

**Cytotoxic Polyhydroxysteroidal Glycosides from Starfish *Culcita novaeguineae***

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Table S1. The  $^{13}\text{C}$  NMR data of compounds echinasteroside C (**3**), linckoside F (**5**) and linckoside L3 (**6**).

Table S2. The inhibition ratio of the new compounds against glioblastoma cell lines at 24h.

Table S3. The inhibition ratio of the new compounds against glioblastoma cell lines at 48h.

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Figure S1.  $^1\text{H}$  NMR (500 MHz, in  $\text{C}_5\text{D}_5\text{N}$ ) spectrum of Culcinoside A (**1**)

Figure S2.  $^{13}\text{C}$  NMR (125 MHz, in  $\text{C}_5\text{D}_5\text{N}$ ) spectrum of Culcinoside A (**1**)

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Figure S17. NOESY spectrum of Culcinoside C (**4**)

Figure S18.  $^1\text{H}$  NMR (500 MHz, in  $\text{C}_5\text{D}_5\text{N}$ ) spectrum of Culcinoside D (**7**)

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Figure S21. HMBC spectrum of Culcinoside D (**7**)

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Figure S26.HRESIMS spectrum of Culcinoside C (4)

Figure S27.HRESIMS spectrum of Culcinoside D (7)

Table S1. The  $^{13}\text{C}$ -NMR (500 MHz) data of compounds echinasteroside C (**3**), linckoside F (**5**) and linckoside L3 (**6**) ( $\delta$  in ppm).

position	<b>3</b> <sup>a</sup>	<b>5</b> <sup>a</sup>	<b>6</b> <sup>b</sup>
1	39.5	39.1	39.8
2	28.3	28.0	28.0
3	76.8	76.4	77.6
4	126.6	126.2	127.0
5	149.5	148.6	148.6
6	76.1	75.7	76.5
7	45.0	44.6	44.5
8	76.1	75.7	76.3
9	57.8	57.4	57.9
10	37.8	37.4	37.8
11	19.7	19.3	19.6
12	43.1	42.7	43.1
13	45.2	44.8	45.2
14	64.3	63.8	63.7
15	81.3	80.9	80.6
16	85.6	82.5	83.1
17	60.9	60.4	61.1
18	17.7	17.3	16.9
19	23.2	22.8	22.9
20	30.7	30.2	31.8
21	19.1	18.6	18.8
22	37.3	35.0	30.2
23	25.1	32.8	31.7
24	35.1	153.9	77.5
25	37.3	43.4	33.9
26	68.1	67.0	17.3

27	18.1	17.5	17.6
28		108.9	66.2
2-OMe-Xyl			
1'	104.9	104.5	104.7
2'	85.6	85.2	85.0
3'	78.1	77.7	77.6
4'	71.7	71.3	71.3
5'	67.5	67.1	66.9
2-OMe	61.3	60.9	61.3

<sup>a</sup> in C<sub>5</sub>D<sub>5</sub>N. <sup>b</sup> in CD<sub>3</sub>OD.

Table S2. The inhibition ratio of the new compounds against glioblastoma cell lines at 24h.

Compounds	Inhibition ratio (%)		
	U87	U251	SHG44
1	19.32	17.71	21.27
2	10.16	8.47	9.33
4	12.14	13.35	9.82
7	7.78	11.22	11.88

The concentration of each compound was 10  $\mu$ mol/L.

Table S3. The inhibition ratio of the new compounds against glioblastoma cell lines at 48h.

Compounds	Inhibition ratio (%)		
	U87	U251	SHG44
1	54.85	48.63	55.68
2	19.91	16.44	18.75
4	26.26	28.45	19.42
7	15.81	25.57	26.14

The concentration of each compound was 10  $\mu$ mol/L.

Table S4. The inhibition ratio of the new compounds against glioblastoma cell lines at 72h.

Compounds	Inhibition ratio (%)		
	U87	U251	SHG44
1	63.42	61.38	65.88
2	25.73	21.82	22.95
4	34.26	37.13	24.35
7	20.64	33.58	34.62

The concentration of each compound was 10  $\mu$ mol/L.

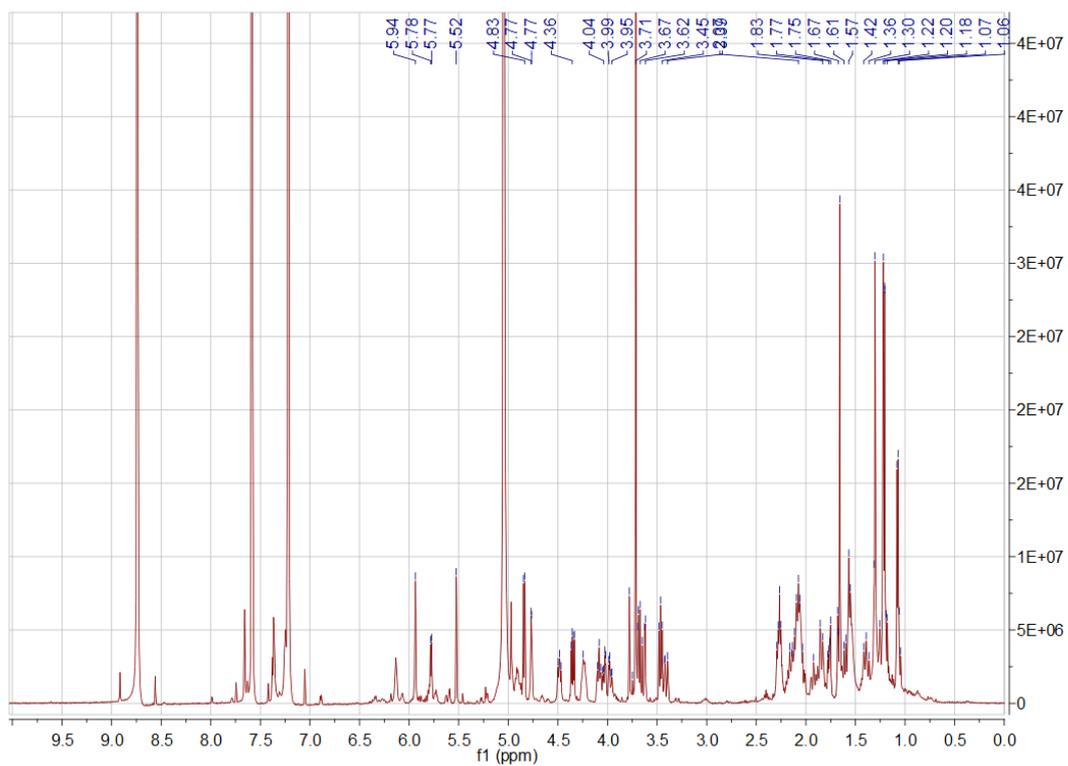


Figure S1.  $^1\text{H}$  NMR (500 MHz, in  $\text{C}_5\text{D}_5\text{N}$ ) spectrum of Culcinoside A (**1**)

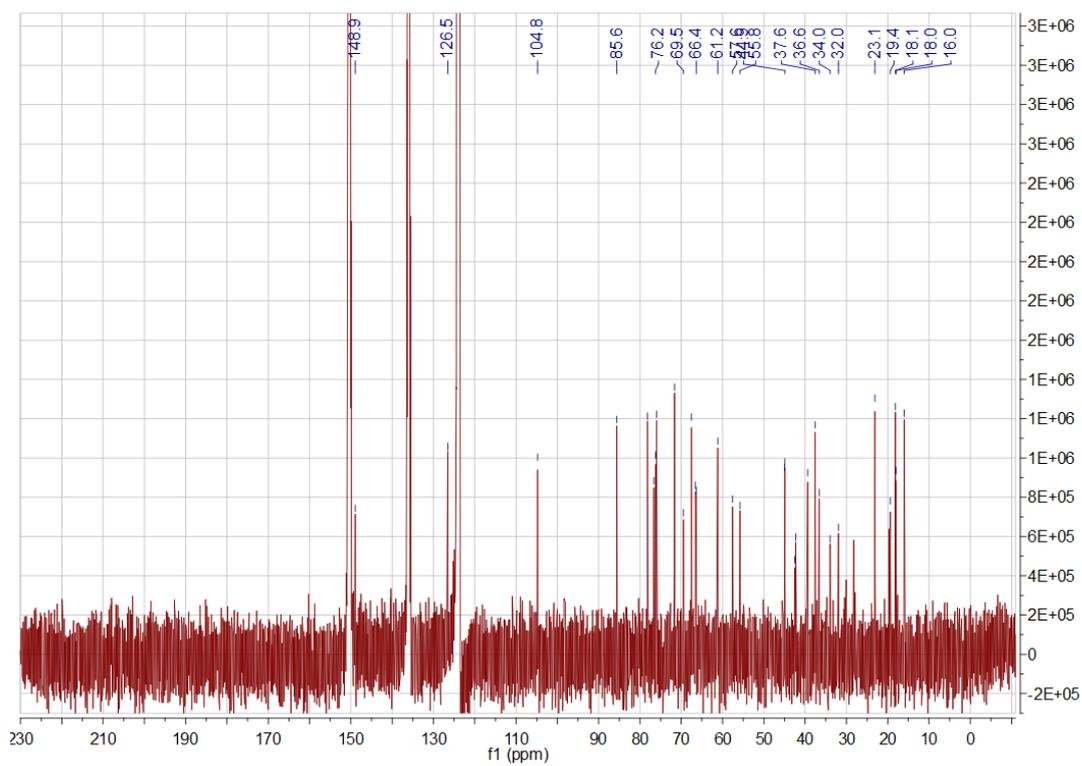


Figure S2.  $^{13}\text{C}$  NMR (125 MHz, in  $\text{C}_5\text{D}_5\text{N}$ ) spectrum of Culcinoside A (**1**)

