Crystal structure of 13-hydroxy-8(9)-abieten-20-oic acid, $C_{20}H_{32}O_3$

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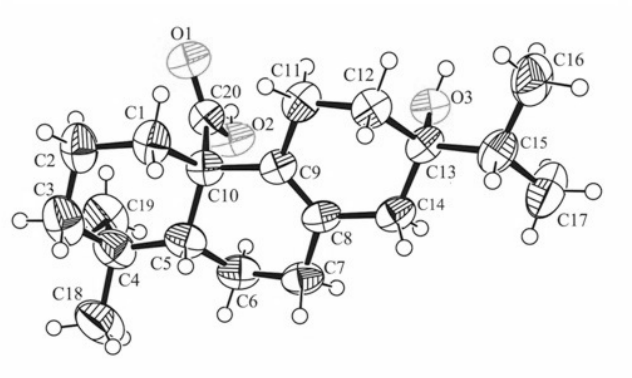

Abstract

$C_{20}H_{32}O_3$, trigonal, $P3_12_1$ (no. 152), a = 14.420(2) Å, c = 31.927(6) Å, V = 5749.8 Å^3, Z = 12, $R_{gt}(F)$ = 0.0539, $wR_{ref}(F^2)$ = 0.1223, T = 293 K.

Source of material

The air-dried and powdered leaves of Isodon flavida (Rabdosia, Labiatae family) were extracted with 90% methanol at room temperature. The carbinol extract was concentrated and treated with activated charcoal and then filtered. The filtrate was concentrated in vacuo to give a residue which was fractionalized by silica gel column chromatography eluted with a solvent system of petroleum ether (PE)/ethyl acetate (9:1) to yield the crude extract of lophanic acid. It was further isolated and purified by silica gel and crystallized from methanol affording the title compound as colourless block crystals.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.21×0.24×0.26 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.73 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ and ω
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	22635, 6736
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3597
$N(param)_{refined}$:	426
Programs:	SHELXL-97 [4]

Experimental details

All of the nonhydrogen 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-X parameter could not be determined with Mo K_α radiation (Flack-X = 2.5(14)) and the absolute structure could not be made sure.

Discussion

The title compound is a typical abietane diterpenoid and shows antimicrobial, antiulcer, and cardiovascular activities [1-2]. In 1995 the crystal structure was analysed, but no crystal data were given in the paper [3]. In order to further confirm the structure and provide complete crystal data, we repeated the work. The compound crystallizes with two molecules in the trigonal crystal system with space group $P3_12_1$ (figure). Geometric parameters of the title compound are in the usual ranges. The intermolecular connection was achieved by hydrogen bonding. The carbon-oxygen double bonds of the carboxyl groups are clearly identified by the distance of 1.214(3) Å between C20 and O1, and 1.203(3) Å between C20A and O1A, respectively. A (C1/C2/C3/C4/C5/C10), B (C5/C6/C7/C8/C9/C10) and C (C8/C9/C11/C12/C13/C14) are six-membered rings. The A ring is in the chair conformation, the B and C ring show a distorted chair conformation.

