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5-Fluoro-3-hydroxy-3-(nitromethyl)-1*H*-indol-2(3*H*)-one

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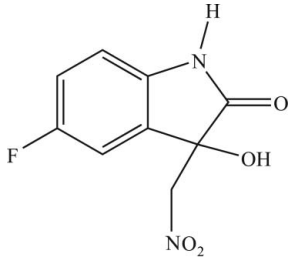
Key indicators: single-crystal X-ray study; T = 150 K; mean σ(C–C) = 0.002 Å; R factor = 0.038; wR factor = 0.101; data-to-parameter ratio = 12.1.

The title compound, $C_9H_7FN_2O_4$, was isolated during a manual crystallization screen on 5-fluoroisatin (5-fluoroindoline-2,3-dione). Hydrogen-bonded ribbons of the oxindole are formed through pairs of N—H···O and O—H···O interactions. These ribbons then pack parallel to (092) and (09$\bar{2}$) such that a herring-bone motif is established.

Related literature

5-Fluoroisatin forms sheets through double N—H···O hydrogen bonds and C—H···O interactions (Naumov *et al.*, 2000). The title oxindole derivative resulted from a side reaction between 5-fluoroisatin and nitromethane. The synthesis of oxindole derivatives of isatins from nitromethane has been previously reported (Conn & Lindwall, 1936). The 1,4-dioxane (Shankland *et al.*, 2007) and DMSO (Mohamed *et al.*, 2007*a*) solvates of 7-fluoroisatin have been prepared, as well as the DMSO solvate of 5-fluoroisatin (Mohamed *et al.*, 2007*b*).



Experimental

Crystal data

$C_9H_7FN_2O_4$
M_r = 226.17
Monoclinic, $P2_1/c$
a = 7.9400 (8) Å
b = 15.7867 (16) Å
c = 7.2980 (8) Å
β = 105.536 (2)°
V = 881.35 (16) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.15 mm^{-1}
T = 150 (2) K
0.45 × 0.30 × 0.25 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.879, T_{max} = 1.000
7682 measured reflections
2170 independent reflections
1966 reflections with $I > 2\sigma(I)$
R_{int} = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.101
S = 1.05
2087 reflections
173 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H4···O1^{i}	0.802 (19)	2.214 (19)	2.9700 (13)	157.4 (17)
O2—H5···O1^{ii}	0.83 (2)	1.98 (2)	2.7799 (13)	161 (2)

Symmetry codes: (i) $-x, -y+1, -z+2$; (ii) $-x+1, -y+1, -z+2$.

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: TK2184).

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supplementary materials

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5-Fluoro-3-hydroxy-3-(nitromethyl)-1*H*-indol-2(3*H*)-one

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Comment

The title compound, (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).

The oxindole derivative, (I), resulted from the side reaction of 5-fluoroisatin with the recrystallization solvent, nitromethane. The synthesis of oxindole derivatives of isatins from nitromethane has been previously reported (Conn & Lindwall, 1936).

Hydrogen-bonded ribbons of the oxindole are formed through pairs of N—H···O and O—H···O interactions (Figure 2 and Table 1). These ribbons then pack parallel to (0 9 2) and (0 9 − 2) such that a herringbone motif is established (Figure 3).

Experimental

Single crystals of the title compound were crystallized by slow solvent evaporation of a saturated solution of 5-fluoroisatin in nitromethane.

Refinement

All H atoms were refined freely so that the C—H distances were in the range 0.939 (16) to 0.981 (16) Å, N—H = 0.802 (19) Å and O—H = 0.83 (2) Å. The (83) reflections present but, which should be systematically absent for the space group P21/c have been omitted from the refinement.

Figures

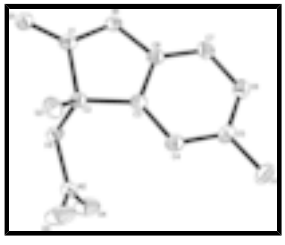

Fig. 1. The asymmetric unit of (I) showing the numbering scheme used. Displacement ellipsoids are drawn at the 50% probability level and hydrogen atoms have been omitted for clarity.

