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 $CDCl_3$

Position	δ_H[a]		J/Hz	^{1}H-^{1}H COSY	NOE	δ_C		HMBC (C)[b]
1								
2						88.7	(s)	
3	2.68	q	6.9 (16)	16	9, 14, 15, 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	3, 19, 23, 9-OH	73.9	(d)	4, 17
10						204.0	(s)	
11	6.28	dd	6.2 (12), 2.0 (13)	12	12, 16	131.7	(d)	2, 10, 12, 13
12	7.78	dd	6.2 (11), 2.0 (13)	11, 13	11, 13, 14, 15	164.1	(d)	2, 10, 11, 13
13	3.10	ddt	12.8 (14A), 5.0 (14B), 2.0 (11, 12)	11, 12, 14	12, 14, 15, 9-OH	52.2	(d)	
14A	1.41	ddq	14.0 (14B), 12.8 (13), 7.3 (15)	13, 15	3, 12, 13, 15, 16	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	3, 12, 13, 14	12.2	(q)	13, 14
16	1.09	d	6.9 (3)	3	3, 11, 14	9.1	(q)	2, 3, 4
17						194.0	(s)	
18						133.1	(s)	
19	8.30	d	7.3 (20)	20	7, 9, 20, 8-CH_3, 9-OH	130.6	(d)	17, 21
20	7.48	t	7.3 (19, 21)	19, 21	19, 21	128.5	(d)	18, 19, 21
21	7.63	t	7.3 (20, 22)	20, 22	20, 22	134.4	(d)	19
22	7.48	t	7.3 (21, 23)	21, 23	21, 23	128.5	(d)	18, 21, 23
23	8.30	d	7.3 (22)	22	7, 9, 22, 8-CH_3, 9-OH	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, 13, 19, 23			9

a ^{1}H chemical shift values (δ ppm from $SiMe_4$) followed by multiplicity and then the coupling constants (J/Hz). Figures in parentheses indicate the proton coupling with that position. *b* Long range ^{1}H-^{13}C correlations from H to C observed in the HMBC experiment.

Table S2 NMR spectral data of **2** in $CDCl_3$

Position	δ_H[a]		J/Hz	^1H-^1H COSY	NOE	δ_C		HMBC (C)[b]
1								
2						87.2	(s)	
3	2.87	q	6.9 (16)	16	9, 14, 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	12, 13	131.5	(d)	2, 10, 13
12	7.76	dd	6.2 (11), 2.0 (13)	11, 13	11, 13, 15	164.7	(d)	10, 11, 13
13	3.09	ddt	10.8 (14A), 6.2 (14B), 2.0 (11, 12)	11, 12, 14	12, 14, 15	51.9	(d)	
14A	1.45	ddq	14.0 (14B), 10.8 (13), 7.3 (15)	13, 15	3, 13, 15, 16	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	9, 12, 13, 14	12.2	(q)	13, 14
16	1.06	d	6.9 (3)	3	3, 11, 14	8.8	(q)	2, 3, 4
17						197.0	(s)	
18						134.3	(s)	
19	8.07	d	7.3 (20)	20	9, 20, 8-OCH_3	129.0	(d)	17, 21
20	7.50	t	7.3 (19, 21)	19, 21	19, 21	128.9	(d)	18, 19, 21
21	7.63	t	7.3 (20, 22)	20, 22	20, 22	134.0	(d)	19
22	7.50	t	7.3 (21, 23)	21, 23	21, 23	128.9	(d)	18, 21, 23
23	8.07	d	7.3 (22)	22	9, 22, 8-OCH_3	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 $CDCl_3$

Position	δ_H[a]		J/Hz	^1H-^1H 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 $CDCl_3$

Position	δ_H[a]		J/Hz	^1H-^1H 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 $CDCl_3$

Position	δ_H[a]		J/Hz	^1H-^1H 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 $CDCl_3$

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 $CDCl_3$

Position	δ_H[a]		J/Hz	^1H-^1H COSY	NOE	δ_C		HMBC (C)[b]
1								
2						88.5	(s)	
3	3.09	q	6.9 (16)	16	14, 15, 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	19, 23, 9-OH	81.8	(d)	4, 17
10						209.0	(s)	
11	6.25	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	167.4	(d)	2, 10, 11, 13
13	3.14	ddt	10.8 (14A), 6.2 (14B), 2.0 (11, 12)	11, 12, 14	12, 14, 15	50.1	(d)	
14A	1.54	ddq	14.0 (14B), 10.8 (13), 7.3 (15)	13, 15	3, 12, 13, 15, 16	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	3, 12, 13, 14	12.2	(q)	13, 14
16	0.97	d	6.9 (3)	3	3, 11, 14	8.6	(q)	2, 3, 4
17						192.7	(s)	
18						133.9	(s)	
19	8.25	d	7.3 (20)	20	7, 9, 20, 8-OCH_3, 9-OH	129.7	(d)	17, 21
20	7.48	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	7.48	t	7.3 (21, 23)	21, 23	21, 23	128.6	(d)	18, 21, 23
23	8.25	d	7.3 (22)	22	7, 9, 22, 8-OCH_3, 9-OH	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, 19, 23			9

a, *b* As in Table S1

Table S8 NMR spectral data of **8** in $CDCl_3$

Position	δ_H[a]		J/Hz	^{1}H-^{1}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 ^{1}H and ^{13}C NMR spectra of **1** in $CDCl_3$

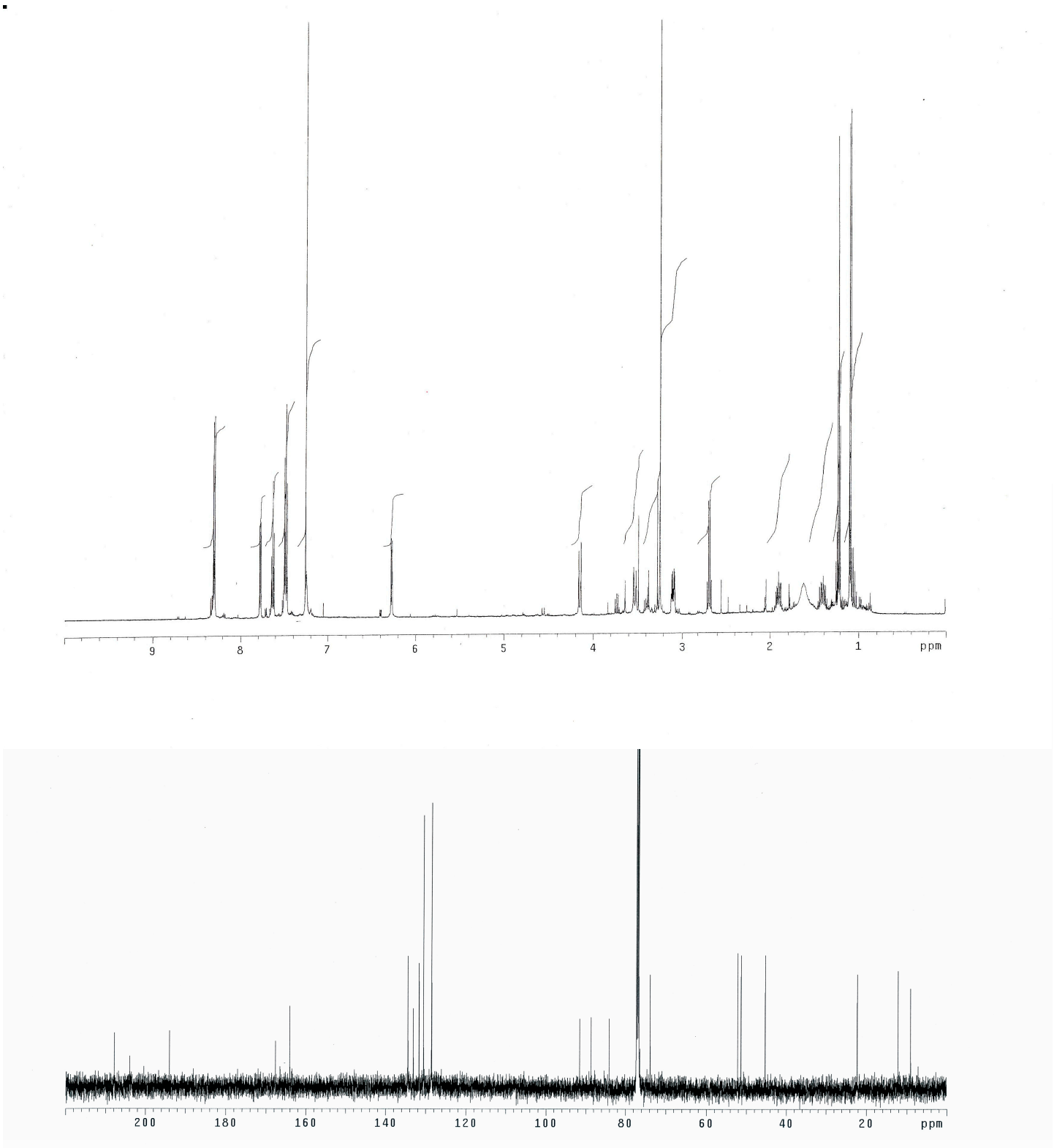
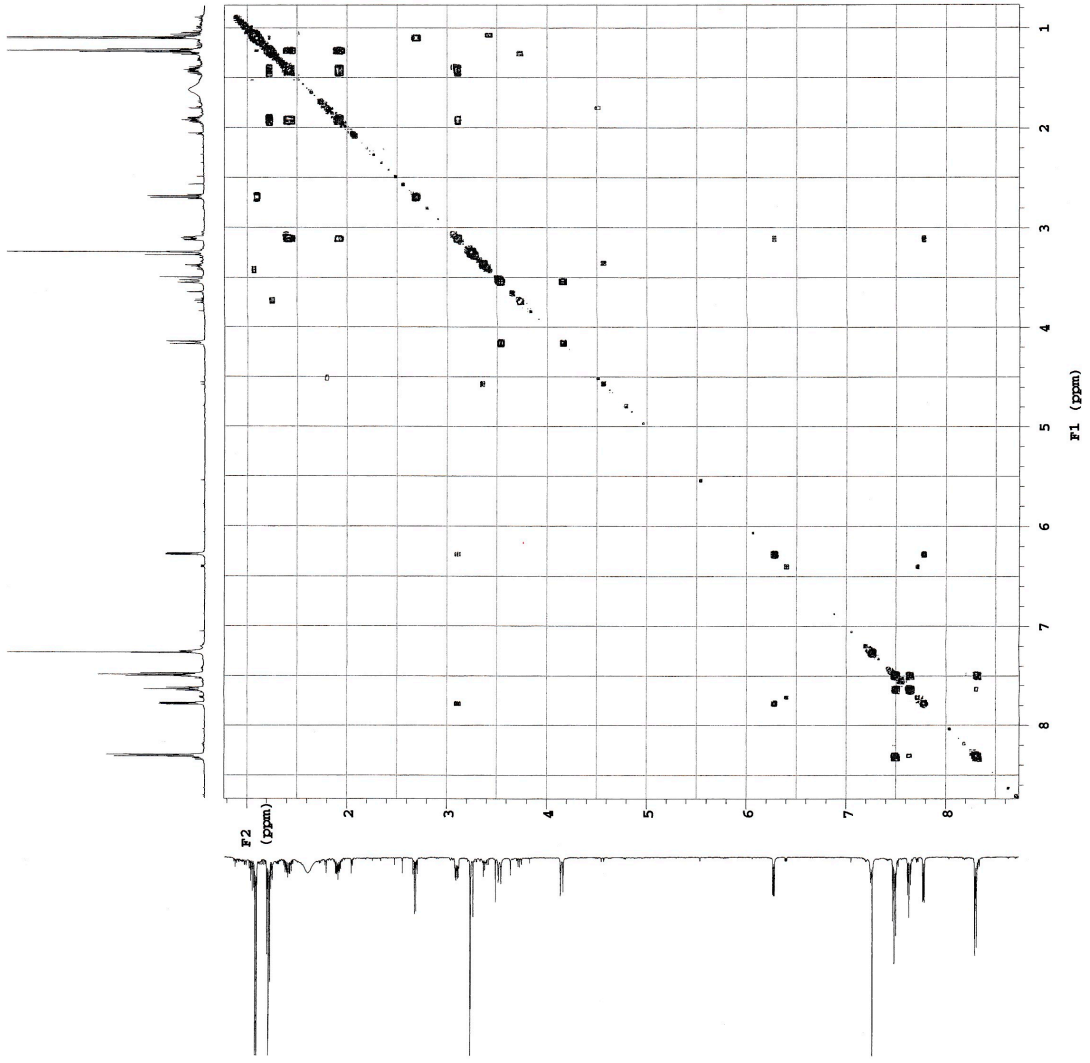


Figure S2 ^{1}H-^{1}H COSY of **1**



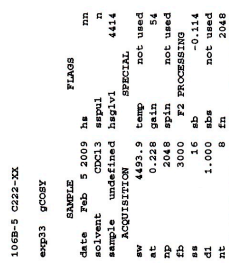
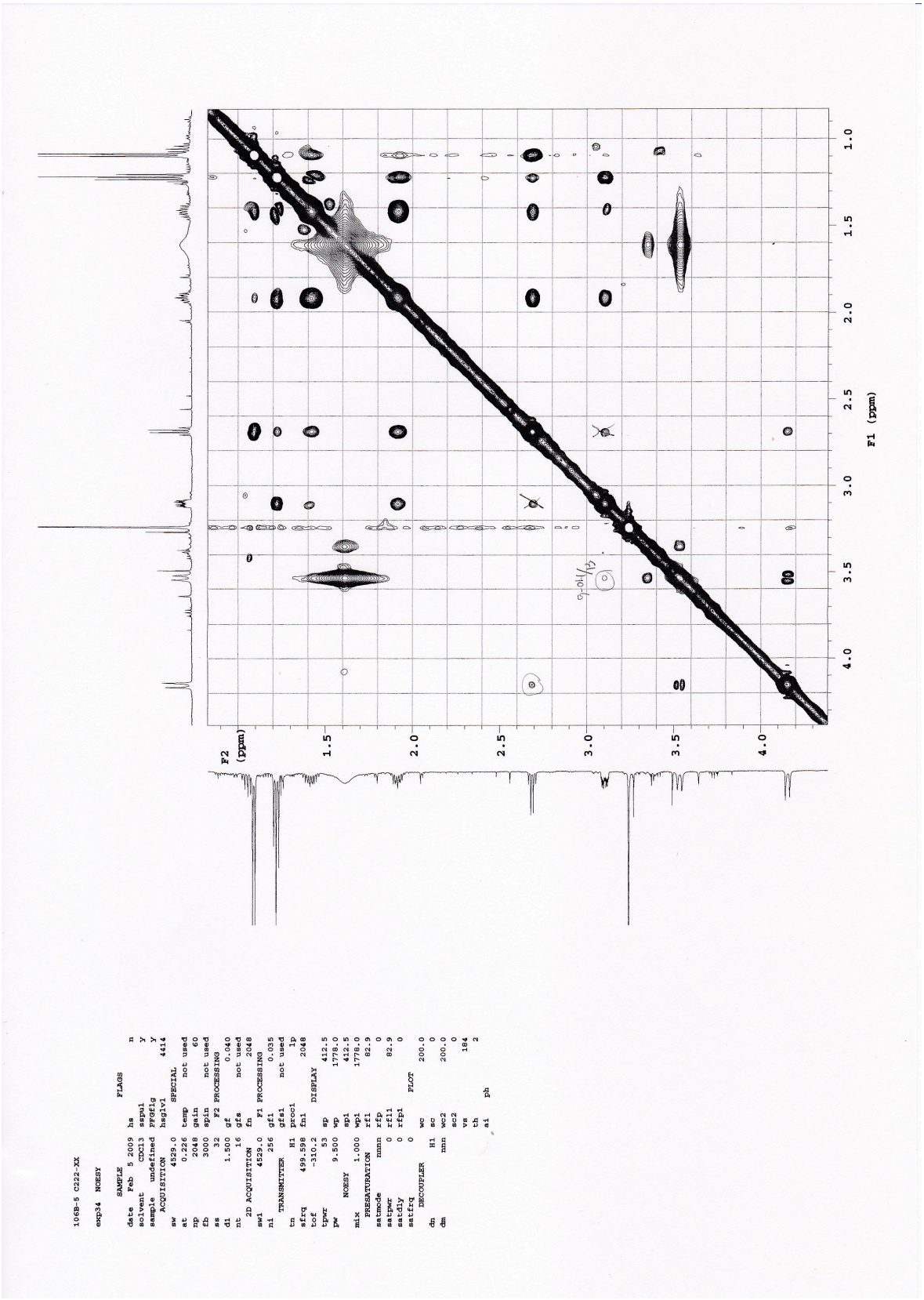
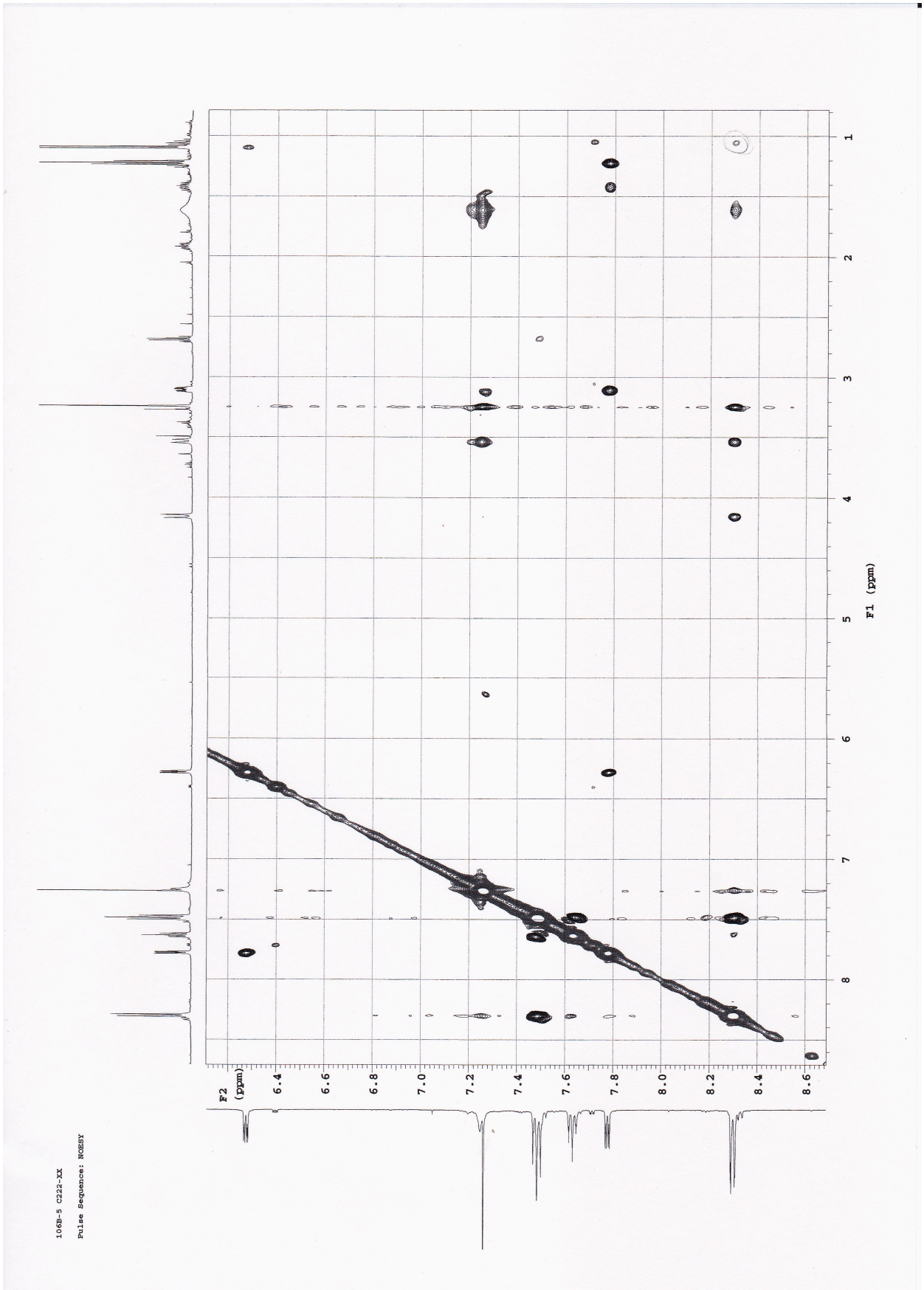


Figure S3 NOESY of **1**

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6.6
6.8
7.0
7.2
7.4
7.6
7.8
8.0
8.2
8.4
8.6
F1 (ppm)
1
2
3
4
5
6
7
8

