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5-Fluoroisatin–dimethyl sufoxide (1/1)

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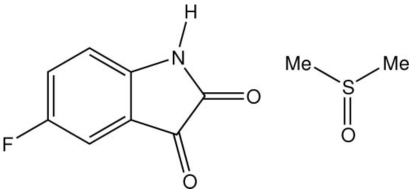
Key indicators: single-crystal X-ray study; T = 150 K; mean σ(C–C) = 0.002 Å; R factor = 0.032; wR factor = 0.087; data-to-parameter ratio = 14.9.

The title dimethyl sulfoxide (DMSO) solvate, $C_8H_4FNO_2 \cdot C_2H_6OS$, was isolated during a manual crystallization screen on 5-fluoroisatin (5-fluoroindoline-2,3-dione). Molecules of 5-fluoroisatin are linked *via* C—H⋯O interactions to form chains parallel to $(0\bar{1}1)$ from which N—H⋯O hydrogen-bonded DMSO molecules protrude.

Related literature

7-Fluoroisatin is reported to be a suitable starting material for the preparation of 7-substituted-2-indolinones, a class of compounds which are understood to display anti-convulsant activity in animals (Canas-Rodriguez & Leeming, 1972). 5-Fluoroisatin forms sheets through double N—H⋯O hydrogen bonds and C—H⋯O interactions (Naumov *et al.*, 2000). The 1,4-dioxane (Shankland *et al.*, 2007) and DMSO (Mohamed *et al.*, 2007*a*) solvates of 7-fluoroisatin have also been prepared, as well as the oxindole derivative of 5-fluoroisatin (Mohamed *et al.*, 2007*b*).



Experimental

Crystal data

$C_{10}H_{10}FNO_3S$
M_r = 243.25
Triclinic, $P\bar{1}$
a = 8.2828 (13) Å
b = 8.3702 (13) Å
c = 8.7613 (14) Å
α = 91.326 (2)°
β = 117.099 (2)°
γ = 97.337 (2)°
V = 534.02 (15) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.31 mm^{-1}
T = 150 (2) K
0.79 × 0.47 × 0.22 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.871, T_{max} = 1.000 (expected range = 0.814–0.934)
4674 measured reflections
2429 independent reflections
2282 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.087
S = 1.04
2426 reflections
163 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1—H4⋯O3	0.89 (2)	1.92 (2)	2.7935 (15)	167.0 (18)
C6—H2⋯O2^{i}	0.92 (2)	2.51 (2)	3.3484 (17)	152.1 (16)

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek 2003).

The authors acknowledge the Research Councils UK Basic Technology Programme for support through the programme ‘Control and Prediction of the Organic Solid State’ (http://www.cposs.org.uk).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2183).

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supplementary materials

Acta Cryst. (2007). E**63**, o3576   [**doi:10.1107/S1600536807035271**]

5-Fluoroisatin-dimethyl sufoxide (1/1)

S. Mohamed, S. A. Barnett and **D. A. Tocher**

Comment

The title solvate, (I), (Figure 1) was isolated during a manual crystallization screen on 5-fluoroisatin. The crystallization screen was motivated by a wider investigation into the potential polymorphism displayed by the isomeric compounds 7-fluoroisatin and 5-fluoroisatin (Shankland *et al.*, 2007; Mohamed *et al.*, 2007*a*,b).

In the crystal structure of (I), molecules of 5-fluoroisatin form chains *via* C—H···O interactions with the DMSO molecules then linked to one side of these chains by N—H···O hydrogen bonds. In the same way as the 7-fluoroisatin DMSO solvate (Mohamed *et al.*, 2007*b*), ribbons are formed by the interdigitation of the DMSO molecules (Figure 2 and Table 1). The C—H···O interactions for this compound occur using O2 (*i.e.* the O not adjacent to the N position) whereas the chain formed by 7-fluoroisatin DMSO (Mohamed *et al.*, 2007*b*) uses O1. Viewing down the *a* axis, these ribbons can be seen to form planes parallel to (0 − 1 1) with separations of 2.38 Å and 3.49 Å where the DMSO molecules interdigitate (Figure 3).