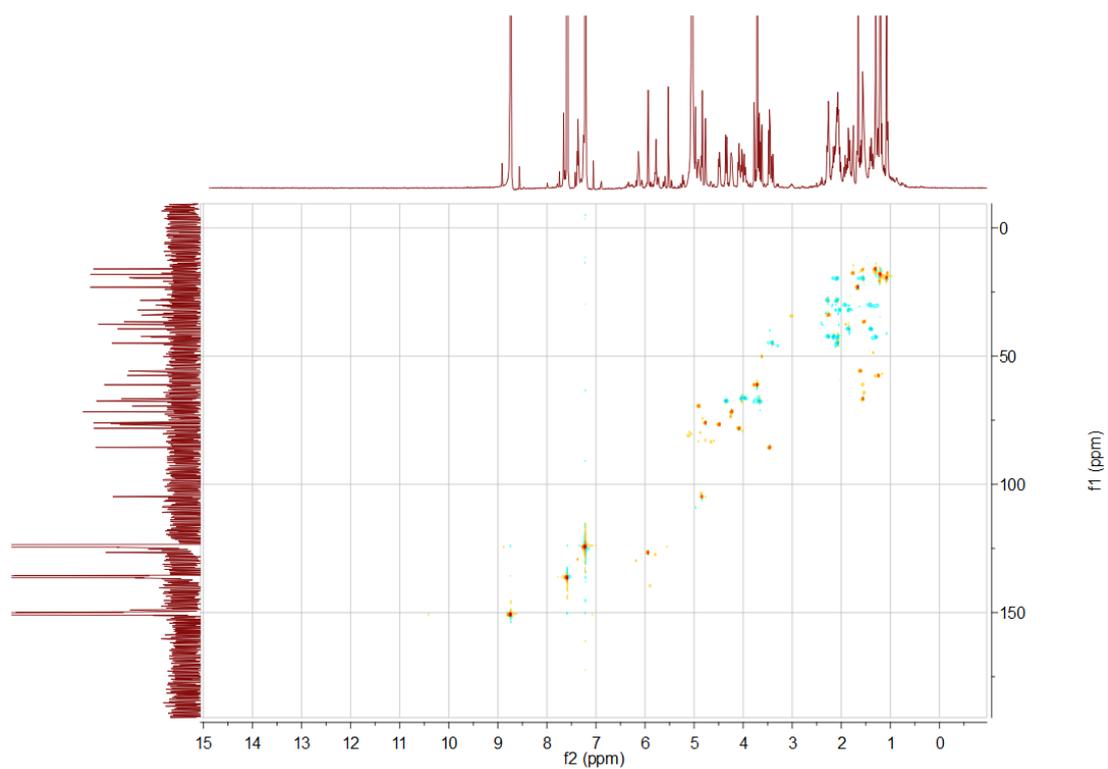


Figure S3. HSQC spectrum of Culciniside A (**1**)

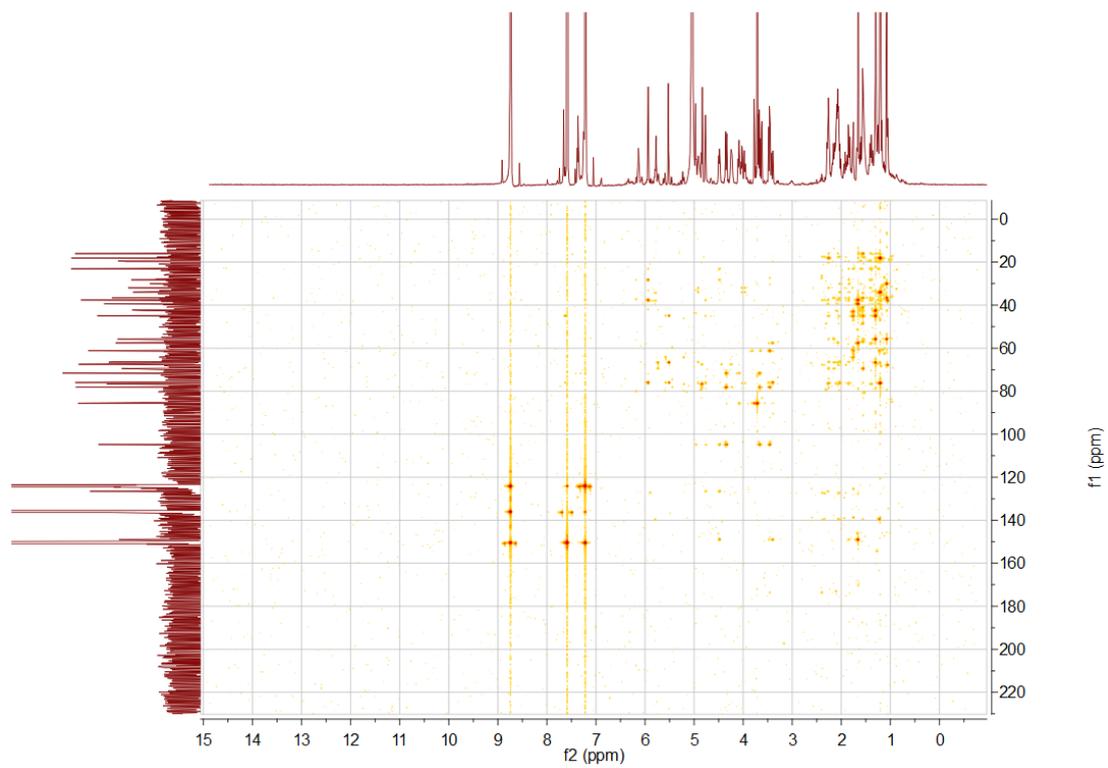


Figure S4. HMBC spectrum of Culciniside A (**1**)

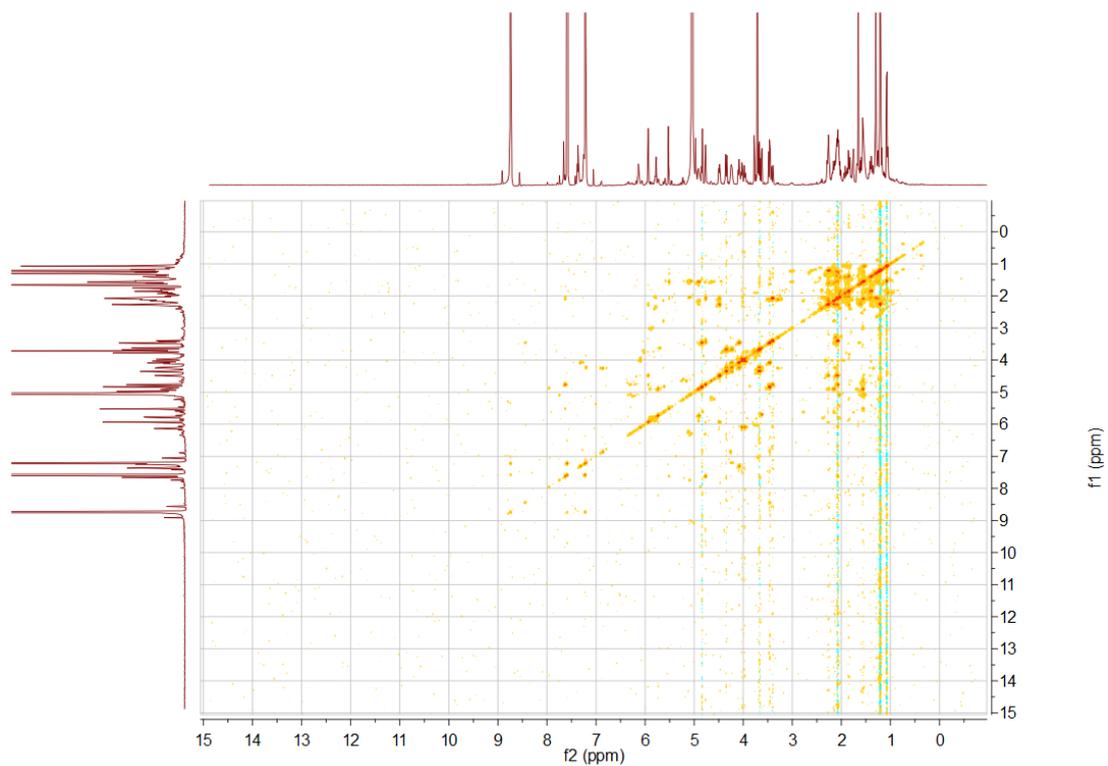


Figure S5.  $^1\text{H}$ - $^1\text{H}$  COSY spectrum of Culcinoside A (1)

