	0.6120 (4)	0.5496 (4)	0.1259 (3)	0.0325 (11)
H14	0.6703	0.5469	0.0674	0.039*
C15	0.6258 (3)	0.4784 (4)	0.2191 (3)	0.0254 (10)
H15	0.6948	0.4268	0.2249	0.030*
C16	0.5411 (3)	0.4812 (3)	0.3036 (3)	0.0209 (9)
H16	0.5513	0.4303	0.3663	0.025*

C17	0.2897 (4)	0.2070 (4)	0.6565 (4)	0.0542 (15)
H17A	0.3571	0.2508	0.6218	0.081*
H17B	0.3084	0.1220	0.6559	0.081*
H17C	0.2803	0.2338	0.7338	0.081*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S	0.0362 (6)	0.0260 (6)	0.0304 (7)	−0.0089 (5)	−0.0004 (5)	−0.0024 (5)
O1	0.0237 (14)	0.0361 (18)	0.0201 (15)	0.0051 (12)	0.0003 (12)	−0.0009 (13)
O2	0.0220 (15)	0.0358 (17)	0.0219 (16)	−0.0052 (12)	0.0058 (12)	−0.0015 (13)
O3	0.0170 (14)	0.0507 (19)	0.0211 (17)	−0.0056 (13)	−0.0029 (12)	−0.0028 (14)
C1	0.0185 (19)	0.031 (2)	0.010 (2)	−0.0033 (17)	0.0002 (16)	0.0005 (18)
C2	0.0172 (19)	0.025 (2)	0.014 (2)	−0.0018 (16)	−0.0062 (16)	−0.0005 (17)
C3	0.022 (2)	0.021 (2)	0.018 (2)	0.0051 (16)	−0.0073 (17)	−0.0021 (17)
C4	0.0222 (19)	0.021 (2)	0.017 (2)	0.0012 (16)	−0.0099 (17)	−0.0011 (16)
C5	0.029 (2)	0.021 (2)	0.019 (2)	−0.0045 (17)	−0.0029 (18)	0.0068 (18)
C6	0.029 (2)	0.024 (2)	0.022 (2)	0.0079 (18)	−0.0023 (18)	−0.0013 (19)
C7	0.0145 (19)	0.030 (2)	0.017 (2)	0.0033 (16)	−0.0011 (16)	0.0004 (18)
C8	0.021 (2)	0.028 (2)	0.020 (2)	−0.0034 (17)	−0.0059 (18)	−0.0004 (18)
C9	0.024 (2)	0.043 (3)	0.025 (2)	−0.0077 (18)	−0.0028 (18)	−0.005 (2)
C10	0.020 (2)	0.023 (2)	0.023 (2)	0.0051 (16)	−0.0024 (18)	0.0014 (17)
C11	0.023 (2)	0.024 (2)	0.012 (2)	−0.0050 (16)	−0.0048 (17)	0.0003 (17)
C12	0.024 (2)	0.034 (2)	0.025 (2)	0.0004 (18)	−0.0076 (19)	0.0031 (19)
C13	0.032 (2)	0.048 (3)	0.019 (2)	−0.005 (2)	−0.0030 (19)	0.008 (2)
C14	0.026 (2)	0.050 (3)	0.022 (2)	−0.004 (2)	0.0050 (19)	−0.002 (2)
C15	0.025 (2)	0.030 (2)	0.021 (2)	0.0002 (17)	−0.0028 (18)	−0.0068 (18)
C16	0.020 (2)	0.030 (2)	0.012 (2)	−0.0023 (17)	−0.0011 (16)	0.0006 (17)
C17	0.069 (3)	0.035 (3)	0.058 (3)	0.012 (2)	−0.030 (3)	0.006 (2)

Geometric parameters (Å, °)

