He Lou*

Crystal structure of 2-amino-4-(3,5-difluorophenyl)-7,7-dimethyl-5-oxo-5,6,7,8-tetrahydro-4*H*-chromene-3-carbonitrile, C₁₈H₁₆F₂N₂O₂

Table 1: Data collection and handling.

Crystal:	Colourless prism
Wavelength:	Size 0.60 × 0.50 × 0.40 mm
μ :	Mo K α radiation (0.71073 Å)
Diffractometer, scan mode:	1.1 cm ⁻¹
2 θ _{max} , completeness:	Brucker P4, ω
$N(hkl)$ _{measured} , $N(hkl)$ _{unique} , R_{int} :	55°, >99%,
Criterion for I_{obs} , $N(hkl)$ _{gt} :	11299, 2734, 0.014
$N(param)$ _{refined} :	$I_{obs} > 2 \sigma(I_{obs})$, 2525
Programs:	217
	SHELX [7], Bruker programs [8]

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	U_{iso}^*/U_{eq}
C1	0.07097(12)	0.26250(14)	0.89375(10)	0.0338(3)
C2	0.12584(12)	0.30672(14)	0.82477(10)	0.0326(3)
C3	0.25652(12)	0.29796(14)	0.83116(10)	0.0332(3)
H3A	0.2743	0.2664	0.7708	0.040*
C4	0.29942(12)	0.18552(15)	0.90287(10)	0.0345(3)
C5	0.24004(12)	0.15389(15)	0.97376(11)	0.0350(3)
C6	0.40843(12)	0.12048(16)	0.90049(10)	0.0383(4)
C7	0.05618(13)	0.36614(16)	0.74153(11)	0.0380(3)
C8	−0.07389(13)	0.3501(2)	0.73189(12)	0.0481(4)
H8A	−0.0952	0.2581	0.7056	0.058*
H8B	−0.1100	0.4205	0.6884	0.058*
C9	−0.12304(12)	0.36518(17)	0.82315(11)	0.0403(4)
C10	−0.05699(12)	0.26439(16)	0.89490(11)	0.0398(4)
H10A	−0.0711	0.2918	0.9564	0.048*
H10B	−0.0874	0.1698	0.8830	0.048*
C11	−0.25227(14)	0.3250(2)	0.80660(15)	0.0600(5)
H11A	−0.2836	0.3344	0.8636	0.090*
H11B	−0.2605	0.2292	0.7853	0.090*
H11C	−0.2936	0.3864	0.7605	0.090*
C12	−0.10949(16)	0.51693(19)	0.85768(14)	0.0552(5)
H12A	−0.1402	0.5255	0.9150	0.083*
H12B	−0.1513	0.5788	0.8121	0.083*
H12C	−0.0285	0.5420	0.8676	0.083*
C13	0.31868(12)	0.43831(15)	0.85461(10)	0.0346(3)
C14	0.42725(13)	0.46141(17)	0.82902(12)	0.0469(4)
H14A	0.4613	0.3938	0.7954	0.056*
C15	0.48367(14)	0.58608(19)	0.85423(13)	0.0518(4)
C16	0.43780(15)	0.69134(18)	0.90220(13)	0.0511(4)

DOI 10.1515/ncrs-2016-0072

Received March 11, 2016; accepted June 13, 2016; available online July 6, 2016

Abstract

C₁₈H₁₆F₂N₂O₂, monoclinic, $P2_1/c$ (no. 14), $a = 11.619(3)$ Å, $b = 9.481(2)$ Å, $c = 14.555(3)$ Å, $\beta = 98.799(4)^\circ$, $V = 1584.6(6)$ Å³, $Z = 4$, $R_{gt}(F) = 0.0404$, $wR_{ref}(F^2) = 0.1139$, $T = 293(2)$ K.

CCDC no.: 1453891

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.

