Crystal structure of 17-(3-methyl-2-butenoyl)-ent-kaurane-7*a*,16*b*-diol, $C_{25}H_{40}O_4$

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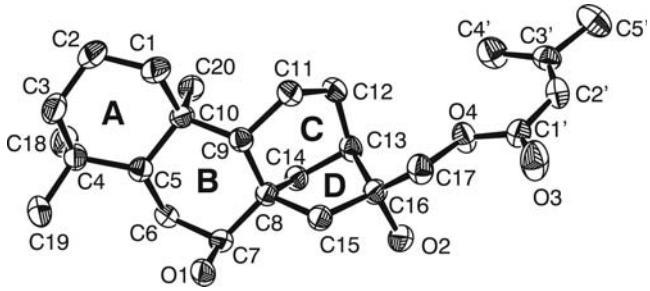

Abstract

$C_{25}H_{40}O_4$, monoclinic, $P2_1$ (no. 4), a = 6.6906(9) Å, b = 14.046(2) Å, c = 11.968(2) Å, β = 101.271(5)°, V = 1103.0 Å^3, Z = 2, $R_{gt}(F)$ = 0.044, $wR_{ref}(F^2)$ = 0.125, T = 293 K.

Source of material

The air-dried powdered leaves of Isodon sculponeata (*Rabdosia*, Labiatae family) were extracted with 95 % methanol at room temperature. The extract was concentrated to give a residue, which was fractionalized by silica gel column chromatography eluted with petroleum ether (PE)/ethyl acetate to yield fractions A-F. Fraction E eluted by 20 % ethyl acetate was separated on silica gel to obtain a sub-fraction (PE/ethyl acetate = 10 : 1), which was further isolated and purified by silica gel and crystallized from methanol affording the title compound as colourless needles (m.p. 343 K; $[\alpha]_D$ = –16.7).

Discussion

The title compound is a natural ent-kaurane diterpenoid and shows some biological activity in treating dysentery and beriberi [1]. In order to confirm the molecular structure and conformation, the crystal structure analysis has been undertaken.

The compound crystallizes with one molecule in the asymmetric unit. Geometric parameters are in the usual ranges. The double bonds are clearly identified by the distance of 1.329(4) Å between C2' and C3' and 1.204(3) Å between C1' and O3. The single bond is identified by the distance of 1.452(3) Å between C17 and O4. The d(C1'—O4) of 1.335(3) Å is less than a typical C—O single bond, indicating that C17-O4-O5 is an ester bond. The rings **A** (C1/C2/C3/C4/C5/C10), **B** (C5/C6/C7/C8/C9/C10) and **C** (C8/C9/C11/C12/C13/C14) are six-membered, while **D** (C8/C13/C14/C15/C16) is a five-membered ring. Rings **A** and **B** have the chair conformation, while ring **C** has a distorted chair conformation.

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Table 1. Data collection and handling.

