

Mexicanolide limonoids with in vitro neuroprotective activities from seeds of *Khaya senegalensis*

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S53: ¹³C-NMR (125 MHz, CDCl₃) Spectrum of Compound **9** (Khasenegasin I)

S54: HSQC Spectrum of Compound **9** (Khasenegasin I)

S55: HMBC Spectrum of Compound **9** (Khasenegasin I)

S56: ROESY Spectrum of Compound **9** (Khasenegasin I)

S57: HR-ESI-MS Spectrum of Compound **10** (Khasenegasin J)

S58: ¹H-NMR (500 MHz, CDCl₃) Spectrum of Compound **10** (Khasenegasin J)

S59: ¹³C-NMR (125 MHz, CDCl₃) Spectrum of Compound **10** (Khasenegasin J)

S60: HSQC Spectrum of Compound **10** (Khasenegasin J)

S61: HMBC Spectrum of Compound **10** (Khasenegasin J)

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S83: ^{13}C -NMR (125 MHz, CDCl_3) Spectrum of Compound **14** (Khasenegasin N)

S84: HSQC Spectrum of Compound **14** (Khasenegasin N)

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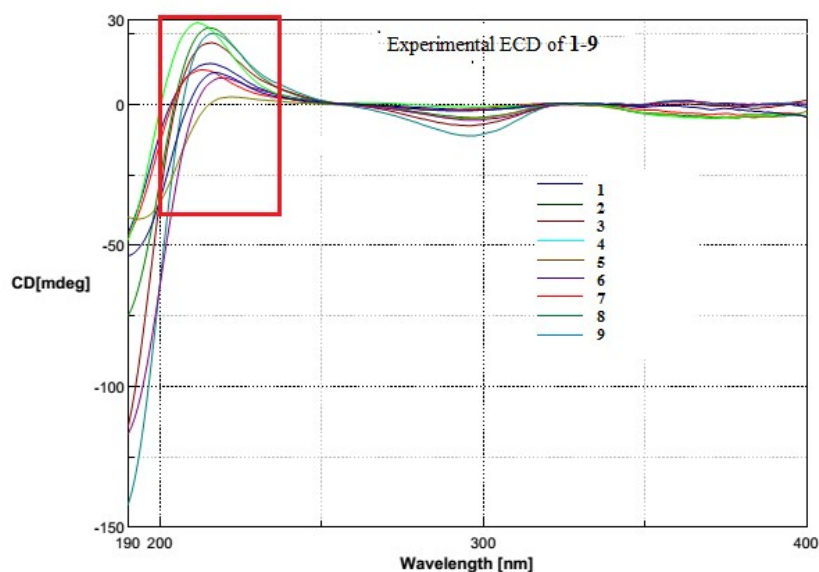
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S1: Experimental ECD of 1-9



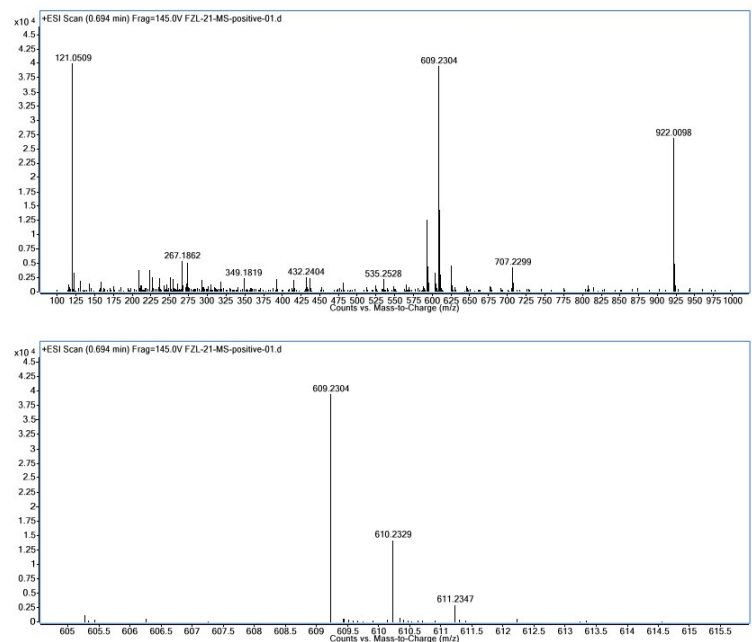
S2: The values of cell viabilities of compounds 2-10, 12-15 and 17

Compound	in vitro cytotoxicities (% of Control at 10 μ M)	in vitro neuroprotective activities (% of Control at 10 μ M)	in vitro neuroprotective activities (% of Control at 1 μ M)
2	78.0 $\pm$ 3.3	53.2 $\pm$ 2.5	ND
3	107.0 $\pm$ 6.4	73.3 $\pm$ 3.9	ND
4	100.6 $\pm$ 10.2	68.3 $\pm$ 3.9	ND
5	85.2 $\pm$ 1.7	58.4 $\pm$ 3.1	ND
6	100.8 $\pm$ 9.8	88.5 $\pm$ 6.4***	87.6 $\pm$ 1.2%***
7	108.3 $\pm$ 7.0	52.5 $\pm$ 2.0	ND
8	104.0 $\pm$ 10.1	71.1 $\pm$ 1.2	ND
9	102.0 $\pm$ 5.9	60.2 $\pm$ 3.6	ND
10	100.7 $\pm$ 9.9	54.1 $\pm$ 3.6	ND
12	96.2 $\pm$ 5.3	56.0 $\pm$ 3.9	ND
13	83.6 $\pm$ 5.6	61.3 $\pm$ 3.7	ND
14	110.7 $\pm$ 1.8	66.3 $\pm$ 3.7	ND
15	109.2 $\pm$ 3.5	64.2 $\pm$ 3.9	ND
17	107.0 $\pm$ 10.4	78.4 $\pm$ 5.7*	76.5 $\pm$ 2.0%*
Edaravone	ND	86.7 $\pm$ 5.6***(50 μ M)	

Cell viabilities were evaluated by MTT assay and the cell viability in control was taken as 100%, and the average value of cell viability under glutamate exposure was 53.6 $\pm$ 4.7%. Glutamate-injured cells differ significantly from the control at a level of $P < 0.001$. The values expressed as mean $\pm$ SD of triplicate experiments. ND = not determined. (* $P < 0.05$, *** $P < 0.001$)

Khasenegasin A (1)

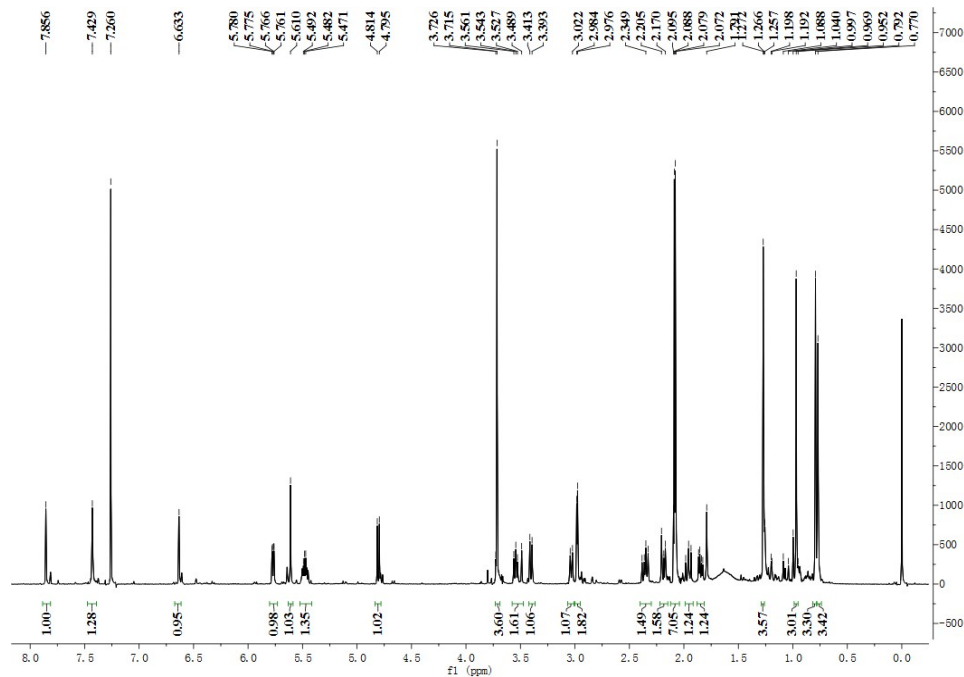
S3 HR-ESI-MS of Khasenegasin A (1)



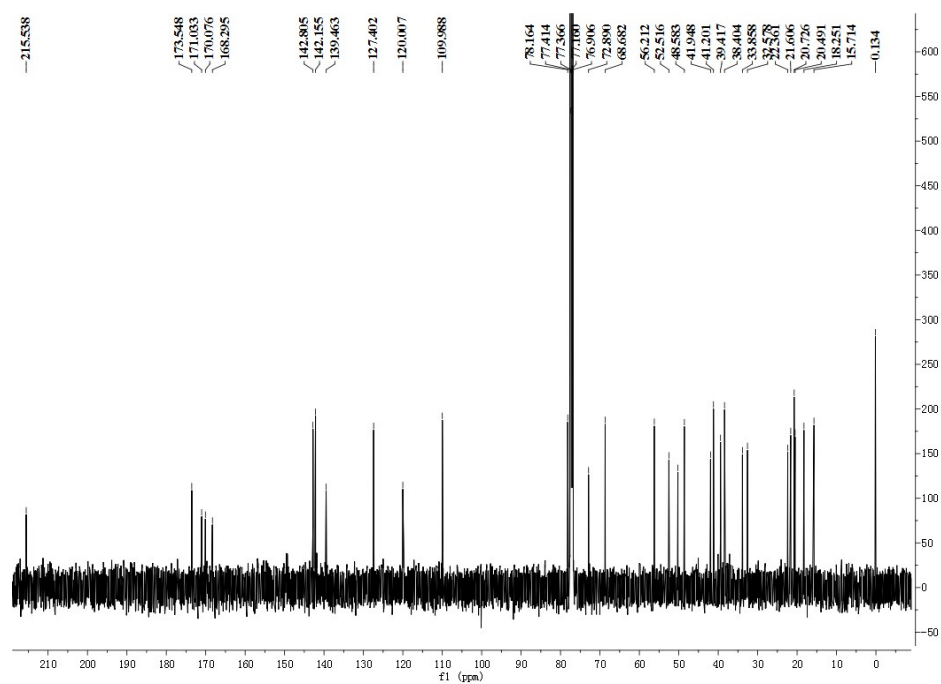
Elemental Composition Calculator

Target m/z:	609.2304	Result type:	Positive ions	Species:	[M+Na] ⁺
Elements:	C (0-80); H (0-120); O (0-30); N(0-10); Na (0-5); S (0-5)				
Ion Formula	Calculated m/z		PPM Error		
C31H38NaO11	609.2306		0.4		

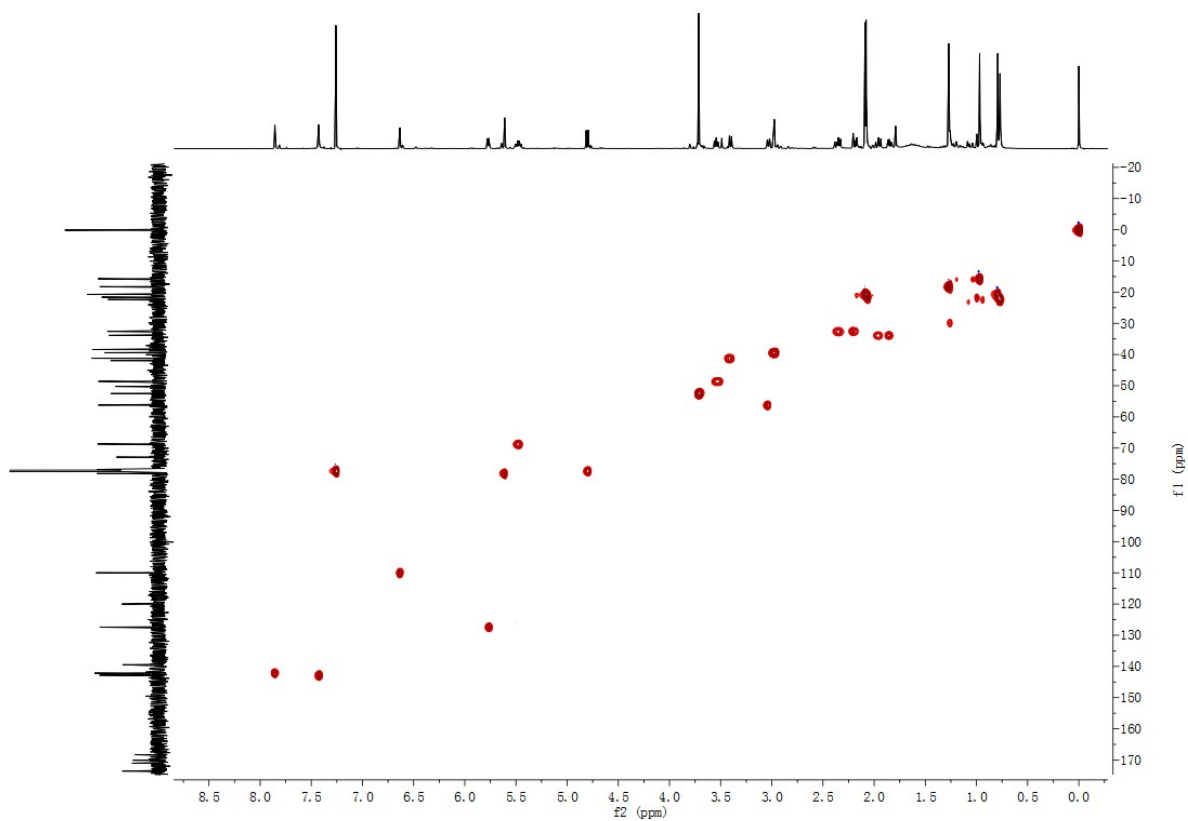
S4 ¹H NMR of Khasenegasin A (1)



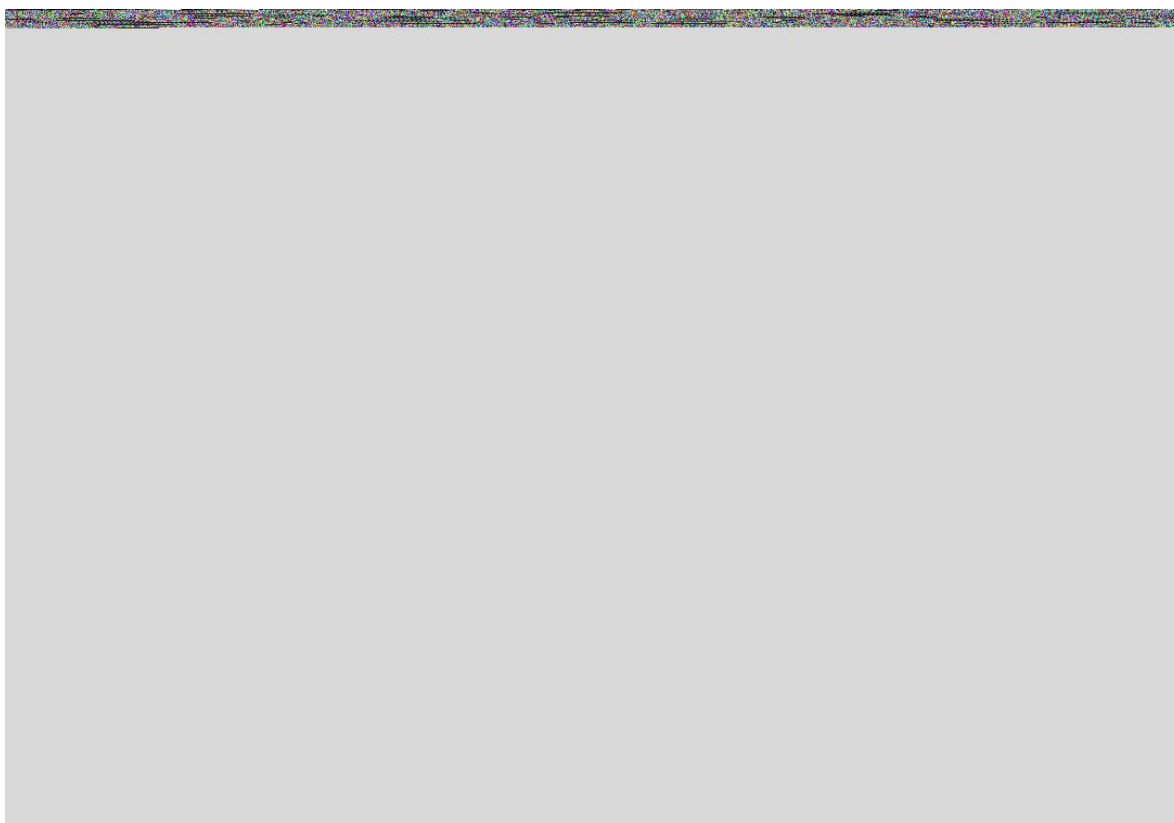
S5 ^{13}C NMR of Khasenegasin A (**1**)



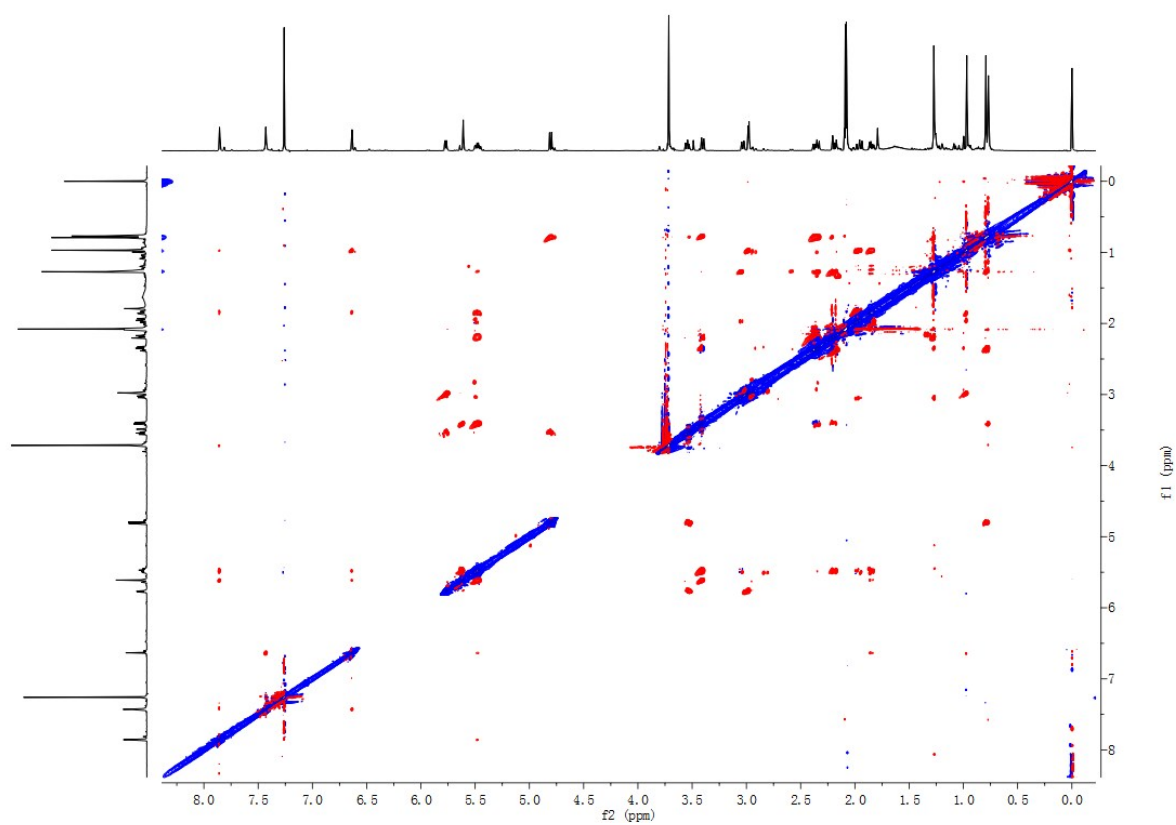
S6 HSQC of Khasenegasin A (**1**)



S7 HMBC of Khasenegasin A (**1**)

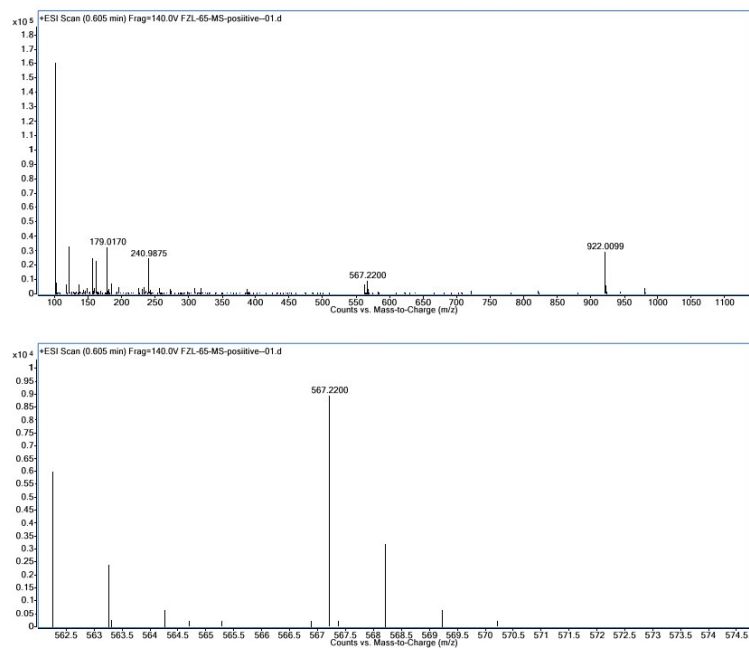


S8 ROSEY of Khasenegasin A (**1**)



Khasenegasin B (2)

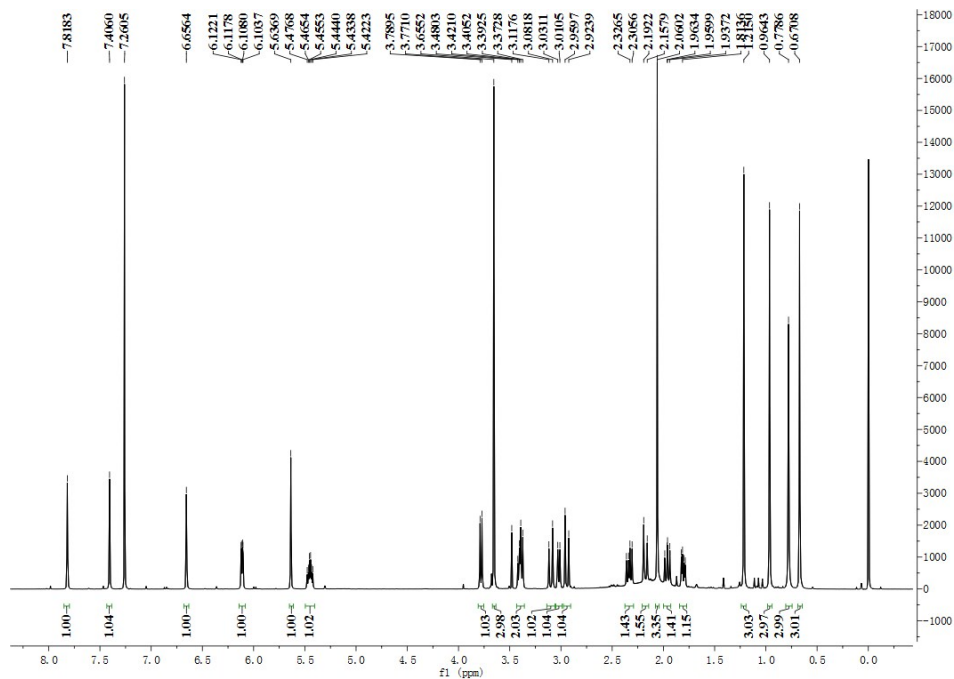
S9 HR-ESI-MS of Khasenegasin B (2)



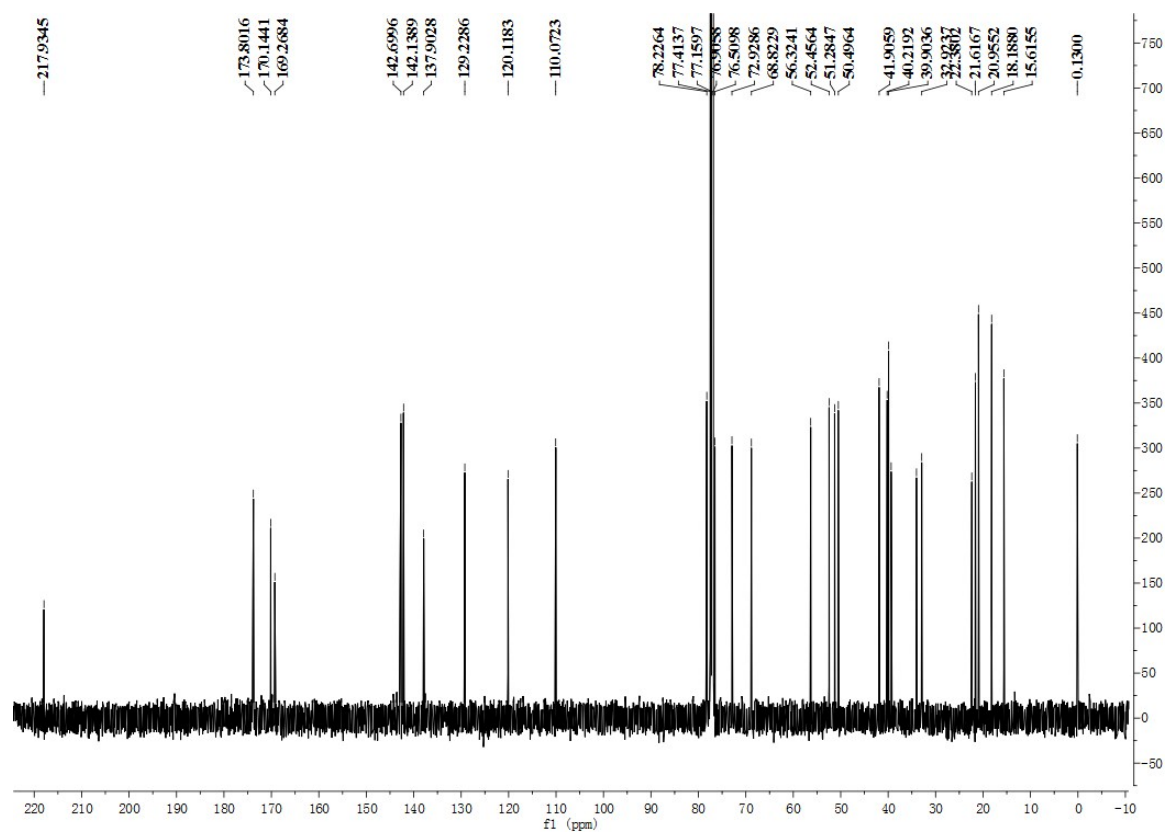
Elemental Composition Calculator

Target m/z:	567.2200	Result type:	Positive ions	Species:	[M+Na] ⁺
Elements:	C (0-80); H (0-120); O (0-30); N(0-10); Na (0-5); S (0-5)				
Ion Formula	Calculated m/z		PPM Error		
C ₂₉ H ₃₆ NaO ₁₀	567.2201		0.15		

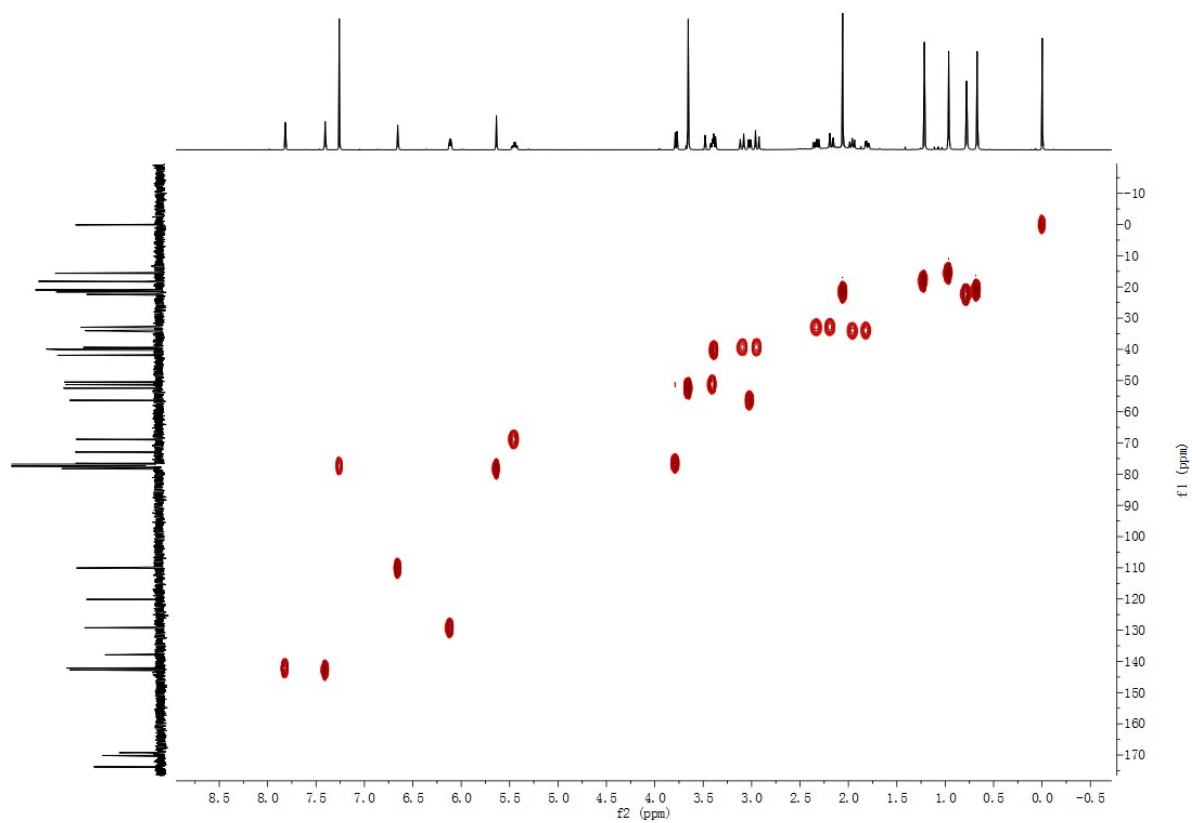
S10 ¹H NMR of Khasenegasin B (2)



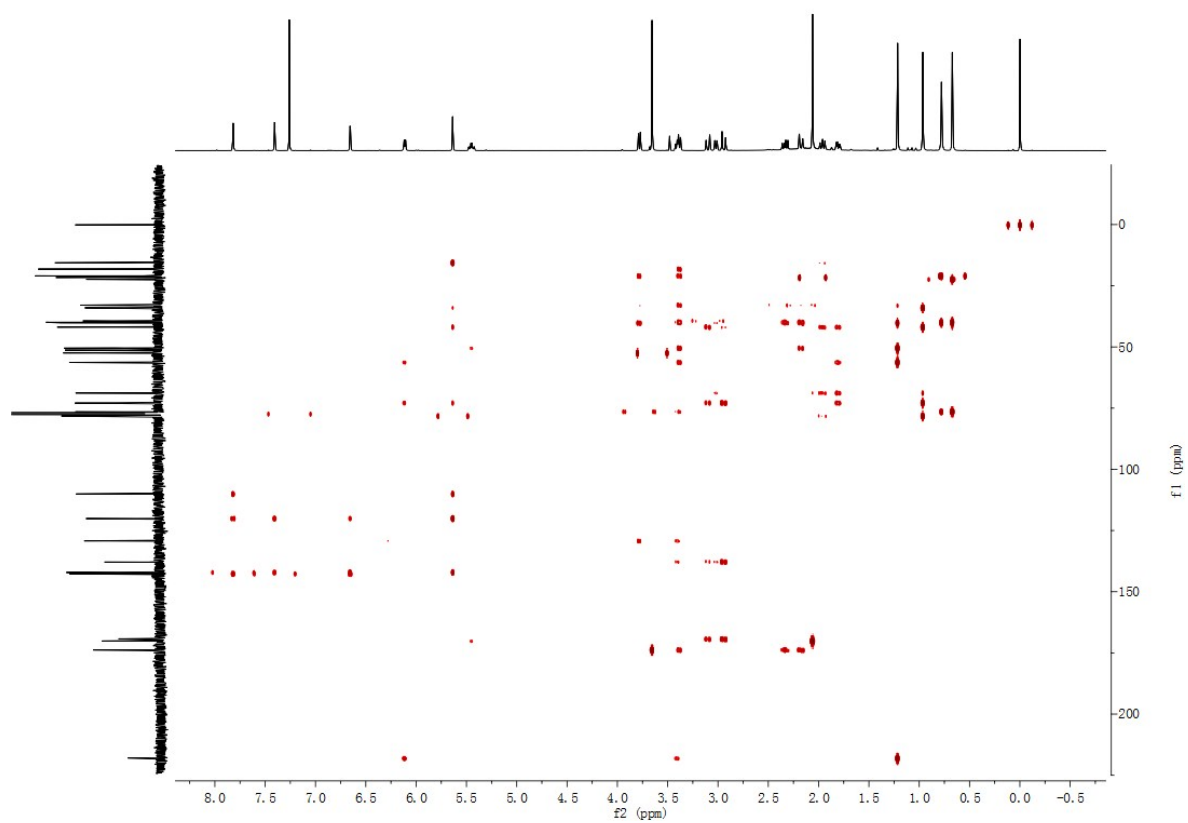
S11 ^{13}C NMR of Khasenegasin B (2)



