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7-Fluoroisatin–1,4-dioxane (1/1)

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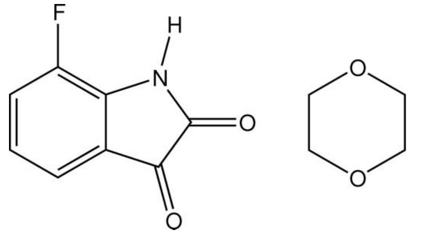
Key indicators: single-crystal X-ray study; T = 123 K; mean σ(C–C) = 0.002 Å; R factor = 0.039; wR factor = 0.103; data-to-parameter ratio = 13.0.

The title 1,4-dioxane solvate, $C_8H_4FNO_2 \cdot C_4H_8O_2$, was isolated during a manual crystallization screen on 7-fluoroisatin (7-fluoroindoline-2,3-dione). The 7-fluoroisatin molecule occupies a general position and each of the independent molecules of 1,4-dioxane is disposed about a centre of inversion, with half of each in the asymmetric unit. Hydrogen-bonded ribbons of 7-fluoroisatin are linked by 1,4-dioxane to form sheets parallel to $(30\overline{1})$. Whilst one solvent molecule has an active role in the sheet formation, the other simply fills the cavity formed within the sheet.

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 anticonvulsant activity in animals (Canas-Rodriguez & Leeming, 1972). The dimethyl sulfoxide solvates of 7-fluoroisatin (Mohamed *et al.*, 2007*a*) and 5-fluoroisatin (Mohamed *et al.*, 2007*b*) have also been prepared, as well as the oxindole derivative of 5-fluoroisatin (Mohamed *et al.*, 2007*c*).



Experimental

Crystal data

$C_{12}H_{12}FNO_4$
M_r = 253.23
Monoclinic, $P2_1/n$
a = 11.5274 (3) Å
b = 8.6508 (2) Å
c = 12.1738 (4) Å
β = 106.701 (3)°
V = 1162.78 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 123 (2) K
0.35 × 0.24 × 0.06 mm

Data collection

Oxford Diffraction Gemini diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2006)
$T_{\min}$ = 0.960, $T_{\max}$ = 0.993
12479 measured reflections
2740 independent reflections
2042 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.041

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.103
S = 1.04
2740 reflections
211 parameters
All H-atom parameters refined
$\Delta\rho_{\max}$ = 0.30 e Å^{-3}
$\Delta\rho_{\min}$ = −0.16 e Å^{-3}

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N1—H4$\cdots$O3	0.92 (2)	1.86 (2)	2.7727 (15)	175.3 (17)
C4—H1$\cdots$O2$^{\rm i}$	0.972 (17)	2.535 (17)	3.2262 (18)	128.1 (13)

Symmetry code: (i) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{3}{2}$.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED*; 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: TK2181).

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supplementary materials

Acta Cryst. (2007). E**63**, o3574   [**doi:10.1107/S1600536807035258**]

7-Fluoroisatin-1,4-dioxane (1/1)

K. Shankland, C. K. Leech, 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 (Mohamed *et al.*, 2007*a*-c).

The crystal structure of (I) is characterized by hydrogen-bonded ribbons of 7-fluoroisatin which are linked into sheets by 1,4-dioxane molecules. Whilst one dioxane molecule uses both oxygen atoms as hydrogen bond acceptors, the second dioxane molecule in the asymmetric unit does not appear to participate in any intermolecular interactions and simply fills the cavities formed within the sheet (Figure 2 and Table 1). The sheets lie parallel to (3 0 − 1) and are separated by 3.84 Å.

Experimental

Single crystals of the title compound were crystallized from a saturated 1,4-dioxane solution by slow evaporation at 278 K over the course of 3 weeks.

Refinement

All H atoms were refined freely so that the C—H distances were in the range 0.944 (18) to 1.006 (16) Å and N—H = 0.92 (2) Å.

