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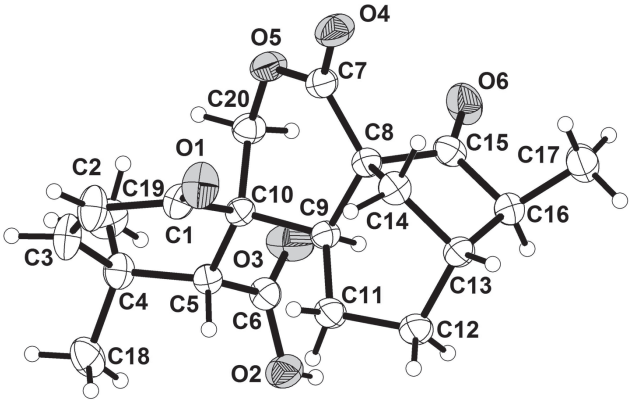
Crystal constructure of 16(*S*)-methyl-6α-carboxy-1, 15-dioxo-6, 7-seco-*ent*-kaur-2-en-7, 20-olide, $C_{20}H_{24}O_6$



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Abstract

$C_{20}H_{24}O_6$, orthorhombic, $P2_12_12_1$ (no. 19), $a = 7.697(14)$ Å, $b = 13.91(2)$ Å, $c = 16.75(3)$ Å, $V = 1794$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0430$, $wR_{ref}(F^2) = 0.1087$, $T = 293$ K.

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The asymmetric unit of the crystal structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

The leaves of *Isodon rubescens* were collected in Xishui city of Guizhou province, China in June 2014 and identified by Prof. J. H. Zhao. The dried and powdered leaves (8 kg) were extracted with MeOH (× 3) and filtered. The filtrate was concentrated and extracted with ethyl acetate to give a residue. The residue

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Table 1: Data collection and handling.

Crystal:	Colourless blocks
Size:	0.24 × 0.23 × 0.21 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	1.0 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$2\theta_{max}$, completeness:	52°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	12305, 3327, 0.056
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2512
$N(param)_{refined}$:	236
Programs:	SHELX [2], Bruker programs [3]

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

Atom	x	y	z	U_{iso}*/U_{eq}
C1	0.2181(3)	0.20318(17)	0.20048(13)	0.0484(6)
C2	0.0937(4)	0.1500(2)	0.24967(15)	0.0645(8)
H2	0.0493	0.0924	0.2303	0.077*
C3	0.0426(4)	0.1804(2)	0.31994(15)	0.0653(8)
H3	−0.0314	0.1401	0.3483	0.078*
C4	0.0909(3)	0.27313(18)	0.35911(13)	0.0503(6)
C5	0.1925(3)	0.33691(15)	0.29904(12)	0.0403(5)
H5	0.1037	0.3665	0.2651	0.048*
C6	0.2808(3)	0.41966(17)	0.34100(13)	0.0466(6)
C7	0.6013(3)	0.21400(18)	0.15093(15)	0.0514(6)
C8	0.5430(3)	0.31356(16)	0.12817(13)	0.0436(6)
C9	0.3919(3)	0.35780(16)	0.17948(12)	0.0408(5)
H9	0.4449	0.4093	0.2111	0.049*
C10	0.3190(3)	0.28497(15)	0.24043(12)	0.0399(5)
C11	0.2579(3)	0.40658(18)	0.12593(14)	0.0520(6)
H11A	0.1925	0.3578	0.0975	0.062*
H11B	0.1771	0.4426	0.1588	0.062*
C12	0.3424(3)	0.47436(19)	0.06578(15)	0.0551(7)
H12A	0.3690	0.5347	0.0920	0.066*
H12B	0.2604	0.4877	0.0233	0.066*
C13	0.5102(3)	0.43320(17)	0.02926(14)	0.0472(6)
H13	0.5198	0.4510	−0.0272	0.057*
C14	0.5083(3)	0.32376(17)	0.03875(13)	0.0490(6)
H14A	0.3966	0.2968	0.0241	0.059*
H14B	0.5989	0.2936	0.0073	0.059*
C15	0.6958(3)	0.38373(18)	0.13675(13)	0.0475(6)
C16	0.6755(3)	0.46194(17)	0.07494(14)	0.0492(6)
H16	0.6560	0.5235	0.1019	0.059*
C17	0.8374(3)	0.4690(2)	0.02353(16)	0.0636(7)

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
H17A	0.8226	0.5192	−0.0152	0.095*
H17B	0.8560	0.4090	−0.0034	0.095*
H17C	0.9358	0.4832	0.0566	0.095*
C18	−0.0779(4)	0.3264(2)	0.38106(16)	0.0664(8)
H18A	−0.0498	0.3867	0.4058	0.100*
H18B	−0.1445	0.2879	0.4174	0.100*
H18C	−0.1446	0.3379	0.3336	0.100*
C19	0.1867(4)	0.2507(2)	0.43728(14)	0.0699(8)
H19A	0.2192	0.3097	0.4629	0.105*
H19B	0.2890	0.2137	0.4259	0.105*
H19C	0.1117	0.2146	0.4719	0.105*
C20	0.4757(3)	0.23948(18)	0.28090(13)	0.0492(6)
H20A	0.5497	0.2895	0.3025	0.059*
H20B	0.4369	0.1995	0.3248	0.059*
O1	0.2457(3)	0.18088(13)	0.13163(10)	0.0695(6)
O2	0.1920(2)	0.49999(13)	0.33388(12)	0.0660(5)
H2A	0.2433	0.5434	0.3571	0.099*
O3	0.4139(3)	0.41425(14)	0.37736(12)	0.0813(7)
O4	0.6800(3)	0.16234(13)	0.10546(11)	0.0695(5)
O5	0.5744(2)	0.18158(12)	0.22487(10)	0.0572(5)
O6	0.8116(2)	0.37705(15)	0.18469(11)	0.0726(6)

was dissolved in MeOH and ethyl acetate, which was applied to silica gel column eluting with chloroform/acetone (8:2). The title compound, isolated from silica gel column with chloroform/acetone (7:3), was obtained as colourless needles by recrystallization in methanol.

Experimental details

Hydrogen atoms were positioned geometrically and refined using a riding model, with C—H = 0.93–0.97 Å, with $U_{iso}(H) = 1.2U_{eq}(C)$ and $U_{iso}(H) = 1.5U_{eq}(O)$.

Discussion

Experimental study shows that the main chemical compositions of *Isodon rubescens* are ent-kaurene diterpenoids. These compounds have antibacterial activity, anti-inflammatory activity and are resistant to a variety of animals with implanted tumor [1].

The title compound contains two ketone, one carboxy, one lactone, one olefine and three methyl-groups. The bond distances of $C_1—O_1$, $C_{15}—O_6$ and $C_7—O_4$ are 1.213(3) Å, 1.203(3) Å and 1.210(3) Å, which are typical for C = O groups. The carboxy group was confirmed by the distance of 1.316(3) Å and 1.194(3) Å, $C_6—O_2$ and $C_6—O_3$, respectively. The bond distances of $C_7—O_5$ and $C_{20}—O_5$ are 1.335(3) Å and 1.452(3) Å, which clearly confirmed olefinic moieties.

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