

Supplementary Materials

Elucidation of the relationship between CD Cotton effects and the absolute configuration of sixteen stereoisomers of spiroheterocyclic-lactams

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Table S1 NMR spectral data of **1** in CDCl₃

Position	δ_{H}^a	J/Hz	$^1\text{H}-^1\text{H}$ COSY	NOE	δ_{C}	HMBC (C) ^b
1						
2					88.7 (s)	
3	2.68	q	6.9 (16)	16	45.3 (d)	2, 4, 10, 13, 16
4					207.9 (s)	
5					84.1 (s)	
6					167.6 (s)	5
7	7.25	br s		19, 23, 8-OCH ₃		
8					91.6 (s)	
9	4.15	d	12.6 (9-OH)	9-OH	73.9 (d)	4, 17
10					204.0 (s)	
11	6.28	dd	6.2 (12), 2.0 (13)	12	131.7 (d)	2, 10, 12, 13
12	7.78	dd	6.2 (11), 2.0 (13)	11, 13	164.1 (d)	2, 10, 11, 13
13	3.10	ddt	12.8 (14A), 5.0 (14B), 2.0 (11, 12)	11, 12, 14	52.2 (d)	
14A	1.41	ddq	14.0 (14B), 12.8 (13), 7.3 (15)	13, 15	22.2 (t)	12, 13, 15
14B	1.91	dqd	14.0 (14A), 7.3 (15), 5.0 (14)			
15	1.22	t	7.3 (14)	14	12.2 (q)	13, 14
16	1.09	d	6.9 (3)	3	9.1 (q)	2, 3, 4
17					194.0 (s)	
18					133.1 (s)	
19	8.30	d	7.3 (20)	20	130.6 (d)	17, 21
20	7.48	t	7.3 (19, 21)	19, 21	128.5 (d)	18, 19, 21
21	7.63	t	7.3 (20, 22)	20, 22	134.4 (d)	19
22	7.48	t	7.3 (21, 23)	21, 23	128.5 (d)	18, 21, 23
23	8.30	d	7.3 (22)	22	130.6 (d)	17, 21
8-OCH ₃	3.24	s		7, 19, 23	51.3 (q)	8
9-OH	3.53	d	12.6 (9)	9		9

^a ^1H chemical shift values (δ ppm from SiMe₄) followed by multiplicity and then the coupling constants (J/Hz). Figures in parentheses indicate the proton coupling with that position. ^b Long range $^1\text{H}-^{13}\text{C}$ correlations from H to C observed in the HMBC experiment.

Table S2 NMR spectral data of **2** in CDCl₃

Position	δ_{H}^a	J/Hz	$^1\text{H}-^1\text{H COSY}$	NOE	δ_{C}	HMBC (C) ^b
1						
2					87.2 (s)	
3	2.87	q	6.9 (16)	16	45.8 (d)	2, 4, 10, 13, 16
4					206.7 (s)	
5					87.2 (s)	
6					168.7 (s)	
7	6.65	br s				
8					87.8 (s)	
9	4.54	s		3, 15, 19, 23, 8-OH ₃	82.1 (d)	8
10					204.5 (s)	
11	6.25	dd	6.2 (12), 2.0 (13)	12, 13	131.5 (d)	2, 10, 13
12	7.76	dd	6.2 (11), 2.0 (13)	11, 13	164.7 (d)	10, 11, 13
13	3.09	ddt	10.8 (14A), 6.2 (14B), 2.0 (11, 12)	11, 12, 14	51.9 (d)	
14A	1.45	ddq	14.0 (14B), 10.8 (13), 7.3 (15)	13, 15	22.4 (t)	13, 15
14B	1.90	dqd	14.0 (14A), 7.3 (15), 6.2 (13)			
15	1.21	t	7.3 (14)	14	12.2 (q)	13, 14
16	1.06	d	6.9 (3)	3	8.8 (q)	2, 3, 4
17					197.0 (s)	
18					134.3 (s)	
19	8.07	d	7.3 (20)	20	129.0 (d)	17, 21
20	7.50	t	7.3 (19, 21)	19, 21	128.9 (d)	18, 19, 21
21	7.63	t	7.3 (20, 22)	20, 22	134.0 (d)	19
22	7.50	t	7.3 (21, 23)	21, 23	128.9 (d)	18, 21, 23
23	8.07	d	7.3 (22)	22	129.0 (d)	17, 21
8-OCH ₃	3.43	s		9, 19, 23	51.7 (q)	8
9-OH	5.05	br s				

a, b As in Table S1

Table S3 NMR spectral data of **3** in CDCl₃

Position	δ_{H}^a		J/Hz	$^1\text{H}-^1\text{H}$ COSY		NOE	δ_{C}	HMBC (C) ^b
1								
2							89.2 (s)	
3	2.91	q	6.9 (16)	16	14, 15, 16		46.2 (d)	2, 4, 10, 13, 16
4							203.4 (s)	
5							89.1 (s)	
6							168.6 (s)	
7	7.14	br s				9, 19, 23, 8-OCH ₃		
8							90.8 (s)	
9	4.36	d	3.2 (9-OH)	9-OH	7, 19, 23, 8-OCH ₃ , 9-OH		76.1 (d)	4, 17
10							211.0 (s)	
11	6.23	dd	6.2 (12), 2.0 (13)	12	12, 16		131.0 (d)	2, 10, 12, 13
12	7.83	dd	6.2 (11), 2.0 (13)	11, 13	11, 13, 15, 16		168.0 (d)	2, 10, 11, 13
13	3.12	ddt	11.8 (14A), 6.5 (14B), 2.0 (11, 12)	11, 12, 14	12, 14, 15		51.1 (d)	2, 14
14A	1.52	ddq	14.0 (14B), 11.8 (13), 7.3 (15)	13, 15	3, 12, 13, 15, 16		21.3 (t)	12, 13, 15
14B	1.97	dqd	14.0 (14A), 7.3 (15), 6.5 (13)					
15	1.25	t	7.3 (14)	14	3, 12, 13, 14, 16		12.1 (q)	13, 14
16	1.00	d	6.9 (3)	3	3, 11, 12, 14, 15, 9-OH		9.2 (q)	2, 3, 4
17							194.9 (s)	
18							132.4 (s)	
19	8.49	d	7.3 (20)	20	7, 9, 20, 8-OCH ₃ , 9-OH		131.2 (d)	17, 21
20	7.47	t	7.3 (19, 21)	19, 21	19, 21		128.4 (d)	19, 21, 23
21	7.62	t	7.3 (20, 22)	20, 22	20, 22		134.4 (d)	19
22	7.47	t	7.3 (21, 23)	21, 23	21, 23		128.4 (d)	19, 21, 23
23	8.49	d	7.3 (22)	22	7, 9, 22, 8-OCH ₃ , 9-OH		131.2 (d)	17, 21
8-OCH ₃	3.37	s			7, 9, 19, 23, 9-OH		51.7 (q)	8
9-OH	5.51	d	3.2 (9)	9	9, 16, 19, 23, 8-OCH ₃			8, 9

a, b As in Table S1

Table S4 NMR spectral data of **4** in CDCl₃

Position	δ_{H}^a		J/Hz	$^1\text{H}-^1\text{H}$ COSY		NOE	δ_{C}	HMBC (C) ^b
1								
2							89.8 (s)	
3	3.03	q	6.9 (16)	16	14, 15, 16		46.1 (d)	2, 4, 10, 13, 16
4							204.8 (s)	
5							86.1 (s)	
6							169.8 (s)	
7	7.12	br s			8-OCH ₃			5, 9
8							94.8 (s)	
9	4.95	d	1.8 (9-OH)	9-OH	16, 19, 23, 8-OCH ₃ , 9-OH		76.4 (d)	4, 6, 8
10							209.0 (s)	
11	6.20	dd	6.2 (12), 2.0 (13)	12, 13	12, 16		130.9 (d)	2, 10, 12, 13
12	7.81	dd	6.2 (11), 2.0 (13)	11, 13	11, 13, 14, 15		167.1 (d)	2, 10, 11, 13
13	3.16	ddt	10.0 (14A), 7.3 (14B), 2.0 (11, 12)	11, 12, 14	12, 14, 15		50.2 (d)	14
14A	1.56	ddq	14.0 (14B), 10.0 (13), 7.3 (15)	13, 15	3, 12, 13, 15, 16		21.3 (t)	2, 12, 13, 15
14B	2.00	d quint	14.0 (14A), 7.3 (13, 15)					
15	1.26	t	7.3 (14)	14	3, 12, 13, 14		12.1 (q)	13, 14
16	1.00	d	6.9 (3)	3	3, 9, 11, 14		9.1 (q)	2, 3, 4
17							193.5 (s)	
18							133.9 (s)	
19	8.27	d	7.3 (20)	20	9, 20, 8-OCH ₃		129.8 (d)	17, 21
20	7.49	t	7.3 (19, 21)	19, 21	19, 21		128.6 (d)	18, 19, 21
21	7.61	t	7.3 (20, 22)	20, 22	20, 22		133.8 (d)	19
22	7.49	t	7.3 (21, 23)	21, 23	21, 23		128.6 (d)	18, 21, 23
23	8.27	d	7.3 (22)	22	9, 22, 8-CH ₃		129.8 (d)	17, 21
8-OCH ₃	3.25	s			7, 9, 19, 23		51.2 (q)	8
9-OH	2.62	d	1.8 (9)	9	9			5, 8, 9

a, b As in Table S1

Table S5 NMR spectral data of **5** in CDCl₃

Position	δ_{H}^a		J/Hz	$^1\text{H}-^1\text{H}$ COSY		NOE	δ_{C}	HMBC (C) ^b
1								
2							87.0 (s)	
3	2.88	q	6.9 (16)	16	9, 14, 15, 16		44.0 (d)	2, 4, 10, 13, 16
4							202.7 (s)	
5							85.1 (s)	
6							168.7 (s)	
7	7.32	br s			8-OCH ₃			
8							92.8 (s)	
9	4.44	s			3, 14, 19, 23, 8-OH ₃ , 9-OH		78.2 (d)	4, 5, 6, 8
10							203.4 (s)	
11	6.29	dd	6.2 (12), 2.0 (13)	12, 13	12, 16		131.6 (d)	2, 10, 12, 13
12	7.74	dd	6.2 (11), 2.0 (13)	11, 13	11, 13, 14, 15		164.4 (d)	2, 10, 11, 13
13	2.95	ddt	10.8 (14A), 6.2 (14B), 2.0 (11, 12)	11, 12, 14	12, 14, 15		51.7 (d)	
14A	1.54	ddq	14.0 (14B), 10.8 (13), 7.3 (15)	13, 15	3, 9, 12, 13, 15, 16		22.3 (t)	2, 12, 13, 15
14B	1.93	dqd	14.0 (14A), 7.3 (15), 6.2 (13)					
15	1.14	t	7.3 (14)	14	3, 12, 13, 14, 9-OH		12.3 (q)	13, 14
16	1.10	d	6.9 (3)	3	3, 11, 14		8.7 (q)	2, 3, 4
17							192.2 (s)	
18							133.9 (s)	
19	8.20	d	7.3 (20)	20	9, 20, 8-OCH ₃		129.5 (d)	17, 21
20	7.49	t	7.3 (19, 21)	19, 21	19, 21		128.7 (d)	18, 19, 21
21	7.62	t	7.3 (20, 22)	20, 22	20, 22		134.0 (d)	19
22	7.49	t	7.3 (21, 23)	21, 23	21, 23		128.7 (d)	18, 21, 23
23	8.20	d	7.3 (22)	22	9, 22, 8-OCH ₃		129.5 (d)	17, 21
8-OCH ₃	3.25	s			7, 9, 19, 23		50.9 (q)	8
9-OH	2.96	br s			9, 15			

a, b As in Table S1

Table S6 NMR spectral data of **6** in CDCl₃

Position	δ_{H}^a	J/Hz	^1H - ^1H COSY	NOE	δ_{C}	HMBC (C) ^b
1						
2					86.3 (s)	
3	2.90	q	6.9 (16)	16	14, 15, 16, 9-OH	45.6 (d) 4, 5, 13, 16
4					205.0 (s)	
5					88.9 (s)	
6					167.7 (s)	
7	7.28	br s		9, 8-OCH ₃		5
8					88.0 (s)	
9	4.19	d	11.0 (9-OH)	9-OH	7, 15, 19, 23, 9-OH	76.5 (d) 4, 5, 17
10					204.7 (s)	
11	6.27	dd	6.4 (12), 2.0 (13)	12	12, 16	131.5 (d) 2, 10, 12, 13
12	7.74	dd	6.4 (11), 2.0 (13)	11, 13	11, 13, 14, 15	164.9 (d) 2, 10, 11, 13
13	2.97	ddt	12.2 (14A), 6.6 (14B), 2.0 (11, 12)	11, 12, 14	12, 14, 15	51.8 (d)
14A	1.43	ddq	14.0 (14B), 12.2 (13), 7.3 (15)	13, 15	3, 12, 13, 15, 16	22.4 (t) 2, 12, 13, 15
14B	1.89	dqd	14.0 (14A), 7.3 (15), 6.6 (13)			
15	1.15	t	7.3 (14)	14	3, 9, 12, 13, 14	12.2 (q) 13, 14
16	1.06	d	6.9 (3)	3	3, 11, 14	8.6 (q) 2, 3, 4
17					194.1 (s)	
18					132.9 (s)	
19	8.18	d	7.3 (20)	20	9, 20, 8-OCH ₃ , 9-OH	130.2 (d) 17, 20, 21
20	7.48	t	7.3 (19, 21)	19, 21	19, 21	128.7 (d) 18, 19, 21, 22
21	7.63	t	7.3 (20, 22)	20, 22	20, 22	134.3 (d) 19, 23
22	7.48	t	7.3 (21, 23)	21, 23	21, 23	128.7 (d) 18, 20, 21, 23
23	8.18	d	7.3 (22)	22	9, 22, 8-OCH ₃ , 9-OH	130.2 (d) 17, 21, 22
8-OCH ₃	3.38	s			7, 19, 23	51.2 (q) 8
9-OH	3.55	d	11.0 (9)	9	3, 9, 19, 23	5, 9, 8-OCH ₃

^a, ^b As in Table 1

Table S7 NMR spectral data of **7** in CDCl₃

Position	δ_{H}^a	J/Hz	$^1\text{H}-^1\text{H}$ COSY	NOE	δ_{C}	HMBC (C) ^b
1						
2					88.5 (s)	
3	3.09	q	6.9 (16)	16	47.2 (d)	2, 4, 10, 13, 16
4					209.4 (s)	
5					83.0 (s)	
6					170.5 (s)	
7	7.14	br s		19, 23, 8-OCH ₃		
8					96.7 (s)	
9	4.99	d	2.6 (9-OH)	9-OH	81.8 (d)	4, 17
10					209.0 (s)	
11	6.25	dd	6.2 (12), 2.0 (13)	12	131.1 (d)	2, 10, 12, 13
12	7.84	dd	6.2 (11), 2.0 (13)	11, 13	167.4 (d)	2, 10, 11, 13
13	3.14	ddt	10.8 (14A), 6.2 (14B), 2.0 (11, 12)	11, 12, 14	50.1 (d)	
14A	1.54	ddq	14.0 (14B), 10.8 (13), 7.3 (15)	13, 15	21.2 (t)	2, 12, 13, 15
14B	1.93	dqd	14.0 (14A), 7.3 (15), 6.2 (13)			
15	1.22	t	7.3 (14)	14	12.2 (q)	13, 14
16	0.97	d	6.9 (3)	3	8.6 (q)	2, 3, 4
17					192.7 (s)	
18					133.9 (s)	
19	8.25	d	7.3 (20)	20	129.7 (d)	17, 21
20	7.48	t	7.3 (19, 21)	19, 21	128.6 (d)	18, 19, 21
21	7.61	t	7.3 (20, 22)	20, 22	133.8 (d)	19
22	7.48	t	7.3 (21, 23)	21, 23	128.6 (d)	18, 21, 23
23	8.25	d	7.3 (22)	22	129.7 (d)	17, 21
8-OCH ₃	3.20	s		7, 19, 23	51.2 (q)	8
9-OH	4.44	d	2.6 (9)	9		9

^a, ^b As in Table S1

Table S8 NMR spectral data of **8** in CDCl₃

Position	δ_{H}^a		J/Hz	$^1\text{H}-^1\text{H COSY}$	NOE	δ_{C}	HMBC (C) ^b
1							
2						89.4 (s)	
3	3.19	q	7.1 (16)	16	14, 15, 16	46.2 (d)	2, 4, 10, 16
4						205.8 (s)	
5						85.0 (s)	
6						169.4 (s)	
7	6.98	br s			8, 9, 8-CH ₃		5, 8, 9
8						93.5 (s)	
9	4.62	d	2.3 (9-OH)	9-OH	7, 16, 19, 23, 8-OCH ₃ , 9-OH	70.1 (d)	4, 17
10						211.9 (s)	
11	6.27	dd	6.2 (12), 2.0 (13)	12	12, 16	131.1 (d)	2, 10, 12, 13
12	7.84	dd	6.2 (11), 2.0 (13)	11, 13	11, 13, 14, 15	167.7 (d)	2, 10, 13
13	3.20	ddt	12.0 (14A), 6.8 (14B), 2.0 (11, 12)	11, 12, 14	12, 14, 15	50.5 (d)	2, 10, 12, 14
14A	1.56	ddq	14.0 (14B), 12.0 (13), 7.3 (15)	13, 15	3, 12, 13, 15, 16	21.0 (t)	2, 12, 13, 15
14B	1.96	dqd	14.0 (14A), 7.3 (15), 6.8 (13)				
15	1.25	t	7.3 (14)	14	3, 12, 13, 14	12.1 (q)	13, 14
16	0.96	d	7.1 (3)	3	3, 9, 11, 14	8.8 (q)	2, 3, 4
17						194.1 (s)	
18						132.7 (s)	
19	8.48	d	7.3 (20)	20	9, 20, 8-OCH ₃ , 9-OH	131.2 (d)	17, 21, 23
20	7.49	t	7.3 (19, 21)	19, 21	19, 21	128.4 (d)	18, 19, 21, 22
21	7.63	t	7.3 (20, 22)	20, 22	20, 22	134.4 (d)	19, 20, 22, 23
22	7.49	t	7.3 (21, 23)	21, 23	21, 23	128.4 (d)	18, 21, 23
23	8.48	d	7.3 (22)	22	9, 22, 8-OCH ₃ , 9-OH	131.2 (d)	17, 19, 21
8-OCH ₃	3.33	s			7, 9, 19, 23, 9-OH	51.8 (q)	8
9-OH	4.52	d	2.3 (9)	9	9, 19, 23, 8-OCH ₃		8, 9

^a, ^b As in Table S1

Figure S1 ^1H and ^{13}C NMR spectra of **1** in CDCl_3

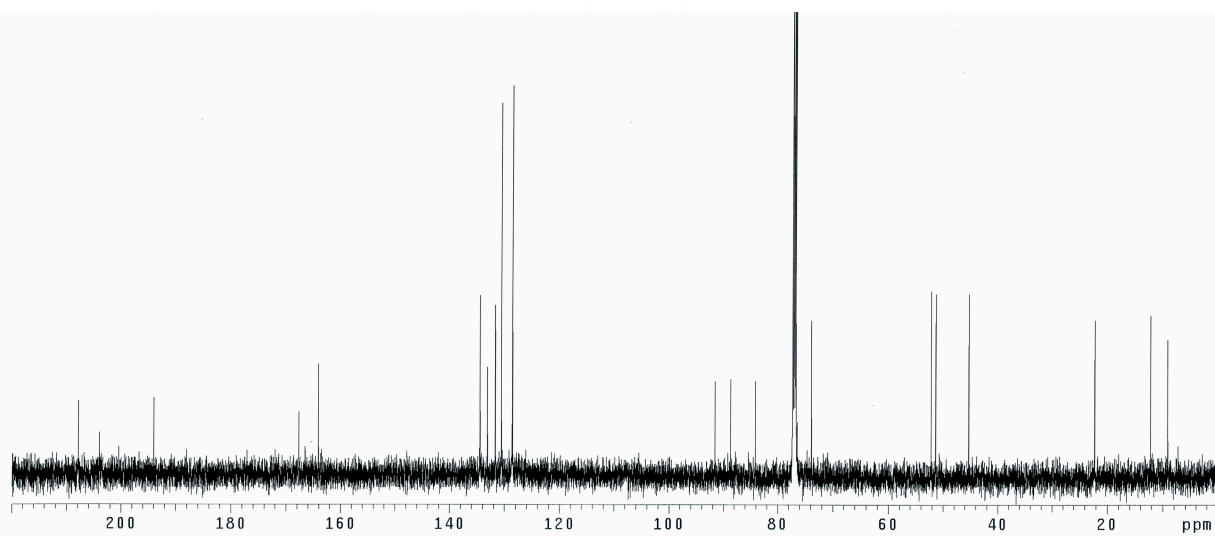
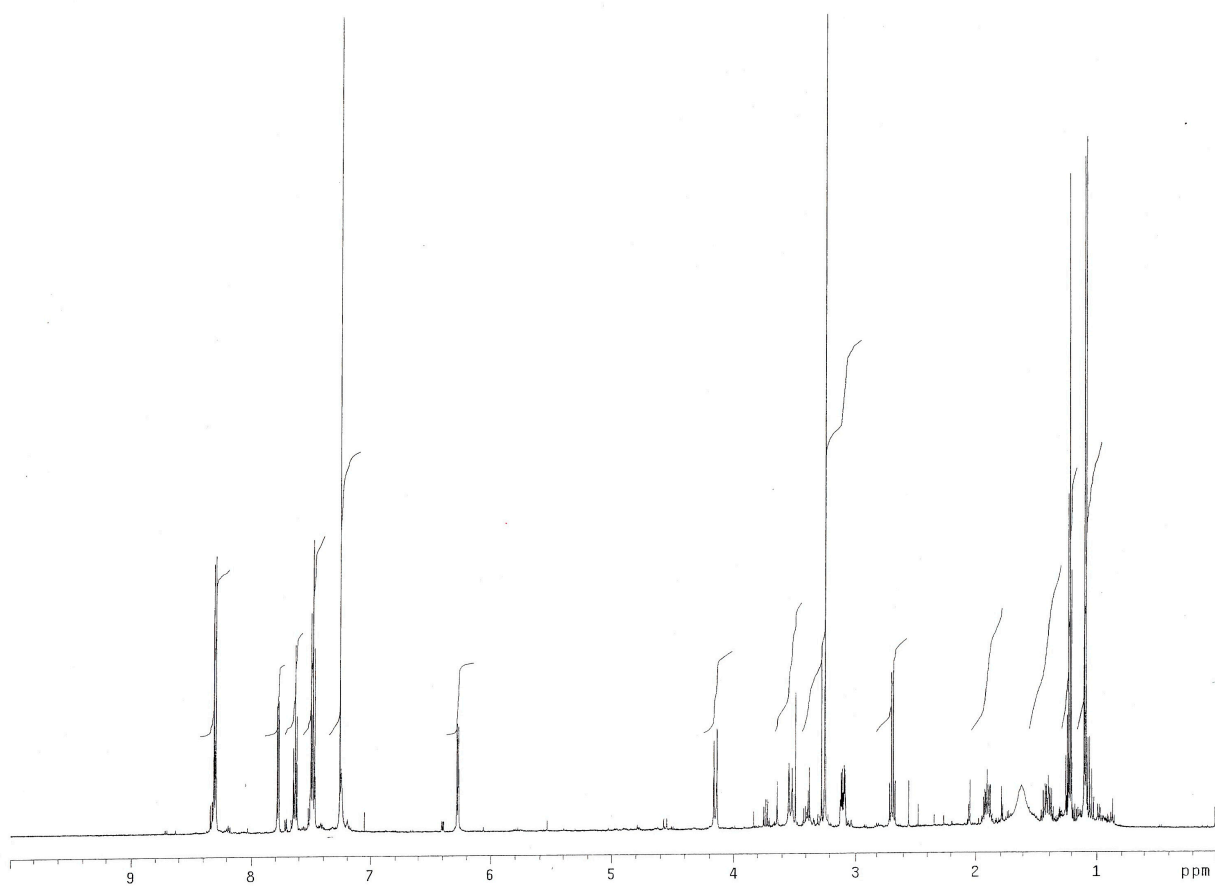


Figure S2 ^1H - ^1H COSY of **1**

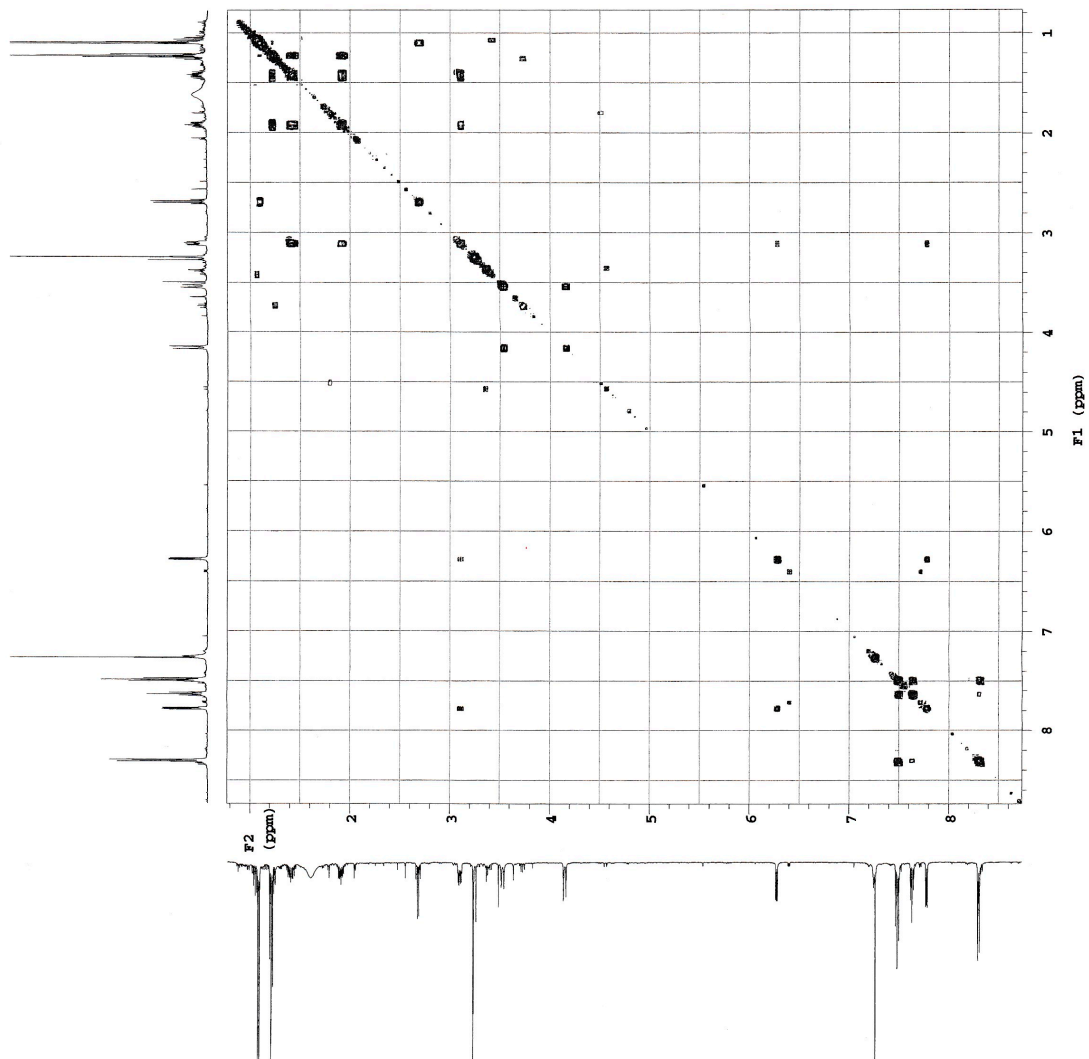
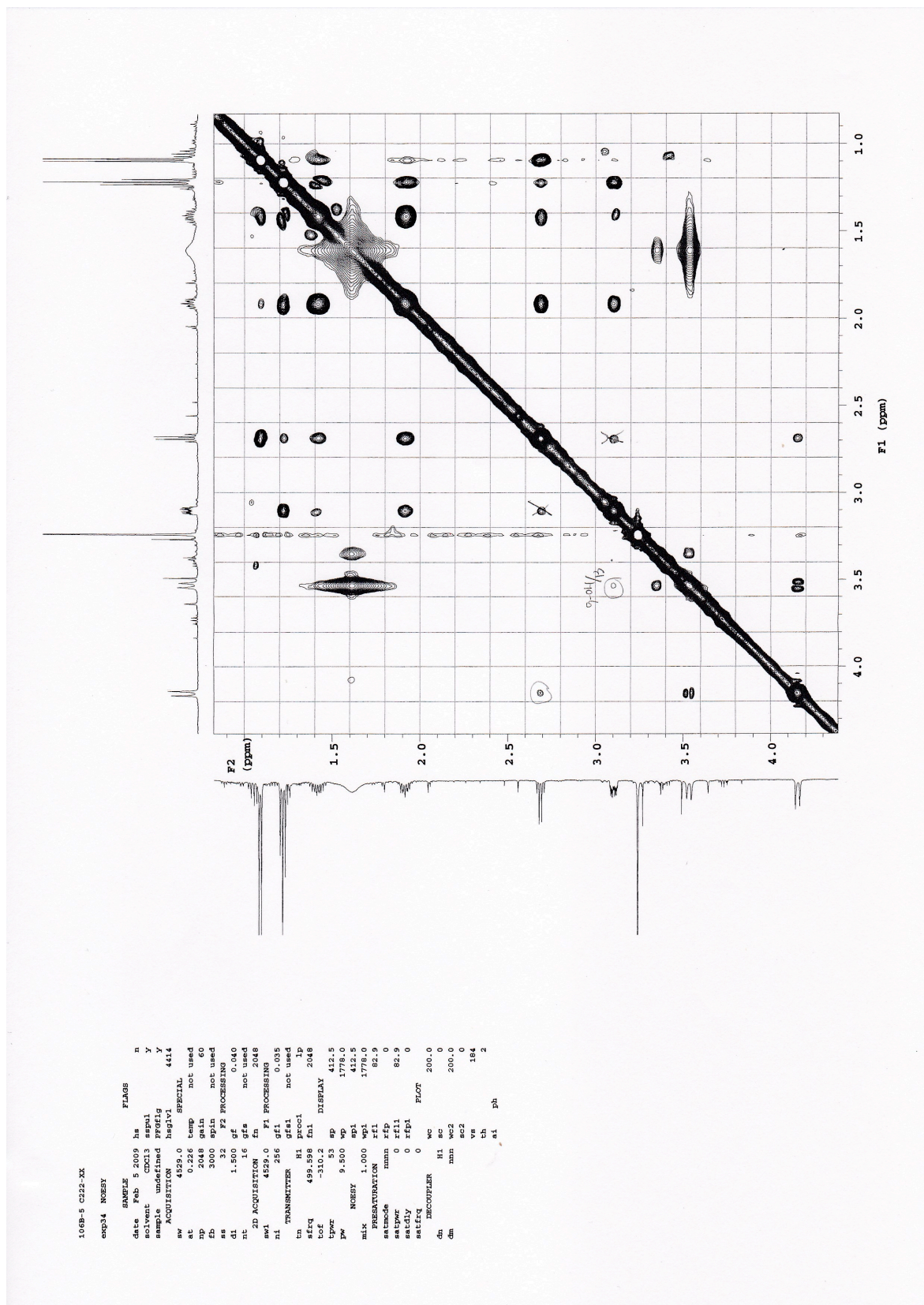
[illegible]

