

Supporting Information

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Biological Activity and Chemical Composition of the Endophytic Fungus *Fusarium* sp. TP-G1 Obtained from the Root of *Dendrobium officinale* Kimura et Migo

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S1. Identification of strain *Fusarium* sp. TP-G1 by 18S rDNA sequence analysis:

1 gtggcctcga ctacctccc aaccctgtg acataccta ctgtgcctc ggcggatcag
61 cccgctccc gtaaacggg acggcccc agaggacccc taaactctgt ttctatatgt
121 aactctgag taaaccata aataaatcaa aacttcaac aacggatctc ttggttctgg
181 catcgatgaa gaacgcagca aatgcgata agtaatgtga attgcagaat tcagtgaatc
241 atcgaatctt tgaacgcaca ttgcgcccc cagtattctg gcgggcatgc ctgttcgagc
301 gtcattcaa ccccaagcc ctcgggttg gtgttgggga tcggcgagcc attctggcaa
361 gccggcccc aatctagtg gcggtctcgc tgcagcctcc attgcgtagt agtaaaaccc
421 tcgcaactgg aacggcgcc ggccaagccg ttaaaccccc aacttctgaa tgttgacctc
481 ggatcaggtg ggaataccc ctgaacttaa gcatatcaat aaggcggagg aa

Table S1. Summary of ¹H (600 MHz) and ¹³C (150 MHz) NMR spectroscopic data for compound **1**.

position	1 ^a		trichosetin (<i>J. Antibiot.</i> ¹)	
	δ_C	δ_H mult. (<i>J</i> in Hz)	δ_C	δ_H mult. (<i>J</i> in Hz)
1	201.1, C ^b		200.6	
2	49.6, C		49.0	
3	45.3, CH	3.36 (1H, brs)	45.0	3.34 (1H, brd, 9.0)
4	127.4, CH	5.38 (1H, brs)	126.6	5.40 (**1H, brs)
5	131.0, CH	5.38 (1H, brs)	130.0	5.41 (**1H, brs)
6	38.7, CH	1.81 (1H, m)	38.5	1.85 (1H, brt, 12.0)
7	42.4, CH ₂	0.86 (1H, m); 1.81 (1H, m)	42.1	0.90 (1H, q, 12.0), 1.85 (1H, brd, 12.0)
8	33.7, CH	1.49 (1H, brs)	33.4	1.52 (1H, m)
9	35.9, CH ₂	1.09 (1H, m); 1.72 (1H, m)	35.6	1.11 (1H, q, 12.0), 1.76 (1H, brd, *12.0)
10	28.4, CH ₂	1.02 (1H, m); 1.92 (1H, brd, 9.6)	28.3	1.05 (1H, q, *12.0), 1.96 (1H, brd, 12.0)
11	40.2, CH	1.66 (1H, brs)	40.0	1.68 (1H, brt, *12.0)
12	14.3, CH ₃	1.43 (3H, s)	13.9	1.46 (3H, s)
13	130.3, CH	5.14 (1H, m)	130.8	5.20 (1H, dd, 15.0, 9.0)
14	126.6, CH	5.25 (1H, m)	127.2	5.27 (1H, dq, 15.0, 6.5)
15	18.1, CH ₃	1.51 (3H, brs)	17.9	1.55 (3H, d, 6.5)
16	22.7, CH ₃	0.89 (3H, d, 10.2)	22.4	0.92 (3H, d, 6.5)
1'		6.99 (1H, brs)		6.44 (1H, brs)
2'	179.2, C ^b		179.1	
3'	100.5, C ^b		99.9	
4'	191.3, C ^b		190.6	
5'	62.8, CH	3.78 (1H, brs)	62.2	3.89 (1H, dd, 5.0, 3.5)
6'	62.8, CH ₂	3.90 (2H, d, 15)	62.9	3.82 (1H, dd, 12.0, 3.5), 3.90 (1H, dd, 12.0, 3.5)

^a Recorded in CDCl₃; ^b Not detected; *Partially obscured; **overlapping.

[1] J. Inokoshi, N. Shigeta, T. Fukuda, R. Uchida, K. Nonaka, R. Masuma, and H. Tomoda (2013). Epi-trichosetin, a new undecaprenyl pyrophosphate synthase inhibitor, produced by *Fusarium oxysporum* FKI-4553, *J. Antibiot.* **66(9)**, 549-554.

Table S2. Summary of ¹H (600 MHz) and ¹³C (150 MHz) NMR spectroscopic data for compound **2**.

position	2 ^a		Beauvericin (<i>Tetrahedron. Lett.</i> ²)	
	δ_C	δ_H mult. (<i>J</i> in Hz)	δ_C	δ_H mult. (<i>J</i> in Hz)
Hiv ^c	3units		3units	
1 C=O	169.9*3		169.3*3	
2	75.8*3	4.86 (3H, d, 8.4)	75.4*3	4.91(3H, d, 8.6)
3	29.9*3	1.96 (3H, m)	29.7*3	2.0 (3H, m)
4	17.4*3	0.38 (9H, d, 6.6)	17.4*3	0.42 (9H, d, 6.8)
4'	18.3*3	0.77 (9H, d, 6)	18.3*3	0.79 (9H, d, 6.7)
NMePHe	3units		3units	
1 C=O	170.2*3		169.9*3	
2	57.3*3	5.54 (3H, d, 7.8)	57.3*3	5.45 (3H, dd, 4.1,11.1)
3	34.9*3	3.36 (3H, dd, 4.2, 14.4)	34.7*3	3.34(3H, dd, 5.0,14.6),2.97(3H, dd, 11.8,14.6)
4	136.7*3	2.94 (3H, t, 13.8)	136.6*3	
5,9	128.7*3	7.15-7.25 (15H, m)	128.8*3	7.14-7.26 (15H, m)
6,8	129.0*3		128.5*3	
7	127.0*3		126.7*3	
NCH ₃	32.3*3	2.99 (9H, s)	32.3*3	2.98 (9H, s)

^a Recorded in CDCl₃; ^c D-2-hydroxyisovaleric acid (Hiv)

[2] A.R.B. Ola, A.H. Aly, W.H. Lin, V. Wray, and A. Debbab (2014). Structural revision and absolute configuration of lateritin, *Tetrahedron. Lett.* **55**(45), 6184-6187.

Table S3. Summary of ¹H (600 MHz) and ¹³C (150 MHz) NMR spectroscopic data for compound **3**.

position	3 ^a		beauvericin A (<i>J. Nat. Prod.</i> ³)	
	δ_C	δ_H mult. (<i>J</i> in Hz)	δ_C	δ_H mult. (<i>J</i> in Hz)
Hiv ^c	2units			
1 C=O	169.8*2		169.2*2	
2	75.8*2	4.91 (1H, d, 9.6) 4.93 (1H, d, 9.6)	75.4*2	4.93 (1H,d,6.2) 4.96 (1H,d,6.5)
3	29.9, 30.0	2.96-3.01 (2H, m)	29.7*2	2.04 (2H,m)
4	17.6, 17.7	0.43 (3H, d, 7.8), 0.45 (3H, d, 7.2)	17.5*2	0.43(3H,d,6.4),0.45(3H,d,6.5)
4'	18.5*2	0.78 (3H, d, 7.2), 0.80 (3H, d, 7.8)	18.3*2	0.76(3H,d,6.6),0.77(3H,d,6.8)
Hmp ^d	1units		1units	
1 C=O	169.8		169.2	
2	74.6	5.02 (1H, d, 7.8)	74.2	5.04 (1H,d,7.7)
3	36.1	1.75 (1H, m)	35.8	1.78 (1H,m)
4	24.6	2.02 (2H, m)	24.5	0.70 (2H,m)
5	11.6	0.68 (3H, d, 6.0)	11.3	0.68 (3H,m)
6	14.6	0.82 (3H, d, 6.8)	14.3	0.81(3H,d,6.8)
NMePHe	3units		3units	
1 C=O	170.2*3		169.9 *3	
2	57.3, 57.4*2	5.55 (3H, m)	57.4, 57.5*2	5.43 (3H,m)
3	34.8, 34.9, 35.0	3.40 (3H, m)	34.7,34.8*2	3.35 (3H,),2.99 (3H,m)
4	136.8*3	7.19-7.30 (15H, m)	136.6*3	7.22 (15H,m)
5,9	128.8*3, 128.7*3		128.5*6	
6,8	129.0*3, 129.1*3		128.9 *6	
7	127.0*3		126.8*3	
NCH ₃	32.3, 32.4*2	3.0 (3H, s); 3.03 (6H, s)	32.37,32.4*2	2.95 (3H,s), 2.99 (6H,s)

^a Recorded in CDCl₃; ^c D-2-hydroxyisovaleric acid (Hiv); ^d2-hydroxy-3-methylpentanoic acid (Hmp)

[3] S. Gupta, C. Montllor, and Y.S. Hwang (1995). Isolation of novel beauvericin analogues from the fungus *Beauveria bassiana*, *J. Nat. Prod.* **58**(5), 733-738.

