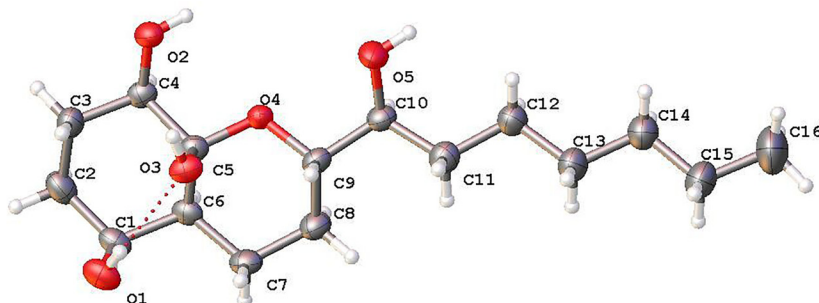


Guangdi Wang, Shihao Qian, Sun Yan and Tingchi Wen*

Crystal structure of (2-(1-hydroxyheptyl) octahydro-8aH-chromene-5,8,8a-triol), C₁₆H₃₀O₅



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Abstract

C₁₆H₃₀O₅, monoclinic, $P2_1$ (no. 4), $a = 5.6814(5)$ Å, $b = 13.0181(12)$ Å, $c = 22.833(2)$ Å, $\beta = 96.473(2)^\circ$, $V = 1677.9(3)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0508$, $wR_{\text{ref}}(F^2) = 0.1346$, $T = 296(2)$ K.

CCDC no.: 2117133

The 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

Stilbella buquetii was cultured on slants of potato dextrose agar (PDA) at 25 °C for 15 days. Agar plugs were cut into small pieces (about 0.5 × 0.5 × 0.5 cm³) under aseptic conditions and 15 of these pieces were used to inoculate in

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.20 × 0.20 × 0.20 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.09 mm ⁻¹
Diffractometer, scan mode:	CCD, ϕ and ω
θ_{max} , completeness:	25.0°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	35,545, 5903, 0.084
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 4005
$N(\text{param})_{\text{refined}}$:	381
Programs:	SHELX [1–4], Diamond [5]

three Erlenmeyer flasks (1 L), each containing 400 mL of media (1% protein, 4% glucose) [6]; 15 flasks of the inoculated media were incubated at 25 °C on a rotary shaker at 170 rpm for five days to prepare the seed culture. Fermentation was carried out in 500 Fernbach flasks (500 mL), each containing 2 g of protein, 8 g of glucose. Distilled H₂O (200 mL) was added to each flask, and autoclaving at 15 psi for 30 min. After cooling to room temperature, each flask was inoculated with 5 mL of the spore inoculum and incubated at 25 °C for 60 days. The culture was filtered to separate the residue (mycelia) and the filtrate (broth). The broth was extracted with EtOAc (3 × 10 L) and concentrated to a brown gum (33 g; extract A). The mycelium was macerated in EtOAc (1.5 L, rt, three days) and filtered. The filtrate was partially concentrated to a brown gum (130 g; Extract B). Combine extract A and extract B, and perform column chromatography on silica gel (10 × 150 cm), using PE/EtOAc as the eluent (gradient from 100:0 to 0:100) to obtain 11 fractions. Fraction 6 (8 g) was repeatedly ODS (manually

*Corresponding author: Tingchi Wen, College of Pharmacy, Guizhou University, Guiyang 550025, G. P, China, E-mail: 10740826@qq.com. <https://orcid.org/0000-0003-1744-5869>

Guangdi Wang, Shihao Qian and Sun Yan, College of Pharmacy, Guizhou University, Guiyang 550025, G. P, China. <https://orcid.org/0000-0002-0448-3324> (G. Wang)