Figure S3 NOESY of **1**



106B-5 C222-XX
Pulse Sequence: NOESY

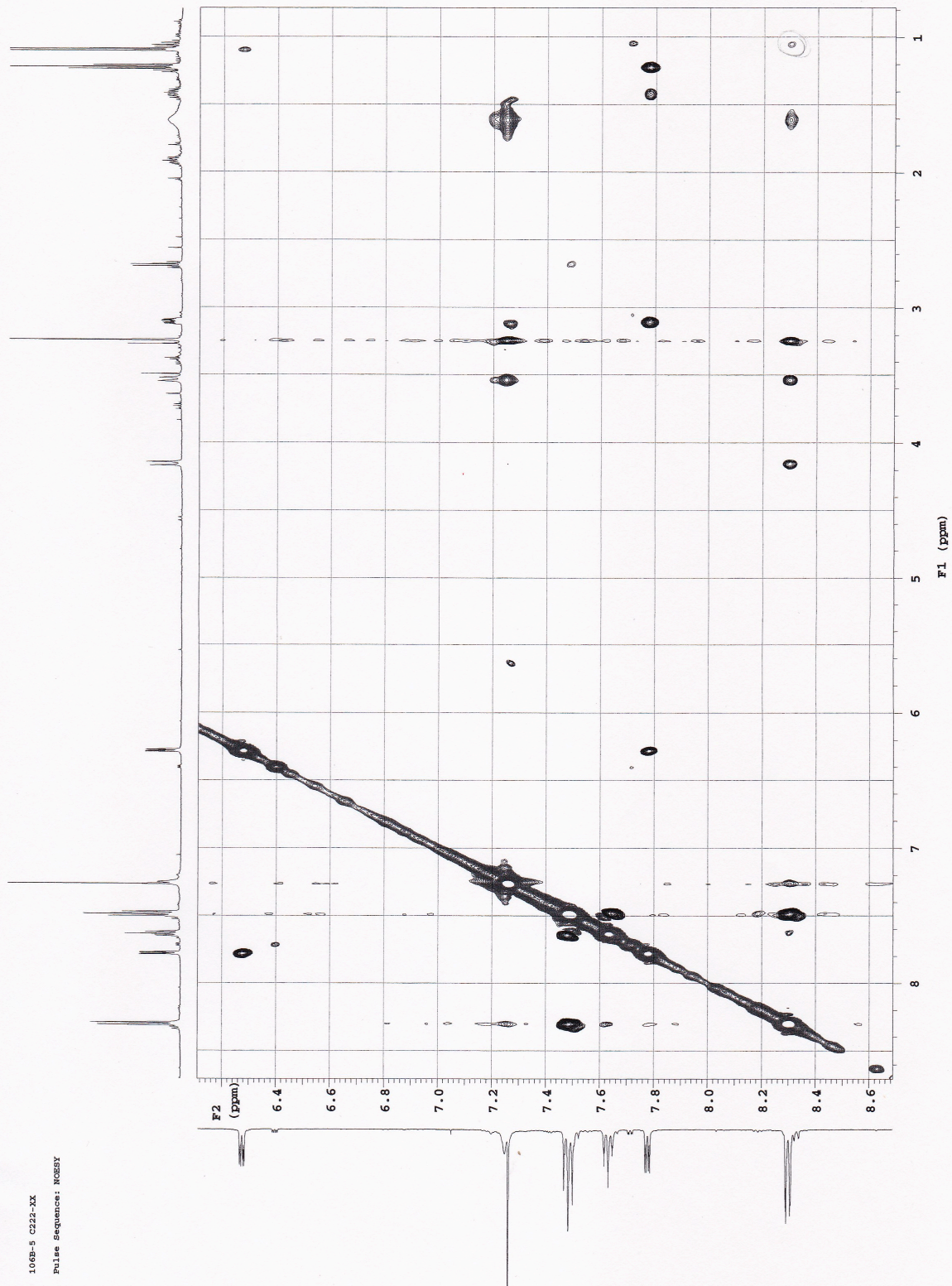


Figure S4 HMQC of **1**

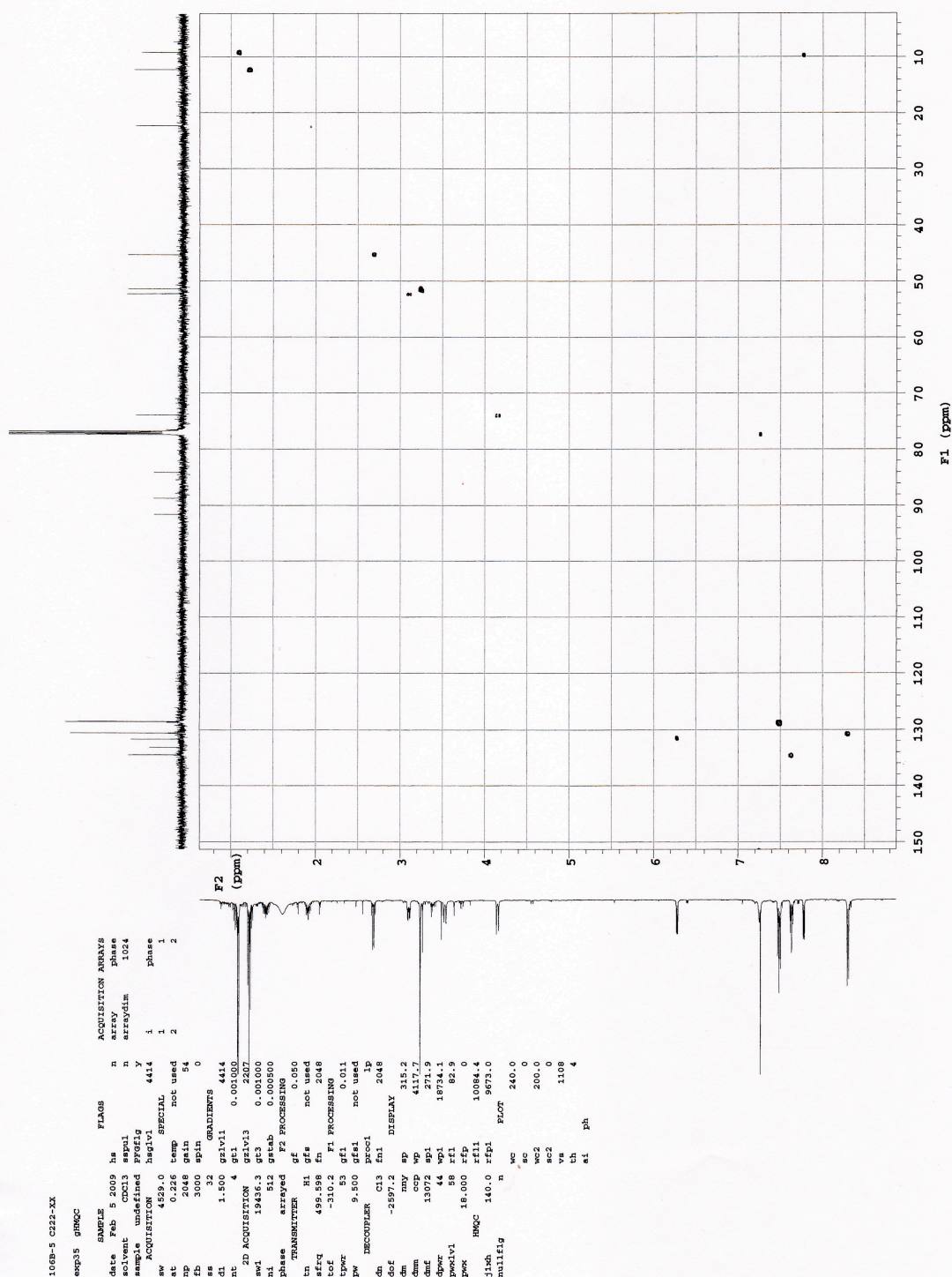


Figure S5 HMBC of 1

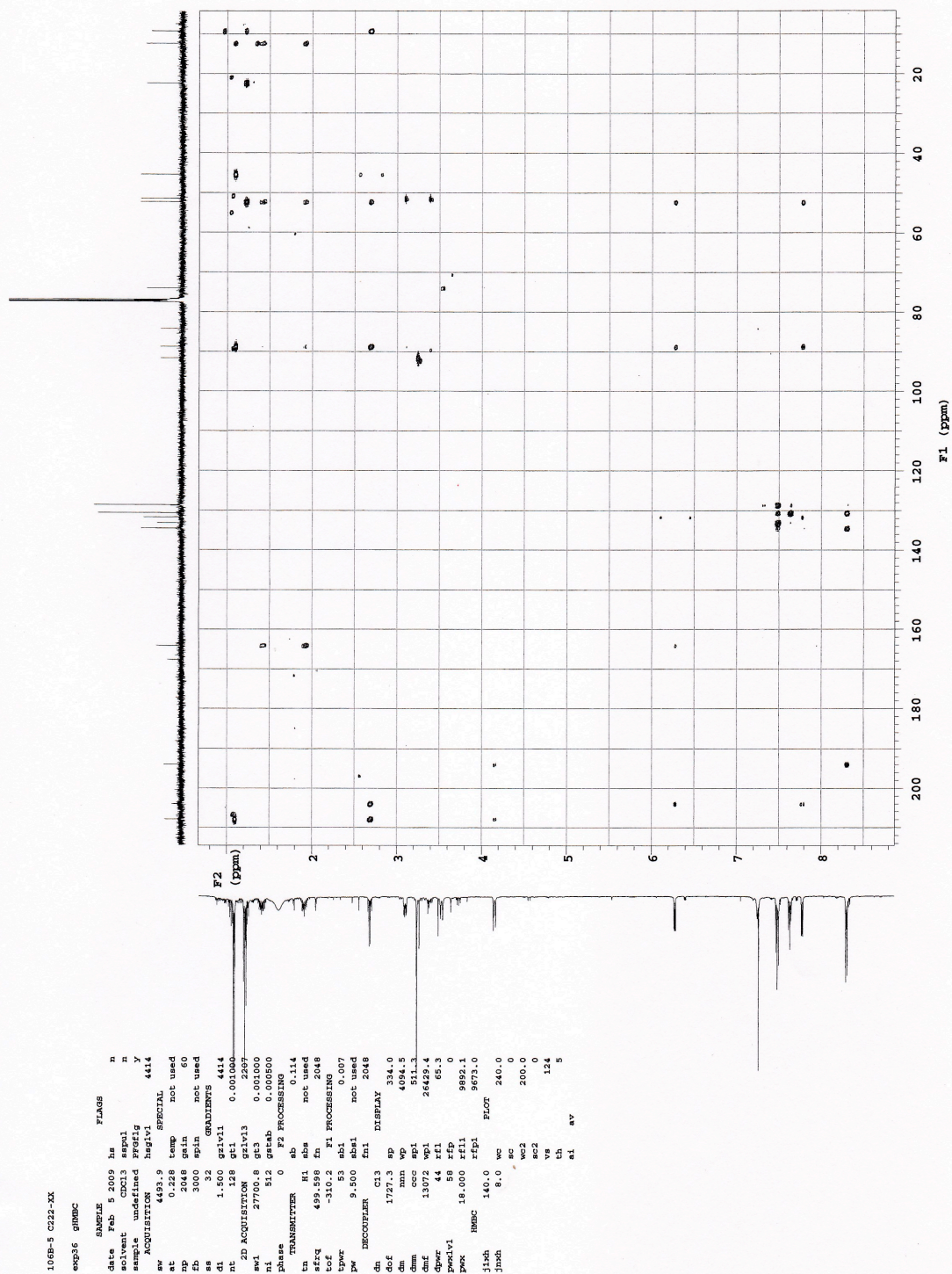


Figure S6 IR spectrum of 1

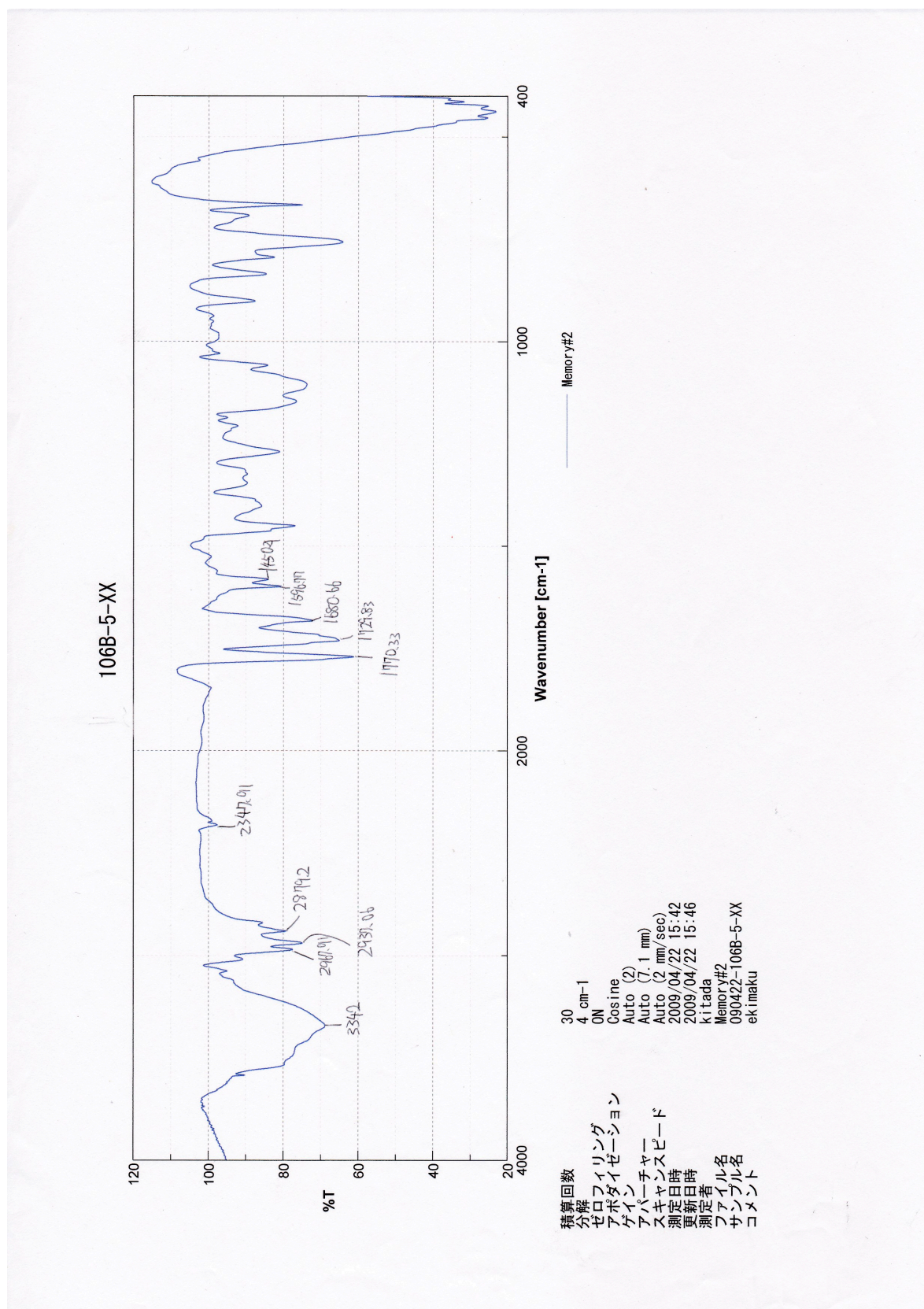


Figure S7 FABMS of 1

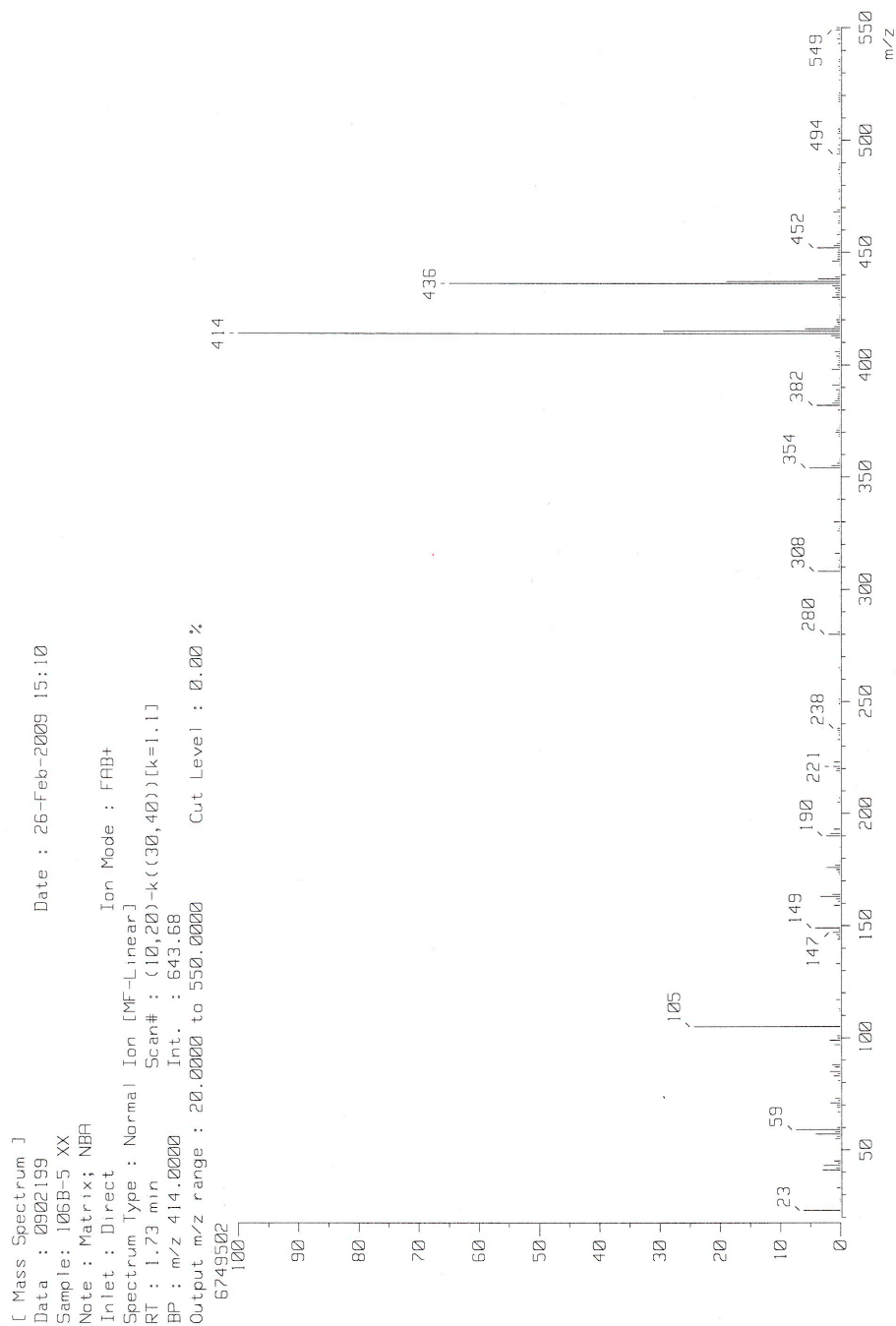


Figure S8 ^1H and ^{13}C NMR spectra of **2** in CDCl_3

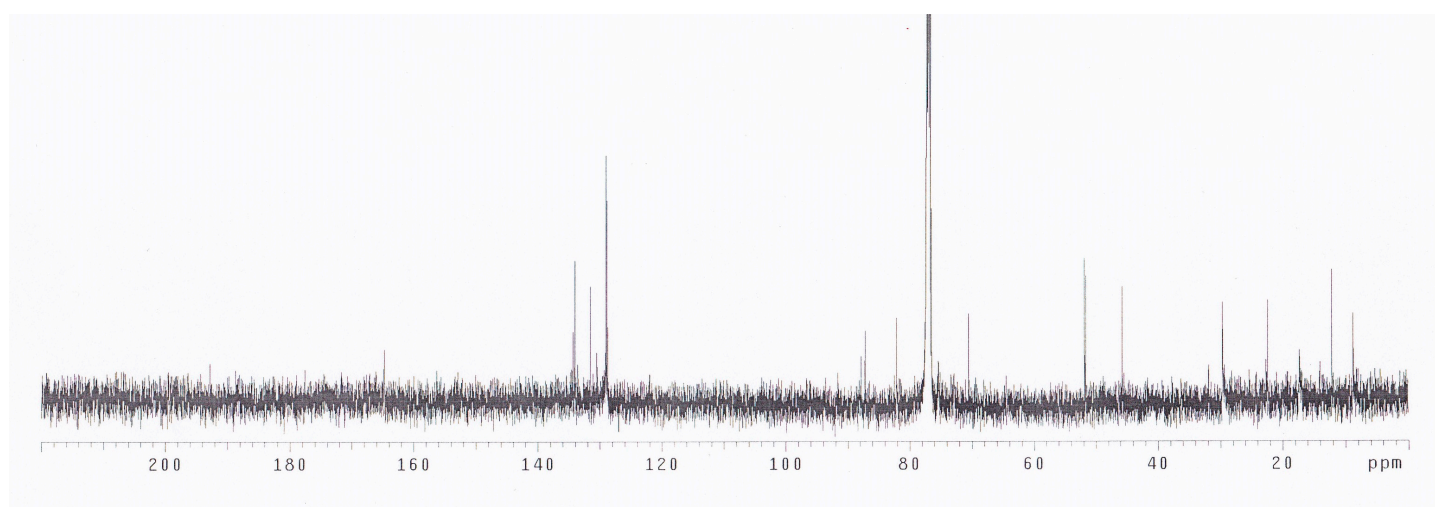
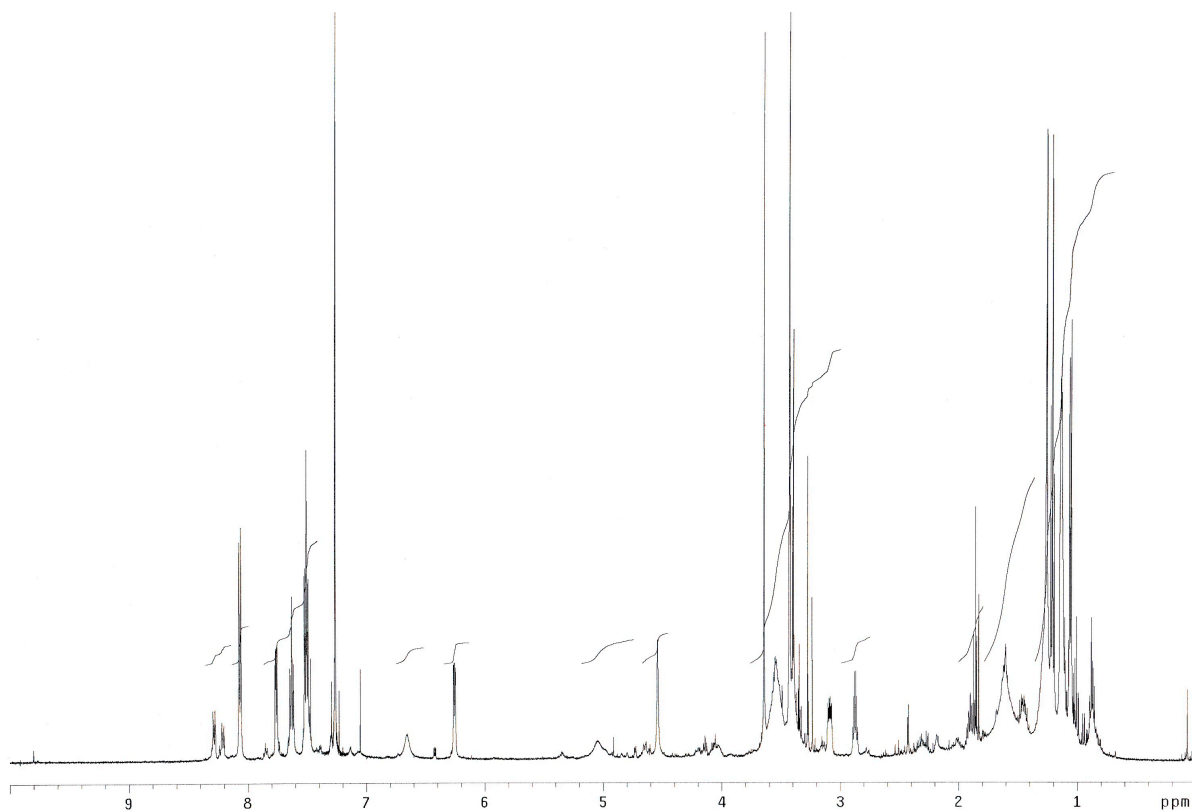


Figure S9 H-¹H COSY of 2

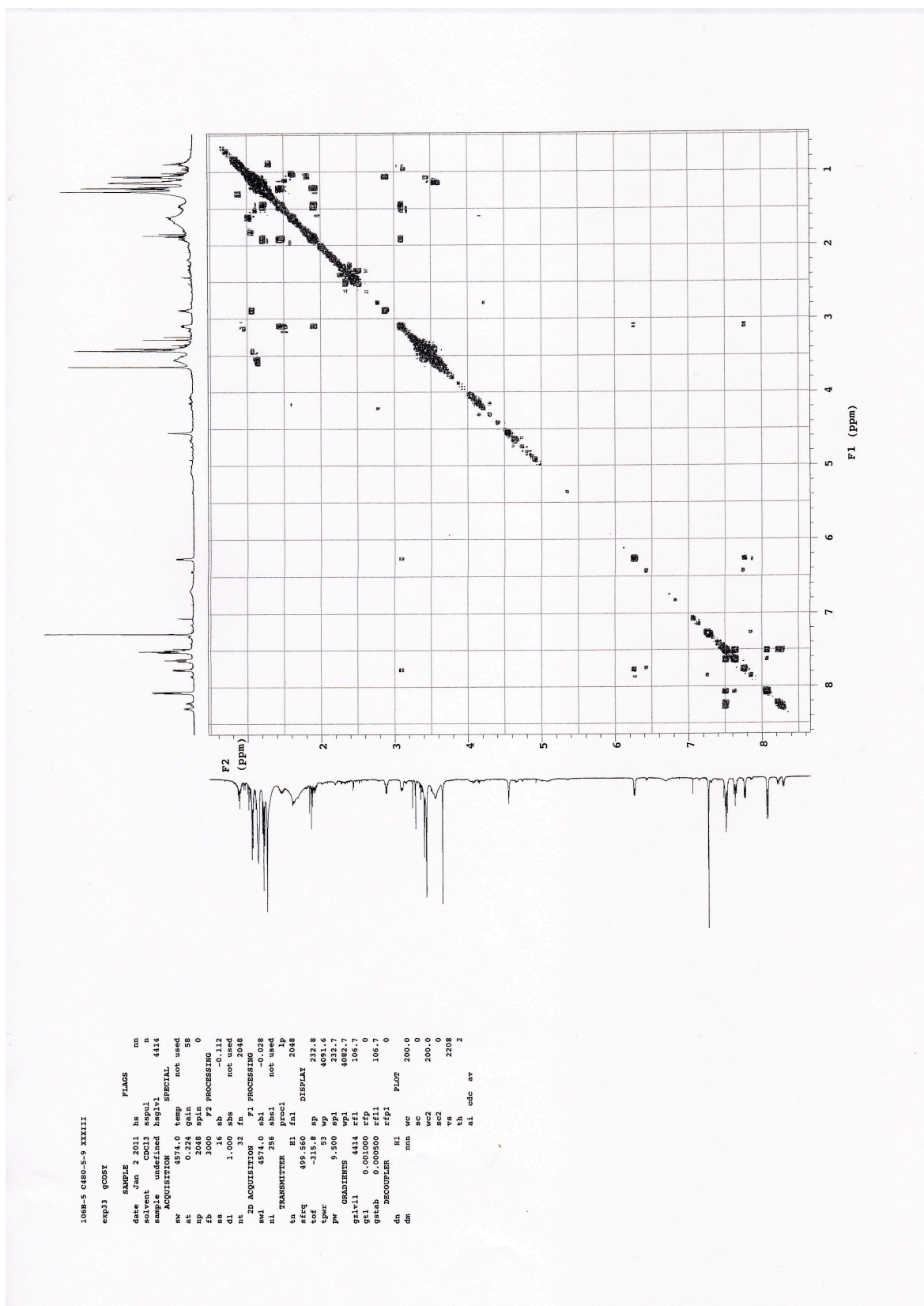
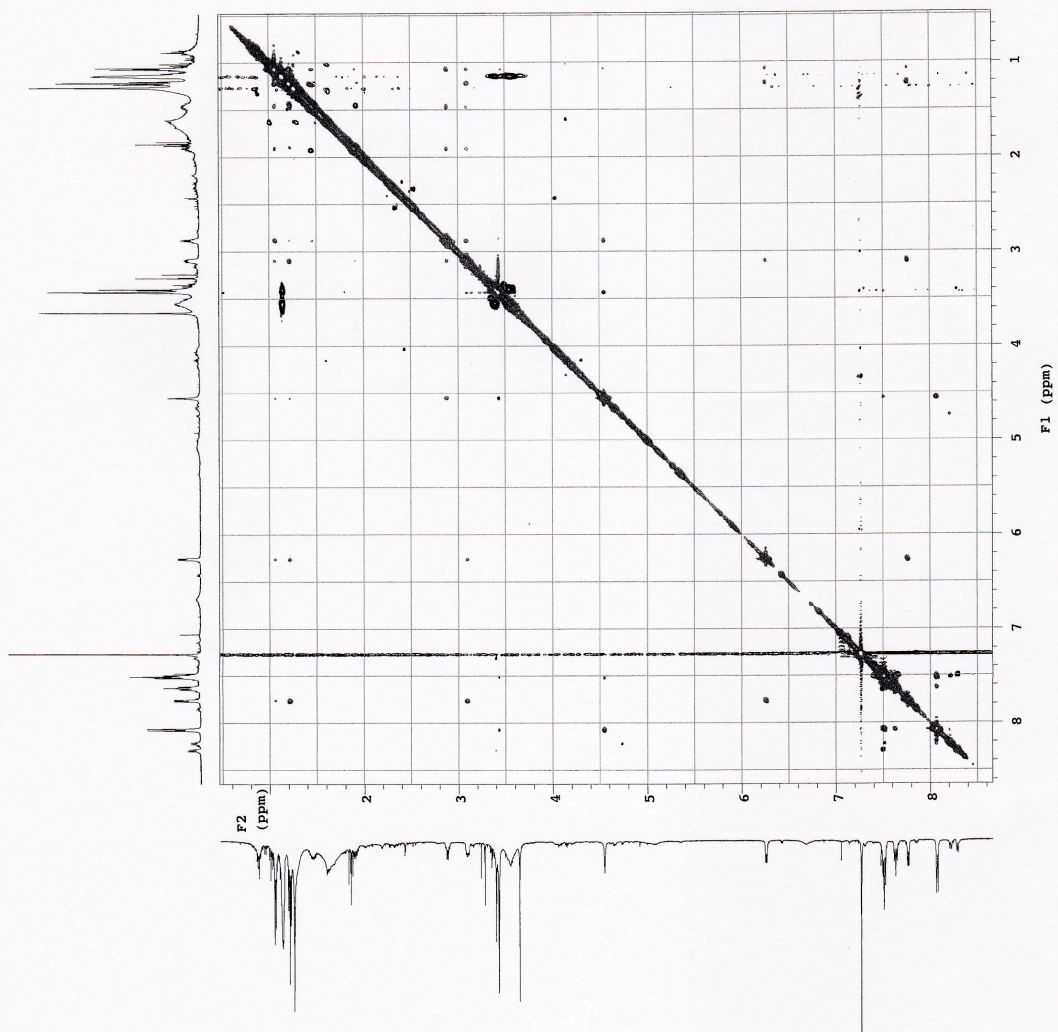


Figure S10 NOESY of 2



exp34	NOISE	SAMPLE	date	Jan	2011	h	FLAGS	n
		acq1	20110101	0000	0000	0000	0000	0
		acq2	20110101	0000	0000	0000	0000	0
		acq3	20110101	0000	0000	0000	0000	0
		acq4	20110101	0000	0000	0000	0000	0
		acq5	20110101	0000	0000	0000	0000	0
		acq6	20110101	0000	0000	0000	0000	0
		acq7	20110101	0000	0000	0000	0000	0
		acq8	20110101	0000	0000	0000	0000	0
		acq9	20110101	0000	0000	0000	0000	0
		acq10	20110101	0000	0000	0000	0000	0
		acq11	20110101	0000	0000	0000	0000	0
		acq12	20110101	0000	0000	0000	0000	0
		acq13	20110101	0000	0000	0000	0000	0
		acq14	20110101	0000	0000	0000	0000	0
		acq15	20110101	0000	0000	0000	0000	0
		acq16	20110101	0000	0000	0000	0000	0
		acq17	20110101	0000	0000	0000	0000	0
		acq18	20110101	0000	0000	0000	0000	0
		acq19	20110101	0000	0000	0000	0000	0
		acq20	20110101	0000	0000	0000	0000	0
		acq21	20110101	0000	0000	0000	0000	0
		acq22	20110101	0000	0000	0000	0000	0
		acq23	20110101	0000	0000	0000	0000	0
		acq24	20110101	0000	0000	0000	0000	0
		acq25	20110101	0000	0000	0000	0000	0
		acq26	20110101	0000	0000	0000	0000	0
		acq27	20110101	0000	0000	0000	0000	0
		acq28	20110101	0000	0000	0000	0000	0
		acq29	20110101	0000	0000	0000	0000	0
		acq30	20110101	0000	0000	0000	0000	0
		acq31	20110101	0000	0000	0000	0000	0
		acq32	20110101	0000	0000	0000	0000	0
		acq33	20110101	0000	0000	0000	0000	0
		acq34	20110101	0000	0000	0000	0000	0
		acq35	20110101	0000	0000	0000	0000	0
		acq36	20110101	0000	0000	0000	0000	0
		acq37	20110101	0000	0000	0000	0000	0
		acq38	20110101	0000	0000	0000	0000	0
		acq39	20110101	0000	0000	0000	0000	0
		acq40	20110101	0000	0000	0000	0000	0
		acq41	20110101	0000	0000	0000	0000	0
		acq42	20110101	0000	0000	0000	0000	0
		acq43	20110101	0000	0000	0000	0000	0
		acq44	20110101	0000	0000	0000	0000	0
		acq45	20110101	0000	0000	0000	0000	0
		acq46	20110101	0000	0000	0000	0000	0
		acq47	20110101	0000	0000	0000	0000	0
		acq48	20110101	0000	0000	0000	0000	0
		acq49	20110101	0000	0000	0000	0000	0
		acq50	20110101	0000	0000	0000	0000	0
		acq51	20110101	0000	0000	0000	0000	0
		acq52	20110101	0000				