Figures

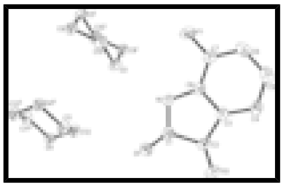

Fig. 1. The molecular structures of the components of (I) showing the atomic numbering scheme used. Displacement ellipsoids are drawn at the 50% probability level and hydrogen atoms have been omitted for clarity. Each 1,4-dioxane molecule is disposed about a centre of inversion. Symmetry codes: (i) = $1 - x, 1 - y, -z$ and (ii) = $1 - x, -y, -z$.

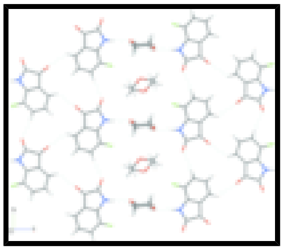

Fig. 2. Part of the sheet formed in (I) showing the hydrogen-bonding interactions as blue dotted lines. Colour code: C - dark grey, H - light grey, N - blue, O - red, F - green.

Fig. 3. Packing diagram showing the stacking of the sheets in (I). Hydrogen-bonding interactions are shown as blue dotted lines. Colour code: C - dark grey, H - light grey, N - blue, O - red, F - green.

7-fluoroindoline-2,3-dione–1,4-dioxane (1/1)

Crystal data

$C_{12}H_{12}FNO_4$

$M_r = 253.23$

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

$a = 11.5274$ (3) Å

$b = 8.6508$ (2) Å

$c = 12.1738$ (4) Å

$\beta = 106.701$ (3)°

$V = 1162.78$ (6) Å^3

$Z = 4$

$F_{000} = 528$

$D_x = 1.447$ Mg m^{-3}

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

Cell parameters from 5725 reflections

$\theta = 2.9$–28.6°

$\mu = 0.12$ mm^{-1}

$T = 123$ (2) K

Plate, yellow

0.35 × 0.24 × 0.06 mm

Data collection

Oxford Diffraction Gemini diffractometer

Radiation source: Enhance (Mo) X-ray Source

Monochromator: graphite

$T = 123(2)$ K

ω scans

Absorption correction: multi-scan
(CrysAlis RED; Oxford Diffraction, 2006)

$T_{\min} = 0.960$, $T_{\max} = 0.993$

12479 measured reflections

2740 independent reflections

2042 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.041$

$\theta_{\max} = 28.7$°

$\theta_{\min} = 2.9$°

$h = -15 \rightarrow 15$

$k = -11 \rightarrow 11$

$l = -16 \rightarrow 16$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.039$

$wR(F^2) = 0.103$

$S = 1.04$

2740 reflections

211 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: structure-invariant direct methods

Hydrogen site location: difference Fourier map

All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0555P)^2 + 0.117P]$
where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\max} = 0.30$ e Å^{-3}

