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

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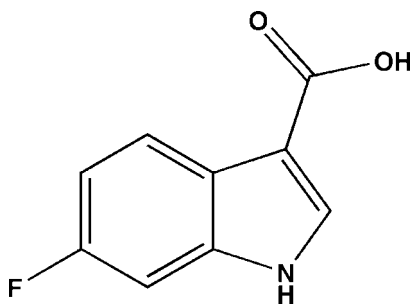
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.043; wR factor = 0.115; data-to-parameter ratio = 13.8.

In the title compound, $\text{C}_9\text{H}_6\text{FNO}_2$, all the non-H atoms are approximately coplanar, the carboxy O atoms deviating by 0.0809 and -0.1279 Å from the indole plane. In the crystal, $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds link the molecules into dimers which are linked *via* $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds and $\pi-\pi$ interactions [centroid-centroid distance = $3.680(2)$ Å]

Related literature

For the origin of the material studied, see: Kunzer & Wendt (2011). For a related structure, see: Luo *et al.* (2011).



Experimental

Crystal data

 $\text{C}_9\text{H}_6\text{FNO}_2$ $M_r = 179.15$

Monoclinic, $P2_1/c$
 $a = 7.0054(14)$ Å
 $b = 11.699(2)$ Å
 $c = 9.2947(19)$ Å
 $\beta = 104.15(3)^\circ$
 $V = 738.7(3)$ Å³

$Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.13$ mm⁻¹
 $T = 293$ K
 $0.3 \times 0.3 \times 0.2$ mm

Data collection

Rigaku SCXmini diffractometer
 Absorption correction: multi-scan
 (*CrystalClear*; Rigaku, 2005)
 $T_{\min} = 0.961$, $T_{\max} = 0.974$

7541 measured reflections
 1693 independent reflections
 1418 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.033$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.115$
 $S = 1.08$
 1693 reflections
 123 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.21$ e Å⁻³
 $\Delta\rho_{\min} = -0.21$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1}\cdots\text{O1}^{\text{i}}$	0.86 (2)	2.159 (19)	2.8925 (17)	142.8 (17)
$\text{O2}-\text{H2}\cdots\text{O1}^{\text{ii}}$	0.82	1.78	2.5954 (17)	170

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

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*.

LM thanks Southeast University, Jiangsu Province, PRC.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RN2099).

References

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 Kunzer, A. R. & Wendt, M. D. (2011). *Tetrahedron*, **52**, 1815–1818.
 Luo, Y.-H., Qian, X.-M., Gao, G., Li, J.-F. & Mao, S.-L. (2011). *Acta Cryst.* **E67**, m172.
 Rigaku. (2005). *CrystalClear*. Rigaku Corporation, Tokyo, Japan.
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supporting information

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6-Fluoro-1*H*-indole-3-carboxylic acid**Ming Lou and Yang-Hui Luo****S1. Comment**

Indole-3-carboxylic acid and its derivatives are important chemical materials, because they are excellent auxins for plants (Kunzer & Wendt, 2011) and drug intermediates for many pharmaceutical products (Luo et al., 2011).

The molecular structure of the title compound is shown in Fig. 1. All the non-H atoms are approximately coplanar: the carboxy O atoms deviating by 0.0809 and -0.1279 Å from the indole plane..

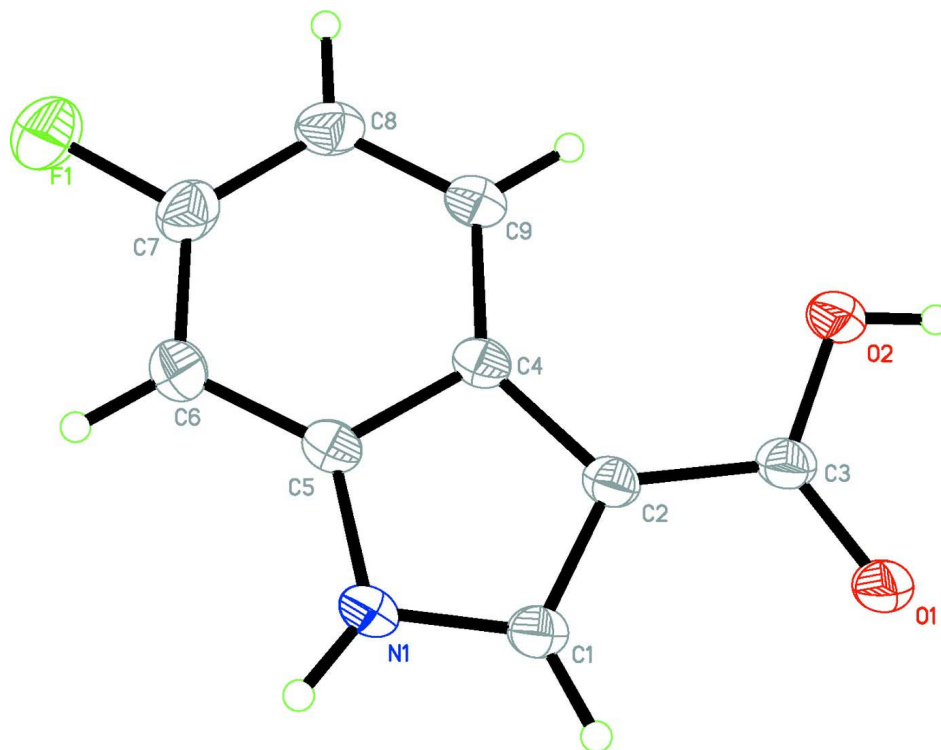
In the crystal structure of the title compound, intermolecular O—H···O hydrogen bonds linked the molecules into dimers and the dimers are linked via intermolecular N—H···O hydrogen bonds and π - π interactions [centroid-centroid distance = 3.680 (2) Å] (Fig. 2).

