

Innovative Approach to Sustainable Marine Invertebrate Chemistry and a Scale-Up Technology for Open Marine Ecosystems.

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Elemental Composition Report

Single Mass Analysis

Tolerance = 20.0 PPM / DBE: min = -1.5, max = 100.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 9

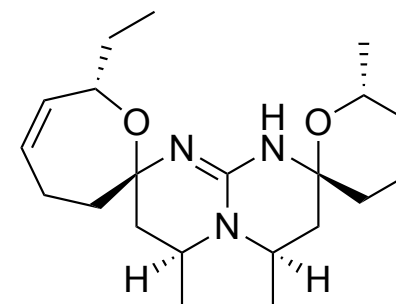
Monoisotopic Mass, Even Electron Ions

460 formula(e) evaluated with 6 results within limits (all results (up to 1000) for each mass)

Elements Used:

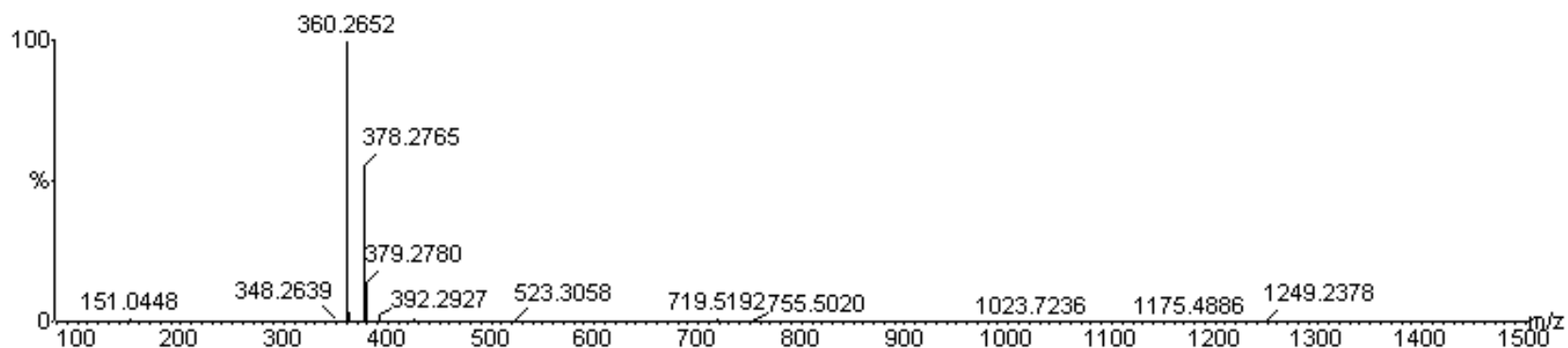
C: 0-50 H: 0-100 N: 0-10 O: 0-10

OUAZZANI_glegoff56-1 23 (0.608) Cm (21:26)



crambescidin 359 (1)

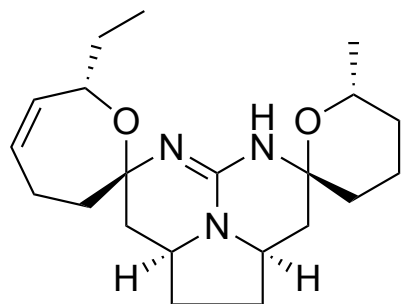
1: TOF MS ES+
8.30e+004



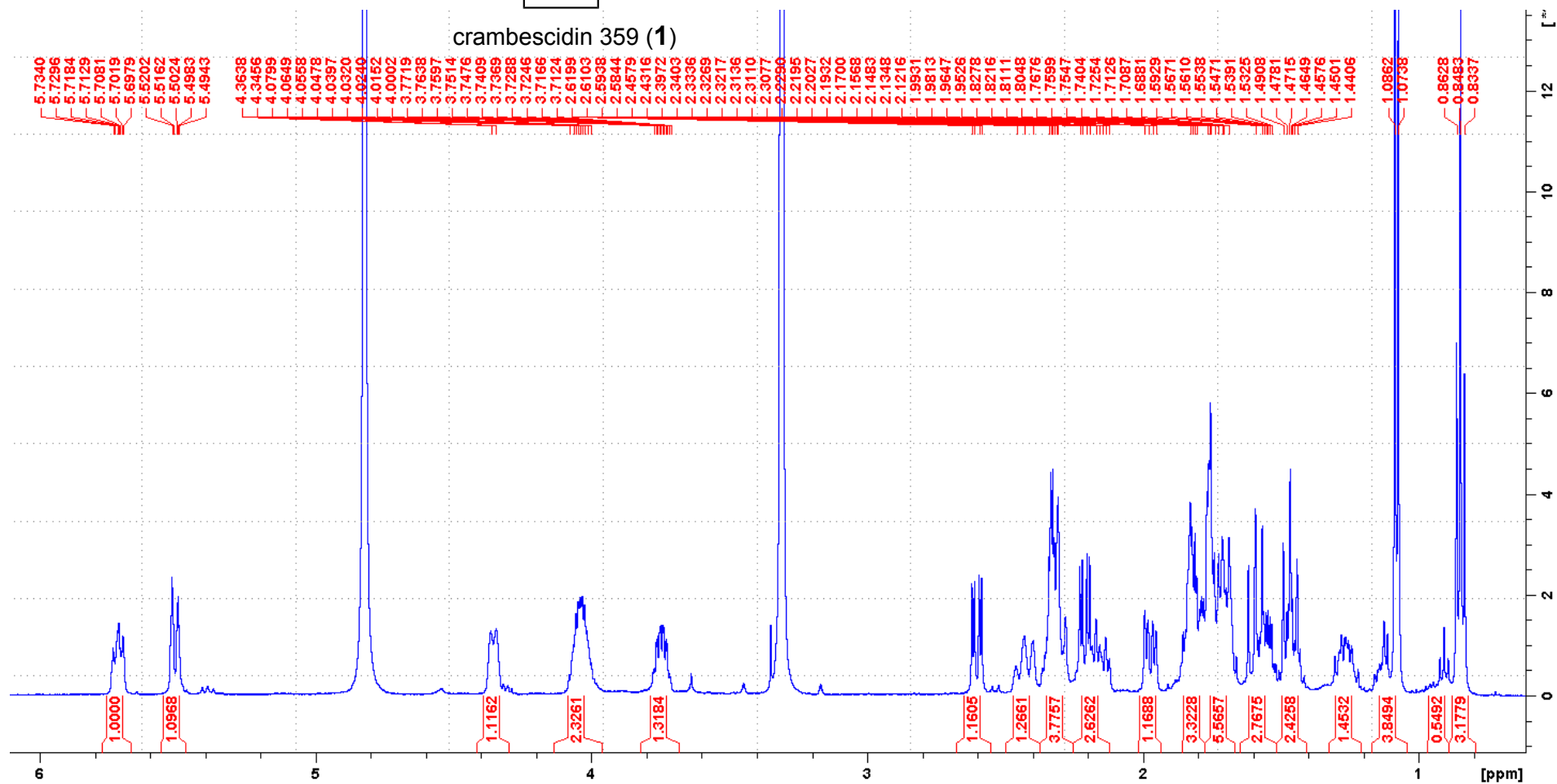
Minimum: -1.5
Maximum: 5.0 20.0 100.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
360.2652	360.2651	0.1	0.3	6.5	999.5	0.0	C21 H34 N3 O2
	360.2624	2.8	7.8	7.5	1006.0	6.4	C17 H30 N9
	360.2683	-3.1	-8.6	-1.5	1014.2	14.7	C10 H34 N9 O5
	360.2691	-3.9	-10.8	10.5	1007.1	7.5	C26 H34 N
	360.2611	4.1	11.4	2.5	1007.5	8.0	C16 H34 N5 O4
	360.2723	-7.1	-19.7	2.5	1008.8	9.3	C15 H34 N7 O3

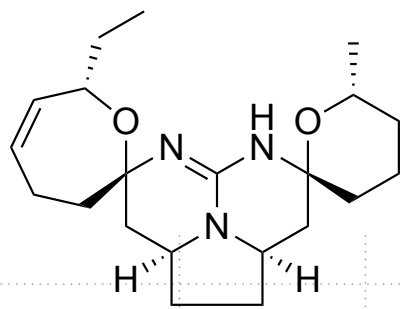
S1- crambescidin 359 (1) HRESIMS



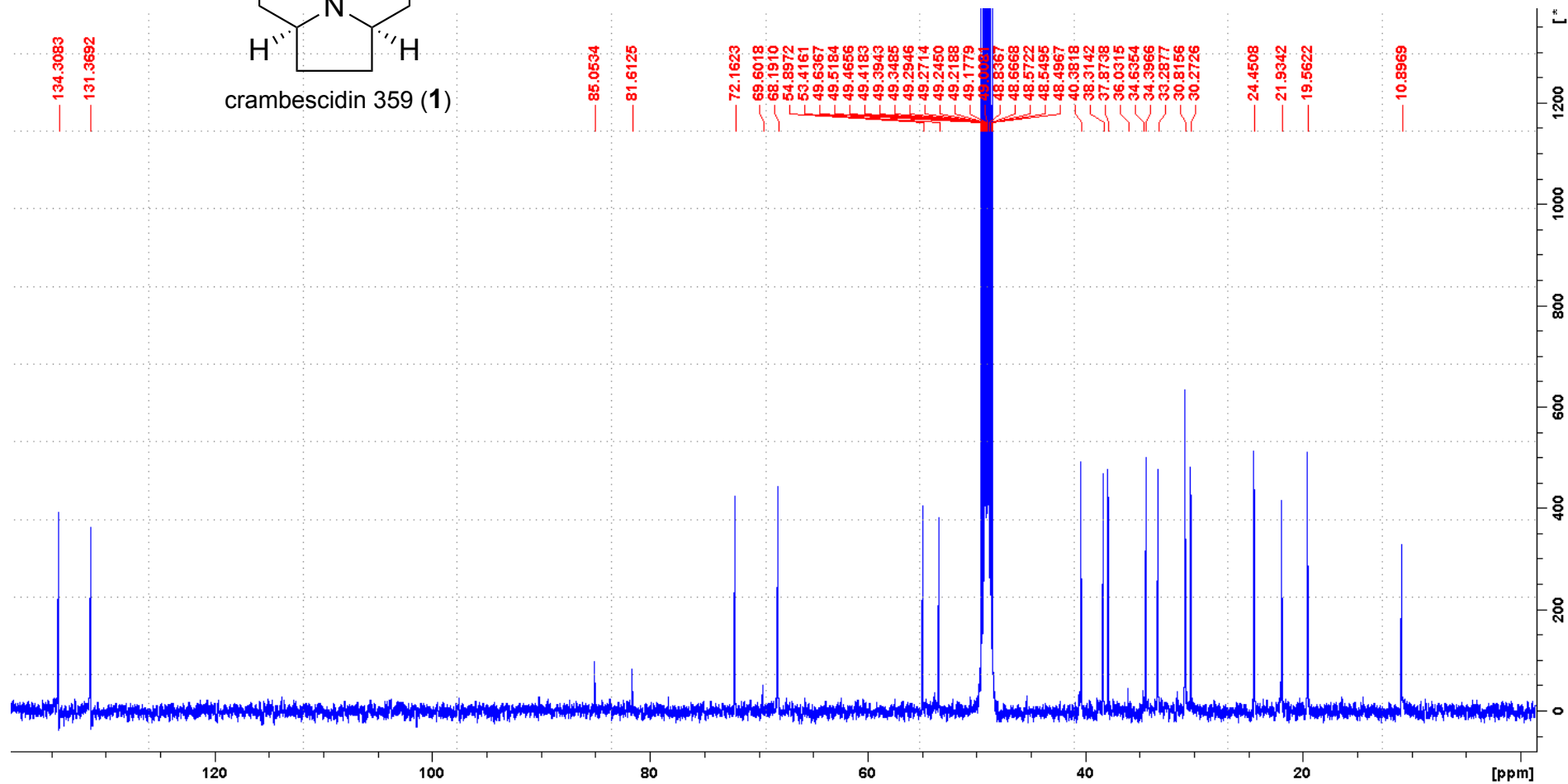
crambescidin 359 (1)



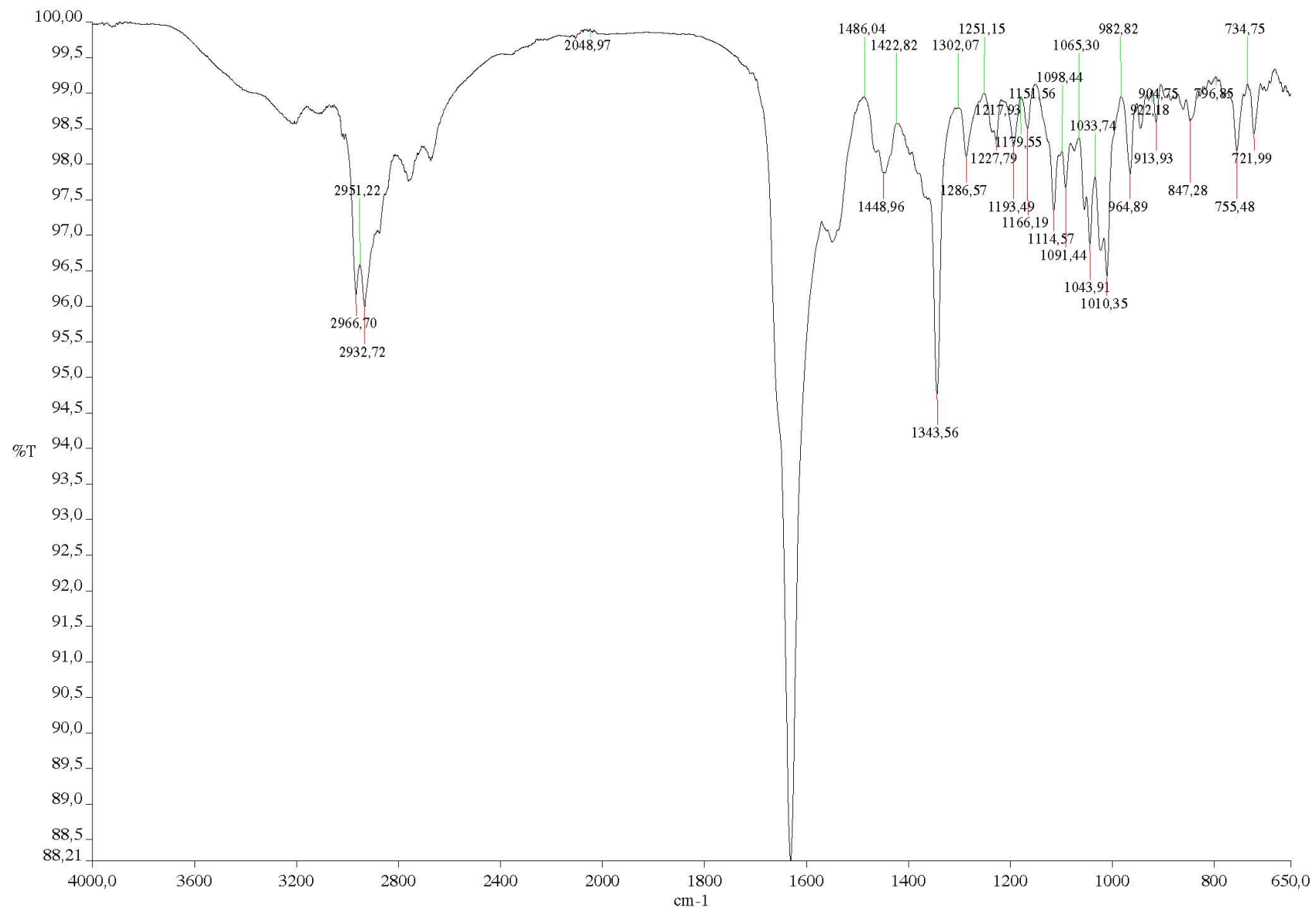
S2- crambescidin 359 (1) ¹H-NMR spectrum (500 MHz, MeOD)



crambescidin 359 (1)