$\Delta\rho_{\min} = -0.16$ e Å^{-3}

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 $F^2 > 2\sigma(F^2)$ 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	0.63568 (11)	0.30973 (16)	0.41226 (12)	0.0227 (3)
C2	0.67610 (11)	0.36091 (17)	0.54023 (12)	0.0241 (3)
C3	0.65915 (11)	0.52886 (17)	0.53806 (12)	0.0232 (3)
C4	0.67938 (13)	0.63897 (19)	0.62395 (13)	0.0288 (3)
H1	0.7103 (14)	0.606 (2)	0.7033 (15)	0.037 (5)*
C5	0.65384 (13)	0.79131 (19)	0.59349 (15)	0.0329 (4)
H2	0.6680 (14)	0.867 (2)	0.6539 (15)	0.038 (5)*
C6	0.60940 (13)	0.83359 (18)	0.47911 (15)	0.0318 (4)
H3	0.5924 (15)	0.937 (2)	0.4561 (15)	0.041 (5)*
C7	0.59034 (13)	0.72222 (17)	0.39484 (13)	0.0274 (3)
C8	0.61464 (11)	0.56981 (16)	0.42251 (12)	0.0215 (3)
N1	0.60153 (10)	0.44135 (13)	0.35030 (10)	0.0230 (3)
H4	0.5719 (16)	0.444 (2)	0.2720 (17)	0.048 (5)*
O1	0.63342 (9)	0.17947 (11)	0.37651 (9)	0.0302 (3)
O2	0.71073 (9)	0.27269 (13)	0.61973 (9)	0.0335 (3)
F1	0.54700 (9)	0.76186 (11)	0.28272 (8)	0.0414 (3)
C9	0.38445 (13)	0.47942 (19)	−0.07452 (13)	0.0272 (3)
H9A	0.3873 (14)	0.370 (2)	−0.0935 (13)	0.032 (4)*
H9B	0.3077 (14)	0.5267 (18)	−0.1169 (13)	0.029 (4)*
C10	0.40659 (12)	0.50121 (19)	0.05195 (12)	0.0277 (3)
H10A	0.3476 (14)	0.4418 (17)	0.0791 (13)	0.030 (4)*
H10B	0.4031 (13)	0.610 (2)	0.0727 (13)	0.030 (4)*
O3	0.52419 (8)	0.44245 (11)	0.11257 (8)	0.0262 (2)
C11	0.46557 (12)	0.05248 (19)	0.09413 (13)	0.0270 (3)
H11A	0.4396 (14)	−0.0409 (19)	0.1234 (13)	0.026 (4)*
H11B	0.4563 (13)	0.1403 (18)	0.1444 (13)	0.024 (4)*
C12	0.59628 (12)	0.03656 (18)	0.09507 (13)	0.0272 (3)
H12A	0.6456 (14)	0.0060 (19)	0.1745 (14)	0.031 (4)*
H12B	0.6237 (14)	0.135 (2)	0.0700 (14)	0.031 (4)*
O4	0.60965 (8)	−0.08326 (11)	0.01931 (8)	0.0256 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0195 (6)	0.0247 (7)	0.0234 (7)	−0.0004 (5)	0.0056 (5)	0.0013 (6)
C2	0.0198 (6)	0.0307 (8)	0.0220 (7)	−0.0005 (6)	0.0060 (5)	0.0043 (6)
C3	0.0188 (6)	0.0294 (8)	0.0217 (7)	−0.0007 (5)	0.0065 (5)	0.0007 (6)
C4	0.0257 (7)	0.0387 (9)	0.0233 (8)	−0.0037 (6)	0.0091 (6)	−0.0043 (7)
C5	0.0296 (8)	0.0351 (9)	0.0361 (9)	−0.0053 (6)	0.0126 (7)	−0.0122 (7)
C6	0.0286 (7)	0.0231 (8)	0.0465 (10)	−0.0007 (6)	0.0153 (7)	−0.0011 (7)
C7	0.0258 (7)	0.0293 (8)	0.0266 (8)	0.0013 (6)	0.0066 (6)	0.0053 (6)
C8	0.0181 (6)	0.0249 (7)	0.0216 (7)	−0.0014 (5)	0.0057 (5)	0.0011 (5)
N1	0.0248 (6)	0.0253 (6)	0.0172 (6)	0.0002 (5)	0.0030 (5)	0.0005 (5)
O1	0.0302 (5)	0.0247 (6)	0.0345 (6)	0.0004 (4)	0.0072 (5)	−0.0011 (5)
O2	0.0347 (6)	0.0387 (6)	0.0261 (6)	0.0048 (5)	0.0072 (5)	0.0127 (5)
F1	0.0563 (6)	0.0334 (5)	0.0314 (5)	0.0093 (4)	0.0080 (5)	0.0128 (4)
C9	0.0226 (7)	0.0316 (8)	0.0242 (7)	−0.0019 (6)	0.0017 (6)	0.0027 (6)
C10	0.0232 (7)	0.0348 (8)	0.0244 (8)	0.0010 (6)	0.0057 (6)	0.0020 (7)
O3	0.0246 (5)	0.0316 (6)	0.0196 (5)	−0.0005 (4)	0.0020 (4)	0.0041 (4)
C11	0.0236 (7)	0.0329 (8)	0.0223 (7)	0.0022 (6)	0.0033 (6)	0.0001 (6)
C12	0.0232 (7)	0.0306 (8)	0.0248 (8)	0.0019 (6)	0.0022 (6)	−0.0013 (6)
O4	0.0211 (5)	0.0277 (5)	0.0250 (5)	0.0045 (4)	0.0018 (4)	0.0015 (4)

