Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# 2-(4-Fluorophenyl)-3-isopropylsulfinyl-5,6-methylenedioxy-1-benzofuran

**Pil Ja Seo,[a] Hong Dae Choi,[a] Byeng Wha Son[b] and Uk Lee[b]***

[a]Department of Chemistry, Dongeui University, San 24 Kaya-dong Busanjin-gu, Busan 614-714, Republic of Korea, and [b]Department of Chemistry, Pukyong National University, 599-1 Daeyeon 3-dong, Nam-gu, Busan 608-737, Republic of Korea
Correspondence e-mail: uklee@pknu.ac.kr

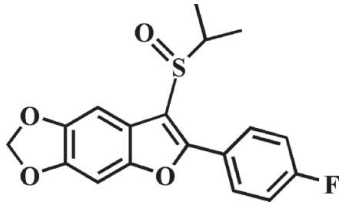
Received 21 November 2011; accepted 28 November 2011

Key indicators: single-crystal X-ray study; $T$ = 173 K; mean $\sigma$(C–C) = 0.002 Å; disorder in main residue; $R$ factor = 0.039; $wR$ factor = 0.101; data-to-parameter ratio = 15.2.

In the title compound, $C_{18}H_{15}FO_4S$, the fluorobenzene ring makes a dihedral angle of 4.3 (1)° with the mean plane of the benzofuran fragment. In the crystal, molecules are linked by weak intermolecular C—H···O hydrogen bonds. The O atom of the sulfinyl group is disordered over two orientations, with site-occupancy factors of 0.940 (3) and 0.060 (3).

## Related literature

For the pharmacological activity of benzofuran compounds, see: Aslam *et al.* (2009); Galal *et al.* (2009); Khan *et al.* (2005). For natural products with benzofuran rings, see: Akgul & Anil (2003); Soekamto *et al.* (2003). For the crystal structures of related compounds, see: Choi *et al.* (2010*a*,*b*).



## Experimental

### Crystal data

$C_{18}H_{15}FO_4S$
$M_r$ = 346.36
Triclinic, $P\overline{1}$
$a$ = 6.2519 (1) Å
$b$ = 9.6773 (2) Å
$c$ = 12.9267 (2) Å
$\alpha$ = 90.122 (1)°
$\beta$ = 94.726 (1)°
$\gamma$ = 101.920 (1)°
$V$ = 762.47 (2) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.24 mm$^{-1}$
$T$ = 173 K
0.45 × 0.21 × 0.14 mm

### Data collection

Bruker SMART APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
$T_{min}$ = 0.898, $T_{max}$ = 0.967
13552 measured reflections
3488 independent reflections
3171 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.024

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.101
$S$ = 1.06
3488 reflections
229 parameters
4 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.56 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.40 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C5—H5$B$···O4$A^{i}$ | 0.99 | 2.27 | 3.231 (2) | 163 |
| C18—H18$A$···O4$A^{ii}$ | 0.98 | 2.49 | 3.354 (2) | 147 |

Symmetry codes: (i) $-x+1, -y, -z$; (ii) $-x+1, -y+1, -z$.

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); 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: FJ2487).

## References

Akgul, Y. Y. & Anil, H. (2003). *Phytochemistry*, **63**, 939–943.
Aslam, S. N., Stevenson, P. C., Kokubun, T. & Hall, D. R. (2009). *Microbiol. Res.* **164**, 191–195.
Brandenburg, K. (1998). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Bruker (2009). *APEX2*, *SADABS* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Choi, H. D., Seo, P. J., Son, B. W. & Lee, U. (2010*a*). *Acta Cryst.* E**66**, o605.
Choi, H. D., Seo, P. J., Son, B. W. & Lee, U. (2010*b*). *Acta Cryst.* E**66**, o1296.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Galal, S. A., Abd El-All, A. S., Abdallah, M. M. & El-Diwani, H. I. (2009). *Bioorg. Med. Chem. Lett.* **19**, 2420–2428.
Khan, M. W., Alam, M. J., Rashid, M. A. & Chowdhury, R. (2005). *Bioorg. Med. Chem.* **13**, 4796–4805.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Soekamto, N. H., Achmad, S. A., Ghisalberti, E. L., Hakim, E. H. & Syah, Y. M. (2003). *Phytochemistry*, **64**, 831–834.

