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7-Fluoroisatin–dimethyl sulfoxide (1/1)

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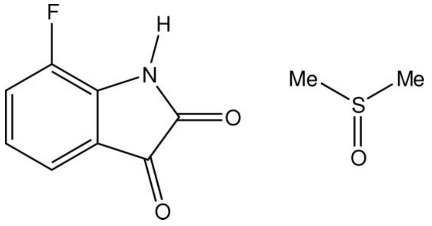
Key indicators: single-crystal X-ray study; T = 150 K; mean σ(C–C) = 0.002 Å; R factor = 0.031; wR factor = 0.085; data-to-parameter ratio = 16.5.

The title dimethyl sulfoxide (DMSO) solvate, $C_8H_4FNO_2\cdot C_2H_6OS$, was isolated during a manual crystallization screen on 7-fluoroisatin (7-fluoroindoline-2,3-dione). Molecules of 7-fluoroisatin are linked *via* C—H···O interactions to form chains parallel to the *a* direction, from which N—H···O hydrogen-bonded molecules of DMSO 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). The 1,4-dioxane solvate of 7-fluoroisatin (Shankland *et al.*, 2007) and the DMSO solvate of 5-fluoroisatin (Mohamed *et al.*, 2007*a*) 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 = 7.4917 (12) Å
b = 8.4911 (14) Å
c = 8.9542 (15) Å
α = 106.207 (2)°
β = 98.648 (2)°
γ = 96.242 (3)°
V = 533.89 (15) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.31 mm^{-1}
T = 150 (2) K
0.53 × 0.26 × 0.19 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.886, T_{max} = 1.000 (expected range = 0.836–0.943)
4718 measured reflections
2439 independent reflections
2355 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.085
S = 1.08
2436 reflections
148 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O3	0.88	1.89	2.7679 (14)	175
C6—H6A···O1^{i}	0.95	2.34	3.2870 (17)	175

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

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).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2182).

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supplementary materials

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7-Fluoroisatin-dimethyl sulfoxide (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 7-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), chains of 7-fluoroisatin are formed by C—H···O interactions and each 7-fluoroisatin is linked to a DMSO molecule *via* a N—H···O hydrogen bond. Two identical chains form ribbons by the interdigitation of the DMSO molecules (Figure 2 and Table 1). The C—H···O interactions for this compound occur using O1 (*i.e.* the O adjacent to the N position) whereas the chain formed by 5-fluoroisatin DMSO (Mohamed *et al.*, 2007*a*) uses O2. On viewing down the *c* axis, these ribbons can be seen to form planes parallel to (1 − 1 0) with separations of 2.36 Å and 3.40 Å where the DMSO molecules interdigitate (Figure 3).

Experimental

Single crystals of the title compound were crystallized from a saturated dimethyl sulfoxide solution by slow evaporation at 298 K over the course of four months.

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 $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(C). All other H atoms were geometrically placed and refined using a riding model with C—H = 0.95 Å and N—H = 0.88 Å, and with $U_{\rm iso}$(H) = 1.20 $U_{\rm eq}$(C, N). The three reflections with the greatest discrepancies were omitted from the refinement.

Figures

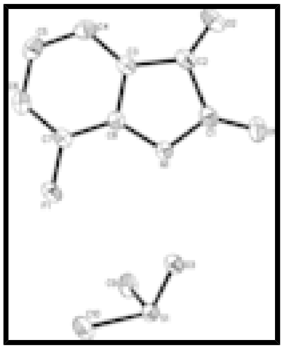

Fig. 1. The asymmetric unit of (I) showing the numbering scheme used. Displacement ellipsoids are drawn at the 50% probability level and hydrogen atoms have been omitted for clarity.

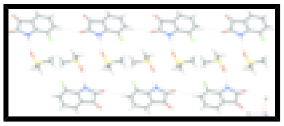

Fig. 2. The ribbon formed by 7-fluoroisatin and DMSO in (I) showing the hydrogen bonding interactions as blue dotted lines. Colour code: C - dark grey, H - light grey, N - blue, O - red, F - green, S - yellow.

