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Crystal structure of ethyl 2-amino-4-(3,4-difluorophenyl)-5-oxo-4*H*,5*H*-pyrano[3,2-*c*]chromene-3-carboxylate, C₂₁H₁₅F₂NO₅

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
$\theta_{\max}$, completeness:	27.5°, 99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	5602, 4062, 0.032
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1708
$N(\text{param})_{\text{refined}}$:	284
Programs:	Olex2 [1], SHELX [2], Bruker [3]

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O5	0.7686(4)	1.0148(2)	0.08040(13)	0.0589(7)
O3	0.1828(4)	1.0571(2)	0.24301(12)	0.0516(6)
O4	0.9584(4)	0.8472(3)	0.15395(14)	0.0675(7)
O1	0.5998(6)	0.7874(3)	0.43994(14)	0.0892(10)
F1	0.6127(5)	0.2774(2)	0.24229(16)	0.0991(9)
N1	−0.0049(5)	1.0476(3)	0.36248(17)	0.0649(9)
H1A	−0.0250(5)	1.0207(3)	0.41185(4)	0.078*
H1B	−0.1144(6)	1.1053(5)	0.3412(5)	0.078*
F2	0.9569(5)	0.3639(2)	0.30888(17)	0.1086(10)
O2	0.2346(6)	0.8978(3)	0.47760(16)	0.1017(11)
C1 ^a	0.817(3)	0.6298(15)	0.5240(7)	0.122(5)
H1C ^a	0.8095(3)	0.5553(18)	0.4894(6)	0.184*
H1D ^a	0.8349(4)	0.5911(6)	0.5772(3)	0.184*
H1E ^a	0.9504(5)	0.6765(7)	0.5057(2)	0.184*
C2 ^a	0.589(2)	0.7344(18)	0.5236(5)	0.092(5)
H2A ^a	0.4490(8)	0.6887(9)	0.5377(3)	0.110*
H2B ^a	0.5880(6)	0.8092(8)	0.5595(4)	0.110*
C3	0.3942(9)	0.8676(4)	0.4250(2)	0.0716(12)
C4	0.3859(6)	0.9099(3)	0.34147(18)	0.0511(9)
C5	0.1956(6)	1.0000(3)	0.31898(18)	0.0497(8)
C7	0.5897(6)	0.8539(3)	0.28247(18)	0.0484(8)
H7	0.7389(4)	0.8642(3)	0.3041(8)	0.058*
C8	0.5741(6)	0.9426(3)	0.20554(17)	0.0428(8)
C9	0.3809(6)	1.0354(3)	0.19060(18)	0.0434(8)
C10	0.3688(6)	1.1239(3)	0.11921(17)	0.0458(8)
C11	0.5706(6)	1.1115(3)	0.06592(19)	0.0510(8)
C12	0.5842(7)	1.1948(4)	−0.0034(2)	0.0638(10)
H12	0.7215(8)	1.1854(6)	−0.0385(5)	0.077*
C13	0.3896(9)	1.2913(4)	−0.0189(2)	0.0760(12)
H13	0.3954(7)	1.3489(6)	−0.0650(5)	0.091*

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Abstract

C₁₈H₂₀BrNO₃S, triclinic, $P\bar{1}$, $a = 5.699(3)$ Å, $b = 9.629(4)$ Å, $c = 16.888(2)$ Å, $\alpha = 86.51(3)^\circ$, $\beta = 84.31(3)^\circ$, $\gamma = 81.06(3)^\circ$, $V = 909.9(7)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0643$, $wR_{\text{ref}}(F^2) = 0.2270$, $T = 296(2)$ K.

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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 4-hydroxycoumarin (10 mmol), 3,4-difluorobenzaldehyde (10 mmol), ethyl cyanoacetate (10 mmol) and 4-(dimethylamino)pyridine (DMAP) (1 mmol) in ethanol

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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}
C14	0.1844(8)	1.3049(4)	0.0328(2)	0.0701(11)
H14	0.053065	1.370406	0.021033	0.084*
C15	0.7780(6)	0.9282(3)	0.14793(19)	0.0509(8)
C16	0.5951(6)	0.6988(3)	0.26863(17)	0.0438(8)
C17	0.4179(6)	0.6529(3)	0.23298(19)	0.0548(9)
H17	0.293280	0.717835	0.215319	0.066*
C18	0.4218(7)	0.5113(4)	0.2229(2)	0.0660(10)
H18	0.301166	0.480768	0.198420	0.079*
C19	0.6028(7)	0.4175(3)	0.2491(2)	0.0642(10)
C20	0.7792(7)	0.4612(3)	0.2839(2)	0.0618(10)
C21	0.7784(6)	0.6004(3)	0.29394(19)	0.0547(9)
H21	0.901584	0.629333	0.317909	0.066*
C6	0.1753(7)	1.2224(3)	0.10076(19)	0.0575(9)
H6	0.037056	1.232043	0.135469	0.069*
C1AA ^b	0.620(4)	0.6145(15)	0.5398(8)	0.121(7)
H1AA ^b	0.682089	0.550450	0.498761	0.182*
H1AB ^b	0.449276	0.622289	0.546433	0.182*
H1AC ^b	0.684027	0.579837	0.588966	0.182*
COAA ^b	0.690(3)	0.7559(17)	0.5170(8)	0.089(6)
HOAA ^b	0.615788	0.825040	0.554822	0.107*
HOAB ^b	0.861279	0.752224	0.513422	0.107*

^aOccupancy: 0.555(15), ^bOccupancy: 0.445(15).

(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

H atoms were positioned geometrically and refined using a riding model, with C–H = 0.93/0.96/0.97 Å and N–H = 0.86 Å with *U*_{iso}(H) = 1.2 times *U*_{eq}(C) and 1.2 times *U*_{eq}(N).

Comment

Coumarins possess diverse pharmacological and biological activities. In particular, the antitumor activity of coumarin compounds has received considerable attention among researchers because of [4, 5].

The molecular structural unit of the title compound is shown in the figure. The bond lengths and angles presented in the title compound are all in their normal ranges. The features of the title compound are comparable with those in the compound ethyl 2-amino-4-(2-methoxyphenyl)-5-oxo-4*H*,5*H*-pyrano[3,2-*c*]chromene-3-carboxylate [6].

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