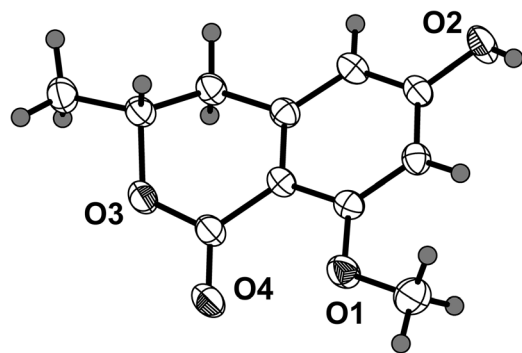


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The crystal structure of (*R*)-6-hydroxy-8-methoxy-3-methylisochroman-1-one, C₁₁H₁₂O₄



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Abstract

C₁₁H₁₂O₄, monoclinic, *P*2₁ (no. 4), *a* = 15.4586(2) Å, *b* = 7.80238(13) Å, *c* = 16.4003(2) Å, β = 91.0103(13)°, *V* = 1977.81(5) Å³, *Z* = 8, *R*_{gt}(*F*) = 0.0413, *wR*_{ref}(*F*²) = 0.1045, *T* = 180 K.

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

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.16 × 0.13 × 0.12 mm
Wavelength:	Cu Kα radiation (1.54184 Å)
μ:	0.90 mm ⁻¹
Diffractometer, scan mode:	SuperNova, ω
θ _{max} , completeness:	73.8°, >99%
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	10,690, 6502, 0.023
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 6386
<i>N</i> (<i>param</i>) _{refined} :	553
Programs:	CrysAlis ^{PRO} [1], SHELX [2], Olex2 [3]

Source of material

All solvents were of analytical grade. The title compound was isolated and purified from the secondary metabolites of *Talaromyces* sp. Colorless crystals were obtained by slow-evaporative solution of the methanol at room temperature.

Experimental details

All hydrogen atoms were placed in calculated positions and refined using a riding model with the relative isotropic parameters. *U*_{iso} values of hydrogen atoms were set to 1.2*U*_{eq} of the parent atoms.

Comment

The endophytic fungus *Talaromyces* sp. was isolated from mangrove tree *Sonneratia apetala* in Dongzhai harbor, Hainan Province, and identified by 18S DNA gene sequence [4]. The specimen was stored at Guangzhou Medical University, Guangzhou, P. R. China. This fungus strain was fermented statically by using rice medium (85 g rice, 160 mL coarse sea salt solution with a mass concentration of 2 g/L) in a 1 L Erlenmeyerflask at 28 °C for forty days. The culture was repetitively extracted with MeOH solvent and then the extract was further fractionated by silica gel chromatography using a stepwise

