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Crystal structure of 3β,6α,23α-trihydroxy-5α,25*R*-spirostane — methanol (1:1), $C_{27}H_{45}O_5$ CH_3OH, isolated from *Agave sisalana*

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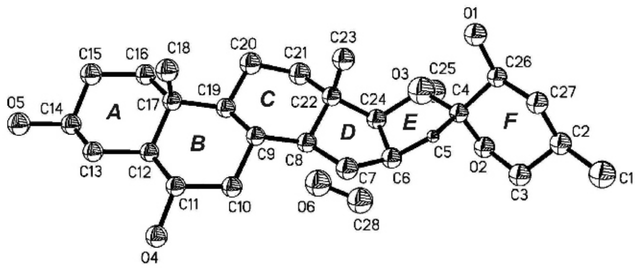

Abstract

$C_{28}H_{49}O_6$, orthorhombic, $P2_12_12_1$ (no. 19), $a = 8.689(2)$ Å, $b = 13.516(3)$ Å, $c = 22.545(5)$ Å, $V = 2647.6$ Å^3, $Z = 4$, $R_{gt}(F) = 0.063$, $wR_{ref}(F^2) = 0.195$, $T = 296$ K.

Source of material

Generally, for the separation of the steroid sapogenins and saponins, paper chromatography, silica gel thin layer chromatography and silica gel column chromatography can be used [1-4]. This separation experiment has made use of a method of silica gel column chromatography, and the main experimental steps are as follows. The ethanol extract of *Agave sisalana* (200.0 g) has been added to a methanol-water solution (1:1, 8 L); Then the upper solution has been separated from the precipitate and concentrated to obtain an extract (108.3 g); The extract has been dissolved in a methanol-water solution (1:1, 4 L), and ethyl acetate (3 L) was added for a liquid-liquid extraction; The upper ethyl acetate layer has been concentrated to obtain a pale yellow extract (23.3 g); Then the ethyl acetate extract was separated by silica gel column chromatography, and the eluting solutions are as follows: petroleum ether - ethyl acetate (2:1), petroleum ether - ethyl acetate (1:1), petroleum ether - ethyl acetate (1:3), ethyl acetate, methanol - ethyl acetate (1:9), methanol - ethyl acetate (1:4), methanol - ethyl acetate (1:1), and methanol. Colorless block-shaped crystals of hongguanggenin (3β,6α,23α-trihydroxy-5α,25*R*-spirostane) were found in the methanol - ethyl acetate (1:9) portion.

Experimental details

All H atoms were positioned geometrically (d(C—H) = 0.82-0.98 Å) and refined as riding with $U_{iso} = 1.2\ U_{eq}$. The Flack parameter is –0.7(17), based on 1997 Friedel pairs.

Discussion

Agave sisalana is an agave, *Agavaceae*, perennial herb. The extract of *Agave sisalana* can be used to treat, e. g., tuberculosis, subcutaneous tissue, hemafecia, dysentery, skin ulcer, hemorrhoids [5]. The steroid sapogenins and saponins from *Agave sisalana* have proved to be cytotoxic, antifungal, antibacterial and anti-inflammatory [6,7]. Research has shown that the activity of the steroidal saponins can be associated with their aglycone moieties and the number and structure of monosaccharide units in their sugar chains. Hongguanggenin (3β,6α,23α-trihydroxy-5α,25*R*-spirostane) is a steroidal sapogenin and isolated from *Agave sisalana* [8]. In order to find out the structure activity relationship of hongguanggenin and its saponin, and increase the utilization of *Agave sisalana*, the data of crystal structure of hongguanggenin are useful.

In the molecule of the title compound, all bond lengths and angles are within normal ranges. Six carbon atoms in ring *A* (C12-C17), ring *B* (C9-C12/C17/C19), ring *C* (C8/C9/C19-C22), and ring *F* (C2/C3/O2/C4/C26/C27) are all in chair conformation. Five carbon atoms in ring *D* (C6-C8/C22/C24) have an envelope form with C8 as the out-of-plane atom. Five carbon atoms in ring *E* (C4-C6/C24/O3) also has an envelope form with C5 as the out-of-plane atom. In the crystal, adjacent molecules are linked by intermolecular C–H   O hydrogen bonding to form a supramolecular network. The intermolecular O–H   O hydrogen-bonding interactions are formed by atoms O1, O4, O5, and O6 belonging to hydroxy groups, resulting in a 3D supramolecular structures.

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Table 1. Data collection and handling.

