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Methyl 5-fluoro-1*H*-indole-2-carboxylate

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.002 Å
R factor = 0.042
wR factor = 0.110
Data-to-parameter ratio = 14.7

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

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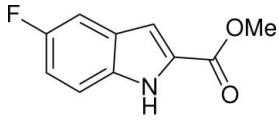
The geometrical parameters for the title compound, $C_{10}H_8FNO_2$, are normal. In the crystal structure, the molecules form inversion-symmetry-generated dimeric pairs by way of two N—H···O hydrogen bonds.

Comment

Several indolecarboxylic acid derivatives show biological activity: methyl indole-3-carboxylate, extracted from a marine microorganism (Hu *et al.*, 2005), is cytotoxic against the K562 human leukaemia strain. Methyl indole-2-carboxylic acid may serve as a glycine site antagonist and hence aid in the treatment of human brain injuries (Morzyk-Ociepa *et al.*, 2004). 5-Fluoroindole-3-acetic acid (Antolic *et al.*, 1996) has plant-growth regulating activity. The crystal structure of methyl indole-2-carboxylate has been deposited [Parsons, S., McNab, H. & Wood, P. (2004). refcode OCAQEP] with the Cambridge Structural Database (CSD; Version 5.27; Allen, 2002). As part of our ongoing research in this area, the structure of the related title compound, (I) (Fig. 1), prepared by the Fischer indole synthesis reaction (Narayana *et al.*, 2005), is now presented.



(I)

The geometrical parameters for (I) are consistent with those of the compounds noted above. In particular, methyl indole-2-carboxylic acid, (II) (Morzyk-Ociepa *et al.*, 2004), has almost identical geometry to (I). For example, the benzene-ring bond lengths (Å) in (I) are C1—C2 = 1.396 (2) [equivalent value in (II) = 1.390 (2) Å], C2—C3 = 1.375 (2) [1.372 (2)], C3—C4 = 1.399 (2) [1.404 (2)], C4—C5 = 1.356 (2) [1.357 (2)], C5—C6 = 1.408 (2) [1.409 (2)] and C6—C1 = 1.416 (2) [1.403 (2)]. Apart from the methyl H atoms, the molecule in (I) is essentially planar [r.m.s. deviation of the non-H atoms from the mean plane = 0.031 Å, max. = 0.0327 (11) Å for N1]. The bond angle sum about N1 is 359.7°. The crystal packing in (I) exhibits inversion-symmetry-generated dimeric pairs of molecules linked by two N—H···O hydrogen bonds (Table 1 and Fig. 2). A similar pairing arrangement was seen in the structure of methyl indole-2-carboxylate (CSD refcode OCAQEP) although the overall structure is different to (I). Conversely, in methyl indole-2-carboxylic acid (Morzyk-Ociepa *et al.*, 2004) a completely different arrangement of N—H···O and O—H···O hydrogen bonds leads to chains of molecules. There are no π–π stacking interactions in (I), the shortest intermolecular ring-centroid separation being 4.35 Å.

Experimental

Methyl pyruvate-4-fluorophenylhydrazone (2 g, 0.0095 mol) was added to 10 g polyphosphoric acid and continuously stirred for proper mixing. The reaction mass was slowly heated to 353–363 K and maintained for 4 h. The progress of the reaction was monitored by TLC. The reaction mass was cooled and water (100 ml) was added to break up the lumps until it became a slurry. The separated solid was filtered off and washed with water. The dried crude product was charcoalized in ethyl acetate, filtered over hyflo/silica gel, slowly cooled to room temperature and kept overnight with stirring. After recrystallization from ethyl acetate, colourless crystals of (I) were obtained in 60% yield (m.p. 474 K). Analysis found (calculated) for $C_{10}H_8FNO_2$: C 62.11 (62.18), H 4.09 (4.17), N 7.13 (7.25)%.

Crystal data

$C_{10}H_8FNO_2$
M_r = 193.17
Monoclinic, $P2_1/n$
a = 12.4420 (7) Å
b = 3.8185 (1) Å
c = 18.269 (1) Å
β = 100.125 (2)°
V = 854.43 (7) Å^3
Z = 4
D_x = 1.502 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 120 (2) K
Needle, colourless
0.41 × 0.07 × 0.05 mm

Data collection

Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2003)
T_{min} = 0.952, T_{max} = 0.994
10458 measured reflections
1937 independent reflections
1311 reflections with $I > 2\sigma(I)$
R_{int} = 0.052
θ_{max} = 27.6°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.110
S = 1.03
1937 reflections
132 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0568P)^2 + 0.104P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.014 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2^{i}	0.883 (18)	2.019 (18)	2.8555 (18)	157.7 (15)

Symmetry code: (i) $-x+1, -y, -z$.

The N-bound H atom was located in a difference map and its position was freely refined with U_{iso}(H) = 1.2U_{eq}(N). The C-bound H atoms were placed in idealized locations (C—H = 0.95–0.99 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C). The methyl group was rotated about its C—N bond to best fit the electron density.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK*, *DENZO* (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*.

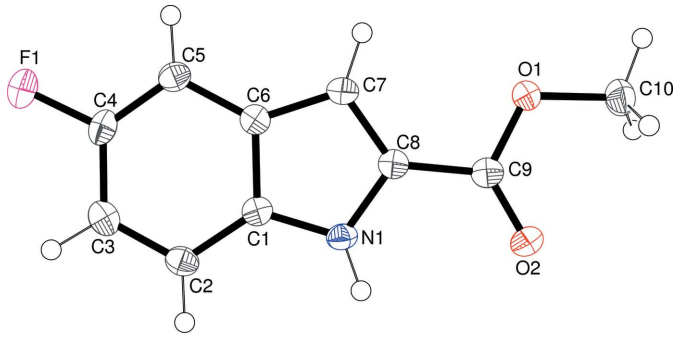


Figure 1
View of (I), showing 50% probability displacement ellipsoids and arbitrary spheres for the H atoms.

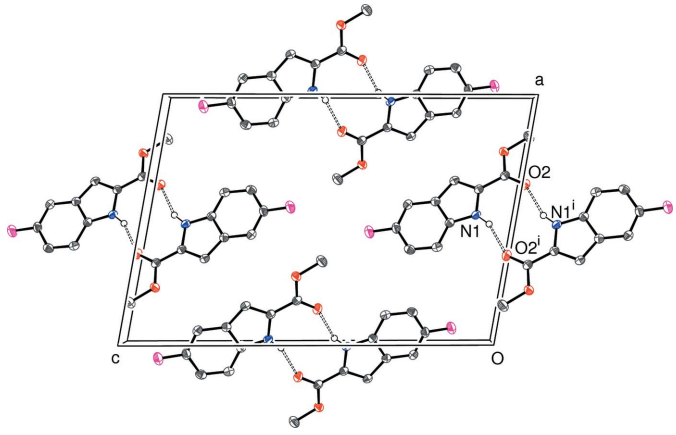


Figure 2
Unit cell packing in (I) with all H atoms except H1 omitted for clarity and hydrogen bonds indicated by dashed lines. See Table 1 for symmetry code.

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