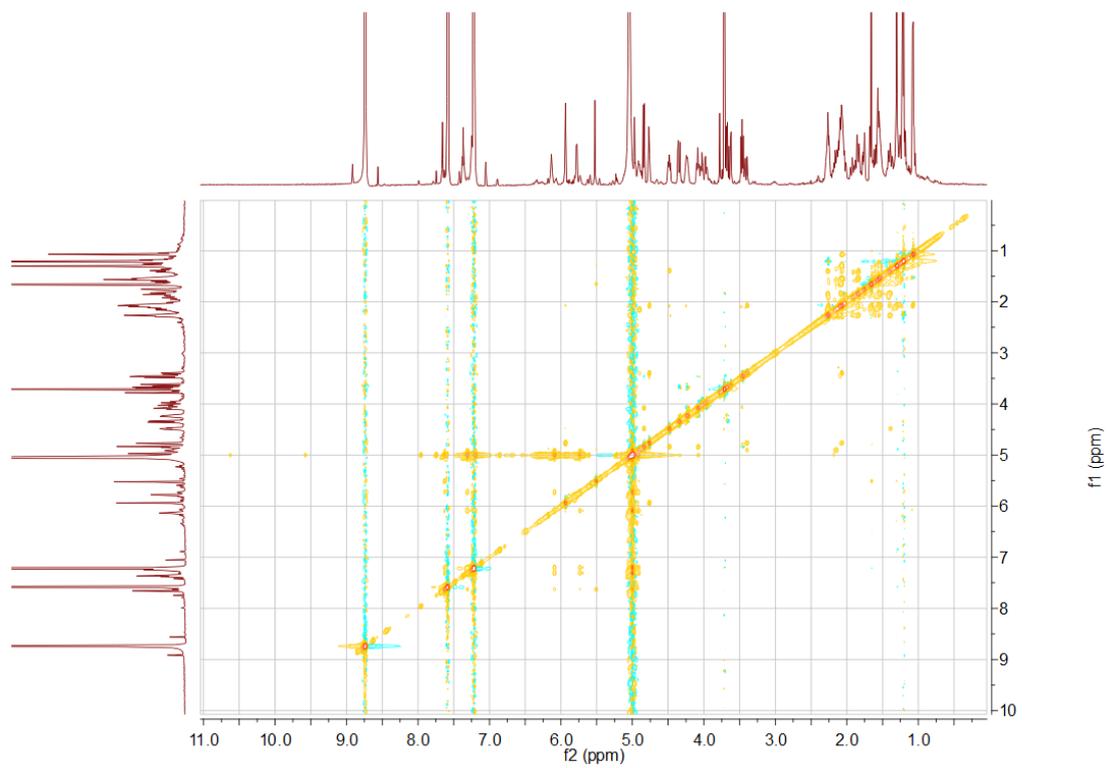


Figure S6. NOESY spectrum of Culcinoside A (1)

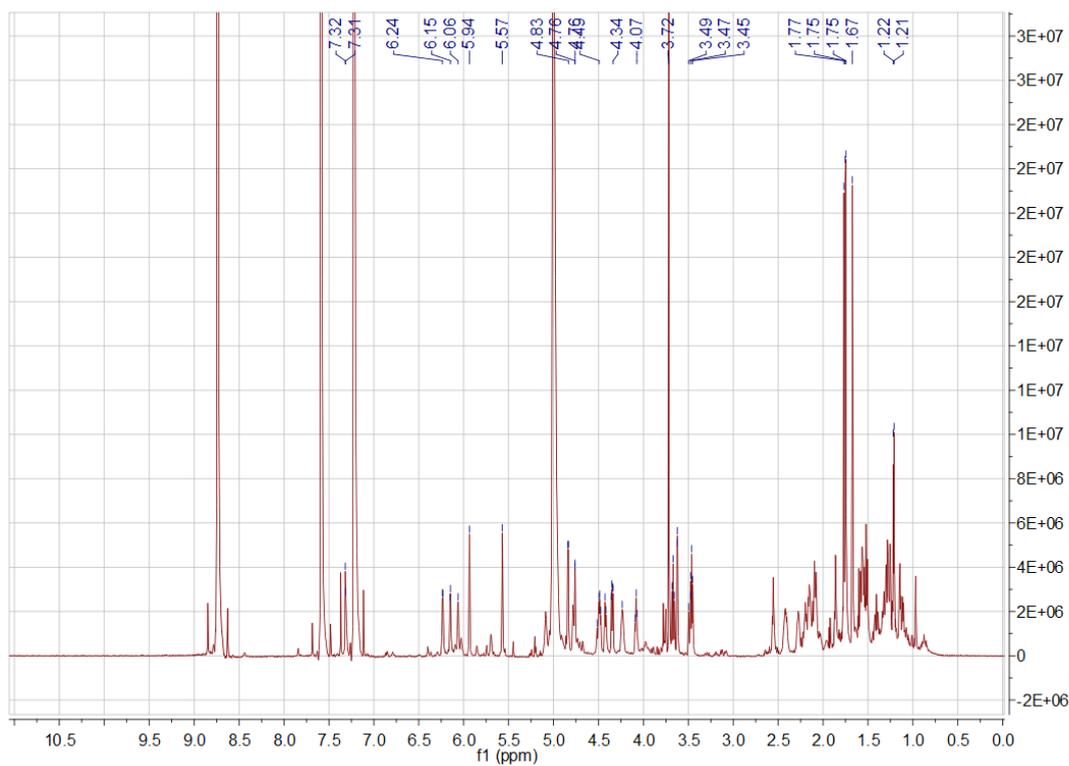


Figure S7.  $^1\text{H}$  NMR (500 MHz, in  $\text{C}_5\text{D}_5\text{N}$ ) spectrum of Culcinoside B (2)

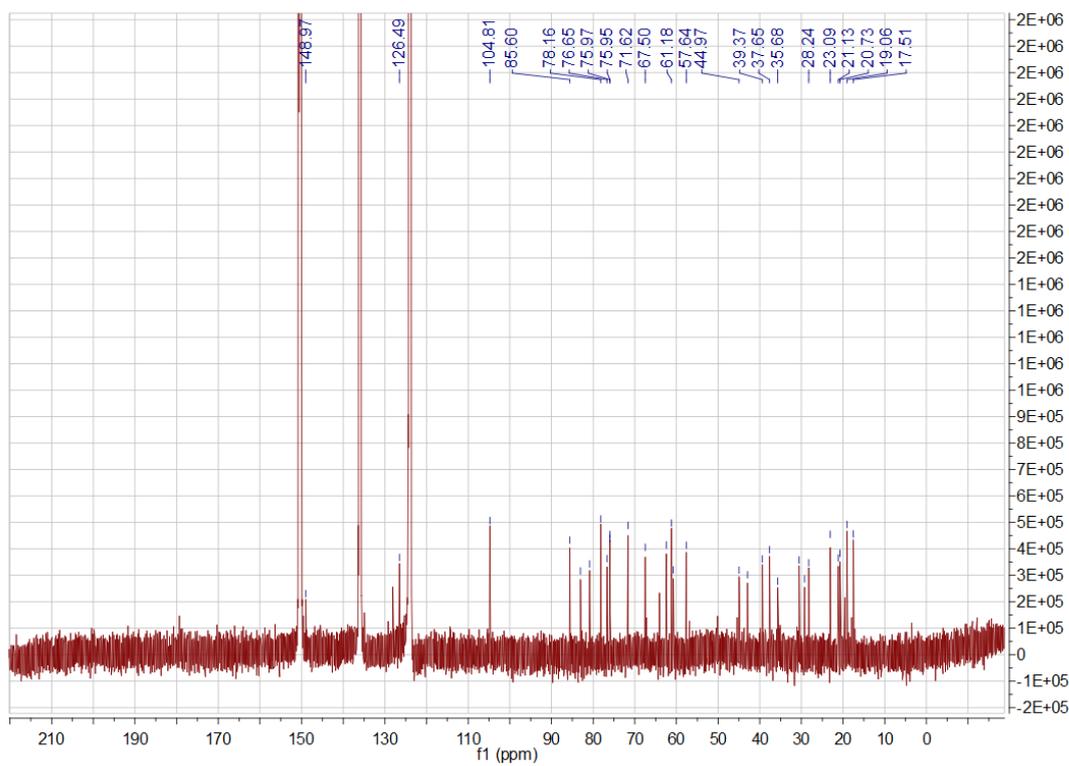


Figure S8.  $^{13}\text{C}$  NMR (125 MHz, in  $\text{C}_5\text{D}_5\text{N}$ ) spectrum of Culcinoside B (2)