Experimental

Single crystals of the title solvate were grown from a saturated dimethylsulfoxide solution by slow solvent evaporation at 298 K over a period of approximately one month.

Refinement

Methyl-H atoms were located from the difference map and refined using a rigid rotor model with C—H = 0.98 Å, and with U_{iso}(H) = 1.5U_{eq}(C). All other H atoms were refined freely so that the C—H distances were in the range 0.92 (2) to 0.958 (19) Å and N—H = 0.89 (2) Å. The three reflections with the greatest discrepancies were omitted from the refinement.

Figures

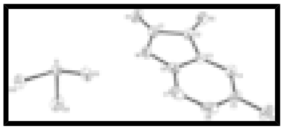


Fig. 1. The asymmetric unit of (I) showing the numbering scheme used. Displacement ellipsoids are drawn at the 50% probability level and hydrogen atoms have been omitted for clarity.

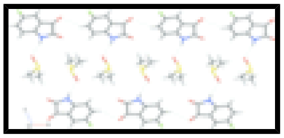
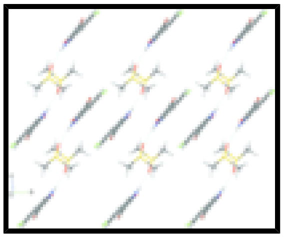


Fig. 2. The ribbon formed by 5-fluoroisatin and DMSO in (I) showing the showing the hydrogen-bonding interactions as blue dotted lines Colour code: C - dark grey, H - light grey, N - blue, O - red, F - green, S - yellow.



5-fluoroindoline-2,3-dione–dimethyl sulfoxide (1/1)

Crystal data

$C_{10}H_{10}FNO_3S$

$M_r = 243.25$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 8.2828$ (13) Å

$b = 8.3702$ (13) Å

$c = 8.7613$ (14) Å

$\alpha = 91.326$ (2)°

$\beta = 117.099$ (2)°

$\gamma = 97.337$ (2)°

$V = 534.02$ (15) Å^3

$Z = 2$

$F_{000} = 252$

$D_x = 1.513$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å

Cell parameters from 3349 reflections

$\theta = 2.6$–28.3°

$\mu = 0.31$ mm^{-1}

$T = 150$ (2) K

Block, orange

0.79 × 0.47 × 0.22 mm

Data collection

Bruker SMART APEX
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 150(2)$ K

ω rotation with narrow frames scans

Absorption correction: multi-scan
(SADABS; Bruker, 2001)

$T_{min} = 0.871$, $T_{max} = 1.000$

4674 measured reflections

2429 independent reflections

2282 reflections with $I > 2\sigma(I)$

$R_{int} = 0.025$

$\theta_{max} = 28.3°$

$\theta_{min} = 2.5°$

$h = -10 \rightarrow 10$

$k = -11 \rightarrow 11$

$l = -11 \rightarrow 11$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.032$

$wR(F^2) = 0.087$

$S = 1.04$

2426 reflections

Secondary atom site location: difference Fourier map

Hydrogen site location: difference Fourier map

H atoms treated by a mixture of
independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0489P)^2 + 0.1972P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.38$ e Å^{-3}