Figure S4 HMQC of **1**

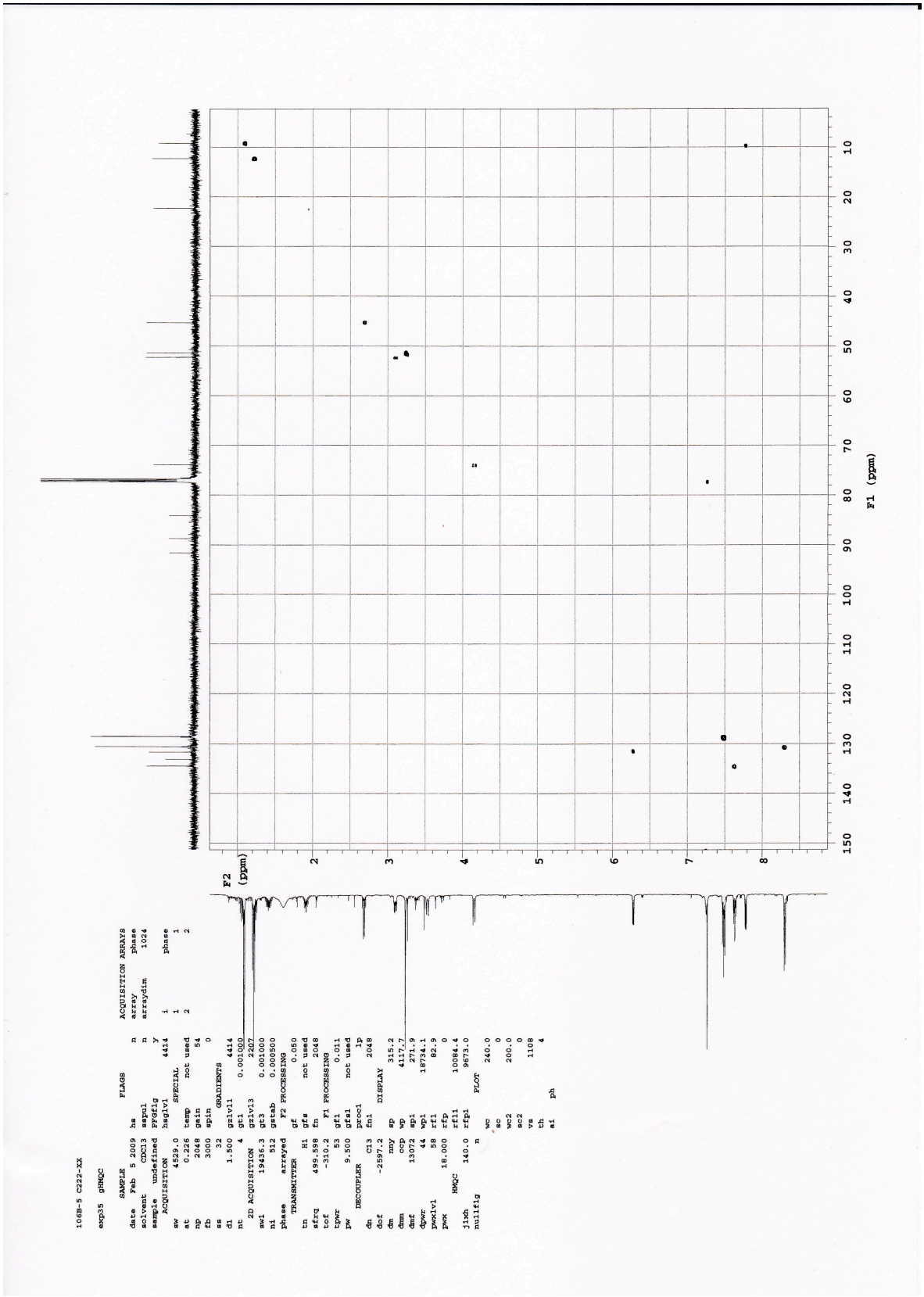


Figure S5 HMBC of **1**

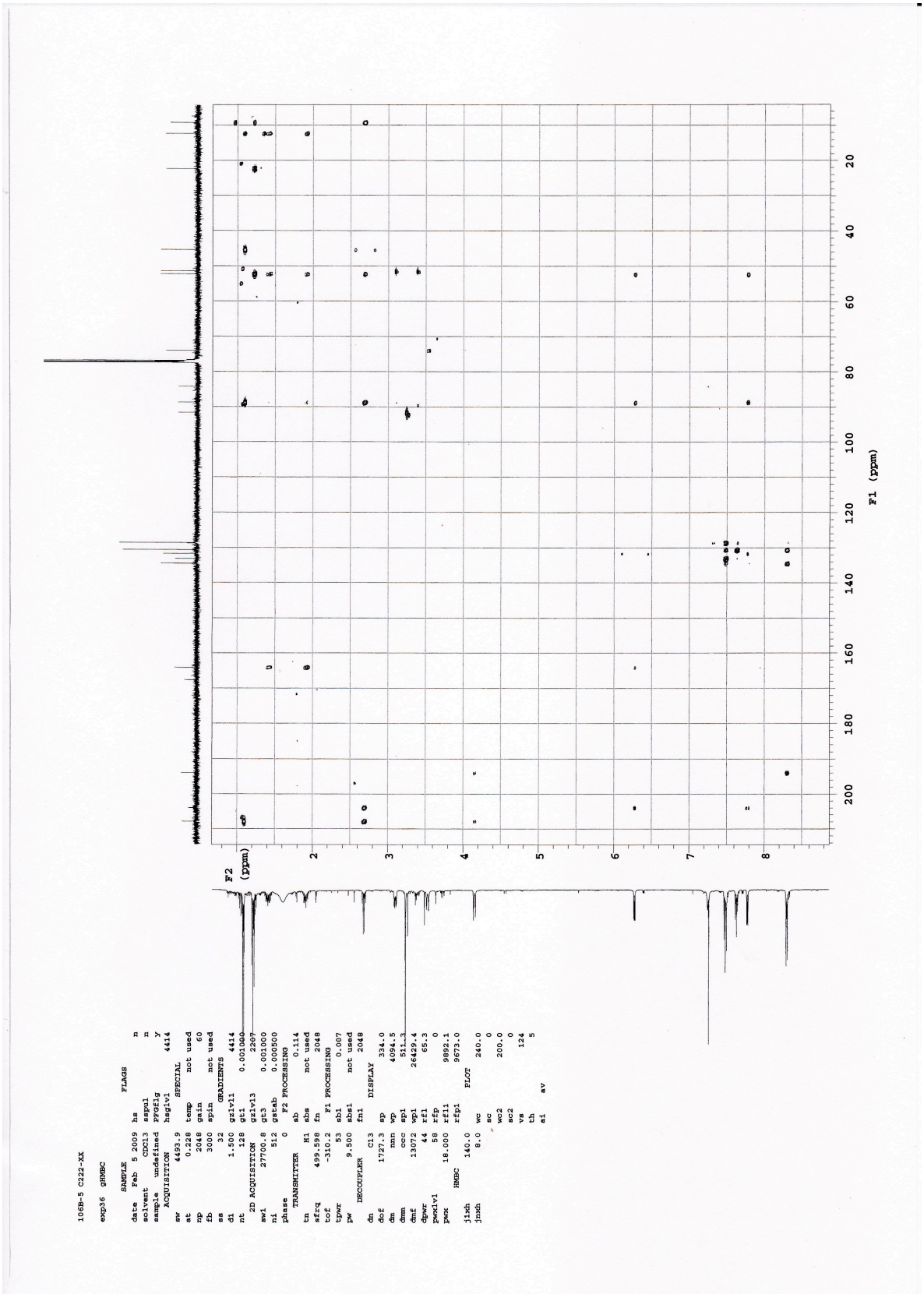


Figure S6 IR spectrum of **1**

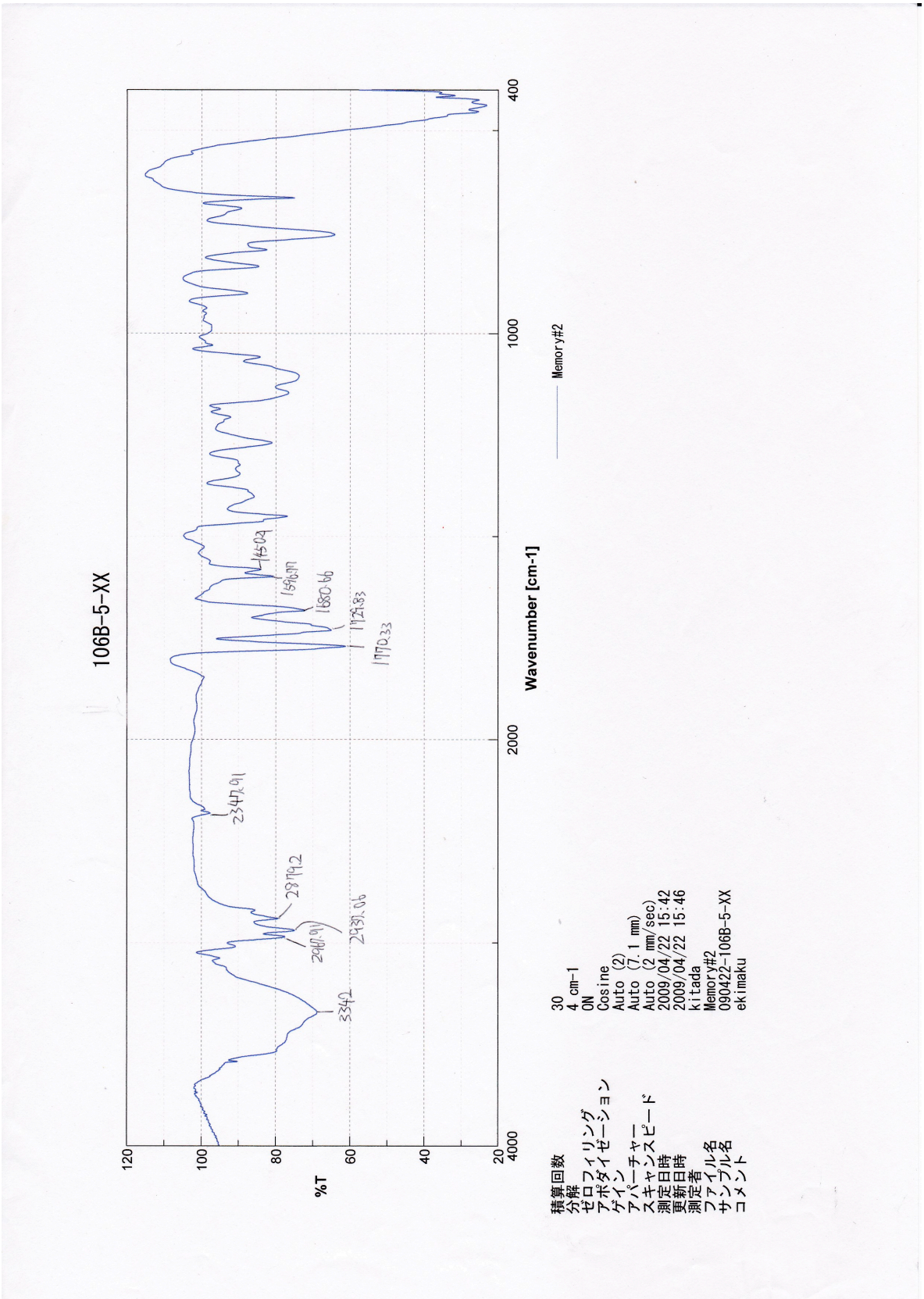


Figure S7 FABMS of **1**

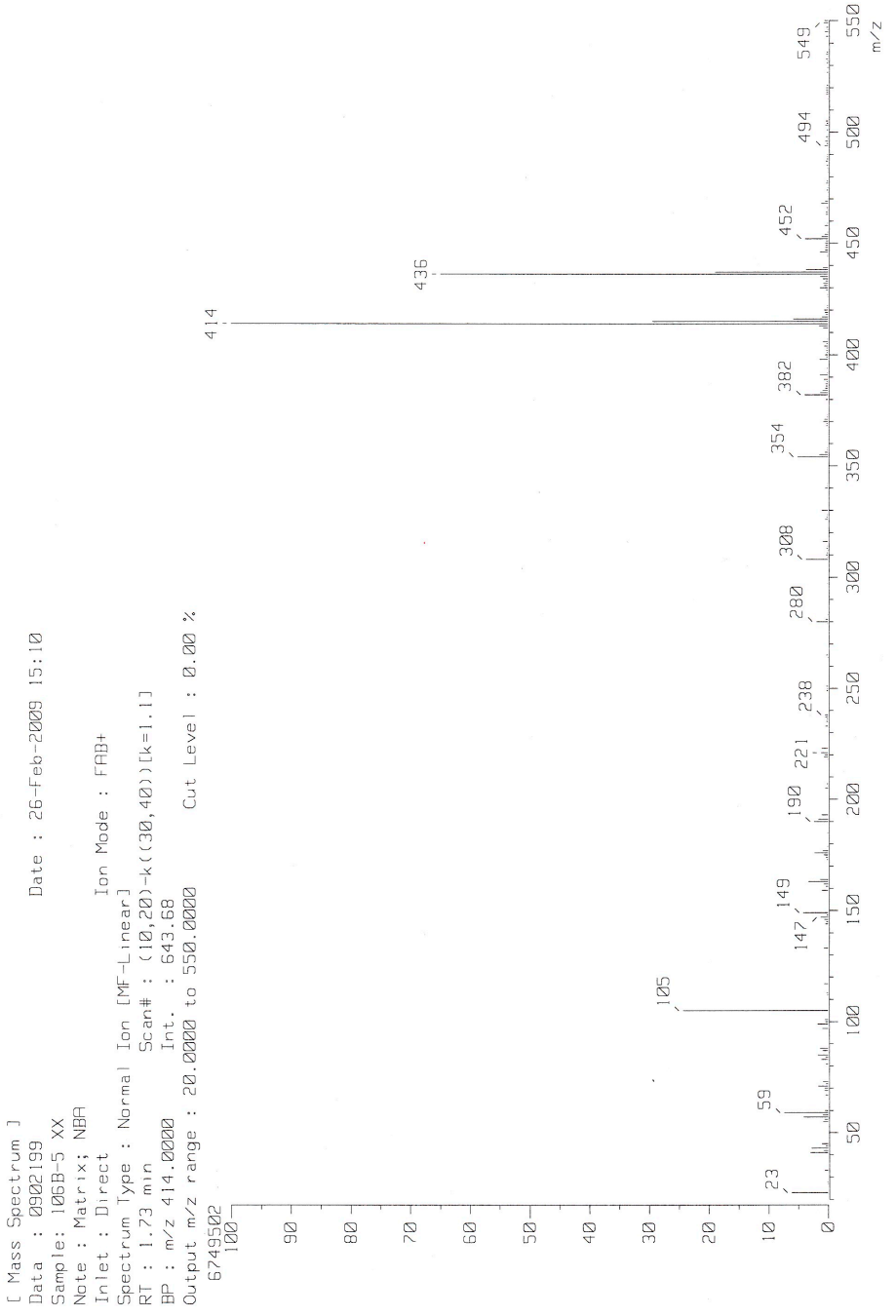


Figure S8 ^{1}H and ^{13}C NMR spectra of **2** in $CDCl_3$

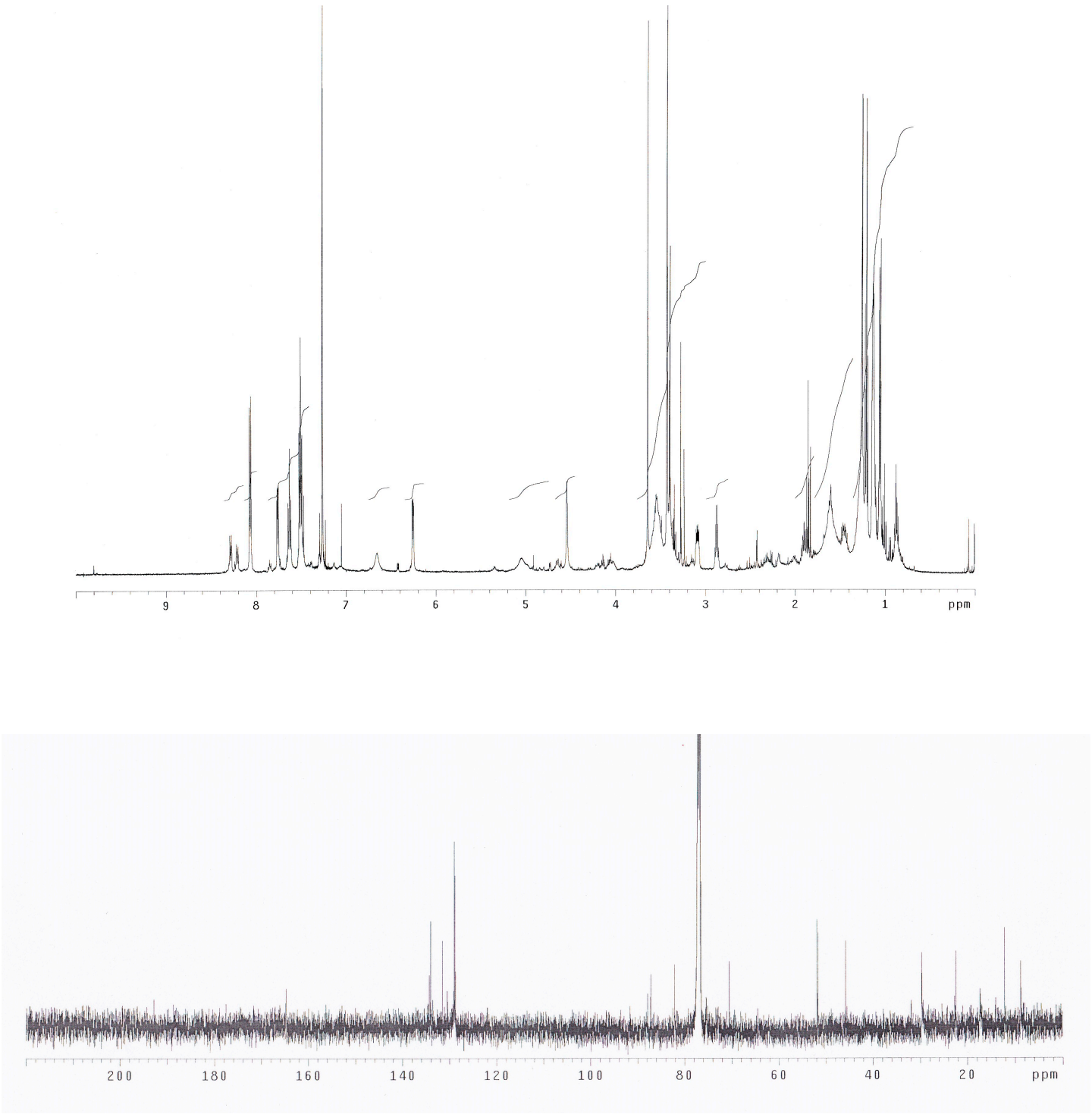


Figure S9 H-^{1}H COSY of **2**

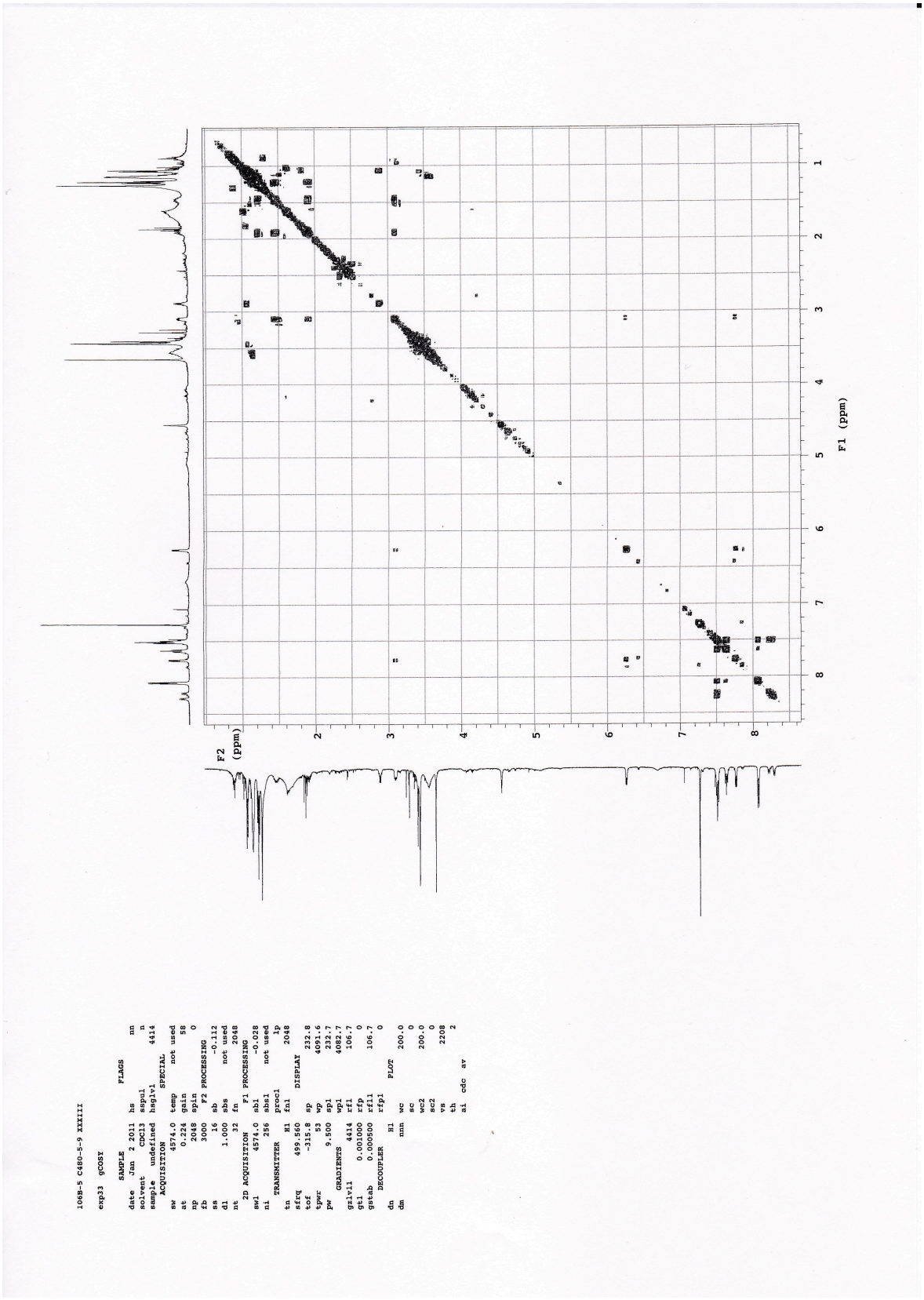


Figure S10 NOESY of **2**

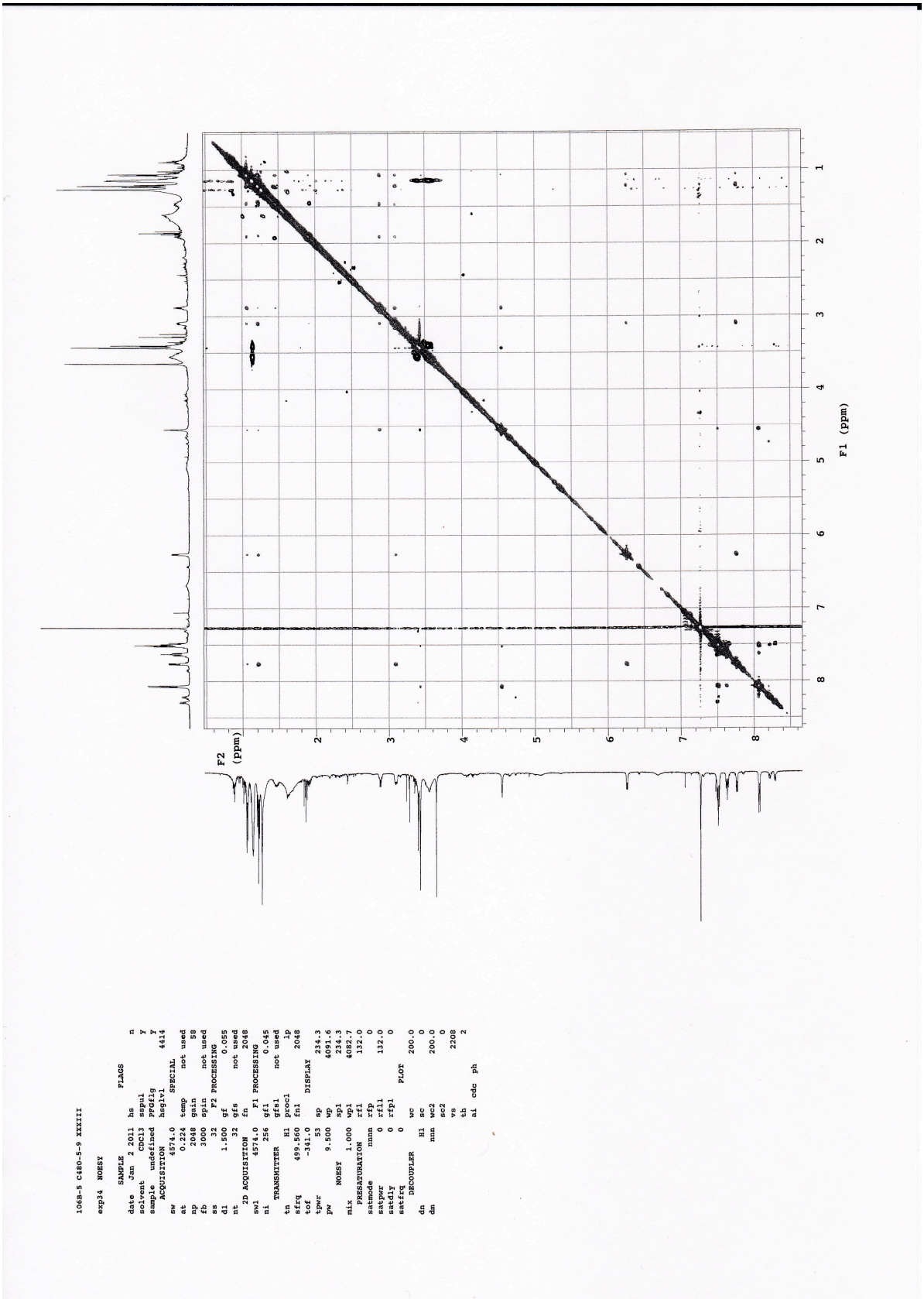


Figure S11 HMQC of **2**

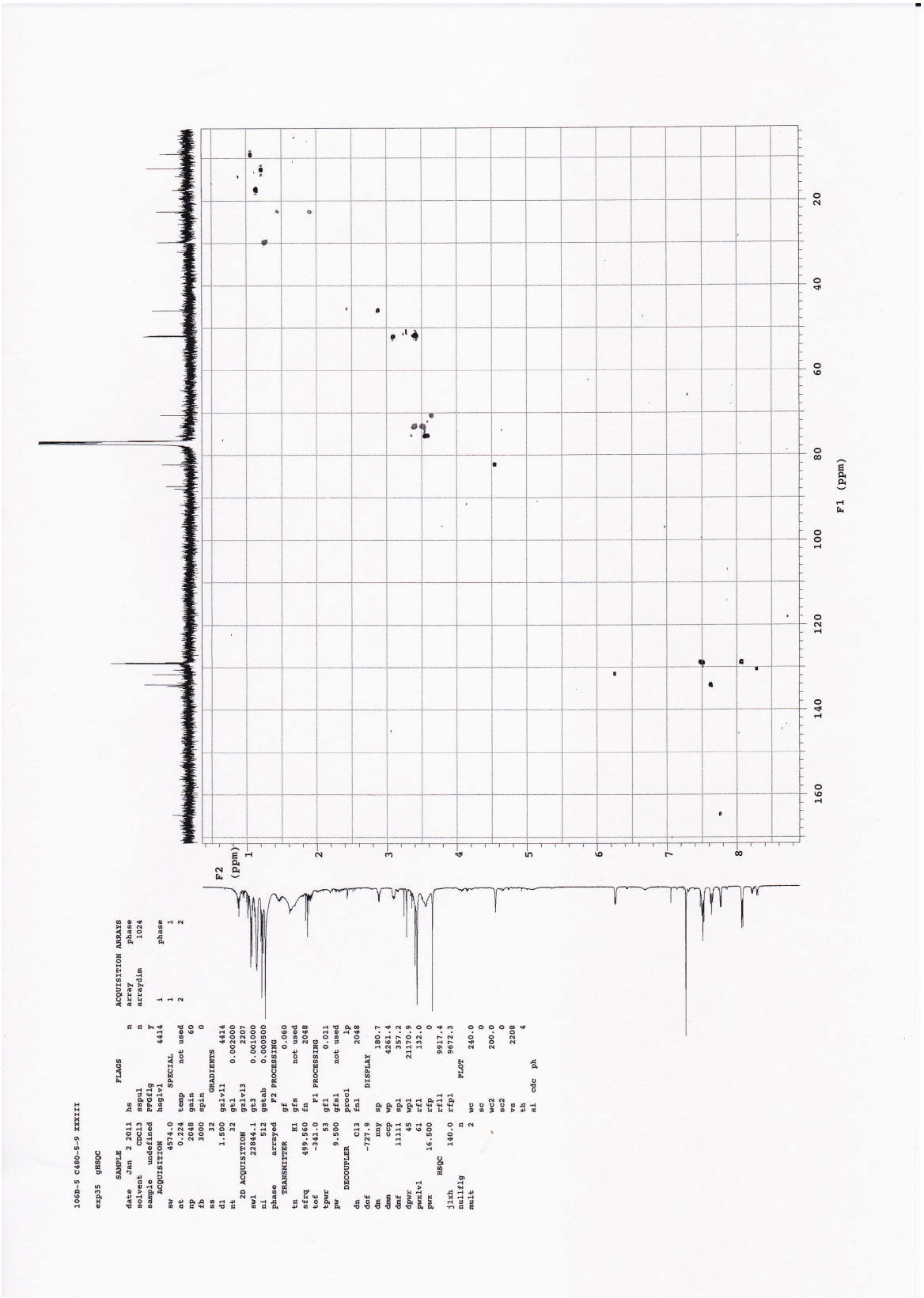


Figure S12 HMBC of **2**

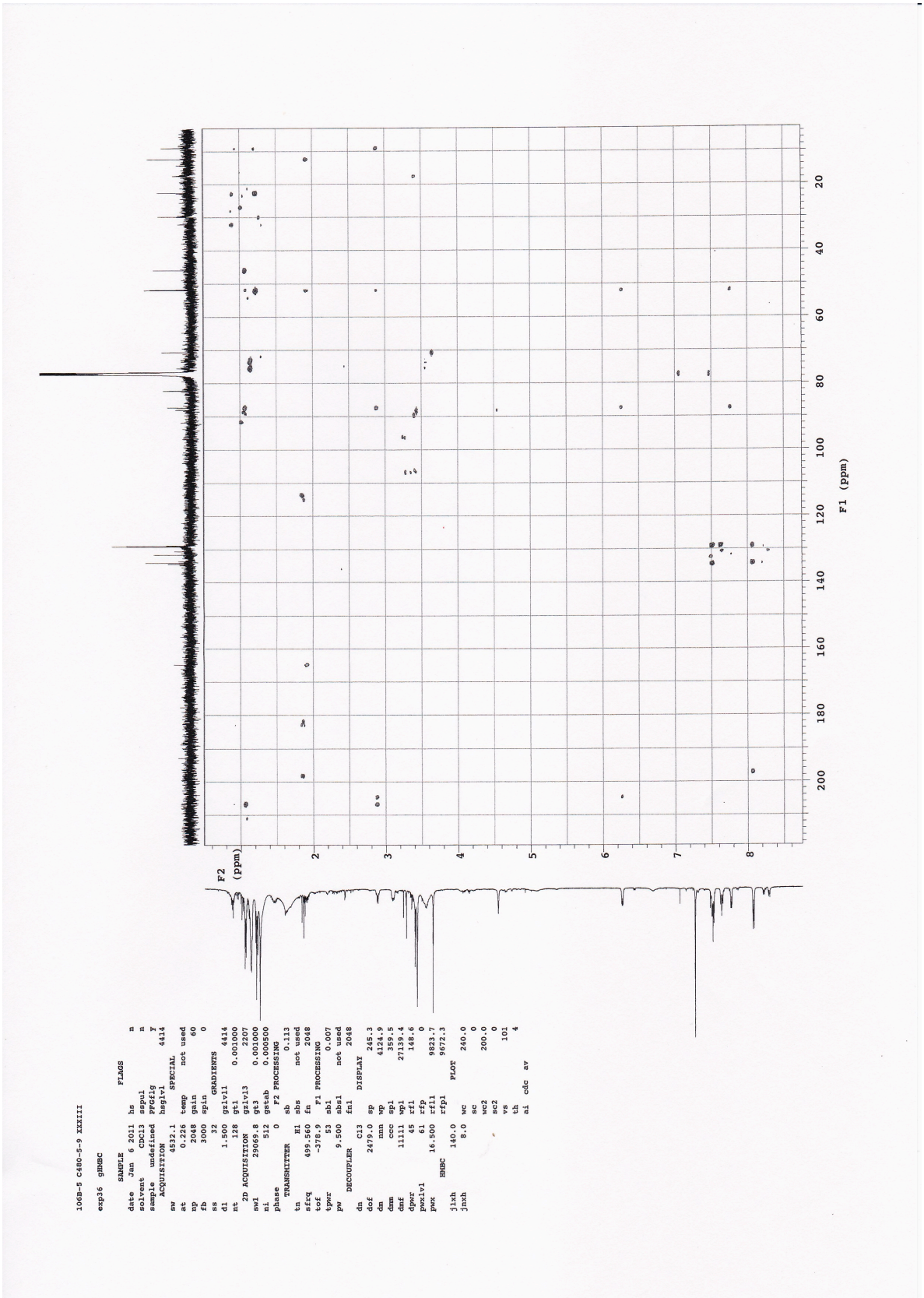


Figure S13 IR spectrum of **2**

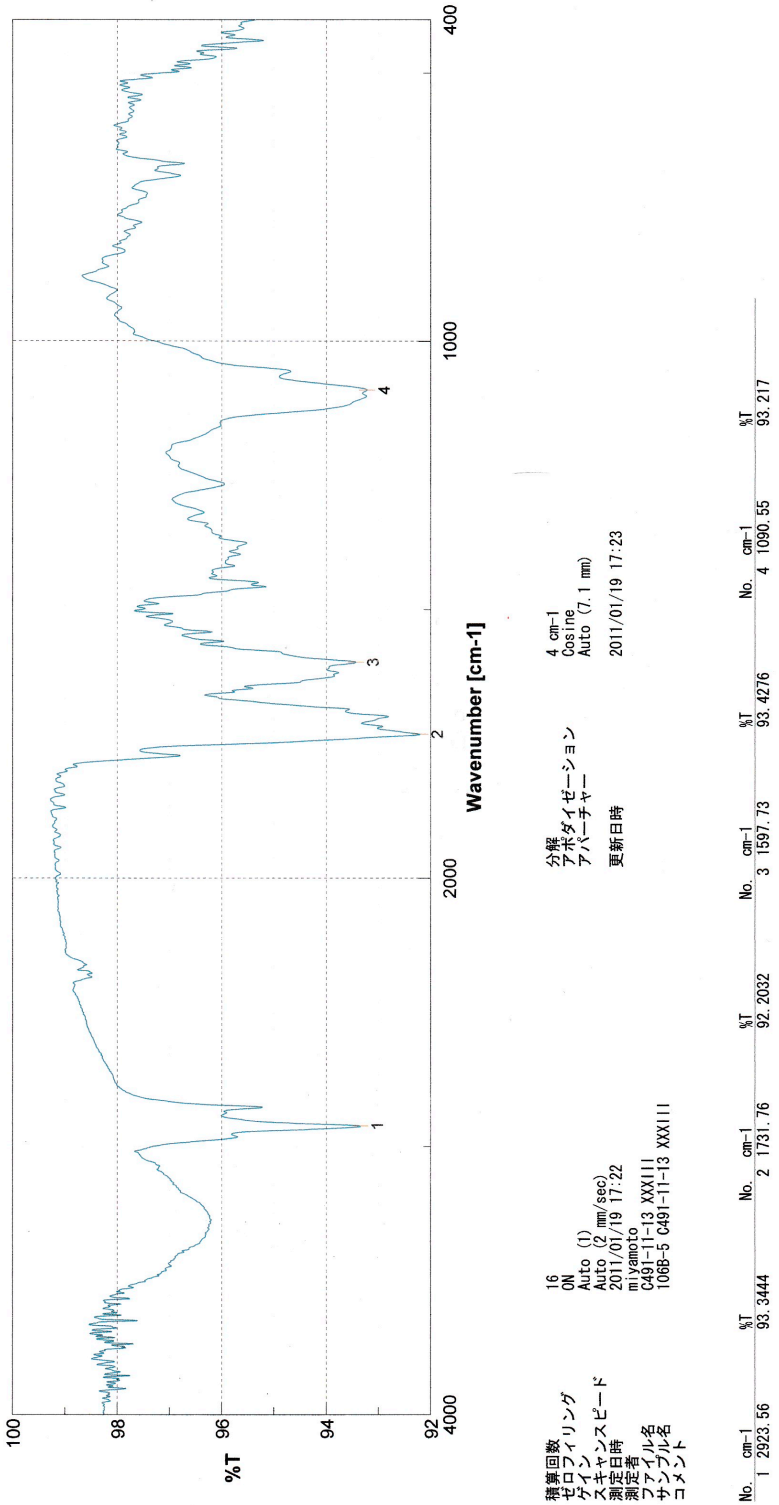


Figure S14 FABMS of **2**

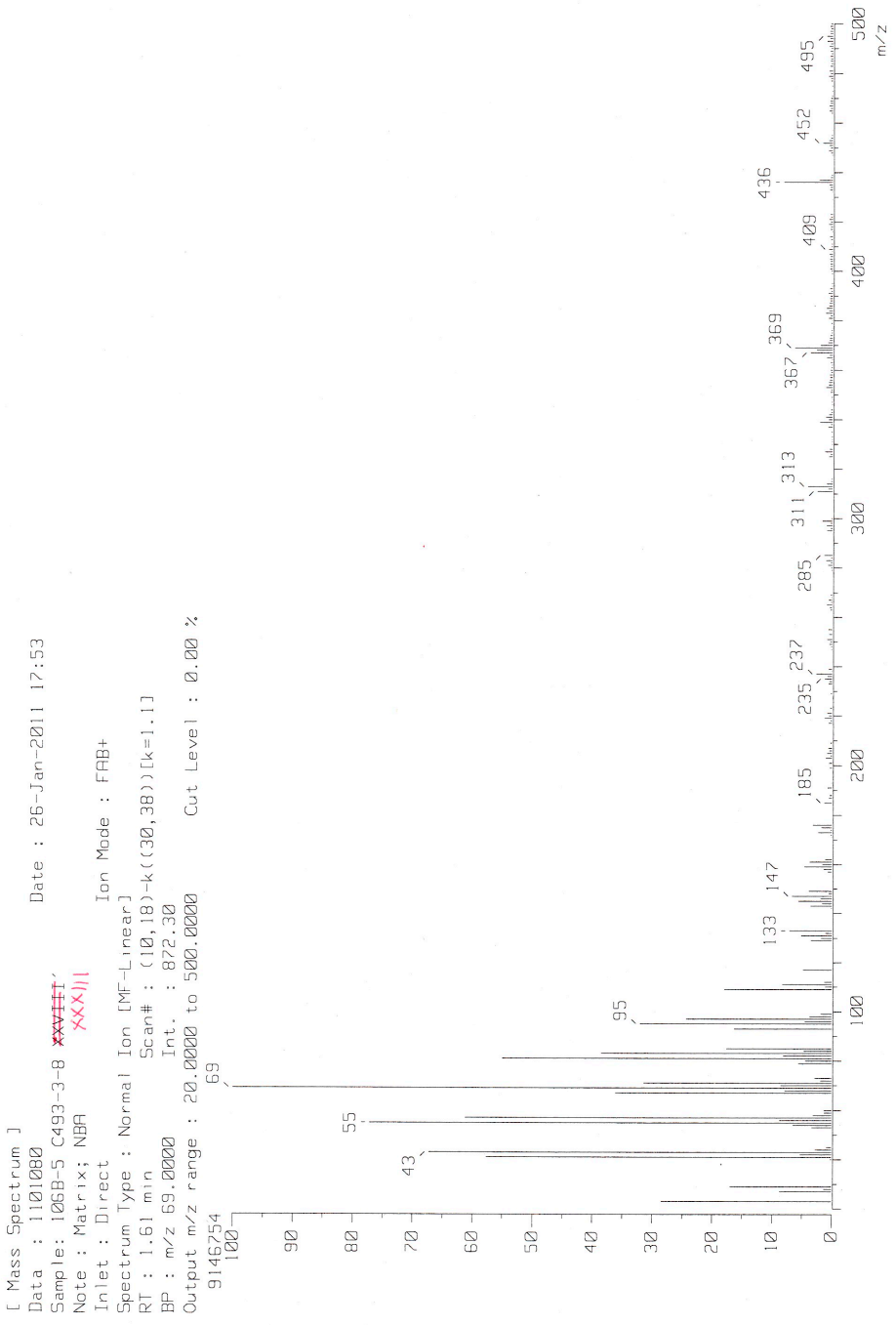


Figure S15 ^{1}H and ^{13}C NMR spectra of **3** in $CDCl_3$

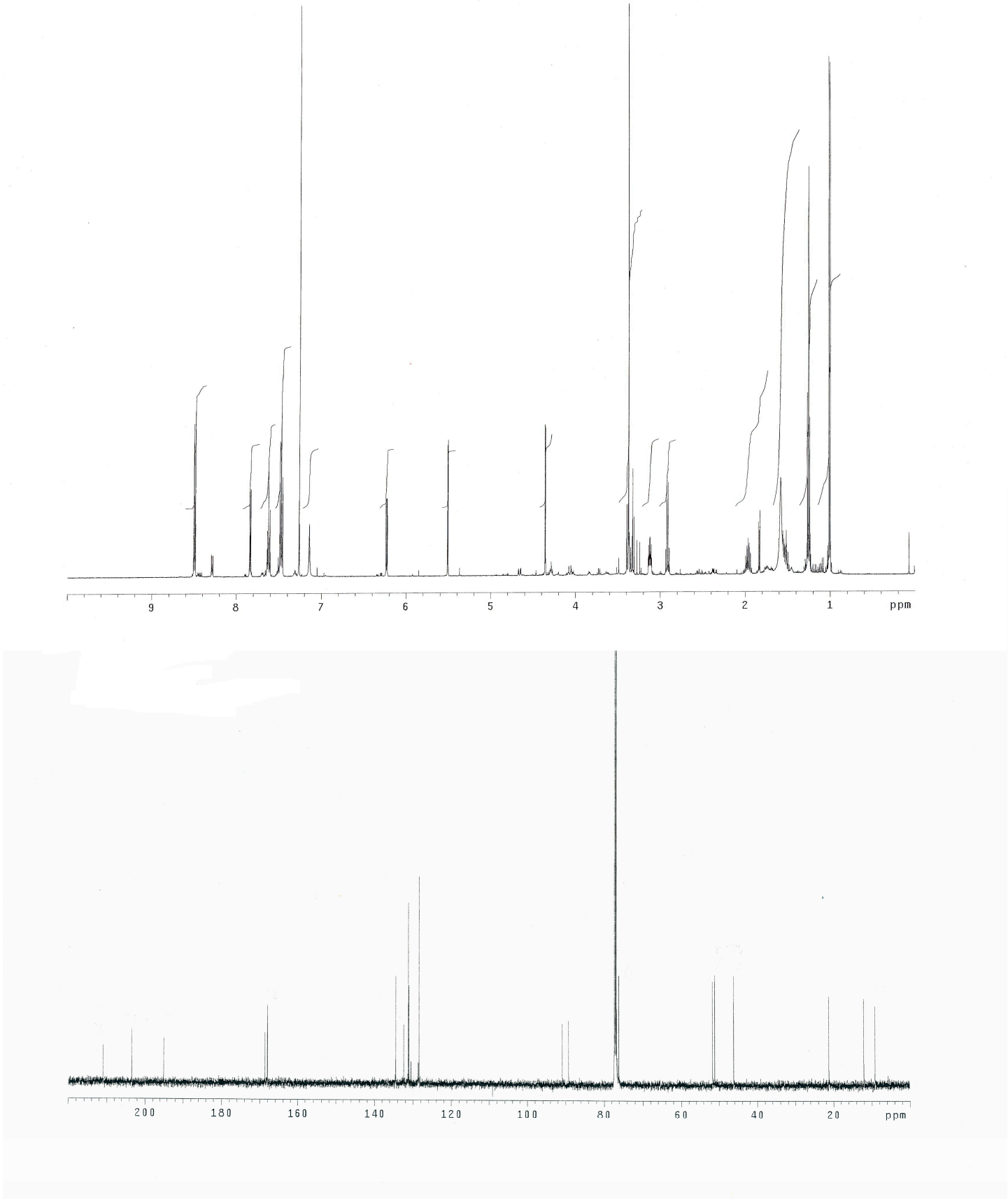
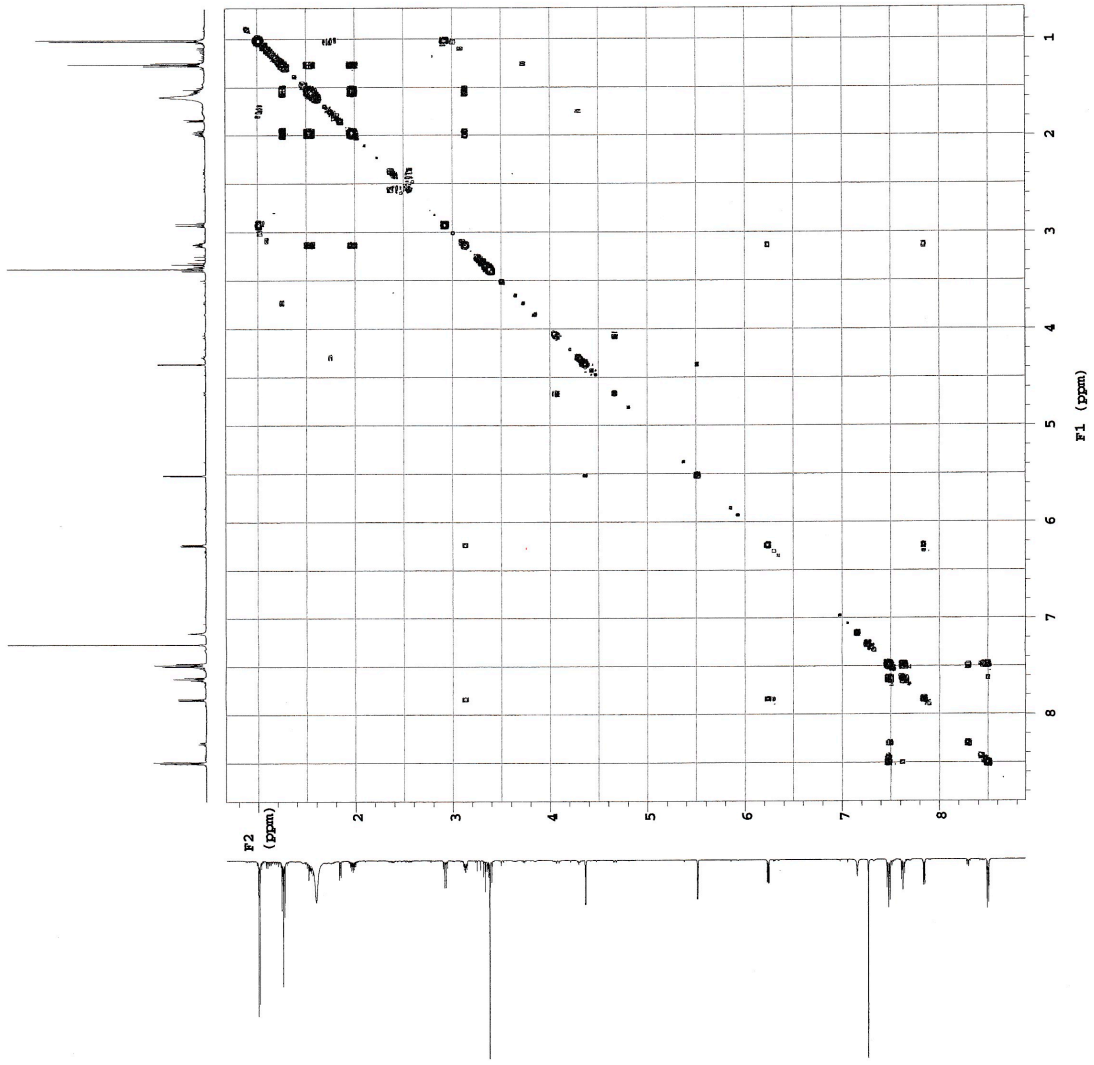


Figure S16 ^{1}H-^{1}H COSY of **3**



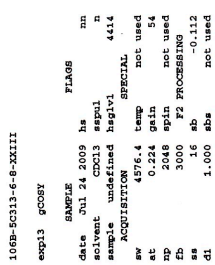
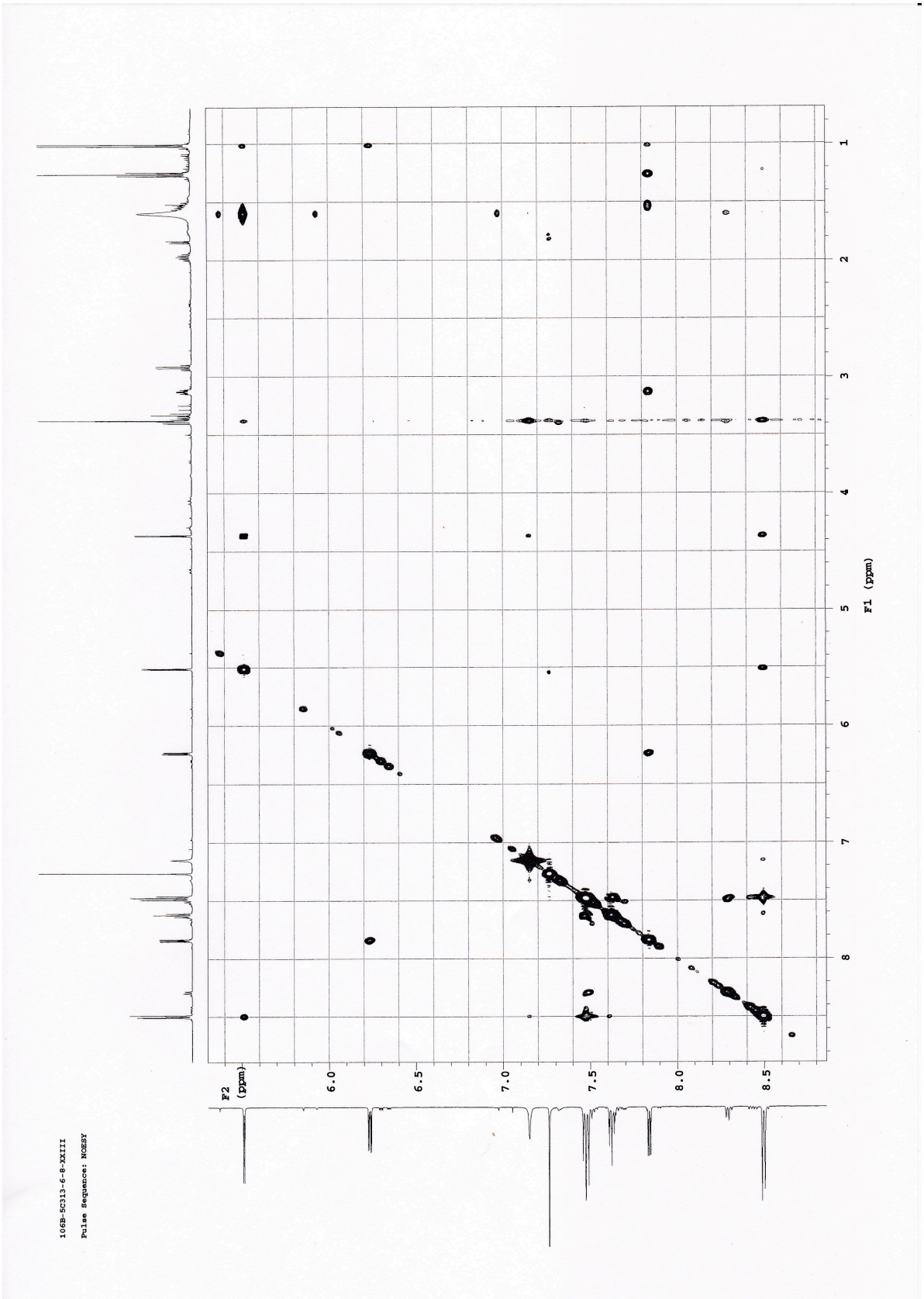


