Xiang Yu, You-Fang Zhao, Yan Qin, Jing Yan and Ya-Fang Chen*

The crystal structure of *N'*-((1*E*,2*E*)-4-(7-methoxy-2-oxo-2*H*-chromen-8-yl)-2-methylbut-2-en-1-ylidene)-3-methylbenzohydrazide, C₂₃H₂₂N₂O₄

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.29 × 0.28 × 0.27 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.09 mm ⁻¹
Diffractometer, scan mode:	SuperNova, ω
$\theta_{\max}$, completeness:	28.5°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	93062, 9585, 0.055
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 6513
$N(\text{param})_{\text{refined}}$:	529
Programs:	CrysAlis ^{PRO} [1], Olex2 [2], SHELX [3, 4]

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 to afford the aldehyde, and then reacted with 4-methylbenzoyl hydrazine (1.0 mmol), and two drops of HOAc in EtOH (10 mL) at 80 °C. Suitable colorless block crystals of this compound were then obtained after recrystallization from methanol.

Experimental details

The structure was solved with the Olex2 program [2] as an interface together with the SHELXT and SHELXL programs [3, 4]. All non-hydrogen atoms were assigned anisotropic displacement parameters in the refinement. Hydrogen atoms were treated using a riding model.

Comment

Coumarins were natural products with diverse bioactivities such as anti-tumor [5], anti-inflammatory [6] and antimicrobial activity [7]. On the other hand, recent studies show that introduction of hydrazone fragments into natural products could displayed the remarkable biological activity [8, 9]. In order to discover more compounds with potential bioactivities we prepared a coumarin-based hydrazone derivative by introduction of hydrazone moiety into the coumarin.

The asymmetric unit of the title structure consists of two crystallographically independent molecules (*cf.* the figure). Geometric parameters of the title structure are in the usual

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Abstract

C₂₃H₂₂N₂O₄, monoclinic, *P*2₁/*c* (no. 14), *a* = 17.7478(4) Å, *b* = 10.0041(2) Å, *c* = 24.4749(6) Å, β = 111.111(2)°, *V* = 4053.88(17) Å³, *Z* = 8, *R*_{gt}(*F*) = 0.0614, *wR*_{ref}(*F*²) = 0.1749, *T* = 290 K.

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The asymmetric unit containing two crystallographically independent molecules 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.