163 parameters	$\Delta\rho_{min}$ = −0.24 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2\mathrm{sigma}(F^2)$ 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}
C1	0.27263 (19)	0.28862 (15)	0.48470 (17)	0.0240 (3)
C2	0.20213 (18)	0.15613 (15)	0.33047 (17)	0.0216 (3)
C3	0.36806 (17)	0.12356 (15)	0.32246 (16)	0.0195 (2)
C4	0.39226 (19)	0.01360 (16)	0.21618 (17)	0.0239 (3)
H1	0.293 (3)	−0.059 (2)	0.128 (2)	0.036 (5)*
C5	0.5705 (2)	0.01559 (18)	0.24401 (18)	0.0274 (3)
C6	0.71742 (19)	0.12245 (19)	0.36432 (19)	0.0300 (3)
H2	0.831 (3)	0.116 (2)	0.372 (2)	0.041 (5)*
C7	0.69151 (18)	0.23337 (18)	0.46907 (18)	0.0273 (3)
H3	0.788 (3)	0.309 (2)	0.551 (2)	0.039 (5)*
C8	0.51515 (17)	0.22925 (15)	0.44893 (16)	0.0206 (2)
N1	0.45677 (16)	0.32363 (13)	0.54376 (14)	0.0239 (2)
H4	0.533 (3)	0.393 (2)	0.635 (2)	0.038 (5)*
O1	0.17722 (16)	0.34426 (13)	0.53751 (14)	0.0355 (3)
O2	0.04277 (13)	0.10042 (12)	0.24154 (14)	0.0305 (2)
F1	0.60203 (14)	−0.09251 (12)	0.14601 (12)	0.0407 (2)
C9	0.7818 (2)	0.41521 (18)	1.11959 (19)	0.0347 (3)
H9A	0.6925	0.3163	1.0666	0.052*
H9B	0.7853	0.4489	1.2291	0.052*
H9C	0.9036	0.3944	1.1405	0.052*
C10	0.90882 (19)	0.72168 (17)	1.10596 (17)	0.0277 (3)
H10A	1.0222	0.6799	1.1285	0.042*
H10B	0.9092	0.7493	1.2154	0.042*
H10C	0.9011	0.8186	1.0438	0.042*
O3	0.73930 (14)	0.52031 (12)	0.82378 (12)	0.0277 (2)
S1	0.71618 (4)	0.57126 (4)	0.97903 (4)	0.02225 (11)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0314 (7)	0.0205 (6)	0.0234 (6)	0.0050 (5)	0.0153 (5)	0.0022 (5)
C2	0.0245 (6)	0.0186 (6)	0.0260 (6)	0.0034 (5)	0.0154 (5)	0.0009 (5)
C3	0.0205 (6)	0.0187 (5)	0.0216 (6)	0.0034 (4)	0.0115 (5)	0.0032 (4)
C4	0.0263 (6)	0.0224 (6)	0.0259 (6)	0.0050 (5)	0.0143 (5)	0.0012 (5)
C5	0.0341 (7)	0.0315 (7)	0.0289 (7)	0.0153 (6)	0.0223 (6)	0.0093 (5)
C6	0.0213 (6)	0.0432 (8)	0.0329 (7)	0.0117 (6)	0.0164 (6)	0.0171 (6)
C7	0.0195 (6)	0.0331 (7)	0.0248 (6)	0.0002 (5)	0.0070 (5)	0.0087 (5)
C8	0.0228 (6)	0.0204 (6)	0.0185 (6)	0.0032 (5)	0.0094 (5)	0.0056 (4)
N1	0.0288 (6)	0.0209 (5)	0.0188 (5)	0.0009 (4)	0.0093 (4)	−0.0016 (4)
O1	0.0441 (6)	0.0347 (6)	0.0374 (6)	0.0116 (5)	0.0261 (5)	−0.0027 (4)
O2	0.0217 (5)	0.0288 (5)	0.0409 (6)	−0.0011 (4)	0.0161 (4)	−0.0056 (4)
F1	0.0485 (6)	0.0484 (6)	0.0435 (5)	0.0244 (4)	0.0325 (5)	0.0054 (4)
C9	0.0532 (9)	0.0262 (7)	0.0314 (7)	0.0078 (6)	0.0247 (7)	0.0061 (6)
C10	0.0280 (7)	0.0254 (7)	0.0241 (6)	−0.0001 (5)	0.0086 (5)	−0.0044 (5)
O3	0.0303 (5)	0.0305 (5)	0.0186 (4)	0.0033 (4)	0.0088 (4)	−0.0030 (4)
S1	0.01999 (17)	0.02330 (18)	0.02206 (17)	0.00339 (12)	0.00868 (13)	−0.00185 (12)

