Crystal structure of stigmasta-4,25-diene-3 β ,6 β -diol, C₂₉H₄₈O₂

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Abstract

C₂₉H₄₈O₂, orthorhombic, *P*2₁2₁2₁ (no. 19), *a* = 8.162(1) Å, *b* = 10.959(2) Å, *c* = 29.056(4) Å, *V* = 2598.9 Å³, *Z* = 4, *R*_{gt}(*F*) = 0.108, *wR*_{ref}(*F*²) = 0.234, *T* = 298 K.

Source of material

Air-dried green alga material (5 kg) was collected along the coast of Fuding of Fujian province, P. R. China, in May 2005. Its extract of 440 g in 95 % EtOH was suspended in water and partitioned successively with petroleum ether, chloroform and *n*-butanol. An aliquot (50 g) of the petroleum ether extract fraction was subjected to silica-gel column chromatography (160–200 mesh, 0.5 kg) and eluted with CH₂Cl₂/MeOH in increasing polarity. The fractions eluted by 10 % methanol in dichloromethane was further eluted with petroleum/EtOAc in increasing polarity. The fraction eluted by petroleum ether/ethyl acetate (3:7, *v/v*) was crystallized at room temperature from chloroform/methanol, which yielded the crystals of the title compound. ¹³C NMR data are available in the CIF.

Experimental details

An exact explanation for the large *R* values could not be found. Those are probably caused by the imperfect crystals. Co-crystallizing solvents were not found.

Discussion

Marine green alga genus *Codium* have been found to be an important source of clerosterol [1,2] and its oxygenated derivatives [3]. Several reports have revealed that some oxygenated sterols possess interesting biological activities [4,5]. Our present investigation of *Codium divaricatum* Holmes led to the isolation of the title compound from this genus for the first time. The structure was elucidated by spectroscopic analysis, including two-dimensional NMR, and confirmed by single-crystal X-ray diffraction analysis.

The molecule contains three six-membered rings (A, atoms C1 – C5/C10; B, C5 – C10; and C, C8/C9/C11 – C14), and a five-membered ring (D, C13 – C17). Ring A adopts a badly distorted chair conformation, while rings B and C adopt chair conformations, respectively. Ring D exhibits an envelope conformation. In the crystal structure, the molecules are interlinked by two hydrogen bonds forming a two-dimensional network parallel to the (001) plane.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.16 × 0.29 × 0.32 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.66 cm ⁻¹
Diffractometer, scan mode:	Bruker SMART APEX CCD, ϕ/ω
2 θ _{max} :	50.04°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	13741, 4589
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ (<i>I</i> _{obs}), 3919
<i>N</i> (<i>param</i>) _{refined} :	287
Program:	SHELX-97 [6]

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(1)	4a	−0.1497	0.2030	0.0320	0.086
H(2)	4a	0.5665	0.2413	0.0245	0.089
H(26A)	4a	1.1840	0.4436	0.4474	0.182
H(26B)	4a	1.2249	0.4128	0.5004	0.182
H(1A)	4a	0.1466	0.4332	0.1391	0.066
H(1B)	4a	0.1169	0.2958	0.1516	0.066
H(2A)	4a	−0.0726	0.3637	0.0941	0.072
H(2B)	4a	0.0748	0.3896	0.0607	0.072
H(3)	4a	−0.0211	0.1597	0.0938	0.056
H(4)	4a	0.2198	0.0942	0.0460	0.051
H(6)	4a	0.4852	0.0720	0.0593	0.055
H(7A)	4a	0.6976	0.0942	0.1148	0.053
H(7B)	4a	0.5288	0.0575	0.1365	0.053

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Table 2. Continued.