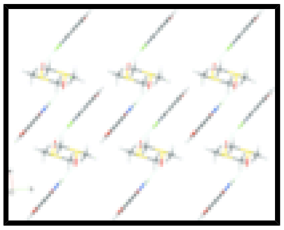

Fig. 3. Packing diagram showing the stacking of the ribbons in (I). Colour code: C - dark grey, H - light grey, N - blue, O - red, F - green, S - yellow.

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

Crystal data

$C_{10}H_{10}FNO_3S$
$M_r = 243.25$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 7.4917\ (12)$ Å
$b = 8.4911\ (14)$ Å
$c = 8.9542\ (15)$ Å
$\alpha = 106.207\ (2)°$
$\beta = 98.648\ (2)°$
$\gamma = 96.242\ (3)°$
$V = 533.89\ (15)$ 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 3848 reflections
$\theta = 2.4–28.3°$
$\mu = 0.31$ mm^{-1}
$T = 150\ (2)$ K
Rhomboid block, orange
0.53 × 0.26 × 0.19 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)
$T_{\min} = 0.886$, $T_{\max} = 1.000$
4718 measured reflections

2439 independent reflections
2355 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.028$
$\theta_{\max} = 28.3°$
$\theta_{\min} = 2.4°$
$h = -9 \rightarrow 9$
$k = -10 \rightarrow 11$
$l = -11 \rightarrow 11$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.031$

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.04P)^2 + 0.2177P]$
where $P = (F_o^2 + 2F_c^2)/3$

$wR(F^2)$ = 0.085	$(\Delta/\sigma)_{max}$ = 0.001
S = 1.08	$\Delta\rho_{max}$ = 0.37 e Å^{-3}
2436 reflections	$\Delta\rho_{min}$ = −0.25 e Å^{-3}
148 parameters	Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.036 (6)
Secondary atom site location: difference Fourier map	

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 $F^2 > 2\sigma(F^2)$ 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	0.48025 (17)	0.24882 (16)	0.36792 (14)	0.0223 (3)
C2	0.33034 (17)	0.10687 (15)	0.37074 (14)	0.0225 (3)
C3	0.33940 (16)	0.12133 (15)	0.53918 (14)	0.0205 (2)
C4	0.24087 (18)	0.03106 (16)	0.61588 (17)	0.0264 (3)
H4A	0.1467	−0.0588	0.5575	0.032*
C5	0.2834 (2)	0.07534 (17)	0.78007 (17)	0.0290 (3)
H5A	0.2168	0.0159	0.8349	0.035*
C6	0.42278 (19)	0.20619 (17)	0.86501 (15)	0.0265 (3)
H6A	0.4514	0.2354	0.9773	0.032*
C7	0.51927 (17)	0.29330 (15)	0.78552 (14)	0.0215 (2)
C8	0.47933 (16)	0.25344 (14)	0.62275 (14)	0.0185 (2)
N1	0.56176 (14)	0.32541 (13)	0.52209 (12)	0.0208 (2)
H1A	0.6534	0.4085	0.5539	0.025*
O1	0.51535 (14)	0.28218 (13)	0.25143 (11)	0.0301 (2)
O2	0.23455 (14)	0.00815 (12)	0.25429 (11)	0.0320 (2)
F1	0.65707 (11)	0.41893 (10)	0.86828 (9)	0.0312 (2)
C9	1.0906 (2)	0.46043 (18)	0.75001 (16)	0.0293 (3)
H9A	0.9919	0.4039	0.7857	0.044*
H9B	1.2019	0.4892	0.8315	0.044*
H9C	1.1139	0.3867	0.6516	0.044*
C10	0.9842 (2)	0.74243 (17)	0.90954 (15)	0.0302 (3)
H10A	0.9334	0.8442	0.9103	0.045*
H10B	1.0995	0.7701	0.9863	0.045*
H10C	0.8972	0.6662	0.9381	0.045*