Crystal:	colorless block, size 0.28 0.31 0.38 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.83 cm ^{1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	14344, 4653
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4340
$N(param)_{refined}$:	309
Programs:	SHELXS-97, SHELXL-97, SHELXTL [9]

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

Atom	Site	x	y	z	U_{iso}
H(1)	4*a*	0.6170	0.0583	0.0834	0.062
H(4)	4*a*	0.1224	0.3451	0.4013	0.058
H(5)	4*a*	0.1341	0.1074	0.6203	0.069
H(6)	4*a*	0.8890	0.3404	0.3419	0.119
H(1B)	4*a*	0.7455	0.2563	0.0886	0.092
H(1C)	4*a*	0.5871	0.2096	0.0706	0.092
H(1D)	4*a*	0.7082	0.1464	0.1056	0.092
H(2A)	4*a*	0.8744	0.1482	0.0239	0.049
H(3A)	4*a*	0.8081	0.2988	0.0164	0.053
H(3B)	4*a*	0.6472	0.2575	0.0359	0.053
H(5A)	4*a*	0.5324	0.0811	0.1266	0.016
H(5B)	4*a*	0.5444	0.1775	0.0875	0.016
H(6A)	4*a*	0.6137	0.2713	0.1654	0.044
H(7A)	4*a*	0.3632	0.1484	0.1891	0.048
H(7B)	4*a*	0.3784	0.2616	0.2059	0.048
H(8A)	4*a*	0.5295	0.2278	0.2860	0.038
H(9A)	4*a*	0.2977	0.0833	0.2992	0.037

Table 2. Continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(10A)	4*a*	0.3089	0.2849	0.3340	0.041
H(10B)	4*a*	0.1885	0.2385	0.2905	0.041
H(11A)	4*a*	0.0577	0.1634	0.3660	0.038
H(12A)	4*a*	0.2893	0.2261	0.4454	0.037
H(13A)	4*a*	0.0494	0.2220	0.4928	0.043
H(13B)	4*a*	0.0327	0.1092	0.4774	0.043
H(14A)	4*a*	0.2651	0.1891	0.5515	0.045
H(15A)	4*a*	0.2190	0.0140	0.5262	0.051
H(15B)	4*a*	0.3480	0.0277	0.5680	0.051
H(16A)	4*a*	0.4489	0.0109	0.4737	0.047
H(16B)	4*a*	0.4730	0.1012	0.4887	0.047
H(18A)	4*a*	0.1404	0.0230	0.4308	0.064
H(18B)	4*a*	0.2787	0.0637	0.3934	0.064
H(18C)	4*a*	0.1614	0.0102	0.3646	0.064
H(19A)	4*a*	0.4851	0.1694	0.3881	0.039
H(20A)	4*a*	0.6170	0.0223	0.4015	0.055

Table 2. Continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(20B)	4*a*	0.5219	0.0282	0.3506	0.055
H(21A)	4*a*	0.7337	0.1293	0.3331	0.055
H(21B)	4*a*	0.7617	0.0197	0.3128	0.055
H(23A)	4*a*	0.6318	0.0493	0.2256	0.063
H(23B)	4*a*	0.5006	0.0100	0.1937	0.063
H(23C)	4*a*	0.4730	0.0335	0.2573	0.063
H(24A)	4*a*	0.7632	0.2042	0.2365	0.041
H(25A)	4*a*	1.0478	0.1026	0.1497	0.079
H(25B)	4*a*	1.0196	0.1229	0.2173	0.079
H(25C)	4*a*	0.9894	0.2067	0.1706	0.079
H(26A)	4*a*	0.8724	0.0309	0.0576	0.041
H(27A)	4*a*	0.5820	0.0758	0.0104	0.047
H(27B)	4*a*	0.7087	0.0136	0.0225	0.047
H(28A)	4*a*	0.8442	0.4639	0.2647	0.110
H(28B)	4*a*	0.8353	0.3601	0.2339	0.110
H(28C)	4*a*	0.9854	0.3919	0.2674	0.110

