BIOINSPIRED SYNTHESIS OF *ILLICIUM*-DERIVED NEOLIGNANS AND THEIR NEUROLOGICAL ACTIVITY

SUPPORTING NMR SPECTRA

In order of appearance from section 7

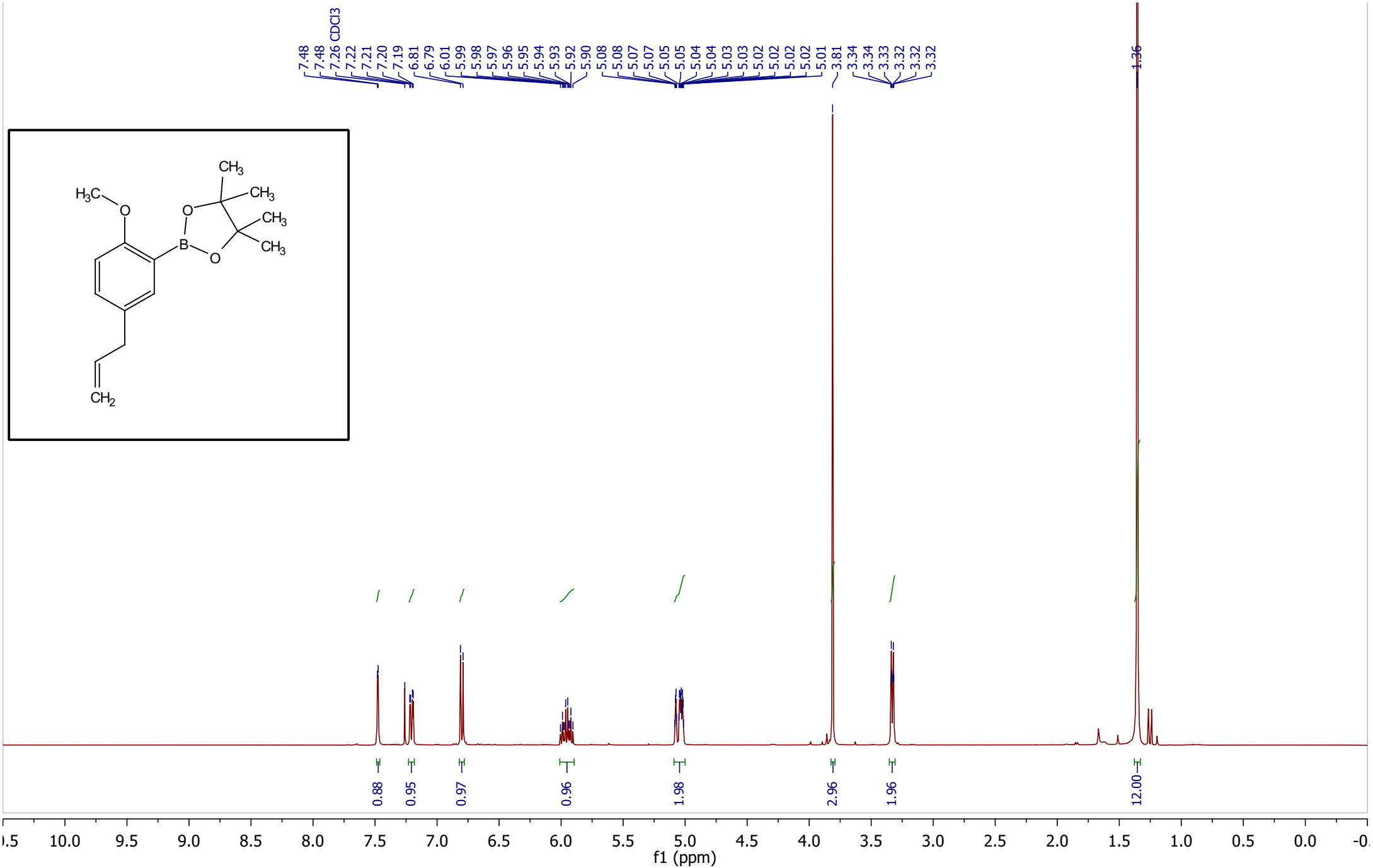
Robert Arnold, MChem

Thesis submitted to the University of Nottingham for

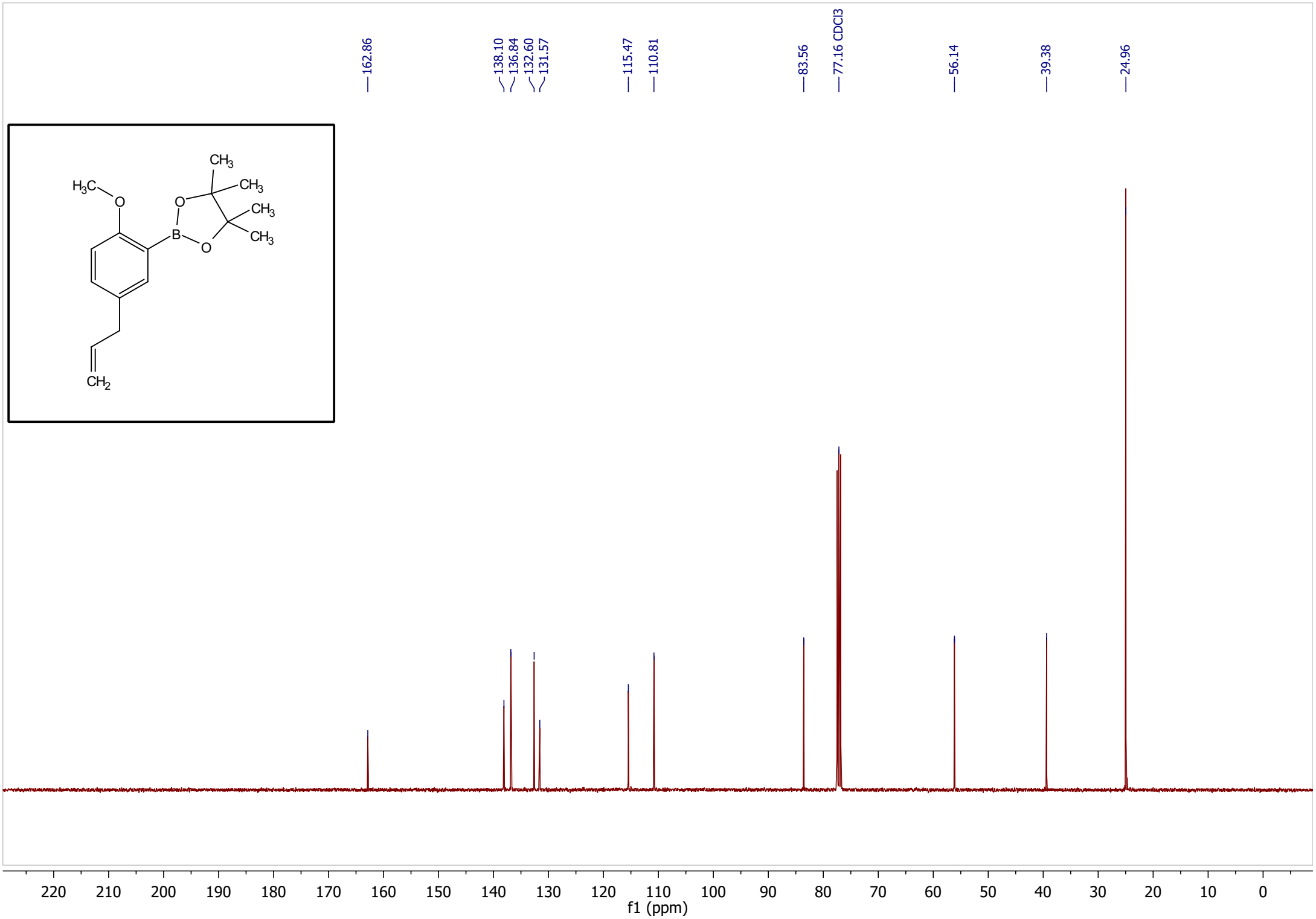
the degree of Doctor of Philosophy

JUNE 2020

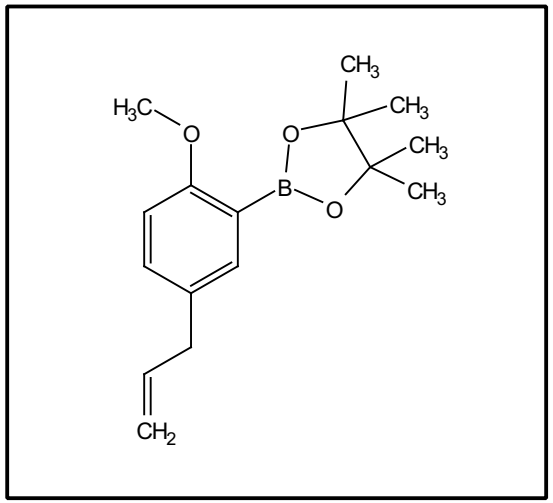
SUPERVISOR: PROF. ROSS DENTON

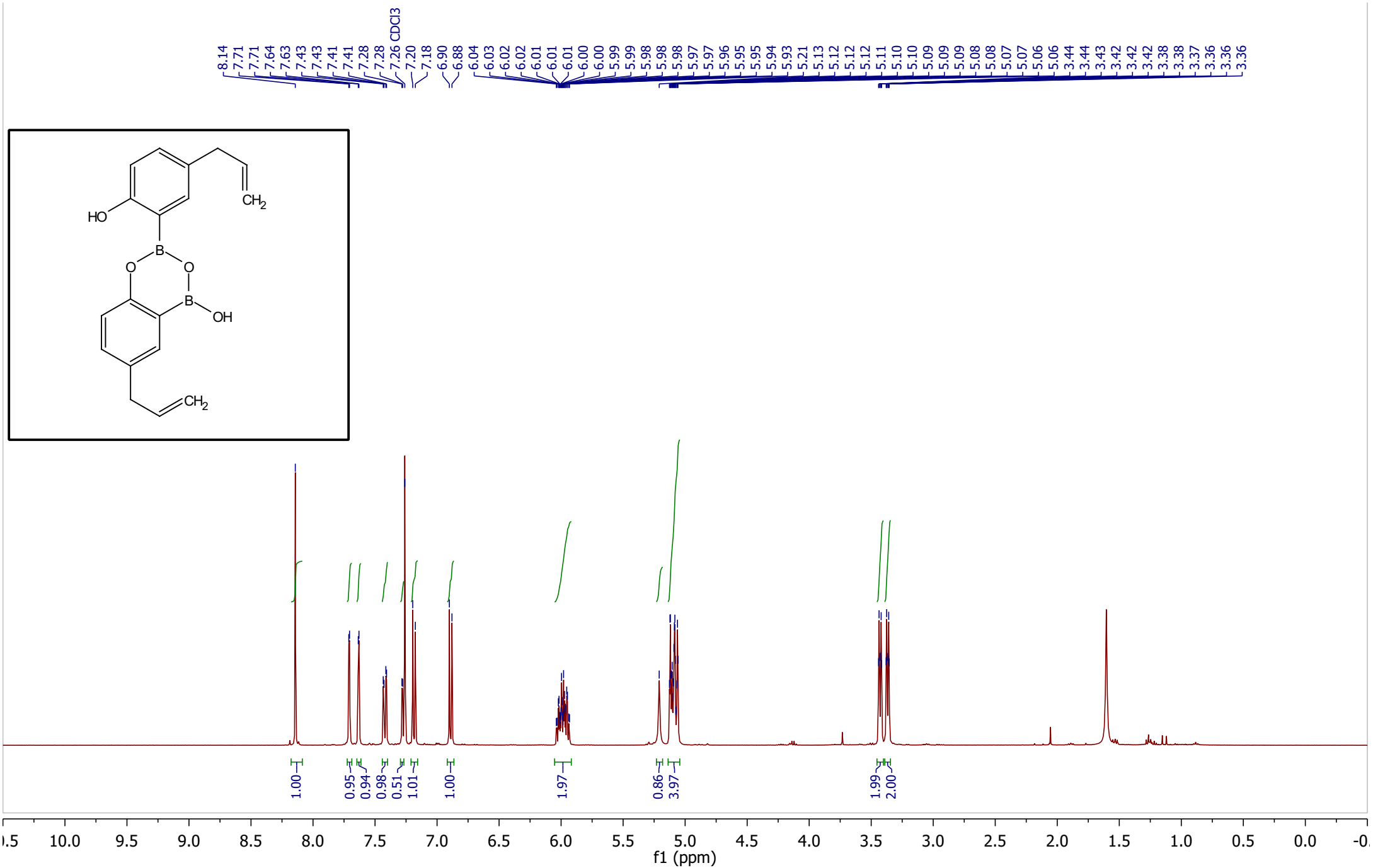


^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.48 (d, *J* = 2.4 Hz, 1H), 7.21 (dd, *J* = 8.4, 2.4 Hz, 1H), 6.80 (d, *J* = 8.4 Hz, 1H), 5.95 (ddt, *J* = 16.8, 10.1, 6.7 Hz, 1H), 5.09 – 5.00 (m, 2H), 3.81 (s, 3H), 3.33 (ddd, *J* = 6.7, 1.5, 1.5 Hz, 2H), 1.36 (s, 12H).

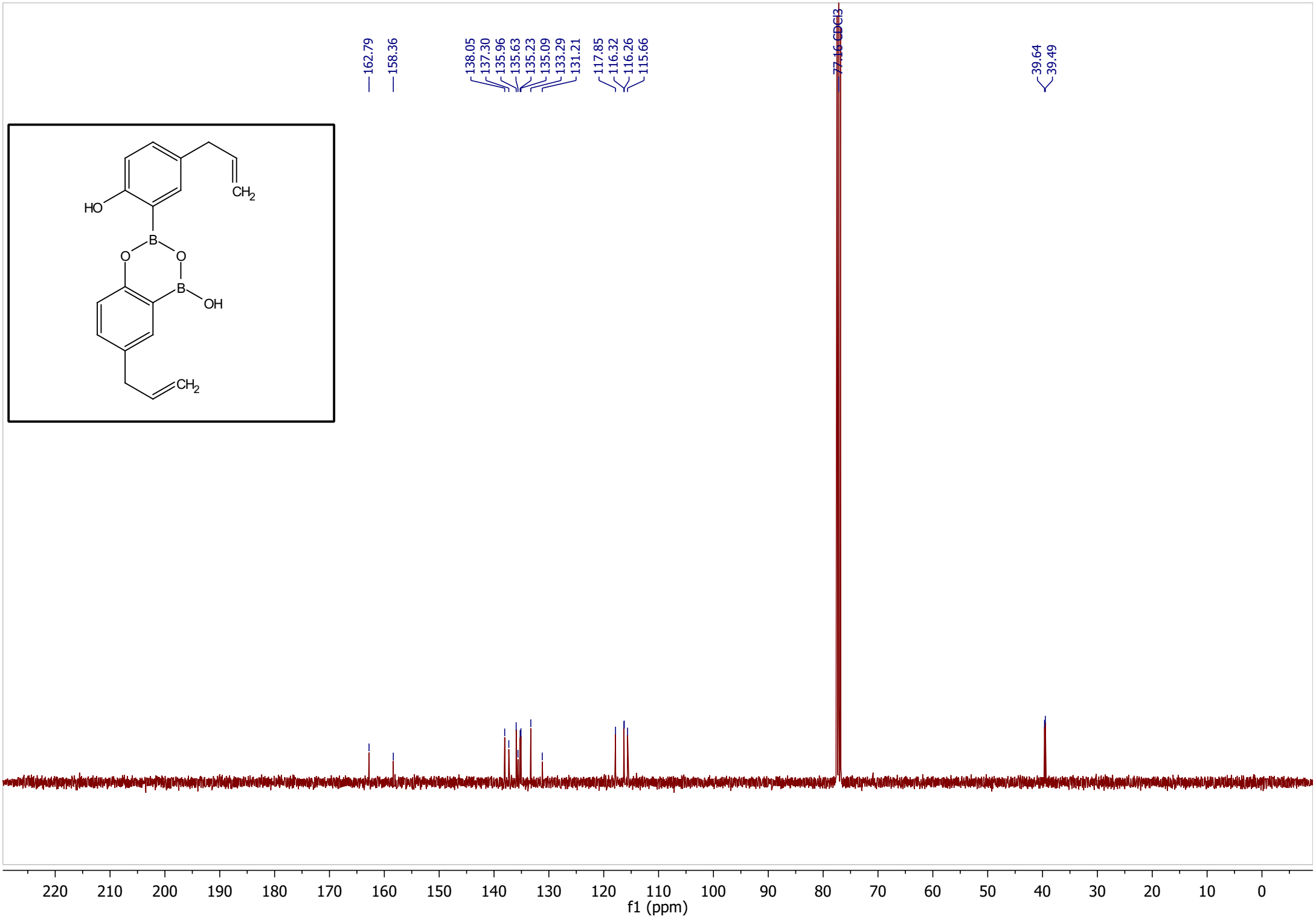

162.86
138.10
136.84
132.60
131.57
115.47
110.81
83.56
77.16 CDCl3
56.14
39.38
24.96
H3C
O
O
B
O
CH3
CH3
CH3
CH3
CH2
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

^{11}B NMR spectra

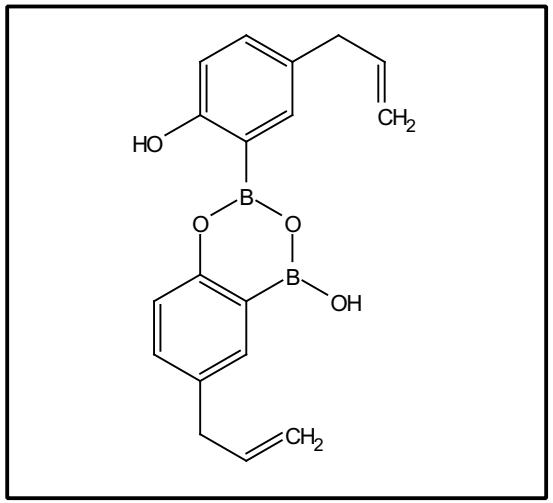


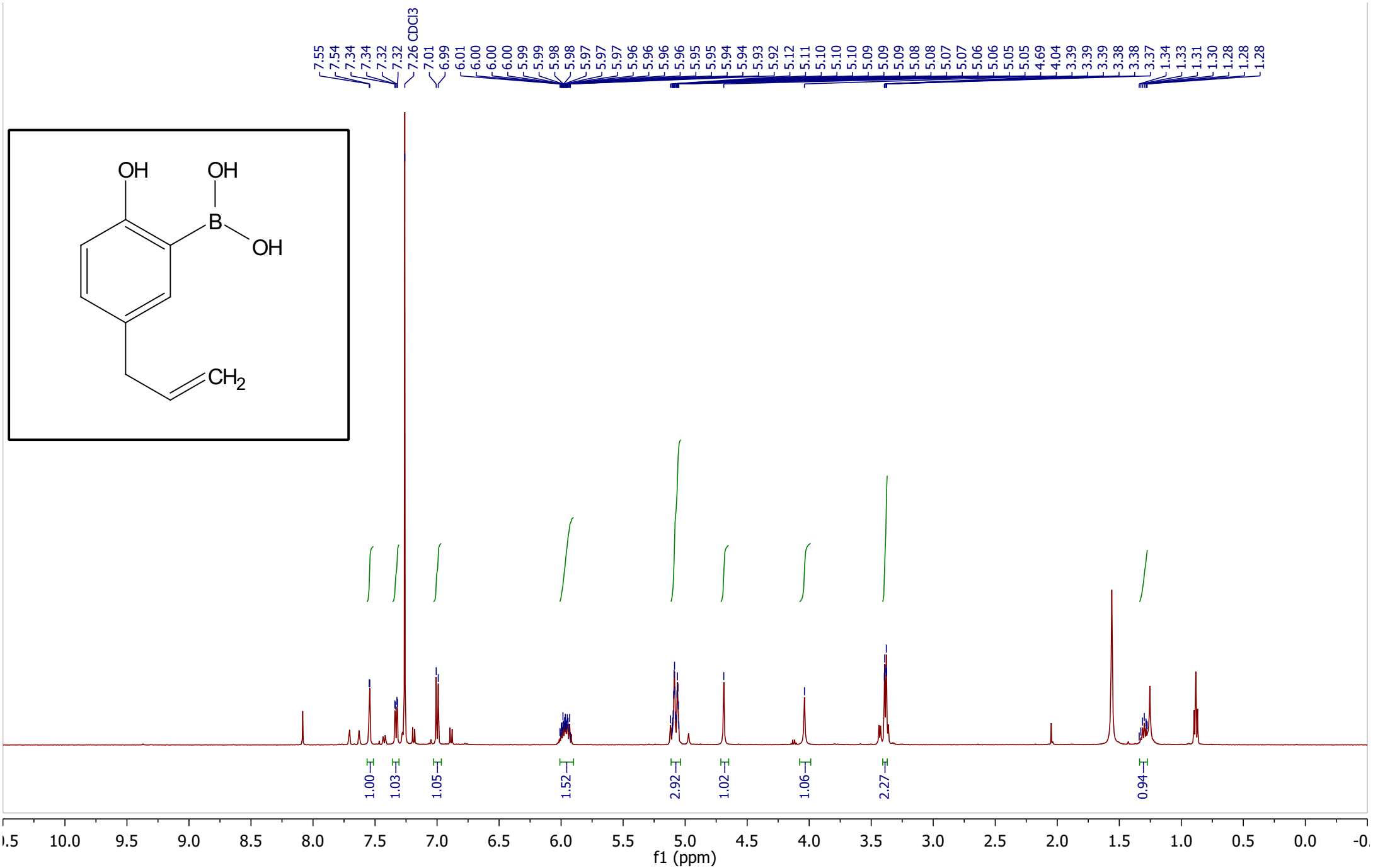


^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.14 (s, 1H), 7.71 (d, *J* = 2.4 Hz, 1H), 7.63 (d, *J* = 2.3 Hz, 1H), 7.42 (dd, *J* = 8.4, 2.3 Hz, 1H), 7.27 (dd, *J* = 8.4, 2.4 Hz, 1H), 7.19 (d, *J* = 8.4 Hz, 1H), 6.89 (d, *J* = 8.4 Hz, 1H), 6.05 – 5.91 (m, 2H), 5.21 (s, 1H), 5.16 – 5.02 (m, 4H), 3.43 (ddd, *J* = 6.6, 1.5, 1.5 Hz, 2H), 3.37 (ddd, *J* = 6.6, 1.5, 1.5 Hz, 2H).

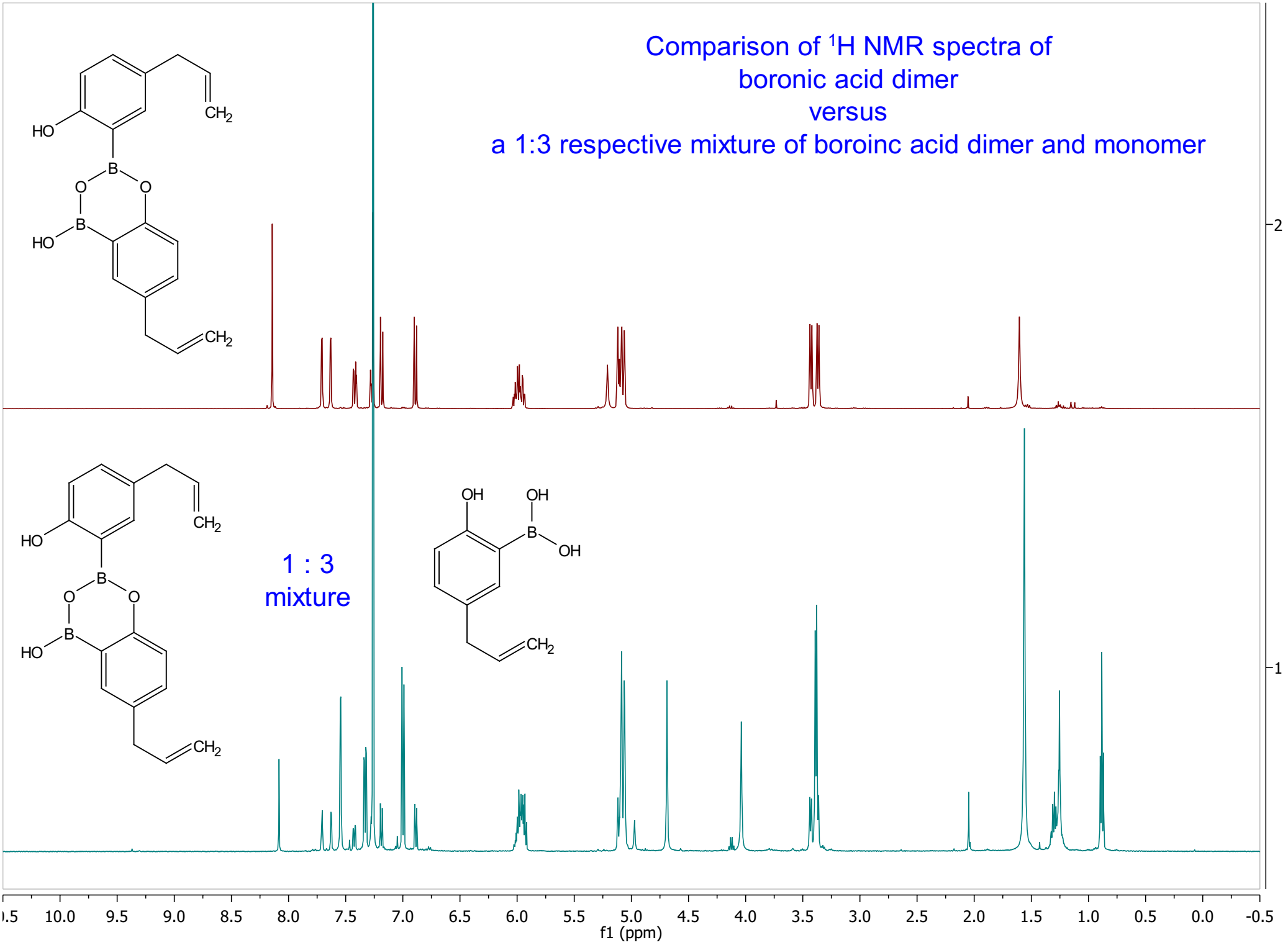

162.79
158.36
138.05
137.30
135.96
135.63
135.23
135.09
133.29
131.21
117.85
116.32
116.26
115.66
77.16 CDCl3
39.64
39.49
HO
CH2
B
O
O
B
OH
CH2
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

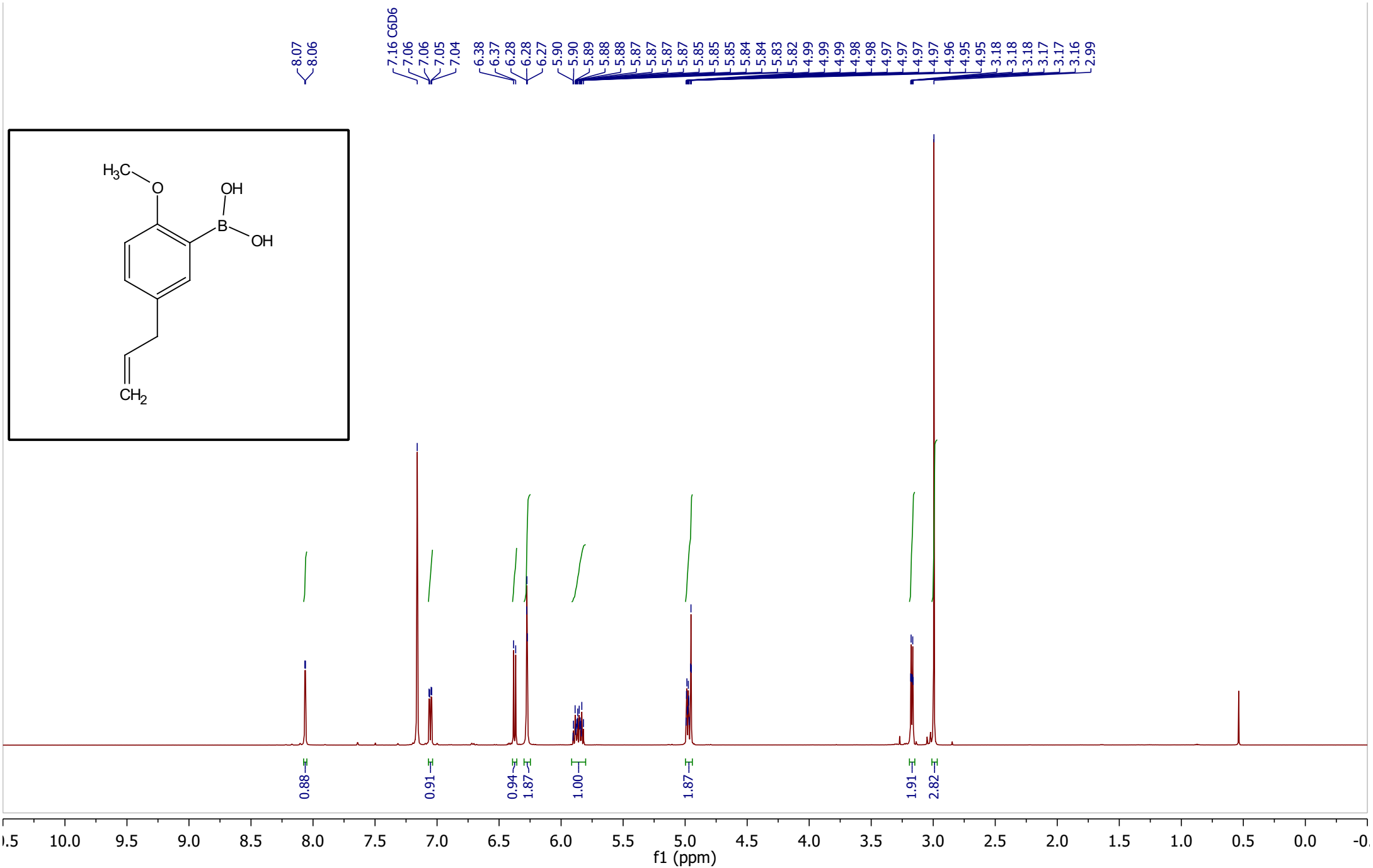
^{11}B NMR spectra



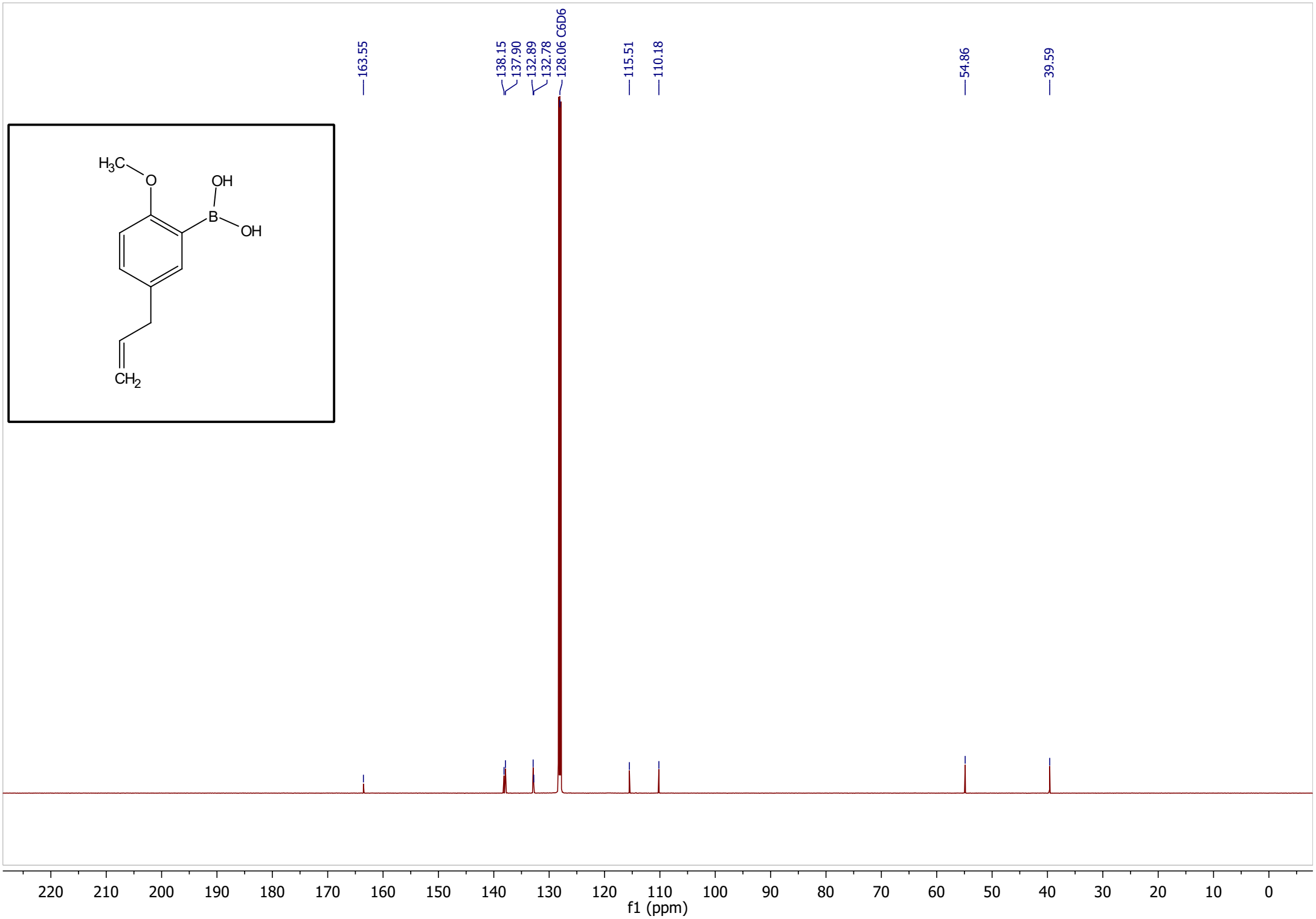


^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.54 (d, *J* = 2.4 Hz, 1H), 7.33 (dd, *J* = 8.3, 2.4 Hz, 1H), 7.00 (d, *J* = 8.3 Hz, 1H), 6.01 – 5.92 (m, 1H), 5.12 – 5.03 (m, 2H), 4.69 (s, 1H), 4.04 (s, 1H), 3.38 (ddd, *J* = 7.3, 1.4, 1.4 Hz, 2H), 1.34 – 1.27 (m, 1H).

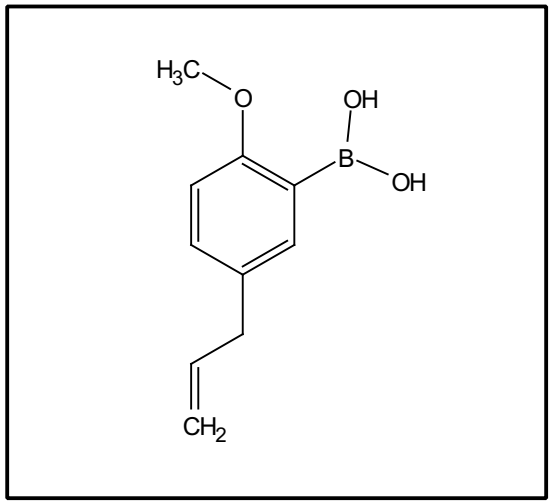

Comparison of ^{1}H NMR spectra of
boronic acid dimer
versus
a 1:3 respective mixture of boroinc acid dimer and monomer
HO
CH2
B
O
O
B
HO
CH2
1 : 3
mixture
OH
OH
B
OH
CH2
2
1
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

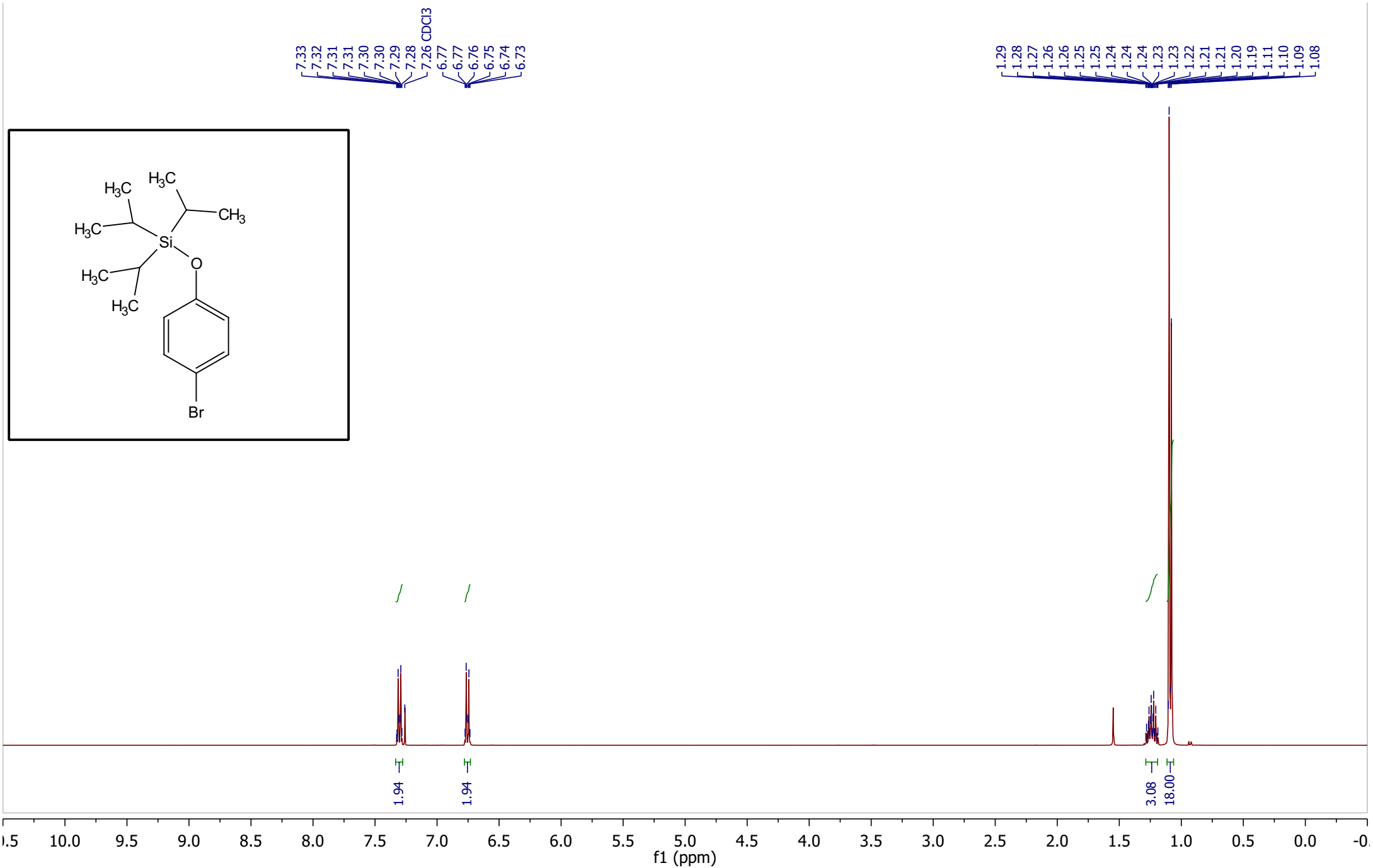


^{1}H NMR (500 MHz, Benzene-d_6) δ 8.06 (d, J = 2.4 Hz, 1H), 7.05 (dd, J = 8.4, 2.4 Hz, 1H), 6.37 (d, J = 8.4 Hz, 1H), 6.28 (s, 2H, 2x B-OH), 5.96 – 5.76 (m, 1H), 4.99 – 4.94 (m, 2H), 3.17 (ddd, J = 6.6, 1.6, 1.6 Hz, 2H), 2.99 (s, 3H).

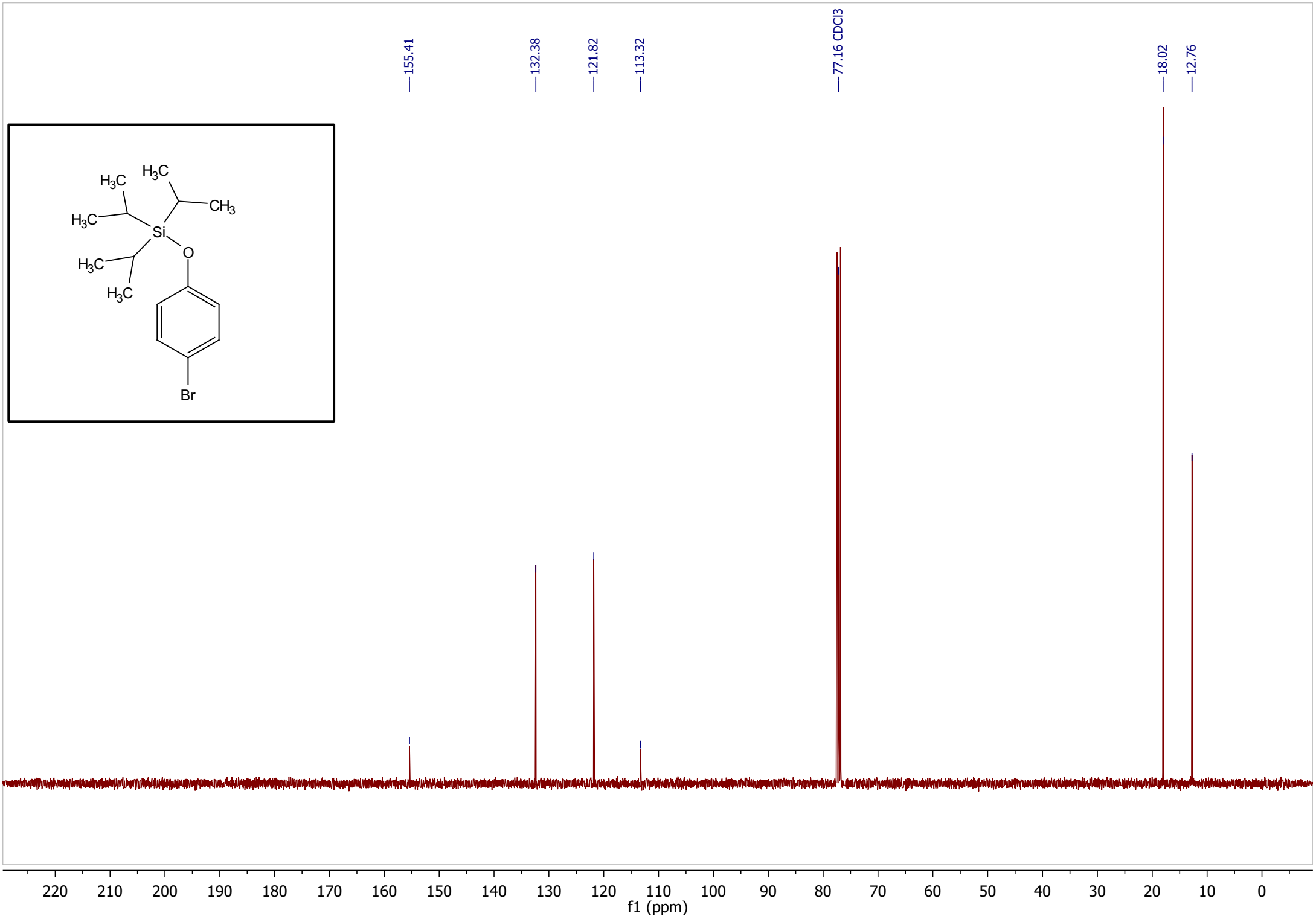

163.55
138.15
137.90
132.89
132.78
128.06 C6D6
115.51
110.18
54.86
39.59
H3C
O
OH
B
OH
CH2
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

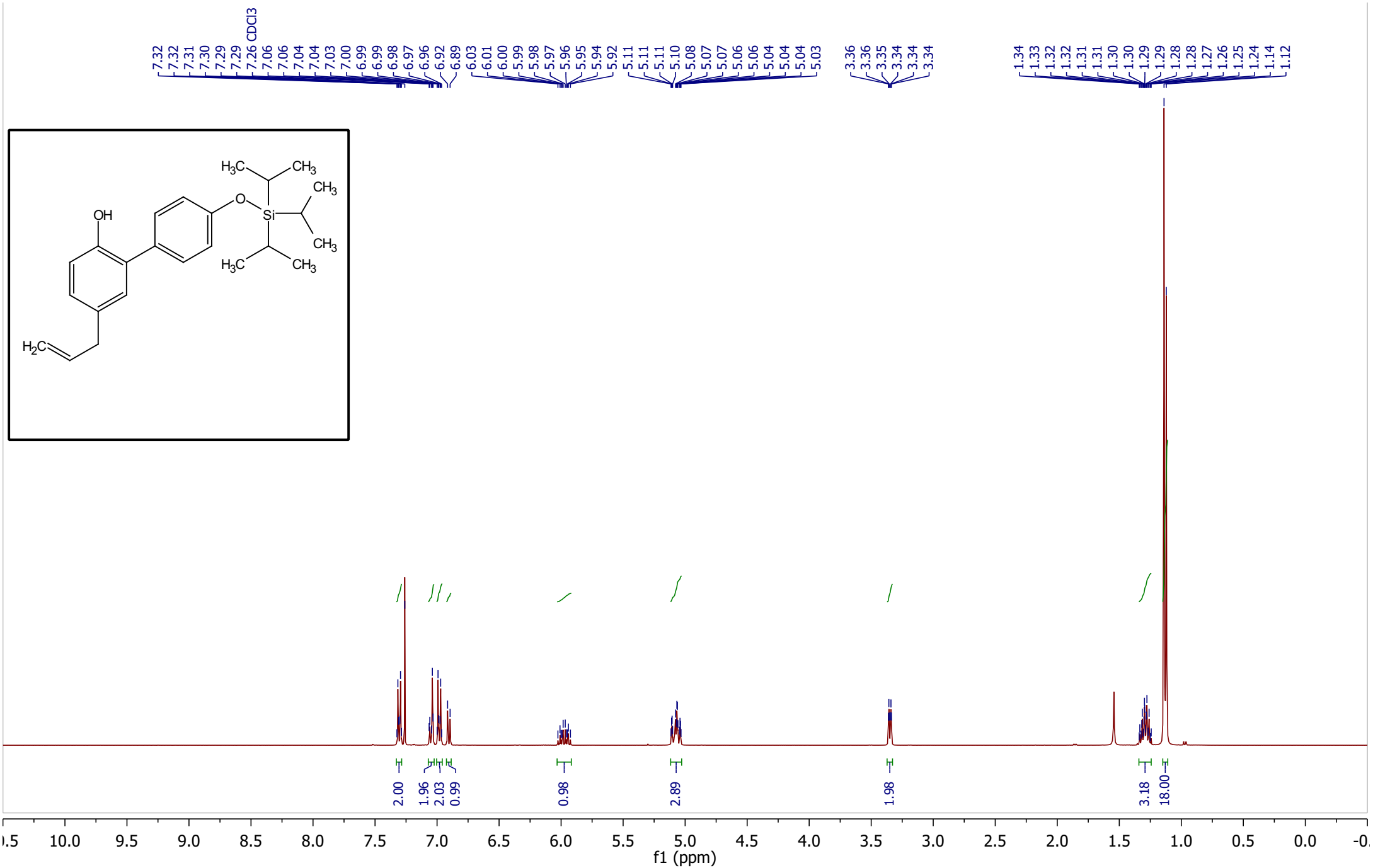
^{11}B NMR spectra



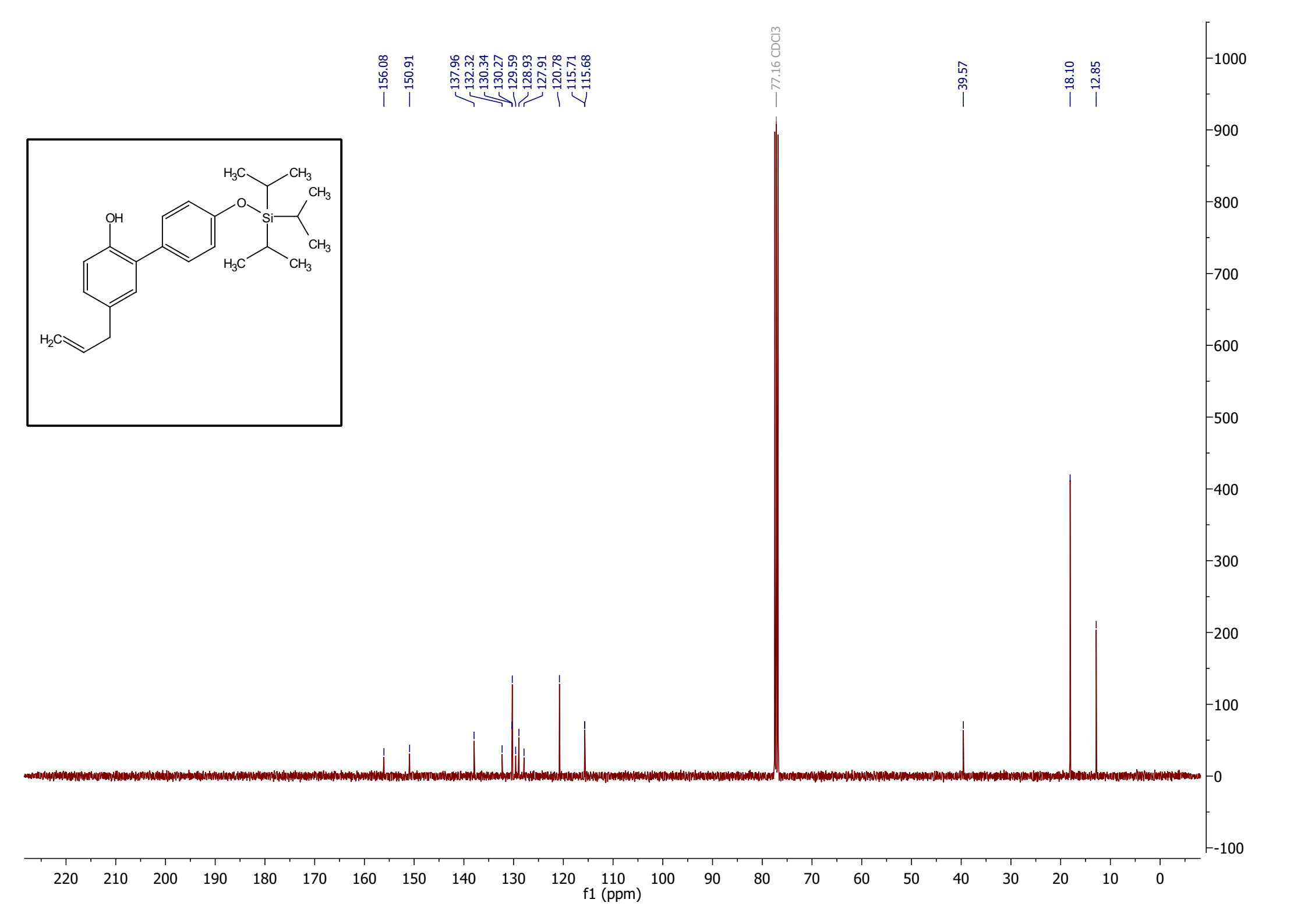


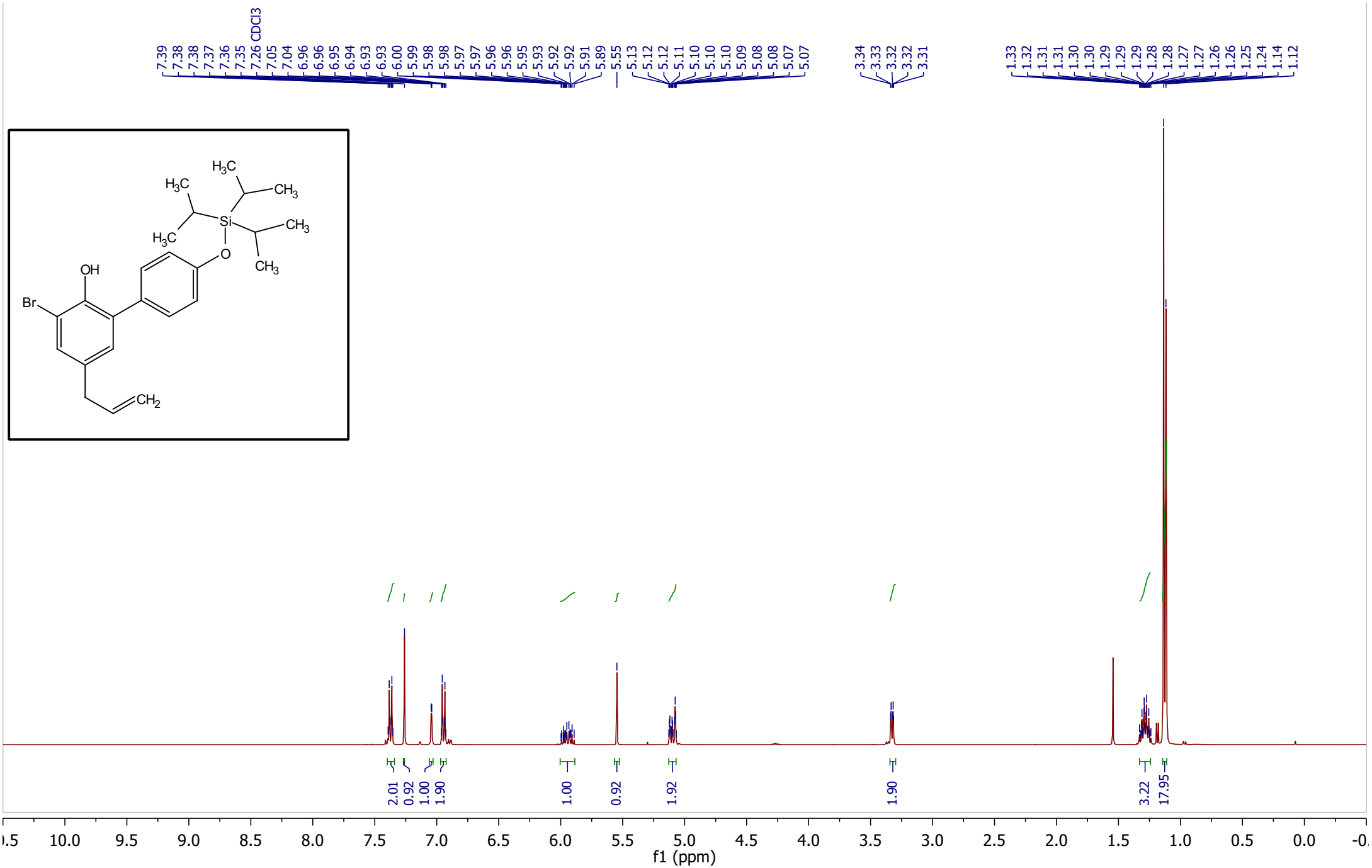
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.33 – 7.28 (m, 2H), 6.78 – 6.73 (m, 2H), 1.29 – 1.19 (m, 3H), 1.09 (d, *J* = 7.3 Hz, 18H).


155.41
132.38
121.82
113.32
77.16 CDCl3
18.02
12.76
H3C
H3C
H3C
CH3
Si
O
H3C
H3C
Br
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

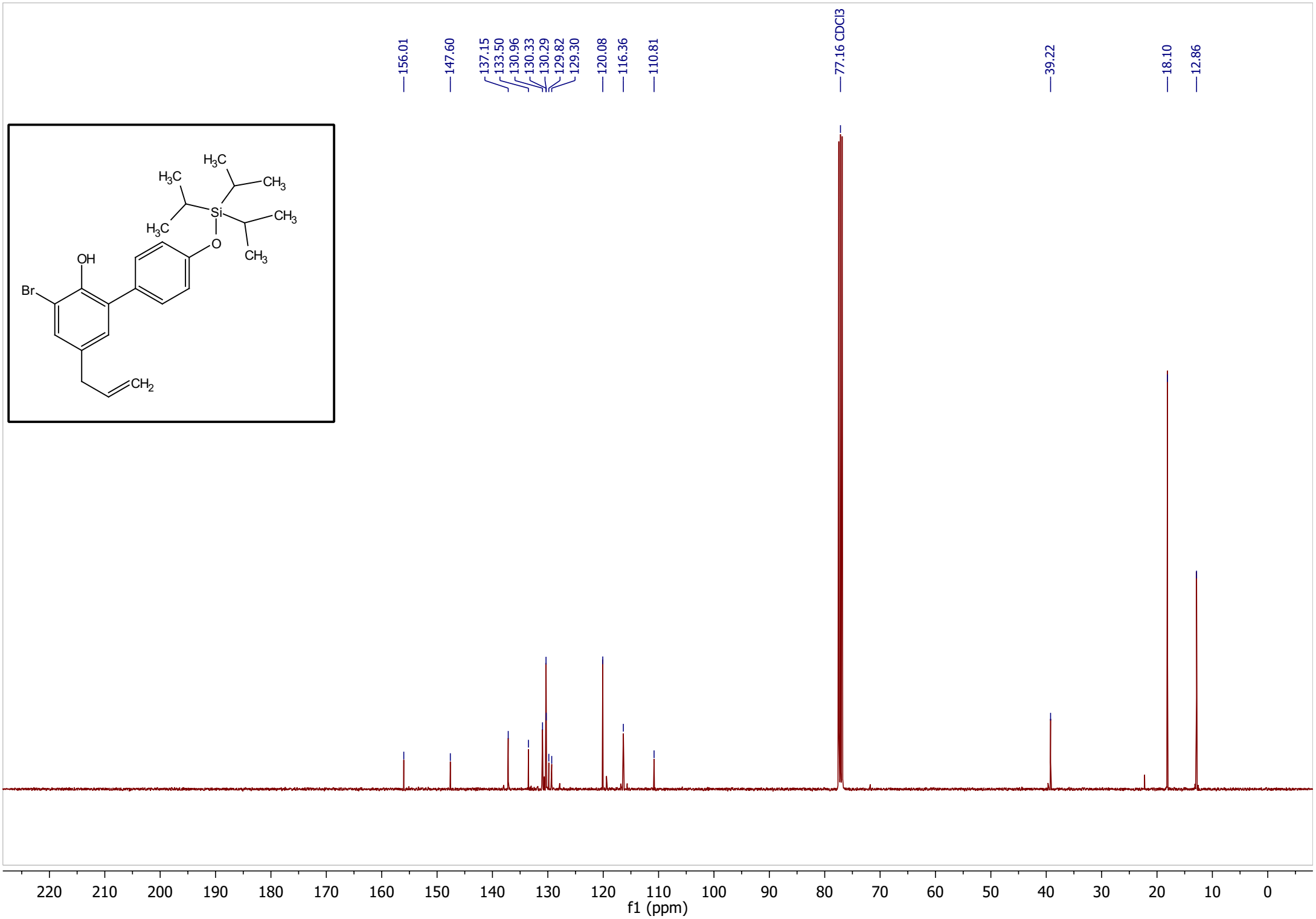


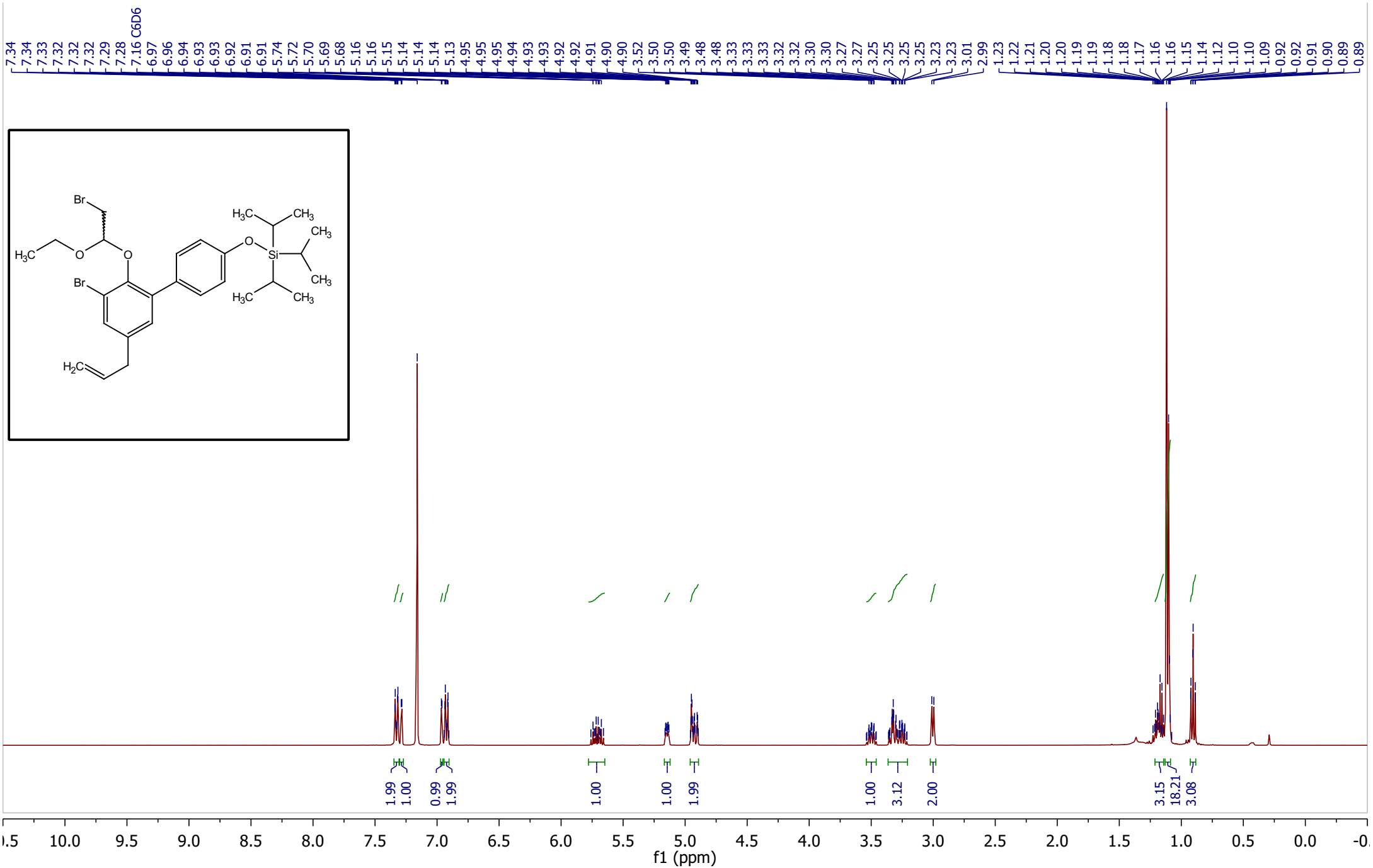
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.33 – 7.28 (m, 2H), 7.06 – 7.02 (m, 2H), 7.00 – 6.96 (m, 2H), 6.90 (d, *J* = 8.1 Hz, 1H), 5.98 (ddt, *J* = 16.8, 10.0, 6.7 Hz, 1H), 5.12 – 5.03 (m, 3H), 3.35 (ddd, *J* = 6.6, 1.4, 1.4 Hz, 2H), 1.34 – 1.24 (m, 3H), 1.13 (d, *J* = 7.3 Hz, 18H).


156.08
150.91
137.96
132.32
130.34
130.27
129.59
128.93
127.91
120.78
115.71
115.68
77.16 CDCl3
39.57
18.10
12.85
OH
O
Si
H3C
CH3
CH3
CH3
H3C
CH3
H2C
1000
900
800
700
600
500
400
300
200
100
0
-100
220
210
200
190
180
170
160
150
140
130
120
110
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90
80
70
60
50
40
30
20
10
0
f1 (ppm)

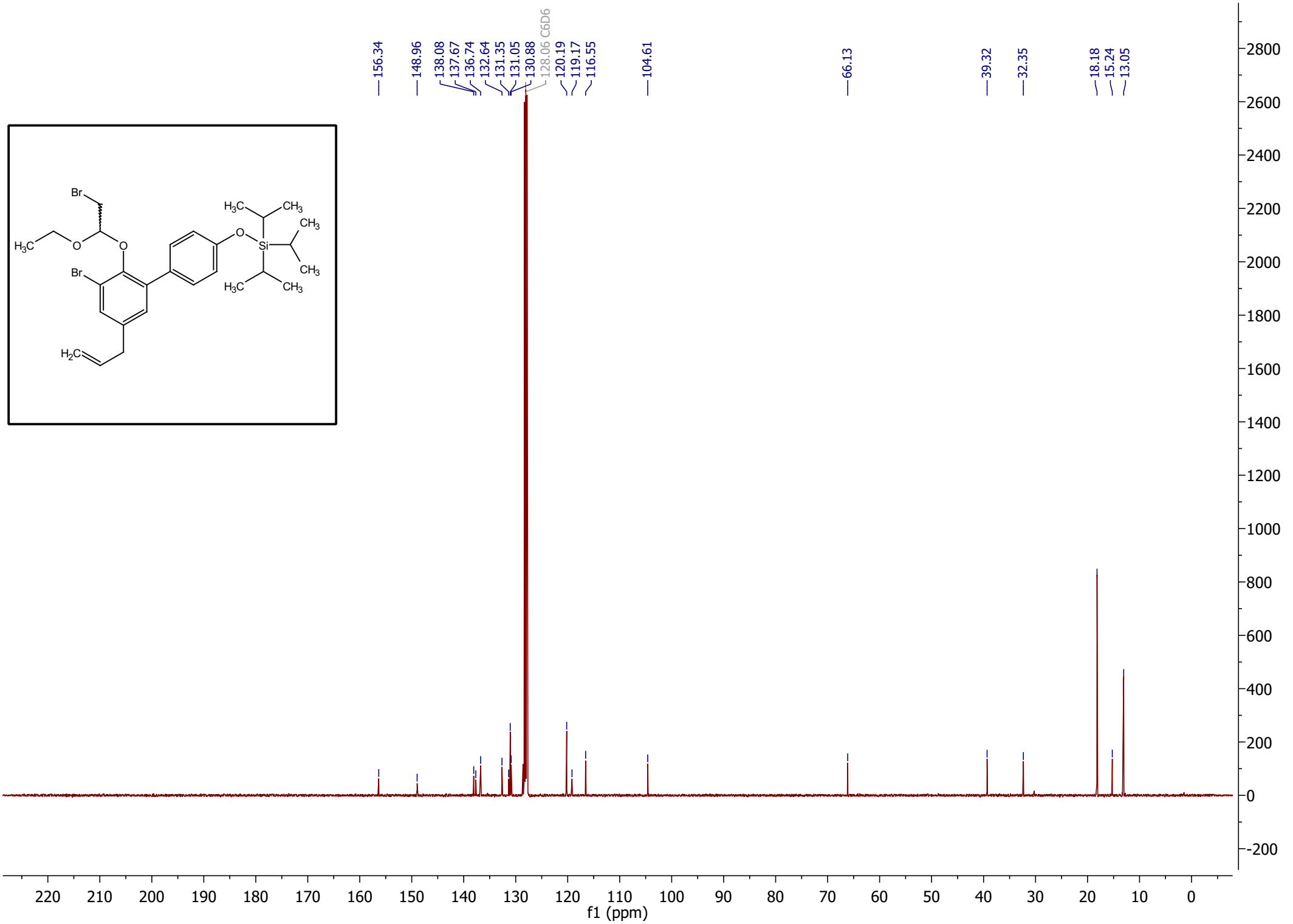


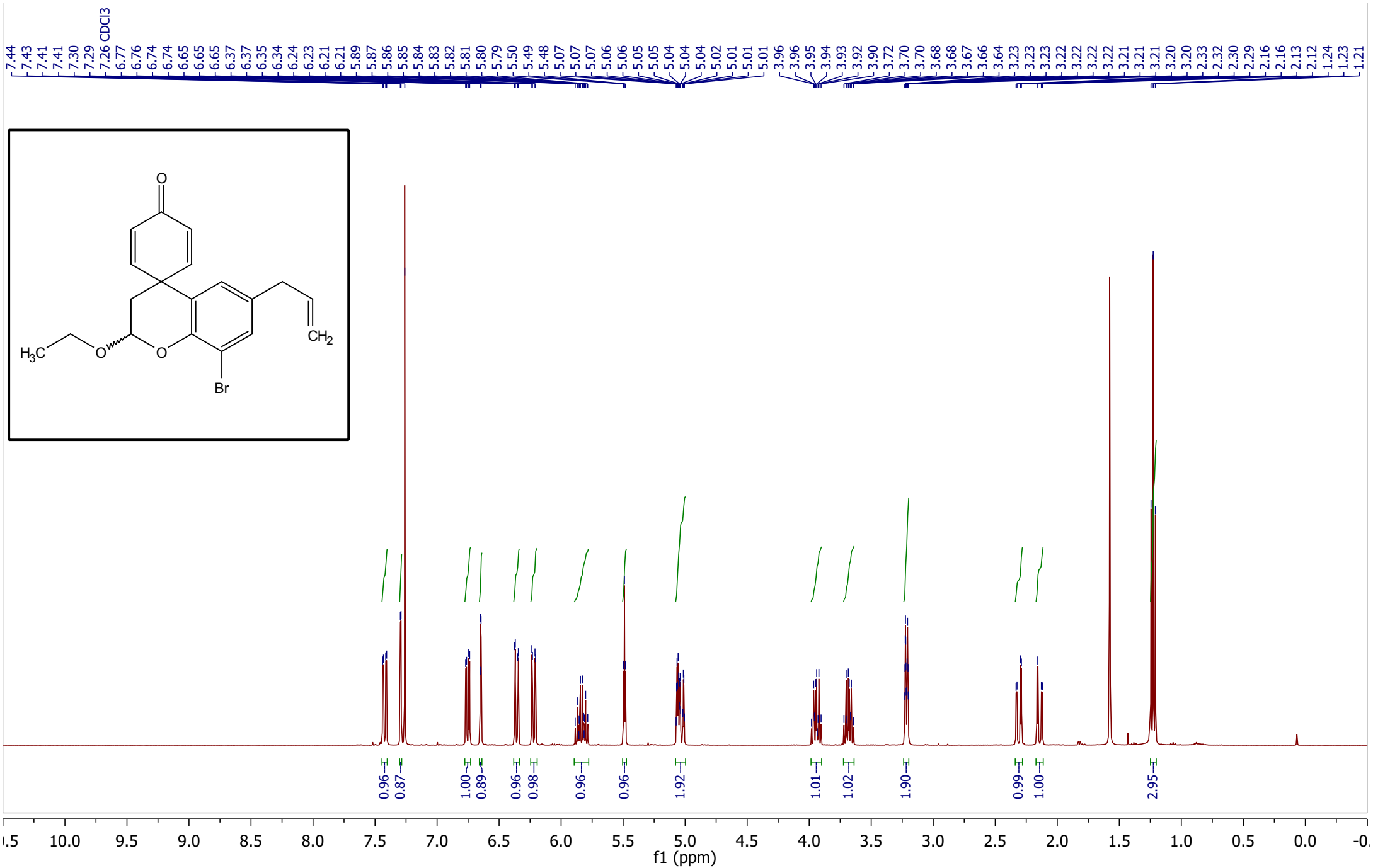
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.40 – 7.34 (m, 2H), 7.26 (d, *J* = 2.1 Hz, 1H) 7.04 (d, *J* = 2.1 Hz, 1H), 6.98 – 6.92 (m, 2H), 5.94 (ddt, *J* = 16.9, 10.2, 6.7 Hz, 1H), 5.55 (s, 1H), 5.14 – 5.06 (m, 2H), 3.33 (ddd, *J* = 6.6, 1.5, 1.5 Hz, 2H), 1.35 – 1.24 (m, 3H), 1.13 (d, *J* = 7.3 Hz, 18H).


156.01
147.60
137.15
133.50
130.96
130.33
130.29
129.82
129.30
120.08
116.36
110.81
77.16 CDCl3
39.22
18.10
12.86
OH
Br
O
Si
H3C
CH3
CH2
220
210
200
190
180
170
160
150
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130
120
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90
80
70
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40
30
20
10
0
f1 (ppm)

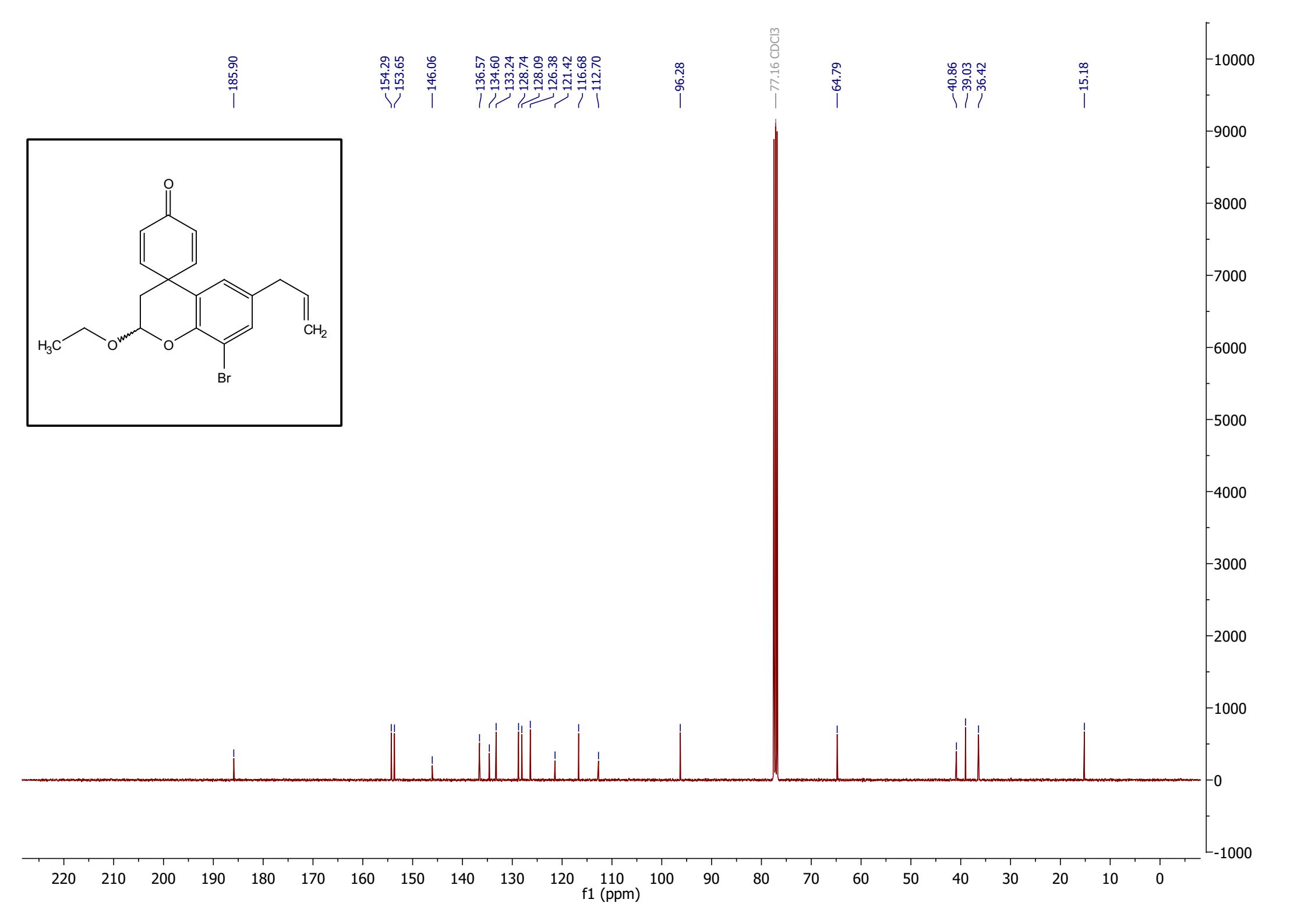


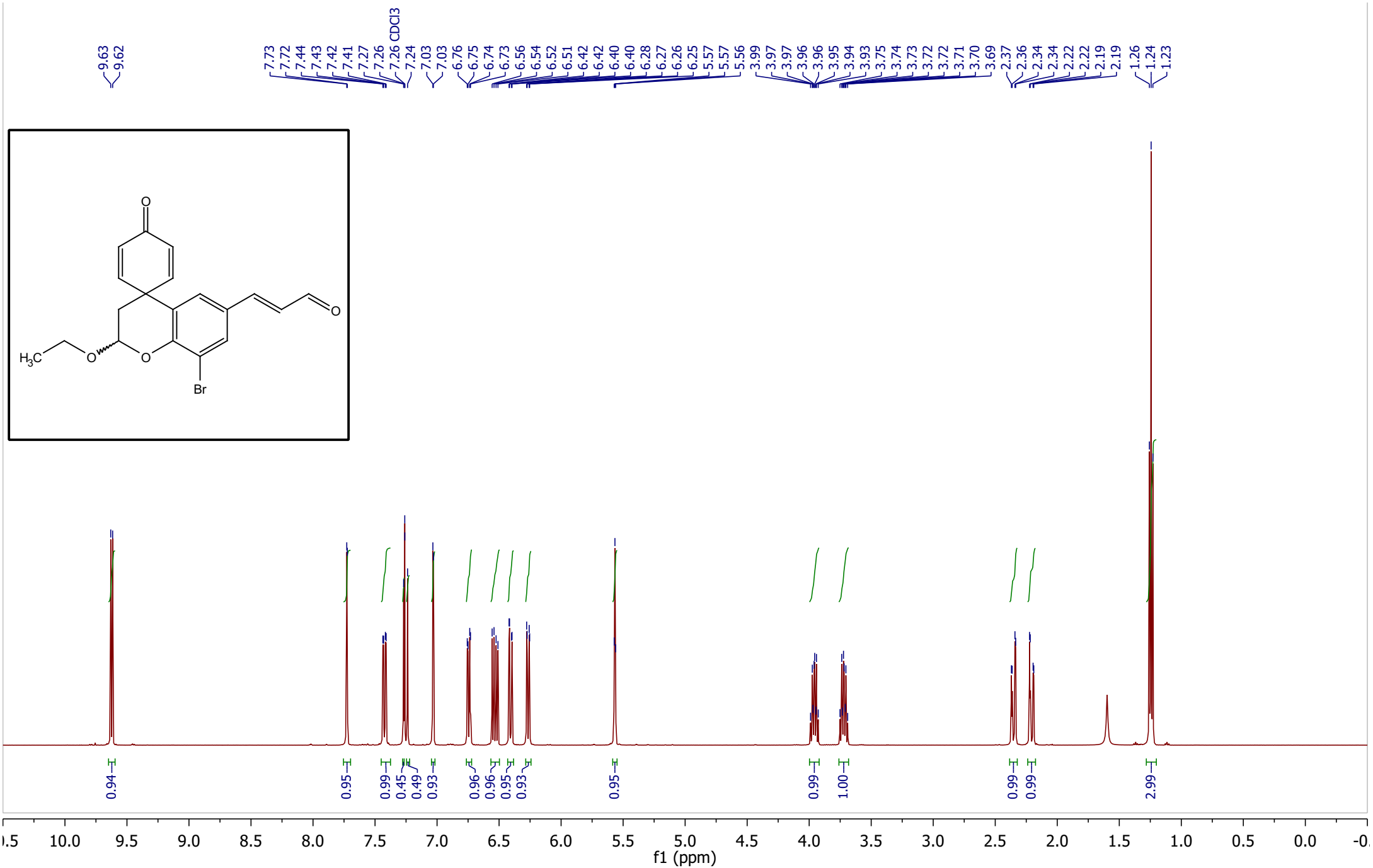
^{1}H NMR (400 MHz, Benzene-d_6) δ 7.35 – 7.31 (m, 2H), 7.29 (d, J = 2.1 Hz, 1H), 6.96 (d, J = 2.1 Hz, 1H), 6.94 – 6.90 (m, 2H), 5.71 (dddd, J = 16.9, 10.3, 6.8, 6.8 Hz, 1H), 5.16 – 5.12 (m, 1H), 4.97 – 4.89 (m, 2H), 3.55 – 3.45 (m, 1H), 3.37 – 3.20 (m, 3H), 3.00 (d, J = 6.7 Hz, 2H), 1.23 – 1.13 (m, 3H), 1.11 (d, J = 6.4 Hz, 18H), 0.90 (dd, J = 7.0, 7.0 Hz, 2H).


156.34
148.96
138.08
137.67
136.74
132.64
131.35
131.05
130.88
128.06 C6D6
120.19
119.17
116.55
104.61
66.13
39.32
32.35
18.18
15.24
13.05
Br
H3C
O
O
Br
H2C
O
Si
H3C
CH3
CH3
CH3
H3C
CH3
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
-200
220
210
200
190
180
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10
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f1 (ppm)

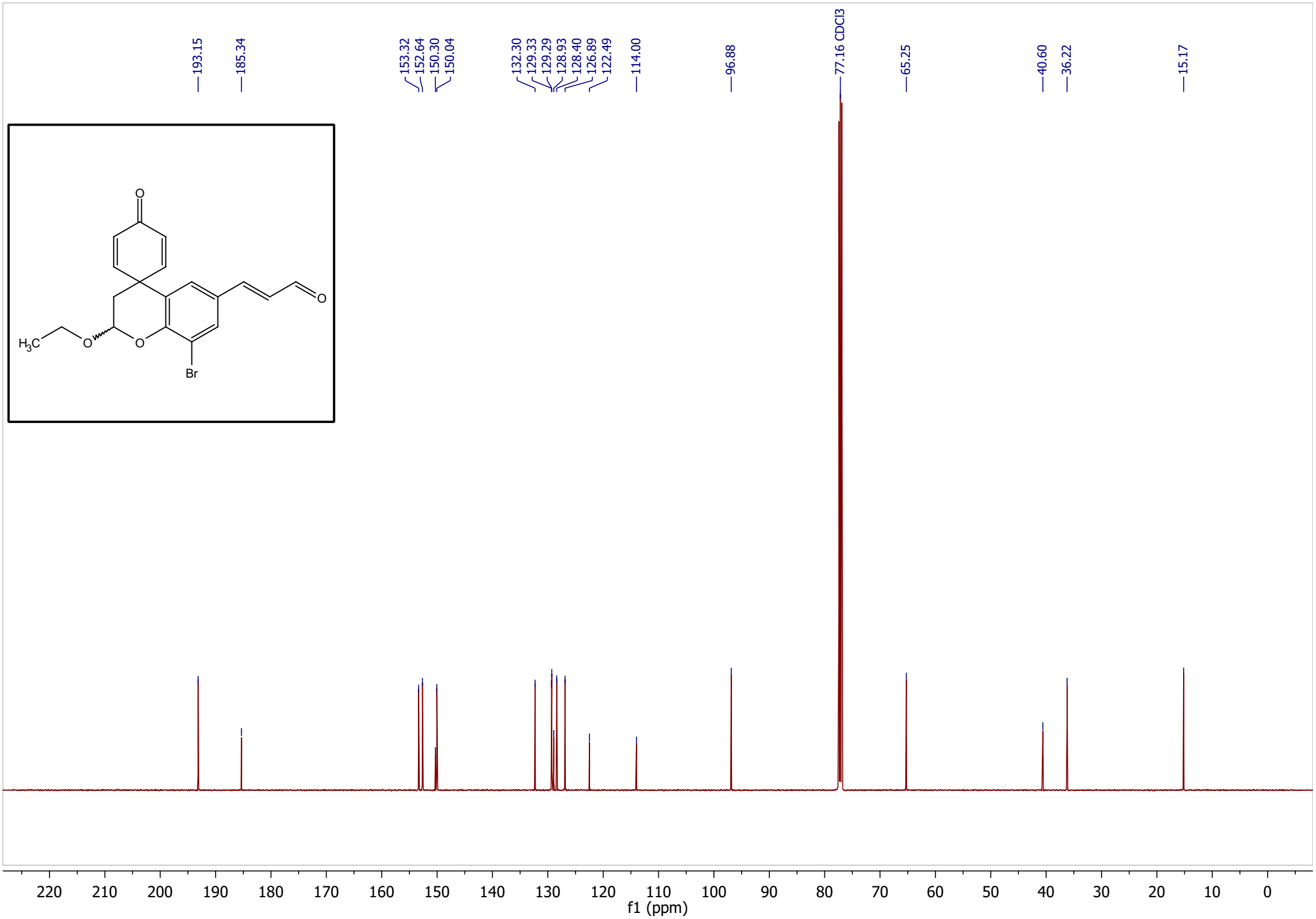


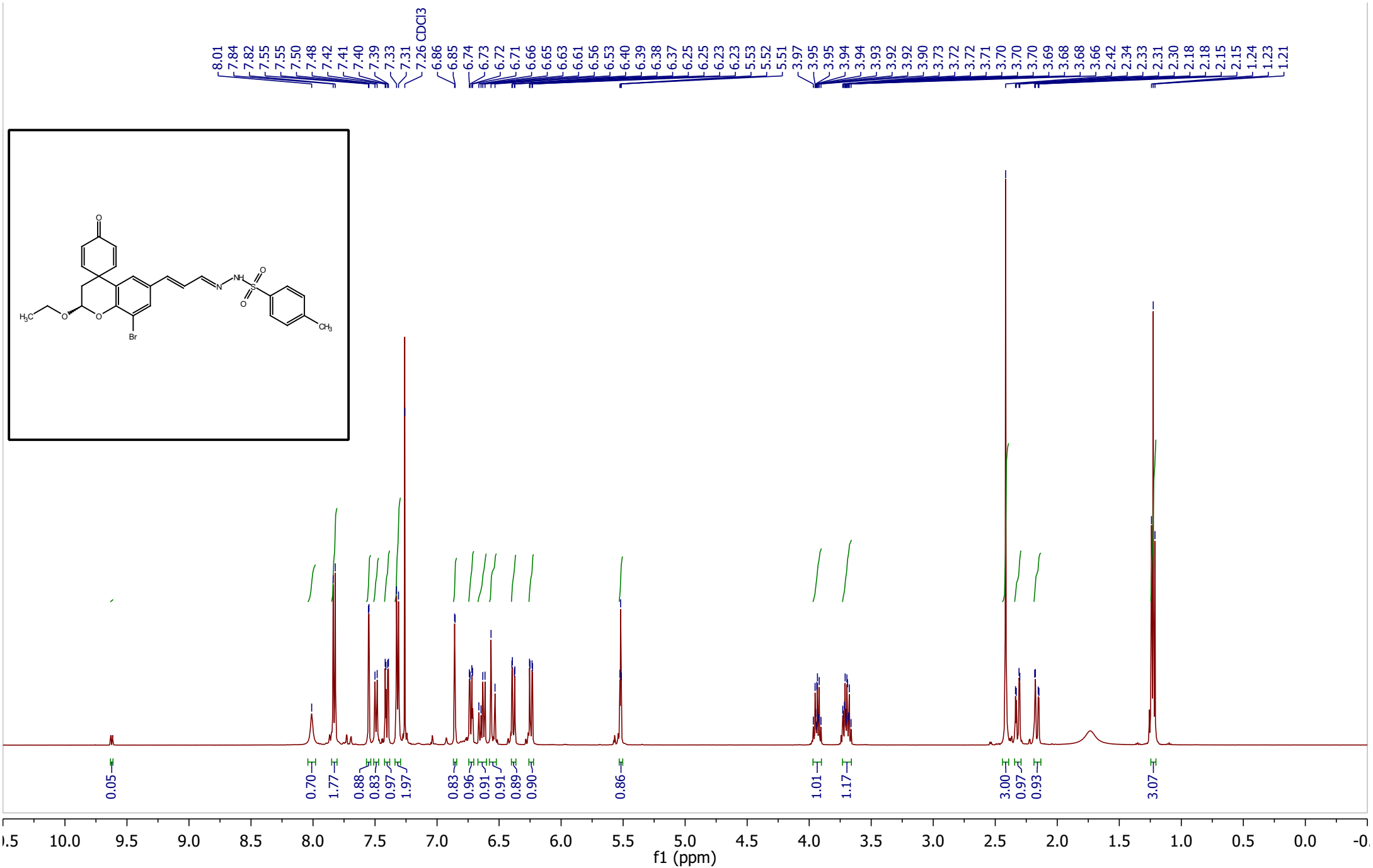
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.42 (dd, *J* = 10.2, 3.0 Hz, 1H), 7.29 (d, *J* = 2.1 Hz, 1H), 6.75 (dd, *J* = 10.0, 3.0 Hz, 1H), 6.65 (d, *J* = 2.1 Hz, 1H), 6.36 (dd, *J* = 10.0, 1.9 Hz, 1H), 6.22 (dd, *J* = 10.2, 1.9 Hz, 1H), 5.84 (dddd, *J* = 16.9, 10.2, 6.7, 6.7 Hz, 1H), 5.49 (dd, *J* = 3.0, 3.0 Hz, 1H), 5.08 – 5.00 (m, 2H), 3.94 (dq, *J* = 9.7, 7.1 Hz, 1H), 3.68 (dq, *J* = 9.7, 7.1 Hz, 1H), 3.22 (ddd, *J* = 6.7, 1.4, 1.4 Hz, 2H), 2.31 (dd, *J* = 14.2, 3.0 Hz, 1H), 2.14 (dd, *J* = 14.2, 3.0 Hz, 1H), 1.23 (dd, *J* = 7.1, 7.1 Hz, 3H).


185.90
154.29
153.65
146.06
136.57
134.60
133.24
128.74
128.09
126.38
121.42
116.68
112.70
96.28
77.16 CDCl3
64.79
40.86
39.03
36.42
15.18
O
H3C
O
O
Br
CH2
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
220
210
200
190
180
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0
f1 (ppm)

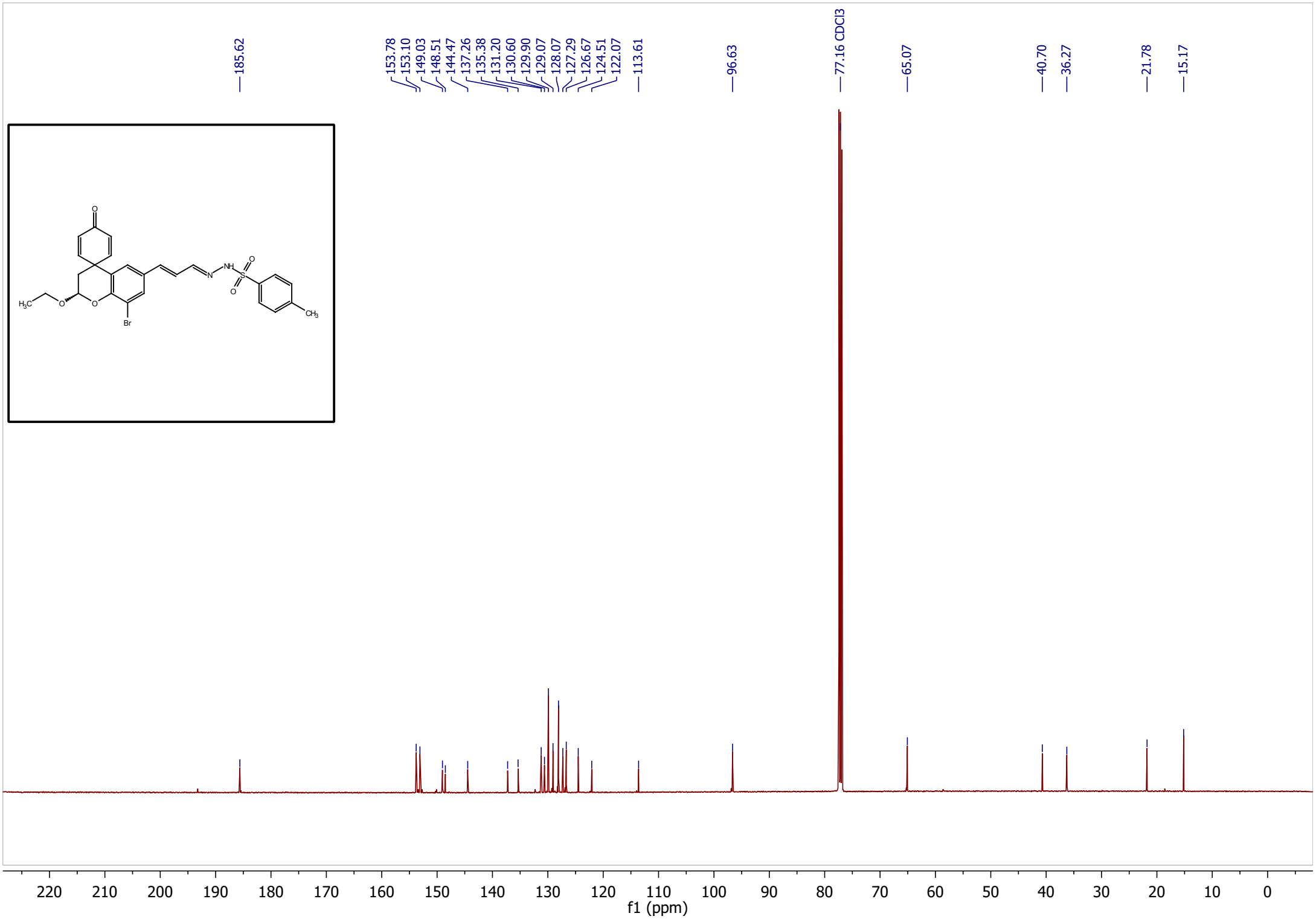


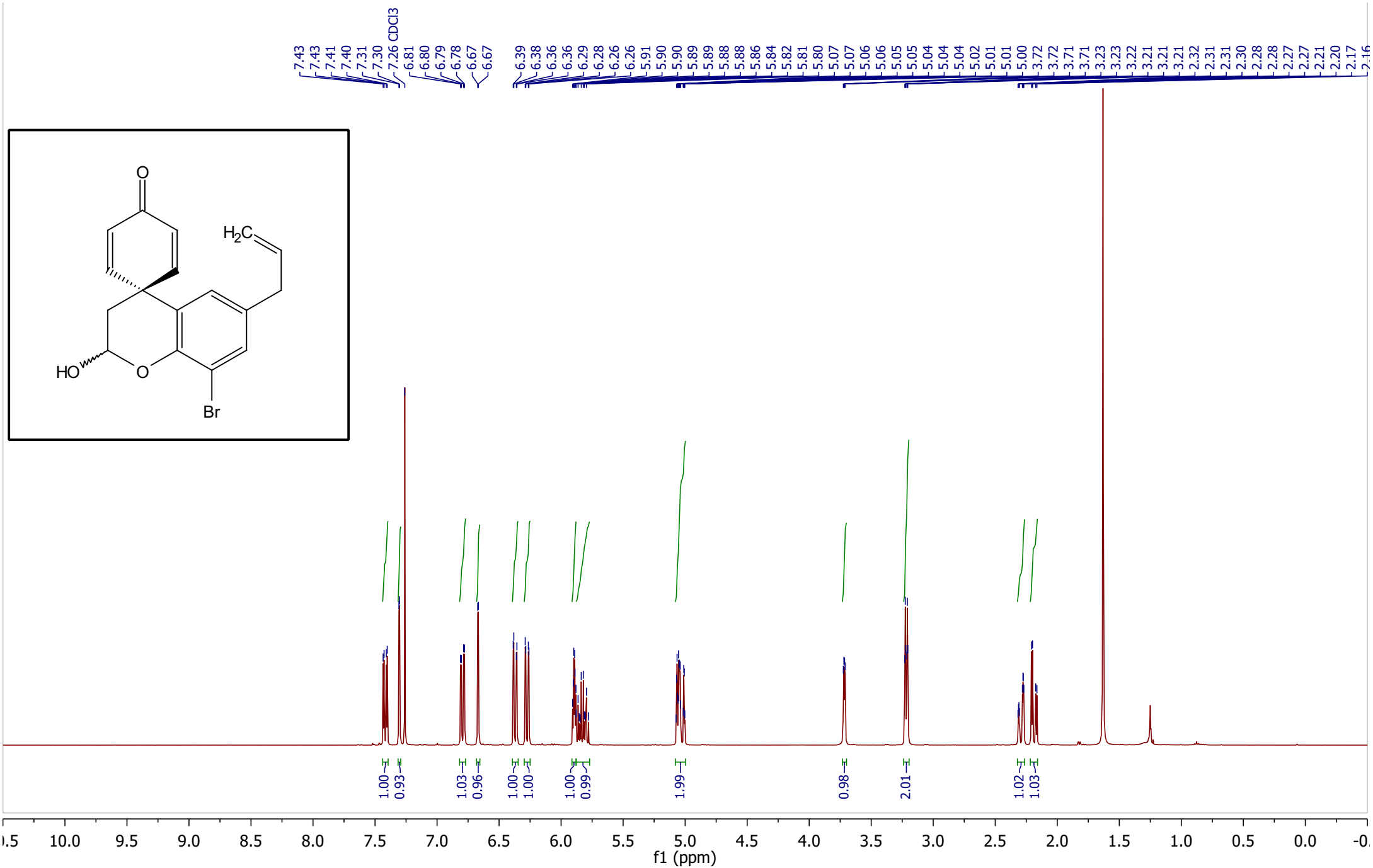
^{1}H NMR (500 MHz, Chloroform-*d*) δ 9.62 (d, *J* = 7.6 Hz, 1H), 7.73 (d, *J* = 2.1 Hz, 1H), 7.42 (dd, *J* = 10.1, 3.0 Hz, 1H), 7.25 (d, *J* = 15.9 Hz, 1H), 7.03 (d, *J* = 2.1 Hz, 1H), 6.74 (dd, *J* = 10.0, 3.0 Hz, 1H), 6.53 (dd, *J* = 15.9, 7.6 Hz, 1H), 6.41 (dd, *J* = 10.0, 1.9 Hz, 1H), 6.27 (dd, *J* = 10.1, 1.9 Hz, 1H), 5.57 (dd, *J* = 2.9, 2.9 Hz, 1H), 3.96 (dq, *J* = 9.7, 7.1 Hz, 1H), 3.72 (dq, *J* = 9.7, 7.1 Hz, 1H), 2.35 (dd, *J* = 14.3, 2.9 Hz, 1H), 2.21 (dd, *J* = 14.3, 2.9 Hz, 1H), 1.24 (dd, *J* = 7.1, 7.1 Hz, 3H).


193.15
185.34
153.32
152.64
150.30
150.04
132.30
129.33
129.29
128.93
128.40
126.89
122.49
114.00
96.88
77.16 CDCl3
65.25
40.60
36.22
15.17
O
O
O
O
H3C
Br
220
210
200
190
180
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20
10
0
f1 (ppm)

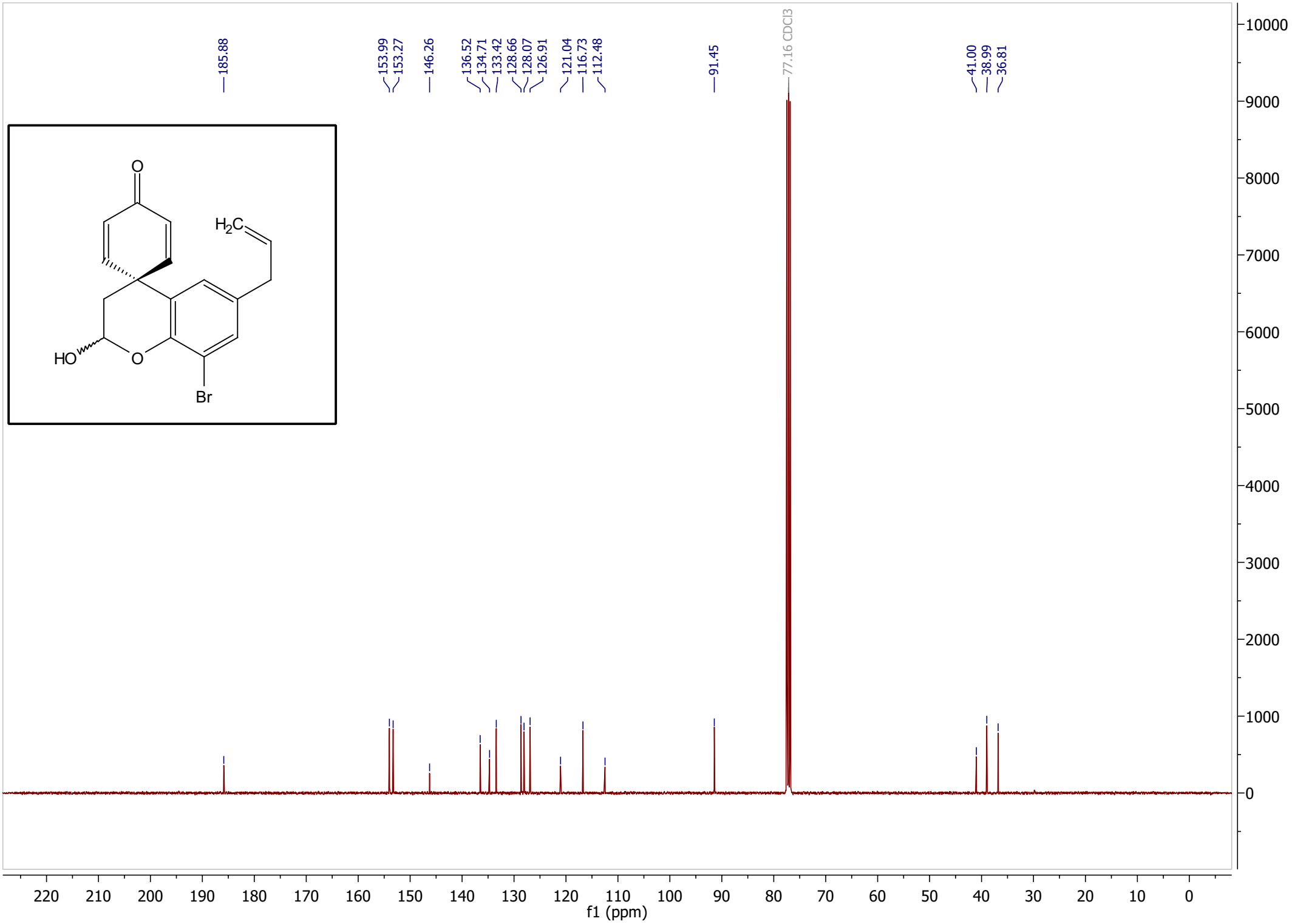


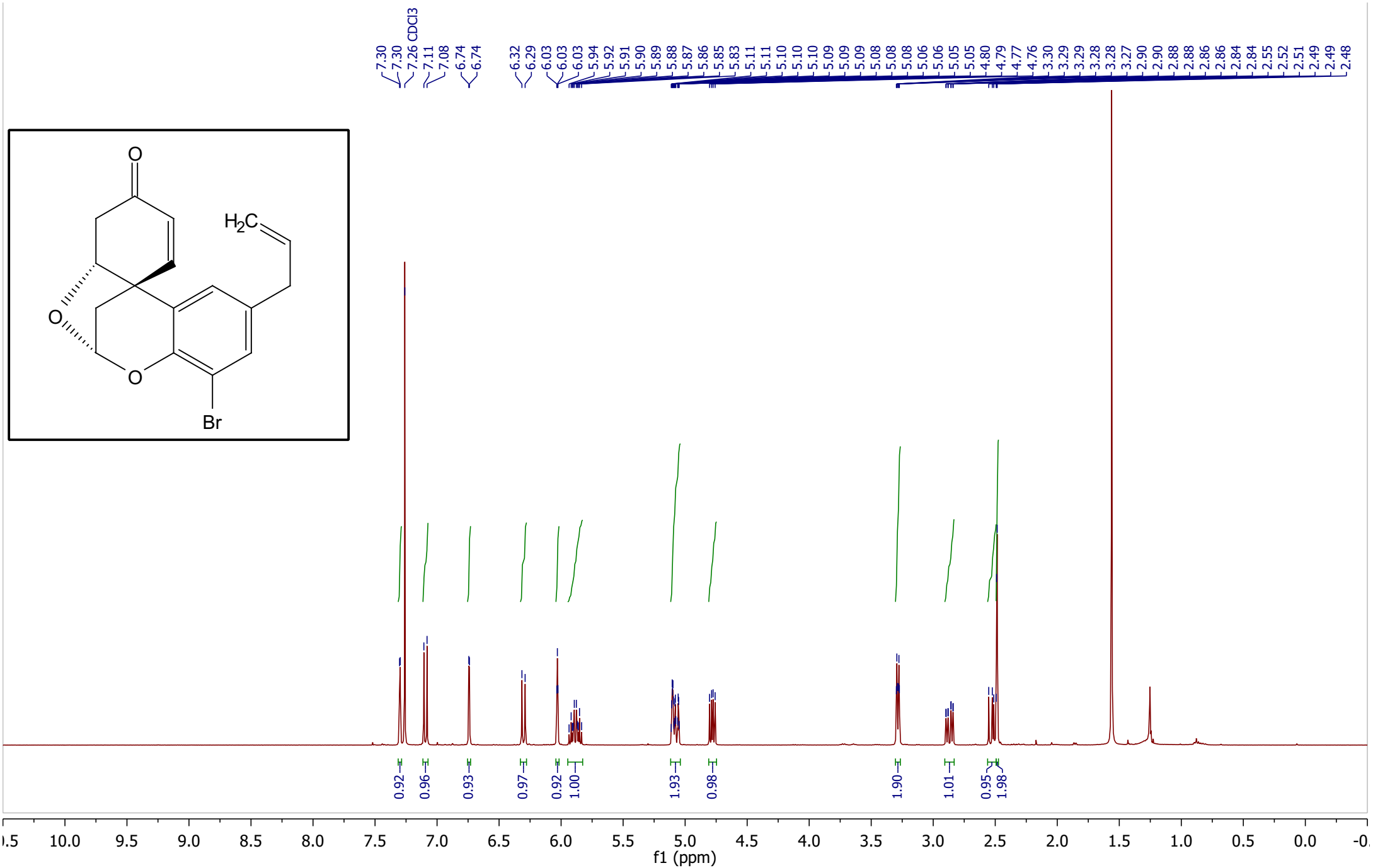
^{1}H NMR (500 MHz, Chloroform-*d*) δ 8.01 (s, 1H, NH), 7.83 (d, *J* = 8.2 Hz, 2H), 7.55 (d, *J* = 2.1 Hz, 1H), 7.49 (d, *J* = 8.8 Hz, 1H), 7.41 (dd, *J* = 10.2, 2.9 Hz, 1H), 7.32 (d, *J* = 8.2 Hz, 2H), 6.86 (d, *J* = 2.1 Hz, 1H), 6.73 (dd, *J* = 10.0, 3.0 Hz, 1H), 6.64 (dd, *J* = 16.0, 8.8 Hz, 1H), 6.55 (d, *J* = 16.0 Hz, 1H), 6.38 (dd, *J* = 10.0, 1.9 Hz, 1H), 6.24 (dd, *J* = 10.2, 1.9 Hz, 1H), 5.52 (dd, *J* = 2.9, 2.9 Hz, 1H), 3.94 (dq, *J* = 9.7, 7.1 Hz, 1H), 3.69 (dq, *J* = 9.7, 7.1 Hz, 1H), 2.42 (s, 3H), 2.32 (dd, *J* = 14.2, 2.9 Hz, 1H), 2.16 (dd, *J* = 14.2, 2.9 Hz, 1H), 1.23 (dd, *J* = 7.1, 7.1 Hz, 3H).


185.62
153.78
153.10
149.03
148.51
144.47
137.26
135.38
131.20
130.60
129.90
129.07
128.07
127.29
126.67
124.51
122.07
113.61
96.63
77.16 CDCl3
65.07
40.70
36.27
21.78
15.17
O
O
O
O
NH
S
O
O
N
H3C
Br
CH3
220
210
200
190
180
170
160
150
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130
120
110
100
90
80
70
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50
40
30
20
10
0
f1 (ppm)

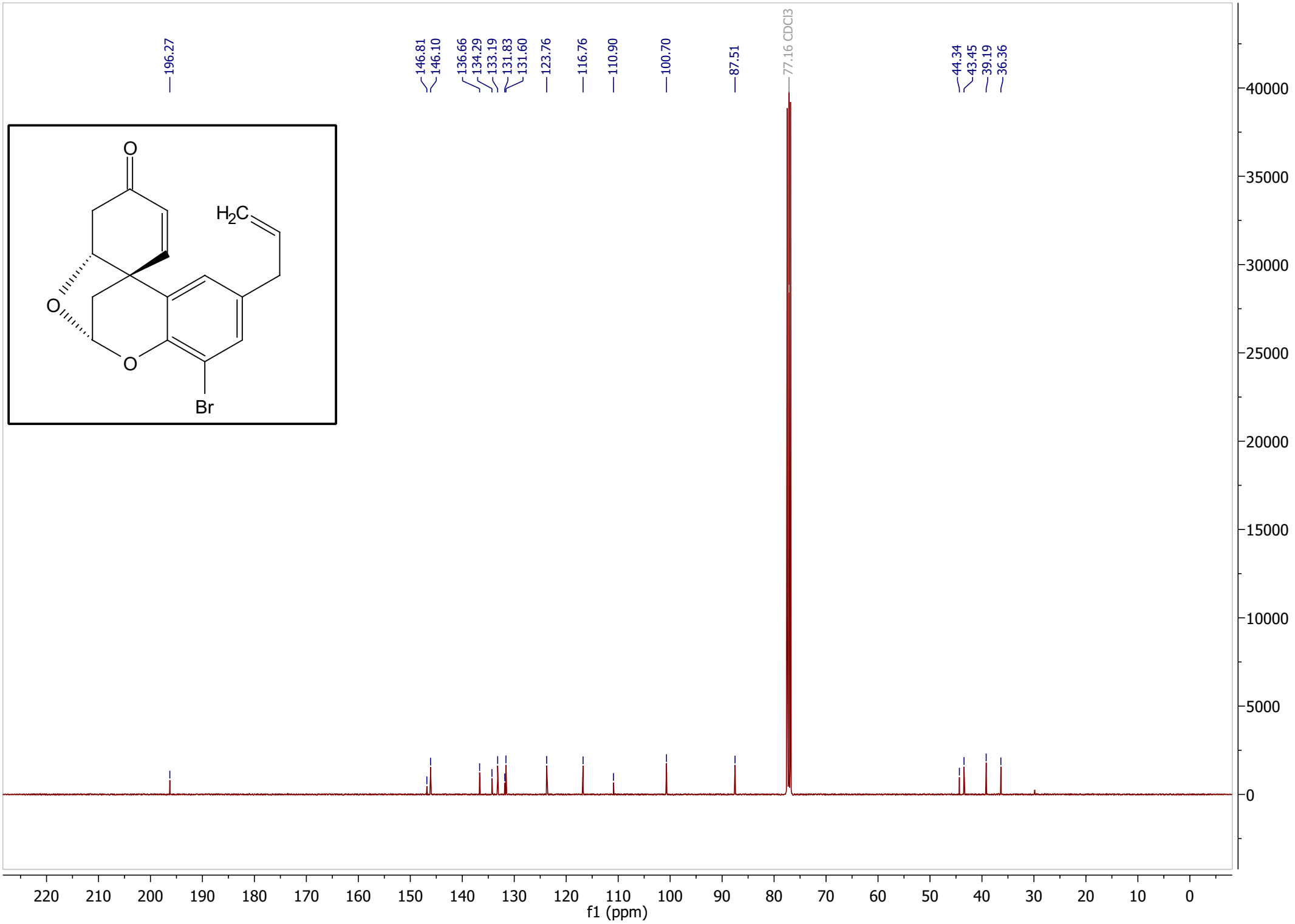


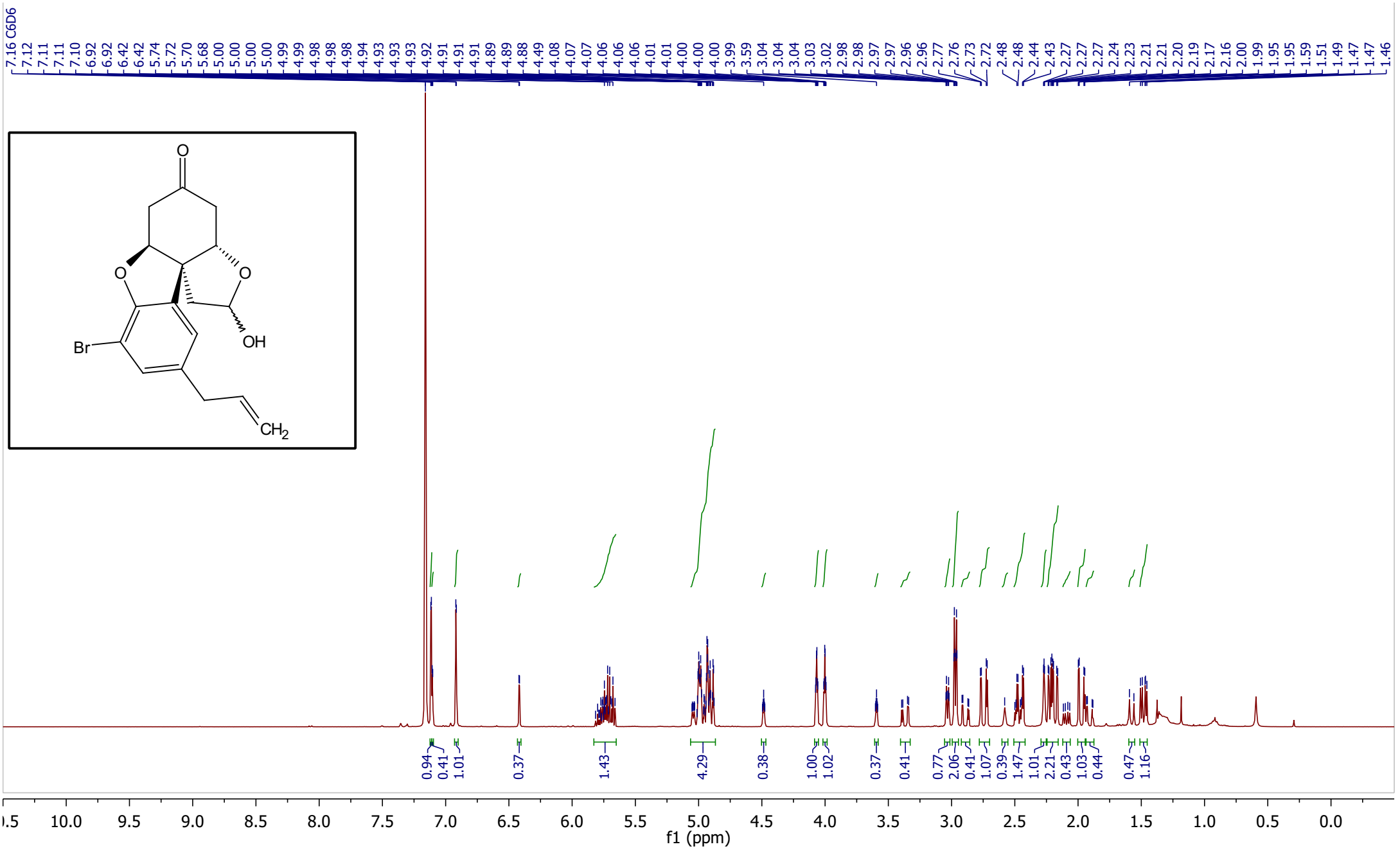
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.42 (dd, *J* = 10.2, 3.0 Hz, 1H), 7.30 (d, *J* = 2.1 Hz, 1H), 6.79 (dd, *J* = 10.0, 3.0 Hz, 1H), 6.67 (d, *J* = 2.1 Hz, 1H), 6.37 (dd, *J* = 10.0, 1.9 Hz, 1H), 6.27 (dd, *J* = 10.2, 1.9 Hz, 1H), 5.89 (ddd, *J* = 3.9, 3.9, 2.9 Hz, 1H), 5.83 (dddd, *J* = 16.9, 10.2, 6.5, 6.5 Hz, 1H), 5.08 – 4.99 (m, 2H), 3.72 (dd, *J* = 3.9, 1.7 Hz, 1H), 3.22 (ddd, *J* = 6.5, 1.5, 1.5 Hz, 2H), 2.29 (ddd, *J* = 14.1, 2.9, 1.7 Hz, 1H), 2.19 (dd, *J* = 14.1, 3.9 Hz, 1H).