O3	0.83993 (12)	0.59461 (11)	0.60778 (10)	0.0224 (2)
S1	1.02562 (4)	0.64525 (4)	0.71564 (3)	0.02143 (12)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0228 (6)	0.0253 (6)	0.0189 (6)	0.0085 (5)	0.0023 (4)	0.0060 (5)
C2	0.0230 (6)	0.0207 (6)	0.0213 (6)	0.0072 (5)	−0.0003 (4)	0.0032 (5)
C3	0.0210 (6)	0.0184 (5)	0.0208 (6)	0.0053 (4)	0.0016 (4)	0.0040 (4)
C4	0.0238 (6)	0.0210 (6)	0.0336 (7)	0.0016 (5)	0.0054 (5)	0.0074 (5)
C5	0.0332 (7)	0.0267 (6)	0.0324 (7)	0.0049 (5)	0.0131 (5)	0.0134 (5)
C6	0.0332 (7)	0.0289 (6)	0.0204 (6)	0.0096 (5)	0.0077 (5)	0.0092 (5)
C7	0.0226 (6)	0.0211 (6)	0.0186 (6)	0.0035 (4)	0.0012 (4)	0.0040 (4)
C8	0.0194 (5)	0.0183 (5)	0.0182 (5)	0.0054 (4)	0.0026 (4)	0.0059 (4)
N1	0.0216 (5)	0.0227 (5)	0.0173 (5)	0.0006 (4)	0.0019 (4)	0.0066 (4)
O1	0.0332 (5)	0.0411 (6)	0.0196 (4)	0.0103 (4)	0.0059 (4)	0.0126 (4)
O2	0.0350 (5)	0.0264 (5)	0.0246 (5)	0.0042 (4)	−0.0070 (4)	−0.0016 (4)
F1	0.0330 (4)	0.0338 (4)	0.0188 (4)	−0.0061 (3)	−0.0036 (3)	0.0038 (3)
C9	0.0341 (7)	0.0335 (7)	0.0249 (6)	0.0150 (6)	0.0060 (5)	0.0124 (5)
C10	0.0391 (7)	0.0267 (6)	0.0197 (6)	0.0048 (5)	−0.0008 (5)	0.0024 (5)
O3	0.0201 (4)	0.0250 (4)	0.0195 (4)	0.0013 (3)	−0.0012 (3)	0.0060 (3)
S1	0.01927 (17)	0.02451 (18)	0.01990 (17)	−0.00010 (11)	0.00060 (11)	0.00866 (12)

Geometric parameters (Å, °)

C1—O1	1.2118 (16)	C7—F1	1.3572 (14)
C1—N1	1.3640 (16)	C7—C8	1.3783 (17)
C1—C2	1.5640 (18)	C8—N1	1.3999 (15)
C2—O2	1.2087 (15)	N1—H1A	0.8800
C2—C3	1.4692 (17)	S1—O3	1.5122 (9)
C3—C4	1.3883 (18)	S1—C9	1.7848 (14)
C3—C8	1.3990 (16)	S1—C10	1.7914 (14)
C4—C5	1.389 (2)	C9—H9A	0.9800
C4—H4A	0.9500	C9—H9B	0.9800
C5—C6	1.393 (2)	C9—H9C	0.9800
C5—H5A	0.9500	C10—H10A	0.9800
C6—C7	1.3816 (18)	C10—H10B	0.9800
C6—H6A	0.9500	C10—H10C	0.9800
O1—C1—N1	127.72 (13)	C7—C8—C3	118.45 (11)
O1—C1—C2	126.38 (12)	C7—C8—N1	129.33 (11)
N1—C1—C2	105.91 (10)	C3—C8—N1	112.22 (10)
O2—C2—C3	130.21 (13)	C1—N1—C8	110.58 (10)
O2—C2—C1	124.66 (12)	C1—N1—H1A	124.7
C3—C2—C1	105.10 (10)	C8—N1—H1A	124.7
C4—C3—C8	121.77 (11)	O3—S1—C9	106.48 (6)
C4—C3—C2	132.07 (12)	O3—S1—C10	105.88 (6)
C8—C3—C2	106.16 (11)	C9—S1—C10	97.75 (6)
C3—C4—C5	118.36 (12)	S1—C9—H9A	109.5

