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Crystal structure of ethyl 2-amino-4-(3,5-difluorophenyl)-7-methyl-5-oxo-4*H*,5*H*-pyrano [4,3-*b*]pyran-3-carboxylate, C₁₈H₁₅F₂NO₅

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.26 × 0.21 × 0.17 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.12 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω -scans
$\theta_{\max}$, completeness:	25°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	8083, 2916, 0.066
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2106
$N(\text{param})_{\text{refined}}$:	246
Programs:	Bruker programs [1], SHELX [2], OLEX2 [3]

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Abstract

C₁₈H₁₅F₂NO₅, monoclinic, $C2/c$ (no. 15), $a = 26.896(3)$ Å, $b = 9.005(3)$ Å, $c = 13.737(2)$ Å, $\beta = 94.971(11)^\circ$, $V = 3314.6(13)$ Å³, $Z = 8$, $R_{\text{gt}}(F) = 0.0560$, $wR_{\text{ref}}(F^2) = 0.1823$, $T = 293(2)$ K.

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The asymmetric unit of the title 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

The title compound was synthesized according to a reported procedure. A mixture of 4-hydroxy-6-methyl-pyran-2-one (10 mmol), 3,5-difluorobenzaldehyde (10 mmol), ethyl cyanoacetate (10 mmol) and 4-(dimethylamino)pyridine

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.19318(9)	0.4103(2)	0.08368(16)	0.0518(6)
H1A	0.164629	0.375518	0.061548	0.062*
H1B	0.217637	0.350783	0.098904	0.062*
O1	0.08515(7)	0.73459(19)	0.01380(14)	0.0549(5)
O2	0.10041(7)	0.4908(2)	0.02594(15)	0.0593(6)
O3	0.24721(6)	0.58577(16)	0.13100(12)	0.0426(5)
O4	0.22800(7)	1.10690(19)	0.11529(15)	0.0564(6)
O5	0.30097(6)	1.00665(16)	0.16085(13)	0.0438(5)
F2	0.04341(6)	1.1446(2)	0.19192(15)	0.0832(7)
F1	0.12534(8)	0.8318(2)	0.42355(13)	0.0888(7)
C2	0.03283(12)	0.7025(4)	−0.0095(3)	0.0795(10)
C1 ^a	0.0085(2)	0.7000(9)	0.0864(6)	0.114(3)
H1C ^a	−0.026480	0.678923	0.073644	0.170*
H1D ^a	0.023842	0.624666	0.128265	0.170*
H1E ^a	0.012650	0.795035	0.117757	0.170*
C1A ^b	0.0010(5)	0.813(2)	0.0342(14)	0.090(7)
H1AA ^b	−0.033452	0.789341	0.017623	0.134*
H1AB ^b	0.007876	0.812583	0.103963	0.134*
H1AC ^b	0.007948	0.910152	0.009462	0.134*
C3	0.11522(9)	0.6174(3)	0.03645(18)	0.0425(6)
C4	0.16462(9)	0.6636(2)	0.07359(17)	0.0374(6)
C5	0.19950(9)	0.5557(3)	0.09433(16)	0.0378(6)
C6	0.26213(9)	0.7309(2)	0.14032(16)	0.0358(6)
C7	0.23065(8)	0.8453(2)	0.12167(16)	0.0339(6)
C8	0.17600(8)	0.8264(2)	0.09280(17)	0.0362(6)
H8	0.167800	0.881257	0.031910	0.043*
C9	0.25060(9)	0.9933(3)	0.13071(19)	0.0406(6)
C10	0.33115(9)	0.8870(3)	0.18050(17)	0.0424(6)
C11	0.31316(9)	0.7498(3)	0.17096(18)	0.0419(6)

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Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
H11	0.333774	0.668048	0.184190	0.050*
C12	0.14462(8)	0.8899(2)	0.17075(17)	0.0365(6)
C13	0.15085(10)	0.8349(3)	0.26540(18)	0.0439(6)
H13	0.175638	0.765765	0.283497	0.053*
C14	0.11943(11)	0.8855(3)	0.33137(19)	0.0526(7)
C15	0.08313(11)	0.9880(3)	0.3105(2)	0.0591(8)
H15	0.062431	1.020155	0.357033	0.071*
C16	0.07874(9)	1.0414(3)	0.2170(2)	0.0525(7)
C17	0.10899(9)	0.9963(3)	0.1471(2)	0.0446(6)
H17	0.105430	1.037206	0.084744	0.054*
C18	0.38241(10)	0.9326(3)	0.2124(2)	0.0561(7)
H18A	0.384545	1.039029	0.212294	0.084*
H18B	0.391328	0.896068	0.277208	0.084*
H18C	0.404881	0.892555	0.168514	0.084*

Occupancies: ^a = 0.770(12), ^b = 0.230(12)

(DMP) (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 ice-cooled water and ethanol and then dried under a vacuum.

Experimental details

The H atoms bonded to C and N were positioned geometrically and refined using a riding model, with C–H = 0.93 Å and 0.86 Å and with *U*_{iso}(H) = 1.2 times *U*_{eq}(C) and *U*_{eq}(N). There is a disorder of the ethyl group (*cf.* the figure; Table 2).

Discussion

The chemistry and diverse applications of organic heterocyclic derivatives for example coumarin have received much attention due to their pronounced biological activity [4–7]. Also some pyrans are well known to exhibit various biological activities.

In the crystal structure of the title compound (*cf.* the figure), only one methyl substituent is located at the oxo-pyrano moiety. The 3,5-difluorophenyl substituent, an ethyl ester group and one amino group are linked to the pyran ring of the 4*H*,5*H*-pyrano[4,3-*b*]pyran core. Angles, torsion angles and bond lengths fall in their normal ranges compared with those in previously studies.

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