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Crystal structure of 1-fluoro-1,3-dihydrobenzo[c]thiophene 2,2-dioxide

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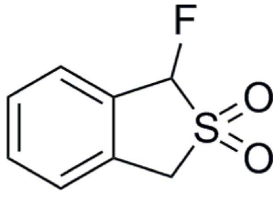
In the title compound, $C_8H_7FO_2S$, the thiophene ring has an envelope conformation, with the S atom bearing the two O atoms being the flap. In the crystal, molecules are linked by C—H···O and C—H···F interactions, generating a three-dimensional network structure.

Keywords: crystal structure; sulfone; fluorine; dihydrobenzothiophene; C—H···O and C—H···F interactions.

CCDC reference: 1421889

1. Related literature

For the use of of α-fluoro sulfones in organic synthesis, see: Fukuzumi *et al.* (2006); Li *et al.* (2006); Prakash *et al.* (2003); Zhao *et al.* (2013). For their synthesis, see: Jiang *et al.* (2014); Ni *et al.* (2008).



2. Experimental

2.1. Crystal data

$C_8H_7FO_2S$
M_r = 186.20
Monoclinic, $P2_1/c$
a = 5.7772 (5) Å
b = 8.3886 (6) Å
c = 16.8717 (12) Å
β = 99.742 (6)°
V = 805.86 (11) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 3.38 mm^{-1}
T = 296 K
0.05 × 0.03 × 0.02 mm

2.2. Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.418, T_{max} = 0.753
6724 measured reflections
1475 independent reflections
1206 reflections with $I > 2\sigma(I)$
R_{int} = 0.080

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.068
$wR(F^2)$ = 0.205
S = 1.13
1475 reflections
109 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.58 e Å^{-3}
$\Delta\rho_{min}$ = −0.98 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O1^{i}	0.93	2.95	3.687 (6)	138
C1—H1···O1^{ii}	0.98	2.47	3.266 (5)	139
C1—H1···O2^{ii}	0.98	3.48	4.331 (6)	147
C4—H4···O2^{iii}	0.93	2.54	3.371 (5)	148
C5—H5···F1^{iv}	0.93	2.81	3.640 (5)	150
C8—H8B···O2^{v}	0.97	2.50	3.406 (5)	156

Symmetry codes: (i) $x-1, y, z$; (ii) $-x+1, -y+2, -z+2$; (iii) $x-1, -y+\frac{3}{2}, z-\frac{1}{2}$; (iv) $x, -y+\frac{3}{2}, z-\frac{1}{2}$; (v) $-x+1, -y+1, -z+2$.

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: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009) and *Mercury* (Macrae, 2006); software used to prepare material for publication: *OLEX2*.

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: WM5205).

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supporting information

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Crystal structure of 1-fluoro-1,3-dihydrobenzo[*c*]thiophene 2,2-dioxide

Ying Zou, Zibin Qiu, Renming Tang, Kaixu Yuan and Ya Li

S1. Synthesis and crystallization

Lithium bis(trimethylsilyl)amide (LiHMDS; 2.2 ml, 1.0 M in THF, 2.2 mmol, 2.2 equiv) and anhydrous $ZnCl_2$ (341 mg, 2.5 mmol, 2.5 equiv) were dissolved in 12 ml THF. 10 minutes later, 1,3-dihydrobenzo[*c*]thiophene-2,2-dioxide (168 mg, 1.0 mmol, 1.0 equiv) was added into the mixture under N_2 atmosphere. The reaction was stirred for one hour. Then *N*-fluorobenzenesulfonimide (NFSI; 2.0 equiv, 632 mg, 2.0 mmol) was added into the mixture in a flash. The reaction was allowed at room temperature for half an hour and quenched by addition of H_2O. After extraction with ethyl acetate, the organic layer was dried over anhydrous Na_2SO_4, filtered and removed under vacuum. The crude product was purified by flash column chromatography on silica gel with ethyl acetate/hexane (1:3) to provide the title compound (97 mg, 52%). The obtained powder was recrystallized from dichloromethane/hexane (1:10) solution to give colourless crystals.