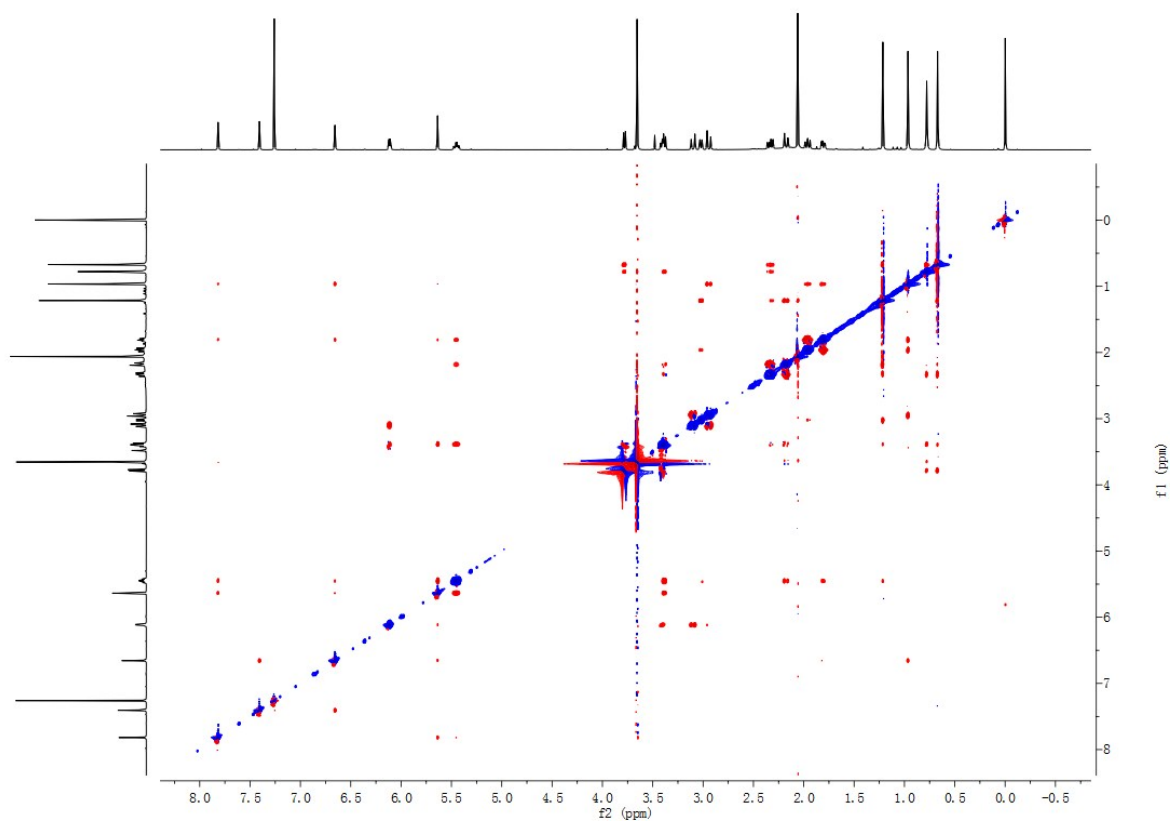
S12 HSQC of Khasenegasin B (2)



S13 HMBC of Khasenegasin B (2)

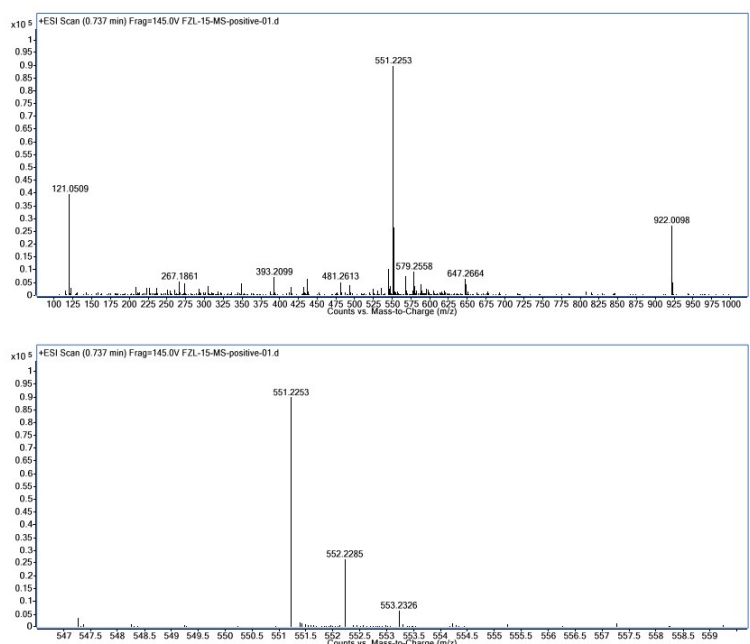


S14 ROSEY of Khasenegasin B (2)



Khasenegasin C (3)

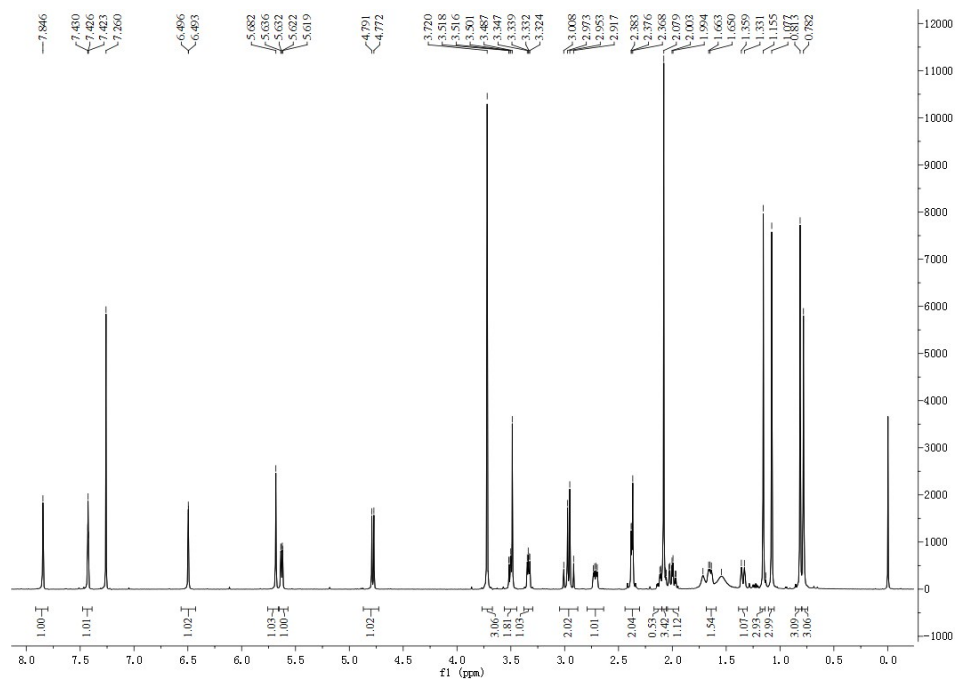
S15 HR-ESI-MS of Khasenegasin C (3)



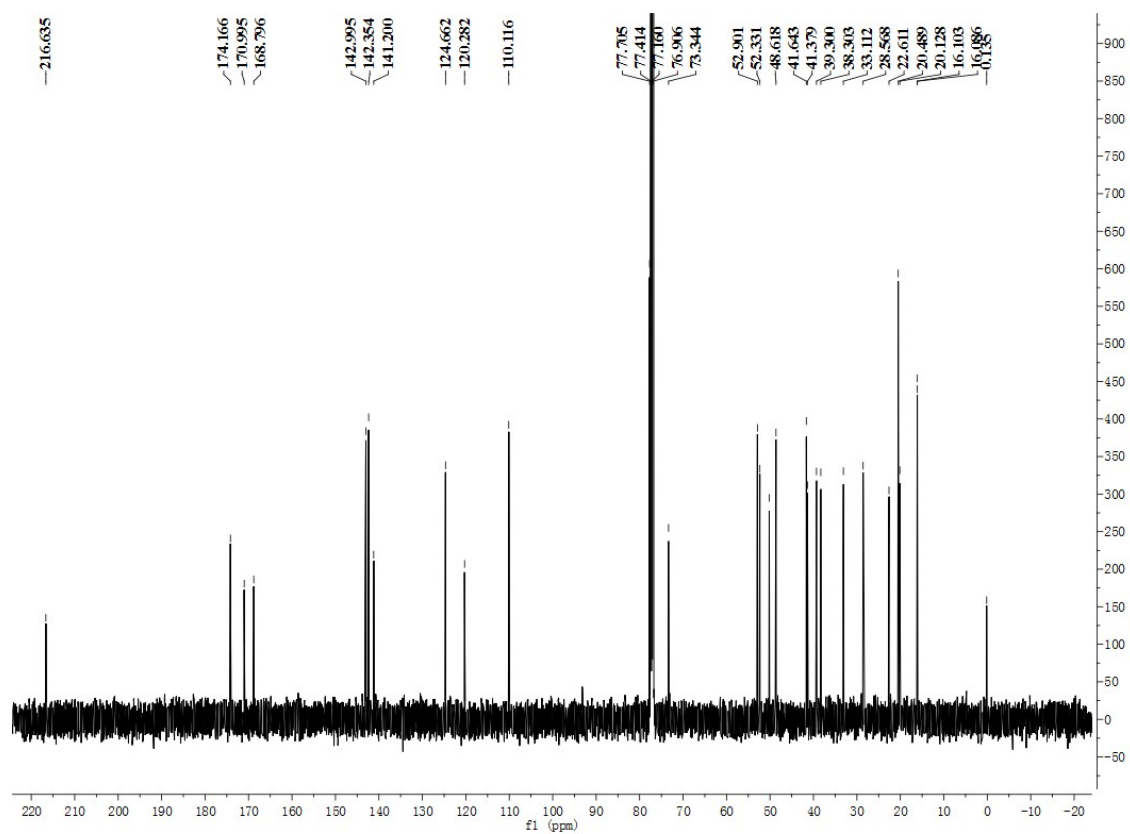
Elemental Composition Calculator

Target m/z:	551.2253	Result type:	Positive ions	Species:	[M+Na] ⁺
Elements:	C (0-80); H (0-120); O (0-30); N(0-10); Na (0-5); S (0-5)				
Ion Formula	Calculated m/z		PPM Error		
C ₂₉ H ₃₆ NaO ₉	551.2252		-0.25		

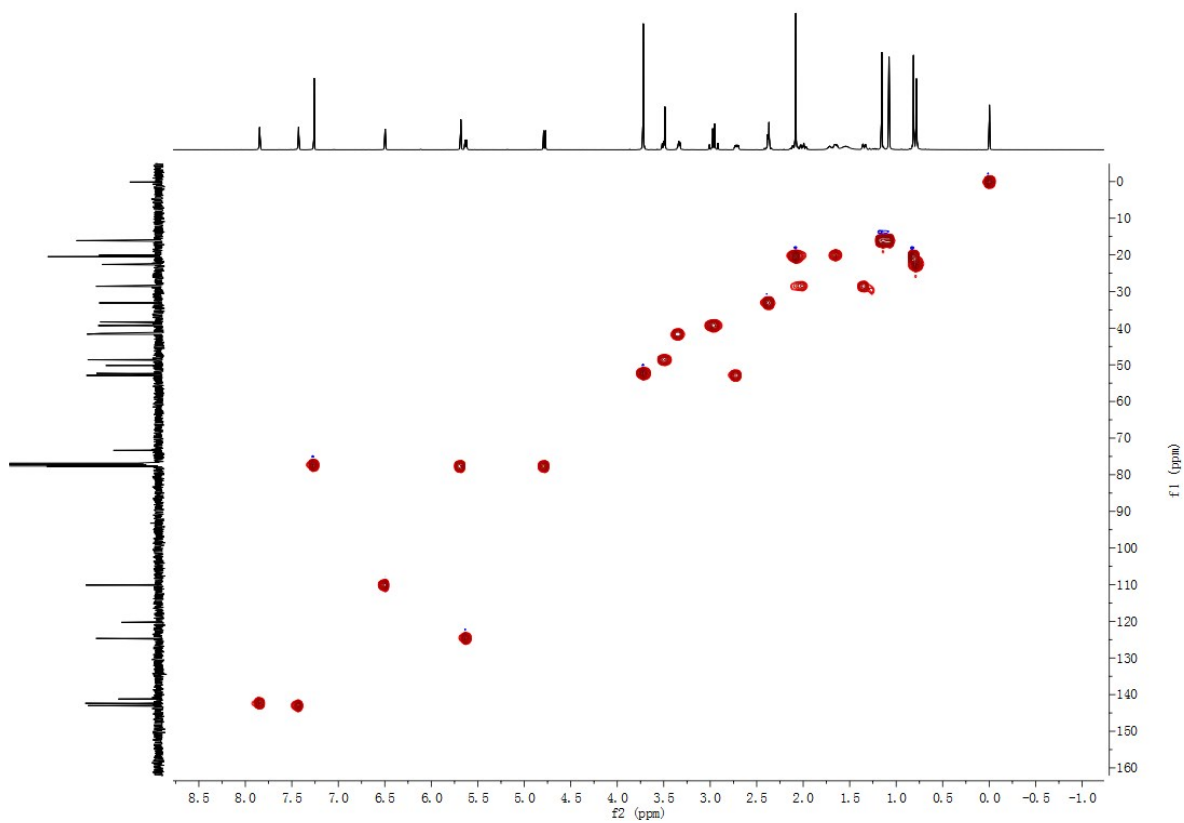
S16 ¹H NMR of Khasenegasin C (3)



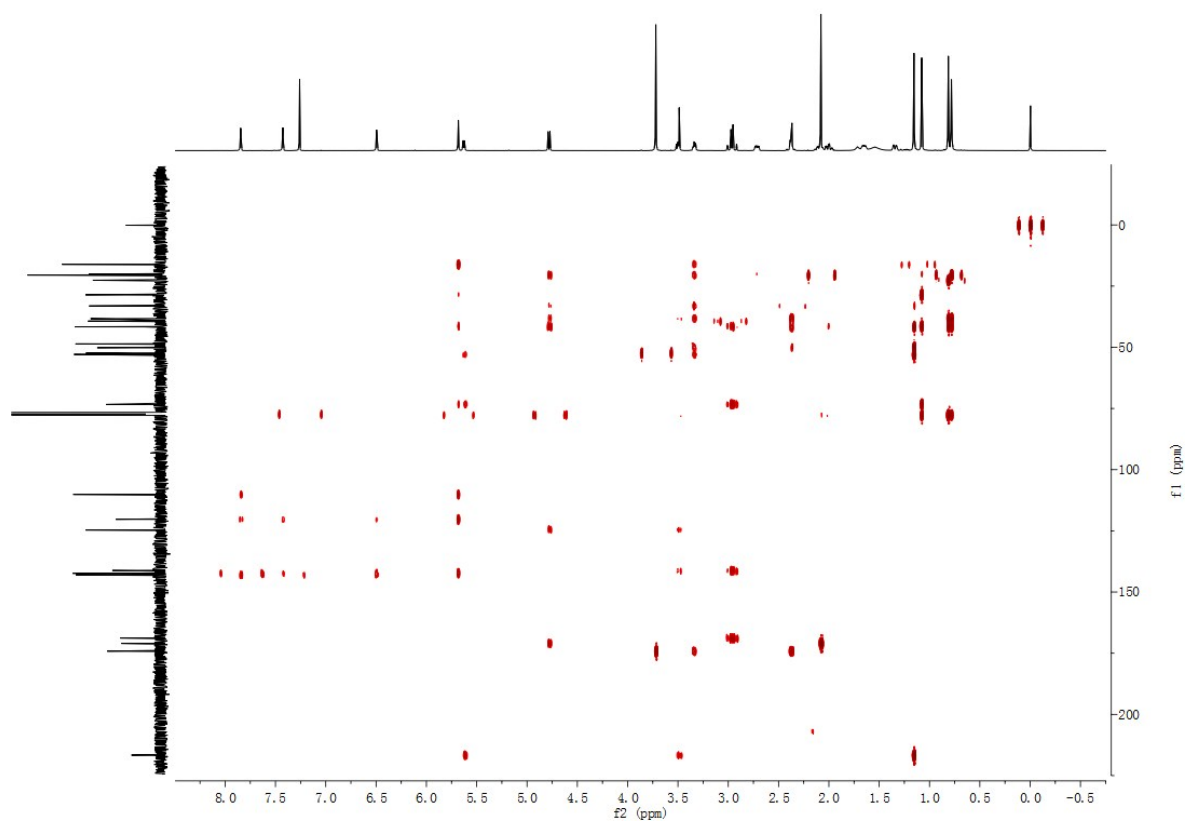
S17 ^{13}C NMR of Khasenegasin C (**3**)



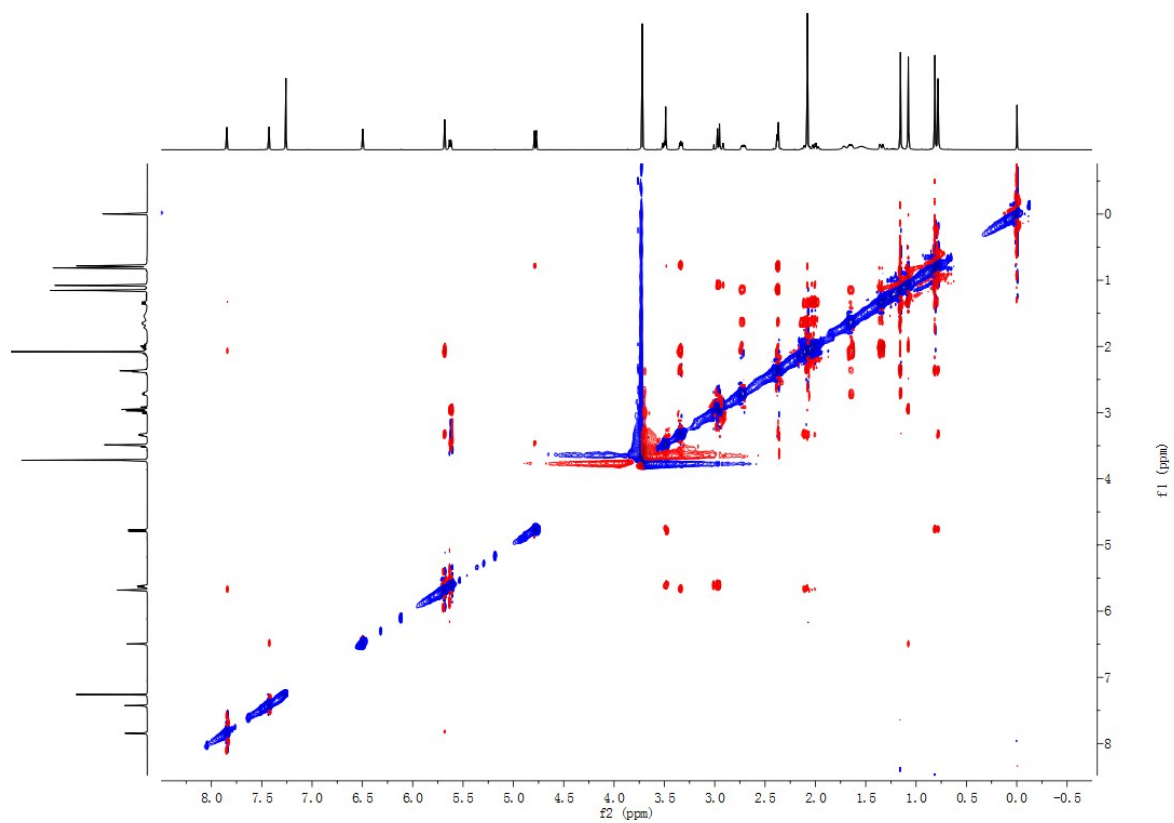
S18 HSQC of Khasenegasin C (**3**)



S19 HMBC of Khasenegasin C (**3**)

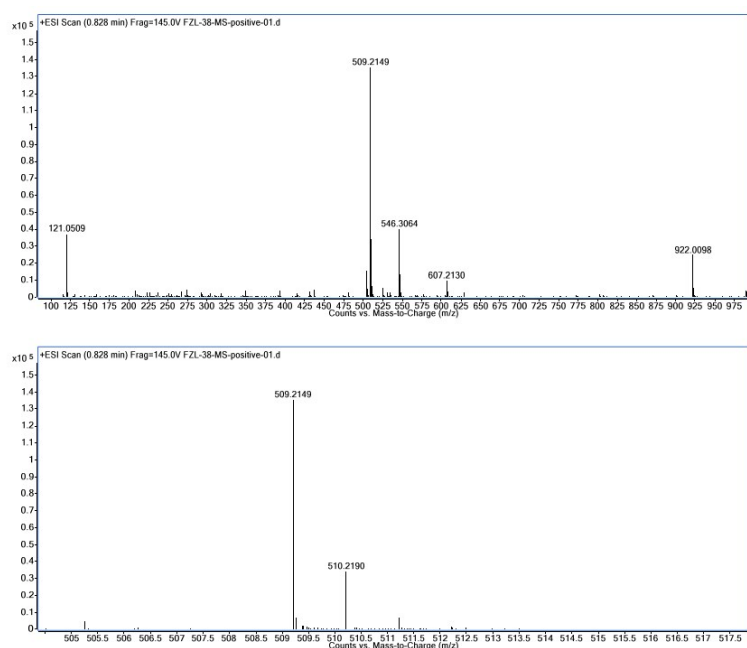


S20 ROSEY of Khasenegasin C (**3**)



Khasenegasin D (4)

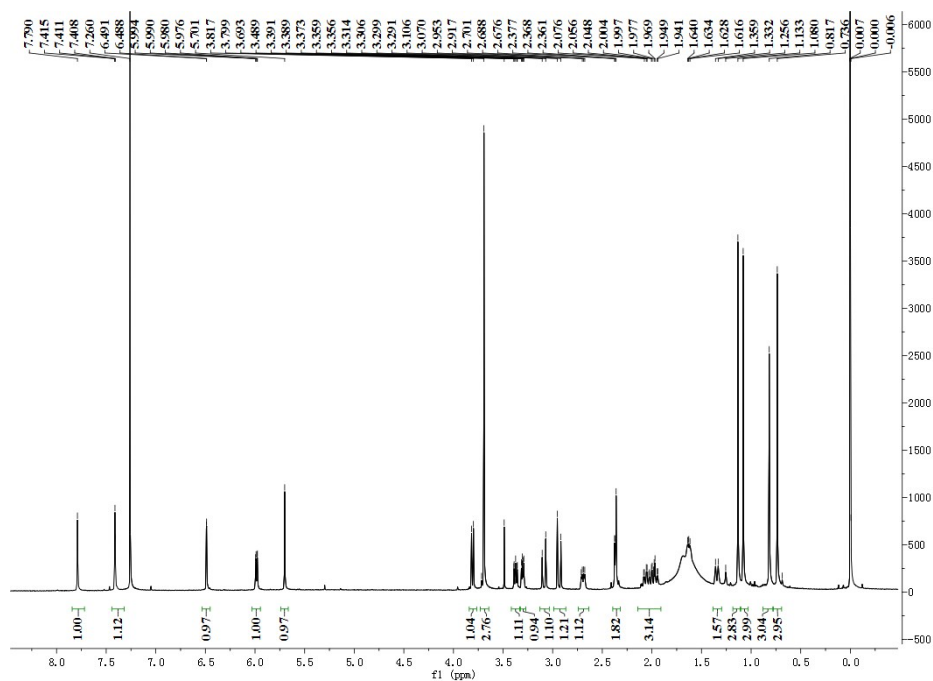
S21 HR-ESI-MS of Khasenegasin D (4)



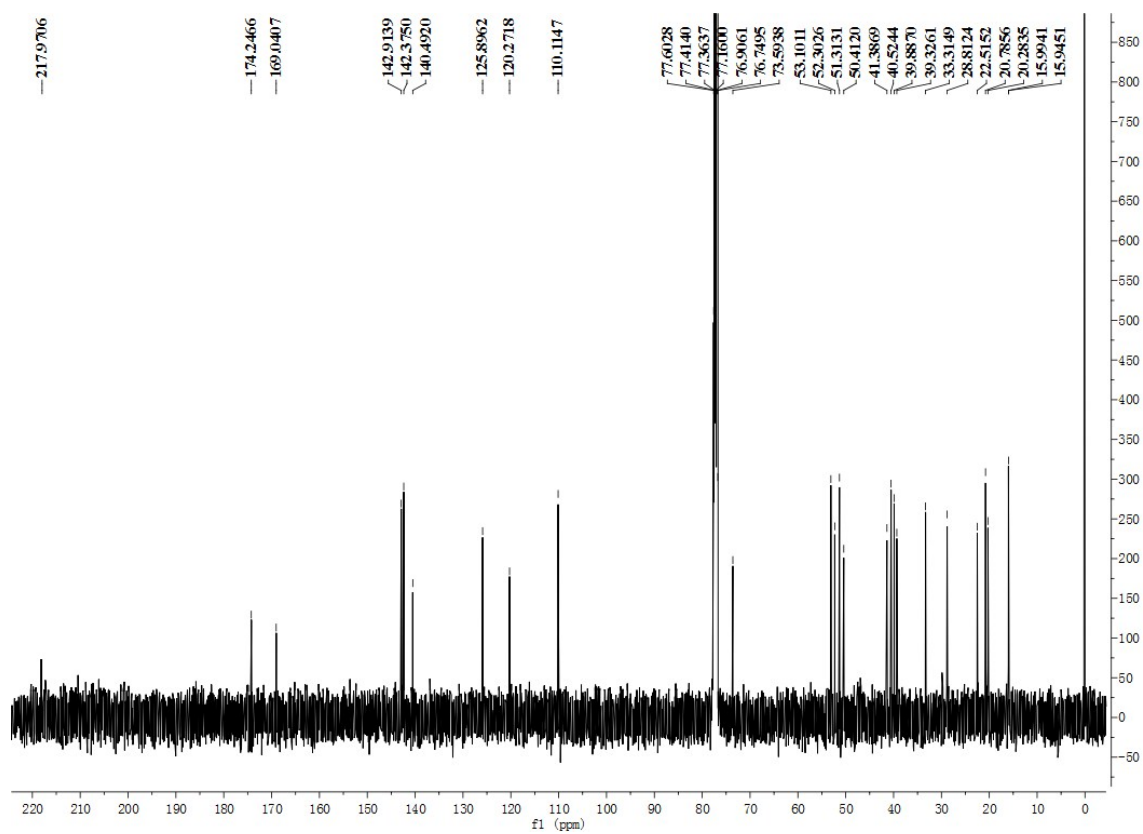
Elemental Composition Calculator

Target m/z:	509.2149	Result type:	Positive ions	Species:	[M+Na] ⁺
Elements:	C (0-80); H (0-120); O (0-30); N(0-10); Na (0-5); S (0-5)				
Ion Formula	Calculated m/z		PPM Error		
C27H34NaO8	509.2146		-0.64		

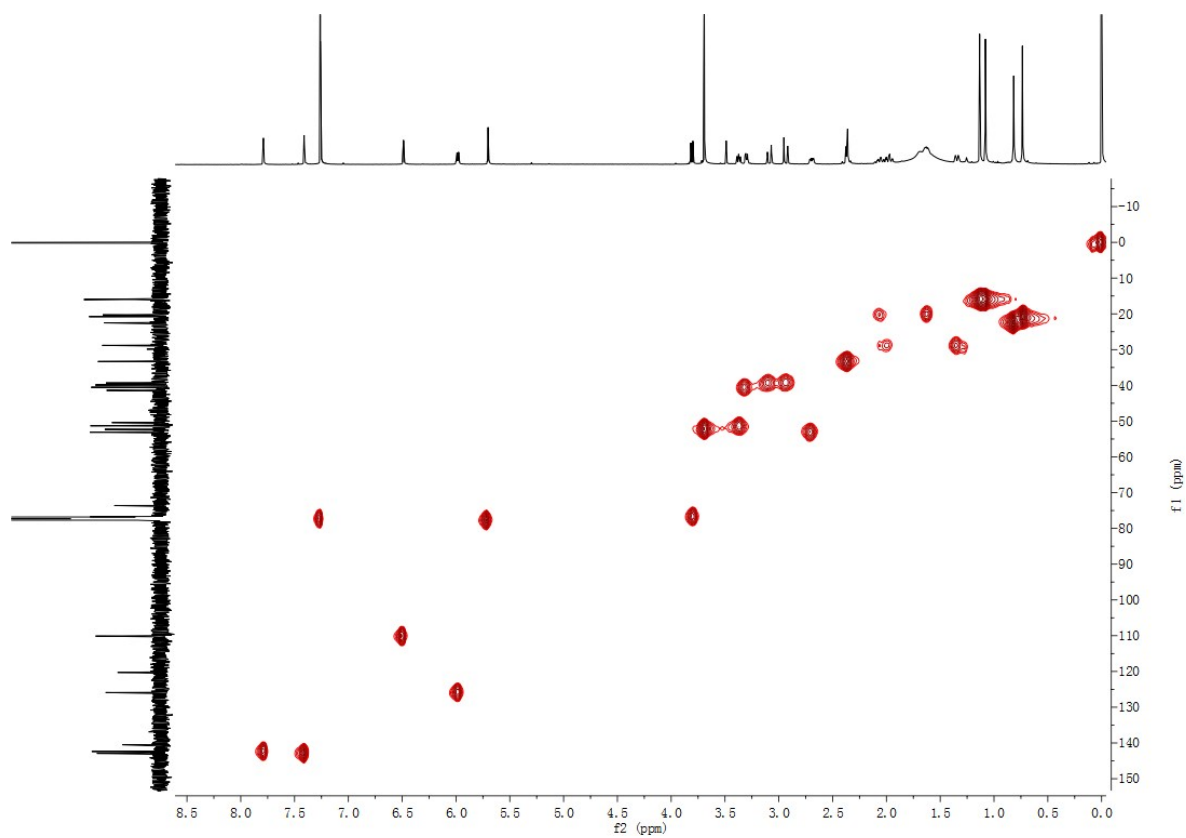
S22 ¹H NMR of Khasenegasin D (4)



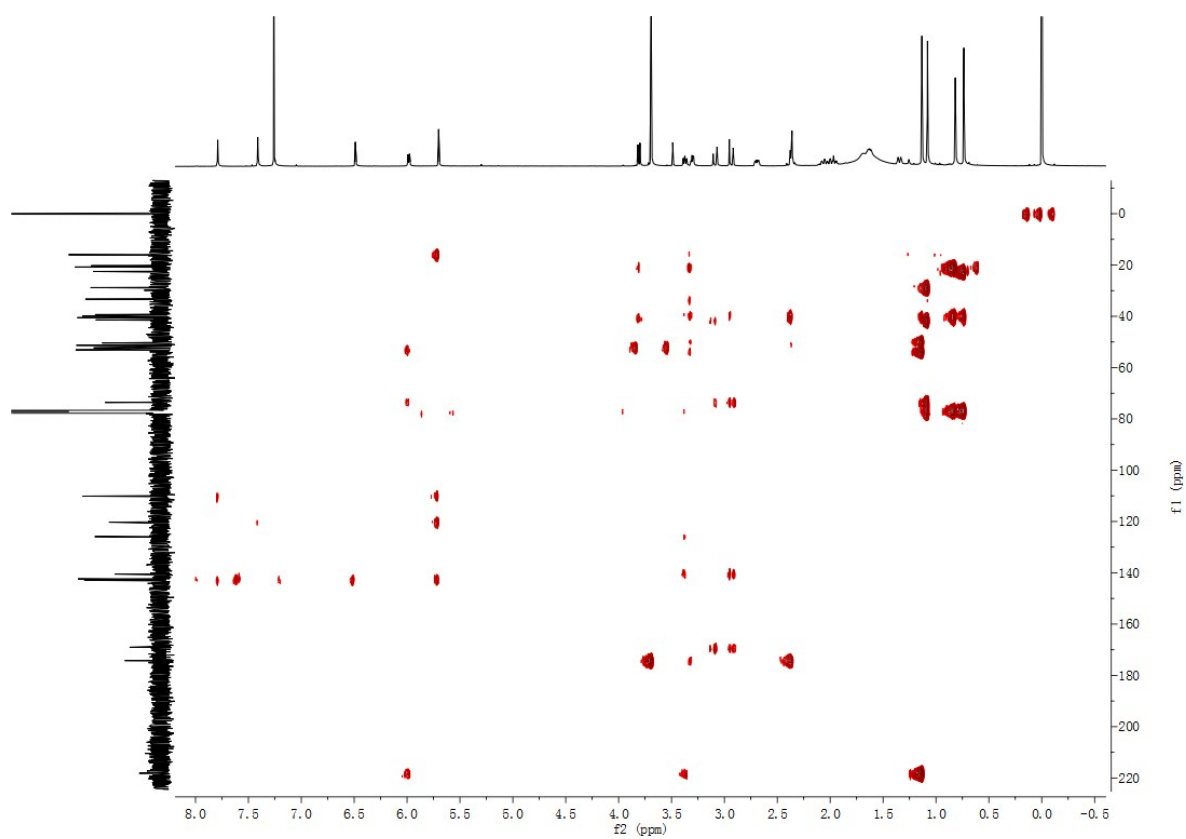
S23 ^{13}C NMR of Khasenegasin D (4)