Atom	Site	x	y	z	U _{iso}
H(8)	4a	0.6647	0.2947	0.1381	0.045
H(9)	4a	0.3597	0.2220	0.1748	0.047
H(11A)	4a	0.4863	0.4595	0.1819	0.073
H(11B)	4a	0.3182	0.4162	0.2020	0.073
H(12A)	4a	0.4482	0.2989	0.2571	0.066
H(12B)	4a	0.5188	0.4319	0.2600	0.066
H(17)	4a	0.6877	0.1961	0.2897	0.051
H(16A)	4a	0.8890	0.0780	0.2679	0.063
H(16B)	4a	0.9999	0.1873	0.2516	0.063
H(15A)	4a	0.9091	0.1699	0.1798	0.055
H(15B)	4a	0.8194	0.0486	0.1949	0.055
H(14)	4a	0.5890	0.1453	0.2142	0.043
H(19A)	4a	0.3874	0.5038	0.1082	0.086
H(19B)	4a	0.3655	0.4389	0.0605	0.086
H(19C)	4a	0.5267	0.4196	0.0889	0.086
H(18A)	4a	0.8054	0.4751	0.2342	0.081
H(18B)	4a	0.7562	0.4354	0.1842	0.081
H(18C)	4a	0.9065	0.3742	0.2086	0.081

Table 2. Continued.

Atom	Site	x	y	z	U _{iso}
H(20)	4a	0.9488	0.3624	0.2972	0.064
H(22A)	4a	0.8322	0.1971	0.3633	0.082
H(22B)	4a	0.9940	0.1803	0.3352	0.082
H(23A)	4a	1.0976	0.3571	0.3730	0.116
H(23B)	4a	0.9423	0.3541	0.4044	0.116
H(24)	4a	1.0251	0.1598	0.4285	0.105
H(27A)	4a	1.1277	0.0970	0.4896	0.356
H(27B)	4a	1.1456	0.1948	0.5286	0.356
H(27C)	4a	1.3004	0.1515	0.5011	0.356
H(21A)	4a	0.6487	0.3857	0.3447	0.119
H(21B)	4a	0.7149	0.4778	0.3078	0.119
H(21C)	4a	0.8040	0.4649	0.3554	0.119
H(28A)	4a	1.1932	0.1109	0.3715	0.189
H(28B)	4a	1.2822	0.0950	0.4190	0.189
H(29A)	4a	1.3397	0.2847	0.3545	0.388
H(29B)	4a	1.4718	0.1960	0.3748	0.388
H(29C)	4a	1.3981	0.3000	0.4055	0.388