Figure S17 NOESY of **3**



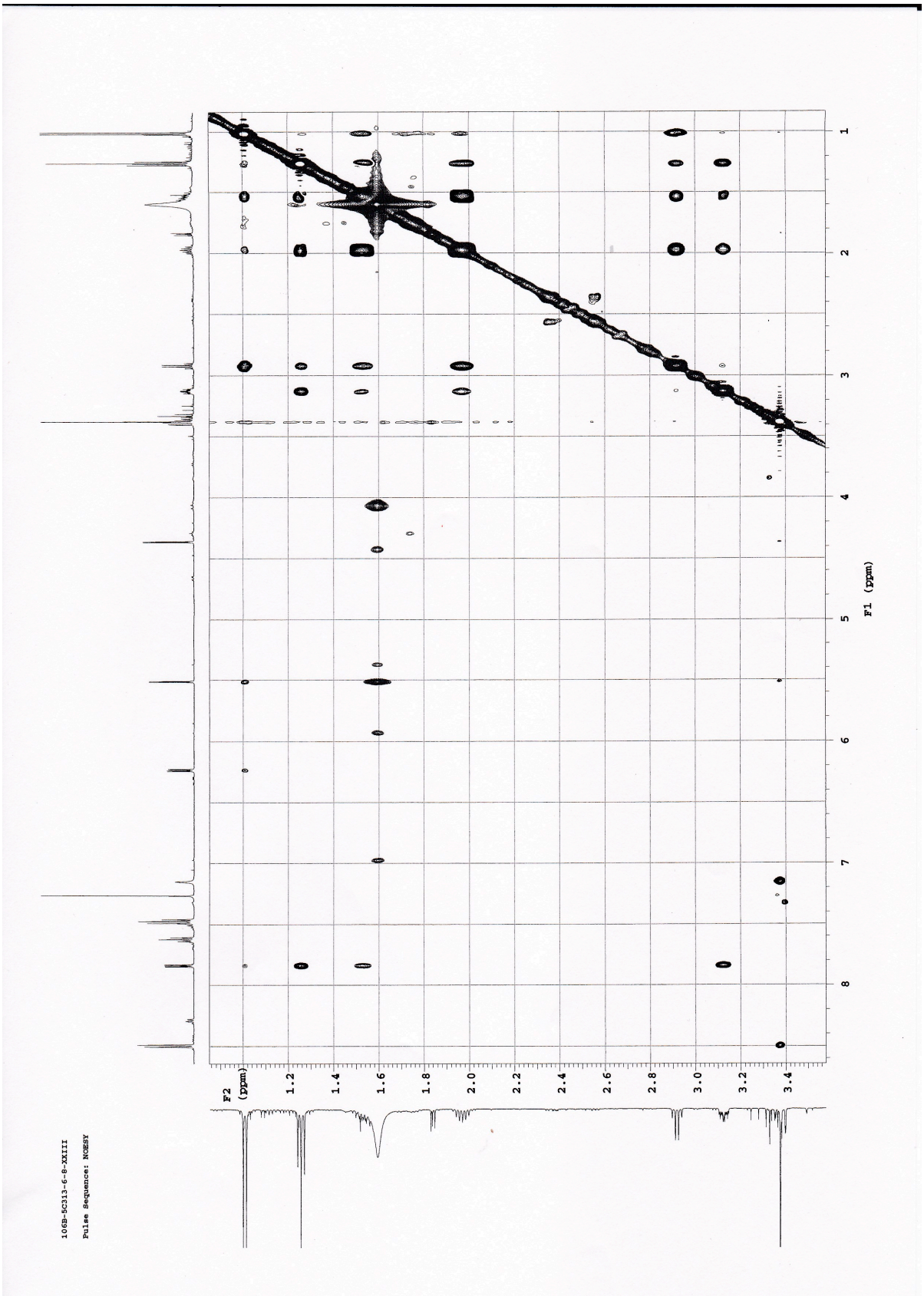

106B-5C313-6-8-XXIII
Pulse Sequence: NOESY
F2 (ppm)
F1 (ppm)

Figure S18 HMQC of **3**

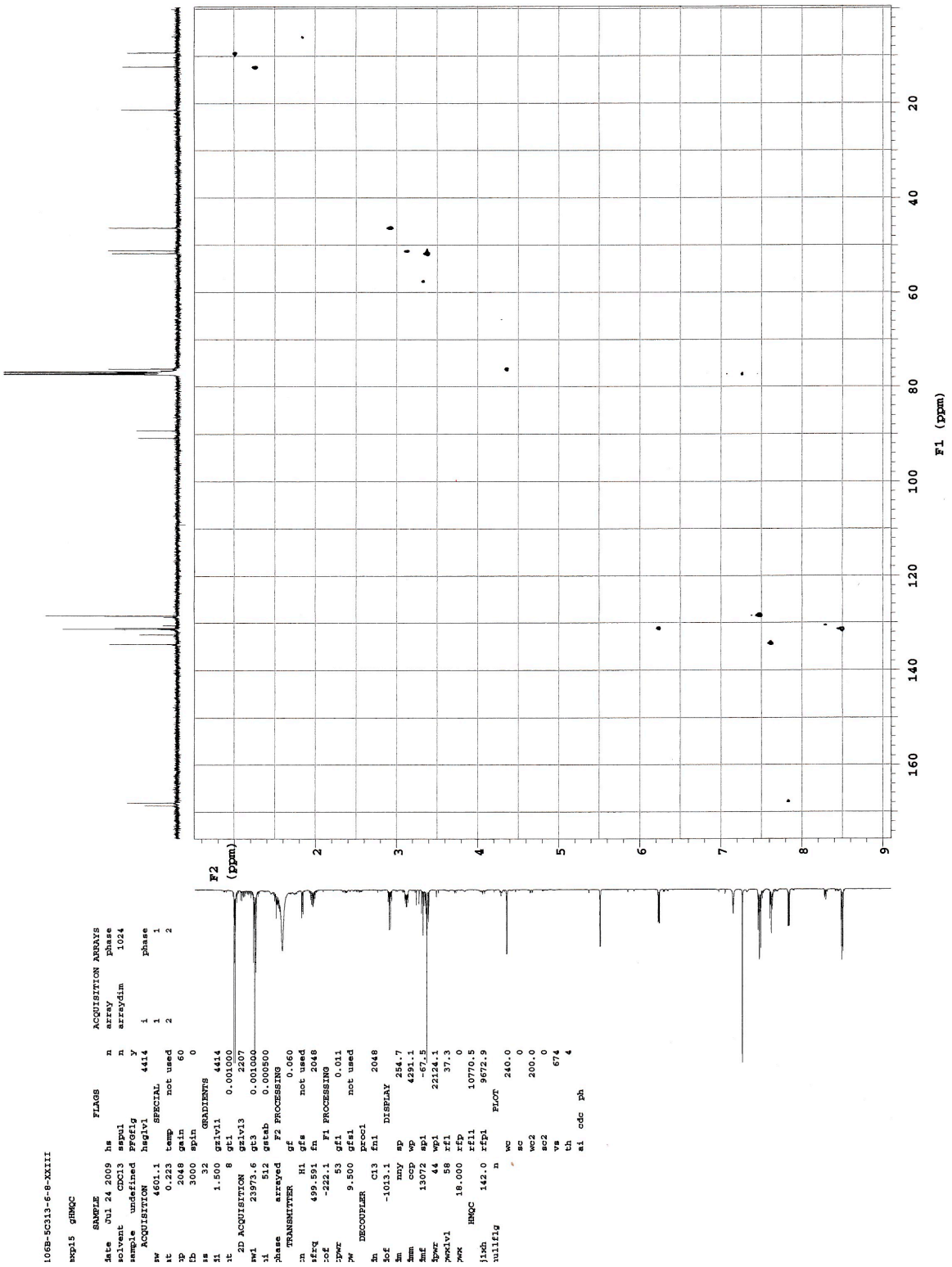


Figure S19 HMBC of **3**

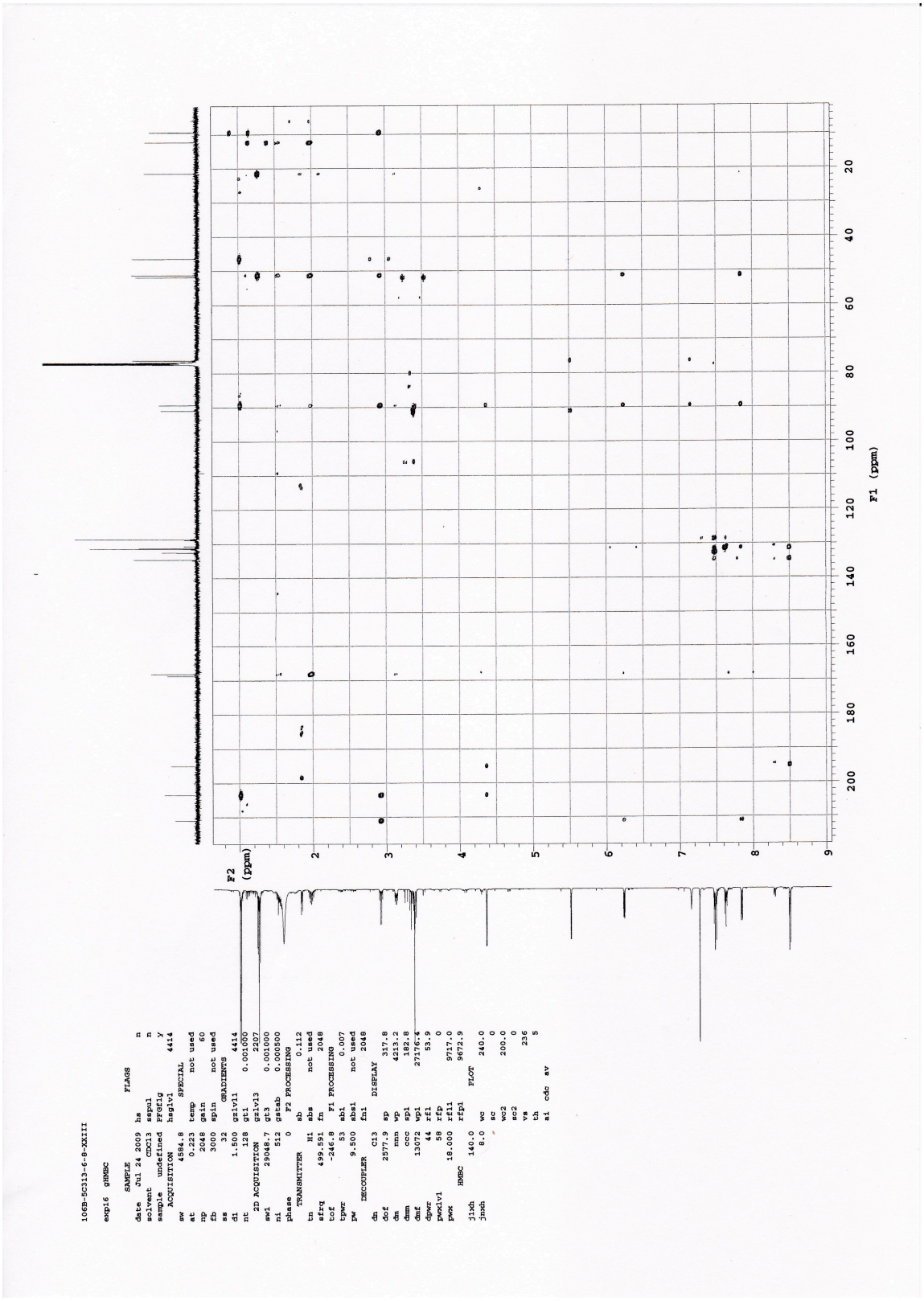


Figure S20 IR spectrum of **3**

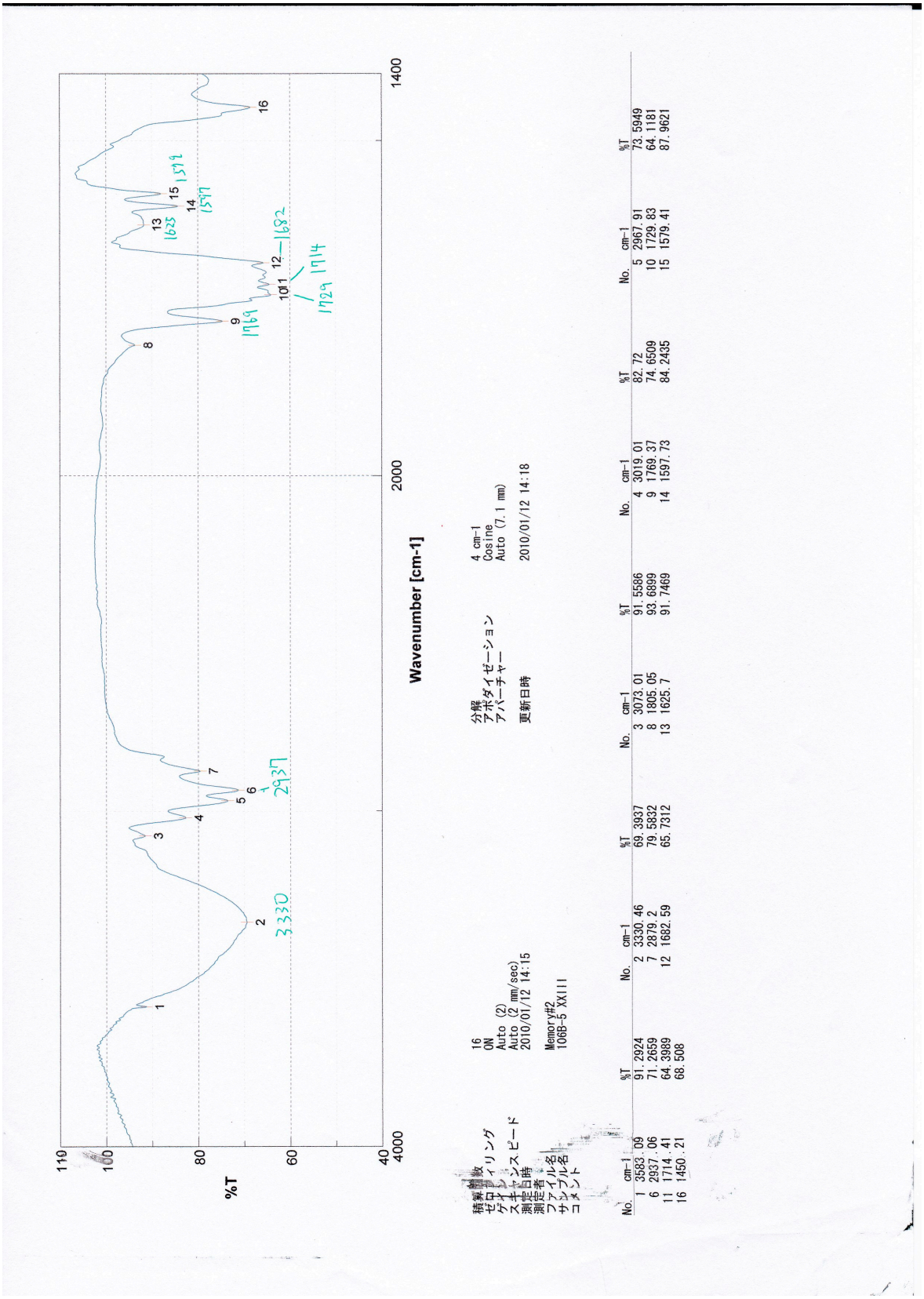


Figure S21 FABMS of **3**

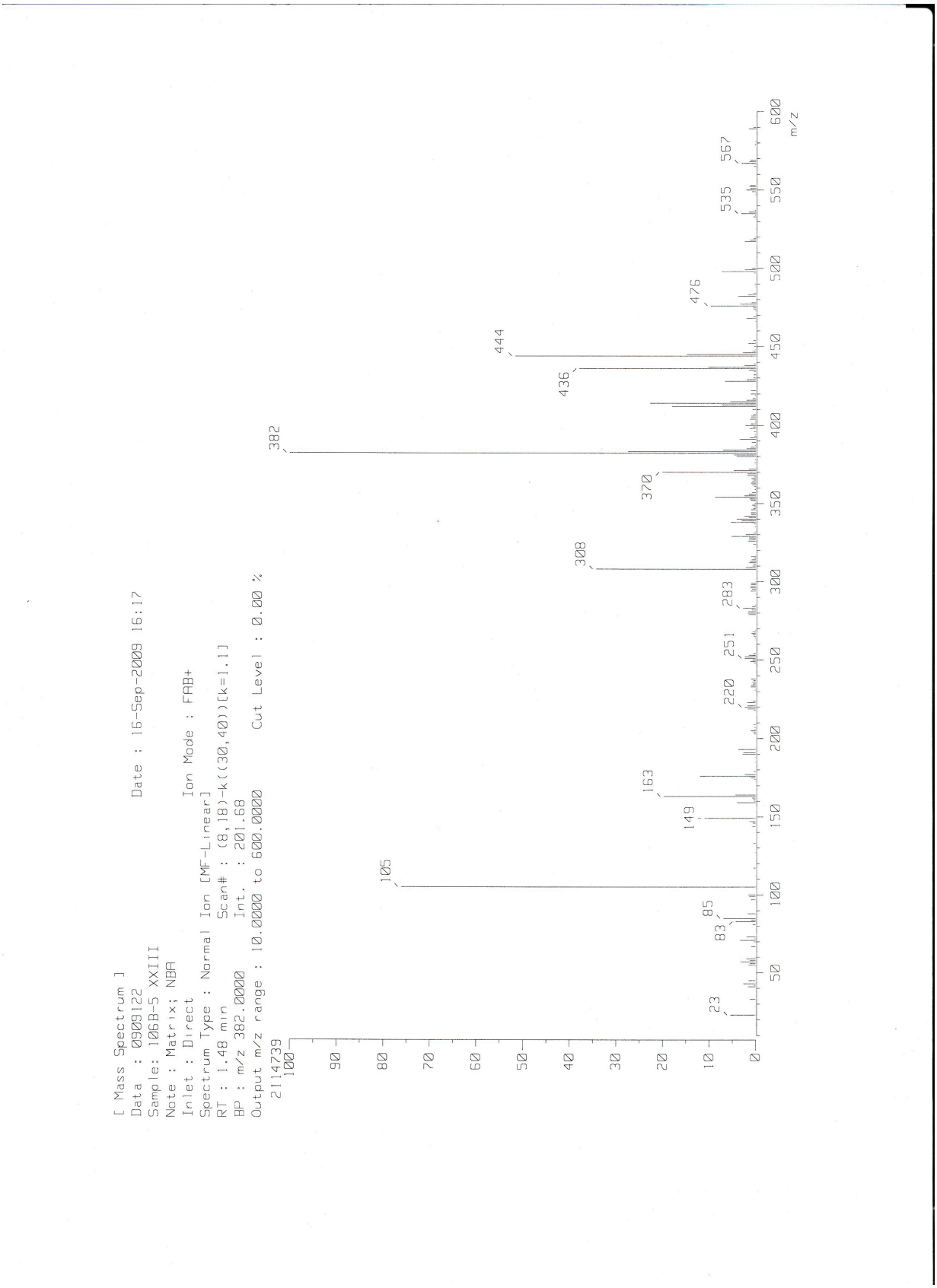


Figure S22 ^{1}H and ^{13}C NMR spectra of **4** in $CDCl_3$

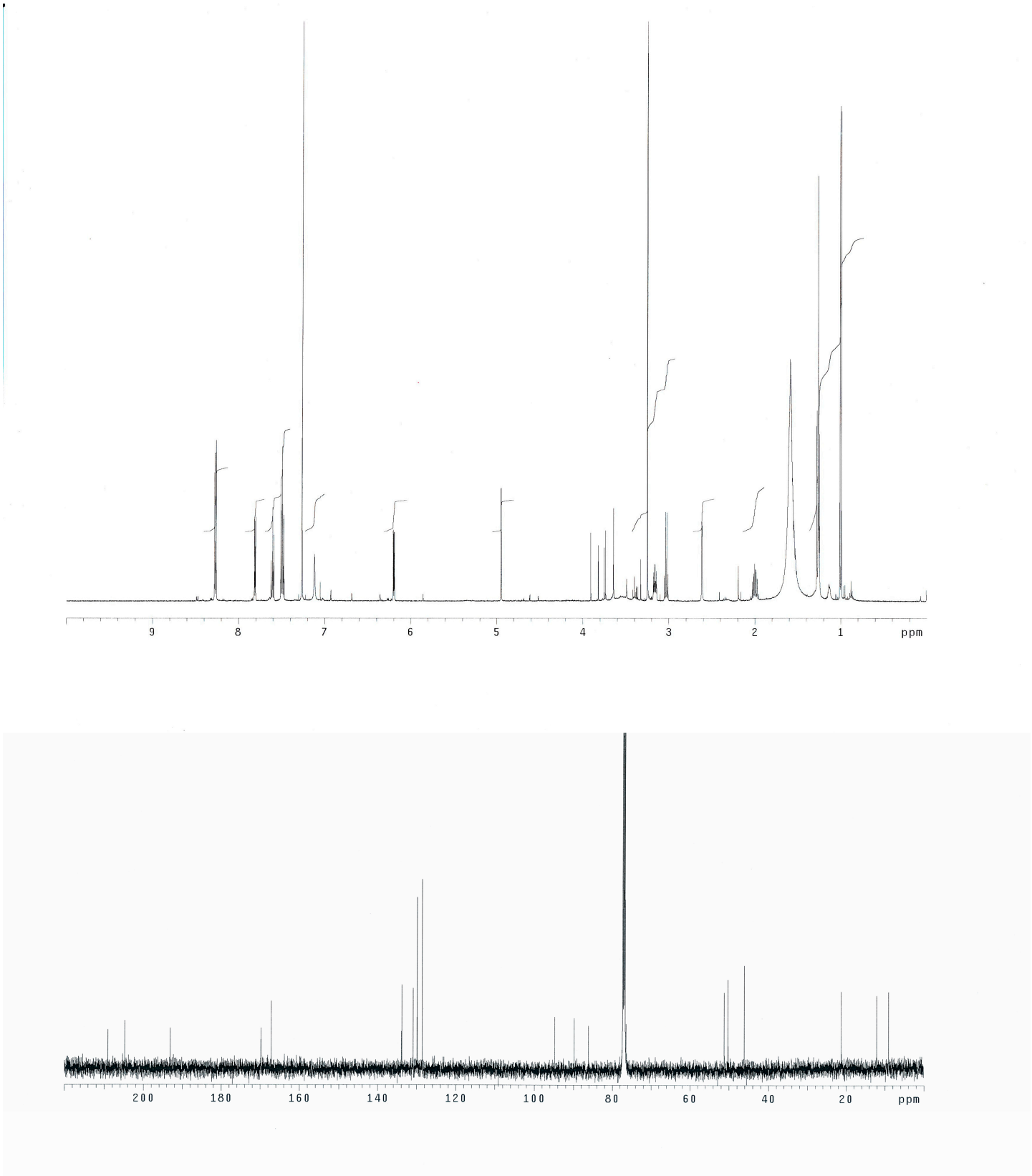
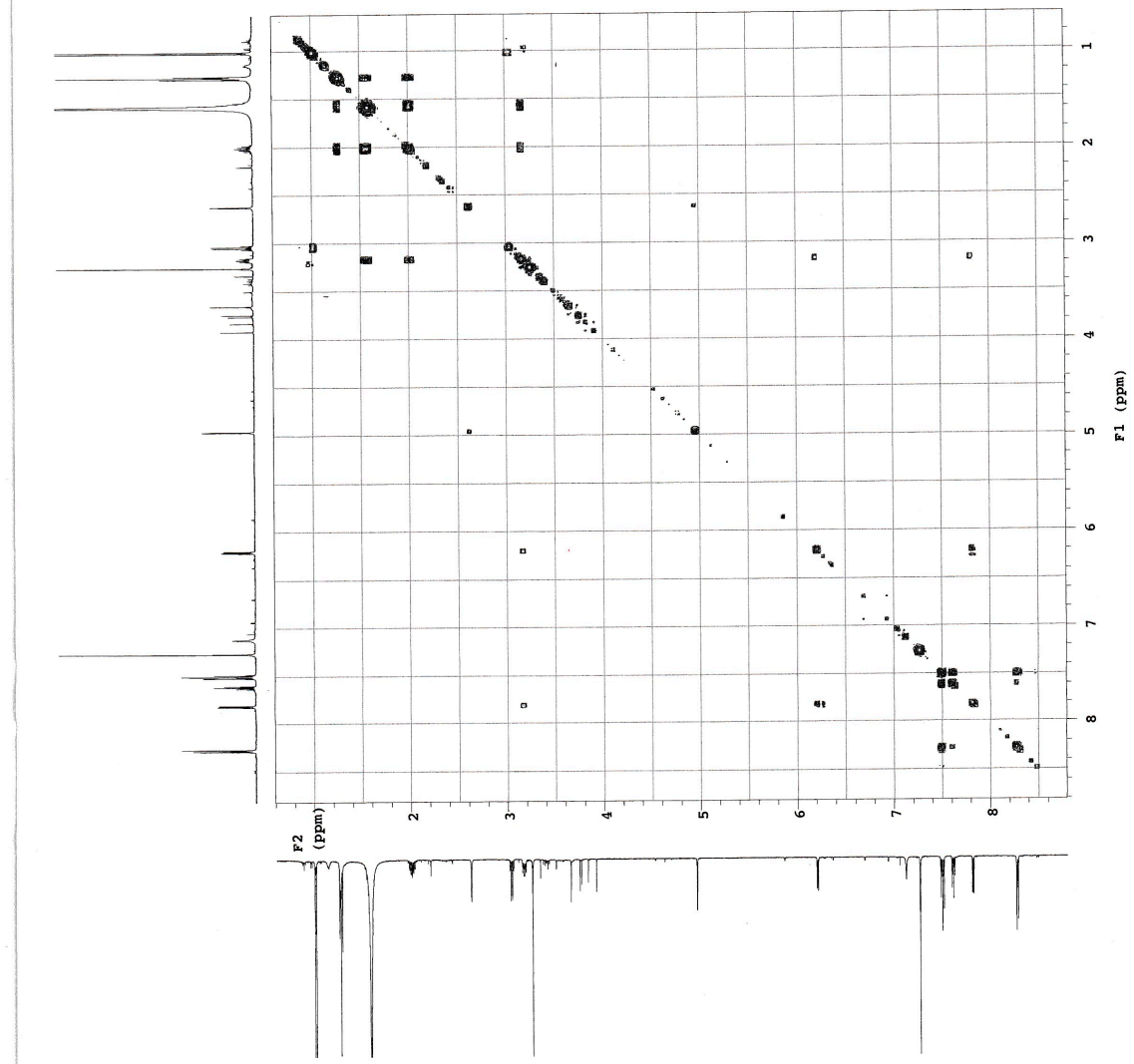


Figure S23 ^{1}H-^{1}H COSY of **4**



106B-5 XXII-metha

exp23 gCOSY

	SAMPLE		FLAGS		nn
date	Aug 21 2010	hs			n
solvent	CDCl3	sspul			
sample	undefined	hsglv1			4414
	ACQUISITION		SPECIAL		
sw	4531.8	temp		not used	
at	0.226	gain		58	
np	2048	spin		0	
fb	3000	F2 PROCESSING			
ss	16	sb		-0.113	
d1	1.000	sbs		not used	
nt	16	fn		2048	
	2D ACQUISITION		F1 PROCESSING		
sw1	4531.8	sb1		-0.028	
n1	256	sbs1		not used	
	TRANSMITTER	H1	fn1		1p
tn				DISPLAY	2048
sfrq	499.568				297.8
tof	-314.7	sp			4093.7
tpwr	53	wp			306.6
pw	9.500	sp1			4093.7
	GRADIENTS	4414	wp1		96.1
gt1v11			rf1		0
gt1	0.001000		rfl1		96.1
gtlab	0.000500		rfp1		0
	DECOUPLER	H1		PLOT	
dn		nnn	wc		200.0
dm			sc		0
			wc2		200.0
			sc2		0
			vs		587
			th		3
			al	av	