185.88
153.99
153.27
146.26
136.52
134.71
133.42
128.66
128.07
126.91
121.04
116.73
112.48
91.45
77.16 CDCl3
41.00
38.99
36.81
O
H2C
HO
O
Br
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
220
210
200
190
180
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90
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0
f1 (ppm)

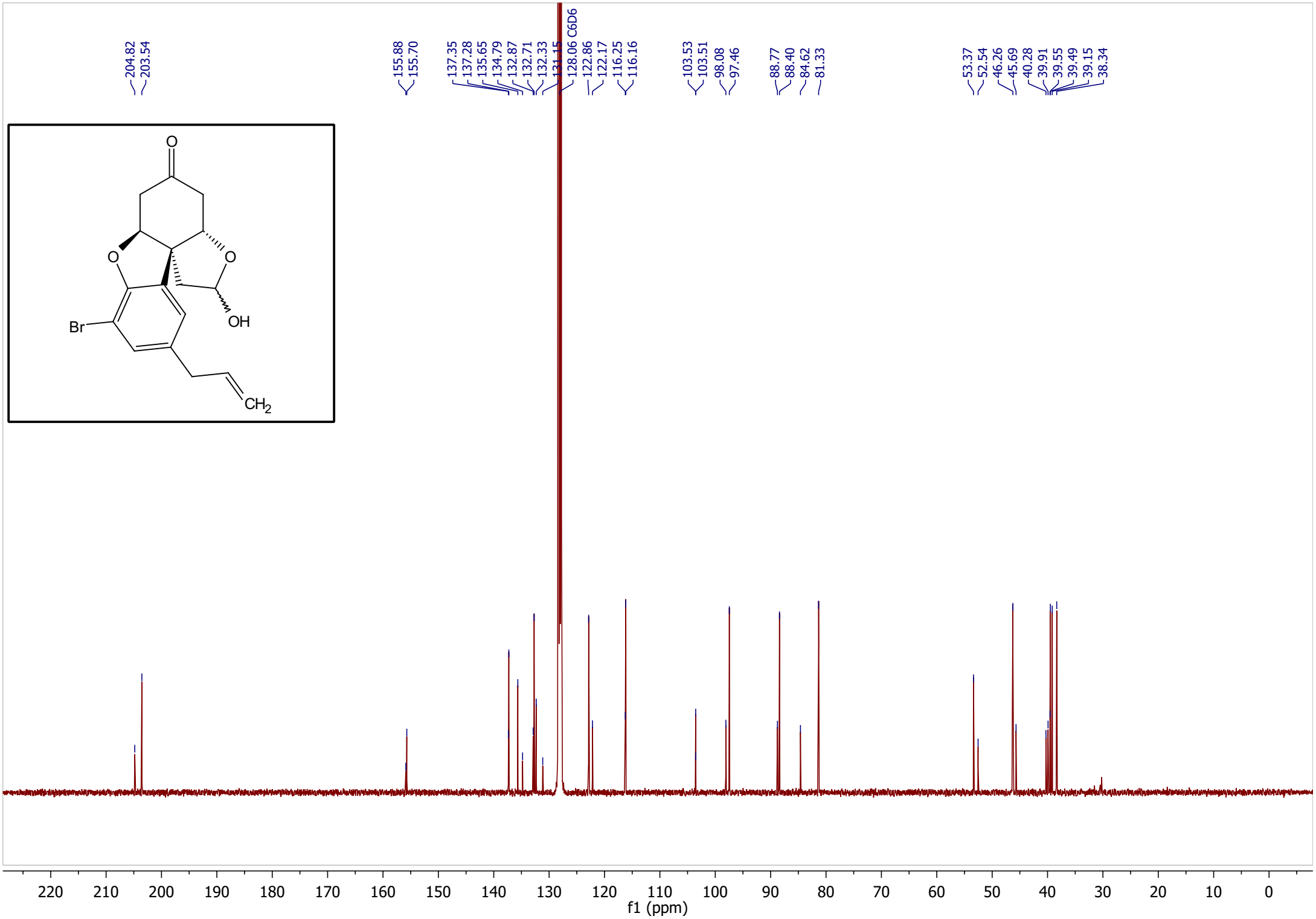


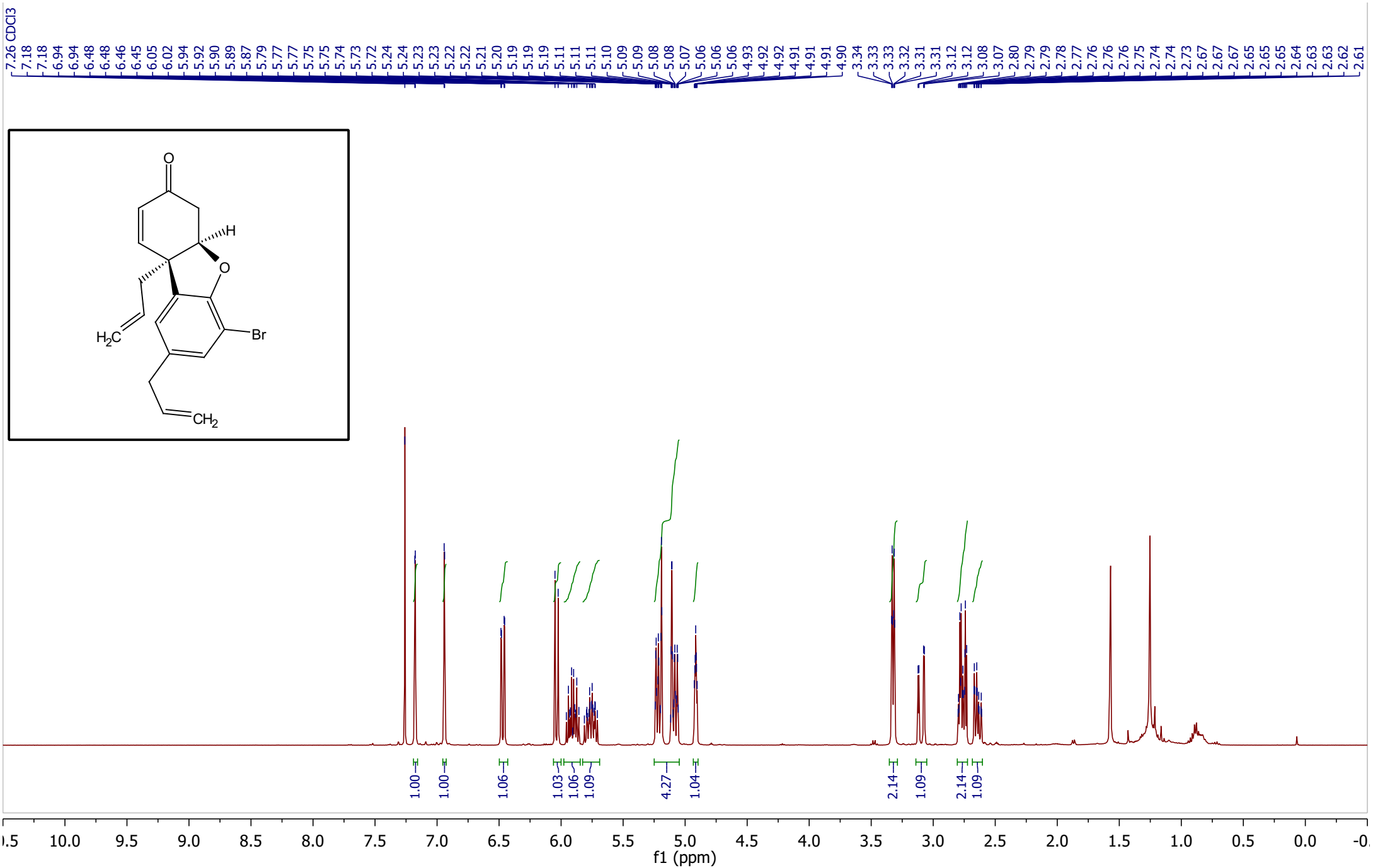
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.30 (d, *J* = 2.0 Hz, 1H), 7.09 (d, *J* = 10.2 Hz, 1H), 6.74 (d, *J* = 2.0 Hz, 1H), 6.30 (d, *J* = 10.2 Hz, 1H), 6.03 (dd, *J* = 1.8, 1.8 Hz, 1H), 5.89 (dddd, *J* = 16.9, 10.3, 6.7, 6.7 Hz, 1H), 5.11 – 5.04 (m, 2H), 4.78 (dd, *J* = 11.3, 6.6 Hz, 1H), 3.29 (ddd, *J* = 6.7, 1.5, 1.5 Hz, 2H), 2.87 (ddd, *J* = 16.1, 6.6, 0.9 Hz, 1H), 2.52 (dd, *J* = 16.1, 11.3 Hz 1H), 2.49 (d, *J* = 1.8 Hz, 2H).


196.27
146.81
146.10
136.66
134.29
133.19
131.83
131.60
123.76
116.76
110.90
100.70
87.51
77.16 CDCl3
44.34
43.45
39.19
36.36
O
H2C
O
O
Br
40000
35000
30000
25000
20000
15000
10000
5000
0
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

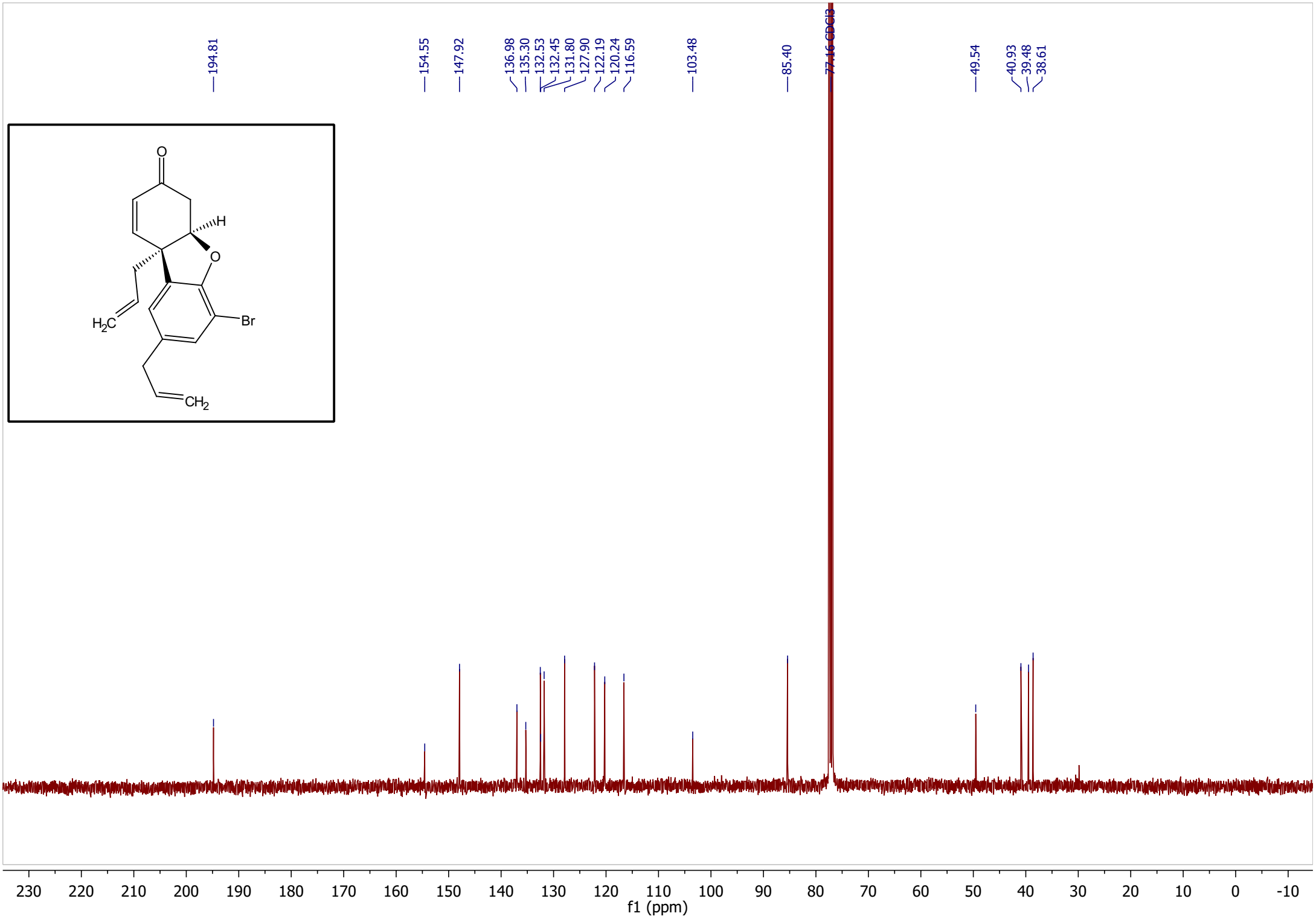


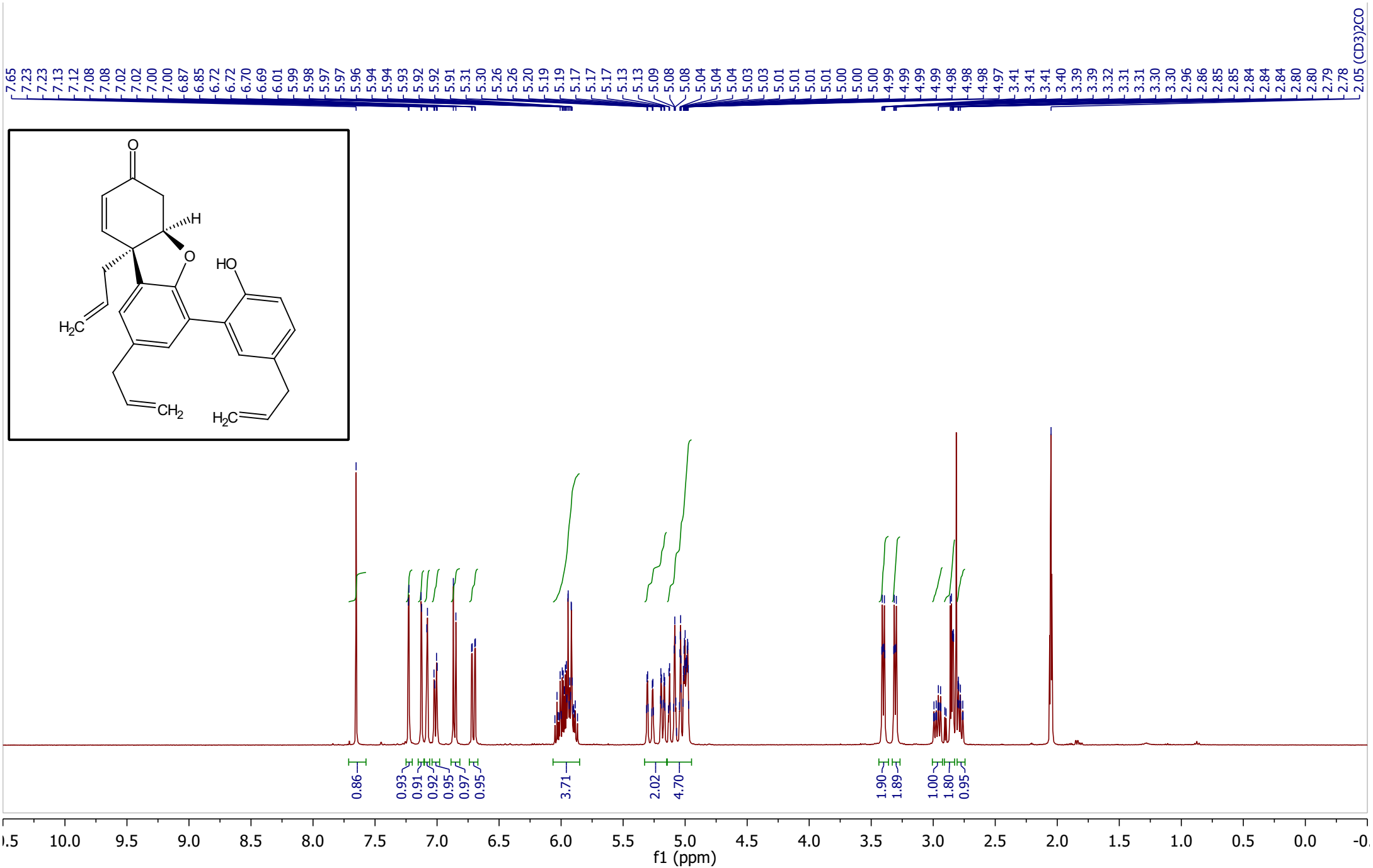
^{1}H NMR (400 MHz, Benzene-d_6) δ 7.12 (d, J = 1.7 Hz, 1H), 7.10 (d, J = 1.7 Hz, 1H), 6.92 (d, J = 1.7 Hz, 1H), 6.42 (d, J = 1.7 Hz, 1H), 5.83 – 5.64 (m, 2H), 5.06 – 4.87 (m, 6H), 4.49 (ddd, J = 3.6, 2.6, 1.1 Hz, 1H), 4.07 (ddd, J = 3.1, 3.1, 1.1 Hz, 1H), 4.00 (ddd, J = 3.1, 3.1, 1.1 Hz, 1H), 3.59 (ddd, J = 3.6, 2.4, 1.1 Hz, 1H), 3.37 (dd, J = 18.5, 3.7 Hz, 1H), 3.03 (ddd, J = 6.8, 1.5, 1.5 Hz, 2H), 2.97 (ddd, J = 6.7, 1.5, 1.5 Hz, 2H), 2.89 (dd, J = 18.5, 2.6 Hz, 1H), 2.75 (dd, J = 18.4, 2.9 Hz, 1H), 2.58 (s, 1H), 2.47 (dd, J = 17.5, 3.6 Hz, 1H), 2.46 (dd, J = 17.7, 3.3 Hz, 1H), 2.29 – 2.25 (m, 1H), 2.21 (dd, J = 14.6, 2.1 Hz, 1H), 2.19 (dd, J = 18.4, 3.2 Hz, 1H), 2.09 (dd, J = 14.0, 6.0 Hz, 1H), 1.97 (dd, J = 17.7, 2.8 Hz, 1H), 1.91 (dd, J = 17.5, 2.5 Hz, 1H), 1.58 (d, J = 14.0 Hz, 1H), 1.52 – 1.44 (m, 1H).


204.82
203.54
155.88
155.70
137.35
137.28
135.65
134.79
132.87
132.71
132.33
131.15
128.06 C6D6
122.86
122.17
116.25
116.16
103.53
103.51
98.08
97.46
88.77
88.40
84.62
81.33
53.37
52.54
46.26
45.69
40.28
39.91
39.55
39.49
39.15
38.34
O
O
O
Br
OH
CH2
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
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50
40
30
20
10
0
f1 (ppm)

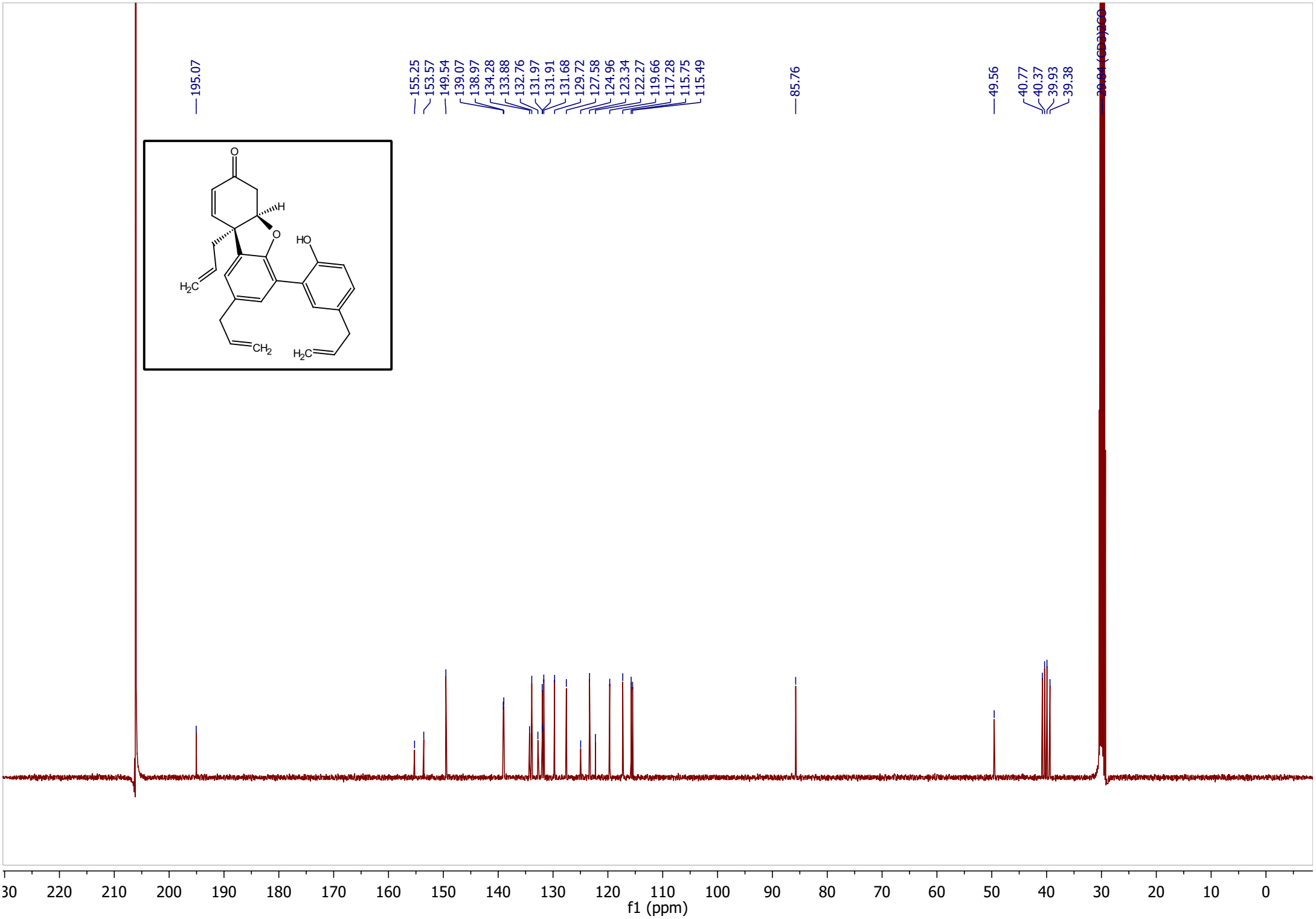


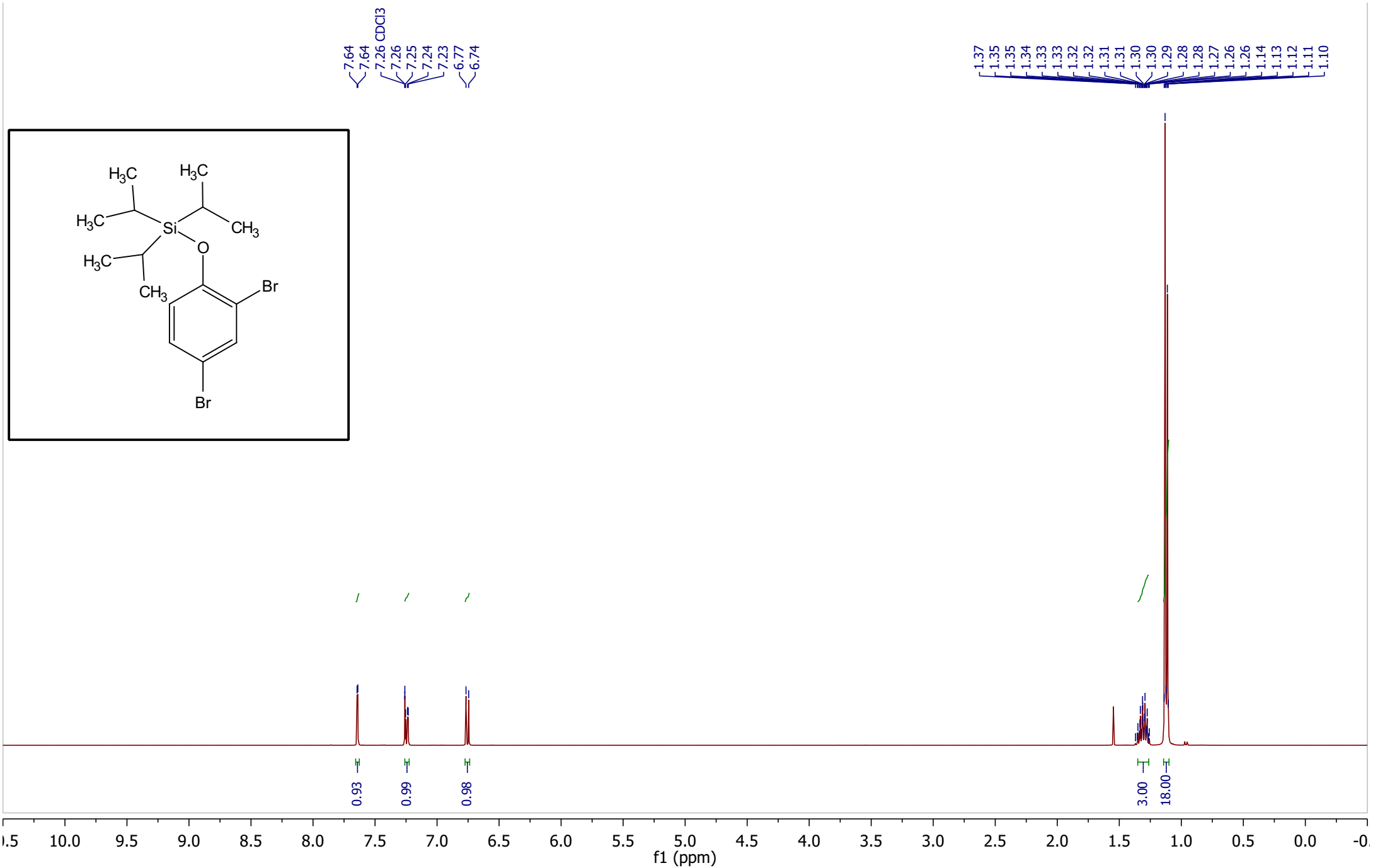
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.17 (d, *J* = 1.6 Hz, 1H), 6.94 (d, *J* = 1.6 Hz, 1H), 6.47 (dd, *J* = 10.2, 1.8 Hz, 1H), 6.03 (dd, *J* = 10.2, 0.8 Hz, 1H), 5.90 (dddd, *J* = 16.9, 10.4, 6.7, 6.7 Hz, 1H), 5.76 (dddd, *J* = 16.9, 10.2, 8.0, 6.8 Hz, 1H), 5.25 – 5.04 (m, 4H), 4.91 (ddd, *J* = 4.5, 2.8, 1.8 Hz, 1H), 3.32 (d, *J* = 6.7 Hz, 2H), 3.09 (ddd, *J* = 17.7, 2.8, 0.8 Hz, 1H), 2.82 – 2.70 (m, 2H), 2.64 (dd, *J* = 14.2, 8.0 Hz, 1H).


194.81
154.55
147.92
136.98
135.30
132.53
132.45
131.80
127.90
122.19
120.24
116.59
103.48
85.40
77.16 CDCl3
49.54
40.93
39.48
38.61
O
H
O
H2C
Br
CH2
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

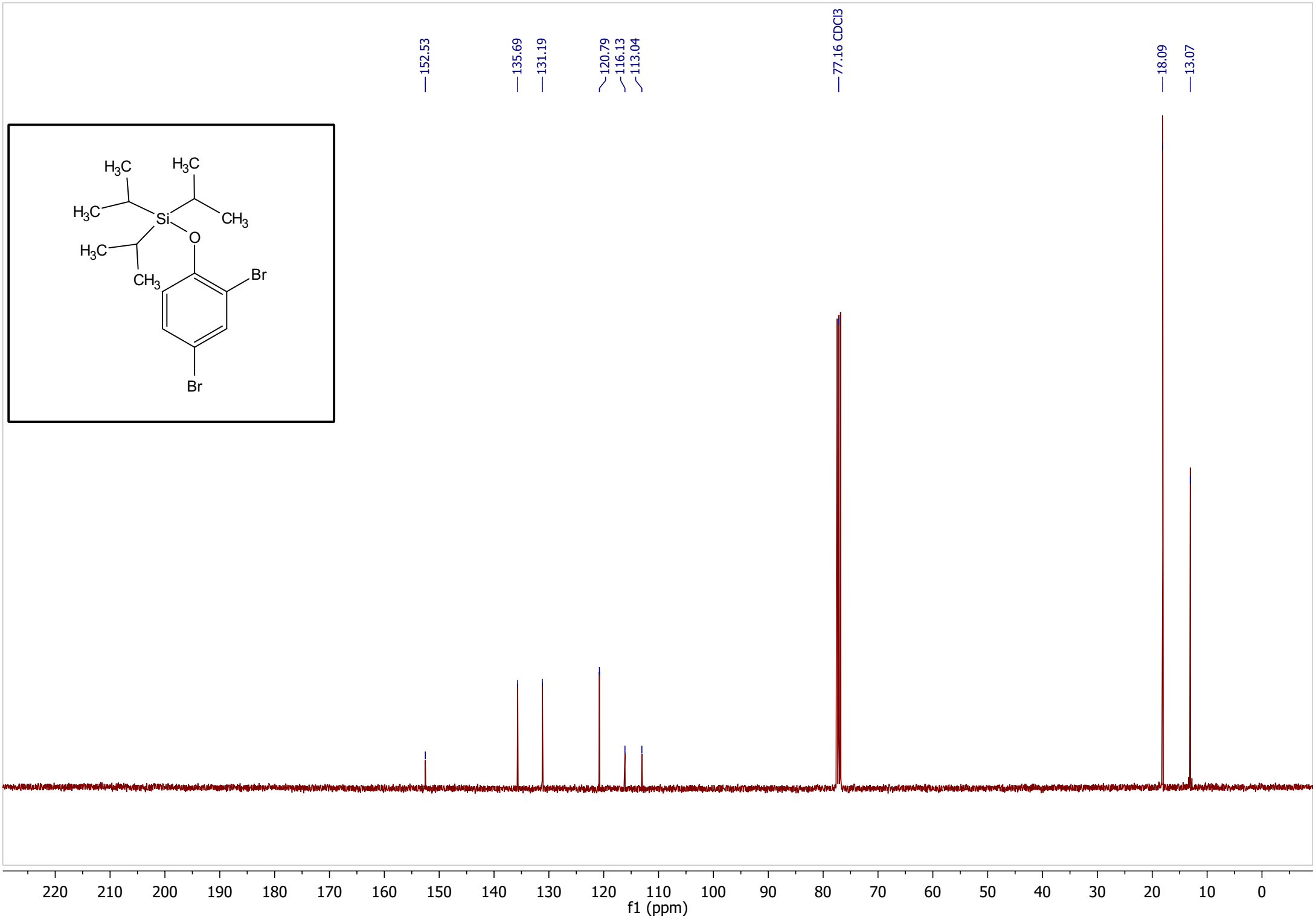


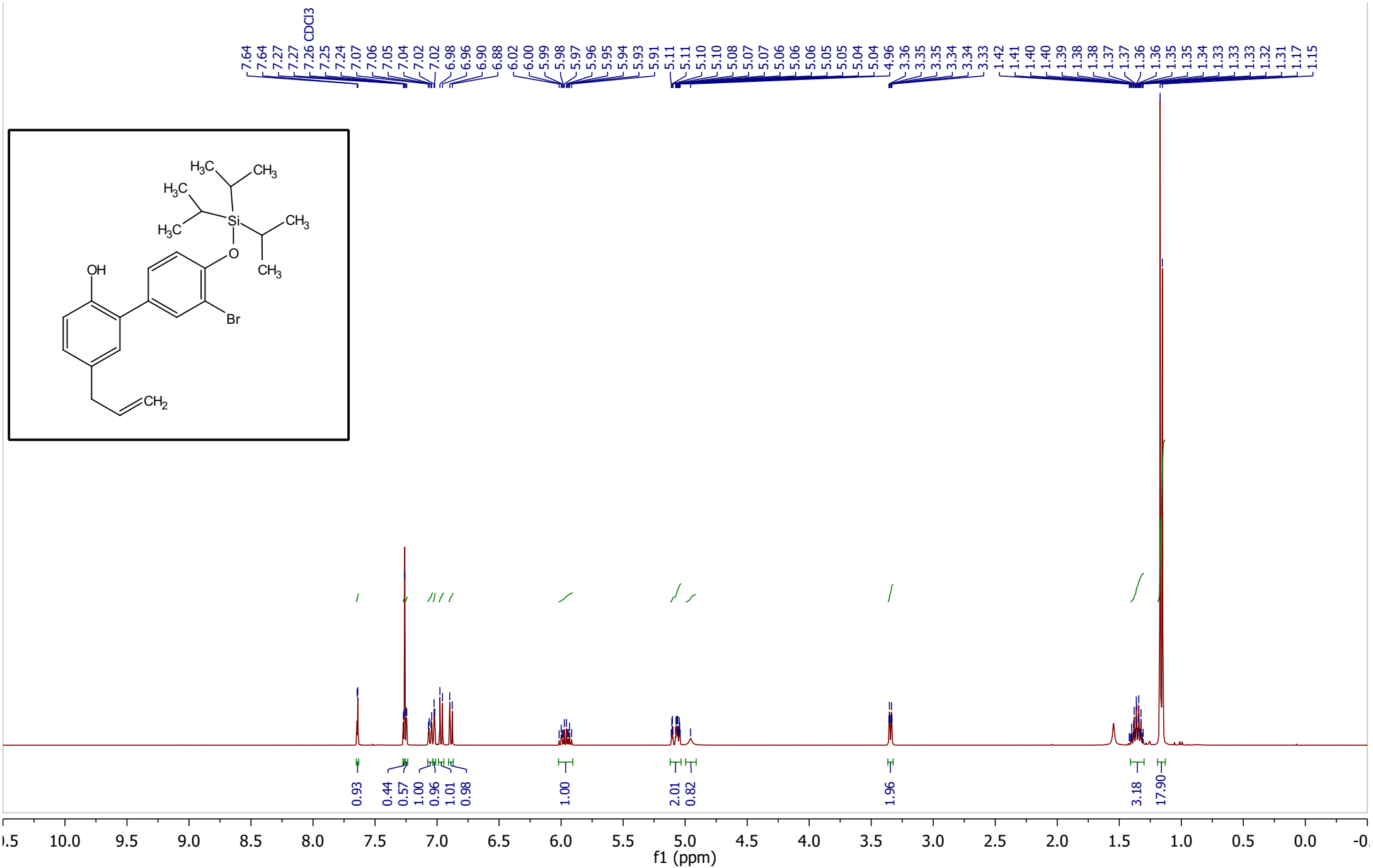
^{1}H NMR (400 MHz, Acetone-d_6) δ 7.65 (s, 1H), 7.23 (d, J = 1.8 Hz, 1H), 7.13 (d, J = 1.8 Hz, 1H), 7.08 (d, J = 2.3 Hz, 1H), 7.01 (dd, J = 8.3, 2.3 Hz, 1H), 6.86 (d, J = 8.3 Hz, 1H), 6.71 (dd, J = 10.3, 1.9 Hz, 1H), 6.07 – 5.84 (m, 3H), 5.93 (dd, J = 10.3, 0.6 Hz, 1H), 5.32 – 5.16 (m, 2H), 5.14 – 4.96 (m, 5H), 3.40 (ddd, J = 6.8, 1.5, 1.5 Hz, 2H), 3.31 (ddd, J = 6.8, 1.5, 1.5 Hz, 2H), 2.97 (dddd, J = 14.1, 7.1, 1.3, 1.3 Hz, 1H), 2.92 – 2.83 (m, 2H), 2.80 – 2.74 (m, 1H).


195.07
155.25
153.57
149.54
139.07
138.97
134.28
133.88
132.76
131.97
131.91
131.68
129.72
127.58
124.96
123.34
122.27
119.66
117.28
115.75
115.49
85.76
49.56
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40.37
39.93
39.38
O
H
O
HO
H2C
CH2
H2C
f1 (ppm)

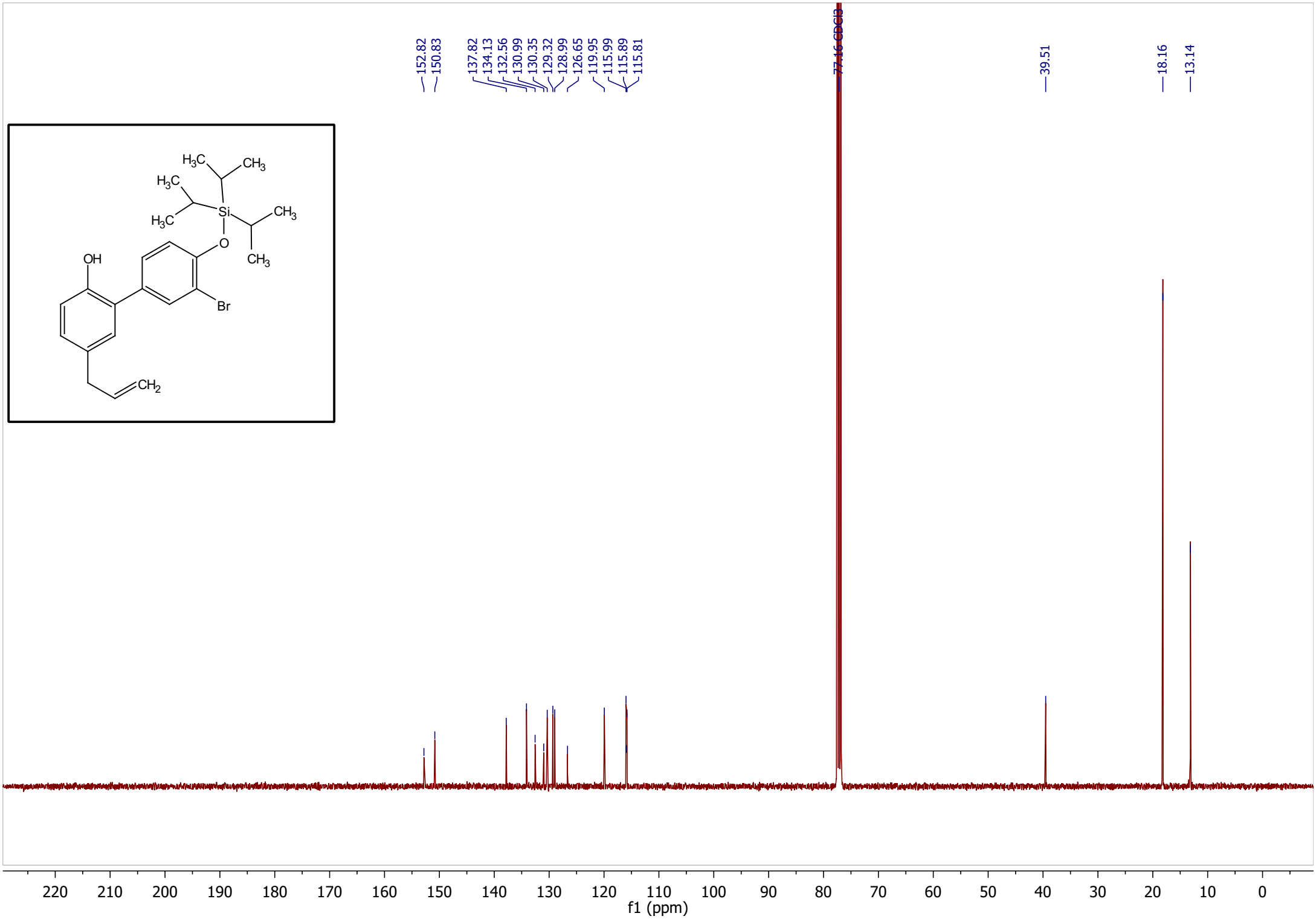


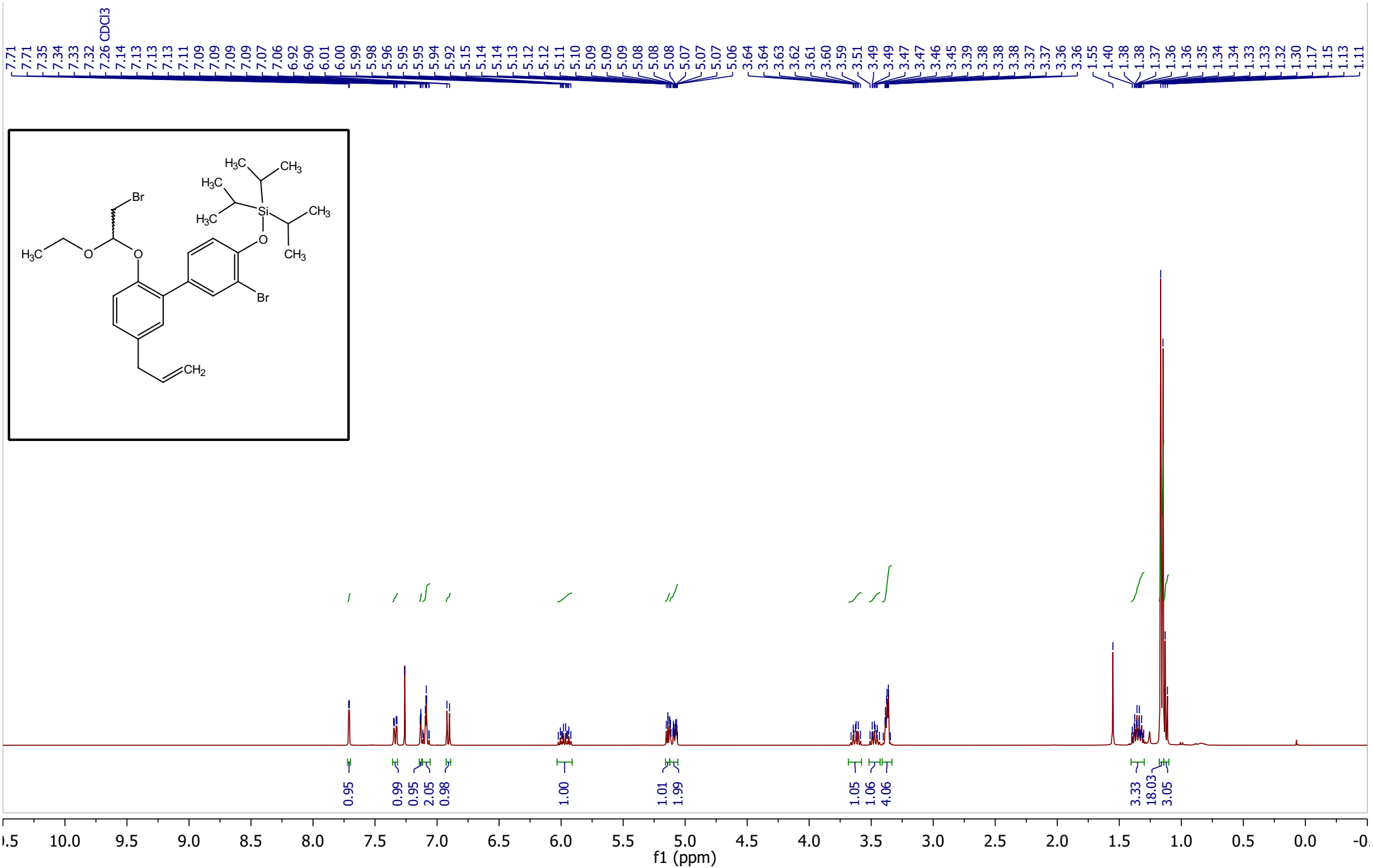
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.64 (d, *J* = 2.5 Hz, 1H), 7.25 (dd, *J* = 8.7, 2.5 Hz, 1H), 6.76 (d, *J* = 8.7 Hz, 1H), 1.35 – 1.25 (m, 3H), 1.12 (d, *J* = 7.4 Hz, 18H).


152.53
135.69
131.19
120.79
116.13
113.04
77.16 CDCl3
18.09
13.07
H3C
H3C
H3C
Si
CH3
H3C
O
CH3
Br
Br
220
210
200
190
180
170
160
150
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130
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110
100
90
80
70
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50
40
30
20
10
0
f1 (ppm)

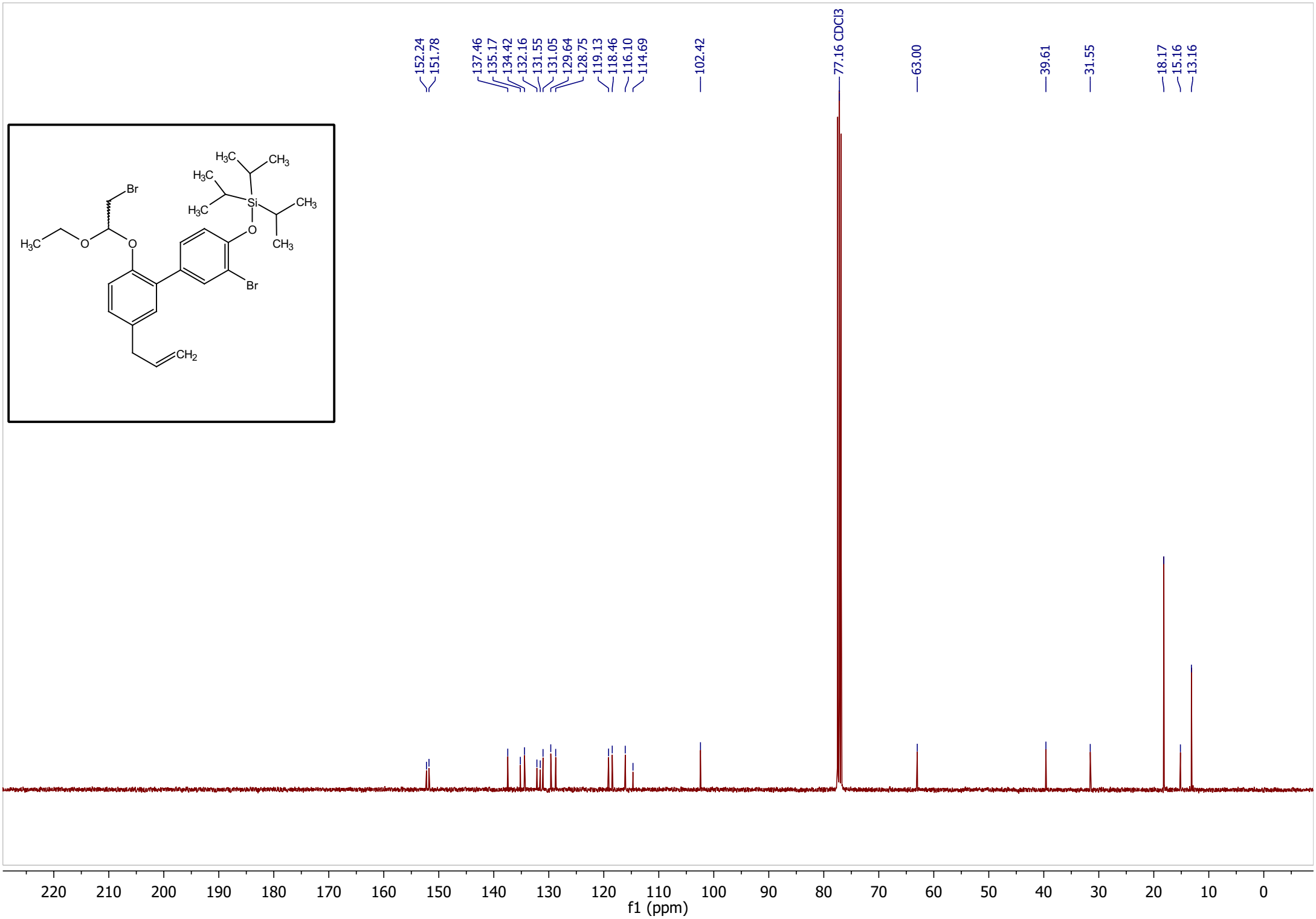


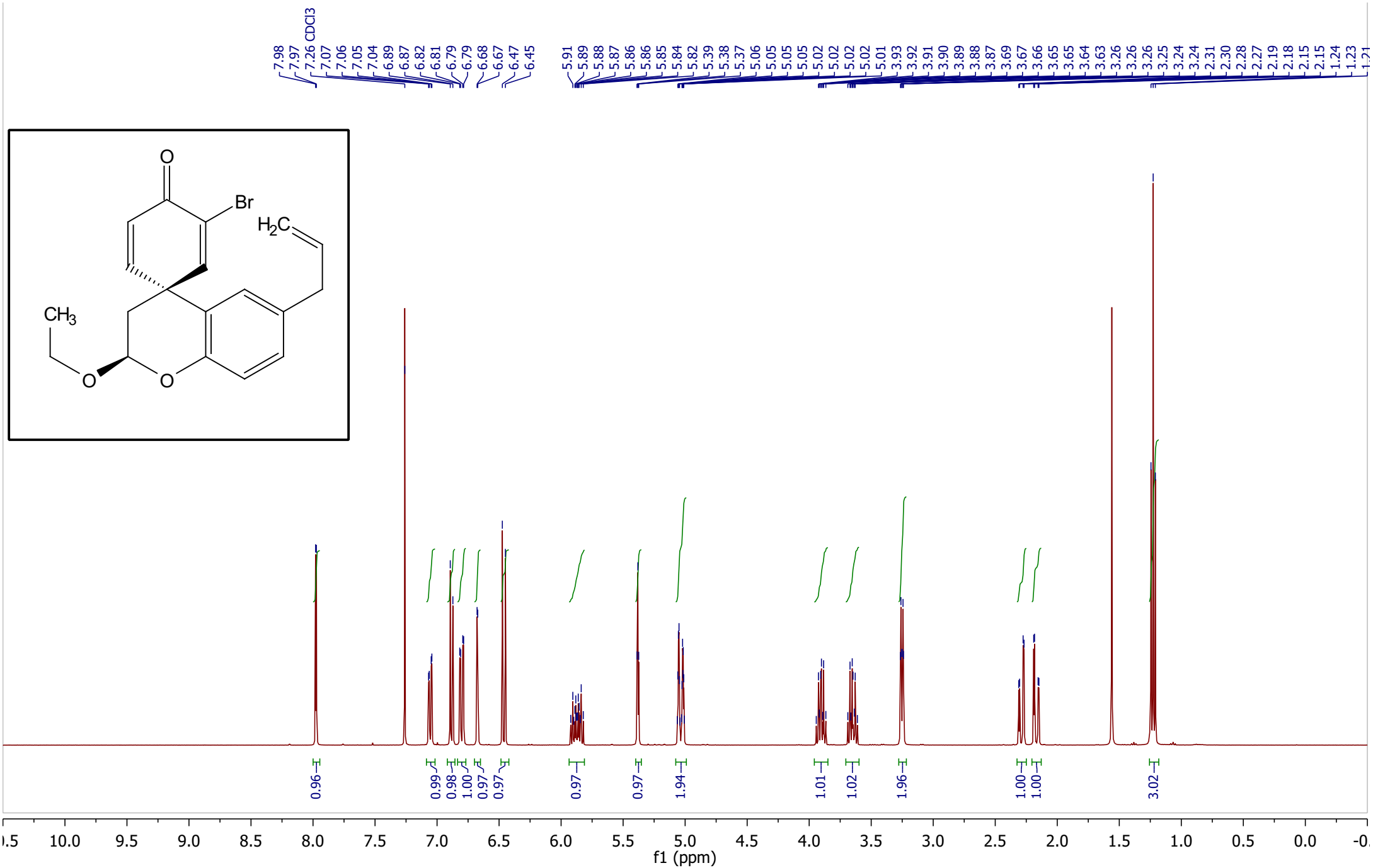
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.64 (d, *J* = 2.2 Hz, 1H), 7.26 (dd, *J* = 8.3, 2.2 Hz, 1H), 7.06 (dd, *J* = 8.2, 2.3 Hz, 1H), 7.02 (d, *J* = 2.2 Hz, 1H), 6.97 (d, *J* = 8.3 Hz, 1H), 6.89 (d, *J* = 8.2 Hz, 1H), 5.97 (ddt, *J* = 16.8, 10.0, 6.7 Hz, 1H), 5.12 – 5.03 (m, 2H), 4.96 (s, 1H), 3.34 (ddd, *J* = 6.7, 1.5, 1.5 Hz, 2H), 1.42 – 1.30 (m, 3H), 1.16 (d, *J* = 7.3 Hz, 18H).


152.82
150.83
137.82
134.13
132.56
130.99
130.35
129.32
128.99
126.65
119.95
115.99
115.89
115.81
77.16 CDCl3
39.51
18.16
13.14
H3C
CH3
H3C
Si
CH3
H3C
O
CH3
OH
Br
CH2
220
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200
190
180
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70
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50
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10
0
f1 (ppm)

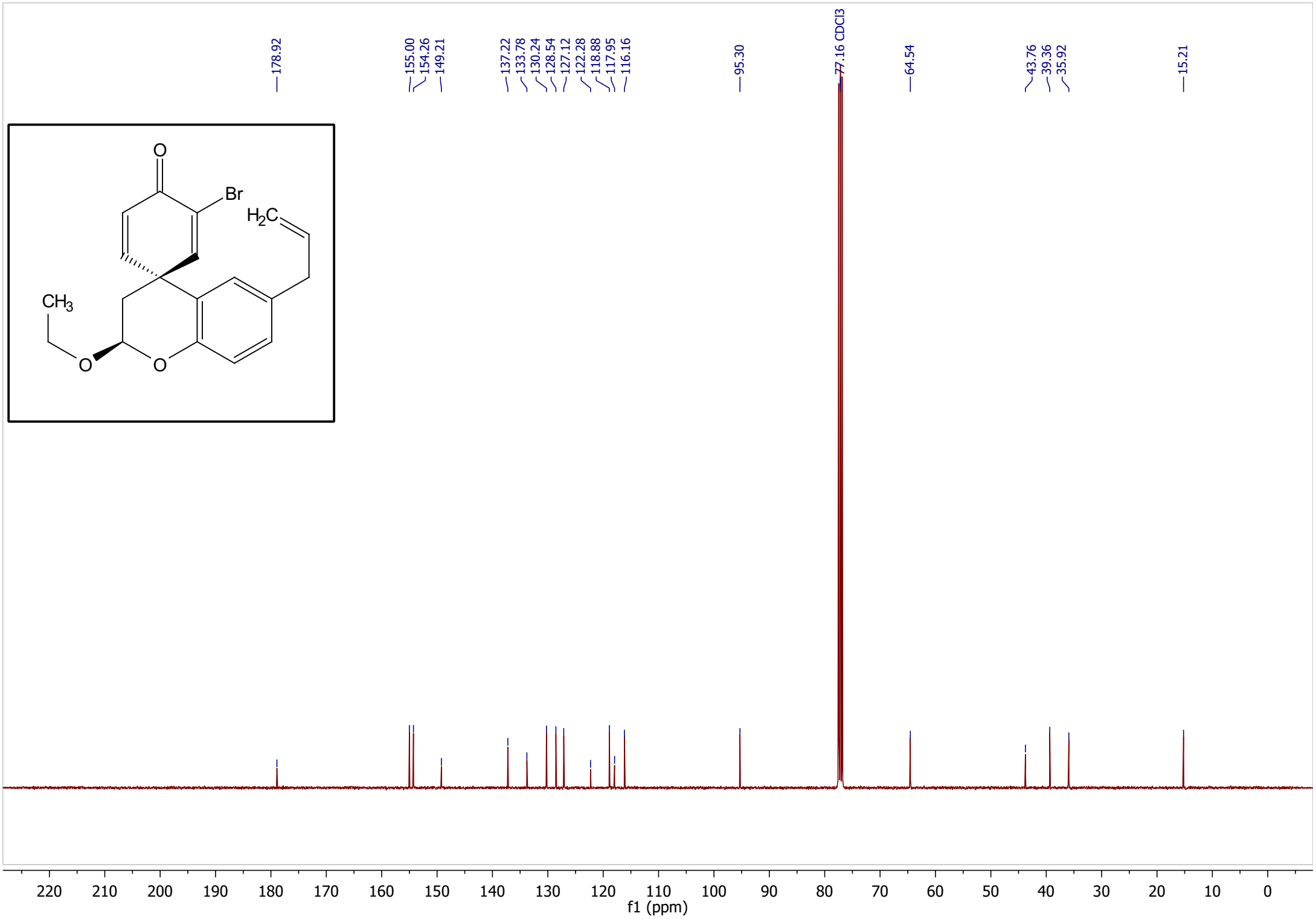


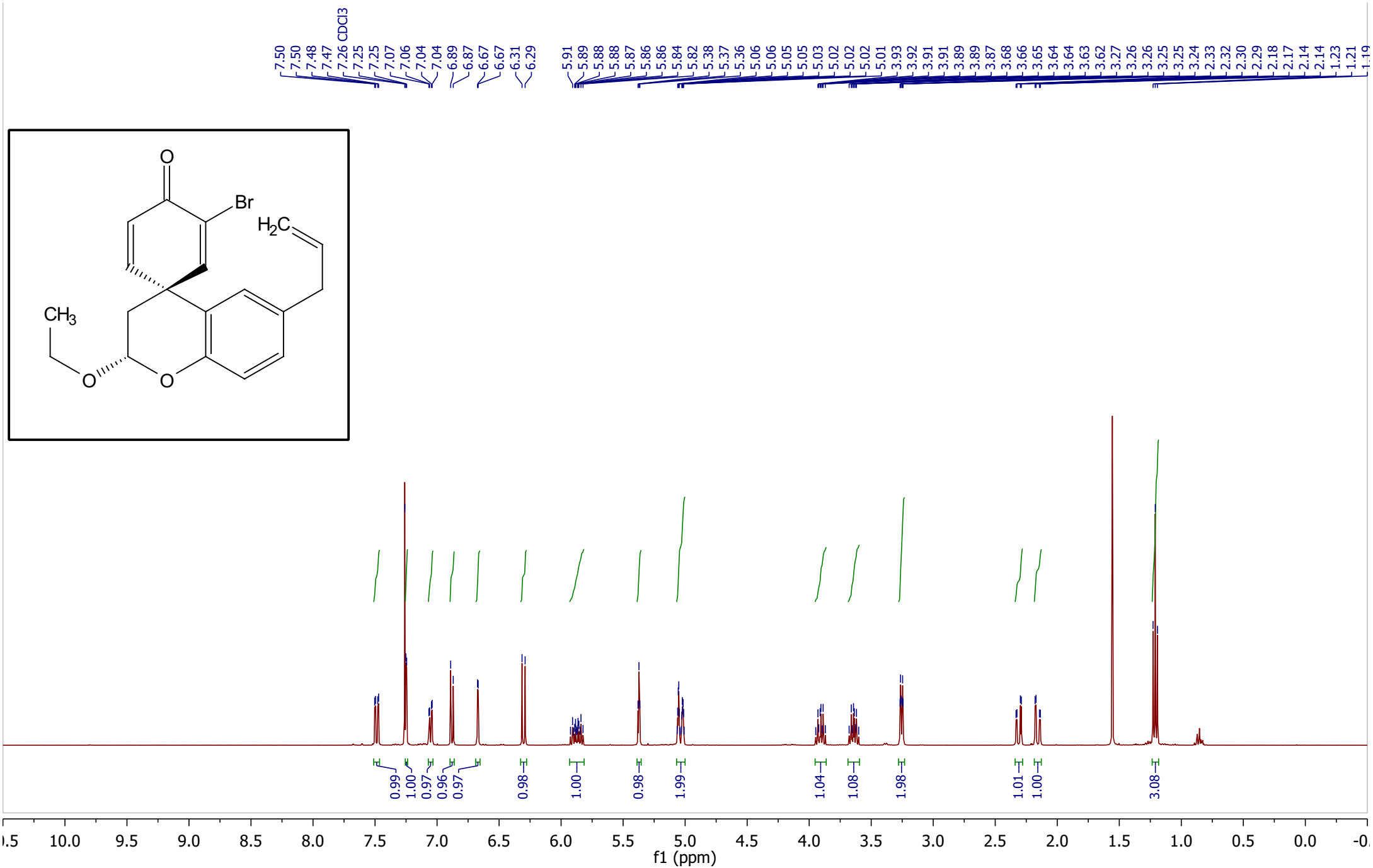
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.71 (d, *J* = 2.2 Hz, 1H), 7.34 (dd, *J* = 8.4, 2.2 Hz, 1H), 7.15 – 7.12 (m, 1H), 7.12 – 7.06 (m, 2H), 6.91 (d, *J* = 8.4 Hz, 1H), 5.97 (dddd, *J* = 16.8, 10.1, 6.7, 6.7 Hz, 1H), 5.16 – 5.12 (m, 1H), 5.12 – 5.05 (m, 2H), 3.62 (dq, *J* = 9.3, 7.0 Hz, 1H), 3.47 (dq, *J* = 9.3, 7.0 Hz, 1H), 3.40 – 3.35 (m, 4H), 1.40 – 1.31 (m, 3H), 1.16 (d, *J* = 7.5 Hz, 18H), 1.13 (dd, *J* = 7.0, 7.0 Hz, 3H).


152.24
151.78
137.46
135.17
134.42
132.16
131.55
131.05
129.64
128.75
119.13
118.46
116.10
114.69
102.42
77.16 CDCl3
63.00
39.61
31.55
18.17
15.16
13.16
H3C
CH3
H3C
H3C
Si
CH3
O
CH3
Br
H3C
O
O
Br
CH2
220
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200
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0
f1 (ppm)

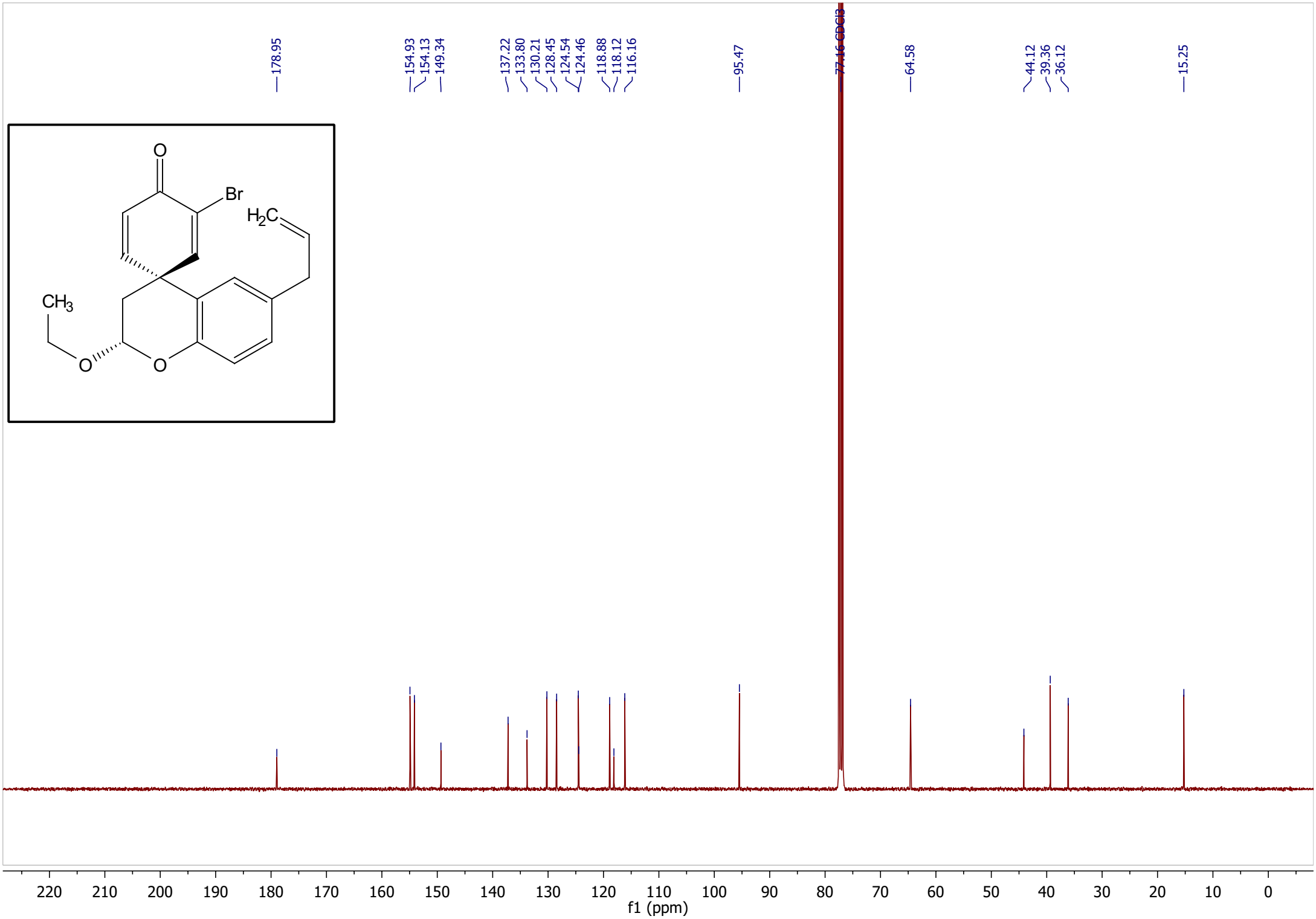


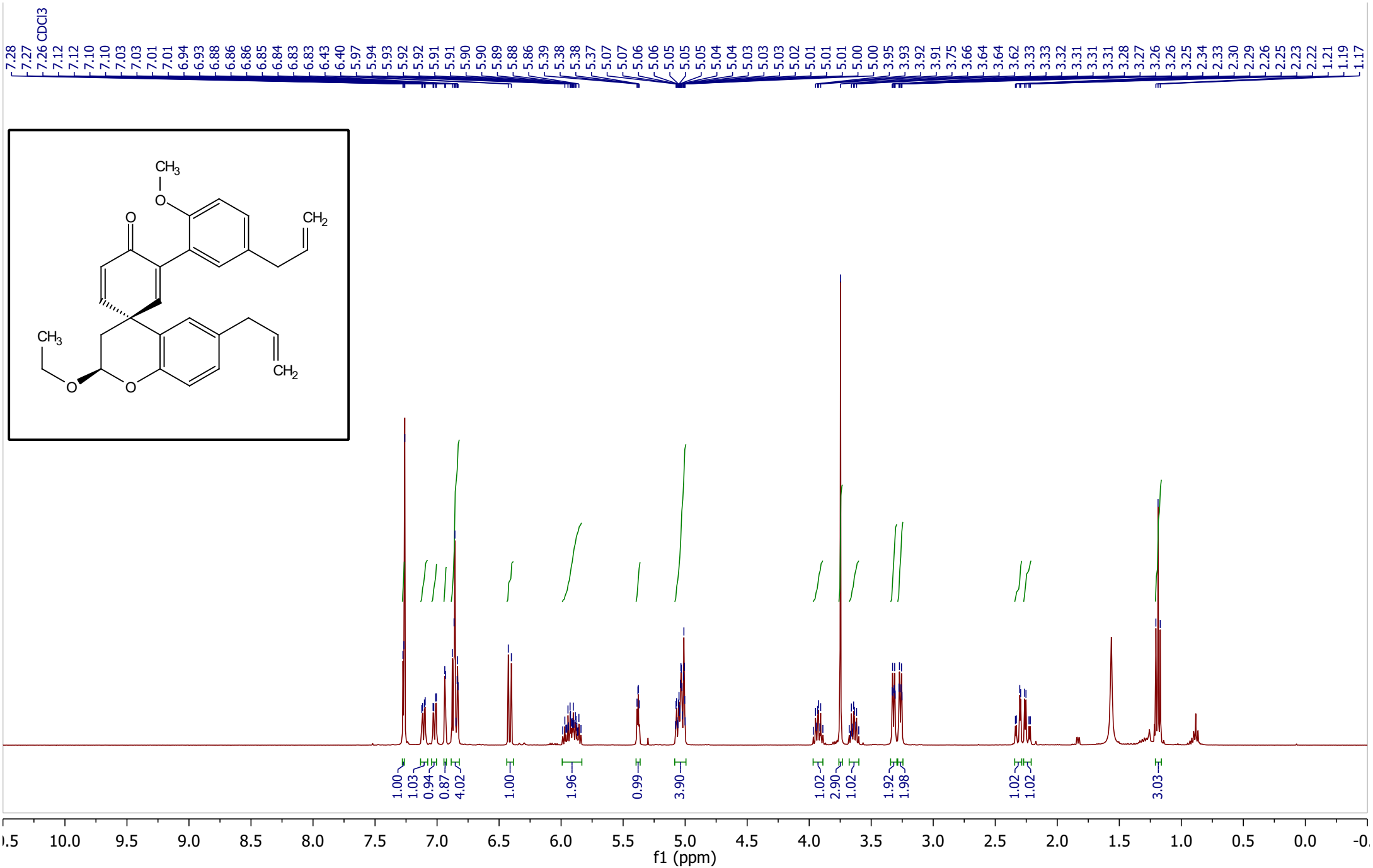
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.98 (d, *J* = 2.7 Hz, 1H), 7.05 (dd, *J* = 8.4, 2.2 Hz, 1H), 6.88 (d, *J* = 8.4 Hz, 1H), 6.80 (dd, *J* = 9.8, 2.7 Hz, 1H), 6.68 (d, *J* = 2.2 Hz, 1H), 6.46 (d, *J* = 9.8 Hz, 1H), 5.93 – 5.82 (m, 1H), 5.38 (dd, *J* = 2.8, 2.8 Hz, 1H), 5.06 – 5.00 (m, 2H), 3.90 (dq, *J* = 9.7, 7.1 Hz, 1H), 3.65 (dq, *J* = 9.7, 7.1 Hz, 1H), 3.25 (ddd, *J* = 6.7, 1.4, 1.4 Hz, 2H), 2.29 (dd, *J* = 14.1, 2.9 Hz, 1H), 2.17 (dd, *J* = 14.1, 2.7 Hz, 1H), 1.23 (dd, *J* = 7.1, 7.1 Hz, 3H).


178.92
155.00
154.26
149.21
137.22
133.78
130.24
128.54
127.12
122.28
118.88
117.95
116.16
95.30
77.16 CDCl3
64.54
43.76
39.36
35.92
15.21
O
Br
H2C
CH3
O
O
220
210
200
190
180
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90
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70
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10
0
f1 (ppm)

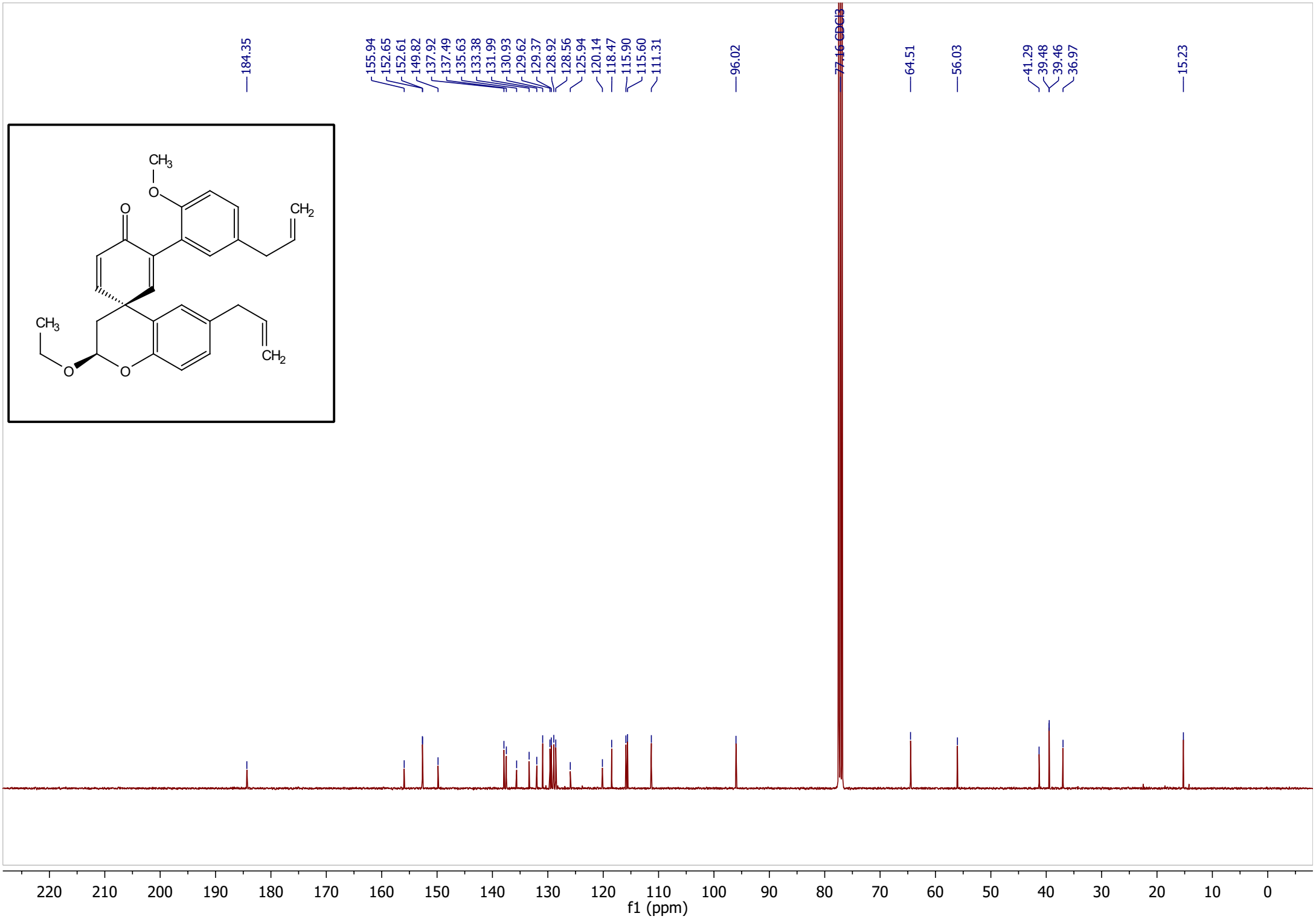


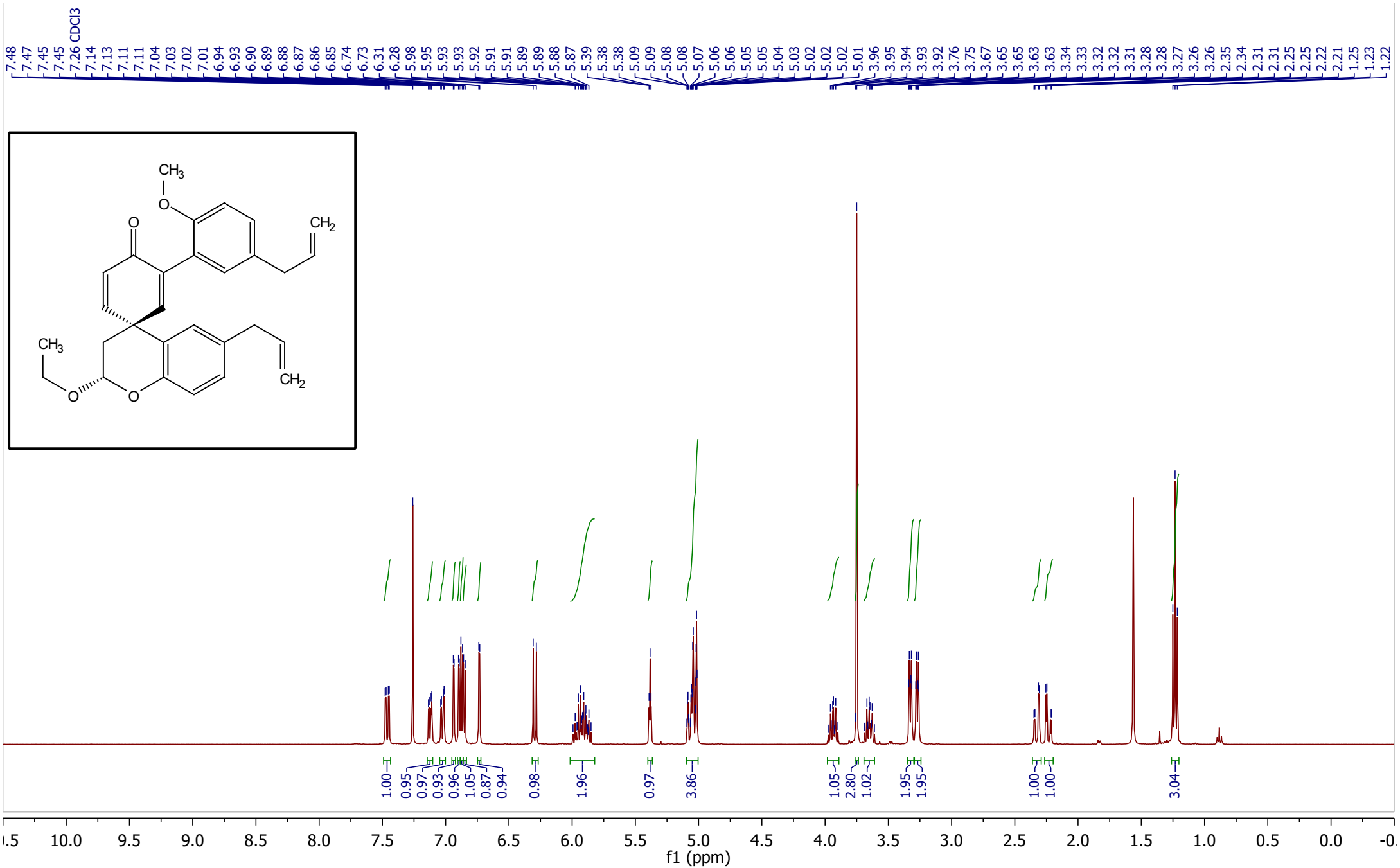
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.49 (dd, *J* = 10.0, 2.7 Hz, 1H), 7.25 (d, *J* = 2.7 Hz, 1H), 7.05 (dd, *J* = 8.3, 2.2 Hz, 1H), 6.88 (d, *J* = 8.3 Hz, 1H), 6.67 (d, *J* = 2.2 Hz, 1H), 6.30 (d, *J* = 10.0 Hz, 1H), 5.92 – 5.81 (m, 1H), 5.37 (dd, *J* = 3.1, 3.1 Hz, 1H), 5.06 – 5.00 (m, 2H), 3.91 (dq, *J* = 9.7, 7.1 Hz, 1H), 3.64 (dq, *J* = 9.7, 7.1 Hz, 1H), 3.25 (ddd, *J* = 6.8, 1.4, 1.4 Hz, 2H), 2.31 (dd, *J* = 14.0, 3.0 Hz, 1H), 2.16 (dd, *J* = 14.0, 3.2 Hz, 1H), 1.21 (dd, *J* = 7.1, 7.1 Hz, 3H).


178.95
154.93
154.13
149.34
137.22
133.80
130.21
128.45
124.54
124.46
118.88
118.12
116.16
95.47
77.16 CDCl3
64.58
44.12
39.36
36.12
15.25
O
Br
H2C
CH3
O
O
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

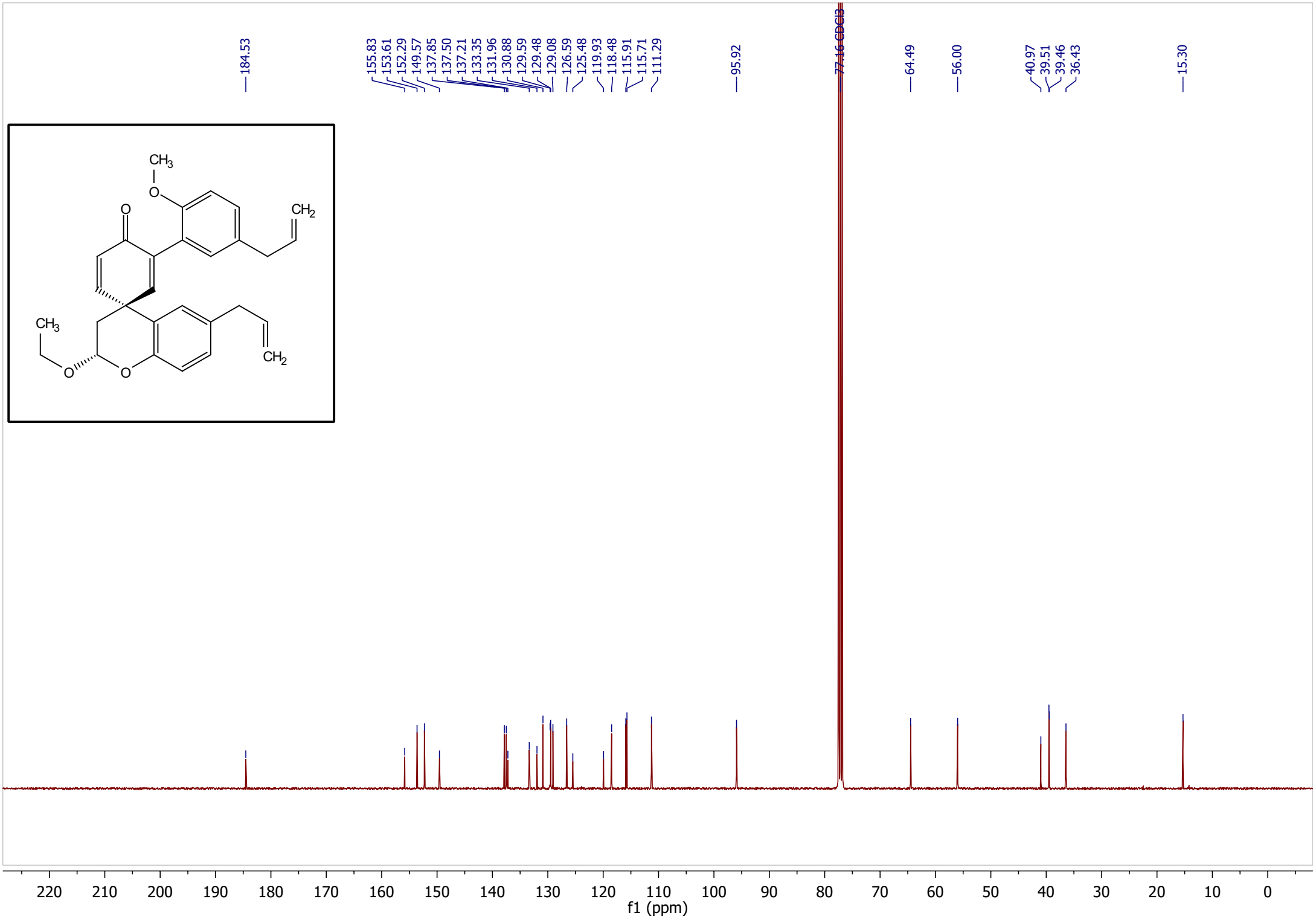


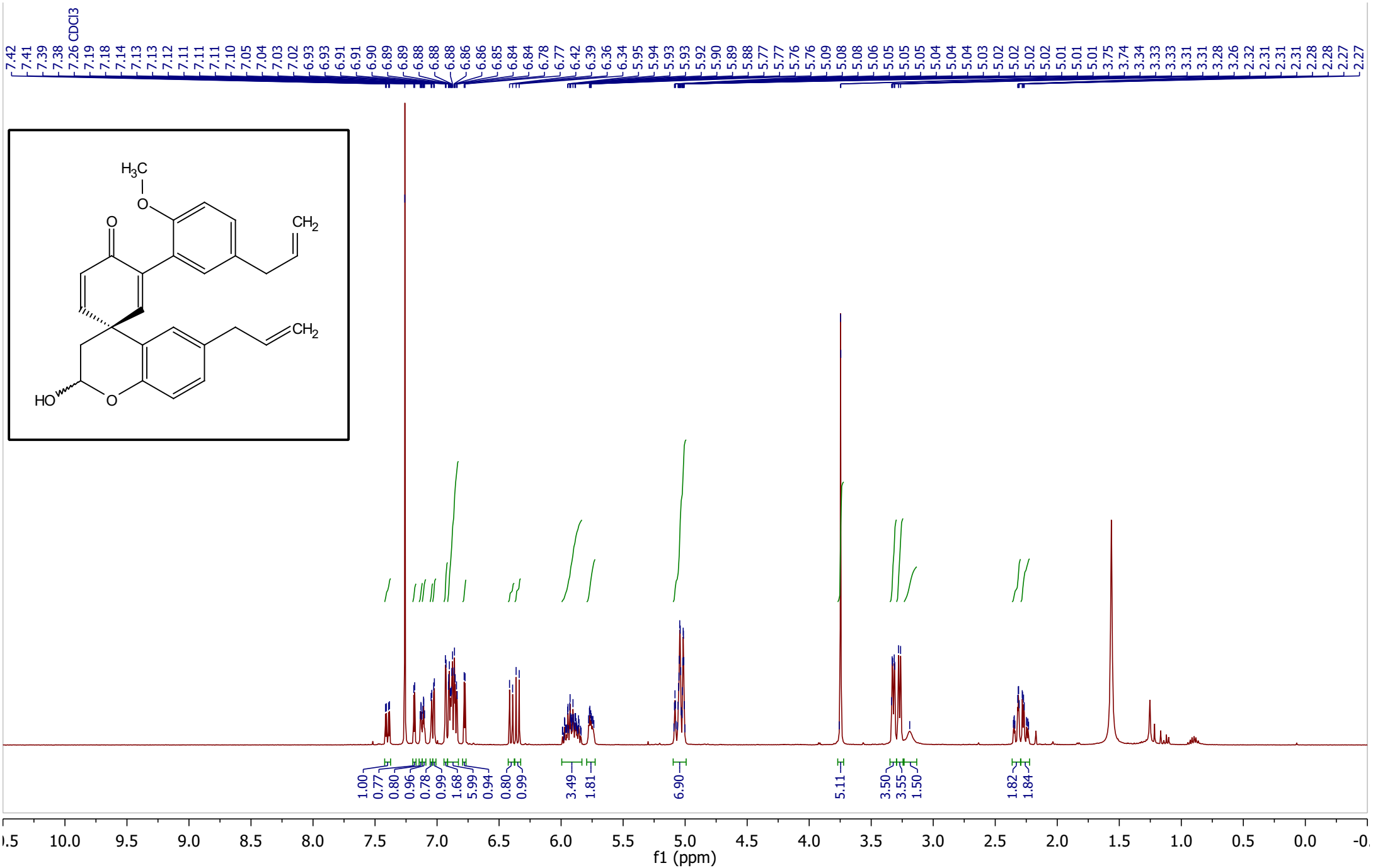
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.27 (d, *J* = 3.0 Hz, 1H), 7.11 (dd, *J* = 8.4, 2.4 Hz, 1H), 7.02 (dd, *J* = 8.4, 2.2 Hz, 1H), 6.93 (d, *J* = 2.3 Hz, 1H), 6.89 – 6.81 (m, 4H), 6.41 (d, *J* = 9.9 Hz, 1H), 6.00 – 5.82 (m, 2H), 5.38 (dd, *J* = 4.2, 2.9 Hz, 1H), 5.08 – 4.98 (m, 4H), 3.93 (dq, *J* = 9.7, 7.1 Hz, 1H), 3.75 (s, 3H), 3.64 (dq, *J* = 9.7, 7.1 Hz, 1H), 3.32 (ddd, *J* = 6.7, 1.5, 1.5 Hz, 2H), 3.27 (ddd, *J* = 6.6, 1.5, 1.5 Hz, 2H), 2.32 (dd, *J* = 14.0, 2.9 Hz, 1H), 2.24 (dd, *J* = 14.0, 4.2 Hz, 1H), 1.19 (dd, *J* = 7.1, 7.1 Hz, 3H).


184.35
155.94
152.65
152.61
149.82
137.92
137.49
135.63
133.38
131.99
130.93
129.62
129.37
128.92
128.56
125.94
120.14
118.47
115.90
115.60
111.31
96.02
77.16 CDCl3
64.51
56.03
41.29
39.48
39.46
36.97
15.23
CH3
O
O
CH2
CH3
O
O
CH2
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

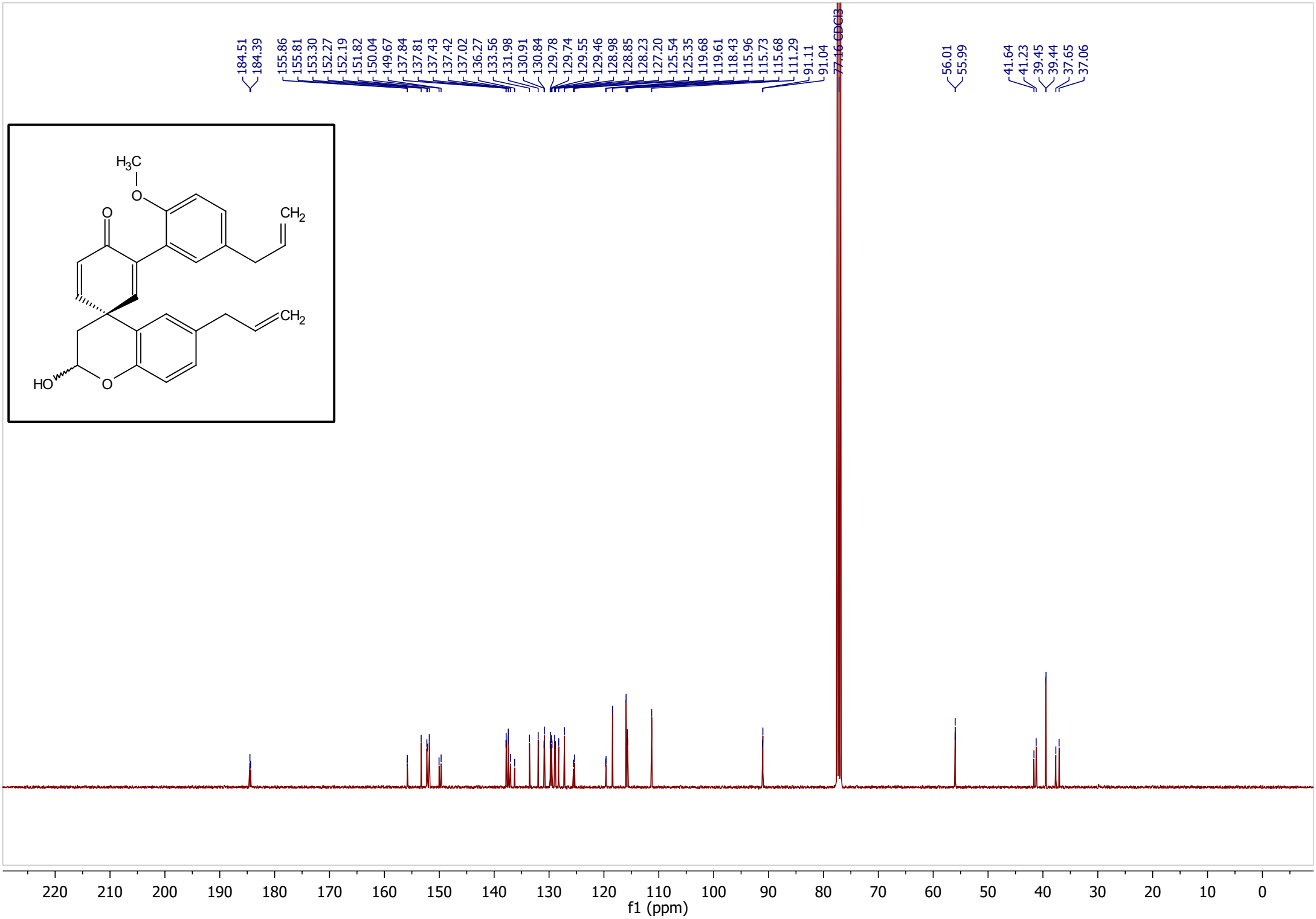


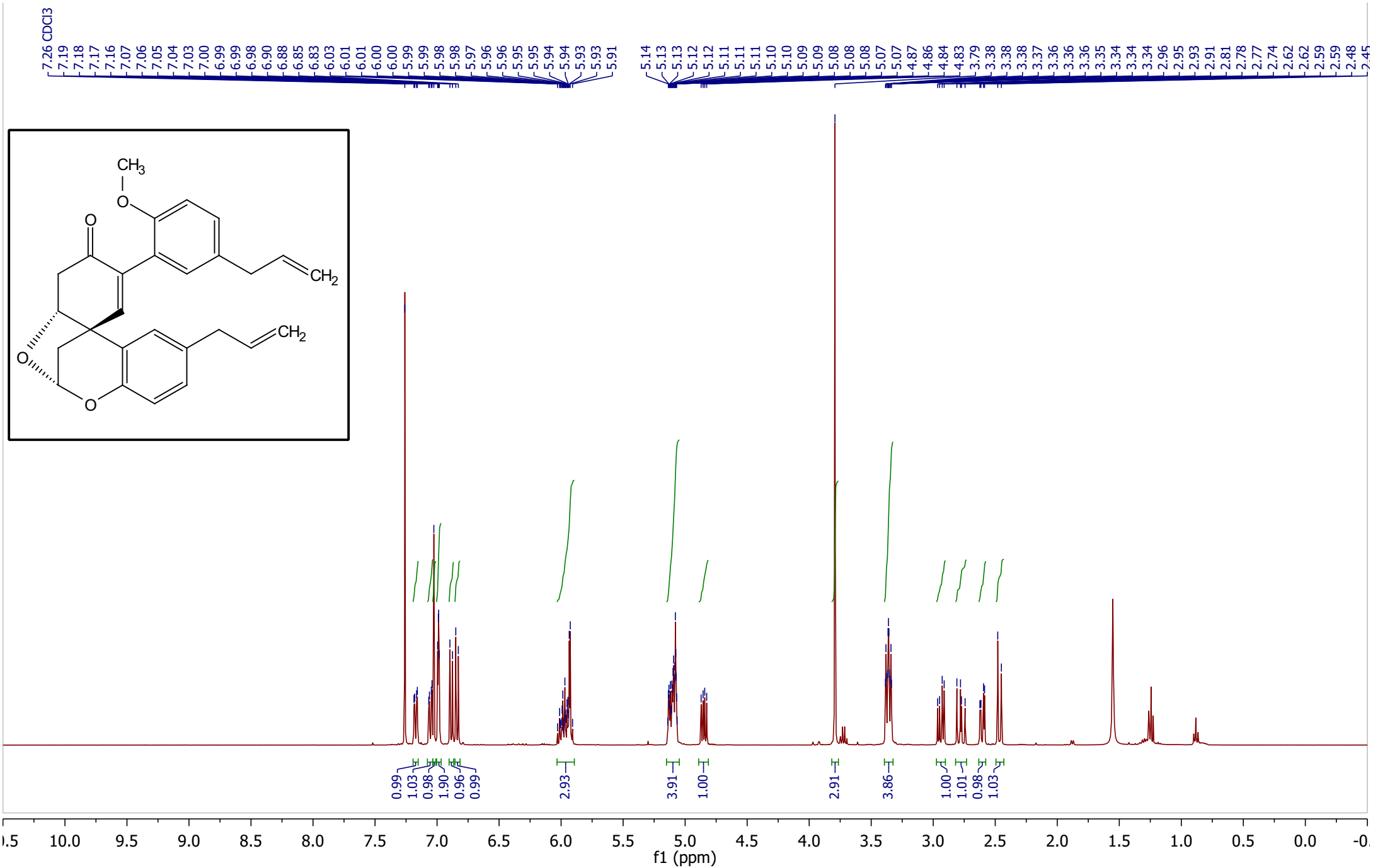
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.46 (dd, *J* = 10.1, 2.9 Hz, 1H), 7.12 (dd, *J* = 8.4, 2.3 Hz, 1H), 7.03 (dd, *J* = 8.3, 2.2 Hz, 1H), 6.94 (d, *J* = 2.3 Hz, 1H), 6.90 (d, *J* = 2.2 Hz, 1H), 6.87 (d, *J* = 8.3 Hz, 1H), 6.86 (d, *J* = 8.4 Hz, 1H), 6.73 (d, *J* = 2.9 Hz, 1H), 6.30 (d, *J* = 10.1 Hz, 1H), 6.00 – 5.84 (m, 2H), 5.38 (dd, *J* = 3.1, 3.1 Hz, 1H), 5.10 – 5.00 (m, 4H), 3.94 (dq, *J* = 9.6, 7.1 Hz, 1H), 3.75 (s, 3H), 3.65 (dq, *J* = 9.6, 7.1 Hz, 1H), 3.33 (ddd, *J* = 6.7, 1.5, 1.5 Hz, 2H), 3.27 (ddd, *J* = 6.7, 1.5, 1.5 Hz, 2H), 2.33 (dd, *J* = 14.1, 3.0 Hz, 1H), 2.23 (dd, *J* = 14.1, 3.2 Hz, 1H), 1.23 (dd, *J* = 7.1, 7.1 Hz, 3H).


184.53
155.83
153.61
152.29
149.57
137.85
137.50
137.21
133.35
131.96
130.88
129.59
129.48
129.08
126.59
125.48
119.93
118.48
115.91
115.71
111.29
95.92
77.16 CDCl3
64.49
56.00
40.97
39.51
39.46
36.43
15.30
CH3
O
O
CH2
CH3
O
O
CH2
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

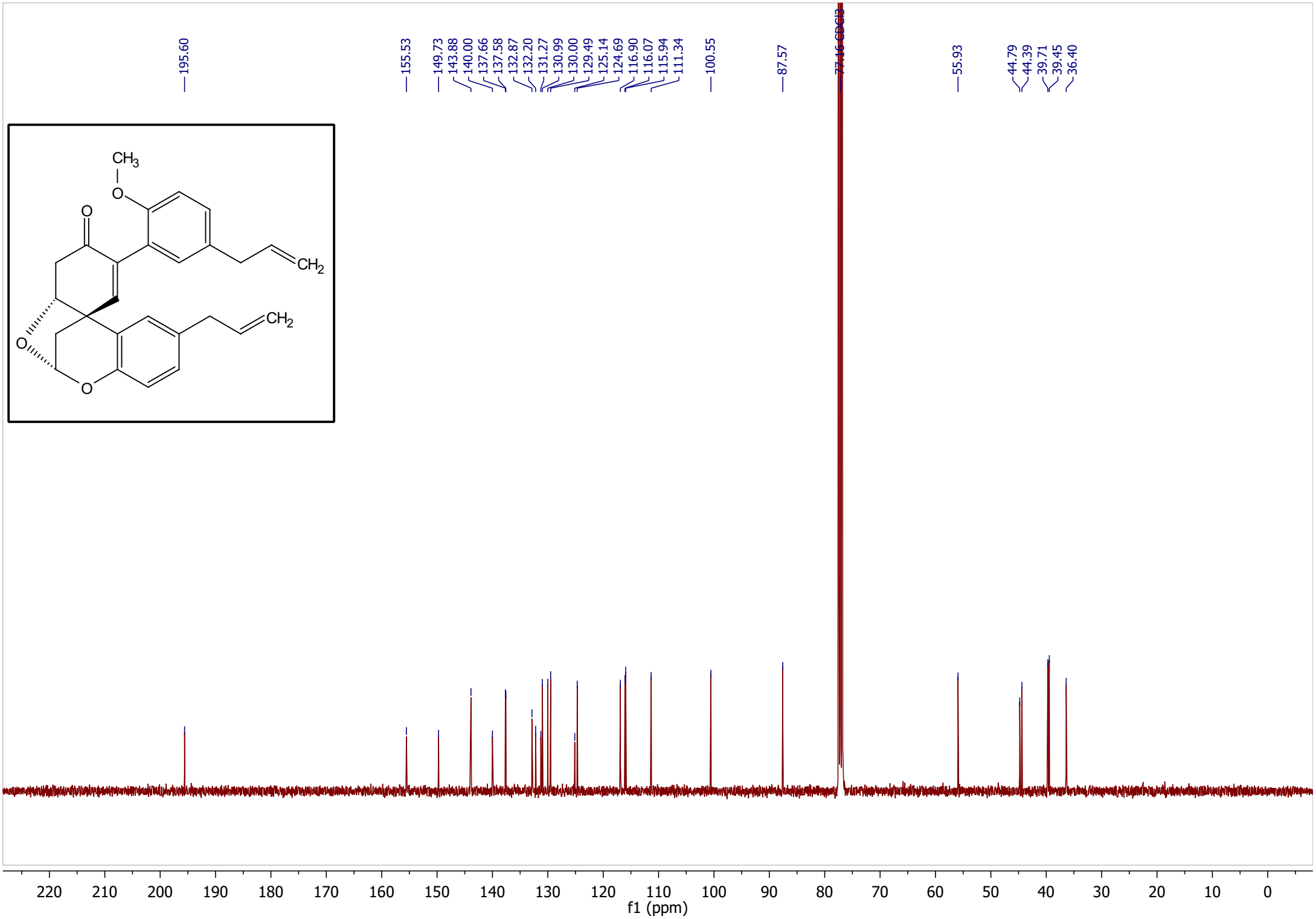


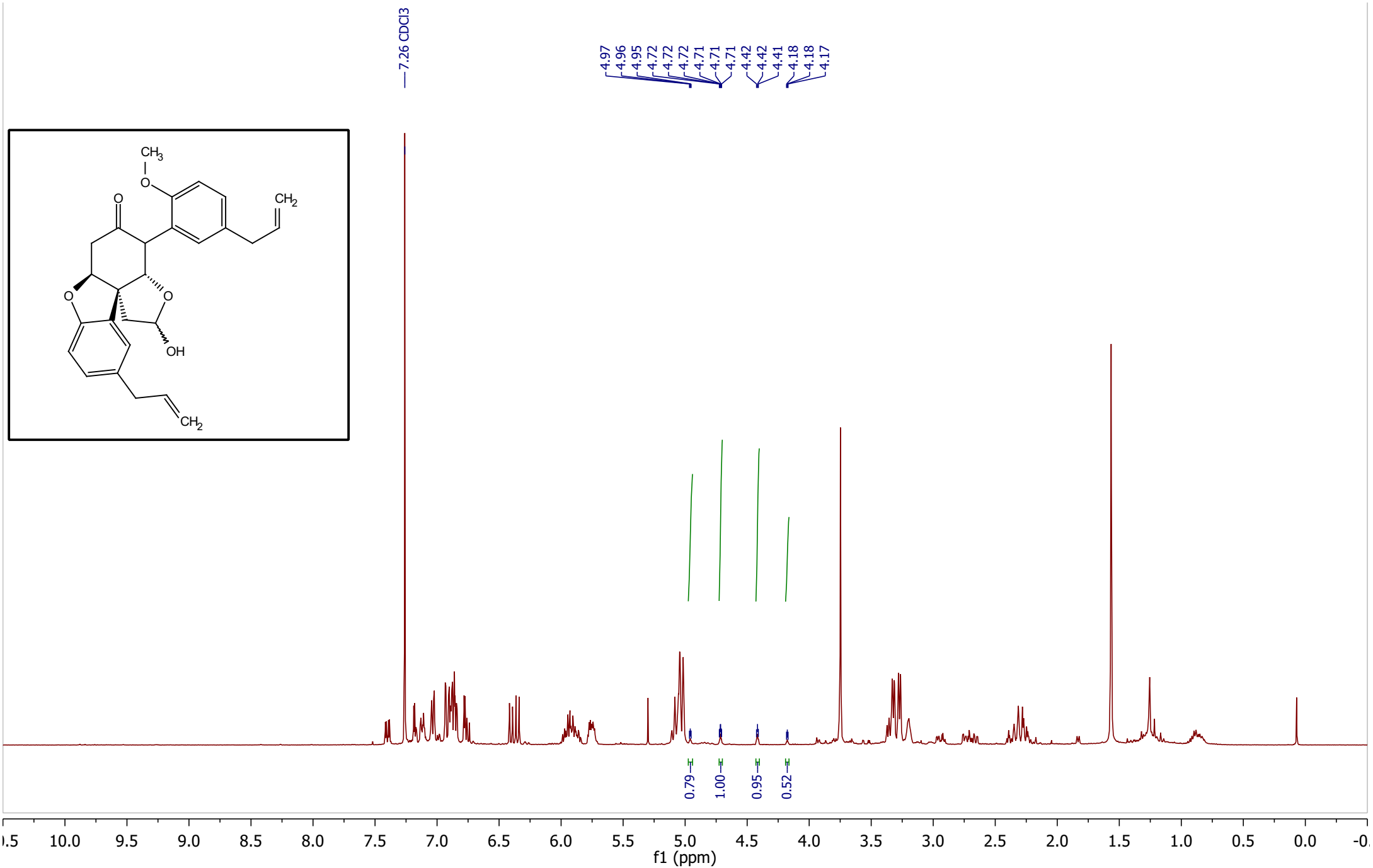
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.40 (dd, *J* = 10.0, 3.0 Hz, 1H), 7.18 (d, *J* = 3.0 Hz, 1H), 7.12 (dd, *J* = 8.3, 2.4 Hz, 1H), 7.11 (dd, *J* = 8.3, 2.4 Hz, 1H), 7.05 (d, *J* = 2.2 Hz, 1H), 7.02 (d, *J* = 2.3 Hz, 1H), 6.94 – 6.92 (m, 2H), 6.91 – 6.90 (m, 1H), 6.90 – 6.87 (m, 2H), 6.87 – 6.83 (m, 3H), 6.78 (d, *J* = 3.0 Hz, 1H), 6.40 (d, *J* = 9.9 Hz, 1H), 6.35 (d, *J* = 10.0 Hz, 1H), 6.00 – 5.83 (m, 4H), 5.79 – 5.72 (m, 2H), 5.10 – 4.98 (m, 8H), 3.75 (s, 3H), 3.74 (s, 3H), 3.35 – 3.30 (m, 4H), 3.30 – 3.24 (m, 4H), 3.19 (s, 2H), 2.37 – 2.30 (m, 2H), 2.29 – 2.22 (m, 2H).


184.51
184.39
155.86
155.81
153.30
152.27
152.19
151.82
150.04
149.67
137.84
137.81
137.43
137.42
137.02
136.27
133.56
131.98
130.91
130.84
129.78
129.74
129.55
129.46
128.98
128.85
128.23
127.20
125.54
125.35
119.68
119.61
118.43
115.96
115.73
115.68
111.29
91.11
91.04
77.16 CDCl3
56.01
55.99
41.64
41.23
39.45
39.44
37.65
37.06
H3C
O
O
CH2
CH2
HO
O
220
210
200
190
180
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110
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90
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50
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30
20
10
0
f1 (ppm)

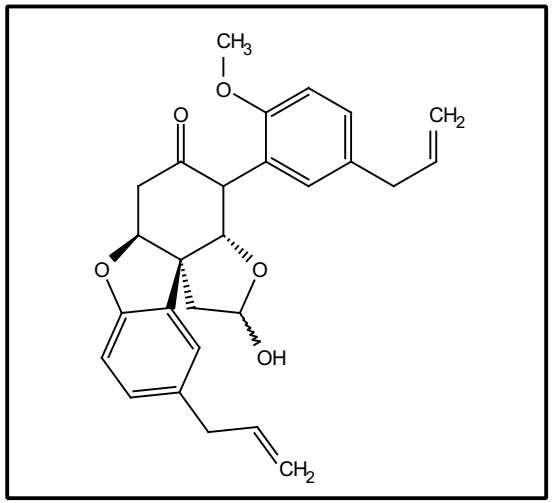


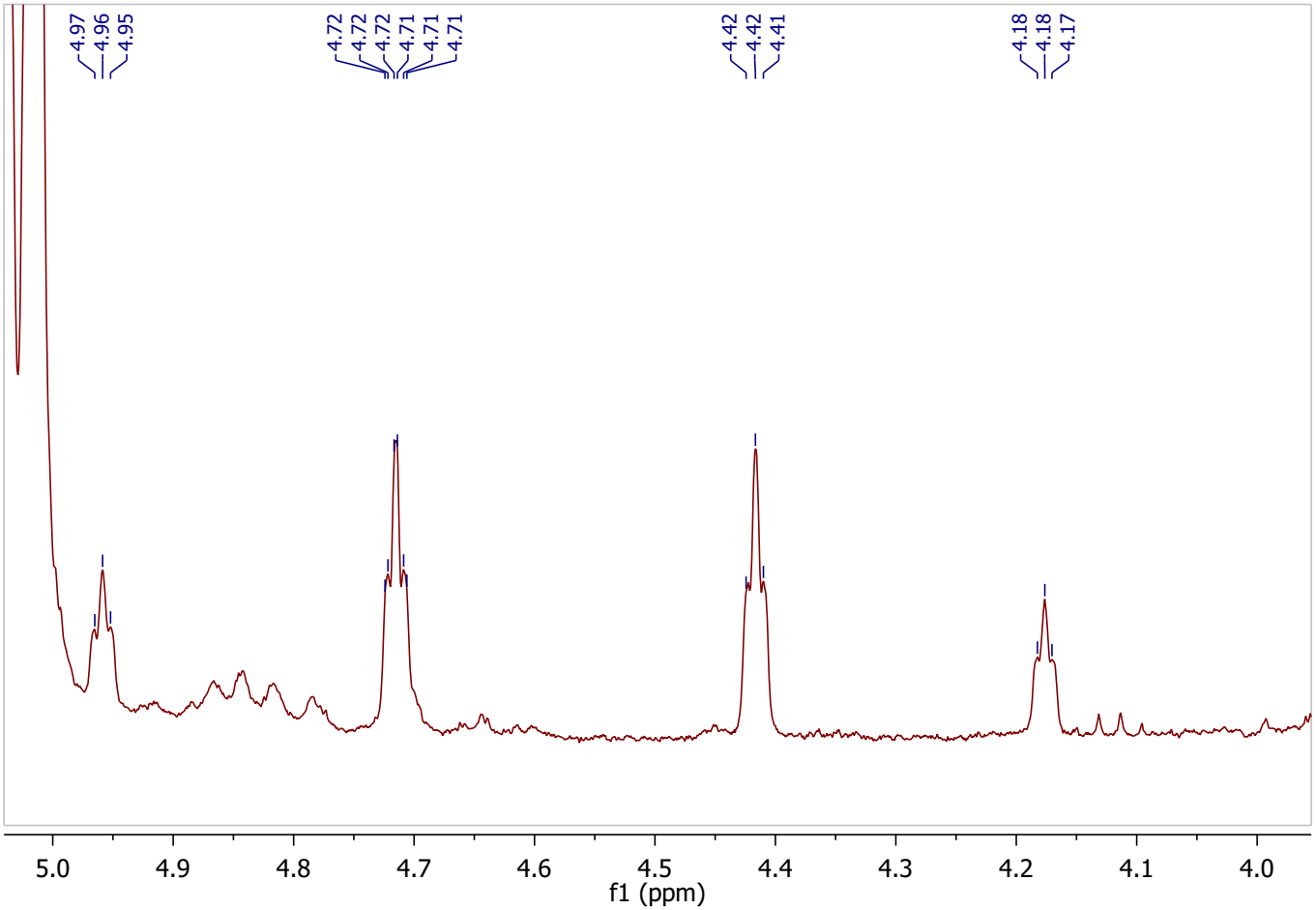
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.17 (dd, *J* = 8.4, 2.3 Hz, 1H), 7.05 (dd, *J* = 8.3, 2.2 Hz, 1H), 7.03 (s, 1H), 6.99 (d, *J* = 2.3 Hz, 1H), 6.98 (d, *J* = 2.2 Hz, 1H), 6.89 (d, *J* = 8.4 Hz, 1H), 6.84 (d, *J* = 8.3 Hz, 1H), 6.05 – 5.88 (m, 2H), 5.93 (d, *J* = 3.3 Hz, 1H), 5.15 – 5.04 (m, 4H), 4.85 (dd, *J* = 11.7, 6.3 Hz, 1H), 3.79 (s, 3H), 3.40 – 3.31 (m, 4H), 2.94 (dd, *J* = 14.7, 6.3 Hz, 1H), 2.78 (dd, *J* = 14.7, 11.7 Hz, 1H), 2.60 (dd, *J* = 11.7, 3.3 Hz, 1H), 2.47 (d, *J* = 11.7 Hz, 1H).

