Seminar (2019 April)

CC: Question 1

Sample 20 g

A

Si gel 100 g

5 times

B

Si gel 1000 g

50 times

1. Which one you prefer? Why?
2. What eluant you will choose?

Make your choose and explain your choose with your experience or your theory.

Question 2

Compound Aa-12 (**1**) was obtained as white amorphous powder from fresh bark of *Ailanthus altissima* by Tian-Hui. The molecular formula of **1** was established as $C_{30}H_{48}O_6$ at *m/z* 527.3349 $[M+Na]^+$ (Calcd 527.3348) in its HR-ESI-MS.

The ^1H- and ^{13}C NMR spectrum was attached (Table 1) and DEPT of **1** displayed signals attributed to 30 C atoms.

According to these information to deduce a reasonable structure for **1** and give your evident to support your proposed structure which must compatible with all the information.

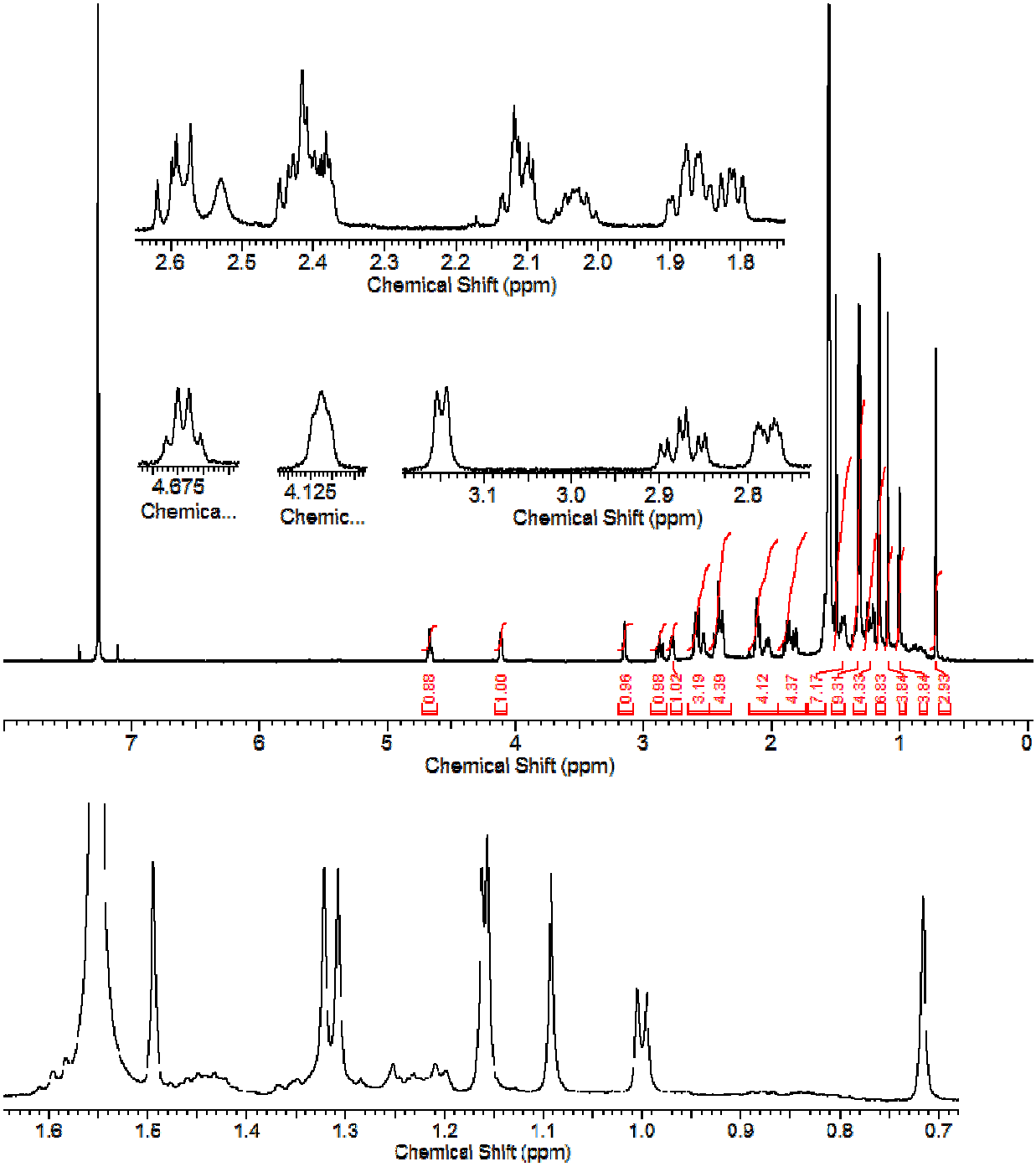
The structure depicted in Figure **1**. is Right? and Why?