S3- crambescidin 359 (1) ^{13}C -NMR spectrum (125 MHz, MeOD)



S4- crambescidin 359 (1) IR spectrum

Elemental Composition Report

Single Mass Analysis

Tolerance = 10.0 PPM / DBE: min = -1.5, max = 100.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 9

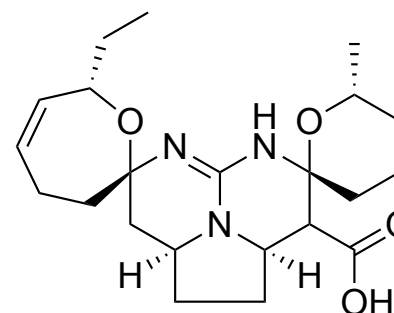
Monoisotopic Mass, Even Electron Ions

526 formula(e) evaluated with 5 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 0-50 H: 0-100 N: 0-10 O: 0-10

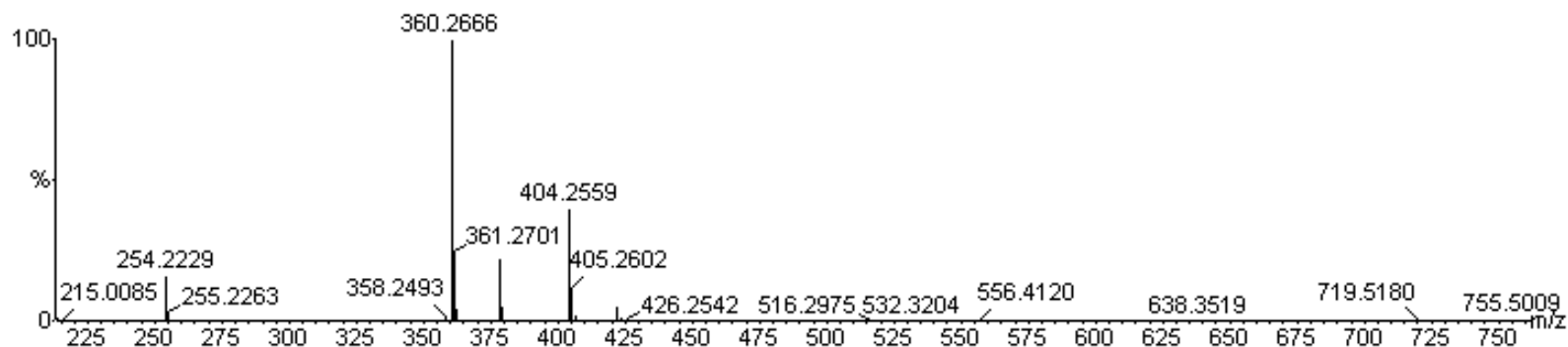
OUAZZANI_glegoff58-2 24 (0.625) Cm (20:28-39:65x2.000)



crambescidin acid (2)

Page 1

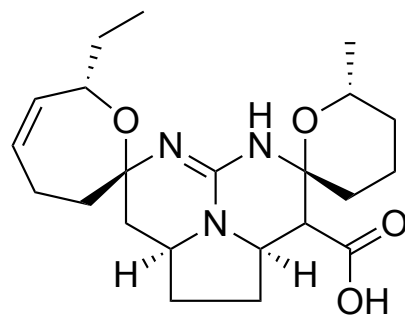
1: TOF MS ES+
4.26e+004



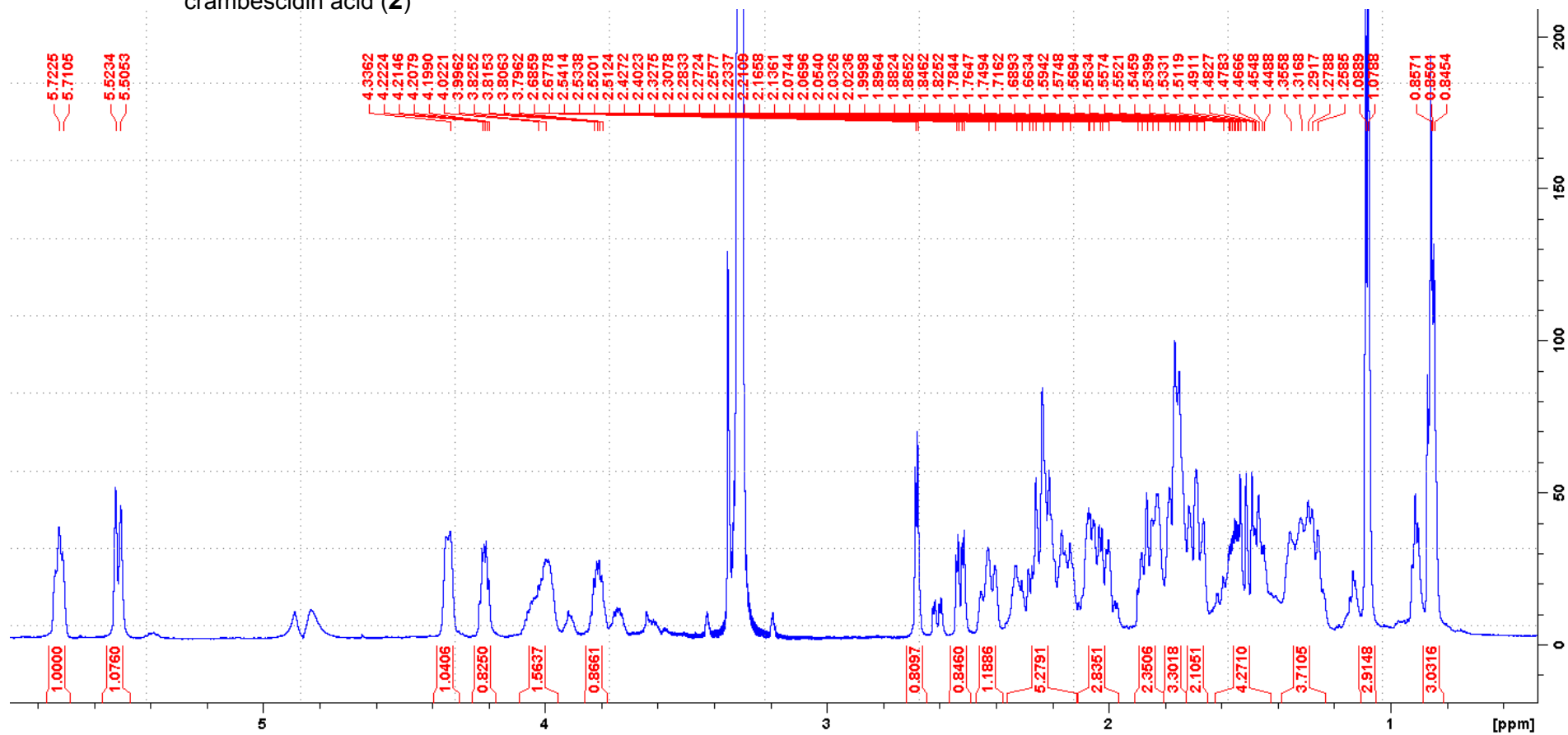
Minimum: -1.5
Maximum: 5.0 10.0 100.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
404.2559	404.2563	-0.4	-1.0	12.5	292.4	3.0	C23 H30 N7
	404.2549	1.0	2.5	7.5	289.5	0.1	C22 H34 N3 O4
	404.2581	-2.2	-5.4	-0.5	306.9	17.5	C11 H34 N9 O7
	404.2590	-3.1	-7.7	11.5	293.7	4.3	C27 H34 N O2
	404.2522	3.7	9.2	8.5	298.9	9.5	C18 H30 N9 O2

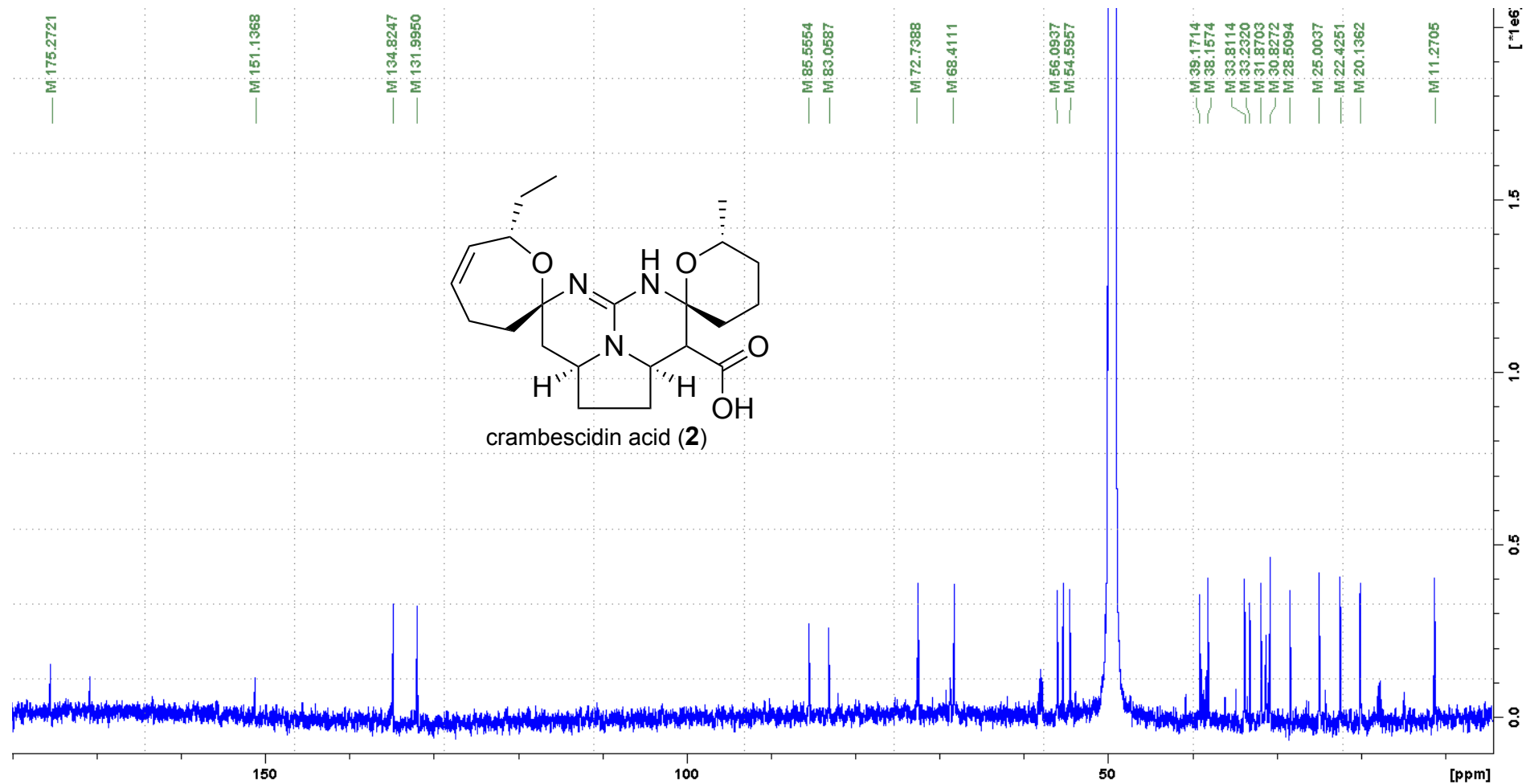
S5- crambescidin acid (2) HRESIMS



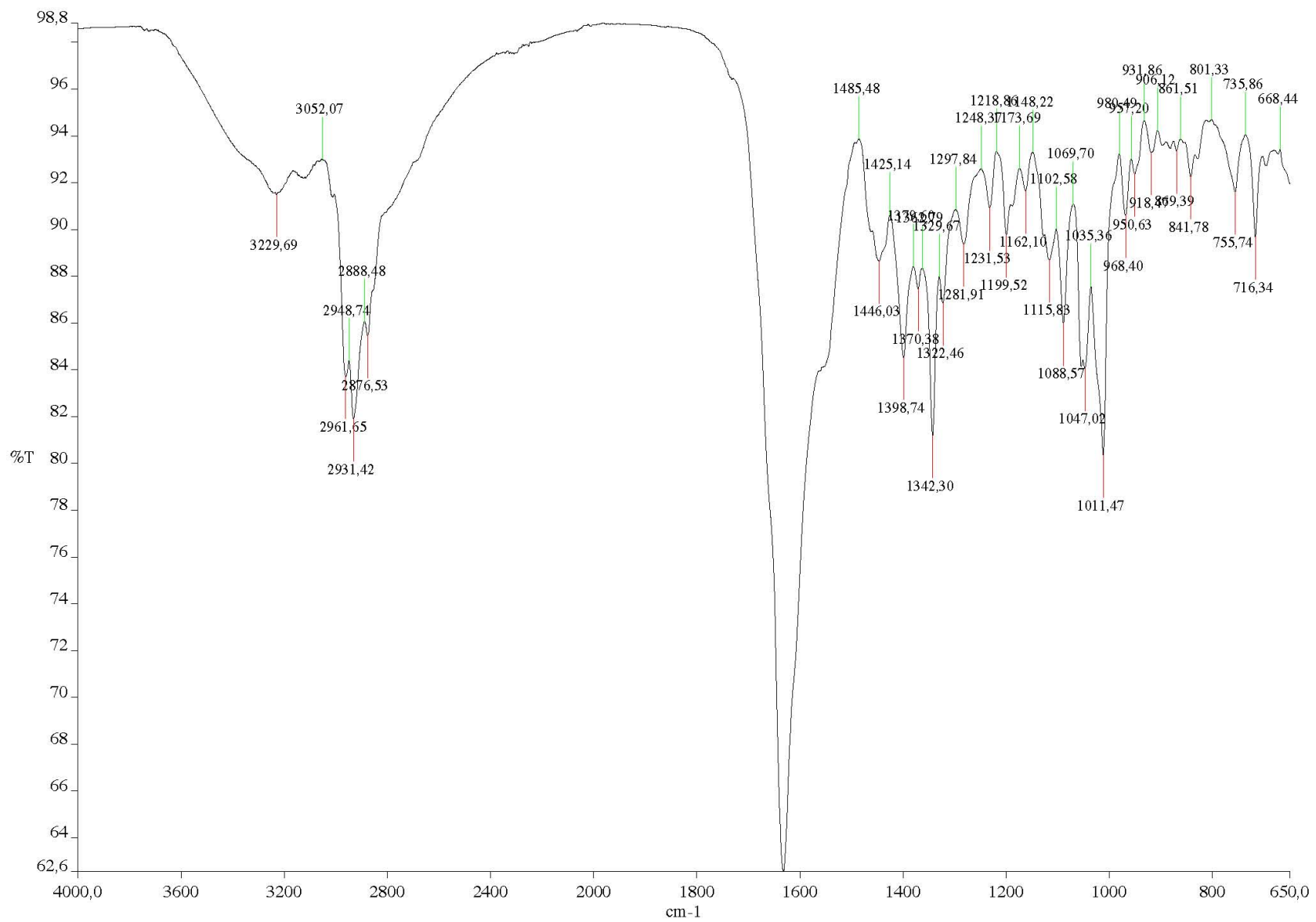
crambescidin acid (**2**)