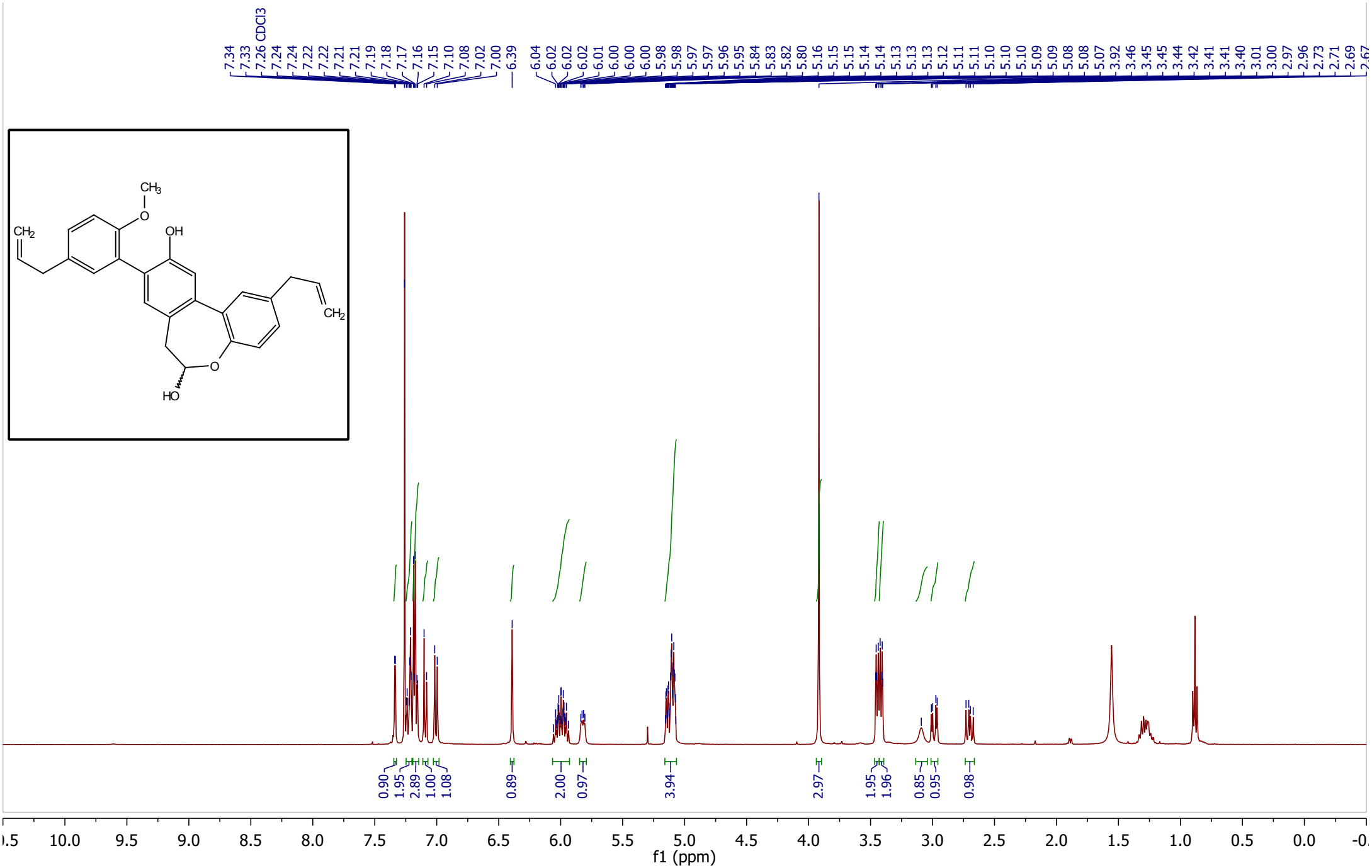

195.60
155.53
149.73
143.88
140.00
137.66
137.58
132.87
132.20
131.27
130.99
130.00
129.49
125.14
124.69
116.90
116.07
115.94
111.34
100.55
87.57
77.16 CDCl3
55.93
44.79
44.39
39.71
39.45
36.40
CH3
O
O
CH2
CH2
O
O
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



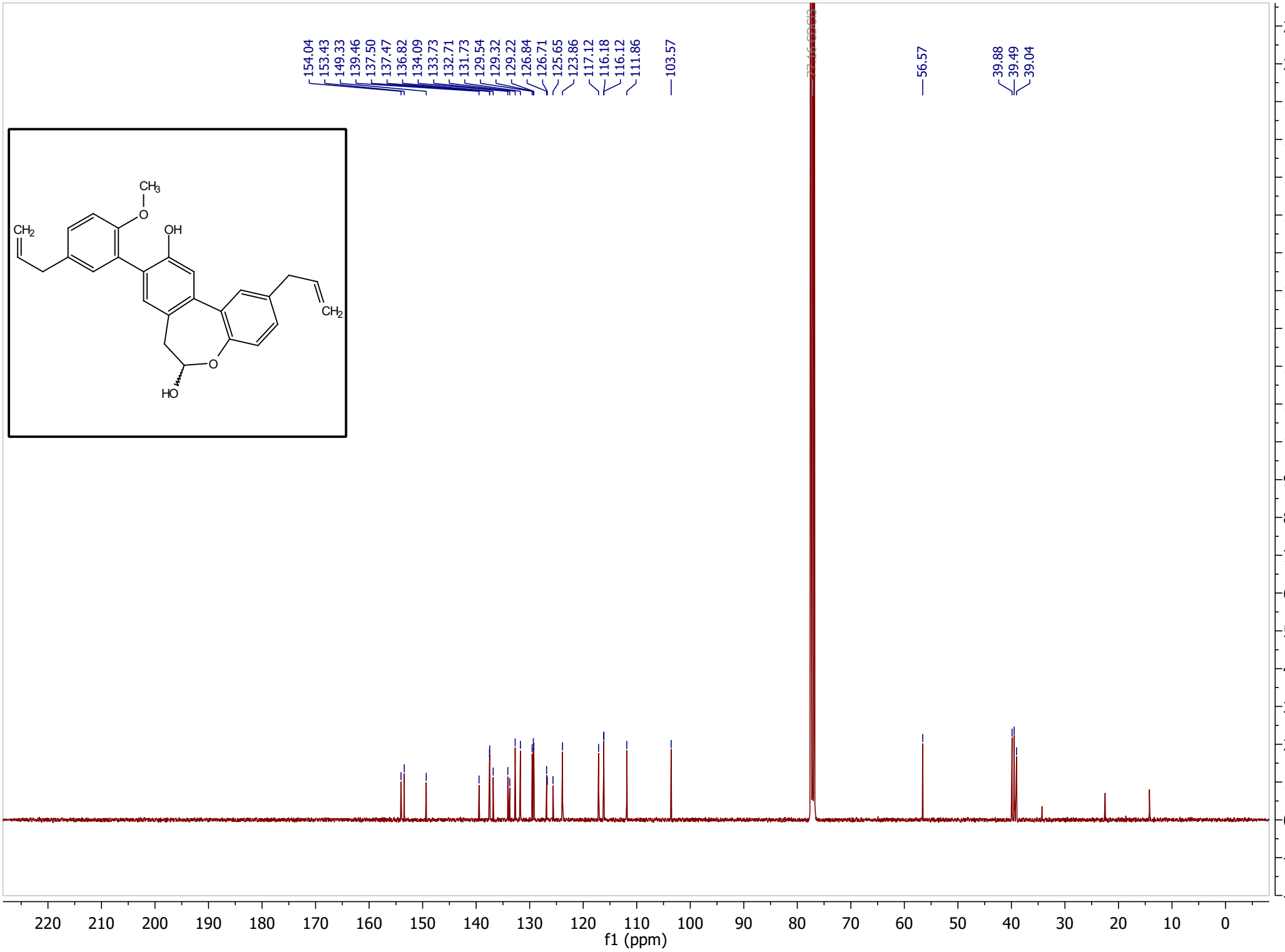
^{1}H NMR (400 MHz, Chloroform-*d*) δ 4.96 (t, *J* = 2.9 Hz, 0H), 4.72 (td, *J* = 3.1, 1.0 Hz, 1H), 4.42 (t, *J* = 2.9 Hz, 1H), 4.18 (t, *J* = 2.7 Hz, 1H).

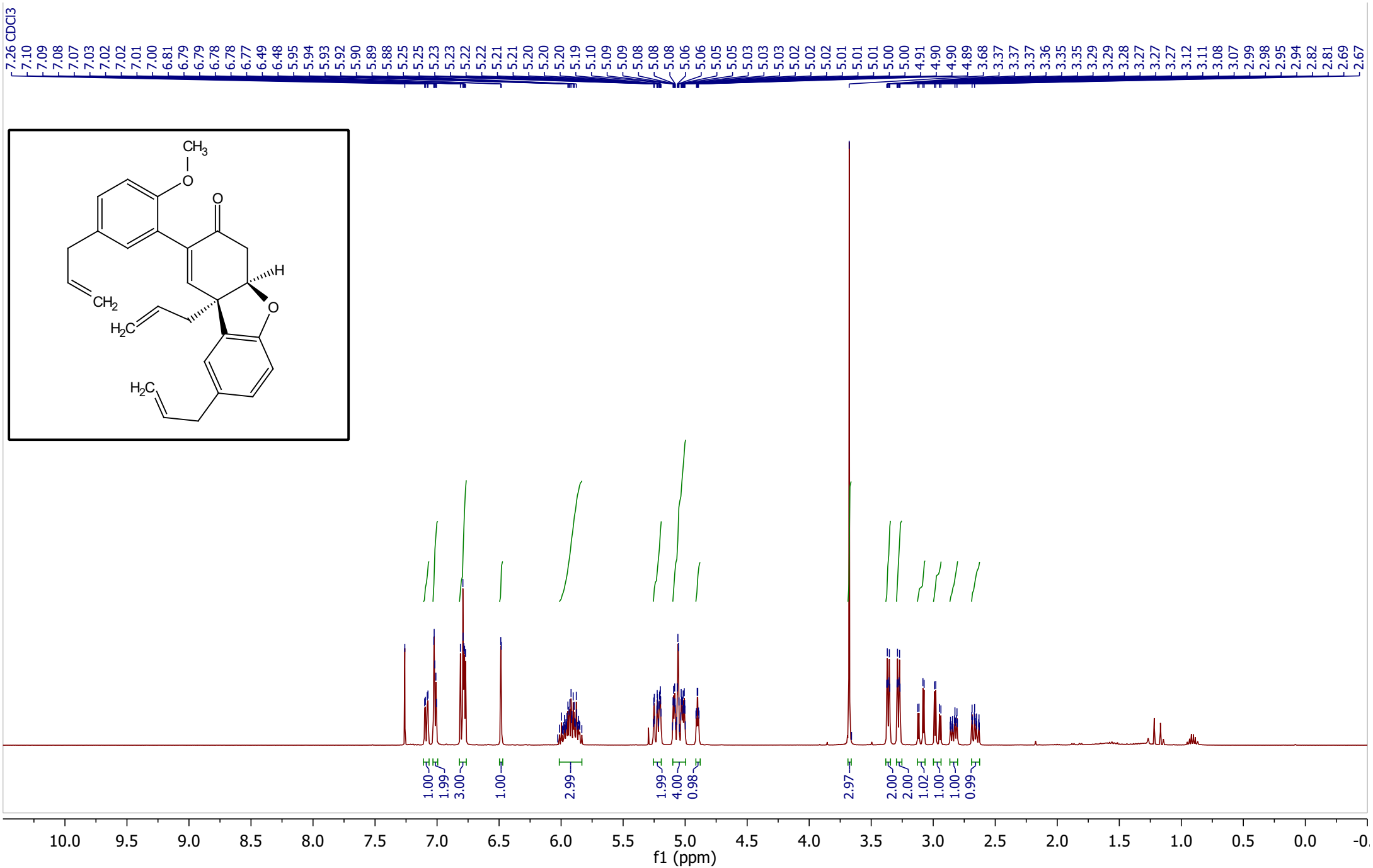

CH3
O
O
CH2
O
O
OH
CH2


4.97
4.96
4.95
4.72
4.72
4.72
4.71
4.71
4.71
4.42
4.42
4.41
4.18
4.18
4.17
5.0
4.9
4.8
4.7
4.6
4.5
4.4
4.3
4.2
4.1
4.0
f1 (ppm)

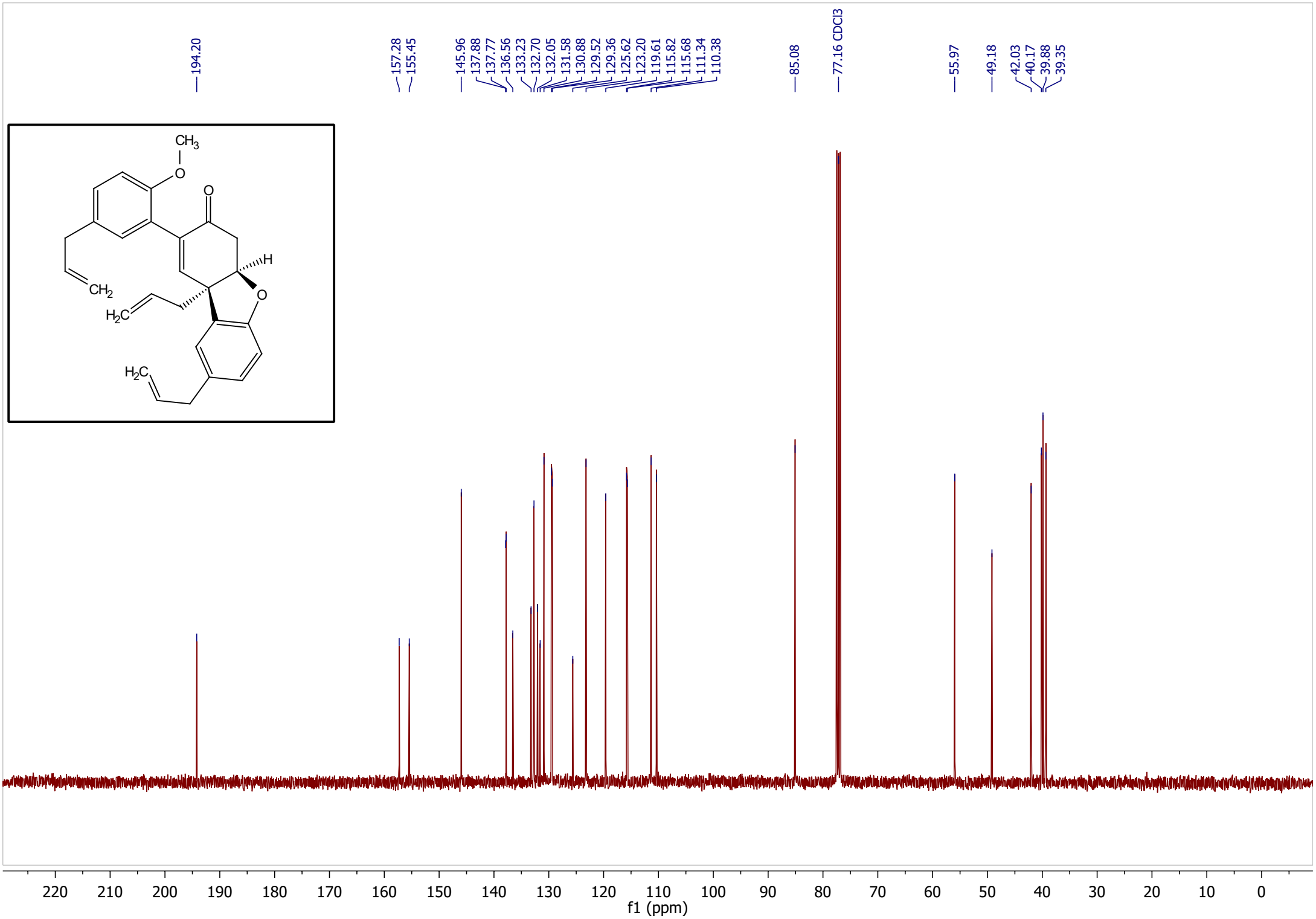


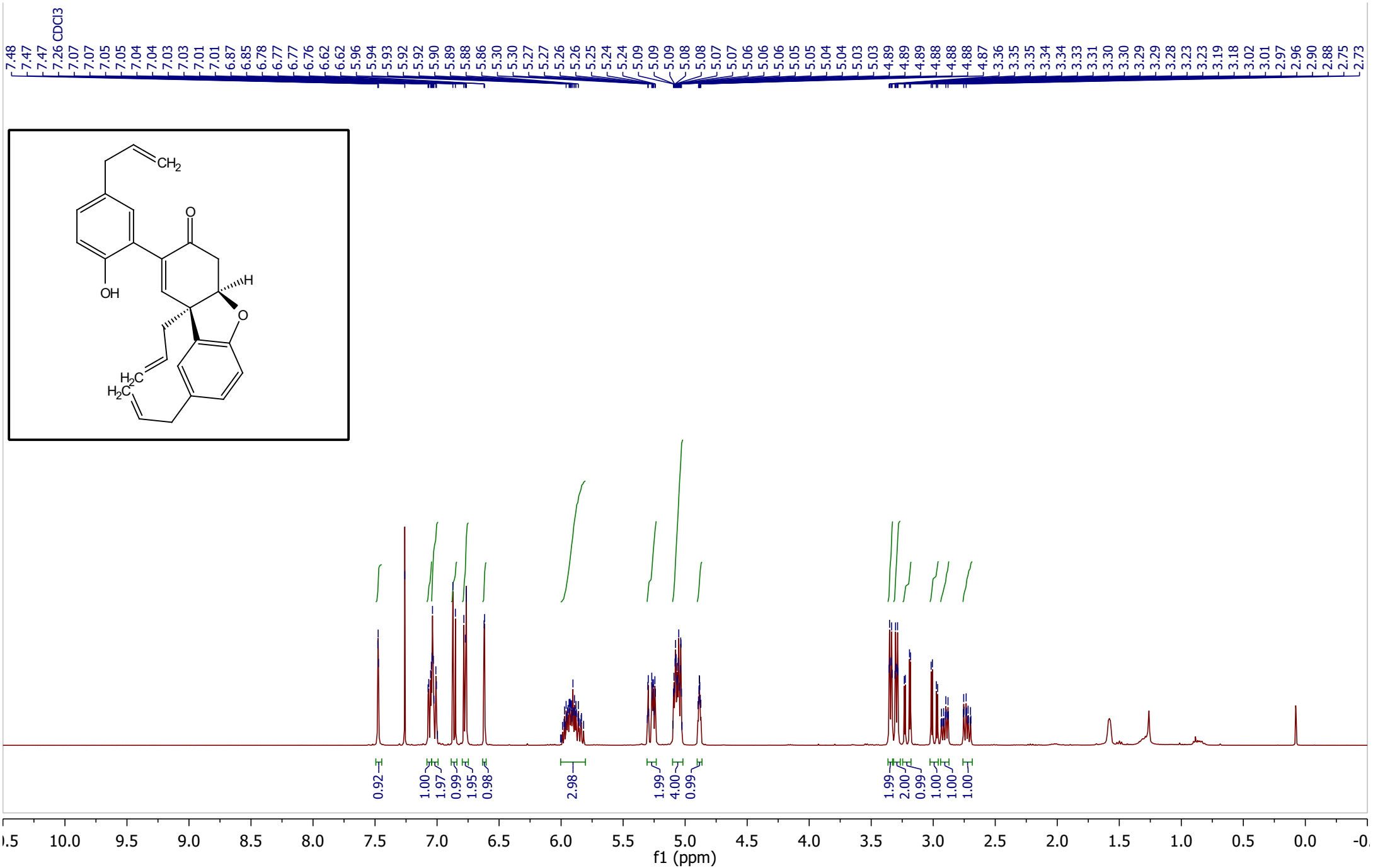
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.34 (d, *J* = 2.2 Hz, 1H), 7.23 (dd, *J* = 8.2, 2.3 Hz, 1H), 7.22 (d, *J* = 2.4 Hz, 1H), 7.19 (s, 1H), 7.17 (s, 1H), 7.16 (dd, *J* = 8.2, 2.2 Hz, 1H), 7.09 (d, *J* = 8.1 Hz, 1H), 7.01 (d, *J* = 8.2 Hz, 1H), 6.39 (s, 1H), 6.06 – 5.92 (m, 2H), 5.82 (dd, *J* = 9.1, 4.1 Hz, 1H), 5.17 – 5.06 (m, 4H), 3.92 (s, 3H), 3.45 (ddd, *J* = 6.7, 1.5, 1.5 Hz, 2H), 3.41 (ddd, *J* = 6.7, 1.5, 1.5 Hz, 2H), 3.09 (s, 1H), 2.99 (dd, *J* = 14.4, 4.1 Hz, 1H), 2.70 (dd, *J* = 14.3, 8.9 Hz, 1H).


154.04
153.43
149.33
139.46
137.50
137.47
136.82
134.09
133.73
132.71
131.73
129.54
129.32
129.22
126.84
126.71
125.65
123.86
117.12
116.18
116.12
111.86
103.57
56.57
39.88
39.49
39.04
CH3
O
OH
CH2
CH2
O
HO
f1 (ppm)

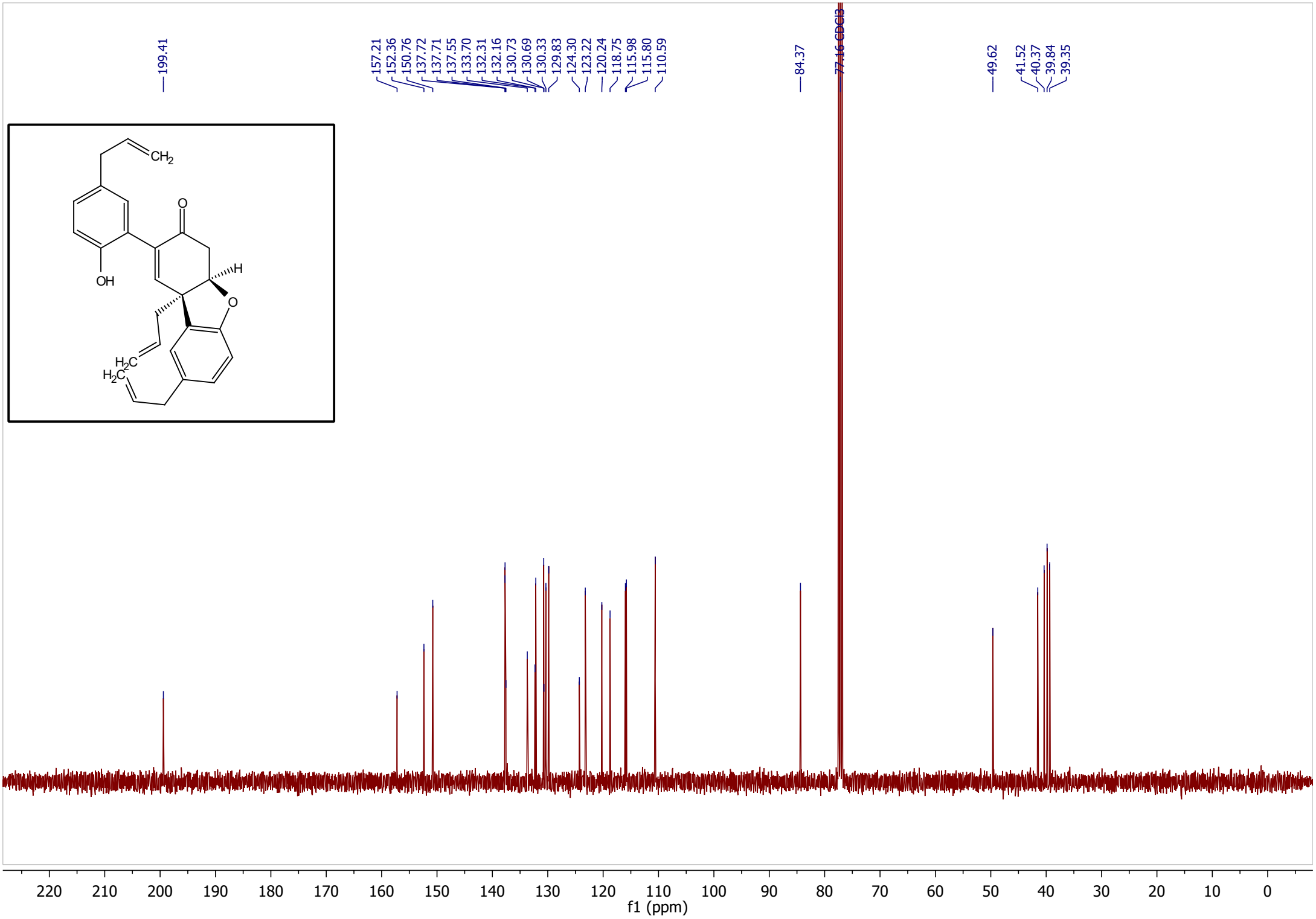


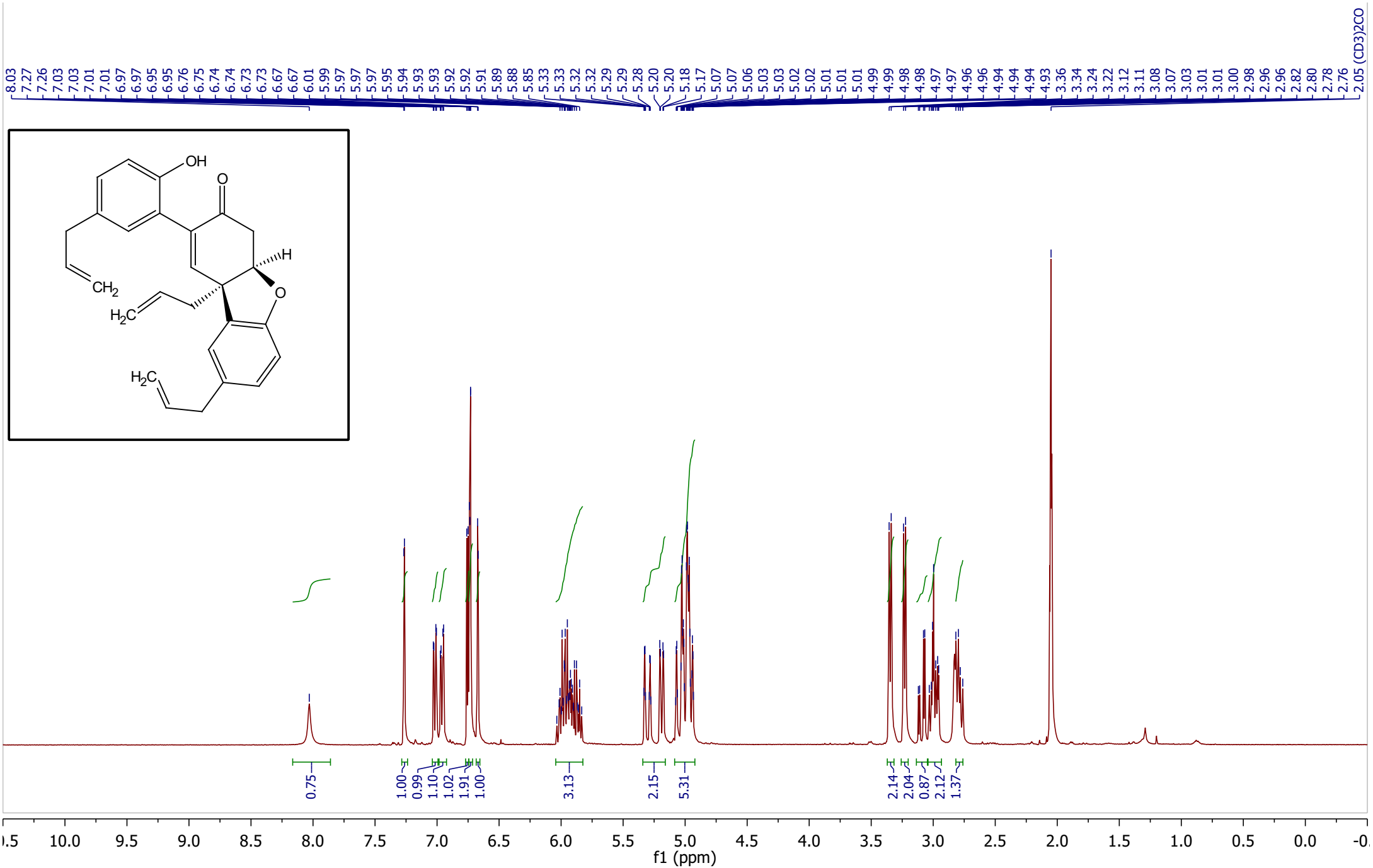
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.09 (dd, *J* = 8.3, 2.3 Hz, 1H), 7.04 – 6.99 (m, 2H), 6.80 (d, *J* = 8.3 Hz, 1H), 6.78 (d, *J* = 2.3 Hz, 1H), 6.78 (d, *J* = 8.3 Hz, 1H), 6.48 (d, *J* = 1.6 Hz, 1H), 6.03 – 5.82 (m, 3H), 5.26 – 5.18 (m, 2H), 5.11 – 4.99 (m, 4H), 4.90 (ddd, *J* = 4.1, 4.1, 1.6 Hz, 1H), 3.68 (s, 3H), 3.36 (ddd, *J* = 6.7, 1.5, 1.5 Hz, 2H), 3.28 (ddd, *J* = 6.7, 1.4, 1.4 Hz, 2H), 3.10 (dd, *J* = 16.5, 3.8 Hz, 1H), 2.96 (dd, *J* = 16.5, 4.3 Hz, 1H), 2.83 (dddd, *J* = 14.1, 6.5, 1.3, 1.3 Hz, 1H), 2.66 (dddd, *J* = 14.1, 8.2, 1.0, 1.0 Hz, 1H).


194.20
157.28
155.45
145.96
137.88
137.77
136.56
133.23
132.70
132.05
131.58
130.88
129.52
129.36
125.62
123.20
119.61
115.82
115.68
111.34
110.38
85.08
77.16 CDCl3
55.97
49.18
42.03
40.17
39.88
39.35
CH3
O
O
H
CH2
H2C
O
H2C
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
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50
40
30
20
10
0
f1 (ppm)

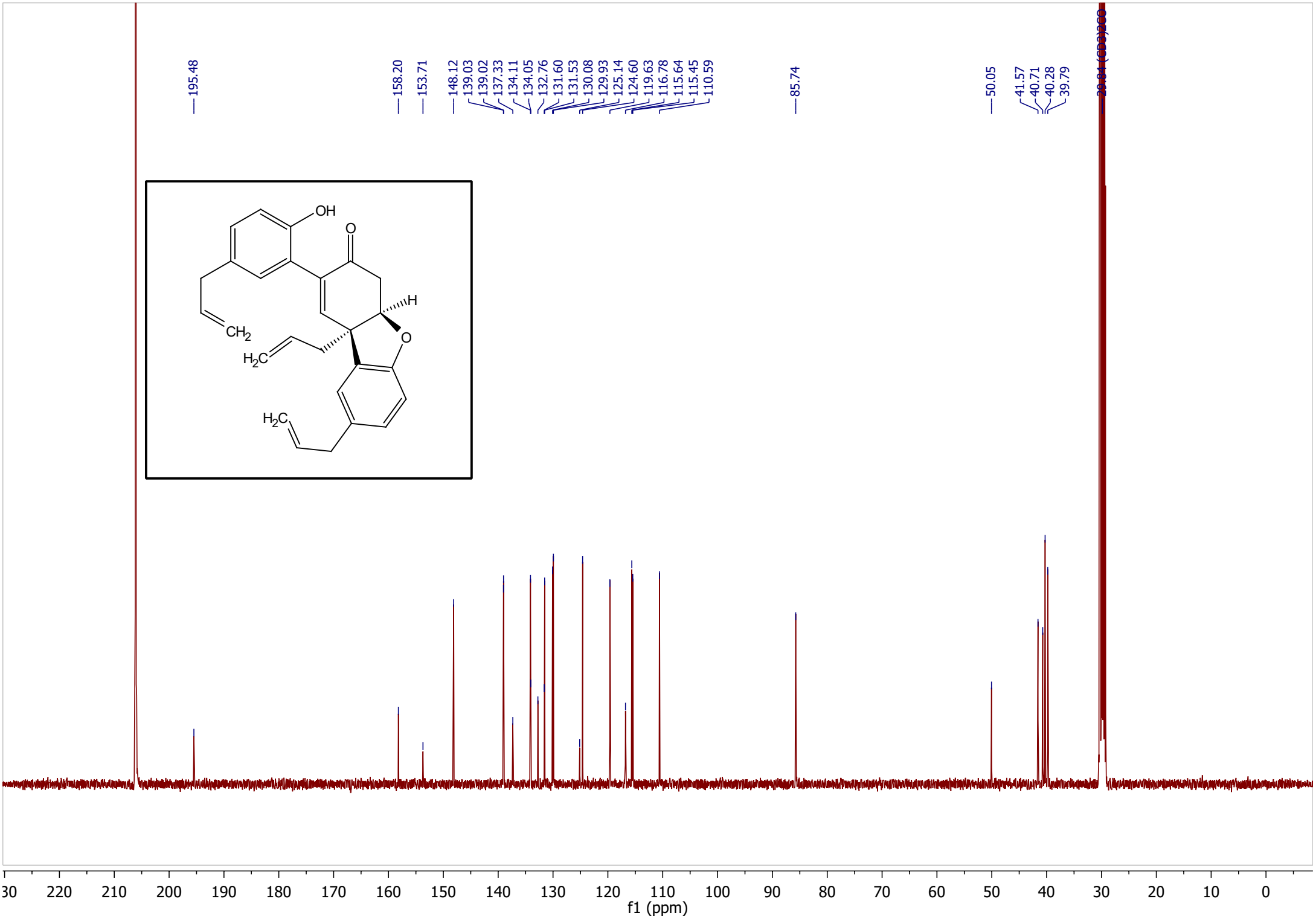


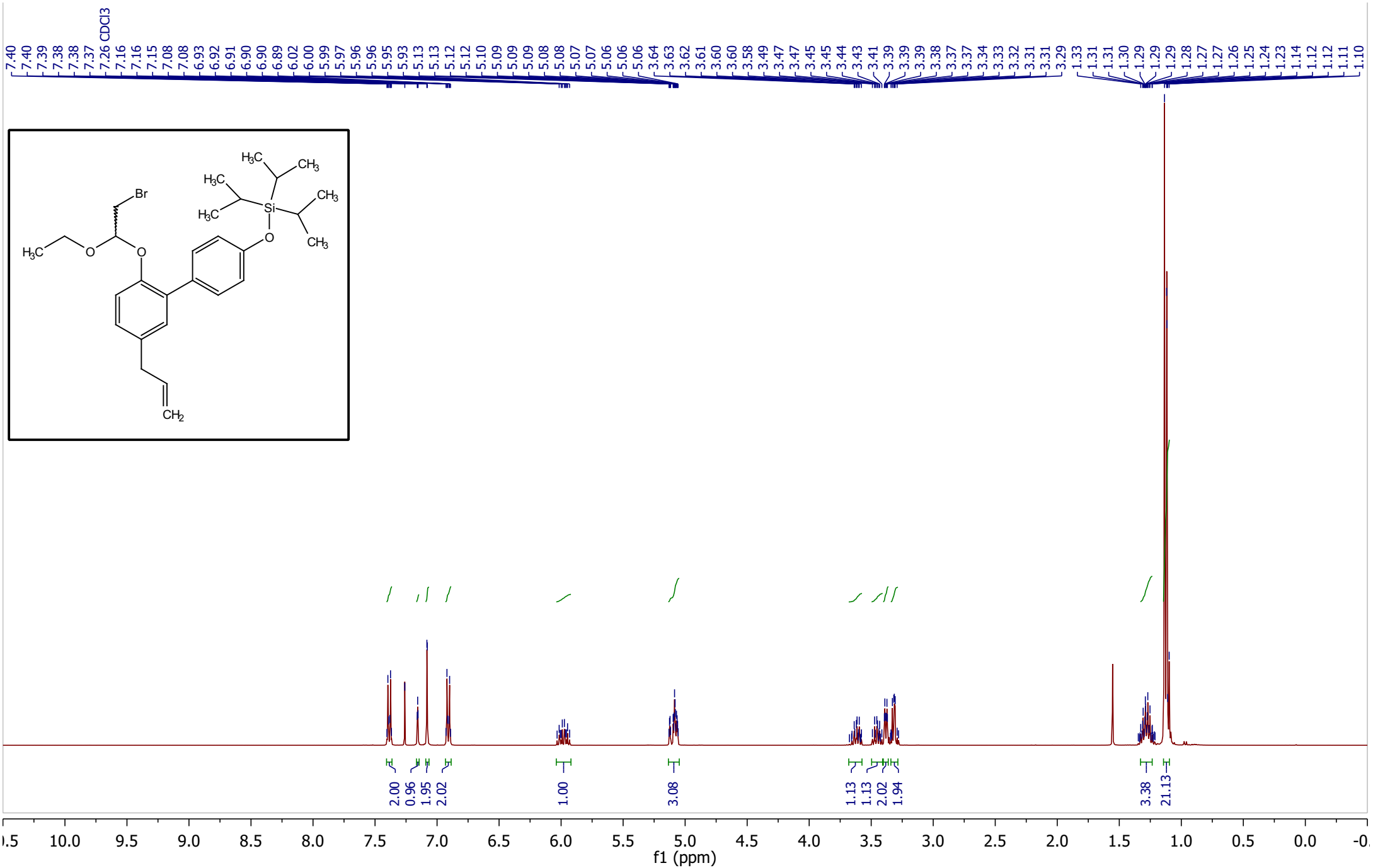
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.49 – 7.45 (m, 1H), 7.06 (dd, *J* = 8.2, 2.3 Hz, 1H), 7.04 – 6.99 (m, 2H), 6.86 (d, *J* = 8.2 Hz, 1H), 6.77 (d, *J* = 8.2 Hz, 1H), 6.77 (d, *J* = 2.3 Hz, 1H), 6.62 (d, *J* = 1.7 Hz, 1H), 6.01 – 5.80 (m, 3H), 5.32 – 5.23 (m, 2H), 5.11 – 5.02 (m, 4H), 4.88 (ddd, *J* = 4.1, 3.2, 1.7 Hz, 1H), 3.34 (ddd, *J* = 6.9, 1.6, 1.6 Hz, 2H), 3.30 (ddd, *J* = 6.9, 1.6, 1.6 Hz, 2H), 3.21 (dd, *J* = 16.8, 3.2 Hz, 1H), 2.99 (dd, *J* = 16.8, 4.2 Hz, 1H), 2.91 (dddd, *J* = 14.2, 7.0, 1.3, 1.3 Hz, 1H), 2.73 (dddd, *J* = 14.0, 7.9, 1.0, 1.0 Hz, 1H).


199.41
157.21
152.36
150.76
137.72
137.71
137.55
133.70
132.31
132.16
130.73
130.69
130.33
129.83
124.30
123.22
120.24
118.75
115.98
115.80
110.59
84.37
77.16 CDCl3
49.62
41.52
40.37
39.84
39.35
CH2
O
OH
H
O
H2C
H2C
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

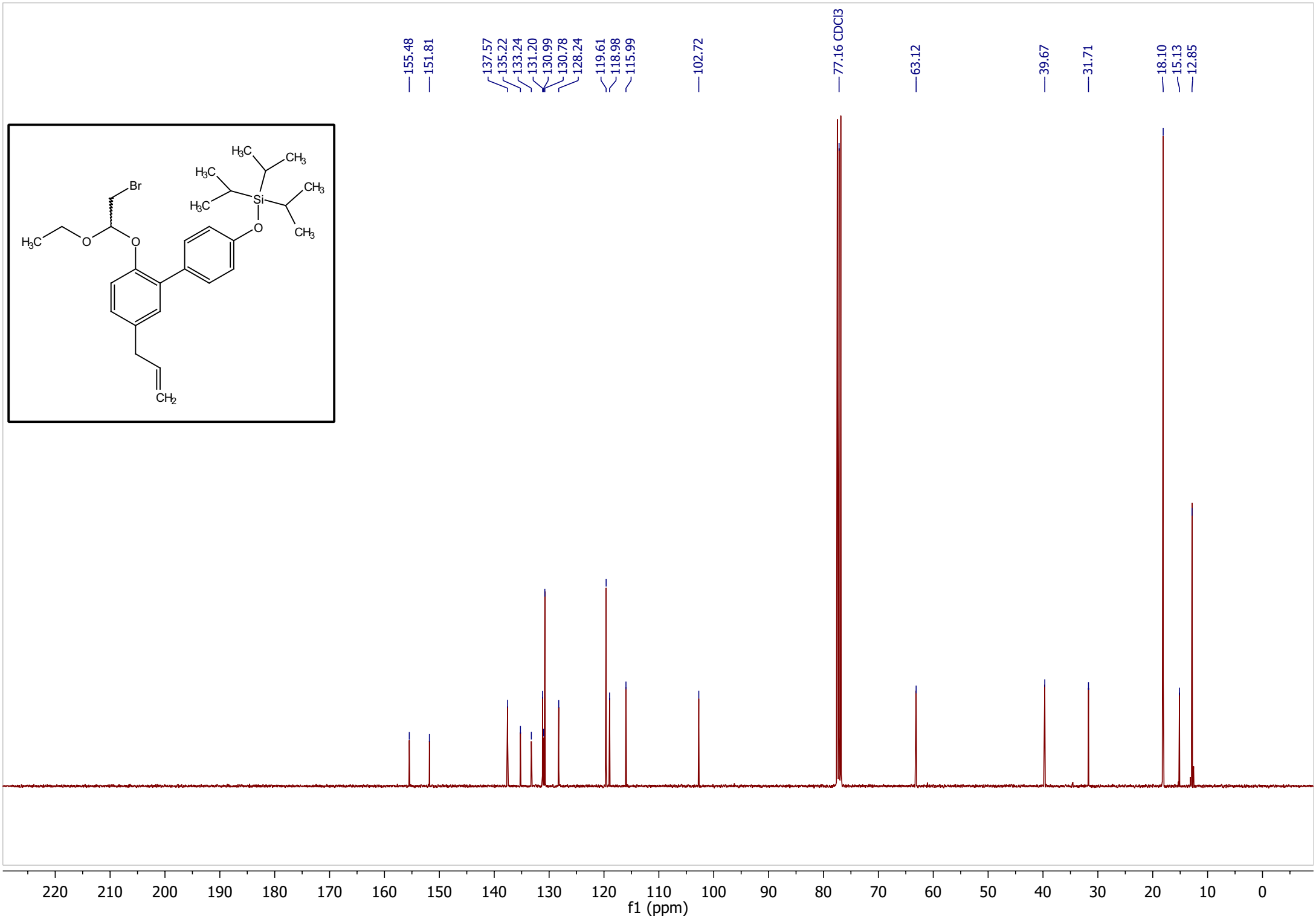


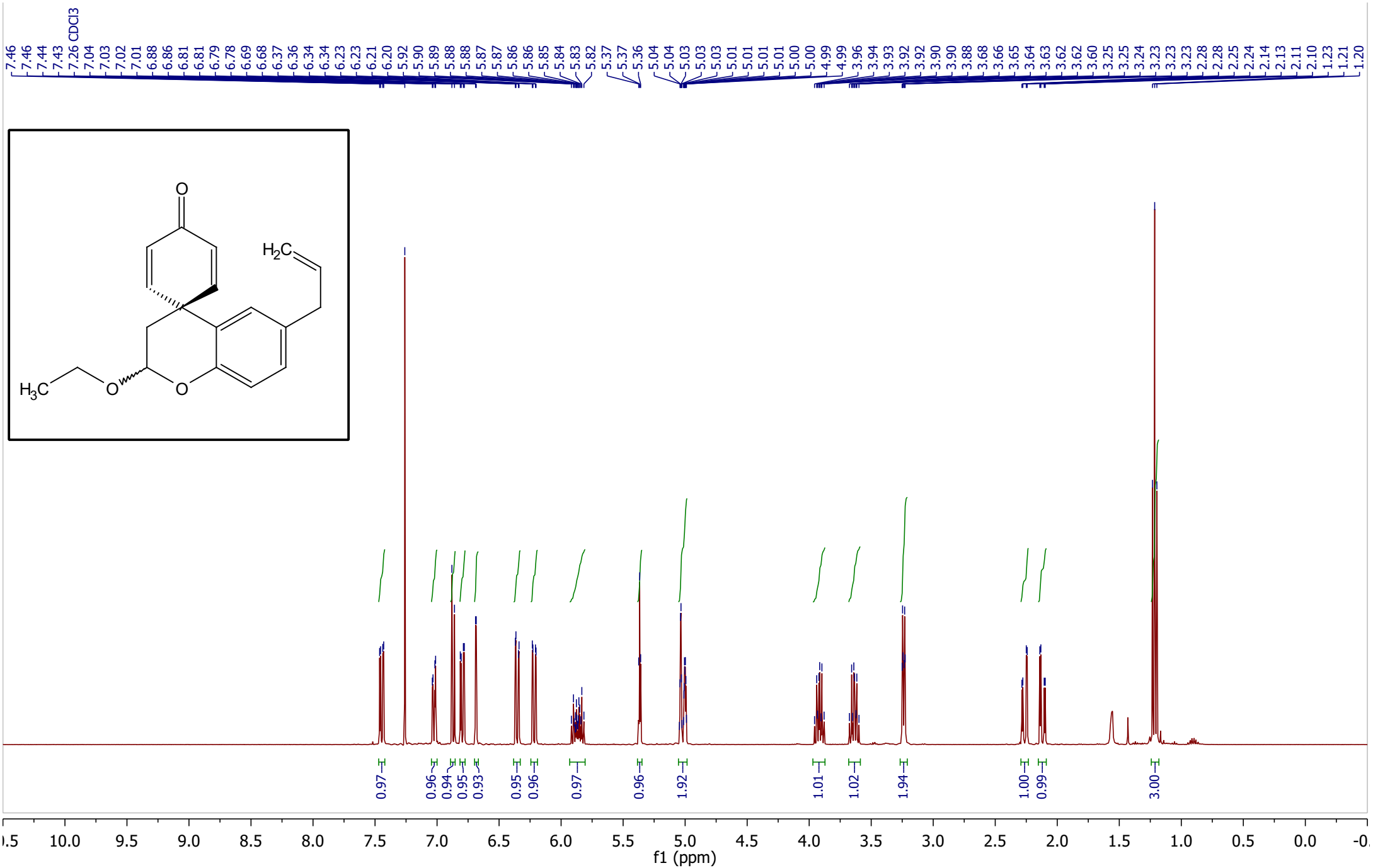
^{1}H NMR (400 MHz, Acetone-d_6) δ 8.03 (s, 1H), 7.27 (d, J = 1.8 Hz, 1H), 7.02 (dd, J = 8.2, 1.8 Hz, 1H), 6.96 (dd, J = 8.2, 2.3 Hz, 1H), 6.75 (d, J = 8.2 Hz, 1H), 6.74 (d, J = 8.0 Hz, 1H), 6.73 (d, J = 2.3 Hz, 1H), 6.67 (d, J = 1.8 Hz, 1H), 6.05 – 5.83 (m, 3H), 5.35 – 5.15 (m, 2H), 5.09 – 4.92 (m, 5H), 3.35 (d, J = 6.7 Hz, 2H), 3.23 (d, J = 6.7 Hz, 2H), 3.09 (dd, J = 16.5, 4.1 Hz, 1H), 3.04 – 2.94 (m, 2H), 2.79 (dd, J = 14.2, 8.1 Hz, 1H).


195.48
158.20
153.71
148.12
139.03
139.02
137.33
134.11
134.05
132.76
131.60
131.53
130.08
129.93
125.14
124.60
119.63
116.78
115.64
115.45
110.59
85.74
50.05
41.57
40.71
40.28
39.79
OH
O
H
O
CH2
H2C
H2C
f1 (ppm)

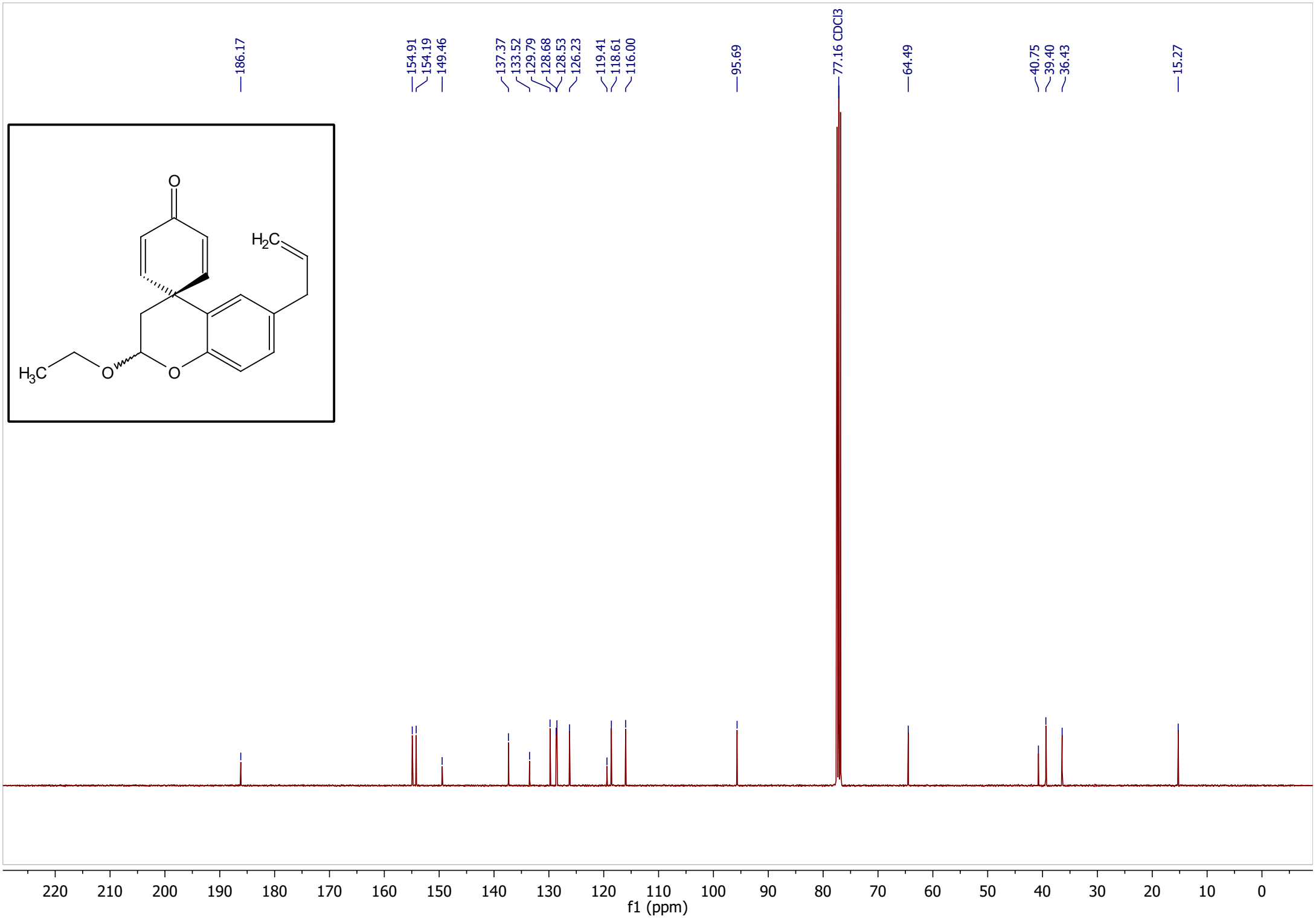


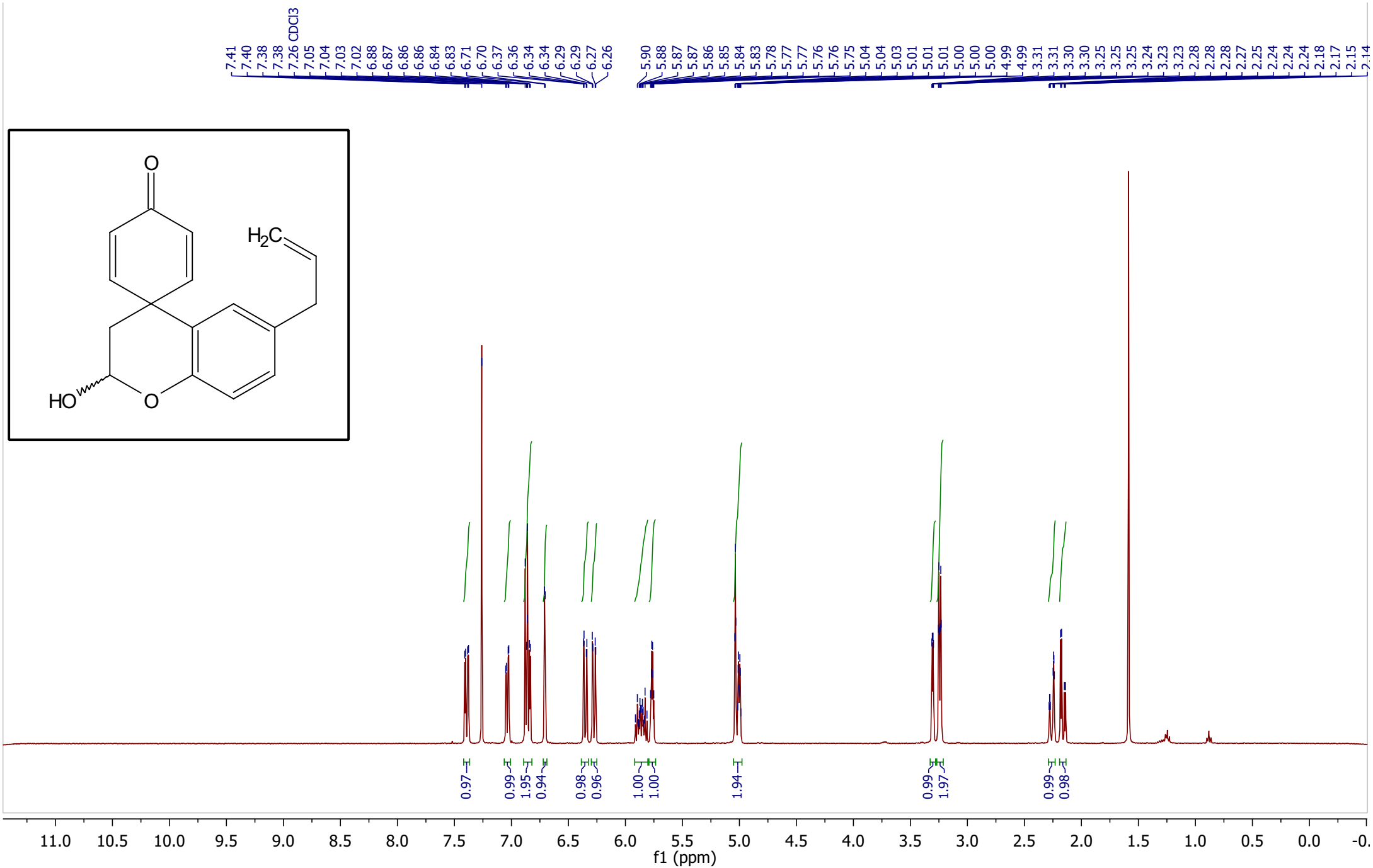
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.41 – 7.36 (m, 2H), 7.17 – 7.14 (m, 1H), 7.09 – 7.07 (m, 2H), 6.93 – 6.89 (m, 2H), 5.98 (dddd, *J* = 16.8, 10.0, 6.7, 6.7 Hz, 1H), 5.15 – 5.04 (m, 3H), 3.62 (dq, *J* = 9.3, 7.0 Hz, 1H), 3.45 (dq, *J* = 9.3, 7.0 Hz, 1H), 3.38 (ddd, *J* = 6.8, 1.5, 1.5 Hz, 2H), 3.35 – 3.28 (m, 2H), 1.36 – 1.21 (m, 3H), 1.15 – 1.10 (m, 21H).


155.48
151.81
137.57
135.22
133.24
131.20
130.99
130.78
128.24
119.61
118.98
115.99
102.72
77.16 CDCl3
63.12
39.67
31.71
18.10
15.13
12.85
Br
O
O
Si
O
CH2
f1 (ppm)

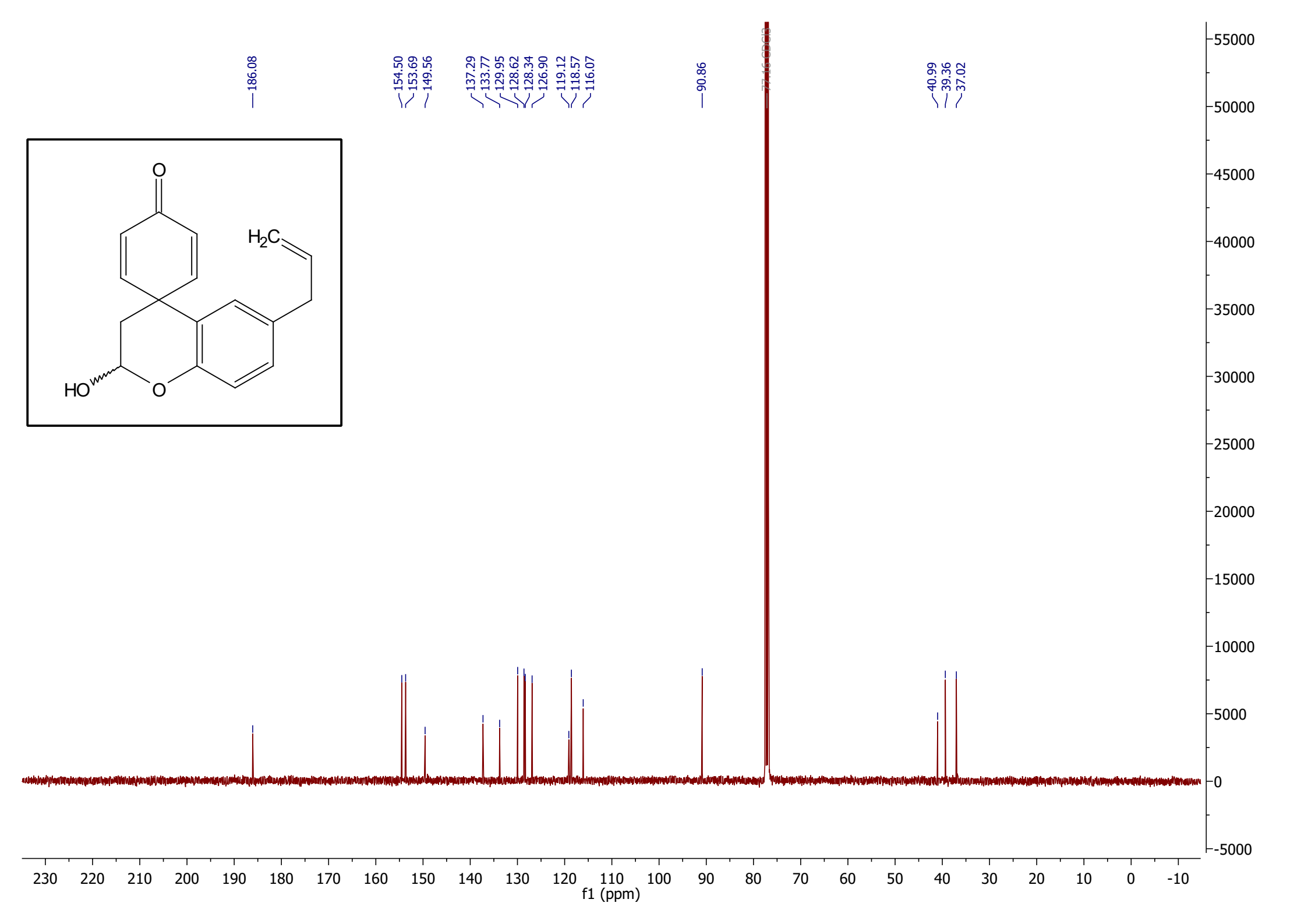


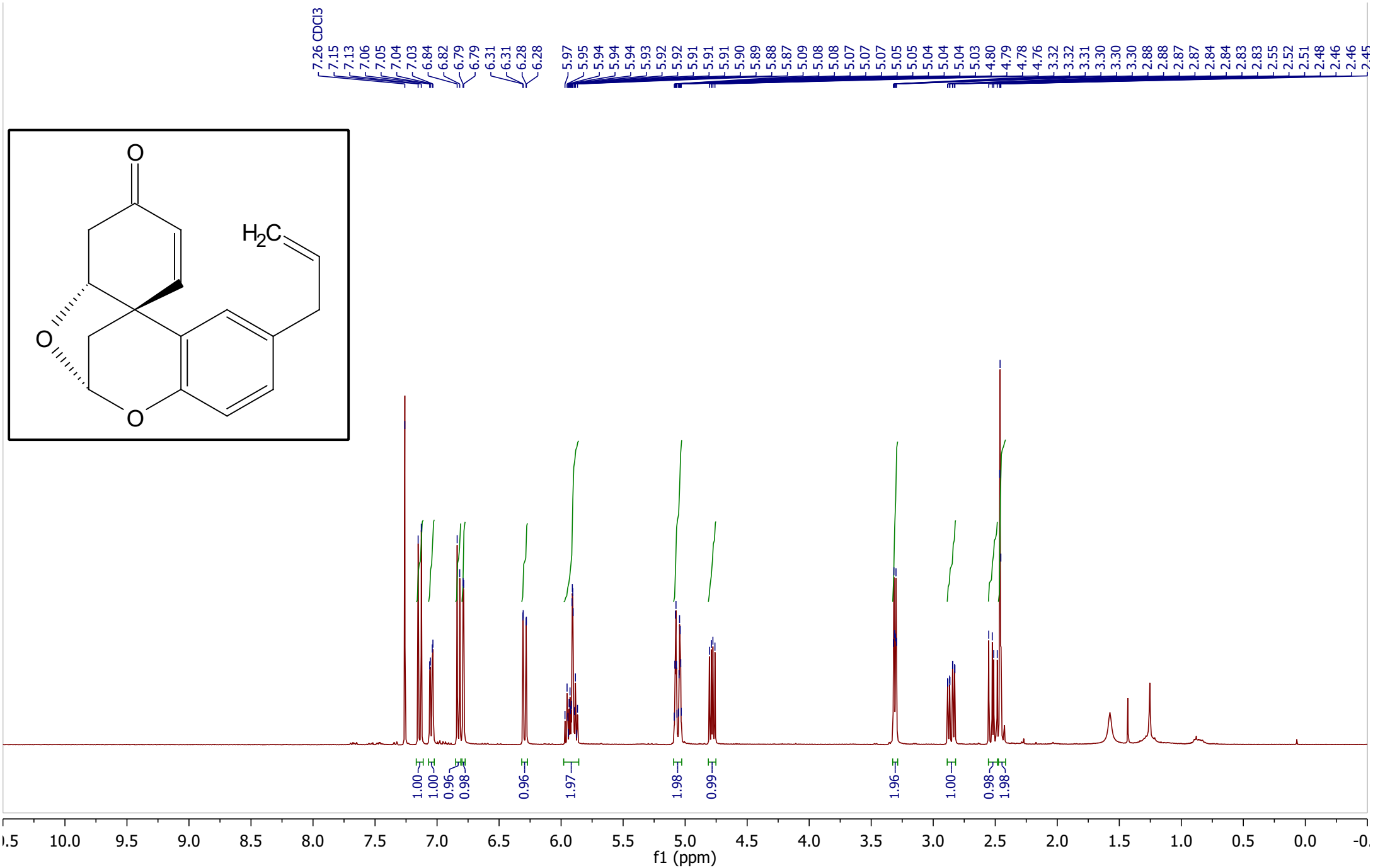
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.45 (dd, *J* = 10.1, 2.9 Hz, 1H), 7.02 (dd, *J* = 8.3, 2.2 Hz, 1H), 6.87 (d, *J* = 8.3 Hz, 1H), 6.80 (dd, *J* = 10.0, 2.9 Hz, 1H), 6.69 (d, *J* = 2.2 Hz, 1H), 6.35 (dd, *J* = 10.0, 1.9 Hz, 1H), 6.22 (dd, *J* = 10.1, 1.9 Hz, 1H), 5.87 (dddd, *J* = 17.6, 9.5, 6.7, 6.7 Hz, 1H), 5.37 (dd, *J* = 3.1, 3.1 Hz, 1H), 5.06 – 4.98 (m, 2H), 3.92 (dq, *J* = 9.6, 7.1 Hz, 1H), 3.64 (dq, *J* = 9.6, 7.1 Hz, 1H), 3.24 (ddd, *J* = 6.7, 1.5, 1.5 Hz, 2H), 2.26 (dd, *J* = 14.1, 2.9 Hz, 1H), 2.12 (dd, *J* = 14.1, 3.3 Hz, 1H), 1.21 (dd, *J* = 7.1, 7.1 Hz, 3H).


186.17
154.91
154.19
149.46
137.37
133.52
129.79
128.68
128.53
126.23
119.41
118.61
116.00
95.69
77.16 CDCl3
64.49
40.75
39.40
36.43
15.27
O
H2C
H3C
O
O
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

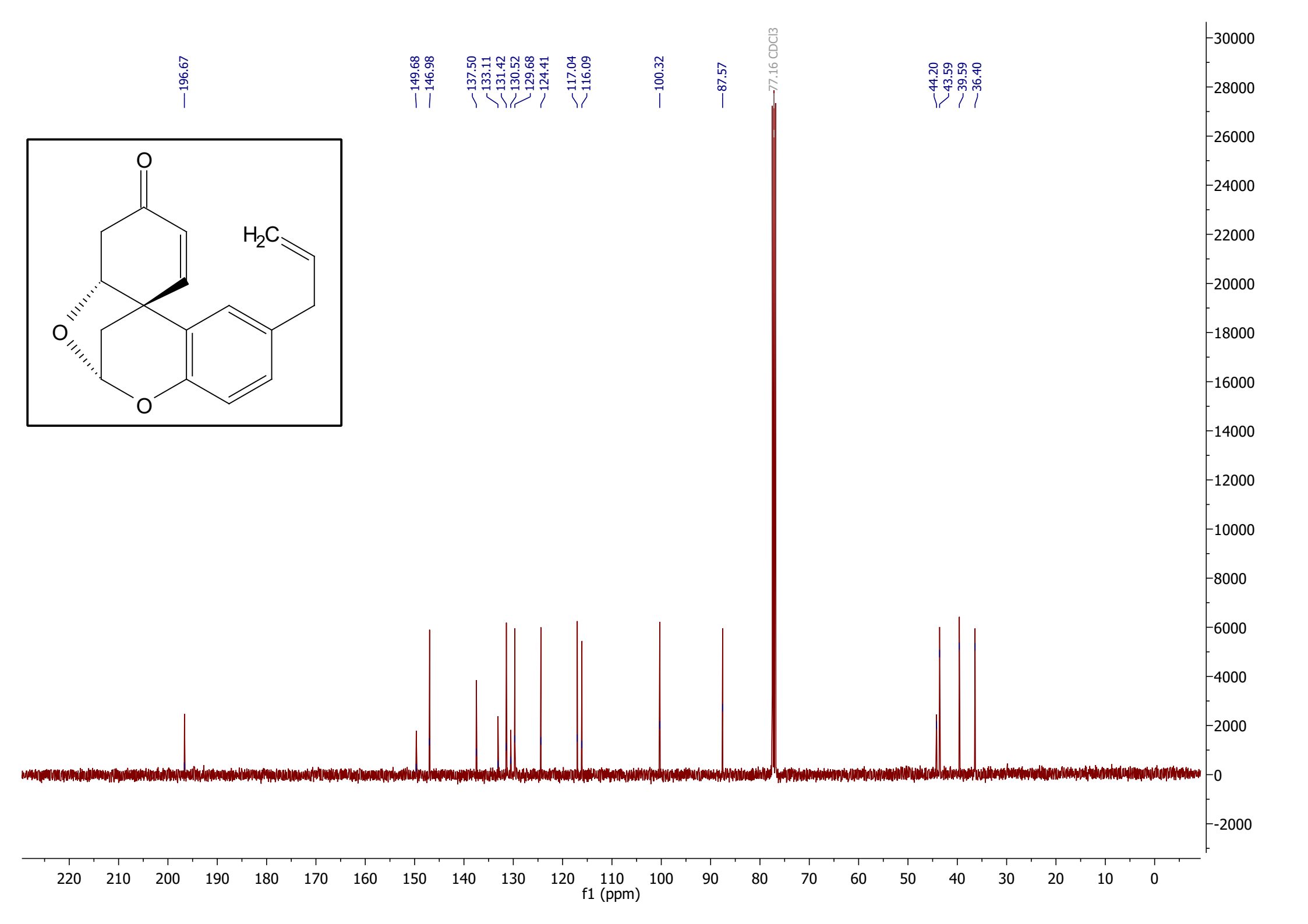


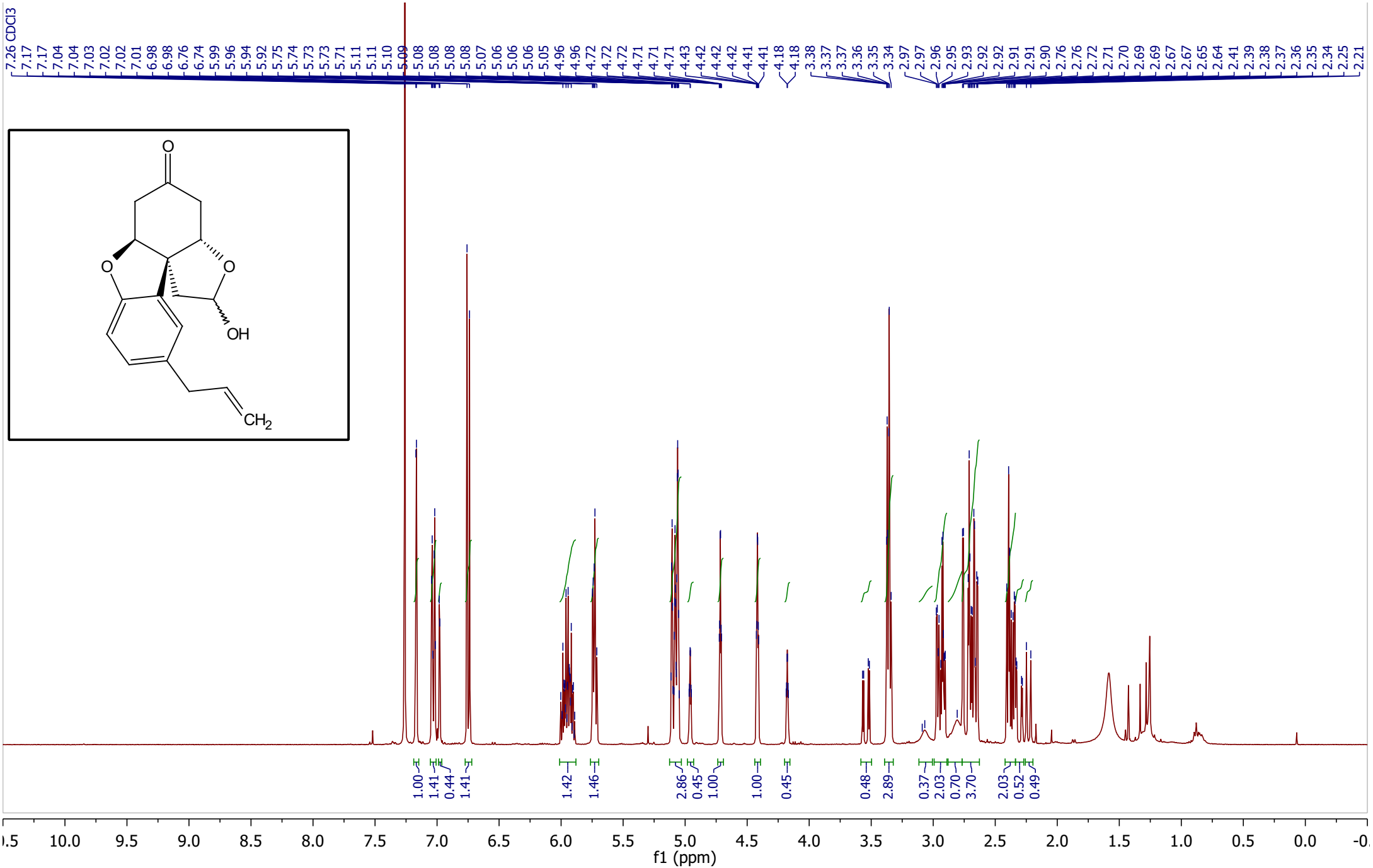
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.39 (dd, *J* = 10.1, 3.0 Hz, 1H), 7.04 (dd, *J* = 8.3, 2.2 Hz, 1H), 6.87 (d, *J* = 8.3 Hz, 1H), 6.85 (dd, *J* = 10.0, 3.0 Hz, 1H), 6.71 (d, *J* = 2.2 Hz, 1H), 6.35 (dd, *J* = 10.0, 1.9 Hz, 1H), 6.28 (dd, *J* = 10.1, 1.9 Hz, 1H), 5.86 (dddd, *J* = 18.2, 9.4, 6.7, 6.7 Hz, 1H), 5.77 (ddd, *J* = 4.2, 4.2, 2.8 Hz, 1H), 5.05 – 4.98 (m, 2H), 3.30 (dd, *J* = 4.1, 1.6 Hz, 1H), 3.24 (ddd, *J* = 6.4, 1.4, 1.4 Hz, 2H), 2.26 (ddd, *J* = 14.0, 2.8, 1.6 Hz, 1H), 2.16 (dd, *J* = 14.0, 4.3 Hz, 1H).


186.08
154.50
153.69
149.56
137.29
133.77
129.95
128.62
128.34
126.90
119.12
118.57
116.07
90.86
77.16 CDCl3
40.99
39.36
37.02
O
H2C
HO
O
55000
50000
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
-5000
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

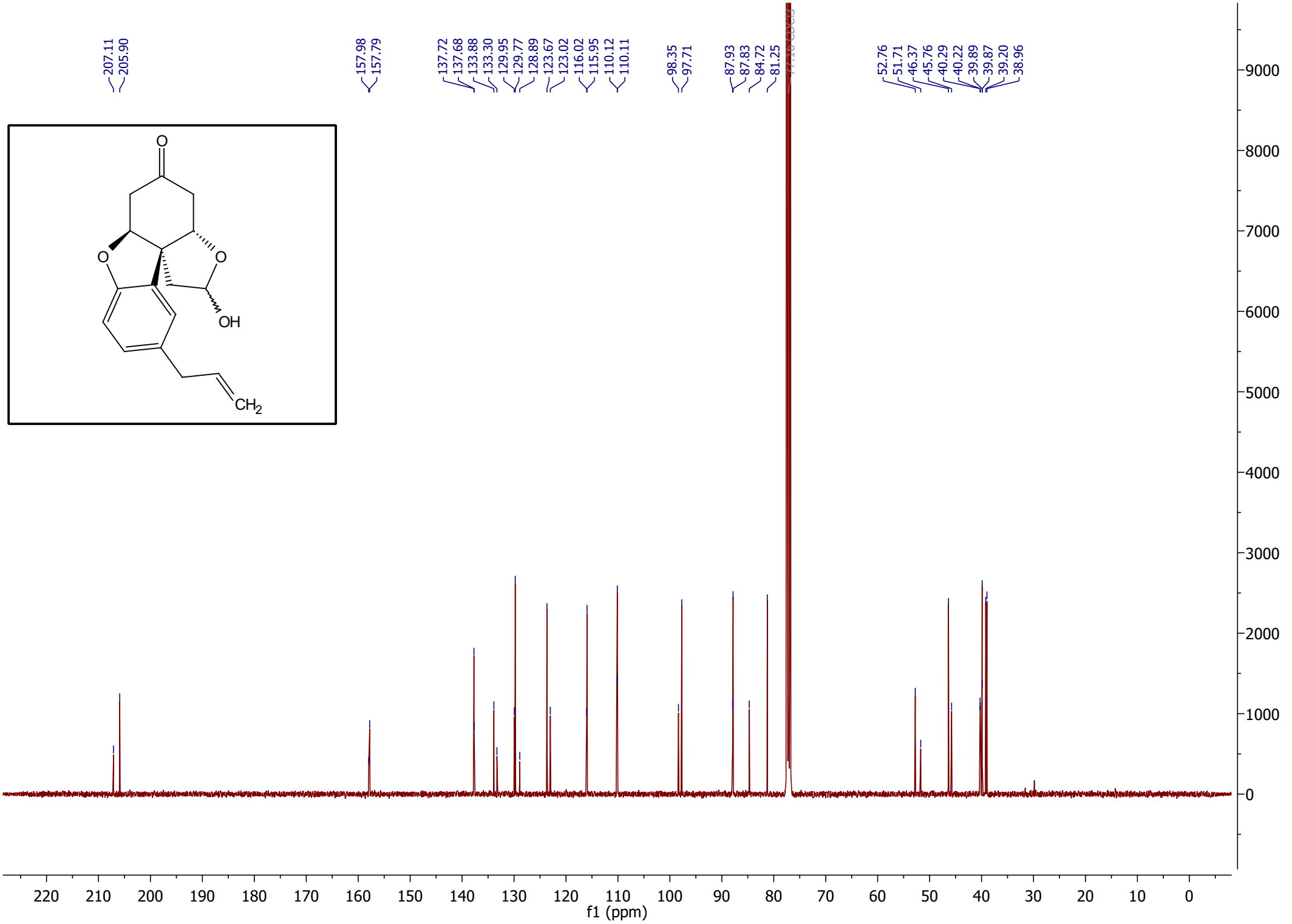


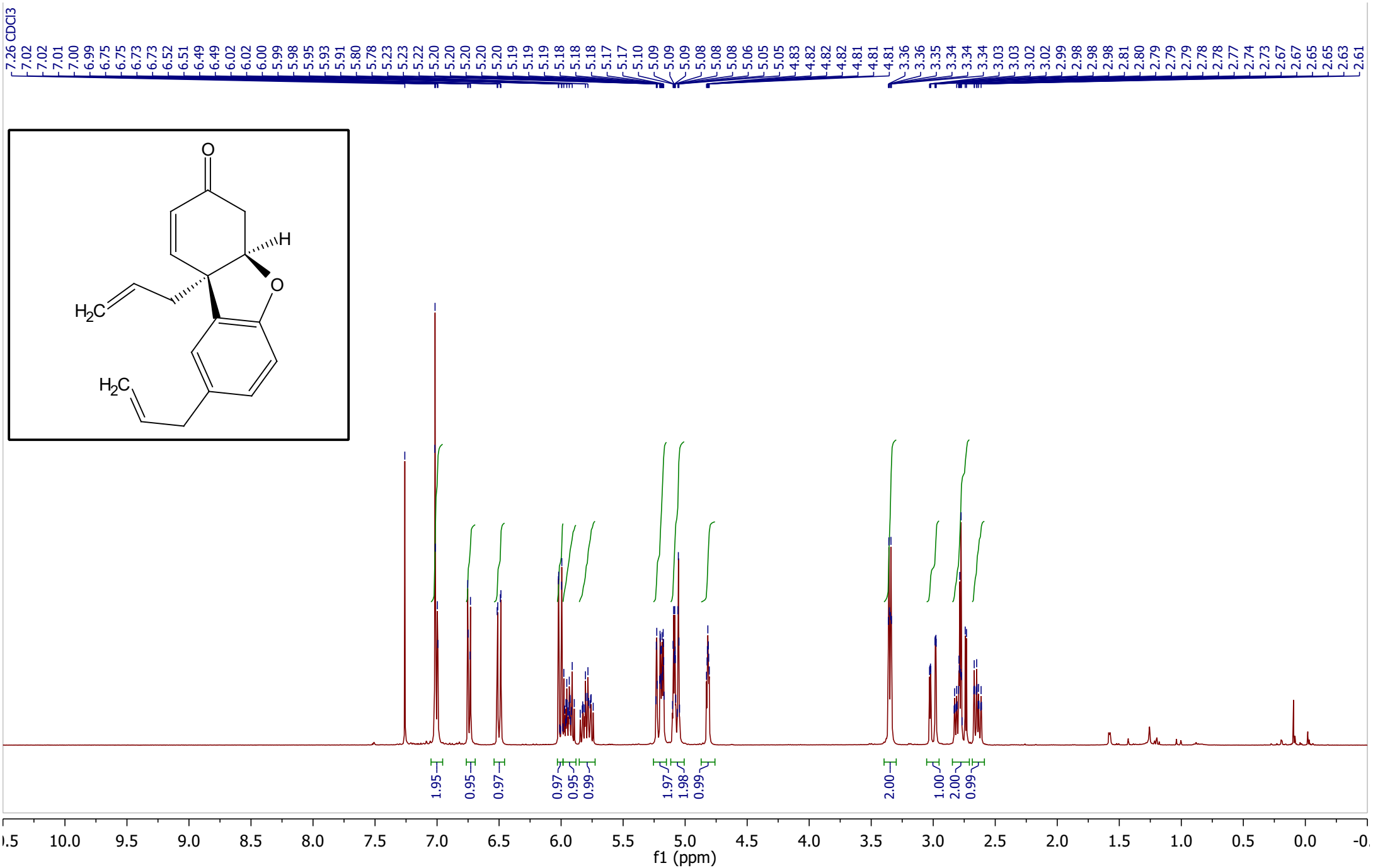
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.14 (d, *J* = 10.3 Hz, 1H), 7.04 (dd, *J* = 8.3, 2.1 Hz, 1H), 6.83 (d, *J* = 8.3 Hz, 1H), 6.79 (d, *J* = 2.1 Hz, 1H), 6.29 (dd, *J* = 10.3, 0.9 Hz, 1H), 5.97 – 5.86 (m, 2H), 5.10 – 5.02 (m, 2H), 4.78 (dd, *J* = 11.3, 6.6 Hz, 1H), 3.31 (ddd, *J* = 6.8, 1.6, 1.6 Hz, 2H), 2.85 (ddd, *J* = 16.1, 6.6, 1.0 Hz, 1H), 2.52 (dd, *J* = 16.1, 11.4 Hz, 1H), 2.47 – 2.44 (m, 2H).


196.67
149.68
146.98
137.50
133.11
131.42
130.52
129.68
124.41
117.04
116.09
100.32
87.57
77.16 CDCl3
44.20
43.59
39.59
36.40
O
O
O
H2C
30000
28000
26000
24000
22000
20000
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
-2000
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

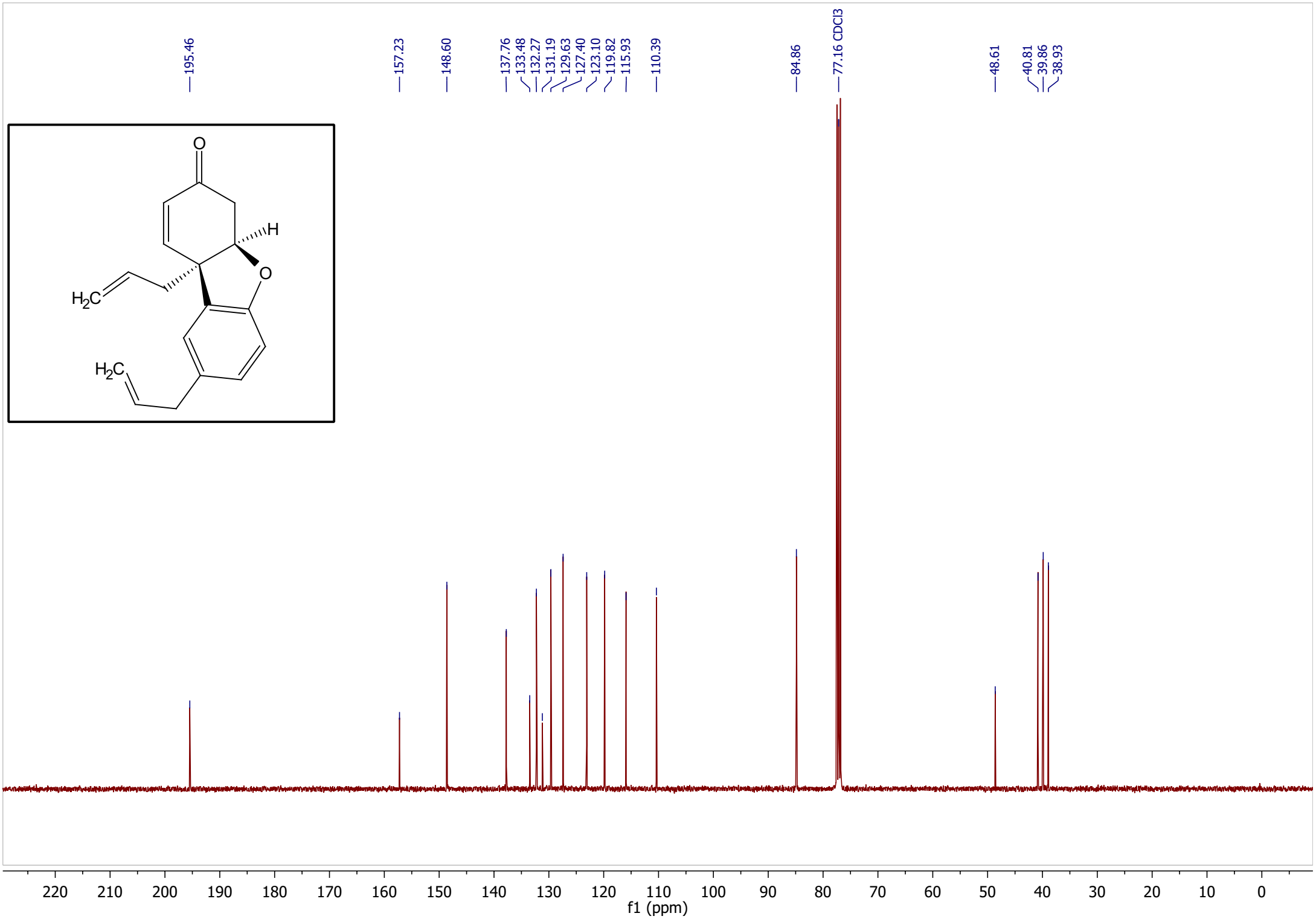


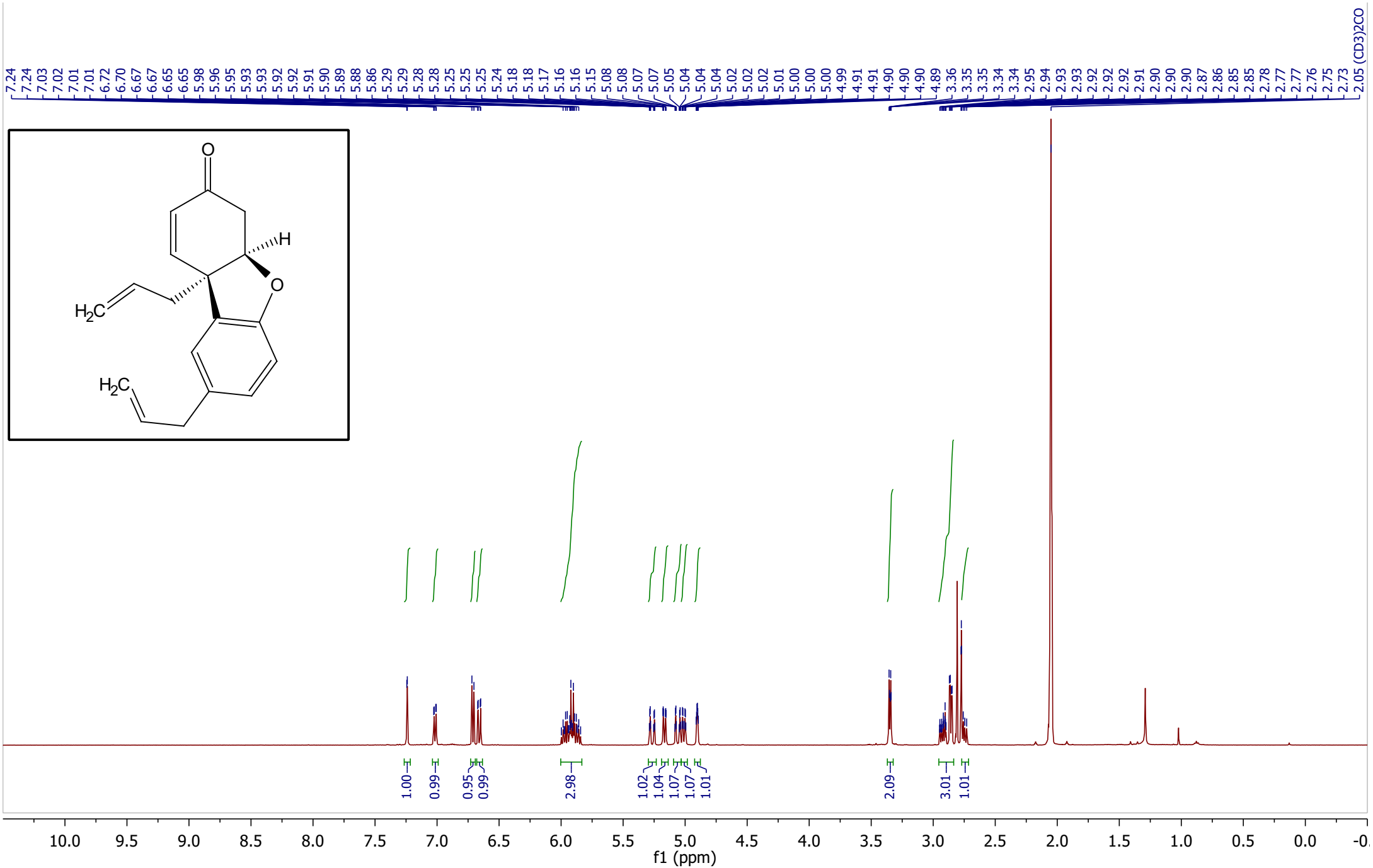
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.17 (d, *J* = 1.8 Hz, 1H), 7.08 – 7.01 (m, 2H), 6.98 (d, *J* = 1.8 Hz, 1H), 6.75 (d, *J* = 8.1 Hz, 2H), 6.01 – 5.89 (m, 2H), 5.76 – 5.70 (m, 2H), 5.12 – 5.04 (m, 4H), 4.96 (ddd, *J* = 3.1, 3.1, 1.0 Hz, 1H), 4.72 (ddd, *J* = 3.0, 3.0, 1.0 Hz, 1H), 4.42 (ddd, *J* = 3.0, 3.0, 1.0 Hz, 1H), 4.18 (ddd, *J* = 3.1, 3.1, 1.0 Hz, 1H), 3.54 (dd, *J* = 18.4, 3.5 Hz, 1H), 3.39 – 3.32 (m, 4H), 3.07 (s, 1H), 2.98 – 2.89 (m, 2H), 2.81 (s, 1H), 2.77 – 2.63 (m, 5H), 2.41 – 2.34 (m, 2H), 2.31 (dd, *J* = 17.8, 2.5 Hz, 1H), 2.23 (d, *J* = 14.2 Hz, 1H).


207.11
205.90
157.98
157.79
137.72
137.68
133.88
133.30
129.95
129.77
128.89
123.67
123.02
116.02
115.95
110.12
110.11
98.35
97.71
87.93
87.83
84.72
81.25
52.76
51.71
46.37
45.76
40.29
40.22
39.89
39.87
39.20
38.96
O
O
O
OH
CH2
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

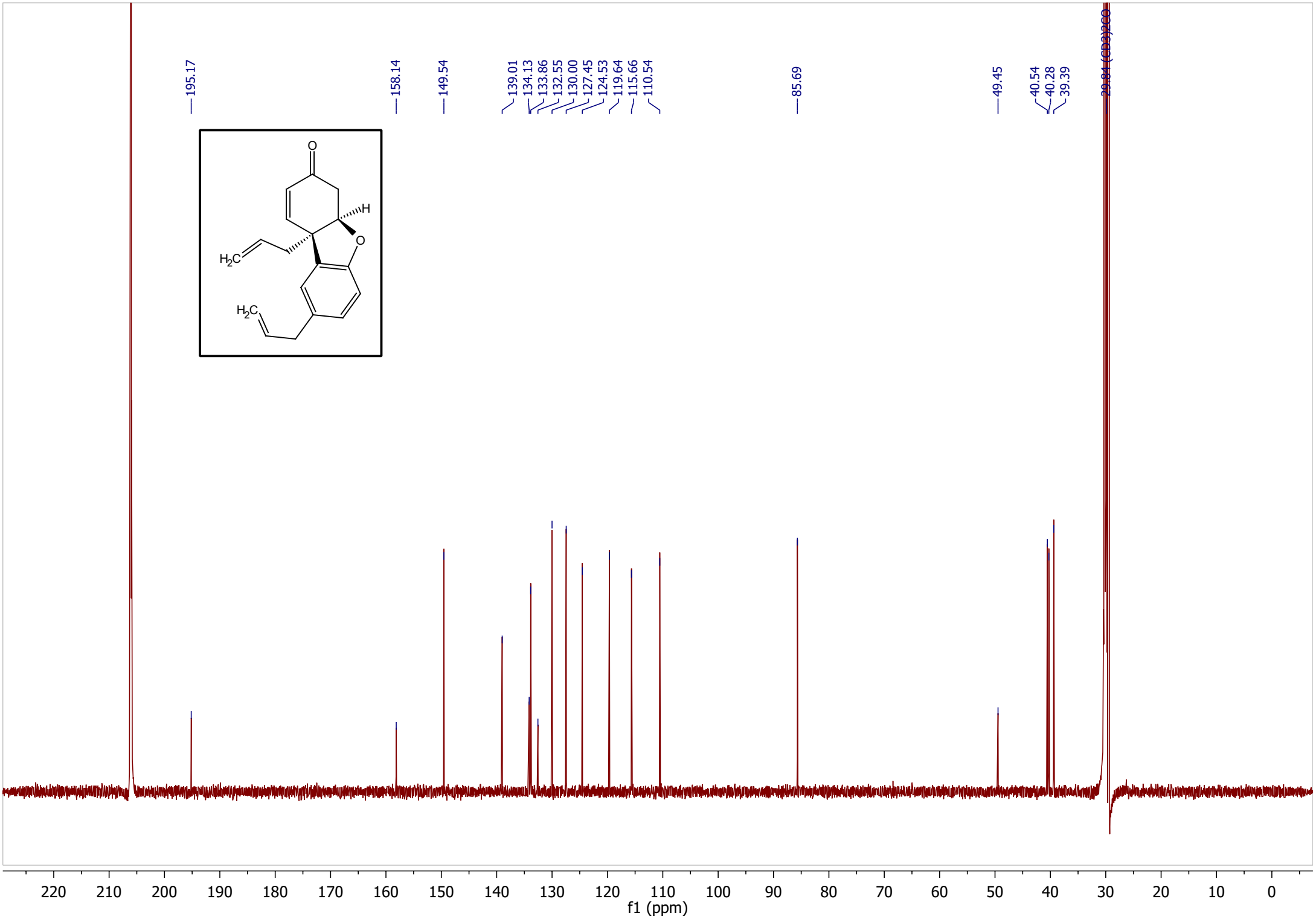


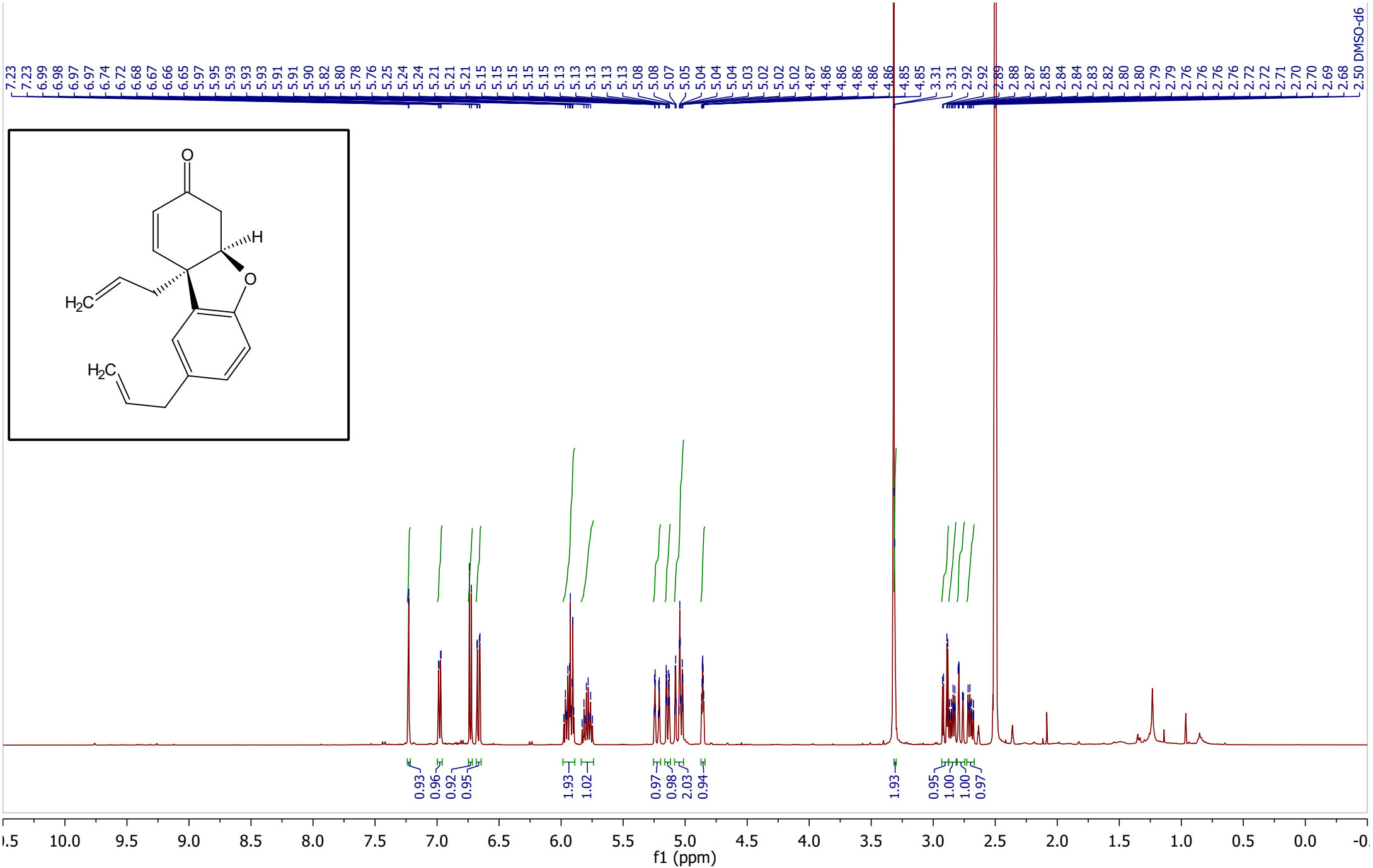
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.03 – 6.99 (m, 2H), 6.76 – 6.72 (m, 1H), 6.50 (dd, *J* = 10.2, 1.9 Hz, 1H), 6.01 (dd, *J* = 10.2, 0.7 Hz, 1H), 6.00 – 5.87 (m, 1H), 5.79 (dddd, *J* = 16.9, 10.1, 8.1, 6.7 Hz, 1H), 5.24 – 5.16 (m, 2H), 5.11 – 5.04 (m, 2H), 4.82 (ddd, *J* = 4.6, 2.9, 1.9 Hz, 1H), 3.35 (ddd, *J* = 6.8, 1.5, 1.5 Hz, 2H), 3.00 (ddd, *J* = 17.6, 2.9, 0.8 Hz, 1H), 2.80 (dddd, *J* = 14.1, 6.7, 1.5, 1.5 Hz, 1H), 2.76 (dd, *J* = 17.6, 4.2 Hz, 1H), 2.64 (dddd, *J* = 14.1, 8.1, 1.0, 1.0 Hz, 1H).


195.46
157.23
148.60
137.76
133.48
132.27
131.19
129.63
127.40
123.10
119.82
115.93
110.39
84.86
77.16 CDCl3
48.61
40.81
39.86
38.93
O
H
O
H2C
H2C
220
210
200
190
180
170
160
150
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130
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110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

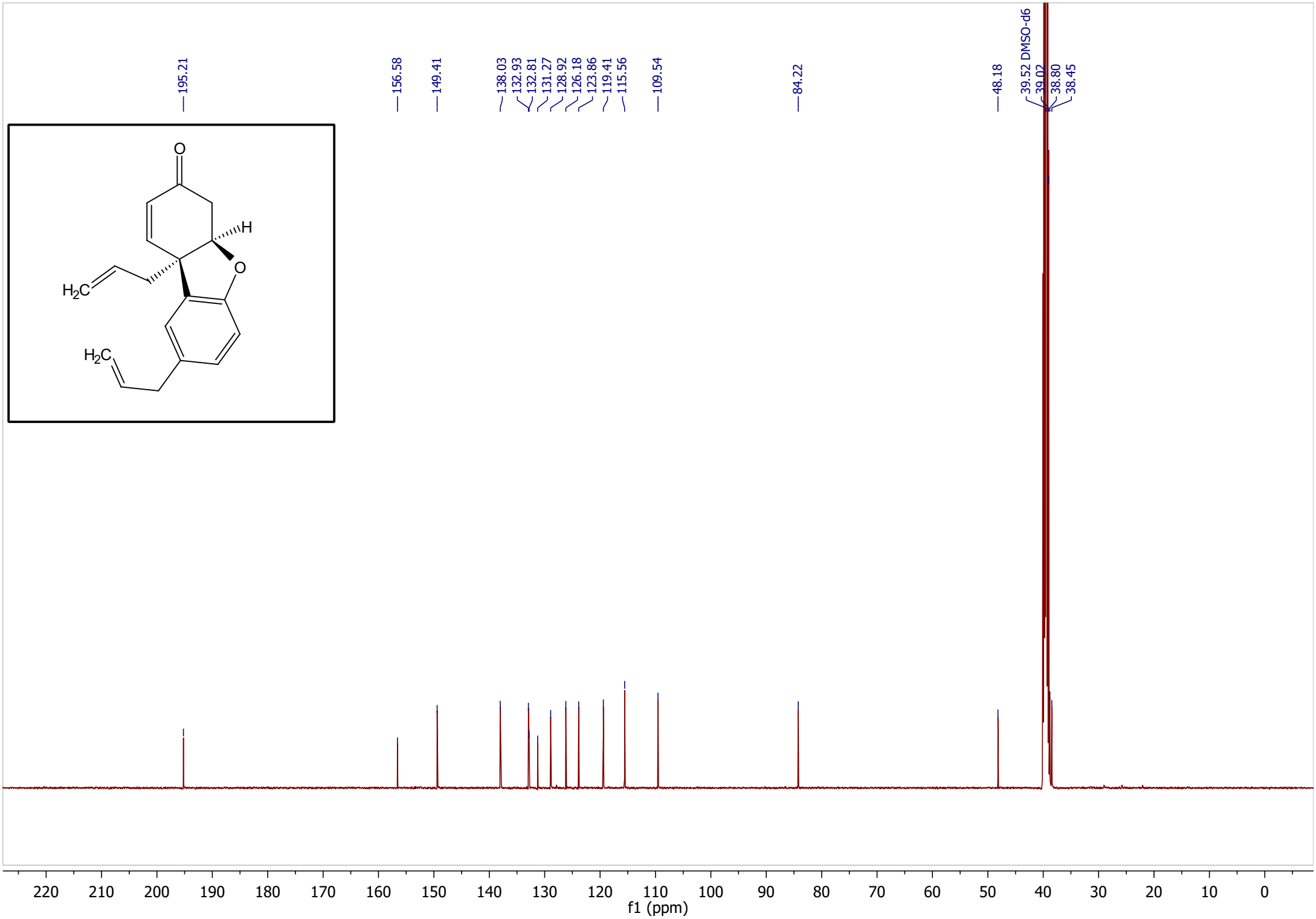


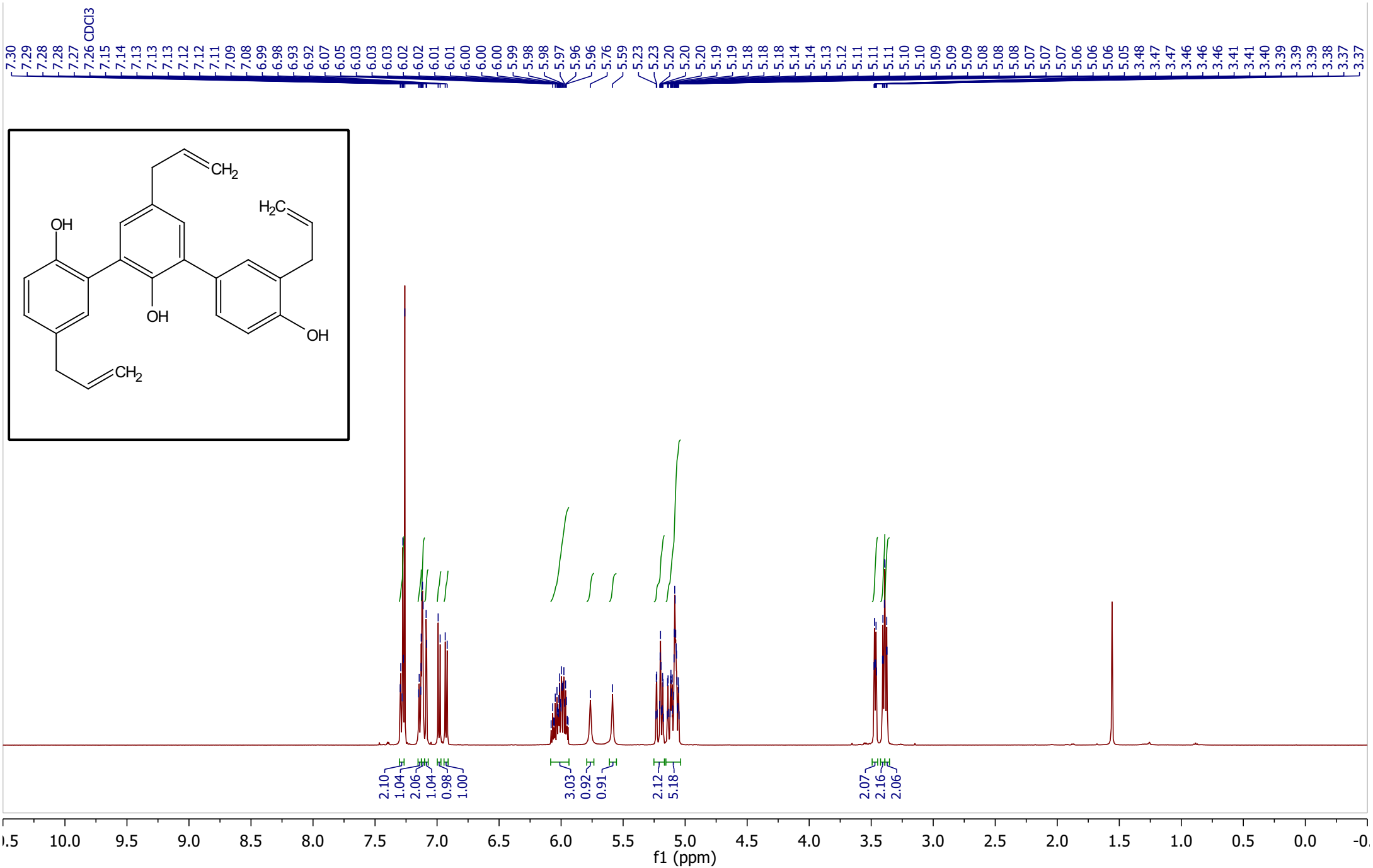
^{1}H NMR (500 MHz, Acetone-d_6) δ 7.24 (d, J = 1.8 Hz, 1H), 7.02 (dd, J = 8.1, 1.8 Hz, 1H), 6.71 (d, J = 8.1 Hz, 1H), 6.66 (dd, J = 10.2, 1.9 Hz, 1H), 6.04 – 5.81 (m, 2H), 5.91 (d, J = 10.2 Hz, 1H), 5.30 – 5.15 (m, 2H), 5.09 – 4.98 (m, 2H), 4.91 – 4.88 (m, 1H), 3.35 (ddd, J = 6.6, 1.5 Hz, 2H), 2.93 (dddd, J = 14.2, 7.2, 1.4, 1.4 Hz, 1H), 2.88 – 2.83 (m, 2H), 2.77 – 2.72 (m, 1H).


195.17
158.14
149.54
139.01
134.13
133.86
132.55
130.00
127.45
124.53
119.64
115.66
110.54
85.69
49.45
40.54
40.28
39.39
29.84
(CD3)2CO
O
H
O
H2C
H2C
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

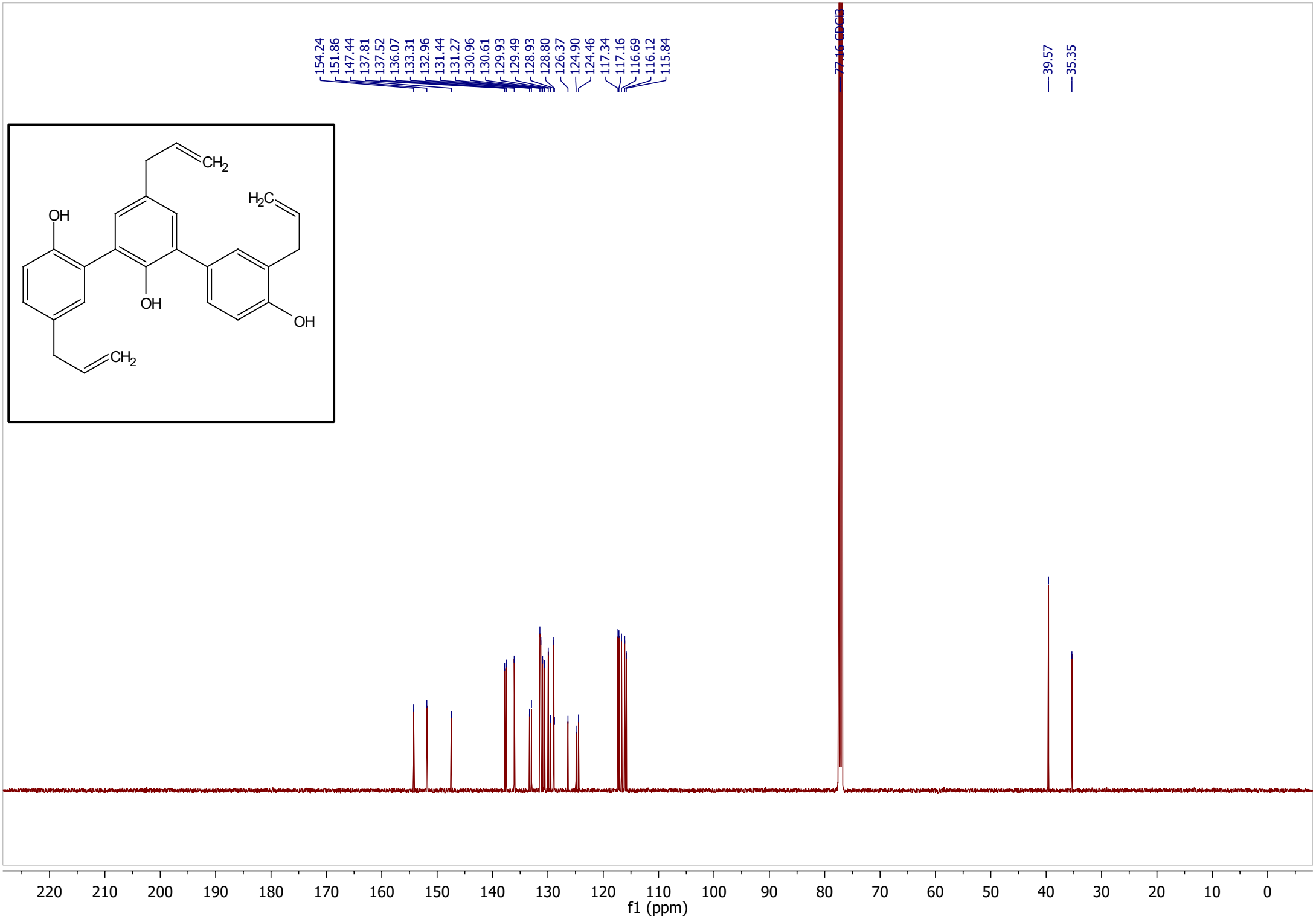


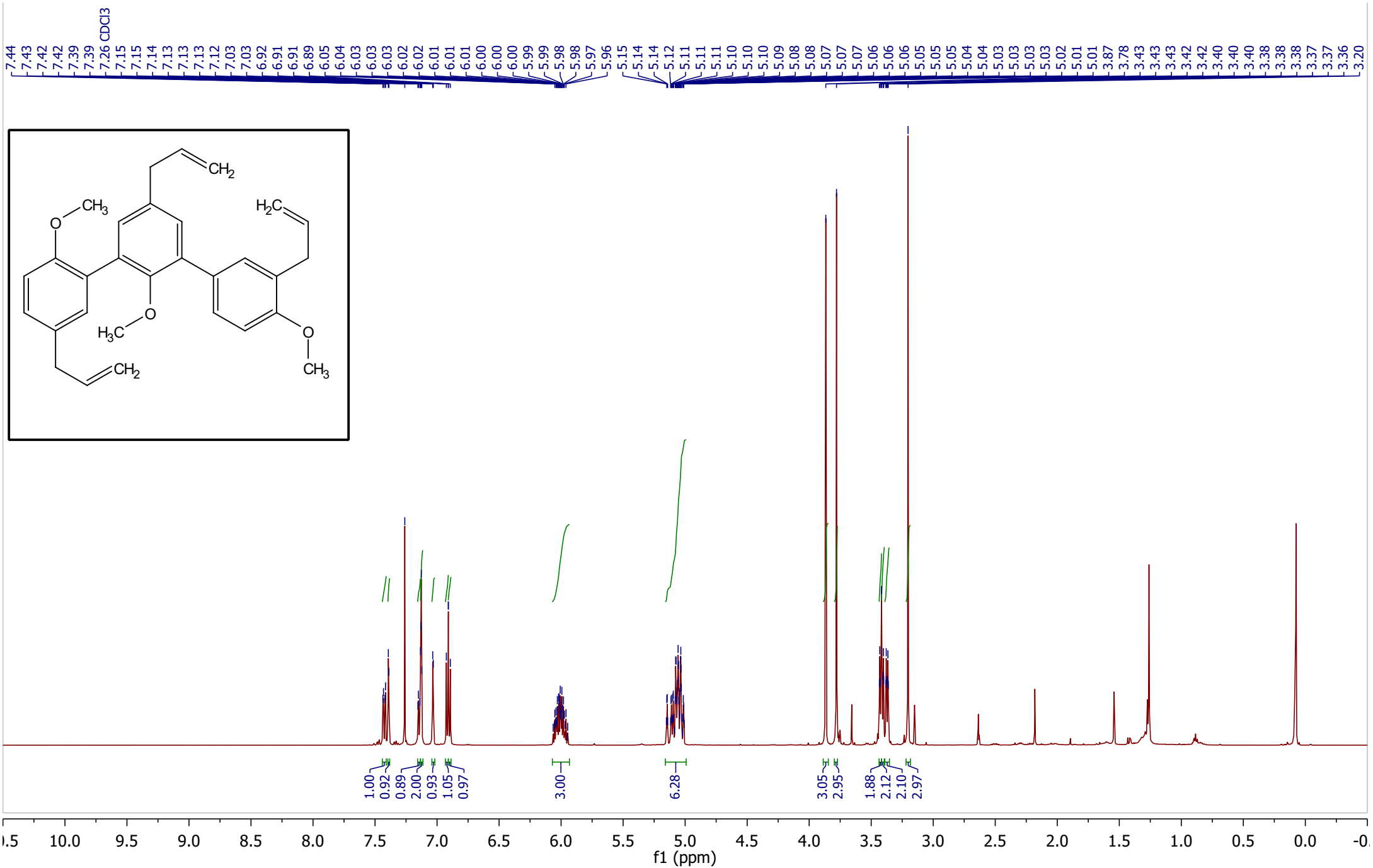
^{1}H NMR (500 MHz, DMSO-d_6) δ 7.23 (d, J = 1.9 Hz, 1H), 6.98 (dd, J = 8.1, 1.9 Hz, 1H), 6.73 (d, J = 8.1 Hz, 1H), 6.67 (dd, J = 10.2, 1.9 Hz, 1H), 5.94 (dddd, J = 16.8, 10.0, 6.7 Hz, 1H), 5.92 (dd, J = 10.2, 0.9 Hz, 1H), 5.79 (dddd, J = 17.4, 10.1, 7.4 Hz, 1H), 5.27 – 5.11 (m, 2H), 5.09 – 5.01 (m, 2H), 4.88 – 4.84 (m, 1H), 3.31 (d, J = 6.7 Hz, 2H), 2.90 (dd, J = 17.5, 3.9 Hz, 1H), 2.85 (dddd, J = 14.0, 7.4, 1.2 Hz, 1H), 2.78 (ddd, J = 17.5, 2.7, 0.9 Hz, 1H), 2.70 (dddd, J = 14.0, 7.4, 1.1 Hz, 1H).