And how to prepare a manuscript with these data?

A friendly reminder:

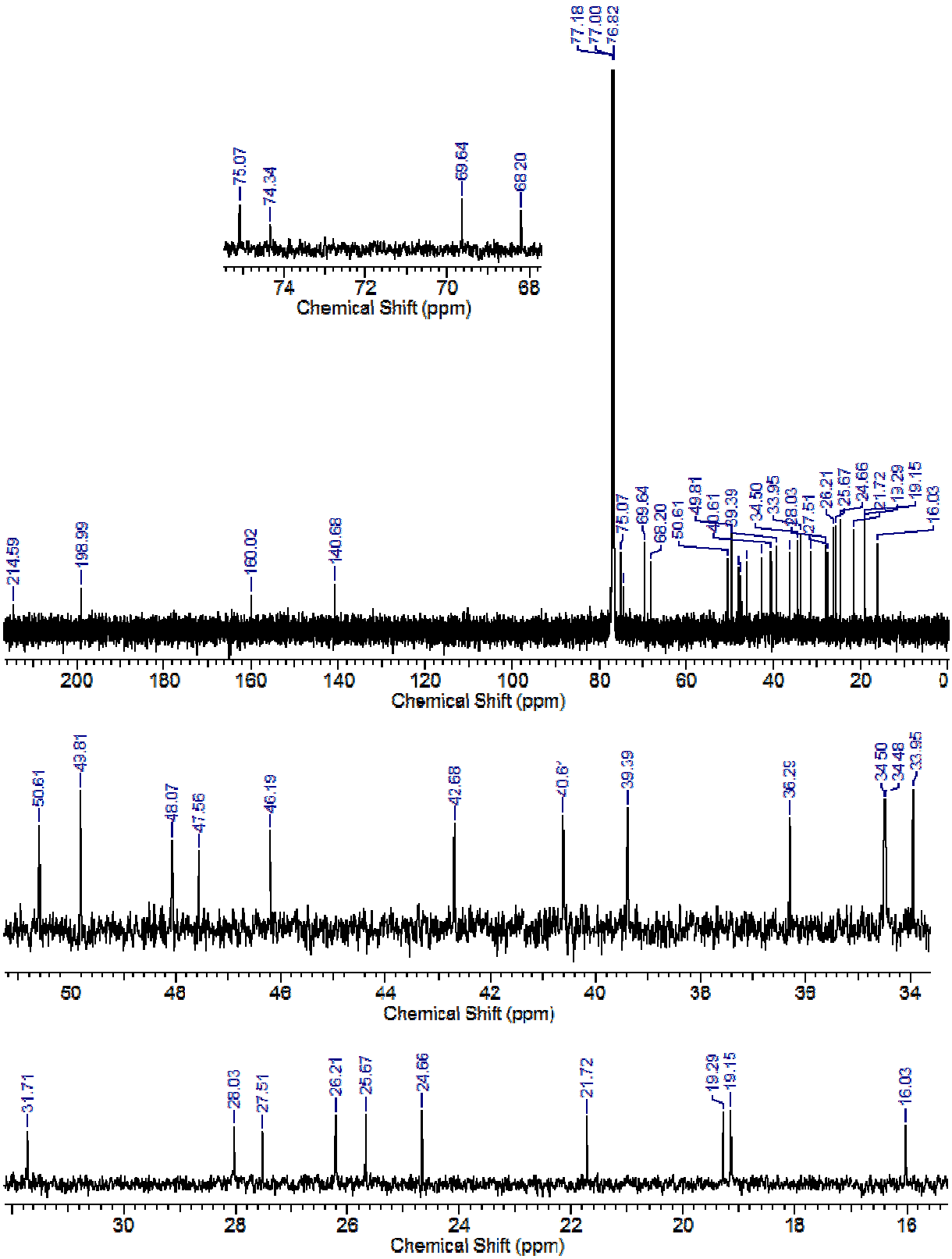
The ^1H- and ^{13}C NMR spectroscopic pattern of **1** accommodated the same characteristics as that of attached reference [*Chemistry & Biodiiversity*, **2013**, *10*: 695-702.].

