Enantiospecific Total Synthesis and Absolute Configuration Assignment of Chabrolobenzoquinone H

Supporting Information

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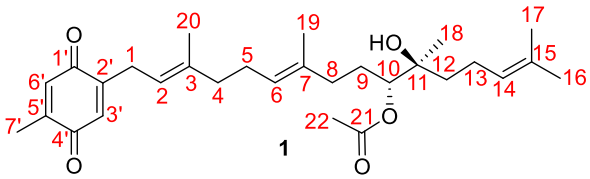
Comparison of natural and synthetic chabrolobenzoquinone H

Table S1. Comparison of NMR data for natural and synthetic chabrolobenzoquinone H (1)



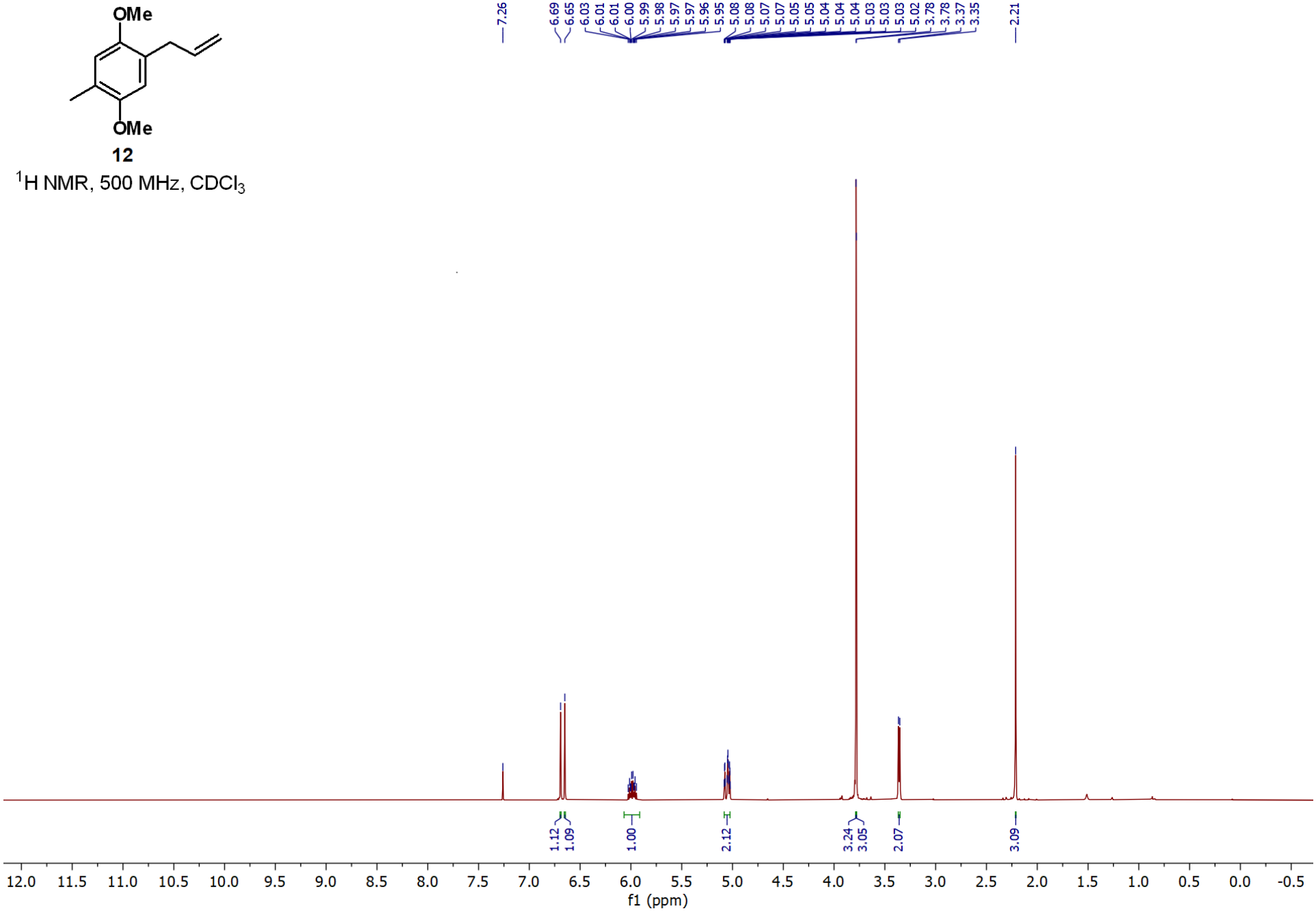
	NATURAL SAMPLE[a]		SYNTHETIC SAMPLE[b]	
Frequency	300 MHz	75 MHz	500 MHz	126 MHz
C/H	δ_H	δ_C	δ_H	δ_C
1′	-	187.9	-	187.9
2′	-	148.6	-	148.5
3′	6.50 s	132.4	6.50 t (1.8 Hz)	132.4
4′	-	188.4	-	188.4
5′	-	145.7	-	145.7
6′	6.60 q (1.5 Hz)	133.6	6.60 q (1.6	133.6
7′	2.04 d (1.5 Hz)	15.5	2.04 d (1.6 Hz)	15.5
1	3.11 d (7.2 Hz)	27.2	3.11 d (6.3 Hz)	27.1
2	5.13 t (7.2 Hz)	118.0	5.14 td (7.3, 1.3 Hz)	118.0
3	-	139.8	-	139.8
4	2.09 m	39.6	2.08 m	39.5
5	2.12 m	26.4	2.12 m	26.4
6	5.11 m	124.3	5.11 m	124.3
7	-	134.6	-	134.6
8	1.96 m	36.2	1.96 m	36.2
9	1.71 m	27.7	1.75-1.66 m	27.6
10	4.85 dd (9.6, 3.0 Hz)	79.1	4.85 dd (10.2, 2.6 Hz)	79.1
11	-	74.2	-	74.2
12	1.43 m, 1.54 m	37.8	1.44 m, 1.55 m	37.8
13	2.05 m	22.1	2.05 m	22.1
14	5.11 m	124.5	5.11 m	124.5
15	-	132.1	-	132.1
16	1.68 s	25.8	1.69 s	25.7
17	1.62 s	17.7	1.62 s	17.7
18	1.17 s	23.6	1.17 s	23.6
19	1.60 s	16.2	1.60 s	16.2
20	1.62 s	16.1	1.62 s	16.1
21	-	171.1	-	171.1
22	2.11 s	21.1	2.11 s	21.1
OH	Not observed	-	Not observed	-

[a]Taken from the isolation report.[1] *Note:* There is no information on the reference shift used in that report. [b]For synthetic **1**, the reference shift of $CDCl_3$ was set to 7.27 for ^1H and 77.00 for ^{13}C.

[1]Su, J-H.; Ahmed, A. F.; Sung, P-J.; Wu, Y-C.; Sheu, J-H. Meroditerpenoids from a Formosan Soft Coral *Nephthea Chabrolii*. *J. Nat. Prod.* **2005**, *68,* 1651–1655.

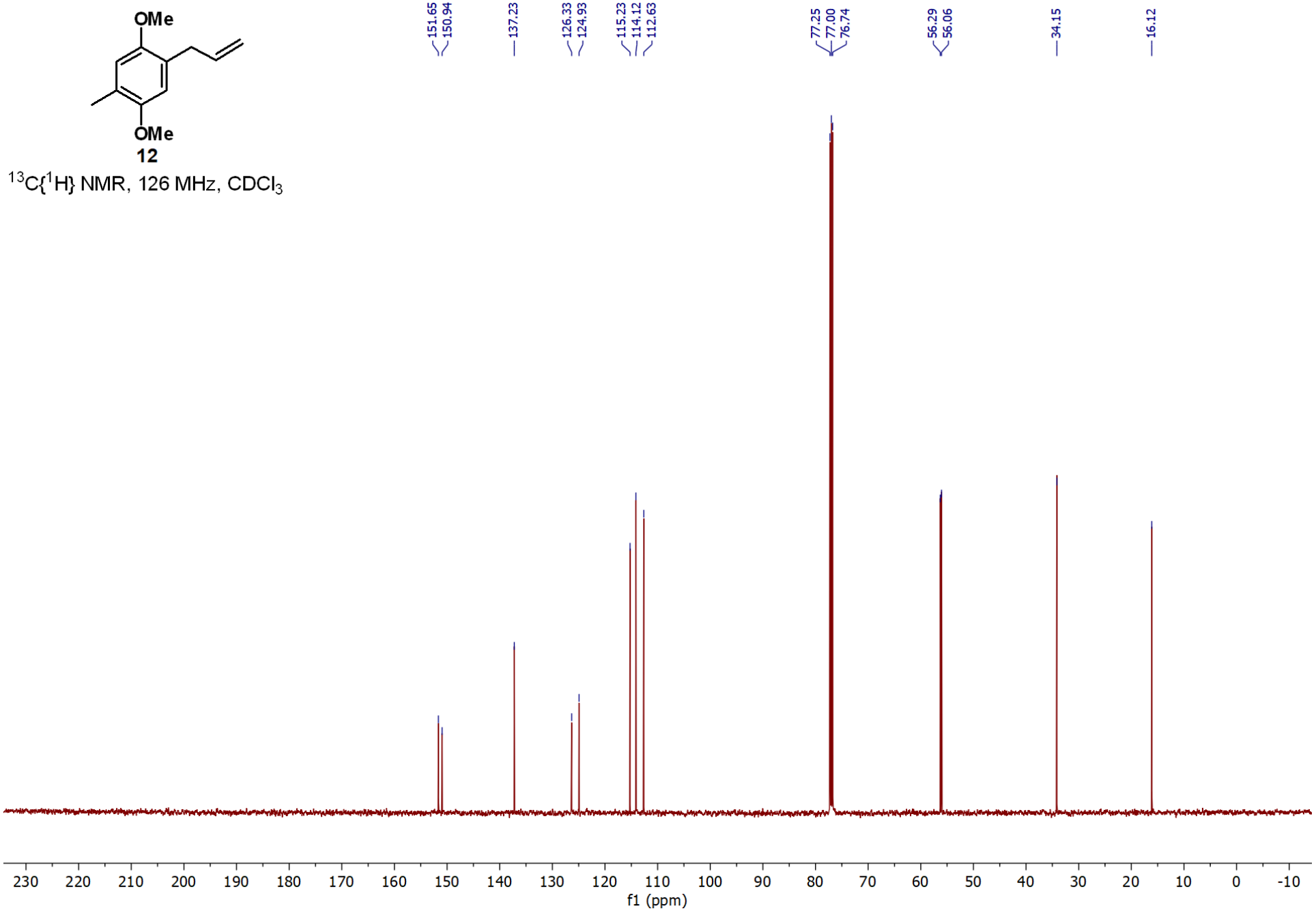
12

^{1}H NMR, 500 MHz, $CDCl_3$



12

$^{13}C\{^{1}H\}$ NMR, 126 MHz, $CDCl_3$



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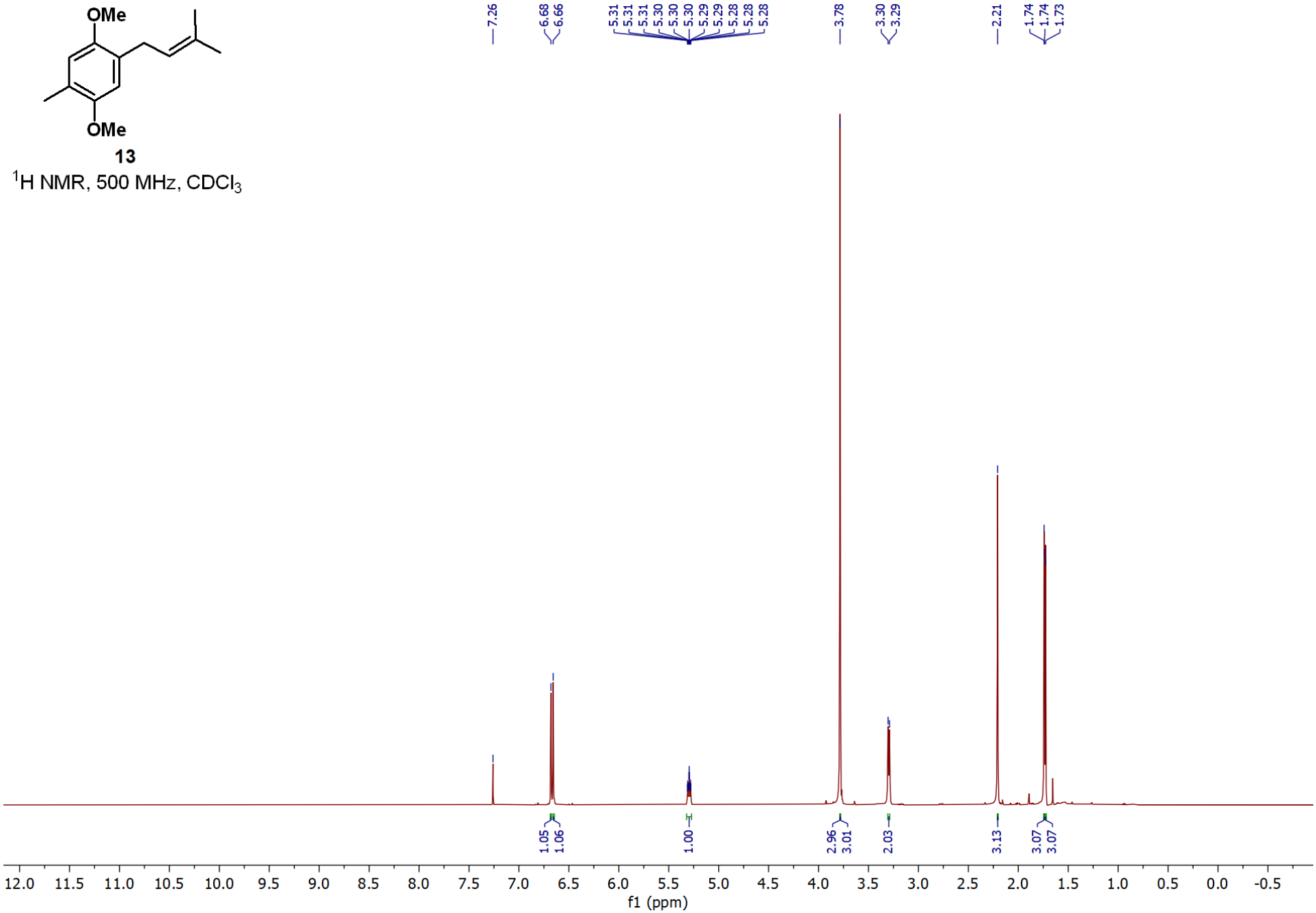
^{1}H NMR, 500 MHz, $CDCl_3$



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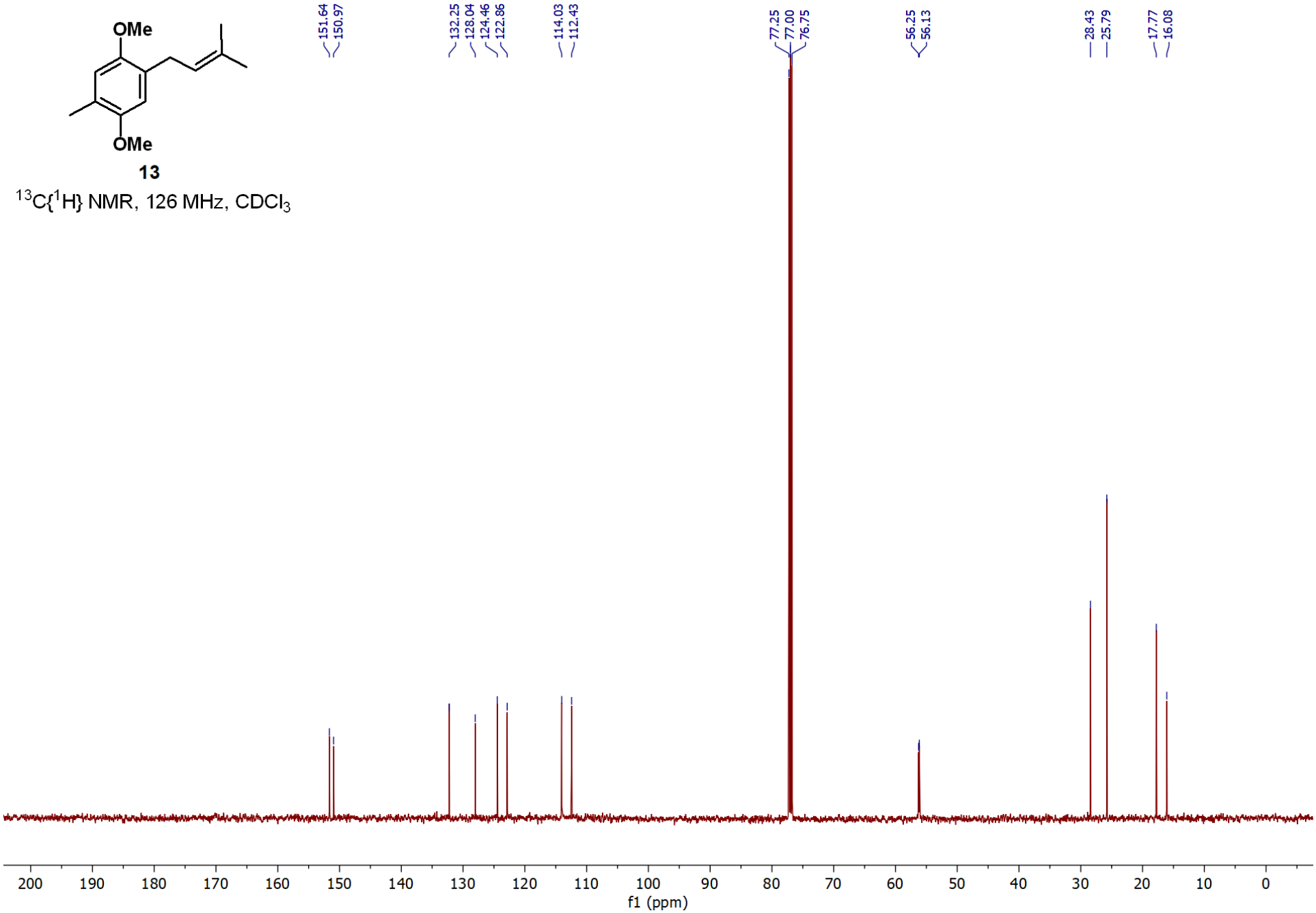
$^{13}C\{^{1}H\}$ NMR, 126 MHz, $CDCl_3$



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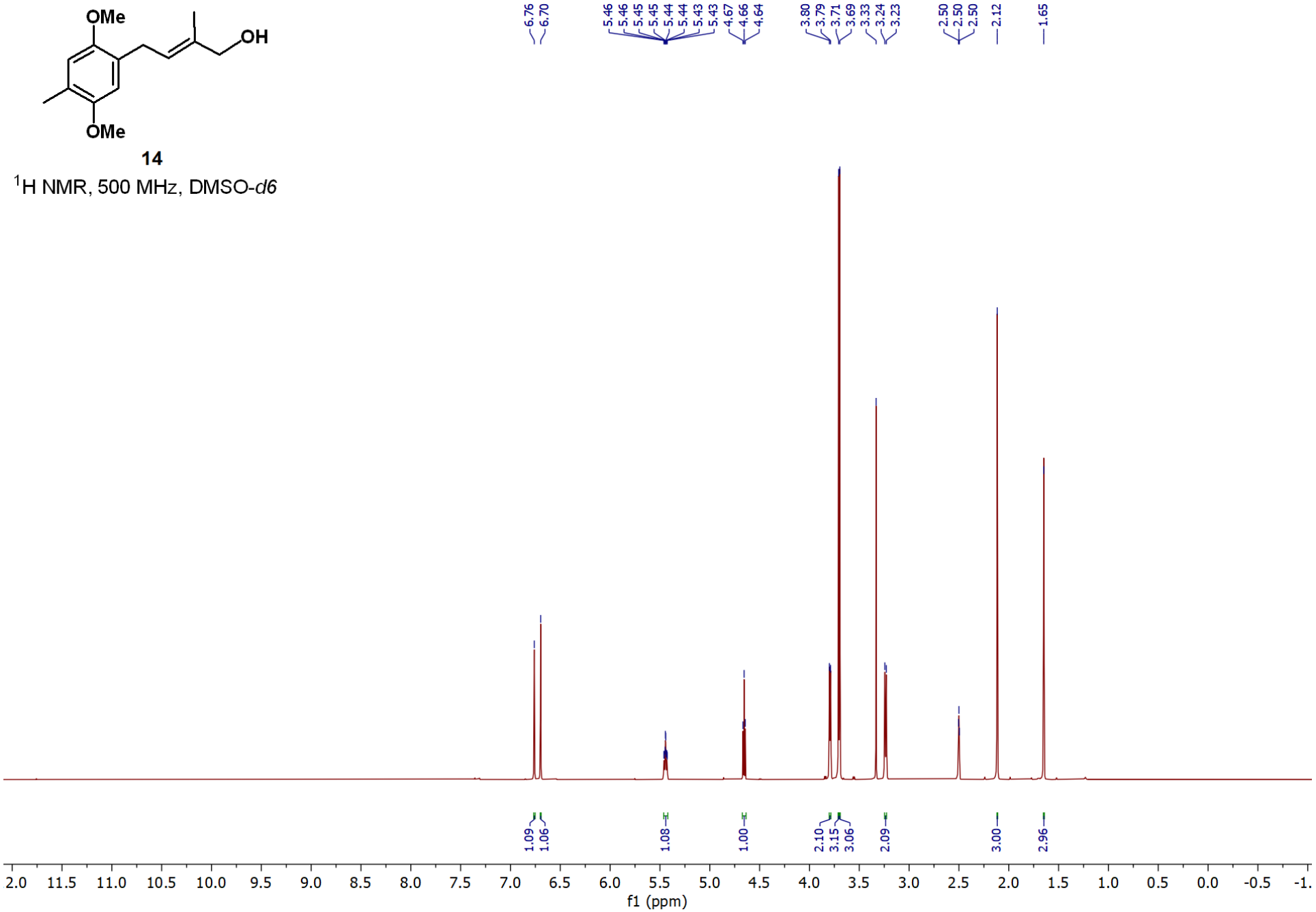
^{1}H NMR, 500 MHz, DMSO-*d6*



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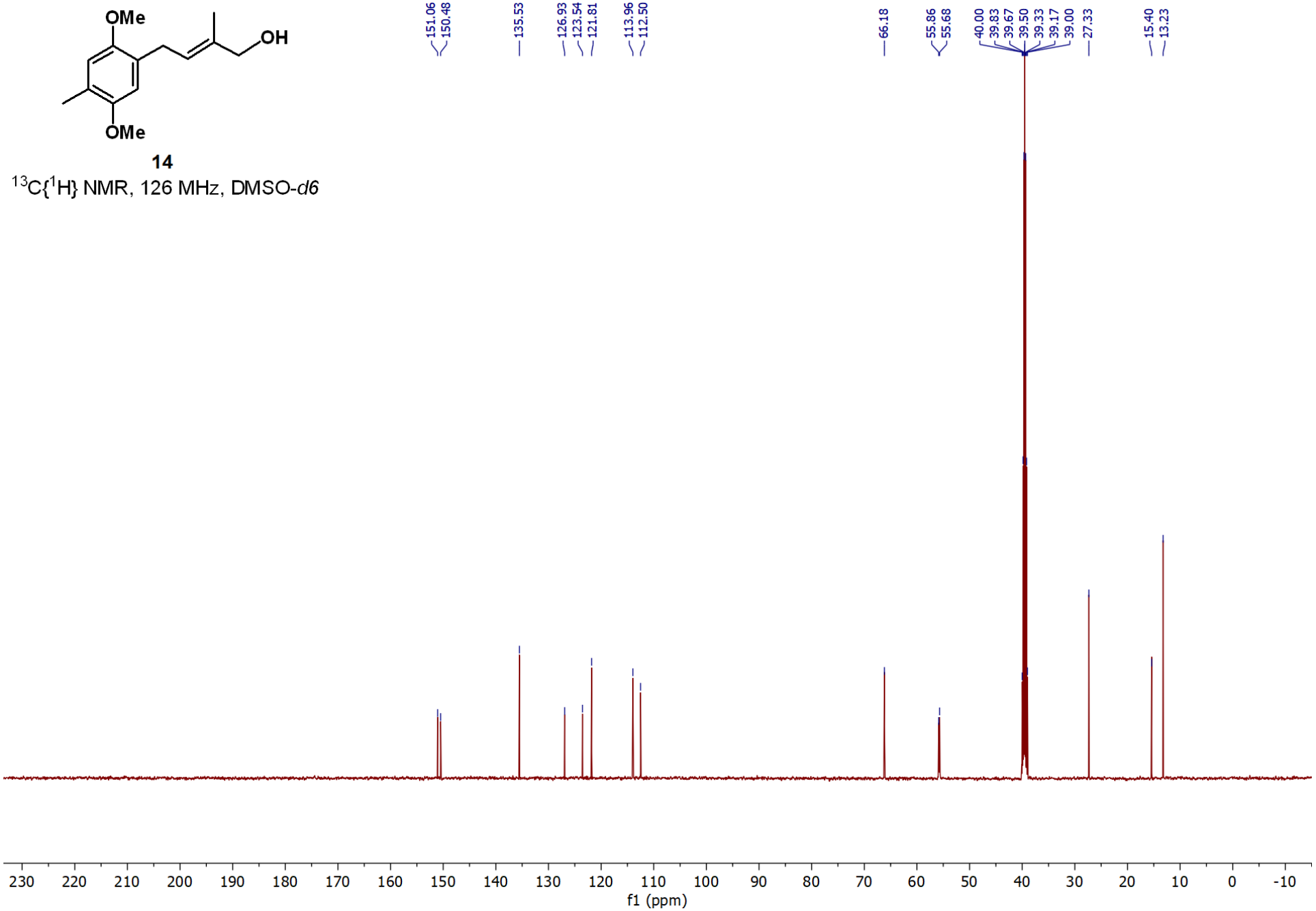
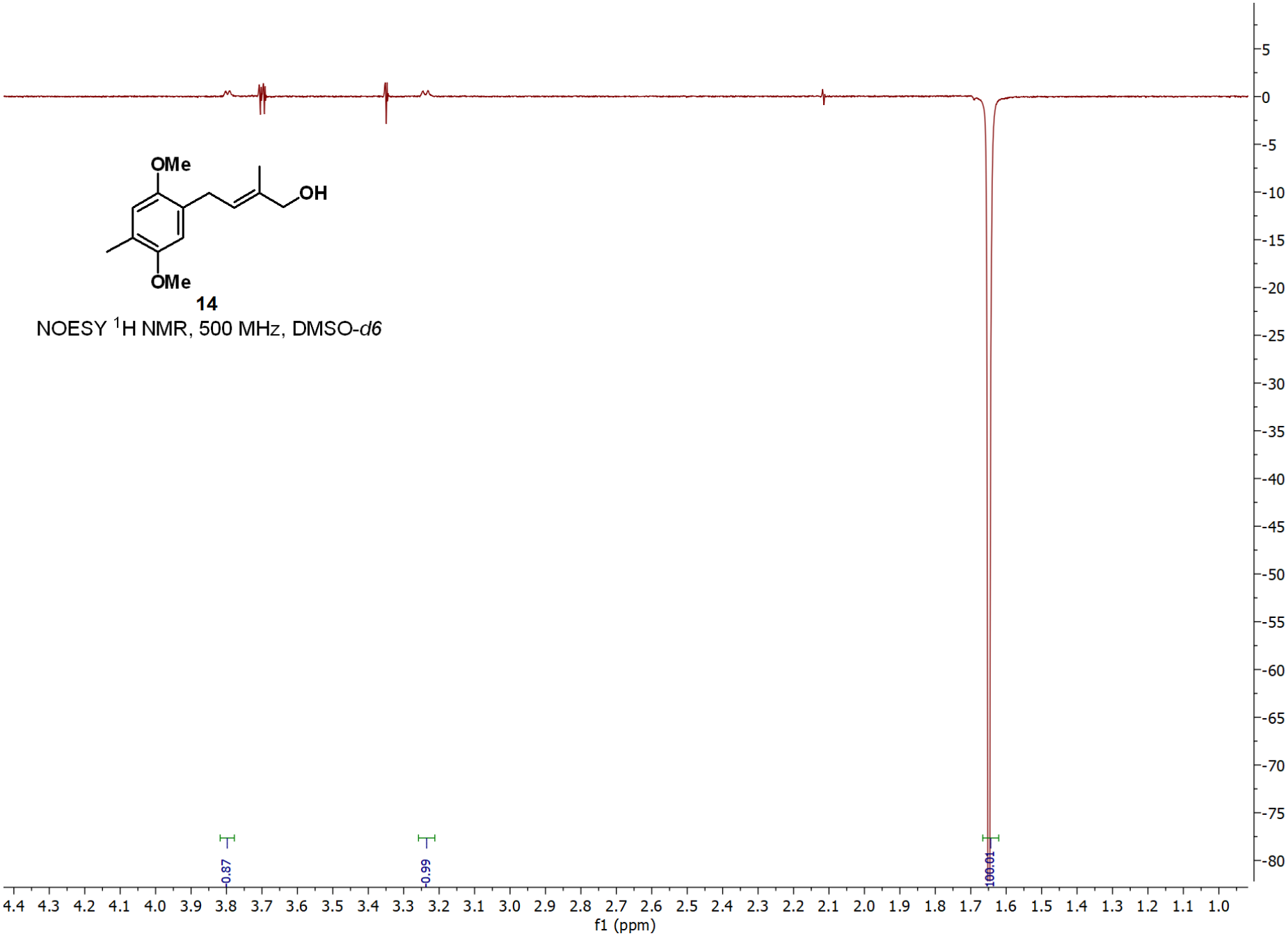
$^{13}C\{^{1}H\}$ NMR, 126 MHz, DMSO-*d6*