S—C1	1.749 (4)	C8—C9	1.483 (5)
S—C17	1.804 (4)	C9—C10	1.511 (5)
O1—C8	1.372 (5)	C9—H9A	0.990
O1—C7	1.397 (4)	C9—H9B	0.990
O2—C10	1.238 (4)	C11—C16	1.393 (5)
O3—C10	1.281 (4)	C11—C12	1.407 (5)
O3—H1	0.97 (6)	C12—C13	1.372 (5)
C1—C8	1.356 (5)	C12—H12	0.950
C1—C2	1.441 (5)	C13—C14	1.378 (6)
C2—C7	1.379 (5)	C13—H13	0.950
C2—C3	1.399 (5)	C14—C15	1.381 (6)
C3—C4	1.401 (5)	C14—H14	0.950
C3—H3	0.950	C15—C16	1.378 (5)
C4—C5	1.397 (5)	C15—H15	0.950
C4—C11	1.487 (5)	C16—H16	0.950
C5—C6	1.384 (5)	C17—H17A	0.980
C5—H5	0.950	C17—H17B	0.980

C6—C7	1.374 (5)	C17—H17C	0.980
C6—H6	0.950		
C1—S—C17	100.4 (2)	C8—C9—H9B	108.0
C8—O1—C7	105.8 (3)	C10—C9—H9B	108.0
C10—O3—H1	117 (4)	H9A—C9—H9B	107.2
C8—C1—C2	105.9 (4)	O2—C10—O3	125.0 (3)
C8—C1—S	126.2 (3)	O2—C10—C9	117.8 (3)
C2—C1—S	127.9 (3)	O3—C10—C9	117.2 (4)
C7—C2—C3	118.9 (3)	C16—C11—C12	118.2 (4)
C7—C2—C1	106.6 (3)	C16—C11—C4	121.8 (3)
C3—C2—C1	134.5 (4)	C12—C11—C4	120.0 (3)
C4—C3—C2	118.9 (3)	C13—C12—C11	119.9 (4)
C4—C3—H3	120.5	C13—C12—H12	120.0
C2—C3—H3	120.5	C11—C12—H12	120.0
C5—C4—C3	119.1 (4)	C12—C13—C14	121.7 (4)
C5—C4—C11	120.2 (3)	C12—C13—H13	119.2
C3—C4—C11	120.6 (3)	C14—C13—H13	119.2
C6—C5—C4	122.9 (4)	C13—C14—C15	118.6 (4)
C6—C5—H5	118.5	C13—C14—H14	120.7
C4—C5—H5	118.5	C15—C14—H14	120.7
C7—C6—C5	115.8 (4)	C16—C15—C14	121.0 (4)
C7—C6—H6	122.1	C16—C15—H15	119.5
C5—C6—H6	122.1	C14—C15—H15	119.5
C6—C7—C2	124.3 (4)	C15—C16—C11	120.6 (4)
C6—C7—O1	126.0 (3)	C15—C16—H16	119.7
C2—C7—O1	109.6 (3)	C11—C16—H16	119.7
C1—C8—O1	112.0 (3)	S—C17—H17A	109.5
C1—C8—C9	132.0 (4)	S—C17—H17B	109.5
O1—C8—C9	115.9 (3)	H17A—C17—H17B	109.5
C8—C9—C10	117.3 (3)	S—C17—H17C	109.5
C8—C9—H9A	108.0	H17A—C17—H17C	109.5
C10—C9—H9A	108.0	H17B—C17—H17C	109.5
C17—S—C1—C8	−105.6 (4)	S—C1—C8—O1	−177.8 (2)
C17—S—C1—C2	76.6 (4)	C2—C1—C8—C9	176.0 (3)
C8—C1—C2—C7	−0.6 (4)	S—C1—C8—C9	−2.1 (6)
S—C1—C2—C7	177.6 (3)	C7—O1—C8—C1	−0.1 (4)
C8—C1—C2—C3	−178.1 (3)	C7—O1—C8—C9	−176.5 (3)
S—C1—C2—C3	0.0 (6)	C1—C8—C9—C10	120.0 (4)
C7—C2—C3—C4	0.9 (4)	O1—C8—C9—C10	−64.4 (4)
C1—C2—C3—C4	178.2 (3)	C8—C9—C10—O2	171.0 (3)
C2—C3—C4—C5	0.8 (5)	C8—C9—C10—O3	−9.4 (5)
C2—C3—C4—C11	−177.1 (3)	C5—C4—C11—C16	136.8 (4)
C3—C4—C5—C6	−0.9 (5)	C3—C4—C11—C16	−45.3 (5)
C11—C4—C5—C6	177.1 (3)	C5—C4—C11—C12	−42.0 (5)
C4—C5—C6—C7	−0.8 (5)	C3—C4—C11—C12	135.9 (4)
C5—C6—C7—C2	2.7 (5)	C16—C11—C12—C13	1.5 (5)
C5—C6—C7—O1	−178.8 (3)	C4—C11—C12—C13	−179.7 (3)
C3—C2—C7—C6	−2.8 (5)	C11—C12—C13—C14	−2.4 (6)

