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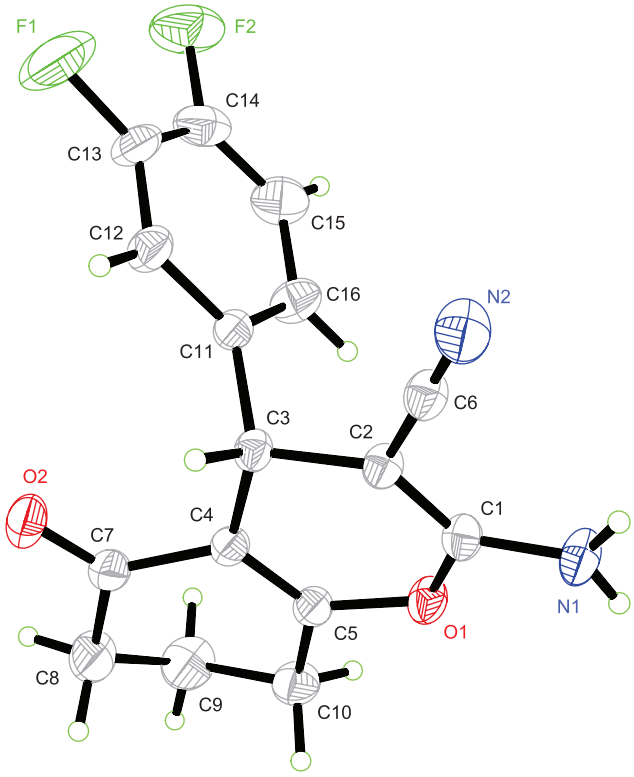
Crystal structure of 2-amino-4-(3,4-difluorophenyl)-5-oxo-5,6,7,8-tetrahydro-4H-chromene-3-carbonitrile, $C_{16}H_{12}N_2O_2F_2$



DOI 10.1515/ncrs-2015-0097
Received May 15, 2015; accepted December 16, 2015; available online February 11, 2016

Abstract

$C_{16}H_{12}F_2N_2O_2$, monoclinic, $C2/c$ (no. 15), $a = 13.2109(11)$ Å, $b = 10.957(1)$ Å, $c = 20.2646(18)$ Å, $\beta = 101.411(9)°$, $V = 2875.4(4)$ Å^3, $Z = 8$, $R_{gt}(F) = 0.0529$, $wR_{ref}(F^2) = 0.1649$, $T = 293$ K.

The crystal structure is shown in the figure, Tables 1—3 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:	Colorless, prism, size 0.16×0.17×0.19 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.11 cm^{-1}
Diffractometer, scan mode:	Area detector, φ and ω scans
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5321, 2528
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1841
$N(param)_{refined}$:	200
Programs:	SHELX [6]

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

Atom	Site	x	y	z	U_{iso}
H(3A)	8*f*	0.1391	1.0562	0.4594	0.045
H(8A)	8*f*	−0.1702	0.8688	0.3585	0.091
H(8B)	8*f*	−0.1160	0.7951	0.4219	0.091
H(9A)	8*f*	−0.0690	0.7784	0.2929	0.092
H(9B)	8*f*	−0.1178	0.6744	0.3298	0.092
H(10A)	8*f*	0.0335	0.6394	0.4029	0.066
H(10B)	8*f*	0.0638	0.6493	0.3320	0.066
H(12A)	8*f*	0.1136	1.2509	0.4100	0.061
H(15A)	8*f*	0.1779	1.1156	0.2066	0.080
H(16A)	8*f*	0.1844	0.9811	0.2956	0.067
H(1A)	8*f*	0.4293	0.8221	0.4668	0.064
H(1B)	8*f*	0.3806	0.7073	0.4364	0.064

Source of material

The title compound was synthesized according to a reported procedure [5]. A mixture of 3,5-cyclohexanedione (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 ice-cooled water and ethanol and then dried under vacuum.

