Liyan Chen*

Crystal structure of methyl 4-(3,5-ditrifluoromethylphenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate — water (2/1), C₂₂H₂₁F₆NO₃

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.29 × 0.28 × 0.21 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.13 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	25.0°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	10918, 3918, 0.057
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1993
$N(\text{param})_{\text{refined}}$:	298
Programs:	Bruker [1], SHELX [2], OLEX2 [3]

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.3773(2)	0.3527(3)	0.18479(11)	0.0475(8)
H1	0.430906	0.373790	0.197638	0.057*
O1	0.1673(2)	0.3183(3)	0.04915(10)	0.0751(9)
O2	0.2613(3)	0.4792(4)	0.04910(14)	0.1245(16)
O3	0.11612(19)	0.1155(3)	0.19811(10)	0.0638(8)
O1W	0.500000	0.4992(4)	0.250000	0.0488(10)
C1	0.1263(4)	0.3531(6)	0.00265(18)	0.108(2)
H1A	0.077610	0.295335	−0.007638	0.163*
H1B	0.102963	0.438297	0.003094	0.163*
H1C	0.171099	0.348910	−0.018547	0.163*
C2	0.2353(3)	0.3876(4)	0.06860(16)	0.0616(12)
C3	0.2721(3)	0.3403(4)	0.11552(13)	0.0458(10)
C4	0.3481(3)	0.3921(3)	0.13908(14)	0.0453(9)
C5	0.4068(3)	0.4934(4)	0.12275(16)	0.0696(13)
H5A	0.456176	0.511228	0.147017	0.104*
H5B	0.430430	0.464963	0.094996	0.104*
H5C	0.371522	0.569341	0.115703	0.104*
C6	0.3242(2)	0.2820(3)	0.20992(13)	0.0400(9)
C7	0.2477(2)	0.2277(3)	0.18862(13)	0.0412(9)
C8	0.2219(2)	0.2321(3)	0.13616(12)	0.0438(9)
H8	0.156542	0.248724	0.129482	0.053*
C9	0.1879(3)	0.1625(3)	0.21669(13)	0.0452(9)
C10	0.2140(3)	0.1592(4)	0.26881(13)	0.0566(11)
H10A	0.188562	0.083034	0.281007	0.068*
H10B	0.186960	0.232055	0.282425	0.068*
C11	0.3156(3)	0.1607(4)	0.28486(13)	0.0531(10)
C12	0.3341(3)	0.1761(5)	0.33784(15)	0.0816(15)
H12A	0.398556	0.176922	0.347598	0.122*
H12B	0.308279	0.254832	0.346739	0.122*
H12C	0.307323	0.106405	0.352699	0.122*

<https://doi.org/10.1515/ncrs-2018-0112>

Received May 14, 2018; accepted June 30, 2018; available online July 18, 2018

Abstract

C₂₂H₂₁F₆NO₃, monoclinic, *C*2/*c* (no. 15), *a* = 14.849(4) Å, *b* = 10.497(3) Å, *c* = 28.694(7) Å, β = 97.682(5)°, *V* = 4432(2) Å³, *Z* = 4, *R*_{gt}(*F*) = 0.0736, *wR*_{ref}(*F*²) = 0.2405, *T* = 293(2) K.

CCDC no.: 1852647

The asymmetric unit of the title 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

The title compound was synthesized according to a reported procedure. A mixture of 5,5-dimethyl-cyclohexane-1,3-dione (10 mmol), 3,5-ditrifluoromethylbenzaldehyde (10 mmol), 3-amino-2-butenic acid methyl ester (10 mmol) in ethanol (100 mL) was refluxed for 2–3 h and then cooled to room

*Corresponding author: Liyan Chen, College of Animal Science and Technology, Inner Mongolia University for Nationalities, Tongliao 028042, P.R. China, e-mail: liyan_chen666@yeah.net

