Ting-Guang Zou, Si-Yu Guo, Jing-Lin Zhu, Sheng Wang, Ling-Yin Lin and Yi-Wen Tao*

The crystal structure of 5-hydroxy-6,8-dimethoxy-2-methyl-4*H*-benzo[*g*]chromen-4-one–rubrofusarin B, C₁₆H₁₄O₅

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.13 × 0.12 × 0.10 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.11 mm ⁻¹
Diffractometer, scan mode:	SuperNova, ω
$\theta_{\max}$, completeness:	25.0°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	5600, 2266, 0.022
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1809
$N(\text{param})_{\text{refined}}$:	194
Programs:	CrysAlis ^{PRO} [1], SHELX [2], Olex2 [3]

<https://doi.org/10.1515/ncrs-2020-0645>

Received December 28, 2020; accepted January 14, 2021;

published online February 15, 2021

Abstract

C₁₆H₁₄O₅, monoclinic, $P2_1/c$ (no. 14), $a = 9.0005(11)$ Å, $b = 16.0386(15)$ Å, $c = 9.8434(11)$ Å, $\beta = 114.861(14)^\circ$, $V = 1289.3(3)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0458$, $wR_{\text{ref}}(F^2) = 0.1245$, $T = 150(1)$ K.

CCDC no.: 2052053

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.

***Corresponding author: Yi-Wen Tao**, Key Laboratory of Molecular Target & Clinical Pharmacology and the State Key Laboratory of Respiratory Disease, School of Pharmaceutical Sciences & The Fifth Affiliated Hospital, Guangzhou Medical University, Guangzhou, Guangdong 511436, P. R. China, E-mail: yywentao@aliyun.com
Ting-Guang Zou, Si-Yu Guo, Jing-Lin Zhu, Sheng Wang and Ling-Yin Lin, Key Laboratory of Molecular Target & Clinical Pharmacology and the State Key Laboratory of Respiratory Disease, School of Pharmaceutical Sciences & The Fifth Affiliated Hospital, Guangzhou Medical University, Guangzhou, Guangdong 511436, P. R. China, E-mail: 158562347@qq.com (S. Wang)

Source of material

All solvents of analytical grade were used. The title compound was isolated and purified from the metabolites of *Aspergillus fumigatus* (for details see the Comment section).

Experimental details

All hydrogen atoms were placed in calculated positions and refined using a riding model with the relative isotropic parameters. U_{iso} values of hydrogen atoms were set to 1.2 U_{eq} of the parent atoms.

Comment

The endophytic fungus *A. fumigatus*. was isolated from mangrove tree *Sonneratia apetala* in Dongzhai harbor, Hainan Province, and identified by 18S DNA gene sequence [4]. The specimen was stored at Guangzhou Medical University, Guangzhou, P. R. China. This fungus strain was fermented statically by using rice medium (85 g rice, 160 mL coarse sea salt solution with a mass concentration of 2 g/L) in a 1 L Erlenmeyer flask at 28 °C for forty days. The metabolites of *A. fumigatus* were extracted with methanol, and then dried with rotary evaporator. The extract was further purified from the culture by various chromatographic purification techniques.

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}
O1	0.78908 (15)	0.84499 (8)	0.50175 (13)	0.0257 (3)
O2	0.58623 (15)	0.59409 (7)	0.60455 (13)	0.0239 (3)
O3	0.36755 (15)	0.59571 (8)	0.70845 (13)	0.0236 (3)
H3	0.299267	0.581240	0.738547	0.035*
O4	0.14519 (15)	0.60616 (8)	0.80276 (13)	0.0263 (3)
O5	0.17684 (14)	0.86012 (7)	0.79365 (12)	0.0217 (3)
C1	0.4779 (2)	0.72043 (11)	0.65159 (17)	0.0171 (4)
C2	0.5882 (2)	0.67855 (11)	0.60250 (17)	0.0185 (4)
C3	0.6868 (2)	0.72326 (11)	0.55541 (18)	0.0213 (4)
H3A	0.758256	0.695236	0.525219	0.026*
C4	0.6824 (2)	0.81084 (11)	0.55165 (17)	0.0199 (4)
C5	0.5790 (2)	0.85395 (11)	0.59614 (18)	0.0197 (4)
H5	0.576086	0.911896	0.592327	0.024*
C6	0.47671 (19)	0.80970 (11)	0.64799 (16)	0.0172 (4)
C7	0.3727 (2)	0.85469 (11)	0.69630 (17)	0.0193 (4)
H7	0.371480	0.912660	0.693797	0.023*
C8	0.2743 (2)	0.81254 (11)	0.74654 (17)	0.0177 (4)
C9	0.2690 (2)	0.72499 (11)	0.75075 (17)	0.0182 (4)
C10	0.3719 (2)	0.67927 (11)	0.70354 (16)	0.0177 (4)
C11	0.1581 (2)	0.68417 (12)	0.80228 (17)	0.0203 (4)
C12	0.0637 (2)	0.73889 (12)	0.85092 (18)	0.0238 (4)
H12	−0.007608	0.716137	0.887134	0.029*
C13	0.0761 (2)	0.82210 (12)	0.84535 (17)	0.0213 (4)
C14	−0.0131 (2)	0.88461 (13)	0.8942 (2)	0.0285 (5)
H14A	0.058527	0.906097	0.990683	0.043*
H14B	−0.106182	0.858697	0.900409	0.043*
H14C	−0.049251	0.929444	0.823070	0.043*
C15	0.6952 (2)	0.55137 (12)	0.5566 (2)	0.0279 (5)
H15A	0.671707	0.567097	0.455485	0.042*
H15B	0.681011	0.492279	0.561256	0.042*
H15C	0.806206	0.566043	0.620811	0.042*
C16	0.7795 (2)	0.93298 (12)	0.4746 (2)	0.0293 (5)
H16A	0.670468	0.947325	0.404625	0.044*
H16B	0.855678	0.948363	0.433978	0.044*
H16C	0.805830	0.962237	0.566944	0.044*