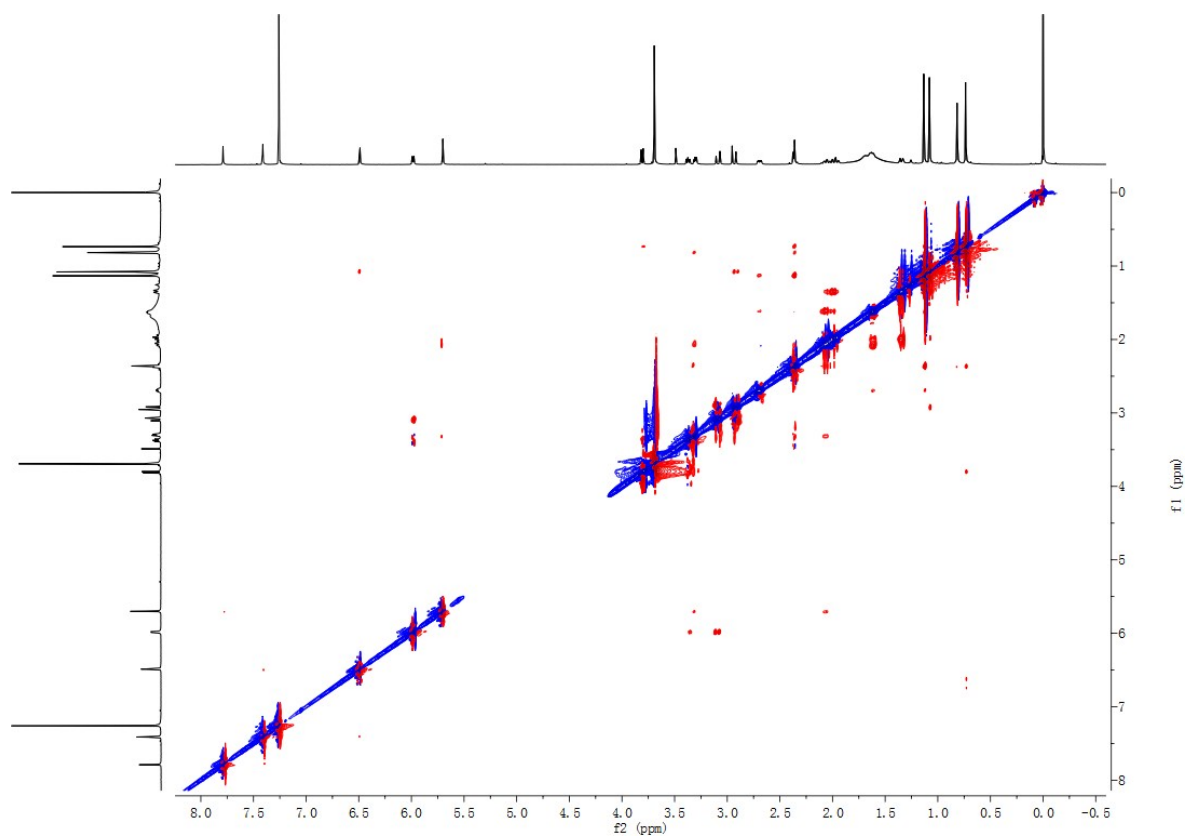
S24 HSQC of Khasenegasin D (4)



S25 HMBC of Khasenegasin D (4)

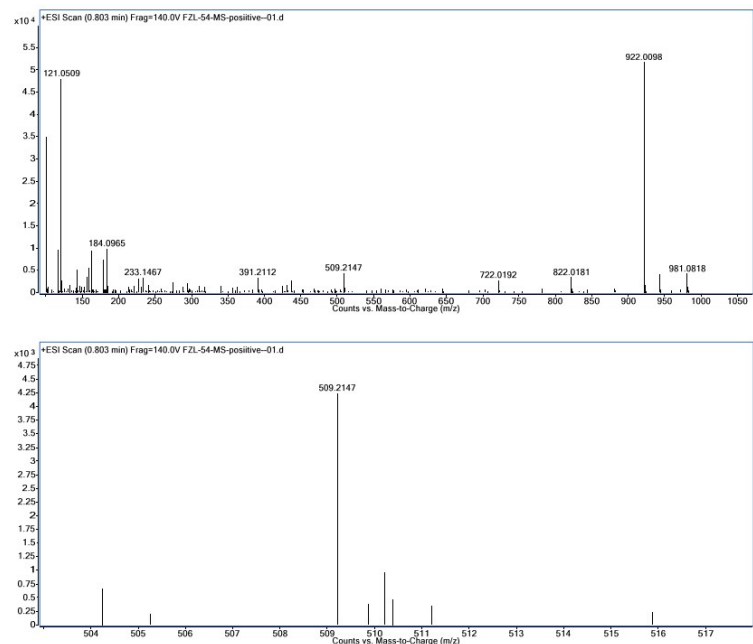


S26 ROSEY of Khasenegasin D (4)



Khasenegasin E (5)

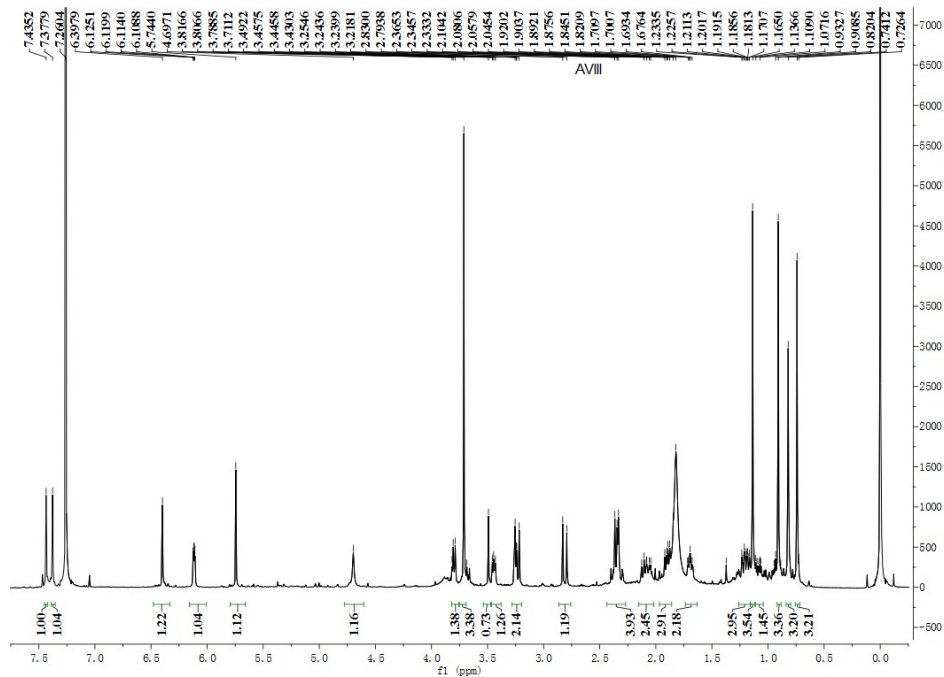
S27 HR-ESI-MS of Khasenegasin E (5)



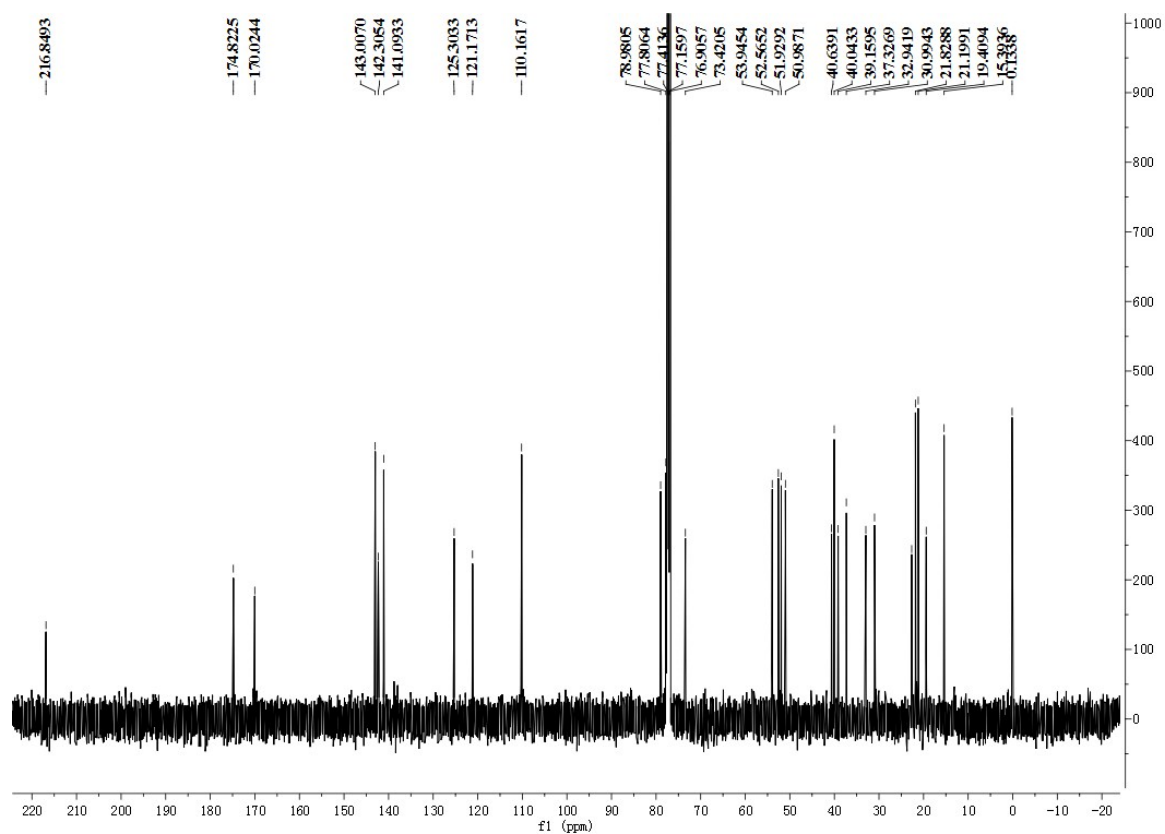
Elemental Composition Calculator

Target m/z:	509.2147	Result type:	Positive ions	Species:	[M+Na] ⁺
Elements:	C (0-80); H (0-120); O (0-30); N(0-10); Na (0-5); S (0-5)				
Ion Formula	Calculated m/z		PPM Error		
C ₂₇ H ₃₄ NaO ₈	509.2146		-0.27		

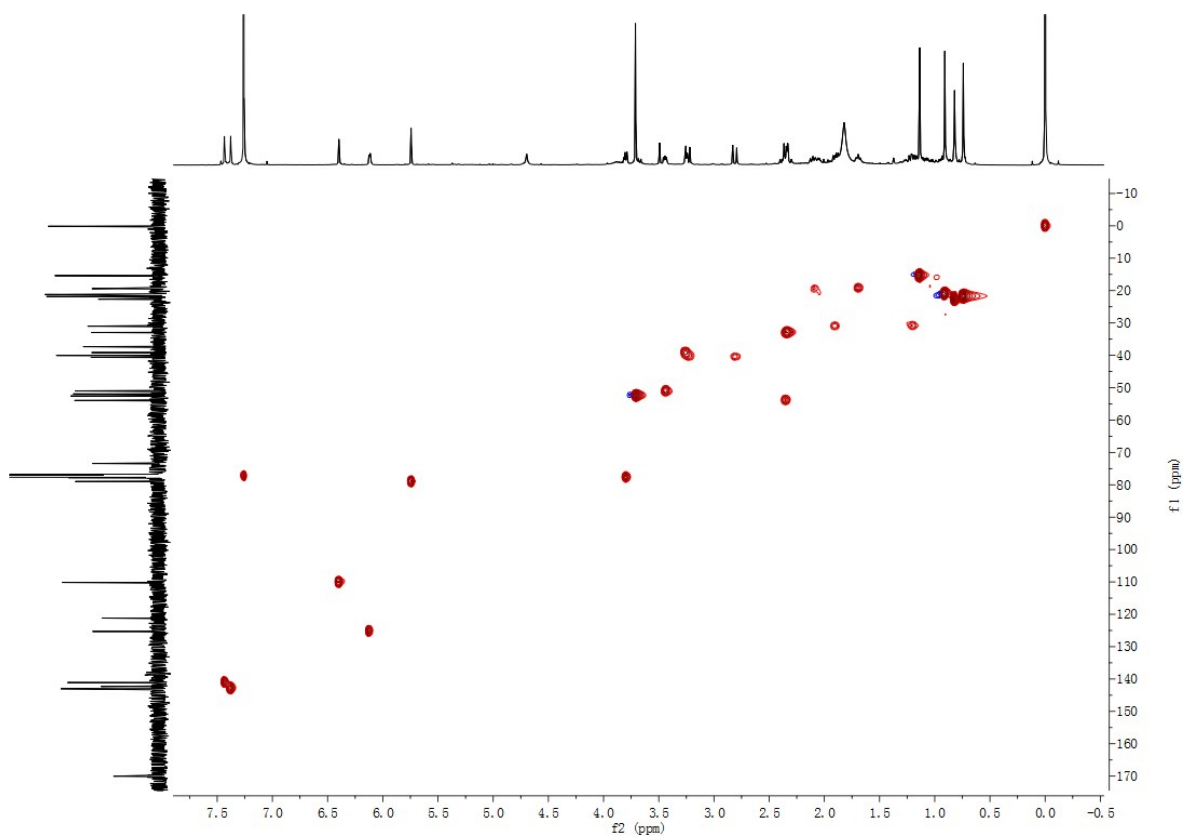
S28 ¹H NMR of Khasenegasin E (5)



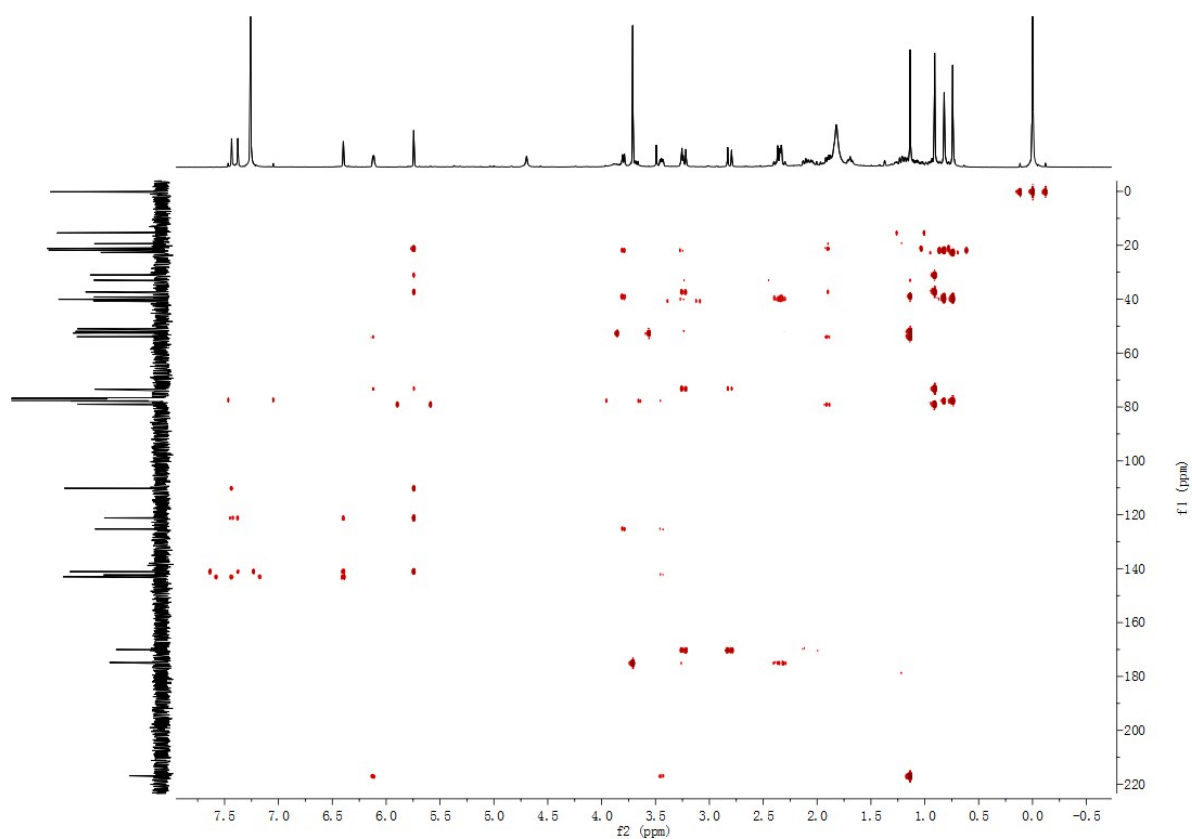
S29 ^{13}C NMR of Khasenegasin E (5)



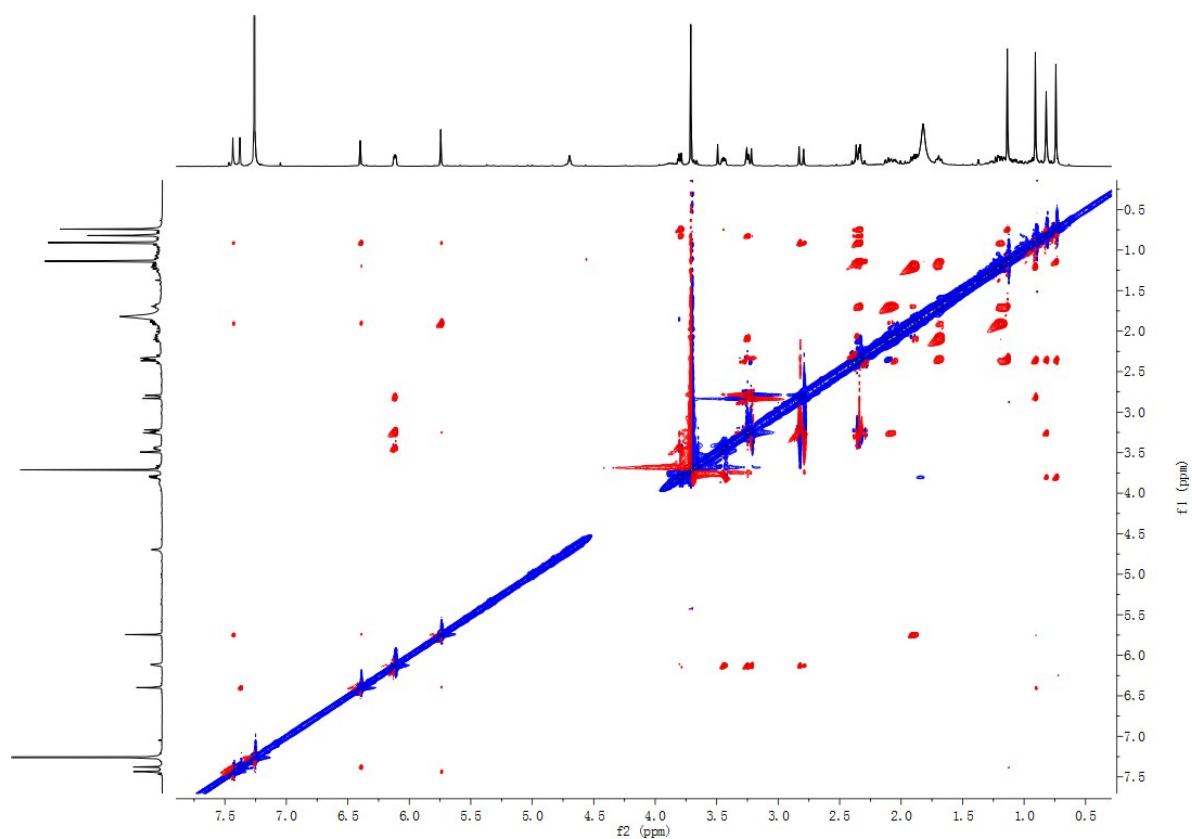
S30 HSQC of Khasenegasin E (5)



S31 HMBC of Khasenegasin E (5)

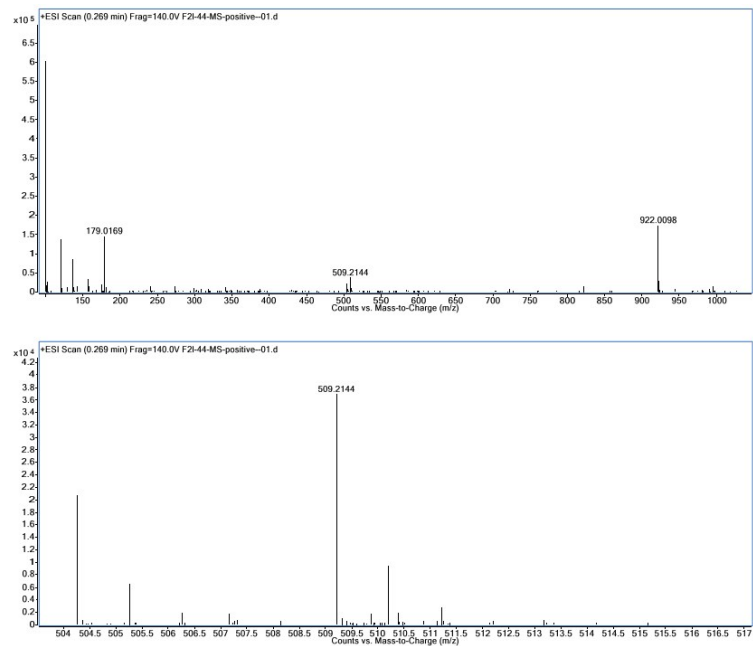


S32 ROSEY of Khasenegasin E (5)



Khasenegasin F (6)

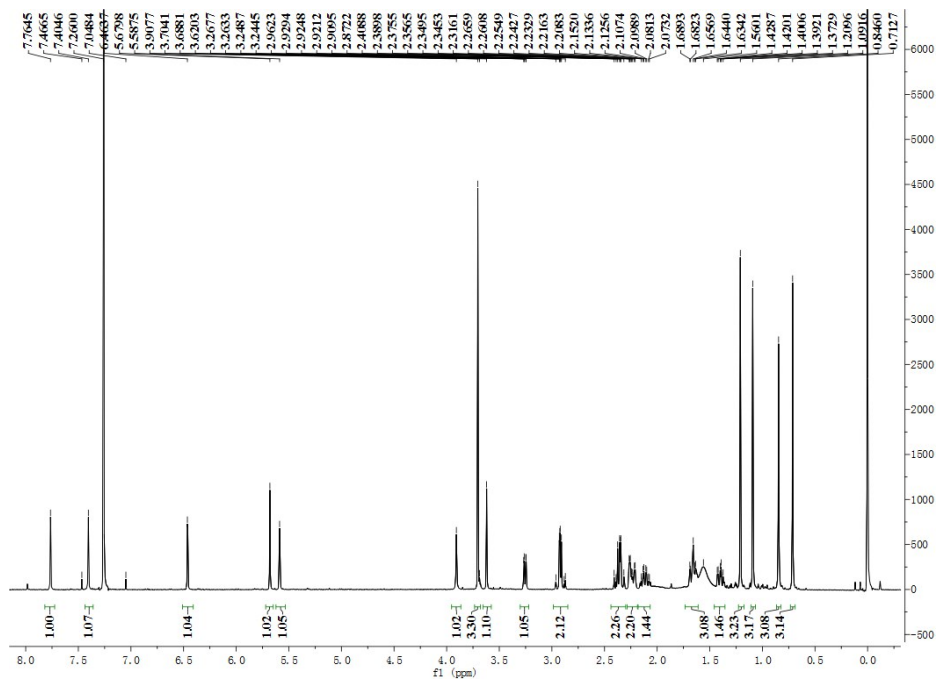
S33 HR-ESI-MS of Khasenegasin F (6)



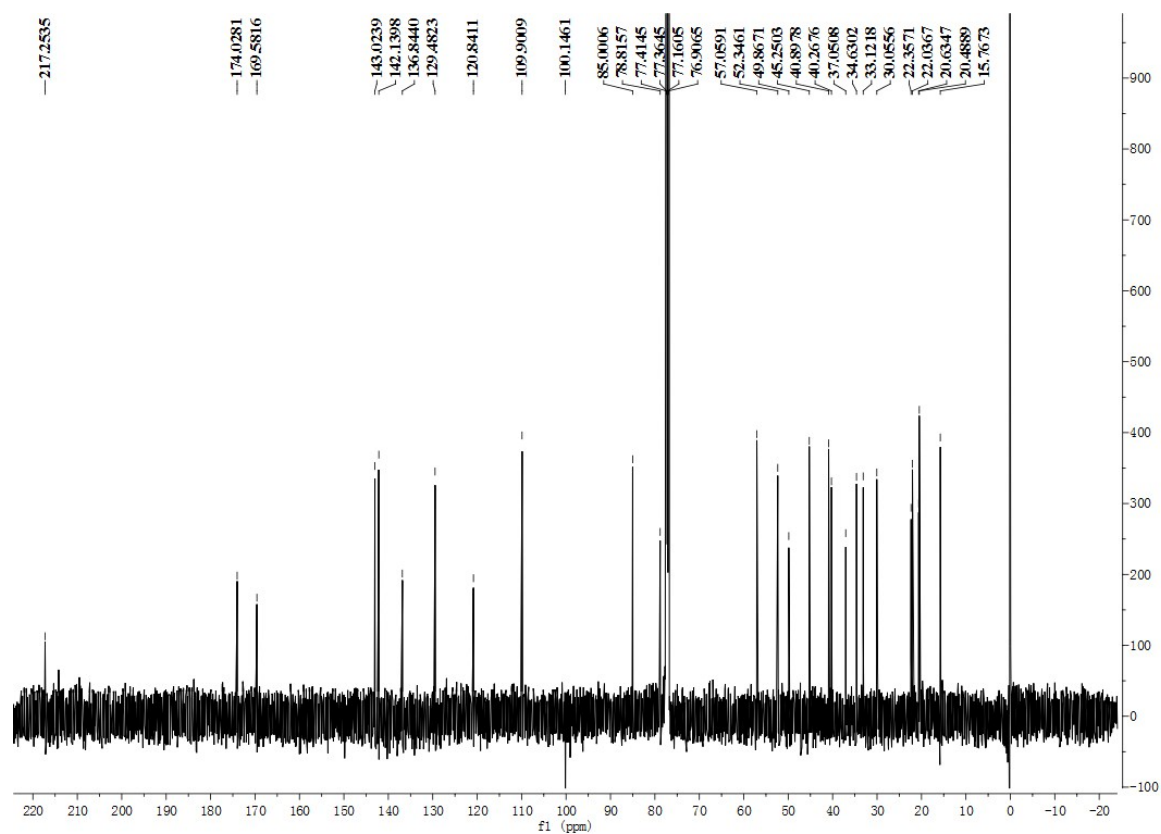
Elemental Composition Calculator

Target m/z:	509.2144	Result type:	Positive ions	Species:	[M+Na] ⁺
Elements:	C (0-80); H (0-120); O (0-30); N(0-10); Na (0-5); S (0-5)				
Ion Formula	Calculated m/z		PPM Error		
C27H34NaO8	509.2146		0.42		

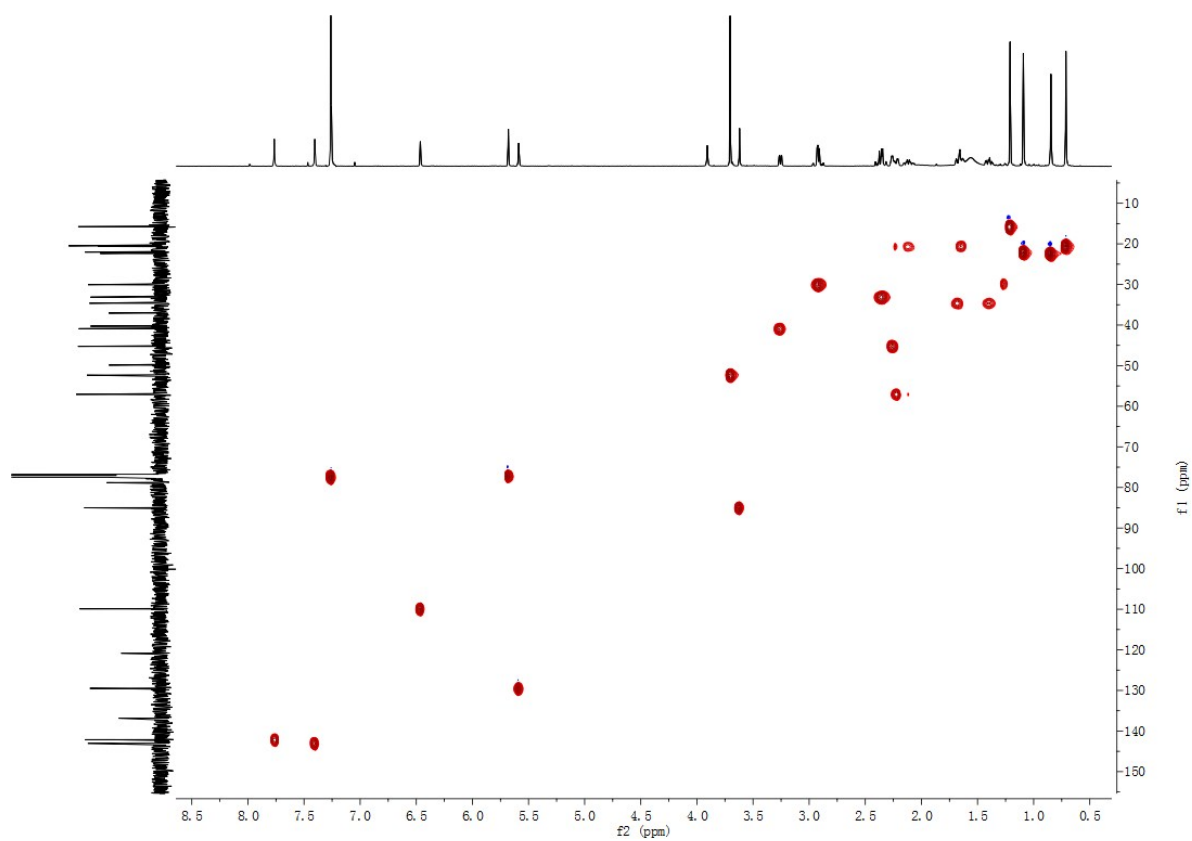
S34 ¹H NMR of Khasenegasin F (6)



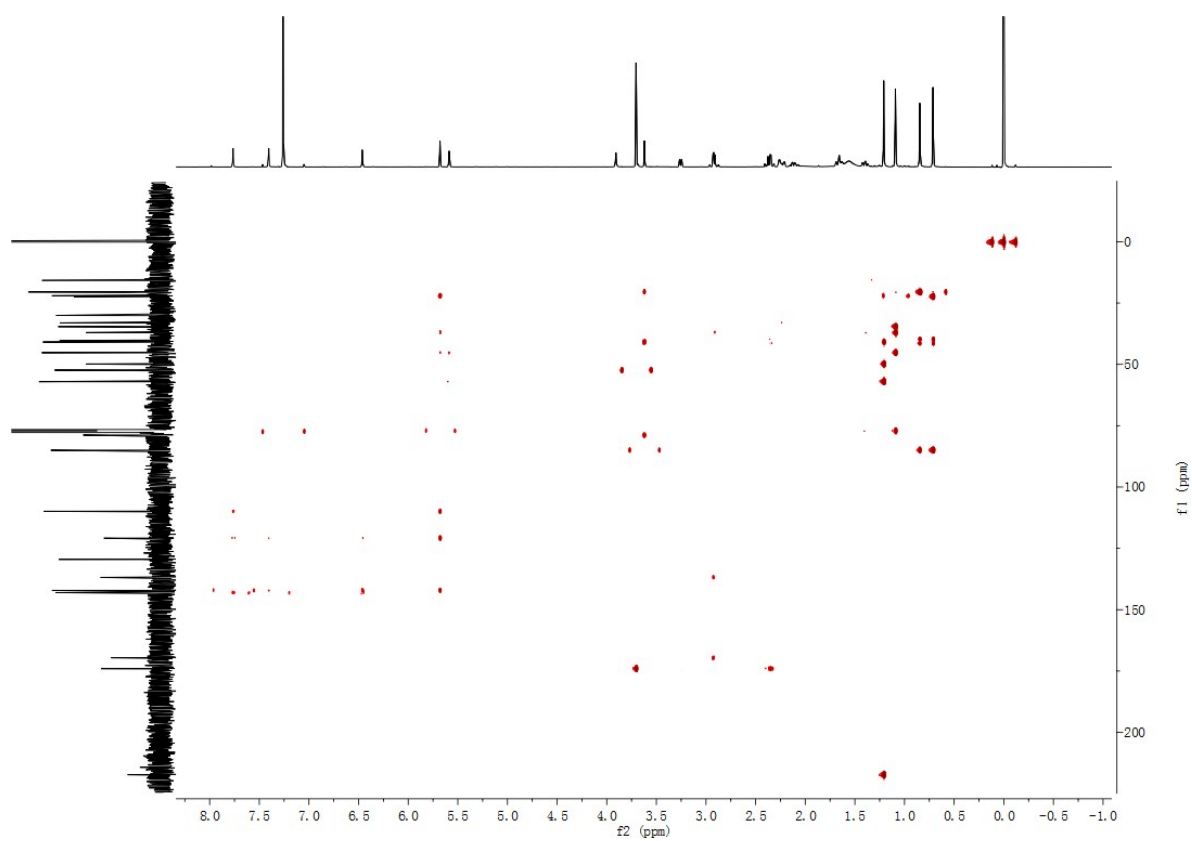
S35 ^{13}C NMR of Khasenegasin F (6)



