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2-(5-Ethyl-3-methylsulfanyl-1-benzofuran-2-yl)-acetic acid

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.002 Å
R factor = 0.035
wR factor = 0.096
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.

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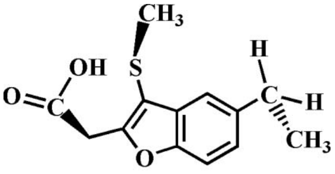
The title compound, $C_{13}H_{14}O_3S$, was prepared by alkaline hydrolysis of ethyl 2-(5-ethyl-3-methylsulfanyl-1-benzofuran-2-yl)acetate. The crystal structure is stabilized by aromatic π–π stacking interactions and inversion-related intermolecular O—H···O hydrogen bonds between adjacent carboxyl groups.

Comment

This work is related to our previous communications on (3-methylsulfanyl-1-benzofuran-5-yl)acetic acid derivatives (Choi *et al.*, 2006, 2007), with the purpose of understanding how the molecular conformation and crystal structure are affected by acetic acid substituents on the 1-benzofuran ring system. The title compound, (I) (Fig. 1), was obtained by hydrolysis of ethyl 2-(5-ethyl-3-methylsulfanyl-1-benzofuran-2-yl)acetate with potassium hydroxide.



(I)

In (I), bond lengths and angles are as expected for this type of compound (Choi *et al.*, 2006, 2007). In the crystal structure (Fig. 2), π–π stacking interactions are observed between the furan and benzene rings of adjacent benzofuran systems, with $Cg1\cdots Cg2^{i}$ = 3.801 (2) Å ($Cg1$ and $Cg2$ are the centroids of the O1/C8/C1/C2/C7 and C2–C7 rings; symmetry code as in Fig. 2). Classical inversion-related O2—H14···O3^{iii} hydrogen bonds link the carboxyl groups of adjacent molecules (Table 1 and Fig. 2).

Experimental

Ethyl 2-(5-ethyl-3-methylsulfanyl-1-benzofuran-2-yl)acetate (834 mg, 3 mmol) was added to a solution of potassium hydroxide (561 mg, 10.0 mmol) in water (10 ml) and methanol (10 ml); the mixture was heated at 333 K for 4 h and then cooled. Water (20 ml) was added, and the solution was washed with dichloromethane. The aqueous layer was acidified to pH = 1.0 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 (hexane–ethyl acetate, 1:2 v/v) to give (I) as a white solid. Crystals suitable for X-ray analysis were grown by slow evaporation of a benzene solution [yield 92%, m.p. 396–397 K; R_F = 0.46 (hexane–ethyl acetate, 1:2 v/v)].

Crystal data

$C_{13}H_{14}O_3S$
$M_r = 250.30$
Triclinic, $P\overline{1}$
$a = 5.0746$ (4) Å
$b = 8.0636$ (6) Å
$c = 15.058$ (1) Å
$\alpha = 94.711$ (1)°
$\beta = 93.153$ (1)°
$\gamma = 90.538$ (1)°
$V = 613.10$ (8) Å^3
$Z = 2$
Mo $K\alpha$ radiation
$\mu = 0.26$ mm^{-1}
$T = 173$ (2) K
0.32 × 0.25 × 0.20 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: none
3461 measured reflections
2368 independent reflections
2145 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.031$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.096$
$S = 1.10$
2368 reflections
159 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\rm max} = 0.28$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.22$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H14···O3$^{\rm i}$	0.82 (3)	1.87 (3)	2.684 (2)	175 (3)

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

Atom H14 of the carboxyl group was found in a difference Fourier map and refined freely with an isotropic displacement parameter. 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 $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$ for aromatic and methylene H atoms and $1.5U_{\rm eq}({\rm C})$ for methyl H atoms.

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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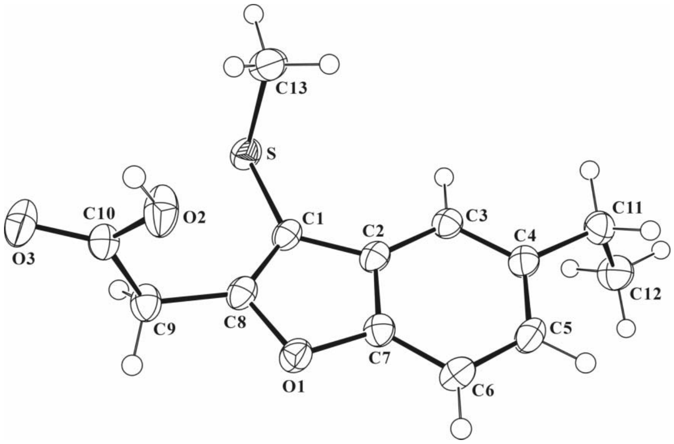

Figure 1
The molecular structure of (I), with displacement ellipsoids drawn at the 50% probability level for non-H atoms.

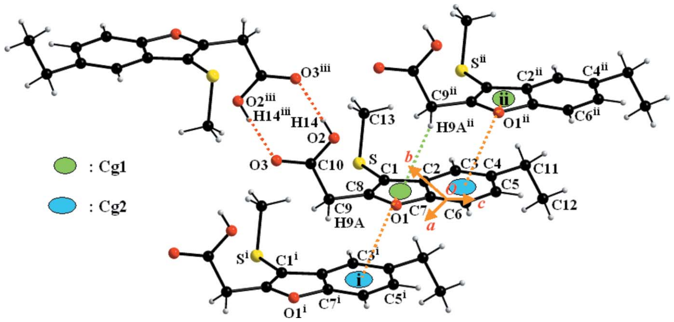

Figure 2
Hydrogen-bond and π–π interactions (dotted lines) in (I). [Symmetry codes: (i) $1 + x, y, z$; (ii) $x - 1, y, z$; (iii) $2 - x, 2 - y, 1 - z$.]

References

Brandenburg, K. (1998). *DIAMOND*. Version 2.1. Crystal Impact GbR, Bonn, Germany.

Bruker (1997). *SMART* (Version 5.631) and *SAINT* (Version 6.12). Versions 5.0. Bruker AXS Inc., Madison, Wisconsin, USA.

Choi, H. D., Seo, P. J., Kang, B. W., Son, B. W. & Lee, U. (2006). *Acta Cryst.* E**62**, o4800–o4801.

Choi, H. D., Seo, P. J., Son, B. W. & Lee, U. (2007). *Acta Cryst.* E**63**, o442–o443.

Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

Acta Cryst. (2007). E**63**, o2048–o2049 Seo *et al.* • $C_{13}H_{14}O_3S$ **o2049**