Geometric parameters (Å, °)

N1—C1	1.3600 (18)	O3—C9$^{\mathrm{i}}$	1.4356 (17)
N1—C8	1.3979 (17)	O3—C10	1.4373 (16)
N1—H4	0.92 (2)	C9—O3$^{\mathrm{i}}$	1.4356 (17)
C1—O1	1.2056 (17)	C9—C10	1.498 (2)
C1—C2	1.5566 (19)	C9—H9A	0.979 (18)
C2—O2	1.2068 (16)	C9—H9B	0.976 (16)
C2—C3	1.465 (2)	C10—H10A	0.983 (16)
C3—C4	1.384 (2)	C10—H10B	0.979 (17)
C3—C8	1.3975 (18)	O4—C12	1.4250 (17)
C4—C5	1.378 (2)	O4—C11$^{\mathrm{ii}}$	1.4288 (17)
C4—H1	0.972 (17)	C11—O4$^{\mathrm{ii}}$	1.4288 (17)
C5—C6	1.388 (2)	C11—C12	1.5097 (19)
C5—H2	0.960 (18)	C11—H11A	0.965 (16)
C6—C7	1.378 (2)	C11—H11B	1.001 (16)
C6—H3	0.944 (18)	C12—H12A	1.006 (16)
C7—F1	1.3558 (17)	C12—H12B	0.990 (17)
C7—C8	1.370 (2)		
C1—N1—C8	110.80 (12)	C9$^{\mathrm{i}}$—O3—C10	109.81 (11)
C1—N1—H4	123.9 (12)	O3$^{\mathrm{i}}$—C9—C10	110.37 (11)
C8—N1—H4	125.3 (12)	O3$^{\mathrm{i}}$—C9—H9A	107.6 (9)
O1—C1—N1	127.55 (13)	C10—C9—H9A	110.9 (9)
O1—C1—C2	126.61 (13)	O3$^{\mathrm{i}}$—C9—H9B	105.6 (9)

N1—C1—C2	105.82 (11)
O2—C2—C3	130.79 (14)
O2—C2—C1	123.89 (13)
C3—C2—C1	105.31 (11)
C4—C3—C8	121.30 (14)
C4—C3—C2	132.56 (13)
C8—C3—C2	106.14 (12)
C5—C4—C3	118.59 (15)
C5—C4—H1	122.6 (10)
C3—C4—H1	118.8 (10)
C4—C5—C6	120.72 (15)
C4—C5—H2	117.8 (10)
C6—C5—H2	121.5 (10)
C7—C6—C5	119.78 (15)
C7—C6—H3	118.0 (11)
C5—C6—H3	122.2 (11)
F1—C7—C8	118.76 (13)
F1—C7—C6	120.42 (13)
C8—C7—C6	120.82 (14)
C7—C8—C3	118.78 (13)
C7—C8—N1	129.28 (13)
C3—C8—N1	111.93 (12)
C10—C9—H9B	110.5 (9)
H9A—C9—H9B	111.6 (13)
O3—C10—C9	109.89 (12)
O3—C10—H10A	106.5 (9)
C9—C10—H10A	110.5 (9)
O3—C10—H10B	108.1 (9)
C9—C10—H10B	112.2 (9)
H10A—C10—H10B	109.5 (13)
C12—O4—C11^{ii}	109.97 (11)
O4^{ii}—C11—C12	110.58 (12)
O4^{ii}—C11—H11A	110.3 (9)
C12—C11—H11A	109.6 (9)
O4^{ii}—C11—H11B	108.0 (8)
C12—C11—H11B	110.3 (8)
H11A—C11—H11B	108.1 (13)
O4—C12—C11	110.78 (12)
O4—C12—H12A	107.1 (9)
C11—C12—H12A	108.6 (9)
O4—C12—H12B	109.0 (10)
C11—C12—H12B	109.0 (9)
H12A—C12—H12B	112.4 (13)