Figure S11 HMQC of **2**

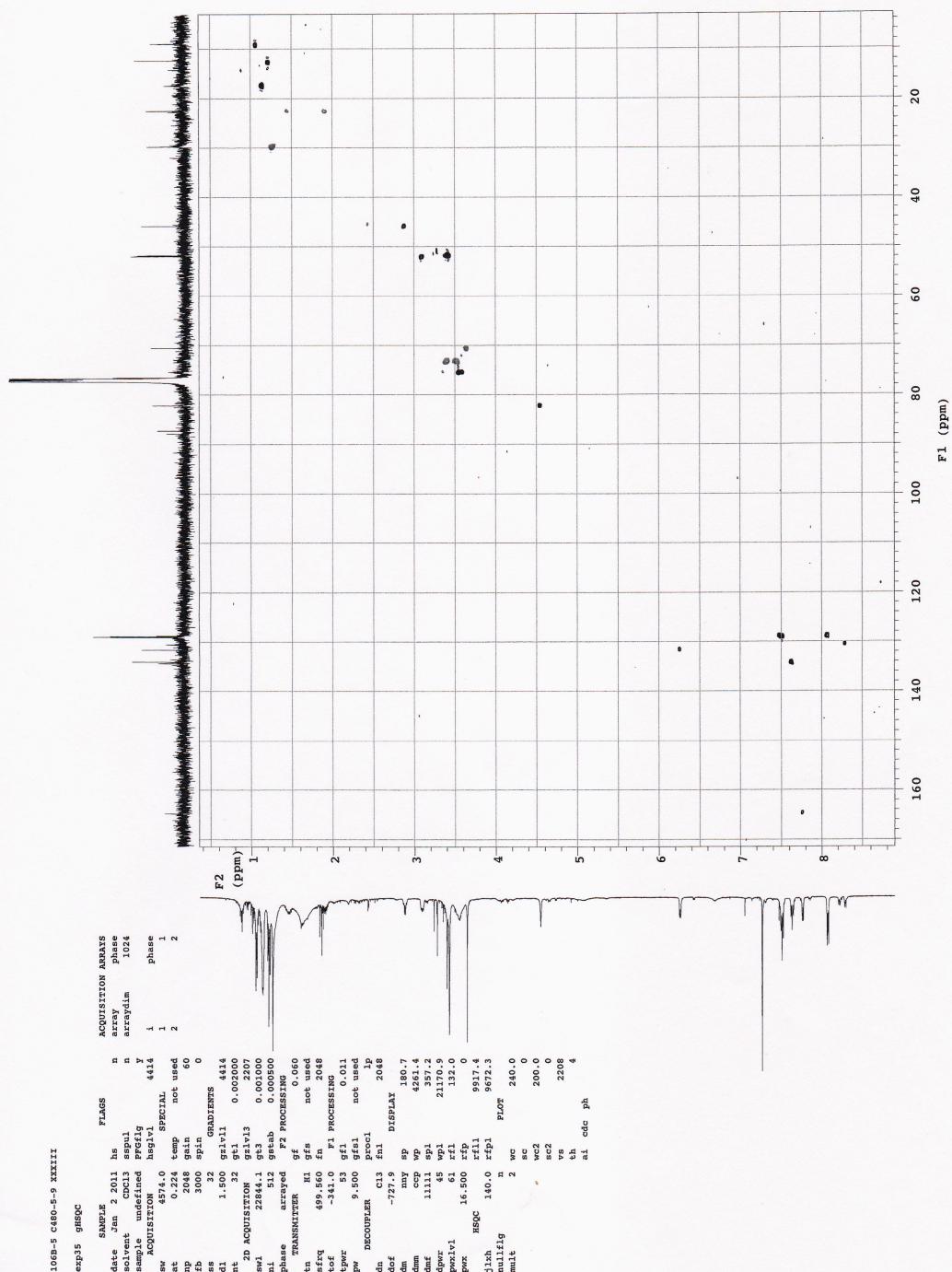


Figure S12 HMBC of **2**

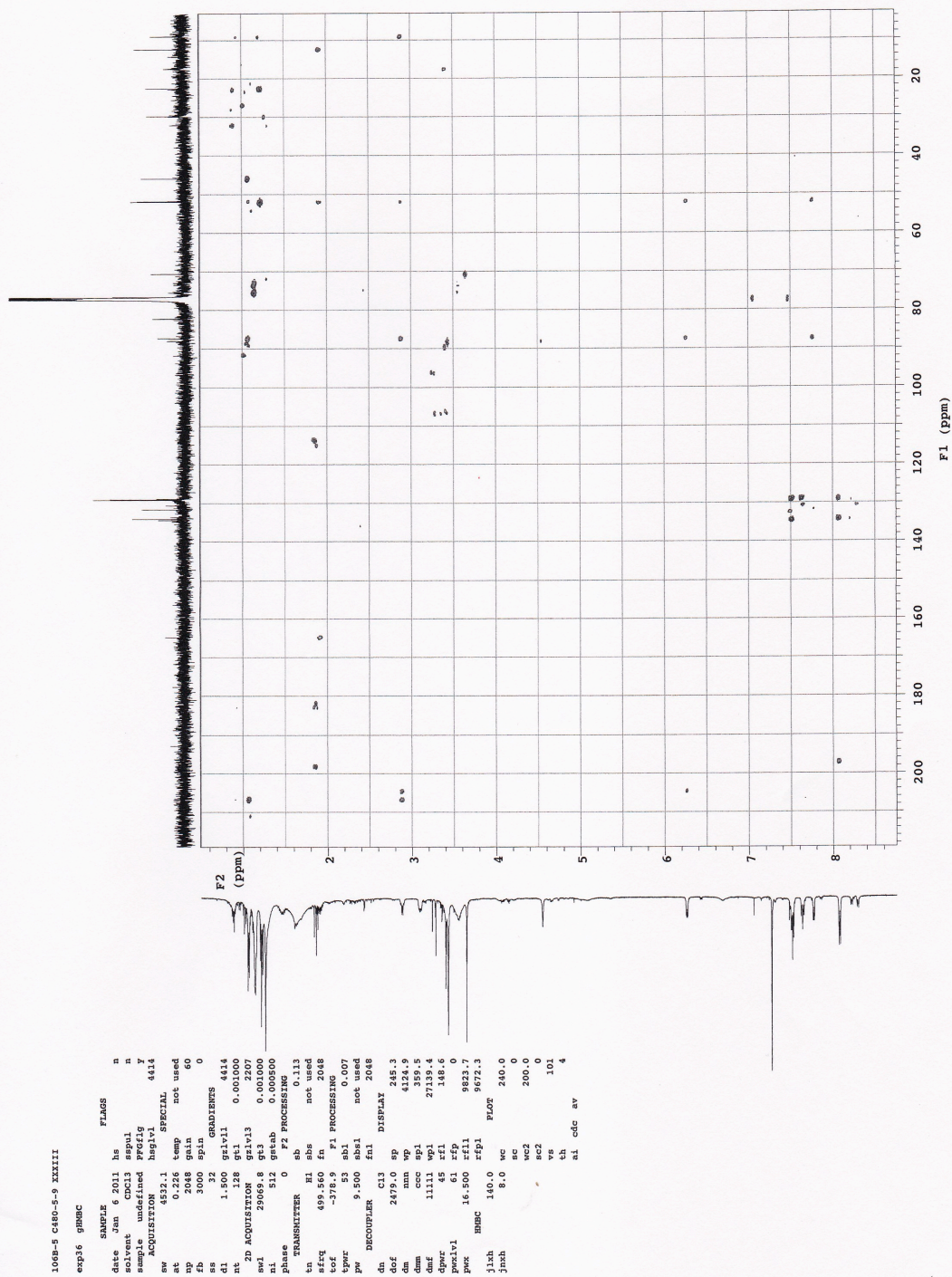


Figure S13 IR spectrum of 2

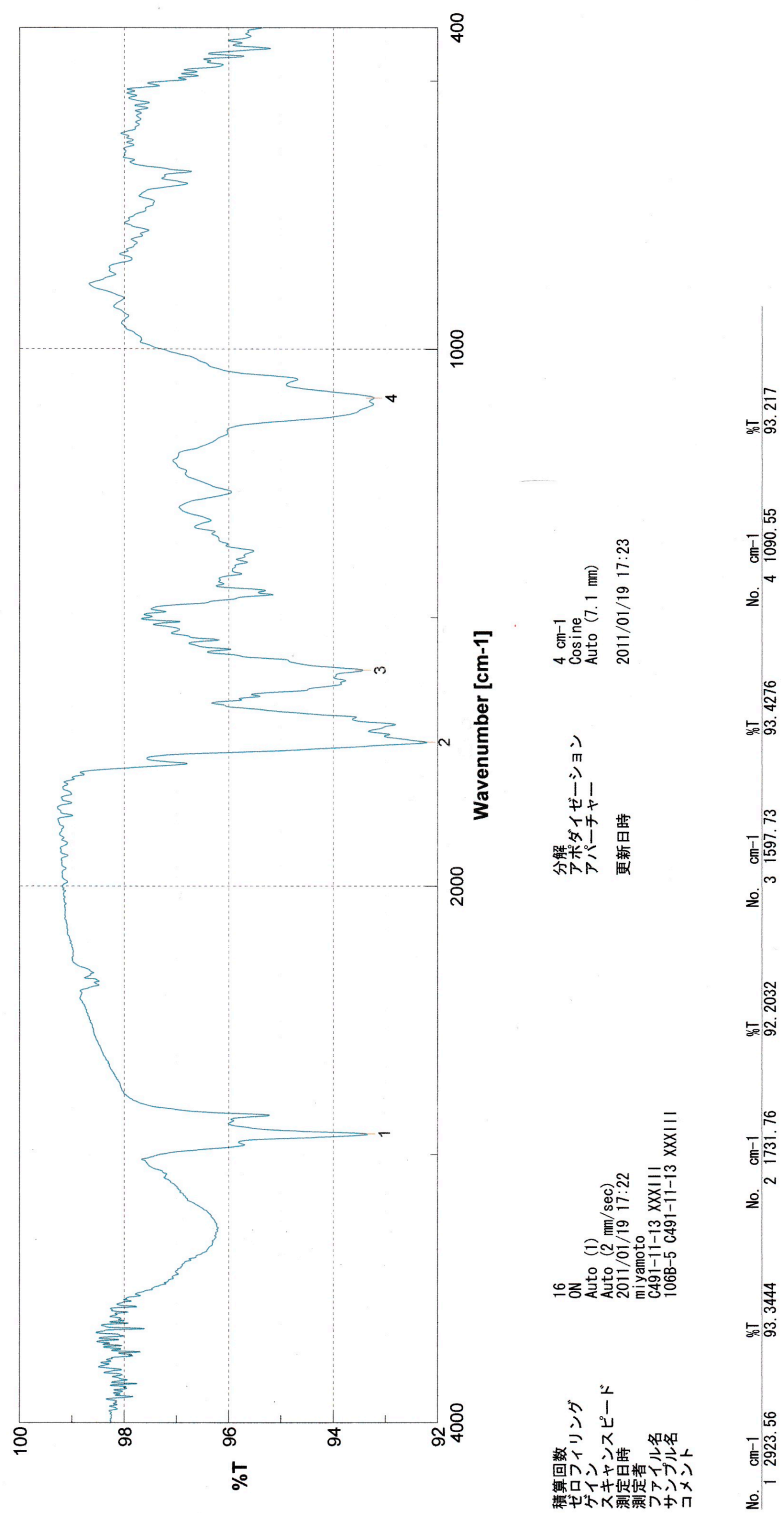


Figure S14 FABMS of 2

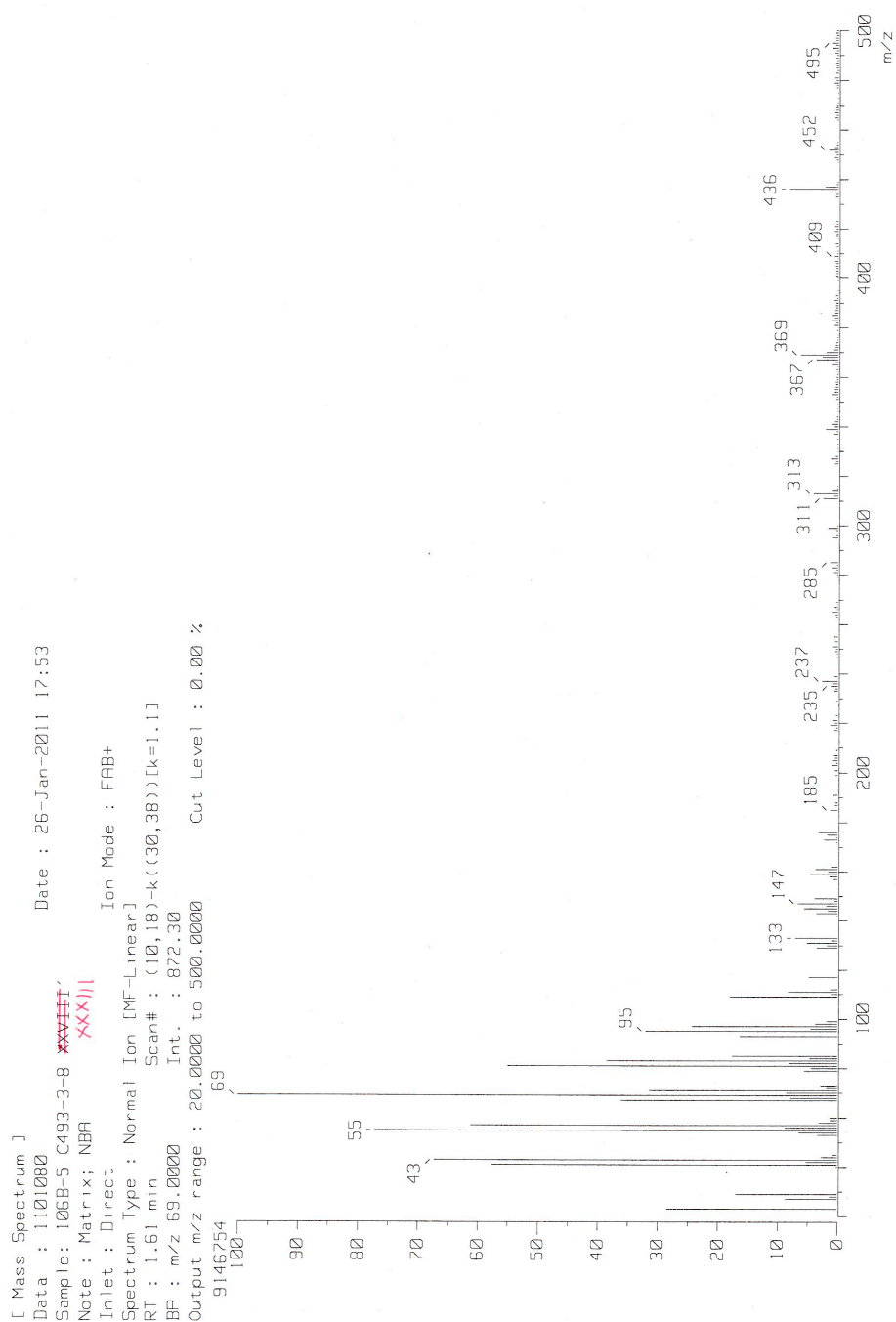


Figure S15 ^1H and ^{13}C NMR spectra of **3** in CDCl_3

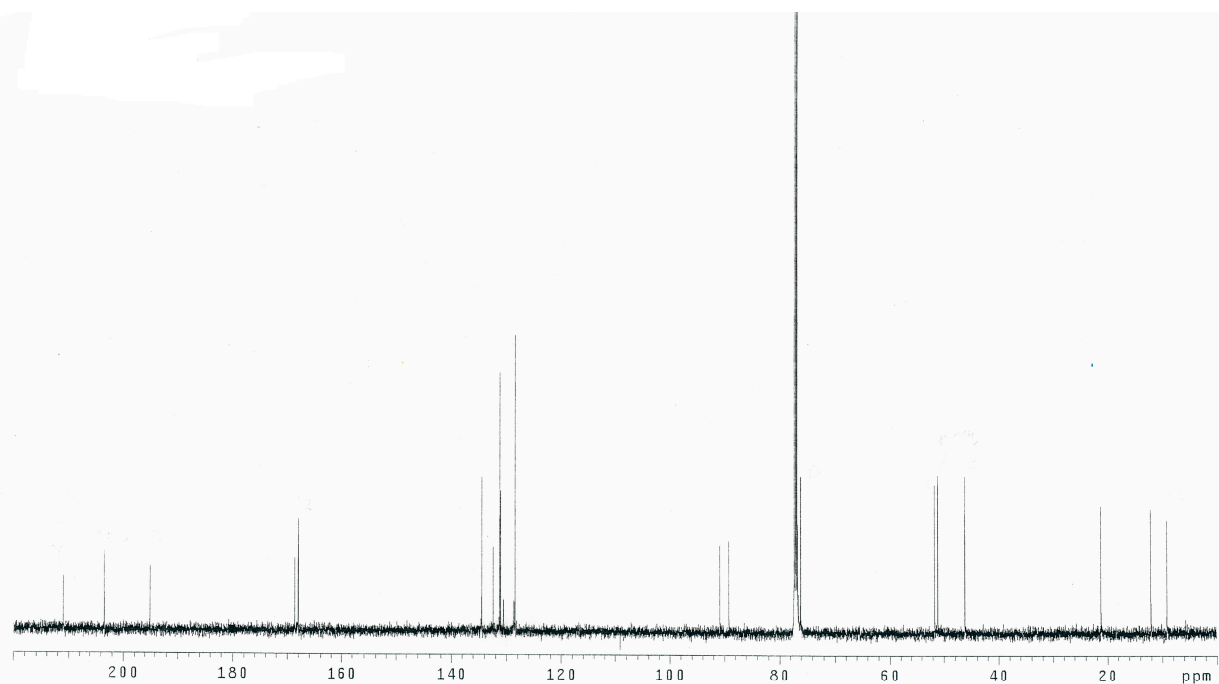
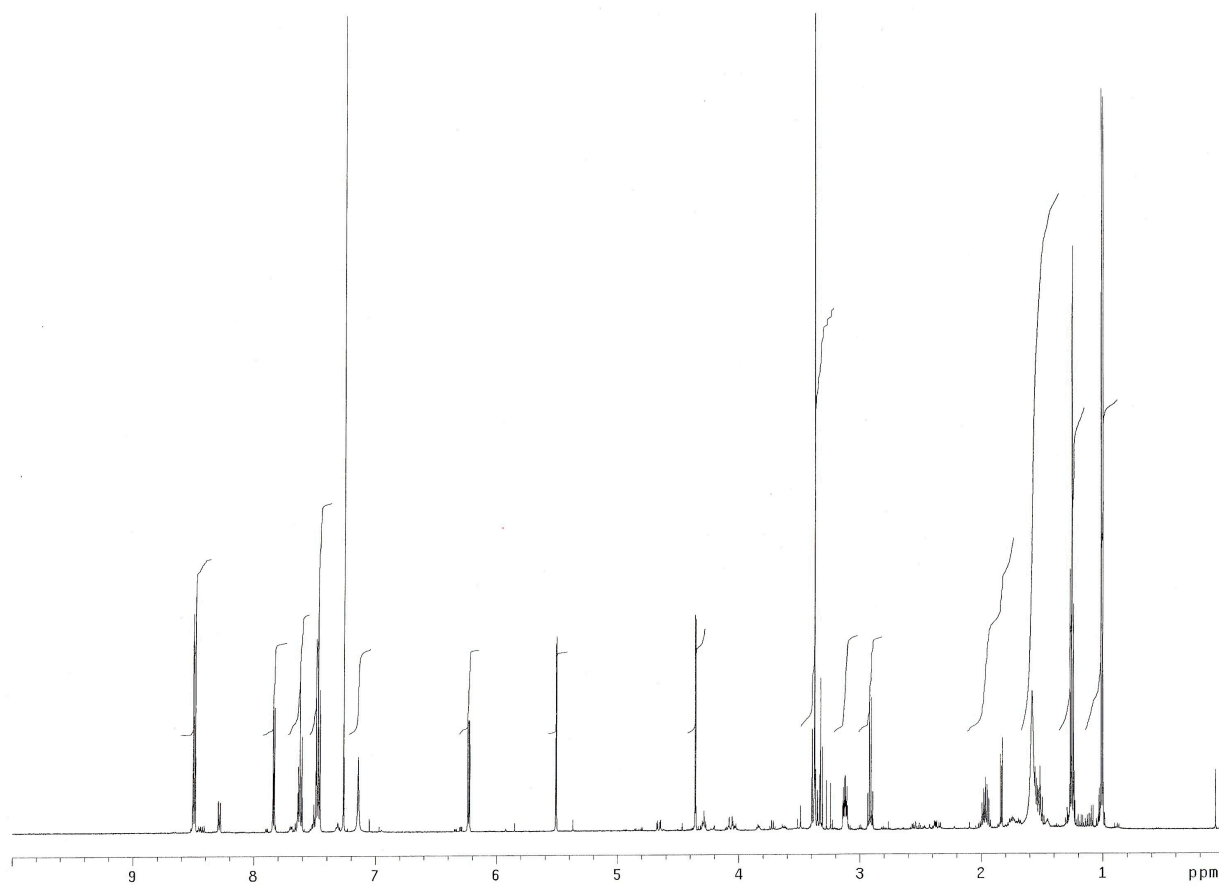
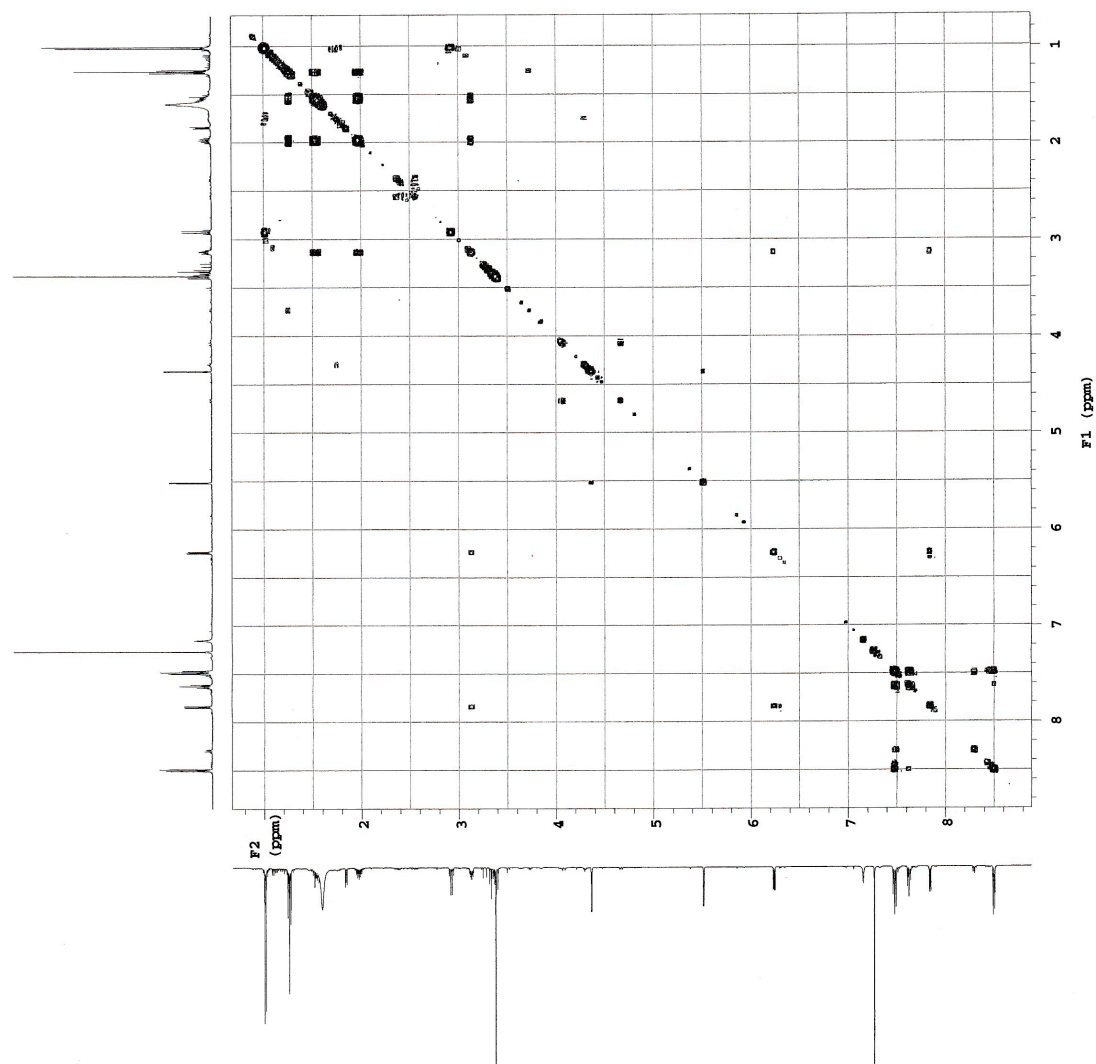


Figure S16 ^1H - ^1H COSY of 3

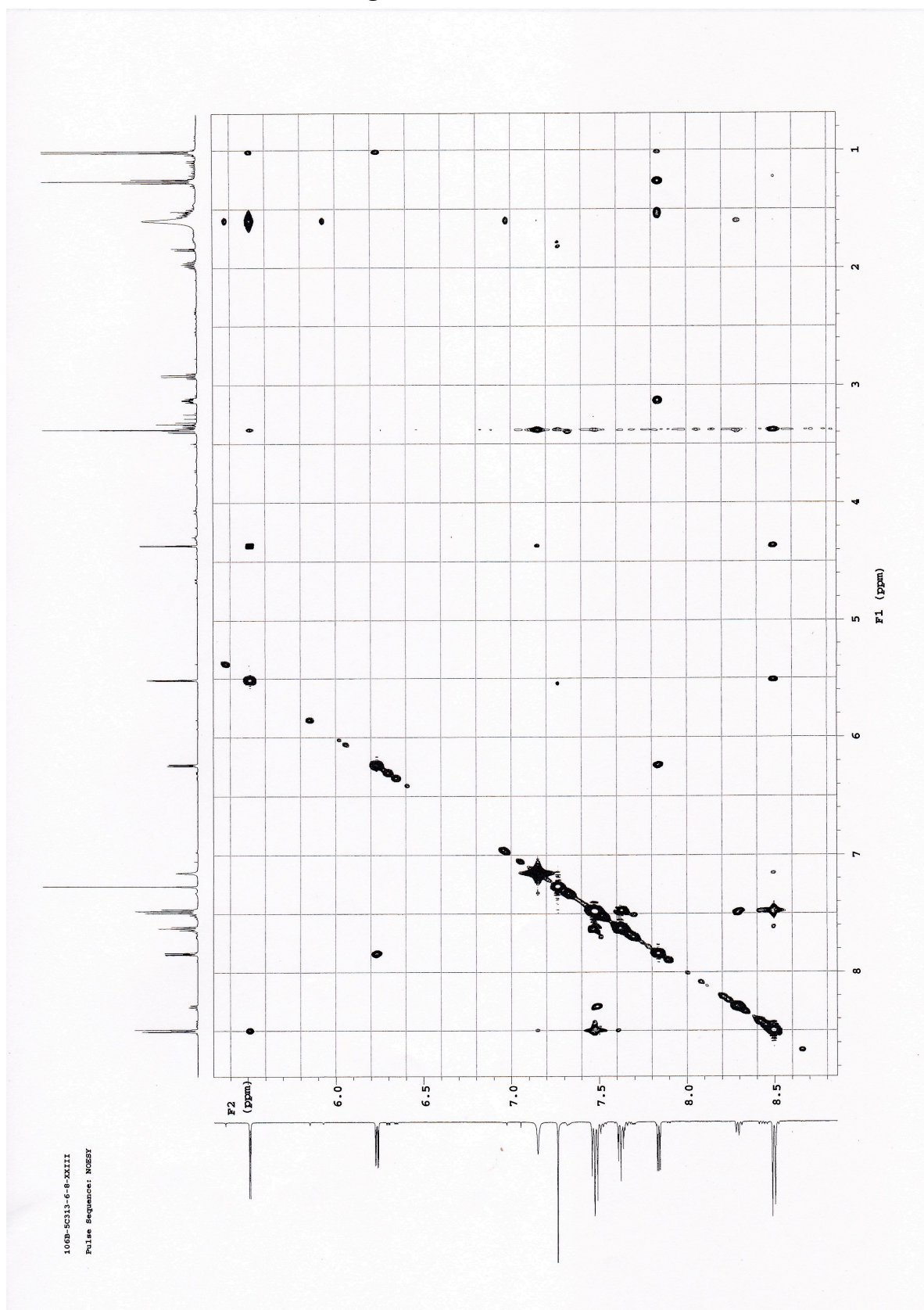


```

106B-EC313-6-8-XXIII
exp33  gCOSY

NAME      FLAGS
date      2009 04 24
solvent   CDCl3
sample    undefined has1v1
acq1      SPECIAL
  at       4576.4 temp not used
  ap       2048 spin not used
  zp       3000 F2 PROCESSING
  d1       1.000 dhs not used
  nt       16 F1
  2D ACQUISITION F1 PROCESSING
  ni       4576.4 ab1 -0.028
  ni       256 ab1 not used
  lp       1p
  TRANSMITTER HI proc1
  ta       499.594 F1
  tof      -289.1 F1 DISPLAY
  tpr      53 sp 4102.7
  pw       9.500 sp1 335.7
  GRADIENTS
  gtlv11   4414 rf1 4107.1
  gtl      0.001000 rf1 62.1
  gtl      0.000500 rf1 62.0
  ch       DECOUPLER HI rf1 0
  ch       min wc 200.0
  ch       wc2 200.0
  ch       ac2 0
  ch       va 236
  ch       en 4
  ch       cdc av 4
  
```

Figure S17 NOESY of 3



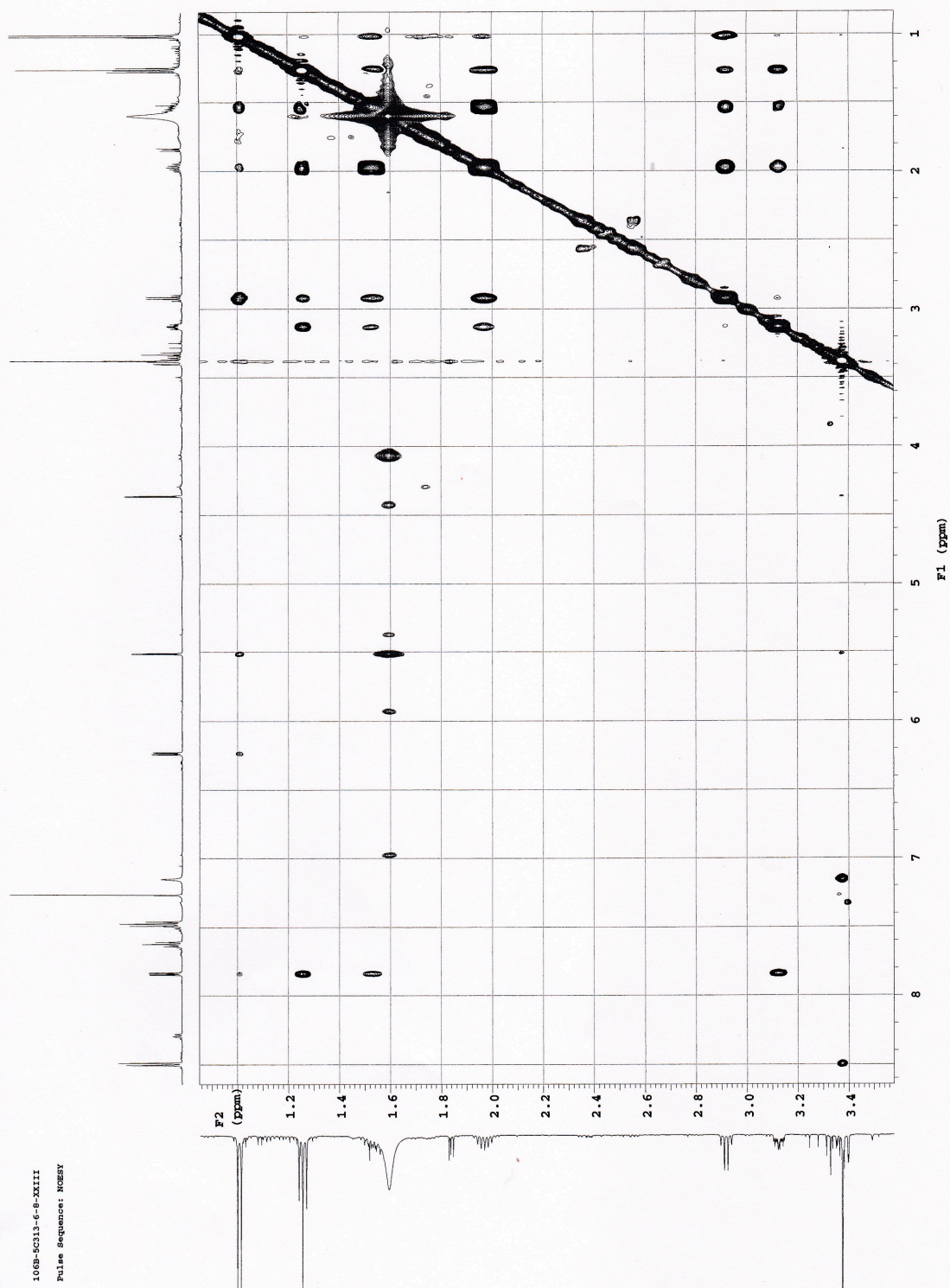


Figure S18 HMQC of **3**

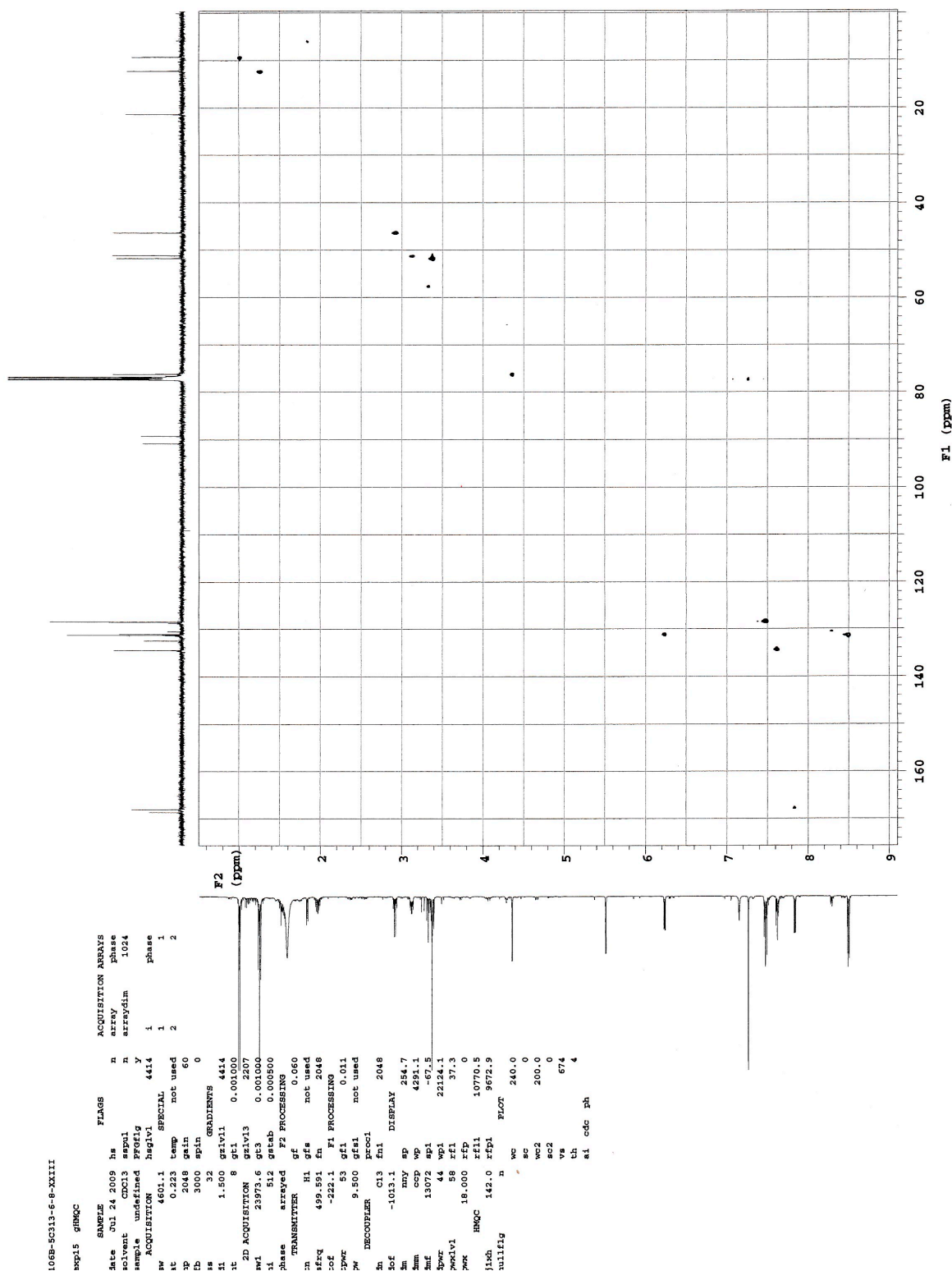
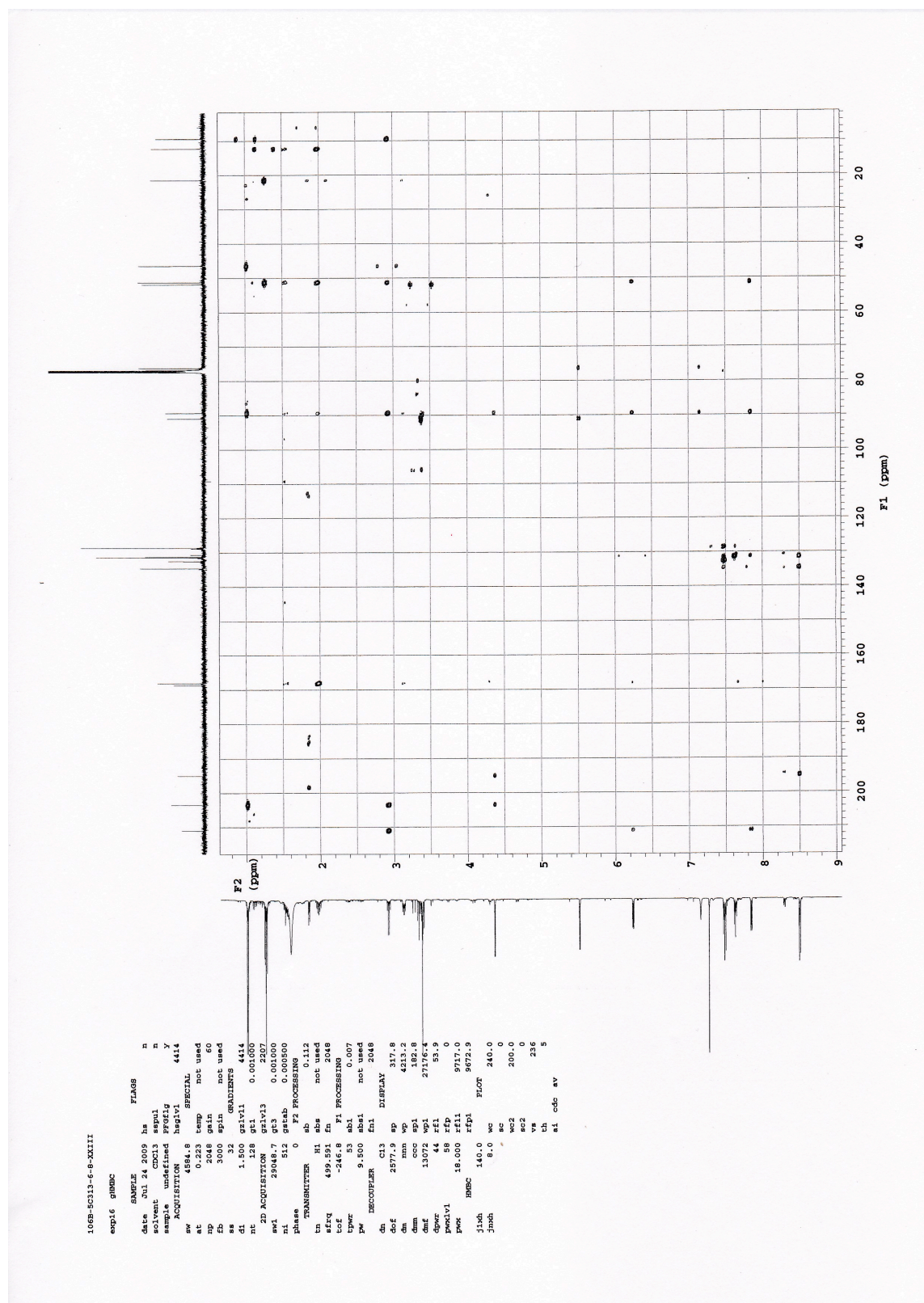


Figure S19 HMBC of 3



32

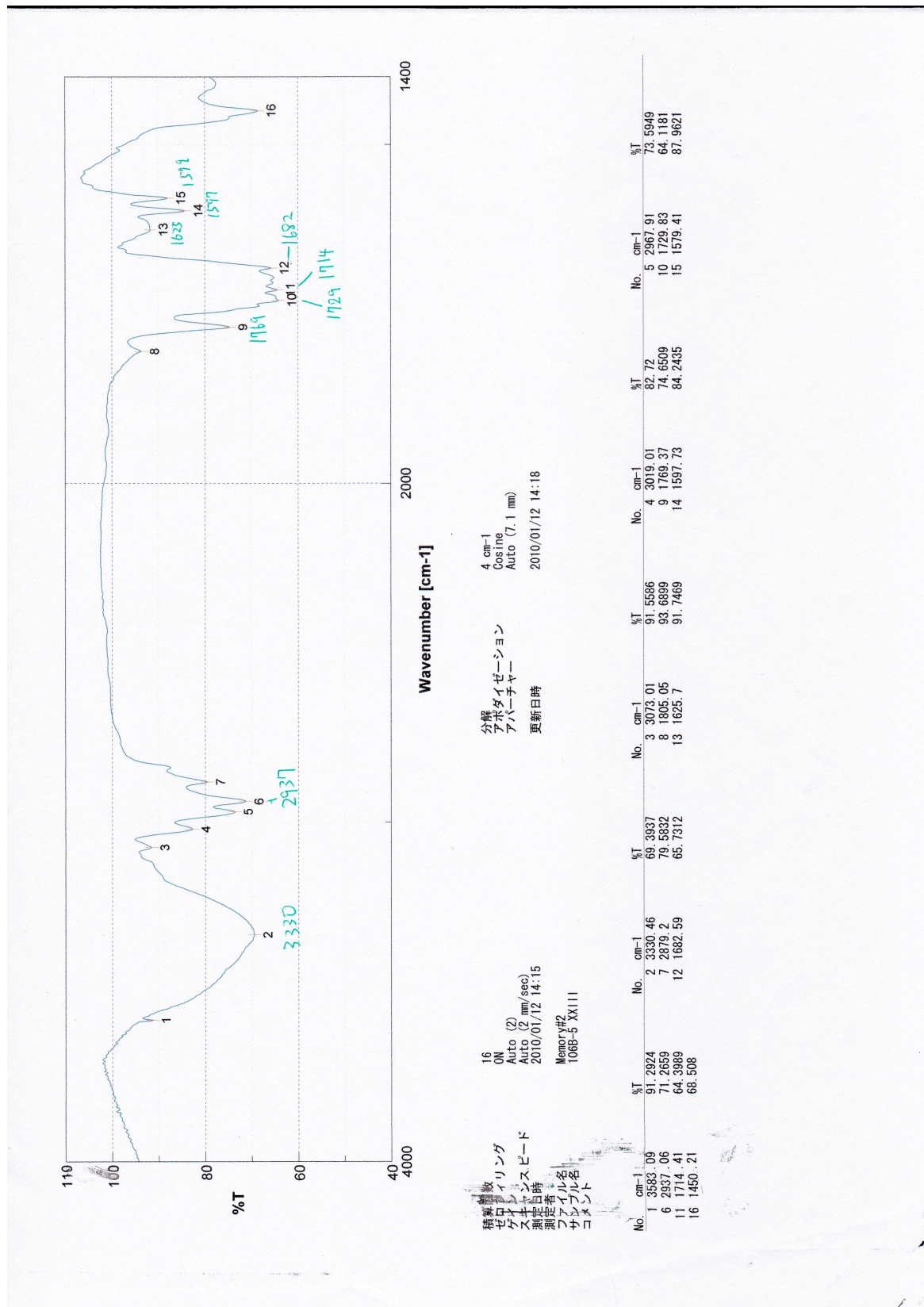


Figure S21 FABMS of 3

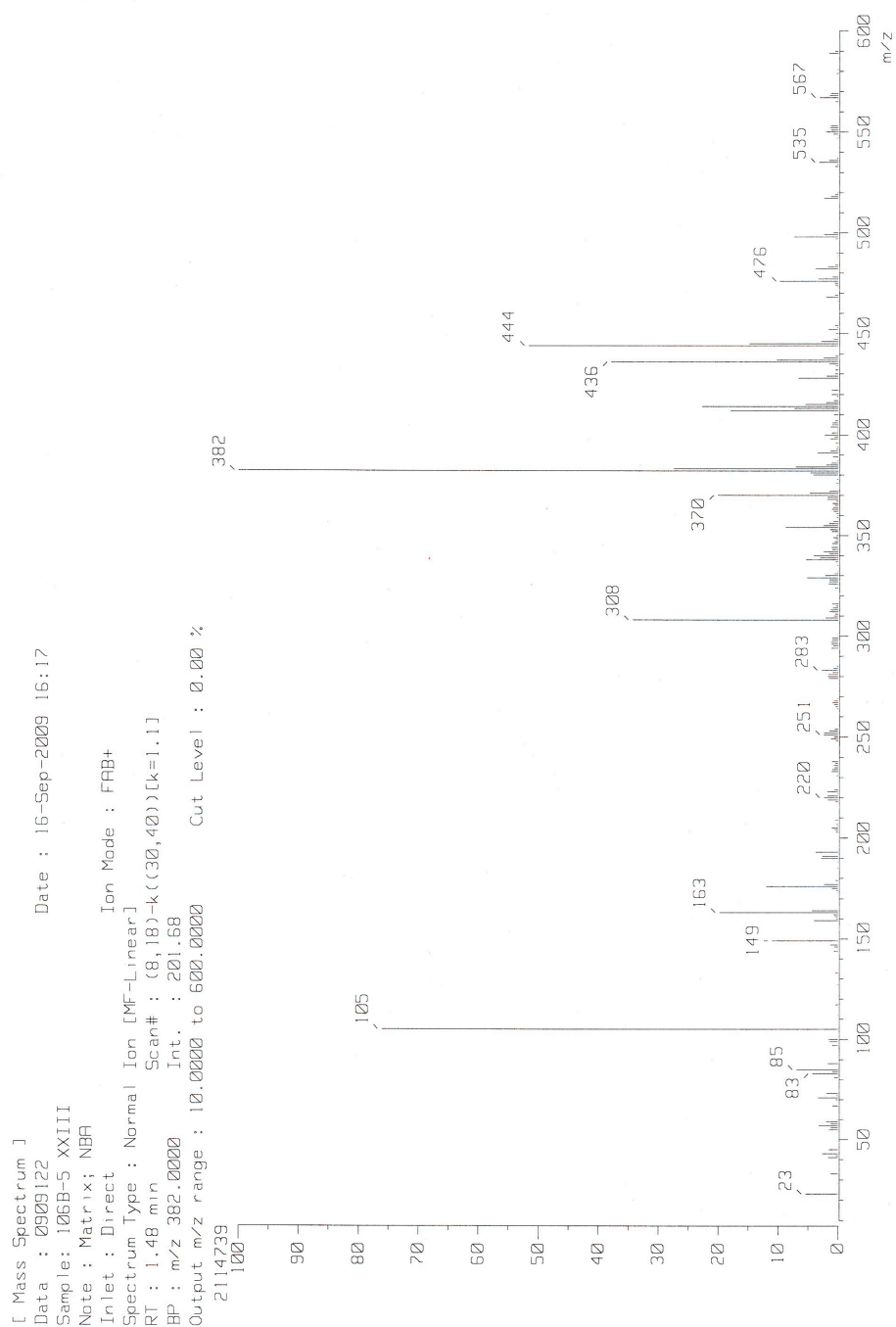


Figure S22 ^1H and ^{13}C NMR spectra of **4** in CDCl_3

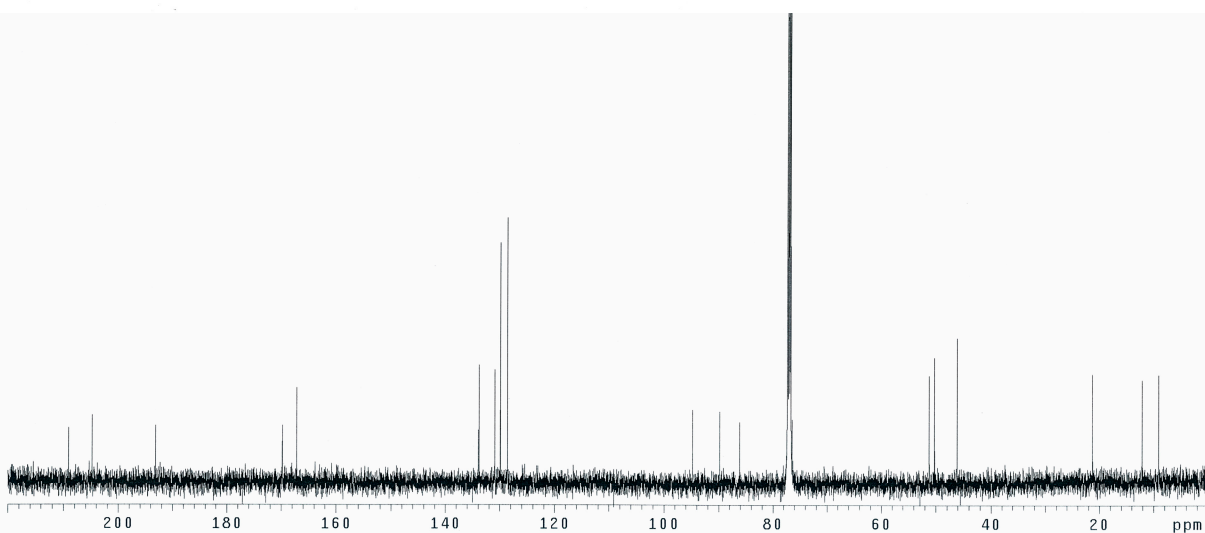
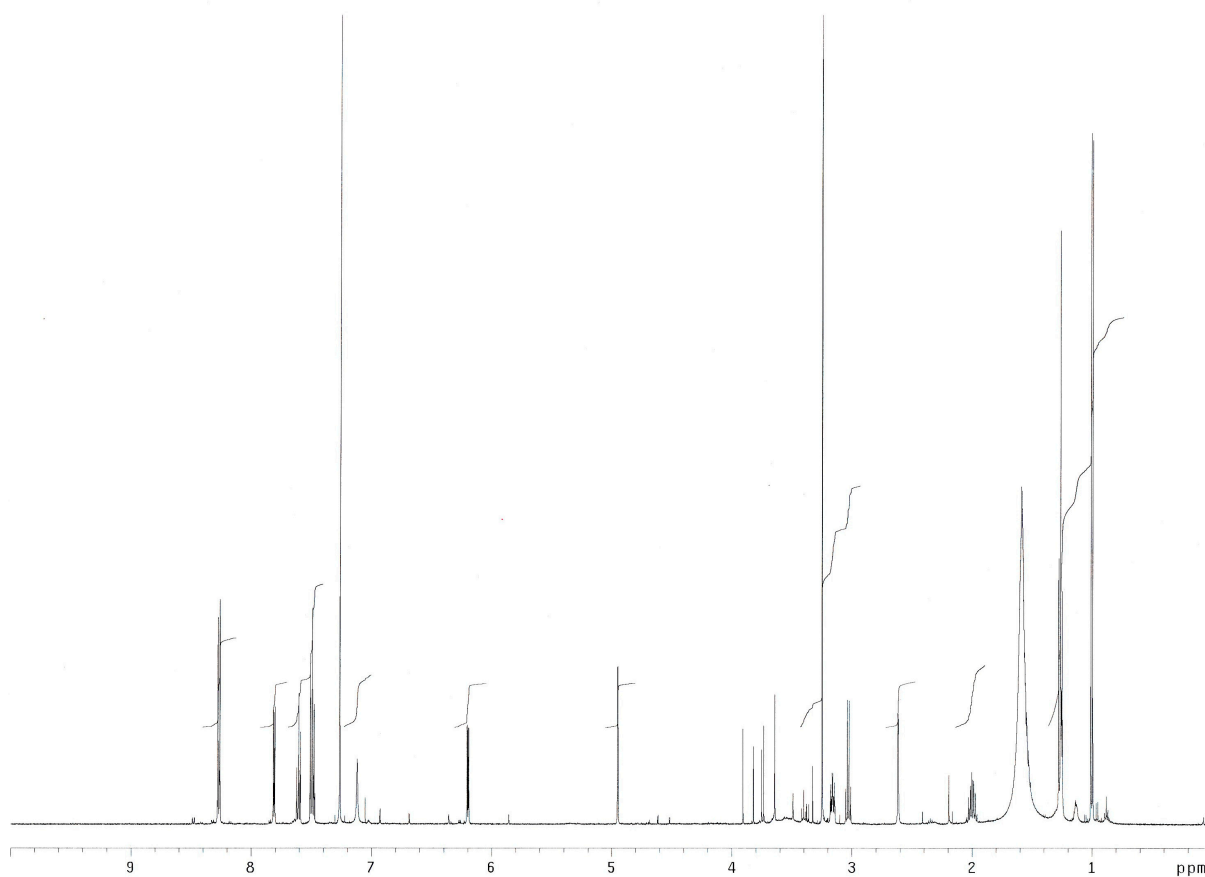
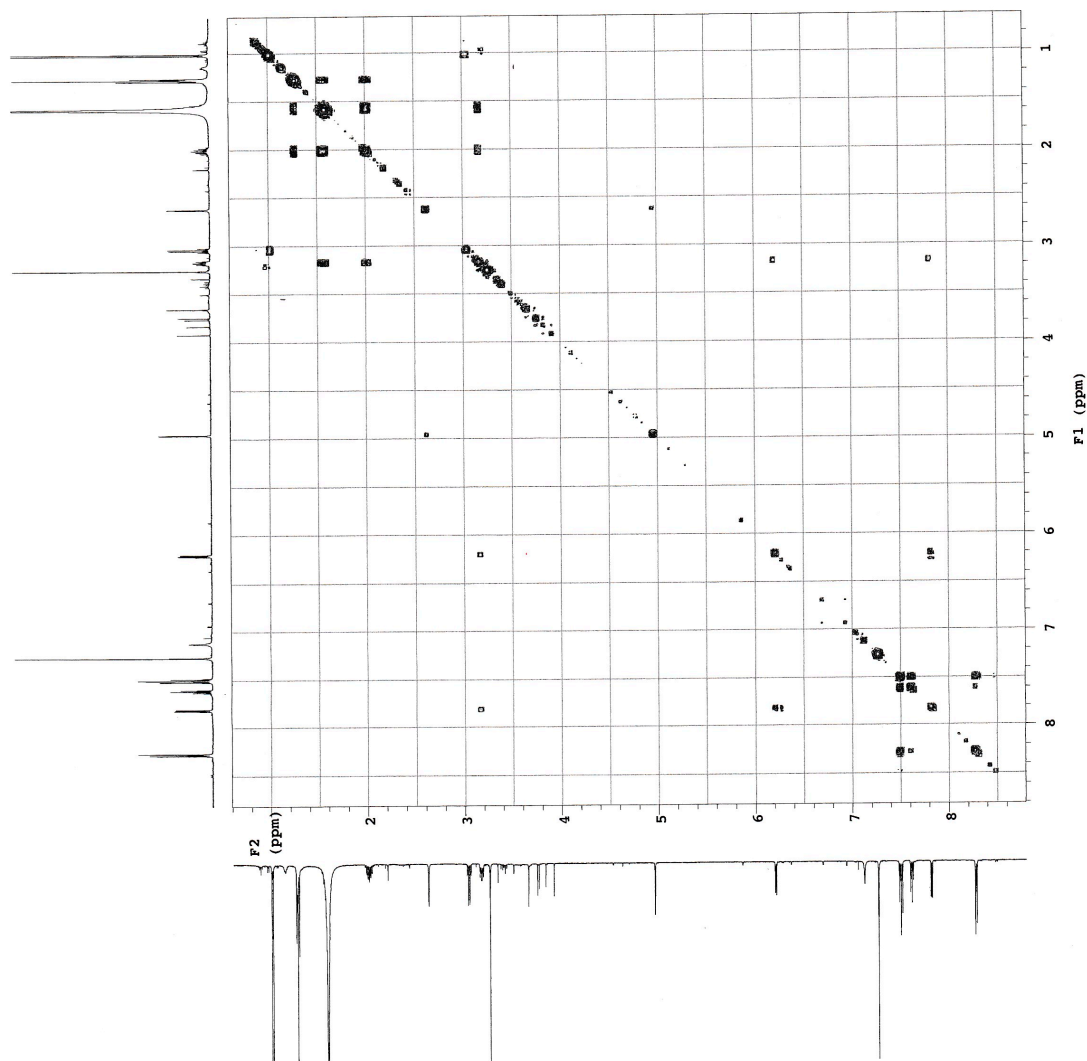