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

Atom	x	y	z	U_{iso}^*/U_{eq}
O1	0.11870 (10)	0.7354 (3)	0.29776 (10)	0.0357 (4)
O2	−0.08576 (10)	0.4933 (3)	0.48163 (11)	0.0382 (4)
H2	−0.120063	0.533356	0.448006	0.057*
O3	0.31167 (10)	0.6880 (3)	0.47316 (10)	0.0334 (4)
O4	0.28372 (11)	0.6905 (3)	0.34198 (11)	0.0458 (5)
C1	0.09698 (14)	0.6621 (3)	0.36961 (13)	0.0265 (5)
C2	0.01304 (14)	0.6156 (3)	0.38727 (14)	0.0272 (5)
H2A	−0.031420	0.635080	0.349462	0.033*
C3	−0.00462 (14)	0.5393 (3)	0.46222 (14)	0.0277 (5)
C4	0.06082 (14)	0.5094 (3)	0.51939 (14)	0.0292 (5)
H4	0.048662	0.455217	0.568370	0.035*
C5	0.14425 (14)	0.5609 (3)	0.50296 (13)	0.0261 (5)
C6	0.16449 (13)	0.6356 (3)	0.42781 (13)	0.0256 (4)
C7	0.25485 (14)	0.6750 (3)	0.41041 (13)	0.0300 (5)
C8	0.27887 (13)	0.6868 (4)	0.55708 (13)	0.0292 (5)
H8	0.248437	0.794441	0.567728	0.035*
C9	0.21706 (14)	0.5393 (3)	0.56432 (14)	0.0293 (5)
H9A	0.194078	0.535786	0.618952	0.035*
H9B	0.247085	0.432374	0.554473	0.035*
C10	0.35783 (15)	0.6741 (4)	0.61237 (15)	0.0362 (6)
H10A	0.385073	0.564769	0.604910	0.054*
H10B	0.340812	0.685677	0.668114	0.054*
H10C	0.397764	0.763727	0.599223	0.054*
C11	0.05132 (17)	0.7581 (4)	0.23809 (15)	0.0415 (6)
H11A	0.074389	0.812465	0.190660	0.062*
H11B	0.006621	0.828727	0.260232	0.062*
H11C	0.027648	0.648516	0.223245	0.062*
O5	0.40796 (10)	0.8674 (3)	0.27311 (11)	0.0381 (4)
H5	0.374934	0.810969	0.301020	0.057*
O6	0.62498 (11)	0.5890 (3)	0.43905 (10)	0.0351 (4)
O7	0.78551 (10)	0.6210 (3)	0.38987 (10)	0.0351 (4)
O8	0.80964 (10)	0.7069 (3)	0.26558 (10)	0.0313 (4)
C44	0.75444 (13)	0.6762 (3)	0.32636 (13)	0.0268 (5)
C12	0.55230 (14)	0.8666 (3)	0.23338 (14)	0.0288 (5)
H12	0.536747	0.931754	0.187971	0.035*
C13	0.48998 (14)	0.8180 (3)	0.28878 (14)	0.0282 (5)
C14	0.51317 (14)	0.7245 (3)	0.35811 (13)	0.0281 (5)
H14	0.471355	0.693561	0.395447	0.034*
C15	0.59892 (14)	0.6775 (3)	0.37133 (13)	0.0262 (4)
C16	0.66248 (13)	0.7198 (3)	0.31381 (13)	0.0250 (5)
C17	0.63707 (13)	0.8181 (3)	0.24576 (13)	0.0256 (4)
C18	0.70565 (14)	0.8732 (3)	0.18732 (14)	0.0294 (5)
H18A	0.679855	0.890185	0.133576	0.035*
H18B	0.730440	0.981355	0.205244	0.035*
C19	0.77542 (14)	0.7406 (3)	0.18286 (13)	0.0287 (5)
H19	0.751372	0.634627	0.159763	0.034*
C20	0.85162 (15)	0.7972 (4)	0.13335 (15)	0.0376 (6)
H20A	0.871677	0.906554	0.152661	0.056*
H20B	0.834327	0.806418	0.077016	0.056*
H20C	0.897339	0.714481	0.138869	0.056*
C21	0.56364 (17)	0.5605 (4)	0.50141 (15)	0.0397 (6)
H21A	0.516201	0.494228	0.479830	0.060*
H21B	0.542557	0.668659	0.520686	0.060*
H21C	0.590832	0.499474	0.545753	0.060*

