Xiang Yu, Lu Peng, Xin Wang and Ya-Fang Chen*

The crystal structure of (*E*)-4-(7-methoxy-2-oxo-2*H*-chromen-8-yl)-2-methylbut-2-en-1-yl 4-nitrobenzoate, C₂₂H₁₉NO₇

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.30 × 0.15 × 0.15 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.10 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω -scans
$\theta_{\max}$, completeness:	30.5°, >81% (up to 25.2°, >99%)
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	81658, 4882, 0.029
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 4438
$N(\text{param})_{\text{refined}}$:	273
Programs:	Bruker programs [1], OLEX2 [2], SHELX [3, 4]

<https://doi.org/10.1515/ncrs-2019-0393>

Received June 6, 2019; accepted July 12, 2019; available online July 25, 2019

Abstract

C₂₂H₁₉NO₇, monoclinic, $P2_1/n$ (no. 14), $a = 7.5742(4)$ Å, $b = 20.0688(12)$ Å, $c = 13.2932(8)$ Å, $\beta = 102.469(2)^\circ$, $V = 1973.0(2)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0726$, $wR_{\text{ref}}(F^2) = 0.1777$, $T = 198$ K.

CCDC no.: 1940081

The crystal 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

In a representative experiment 7-methoxy-8-(3-methylbut-2-en-1-yl)-2*H*-chromen-2-one (1.0 mmol) reacted with SeO₂ (1.1 mmol) in dioxane (10 mL) at 80 °C, and then reacted with sodium borohydride at 0 °C to give the corresponding alcohol. Finally, a mixture of the aldehyde, 4-nitrobenzoic acid, *N,N'*-dicyclohexylcarbodiimide and 4-dimethylaminopyridine in dry dichloromethane was stirred at room temperature.

*Corresponding author: Ya-Fang Chen, Guizhou University of Traditional Chinese Medicine, Guiyang 550025, P.R. China, e-mail: 985566851@qq.com

Xiang Yu, Lu Peng and Xin Wang: Guizhou University of Traditional Chinese Medicine, Guiyang 550025, P.R. China

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.4098(2)	0.52727(7)	0.14215(12)	0.0408(4)
O2	0.2582(4)	0.61446(10)	0.1701(2)	0.0798(8)
O3	0.7009(3)	0.33155(8)	0.09057(14)	0.0508(5)
O4	0.3060(3)	0.40041(7)	0.39397(12)	0.0456(4)
O5	0.2941(4)	0.36713(9)	0.54588(15)	0.0724(7)
O6	0.1127(4)	0.69025(11)	0.64593(18)	0.0723(7)
O7	0.1919(4)	0.72439(10)	0.51883(19)	0.0852(8)
C1	0.4019(4)	0.59461(12)	0.1605(2)	0.0531(7)
C2	0.5651(5)	0.63380(12)	0.1665(2)	0.0548(7)
H2	0.561481	0.680510	0.177448	0.066*
C3	0.7177(4)	0.60586(11)	0.15708(18)	0.0473(6)
H3	0.822942	0.632428	0.161574	0.057*
C4	0.7261(3)	0.53550(11)	0.14006(15)	0.0372(5)
C5	0.5664(3)	0.49769(10)	0.13170(14)	0.0325(4)
C6	0.5556(3)	0.42938(10)	0.11403(14)	0.0323(4)
C7	0.7157(3)	0.39850(11)	0.10620(16)	0.0387(5)
C8	0.8773(4)	0.43488(13)	0.11383(19)	0.0468(6)
H8	0.984683	0.412436	0.107268	0.056*
C9	0.8810(4)	0.50263(13)	0.13070(18)	0.0454(6)
H9	0.990646	0.526664	0.135871	0.054*
C10	0.3823(3)	0.39017(10)	0.10885(16)	0.0361(5)
H10A	0.278203	0.413712	0.065603	0.043*
H10B	0.391997	0.345370	0.079326	0.043*
N24	0.1610(4)	0.68028(11)	0.57146(18)	0.0544(6)
C11	0.3605(3)	0.38504(10)	0.21286(16)	0.0345(5)
H11	0.339298	0.425397	0.245694	0.041*
C12	0.3667(3)	0.33028(10)	0.26756(17)	0.0372(5)
C13	0.3363(4)	0.33230(10)	0.36947(18)	0.0433(6)
H13A	0.442671	0.314362	0.418741	0.052*