Geometric parameters (Å, °)

C1—O1	1.2135 (16)	C7—C8	1.3851 (18)
C1—N1	1.3563 (18)	C7—H3	0.94 (2)
C1—C2	1.5630 (18)	C8—N1	1.4076 (16)
C2—O2	1.2066 (16)	N1—H4	0.89 (2)
C2—C3	1.4666 (17)	C9—S1	1.7787 (15)
C3—C4	1.3899 (17)	C9—H9A	0.9800
C3—C8	1.3943 (18)	C9—H9B	0.9800
C4—C5	1.3805 (19)	C9—H9C	0.9800
C4—H1	0.958 (19)	C10—S1	1.7814 (13)
C5—F1	1.3610 (15)	C10—H10A	0.9800
C5—C6	1.379 (2)	C10—H10B	0.9800
C6—C7	1.393 (2)	C10—H10C	0.9800
C6—H2	0.92 (2)	O3—S1	1.5166 (10)
O1—C1—N1	128.94 (13)	C7—C8—C3	121.03 (12)
O1—C1—C2	125.24 (13)	C7—C8—N1	127.83 (12)
N1—C1—C2	105.82 (10)	C3—C8—N1	111.14 (11)
O2—C2—C3	130.81 (12)	C1—N1—C8	111.19 (11)
O2—C2—C1	124.37 (11)	C1—N1—H4	125.1 (12)
C3—C2—C1	104.81 (11)	C8—N1—H4	123.6 (12)
C4—C3—C8	121.86 (12)	S1—C9—H9A	109.5
C4—C3—C2	131.19 (12)	S1—C9—H9B	109.5
C8—C3—C2	106.95 (11)	H9A—C9—H9B	109.5
C5—C4—C3	115.84 (12)	S1—C9—H9C	109.5
C5—C4—H1	121.2 (11)	H9A—C9—H9C	109.5
C3—C4—H1	123.0 (11)	H9B—C9—H9C	109.5

F1—C5—C6	118.51 (12)	S1—C10—H10A	109.5
F1—C5—C4	118.10 (13)	S1—C10—H10B	109.5
C6—C5—C4	123.39 (13)	H10A—C10—H10B	109.5
C5—C6—C7	120.32 (12)	S1—C10—H10C	109.5
C5—C6—H2	117.3 (12)	H10A—C10—H10C	109.5
C7—C6—H2	122.3 (12)	H10B—C10—H10C	109.5
C8—C7—C6	117.46 (13)	O3—S1—C9	106.19 (6)
C8—C7—H3	120.1 (12)	O3—S1—C10	106.02 (6)
C6—C7—H3	122.4 (12)	C9—S1—C10	97.29 (7)

O1—C1—C2—O2	3.8 (2)	C4—C5—C6—C7	1.8 (2)
N1—C1—C2—O2	−176.78 (13)	C5—C6—C7—C8	1.1 (2)
O1—C1—C2—C3	−177.15 (13)	C6—C7—C8—C3	−3.22 (19)
N1—C1—C2—C3	2.24 (13)	C6—C7—C8—N1	177.15 (12)
O2—C2—C3—C4	−3.7 (2)	C4—C3—C8—C7	2.69 (19)
C1—C2—C3—C4	177.36 (13)	C2—C3—C8—C7	−177.12 (11)
O2—C2—C3—C8	176.07 (14)	C4—C3—C8—N1	−177.62 (11)
C1—C2—C3—C8	−2.86 (13)	C2—C3—C8—N1	2.58 (14)
C8—C3—C4—C5	0.11 (19)	O1—C1—N1—C8	178.59 (13)
C2—C3—C4—C5	179.86 (13)	C2—C1—N1—C8	−0.76 (14)
C3—C4—C5—F1	178.45 (11)	C7—C8—N1—C1	178.55 (13)
C3—C4—C5—C6	−2.3 (2)	C3—C8—N1—C1	−1.12 (15)
F1—C5—C6—C7	−179.01 (12)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H4···O3	0.89 (2)	1.92 (2)	2.7935 (15)	167.0 (18)
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Fig. 1