H-NMR

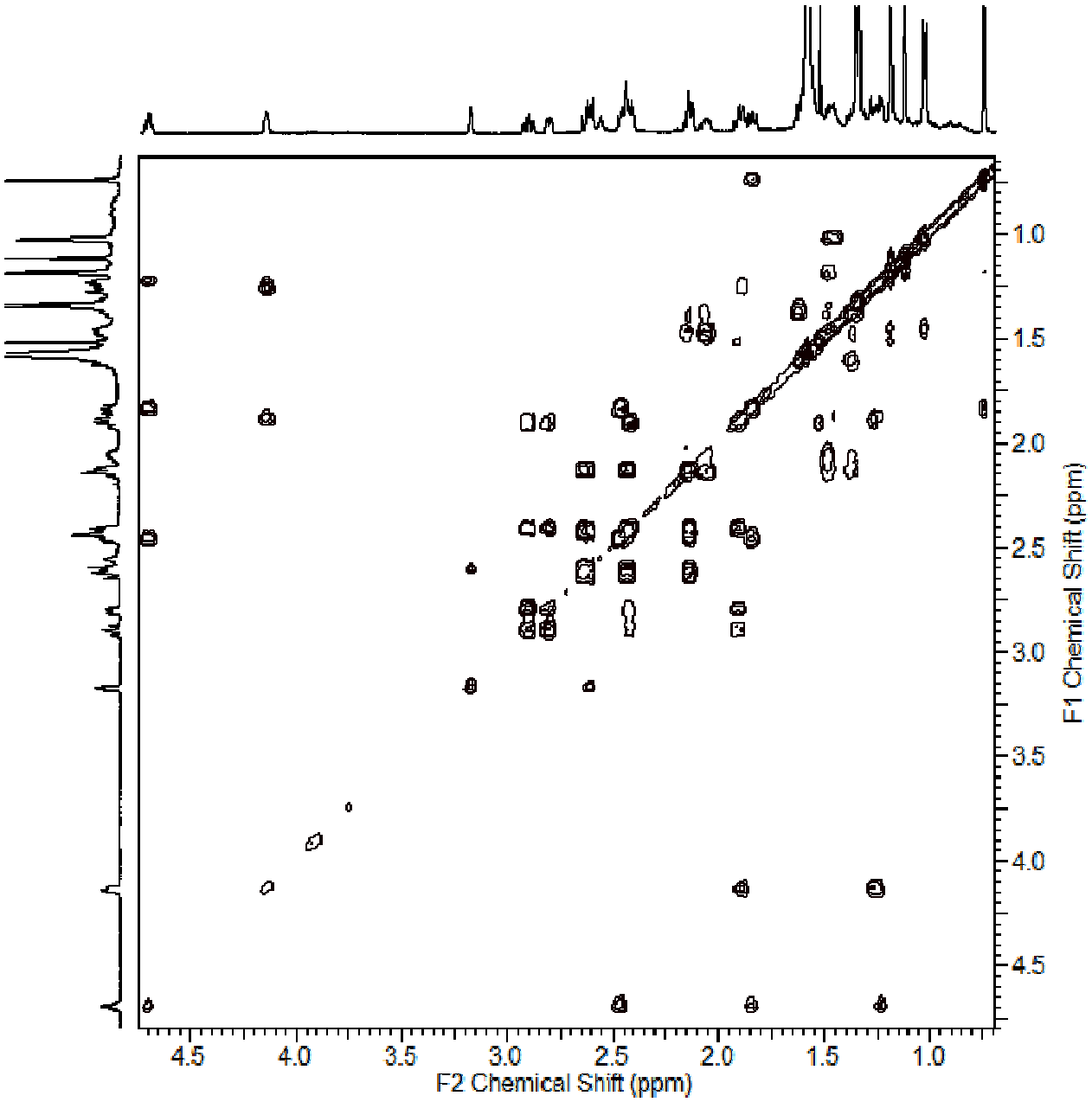

2.6 2.5 2.4 2.3 2.2 2.1 2.0 1.9 1.8
Chemical Shift (ppm)
4.675
Chemica...
4.125
Chemic...
3.1 3.0 2.9 2.8
Chemical Shift (ppm)
0.88 1.00 0.96 0.98 1.02 3.19 4.39 