# supporting information

*Acta Cryst.* (2011). E**67**, o3504 [https://doi.org/10.1107/S1600536811051178]

## 2-(4-Fluorophenyl)-3-isopropylsulfinyl-5,6-methylenedioxy-1-benzofuran

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

### S1. Comment

Recently, substituted benzofuran derivatives have drawn much attention due to their valuable pharmacological properties such as antibacterial and antifungal, antitumor and antiviral, and antimicrobial activities (Aslam *et al.*, 2009, Galal *et al.*, 2009, Khan *et al.*, 2005). These benzofuran derivatives occur in a wide range of natural products (Akgul & Anil, 2003; Soekamto *et al.*, 2003). As a part of our ongoing study of 5,6-(methylenedioxy)benzofuran derivatives containing either 3-methylsulfinyl (Choi *et al.*, 2010*a*) or 3-ethylsulfinyl (Choi *et al.*, 2010*b*) substituents, we report herein the crystal structure of the title compound.

In the title molecule (Fig. 1), the benzofuran unit is essentially planar, with a mean deviation of 0.006 (1) Å from the least-squares plane defined by the nine constituent atoms. The dihedral angle formed by the 4-fluorophenyl ring and the mean plane of the benzofuran fragment is 4.3 (1)°. The O atom of the sulfinyl group is disordered over two positions with site-occupancy factors, from refinement, of 0.940 (3) (Part A) and 0.060 (3) (part B). The crystal packing is stabilized by weak intermolecular C—H···O hydrogen bonds; the first one between a methylene H atom and the O atom of the S═O unit (Table 1, first entry & Fig. 2), and the second one between a methyl H atom of the isopropyl group and the O atom of the S═O unit (Table 1, second entry & Fig. 2).

### S2. Experimental

77% 3-chloroperoxybenzoic acid (224 mg, 1.0 mmol) was added in small portions to a stirred solution of 2-(4-fluorophenyl)-3-isopropylsulfanyl-5,6-methylenedioxy-1-benzofuran (251 mg, 0.8 mmol) in dichloromethane (30 mL) at 273 K. After being stirred at room temperature for 3h, the mixture was washed with saturated sodium bicarbonate solution and the organic layer was separated, dried over magnesium sulfate, filtered and concentrated at reduced pressure. The residue was purified by column chromatography (hexane–ethyl acetate, 1:2 v/v) to afford the title compound as a colorless solid [yield 71%, m.p. 437–438 K; $R_f$ = 0.55 (hexane–ethyl acetate, 1:2 v/v)]. Single crystals suitable for X-ray diffraction were prepared by slow evaporation of a solution of the title compound in ethyl acetate at room temperature.

### S3. Refinement

All H atoms were positioned geometrically and refined using a riding model, with C—H = 0.95 Å for the aryl, 1.00 Å for the methine, 0.99 Å for the methylene, and 0.98 Å for the methyl H atoms. $U_{iso}$(H) =1.2$U_{eq}$(C) for the aryl, methine, and methylene H atoms, and 1.5$U_{eq}$(C) for the methyl H atoms. The O atom of sulfinyl group is disordered over two positions with site-ccupancy factors, from refinement of 0.940 (3) (part A) and 0.060 (3) (part B). The distance of S—O sets was restrained to 0.001 Å using command SADI and DELU.

