Electronic supplementary material (ESM)

Structural Revision and Pharmacological Activity of Hemslecin C

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1. Synthesis of hemslecin C.

This paper reports on the synthesis of hemslecin C, according to the preliminary work of literature[1], as shown in Scheme 1. Starting from dissolving hemslecin A in EtOH, adding KOH in reaction system when it dissolves completely. Reflux for 4 hours, and checking by TLC. After air-cooling the reaction, the reaction mixture was poured into separatory funnel and separated. The aqueous layer was extracted with ethyl acetate three times. The combined organic layers were washed with a saturated salt water and dried over Na_2SO_4. The organic layer was filtered and concentrated under reduced pressure to dryness to provide crude product. The crude product was purified by silica gel chromatography eluted with CH_2Cl_2: CH_3OH = 20: 1 to give product as white solid. HRMS (ESI) calcd. for $C_{32}H_{49}O_7$ $(M+H)^+$ 545.3478, found 545.3442, and $(2M+Na)^+$ was found for 1111. 6671. $[\alpha]_D^{25}$ = +111.0 (*c* = 0.1, EtOH) M..p: 160-162 °C. IR (KBr, cm^{-1}): 3419, 2996, 2928, 2872, 1693, 1618, 1413, 1374. ^1H NMR (400 MHz, CD_3OD, ppm): δ 5.73 (d, *J* = 5.6 Hz, 1H), 4.18 (t, *J* = 7.9 Hz, 1H), 3.55 (ddd, *J* = 11.4, 9.2, 4.1 Hz, 1H), 3.33 (s, 1H), 2.84 (d, *J* = 9.3 Hz, 1H), 2.58 (d, *J* = 7.0 Hz, 1H), 2.53-2.32 (m, 4H), 2.28 (d, *J* = 4.7 Hz, 4H), 1.99-1.82 (m, 3H), 1.78 (dt, *J* = 12.4, 4.1 Hz, 1H), 1.40 (s, 4H), 1.29 (s, 3H), 1.22 (s, 3H), 1.16 (s, 3H), 1.13 (s, 3H), 1.06 (s, 3H), 1.01 (m, 1H), 0.96 (s, 3H), 0.86 (s, 3H). ^{13}C NMR (100 MHz, CD_3OD, ppm): δ 215.5, 209.8, 180.1, 142.7, 119.8, 112.7, 90.6, 81.9, 72.8, 71.5, 71.2, 59.6, 51.5, 49.8, 49.4, 48.8, 46.5, 44.4, 43.4, 36.1, 34.9, 34.7, 29.8, 28.9, 25.3, 24.7, 22.6, 22.3, 20.5, 20.4, 19.5, 15.7.

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Figure S1 ^{1}H NMR spectrum of hemslecin C

