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Crystal structure of 5,7,7-trimethyl-4,6,7,8-tetrahydrocyclopenta[*g*]isochromen-1(3*H*)-one, C₁₅H₁₈O₂

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.19 × 0.15 × 0.12 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.08 mm ⁻¹
Diffractometer, scan mode:	D8 VENTURE, φ and ω
$\theta_{\max}$, completeness:	26.0°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	23,718, 4971, 0.064
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3594
$N(\text{param})_{\text{refined}}$:	313
Programs:	SHELX [1], Olex2 [2], CrysAlis ^{PRO} [3]

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Abstract

C₁₅H₁₈O₂, monoclinic, $P2_1/n$ (no. 14), $a = 12.7072(5)$ Å, $b = 11.2947(6)$ Å, $c = 17.7857(7)$ Å, $\beta = 95.807(1)^\circ$, $V = 2539.6(2)$ Å³, $Z = 8$, $R_{\text{gt}}(F) = 0.0606$, $wR_{\text{ref}}(F^2) = 0.1759$, $T = 170$ K.

CCDC no.: 2047041

Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

The title compound was prepared according to the known procedure in five steps with 3-iodocyclopent-2-enone as

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

Atom	X	Y	Z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.81040 (17)	−0.0797 (2)	0.53443 (14)	0.0386 (5)
C2	0.87213 (16)	0.03224 (18)	0.54322 (12)	0.0319 (5)
C3	0.87155 (16)	0.11269 (19)	0.48319 (12)	0.0324 (5)
C4	0.80017 (19)	0.0878 (2)	0.41239 (13)	0.0410 (6)
H4A	0.840492	0.046224	0.375555	0.049*
H4B	0.773931	0.163383	0.389388	0.049*
C5	0.7080 (2)	0.0127 (2)	0.42972 (17)	0.0511 (7)
H5A	0.661042	0.060088	0.459163	0.061*
H5B	0.666980	−0.010736	0.381761	0.061*
C6	0.93593 (17)	0.05035 (19)	0.61108 (13)	0.0340 (5)
H6	0.936081	−0.005029	0.651295	0.041*
C7	0.99852 (18)	0.1501 (2)	0.61843 (13)	0.0363 (5)
C8	1.0748 (2)	0.1879 (2)	0.68364 (16)	0.0548 (7)
H8A	1.137617	0.135570	0.688631	0.066*
H8B	1.040950	0.185263	0.731357	0.066*
C9	1.10672 (19)	0.3162 (2)	0.66557 (14)	0.0407 (6)
C10	1.2225 (3)	0.3399 (4)	0.6866 (2)	0.0843 (11)
H10A	1.264626	0.283127	0.660708	0.126*
H10B	1.238828	0.331544	0.741393	0.126*
H10C	1.239449	0.420571	0.671440	0.126*
C11	1.0375 (3)	0.4002 (3)	0.70572 (19)	0.0746 (10)
H11A	1.055541	0.482155	0.694289	0.112*
H11B	1.049383	0.387034	0.760388	0.112*
H11C	0.962980	0.385507	0.688390	0.112*
C12	1.0780 (2)	0.3283 (2)	0.57901 (14)	0.0458 (6)
H12A	1.045904	0.406617	0.566384	0.055*
H12B	1.141722	0.319033	0.551800	0.055*
C13	0.99932 (17)	0.22975 (18)	0.55811 (12)	0.0334 (5)
C14	0.93794 (17)	0.21327 (18)	0.49002 (12)	0.0324 (5)

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Table 2: (continued)