Table S4. Summary of ¹H (600 MHz) and ¹³C (150 MHz) NMR spectroscopic data for compound **4**

position	4 ^a		enniatin B (<i>Food. Chem.</i> ⁴)	
	δ_C	δ_H mult. (<i>J</i> in Hz)	δ_C	δ_H mult. (<i>J</i> in Hz)
NMeVal	3units			
1 C=O	170.6*3		171.4*3	
2	63.4*3	4.51 (3H, d, 8.4)	63.6*3	4.7 (3H)
3	28.1*3	2.25 (3H, m)	29.1*3	2.29 (3H)
4	20.6*3	1.04 (9H, d, 5.4)	20.2*3	1.09 (9H)
4'	19.7*3	0.88 (9H, d, 6.0)	20.2*3	0.95 (9H)
N-CH ₃	33.4*3	3.11 (9H, s)	32.9*3	3.21 (9H)
Hiv ^c	3units		3units	
1 C=O	169.6*3	5.11 (3H, d, 7.8)	171.9*3	5.22 (3H)
2	75.9*3	2.17 (3H, m)	76.4*3	2.19 (3H)
3	30.2*3		31.2*3	
4	18.9*3	0.94 (9H, d, 6)	18.7*3	0.98 (9H)
4'	18.8*3	0.96 (9H, d, 6.6)	18.7*3	1.02 (9H)

^a Recorded in CDCl₃; ^c D-2-hydroxyisovaleric acid (Hiv)

[4] V. Cuomo, A. Randazzo, G. Meca, A. Moretti, A. Cascone, O. Eriksson, E. Novellino, and A. Ritieni (2013). Production of enniatins A, A1, B, B1, B4, J1 by *Fusarium tricinctum* in solid corn culture: structural analysis and effects on mitochondrial respiration, *Food. Chem.* **140**(4), 784-793.

Table S5. Summary of ^1H (600 MHz) and ^{13}C (150 MHz) NMR spectroscopic data for compound **5**.

position	5 ^a		enniatin H (<i>Tetrahedron</i> ⁵)	
	δ_{C}	δ_{H} mult. (<i>J</i> in Hz)	δ_{C}	δ_{H} mult. (<i>J</i> in Hz)
NMeVal	3units		3units	
1 C=O	170.5*3		170.3*3	
2	63.3, 63.4*2	4.50-4.53 (3H,m)	63.1, 63.2, 63.3	4.55-4.57 (3H, m)
3	28.0, 28.1,28.2	2.23-2.29 (3H,m)	27.8, 27.9, 28.0	2.28-2.29 (3H, m)
4	20.5*2, 20.6	1.03 (9H ,m)	20.3*2, 20.4	1.06 (9H, m)
4'	19.5, 19.6,19.7	0.87-0.89 (9H, m)	19.3, 19.4, 19.5	0.89-0.90 (9H, m)
N-CH ₃	33.1*2, 33.3	3.08 (3H, s) 3.09 (3H, s) 3.11 (3H, s)	32.9*2, 33.1	3.11 (3H, s) 3.13 (3H, s) 3.14 (3H, s)
Hiv ^c	2units		2units	
1 C=O	169.5*2		169.3*2	
2	75.8, 76	5.12 (2H, dd, 9,13.8)	75.9, 75.6	5.13-5.15 (2H, m)
3	30.1*2	2.26-2.29 (2H, m)	29.9*2	2.28 (2H, m)
4	18.8, 18.9	0.96 (6H, d, 6.0)	18.6, 18.7	0.98 (6H, m)
4'	18.6, 18.7	0.94 (9H, d, 6.6)	18.5*2	0.96 (6H, m)
Hmp ^d	1unit		1unit	
1 C=O	169.5		169.3	
2	74.4	5.24 (1H, d, 6)	74.3	5.27 (1H, d, 6.8)
3	36.3	1.98(1H, m)	36.1	2.00 (1H, m)
4	25.6	1.44(1H, m) 1.17(1H, m)	25.4	1.46 (1H, m) 1.19 (1H, m)
5	11.5	0.89-0.90 (3H, m)	11.3	0.92 (3H, m)
3-CH ₃	14.8	0.94 (9H, d, 6.6)	14.6	0.96 (3H, m)

^a Recorded in CDCl₃; ^c D-2-hydroxyisovaleric acid (Hiv); ^d2-hydroxy-3-methylpentanoic acid (Hmp)

[5] C. Nilanonta, M. Isaka, R. Chanphen, N. Thong-Orn, M. Tanticharoen, and Y. Thebtaranonth (2003). Unusual enniatins produced by the insect pathogenic fungus *Verticillium hemipterigenum*: Isolation and studies on precursor-directed biosynthesis, *Tetrahedron* **59(7)**, 1015-1020.

Table S6. Summary of ¹H (600 MHz) and ¹³C (150 MHz) NMR spectroscopic data for compound **6**.

position	6 ^a		enniatin I (<i>Tetrahedron</i> ⁵)	
	δ_C	δ_H mult. (<i>J</i> in Hz)	δ_C	δ_H mult. (<i>J</i> in Hz)
NMeVal	3units		3units	
1 C=O	170.6*3		170.3*3	
2	63.2, 63.3, 63.4	4.53-4.55 (3H, m)	63.1*3	4.55-4.56 (3H, m)
3	27.9, 28.0*2	2.27 (3H, m)	27.8, 27.9*2	2.28-2.30 (3H, m)
4	20.5, 20.6*2	1.04 (9H, m)	20.3*3	1.06 (9H, m)
4'	19.4, 19.5, 19.6	0.86-0.88 (9H, m)	19.2, 19.3, 19.4	0.89 (9H, m)
N-CH ₃	33.0, 33.1*2	3.07 (3H, s)	32.7, 32.9*2	3.09 (3H, s)
		3.09 (3H, s)		3.11 (3H, s)
		3.10 (3H, s)		3.12 (3H, s)
Hiv ^c	1unit		1unit	
1 C=O	169.4		169.2*1	
2	75.9	5.13 (1H, d, 7.8)	75.7	5.15(1H, d, 8.2)
3	30.1	2.27 (1H, m)	29.9	2.29(1H, m)
4	18.7	0.96 (3H, d, 6.6)	18.6	0.98(3H, m)
4'	18.6	0.94 (3H, m)	18.5	0.95(3H, m)
Hmp ^d	2units		2units	
1 C=O	169.4*2		169.2*2	
2	74.6, 74.5	5.26 (2H, dd, 6.6,11.4)	74.2, 74.4	5.27-5.28 (2H, m)
3	36.3, 36.4	2.0 (2H, m)	36.1*2	2.02 (2H, m)
4	25.6*2	1.44 (2H, m)	25.3*2	1.46 (2H, m)
		1.14-1.19 (2H, m)		1.18-1.19 (2H, m)
5	11.5*2	0.90 (6H, t, 7.2)	11.3*2	0.92 (6H, t, 7.5)
3-CH ₃	14.7*2	0.94 (6H, m)	14.6*2	0.96 (6H, m)

^a Recorded in CDCl₃; ^c D-2-hydroxyisovaleric acid (Hiv); ^d2-hydroxy-3-methylpentanoic acid (Hmp)

[5] C. Nilanonta, M. Isaka, R. Chanphen, N. Thong-Orn, M. Tanticharoen, and Y. Thebtaranonth (2003). Unusual enniatins produced by the insect pathogenic fungus *Verticillium hemipterigenum*: Isolation and studies on precursor-directed biosynthesis, *Tetrahedron* **59**(7), 1015-1020.

Table S7. Summary of ¹H (600 MHz) and ¹³C (150 MHz) NMR spectroscopic data for compound **7**.

position	7 ^a		enniatin MK1688 (<i>Tetrahedron</i> ⁵)	
	δ_C	δ_H mult. (<i>J</i> in Hz)	δ_C	δ_H mult. (<i>J</i> in Hz)
NMeVal	3units		3units	
1 C=O	170.6*3		170.4*3	
2	63.3*3	4.56 (3H, d, 9)	63.1*3	4.59 (3H, brd, 9.4)
3	28.0*3	2.25 (3H, m)	27.8*3	2.29 (3H, m)
4	20.5*3	1.01 (9H, d, 6)	20.3*3	1.06 (9H, d, 6.1)
4'	19.6*3	0.86 (9H, d, 6.6)	19.3*3	0.89 (9H, d, 6.9)
N-CH ₃	32.8*3	3.07 (9H, s)	32.7*3	3.10 (9H, s)
Hmp ^d	3units		3units	
1 C=O	169.5*3		169.2*3	
2	74.5*3	5.25 (3H, d, 4.8)	74.3*3	5.28 (3H, brd, 5.6)
3	36.4*3	1.98 (3H, m)	36.2*3	2.02 (3H, m)
4	25.6*3	1.42 (3H, m)	25.4*3	1.45 (3H, m)
		1.16 (3H, m)		1.19 (3H, m)
5	11.5*3	0.89 (9H, t, 7.2)	11.3*3	0.92 (9H, t, 7.4)
3-CH ₃	14.8*3	0.92 (9H, d, 6.6)	14.6*3	0.96 (9H, d, 6.4)

^a Recorded in CDCl₃; ^d2-hydroxy-3-methylpentanoic acid (Hmp)

[5] C. Nilanonta, M. Isaka, R. Chanphen, N. Thong-Orn, M. Tanticharoen, and Y. Thebtaranonth (2003). Unusual enniatins produced by the insect pathogenic fungus *Verticillium hemipterigenum*: Isolation and studies on precursor-directed biosynthesis, *Tetrahedron* **59** (7), 1015-1020.