195.21
156.58
149.41
138.03
132.93
132.81
131.27
128.92
126.18
123.86
119.41
115.56
109.54
84.22
48.18
39.52 DMSO-d6
39.02
38.80
38.45
O
H
O
H2C
H2C
220
210
200
190
180
170
160
150
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130
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100
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70
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50
40
30
20
10
0
f1 (ppm)

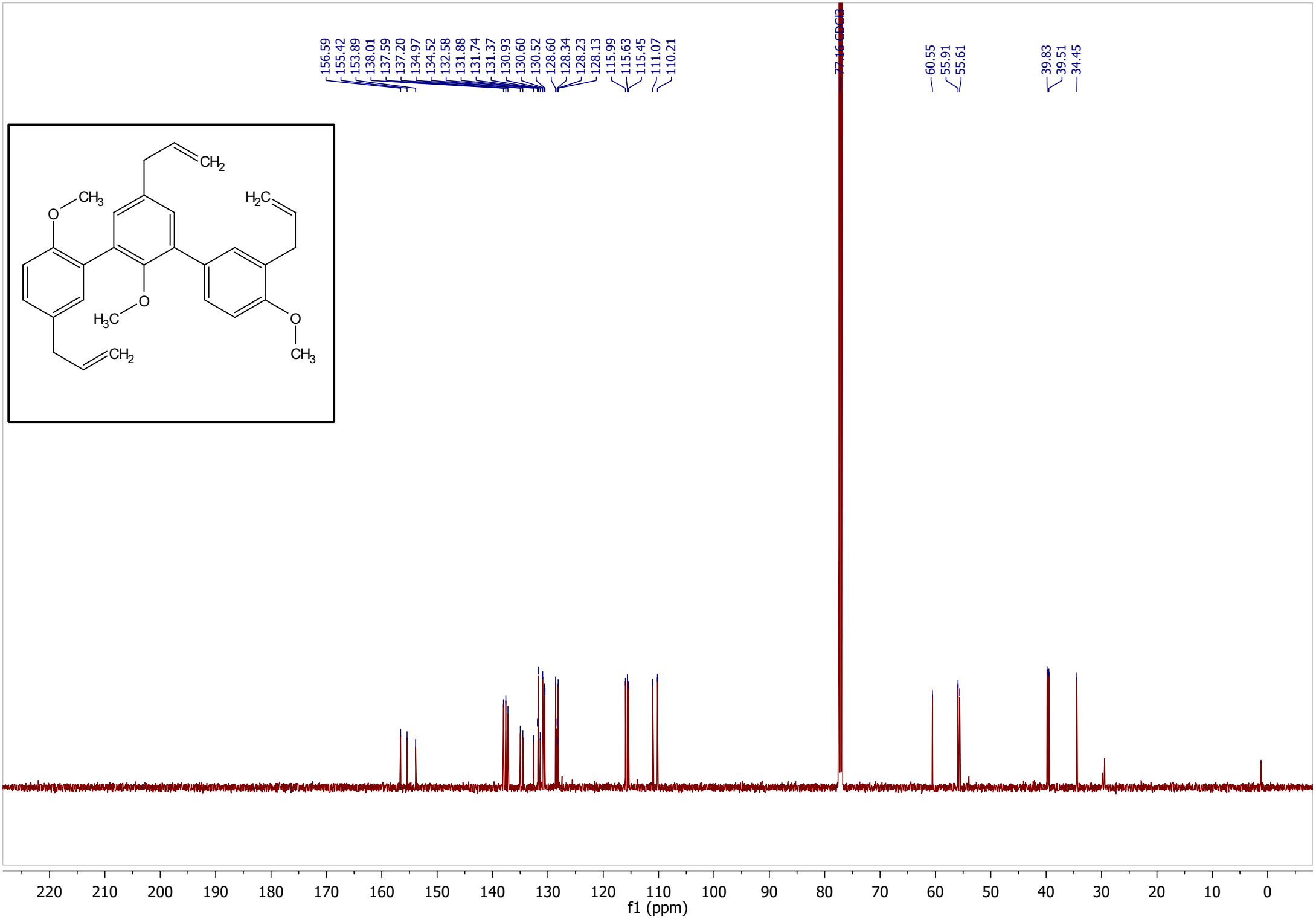


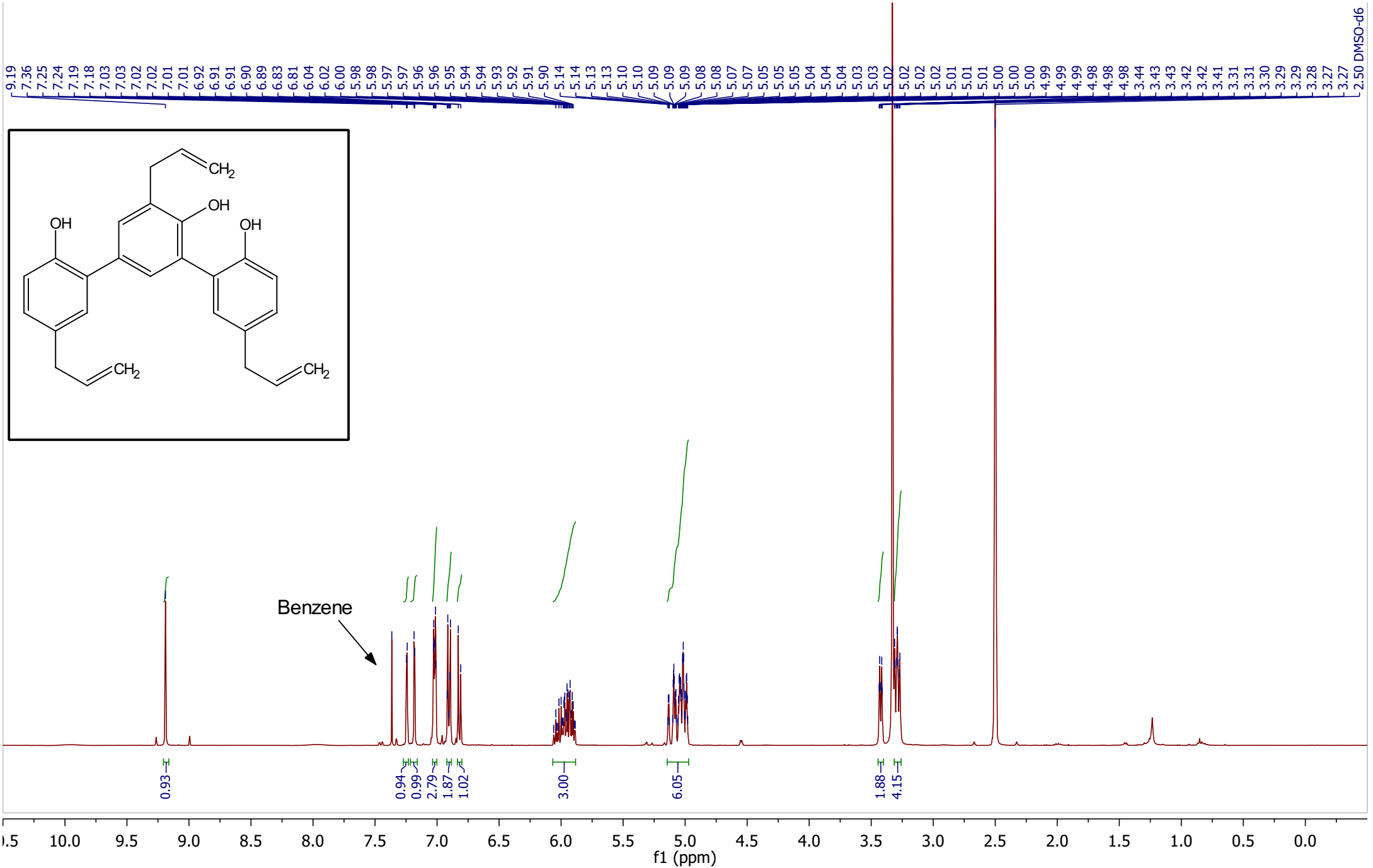
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.30 – 7.27 (m, 2H), 7.14 (dd, *J* = 8.0, 1.8 Hz, 1H), 7.13 – 7.11 (m, 2H), 7.09 (d, *J* = 2.2 Hz, 1H), 6.98 (d, *J* = 8.1 Hz, 1H), 6.93 (d, *J* = 7.9 Hz, 1H), 6.08 – 5.94 (m, 3H), 5.76 (s, 1H), 5.59 (s, 1H), 5.24 – 5.17 (m, 2H), 5.15 – 5.04 (m, 5H), 3.47 (ddd, *J* = 6.4, 1.6, 1.6 Hz, 2H), 3.40 (ddd, *J* = 7.4, 1.4, 1.4 Hz, 2H), 3.38 (ddd, *J* = 7.4, 1.4, 1.4 Hz, 2H).


154.24
151.86
147.44
137.81
137.52
136.07
133.31
132.96
131.44
131.27
130.96
130.61
129.93
129.49
128.93
128.80
126.37
124.90
124.46
117.34
117.16
116.69
116.12
115.84
77.16 CDCl3
39.57
35.35
OH
OH
OH
CH2
H2C
CH2
220
210
200
190
180
170
160
150
140
130
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100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

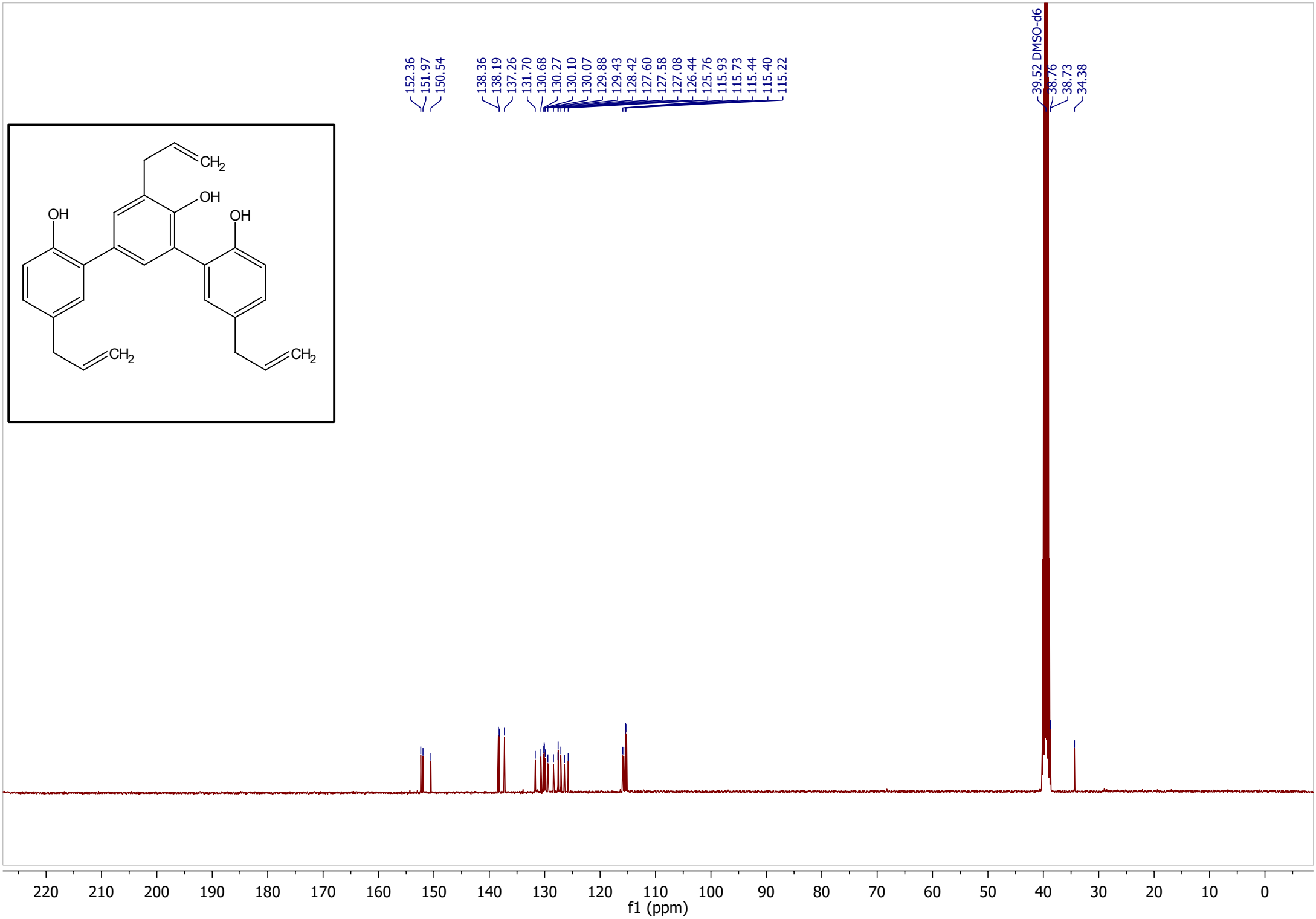


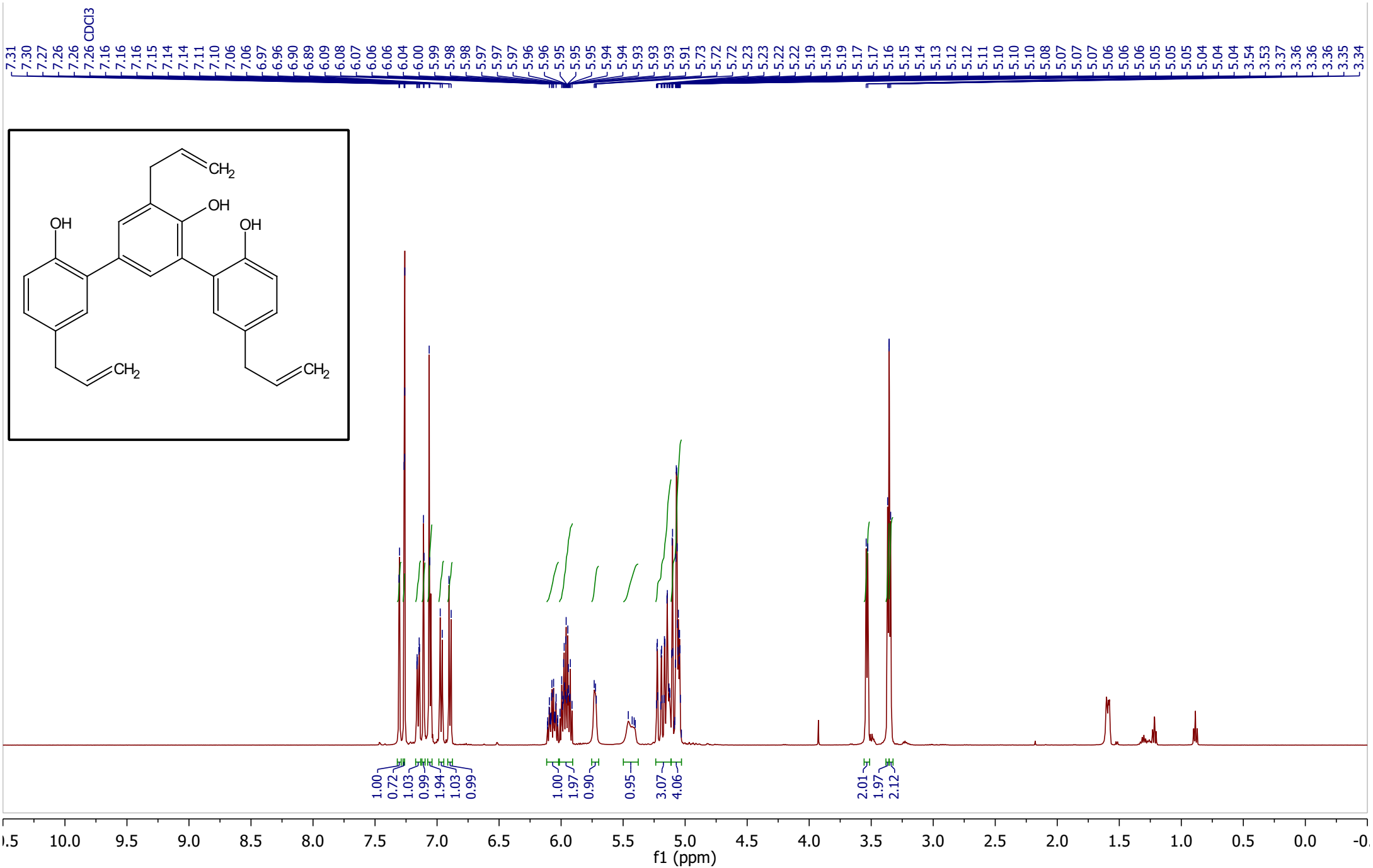
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.43 (dd, *J* = 8.4, 2.3 Hz, 1H), 7.39 (d, *J* = 2.3 Hz, 1H), 7.15 (dd, *J* = 8.3, 2.2 Hz, 1H), 7.14 – 7.12 (m, 2H), 7.03 (d, *J* = 2.4 Hz, 1H), 6.92 (d, *J* = 8.3 Hz, 1H), 6.90 (d, *J* = 8.4 Hz, 1H), 6.08 – 5.93 (m, 3H), 5.17 – 5.00 (m, 6H), 3.87 (s, 3H), 3.78 (s, 3H), 3.43 (ddd, *J* = 7.1, 1.6, 1.6 Hz, 1H), 3.41 (ddd, *J* = 7.1, 1.4, 1.4 Hz, 2H), 3.37 (ddd, *J* = 6.7, 1.5, 1.5 Hz, 2H), 3.20 (s, 3H).


156.59
155.42
153.89
138.01
137.59
137.20
134.97
134.52
132.58
131.88
131.74
131.37
130.93
130.60
130.52
128.60
128.34
128.23
128.13
115.99
115.63
115.45
111.07
110.21
77.16 CDCl3
60.55
55.91
55.61
39.83
39.51
34.45
CH2
CH3
O
H2C
O
H3C
O
CH2
CH3
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

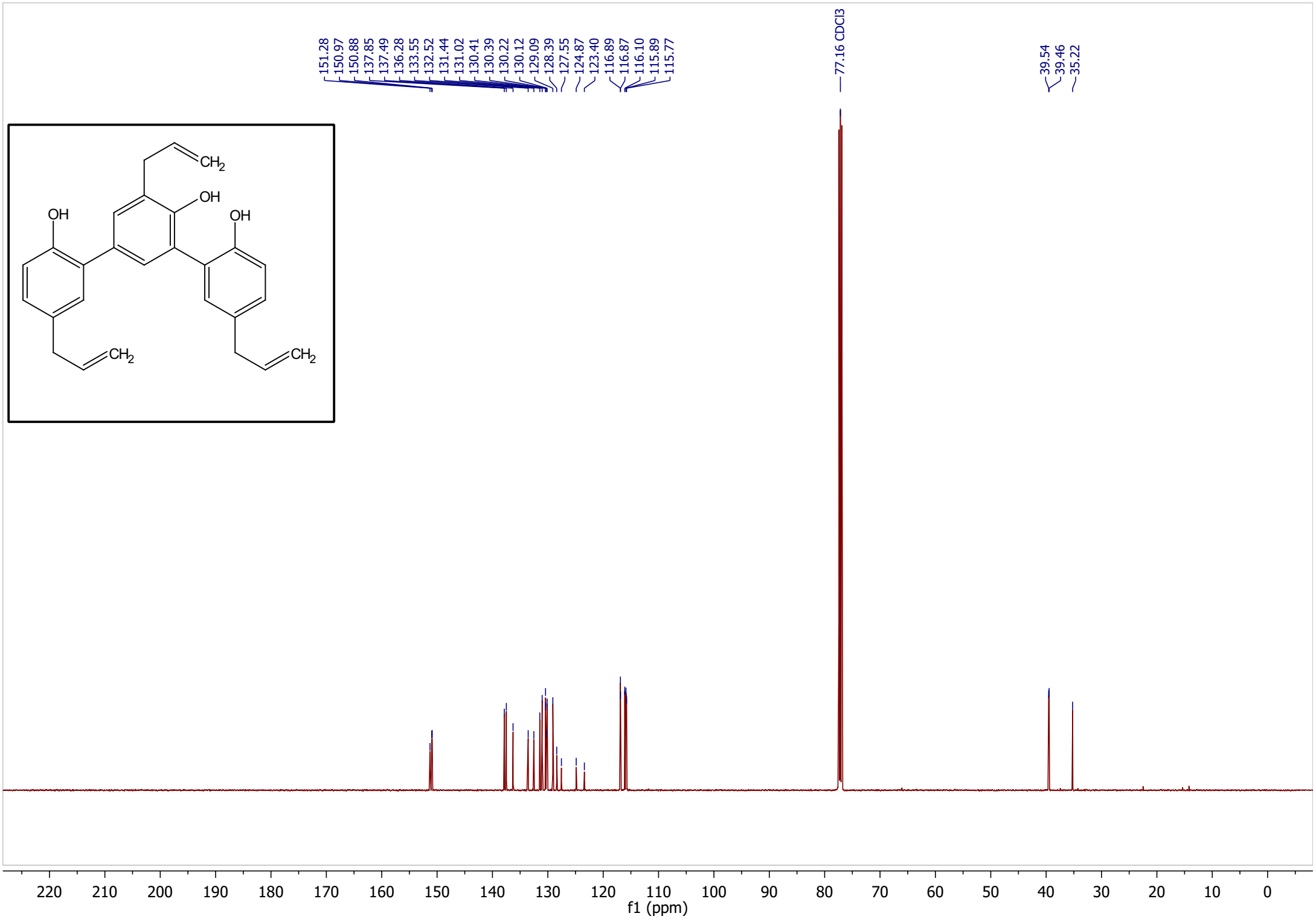


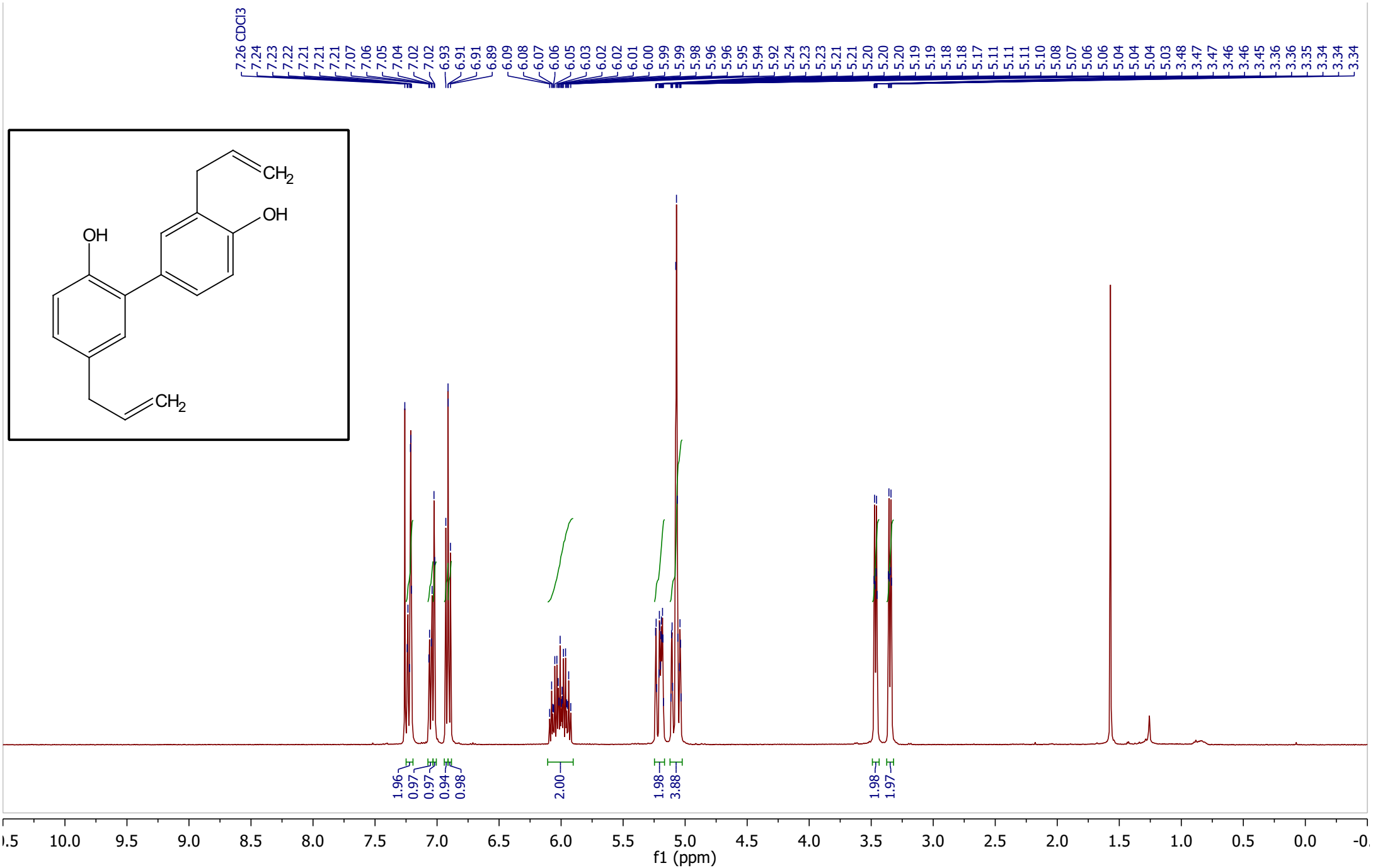
^{1}H NMR (400 MHz, DMSO-d_6) δ 9.19 (s, 1H), 7.24 (d, J = 2.3 Hz, 1H), 7.18 (d, J = 2.3 Hz, 1H), 7.04 – 7.00 (m, 3H), 6.93 – 6.88 (m, 2H), 6.82 (d, J = 8.1 Hz, 1H), 6.07 – 5.88 (m, 3H), 5.15 – 4.97 (m, 6H), 3.43 (ddd, J = 6.7, 1.5, 1.5 Hz, 2H), 3.30 (ddd, J = 6.5, 1.9, 1.9 Hz, 2H), 3.28 (ddd, J = 6.2, 1.8, 1.8 Hz, 2H).


152.36
151.97
150.54
138.36
138.19
137.26
131.70
130.68
130.27
130.10
130.07
129.88
129.43
128.42
127.60
127.58
127.08
126.44
125.76
115.93
115.73
115.44
115.40
115.22
39.52 DMSO-d6
38.76
38.73
34.38
OH
OH
OH
CH2
CH2
CH2
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

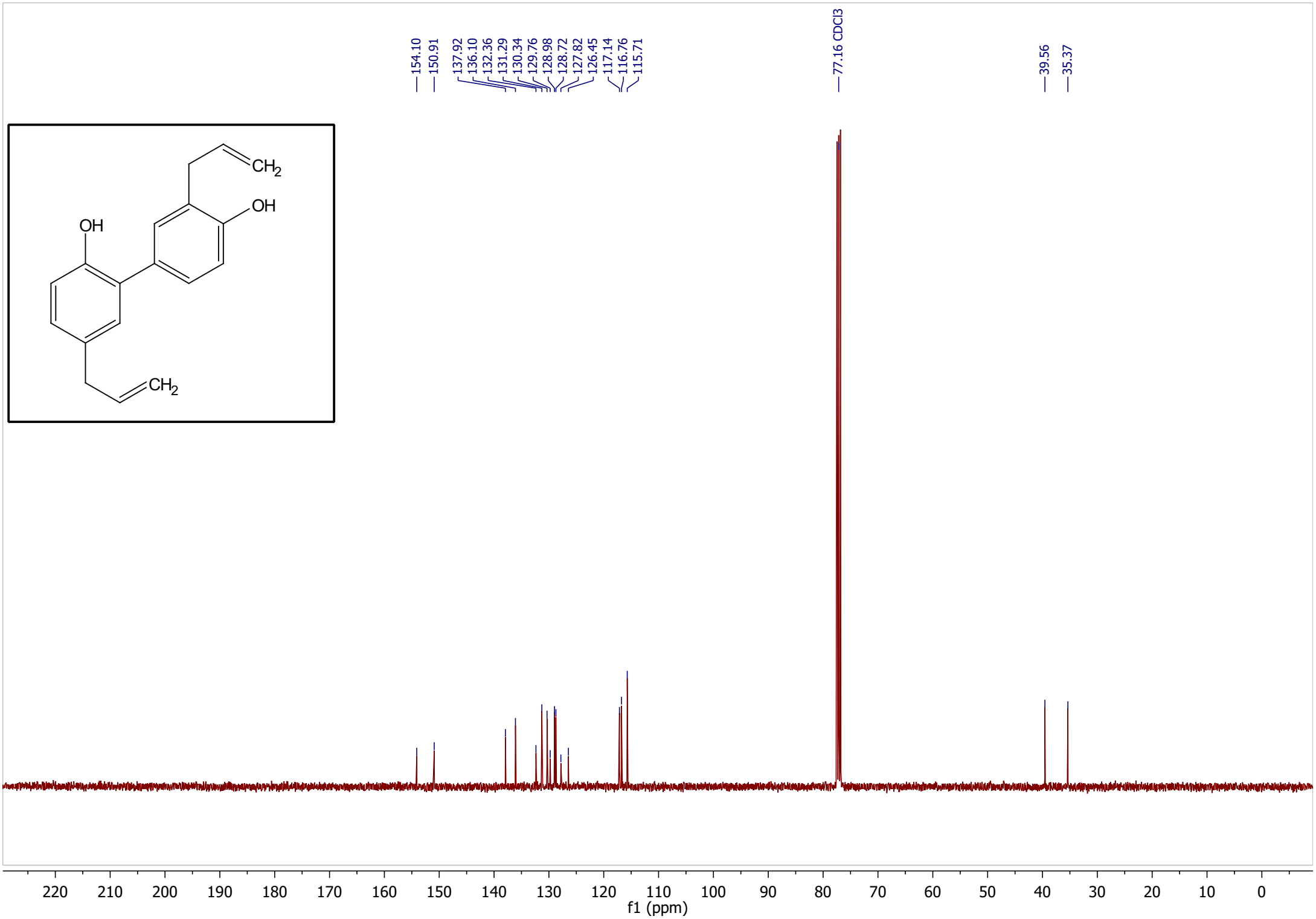


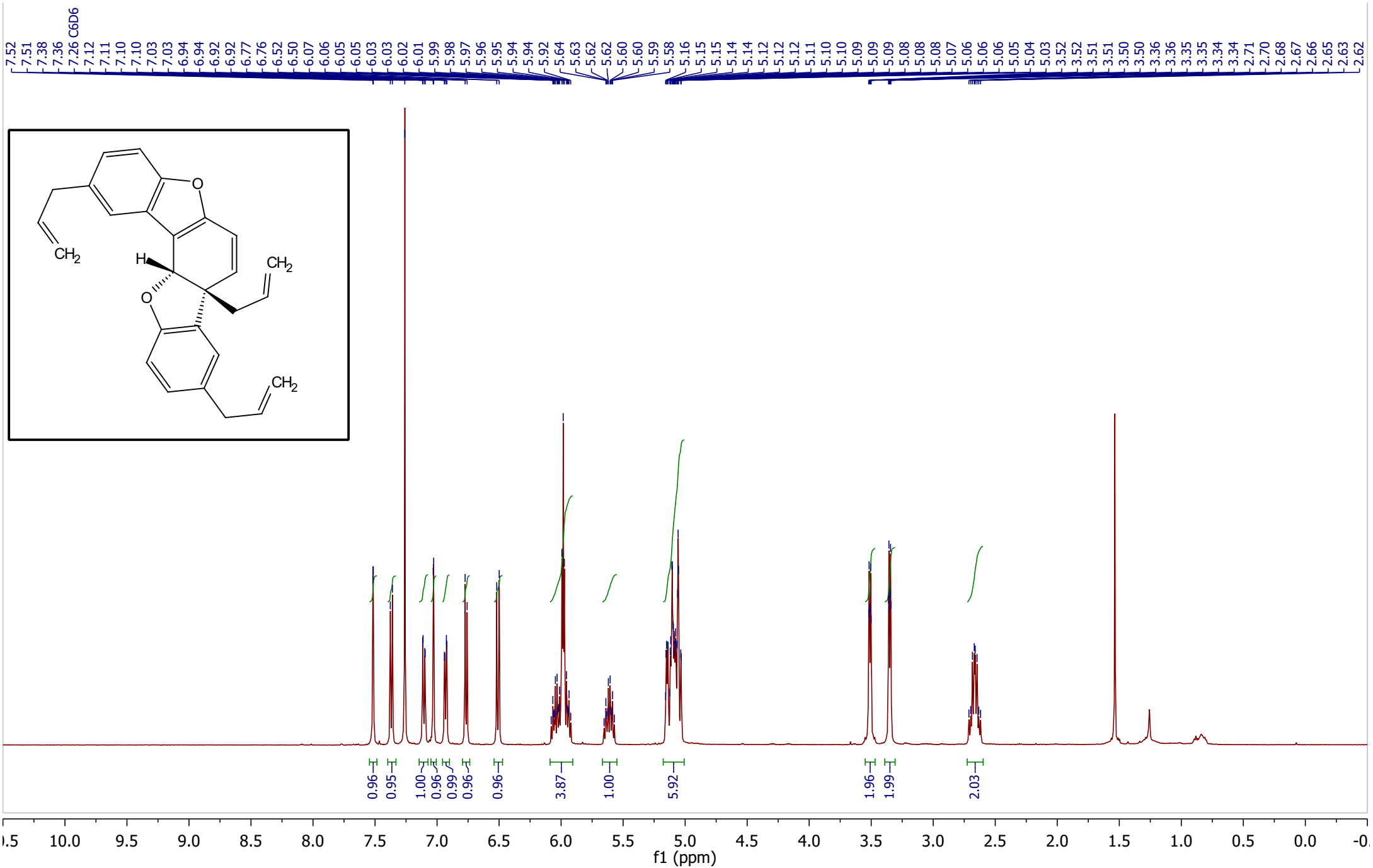
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.30 (d, *J* = 2.2 Hz, 1H), 7.26 (d, *J* = 2.2 Hz, 1H), 7.15 (dd, *J* = 8.3, 2.3 Hz, 1H), 7.11 (d, *J* = 2.2 Hz, 1H), 7.06 (d, *J* = 2.2 Hz, 1H), 7.06 (dd, *J* = 8.9, 2.2 Hz, 1H), 6.97 (d, *J* = 8.2 Hz, 1H), 6.90 (d, *J* = 8.9 Hz, 1H), 6.07 (dddd, *J* = 16.8, 10.0, 6.7, 1.2 Hz, 1H), 5.98 (ddt, *J* = 16.9, 10.3, 6.6 Hz, 1H), 5.93 (ddt, *J* = 16.9, 10.3, 6.6 Hz, 1H), 5.75 – 5.70 (m, 1H), 5.49 – 5.38 (m, 1H), 5.24 – 5.13 (m, 2H), 5.13 – 5.11 (m, 1H), 5.11 – 5.03 (m, 4H), 3.53 (d, *J* = 6.6 Hz, 2H), 3.36 (d, *J* = 6.6 Hz, 2H), 3.35 (d, *J* = 6.6 Hz, 2H).


151.28
150.97
150.88
137.85
137.49
136.28
133.55
132.52
131.44
131.02
130.41
130.39
130.22
130.12
129.09
128.39
127.55
124.87
123.40
116.89
116.87
116.10
115.89
115.77
77.16 CDCl3
39.54
39.46
35.22
OH
OH
OH
CH2
CH2
CH2
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

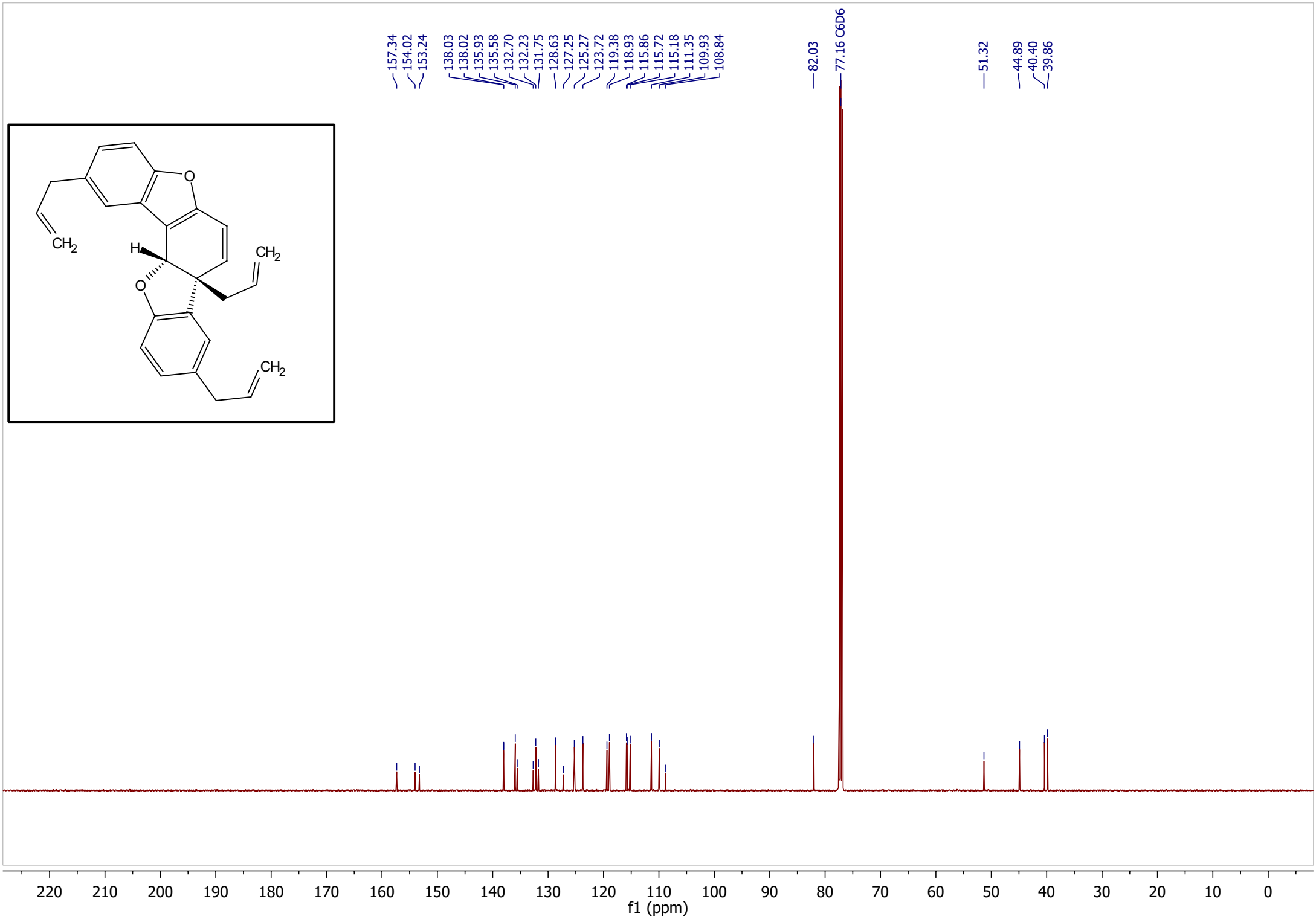


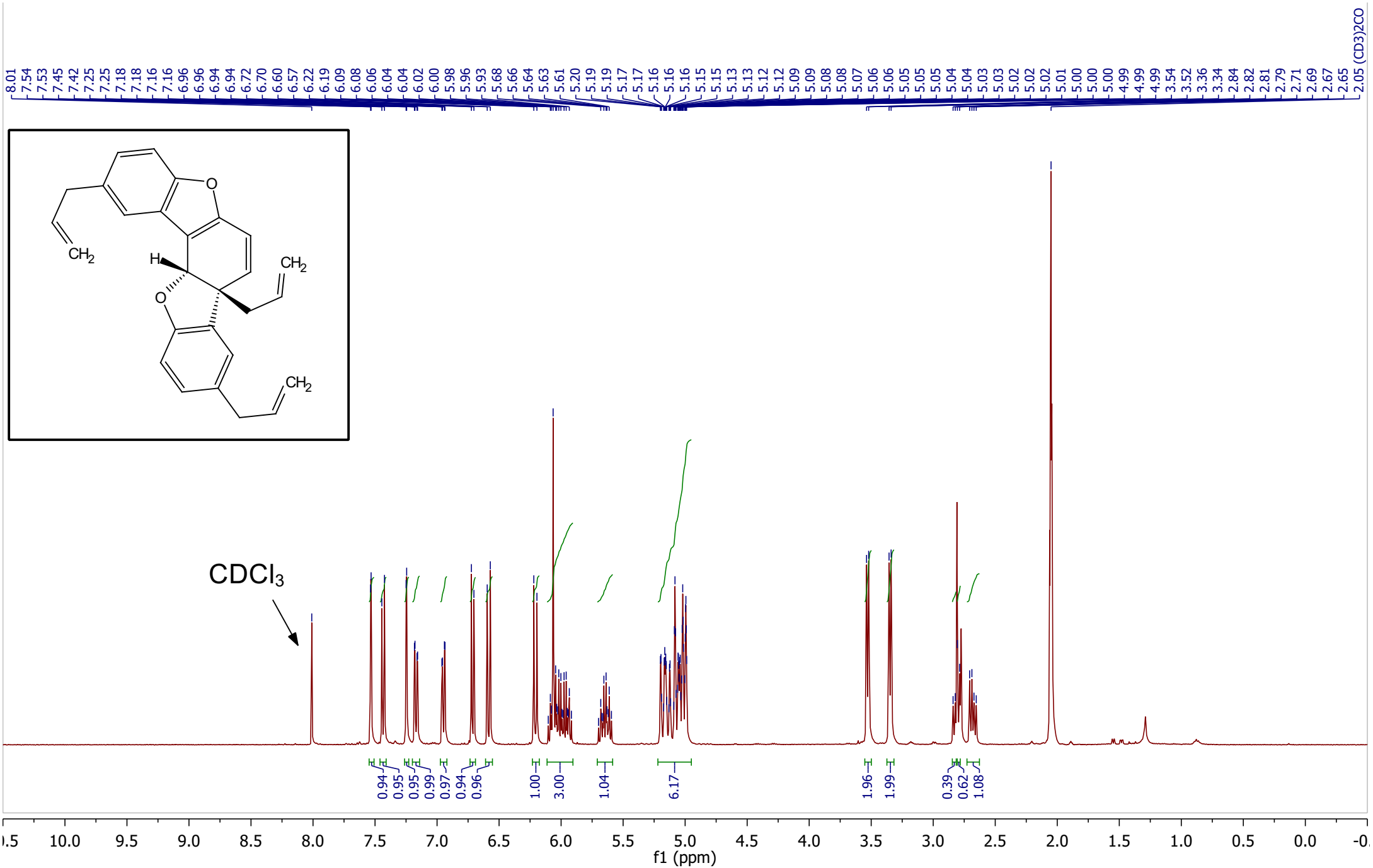
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.25 – 7.20 (m, 2H), 7.05 (dd, *J* = 8.2, 2.2 Hz, 1H), 7.02 (d, *J* = 2.2 Hz, 1H), 6.92 (d, *J* = 8.2 Hz, 1H), 6.90 (d, *J* = 8.2 Hz, 1H), 6.11 – 5.90 (m, 2H), 5.25 – 5.16 (m, 2H), 5.14 – 5.01 (m, 4H), 3.46 (ddd, *J* = 6.4, 1.5, 1.5 Hz, 2H), 3.35 (ddd, *J* = 6.7, 1.5, 1.5 Hz, 2H).


154.10
150.91
137.92
136.10
132.36
131.29
130.34
129.76
128.98
128.72
127.82
126.45
117.14
116.76
115.71
77.16 CDCl3
39.56
35.37
OH
OH
CH2
CH2
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

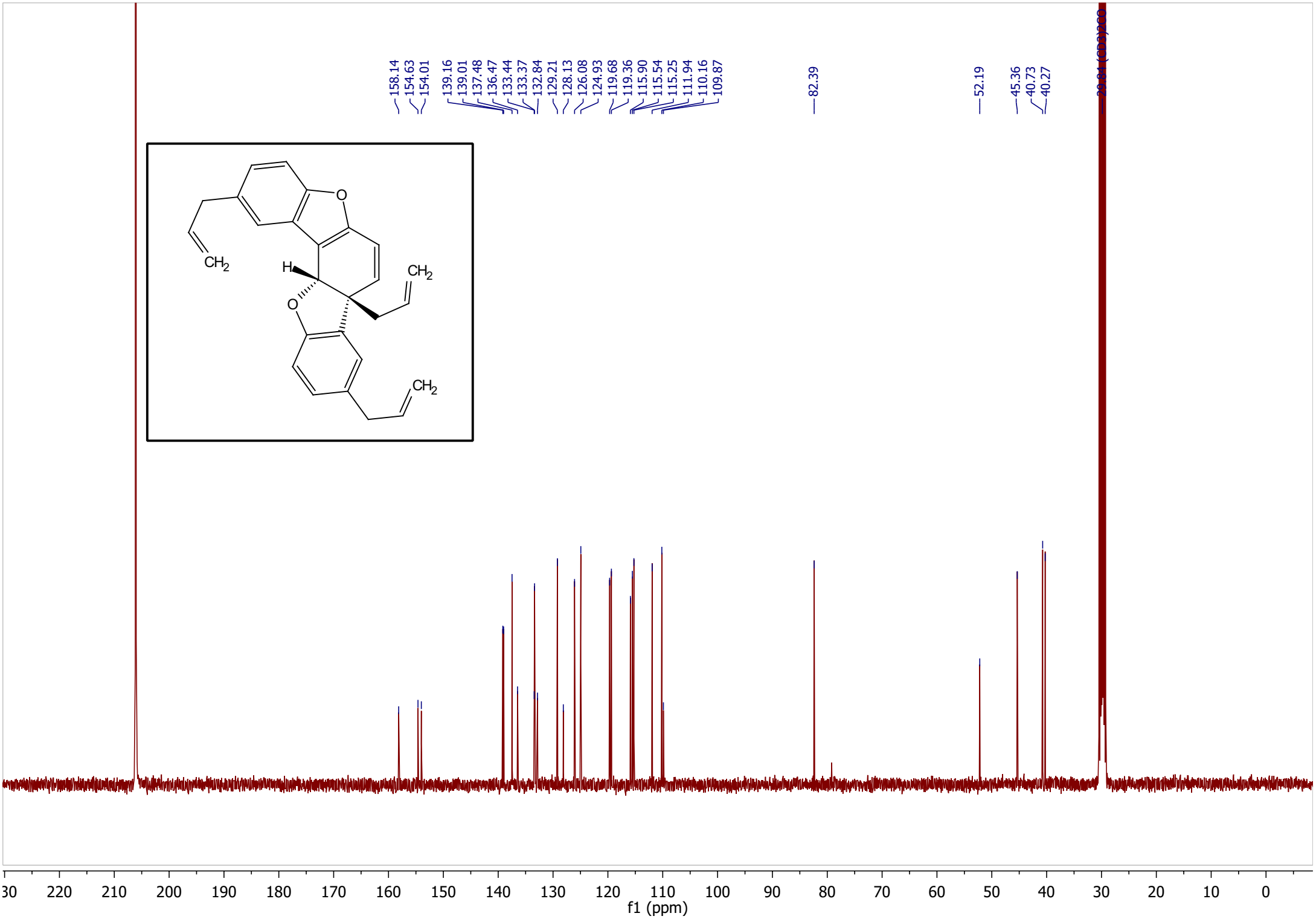


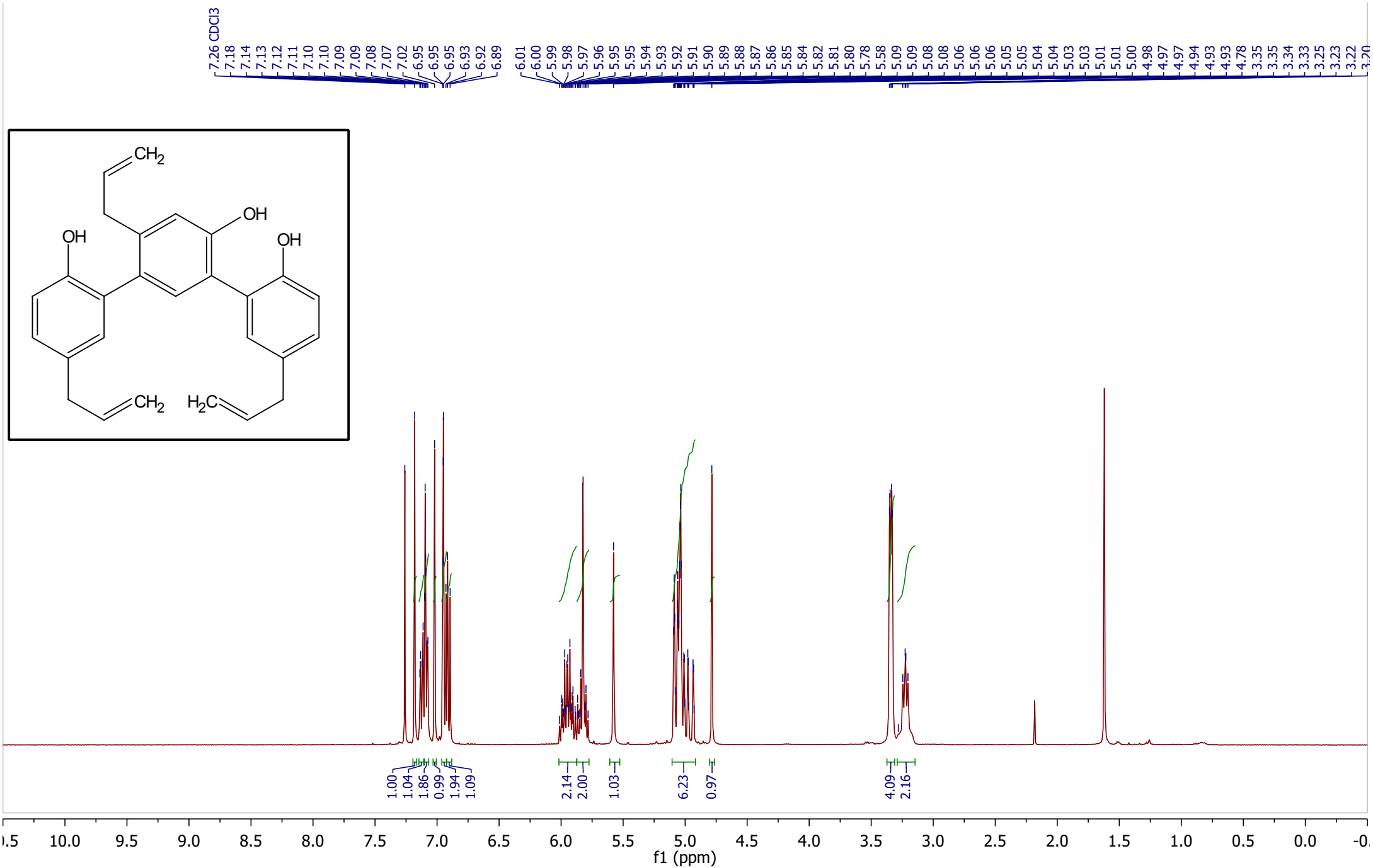
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.52 (d, *J* = 1.8 Hz, 1H), 7.37 (d, *J* = 8.4 Hz, 1H), 7.11 (dd, *J* = 8.4, 1.8 Hz, 1H), 7.03 (d, *J* = 1.9 Hz, 1H), 6.93 (dd, *J* = 8.2, 1.9 Hz, 1H), 6.77 (d, *J* = 8.2 Hz, 1H), 6.51 (d, *J* = 9.9 Hz, 1H), 6.10 – 5.90 (m, 2H), 5.98 (d, *J* = 9.9 Hz, 1H), 5.98 (s, 1H), 5.61 (dddd, *J* = 17.2, 10.1, 7.3, 7.3 Hz, 1H), 5.17 – 5.00 (m, 6H), 3.51 (dddd, *J* = 6.7, 1.6, 1.6 Hz, 2H), 3.35 (ddd, *J* = 6.4, 1.4, 1.4 Hz, 2H), 2.69 (dd, *J* = 14.3, 7.3 Hz, 1H), 2.64 (dd, *J* = 14.3, 7.3 Hz, 1H).


157.34
154.02
153.24
138.03
138.02
135.93
135.58
132.70
132.23
131.75
128.63
127.25
125.27
123.72
119.38
118.93
115.86
115.72
115.18
111.35
109.93
108.84
82.03
77.16 C6D6
51.32
44.89
40.40
39.86
O
CH2
H
CH2
O
CH2
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

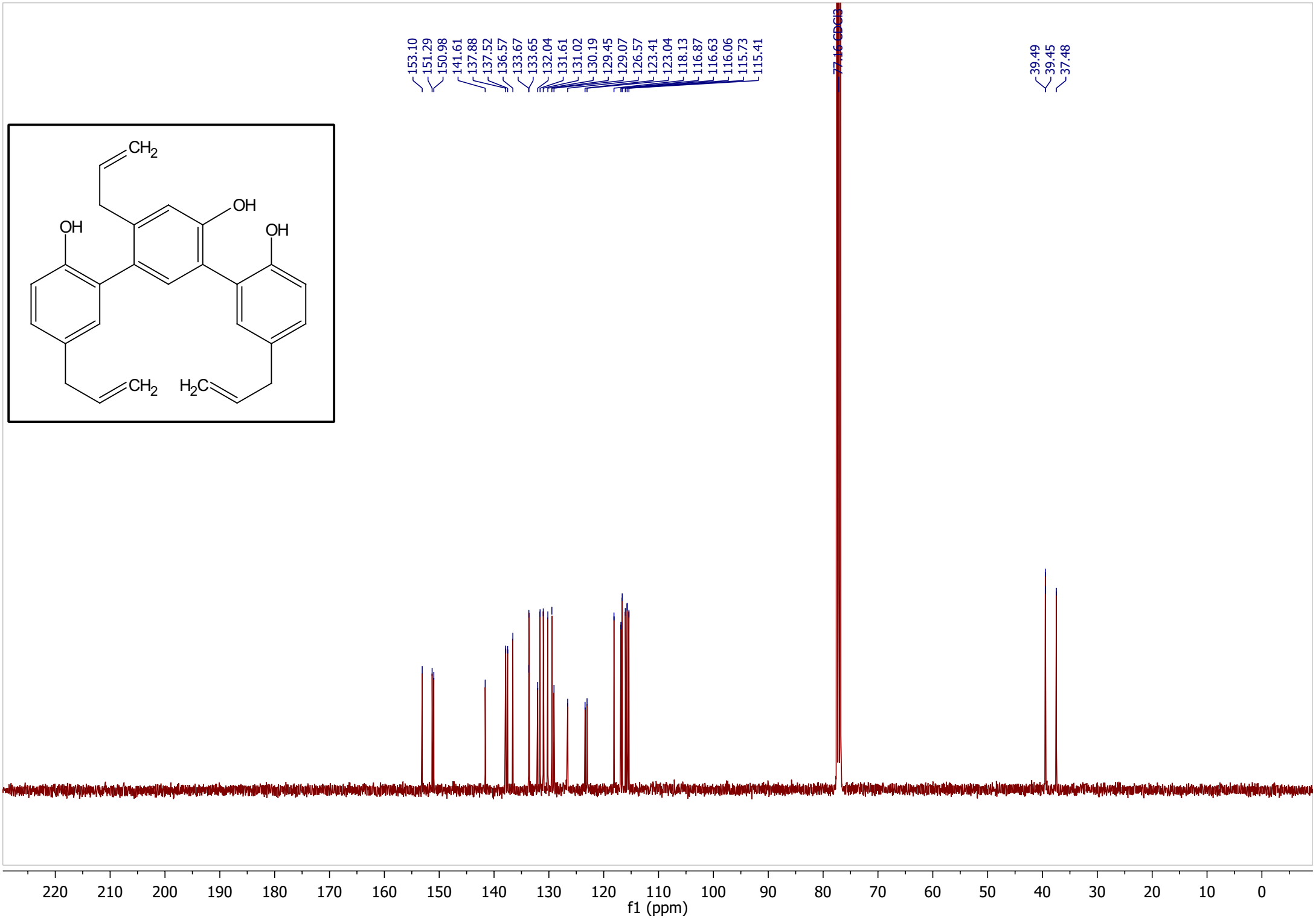


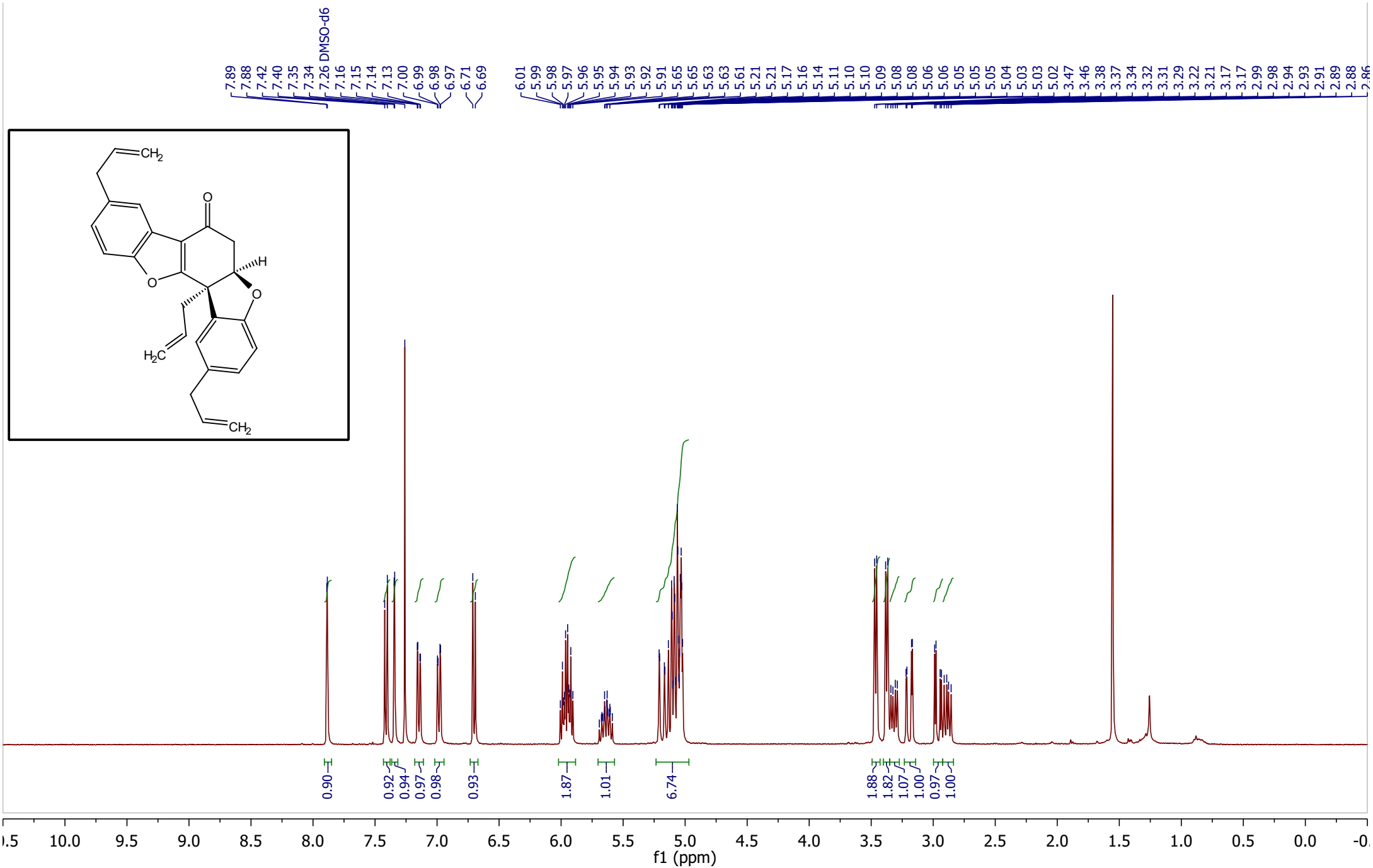
^{1}H NMR (400 MHz, Acetone-d_6) δ 7.53 (d, J = 1.7 Hz, 1H), 7.43 (d, J = 8.4 Hz, 1H), 7.25 (d, J = 1.8 Hz, 1H), 7.17 (dd, J = 8.4, 1.7 Hz, 1H), 6.95 (dd, J = 8.1, 1.8 Hz, 1H), 6.71 (d, J = 8.1 Hz, 1H), 6.58 (d, J = 10.0 Hz, 1H), 6.21 (d, J = 10.0 Hz, 1H), 6.06 (s, 1H), 6.13 – 5.89 (m, 2H), 5.65 (dddd, J = 17.3, 10.2, 7.2, 7.2 Hz, 1H), 5.21 – 4.96 (m, 6H), 3.53 (d, J = 6.8 Hz, 2H), 3.35 (d, J = 6.7 Hz, 2H), 2.81 (dd, J = 14.0, 7.2 Hz, 1H), 2.68 (dd, J = 14.0, 7.2 Hz, 1H).


158.14
154.63
154.01
139.16
139.01
137.48
136.47
133.44
133.37
132.84
129.21
128.13
126.08
124.93
119.68
119.36
115.90
115.54
115.25
111.94
110.16
109.87
82.39
52.19
45.36
40.73
40.27
30.04 (CD3)2CO
O
CH2
H
CH2
O
CH2
30
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

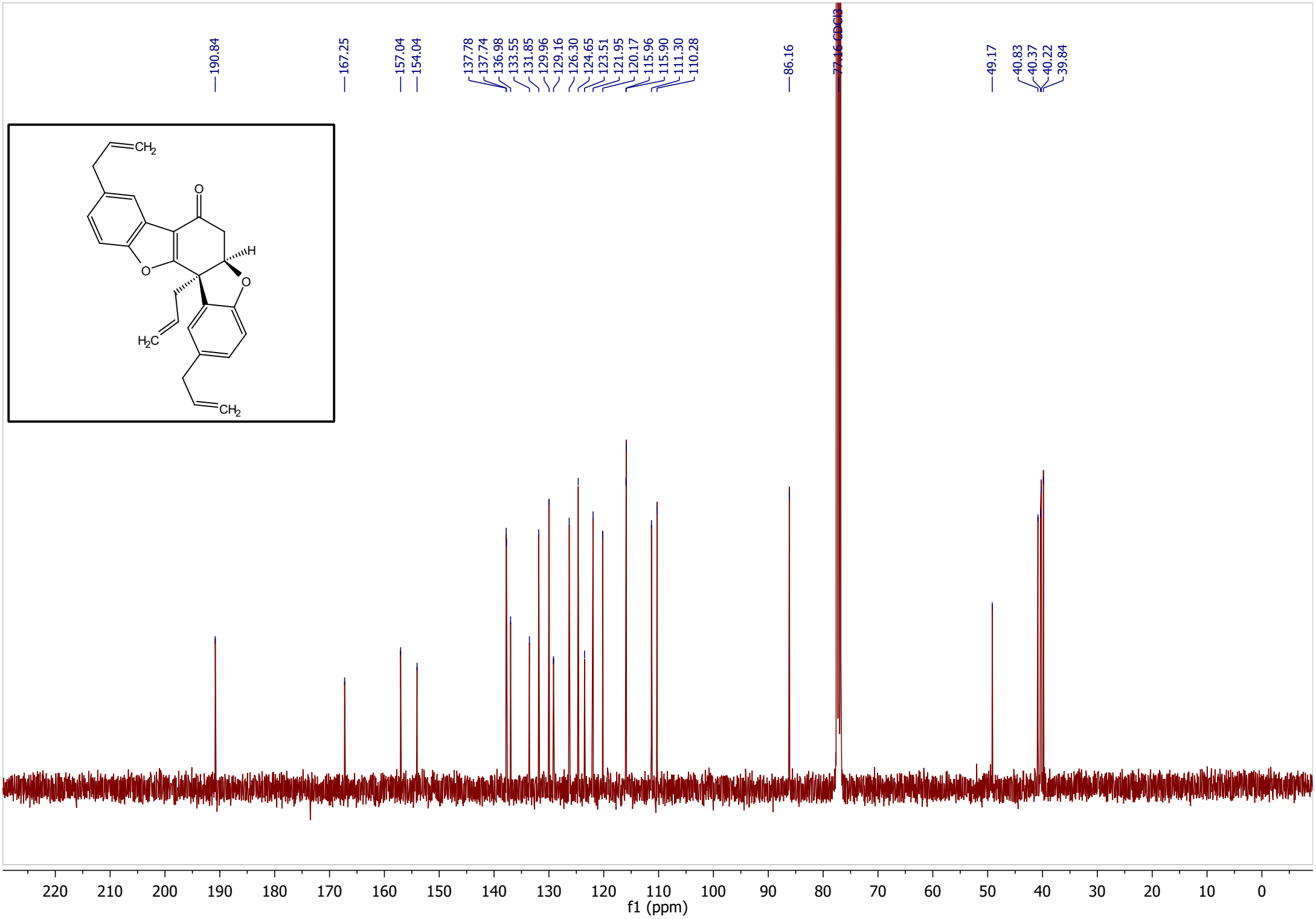


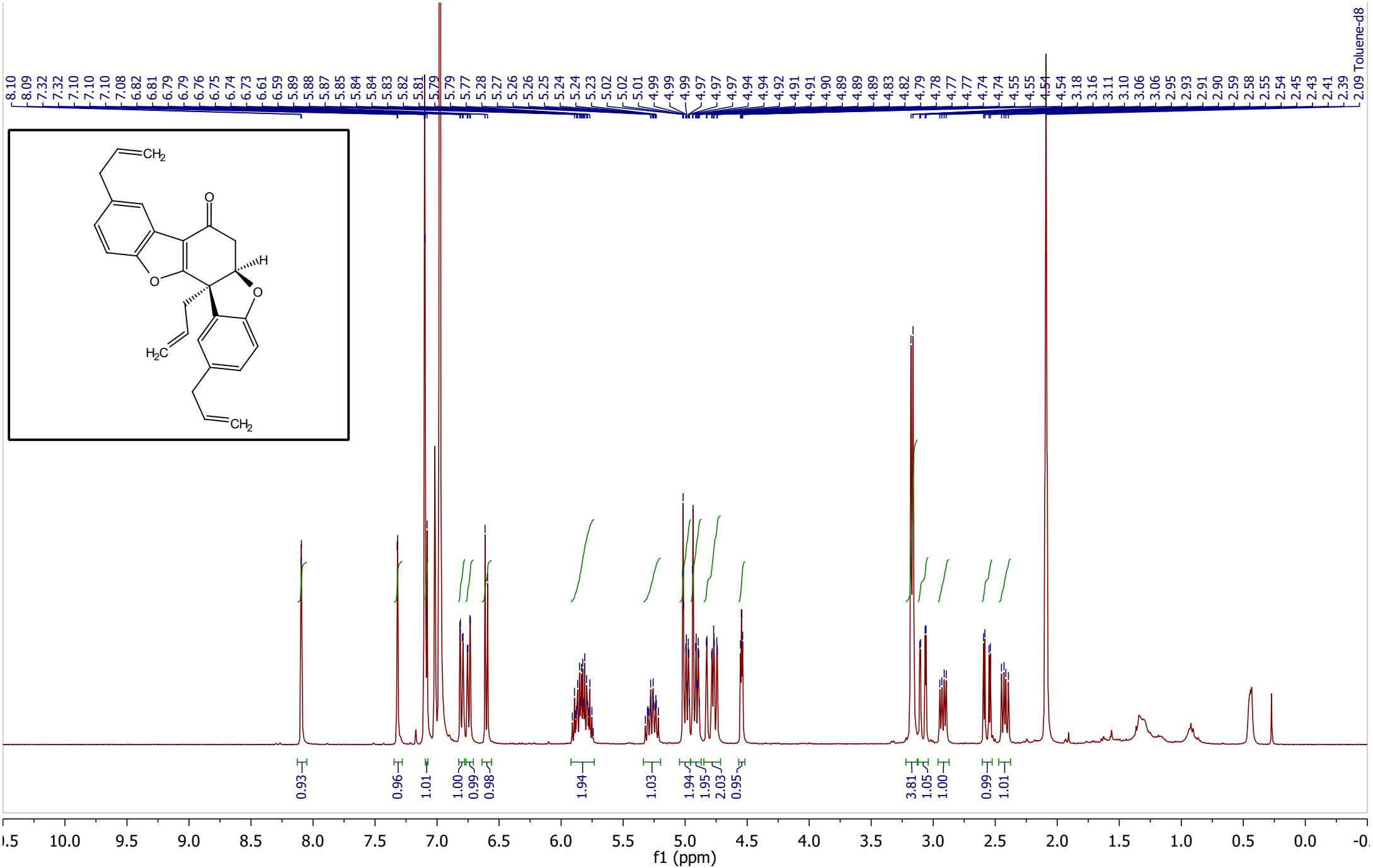
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.18 (s, 1H), 7.13 (dd, *J* = 8.2, 2.3 Hz, 1H), 7.09 (d, *J* = 2.3 Hz, 1H), 7.09 (dd, *J* = 8.2, 2.3 Hz, 1H), 7.02 (s, 1H), 6.95 (d, *J* = 2.3 Hz, 1H), 6.94 (d, *J* = 8.2 Hz, 1H), 6.90 (d, *J* = 8.2 Hz, 1H), 6.02 – 5.88 (m, 2H), 5.89 – 5.76 (m, 1H), 5.82 (s, 1H), 5.58 (s, 1H), 5.11 – 4.92 (m, 6H), 4.78 (s, 1H), 3.37 – 3.31 (m, 4H), 3.29 – 3.15 (m, 2H).


153.10
151.29
150.98
141.61
137.88
137.52
136.57
133.67
133.65
132.04
131.61
131.02
130.19
129.45
129.07
126.57
123.41
123.04
118.13
116.87
116.63
116.06
115.73
115.41
77.16 CDCl3
39.49
39.45
37.48
OH
OH
OH
CH2
CH2
H2C
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

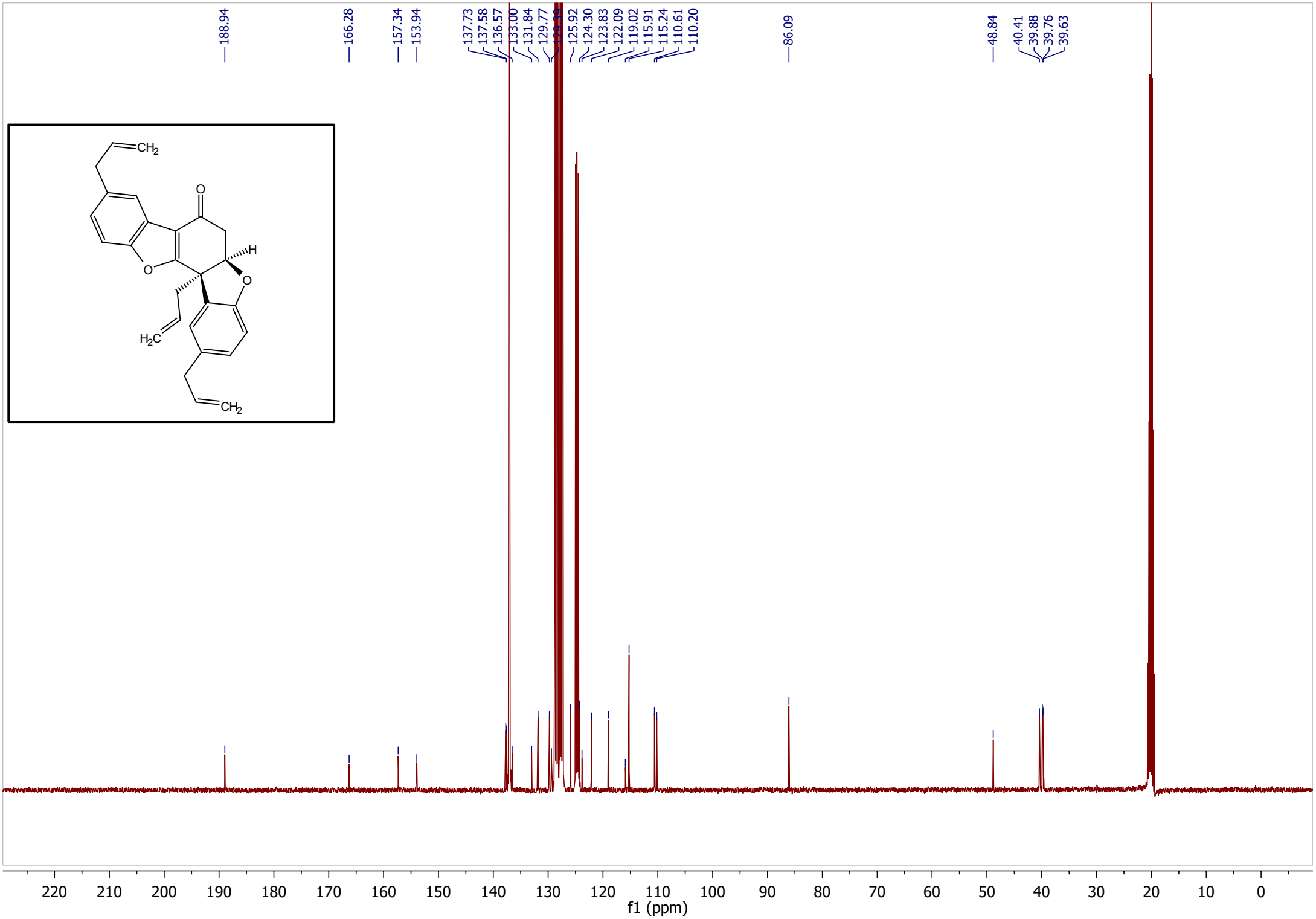


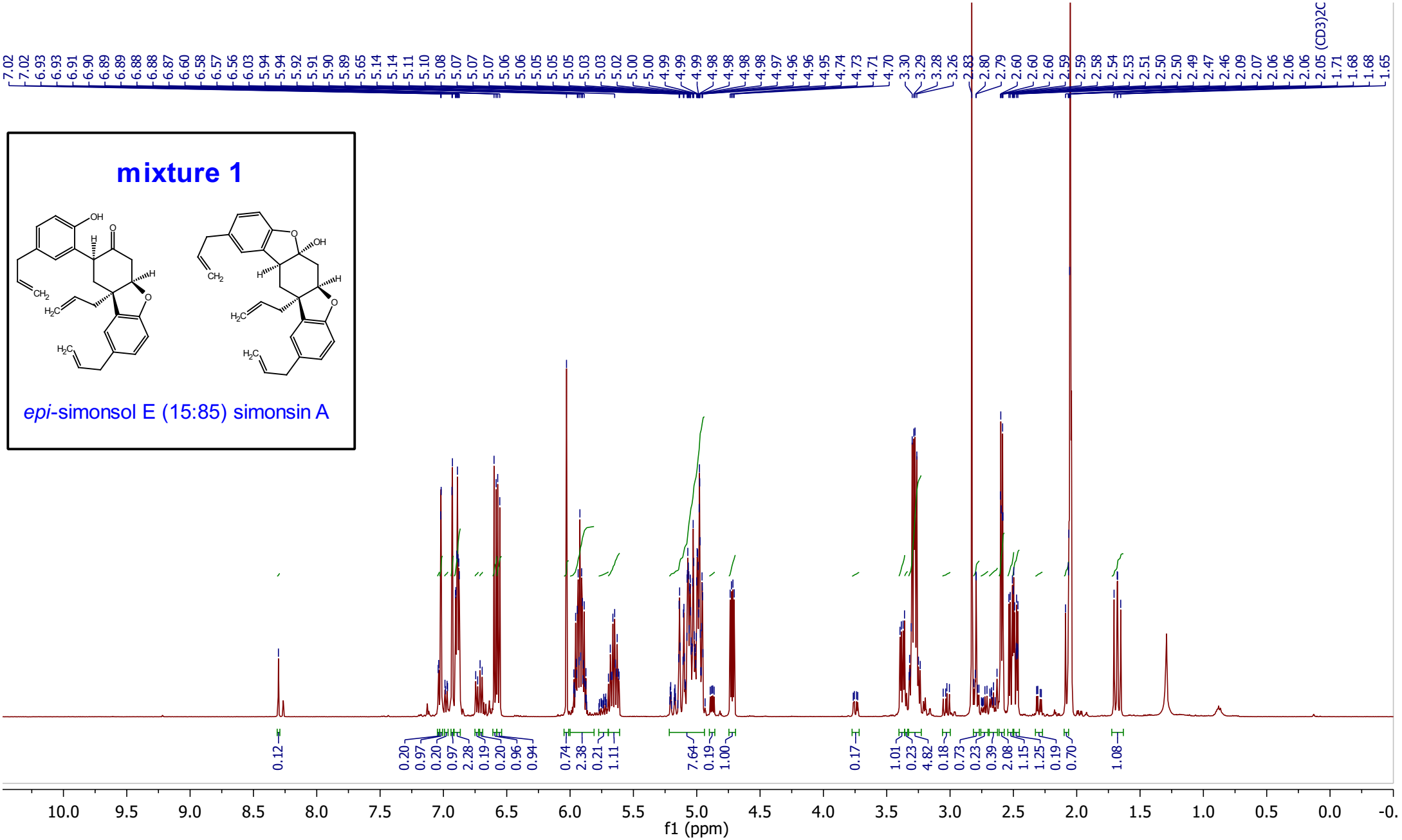
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.89 (d, *J* = 1.8 Hz, 1H), 7.41 (d, *J* = 8.5 Hz, 1H), 7.34 (d, *J* = 1.8 Hz, 1H), 7.15 (dd, *J* = 8.5, 1.8 Hz, 1H), 6.98 (dd, *J* = 8.2, 1.8 Hz, 1H), 6.70 (d, *J* = 8.2 Hz, 1H), 5.96 (dddd, *J* = 16.9, 10.2, 6.7, 6.7 Hz, 2H), 5.64 (dddd, *J* = 16.6, 10.0, 8.2, 6.5, 6.5 Hz, 1H), 5.23 – 5.00 (m, 7H), 3.46 (d, *J* = 6.7 Hz, 2H), 3.38 (d, *J* = 6.7 Hz, 2H), 3.32 (dd, *J* = 14.2, 6.5 Hz, 1H), 3.19 (dd, *J* = 17.7, 2.7 Hz, 1H), 2.96 (dd, *J* = 17.7, 4.4 Hz, 1H), 2.88 (dd, *J* = 14.2, 8.2 Hz, 1H).


190.84
167.25
157.04
154.04
137.78
137.74
136.98
133.55
131.85
129.96
129.16
126.30
124.65
123.51
121.95
120.17
115.96
115.90
111.30
110.28
86.16
77.16 CDCl3
49.17
40.83
40.37
40.22
39.84
CH2
O
H
O
O
H2C
CH2
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

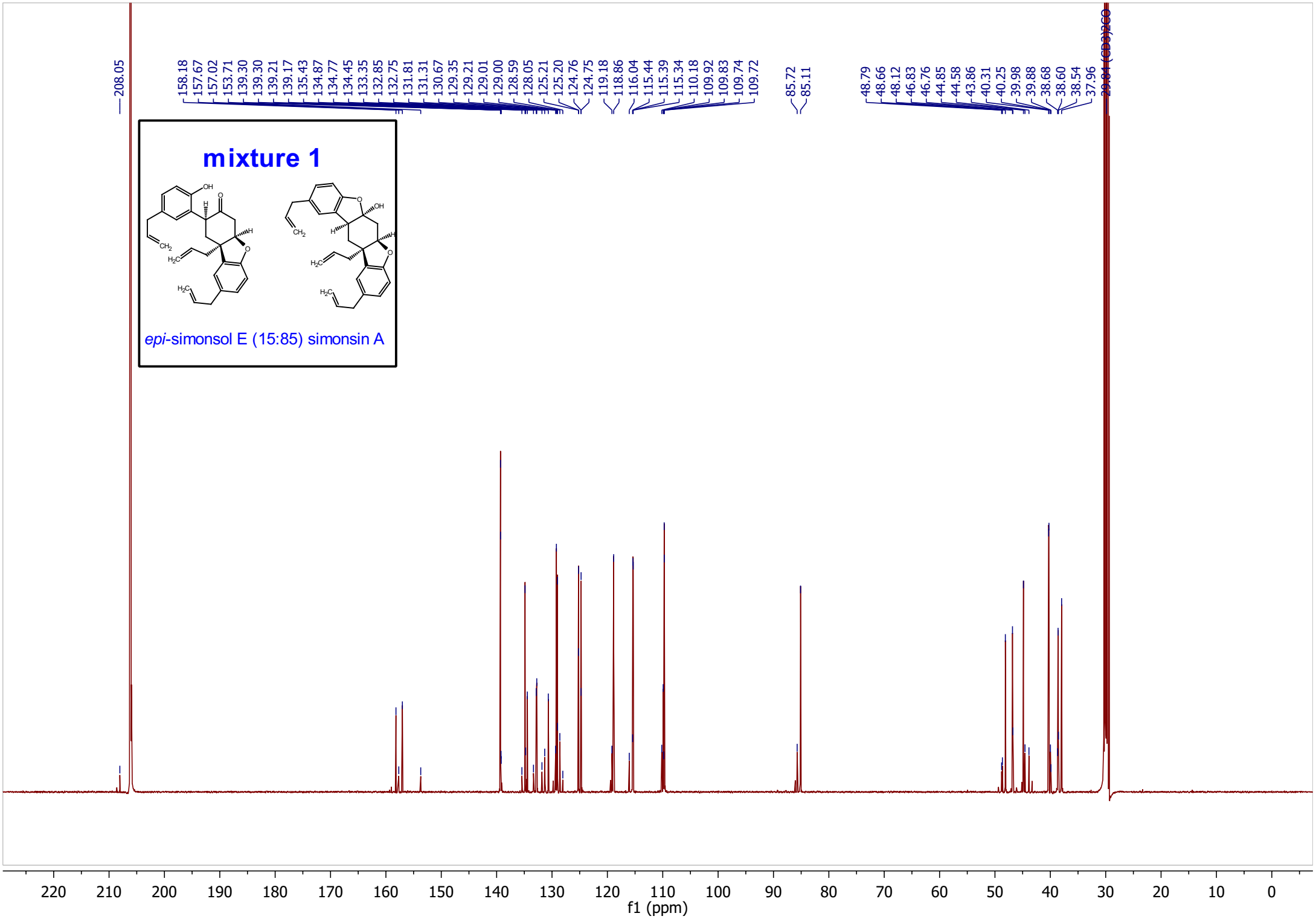


^{1}H NMR (400 MHz, Toluene-d_8) δ 8.10 (d, J = 1.8 Hz, 1H), 7.32 (d, J = 1.8 Hz, 1H), 7.09 (d, J = 8.5 Hz, 1H), 6.80 (dd, J = 8.5, 1.8 Hz, 1H), 6.74 (dd, J = 8.2, 1.8 Hz, 1H), 6.60 (d, J = 8.2 Hz, 1H), 5.93 – 5.73 (m, 2H), 5.27 (dddd, J = 16.8, 10.1, 8.1, 6.6 Hz, 1H), 5.04 – 4.95 (m, 2H), 4.96 – 4.87 (m, 2H), 4.86 – 4.70 (m, 2H), 4.55 (dd, J = 4.3, 2.8 Hz, 1H), 3.17 (d, J = 6.6 Hz, 4H), 3.08 (dd, J = 17.5, 2.8 Hz, 1H), 2.92 (dd, J = 14.1, 6.6 Hz, 1H), 2.57 (dd, J = 17.5, 4.3 Hz, 1H), 2.42 (dd, J = 14.1, 8.1 Hz, 1H).


188.94
166.28
157.34
153.94
137.73
137.58
136.57
133.00
131.84
129.77
125.92
124.30
123.83
122.09
119.02
115.91
115.24
110.61
110.20
86.09
48.84
40.41
39.88
39.76
39.63
CH2
O
H
O
O
H2C
CH2
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

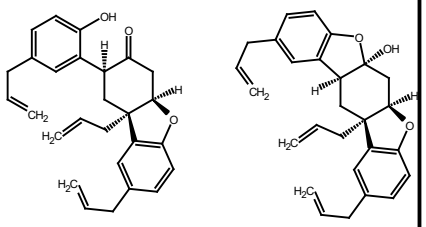


^{1}H NMR (500 MHz, Acetone-d_6) δ 8.30 (s, 0H), 7.04 (d, J = 1.9 Hz, 0H), 7.02 (d, J = 1.9 Hz, 0H), 6.98 (dd, J = 8.1, 1.9 Hz, 0H), 6.93 (d, J = 1.9 Hz, 1H), 6.90 (dd, J = 8.1, 1.9 Hz, 2H), 6.88 (dd, J = 8.1, 1.9 Hz, 1H), 6.74 (d, J = 7.8 Hz, 0H), 6.70 (d, J = 8.2 Hz, 0H), 6.59 (d, J = 8.2 Hz, 1H), 6.56 (d, J = 8.1 Hz, 1H), 6.03 (s, 1H), 5.92 (dddd, J = 16.8, 10.0, 6.6, 6.6 Hz, 0H), 5.92 (dddd, J = 16.8, 10.0, 6.6, 6.6 Hz, 2H), 5.74 (dddd, 0H), 5.66 (dddd, J = 17.3, 10.1, 7.2, 7.2 Hz, 1H), 5.22 – 4.94 (m, 7H), 4.88 (dd, J = 9.9, 5.4 Hz, 0H), 4.72 (dd, J = 11.1, 5.1 Hz, 1H), 3.75 (dd, J = 13.3, 4.4 Hz, 0H), 3.38 (dd, J = 12.4, 5.4 Hz, 1H), 3.32 (ddd, J = 6.6, 1.5 Hz, 0H), 3.29 (d, J = 6.7 Hz, 2H), 3.27 (d, J = 7.0 Hz, 2H), 3.24 (d, J = 7.1 Hz, 0H), 3.03 (dd, J = 14.7, 10.0 Hz, 0H), 2.79 (d, J = 1.2 Hz, 0H), 2.79 (dd, J = 14.5, 5.4 Hz, 0H), 2.73 (dddd, J = 12.7, 6.8, 2.3, 2.3 Hz, 0H), 2.67 (dd, J = 12.7, 8.3 Hz, 0H), 2.64 (d, J = 13.7 Hz, 0H), 2.59 (ddd, J = 7.2, 1.2, 1.2 Hz, 2H), 2.52 (dd, J = 13.9, 5.3 Hz, 1H), 2.48 (dd, J = 13.1, 5.3 Hz, 1H), 2.30 (dd, J = 13.9, 4.5 Hz, 0H), 2.07 (dd, J = 13.9, 11.1 Hz, 1H), 1.68 (dd, J = 13.9, 12.4 Hz, 1H).


208.05
158.18
157.67
157.02
153.71
139.30
139.30
139.21
139.17
135.43
134.87
134.77
134.45
133.35
132.85
132.75
131.81
131.31
130.67
129.35
129.21
129.01
129.00
128.59
128.05
125.21
125.20
124.76
124.75
119.18
118.86
116.04
115.44
115.39
115.34
110.18
109.92
109.83
109.74
109.72
85.72
85.11
48.79
48.66
48.12
46.83
46.76
44.85
44.58
43.86
40.31
40.25
39.98
39.88
38.68
38.60
38.54
37.96
29.84 (CD3)2CO
mixture 1
epi-simonsol E (15:85) simonsin A
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

^{13}C DEPT 135

mixture 1

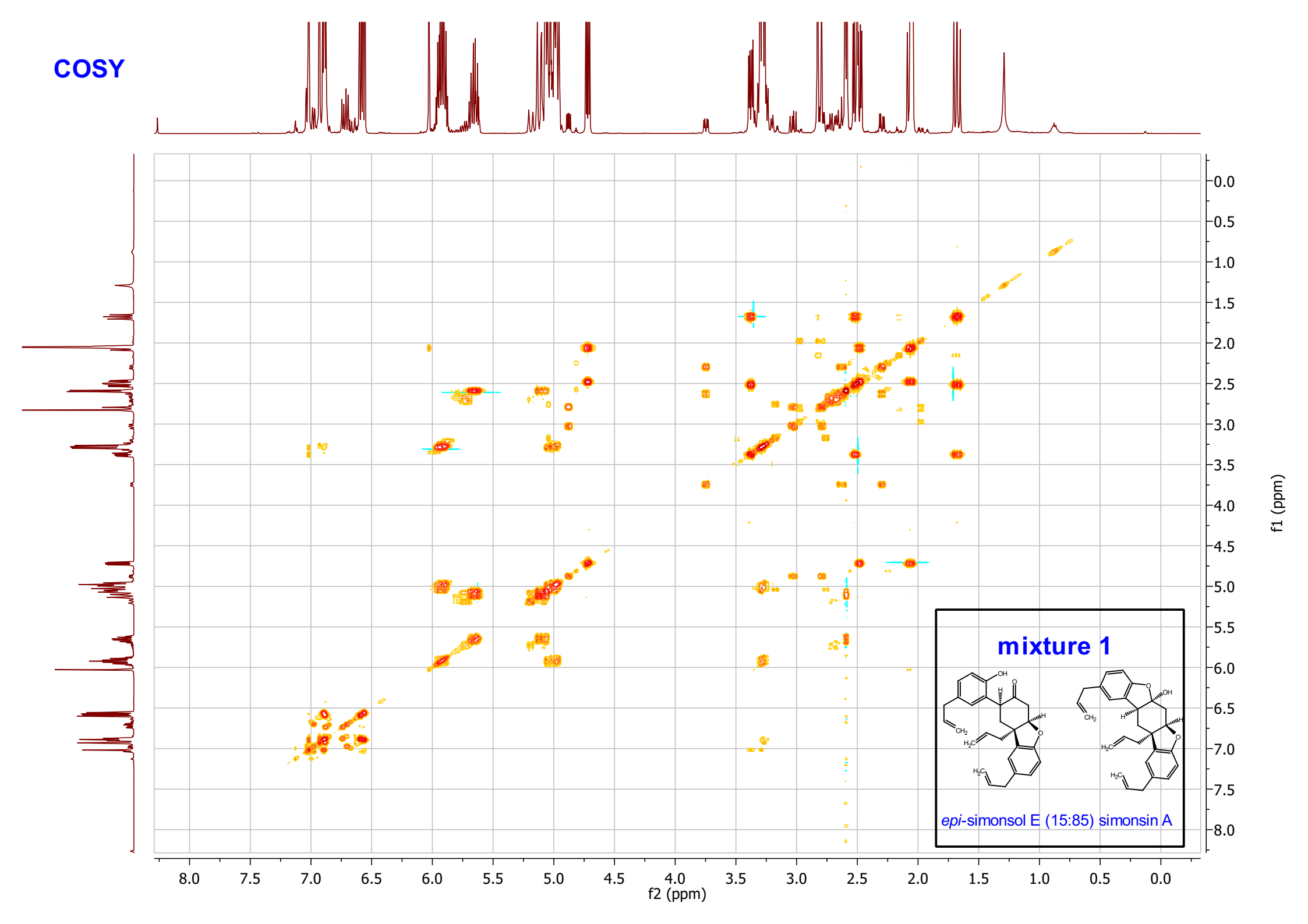


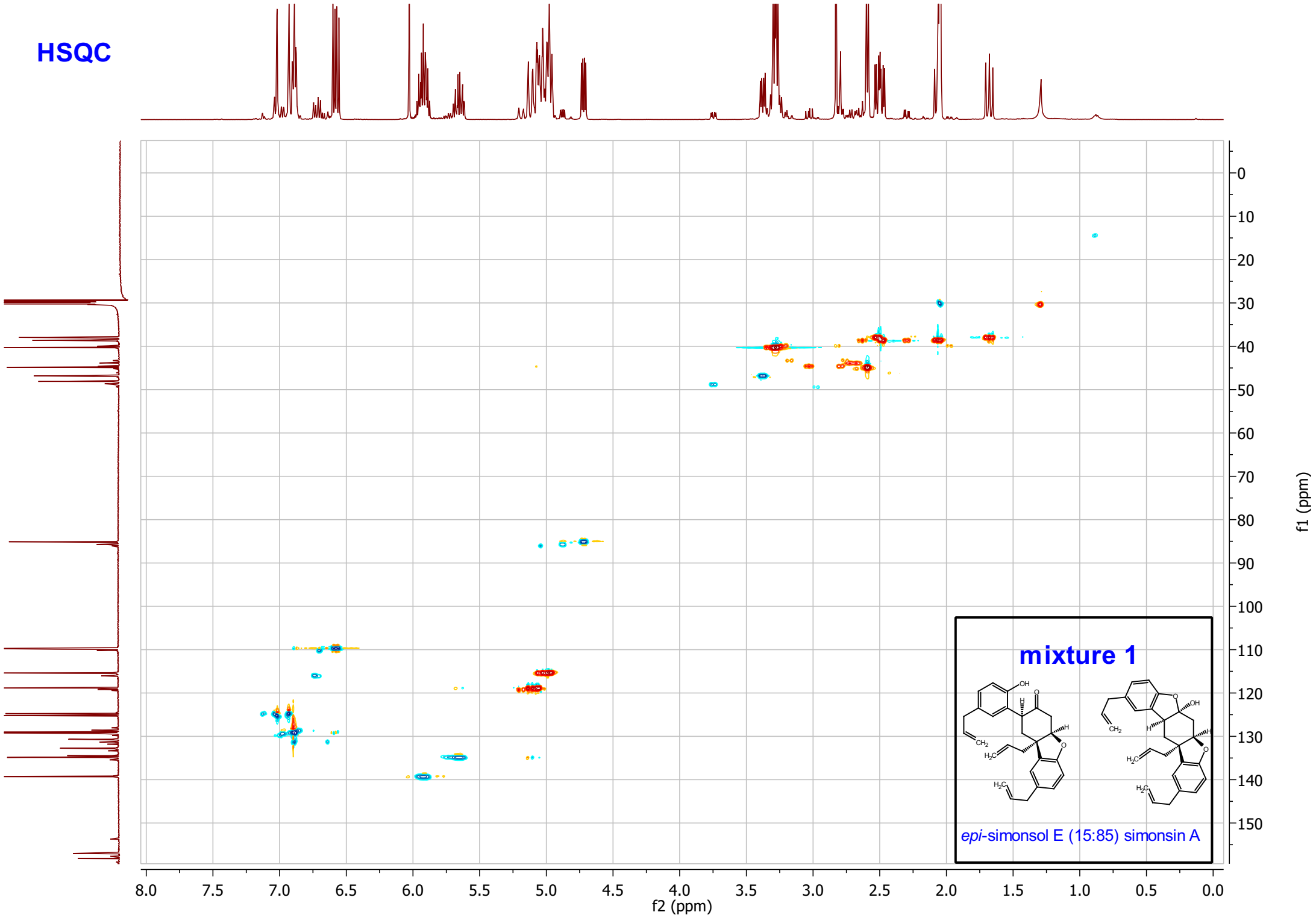
epi-simonsol E (15:85) simonsin A

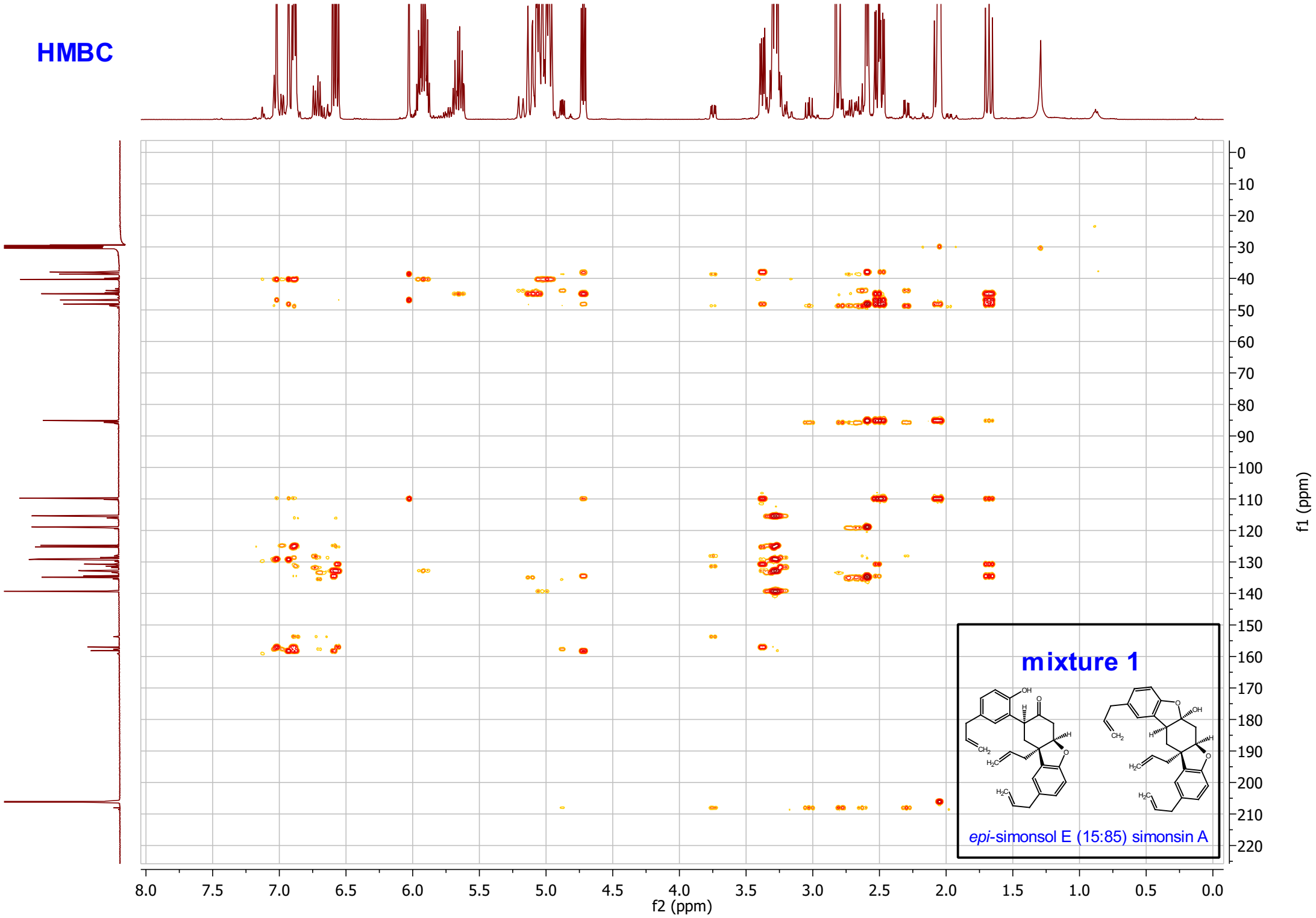
139.30
139.21
139.17
134.87
134.77
131.32
129.35
129.21
129.01
128.59
125.21
124.75
119.18
118.86
116.04
115.44
115.39
115.35
115.34
110.18
109.74
109.72
85.72
85.11 (CD3)2CO
48.80
46.83
46.76
44.85
44.58
43.86
40.31
40.25
39.98
39.88
38.68
38.60
38.54
37.96

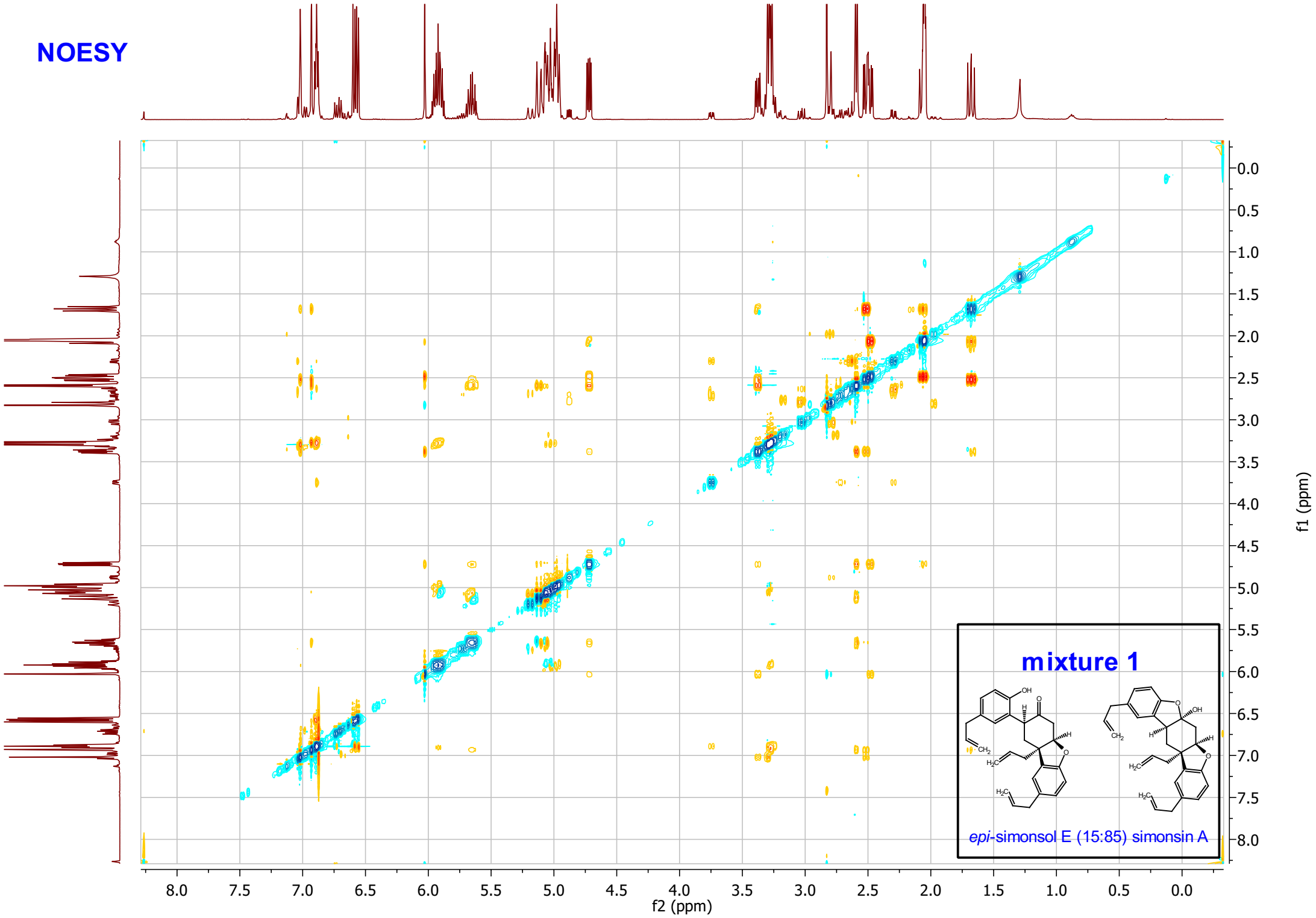
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

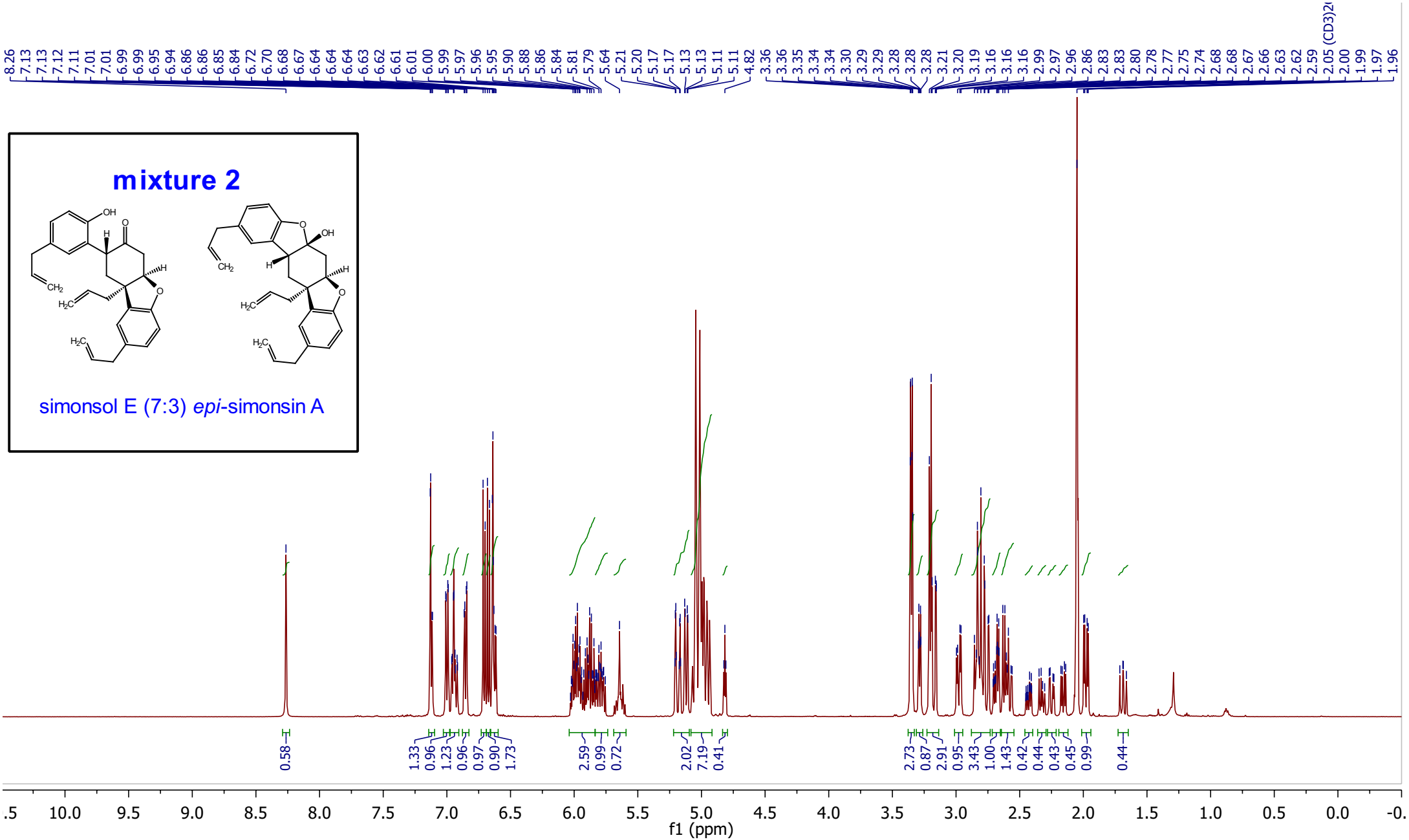
f1 (ppm)


COSY
mixture 1
epi-simonsol E (15:85) simonsin A
f2 (ppm)
f1 (ppm)

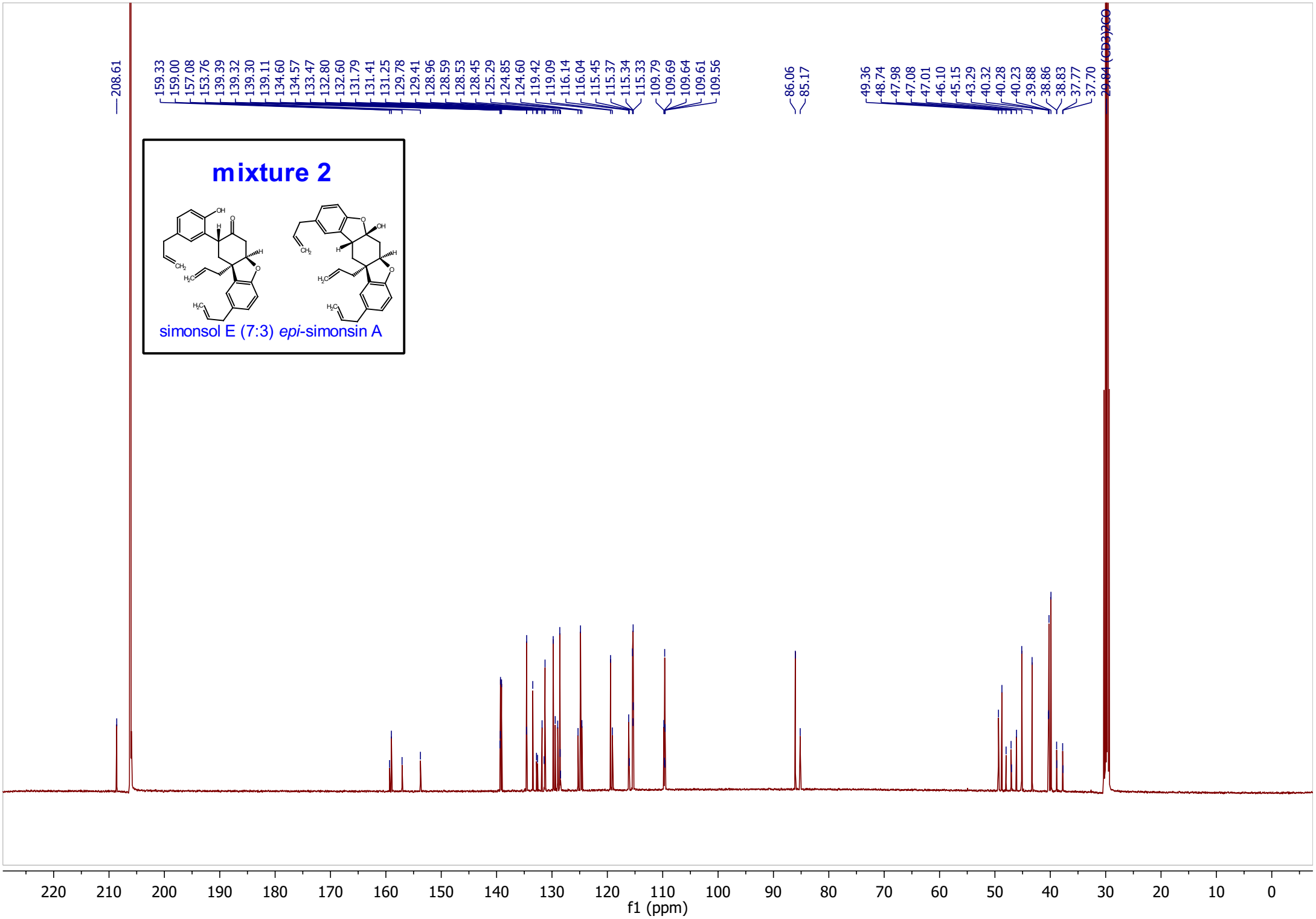

HSQC
mixture 1
epi-simonsol E (15:85) simonsin A
f2 (ppm)
f1 (ppm)
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150


HMBC
mixture 1
epi-simonsol E (15:85) simonsin A
f2 (ppm)
f1 (ppm)


NOESY
mixture 1
epi-simonsol E (15:85) simonsin A
f2 (ppm)
f1 (ppm)
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

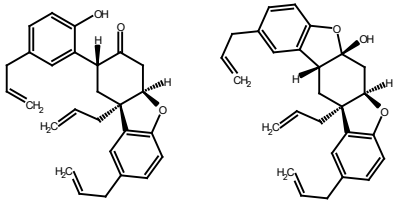


^{1}H NMR (500 MHz, Acetone-d_6) δ 8.26 (s, 1H), 7.13 (d, J = 1.9 Hz, 1H), 7.11 (d, J = 1.9 Hz, 0H), 7.00 (dd, J = 8.2, 1.9 Hz, 1H), 6.96 (d, J = 1.9 Hz, 0H), 6.95 (d, J = 1.8 Hz, 0H), 6.93 (dd, J = 8.2, 1.8 Hz, 1H), 6.85 (dd, J = 8.2, 2.2 Hz, 1H), 6.71 (d, J = 8.2 Hz, 1H), 6.67 (d, J = 8.2 Hz, 1H), 6.64 (d, J = 2.2 Hz, 1H), 6.63 (d, J = 8.2 Hz, 0H), 6.62 (d, J = 8.2 Hz, 0H), 5.98 (dddd, J = 16.8, 10.0, 6.6, 6.6 Hz, 1H), 5.88 (dddd, J = 16.8, 10.0, 6.6, 6.6 Hz, 1H), 5.80 (dddd, J = 16.8, 10.2, 8.2, 6.5 Hz, 1H), 5.64 (s, 0H), 5.22 – 5.09 (m, 2H), 4.82 (dd, J = 4.2, 3.6 Hz, 0H), 3.35 (ddd, J = 6.6, 1.6, 1.6 Hz, 3H), 3.29 (ddd, J = 6.8, 1.6, 1.6 Hz, 1H), 3.20 (d, J = 6.6 Hz, 2H), 3.17 (d, J = 16.1 Hz, 0H), 3.16 (d, J = 3.3 Hz, 1H), 2.98 (dd, J = 13.5, 4.2 Hz, 1H), 2.85 (d, J = 7.8 Hz, 1H), 2.85 – 2.77 (m, 2H), 2.76 (dd, J = 16.1, 3.0 Hz, 2H), 2.68 (dddd, J = 13.8, 6.5, 1.4 Hz, 1H), 2.61 (dd, J = 13.8, 8.2 Hz, 1H), 2.56 (d, J = 4.2 Hz, 0H), 2.43 (ddd, J = 13.8, 6.5, 1.4, 1.4 Hz, 0H), 2.33 (dd, J = 13.8, 8.2 Hz, 0H), 2.25 (dd, J = 14.8, 3.6 Hz, 0H), 2.16 (dd, J = 13.9, 5.0 Hz, 0H), 1.98 (dd, J = 13.6, 4.2 Hz, 1H), 1.69 (dd, J = 13.9, 11.9 Hz, 0H).


