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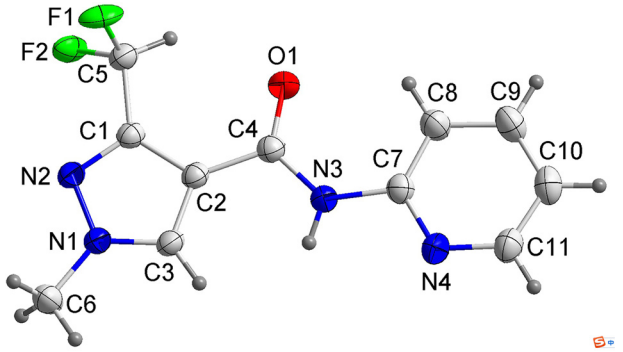
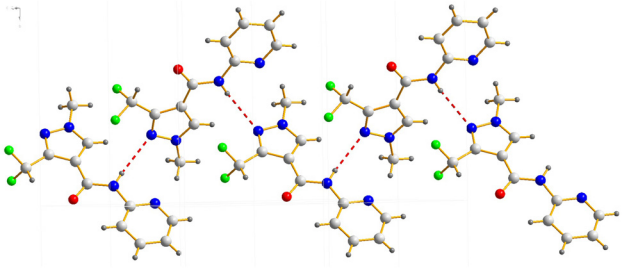
# Crystal structure of 3-(difluoromethyl)-1-methyl-*N*-(pyridin-2-yl)-1*H*-pyrazole-4-carboxamide, $C_{11}H_{10}F_2N_4O$



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**Abstract**

$C_{11}H_{10}F_2N_4O$, monoclinic, $P2_1/n$ (no. 14), $a$ = 8.7616(10) Å, $b$ = 14.2418(17) Å, $c$ = 9.7191(12) Å, $\beta$ = 107.6020°, $V$ = 1156.0(2) Å$^3$, $Z$ = 4, $R_{gt}(F)$ = 0.0431, $wR_{ref}(F^2)$ = 0.1263, T = 296(2) K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

## 1 Source of materials

4-(Difluoromethyl)-1-methyl-1*H*-pyrazole-4-carboxylic acid (1.76 g, 10.0 mmol), thionyl chloride (30.0 mmol) and *N*,*N*-dimethylformamide (0.5 mL) reacted at 95 °C for 4 h to obtain 3-(difluoromethyl)-1-methyl-1*H*-pyrazole-4-carbonyl chloride. The product was dissolved in $CH_2Cl_2$ (30.0 mL) for later use. Dissolve pyridin-2-amine (0.94 g, 10.0 mmol) and triethylamine (2.02 g, 20.0 mmol) in $CH_2Cl_2$ (30.0 mL) and cool to 0 °C. Add the solution of 3-(difluoromethyl)-1-methyl-1*H*-pyrazole-4-carbonyl chloride slowly under stirring conditions and react at room temperature for 6 h. Use TLC to detect the reaction until the raw material completely disappears. After the reaction is completed, elute and purify with column chromatography to obtain the title product in methanol.

## 2 Experimental details

All H atoms were included in calculated positions and refined as riding atoms, with C–H = 0.90–0.97 Å with $U_{iso}$(H) = 1.5 $U_{eq}$(C) for methyl H atoms and 1.2 $U_{eq}$(C) for all other H atoms.

**Table 1:** Data collection and handling.

| | |
|---|---|
| Crystal: | Colorless block |
| Size: | 0.20 × 0.18 × 0.14 mm |
| Wavelength: | Mo *K*α radiation (0.71073 Å) |
| $\mu$: | 0.12 mm$^{-1}$ |
| Diffractometer, scan mode: | Bruker APEX-II, $\varphi$ and $\omega$ |
| $\theta_{max}$, completeness: | 25.5°, >99 % |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$, $R_{int}$: | 8788, 2148, 0.024 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\sigma(I_{obs})$, 1745 |
| $N(param)_{refined}$: | 165 |
| Programs: | Bruker [1], SHELX [2, 3], Diamond [4] |