Figure S24 NOESY of **4**

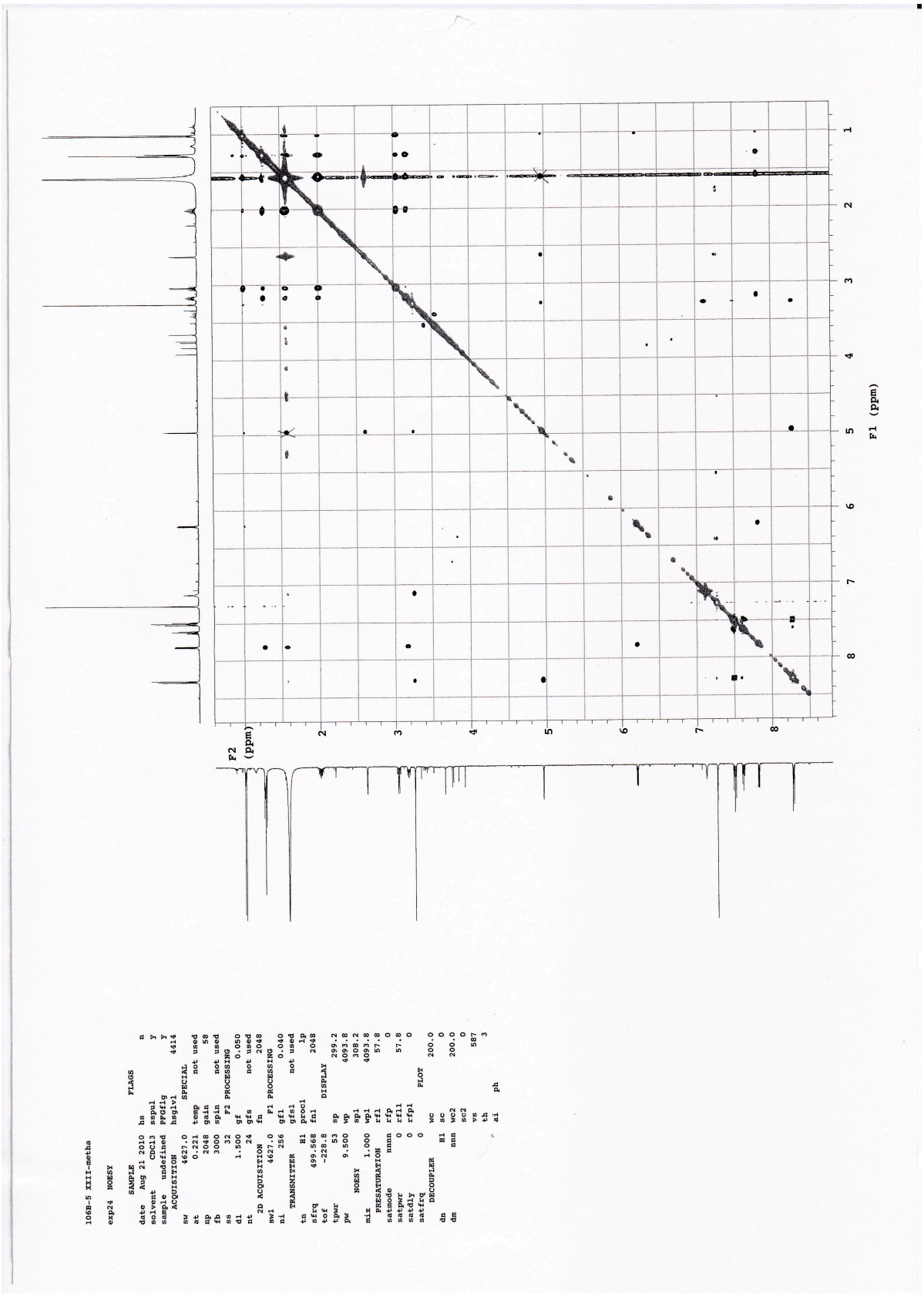


Figure S25 HMQC of **4**

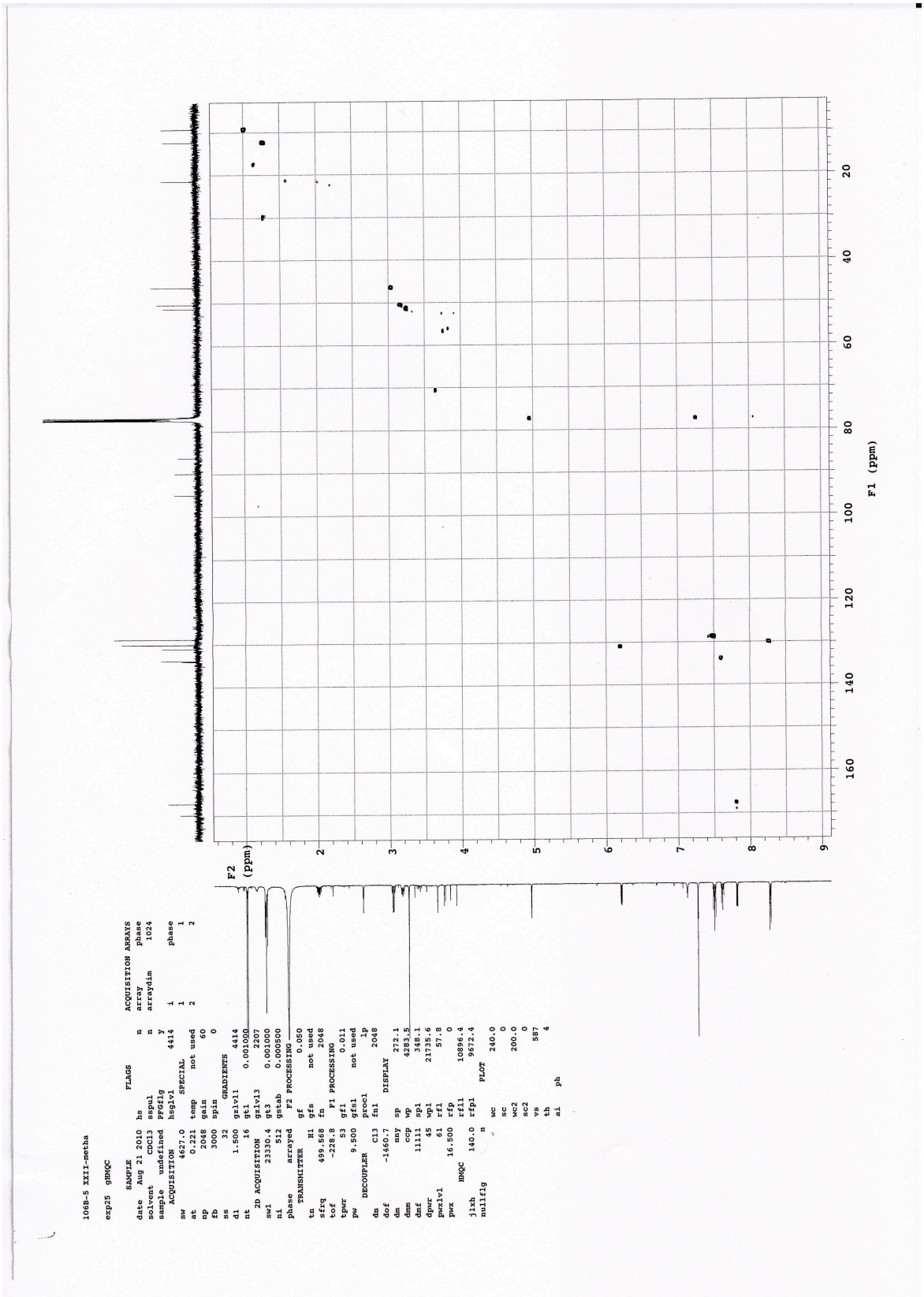


Figure S26 HMBC of **4**

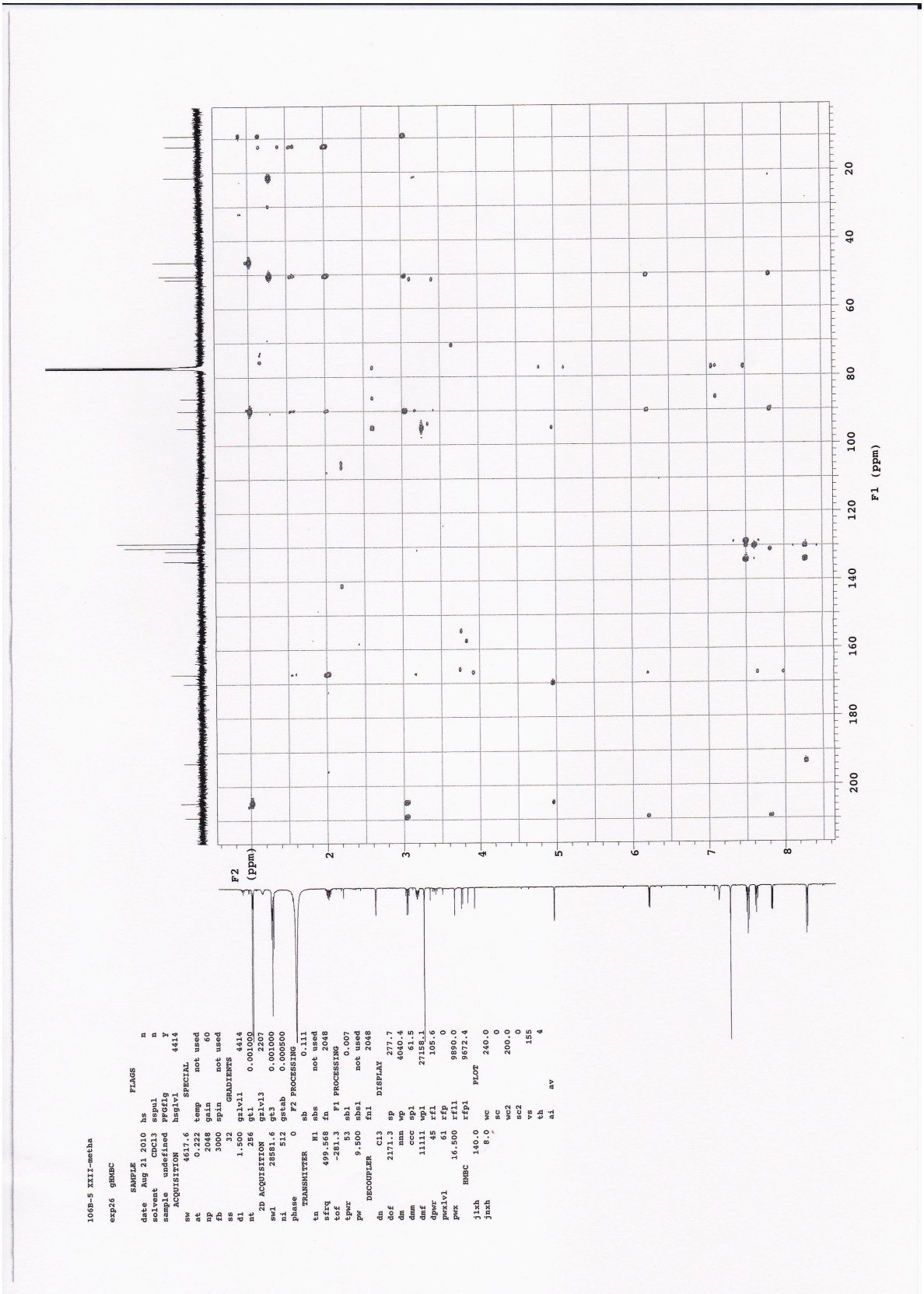


Figure S27 IR spectrum of **4**

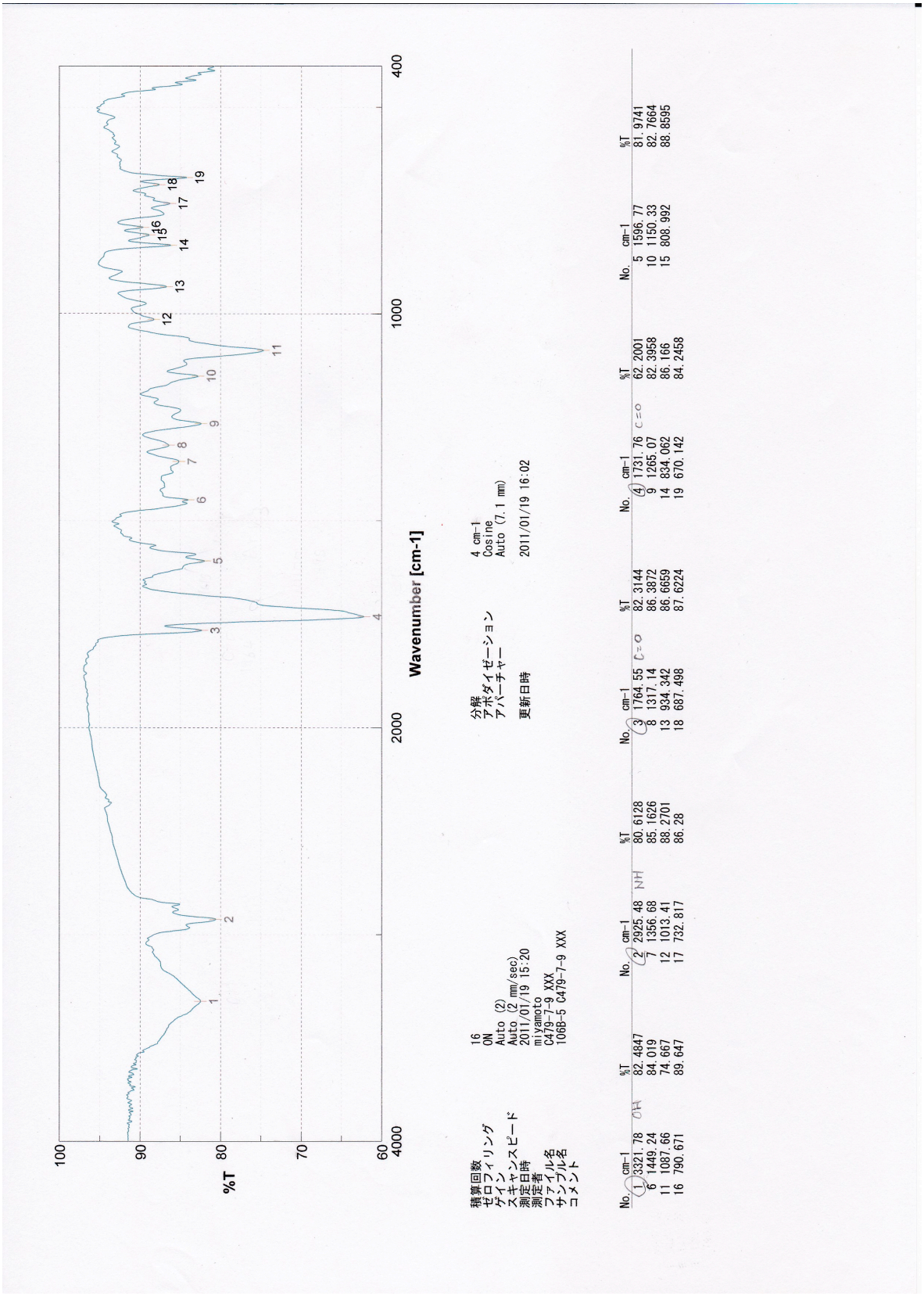


Figure S28 FABMS of **4**

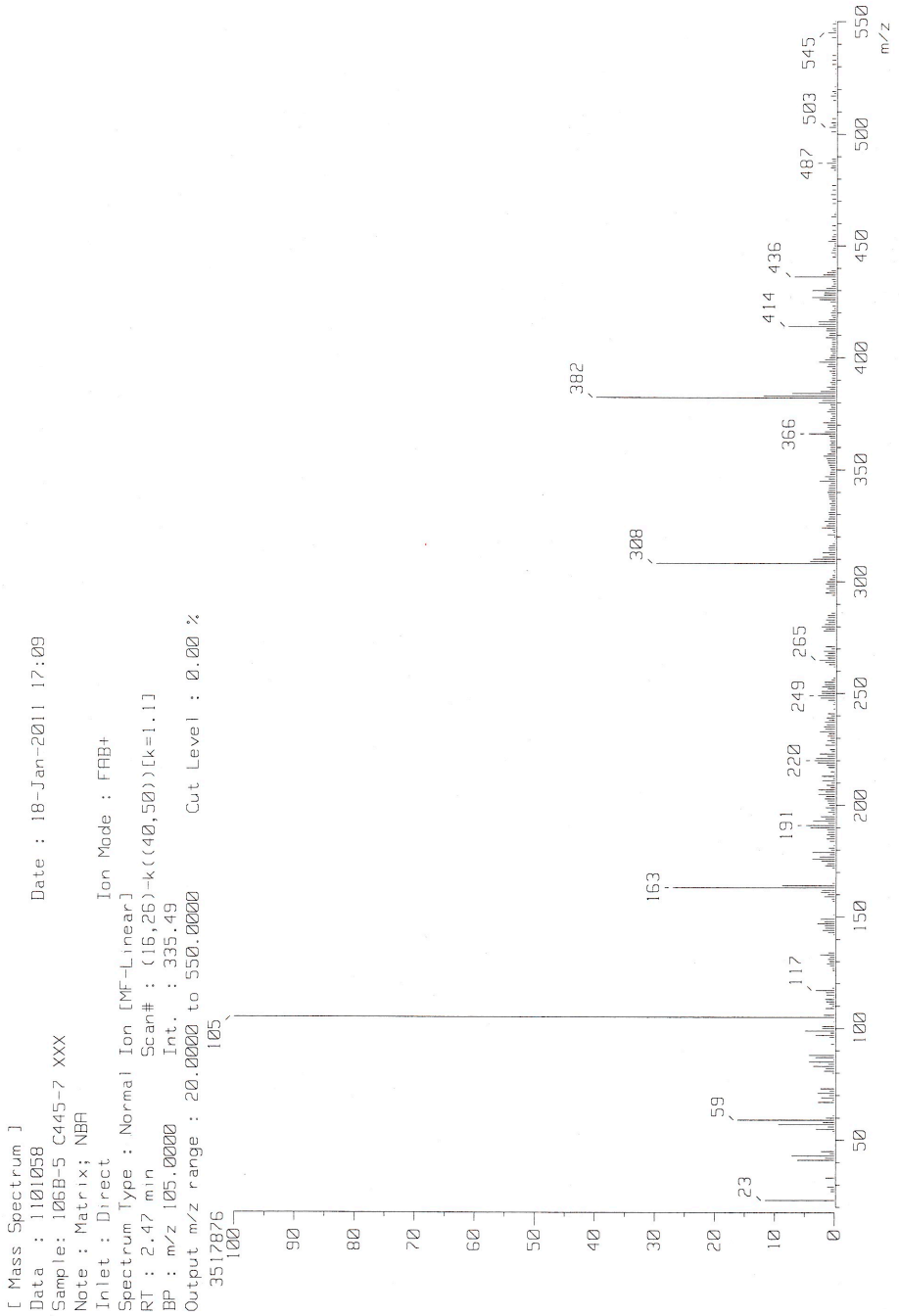


Figure S29 ^{1}H and ^{13}C NMR spectra of **5** in $CDCl_3$

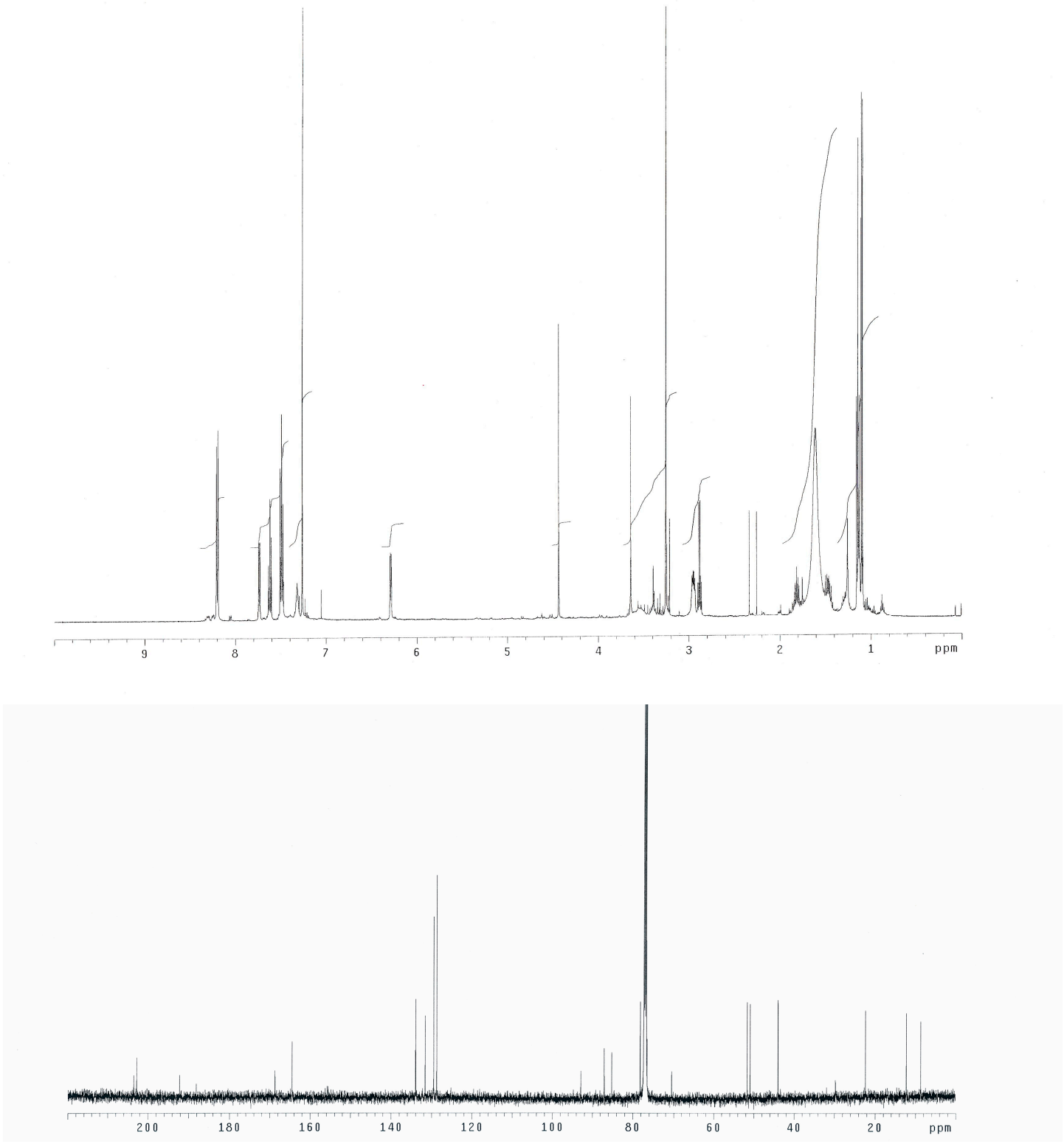


Figure S30 ^{1}H-^{1}H COSY of **5**

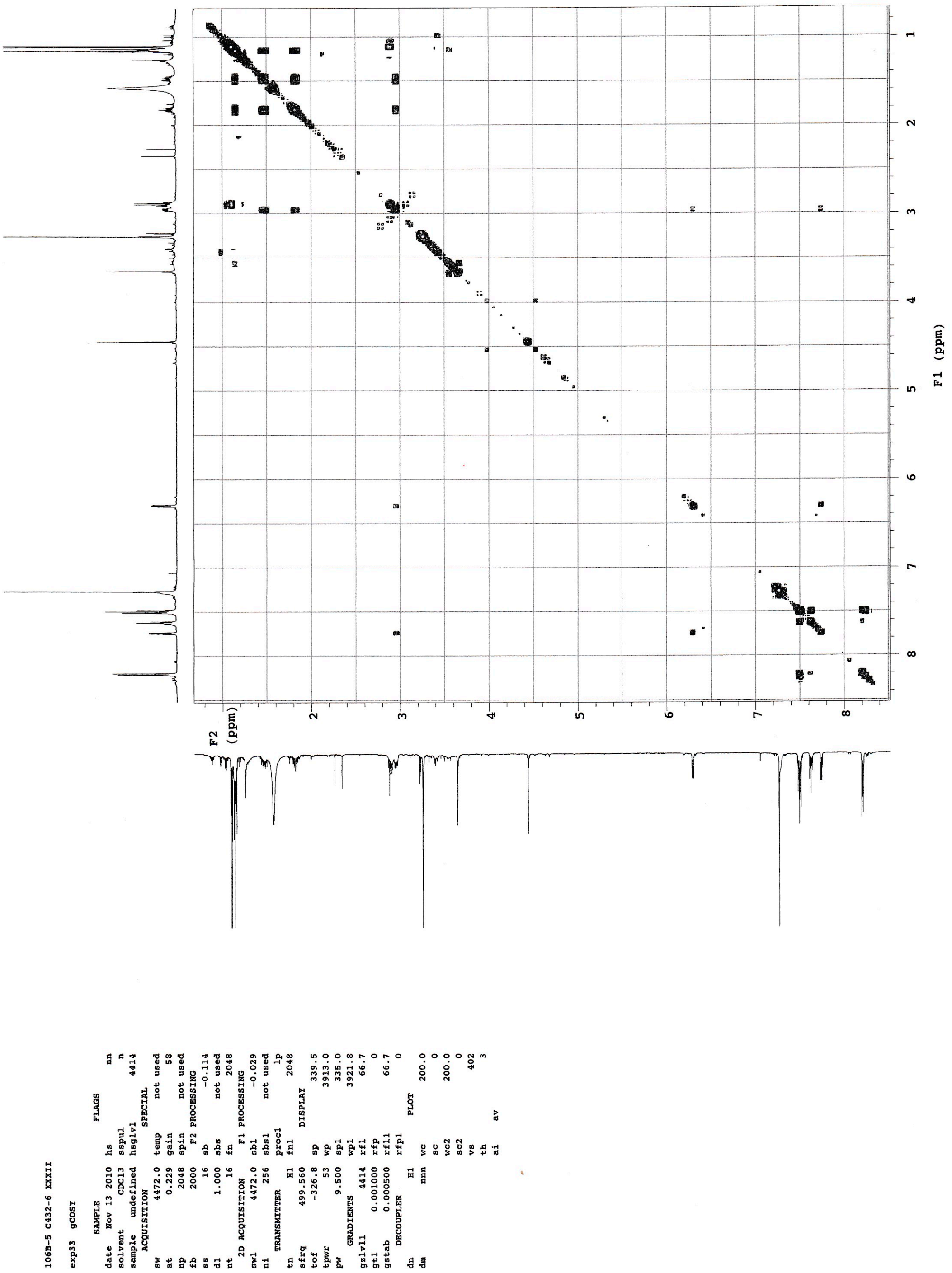
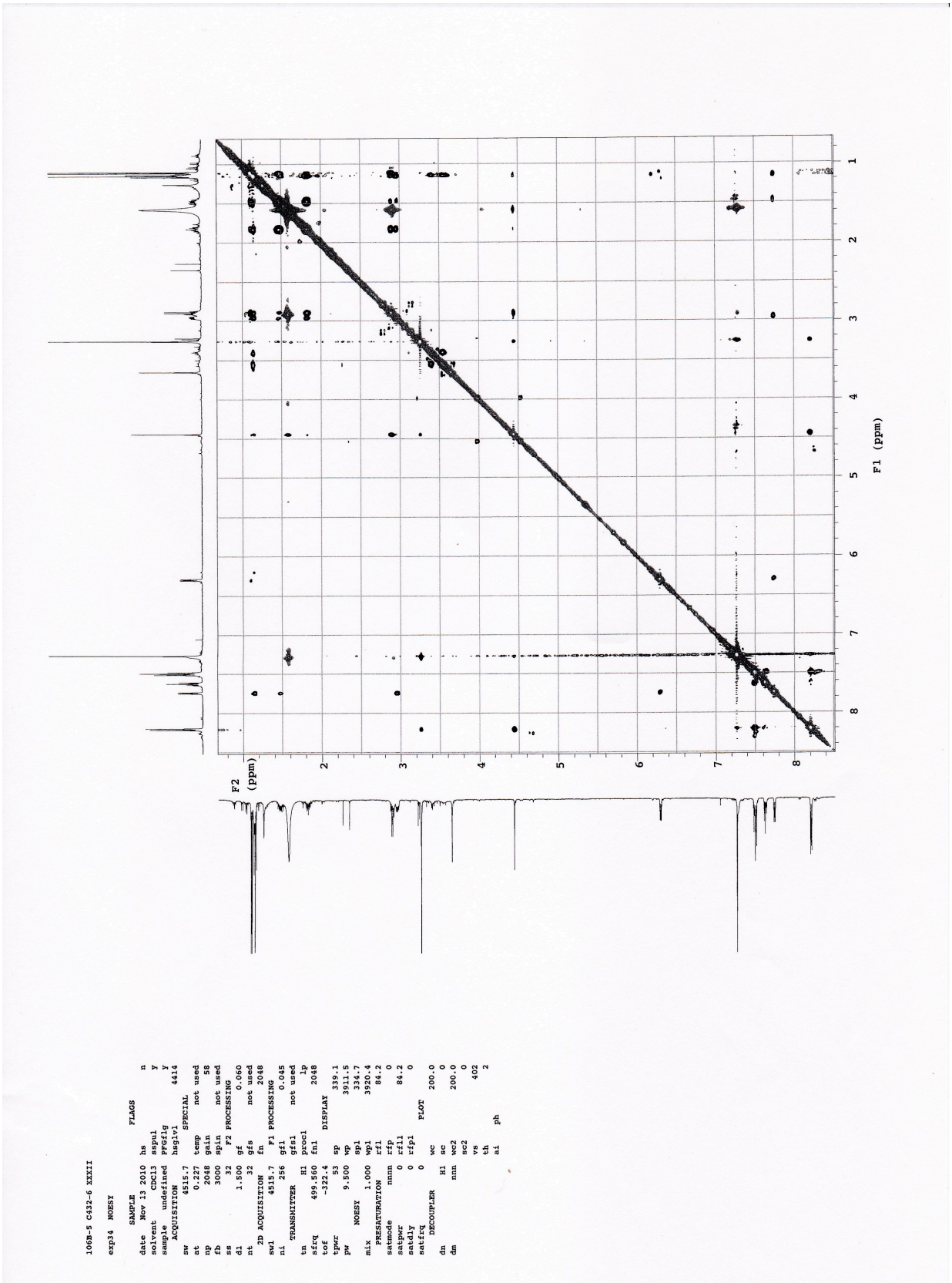


Figure S31 NOESY of **5**



106B-5 C432-6 XXXII

exp34 NOESY

SAMPLE		FLAGS	
date	Nov 13 2010	hs	n
solvent	CDCl3	sspul	Y
sample	undefined	PFGflg	Y
ACQUISITION		hsglvl	4414
sw	4515.7	SPECIAL	
at	0.227	temp	not used
np	2048	gain	58
fb	3000	spin	not used
ss	32	F2 PROCESSING	
d1	1.500	gf	0.060
nt	32	gfs	not used
2D ACQUISITION		fn	2048
sw1	4515.7	F1 PROCESSING	
ni	256	gf1	0.045
TRANSMITTER		gfs1	not used
tn	H1	proc1	lp
sfrq	499.560	fn1	2048
tof	-322.4	DISPLAY	
tpwr	53	sp	339.1
pw	9.500	wp	3911.5
NOESY		spl	334.7
mix	1.000	wp1	3920.4
PRESATURATION		rf1	84.2
satmode	nnnn	rfp	0
satpwr	0	rfl1	84.2
satdly	0	rfp1	0
satfrq	0	PLOT	
DECOUPLER		wc	200.0
dn	H1	sc	0
dm	nnn	wc2	200.0
		sc2	0
		vs	402
		th	2
		ai	ph

Figure S32 HMQC of **5**

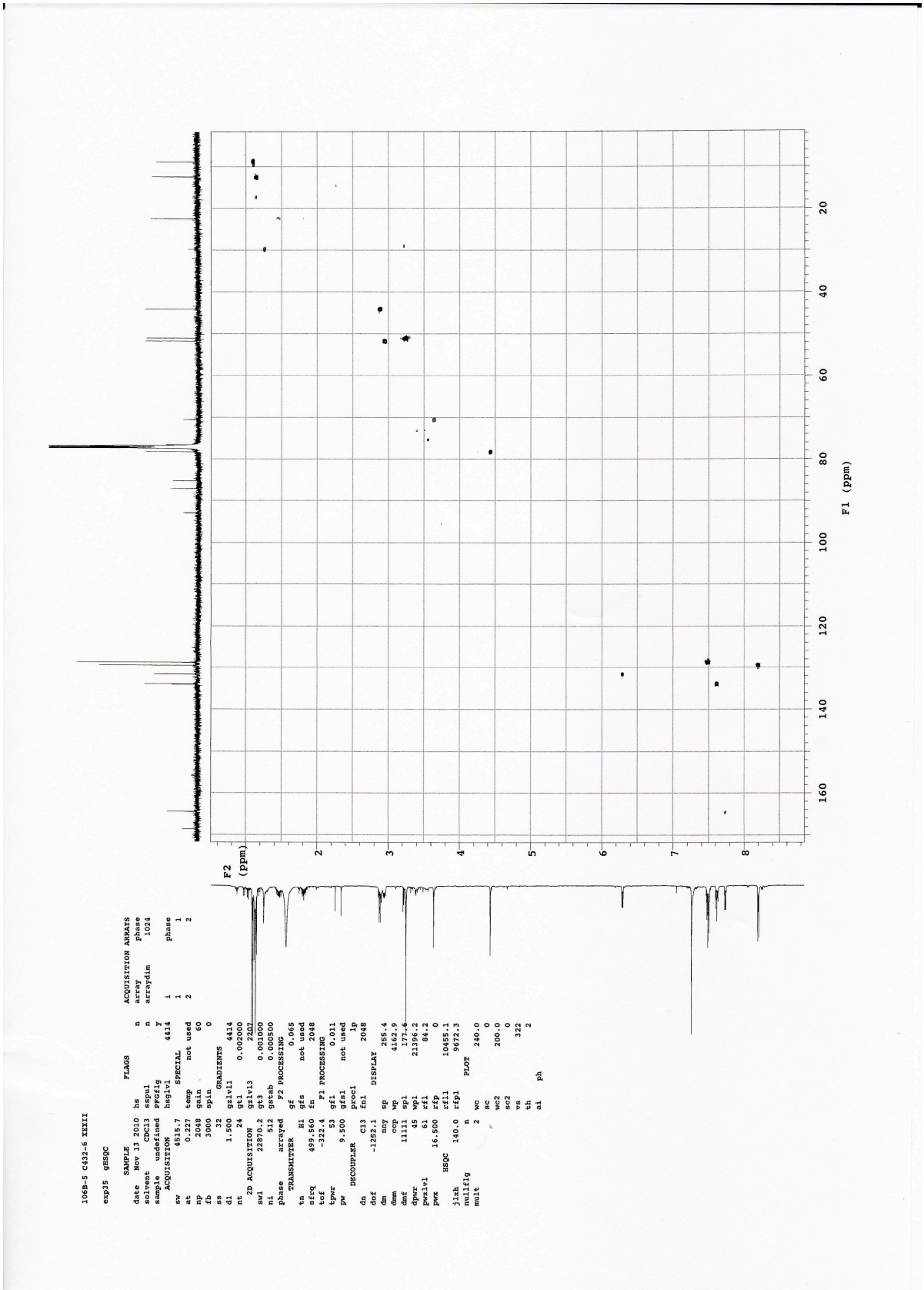


Figure S33 HMBC of **5**

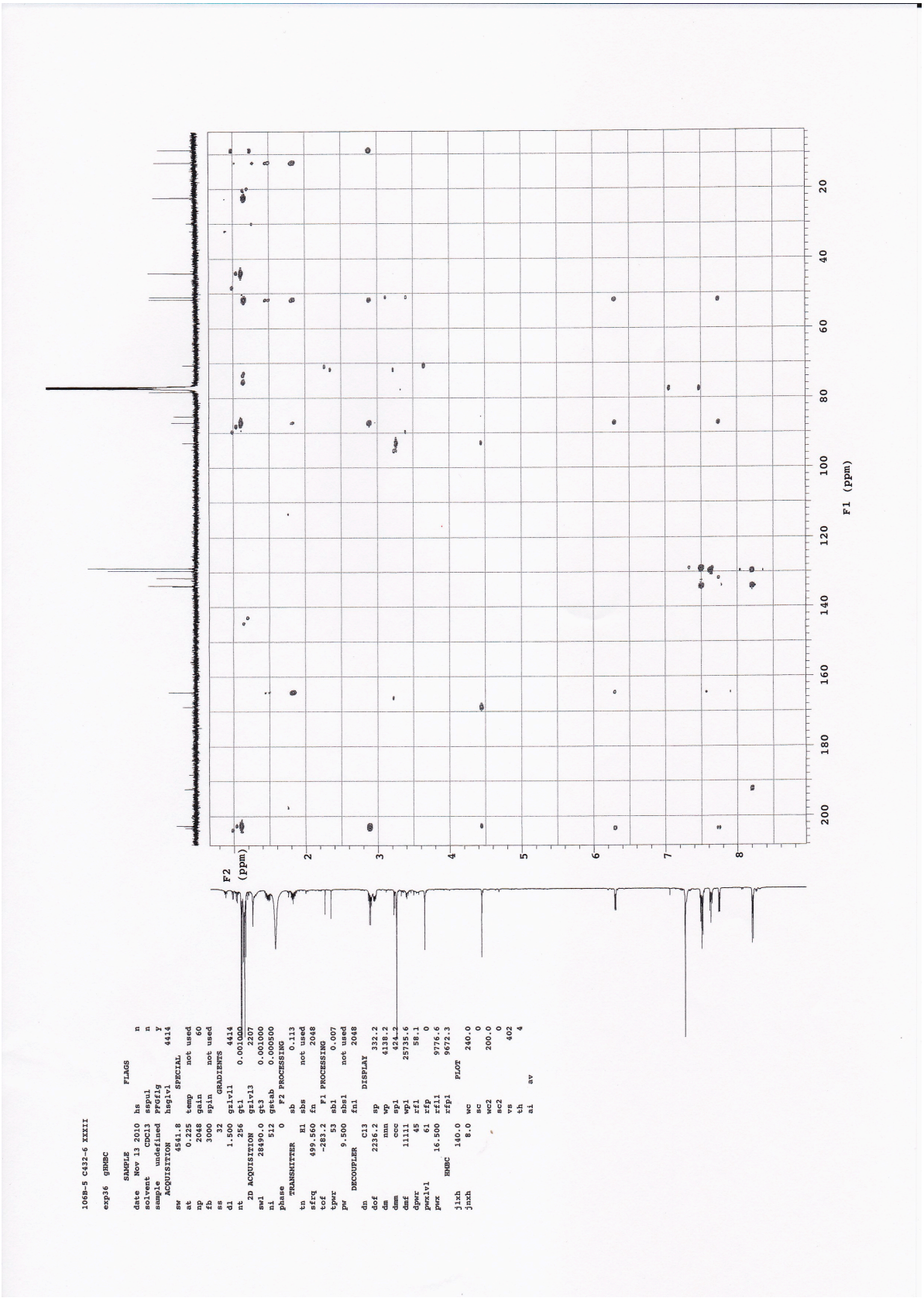


Figure S34 IR spectrum of **5**

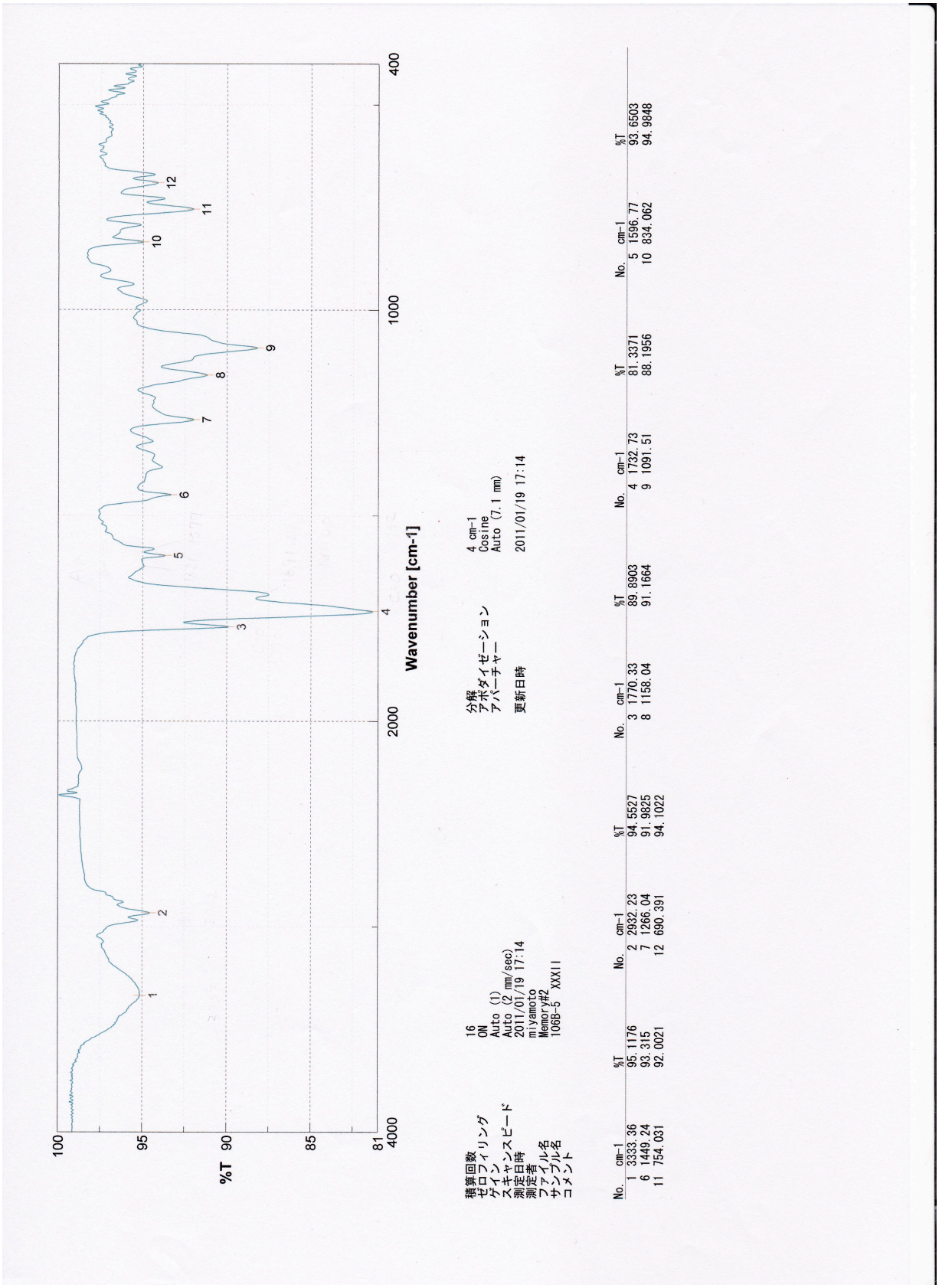


Figure S35 FABMS of **5**

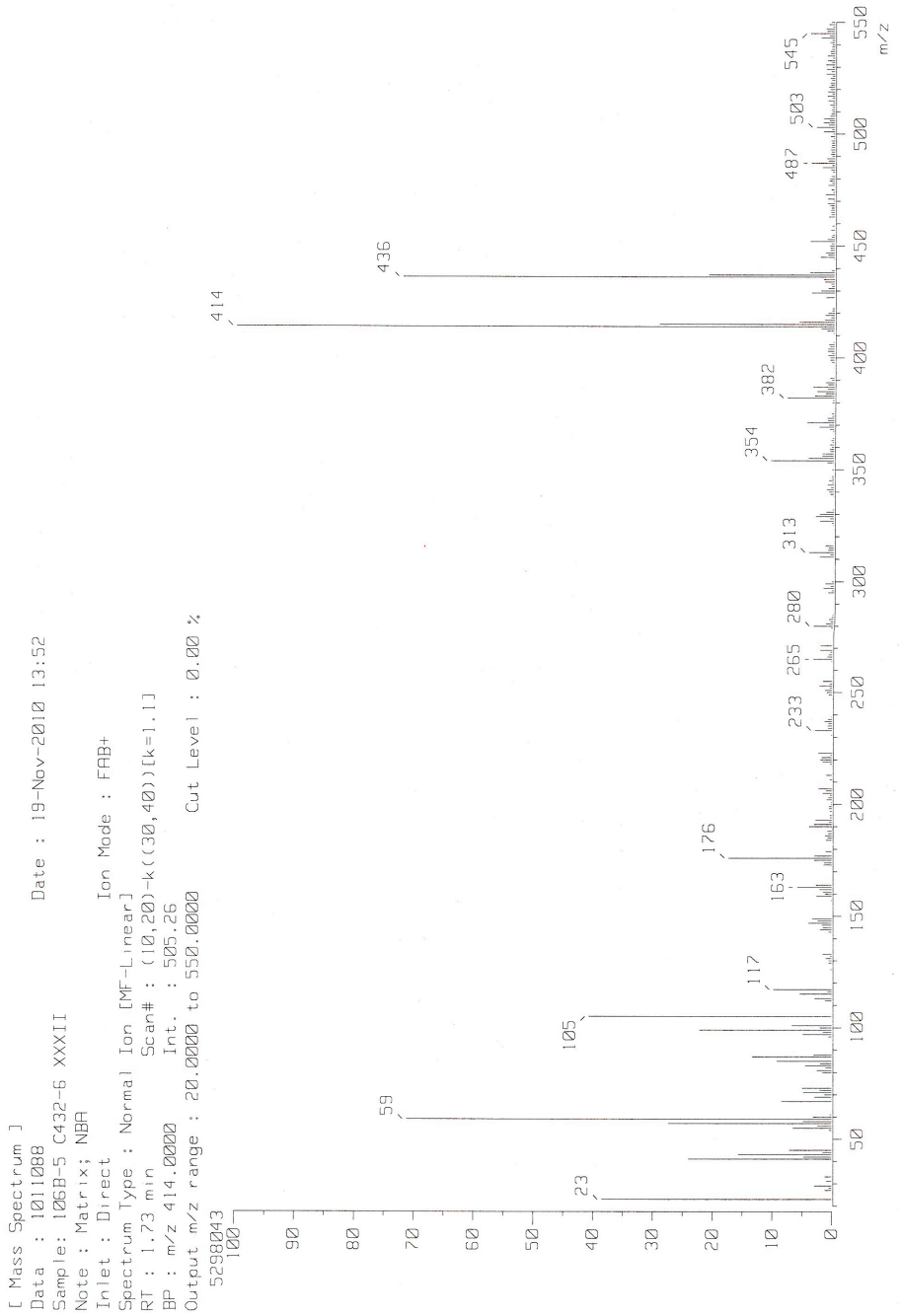
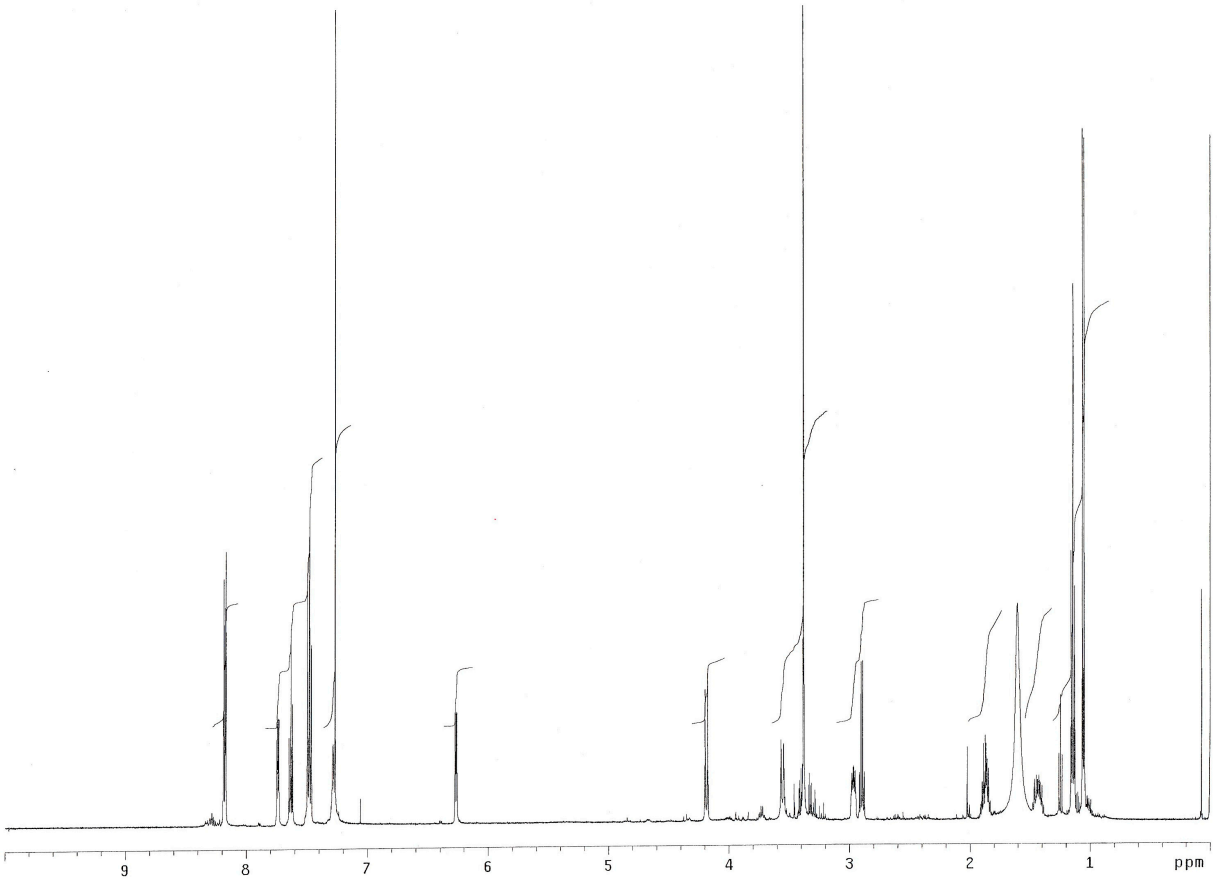


