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Crystal structure of *rac*-3,6-dimethyl-5-(prop-1-en-2-yl)-6-vinyl-1,4,5,6-tetrahydro-2*H*-indol-2-one, C₁₅H₁₉NO

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.28 × 0.15 × 0.12 mm
Wavelength:	Cu K α radiation (1.54184 Å)
μ :	0.56 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II CCD, φ and ω
$\theta_{\max}$, completeness:	63.6°, 96%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	9746, 2069, 0.050
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1768
$N(\text{param})_{\text{refined}}$:	157
Programs:	CrysAlis ^{PRO} [1], OLEX2 [2], SHELX [3]

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Abstract

C₁₅H₁₉NO, $M = 229.32$, triclinic, $P\bar{1}$ (no. 2), $a = 6.9535(5)$ Å, $b = 7.4037(6)$ Å, $c = 14.5179(9)$ Å, $\alpha = 97.879(4)^\circ$, $\beta = 93.543(5)^\circ$, $\gamma = 116.785(5)^\circ$, $V = 654.43(9)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0520$, $wR_{\text{ref}}(F^2) = 0.1518$, $T = 173$ K.

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The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

The title compound was prepared according to the known procedure in 7 steps with (+)-verbenone as starting material affording the product as a white solid [4]. The crude product was recrystallized from ethyl acetate as single crystals suitable for X-ray diffraction analysis.

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.6563(3)	0.2502(2)	0.59104(10)	0.0403(4)
N1	0.5548(3)	0.1902(2)	0.43052(12)	0.0345(5)
C1	0.6576(3)	0.3111(3)	0.51614(14)	0.0317(5)
H1	0.4728	0.0559	0.4208	0.041*
C2	0.7725(3)	0.5273(3)	0.50022(14)	0.0305(5)
C3	0.7387(3)	0.5225(3)	0.40802(14)	0.0301(5)
C4	0.8984(4)	0.7053(3)	0.57884(15)	0.0381(5)
H4A	0.7985	0.7456	0.6096	0.057*
H4B	1.0044	0.8215	0.5540	0.057*
H4C	0.9748	0.6655	0.6245	0.057*
C5	0.5983(3)	0.3099(3)	0.36066(14)	0.0311(5)
C6	0.5272(3)	0.2536(3)	0.26954(15)	0.0348(5)
H6	0.4345	0.1123	0.2442	0.042*
C7	0.5897(3)	0.4090(3)	0.20427(14)	0.0325(5)
C8	0.8141(3)	0.5995(3)	0.24824(14)	0.0328(5)
H8	0.9227	0.5460	0.2511	0.039*
C9	0.8114(3)	0.6876(3)	0.35023(14)	0.0344(5)
H9A	0.9591	0.7970	0.3776	0.041*
H9B	0.7118	0.7499	0.3511	0.041*
C10	0.8959(3)	0.7649(3)	0.18803(15)	0.0397(6)
C11	0.8490(4)	0.9225(4)	0.19745(18)	0.0534(7)
H11A	0.9075	1.0251	0.1599	0.064*
H11B	0.7572	0.9318	0.2417	0.064*
C12	1.0387(5)	0.7477(4)	0.1194(2)	0.0636(8)
H12A	1.1599	0.7368	0.1517	0.095*
H12B	1.0959	0.8702	0.0902	0.095*
H12C	0.9549	0.6249	0.0708	0.095*
C13	0.4078(4)	0.4691(3)	0.19024(16)	0.0408(6)

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
H13A	0.3955	0.5388	0.2503	0.061*
H13B	0.2699	0.3451	0.1665	0.061*
H13C	0.4416	0.5620	0.1449	0.061*
C14	0.6172(4)	0.3029(3)	0.11268(15)	0.0404(6)
H14	0.7327	0.2676	0.1154	0.048*
C15	0.5008(5)	0.2542(4)	0.03036(17)	0.0536(7)
H15A	0.3831	0.2856	0.0232	0.064*
H15B	0.5341	0.1875	−0.0223	0.064*

Experimental details

All the Friedel pairs were merged [1–3]. All H atoms were positioned geometrically and treated as riding, with C–H bond lengths constrained to 0.98° for methyl H atoms, 0.99° for ethyl H atoms, 1.00° for tert-butyl H atoms, 0.95° for alkenyl H atoms, and 0.88° for nitric H atoms, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for alkenyl H atoms, and $1.5U_{\text{eq}}(\text{C})$ for methyl H atoms, ethyl H atoms, and tert-butyl H atoms.

Comment

X-ray diffraction structure analysis is a powerful tool to determine the structure of solid natural products. Elema–1, 3,7(11),8–tetraen-8,12-lactam, isolated from the rhizomes of *Curcuma wenyujin* in 2013, [5] is one of γ -elemene-type sesquiterpenes. Previously, we reported the total synthesis of this natural product [4]. Here we report the crystal structure of the title compound.

There is one molecule in the asymmetric unit (see the figure). In the crystal structure, the crystal packing is stabilized in layers and held together by van der Waals.

Geometric parameters derived from the title crystal structure are in accord with related ones [6].

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References

1. Agilent Technologies: CrysAlis^{Pro} Software System. Agilent Technologies UK Ltd, Version 1.171.37.35, Oxford, UK (2011).
2. Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H.: OLEX2: a complete structure solution, refinement and analysis program. *J. Appl. Cryst.* **42** (2015) 339–341.
3. Sheldrick, G. M.: SHELXT—integrated space-group and crystal-structure determination. *Acta Crystallogr.* **A71** (2015) 3–8.
4. Yuan, C.; Zhong, S.; Li, X.; Wang, Y.; Xun, M.; Bai, Y.; Zhu, K.: Total synthesis, structural revision and biological evaluation of γ -elemene-type sesquiterpenes. *Org. Biomol. Chem.* **42** (2018) 7843.
5. Qiu, G.; Yan, P.; Shao, W.; Zhou, J.; Lin, W.; Fang, L.; Zhao, X.; Dong, J.: Two new sesquiterpenoids including a sesquiterpenoid lactam from *Curcuma wenyujin*. *Chem. Pharm. Bull.* **61** (2013) 983–986.
6. Hökelek, T.; Erçag, A.; Çoruh, U.; Vazquez-Lopez, E. M.; Üstün Özgür, M.: Crystal structure of (3)-3-[(4-ethoxyphenyl)imino]-1,3-dihydro-2*H*-indol-2-one. *Anal. Sci.: X-ray Struct. Anal. Online* **21** (2005) x129–x130.