Bao-Long Lai, Hai-Ming Zhou, Zan-Hong Chen, Hui-Ying Chen, Qin-Wen Zhang and Yi-Wen Tao*

The crystal structure of $C_{19}H_{20}O_8$

Table 1: Data collection and handling.

Crystal:	Block, colourless
Size:	0.14 × 0.12 × 0.1 mm
Wavelength:	Cu K α radiation (1.54184 Å)
μ :	0.94 mm ⁻¹
Diffractometer, scan mode:	SuperNova, φ and ω -scans
$\theta_{\max}$, completeness:	74°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	5703, 3448, 0.01
R_{int} :	
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3217
$N(\text{param})_{\text{refined}}$:	252
Programs:	CrysAlis ^{PRO} [1], SHELX [2, 3], OLEX2 [4]

<https://doi.org/10.1515/ncrs-2021-0405>

Received October 19, 2021; accepted December 2, 2021;

published online January 4, 2022

Abstract

$C_{19}H_{20}O_8$, triclinic, $P\bar{1}$ (no. 2), $a = 7.0708(4)$ Å, $b = 8.1261(4)$ Å, $c = 16.1067(6)$ Å, $\alpha = 81.252(4)^\circ$, $\beta = 81.500(4)^\circ$, $\gamma = 77.890(4)^\circ$, $V = 887.88(8)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0418$, $wR_{\text{ref}}(F^2) = 0.1158$, $T = 150$ K.

CCDC no.: 2125914

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.

Hai-Ming Zhou: Co-first author.

*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: ywentaotao@aliyun.com. <https://orcid.org/0000-0002-2863-3913>

Bao-Long Lai, Department of Pharmacy of Respiratory Disease, The 7th Affiliated Hospital of Sun Yat-Sen University, Shenzhen, Guangdong 518107, P. R. China

Hai-Ming Zhou, Zan-Hong Chen, Hui-Ying Chen and Qin-Wen Zhang, 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

Source of materials

Light yellow crystals of the title compound were obtained by slow evaporation in the methanol and dichloromethane solution in a ratio of 1:2 at 4 °C.

Experimental details

All hydrogen atoms were placed in calculated positions and refined using a riding model with the relative isotropic parameters.

Comment

Strain *Aspergillus fumigatus* was collected from Dongzhai Harbor, Hainan province, China, and identified by 18S RNA gene sequence. The specimen was stored at Guangzhou Medical University, Guangzhou, P. R. China. The stored strain was cultured and fermented on rice medium (sea salt 2 g/L, rice 350 g/L) at 28 °C for 30 days [5]. The fermentation was soaked in an equal volume of methanol. After being concentrated with a rotary evaporator, it was extracted with ethyl acetate. The ethyl acetate extract was repeatedly washed with methanol after drying. Methanol insoluble and methanol soluble portions were obtained afterwards. The methanol-insoluble substance was then identified as trypacidin. Trypacidin was added with 2 mL sodium hydroxide methanol solution (1 mol/L), stirred and reacted for 12 h at room temperature. The reaction was ended by adding

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

Atom	x	y	z	U _{iso} */U _{eq}
O1	0.28517 (18)	0.09974 (14)	0.49229 (7)	0.0306 (3)
H1	0.318511	0.036168	0.455598	0.046*
O2	0.38647 (16)	0.51210 (13)	0.25448 (6)	0.0236 (3)
O3	0.36190 (17)	0.02077 (13)	0.34200 (7)	0.0290 (3)
O4	0.02217 (15)	0.37782 (14)	0.25178 (6)	0.0245 (3)
O5	0.72670 (15)	0.10598 (13)	0.24897 (7)	0.0261 (3)
O6	0.74456 (17)	−0.09407 (14)	0.16472 (7)	0.0306 (3)
O7	0.61026 (16)	0.11270 (14)	0.01399 (7)	0.0269 (3)
O8	0.25843 (18)	0.29744 (15)	−0.03545 (7)	0.0312 (3)
H8	0.314942	0.215291	−0.059214	0.047*
C1	0.3272 (2)	0.30330 (18)	0.36853 (9)	0.0200 (3)
C2	0.2894 (2)	0.26020 (19)	0.45730 (10)	0.0227 (3)
C3	0.2498 (2)	0.3838 (2)	0.51176 (10)	0.0256 (3)
H3	0.223409	0.353111	0.569684	0.031*
C4	0.2494 (2)	0.5520 (2)	0.48048 (10)	0.0246 (3)
C5	0.2940 (2)	0.59814 (19)	0.39370 (10)	0.0232 (3)
H5	0.296741	0.710901	0.372822	0.028*
C6	0.3340 (2)	0.47659 (18)	0.33881 (9)	0.0206 (3)
C7	0.3449 (2)	0.17365 (18)	0.31313 (9)	0.0202 (3)
C8	0.3325 (2)	0.21632 (17)	0.21981 (9)	0.0199 (3)
C9	0.4823 (2)	0.14568 (17)	0.16078 (9)	0.0206 (3)
C10	0.4630 (2)	0.17475 (18)	0.07444 (9)	0.0220 (3)
C11	0.2890 (2)	0.26826 (19)	0.04760 (9)	0.0231 (3)
C12	0.1398 (2)	0.34005 (18)	0.10547 (10)	0.0224 (3)
H12	0.025385	0.404527	0.086877	0.027*
C13	0.1620 (2)	0.31536 (18)	0.19082 (9)	0.0204 (3)
C14	0.6641 (2)	0.03803 (18)	0.19039 (9)	0.0216 (3)
C15	0.8868 (3)	0.0020 (2)	0.29061 (12)	0.0342 (4)
H15A	1.004143	−0.006154	0.251686	0.051*
H15B	0.859065	−0.109317	0.309234	0.051*
H15C	0.903369	0.052617	0.338518	0.051*
C16	0.2051 (2)	0.6858 (2)	0.53927 (11)	0.0307 (4)
H16A	0.324327	0.701926	0.555742	0.046*
H16B	0.122181	0.650514	0.588638	0.046*
H16C	0.140345	0.790551	0.510958	0.046*
C17	−0.1453 (2)	0.4931 (2)	0.22468 (11)	0.0306 (4)
H17A	−0.105157	0.586860	0.187517	0.046*
H17B	−0.227882	0.534055	0.273037	0.046*
H17C	−0.215548	0.435839	0.195236	0.046*
C18	0.3681 (3)	0.6854 (2)	0.21888 (11)	0.0337 (4)
H18A	0.414300	0.691825	0.159579	0.051*
H18B	0.443894	0.740677	0.246618	0.051*
H18C	0.233743	0.740409	0.226462	0.051*
C19	0.7703 (3)	0.2003 (2)	−0.00111 (11)	0.0356 (4)
H19A	0.861445	0.157026	−0.046706	0.053*
H19B	0.833769	0.183181	0.049097	0.053*
H19C	0.723024	0.319289	−0.015850	0.053*

