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4-(4-Fluorophenyl)-3-methyl-6-oxo-1-phenyl-4,5,6,7-tetrahydro-1*H*-pyrazolo[3,4-*b*]pyridine-5-carbonitrile

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.005 Å
R factor = 0.057
wR factor = 0.165
Data-to-parameter ratio = 13.0

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

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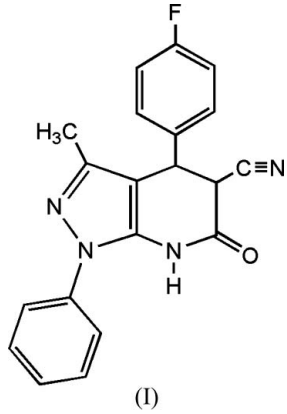
The title compound, $C_{20}H_{15}FN_4O$, was synthesized by the reaction of 5-amino-3-methyl-1-phenylpyrazole with ethyl 2-cyano-3-(4-fluorophenyl)-1-acylate in glycol under microwave irradiation. The tetrahydropyridine ring adopts a distorted envelope conformation. The pyrazole ring forms a dihedral angle of 39.2 (3)° with the attached phenyl ring.

Comment

The pyrazolo[3,4-*b*]pyridine system has many interesting biological and pharmacological properties and is used in the treatment of a wide variety of stress-related illnesses (Sekikawa *et al.*, 1973; Kuczynski *et al.*, 1979; El-Dean *et al.*, 1991). As part of our program aimed at employing microwave irradiation for the preparation of heterocyclic compounds (Tu *et al.*, 2004), we have recently synthesized the title pyrazolo[3,4-*b*]pyridine derivative, (I), under microwave irradiation and we report here its crystal structure.



(I)

The molecular structure of (I) is shown in Fig. 1. The tetrahydropyridine ring adopts a distorted envelope conformation, with atom C1 and C2 deviating from the C3/C4/C5/N1 plane by 0.231 (1) and 0.731 (1) Å, respectively, so that C2 is the main flap atom. The pyrazole ring forms a dihedral angle of 39.2 (3)° with the attached phenyl ring.

Experimental

A dry flask (25 ml) was charged with 5-amino-3-methyl-1-phenylpyrazole (2 mmol), ethyl 2-cyano-3-(4-fluorophenyl)-1-acylate (2 mmol) and glycol (1 ml). The unsealed reaction vessel was put into a modified household microwave oven and connected to refluxing equipment. After microwave irradiation for 5 min (250 W), the reaction mixture was cooled and washed with a small amount of ethanol. The crude product was filtered off and single crystals of (I) were obtained by recrystallization from a 95% ethanol solution (yield

80%). Spectroscopic analysis: IR (KBr, ν, cm^{-1}): 3360, 3074 (NH_2), 2228 (CN), 1703 (CO); ^{1}H NMR (300 MHz, DMSO-d_6): 2.04 (3H, *s*, CH_3), 4.96–5.18 (1H, *m*, CH), 4.68–4.77 (1H, *m*, CH), 7.16–7.81 (9H, *m*, ArH), 11.30(1H, *s*, NH).

Crystal data

$C_{20}H_{15}FN_4O$
M_r = 346.36
Orthorhombic, *Pbca*
a = 10.856 (5) Å
b = 8.245 (4) Å
c = 38.898 (17) Å
V = 3482 (3) Å^3
Z = 8
D_x = 1.322 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2536 reflections
θ = 2.1–21.1°
μ = 0.09 mm^{-1}
T = 298 (2) K
Block, colorless
0.45 × 0.41 × 0.35 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
17020 measured reflections
3069 independent reflections
1712 reflections with $I > 2\sigma(I)$
R_{int} = 0.061
θ_{max} = 25.0°
$h = -12 \rightarrow 12$
$k = -8 \rightarrow 9$
$l = -34 \rightarrow 46$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.165
S = 1.03
3069 reflections
236 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.054P)^2 + 3.1712P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C1	1.368 (4)	C2—C3	1.561 (4)
N1—C5	1.389 (4)	C3—C4	1.503 (4)
C1—C2	1.532 (5)	C4—C5	1.351 (4)
C1—N1—C5	119.8 (3)	C4—C3—C8	115.3 (3)
N1—C1—C2	114.5 (3)	C4—C3—C2	104.9 (3)
C7—C2—C1	109.1 (3)	C8—C3—C2	113.8 (3)
C7—C2—C3	111.5 (3)	C5—C4—C3	121.5 (3)
C1—C2—C3	115.3 (2)	C4—C5—N1	124.9 (3)

Methyl H atoms were placed in calculated positions, with C—H = 0.96 Å and torsion angles refined to fit the electron density, with U_{iso}(H) = 1.5U_{eq}(C). Other H atoms were placed in idealized positions, with C—H = 0.93 − 0.98 Å and N—H = 0.86 Å, and allowed to ride on their parent atoms, with U_{iso}(H) = 1.2 U_{eq}(carrier).

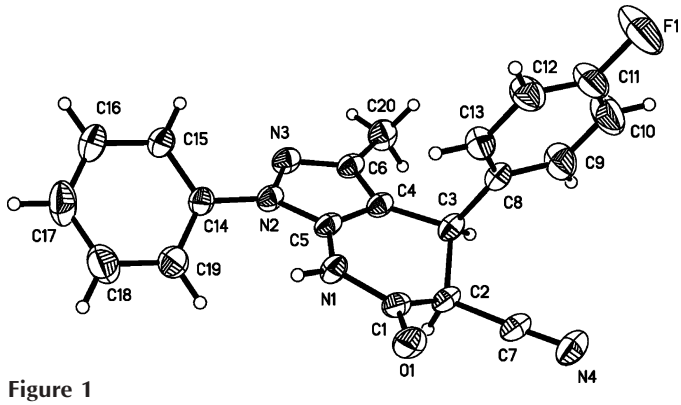


Figure 1
The molecular structure of (I), showing 40% probability displacement ellipsoids (arbitrary spheres for H atoms).

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); 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, 1999); software used to prepare material for publication: *SHELXTL*.

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References

Bruker (1999). *SMART*, *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
El-Dean, A. M. K., Atalla, A. A., Mohamed, T. A. & Geies, A. A. (1991). *Z. Naturforsch. Teil B*, **46**, 541–546.
Kuczynski, L., Mrozikiewic, A., Banaszkiewicz, W. & Poreba, K. (1979). *J. Pharmacol. Pharm.* **31**, 217–225.
Sekikawa, I., Nishie, J., Tono-oka, S., Tanaka, Y. & Kakimoto, S. (1973). *J. Heterocycl. Chem.* **10**, 931–932.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Tu, S.-J., Fang, F., Zhu, S.-L., Li, T.-J., Zhang, X.-J. & Zhuang, Q.-Y. (2004). *Synlett*, pp. 537–539.