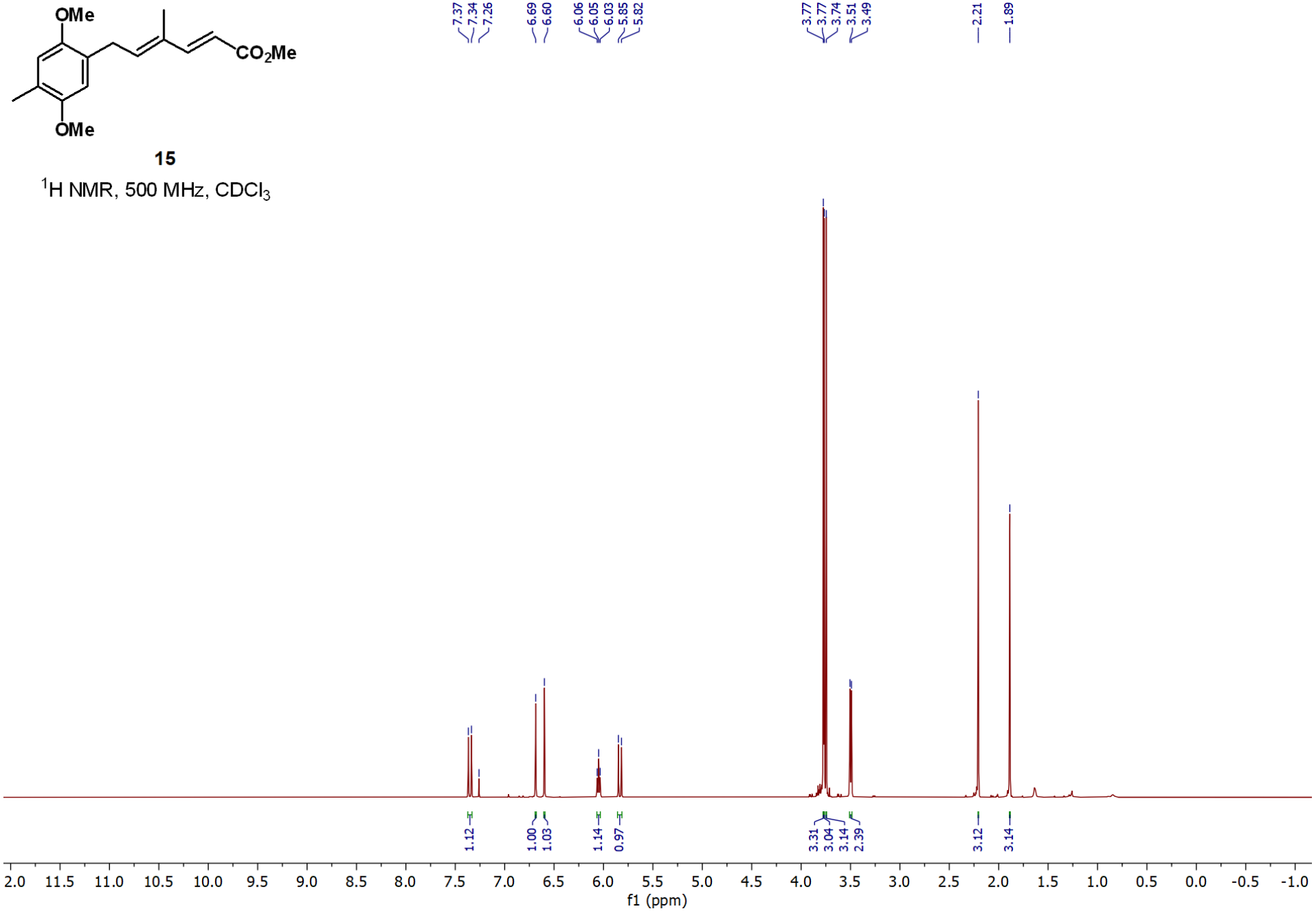




NOESY ^{1}H NMR, 500 MHz, DMSO-*d6*

15

^{1}H NMR, 500 MHz, $CDCl_3$



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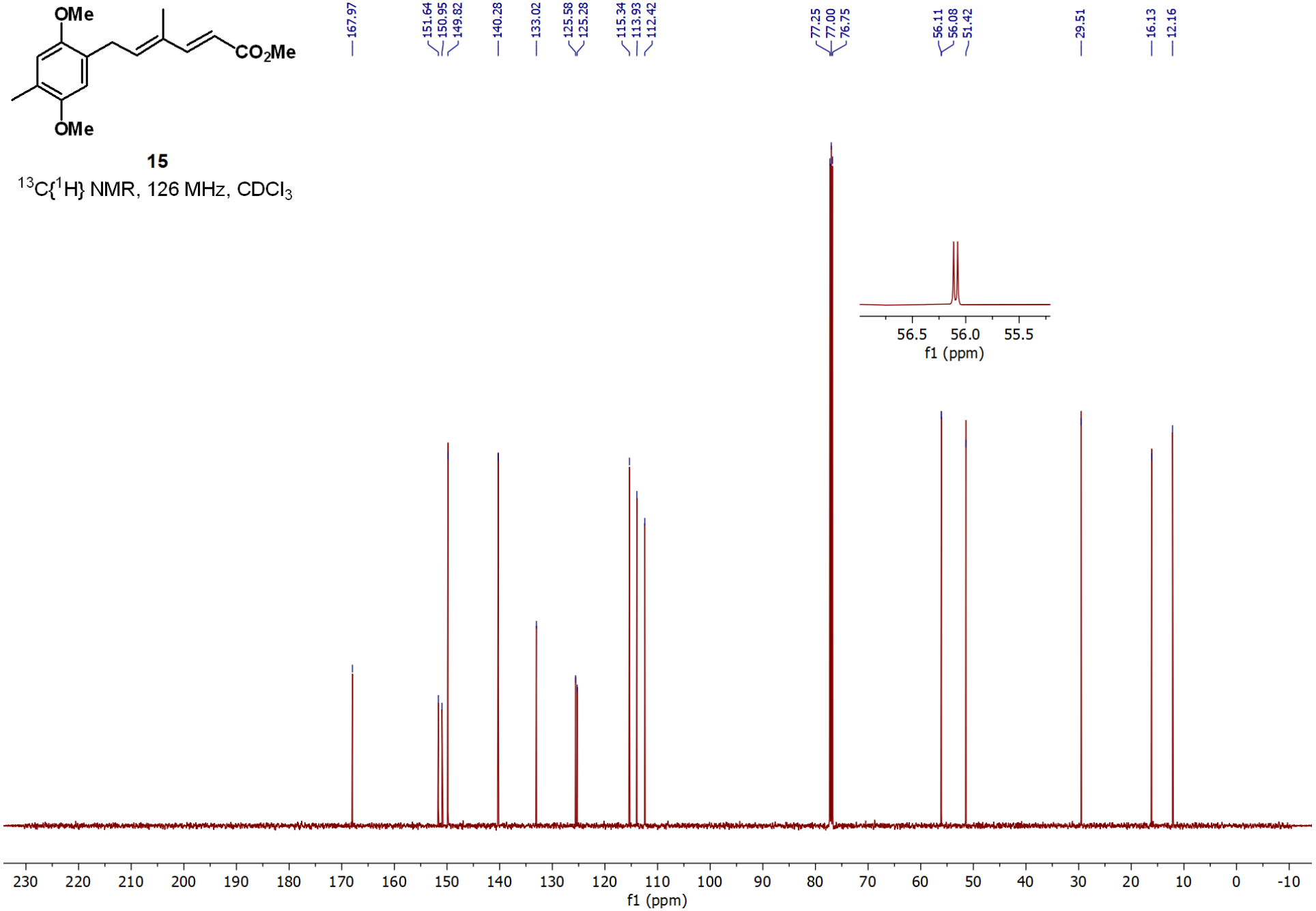
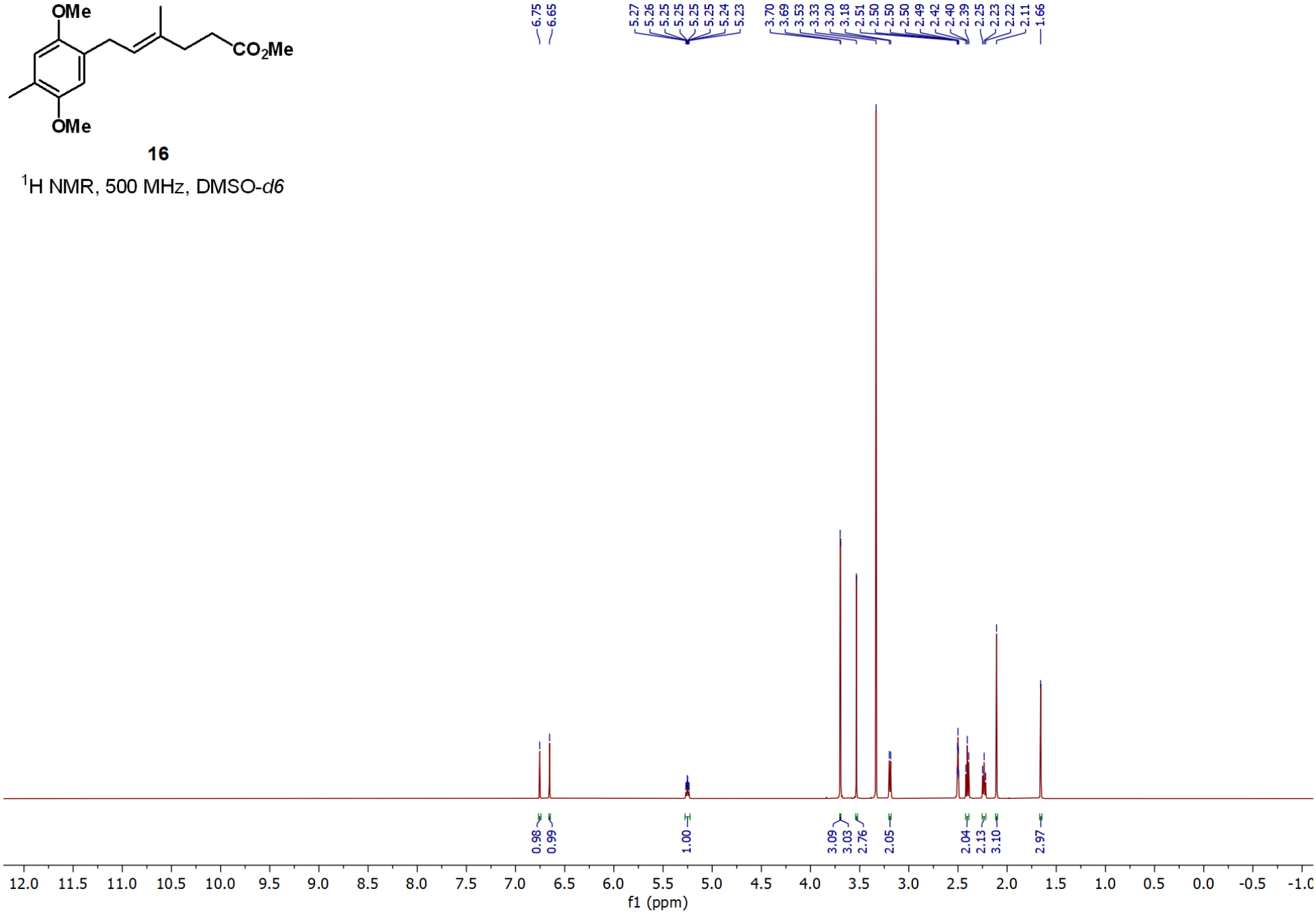
$^{13}C\{^{1}H\}$ NMR, 126 MHz, $CDCl_3$



16

^{1}H NMR, 500 MHz, DMSO-*d6*



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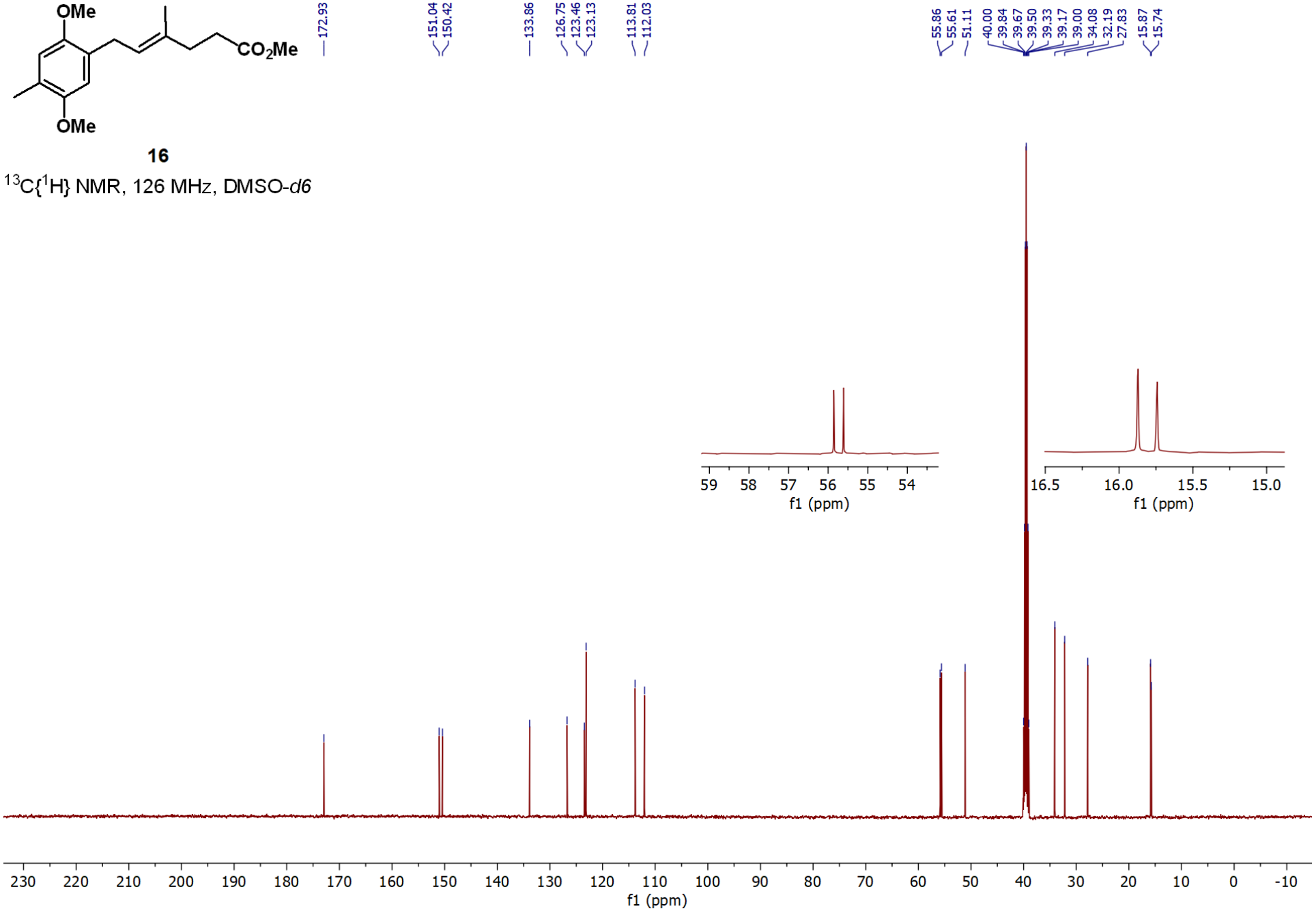
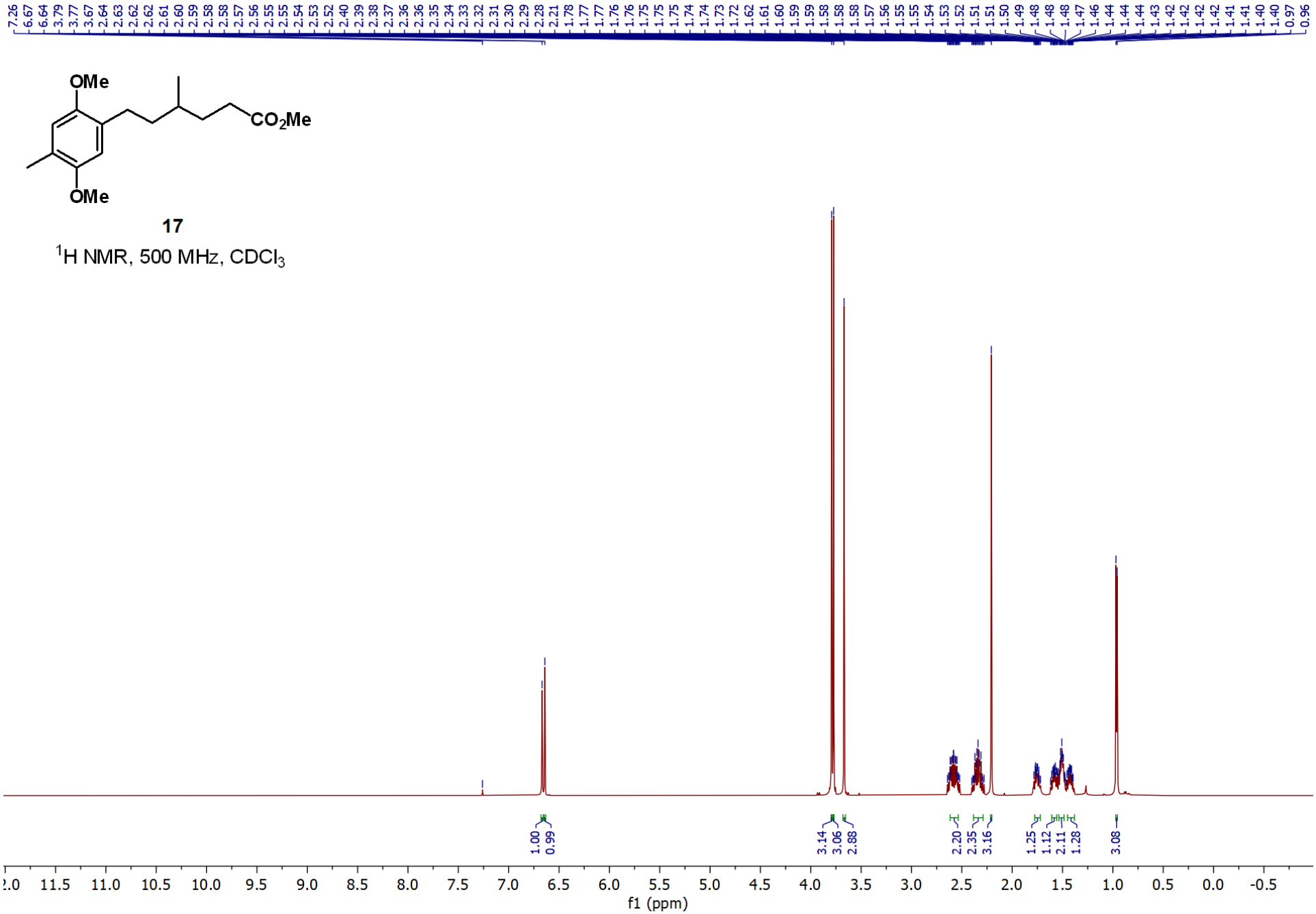
$^{13}C\{^{1}H\}$ NMR, 126 MHz, DMSO-*d6*



17

^{1}H NMR, 500 MHz, $CDCl_3$



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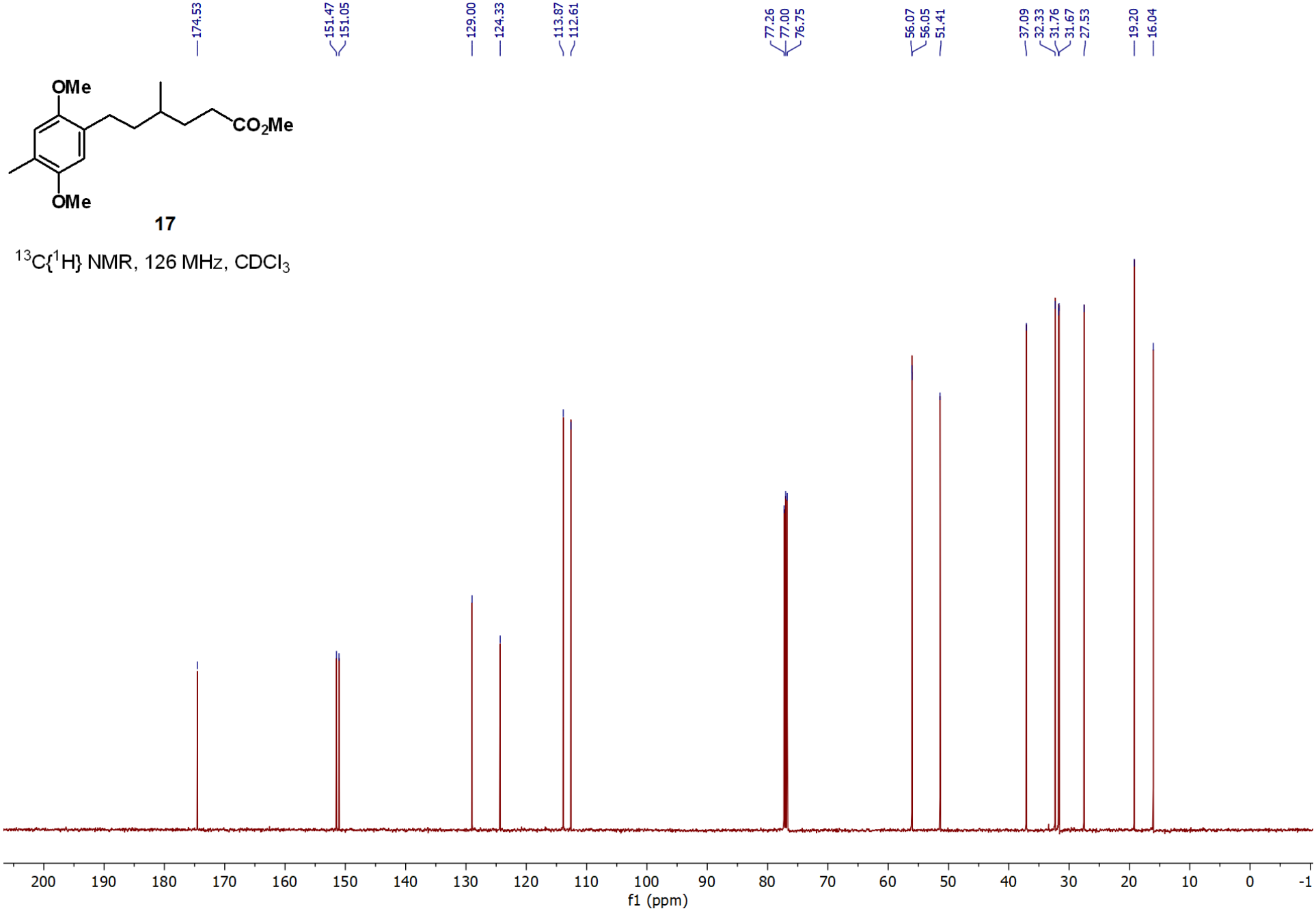
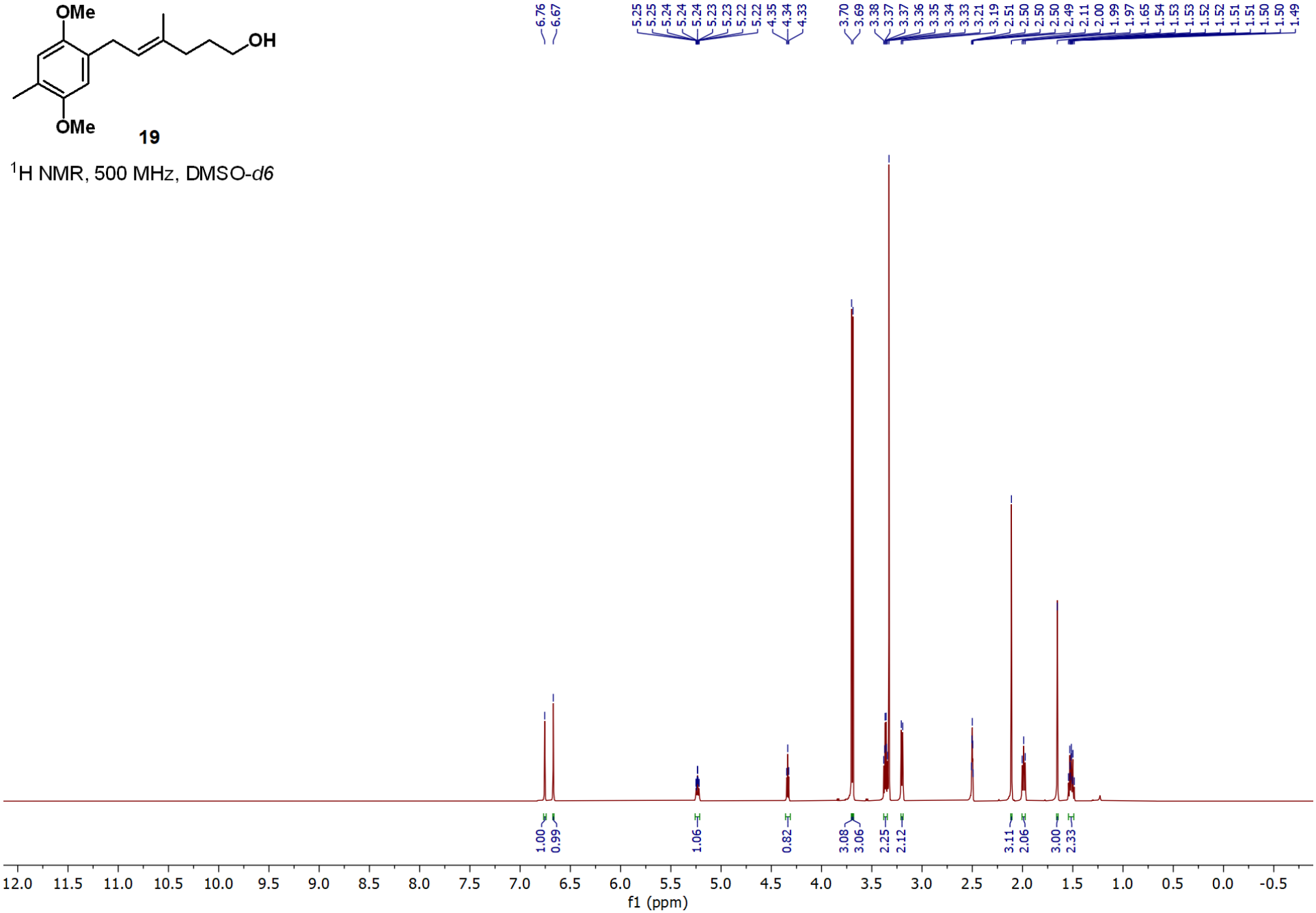
$^{13}C\{^{1}H\}$ NMR, 126 MHz, $CDCl_3$