Table S8. Summary of ^1H (600 MHz) and ^{13}C (150 MHz) NMR spectroscopic data for compounds **8–9**.

position	8^a		fusaric acid (<i>Appl Environ Microbiol</i> ⁶)		9^a	
	δ_{C}	δ_{H} mult. (J in Hz)	δ_{C}	δ_{H} mult. (J in Hz)	δ_{C}	δ_{H} mult. (J in Hz)
2	145.3, C		147.8, C		144.7, C	
3	124.8, CH	8.16 (1H, d, 7.2) ^b	128.8, CH	8.26d (1H, d, 8.2)	123.9, CH	8.14 (1H, d, 7.2) ^c
4	138.7, CH	7.73 (1H, s) ^b	143.1, CH	8.47dd (1H, 1.8, 8.2)	136.3, CH	7.73 (1H, d, 7.2) ^c
5	143.2, C		146.1, C		142.0, C	
6	147.7, CH	8.74 (1H, s)	149.7, CH	8.57 (1H, brs)	148.2, CH	8.55 (1H, s)
7	165.5, COOH	13.16 (1H, s)	166.6, COOH		164.9, COOH	7.24 (1H, s)
8	33.0, CH ₂	2.68 (2H, t, 7.2)	34.4, CH ₂	2.87 (2H, t, 7.6)	34.6, CH ₂	2.81 (2H, t, 7.2)
9	32.9, CH ₂	1.58 (2H, m)	34.3, CH ₂	1.66 (2H, m)	32.3, CH ₂	2.39 (2H, dd, 7.2)
10	22.3, CH ₂	1.30 (2H, m)	24.1, CH ₂	1.32 (2H, m)	138.8, CH	5.78 (1H, m)
11	13.8, CH ₃	0.86 (3H, t, 7.2)	15.7, CH ₃	0.89 (3H, t, 7.2)	116.3, CH ₂	4.99 (2H, d, 12.6)

^a Recorded in CDCl₃; Fusaric acid was recorded in D₂O in the literature; ^{b&c} According to the structure of compounds **8** and **9**, the chemical shifts of position 3 and 4 should be assigned as it is in this paper, not as it is in the literature.

[6] H.R. Burmeister, M.D. Grove, R.E. Peterson, D. Weisleder, and R.D. Plattner (1985). Isolation and characterization of two new fusaric acid analogs from *Fusarium moniliforme* NRRL 13,163, *Appl Environ Microbiol* **50**(2), 311-314.

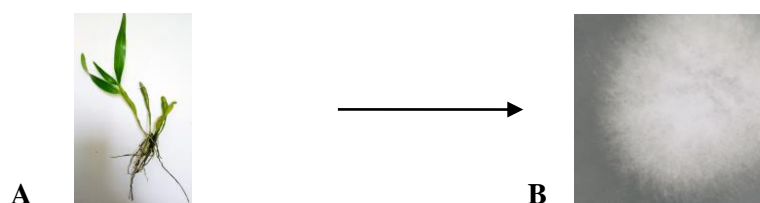


Figure S1. *D. officinale* kimura et Migo (A) and pure culture of *Fusarium* sp. TP-G1 obtained from the root of *D. officinale* kimura et Migo at 28 °C for 7 days (B).

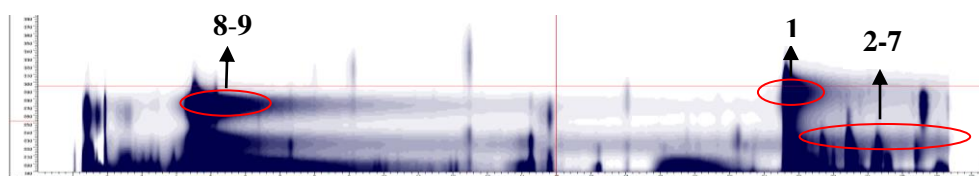


Figure S2. HPLC-DAD screening of the fermentation extracts of *Fusarium* sp. TP-G1.

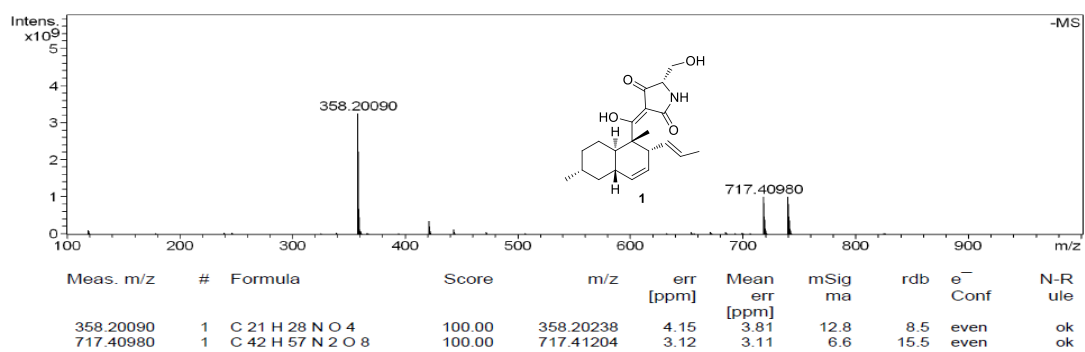


Figure S3. HRESIMS spectrum of trichosetin (1)