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.64-7.66 (d, J = 8.0 Hz, 1H), 7.56-7.59 (m, 1H), 7.48-7.52 (m, 1H), 7.36-7.38 (d, J = 8.0 Hz, 1H), 6.03 (d, J = 56.4 Hz, 1H), 4.40 (dd, J = 60.0, 16.0 Hz, 2H). ^{19}F NMR (376 MHz, $CDCl_3$) δ = -153.70 (d, J = 56.4 Hz). ^{13}C NMR (101 MHz, $CDCl_3$) δ = 128.6 (d, J = 4.0 Hz), 126.1, 125.5 (d, J = 4.0 Hz), 124.8, 122.5, 122.1, 94.2 (d, J = 86.4 Hz), 49.9. MS (EI) m/z: 168 $[M + H - 19]^+$. HRMS (EI) m/z: calcd for $C_8H_8O_2S$ $[M + H - 19]^+$ 168.0245, found 168.0251.

S2. Refinement

All H atoms of the phenyl groups were placed at calculated positions and treated as riding on their parent atoms, with C—H = 0.93 Å and $U_{iso}(H) = 1.2U_{eq}(C)$. The methylene H atoms were found from a difference map. Their positions were refined with C—H = 0.97 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

S3. Results and discussion

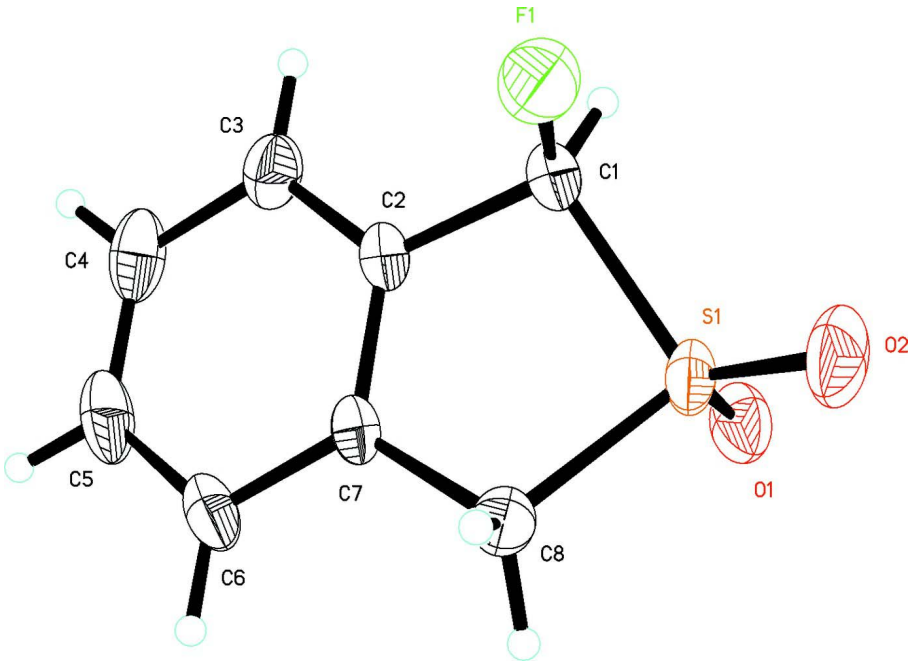

Figure 1

Molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level.

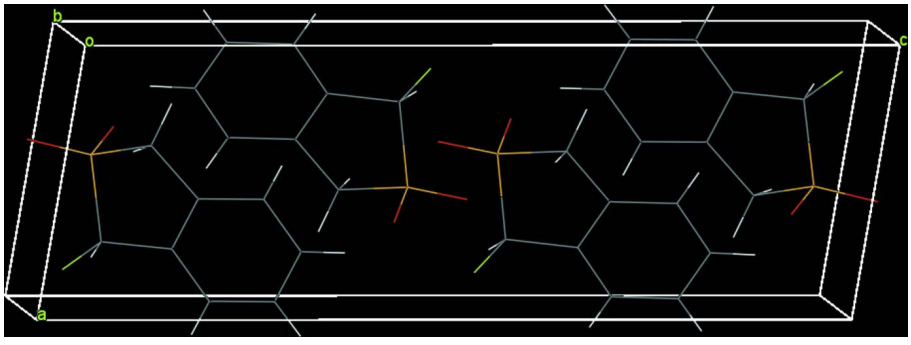

Figure 2

Packing of the molecules in the unit cell in a view approximately along [010].