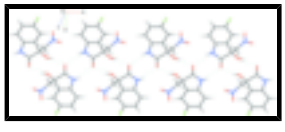

Fig. 2. Illustration of the ribbon motif in (I) showing the hydrogen-bonding interactions as blue dotted lines. C - dark grey, H - light grey, N - blue, O - red, F - green.

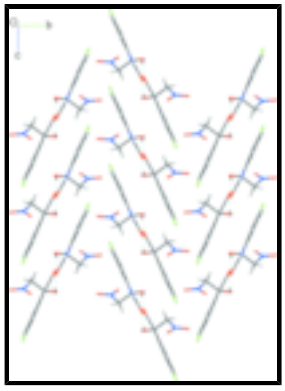

Fig. 3. Packing diagram for (I) showing the stacking of the ribbons. C - dark grey, H - light grey, N - blue, O - red, F - green.

5-Fluoro-3-hydroxy-3-(nitromethyl)-1*H*-indol-2(3*H*)-one

Crystal data

$C_9H_7FN_2O_4$	F_{000} = 464
M_r = 226.17	D_x = 1.704 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 5148 reflections
a = 7.9400 (8) Å	θ = 2.6–28.2º
b = 15.7867 (16) Å	μ = 0.15 mm^{-1}
c = 7.2980 (8) Å	T = 150 (2) K
β = 105.536 (2)º	Block, colourless
V = 881.35 (16) Å^3	0.45 × 0.30 × 0.25 mm
Z = 4	

Data collection

Bruker SMART APEX diffractometer	2170 independent reflections
Radiation source: fine-focus sealed tube	1966 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.029
T = 150(2) K	θ_{max} = 28.3º
ω rotation with narrow frames scans	θ_{min} = 2.7º
Absorption correction: multi-scan (SADABS; Bruker, 2001)	h = −10→10
T_{min} = 0.879, T_{max} = 1.000	k = −20→20
7682 measured reflections	l = −9→9

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)]$ = 0.038	All H-atom parameters refined
$wR(F^2)$ = 0.101	$w = 1/[\sigma^2(F_o^2) + (0.056P)^2 + 0.3897P]$

