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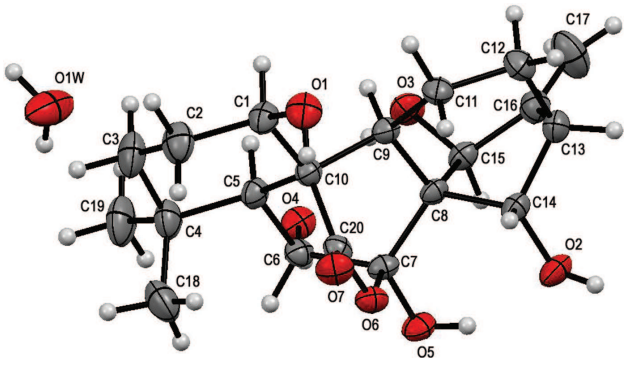
Crystal structure of *Ent*-7*β*,20-epoxy-kaur-16-en-1*β*,6*α*,7*α*,14*α*,15*α*-pentaol-20-one, $C_{20}H_{30}O_8$



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Abstract

$C_{20}H_{30}O_8$, monoclinic, $C2$ (no. 5), $a = 21.684(13)$ Å, $b = 7.275(4)$ Å, $c = 14.320(9)$ Å, $\beta = 118.975(11)$, $V = 1976(2)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0362$, $wR_{ref}(F^2) = 0.0920$, $T = 298$ K.

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The title crystal structure (systematic name: (1*R*,4a*S*,5*R*,6a*S*,7*S*,9*R*,11a*S*,14*S*)-1,5,6,7,14-pentahydroxy-4,4-dimethyl-8-methylenedodecahydro-1*H*-6,11b-(epoxymethano)-6a,9-methanocyclohepta[*a*]naphthalen-12-one) 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 experimental material was collected in Zhong county, Chongqing city, PRC, in the August of 2009, authenticated by Professor Junhua Zhao of the Guiyang College of Traditional Chinese Medicine as Isodon rubescens. The air-dried material (30 kg) was extracted with 95% methanol (30 L × 3) after comminution. The extraction was then concentrated to

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.28 × 0.15 × 0.12 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.10 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II CCD, φ and ω
θ_{max}, completeness:	25.0°, 99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	6731, 3180, 0.028
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2782
$N(param)_{refined}$:	265
Programs:	OLEX2 [1], Bruker [3], SHELX [4, 5]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O6	0.82004(10)	0.1521(3)	0.83061(14)	0.0431(5)
O4	0.70911(11)	0.2527(3)	0.54417(16)	0.0513(5)
H4	0.6972	0.1516	0.5156	0.077*
O2	0.94759(11)	0.0481(3)	0.77058(18)	0.0552(6)
H2	0.9854	0.0062	0.8160	0.083*
O5	0.81147(10)	−0.0093(3)	0.68756(16)	0.0491(5)
H5	0.8528	−0.0352	0.7077	0.074*
O3	0.81428(10)	0.4196(3)	0.53502(16)	0.0568(6)
H3	0.7749	0.3795	0.5180	0.085*
O1	0.84642(11)	0.6845(3)	0.95572(17)	0.0595(6)
H1	0.8487	0.6006	0.9956	0.089*
O7	0.83642(12)	0.3135(3)	0.97054(17)	0.056(6)
C7	0.80727(14)	0.1664(4)	0.7188(2)	0.0386(6)
C8	0.86364(13)	0.2984(4)	0.7232(2)	0.0354(6)
C10	0.79511(13)	0.4740(4)	0.8000(2)	0.0381(6)
C13	0.98124(15)	0.3701(4)	0.7687(2)	0.0471(7)
H13	1.0294	0.3269	0.7922	0.057*
C5	0.72399(13)	0.4222(4)	0.6981(2)	0.0424(7)
H5A	0.7180	0.5125	0.6436	0.051*
C20	0.81831(14)	0.3115(4)	0.8752(2)	0.0408(7)
C1	0.78786(14)	0.6534(5)	0.8511(2)	0.0472(7)
H1A	0.7873	0.7548	0.8057	0.057*
C9	0.85221(13)	0.4900(4)	0.7619(2)	0.0362(6)
H9	0.8327	0.5719	0.6998	0.043*
C6	0.73157(13)	0.2358(4)	0.6551(2)	0.0410(7)
H6	0.7005	0.1477	0.6638	0.049*
C11	0.92205(14)	0.5767(4)	0.8466(2)	0.0453(7)
H11A	0.9143	0.7059	0.8541	0.054*
H11B	0.9365	0.5177	0.9148	0.054*
C15	0.86578(14)	0.3079(4)	0.6163(2)	0.0423(7)
H15	0.8615	0.1827	0.5885	0.051*
C14	0.93987(14)	0.2325(4)	0.7985(2)	0.0423(7)
H14	0.9521	0.2427	0.8738	0.051*
C4	0.65640(15)	0.4365(5)	0.7094(3)	0.0600(9)
C16	0.93917(16)	0.3777(5)	0.6494(3)	0.0522(8)