1-Fluoro-1,3-dihydrobenzo[*c*]thiophene 2,2-dioxide

Crystal data

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c = 16.8717 (12) Å
β = 99.742 (6)°
V = 805.86 (11) Å^3
Z = 4
F(000) = 384
D_x = 1.535 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 1262 reflections
θ = 5.3–66.3°
μ = 3.38 mm^{-1}
T = 296 K
Block, colourless
0.05 × 0.03 × 0.02 mm

Data collection

Bruker APEXII CCD diffractometer
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.418, T_{max} = 0.753
6724 measured reflections
1475 independent reflections
1206 reflections with $I > 2\sigma(I)$
R_{int} = 0.080
θ_{max} = 69.6°, θ_{min} = 5.3°
$h = -6 \rightarrow 6$
$k = -10 \rightarrow 9$
$l = -19 \rightarrow 17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.068
$wR(F^2)$ = 0.205
S = 1.13
1475 reflections
109 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1242P)^2 + 0.3031P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.58 e Å^{-3}
$\Delta\rho_{min}$ = −0.98 e Å^{-3}

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}
S1	0.58177 (16)	0.76462 (12)	0.95887 (5)	0.0438 (4)
F1	0.1554 (5)	0.6784 (4)	0.96561 (16)	0.0686 (8)
O1	0.6976 (5)	0.9083 (4)	0.94354 (18)	0.0593 (9)
O2	0.6325 (6)	0.6952 (5)	1.03759 (17)	0.0675 (10)
C1	0.2672 (7)	0.7960 (5)	0.9290 (2)	0.0442 (9)
H1	0.2194	0.9030	0.9431	0.053*
C2	0.2387 (7)	0.7727 (4)	0.8397 (2)	0.0381 (8)
C3	0.0537 (8)	0.8364 (5)	0.7852 (3)	0.0521 (10)
H3	−0.0632	0.8964	0.8027	0.063*
C4	0.0492 (10)	0.8075 (6)	0.7037 (3)	0.0633 (13)
H4	−0.0734	0.8477	0.6661	0.076*
C5	0.2237 (10)	0.7203 (6)	0.6781 (3)	0.0638 (13)
H5	0.2178	0.7029	0.6233	0.077*
C6	0.4093 (8)	0.6576 (5)	0.7325 (2)	0.0512 (10)
H6	0.5276	0.5994	0.7147	0.061*
C7	0.4136 (6)	0.6840 (4)	0.8142 (2)	0.0373 (8)
C8	0.6028 (7)	0.6252 (5)	0.8808 (2)	0.0442 (9)
H8A	0.7564	0.6286	0.8649	0.053*
H8B	0.5711	0.5175	0.8969	0.053*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0448 (6)	0.0535 (7)	0.0294 (6)	−0.0075 (4)	−0.0046 (4)	−0.0040 (3)
F1	0.0590 (15)	0.098 (2)	0.0493 (15)	−0.0118 (15)	0.0102 (11)	0.0065 (13)
O1	0.0623 (18)	0.0629 (19)	0.0519 (16)	−0.0229 (15)	0.0075 (13)	−0.0147 (14)
O2	0.068 (2)	0.095 (2)	0.0328 (16)	−0.0078 (18)	−0.0109 (14)	0.0109 (15)
C1	0.047 (2)	0.054 (2)	0.0302 (19)	−0.0005 (17)	0.0026 (14)	−0.0082 (15)
C2	0.045 (2)	0.0384 (19)	0.0283 (18)	−0.0040 (15)	−0.0003 (14)	−0.0037 (13)
C3	0.053 (2)	0.051 (2)	0.047 (2)	0.0017 (18)	−0.0075 (17)	0.0000 (17)
C4	0.078 (3)	0.063 (3)	0.038 (2)	−0.013 (2)	−0.019 (2)	0.0103 (19)
C5	0.092 (4)	0.069 (3)	0.027 (2)	−0.021 (3)	−0.001 (2)	−0.0043 (18)
C6	0.068 (3)	0.052 (2)	0.0358 (19)	−0.0143 (19)	0.0140 (17)	−0.0135 (16)
C7	0.0453 (19)	0.0335 (18)	0.0313 (17)	−0.0072 (14)	0.0011 (14)	−0.0070 (13)
C8	0.0427 (19)	0.043 (2)	0.045 (2)	0.0018 (16)	0.0020 (15)	−0.0037 (16)