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
C1	−0.1136 (7)	0.0479 (3)	0.75195 (19)	0.0367 (11)
H1	−0.0908	−0.0106	0.7790	0.044*
C2	−0.0993 (8)	0.0069 (3)	0.6903 (2)	0.0454 (12)
H2A	−0.2378	−0.0351	0.6786	0.054*
H2B	0.0395	−0.0366	0.6906	0.054*
C3	−0.0855 (8)	0.0929 (4)	0.64492 (19)	0.0404 (11)
H3A	−0.0645	0.0633	0.6069	0.049*
H3B	−0.2326	0.1315	0.6408	0.049*
C4	0.1175 (7)	0.1640 (3)	0.66384 (17)	0.0318 (11)
H4	0.2659	0.1251	0.6658	0.038*
C5	0.0925 (7)	0.2083 (3)	0.72393 (18)	0.0299 (10)
C6	0.0856 (7)	0.1237 (3)	0.76975 (18)	0.0321 (10)
H6	0.2339	0.0853	0.7698	0.039*
C7	0.0889 (8)	0.1710 (3)	0.83109 (18)	0.0386 (11)
H7A	−0.0593	0.2067	0.8340	0.046*
H7B	0.1042	0.1170	0.8605	0.046*
C8	0.2949 (8)	0.2462 (4)	0.84296 (19)	0.0414 (11)
H8A	0.4433	0.2087	0.8460	0.050*
H8B	0.2846	0.2808	0.8802	0.050*
C9	0.2914 (8)	0.3259 (3)	0.79368 (18)	0.0336 (11)
H9	0.1455	0.3663	0.7928	0.040*
C10	0.5011 (8)	0.3990 (3)	0.79911 (17)	0.0342 (11)
H10	0.6465	0.3580	0.8015	0.041*
C11	0.5095 (8)	0.4641 (4)	0.8543 (2)	0.0420 (12)
H11A	0.3653	0.5045	0.8522	0.050*
H11B	0.5103	0.4186	0.8880	0.050*
C12	0.7189 (8)	0.5364 (4)	0.8653 (2)	0.0456 (12)
H12A	0.8643	0.4975	0.8651	0.055*
H12B	0.7122	0.5863	0.8336	0.055*
C13	0.7233 (9)	0.5926 (4)	0.9235 (2)	0.0518 (13)
H13A	0.5775	0.6315	0.9231	0.062*
H13B	0.7246	0.5419	0.9547	0.062*
C14	0.9286 (9)	0.6649 (4)	0.9387 (2)	0.0549 (14)
H14A	0.9272	0.7166	0.9081	0.066*
H14B	1.0753	0.6266	0.9394	0.066*
C15	0.9228 (10)	0.7176 (5)	0.9974 (2)	0.0668 (17)
H15A	0.7758	0.7558	0.9964	0.080*
H15B	0.9215	0.6654	1.0277	0.080*
C16	1.1256 (13)	0.7900 (6)	1.0149 (3)	0.102 (3)
H16A	1.2729	0.7549	1.0126	0.153*
H16B	1.1180	0.8134	1.0545	0.153*
H16C	1.1151	0.8478	0.9886	0.153*
C17	−0.3180 (8)	0.5036 (4)	0.4719 (2)	0.0434 (12)
H17	−0.3572	0.5537	0.4403	0.052*
C18	−0.5337 (8)	0.4342 (5)	0.4738 (2)	0.0548 (14)
H18A	−0.5517	0.3914	0.4388	0.066*
H18B	−0.6744	0.4766	0.4733	0.066*
C19	−0.5144 (8)	0.3654 (4)	0.52838 (19)	0.0452 (12)
H19A	−0.6612	0.3277	0.5293	0.054*
H19B	−0.3879	0.3160	0.5262	0.054*
C20	−0.4650 (7)	0.4281 (4)	0.58418 (18)	0.0359 (11)
H20	−0.5985	0.4748	0.5870	0.043*
C21	−0.2427 (7)	0.4916 (3)	0.58211 (19)	0.0335 (11)