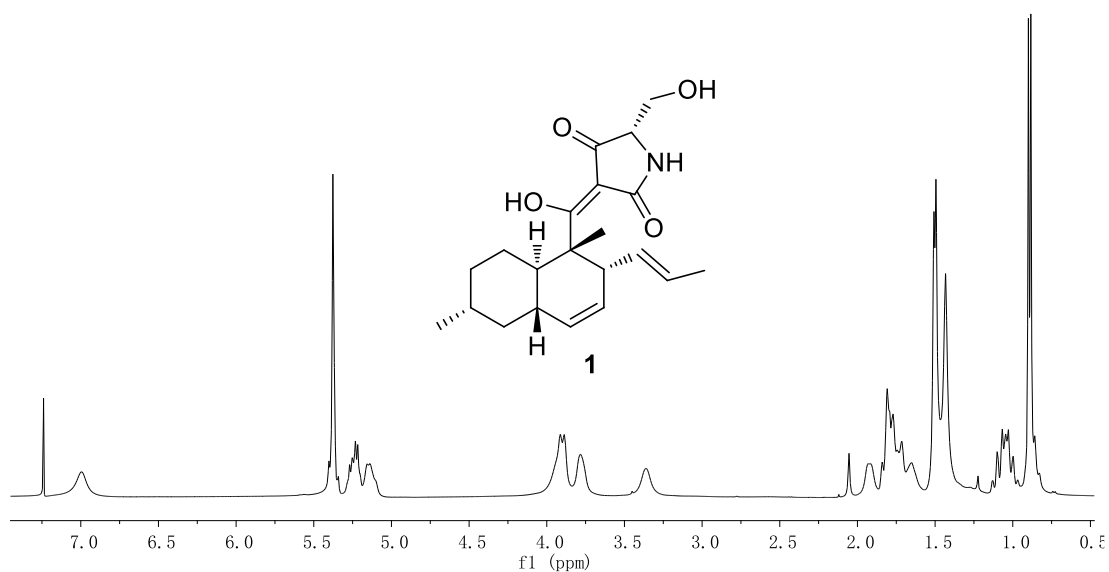


Figure S4. ¹H NMR (600 MHz) spectrum of trichosetin (1) in CDCl₃

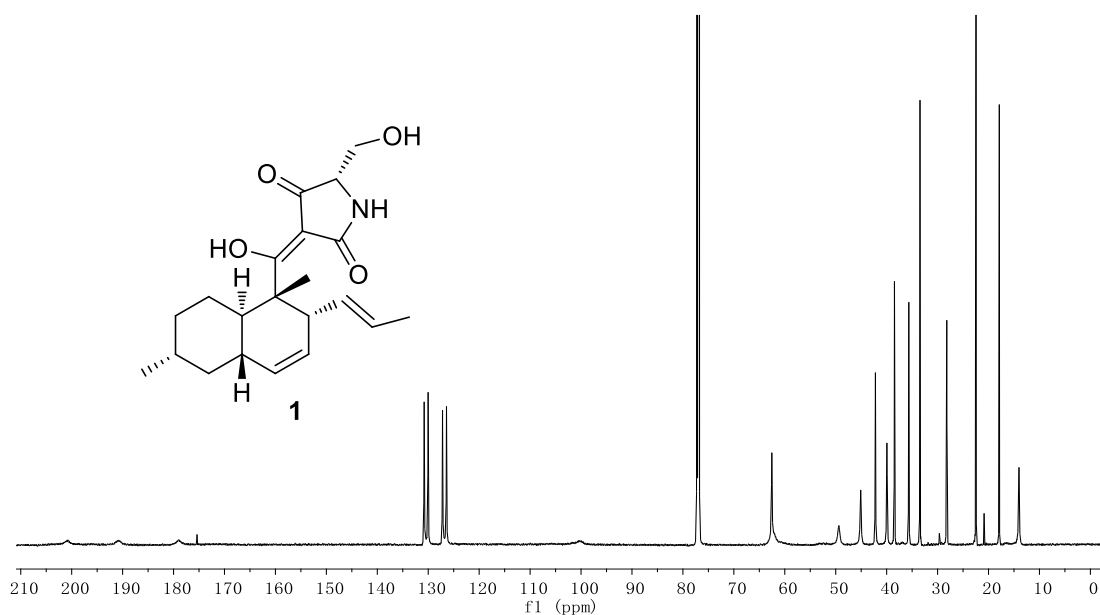
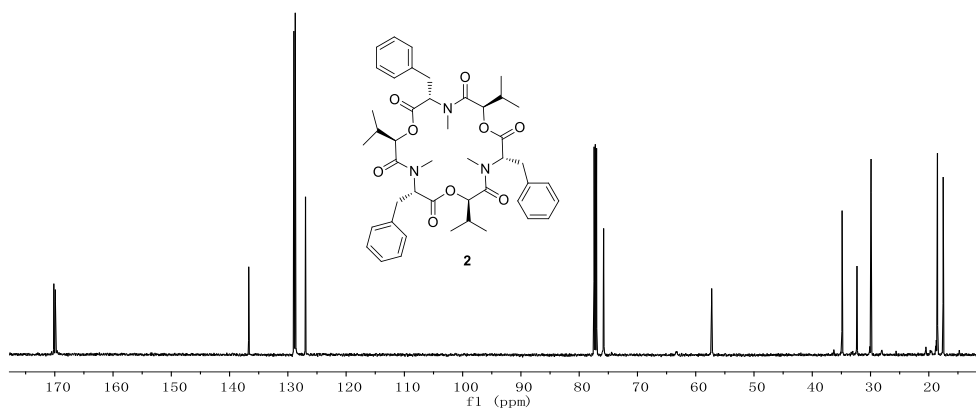
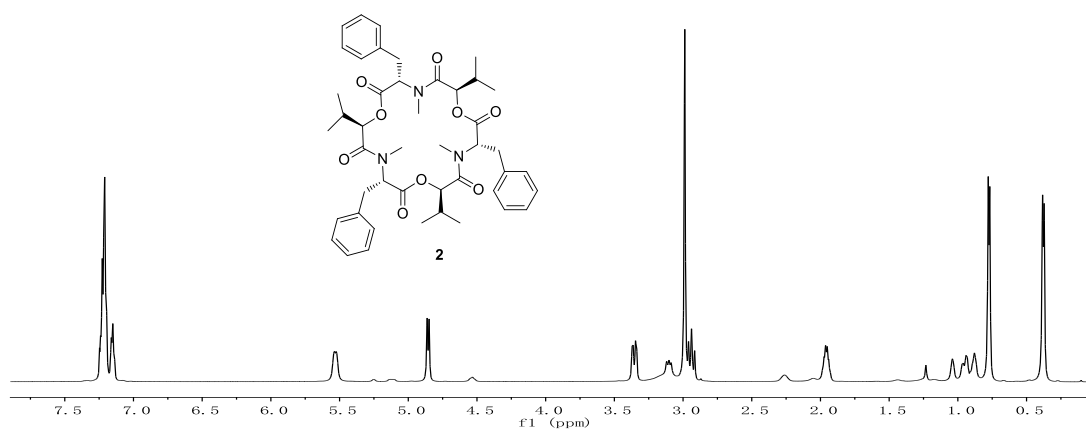
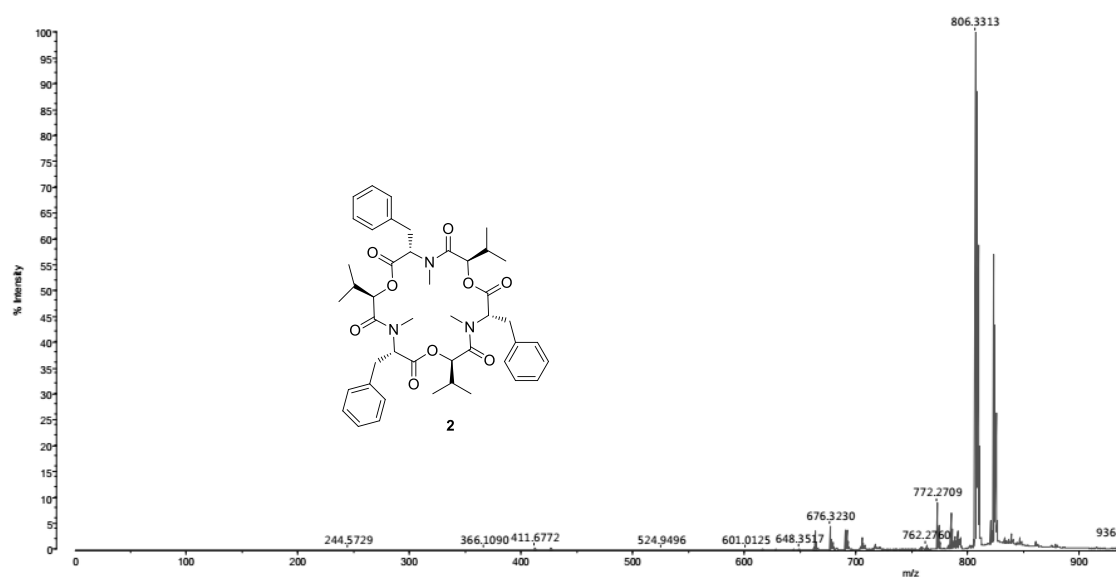


Figure S5. ¹³C NMR (150 MHz) spectrum of trichosetin (1) in CDCl₃



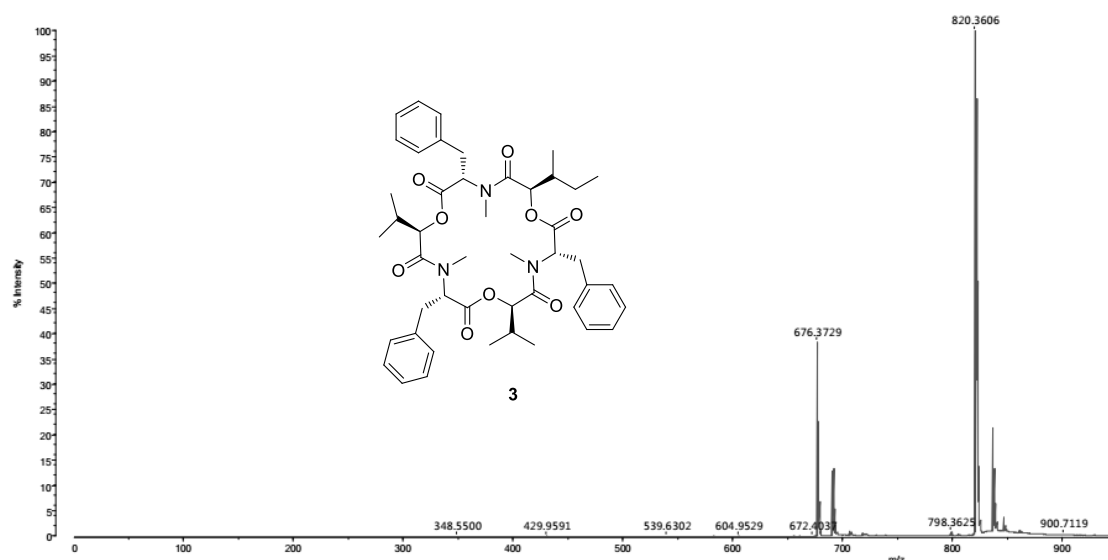


Figure S9. MS spectrum of beavericin A (3)