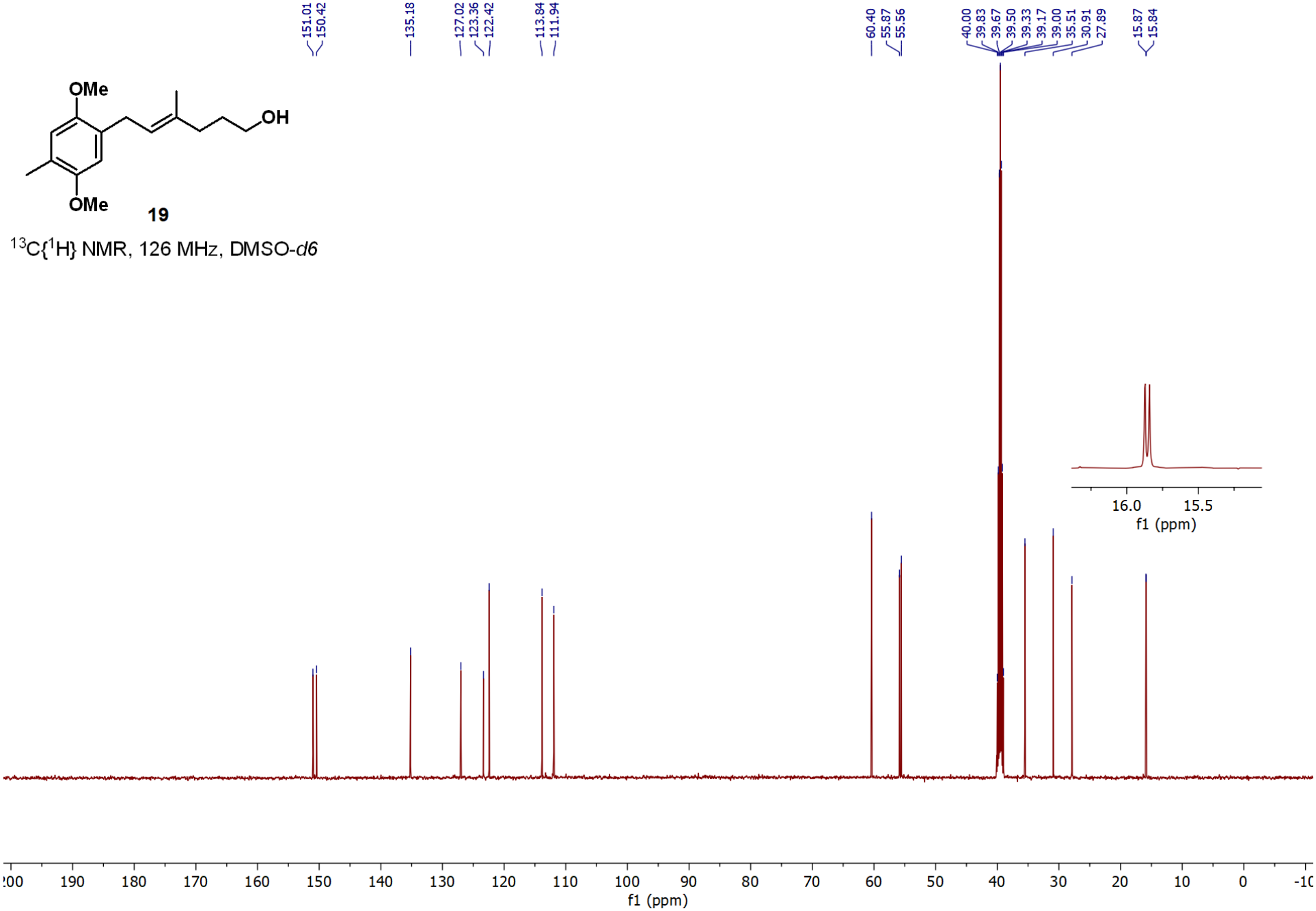
19

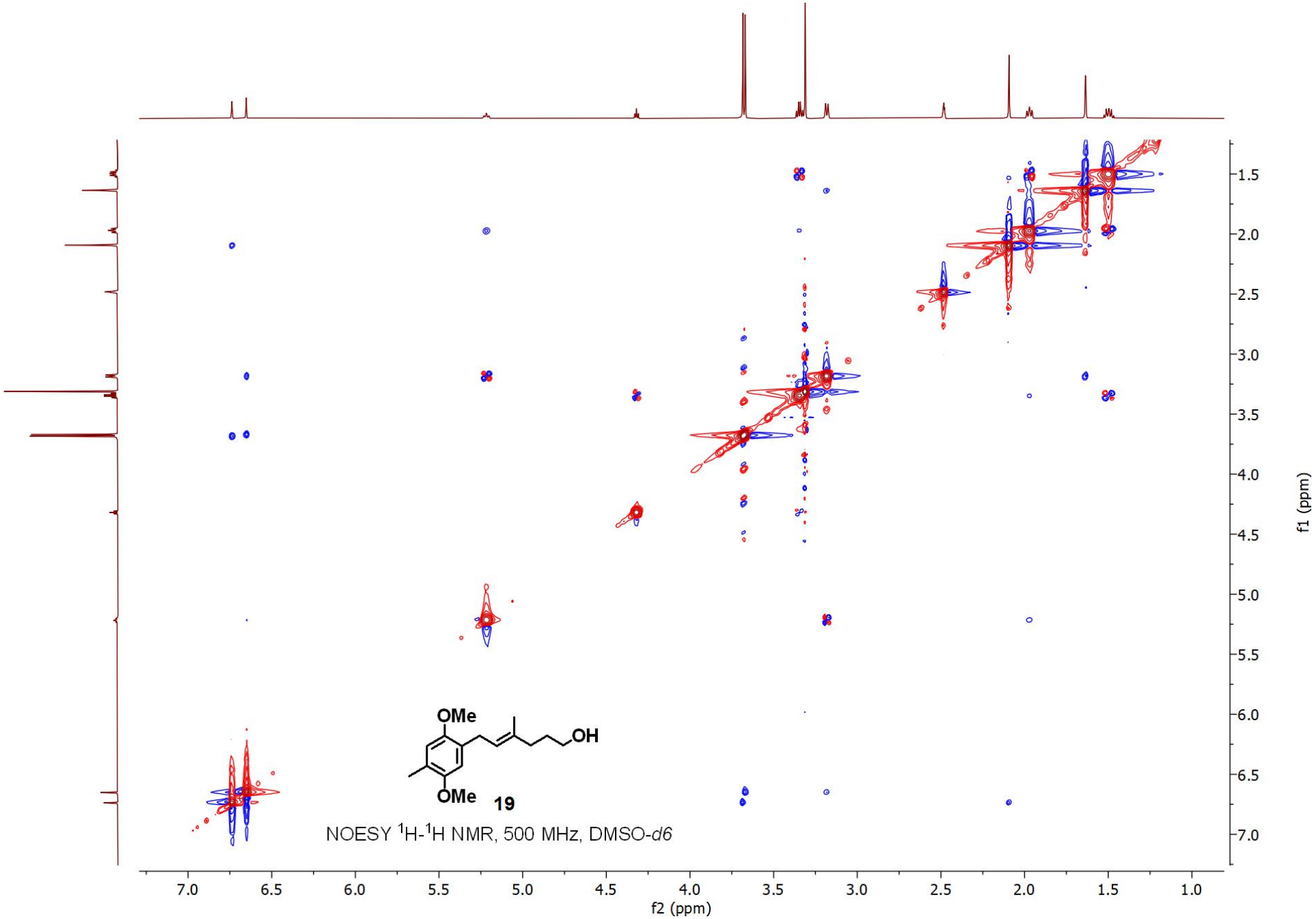
^{1}H NMR, 500 MHz, DMSO-*d6*



19

$^{13}C\{^{1}H\}$ NMR, 126 MHz, DMSO-*d6*

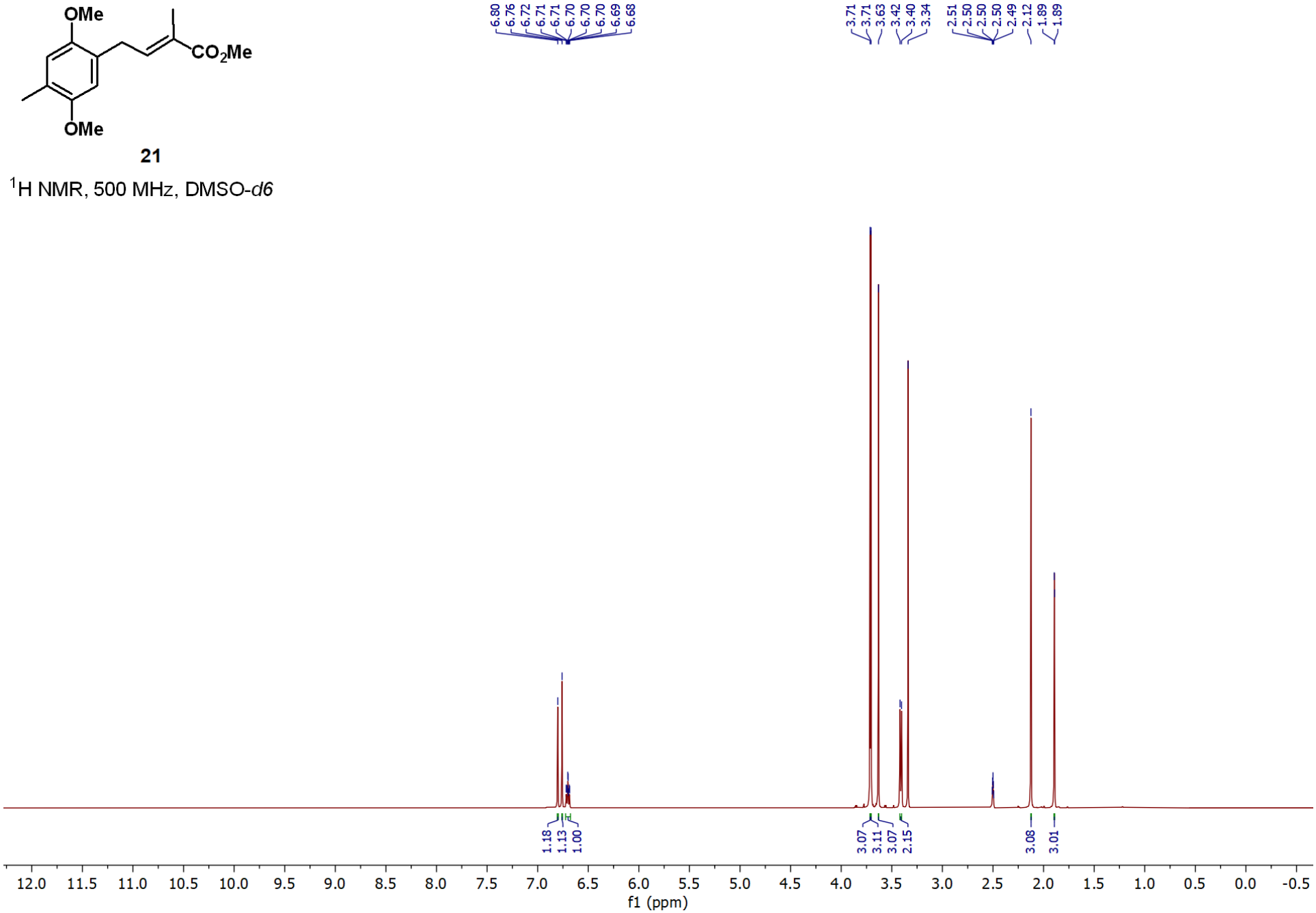




NOESY ^{1}H-^{1}H NMR, 500 MHz, DMSO-*d6*

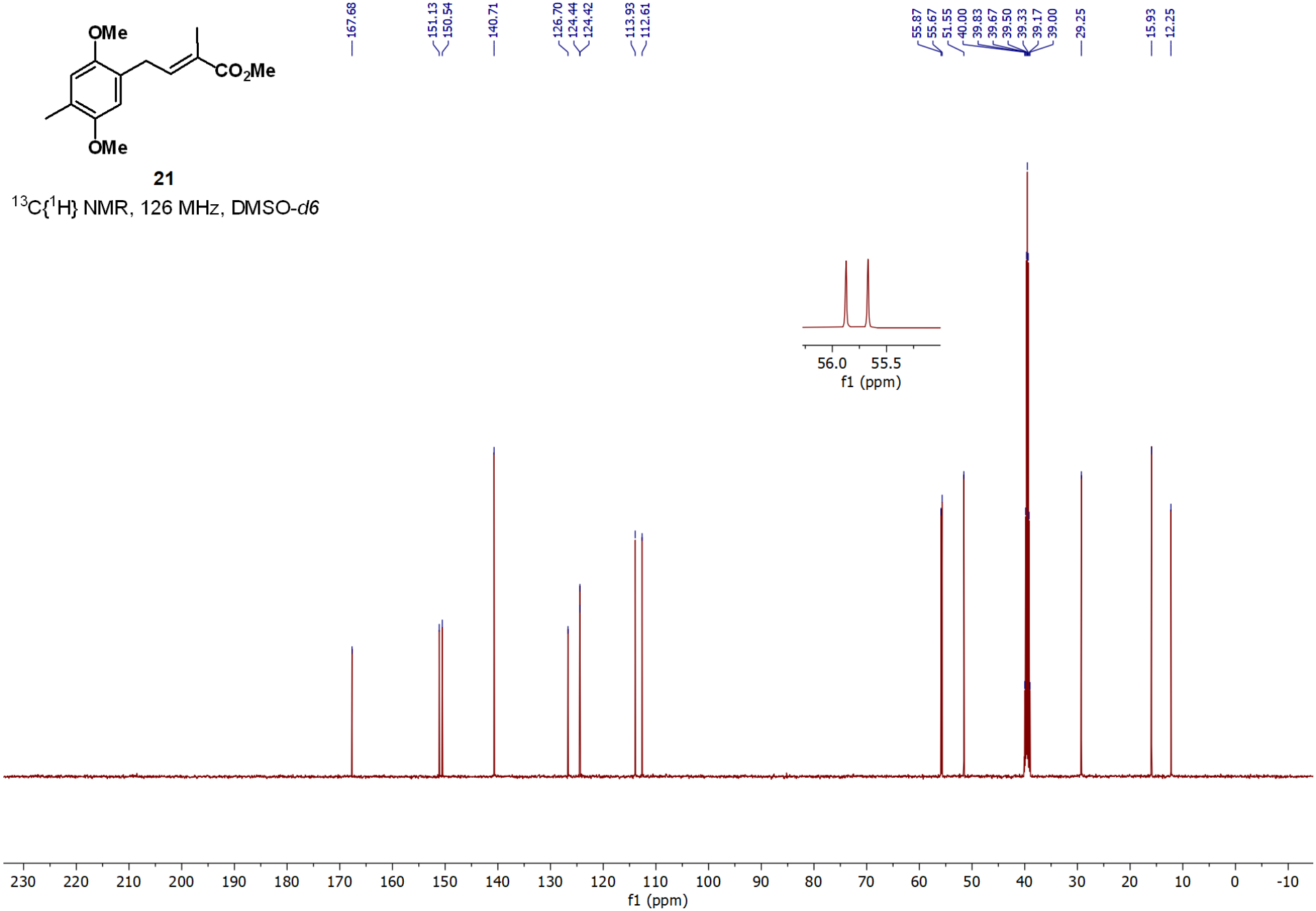
21

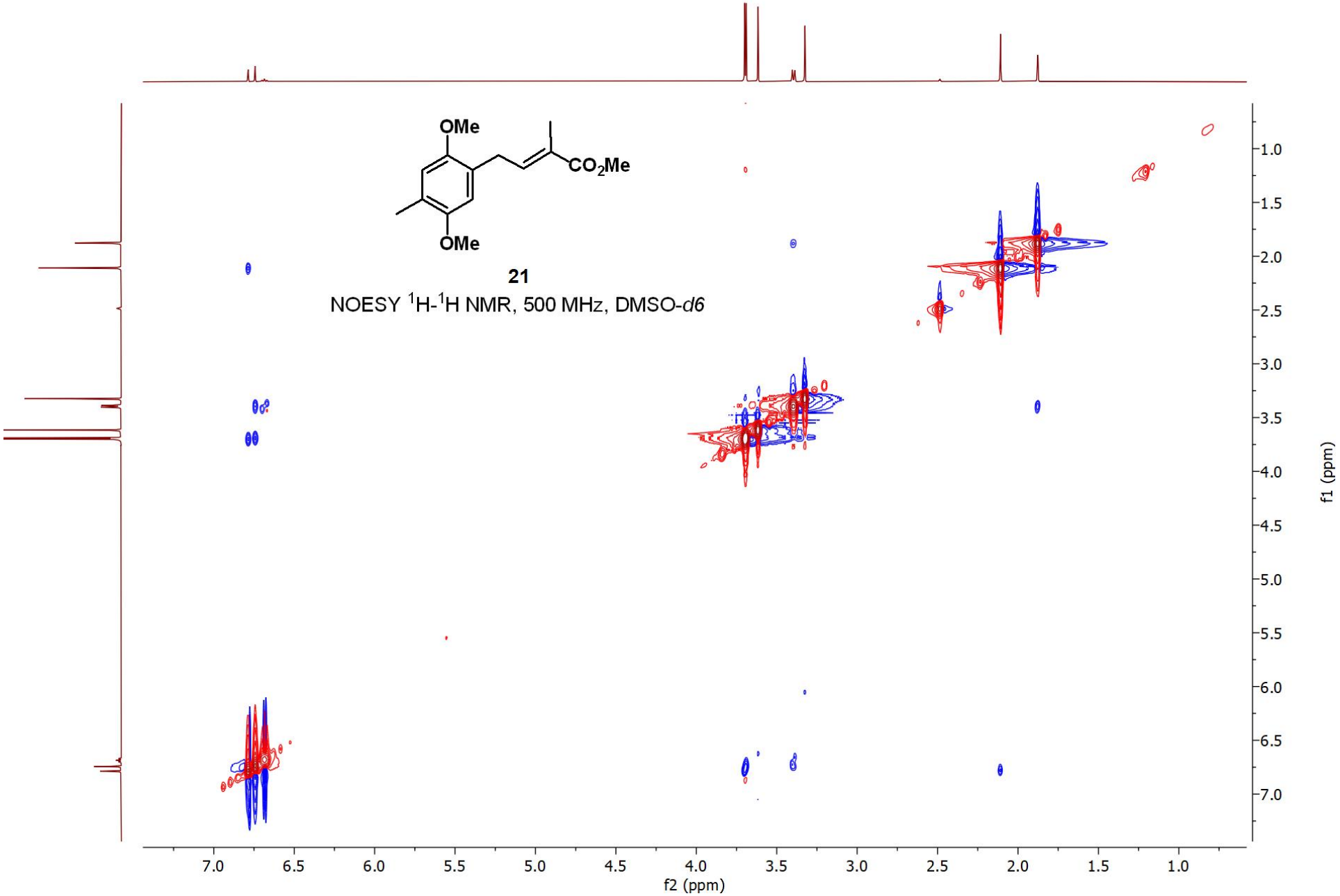
^{1}H NMR, 500 MHz, DMSO-*d6*



21

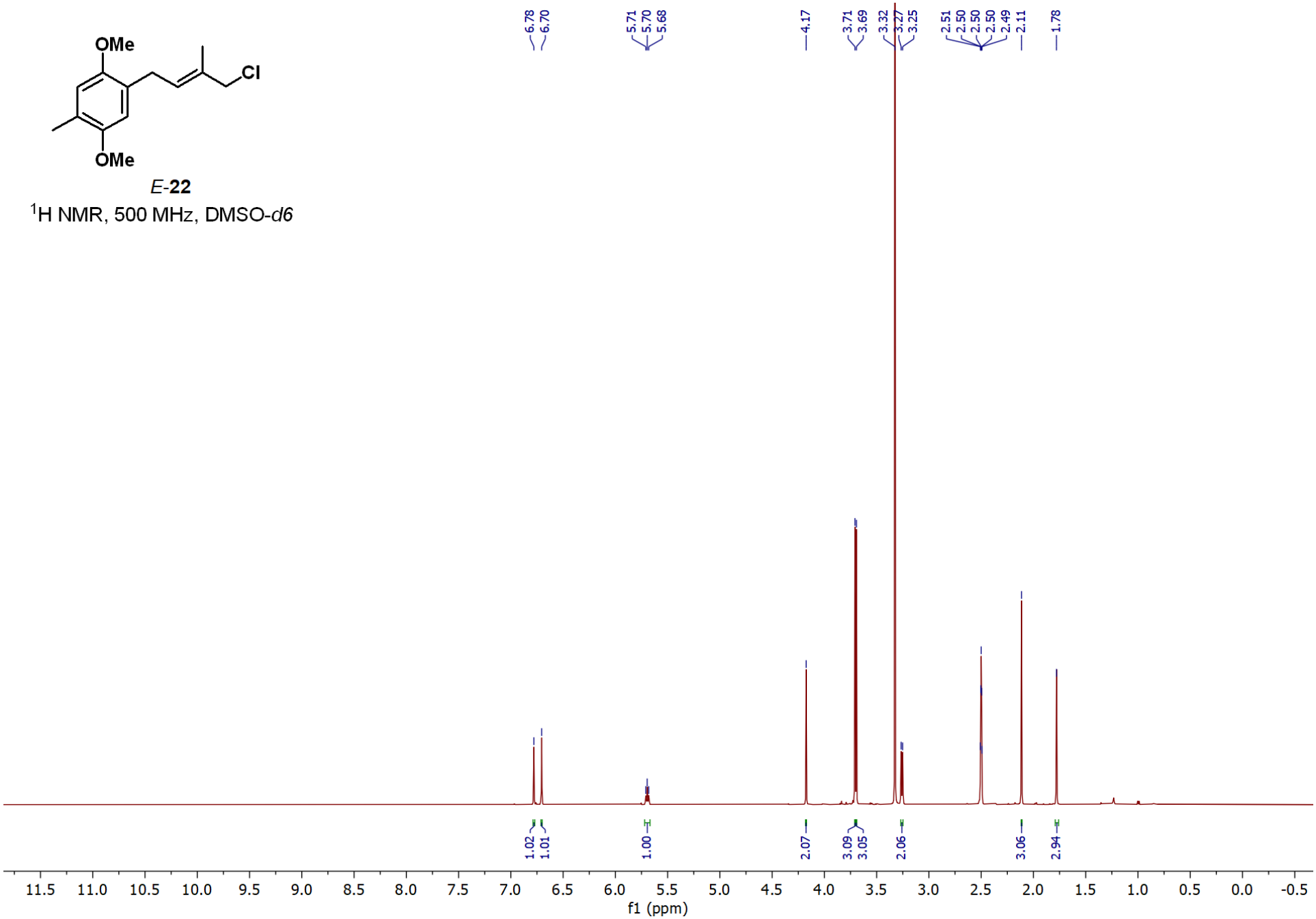
$^{13}C\{^{1}H\}$ NMR, 126 MHz, DMSO-*d6*




OMe
CO2Me
OMe
21
NOESY 1H-1H NMR, 500 MHz, DMSO-d6
f2 (ppm)
f1 (ppm)

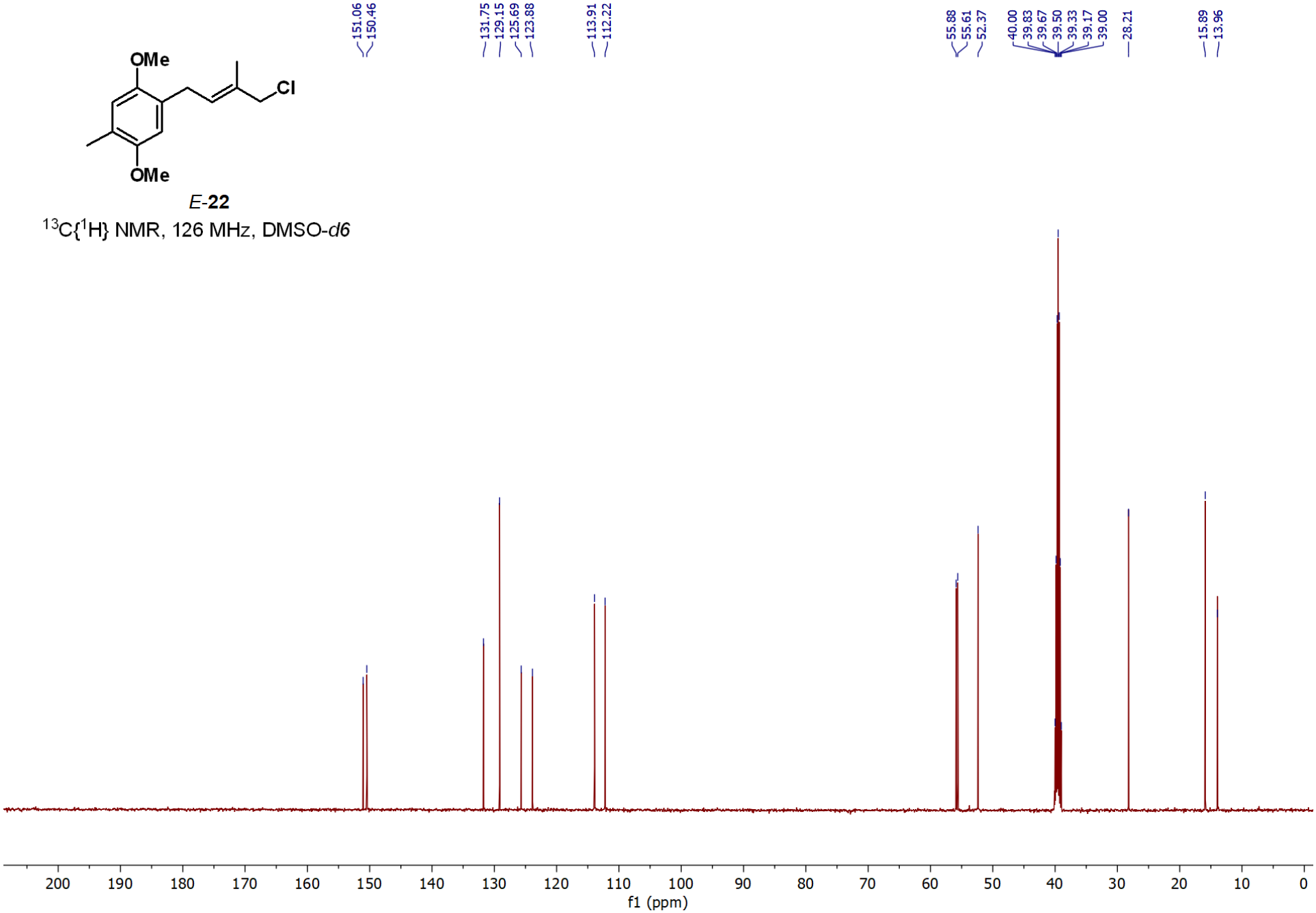
E-**22**

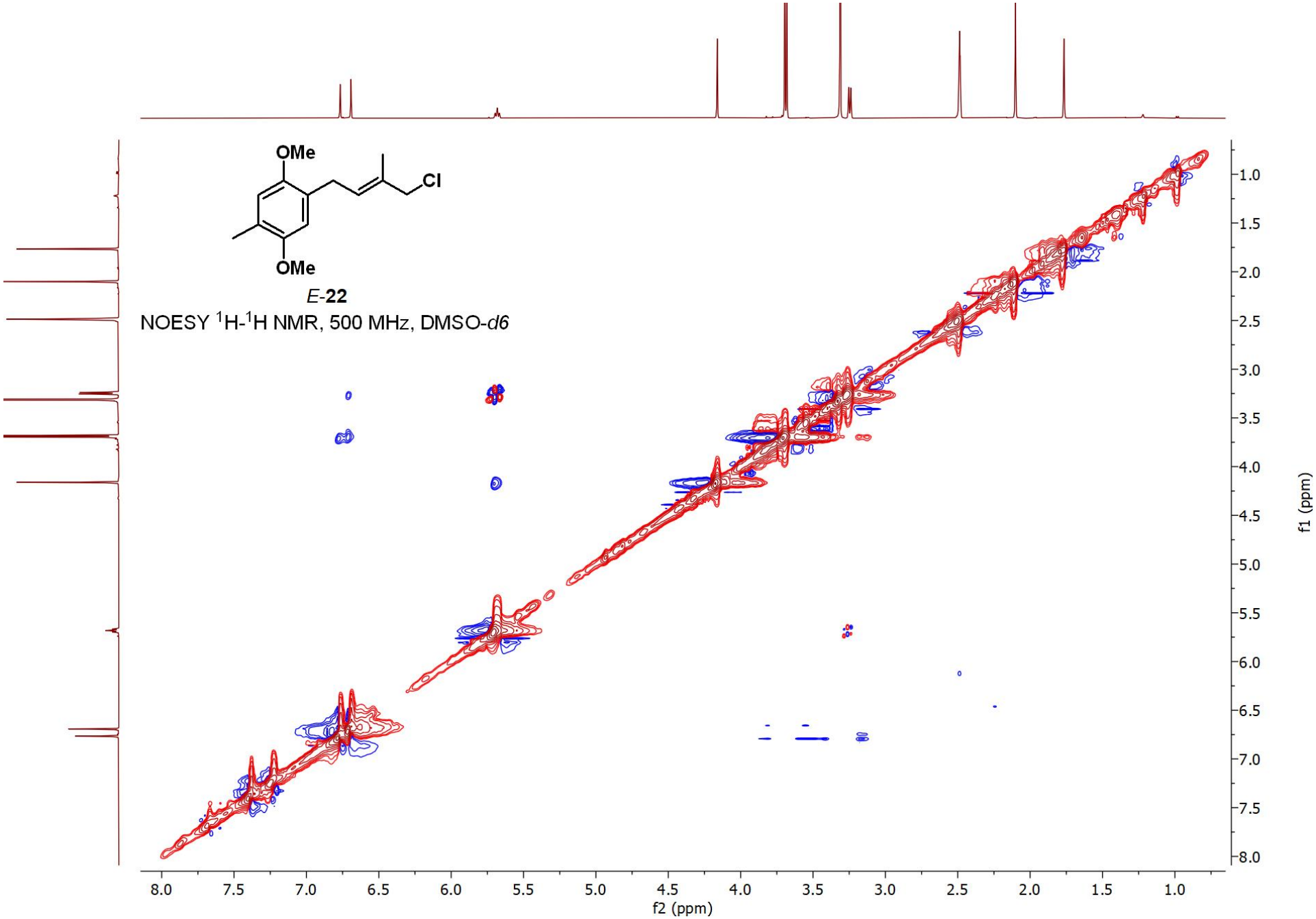
^{1}H NMR, 500 MHz, DMSO-*d6*



E-**22**

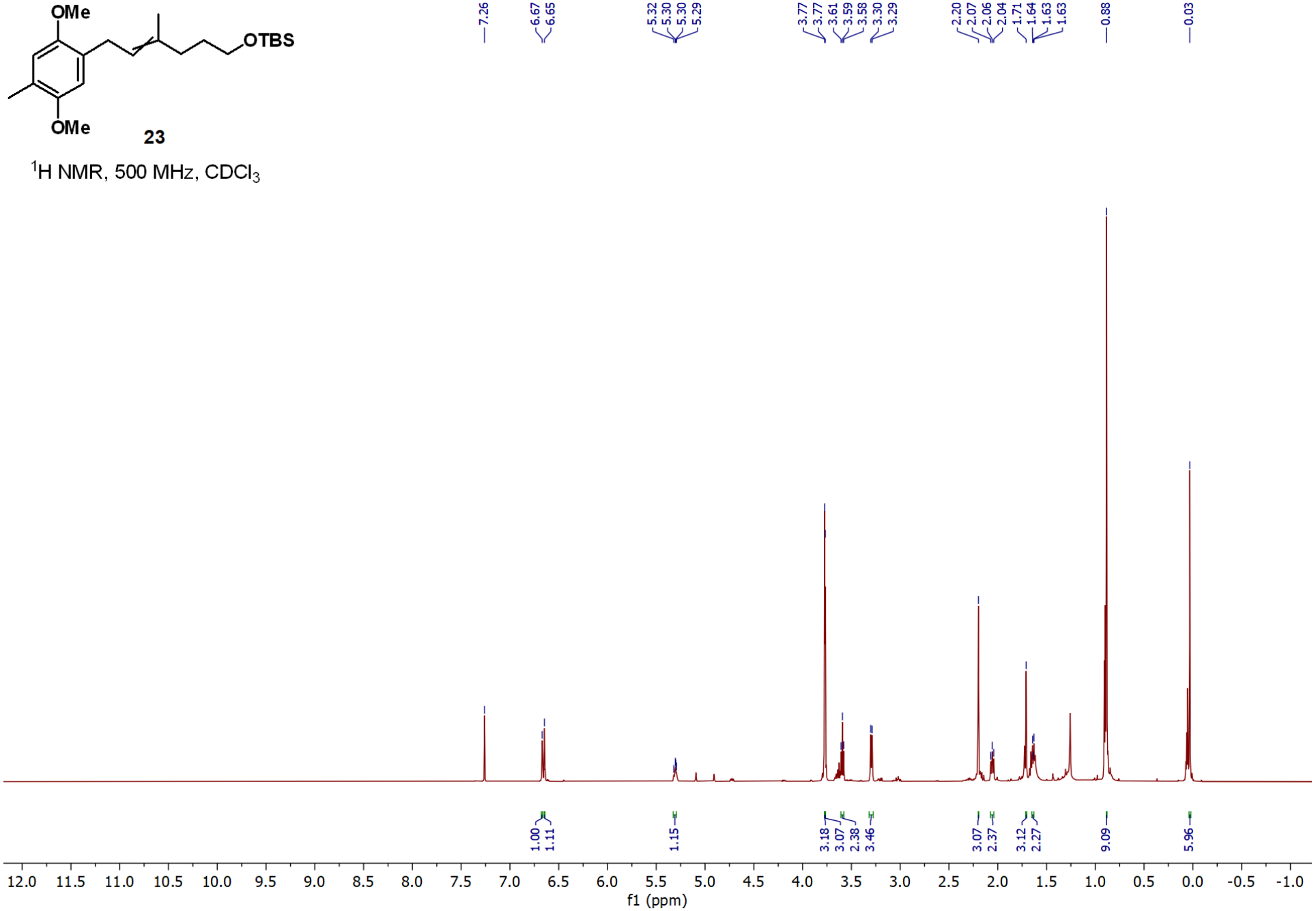
$^{13}C\{^{1}H\}$ NMR, 126 MHz, DMSO-*d6*




OMe
Cl
OMe
E-**22**
NOESY ^{1}H-^{1}H NMR, 500 MHz, DMSO-*d6*
f2 (ppm)
f1 (ppm)