Table 2: (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
C22	−0.2662 (7)	0.5634 (3)	0.52925 (18)	0.0374 (11)
H22	−0.4060	0.6061	0.5328	0.045*
C23	−0.0549 (8)	0.6369 (4)	0.5323 (2)	0.0451 (12)
H23A	0.0874	0.5987	0.5267	0.054*
H23B	−0.0812	0.6873	0.5009	0.054*
C24	−0.0216 (10)	0.6914 (4)	0.5915 (2)	0.0531 (14)
H24A	0.1204	0.7333	0.5941	0.064*
H24B	−0.1556	0.7362	0.5951	0.064*
C25	−0.0007 (8)	0.6131 (3)	0.64125 (19)	0.0400 (12)
H25	0.1398	0.5707	0.6380	0.048*
C26	0.0117 (8)	0.6542 (4)	0.7037 (2)	0.0434 (12)
H26	−0.1327	0.6936	0.7076	0.052*
C27	0.2246 (9)	0.7224 (4)	0.7214 (2)	0.0497 (13)
H27A	0.3681	0.6833	0.7186	0.060*
H27B	0.2230	0.7794	0.6940	0.060*
C28	0.2296 (9)	0.7644 (4)	0.7835 (2)	0.0555 (14)
H28A	0.0836	0.8019	0.7862	0.067*
H28B	0.2327	0.7070	0.8106	0.067*
C29	0.4351 (9)	0.8343 (4)	0.8033 (2)	0.0559 (14)
H29A	0.4370	0.8906	0.7755	0.067*
H29B	0.5817	0.7963	0.8028	0.067*
C30	0.4248 (10)	0.8779 (5)	0.8644 (2)	0.0618 (15)
H30A	0.2769	0.9151	0.8646	0.074*
H30B	0.4222	0.8211	0.8918	0.074*
C31	0.6232 (11)	0.9479 (5)	0.8862 (3)	0.0713 (17)
H31A	0.6306	1.0033	0.8581	0.086*
H31B	0.7709	0.9101	0.8880	0.086*
C32	0.6017 (14)	0.9936 (6)	0.9461 (3)	0.094 (2)
H32A	0.4631	1.0362	0.9441	0.141*
H32B	0.7396	1.0343	0.9583	0.141*
H32C	0.5888	0.9394	0.9741	0.141*
O1	−0.3429 (5)	0.0896 (2)	0.75762 (13)	0.0463 (8)
H1A	−0.3465	0.1501	0.7476	0.069*
O2	0.1230 (5)	0.2441 (2)	0.62126 (11)	0.0357 (7)
H2	0.2453	0.2774	0.6284	0.054*
O3	−0.1206 (5)	0.2655 (2)	0.72367 (12)	0.0343 (7)
H3	−0.1512	0.2936	0.6916	0.051*
O4	0.2920 (5)	0.2739 (2)	0.73800 (11)	0.0308 (7)
O5	0.4909 (6)	0.4626 (2)	0.74689 (12)	0.0438 (8)
H5	0.6027	0.5028	0.7506	0.066*
O7	−0.1194 (5)	0.4453 (3)	0.45705 (14)	0.0514 (9)
H7	−0.0573	0.4156	0.4865	0.077*
O8	−0.0430 (4)	0.4282 (2)	0.57679 (12)	0.0375 (8)
H8	−0.0502	0.3768	0.5973	0.056*
O9	−0.2074 (5)	0.5470 (2)	0.63589 (12)	0.0390 (8)
O10	0.0225 (6)	0.5695 (3)	0.74382 (13)	0.0530 (9)
H10A	0.1055	0.5240	0.7321	0.079*
O6	−0.4460 (5)	0.3606 (2)	0.63424 (12)	0.0386 (7)
H6A	−0.4306	0.3948	0.6646	0.058*

assembled C18 reverse column chromatography) column using a step gradient elution with hexane/EtOAc (from 100:0 to 60:40) to obtain nine fractions. The product

(2-(1-hydroxyheptyl)octahydro-8aH-chromene-5,8,8a-triol) (33 mg) was obtained using ethyl acetate and methyl alcohol recrystallization in the fraction 6–6.

Experimental details

Carbon-bound H atoms were placed in calculated positions (C–H = 0.97 Å (methylene), C–H = 0.96 Å, (methyl), and O–H = 0.82 Å(–OH)). The absolute configuration was derived from the configuration of the educts.

Comment

A series of complex pyranones discovered from *Trichoderma* conring are called koninginin. The main types of koninginin discovered so far are koninginin A–E and Koninginin G [7–9]. Koninginin G is also named (2*S*,4*aR*,5*R*,8*S*, 8*aS*)-2-((*S*)-1-hydroxyheptyl)octahydro-2*H*-chromene-5. Although its molecular structure has been discovered [10], its crystal structure has not yet been reported. It should be noted that 1,4,5,6-tetra-epi-koninginin G was extracted from *Stilbella buquetii* for the first time.

The structure solution shows that the title compound is a derivative of koninginin A. There are two crystallographically independent molecules in the asymmetric unit. The bond length in both molecules are within the expected range.

We speculate that the C7–O–C10 position of koninginin A is hydrolyzed by hydrolase to form two hydroxyl groups to form koninginin G (C5–OH, C10–OH).

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

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