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

Atom	Site	x	y	z	U_{iso}
H(1A)	6*c*	0.1247	–0.1052	0.1378	0.0690
H(1B)	6*c*	0.0335	–0.2032	0.1138	0.0690
H(1C)	6*c*	0.5053	0.6346	0.1618	0.0740
H(1D)	6*c*	0.5906	0.7419	0.1406	0.0740
H(2A)	6*c*	0.1843	–0.2242	0.1207	0.0840
H(2B)	6*c*	0.1599	–0.2187	0.0731	0.0840
H(2D)	6*c*	0.4475	0.7593	0.1664	0.0890
H(2C)	6*c*	0.4564	0.7752	0.1177	0.0890
H(3A)	6*c*	0.3228	–0.0483	0.1198	0.0980
H(3B)	6*c*	0.3426	–0.1093	0.0835	0.0980
H(3C)	6*c*	0.2781	0.6642	0.1350	0.0980
H(3D)	6*c*	0.3056	0.5865	0.1592	0.0980
H(5)	6*c*	0.2368	0.0602	0.1016	0.040(8)
H(5A)	6*c*	0.3765	0.4886	0.1187	0.045(8)
H(6A)	6*c*	0.2937	0.1789	0.0449	0.0740
H(6B)	6*c*	0.1903	0.0966	0.0205	0.0740
H(6D)	6*c*	0.2986	0.3981	0.0565	0.0750
H(6C)	6*c*	0.3926	0.4912	0.0313	0.0750
H(7A)	6*c*	0.1985	0.2099	0.0935	0.0700
H(7B)	6*c*	0.1443	0.2099	0.0510	0.0700
H(7C)	6*c*	0.4453	0.3700	0.0386	0.0720
H(7D)	6*c*	0.4058	0.3451	0.0851	0.0720
H(11A)	6*c*	–0.0744	–0.1507	0.1271	0.0640
H(11B)	6*c*	–0.1260	–0.1462	0.0845	0.0640
H(11E)	6*c*	0.6932	0.6768	0.1379	0.0700
H(11F)	6*c*	0.7324	0.7016	0.0914	0.0700
H(12A)	6*c*	–0.2164	–0.1115	0.1347	0.0610
H(12B)	6*c*	–0.1104	–0.0294	0.1575	0.0610
H(12E)	6*c*	0.8330	0.6392	0.1220	0.0710
H(12F)	6*c*	0.7322	0.5422	0.1425	0.0710
H(14C)	6*c*	–0.0237	0.1648	0.0751	0.0620
H(14D)	6*c*	0.0021	0.1635	0.1225	0.0620

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Table 2. contiued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(14E)	6*c*	0.6107	0.3787	0.0937	0.0670
H(14F)	6*c*	0.6208	0.4076	0.0461	0.0670
H(15)	6*c*	–0.1671	0.1103	0.1548	0.0740
H(15A)	6*c*	0.7844	0.4124	0.1083	0.0830
H(16A)	6*c*	–0.3520	0.0361	0.1512	0.1230
H(16B)	6*c*	–0.3228	–0.0549	0.1542	0.1230
H(16C)	6*c*	–0.3561	–0.0284	0.1110	0.1230
H(16G)	6*c*	0.9669	0.4952	0.0944	0.1590
H(16H)	6*c*	0.9370	0.5682	0.1207	0.1590
H(16I)	6*c*	0.9608	0.5895	0.0727	0.1590
H(17D)	6*c*	–0.2379	0.2021	0.1176	0.1450
H(17E)	6*c*	–0.2209	0.1557	0.0757	0.1450
H(17F)	6*c*	–0.1219	0.2344	0.1029	0.1450
H(17G)	6*c*	0.7290	0.3432	0.0415	0.1430
H(17H)	6*c*	0.8409	0.3550	0.0514	0.1430
H(17I)	6*c*	0.8334	0.4386	0.0224	0.1430

Table 2. continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(18D)	6*c*	0.4281	0.1471	0.0432	0.1480
H(18E)	6*c*	0.4677	0.0773	0.0663	0.1480
H(18F)	6*c*	0.4190	0.1352	0.0921	0.1480
H(18G)	6*c*	0.1729	0.4291	0.0732	0.1550
H(18H)	6*c*	0.1410	0.4915	0.1048	0.1550
H(18I)	6*c*	0.1908	0.4236	0.1213	0.1550
H(19D)	6*c*	0.2876	0.0074	–0.0024	0.1290
H(19E)	6*c*	0.2298	–0.1107	0.0143	0.1290
H(19F)	6*c*	0.3548	–0.0467	0.0099	0.1290
H(19G)	6*c*	0.3021	0.5941	0.0314	0.1210
H(19H)	6*c*	0.3719	0.7005	0.0564	0.1210
H(19I)	6*c*	0.2467	0.6446	0.0578	0.1210
H(1A1)	6*c*	0.5546	0.7066	0.0058	0.0910
H(2)	6*c*	0.0311	–0.1138	–0.0149	0.0920
H(3)	6*c*	–0.2501	–0.0505	0.0673	0.0750
H(3A1)	6*c*	0.8444	0.6296	0.0449	0.0810

