Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

5-Fluoro-1*H*-indole-3-carboxylic acid

Wen-Jun Lu, Zhi-Hong Zou* and Yang-Hui Luo

Ordered Matter Science Research Center, College of Chemistry and Chemical Engineering, Southeast University, Nanjing 210096, People's Republic of China
Correspondence e-mail: seuzzh@sina.com

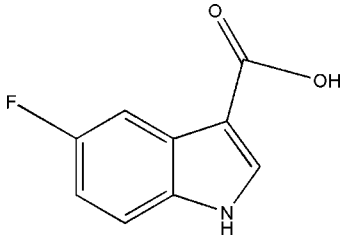
Received 2 December 2011; accepted 12 December 2011

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.054; wR factor = 0.137; data-to-parameter ratio = 14.5.

In the title compound, $C_9H_6FNO_2$, the carboxyl group is twisted slightly away from the indole-ring plane [dihedral angle = 7.39 (10)°]. In the crystal, carboxyl inversion dimers linked by pairs of O—H···O hydrogen bonds generate $R_2^2(8)$ loops and N—H···O hydrogen bonds connect the dimers into $(10\bar{1})$ sheets.

Related literature

For background to indoles as pharmaceuticals, see: Lang *et al.* (2011).



Experimental

Crystal data

$C_9H_6FNO_2$
M_r = 179.15
Monoclinic, $P2_1/n$
a = 4.4176 (9) Å
b = 11.073 (2) Å
c = 16.014 (3) Å
β = 96.63 (3)°
V = 778.1 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 293 K
0.30 × 0.20 × 0.10 mm

Data collection

Rigaku SCXmini CCD diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.982, T_{max} = 0.993
7874 measured reflections
1788 independent reflections
1153 reflections with $I > 2\sigma(I)$
R_{int} = 0.068

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.137
S = 1.02
1788 reflections
123 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2^{i}	0.82	1.86	2.669 (2)	171
N1—H1B···O2^{ii}	0.89 (3)	2.18 (3)	3.026 (2)	159 (2)

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

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2005); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6548).

References

Brandenburg, K. & Putz, H. (2005). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Lang, L., Wu, J.-L., Shi, L.-J., Xia, C.-G. & Li, F.-W. (2011). *Chem. Commun.* **47**, 12553–12555.
Rigaku. (2005). *CrystalClear*. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2012). E**68**, o187 [doi:10.1107/S1600536811053426]

5-Fluoro-1*H*-indole-3-carboxylic acid

Wen-Jun Lu, Zhi-Hong Zou and Yang-Hui Luo

S1. Experimental

A commercial sample of the title compound was obtained. Colourlss prisms were obtained by slow evaporation of a methanol solution over a period of seven days.

S2. Refinement

All H atoms attached to C atoms and O atoms were fixed geometrically and treated as riding with C—H = 0.93 Å (CH), N—H = 0.86 Å and O—H = 0.86 Å with U_{iso}(H) = 1.2U_{eq}(CH), U_{iso}(H) = 1.3U_{eq}(N) and U_{iso}(H) = 1.5U_{eq}(O).