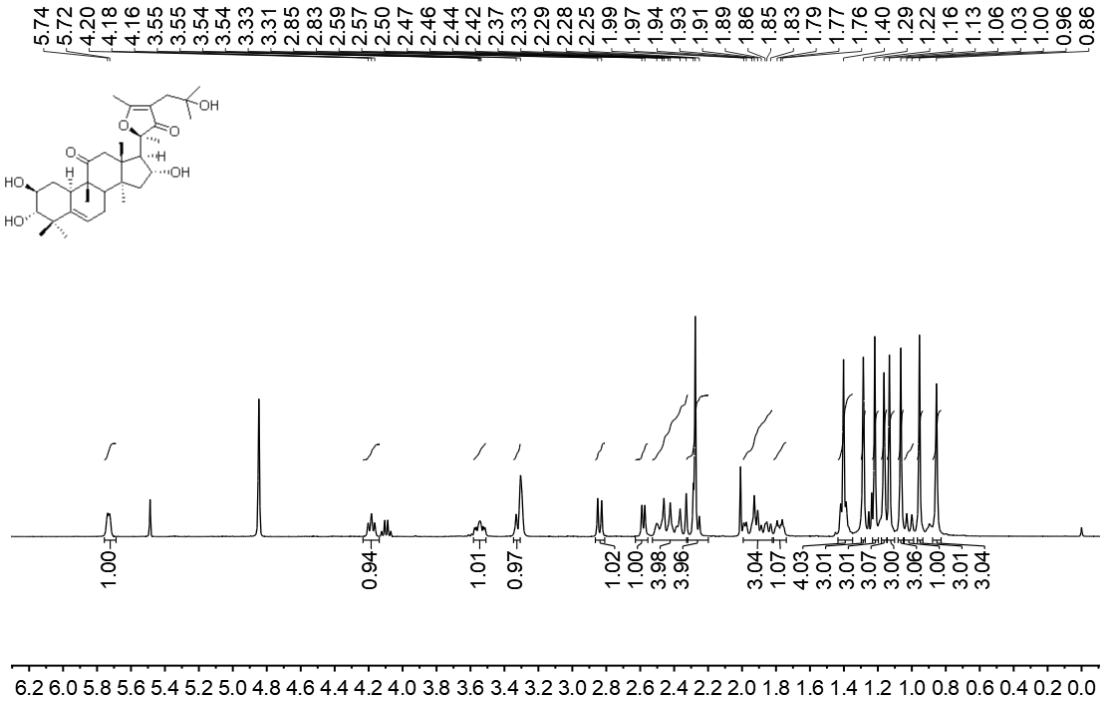


Figure S2 ^{13}C NMR spectrum of hemslecin C

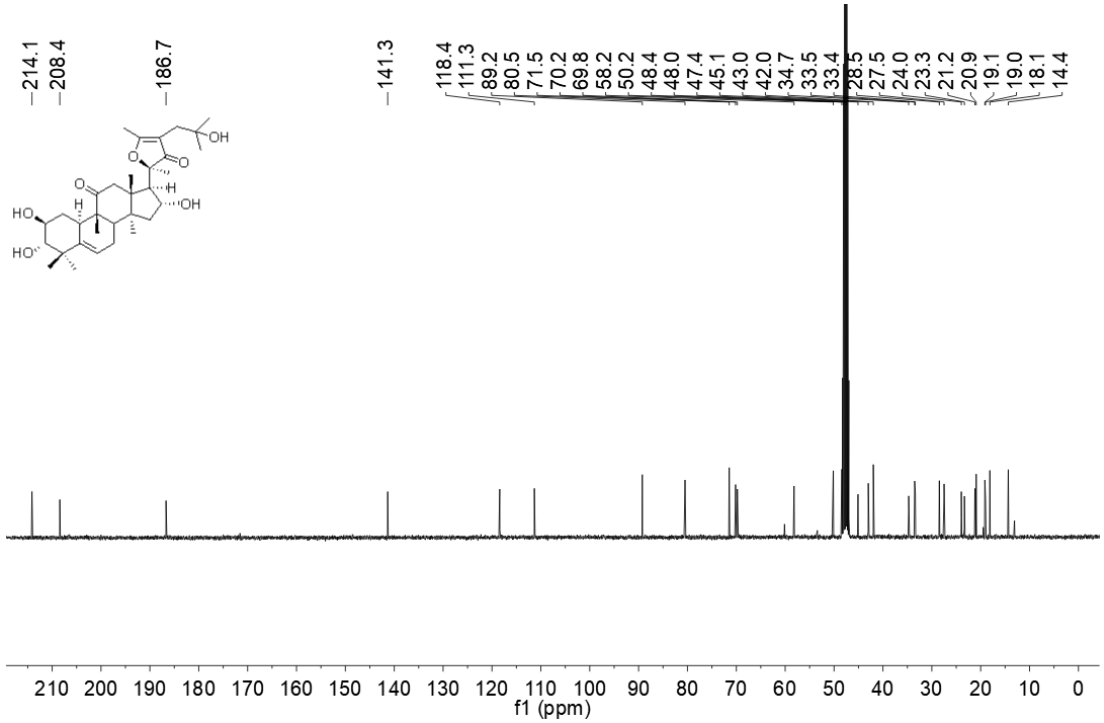


Figure S3 HSQC spectrum of hemslecin C

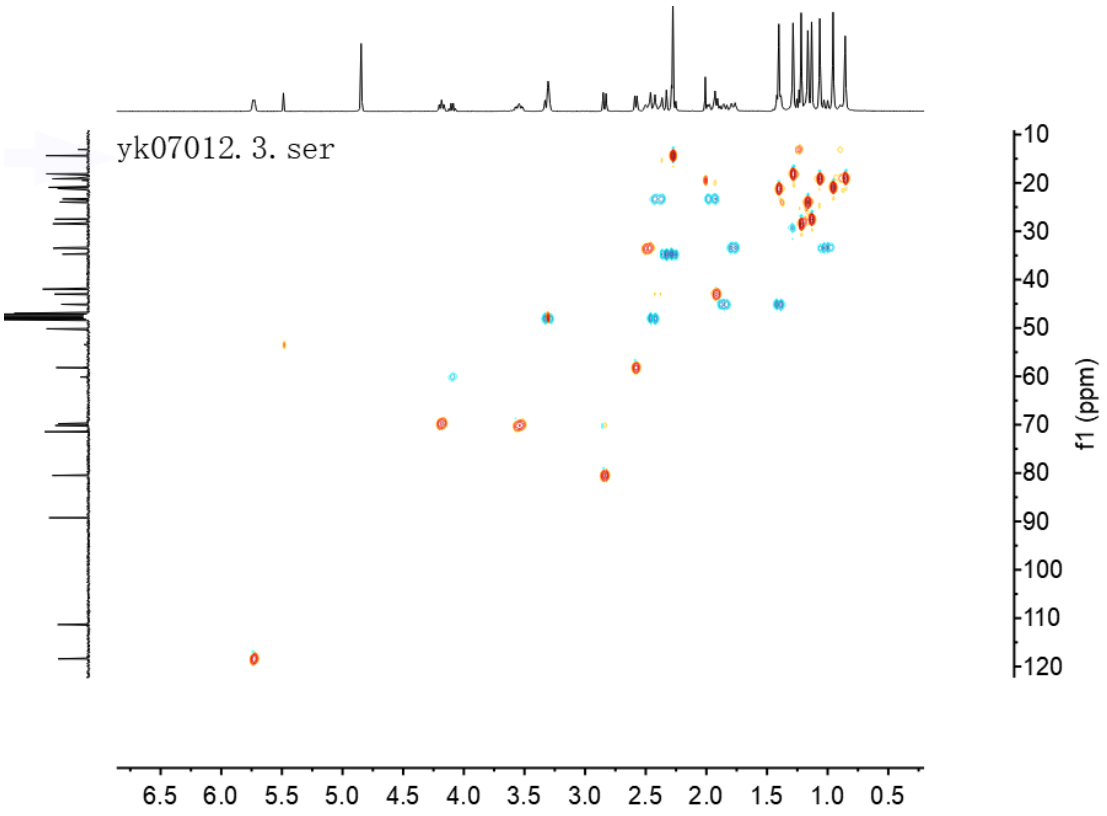


Figure S4 HRMS spectrum of hemslecin C

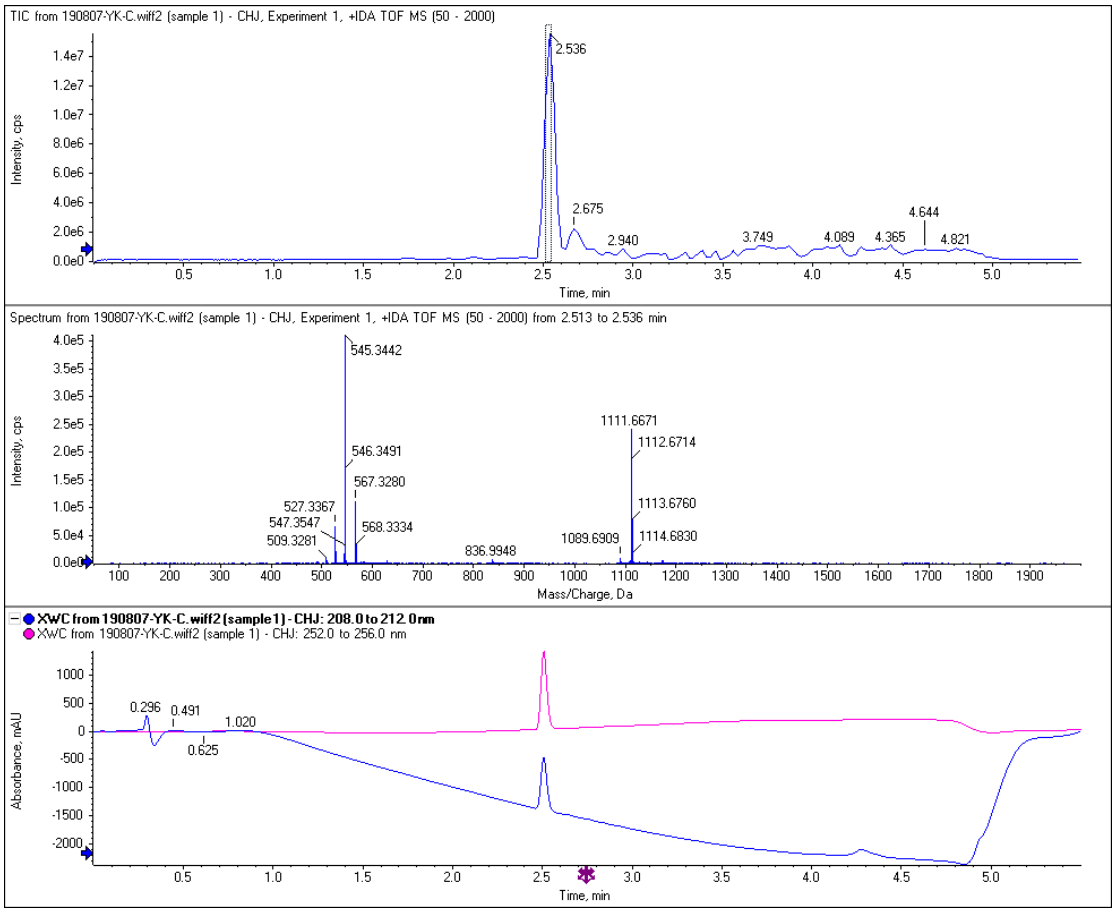


Figure S5 IR spectrum of hemslecin C

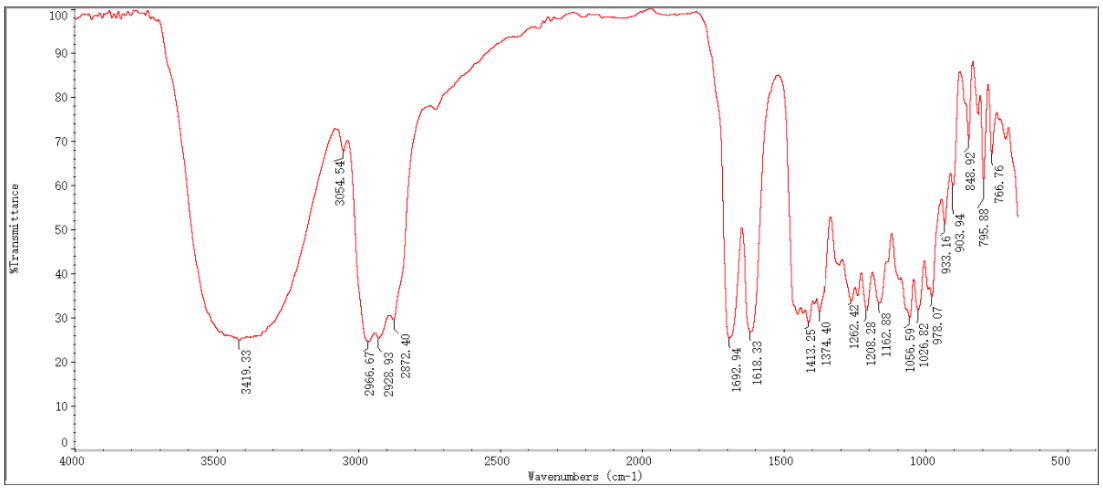
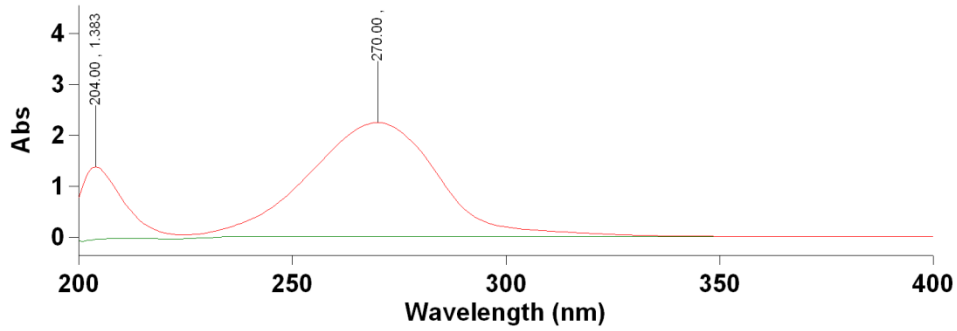


Table S1. Crystal data and structure refinement for hemslecin C.

Identification code	hemslecin C	