Figure S36 ^{1}H and ^{13}C NMR spectra of **6** in $CDCl_3$



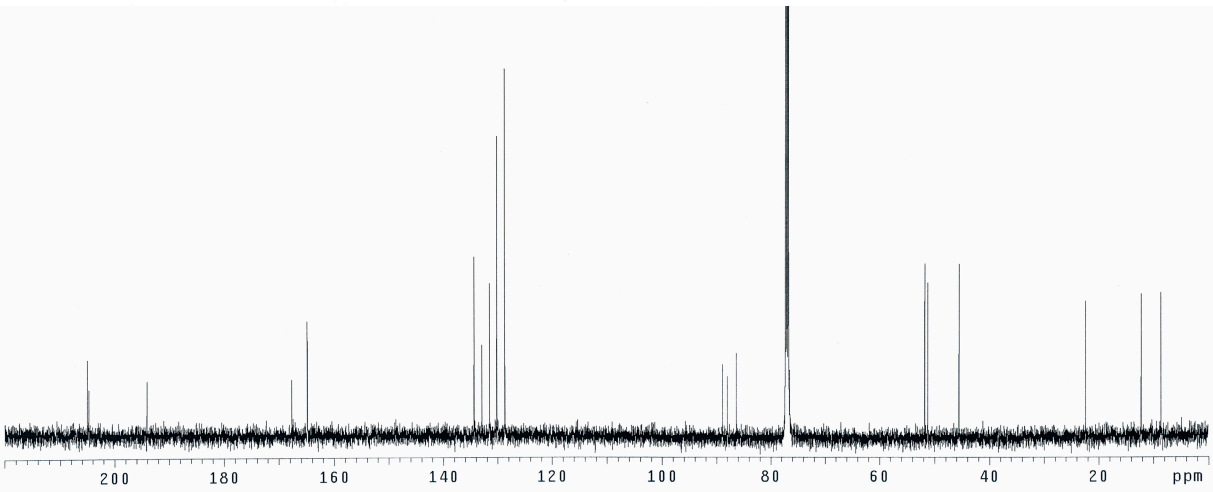
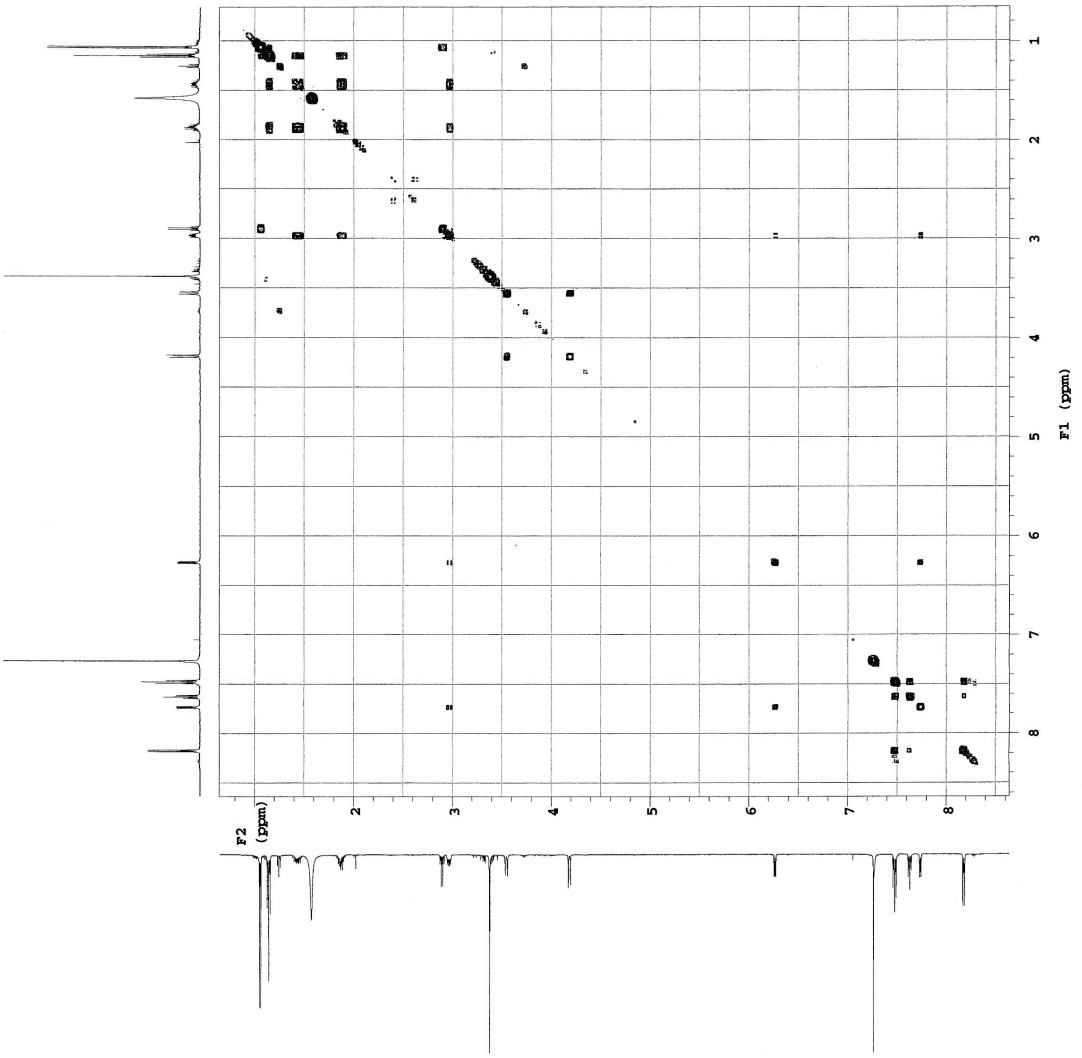


Figure S37 ^{1}H-^{1}H COSY of **6**



106B-5 C356 4-5 XXVIII

exp3 gCOSY

SAMPLE FLAGS

date Dec 11 2009 ha nn
solvent CDCl3 segvul n
sample undefined hsglvl 4414
ACQUISITION SPECIAL
sw 4672.1 temp not used
at 0.219 gain 58
np 2048 spin not used
fb 3000 F2 PROCESSING
ss 16 ab -0.110
d1 1.000 abs not used
nt 16 fn 2048
2D ACQUISITION F1 PROCESSING
sw1 4672.1 ab1 -0.027
ni 256 abs1 not used
TRANSMITTER pcoc1 lp
tn H1 fn1 2048
sfrq 499.583 DISPLAY
tof -298.6 sp 321.8
tpwr 53 wp 3992.3
pw 9.500 sp1 326.3
GRADIENTS wp1 3987.7
gzlvl1 4414 rf1 139.0
gt1 0.001000 rfp 0
gstab 0.000500 rfl1 139.1
DECOUPLER rfp1 0
dn H1 PLOT
dm nnn wc 200.0
sc 0
wc2 200.0
sc2 0
vs 261
th 4
al av

Figure S38 NOESY of **6**

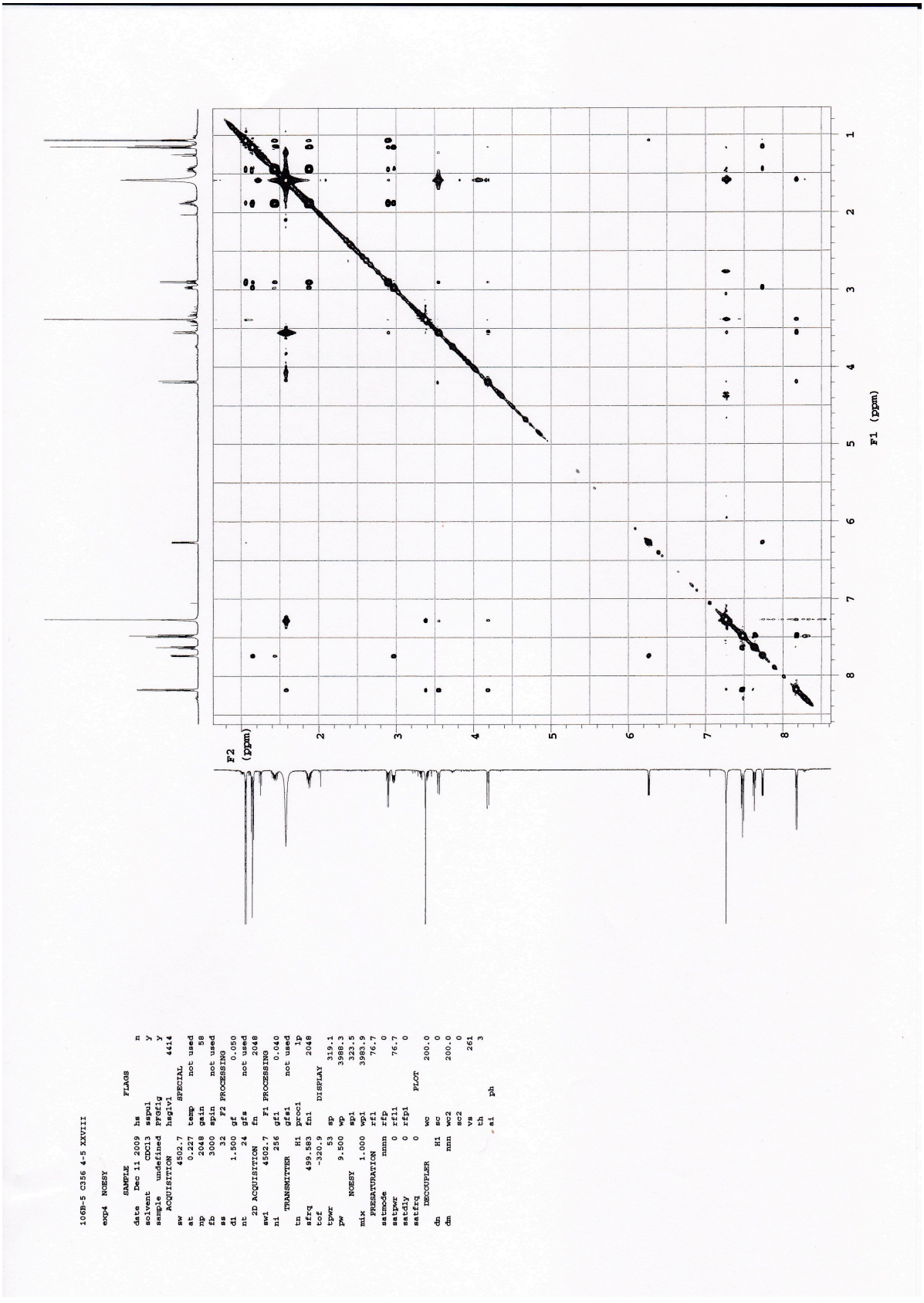


Figure S39 HMQC of **6**

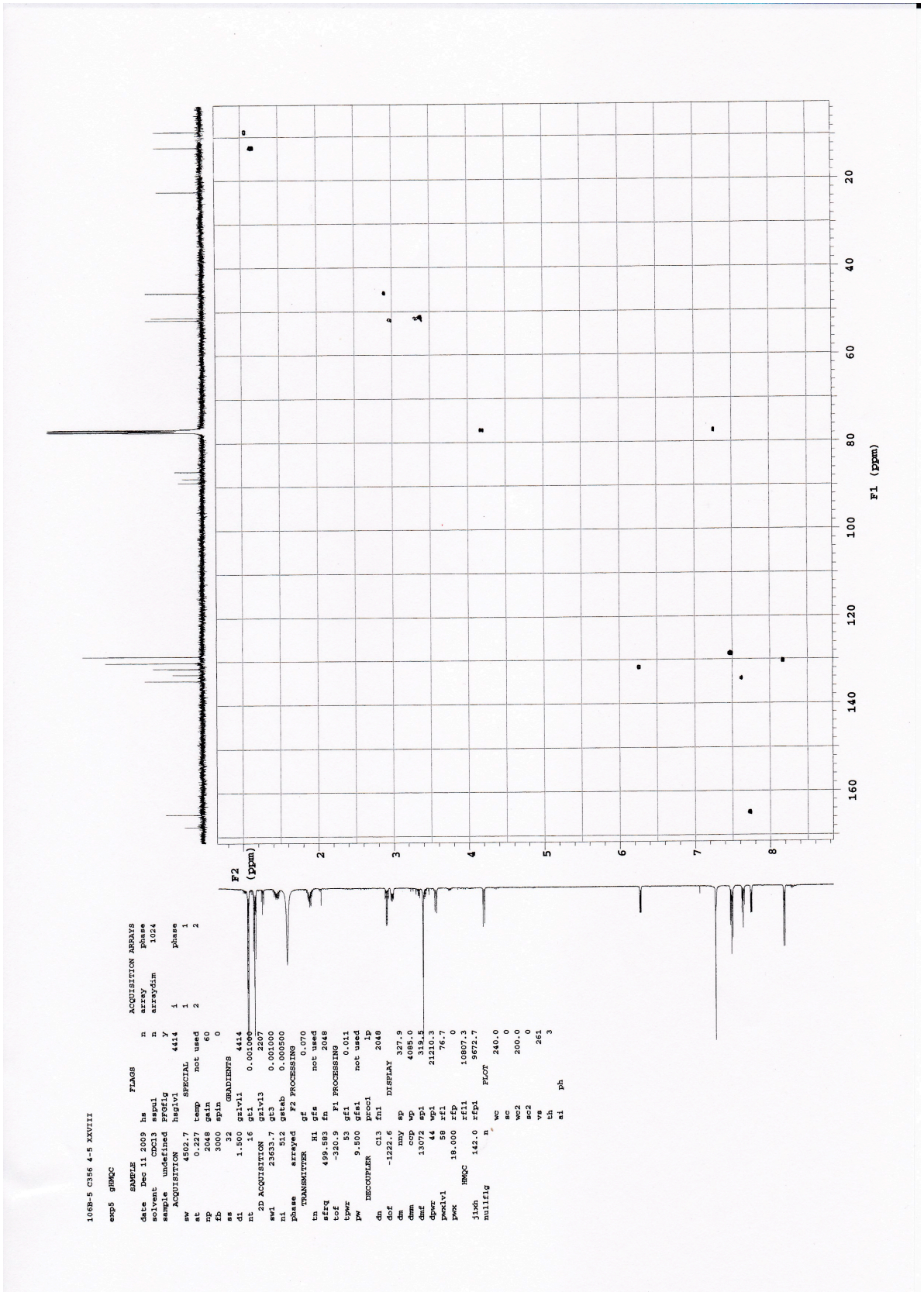


Figure S40 HMBC of **6**

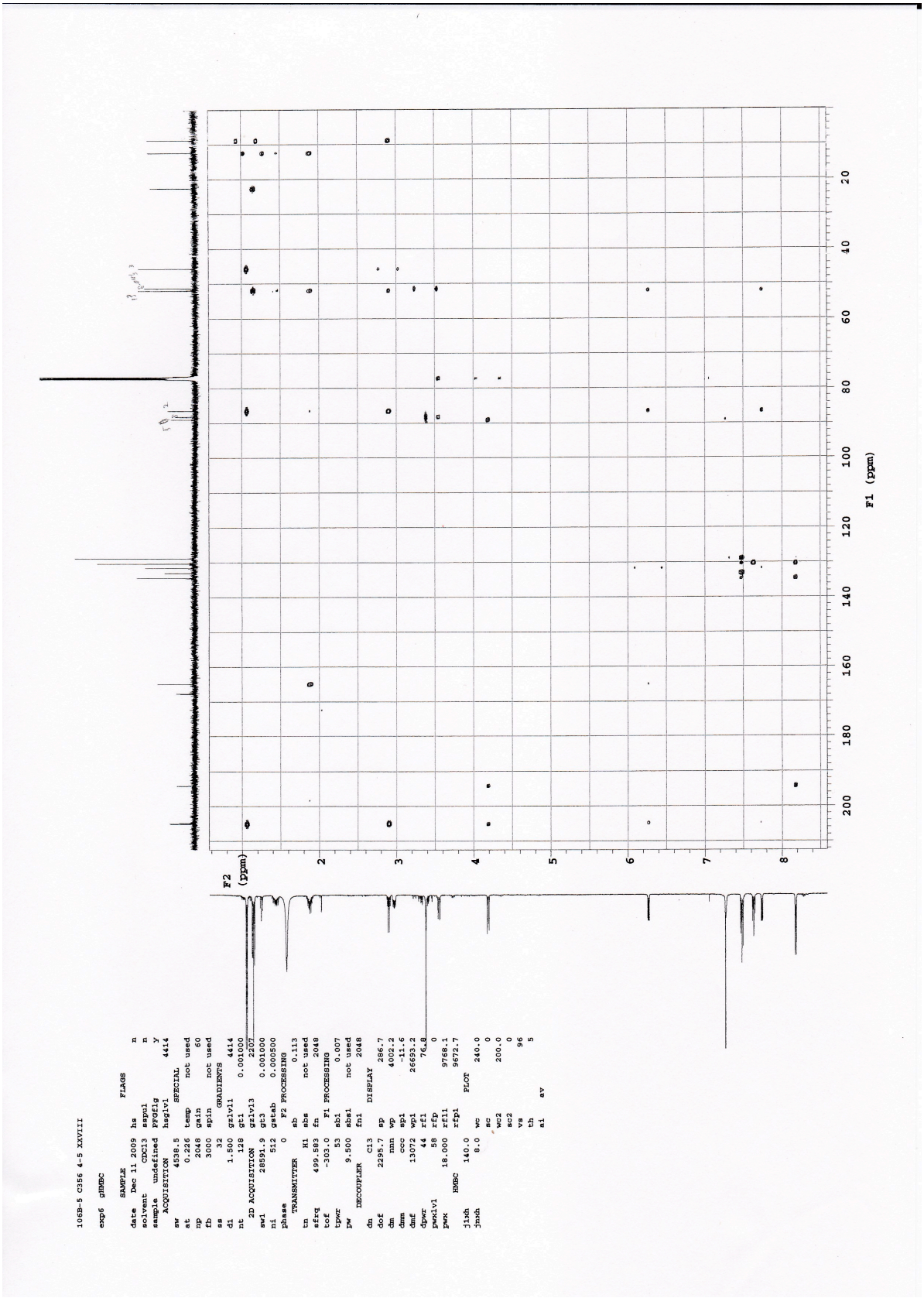


Figure S41 IR spectrum of **6**

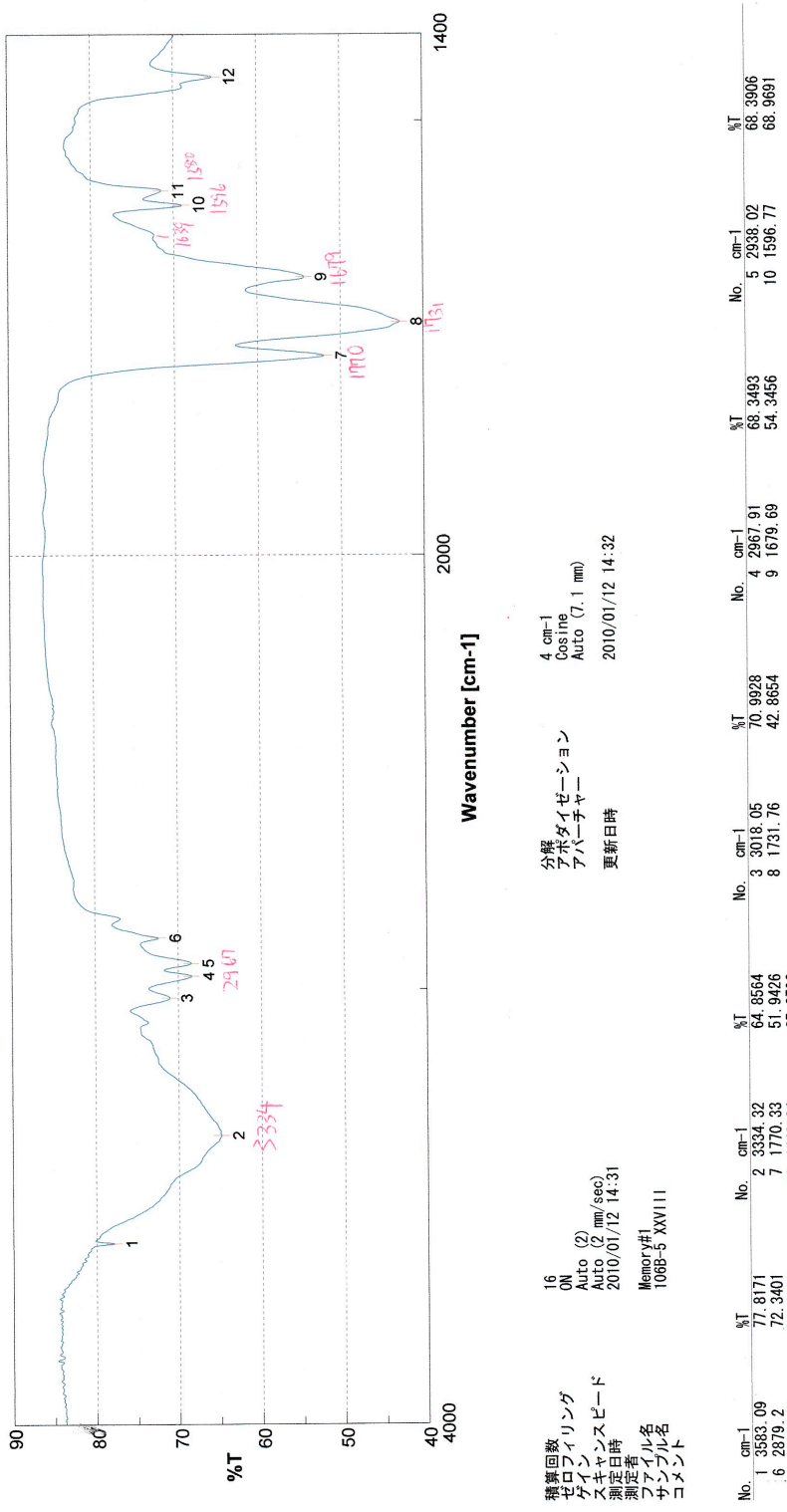


Figure S42 FABMS of **6**

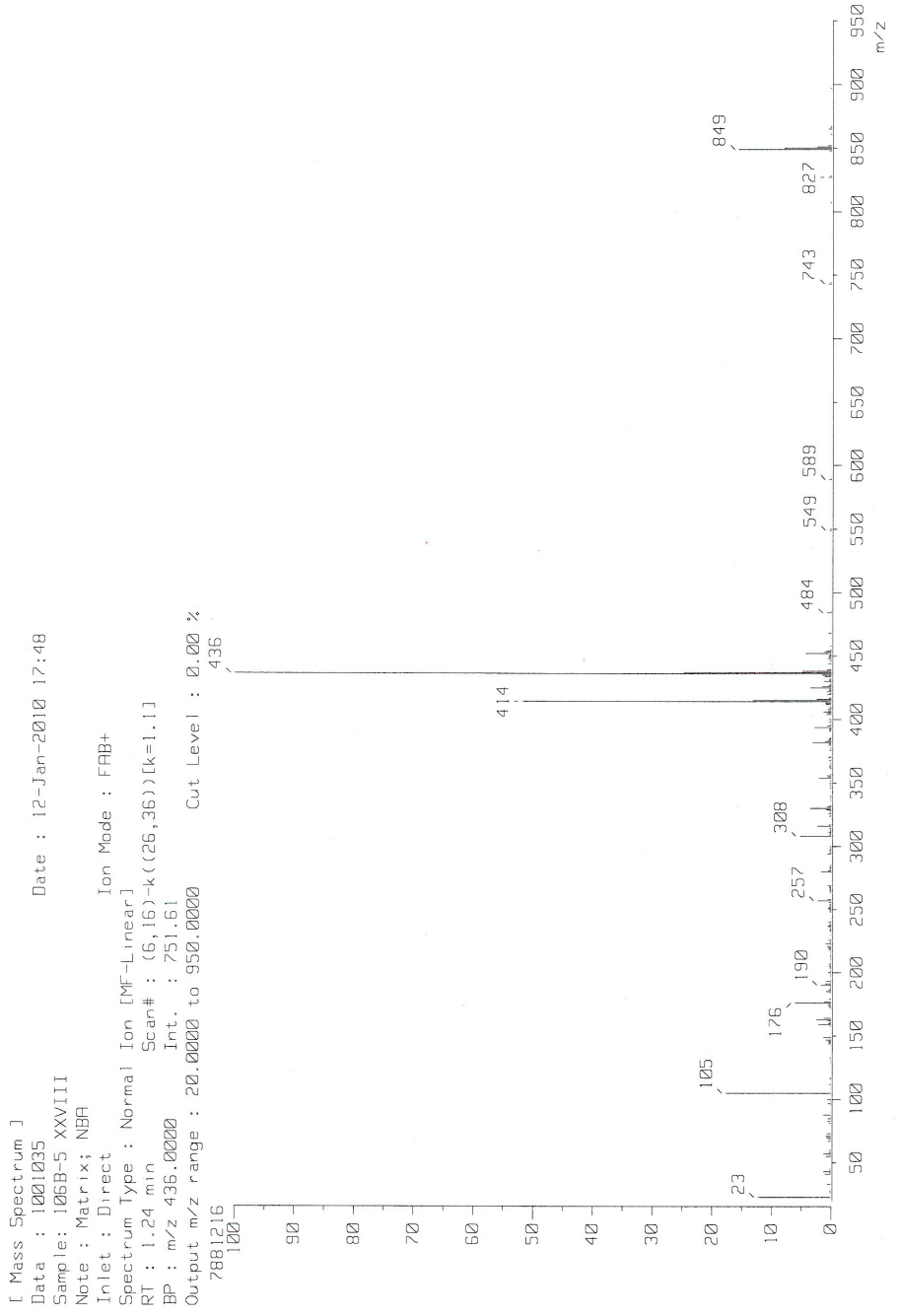


Figure S43 H and ^{13}C NMR spectra of **7** in $CDCl_3$

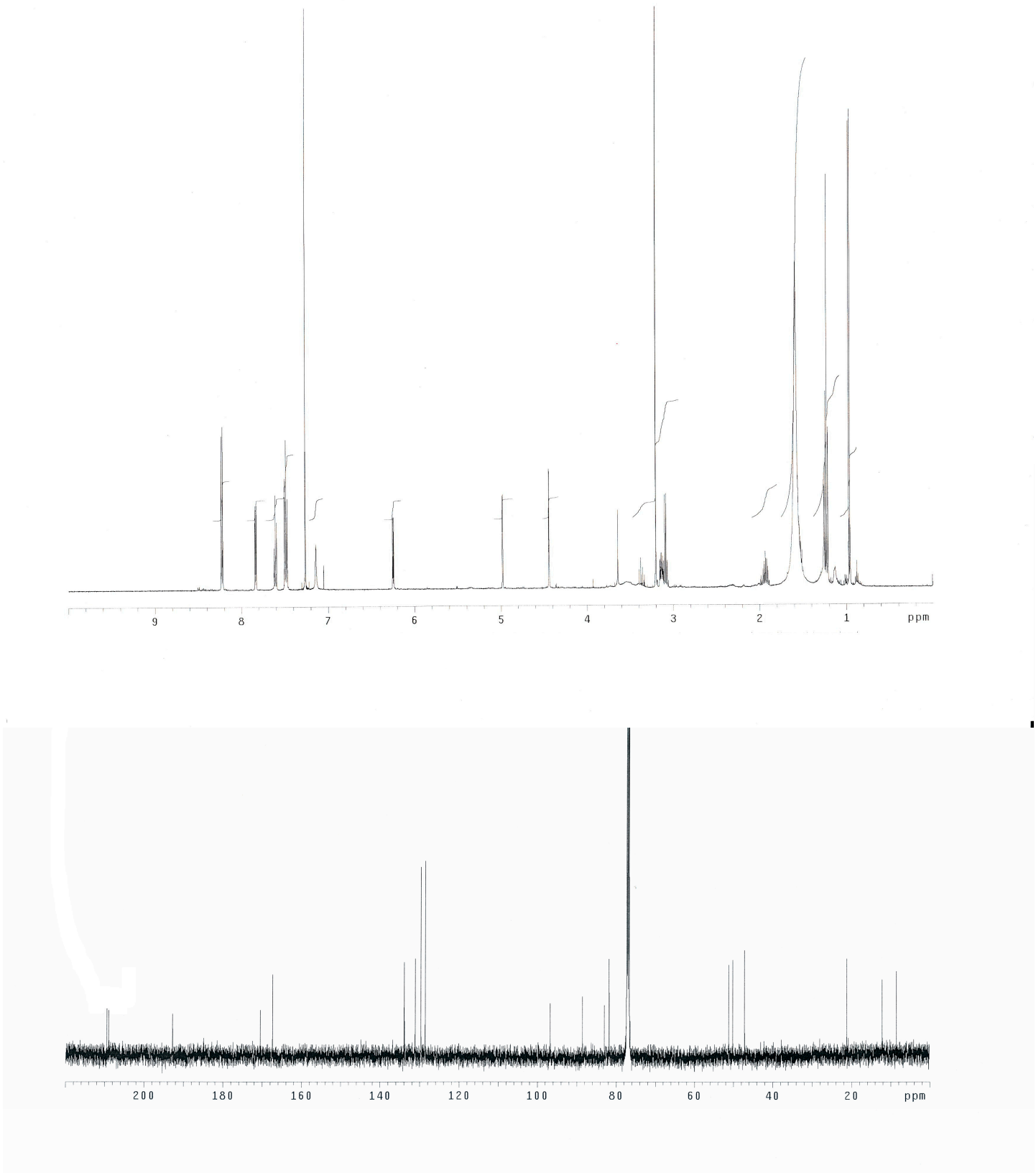
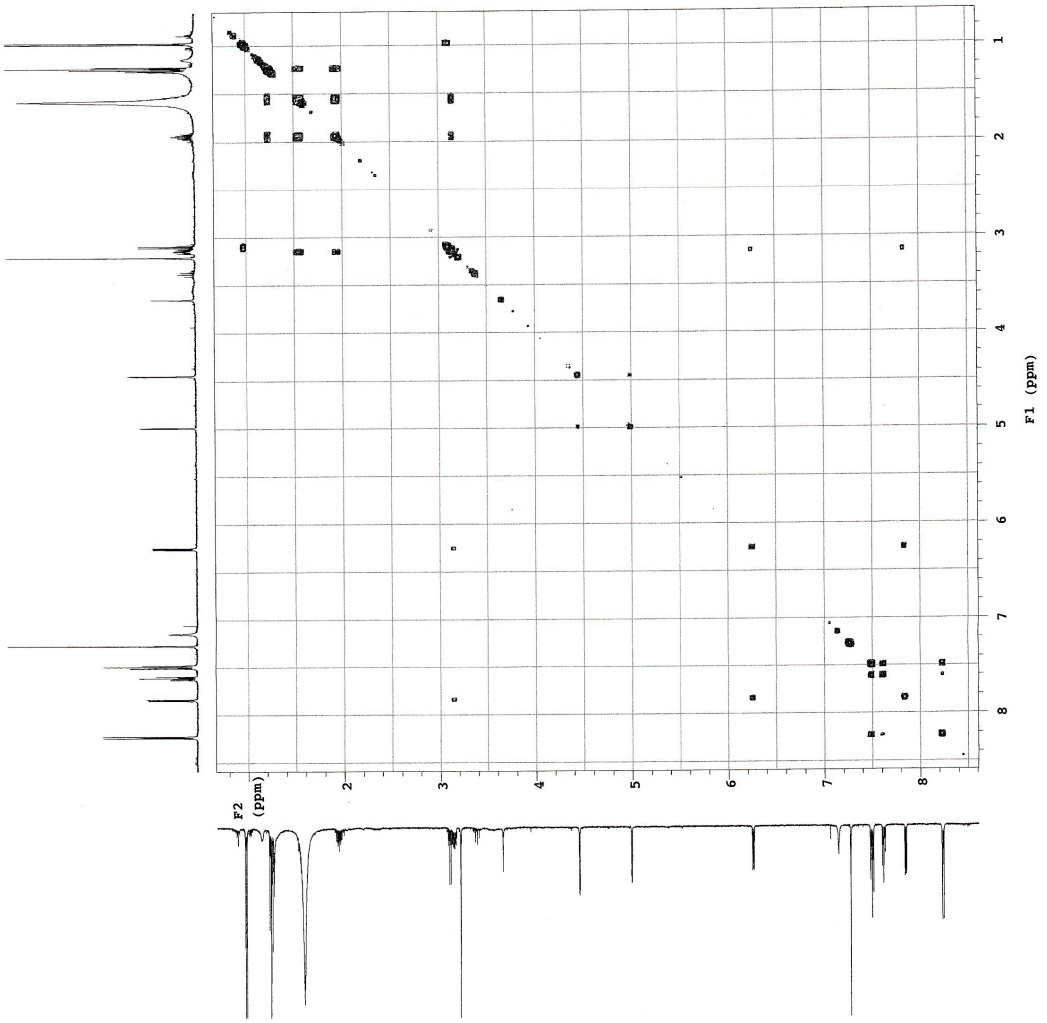