4.12 4.37 7.17 9.31 4.33 6.83 3.84 3.84 2.93
7 6 5 4 3 2 1 0
Chemical Shift (ppm)

1.6 1.5 1.4 1.3 1.2 1.1 1.0 0.9 0.8 0.7
Chemical Shift (ppm)

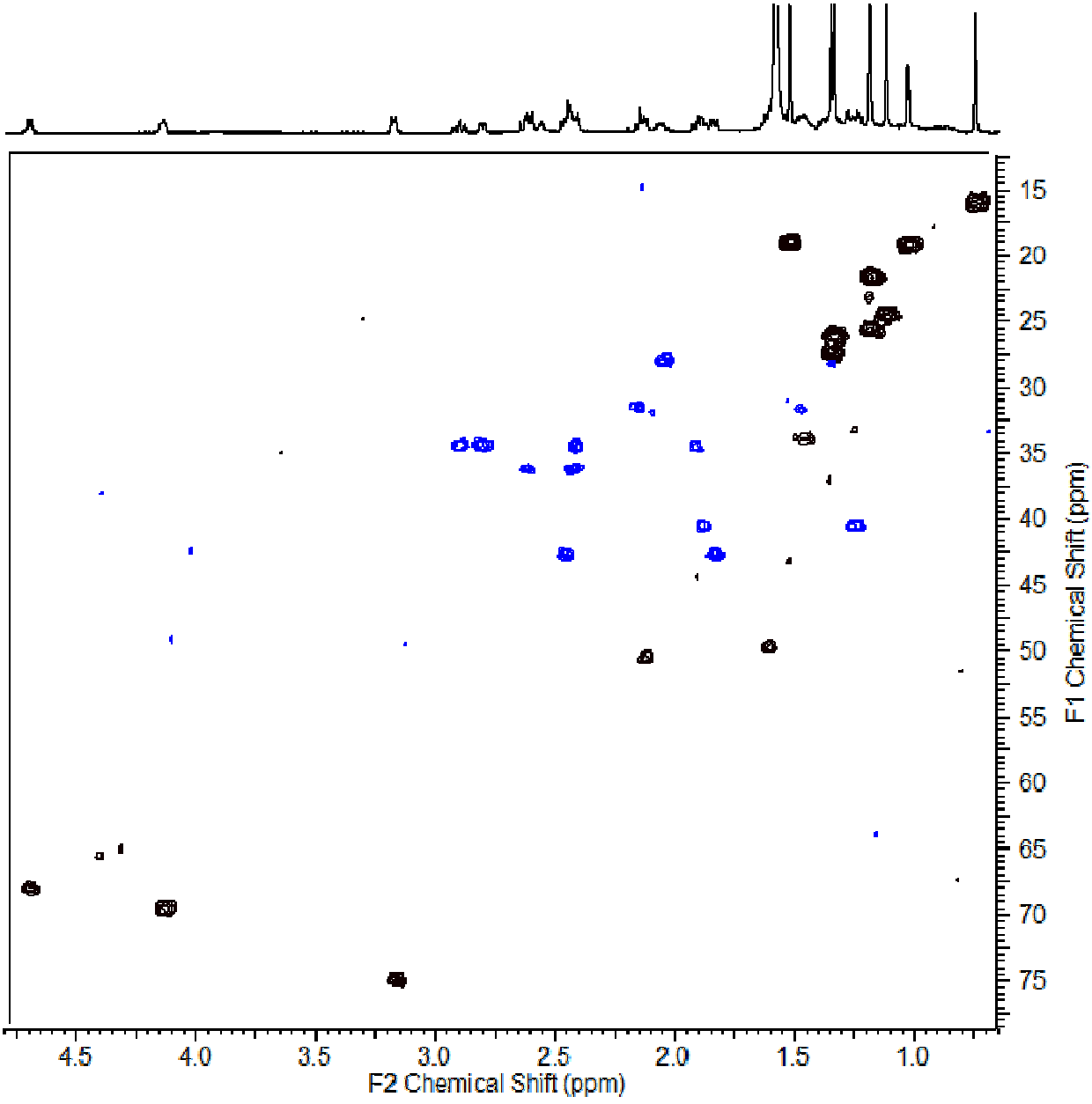
C13-NMR (ns 28,208)