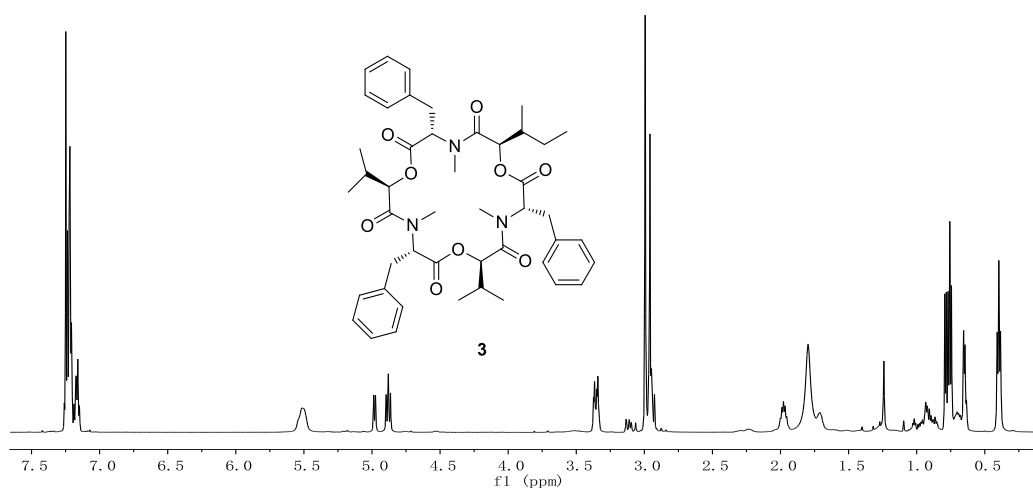


Figure S10. ^1H NMR (600 MHz) spectrum of beavericin A (3) in CDCl_3

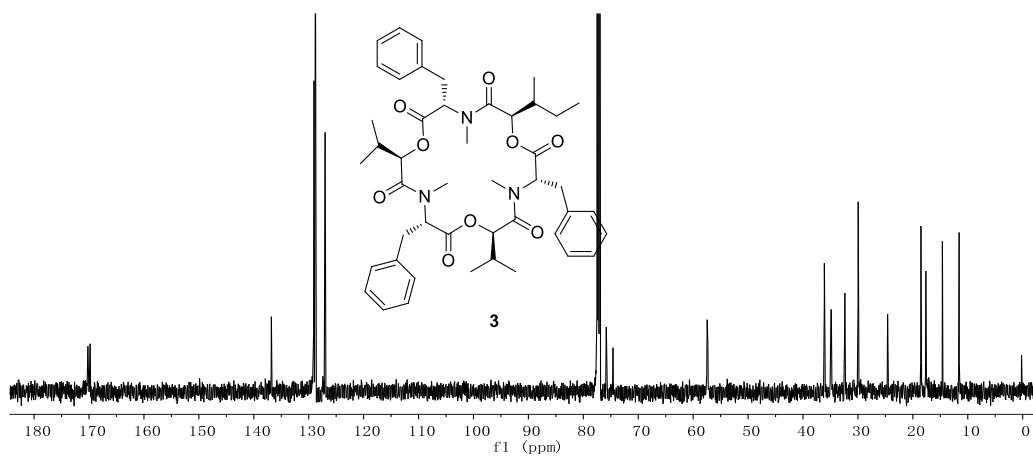


Figure S11. ^{13}C NMR (150 MHz) spectrum of beavericin A (3) in CDCl_3

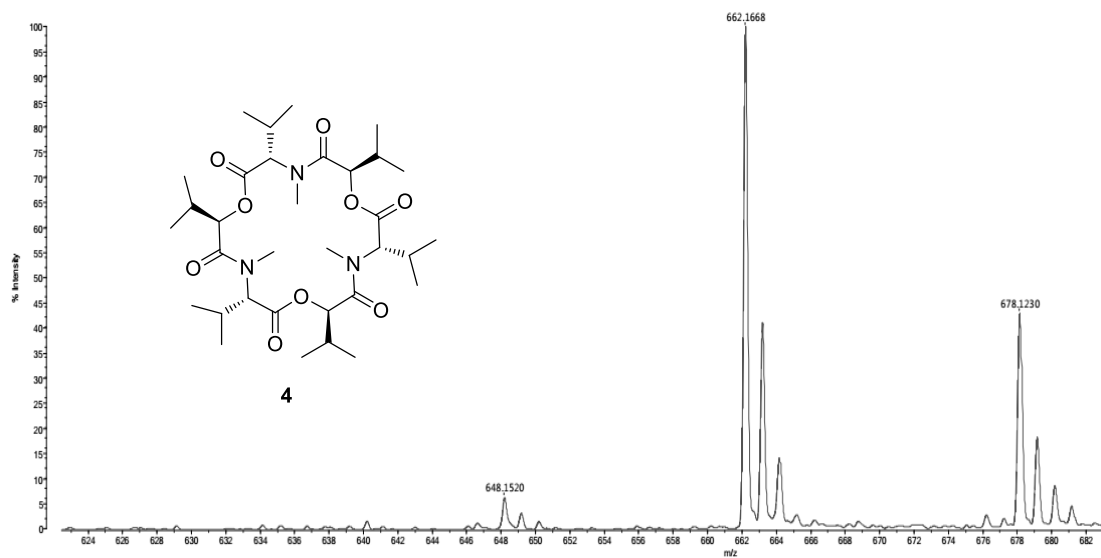


Figure S12. MS spectrum of enniatin B (**4**)