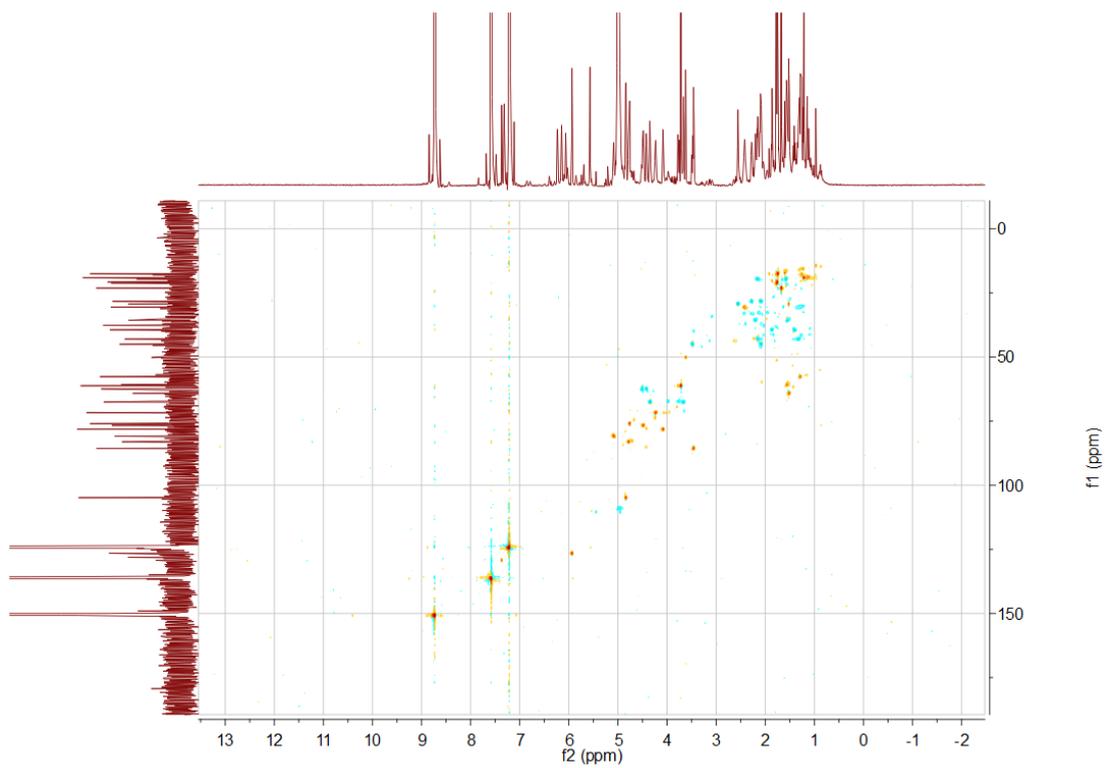


Figure S9. HSQC spectrum of Culcinoside B (2)

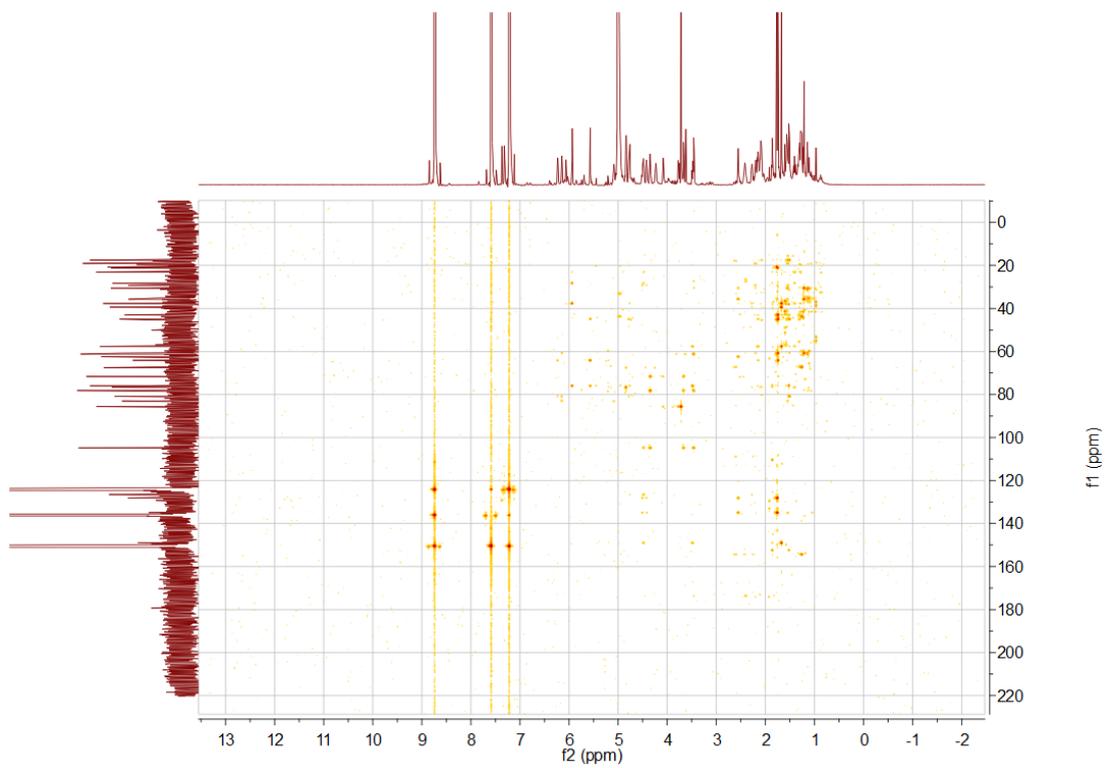


Figure S10. HMBC spectrum of Culcinoside B (2)

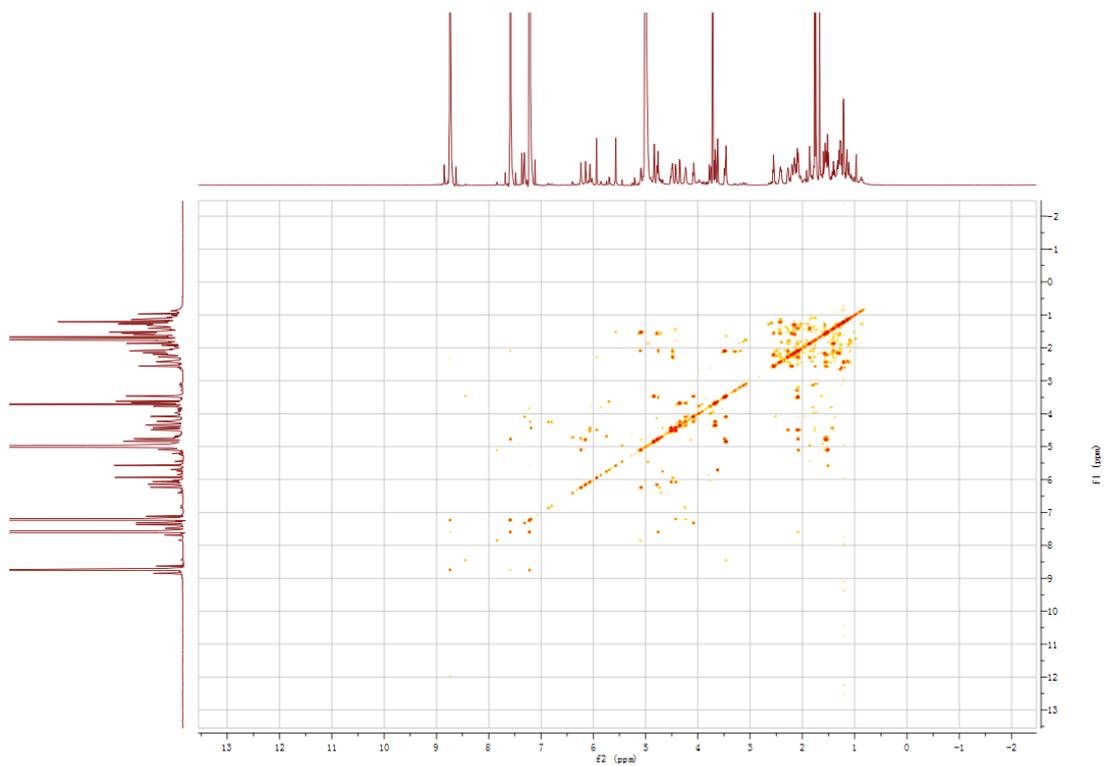


Figure S11.  $^1\text{H}$ - $^1\text{H}$  COSY spectrum of Culcinoside B (**2**)

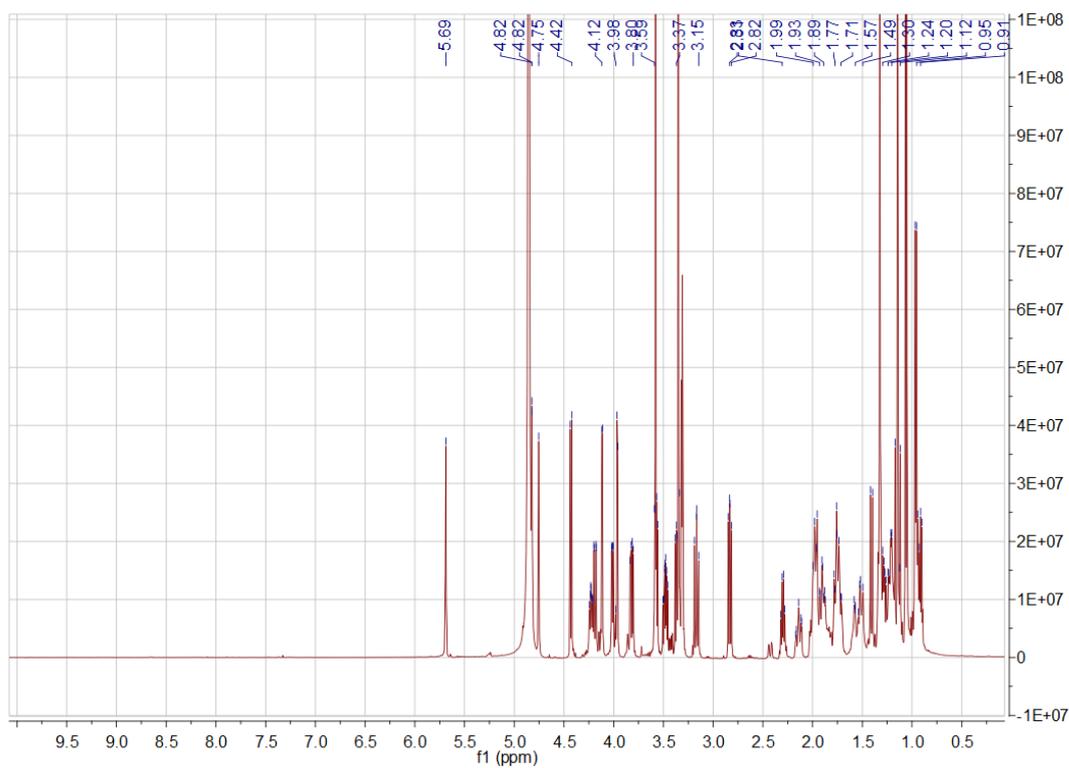


Figure S12.  $^1\text{H}$  NMR (500 MHz, in  $\text{CD}_3\text{OD}$ ) spectrum of Culcinoside C (**4**)