C3—C4—H4A	120.8	S1—C9—H9B	109.5
C5—C4—H4A	120.8	H9A—C9—H9B	109.5
C4—C5—C6	120.58 (12)	S1—C9—H9C	109.5
C4—C5—H5A	119.7	H9A—C9—H9C	109.5
C6—C5—H5A	119.7	H9B—C9—H9C	109.5
C7—C6—C5	119.82 (12)	S1—C10—H10A	109.5
C7—C6—H6A	120.1	S1—C10—H10B	109.5
C5—C6—H6A	120.1	H10A—C10—H10B	109.5
F1—C7—C8	119.24 (11)	S1—C10—H10C	109.5
F1—C7—C6	119.73 (11)	H10A—C10—H10C	109.5
C8—C7—C6	121.02 (12)	H10B—C10—H10C	109.5

O1—C1—C2—O2	2.9 (2)	C5—C6—C7—C8	0.33 (19)
N1—C1—C2—O2	−177.03 (12)	F1—C7—C8—C3	178.67 (10)
O1—C1—C2—C3	−178.51 (12)	C6—C7—C8—C3	−0.56 (18)
N1—C1—C2—C3	1.60 (12)	F1—C7—C8—N1	−0.18 (19)
O2—C2—C3—C4	−1.6 (2)	C6—C7—C8—N1	−179.41 (12)
C1—C2—C3—C4	179.85 (13)	C4—C3—C8—C7	0.20 (18)
O2—C2—C3—C8	177.65 (13)	C2—C3—C8—C7	−179.16 (10)
C1—C2—C3—C8	−0.88 (12)	C4—C3—C8—N1	179.24 (11)
C8—C3—C4—C5	0.37 (19)	C2—C3—C8—N1	−0.12 (13)
C2—C3—C4—C5	179.55 (13)	O1—C1—N1—C8	178.40 (12)
C3—C4—C5—C6	−0.6 (2)	C2—C1—N1—C8	−1.72 (13)
C4—C5—C6—C7	0.3 (2)	C7—C8—N1—C1	−179.85 (12)
C5—C6—C7—F1	−178.89 (12)	C3—C8—N1—C1	1.25 (14)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1A···O3	0.88	1.89	2.7679 (14)	175
C6—H6A···O1^{i}	0.95	2.34	3.2870 (17)	175