C1—C2—C7—C6	179.2 (3)	C12—C13—C14—C15	1.4 (6)
C3—C2—C7—O1	178.5 (3)	C13—C14—C15—C16	0.5 (6)
C1—C2—C7—O1	0.5 (4)	C14—C15—C16—C11	−1.4 (5)
C8—O1—C7—C6	−178.9 (3)	C12—C11—C16—C15	0.3 (5)
C8—O1—C7—C2	−0.3 (3)	C4—C11—C16—C15	−178.5 (3)
C2—C1—C8—O1	0.4 (4)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H1···O2^{i}	0.97 (6)	1.66 (6)	2.630 (4)	174 (6)

Symmetry codes: (i) −*x*, −*y*+1, −*z*+2.

Fig. 1

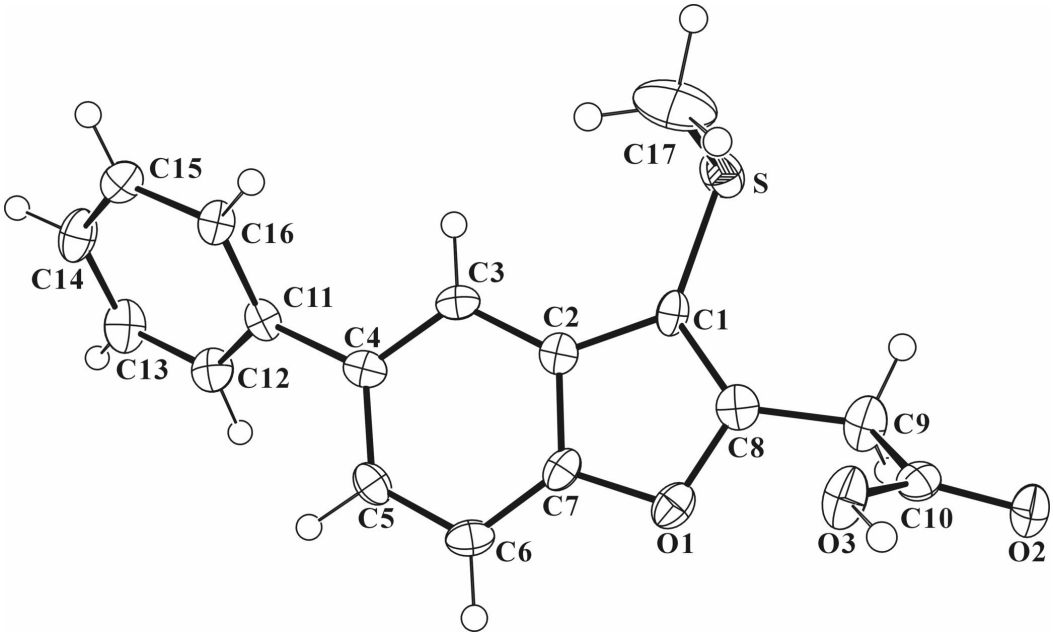

Fig. 2