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

Xiang Yu, You-Fang Zhao, Yan Qin and Jing Yan: 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>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
O1	0.26905(9)	0.96583(15)	0.33980(6)	0.0499(4)
O2	0.24264(14)	1.1723(2)	0.30743(9)	0.0881(6)
O3	0.34233(11)	0.54040(16)	0.42024(8)	0.0645(5)
O4	0.16120(10)	1.24119(13)	0.61106(7)	0.0517(4)
N1	0.20741(10)	1.05625(16)	0.54586(7)	0.0422(4)
N2	0.15460(11)	1.03074(16)	0.57499(8)	0.0448(4)
H2	0.1341	0.9522	0.5731	0.054 [*]
C1	0.23379(15)	1.0559(3)	0.29467(11)	0.0613(6)
C2	0.19009(17)	1.0006(3)	0.23797(12)	0.0736(8)
H2A	0.1672	1.0577	0.2063	0.088 [*]
C3	0.18169(16)	0.8688(3)	0.22975(11)	0.0708(7)
H3	0.1515	0.8360	0.1928	0.085 [*]
C4	0.21820(14)	0.7768(3)	0.27665(10)	0.0534(6)
C5	0.21527(16)	0.6387(3)	0.27125(12)	0.0653(7)
H5	0.1853	0.6008	0.2353	0.078 [*]
C6	0.25518(16)	0.5564(3)	0.31733(12)	0.0626(7)
H6	0.2522	0.4641	0.3127	0.075 [*]
C7	0.30073(14)	0.6131(2)	0.37172(10)	0.0499(5)
C8	0.30533(12)	0.7516(2)	0.37966(9)	0.0407(5)
C9	0.26371(12)	0.8292(2)	0.33160(9)	0.0429(5)
C10	0.3495(2)	0.3997(3)	0.41407(17)	0.0938(10)
H10A	0.3751	0.3827	0.3862	0.141 [*]
H10B	0.2968	0.3598	0.4006	0.141 [*]
H10C	0.3815	0.3619	0.4513	0.141 [*]
C11	0.35239(12)	0.8148(2)	0.43816(9)	0.0415(4)
H11A	0.3963	0.7561	0.4602	0.050 [*]
H11B	0.3755	0.8986	0.4319	0.050 [*]
C12	0.29926(11)	0.84007(19)	0.47269(8)	0.0379(4)
H12	0.2756	0.7650	0.4822	0.045 [*]
C13	0.28159(11)	0.95671(19)	0.49148(8)	0.0380(4)
C14	0.22740(12)	0.9522(2)	0.52403(9)	0.0402(4)
H14	0.2064	0.8700	0.5292	0.048 [*]
C15	0.31369(16)	1.0894(2)	0.48191(12)	0.0609(6)
H15A	0.3712	1.0912	0.5018	0.091 [*]
H15B	0.2895	1.1589	0.4971	0.091 [*]
H15C	0.3010	1.1031	0.4407	0.091 [*]
C16	0.13491(12)	1.12703(19)	0.60623(9)	0.0398(4)
C17	0.07825(13)	1.08329(19)	0.63543(9)	0.0439(5)
C18	0.09078(14)	1.1311(2)	0.69118(9)	0.0458(5)
H18	0.1341	1.1879	0.7094	0.055 [*]
C19	0.03929(16)	1.0952(2)	0.72021(11)	0.0569(6)
C20	−0.0245(2)	1.0119(3)	0.69171(15)	0.0821(10)
H20	−0.0594	0.9869	0.7106	0.099 [*]
C21	−0.0380(2)	0.9649(3)	0.63670(17)	0.0982(12)
H21	−0.0819	0.9095	0.6184	0.118 [*]
C22	0.01351(18)	0.9998(3)	0.60823(14)	0.0771(9)
H22	0.0047	0.9671	0.5709	0.093 [*]
C23	0.0528(2)	1.1483(4)	0.78048(13)	0.0904(10)
H23A	0.1056	1.1874	0.7966	0.136 [*]
H23B	0.0486	1.0764	0.8052	0.136 [*]
H23C	0.0128	1.2149	0.7781	0.136 [*]
O5	0.56386(10)	0.46774(18)	0.70392(8)	0.0614(4)
O6	0.59196(16)	0.6733(3)	0.73497(13)	0.1088(8)
O7	0.50174(11)	0.04222(18)	0.62100(9)	0.0744(5)
O8	0.11940(10)	0.74961(14)	0.56374(9)	0.0658(5)
N3	0.23454(9)	0.55897(16)	0.58128(7)	0.0373(4)

