

William T. A. Harrison,^{a*} H. S. Yathirajan,^b B. V. Ashalatha,^c K. K. Vijaya Raj^c and B. Narayana^c

^aDepartment of Chemistry, University of Aberdeen, Meston Walk, Aberdeen AB24 3UE, Scotland, ^bDepartment of Studies in Chemistry, University of Mysore, Manasagangotri, Mysore 570 006, India, and ^cDepartment of Chemistry, Mangalore University, Mangalagangothri 574 199, India

Correspondence e-mail:
w.harrison@abdn.ac.uk

Key indicators

Single-crystal X-ray study
 T = 120 K
 Mean $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$
 R factor = 0.032
 wR factor = 0.089
 Data-to-parameter ratio = 12.4

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

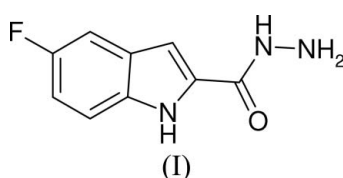
5-Fluoro-1*H*-indole-2-carbohydrazide

The geometric parameters for the essentially planar molecule of the title compound, $\text{C}_9\text{H}_8\text{FN}_3\text{O}$, are normal. A network of $\text{N}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{N}$ hydrogen bonds helps to establish the crystal packing.

Received 4 October 2006
 Accepted 4 October 2006

Comment

As part of our ongoing research into indole carboxylic acid derivatives (Harrison *et al.*, 2006), the synthesis and crystal structure of the title compound, (I) (Fig. 1), are now presented.



The geometric parameters for (I) fall within their expected ranges (Allen *et al.*, 1987). The indole ring system is essentially flat (r.m.s. deviation from the mean plane = 0.005 Å). The mean plane of atoms C9, O1, N2 and N3 of the carbohydrazide side chain is slightly twisted away from the indole mean plane [dihedral angle = 5.27 (9)°]. The bond angle sum about N2 is 359°, suggesting sp^2 -hybridization for this atom. Conversely, the average bond angle for N3 of 108° suggests sp^3 -hybridization.

The crystal packing in (I) is influenced by $\text{N}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{N}$ hydrogen bonds (Table 1). Inversion-generated dimeric pairs of molecules are linked by a pair of $\text{N}3-\text{H}3\cdots\text{O}1^{\text{iii}}$ hydrogen bonds (Fig. 2). Adjacent molecules are then linked into ribbons by a combination of the $\text{N}2-\text{H}2\cdots\text{O}1^{\text{ii}}$ and $\text{N}1\cdots\text{H}1-\text{N}3^{\text{i}}$ bonds. In terms of graph theory (Bernstein *et al.*, 1995) these two hydrogen-bonding motifs result in $R_2^2(10)$ and $R_2^2(8)$ loops, respectively. Combining the two results in (001) sheets of molecules. Atom H4, attached to N3, does not participate in hydrogen bonds. A PLATON (Spek, 2003) analysis of (I) indicated a short $\text{C}-\text{H}\cdots\text{F}$ contact that may also help to consolidate the crystal packing. In the packing of (I), a zigzag stacking of molecules with respect to the *c* direction is seen (Fig. 3). Any $\pi-\pi$ stacking interactions in (I) must be very weak, the shortest intermolecular ring-centroid separation being 4.08 Å.

Experimental

Methyl-5-fluoroindole-2-carboxylate (2.34 g, 0.01 mol) (Harrison *et al.*, 2006) in 25 ml of absolute ethanol was refluxed with 1.0 ml of hydrazine hydrate for 2 h, with the reaction progress monitored by

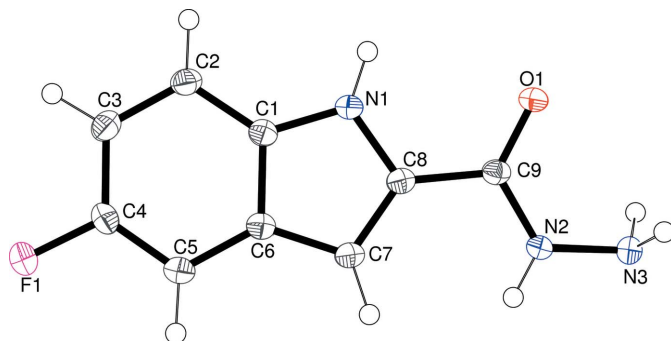


Figure 1
View of the molecular structure of (I), showing 50% probability displacement ellipsoids and arbitrary spheres for the H atoms.

thin-layer chromatography. Upon completion, the mixture was cooled to room temperature. The separated solid was filtered off and washed with cold ethanol; cubes of (I) were recrystallized from ethanol (m.p. 505–507 K). Analysis found (calculated) for $C_9H_8FN_3O$: C 55.70 (55.96), H 4.12 (4.17), N 21.65 (21.75)%.

