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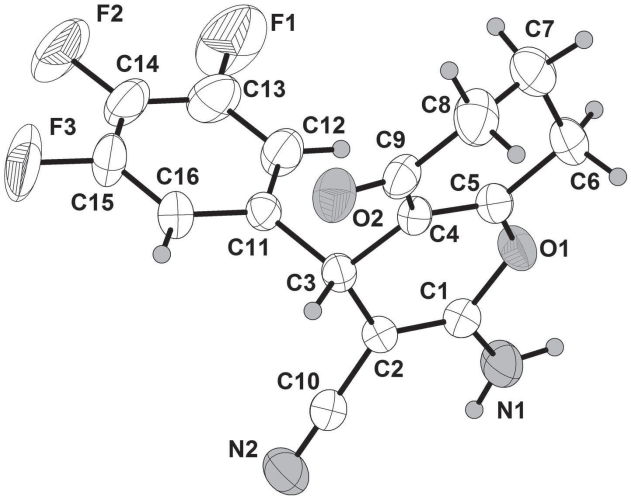
Crystal structure of 2-amino-4-(3,4,5-trifluorophenyl)-5-oxo-5,6,7,8-tetrahydro-4*H*-chromene-3-carbonitrile, $C_{16}H_{11}F_3N_2O_2$



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Abstract

$C_{16}H_{11}F_3N_2O_2$, triclinic, $P\bar{1}$ (No. 2), $a = 8.3288(5)$ Å, $b = 8.6385(8)$ Å, $c = 11.6004(8)$ Å, $\alpha = 111.488(7)°$, $\beta = 94.705(6)°$, $\gamma = 99.107(6)°$, $V = 757.87(10)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0435$, $wR_{ref}(F^2) = 0.0954$, $T = 293(2)$ K.

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The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details 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, block
	Size 0.20 × 0.18 × 0.17 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.2 cm^{-1}
Diffractometer, scan mode:	Bruker FRAMBO, φ and ω
$2\theta_{max}$, completeness:	50°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	4859, 2654, 0.018
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1998
$N(param)_{refined}$:	209
Programs:	SHELX [7], Bruker programs [8]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O1	0.48780(16)	0.40895(17)	0.31360(14)	0.0463(4)
O2	1.05477(18)	0.4175(2)	0.35629(16)	0.0587(4)
C3	0.8135(2)	0.6104(2)	0.35036(17)	0.0351(4)
H3A	0.9062	0.6643	0.4190	0.042*
C2	0.6647(2)	0.6809(2)	0.39365(17)	0.0366(4)
C4	0.7764(2)	0.4226(2)	0.32121(18)	0.0367(4)
C10	0.6822(2)	0.8582(3)	0.45636(19)	0.0426(5)
C5	0.6230(2)	0.3340(2)	0.29954(19)	0.0405(5)
C11	0.8612(2)	0.6444(2)	0.23621(18)	0.0365(4)
N1	0.3722(2)	0.6309(2)	0.39549(18)	0.0547(5)
H1A	0.3707	0.7371	0.4315	0.066*
H1B	0.2824	0.5562	0.3760	0.066*
C9	0.9140(3)	0.3369(3)	0.3229(2)	0.0454(5)
C1	0.5133(2)	0.5822(2)	0.37001(18)	0.0399(5)
N2	0.6974(2)	1.0025(2)	0.5071(2)	0.0621(6)
C6	0.5738(3)	0.1470(3)	0.2577(2)	0.0542(6)
H6A	0.4765	0.1055	0.1944	0.065*
H6B	0.5470	0.1181	0.3281	0.065*
C16	1.0141(3)	0.7360(3)	0.2405(2)	0.0506(6)
H16A	1.0909	0.7771	0.3131	0.061*
F2	0.9825(3)	0.7442(3)	−0.06930(17)	0.1153(7)
C12	0.7503(3)	0.5854(3)	0.1275(2)	0.0547(6)
H12A	0.6462	0.5234	0.1229	0.066*
C8	0.8746(3)	0.1492(3)	0.2888(3)	0.0673(7)
H8A	0.8718	0.1273	0.3649	0.081*
H8B	0.9618	0.1001	0.2477	0.081*

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
F1	0.6855(3)	0.5624(3)	−0.08032(16)	0.1287(9)
C15	1.0526(3)	0.7662(3)	0.1370(3)	0.0670(7)
F3	1.2013(2)	0.8560(3)	0.1397(2)	0.1274(8)
C7	0.7129(3)	0.0627(3)	0.2038(3)	0.0697(8)
H7A	0.6888	−0.0559	0.1927	0.084*
H7B	0.7210	0.0676	0.1222	0.084*
C14	0.9440(4)	0.7100(3)	0.0303(3)	0.0664(7)
C13	0.7936(3)	0.6183(4)	0.0261(2)	0.0668(7)

Source of material

The title compound was synthesized according to a reported procedure [6]. A mixture of 3,5-cyclohexanedione (10 mmol), 3,4,5-trifluorobenzaldehyde (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 ice-cooled water and ethanol and then dried under 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 growing interest in bioactive pyrans has led to an increasing demand for efficient syntheses of this class of heterocyclic compounds [1]. Several reports have found diverse applications for pyrans in medicine such as antitumor (including breast cancer and liver cancer), antiviral (including hepatitis B virus and influenza virus), anti-inflammatory agents, or kinase inhibitors for the treatment of type 2 diabetes, hyperlipidemia, and obesity [2, 3]. Moreover, these compounds have remarkable potential in nanomedicine against malignant gliomas [4, 5].

In the crystal structure of the title compound, the pyran ring is nearly planar whereas the ring containing the keto group is folded. The plane of the pyran ring is almost perpendicular to the plane of the triflorophenyl moiety. All bond lengths and angles are in the expected ranges.

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