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
N4	0.15676(9)	0.53398(15)	0.57946(7)	0.0366(4)
H4	0.1431	0.4539	0.5847	0.044 [*]
C24	0.60162(18)	0.5575(4)	0.74803(15)	0.0775(8)
C25	0.6489(2)	0.5020(4)	0.80416(15)	0.0917(10)
H25	0.6753	0.5593	0.8351	0.110 [*]
C26	0.65588(19)	0.3688(4)	0.81295(13)	0.0879(10)
H26	0.6870	0.3360	0.8497	0.106 [*]
C27	0.61669(14)	0.2782(3)	0.76716(11)	0.0656(7)
C28	0.62150(16)	0.1388(3)	0.77156(13)	0.0756(9)
H28	0.6511	0.1004	0.8075	0.091 [*]
C29	0.58408(16)	0.0571(3)	0.72479(14)	0.0710(8)
H29	0.5879	−0.0353	0.7291	0.085 [*]
C30	0.54028(13)	0.1141(3)	0.67072(12)	0.0570(6)
C31	0.53350(12)	0.2530(2)	0.66384(10)	0.0477(5)
C32	0.57150(13)	0.3314(3)	0.71235(11)	0.0531(6)
C33	0.5124(2)	−0.0993(3)	0.62216(19)	0.1038(12)
H33A	0.4833	−0.1354	0.5840	0.156 [*]
H33B	0.4923	−0.1379	0.6501	0.156 [*]
H33C	0.5688	−0.1196	0.6331	0.156 [*]
C34	0.48551(12)	0.3149(2)	0.60558(10)	0.0490(5)
H34A	0.5104	0.3986	0.6013	0.059 [*]
H34B	0.4860	0.2556	0.5743	0.059 [*]
C35	0.40024(11)	0.3401(2)	0.60045(9)	0.0419(5)
H35	0.3710	0.2659	0.6044	0.050 [*]
C36	0.36113(11)	0.45670(19)	0.59091(8)	0.0370(4)
C37	0.27756(11)	0.45395(19)	0.58787(8)	0.0370(4)
H37	0.2546	0.3720	0.5909	0.044 [*]
C38	0.39748(13)	0.5875(2)	0.58287(11)	0.0551(6)
H38A	0.4054	0.5870	0.5461	0.083 [*]
H38B	0.4485	0.5998	0.6141	0.083 [*]
H38C	0.3617	0.6592	0.5832	0.083 [*]
C39	0.10250(12)	0.63346(19)	0.56957(9)	0.0388(4)
C40	0.01956(11)	0.59498(19)	0.56642(8)	0.0365(4)
C41	0.00208(11)	0.4783(2)	0.58977(9)	0.0390(4)
H41	0.0433	0.4174	0.6074	0.047 [*]
C42	−0.07599(12)	0.4500(2)	0.58743(9)	0.0451(5)
C43	−0.13603(12)	0.5427(2)	0.56054(10)	0.0497(5)
H43	−0.1883	0.5264	0.5591	0.060 [*]
C44	−0.12031(13)	0.6574(2)	0.53608(10)	0.0515(6)
H44	−0.1618	0.7173	0.5178	0.062 [*]
C45	−0.04303(12)	0.6843(2)	0.53851(9)	0.0456(5)
H45	−0.0325	0.7619	0.5216	0.055 [*]
C46	−0.09364(15)	0.3244(3)	0.61374(14)	0.0766(8)
H46A	−0.0903	0.2492	0.5903	0.115 [*]
H46B	−0.1470	0.3292	0.6150	0.115 [*]
H46C	−0.0550	0.3140	0.6528	0.115 [*]

ranges [10]. The compound contains one methoxy group, one lactone and two methyl-groups. The length of C—C double bond are, longer than the length of C—N double bond. Both of the configuration of the C—C double bond and the configuration C—N double bond are in type E, as well as the similar structure that reported [11].

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References

1. Oxford Diffraction. CrysAlis^{Pro} 1.171.39.46, SCALE3 ABSPACK, Oxford Diffraction Ltd, Yarnton, England (2018).
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. Crystallogr.* **42** (2009) 339–341.
3. Sheldrick, G. M.: SHELXT – 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. 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.
6. 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.
7. Rosselli, S.; Maggio, A.; Bellone, G.: Antibacterial and anticoagulant activities of coumarins isolated from the flowers of *Magydaris tomentosa*. *Planta Med.* **2** (2007) 116–120.
8. Qu, H.; Lv, M.; Yu, X.; Lian, X. H.; Xu, H.: Discovery of some piperine-based phenylsulfonylhydrazone derivatives as potent botanically narcotic agents. *Sci. Rep.* **5** (2015) 13077.
9. Yu, X.; Feng, G.; Huang, J. L.; Xu, H.: Evaluation of some quinoline-based hydrazone derivatives as insecticidal agents. *RSC Adv.* **6** (2016) 30405–30411.
10. Yu, X.; Qin, Y.; Yan, J.; Chen, Y.-F.: Crystal structure of N'-((1*E*, 2*E*)-4-(7-methoxy-2-oxo-2H-chromen-8-yl)-2-methylbut-2-en-1-ylidene)-4-methylbenzenesulfonohydrazide, $C_{22}H_{22}O_5N_2S$. *Z. Kristallogr. NCS* **234** (2019) 367–368.
11. Qu, H.; Yu, X.; Zhi, X. Y.; Lv, M.; Xu, H.: Natural-product-based insecticidal agents 14. semisynthesis and insecticidal activity of new piperine-based hydrazone derivatives against *Mythimna separata* Walker *in vivo*. *Bioorg. Med. Chem. Lett.* **20** (2013) 5552–5557.