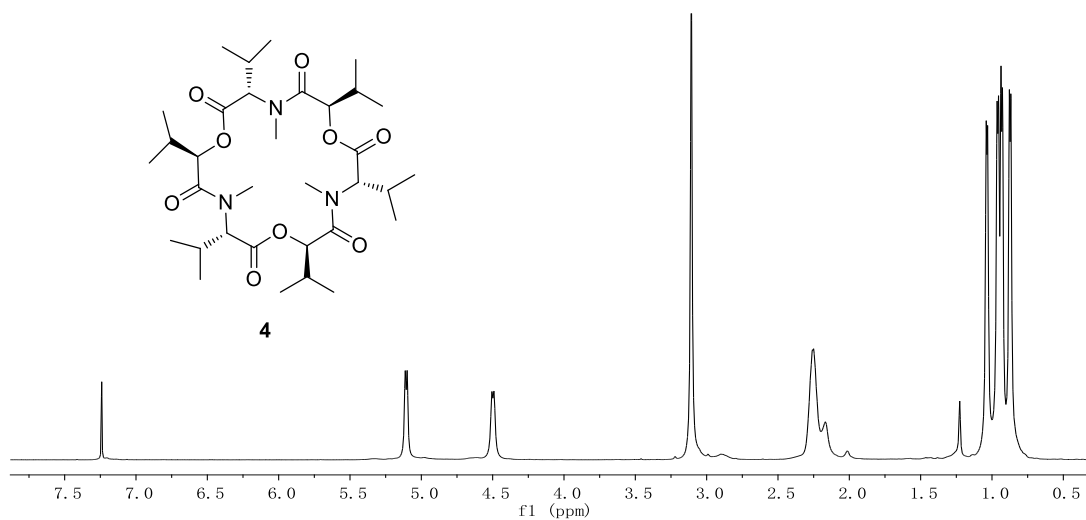


Figure S13. ^1H NMR (600 MHz) spectrum of enniatin B (**4**) in CDCl_3

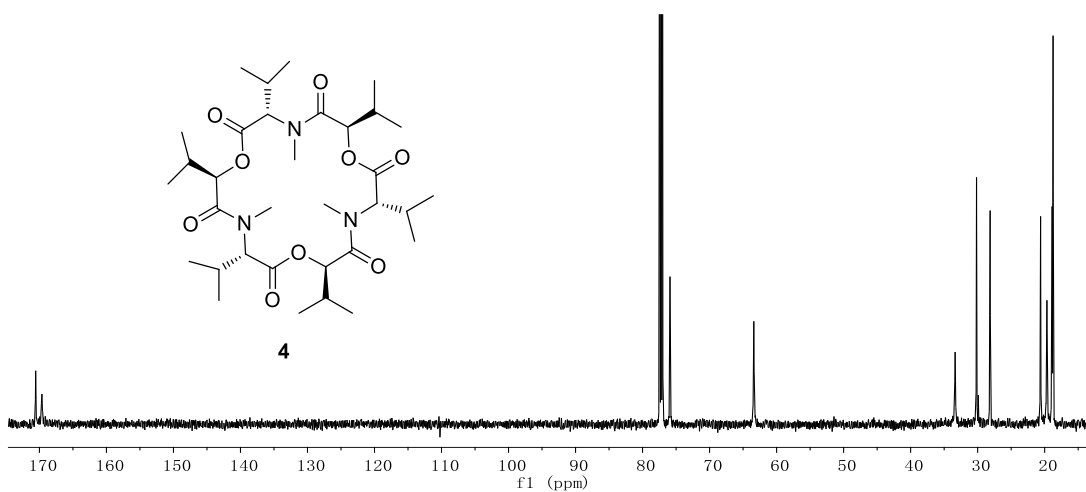


Figure S14. ^{13}C NMR (150 MHz) spectrum of enniatin B (**4**) in CDCl_3

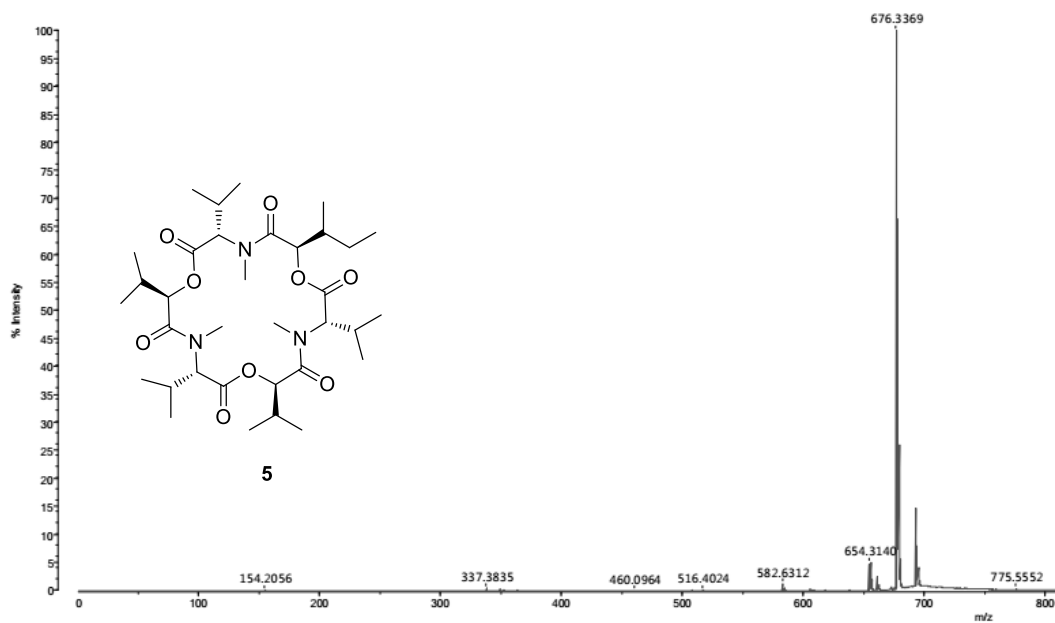


Figure S15. MS spectrum of enniatin H (**5**)

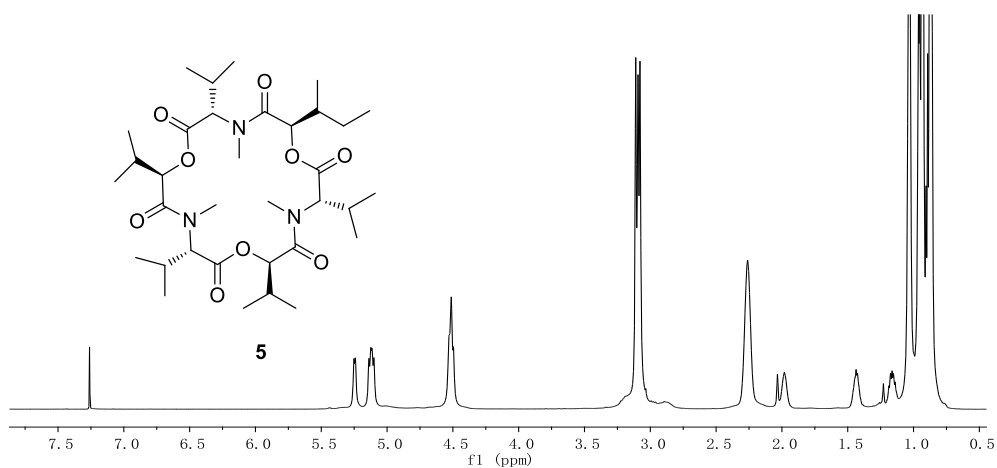


Figure S16. ¹H NMR (600 MHz) spectrum of enniatin H (**5**) in CDCl₃

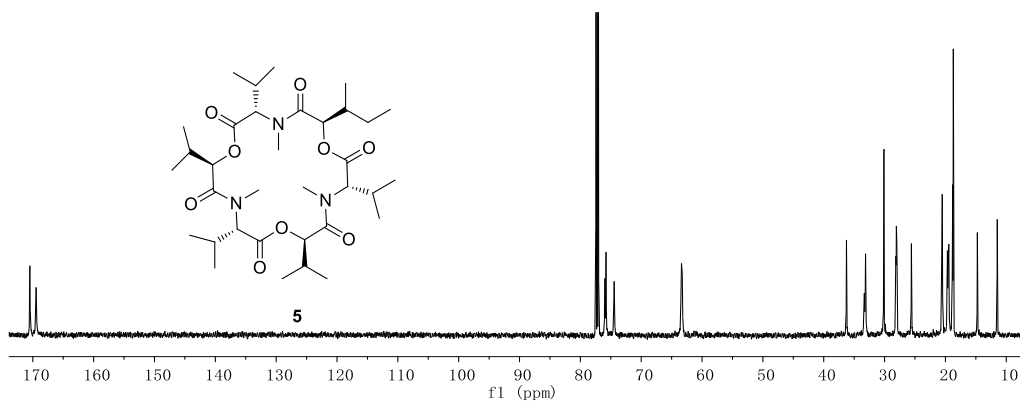


Figure S17. ¹³C NMR (150 MHz) spectrum of enniatin H (**5**) in CDCl₃

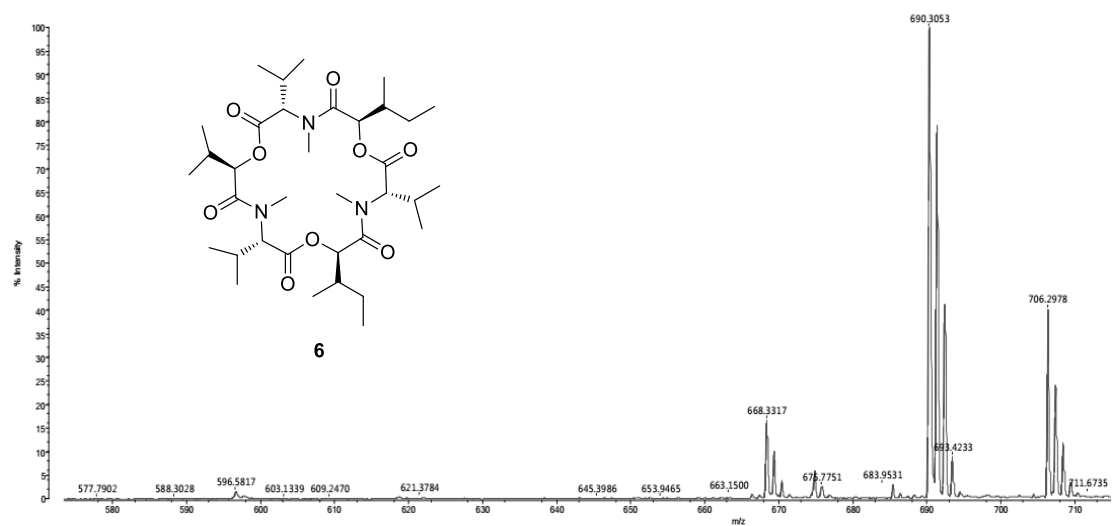


Figure S18 MS spectrum of enniatin I (**6**)