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
C12	0.98131(16)	0.5588(4)	0.8184(3)	0.0527(8)
H12A	1.0262	0.5761	0.8825	0.063*
H12B	0.9763	0.6552	0.7684	0.063*
C2	0.72062(17)	0.6673(6)	0.8585(3)	0.0668(9)
H2A	0.7226	0.5801	0.9111	0.080*
H2B	0.7171	0.7897	0.8825	0.080*
C3	0.65578(18)	0.6292(6)	0.7524(3)	0.0732(11)
H3A	0.6529	0.7199	0.7007	0.088*
H3B	0.6141	0.6420	0.7606	0.088*
C18	0.6521(2)	0.2901(6)	0.7828(3)	0.0810(12)
H18A	0.6485	0.1707	0.7520	0.122*
H18B	0.6114	0.3123	0.7910	0.122*
H18C	0.6937	0.2954	0.8513	0.122*
C19	0.59212(17)	0.4185(7)	0.5972(3)	0.0851(13)
H19A	0.5932	0.5150	0.5523	0.128*
H19B	0.5496	0.4279	0.6020	0.128*
H19C	0.5935	0.3015	0.5672	0.128*
C17	0.9603(2)	0.4375(8)	0.5836(3)	0.0886(14)
H17A	1.0060	0.4816	0.6099	0.106*
H17B	0.9296	0.4360	0.5105	0.106*
O1W	0.42572(14)	0.4888(4)	0.0620(2)	0.0963(10)
H1WA	0.3991	0.5821	0.0283	0.144*
H1WB	0.4037	0.3941	0.0229	0.144*

give a crude product (3 kg), which was continuously divided into 5 fractions (A—E) after subjected to silica gel column eluting with petroleum ether/acetic ether (95:5—6:4). Subsequently, fraction C (653 g) was applied to MCI column separation to afford subfraction C1—C3. C2 was further separated by silica gel column, Sephadex LH-20 to produce the title compound (18 mg). Suitable colorless block crystals of this compound were then obtained after recrystallization from methanol.

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. The hydrogen atoms at the hydroxyl groups are refined freely.

Comment

Recent studies show that there are abundant diterpenoids with various structures possessing anti-cancer activity found from Isodon rubescens [2]. In order to discover more compounds with diverse bioactivities, our group has studied on the same species distributed in Zhong county, Chongqing city and the title compound, namely rabdoternin B, has been isolated.

The asymmetric unit of the title compound consists of one formula unit. Geometric parameters of the title structure are in the usual ranges. The compound contains five hydroxyl groups, one lactone and three methyl-groups. Moreover, the skeleton of this compound contains three six-membered carbon rings and one five-membered ring. The six-membered ring A (atoms C1—C5/C10) shows a chair conformation, while the other six-membered rings B (atoms C5—C10) and C (atoms C8—C9/C11—C14) present boat conformations. In addition, the five-membered carbon ring is in a twist-chair conformation. The intermolecular connection was achieved through hydrogen bonds.

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