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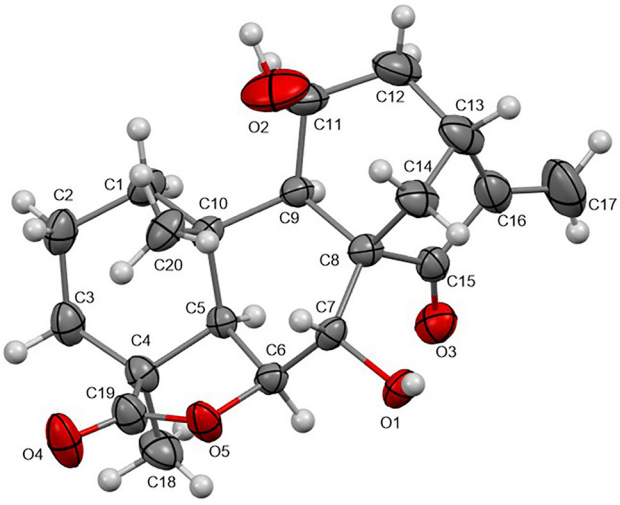
Crystal structure of 7α,11α-dihydroxy-15-oxo-ent-kauran-16-en-19,6β-olide, $C_{20}H_{26}O_5$



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Abstract

$C_{20}H_{26}O_5$, monoclinic, $P2_12_12_1$ (no. 19), a = 7.5455(15) Å, b = 12.735(3) Å, c = 18.333(4) Å, V = 1761.6(6) Å^3, Z = 4, $R_{gt}(F)$ = 0.0529, $wR_{ref}(F^2)$ = 0.1407, T = 273(2) K.

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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.

1 Source of material

The acrial part of *Pteris dispar* Kunze collected from Liping County, Guizhou Province were extracted with methanol for three times (once every five days) at room temperature. The combined filtrate was concentrated and extracted with ethyl acetate. The crude product (348 g) was treated by MCI column with methanol/water (volume radio 50–95%) to obtain five fractions of A–F. The title compound was further purified and isolated from D fraction by silica gel column chromatography. The white needle crystal of the title compound (28 mg) suitable for X-ray diffraction was obtained after recrystallization with ethyl acetate for three days.

Table 1: Data collection and handling.

Crystal:	Colourless needle
Size:	0.24 × 0.21 × 0.19 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.09 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	28.3°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	27,421, 4336, 0.061
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2967
$N(param)_{refined}$:	232
Programs:	Bruker [1], SHELX [2, 3], Olex2 [4]

2 Experimental details

The carbon-bound hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms with d(C–H) = 0.82–0.98 Å, U_{iso}(H) = 1.5times U_{eq}(C) and 1.2times U_{eq}(O).

3 Comment

Ent-kauranediterpene is the main active ingredient of *Pteris*. In recent years, many scholars have reported that there were a certain amount of ent-kauranediterpenes found in *Pteris dispar* Kunze. Such as geopyxin B, geopyxin E, neolaxiflorin L and so on [5, 6]. Combined with the literature review, ent-kauranediterpene showed good inhibitory activity against breast cancer (MCF-7,MDA–MB-231 and SK–BR-3), colorectal cancer, nasopharyngeal cancer(CNE-2Z), liver cancer, lung cancer (A549), laryngeal cancer and other tumors [7–12]. The title compound is a member of ent-kauranediterpene, and we hypothesize that it also has