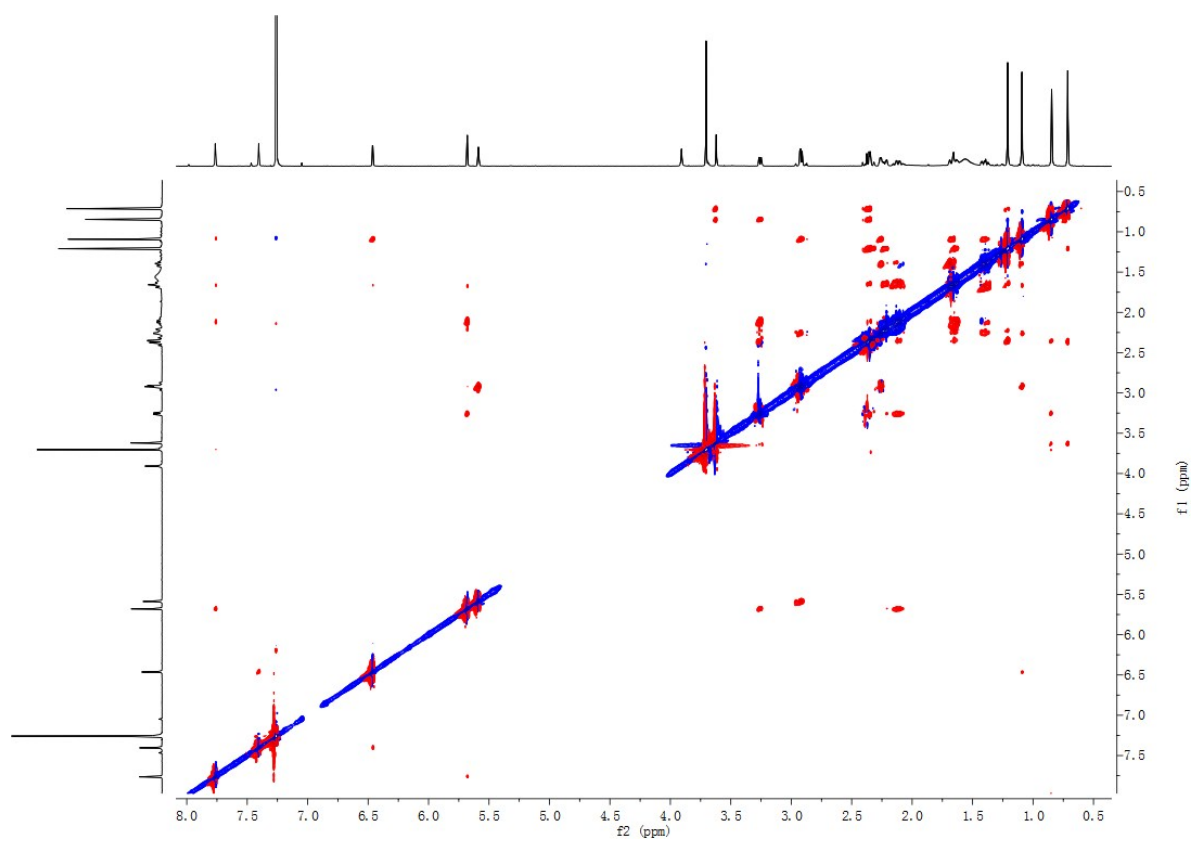
S36 HSQC of Khasenegasin F (6)



S37 HMBC of Khasenegasin F (6)

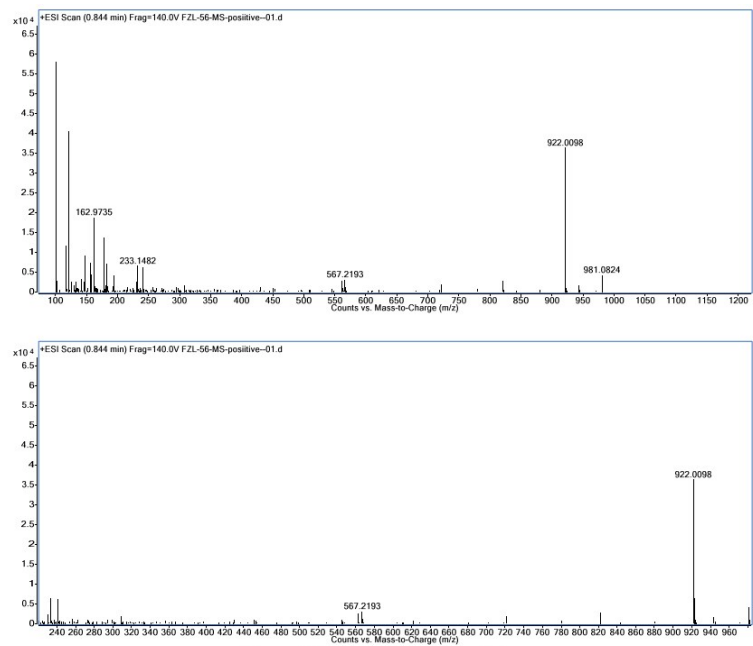


S38 ROSEY of Khasenegasin F (6)



Khasenegasin F (7)

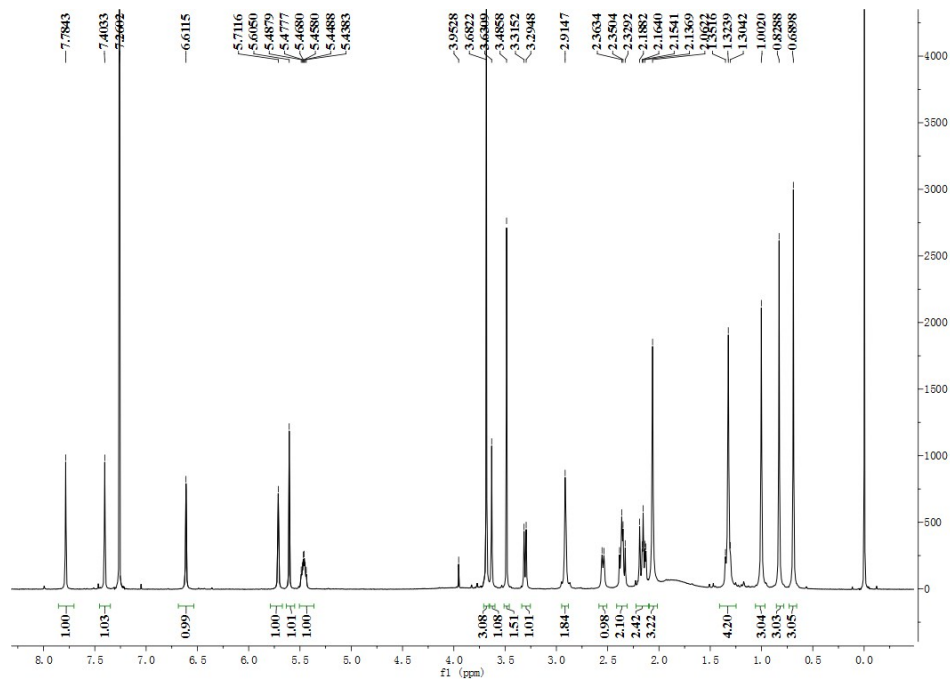
S39 HR-ESI-MS of Khasenegasin F (7)



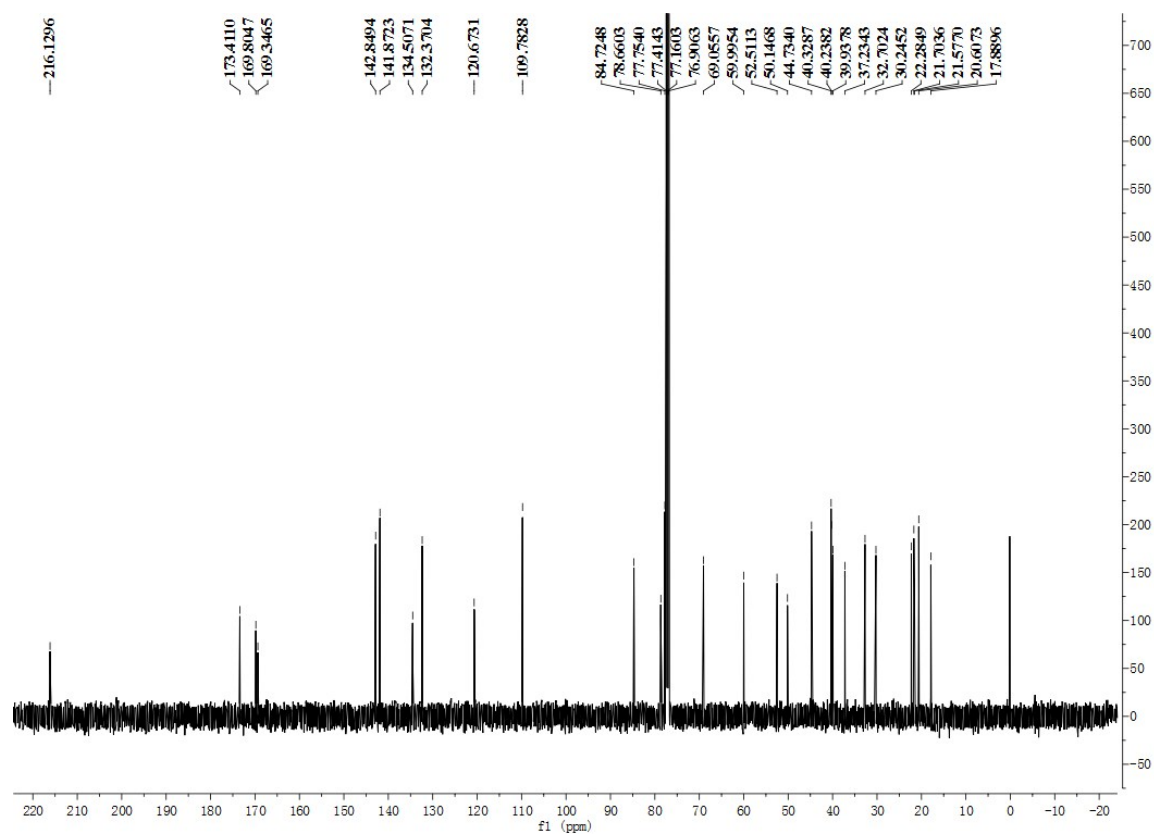
Elemental Composition Calculator

Target m/z:	567.2196	Result type:	Positive ions	Species:	[M+Na] ⁺
Elements:	C (0-80); H (0-120); O (0-30); N(0-10); Na (0-5); S (0-5)				
Ion Formula	Calculated m/z		PPM Error		
C ₂₉ H ₃₆ NaO ₁₀	567.2201		0.81		

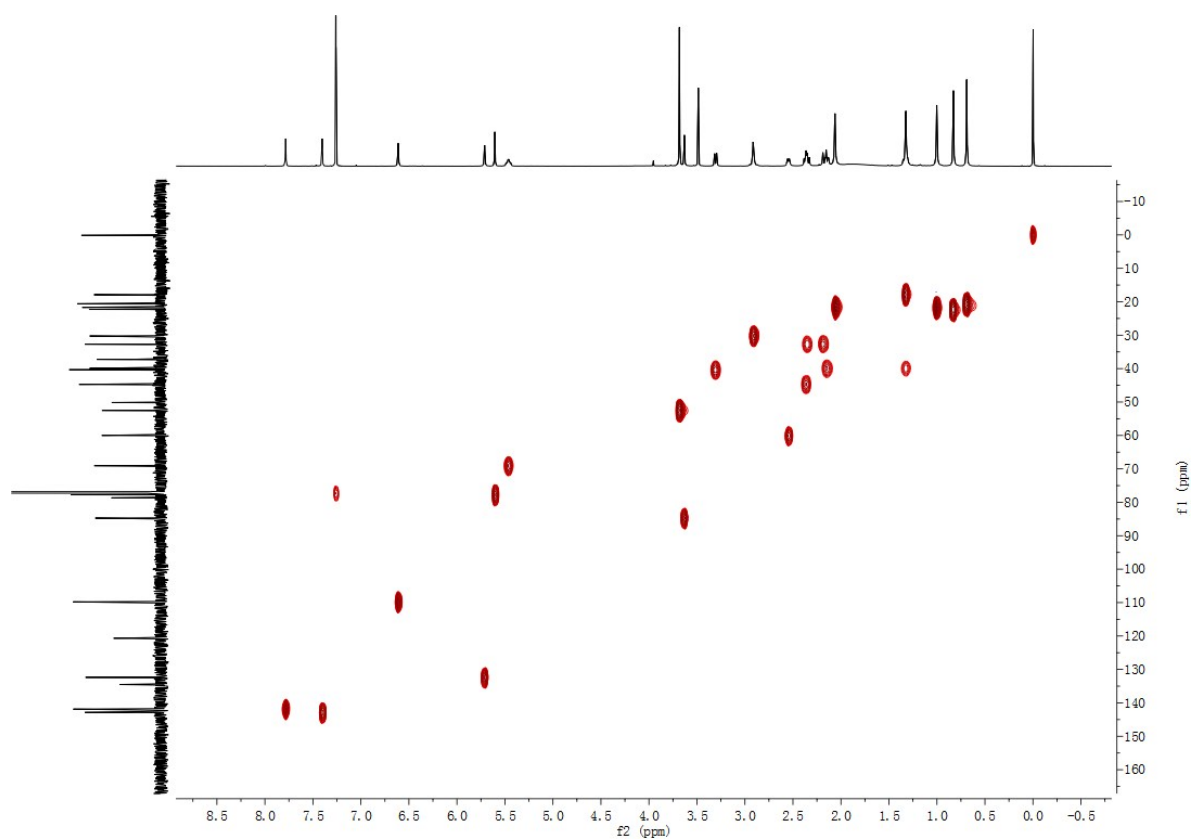
S40 ¹H NMR of Khasenegasin F (7)



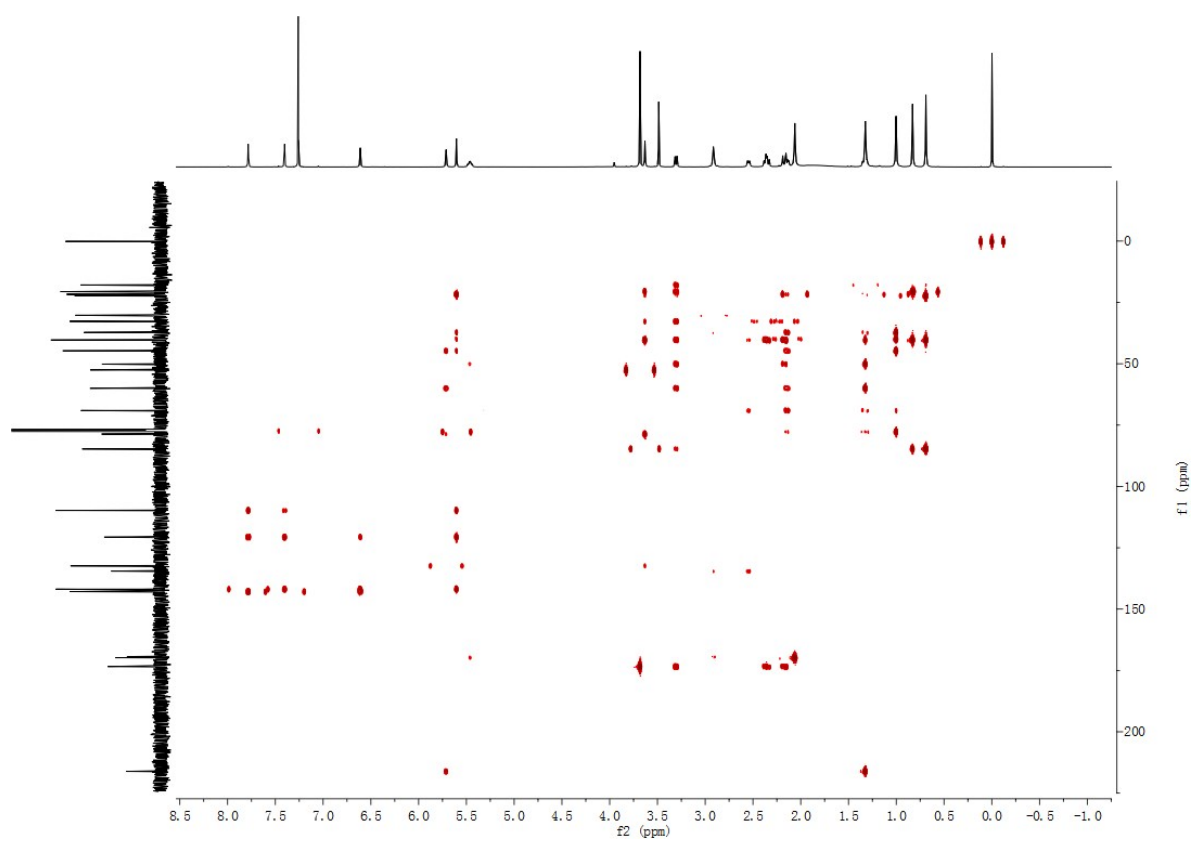
S41 ^{13}C NMR of Khasenegasin F (7)



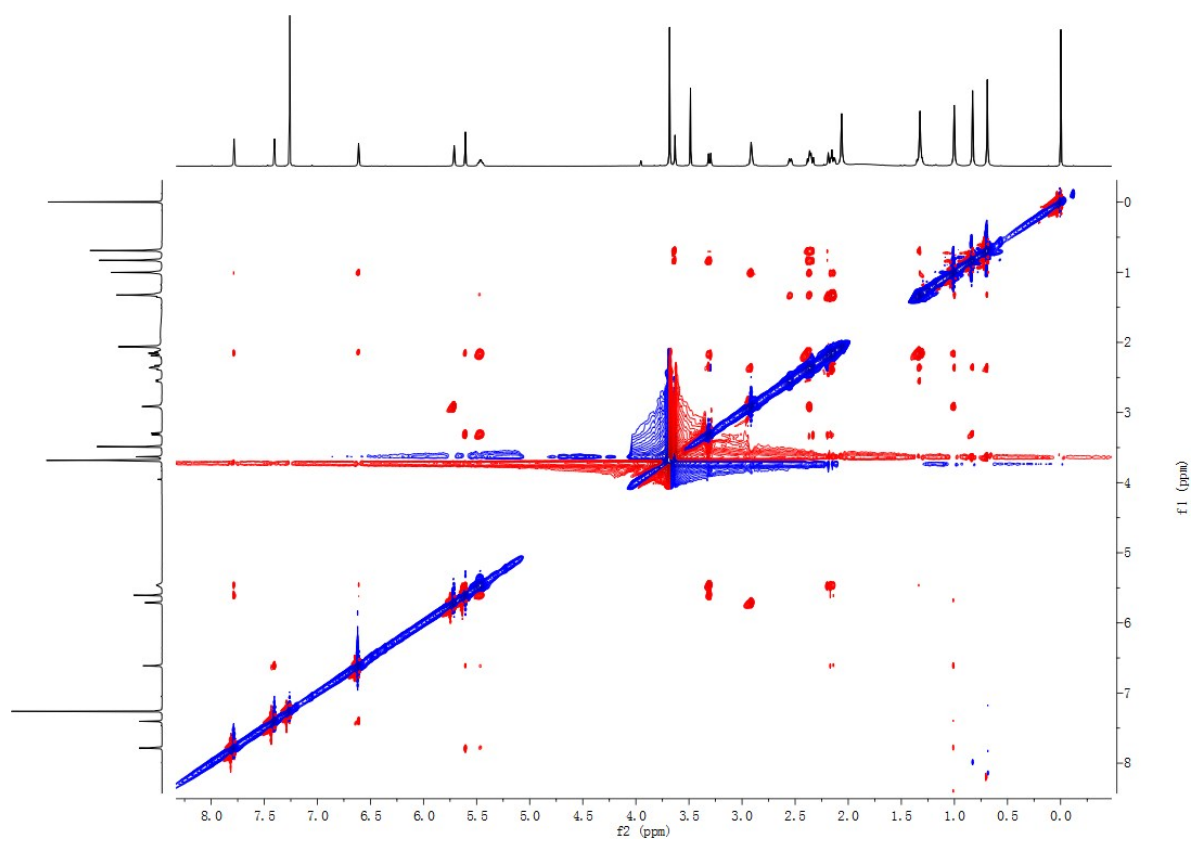
S42 HSQC of Khasenegasin F (7)



S43 HMBC of Khasenegasin F (7)

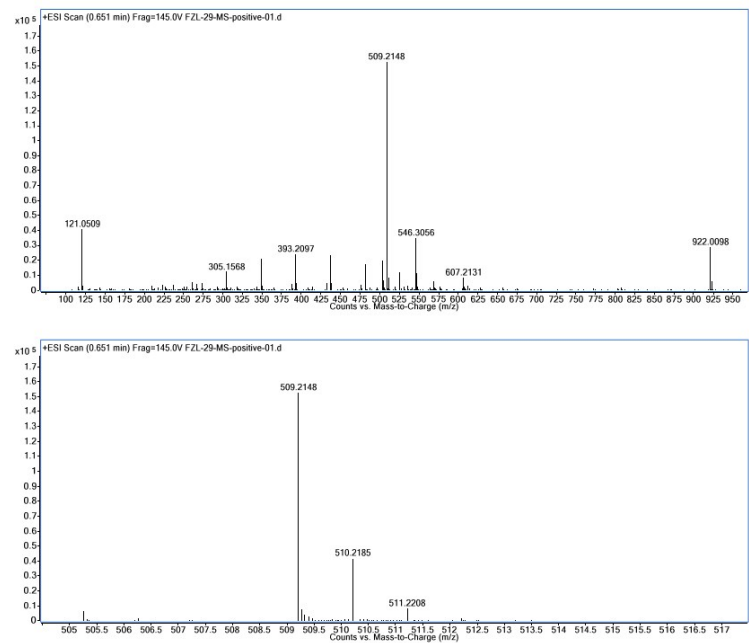


S44 ROSEY of Khasenegasin F (7)



Khasenegasin H (8)

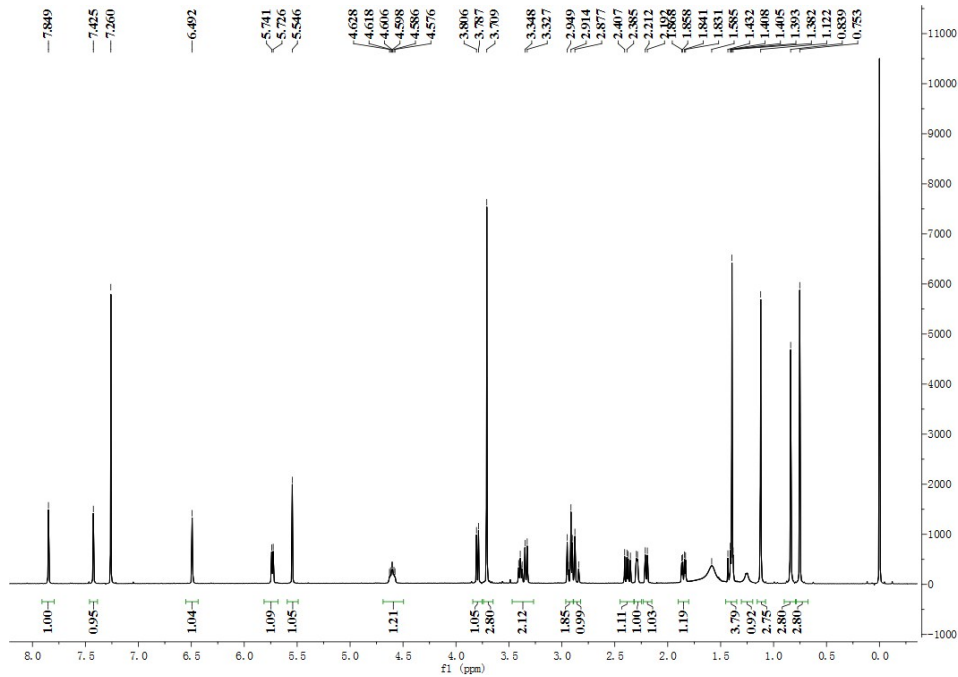
S45 HR-ESI-MS of Khasenegasin H (8)



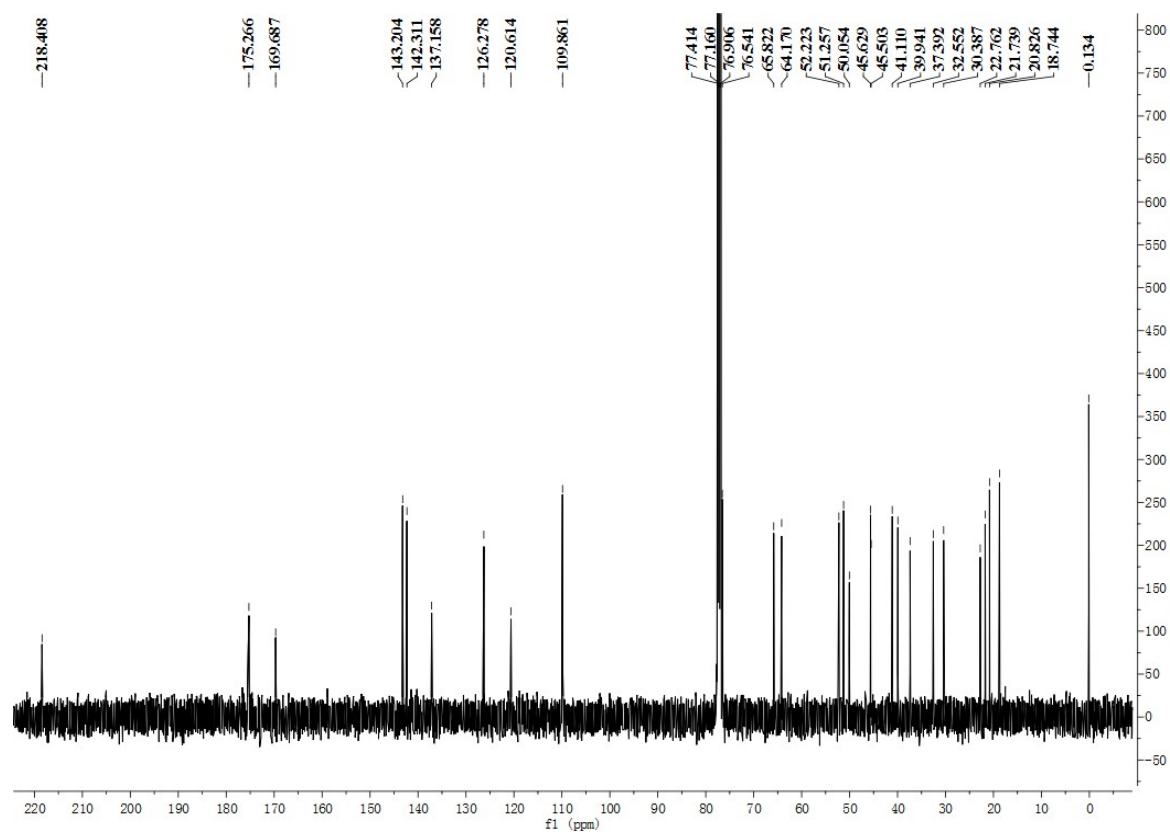
Elemental Composition Calculator

Target m/z:	509.2148	Result type:	Positive ions	Species:	[M+Na] ⁺
Elements:	C (0-80); H (0-120); O (0-30); N(0-10); Na (0-5); S (0-5)				
Ion Formula	Calculated m/z		PPM Error		
C27H34NaO8	509.2146		-0.38		

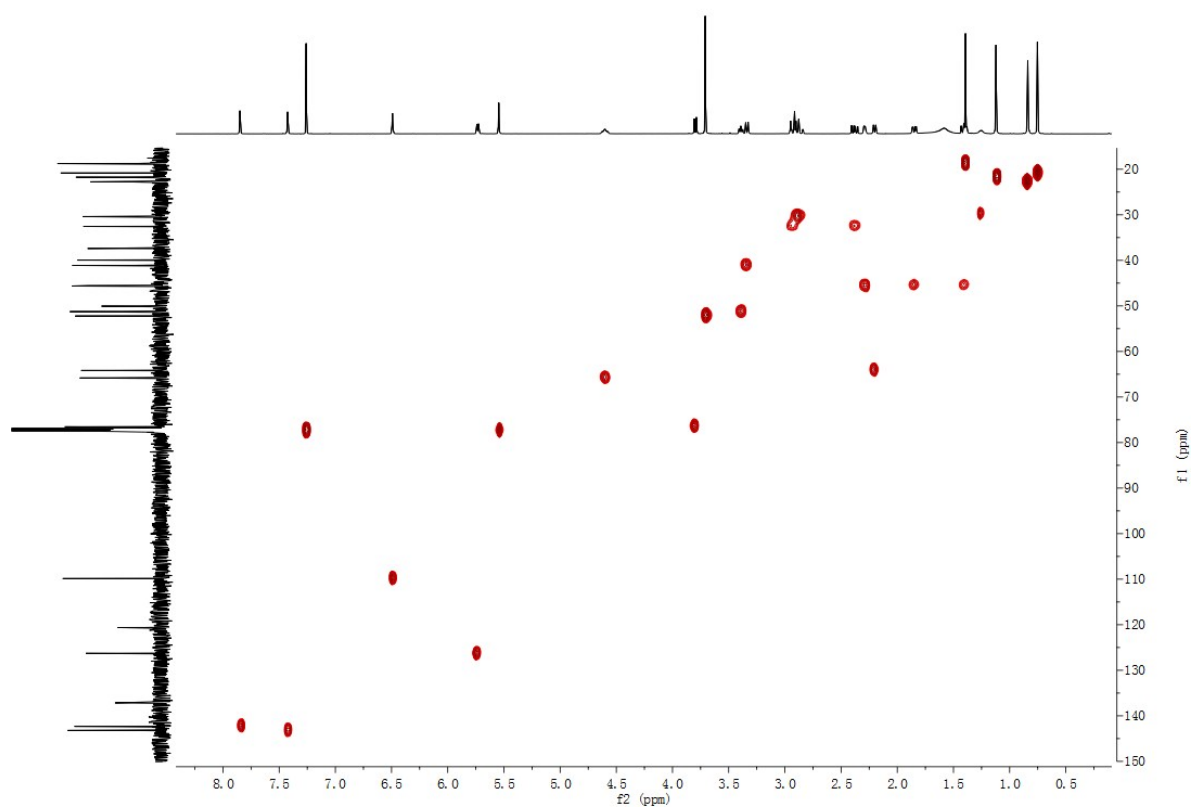
S46 ¹H NMR of Khasenegasin H (8)



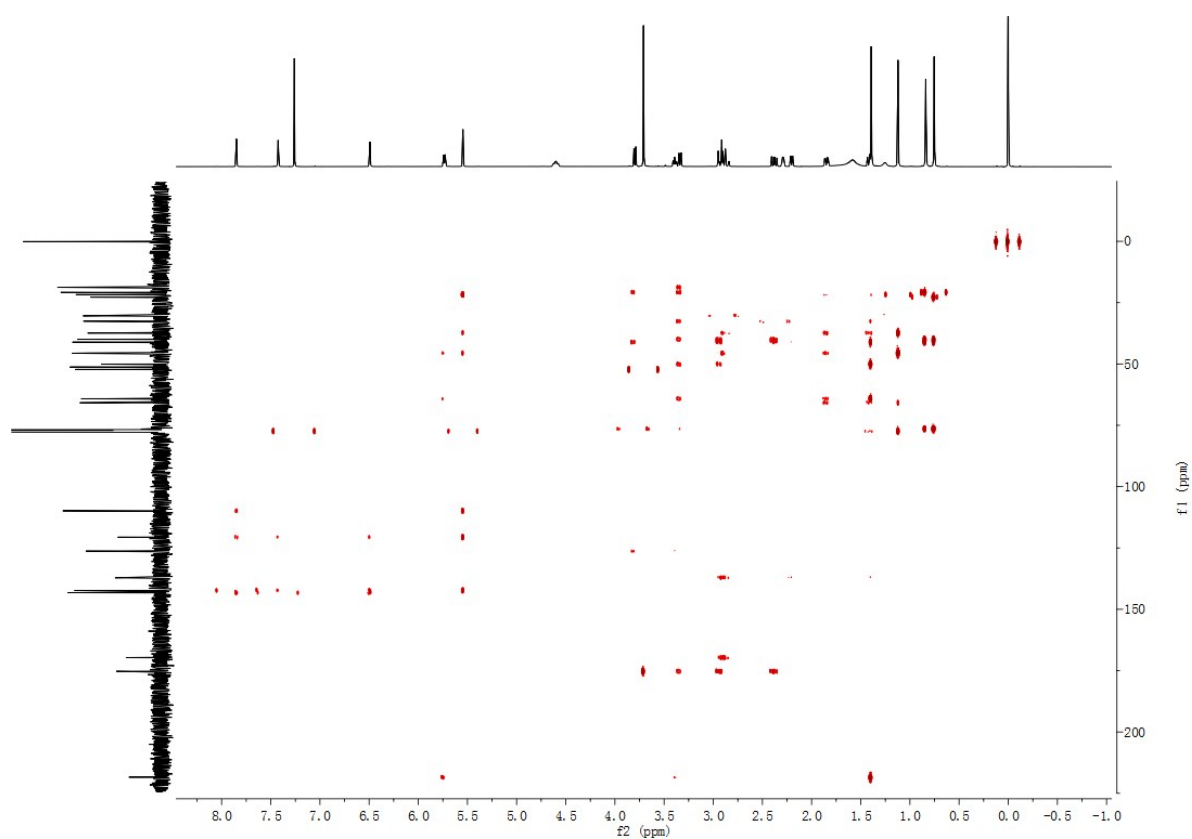
S47 ^{13}C NMR of Khasenegasin H (8)



