Structure and Absolute Stereochemistry of Phormidolide, a New Toxic Metabolite from the Marine Cyanobacterium *Phormidium* sp.

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Supporting Information:

Figure S1. ¹H NMR spectrum of phormidolide (1)

Figure S2. ¹³C NMR spectrum of phormidolide (1)

Figure S3. E.COSY spectrum of phormidolide (1)

Figure S4. PEP-HSQC spectrum of phormidolide (1)

Figure S5. GHMBC spectrum of phormidolide (1)

Figure S6. Pure absorbtion TOCSY spectrum of phormidolide (1)

Figure S7. GROESY spectrum of phormidolide (1)

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Figure S10. ¹H NMR spectrum of the diacetone derivative (3)

Figure S11. HSQC spectrum of the diacetone derivative (3)

Figure S12. HSQC spectrum of the diacetone derivative (3) showing expansion of
acetone methyl groups

Figure S13. ROESY spectrum of the diacetone derivative (3)

Figure S14. Expansion of ROESY spectrum of the diacetone derivative (3)

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Figure S16. DQF-COSY spectrum of the diacetone-diMPA derivative (4)

Table S1. Table of ¹H NMR chemical shifts for diacetone (3) and diacetone diMPA
(4) derivatives

Figure S1. ¹H NMR spectrum of phormidolide (1)

Current Data Parameters
 NAME avph058
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20000318
 Time 20.54
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 PULPROG zg30
 TD 32768
 SOLVENT CDCl₃
 NS 64
 DS 2
 SWH 5995.204 Hz
 FIDRES 0.182959 Hz
 AQ 2.7329011 sec
 RG 90.5
 DM 83.400 usec
 DE 6.00 usec
 TE 300.0 K
 D1 2.00000000 sec

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 P1 8.25 usec
 PL1 0.00 dB
 SF01 400.1324000 MHz

F2 - Processing parameters
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 SF 400.1300094 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
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 FIP 8.000 ppm
 F1 3201.04 Hz
 F2P 0.000 ppm
 F2 0.00 Hz
 PPMCM 0.40000 ppm/cm
 HZCM 160.05200 Hz/cm

Figure S2. ^{13}C NMR spectrum of phormidolide (1)

Figure S3. E.COSY spectrum of phormidolide (1)

Figure S4. PEP-HSQC spectrum of phormidolide (1)

Figure S5. GHMBC spectrum of phormidolide (1)

Figure S6. Pure absorbtion TOCSY spectrum of phormidolide (1)

Figure S7. GROESY spectrum of phormidolide (1)

Figure S8. FAB-MS spectrum of phormidolide (1) in 3-NBA

Figure S9. HSQMBBC spectrum of phormidolide (1)

Figure S10. ¹H NMR spectrum of the diacetone derivative (3)

Figure S11. HSQC spectrum of the diacetonide derivative (3)

Figure S12. HSQC spectrum of the diacetonide derivative (3) showing expansion of acetonide methyl groups

NAME	Current Data Parameters
NAME	NAME
EXPNO	EXPNO
PROCNO	PROCNO
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TIME	TIME
INSTRUM	INSTRUM
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DELTA100	DELTA100

Figure S13. ROESY spectrum of the diacetone derivative (3)

NAME	Current Date Parameters
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00000001	2
00000002	00000004

2.2 - Acquisition Parameters

[illegible]

F1 - Acquisition per poster.

Figure S14. Expansion of ROESY spectrum of the diacetone derivative (3)

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S15

Figure S15. ^1H NMR spectrum of the diacetone-diMPA derivative (4)

Figure S16. DQF-COSY spectrum of the diacetone-diMPAderivative (4)

Table S1. Table of ^1H NMR chemical shifts for diacetonide (3) and diacetonide diMPA (4) derivatives

Carbon	diacetonide	diacetonide-diMPA
1		
2	5.8, s	5.76, s
3		
4	6.19, d (14.6)	6.14, d (14.8)
5	5.88, ddd (15.8, 11.8, 3.9)	5.80, ddd (15.4, 11.1, 3.9)
6	2.85, ddd (11.0, 4.0, 3.6)	2.62, m
	2.85, ddd (12.0, 11.6, 10.0)	2.10, m
7	4.02, ovlp	4.01, m
8	2.47, d (15.4)	2.47, d (15.0)
	1.81, ovlp	1.77, ovlp
9		
10	5.28, bs	5.24, bs
11		
12	2.33, ovlp	2.33, overlapped
	2.59, ovlp	2.58, overlapped
13	4.49, m	4.48, m
14	1.57, ovlp	1.57, overlapped
	2.33, ovlp	2.33, overlapped
15	5.15, d (4.4)	5.15, d (4.4)
16		
17	4.67, d (9.2)	4.66, d (9.2)
18	5.40, ovlp	5.32, ovlp
19		
20	2.06, dd (13.1, 10.5)	3.66, dd (13.1, 10.5)
	2.24, ovlp	2.22, ovlp
21	3.67, dd (8.8, 2.0)	3.52, dd (8.0, 2.5)
22		
23	3.47, dd (10.6, 6.6)	3.47, dd (10.5, 6.8)
24	1.65, m	1.65, m
	1.47, d (14.6)	1.47, d (14.0)
25	4.06, ovlp	4.07, ovlp
26	1.49, m	1.49, m
27	3.98, dd (4.4)	3.97, dd (4.4)
28	1.84 / 1.70 ovlp	1.84 / 1.70 ovlp
29	5.08, m	5.08, m
30	2.53 / 2.53 ovlp	2.53 / 2.53 ovlp
31		
32		
33	5.33, s	5.30, s
34	2.09, s	2.06, s
35	4.76, bs	4.80, bs
	4.98, bs	5.14, bs
36	1.58, s	1.58, s
37	1.16, s	1.16, s
38	1.80, s	1.80, s
39	0.85, s	0.85, s
40	0.76, s	0.76, s
41	0.86, ovlp	0.86, ovlp
42	5.37, s	5.37, s
	5.42, ovlp	5.42, ovlp
43	3.59, s	3.59, s
44		
45	2.27 / 2.27 ovlp	2.27 / 2.27 ovlp
46	1.61 / 1.61 ovlp	1.61 / 1.61 ovlp
47-56	1.25 / 1.25 ovlp	1.25 / 1.25 ovlp
57	1.30 / 1.30 ovlp	1.30 / 1.30 ovlp
58	1.25 / 1.25 ovlp	1.25 / 1.25 ovlp
59	0.87, t	0.87, t
60		
61	1.39, s (^{13}C -19.0 ppm)	1.39, s
62	1.37, s (^{13}C -29.9 ppm)	1.37, s
63		
64	1.41, s (^{13}C -19.5 ppm)	1.41, s
65	1.36, s (^{13}C -29.9 ppm)	1.36, s
66		
67	-	4.75, s
68		
69	-	7.43, d (8.1)
70	-	7.37, dd (8.0, 7.3)
71	-	7.34, t (7.3)
72	-	3.42, s
73		
74	-	4.75, s
75		
76	-	7.43, d (8.1)
77	-	7.37, dd (8.0, 7.3)
78	-	7.34, t (7.3)
79	-	3.42, s