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)	6*c*	0.1043(3)	–0.1410(3)	0.11087(9)	0.077(3)	0.073(2)	0.034(2)	0.047(2)	–0.008(2)	–0.002(2)
C(1A)	6*c*	0.5182(3)	0.6819(3)	0.13818(9)	0.076(2)	0.073(2)	0.038(2)	0.041(2)	0.009(2)	0.005(2)
C(2)	6*c*	0.1833(3)	–0.1778(3)	0.0989(1)	0.087(3)	0.097(3)	0.050(2)	0.063(3)	–0.014(2)	–0.012(2)
C(2A)	6*c*	0.4396(3)	0.7233(3)	0.1399(1)	0.098(3)	0.089(3)	0.050(2)	0.058(3)	0.018(2)	0.003(2)
C(3)	6*c*	0.2961(3)	–0.0829(3)	0.0930(1)	0.083(3)	0.115(4)	0.064(3)	0.063(3)	–0.015(2)	–0.023(3)
C(3A)	6*c*	0.3246(3)	0.6336(3)	0.1352(1)	0.084(3)	0.110(3)	0.074(3)	0.065(3)	0.031(2)	0.022(3)
C(4)	6*c*	0.3025(3)	0.0001(3)	0.0617(1)	0.061(3)	0.083(3)	0.059(3)	0.033(2)	–0.005(2)	–0.015(2)
C(4A)	6*c*	0.3047(3)	0.5672(3)	0.0951(1)	0.064(2)	0.076(3)	0.062(3)	0.038(2)	0.016(2)	0.017(2)
C(5)	6*c*	0.2168(3)	0.0308(3)	0.0732(1)	0.059(2)	0.065(2)	0.032(2)	0.024(2)	–0.011(2)	–0.012(2)
C(5A)	6*c*	0.3895(3)	0.5312(3)	0.0932(1)	0.057(2)	0.058(2)	0.050(2)	0.024(2)	0.009(2)	0.012(2)
C(6)	6*c*	0.2200(3)	0.1220(3)	0.0482(1)	0.063(2)	0.060(2)	0.047(2)	0.019(2)	0.001(2)	–0.006(2)
C(6A)	6*c*	0.3735(3)	0.4532(3)	0.0578(1)	0.058(2)	0.055(2)	0.060(2)	0.017(2)	0.003(2)	0.008(2)
C(7)	6*c*	0.1564(2)	0.1652(2)	0.07031(9)	0.064(2)	0.049(2)	0.048(2)	0.018(2)	–0.009(2)	–0.005(2)
C(7A)	6*c*	0.4410(2)	0.4021(3)	0.0647(1)	0.059(2)	0.051(2)	0.053(2)	0.015(2)	0.007(2)	–0.001(2)
C(8)	6*c*	0.0508(3)	0.0796(3)	0.08666(9)	0.055(2)	0.042(2)	0.032(2)	0.017(2)	–0.001(2)	–0.004(2)
C(8A)	6*c*	0.5533(3)	0.4786(3)	0.07976(9)	0.059(2)	0.049(2)	0.038(2)	0.024(2)	0.006(2)	0.005(2)
C(9)	6*c*	0.0253(2)	–0.0222(3)	0.09177(8)	0.055(2)	0.047(2)	0.025(2)	0.023(2)	–0.000(2)	–0.000(2)
C(9A)	6*c*	0.5823(2)	0.5733(3)	0.09668(9)	0.055(2)	0.050(2)	0.032(2)	0.023(2)	0.001(2)	0.000(2)
C(10)	6*c*	0.1000(2)	–0.0644(2)	0.07840(9)	0.055(2)	0.054(2)	0.026(2)	0.025(2)	–0.004(2)	–0.002(2)
C(10A)	6*c*	0.5080(2)	0.6209(2)	0.09713(9)	0.056(2)	0.050(2)	0.034(2)	0.028(2)	0.005(2)	0.002(2)
C(11)	6*c*	–0.0828(3)	–0.1033(3)	0.10788(9)	0.070(2)	0.054(2)	0.040(2)	0.033(2)	0.006(2)	0.008(2)
C(11A)	6*c*	0.6951(3)	0.6443(3)	0.1116(1)	0.068(2)	0.059(2)	0.053(2)	0.036(2)	–0.011(2)	–0.008(2)
C(12)	6*c*	–0.1427(3)	–0.0558(2)	0.13030(9)	0.062(2)	0.055(2)	0.035(2)	0.028(2)	0.003(2)	0.003(2)
