Crystal structure of 1-oxo-6,15-dihydroxy-6,7-seco-ent-kaura-2,16-dien-7,20-olide monohydrate, $C_{20}H_{26}O_5 \cdot H_2O$

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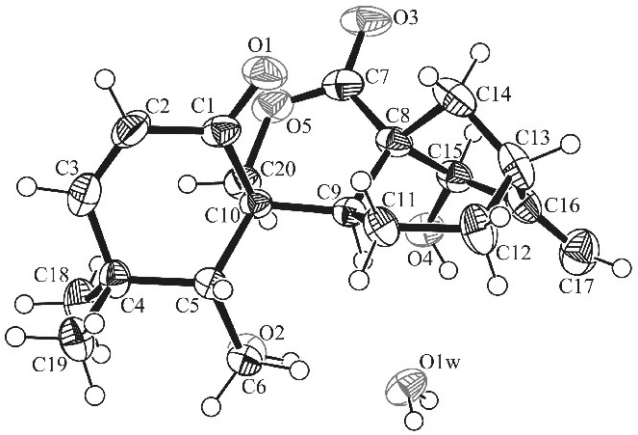

Abstract

$C_{20}H_{28}O_6$, orthorhombic, $P2_12_12_1$ (no. 19), a = 7.444(2) Å, b = 14.026(3) Å, c = 17.298(4) Å, V = 1806.0 Å^3, Z = 4, $R_{gt}(F)$ = 0.036, $wR_{ref}(F^2)$ = 0.086, T = 293 K.

Source of material

The powdered air-dried leaves of *Isodon eriocalyx* var. *laxiflora* were extracted with diethyl ether and the solvent removed under vacuum. The residue was dissolved in methanol and decolorized with activated charcoal. The methanol was extracted by ethyl acetate and the extraction was combined by monitoring with TLC, followed by re-crystallization to yield the compound as a colorless cubic crystal.

Experimental details

The molecule contains no heavy atoms, therefore a meaningful Flack parameter could not be determined with Mo K_α radiation (x= 0.6(10)) and the absolute structure could not be made sure.

Discussion

The title compound is a classic 6,7-seco-ent-kaurane diterpenoid [1]. The geometric parameters of the title compound are in the usual ranges. The compound contains two hydroxyl groups, two olefinic bonds, one carbonyl group, and one lactone. The two hydroxyl groups were confirmed by the distances d(C6—O2) = 1.432(2) Å and d(C15—O4) = 1.403(2) Å. Two olefinic bonds were clearly identified by the distances d(C2—C3) = 1.317(3) Å and d(C16—C17) = 1.313(3) Å. The distances C1—O1 and C7—O3 are 1.223(2) Å and 1.211(2) Å, respectively. The intermolecular connection was established through hydrogen bonding of the water molecule.

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Table 1. Data collection and handling.

Crystal:	colorless block, size 0.24 × 0.26 × 0.28 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.98 cm^{-1}
Diffractometer, scan mode:	Bruker CCD, φ/ω
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	8152, 3126
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2919
$N(param)_{refined}$:	240
Program:	SHELXL-97 [2]

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

Atom	Site	x	y	z	U_{iso}
H(2)	4*a*	0.8470	0.6269	0.3474	0.053
H(3)	4*a*	0.8717	0.5499	0.4570	0.051
H(5)	4*a*	0.7094	0.3638	0.3307	0.031
H(9)	4*a*	0.3515	0.3705	0.2611	0.032
H(20A)	4*a*	0.2675	0.4760	0.3657	0.043
H(20B)	4*a*	0.4105	0.5412	0.4064	0.043
H(15)	4*a*	0.0359	0.4982	0.1654	0.041
H(14A)	4*a*	0.2867	0.5492	0.0863	0.054
H(14B)	4*a*	0.4857	0.5198	0.1081	0.054
H(13)	4*a*	0.3160	0.4000	0.0271	0.055
H(12A)	4*a*	0.4246	0.2720	0.1232	0.055
H(12B)	4*a*	0.5652	0.3276	0.0730	0.055
H(11A)	4*a*	0.6569	0.4180	0.1727	0.041
H(11B)	4*a*	0.6215	0.3201	0.2138	0.041
H(18A)	4*a*	0.5560	0.3730	0.5219	0.071
H(18B)	4*a*	0.5168	0.4808	0.5050	0.071
H(18C)	4*a*	0.6867	0.4517	0.5531	0.071
H(19A)	4*a*	0.8363	0.2975	0.4624	0.061
H(19B)	4*a*	0.9479	0.3837	0.4945	0.061
H(19C)	4*a*	0.9557	0.3582	0.4063	0.061
H(6A)	4*a*	0.4757	0.2669	0.3358	0.037
H(6B)	4*a*	0.5672	0.2592	0.4172	0.037
H(17A)	4*a*	0.0654	0.2673	0.0611	0.065
H(17B)	4*a*	–0.0712	0.2997	0.1283	0.065
H(4)	4*a*	0.0078	0.3547	0.2462	0.058
H(2A)	4*a*	0.2504	0.3030	0.3950	0.09(1)
H(1WB)	4*a*	0.184(2)	0.183(1)	0.2999(3)	0.09(1)
H(1WA)	4*a*	0.004(3)	0.2128(4)	0.3146(1)	0.064(8)