Crystal:	colorless needle, size 0.21 × 0.23 × 0.25 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.80 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	49.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	9545, 3802
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3358
$N(param)_{refined}$:	262
Programs:	SHELXS-97, SHELXL-97 [2]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1A)	2*a*	0.9516	0.1891	0.5119	0.054
H(1B)	2*a*	0.8153	0.1026	0.4616	0.054
H(2A)	2*a*	0.7093	0.2416	0.3571	0.061
H(2B)	2*a*	0.5319	0.2022	0.4127	0.061
H(2')	2*a*	1.4943	–0.4158	0.8949	0.049
H(3A)	2*a*	0.5616	0.3651	0.4424	0.055
H(3B)	2*a*	0.7916	0.3539	0.4993	0.055
H(4'1)	2*a*	0.9077	–0.4187	0.7623	0.079
H(4'2)	2*a*	1.0134	–0.3245	0.7342	0.079
H(4'3)	2*a*	0.9687	–0.3407	0.8566	0.079
H(5)	2*a*	0.8782	0.2690	0.6890	0.037
H(5'1)	2*a*	1.0741	–0.5429	0.7758	0.089
H(5'2)	2*a*	1.2547	–0.5542	0.8803	0.089
H(5'3)	2*a*	1.2999	–0.5378	0.7581	0.089
H(6A)	2*a*	0.6728	0.2858	0.8305	0.040
H(6B)	2*a*	0.5859	0.1839	0.7955	0.040
H(7)	2*a*	0.8559	0.1686	0.9488	0.041
H(9)	2*a*	1.0667	0.1345	0.6921	0.037
H(11A)	2*a*	0.9615	–0.0067	0.5706	0.047
H(11B)	2*a*	1.1580	–0.0080	0.6658	0.047
H(12A)	2*a*	0.7938	–0.1068	0.6647	0.047
H(12B)	2*a*	1.0159	–0.1475	0.6934	0.047
H(13)	2*a*	0.8745	–0.1406	0.8617	0.040
H(14A)	2*a*	0.7860	0.0082	0.9277	0.039
H(14B)	2*a*	0.6784	0.0058	0.7977	0.039
H(15A)	2*a*	1.2638	0.0641	0.8474	0.042
H(15B)	2*a*	1.1936	0.0804	0.9640	0.042
H(17A)	2*a*	1.3494	–0.0927	0.8053	0.049
H(17B)	2*a*	1.4421	–0.0997	0.9364	0.049
H(18A)	2*a*	0.3400	0.2914	0.6696	0.076
H(18B)	2*a*	0.3399	0.2376	0.5549	0.076
H(18C)	2*a*	0.2962	0.3473	0.5540	0.076
H(19A)	2*a*	0.7875	0.4279	0.6733	0.078
H(19B)	2*a*	0.6085	0.4123	0.7385	0.078
H(19C)	2*a*	0.5667	0.4619	0.6189	0.078
H(20A)	2*a*	0.5320	0.0697	0.6738	0.062
H(20B)	2*a*	0.5940	0.0228	0.5667	0.062
H(20C)	2*a*	0.4595	0.1150	0.5530	0.062
H(1)	2*a*	1.1493	0.2309	0.9263	0.068
H(2)	2*a*	1.1248	–0.1307	1.0353	0.067