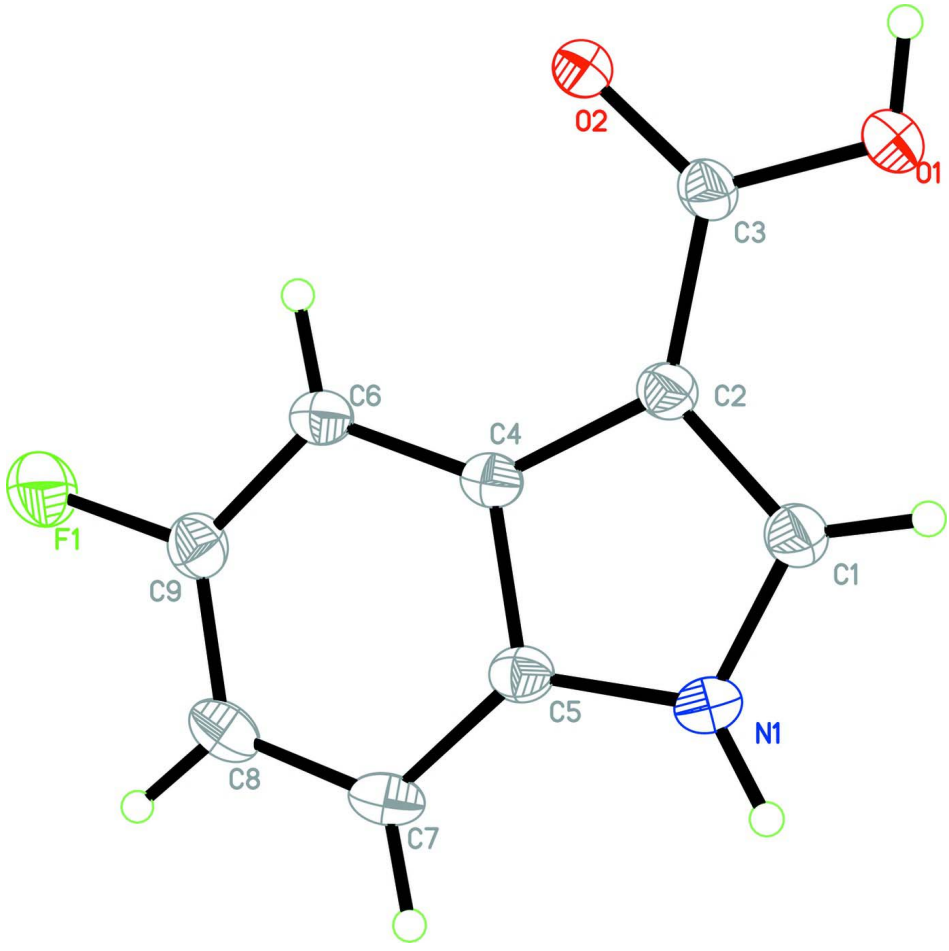


Figure 1

The molecular structure of the title compound with displacement ellipsoids drawn at the 30% probability level.