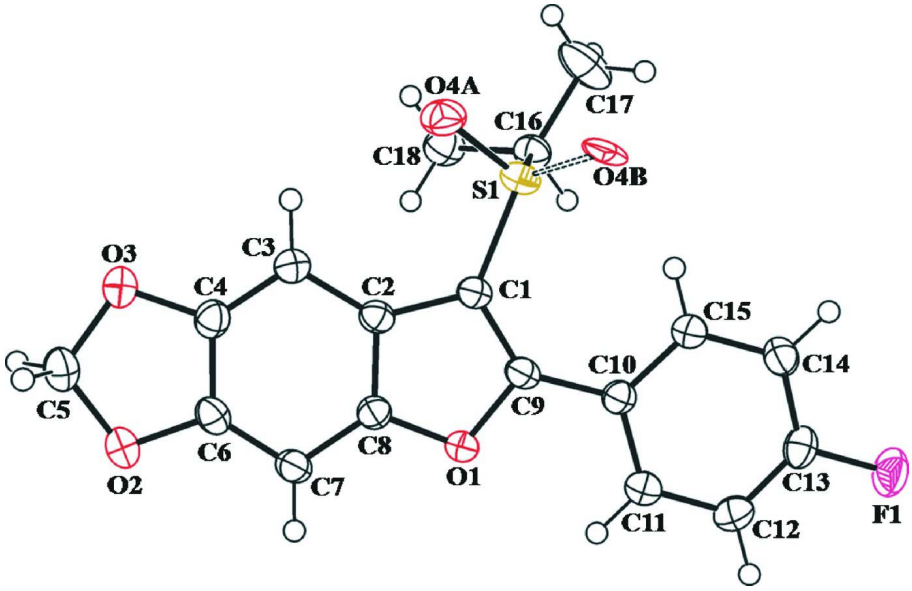



**Figure 1**

The molecular structure of the title compound with the atom numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are presented as small spheres of arbitrary radius. The O atom of the sulfinyl group is disordered over two positions with site-occupancy factors, from refinement of 0.940 (3) (Part A) and 0.060 (3) (part B).

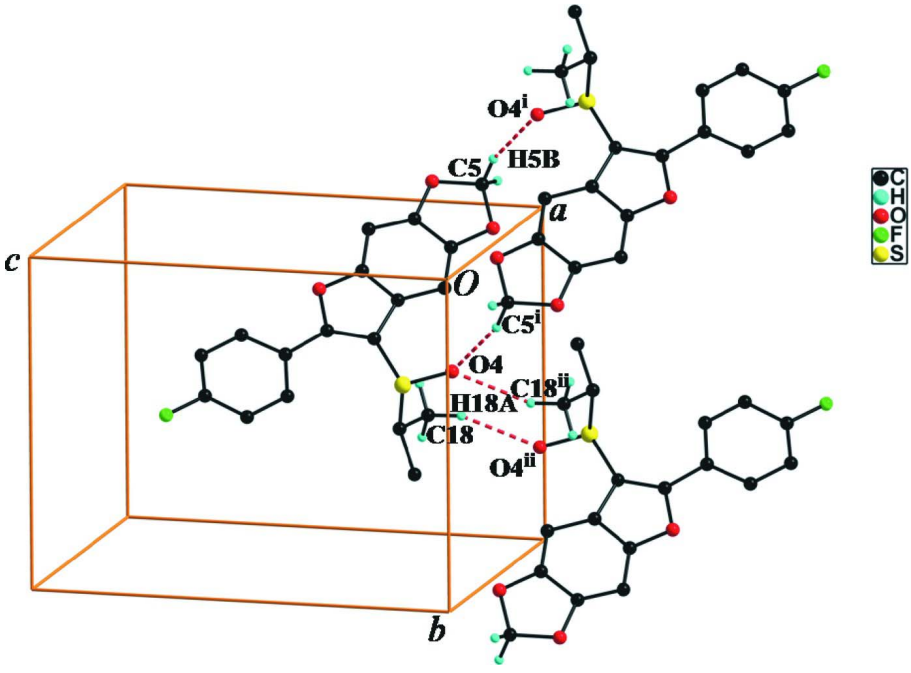



**Figure 2**

A view of the C—H···O interactions (dotted lines) in the crystal structure of the title compound. H atoms non-participating in hydrogen- bonding were omitted for clarity. [Symmetry codes: (i) - *x* + 1, - *y*, - *z*; (ii) - *x* + 1, 1- *y*, - *z*.]

