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Crystal structure of isopimara-7,15-dien-3-one, $C_{20}H_{30}O$

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Abstract

$C_{20}H_{30}O$, triclinic, $P\bar{1}$ (no. 1), $a = 6.6084(9)$ Å, $b = 10.8811(16)$ Å, $c = 12.0845(16)$ Å, $\alpha = 85.719(6)^\circ$, $\beta = 79.547(5)^\circ$, $\gamma = 89.903(5)^\circ$, $V = 852.1(2)$ Å³, $Z = 2$, $R_{gt}(F) = 0.0744$, $wR_{ref}(F^2) = 0.1953$, $T = 173(2)$ K.

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The title molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

The leaves and stems of *Isodon amethystoides* collected from Libo country, Guizhou province, were extracted by methanol three times at room temperature. The combined filtrate was concentrated and extracted with petroleum ether. The crude (1.0 kg) product was purified by TLC (silica gel), then divided into A-D fractions by silica gel column with petroleum ether/ethyl acetate (volume ratio 100:1). The colourless block crystals of the title compound were isolated from fraction A and

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.18 × 0.20 × 0.24 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.066 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω -scans
θ_{max} , completeness:	28.5, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	27636, 8549, 0.050
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 7322
$N(param)_{refined}$:	387
Programs:	Bruker programs [1], SHELX [2], OLEX2 [3], DIAMOND [4]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	U_{iso}^*/U_{eq}
O1	−0.1182(5)	−0.1221(3)	0.3766(3)	0.0474(8)
C1	0.0664(5)	0.1781(3)	0.3772(3)	0.0284(7)
H1A	0.040557	0.248408	0.425155	0.034*
H1AB	−0.011683	0.192069	0.314841	0.034*
C2	−0.0114(6)	0.0595(4)	0.4474(3)	0.0327(8)
H2A	0.062911	0.046458	0.511351	0.039*
H2AB	−0.159771	0.066759	0.478526	0.039*
C3	0.0213(5)	−0.0484(3)	0.3765(3)	0.0299(7)
C4	0.2342(5)	−0.0658(3)	0.3075(3)	0.0257(6)
C5	0.3359(5)	0.0608(3)	0.2556(3)	0.0226(6)
H5	0.273041	0.082527	0.187532	0.027*
C6	0.5670(5)	0.0504(3)	0.2106(3)	0.0302(7)
H6A	0.638447	0.024796	0.273660	0.036*
H6AB	0.590071	−0.013489	0.155544	0.036*
C7	0.6551(6)	0.1715(4)	0.1545(3)	0.0311(7)
H7	0.786400	0.171366	0.106912	0.037*
C8	0.5604(5)	0.2789(3)	0.1675(3)	0.0260(6)
C9	0.3559(5)	0.2905(3)	0.2461(3)	0.0238(6)
H9	0.249213	0.297787	0.196961	0.029*
C10	0.2974(5)	0.1737(3)	0.3276(3)	0.0211(6)
C11	0.3455(6)	0.4091(3)	0.3069(3)	0.0349(8)
H11A	0.201818	0.421305	0.344750	0.042*
H11B	0.431672	0.400031	0.365943	0.042*
C12	0.4179(6)	0.5229(3)	0.2283(4)	0.0348(8)
H12A	0.411805	0.595555	0.273369	0.042*
H12B	0.322878	0.537101	0.174127	0.042*
C13	0.6373(6)	0.5111(3)	0.1625(3)	0.0293(7)
C1_1	0.9380(5)	0.6550(4)	0.6204(3)	0.0306(7)
H1A_1	0.964878	0.732791	0.571913	0.037*