S6- crambescidin acid (**2**) ¹H-NMR spectrum (600 MHz, MeOD)

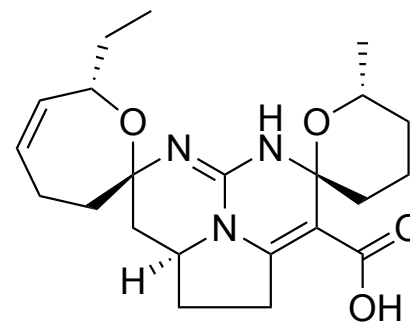


S7- crambescidin acid (2) ^{13}C -NMR spectrum (150 MHz, MeOD)



c:\pel_data\spectra\eq95\crambescidin 403.001

S8- crambescidin acid (2) IR spectrum



crambescidin 401 (3)

Elemental Composition Report

Single Mass Analysis

Tolerance = 10.0 PPM / DBE: min = -1.5, max = 100.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 9

Monoisotopic Mass, Even Electron Ions

519 formula(e) evaluated with 4 results within limits (all results (up to 1000) for each mass)

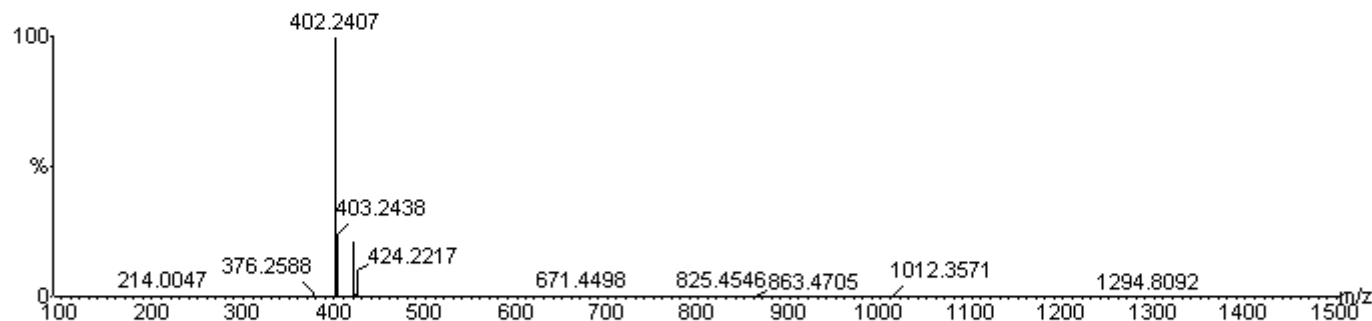
Elements Used:

C: 0-50 H: 0-100 N: 0-10 O: 0-10

OUAZZANI_glegoff58-1 23 (0.607) Cm (18:28-34:67x2.000)

1: TOF MS ES+

4.44e+004



Minimum:

-1.5

Maximum:

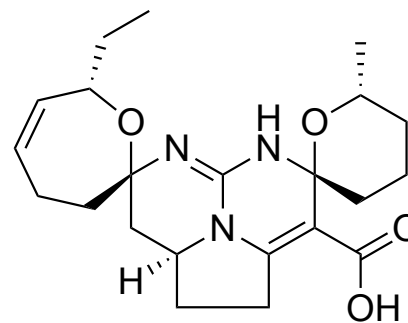
5.0

10.0

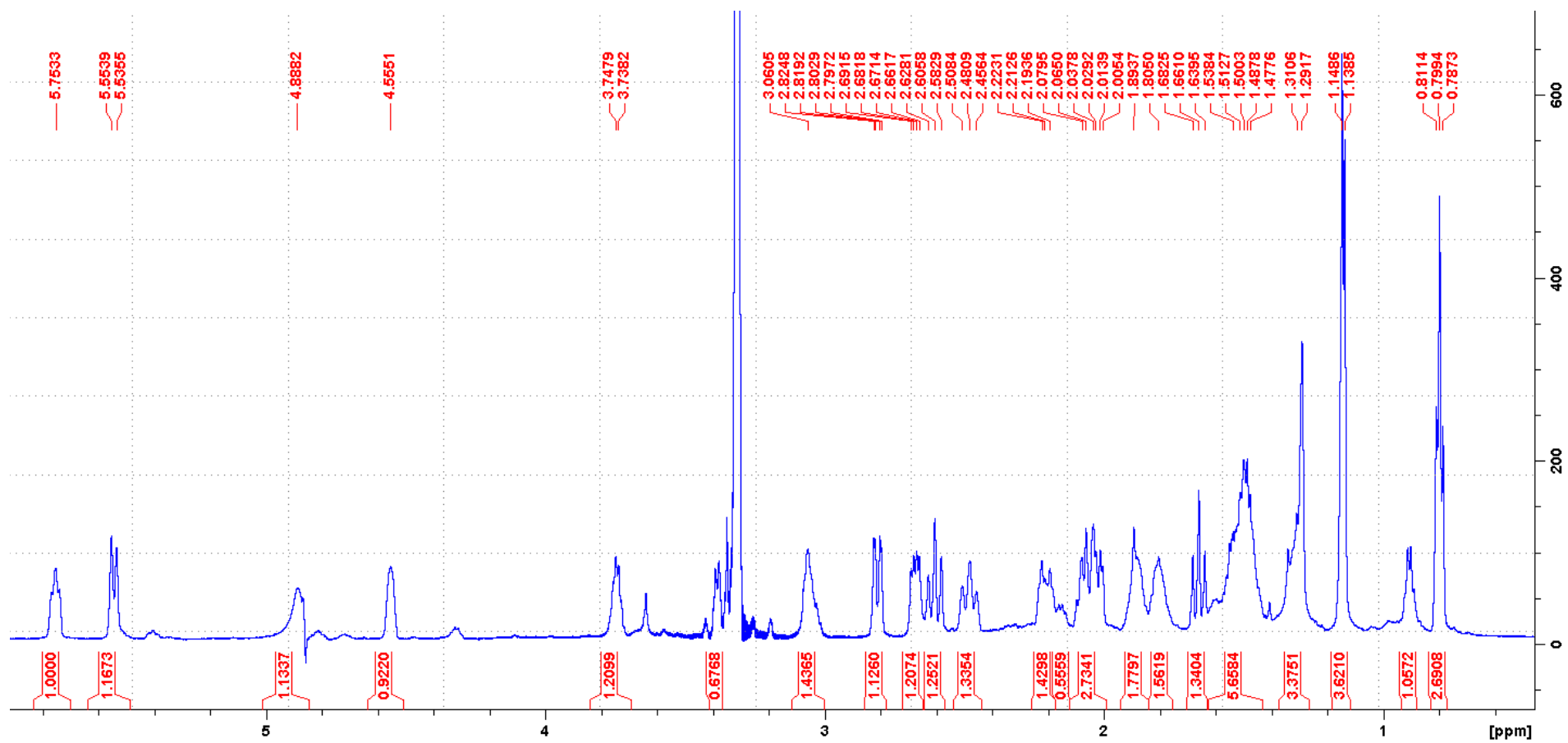
100.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
402.2407	402.2406	0.1	0.2	13.5	520.2	3.4	C23 H28 N7
	402.2393	1.4	3.5	8.5	516.8	0.0	C22 H32 N3 O4
	402.2425	-1.8	-4.5	0.5	521.2	4.4	C11 H32 N9 O7
	402.2433	-2.6	-6.5	12.5	525.0	8.2	C27 H32 N O2

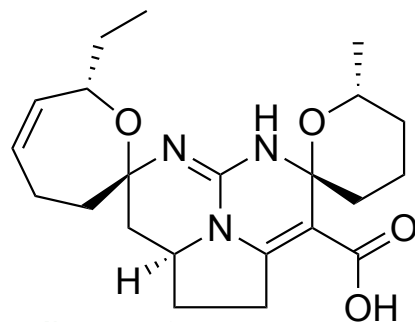
S9- crambescidin 401 (3) HRESIMS



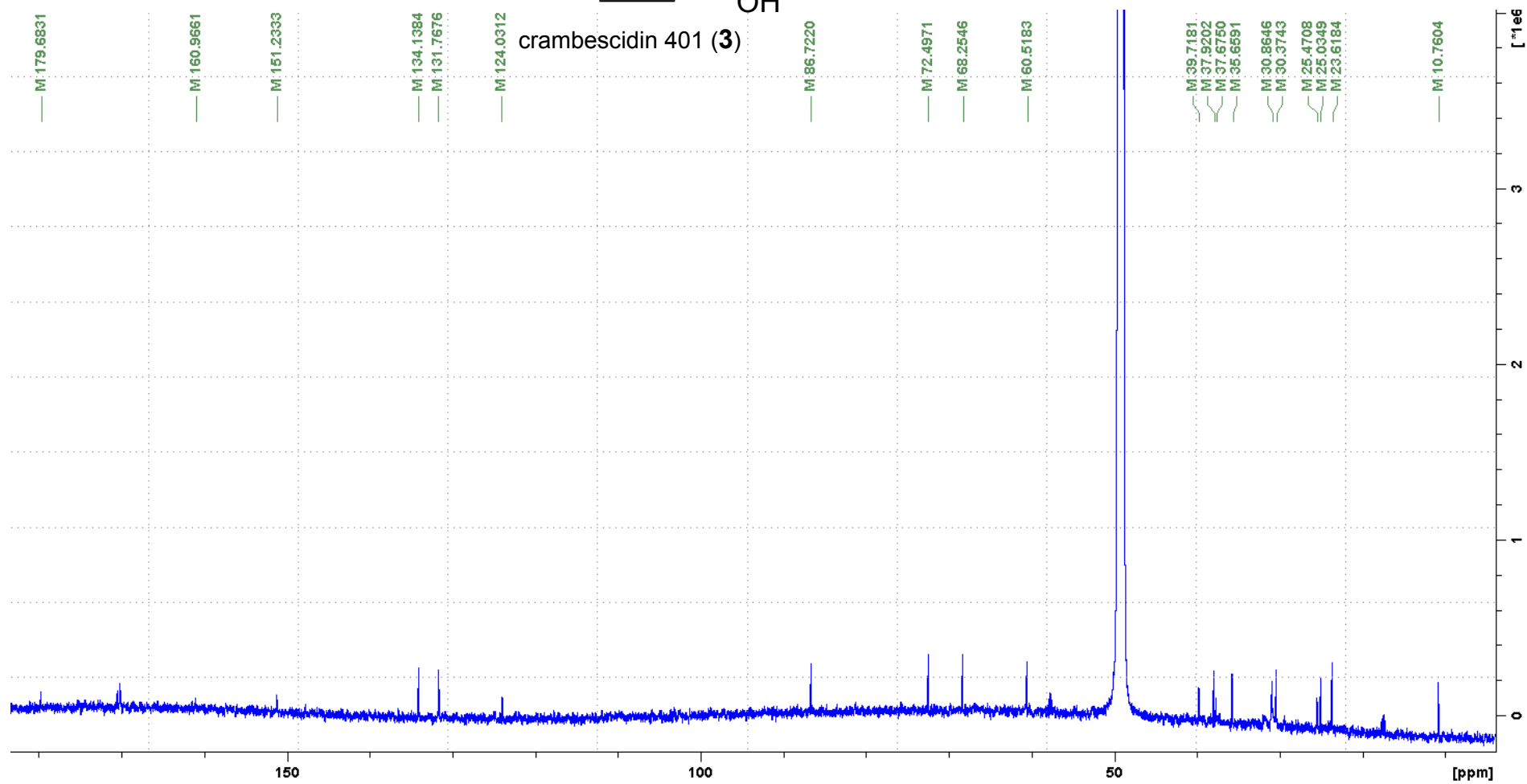
crambescidin 401 (**3**)



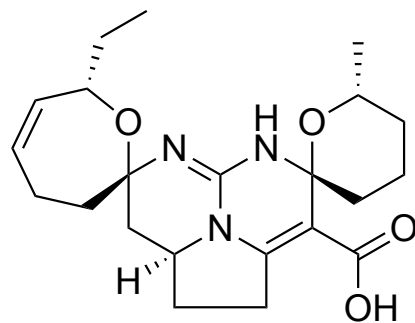
S10- crambescidin 401 (**3**) $^1\text{H-NMR}$ spectrum (600 MHz, MeOD)



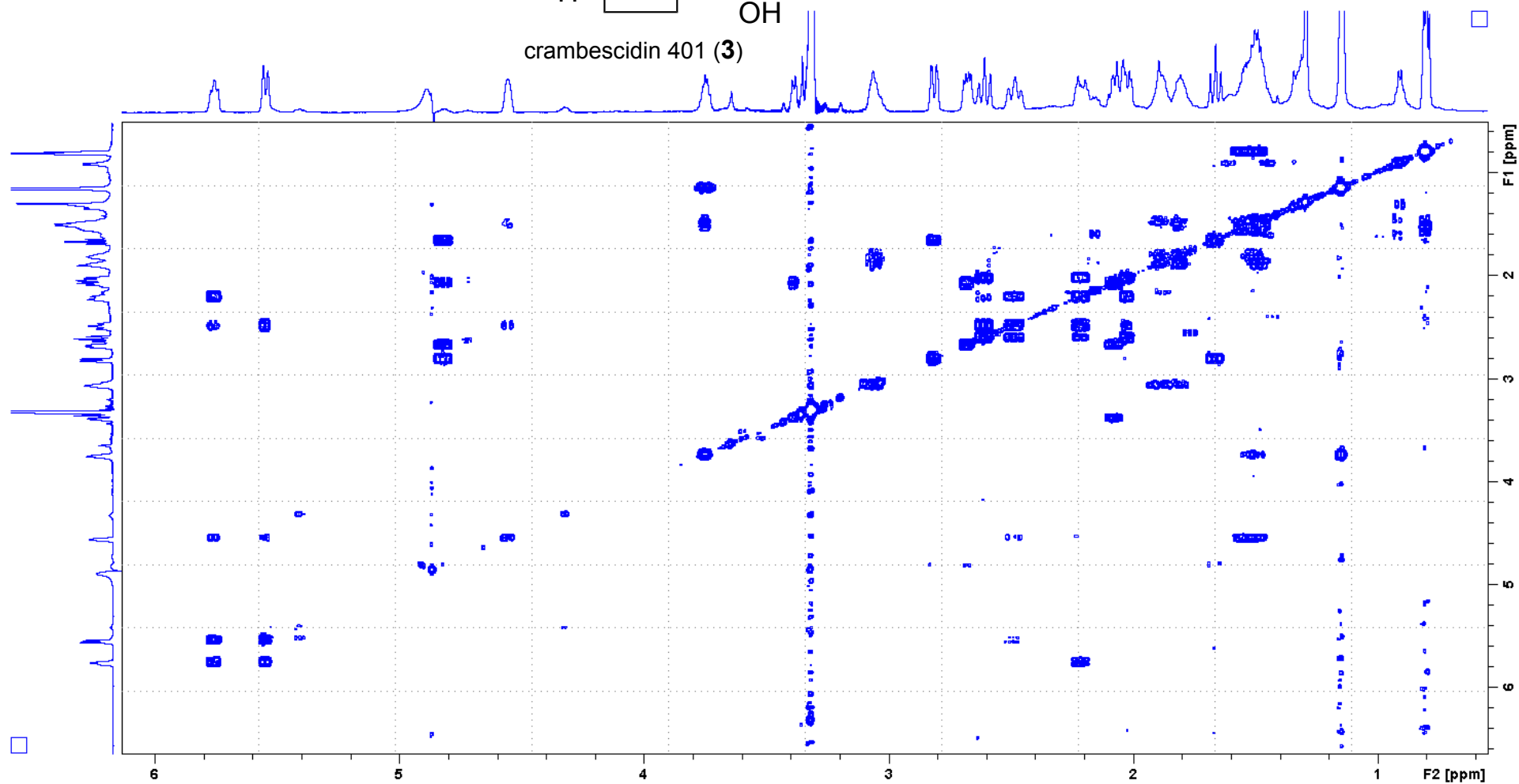
crambescidin 401 (**3**)