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

| Atom | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.9350 (3) | 0.56118 (15) | 1.2027 (2) | 0.0615 (6) |
| H1 | 0.9816 | 0.5561 | 1.3020 | 0.074* |
| C2 | 0.9831 (3) | 0.63278 (15) | 1.1330 (3) | 0.0661 (6) |
| H2 | 1.0607 | 0.6753 | 1.1830 | 0.079* |
| C3 | 0.9137 (3) | 0.64052 (16) | 0.9866 (3) | 0.0697 (6) |
| H3 | 0.9442 | 0.6886 | 0.9358 | 0.084* |
| C4 | 0.7985 (3) | 0.57676 (15) | 0.9150 (2) | 0.0616 (6) |
| H4 | 0.7491 | 0.5813 | 0.8160 | 0.074* |
| C5 | 0.7593 (2) | 0.50616 (13) | 0.9952 (2) | 0.0482 (5) |
| C6 | 0.5493 (2) | 0.42514 (13) | 0.8007 (2) | 0.0492 (5) |
| C7 | 0.4370 (2) | 0.34540 (12) | 0.77671 (19) | 0.0446 (4) |
| C8 | 0.3235 (2) | 0.31971 (12) | 0.64576 (19) | 0.0447 (4) |
| C9 | 0.2908 (3) | 0.36607 (14) | 0.5021 (2) | 0.0545 (5) |
| H9 | 0.3780 | 0.4090 | 0.5014 | 0.065* |
| C10 | 0.4122 (2) | 0.28160 (13) | 0.87412 (19) | 0.0479 (5) |
| H10 | 0.4678 | 0.2790 | 0.9721 | 0.057* |
| C11 | 0.2273 (3) | 0.14658 (15) | 0.8623 (2) | 0.0630 (6) |
| H11A | 0.2549 | 0.0885 | 0.8258 | 0.094* |
| H11B | 0.1129 | 0.1528 | 0.8343 | 0.094* |
| H11C | 0.2693 | 0.1471 | 0.9657 | 0.094* |
| N1 | 0.8249 (2) | 0.49765 (12) | 1.13706 (18) | 0.0568 (5) |
| N2 | 0.64840 (19) | 0.43440 (11) | 0.93725 (17) | 0.0521 (4) |
| H2A | 0.6427 | 0.3905 | 0.9963 | 0.063* |
| N3 | 0.29479 (18) | 0.22431 (10) | 0.80345 (16) | 0.0479 (4) |
| N4 | 0.23787 (19) | 0.24589 (11) | 0.66193 (16) | 0.0493 (4) |
| O1 | 0.5494 (2) | 0.47916 (11) | 0.70316 (17) | 0.0755 (5) |
| F1 | 0.2688 (2) | 0.30146 (10) | 0.39675 (13) | 0.0867 (5) |
| F2 | 0.1503 (2) | 0.41216 (12) | 0.46903 (16) | 0.1016 (6) |

## 3 Comment

Pyrazolamide compounds have excellent insecticidal and bactericidal activities, which have attracted the attention of researchers [5–8]. For example, Zhong et al. reported on design, synthesis and anti-inflammatory activity of novel pyrazole amines derivatives [9, 10]. Qiao et al. studied the synthesis, crystal structure, antifungal activity, and molecular docking of difluoromethylpyrazole derivatives [11]. Sun et al. discovered that novel pyrazole amides could be used as promising candidate drugs for plant fungicides [12]. Many of these reports contain structures of difluoromethylpyrazolamide, and similar structural fungicides have also been launched in recent years. So In this paper, we use 4-(difluoromethyl)-1-methyl-1*H*-pyrazole-4-carboxylic acid as the raw material to design and synthesize 3-(difluoromethyl)-1-methyl-*N*-(pyridin-2-yl)-1*H*-pyrazole-4-carboxylamide.

In the molecule of the title compound, bond lengths and angles are very similar to those given in the literature [9, 10, 13]. The dihedral angle of the pyrazole ring and the pyridine ring plane is 3.60(7)°. The torsion angles of C8–C7–C6–O1, C8–C7–C6–N2, C7–C6–N2–C5, and C6–N2–C5–C4 are 1.7(3)°, −179.3(2)°, −175.2(2)°, and −7.4(3)°, respectively. Intermolecular N–H···N hydrogen bonds between the nitrogen atom (N3) of the amide group and the nitrogen atom (N2) of the pyrazole ring linked the molecules in a one-dimensional chain structure (lower part of the figure).

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**Conflict of interest statement:** The authors declare no conflicts of interest regarding this article.

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