S2. Experimental

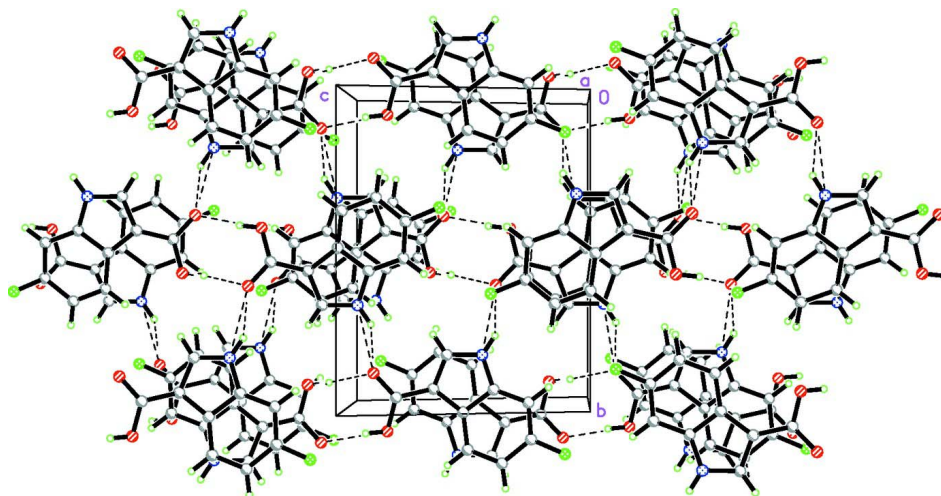
The title compound was purchased commercially from ChemFuture PharmaTech, Ltd (Jiangsu) and used as received without further purification. Crystals of it were obtained by slow evaporation of a methanol solution.

S3. Refinement

All H atoms attached to C, N and O atoms were fixed geometrically and treated as riding with C—H = 0.93 Å (CH), O—H = 0.82 Å and N—H = 0.86 Å with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{CH})$, $U_{\text{iso}}(\text{H}) = 1.35U_{\text{eq}}(\text{N})$ and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$.

**Figure 1**

The molecular structure of the title compound with the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented as small spheres of arbitrary radii.

**Figure 2**

A packing view down the *a* axis showing the three dimensional network. Intermolecular hydrogen bonds are shown as dashed lines.

6-Fluoro-1*H*-indole-3-carboxylic acid*Crystal data*C₉H₆FNO₂ $M_r = 179.15$ Monoclinic, $P2_1/c$ Hall symbol: - P 2ybc $a = 7.0054$ (14) Å $b = 11.699$ (2) Å $c = 9.2947$ (19) Å $\beta = 104.15$ (3)° $V = 738.7$ (3) Å³ $Z = 4$ $F(000) = 368$ $D_x = 1.611$ Mg m⁻³Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 1693 reflections

 $\theta = 3.5$ – 27.5 ° $\mu = 0.13$ mm⁻¹ $T = 293$ K

Block, brown

 $0.3 \times 0.3 \times 0.2$ mm*Data collection*

Rigaku SCXmini

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)

 $T_{\min} = 0.961$, $T_{\max} = 0.974$

7541 measured reflections

1693 independent reflections

1418 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.033$ $\theta_{\text{max}} = 27.5$ °, $\theta_{\text{min}} = 3.5$ ° $h = -9 \rightarrow 9$ $k = -15 \rightarrow 15$ $l = -12 \rightarrow 12$ *Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.043$ $wR(F^2) = 0.115$ $S = 1.08$

1693 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

 $w = 1/[\sigma^2(F_o^2) + (0.0591P)^2 + 0.1428P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\text{max}} < 0.001$ $\Delta\rho_{\text{max}} = 0.21$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.21$ 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 (Å²)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O2	0.60596 (19)	-0.07115 (8)	0.16978 (12)	0.0489 (3)
H2	0.5681	-0.0785	0.0795	0.073*
O1	0.5438 (2)	0.11097 (9)	0.10977 (12)	0.0514 (3)