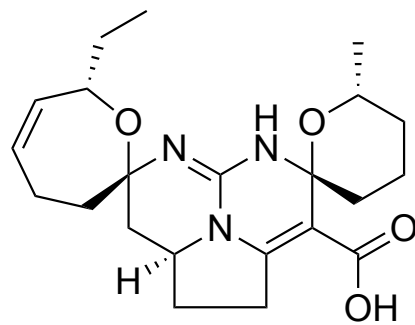
S11- crambescidin 401 (**3**) ^{13}C -NMR spectrum (150 MHz, MeOD)



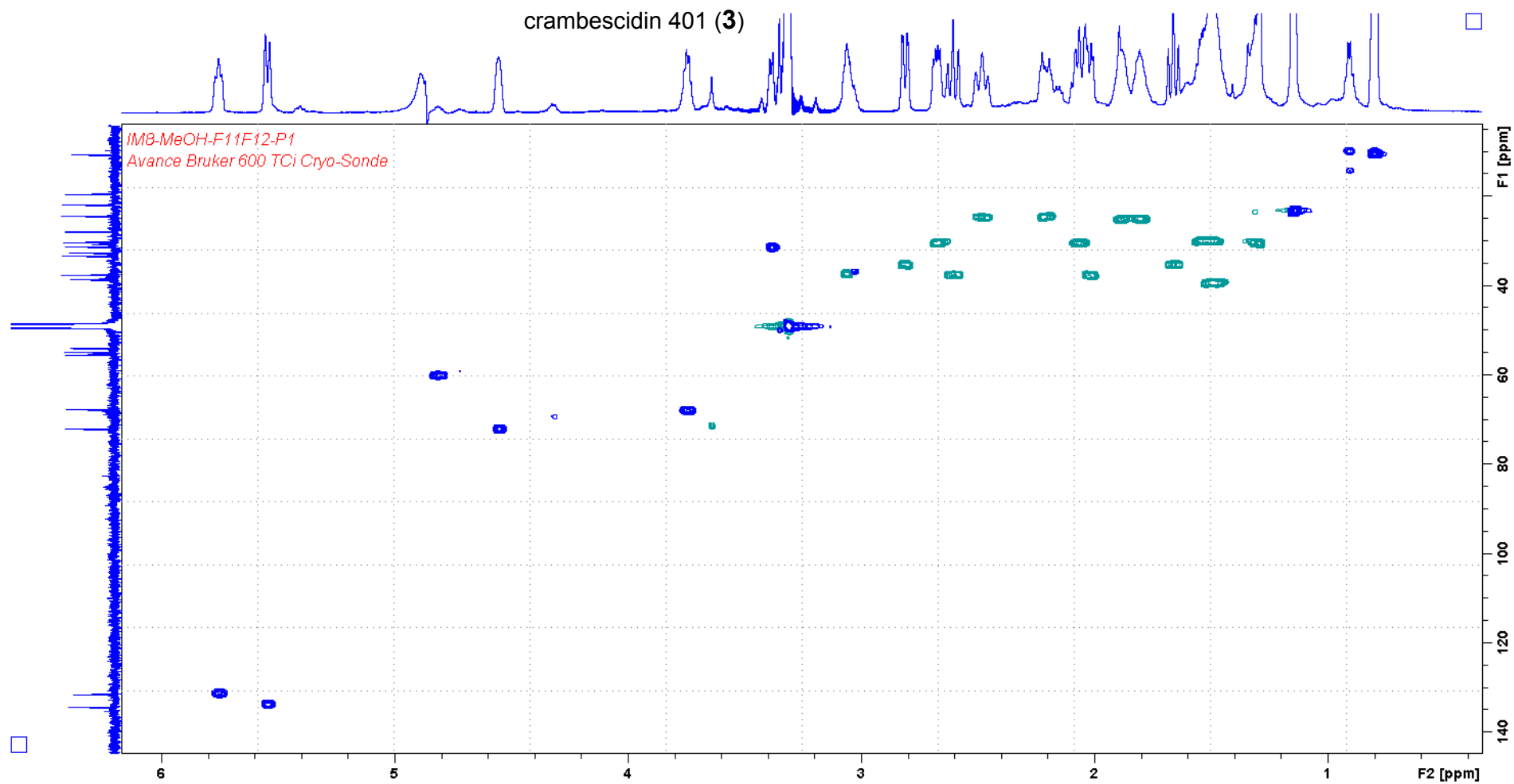
crambescidin 401 (3)



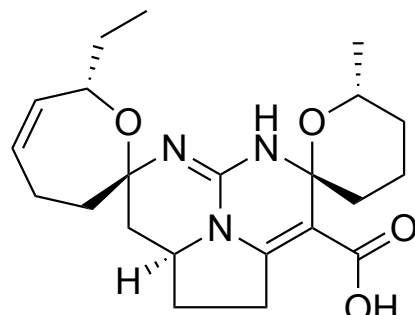
S12- crambescidin 401 (3) ^1H - ^1H COSY spectrum (600 MHz, MeOD)



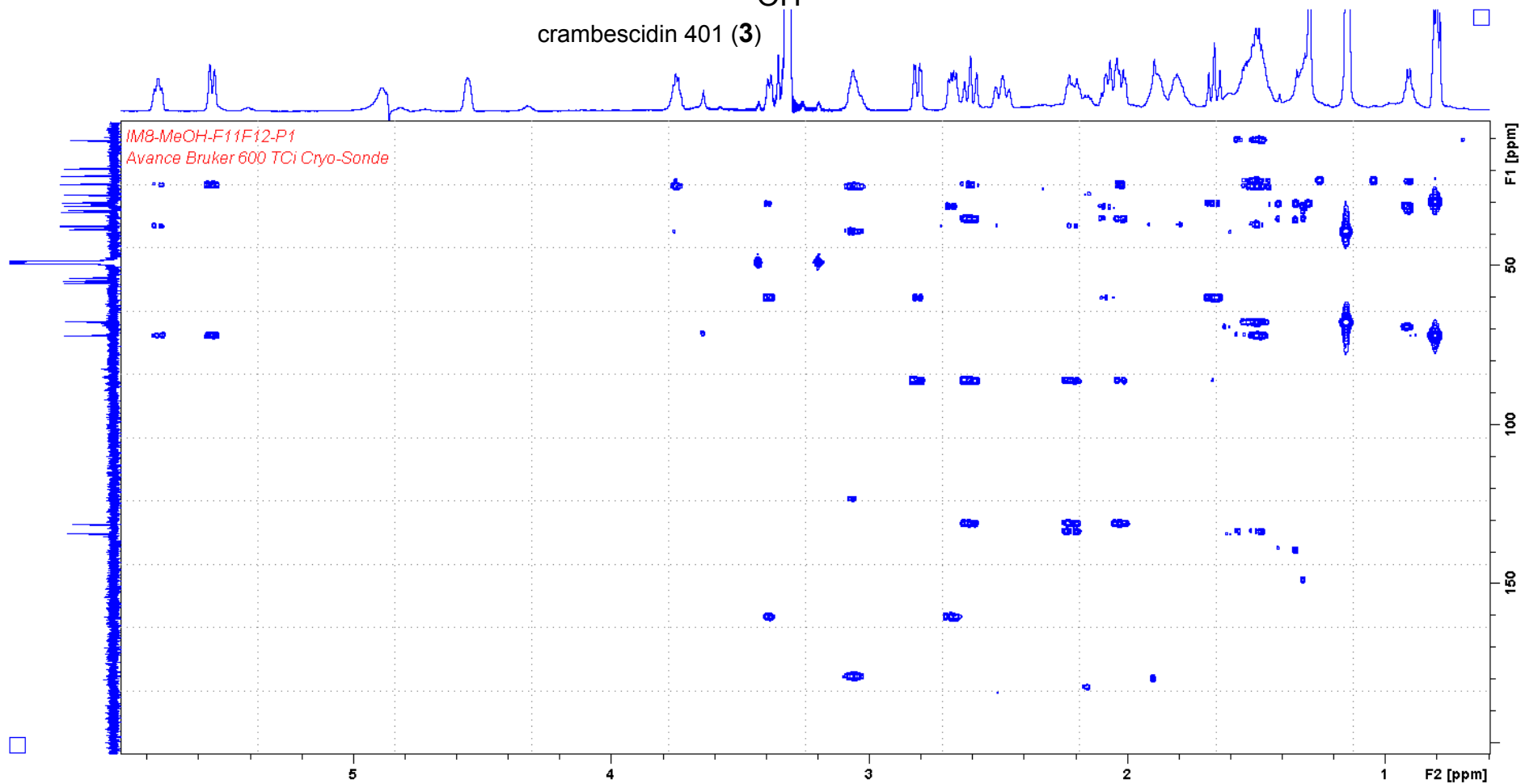
crambescidin 401 (3)



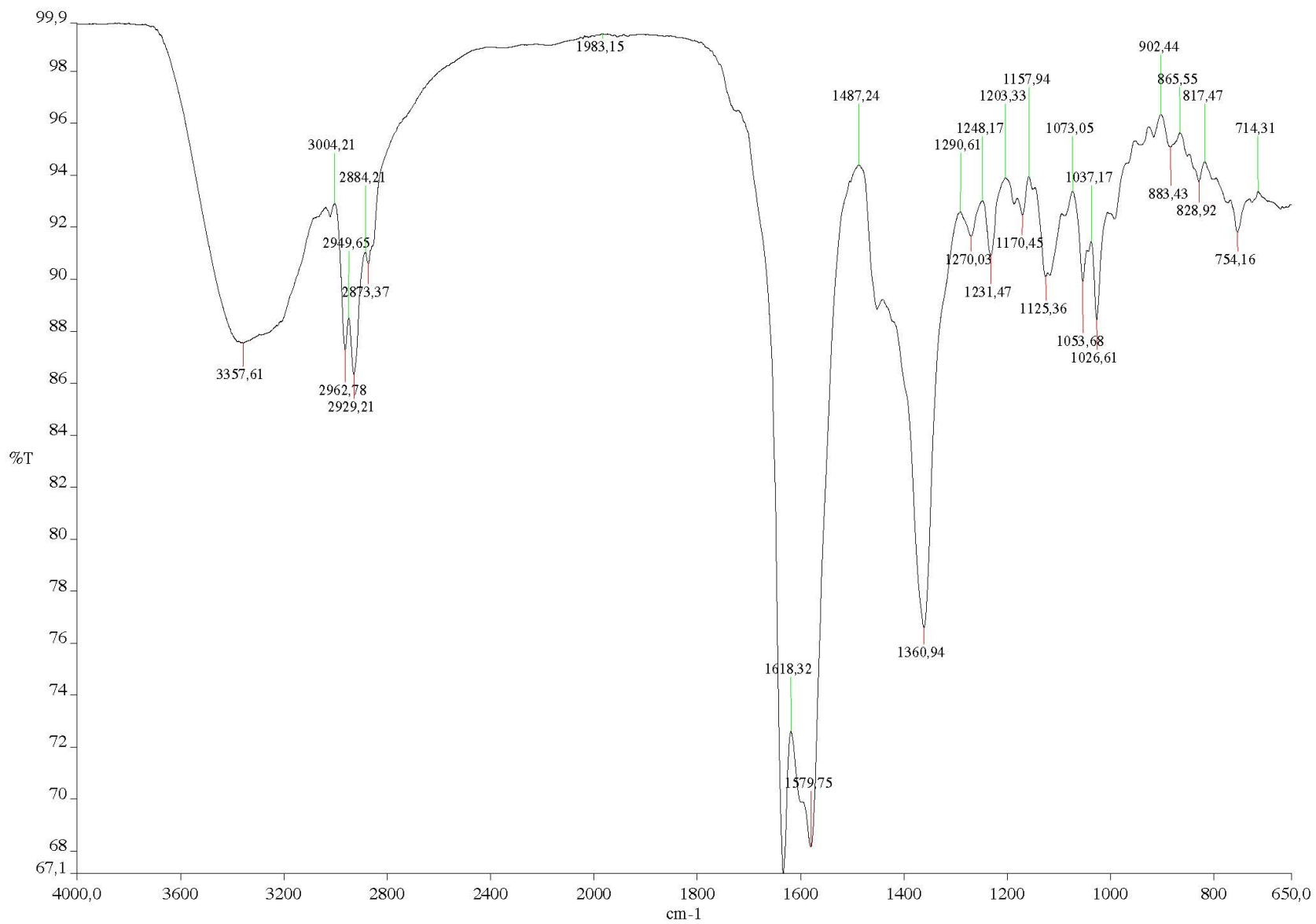
S13- crambescidin 401 (3) ^1H - ^{13}C HSQC spectrum (600 MHz, MeOH)



crambescidin 401 (3)



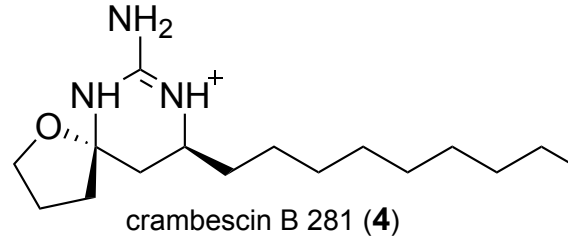
S14- crambescidin 401 (3) ^1H - ^{13}C HMBC spectrum (600 MHz, MeOD)



c:\pel_data\spectra\eq95\crambescidin 401.001

S15- crambescidin 401 (3) IR spectrum

Elemental Composition Report



Page 1

Single Mass Analysis

Tolerance = 20.0 PPM / DBE: min = -1.5, max = 100.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 9

Monoisotopic Mass, Even Electron Ions

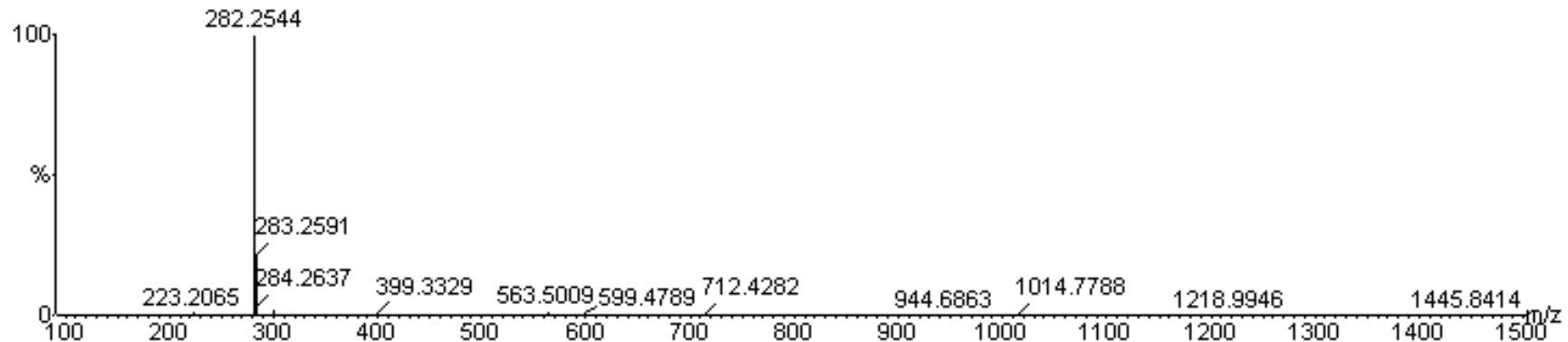
344 formula(e) evaluated with 2 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 0-50 H: 0-100 N: 0-10 O: 0-10