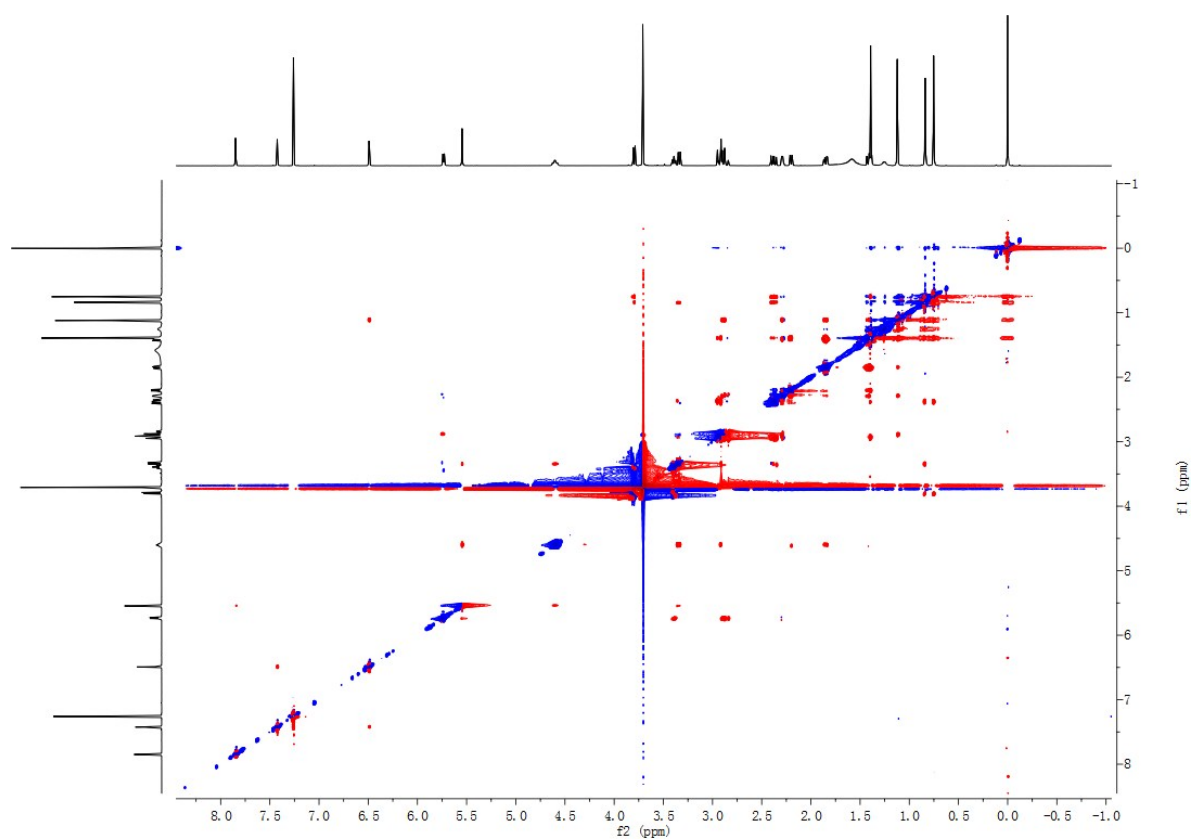
S48 HSQC of Khasenegasin H (8)



S49 HMBC of Khasenegasin H (**8**)

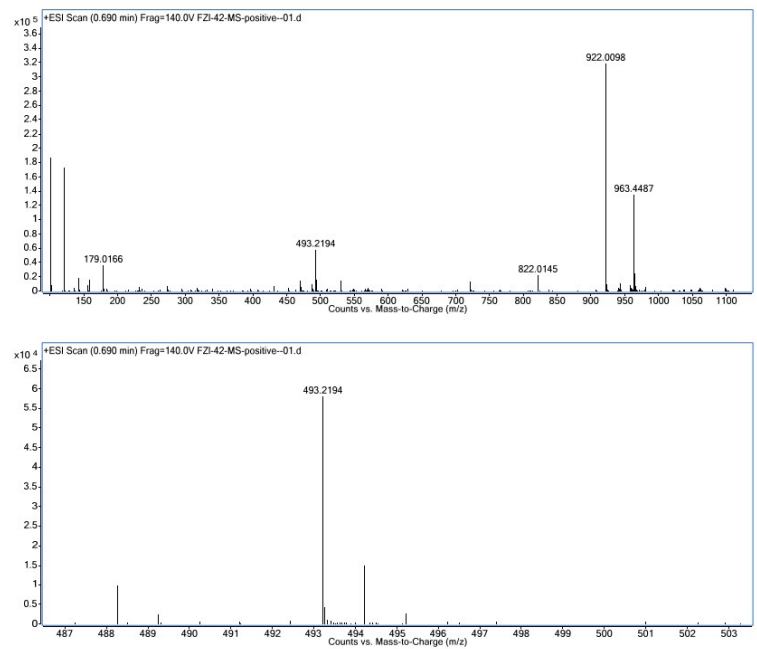


S50 ROSEY of Khasenegasin H (**8**)



Khasenegasin I (9)

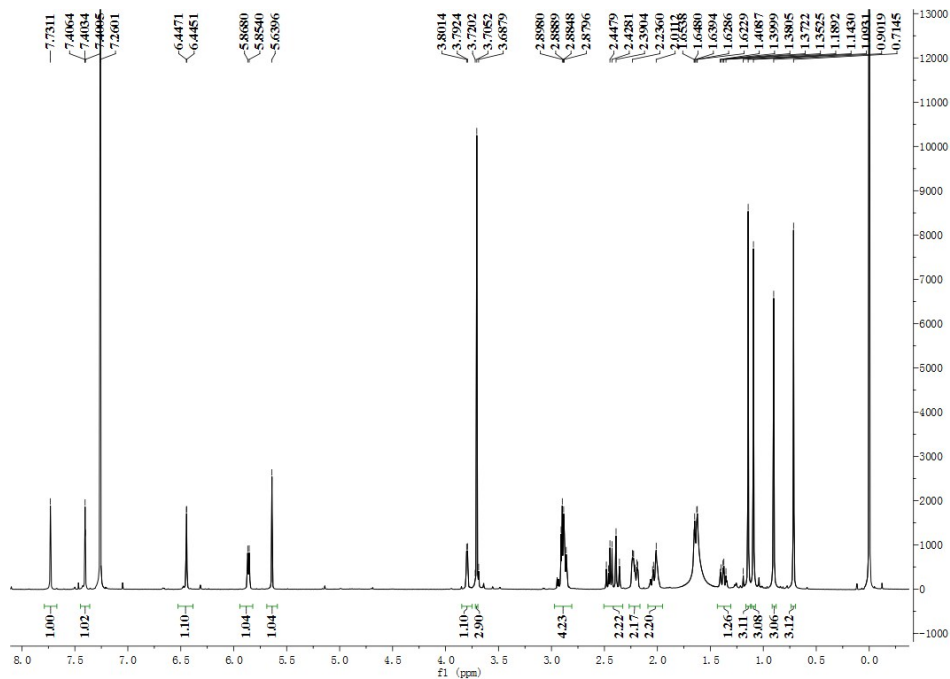
S51 HR-ESI-MS of Khasenegasin I (9)



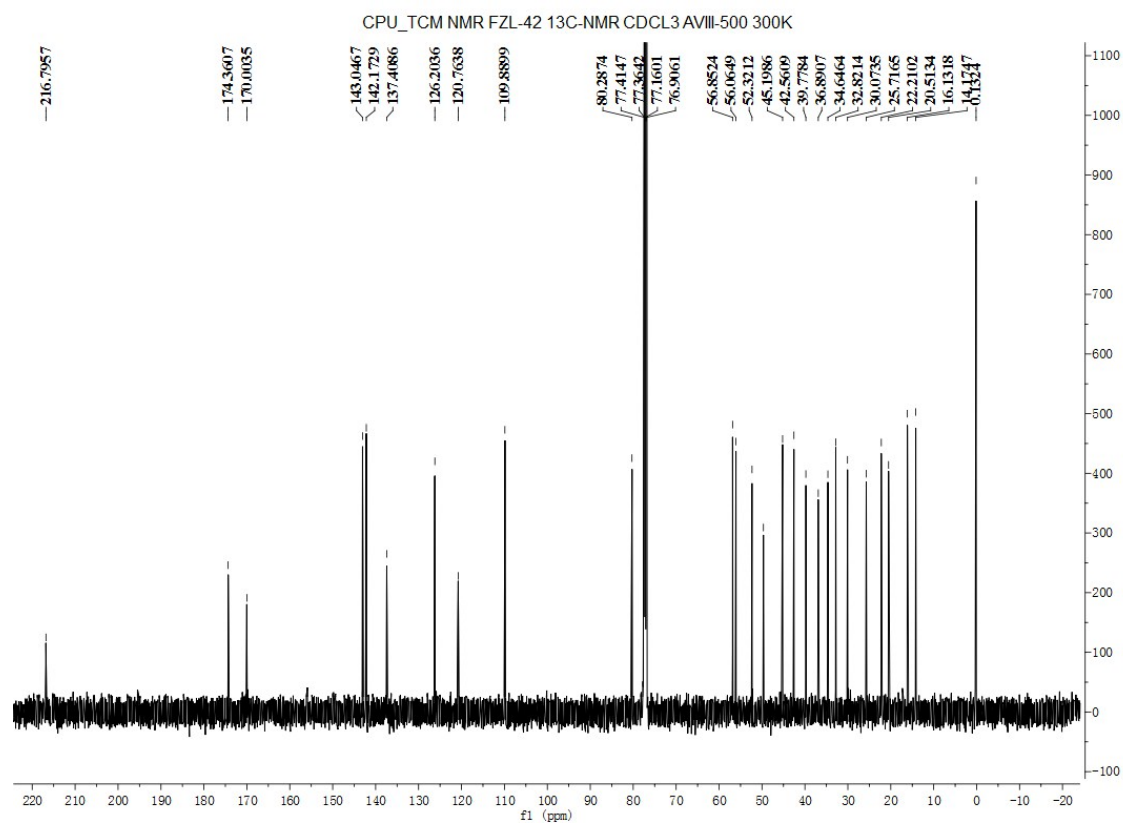
Elemental Composition Calculator

Target m/z:	493.2194	Result type:	Positive ions	Species:	[M+Na] ⁺
Elements:	C (0-80); H (0-120); O (0-30); N(0-10); Na (0-5); Cl (0-5)				
Ion Formula	Calculated m/z		PPM Error		
C27H34NaO7	493.2197		0.51		

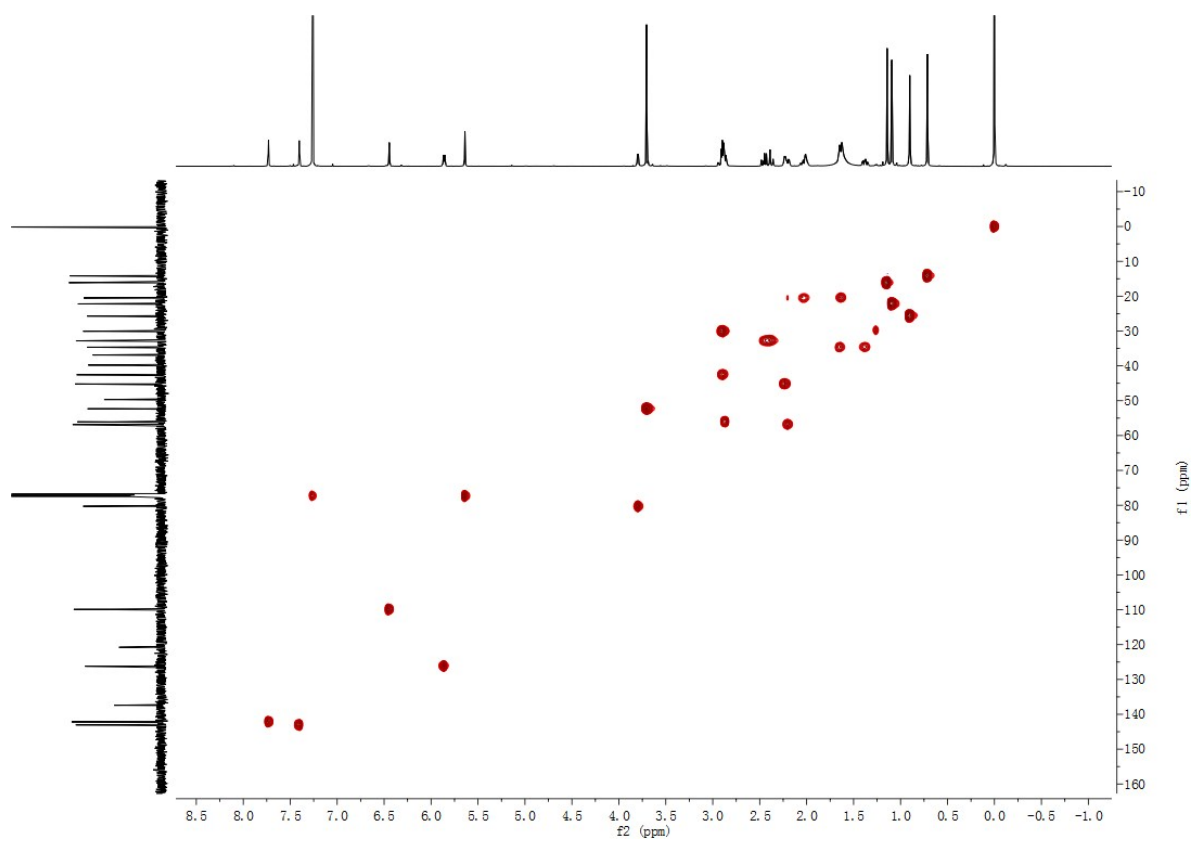
S52 ¹H NMR of Khasenegasin I (9)



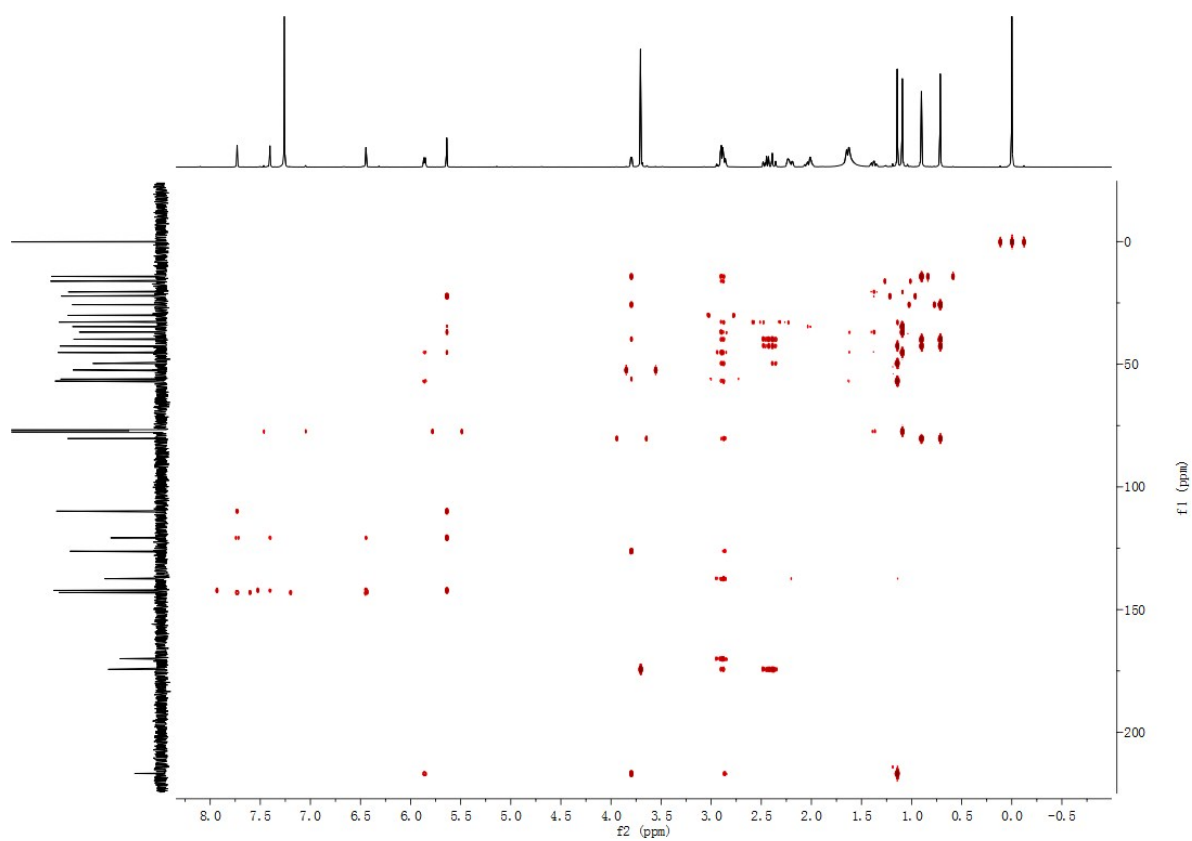
S53 ^{13}C NMR of Khasenegasin I (9)



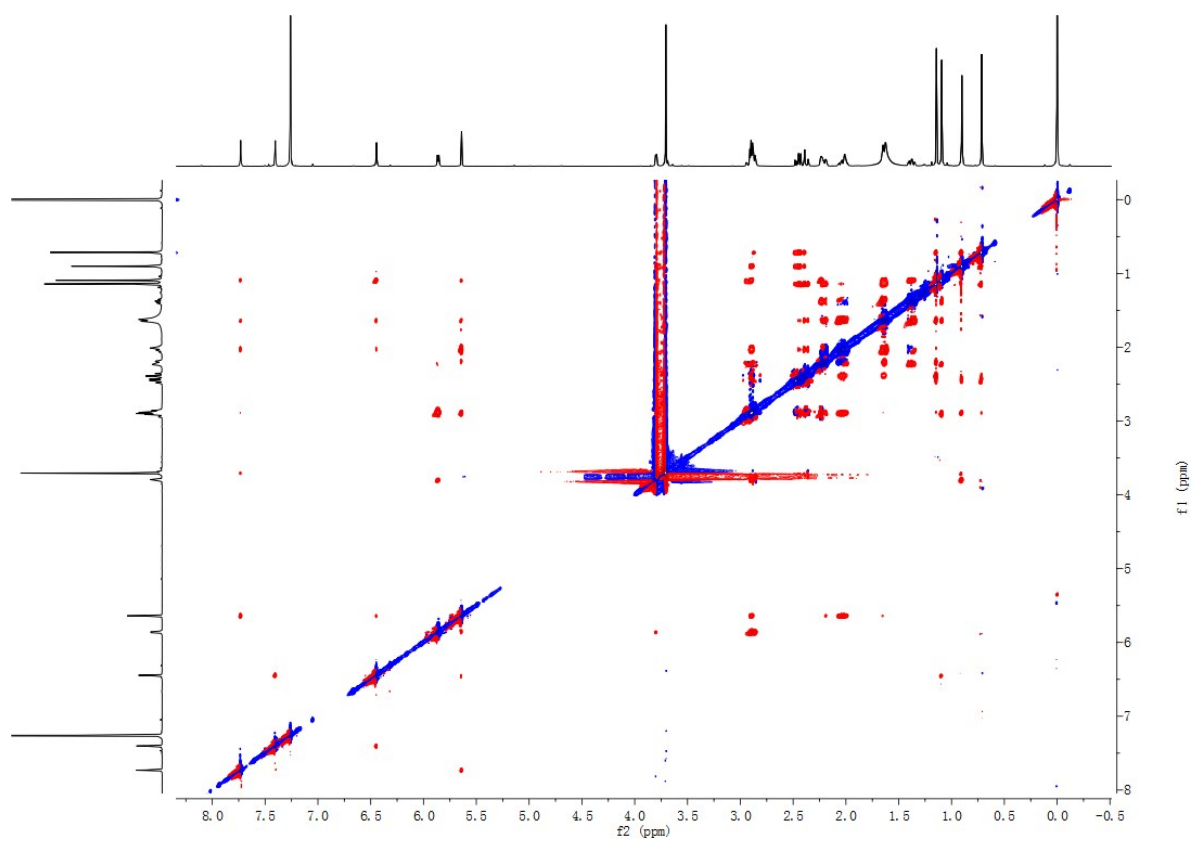
S54 HSQC of Khasenegasin I (9)



S55 HMBC of Khasenegasin I (9)

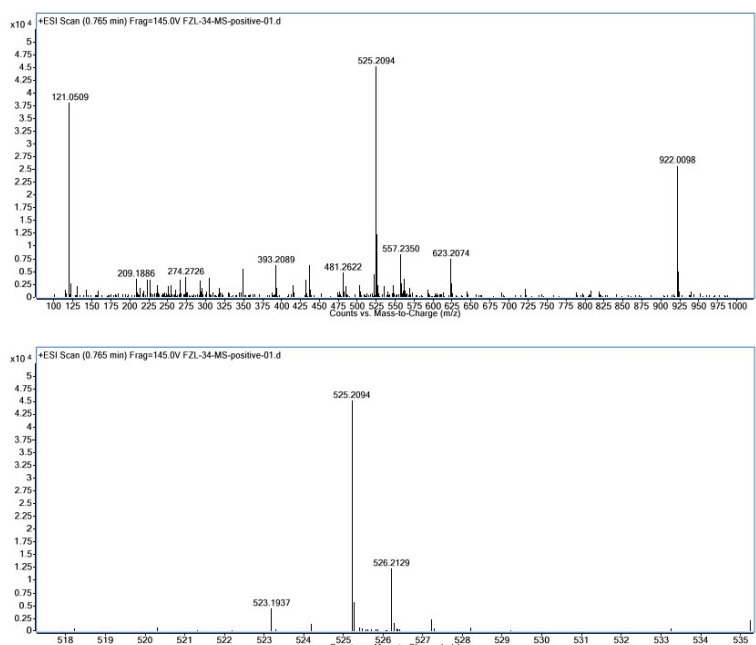


S56 ROSEY of Khasenegasin I (9)



Khasenegasin J (10)

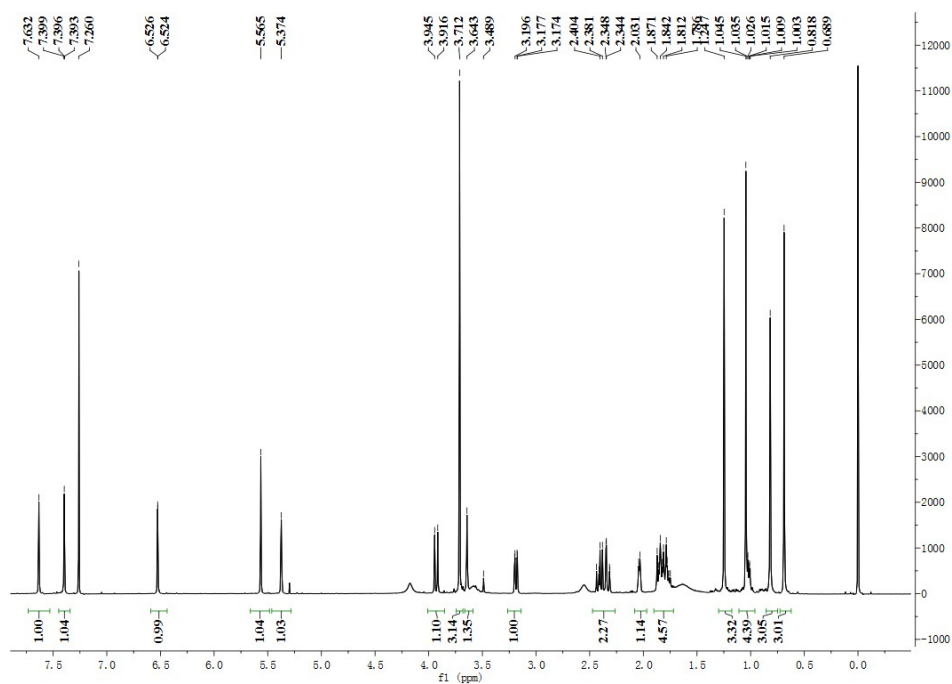
S57 HR-ESI-MS of Khasenegasin J (10)



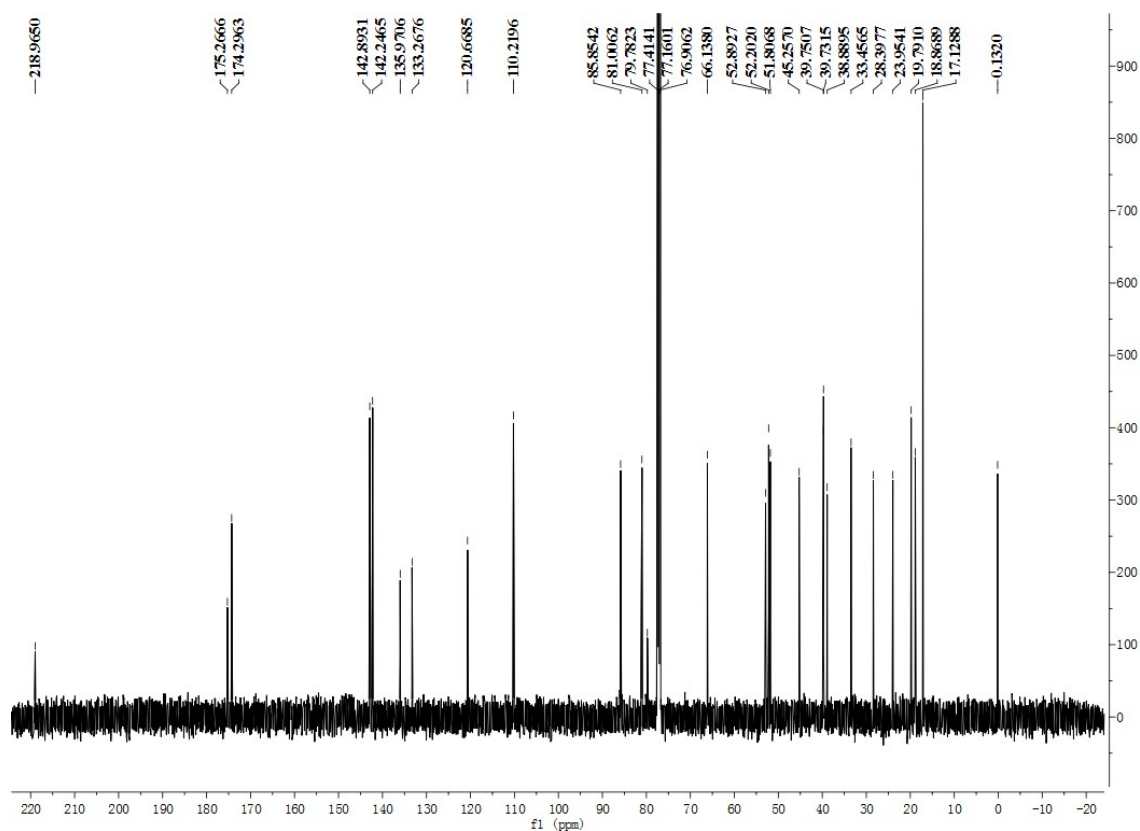
Elemental Composition Calculator

Target m/z:	525.2094	Result type:	Positive ions	Species:	[M+Na] ⁺
Elements:	C (0-80); H (0-120); O (0-30); N(0-10); Na (0-5); S (0-5)				
Ion Formula	Calculated m/z		PPM Error		
C27H34NaO9	525.2095		0.19		

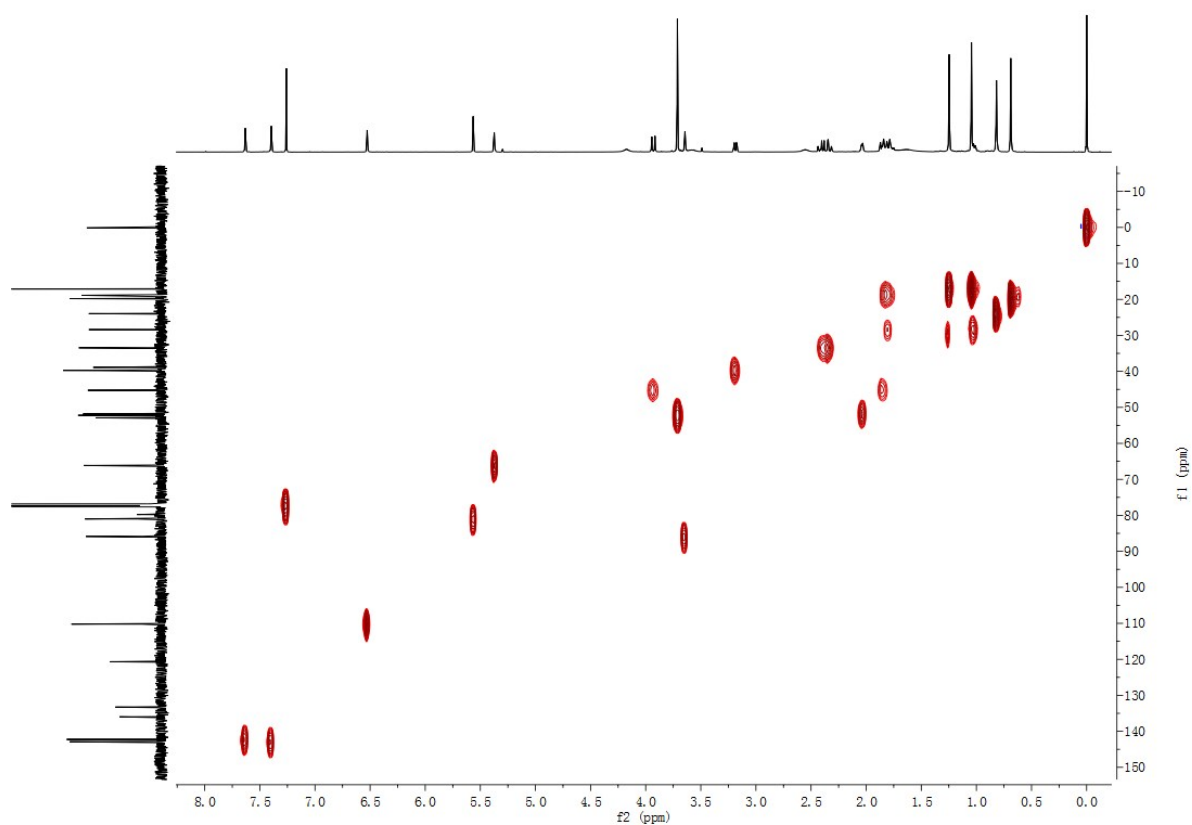
S58 ¹H NMR of Khasenegasin J (10)