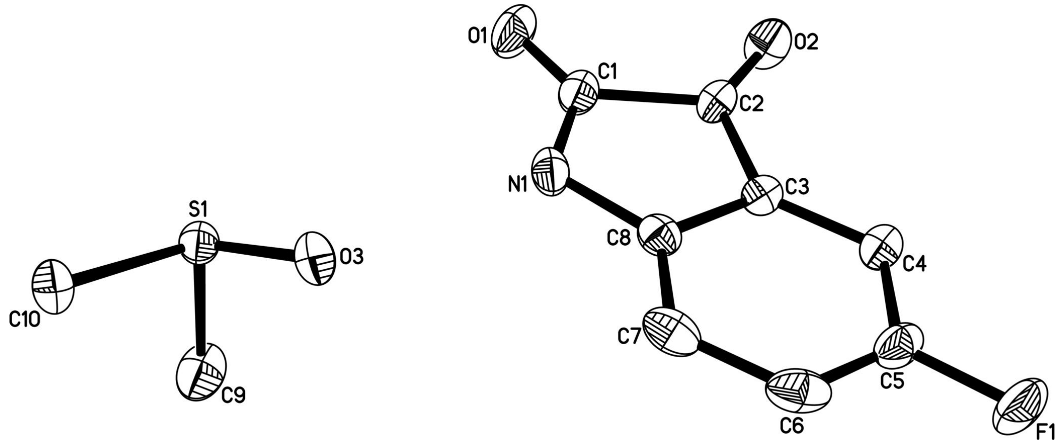

Fig. 2

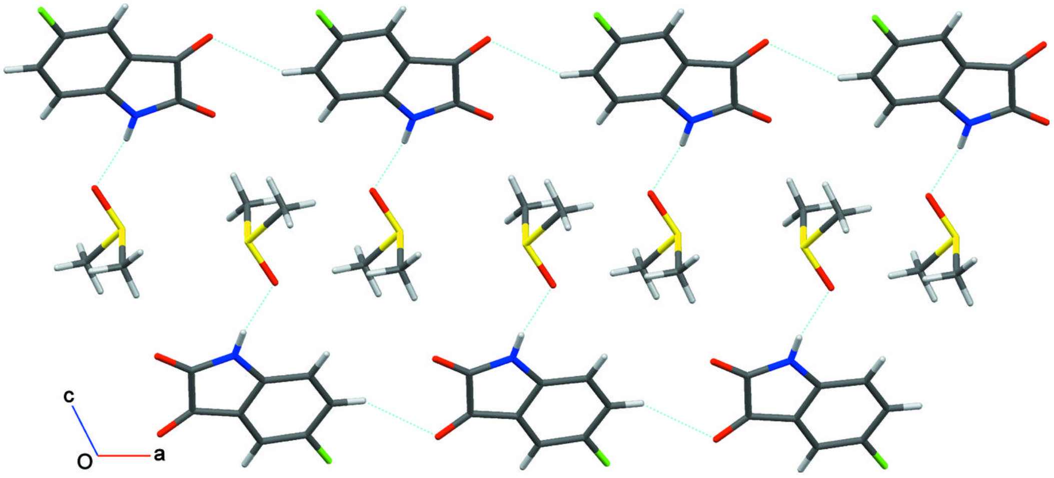

Fig. 3