OUAZZANI_glegoff57-4 22 (0.590) Cm (19:28-35:70x2.000)

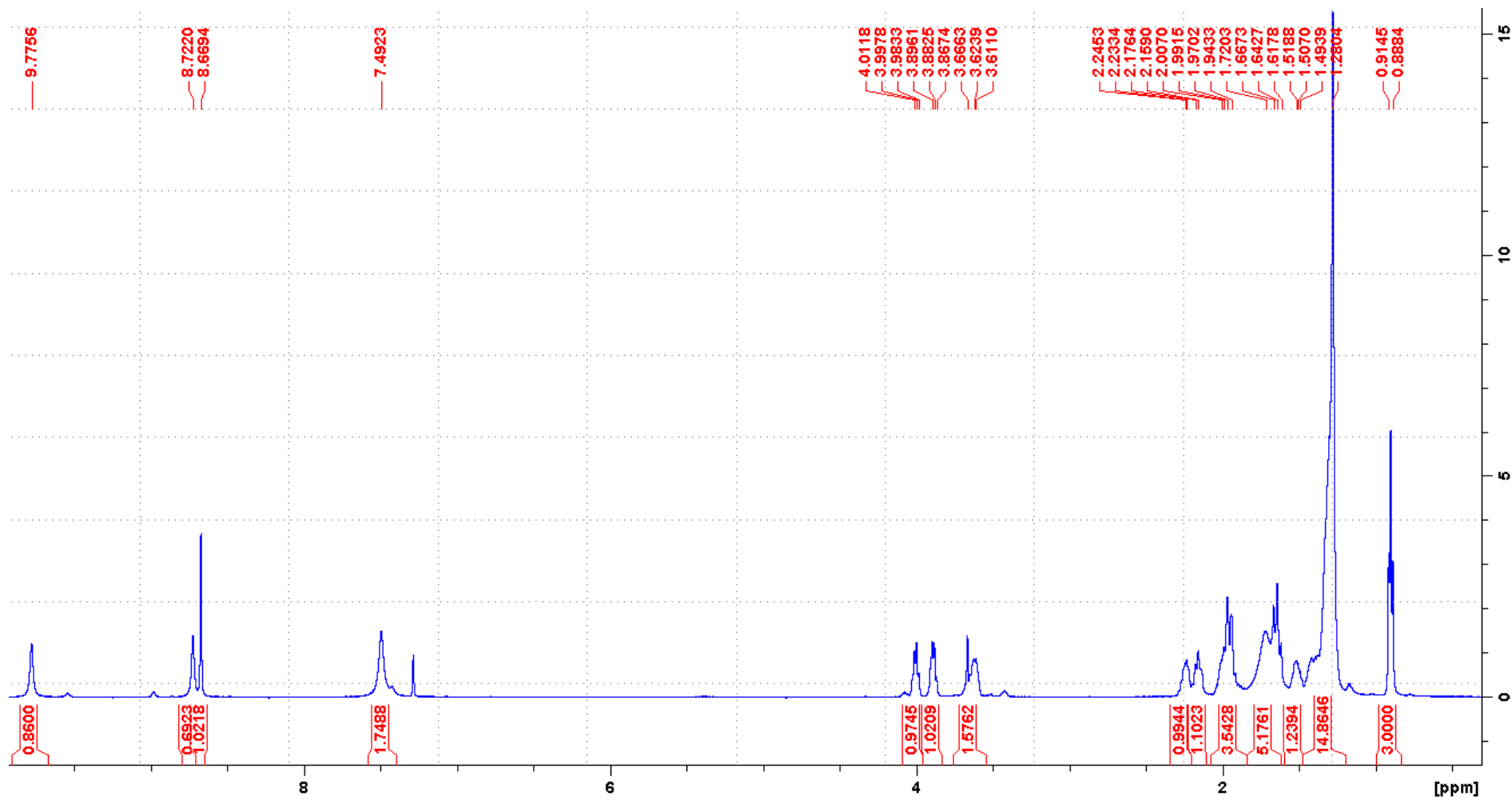
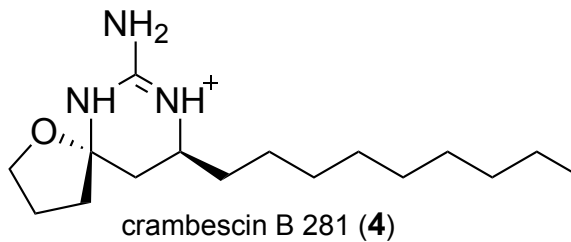
1: TOF MS ES+
2.26e+005



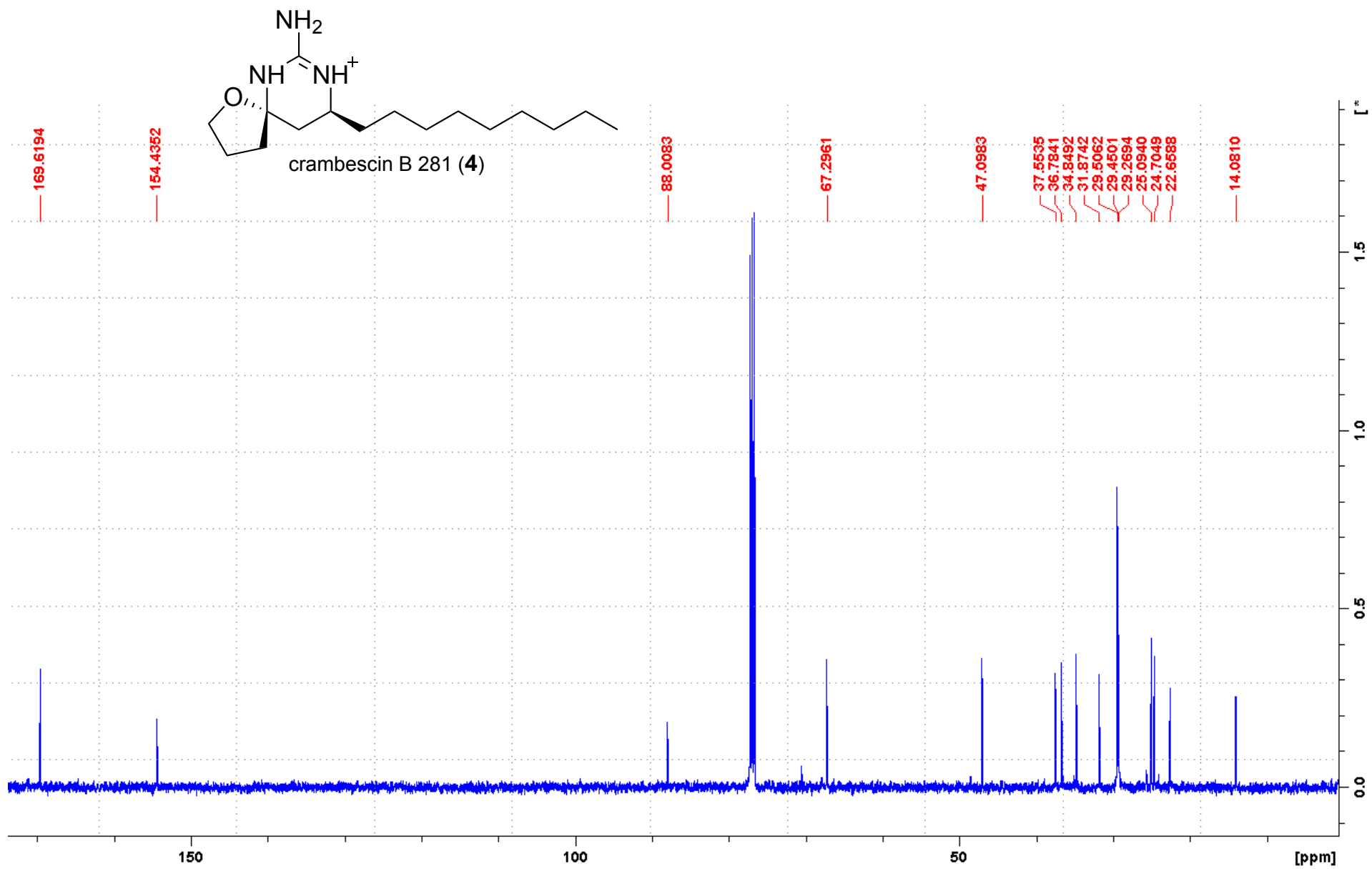
Minimum: -1.5
Maximum: 5.0 20.0 100.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
282.2544	282.2545	-0.1	-0.4	2.5	950.5	0.0	C16 H32 N3 O
	282.2505	3.9	13.8	-1.5	958.8	8.3	C11 H32 N5 O3

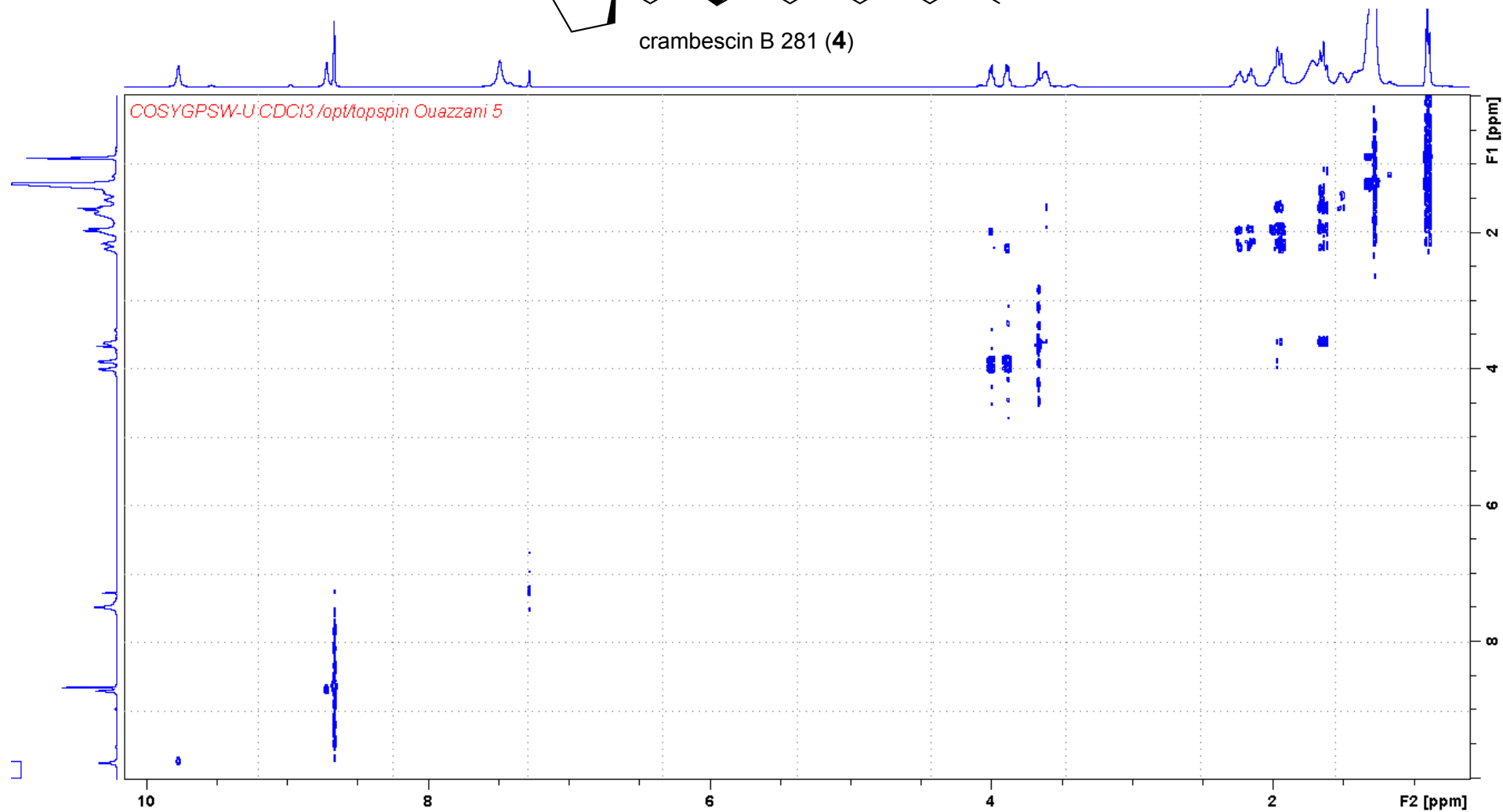
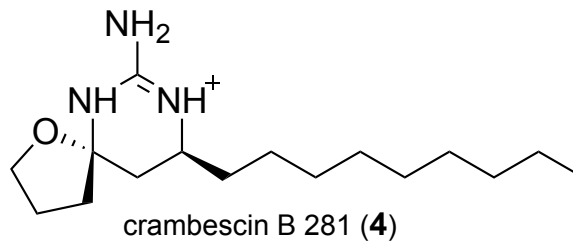
S16- crambescin B 281 (4) HRESIMS