**11-(4-fluorophenyl)-12-(propane-2-sulfinyl)-4,6,10- trioxatricyclo[7.3.0.0$^{3,7}$]dodeca-1(9),2,7-triene**

*Crystal data*

$C_{18}H_{15}FO_4S$
$M_r = 346.36$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 6.2519$ (1) Å
$b = 9.6773$ (2) Å
$c = 12.9267$ (2) Å
$\alpha = 90.122$ (1)°
$\beta = 94.726$ (1)°
$\gamma = 101.920$ (1)°
$V = 762.47$ (2) Å$^3$
$Z = 2$
$F(000) = 360$
$D_x = 1.509$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 7633 reflections
$\theta = 2.6$–$27.5°$
$\mu = 0.24$ mm$^{-1}$
$T = 173$ K
Block, colourless
0.45 × 0.21 × 0.14 mm

*Data collection*

Bruker SMART APEXII CCD diffractometer
Radiation source: rotating anode
Graphite multilayer monochromator
Detector resolution: 10.0 pixels mm$^{-1}$
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
$T_{min} = 0.898$, $T_{max} = 0.967$
13552 measured reflections
3488 independent reflections
3171 reflections with $I > 2\sigma(I)$
$R_{int} = 0.024$
$\theta_{max} = 27.5°$, $\theta_{min} = 1.6°$
$h = -7 \rightarrow 8$
$k = -12 \rightarrow 12$
$l = -15 \rightarrow 16$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.101$
$S = 1.06$
3488 reflections
229 parameters
4 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: difference Fourier map
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0457P)^2 + 0.4198P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.56$ e Å$^{-3}$
$\Delta\rho_{min} = -0.40$ e Å$^{-3}$

*Special details*

**Geometry**. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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$^2$ > 2sigma(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* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| S1 | 0.15386 (6) | 0.36031 (4) | 0.14314 (3) | 0.02733 (12) | |
| F1 | −0.43070 (18) | 0.35212 (13) | 0.57633 (8) | 0.0438 (3) | |
| O1 | 0.31016 (17) | 0.12210 (11) | 0.37742 (8) | 0.0264 (2) | |

