Juan Zou, Chen-Liang Zhao, Jiang-Hai Ye, Kang He, Ren-Tao Tang and Lu-Tai Pan*

Crystal structure of 7 β ,9 β -dihydroxy-15-oxo-*ent*-kauran-16-en-19,6 β -olide, C₂₀H₂₆O₅

Table 1: Data collection and handling.

Crystal:	Prism, colorless
Size:	0.23 × 0.22 × 0.21 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:	25°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	11424, 2953, 0.122
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2025
$N(\text{param})_{\text{refined}}$:	226
Programs:	Bruker programs [1], SHELX [2, 3], OLEX2 [4]

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

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.2622(7)	0.6275(5)	0.6753(2)	0.0549(15)
O2	0.2016(6)	0.5187(4)	0.5873(2)	0.0394(12)
O3	0.7033(5)	0.3773(3)	0.45170(18)	0.0241(10)
H3	0.8118	0.3914	0.4629	0.036*
O4	0.3849(7)	0.2327(3)	0.4272(2)	0.0321(11)
O5	0.0927(6)	0.3874(4)	0.4547(2)	0.0381(12)
H5A	0.1081	0.3633	0.4162	0.057*
C1	0.7833(8)	0.5666(5)	0.5463(3)	0.0241(13)
H1A	0.8482	0.4902	0.5512	0.029*
H1B	0.8559	0.6175	0.5166	0.029*
C2	0.7645(9)	0.6262(5)	0.6131(3)	0.0307(15)
H2A	0.8880	0.6427	0.6309	0.037*
H2B	0.7004	0.7028	0.6076	0.037*
C3	0.6598(10)	0.5506(6)	0.6611(3)	0.0362(17)
H3A	0.6190	0.6017	0.6977	0.043*
H3B	0.7451	0.4912	0.6795	0.043*
C4	0.4920(9)	0.4854(5)	0.6339(3)	0.0307(15)
C5	0.4989(9)	0.4536(5)	0.5608(3)	0.0229(13)
H5	0.5644	0.3765	0.5558	0.028*
C6	0.2977(8)	0.4351(5)	0.5442(3)	0.0277(14)
H6	0.2614	0.3525	0.5559	0.033*
C7	0.2447(8)	0.4598(5)	0.4736(3)	0.0257(14)
H7	0.2014	0.5437	0.4720	0.031*
C8	0.4038(8)	0.4490(5)	0.4233(3)	0.0225(13)
C9	0.6017(8)	0.4875(4)	0.4477(3)	0.0213(12)
C10	0.5911(8)	0.5452(4)	0.5167(3)	0.0201(13)
C11	0.6959(9)	0.5652(5)	0.3964(3)	0.0266(14)
H11A	0.6365	0.6439	0.3959	0.032*
H11B	0.8257	0.5765	0.4089	0.032*
C12	0.6878(10)	0.5125(6)	0.3278(3)	0.0362(16)
H12A	0.7806	0.4490	0.3241	0.043*
H12B	0.7186	0.5745	0.2954	0.043*

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Abstract

C₂₀H₂₆O₅, orthorhombic, $P2_12_12_1$ (no. 19), $a = 7.302(4)$ Å, $b = 11.307(5)$ Å, $c = 20.414(10)$ Å, $V = 1685.5(14)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0670$, $wR_{\text{ref}}(F^2) = 0.1748$, $T = 296(2)$ K.

CCDC no.: 1818254

The crystal structure of the title compound (systematic name: (3aR,3a1S,6aS,9R,11aR,11bR)-6,11a-dihydroxy-3a,8,11b-trimethyldodecahydro-1H-6a,9-methanocyclohepta[3,4]naphtho[1,8-bc]furan-4,7-dione is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

The aerial part of *Pteris semipinnata* were collected and identified by our research in the October of 2016. After crushing the material into powder (14 kg), it was extracted with 90% ethyl acetate and filtered. The solvent was then distilled off

*Corresponding author: Lu-Tai Pan, Guiyang College of Traditional Chinese Medicine, Guiyang 550002, P.R. China, e-mail: ltpan@sina.cn

Juan Zou, Chen-Liang Zhao, Jiang-Hai Ye, Kang He and Ren-Tao Tang: Guiyang College of Traditional Chinese Medicine, Guiyang 550002, P.R. China

Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
C13	0.4979(10)	0.4613(5)	0.3117(3)	0.0339(16)
H13	0.4637	0.4764	0.2650	0.041*
C14	0.3557(9)	0.5131(5)	0.3589(3)	0.0304(15)
H14A	0.2293	0.4947	0.3446	0.036*
H14B	0.3696	0.5999	0.3633	0.036*
C15	0.4236(8)	0.3220(5)	0.3979(3)	0.0222(13)
C16	0.4844(9)	0.3309(5)	0.3285(3)	0.0277(14)
C17	0.5124(11)	0.2366(6)	0.2907(3)	0.0411(18)
H17A	0.4935	0.1595	0.3078	0.049*
H17B	0.5513	0.2465	0.2466	0.049*
C18	0.4576(10)	0.3736(6)	0.6761(3)	0.0453(19)
H18A	0.5683	0.3245	0.6765	0.068*
H18B	0.4271	0.3972	0.7210	0.068*
H18C	0.3558	0.3282	0.6574	0.068*
C19	0.3130(10)	0.5534(6)	0.6370(3)	0.0382(17)
C20	0.4963(10)	0.6654(5)	0.5110(3)	0.0320(15)
H20A	0.5670	0.7166	0.4817	0.048*
H20B	0.3728	0.6545	0.4932	0.048*
H20C	0.4882	0.7020	0.5544	0.048*

in rotary evaporator and the residue (286 g) was subjected to MCI in order to remove the chlorophyll. The residue was subsequently separated by silica gel column, Sephadex LH-20 and semi-preparative HPLC (Agilent 1100) to afford the crude product (25 mg), after which it was crystallized from methanol to yield the title compound as colorless block crystals (15 mg).

Experimental details

The hydrogen atoms were assigned with common isotropic displacement factors $U_{iso}(H) = 1.2U_{eq}(C)$ and $1.5U_{eq}(O)$, respectively, and included in the final refinement with $d(C-H) = 0.93-0.98$ Å.

Discussion

Species of *Pteris* are widely distributed in China, and many of them are used in folk medicine. *Pteris semipinnata* has

been studied for its biologically active ingredients for years. Remarkably, in recent years, it has been proved to have bioactive diterpenoid compounds, which could be recognized as potential medication in the future. In our continuous endeavor to search for structurally fascinating natural products from *Pteris semipinnata*, the title compound was found.

The bond distances and bond angles in the title structure are all in expected ranges [5, 6]. The title structure with a 6/6/6/5 ring system of carbon skeleton contains two hydroxyls, one carbonyl, one alkenyl and two methyl groups. These functional groups were identified by the distances $d(C9-O3) = 1.452(5)$ Å, $d(C7-O5) = 1.432(5)$ Å, $d(C15-O4) = 1.208(5)$ Å, $d(C16-C17) = 1.330(6)$ Å, respectively. It is noticeable that this compound also has a lactone ring which was determined by the distances $d(C19-O1) = 1.206(6)$ Å, $d(C19-O2) = 1.357(6)$ Å and $d(C6-O2) = 1.469(5)$ Å.

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