Empirical formula	C32 H48 O7	
Formula weight	544.70	
Temperature	193(2) K	
Wavelength	1.54178 Å	
Crystal system	Orthorhombic	
Space group	$P2_12_12_1$	
Unit cell dimensions	a = 6.4718(5) Å	□= 90°.
	b = 18.8334(15) Å	□= 90°.
	c = 27.629(2) Å	□ = 90°.
Volume	3367.6(5) $Å^3$	
Z	4	
Density (calculated)	1.074 Mg/m^3	
Absorption coefficient	0.598 mm^{-1}	
F(000)	1184	
Crystal size	0.190 x 0.160 x 0.110 mm^3	
Theta range for data collection	5.346 to 68.535°.	
Index ranges	-7<=h<=7, -22<=k<=20, -31<=l<=32	
Reflections collected	17476	
Independent reflections	5953 [R(int) = 0.0382]	
Completeness to theta = 67.679°	99.0 %	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5953 / 1 / 365	
Goodness-of-fit on F^2	1.021	
Final R indices [I>2sigma(I)]	R1 = 0.0517, wR2 = 0.1507	
R indices (all data)	R1 = 0.0550, wR2 = 0.1542	
Absolute structure parameter	1.23(11)	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.303 and -0.220 $e.Å^{-3}$	

Figure S6 UV spectrum of hemslecin C



References

1. Wang F and Liang XT. *Huaxue Xuebao* 1983; 41: 95.