The title compound was isolated and purified from 20% petroleum 80% ethyl acetate fraction through silica gel column chromatography. Its structure was identified by MS and NMR spectroscopic data, and confirmed by X-ray crystallography. Crystallization of the title compound in CHCl₃–N-hexane (6:1, v/v) gave yellow crystals at room temperature. It has been reported that this compound displayed antibacterial activity against *C. Albi-cans* and *Escherichia coli*, and shown to be cytotoxic to the colon cancer cell line SW1116 [5–9]. What's more, the X-ray crystallography data of the title compound are reported here for the first time. The geometric parameters are similar to those reported for the parent compound rubrofusarin [10].

Author contributions: Ting-Guang Zou, Si-Yu Guo, Jing-Lin Zhu and Sheng Wang contributed equally to this work and all the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Research funding: Guangdong Natural Science Foundation of China (2018A0303130201), Guangzhou Education Bureau Yangcheng Scholars Project (202032774), National Student Training Programs for innovation and Entrepreneurship (202010570028), Special Funds for Undergraduates Scientific and Technological Innovation Training Programs in Guangdong (pdjh2020b0487, S201910570075, S201910570069), and Undergraduate Training Programs for Innovation and Entrepreneurship in GZHMU (2019A076, 2018A107, 2018A103).

Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

References

- Agilent Technologies. CrysAlis^{PRO} Software System, Version 1.171.37.35; Agilent Technologies UK Ltd: Oxford, UK, 2011.
- Sheldrick G. M. A short history of SHELX. *Acta Crystallogr.* 2008, *A64*, 112–122.
- 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.* 2009, *42*, 339–341.
- Siriwardane A. M. D. A., Savitri Kumar N., Jayasinghe L., Fujimoto Y. Chemical investigation of metabolites produced by an endophytic *Aspergillus* sp. isolated from *Limonia acidissima*. *Nat. Prod. Res.* 2015, *29*, 1384–1387.
- Hua Y., Pan R., Bai X., Wei B., Chen J., Wang H., Zhang H. Aromatic polyketides from a symbiotic strain *Aspergillus fumigatus* D and characterization of their biosynthetic gene D8.t287. *Mar. Drugs* 2020, *18*, 324.
- Ma Y.-M., Li T., Ma C.-C. A new pyrone derivative from an endophytic *Aspergillus tubingensis* of *Lycium ruthenicum*. *Nat. Prod. Res.* 2016, *30*, 1499–1503.
- Song Y. C., Li H., Ye Y. H., Shan C. Y., Yang Y. M., Tan R. X. Endophytic naphthopyrone metabolites are co-inhibitors of xanthine oxidase, SW1116 cell and some microbial growths. *FEMS Microbiol. Lett.* 2006, *241*, 67–72.
- Huang H.-B., Feng X.-J., Liu L., Chen B., Lu Y.-J., Ma L., She Z.-G., Lin Y.-C. Three dimeric naphtho-γ-pyrones from the mangrove Endophytic Fungus *Aspergillus tubingensis* isolated from *Pongamia pinnata*. *Planta Med.* 2010, *76*, 1888–1891.
- Shaaban M., Shaaban K. A., Abdel-Aziz M. S. Seven naphtho-γ-pyrones from the marine-derived fungus *Alternaria alternata*: structure elucidation and biological properties. *Org. Med. Chem. Lett.* 2012, *2*, 8.
- Graham J. G., Zhang H., Pendland S. L., Santarsiero B. D., Mesecar A. D., Cabieses F., Farnsworth N. R. Antimycobacterial naphthopyrones from *Senna obliqua*. *J. Nat. Prod.* 2004, *67*, 225–227.