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Table 3: Atomic displacement parameters (Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	8*f*	0.2847(2)	0.8399(2)	0.4308(1)	0.036(1)	0.042(2)	0.041(1)	0.003(1)	0.004(1)	−0.003(1)
C(2)	8*f*	0.2629(2)	0.9571(2)	0.4427(1)	0.035(1)	0.035(1)	0.041(1)	0.002(1)	0.002(1)	−0.004(1)
C(3)	8*f*	0.1563(2)	1.0121(2)	0.4210(1)	0.037(1)	0.033(1)	0.042(1)	0.003(1)	0.008(1)	−0.004(1)
C(4)	8*f*	0.0801(2)	0.9099(2)	0.4027(1)	0.036(1)	0.035(1)	0.045(1)	0.001(1)	0.006(1)	0.004(1)
C(5)	8*f*	0.1083(2)	0.7964(2)	0.3909(1)	0.035(1)	0.037(1)	0.050(1)	−0.001(1)	0.005(1)	0.002(1)
C(6)	8*f*	0.3438(2)	1.0333(2)	0.4746(1)	0.041(1)	0.039(2)	0.046(1)	0.005(1)	0.005(1)	−0.006(1)
C(7)	8*f*	−0.0292(2)	0.9371(3)	0.4004(1)	0.039(1)	0.046(2)	0.063(2)	0.003(1)	0.010(1)	0.012(1)
C(8)	8*f*	−0.1047(2)	0.8351(3)	0.3813(2)	0.037(2)	0.057(2)	0.132(3)	−0.005(1)	0.012(2)	0.010(2)
C(9)	8*f*	−0.0694(2)	0.7420(3)	0.3365(2)	0.050(2)	0.059(2)	0.110(3)	−0.018(2)	−0.007(2)	−0.005(2)
C(10)	8*f*	0.0373(2)	0.6946(3)	0.3660(2)	0.054(2)	0.040(2)	0.071(2)	−0.009(1)	0.009(1)	−0.003(1)
C(11)	8*f*	0.1503(2)	1.1016(2)	0.3626(1)	0.030(1)	0.032(1)	0.049(1)	−0.0027(9)	0.001(1)	−0.001(1)
C(12)	8*f*	0.1270(2)	1.2225(2)	0.3694(1)	0.055(2)	0.036(2)	0.061(2)	0.004(1)	0.011(1)	−0.003(1)
C(13)	8*f*	0.1233(2)	1.3012(3)	0.3166(2)	0.062(2)	0.031(2)	0.089(2)	0.003(1)	0.009(2)	0.012(2)
C(14)	8*f*	0.1415(2)	1.2616(3)	0.2569(2)	0.060(2)	0.066(2)	0.064(2)	−0.014(2)	0.003(1)	0.022(2)
C(15)	8*f*	0.1649(2)	1.1426(3)	0.2477(2)	0.077(2)	0.069(2)	0.054(2)	−0.010(2)	0.009(2)	0.003(2)
C(16)	8*f*	0.1689(2)	1.0628(3)	0.3011(1)	0.070(2)	0.041(2)	0.055(2)	−0.000(1)	0.010(1)	−0.003(1)
O(1)	8*f*	0.2095(1)	0.7601(2)	0.40019(9)	0.0394(9)	0.033(1)	0.064(1)	0.0017(7)	0.0058(8)	−0.0095(8)
O(2)	8*f*	−0.0563(1)	1.0376(2)	0.4157(1)	0.049(1)	0.050(1)	0.105(2)	0.0141(9)	0.022(1)	0.005(1)
N(1)	8*f*	0.3757(2)	0.7830(2)	0.4465(1)	0.043(1)	0.044(1)	0.067(1)	0.011(1)	−0.002(1)	−0.009(1)
N(2)	8*f*	0.4092(2)	1.0964(2)	0.5002(1)	0.050(1)	0.054(2)	0.079(2)	−0.004(1)	0.000(1)	−0.018(1)
F(1)	8*f*	0.1039(2)	1.4199(2)	0.3230(2)	0.183(3)	0.048(1)	0.154(2)	0.024(1)	0.051(2)	0.024(1)
F(2)	8*f*	0.1385(2)	1.3436(2)	0.2062(1)	0.123(2)	0.095(2)	0.091(2)	−0.015(1)	0.012(1)	0.049(1)

Discussion

Dihydropyran and its derivatives have received considerable attention in the past few years for their versatile biological and medical activities. Different substituents at the aromatic ring lead to multiple chemical modifications which generates antioxidant, anti-inflammatory, antibacterial and anticancer activities for these compounds [1, 2]. Recognizing the considerable importance of the compounds, research focused on the synthesis of dihydropyran derivatives [3, 4].

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

Acknowledgements: This work was supported by grants from the Youth Fund Project of Shaanxi University of Chinese Medicine (No. 2015QN22).

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