formic acid to adjust pH value to neutrality, and then the products were further isolated and purified by silica gel chromatography using a solvent system of 2:1 CH₂Cl₂/MeOH to afford the title compound. Its crystals were obtained by

being dissolved in mixed solvent of CH₂Cl₂/MeOH(2/1, v/v) and slowly evaporated at 4 °C.

The structure was clarified by comprehensive analysis of spectral data and confirmed by X-ray crystallography. Geometric parameters are all in the expected ranges. The title compound was named as asperfumin. Liu *et al.* [6] first purified, identified, and enumerated the ¹H NMR and ¹³C NMR data of asperfumin with a benzophenone structure. Asperfumin is NRPS gene clusters dependent [7] and could inhibit the secretion of IL-6 and decrease collagen IV and fibronectin production at 10 μM in mesangial cells treated with high glucose. Moreover, it didn't exhibit cytotoxicity against mesangial cells at 10 μM using 3-(4,5-dimethylthiazol-2-yl)-2,5-diphenyl tetrazoliumbromide (MTT) assay [8].

Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Research funding: This work was supported by Guangzhou Education Bureau Yangcheng Scholars Project (202032774), Scientific Research Foundation of Traditional Chinese Medicine of Health and Family Planning Bureau from Guangming District (GM2019020026), National Students Training Programs for Innovation and Entrepreneurship (202010570028), Special Funds for Undergraduates' Scientific and Technological Innovation Training Programs in Guangdong (pdjh2021b0417, pdjh2020b0487, S201910570075, S201910570069), Undergraduate Training Programs for Innovation and Entrepreneurship in GZHMU (2020A068, 2019A076, 2018A107, 2018A103), and High-level University Construction Fund of Guangdong Province (06-410-2107242, 06-410-2107249, 06-410-2107273).

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

References

1. Oxford Diffraction Ltd. CrysAlis^{PRO}: Abingdon, Oxfordshire, England, 2006.
2. Sheldrick G. M. SHELXT - integrated space-group and crystal-structure determination. *Acta Crystallogr.* 2015, *A71*, 3–8.
3. Sheldrick G. M. Crystal structure refinement with SHELXL. *Acta Crystallogr.* 2015, *C71*, 3–8.
4. 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.
5. Zhang J. Y., Lai Z. Z., Huang W. J., Ling H. P., Lin M. T., Tang S. L., Liu Y., Tao Y. W. Apicidin inhibited proliferation and invasion and induced apoptosis via mitochondrial pathway in non-small cell lung cancer GLC-82 cells. *Anti Cancer Agents Med. Chem.* 2017, *17*, 1374–1382.
6. Liu J. Y., Song Y. C., Zhang Z., Wang L., Guo Z. J., Zou W. X., Tan R. X. *Aspergillus fumigatus* CY018, an endophytic fungus in Cynodon

- dactylon as a versatile producer of new and bioactive metabolites. *J. Biotechnol.* 2004, 114, 279–287.
7. Frisvad J. C., Rank C., Nielsen K. F., Larsen T. O. Metabolomics of *Aspergillus fumigatus*. *Med. Mycol. J.* 2009, 47, S53–S71.
8. Yang Y., Yan Y. M., Wei W., Luo J., Zhang L. S., Zhou X. J., Wang P. C., Yang Y. X., Cheng Y. X. Anthraquinone derivatives from Rumex plants and endophytic *Aspergillus fumigatus* and their effects on diabetic nephropathy. *Bioorg. Med. Chem. Lett.* 2013, 23, 3905–3909.