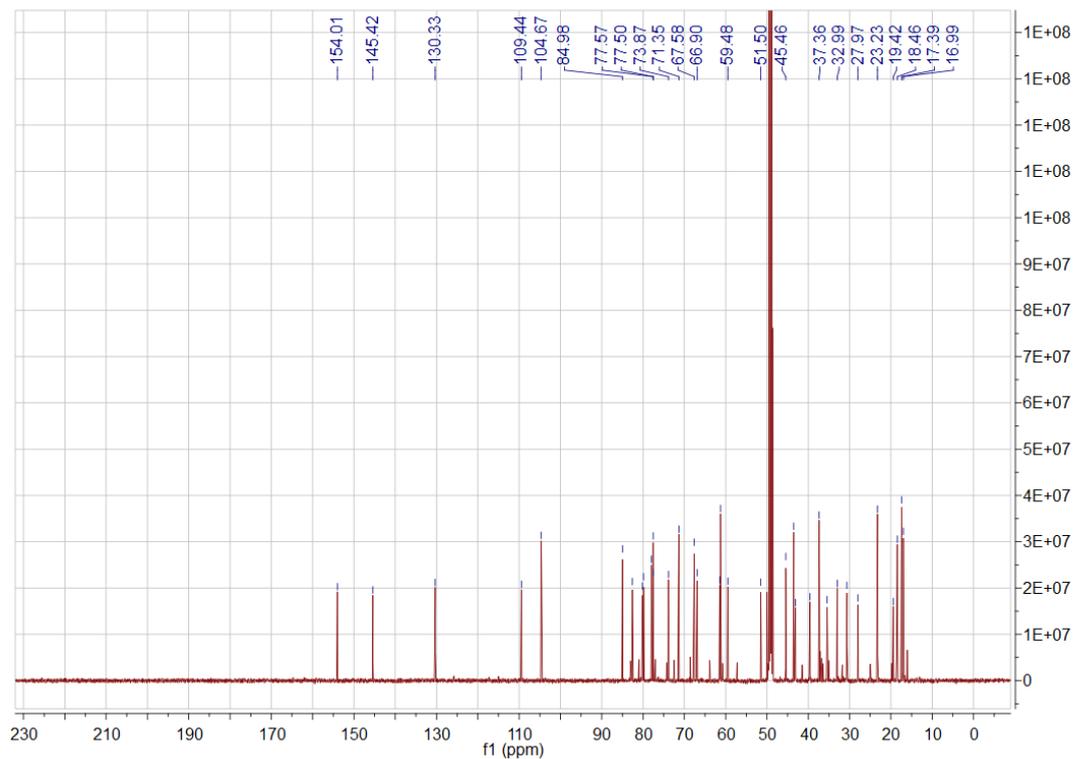


Figure S13.  $^{13}\text{C}$  NMR (125 MHz, in  $\text{CD}_3\text{OD}$ ) spectrum of Culcinoside C (**4**)

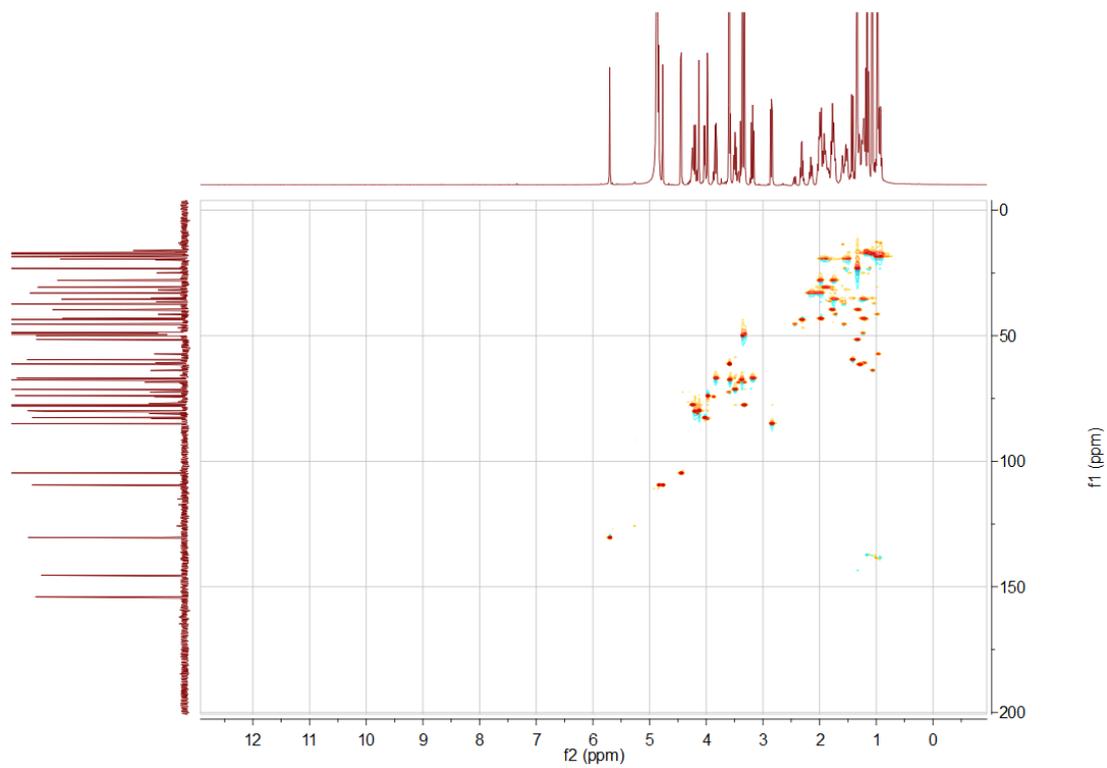


Figure S14. HSQC spectrum of Culcinoside C (**4**)

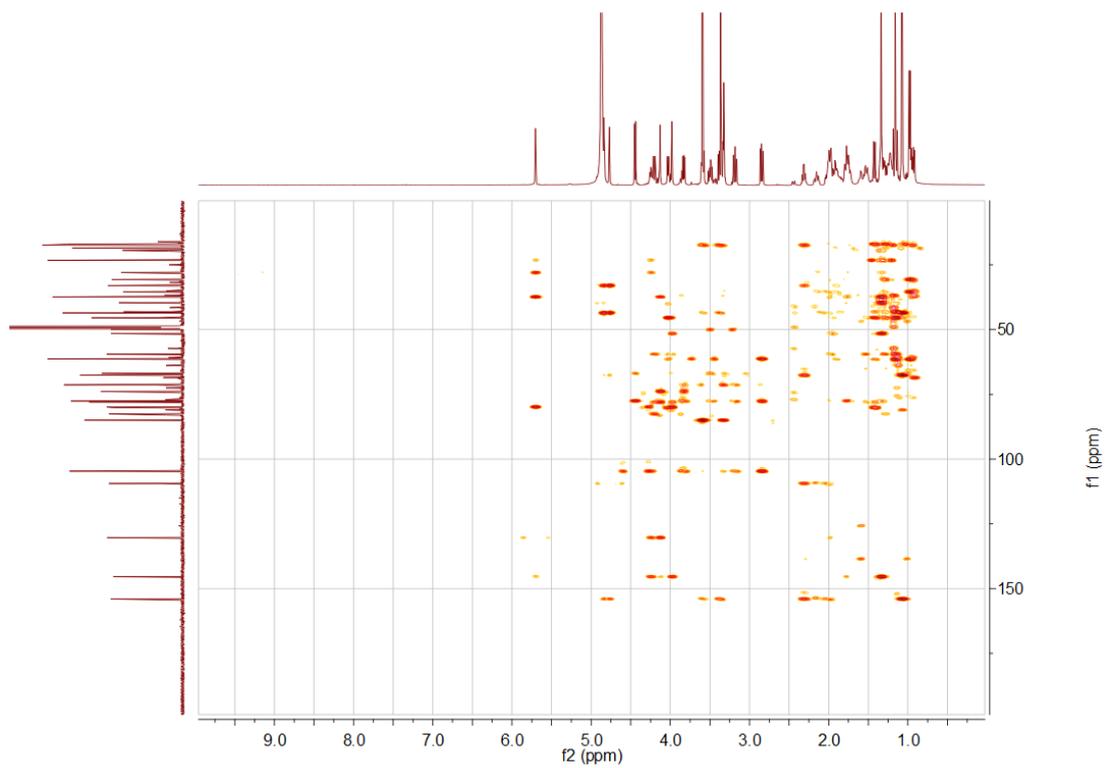


Figure S15. HMBC spectrum of Culcinoside C (4)

