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Crystal structure of-(1*S*,4a*R*,5*S*)-5,6,7-trihydroxy-8-isopropyl-1-methyl-1,2,3,4,5,10,11,11a-octahydro-4a,1-(epoxymethano)dibenzo[*a*,*d*][7]annulen-13-one $C_{20}H_{26}O_5$

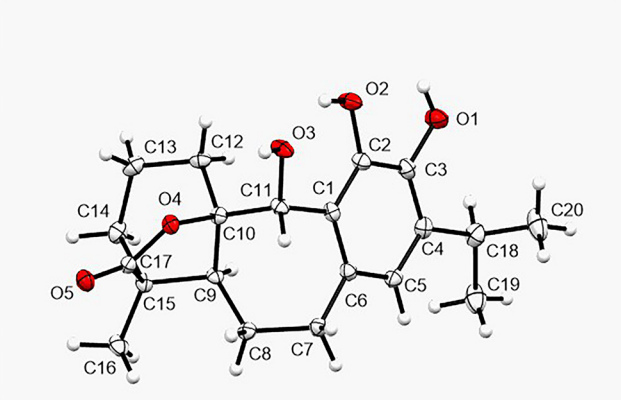

Table 1: Data collection and handling.

Crystal:	Clear light colourless block
Size:	0.15 × 0.13 × 0.12 mm
Wavelength:	Cu *K*α radiation (1.54184 Å)
μ:	0.76 mm^{-1}
Diffractometer, scan mode:	Rigaku Oxford Diffraction, ω scans
θ_{max}, completeness:	75.0°, 99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	21438, 6770, 0.015
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 6,753
$N(param)_{refined}$:	464
Programs:	Rigaku[1], Olex2[2], SHELX[3,4]

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Abstract

$C_{20}H_{26}O_5$, monoclinic, $P2_1$, a = 14.0782(12) Å, b = 8.6399(7) Å, c = 14.6891(13) Å, β = 101.9593(9), V = 1747.93(3) $Å^3$, Z = 4, $R_{gt}(F)$ = 0.0275, $wR_{ref}(F^2)$ = 0.0736, T = 100(1) K.

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The molecular structure is shown in the figure. Table 1 contains the crystallographic data and the list of the atoms including atomic coordinates and displacement parameters can be found in the cif-file attached to this article.

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1 Source of material

Isodon lophanthoides is a plant of the genus Isodon, belonging to Labiatae family. It has a variety of chemical constituents, and has anti-viral, anti-tumor and anti-inflammatory activities. It has broad research prospects. However, there are few chemical studies on *I. lophanthoides*. In order to explore the bioacive material basis of chemical constituents in *I. lophanthoides*, we systematically studied the chemical constituents of *I. lophanthoides*.

The aerial part (7.16 kg) of *I. lophanthoides* was collected from Baise City, Guangxi Zhuang Autonomous Region. The plants were pulverized after air-drying, and impregnated with 70 %, 90 %, and 100 % methanol at room temperature for 2 times (7 days each time). The total aqueous extract (1.68 kg) was obtained by vacuum concentration. The extract was dissolved in 55 °C hot water and extracted with equal volume of petroleum ether, ethyl acetate and n-butanol to obtain three corresponding parts. Petroleum ether fraction (154.2 g), was subjected to silica gel column chromatography, eluted with petroleum/ether-ethyl acetate (50:1, 20:1, 10:1, 5:1, 1:1) to obtain ten sub-fractions (A1–A10), some of which were isolated and purified from A6 by repeated silica gel column chromatography and Sephadex LH-20 gel column chromatography to obtain the crude title compound (60 mg). The title compound was then further recrystallized with methanol to give white bulk crystals, and its systematic name was (1*S*,4a*R*,5*S*)-5,6,7-trihydroxy-8-isopropyl-1-methyl-

1,2,3,4,5,10,11,11a-octahydro-4a,1-(epoxymethano)dibenzo[*a*, *d*][7]annulen-13-one.

2 Comment

I. lophanthoides is a plant of the genus *Isodon*.[5] It is a ethnic medicine with the effects of clearing damp heat, promoting blood circulation and removing blood stasis, detoxification and detumescence. Therefore, it is used in the treatment of acute icteric hepatitis, acute cholecystitis, laryngopharyngitis, gynecological diseases, tumor leprosy.[6] Previous studies have shown that the plants of this genus have good biological activities, including antioxidant, antibacterial, anti-inflammatory, anti-tumor and so on.[7,8] In order to better develop and utilize *Pteris multifida*, its chemical constituents were preliminarily studied and the title compound was obtained. The title compound is composed of three methyl groups, two six-membered rings, one seven-membered ring, one ester group and three hydroxyl groups (see the figure), some of which were confirmed by the distances *d*(O1–C3) = 1.369(2) Å, *d*(O2–C2) = 1.372(2) Å, *d*(O3–C11) = 1.429(2) Å, *d*(O4–C17) = 1.349(2) Å, *d*(O5–C17) = 1.211(2) Å. And the substituents on positions C-11, is α configuration, C-15 is β configuration.

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