| | | | | | |
|---|---|---|---|---|---|
| O2 | 0.8622 (2) | −0.09128 (13) | 0.23234 (10) | 0.0377 (3) | |
| O3 | 0.8308 (2) | 0.03336 (14) | 0.08091 (10) | 0.0415 (3) | |
| O4A | 0.2282 (2) | 0.32964 (14) | 0.04204 (9) | 0.0358 (4) | 0.940 (3) |
| O4B | −0.0806 (7) | 0.3600 (18) | 0.1198 (15) | 0.030 (4) | 0.060 (3) |
| C1 | 0.2601 (2) | 0.25039 (15) | 0.23544 (11) | 0.0241 (3) | |
| C2 | 0.4209 (2) | 0.16817 (15) | 0.21565 (12) | 0.0245 (3) | |
| C3 | 0.5457 (3) | 0.15352 (16) | 0.13162 (12) | 0.0282 (3) | |
| H3 | 0.5335 | 0.2030 | 0.0687 | 0.034* | |
| C4 | 0.6853 (3) | 0.06270 (17) | 0.14767 (13) | 0.0289 (3) | |
| C5 | 0.9256 (3) | −0.07578 (18) | 0.12790 (14) | 0.0343 (4) | |
| H5A | 1.0874 | −0.0506 | 0.1285 | 0.041* | |
| H5B | 0.8725 | −0.1656 | 0.0882 | 0.041* | |
| C6 | 0.7051 (3) | −0.01235 (16) | 0.23908 (13) | 0.0279 (3) | |
| C7 | 0.5854 (3) | −0.00188 (16) | 0.32195 (12) | 0.0278 (3) | |
| H7 | 0.5975 | −0.0530 | 0.3841 | 0.033* | |
| C8 | 0.4441 (2) | 0.09170 (15) | 0.30558 (12) | 0.0248 (3) | |
| C9 | 0.1973 (2) | 0.21924 (15) | 0.33327 (12) | 0.0247 (3) | |
| C10 | 0.0418 (2) | 0.26129 (16) | 0.39856 (12) | 0.0249 (3) | |
| C11 | 0.0148 (3) | 0.20327 (18) | 0.49683 (13) | 0.0308 (3) | |
| H11 | 0.1053 | 0.1409 | 0.5222 | 0.037* | |
| C12 | −0.1410 (3) | 0.23498 (19) | 0.55767 (13) | 0.0343 (4) | |
| H12 | −0.1584 | 0.1954 | 0.6244 | 0.041* | |
| C13 | −0.2699 (3) | 0.32515 (18) | 0.51912 (13) | 0.0309 (3) | |
| C14 | −0.2463 (3) | 0.38780 (18) | 0.42458 (13) | 0.0311 (3) | |
| H14 | −0.3354 | 0.4518 | 0.4010 | 0.037* | |
| C15 | −0.0895 (3) | 0.35560 (17) | 0.36425 (12) | 0.0294 (3) | |
| H15 | −0.0709 | 0.3982 | 0.2986 | 0.035* | |
| C16 | 0.3187 (3) | 0.53086 (16) | 0.19013 (13) | 0.0289 (3) | |
| H16 | 0.3011 | 0.5410 | 0.2658 | 0.035* | |
| C17 | 0.2245 (4) | 0.6446 (2) | 0.1325 (2) | 0.0562 (6) | |
| H17A | 0.3062 | 0.7380 | 0.1572 | 0.084* | |
| H17B | 0.0697 | 0.6346 | 0.1452 | 0.084* | |
| H17C | 0.2369 | 0.6346 | 0.0579 | 0.084* | |
| C18 | 0.5593 (3) | 0.54184 (19) | 0.17658 (15) | 0.0365 (4) | |
| H18A | 0.5814 | 0.5424 | 0.1024 | 0.055* | |
| H18B | 0.6082 | 0.4608 | 0.2087 | 0.055* | |
| H18C | 0.6443 | 0.6294 | 0.2099 | 0.055* | |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| S1 | 0.0300 (2) | 0.0256 (2) | 0.0255 (2) | 0.00633 (14) | −0.00408 (14) | 0.00327 (14) |
| F1 | 0.0410 (6) | 0.0612 (7) | 0.0347 (6) | 0.0199 (5) | 0.0117 (4) | −0.0051 (5) |
| O1 | 0.0268 (5) | 0.0275 (5) | 0.0268 (5) | 0.0092 (4) | 0.0047 (4) | 0.0070 (4) |
| O2 | 0.0415 (7) | 0.0373 (7) | 0.0412 (7) | 0.0215 (5) | 0.0103 (5) | 0.0049 (5) |
| O3 | 0.0502 (8) | 0.0434 (7) | 0.0398 (7) | 0.0246 (6) | 0.0185 (6) | 0.0069 (5) |