C8—N1—C1—O1	178.58 (13)
C8—N1—C1—C2	−0.21 (14)
O1—C1—C2—O2	0.1 (2)
N1—C1—C2—O2	178.91 (13)
O1—C1—C2—C3	−178.55 (13)
N1—C1—C2—C3	0.25 (13)
O2—C2—C3—C4	1.7 (3)
C1—C2—C3—C4	−179.74 (14)
O2—C2—C3—C8	−178.72 (14)
C1—C2—C3—C8	−0.19 (13)
C8—C3—C4—C5	0.5 (2)
C2—C3—C4—C5	179.97 (14)
C3—C4—C5—C6	−0.4 (2)
C4—C5—C6—C7	0.1 (2)
C5—C6—C7—F1	−179.98 (13)
C5—C6—C7—C8	0.1 (2)
F1—C7—C8—C3	−179.91 (11)
C6—C7—C8—C3	0.1 (2)
F1—C7—C8—N1	0.1 (2)
C6—C7—C8—N1	−179.96 (13)
C4—C3—C8—C7	−0.33 (19)
C2—C3—C8—C7	−179.94 (12)
C4—C3—C8—N1	179.69 (12)
C2—C3—C8—N1	0.07 (14)
C1—N1—C8—C7	−179.89 (14)
C1—N1—C8—C3	0.09 (15)
C9^{i}—O3—C10—C9	58.41 (17)
O3^{i}—C9—C10—O3	−58.75 (17)
C11^{ii}—O4—C12—C11	57.46 (17)
O4^{ii}—C11—C12—O4	−57.81 (17)

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H4···O3	0.92 (2)	1.86 (2)	2.7727 (15)	175.3 (17)
C4—H1···O2^{iii}	0.972 (17)	2.535 (17)	3.2262 (18)	128.1 (13)

Symmetry codes: (iii) −*x*+3/2, *y*+1/2, −*z*+3/2.

Fig. 1

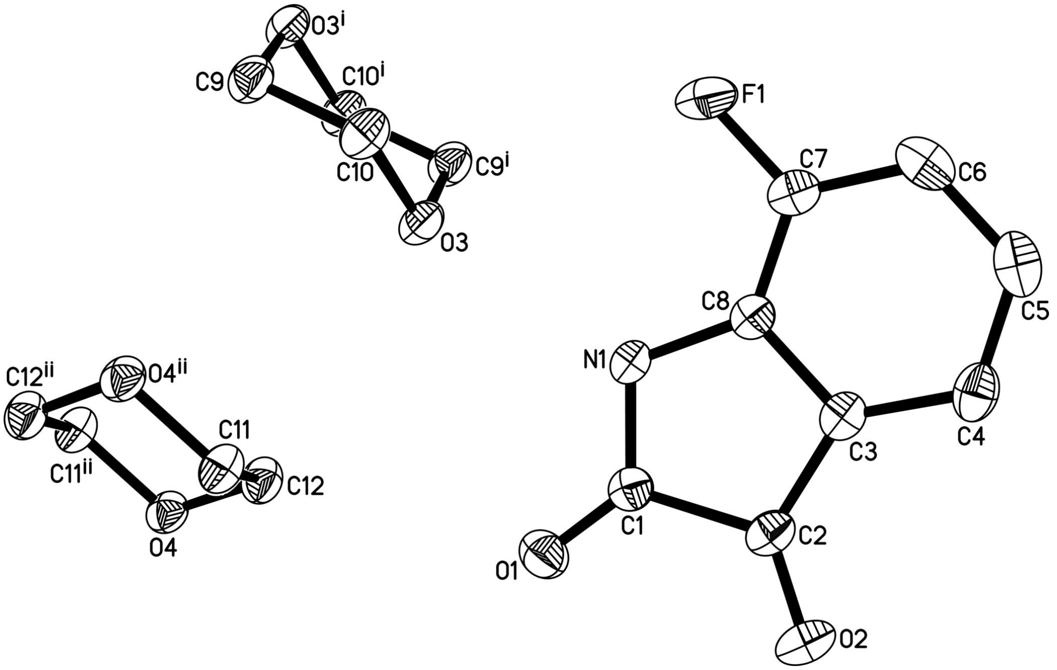
O3i
C9
C10i
C10
C9i
O3
F1
C7
C6
C5
C8
N1
C3
C4
C12ii
O4ii
C11
C11ii
C12
O4
C1
C2
O1
O2