Figure S23 ^1H - ^1H COSY of 4



1069-5 XIII-metha
exp23 gcosy

NAME
date Aug 21 2010 ha nn
solvent CDCl3 sspul n
sample undefined hsgl1 414
ACQUISITION SPECIAL
sw 400.13 temp not used
ns 3206 sspul 58
sp 2048 sspul 0
fb 3000 F2 PROCESSING
ss 16 ab -0.113
dl 1.000 abs not used
nt 16 fn 2048
2D ACQUISITION F1 PROCESSING
sw1 400.13 temp not used
sp1 2048 sspul 0
fb1 3000 F2 PROCESSING
ss1 16 ab -0.113
dl1 1.000 abs not used
nt1 16 fn 2048
TRANSMITTER H1 fml 2048
tn 499.568
ffrq -314.7 sp DISPLAY
tof 53 up 4093.7
tpwr 9.500 sp1
pw GRADIENTS 414 fml 4093.7
gt1v11 0.001000 rfp 96.1
gt1 0.001000 rfp 96.1
gstab 0.000500 rfp1
dc DECOUPLER H1 fml 2048
dm nn wc
scf1 200.0
scf2 200.0
sc2 0
vs 587
th 3
al av

Figure S24 NOESY of 4

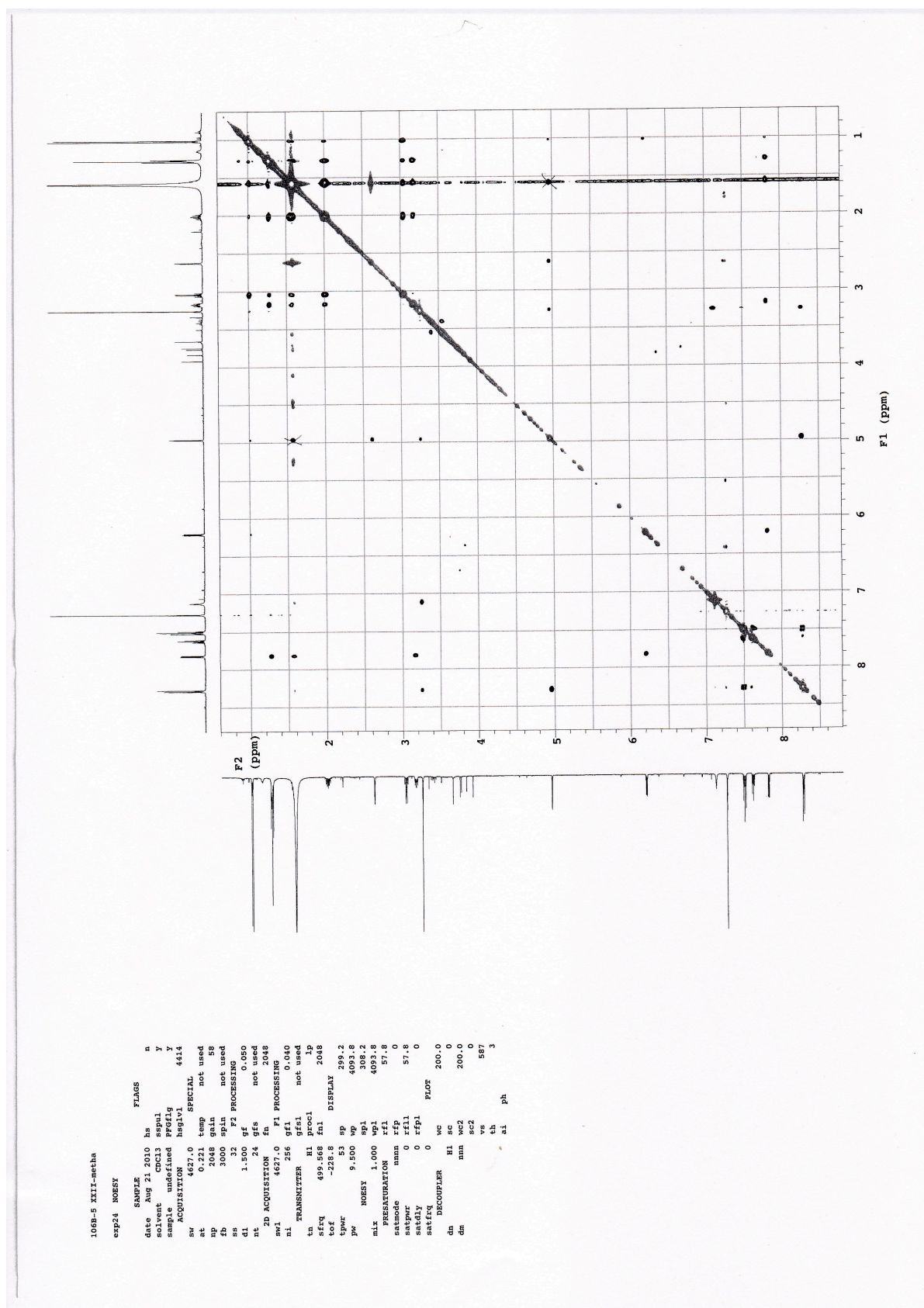


Figure S25 HMQC of 4

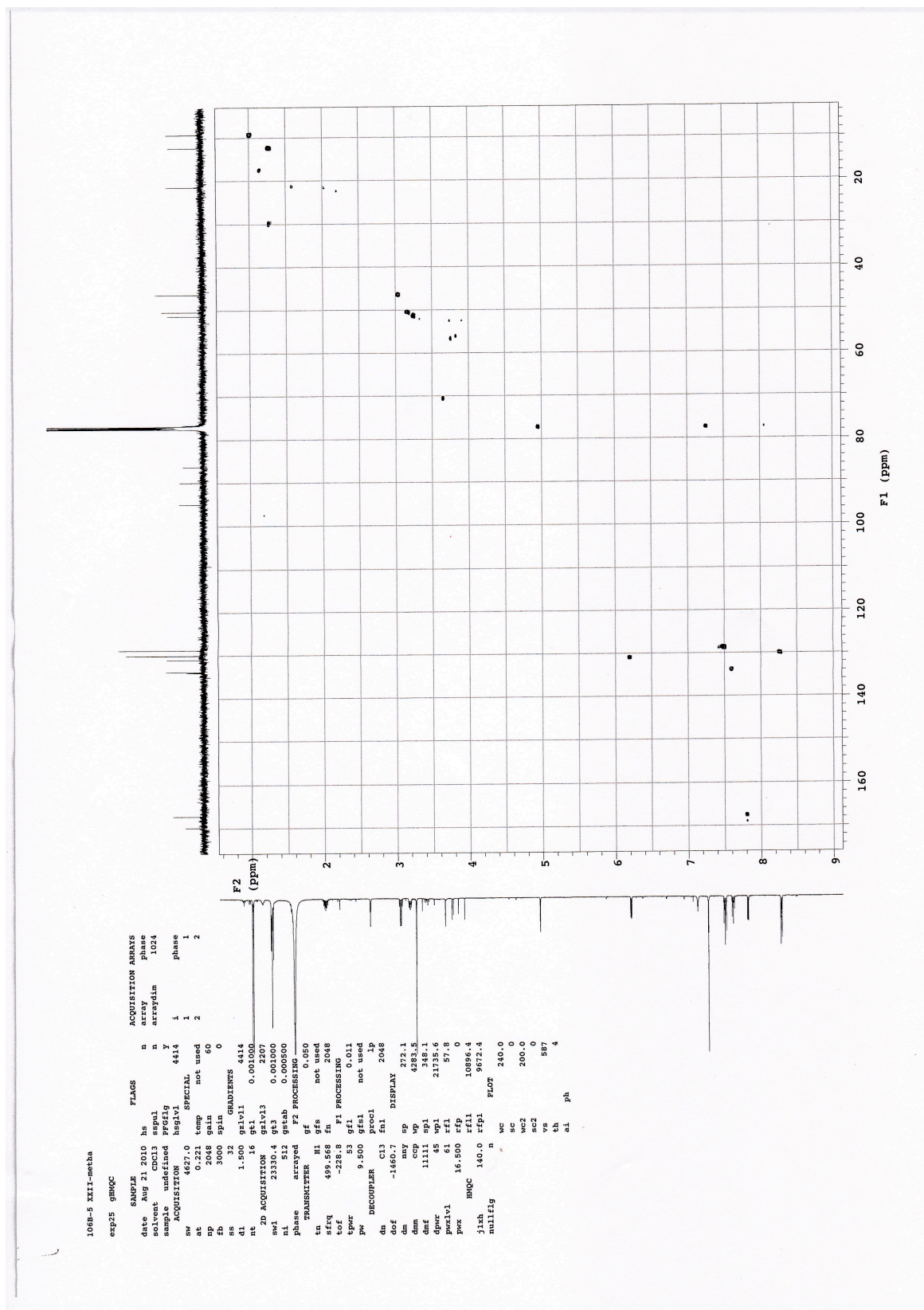


Figure S26 HMBC of 4

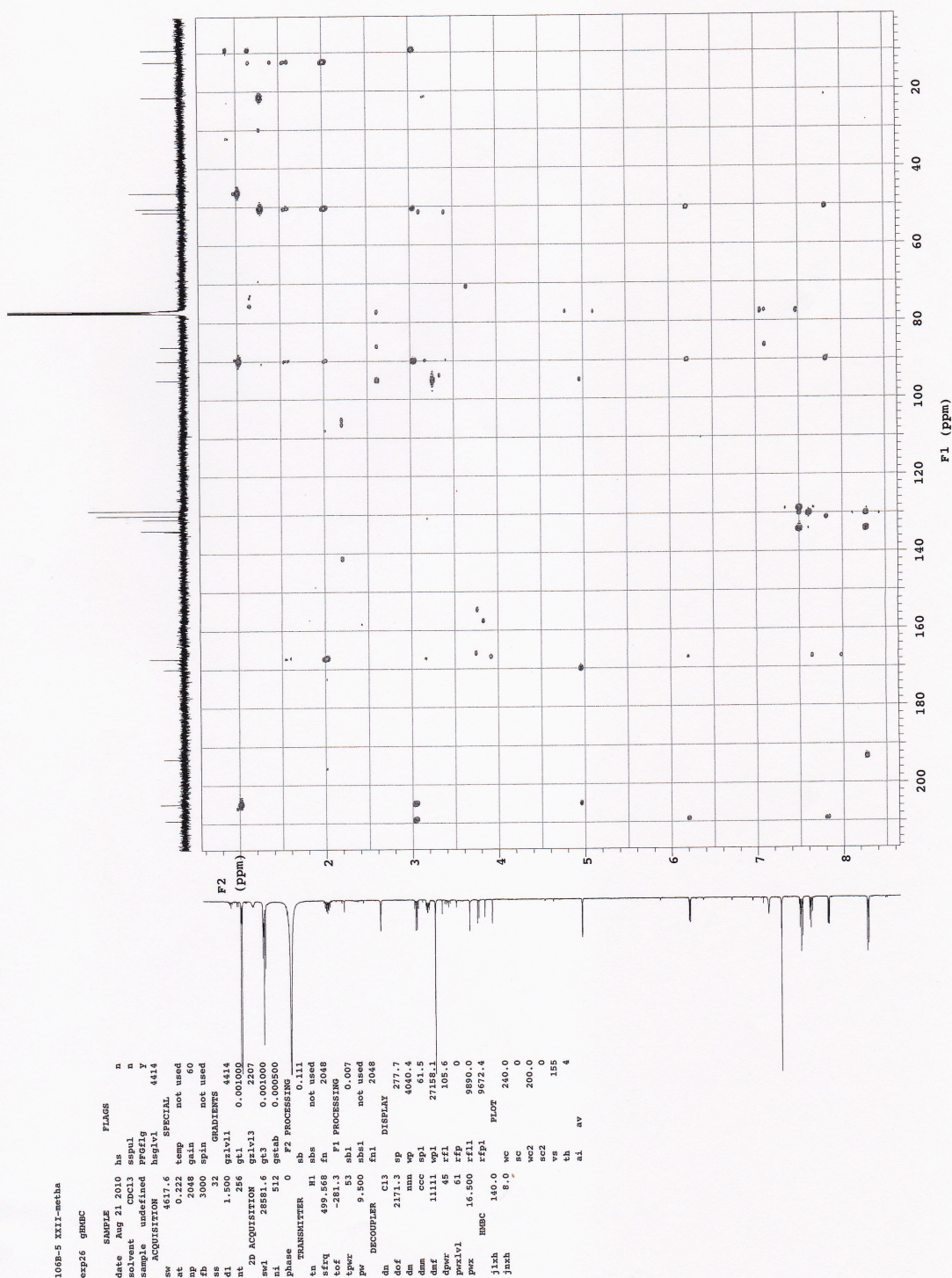


Figure S27 IR spectrum of 4

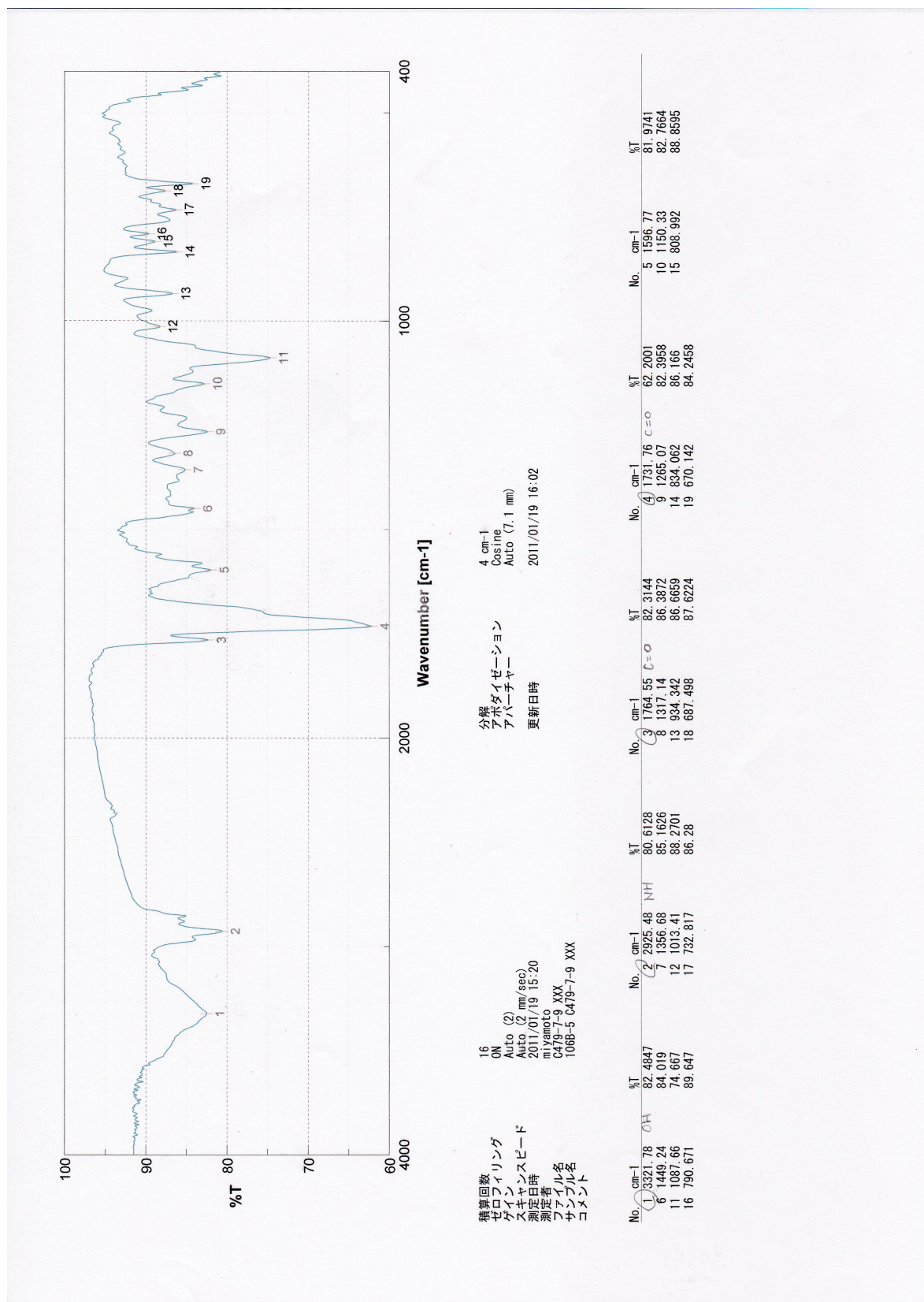


Figure S28 FABMS of 4

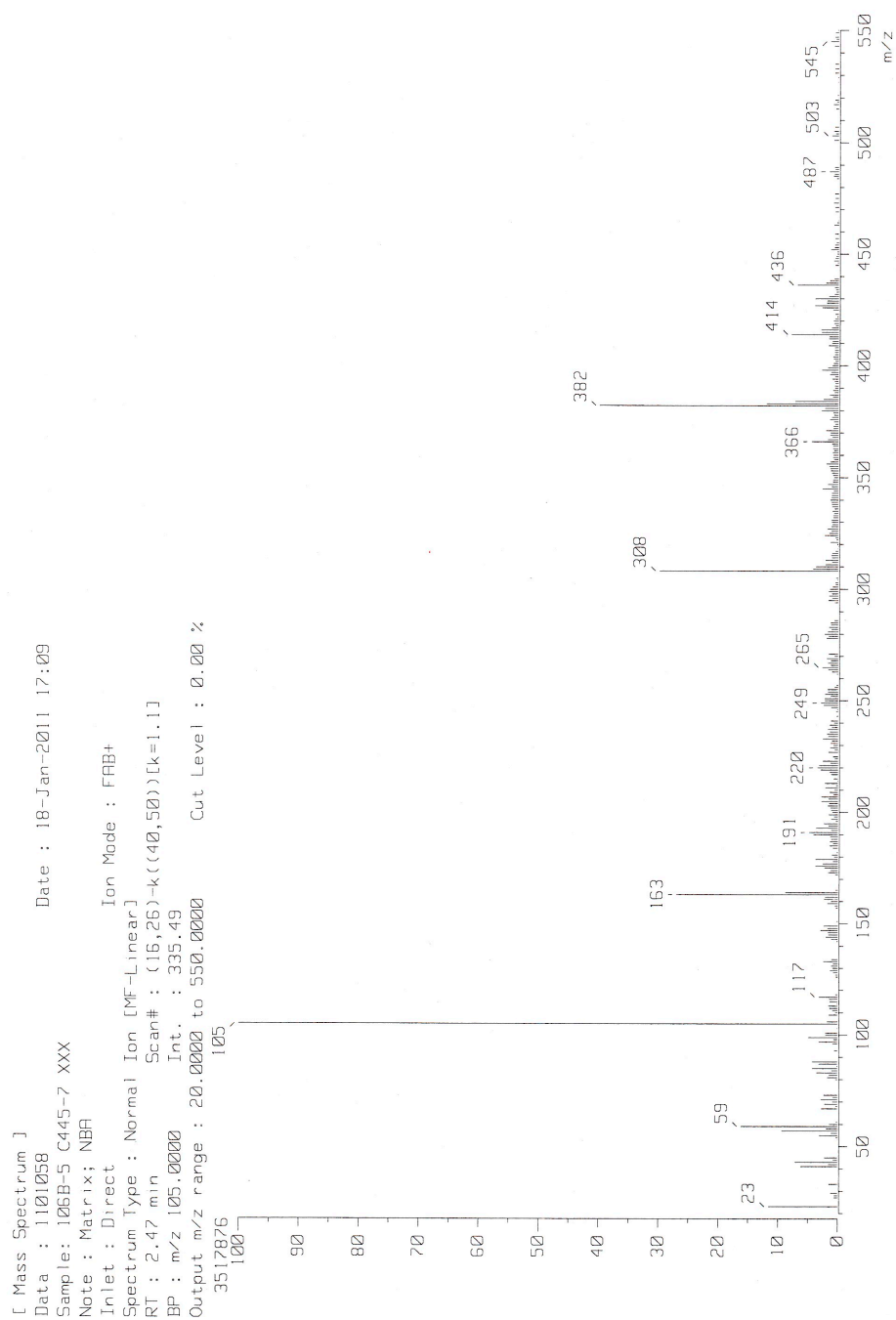
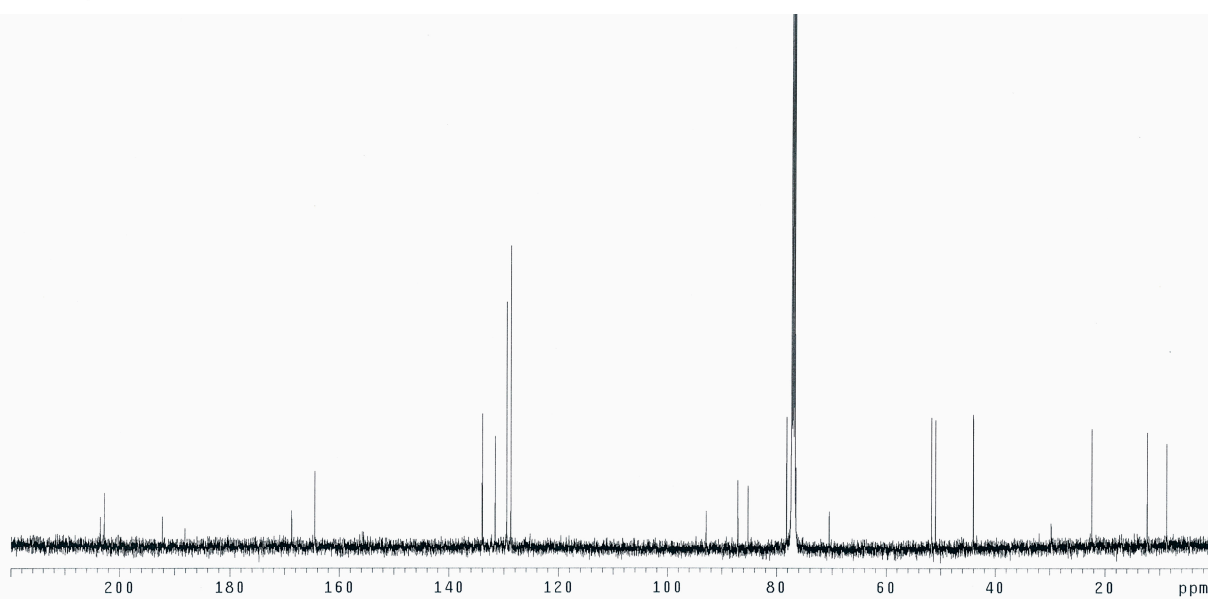
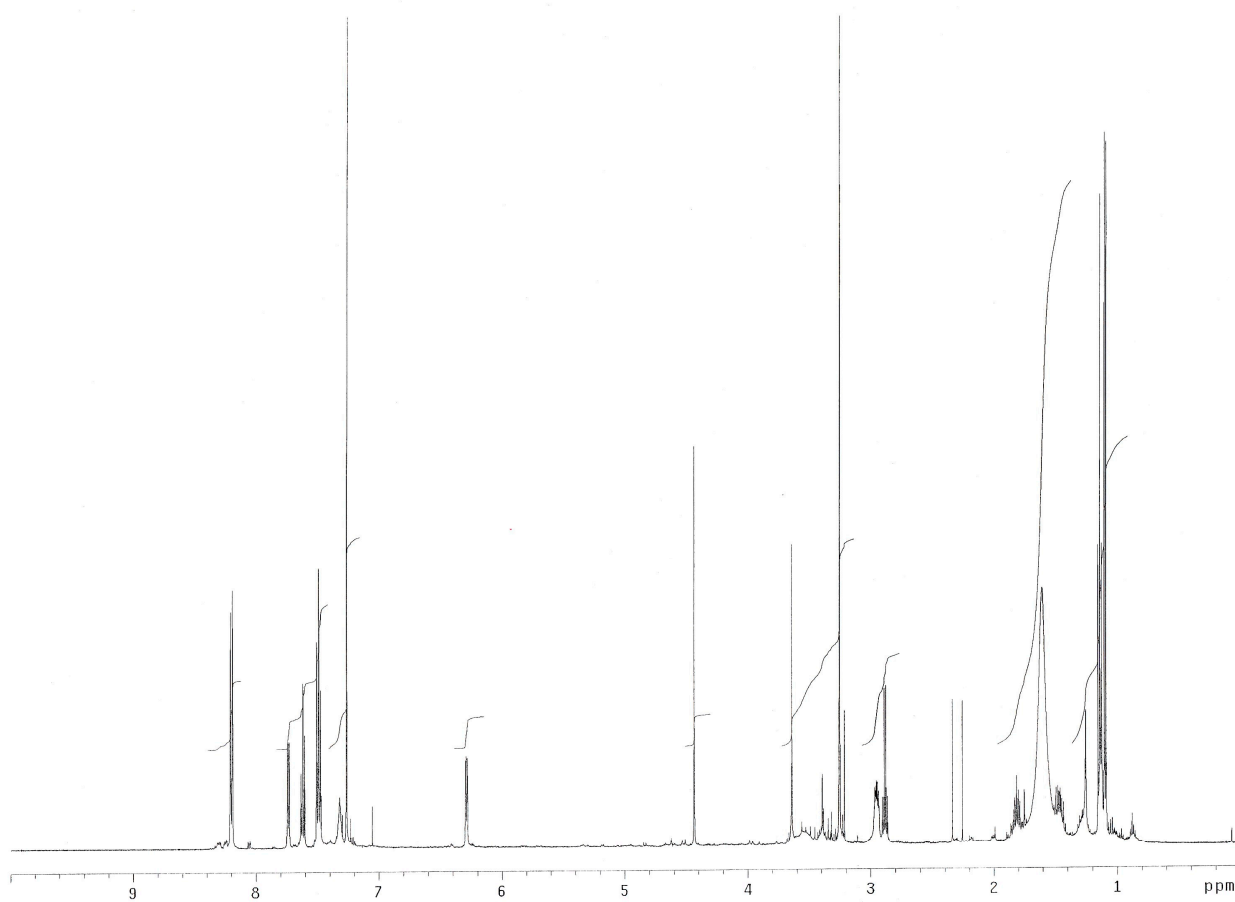
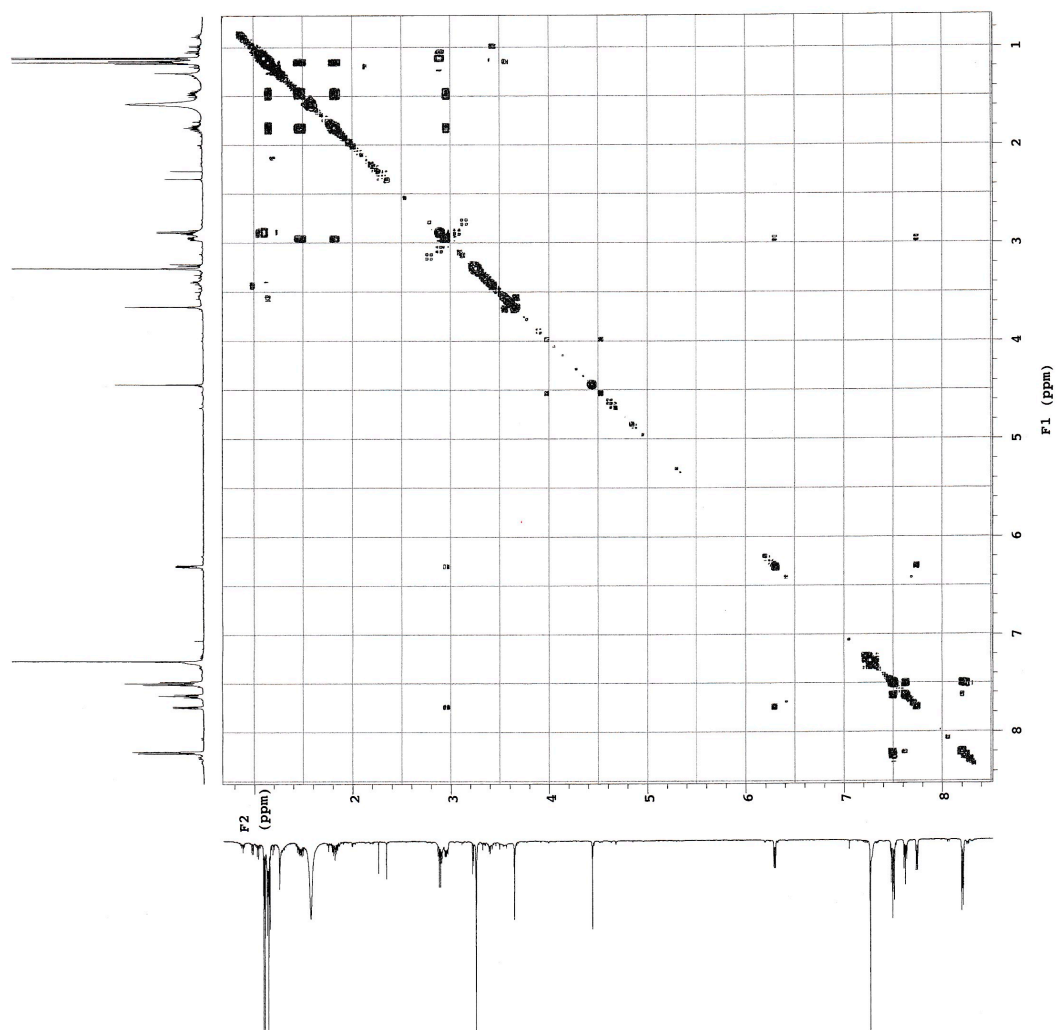


Figure S29 ^1H and ^{13}C NMR spectra of **5** in CDCl_3





exp33 gc031		SAMPLE		FLAGS	
date	Nov 19 2010	ha			nn
time	12:00:00	h1			nn
sample	undefined	bag1v1			4414
ACQUISITION					
rw	4472.0 temp				
ns	0.229 gain				58
up	2048				not used
sp	2048				not used
pr	2048				not used
ab	16				-0.114
dt	1.000	abs			not used
td	16	fn			2048
2D ACQUISITION					
rw	4472.0	ba			-0.029
ns	256	ba			not used
up	256	ba			not used
sp	256	ba			not used
pr	256	ba			not used
ab	16	ba			not used
dt	1.000	ba			not used
td	16	ba			not used
1D TRANSMITTER					
rw	4472.0	ba			-0.029
ns	256	ba			not used
up	256	ba			not used
sp	256	ba			not used
pr	256	ba			not used
ab	16	ba			not used
dt	1.000	ba			not used
td	16	ba			not used
1D PROCESSING					
rw	4472.0	ba			-0.029
ns	256	ba			not used
up	256	ba			not used
sp	256	ba			not used
pr	256	ba			not used
ab	16	ba			not used
dt	1.000	ba			not used
td	16	ba			not used
1D DISPLAY					
rw	4472.0	ba			-0.029
ns	256	ba			not used
up	256	ba			not used
sp	256	ba			not used
pr	256	ba			not used
ab	16	ba			not used
dt	1.000	ba			not used
td	16	ba			not used
1D TRANSMITTER					
rw	4472.0	ba			-0.029
ns	256	ba			not used
up	256	ba			not used
sp	256	ba			not used
pr	256	ba			not used
ab	16	ba			not used
dt	1.000	ba			not used
td	16	ba			not used
1D PROCESSING					
rw	4472.0	ba			-0.029
ns	256	ba			not used
up	256	ba			not used
sp	256	ba			not used
pr	256	ba			not used
ab	16	ba			not used
dt	1.000	ba			not used
td	16	ba			not used
1D DISPLAY					
rw	4472.0	ba			-0.029
ns	256	ba			not used
up	256	ba			not used
sp	256	ba			not used
pr	256	ba			not used
ab	16	ba			not used
dt	1.000	ba			not used
td	16	ba			not used
1D TRANSMITTER					
rw	4472.0	ba			-0.029
ns	256	ba			not used
up	256	ba			not used
sp	256	ba			not used
pr	256	ba			not used
ab	16	ba			not used
dt	1.000	ba			not used
td	16	ba			not used
1D PROCESSING					
rw	4472.0	ba			-0.029
ns	256	ba			not used</

