Crystal structure of 7-deacetylazorellanol, $C_{20}H_{34}O_2$, a diterpenoid from *Azorella Compacta*

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Abstract

$C_{20}H_{34}O_2$, orthorhombic, $C222$ (No. 21), $a = 15.885(3)$ Å, $b = 17.168(3)$ Å, $c = 13.920(3)$ Å, $V = 3796.2$ Å³, $Z = 8$, $R_{\text{gt}}(F) = 0.045$, $wR_{\text{ref}}(F^2) = 0.071$, $T = 293$ K.

Source of material

The new diterpenoid azorellanol has been isolated from the aerial parts of *Azorella Compacta* Phil. (Umbelliferae). White crystals were obtained from petroleum ether-EtOAc solution [1]. *Azorella Compacta*, popularly known as "llareta", is a yellow-green, compact resinous cushion shrub, which grows in the high Andes of Southern Peru, Bolivia, northeastern Chile and northwestern Argentina [2]. Bitter taste infusions of the whole plant are used in folk medicine, principally in the treatment of diabetes as well as for asthma, colds, bronchitis, kidney and womb complaints [2].

Discussion

In a previous communication [1] we have reported the absolute structure of 7-p-bromobenzoyl-7-deacetylazorellanol determined by combination of spectroscopic and single crystal X-ray diffraction analysis.

7-Deacetylazorellanol has the molecular formula $C_{20}H_{34}O_2$ and possesses a tricyclic carbon skeleton, with a six-member ring (A) with a methylene bridge between C(9) and C(12). It also has a six membered ring (B) fused to a five ring (C), the methyl substituents at C(5) and C(8) are axial on one side of molecule, and the hydroxyl group at C(13) is axially disposed on the other side. The isopropyl substituent and the hydroxyl group at C(7) are equatorially bonded to the molecule. The ring A is in a distorted half-chair conformation with C(9) deviate for the plane defined by C(8), C(12), C(13) and C(15) -0.35 Å, as compared to 0.49 Å and 0.70 Å for C(14) and C(11). This flattening of the ring A is

essentially determined by the presence of C(11) bonded to C(9) and C(12) to form a cyclopropane ring, that tends to constrain half of the ring to a planar arrangement. The cyclohexane ring B adopts a twist boat conformation and the five member ring C is an envelope. The torsional angles of ring A and B are about the common C(8)—C(9) bond are 126.2° and 131.5° , whereas those of ring B and C about C(5) and C(10) are 168.8° and 179.6° .

Table 1. Data collection and handling.

Crystal:	white block, size $0.30 \times 0.90 \times 0.20$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.67 cm^{-1}
Diffractometer, scan mode:	Siemens R3mV, $2\theta/\theta$
$2\theta_{\text{max}}$:	45.06°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	1457, 1457
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1417
$N(\text{param})_{\text{refined}}$:	199
Programs:	SHELXS-97 [3] SHELXL-97 [4]

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(1A)	8I	0.0349	0.5863	0.4864	0.08
H(2A)	8I	0.0400	0.5000	0.1251	0.08
H(1B)	8I	0.3895	0.5089	0.3646	0.08
H(1C)	8I	0.3367	0.4527	0.4296	0.08
H(2B)	8I	0.3927	0.3471	0.3615	0.08
H(2C)	8I	0.4497	0.4025	0.3023	0.08
H(3A)	8I	0.3038	0.3338	0.2347	0.08
H(4A)	8I	0.4533	0.3716	0.1418	0.08
H(6A)	8I	0.2150	0.3951	0.1140	0.08
H(6B)	8I	0.2407	0.4751	0.0704	0.08
H(7A)	8I	0.1274	0.4503	0.2265	0.08
H(10A)	8I	0.2319	0.4423	0.3202	0.08
H(11A)	8I	0.2625	0.6790	0.3051	0.08
H(11B)	8I	0.3358	0.6216	0.3437	0.08
H(12A)	8I	0.2371	0.5612	0.4522	0.08
H(14A)	8I	0.1107	0.6938	0.3046	0.08
H(14B)	8I	0.0268	0.6771	0.3591	0.08
H(15A)	8I	0.0638	0.5461	0.3126	0.08
H(15B)	8I	0.0291	0.5932	0.2248	0.08
H(16A)	8I	0.1802	0.6817	0.5526	0.08
H(16B)	8I	0.1805	0.7388	0.4647	0.08
H(16C)	8I	0.0962	0.7212	0.5191	0.08
H(17A)	8I	0.2378	0.6152	0.1225	0.08
H(17B)	8I	0.1420	0.6226	0.0971	0.08
H(17C)	8I	0.1827	0.6794	0.1721	0.08