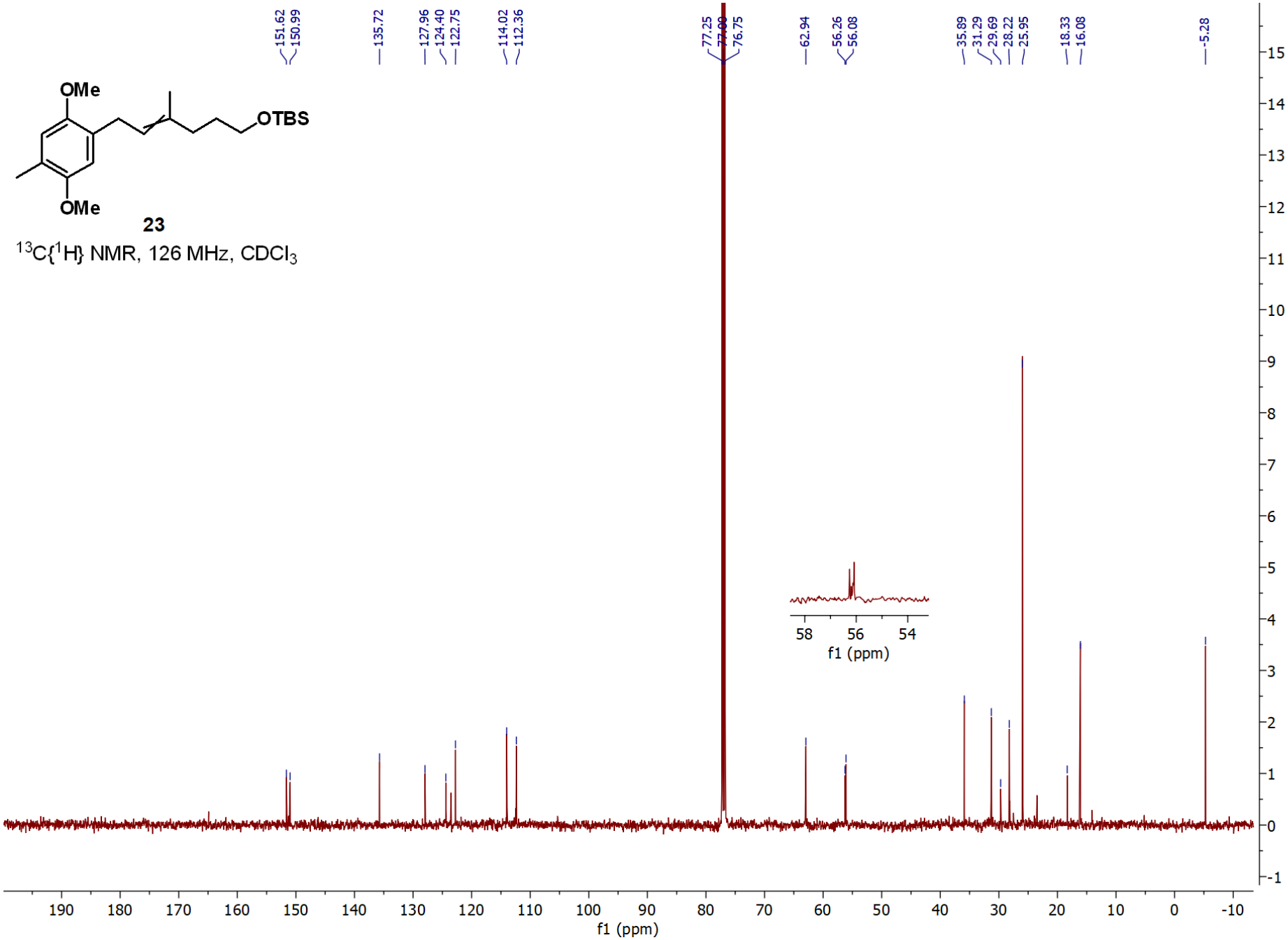
23

^{1}H NMR, 500 MHz, $CDCl_3$



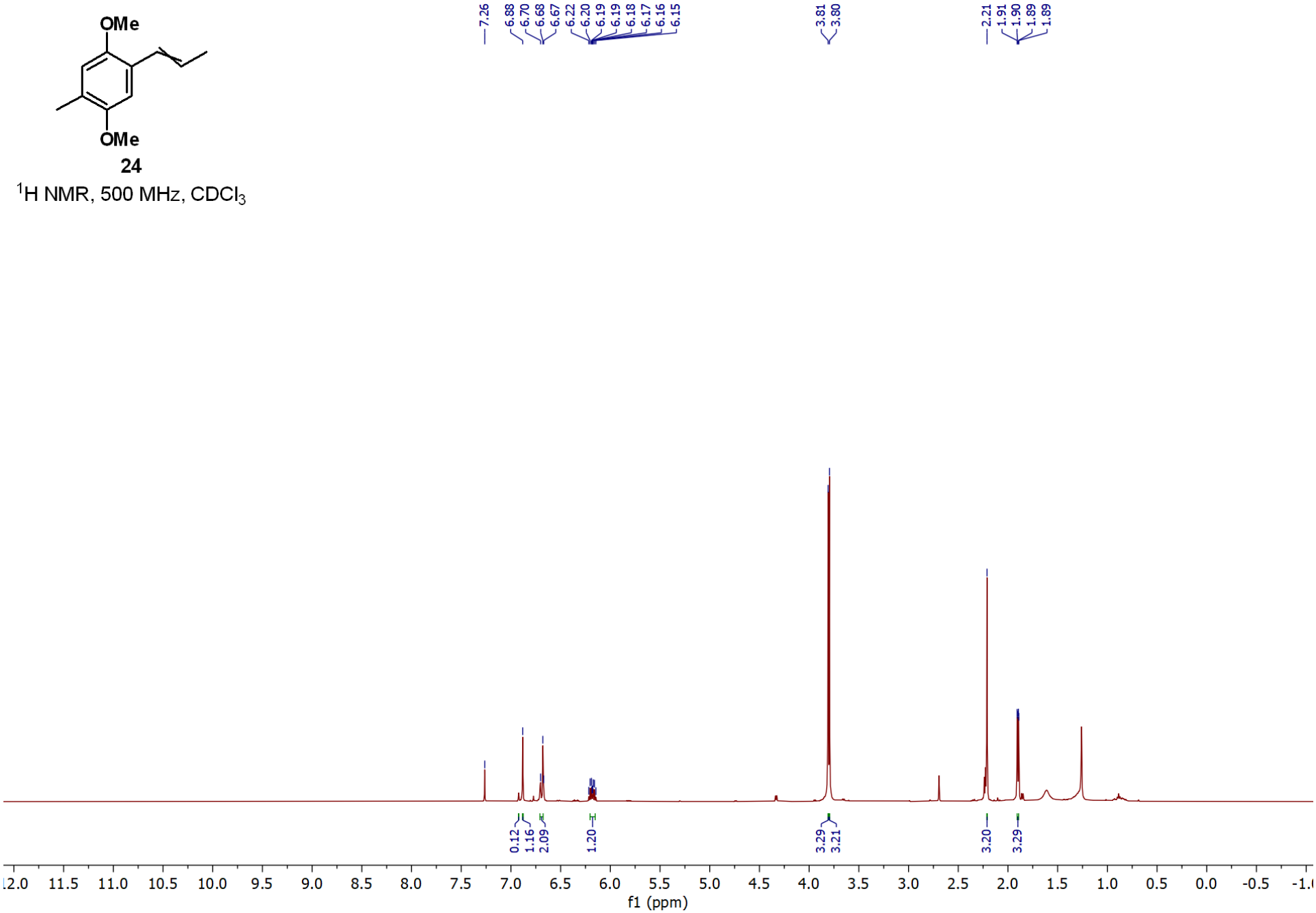
23

$^{13}C\{^{1}H\}$ NMR, 126 MHz, $CDCl_3$



24

^{1}H NMR, 500 MHz, $CDCl_3$



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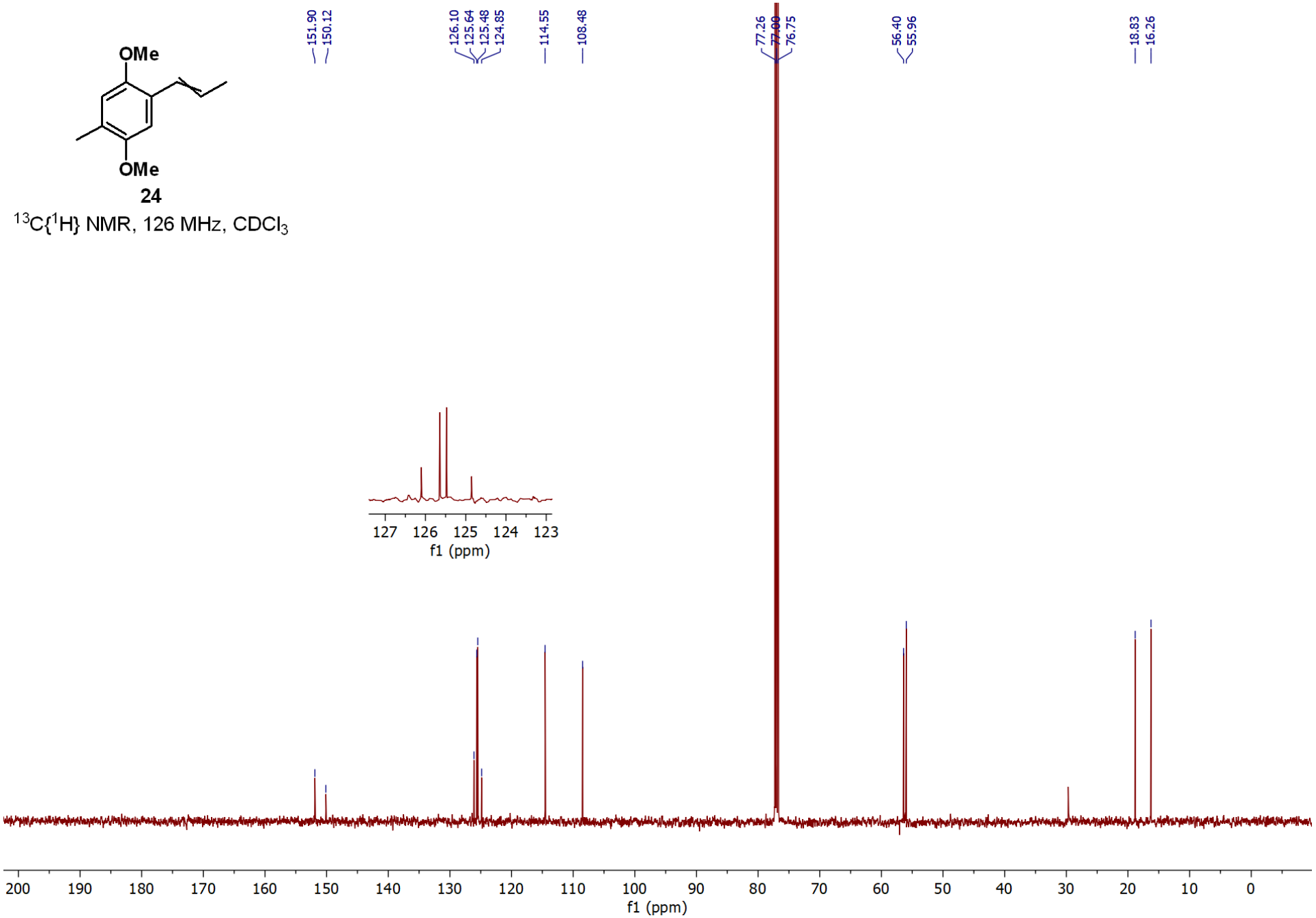
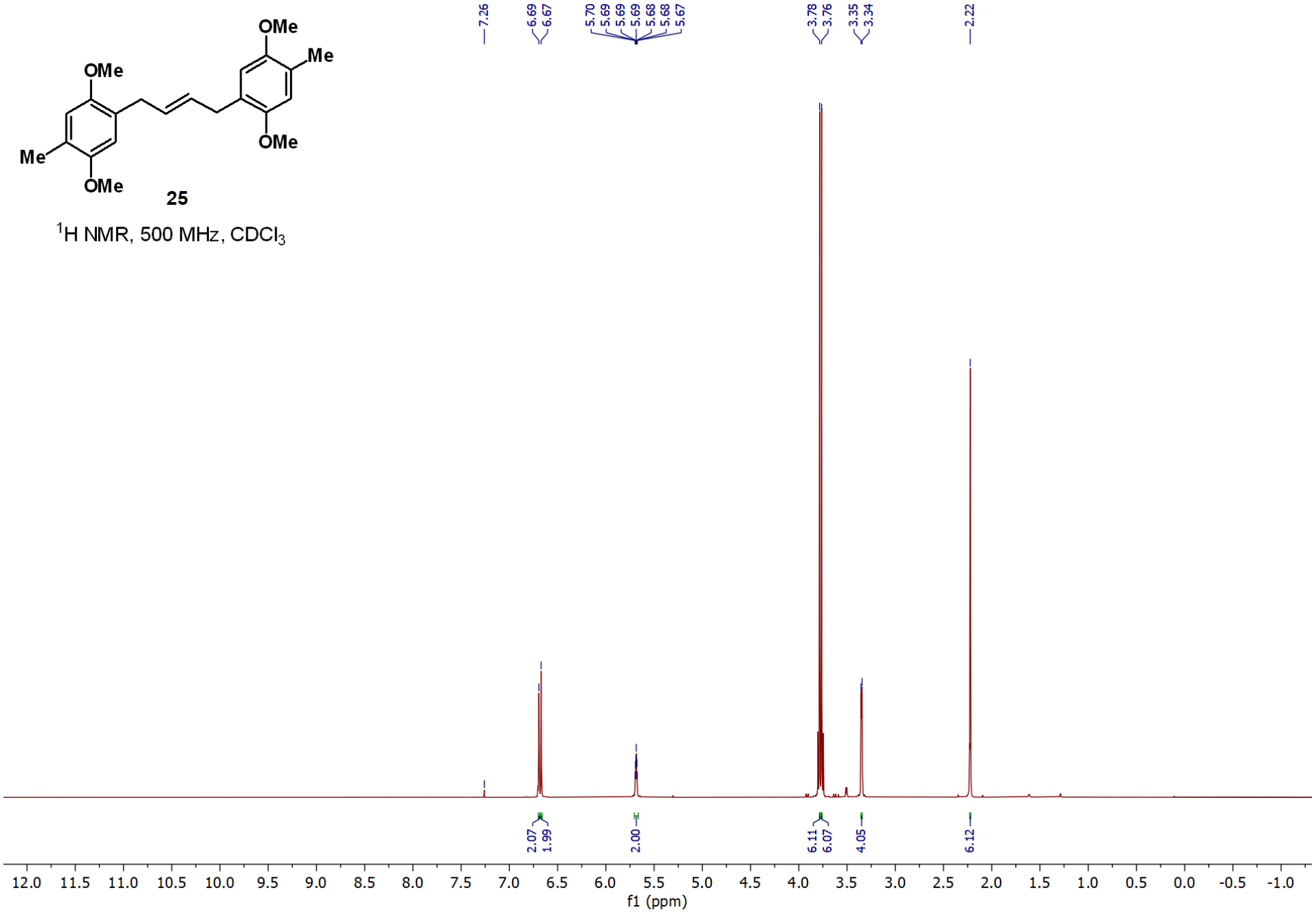
$^{13}C\{^{1}H\}$ NMR, 126 MHz, $CDCl_3$



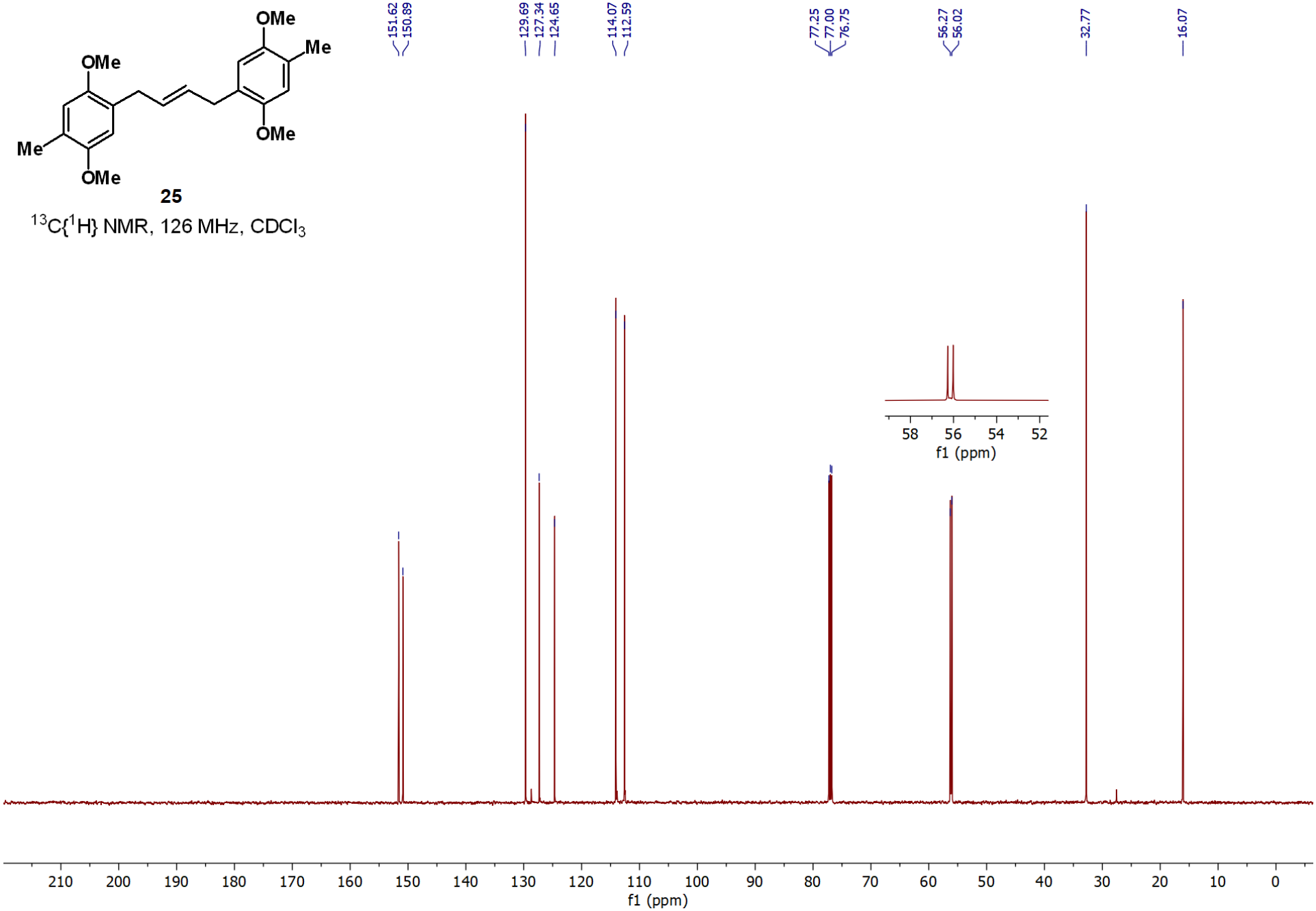
25

^{1}H NMR, 500 MHz, $CDCl_3$



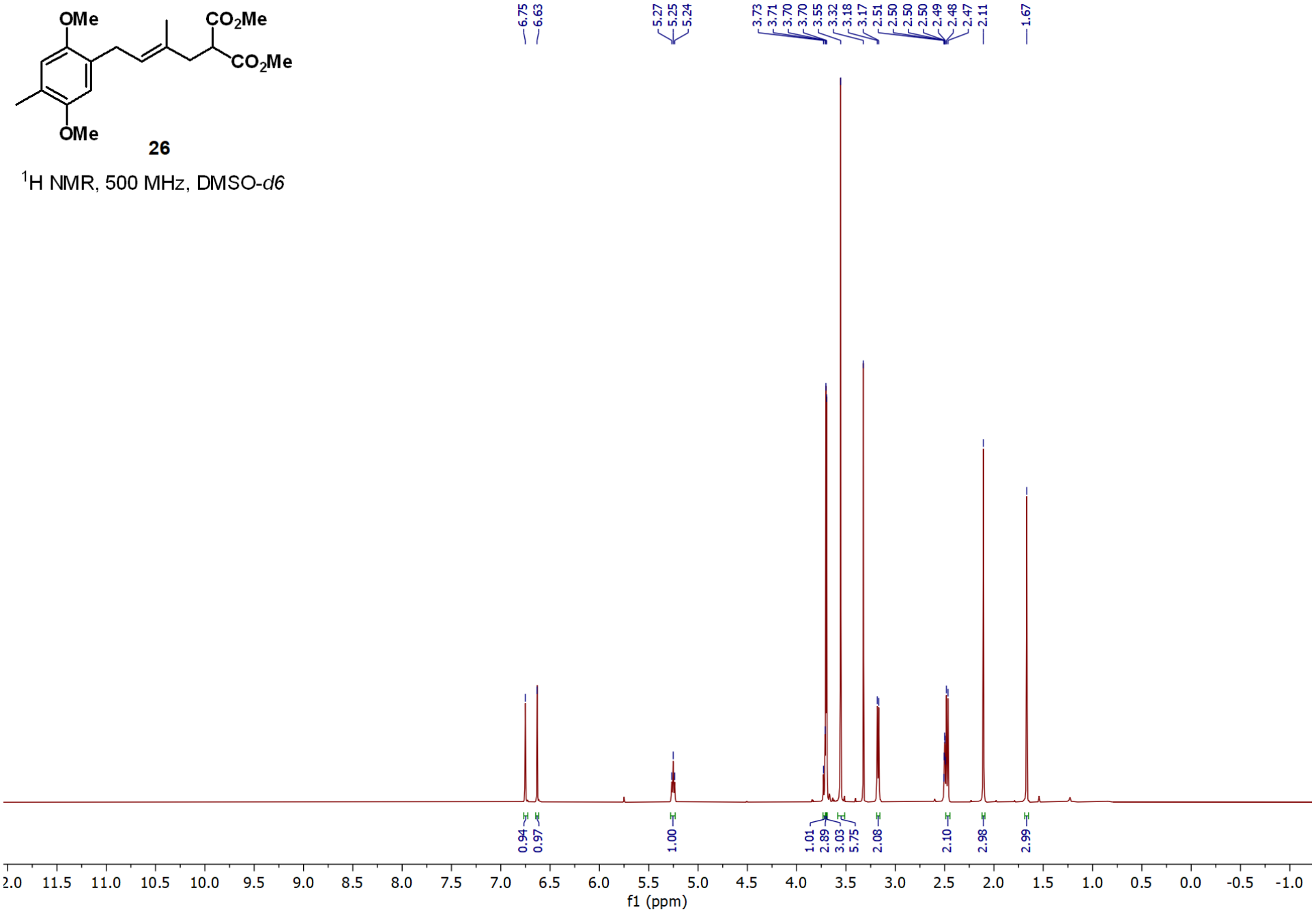
25

$^{13}C\{^{1}H\}$ NMR, 126 MHz, $CDCl_3$



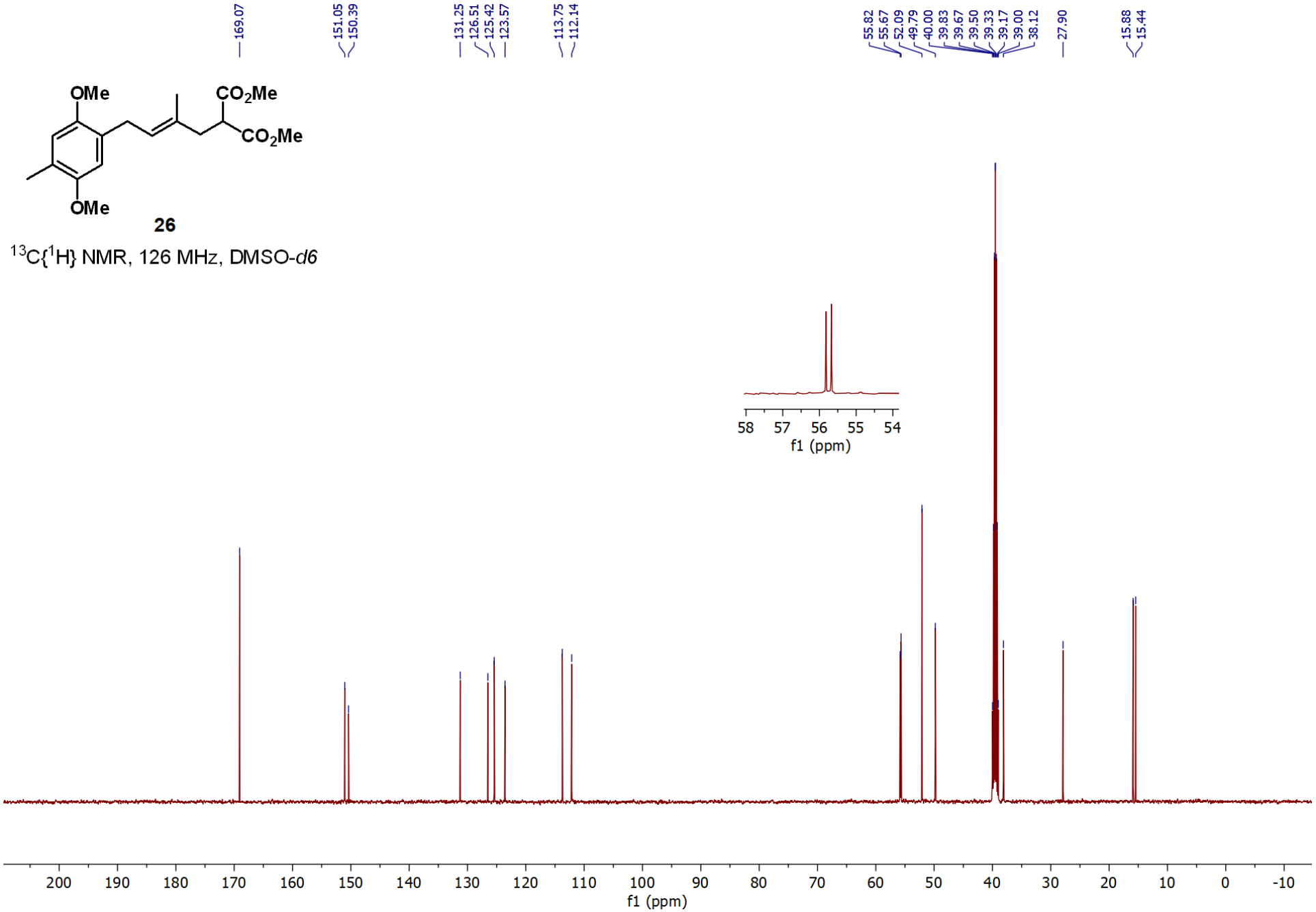
26

^{1}H NMR, 500 MHz, DMSO-*d6*



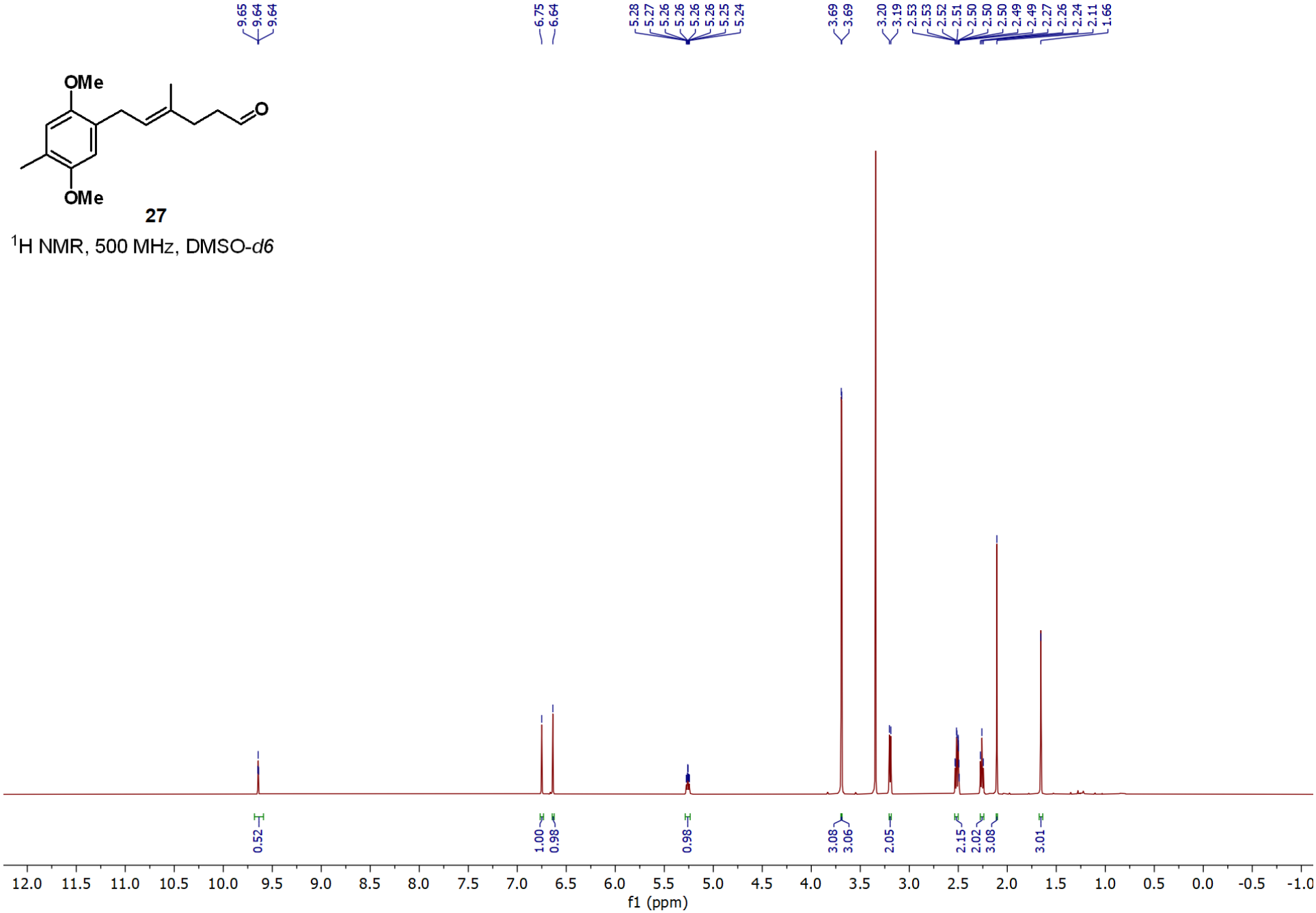
26

$^{13}C\{^{1}H\}$ NMR, 126 MHz, DMSO-*d6*



27

^{1}H NMR, 500 MHz, DMSO-*d6*



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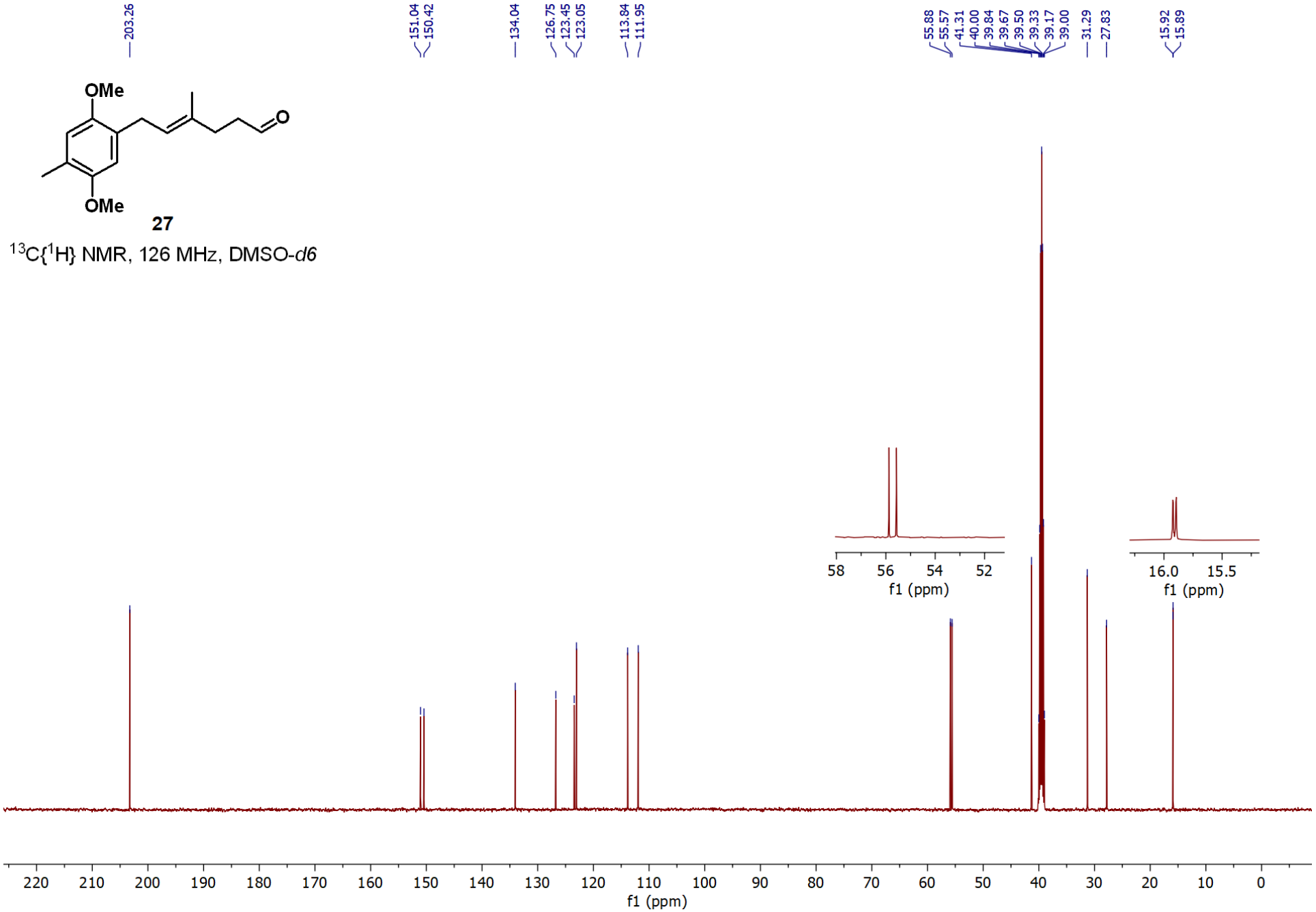
$^{13}C\{^{1}H\}$ NMR, 126 MHz, DMSO-*d6*



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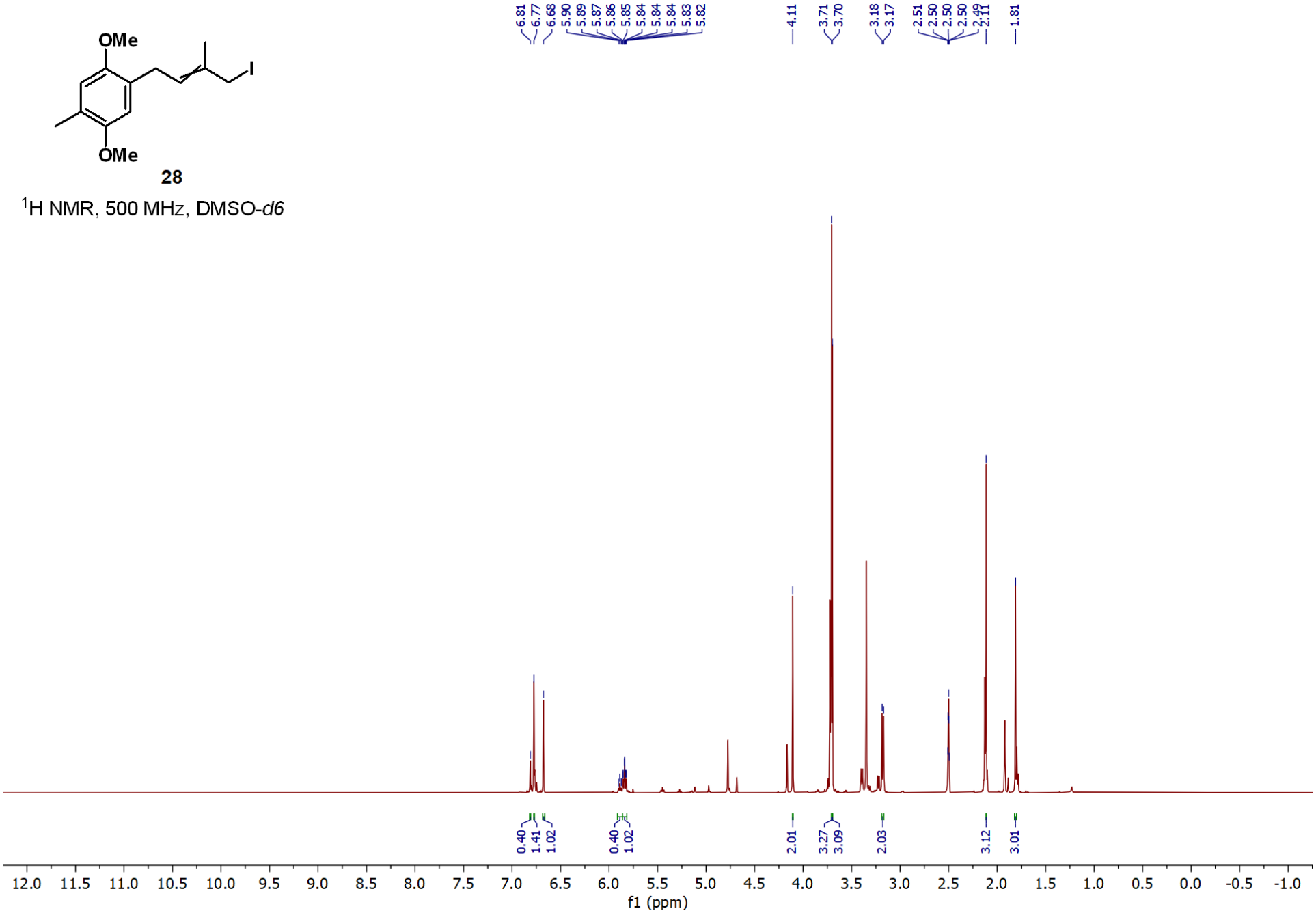
^{1}H NMR, 500 MHz, DMSO-*d6*