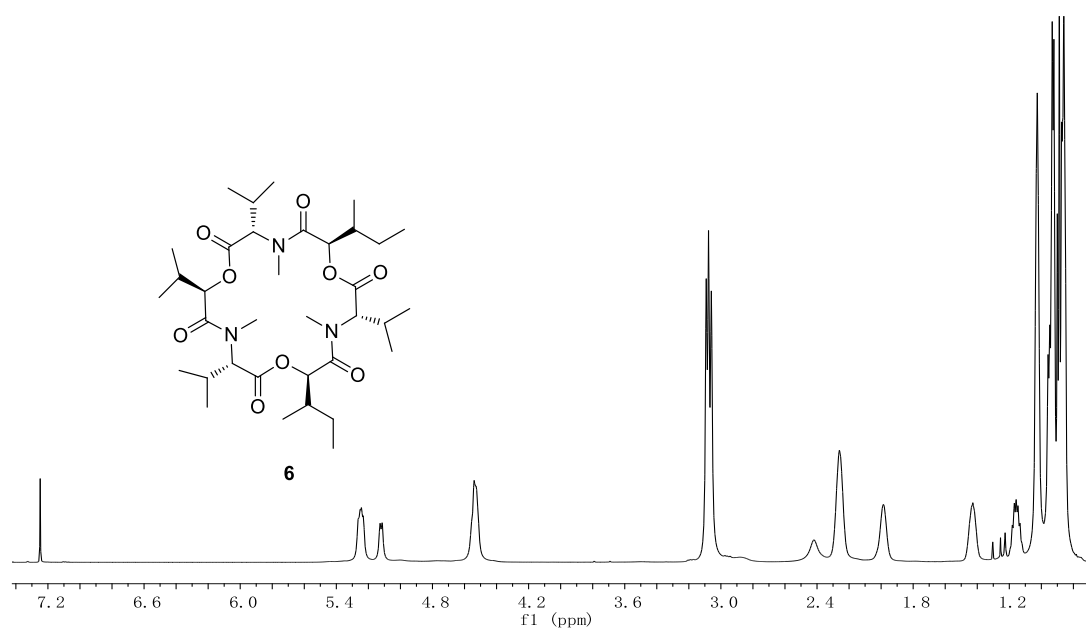


Figure S19. ¹H NMR (600 MHz) spectrum of enniatin I (**6**) in CDCl₃

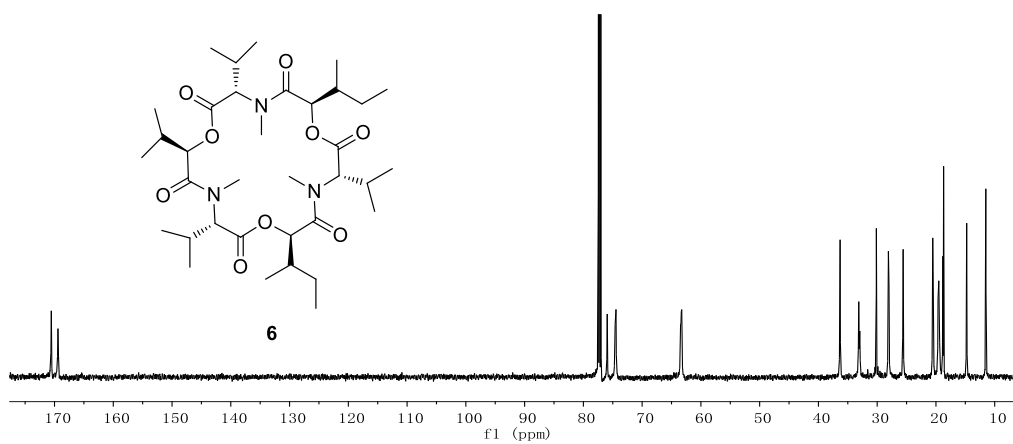


Figure S20. ¹³C NMR (150 MHz) spectrum of enniatin I (**6**) in CDCl₃

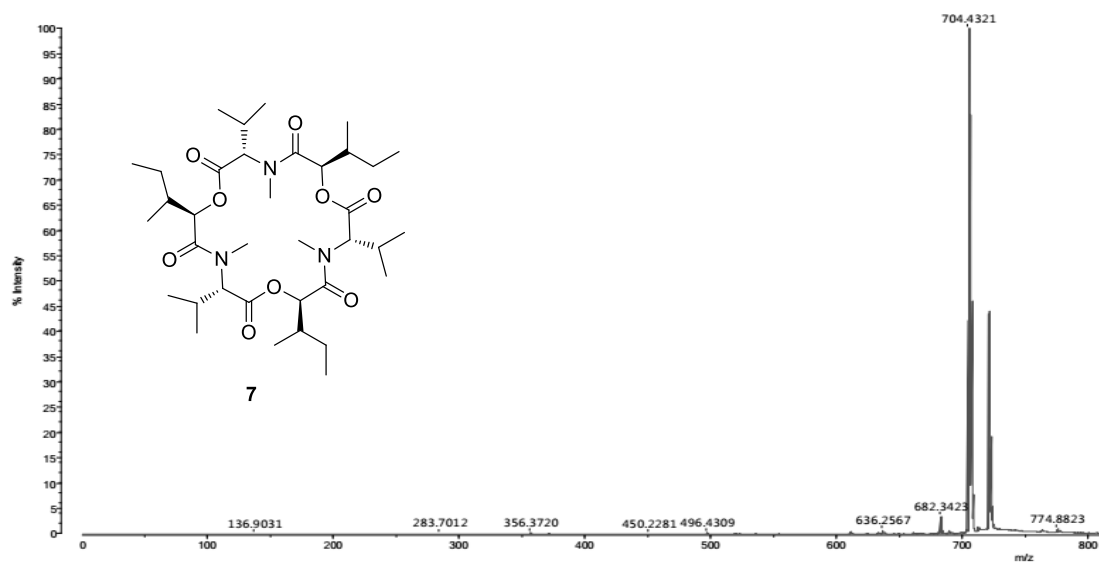


Figure S21. MS spectrum of enniatin MK1688 (7)

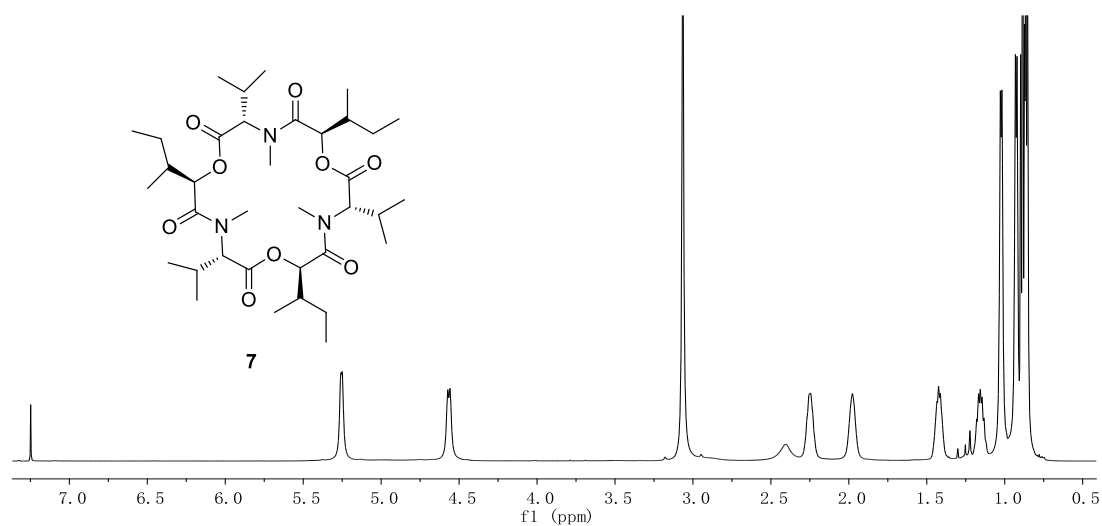


Figure S22. ^1H NMR (600 MHz) spectrum of enniatin MK1688 (7) in CDCl_3

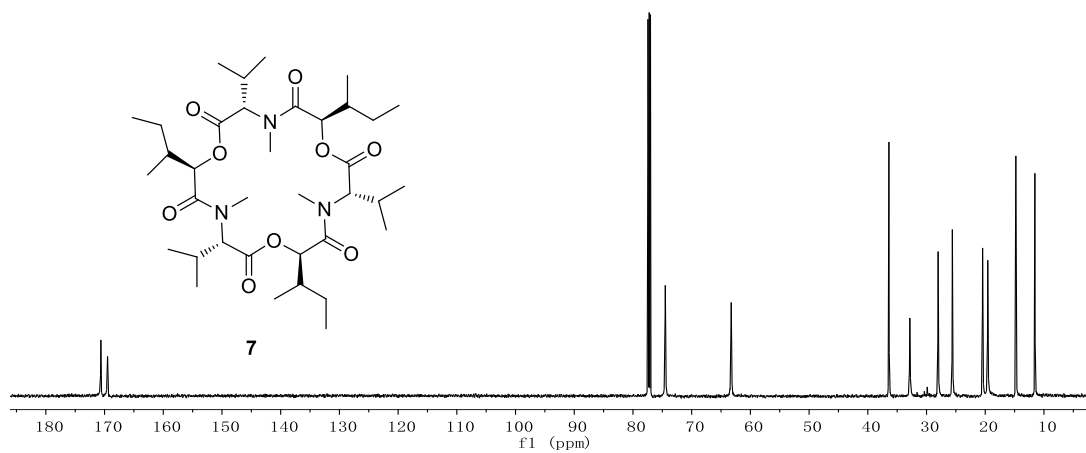


Figure S23. ^{13}C NMR (150 MHz) spectrum of enniatin MK1688 (7) in CDCl_3

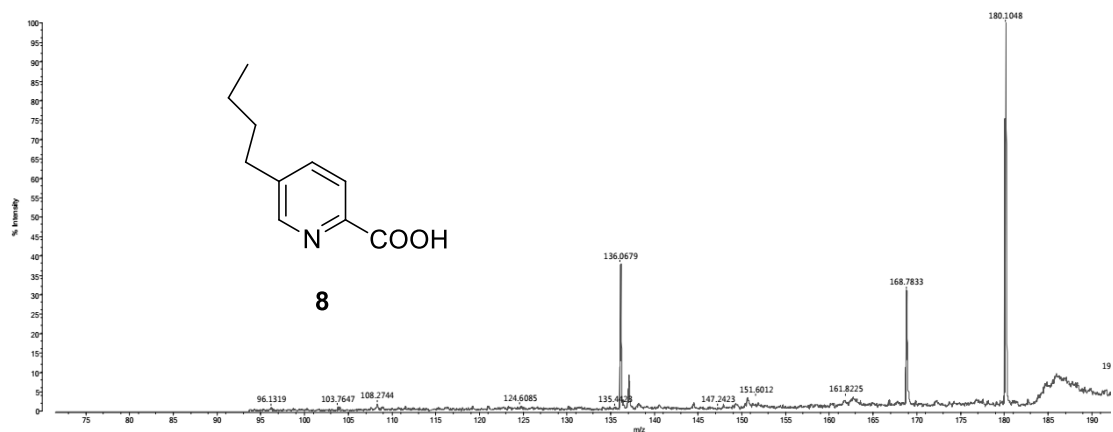


Figure S24. MS spectrum of fusaric acid (**8**)