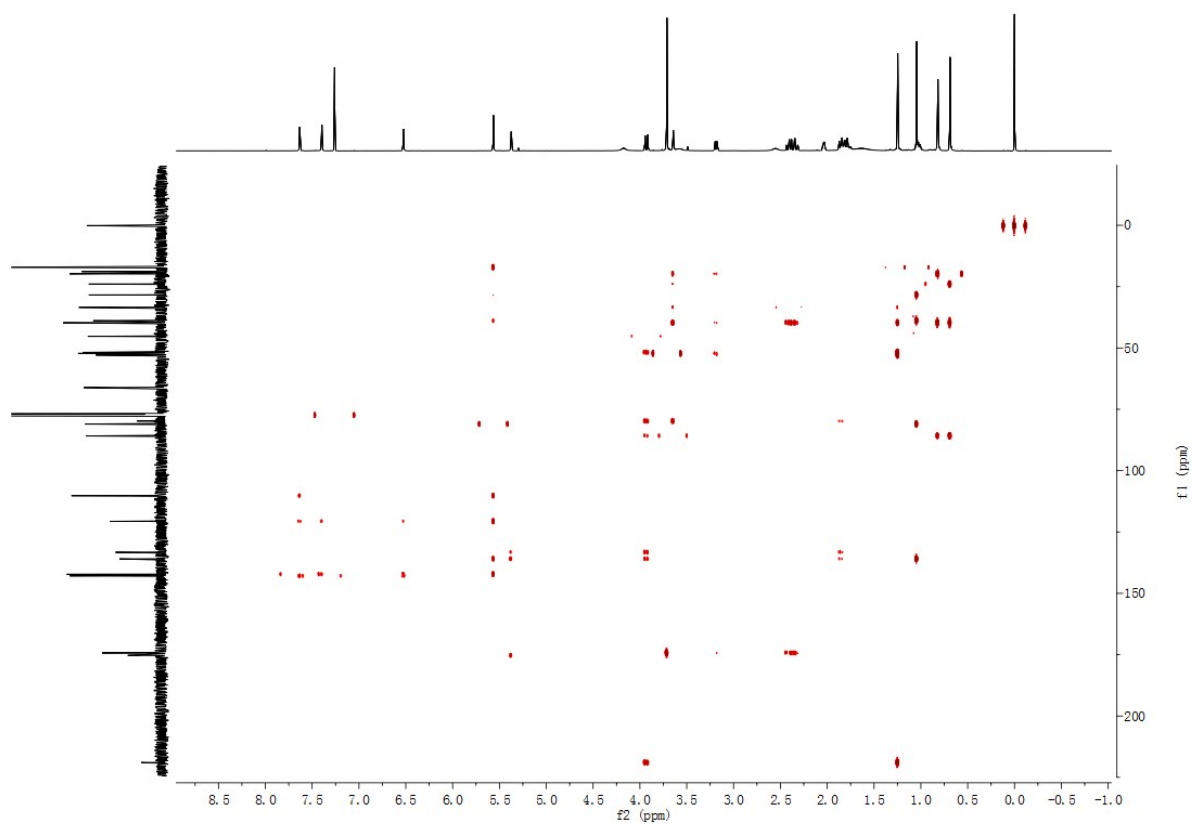
S59 ^{13}C NMR of Khasenegasin J (**10**)



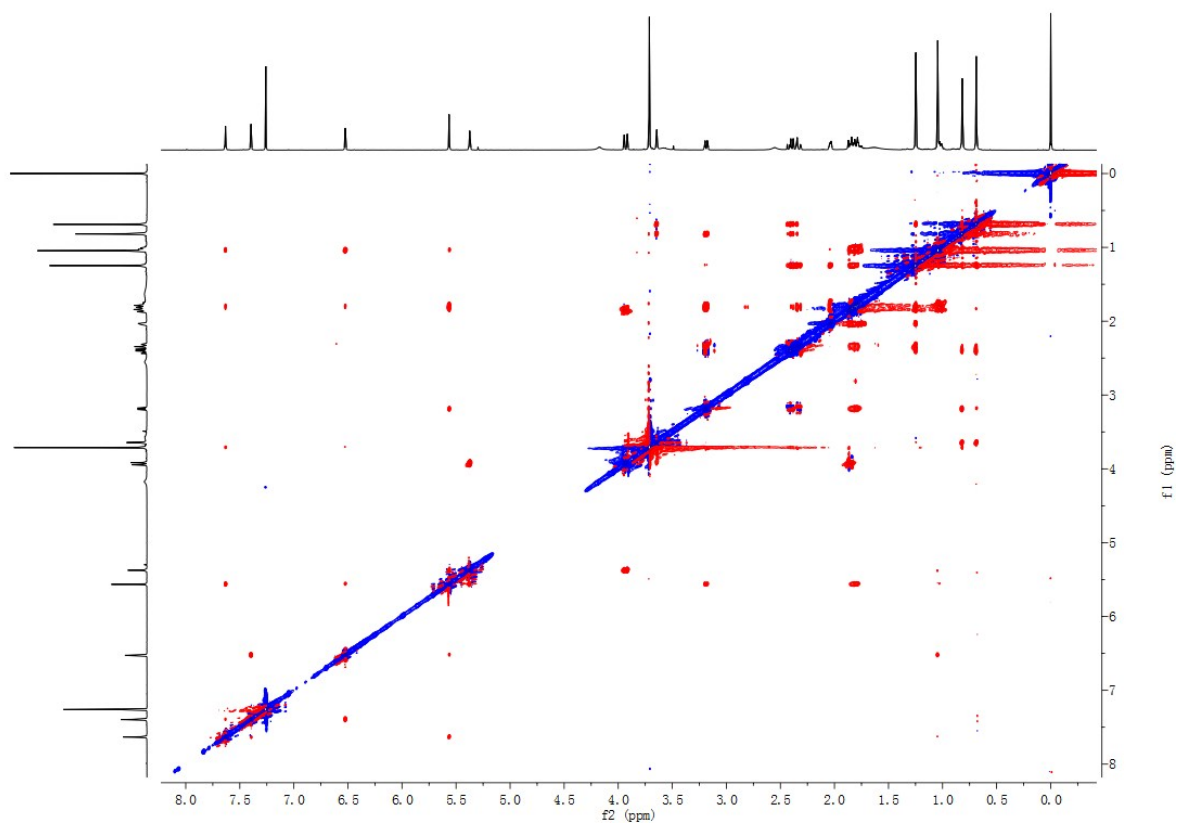
S60 HSQC of Khasenegasin J (**10**)



S61 HMBC of Khasenegasin J (10)

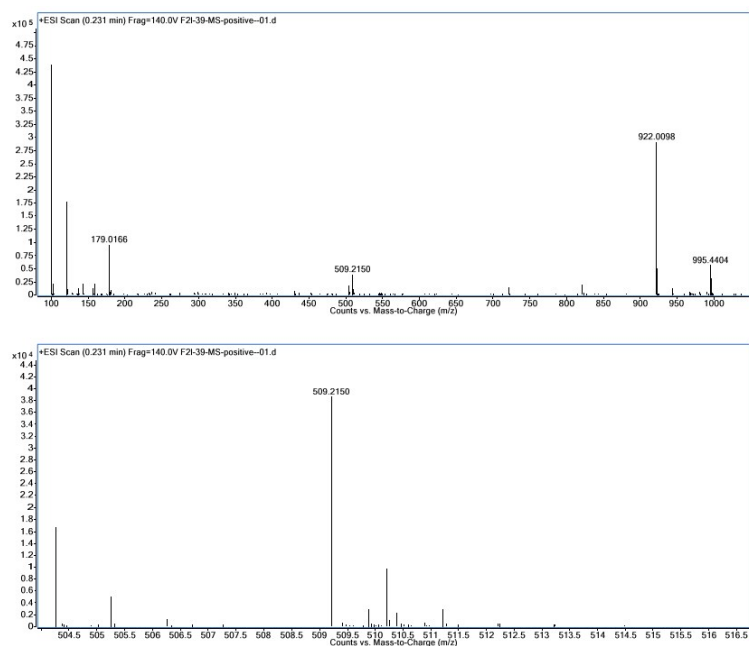


S62 ROSEY of Khasenegasin J (10)



Khasenegasin K (11)

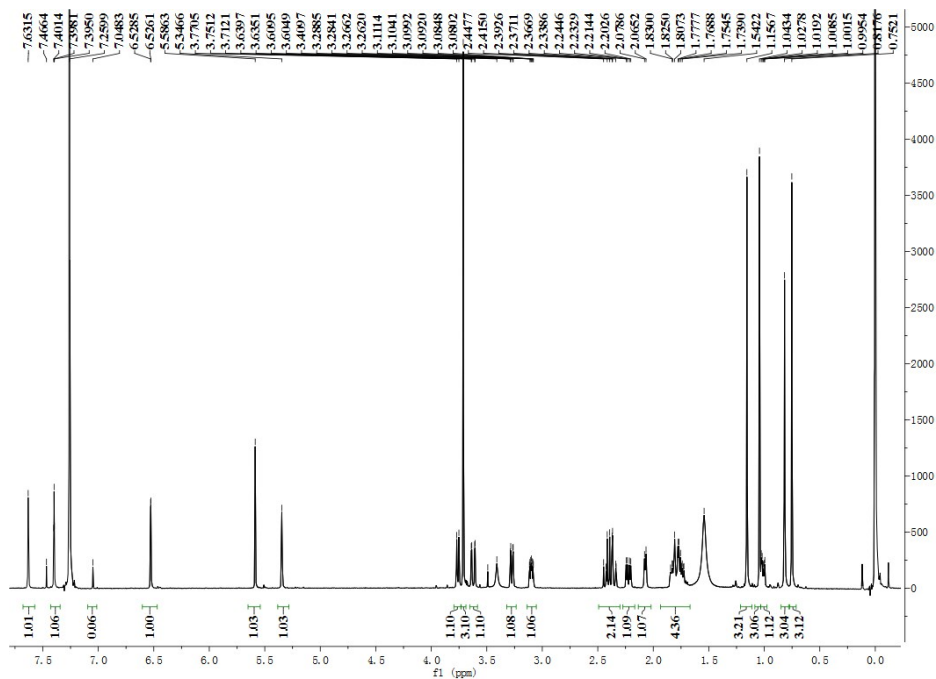
S63 HR-ESI-MS of Khasenegasin K (11)



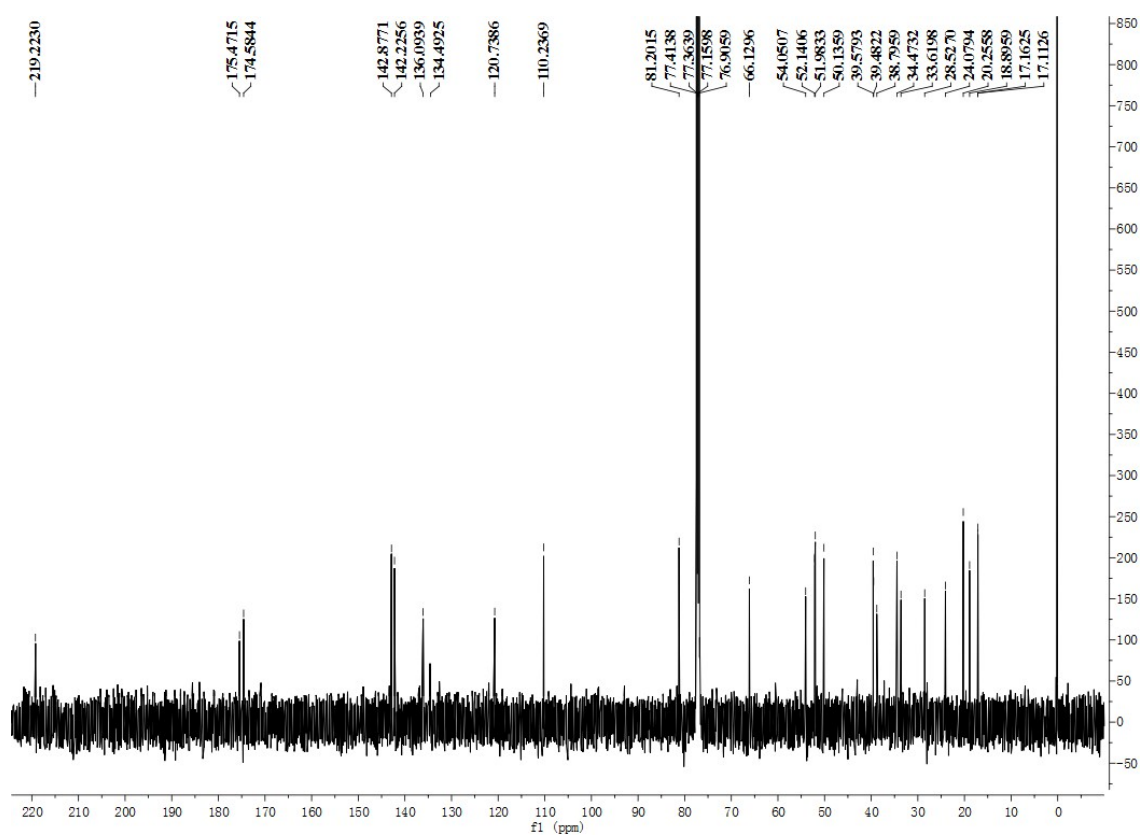
Elemental Composition Calculator

Target m/z:	509.2150	Result type:	Positive ions	Species:	[M+Na] ⁺
Elements:	C (0-80); H (0-120); O (0-30); N(0-10); Na (0-5); S (0-5)				
Ion Formula	Calculated m/z		PPM Error		
C ₂₇ H ₃₄ NaO ₈	509.2146		-0.83		

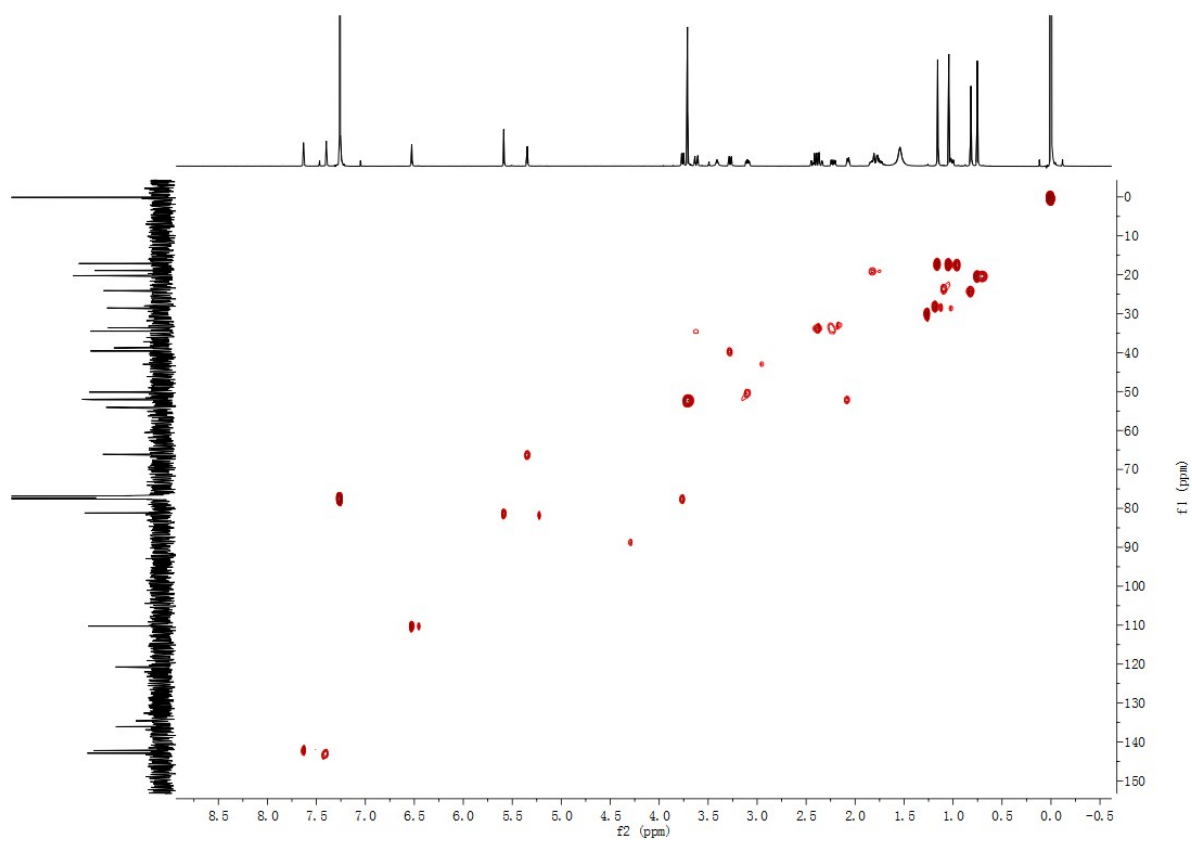
S64 ¹H NMR of Khasenegasin K (11)



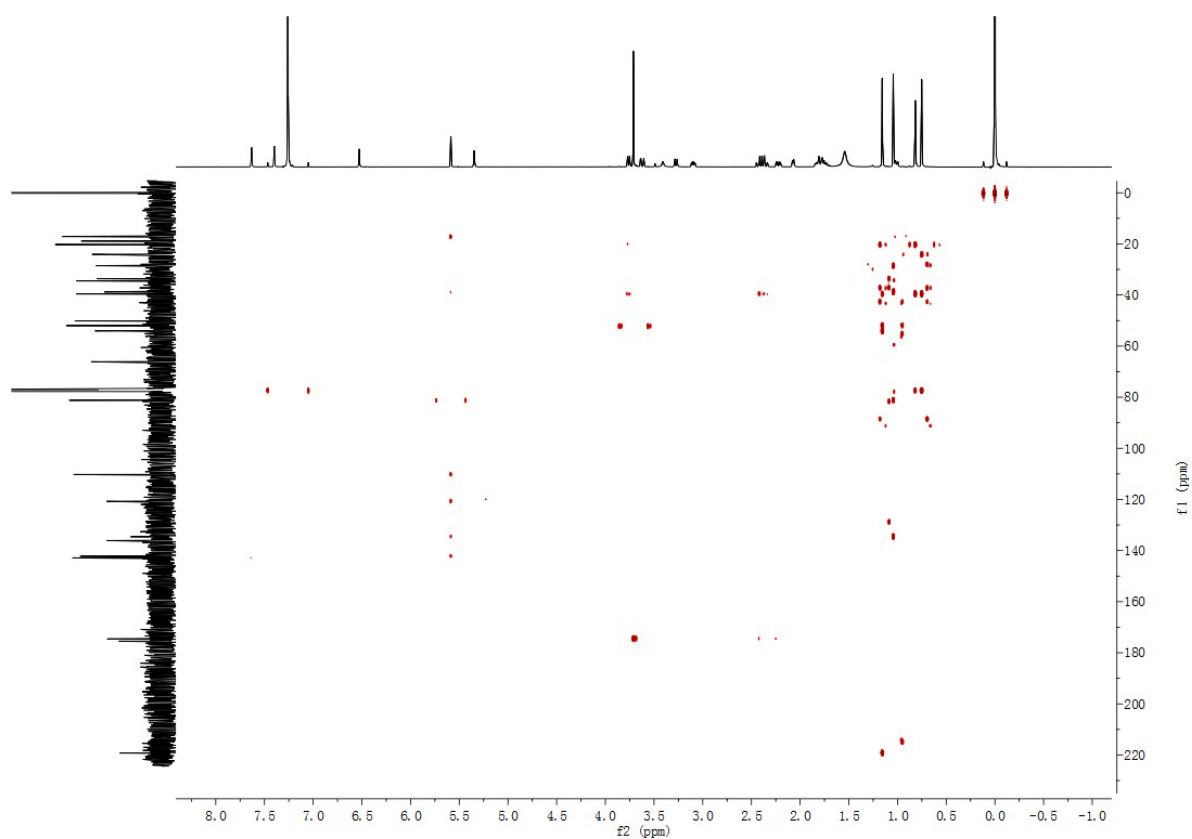
S65 ^{13}C NMR of Khasenegasin K (**11**)



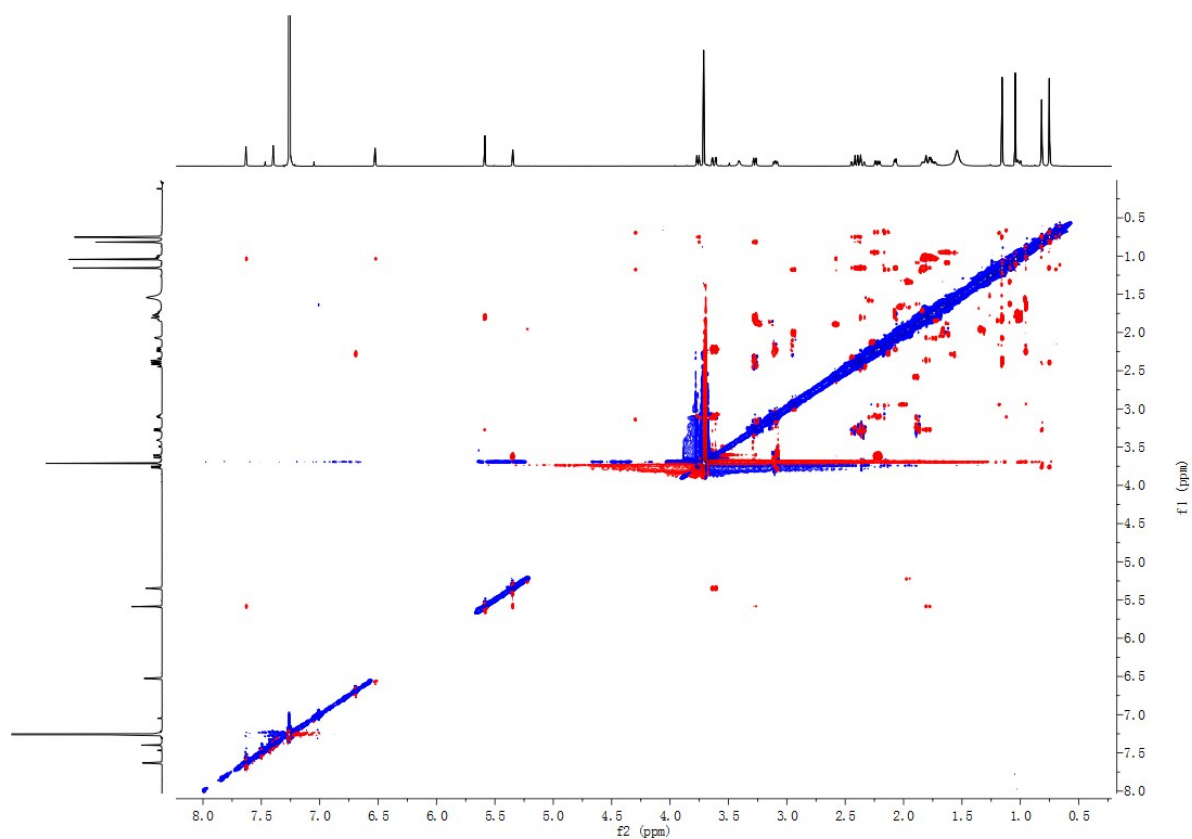
S66 HSQC of Khasenegasin K (**11**)



S67 HMBC of Khasenegasin K (**11**)

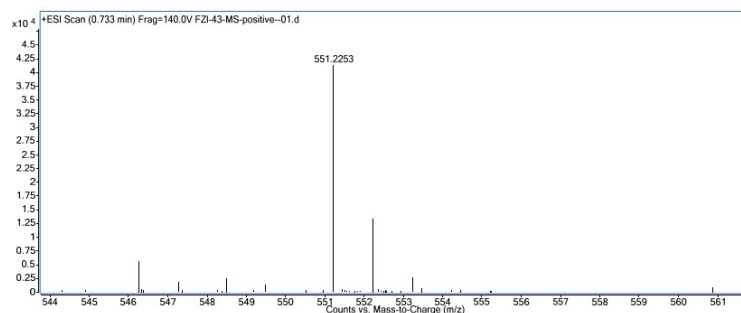
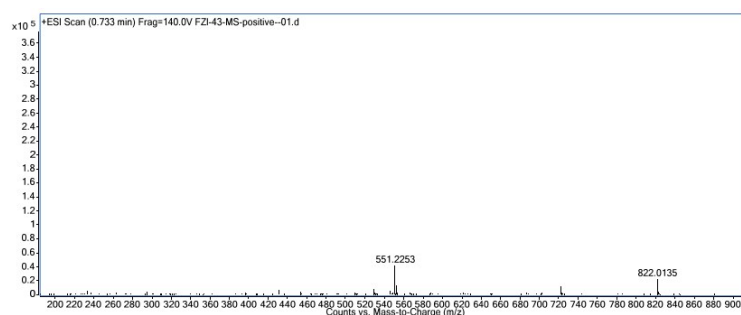


S68 ROSEY of Khasenegasin K (**11**)



Khasenegasin L (12)

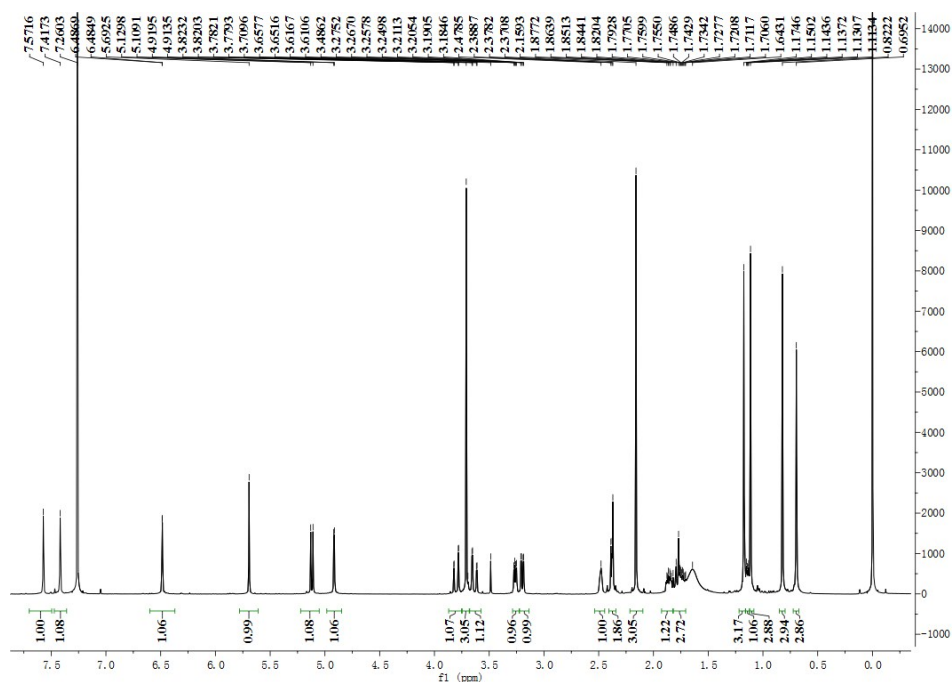
S69 HR-ESI-MS of Khasenegasin L (12)



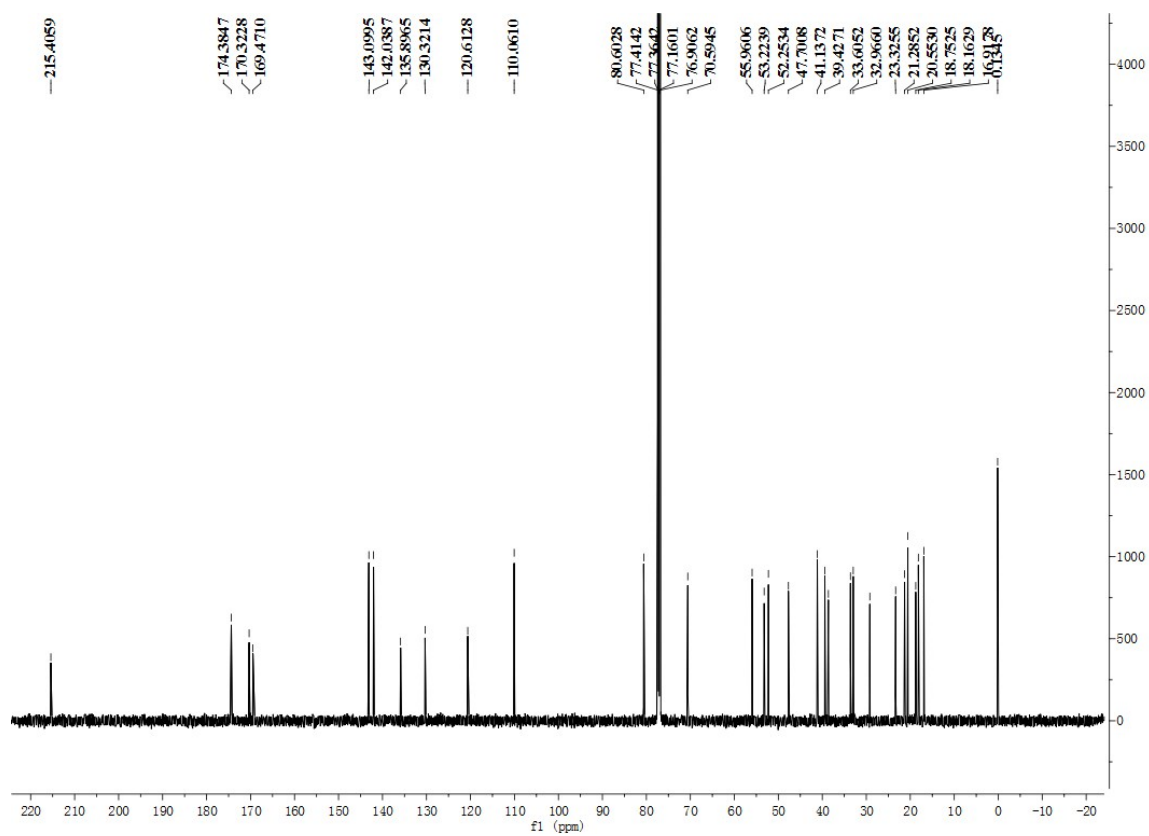
Elemental Composition Calculator

Target m/z:	551.2253	Result type:	Positive ions	Species:	[M+Na] ⁺
Elements:	C (0-80); H (0-120); O (0-30); N(0-10); Na (0-5); S (0-5)				
Ion Formula	Calculated m/z		PPM Error		
C ₂₉ H ₃₆ NaO ₉	551.2252		-0.32		

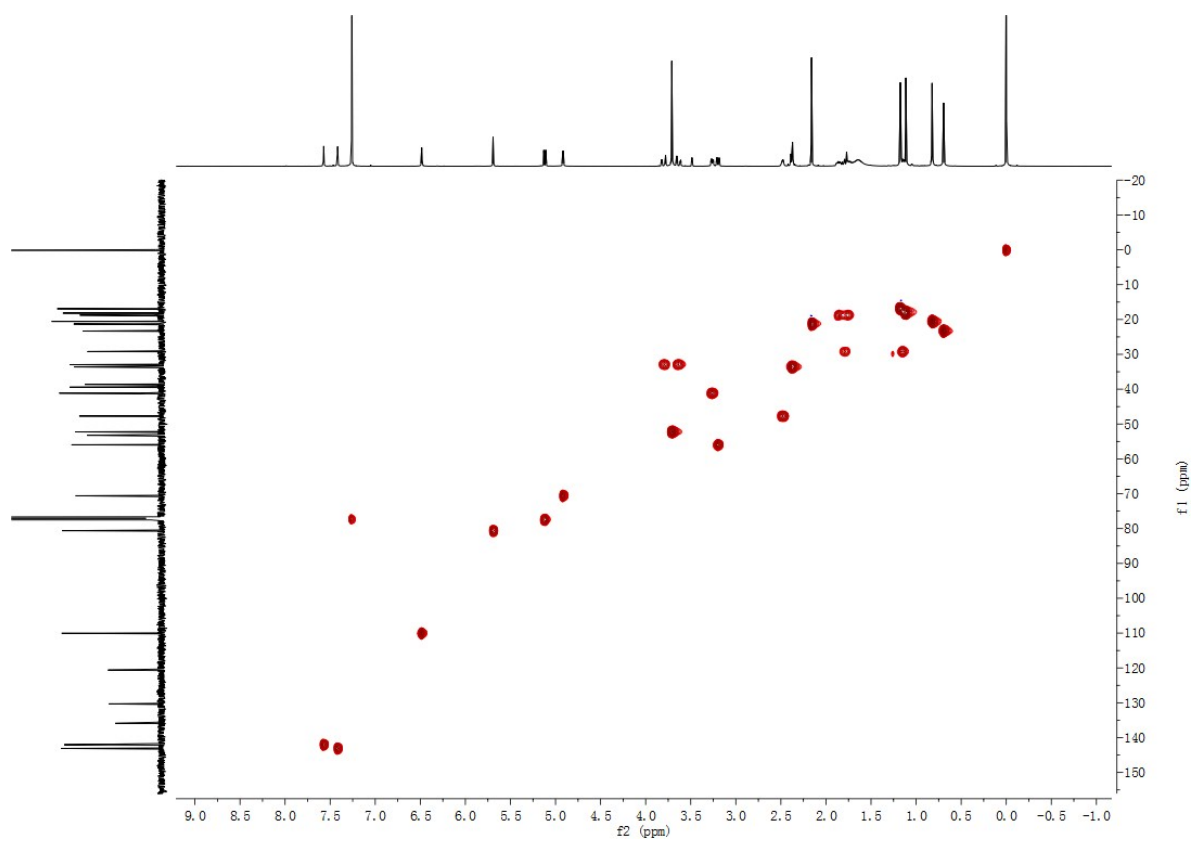
S70 ¹H NMR of Khasenegasin L (12)