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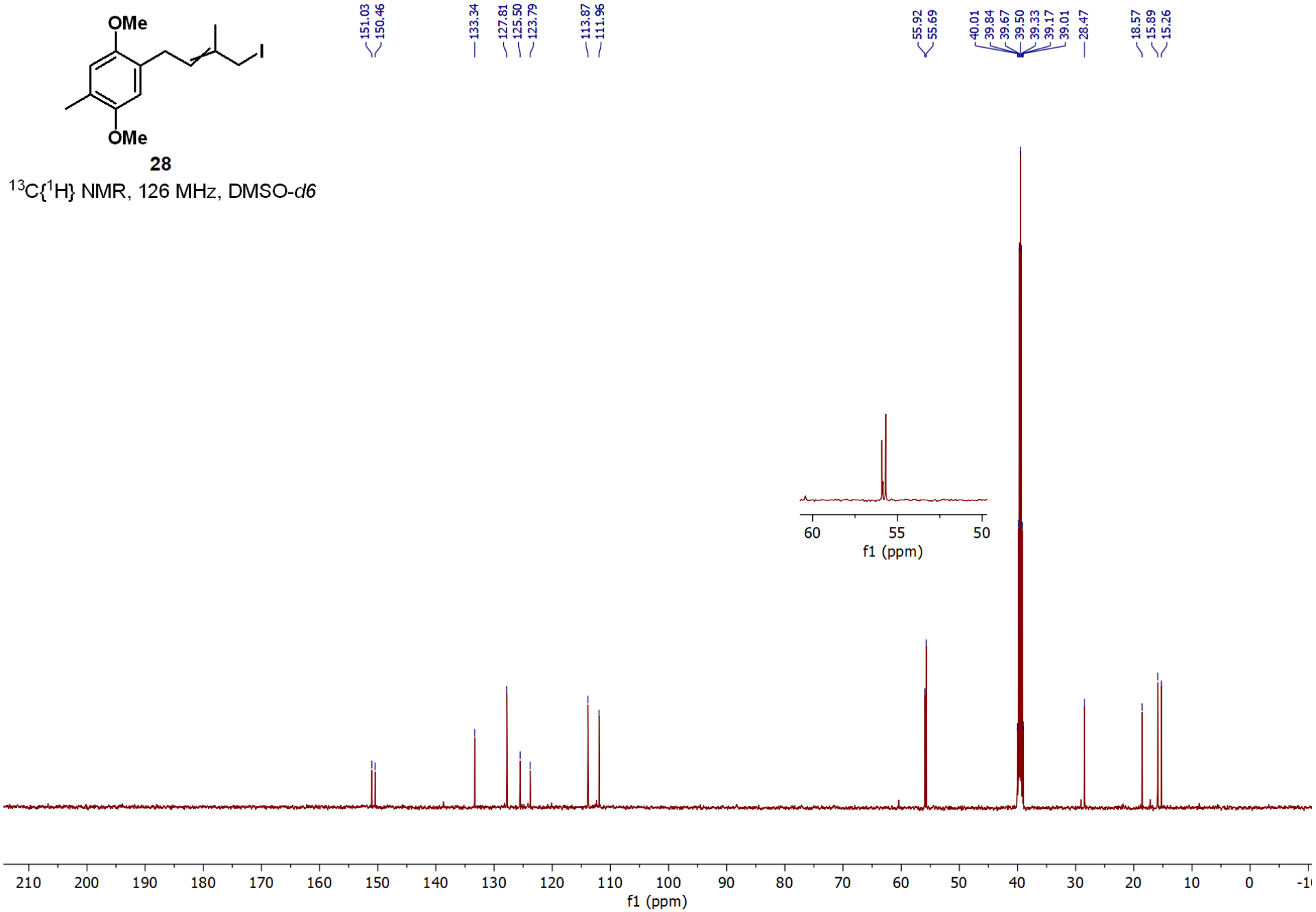
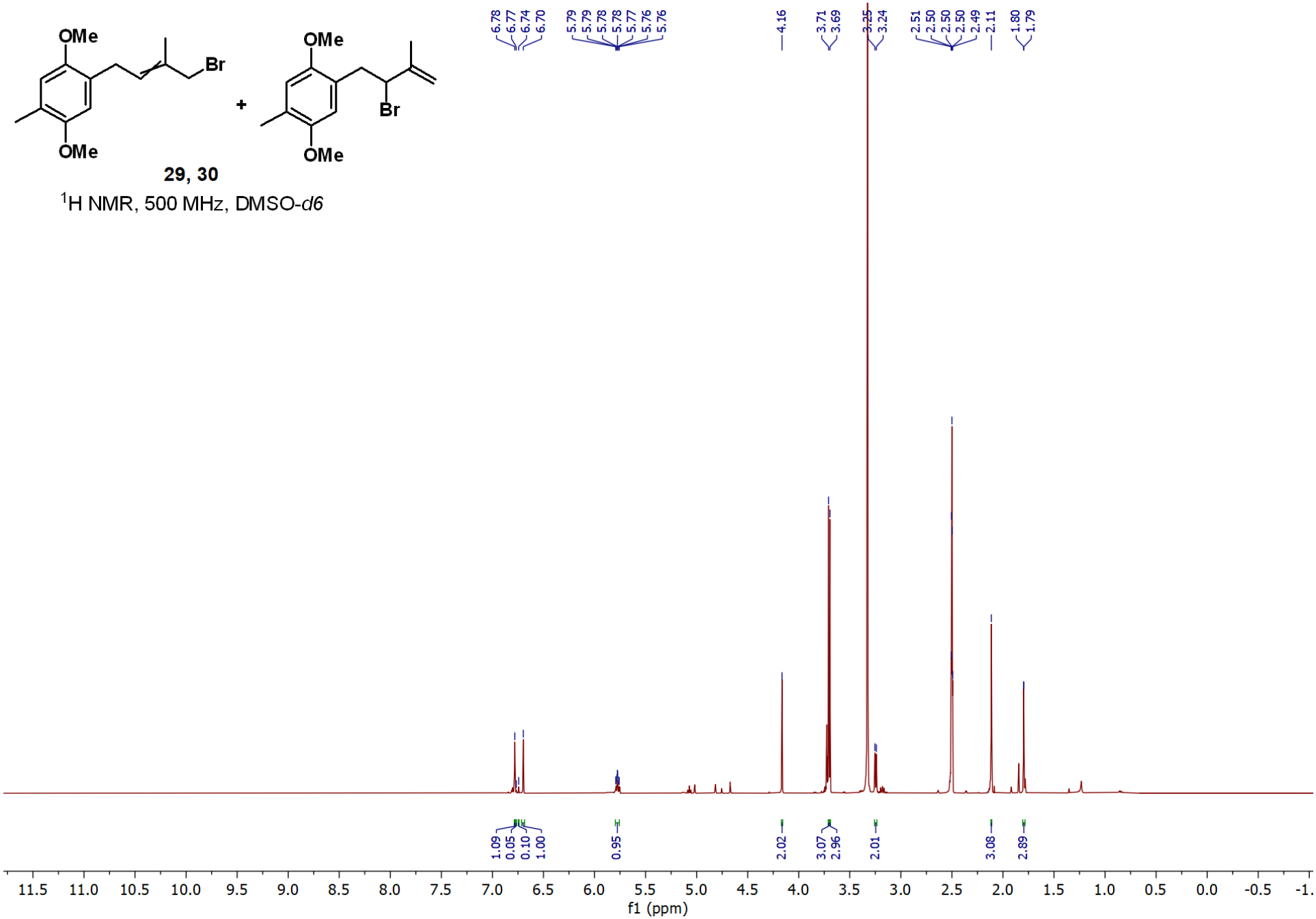
$^{13}C\{^{1}H\}$ NMR, 126 MHz, DMSO-*d6*



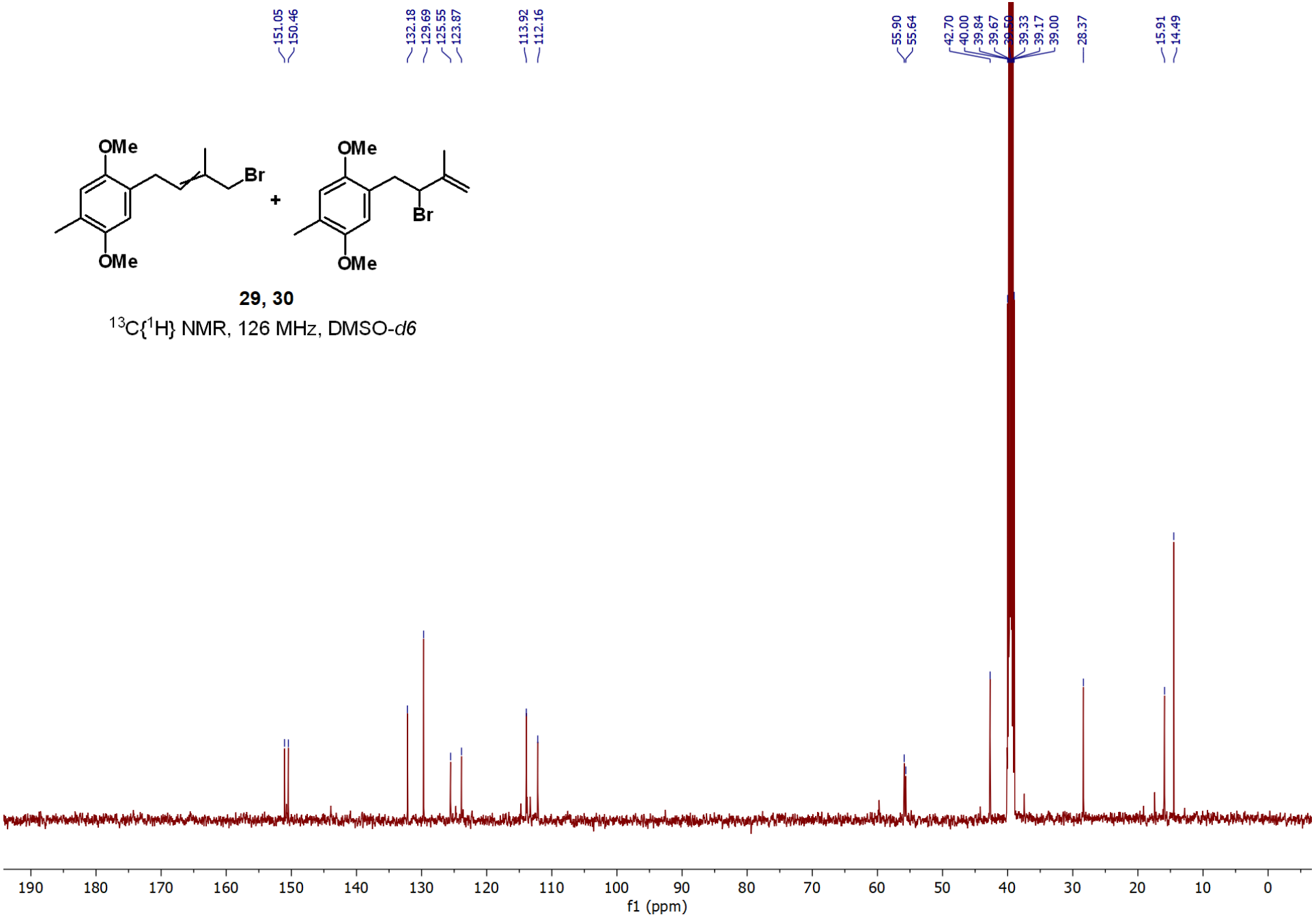
29, 30

^{1}H NMR, 500 MHz, DMSO-*d6*



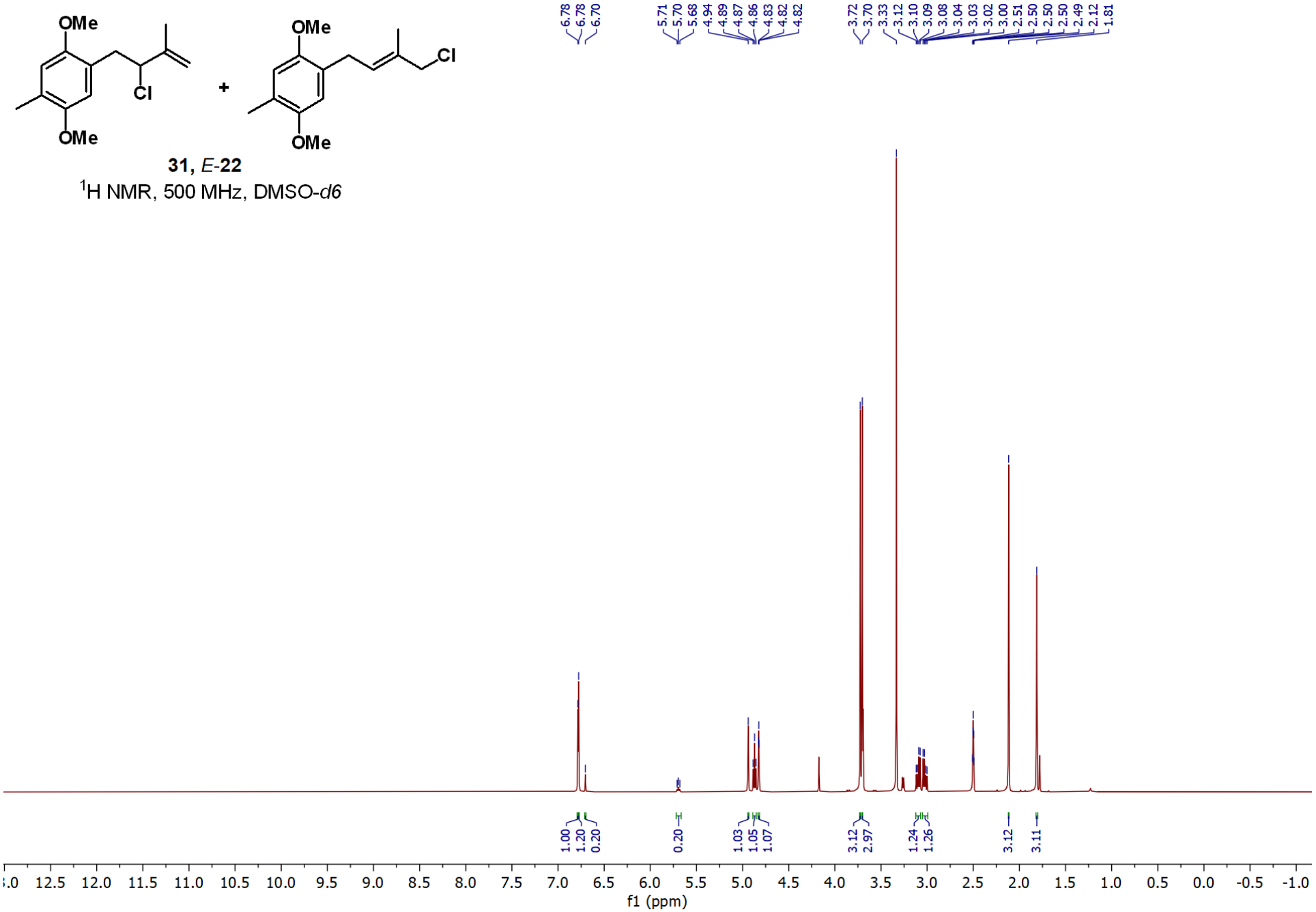
29, 30

$^{13}C\{^{1}H\}$ NMR, 126 MHz, DMSO-*d6*



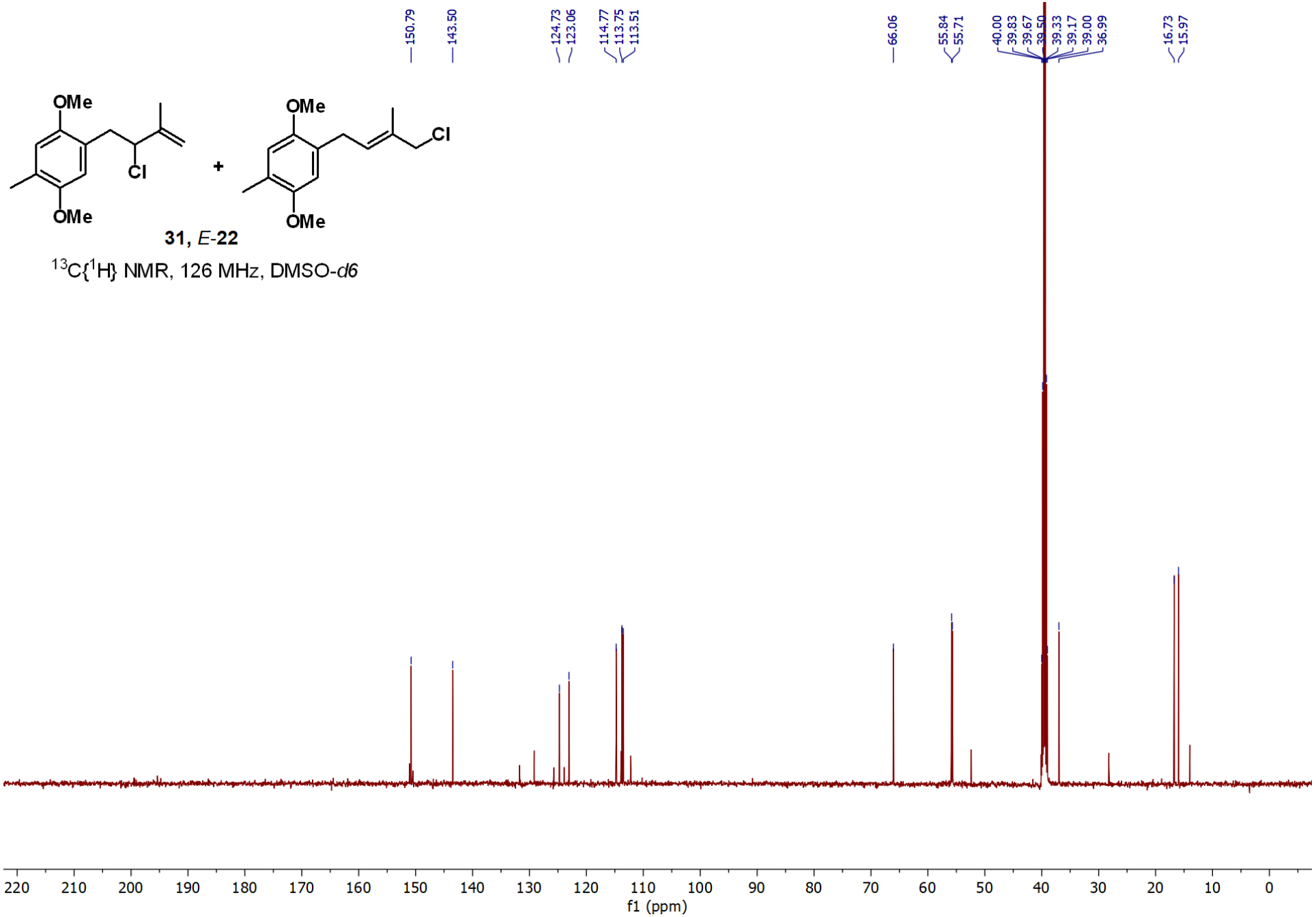
31, *E*-**22**

^{1}H NMR, 500 MHz, DMSO-*d6*



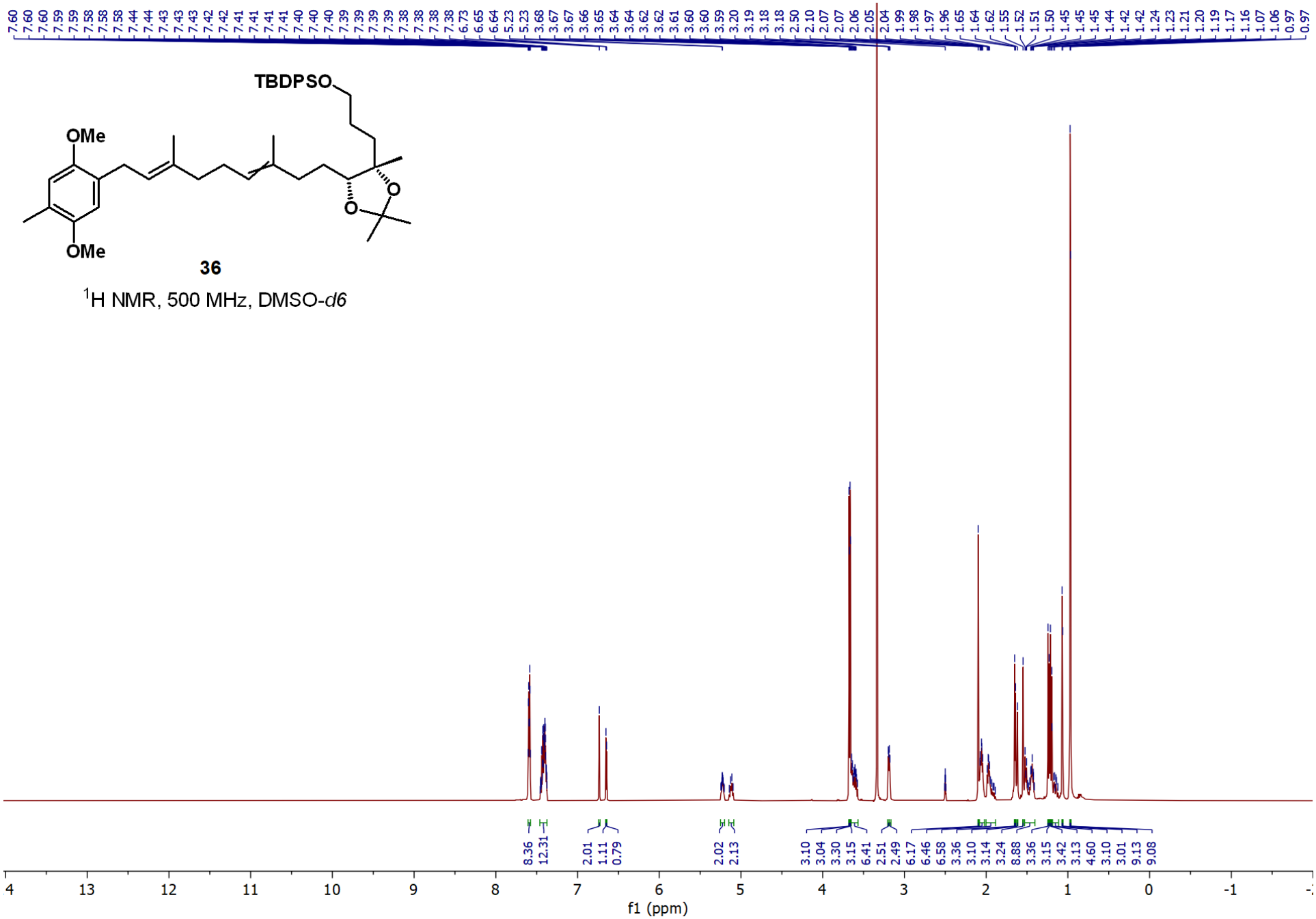
31, *E*-**22**

$^{13}C\{^{1}H\}$ NMR, 126 MHz, DMSO-*d6*



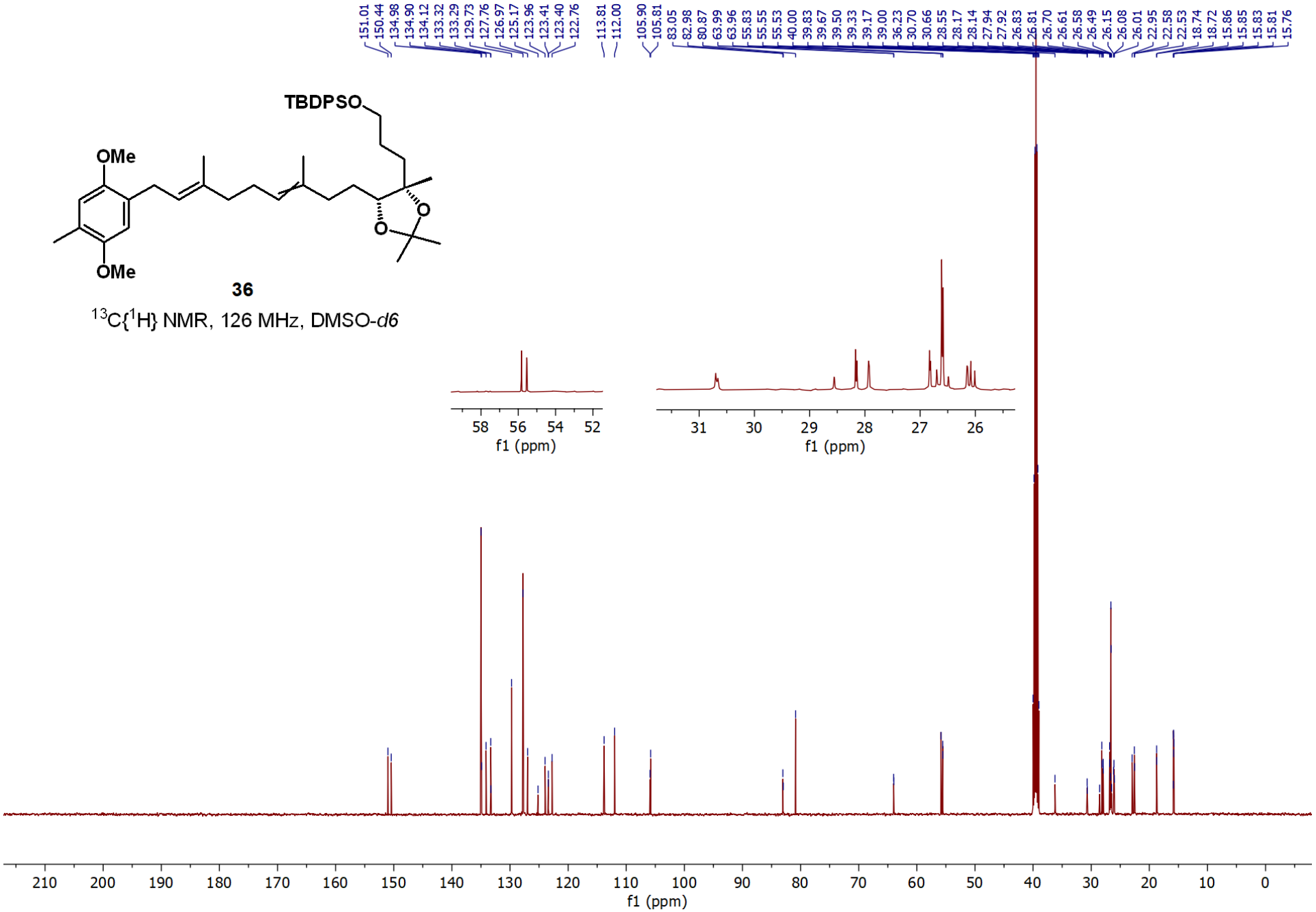
36

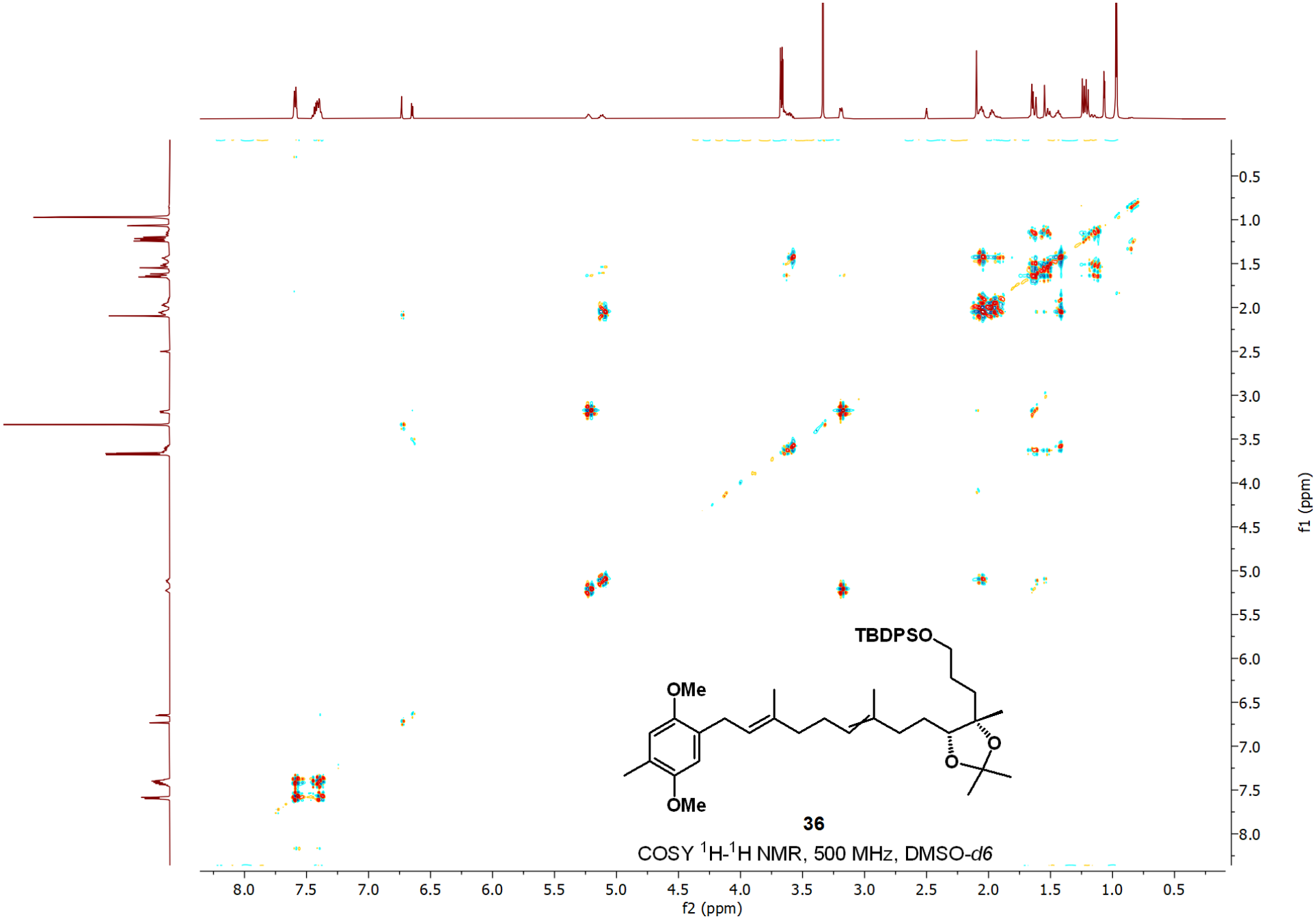
^{1}H NMR, 500 MHz, DMSO-*d6*



36

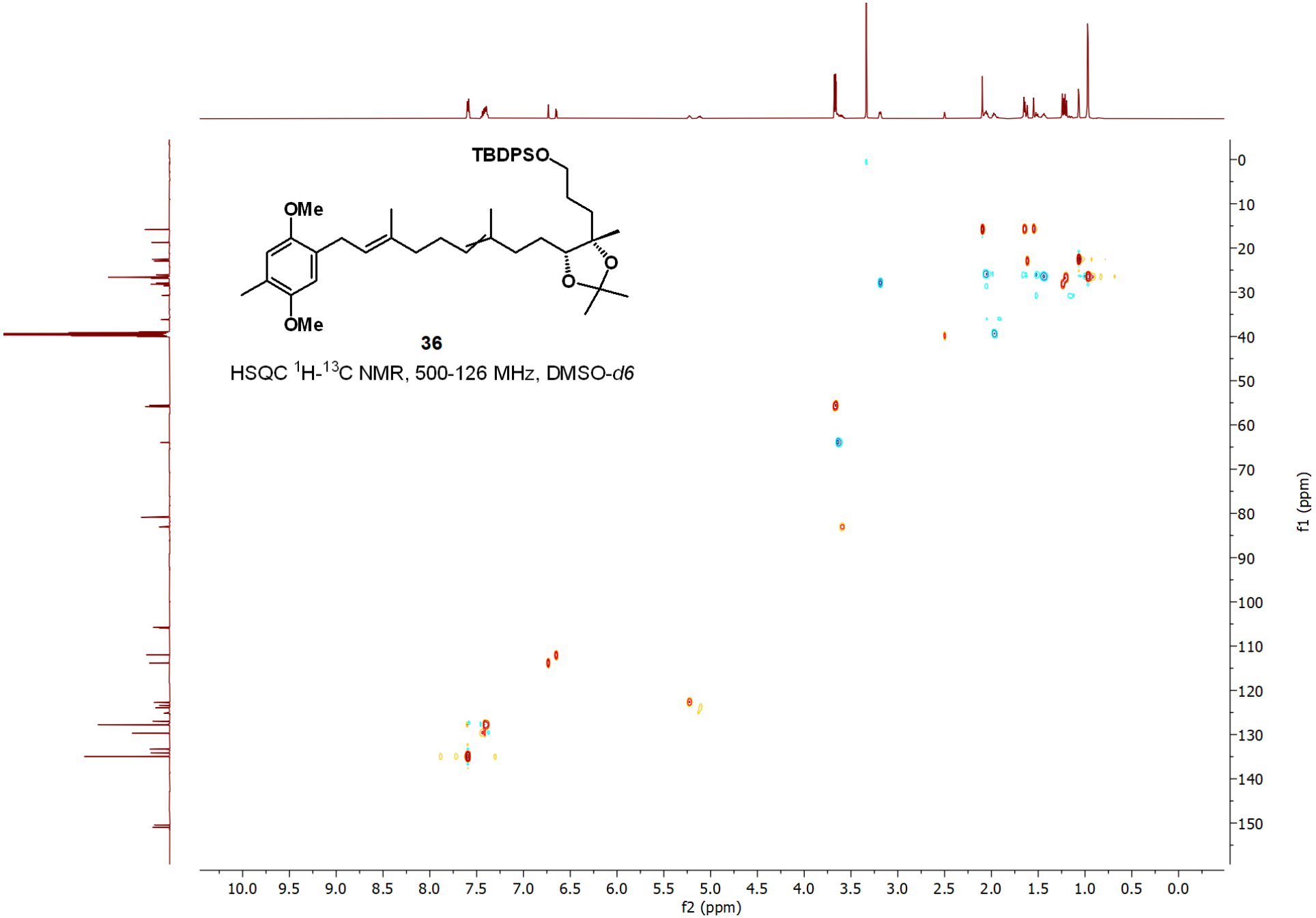
$^{13}C\{^{1}H\}$ NMR, 126 MHz, DMSO-*d6*