	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{max} < 0.001$
2087 reflections	$\Delta\rho_{max} = 0.40$ e Å^{-3}
173 parameters	$\Delta\rho_{min} = -0.29$ 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 $F^2 > 2\text{sigma}(F^2)$ 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	0.18906 (15)	0.46235 (7)	0.87480 (17)	0.0181 (2)
C2	0.32534 (14)	0.43472 (7)	0.76650 (16)	0.0172 (2)
C3	0.20654 (15)	0.40255 (7)	0.58143 (16)	0.0173 (2)
C4	0.24608 (15)	0.37339 (8)	0.41905 (17)	0.0197 (2)
H1	0.365 (2)	0.3737 (10)	0.402 (2)	0.021 (4)*
C5	0.10726 (16)	0.34140 (8)	0.27745 (17)	0.0204 (3)
C6	−0.06236 (16)	0.33844 (8)	0.29063 (17)	0.0215 (3)
H2	−0.151 (2)	0.3143 (11)	0.192 (2)	0.026 (4)*
C7	−0.10177 (15)	0.37019 (8)	0.45338 (18)	0.0205 (3)
H3	−0.217 (2)	0.3701 (11)	0.465 (3)	0.027 (4)*
C8	0.03515 (15)	0.40167 (7)	0.59618 (16)	0.0176 (2)
C9	0.42671 (16)	0.36201 (8)	0.88774 (17)	0.0190 (2)
H6	0.351 (2)	0.3207 (10)	0.913 (2)	0.020 (4)*
H7	0.504 (2)	0.3841 (10)	1.004 (2)	0.024 (4)*
N1	0.02989 (13)	0.43790 (7)	0.77204 (14)	0.0191 (2)
H4	−0.058 (2)	0.4466 (12)	0.803 (3)	0.031 (4)*
N2	0.54238 (13)	0.31607 (7)	0.78830 (14)	0.0196 (2)
O1	0.22624 (11)	0.49891 (6)	1.02900 (13)	0.0225 (2)
O2	0.43033 (12)	0.50392 (6)	0.74659 (13)	0.0223 (2)
H5	0.534 (3)	0.4928 (14)	0.799 (3)	0.052 (6)*
O3	0.64238 (16)	0.35790 (7)	0.72435 (19)	0.0416 (3)
O4	0.53062 (14)	0.23967 (6)	0.77521 (17)	0.0350 (3)
F1	0.14101 (10)	0.31091 (5)	0.11608 (11)	0.0274 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0173 (5)	0.0194 (5)	0.0192 (5)	0.0016 (4)	0.0076 (4)	0.0005 (4)
C2	0.0158 (5)	0.0202 (5)	0.0170 (5)	−0.0009 (4)	0.0068 (4)	−0.0017 (4)
C3	0.0163 (5)	0.0188 (5)	0.0174 (5)	−0.0010 (4)	0.0055 (4)	−0.0001 (4)
C4	0.0188 (5)	0.0223 (6)	0.0197 (6)	−0.0002 (4)	0.0081 (4)	−0.0001 (4)
C5	0.0254 (6)	0.0215 (6)	0.0163 (5)	−0.0004 (4)	0.0089 (4)	−0.0017 (4)
C6	0.0212 (6)	0.0232 (6)	0.0189 (6)	−0.0029 (4)	0.0035 (4)	−0.0013 (4)
C7	0.0165 (5)	0.0242 (6)	0.0213 (6)	−0.0014 (4)	0.0059 (4)	0.0006 (4)
C8	0.0182 (5)	0.0186 (5)	0.0175 (5)	0.0007 (4)	0.0074 (4)	0.0007 (4)
C9	0.0184 (5)	0.0224 (6)	0.0184 (5)	0.0015 (4)	0.0084 (4)	−0.0012 (4)
N1	0.0148 (5)	0.0255 (5)	0.0188 (5)	0.0012 (4)	0.0075 (4)	−0.0022 (4)
N2	0.0157 (5)	0.0237 (5)	0.0192 (5)	0.0006 (4)	0.0046 (4)	−0.0026 (4)
O1	0.0197 (4)	0.0291 (5)	0.0197 (4)	0.0009 (3)	0.0068 (3)	−0.0057 (3)
O2	0.0175 (4)	0.0233 (5)	0.0257 (5)	−0.0046 (3)	0.0052 (4)	0.0014 (3)
O3	0.0421 (6)	0.0352 (6)	0.0622 (8)	−0.0112 (5)	0.0393 (6)	−0.0132 (5)
O4	0.0381 (6)	0.0212 (5)	0.0519 (7)	0.0048 (4)	0.0226 (5)	−0.0009 (4)
F1	0.0308 (4)	0.0348 (4)	0.0189 (4)	−0.0034 (3)	0.0109 (3)	−0.0081 (3)

Geometric parameters (Å, °)

C1—O1	1.2283 (15)	C6—C7	1.3996 (17)
C1—N1	1.3420 (15)	C6—H2	0.943 (17)
C1—C2	1.5634 (15)	C7—C8	1.3813 (17)
C2—O2	1.4052 (14)	C7—H3	0.940 (18)
C2—C3	1.5131 (15)	C8—N1	1.4159 (14)
C2—C9	1.5383 (16)	C9—N2	1.5011 (15)
C3—C4	1.3835 (16)	C9—H6	0.939 (16)
C3—C8	1.3936 (16)	C9—H7	0.968 (16)
C4—C5	1.3885 (17)	N1—H4	0.802 (19)
C4—H1	0.981 (16)	N2—O4	1.2114 (15)
C5—F1	1.3636 (13)	N2—O3	1.2177 (15)
C5—C6	1.3764 (18)	O2—H5	0.83 (2)
O1—C1—N1	127.33 (11)	C7—C6—H2	120.0 (10)
O1—C1—C2	124.42 (10)	C8—C7—C6	117.35 (11)
N1—C1—C2	108.24 (10)	C8—C7—H3	121.3 (11)
O2—C2—C3	114.45 (9)	C6—C7—H3	121.4 (11)
O2—C2—C9	113.96 (9)	C7—C8—C3	122.19 (11)
C3—C2—C9	111.60 (10)	C7—C8—N1	128.38 (11)
O2—C2—C1	110.21 (9)	C3—C8—N1	109.42 (10)
C3—C2—C1	101.22 (9)	C1—N1—C8	111.76 (10)
C9—C2—C1	104.11 (9)	C1—N1—H4	123.7 (13)
C4—C3—C8	120.88 (11)	C8—N1—H4	124.4 (13)
C4—C3—C2	130.24 (10)	C2—O2—H5	109.3 (16)
C8—C3—C2	108.82 (10)	N2—C9—C2	112.09 (9)
C3—C4—C5	116.20 (11)	N2—C9—H6	105.5 (10)