| O4A | 0.0480 (8) | 0.0365 (7) | 0.0234 (6) | 0.0108 (6) | 0.0003 (5) | 0.0014 (5) |
| O4B | 0.031 (3) | 0.022 (9) | 0.037 (10) | 0.006 (7) | −0.004 (4) | 0.018 (7) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C1 | 0.0246 (7) | 0.0230 (7) | 0.0245 (7) | 0.0049 (5) | 0.0009 (5) | 0.0031 (5) |
| C2 | 0.0245 (7) | 0.0216 (7) | 0.0266 (7) | 0.0035 (5) | 0.0011 (6) | 0.0024 (5) |
| C3 | 0.0311 (8) | 0.0276 (7) | 0.0270 (8) | 0.0072 (6) | 0.0051 (6) | 0.0033 (6) |
| C4 | 0.0299 (8) | 0.0261 (7) | 0.0308 (8) | 0.0049 (6) | 0.0063 (6) | −0.0013 (6) |
| C5 | 0.0355 (9) | 0.0305 (8) | 0.0390 (9) | 0.0108 (7) | 0.0053 (7) | −0.0041 (7) |
| C6 | 0.0271 (7) | 0.0216 (7) | 0.0354 (8) | 0.0064 (6) | 0.0013 (6) | −0.0002 (6) |
| C7 | 0.0281 (8) | 0.0250 (7) | 0.0309 (8) | 0.0073 (6) | 0.0022 (6) | 0.0053 (6) |
| C8 | 0.0234 (7) | 0.0234 (7) | 0.0272 (7) | 0.0038 (5) | 0.0033 (6) | 0.0020 (6) |
| C9 | 0.0233 (7) | 0.0227 (7) | 0.0277 (7) | 0.0050 (5) | −0.0003 (6) | 0.0045 (6) |
| C10 | 0.0224 (7) | 0.0245 (7) | 0.0268 (7) | 0.0028 (5) | 0.0018 (5) | 0.0016 (6) |
| C11 | 0.0322 (8) | 0.0335 (8) | 0.0285 (8) | 0.0106 (6) | 0.0027 (6) | 0.0053 (6) |
| C12 | 0.0388 (9) | 0.0406 (9) | 0.0247 (8) | 0.0093 (7) | 0.0060 (7) | 0.0051 (7) |
| C13 | 0.0273 (8) | 0.0370 (8) | 0.0285 (8) | 0.0063 (6) | 0.0045 (6) | −0.0073 (6) |
| C14 | 0.0284 (8) | 0.0341 (8) | 0.0325 (8) | 0.0110 (6) | 0.0005 (6) | −0.0001 (6) |
| C15 | 0.0284 (8) | 0.0329 (8) | 0.0284 (8) | 0.0091 (6) | 0.0037 (6) | 0.0057 (6) |
| C16 | 0.0341 (8) | 0.0233 (7) | 0.0292 (8) | 0.0061 (6) | 0.0008 (6) | 0.0002 (6) |
| C17 | 0.0508 (12) | 0.0306 (9) | 0.0863 (17) | 0.0127 (8) | −0.0105 (11) | 0.0137 (10) |
| C18 | 0.0325 (9) | 0.0340 (9) | 0.0409 (9) | 0.0021 (7) | 0.0025 (7) | 0.0068 (7) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| S1—O4B | 1.4716 (17) | C7—H7 | 0.9500 |
| S1—O4A | 1.4724 (13) | C9—C10 | 1.456 (2) |
| S1—C1 | 1.7783 (15) | C10—C15 | 1.398 (2) |
| S1—C16 | 1.8290 (16) | C10—C11 | 1.399 (2) |
| F1—C13 | 1.3623 (18) | C11—C12 | 1.382 (2) |
| O1—C8 | 1.3707 (18) | C11—H11 | 0.9500 |
| O1—C9 | 1.3847 (17) | C12—C13 | 1.373 (2) |
| O2—C6 | 1.3712 (19) | C12—H12 | 0.9500 |
| O2—C5 | 1.437 (2) | C13—C14 | 1.371 (2) |
| O3—C4 | 1.3740 (19) | C14—C15 | 1.384 (2) |
| O3—C5 | 1.428 (2) | C14—H14 | 0.9500 |
| C1—C9 | 1.371 (2) | C15—H15 | 0.9500 |
| C1—C2 | 1.443 (2) | C16—C18 | 1.510 (2) |
| C2—C8 | 1.393 (2) | C16—C17 | 1.521 (2) |
| C2—C3 | 1.412 (2) | C16—H16 | 1.0000 |
| C3—C4 | 1.366 (2) | C17—H17A | 0.9800 |
| C3—H3 | 0.9500 | C17—H17B | 0.9800 |
| C4—C6 | 1.398 (2) | C17—H17C | 0.9800 |
| C5—H5A | 0.9900 | C18—H18A | 0.9800 |
| C5—H5B | 0.9900 | C18—H18B | 0.9800 |
| C6—C7 | 1.371 (2) | C18—H18C | 0.9800 |
| C7—C8 | 1.395 (2) | | |