208.61
159.33
159.00
157.08
153.76
139.39
139.32
139.30
139.11
134.60
134.57
133.47
132.80
132.60
131.79
131.41
131.25
129.78
129.41
128.96
128.59
128.53
128.45
125.29
124.85
124.60
119.42
119.09
116.14
116.04
115.45
115.37
115.34
115.33
109.79
109.69
109.64
109.61
109.56
86.06
85.17
49.36
48.74
47.98
47.08
47.01
46.10
45.15
43.29
40.32
40.28
40.23
39.88
38.86
38.83
37.77
37.70
29.84 (CD3)2CO
mixture 2
simonsol E (7:3) epi-simonsin A
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

^{13}C DEPT 135

mixture 2



simonsol E (7:3) *epi*-simonsin A

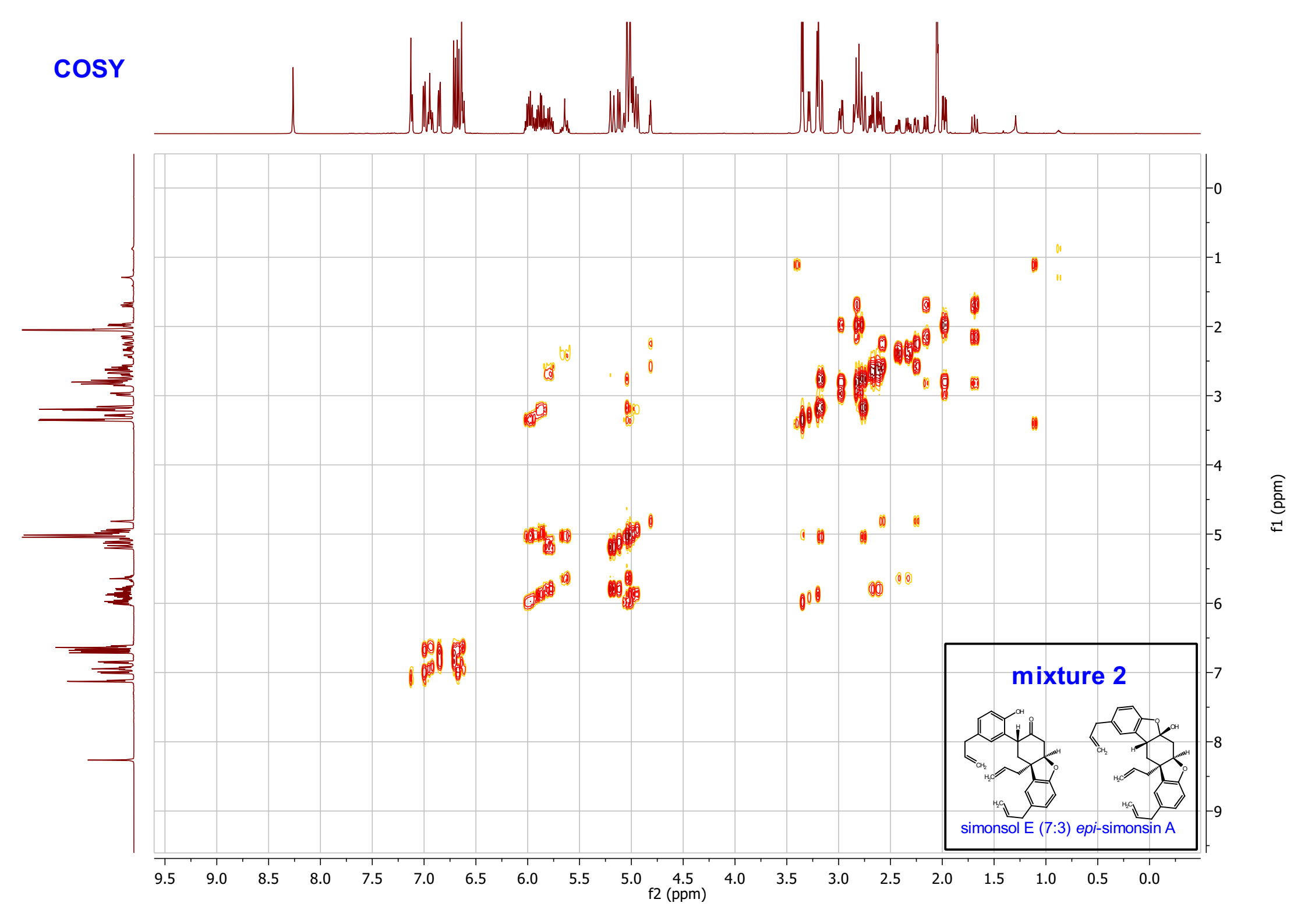
139.39
139.32
139.30
139.11
134.60
134.57
131.25
129.78
128.96
128.59
125.29
124.85
124.60
119.42
119.10
116.14
116.04
115.45
115.37
115.34
109.79
109.64
109.56

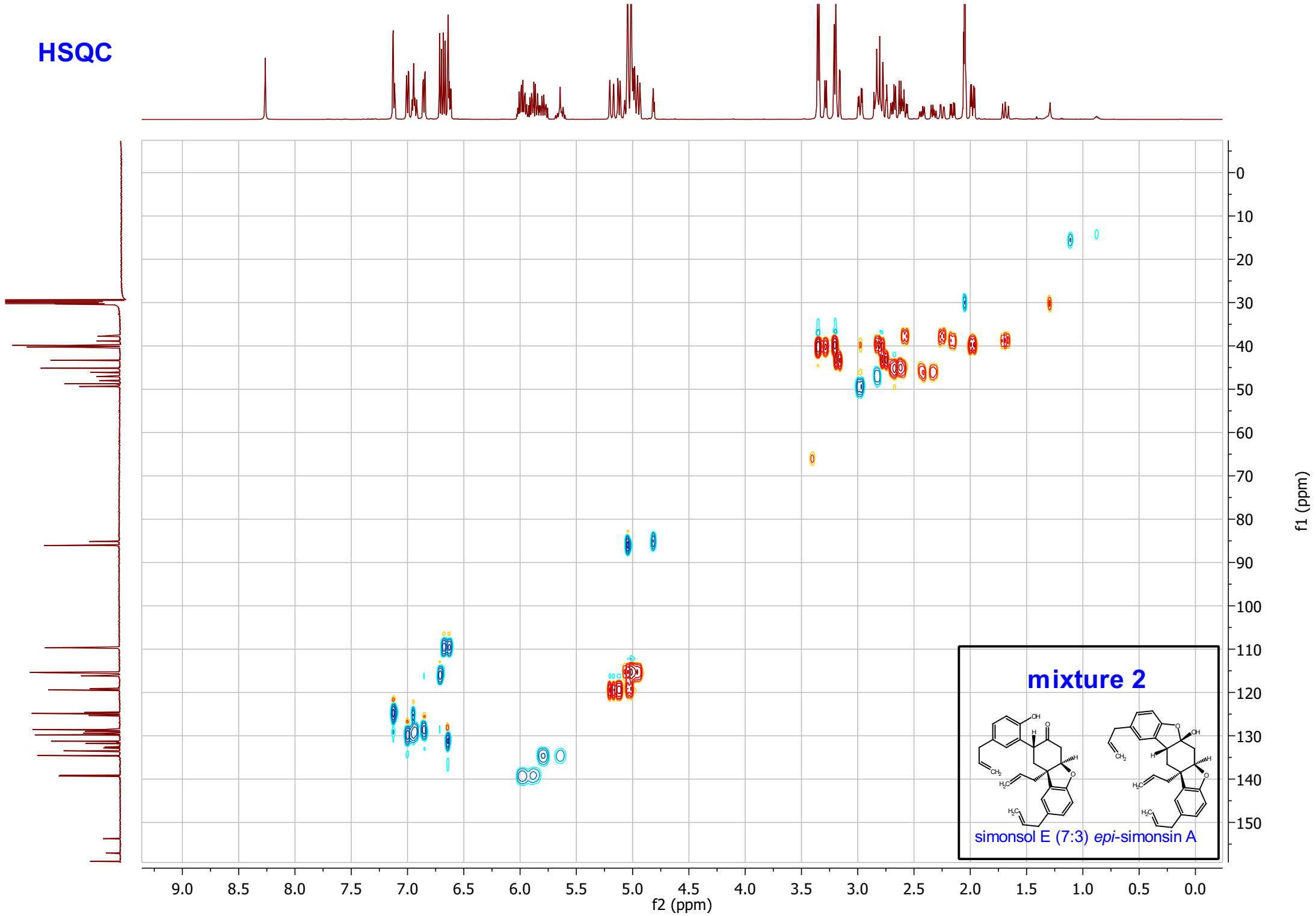
86.06 (CD3)2CO
85.17

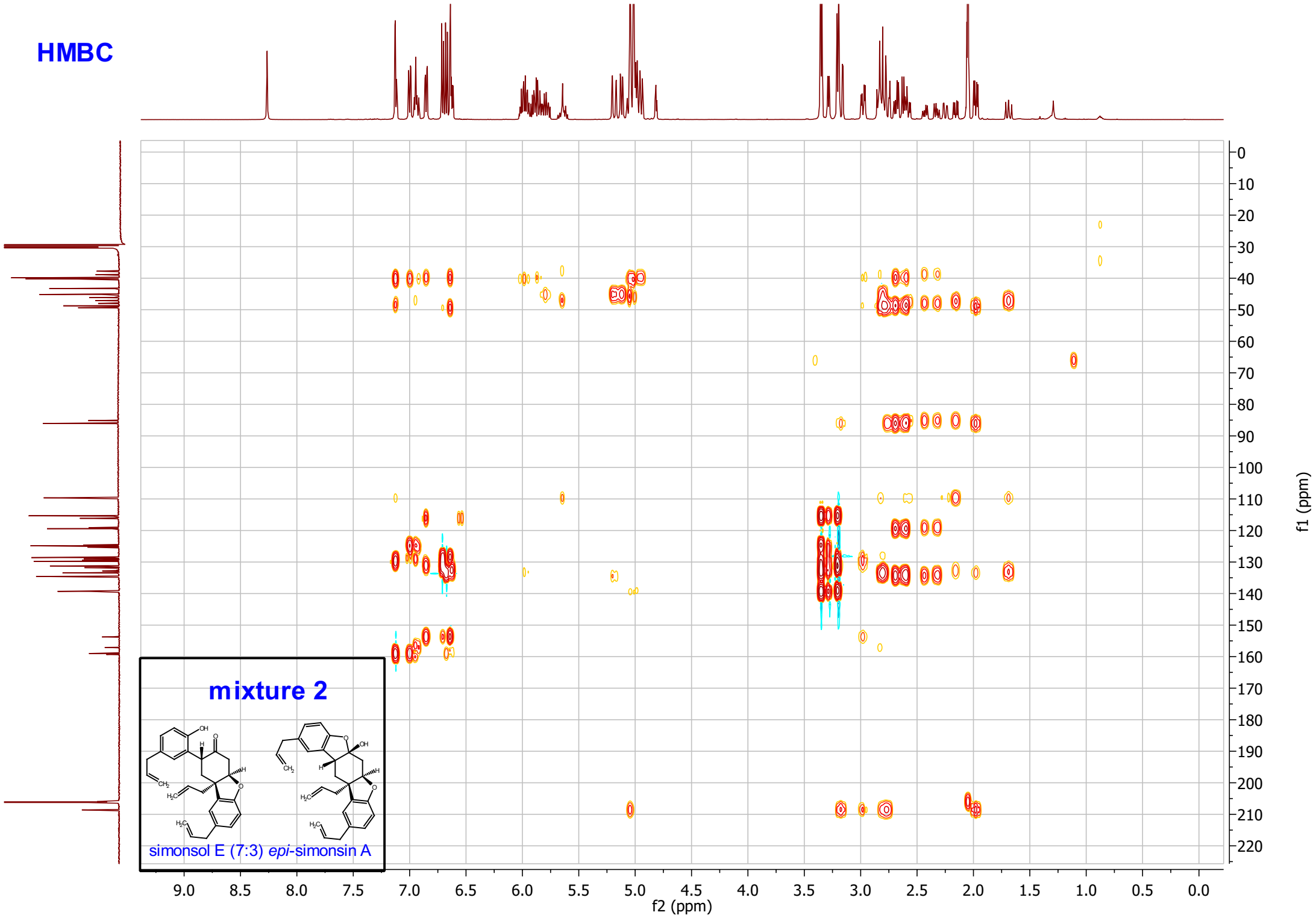
49.36
47.08
47.01
46.10
45.15
43.29
40.32
40.28
40.23
39.87
38.86
37.77

220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

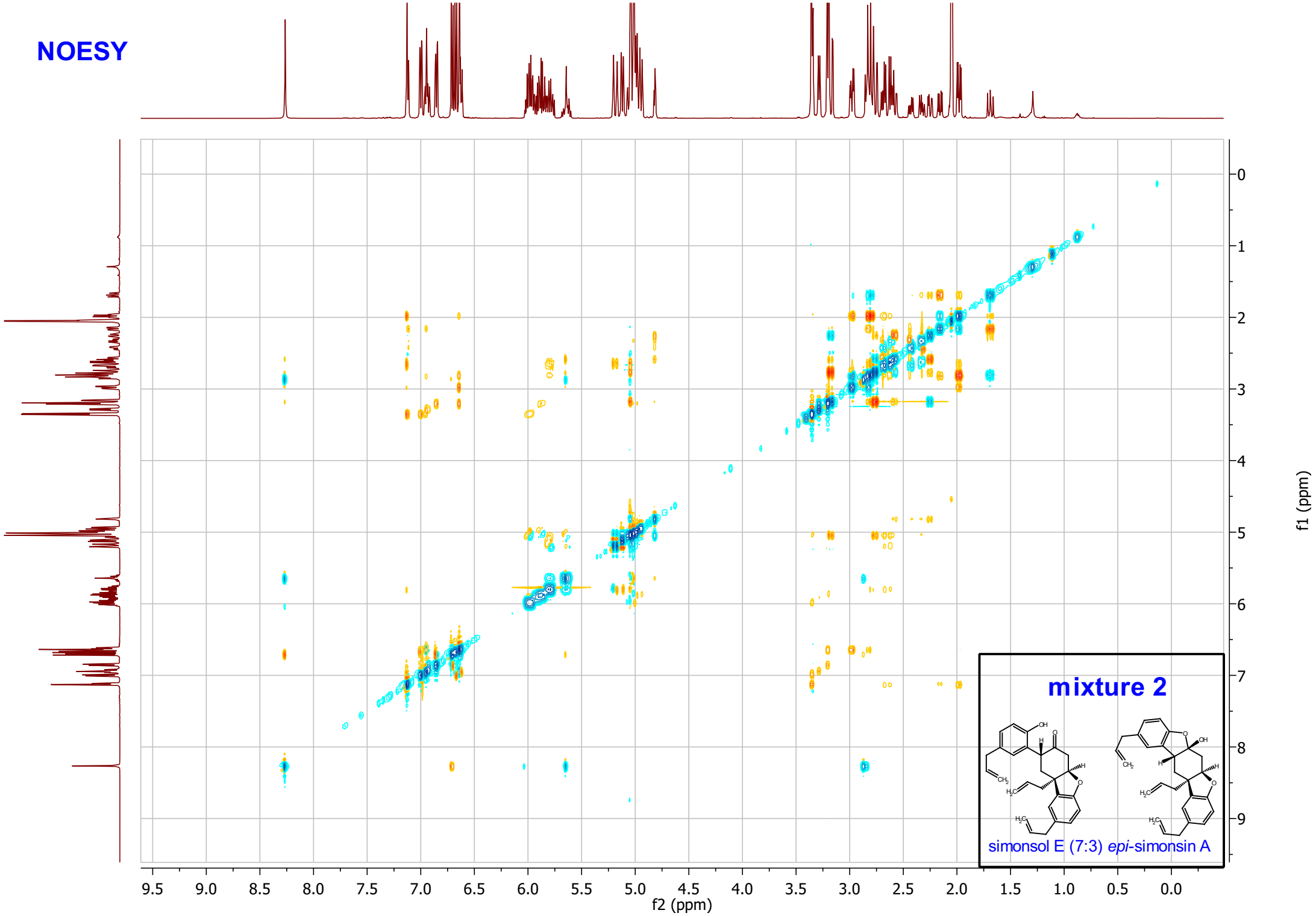
f1 (ppm)

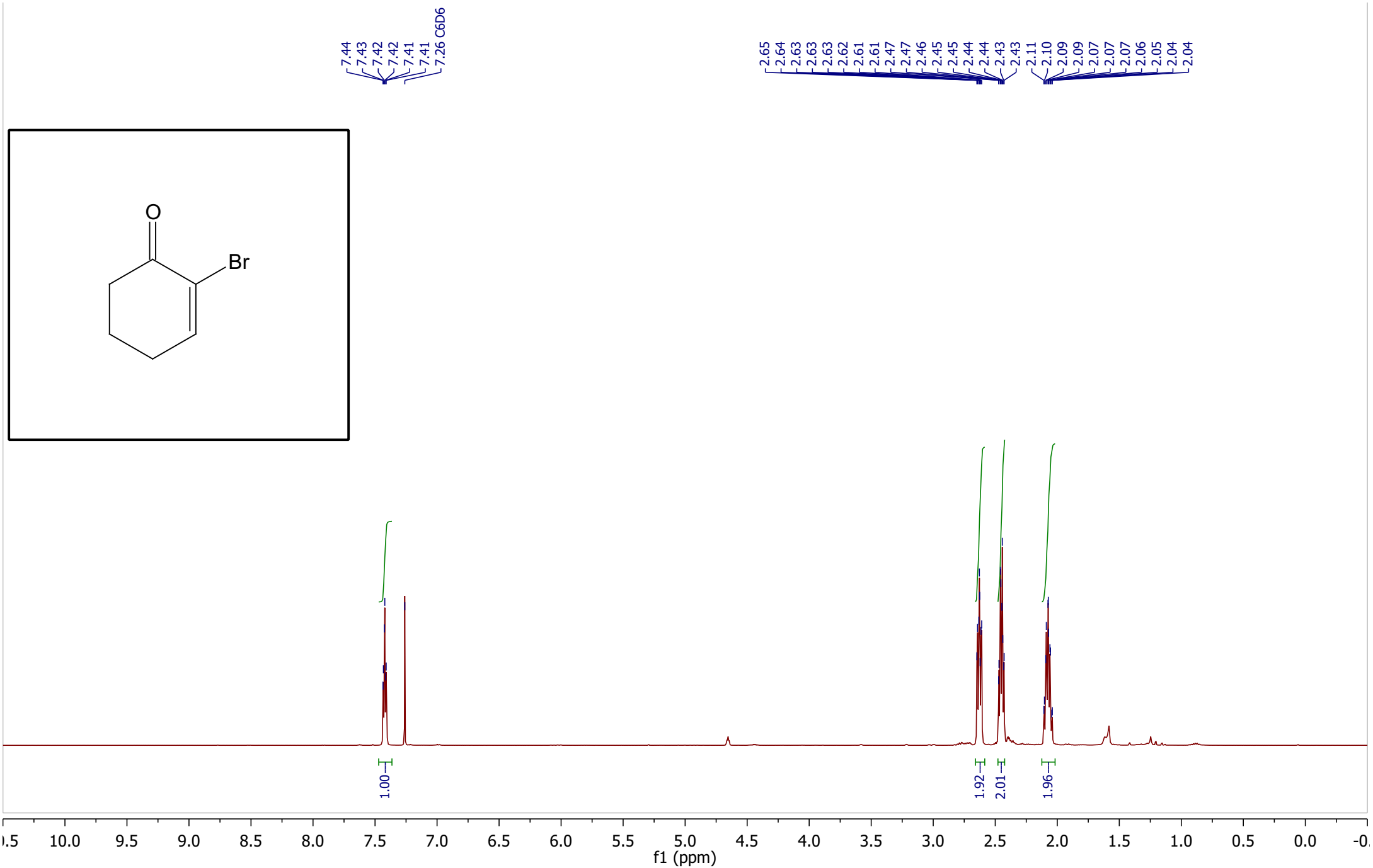

COSY
mixture 2
simonsol E (7:3) epi-simonsin A
f2 (ppm)
f1 (ppm)


HSQC
mixture 2
simonsol E (7:3) epi-simonsin A
f2 (ppm)
f1 (ppm)

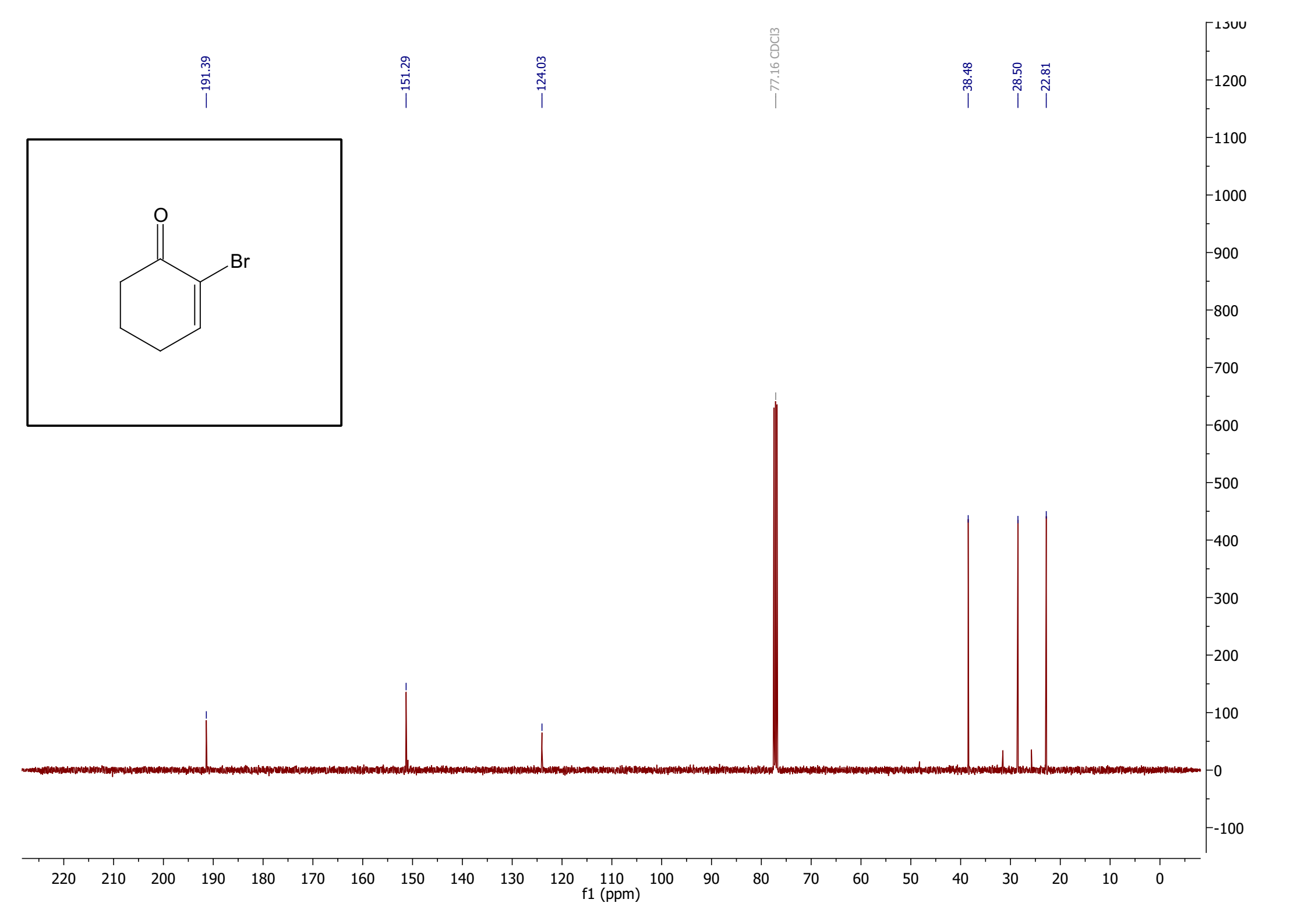

HMBC
mixture 2
simonsol E (7:3) epi-simonsin A
f2 (ppm)
f1 (ppm)

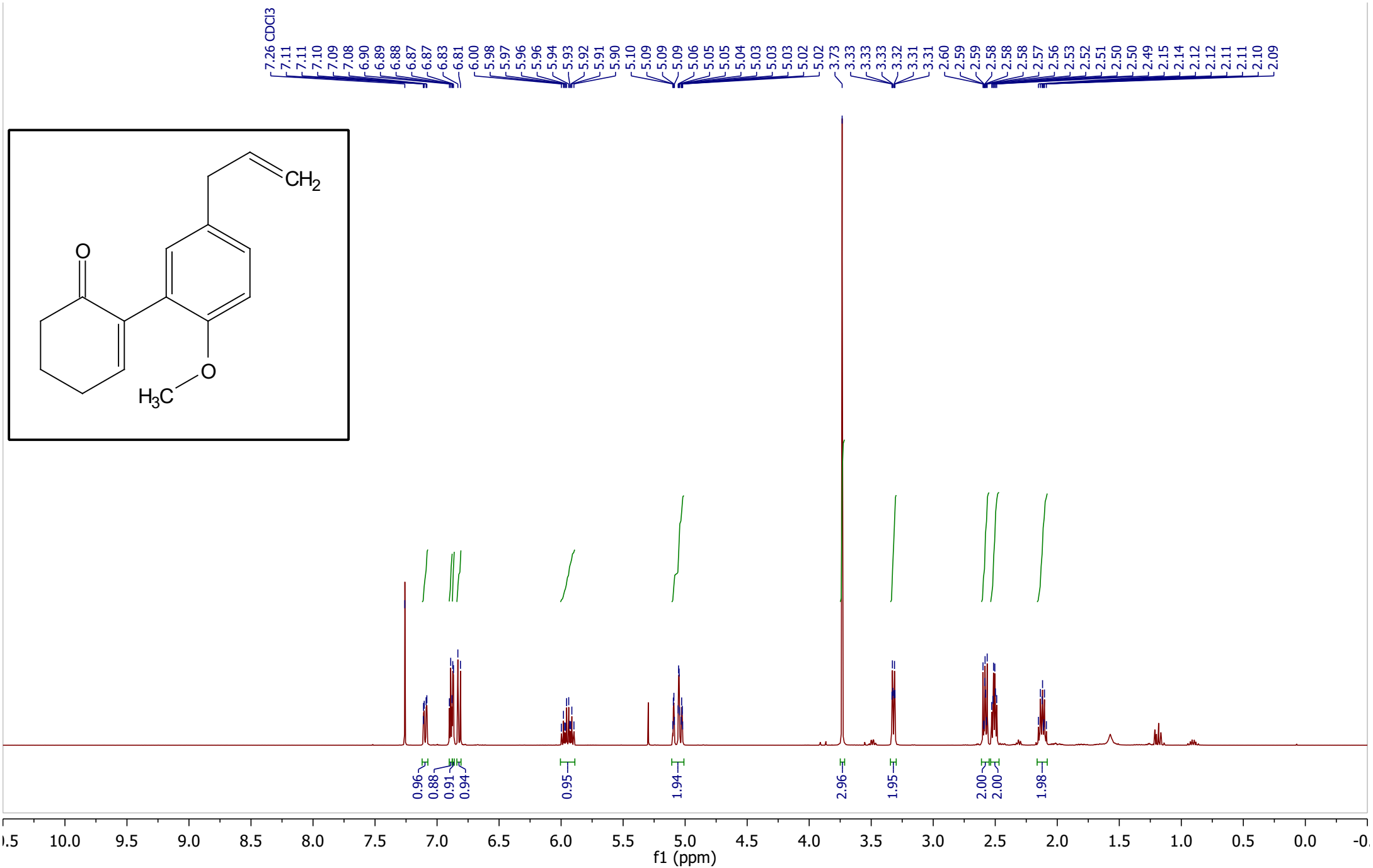
NOESY



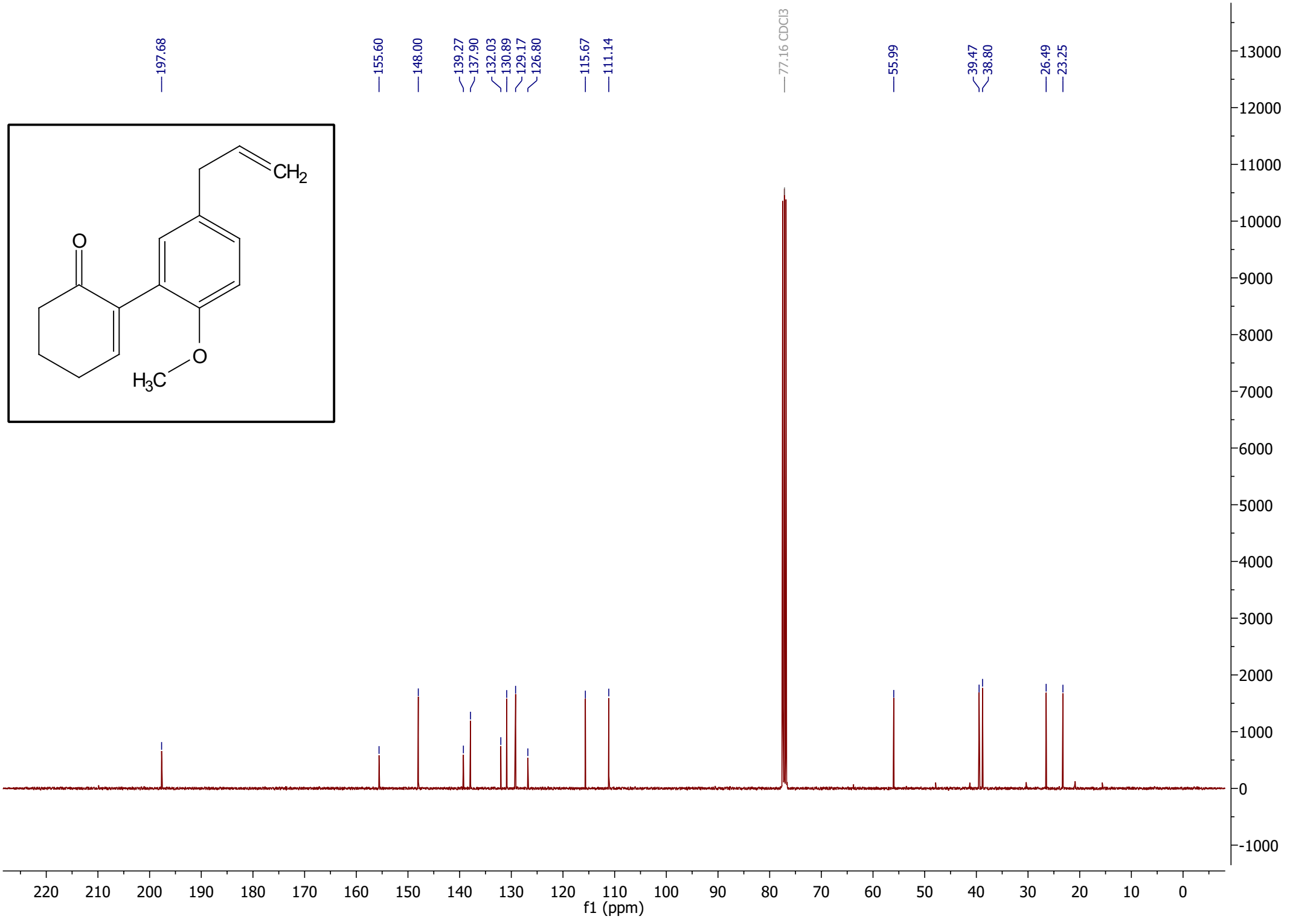


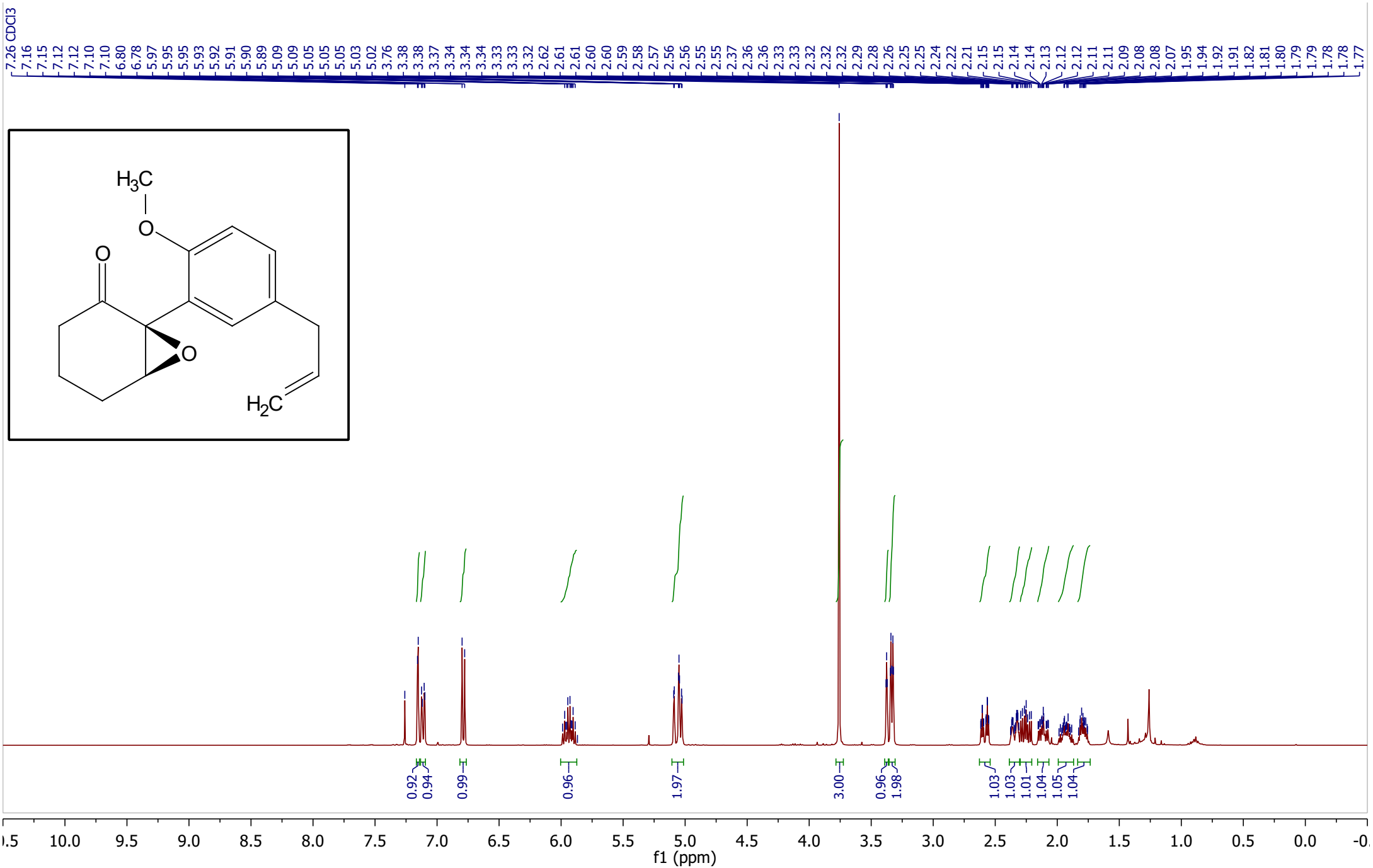
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.42 (td, *J* = 4.5, 1.4 Hz, 1H), 2.65 – 2.60 (m, 2H), 2.47 – 2.41 (m, 2H), 2.12 – 2.04 (m, 2H).


191.39
151.29
124.03
77.16 CDCl3
38.48
28.50
22.81
O
Br
f1 (ppm)

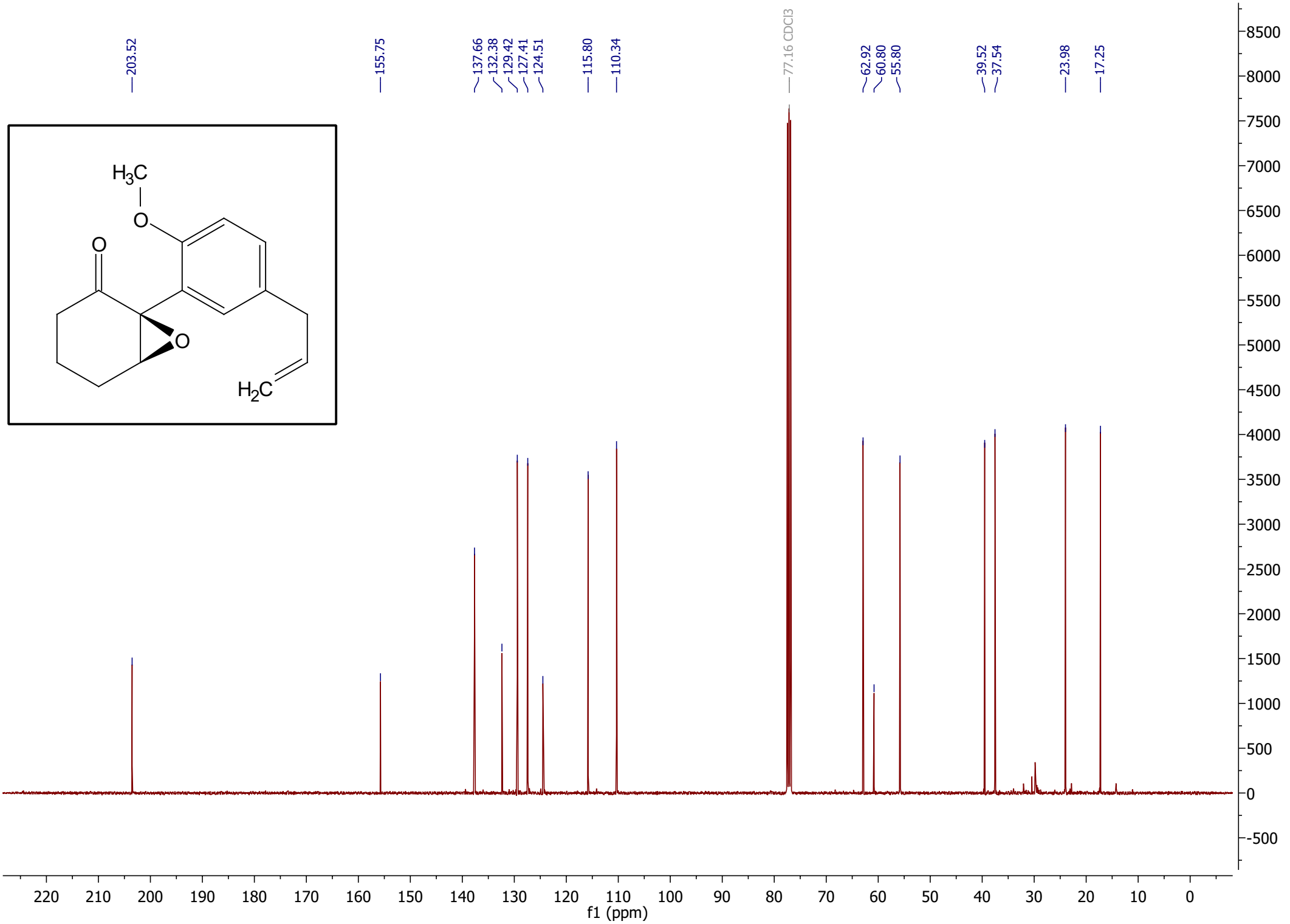


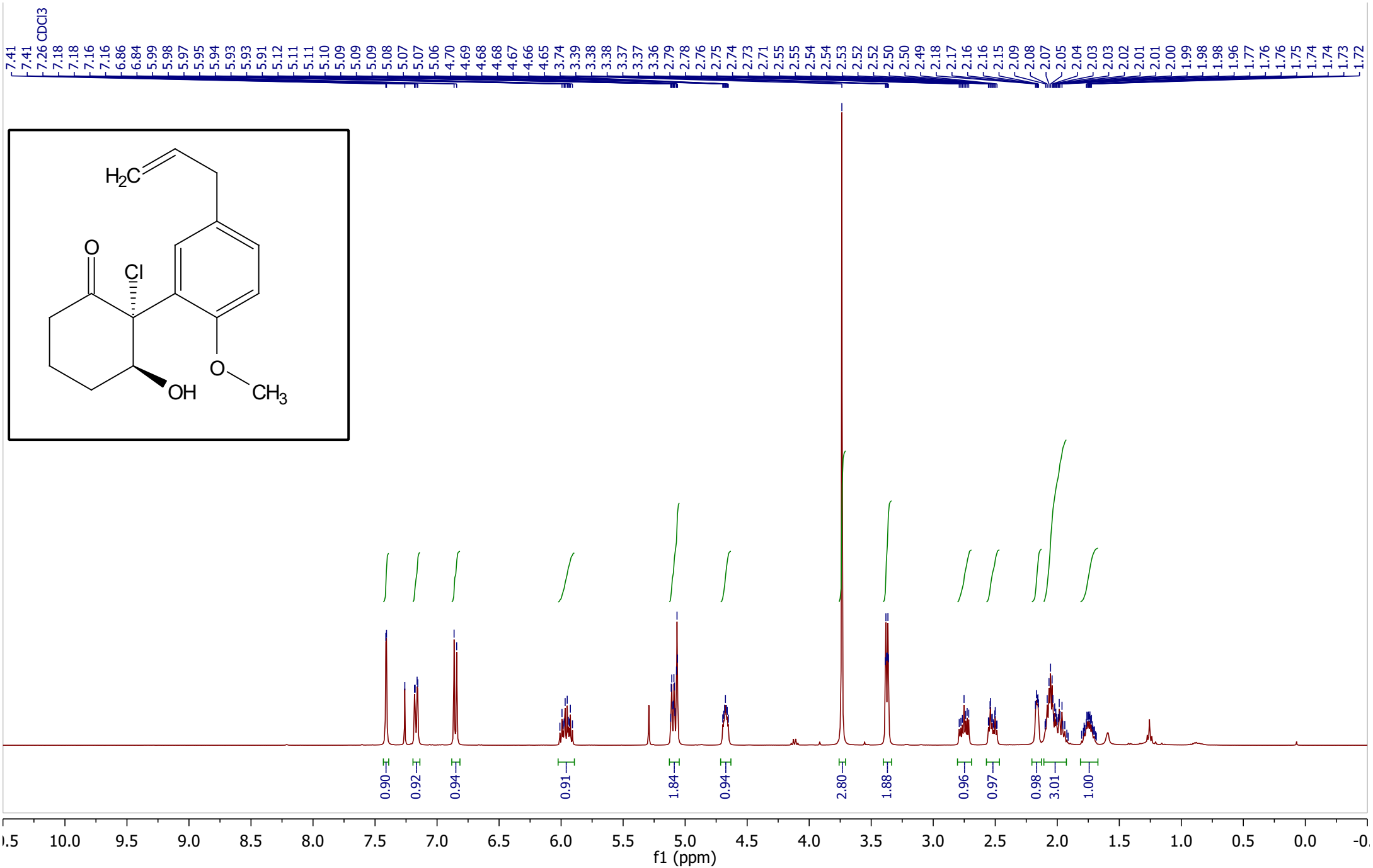
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.10 (dd, *J* = 8.4, 2.3 Hz, 1H), 6.89 (t, *J* = 4.2 Hz, 1H), 6.87 (d, *J* = 2.3 Hz, 1H), 6.82 (d, *J* = 8.4 Hz, 1H), 5.95 (ddt, *J* = 16.8, 10.0, 6.7 Hz, 1H), 5.10 – 5.01 (m, 2H), 3.73 (s, 3H), 3.32 (ddd, *J* = 6.8, 1.4, 1.4 Hz, 2H), 2.61 – 2.56 (m, 2H), 2.51 (td, *J* = 6.0, 4.2 Hz, 2H), 2.16 – 2.08 (m, 2H).


197.68
155.60
148.00
139.27
137.90
132.03
130.89
129.17
126.80
115.67
111.14
77.16 CDCl3
55.99
39.47
38.80
26.49
23.25
CH2
O
O
H3C
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

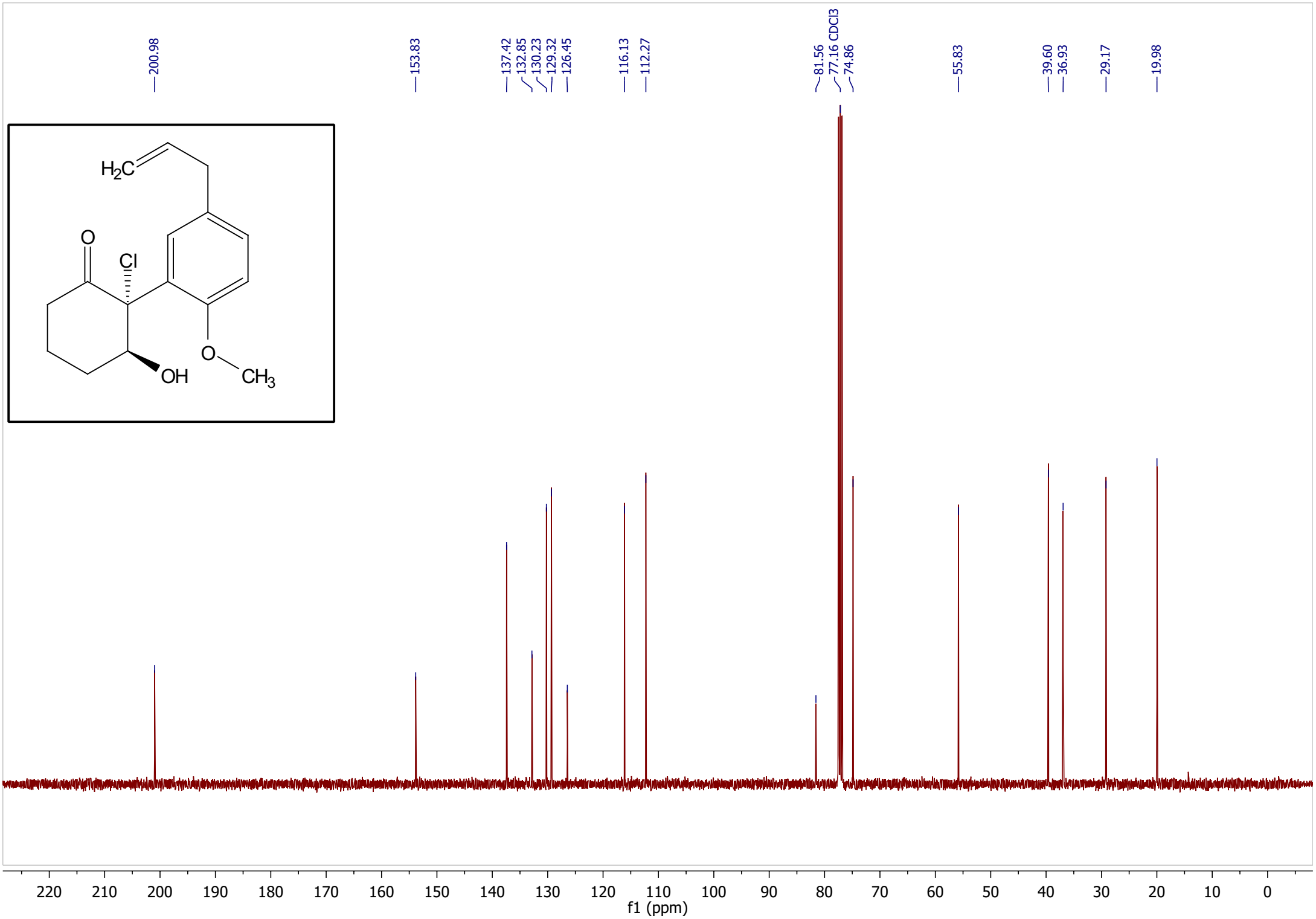


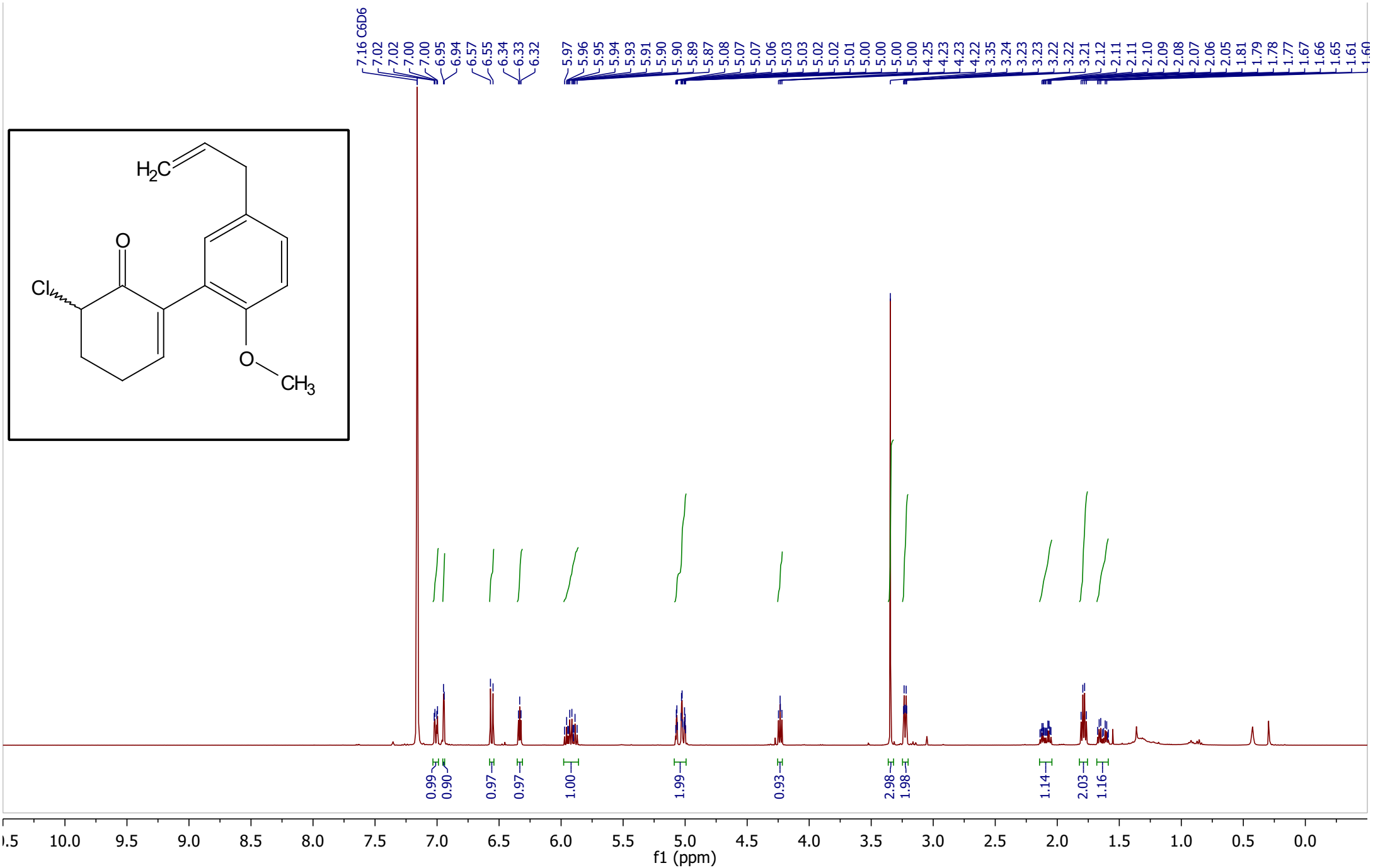
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.15 (d, *J* = 2.3 Hz, 1H), 7.11 (dd, *J* = 8.3, 2.3 Hz, 1H), 6.79 (d, *J* = 8.3 Hz, 1H), 5.94 (dddd, *J* = 16.8, 10.0, 6.8, 6.8 Hz, 1H), 5.12 – 5.01 (m, 2H), 3.76 (s, 3H), 3.38 (dd, *J* = 2.0, 2.0 Hz, 1H), 3.33 (ddd, *J* = 6.7, 1.6, 1.6 Hz, 2H), 2.58 (dddd, *J* = 16.5, 4.3, 4.3, 1.0 Hz, 1H), 2.38 – 2.31 (m, 1H), 2.25 (ddd, *J* = 16.4, 12.1, 5.6 Hz, 1H), 2.16 – 2.06 (m, 1H), 1.99 – 1.87 (m, 1H), 1.83 – 1.75 (m, 1H).


203.52
155.75
137.66
132.38
129.42
127.41
124.51
115.80
110.34
77.16 CDCl3
62.92
60.80
55.80
39.52
37.54
23.98
17.25
H3C
O
O
O
H2C
f1 (ppm)

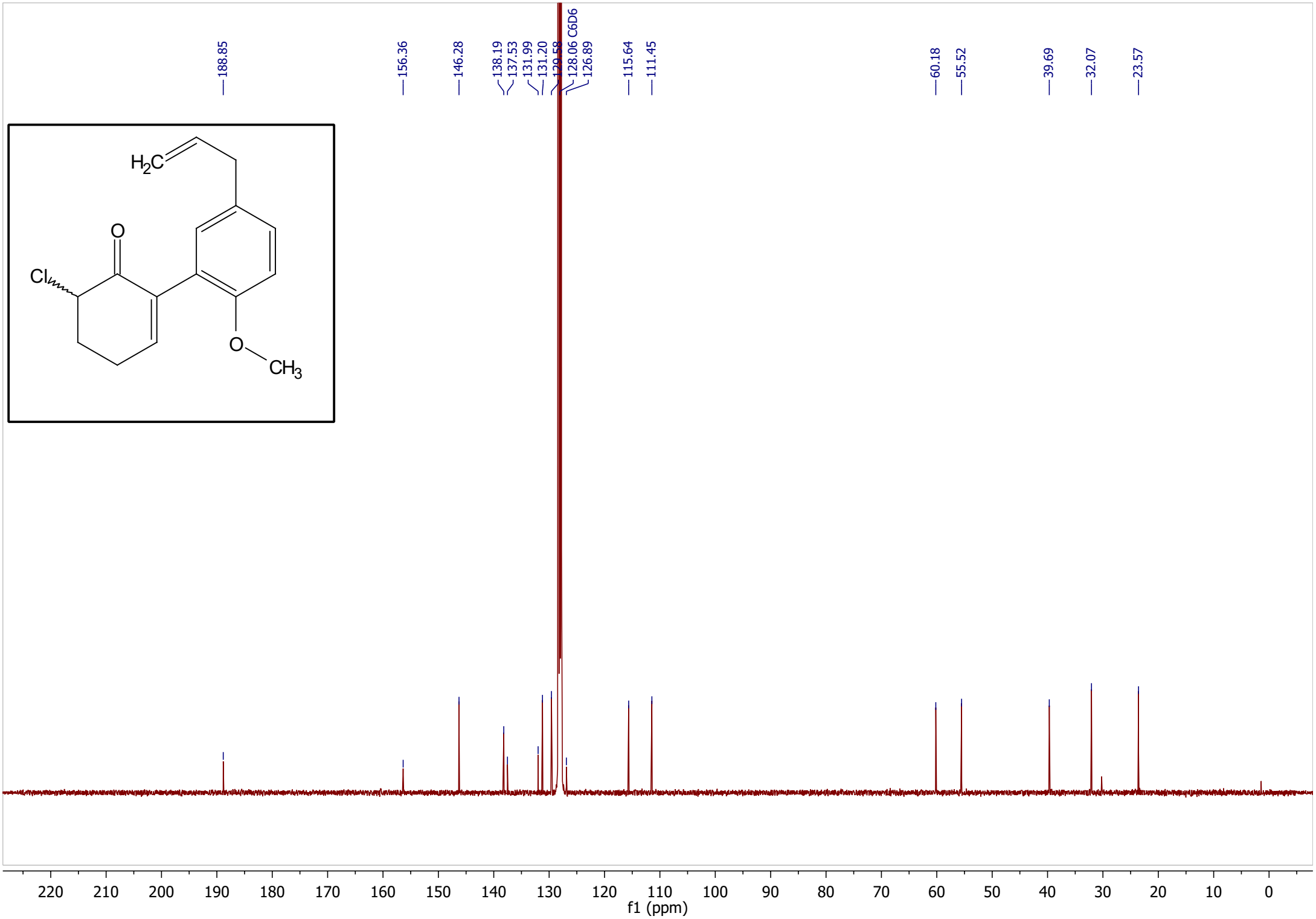


^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.41 (d, *J* = 2.2 Hz, 1H), 7.17 (dd, *J* = 8.4, 2.2 Hz, 1H), 6.85 (d, *J* = 8.4 Hz, 1H), 5.96 (dddd, *J* = 16.9, 10.3, 6.8, 6.8 Hz, 1H), 5.13 – 5.04 (m, 2H), 4.67 (ddd, *J* = 9.3, 6.1, 3.3 Hz, 1H), 3.74 (s, 3H), 3.37 (ddd, *J* = 6.6, 1.7, 1.7 Hz, 2H), 2.75 (ddd, *J* = 15.2, 10.1, 5.2 Hz, 1H), 2.52 (dddd, *J* = 15.2, 5.6, 5.6, 1.5 Hz, 1H), 2.20 – 2.12 (m, 1H), 2.11 – 1.91 (m, 3H), 1.82 – 1.68 (m, 1H).

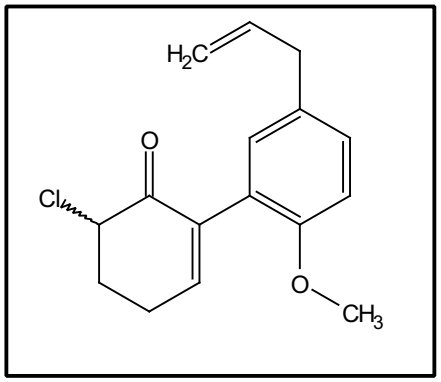

200.98
153.83
137.42
132.85
130.23
129.32
126.45
116.13
112.27
81.56
77.16 CDCl3
74.86
55.83
39.60
36.93
29.17
19.98
H2C
O
Cl
OH
O
CH3
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



^{1}H NMR (400 MHz, Benzene-d_6) δ 7.01 (dd, J = 8.3, 2.3 Hz, 1H), 6.95 (d, J = 2.3 Hz, 1H), 6.56 (d, J = 8.3 Hz, 1H), 6.33 (dd, J = 4.1, 4.1 Hz, 1H), 5.92 (dddd, J = 16.8, 10.0, 6.6, 6.6 Hz, 1H), 5.09 – 4.98 (m, 2H), 4.27 – 4.20 (m, 1H), 3.35 (s, 3H), 3.23 (ddd, J = 6.6, 1.5, 1.5 Hz, 2H), 2.09 (dddd, J = 19.2, 6.2, 6.2, 3.7 Hz, 1H), 1.82 – 1.75 (m, 2H), 1.63 (dddd, J = 19.2, 5.1, 5.1, 4.5 Hz, 1H).


188.85
156.36
146.28
138.19
137.53
131.99
131.20
129.58
128.06 C6D6
126.89
115.64
111.45
60.18
55.52
39.69
32.07
23.57
H2C
O
Cl
O
CH3
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

^{13}C DEPT

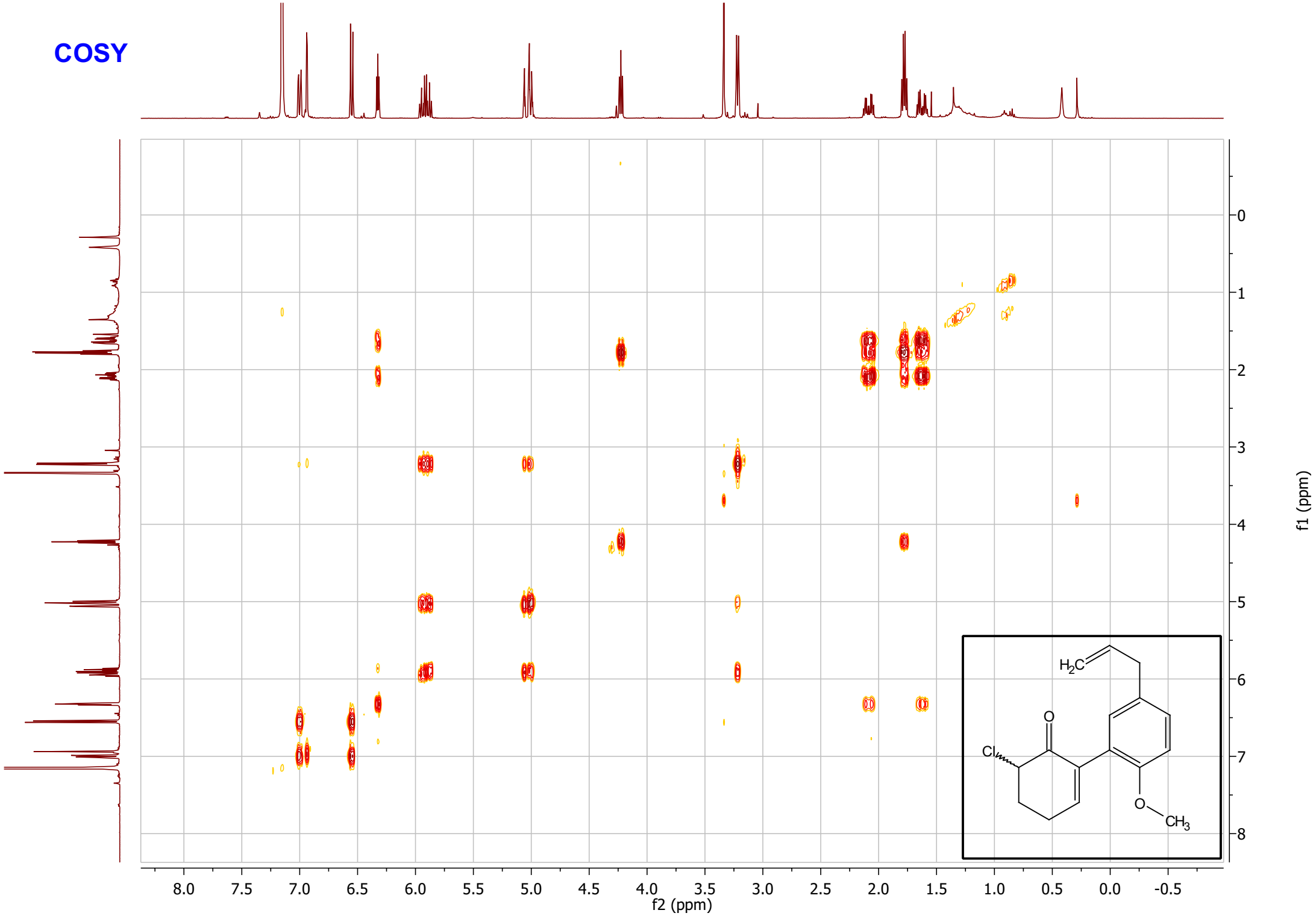


146.28 C6D6
138.19
131.21
129.59
128.35
115.64
111.45
60.18
55.52
39.69
32.07
23.57

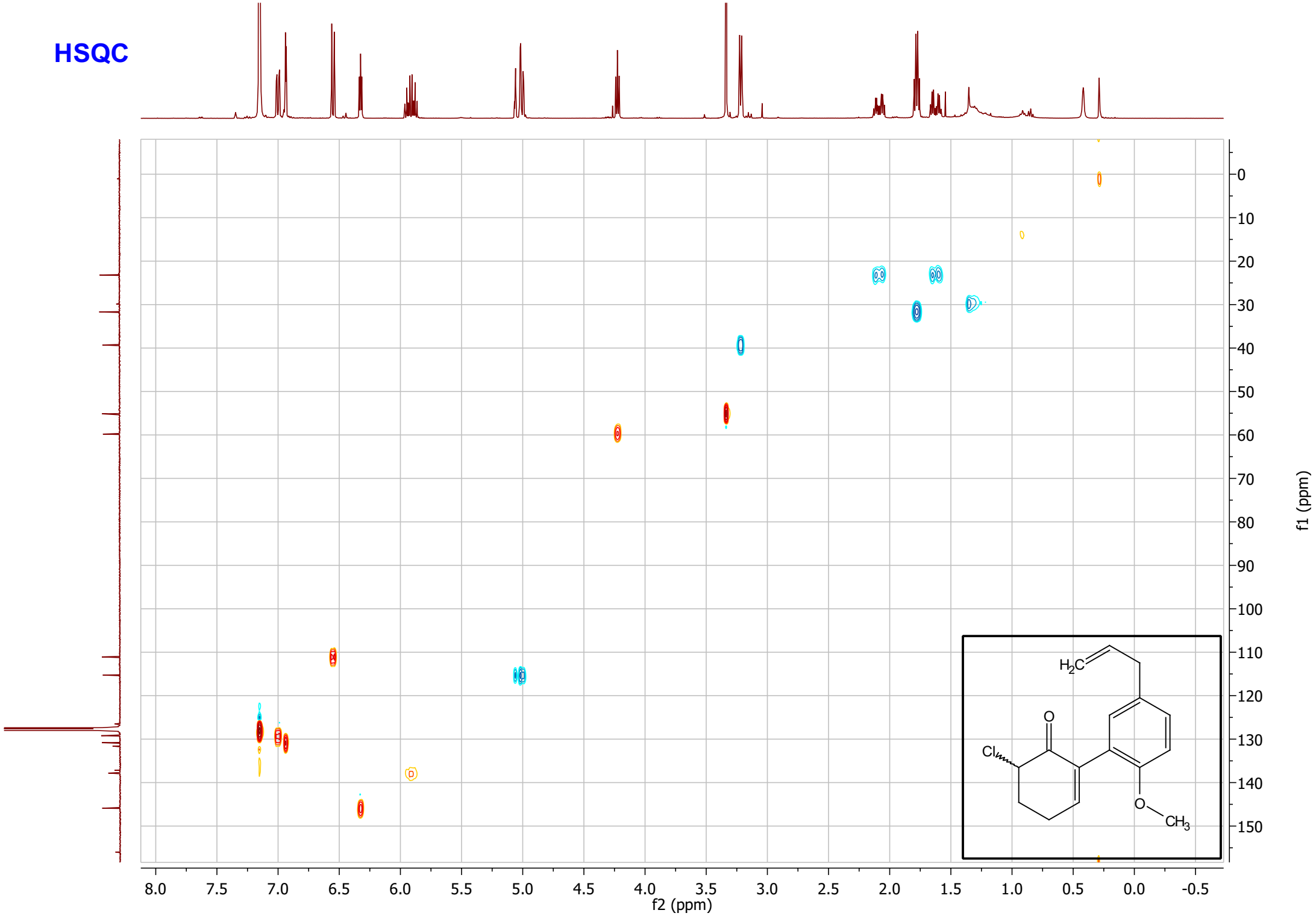
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

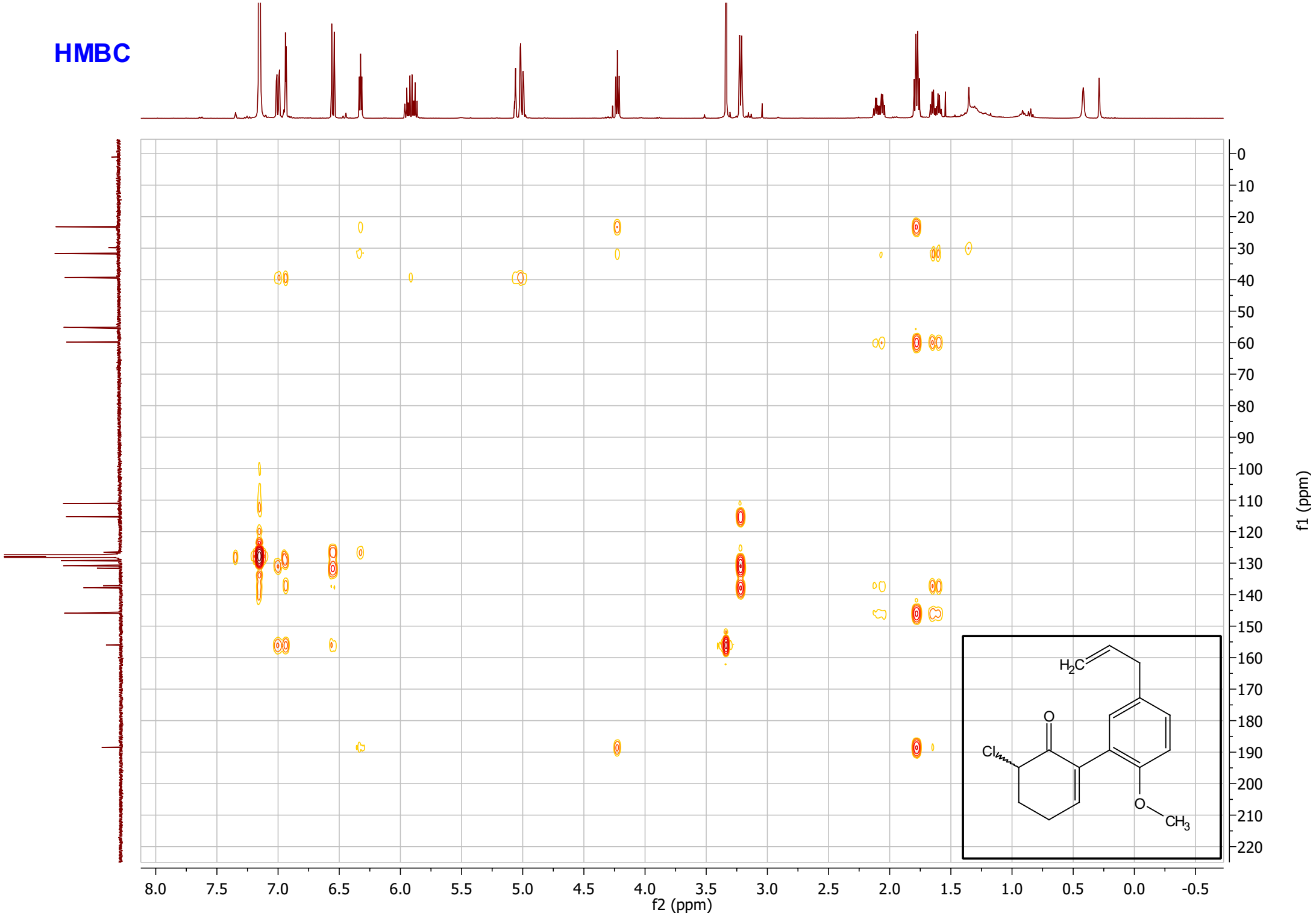
f1 (ppm)

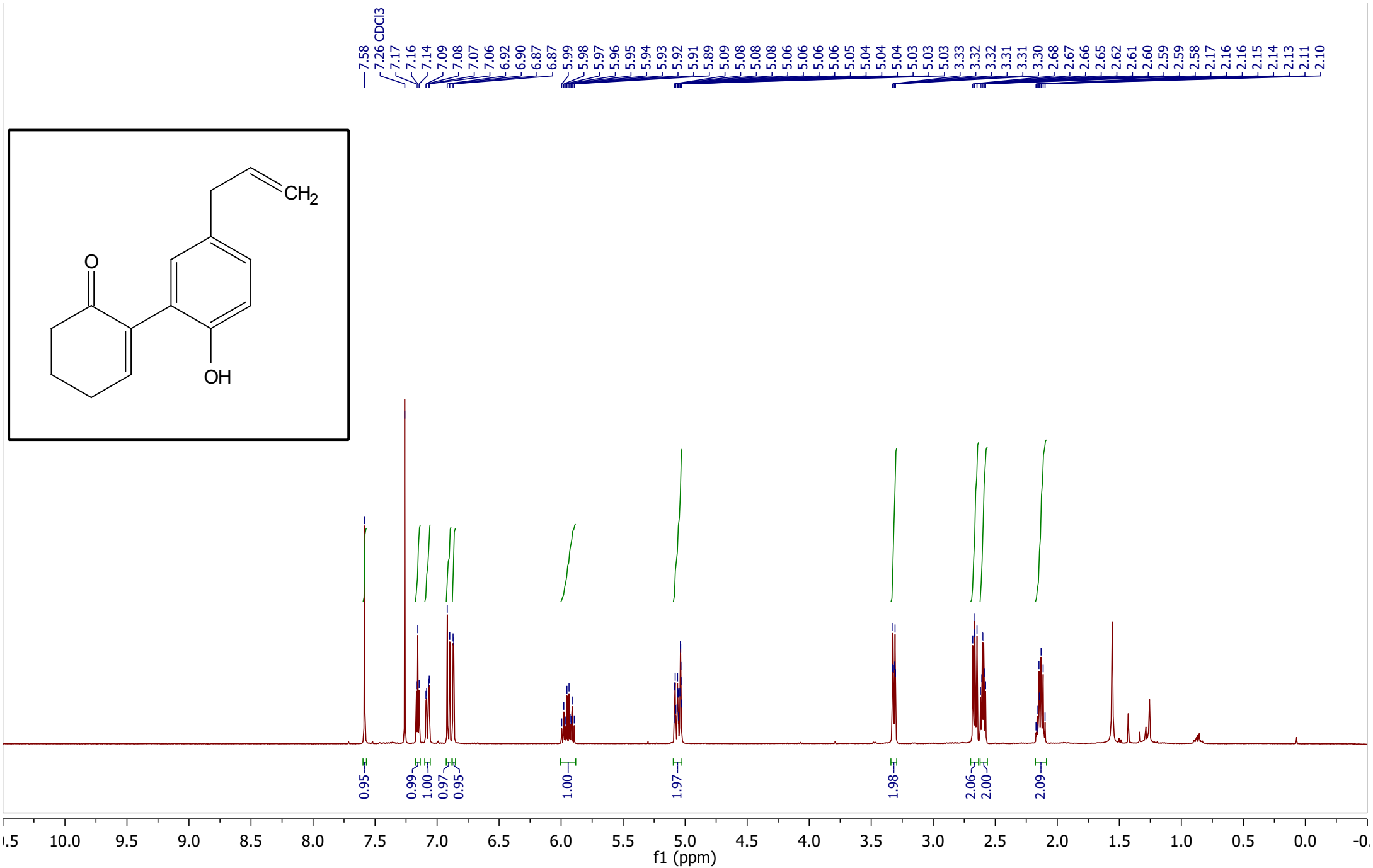
COSY


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
-0.5
f2 (ppm)
0
1
2
3
4
5
6
7
8
f1 (ppm)
H2C
O
Cl
O
CH3

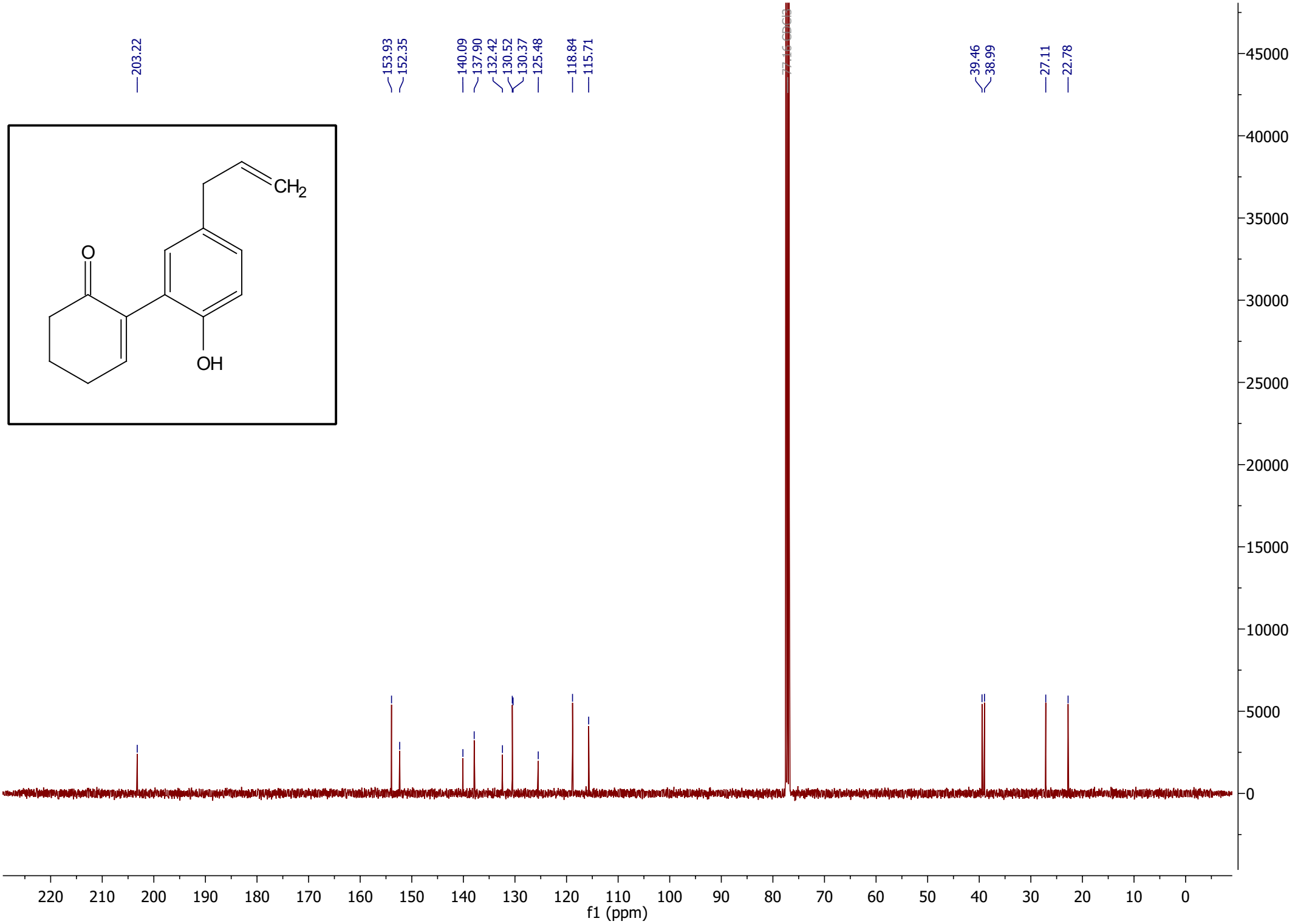
HSQC

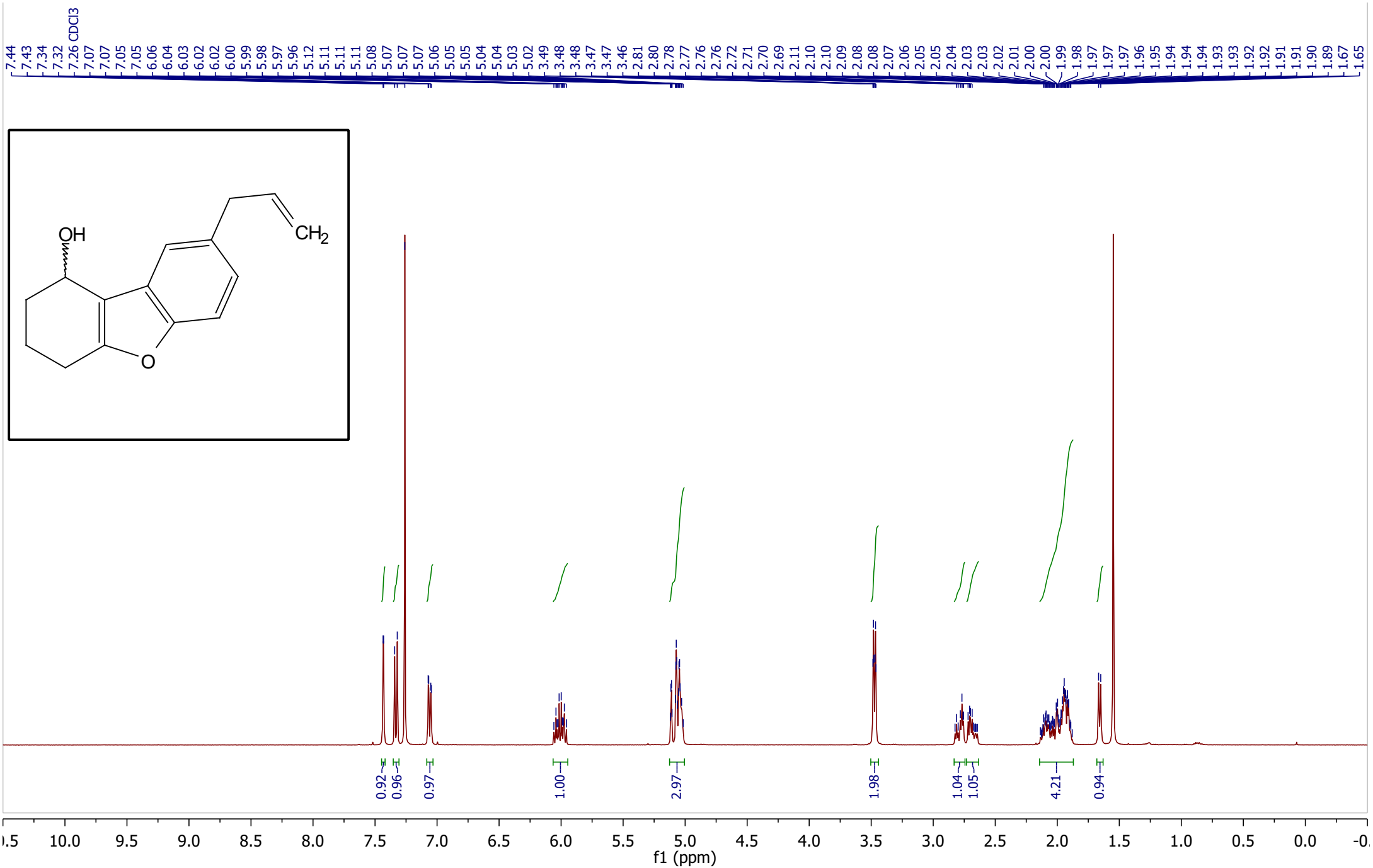

f2 (ppm)
f1 (ppm)
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
H2C
O
Cl
O
CH3


HMBC
f2 (ppm)
f1 (ppm)
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
-0.5
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
210
220
H2C
O
Cl
O
CH3

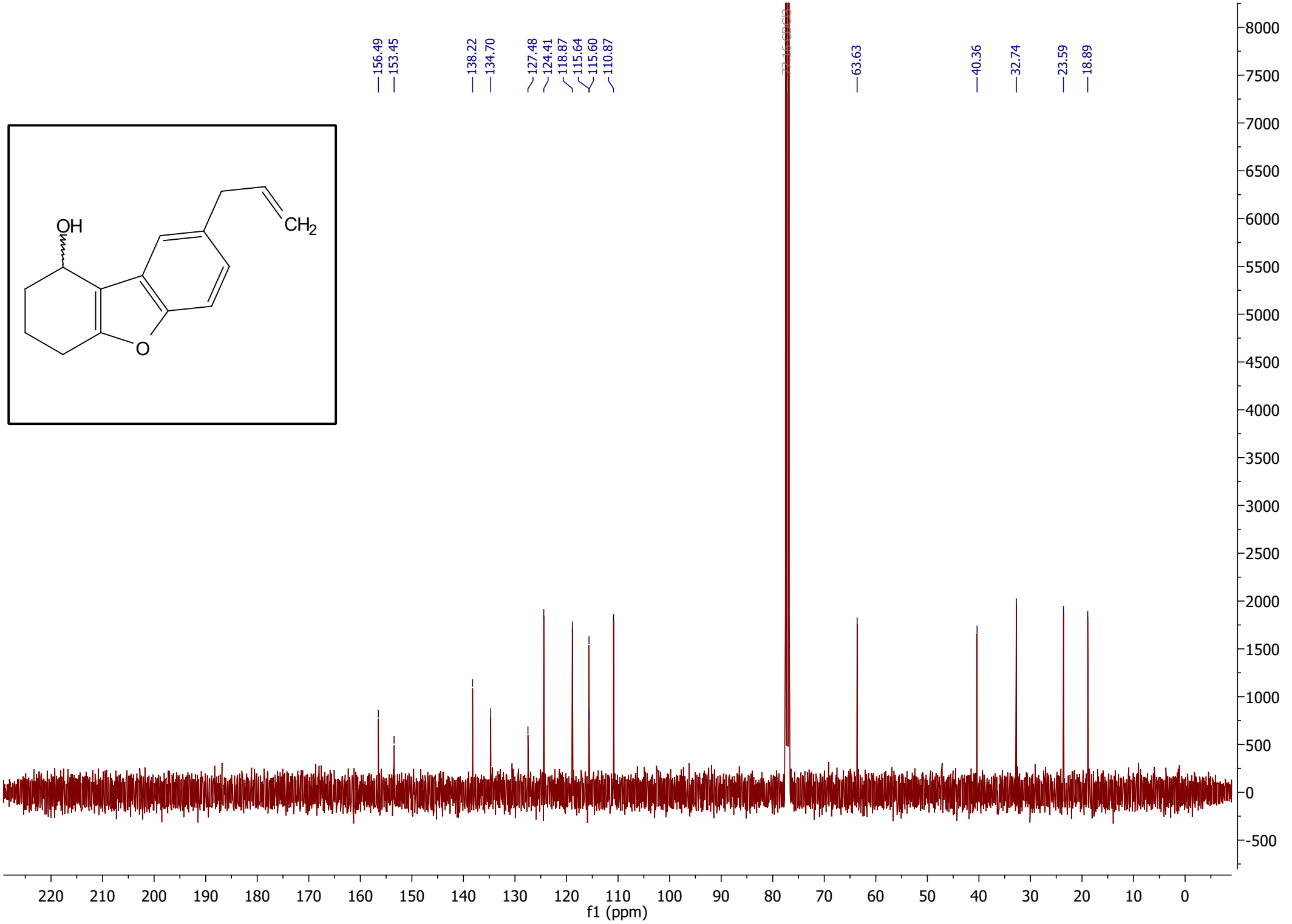


^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.58 (s, 1H), 7.16 (t, *J* = 4.3 Hz, 1H), 7.08 (dd, *J* = 8.2, 2.2 Hz, 1H), 6.91 (d, *J* = 8.2 Hz, 1H), 6.87 (d, *J* = 2.2 Hz, 1H), 5.94 (ddt, *J* = 16.9, 10.1, 6.7 Hz, 1H), 5.10 – 5.02 (m, 2H), 3.32 (ddd, *J* = 6.7, 1.4, 1.4 Hz, 2H), 2.70 – 2.64 (m, 2H), 2.60 (td, *J* = 6.0, 4.3 Hz, 2H), 2.19 – 2.07 (m, 2H).

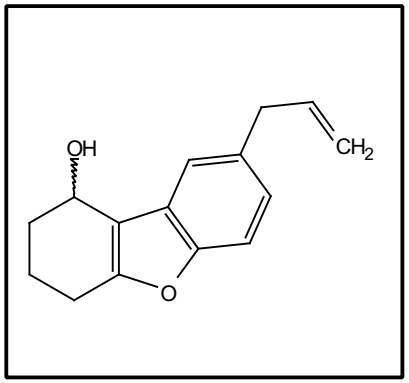

203.22
153.93
152.35
140.09
137.90
132.42
130.52
130.37
125.48
118.84
115.71
77.46
39.46
38.99
27.11
22.78
CH2
O
OH
45000
40000
35000
30000
25000
20000
15000
10000
5000
0
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.43 (d, *J* = 1.8 Hz, 1H), 7.33 (d, *J* = 8.4 Hz, 1H), 7.06 (dd, *J* = 8.4, 1.8 Hz, 1H), 6.01 (dddd, *J* = 16.8, 10.0, 6.7, 6.7 Hz, 1H), 5.12 – 5.01 (m, 3H), 3.48 (ddd, *J* = 6.7, 1.5, 1.5 Hz, 2H), 2.79 (ddd, *J* = 17.4, 5.1, 5.1 Hz, 1H), 2.68 (ddd, *J* = 17.4, 7.4, 4.7 Hz, 1H), 2.14 – 1.87 (m, 4H), 1.66 (d, *J* = 6.9 Hz, 1H).


156.49
153.45
138.22
134.70
127.48
124.41
118.87
115.64
115.60
110.87
77.16 CDCl3
63.63
40.36
32.74
23.59
18.89
OH
CH2
O
8000
7500
7000
6500
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

^{13}C DEPT

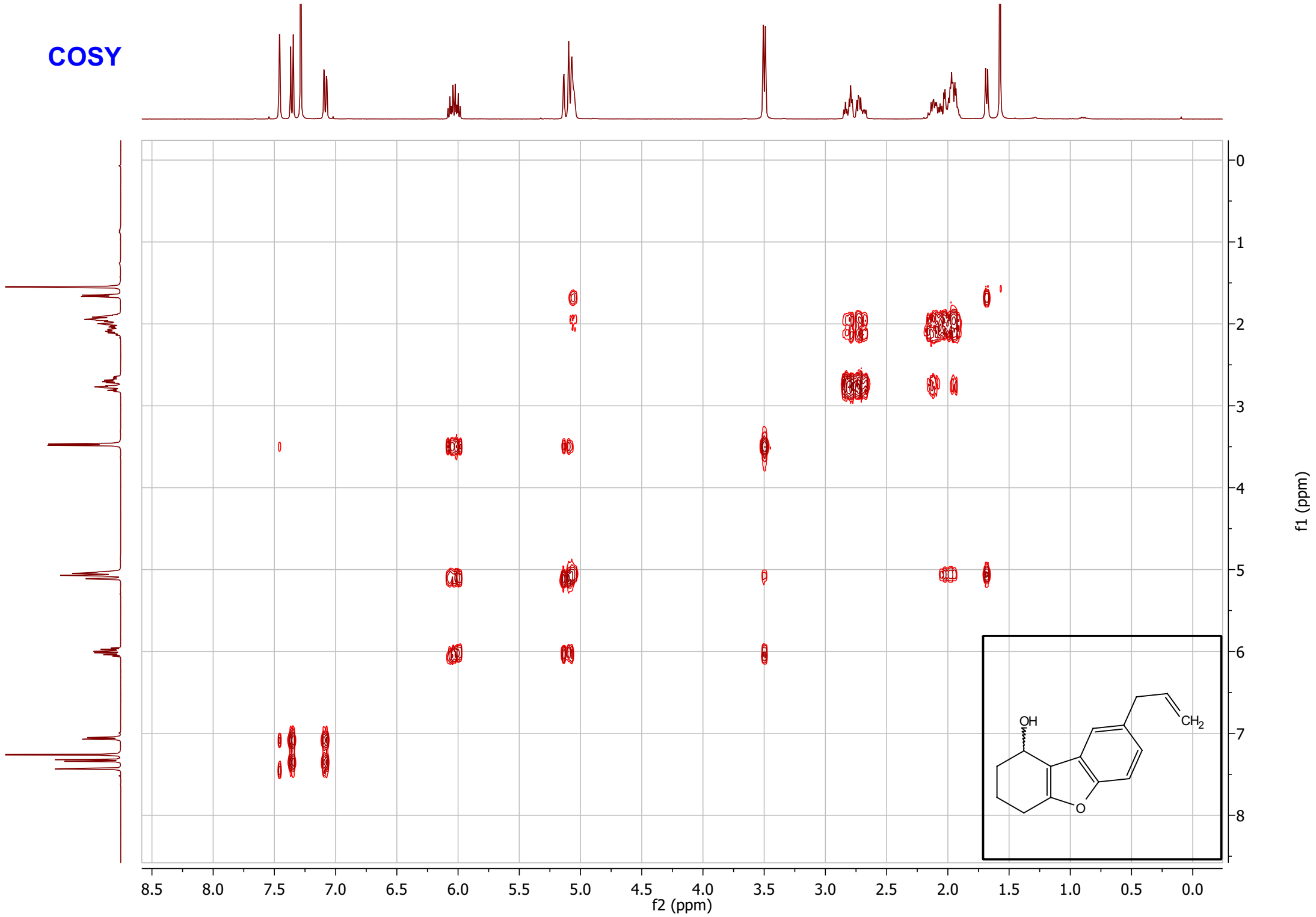


138.23
124.42
118.88
115.64
110.87
63.63 CDCl3
40.36
32.74
23.60
18.90

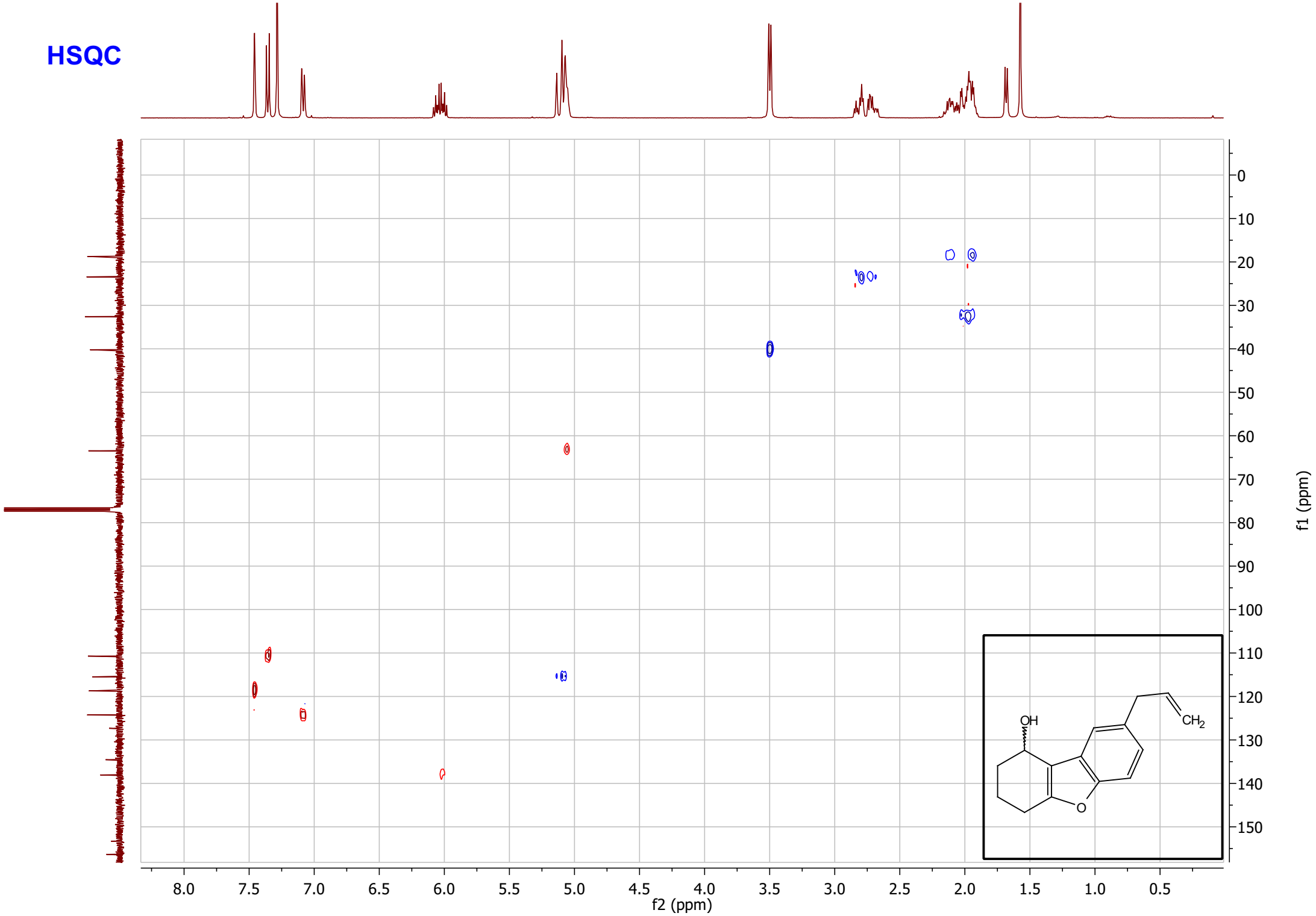
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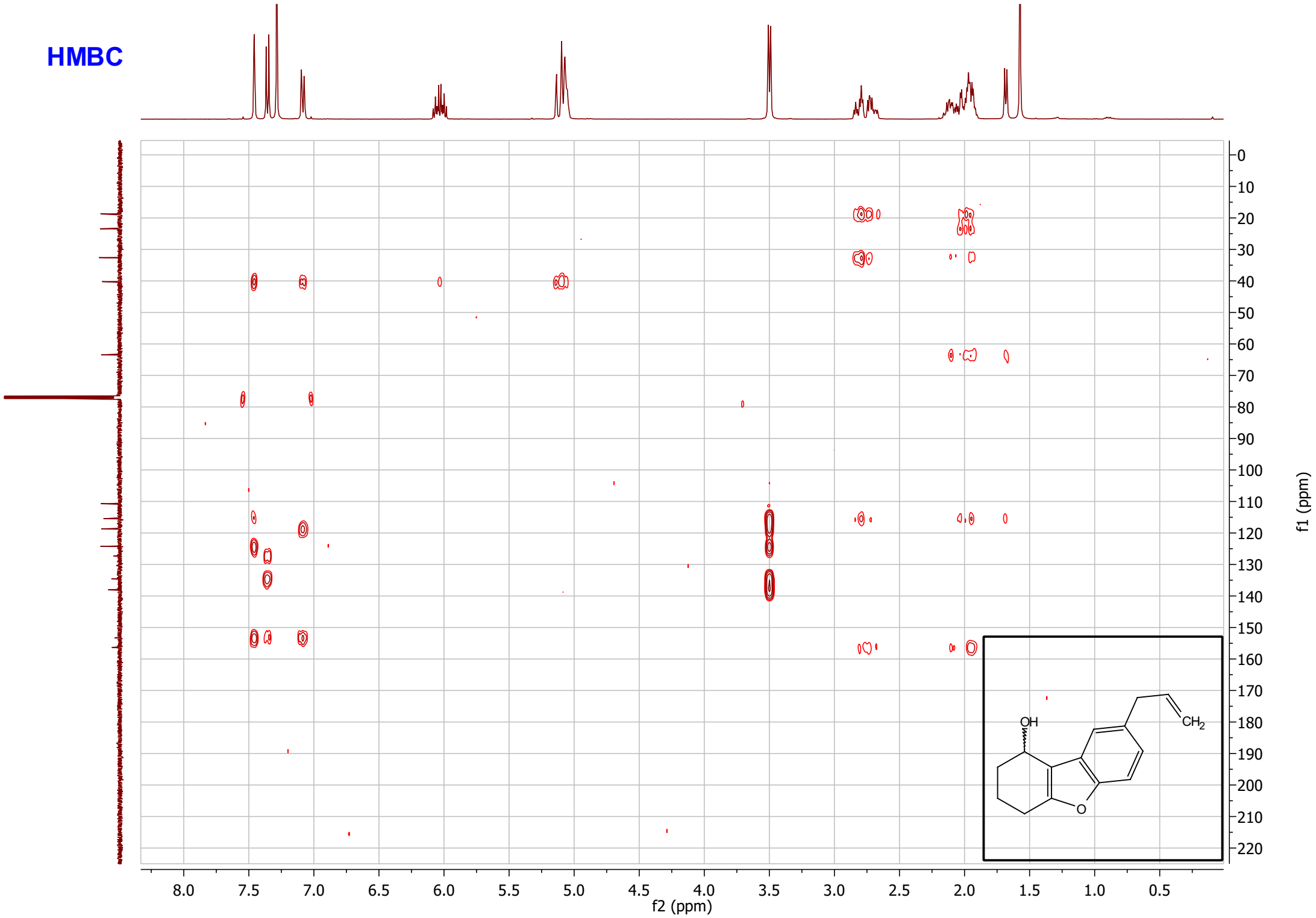
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0

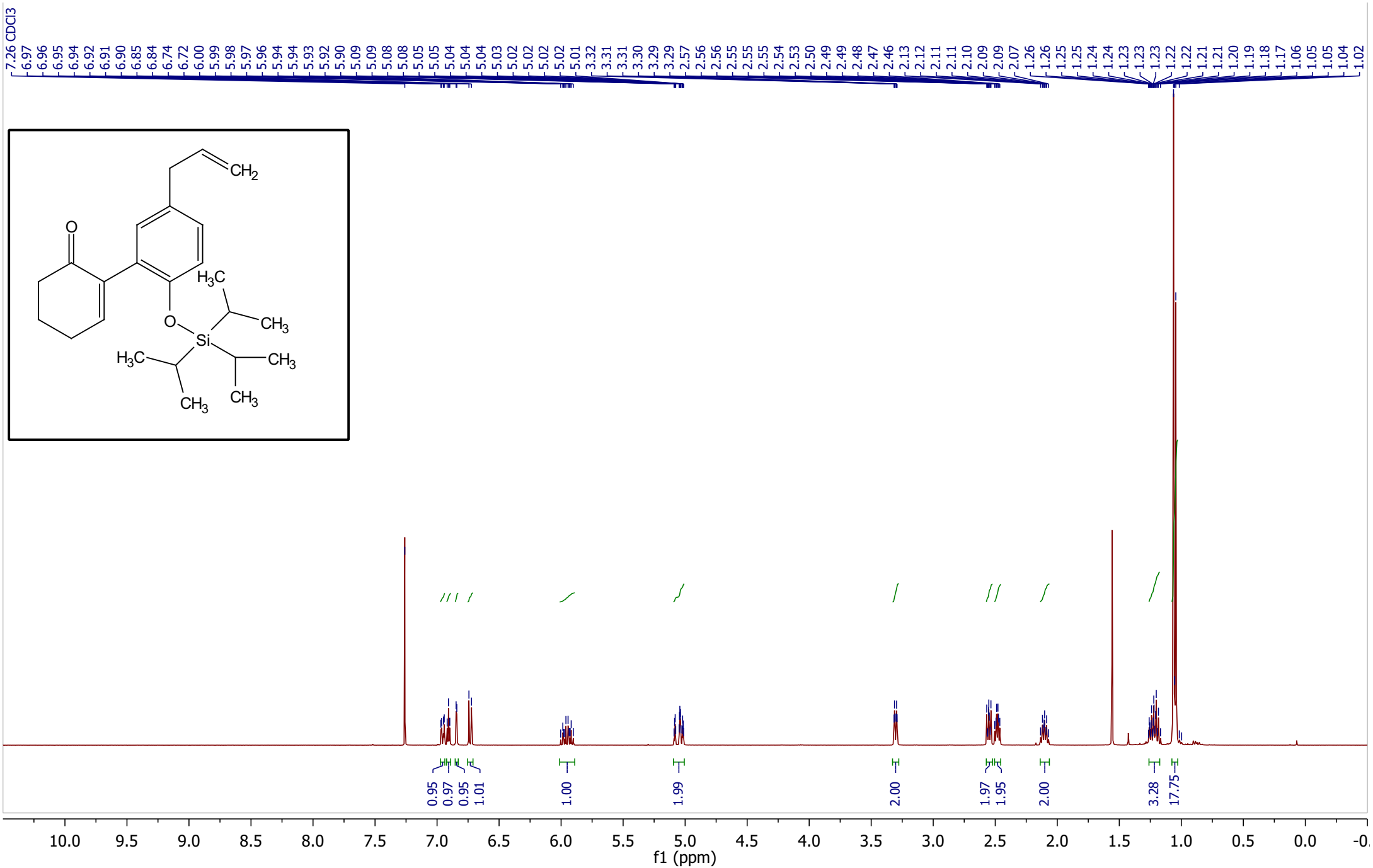
f1 (ppm)


COSY
f2 (ppm)
f1 (ppm)
OH
CH2
O

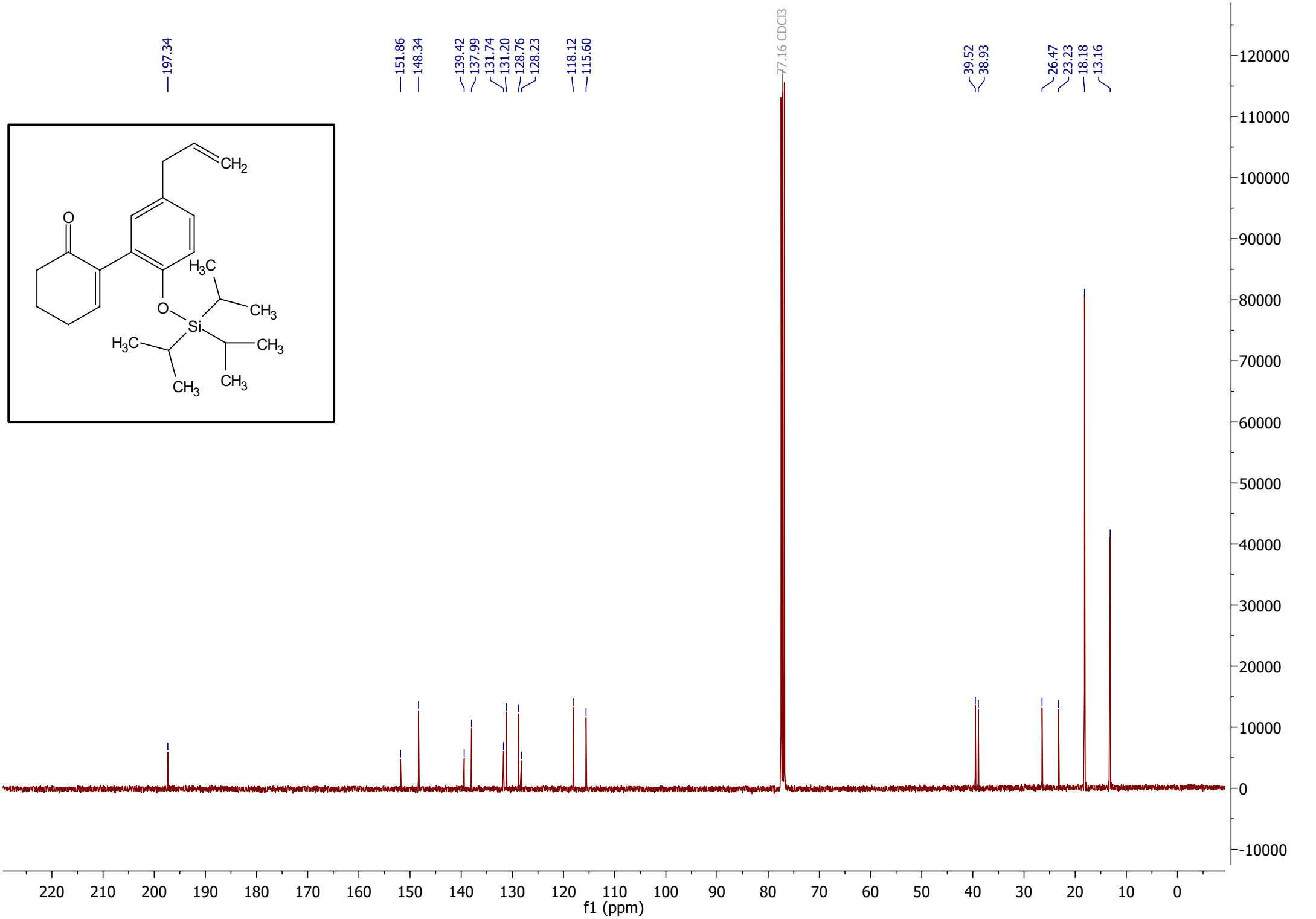
HSQC

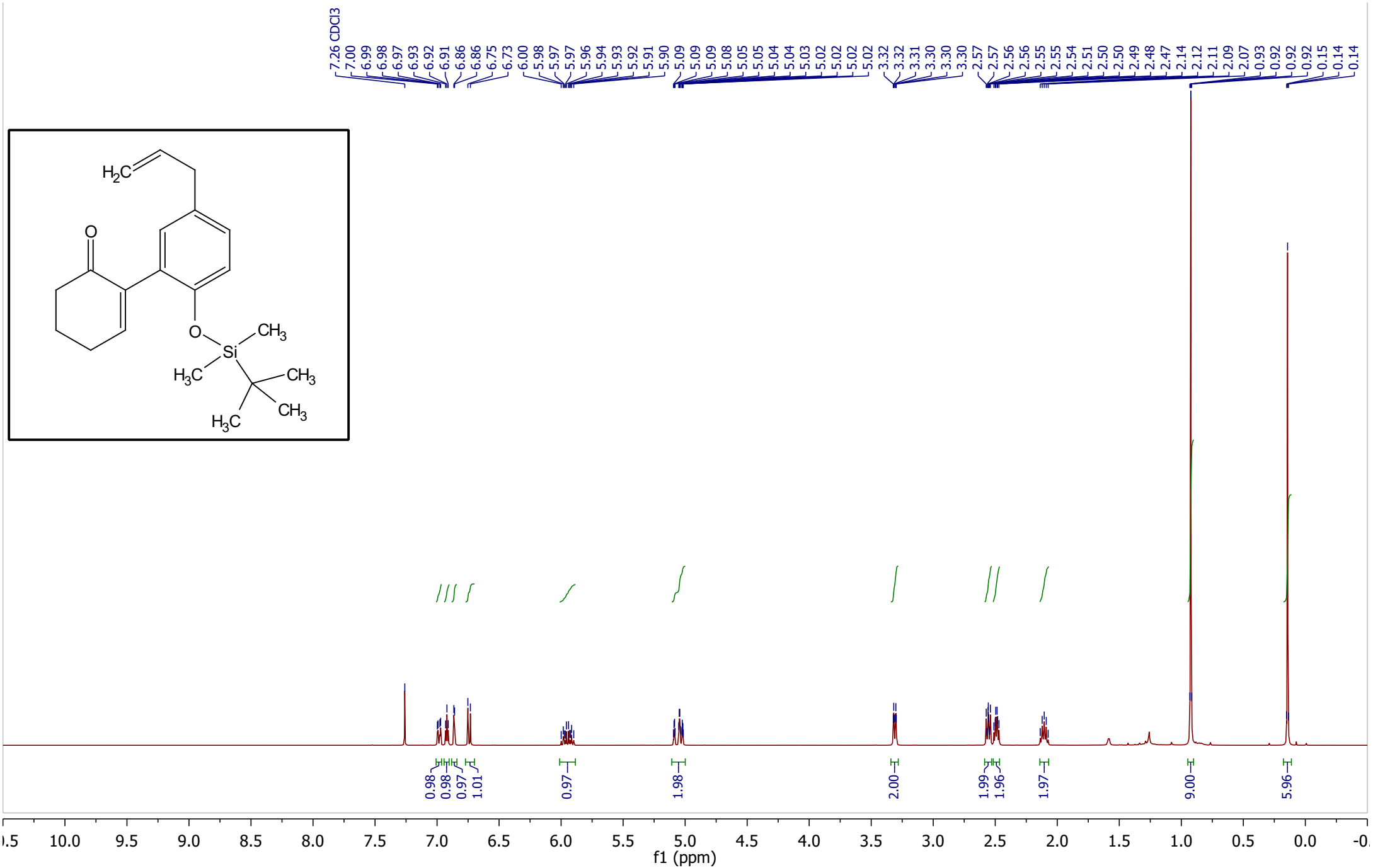

8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f2 (ppm)
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
f1 (ppm)
OH
CH2
O


HMBC
f2 (ppm)
f1 (ppm)
OH
CH2
O

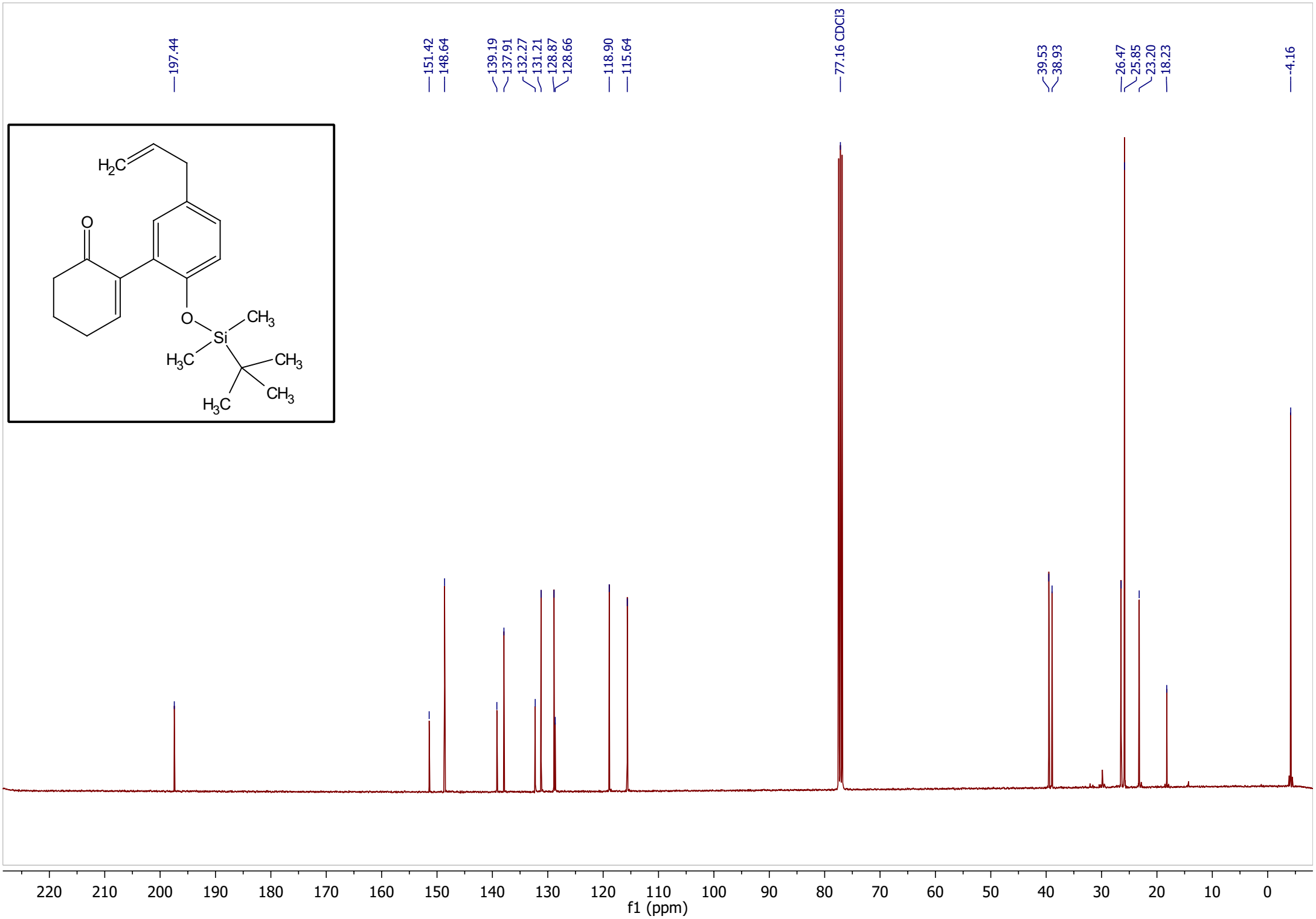


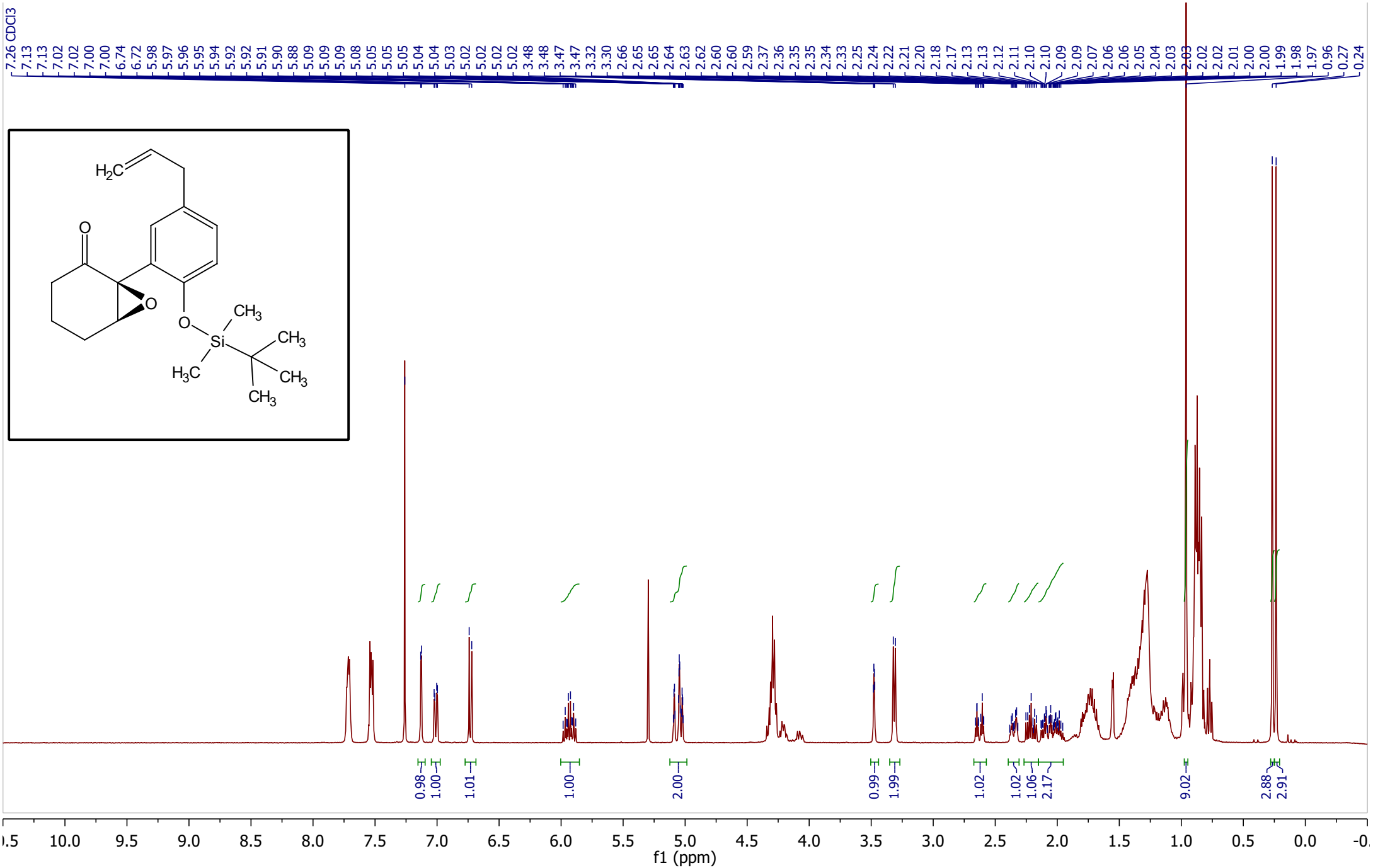
^{1}H NMR (400 MHz, Chloroform-*d*) δ 6.96 (dd, *J* = 8.3, 2.4 Hz, 1H), 6.91 (t, *J* = 4.2 Hz, 1H), 6.84 (d, *J* = 2.4 Hz, 1H), 6.73 (d, *J* = 8.3 Hz, 1H), 5.95 (ddt, *J* = 16.8, 10.0, 6.8 Hz, 1H), 5.09 – 5.01 (m, 2H), 3.30 (ddd, *J* = 6.8, 1.4, 1.4 Hz, 2H), 2.59 – 2.52 (m, 2H), 2.48 (td, *J* = 6.0, 4.2 Hz, 2H), 2.14 – 2.06 (m, 2H), 1.27 – 1.16 (m, 3H), 1.05 (d, *J* = 7.3 Hz, 18H).