C3—C4—H1	123.4 (9)	C2—C9—H6	111.6 (10)
C5—C4—H1	120.3 (9)	N2—C9—H7	105.5 (10)
F1—C5—C6	118.08 (11)	C2—C9—H7	110.3 (10)
F1—C5—C4	118.08 (11)	H6—C9—H7	111.7 (14)
C6—C5—C4	123.84 (11)	O4—N2—O3	123.65 (11)
C5—C6—C7	119.50 (11)	O4—N2—C9	118.35 (10)
C5—C6—H2	120.5 (10)	O3—N2—C9	118.00 (11)
O1—C1—C2—O2	−52.66 (15)	C4—C5—C6—C7	1.0 (2)
N1—C1—C2—O2	128.41 (11)	C5—C6—C7—C8	−1.38 (18)
O1—C1—C2—C3	−174.17 (11)	C6—C7—C8—C3	0.16 (18)
N1—C1—C2—C3	6.90 (12)	C6—C7—C8—N1	179.16 (11)
O1—C1—C2—C9	69.93 (14)	C4—C3—C8—C7	1.57 (18)
N1—C1—C2—C9	−109.00 (11)	C2—C3—C8—C7	−175.80 (11)
O2—C2—C3—C4	57.41 (17)	C4—C3—C8—N1	−177.59 (11)
C9—C2—C3—C4	−73.87 (15)	C2—C3—C8—N1	5.04 (13)
C1—C2—C3—C4	175.90 (12)	O1—C1—N1—C8	176.70 (12)
O2—C2—C3—C8	−125.55 (11)	C2—C1—N1—C8	−4.41 (13)
C9—C2—C3—C8	103.17 (11)	C7—C8—N1—C1	−179.39 (12)
C1—C2—C3—C8	−7.06 (12)	C3—C8—N1—C1	−0.29 (14)
C8—C3—C4—C5	−1.95 (17)	O2—C2—C9—N2	−70.42 (12)
C2—C3—C4—C5	174.79 (11)	C3—C2—C9—N2	61.11 (12)
C3—C4—C5—F1	−179.11 (10)	C1—C2—C9—N2	169.48 (9)
C3—C4—C5—C6	0.71 (19)	C2—C9—N2—O4	−127.94 (12)
F1—C5—C6—C7	−179.20 (11)	C2—C9—N2—O3	51.69 (15)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H4···O1^{i}	0.802 (19)	2.214 (19)	2.9700 (13)	157.4 (17)
O2—H5···O1^{ii}	0.83 (2)	1.98 (2)	2.7799 (13)	161 (2)

Symmetry codes: (i) −*x*, −*y*+1, −*z*+2; (ii) −*x*+1, −*y*+1, −*z*+2.