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}
O(1)	4*a*	0.7107(3)	0.0543(2)	0.0870(1)	0.041(1)	0.032(1)	0.051(1)	0.001(1)	0.000(1)	0.005(1)
O(2)	4*a*	0.8195(3)	0.2082(2)	0.0856(1)	0.045(1)	0.032(1)	0.041(1)	0.005(1)	0.003(1)	0.004(1)
O(3)	4*a*	0.8178(4)	0.1019(3)	0.1713(2)	0.059(2)	0.075(2)	0.068(2)	0.002(2)	0.003(2)	0.005(2)
O(4)	4*a*	0.0593(3)	0.3045(2)	0.3896(1)	0.034(1)	0.039(1)	0.043(1)	0.011(1)	0.001(1)	0.001(1)
O(5)	4*a*	0.0874(3)	0.1228(2)	0.5901(1)	0.040(1)	0.065(2)	0.034(1)	0.004(1)	0.008(1)	0.005(1)
O(6)	4*a*	0.8173(4)	0.3546(3)	0.3198(1)	0.060(2)	0.127(3)	0.053(2)	0.024(2)	0.011(2)	0.004(2)
C(1)	4*a*	0.6949(6)	0.1968(4)	0.0760(2)	0.074(3)	0.071(3)	0.040(2)	0.016(2)	0.003(2)	0.014(2)
C(2)	4*a*	0.7651(4)	0.1615(3)	0.0169(2)	0.043(2)	0.047(2)	0.032(2)	0.008(2)	0.004(1)	0.006(1)
C(3)	4*a*	0.7544(5)	0.2401(3)	0.0299(2)	0.051(2)	0.039(2)	0.041(2)	0.006(2)	0.007(2)	0.009(2)
C(4)	4*a*	0.7462(3)	0.1224(2)	0.1104(1)	0.029(2)	0.030(2)	0.034(2)	0.001(1)	0.006(1)	0.002(1)
C(5)	4*a*	0.5891(3)	0.1422(2)	0.12073(9)	0.014(1)	0.019(1)	0.006(1)	0.0052(9)	0.0029(8)	0.0003(9)
C(6)	4*a*	0.5852(4)	0.2027(2)	0.1742(1)	0.045(2)	0.034(2)	0.033(2)	0.005(2)	0.002(2)	0.000(1)
C(7)	4*a*	0.4301(4)	0.1980(3)	0.2064(1)	0.046(2)	0.046(2)	0.028(2)	0.016(2)	0.003(2)	0.004(1)
C(8)	4*a*	0.4787(4)	0.1692(2)	0.2695(1)	0.034(2)	0.032(2)	0.029(2)	0.005(1)	0.001(1)	0.000(1)
C(9)	4*a*	0.3570(4)	0.1393(2)	0.3147(1)	0.034(2)	0.032(2)	0.027(1)	0.004(1)	0.001(1)	0.003(1)
C(10)	4*a*	0.2476(4)	0.2263(2)	0.3263(1)	0.038(2)	0.036(2)	0.029(2)	0.007(1)	0.001(1)	0.006(1)
C(11)	4*a*	0.1354(4)	0.2121(2)	0.3778(1)	0.029(2)	0.032(2)	0.033(2)	0.003(1)	0.002(1)	0.001(1)
C(12)	4*a*	0.2165(4)	0.1743(2)	0.4337(1)	0.029(2)	0.033(2)	0.030(2)	0.001(1)	0.000(1)	0.002(1)
C(13)	4*a*	0.1061(4)	0.1611(3)	0.4864(1)	0.034(2)	0.040(2)	0.033(2)	0.004(1)	0.005(1)	0.002(1)
C(14)	4*a*	0.1948(4)	0.1347(3)	0.5420(1)	0.034(2)	0.045(2)	0.032(2)	0.002(2)	0.006(1)	0.001(1)
C(15)	4*a*	0.2881(4)	0.0414(3)	0.5326(2)	0.047(2)	0.048(2)	0.033(2)	0.008(2)	0.000(2)	0.013(2)
C(16)	4*a*	0.3959(4)	0.0515(3)	0.4796(1)	0.042(2)	0.047(2)	0.030(2)	0.013(2)	0.003(1)	0.006(1)
C(17)	4*a*	0.3142(4)	0.0806(2)	0.4211(1)	0.037(2)	0.028(1)	0.029(2)	0.004(1)	0.001(1)	0.004(1)
C(18)	4*a*	0.2142(5)	0.0073(2)	0.4005(2)	0.058(2)	0.033(2)	0.037(2)	0.002(2)	0.005(2)	0.000(1)
C(19)	4*a*	0.4352(4)	0.1093(2)	0.3733(1)	0.033(2)	0.035(2)	0.029(2)	0.005(1)	0.001(1)	0.002(1)
C(20)	4*a*	0.5655(5)	0.0341(3)	0.3639(2)	0.048(2)	0.055(2)	0.033(2)	0.022(2)	0.004(2)	0.012(2)
C(21)	4*a*	0.6835(4)	0.0701(3)	0.3183(2)	0.037(2)	0.063(2)	0.037(2)	0.018(2)	0.002(2)	0.004(2)
C(22)	4*a*	0.6080(4)	0.0927(2)	0.2590(1)	0.034(2)	0.034(2)	0.030(2)	0.003(1)	0.002(1)	0.002(1)
C(23)	4*a*	0.5476(4)	0.0042(3)	0.2313(2)	0.048(2)	0.031(2)	0.048(2)	0.000(2)	0.011(2)	0.003(2)
C(24)	4*a*	0.7071(4)	0.1528(2)	0.2148(1)	0.036(2)	0.036(2)	0.032(2)	0.003(1)	0.002(1)	0.001(1)
C(25)	4*a*	0.9841(4)	0.1368(3)	0.1779(2)	0.039(2)	0.074(3)	0.045(2)	0.001(2)	0.001(2)	0.001(2)
C(26)	4*a*	0.7618(4)	0.0384(2)	0.0648(2)	0.031(2)	0.033(2)	0.038(2)	0.000(1)	0.006(1)	0.000(1)
C(27)	4*a*	0.6921(4)	0.0665(3)	0.0059(2)	0.040(2)	0.043(2)	0.034(2)	0.002(2)	0.004(2)	0.003(1)
C(28)	4*a*	0.8751(7)	0.3959(4)	0.2673(2)	0.089(4)	0.059(3)	0.072(3)	0.002(3)	0.015(3)	0.005(2)

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