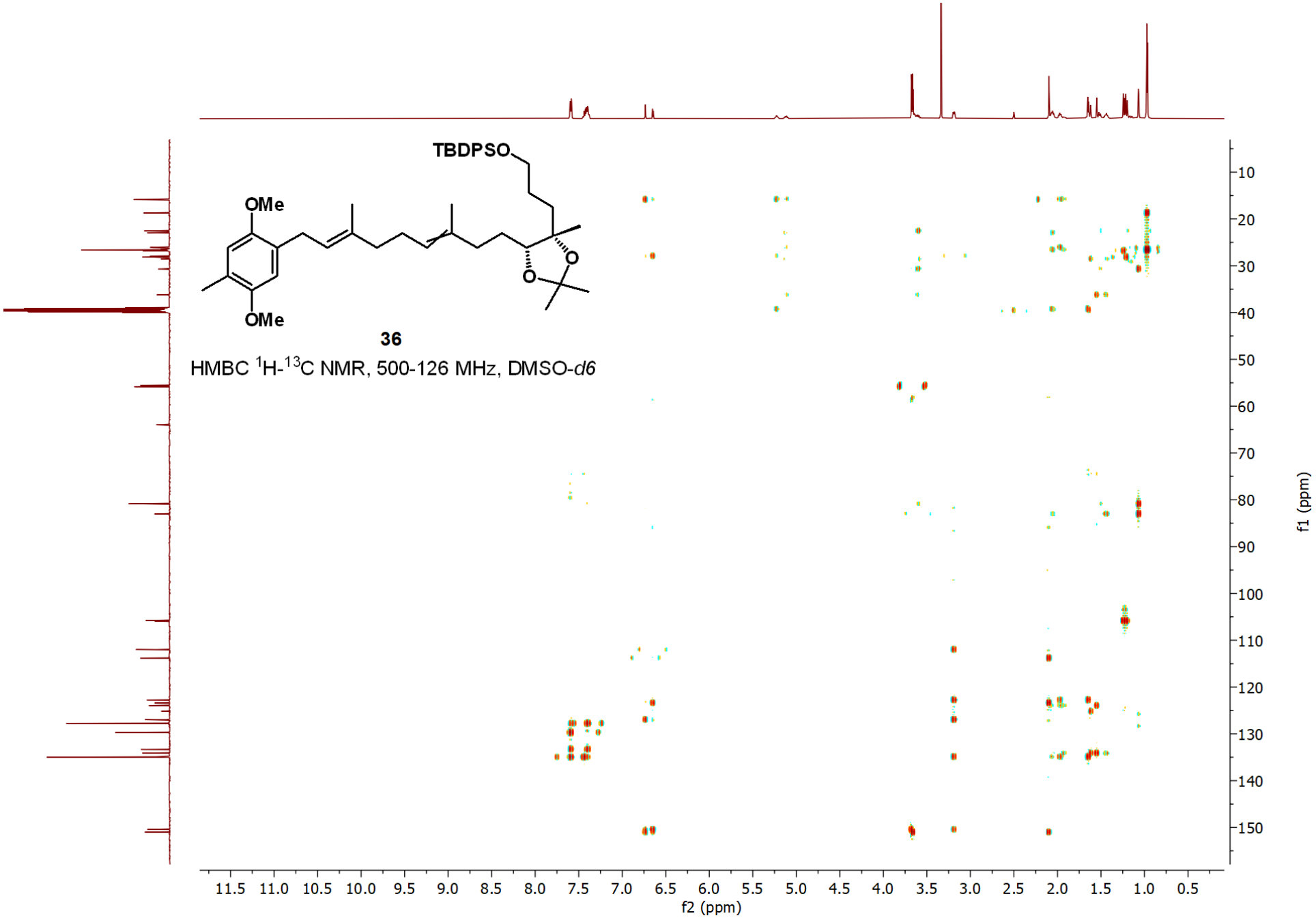




36

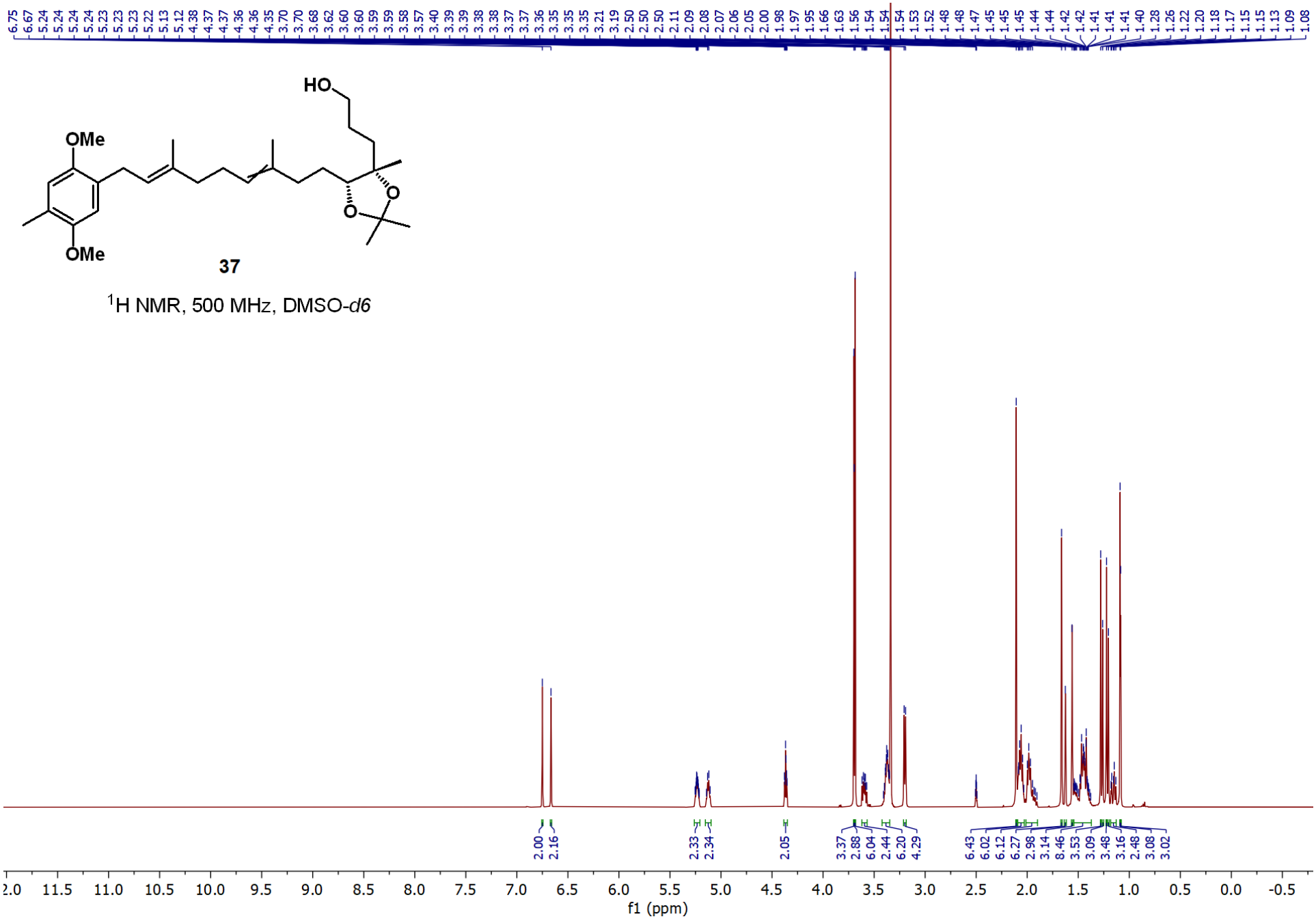
COSY ^{1}H-^{1}H NMR, 500 MHz, DMSO-*d6*

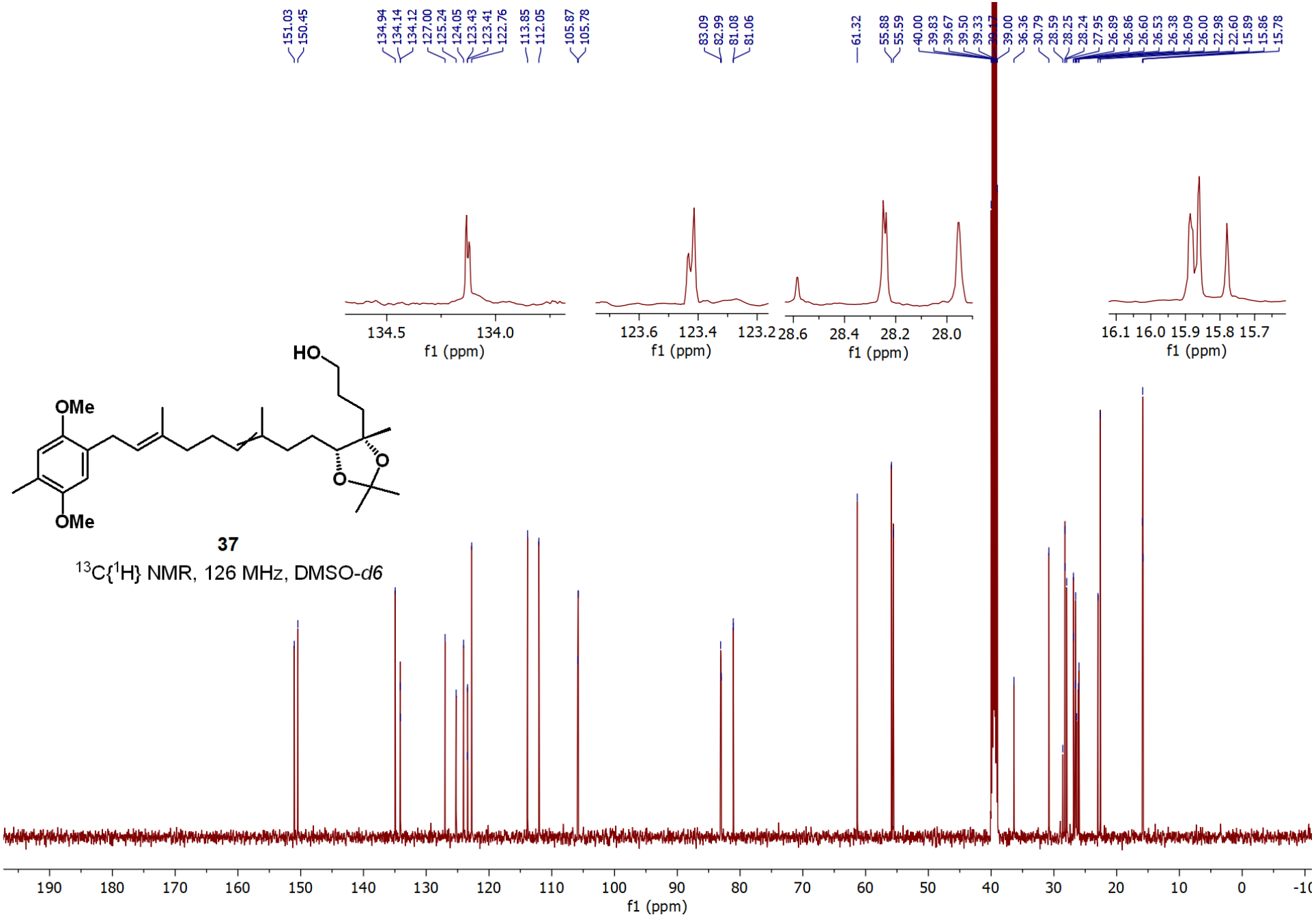

TBDPSO
OMe
OMe
O
O
36
HSQC ^{1}H-^{13}C NMR, 500-126 MHz, DMSO-*d6*
f2 (ppm)
f1 (ppm)

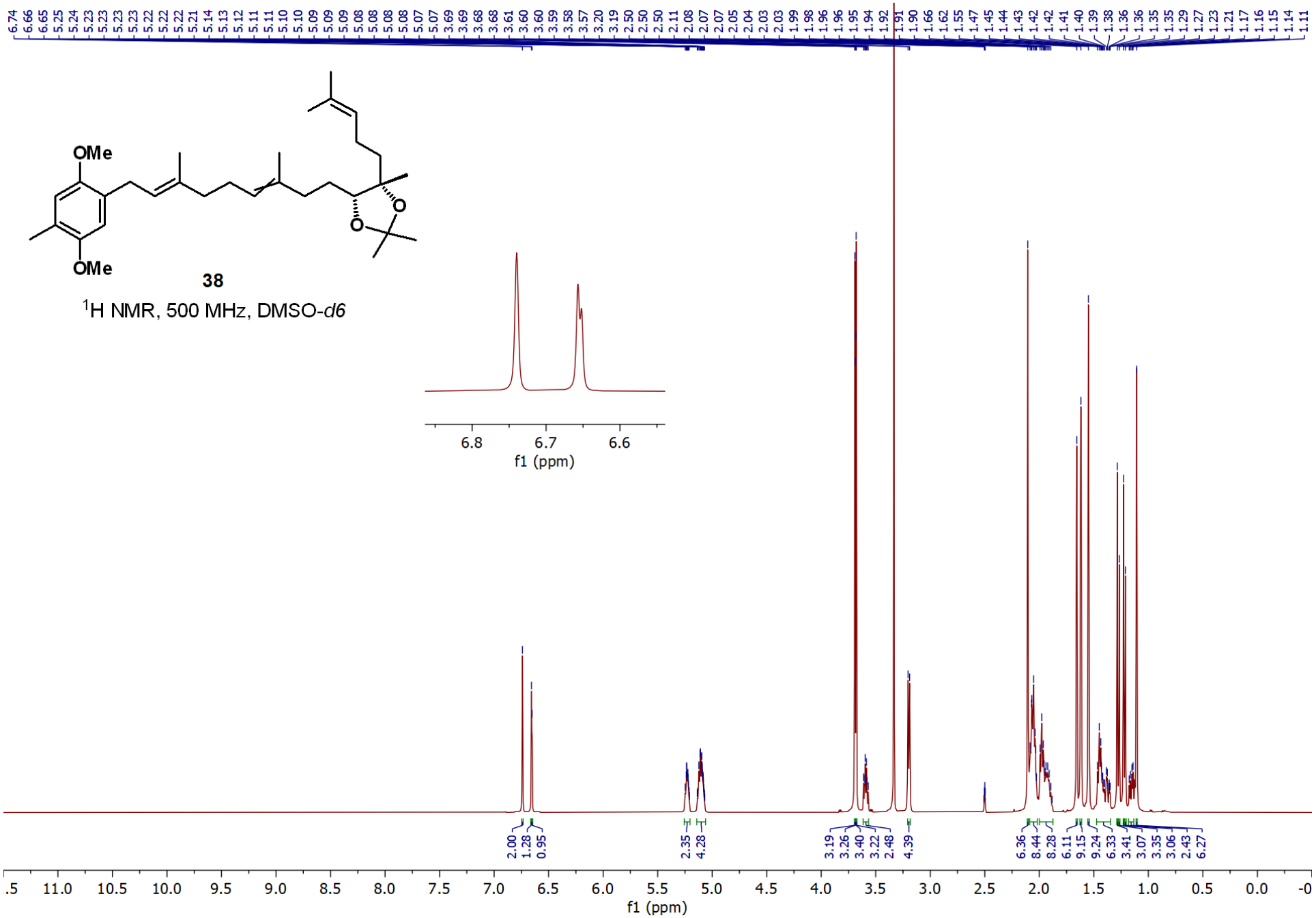

TBDPSO
OMe
OMe
O
O
36
HMBC ^{1}H-^{13}C NMR, 500-126 MHz, DMSO-*d6*
f2 (ppm)
f1 (ppm)

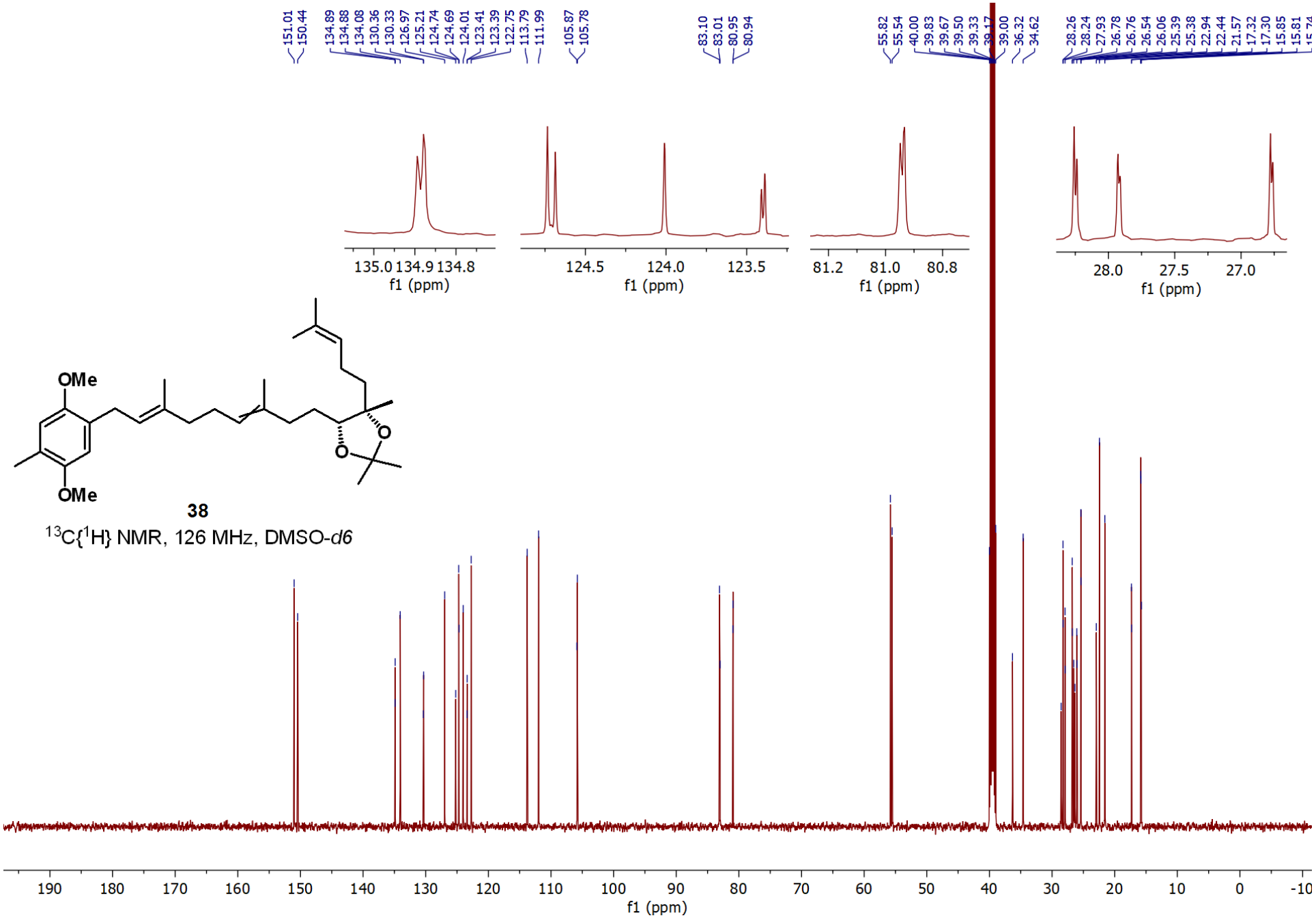
37

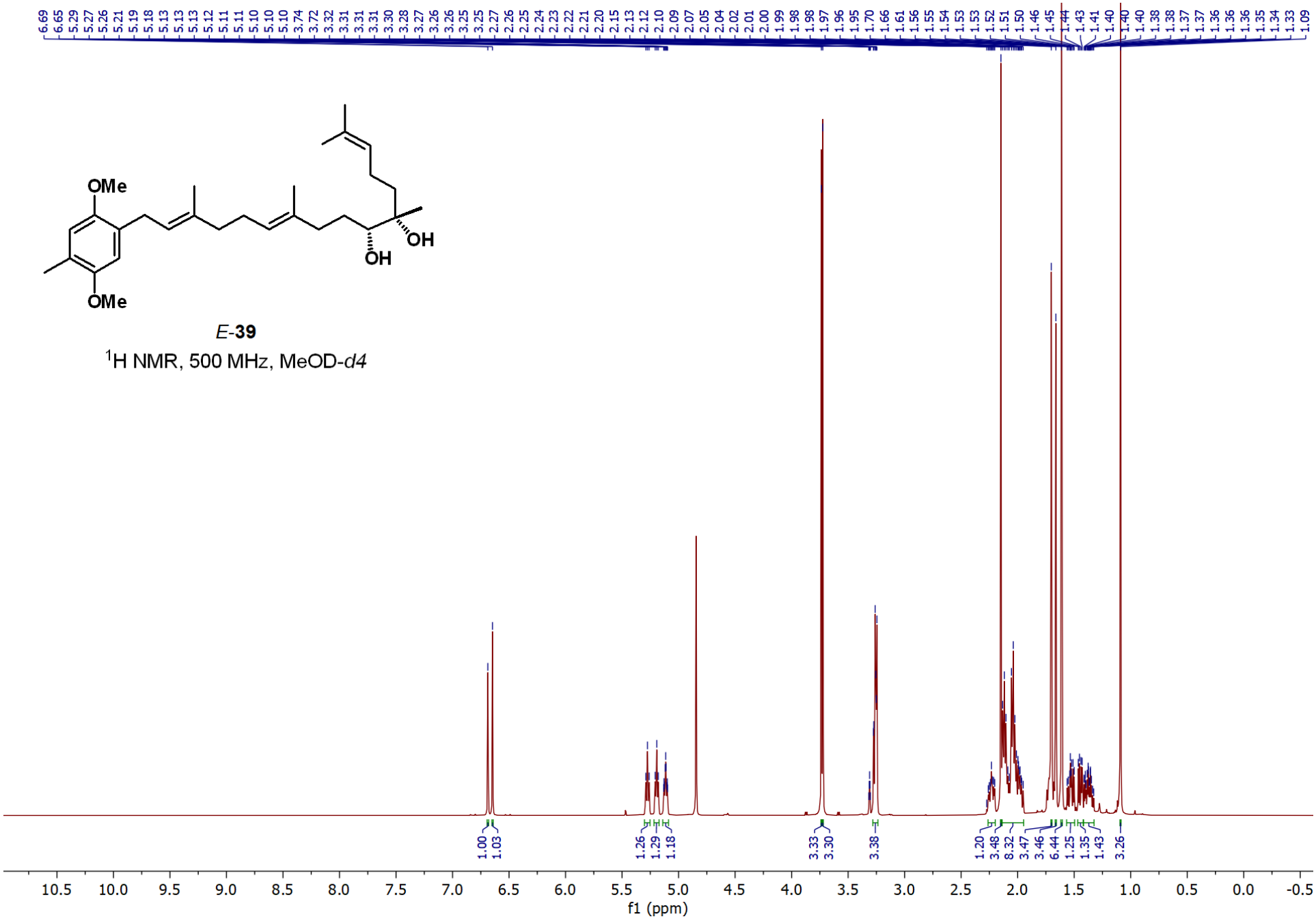
^{1}H NMR, 500 MHz, DMSO-*d6*




151.03
150.45
134.94
134.14
134.12
127.00
125.24
124.05
123.43
123.41
122.76
113.85
112.05
105.87
105.78
83.09
82.99
81.08
81.06
61.32
55.88
55.59
40.00
39.83
39.67
39.50
39.33
39.17
39.00
36.36
30.79
28.59
28.25
28.24
27.95
26.89
26.86
26.60
26.53
26.38
26.09
26.00
22.98
22.60
15.89
15.86
15.78
134.5
134.0
f1 (ppm)
123.6
123.4
123.2
f1 (ppm)
28.6
28.4
28.2
28.0
f1 (ppm)
16.1
16.0
15.9
15.8
15.7
f1 (ppm)
HO
OMe
OMe
O
O
37
13C{1H} NMR, 126 MHz, DMSO-d6
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

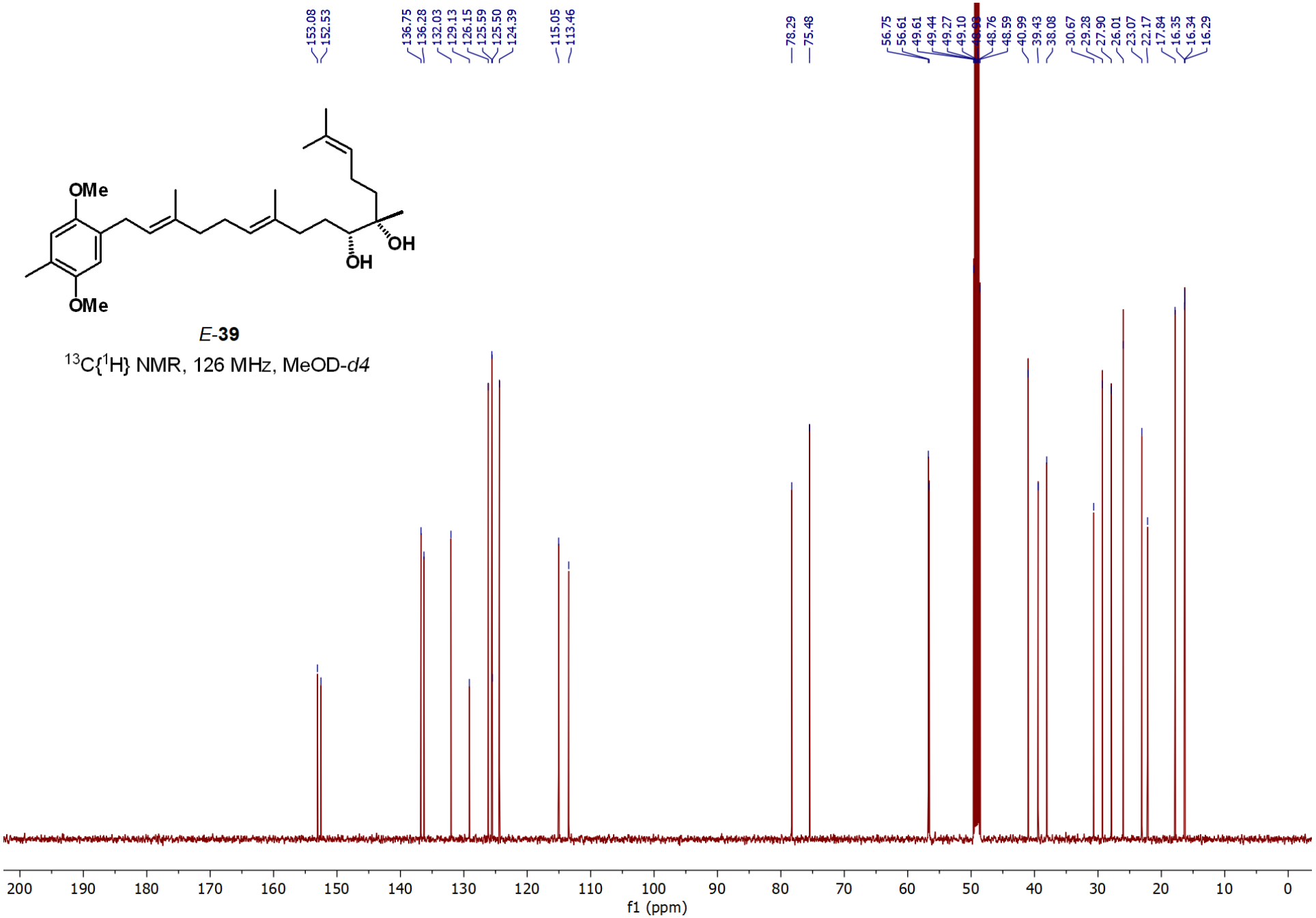

OMe
OMe
O
O
38
^{1}H NMR, 500 MHz, DMSO-*d6*
6.8
6.7
6.6
f1 (ppm)
2.00
1.28
0.95
2.35
4.28
3.19
3.26
3.40
3.22
2.48
4.39
6.36
8.44
8.28
6.11
9.15
9.24
6.33
3.41
3.07
3.35
3.06
2.43
6.27
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

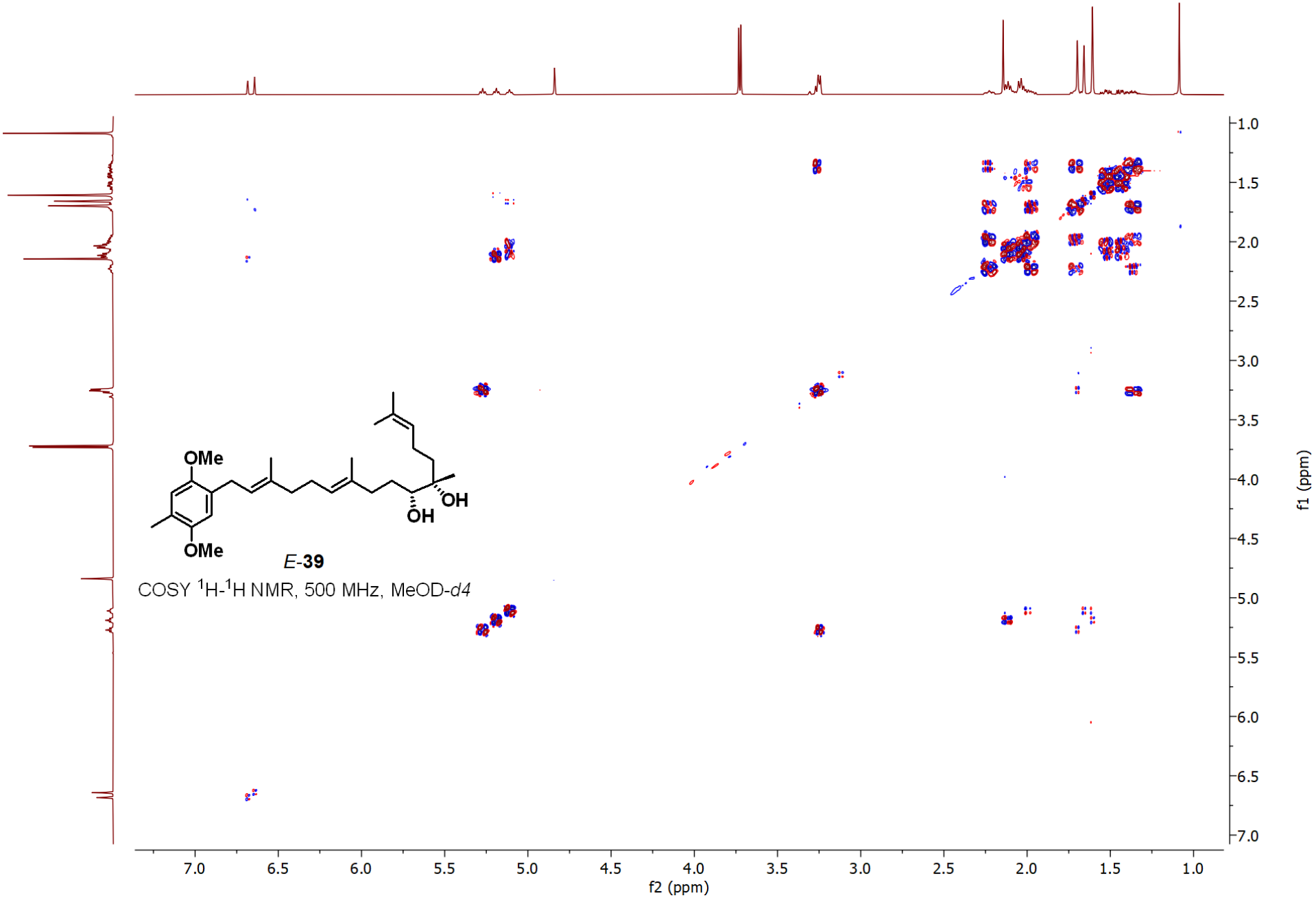

151.01
150.44
134.89
134.88
134.08
130.36
130.33
126.97
125.21
124.74
124.69
124.01
123.41
123.39
122.75
113.79
111.99
105.87
105.78
83.10
83.01
80.95
80.94
55.82
55.54
40.00
39.83
39.67
39.50
39.33
39.17
39.00
36.32
34.62
28.26
28.24
27.93
26.78
26.76
26.54
26.06
25.39
25.38
22.94
22.44
21.57
17.32
17.30
15.85
15.81
135.0 134.9 134.8
f1 (ppm)
124.5
124.0
123.5
f1 (ppm)
81.2
81.0
80.8
f1 (ppm)
28.0
27.5
27.0
f1 (ppm)
OMe
OMe
O
O
38
$^{13}C\{^{1}H\}$ NMR, 126 MHz, DMSO-*d6*
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)