197.34
151.86
148.34
139.42
137.99
131.74
131.20
128.76
128.23
118.12
115.60
77.16 CDCl3
39.52
38.93
26.47
23.23
18.18
13.16
O
CH2
H3C
CH3
O
Si
H3C
CH3
CH3
CH3
120000
110000
100000
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
-10000
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

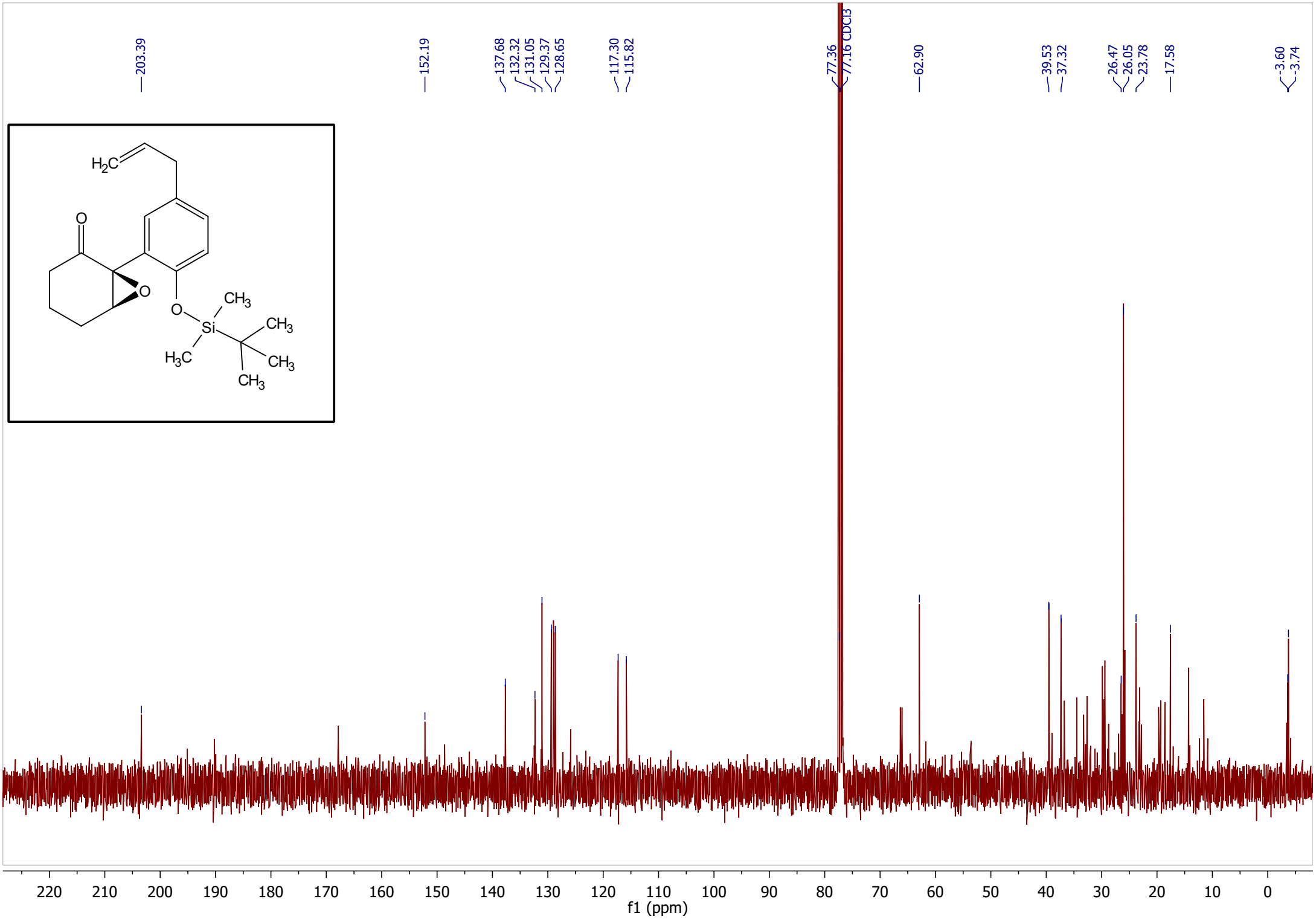


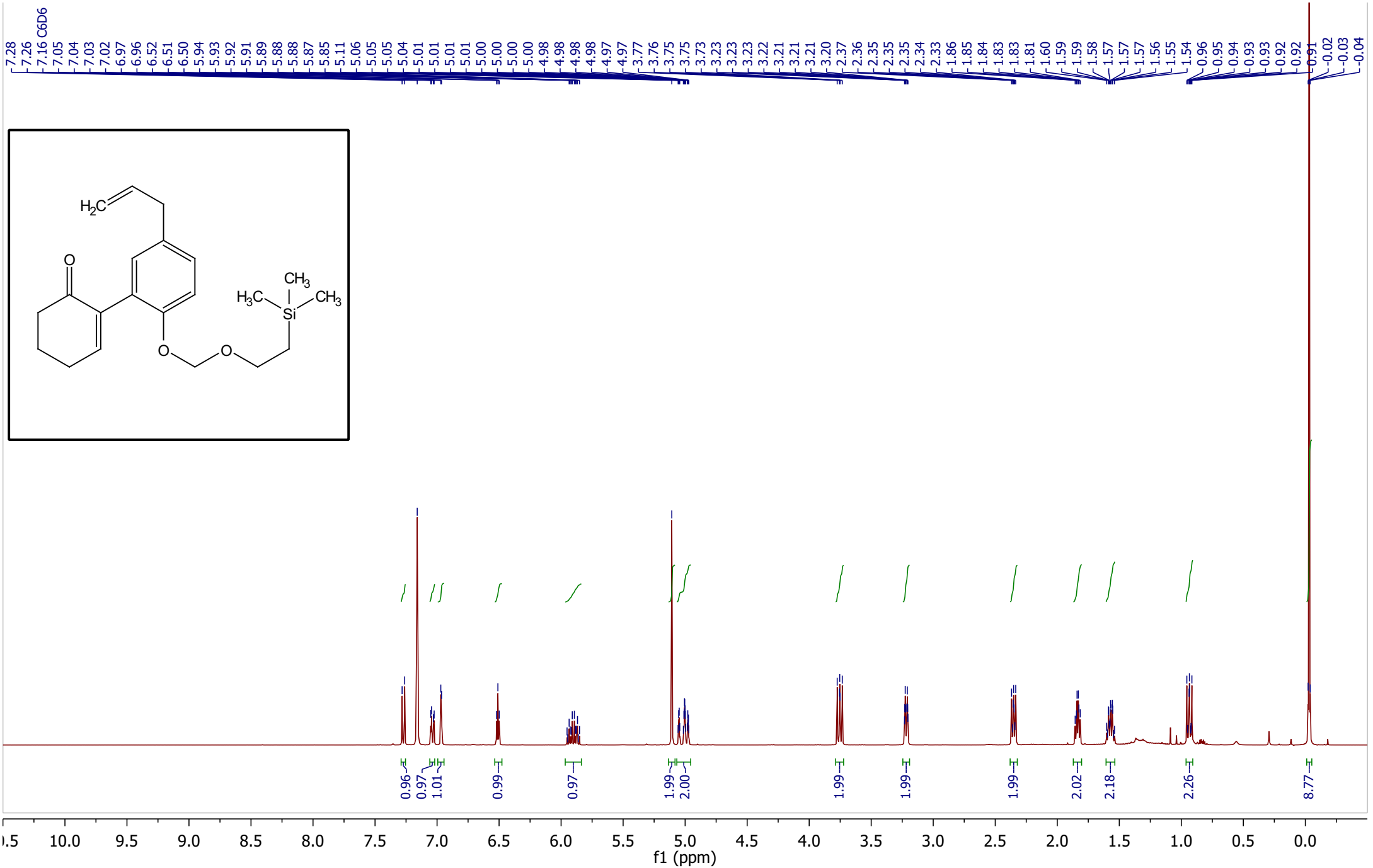
^{1}H NMR (400 MHz, Chloroform-*d*) δ 6.98 (dd, *J* = 8.2, 2.4 Hz, 1H), 6.92 (t, *J* = 4.2 Hz, 1H), 6.86 (d, *J* = 2.4 Hz, 1H), 6.74 (d, *J* = 8.2 Hz, 1H), 5.95 (ddt, *J* = 16.8, 10.0, 6.8 Hz, 1H), 5.11 – 5.00 (m, 2H), 3.31 (ddd, *J* = 6.7, 1.4, 1.4 Hz, 2H), 2.58 – 2.52 (m, 2H), 2.49 (td, *J* = 6.0, 4.2 Hz, 2H), 2.15 – 2.07 (m, 2H), 0.92 (s, 9H), 0.14 (s, 6H).


197.44
151.42
148.64
139.19
137.91
132.27
131.21
128.87
128.66
118.90
115.64
77.16 CDCl3
39.53
38.93
26.47
25.85
23.20
18.23
-4.16
H2C
O
O
Si
CH3
H3C
CH3
CH3
H3C
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

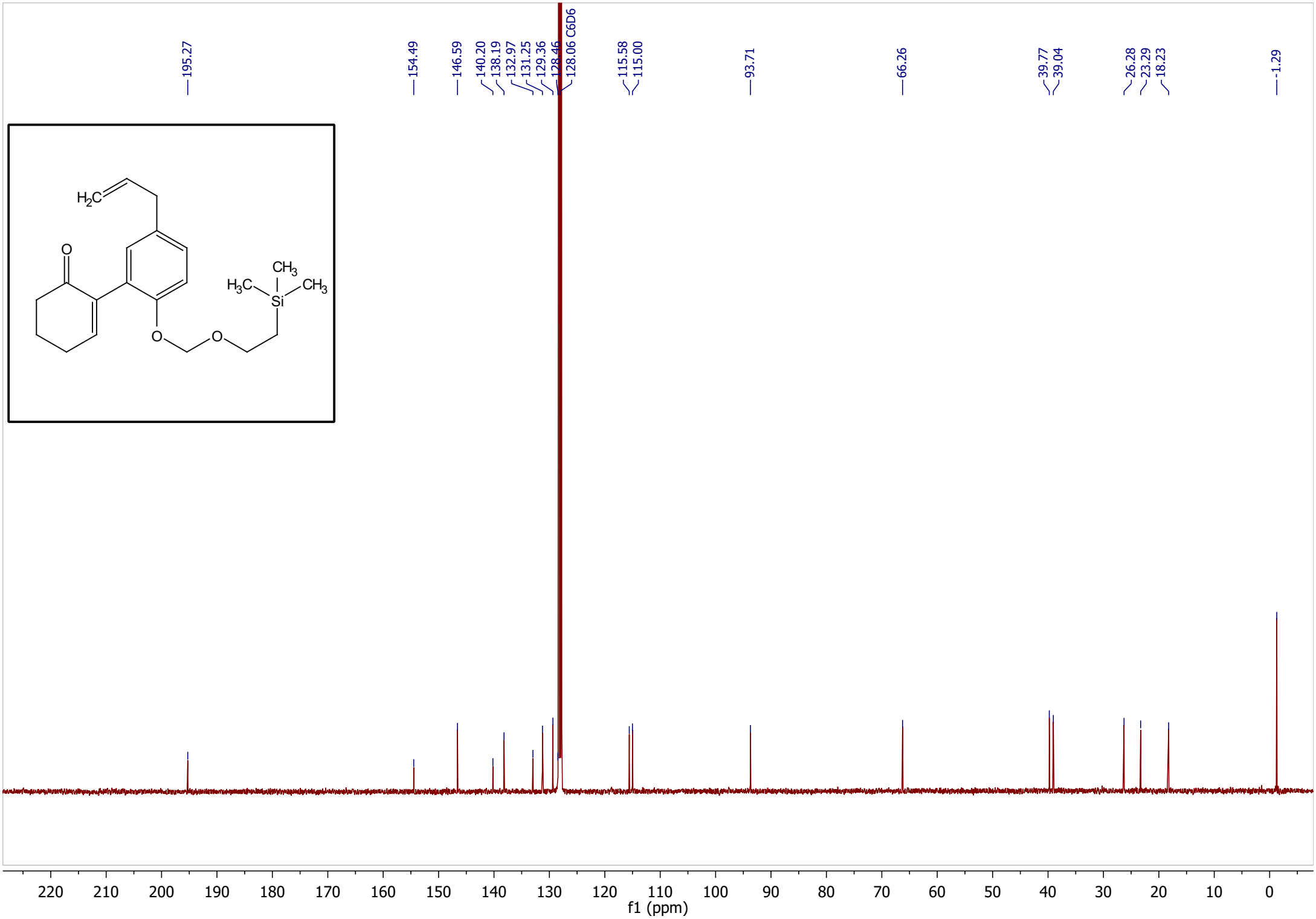


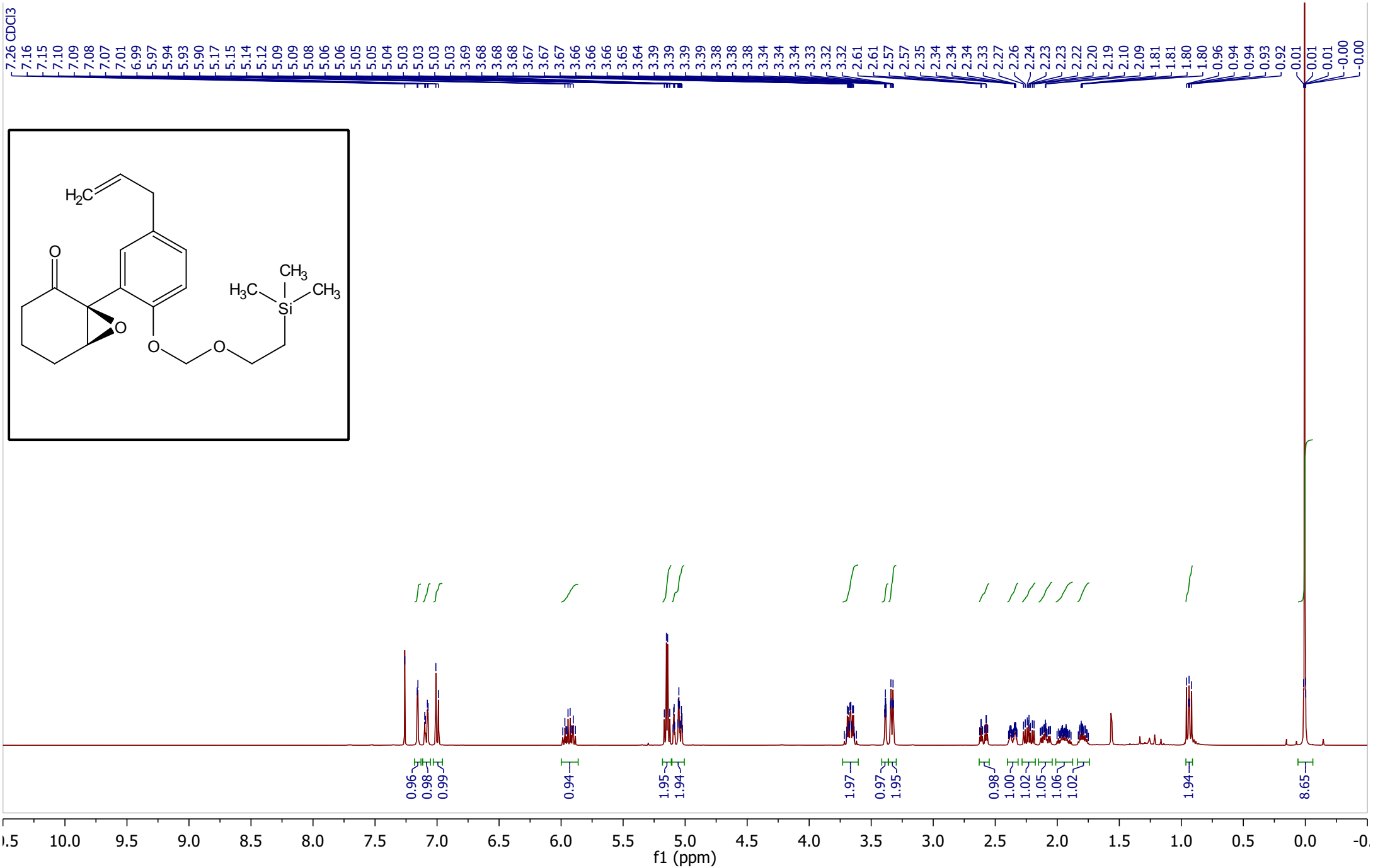
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.13 (d, *J* = 2.4 Hz, 1H), 7.01 (dd, *J* = 8.3, 2.4 Hz, 1H), 6.73 (d, *J* = 8.3 Hz, 1H), 5.93 (dddd, *J* = 16.9, 10.0, 6.8, 6.8 Hz, 1H), 5.11 – 5.01 (m, 2H), 3.49 – 3.46 (m, 1H), 3.31 (d, *J* = 6.8 Hz, 2H), 2.68 – 2.57 (m, 1H), 2.40 – 2.30 (m, 1H), 2.26 – 2.15 (m, 1H), 2.14 – 1.95 (m, 2H), 0.96 (s, 9H), 0.27 (s, 3H), 0.24 (s, 3H).


203.39
152.19
137.68
132.32
131.05
129.37
128.65
117.30
115.82
77.36
77.16 CDCl3
62.90
39.53
37.32
26.47
26.05
23.78
17.58
-3.60
-3.74
H2C
O
O
O
Si
CH3
CH3
CH3
H3C
CH3
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

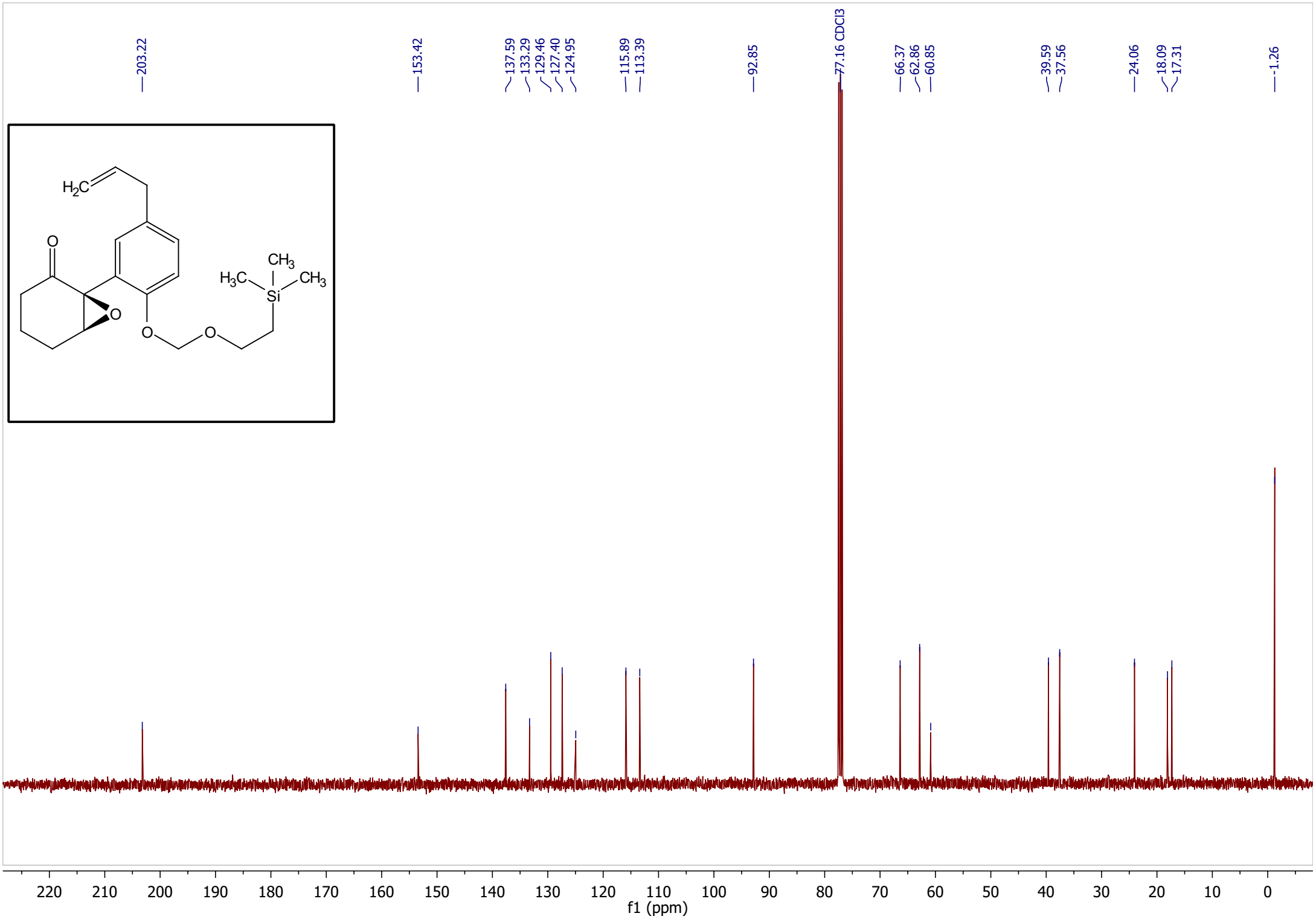


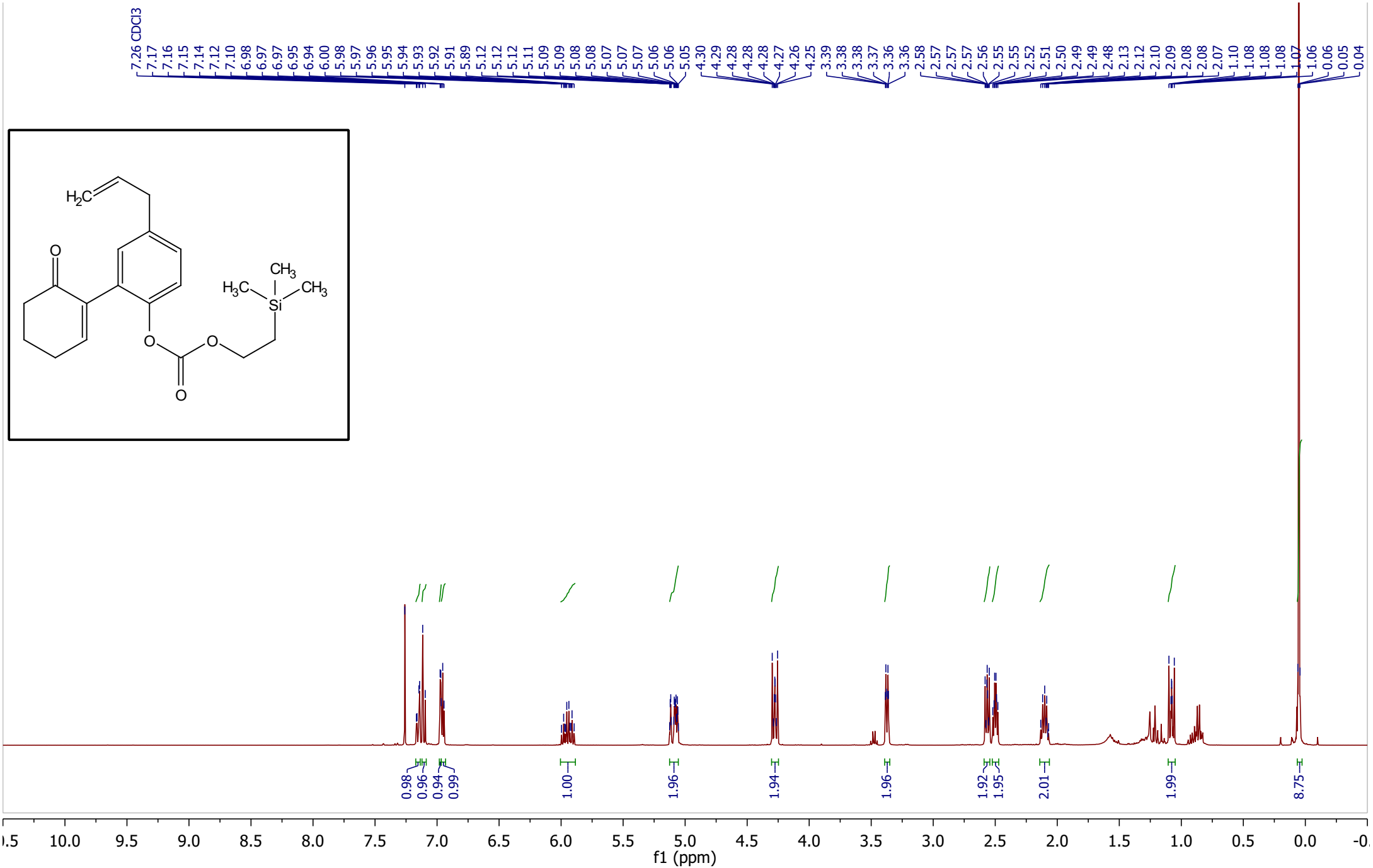
^{1}H NMR (400 MHz, Benzene-d_6) δ 7.27 (d, J = 8.3 Hz, 1H), 7.04 (dd, J = 8.3, 2.3 Hz, 1H), 6.97 (d, J = 2.3 Hz, 1H), 6.51 (t, J = 4.2 Hz, 1H), 5.90 (ddt, J = 16.7, 10.0, 6.7 Hz, 1H), 5.11 (s, 2H), 5.06 – 4.96 (m, 2H), 3.79 – 3.70 (m, 2H), 3.22 (ddd, J = 6.5, 1.3, 1.3 Hz, 2H), 2.39 – 2.29 (m, 2H), 1.84 (td, J = 6.0, 4.2 Hz, 2H), 1.62 – 1.52 (m, 2H), 0.98 – 0.90 (m, 2H), -0.03 (s, 9H).


195.27
154.49
146.59
140.20
138.19
132.97
131.25
129.36
128.46
128.06 C6D6
115.58
115.00
93.71
66.26
39.77
39.04
26.28
23.29
18.23
-1.29
H2C
O
CH3
H3C
Si
CH3
O
O
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

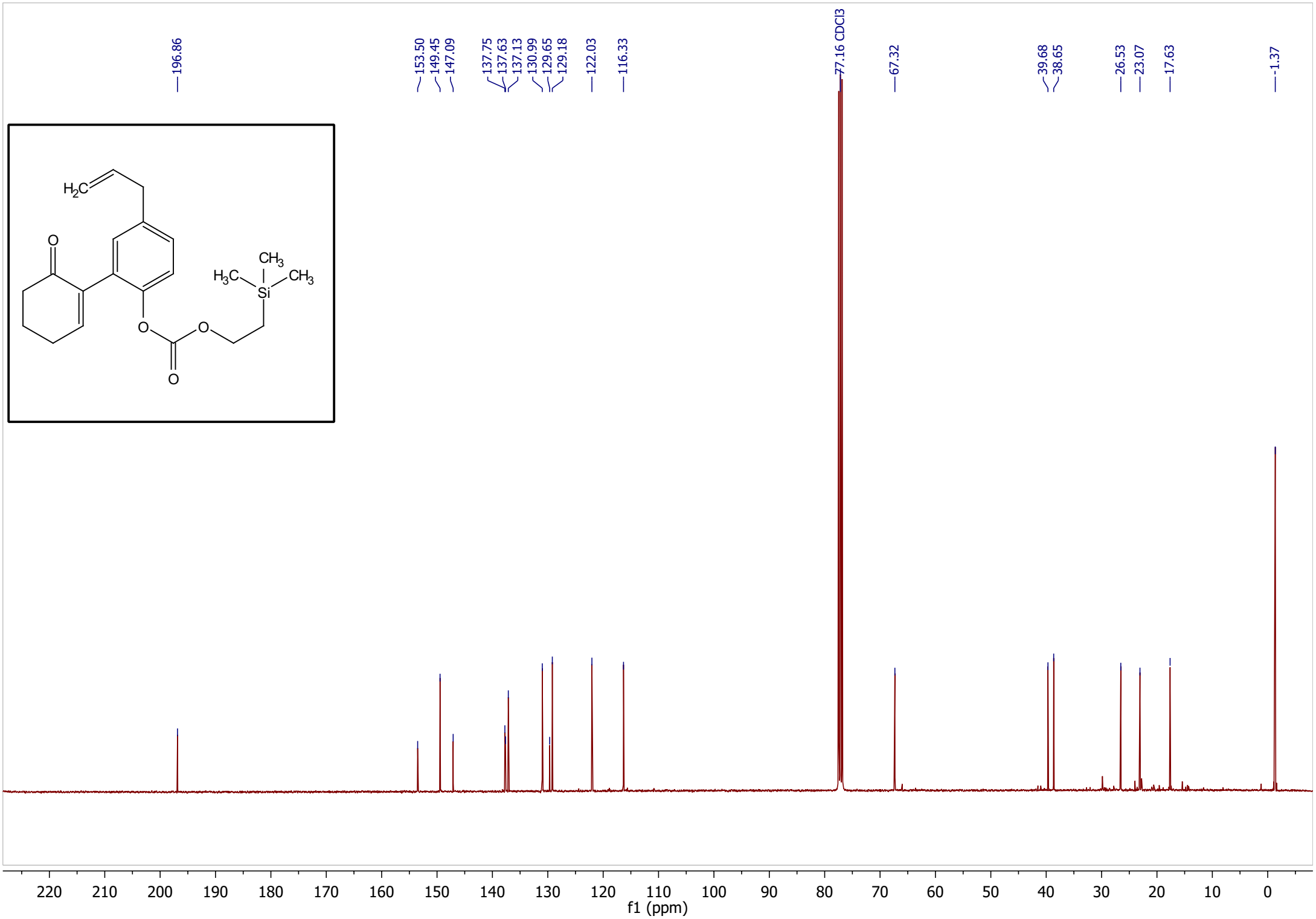


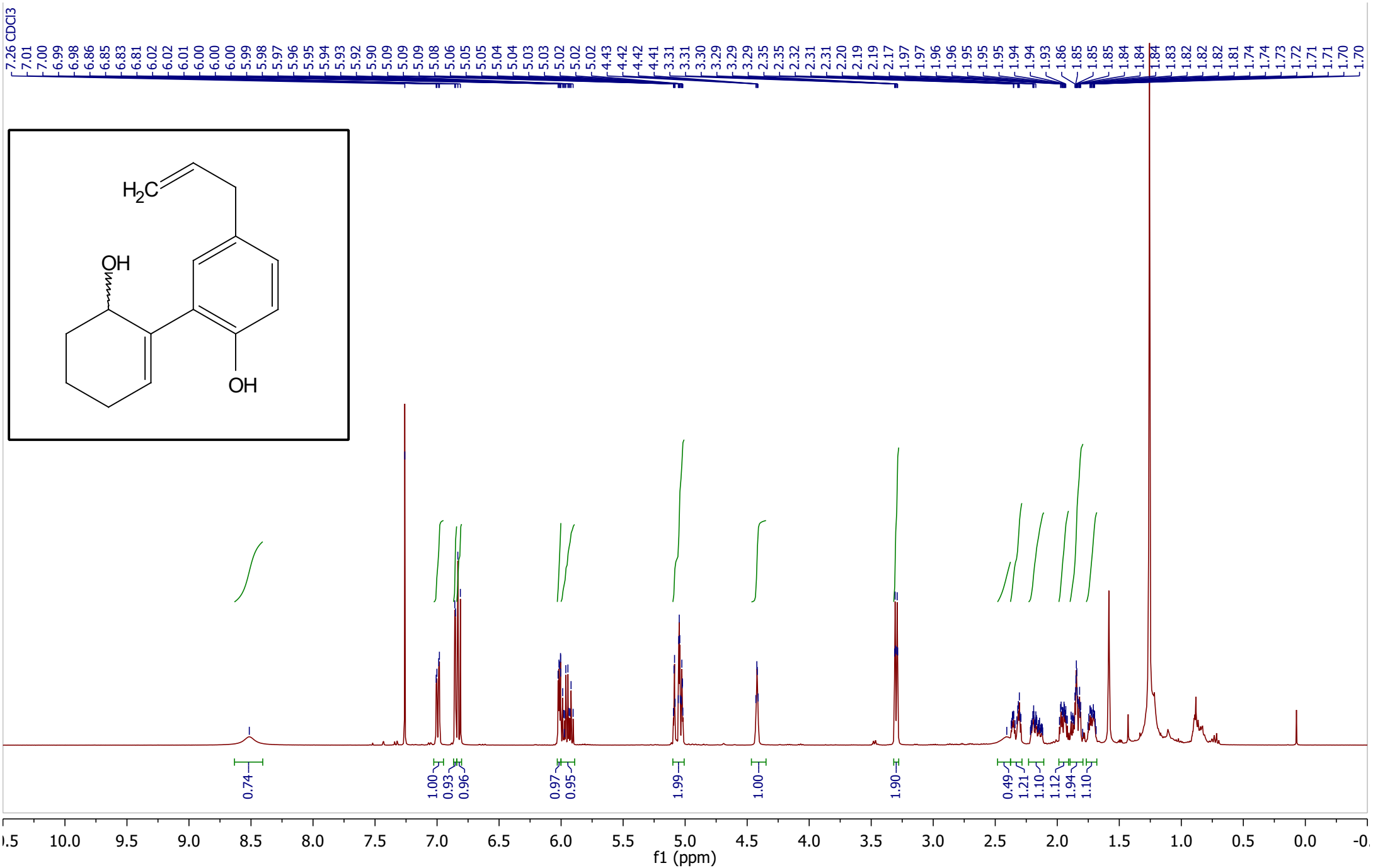
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.16 (d, *J* = 2.2 Hz, 1H), 7.09 (dd, *J* = 8.4, 2.2 Hz, 1H), 7.00 (d, *J* = 8.4 Hz, 1H), 5.94 (dddd, *J* = 16.9, 10.0, 6.8, 6.8 Hz, 1H), 5.17 – 5.12 (m, 2H), 5.10 – 5.01 (m, 2H), 3.72 – 3.62 (m, 2H), 3.39 (ddd, *J* = 2.4, 1.6, 0.6 Hz, 1H), 3.33 (ddd, *J* = 6.8, 1.4, 1.4 Hz, 2H), 2.59 (dddd, *J* = 16.4, 4.3, 4.3, 1.0 Hz, 1H), 2.40 – 2.32 (m, 1H), 2.23 (ddd, *J* = 16.4, 12.0, 5.6 Hz, 1H), 2.10 (dddd, *J* = 14.9, 11.1, 5.3, 1.6 Hz, 1H), 2.00 – 1.88 (m, 1H), 1.84 – 1.75 (m, 1H), 0.96 – 0.91 (m, 2H), 0.01 (s, 9H).


203.22
153.42
137.59
133.29
129.46
127.40
124.95
115.89
113.39
92.85
77.16 CDCl3
66.37
62.86
60.85
39.59
37.56
24.06
18.09
17.31
-1.26
H2C
O
O
O
O
CH3
H3C
CH3
Si
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

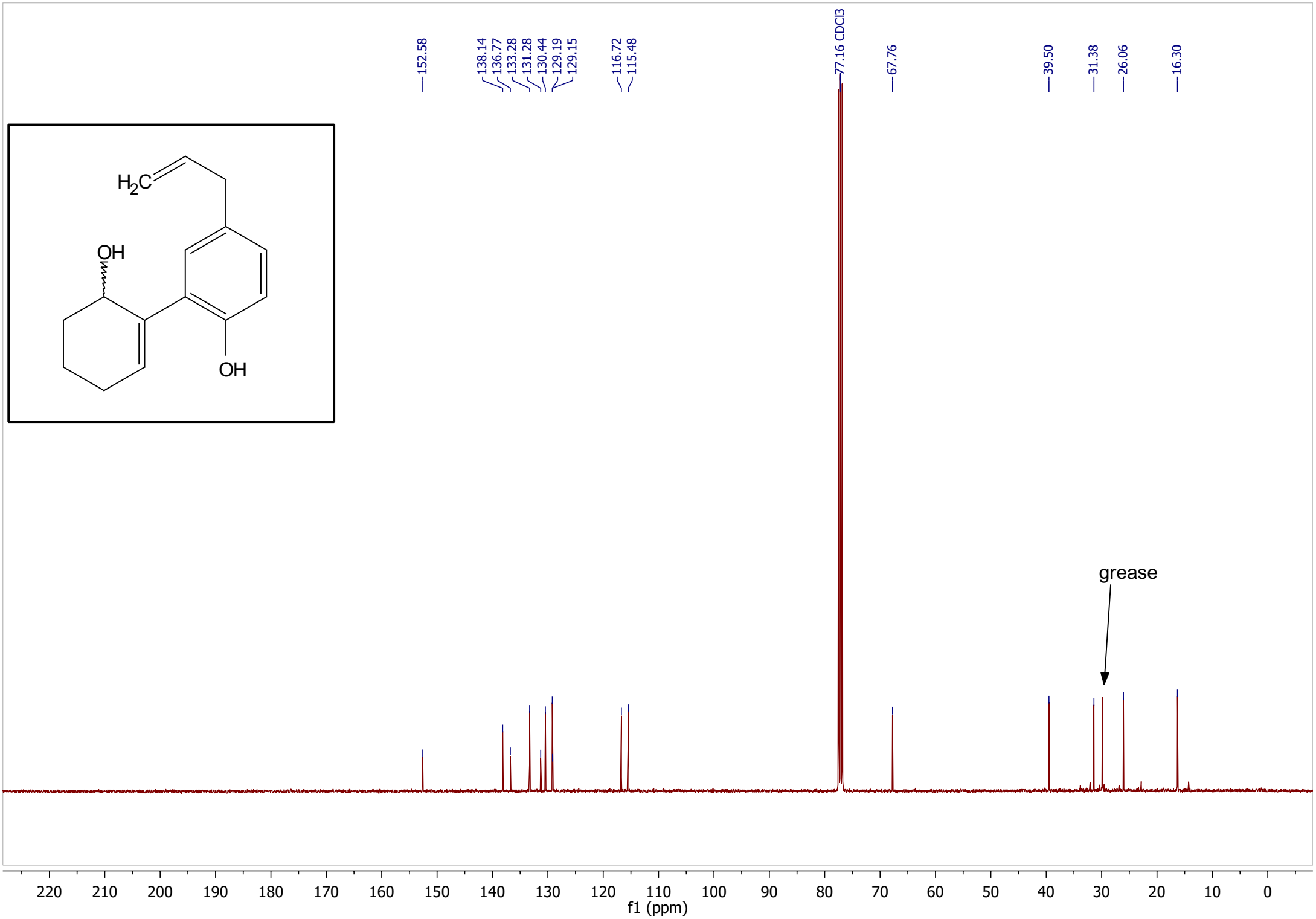


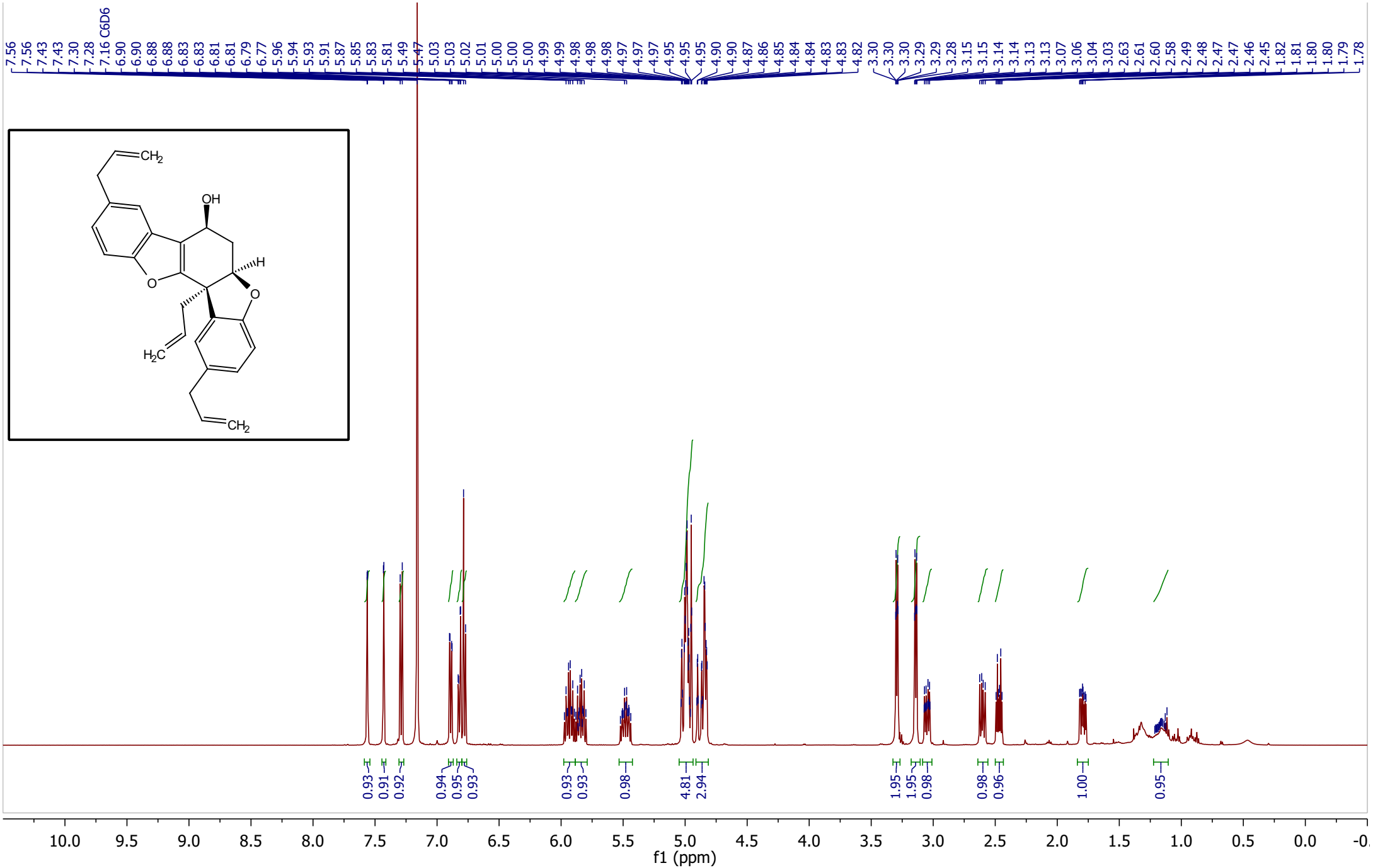
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.15 (dd, *J* = 8.3, 2.2 Hz, 1H), 7.11 (d, *J* = 8.3 Hz, 1H), 6.97 (d, *J* = 2.2 Hz, 1H), 6.95 (t, *J* = 4.2 Hz, 1H), 5.95 (ddt, *J* = 16.9, 10.0, 6.8 Hz, 1H), 5.18 – 4.98 (m, 2H), 4.34 – 4.21 (m, 2H), 3.37 (ddd, *J* = 6.8, 1.4, 1.4 Hz, 2H), 2.59 – 2.53 (m, 2H), 2.50 (td, *J* = 6.0, 4.1 Hz, 2H), 2.15 – 2.06 (m, 2H), 1.11 – 1.04 (m, 2H), 0.05 (s, 9H).


196.86
153.50
149.45
147.09
137.75
137.63
137.13
130.99
129.65
129.18
122.03
116.33
77.16 CDCl3
67.32
39.68
38.65
26.53
23.07
17.63
-1.37
O
O
O
O
H2C
CH3
H3C
CH3
Si
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

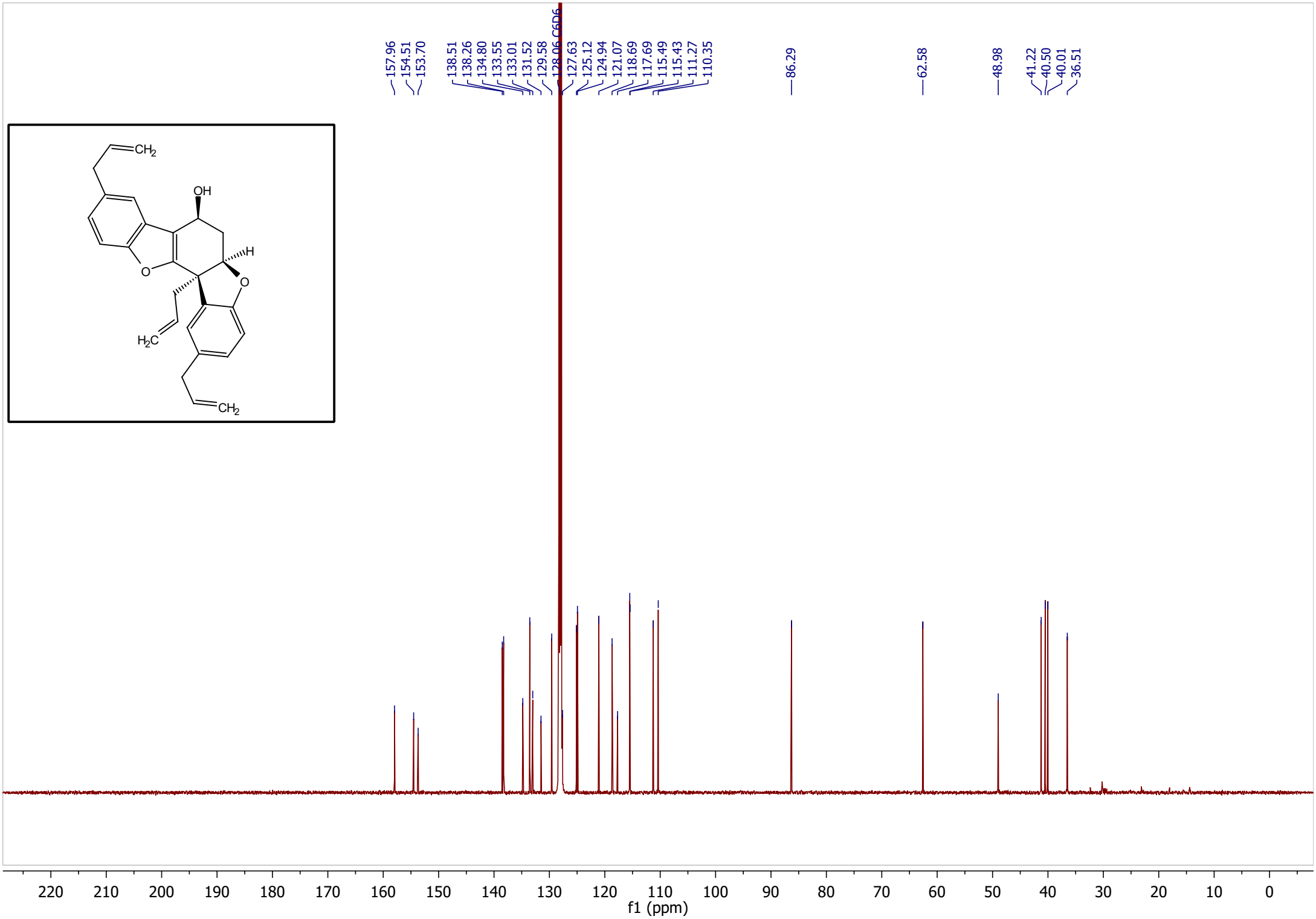


^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.51 (s, 1H), 6.99 (dd, *J* = 8.2, 2.3 Hz, 1H), 6.85 (d, *J* = 2.3 Hz, 1H), 6.82 (d, *J* = 8.2 Hz, 1H), 6.01 (dd, *J* = 4.8, 3.0 Hz, 1H), 5.95 (dddd, *J* = 16.8, 10.0, 6.6, 6.6 Hz, 1H), 5.10 – 5.01 (m, 2H), 4.46 – 4.39 (m, 1H), 3.30 (ddd, *J* = 6.7, 1.5, 1.5 Hz, 2H), 2.41 (s, 1H, alkylOH), 2.38 – 2.28 (m, 1H), 2.24 – 2.10 (m, 1H), 1.99 – 1.91 (m, 1H), 1.89 – 1.79 (m, 2H), 1.77 – 1.67 (m, 1H).

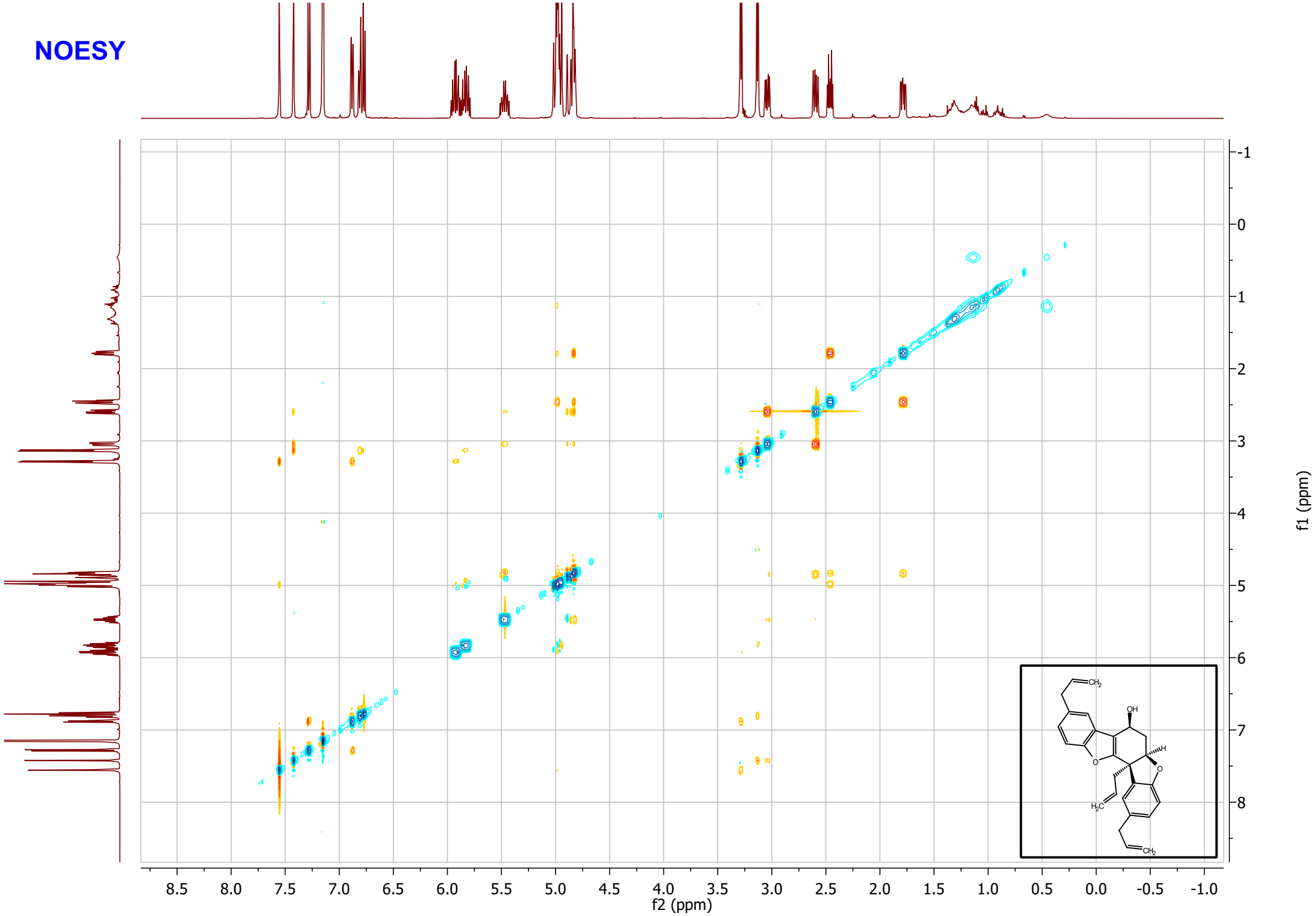

152.58
138.14
136.77
133.28
131.28
130.44
129.19
129.15
116.72
115.48
77.16 CDCl3
67.76
39.50
31.38
26.06
16.30
H2C
OH
OH
grease
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

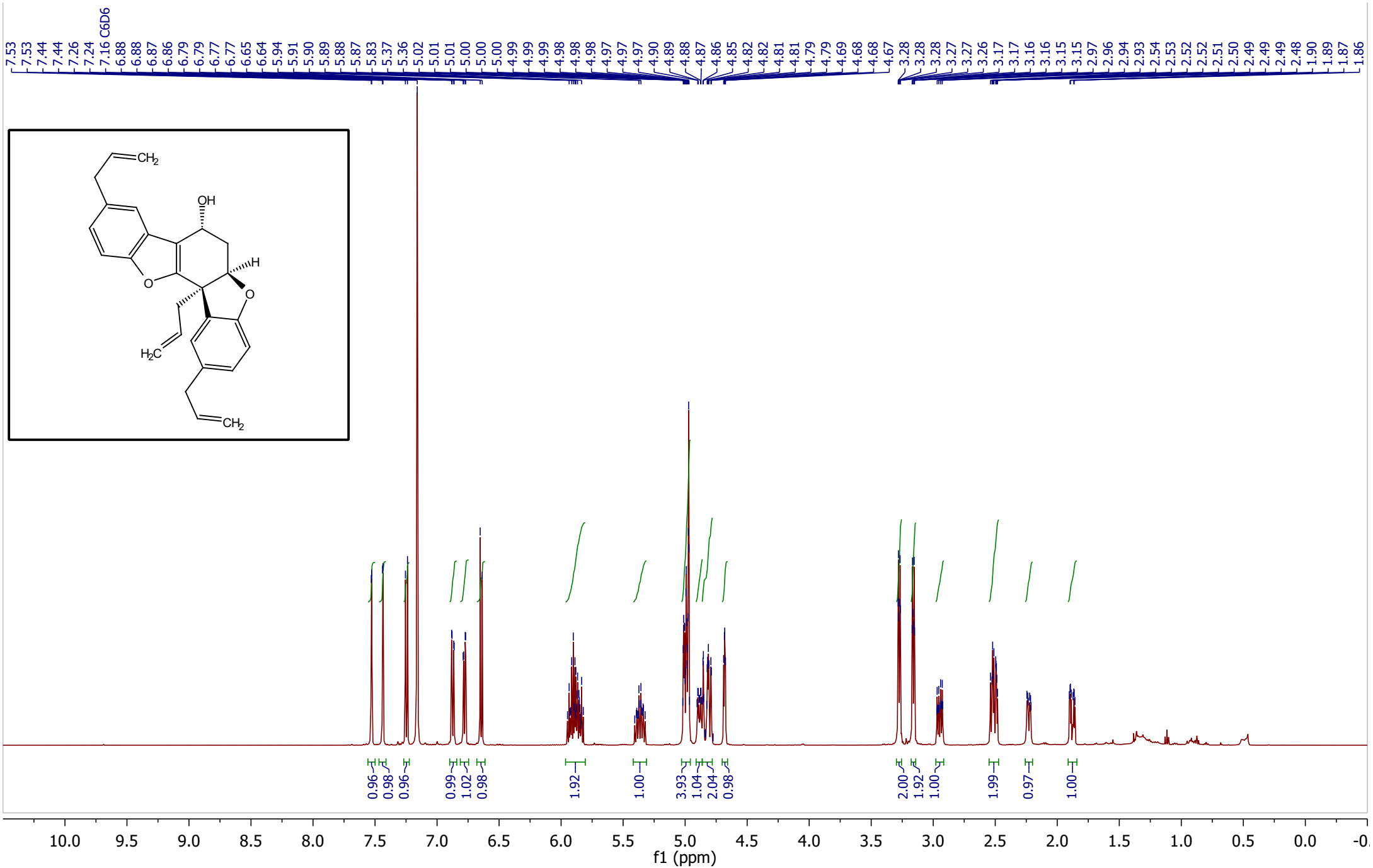


^{1}H NMR (500 MHz, Benzene-d_6) δ 7.56 (d, J = 1.8 Hz, 1H), 7.43 (d, J = 1.8 Hz, 1H), 7.29 (d, J = 8.4 Hz, 1H), 6.89 (dd, J = 8.4, 1.8 Hz, 1H), 6.82 (dd, J = 8.2, 1.8 Hz, 1H), 6.78 (d, J = 8.2 Hz, 1H), 5.93 (dddd, J = 16.8, 10.1, 6.6, 6.6 Hz, 1H), 5.84 (dddd, J = 17.0, 10.3, 6.6, 6.6 Hz, 1H), 5.48 (dddd, J = 16.7, 10.1, 8.2, 6.4 Hz, 1H), 5.04 – 4.94 (m, 5H), 4.91 – 4.80 (m, 3H), 3.29 (ddd, J = 6.6, 1.3, 1.3 Hz, 2H), 3.14 (ddd, J = 6.6, 1.3, 1.3 Hz, 2H), 3.05 (dddd, J = 14.1, 6.4, 1,4, 1.4 Hz, 1H), 2.60 (dd, J = 14.1, 8.2 Hz, 1H), 2.47 (ddd, J = 13.8, 5.0, 5.0 Hz, 1H), 1.79 (ddd, J = 13.8, 8.4, 3.4 Hz, 1H), 1.22 – 1.11 (m, 1H).

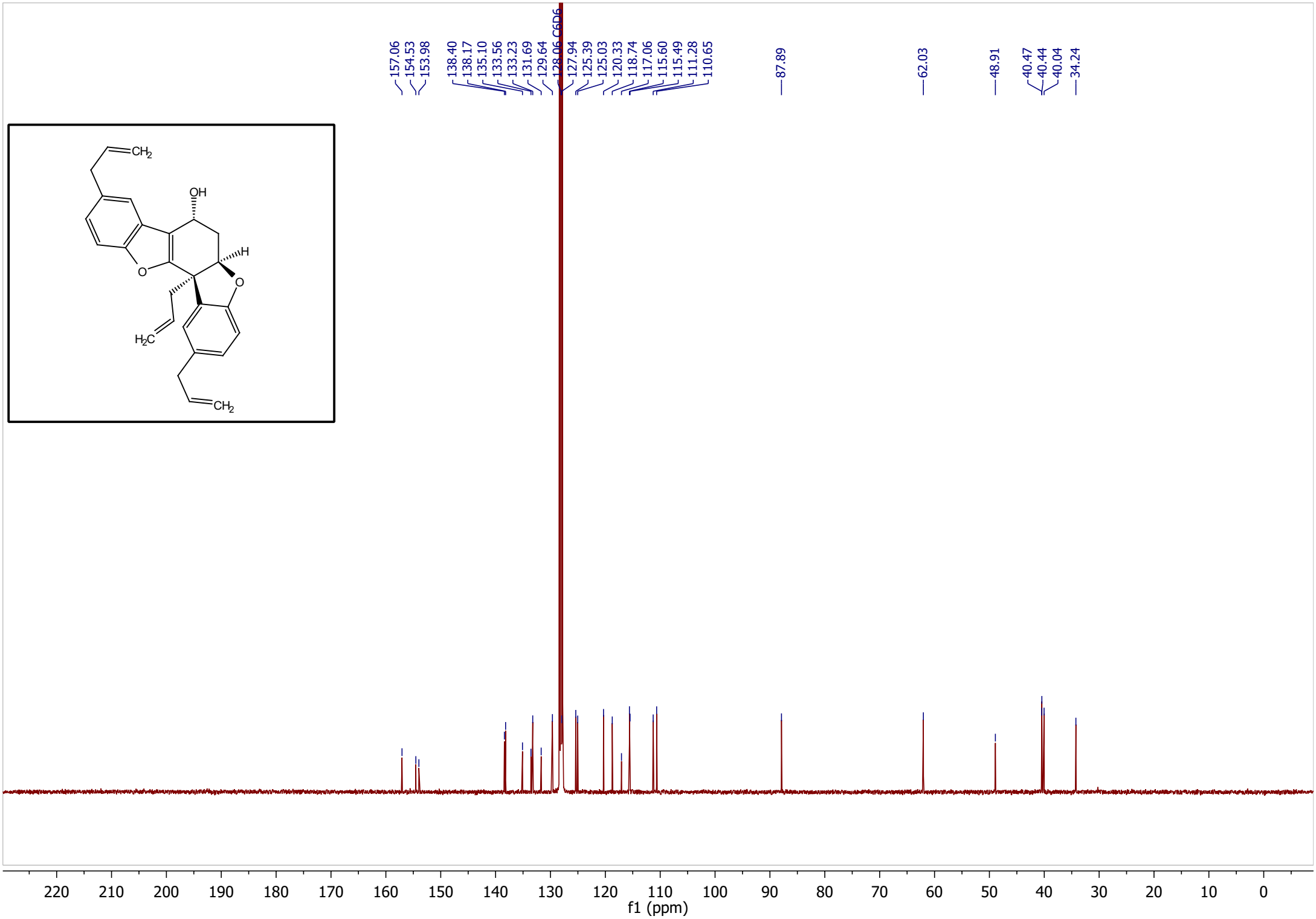

157.96
154.51
153.70
138.51
138.26
134.80
133.55
133.01
131.52
129.58
128.06 C6D6
127.63
125.12
124.94
121.07
118.69
117.69
115.49
115.43
111.27
110.35
86.29
62.58
48.98
41.22
40.50
40.01
36.51
OH
H
O
O
H_2C
CH_2
CH_2
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

NOESY

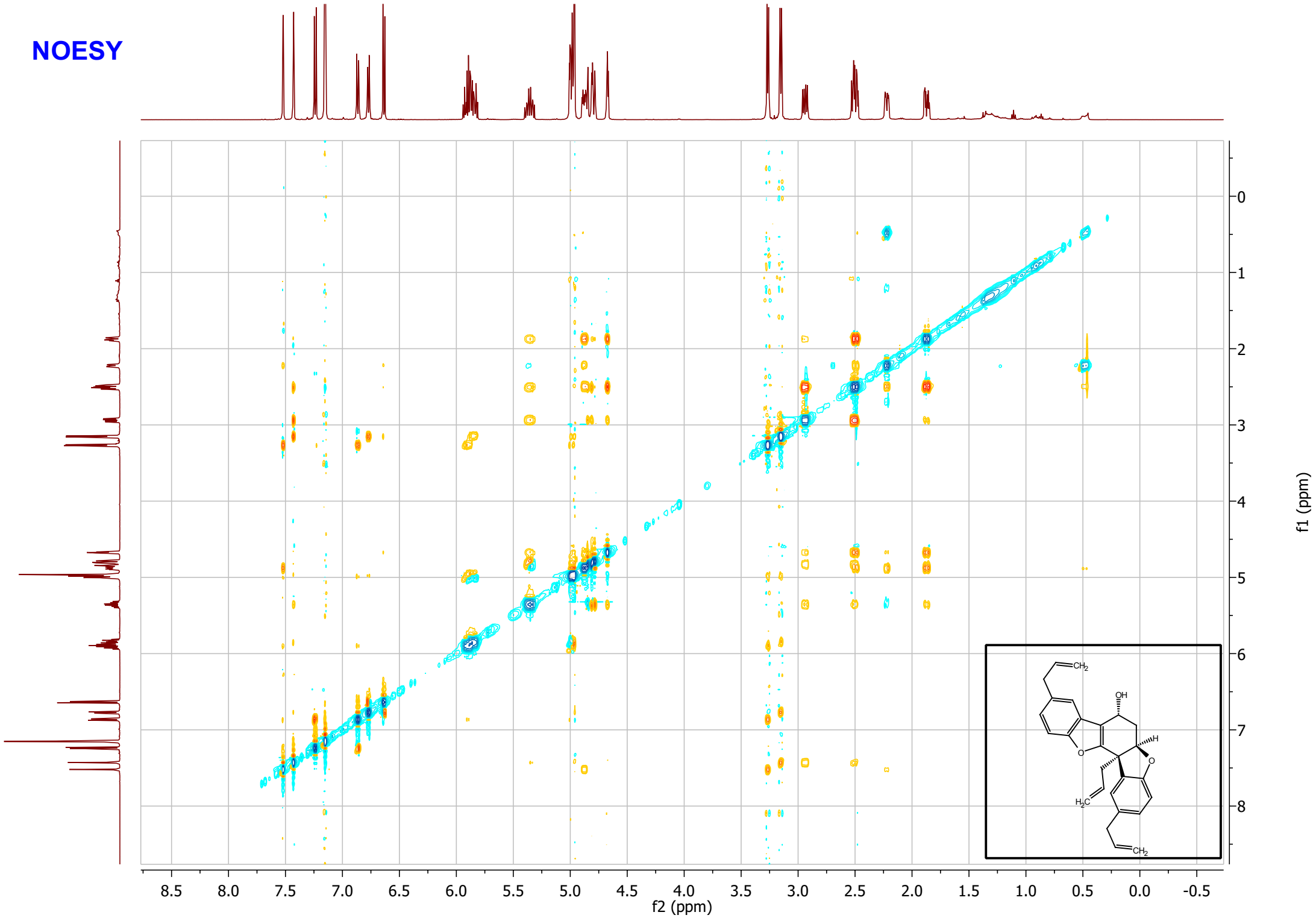

8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f2 (ppm)
-1
0
1
2
3
4
5
6
7
8
f1 (ppm)
CH2
OH
H
O
O
H2C
CH2



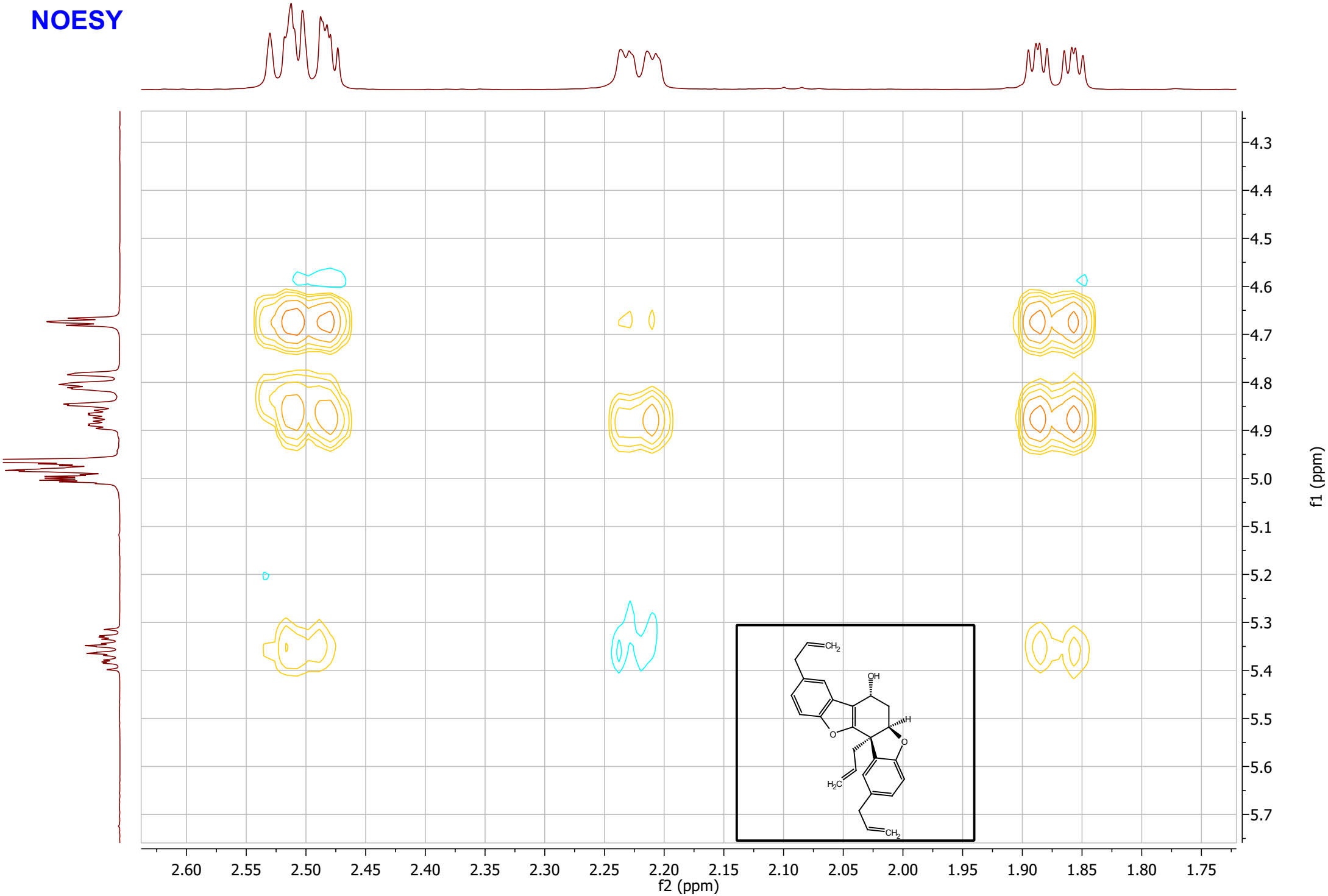
^{1}H NMR (500 MHz, Benzene-d_6) δ 7.53 (d, J = 1.8 Hz, 1H), 7.44 (d, J = 1.9 Hz, 1H), 7.25 (d, J = 8.4 Hz, 1H), 6.87 (dd, J = 8.4, 1.8 Hz, 1H), 6.78 (dd, J = 8.1, 1.9 Hz, 1H), 6.64 (d, J = 8.1 Hz, 1H), 5.96 – 5.82 (m, 2H), 5.36 (dddd, J = 16.8, 10.1, 8.1, 6.6 Hz, 1H), 5.03 – 4.95 (m, 4H), 4.89 (ddd, J = 11.2, 4.7, 3.0 Hz, 1H), 4.86 – 4.77 (m, 2H), 4.68 (dd, J = 4.4, 3.0 Hz, 1H), 3.27 (ddd, J = 6.3, 1.0, 1.0 Hz, 2H), 3.16 (ddd, J = 6.9, 1.5, 1.5 Hz, 1H), 2.95 (dddd, J = 14.1, 6.6, 1.4, 1.4 Hz, 1H), 2.56 – 2.47 (m, 2H), 2.23 (ddd, J = 11.2, 2.7, 2.7 Hz, 1H), 1.88 (ddd, J = 14.9, 4.7, 3.0 Hz,

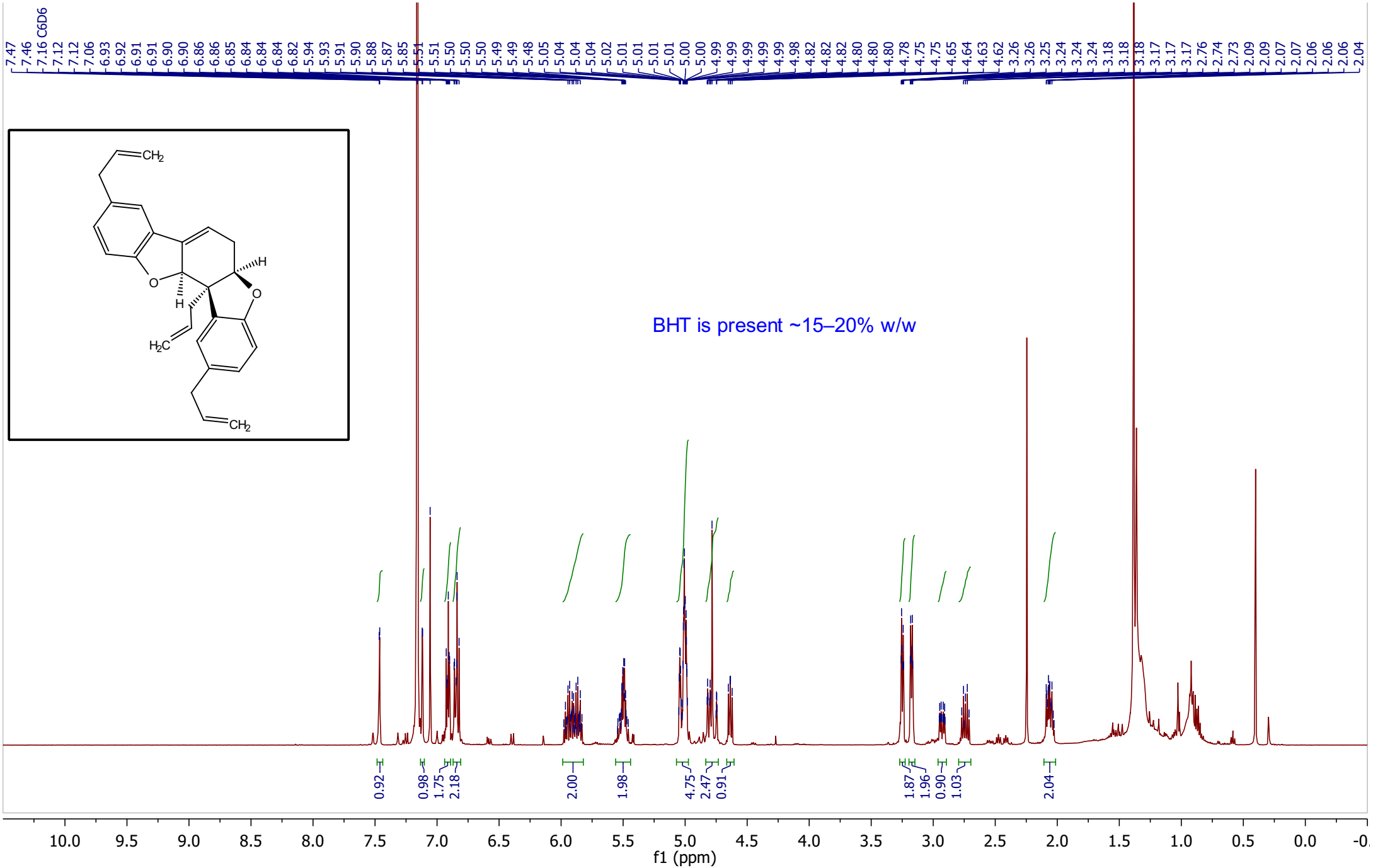

157.06
154.53
153.98
138.40
138.17
135.10
133.56
133.23
131.69
129.64
128.06 C6D6
127.94
125.39
125.03
120.33
118.74
117.06
115.60
115.49
111.28
110.65
87.89
62.03
48.91
40.47
40.44
40.04
34.24
OH
H
O
O
CH2
H2C
CH2
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

NOESY

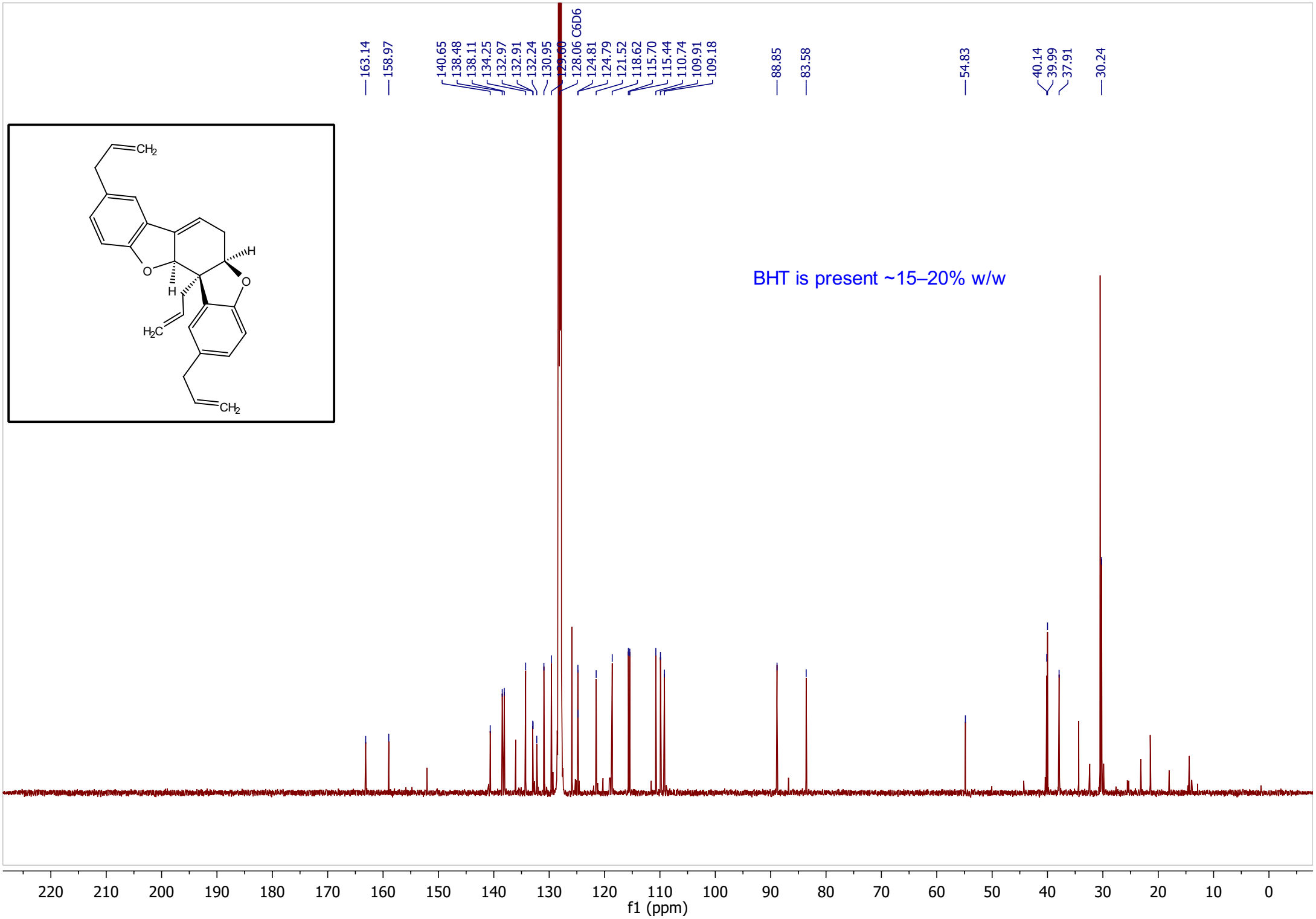

f2 (ppm)
f1 (ppm)
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
0
1
2
3
4
5
6
7
8
CH2
OH
H
O
O
H2C
CH2

NOESY

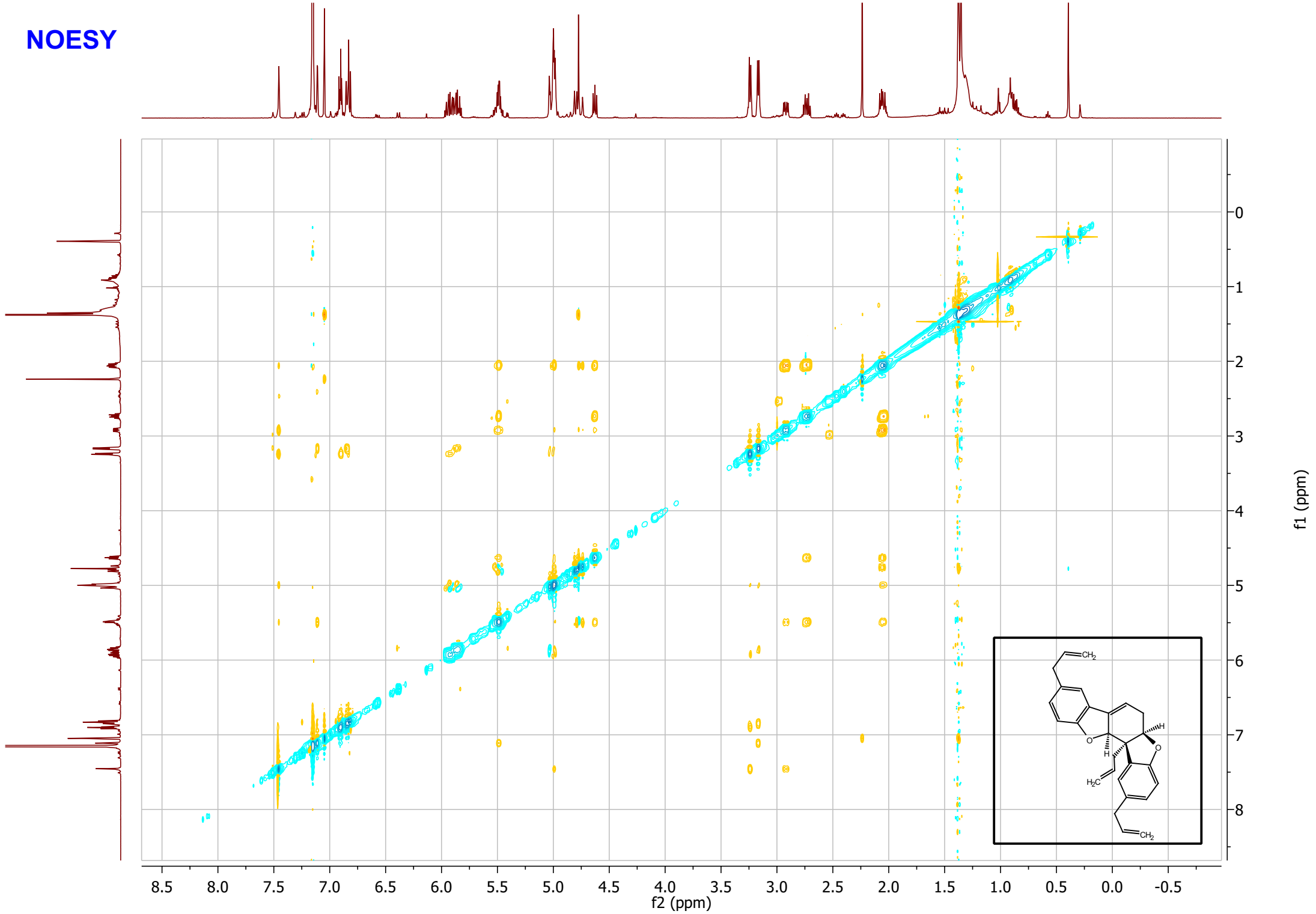

4.3
4.4
4.5
4.6
4.7
4.8
4.9
5.0
5.1
5.2
5.3
5.4
5.5
5.6
5.7
f1 (ppm)
2.60
2.55
2.50
2.45
2.40
2.35
2.30
2.25
2.20
2.15
2.10
2.05
2.00
1.95
1.90
1.85
1.80
1.75
f2 (ppm)
CH2
OH
H
O
O
H2C
CH2



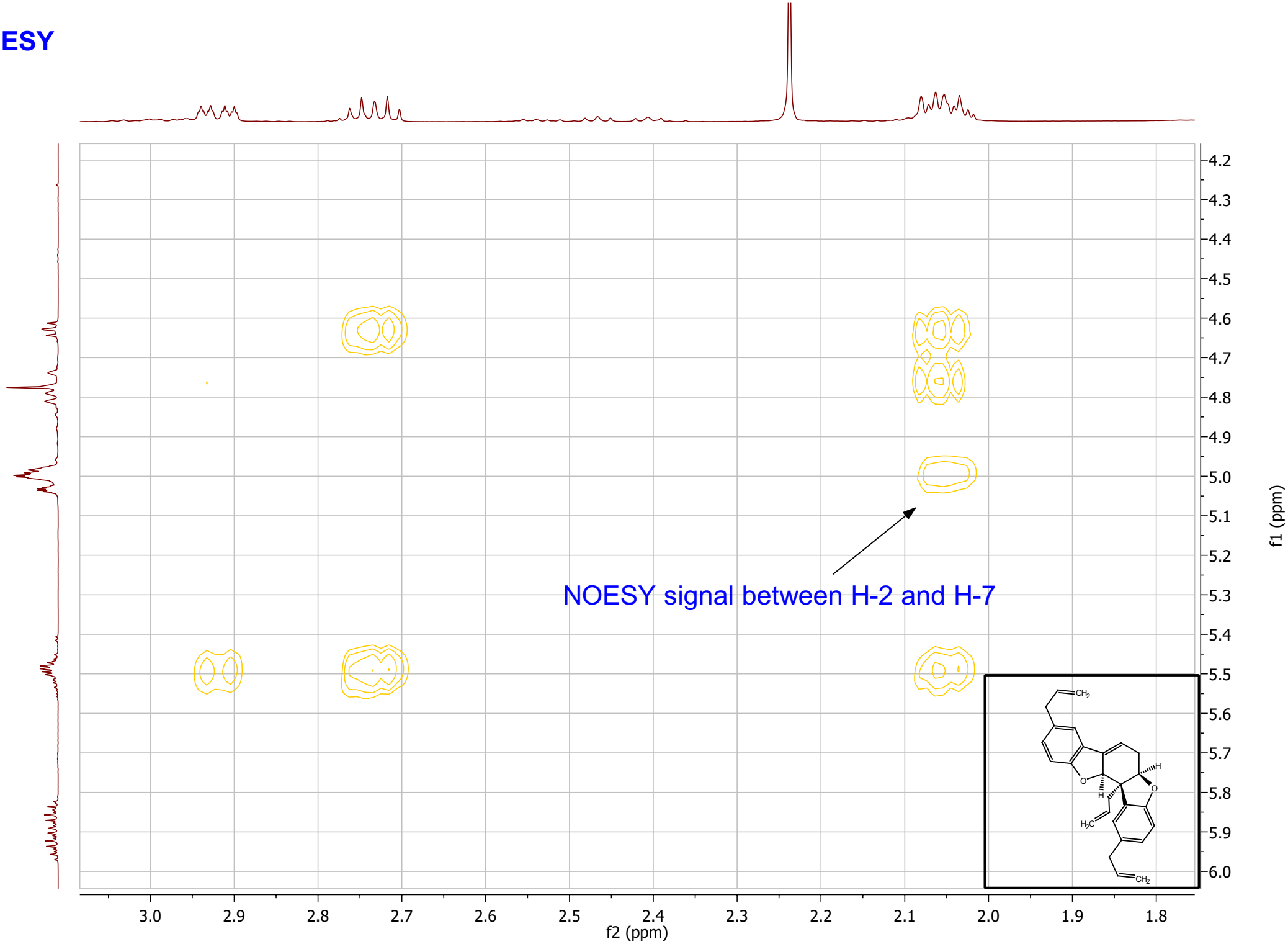
^{1}H NMR (500 MHz, Benzene-d_6) δ 7.46 (d, J = 1.9 Hz, 1H), 7.12 (d, J = 1.9 Hz, 1H), 6.92 (d, J = 8.3 Hz, 1H), 6.91 (dd, J = 8.3, 1.9 Hz, 1H), 6.85 (dd, J = 8.1, 1.9 Hz, 1H), 6.83 (d, J = 8.1 Hz, 1H), 5.94 (ddd, J = 16.8, 10.1, 6.6 Hz, 1H), 5.87 (ddd, J = 16.8, 10.1, 6.6 Hz, 1H), 5.55 – 5.45 (m, 2H), 5.06 – 4.98 (m, 5H), 4.84 – 4.72 (m, 2H), 4.64 (dd, J = 8.5, 7.1 Hz, 1H), 3.25 (ddd, J = 6.6, 1.6 Hz, 2H), 3.18 (ddd, J = 6.6, 1.6 Hz, 2H), 2.93 (dddd, J = 14.2, 5.6, 1.7 Hz, 1H), 2.74 (ddd, J = 14.8, 7.1 Hz, 1H), 2.10 – 2.02 (m, 2H).

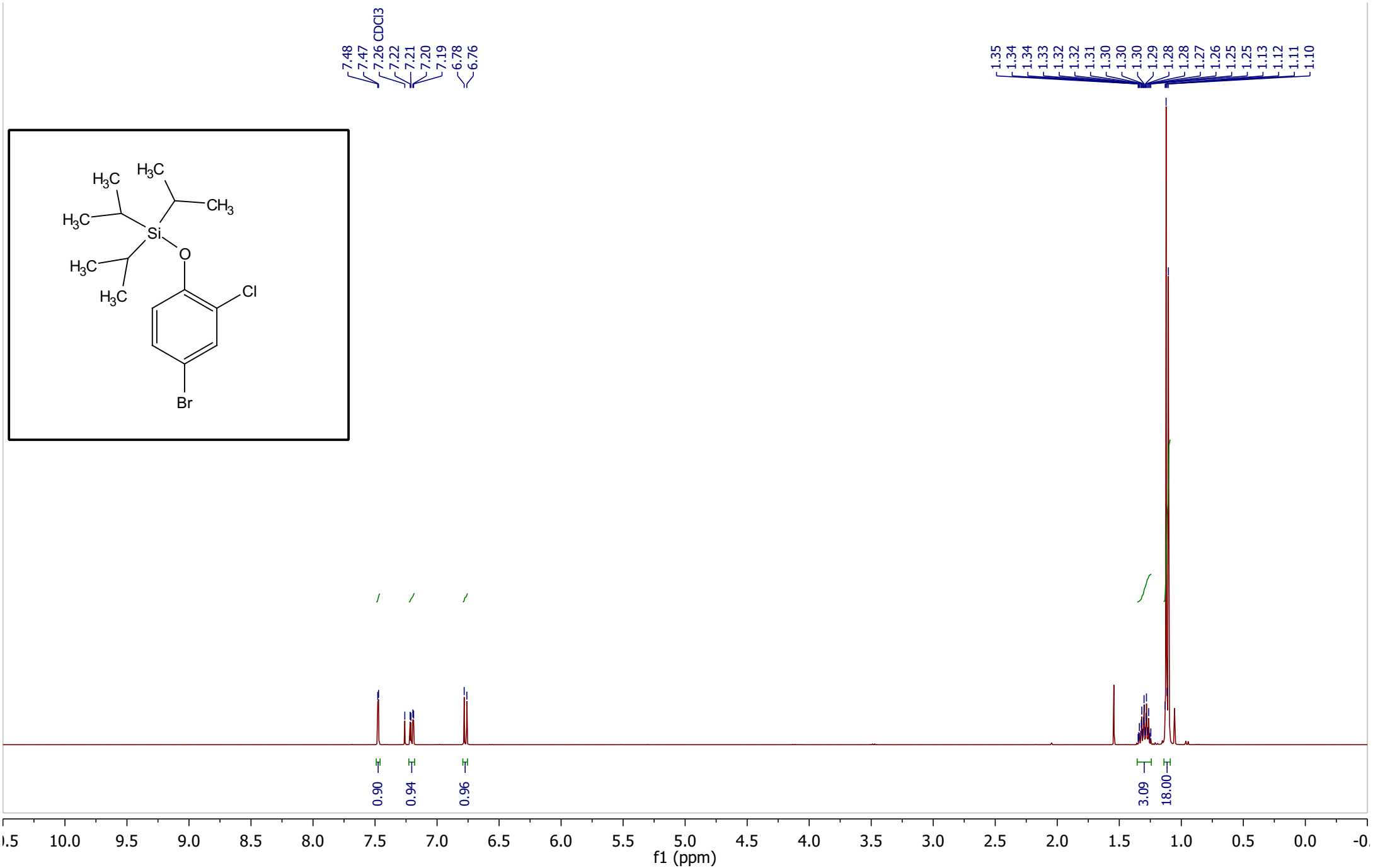

163.14
158.97
140.65
138.48
138.11
134.25
132.97
132.91
132.24
130.95
129.60
128.06 C6D6
124.81
124.79
121.52
118.62
115.70
115.44
110.74
109.91
109.18
88.85
83.58
54.83
40.14
39.99
37.91
30.24
BHT is present ~15–20% w/w
CH2
H
O
O
H
H2C
CH2
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

NOESY

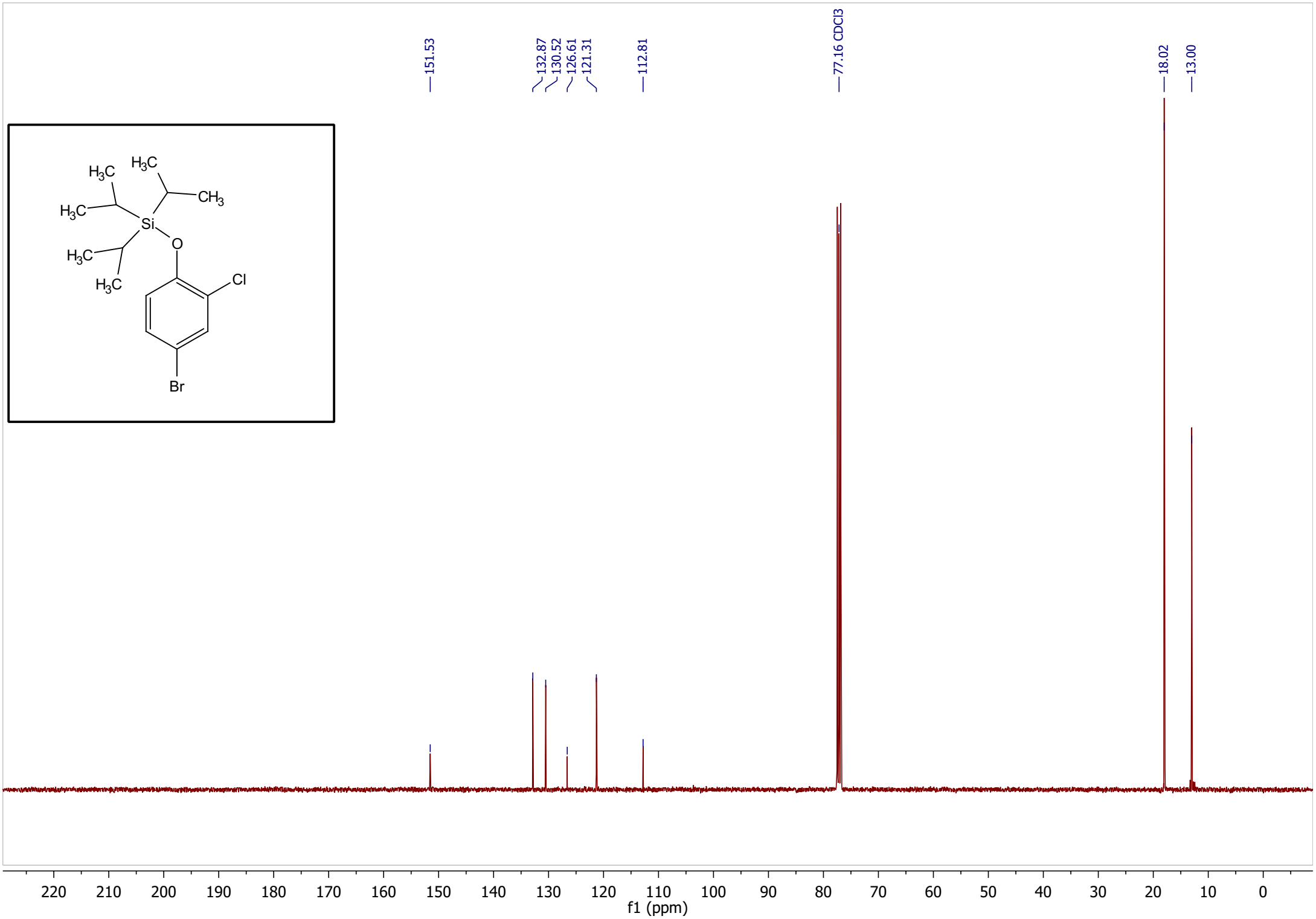

8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f2 (ppm)
0
1
2
3
4
5
6
7
8
f1 (ppm)
CH2
O
H
O
H2C
CH2

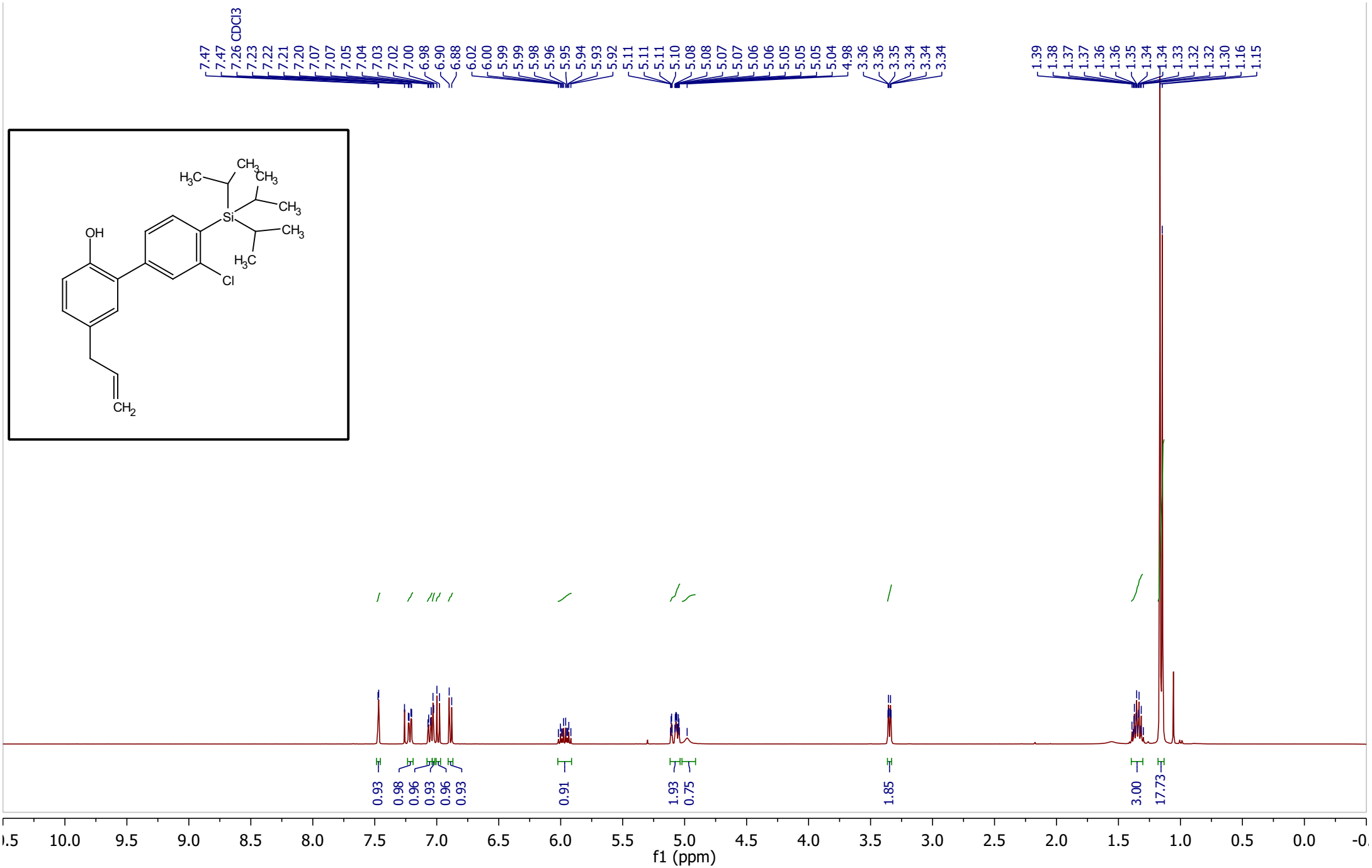
NOESY


NOESY signal between H-2 and H-7
f2 (ppm)
f1 (ppm)
3.0
2.9
2.8
2.7
2.6
2.5
2.4
2.3
2.2
2.1
2.0
1.9
1.8
4.2
4.3
4.4
4.5
4.6
4.7
4.8
4.9
5.0
5.1
5.2
5.3
5.4
5.5
5.6
5.7
5.8
5.9
6.0
CH2
O
H
H2C
O
CH2

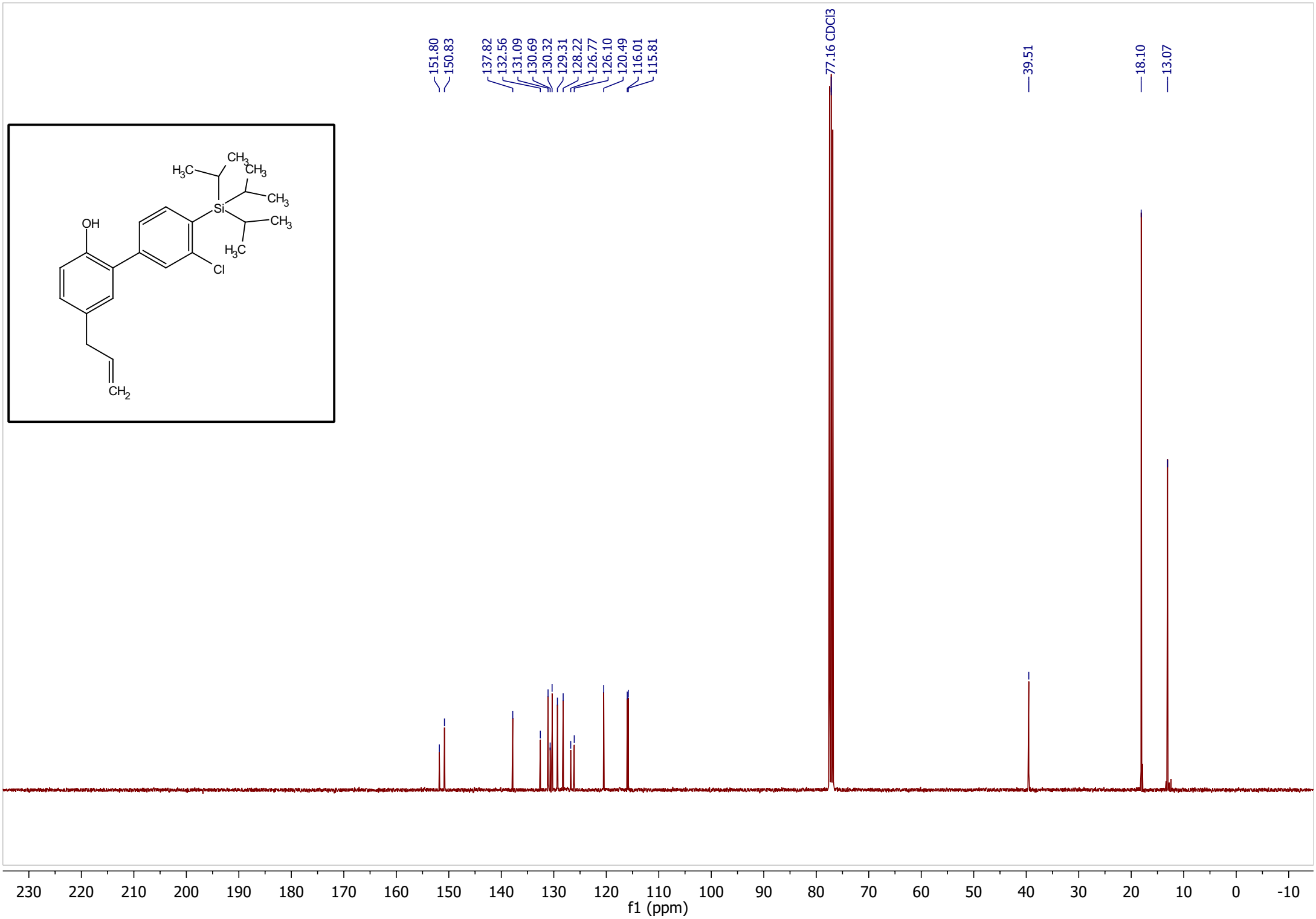


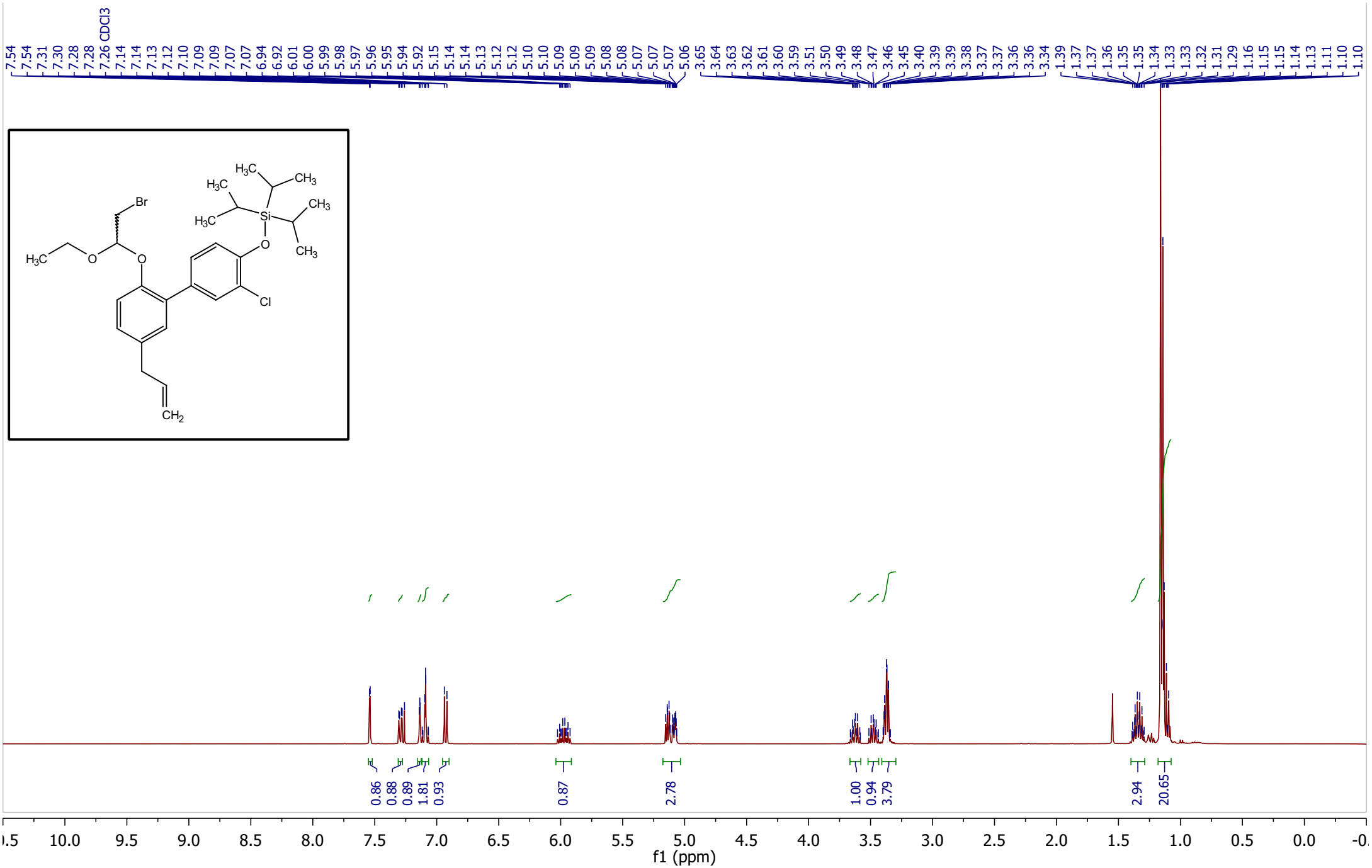
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.47 (d, *J* = 2.5 Hz, 1H), 7.20 (dd, *J* = 8.7, 2.5 Hz, 1H), 6.77 (d, *J* = 8.7 Hz, 1H), 1.35 – 1.24 (m, 3H), 1.11 (d, *J* = 7.3 Hz, 18H).


151.53
132.87
130.52
126.61
121.31
112.81
77.16 CDCl3
18.02
13.00
H3C
H3C
CH3
H3C
Si
O
H3C
H3C
Cl
Br
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

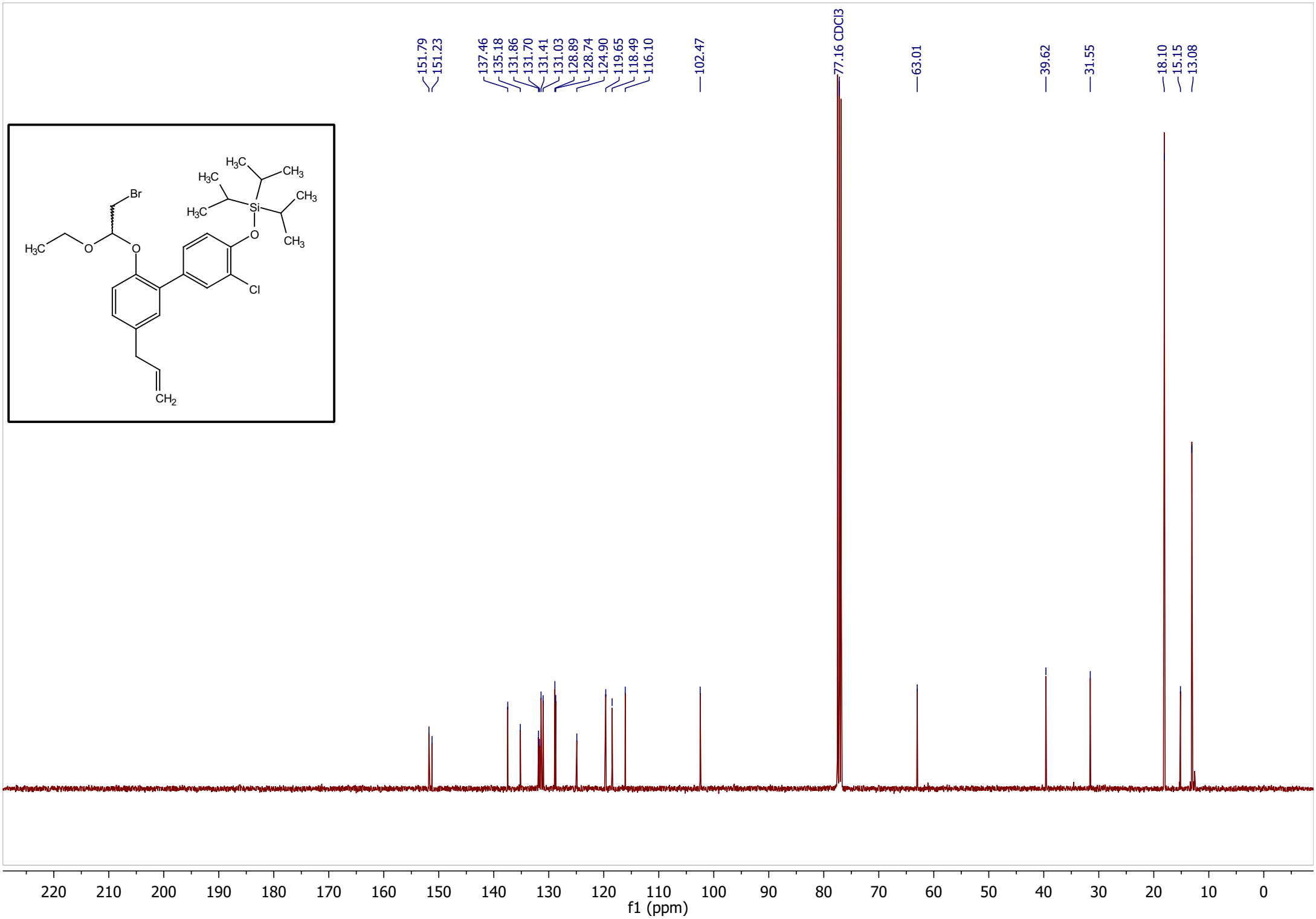


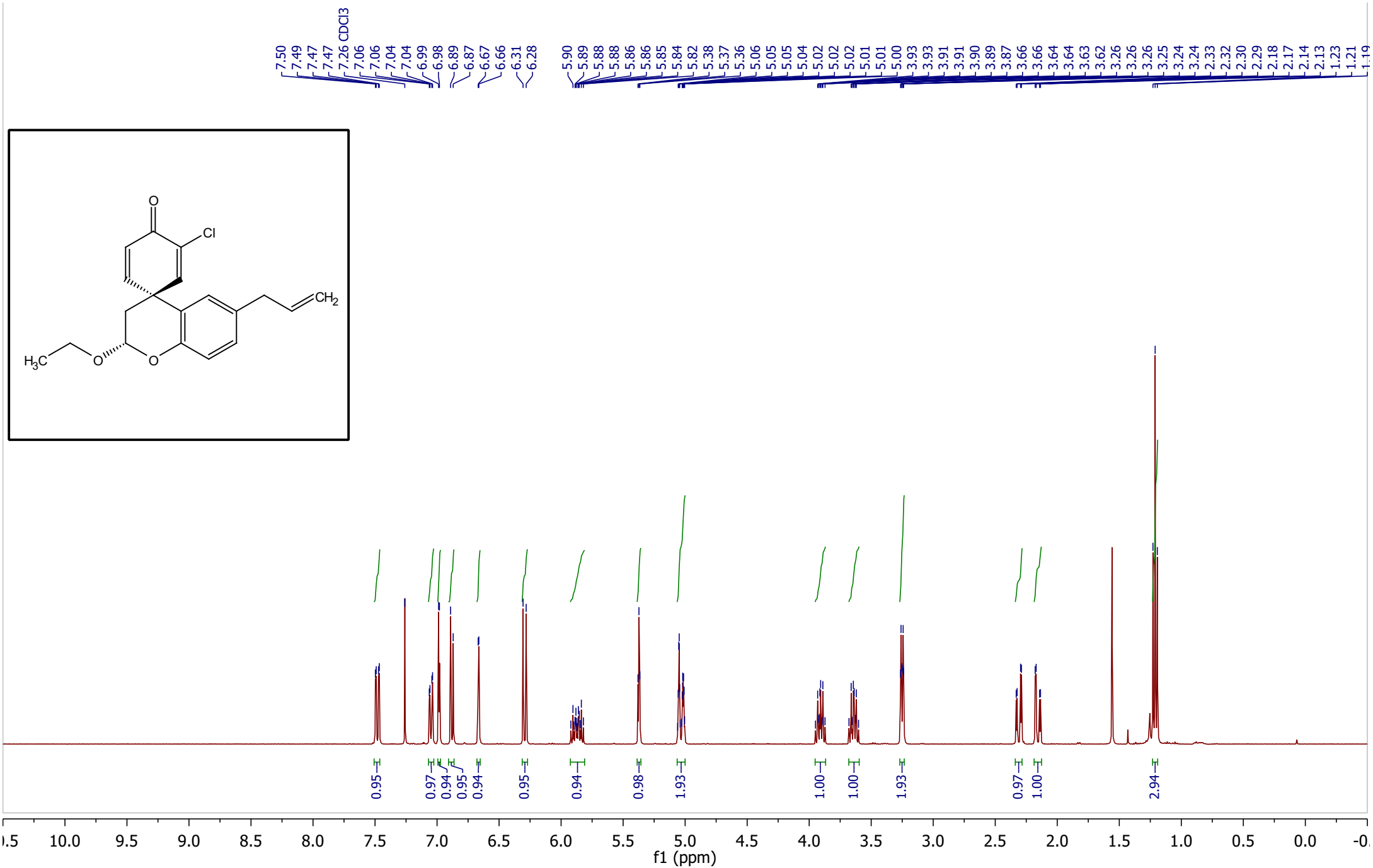
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.47 (d, *J* = 2.3 Hz, 1H), 7.21 (dd, *J* = 8.3, 2.3 Hz, 1H), 7.06 (dd, *J* = 8.1, 2.2 Hz, 1H), 7.03 (d, *J* = 2.2 Hz, 1H), 6.99 (d, *J* = 8.3 Hz, 1H), 6.89 (d, *J* = 8.1 Hz, 1H), 5.97 (ddt, *J* = 16.8, 10.0, 6.7 Hz, 1H), 5.12 – 5.03 (m, 2H), 4.98 (s, 1H), 3.35 (ddd, *J* = 6.8, 1.4, 1.4 Hz, 2H), 1.43 – 1.29 (m, 3H), 1.16 (d, *J* = 7.4 Hz, 18H).


