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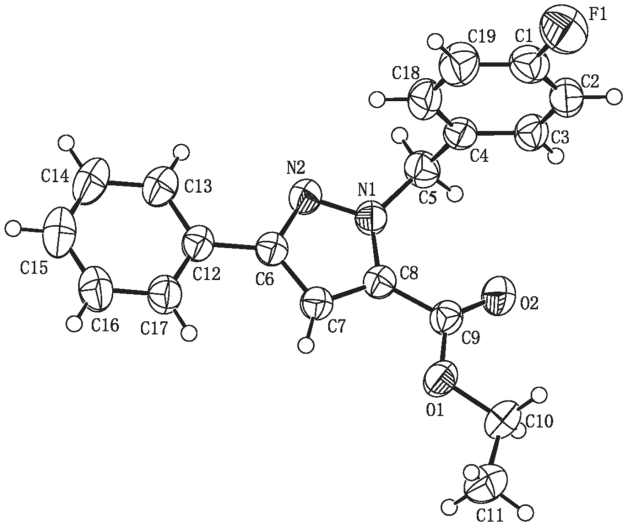
Crystal structure of ethyl 1-(4-fluorobenzyl)-3-phenyl-1*H*-pyrazole-5-carboxylate, $C_{19}H_{17}FN_2O_2$



https://doi.org/10.1515/ncrs-2017-0266
Received September 4, 2017; accepted January 18, 2018; available online February 7, 2018

Abstract

$C_{19}H_{17}FN_2O_2$, triclinic, $P\bar{1}$ (no. 2), $a = 7.8593(19)$ Å, $b = 10.3322(18)$ Å, $c = 10.9747(19)$ Å, $\alpha = 108.914(18)°$, $\beta = 92.931(3)°$, $\gamma = 99.544(3)°$, $V = 826.1(3)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0431$, $wR_{ref}(F^2) = 0.1170$, $T = 296.15$ K.

CCDC no.: 1817865

The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

A mixture of methyl 3-phenyl-1*H*-pyrazole-5-carboxylate (2.02 g, 0.01 mol), 1-(chloromethyl)-4-fluorobenzene (1.44 g, 0.01 mmol) and K_2CO_3 (1.38 g, 0.01 mmol) in acetonitrile (25 mL) was refluxed for 3 h. The resulting solution was concentrated by vacuum evaporation and the residue was purified by column chromatography on silica gel using ethyl acetate and petroleum ether (1:5, *v*/*v*) as eluent, to obtain the target compound in 78% yields. Crystals suitable for X-ray diffraction were obtained by slow evaporation of a solution in ethanol at room temperature.

Table 1: Data collection and handling.

Crystal:	Block, colorless
Size:	0.28 × 0.24 × 0.16 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.09 mm^{-1}
Diffractometer, scan mode:	Bruker P4, φ and ω-scans
θ_{max}, completeness:	27,5°, >92% (>99% up to 25°)
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	6650, 3524, 0.014
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2739
$N(param)_{refined}$:	218
Programs:	Bruker programs [1], SHELX [2, 3]

Experimental details

All H atoms were placed in idealized positions and treated as riding on their parent atoms, with d(C—H) = 0.96 (methyl) and 0.97 Å (methylene), $U_{iso}(H) = 1.5U_{eq}(C)$ and d(C—H) = 0.93 Å (aromatic), $U_{iso}(H) = 1.2U_{eq}(C)$.

Discussion

In recent years, much effort has been focused on the pyrazole derivatives due to their wide range of biological properties such as antiviral, anti-bacterial, anti-inflammatory, analgesic activities [4–7]. Pyrazoles were also found to possess inhibitory activities against xanthine oxidase, cyclooxygenase, and alkaline phosphatases [8–10]. The modification of pyrazole such as substituent moiety should provide potential biological activities [11, 12]. In continuation of interest in the development of pyrazole derivatives, we report here the crystal structure of ethyl 1-(4-fluorobenzyl)-3-phenyl-1*H*-pyrazole-5-carboxylate.