E-**39**

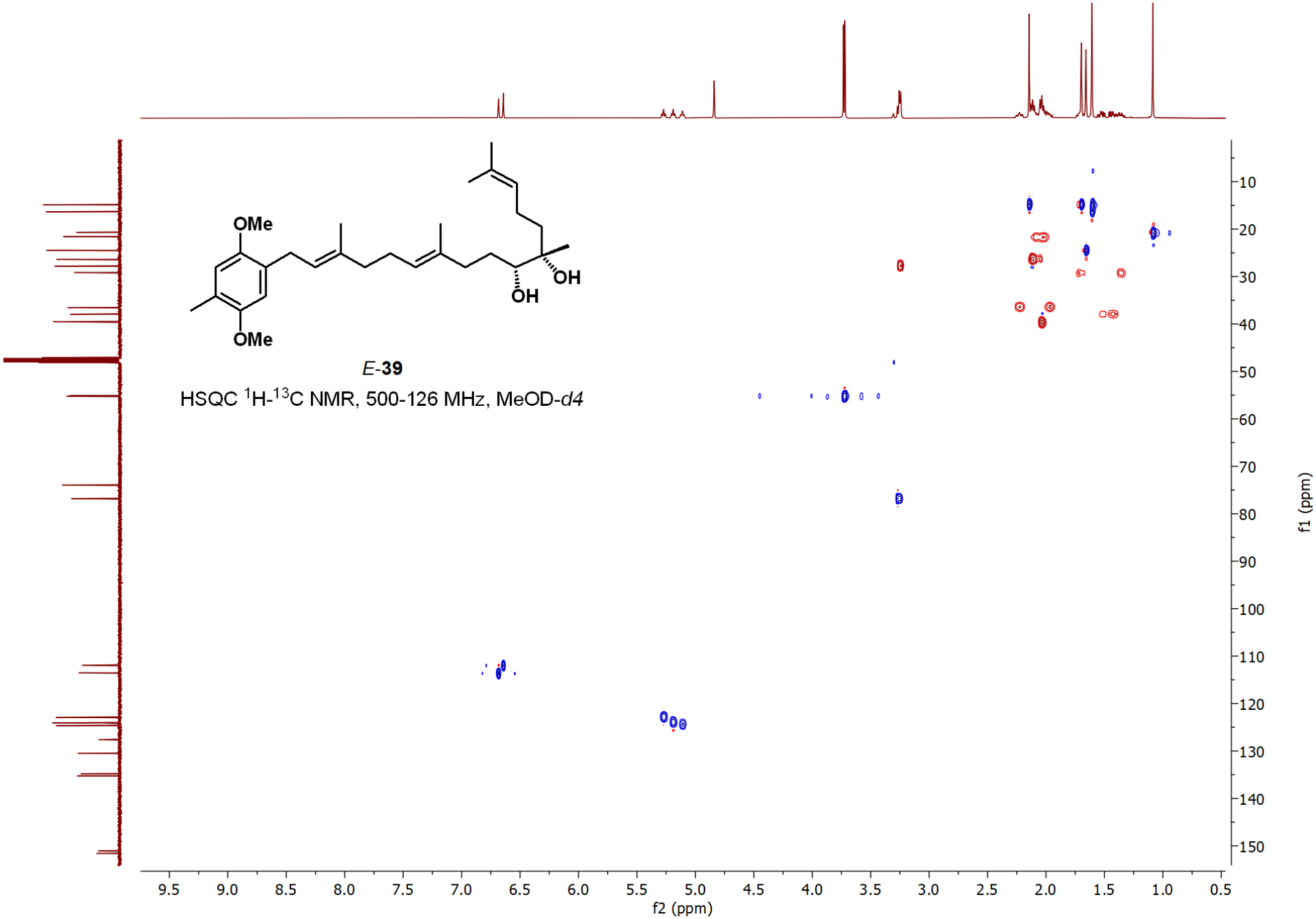
^{1}H NMR, 500 MHz, MeOD-*d4*


153.08
152.53
136.75
136.28
132.03
129.13
126.15
125.59
125.50
124.39
115.05
113.46
78.29
75.48
56.75
56.61
49.61
49.44
49.27
49.10
48.93
48.76
48.59
40.99
39.43
38.08
30.67
29.28
27.90
26.01
23.07
22.17
17.84
16.35
16.34
16.29
OMe
OMe
OH
OH
E-39
13C{1H} NMR, 126 MHz, MeOD-d4
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



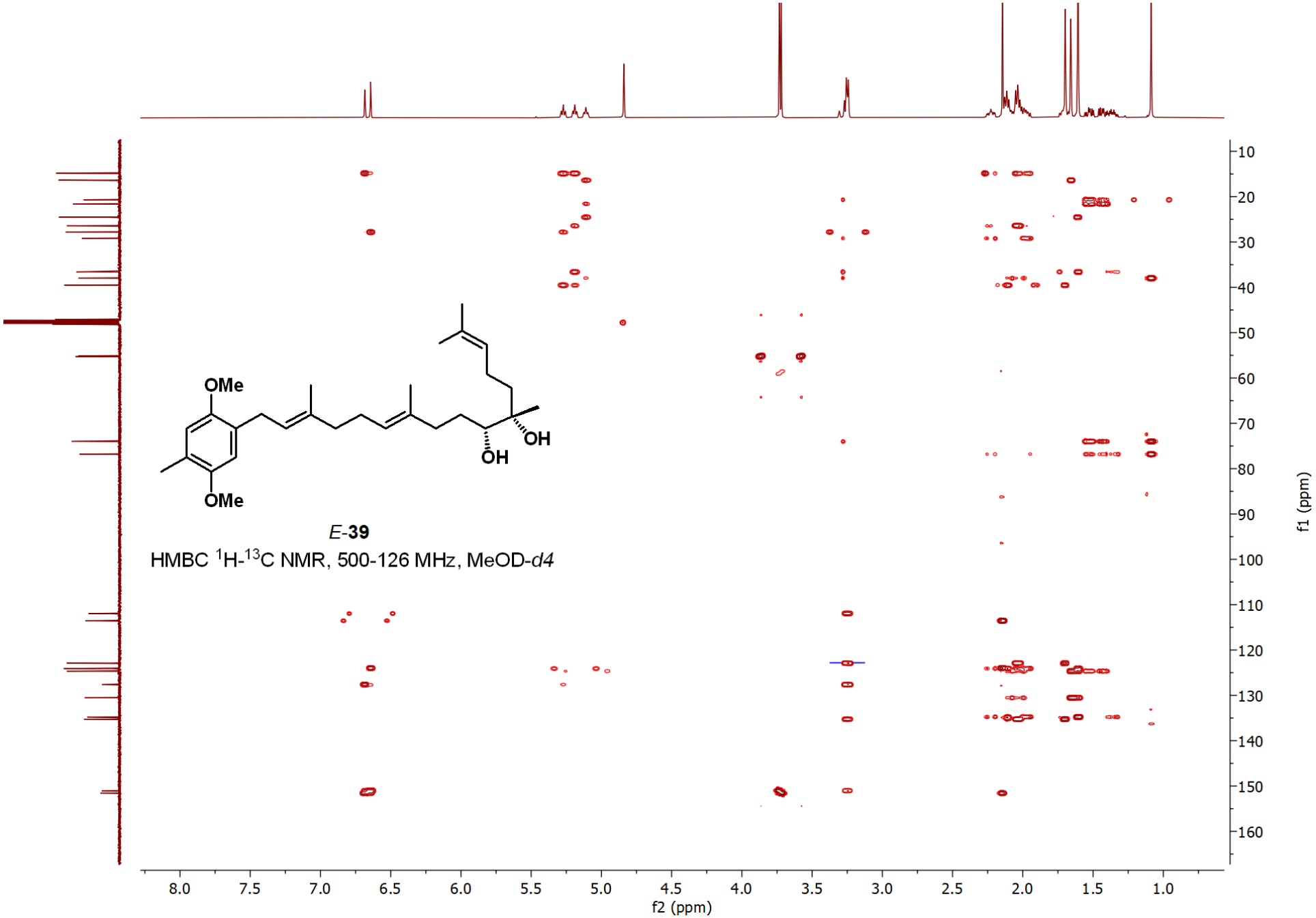
E-**39**

COSY ^{1}H-^{1}H NMR, 500 MHz, MeOD-*d4*



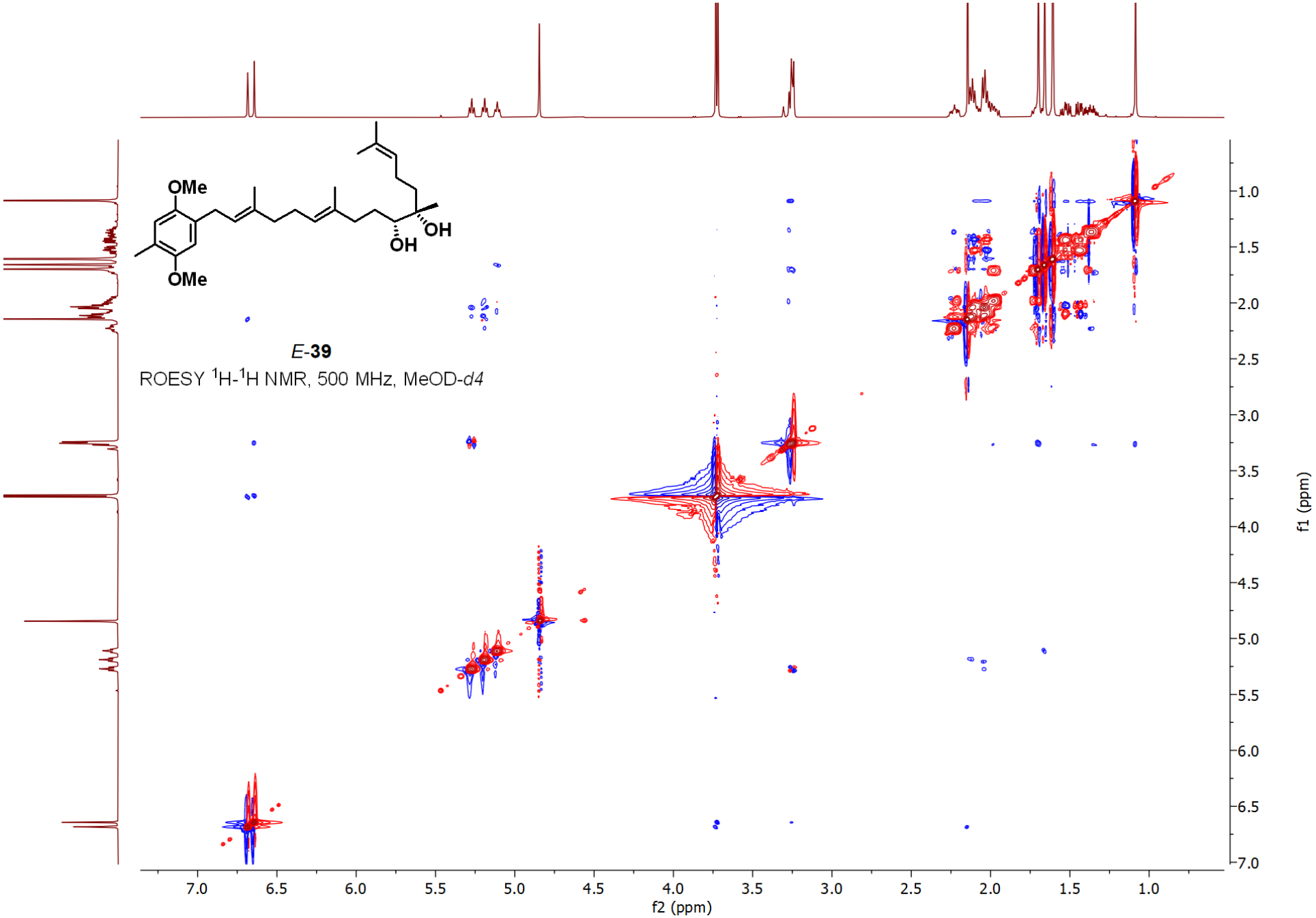
E-**39**

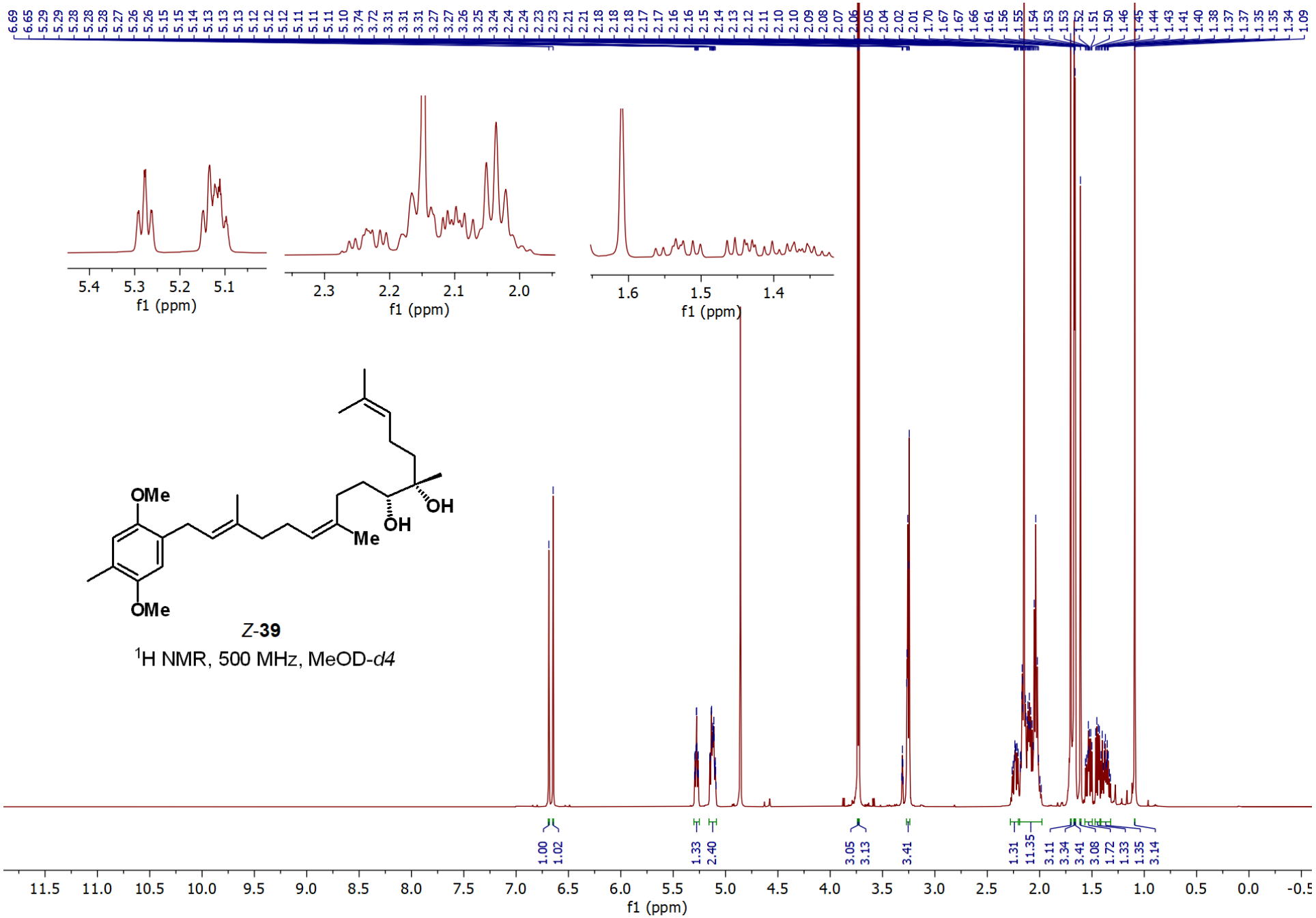
HSQC ^{1}H-^{13}C NMR, 500-126 MHz, MeOD-*d4*



E-**39**

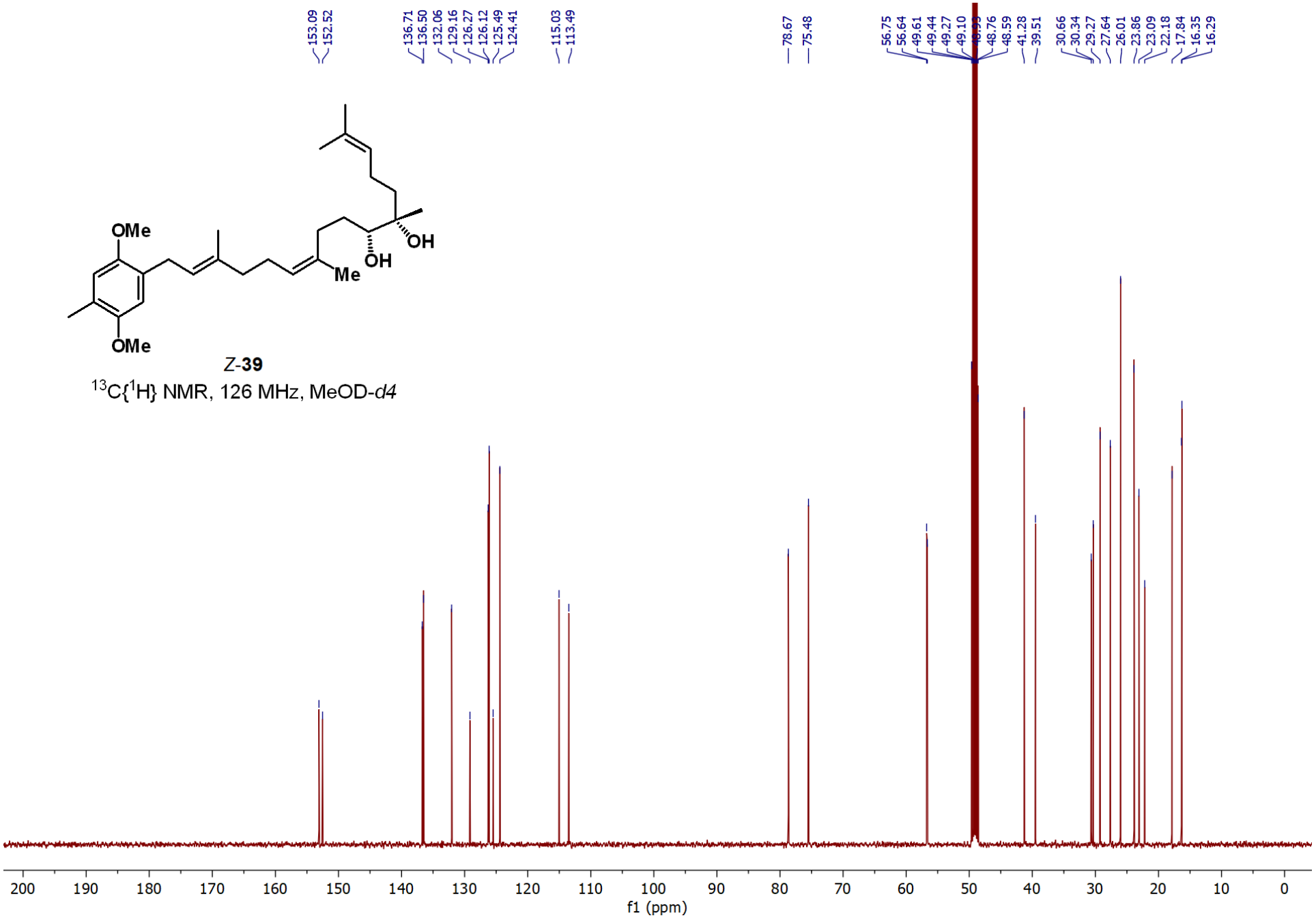
HMBC ^{1}H-^{13}C NMR, 500-126 MHz, MeOD-*d4*


OMe
OMe
OH
OH
E-**39**
ROESY ^{1}H-^{1}H NMR, 500 MHz, MeOD-*d4*
f2 (ppm)
f1 (ppm)