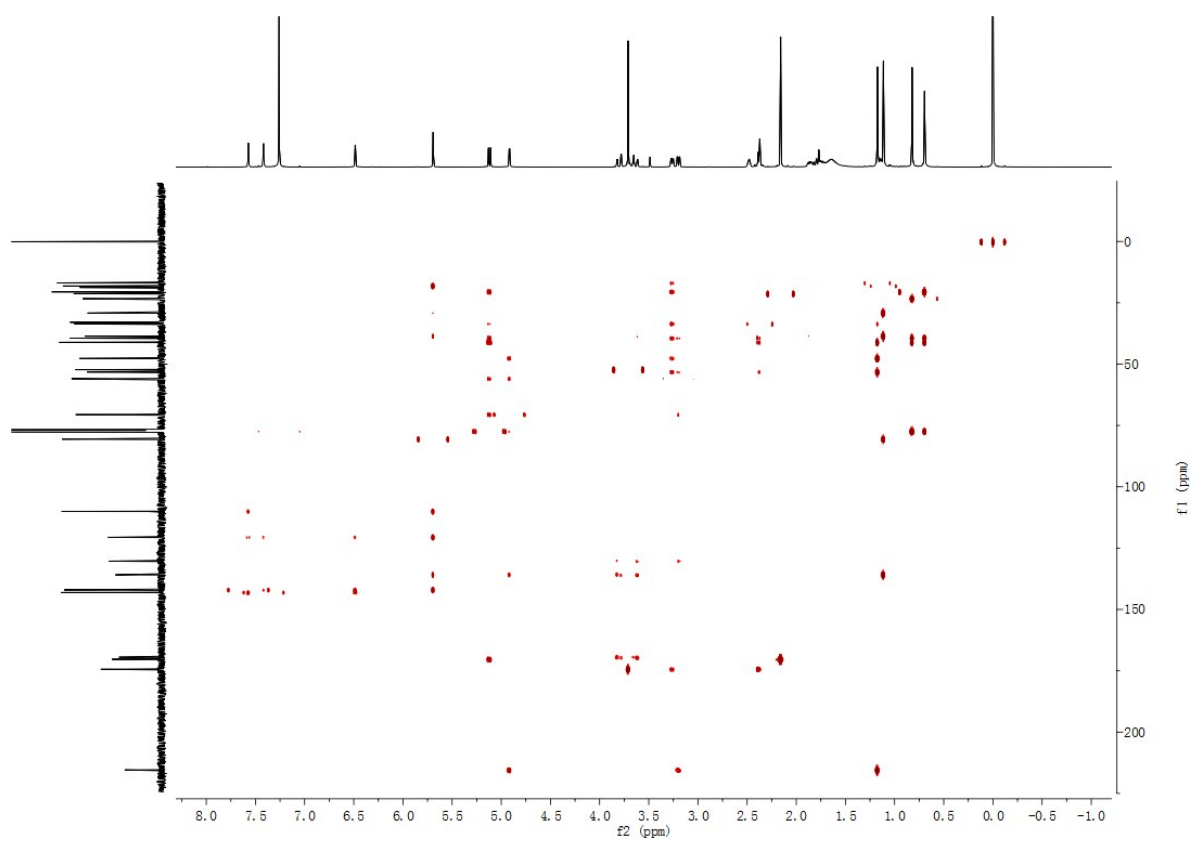
S71 ^{13}C NMR of Khasenegasin L (12)



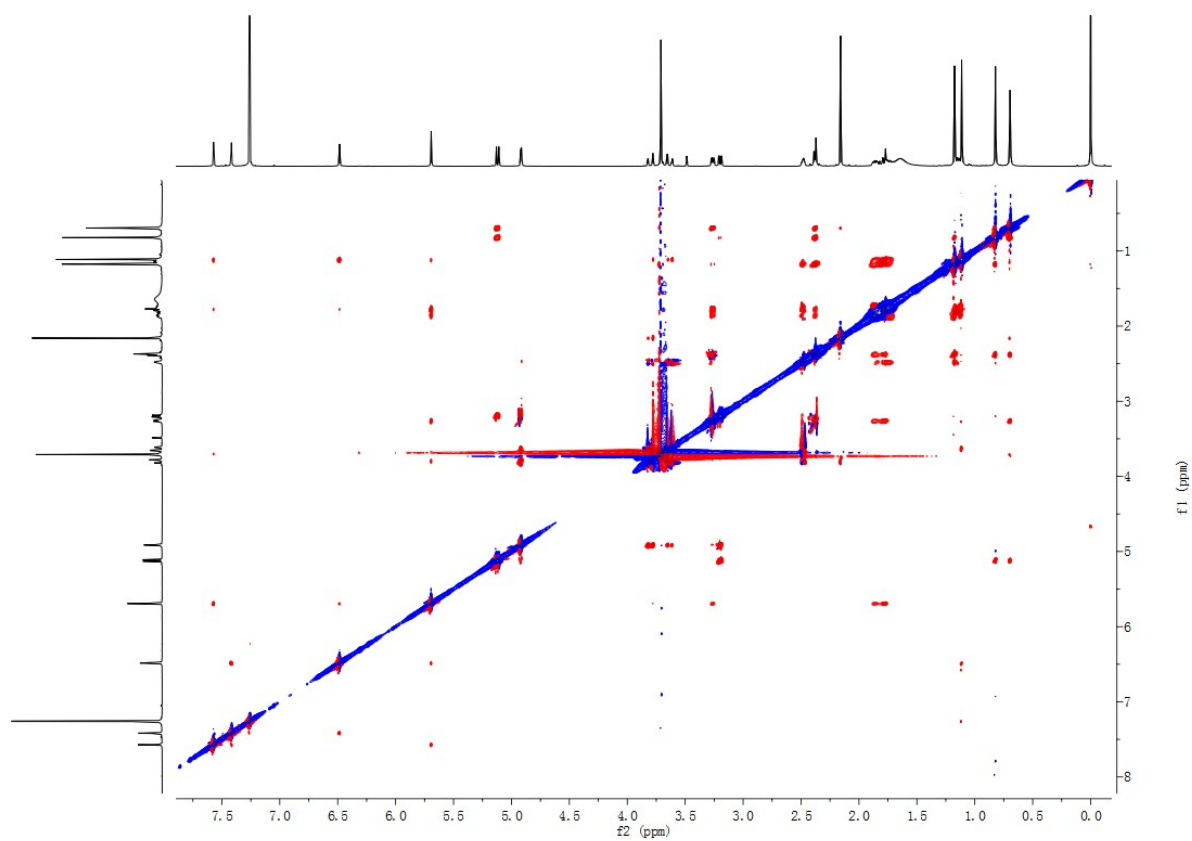
S72 HSQC of Khasenegasin L (12)



S73 HMBC of Khasenegasin L (**12**)

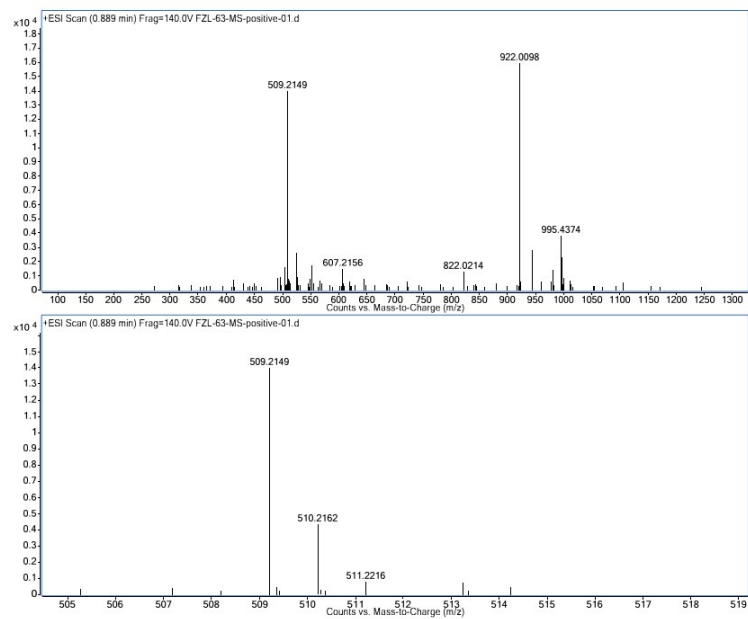


S74 ROSEY of Khasenegasin L (**12**)



Khasenegasin M (13)

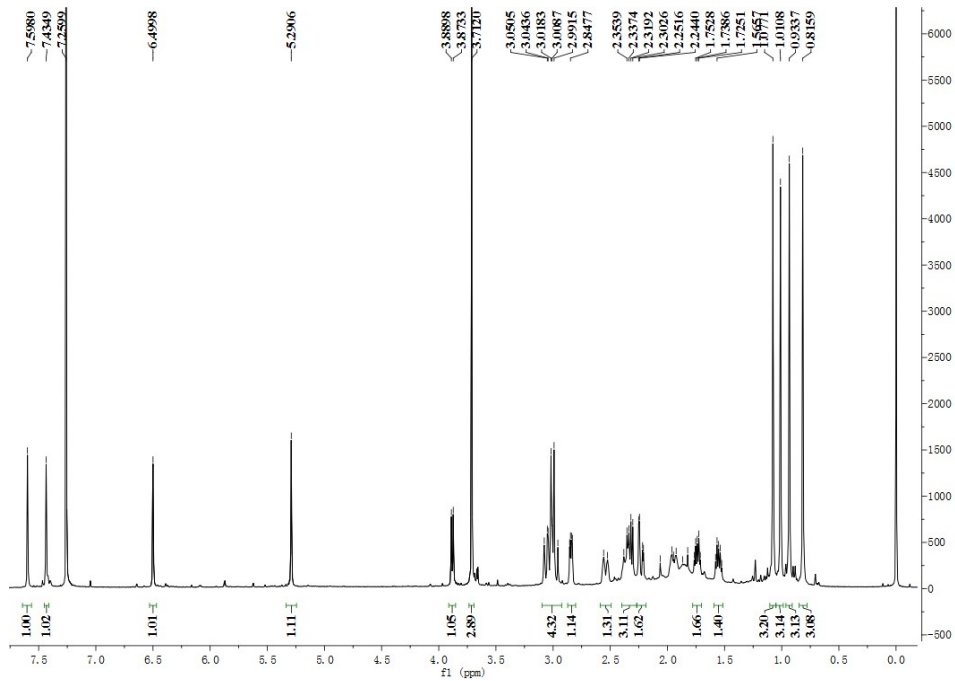
S75 HR-ESI-MS of Khasenegasin M (13)



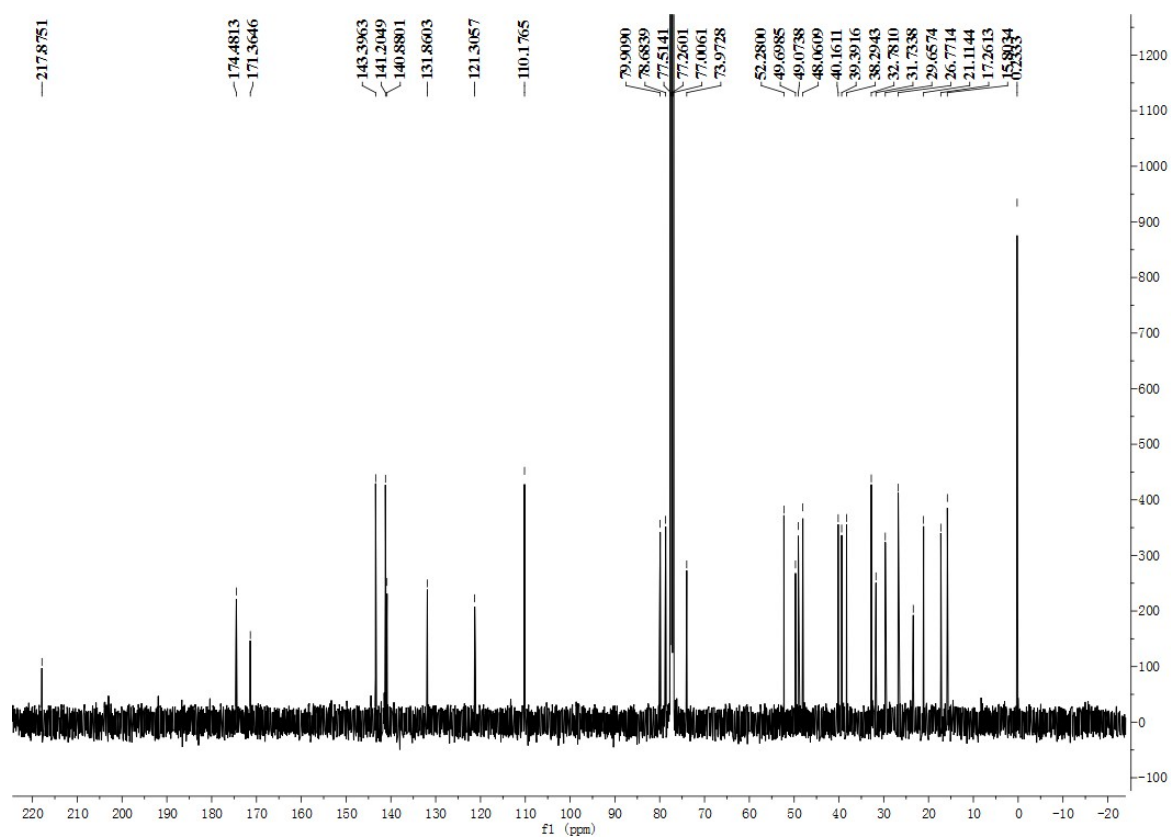
Elemental Composition Calculator

Target m/z:	509.2149	Result type:	Positive ions	Species:	[M+Na] ⁺
Elements:	C (0-80); H (0-120); O (0-30); N(0-10); Na (0-5)				
Ion Formula	Calculated m/z		PPM Error		
C ₂₇ H ₃₄ NaO ₈	509.2146		-0.67		

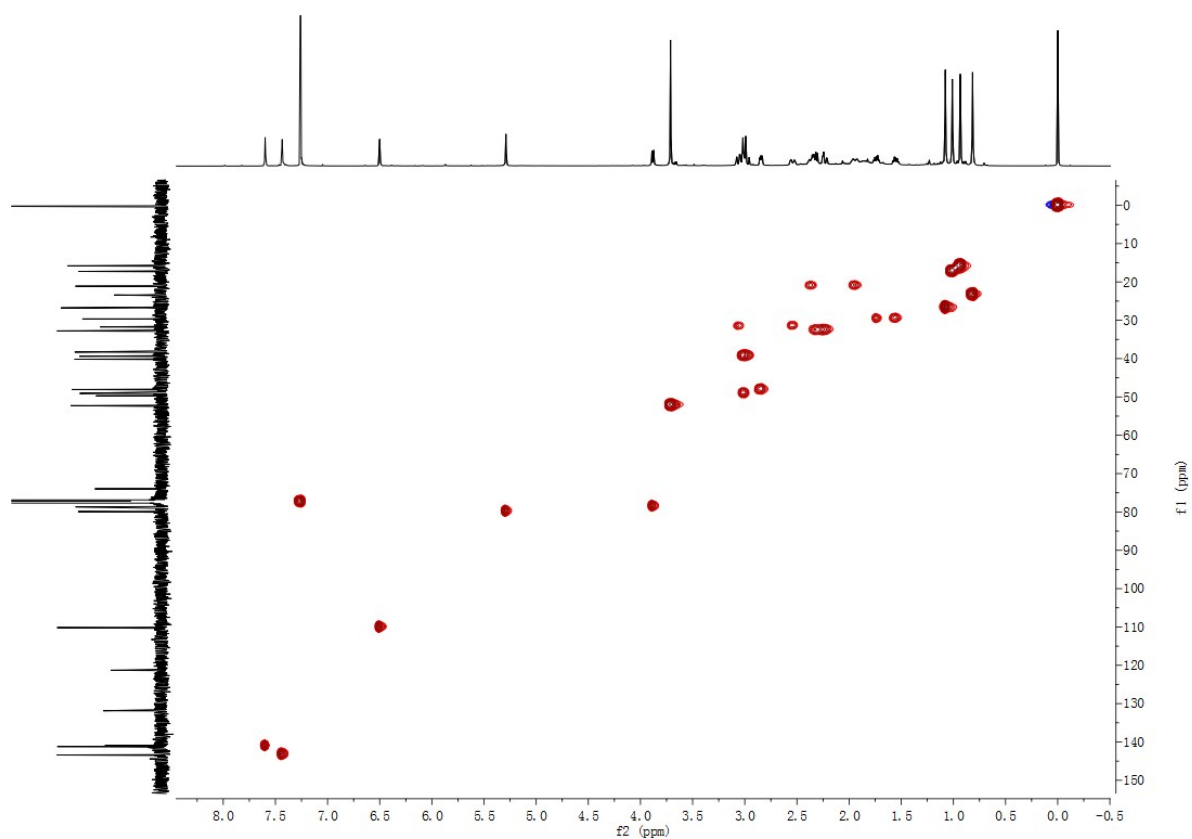
S76 ¹H NMR of Khasenegasin M (13)



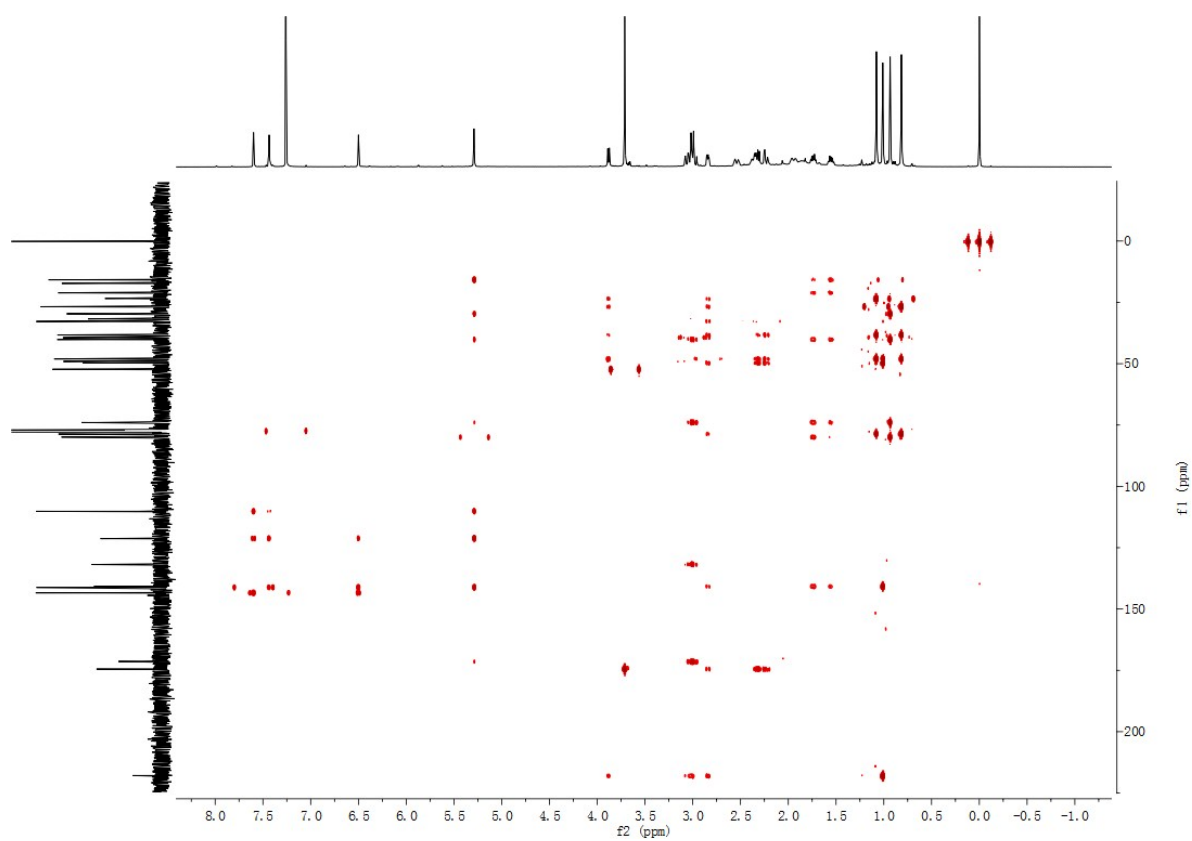
S77 ^{13}C NMR of Khasenegasin M (13)



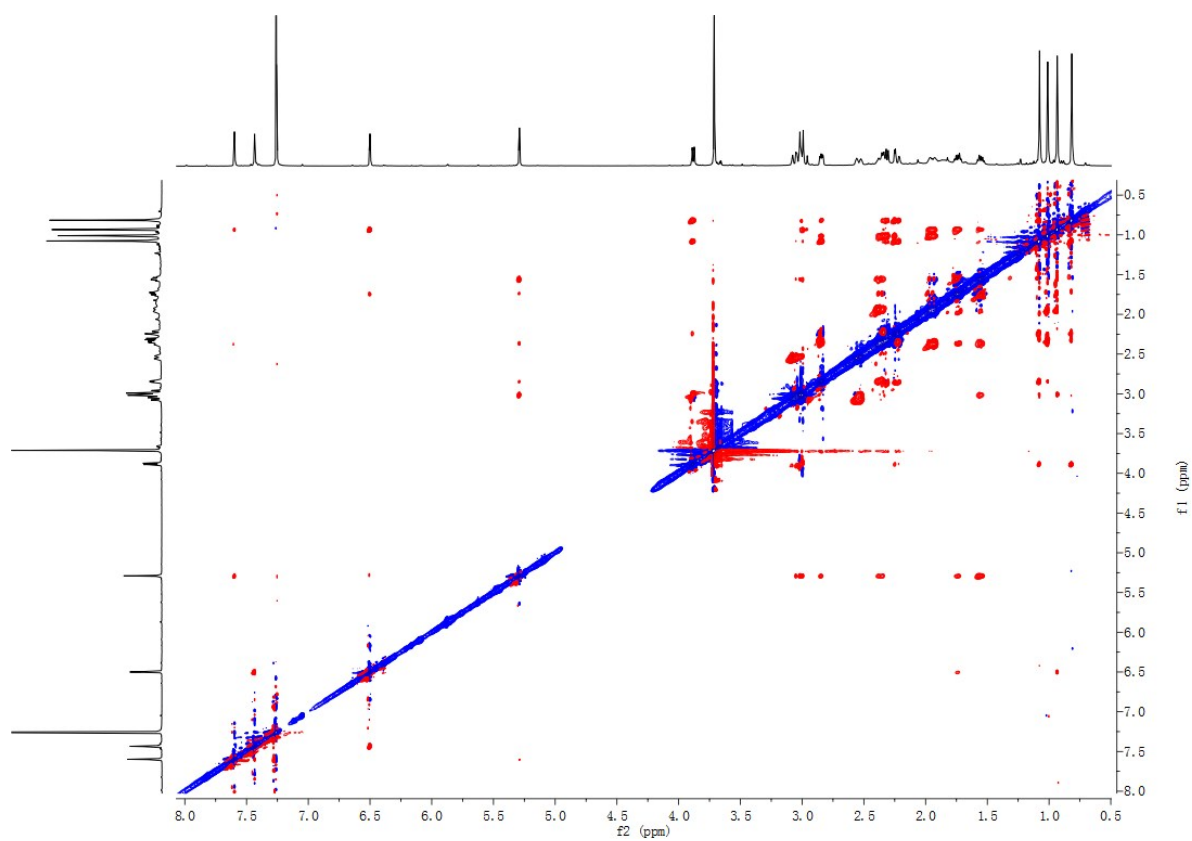
S78 HSQC of Khasenegasin M (13)



S79 HMBC of Khasenegasin M (**13**)

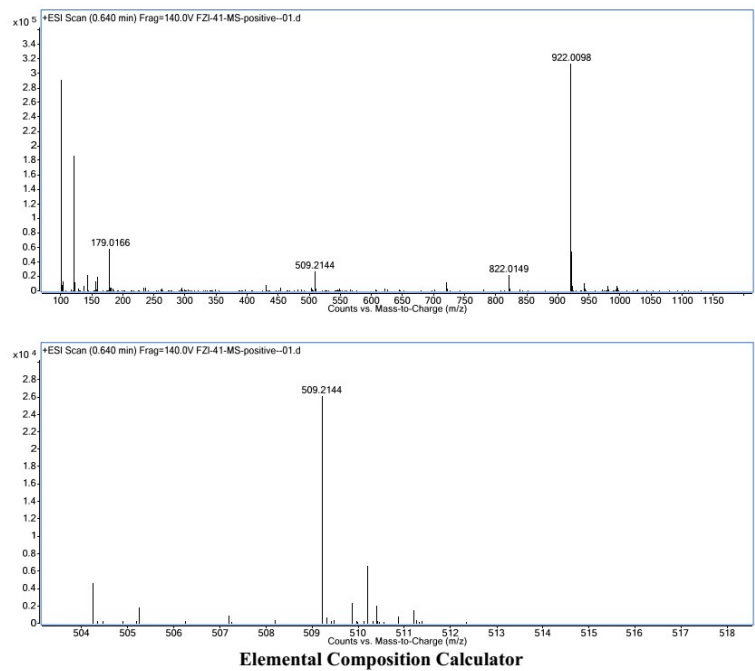


S80 ROSEY of Khasenegasin M (**13**)



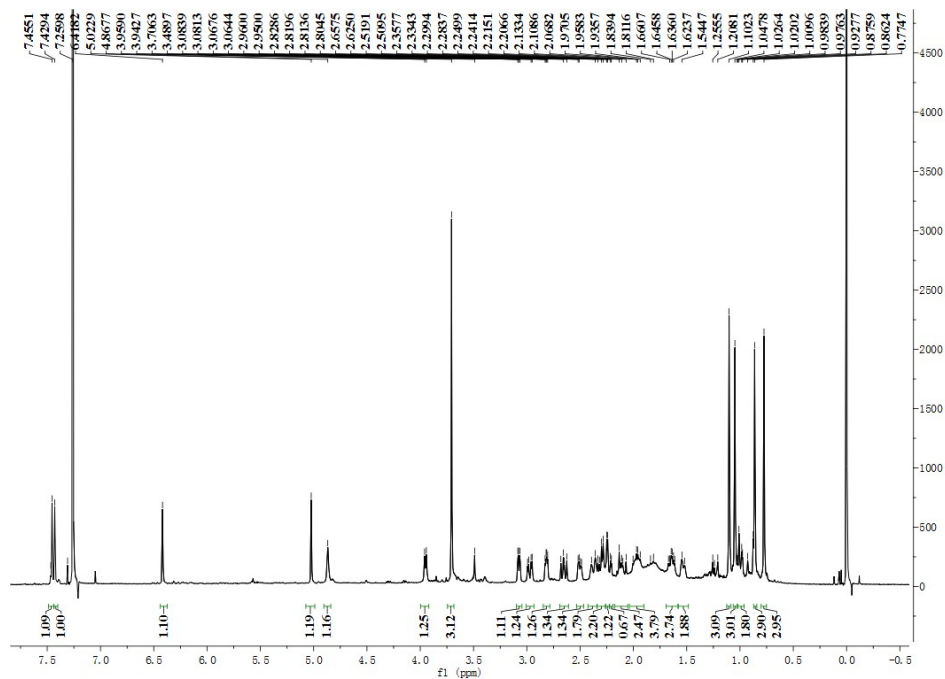
Khasenegasin N (**14**)

S81 HR-ESI-MS of Khasenegasin N (**14**)

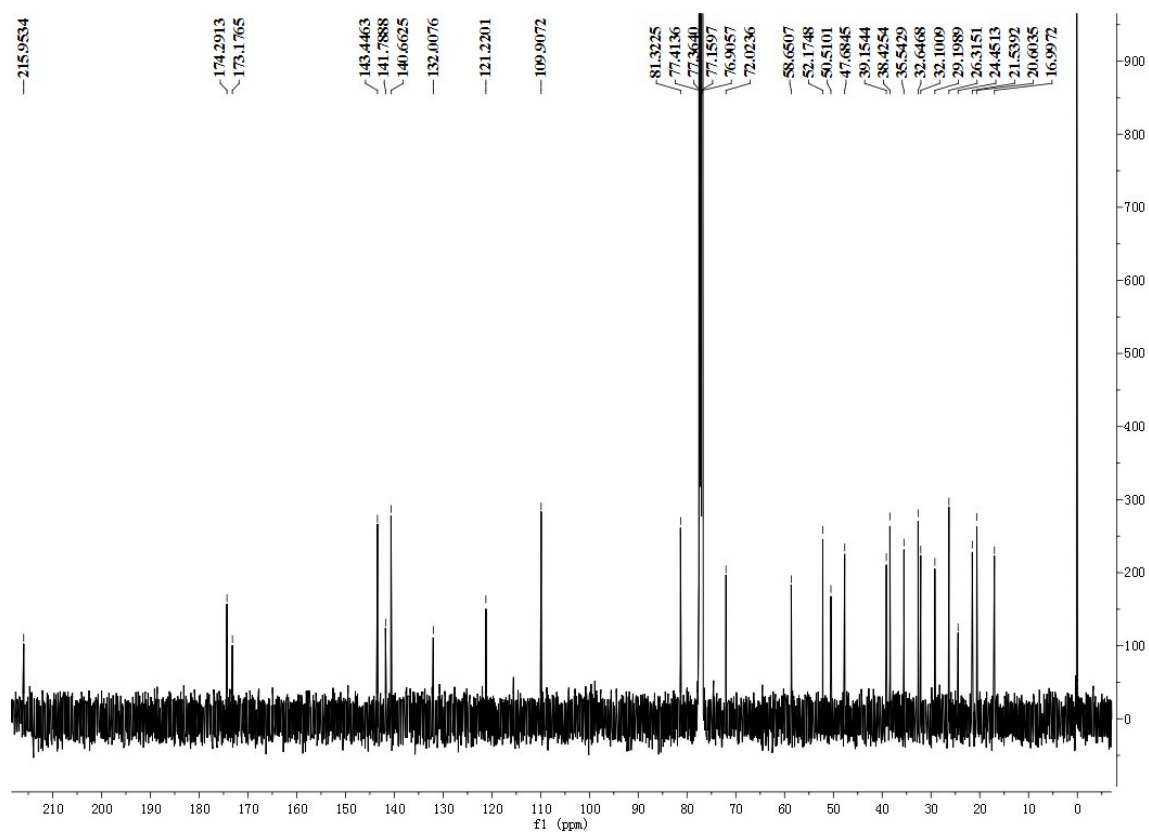


Target m/z:	509.2144	Result type:	Positive ions	Species:	[M+Na] ⁺
Elements:	C (0-80); H (0-120); O (0-30); N(0-10); Na (0-5); Cl (0-5)				
Ion Formula	Calculated m/z		PPM Error		
C27H34NaO8	509.2146		0.39		

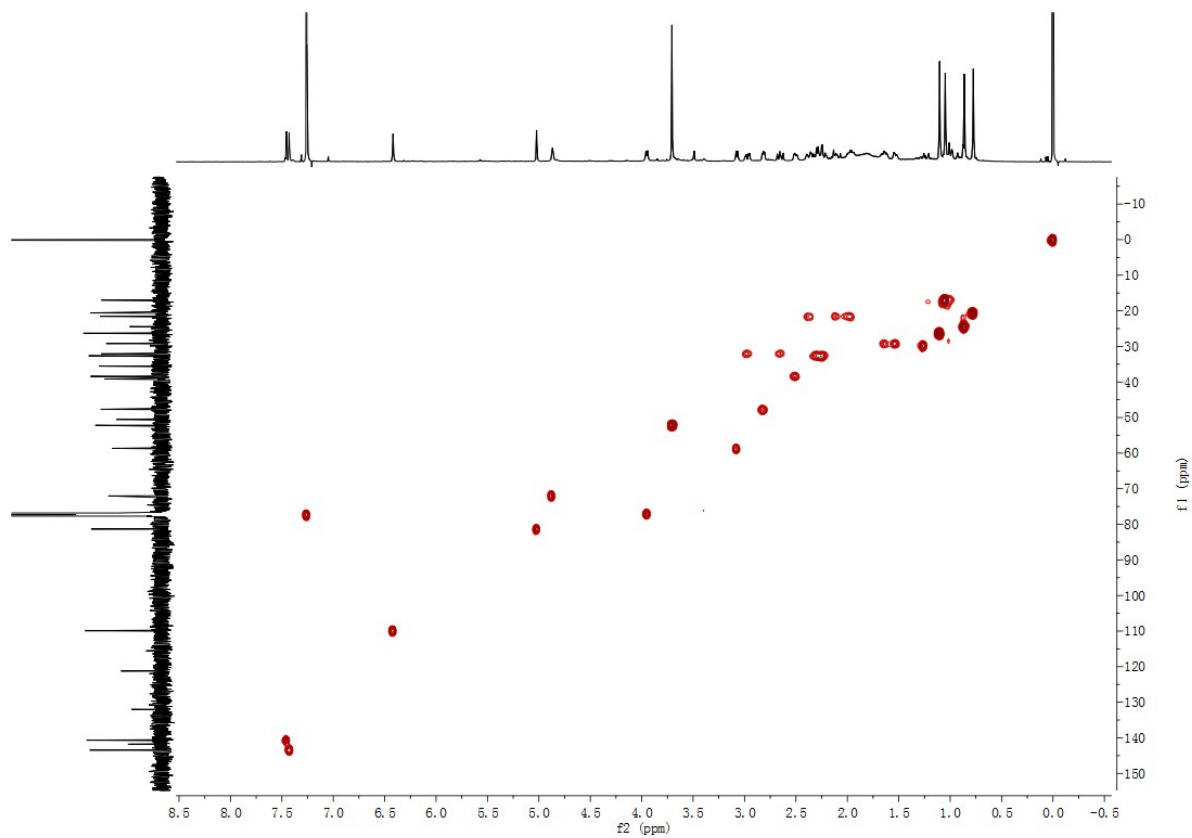
S82 ¹H NMR of Khasenegasin N (**14**)