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Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
H1AB_1	1.015895	0.658554	0.682819	0.037*
C14	0.6424(6)	0.3953(4)	0.0968(3)	0.0333(8)
H14A	0.560841	0.410859	0.036226	0.040*
H14B	0.786328	0.381290	0.060342	0.040*
C15	0.7944(8)	0.5010(4)	0.2418(4)	0.0460(10)
H15A	0.933900	0.503389	0.197005	0.069*
H15B	0.772347	0.423131	0.288766	0.069*
H15C	0.777250	0.570042	0.290146	0.069*
C16	0.6820(7)	0.6215(4)	0.0784(3)	0.0365(8)
H16	0.603041	0.625889	0.019955	0.044*
C17	0.8120(9)	0.7110(5)	0.0742(5)	0.0652(17)
H17A	0.896394	0.712830	0.129952	0.078*
H17B	0.823287	0.775048	0.015464	0.078*
C18	0.3537(6)	−0.1400(3)	0.3880(3)	0.0331(8)
H18A	0.284887	−0.219717	0.412138	0.050*
H18B	0.357766	−0.093892	0.454279	0.050*
H18C	0.494473	−0.153355	0.348864	0.050*
C19	0.2178(7)	−0.1469(4)	0.2111(4)	0.0409(9)
H19A	0.136213	−0.104717	0.159996	0.061*
H19B	0.150865	−0.225445	0.242179	0.061*
H19C	0.355922	−0.162402	0.169394	0.061*
C20	0.4231(6)	0.1691(3)	0.4232(3)	0.0290(7)
H20A	0.565372	0.194901	0.392344	0.043*
H20B	0.421355	0.084724	0.458054	0.043*
H20C	0.362528	0.224635	0.480146	0.043*
O1_1	1.1184(5)	0.3537(3)	0.6248(3)	0.0535(9)
C2_1	1.0136(6)	0.5470(4)	0.5513(3)	0.0354(8)
H2A_1	0.938153	0.543994	0.487818	0.042*
H2AB_1	1.161857	0.558334	0.519419	0.042*
C3_1	0.9804(5)	0.4280(4)	0.6240(3)	0.0322(8)
C4_1	0.7659(5)	0.4016(3)	0.6943(3)	0.0280(7)
C5_1	0.6653(5)	0.5210(3)	0.7434(3)	0.0227(6)
H5_1	0.727282	0.532541	0.811560	0.027*
C6_1	0.4340(5)	0.5047(3)	0.7878(3)	0.0294(7)
H6A_1	0.363190	0.489163	0.724554	0.035*
H6AB_1	0.409628	0.432439	0.843501	0.035*
C7_1	0.3468(5)	0.6177(3)	0.8422(3)	0.0299(7)
H7_1	0.215437	0.610964	0.889785	0.036*
C8_1	0.4431(5)	0.7268(3)	0.8277(3)	0.0246(6)
C9_1	0.6490(5)	0.7483(3)	0.7495(3)	0.0236(6)
H9_1	0.755049	0.747987	0.798961	0.028*
C10_1	0.7075(5)	0.6436(3)	0.6694(3)	0.0213(6)
C11_1	0.6591(7)	0.8769(4)	0.6874(3)	0.0369(9)
H11A_1	0.802139	0.894125	0.647912	0.044*
H11B_1	0.570435	0.877492	0.629565	0.044*
C12_1	0.5914(6)	0.9791(3)	0.7649(3)	0.0349(8)
H12A_1	0.597429	1.058644	0.718945	0.042*
H12B_1	0.688779	0.984388	0.817762	0.042*
C13_1	0.3723(6)	0.9586(3)	0.8332(3)	0.0295(7)
C14_1	0.3665(6)	0.8319(3)	0.8985(3)	0.0310(7)
H14A_1	0.450695	0.836375	0.957993	0.037*
H14B_1	0.222969	0.813258	0.936376	0.037*
C15_1	0.2132(8)	0.9647(5)	0.7561(5)	0.0497(11)
H15A_1	0.227864	1.043370	0.710196	0.075*
H15B_1	0.074707	0.957956	0.802015	0.075*
H15C_1	0.234756	0.896672	0.706668	0.075*

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
C16_1	0.3382(7)	1.0562(4)	0.9175(4)	0.0376(8)
H16_1	0.427308	1.052999	0.971088	0.045*
C17_1	0.2027(10)	1.1442(6)	0.9263(6)	0.0677(17)
H17A_1	0.108881	1.152606	0.875370	0.081*
H17B_1	0.198101	1.199728	0.983754	0.081*
C18_1	0.6464(6)	0.3385(4)	0.6149(4)	0.0339(8)
H18A_1	0.636614	0.395536	0.549392	0.051*
H18B_1	0.507820	0.315755	0.655444	0.051*
H18C_1	0.719133	0.264262	0.589372	0.051*
C19_1	0.7843(8)	0.3071(4)	0.7929(4)	0.0436(10)
H19A_1	0.844457	0.231192	0.763186	0.065*
H19B_1	0.647463	0.288897	0.837988	0.065*
H19C_1	0.872867	0.340997	0.840276	0.065*
C20_1	0.5809(6)	0.6543(3)	0.5739(3)	0.0297(7)
H20A_1	0.640646	0.719205	0.516787	0.045*
H20B_1	0.438537	0.674946	0.604999	0.045*
H20C_1	0.583098	0.575606	0.539283	0.045*

recrystallized with ethyl acetate. The title compound (56 mg; systematic name: 1,1,4a,7-tetramethyl-7-vinyl-3,4,4a,4b,5,6,7,8,10,10a-decahydrophenanthren-2(1*H*)-one) were obtained after five days.

Experimental details

The carbon-bound hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms with $d(\text{C}—\text{H}) = 0.95–1.00 \text{ \AA}$, $U_{\text{iso}}(\text{H}) = 1.5$ times $U_{\text{eq}}(\text{C})$ and 1.2 times $U_{\text{eq}}(\text{O})$.

Comment

In recent years, many groups have studied the chemical composition and pharmacological activity of the *Isodon* plants, and have shown that they are rich in diterpenoids and have good biological activity including antibacterial, antitumor and immunosuppressive effects [5, 6]. Thus it is necessary to investigate further examples of diterpenoids. The crystal structure of the title compound has not been previously reported.

The title molecular structure contains two alkenyl, one keto, and four methyl groups, respectively. The bond lengths and angles which were derived from the title structure are within normal ranges. The olefinic bonds were identified by the distance $d(\text{C}_7–\text{C}_8) = 1.331(5) \text{ \AA}$ and $d(\text{C}_{16}–\text{C}_{17}) = 1.292(6) \text{ \AA}$, and the keto bonds were confirmed by the distances $d(\text{C}_3–\text{O}_1) = 1.222(5) \text{ \AA}$, respectively.

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