Atom	X	Y	Z	U_{iso}^*/U_{eq}
C15	0.9430 (2)	0.2974 (2)	0.42444 (13)	0.0414 (6)
H15A	0.966430	0.254402	0.381261	0.062*
H15B	0.993138	0.361217	0.439179	0.062*
H15C	0.872723	0.331142	0.410332	0.062*
C16	0.57985 (18)	0.3512 (2)	0.3883 (13)	0.0391 (5)
C17	0.49736 (16)	0.44431 (18)	0.37922 (12)	0.0308 (5)
C18	0.40918 (16)	0.43965 (18)	0.42091 (12)	0.0297 (5)
C19	0.4465 (2)	0.2274 (2)	0.43416 (16)	0.0474 (6)
H19A	0.442412	0.157351	0.467213	0.057*
H19B	0.405190	0.209866	0.385213	0.057*
C20	0.39868 (19)	0.33172 (19)	0.46990 (13)	0.0395 (5)
H20A	0.435414	0.345573	0.520925	0.047*
H20B	0.323095	0.316195	0.475133	0.047*
C21	0.33433 (16)	0.53141 (18)	0.41471 (12)	0.0305 (5)
C22	0.35049 (16)	0.62481 (19)	0.36577 (12)	0.0316 (5)
C23	0.43771 (16)	0.62848 (19)	0.32354 (12)	0.0316 (5)
C24	0.51170 (16)	0.53907 (19)	0.33027 (12)	0.0330 (5)
H24	0.571463	0.541492	0.302251	0.040*
C25	0.43246 (19)	0.7368 (2)	0.27311 (14)	0.0415 (6)
H25A	0.409461	0.715360	0.220016	0.050*
H25B	0.502122	0.776626	0.275294	0.050*
C26	0.34980 (18)	0.81710 (19)	0.30597 (13)	0.0368 (5)
C27	0.4084 (2)	0.8974 (2)	0.36643 (17)	0.0572 (7)
H27A	0.357097	0.946665	0.389681	0.086*
H27B	0.458130	0.948348	0.342783	0.086*
H27C	0.447355	0.848354	0.405291	0.086*
C28	0.2884 (2)	0.8924 (3)	0.24597 (18)	0.0651 (8)
H28A	0.250289	0.840803	0.208128	0.098*
H28B	0.337514	0.942886	0.221476	0.098*
H28C	0.237650	0.941998	0.269549	0.098*
C29	0.28053 (19)	0.7306 (2)	0.34606 (16)	0.0441 (6)
H29A	0.257240	0.766908	0.392237	0.053*
H29B	0.217290	0.707268	0.312178	0.053*
C30	0.23743 (19)	0.5270 (2)	0.45732 (14)	0.0443 (6)
H30A	0.190432	0.463553	0.436717	0.066*
H30B	0.200130	0.602953	0.451977	0.066*
H30C	0.258878	0.511660	0.510917	0.066*
O1	0.55623 (14)	0.24826 (15)	0.42165 (11)	0.0511 (5)
O2	0.66797 (13)	0.36239 (16)	0.36863 (11)	0.0513 (5)
O3	0.74133 (13)	−0.09309 (15)	0.47225 (11)	0.0508 (5)
O4	0.82093 (13)	−0.16116 (14)	0.57877 (10)	0.0483 (5)

starting material affording the product as a white solid [4]. The crude product was recrystallized from ethyl acetate.

Experimental details

All H atoms were positioned geometrically and treated as riding, with C–H bond lengths constrained to 0.98 Å for

methyl H atoms, 0.99 Å for methylene H atoms, 0.95 Å for aryl H atoms, and with $U_{iso}(H) = 1.2U_{eq}(C)$ and $1.5U_{eq}(C)$.

Comment

5,7,7-trimethyl-4,6,7,8-tetrahydrocyclopenta[g]isochromen-1(3H)-one, isolated from an agar plate culture of *Granulobasidium vellereum* [5], is a illudalane sesquiterpene. Previously, we reported the total synthesis of this natural product [4]. Here we report the crystal structure of the title compound.

The figure shows one of the two crystallographically independent molecules which define the asymmetric unit. All geometric parameters are in the expected ranges [6]. In the crystal structure, the crystal packing is stabilized in layers and held together by Van der Waals forces.

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References

- Sheldrick G. M. *SHELXTL* – Integrated space-group and crystal-structure determination. *Acta Crystallogr.* 2015, *A71*, 3–8.
- 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. Crystallogr.* 2009, *42*, 339–341.
- Agilent Technologies. *CrysAlis^{PRO}, Version 1.171.37.35*; Agilent Technologies: Santa Clara, CA, USA, 2017.
- Xun M., Bai Y., Wang Y., Hu Z., Fu K., Ma W., Yuan C. Synthesis of four illudalane sesquiterpenes utilizing a one-pot Diels–Alder/oxidative aromatization sequence. *Org. Lett.* 2019, *21*, 6879–6883.
- Kokubun T., Scott-Brown A., Kite G. C., Simmonds M. S. J. Protoilludane, illudane, illudalane, and norilludane sesquiterpenoids from *Granulobasidium vellereum*. *J. Nat. Prod.* 2016, *79*, 1698–1701.
- Zeng Z., Zhao Y., Zhang Y. Divergent total syntheses of five illundalane sesquiterpenes and assignment of the absolute configuration. *Chem. Commun.* 2019, *55*, 4250–4253.