COSY

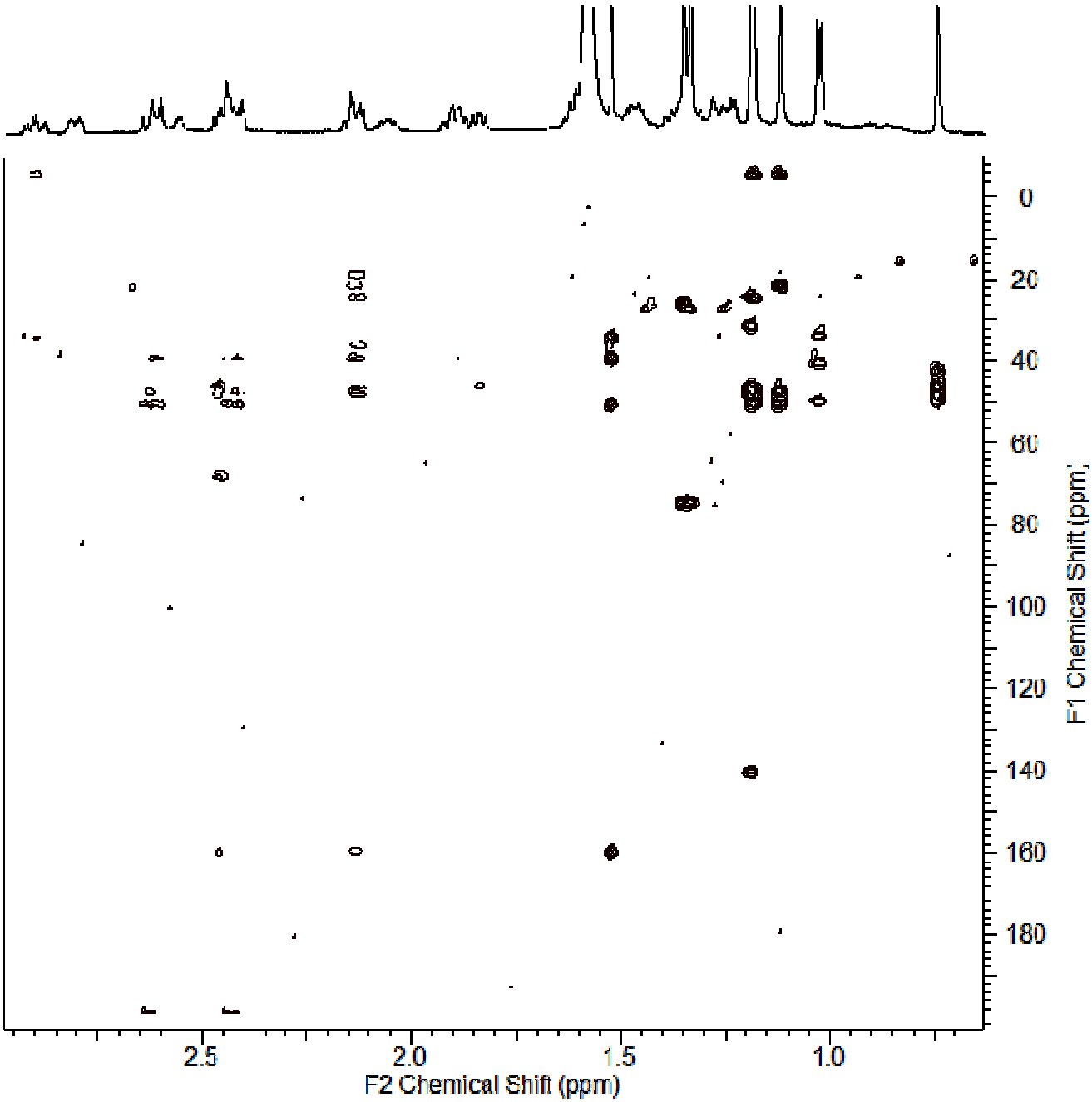

4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
F2 Chemical Shift (ppm)
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
F1 Chemical Shift (ppm)