| | | | |
|---|---|---|---|
| O4B—S1—O4A | 104.0 (8) | O1—C9—C10 | 114.53 (13) |
| O4B—S1—C1 | 124.7 (6) | C15—C10—C11 | 118.20 (15) |
| O4A—S1—C1 | 107.07 (7) | C15—C10—C9 | 121.63 (14) |
| O4B—S1—C16 | 114.1 (7) | C11—C10—C9 | 120.13 (14) |

| | | | |
|---|---|---|---|
| O4A—S1—C16 | 107.44 (8) | C12—C11—C10 | 121.20 (15) |
| C1—S1—C16 | 98.56 (7) | C12—C11—H11 | 119.4 |
| C8—O1—C9 | 107.04 (11) | C10—C11—H11 | 119.4 |
| C6—O2—C5 | 105.72 (13) | C13—C12—C11 | 118.27 (15) |
| C4—O3—C5 | 106.07 (13) | C13—C12—H12 | 120.9 |
| C9—C1—C2 | 107.40 (13) | C11—C12—H12 | 120.9 |
| C9—C1—S1 | 127.89 (12) | F1—C13—C14 | 118.35 (15) |
| C2—C1—S1 | 124.56 (11) | F1—C13—C12 | 118.79 (15) |
| C8—C2—C3 | 120.03 (14) | C14—C13—C12 | 122.85 (15) |
| C8—C2—C1 | 105.09 (13) | C13—C14—C15 | 118.45 (15) |
| C3—C2—C1 | 134.88 (14) | C13—C14—H14 | 120.8 |
| C4—C3—C2 | 114.84 (14) | C15—C14—H14 | 120.8 |
| C4—C3—H3 | 122.6 | C14—C15—C10 | 120.98 (15) |
| C2—C3—H3 | 122.6 | C14—C15—H15 | 119.5 |
| C3—C4—O3 | 126.86 (15) | C10—C15—H15 | 119.5 |
| C3—C4—C6 | 123.73 (15) | C18—C16—C17 | 112.60 (15) |
| O3—C4—C6 | 109.37 (14) | C18—C16—S1 | 111.75 (12) |
| O3—C5—O2 | 107.97 (13) | C17—C16—S1 | 107.09 (12) |
| O3—C5—H5A | 110.1 | C18—C16—H16 | 108.4 |
| O2—C5—H5A | 110.1 | C17—C16—H16 | 108.4 |
| O3—C5—H5B | 110.1 | S1—C16—H16 | 108.4 |
| O2—C5—H5B | 110.1 | C16—C17—H17A | 109.5 |
| H5A—C5—H5B | 108.4 | C16—C17—H17B | 109.5 |
| O2—C6—C7 | 127.12 (15) | H17A—C17—H17B | 109.5 |
| O2—C6—C4 | 109.74 (14) | C16—C17—H17C | 109.5 |
| C7—C6—C4 | 123.13 (14) | H17A—C17—H17C | 109.5 |
| C6—C7—C8 | 113.00 (14) | H17B—C17—H17C | 109.5 |
| C6—C7—H7 | 123.5 | C16—C18—H18A | 109.5 |
| C8—C7—H7 | 123.5 | C16—C18—H18B | 109.5 |
| O1—C8—C2 | 110.64 (13) | H18A—C18—H18B | 109.5 |
| O1—C8—C7 | 124.08 (13) | C16—C18—H18C | 109.5 |
| C2—C8—C7 | 125.27 (14) | H18A—C18—H18C | 109.5 |
| C1—C9—O1 | 109.82 (13) | H18B—C18—H18C | 109.5 |
| C1—C9—C10 | 135.63 (14) | | |