C(12A)	6*c*	0.7581(3)	0.5868(3)	0.1177(1)	0.069(2)	0.069(2)	0.046(2)	0.040(2)	–0.007(2)	–0.005(2)
C(13)	6*c*	–0.1411(3)	0.0349(2)	0.10575(9)	0.066(2)	0.046(2)	0.026(2)	0.029(2)	–0.000(2)	–0.004(2)
C(13A)	6*c*	0.7477(3)	0.5176(3)	0.0802(1)	0.067(2)	0.056(2)	0.039(2)	0.035(2)	0.002(2)	0.010(2)
C(14)	6*c*	–0.0251(3)	0.1192(2)	0.0977(1)	0.068(2)	0.046(2)	0.038(2)	0.026(2)	–0.010(2)	–0.005(2)
C(14A)	6*c*	0.6303(3)	0.4371(2)	0.0741(1)	0.074(2)	0.049(2)	0.044(2)	0.031(2)	0.013(2)	0.008(2)
C(15)	6*c*	–0.2021(3)	0.0828(3)	0.1278(1)	0.084(3)	0.060(2)	0.052(2)	0.043(2)	0.002(2)	–0.006(2)
C(15A)	6*c*	0.8142(3)	0.4614(3)	0.0845(1)	0.088(3)	0.077(3)	0.060(3)	0.056(3)	0.004(2)	0.010(2)
C(16)	6*c*	–0.3191(3)	0.0013(3)	0.1369(1)	0.093(3)	0.091(3)	0.079(3)	0.058(3)	0.020(3)	–0.012(2)
C(16A)	6*c*	0.9301(3)	0.5352(4)	0.0939(2)	0.096(3)	0.114(4)	0.142(5)	0.078(3)	–0.014(3)	0.002(3)
C(17)	6*c*	–0.1951(3)	0.1775(3)	0.1038(1)	0.128(4)	0.087(3)	0.106(3)	0.077(3)	0.023(3)	0.013(3)
C(17A)	6*c*	0.8034(4)	0.3932(3)	0.0464(1)	0.143(4)	0.115(3)	0.077(3)	0.100(3)	0.011(3)	–0.001(3)
C(18)	6*c*	0.4149(3)	0.0993(3)	0.0663(1)	0.061(3)	0.121(4)	0.104(4)	0.037(3)	–0.005(2)	–0.024(3)
C(18A)	6*c*	0.1917(3)	0.4686(4)	0.0990(1)	0.061(3)	0.114(4)	0.128(4)	0.038(3)	0.018(3)	0.022(3)
C(19)	6*c*	0.2927(3)	–0.0414(3)	0.0166(1)	0.090(3)	0.123(4)	0.055(2)	0.060(3)	0.008(2)	–0.019(2)
C(19A)	6*c*	0.3065(3)	0.6328(3)	0.0565(1)	0.078(3)	0.101(3)	0.073(3)	0.052(3)	0.003(2)	0.017(2)
C(20)	6*c*	0.0458(3)	–0.1303(3)	0.0391(1)	0.057(2)	0.053(2)	0.039(2)	0.028(2)	–0.000(2)	0.002(2)
C(20A)	6*c*	0.5534(3)	0.7038(3)	0.0616(1)	0.050(2)	0.055(2)	0.036(2)	0.025(2)	0.004(2)	0.003(2)
O(1)	6*c*	–0.0131(2)	–0.2267(2)	0.03991(6)	0.075(2)	0.054(2)	0.042(1)	0.024(1)	–0.003(1)	–0.002(1)
O(1A)	6*c*	0.5291(2)	0.6594(2)	0.02361(6)	0.079(2)	0.052(1)	0.036(1)	0.021(1)	0.002(1)	0.005(1)
O(2)	6*c*	0.0623(2)	–0.0736(2)	0.00476(6)	0.082(2)	0.052(1)	0.032(1)	0.020(1)	–0.010(1)	–0.001(1)
O(2A)	6*c*	0.6116(2)	0.7984(2)	0.06656(7)	0.073(2)	0.045(2)	0.055(2)	0.022(1)	0.001(1)	0.000(1)
O(3)	6*c*	–0.1869(2)	–0.0045(2)	0.06473(6)	0.058(1)	0.052(1)	0.035(1)	0.023(1)	–0.000(1)	–0.001(1)
O(3A)	6*c*	0.7808(2)	0.5844(2)	0.04300(6)	0.058(1)	0.056(1)	0.044(1)	0.025(1)	–0.002(1)	0.009(1)

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