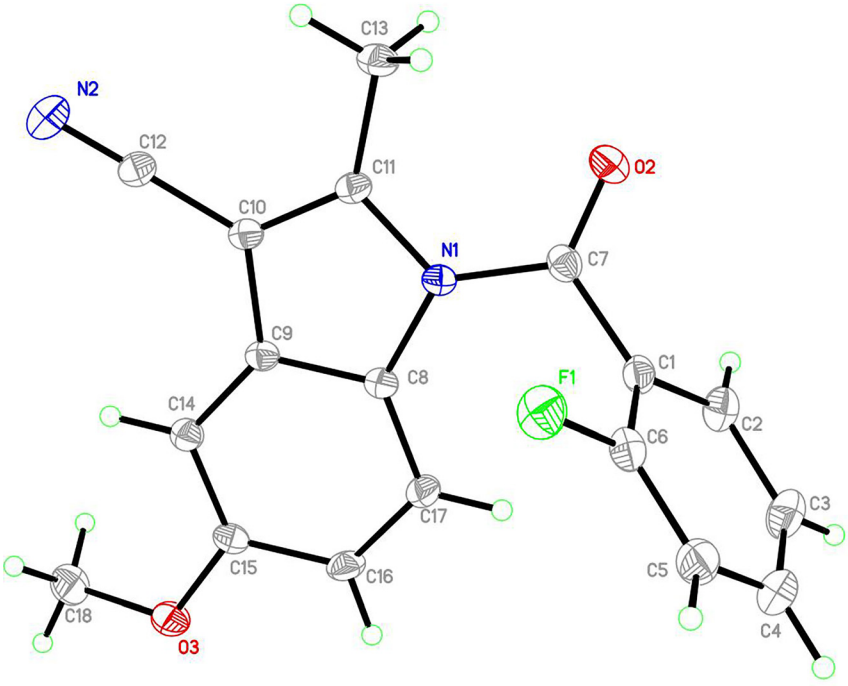
Ma Siyuan and Guo Mengping*

The crystal structure of 1-(2-fluorobenzoyl)-5-methoxy-2-methyl-1*H*-indole-3-carbonitrile, $C_{18}H_{13}FN_2O_2$



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Abstract

$C_{18}H_{13}FN_2O_2$, orthorhombic, *Pbca* (no. 61), a = 13.8082(4) Å, b = 9.7555(3) Å, c = 21.8116(7) Å, V = 2938.15(16) Å^3, Z = 8, $R_{gt}(F)$ = 0.0402, w$R_{ref}(F^2)$ = 0.1131, T = 293 K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless prism
Size:	0.13 × 0.10 × 0.08 mm
Wavelength:	Cu *K*α radiation (1.54184 Å)
μ:	0.84 mm^{-1}
Diffractometer, scan mode:	SuperNova, ω
θ_{max}, completeness:	73.9°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	6784, 2903, 0.026
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2374
$N(param)_{refined}$:	211
Programs:	Bruker [1], SHELX [2, 3]

1 Source of material

The chemical reagents and catalysts used in the reaction were all purchased and used without further purification. First, a mixture of 5-methoxy-2-methyl-1*H*-indole (30 mmol), $K_4[Fe(CN)_6]$ (15 mmol), $PdCl_2(NH_2CH_2COOH)_2$ (1.2 mmol) and $Cu(OAc)_2$ (90 mmol) was dissolved in 50 mL dimethyl sulfoxide; then it was stirred at 130 °C for 2.5 h. The reaction

***Corresponding author: Guo Mengping**, College of Chemistry and Bioengineering, Yichun University, Yichun 336000, Jiangxi, P. R. China, E-mail: guomengping65@163.com. https://orcid.org/0000-0003-1801-6740
Ma Siyuan, College of Chemistry and Bioengineering, Yichun University, Yichun 336000, Jiangxi, P. R. China