Fig. 1

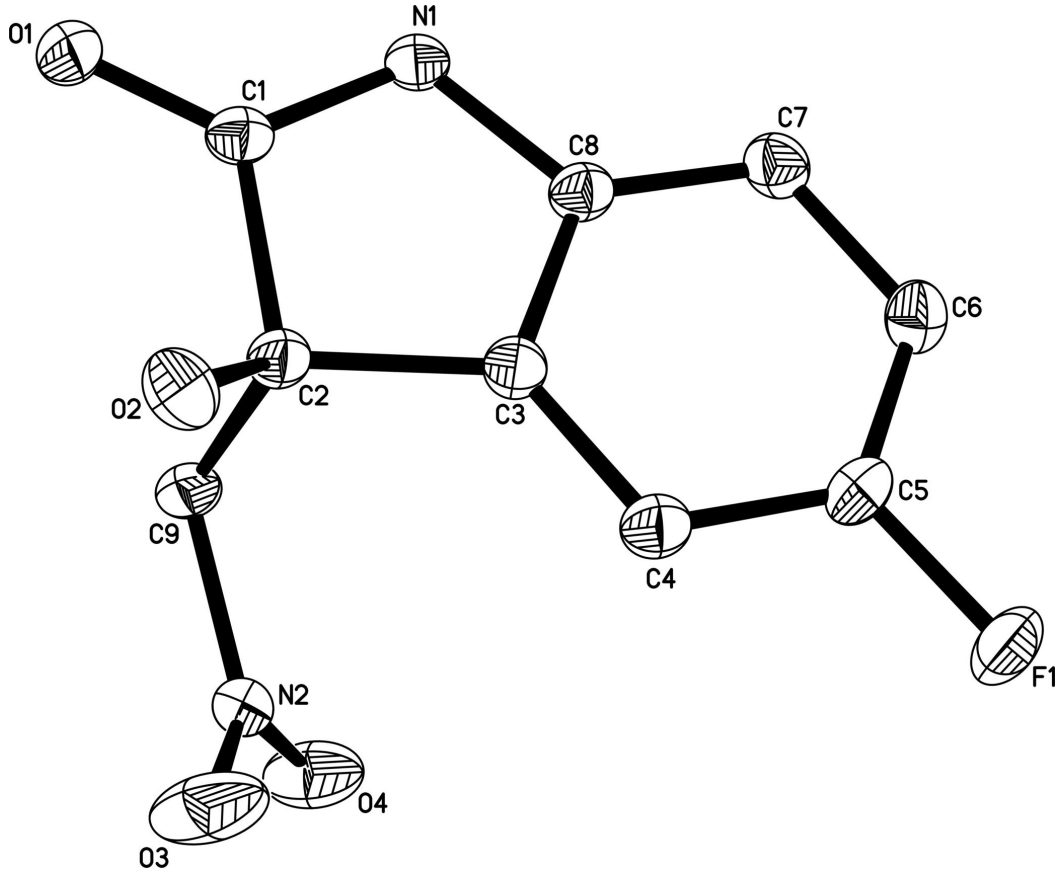

Fig. 2

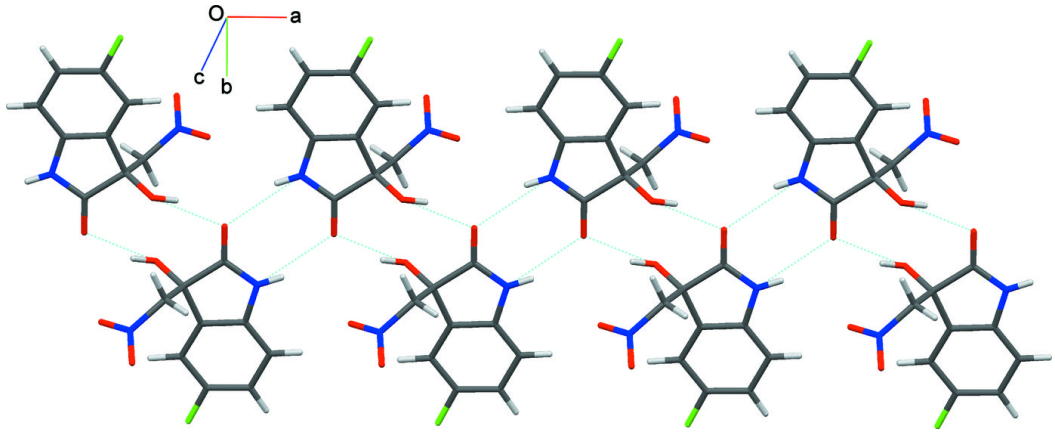

Fig. 3