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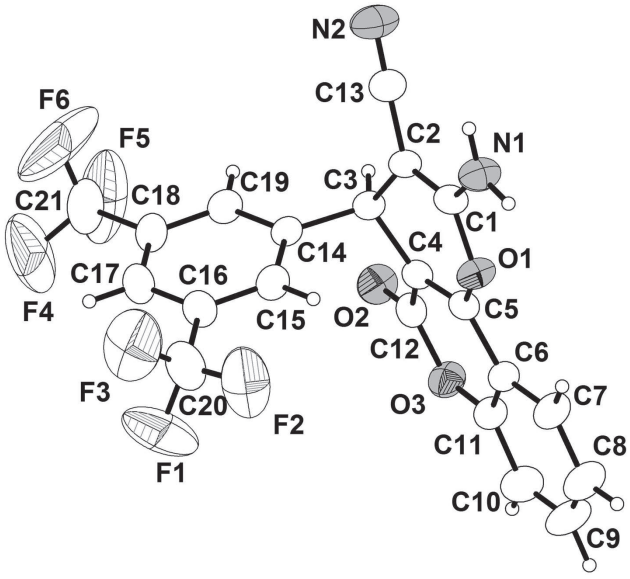
Crystal structure of 2-amino-5-oxo-4-(3,5-bis(trifluoromethyl)phenyl)-4*H*,5*H*-pyrano[3,2-*c*]chromene-3-carbonitrile, $C_{21}H_{10}F_6N_2O_3$



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Abstract

$C_{21}H_{10}F_6N_2O_3$, monoclinic, $P2_1/c$ (no. 14), $a = 11.8113(7)$ Å, $b = 7.8929(3)$ Å, $c = 21.4129(18)$ Å, $\beta = 105.012(7)°$, $V = 1928.1(2)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0632$, $wR_{ref}(F^2) = 0.1347$, $T = 293(2)$ K.

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The 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.

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:	1.4 cm^{-1}
Diffractometer, scan mode:	Bruker FRAMBO, φ and ω
$2\theta_{max}$, completeness:	50.4°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	7293, 3389, 0.026
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2040
$N(param)_{refined}$:	290
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	0.4038(3)	0.5813(4)	0.34440(15)	0.0349(8)
C2	0.4186(3)	0.7272(4)	0.37805(14)	0.0335(7)
C3	0.3737(3)	0.8947(4)	0.34730(14)	0.0329(7)
H3A	0.4303	0.9840	0.3654	0.040*
O3	0.3296(2)	1.0172(3)	0.17218(10)	0.0479(6)
C4	0.3624(3)	0.8800(4)	0.27595(14)	0.0317(7)
C5	0.3524(3)	0.7299(4)	0.24608(14)	0.0324(7)
C6	0.3272(3)	0.7130(4)	0.17705(15)	0.0367(8)
C7	0.3145(3)	0.5585(5)	0.14430(17)	0.0505(10)
H7A	0.3259	0.4572	0.1673	0.061*
C9	0.2670(4)	0.7082(6)	0.04408(19)	0.0719(13)
H9A	0.2450	0.7058	−0.0009	0.086*
C8	0.2852(4)	0.5571(6)	0.07781(19)	0.0656(12)
H8A	0.2775	0.4548	0.0556	0.079*
C10	0.2806(3)	0.8614(6)	0.07519(17)	0.0606(11)
H10A	0.2692	0.9623	0.0520	0.073*
C13	0.4735(3)	0.7213(4)	0.44501(16)	0.0417(8)
C12	0.3579(3)	1.0326(4)	0.23830(16)	0.0387(8)
C11	0.3118(3)	0.8621(5)	0.14206(16)	0.0421(8)
C14	0.2539(3)	0.9412(4)	0.35809(14)	0.0329(7)
C15	0.1575(3)	0.8369(4)	0.33414(16)	0.0435(9)
H15A	0.1669	0.7368	0.3131	0.052*
C16	0.0484(3)	0.8795(5)	0.34105(17)	0.0493(9)
C17	0.0334(3)	1.0271(5)	0.37282(18)	0.0550(10)
H17A	−0.0402	1.0561	0.3776	0.066*
C18	0.1280(3)	1.1303(4)	0.39729(17)	0.0487(9)
C21	0.1109(5)	1.2895(6)	0.4309(3)	0.0829(15)

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Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
C20	−0.0550(4)	0.7714(7)	0.3132(3)	0.0818(15)
O1	0.36109(19)	0.5793(3)	0.27819(10)	0.0406(6)
O2	0.3770(2)	1.1743(3)	0.25990(12)	0.0524(7)
N1	0.4249(2)	0.4225(3)	0.36526(13)	0.0482(8)
H1A	0.4522	0.4025	0.4059	0.058*
H1B	0.4112	0.3400	0.3381	0.058*
C19	0.2374(3)	1.0874(4)	0.38999(15)	0.0415(8)
H19A	0.3009	1.1583	0.4069	0.050*
N2	0.5196(3)	0.7181(4)	0.49902(15)	0.0644(10)
F1	−0.1329(3)	0.8528(5)	0.26723(19)	0.1527(16)
F2	−0.0311(2)	0.6310(4)	0.28772(19)	0.1421(16)
F3	−0.1126(3)	0.7297(5)	0.35460(19)	0.1388(14)
F4	0.0086(3)	1.3501(5)	0.4135(2)	0.176(2)
F5	0.1755(4)	1.4141(4)	0.4201(2)	0.1686(19)
F6	0.1354(6)	1.2770(5)	0.49065(18)	0.235(3)

Source of material

A mixture of 3,5-bis-trifluoromethyl-benzaldehyde (10 mmol) and 4-hydroxycoumarin (20 mmol) was dissolved in 100 mL of EtOH. A few drops of piperidine were added, and the mixture was stirred for 3 h at room temperature. After reaction completion as determined by TLC, water was added until precipitation occurred. After filtering the precipitates, they were sequentially washed with ice-cooled water and ethanol and then dried in a vacuum.

Experimental details

Carbon-bound and Nitrogen-bound hydrogen atoms were placed in calculated positions and were included in the refinement in the riding model approximation, with U_{iso}(H) set to 1.2U_{eq}(C).

Discussion

The synthesis of pyran and their derivatives has attracted considerable attention from organic and medicinal chemists for many years as a large number of natural and synthetic products contain this heterocyclic nucleus [1]. Pyrans possess diverse pharmacological and biological activities such as antitumor, analgesic and ulcerogenic, anti-inflammatory, anticoagulant, phototriggering, and fungicidal properties, and can act as anticoagulants in the production of pesticides [2, 3]. In particular, the antitumor activity of pyran compounds has received considerable attention among researchers because of their cytotoxic activity against numerous types of cancers, including malignant melanoma, leukemia, renal cell carcinoma, prostate and breast cancer cell progression [4, 5].

In the crystal structures of the title compound, the new formed pyran ring and the adjacent coumarin ring are both basically planar, and the two planes are also essentially parallel to each other. However, the phenyl ring makes a torsion angle to the pyran ring in the compound.

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