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
F1	0.79540 (7)	0.58714 (11)	0.64414 (4)	0.0441 (3)
O2	0.56463 (9)	0.40184 (12)	0.64281 (5)	0.0385 (3)
O3	0.62630 (9)	1.01738 (11)	0.42774 (5)	0.0371 (3)
N1	0.61706 (8)	0.54114 (12)	0.56578 (5)	0.0241 (3)
N2	0.64742 (11)	0.39501 (16)	0.35930 (7)	0.0421 (4)
C1	0.63279 (10)	0.61351 (16)	0.67371 (6)	0.0279 (3)
C2	0.56527 (12)	0.67561 (19)	0.71208 (7)	0.0369 (4)
H2	0.500642	0.649012	0.710518	0.044*
C3	0.59419 (14)	0.7769 (2)	0.75256 (8)	0.0454 (4)
H3	0.548560	0.820632	0.777090	0.054*
C4	0.69090 (14)	0.8134 (2)	0.75663 (8)	0.0464 (4)
H4	0.709743	0.881840	0.783816	0.056*
C5	0.75975 (12)	0.7491 (2)	0.72066 (7)	0.0415 (4)
H5	0.824988	0.771553	0.724030	0.050*
C6	0.72899 (11)	0.65097 (17)	0.67976 (7)	0.0316 (3)
C7	0.60157 (10)	0.50908 (16)	0.62788 (7)	0.0272 (3)
C8	0.62103 (9)	0.67404 (15)	0.53889 (6)	0.0238 (3)
C9	0.62922 (9)	0.65666 (15)	0.47537 (6)	0.0231 (3)
C10	0.63063 (9)	0.51107 (15)	0.46441 (6)	0.0240 (3)
C11	0.62252 (9)	0.44331 (15)	0.51898 (7)	0.0241 (3)
C12	0.64033 (10)	0.44601 (16)	0.40664 (7)	0.0284 (3)
C13	0.62605 (10)	0.29257 (15)	0.52887 (7)	0.0301 (3)
H13A	0.675527	0.271378	0.558305	0.045*
H13B	0.640395	0.247579	0.490793	0.045*
H13C	0.564521	0.261439	0.543898	0.045*
C14	0.63162 (9)	0.76835 (15)	0.43545 (7)	0.0264 (3)
H14	0.637450	0.756126	0.393326	0.032*
C15	0.62503 (10)	0.89790 (15)	0.46084 (7)	0.0270 (3)
C16	0.61627 (11)	0.91523 (16)	0.52429 (7)	0.0309 (3)
H16	0.612160	1.003503	0.540180	0.037*
C17	0.61355 (11)	0.80522 (16)	0.56392 (7)	0.0286 (3)
H17	0.606950	0.818029	0.605968	0.034*
C18	0.60779 (14)	1.00748 (19)	0.36355 (8)	0.0420 (4)
H18A	0.659488	0.957984	0.344159	0.063*
H18B	0.603645	1.097815	0.346297	0.063*
H18C	0.547793	0.959973	0.357007	0.063*

mixture was poured into 500 mL cool water to produce a crude product, which was purified by flash column chromatography over silica gel (dichloromethane/*n*-hexane 4/1) to afford the intermediate 5-methoxy-2-methyl-1*H*-indole-3-carbonitrile. Second, in a 25 mL round bottom flask, 4-fluorobenzoyl chloride dissolved in 20 mL dichloromethane was dripped into the mixture of 5-methoxy-2-methyl-1*H*-indole-3carbonitrile (4 mmol), triethylamine (6 mmol), 4-dimethylaminopyridine (0.2 mmol) and dichloromethane (2 mL) at room temperature. The reaction was monitored by TLC until completed. The solvent was removed under vacuum. The crude compound was isolated by thin-layer chromatography (dichloromethane/*n*-hexane 2/1) to obtain the title product. Grayish white crystals of the title compound were obtained by recrystallizing from an anhydrous ethanol solution at room temperature over a period of several days.

2 Experimental details

The H atoms were geometrically placed (C—H = 0.95–0.98 Å) and refined as riding with U_{iso}(H) = 1.2–1.5 U_{eq}(C).

3 Comment

The indole scaffold is one of the most widespread heterocycles in natural products and bioactive molecules [4–8]. Due to its biodiversity and versatility, it has been used as a privileged scaffold for the design of bioactive molecules.

The title compound molecular structure contains one molecule in the asymmetric unit as shown in the figure. The bond lengths and angles within the title structure are all in normal ranges [9, 10]. The atoms in indole ring are all coplanar. The torsion angle of C(12)–N(10)–C(11)–N(1) and C(12)–C(10)–C(9)–C(14) are 178.36(12)° and 3.6(2)° respectively, which indicate the carbonitrile group is almost coplanar with the indole ring. It can be seen that there is a twist angle between the carbonyl group and the indole ring, which is different from the reported results [10]. This may be caused by larger benzene group.

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