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.8471 (5)	0.2771 (3)	0.4121 (2)	0.0518 (9)
C2	0.8452 (5)	0.1959 (3)	0.3505 (2)	0.0569 (10)
C3	0.6592 (5)	0.1614 (3)	0.3310 (2)	0.0527 (9)
C4	0.5187 (5)	0.2482 (2)	0.32738 (17)	0.0414 (7)
C5	0.5469 (4)	0.3384 (2)	0.38096 (15)	0.0321 (6)
C6	0.4294 (4)	0.4240 (2)	0.34983 (16)	0.0368 (7)
C7	0.4855 (4)	0.5357 (2)	0.36708 (16)	0.0354 (7)
C8	0.5843 (4)	0.5440 (2)	0.44028 (18)	0.0371 (7)
C9	0.7307 (4)	0.4558 (2)	0.45429 (17)	0.0373 (7)
C10	0.7414 (4)	0.3755 (2)	0.39092 (17)	0.0374 (7)
C11	0.9037 (5)	0.5030 (3)	0.4829 (3)	0.0613 (11)
C12	0.8702 (6)	0.5773 (3)	0.5468 (3)	0.0694 (12)
C13	0.7071 (6)	0.6488 (3)	0.5343 (2)	0.0668 (12)
C14	0.6623 (5)	0.6541 (3)	0.4531 (2)	0.0545 (9)
C15	0.4622 (5)	0.5354 (3)	0.50565 (17)	0.0431 (8)
C16	0.5398 (6)	0.6022 (3)	0.5643 (2)	0.0602 (10)
C17	0.4570 (9)	0.6200 (5)	0.6270 (2)	0.1008 (19)
C18	0.3348 (5)	0.1973 (3)	0.3375 (2)	0.0611 (10)
C19	0.5020 (5)	0.3094 (3)	0.25723 (18)	0.0495 (8)
C20	0.8288 (5)	0.4218 (3)	0.3219 (2)	0.0524 (9)
H3	0.663376	0.126622	0.283979	0.063*
H5	0.499969	0.317523	0.428649	0.039*
H5A	0.852105	0.536358	0.590738	0.083*
H6	0.767329	0.665714	0.423659	0.065*
H7	0.576090	0.708646	0.442898	0.065*
H8	0.727567	0.719160	0.554172	0.080*
H9	0.974162	0.620896	0.554102	0.083*
H10	0.980744	0.445854	0.499507	0.074*
H11	0.796315	0.246362	0.455747	0.062*
H12	0.913651	0.135097	0.365330	0.068*
H14	0.244240	0.248492	0.328137	0.092*
H16	0.322155	0.139721	0.304160	0.092*
H17	0.564196	0.560466	0.328185	0.043*
H17A	0.346407	0.590269	0.635595	0.121*
H17B	0.509614	0.662205	0.662257	0.121*
H18A	0.307287	0.413062	0.366314	0.044*
H18	0.323600	0.172052	0.386668	0.092*
H20	0.901358	0.225795	0.307694	0.068*
H21	0.968583	0.296653	0.422828	0.062*
H22	0.955360	0.420081	0.327170	0.079*
H23	0.794863	0.381076	0.280111	0.079*
H24	0.790580	0.493137	0.315459	0.079*
H25	0.684333	0.415208	0.495478	0.045*
H33	0.621109	0.109786	0.366628	0.063*
O1	0.3321 (3)	0.6009 (2)	0.36854 (13)	0.0456 (6)
H1A	0.358 (2)	0.666 (3)	0.345 (2)	0.068*
O2	0.9913 (5)	0.5581 (4)	0.4242 (2)	0.1079 (13)
H2A	1.119 (12)	0.555 (5)	0.431 (2)	0.162*
O3	0.3277 (3)	0.4826 (2)	0.50977 (13)	0.0564 (7)
O4	0.5312 (5)	0.2806 (2)	0.19502 (13)	0.0695 (9)
O5	0.4395 (4)	0.40646 (18)	0.27029 (12)	0.0506 (6)

antitumor effects. Therefore, it is significant to investigate further examples of ent-kauranediterpene along with their unambiguous structures.

The title molecular structure consists of three six-membered rings, one five-membered ring and one lactone ring, it contains one keto, one alkenyl, two hydroxyl and two methyl groups (see the figure). The bond lengths and angles derived from the compound are within the normal range. The keto bonds were confirmed by the distance of 1.220(4) Å(C_{15}–O_3). The olefinic bond was identified by the distance of 1.327(7) Å(C16–C17). And the hydroxyl groups were confirmed by the distance of 0.95(4) Å(O1–H1A) and 0.97(9) Å(O2–H2A), respectively.

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

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