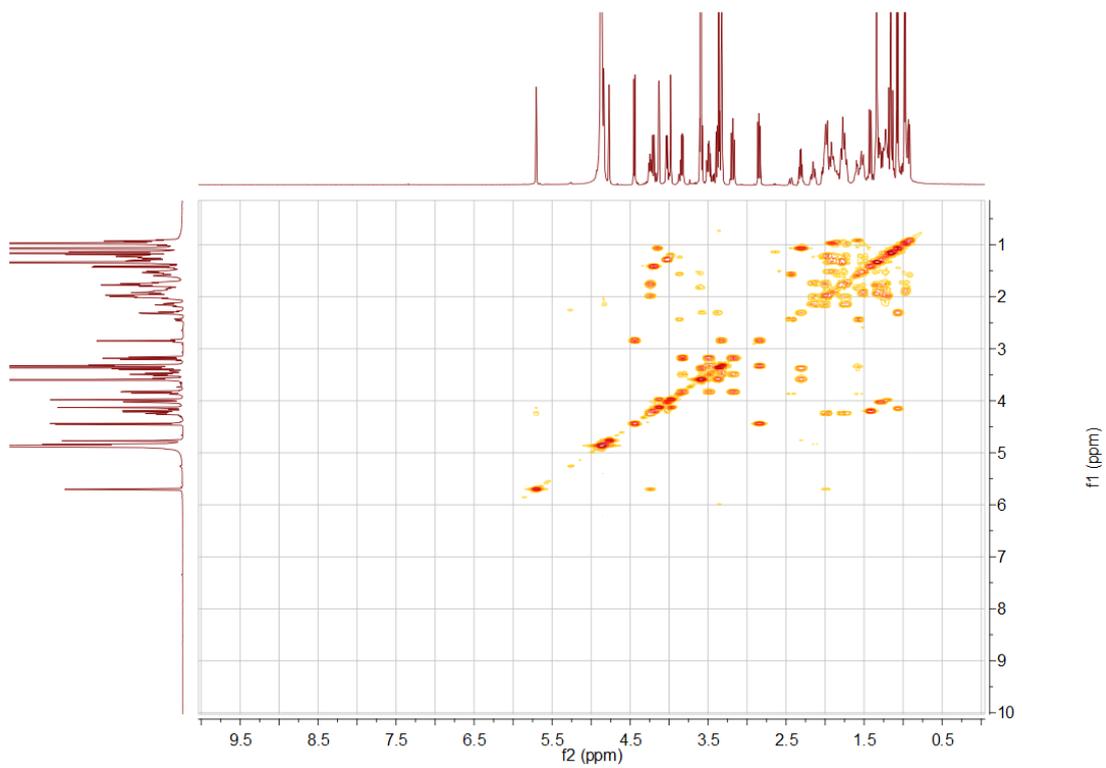


Figure S16. <sup>1</sup>H-<sup>1</sup>H COSY spectrum of Culcinoside C (4)

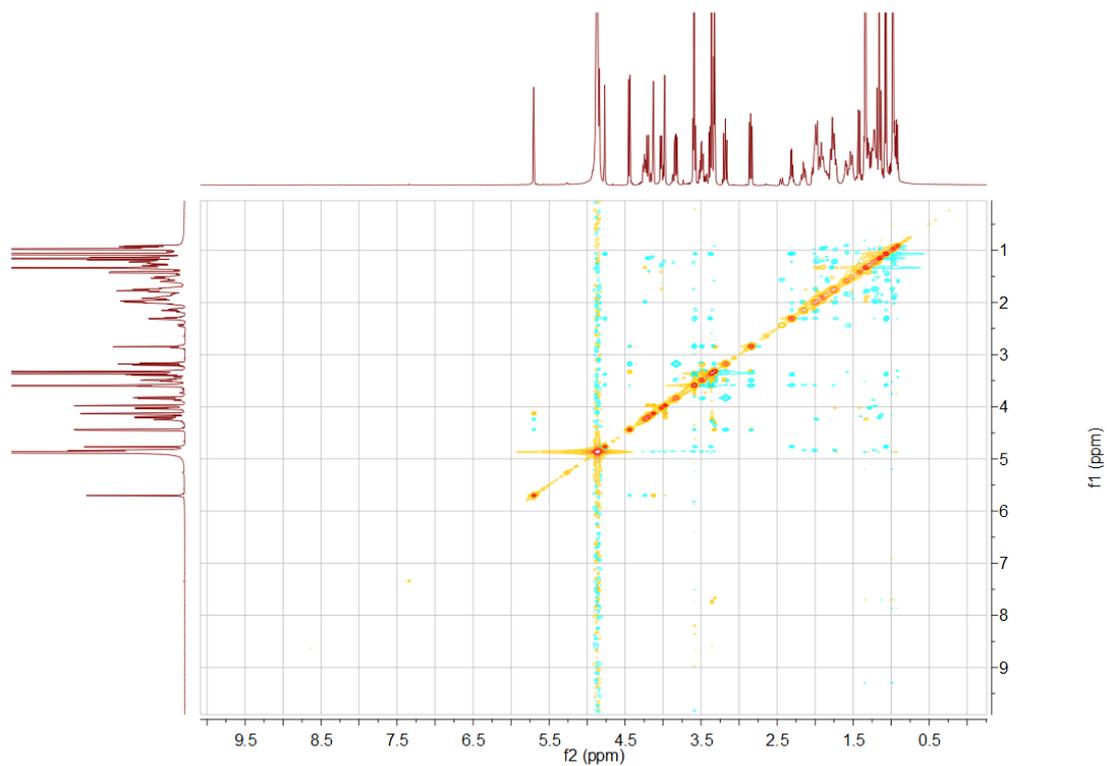


Figure S17. NOESY spectrum of Culcinoside C (**4**)

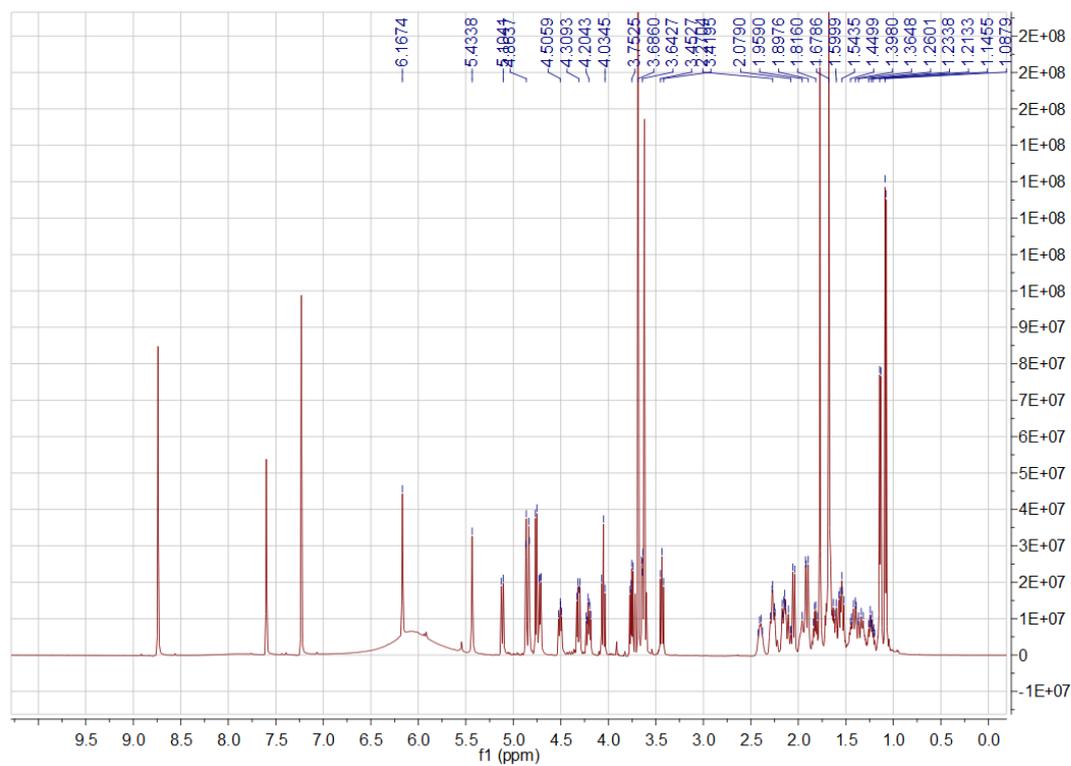


Figure S18.  $^1\text{H}$  NMR (500 MHz, in  $\text{C}_5\text{D}_5\text{N}$ ) spectrum of Culcinoside D (**7**)