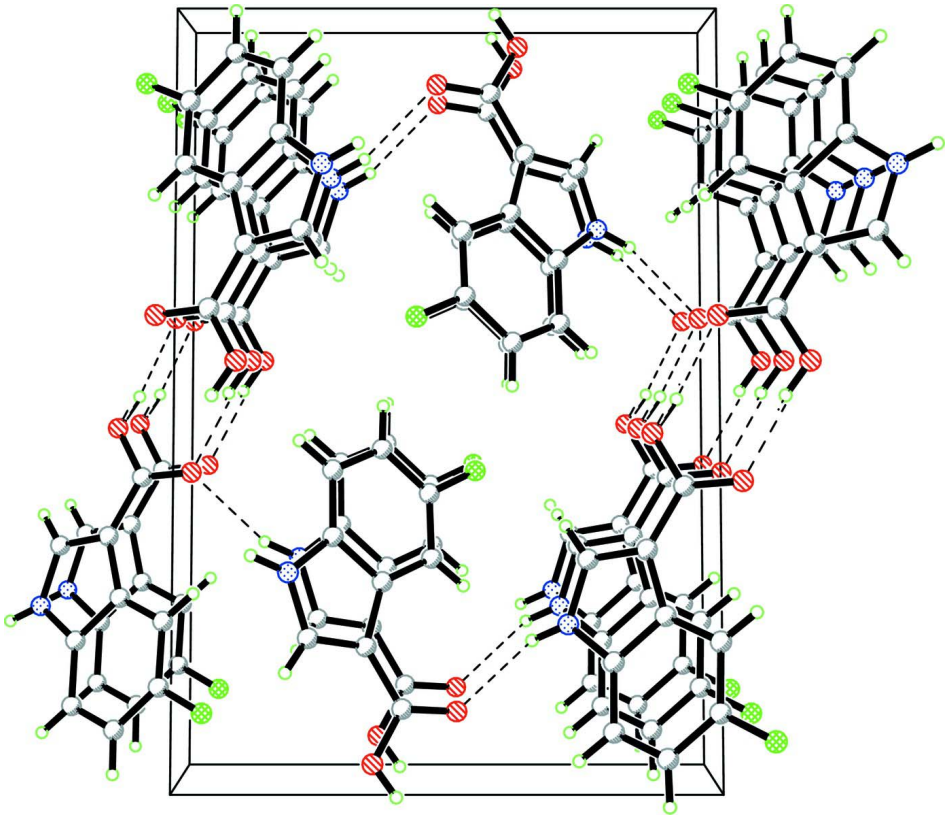


Figure 2

A packing view down the *a* axis showing hydrogen bonds as dashed lines.

5-Fluoro-1*H*-indole-3-carboxylic acid

Crystal data

$C_9H_6FNO_2$
$M_r = 179.15$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 4.4176\ (9)$ Å
$b = 11.073\ (2)$ Å
$c = 16.014\ (3)$ Å
$\beta = 96.63\ (3)°$
$V = 778.1\ (3)$ Å^3
$Z = 4$
$F(000) = 368$
$D_x = 1.529$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1954 reflections
$\theta = 3.2–25.0°$
$\mu = 0.13$ mm^{-1}
$T = 293$ K
Prism, colourless
0.30 × 0.20 × 0.10 mm

Data collection

Rigaku SCXmini CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 13.6612 pixels mm^{-1}
CCD profile–fitting scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
$T_{min} = 0.982$, $T_{max} = 0.993$
7874 measured reflections
1788 independent reflections
1153 reflections with $I > 2\sigma(I)$
$R_{int} = 0.068$

θ_{max} = 27.5°, θ_{min} = 3.2°
h = −5→5
k = −14→14
l = −20→20

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.137
S = 1.02
1788 reflections
123 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0575P)^2 + 0.142P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 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.

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 $F^2 > \sigma(F^2)$ 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}
O2	1.0621 (4)	0.51786 (13)	0.89866 (9)	0.0431 (4)
F1	1.4484 (3)	0.55381 (14)	0.59940 (9)	0.0624 (5)
N1	0.7010 (4)	0.21726 (17)	0.71693 (12)	0.0388 (5)
C4	1.0013 (4)	0.38262 (17)	0.73456 (12)	0.0295 (5)
C7	0.9558 (5)	0.2922 (2)	0.59302 (14)	0.0410 (6)
H7	0.8767	0.2328	0.5556	0.049*
O1	0.8085 (4)	0.36747 (14)	0.95496 (9)	0.0544 (5)
H1	0.8432	0.4097	0.9970	0.082*
C5	0.8845 (5)	0.29428 (17)	0.67573 (14)	0.0322 (5)
C3	0.9254 (5)	0.41935 (19)	0.89110 (13)	0.0341 (5)
C6	1.1989 (4)	0.47175 (18)	0.70871 (13)	0.0333 (5)
H6	1.2837	0.5308	0.7456	0.040*
C2	0.8776 (5)	0.35426 (18)	0.81256 (13)	0.0320 (5)
C1	0.6979 (5)	0.25319 (19)	0.79748 (14)	0.0367 (5)
H1A	0.5906	0.2154	0.8368	0.044*
C9	1.2604 (5)	0.4674 (2)	0.62638 (14)	0.0387 (5)
C8	1.1466 (5)	0.3805 (2)	0.56824 (14)	0.0441 (6)
H8	1.1984	0.3819	0.5136	0.053*
H1B	0.599 (5)	0.154 (2)	0.6945 (16)	0.052 (7)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O2	0.0677 (11)	0.0344 (9)	0.0267 (8)	−0.0101 (8)	0.0033 (8)	−0.0006 (6)
F1	0.0725 (10)	0.0703 (10)	0.0478 (9)	−0.0232 (8)	0.0220 (8)	0.0004 (7)
N1	0.0464 (11)	0.0336 (10)	0.0360 (11)	−0.0070 (9)	0.0027 (9)	−0.0069 (8)
C4	0.0299 (10)	0.0297 (11)	0.0280 (11)	0.0059 (8)	−0.0011 (9)	−0.0021 (9)
C7	0.0449 (13)	0.0454 (14)	0.0320 (13)	0.0019 (11)	0.0012 (10)	−0.0135 (10)
O1	0.0903 (14)	0.0477 (10)	0.0262 (9)	−0.0222 (9)	0.0116 (9)	−0.0019 (7)
C5	0.0328 (11)	0.0304 (11)	0.0326 (12)	0.0038 (9)	0.0005 (9)	−0.0034 (9)
C3	0.0436 (12)	0.0324 (11)	0.0253 (11)	0.0029 (10)	0.0004 (9)	0.0039 (9)
C6	0.0339 (11)	0.0332 (11)	0.0318 (12)	0.0017 (9)	−0.0002 (9)	−0.0039 (9)
C2	0.0383 (11)	0.0298 (11)	0.0270 (11)	0.0016 (9)	0.0006 (9)	−0.0002 (9)
C1	0.0449 (12)	0.0331 (11)	0.0322 (12)	0.0008 (10)	0.0046 (10)	0.0006 (9)
C9	0.0378 (12)	0.0425 (13)	0.0372 (13)	−0.0015 (10)	0.0099 (10)	0.0018 (10)
C8	0.0489 (14)	0.0565 (15)	0.0281 (12)	0.0038 (12)	0.0092 (11)	−0.0048 (11)