Geometric parameters (Å, º)

S1—O1	1.423 (3)	C3—C4	1.392 (6)
S1—O2	1.434 (3)	C4—H4	0.9300
S1—C1	1.821 (4)	C4—C5	1.373 (8)
S1—C8	1.781 (4)	C5—H5	0.9300
F1—C1	1.381 (5)	C5—C6	1.391 (7)
C1—H1	0.9800	C6—H6	0.9300
C1—C2	1.502 (5)	C6—C7	1.392 (5)
C2—C3	1.393 (5)	C7—C8	1.512 (5)
C2—C7	1.381 (5)	C8—H8A	0.9700
C3—H3	0.9300	C8—H8B	0.9700
O1—S1—O2	118.9 (2)	C3—C4—H4	119.6
O1—S1—C1	107.8 (2)	C5—C4—C3	120.7 (4)
O1—S1—C8	109.19 (19)	C5—C4—H4	119.6
O2—S1—C1	110.6 (2)	C4—C5—H5	119.4
O2—S1—C8	112.9 (2)	C4—C5—C6	121.3 (4)
C8—S1—C1	94.61 (18)	C6—C5—H5	119.4
S1—C1—H1	112.0	C5—C6—H6	120.8
F1—C1—S1	107.1 (3)	C5—C6—C7	118.4 (4)
F1—C1—H1	112.0	C7—C6—H6	120.8
F1—C1—C2	112.1 (3)	C2—C7—C6	120.2 (4)
C2—C1—S1	101.1 (3)	C2—C7—C8	114.8 (3)
C2—C1—H1	112.0	C6—C7—C8	125.0 (4)
C3—C2—C1	123.6 (4)	S1—C8—H8A	111.4
C7—C2—C1	114.9 (3)	S1—C8—H8B	111.4
C7—C2—C3	121.4 (4)	C7—C8—S1	101.8 (2)
C2—C3—H3	121.0	C7—C8—H8A	111.4
C4—C3—C2	118.0 (4)	C7—C8—H8B	111.4
C4—C3—H3	121.0	H8A—C8—H8B	109.3

S1—C1—C2—C3	−156.5 (3)	C1—C2—C7—C8	0.2 (5)
S1—C1—C2—C7	22.5 (4)	C2—C3—C4—C5	−0.7 (7)
F1—C1—C2—C3	89.7 (5)	C2—C7—C8—S1	−23.5 (4)
F1—C1—C2—C7	−91.2 (4)	C3—C2—C7—C6	0.7 (6)
O1—S1—C1—F1	−162.2 (2)	C3—C2—C7—C8	179.3 (4)
O1—S1—C1—C2	80.4 (3)	C3—C4—C5—C6	0.3 (8)
O1—S1—C8—C7	−79.0 (3)	C4—C5—C6—C7	0.6 (7)
O2—S1—C1—F1	−30.6 (3)	C5—C6—C7—C2	−1.0 (6)
O2—S1—C1—C2	−148.1 (3)	C5—C6—C7—C8	−179.6 (4)
O2—S1—C8—C7	146.3 (3)	C6—C7—C8—S1	155.1 (3)
C1—S1—C8—C7	31.6 (3)	C7—C2—C3—C4	0.2 (6)
C1—C2—C3—C4	179.3 (4)	C8—S1—C1—F1	86.0 (3)
C1—C2—C7—C6	−178.4 (3)	C8—S1—C1—C2	−31.5 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O1^{i}	0.93	2.95	3.687 (6)	138
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