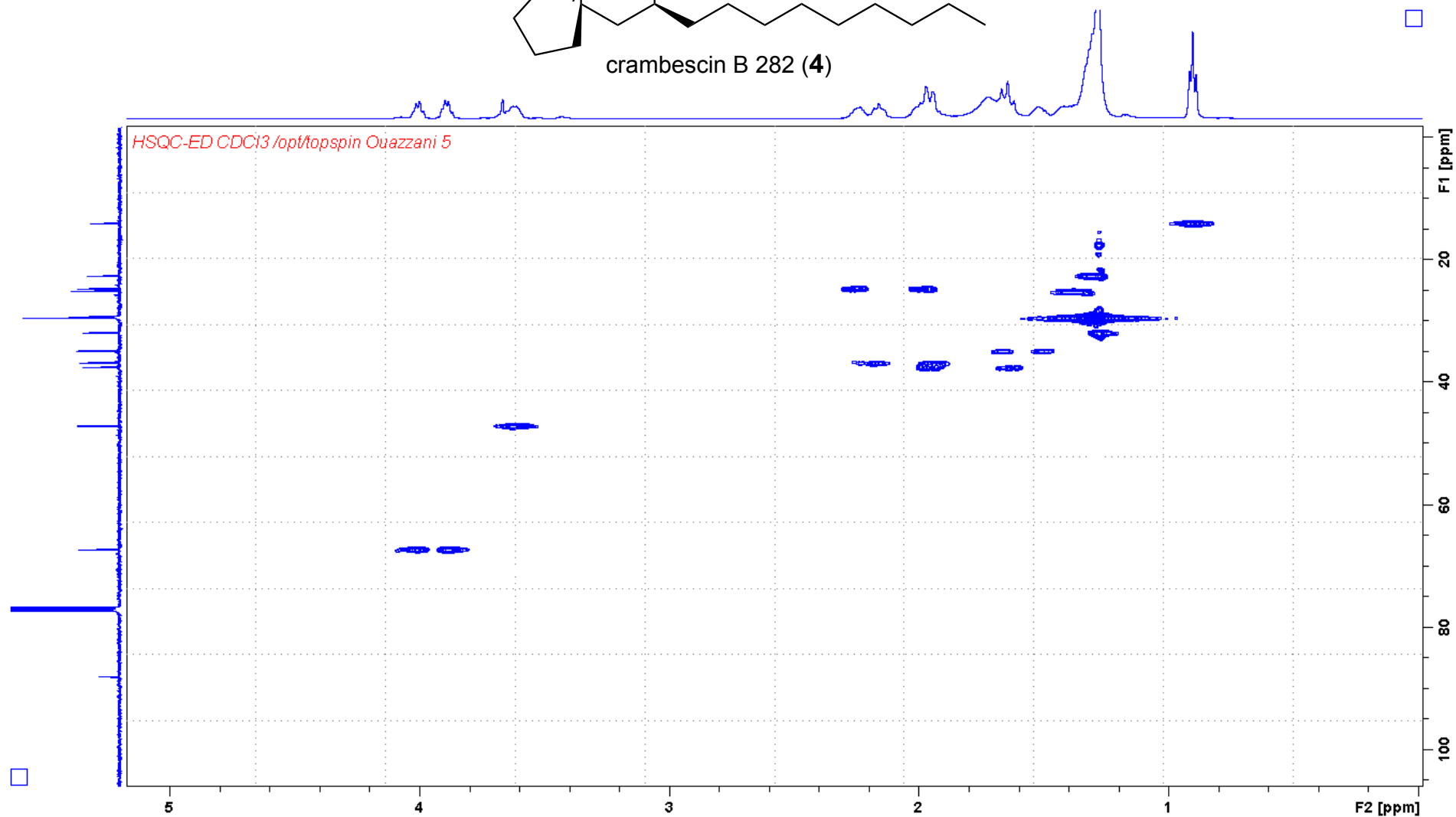
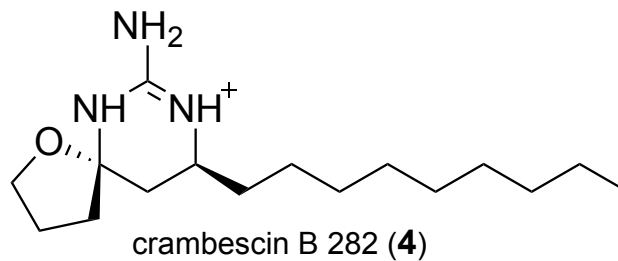
S17- crambescin B 281 (4) $^1\text{H-NMR}$ spectrum (500 MHz, CDCl_3)



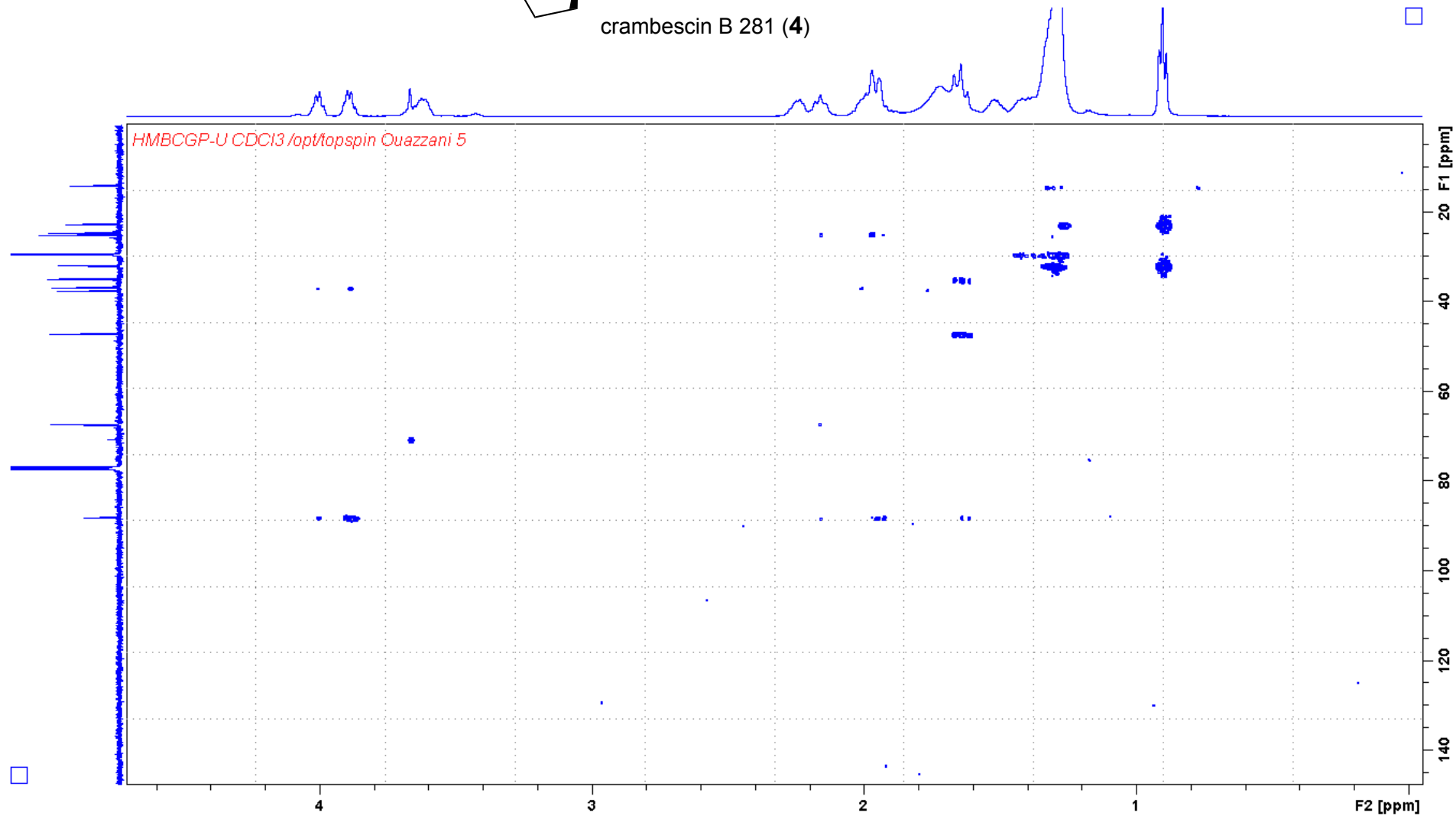
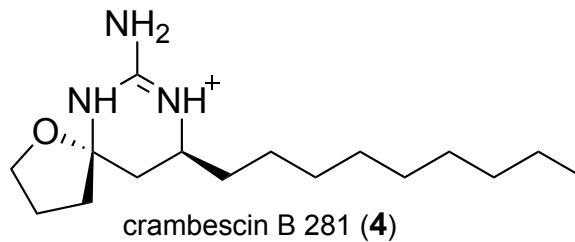
S18- crambescin B 281 (4) ¹³C-NMR spectrum (125 MHz, CDCl₃)



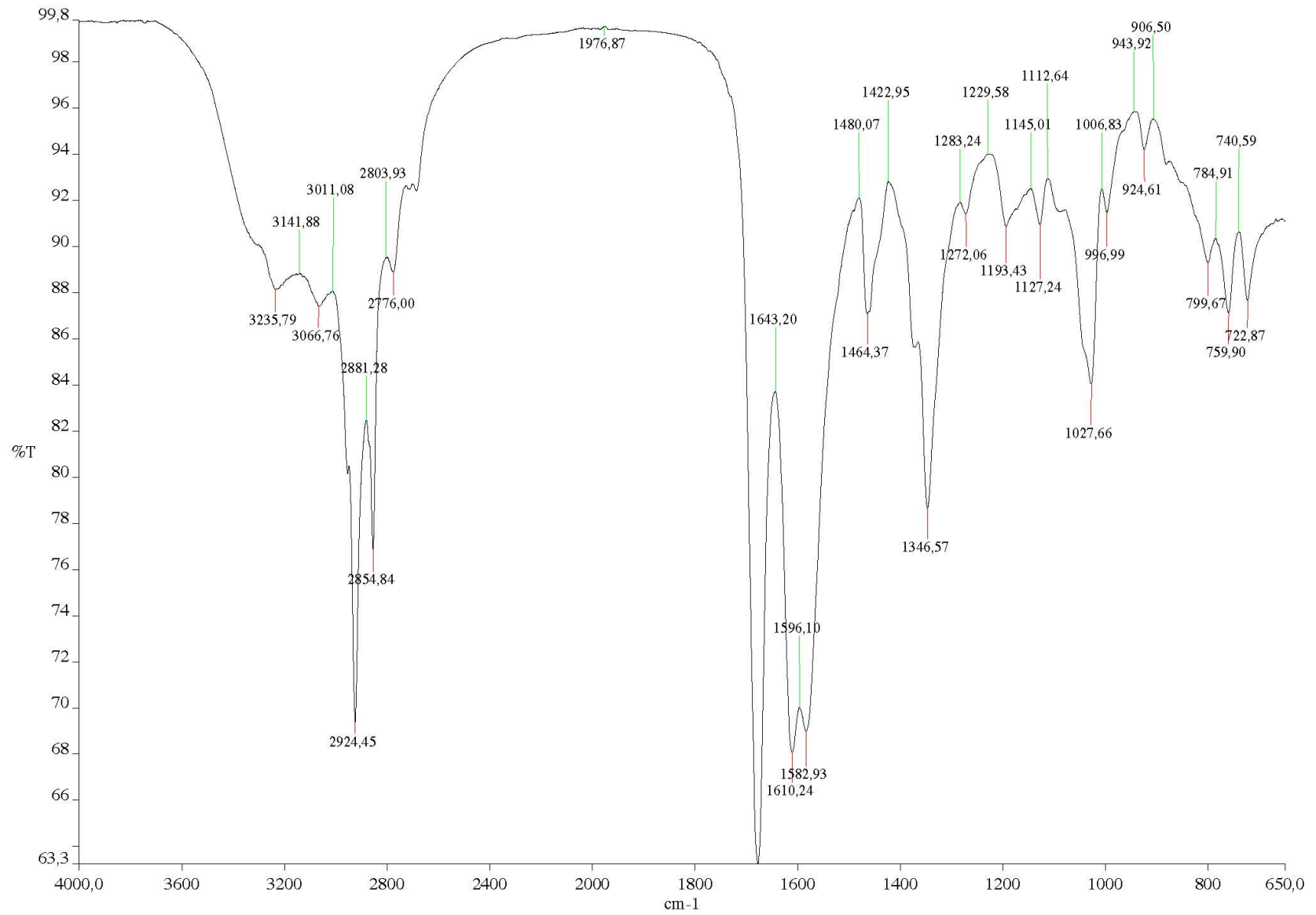
S19- crambescin B 281 (4) ¹H-¹H COSY spectrum (500 MHz, CDCl₃)



S20- crambescin B 282 (4) ¹H-¹³C HSQC spectrum (500 MHz, CDCl₃)



S21- crambescin B 281 (4) ¹H-¹³C HMBC spectrum (500 MHz, CDCl₃)



c:\pel_data\spectra\eq95\crambescin b281.001

S22- crambescin B 281 (4) IR spectrum

Elemental Composition Report

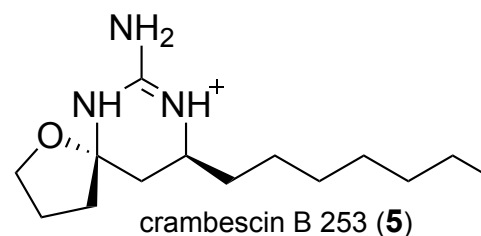
Page 1

Single Mass Analysis

Tolerance = 20.0 PPM / DBE: min = -1.5, max = 100.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 9



Monoisotopic Mass, Even Electron Ions

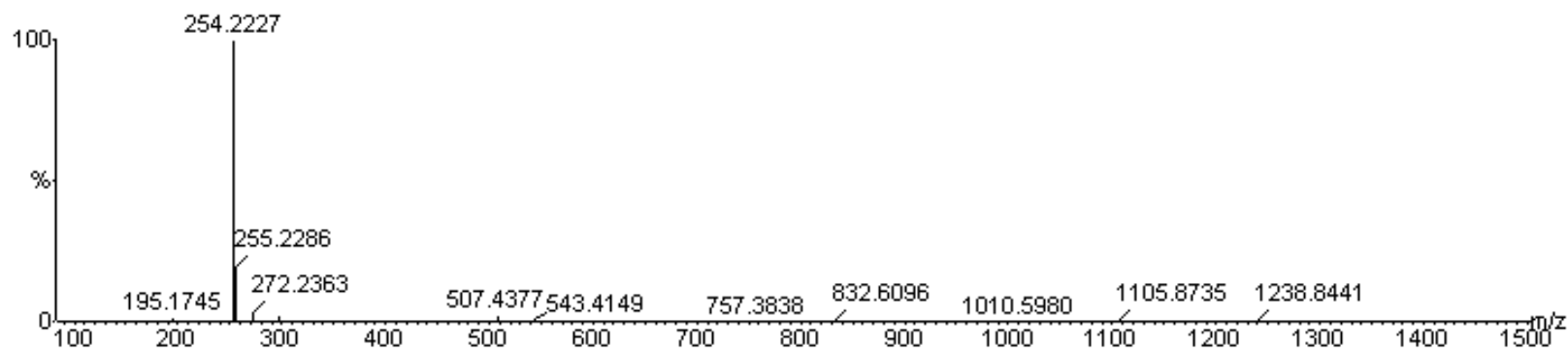
296 formula(e) evaluated with 2 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 0-50 H: 0-100 N: 0-10 O: 0-10

QUAZZANI_glegoff57-2 23 (0.606) Cm (19:27-41:68x2.000)

