Jie Wang, Li-Sha Luo, Yu-Cong Deng, Zhong-Chao Li* and Da-Yong Peng*

Crystal structure of 6-methyl-3-(pyrrolidine-1-carbonyl)-2*H*-chromen-2-one, C₁₅H₁₅N₁O₃

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Abstract

C₁₅H₁₅N₁O₃, triclinic, $P\bar{1}$ (no. 2), $a = 7.1053(11)$ Å, $b = 8.3014(12)$ Å, $c = 11.0297(16)$ Å, $\alpha = 85.810(2)^\circ$, $\beta = 79.896(2)^\circ$, $\gamma = 87.055(2)^\circ$, $V = 638.28(16)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0371$, $wR_{\text{ref}}(F^2) = 0.1062$, $T = 296(2)$ K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

To a stirred solution of 6-methyl-2-oxo-2*H*-chromene-3-carboxylic acid (0.612 g, 3 mmol), *N,N*-Dimethylformamide (DMF) (0.007 g, 0.1 mmol) was added dropwise SOCl₂ (2.975 g, 25 mmol) at 0 °C for 30 min, then enhanced to 95 °C for 4 h. The reaction was monitored by thin-layer chromatography (TLC). Next, unreacted SOCl₂ was removed.

*Corresponding authors: Zhong-Chao Li, Saerfu(Henan)

Agrochemical Co., Ltd, Mengzhou 454750, People's Republic of China, E-mail: 3265909@qq.com; and Da-Yong Peng, Key Laboratory of Chemical Utilization of Plant Resources of Nanchang, College of Chemistry and Materials, Jiangxi Agricultural University, Nanchang 330045, People's Republic of China, E-mail: dayongpeng@163.com. <https://orcid.org/0000-0003-2527-8714>

Jie Wang, Li-Sha Luo and Yu-Cong Deng, College of Chemistry and Materials, Jiangxi Agricultural University, Nanchang 330045, People's Republic of China

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.16 × 0.16 × 0.12 mm
Wavelength:	Mo Kα radiation (0.71073 Å)
μ:	0.09 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ _{max} , completeness:	25.5°, 99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	4943, 2357, 0.019
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2003
$N(\text{param})_{\text{refined}}$:	174
Programs:	Bruker [1], SHELX [2, 3], Diamond [4]

Then 15 mL of anhydrous CH₂Cl₂ was added. Pyrrolidine (0.22 g, 3.1 mmol) and TEA (0.607 g, 6 mmol) were dissolved in 10 mL of anhydrous CH₂Cl₂. This solution was added dropwise to the reaction mixture at room temperature. The reaction was monitored by TLC. After, CH₂Cl₂ was removed. At last, the reaction mixture was allowed to recrystallize from methanol to obtain 6-methyl-3-(pyrrolidine-1-carbonyl)-2*H*-chromen-2-one.

Experimental details

All H atoms were included in calculated positions and refined as riding atoms, with C–H = 0.90–0.97 Å with $U_{\text{iso}}(\text{H}) = 1.5 U_{\text{eq}}(\text{C})$ for methyl H atoms and $1.2 U_{\text{eq}}(\text{C})$ for all other H atoms.

Comment

Coumarin and its derivatives are heterocyclic compounds with a benzopyrone structure [5, 6], which may have excellent antiviral [7], antioxidation [8] antibacterial [9, 10], anticancer [11] and many other physiological, pharmacological activities and optical properties [12]. Among them, coumarin-3-carboxylic acids are vital precursors for the synthesis of active compounds such as dicoumarol, fluorocoumarin carboxylate and coumarin amide [13, 14]. Therefore, it is a research hotspot to explore the efficient synthesis of coumarin-3-carboxylic acids [15–17].

In the molecule of the title compound (fig.), bond lengths and angles are very similar to those given in the

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

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
C1	0.25591 (18)	0.49996 (16)	0.42669 (13)	0.0406 (3)
C2	0.2102 (2)	0.64990 (18)	0.47322 (15)	0.0505 (4)
H2	0.1932	0.7405	0.4209	0.061*
C3	0.1905 (2)	0.66205 (19)	0.59861 (15)	0.0537 (4)
H3	0.1613	0.7628	0.6303	0.064*
C4	0.21277 (19)	0.5279 (2)	0.68005 (14)	0.0496 (4)
C5	0.26202 (19)	0.38007 (19)	0.63054 (13)	0.0451 (3)
H5	0.2803	0.2897	0.6828	0.054*
C6	0.28481 (18)	0.36396 (16)	0.50350 (12)	0.0393 (3)
C7	0.34343 (19)	0.21508 (16)	0.44652 (12)	0.0407 (3)
H7	0.3682	0.1232	0.4956	0.049*
C8	0.36317 (18)	0.20602 (16)	0.32403 (12)	0.0389 (3)
C9	0.32487 (19)	0.34800 (16)	0.24441 (13)	0.0428 (3)
C10	0.44890 (19)	0.05567 (16)	0.26349 (12)	0.0396 (3)
C11	0.1470 (2)	0.0181 (2)	0.18220 (17)	0.0617 (5)
H11A	0.1375	0.1069	0.1211	0.074*
H11B	0.0694	0.0450	0.2601	0.074*
C12	0.0876 (3)	−0.1380 (2)	0.14008 (16)	0.0636 (5)
H12A	0.0443	−0.2129	0.2099	0.076*
H12B	−0.0138	−0.1183	0.0916	0.076*
C13	0.2681 (3)	−0.2022 (2)	0.06258 (15)	0.0593 (4)
H13A	0.2867	−0.1495	−0.0198	0.071*
H13B	0.2642	−0.3179	0.0565	0.071*
C14	0.4249 (2)	−0.16274 (17)	0.13075 (13)	0.0482 (4)
H14A	0.4505	−0.2514	0.1886	0.058*
H14B	0.5420	−0.1393	0.0738	0.058*
C15	0.1845 (3)	0.5447 (3)	0.81726 (16)	0.0681 (5)
H15A	0.0526	0.5299	0.8525	0.102*
H15B	0.2196	0.6504	0.8327	0.102*
H15C	0.2631	0.4644	0.8539	0.102*
N1	0.34679 (16)	−0.01884 (14)	0.19575 (11)	0.0456 (3)
O1	0.27427 (14)	0.49030 (11)	0.30093 (9)	0.0456 (3)
O2	0.33608 (18)	0.35333 (13)	0.13411 (9)	0.0594 (3)
O3	0.60951 (15)	0.00625 (13)	0.27901 (10)	0.0578 (3)

literature for coumarin derivatives [15–18]. In the molecules of the title structure, the coumarin moiety is approximately planar. The dihedral angle between the coumarin moiety and the plane of amides part (C10–N1–O3) is 62.8°. The pyrrolidine part adopts an envelope conformation. The torsion angles of C8–C10–N1–C11, C10–N1–C11–C12, N1–C11–C12–C13, C11–C12–C13–C14 and C12–C13–C14–N1 are −7.0(2)°, −158.5(1)°, −33.7(2)°, 38.6(2)° and −27.7(2)°, respectively.

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