Table 2: (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
O9	1.02791 (10)	0.3470 (3)	0.05186 (10)	0.0385 (4)
O10	0.80644 (11)	0.2873 (3)	0.25194 (10)	0.0379 (4)
H10	0.775040	0.304914	0.212003	0.057*
O11	1.21349 (10)	0.2112 (3)	0.22487 (10)	0.0390 (4)
O12	1.17659 (11)	0.1847 (3)	0.09616 (11)	0.0473 (5)
C31	1.15305 (15)	0.2186 (4)	0.16495 (14)	0.0342 (5)
C22	0.88945 (15)	0.2825 (3)	0.22878 (14)	0.0306 (5)
C23	0.91322 (14)	0.3167 (4)	0.14829 (14)	0.0308 (5)
H23	0.871473	0.345647	0.109156	0.037*
C24	0.99985 (15)	0.3067 (4)	0.12767 (13)	0.0300 (5)
C25	1.06338 (14)	0.2574 (3)	0.18601 (14)	0.0290 (5)
C26	1.03678 (15)	0.2300 (3)	0.26683 (14)	0.0303 (5)
C27	0.95162 (15)	0.2438 (4)	0.28778 (14)	0.0332 (5)
H27	0.935583	0.227112	0.341588	0.040*
C28	1.10551 (15)	0.1868 (4)	0.32939 (14)	0.0362 (6)
H28A	1.112133	0.063335	0.332757	0.043*
H28B	1.088174	0.228217	0.382413	0.043*
C29	1.19049 (15)	0.2668 (4)	0.30723 (14)	0.0362 (6)
H29	1.184786	0.391920	0.308074	0.043*
C30	1.26421 (18)	0.2139 (6)	0.36337 (17)	0.0525 (8)
H30A	1.274193	0.093058	0.357983	0.079*
H30B	1.249735	0.239711	0.418716	0.079*
H30C	1.315567	0.275398	0.349102	0.079*
C41	0.96584 (17)	0.4097 (5)	−0.00603 (15)	0.0453 (7)
H41A	0.994415	0.440147	−0.055468	0.068*
H41B	0.937651	0.508835	0.015750	0.068*
H41C	0.923620	0.322261	−0.017400	0.068*
O13	0.71649 (11)	0.2265 (3)	0.02857 (12)	0.0397 (4)
O14	0.68208 (11)	0.3443 (3)	0.14428 (11)	0.0406 (5)
O15	0.52403 (11)	0.3026 (3)	0.19775 (10)	0.0398 (4)
O16	0.30999 (10)	0.1394 (3)	−0.00258 (10)	0.0387 (4)
H16	0.277822	0.156006	0.035851	0.058*
C33	0.56455 (15)	0.2413 (3)	0.06262 (14)	0.0295 (5)
C34	0.65577 (15)	0.2743 (3)	0.08202 (15)	0.0327 (5)
C32	0.49920 (15)	0.2543 (3)	0.12123 (14)	0.0305 (5)
C42	0.41374 (15)	0.2181 (3)	0.10082 (14)	0.0313 (5)
H42	0.371252	0.223186	0.140192	0.038*
C43	0.39216 (14)	0.1738 (3)	0.02059 (14)	0.0297 (5)
C35	0.45472 (15)	0.1655 (3)	−0.03923 (14)	0.0309 (5)
H35	0.439303	0.137992	−0.092711	0.037*
C36	0.54001 (14)	0.1986 (3)	−0.01821 (14)	0.0289 (5)
C37	0.60949 (15)	0.1943 (4)	−0.08069 (15)	0.0346 (5)
H37A	0.618999	0.308958	−0.101531	0.042*
H37B	0.591335	0.121907	−0.125943	0.042*
C38	0.69245 (16)	0.1259 (4)	−0.04365 (17)	0.0395 (6)
H38	0.683867	0.006247	−0.027594	0.047*
C39	0.76802 (18)	0.1364 (6)	−0.1005 (2)	0.0573 (9)
H39A	0.776540	0.253348	−0.116717	0.086*
H39B	0.756306	0.067496	−0.147965	0.086*
H39C	0.819267	0.094975	−0.073127	0.086*
C40	0.45859 (17)	0.3108 (4)	0.25805 (15)	0.0418 (6)
H40A	0.414461	0.390105	0.240841	0.063*
H40B	0.483794	0.348421	0.308884	0.063*
H40C	0.433549	0.199221	0.264962	0.063*

isocratic solvent system according to the increasing polarity starting from 100% petroleum to 100% methanol using normal-phase vacuum liquid chromatography (VLC) to obtain nine fractions (Fr. A–I). The title compound was isolated and purified from Fraction F (20% petroleum/80% ethyl acetate) through silica gel column chromatography [5]. Its structure was elucidated by comprehensive analysis of spectroscopic data and confirmed by X-ray crystallography.

The asymmetric unit of the title compound contains four crystallographically independent molecules (only one of the molecules is shown in the figure). And the crystal structure of two similar compounds, (R)-8-hydroxy-6-methoxy-3-methylisochroman-1-one [6] and (R)-6,8-dihydroxy-3-methylisochroman-1-one [7], have been previously reported. The absolute configuration of the chiral center at C-2 both was determined to be R.

The title compound has been also reported from *Penicillium* sp. and *Talaromyces thailandiasis*., containing its ^1H NMR, ^{13}C NMR data [6–9]. It exhibited a moderate cytotoxic activity against the mouse lymphoma cells (L5178Y) with IC_{50} value of 13.70 μM and weak anti-HIV activities with the IC_{50} values of 69.3 μM [6, 9]. What's more, the X-ray crystallography data of the title compound was reported here for the first time. The geometric parameters are similar to those reported for the (–)-(3R)-6-methoxy-mellein [10].

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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