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

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
O(1)	4a	-0.0513(4)	0.1921(4)	0.0279(1)	0.021(2)	0.103(3)	0.048(2)	-0.006(2)	-0.003(1)	0.003(2)
O(2)	4a	0.6109(4)	0.2233(4)	0.0489(1)	0.023(2)	0.109(3)	0.046(2)	0.004(2)	0.004(1)	0.015(2)
C(26)	4a	1.192(2)	0.3874(9)	0.4713(3)	0.25(1)	0.114(8)	0.092(6)	-0.002(9)	-0.030(8)	-0.033(6)
C(1)	4a	0.1529(6)	0.3505(5)	0.1274(2)	0.033(3)	0.062(3)	0.070(3)	0.009(2)	0.007(2)	-0.007(3)
C(2)	4a	0.0370(6)	0.3378(6)	0.0856(2)	0.024(2)	0.089(4)	0.069(3)	0.018(3)	-0.001(2)	0.009(3)
C(3)	4a	0.0334(5)	0.2086(5)	0.0701(2)	0.027(2)	0.070(3)	0.044(3)	-0.009(2)	0.006(2)	0.011(3)
C(4)	4a	0.2053(5)	0.1625(5)	0.0645(2)	0.031(2)	0.056(3)	0.040(3)	-0.006(2)	0.006(2)	0.002(2)
C(5)	4a	0.3383(5)	0.2100(4)	0.0834(2)	0.021(2)	0.052(3)	0.035(2)	-0.005(2)	0.002(2)	0.011(2)
C(6)	4a	0.5030(5)	0.1492(5)	0.0756(2)	0.028(2)	0.071(3)	0.038(3)	0.005(2)	-0.003(2)	-0.015(2)
C(7)	4a	0.5871(6)	0.1226(4)	0.1209(2)	0.026(2)	0.046(3)	0.061(3)	0.003(2)	0.006(2)	-0.010(2)
C(8)	4a	0.5954(5)	0.2325(4)	0.1526(2)	0.032(2)	0.037(2)	0.046(3)	-0.001(2)	0.007(2)	0.003(2)
C(9)	4a	0.4245(5)	0.2870(4)	0.1605(2)	0.030(2)	0.037(2)	0.051(3)	-0.008(2)	0.004(2)	-0.011(2)
C(10)	4a	0.3326(5)	0.3210(4)	0.1150(2)	0.025(2)	0.046(3)	0.049(3)	-0.001(2)	0.002(2)	0.006(2)
C(11)	4a	0.4297(7)	0.3906(5)	0.1956(2)	0.045(3)	0.060(3)	0.077(4)	0.022(3)	-0.011(3)	-0.009(3)
C(12)	4a	0.5129(7)	0.3594(5)	0.2409(2)	0.054(3)	0.051(3)	0.058(3)	0.018(3)	-0.005(3)	-0.020(3)
C(13)	4a	0.6861(5)	0.3095(4)	0.2331(2)	0.040(2)	0.037(2)	0.037(2)	-0.002(2)	-0.002(2)	-0.008(2)
C(17)	4a	0.7719(6)	0.2450(4)	0.2742(2)	0.049(3)	0.037(3)	0.043(3)	-0.005(2)	0.000(2)	0.003(2)
C(16)	4a	0.8893(7)	0.1549(5)	0.2514(2)	0.050(3)	0.050(3)	0.058(3)	0.009(3)	-0.006(3)	-0.001(3)
C(15)	4a	0.8321(6)	0.1348(5)	0.2015(2)	0.048(3)	0.048(3)	0.043(3)	0.006(2)	-0.006(2)	-0.005(2)
C(14)	4a	0.6680(5)	0.2006(4)	0.1997(1)	0.031(2)	0.035(2)	0.041(2)	-0.001(2)	0.008(2)	0.009(2)
C(19)	4a	0.4103(7)	0.4311(5)	0.0909(2)	0.045(3)	0.062(3)	0.066(4)	-0.001(3)	-0.009(3)	0.011(3)
C(18)	4a	0.7991(7)	0.4077(5)	0.2132(2)	0.057(3)	0.050(3)	0.055(3)	-0.005(3)	-0.007(3)	-0.002(2)
C(20)	4a	0.8552(7)	0.3216(5)	0.3117(2)	0.060(3)	0.050(3)	0.050(3)	0.003(3)	-0.004(3)	-0.003(2)
C(22)	4a	0.9230(8)	0.2406(5)	0.3494(2)	0.093(4)	0.059(3)	0.052(3)	-0.007(3)	-0.023(3)	-0.004(3)
C(23)	4a	1.018(1)	0.3034(7)	0.3871(2)	0.123(6)	0.083(5)	0.084(5)	0.015(5)	-0.027(5)	0.000(4)
C(24)	4a	1.1060(9)	0.2223(7)	0.4204(2)	0.094(5)	0.099(5)	0.071(4)	0.012(5)	-0.024(4)	0.009(4)
C(25)	4a	1.157(1)	0.271(1)	0.4638(3)	0.102(6)	0.153(9)	0.087(6)	0.001(6)	-0.021(5)	0.012(6)
C(27)	4a	1.185(2)	0.169(1)	0.4991(4)	0.19(1)	0.33(2)	0.19(1)	-0.04(1)	-0.07(1)	0.18(1)
C(21)	4a	0.7454(9)	0.4219(6)	0.3317(2)	0.100(5)	0.082(4)	0.056(4)	0.011(4)	-0.020(4)	-0.027(3)
C(28)	4a	1.237(1)	0.155(1)	0.3978(4)	0.144(9)	0.19(1)	0.143(9)	0.072(9)	-0.048(8)	-0.052(9)
C(29)	4a	1.375(2)	0.242(1)	0.3816(6)	0.15(1)	0.19(1)	0.44(3)	0.04(1)	0.16(1)	0.12(2)

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