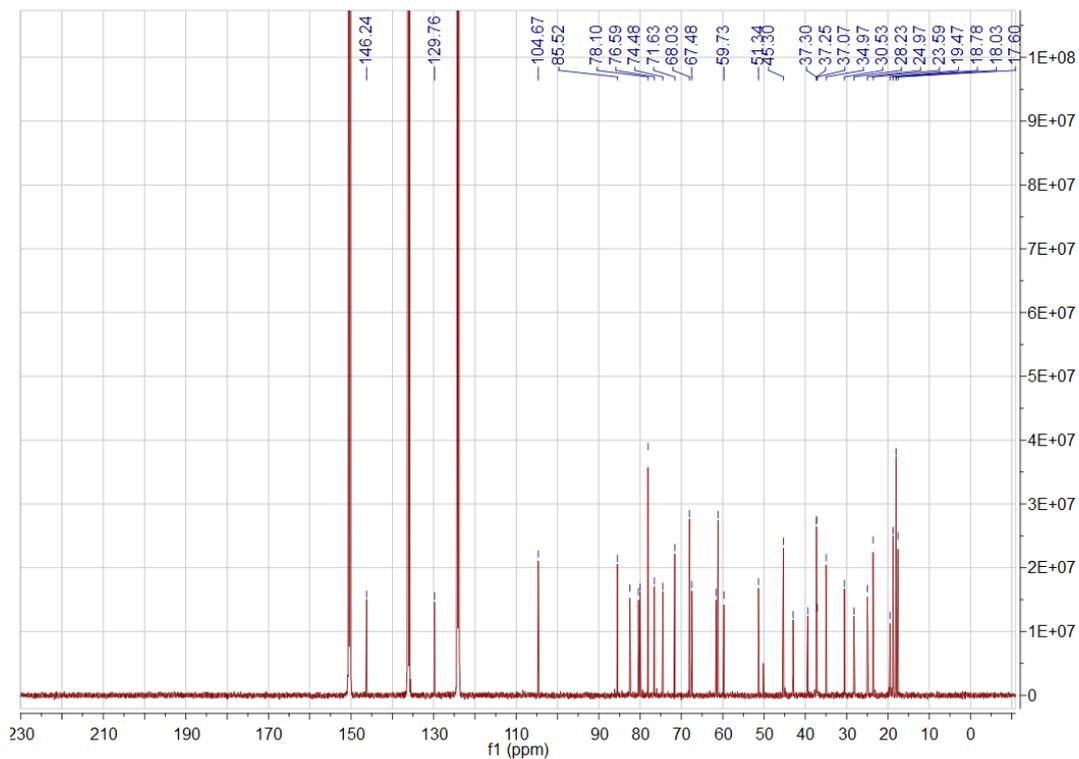


Figure S19.  $^{13}\text{C}$  NMR (125 MHz, in  $\text{C}_5\text{D}_5\text{N}$ ) spectrum of Culcinoside D (7)

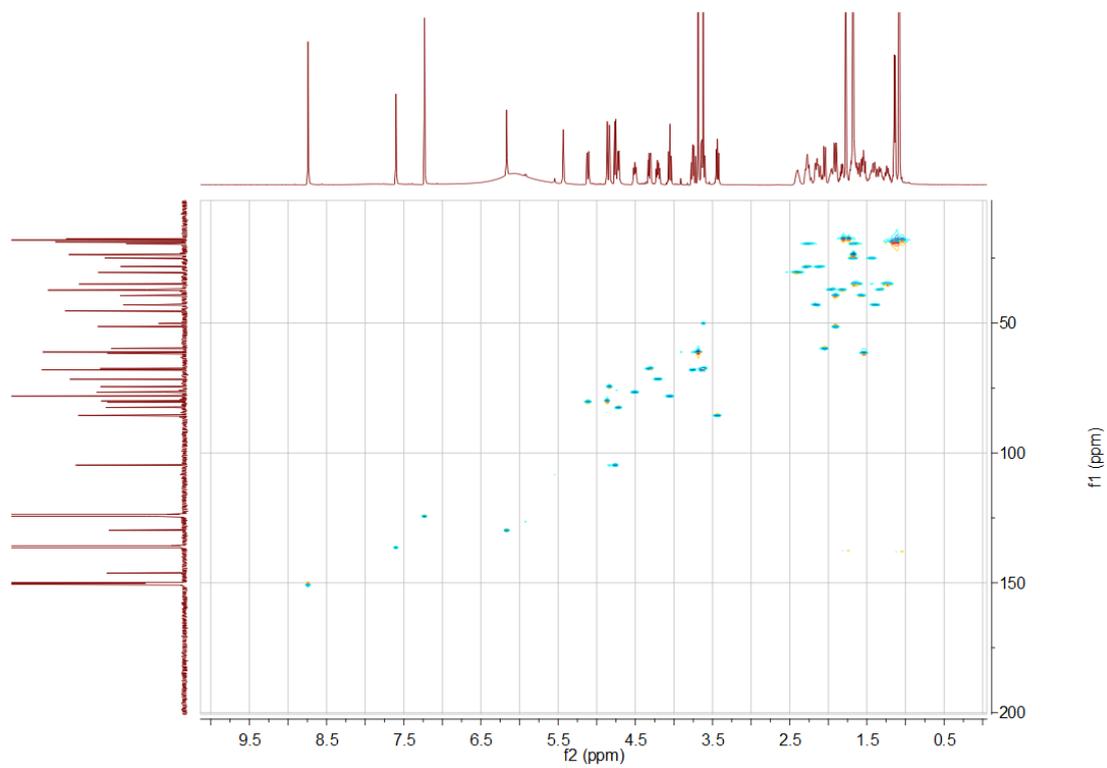


Figure S20. HSQC spectrum of Culcinoside D (7)

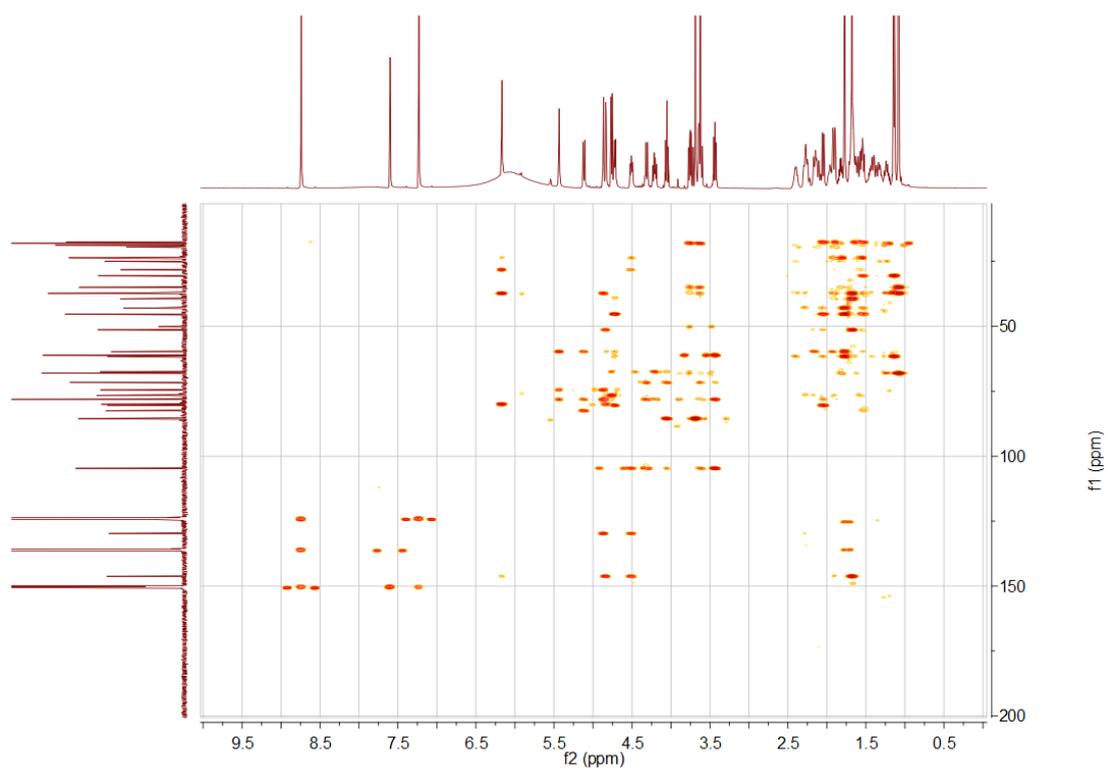


Figure S21. HMBC spectrum of Culcinoside D (7)

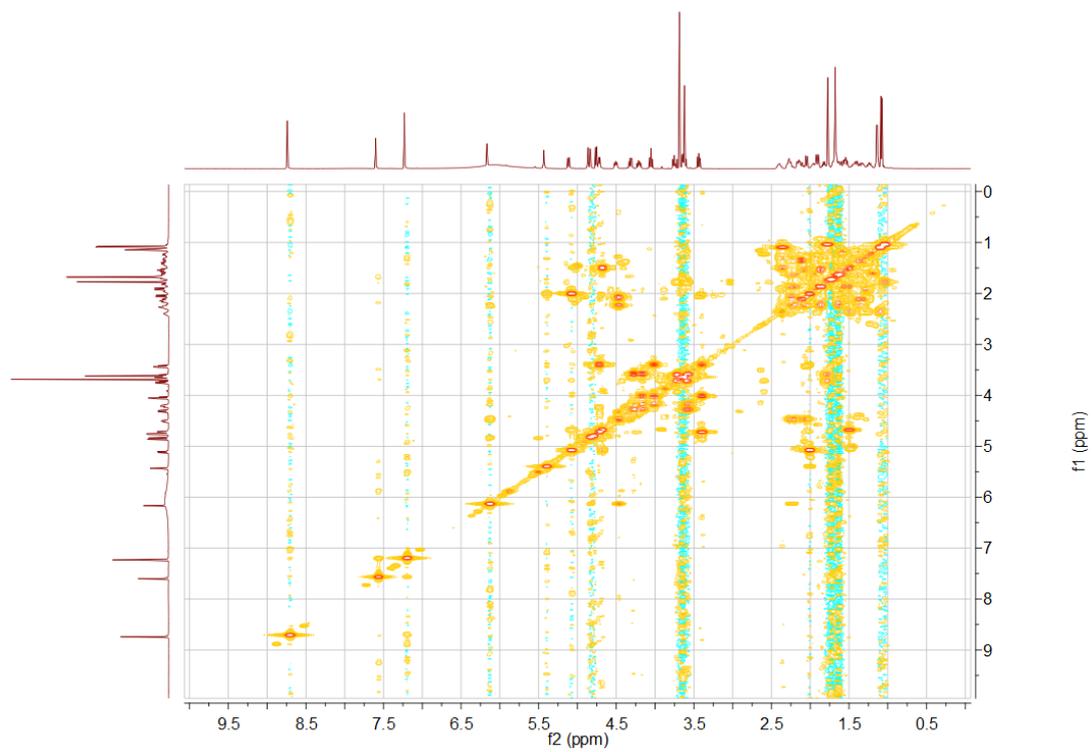


Figure S22.  $^1\text{H}$ - $^1\text{H}$  COSY spectrum of Culcinoside D (7)