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	2*a*	0.8153(4)	0.1626(2)	0.5017(2)	0.055(2)	0.051(2)	0.030(1)	0.006(1)	0.011(1)	0.001(1)
C(1')	2*a*	1.4343(4)	–0.2739(2)	0.8976(2)	0.033(1)	0.037(1)	0.039(1)	0.002(1)	0.004(1)	0.005(1)
C(2)	2*a*	0.6667(5)	0.2305(2)	0.4290(2)	0.066(2)	0.058(2)	0.029(1)	0.008(1)	0.007(1)	0.006(1)
C(2')	2*a*	1.3848(4)	–0.3745(2)	0.8739(2)	0.038(2)	0.035(1)	0.051(2)	0.009(1)	0.009(1)	0.005(1)
C(3')	2*a*	1.2106(4)	–0.4169(2)	0.8277(2)	0.048(2)	0.036(1)	0.036(1)	–0.004(1)	0.014(1)	–0.003(1)
C(3)	2*a*	0.6589(5)	0.3238(2)	0.4901(2)	0.053(2)	0.042(2)	0.040(2)	0.003(1)	0.001(1)	0.012(1)
C(4)	2*a*	0.6005(4)	0.3164(2)	0.6071(2)	0.037(1)	0.031(1)	0.040(1)	0.002(1)	0.001(1)	0.002(1)
C(4')	2*a*	1.0070(4)	–0.3711(2)	0.7920(3)	0.042(2)	0.045(2)	0.068(2)	–0.007(1)	0.006(1)	–0.007(1)
C(5)	2*a*	0.7417(3)	0.2409(2)	0.6776(2)	0.027(1)	0.032(1)	0.033(1)	–0.003(1)	0.0026(9)	0.000(1)
C(5')	2*a*	1.2098(5)	–0.5224(2)	0.8088(3)	0.067(2)	0.040(2)	0.076(2)	–0.008(1)	0.025(2)	–0.012(1)
C(6)	2*a*	0.7029(4)	0.2253(2)	0.7985(2)	0.039(1)	0.028(1)	0.034(1)	0.001(1)	0.007(1)	–0.0045(9)
C(7)	2*a*	0.8881(4)	0.1809(2)	0.8736(2)	0.042(1)	0.029(1)	0.029(1)	–0.002(1)	0.003(1)	–0.0006(9)
C(8)	2*a*	0.9484(3)	0.0860(2)	0.8250(2)	0.029(1)	0.026(1)	0.032(1)	–0.0042(9)	0.0030(9)	0.001(1)
C(9)	2*a*	0.9507(3)	0.0930(2)	0.6962(2)	0.030(1)	0.029(1)	0.033(1)	–0.0018(9)	0.0047(9)	–0.001(1)
C(10)	2*a*	0.7632(4)	0.1438(2)	0.6195(2)	0.036(1)	0.032(1)	0.027(1)	–0.004(1)	0.0030(9)	0.0003(9)
C(11)	2*a*	1.0104(4)	–0.0045(2)	0.6524(2)	0.046(2)	0.035(1)	0.036(1)	0.006(1)	0.010(1)	–0.002(1)
C(12)	2*a*	0.9316(4)	–0.0936(2)	0.7047(2)	0.043(1)	0.028(1)	0.042(1)	0.001(1)	0.001(1)	–0.006(1)
C(13)	2*a*	0.9316(4)	–0.0836(2)	0.8325(2)	0.037(1)	0.022(1)	0.041(1)	–0.005(1)	0.007(1)	0.003(1)
C(14)	2*a*	0.8087(4)	0.0047(2)	0.8502(2)	0.035(1)	0.029(1)	0.035(1)	–0.002(1)	0.0066(9)	0.003(1)
C(15)	2*a*	1.1585(4)	0.0498(2)	0.8902(2)	0.037(1)	0.028(1)	0.038(1)	–0.005(1)	–0.001(1)	0.0004(9)
C(16)	2*a*	1.1402(4)	–0.0594(2)	0.9048(2)	0.039(1)	0.028(1)	0.035(1)	–0.004(1)	0.001(1)	0.002(1)
C(17)	2*a*	1.3222(4)	–0.1129(2)	0.8784(2)	0.036(1)	0.030(1)	0.054(2)	–0.007(1)	0.002(1)	0.003(1)
C(18)	2*a*	0.3730(4)	0.2963(2)	0.5953(3)	0.040(2)	0.053(2)	0.055(2)	0.008(1)	0.001(1)	0.004(1)
C(19)	2*a*	0.6450(5)	0.4137(2)	0.6648(3)	0.063(2)	0.032(1)	0.056(2)	0.009(1)	–0.001(1)	0.003(1)
C(20)	2*a*	0.5688(4)	0.0820(2)	0.6015(2)	0.040(1)	0.036(1)	0.042(1)	–0.006(1)	–0.006(1)	–0.002(1)
O(1)	2*a*	1.0453(3)	0.2517(1)	0.8859(2)	0.047(1)	0.0261(8)	0.054(1)	–0.0043(8)	–0.0136(8)	–0.0031(8)
O(2)	2*a*	1.1357(3)	–0.0737(1)	1.0230(1)	0.061(1)	0.0342(9)	0.0358(9)	0.0000(9)	0.0032(8)	0.0060(7)
O(3)	2*a*	1.6058(3)	–0.2468(2)	0.9325(2)	0.037(1)	0.046(1)	0.097(2)	0.0006(9)	–0.011(1)	0.005(1)
O(4)	2*a*	1.2764(2)	–0.2140(1)	0.8758(2)	0.032(1)	0.0280(9)	0.060(1)	0.0005(7)	0.0023(8)	–0.0001(8)

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