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

Fig. 1

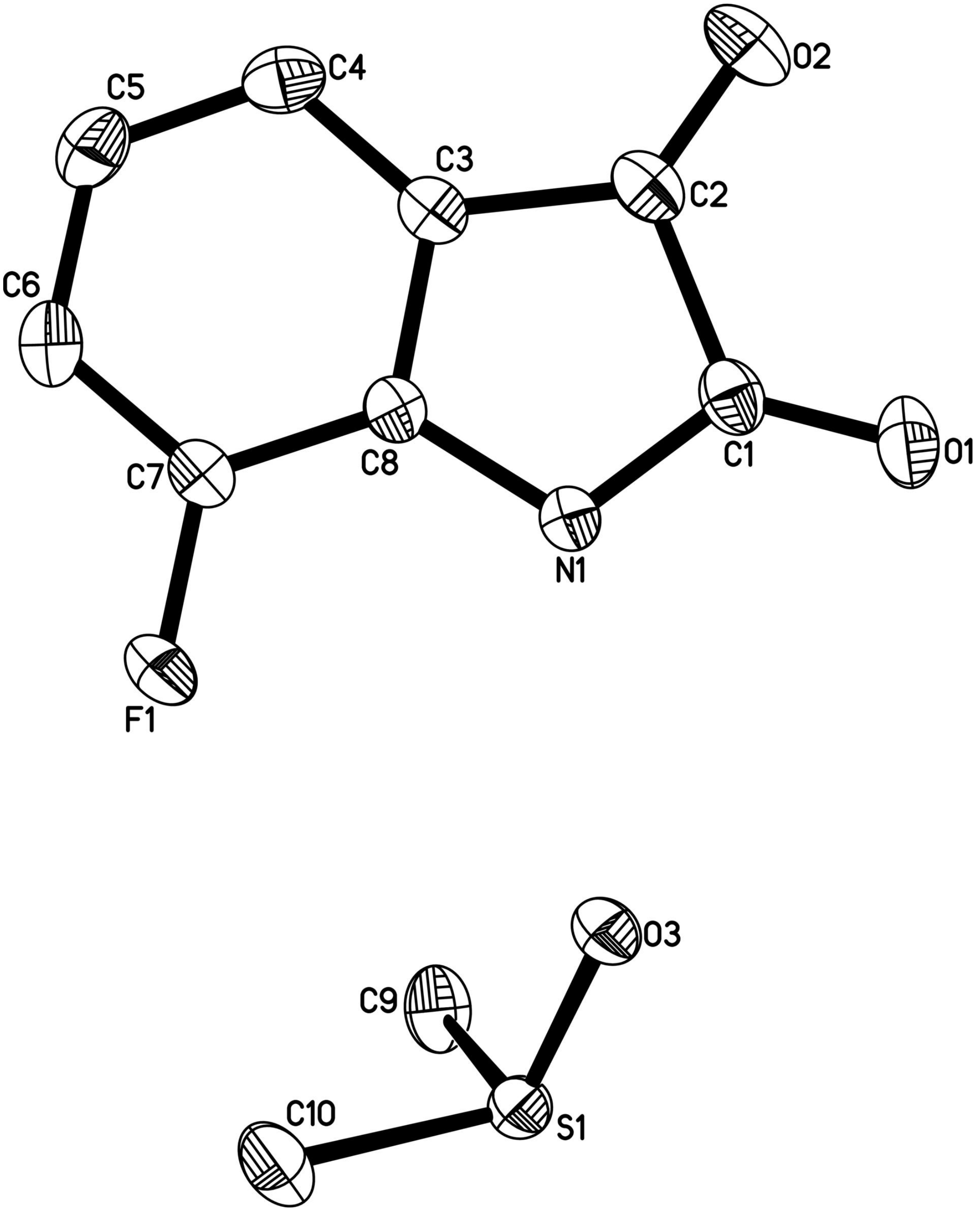

Fig. 2

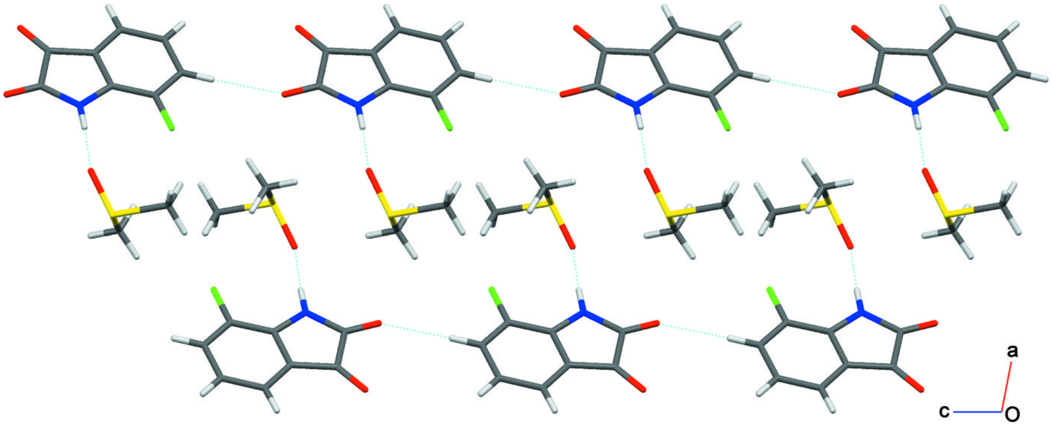

Fig. 3