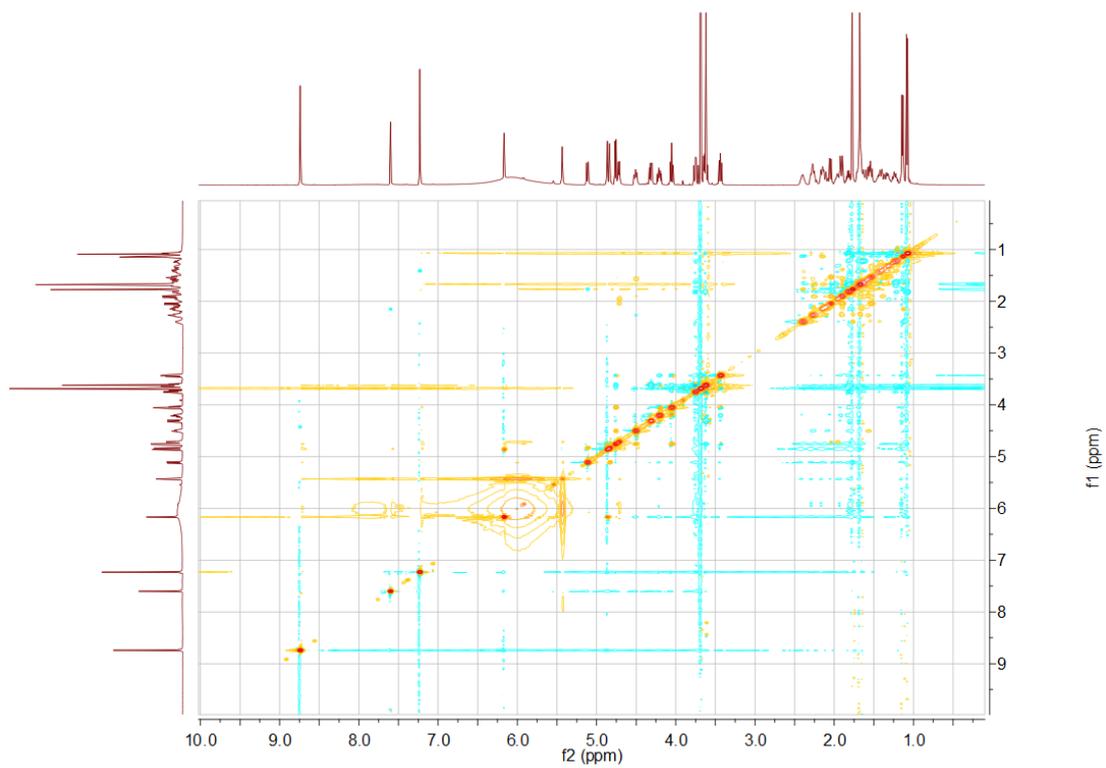


Figure S23. NOESY spectrum of Culcinoside D (7)

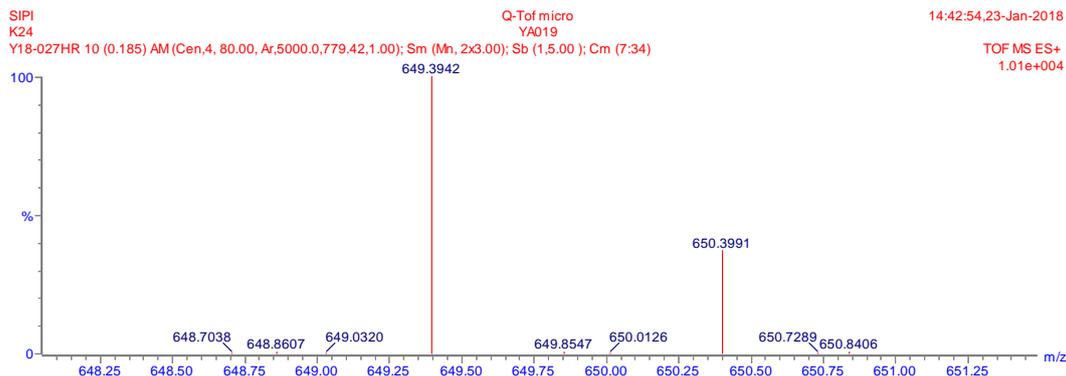


Figure S24.HRESIMS spectrum of Culcinocide A (1)

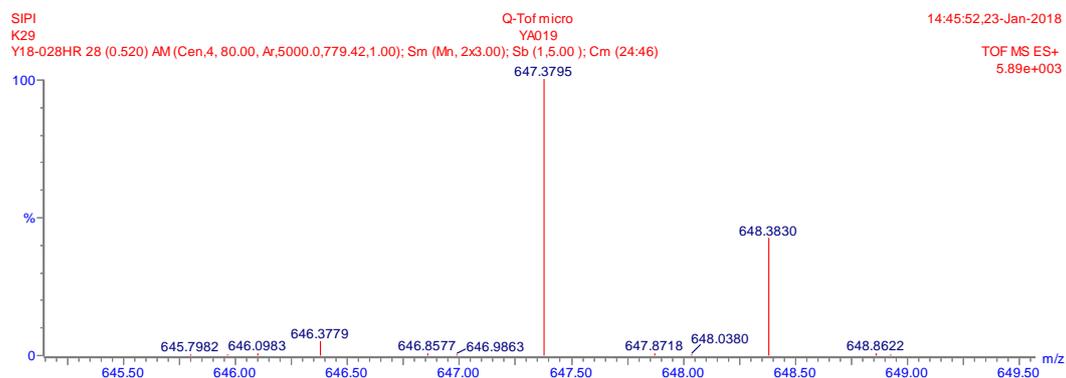


Figure S25. HRESIMS spectrum of Culcinocide B (2)

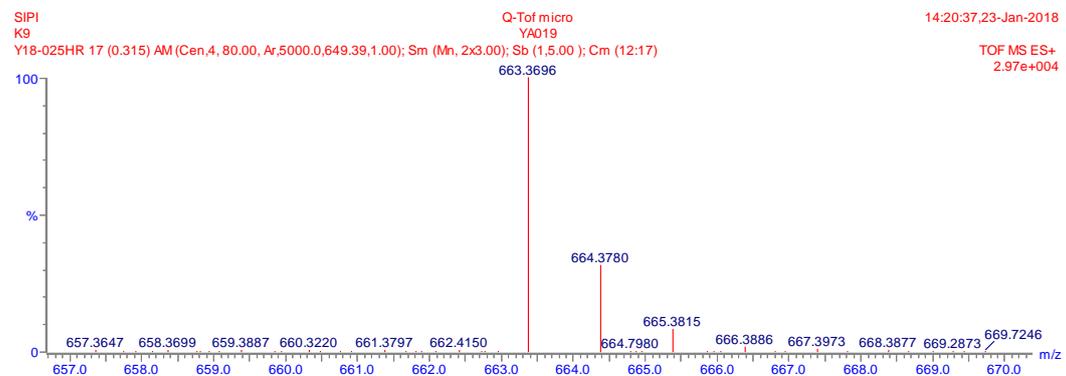


Figure S26.HRESIMS spectrum of Culcinocide C (4)

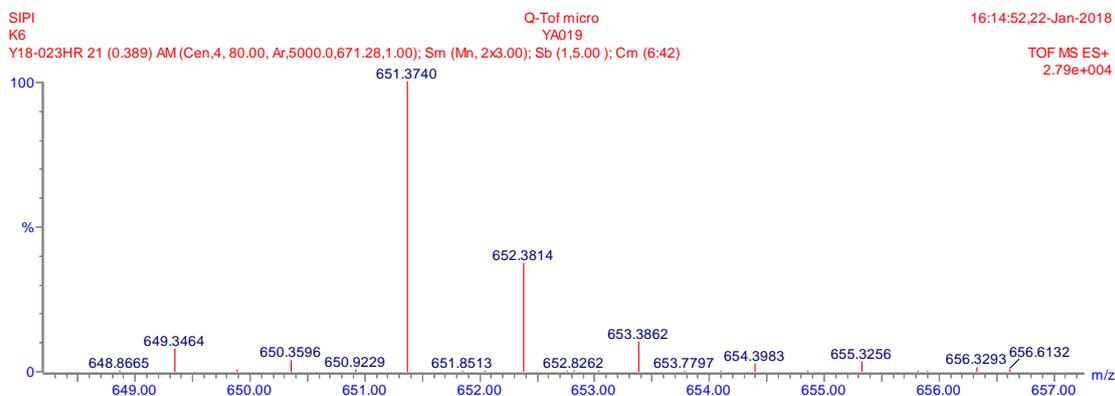


Figure S27.HRESIMS spectrum of Culcinocide D (7)