Figure S31 NOESY of 5

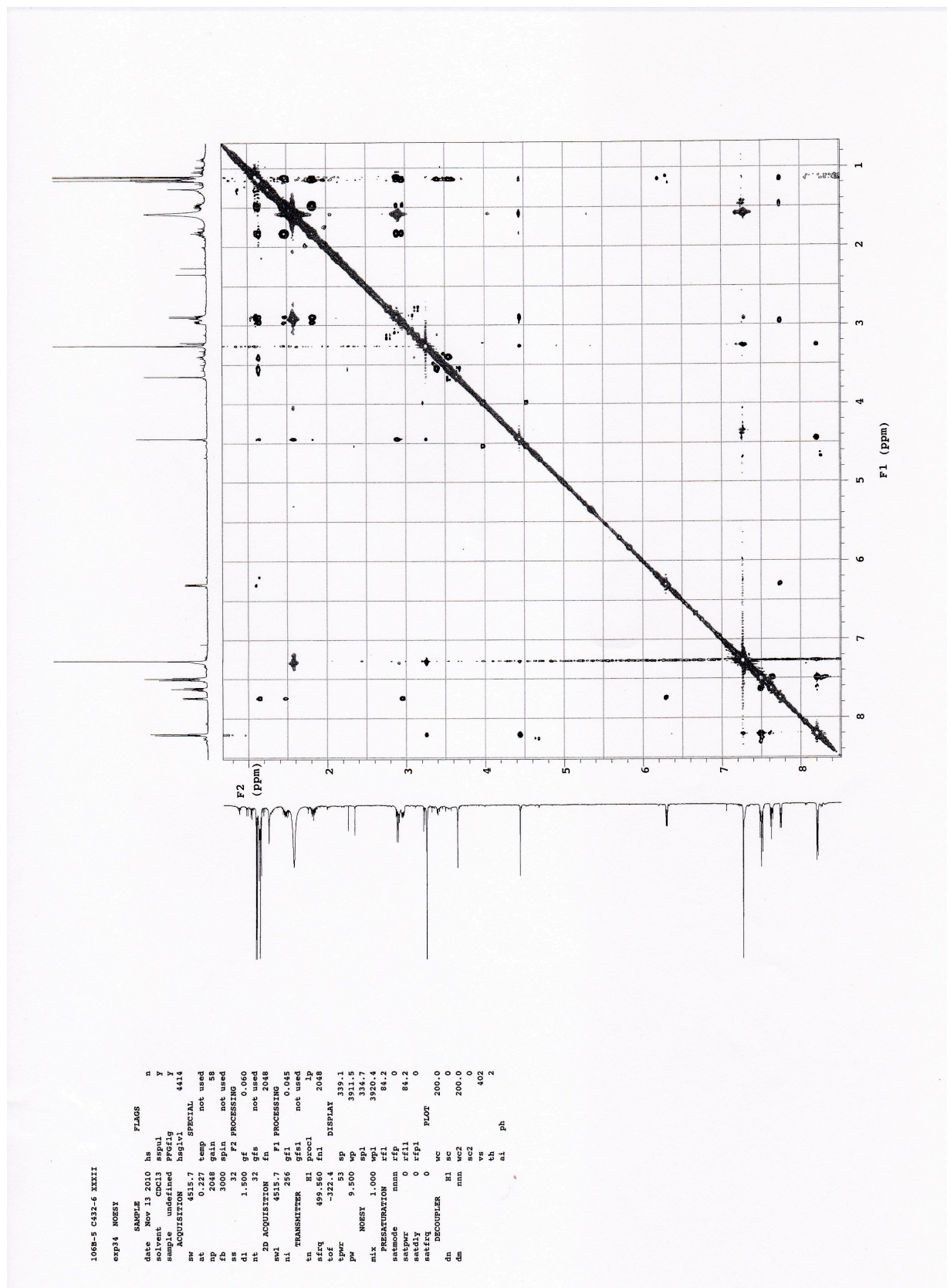


Figure S33 HMBC of 5

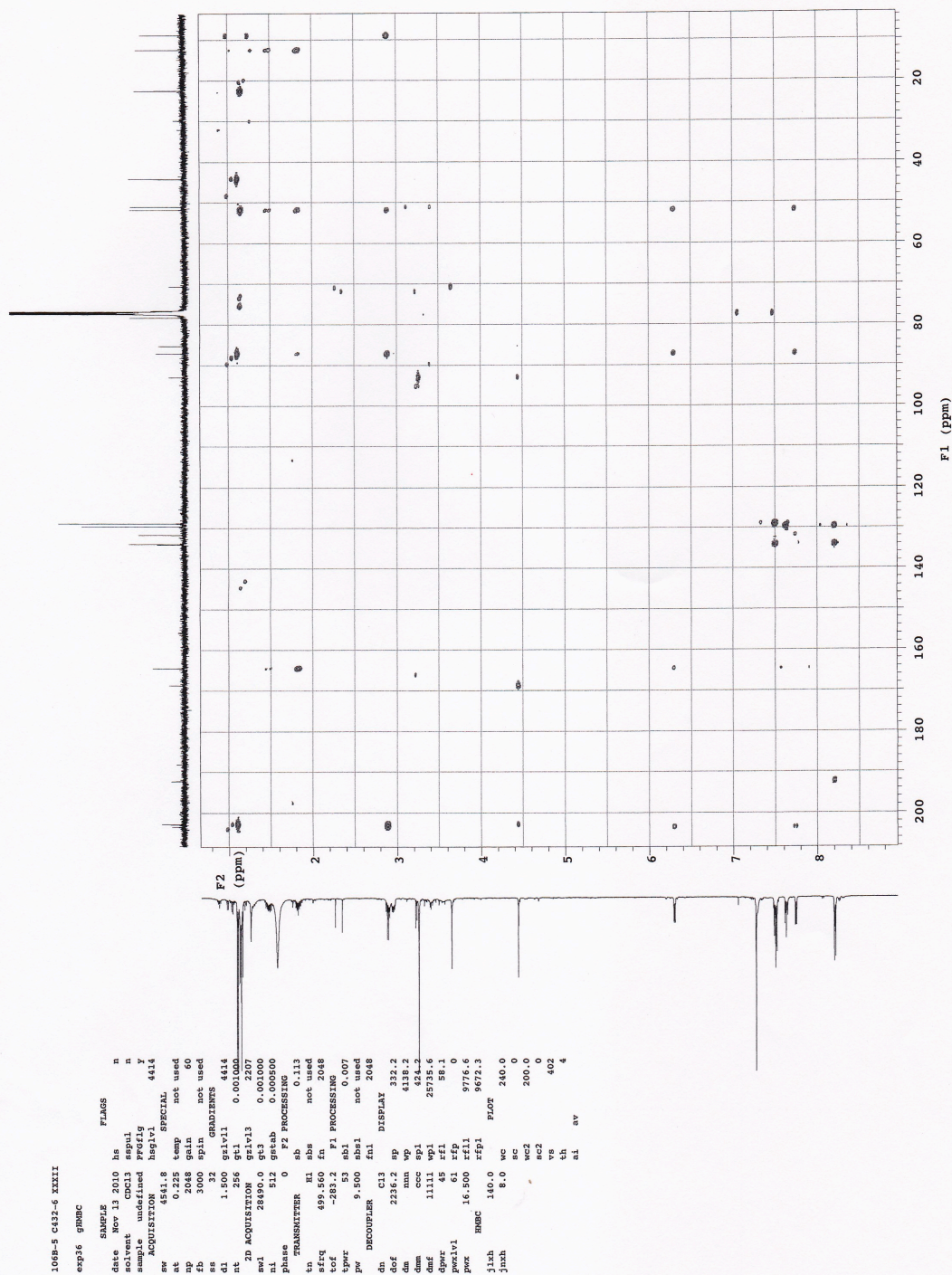


Figure S34 IR spectrum of 5

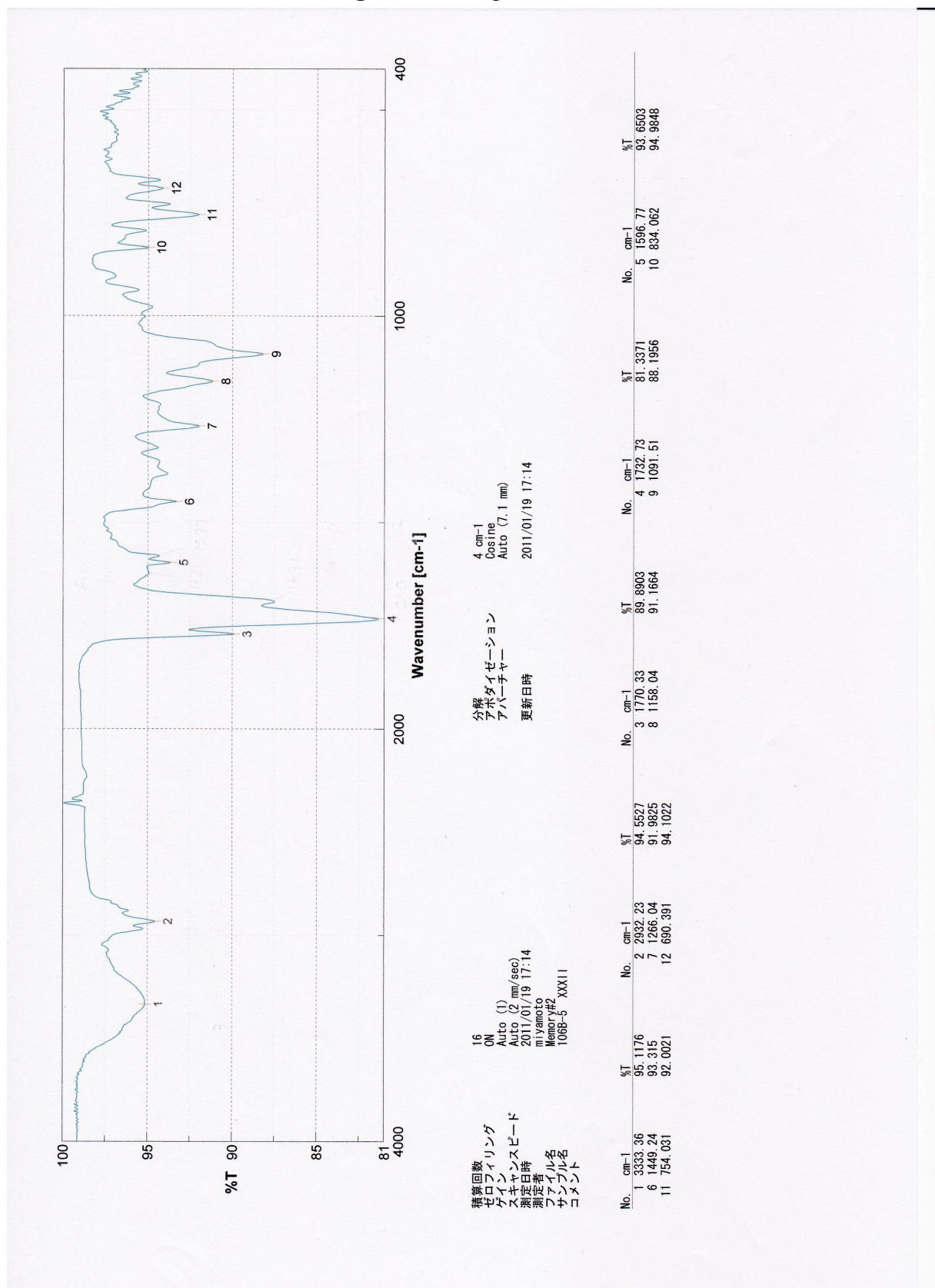


Figure S35 FABMS of 5

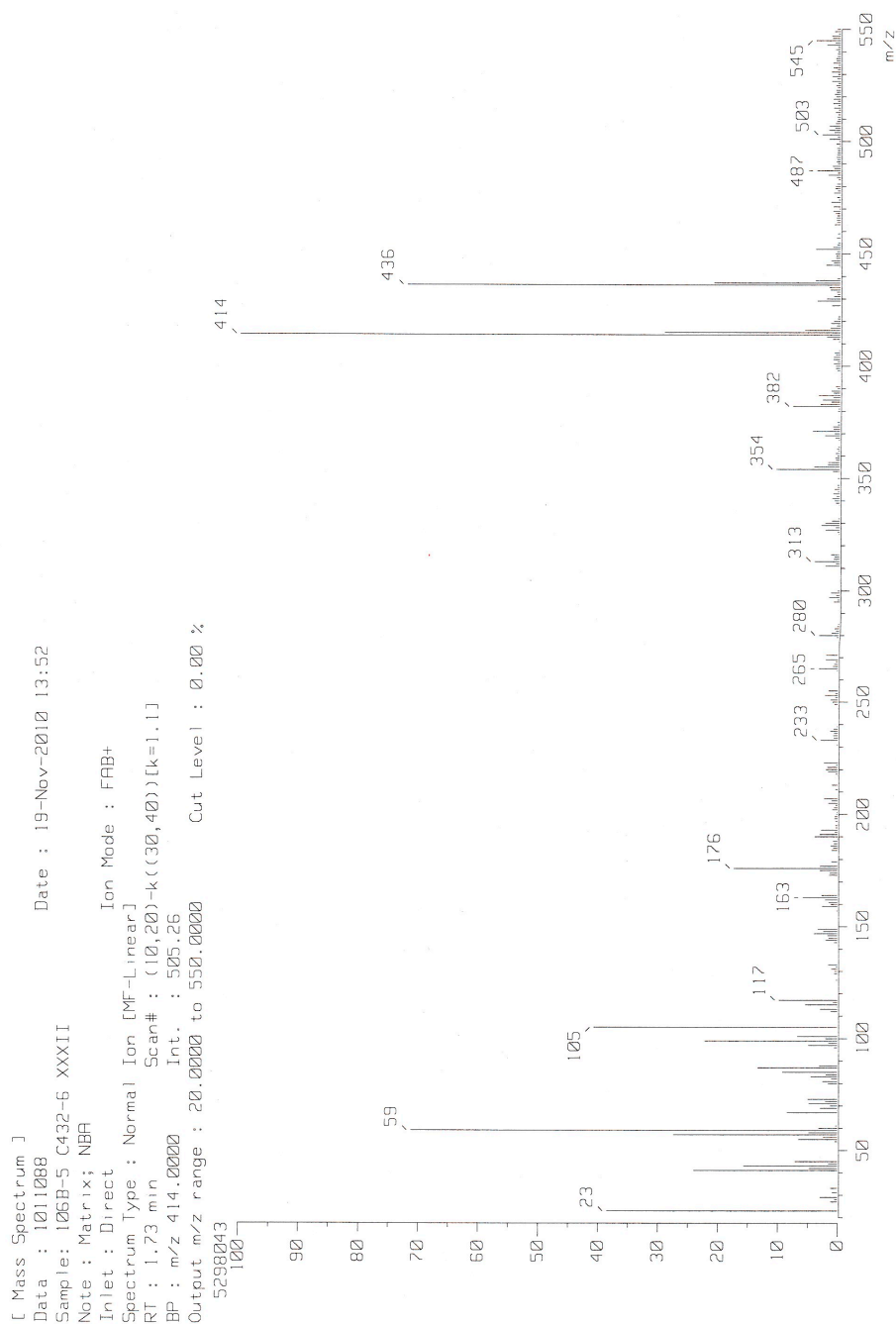


Figure S36 ^1H and ^{13}C NMR spectra of **6** in CDCl_3

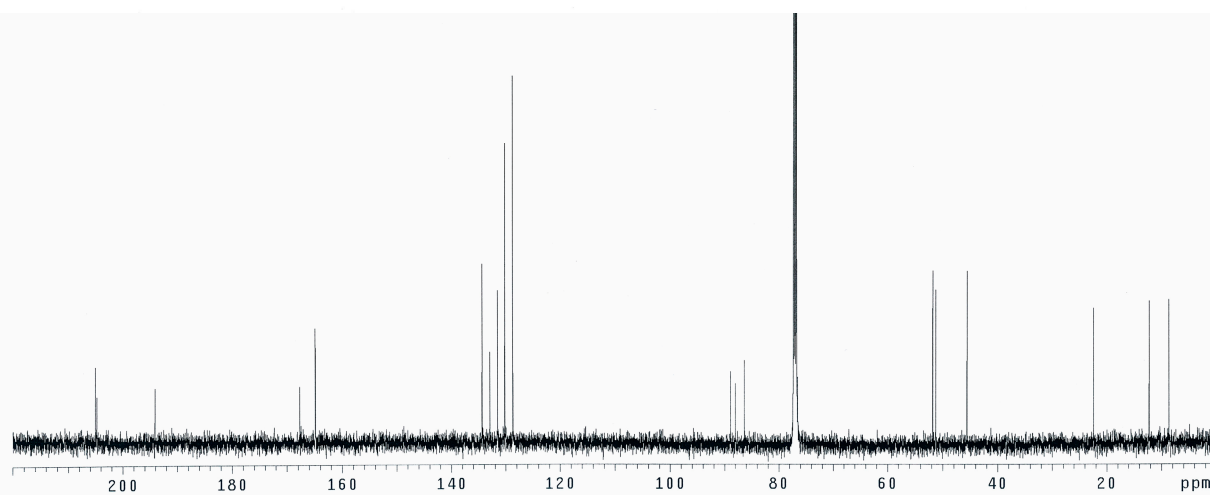
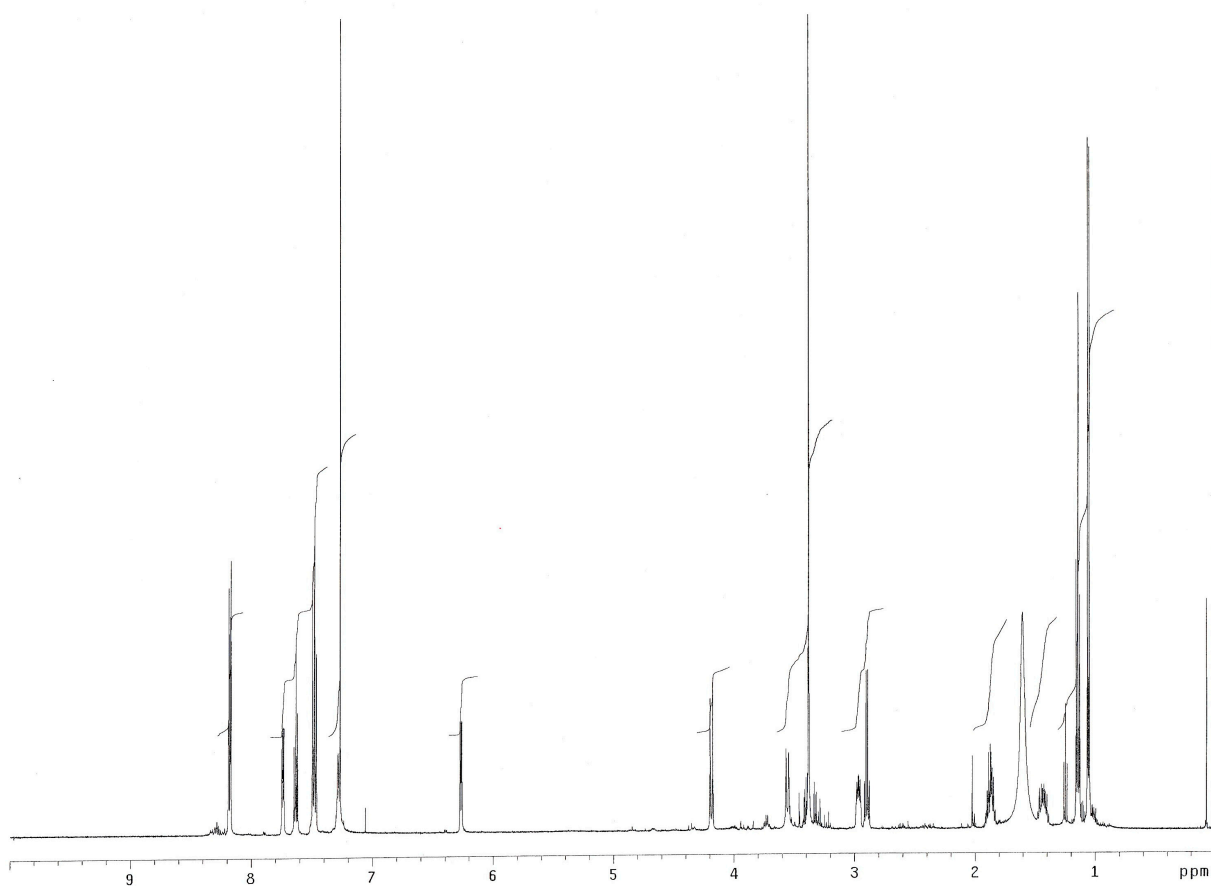
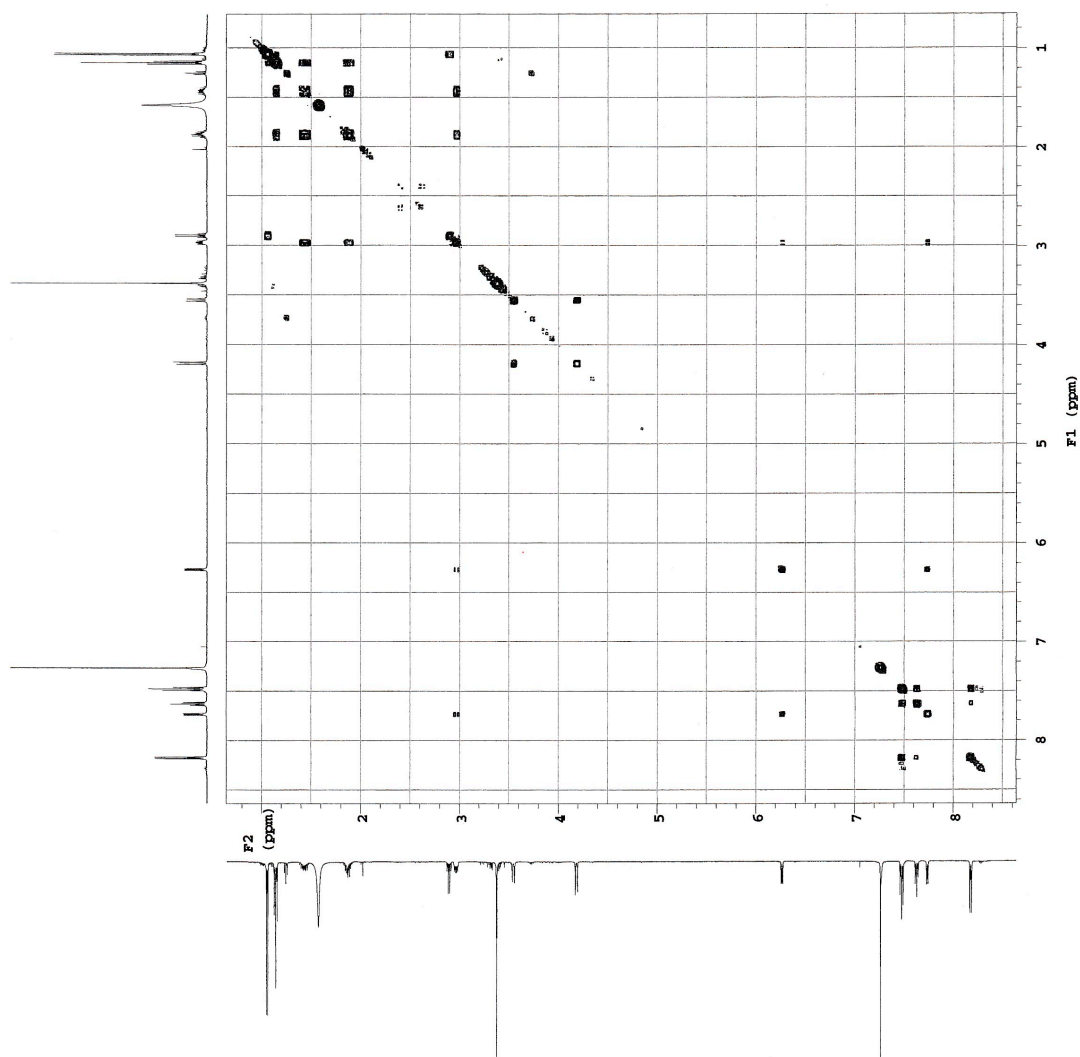


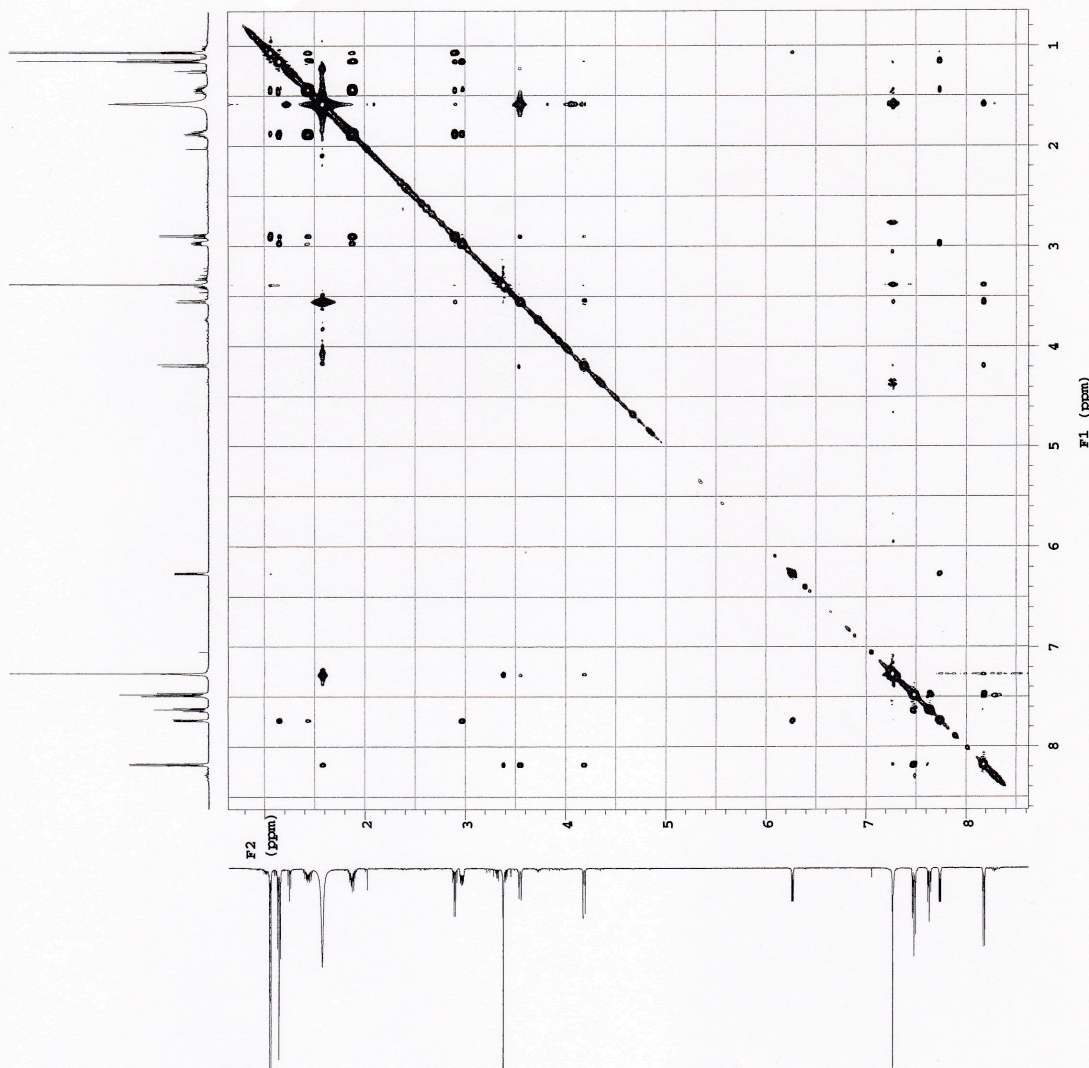
Figure S37 ^1H - ^1H COSY of 6



1068-5 C386 4-5 XVIII
exp3 gcosy

date	Dec 11 2009	ha	nm
solvent	dms-d ₆	spul	n
nmr	undecoupled	mag1	441.4
ACQUISITION	SPECIAL		
sw	4672.1	temp	not used
at	0.219	gain	58
np	2048	spin	not used
fb	3000	f2	PROCESSING
sa	16	ab	-0.110
sc	1.000	as	not used
nt	16	fn	2048
2D ACQUISITION	F1 PROCESSING		
sw1	4672.1	ab1	-0.027
ni	256	ab1	not used
TRANSMITTER	lp		
tn	H1	fn1	2048
freq	499.850	sp	321.8
ref	-298.6	wp	3992.3
tpwr	53	wp	3987.7
pw	9.500	wp1	326.3
GRADIENTS	F1		
gvlv11	441.4	rf1	139.0
gt1	0.001000	rfp	139.1
gfab	0.000500	rf11	139.1
dc	RECOUPLED	rf1	0
dn	nm	nc	200.0
ec	0	ec2	200.0
ec2	0	va	261
va	261	ab	4
ab	4	sv	

Figure S38 NOESY of **6**



106B-5 C356 4-5 XXVIII

exp4 NOESY

SAMPLE		date		Dec 11 2009		hs		FLAGS	
sample	solvent	CD13	aspl	sample	CD13	aspl	sample	CD13	aspl
ACQUISITION	ACQUISITION	hasplv1	4414	ACQUISITION	hasplv1	4414	ACQUISITION	hasplv1	4414
4502.7	4502.7	0.227	temp	not used	0.227	temp	not used	0.227	temp
2048	2048	gain	58	2048	gain	58	2048	gain	58
3000	3000	spin	not used	3000	spin	not used	3000	spin	not used
41	41	F2 PROCESSING	0.050	41	F2 PROCESSING	0.050	41	F2 PROCESSING	0.050
24	24	gfs	not used	24	gfs	not used	24	gfs	not used
2D ACQUISITION	2D ACQUISITION	F1 PROCESSING	2048	2D ACQUISITION	F1 PROCESSING	2048	2D ACQUISITION	F1 PROCESSING	2048
256	256	gfi	0.040	256	gfi	0.040	256	gfi	0.040
H1	H1	proci	1p	H1	proci	1p	H1	proci	1p
499.983	499.983	fml	2048	499.983	fml	2048	499.983	fml	2048
320.9	320.9	sp	310.9	320.9	sp	310.9	320.9	sp	310.9
9.500	9.500	wp	3980.3	9.500	wp	3980.3	9.500	wp	3980.3
1.000	1.000	wp1	323.5	1.000	wp1	323.5	1.000	wp1	323.5
PRESENTATION	PRESENTATION	zf1	3983.9	PRESENTATION	zf1	3983.9	PRESENTATION	zf1	3983.9
nmnm	nmnm	zf0	76.7	nmnm	zf0	76.7	nmnm	zf0	76.7
0	0	zf1	76.7	0	zf1	76.7	0	zf1	76.7
0	0	zf0	0	0	zf0	0	0	zf0	0
0	0	zf1	0	0	zf1	0	0	zf1	0
200.0	200.0	wp	200.0	200.0	wp	200.0	200.0	wp	200.0
200.0	200.0	zf0	200.0	200.0	zf0	200.0	200.0	zf0	200.0
261	261	wp	261	261	wp	261	261	wp	261
261	261	zf0	261	261	zf0	261	261	zf0	261
261	261	zf1	261	261	zf1	261	261	zf1	261
261	261	zf0	261	261	zf0	261	261	zf0	261
261	261	zf1	261	261	zf1	261	261	zf1	261
261	261	zf0	261	261	zf0	261	261	zf0	261
261	261	zf1	261	261	zf1	261	261	zf1	261
261	261	zf0	261	261	zf0	261	261	zf0	261
261	261	zf1	261	261	zf1	261	261	zf1	261
261	261	zf0	261	261	zf0	261	261	zf0	261
261	261	zf1	261	261	zf1	261	261	zf1	261
261	261	zf0	261	261	zf0	261	261	zf0	261
261	261	zf1	261	261	zf1	261	261	zf1	261
261	261	zf0	261	261	zf0	261	261	zf0	261
261	261	zf1	261	261	zf1	261	261	zf1	261
261	261	zf0	261	261	zf0	261	261	zf0	261
261	261	zf1	261	261	zf1	261	261	zf1	261
261	261	zf0	261	261	zf0	261	261	zf0	261
261	261	zf1	261	261	zf1	261	261	zf1	261
261	261	zf0	261	261	zf0	261	261	zf0	261
261	261	zf1	261	261	zf1	261	261	zf1	261
26									

Figure S39 HMQC of **6**

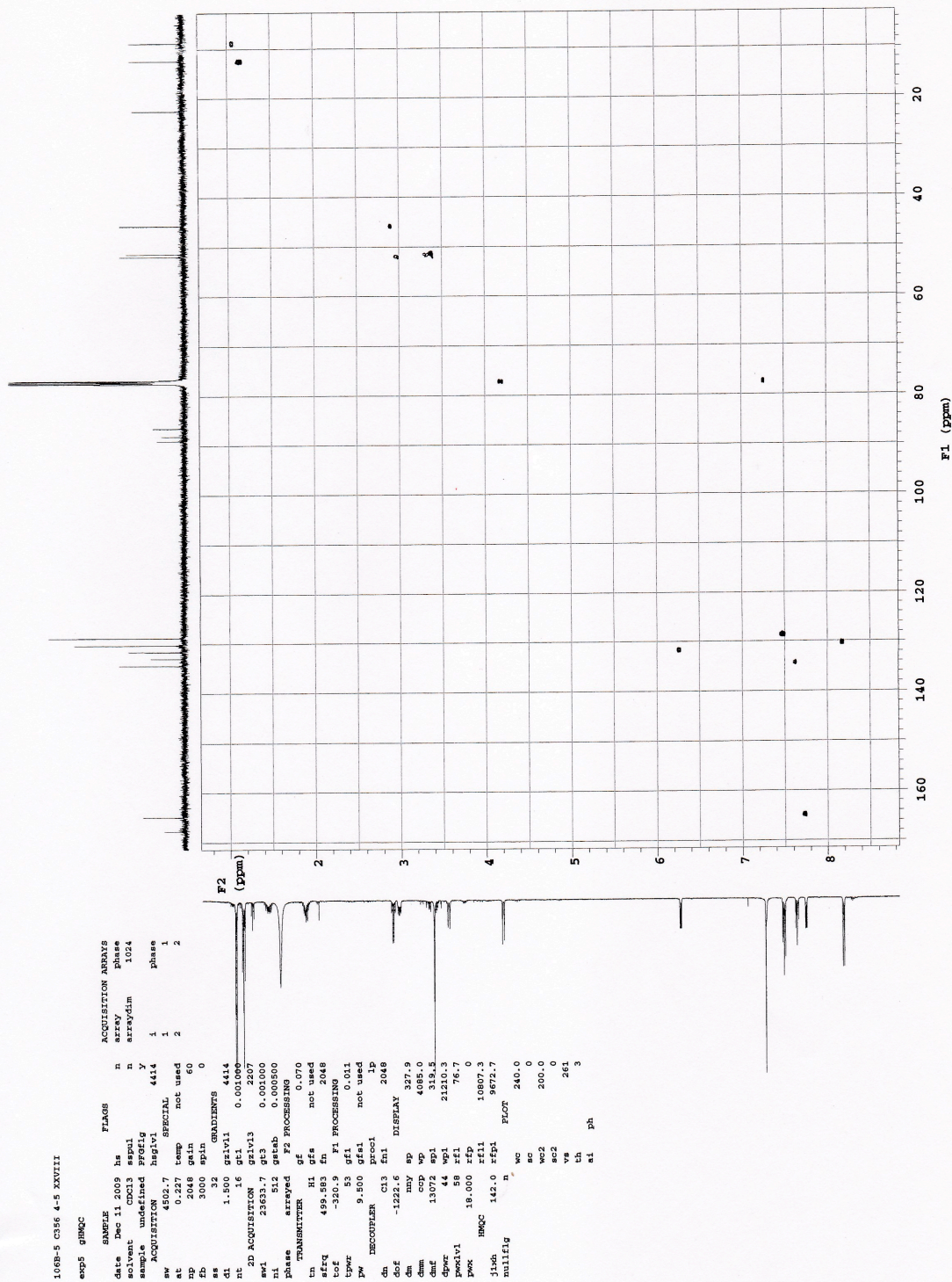


Figure S41 IR spectrum of 6

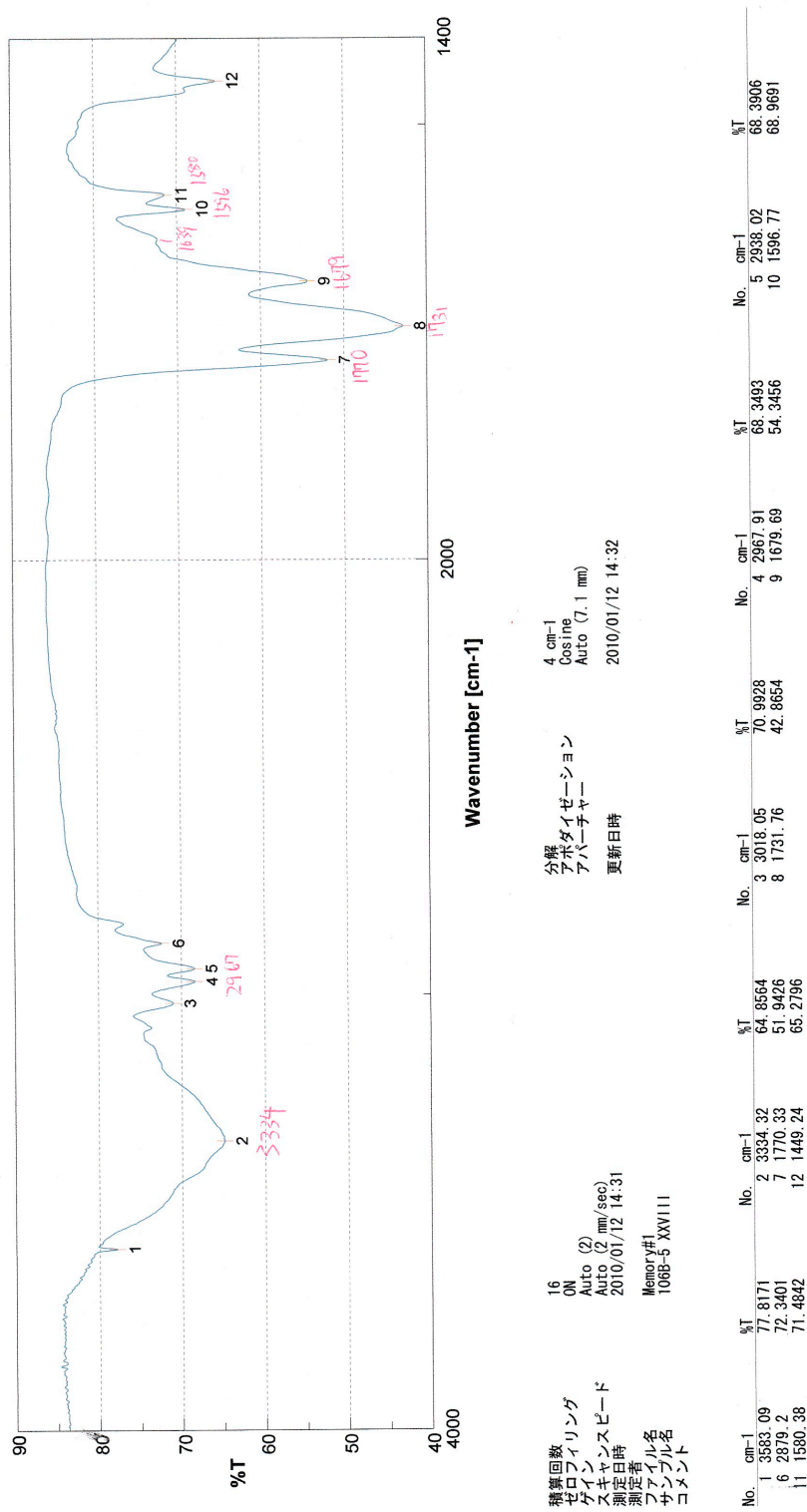


Figure S42 FABMS of 6

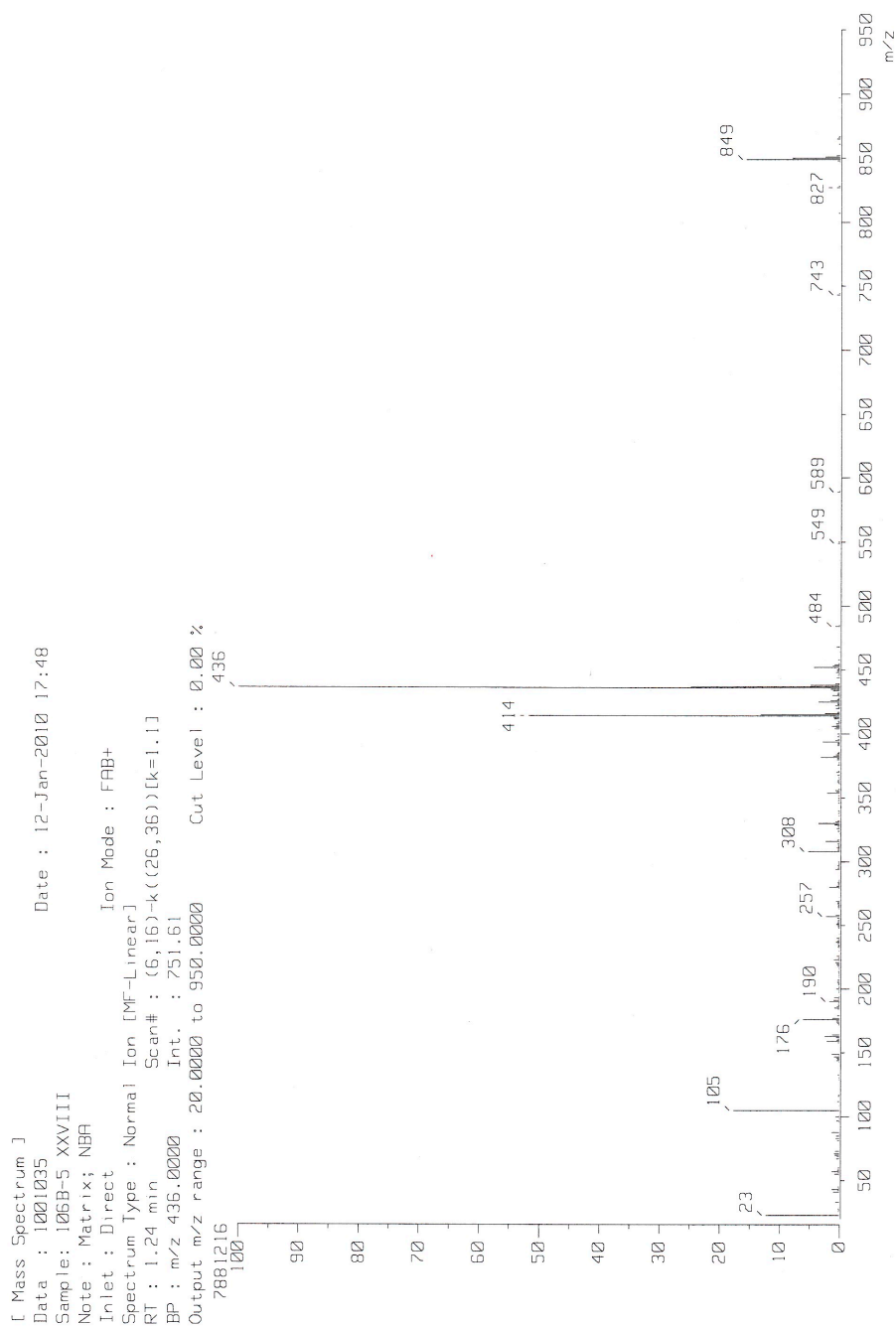


Figure S43 H and ^{13}C NMR spectra of **7** in CDCl_3

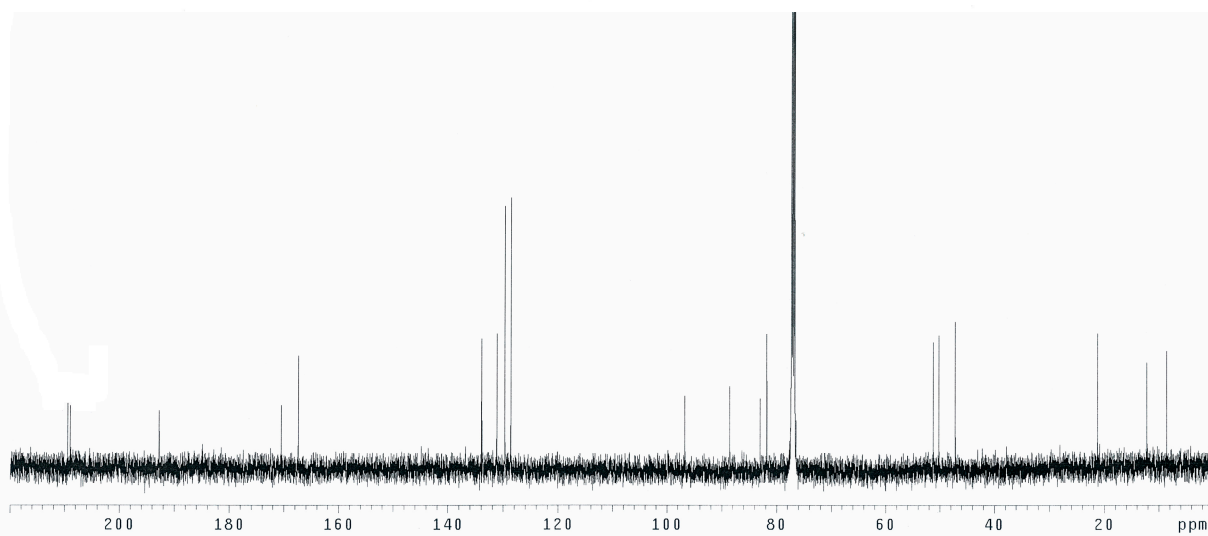
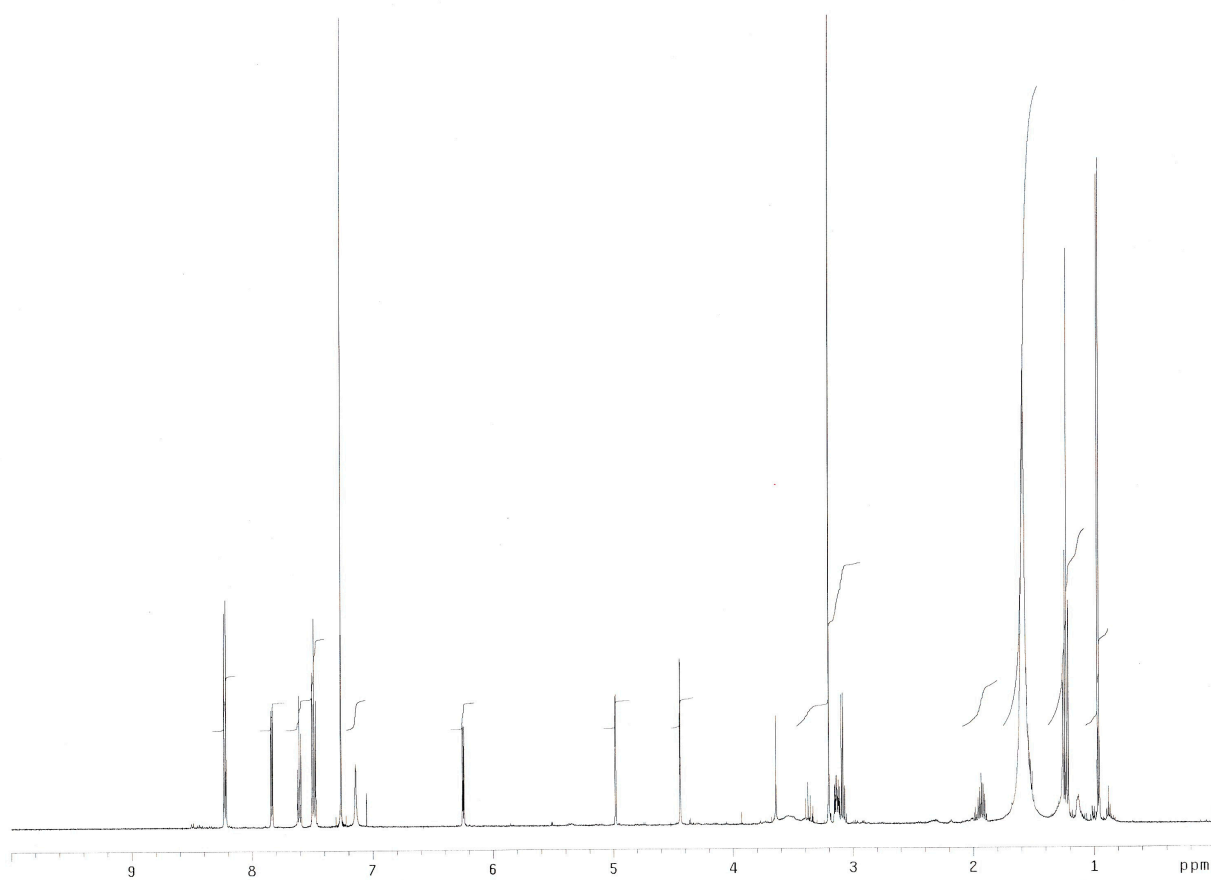
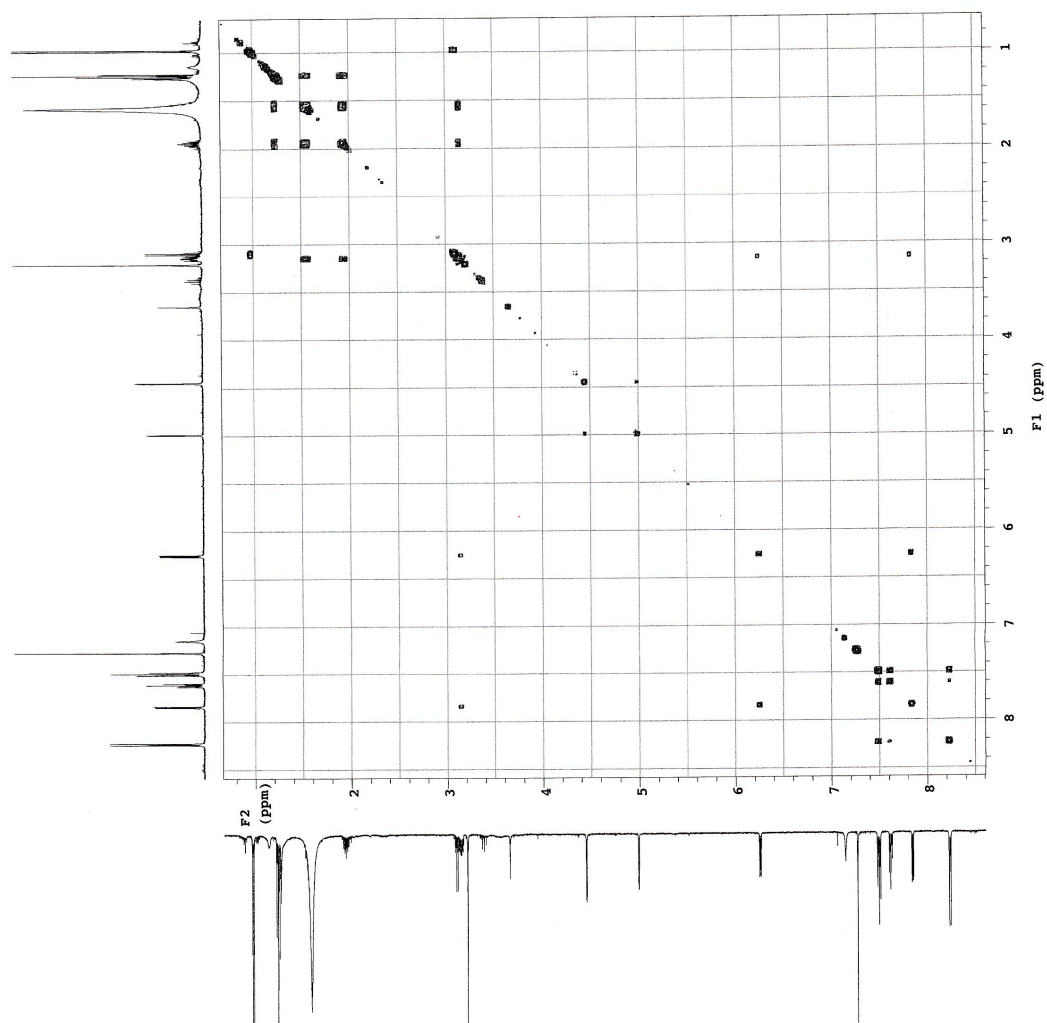