Figure S44 ^{1}H-^{1}H COSY of **7**

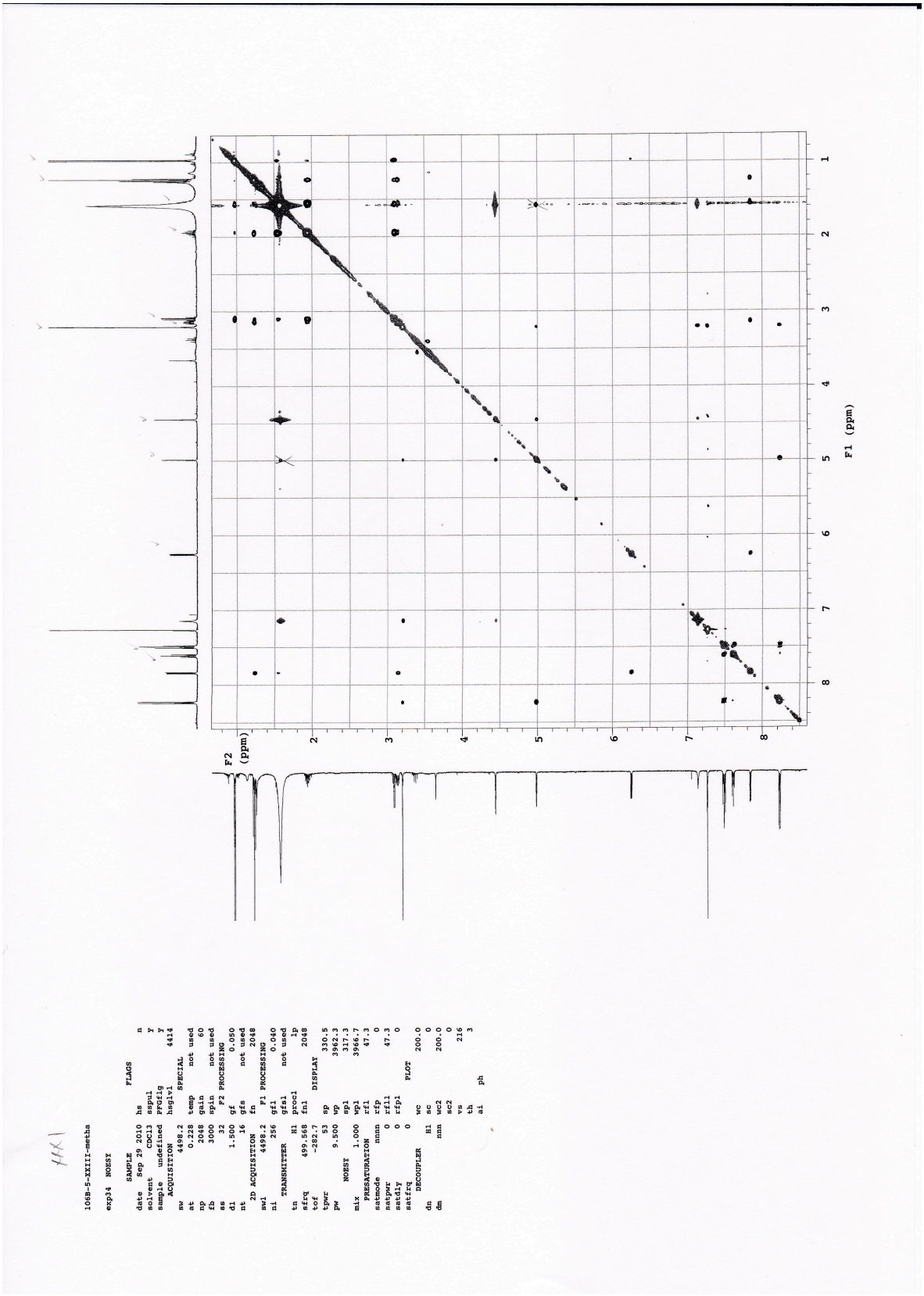


1068-5-XXIII-metha

exp33 gCOSY

SAMPLE FLAGS nn
date Sep 29 2010 hs nn
solvent CDCl3 sspul n
sample undefined hsglvl 4414
ACQUISITION SPECIAL
sw 4442.7 temp not used
at 0.230 gain 58
np 2048 spin not used
fb 2000 F2 PROCESSING 0
ss 16 ab -0.115
d1 1.000 sbs not used
nt 8 fn not used 2048
2D ACQUISITION F1 PROCESSING
sw1 4442.7 ab1 -0.029
ni 256 abs1 not used
TRANSMITTER pccol lp
tn H1 fn1 2048
sfrq 499.568 DISPLAY
tof -347.7 sp 332.1
tpwr 53 wp 3965.5
pw 9.500 sp1 319.0
GRADIENTS wp1 3969.8
gzlvl1 4414 rf1 84.4
gt1 0.001000 rfp 0
gstab 0.000500 rfl1 84.5
DECOUPLER rfp1 0
dn H1 PLOT
dm nnn wc 200.0
sc 0
wc2 200.0
sc2 0
vs 216
th 4
ai av

Figure S45 NOESY of **7**



1068-5-XXIII-metha

exp34 NOESY

SAMPLE FLAGS
date Sep 29 2010 hs n
solvent CDCl3 ssgnl y
sample undefined ffGflg y
acqfil 4414
ACQUISITION hsglvl
sw 4498.2 SPECIAL
at 0.228 temp not used
np 2048 gain 60
fb 3000 spin not used
ss 32 F2 PROCESSING
d1 1.500 gf 0.050
nt 16 gfs not used
2D ACQUISITION fn 2048
sw1 4498.2 F1 PROCESSING
ni 256 gfl 0.040
TRANSMITTER gfs1 not used
tn H1 proc1 lp
sfrq 499.568 fn1 2048
tof -282.7 DISPLAY
tpwr 53 sp 330.5
pw 9.500 wp 3962.3
NOESY sp1 317.3
mix 1.000 wp1 3966.7
PRESATURATION sf1 47.3
satmode nnn rfl 0
satpwr 0 rfl1 47.3
satdly 0 rfp1 0
satfrq PLOT
DECOUPLER wc 200.0
dn H1 sc 0
dm nnn wc2 200.0
sc2 0
vs 216
th 3
ai ph

Figure S46 HMQC of **7**

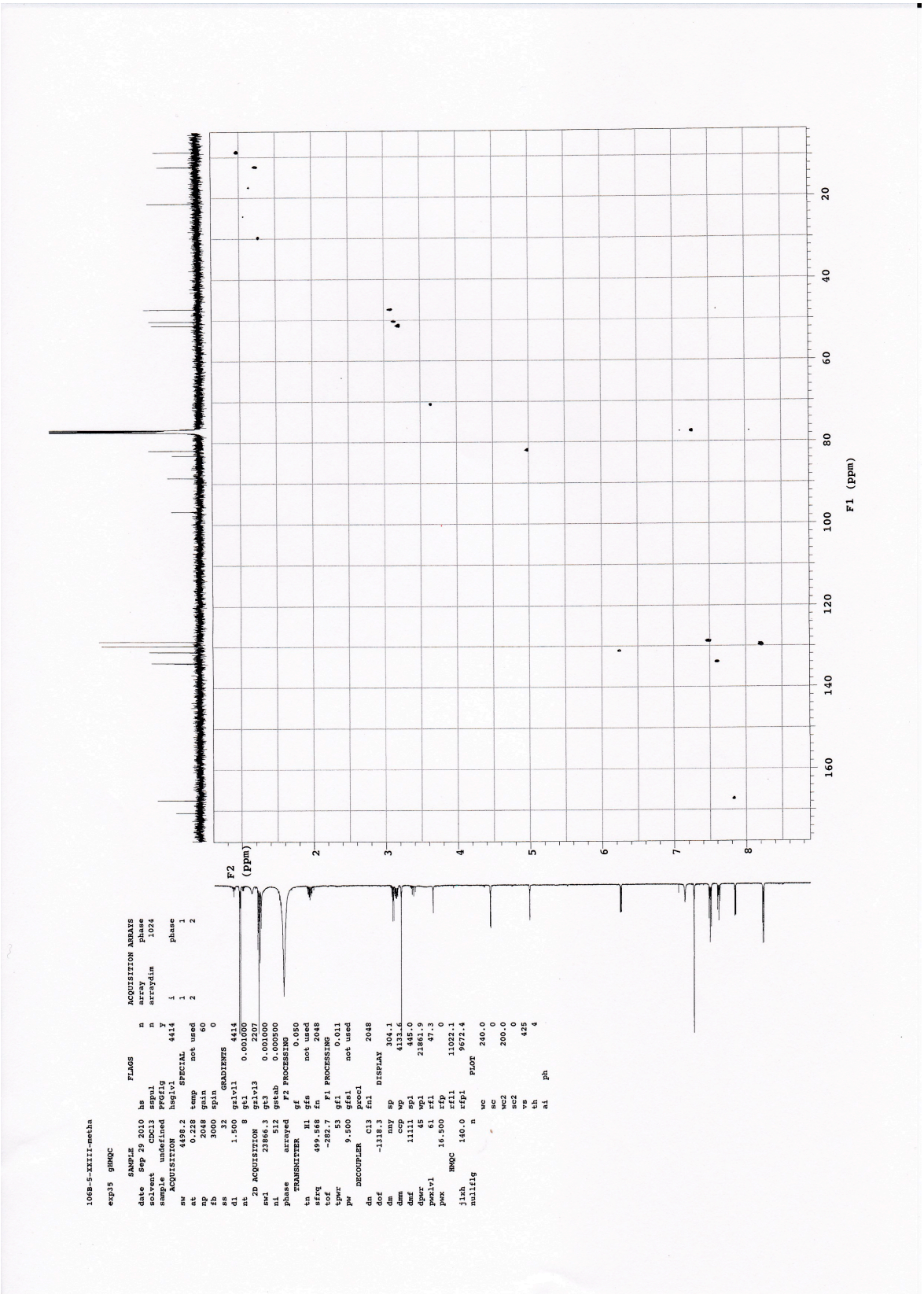


Figure S47 HMBC of **7**

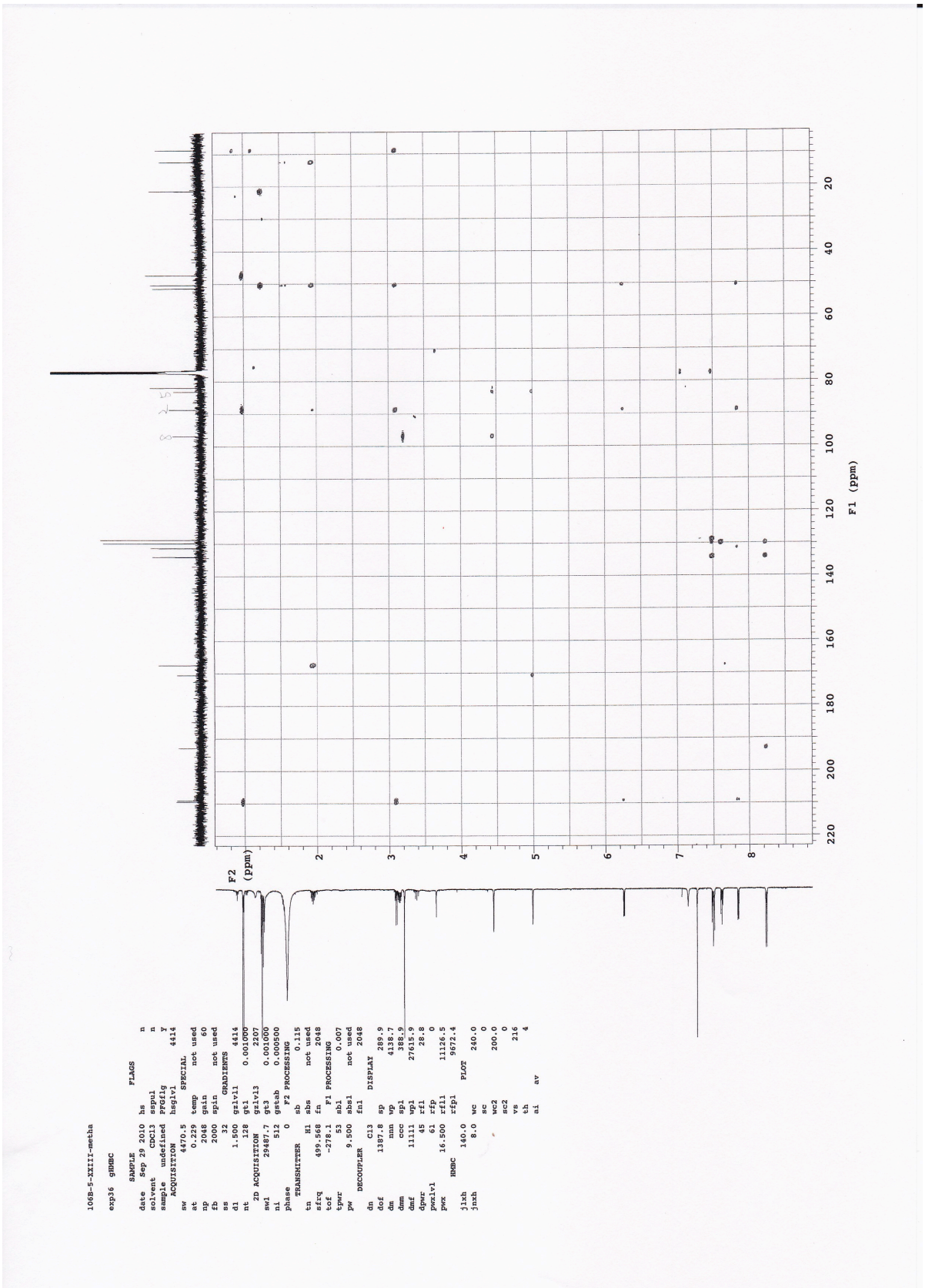


Figure S48 IR spectrum of **7**

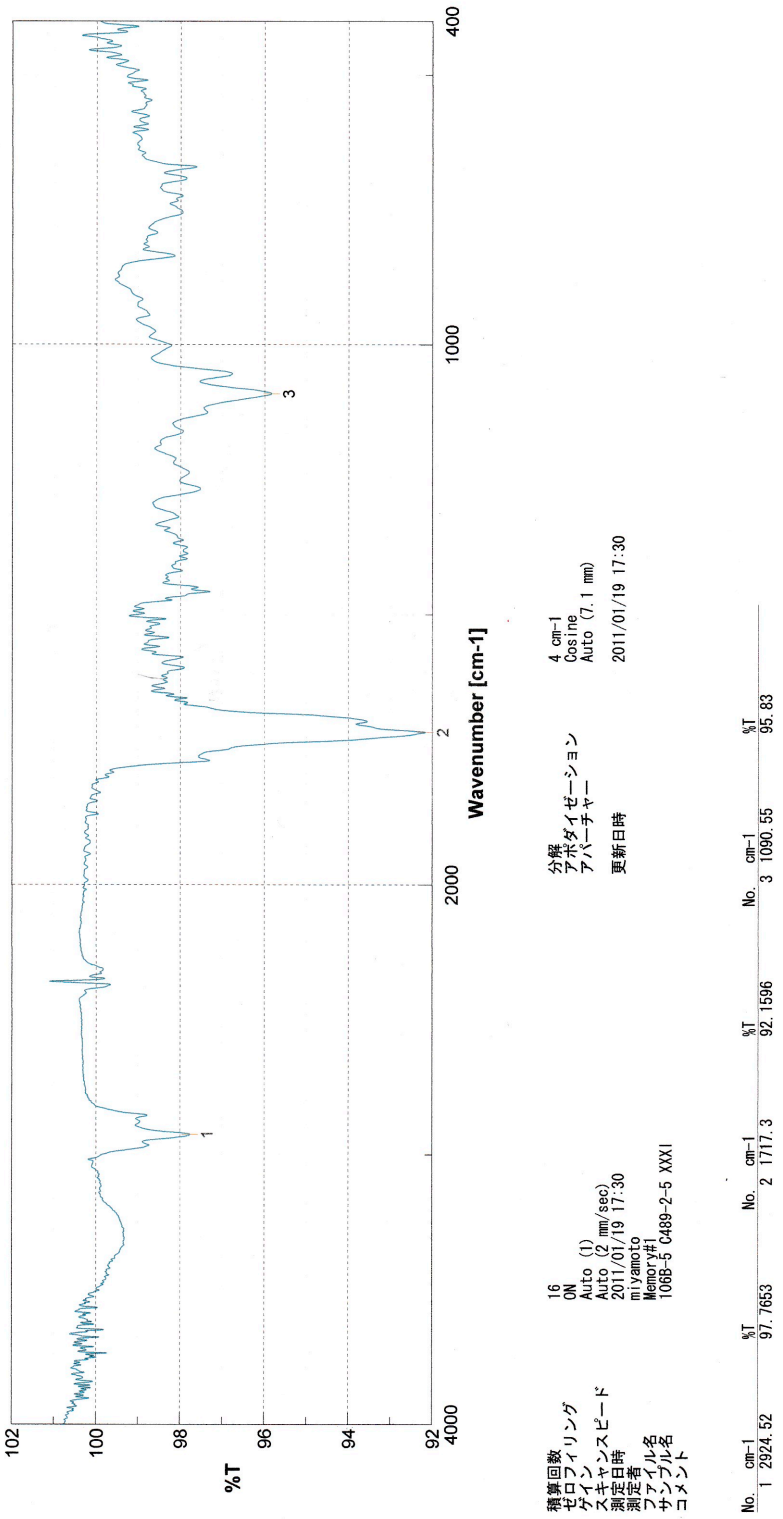


Figure S49 FABMS of **7**

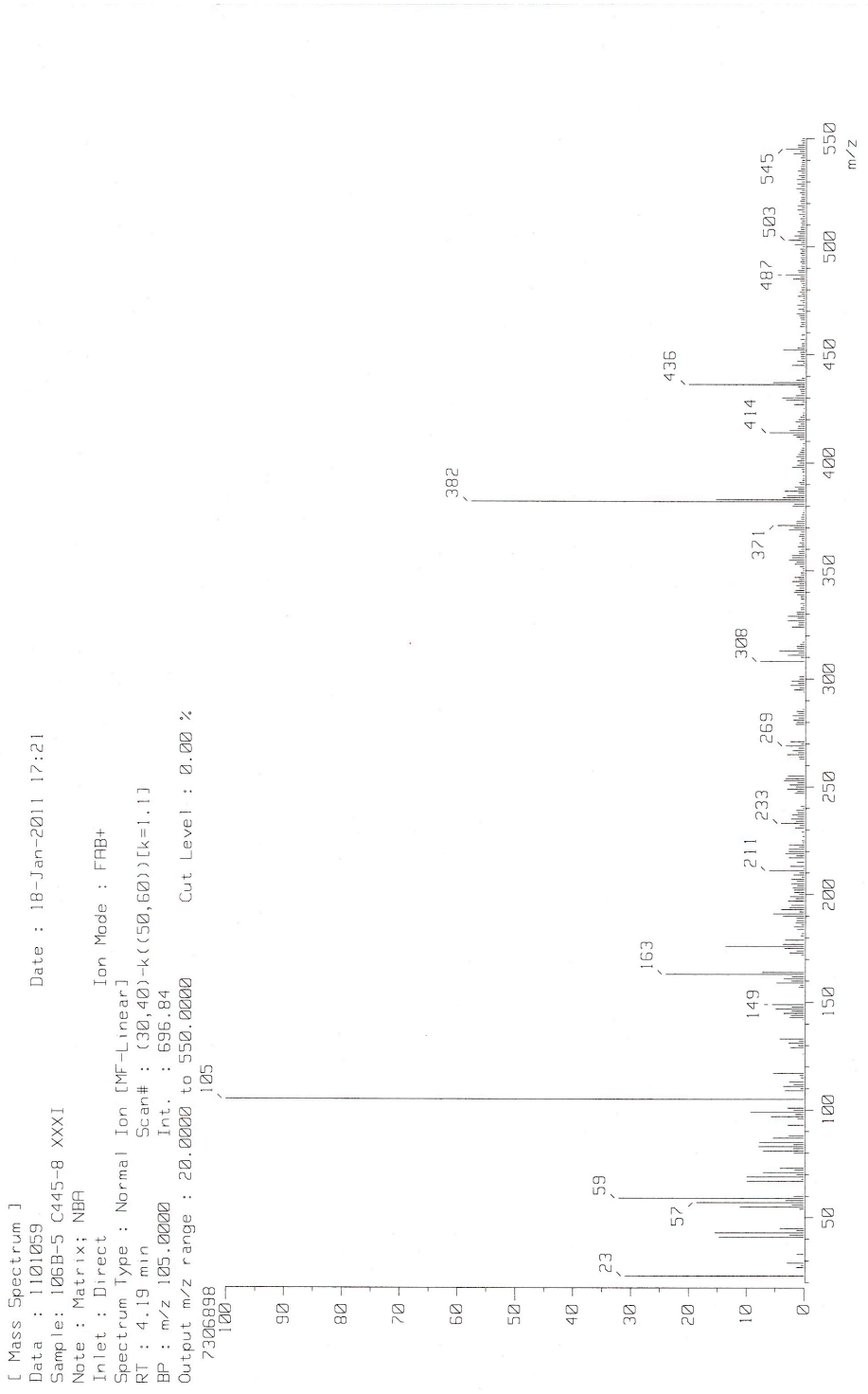


Figure S50^{1}H and ^{13}C NMR spectra of **8** in $CDCl_3$

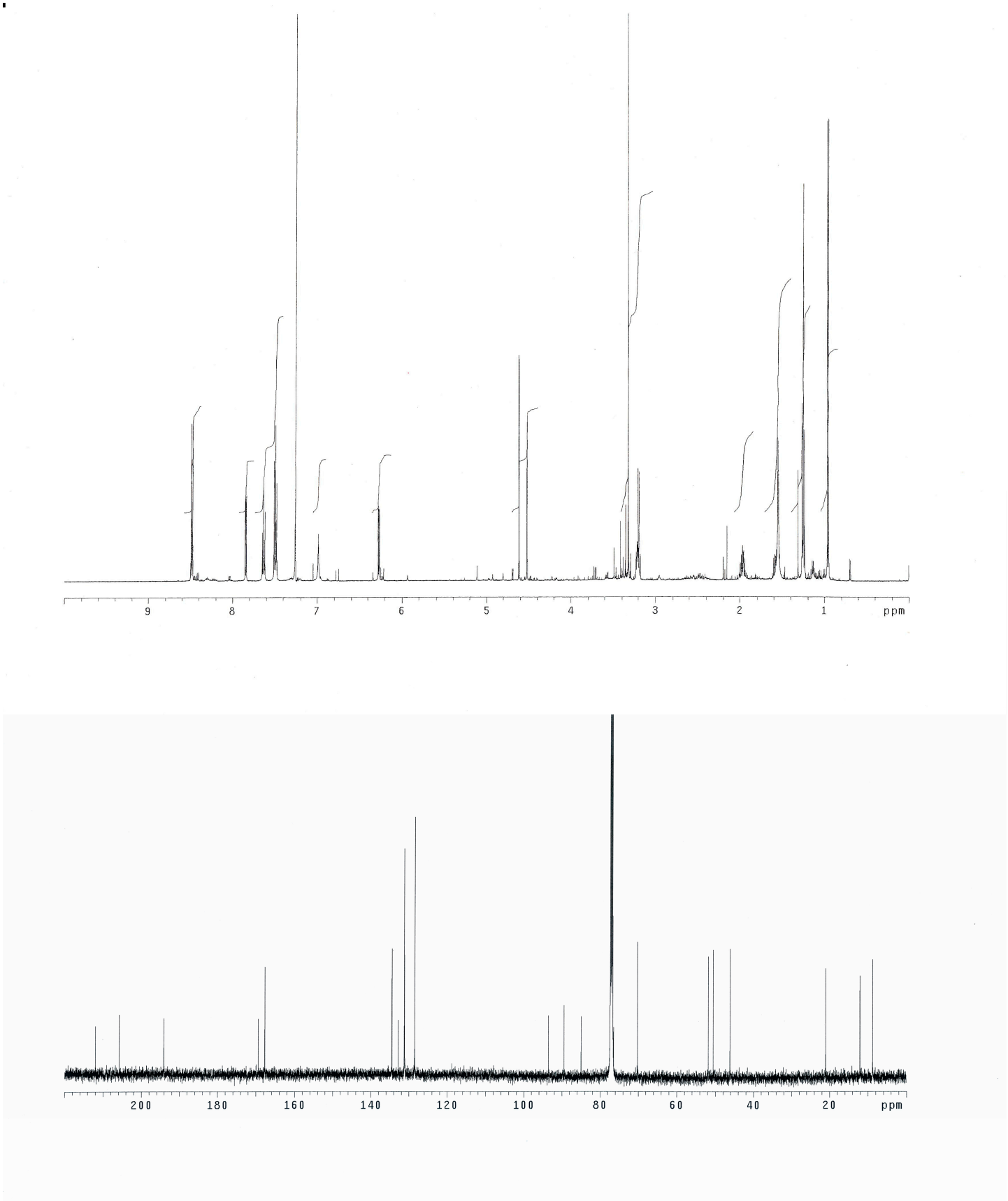
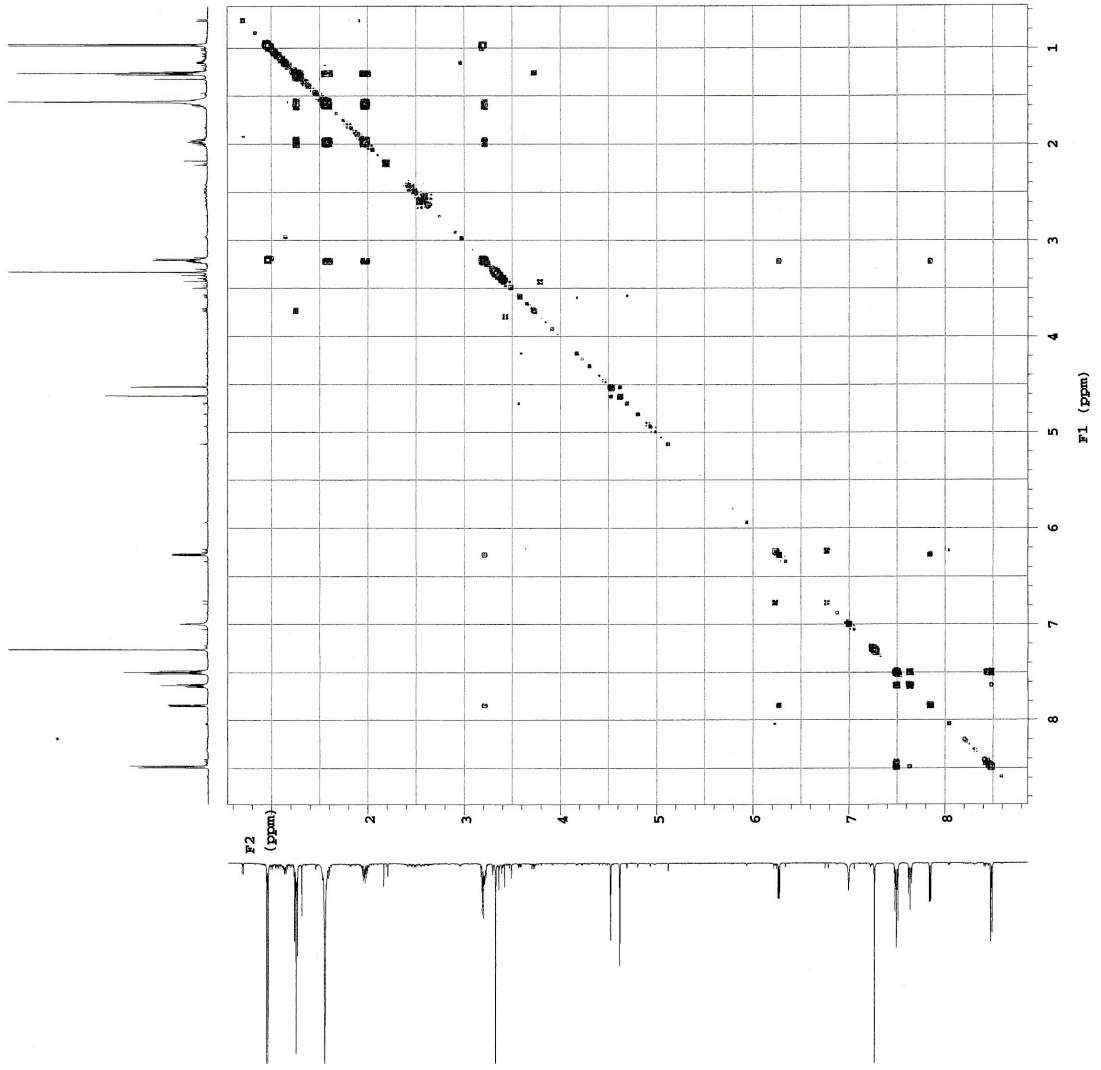


Figure S51 ^{1}H-^{1}H COSY of **8**



1068-5 C259-8 XXII

exp3 gCOSY

SAMPLE FLAGS
date Apr 2 2009 hs nn
solvent CDCl3 sspul n
sample undefined hsglv1 4414
ACQUISITION SPECIAL
sw 4524.4 temp not used
at 0.226 gain 58
np 2048 spin not used
fb 3000 F2 PROCESSING
ss 16 sb -0.113
d1 1.000 sba not used
nt 16 fn 2048
2D ACQUISITION F1 PROCESSING
sw1 4524.4 sb1 -0.028
ni 256 sba1 not used
TRANSMITTER prcol 1p
tn H1 fn1 2048
sfrq 499.598 DISPLAY
tof -305.6 sp 272.6
tpwr 53 wp 4153.2
pw 9.500 sp1 277.1
GRADIENTS wp1 4157.7
gzlvl1 4414 rf1 76.4
gt1 0.001000 rfp 0
gstab 0.000500 rfl1 76.4
DECOUPLER rfp1 0
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sc 0
wc2 200.0
sc2 0
vs 798
th 4
ai cdc av