Crystal data

$C_9H_8FN_3O$
 $M_r = 193.18$
 Orthorhombic, $Pbca$
 $a = 10.0451$ (3) Å
 $b = 9.4978$ (2) Å
 $c = 18.5293$ (6) Å
 $V = 1767.81$ (9) Å³

$Z = 8$
 $D_x = 1.452$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 0.11$ mm⁻¹
 $T = 120$ (2) K
 Cube, colourless
 $0.20 \times 0.20 \times 0.20$ mm

Data collection

Nonius KappaCCD diffractometer
 φ and ω scans
 Absorption correction: multi-scan
 (SADABS; Bruker, 2003)
 $T_{\min} = 0.978$, $T_{\max} = 0.978$

11208 measured reflections
 1733 independent reflections
 1497 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.029$
 $\theta_{\text{max}} = 26.1^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.032$
 $wR(F^2) = 0.089$
 $S = 1.06$
 1733 reflections
 140 parameters
 H atoms treated by a mixture of
 independent and constrained
 refinement

$w = 1/[\sigma^2(F_o^2) + (0.0434P)^2 + 0.7328P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.25$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.19$ e Å⁻³
 Extinction correction: *SHELXL97*
 Extinction coefficient: 0.015 (2)

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N1-H1\cdots N3^i$	0.898 (16)	2.096 (16)	2.9864 (15)	171.3 (13)
$N2-H2\cdots O1^{ii}$	0.870 (16)	2.073 (16)	2.9193 (14)	163.9 (14)
$N3-H3\cdots O1^{iii}$	0.920 (16)	2.124 (16)	3.0241 (15)	165.8 (13)
$C3-H3A\cdots F1^{iv}$	0.95	2.55	3.2082 (15)	127

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

The N-bound H atoms were located in difference maps and their positions were freely refined with $U_{\text{iso}}(\text{H})$ set equal to $1.2U_{\text{eq}}(\text{N})$. The

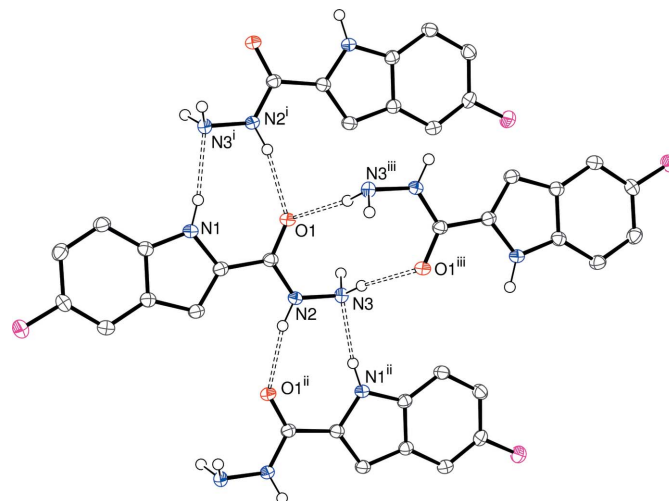


Figure 2
Fragment of the crystal structure of (I), showing the hydrogen-bonding (dashed lines) scheme, with C-bound H atoms omitted for clarity. Symmetry codes as in Table 1.

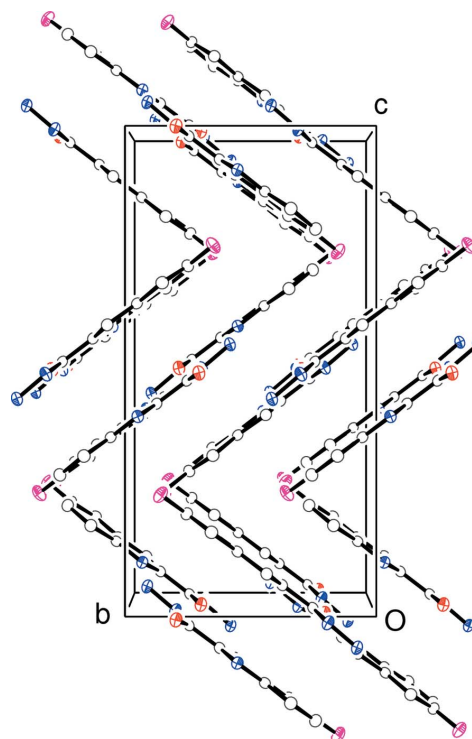


Figure 3
The packing for (I), with all H atoms omitted for clarity.

C-bound H atoms were placed in idealized locations ($C-H = 0.95$ Å) and refined as riding with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor 1997); data reduction: *HKL DENZO* and *SCALEPACK* (Otwinowski & Minor 1997), and *SORTAV* (Blessing, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

The authors thank the EPSRC National Crystallographic Service (University of Southampton) for data collection. ABV thanks Mangalore University for provision of research facilities.

References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
- Bernstein, J., Davis, R. E., Shimon, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
- Blessing, R. H. (1995). *Acta Cryst.* **A51**, 33–38.
- Bruker (2003). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Harrison, W. T. A., Yathirajan, H. S., Ashalatha, B. V., Vijaya Raj, K. K. & Narayana, B. (2006). *Acta Cryst.* **E62**, o4050–o4051.
- Nonius (1998). *COLLECT*. Nonius, Delft, The Netherlands.
- Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.