Figure S44 ^1H - ^1H COSY of 7



1068-S-XXIII-metha
exp33 90081

date	Sep 29 2010	hs	PLANS	nn
sample	undefined	hspl		n
solvent	CNCl3			4414
ACQUISITION			SPECIAL	
nu	4442.7	temp	not used	
sc	0.210	gain	not used	88
fb	2048	spin	not used	
dp	2000	F2 PROCESSING		
ss	16	ab	-0.115	
dl	1.000	sbs	not used	
nt	4442.7	nu	2048	
nu2	4442.7	nu1	PROCESSING	
ni	256	sbal	not used	
tn	TRANSMITTER	proc	ip	
ntq	459.5	wp	312.1	
tpw	-347.7	wp	3565.5	
pw	9.500	wp1	319.0	
GRADIENTS		wp1	3569.8	
g1v11	4414	rf1	84.4	
g1v10	0.0000	rf1	84.5	
g1v10	0.0000	rf1	84.5	
DECOUPLER	rf1	0		
dn	mm	mc	200.0	
sc	0			
sc2	200.0			
vs	216			
th	4			
si	av			

Figure S45 NOESY of 7

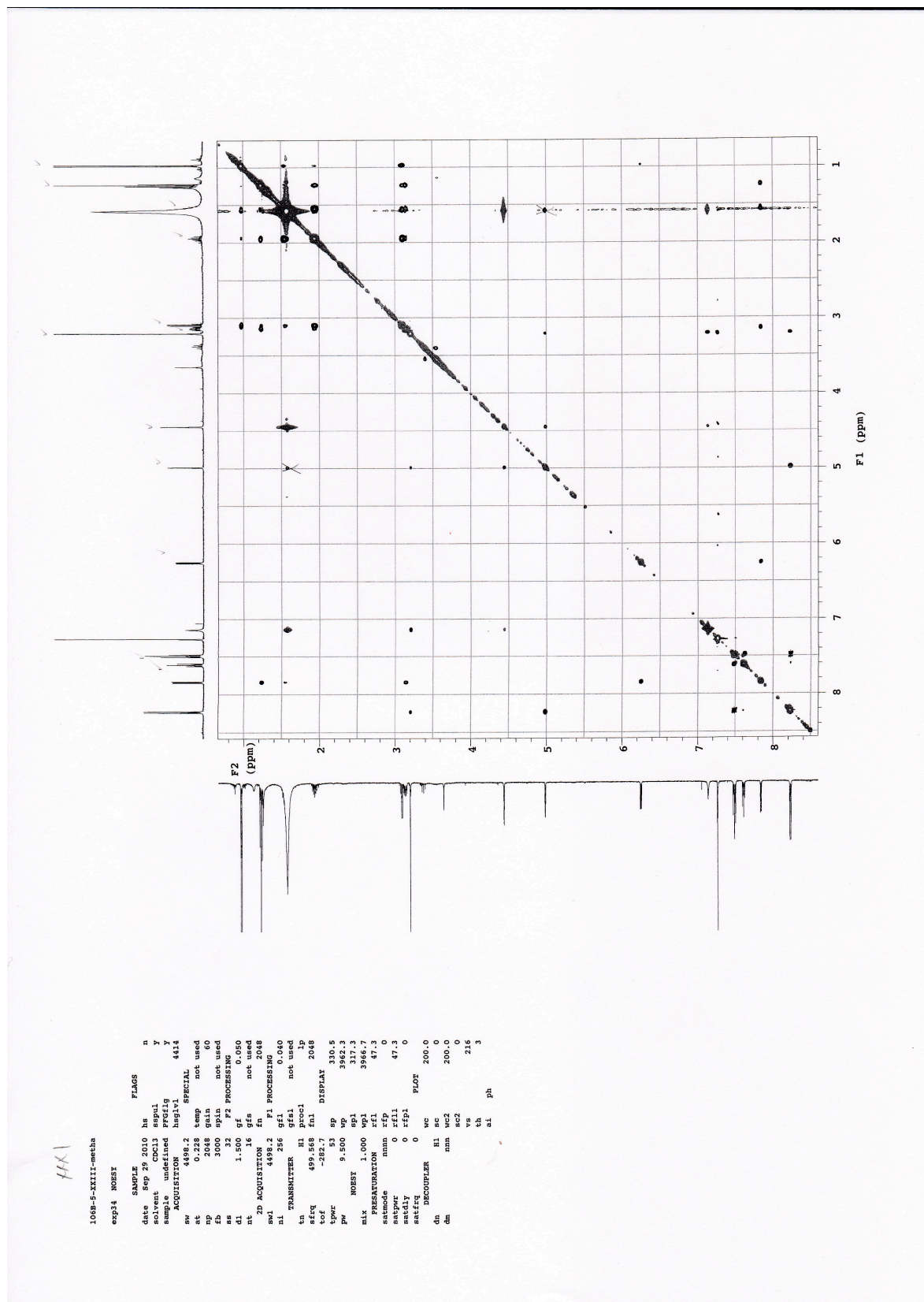


Figure S46 HMQC of 7

106B-S-XIII-metha

ex235 ghmqc

SAMPLE NAME ACQUISITION ARRAYS
 Date Sep 2010 hs 1024
 solvent CDCl3 setup n arraydim
 sample undefined pfgflg y
 ACQUISITION hsglvl 4414 i phase
 AN 4498.2 SPECIAL 1 1
 at 0.228 temp not used 2 2
 p2 0.00500 gfm 40
 Zb 3000 spin 0
 ss 32 GRADIENTS 0
 d1 1.500 gvlvl 4414
 nt 8 9tl 0.001000
 2D ACQUISITION 9tlv13 0.001000
 ncl 2500.3 gvlv13 0.001000
 si 512 gtab 0.000500
 phase arrayed F2 PROCESSING
 TRANSMITTER gf 0.050
 tn hl gfa not used
 hfq 498.5 gfm 4414
 tpr -282.7 F1 PROCESSING
 tpr 53 gfl 0.011
 PM 9.500 gfa1 not used
 DECOUPLER C13 pccol
 dn C13 fml 2048
 scf -1318.3 wp 304.1
 dm csp wp 4133.6
 dmf 11111 spl 445.0
 dpr 45 wpl 21861.9
 pwtvl 61 ffl 47.3
 pwr 16.500 ffl 11022.1
 j1sh 146.0 ffl 9672.4
 nullflg n wc 240.0
 ac 0
 mc2 200.0
 vs 425
 th 4
 ai ph

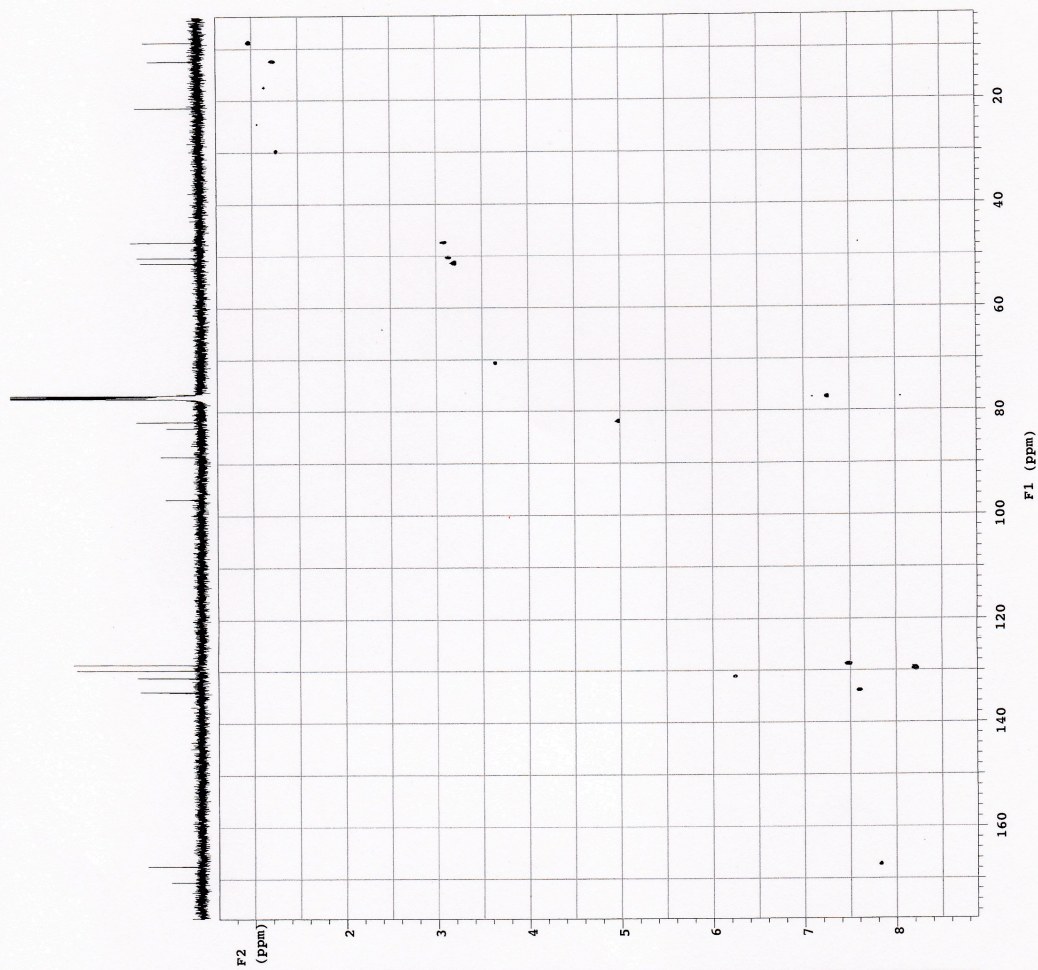


Figure S47 HMBC of 7

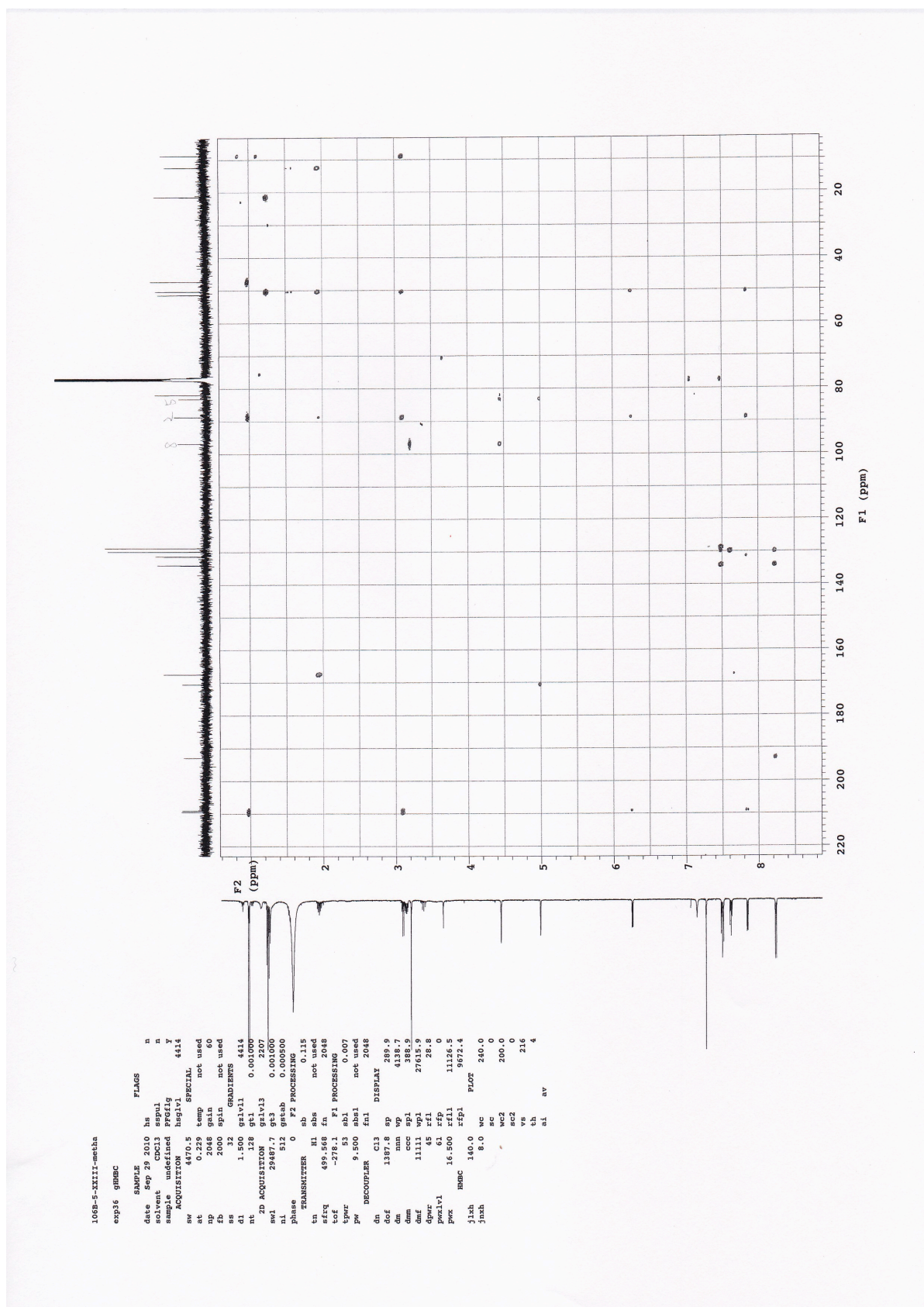


Figure S48 IR spectrum of 7

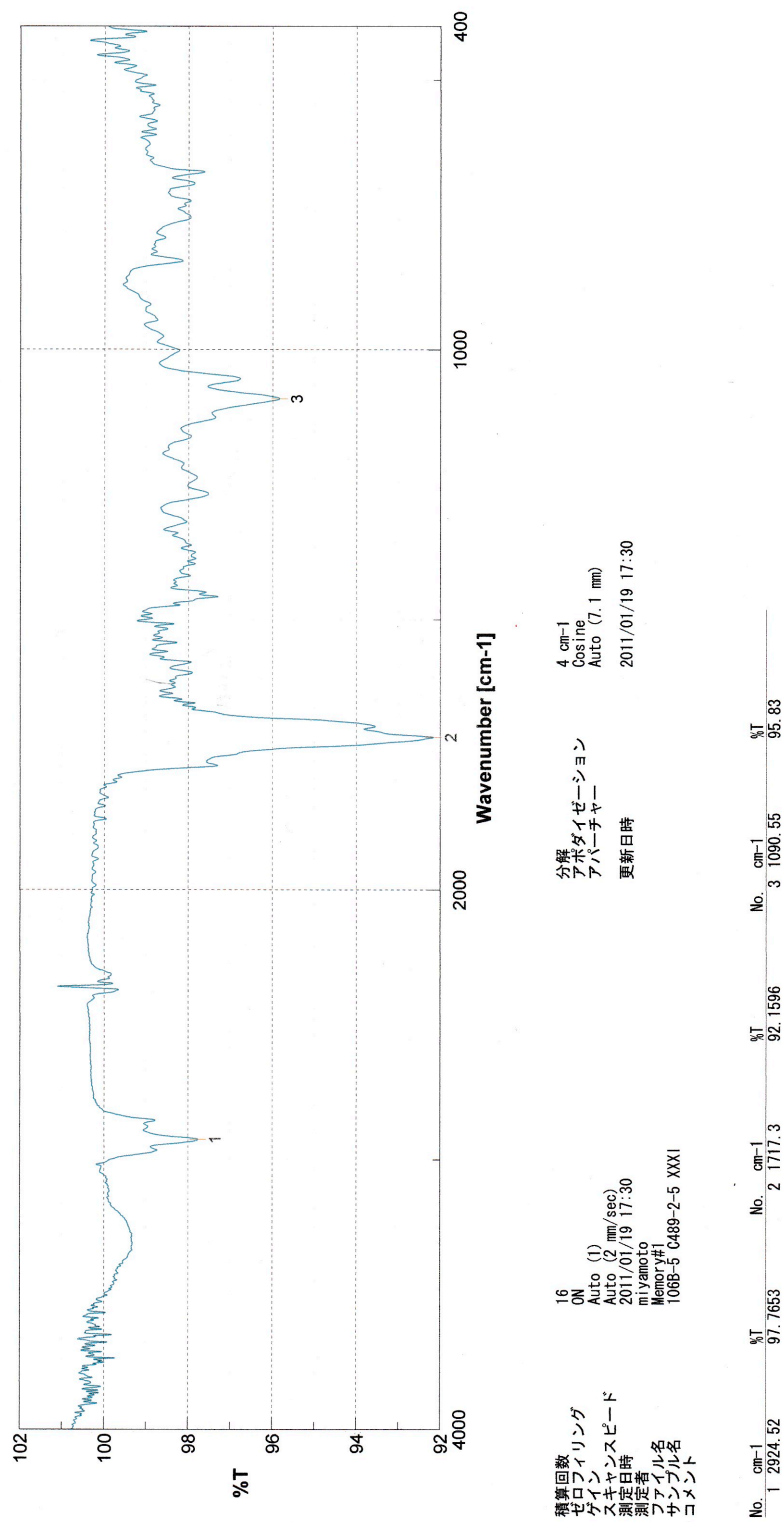


Figure S49 FABMS of 7

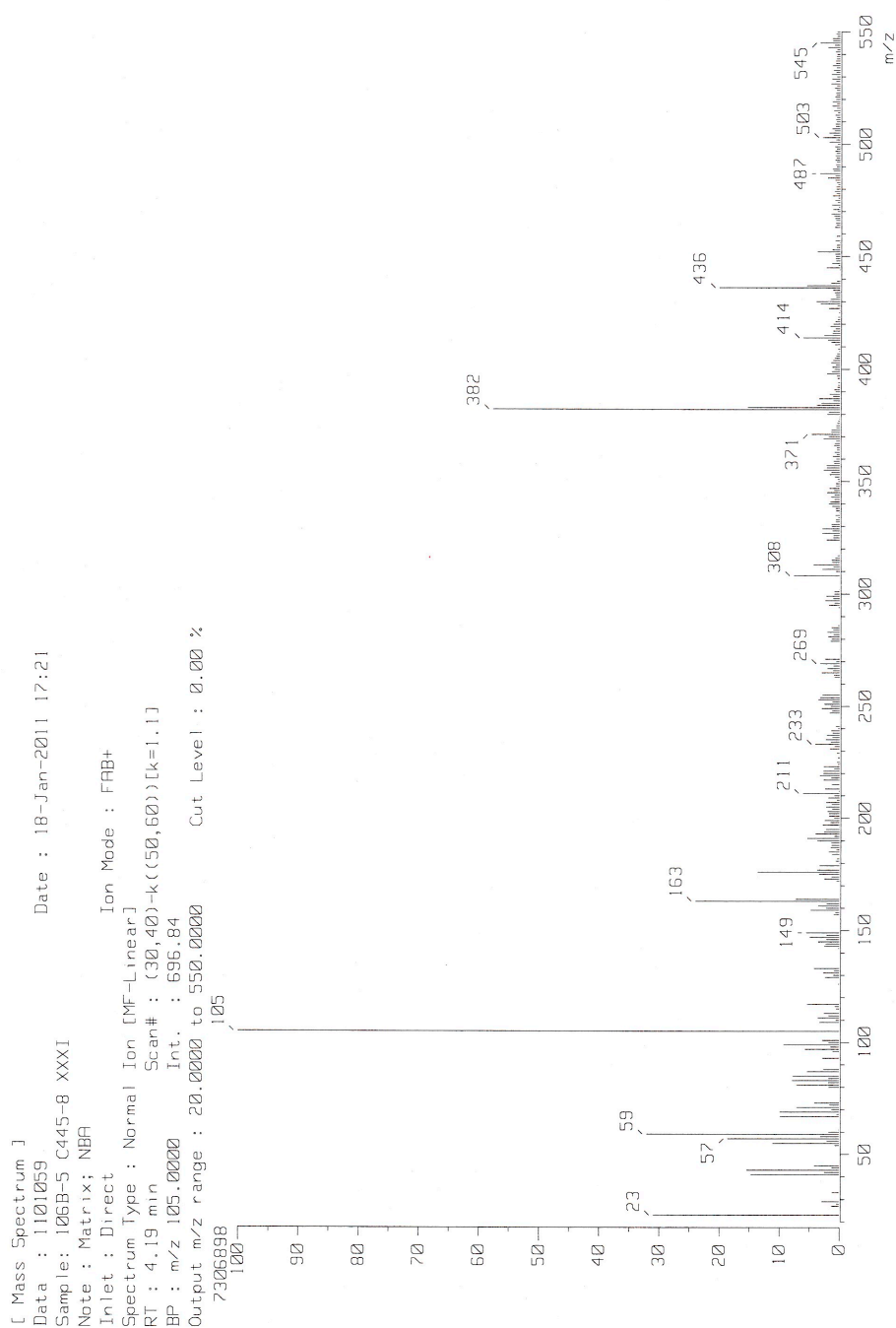


Figure S50 ^1H and ^{13}C NMR spectra of **8** in CDCl_3

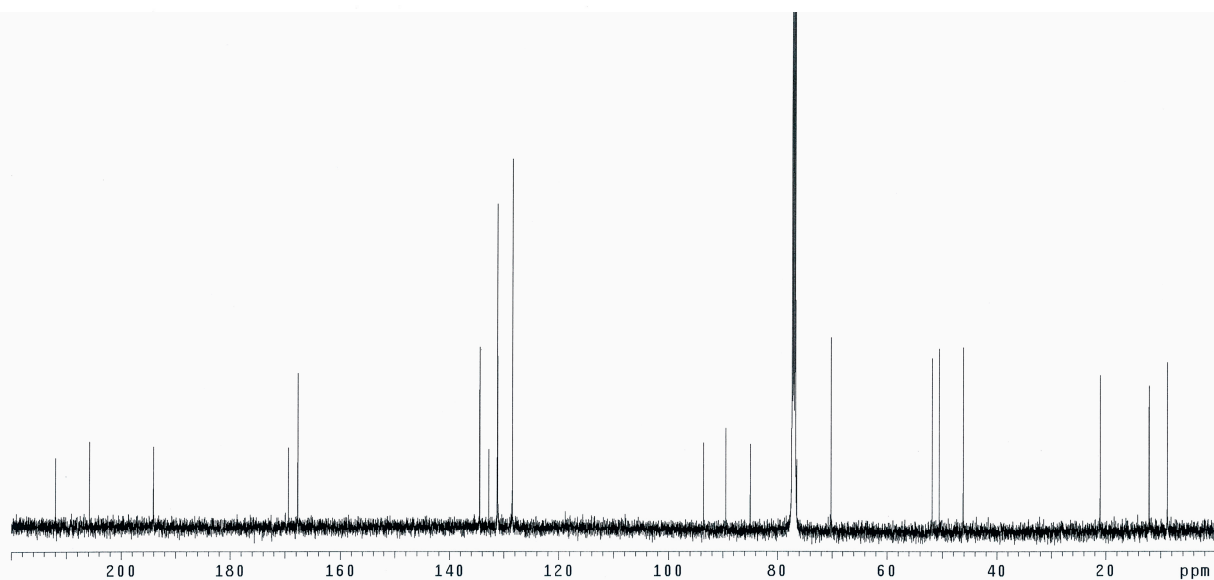
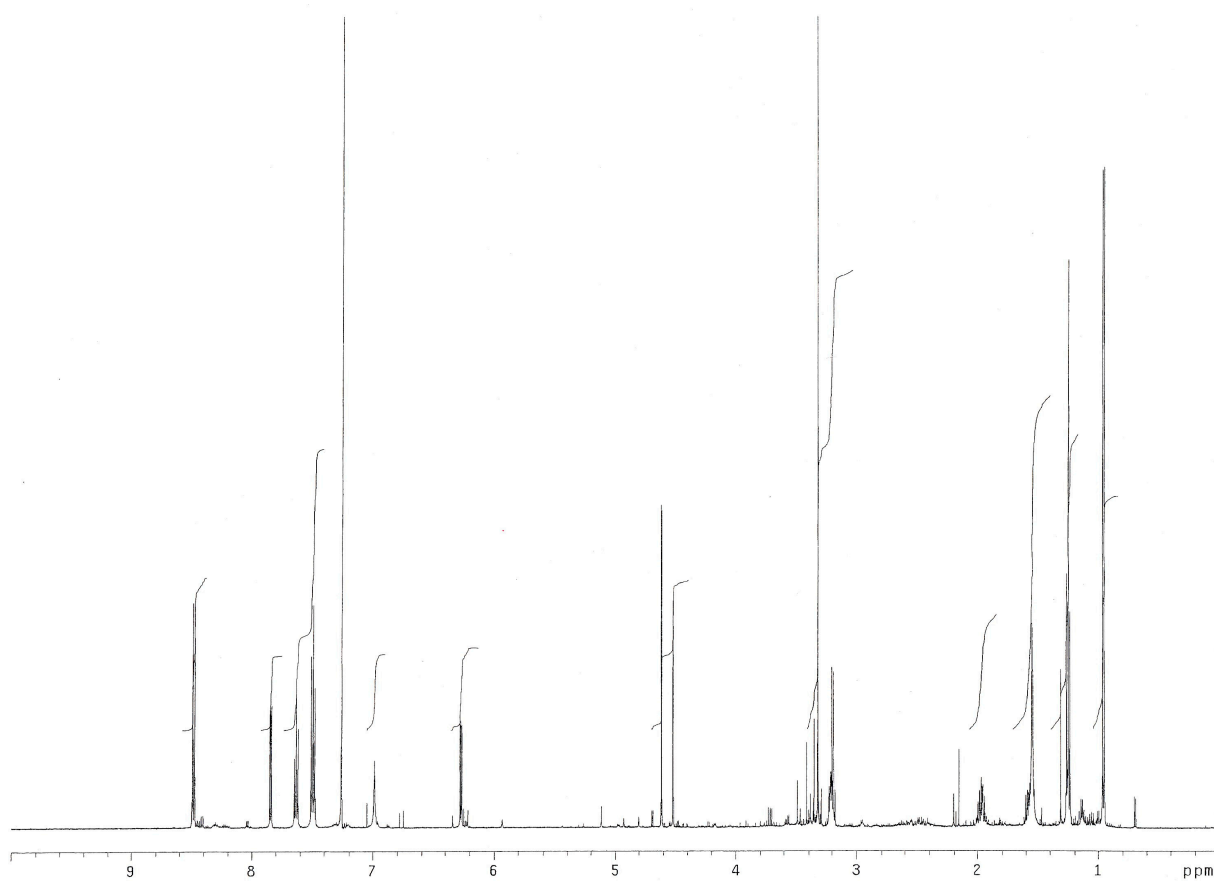
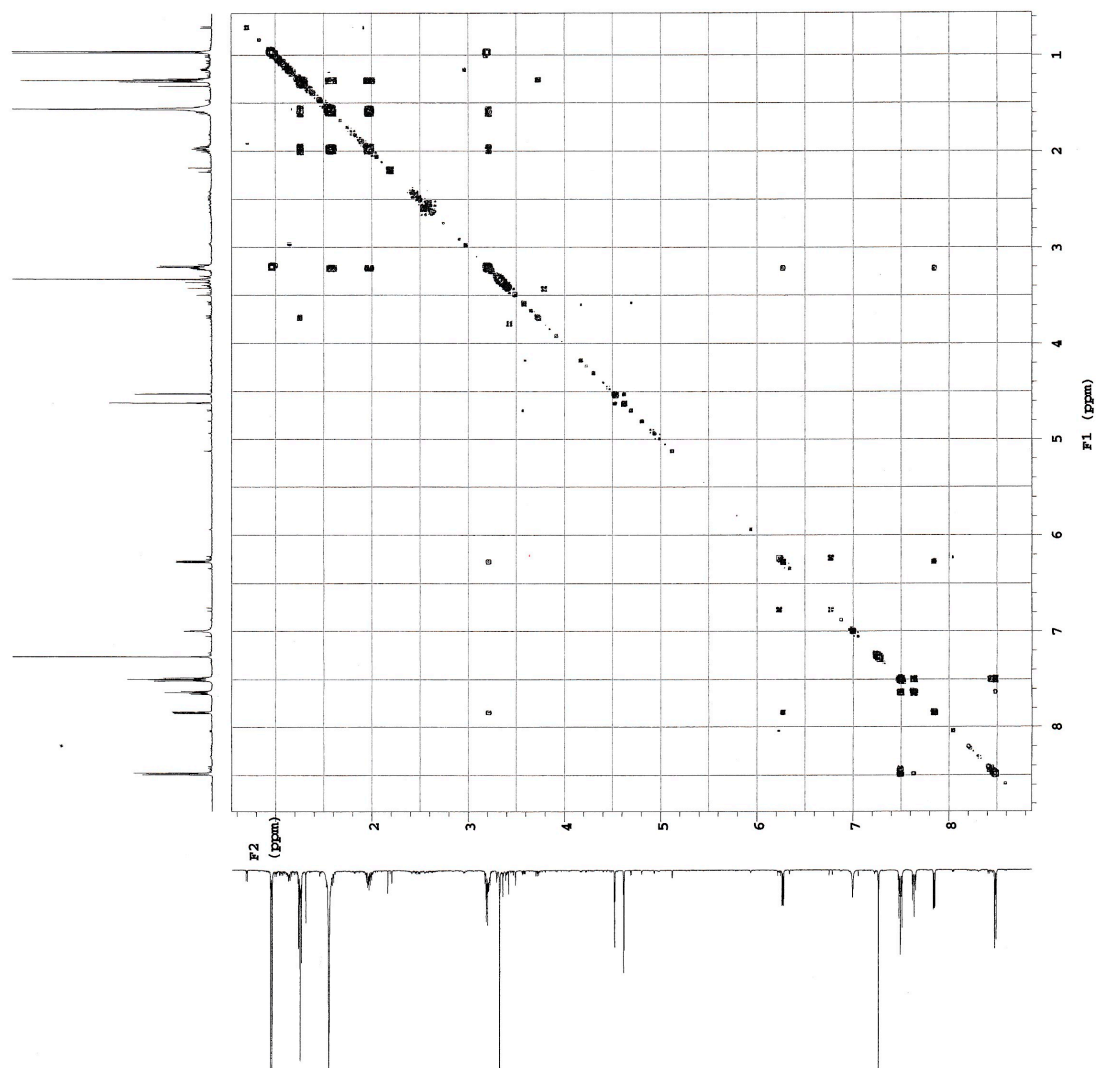


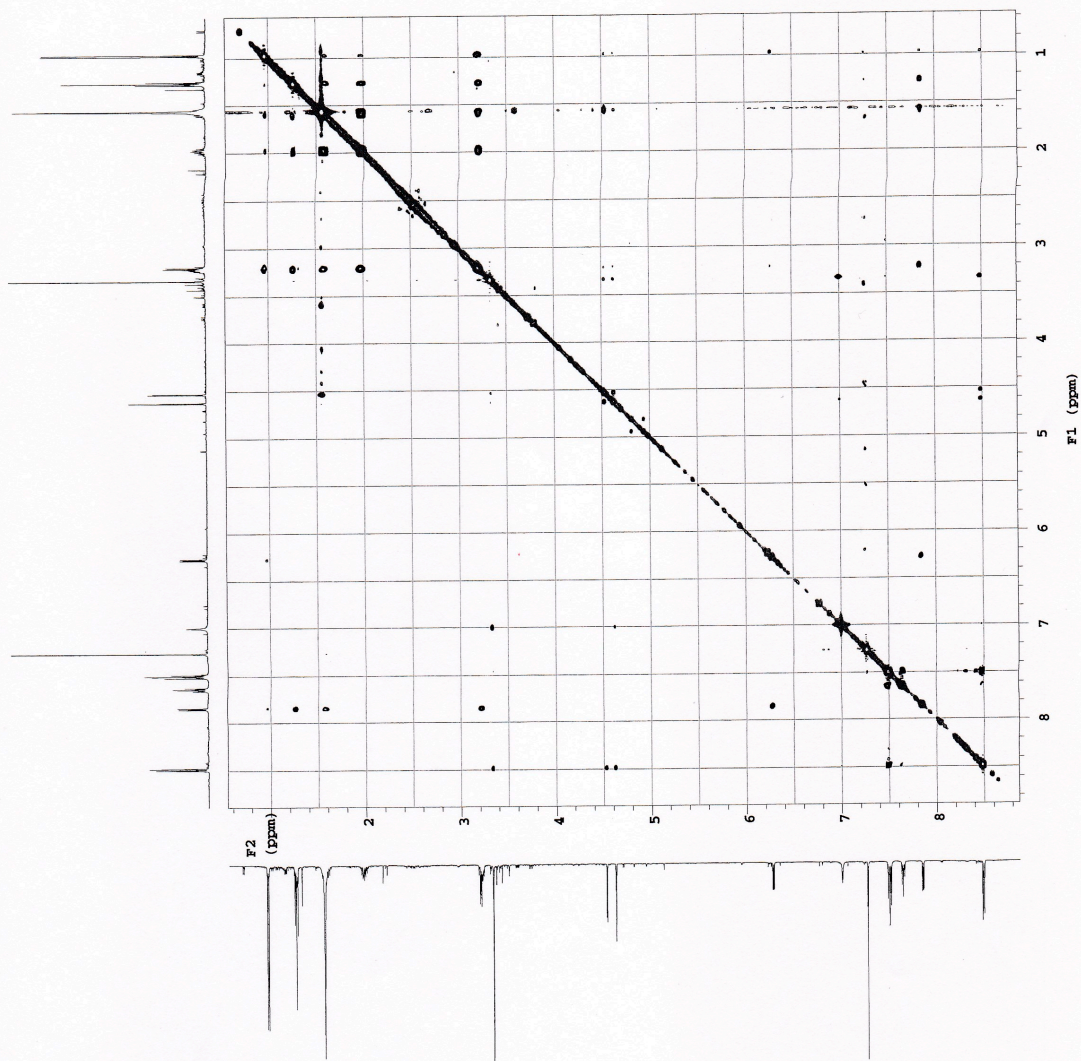
Figure S51 ^1H - ^1H COSY of 8



```

exp3 gCOSY
NAMEZ 1068-1 C259-8 XIII
date Apr 2 2009 hs
solvent CDCl3
sample undefined hspv1 4414
ACQUISITION SPECIAL
sw 4524.4 temp not used
at 0.256 gain 58
np 2048 spin not used
zb 3000 f2 PROCESSING
dt 0.163
d1 1.000 sbx not used
nt 2048
2D ACQUISITION F1 PROCESSING
sw1 4524.4 sb1 -0.028
ni 256 sbal not used
TRANSMITTER H1
fn 2048
tq 498.450
tof -305.6 wp 4153.2
tpwr 9.500 wpl 277.1
GRADIENTS
g2lv11 4414 rfl 4157.7
gt1 0.001000 rfp 0
gscab 0.000500 rll 76.4
dn RECOUPLED rfp 0
dm min wc 200.0
sc 0
wc2 200.0
sc2 0
vs 798
th 4
al cdc av
  
