

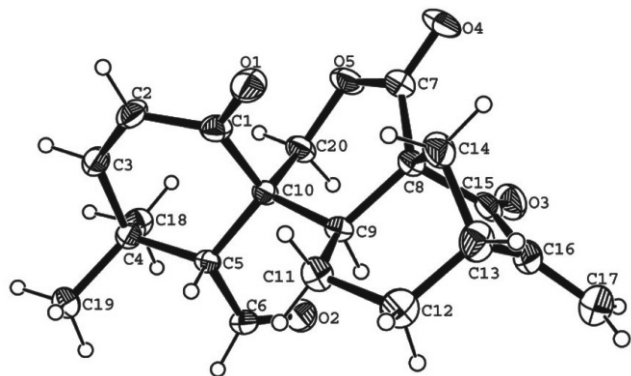
Crystal structure of 2,16-dien-1,15-dion-6,7-seco-7,20-olide-ent-kaur-6-al, C₂₀H₂₂O₅

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Abstract

C₂₀H₂₂O₅, orthorhombic, *P*2₁2₁2₁ (no. 19), *a* = 7.770(2) Å, *b* = 13.761(3) Å, *c* = 16.008(4) Å, *V* = 1711.7 Å³, *Z* = 4, *R*_{gt}(*F*) = 0.041, *wR*_{ref}(*F*²) = 0.091, *T* = 293 K.

Source of material

The compound ericalyxin B has been isolated from the dried and powdered leaves of *Isodon ericalyx*. Eriocalyxin B (100 mg) was dissolved in 25 ml methanol and mixed with 10 ml 80 g/L sodium periodate and set aside for two days. The product was purified by silica gel column chromatography with petroleum ether/ethyl acetate (volume ratio 8:1). Colorless crystals of the title compound were obtained from ethyl acetate solution [1].

Experimental details

All of the non-hydrogen atoms were refined anisotropically. The hydrogen atoms were assigned with common isotropic displacement factors *U*_{iso}(H) = 1.2 *U*_{eq}(C) and 1.5 *U*_{eq}(O), respectively, and included in the final refinement with *d*(C—H) = 0.93–0.98 Å. The molecule contains no heavy atoms, therefore a meaningful Flack parameter could not be determined with Mo *K*_α radiation (*x* = 0.0(12)) and the absolute structure could not be made sure.

Discussion

The bond distances and bond angles in the title crystal structure are all in normal range. Two olefinic bonds were clearly identified by the distances *d*(C2—C3) = 1.321(3) Å and

d(C16—C17) = 1.324(3) Å. Four ketonic bonds were confirmed by the distances *d*(C1—O1) = 1.221(2) Å, *d*(C6—O2) = 1.188(2) Å, *d*(C7—O3) = 1.210(3) Å and *d*(C15—O4) = 1.211(3) Å. The intermolecular connection was achieved through hydrogen bonding.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.24 × 0.25 × 0.29 mm
Wavelength:	Mo <i>K</i> _α radiation (0.71073 Å)
<i>μ</i> :	0.95 cm ^{−1}
Diffractometer, scan mode:	Bruker Smart APEX II, <i>φ/ω</i>
2 θ _{max} :	50°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	8684, 2986
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ (<i>I</i> _{obs}), 2568
<i>N</i> (<i>param</i>) _{refined} :	226
Program:	SHELXL-97 [2]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(2)	4a	0.6463	0.3803	0.5747	0.054
H(3)	4a	0.8355	0.4257	0.6676	0.049
H(5)	4a	0.9877	0.1822	0.6261	0.035
H(9)	4a	0.7993	0.0103	0.6472	0.037
H(20A)	4a	0.6402	0.0947	0.7565	0.050
H(20B)	4a	0.6038	0.2067	0.7507	0.050
H(13)	4a	0.6433	−0.1376	0.4373	0.064
H(14A)	4a	0.4160	−0.0434	0.4894	0.060
H(14B)	4a	0.5541	0.0355	0.4634	0.060
H(12A)	4a	0.9267	−0.0957	0.5147	0.062
H(12B)	4a	0.8840	−0.0478	0.4283	0.062
H(11A)	4a	0.8086	0.0950	0.4870	0.051
H(11B)	4a	0.9689	0.0637	0.5401	0.051
H(18A)	4a	0.9735	0.2654	0.8334	0.069
H(18B)	4a	0.7821	0.2923	0.8130	0.069
H(18C)	4a	0.9227	0.3739	0.8167	0.069
H(19A)	4a	1.2025	0.2835	0.7202	0.070
H(19B)	4a	1.1411	0.3910	0.7070	0.070
H(19C)	4a	1.1482	0.3192	0.6310	0.070
H(6)	4a	1.1197	0.1268	0.7438	0.046
H(17A)	4a	0.6425	−0.2815	0.6290	0.086
H(17B)	4a	0.7136	−0.2911	0.5348	0.086

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
C(1)	4a	0.6614(3)	0.2312(2)	0.5873(1)	0.035(1)	0.043(1)	0.040(1)	0.005(1)	−0.007(1)	0.000(1)
C(2)	4a	0.7047(3)	0.3322(2)	0.6041(1)	0.050(1)	0.033(1)	0.052(1)	0.008(1)	−0.015(1)	0.006(1)

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Table 3. Continued.