151.80
150.83
137.82
132.56
131.09
130.69
130.32
129.31
128.22
126.77
126.10
120.49
116.01
115.81
77.16 CDCl3
39.51
18.10
13.07
OH
Si
Cl
CH2
f1 (ppm)

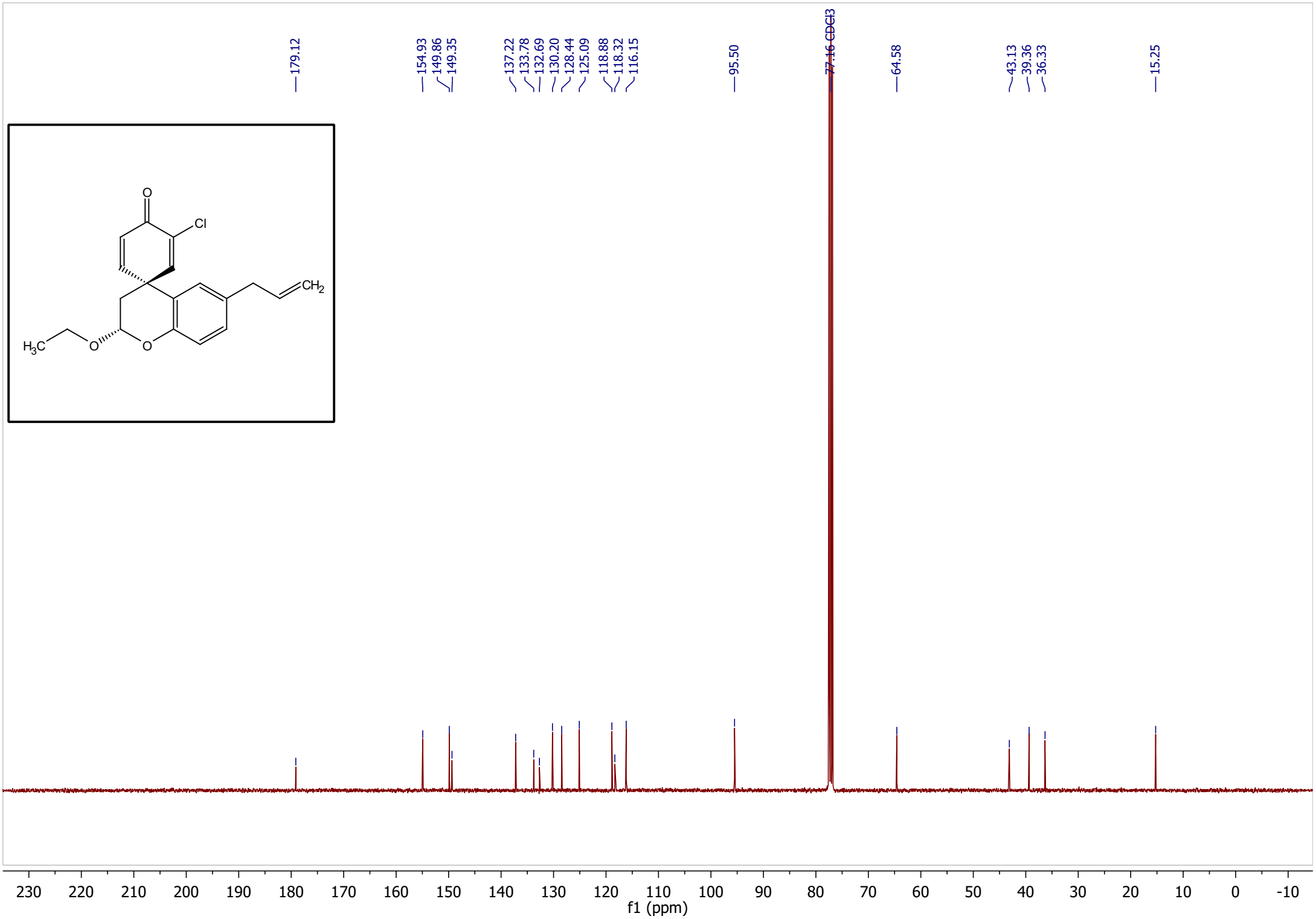


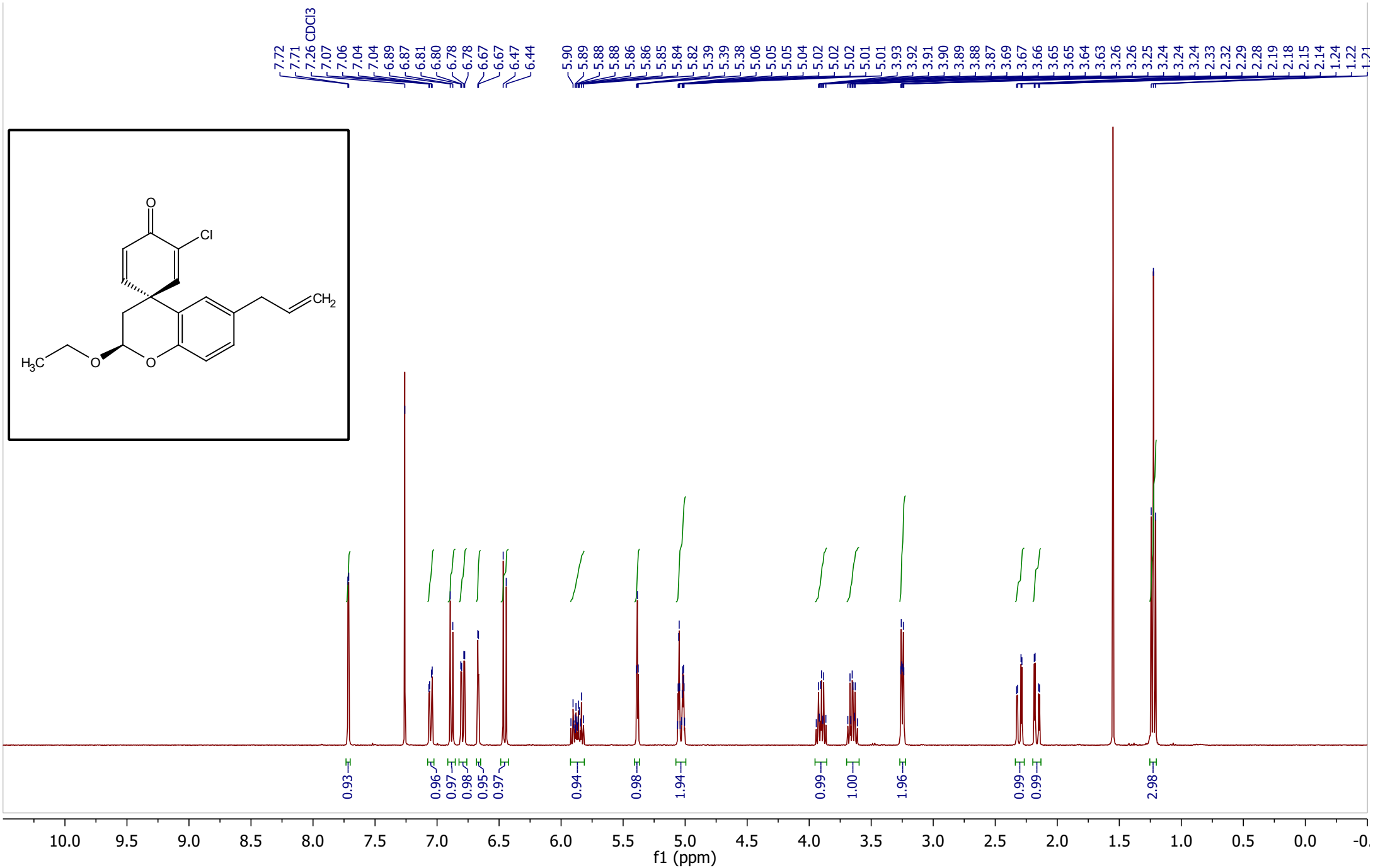
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.54 (d, *J* = 2.3 Hz, 1H), 7.29 (dd, *J* = 8.4, 2.3 Hz, 1H), 7.15 – 7.13 (m, 1H), 7.12 – 7.06 (m, 2H), 6.93 (d, *J* = 8.4 Hz, 1H), 5.97 (dddd, *J* = 16.8, 10.1, 6.7, 6.7 Hz, 1H), 5.16 – 5.06 (m, 3H), 3.63 (dq, *J* = 9.2, 7.1 Hz, 1H), 3.48 (dq, *J* = 9.2, 7.1 Hz, 1H), 3.40 – 3.34 (m, 4H), 1.41 – 1.28 (m, 3H), 1.15 (d, *J* = 7.4 Hz, 18H), 1.15 – 1.09 (m, 3H).


151.79
151.23
137.46
135.18
131.86
131.70
131.41
131.03
128.89
128.74
124.90
119.65
118.49
116.10
102.47
77.16 CDCl3
63.01
39.62
31.55
18.10
15.15
13.08
Br
H3C
O
O
Si
O
Cl
CH2
f1 (ppm)

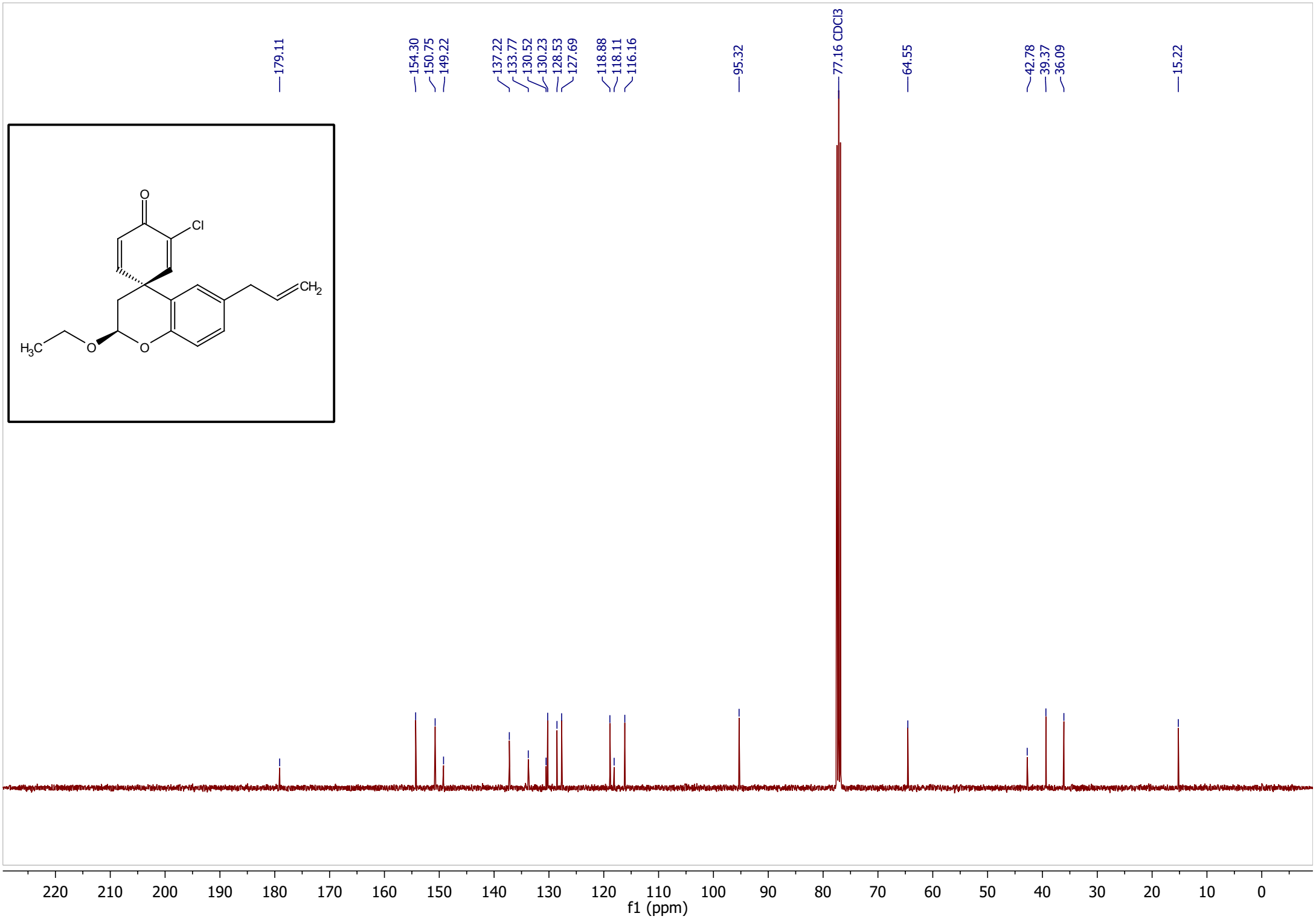


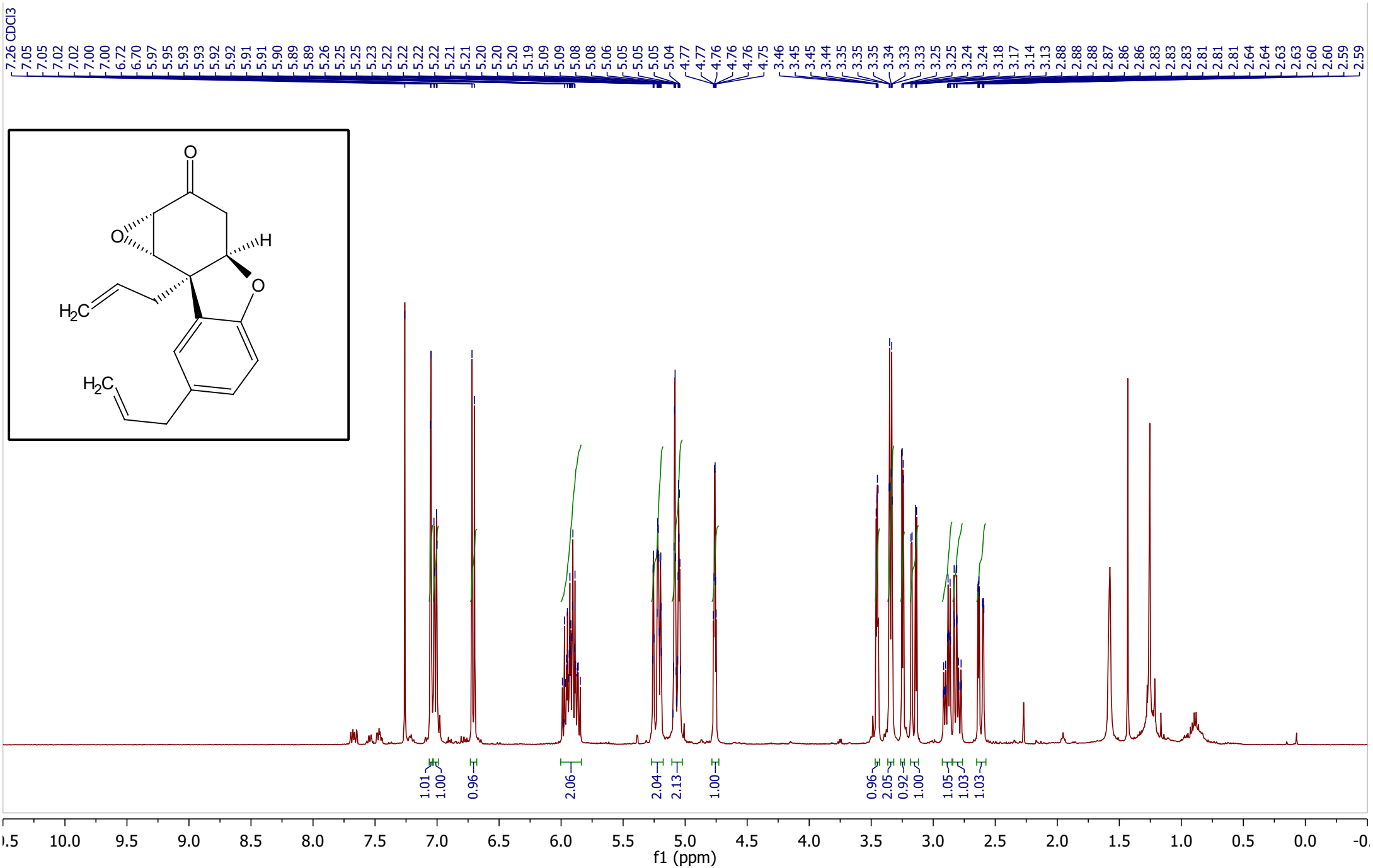
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.48 (dd, *J* = 10.0, 2.7 Hz, 1H), 7.05 (dd, *J* = 8.3, 2.2 Hz, 1H), 6.98 (d, *J* = 2.7 Hz, 1H), 6.88 (d, *J* = 8.3 Hz, 1H), 6.67 (d, *J* = 2.2 Hz, 1H), 6.29 (d, *J* = 10.0 Hz, 1H), 5.87 (dddd, *J* = 17.5, 9.6, 6.7, 6.7 Hz, 1H), 5.37 (dd, *J* = 3.0, 3.0 Hz, 1H), 5.06 – 5.00 (m, 2H), 3.91 (dq, *J* = 9.6, 7.1 Hz, 1H), 3.64 (dq, *J* = 9.6, 7.1 Hz, 1H), 3.25 (d, *J* = 6.8, 1.5 Hz, 2H), 2.31 (dd, *J* = 14.0, 3.0 Hz, 1H), 2.16 (dd, *J* = 14.0, 3.1 Hz, 1H), 1.21 (dd, *J* = 7.1, 7.1 Hz, 3H).


179.12
154.93
149.86
149.35
137.22
133.78
132.69
130.20
128.44
125.09
118.88
118.32
116.15
95.50
77.16 CDCl3
64.58
43.13
39.36
36.33
15.25
O
Cl
CH2
H3C
O
O
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

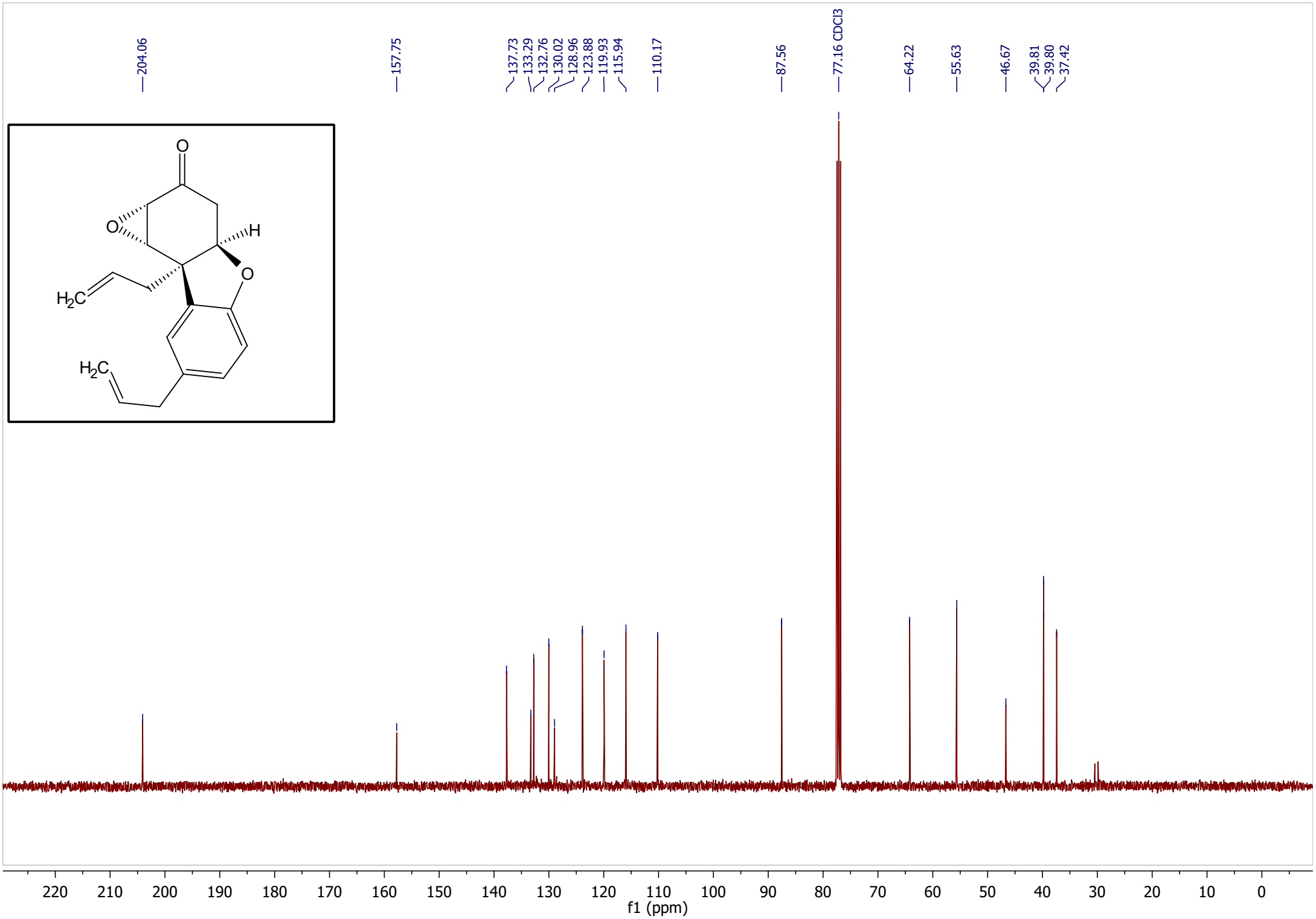


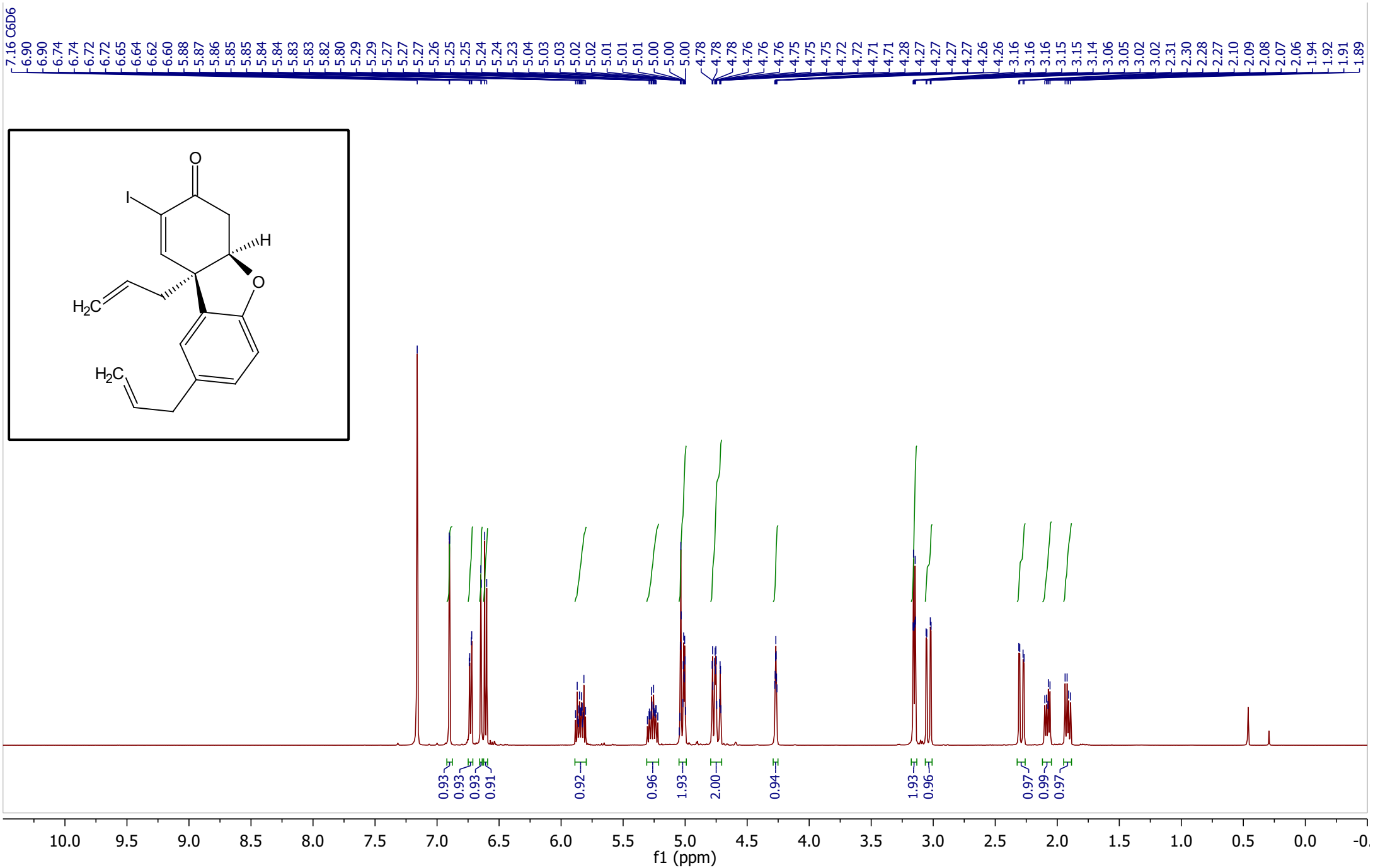
^{1}H NMR (400 MHz, Chloroform-*d*) δ7.72 (d, *J* = 2.7 Hz, 1H), 7.05 (dd, *J* = 8.3, 2.2 Hz, 1H), 6.88 (d, *J* = 8.3 Hz, 1H), 6.79 (dd, *J* = 9.8, 2.7 Hz, 1H), 6.67 (d, *J* = 2.2 Hz, 1H), 6.46 (d, *J* = 9.8 Hz, 1H), 5.87 (dddd, *J* = 17.5, 9.6, 6.7, 6.7 Hz, 1H), 5.39 (dd, *J* = 2.8, 2.8 Hz, 1H), 5.07 – 4.99 (m, 2H), 3.90 (dq, *J* = 9.7, 7.1 Hz, 1H), 3.65 (dq, *J* = 9.7, 7.1 Hz, 1H), 3.25 (ddd, *J* = 6.8, 1.5, 1.5 Hz, 2H), 2.30 (dd, *J* = 14.1, 2.9 Hz, 1H), 2.17 (dd, *J* = 14.1, 2.6 Hz, 1H), 1.22 (dd, *J* = 7.1, 7.1 Hz, 3H).


179.11
154.30
150.75
149.22
137.22
133.77
130.52
130.23
128.53
127.69
118.88
118.11
116.16
95.32
77.16 CDCl3
64.55
42.78
39.37
36.09
15.22
O
Cl
CH2
H3C
O
O
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

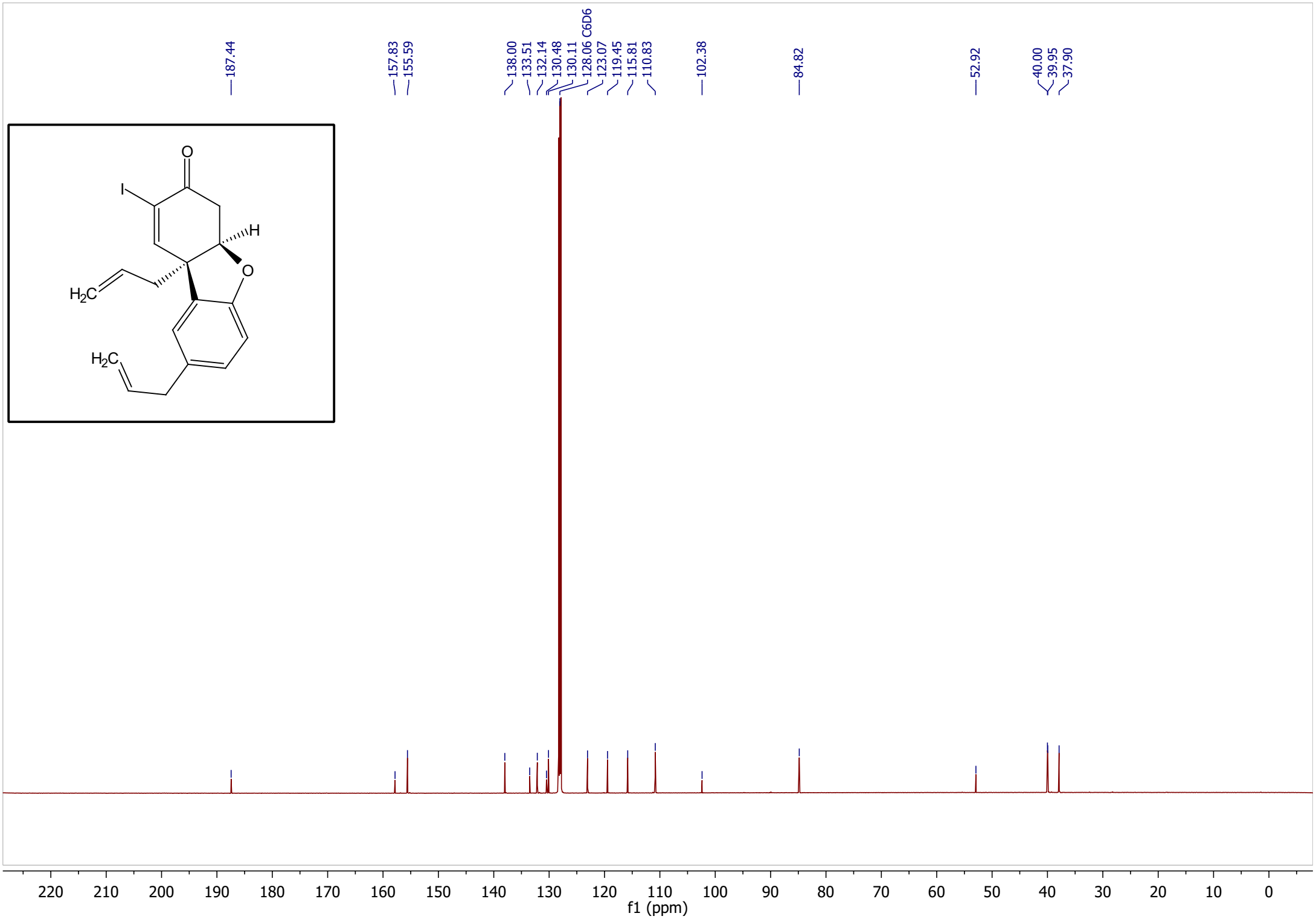


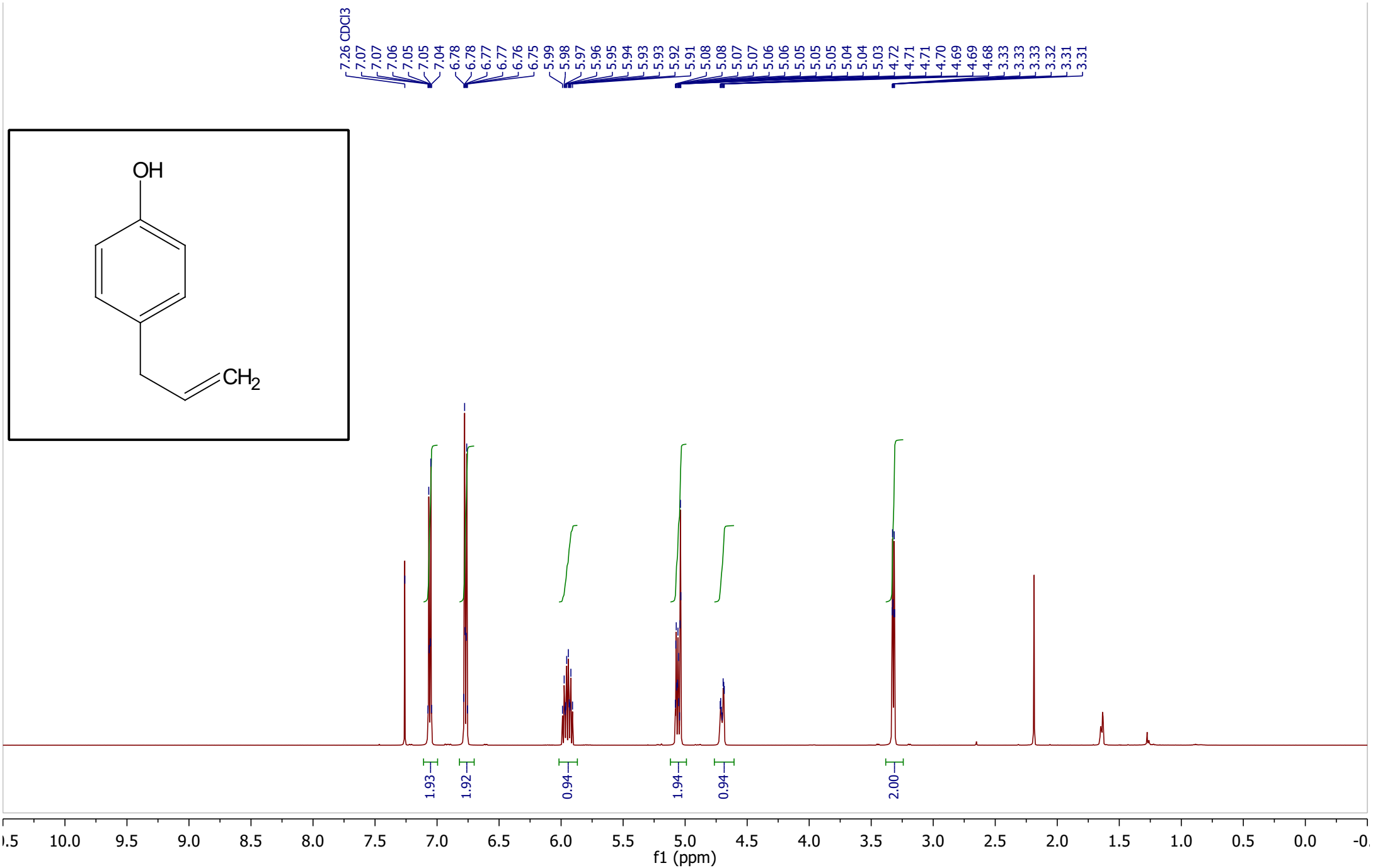
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.05 (d, *J* = 1.9 Hz, 1H), 7.01 (dd, *J* = 8.2, 1.9 Hz, 1H), 6.71 (d, *J* = 8.2 Hz, 1H), 6.00 – 5.83 (m, 2H), 5.28 – 5.18 (m, 2H), 5.10 – 5.03 (m, 2H), 4.79 – 4.71 (m, 1H), 3.45 (dd, *J* = 4.1, 2.1 Hz, 1H), 3.34 (ddd, *J* = 6.7, 1.5, 1.5 Hz, 2H), 3.25 (dd, *J* = 4.1, 1.0 Hz, 1H), 3.16 (dd, *J* = 14.6, 3.4 Hz, 1H), 2.89 (dddd, *J* = 14.2, 6.5, 1.4, 1.4 Hz, 1H), 2.80 (dddd, *J* = 14.2, 8.2, 1.0, 1.0 Hz, 1H), 2.62 (ddd, *J* = 14.6, 3.4, 1.1 Hz, 1H).


204.06
157.75
137.73
133.29
132.76
130.02
128.96
123.88
119.93
115.94
110.17
87.56
77.16 CDCl3
64.22
55.63
46.67
39.81
39.80
37.42
O
O
H
O
H2C
H2C
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

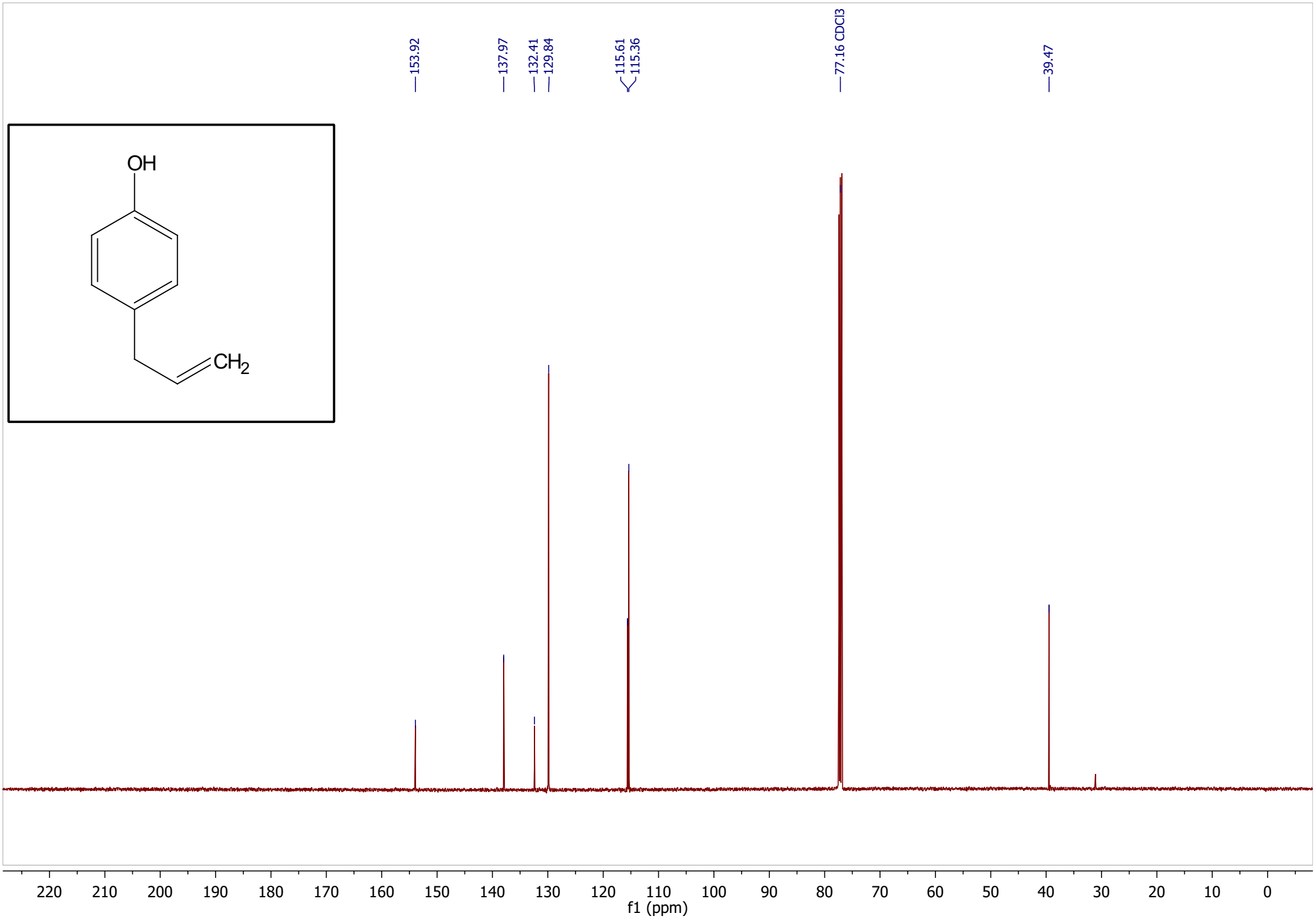


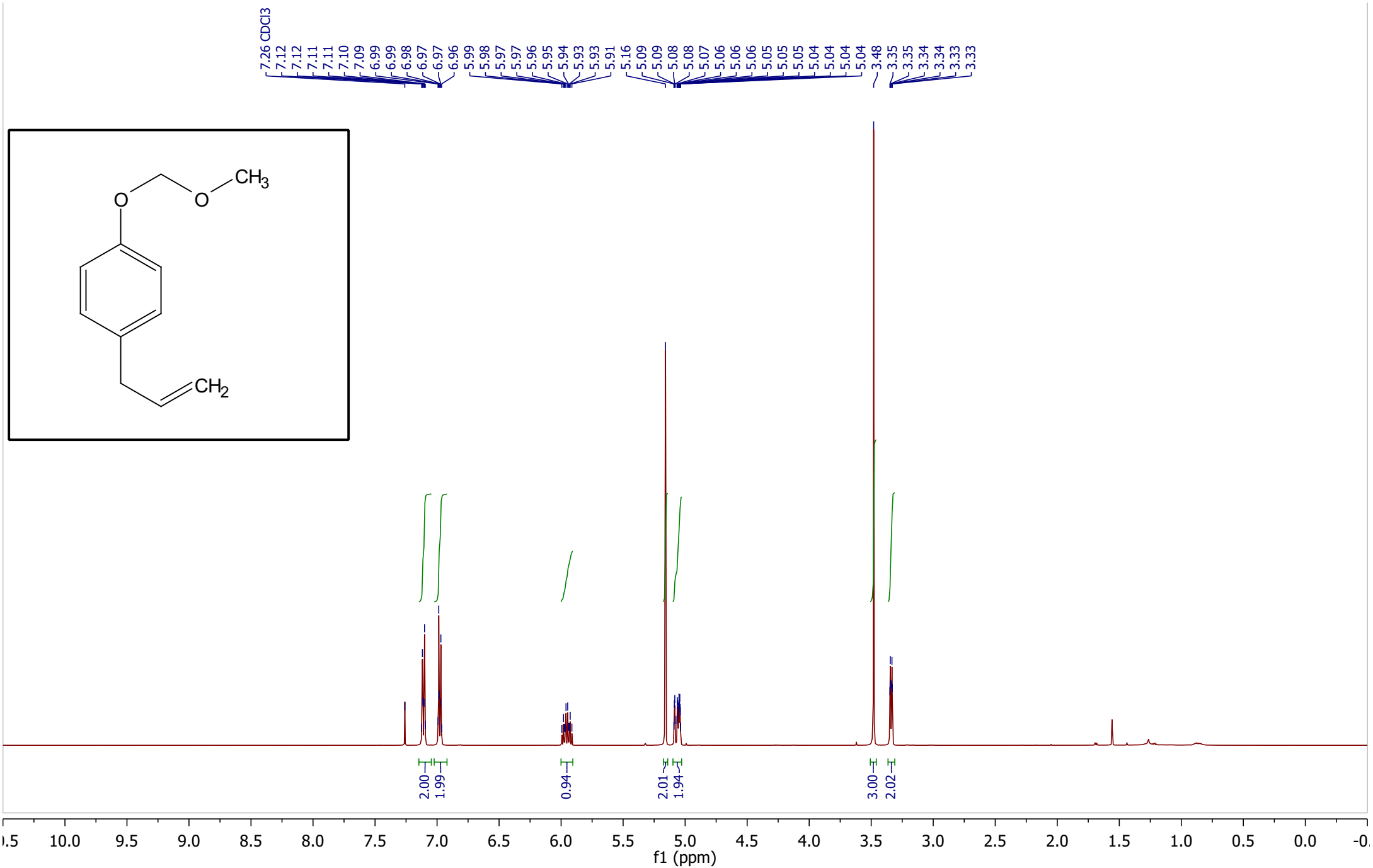
^{1}H NMR (500 MHz, Benzene-d_6) δ 6.90 (d, J = 1.7 Hz, 1H), 6.73 (dd, J = 8.2, 1.9 Hz, 1H), 6.65 (d, J = 1.9 Hz, 1H), 6.61 (d, J = 8.2 Hz, 1H), 5.89 – 5.80 (m, 1H), 5.26 (dddd, J = 16.9, 10.1, 8.0, 6.7 Hz, 1H), 5.05 – 4.99 (m, 2H), 4.80 – 4.69 (m, 2H), 4.27 (ddd, J = 4.0, 3.0, 1.7 Hz, 1H), 3.15 (ddd, J = 6.4, 1.4, 1.4 Hz, 2H), 3.04 (dd, J = 17.1, 3.0 Hz, 1H), 2.29 (dd, J = 17.1, 4.0 Hz, 1H), 2.08 (dd, J = 14.2, 6.7 Hz, 1H), 1.91 (dd, J = 14.2, 8.0 Hz, 1H).


187.44
157.83
155.59
138.00
133.51
132.14
130.48
130.11
128.06 C6D6
123.07
119.45
115.81
110.83
102.38
84.82
52.92
40.00
39.95
37.90
O
I
H
O
H2C
H2C
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

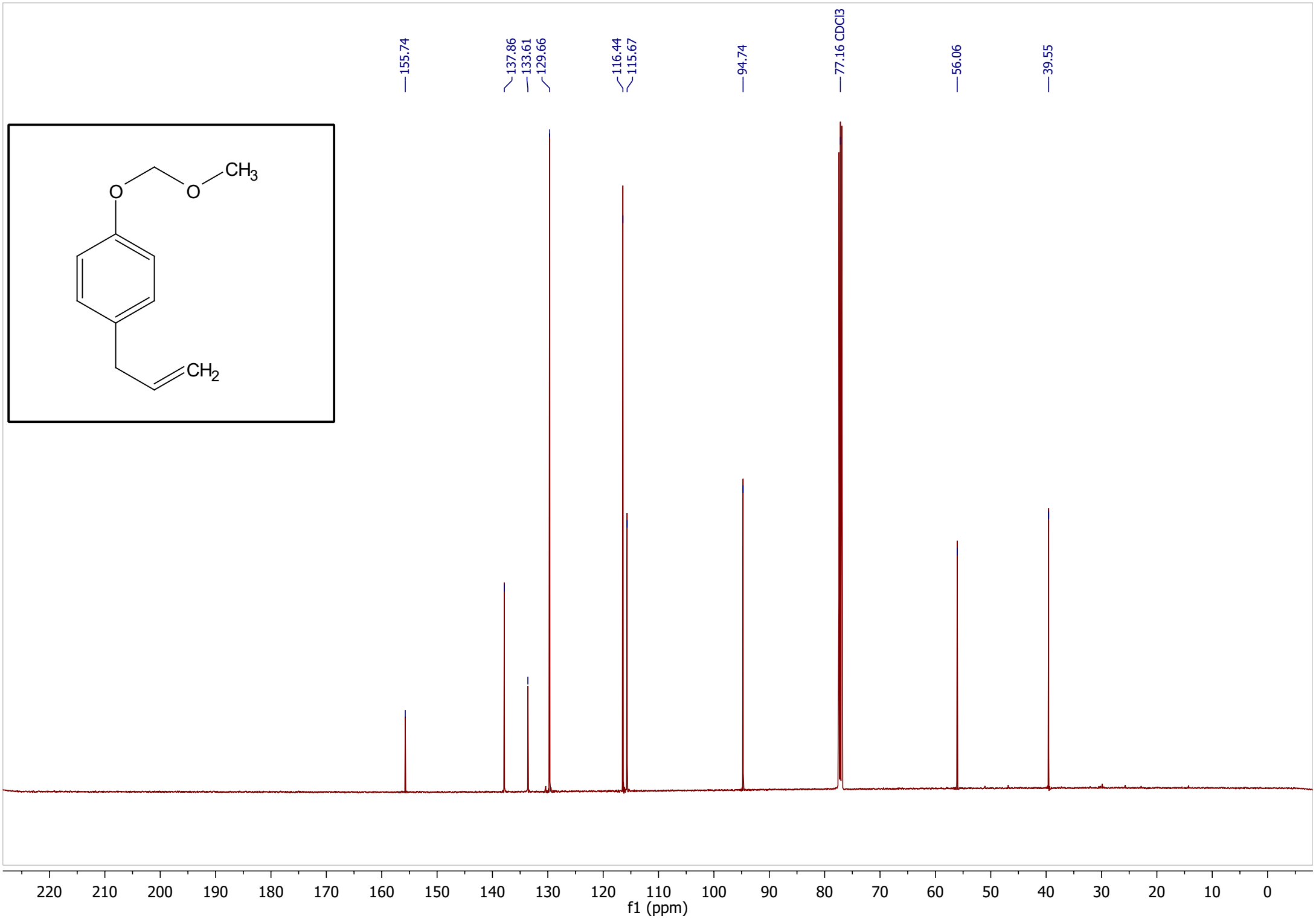


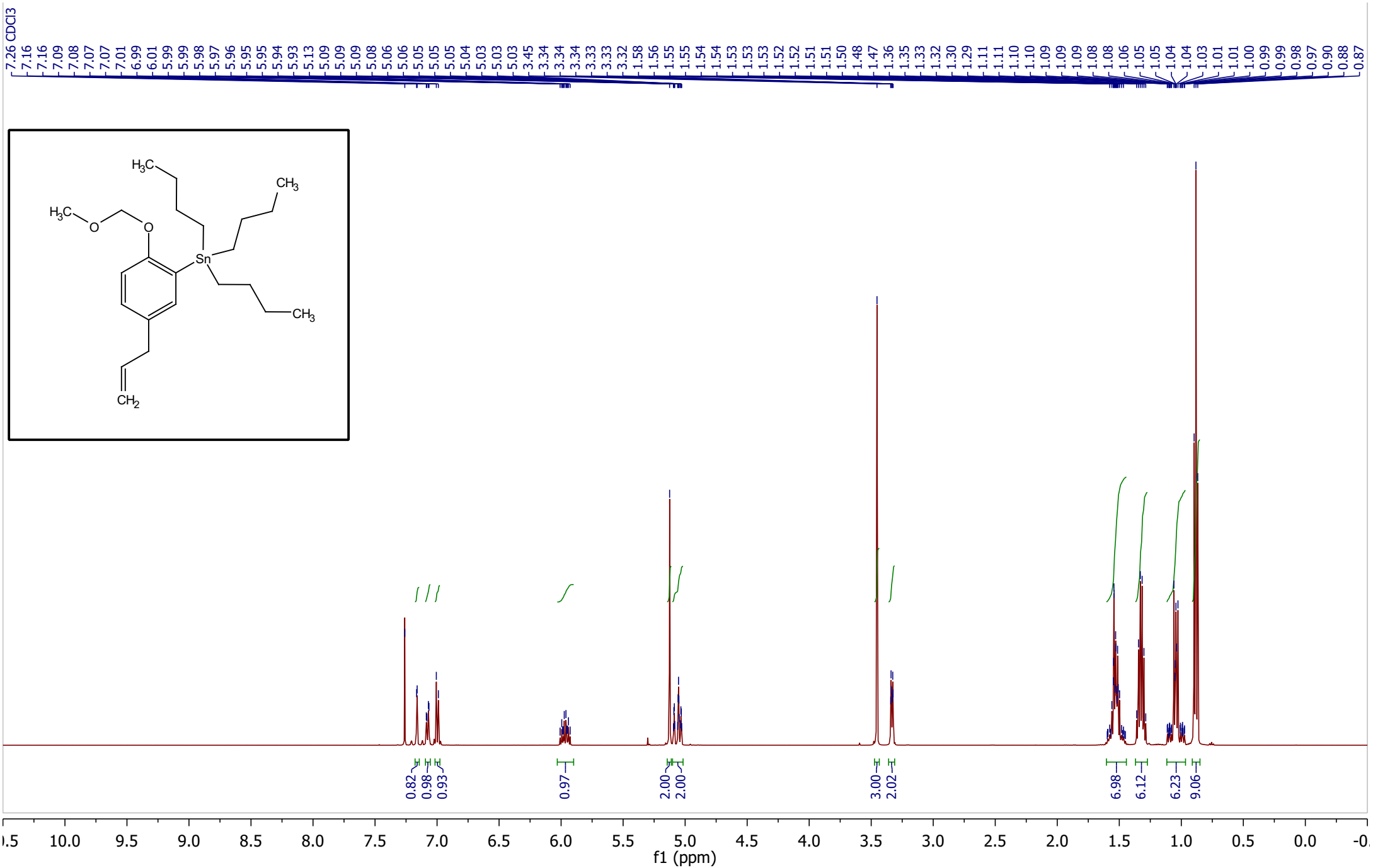
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.09 – 7.04 (m, 2H), 6.79 – 6.74 (m, 2H), 5.95 (ddt, *J* = 16.9, 10.2, 6.7 Hz, 1H), 5.10 – 5.01 (m, 2H), 4.74 – 4.67 (m, 1H), 3.32 (ddd, *J* = 6.7, 1.4, 1.4 Hz, 2H).


153.92
137.97
132.41
129.84
115.61
115.36
77.16 CDCl3
39.47
OH
CH2
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

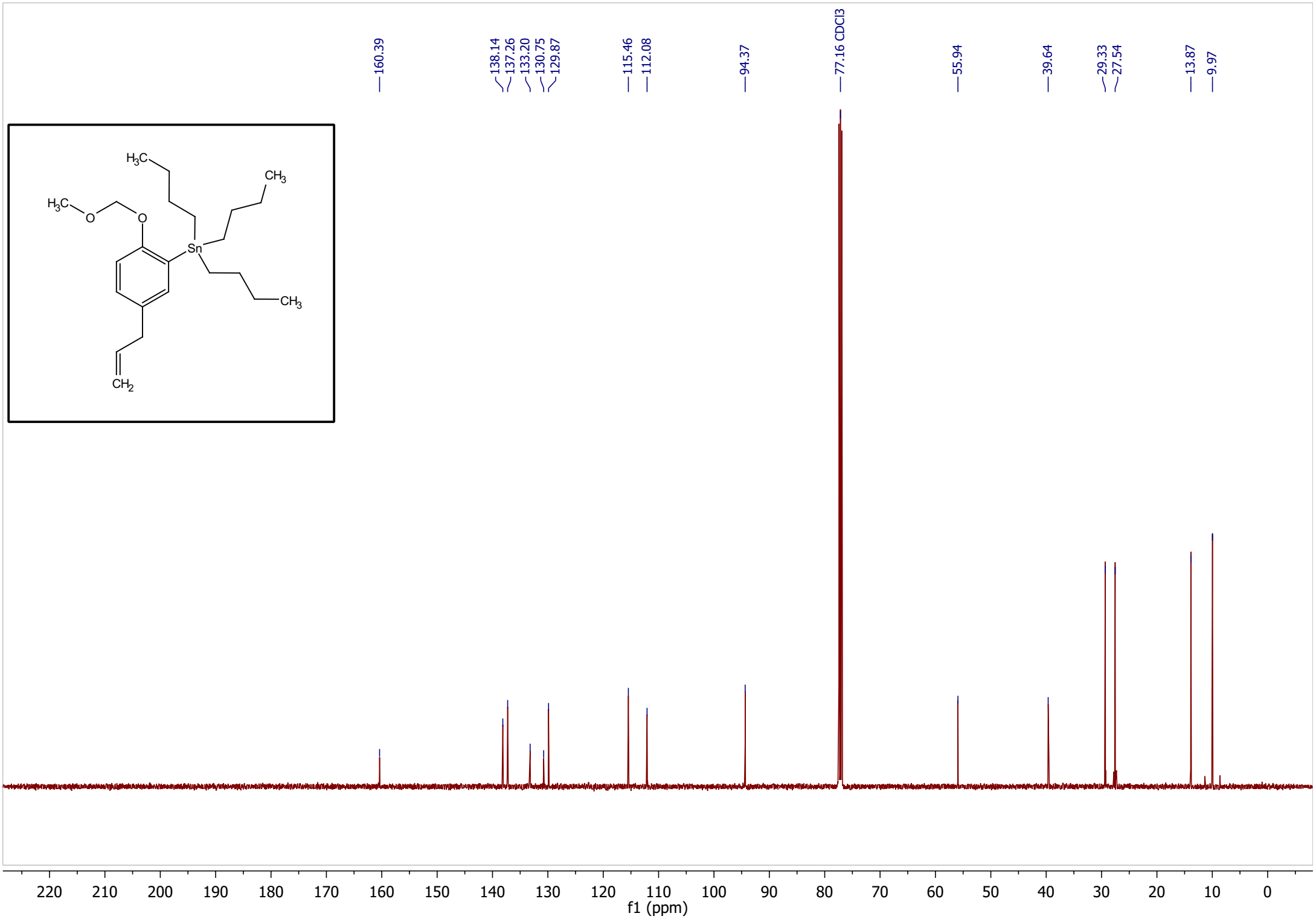


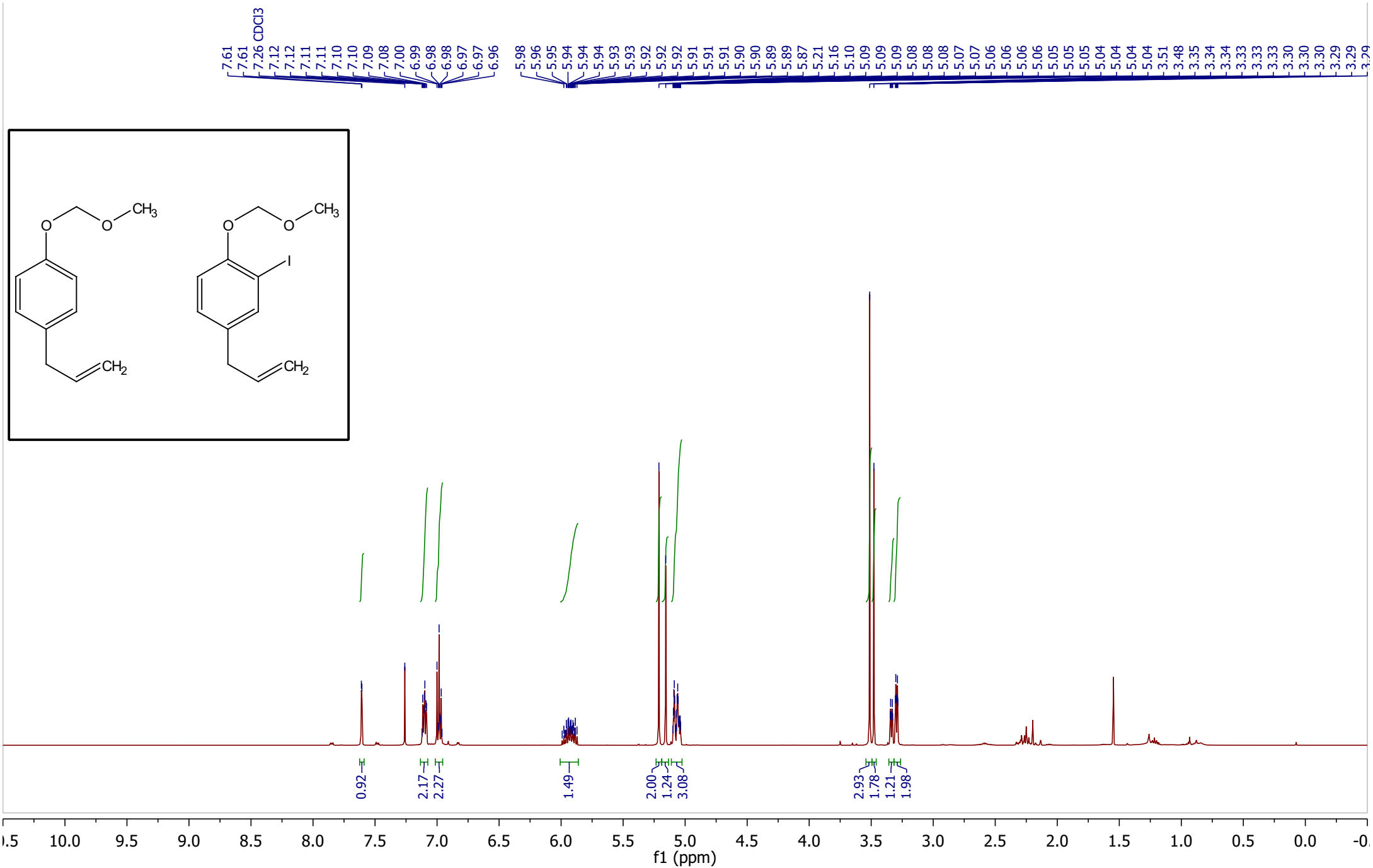
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.13 – 7.09 (m, 2H), 7.00 – 6.95 (m, 2H), 5.95 (ddt, *J* = 16.8, 10.1, 6.7 Hz, 1H), 5.16 (s, 2H), 5.10 – 5.03 (m, 2H), 3.48 (s, 3H), 3.34 (ddd, *J* = 6.7, 1.4, 1.4 Hz, 2H).


155.74
137.86
133.61
129.66
116.44
115.67
94.74
77.16 CDCl3
56.06
39.55
O
O
CH3
CH2
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

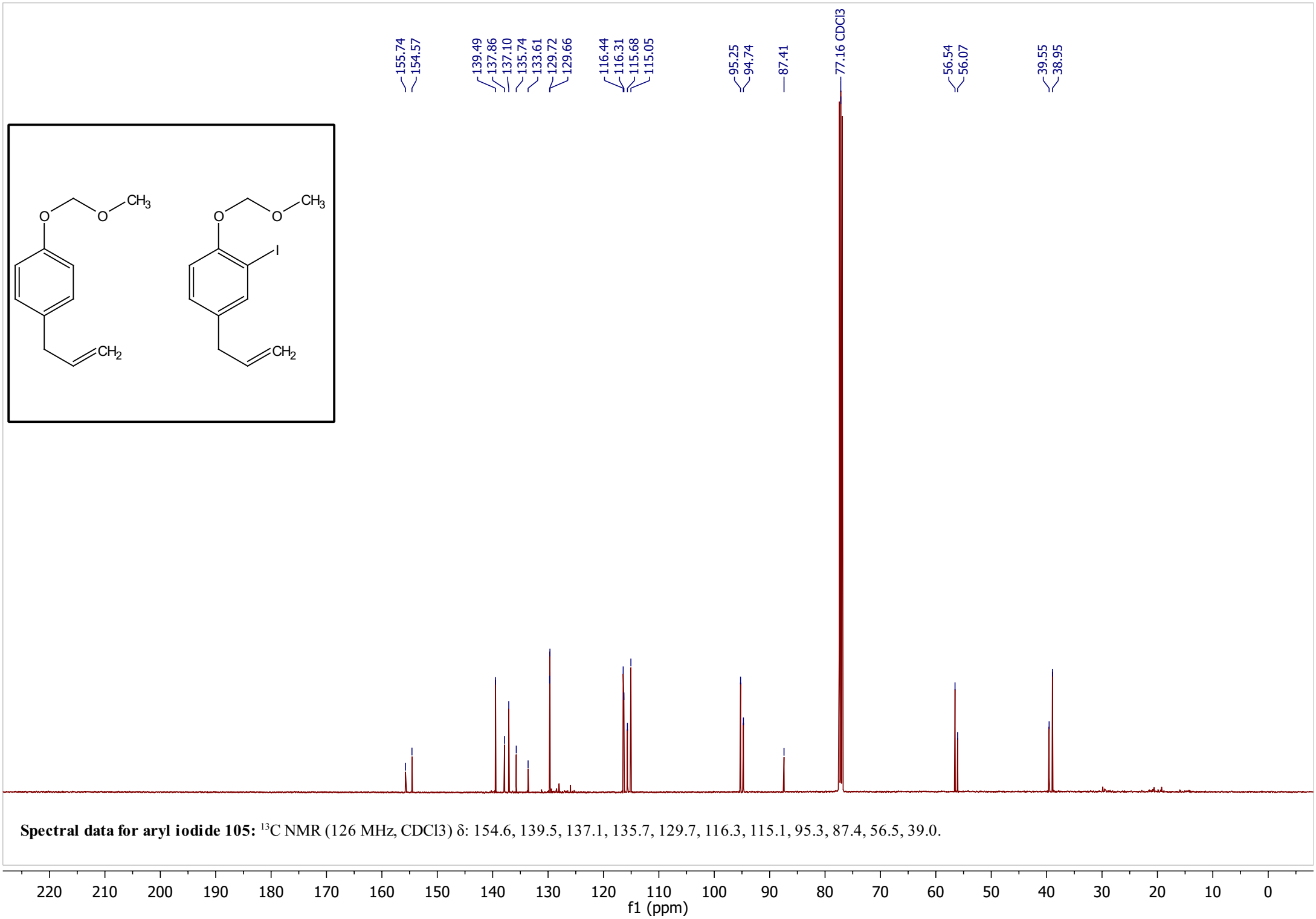


^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.16 (d, *J* = 2.3 Hz, 1H), 7.08 (dd, *J* = 8.4, 2.3 Hz, 1H), 7.00 (d, *J* = 8.4 Hz, 1H), 5.97 (ddt, *J* = 16.8, 10.0, 6.7 Hz, 1H), 5.13 (s, 2H), 5.09 – 5.02 (m, 2H), 3.45 (s, 3H), 3.33 (ddd, *J* = 6.7, 1.4, 1.4 Hz, 2H), 1.60 – 1.45 (m, 6H), 1.37 – 1.28 (m, 6H), 1.12 – 0.97 (m, 6H), 0.88 (t, *J* = 7.3 Hz, 9H).

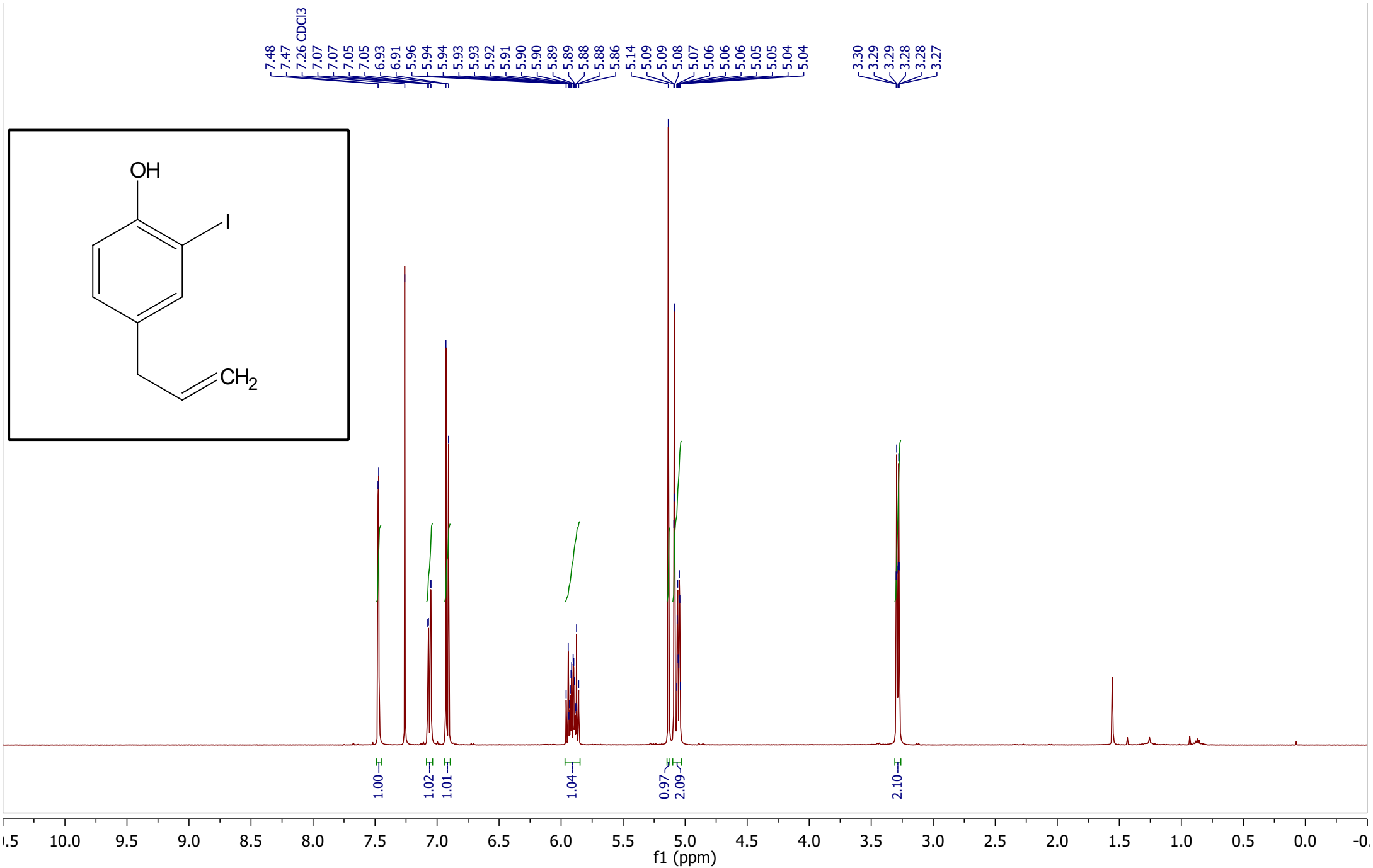

160.39
138.14
137.26
133.20
130.75
129.87
115.46
112.08
94.37
77.16 CDCl3
55.94
39.64
29.33
27.54
13.87
9.97
H3C
CH3
H3C
O
O
Sn
CH3
CH2
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



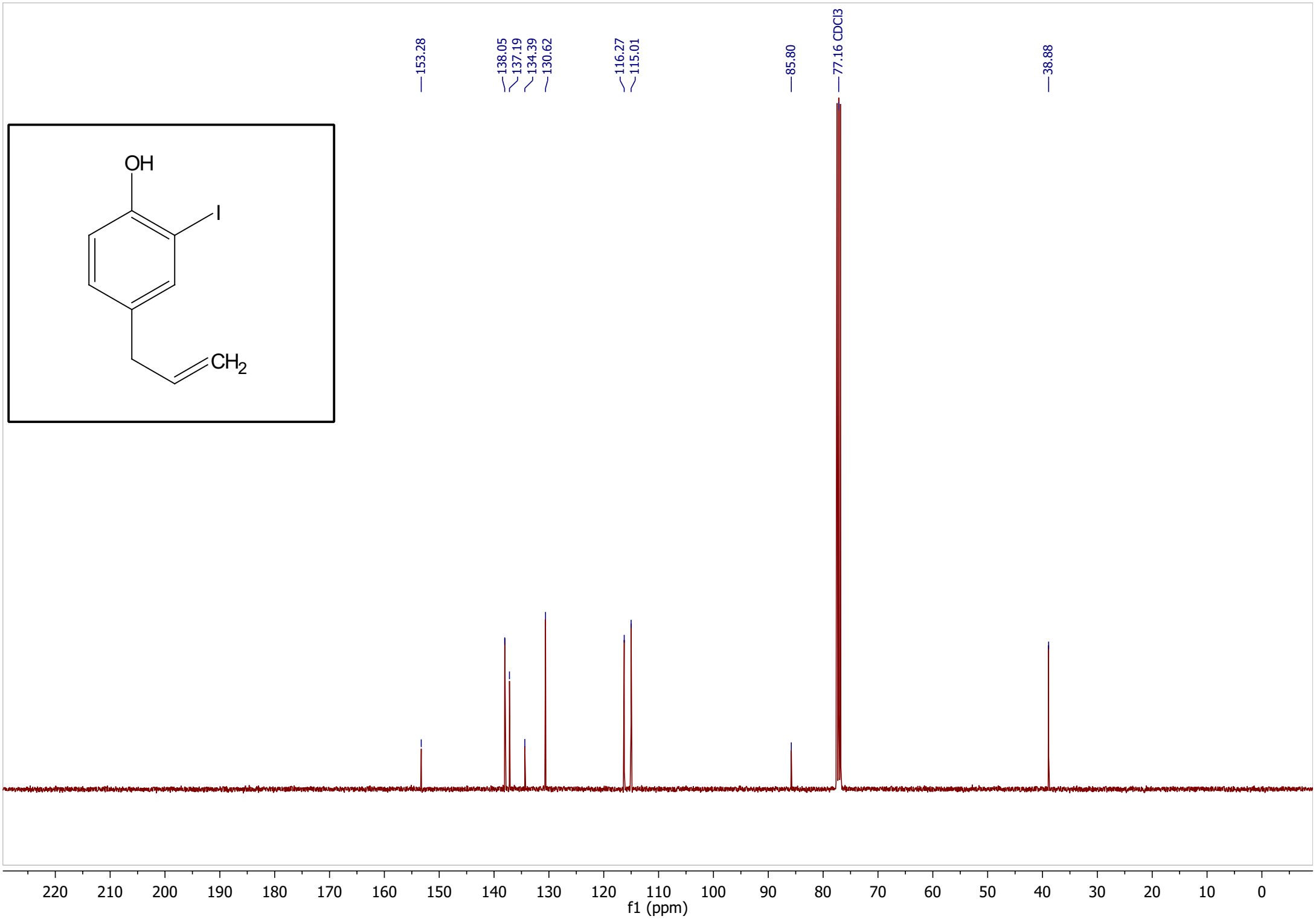
Spectral data for aryl iodide 105: ^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.61 (d, *J* = 2.1 Hz, 1H), 7.10 (d, *J* = 8.4 Hz, 1H), 6.98 (dd, *J* = 8.4, 2.1 Hz, 1H), 6.00 – 5.86 (m, 1H), 5.21 (s, 2H), 5.11 – 5.05 (m, 2H), 3.51 (s, 3H), 3.30 (ddd, *J* = 6.7, 1.5, 1.5 Hz, 2H). Spectral data for aryl iodide 105

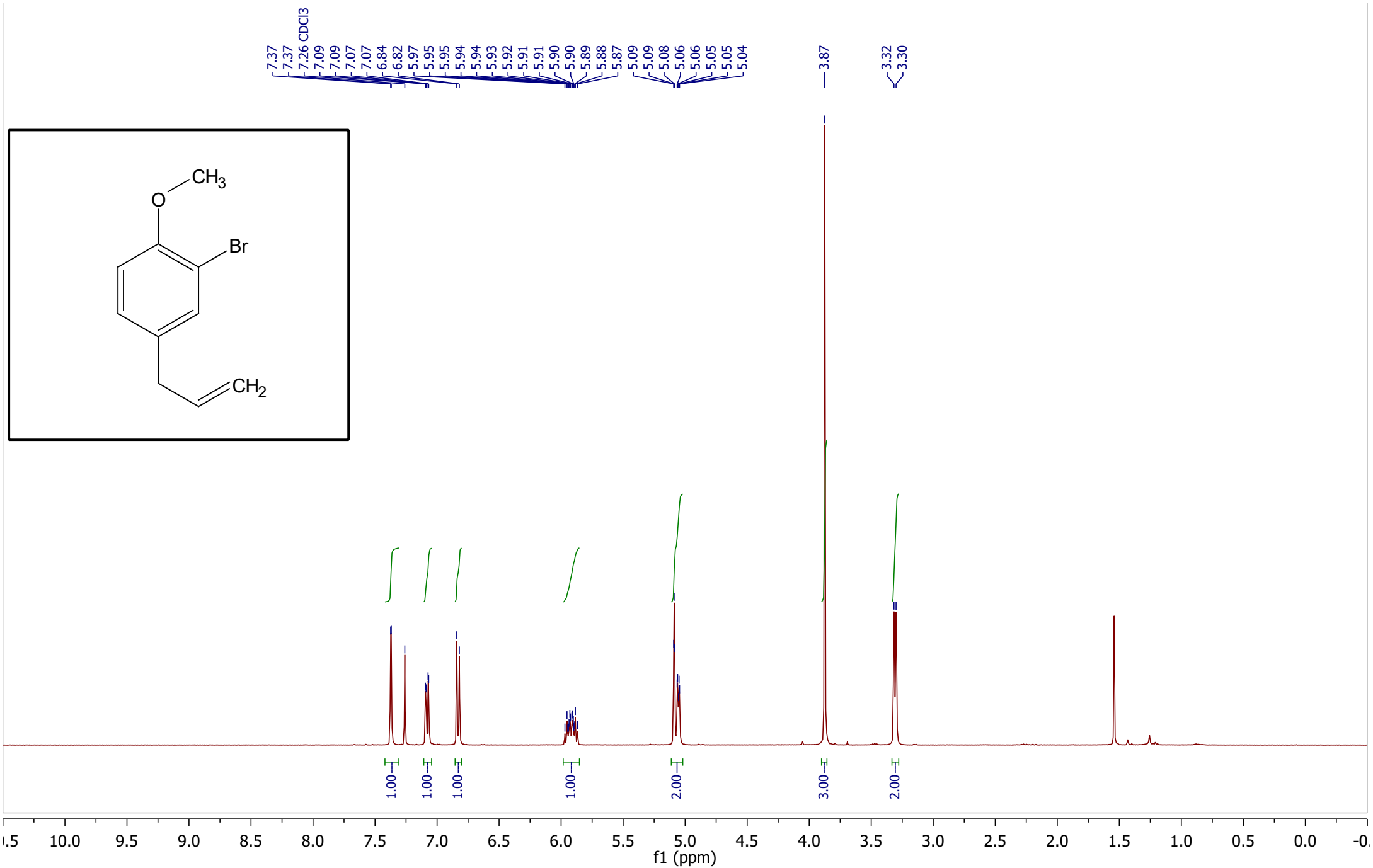


Spectral data for aryl iodide 105: ^{13}C NMR (126 MHz, CDCl3) δ: 154.6, 139.5, 137.1, 135.7, 129.7, 116.3, 115.1, 95.3, 87.4, 56.5, 39.0.

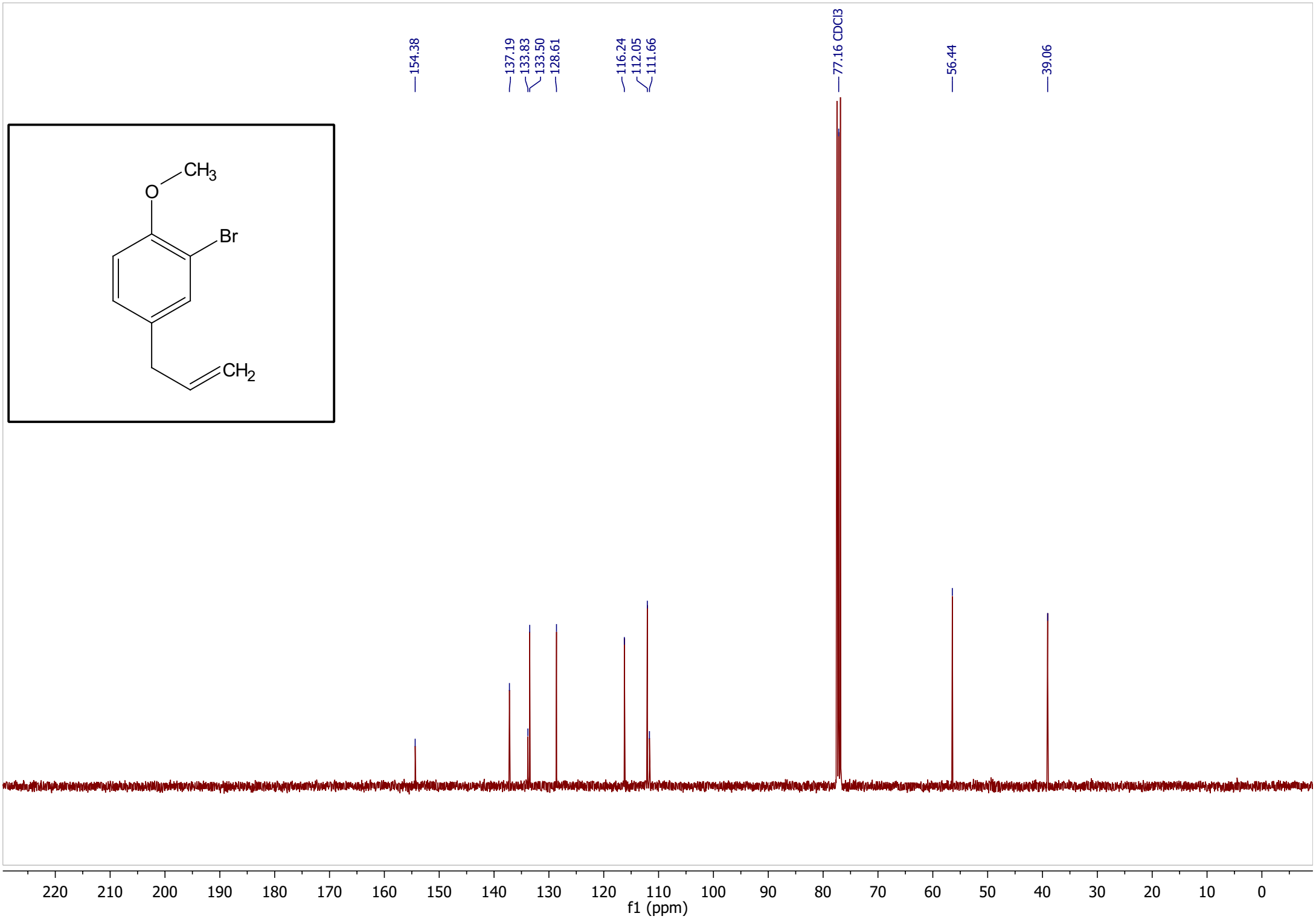


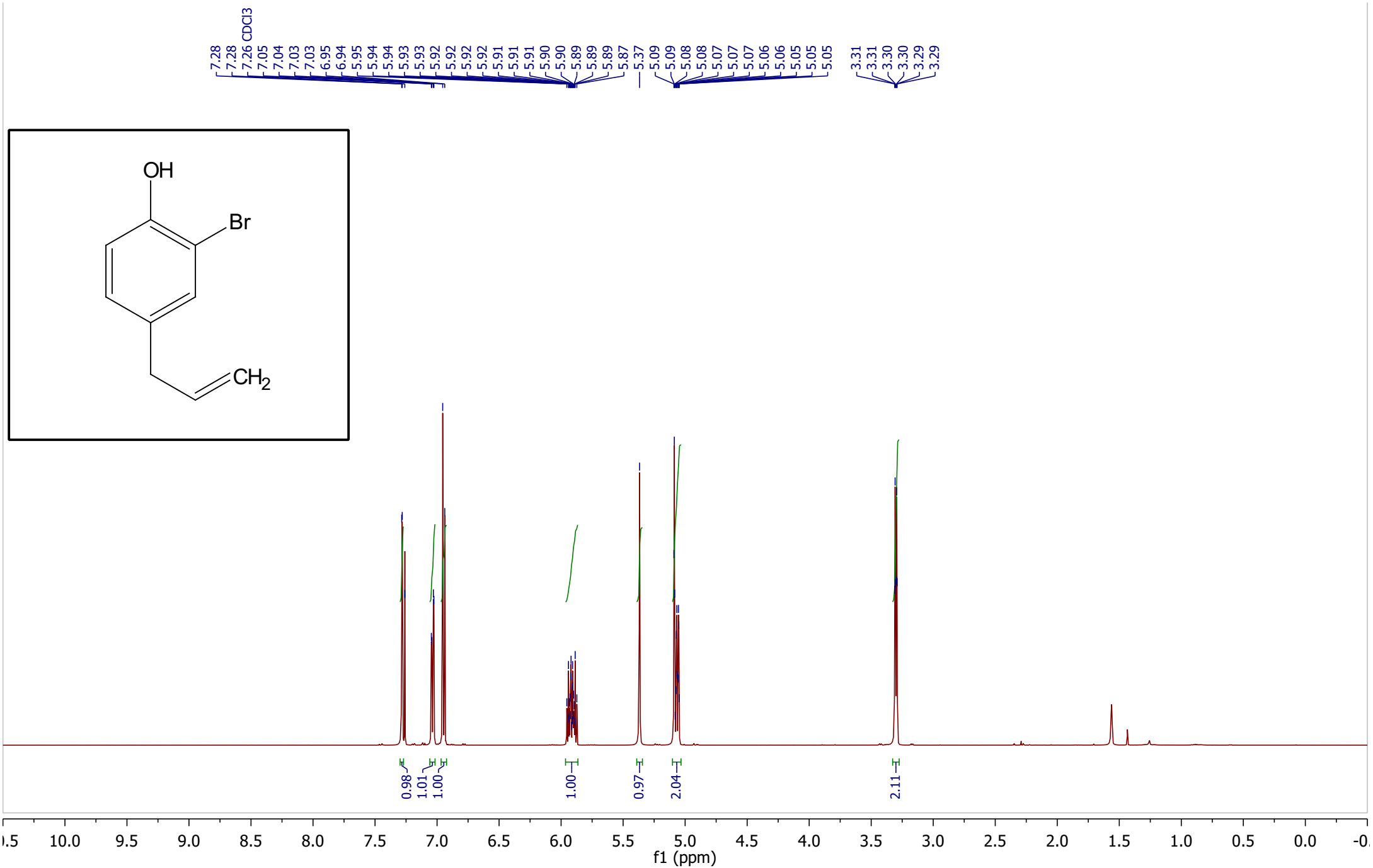
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.47 (d, *J* = 2.1 Hz, 1H), 7.06 (dd, *J* = 8.3, 2.1 Hz, 1H), 6.92 (d, *J* = 8.3 Hz, 1H), 5.91 (ddt, *J* = 17.8, 9.1, 6.7 Hz, 1H), 5.14 (s, 1H), 5.10 – 5.03 (m, 2H), 3.29 (ddd, *J* = 6.7, 1.5, 1.5 Hz, 2H).


153.28
138.05
137.19
134.39
130.62
116.27
115.01
85.80
77.16 CDCl3
38.88
OH
I
CH2
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

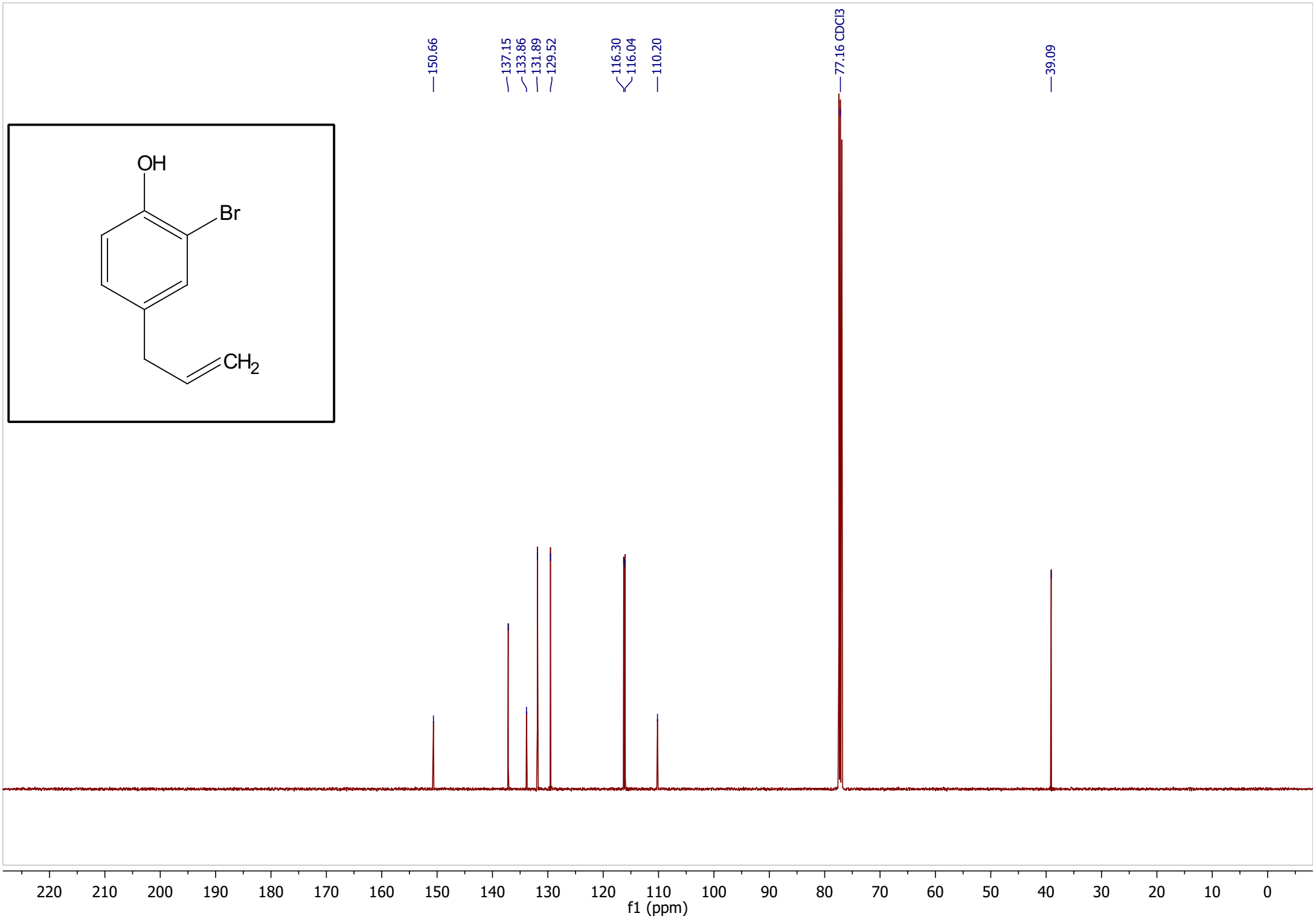


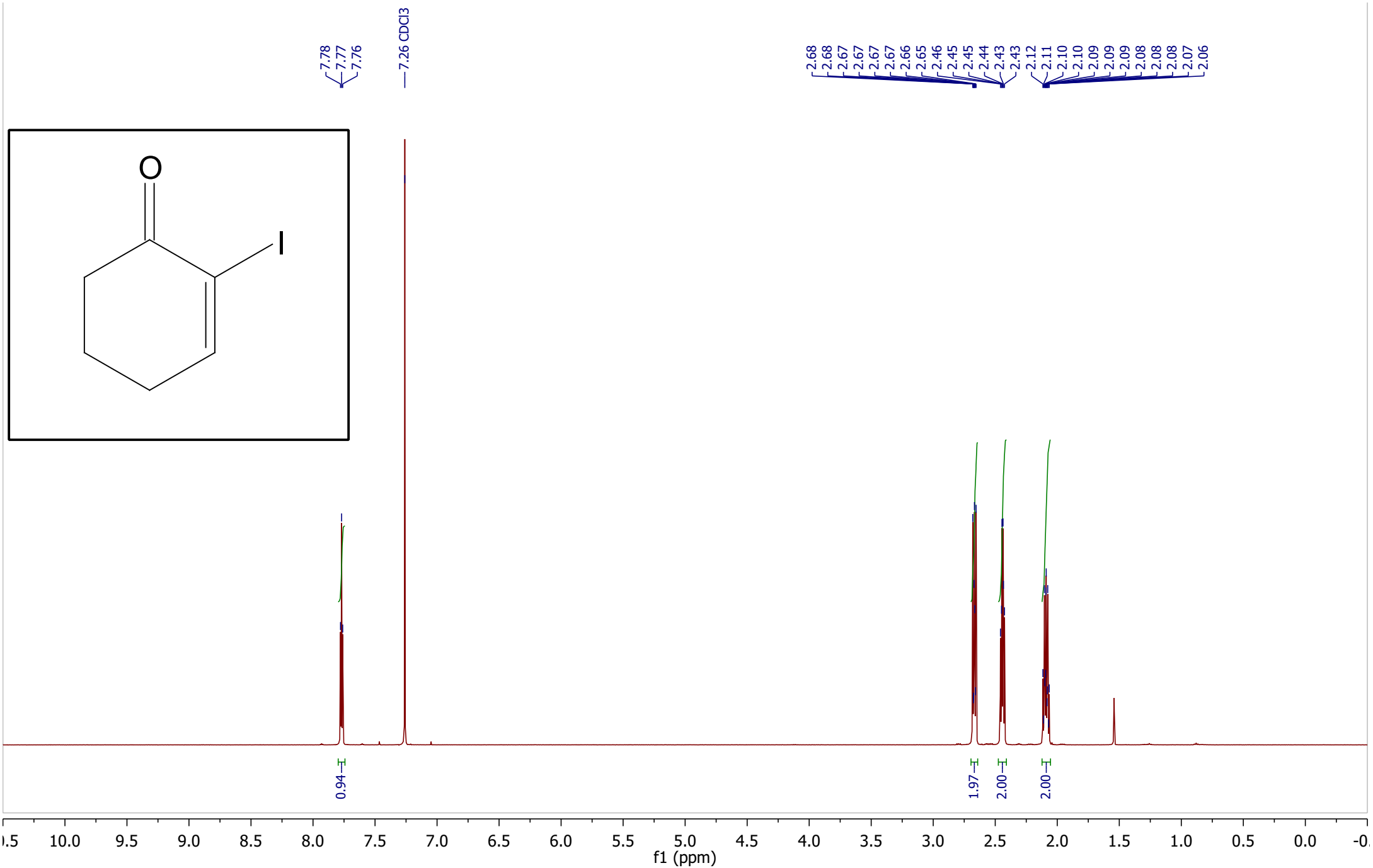
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.37 (d, *J* = 2.0 Hz, 1H), 7.08 (dd, *J* = 8.4, 2.0 Hz, 1H), 6.83 (d, *J* = 8.4 Hz, 1H), 5.92 (ddt, *J* = 17.9, 9.1, 6.7 Hz, 1H), 5.15 – 5.00 (m, 2H), 3.87 (s, 3H), 3.31 (d, *J* = 6.7 Hz, 2H).


154.38
137.19
133.83
133.50
128.61
116.24
112.05
111.66
77.16 CDCl3
56.44
39.06
CH3
O
Br
CH2
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

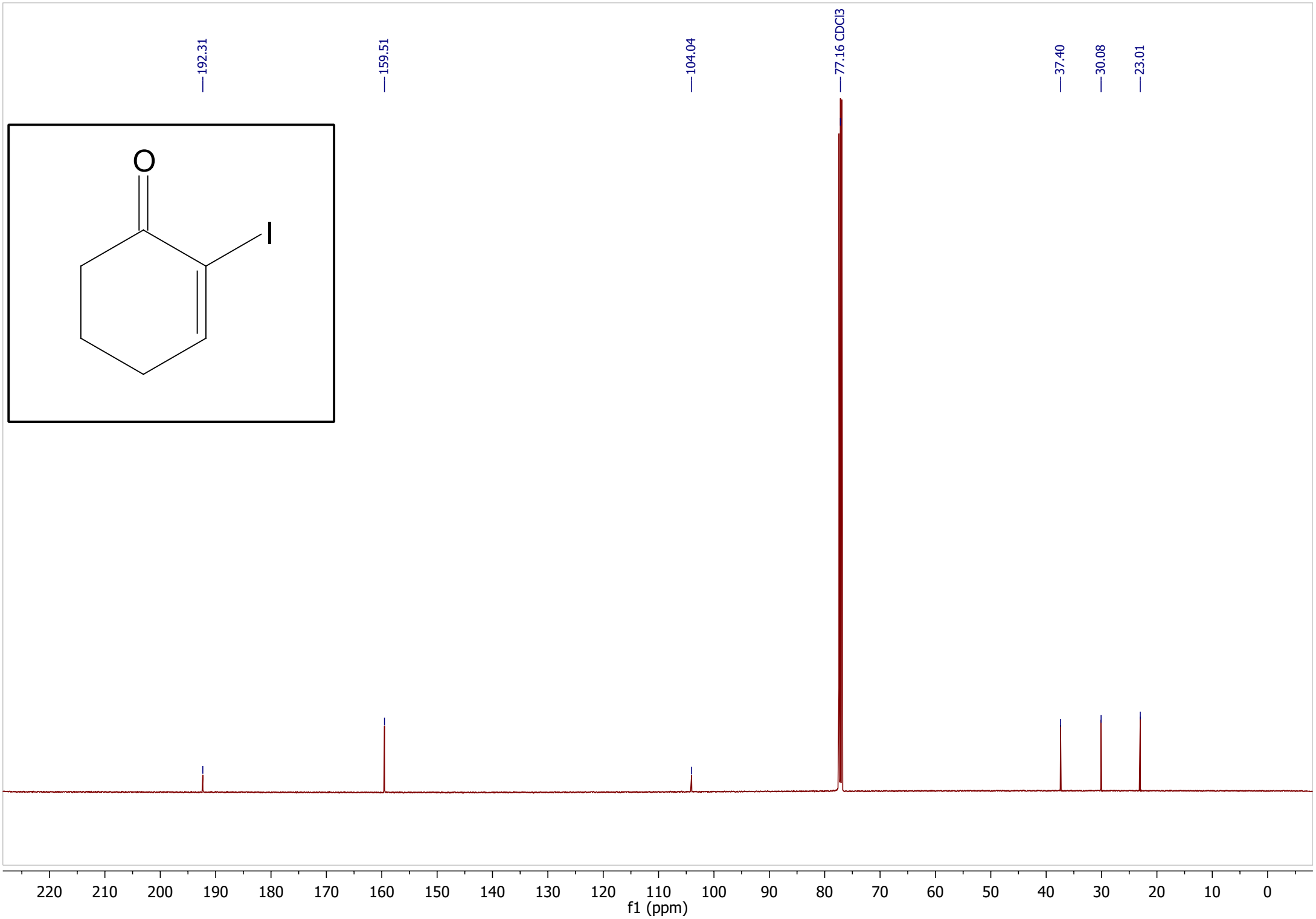


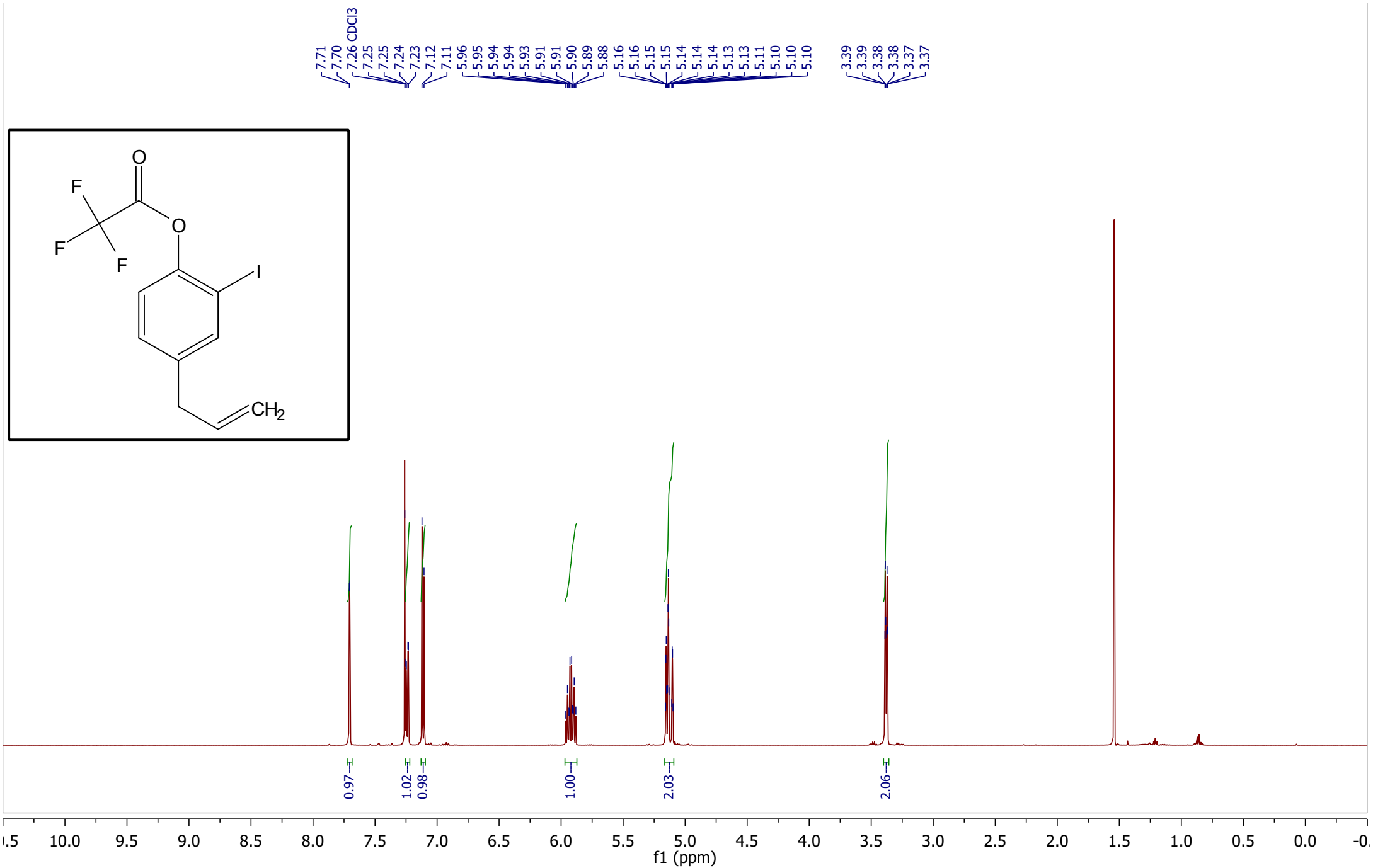
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.28 (d, *J* = 2.1 Hz, 1H), 7.04 (dd, *J* = 8.3, 2.1 Hz, 1H), 6.95 (d, *J* = 8.3 Hz, 1H), 5.91 (ddt, *J* = 16.4, 10.6, 6.7 Hz, 1H), 5.37 (s, 1H), 5.11 – 5.04 (m, 2H), 3.30 (ddd, *J* = 6.7, 1.5, 1.5 Hz, 2H).


150.66
137.15
133.86
131.89
129.52
116.30
116.04
110.20
77.16 CDCl3
39.09
OH
Br
CH2
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

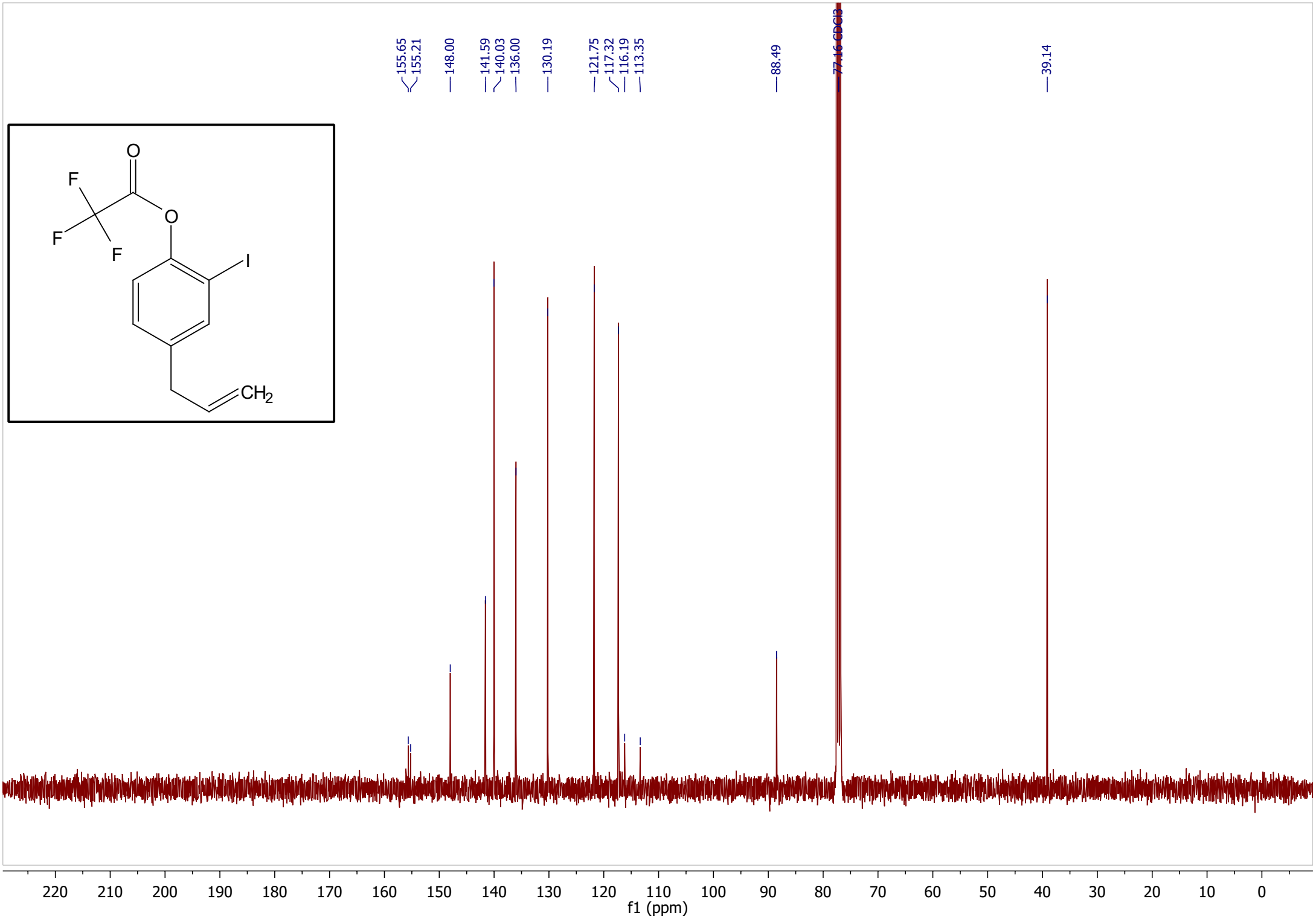


^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.77 (t, *J* = 4.4 Hz, 1H), 2.70 – 2.64 (m, 2H), 2.44 (td, *J* = 6.0, 4.4 Hz, 2H), 2.14 – 2.04 (m, 2H).

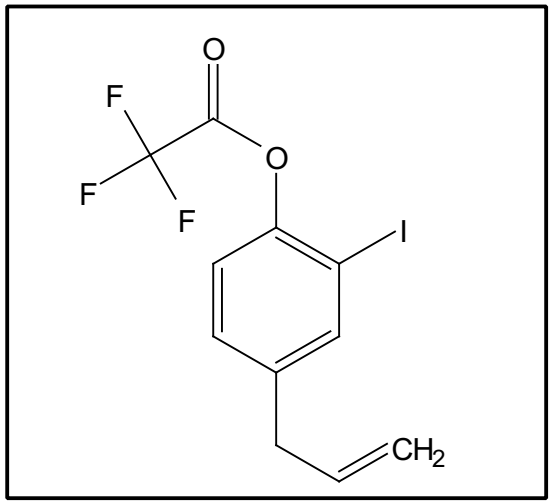

192.31
159.51
104.04
77.16 CDCl3
37.40
30.08
23.01
O
I
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

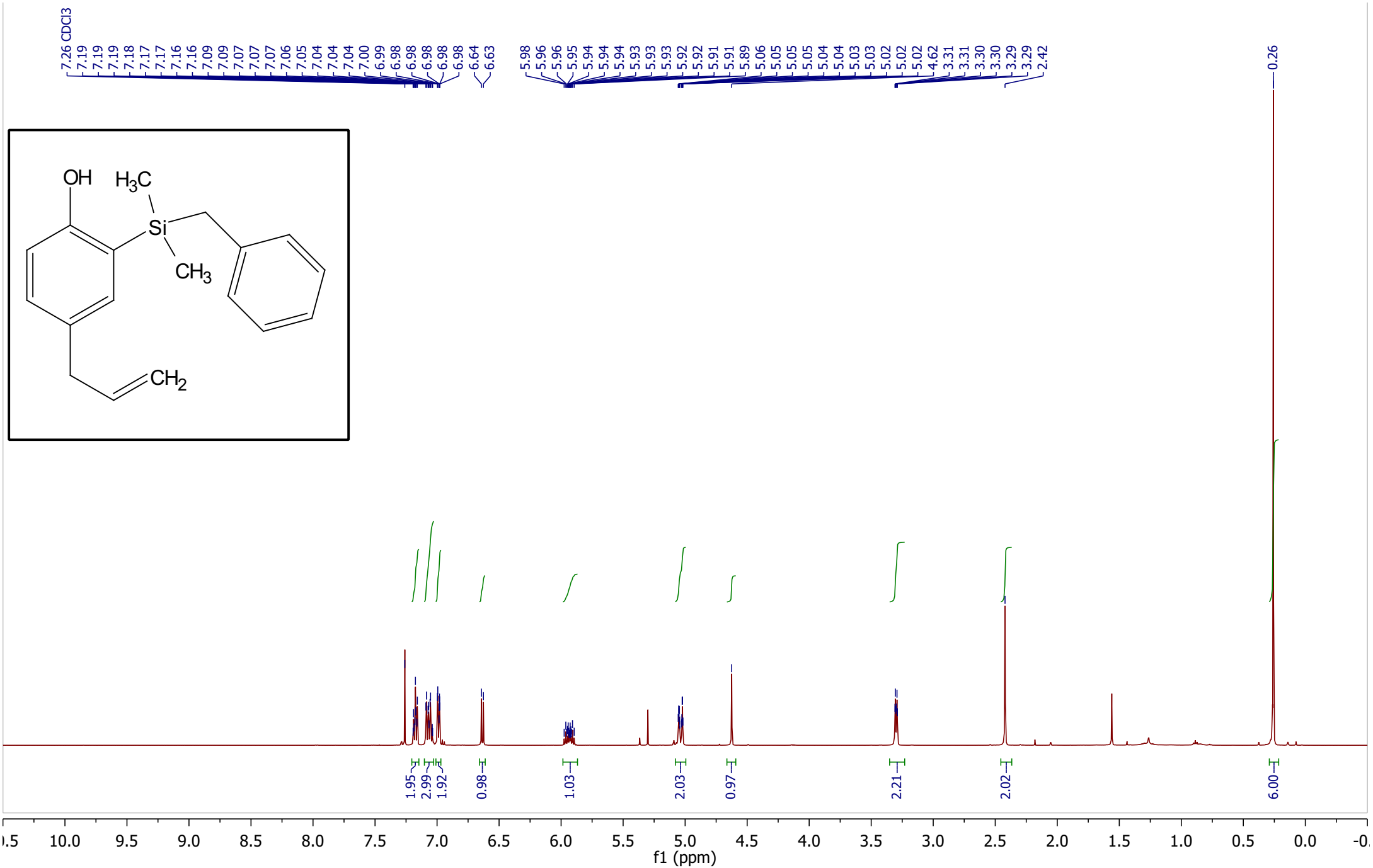


^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.71 (d, *J* = 2.1 Hz, 1H), 7.24 (dd, *J* = 8.3, 2.1 Hz, 1H), 7.11 (d, *J* = 8.3 Hz, 1H), 5.92 (ddt, *J* = 16.9, 10.1, 6.7 Hz, 1H), 5.17 – 5.09 (m, 2H), 3.38 (ddd, *J* = 6.7, 1.4, 1.4 Hz, 2H).

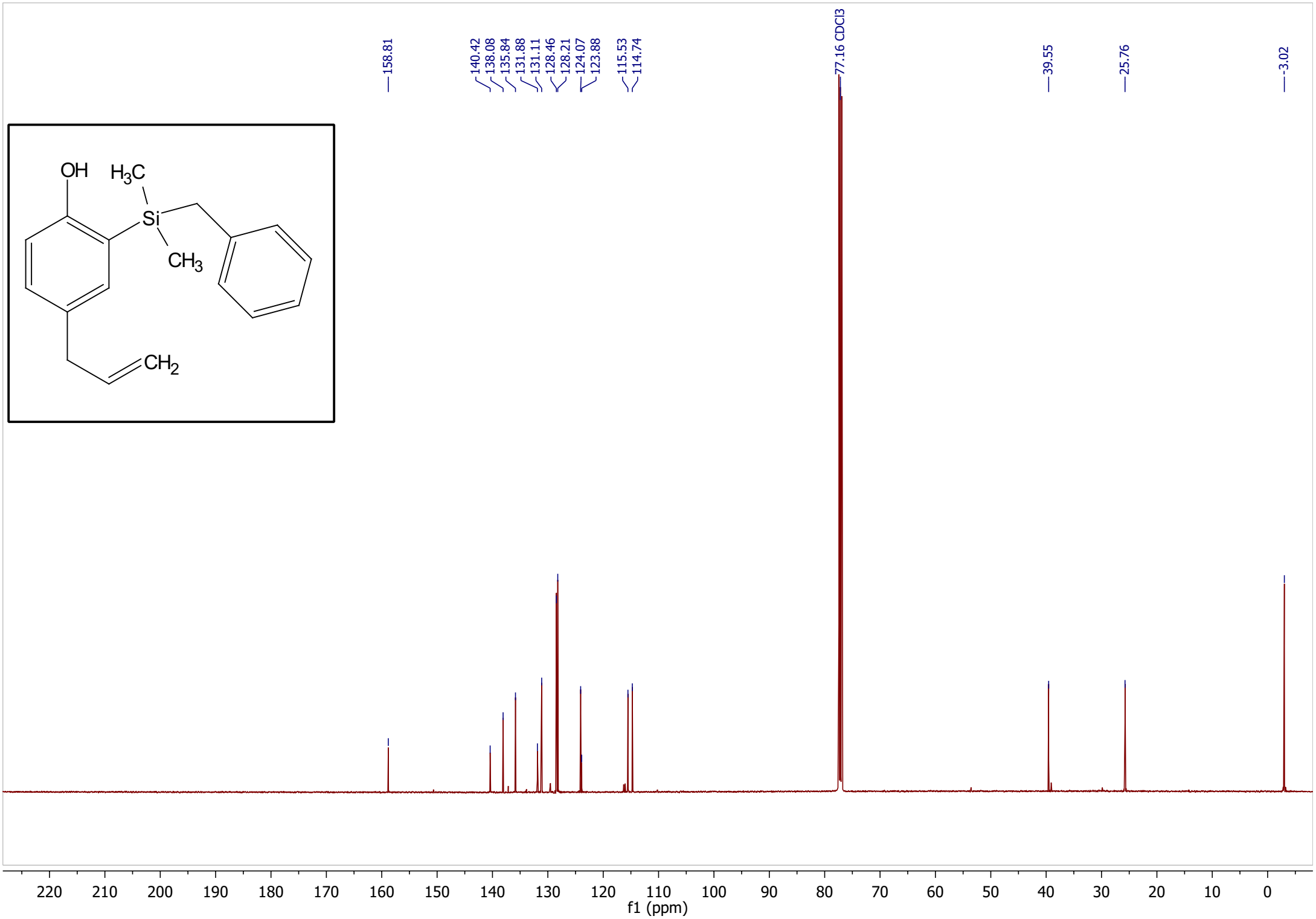

155.65
155.21
148.00
141.59
140.03
136.00
130.19
121.75
117.32
116.19
113.35
88.49
77.16 CDCl3
39.14
O
F
F
F
O
I
CH2
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

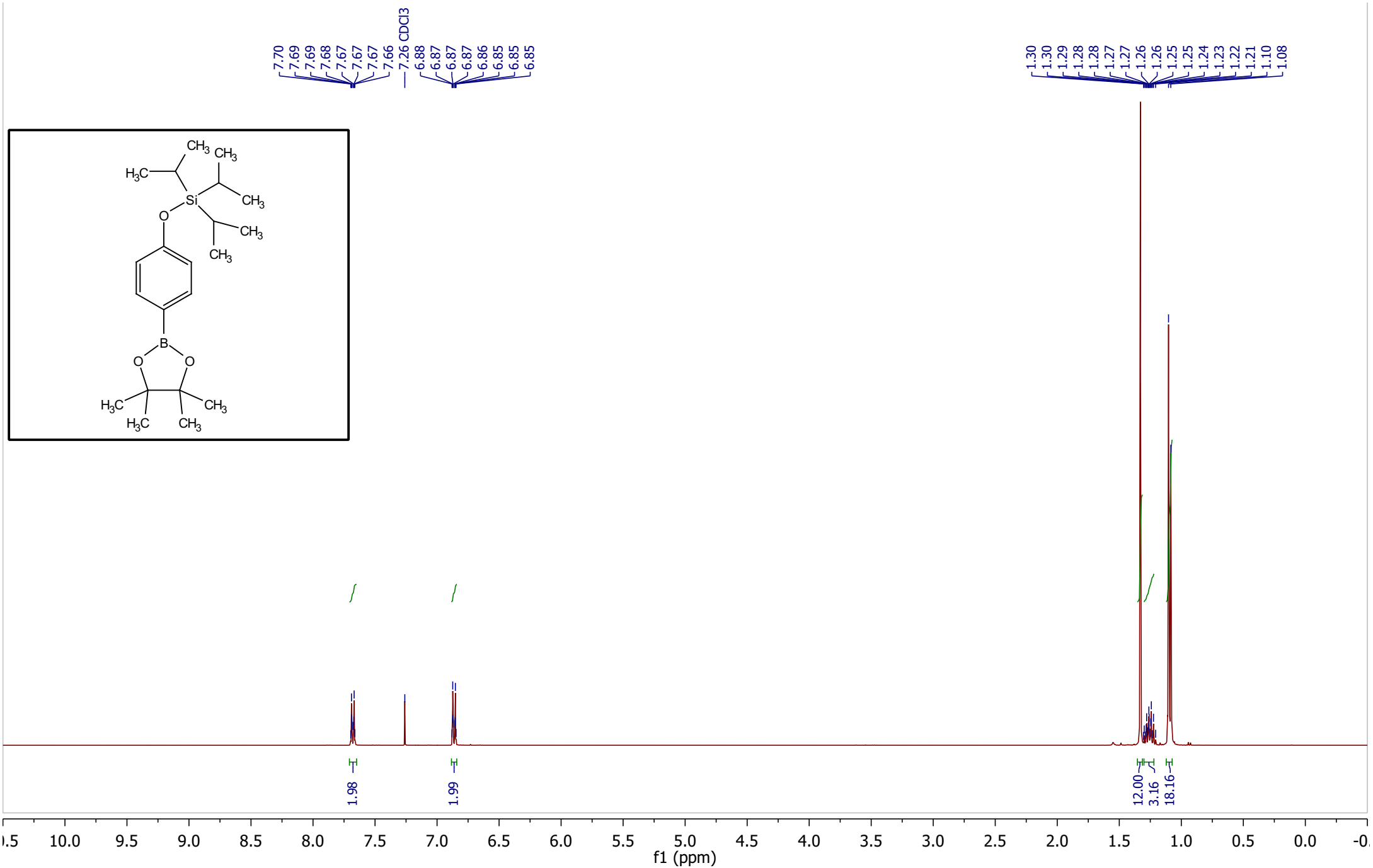
^{19}F NMR spectra



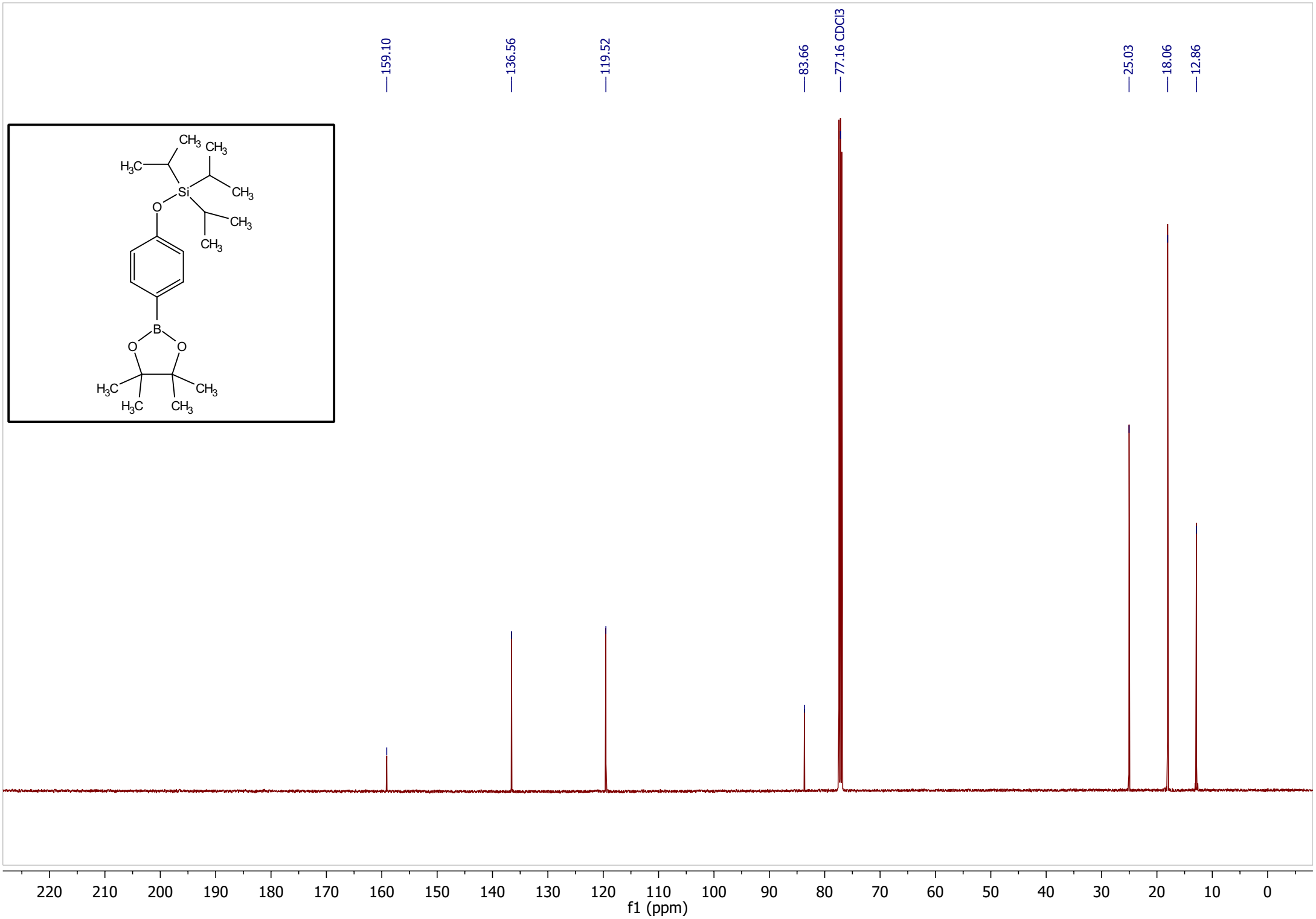


^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.20 – 7.15 (m, 2H), 7.11 – 7.03 (m, 3H), 7.00 – 6.96 (m, 2H), 6.63 (d, *J* = 8.0 Hz, 1H), 5.93 (ddt, *J* = 17.6, 9.5, 6.7 Hz, 1H), 5.08 – 5.00 (m, 2H), 4.62 (s, 1H), 3.30 (ddd, *J* = 6.7, 1.5, 1.5 Hz, 2H), 2.42 (s, 2H), 0.26 (s, 6H).