HSQC-DEPT


15
20
25
30
35
40
45
50
55
60
65
70
75
F1 Chemical Shift (ppm)
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
F2 Chemical Shift (ppm)

HMBC

Ketone C3 fold over.



NOESY

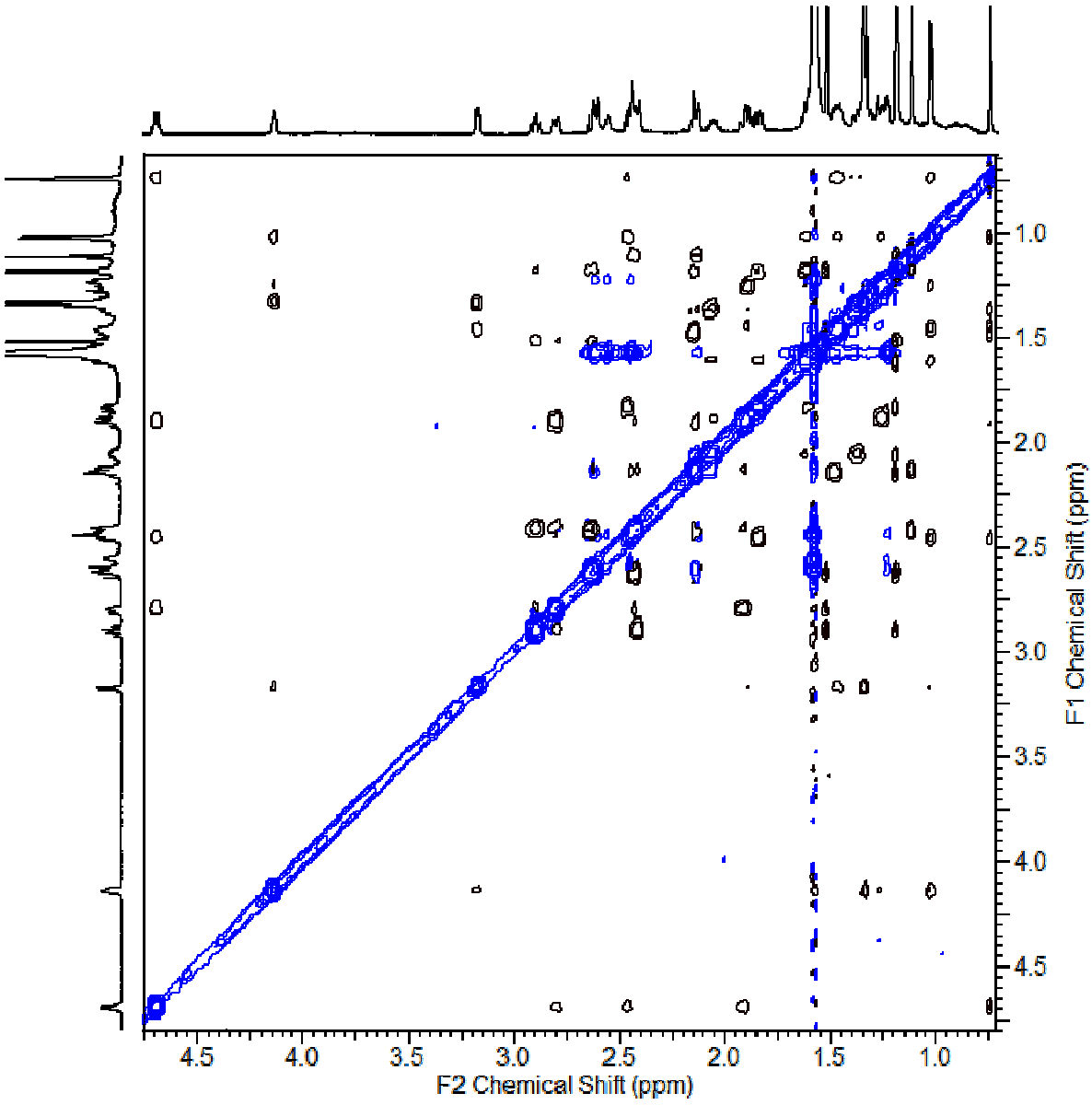

4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
F2 Chemical Shift (ppm)
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
F1 Chemical Shift (ppm)