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)	4*a*	0.6573(3)	0.5420(1)	0.2937(1)	0.029(1)	0.026(1)	0.049(1)	0.0016(8)	–0.0005(9)	0.0042(9)
C(2)	4*a*	0.7803(3)	0.5715(2)	0.3543(1)	0.040(1)	0.032(1)	0.060(1)	–0.0106(9)	–0.007(1)	–0.003(1)
C(3)	4*a*	0.8007(3)	0.5226(2)	0.4187(1)	0.039(1)	0.043(1)	0.047(1)	–0.005(1)	–0.008(1)	–0.012(1)
C(4)	4*a*	0.7204(2)	0.4273(1)	0.4359(1)	0.032(1)	0.038(1)	0.0286(9)	0.0032(8)	–0.0023(8)	–0.0060(9)
C(5)	4*a*	0.6152(2)	0.3887(1)	0.3648(1)	0.0224(8)	0.0276(9)	0.0283(9)	0.0040(7)	0.0028(7)	0.0007(7)
C(10)	4*a*	0.5185(2)	0.4659(1)	0.3143(1)	0.0236(8)	0.0238(9)	0.0324(9)	0.0017(7)	–0.0008(8)	0.0023(8)
C(9)	4*a*	0.4301(2)	0.4213(1)	0.2419(1)	0.0209(8)	0.027(1)	0.0326(9)	–0.0012(7)	0.0013(7)	0.0049(8)
C(8)	4*a*	0.3045(2)	0.4930(1)	0.1983(1)	0.0241(9)	0.034(1)	0.040(1)	0.0003(8)	–0.0018(8)	0.0107(9)
C(7)	4*a*	0.2788(2)	0.5892(2)	0.2359(1)	0.0200(9)	0.034(1)	0.063(1)	–0.0013(8)	–0.0040(9)	0.013(1)
O(5)	4*a*	0.3057(2)	0.6000(1)	0.31144(9)	0.0400(8)	0.0285(7)	0.063(1)	0.0104(6)	–0.0044(7)	–0.0006(7)
C(20)	4*a*	0.3671(3)	0.5191(1)	0.3567(1)	0.034(1)	0.031(1)	0.043(1)	0.0075(9)	0.0014(9)	–0.0015(9)
C(15)	4*a*	0.1135(2)	0.4478(1)	0.1860(1)	0.0245(9)	0.037(1)	0.042(1)	0.0009(8)	–0.0045(9)	0.0093(9)
C(16)	4*a*	0.1456(3)	0.3774(2)	0.1208(1)	0.033(1)	0.048(1)	0.037(1)	0.007(1)	–0.0082(9)	0.007(1)
C(14)	4*a*	0.3600(3)	0.5026(2)	0.1132(1)	0.030(1)	0.062(2)	0.042(1)	–0.002(1)	–0.0025(9)	0.024(1)
C(13)	4*a*	0.3244(3)	0.4014(2)	0.0837(1)	0.037(1)	0.073(2)	0.028(1)	0.001(1)	–0.0056(9)	0.009(1)
C(12)	4*a*	0.4749(3)	0.3345(2)	0.1130(1)	0.037(1)	0.068(2)	0.032(1)	0.008(1)	0.0005(9)	–0.005(1)
C(11)	4*a*	0.5642(2)	0.3726(2)	0.1869(1)	0.0277(9)	0.044(1)	0.032(1)	0.0056(9)	–0.0003(8)	0.0018(9)
C(18)	4*a*	0.6095(3)	0.4338(2)	0.5111(1)	0.044(1)	0.067(2)	0.032(1)	0.003(1)	0.0017(9)	–0.010(1)
C(19)	4*a*	0.8801(3)	0.3604(2)	0.4512(1)	0.034(1)	0.051(1)	0.037(1)	0.005(1)	–0.0078(9)	–0.001(1)
C(6)	4*a*	0.5004(2)	0.3013(1)	0.3832(1)	0.030(1)	0.030(1)	0.0323(9)	0.0031(8)	0.0009(8)	0.0043(8)
C(17)	4*a*	0.0359(3)	0.3081(2)	0.1015(1)	0.046(1)	0.058(2)	0.059(1)	0.002(1)	–0.008(1)	–0.009(1)
O(1)	4*a*	0.6568(2)	0.5814(1)	0.23057(9)	0.0392(8)	0.0466(9)	0.0592(9)	–0.0113(7)	–0.0072(7)	0.0237(8)
O(3)	4*a*	0.2253(2)	0.6579(1)	0.2003(1)	0.0353(8)	0.0373(8)	0.091(1)	0.0046(7)	–0.0092(8)	0.0275(8)
O(4)	4*a*	0.0283(2)	0.4117(1)	0.25215(8)	0.0300(6)	0.0376(8)	0.0487(8)	–0.0045(6)	0.0073(6)	0.0028(7)
O(2)	4*a*	0.3340(2)	0.3263(1)	0.41945(8)	0.0297(7)	0.0477(9)	0.0447(8)	–0.0023(7)	0.0059(6)	0.0045(7)
O(1W)	4*a*	0.1168(2)	0.2298(1)	0.31048(9)	0.0311(8)	0.0337(8)	0.064(1)	–0.0017(6)	0.0038(7)	–0.0074(7)

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