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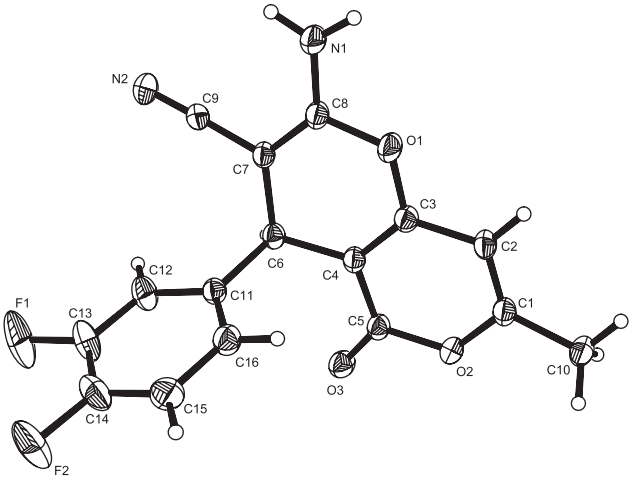
Crystal structure of 2-amino-7-methyl-4-(3,4-difluoro-phenyl)-5-oxo-4*H*,5*H*-pyrano[4,3-*b*]pyran-3-carbonitrile, $C_{16}H_{10}F_2N_2O_3$



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Abstract

$C_{16}H_{10}F_2N_2O_3$, monoclinic, $P2_1/c$ (no. 14), $a = 9.4647(7)$ Å, $b = 11.8726(7)$ Å, $c = 12.5732(8)$ Å, $\beta = 92.262(4)°$, $V = 1411.76(16)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0459$, $wR_{ref}(F^2) = 0.1285$, $T = 293$ K.

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

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Table 1: Data collection and handling.

Crystal:	Colourless blocks Size 0.20 × 0.18 × 0.17 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	1.2 cm^{-1}
Diffractometer, scan mode:	Bruker P4, ω
$2\theta_{max}$, completeness:	50°, >98%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	8227, 2452, 0.012
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2251
$N(param)_{refined}$:	208
Programs:	SHELX [6], Bruker programs [7]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.22244(17)	0.00642(15)	0.55569(13)	0.0375(4)
C2	0.32215(18)	0.08493(15)	0.54663(13)	0.0391(4)
H2A	0.3529	0.1274	0.6052	0.047*
C3	0.38022(17)	0.10216(14)	0.44535(12)	0.0347(4)
C4	0.33914(17)	0.04145(14)	0.35849(12)	0.0330(4)
C5	0.23653(17)	−0.04615(14)	0.37082(12)	0.0348(4)
C6	0.39481(16)	0.06182(14)	0.24986(12)	0.0325(4)
H6A	0.4317	−0.0098	0.2239	0.039*
C7	0.51783(17)	0.14392(14)	0.26144(12)	0.0340(4)
C8	0.55089(17)	0.20203(14)	0.35169(12)	0.0353(4)
C9	0.60416(17)	0.15720(15)	0.17294(13)	0.0371(4)
C10	0.1437(2)	−0.02112(18)	0.65199(14)	0.0476(5)
H10A	0.1760	0.0263	0.7098	0.071*
H10B	0.1596	−0.0986	0.6708	0.071*
H10C	0.0445	−0.0089	0.6377	0.071*
C11	0.28125(17)	0.10314(14)	0.16882(12)	0.0341(4)
C12	0.3005(2)	0.08279(17)	0.06166(14)	0.0487(5)
H12A	0.3785	0.0421	0.0406	0.058*
C13	0.2038(3)	0.1230(2)	−0.01291(15)	0.0619(6)
C14	0.0854(2)	0.18031(19)	0.01723(18)	0.0610(6)
C15	0.0643(2)	0.20137(19)	0.12219(18)	0.0565(5)
H15A	−0.0153	0.2404	0.1427	0.068*
C16	0.16383(19)	0.16343(16)	0.19746(14)	0.0438(4)
H16A	0.1514	0.1789	0.2690	0.053*
O1	0.47953(13)	0.18546(11)	0.44358(9)	0.0437(3)

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
O2	0.18092(12)	−0.05863(10)	0.47041(9)	0.0404(3)
O3	0.19199(13)	−0.10951(11)	0.30186(10)	0.0450(3)
N1	0.65170(17)	0.27926(13)	0.36867(11)	0.0464(4)
H1A	0.7059	0.2977	0.3182	0.056*
H1B	0.6625	0.3106	0.4301	0.056*
N2	0.67250(18)	0.16320(16)	0.10007(12)	0.0537(4)
F1	0.2254(2)	0.10824(16)	−0.11733(10)	0.1076(7)
F2	−0.00792(18)	0.21701(17)	−0.05935(13)	0.1013(6)

Source of material

The title compound was synthesized according to a reported procedure [6]. A mixture of 4-hydroxy-6-methylpyran-2-one (10 mmol), 3,4-difluorobenzaldehyde (10 mmol), malononitrile (10 mmol) and 4-(dimethylamino)pyridine (DMAP) (1 mmol) in ethanol (100 mL) was refluxed for 2–3 h and then cooled to room temperature. After filtering the precipitates, they were sequentially washed with icy water and ethanol and then dried under a vacuum.

Experimental details

H atoms bonded to C and N were positioned geometrically and refined using a riding model, with C—H = 0.93 Å and N—H = 0.86 Å with $U_{iso}(H) = 1.2$ times $U_{eq}(C)$ or $U_{eq}(N)$.

Discussion

Pyrans have a variety of pharmacological and biological activities including antitumor, analgesic and ulcerogenic, anti-inflammatory, anticoagulant, phototriggering, and fungicidal properties, moreover, they can be used as anticoagulants in the production of pesticides [1, 2]. In particular, the antitumor activity of pyran compounds has received considerable attention because of their cytotoxic activity against numerous types of cancers, including malignant melanoma, leukemia, renal cell carcinoma, prostate and breast cancer cell progression [3–5].

In the crystal structure of the title compound, the 4*H*-pyran ring is nearly planar and the adjacent ring with the keto group adopts an almost planar conformation. The 4*H*-pyran ring plane is almost perpendicular to the plane of the difluorophyenyl moiety. All bond lengths and angles are in the expected ranges.

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