Figure S52 NOESY of **8**

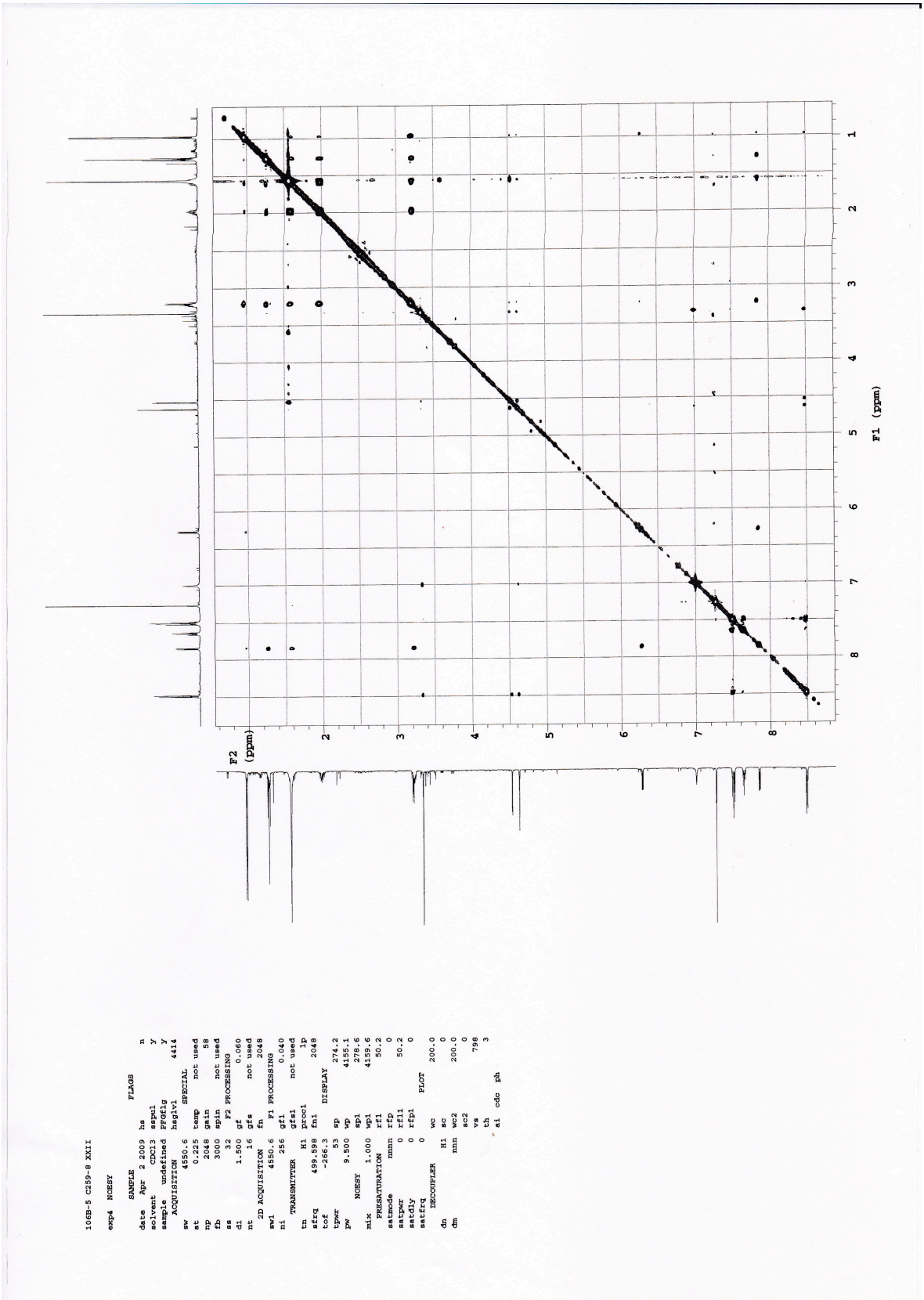


Figure S53 HMQC of **8**

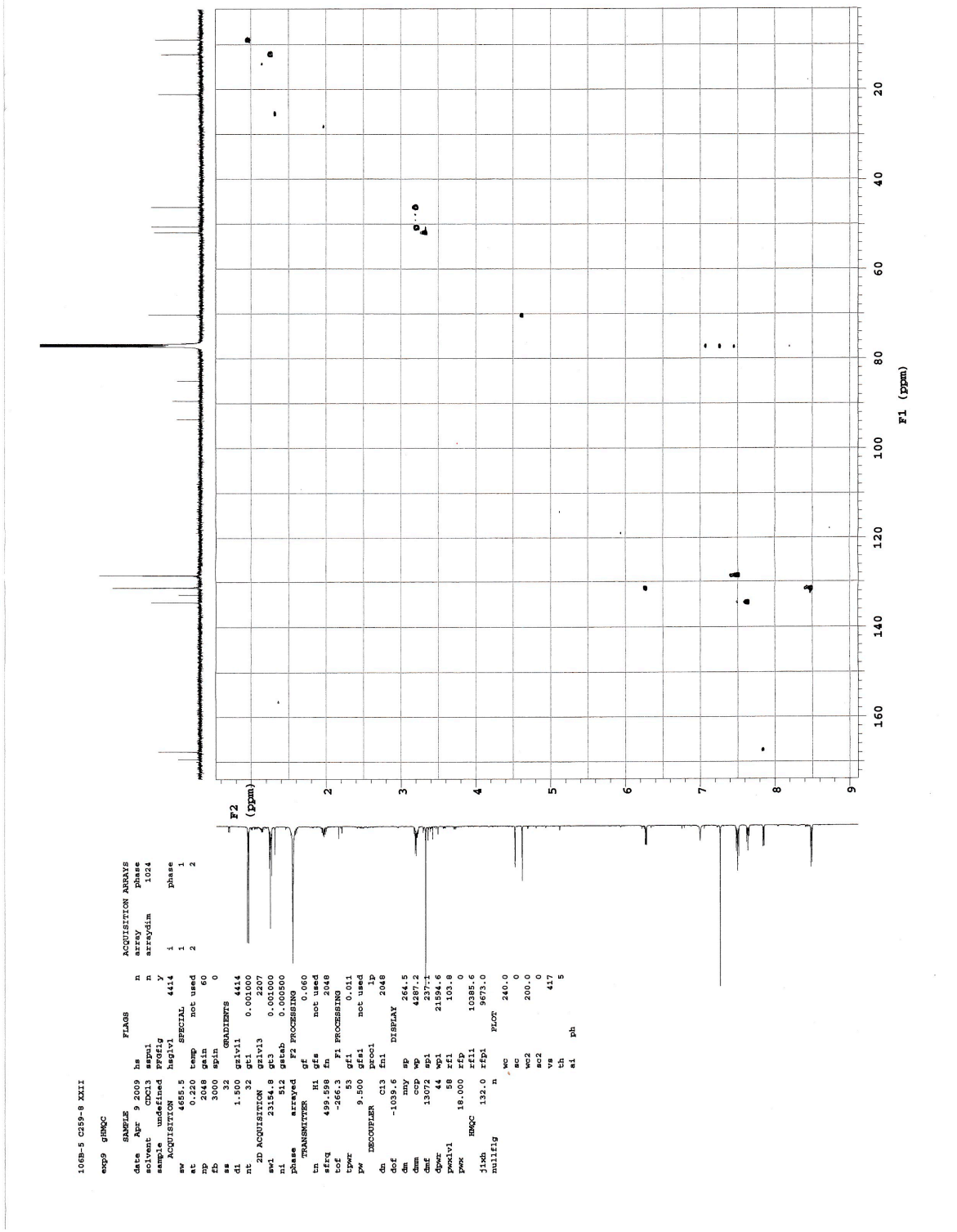


Figure S54 HMBC of **8**

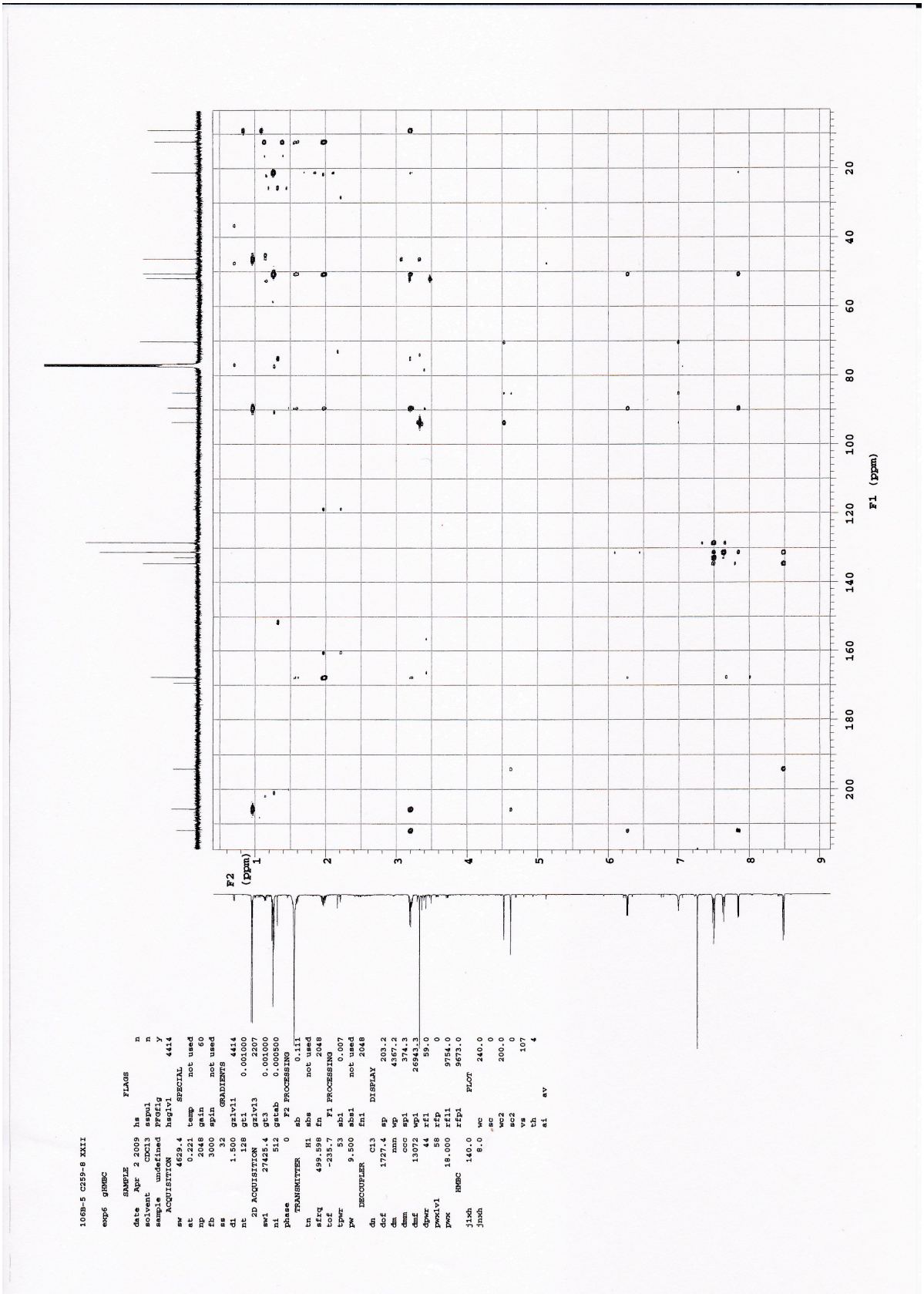


Figure S55 IR spectrum of **8**

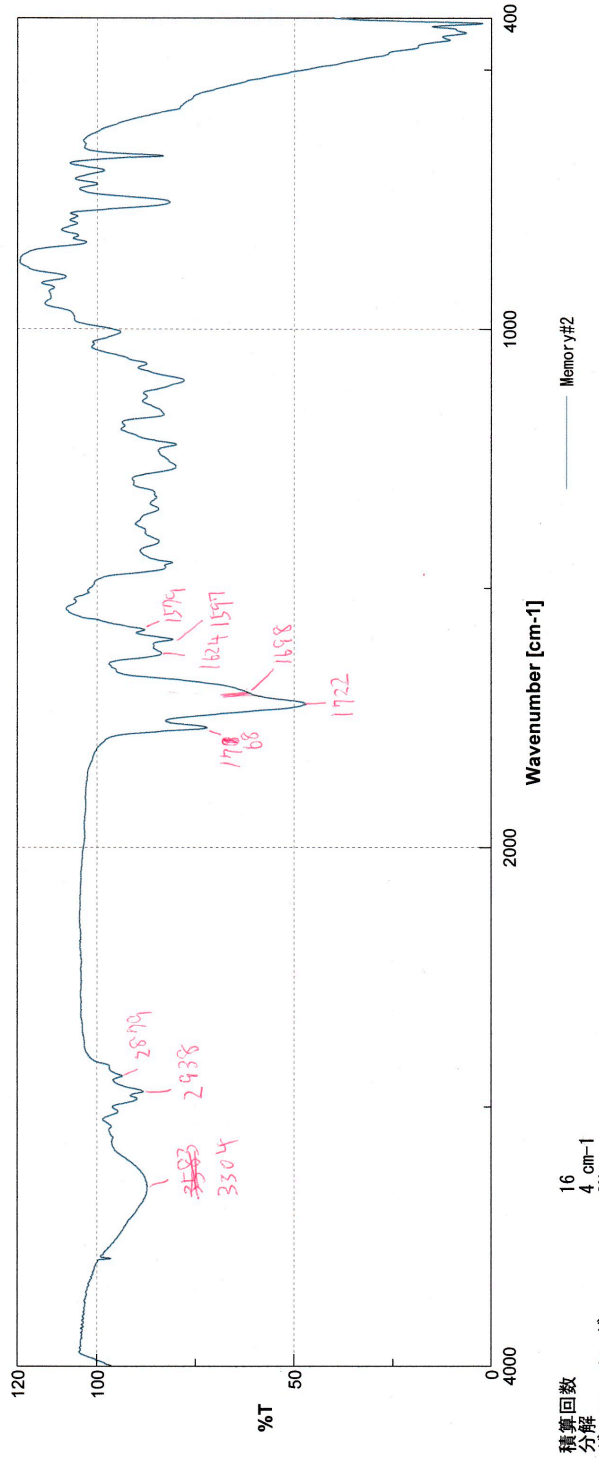


Figure S56 FABMS of **8**

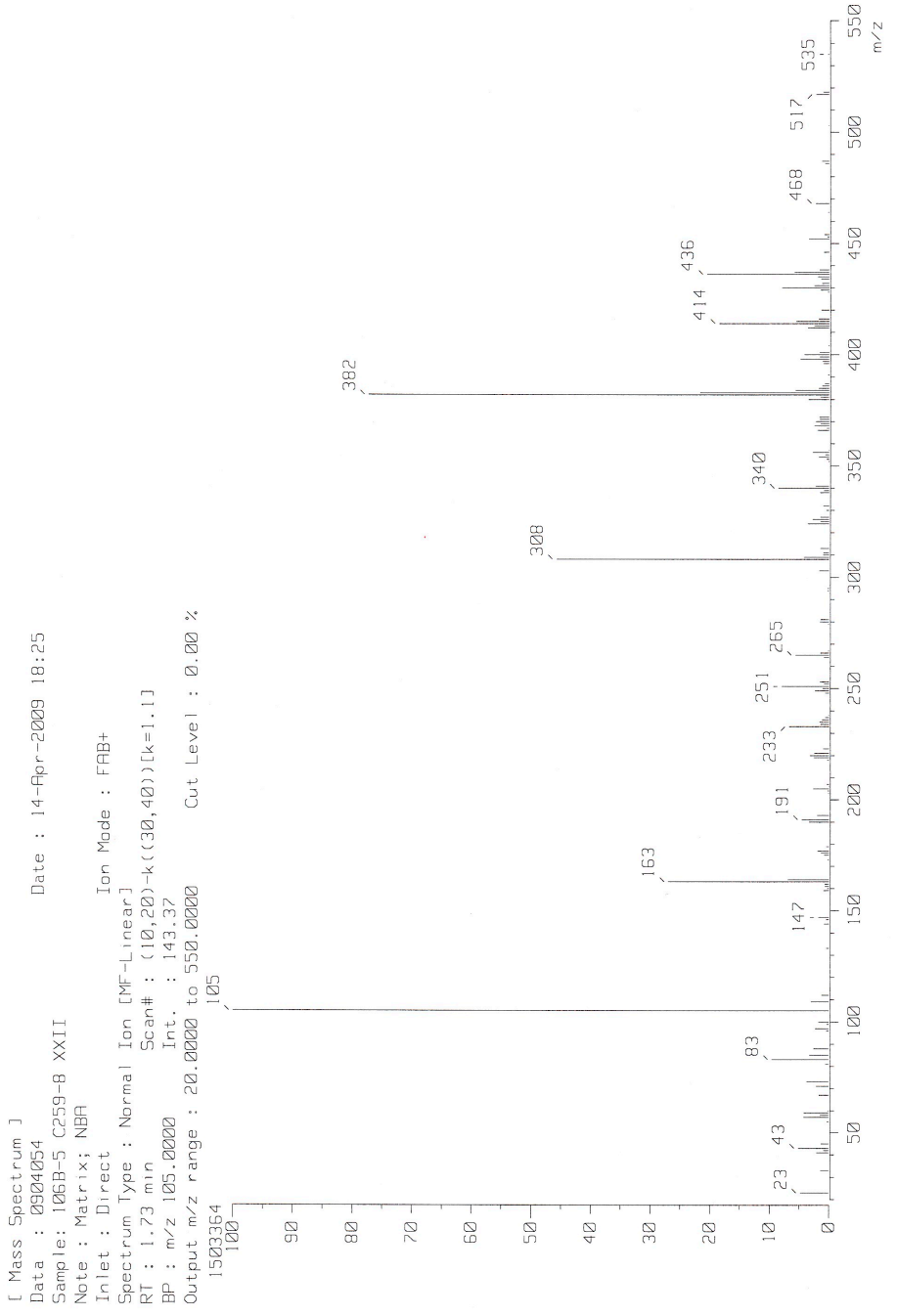


Figure S57 ^{1}H NMR spectrum of **1'**

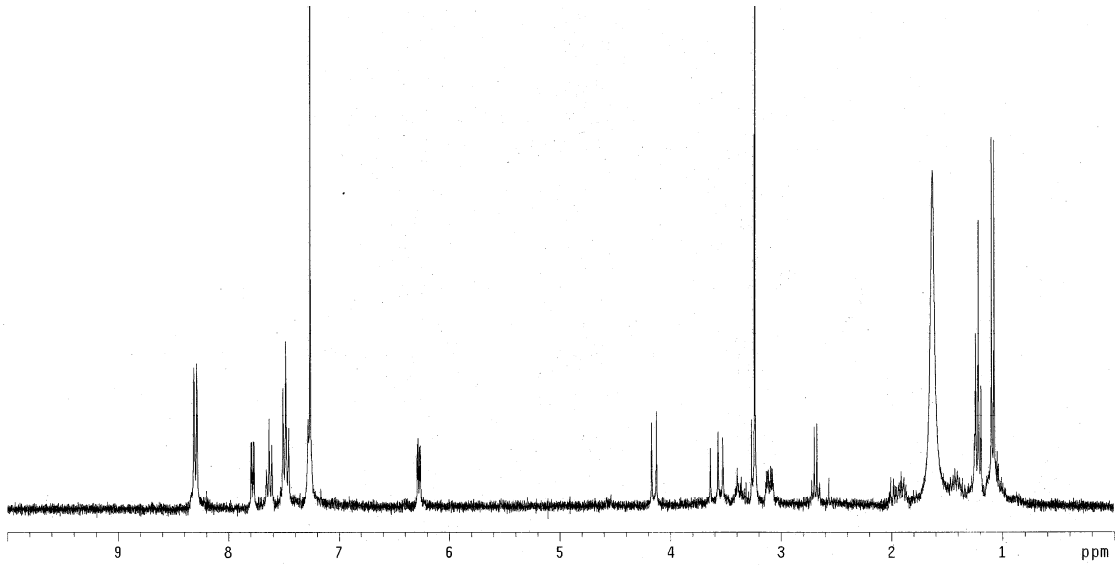


Figure S58 ^{1}H NMR spectrum of **2'**

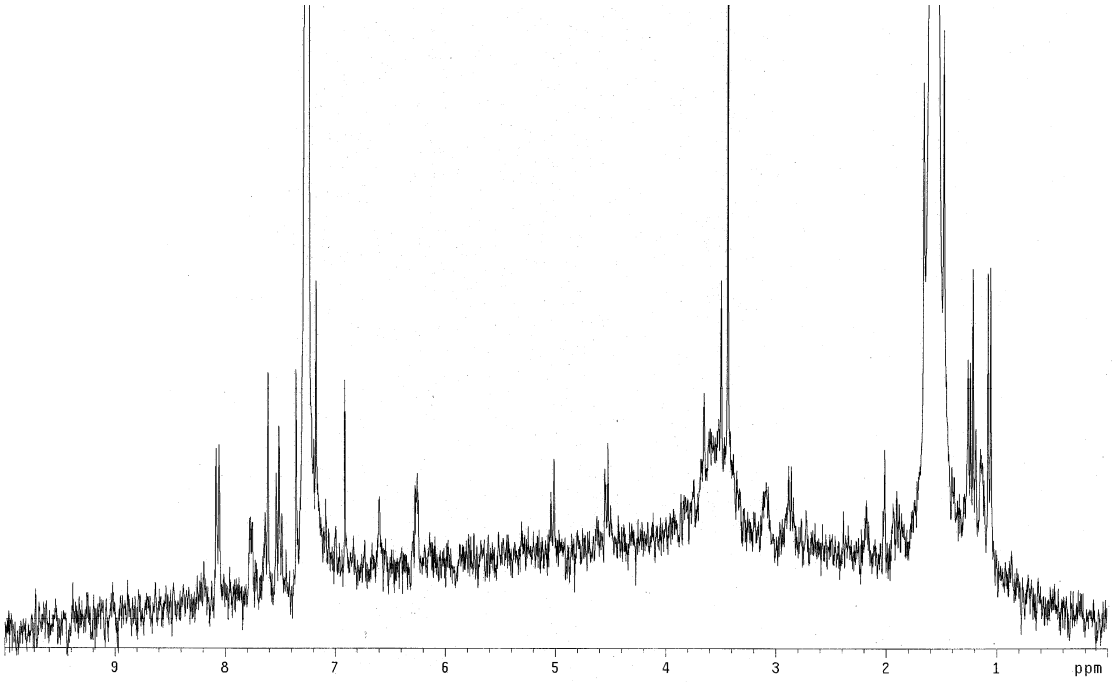


Figure S59 ^{1}H NMR spectrum of **3'**

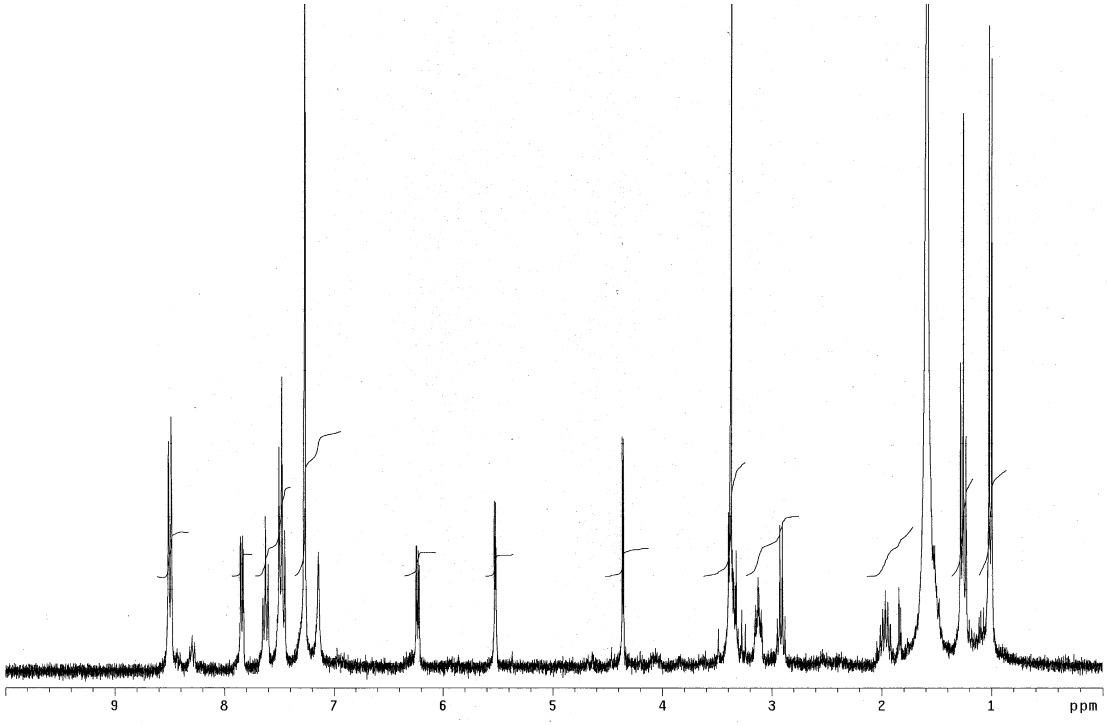


Figure S60 ^{1}H NMR spectrum of **4'**

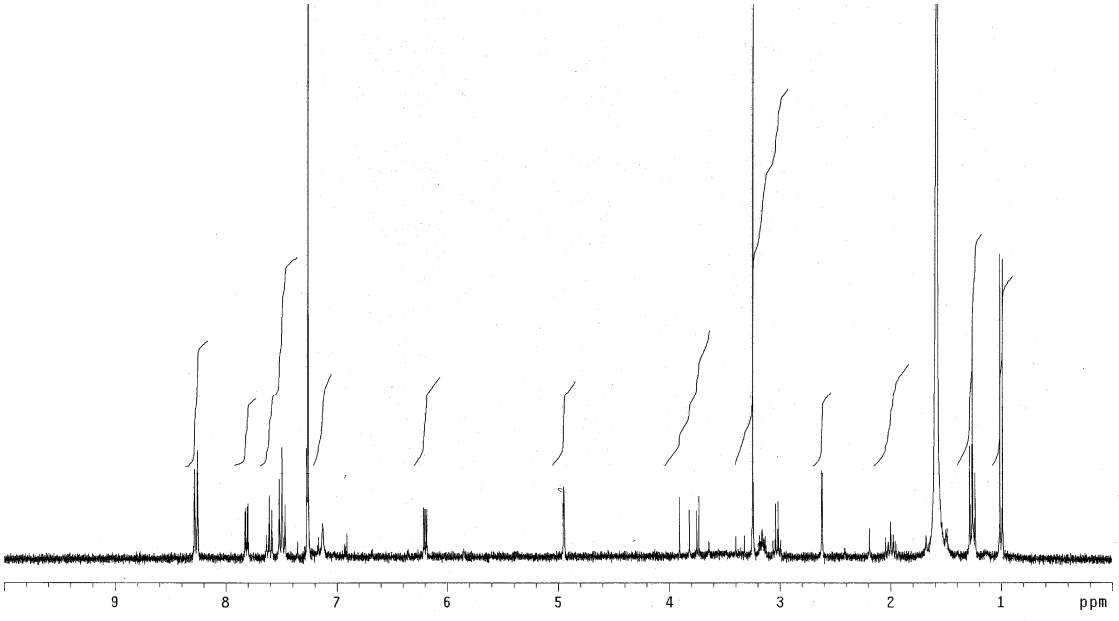


Figure S61 ^{1}H NMR spectrum of **5'**

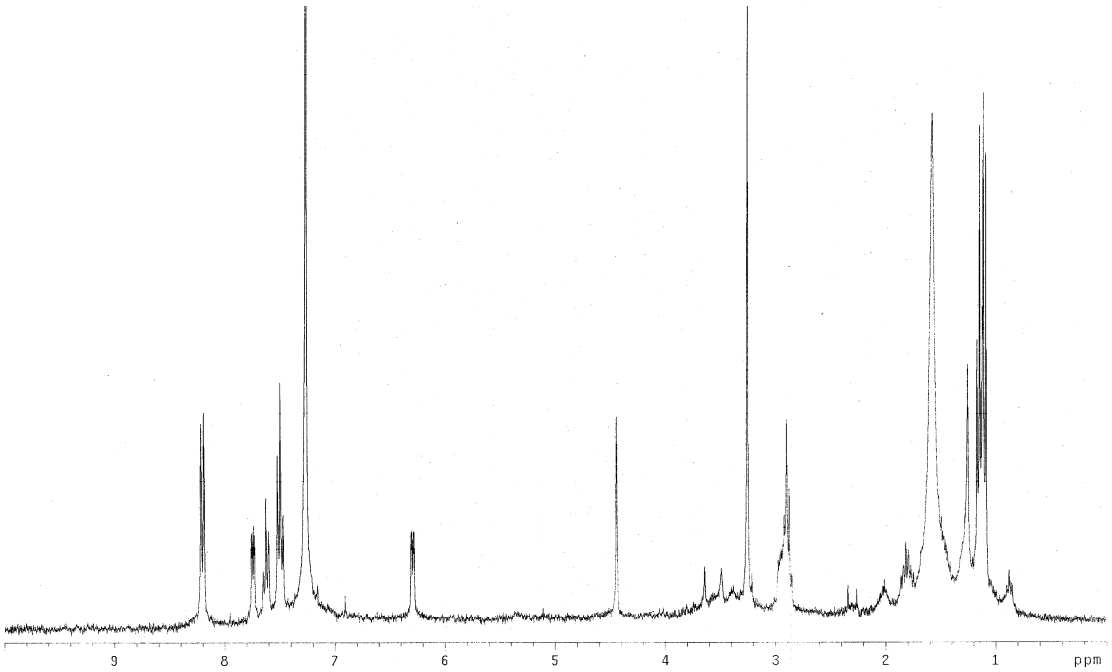


Figure S62 ^{1}H NMR spectrum of **6'**

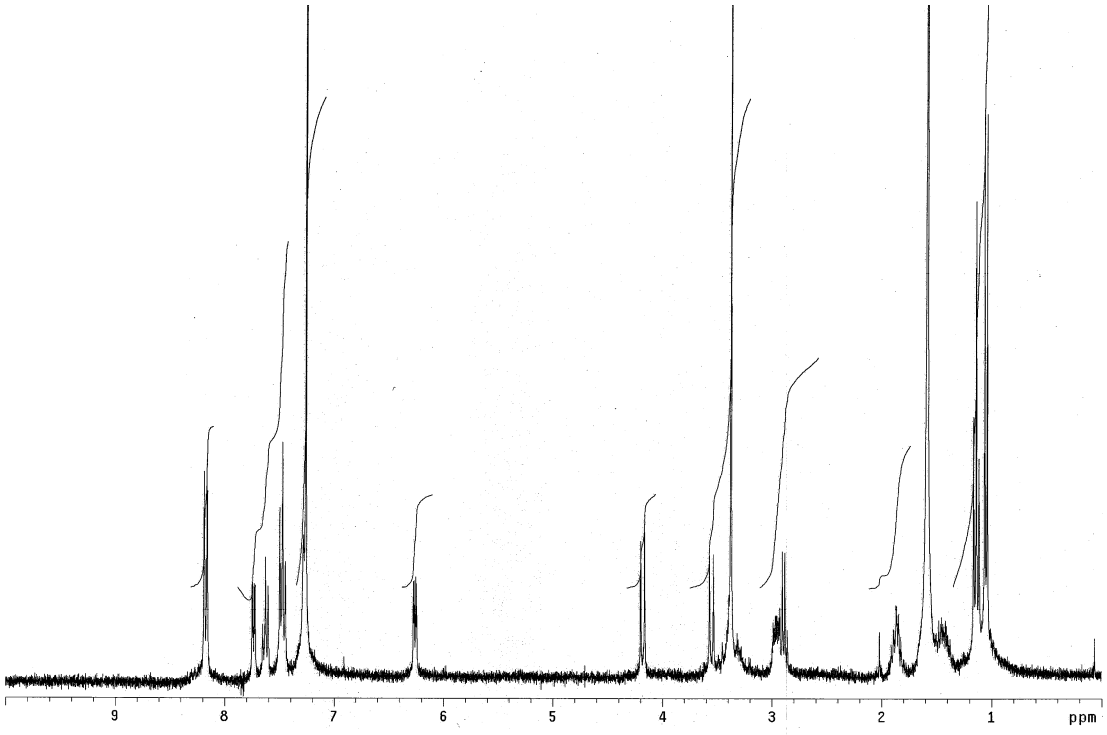


Figure S63 ^{1}H NMR spectrum of **7'**

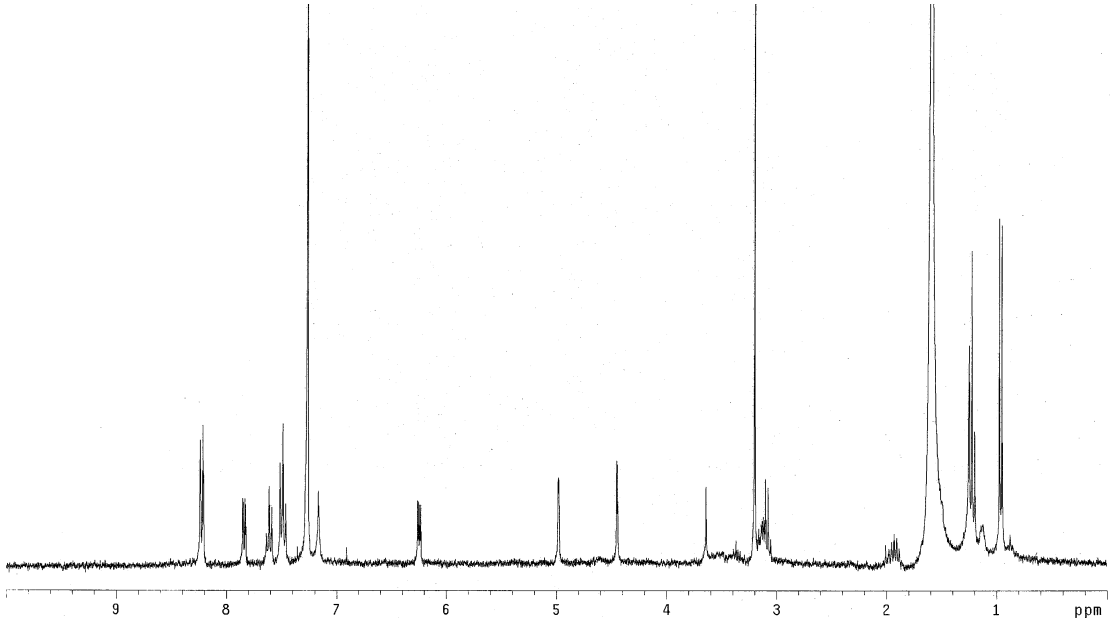
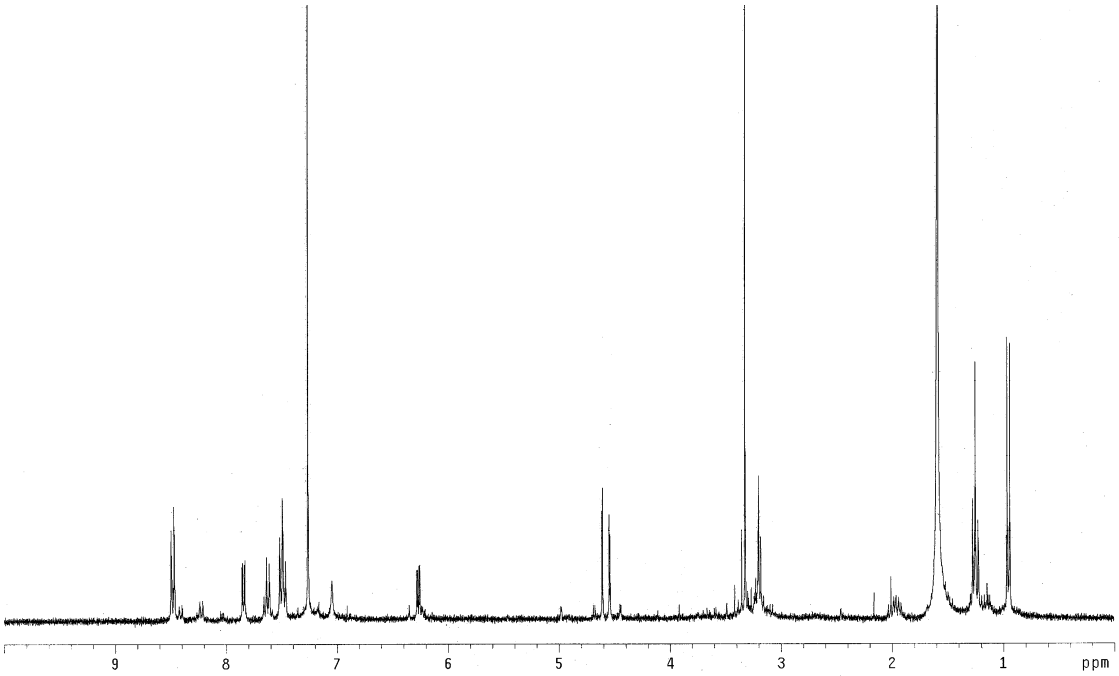


Figure S64 ^{1}H NMR spectrum of **8'**



cephalimysin E (**1**)

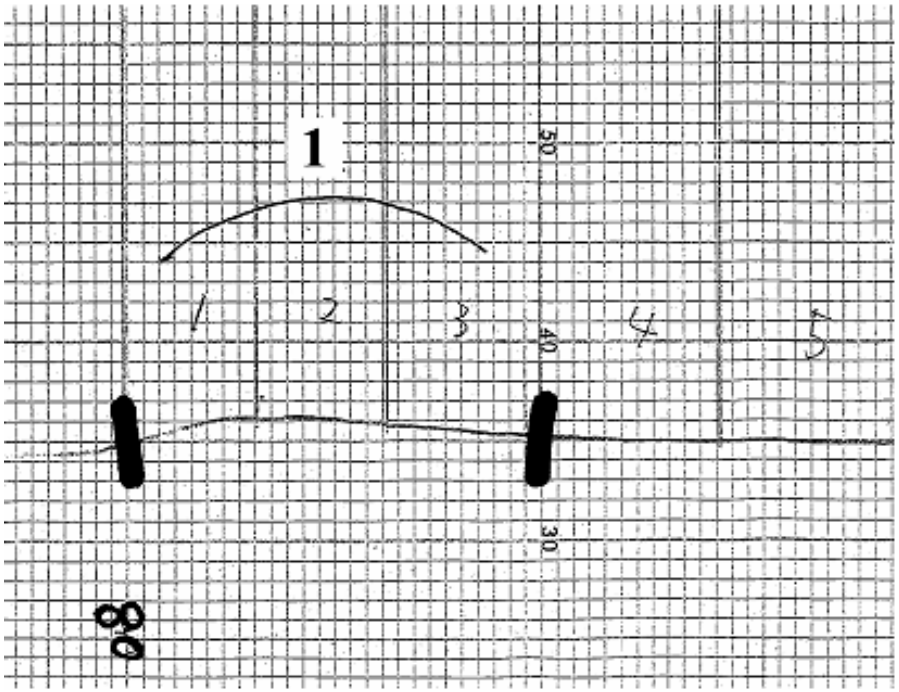


Figure S65

Moble phase CH_3CN / H_2O = 40 / 60, 4ml/min

cephalimysin F (**2**)

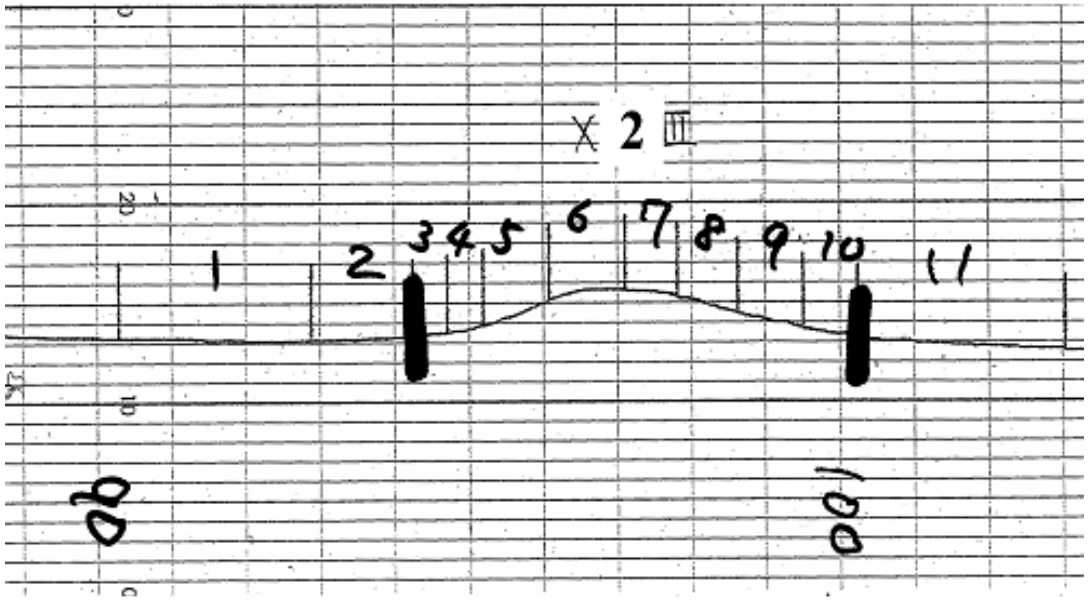
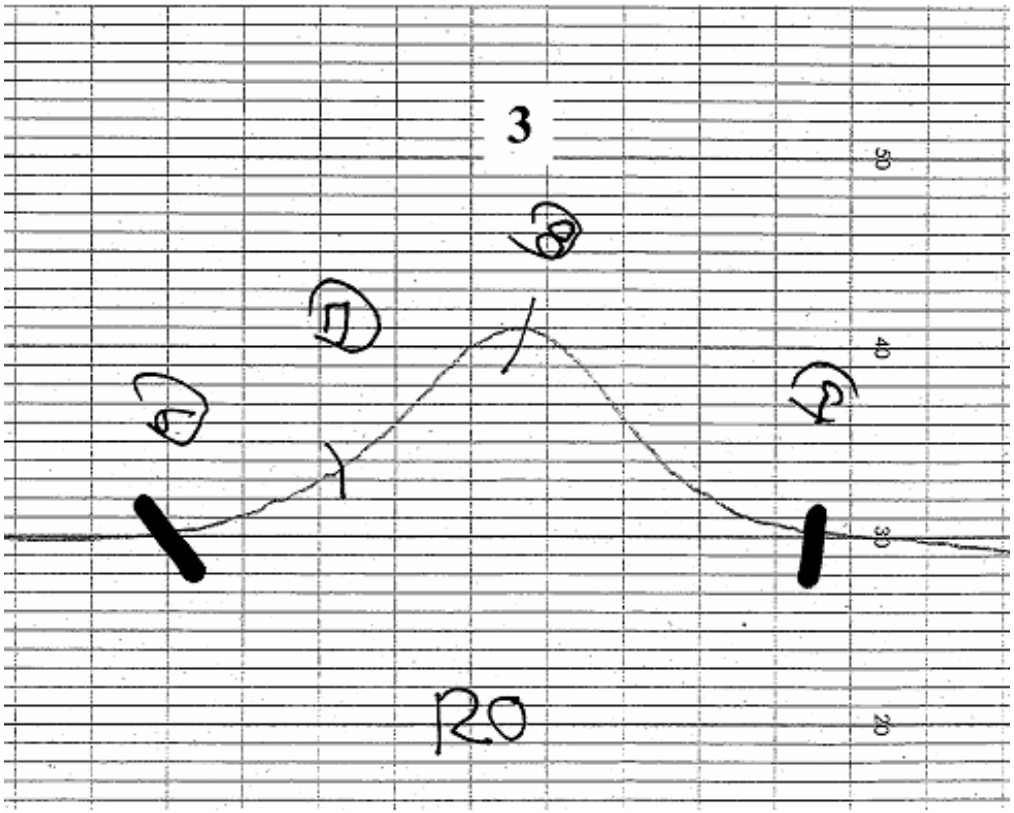


Figure S66

Moble phase CH_3CN / H_2O = 30 / 70, 4ml/min

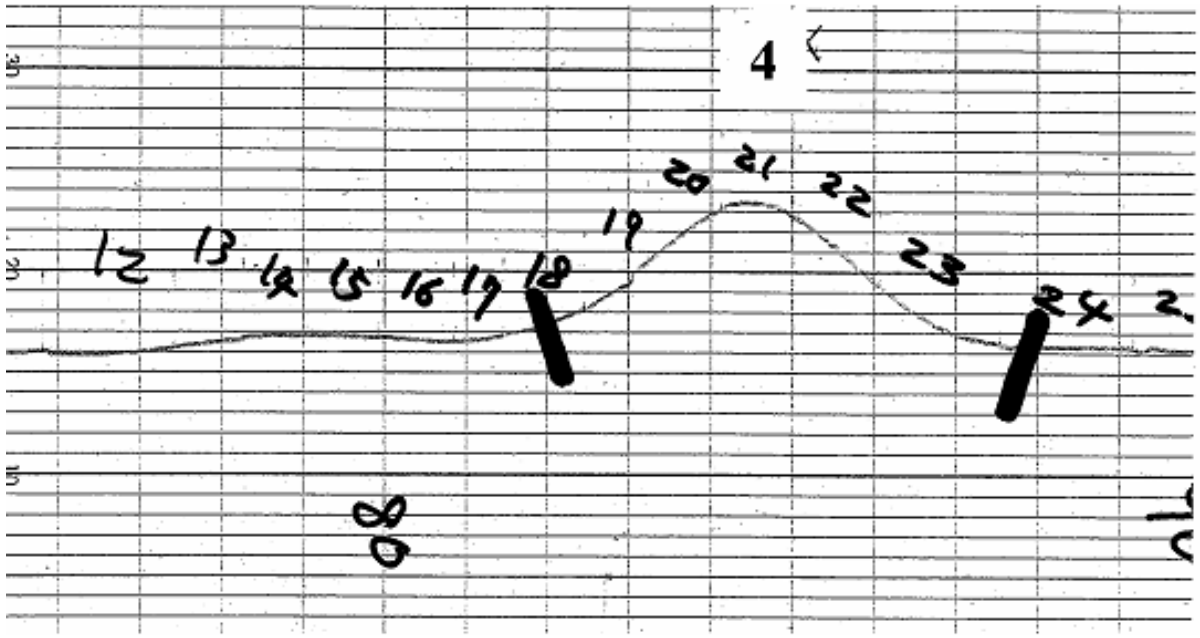
cephalimysin G (**3**)