In the title crystal structure, all bond lengths and angles are within normal ranges [13]. The pyrazole ring

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
F1	−0.01323(14)	−0.58034(13)	−0.21251(12)	0.0901(4)
O1	−0.64722(14)	−1.23471(11)	−0.23397(10)	0.0546(3)
O2	−0.65599(16)	−1.09658(12)	−0.35449(10)	0.0631(3)
N1	−0.74054(15)	−0.89038(12)	−0.12490(11)	0.0445(3)
N2	−0.78245(15)	−0.82174(12)	−0.00687(11)	0.0478(3)
C1	−0.1830(2)	−0.63934(18)	−0.21325(18)	0.0607(4)
C2	−0.2740(2)	−0.72360(17)	−0.32873(15)	0.0565(4)
H2	−0.2223	−0.7409	−0.4053	0.068*
C3	−0.4450(2)	−0.78248(16)	−0.32874(14)	0.0511(4)
H3	−0.5088	−0.8409	−0.4064	0.061*
C4	−0.52324(19)	−0.75619(14)	−0.21538(13)	0.0456(3)
C5	−0.7127(2)	−0.81596(16)	−0.21714(15)	0.0515(4)
H5A	−0.7774	−0.7409	−0.1966	0.062*
H5B	−0.7572	−0.8794	−0.3037	0.062*
C6	−0.78467(16)	−0.91048(14)	0.05959(13)	0.0418(3)
C7	−0.74270(17)	−1.03579(14)	−0.01670(13)	0.0434(3)
H7	−0.7356	−1.1136	0.0069	0.052*
C8	−0.71420(17)	−1.01971(14)	−0.13383(13)	0.0418(3)
C9	−0.66953(18)	−1.11765(15)	−0.25349(13)	0.0450(3)
C10	−0.6040(2)	−1.34105(16)	−0.34517(15)	0.0589(4)
H10A	−0.4985	−1.3049	−0.3751	0.071*
H10B	−0.6972	−1.3712	−0.4156	0.071*
C11	−0.5793(3)	−1.45924(19)	−0.3017(2)	0.0777(5)
H11A	−0.5523	−1.5329	−0.3731	0.117*
H11B	−0.6840	−1.4930	−0.2710	0.117*
H11C	−0.4857	−1.4283	−0.2330	0.117*
C12	−0.82513(17)	−0.86888(15)	0.19470(13)	0.0447(3)
C13	−0.8751(2)	−0.74226(17)	0.25307(16)	0.0572(4)
H13	−0.8883	−0.6838	0.2056	0.069*
C14	−0.9056(2)	−0.7029(2)	0.38276(17)	0.0695(5)
H14	−0.9373	−0.6174	0.4220	0.083*
C15	−0.8892(2)	−0.7896(2)	0.45337(17)	0.0704(5)
H15	−0.9096	−0.7625	0.5399	0.084*
C16	−0.8429(2)	−0.9155(2)	0.39602(16)	0.0663(5)
H16	−0.8329	−0.9745	0.4434	0.080*
C17	−0.8108(2)	−0.95507(17)	0.26763(15)	0.0549(4)
H17	−0.7791	−1.0408	0.2295	0.066*
C18	−0.4251(2)	−0.67022(18)	−0.10037(15)	0.0623(4)
H18	−0.4756	−0.6519	−0.0233	0.075*
C19	−0.2534(2)	−0.6114(2)	−0.09867(18)	0.0722(5)
H19	−0.1876	−0.5542	−0.0214	0.087*

(N1/N2/C9/C8/C7) and the aryl ring (C1/C2/C3/C4/C5/C6) are almost coplanar with a dihedral angle of 6.0°. The dihedral angle between the pyrazole ring (N1/N2/C9/C8/C7) and the aryl moiety (C12/C13/C14/C15/C16/C17) is 81.9°. The two aryl moieties are twisted by an angle of 87.3°.

Acknowledgements: This work was supported by Project of Science and Technology Department of Zhejiang Province of China (no. 2017C33098).

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