```

Figure S52 NOESY of **8**



106B-5 C259-8 XXII

exp4 NOESY

[illegible]

Figure S53 HMQC of 8

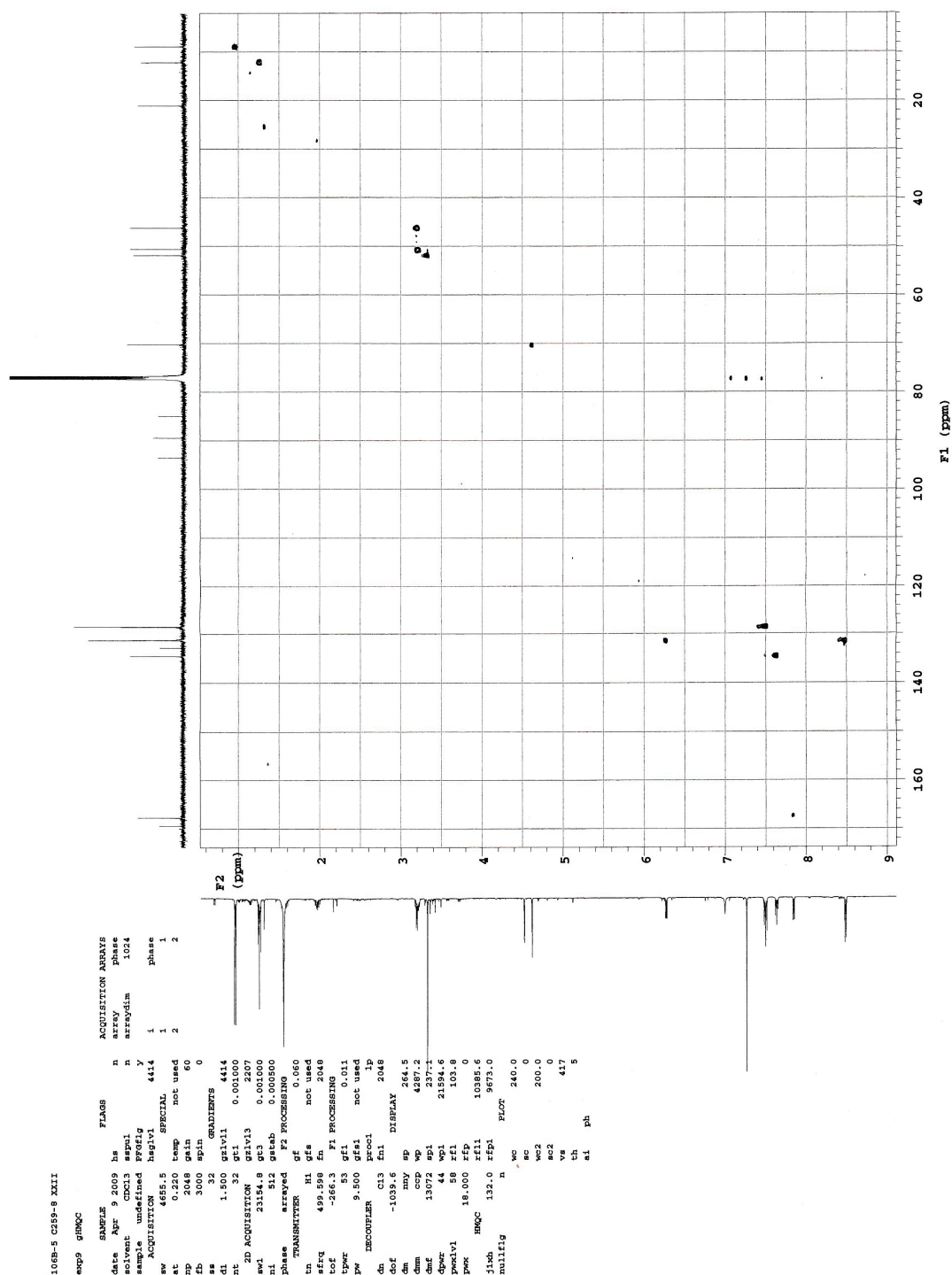


Figure S54 HMBC of 8

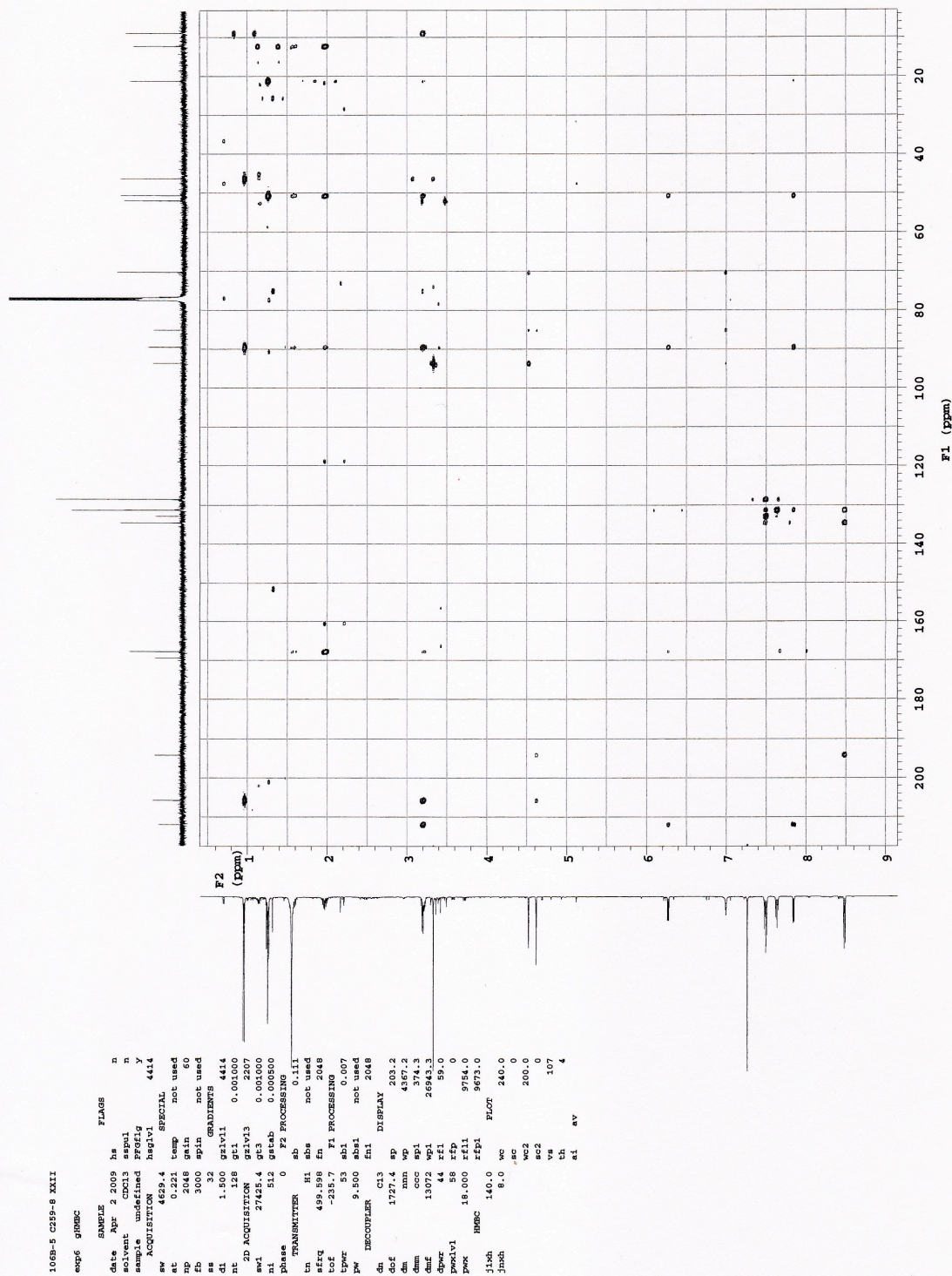


Figure S55 IR spectrum of 8

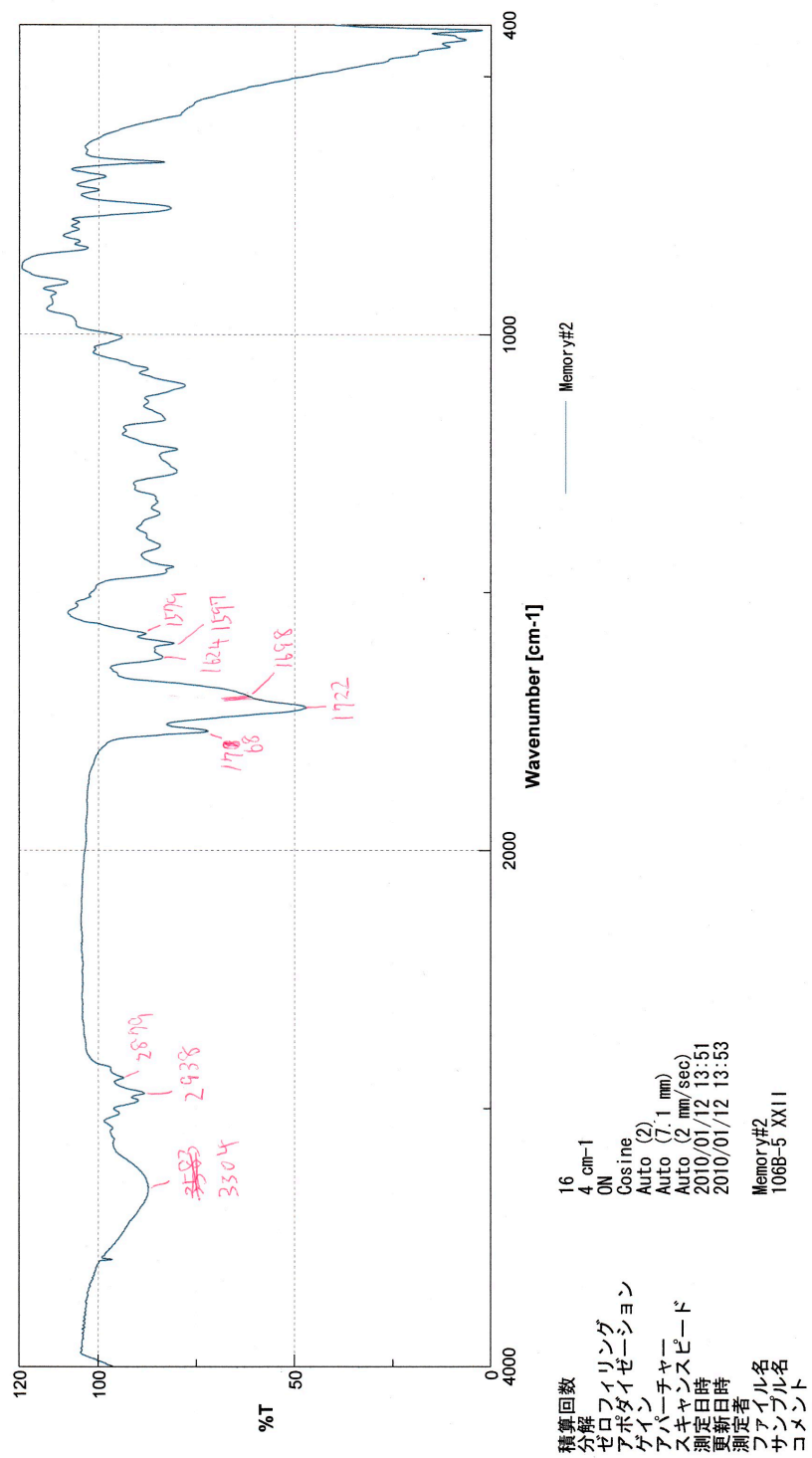


Figure S56 FABMS of 8

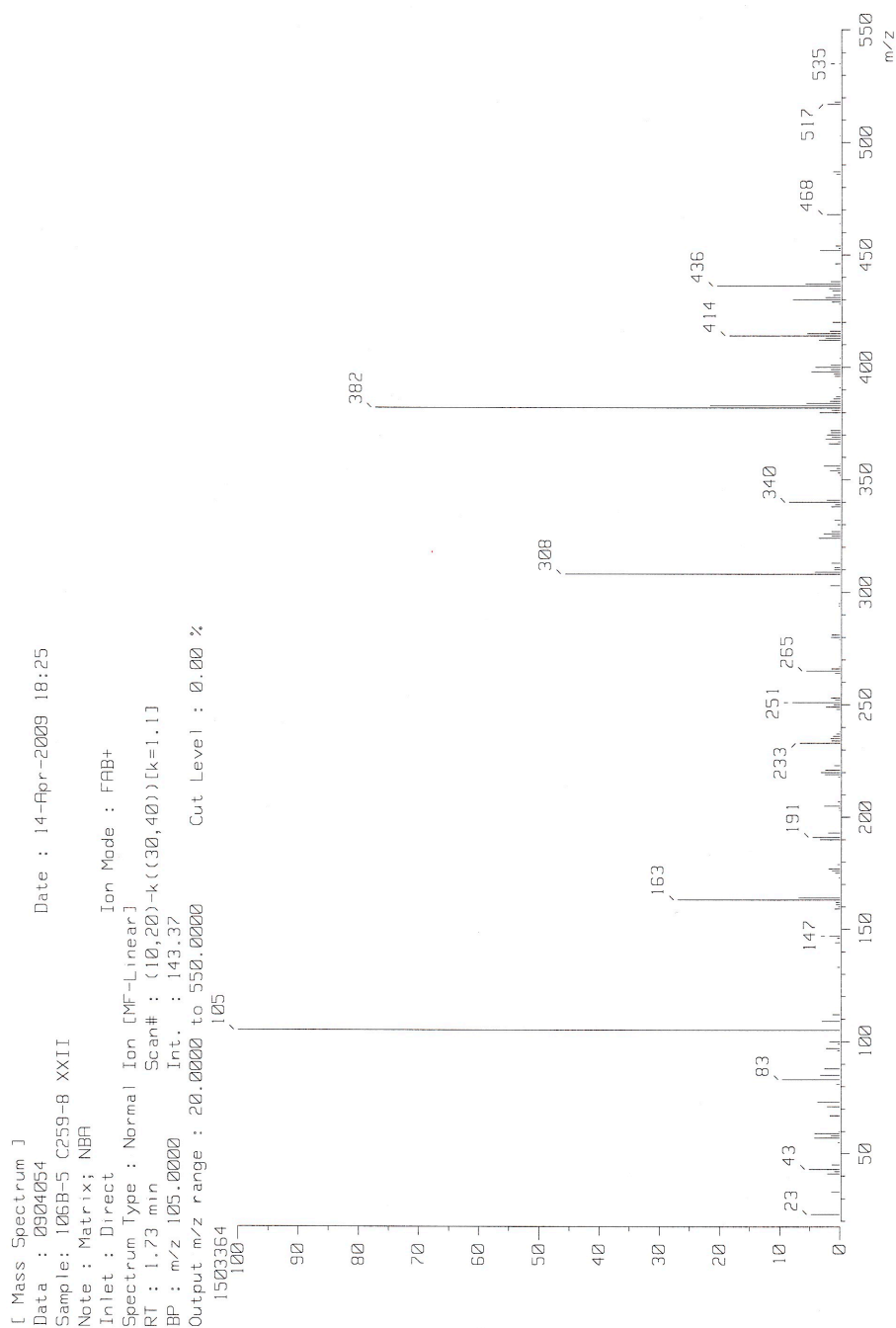


Figure S57 ^1H NMR spectrum of **1'**

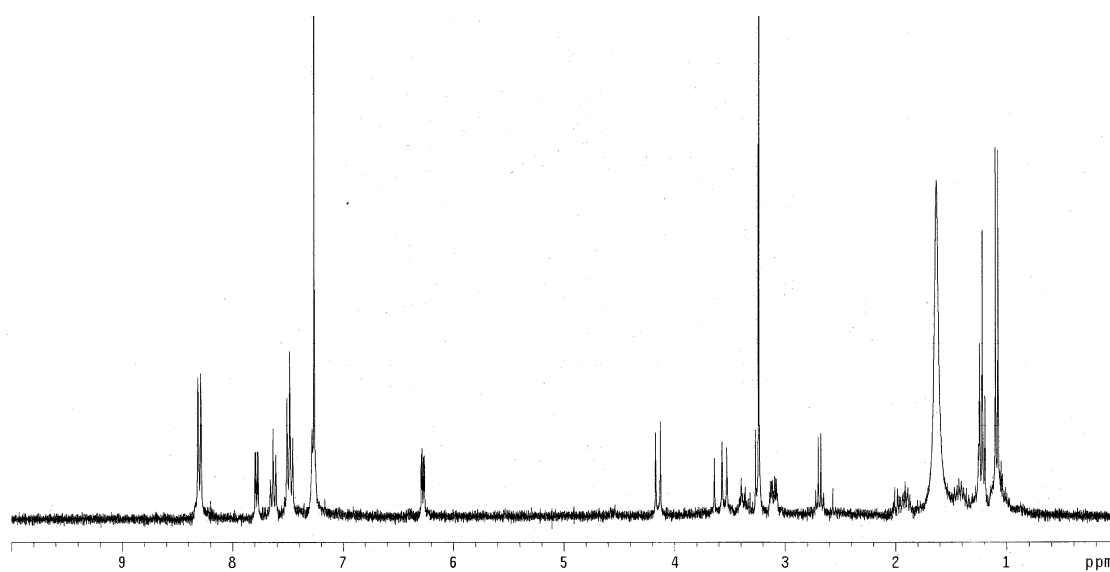


Figure S58 ^1H NMR spectrum of **2'**

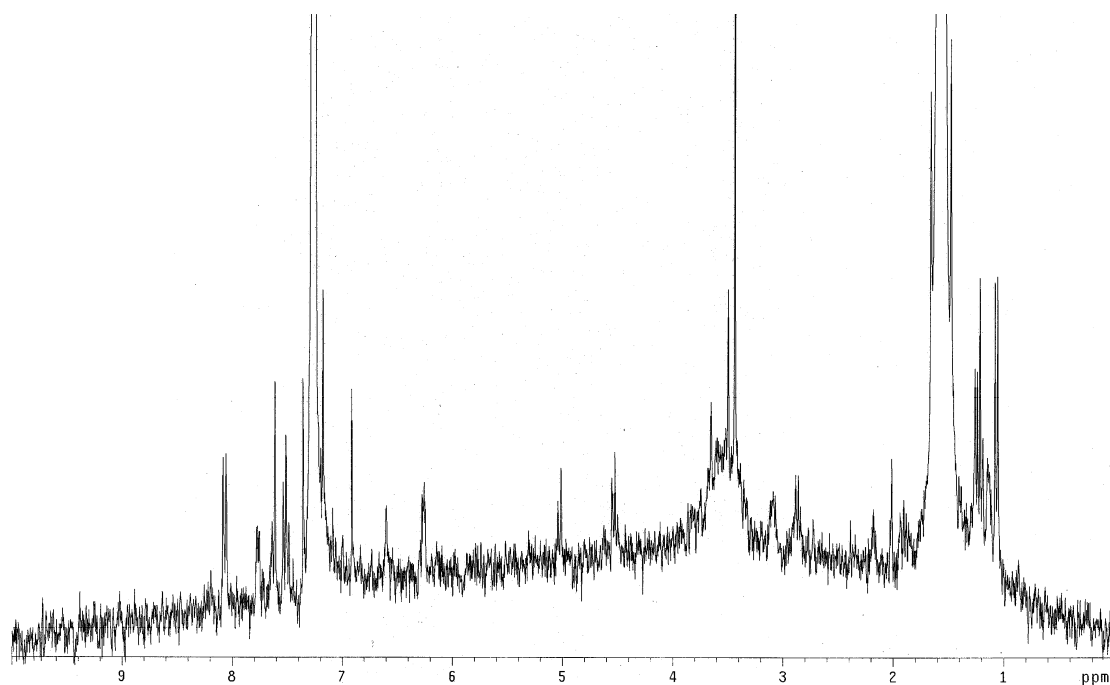


Figure S59 ^1H NMR spectrum of **3'**

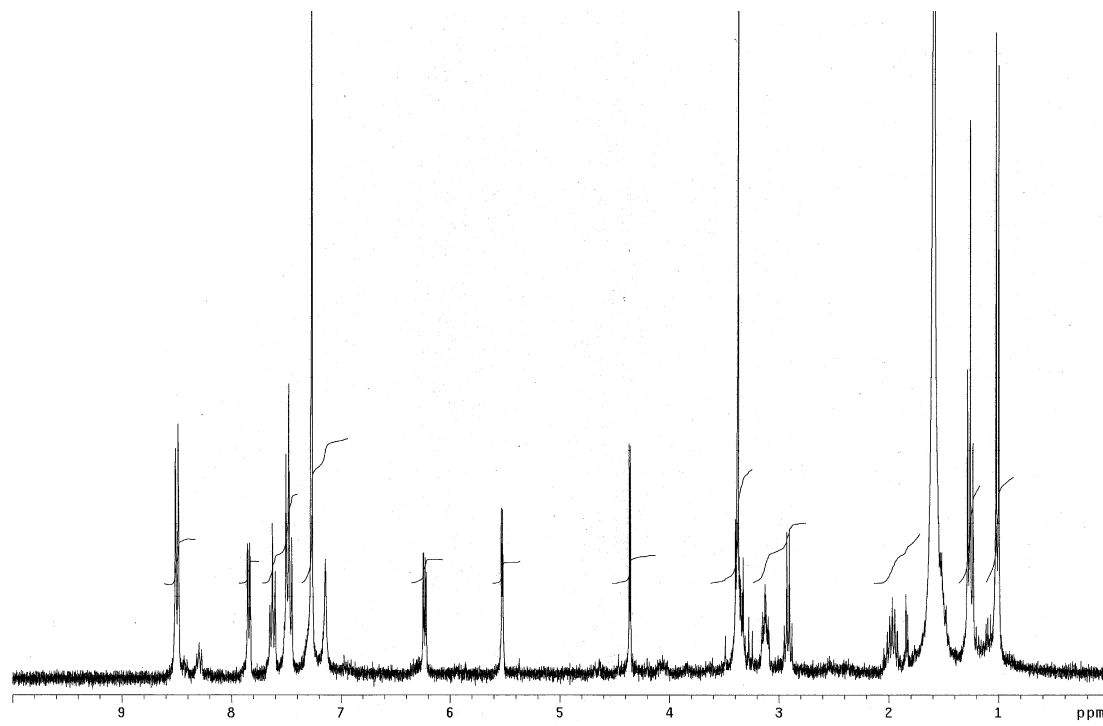


Figure S60 ^1H NMR spectrum of **4'**

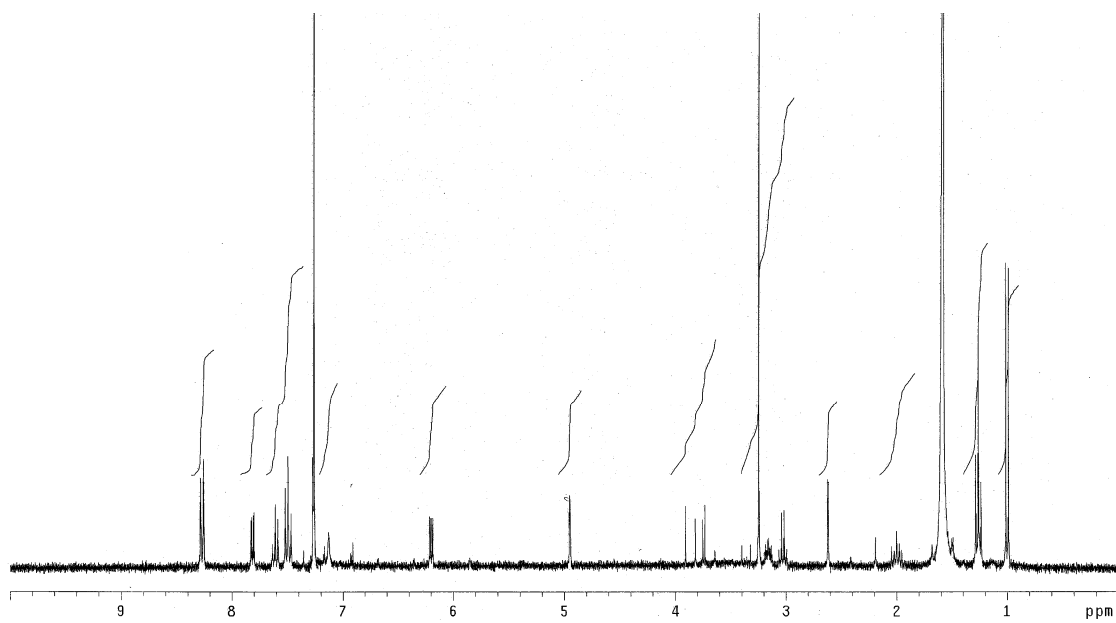


Figure S61 ^1H NMR spectrum of **5'**

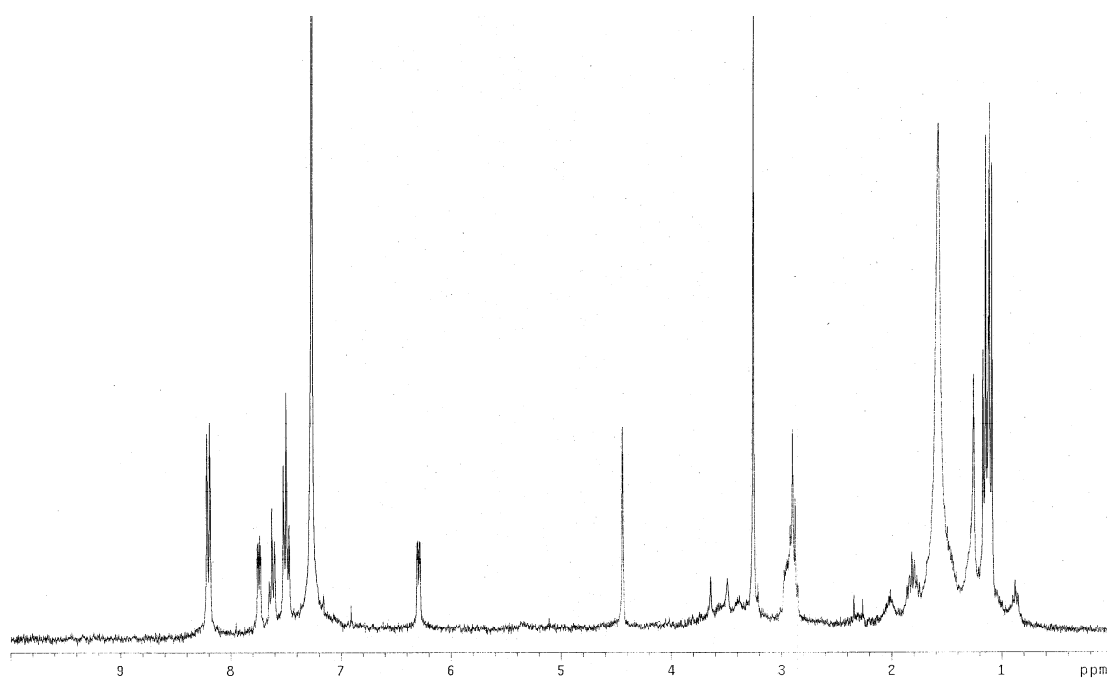


Figure S62 ^1H NMR spectrum of **6'**

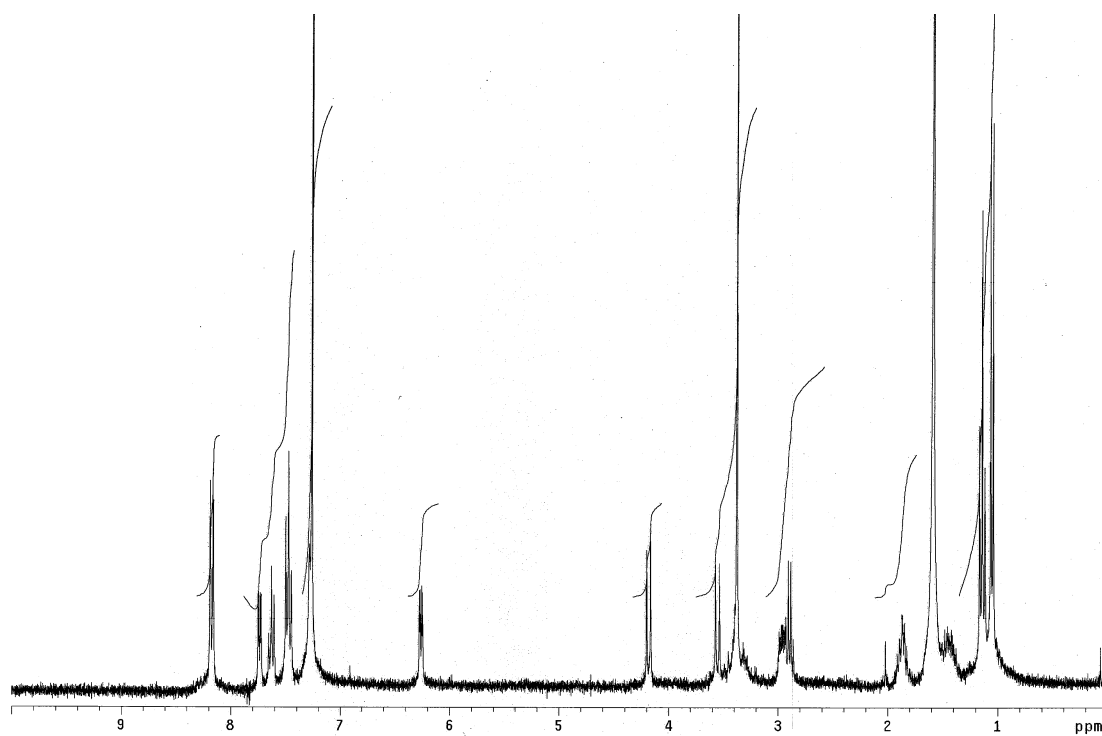


Figure S63 ^1H NMR spectrum of **7'**

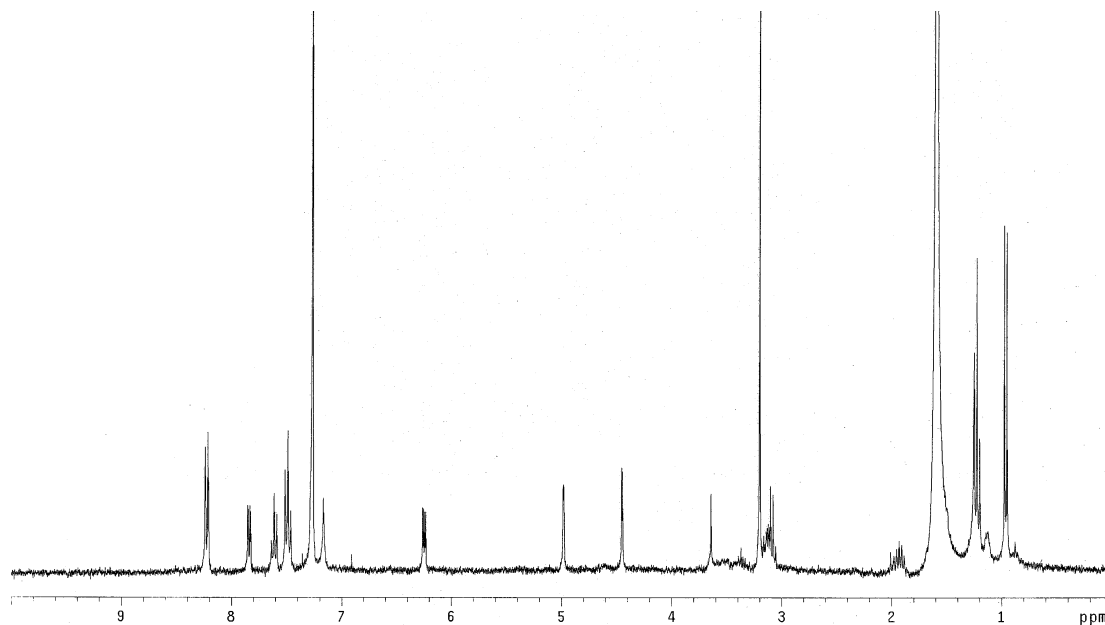
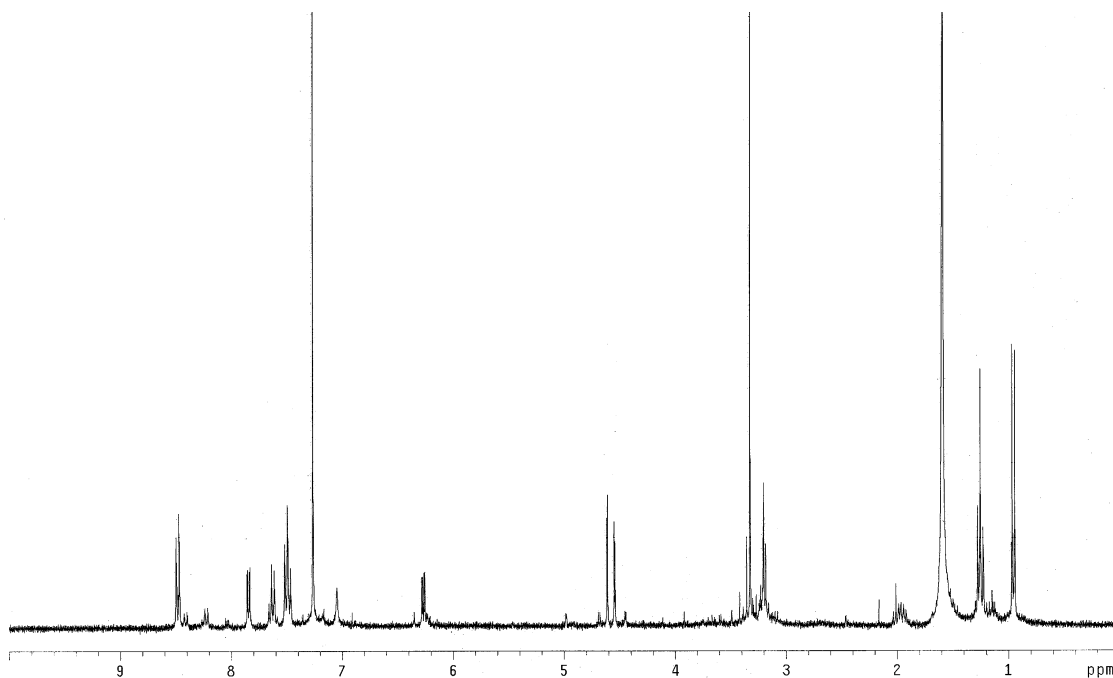
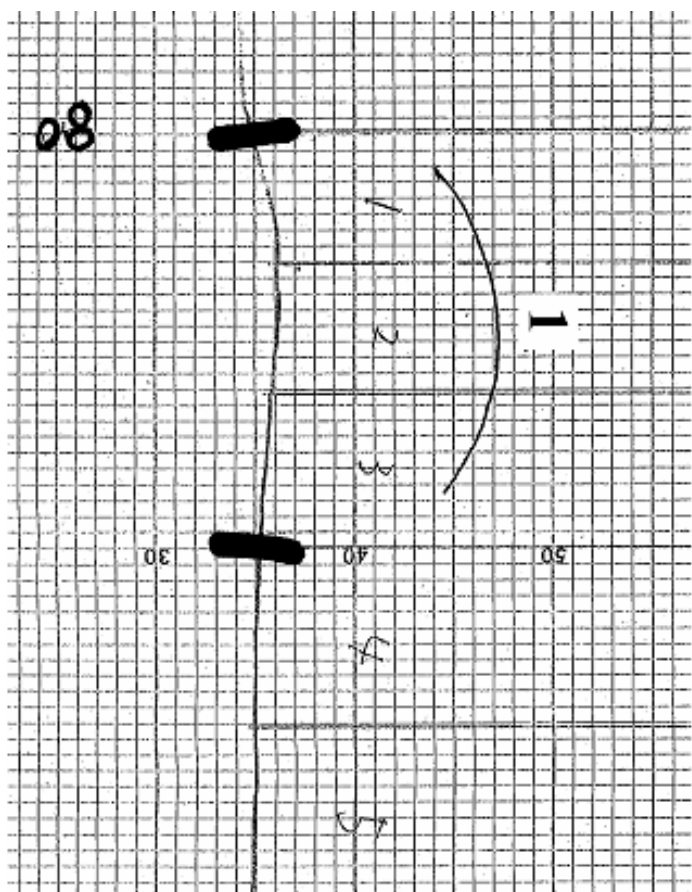


Figure S64 ^1H NMR spectrum of **8'**



cephalimysin E (1)

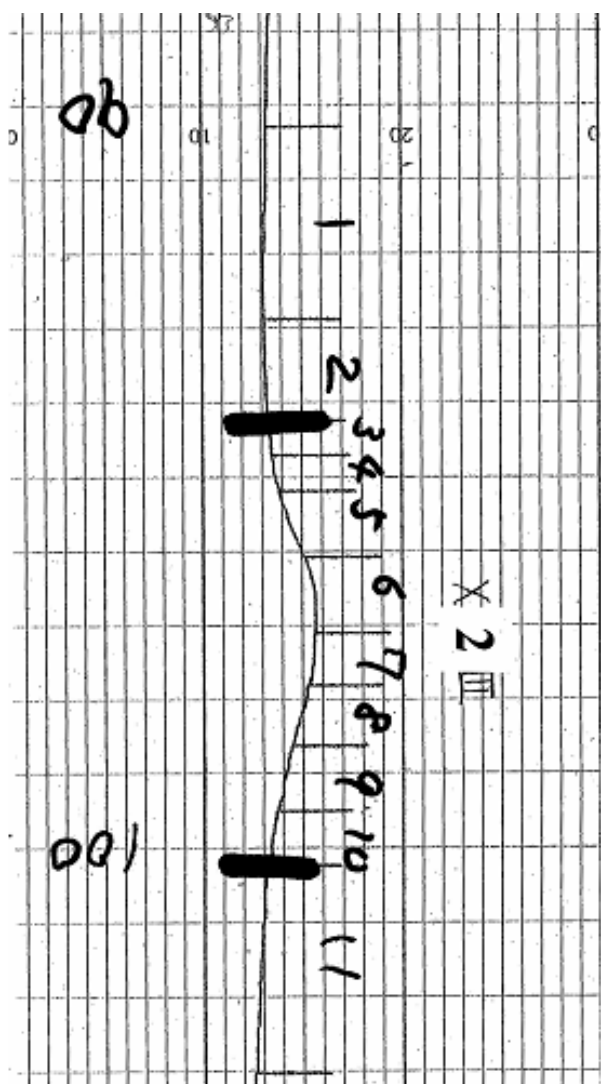
Figure S65



Mobile phase $\text{CH}_3\text{CN} / \text{H}_2\text{O} = 40 / 60$, 4ml/min

cephalimysin F (2)

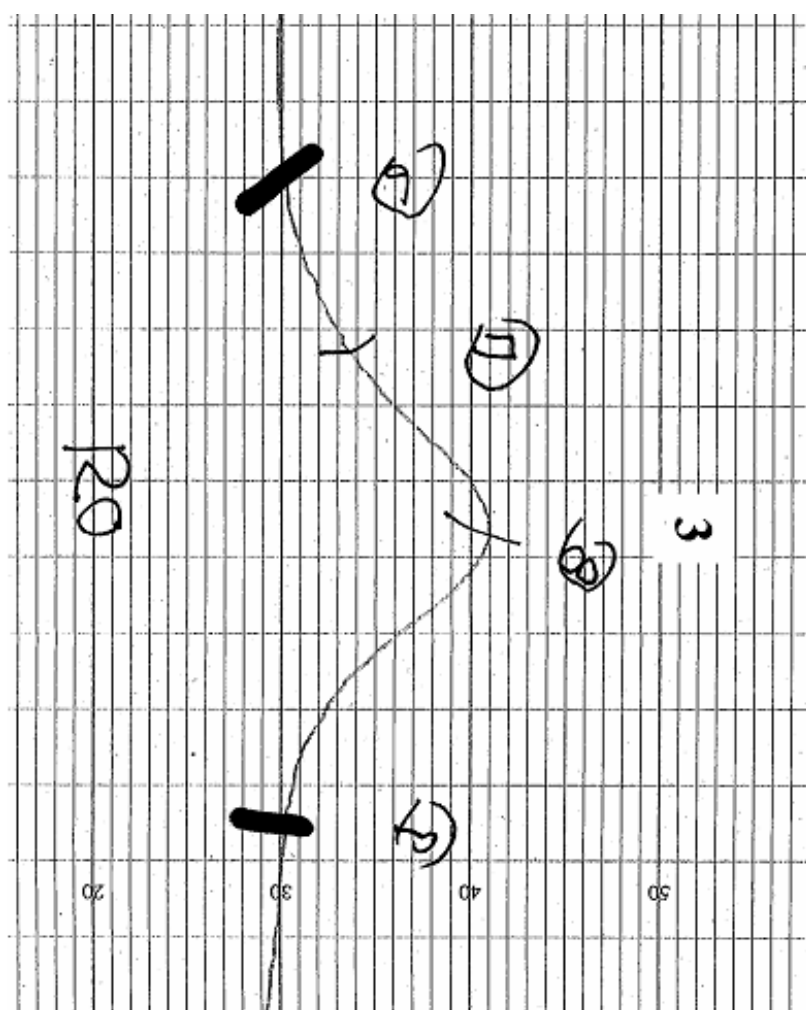
Figure S66



Mobile phase $\text{CH}_3\text{CN} / \text{H}_2\text{O} = 30 / 70$, 4ml/min

cephalimysin G (3)