Moble phase CH_3CN / H_2O = 40 / 60, 4ml/min

Figure S67

cephalimysin H (**4**)



Moble phase CH_3CN / H_2O = 43 / 57, 4ml/min

Figure S68

cephalimysin I (**5**)

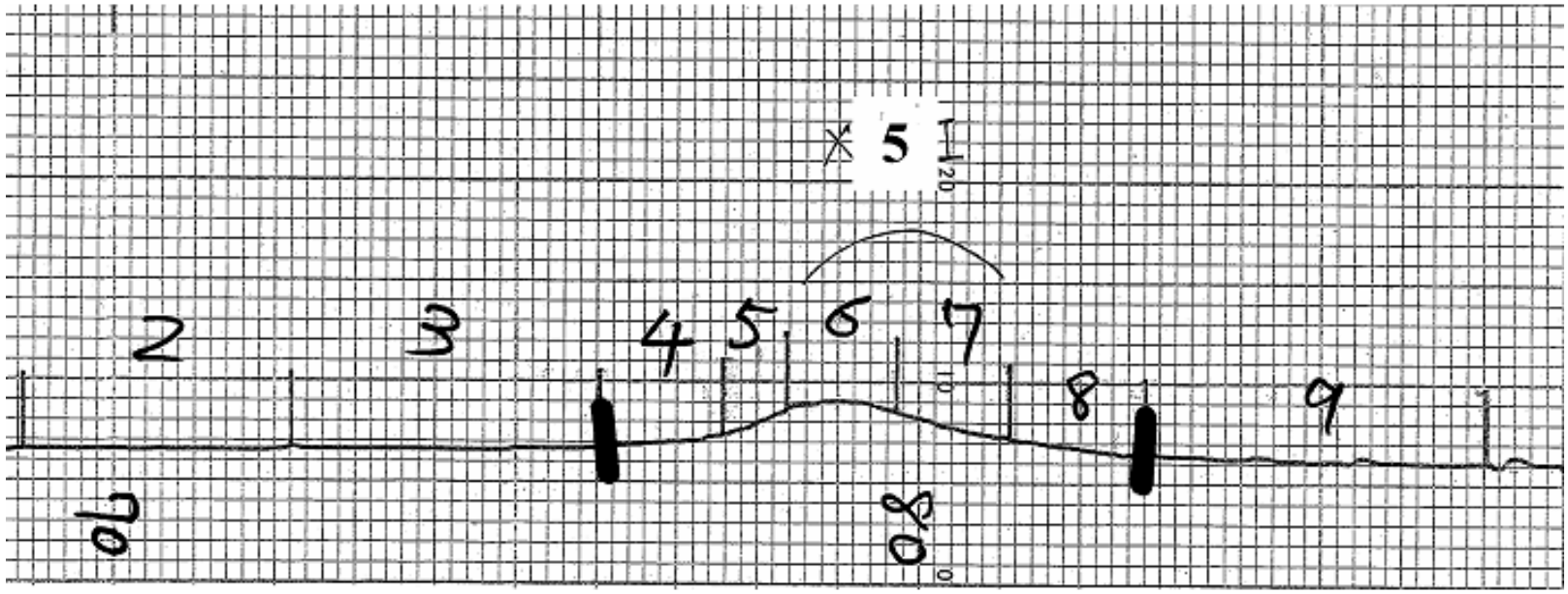
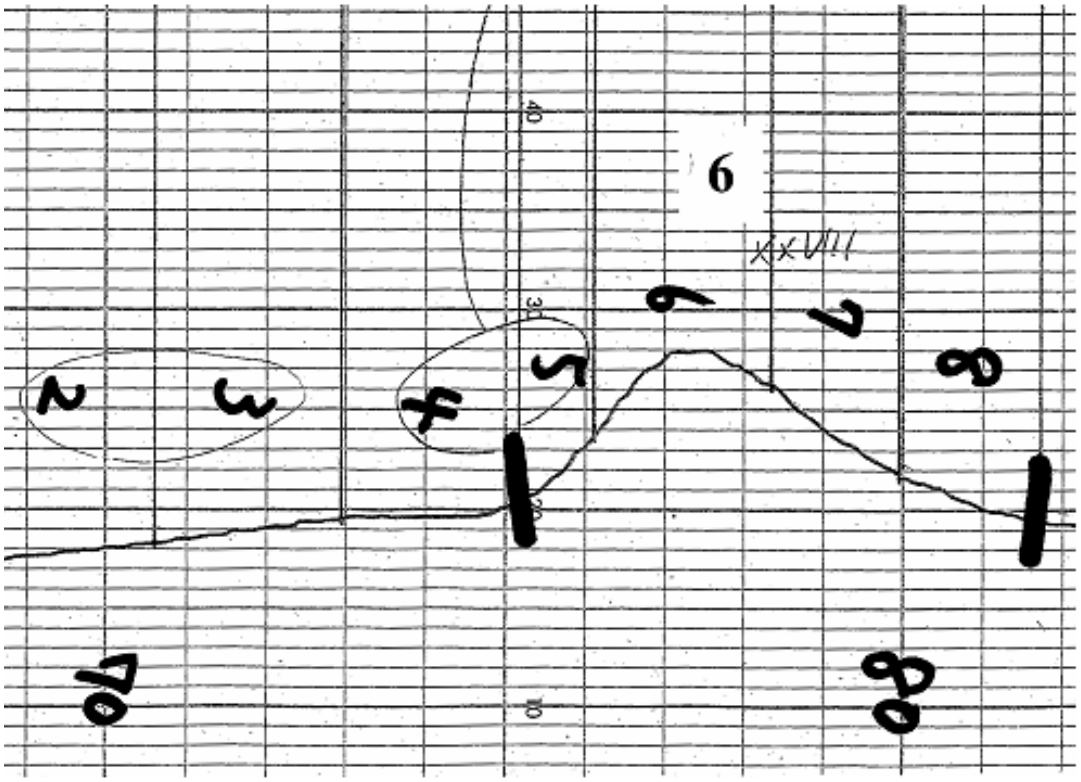


Figure S69

Moble phase CH_3CN / H_2O = 40 / 60, 4ml/min

cephalimysin J(**6**)



Moble phase CH_3CN / H_2O = 38 / 62, 4ml/min

Figure S70

cephalimysin K (7)

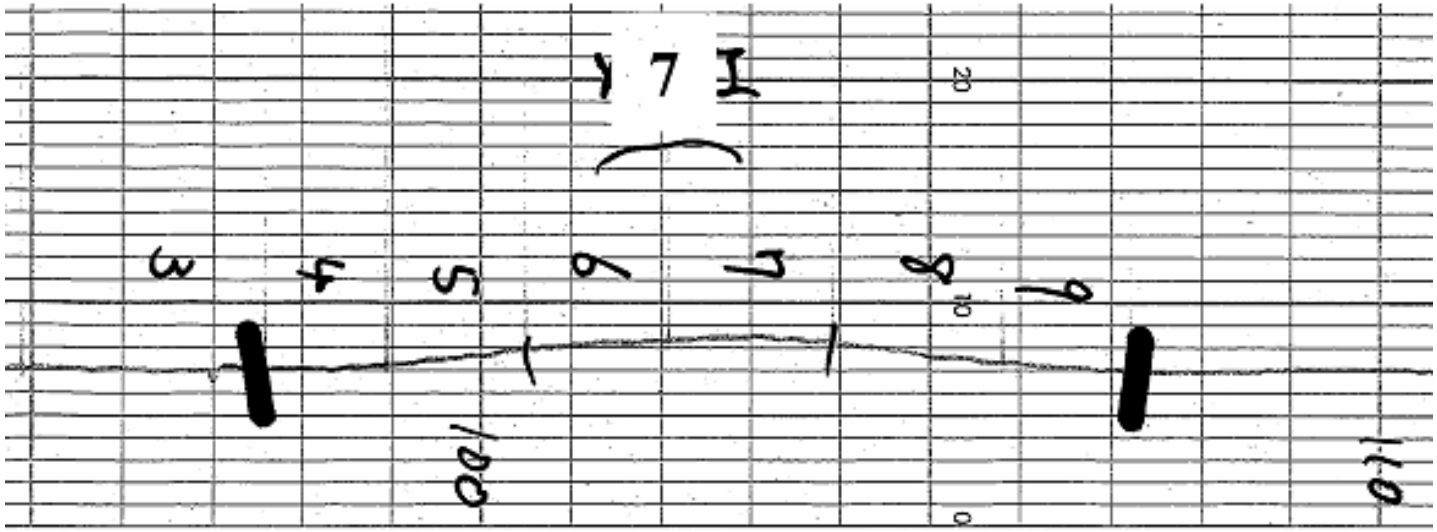
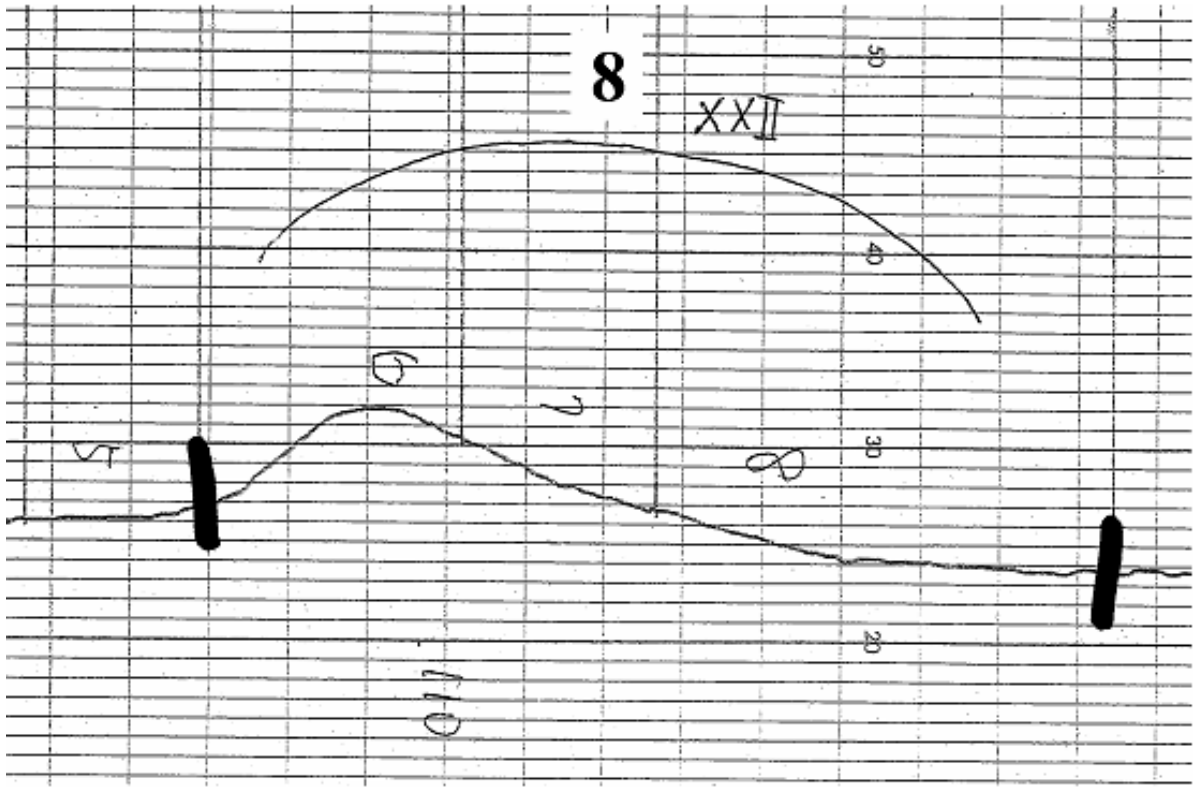


Figure S71

Moble phase CH_3CN / H_2O = 43 / 57, 4ml/min

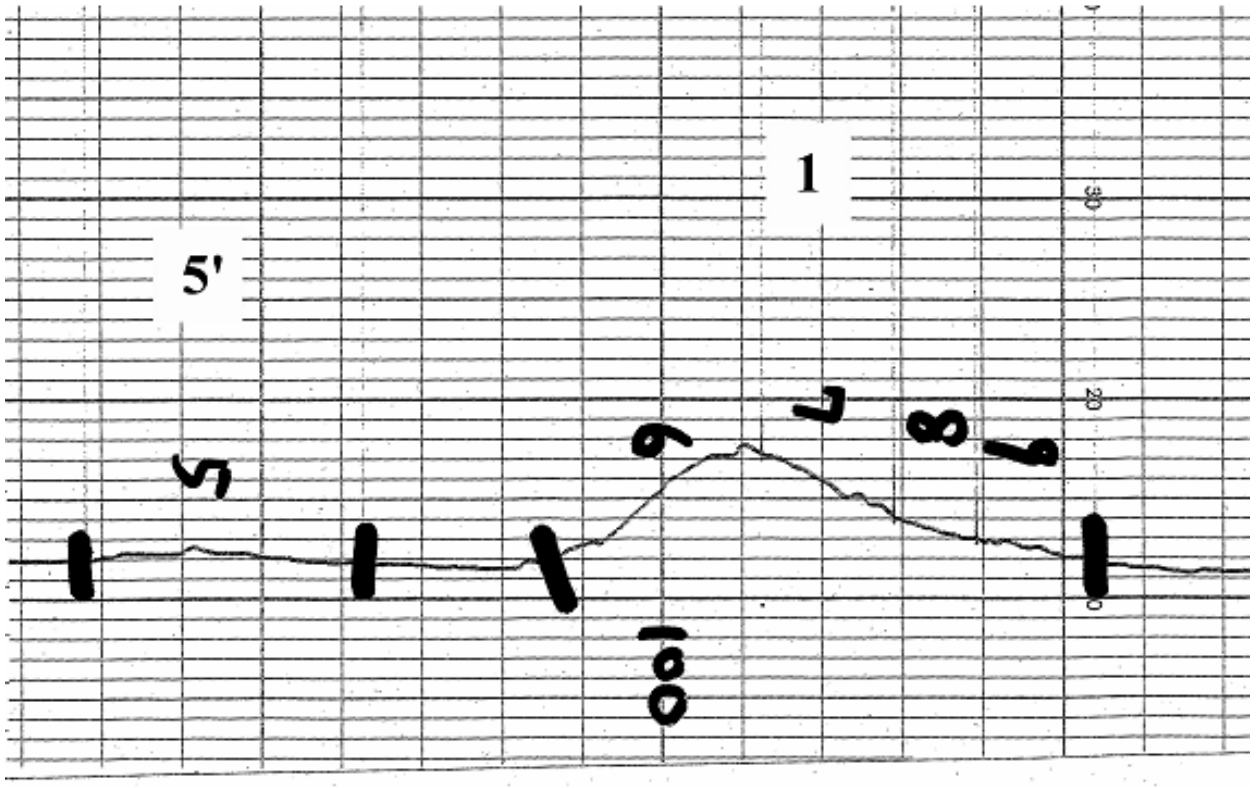
cephalimysin L (**8**)



Moble phase CH_3CN / H_2O = 40 / 60, 4ml/min

Figure S72

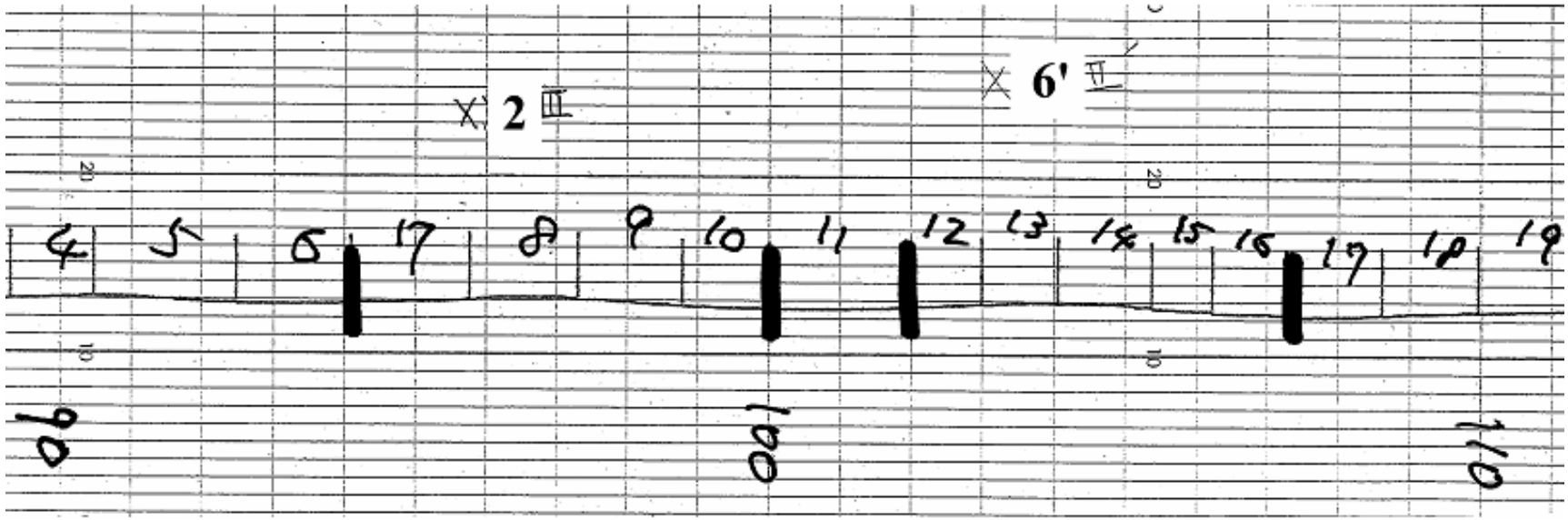
Acid treatment of **1**



Moble phase CH_3CN / H_2O = 38 / 62, 4ml/min

Figure S73

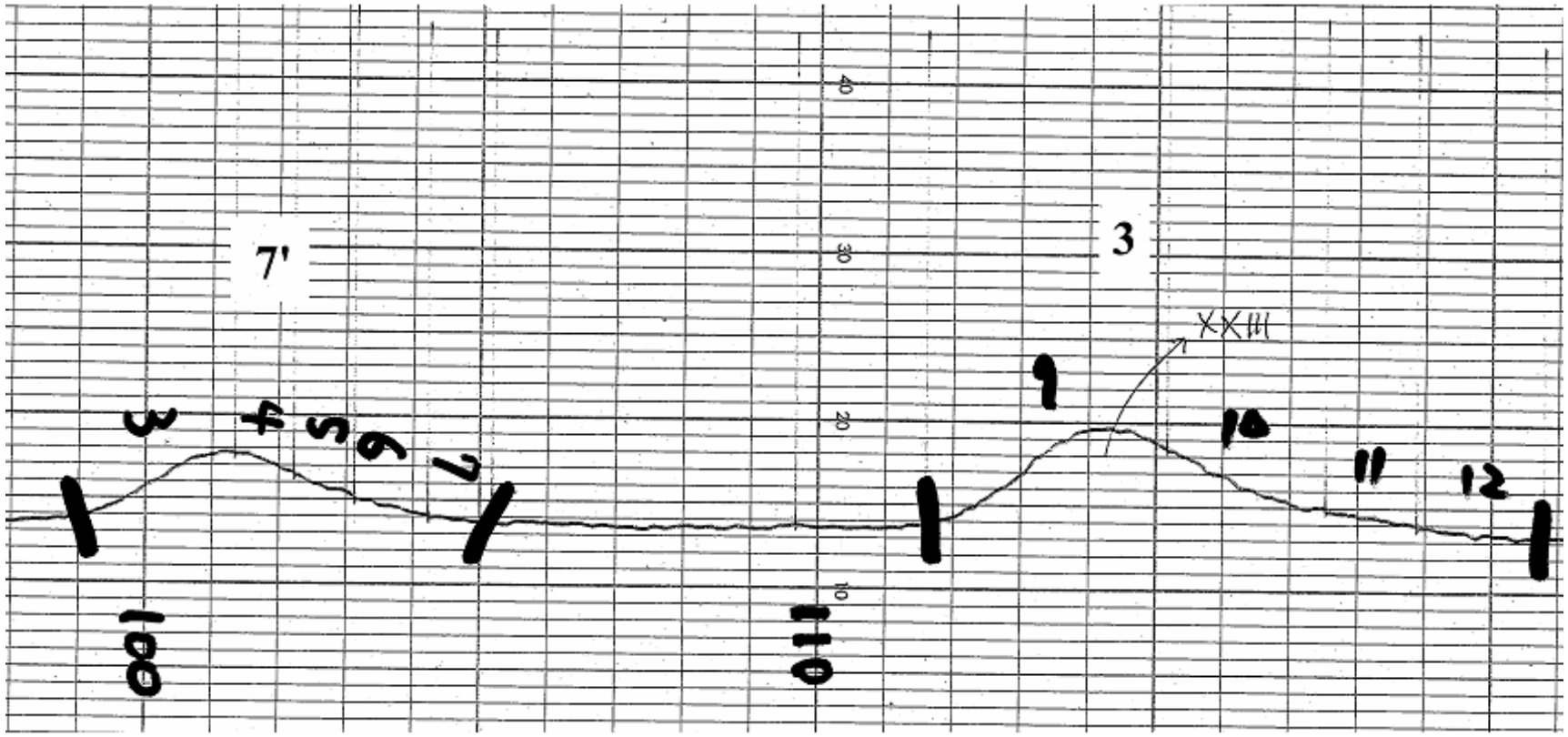
Acid treatment of **2**



Moble phase CH_3CN / H_2O = 35 / 65, 4ml/min

Figure S74

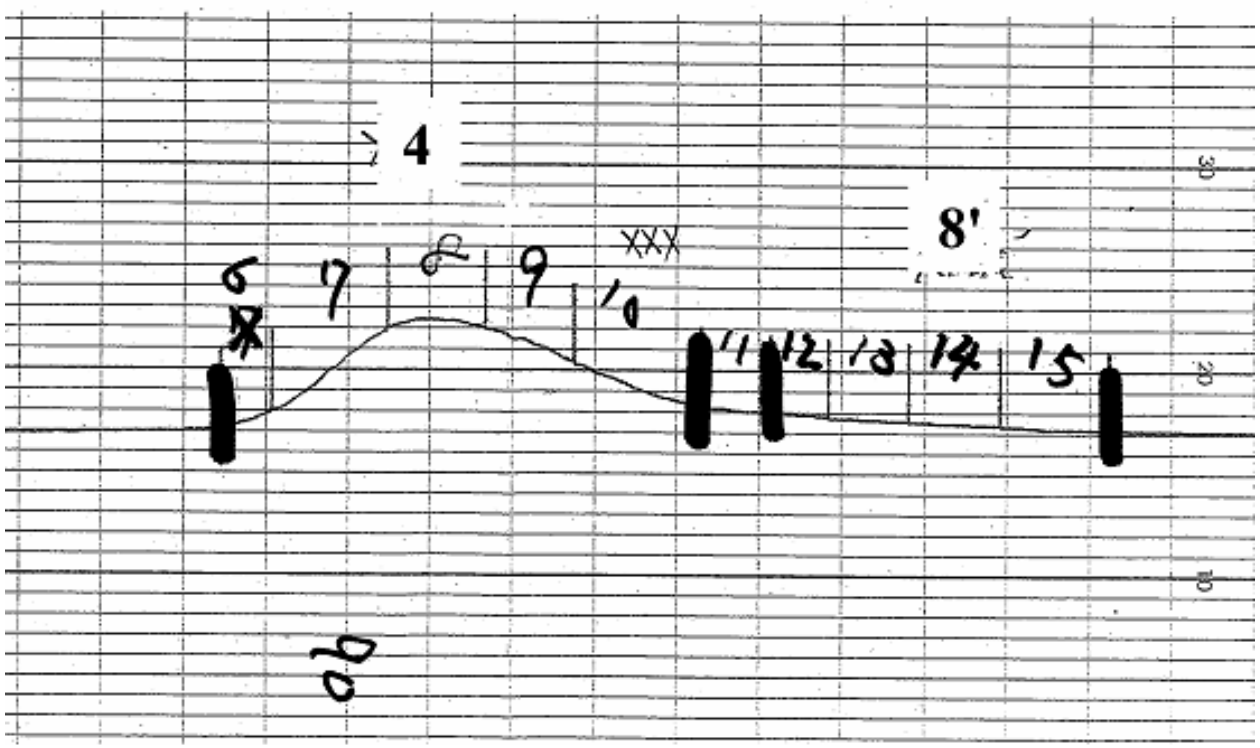
Acid treatment of **3**



Moble phase CH_3CN / H_2O = 43 / 57, 4ml/min

Figure S75

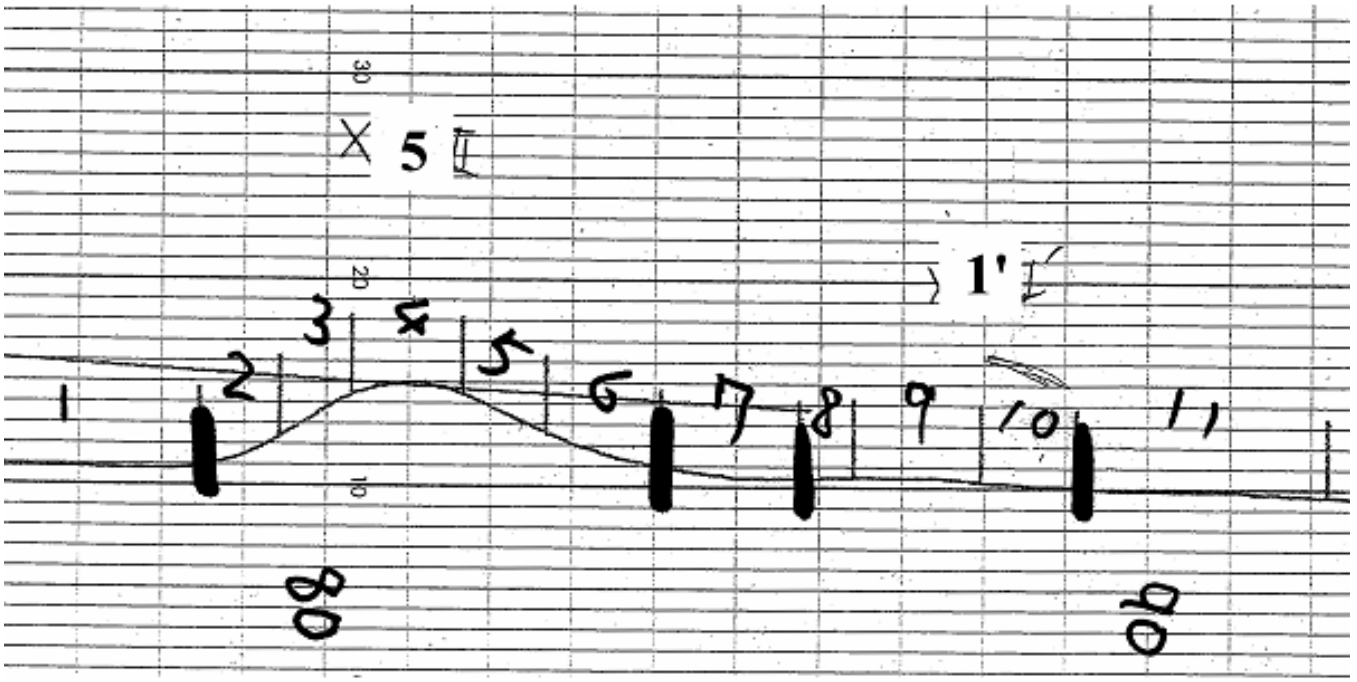
Acid treatment of **4**



Moble phase CH_3CN / H_2O = 43 / 57, 4ml/min

Figure S76

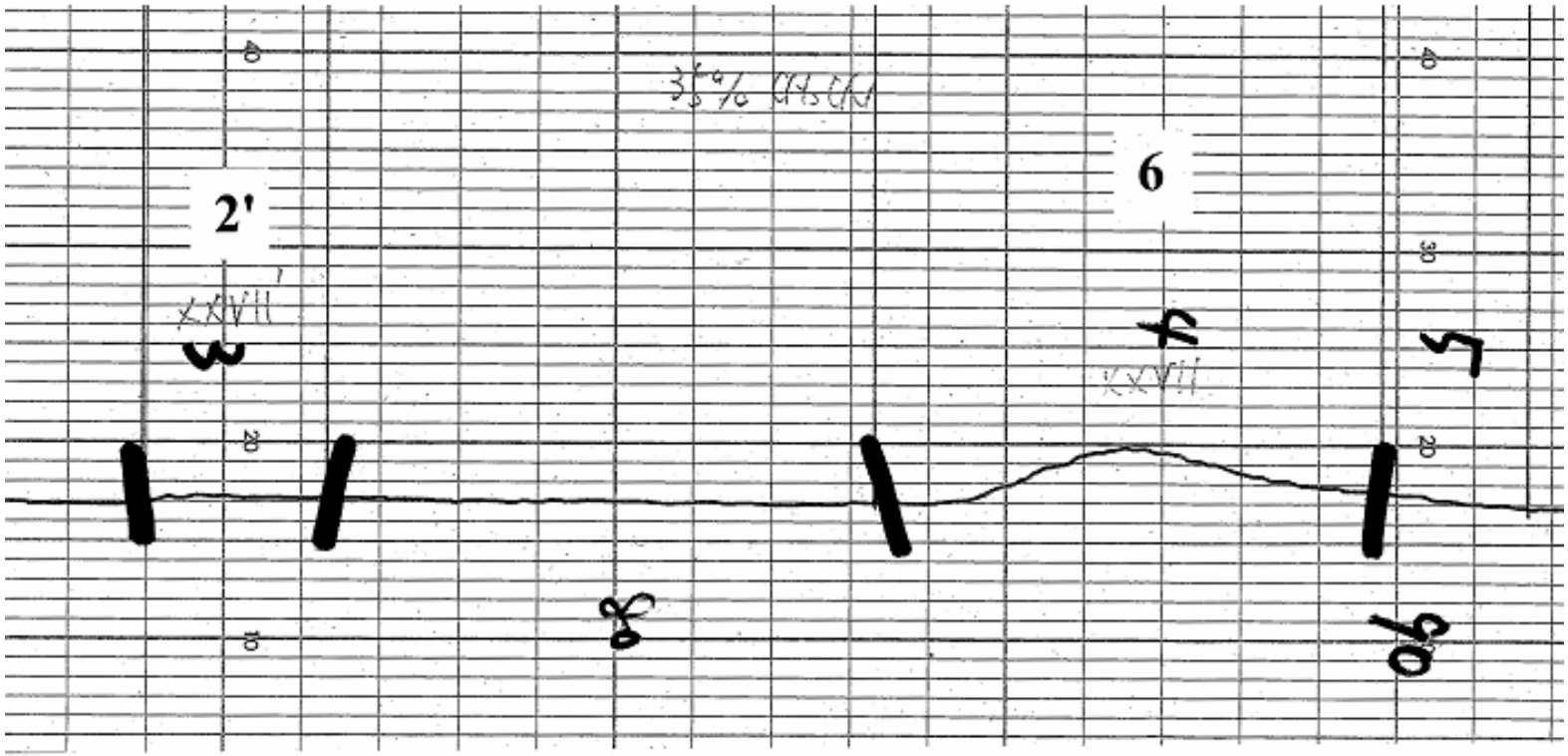
Acid treatment of **5**



Moble phase CH_3CN / H_2O = 40 / 60, 4ml/min

Figure S77

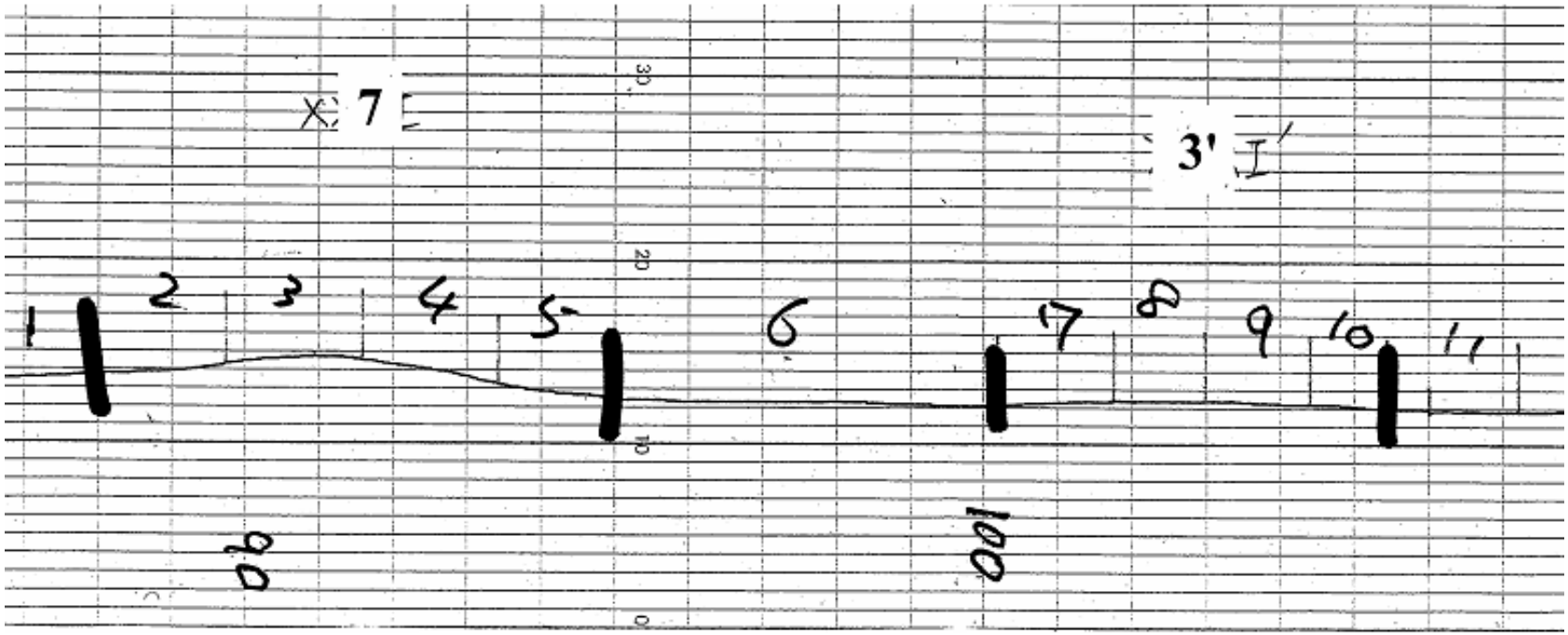
Acid treatment of **6**



Moble phase CH_3CN / H_2O = 36 / 64, 4ml/min

Figure S78

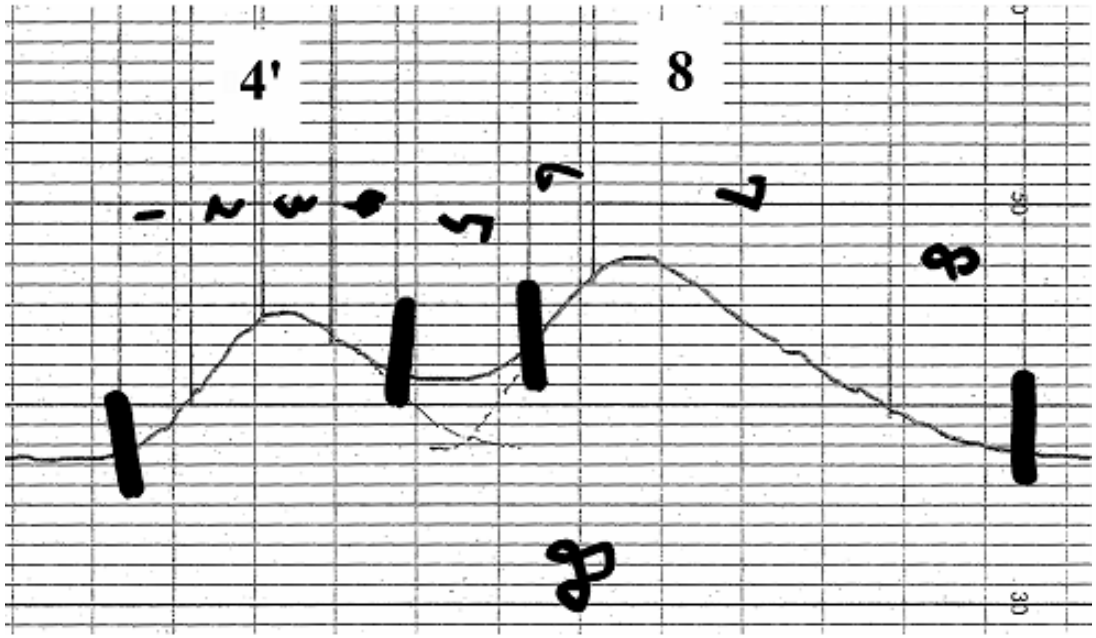
Acid treatment of **7**



Moble phase CH_3CN / H_2O = 43 / 57, 4ml/min

Figure S79

Acid treatment of **8**



Moble phase CH_3CN / H_2O = 45 / 55, 4ml/min

Figure S80

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