Geometric parameters (Å, º)

O2—C3	1.246 (2)	C7—H7	0.9300
F1—C9	1.369 (2)	O1—C3	1.328 (2)
N1—C1	1.351 (3)	O1—H1	0.8200
N1—C5	1.394 (3)	C3—C2	1.444 (3)
N1—H1B	0.89 (3)	C6—C9	1.378 (3)
C4—C5	1.414 (3)	C6—H6	0.9300
C4—C6	1.411 (3)	C2—C1	1.377 (3)
C4—C2	1.454 (3)	C1—H1A	0.9300
C7—C8	1.378 (3)	C9—C8	1.392 (3)
C7—C5	1.397 (3)	C8—H8	0.9300
C1—N1—C5	110.02 (18)	C9—C6—C4	116.95 (19)
C1—N1—H1B	123.9 (16)	C9—C6—H6	121.5
C5—N1—H1B	126.1 (16)	C4—C6—H6	121.5
C5—C4—C6	118.62 (19)	C1—C2—C3	125.57 (19)
C5—C4—C2	106.17 (18)	C1—C2—C4	106.92 (18)
C6—C4—C2	135.20 (19)	C3—C2—C4	127.49 (19)
C8—C7—C5	118.3 (2)	N1—C1—C2	109.7 (2)
C8—C7—H7	120.9	N1—C1—H1A	125.1
C5—C7—H7	120.9	C2—C1—H1A	125.1
C3—O1—H1	109.5	F1—C9—C6	118.10 (19)
C7—C5—N1	130.24 (19)	F1—C9—C8	117.23 (19)
C7—C5—C4	122.58 (19)	C6—C9—C8	124.7 (2)
N1—C5—C4	107.18 (18)	C7—C8—C9	118.9 (2)
O2—C3—O1	122.08 (19)	C7—C8—H8	120.6
O2—C3—C2	122.83 (19)	C9—C8—H8	120.6
O1—C3—C2	115.09 (19)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2^{i}	0.82	1.86	2.669 (2)	171
N1—H1*B*···O2^{ii}	0.89 (3)	2.18 (3)	3.026 (2)	159 (2)

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