| | | | |
|---|---|---|---|
| O4B—S1—C1—C9 | −43.3 (10) | C3—C2—C8—C7 | 0.3 (2) |
| O4A—S1—C1—C9 | −164.61 (14) | C1—C2—C8—C7 | −179.36 (14) |
| C16—S1—C1—C9 | 84.09 (15) | C6—C7—C8—O1 | −178.87 (13) |
| O4B—S1—C1—C2 | 131.6 (10) | C6—C7—C8—C2 | 0.3 (2) |
| O4A—S1—C1—C2 | 10.31 (15) | C2—C1—C9—O1 | 0.33 (16) |
| C16—S1—C1—C2 | −100.99 (13) | S1—C1—C9—O1 | 175.95 (10) |
| C9—C1—C2—C8 | −0.13 (16) | C2—C1—C9—C10 | −178.34 (16) |
| S1—C1—C2—C8 | −175.93 (11) | S1—C1—C9—C10 | −2.7 (3) |
| C9—C1—C2—C3 | −179.69 (16) | C8—O1—C9—C1 | −0.41 (16) |
| S1—C1—C2—C3 | 4.5 (3) | C8—O1—C9—C10 | 178.57 (12) |
| C8—C2—C3—C4 | −0.7 (2) | C1—C9—C10—C15 | 0.4 (3) |
| C1—C2—C3—C4 | 178.84 (16) | O1—C9—C10—C15 | −178.21 (13) |
| C2—C3—C4—O3 | −177.24 (15) | C1—C9—C10—C11 | 178.06 (17) |

| | | | |
|---|---|---|---|
| C2—C3—C4—C6 | 0.6 (2) | O1—C9—C10—C11 | −0.6 (2) |
| C5—O3—C4—C3 | −175.24 (16) | C15—C10—C11—C12 | 1.7 (2) |
| C5—O3—C4—C6 | 6.70 (18) | C9—C10—C11—C12 | −176.06 (15) |
| C4—O3—C5—O2 | −10.48 (18) | C10—C11—C12—C13 | 0.1 (3) |
| C6—O2—C5—O3 | 10.29 (17) | C11—C12—C13—F1 | 177.04 (15) |
| C5—O2—C6—C7 | 175.46 (16) | C11—C12—C13—C14 | −1.9 (3) |
| C5—O2—C6—C4 | −6.21 (17) | F1—C13—C14—C15 | −177.15 (14) |
| C3—C4—C6—O2 | −178.41 (15) | C12—C13—C14—C15 | 1.8 (3) |
| O3—C4—C6—O2 | −0.28 (18) | C13—C14—C15—C10 | 0.1 (2) |
| C3—C4—C6—C7 | 0.0 (3) | C11—C10—C15—C14 | −1.8 (2) |
| O3—C4—C6—C7 | 178.12 (14) | C9—C10—C15—C14 | 175.91 (14) |
| O2—C6—C7—C8 | 177.70 (14) | O4B—S1—C16—C18 | −159.6 (8) |
| C4—C6—C7—C8 | −0.4 (2) | O4A—S1—C16—C18 | −44.95 (14) |
| C9—O1—C8—C2 | 0.32 (16) | C1—S1—C16—C18 | 66.05 (13) |
| C9—O1—C8—C7 | 179.57 (14) | O4B—S1—C16—C17 | −35.9 (8) |
| C3—C2—C8—O1 | 179.52 (13) | O4A—S1—C16—C17 | 78.81 (15) |
| C1—C2—C8—O1 | −0.12 (16) | C1—S1—C16—C17 | −170.20 (14) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C5—H5*B*···O4*A*$^{i}$ | 0.99 | 2.27 | 3.231 (2) | 163 |
| C18—H18*A*···O4*A*$^{ii}$ | 0.98 | 2.49 | 3.354 (2) | 147 |

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