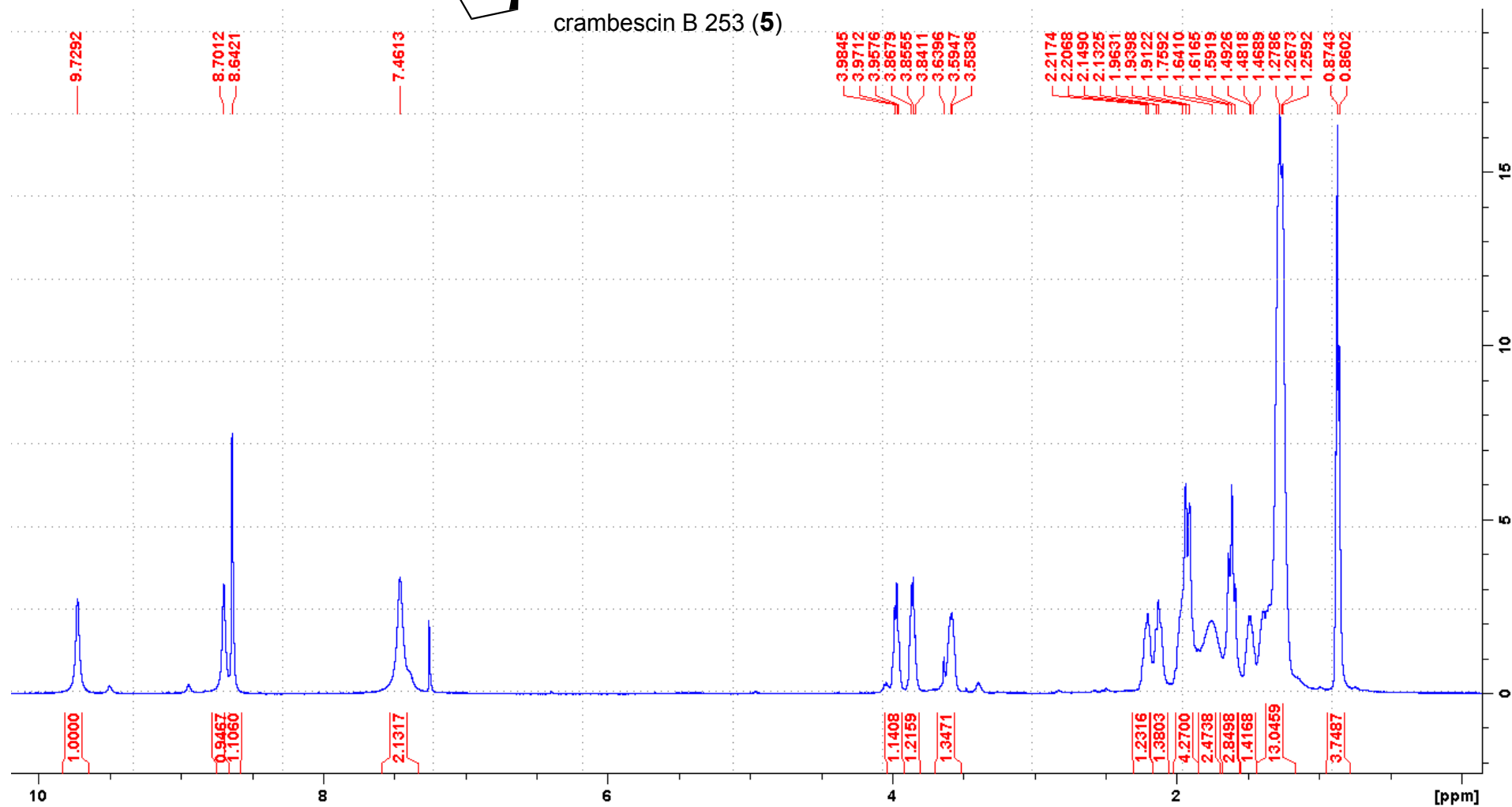
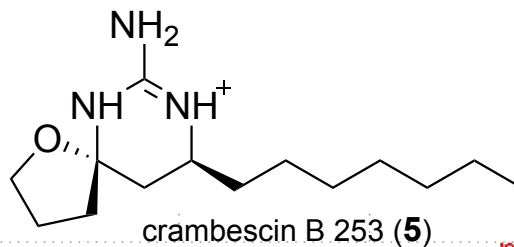
1: TOF MS ES+
2.58e+005



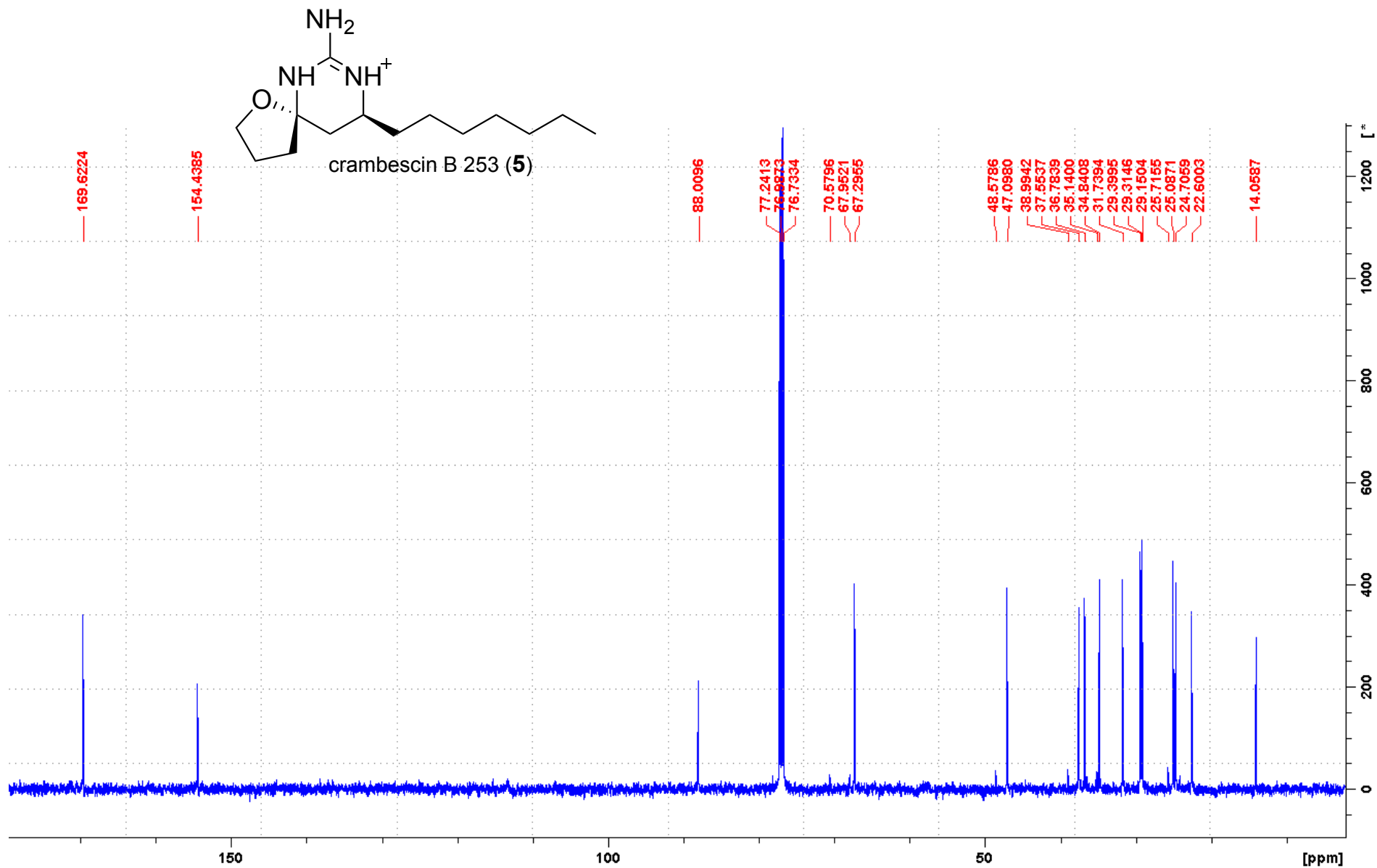
Minimum: -1.5
Maximum: 5.0 20.0 100.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
254.2227	254.2232	-0.5	-2.0	2.5	985.5	0.0	C14 H28 N3 O
	254.2192	3.5	13.8	-1.5	999.5	14.0	C9 H28 N5 O3

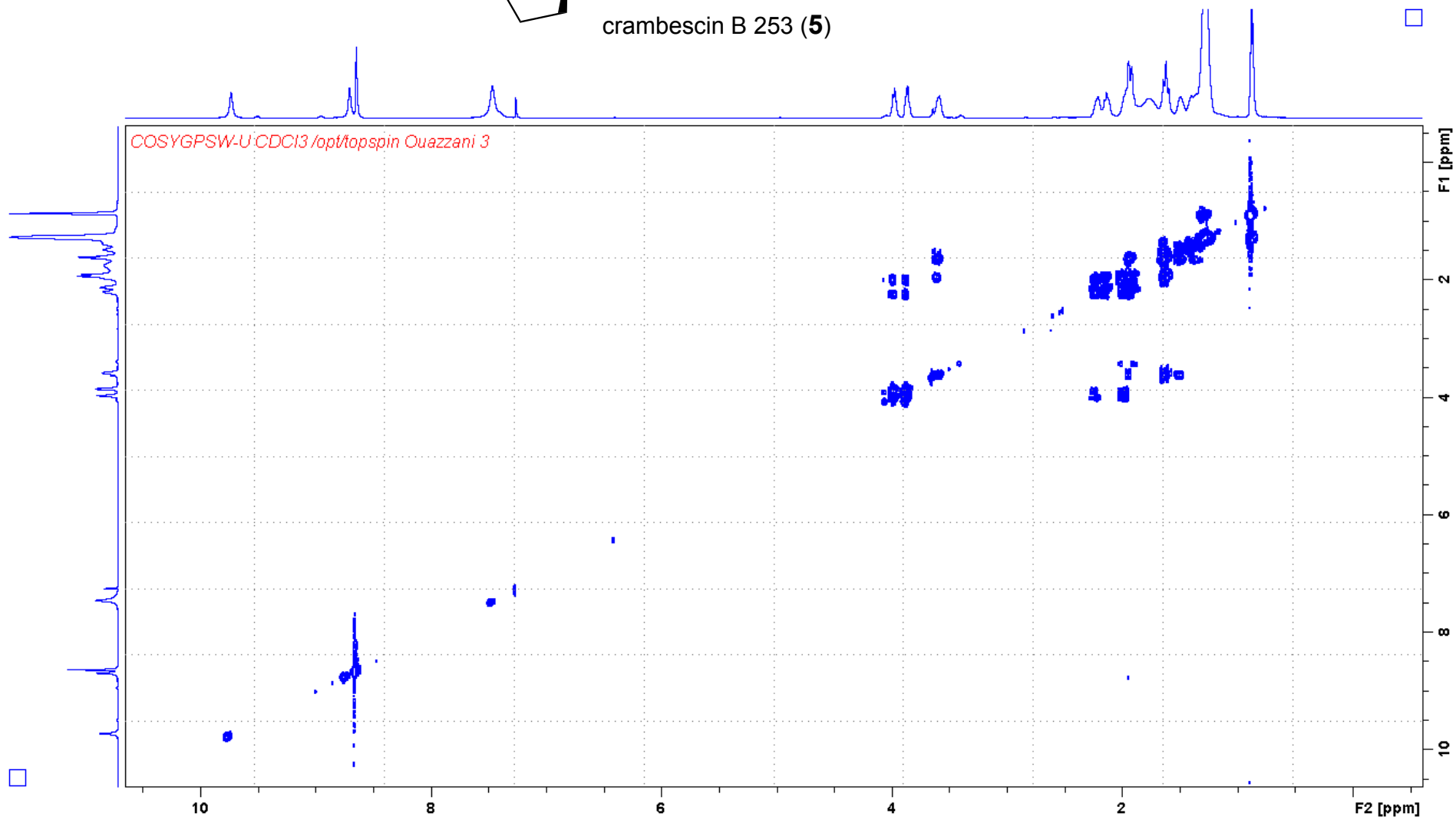
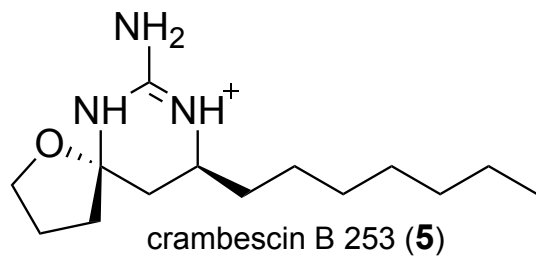
S23- crambescin B 253 (5) HRESIMS



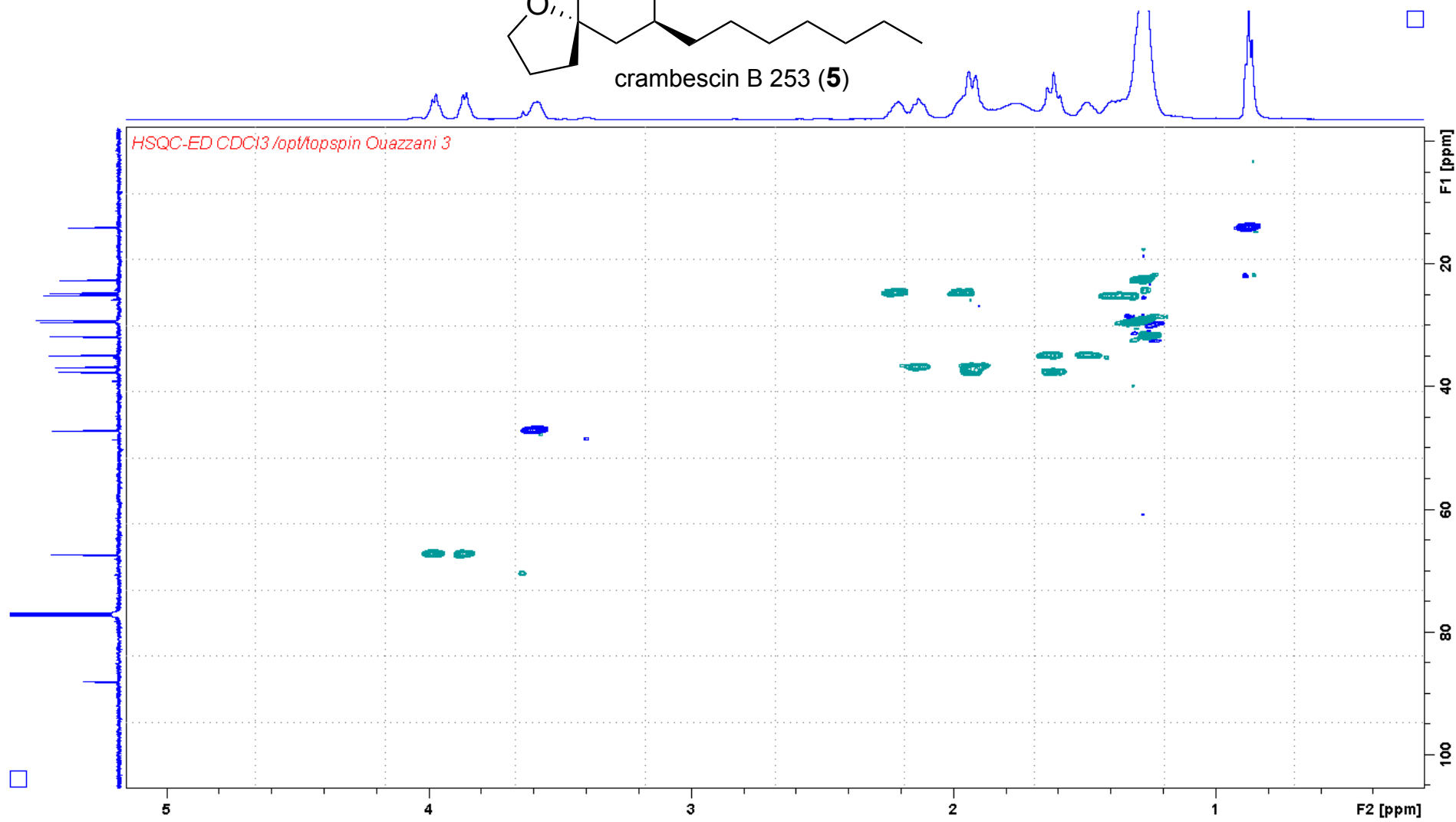
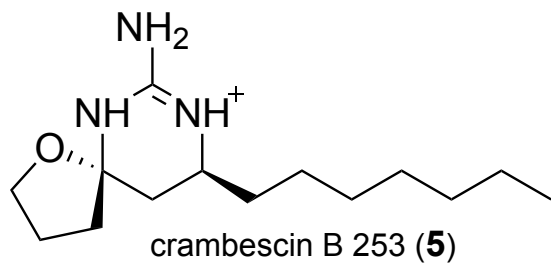
S24- crambescin B 253 (**5**) ¹H-NMR spectrum (500 MHz, CDCl₃)