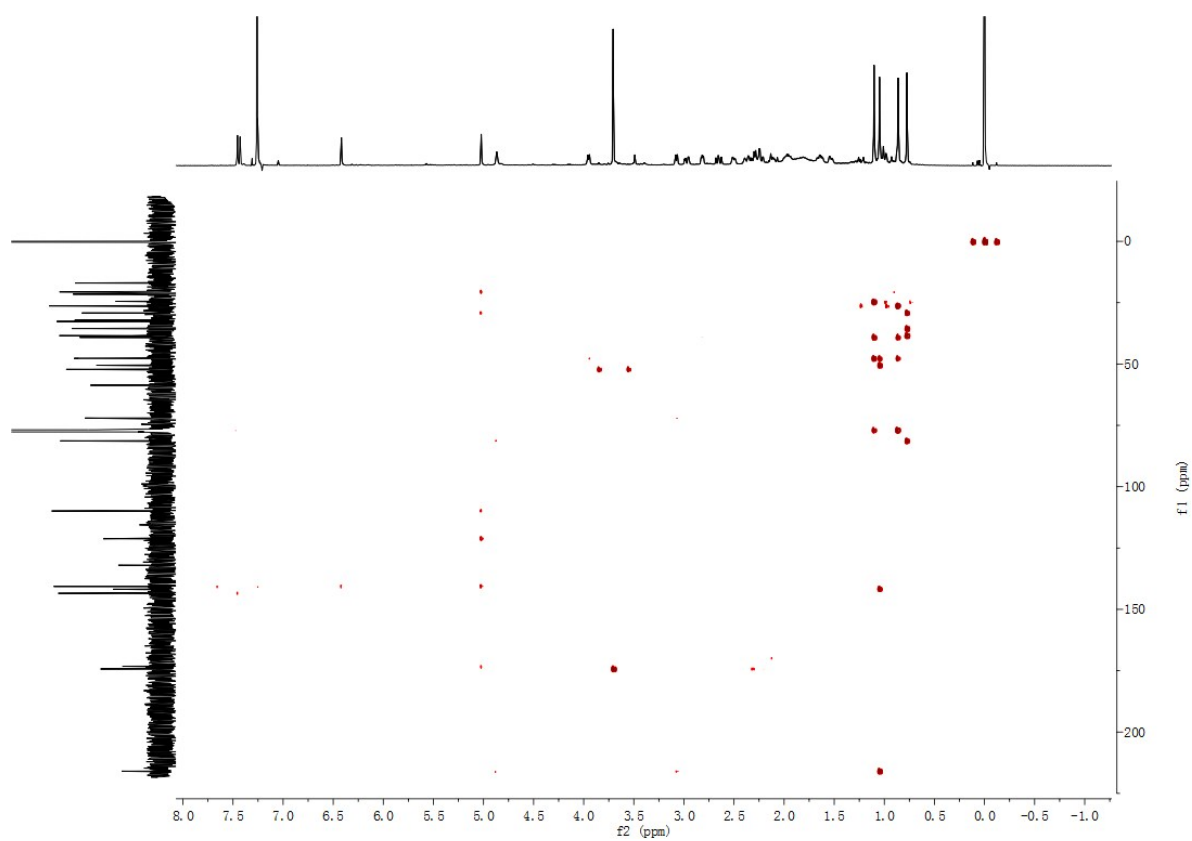
S83 ^{13}C NMR of Khasenegasin N (**14**)



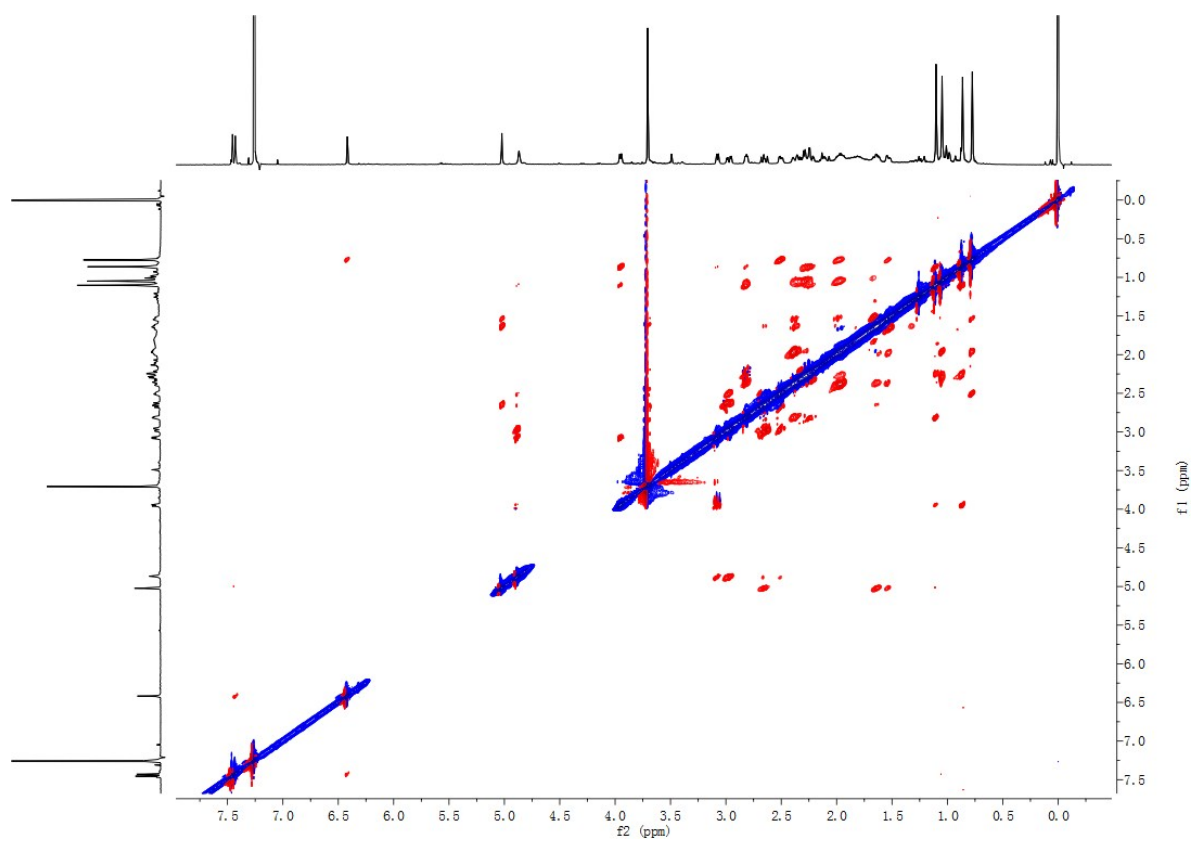
S84 HSQC of Khasenegasin N (**14**)



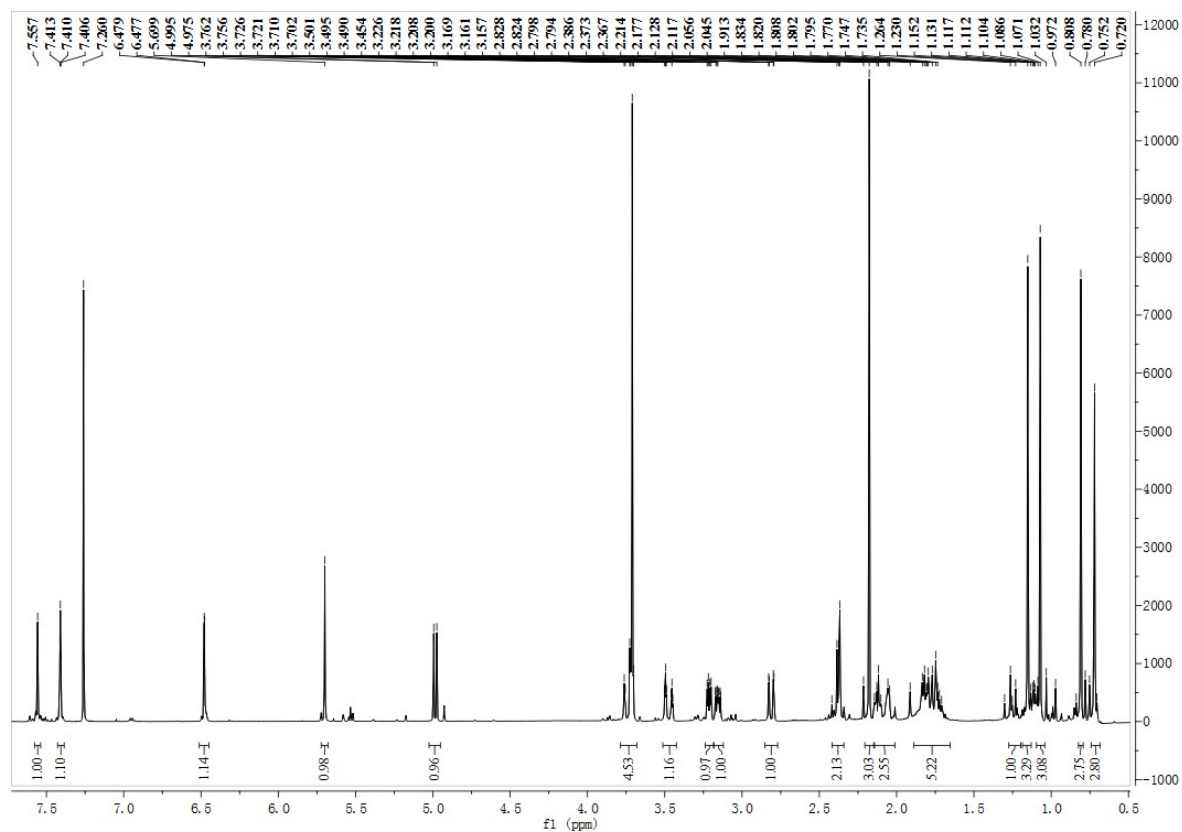
S85 HMBC of Khasenegasin N (**14**)



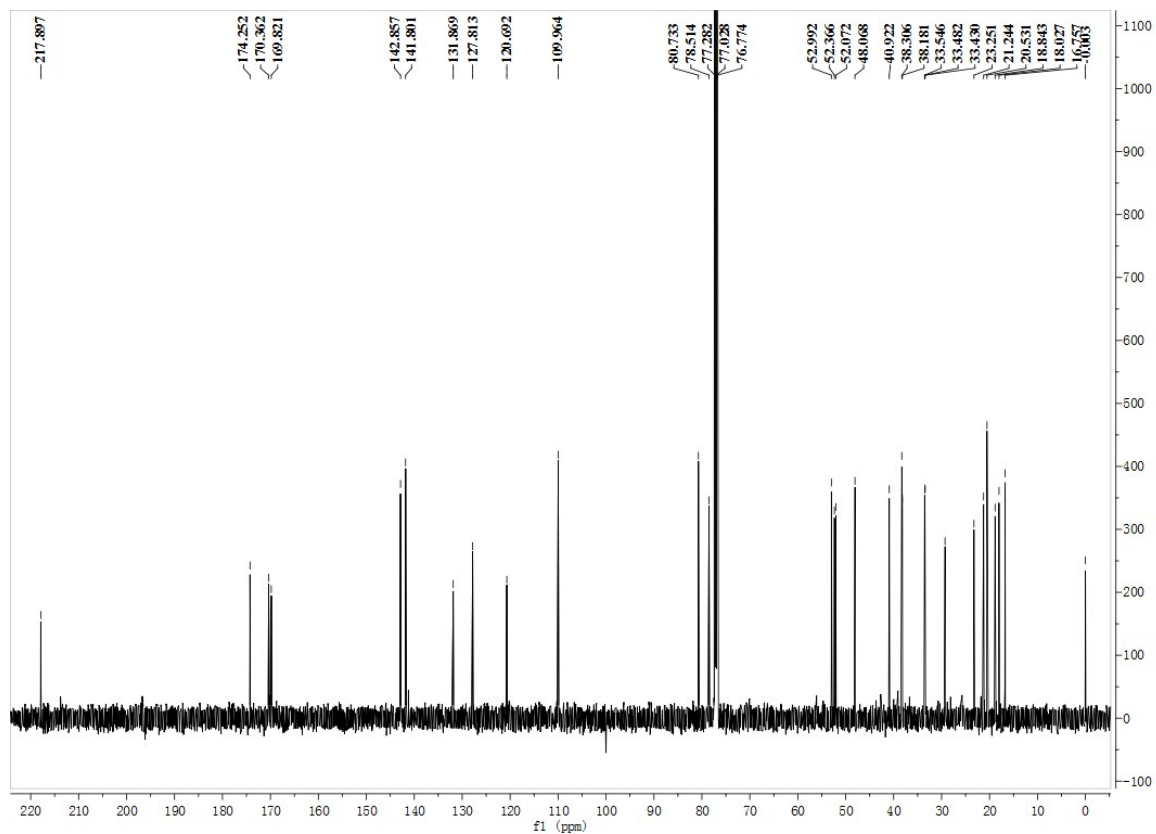
S86 ROSEY of Khasenegasin N (**14**)



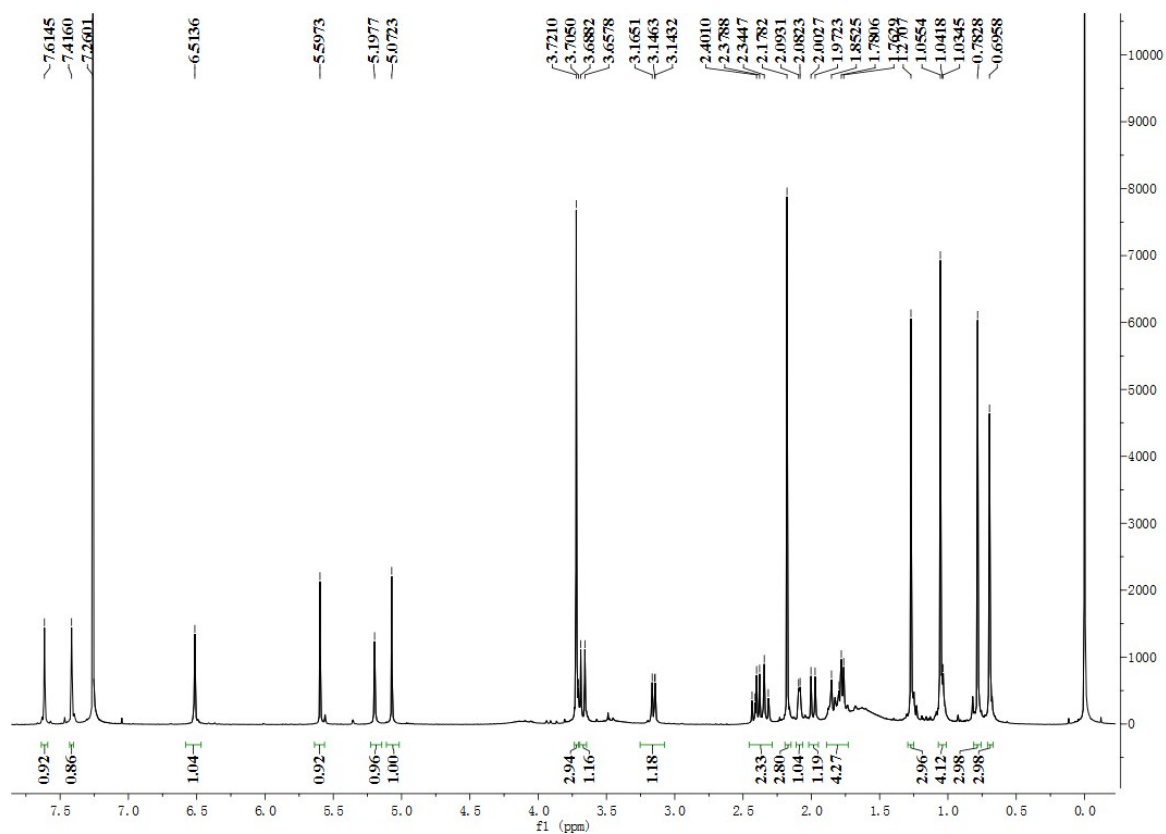
S87: ^1H -NMR (500 MHz, CDCl_3) Spectrum of Compound **15** (fissinolide)



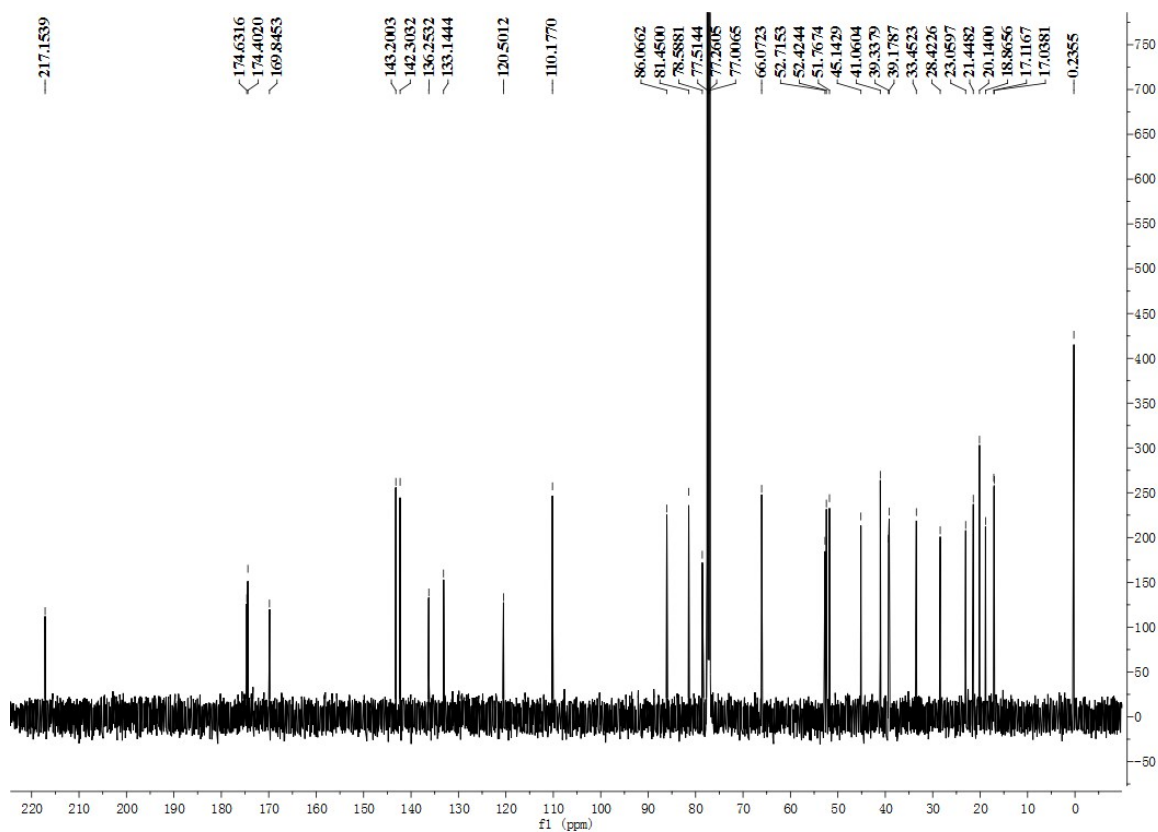
S88: ^{13}C -NMR (125 MHz, CDCl_3) Spectrum of Compound **15** (fissinolide)



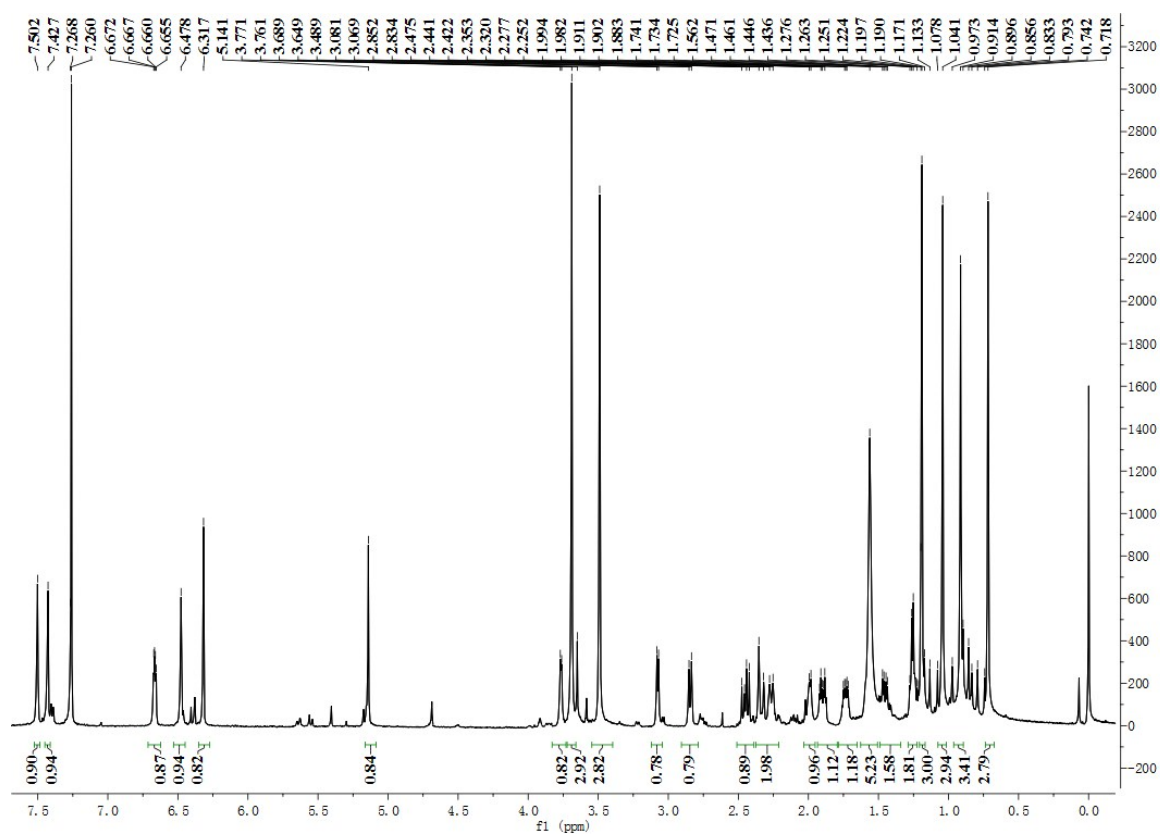
S89: ^1H -NMR (500 MHz, CDCl_3) Spectrum of Compound **16** (swietmanin F)



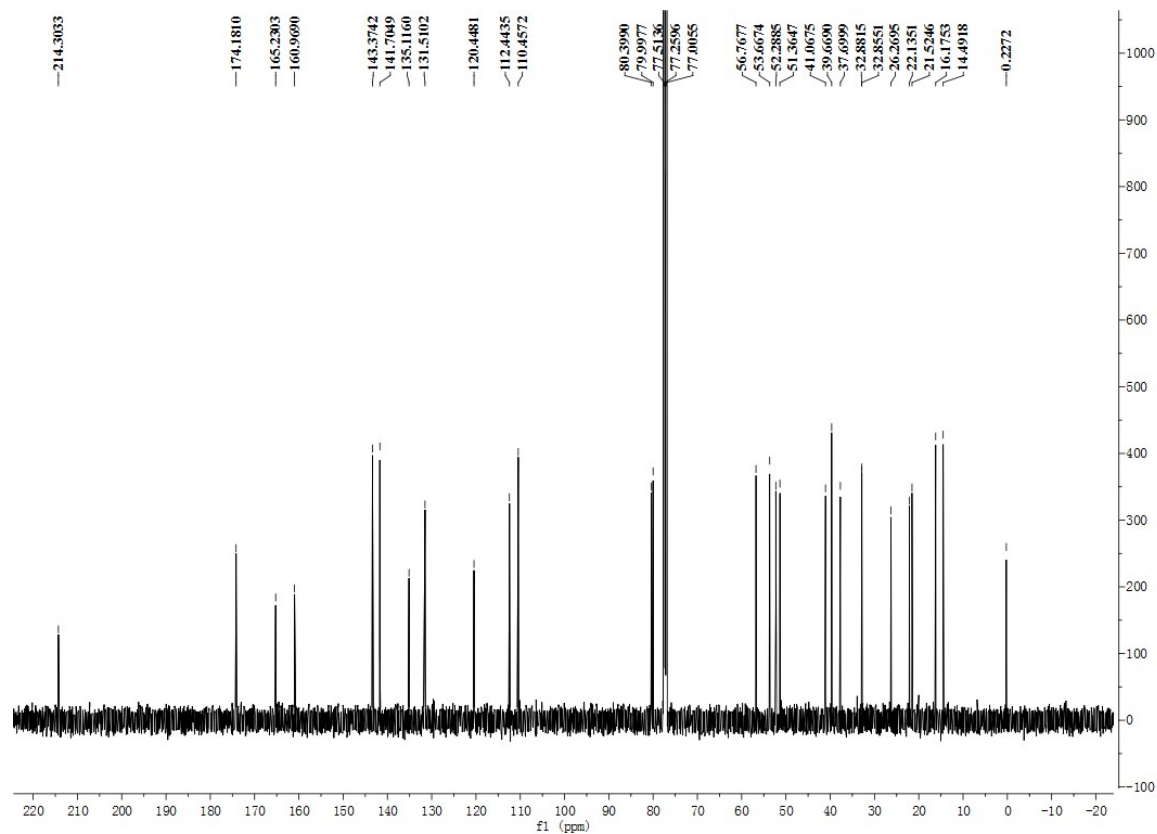
S90: ^{13}C -NMR (125 MHz, CDCl_3) Spectrum of Compound **16** (swietmanin F)



S91: ^1H -NMR (500 MHz, CDCl_3) Spectrum of Compound **17** (seneganolide A)



S92: ^{13}C -NMR (125 MHz, CDCl_3) Spectrum of Compound **17** (seneganolide A)



S93: NMR data comparing compound **15** with published NMR data (fissinolide)

15			fissinolide published NMR data ^a	
Position	δ_{H} (multi, J in Hz) ^b	δ_{C} ^c	δ_{H} (multi, J in Hz) ^d	δ_{C} ^e
1		217.9		218.1
2	3.15, m	48.1	3.15, m	48.0
3	4.98, d (10.0)	78.5	4.95, d (10.5)	78.4
4		38.3		38.3
5	3.21, dd (9.0, 4.0)	40.9	3.22, m	40.8
6	2.40, dd (16.0, 9.0) 2.35, br d (16.0)	33.4	2.38, m	33.4
7		174.3		174.3
8		127.8		127.8
9	1.82, m	52.1	1.8*	52.2
10		52.4		53.0
11 α	1.80, m	18.8	1.7*	18.8
11 β	1.70, m		1.8*	
12 α	1.80, m	29.3	1.0*	29.1
12 β	1.00, m		1.8*	
13		38.2		38.1
14		131.9		131.8
15 α	3.47, dt (21.0, 3.0)	33.5	3.48, dt (20, 2.5)	33.5
15 β	3.75, m		3.80, dm (20)	
16		169.8		170.0
17	5.70, s	80.7	5.70, s	80.7
18	1.07, s, 3H	18.1	1.08, s, 3H	17.9
19	1.15, s, 3H	16.8	1.16, s, 3H	16.8
20		120.7		120.6
21	7.58, s	142.9	7.56, d (1.8)	142.9
22	6.48, d (1.5)	110.0	6.48, d (1.8)	109.9
23	7.41, t (1.5)	141.8	7.42, t (1.8)	141.8
28	0.81, s, 3H	20.5	0.81, s, 3H	20.5
29	0.70, s, 3H	23.3	0.72, s, 3H	23.2
30 α	2.05, m	33.5	2.05, dm (15)	33.4
30 β	2.81, dd (15.0, 2.0)		2.81 dd (15, 2.5)	
7-OCH ₃	3.72, s, 3H	53.0	3.85, s, 3H	53.3
3-OAc	2.18, s, 3H	170.4	2.18, s, 3H	170.4
		21.2		21.3

^a S. A. Khalid, G. M. Friedrichsen, A. Kharazmi, T. G. Theander, C. E. Olsen and S. B. Christensen, *Phytochemistry*, 1998, **49**, 1769-1772 ; ^b measured in CDCl₃ at 500 MHz; ^c measured in CDCl₃ at 125 MHz; ^d measured in CDCl₃ at 300 MHz, * Severe overlapping of signals prevents a detailed interpretation of the coupling pattern. n.o. Not observed; ^e measured in CDCl₃ at 75 MHz.

S94: NMR data comparing compound **16** with published NMR data (swietmanin F)

16			swietmanin F published NMR data ^a	
Position	δ_{H} (multi, J in Hz) ^b	δ_{C} ^c	δ_{H} (multi, J in Hz) ^d	δ_{C} ^e
1		216.9		216.9
2		78.6		78.3
3	5.07, s	86.1	5.07, s	85.7
4		39.3		39.0
5	3.16, dd (11.0, 1.5)	41.1	3.15, dd (11.1, 1.9)	40.7
6	2.40, dd (16.5, 11.0)	33.5	2.41, dd (16.5, 11.1)	33.1
	2.33, dd (16.5, 1.5)		2.33, dd (16.5, 1.9)	
7		174.4		174.1
8		133.1		132.8
9	2.09, br d (5.4)	51.8	2.08, br d (4.5)	51.4
10		52.7		52.4
11 α	1.86, m	18.9	1.88, m	18.5
11 β	1.80, m		1.80, m	
12 α	1.12, m	28.4	1.12, m	28.0
12 β	1.68, m		1.78, m	
13		39.2		38.9
14		136.3		135.9
15	5.20, s	66.1	5.02, s	65.7
16		174.6		174.4
17	5.60, s	81.5	5.60, s	81.1
18	1.06, s, 3H	17.1	1.06, s, 3H	16.9
19	1.27, s, 3H	17.0	1.27, s, 3H	16.7
20		120.5		120.1
21	7.61, s	142.3	7.62, br d (0.6)	142.0
22	6.51, br s	110.2	6.51, br t (0.9)	109.9
23	7.42, br s	143.2	7.42, br t (1.6)	142.9
28	0.78, s, 3H	23.1	0.78, s, 3H	22.7
29	0.70, s, 3H	20.1	0.69, s, 3H	19.8
30	3.66, d (15.2)	45.1	3.67, d (15.2)	44.8
	1.99, d (15.2)		1.99 d (15.2)	
7-OCH ₃	3.72, s, 3H	52.4	3.72, s, 3H	52.2
3-OAc	2.18, s, 3H	169.8	2.18, s, 3H	169.6
		21.4		21.2

^aB. D. Lin, T. Yuan, C. R. Zhang, L. Dong, B. Zhang, Y. Wu and J. M. Yue, *J. Nat. Prod.*, 2009, **72**, 2084–2090.^b measured in CDCl₃ at 500 MHz; ^c measured in CDCl₃ at 125 MHz; ^d measured in CDCl₃ at 400 MHz; ^e measured in CDCl₃ at 125 MHz.

S95: NMR data comparing compound **17** with published NMR data (seneganolide A)

17			seneganolide A published NMR data ^a	
Position	δ_{H} (multi, J in Hz) ^b	δ_{C} ^c	δ_{H} (multi, J in Hz) ^d	δ_{C} ^e
1		214.3		213.9
2	3.07, d (6.0)	56.8	3.07, d (5.9)	56.5
3	3.77, d (5.0)	80.4	3.77, d (5.2)	80.2
4		39.7		39.5
5	2.84, br d (9.0)	41.1	2.85, dd (9.7, 1.5)	40.9
6	2.44, dd (17.0, 9.0)	32.9	2.46, dd (16.8, 9.7)	32.7
	2.33, br d (17.0)		2.35, dd (17.2, 1.6)	
7		174.2		173.9
8		135.1		134.9
9	2.26, br d (12.5)	53.7	2.27, br dt (12.2, 2.8)	53.6
10		51.4		51.2
11 α	1.73, ddd (12.5, 8.0, 4.5)	21.5	1.73, ddd (13.2, 9.1, 4.3)	21.3
11 β	1.45 ddd (14.0, 12.5, 5.0)		1.47, ddd (14.5, 12.1, 5.2)	
12 α	1.26, m	32.9	1.26, ddd (14.1, 11.0, 5.3)	32.7
12 β	1.90, dt (14.0, 4.5)		1.90, dt (14.1, 4.5)	
13		37.7		37.5
14		161.0		160.7
15	6.32, s	112.4	6.31, s	112.3
16		165.2		164.9
17	5.14, s	80.0	5.14, s	79.8
18	1.04, s, 3H	22.1	1.05, s, 3H	21.9
19	1.20, s, 3H	16.2	1.20, s, 3H	15.9
20		120.4		120.3
21	7.43, br s	141.7	7.50, d (1.5)	141.5
22	6.48, br s	110.5	6.48 br d (1.0)	110.2
23	7.50, br s	143.4	7.42, t (1.6)	143.2
28	0.91, s, 3H	26.3	0.92, s, 3H	26.1
29	0.72, s, 3H	14.5	0.72, s, 3H	14.2
30	6.66, dd (6.0, 2.5)	131.5	6.67, dd (6.0, 2.7)	131.2
7-OCH ₃	3.69, s, 3H	52.3	3.69, s, 3H	52.0

^a S. A. M. Abdelgaleil, T. Iwagawa, M. Doe and M. Nakatani, *Fitoterapia*, 2004, **75**, 566-572. ^b measured in CDCl₃ at 500 MHz; ^c measured in CDCl₃ at 125 MHz; ^d measured in CDCl₃ at 600 MHz; ^e measured in CDCl₃ at 150 MHz.