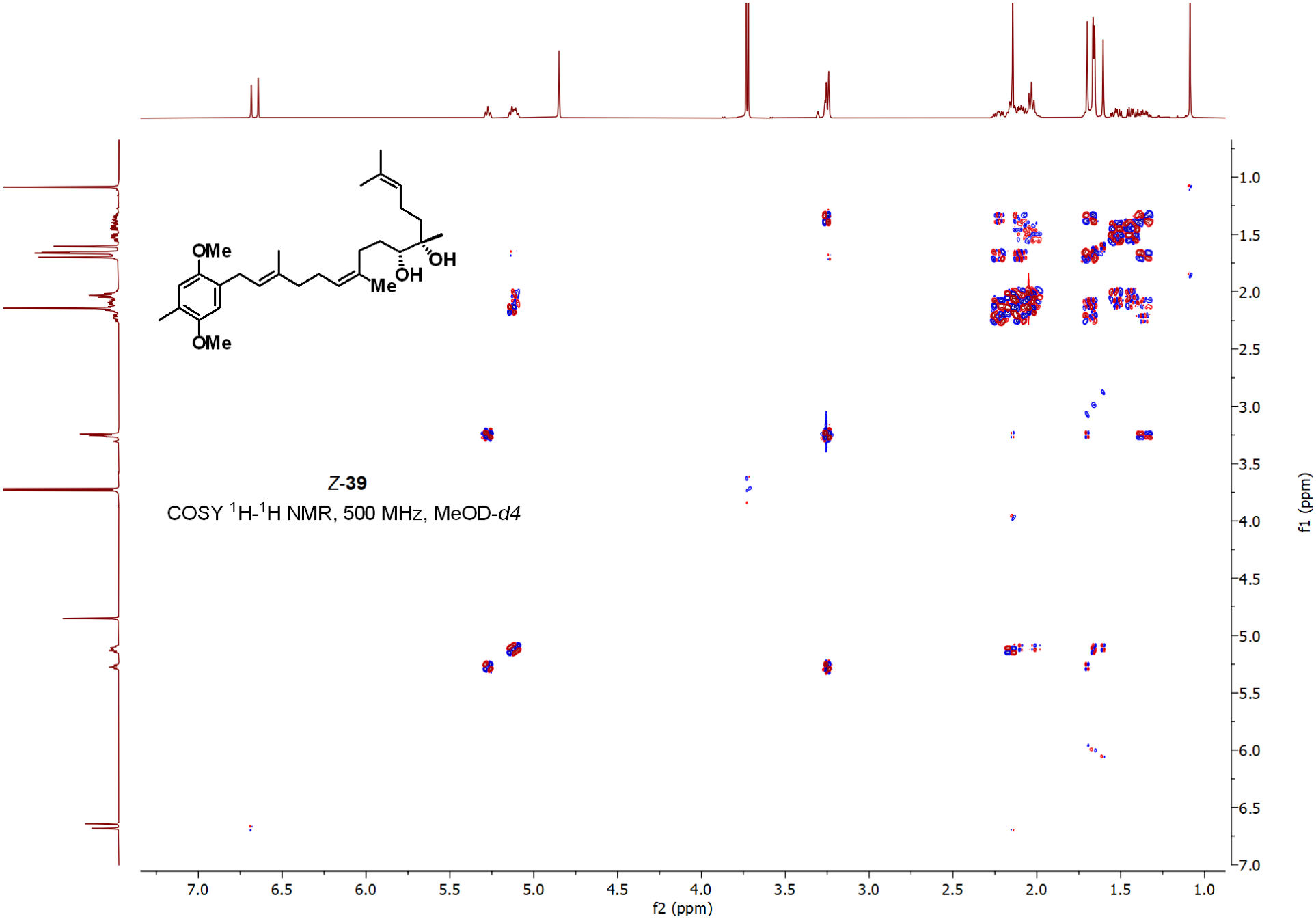
Z-**39**

^{1}H NMR, 500 MHz, MeOD-*d4*



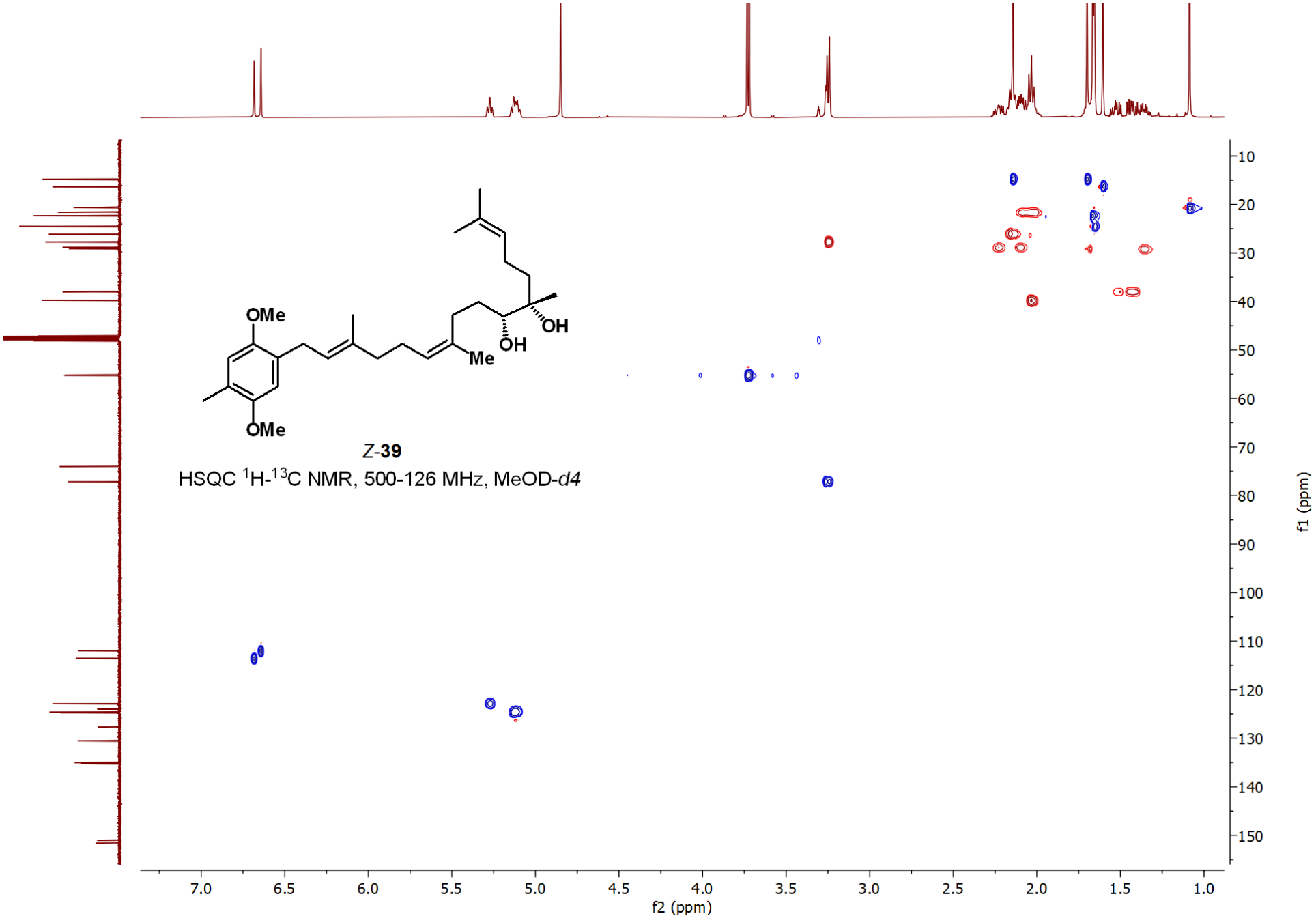
Z-**39**

$^{13}C\{^{1}H\}$ NMR, 126 MHz, MeOD-*d4*



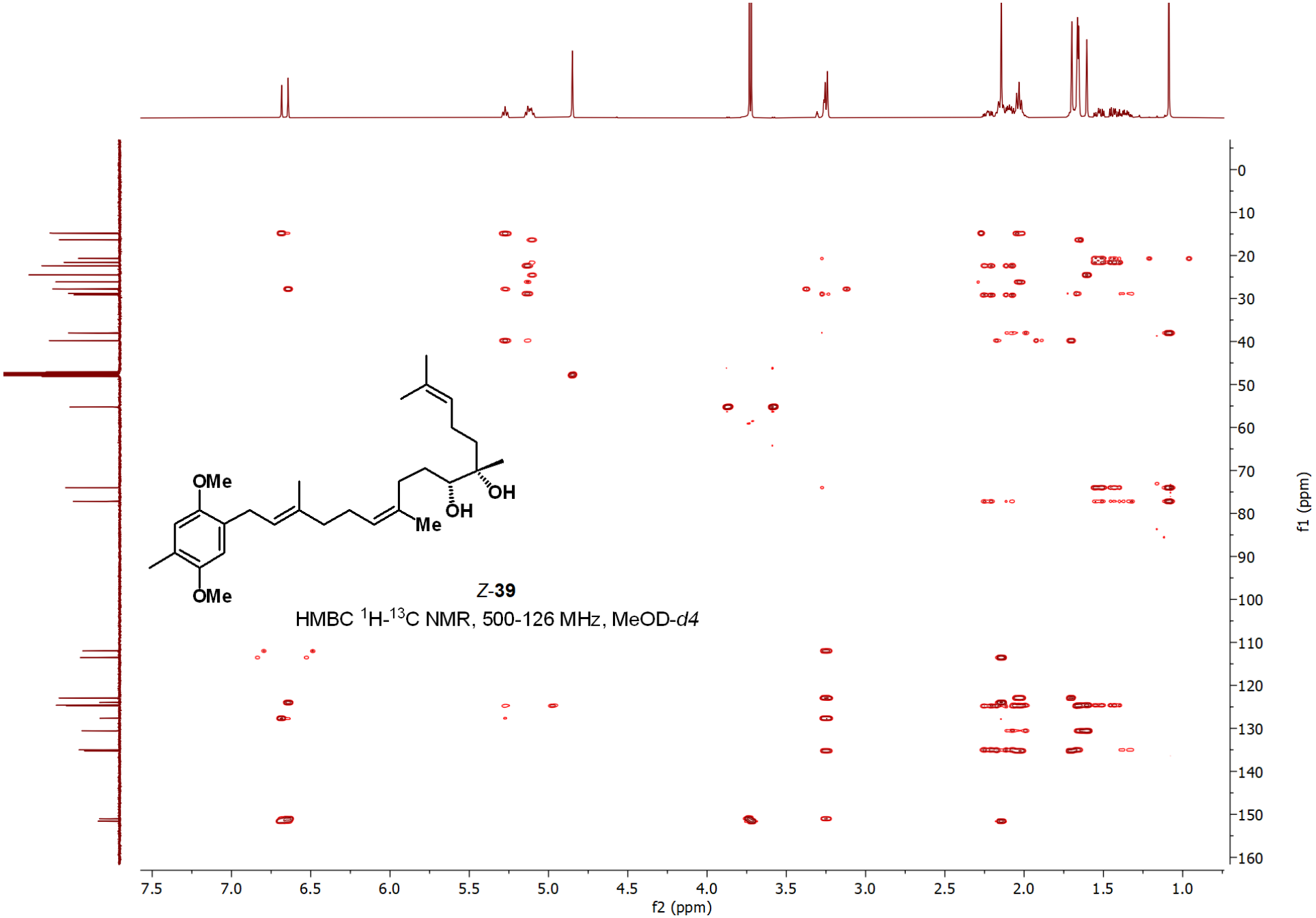
Z-**39**

COSY ^{1}H-^{1}H NMR, 500 MHz, MeOD-*d4*



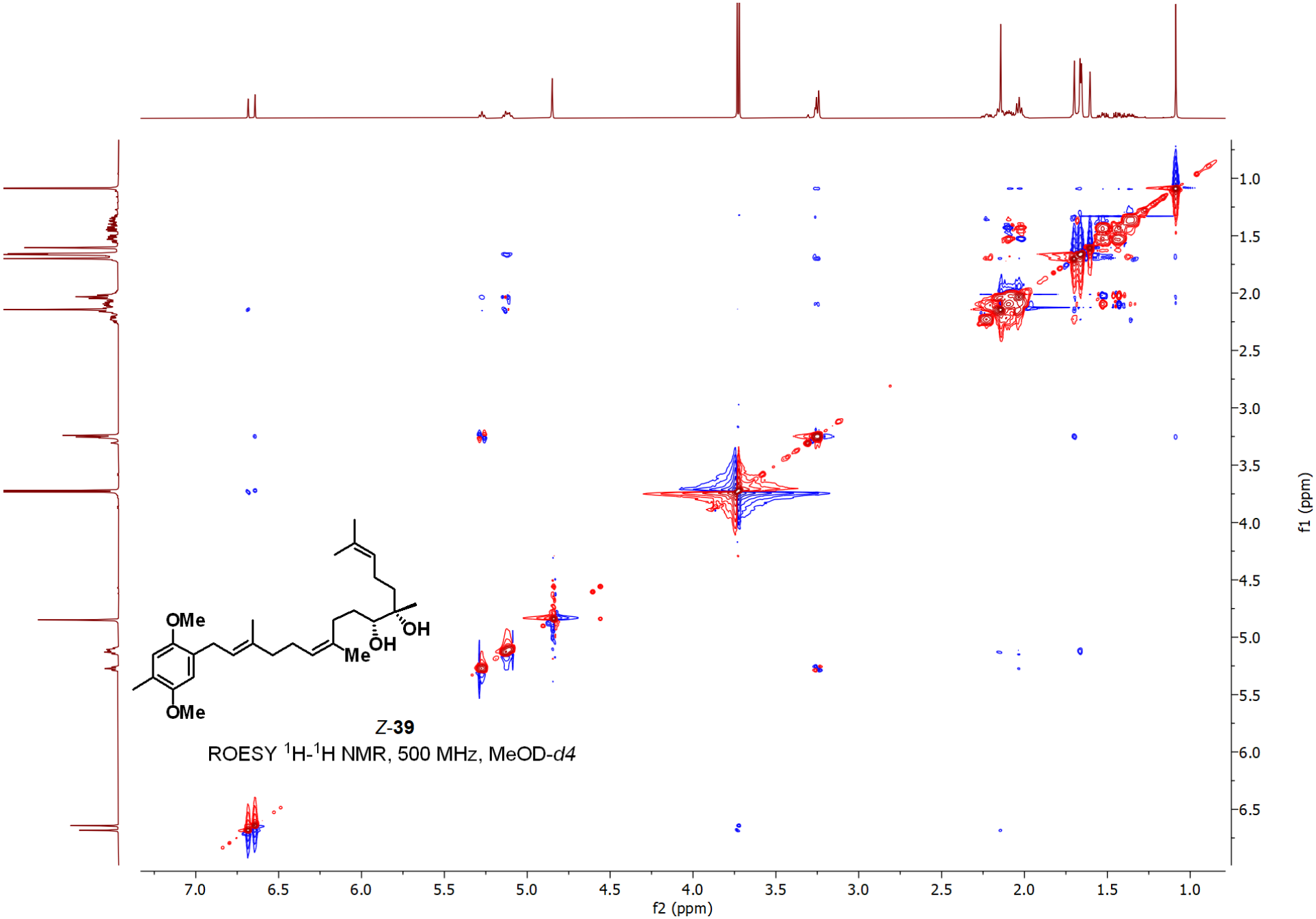
Z-**39**

HSQC ^{1}H-^{13}C NMR, 500-126 MHz, MeOD-*d4*



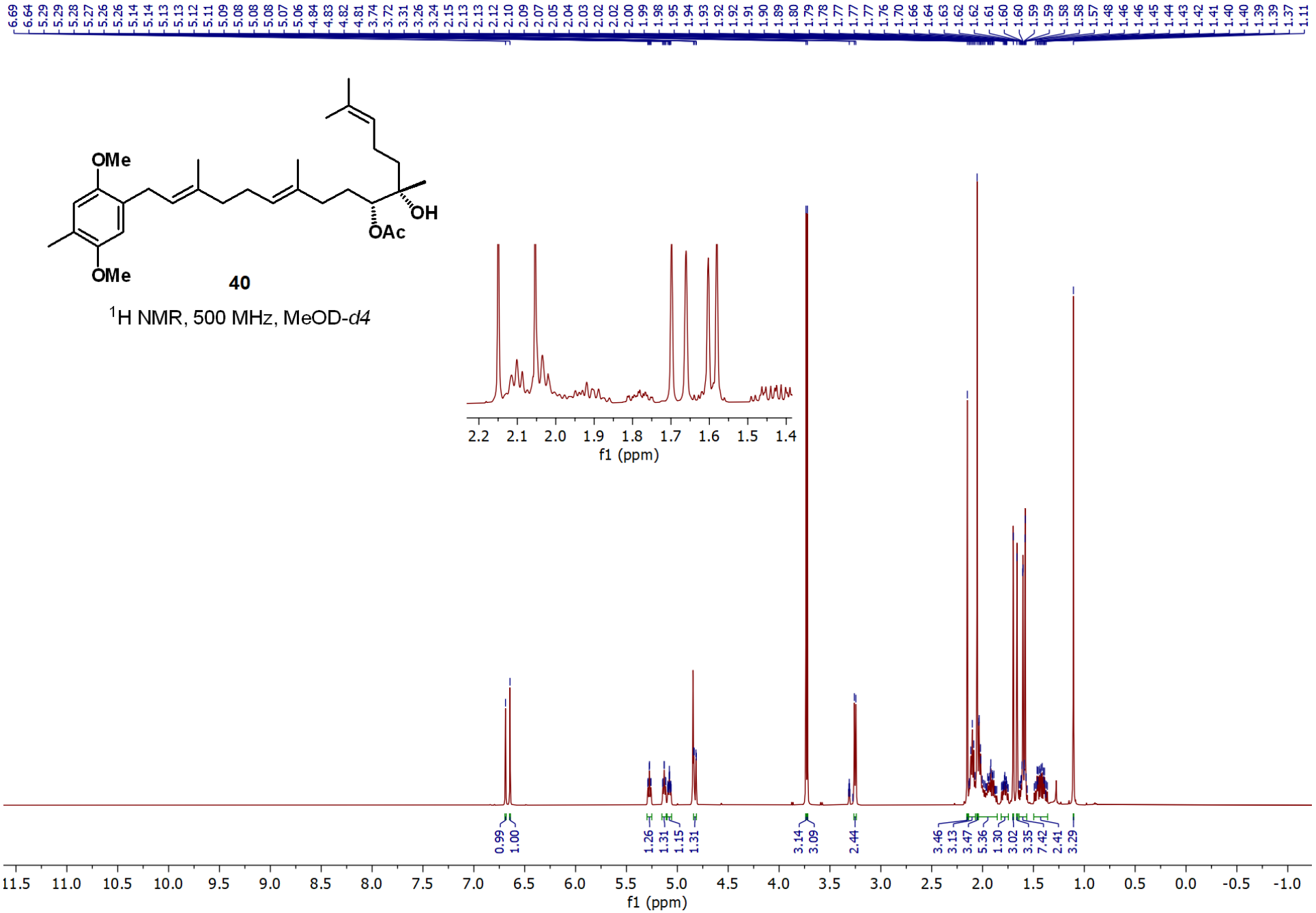
Z-**39**

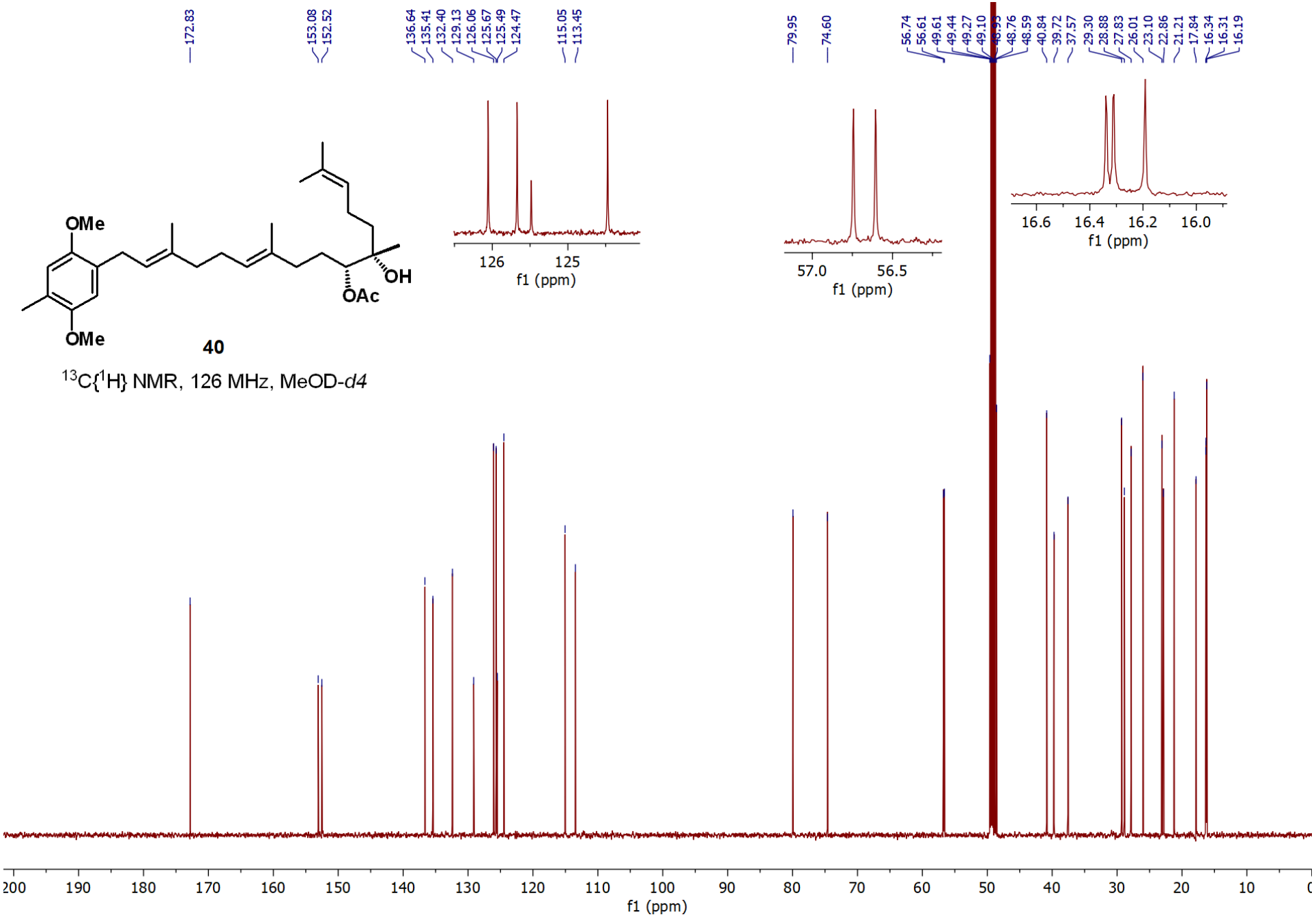
HMBC ^{1}H-^{13}C NMR, 500-126 MHz, MeOD-*d4*

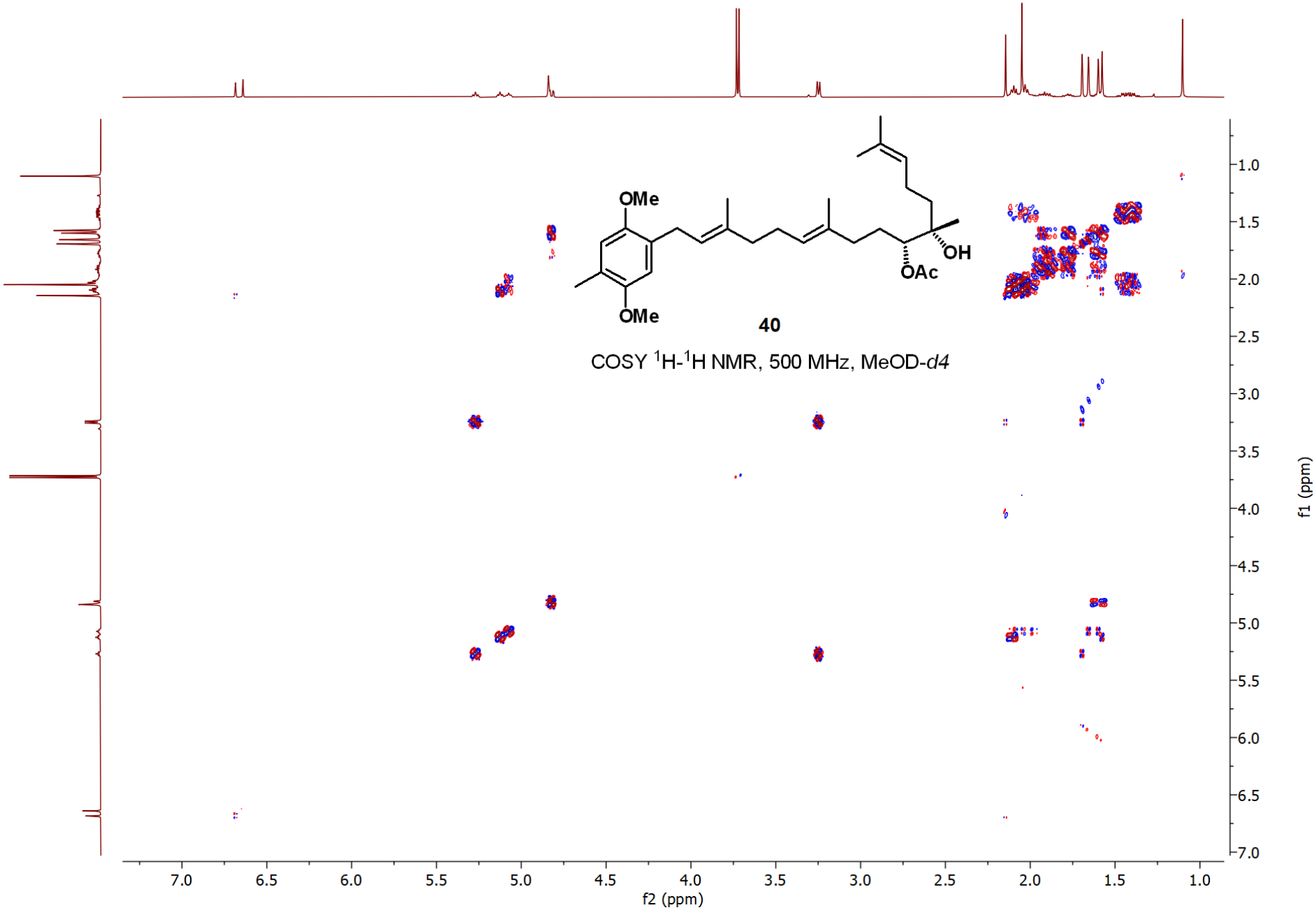


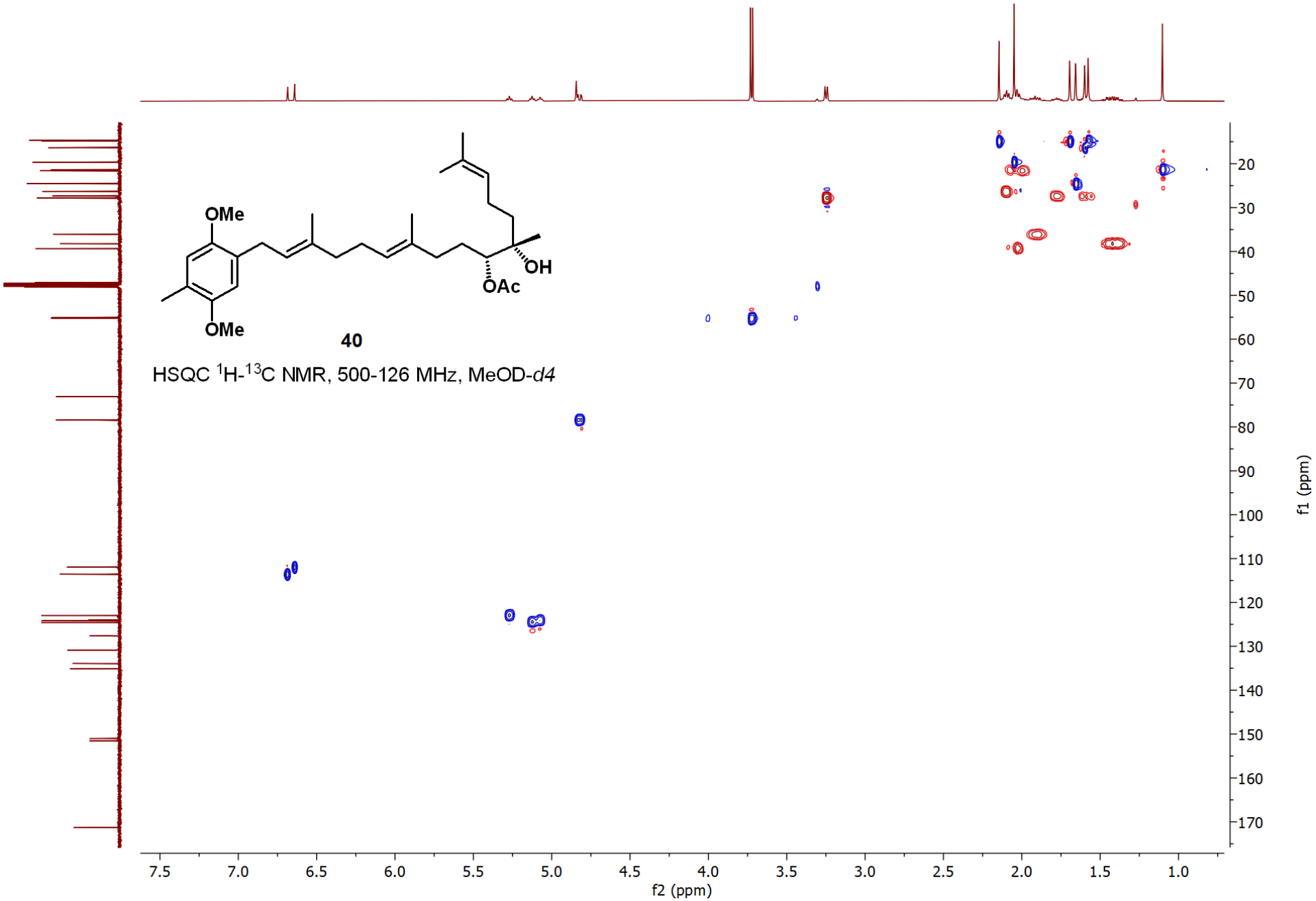
Z-**39**

ROESY ^{1}H-^{1}H NMR, 500 MHz, MeOD-*d4*

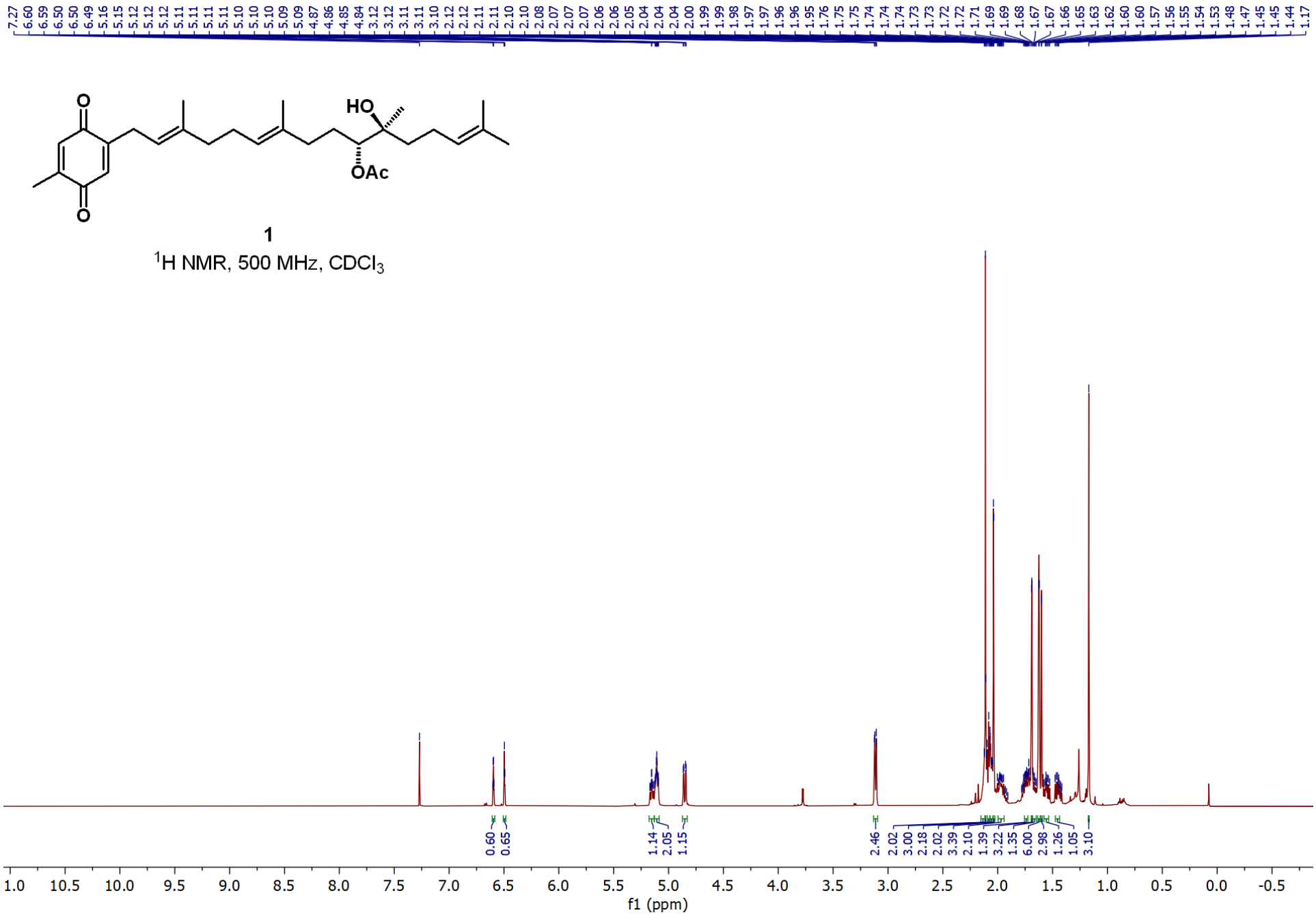

OMe
OMe
OH
OAc
40
^{1}H NMR, 500 MHz, MeOD-*d4*
f1 (ppm)

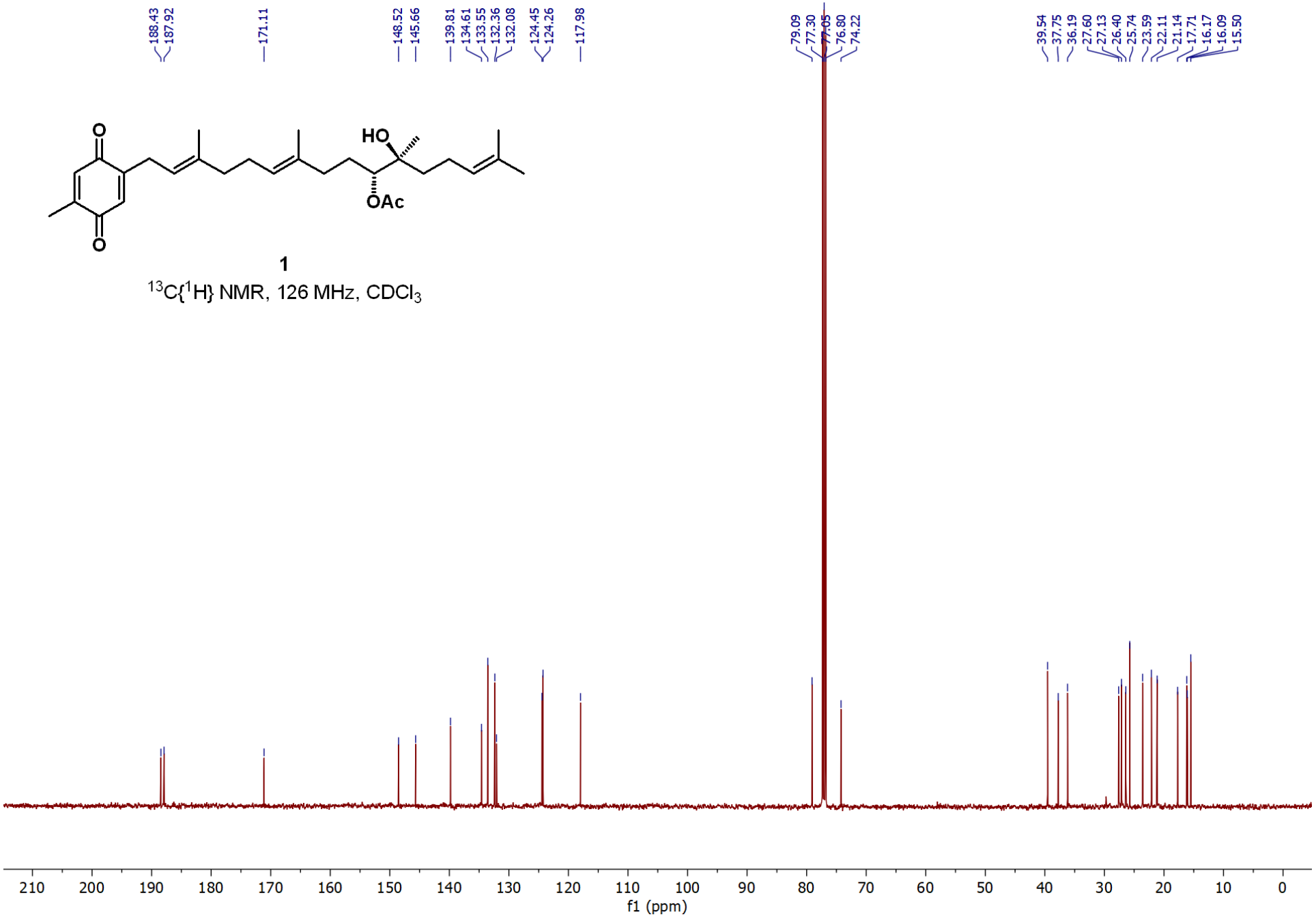

OMe
OMe
OH
OAc
40
$^{13}C\{^{1}H\}$ NMR, 126 MHz, MeOD-*d4*
172.83
153.08
152.52
136.64
135.41
132.40
129.13
126.06
125.67
125.49
124.47
115.05
113.45
79.95
74.60
56.74
56.61
49.61
49.44
49.27
49.10
48.93
48.76
48.59
40.84
39.72
37.57
29.30
28.88
27.83
26.01
23.10
22.86
21.21
17.84
16.34
16.31
16.19
f1 (ppm)

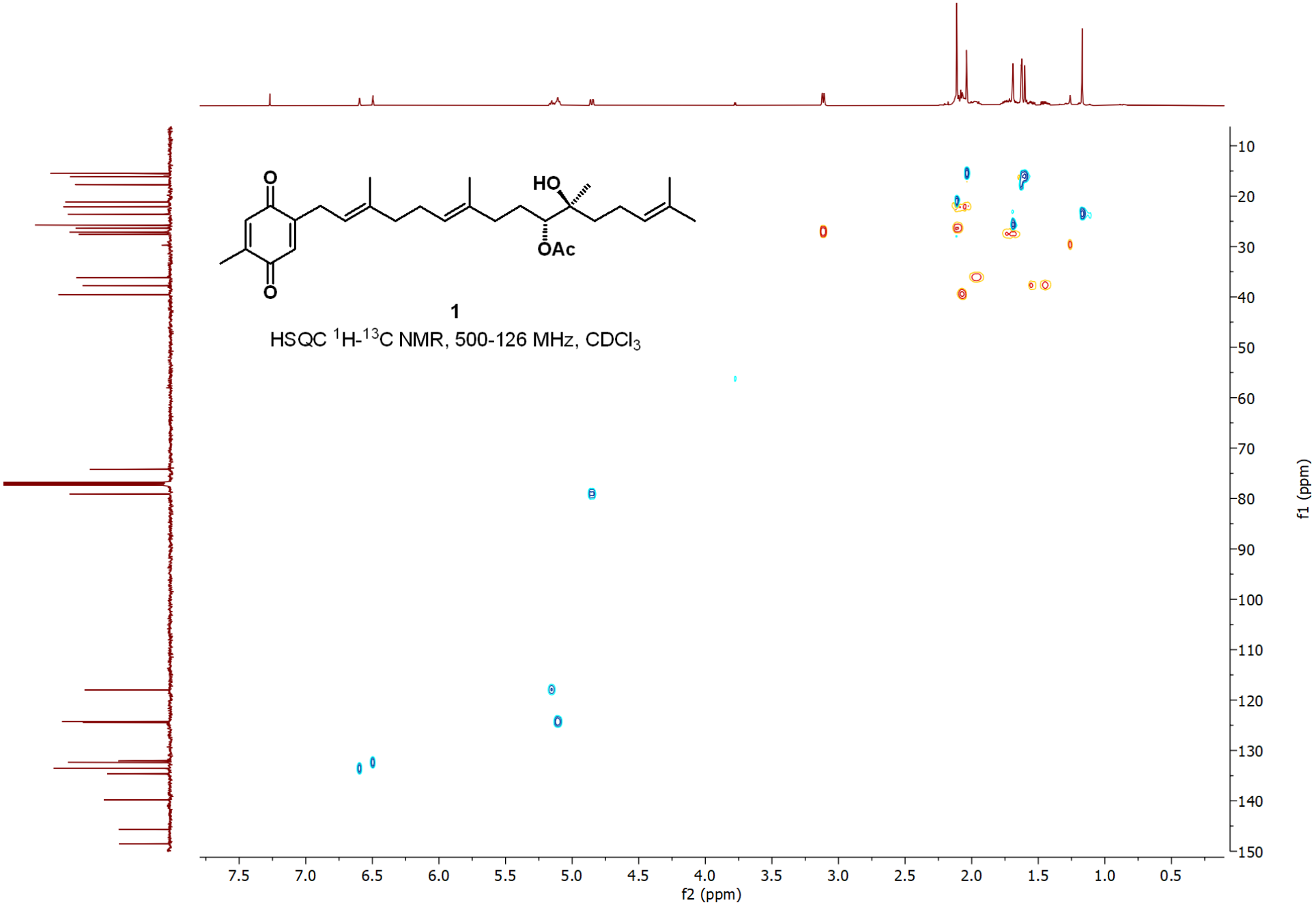

OMe
OMe
OH
OAc
40
COSY ^{1}H-^{1}H NMR, 500 MHz, MeOD-*d4*
f2 (ppm)
f1 (ppm)



HSQC ^{1}H-^{13}C NMR, 500-126 MHz, MeOD-*d4*


HO
OAc
O
O
1
^{1}H NMR, 500 MHz, $CDCl_3$
f1 (ppm)


188.43
187.92
171.11
148.52
145.66
139.81
134.61
133.55
132.36
132.08
124.45
124.26
117.98
79.09
77.30
77.05
76.80
74.22
39.54
37.75
36.19
27.60
27.13
26.40
25.74
23.59
22.11
21.14
17.71
16.17
16.09
15.50
O
O
HO
OAc
1
$^{13}C\{^{1}H\}$ NMR, 126 MHz, $CDCl_3$
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


O
HO
OAc
O
1
HSQC ^{1}H-^{13}C NMR, 500-126 MHz, $CDCl_3$
f2 (ppm)
f1 (ppm)