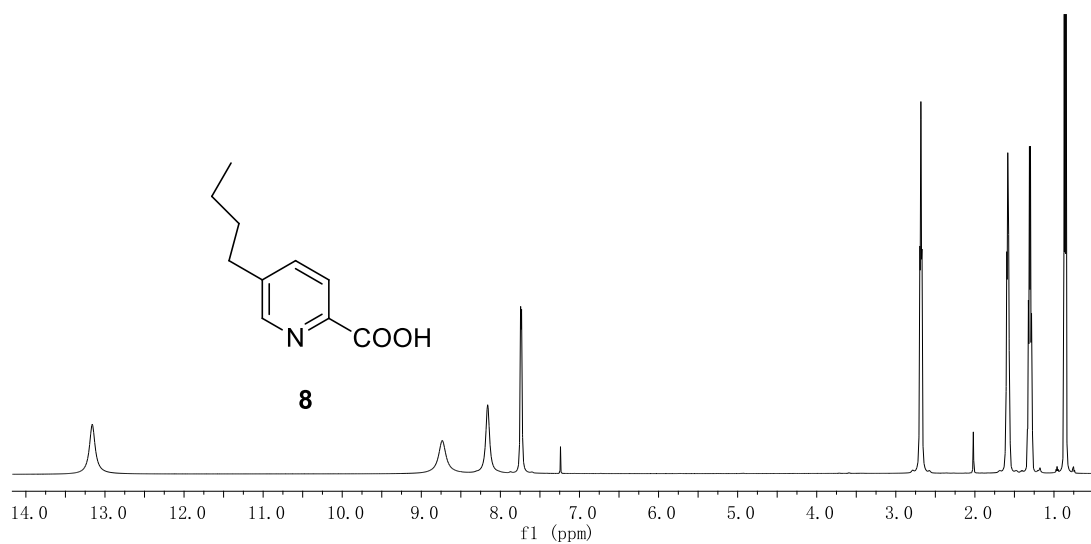


Figure S25. ^1H NMR (600 MHz) spectrum of fusaric acid (**8**) in CDCl_3

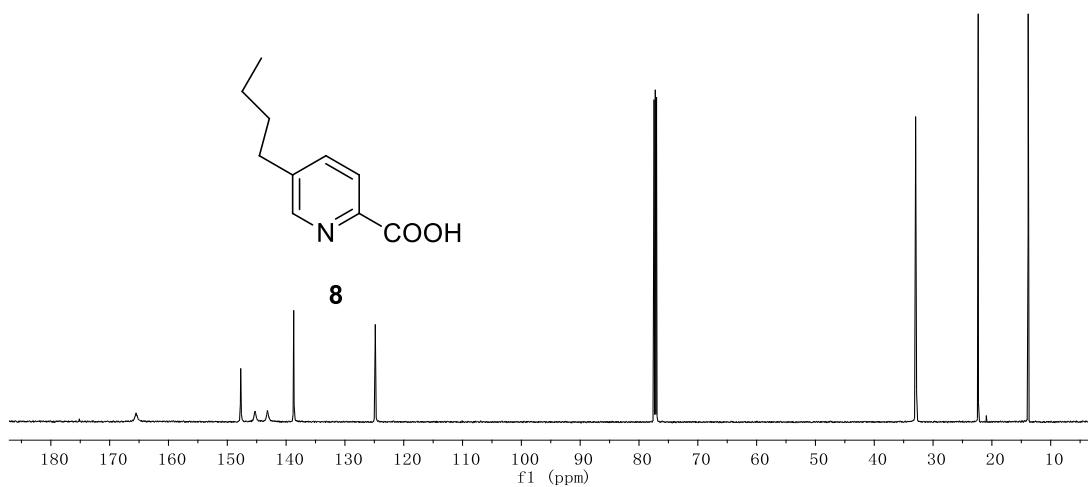


Figure S26. ^{13}C NMR (150 MHz) spectrum of fusaric acid (**8**) in CDCl_3

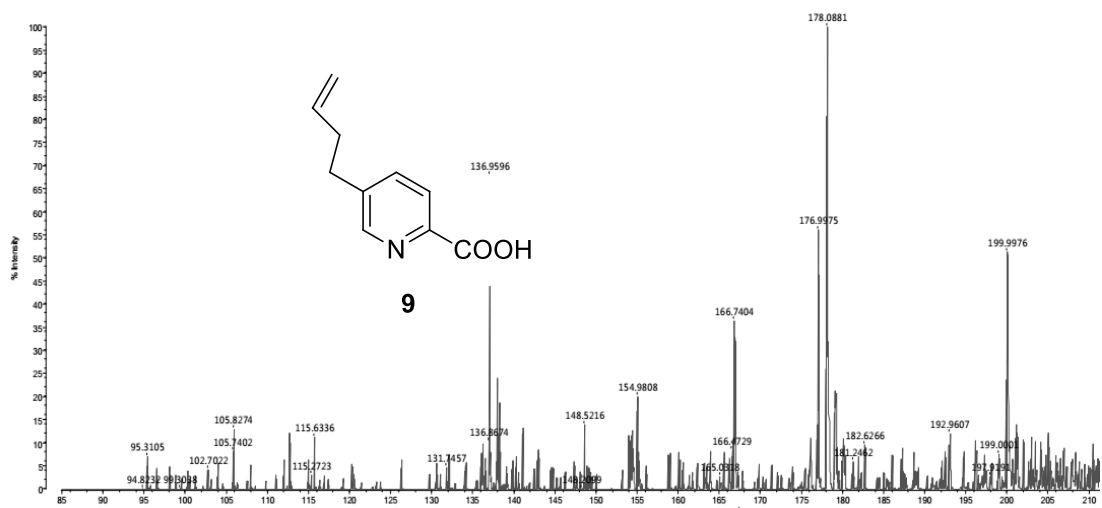


Figure S27. MS spectrum of dehydrofusaric acid (9)

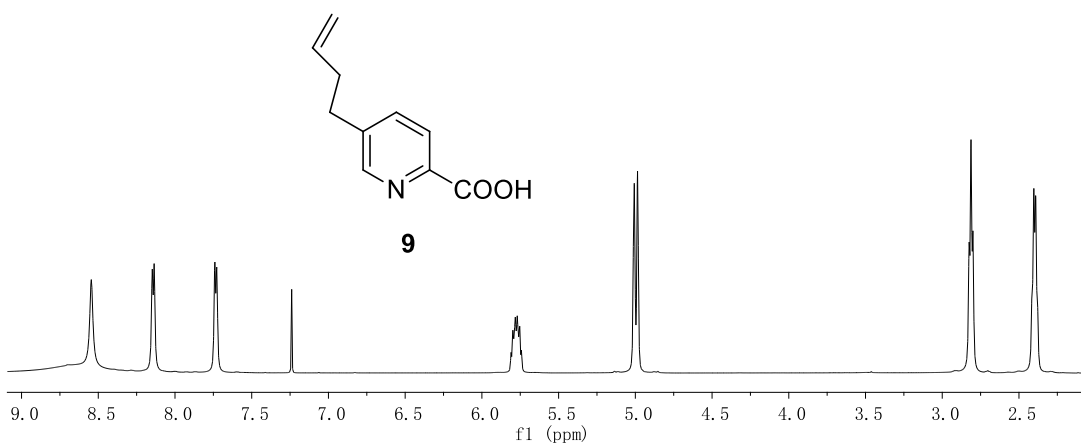


Figure S28. ^1H NMR (600 MHz) spectrum of dehydrofusaric acid (9) in CDCl_3

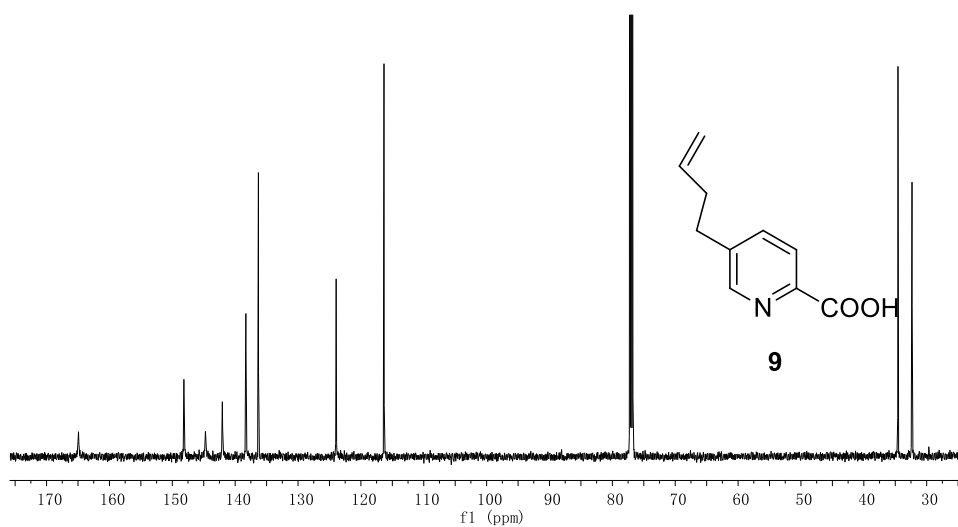


Figure S29. ^{13}C NMR (150 MHz) spectrum of dehydrofusaric acid (9) in CDCl_3