Figure S67



Mobile phase $\text{CH}_3\text{CN} / \text{H}_2\text{O} = 40 / 60$, 4ml/min

cephalimysin H (4)

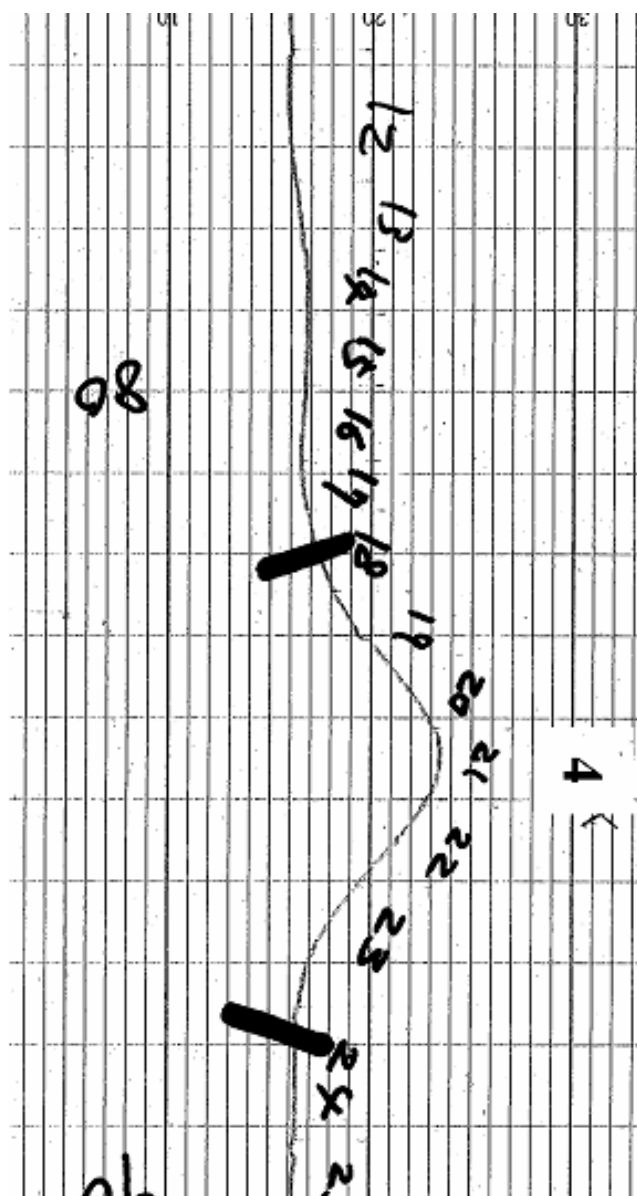
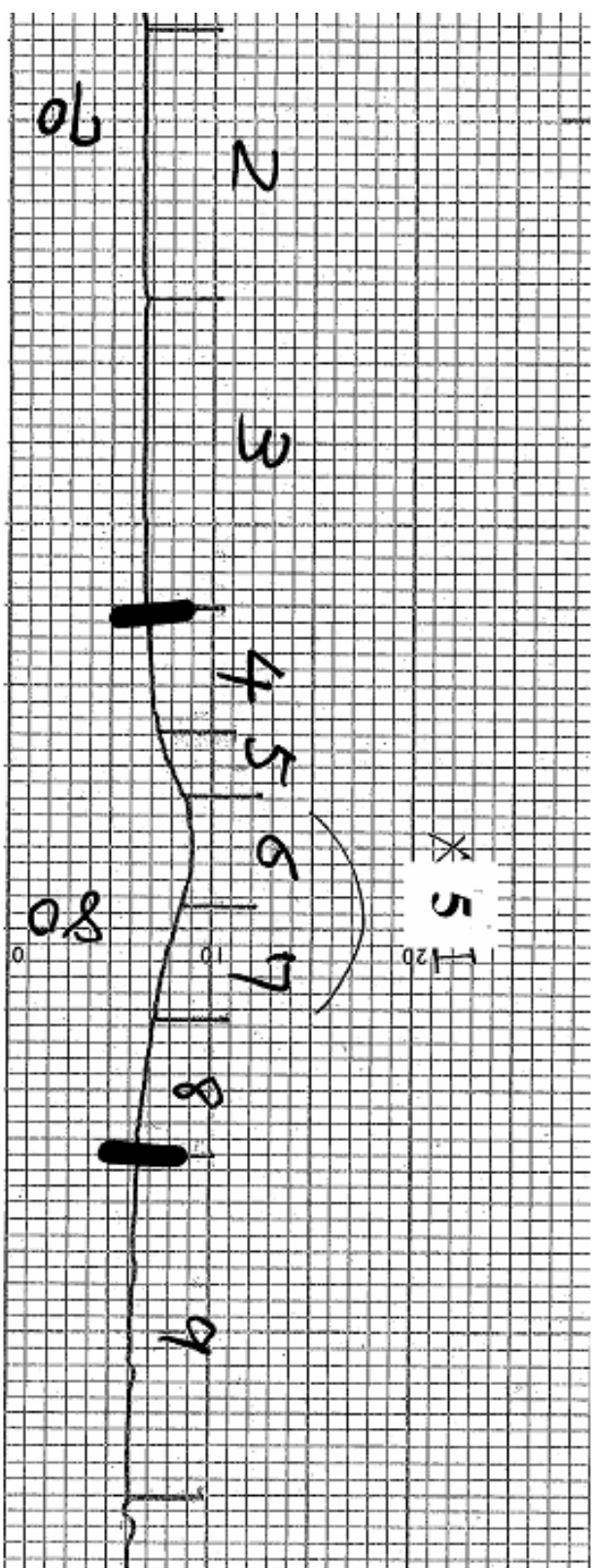


Figure S68

Mobile phase $\text{CH}_3\text{CN} / \text{H}_2\text{O} = 43 / 57$, 4ml/min

cephalimysin I (5)

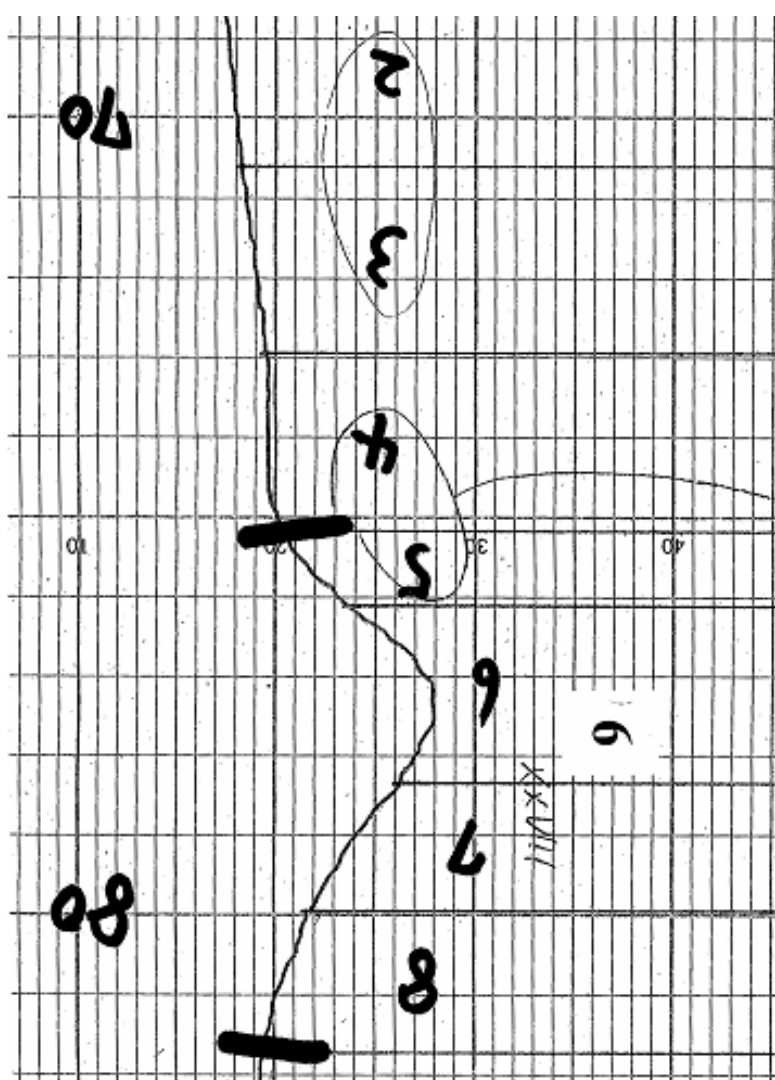
Figure S69



Mobile phase $\text{CH}_3\text{CN} / \text{H}_2\text{O} = 40 / 60$, 4ml/min

cephalimysin J(6)

Figure S70



Mobile phase $\text{CH}_3\text{CN} / \text{H}_2\text{O} = 38 / 62$, 4ml/min

cephalimysin K (7)

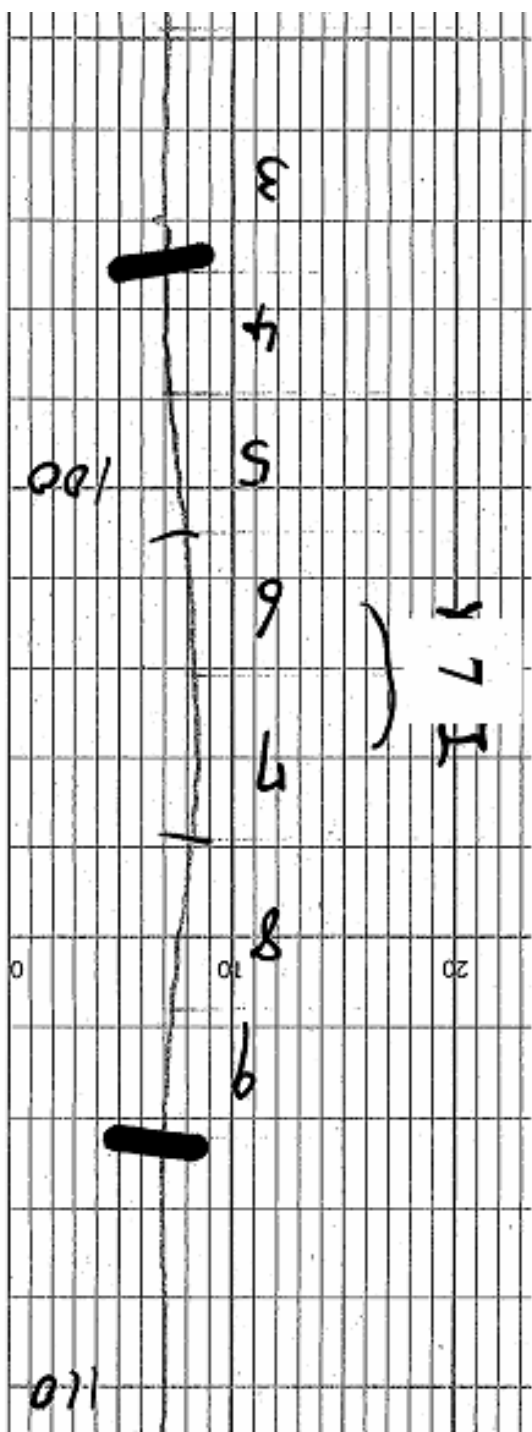
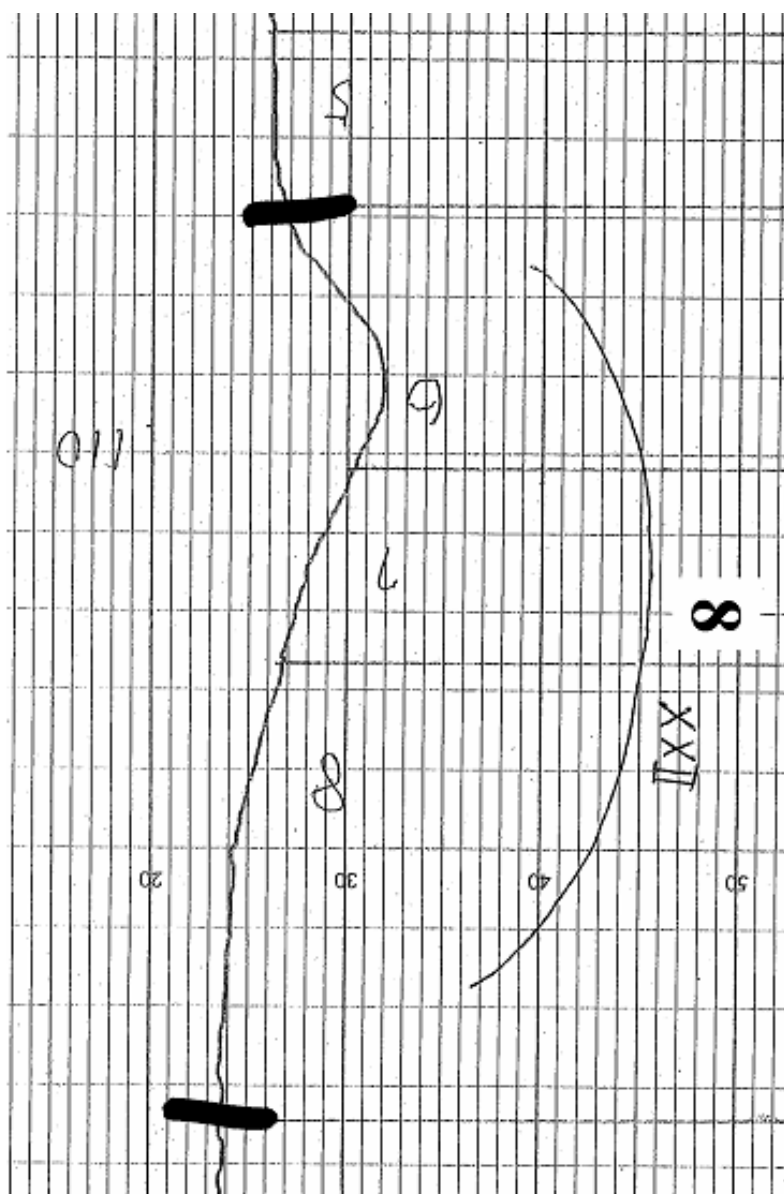


Figure S71

Mobile phase $\text{CH}_3\text{CN} / \text{H}_2\text{O} = 43 / 57$, 4ml/min

cephalimysin L (8)

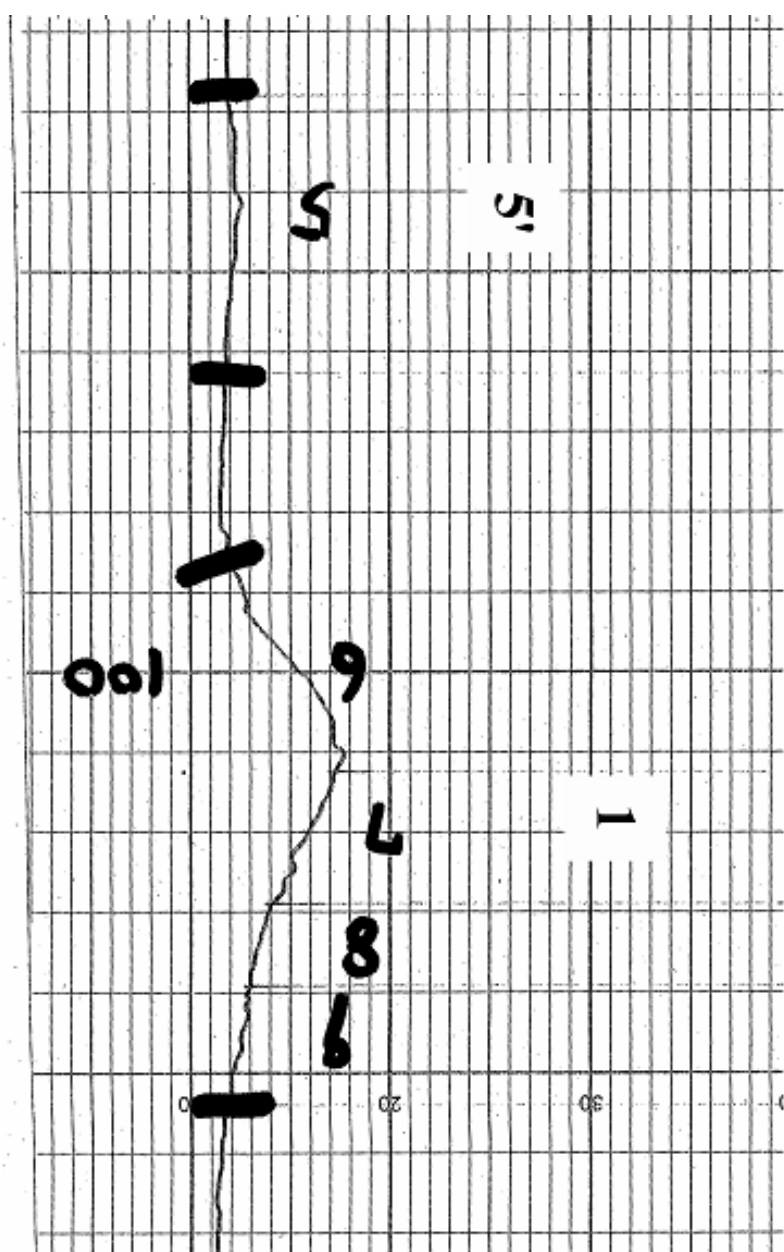


Mobile phase $\text{CH}_3\text{CN} / \text{H}_2\text{O} = 40 / 60$, 4ml/min

Figure S72

Acid treatment of 1

Figure S73



Mobile phase $\text{CH}_3\text{CN} / \text{H}_2\text{O} = 38 / 62$, 4ml/min

Acid treatment of 2

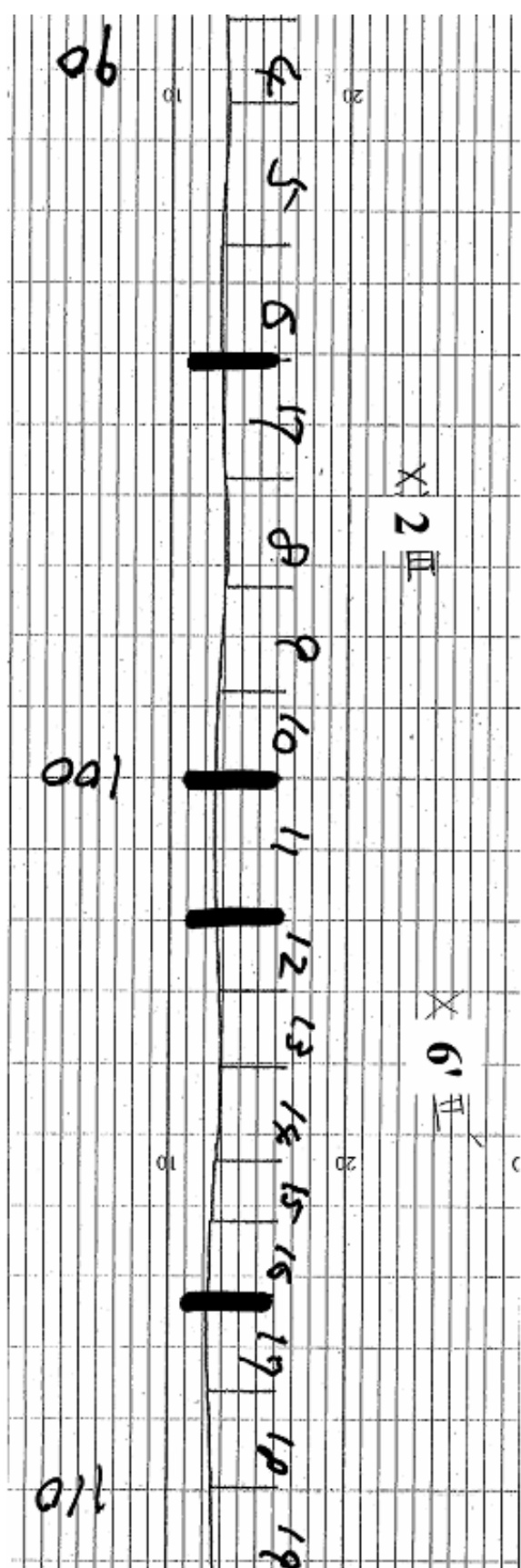
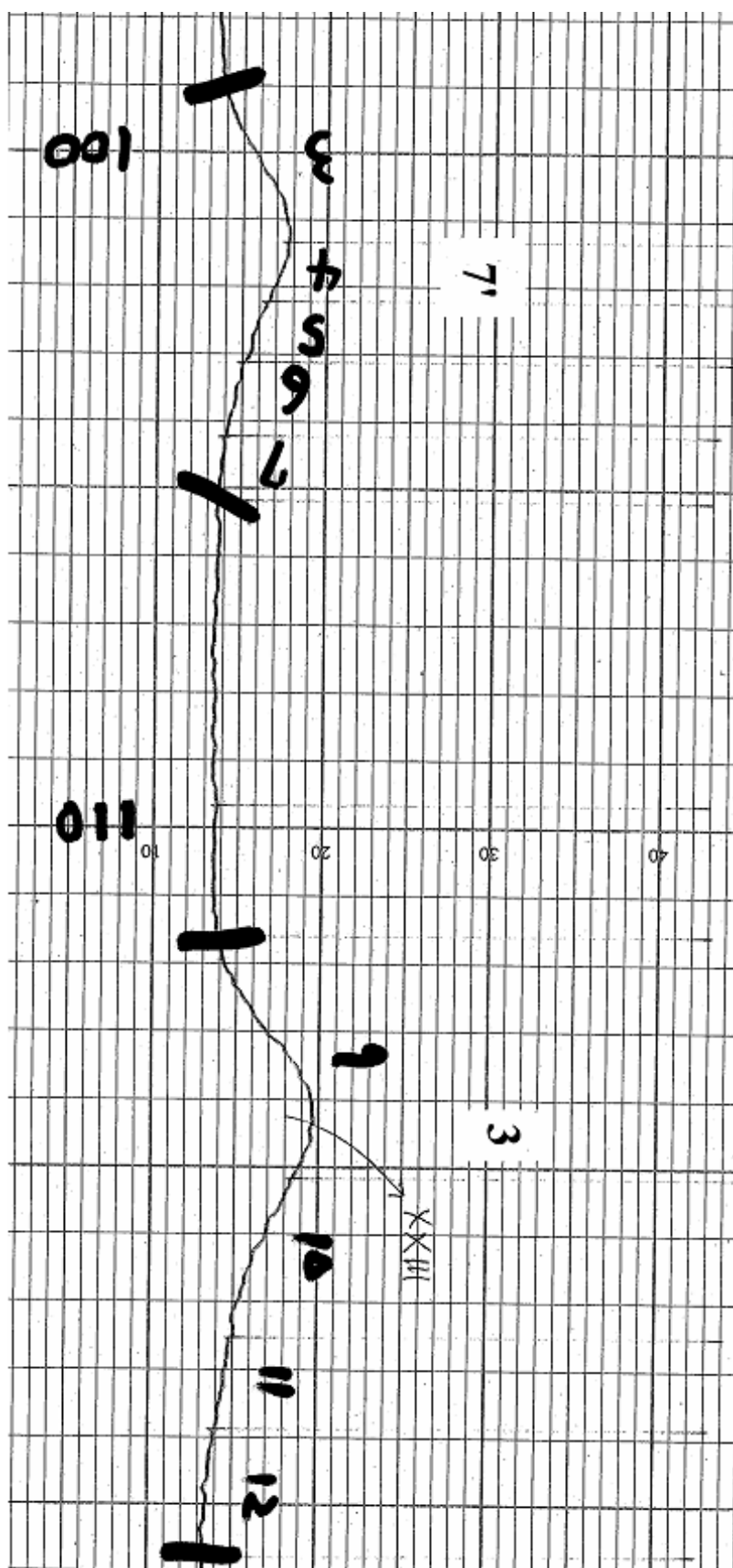


Figure S74

Mobile phase $\text{CH}_3\text{CN} / \text{H}_2\text{O} = 35 / 65$, 4ml/min

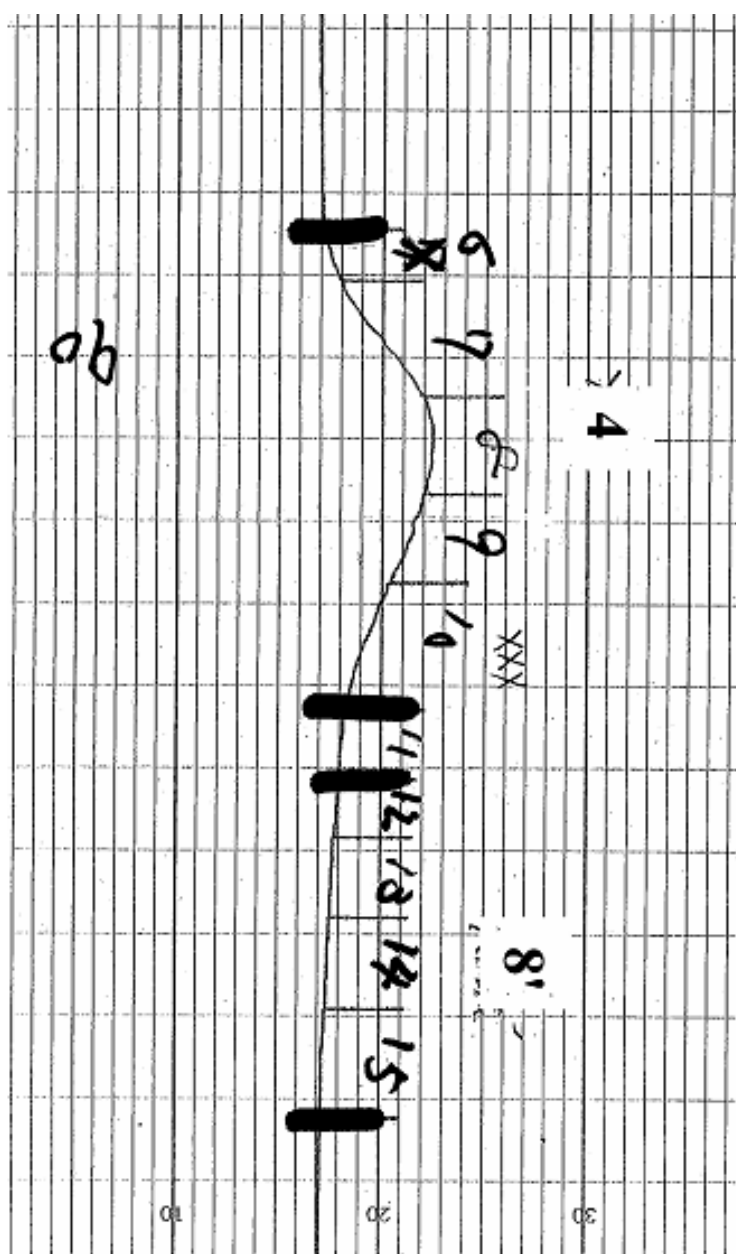
Acid treatment of 3

Figure S75



Mobile phase $\text{CH}_3\text{CN} / \text{H}_2\text{O} = 43 / 57$, 4ml/min

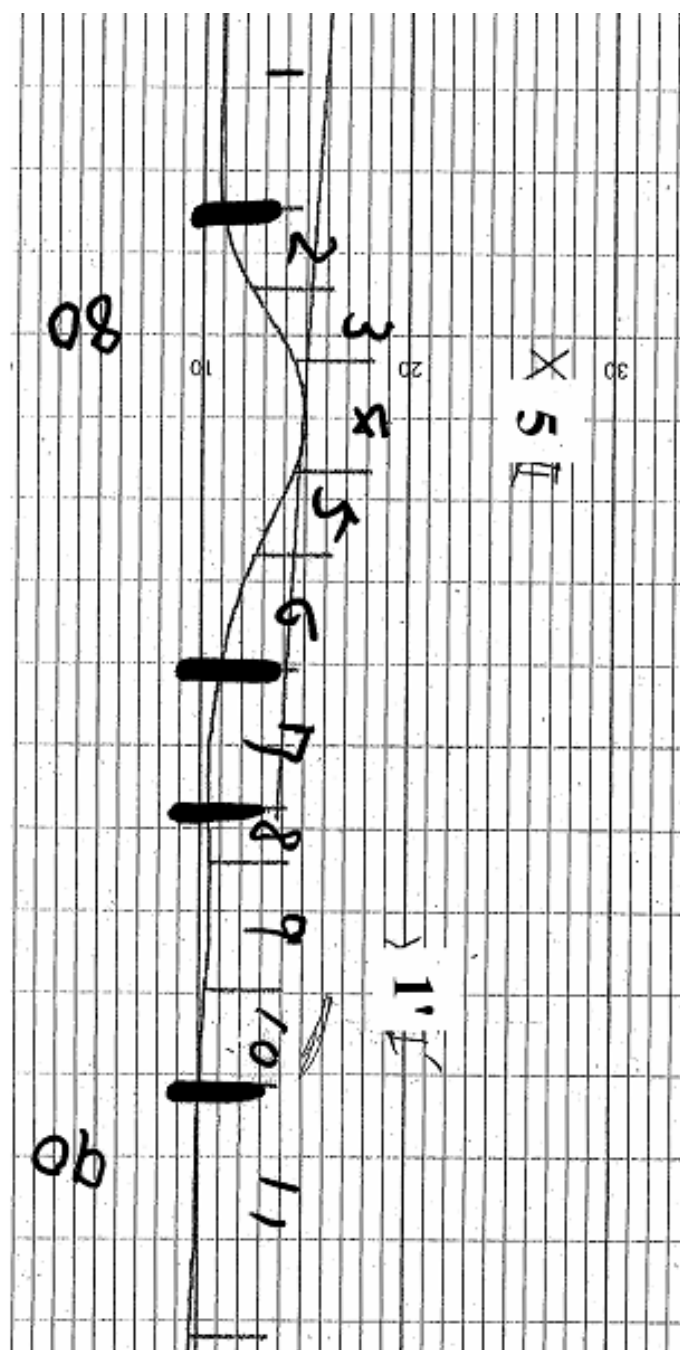
Acid treatment of 4



Mobile phase $\text{CH}_3\text{CN} / \text{H}_2\text{O} = 43 / 57$, 4ml/min

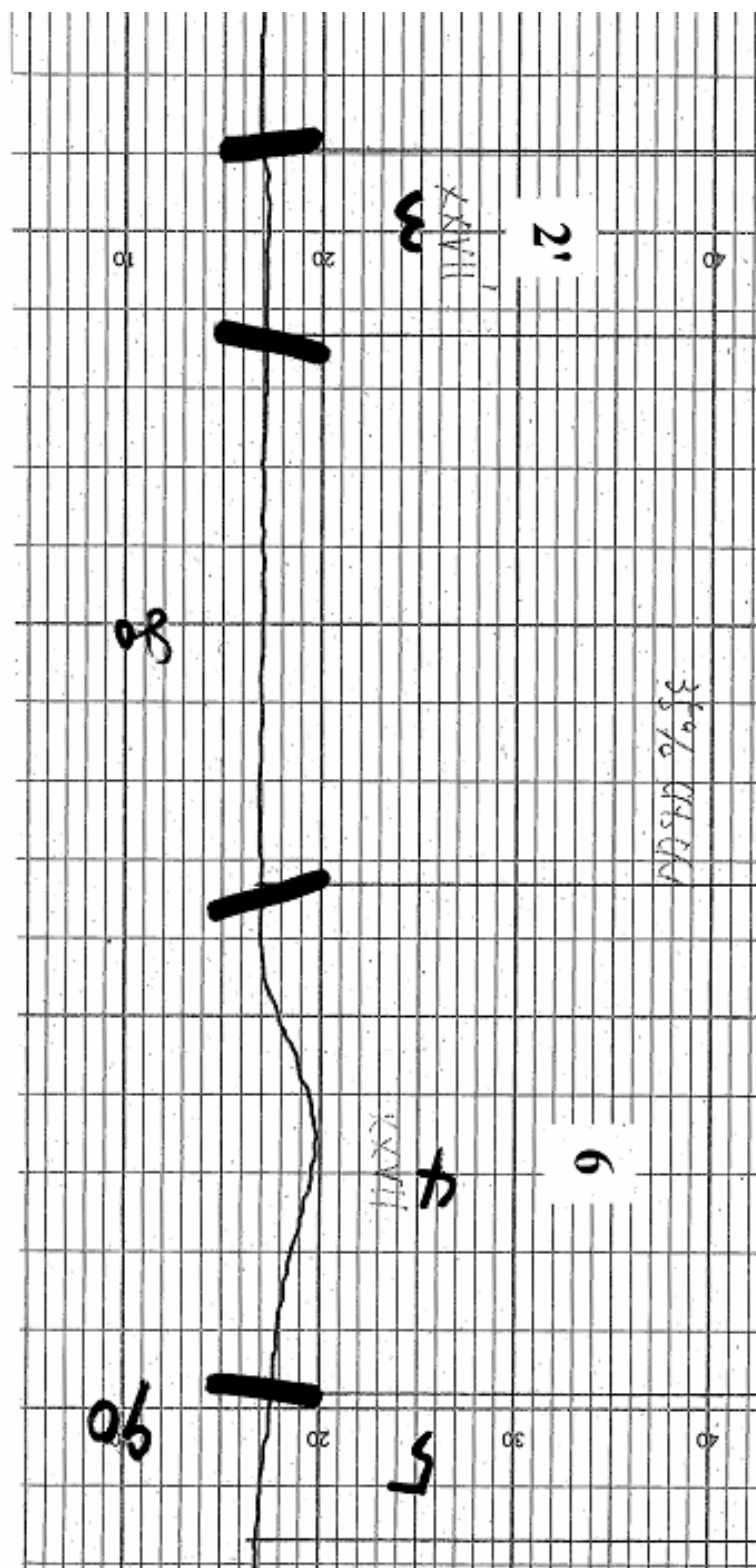
Figure S76

Acid treatment of 5



Mobile phase $\text{CH}_3\text{CN} / \text{H}_2\text{O} = 40 / 60$, 4ml/min

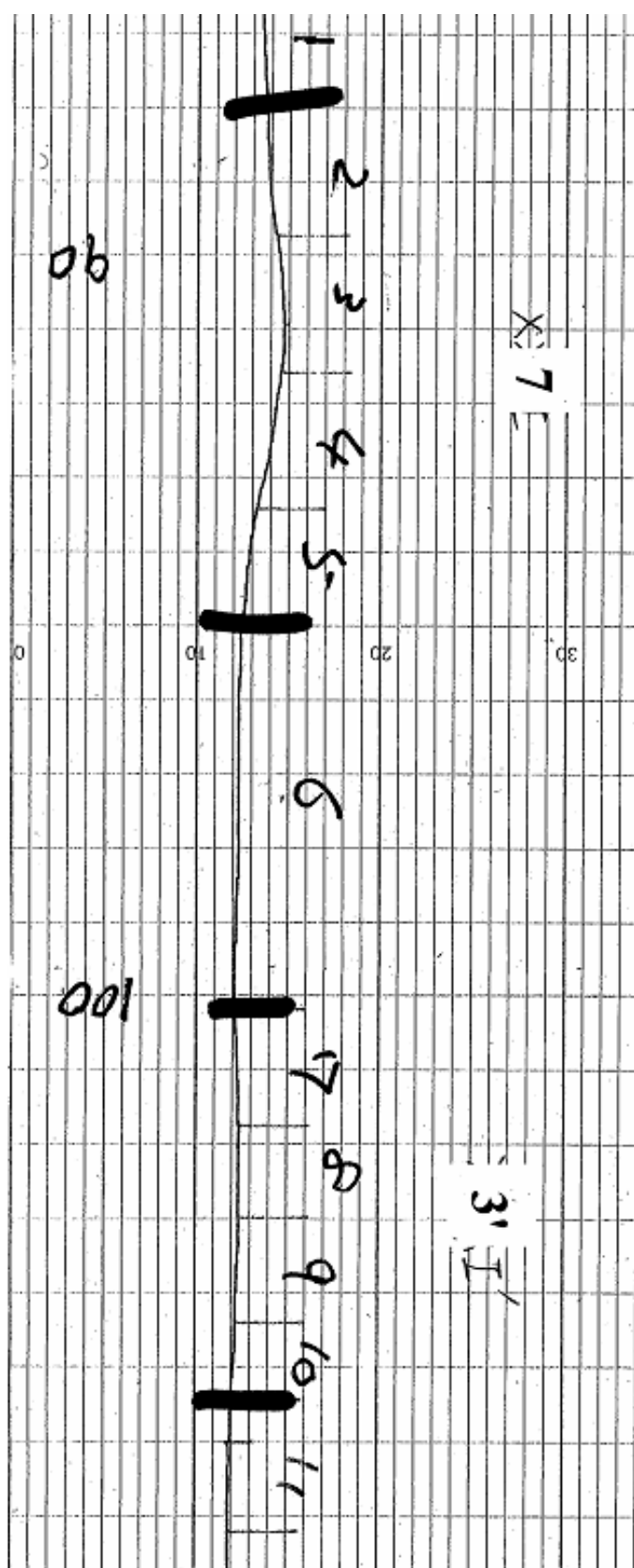
Acid treatment of 6



Mobile phase CH₃CN / H₂O = 36 / 64, 4ml/min

Acid treatment of 7

Figure S79



Mobile phase $\text{CH}_3\text{CN} / \text{H}_2\text{O} = 43 / 57$, 4ml/min

Acid treatment of 8

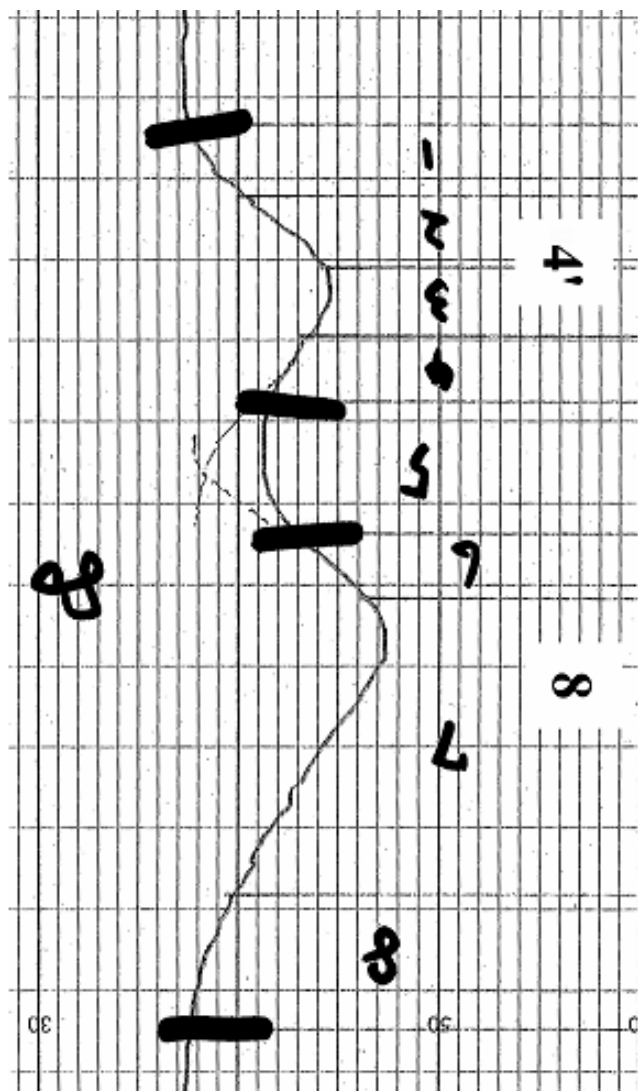


Figure S80

Mobile phase $\text{CH}_3\text{CN} / \text{H}_2\text{O} = 45 / 55$, 4ml/min

Figure S81 the CD spectra of the 16 stereoisomers 1–8 and 1'–8', symmetrical Cotton effects between enantiomers