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
C13	0.3583(3)	0.0377(4)	0.27014(17)	0.0746(14)
H13A	0.346289	0.028670	0.236589	0.112*
H13B	0.422774	0.040237	0.279674	0.112*
H13C	0.332800	−0.033384	0.284867	0.112*
C14	0.3551(3)	0.2741(3)	0.26127(13)	0.0476(10)
H14A	0.337964	0.351892	0.276097	0.057*
H14B	0.420877	0.268376	0.266350	0.057*
C15	0.2415(3)	0.1050(3)	0.11411(12)	0.0420(9)
C16	0.1739(3)	0.0361(4)	0.08728(13)	0.0497(10)
H16	0.115028	0.068081	0.082212	0.060*
C17	0.1931(3)	−0.0793(4)	0.06801(13)	0.0498(10)
C18	0.1197(3)	−0.1511(5)	0.04078(18)	0.0709(13)
C19	0.2798(3)	−0.1285(4)	0.07502(13)	0.0536(11)
H19	0.292284	−0.206710	0.062084	0.064*
C20	0.3477(3)	−0.0607(4)	0.10138(14)	0.0498(10)
C21	0.3285(3)	0.0555(3)	0.12077(13)	0.0478(10)
H21	0.374828	0.100824	0.138514	0.057*
C22	0.4400(3)	−0.1107(5)	0.1093(2)	0.0799(15)
F1	0.0532(2)	−0.1772(5)	0.06250(14)	0.1550(18)
F2	0.0823(3)	−0.0946(4)	0.00388(15)	0.184(2)
F3	0.1419(3)	−0.2587(4)	0.0254(2)	0.189(2)
F4	0.4563(2)	−0.2125(3)	0.08651(14)	0.1199(13)
F5	0.4647(3)	−0.1440(6)	0.15325(17)	0.182(2)
F6	0.5013(2)	−0.0283(4)	0.1026(2)	0.177(2)
H1W	0.460(3)	0.545(4)	0.2656(15)	0.069(13)*

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 $U_{\text{iso}}(\text{H}) = 1.2$ times $U_{\text{eq}}(\text{C})$ and 1.2 times $U_{\text{eq}}(\text{N})$.

Comment

Microorganisms are critical to nutrient recycling in ecosystems, because they act as decomposers. Because some microorganisms can also take nitrogen out of the air, they are an important part of the nitrogen cycle. Pathogenic, or harmful, microbes can invade other organisms and cause

disease [4]. 4-Arylpolyhydroquinolines possess several types of pharmacological properties such as anticancer, anti-HIV, anticoagulant, spasmolytic, and antibacterial activity. A large number of structurally novel 4-arylpolyhydroquinolines have been reported as substantial antimicrobial drugs. Considering their importance, some research focused on the synthesis of the 4-arylpolyhydroquinolines [5–7].

In the crystal structure of the title compound (*cf.* the figure), the six-membered ring containing nitrogen atom is nearly planar and the adjacent hexenone adopts a flattened chair conformation. Geometric parameters are in agreement with those reported in the literature. A closely related compound is the compound which has a nitro substituent at the phenyl instead of the two CF₃ groups in the title compound [8].

References

1. Bruker. SAINT, APEX2 and SADABS. Bruker AXS Inc., Madison, Wisconsin, USA (2009).
2. Sheldrick, G. M.: Crystal structure refinement with SHELXL. *Acta Crystallogr.* **C71** (2015) 3–8.
3. Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H.: OLEX2: a complete structure solution, refinement and analysis program. *J. Appl. Crystallogr.* **42** (2009) 339–341.
4. 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.
5. Song, Y.; Li, N.; Gu, J.; Fu, S.; Peng, Z.; Zhao, C.; Zhang, Y.; Li, X.; Wang, Z.; Li, X.; Liu, G.: β-Hydroxybutyrate induces bovine hepatocyte apoptosis via an ROS-p38 signaling pathway. *J. Dairy Sci.* **99** (2016) 9184–9198.
6. Sun, X.; Yuan, X.; Chen, L.; Wang, T.; Wang, Z.; Sun, G.; Li, X.; Li, X.; Liu, G.: Histamine induces bovine rumen epithelial cell inflammatory response via NF-κB pathway. *Cell. Physiol. Biochem.* **42** (2017) 1109–1119.
7. Liang, L.; Wang, F. Y.; Shi, J. F.; Hu, Q.; Zhang, L.; Ni, H. B.: One-pot synthesis of 4-arylpolyhydroquinoline derivatives and evaluation of their anti-gastric cancer activity. *Lat. Am. J. Pharm.* **37** (2018) 80–84.
8. Meng, J.; Zhou, H.-Y.; Sui, F.: Crystal structure of methyl 2,7,7-trimethyl-4-(4-nitrophenyl)-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate, C₂₀H₂₂N₂O₅. *Z. Kristallogr. NCS* **232** (2017) 527–528.