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
C(3)	4a	0.8226(3)	0.3593(2)	0.6588(1)	0.045(1)	0.031(1)	0.048(1)	0.002(1)	−0.005(1)	0.001(1)
C(4)	4a	0.9366(3)	0.2934(1)	0.7075(1)	0.033(1)	0.032(1)	0.039(1)	−0.0008(9)	−0.006(1)	0.0006(9)
C(5)	4a	0.9155(2)	0.1866(1)	0.6761(1)	0.024(1)	0.032(1)	0.031(1)	0.0019(9)	0.0001(8)	0.0010(8)
C(10)	4a	0.7324(3)	0.1546(1)	0.6471(1)	0.025(1)	0.032(1)	0.033(1)	−0.0003(9)	0.0001(8)	0.0022(8)
C(9)	4a	0.7420(3)	0.0525(1)	0.6064(1)	0.026(1)	0.036(1)	0.031(1)	0.0017(9)	−0.0017(9)	−0.0005(8)
C(8)	4a	0.5573(3)	0.0072(1)	0.5919(1)	0.028(1)	0.036(1)	0.044(1)	−0.003(1)	−0.008(1)	0.0016(9)
C(7)	4a	0.4074(3)	0.0612(2)	0.6286(2)	0.031(1)	0.047(1)	0.068(2)	−0.002(1)	−0.005(1)	0.003(1)
O(5)	4a	0.4326(2)	0.1265(1)	0.6894(1)	0.0241(8)	0.060(1)	0.073(1)	0.0021(8)	0.0056(8)	−0.0122(9)
C(20)	4a	0.6042(3)	0.1464(2)	0.7192(1)	0.028(1)	0.049(1)	0.048(1)	−0.001(1)	0.004(1)	−0.005(1)
C(15)	4a	0.5661(3)	−0.0951(2)	0.6308(2)	0.028(1)	0.046(1)	0.056(1)	−0.011(1)	−0.010(1)	0.003(1)
C(16)	4a	0.6388(3)	−0.1597(2)	0.5654(2)	0.046(1)	0.039(1)	0.067(2)	−0.005(1)	−0.015(1)	−0.005(1)
C(13)	4a	0.6658(3)	−0.0997(2)	0.4879(1)	0.066(2)	0.049(1)	0.046(1)	−0.004(1)	−0.012(1)	−0.012(1)
C(14)	4a	0.5316(3)	−0.0195(2)	0.4997(1)	0.049(1)	0.049(1)	0.052(1)	−0.009(1)	−0.019(1)	0.002(1)
C(12)	4a	0.8465(3)	−0.0534(2)	0.4859(1)	0.059(2)	0.057(1)	0.040(1)	0.001(1)	0.006(1)	−0.014(1)
C(11)	4a	0.8508(3)	0.0473(2)	0.5267(1)	0.042(1)	0.046(1)	0.039(1)	−0.005(1)	0.003(1)	−0.004(1)
C(18)	4a	0.9003(3)	0.3076(2)	0.8015(1)	0.052(1)	0.043(1)	0.044(1)	0.005(1)	−0.006(1)	−0.010(1)
C(19)	4a	1.1251(3)	0.3248(2)	0.6897(2)	0.038(1)	0.042(1)	0.060(2)	−0.008(1)	−0.007(1)	−0.002(1)
C(6)	4a	1.0007(3)	0.1213(2)	0.7393(1)	0.032(1)	0.037(1)	0.048(1)	0.005(1)	−0.004(1)	−0.001(1)
C(17)	4a	0.6679(4)	−0.2534(2)	0.5776(2)	0.072(2)	0.047(2)	0.096(2)	−0.002(2)	−0.009(2)	−0.005(1)
O(4)	4a	0.5219(2)	−0.1169(1)	0.7009(1)	0.052(1)	0.058(1)	0.063(1)	−0.0130(9)	0.0010(9)	0.0161(9)
O(3)	4a	0.2613(2)	0.0447(1)	0.6067(1)	0.0249(9)	0.077(1)	0.108(2)	−0.0022(9)	−0.0150(9)	−0.011(1)
O(1)	4a	0.5645(2)	0.2089(1)	0.5305(1)	0.061(1)	0.0472(9)	0.064(1)	0.0027(9)	−0.033(1)	−0.0004(8)
O(2)	4a	0.9338(2)	0.0637(1)	0.7840(1)	0.055(1)	0.053(1)	0.057(1)	−0.0015(9)	−0.0091(9)	0.0200(8)

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