Table 1 NMR spectroscopic data of **1**in CDCl3, 700 MHz for ^{1}H and 175 MHz for ^{13}C.

Position	δ_H (mult[a])	J [Hz]	δ_C	HMBC	NOESY[b]
1e	2.78 (ddd)	12.8, 5.1, 2.7	34.5	19	$1a^{s}$, 11^{m},19^{m}
1a	1.88 (o)				
2a	2.88 (td)	14.9, 5.6	34.48		$2e^{s}$, $1e^{m}$, 19^{m}, 29^{m}, $1a^{m}$
2e	2.40 (o)				
3	--		214.58	28, 29	
4	--		47.56	28, 29	
5	2.11 (o)		50.62	19, 28, 29	
6a	2.60 (dd)	18.5, 13.9	36.29		$6e^{s}$, 19^{s}, 29^{s}
6e	2.42 (o)				
7	--		198.98		
8	--		140.67	30	
9	--		160.01	19	
10	--		39.39	19	
11	4.67 (q) 1.20 (d)	7.8 7.6	68.2 --		18^{s}, $12a^{m}$, $1e^{m}$, $1a/12^{w}$
12a	2.45 (m)		42.69	18	11^{m}, 21^{s}
12b	1.81 (dd)	12.8, 8.4			
13	--		48.07 or 46.20	18, 30	
14	--		48.07 or 46.20	18, 30	
15a	2.13 (m)		31.71	30	
15b	1.46 (o)				
16a	2.01 (m)		28.03		
16b	1.35 (m)				
17	1.59 (o)		49.81	18, 21	30^{s}, 21^{w}
18	0.72 (s)		16.04	12, 13, 14, 17	11^{s}, 20^{s}, 21^{s}

19	1.50 (s)		19.16	1, 5, 9, 10	$1e^{m}$, $2a^{m}$, $6a^{m}$, 29^{s}
20	1.44 (m)		33.96	21	18^{s}, 21^{w}, 24^{w}
21	1.00 (d)	6.4	19.29	17, 20, 22	$12a^{s}$, 17^{w}, 18^{s}, 20^{w}, 23^{s}
22a	1.87 (m)		40.61	21	
22b	1.22 (m)				
23	4.11 (br.t)	5.7	69.64		27^{s}, 21^{s}
24	3.15 (d)	7.5	75.01	26, 27	26^{s}, 27^{s}, 20^{w}
25	--		74.33	26, 27	
26	1.33 (s)		27.51	24, 25, Me-27	24^{s}
27	1.31 (s)		26.21	24, 25, Me-26	23^{s}, 24^{s}
28	1.09 (s)		24.67	3, 4, 5, Me-29	
29	1.157 (s)		21.72	3, 4, 5, Me-28	$6a^{m}$, 19^{s}
30	1.162 (s)		25.68	8, 13, 14, 15	17^{s}

a Multiplicity: s, singlet; d, doublet; dd, doublet of doublets; m, multiplet; o, overlapped.

b NOESY intensities are marked as strong (s), medium (m), or weak (w).

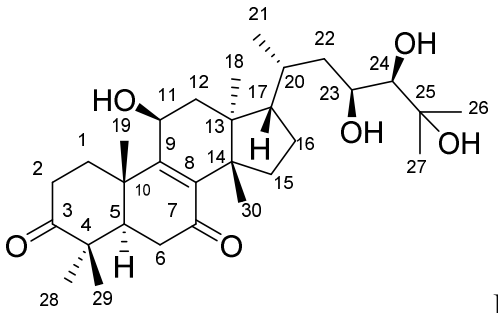


Fig. 1