F1	0.96927 (19)	-0.13190 (10)	0.89741 (12)	0.0727 (4)
N1	0.70843 (19)	0.17044 (11)	0.55849 (14)	0.0394 (3)
C3	0.5997 (2)	0.03550 (12)	0.20426 (16)	0.0377 (3)
C2	0.6606 (2)	0.06339 (11)	0.35810 (16)	0.0343 (3)
C4	0.74187 (19)	-0.00793 (11)	0.48256 (15)	0.0330 (3)
C9	0.7914 (2)	-0.12279 (12)	0.50269 (18)	0.0397 (4)
H9	0.7736	-0.1718	0.4217	0.048*
C6	0.8487 (2)	0.02441 (13)	0.74928 (18)	0.0432 (4)
H6	0.8692	0.0722	0.8316	0.052*
C8	0.8663 (2)	-0.16262 (13)	0.6426 (2)	0.0465 (4)
H8	0.8991	-0.2394	0.6583	0.056*
C5	0.7710 (2)	0.06329 (12)	0.60672 (16)	0.0351 (3)
C1	0.6428 (2)	0.17026 (12)	0.41164 (16)	0.0382 (3)
H1A	0.5925	0.2335	0.3542	0.046*
C7	0.8931 (2)	-0.08866 (15)	0.76066 (19)	0.0475 (4)
H1	0.702 (3)	0.2305 (17)	0.611 (2)	0.053 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O2	0.0787 (8)	0.0289 (6)	0.0382 (6)	-0.0018 (5)	0.0127 (6)	-0.0044 (4)
O1	0.0856 (9)	0.0304 (6)	0.0364 (6)	-0.0084 (5)	0.0116 (6)	0.0019 (4)
F1	0.0872 (8)	0.0638 (8)	0.0524 (7)	0.0055 (6)	-0.0111 (6)	0.0161 (5)
N1	0.0514 (7)	0.0270 (6)	0.0399 (7)	-0.0006 (5)	0.0114 (6)	-0.0068 (5)
C3	0.0479 (8)	0.0291 (7)	0.0388 (8)	-0.0050 (6)	0.0156 (6)	-0.0007 (5)
C2	0.0397 (7)	0.0281 (7)	0.0370 (8)	-0.0039 (5)	0.0128 (6)	-0.0025 (5)
C4	0.0321 (6)	0.0291 (7)	0.0393 (8)	-0.0030 (5)	0.0115 (6)	-0.0025 (5)
C9	0.0417 (8)	0.0300 (7)	0.0475 (9)	0.0004 (6)	0.0114 (7)	-0.0042 (6)
C6	0.0450 (8)	0.0430 (8)	0.0388 (8)	-0.0053 (7)	0.0046 (6)	-0.0044 (6)
C8	0.0456 (9)	0.0329 (8)	0.0585 (10)	0.0055 (6)	0.0079 (7)	0.0050 (7)
C5	0.0342 (7)	0.0306 (7)	0.0411 (8)	-0.0034 (5)	0.0101 (6)	-0.0038 (6)
C1	0.0477 (8)	0.0287 (7)	0.0392 (8)	-0.0010 (6)	0.0124 (6)	0.0001 (6)
C7	0.0442 (8)	0.0477 (9)	0.0447 (9)	0.0006 (7)	-0.0005 (7)	0.0089 (7)

Geometric parameters (Å, °)

O2—C3	1.2916 (17)	C4—C9	1.389 (2)
O2—H2	0.8200	C4—C5	1.3974 (19)
O1—C3	1.2394 (18)	C9—C8	1.360 (2)
F1—C7	1.351 (2)	C9—H9	0.9300
N1—C1	1.330 (2)	C6—C7	1.357 (2)
N1—C5	1.3668 (19)	C6—C5	1.381 (2)
N1—H1	0.86 (2)	C6—H6	0.9300
C3—C2	1.426 (2)	C8—C7	1.373 (3)
C2—C1	1.363 (2)	C8—H8	0.9300
C2—C4	1.427 (2)	C1—H1A	0.9300
C3—O2—H2	109.5	C7—C6—C5	115.15 (14)

C1—N1—C5	109.75 (12)	C7—C6—H6	122.4
C1—N1—H1	121.7 (13)	C5—C6—H6	122.4
C5—N1—H1	128.4 (13)	C9—C8—C7	119.56 (15)
O1—C3—O2	122.44 (14)	C9—C8—H8	120.2
O1—C3—C2	120.84 (13)	C7—C8—H8	120.2
O2—C3—C2	116.72 (13)	N1—C5—C6	129.53 (14)
C1—C2—C3	122.98 (14)	N1—C5—C4	107.80 (13)
C1—C2—C4	107.12 (12)	C6—C5—C4	122.67 (14)
C3—C2—C4	129.88 (13)	N1—C1—C2	109.68 (13)
C9—C4—C5	118.97 (13)	N1—C1—H1A	125.2
C9—C4—C2	135.39 (13)	C2—C1—H1A	125.2
C5—C4—C2	105.64 (12)	F1—C7—C6	117.94 (16)
C8—C9—C4	119.04 (14)	F1—C7—C8	117.45 (15)
C8—C9—H9	120.5	C6—C7—C8	124.61 (15)
C4—C9—H9	120.5		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H1 \cdots O1 ⁱ	0.86 (2)	2.159 (19)	2.8925 (17)	142.8 (17)
O2—H2 \cdots O1 ⁱⁱ	0.82	1.78	2.5954 (17)	170

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