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Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(18A)	8f	0.4709	0.2679	0.2440 ^δ	0.08
H(18B)	8f	0.3921	0.2301	0.1950	0.08
H(18C)	8f	0.4779	0.2342	0.1396	0.08
H(19A)	8f	0.3486	0.3748	0.0235	0.08
H(19B)	8f	0.4020	0.2991	0.0082	0.08

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(19C)	8f	0.3162	0.2949	0.0636	0.08
H(20A)	8f	0.4175	0.5148	0.2048	0.08
H(20B)	8f	0.3917	0.4939	0.0992	0.08
H(20C)	8f	0.345	0.5610	0.1539	0.08

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)	8f	0.0869(3)	0.5746(3)	0.4861(5)	0.043(4)	0.068(4)	0.104(5)	0.003(4)	0.022(4)	0.006(5)
O(2)	8f	0.0871(3)	0.4892(4)	0.0995(4)	0.049(4)	0.122(5)	0.103(5)	0.031(4)	-0.025(4)	-0.038(5)
C(1)	8f	0.3522(5)	0.4649(5)	0.3647(6)	0.050(6)	0.100(9)	0.048(6)	0.019(6)	0.017(5)	0.022(6)
C(2)	8f	0.3923(5)	0.3906(5)	0.3181(7)	0.071(8)	0.092(9)	0.075(9)	0.019(7)	0.012(7)	0.014(7)
C(3)	8f	0.3457(6)	0.3726(6)	0.2212(7)	0.072(8)	0.079(8)	0.052(7)	0.008(7)	0.009(7)	0.012(7)
C(4)	8f	0.4060(5)	0.3374(5)	0.1494(8)	0.057(7)	0.085(9)	0.059(7)	0.022(6)	0.011(6)	-0.003(7)
C(5)	8f	0.3042(5)	0.4539(5)	0.1995(6)	0.038(6)	0.054(7)	0.055(7)	0.005(5)	-0.001(5)	0.010(6)
C(6)	8f	0.2264(5)	0.4487(4)	0.1289(5)	0.051(7)	0.076(7)	0.061(7)	0.005(6)	-0.016(6)	-0.007(6)
C(7)	8f	0.1469(5)	0.4842(5)	0.1765(6)	0.044(6)	0.070(8)	0.045(7)	0.003(6)	-0.017(6)	0.005(6)
C(8)	8f	0.1608(6)	0.5650(5)	0.2238(7)	0.068(7)	0.053(7)	0.044(7)	0.009(6)	-0.006(6)	0.005(6)
C(9)	8f	0.2291(5)	0.5581(5)	0.3013(6)	0.029(5)	0.050(6)	0.045(7)	0.005(6)	-0.003(5)	-0.003(6)
C(10)	8f	0.2740(5)	0.4789(5)	0.2997(6)	0.026(5)	0.045(7)	0.052(6)	0.001(5)	-0.003(5)	0.004(5)
C(11)	8f	0.2768(5)	0.6291(4)	0.3316(6)	0.053(6)	0.054(6)	0.081(8)	-0.012(6)	0.000(7)	-0.012(6)
C(12)	8f	0.2141(5)	0.5921(5)	0.4010(6)	0.051(7)	0.064(7)	0.063(7)	-0.002(6)	-0.010(6)	-0.002(7)
C(13)	8f	0.1309(6)	0.6307(5)	0.4299(7)	0.058(7)	0.045(7)	0.071(8)	-0.010(6)	0.011(7)	-0.017(6)
C(14)	8f	0.0808(5)	0.6559(5)	0.3422(7)	0.059(6)	0.075(8)	0.059(8)	0.016(6)	0.003(7)	-0.004(7)
C(15)	8f	0.0742(5)	0.5893(5)	0.2704(7)	0.037(6)	0.089(8)	0.090(8)	0.024(6)	-0.024(6)	0.000(7)
C(16)	8f	0.1488(4)	0.6996(4)	0.4980(7)	0.051(6)	0.100(8)	0.074(7)	-0.014(6)	0.034(7)	-0.025(8)
C(17)	8f	0.1830(5)	0.6272(5)	0.1475(6)	0.12(1)	0.095(8)	0.055(7)	0.020(7)	0.008(7)	0.016(8)
C(18)	8f	0.4417(5)	0.2607(5)	0.1843(6)	0.15(1)	0.16(1)	0.075(8)	0.117(9)	-0.005(8)	0.006(8)
C(19)	8f	0.3652(5)	0.3261(5)	0.0516(6)	0.090(9)	0.11(1)	0.09(1)	0.045(8)	0.000(8)	-0.004(8)
C(20)	8f	0.3713(4)	0.5109(4)	0.1606(5)	0.053(6)	0.084(7)	0.060(7)	-0.004(6)	0.004(5)	0.038(7)

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