*Corresponding author: He Lou, Institute of Chemical and Electrochemical Process Engineering, Clausthal University of Technology, Clausthal-Zellerfeld, Niedersachsen, Germany, e-mail: lou_he666@yeah.net

Table 2 (continued)

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
H16A	0.4777	0.7749	0.9184	0.061*
C17	0.32970(15)	0.66567(18)	0.92461(12)	0.0477(4)
C18	0.26973(13)	0.54230(17)	0.90315(11)	0.0425(4)
H18A	0.1970	0.5287	0.9210	0.051*
N1	0.27219(11)	0.07502(15)	1.04846(9)	0.0466(4)
H1A	0.3396	0.0354	1.0569	0.056*
H1B	0.2257	0.0634	1.0885	0.056*
N2	0.49584(12)	0.06725(17)	0.89667(11)	0.0553(4)
O1	0.12918(8)	0.20508(11)	0.97425(7)	0.0398(3)
O2	0.10285(10)	0.42306(14)	0.68146(8)	0.0528(3)
F1	0.59006(10)	0.60887(14)	0.82958(11)	0.0866(4)
F2	0.27743(11)	0.76884(12)	0.96812(10)	0.0779(4)

Source of material

The title compound was synthesized according to a reported procedure [6]. A mixture of 3,5-cyclohexanedione (10 mmol), 3,5-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 precipitate, it was sequentially washed with ice-cooled water and ethanol and then dried under a vacuum.

Experimental details

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

Discussion

The importance of the pyranil moiety has been well proved as illustrated by a large number of patents, as chemotherapeutic agents [1, 2]. A number of biological activities have been associated with these compounds, such as antihypertensive, antiasthmatic, analgesic, cardiovascular, platelet aggregation

inhibitor and Ca antagonist [3, 4]. With this in mind, some new pyran derivatives have been synthesized in search of better therapeutic agents, by a convenient single pot method [5].

In the crystal structure of the title compound, the 4*H*-pyran ring is nearly planar whereas the directly attached ring containing the keto group is folded. The plane of the 4*H*-pyran ring is almost perpendicular to the plane of the difluorophenyl moiety. All bond lengths and angles are in the expected ranges.

References

- Li, J.; Xue, X. Y.; Li, X.; Hou, Z.; Yang, X. H.; Qu, D.; Zhou, Y.; Zhang, Z. D.; Luo, X. X.; Li, J. T.; Li, M. K.: Synthesis of biscoumarin and dihydropyran derivatives as two novel classes of potential anti-bacterial derivatives. Arch. Pharm. Res. 2015, DOI 10.1007/s12272-015-0614-7.
- Li, J.; Meng, J.; Qu, D.; Zhang, Z.-D.; Li, F.; Yang, X.-H.; Li, J.-T.; Li, M.-K.: New biscoumarin and dihydropyran derivatives as antimicrobials. Res. Chem. Intermed. **41** (2015) 8257–8267.
- Li, J.; Lv, C. W.; Li, X. J.; Qu, D.; Hou, Z.; Jia, M.; Luo, X. X.; Li, X.; Li, M. K.: Synthesis of biscoumarin and dihydropyran derivatives and evaluation of their antibacterial activity. Molecules **20** (2015) 17469–17482.
- Li, J.; Zhou, P.; Liu, Z.; Li, J.: Crystal structure, photoluminescence, and theoretical studies of dihydropyran derivatives. Chinese J. Struct. Chem. **34** (2015) 319–323.
- Li, J.; Xin, J.-J.; Liu, Z.; Li, M.-K.; Hu, X.-B.: Synthesis, crystal structures and anti-cervical cancer activity of two dihydropyran Derivatives. Lat. Am. J. Pharm. **34** (2015) 404–407.
- Li, J.; Sui, Y. P.; Xin, J. J.; Du, X. L.; Li, J. T.; Huo, H. R.; Ma, H.; Wang, W. H.; Zhou, H. Y.; Zhan, H. D.; Wang, Z. J.; Li, C.; Sui, F.; Li, X.: Synthesis of biscoumarin and dihydropyran derivatives with promising antitumor and antibacterial activities. Bioorg. Med. Chem. Lett. **25** (2015) 5520–5523.
- Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112–122.
- Bruker. APEX2, SAINT and SADABS. Bruker AXS Inc., Madison, WI, USA, 2009.