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
H13B	0.229628	0.304863	0.373963	0.052*
C14	0.2878(4)	0.41052(11)	0.48531(17)	0.0420(5)
C15	0.2557(3)	0.48194(10)	0.50344(16)	0.0376(5)
C16	0.2482(4)	0.53175(11)	0.43182(17)	0.0444(6)
H16	0.265159	0.520655	0.365067	0.053*
C17	0.2170(4)	0.59728(12)	0.45350(19)	0.0486(6)
H17	0.213701	0.631362	0.403531	0.058*
C18	0.1919(4)	0.61069(11)	0.54623(18)	0.0427(5)
C19	0.1954(4)	0.56236(12)	0.61747(17)	0.0436(5)
H19	0.173727	0.573471	0.683208	0.052*
C20	0.2301(4)	0.49730(11)	0.59580(16)	0.0413(5)
H20	0.235570	0.463722	0.646759	0.050*
C21	0.8637(5)	0.29654(15)	0.0870(3)	0.0720(9)
H21A	0.838290	0.248706	0.079513	0.108*
H21B	0.953517	0.304555	0.150910	0.108*
H21C	0.910931	0.312353	0.028227	0.108*
C22	0.4016(5)	0.26075(11)	0.2372(2)	0.0561(8)
H22A	0.449909	0.261638	0.174519	0.084*
H22B	0.288367	0.235402	0.224268	0.084*
H22C	0.489530	0.239526	0.292940	0.084*

Suitable colorless block crystals of this compound were obtained after recrystallization from methanol.

Experimental details

All non-hydrogen atoms were assigned anisotropic displacement parameters in the refinement. Hydrogen atoms were treated using a riding model.

Discussion

Coumarins are natural products with diverse bioactivities such as anti-inflammatory [5], anti-tumor [6], and antimicrobial activity [7]. Recently, we prepared a coumarin-based hydrazone derivative by introduction of hydrazone moiety into the coumarin [8]. Here we introduced the 4-nitrobenzoate moiety at the coumarin to find compounds with potential activity.

There is one crystallographically independent molecule in the asymmetric unit, and all bond lengths are in normal

ranges and they are similar with the known compound [9]. The compound contains one methoxy group, one lactone, one methyl-group and one C—C double bond. The length of C—C double bond (atoms C11—C12) was 1.423(3) Å. In addition, the configuration of the C—C double bond is in *E* configuration.

Acknowledgements: The present research was supported by the Science and Technology Fund of Guizhou [No.(2017)5735-22], the Youth Talent Development Project of Guizhou Provincial Department of Education [2017]169, and Undergraduate Training Program for Innovation and Entrepreneurship [2017]158 and [2018]32.

References

1. Bruker. APEX2, SAINT and SADABS. Bruker AXS Inc., Madison, WI, USA (2012).
2. 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. Cryst.* **42** (2009) 339–341.
3. Sheldrick, G. M.: SHELXT C Integrated space-group and crystal-structure determination. *Acta Crystallogr.* **A71** (2015) 3–8.
4. Sheldrick, G. M.: Crystal structure refinement with SHELXL. *Acta Crystallogr.* **C71** (2015) 3–8.
5. Adad, M. J.; Delas, H. B.; Silvan, A. M.: Effects of furocoumarins from *Cachrys trifida* on some macrophage functions. *J. Pharm. Pharmacol.* **8** (2001) 1163–1168.
6. Kim, Y. K.; Kim, Y. S.; Ryu, S. Y.: Antiproliferative effect of furocoumarins from the root of angelica on cultured human tumor cell lines. *Phytother. Res.* **3** (2007) 288–300.
7. Rosselli, S.; Maggio, A.; Bellone, G.: Antibacterial and anti-coagulant activities of coumarins isolated from the flowers of *Magydaris tomentosa*. *Planta Med.* **2** (2007) 116–120.
8. Yu, X.; Qin, Y.; Yan, J.; Chen, Y. F.: Crystal structure of *N'*-(1*E*,2*E*)-4-(7-methoxy-2-oxo-2*H*-chromen-8-yl)-2-methylbut-2-en-1-ylidene)-4-methylbenzenesulfonohydrazide, C₂₂H₂₂O₅N₂S. *Z. Kristallogr. NCS* **234** (2019) 367–368.
9. Yu, X.; Ding, G. D.; Zhi, X. Y.; Xu, H.: Insight into reduction of obacunone, and their ester derivatives as insecticidal agents against *Mythimna separata* Walker. *Bioorg. Med. Chem. Lett.* **25** (2015) 25–29.