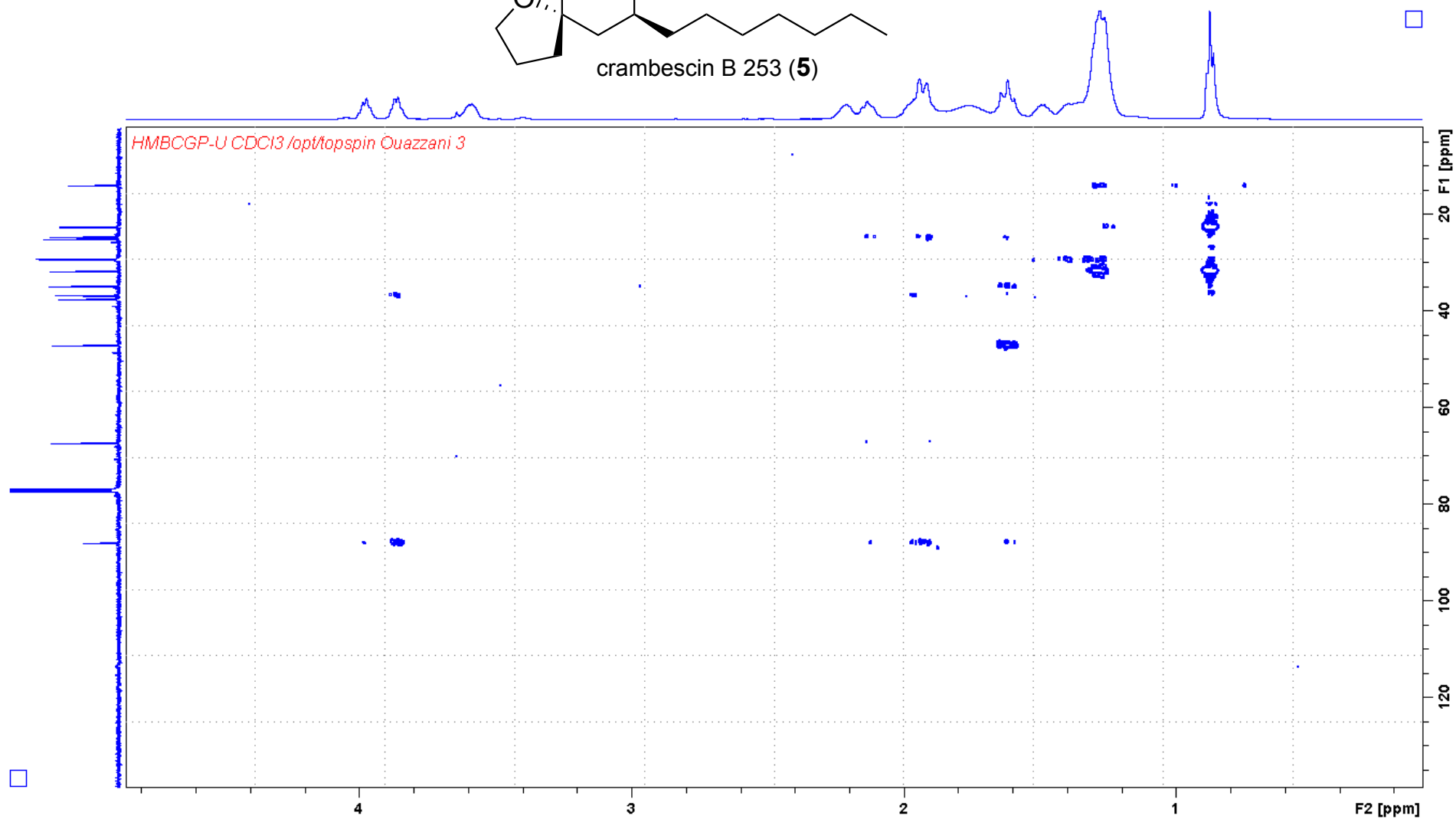
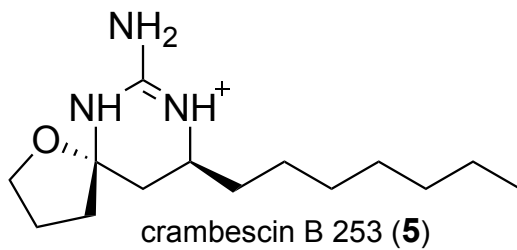
S25- crambescin B 253 (5) ^{13}C -NMR spectrum (125 MHz, CDCl_3)



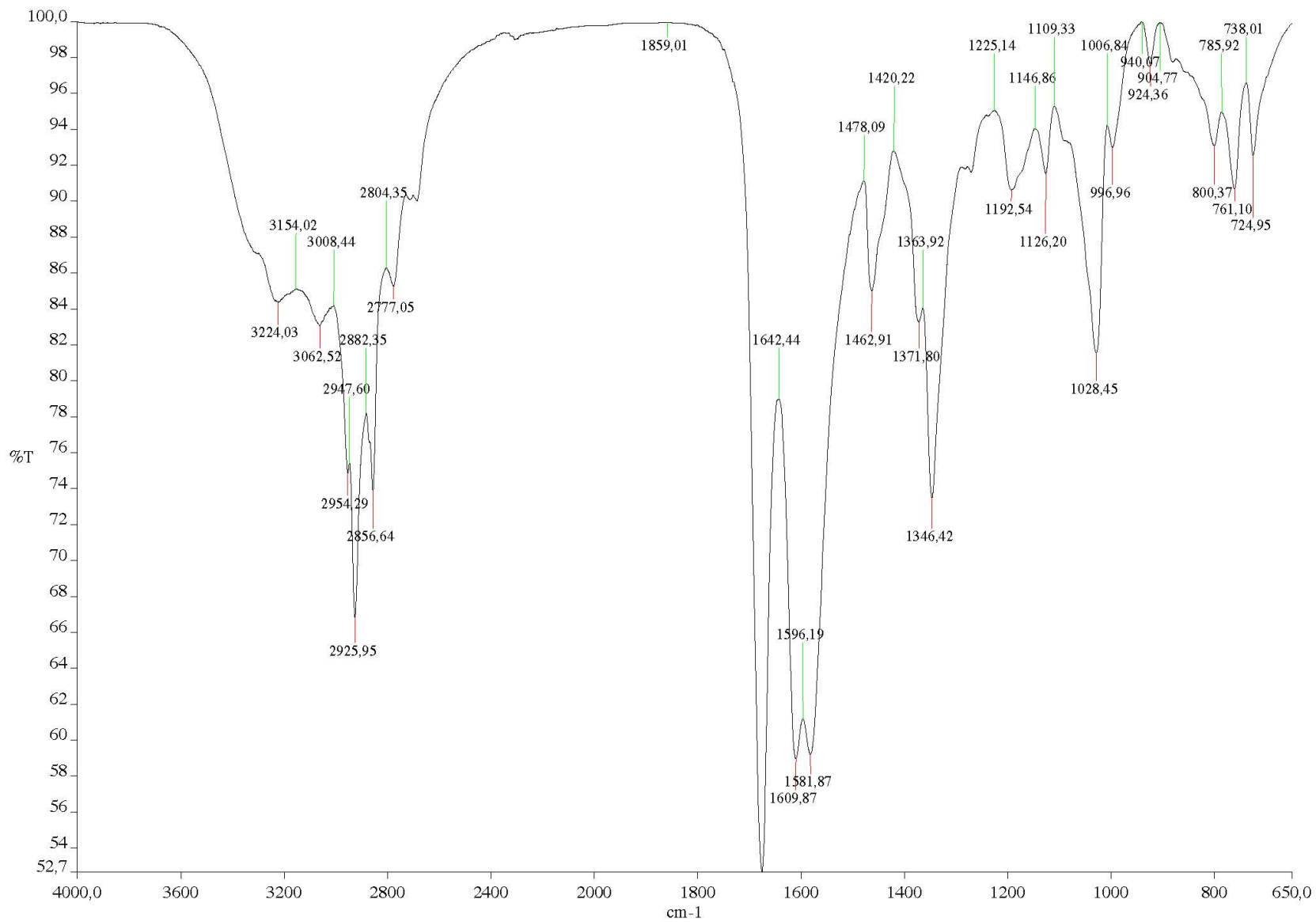
S26- crambescin B 253 (5) ¹H-¹H COSY spectrum (500 MHz, CDCl₃)



S27- crambescin B 253 (5) ¹H-¹³C HSQC-ED spectrum (500 MHz, CDCl₃)



S28- crambescin B 253 (5) ¹H-¹³C HMBC spectrum (500 MHz, CDCl₃)



c:\pel_data\spectra\eq95\crambescidin b253.002

S29- crambescidin 8 253 (5) IR spectrum

Crambescidin 359				Crambescidin acid			Crambescidin 401		
position	δ_C , type	δ_H , mult, <i>J</i> in Hz	HMBC	δ_C , type	δ_H , mult, <i>J</i> in Hz	HMBC	δ_C , type	δ_H , mult, <i>J</i> in Hz	HMBC
1	10.9, CH ₃	0.84; t (7.4)	C-2, C-3	10.7, CH ₃	0.80; t (7.3)	C-2, C-3	10.9, CH ₃	0.88; m	C-2, C-3
2	30.3, CH ₂	1.54, m; 1.46, m	-	30.8, CH ₂	1.54, m; 1.47, m	-	30.2, CH ₂	1.54, m; 1.46, m	-
3	72.2, CH	4.36, br d (9.5)	-	72.2, CH	4.34, br m	-	72.2, CH	4.56, br m	-
4	134.3, CH	5.51, br d (11.1)	C-3, C-6	134.5, CH	5.51, br d (11.2)	C-2, C-3, C-6	134.3, CH	5.54, br d (11.1)	C-3, C-6
5	131.4, CH	5.72, br dd (9.8, 11.1)	C-3	131.7, CH	5.72, br m	C-3, C-6, C-7	131.4, CH	5.76, br m	C-3, C-6, C-7
6	24.5, CH ₂	2.15, m; 2.43, m	C-4, C-5, C-8	24.6, CH ₂	2.15, m; 2.43, m	C-4, C-5, C-7, C-8	24.5, CH ₂	2.15, m; 2.43, m	C-4, C-5, C-7, C-8
7	38.4, CH ₂	1.97, dd (5.9, 14.2); 2.34, m	C-5, C-6, C-8, C-9	37.4, CH ₂	2.02, m; 2.24 m	C-5, C-6, C-8, C-9	37.8, CH ₂	2.02, m; 2.61 br t (13.4)	C-5, C-6, C-8, C-9
8	85.1, C	-	-	85.2, C	-	-	86.6, C	-	-
9	37.9, CH ₂	1.47, m; 2.60 dd (4.6, 13.1)	C-8, C-10, C-11	37.8, CH ₂	1.50 m; 2.53 dd (3.3, 12.9)	C-8, C-10, C-11	35.4, CH ₂	1.6 t (12.9); 2.81 dd (3.3, 12.9)	C-8, C-10, C-11
10	55.1, CH	3.99-4.08, m	-	55.8, CH	4.00, m	C-9	60.1, CH	4.82, m	-
11	30.9, CH ₂	1.74, m; 2.32, m	-	30.9, CH ₂	1.74, m; 2.32, m	-	30.5, CH ₂	2.06, m; 2.68, m	C-12, C-13
12	30.2, CH ₂	1.46-1.54, br m	-	28.5, CH ₂	2.06, m; 2.25, m	C-10, C-11, C-13	31.4, CH ₂	3.38, m	C-10, C-11, C-13
13	53.5, CH	3.99-4.08, m	-	54.9, CH	4.21, m	C-12, C-14, C-22	160.7, C	-	-
14	40.4, CH ₂	1.59, m; 2.60, dd (4.7, 13.2)	C-13, C-15, C-16	54.1, CH	2.68, d (4.7)	C-13, C-15, C-16, C-22	123.6, C	-	-
15	81.6, C	-	-	82.8, C	-	-	86.6, C	-	-
16	34.4, CH ₂	1.69-1.75, br m	-	32.8, CH ₂	1.27, m; 1.68, m	-	37.4, CH ₂	3.01-3.08, br m	C-14, C-17, C-18, C-22
17	19.5, CH ₂	1.82, m	-	19.8, CH ₂	1.80, m; 1.87, m	C-15, C-16, C-22	25.1, CH ₂	1.80, m; 1.87, m	C-15, C-16, C-22
18	33.3, CH ₂	1.26, m; 1.69, m	C-17, C-19	34.5, CH ₂	1.48, br m	-	39.5, CH ₂	1.48, br m	-
19	66.3, CH	3.75, m	-	67.8, CH	3.81, m	C-17, C-18	68.0, CH	3.74, m	C-17
20	21.9, CH ₃	1.08, d (6.2)	C-18, C-19	22.1, CH ₃	1.08, d (6.2)	C-18, C-19	21.9, CH ₃	1.14, d (6.2)	C-18, C-19
21	148.9, C	-	-	150.8, C	-	-	151.3, C	-	-
22	175.1, C	-	-	175.1, C	-	-	179.5, C	-	-

S30- ¹H NMR (500 MHz) and ¹³C NMR (125 MHz) for crambescidin 359

¹H NMR (600 MHz cryoprobe) and ¹³C NMR (150 MHz) for crambescidin acid and crambescidin 401

Crambescin B 281				Crambescin B 253		
position	δ_C , type	δ_H , mult, J in Hz	HMBC	δ_C , type	δ_H , mult, J in Hz	HMBC
1	154.4, C	-	-	154.4, C	-	-
2	88.0, C	-	-	88.0, C	-	-
3	67.3, CH ₂	3.86, 3.98; dd (6.5, 12.8)	C-2, C-4	67.3, CH ₂	3.86, 3.98; dd (6.5, 12.8)	C-2, C-4
4	54.7, CH ₂	1.97, 2.22; m	-	54.7, CH ₂	1.97, 2.22; m	-
5	36.8, CH ₂	1.91, 2.14; m	C-2, C-4	36.8, CH ₂	1.91, 2.14; m	C-2, C-4
6	37.7, CH ₂	1.62, 1.93; m	C-2, C-7, C-8, C-9	37.7, CH ₂	1.62, 1.93; m	C-2, C-7, C-8, C-9
7	47.1, CH	3.59; m	-	47.1, CH	3.59; m	-
8	34.9, CH ₂	1.49, 1.63; m	C-7	34.9, CH ₂	1.49, 1.63; m	C-7
9	25.1, CH ₂	1.22-1.14; m	-	25.1, CH ₂	1.22-1.13; m	-
10	29.5, CH ₂	1.22-1.14; m		29.4, CH ₂	1.22-1.13; m	-
11	29.5, CH ₂	1.22-1.14; m		29.2, CH ₂	1.22-1.13; m	-
12	29.4, CH ₂	1.22-1.14; m	-	38.1, CH ₂	1.22-1.13; m	-
13	29.3, CH ₂	1.22-1.14; m	-	22.9, CH ₂	1.22-1.13; m	-
14	38.1, CH ₂	1.22-1.14; m	-	14.1, CH ₃	0.87; t (7.1)	C-12, C-13
15	22.9, CH ₂	1.22-1.14; m	-			
16	14.1, CH ₃	0.87; t (7.1)	C-14, C-15			

S31- ¹H NMR (500 MHz) and ¹³C NMR (125 MHz) for crambescin 281 and crambescin 253