Fig. 2

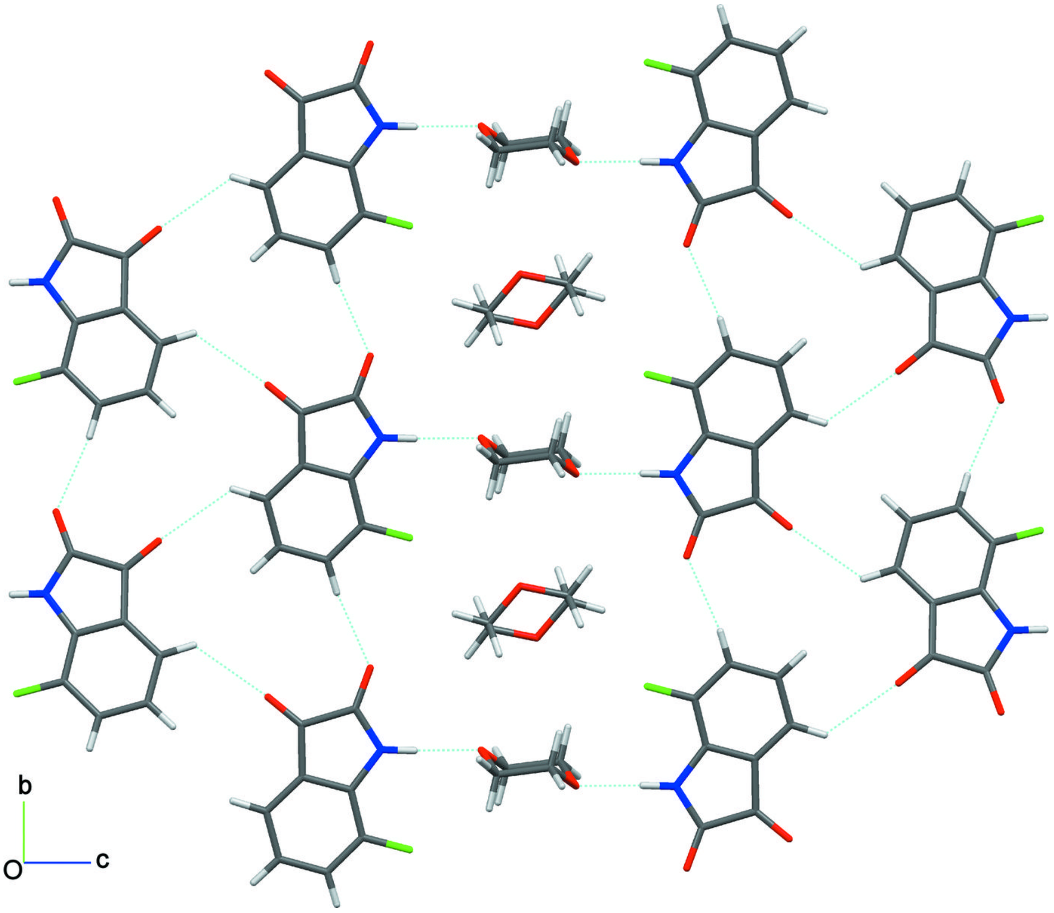

Fig. 3