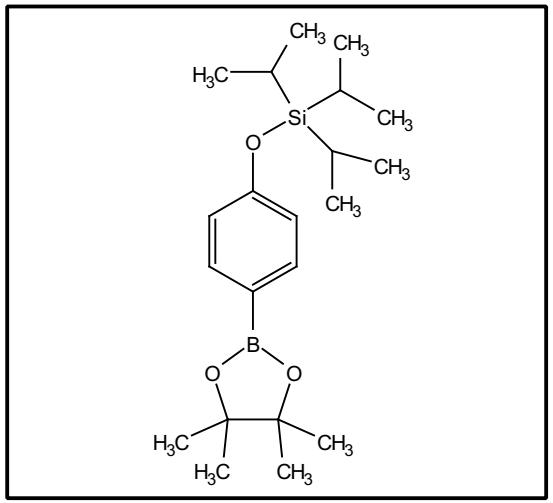

158.81
140.42
138.08
135.84
131.88
131.11
128.46
128.21
124.07
123.88
115.53
114.74
77.16 CDCl3
39.55
25.76
-3.02
OH
H3C
Si
CH3
CH2
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



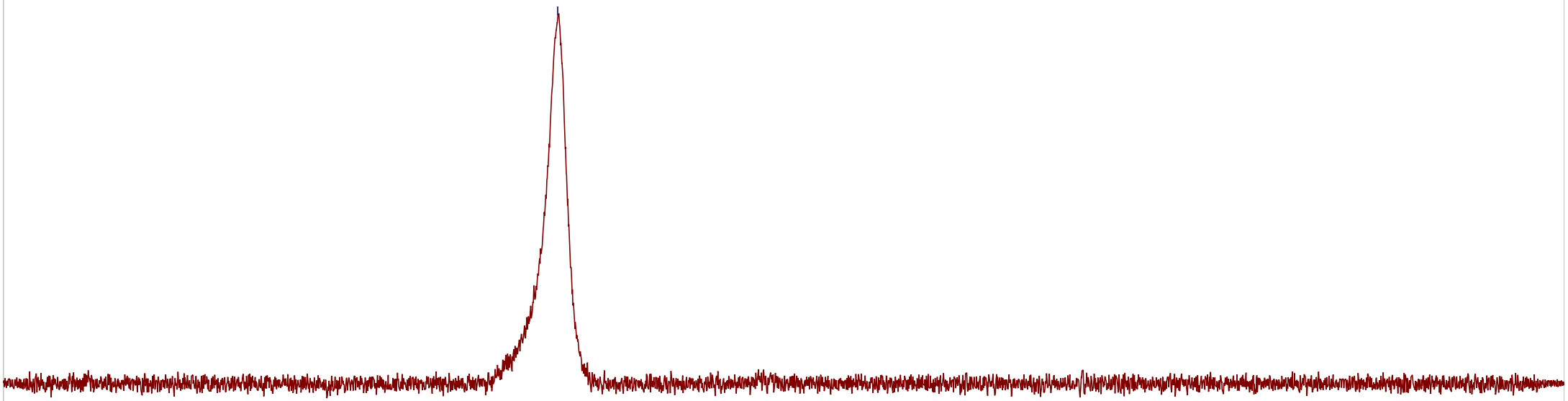
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.70 – 7.66 (m, 2H), 6.88 – 6.84 (m, 2H), 1.33 (s, 12H), 1.31 – 1.21 (m, 3H), 1.09 (d, *J* = 7.3 Hz, 18H).

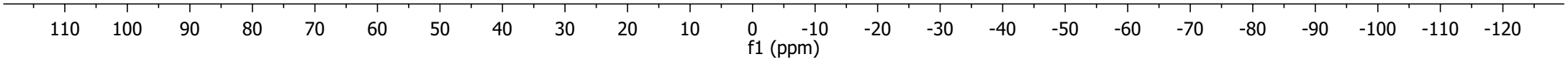

159.10
136.56
119.52
83.66
77.16 CDCl3
25.03
18.06
12.86
f1 (ppm)

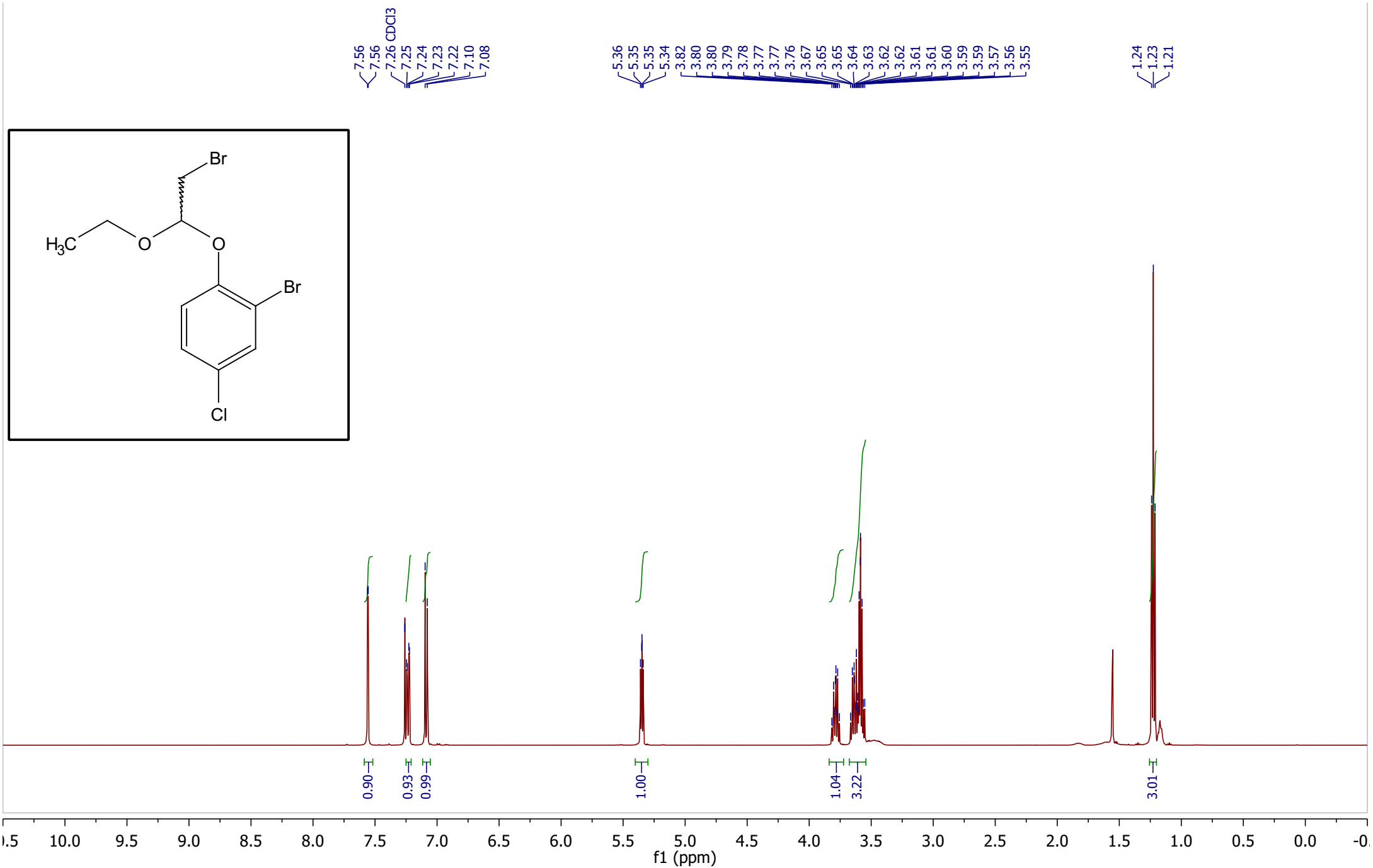
^{11}B NMR spectra



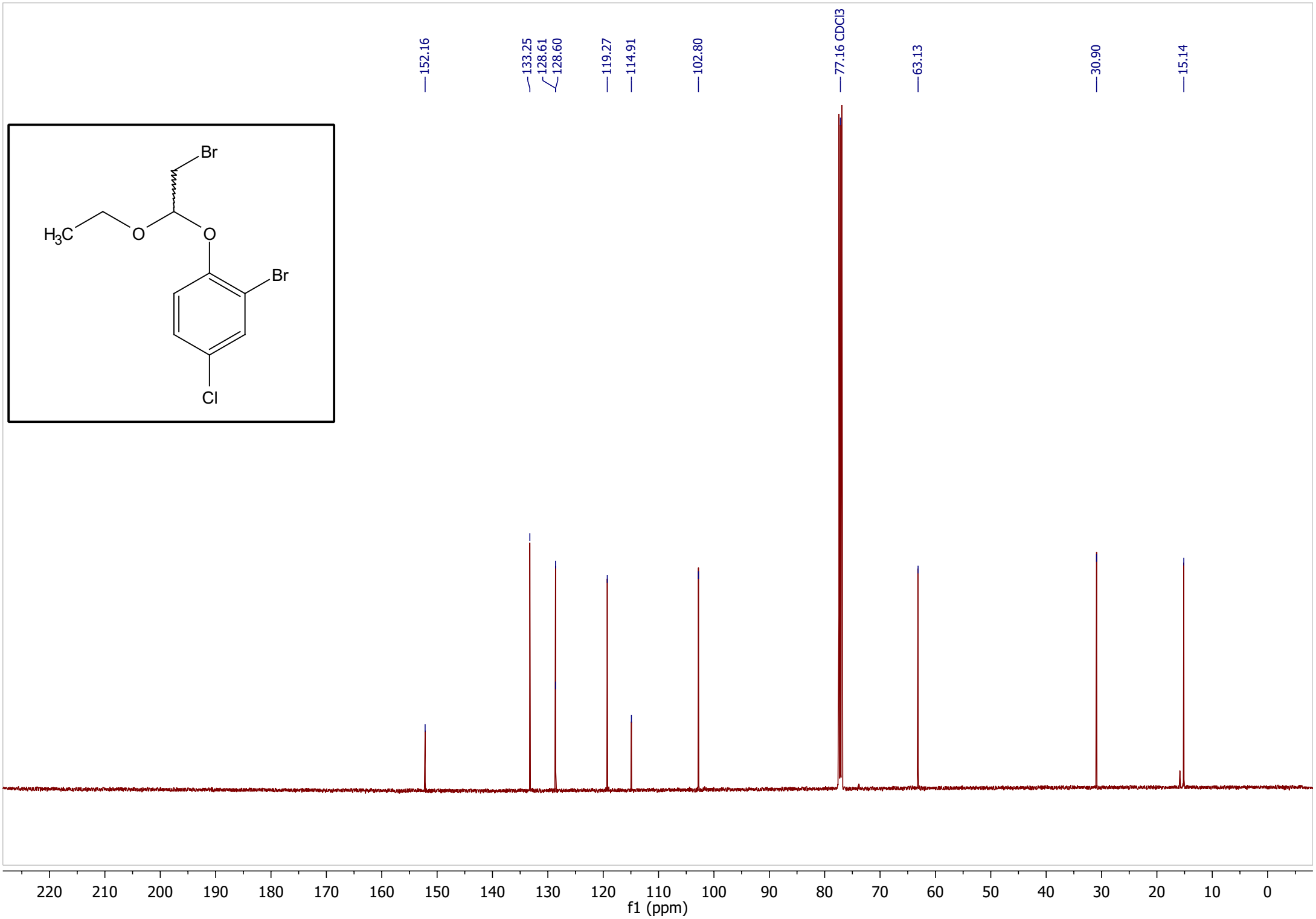
31.17

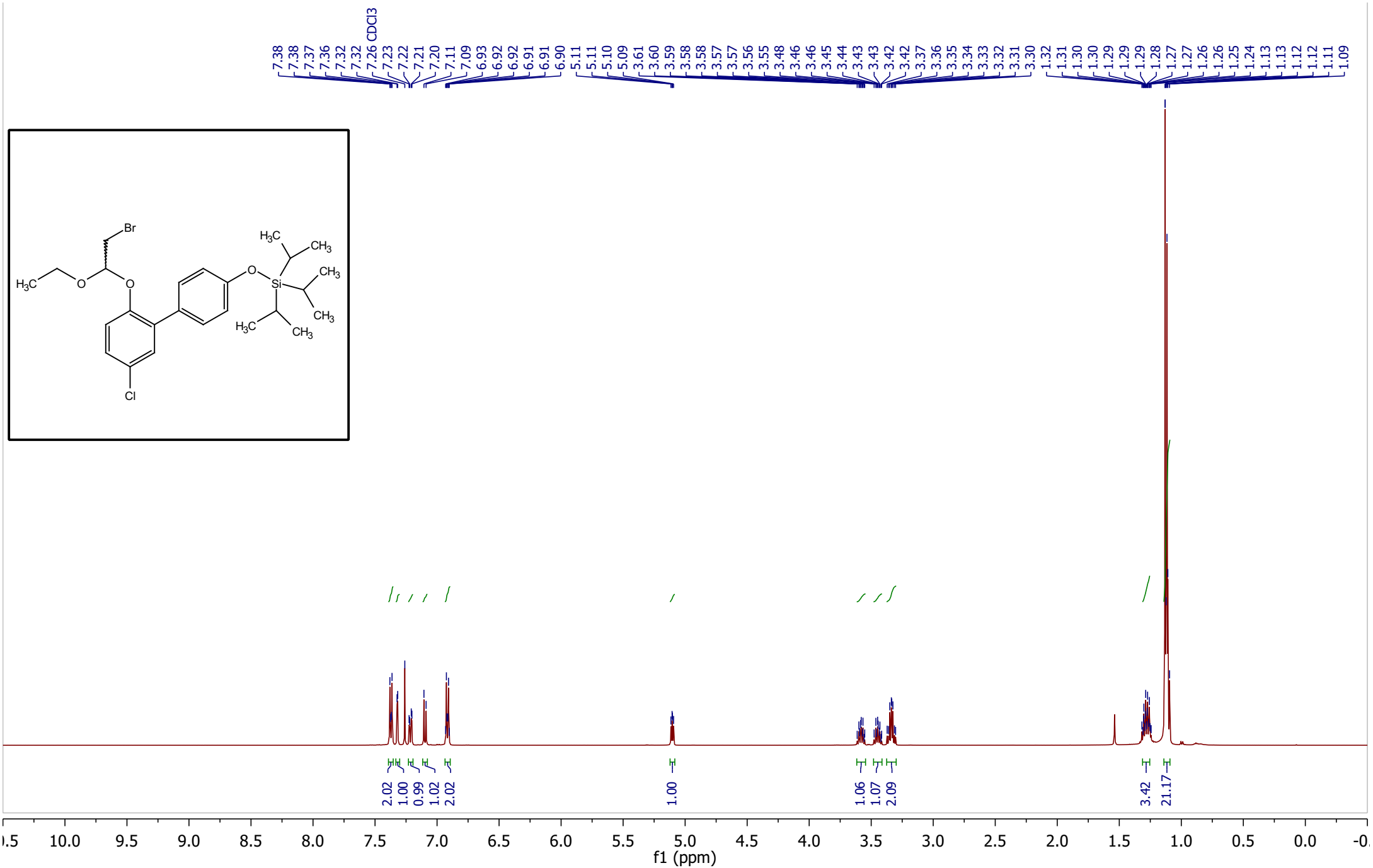




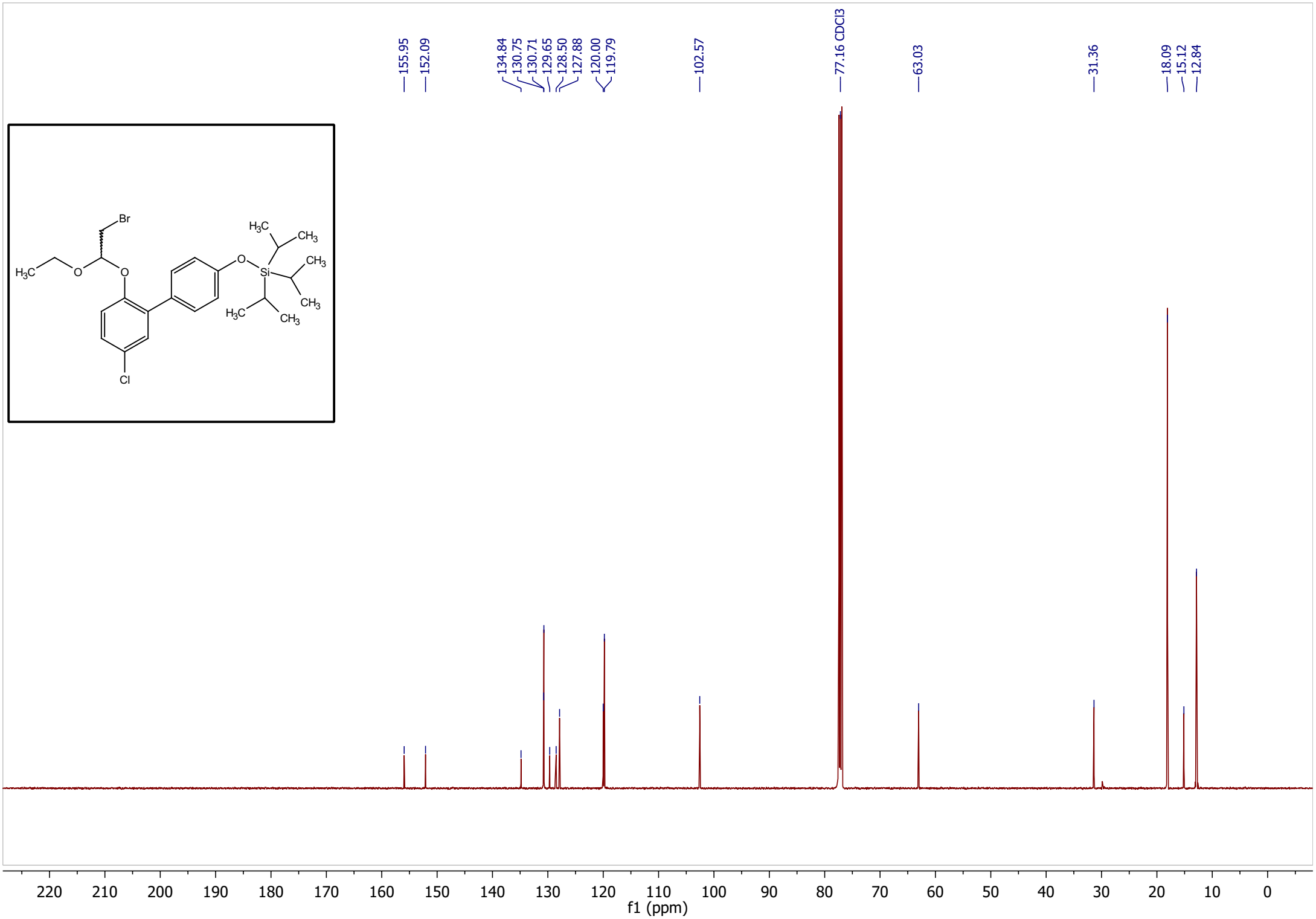


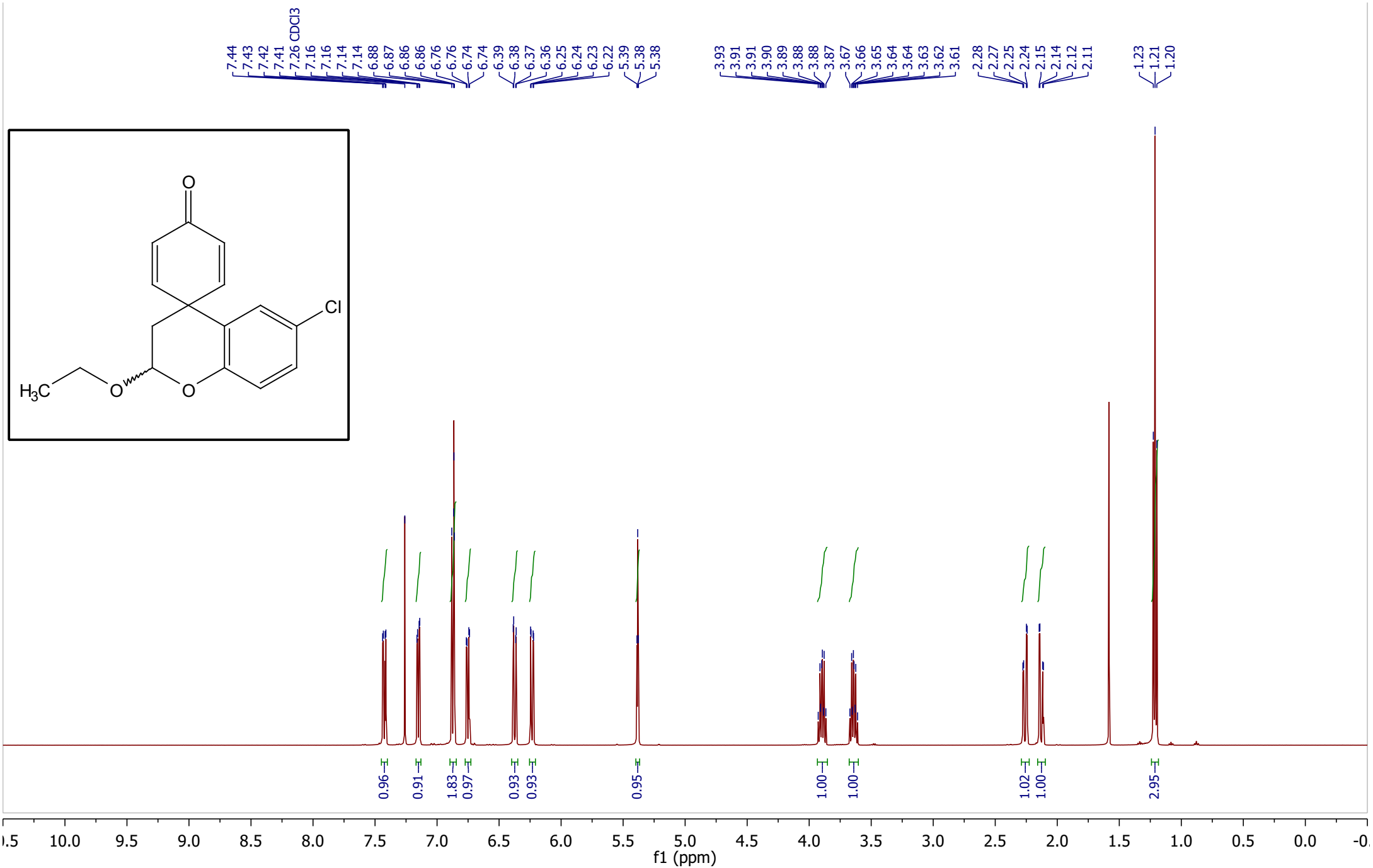
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.56 (d, *J* = 2.5 Hz, 1H), 7.23 (dd, *J* = 8.8, 2.5 Hz, 1H), 7.09 (d, *J* = 8.8 Hz, 1H), 5.35 (dd, *J* = 6.1, 4.8 Hz, 1H), 3.79 (dq, *J* = 9.3, 7.0 Hz, 1H), 3.68 – 3.54 (m, 3H), 1.23 (dd, *J* = 7.0, 7.0 Hz, 3H).


152.16
133.25
128.61
128.60
119.27
114.91
102.80
77.16 CDCl3
63.13
30.90
15.14
Br
H3C
O
O
Br
Cl
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

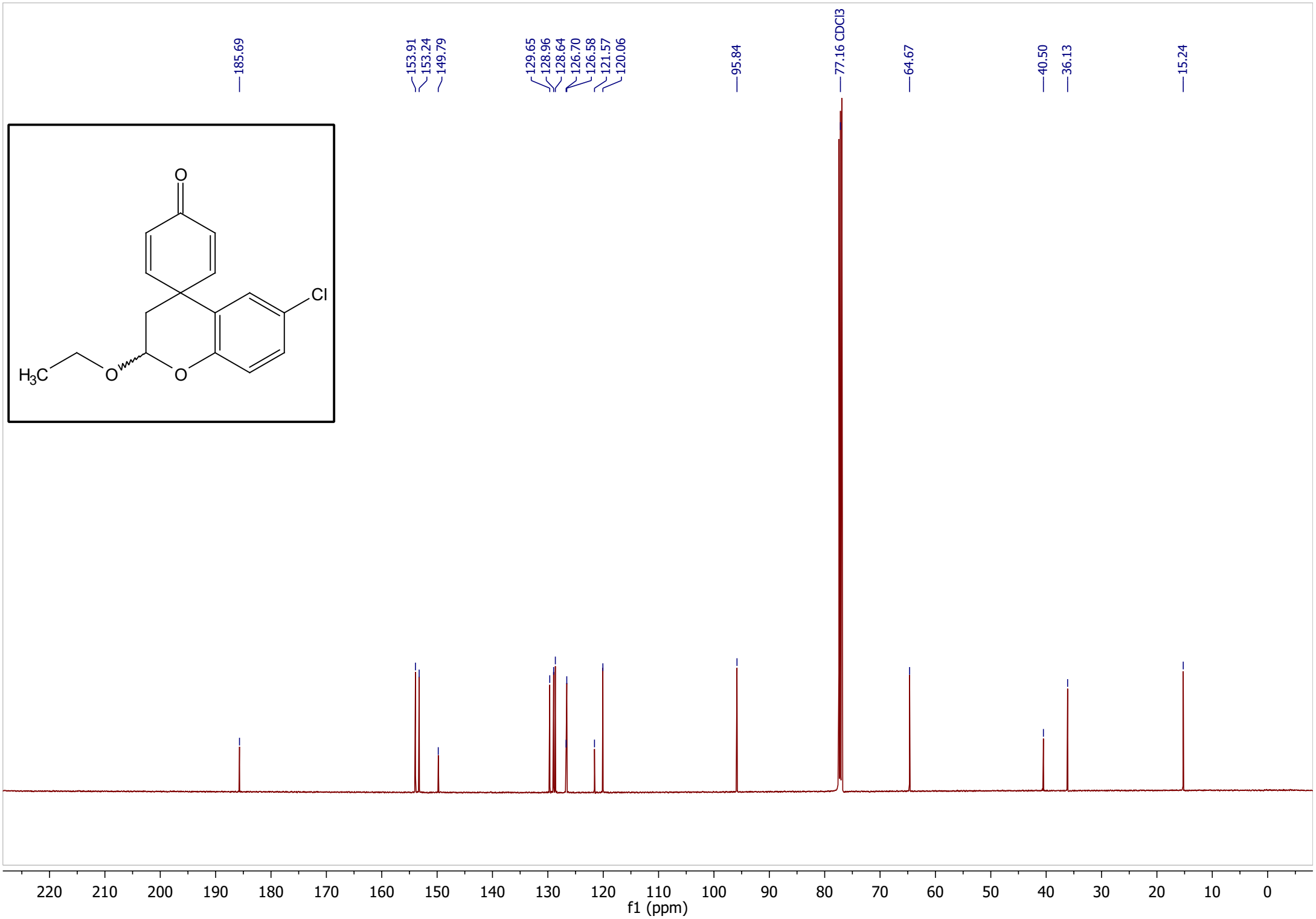


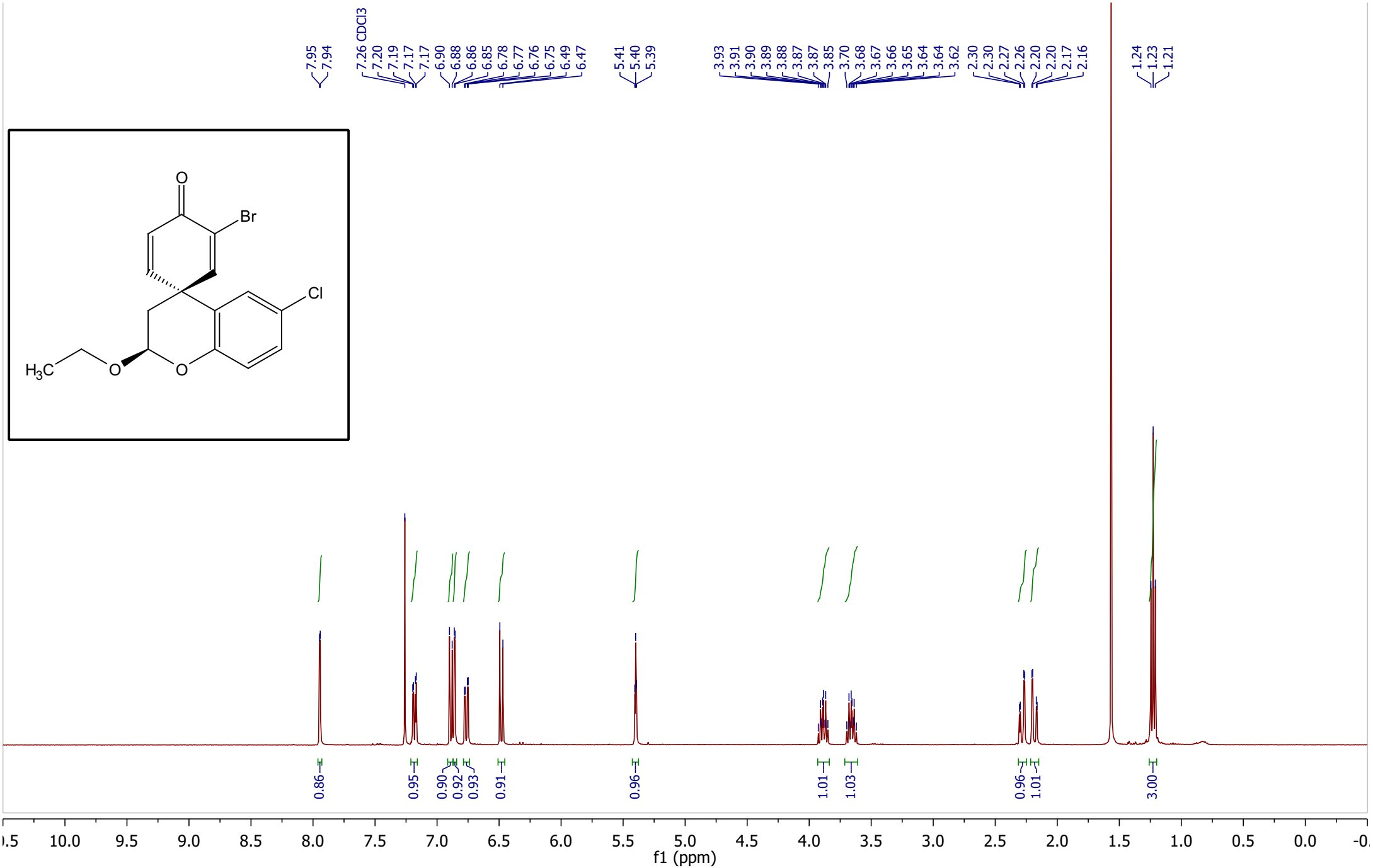
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.39 – 7.35 (m, 2H), 7.32 (d, *J* = 2.7 Hz, 1H), 7.21 (dd, *J* = 8.7, 2.7 Hz, 1H), 7.10 (d, *J* = 8.7 Hz, 1H), 6.95 – 6.89 (m, 2H), 5.10 (dd, *J* = 6.1, 4.6 Hz, 1H), 3.58 (dq, *J* = 9.3, 7.1 Hz, 1H), 3.45 (dq, *J* = 9.3, 7.1 Hz, 1H), 3.38 – 3.30 (m, 2H), 1.32 – 1.24 (m, 3H), 1.12 (d, *J* = 7.4 Hz, 18H), 1.11 (dd, *J* = 7.1, 7.1 Hz, 3H).


155.95
152.09
134.84
130.75
130.71
129.65
128.50
127.88
120.00
119.79
102.57
77.16 CDCl3
63.03
31.36
18.09
15.12
12.84
Br
H3C
O
O
O
Si
H3C
CH3
CH3
CH3
H3C
CH3
Cl
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

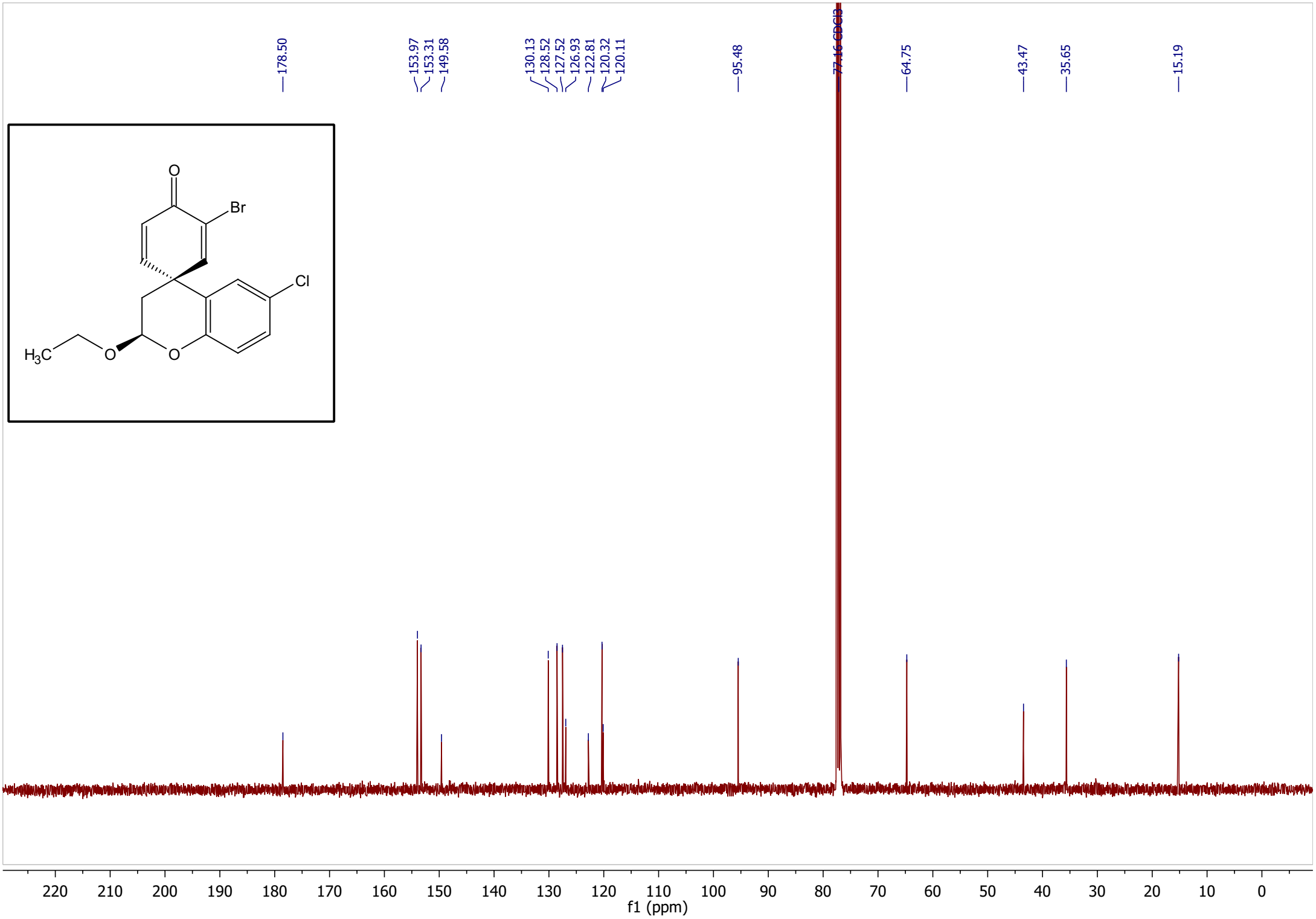


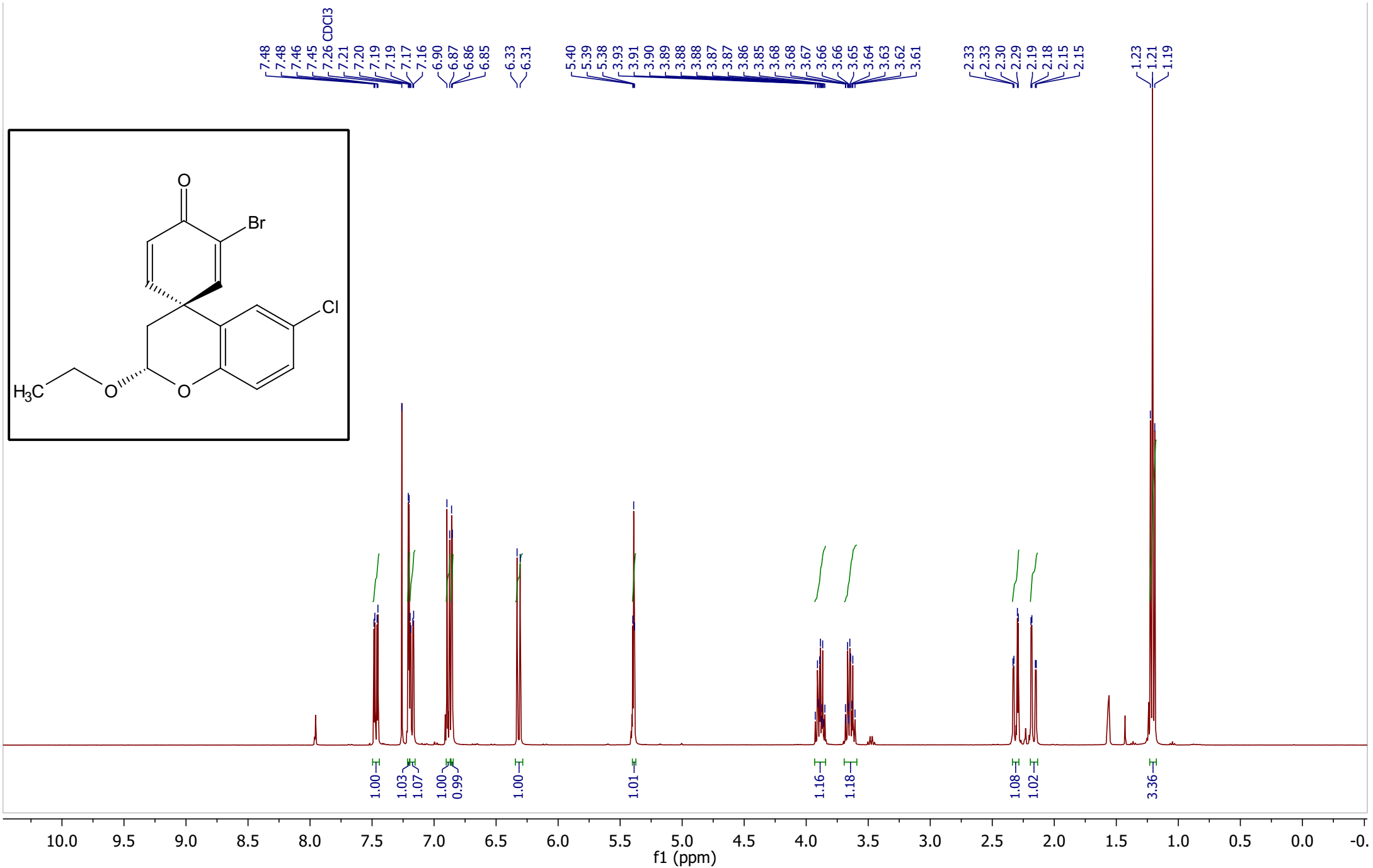
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.43 (dd, *J* = 10.2, 3.0 Hz, 1H), 7.15 (dd, *J* = 8.7, 2.7 Hz, 1H), 6.87 (d, *J* = 8.7 Hz, 1H), 6.86 (d, *J* = 2.7 Hz, 1H), 6.75 (dd, *J* = 10.0, 3.0 Hz, 1H), 6.37 (dd, *J* = 10.0, 1.9 Hz, 1H), 6.23 (dd, *J* = 10.2, 1.9 Hz, 1H), 5.38 (dd, *J* = 3.0, 3.0 Hz, 1H), 3.90 (dq, *J* = 9.7, 7.1 Hz, 1H), 3.64 (dq, *J* = 9.7, 7.1 Hz, 1H), 2.26 (dd, *J* = 14.1, 3.0 Hz, 1H), 2.13 (dd, *J* = 14.1, 3.0 Hz, 1H), 1.21 (dd, *J* = 7.1, 7.1 Hz, 3H).


185.69
153.91
153.24
149.79
129.65
128.96
128.64
126.70
126.58
121.57
120.06
95.84
77.16 CDCl3
64.67
40.50
36.13
15.24
O
Cl
H3C
O
O
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

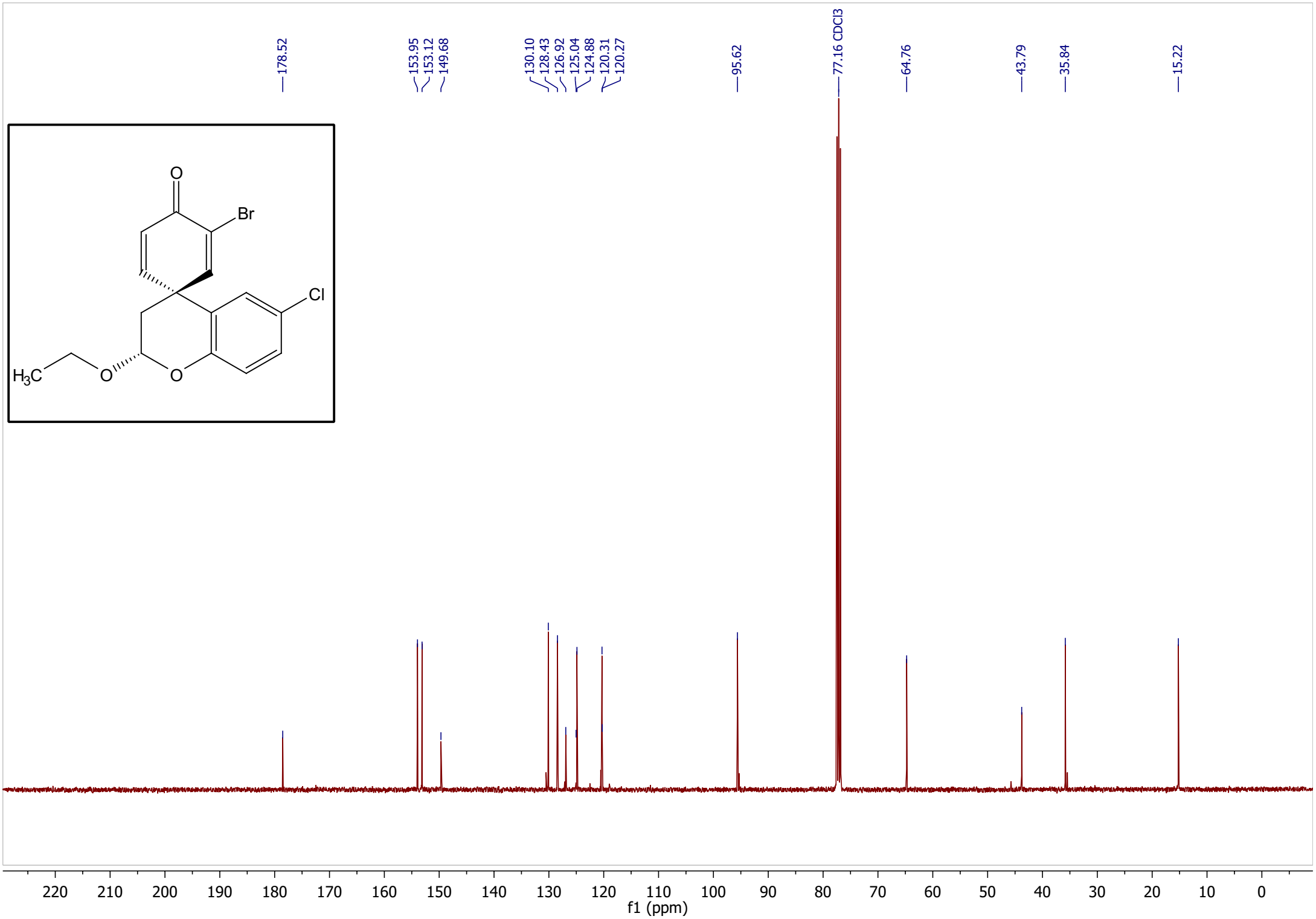


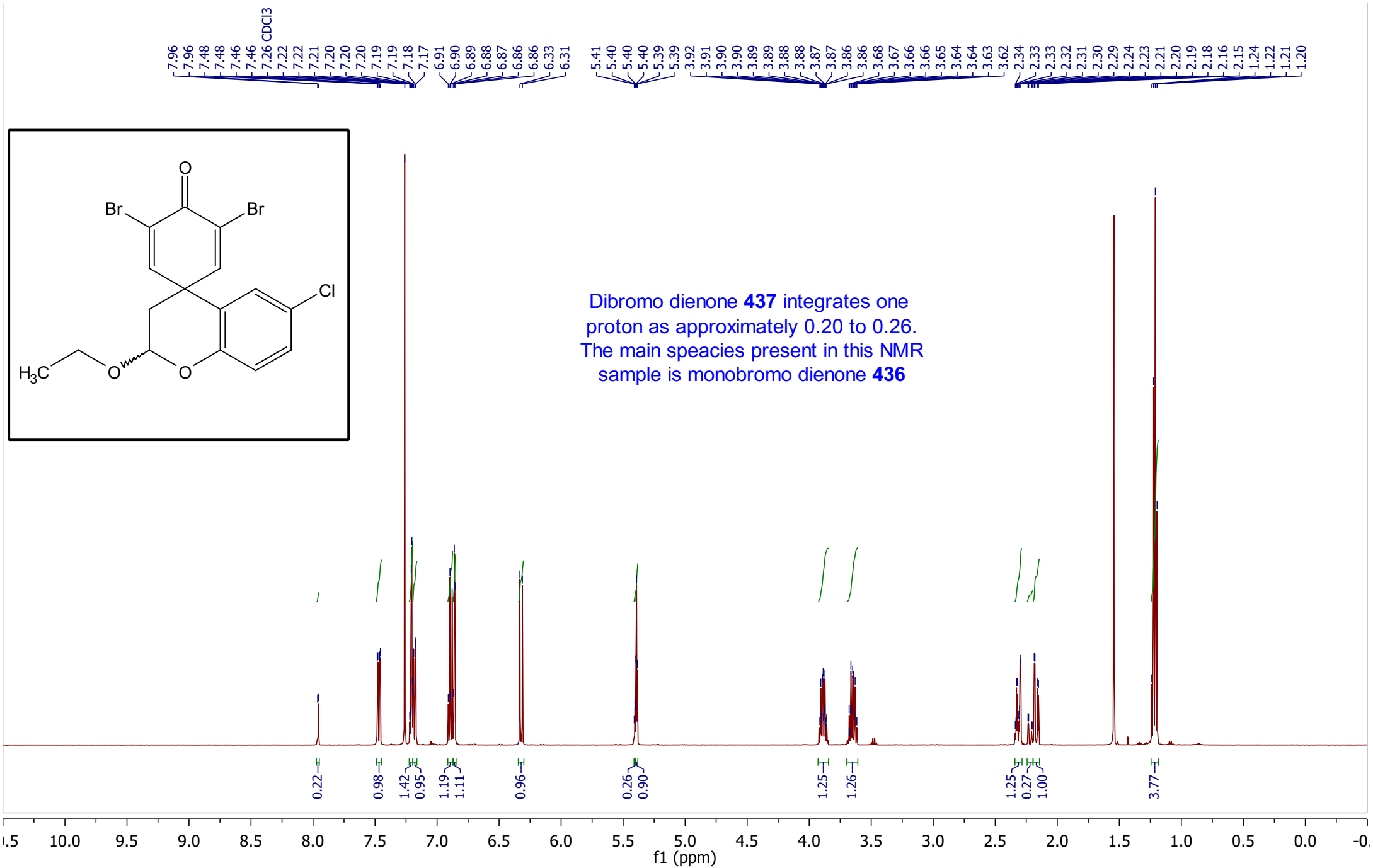
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.94 (d, *J* = 2.7 Hz, 1H), 7.18 (dd, *J* = 8.7, 2.5 Hz, 1H), 6.89 (d, *J* = 8.7 Hz, 1H), 6.86 (d, *J* = 2.5 Hz, 1H), 6.76 (dd, *J* = 9.8, 2.7 Hz, 1H), 6.48 (d, *J* = 9.8 Hz, 1H), 5.40 (dd, *J* = 2.9, 2.9 Hz, 1H), 3.89 (dq, *J* = 9.6, 7.1 Hz, 1H), 3.66 (dq, *J* = 9.7, 7.1 Hz, 1H), 2.28 (dd, *J* = 14.2, 2.9 Hz, 1H), 2.18 (dd, *J* = 14.2, 2.9 Hz, 1H), 1.23 (dd, *J* = 7.1, 7.1 Hz, 3H).


178.50
153.97
153.31
149.58
130.13
128.52
127.52
126.93
122.81
120.32
120.11
95.48
77.16 CDCl3
64.75
43.47
35.65
15.19
O
Br
Cl
H3C
O
O
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
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50
40
30
20
10
0
f1 (ppm)

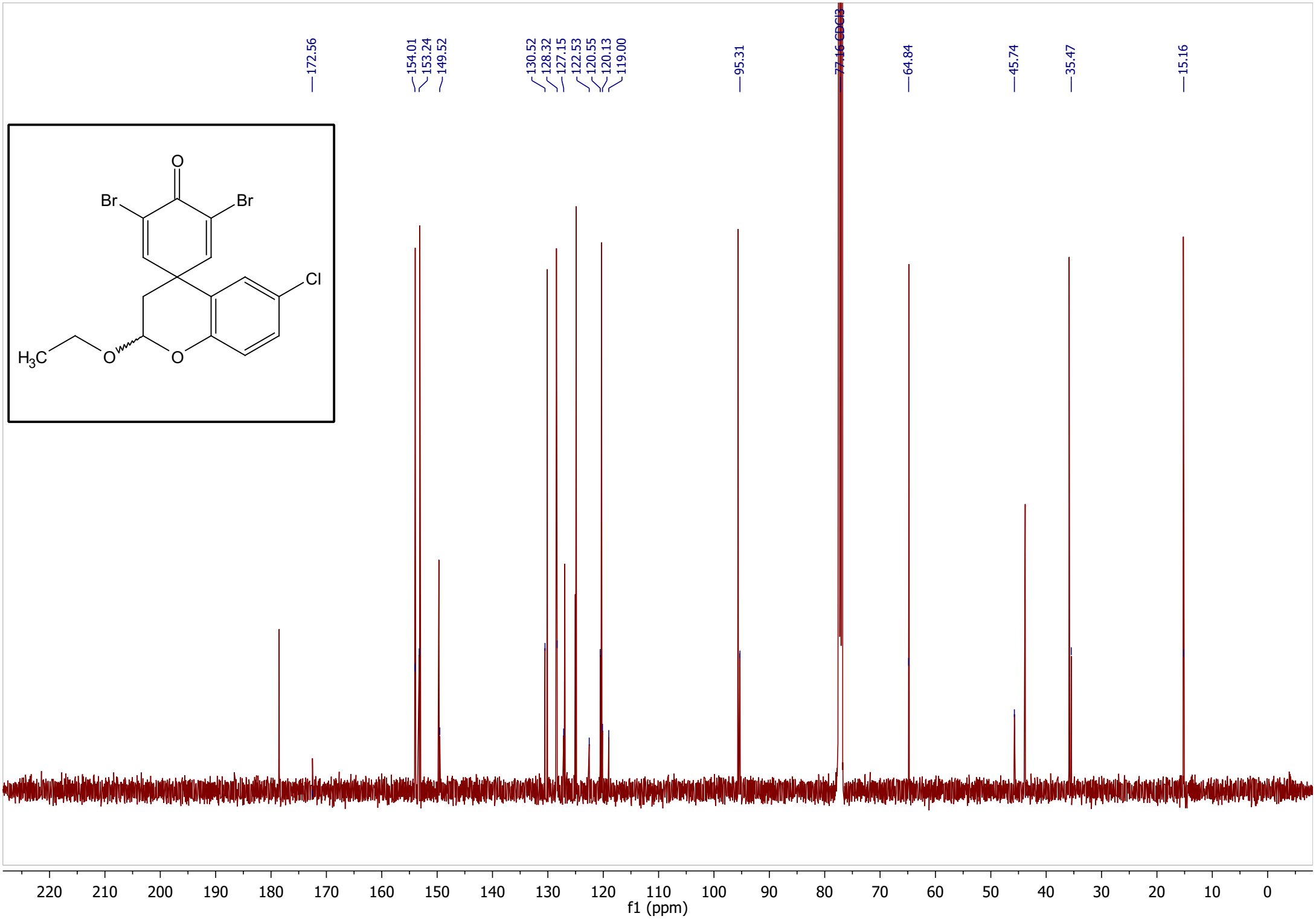


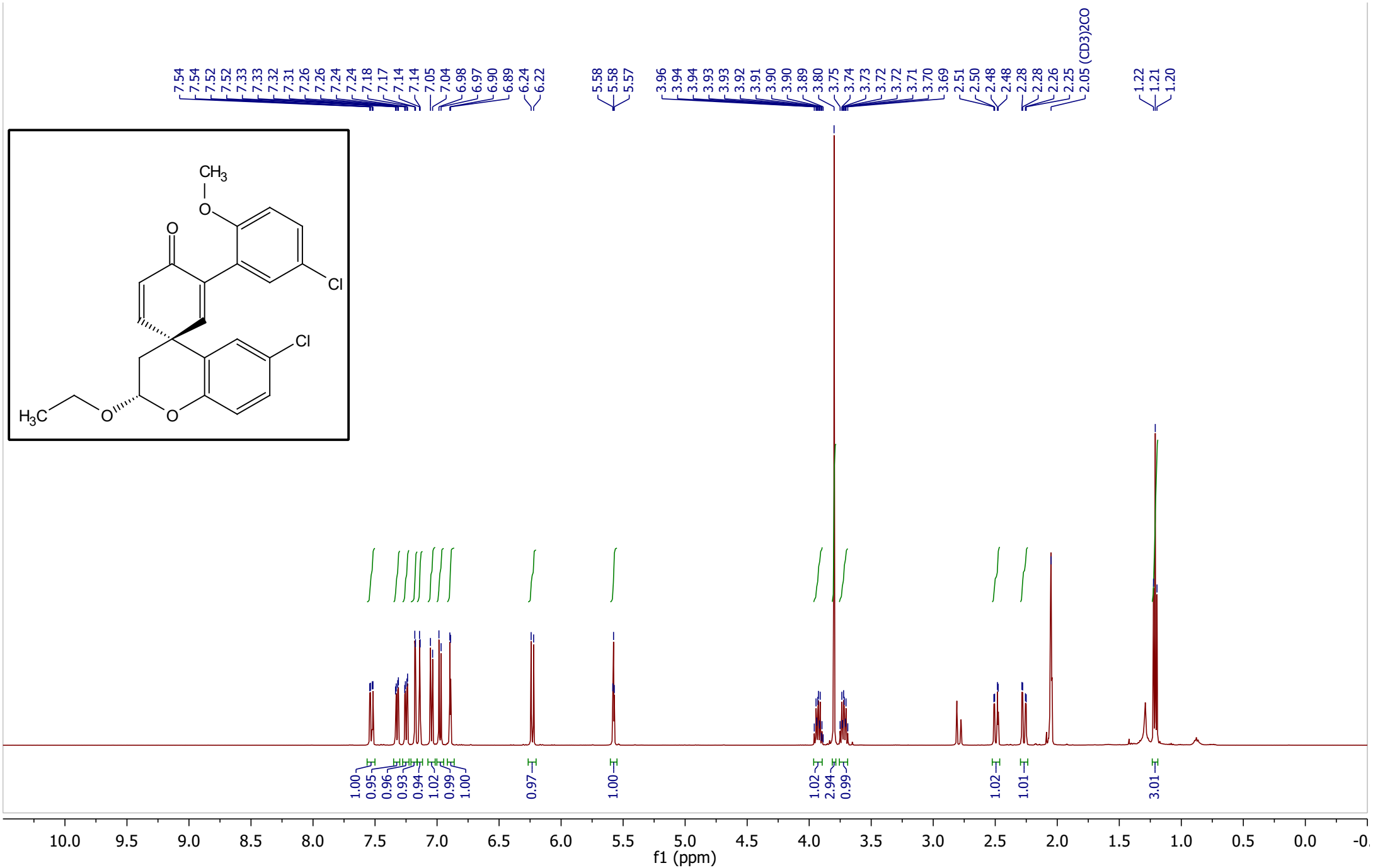
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.47 (dd, *J* = 10.0, 2.7 Hz, 1H), 7.20 (d, *J* = 2.7 Hz, 1H), 7.18 (dd, *J* = 8.7, 2.5 Hz, 1H), 6.89 (d, *J* = 8.7 Hz, 1H), 6.86 (d, *J* = 2.5 Hz, 1H), 6.34 (d, *J* = 10.0 Hz, 1H), 5.39 (dd, *J* = 3.0, 3.0 Hz, 1H), 3.88 (dq, *J* = 9.7, 7.1 Hz, 1H), 3.65 (dq, *J* = 9.7, 7.1 Hz, 1H), 2.31 (dd, *J* = 14.2, 3.0 Hz, 1H), 2.17 (dd, *J* = 14.2, 3.0 Hz, 1H), 1.21 (dd, *J* = 7.1, 7.1 Hz, 3H).


178.52
153.95
153.12
149.68
130.10
128.43
126.92
125.04
124.88
120.31
120.27
95.62
77.16 CDCl3
64.76
43.79
35.84
15.22
O
Br
Cl
H3C
O
O
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

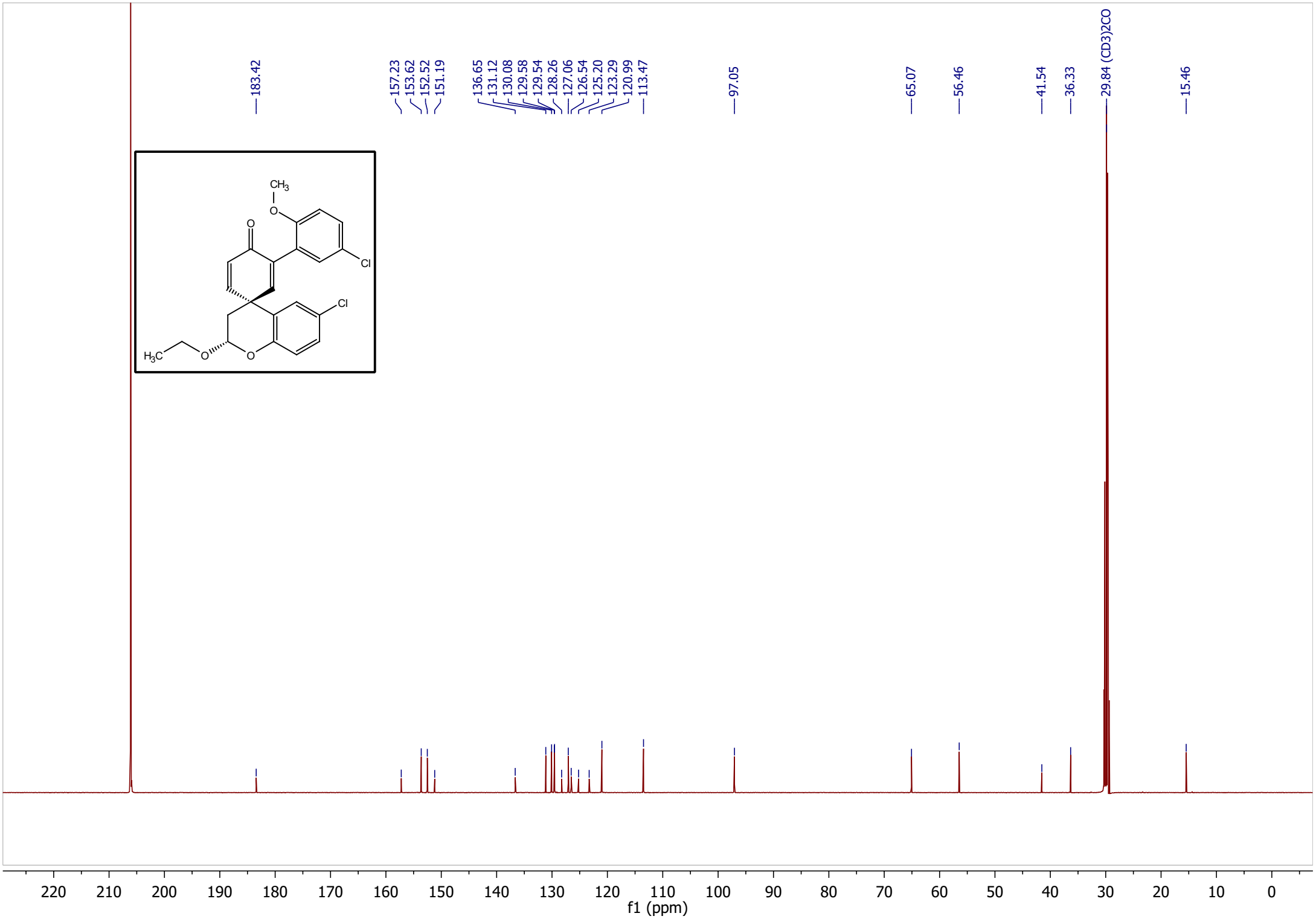


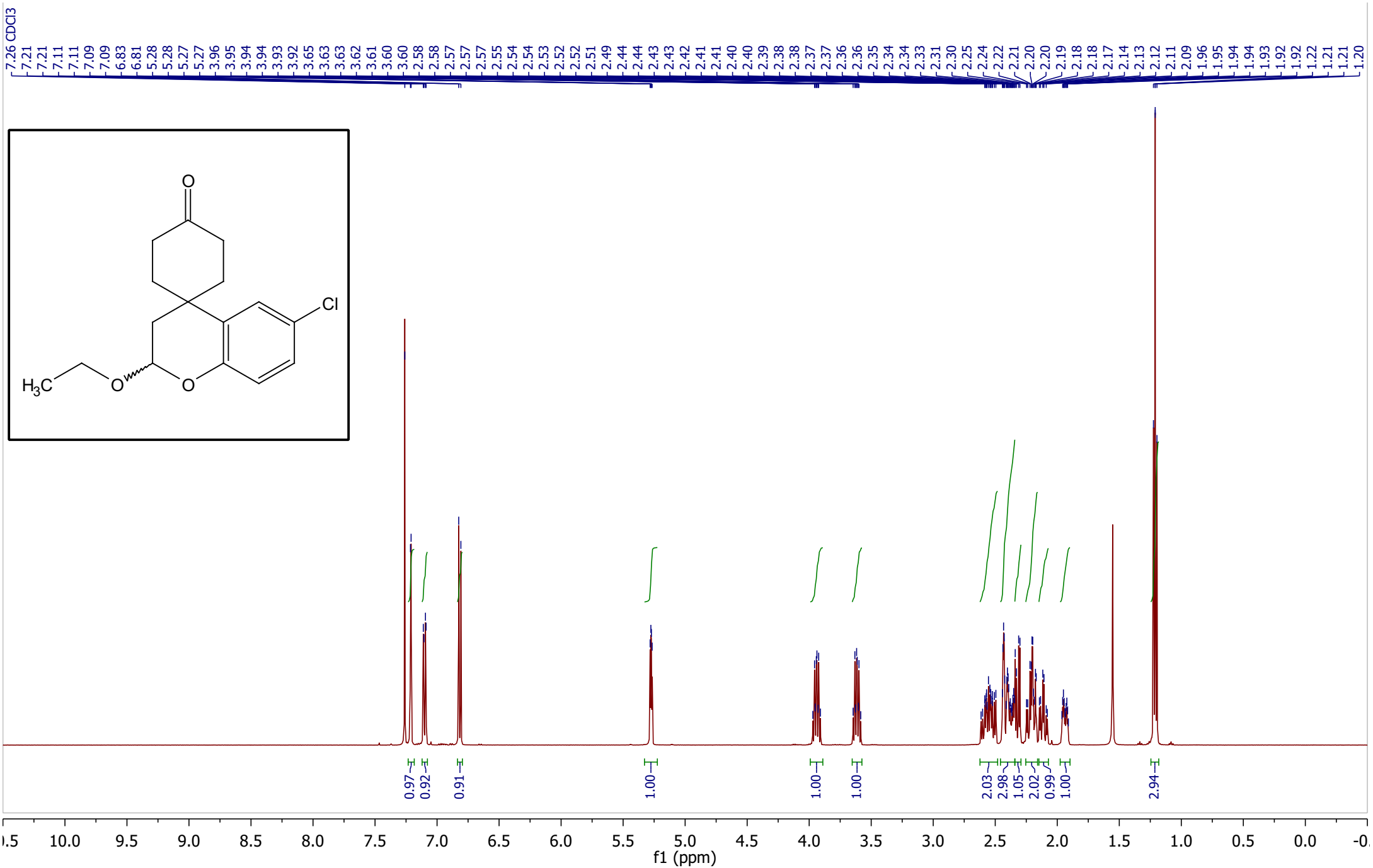
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.96 (d, *J* = 2.6 Hz, 1H), 7.21 (dd, ~*J* = 8.8, 2.5 Hz, 1H), 7.20 (d, ~*J* = 2.6 Hz, 1H), 6.90 (d, ~*J* = 8.8 Hz, 1H), 6.86 (d, ~*J* = 2.5 Hz, 1H), 5.40 (dd, ~*J* = 2.9, 2.9 Hz, 1H), 3.88 (dq, ~*J* = 9.7, 7.1 Hz, 1H), 3.66 (dq, ~*J* = 9.7, 7.1 Hz, 1H), 2.32 (dd, ~*J* = 14.2, 2.9 Hz, 1H), 2.22 (dd, ~*J* = 14.2, 2.9 Hz, 1H), 1.22 (dd, ~*J* = 7.1, 7.1 Hz, 3H).


172.56
154.01
153.24
149.52
130.52
128.32
127.15
122.53
120.55
120.13
119.00
95.31
77.16 CDCl3
64.84
45.74
35.47
15.16
Br
Br
O
Cl
H3C
O
O
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

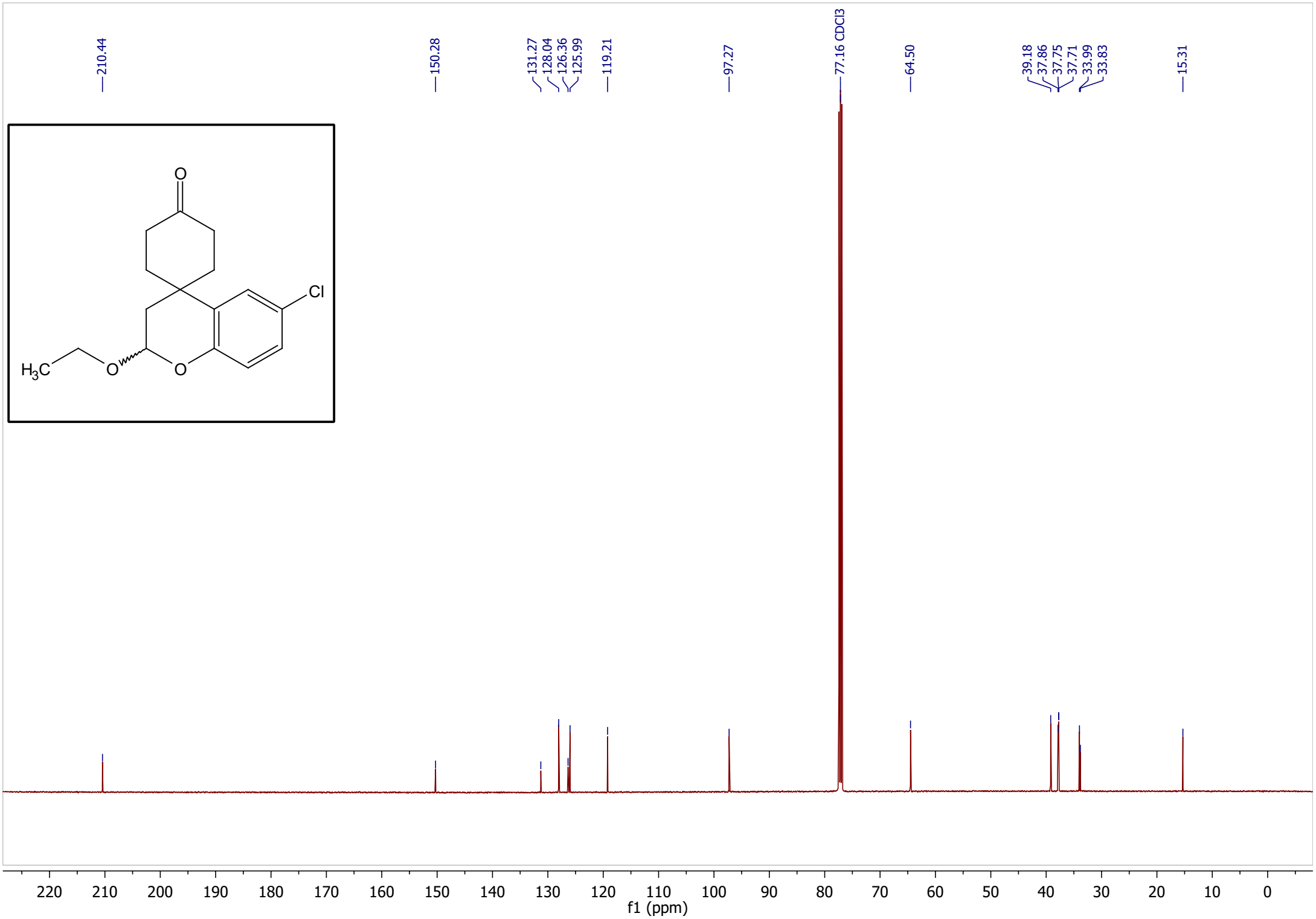


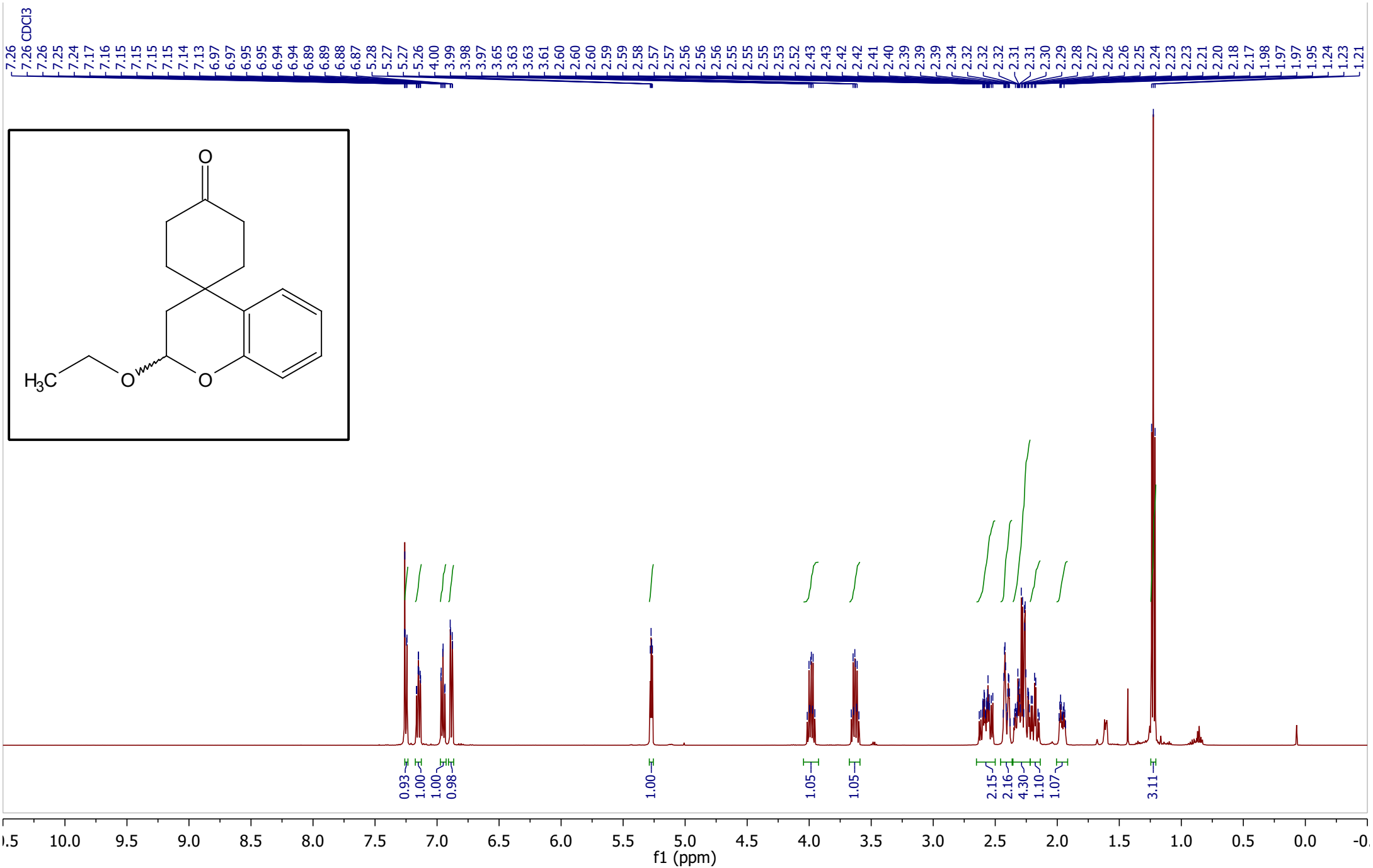
^{1}H NMR (500 MHz, Acetone-d_6) δ 7.53 (dd, J = 10.1, 3.0 Hz, 1H), 7.32 (dd, J = 8.8, 2.7 Hz, 1H), 7.25 (dd, J = 8.7, 2.6 Hz, 1H), 7.18 (d, J = 2.7 Hz, 1H), 7.14 (d, J = 2.6 Hz, 1H), 7.04 (d, J = 8.8 Hz, 1H), 6.98 (d, J = 8.7 Hz, 1H), 6.89 (d, J = 3.0 Hz, 1H), 6.23 (d, J = 10.1 Hz, 1H), 5.58 (dd, J = 3.0, 3.0 Hz, 1H), 3.93 (dq, J = 9.7, 7.1 Hz, 1H), 3.80 (s, 3H), 3.72 (dq, J = 9.7, 7.1 Hz, 1H), 2.49 (dd, J = 14.2, 3.0 Hz, 1H), 2.27 (dd, J = 14.2, 3.0 Hz, 1H), 1.21 (dd, J = 7.1, 7.1 Hz, 3H).


183.42
157.23
153.62
152.52
151.19
136.65
131.12
130.08
129.58
129.54
128.26
127.06
126.54
125.20
123.29
120.99
113.47
97.05
65.07
56.46
41.54
36.33
29.84 (CD3)2CO
15.46
CH3
O
O
Cl
Cl
H3C
O
O
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

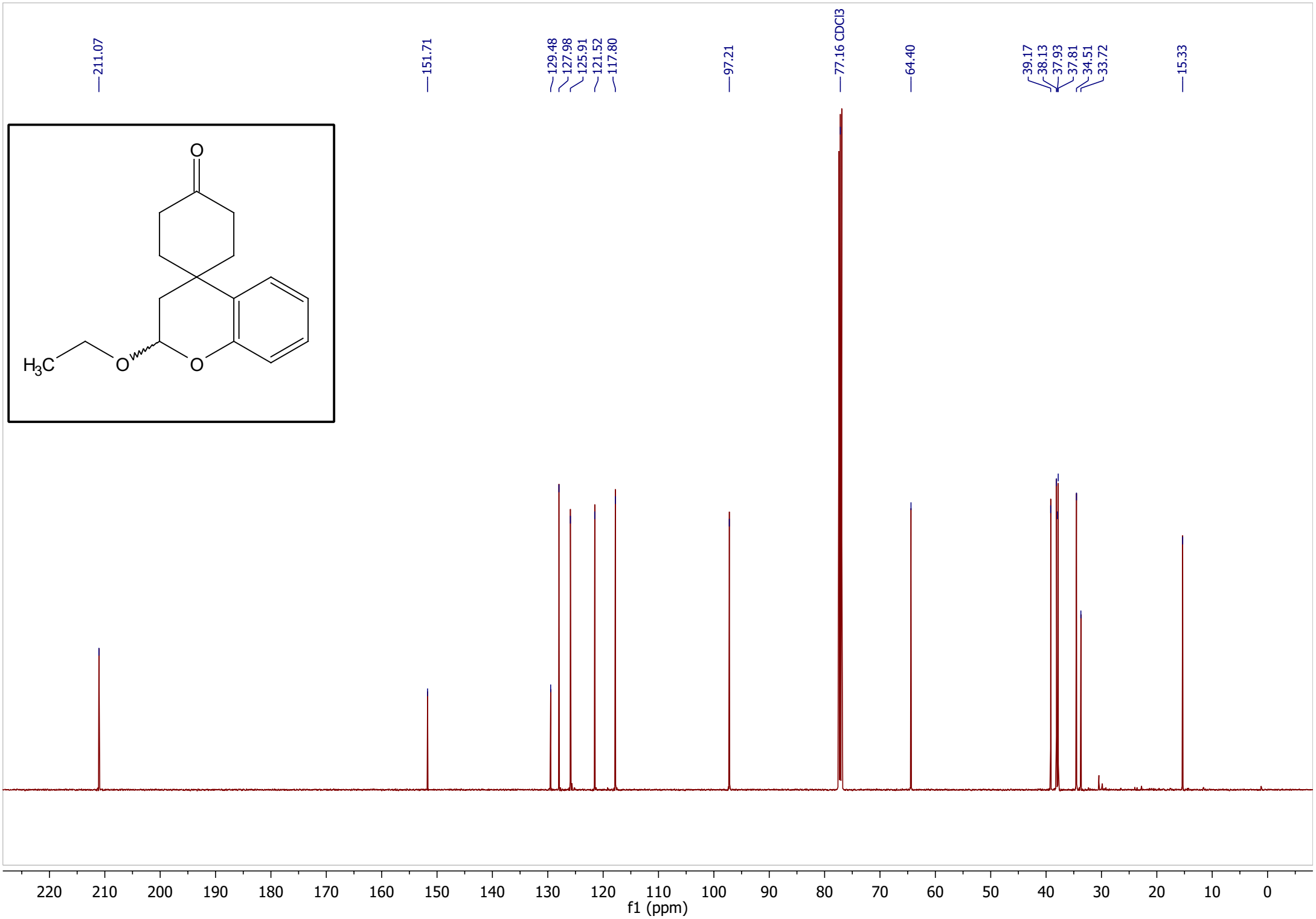


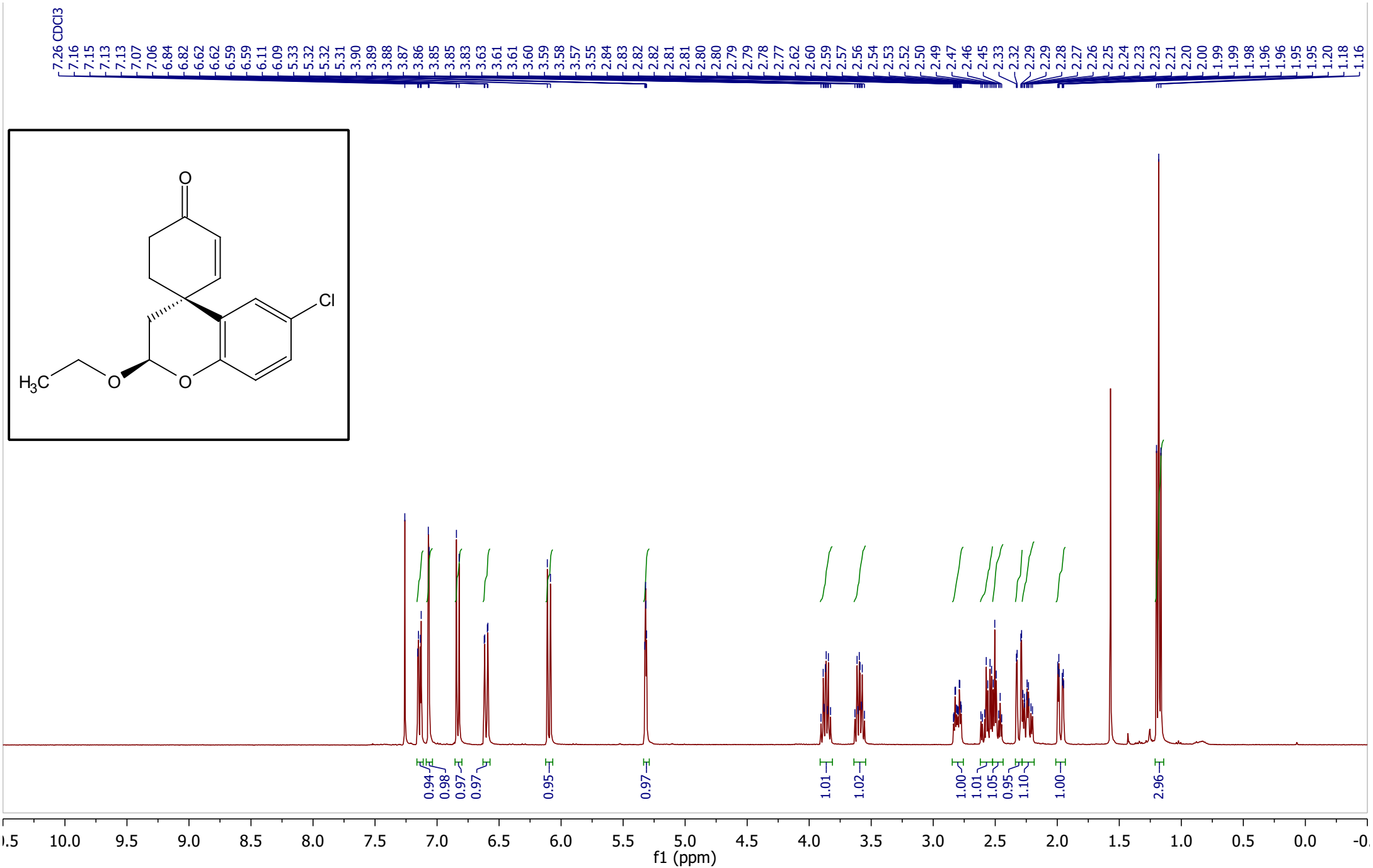
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.21 (d, *J* = 2.5 Hz, 1H), 7.10 (dd, *J* = 8.7, 2.5 Hz, 1H), 6.82 (d, *J* = 8.7 Hz, 1H), 5.27 (dd, *J* = 5.2, 3.0 Hz, 1H), 3.94 (dq, *J* = 9.6, 7.1 Hz, 1H), 3.62 (dq, *J* = 9.6, 7.1 Hz, 1H), 2.62 – 2.49 (m, 2H), 2.45 – 2.34 (m, 3H), 2.32 (dd, *J* = 14.0, 5.2 Hz, 1H), 2.22 (ddd, *J* = 13.8, 13.8, 4.9 Hz, 1H), 2.19 (dd, *J* = 14.0, 3.0 Hz, 1H), 2.11 (ddd, *J* = 13.8, 13.8, 4.6 Hz, 1H), 1.94 (dddd, *J* = 13.8, 6.1, 3.0, 3.0 Hz, 1H), 1.21 (dd, *J* = 7.1, 7.1 Hz, 3H).


210.44
150.28
131.27
128.04
126.36
125.99
119.21
97.27
77.16 CDCl3
64.50
39.18
37.86
37.75
37.71
33.99
33.83
15.31
O
Cl
H3C
O
O
220
210
200
190
180
170
160
150
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130
120
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100
90
80
70
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50
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30
20
10
0
f1 (ppm)

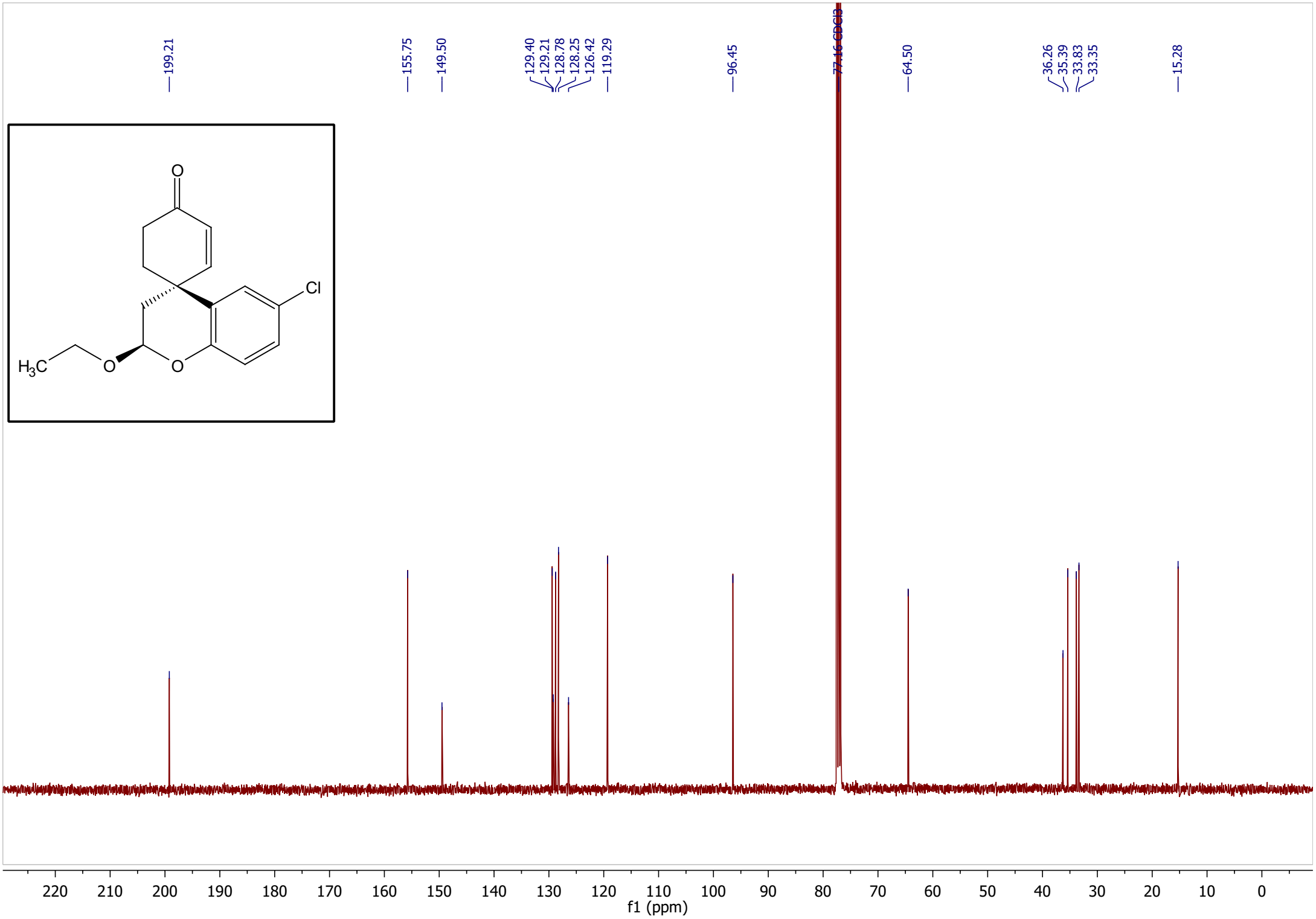


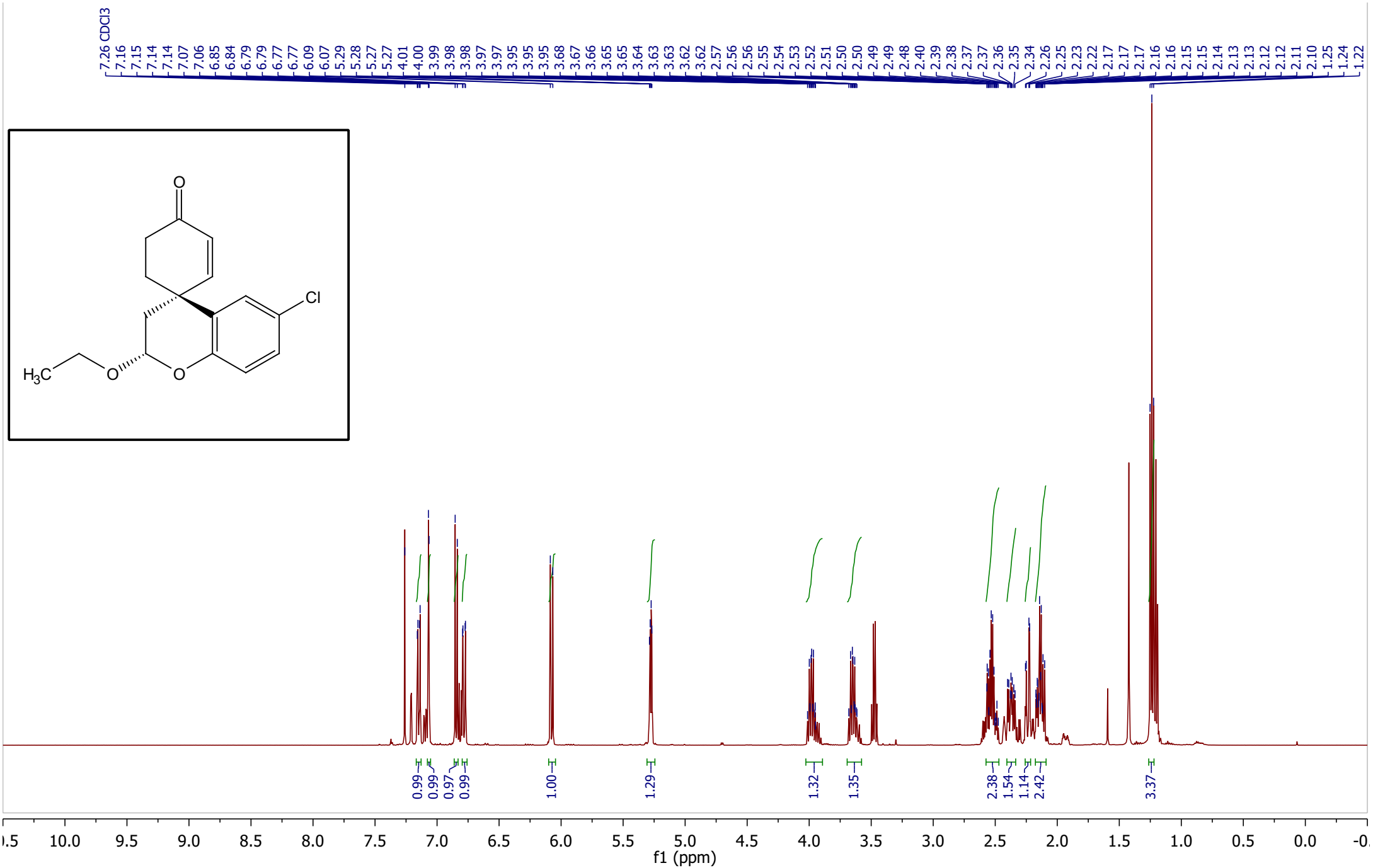
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.25 (dd, *J* = 8.1, 1.7 Hz, 1H), 7.15 (ddd, *J* = 8.1, 7.2, 1.7 Hz, 1H), 6.95 (ddd, *J* = 8.1, 7.2, 1.4 Hz, 1H), 6.88 (dd, *J* = 8.1, 1.4 Hz, 1H), 5.27 (dd, *J* = 5.4, 3.1 Hz, 1H), 3.98 (dq, *J* = 9.5, 7.1 Hz, 1H), 3.63 (dq, *J* = 9.5, 7.1 Hz, 1H), 2.65 – 2.50 (m, 2H), 2.45 – 2.37 (m, 2H), 2.36 – 2.22 (m, 4H), 2.18 (ddd, *J* = 13.8, 13.8, 4.6 Hz, 1H), 1.96 (dddd, *J* = 13.8, 6.1, 3.0, 3.0 Hz, 1H), 1.23 (dd, *J* = 7.1, 7.1 Hz, 3H).


211.07
151.71
129.48
127.98
125.91
121.52
117.80
97.21
77.16 CDCl3
64.40
39.17
38.13
37.93
37.81
34.51
33.72
15.33
O
H3C
O
O
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

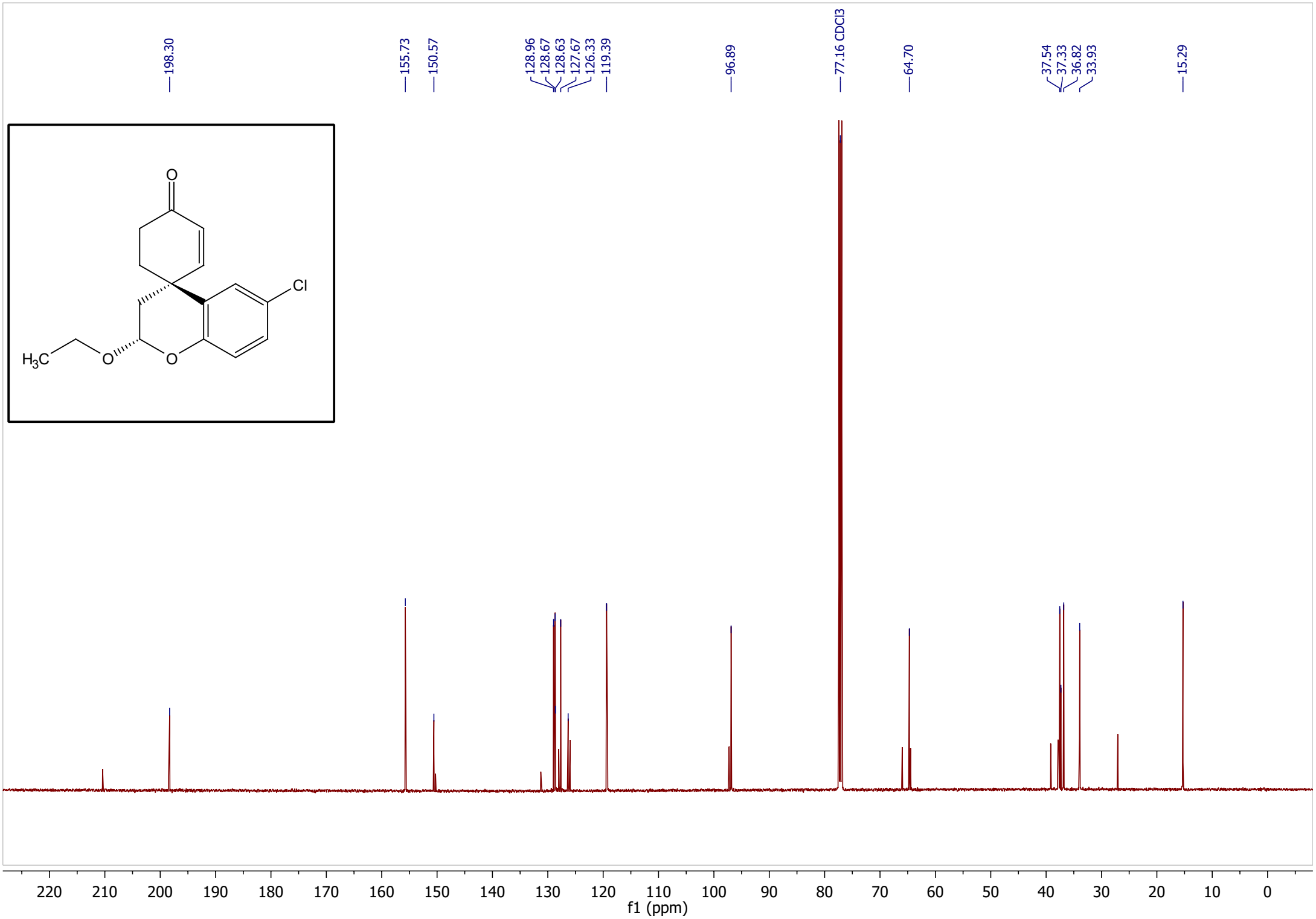


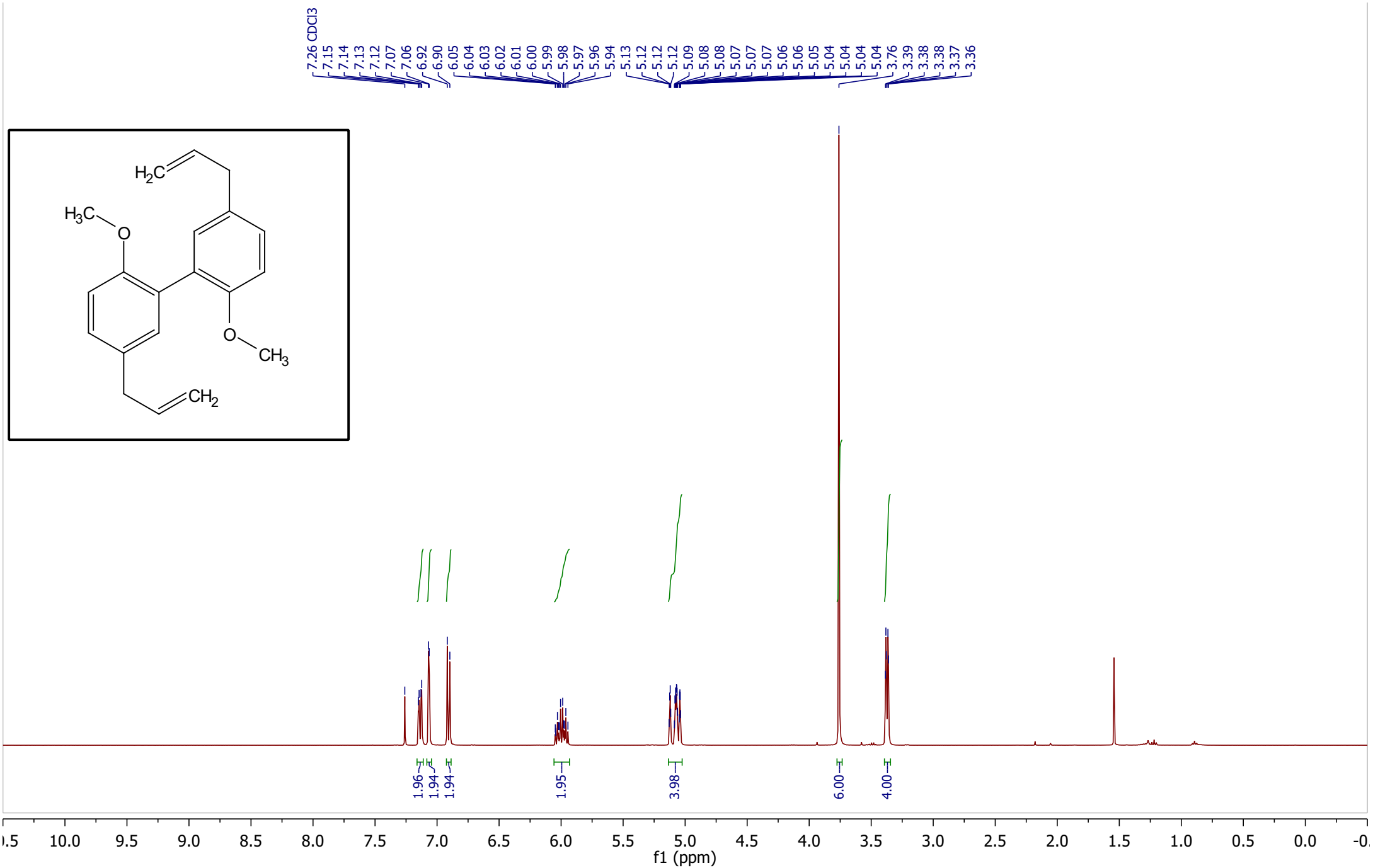
1H NMR (500 MHz, Chloroform-d) δ 7.13 (dd, J = 8.7, 2.5 Hz, 1H), 7.06 (d, J = 2.5 Hz, 1H), 6.83 (d, J = 8.7 Hz, 1H), 6.60 (dd, J = 10.1, 1.8 Hz, 1H), 6.09 (dd, J = 10.1, 0.8 Hz, 1H), 5.31 (dd, J = 3.4, 2.4 Hz, 1H), 3.86 (dq, J = 9.6, 7.1 Hz, 1H), 3.59 (dq, J = 9.6, 7.1 Hz, 1H), 2.80 (dddd, J = 13.7, 4.9, 4.8, 1.8 Hz, 1H), 2.56 (dddd, J = 17.5, 12.4, 4.9, 4.8 Hz, 1H), 2.48 (dddd, J = 17.5, 4.9, 4.8, 0.8 Hz, 1H), 2.30 (dd, J = 14.3, 2.4 Hz, 1H), 2.23 (ddddd, J = 13.7, 12.4, 4.9, 4.8, 1.1 Hz, 1H), 1.97 (ddd, J = 14.3, 3.4, 1.1 Hz, 1H), 1.18 (dd, J = 7.1, 7.1 Hz, 3H).


199.21
155.75
149.50
129.40
129.21
128.78
128.25
126.42
119.29
96.45
77.16 CDCl3
64.50
36.26
35.39
33.83
33.35
15.28
O
Cl
H3C
O
O
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

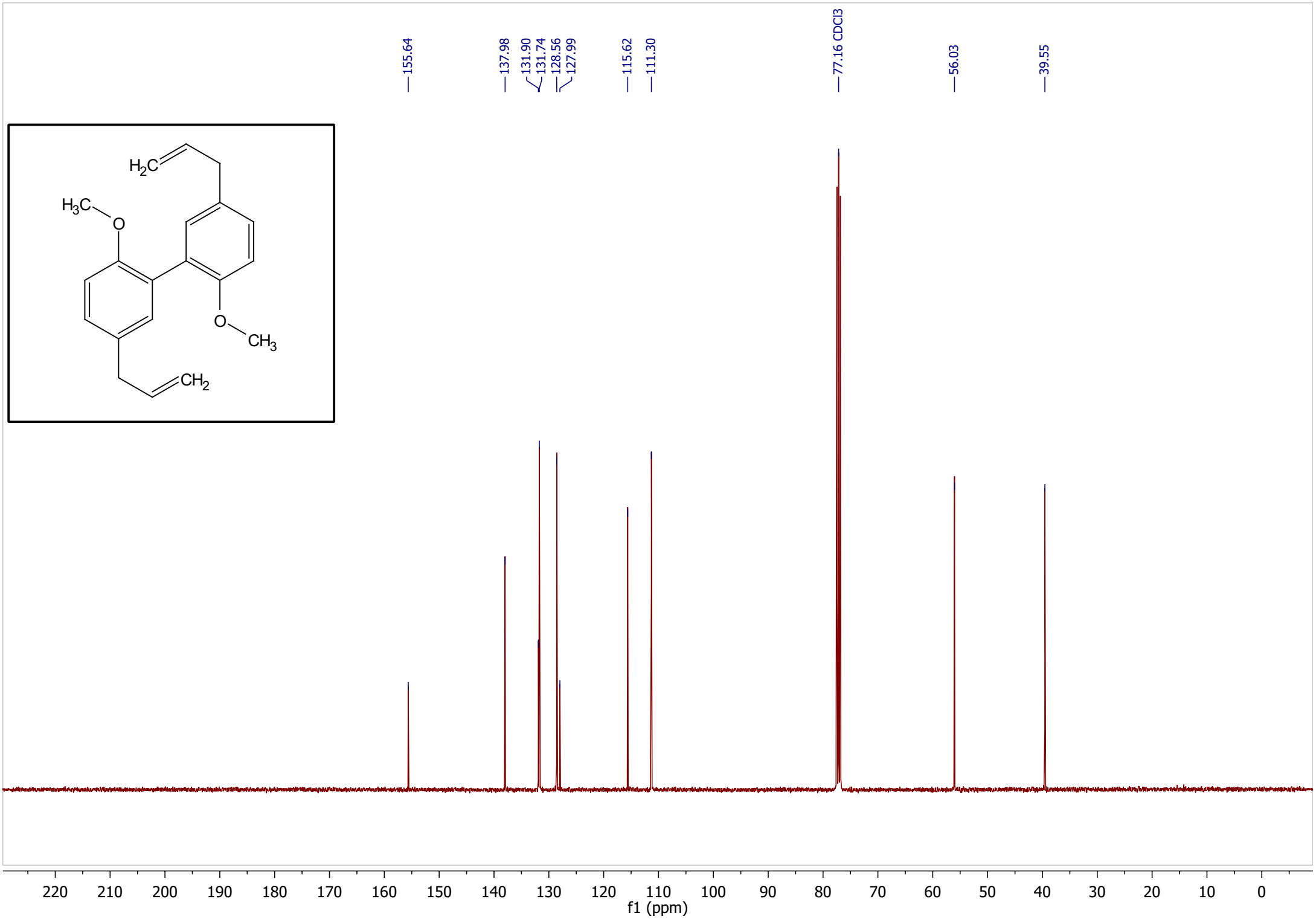


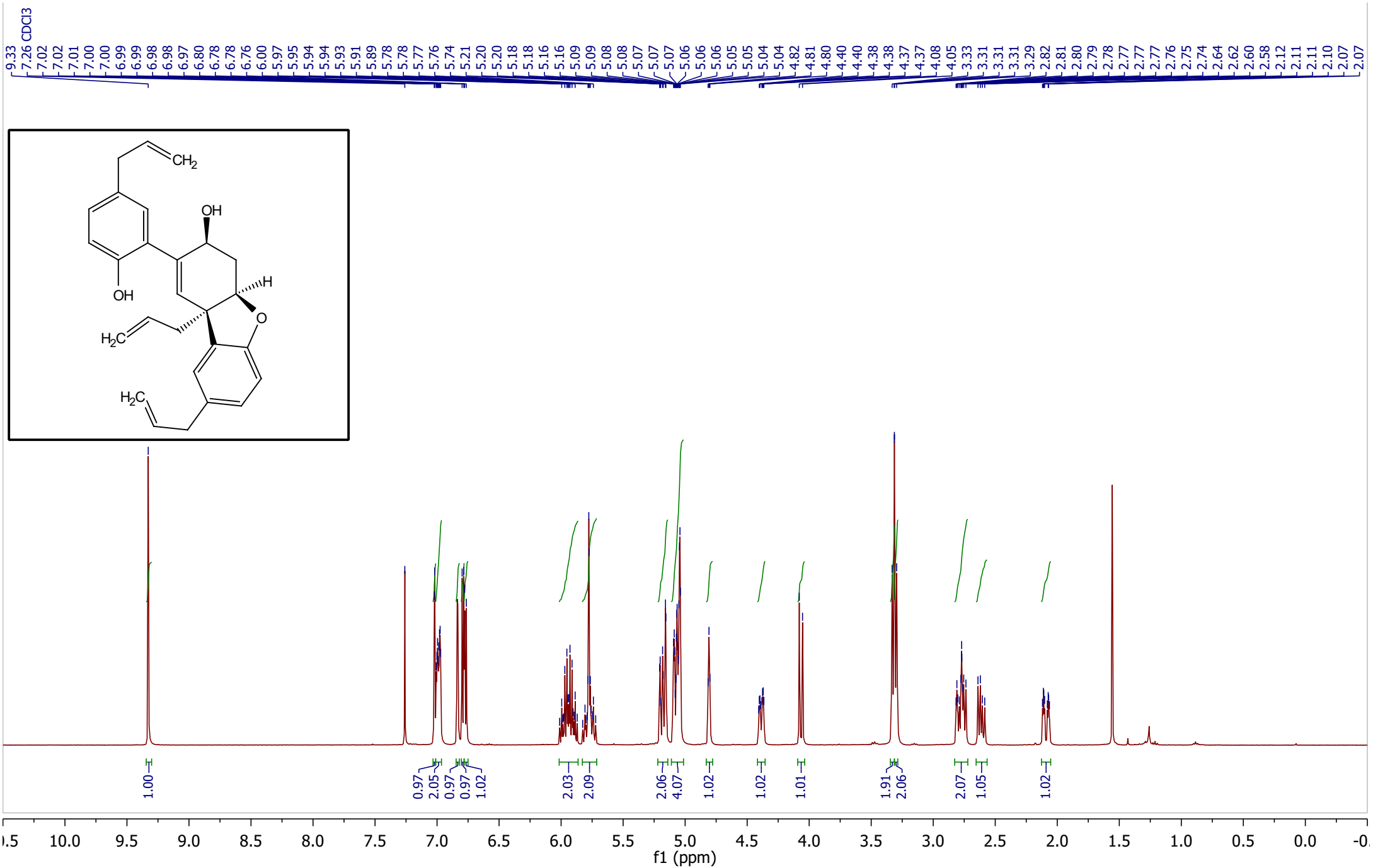
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.15 (dd, *J* = 8.7, 2.5 Hz, 1H), 7.07 (d, *J* = 2.5 Hz, 1H), 6.85 (d, *J* = 8.7 Hz, 1H), 6.78 (dd, *J* = 10.1, 1.4 Hz, 1H), 6.08 (d, *J* = 10.1 Hz, 1H), 5.28 (dd, *J* = 6.8, 2.9 Hz, 1H), 3.98 (dq, *J* = 9.5, 7.1 Hz, 1H), 3.65 (dq, *J* = 9.5, 7.1 Hz, 1H), 2.57 – 2.47 (m, 2H), 2.41 – 2.34 (m, 1H), 2.24 (dd, *J* = 13.8, 2.9 Hz, 1H), 2.17 – 2.09 (m, 2H), 1.24 (dd, *J* = 7.1, 7.1 Hz, 3H).


198.30
155.73
150.57
128.96
128.67
128.63
127.67
126.33
119.39
96.89
77.16 CDCl3
64.70
37.54
37.33
36.82
33.93
15.29
O
Cl
H3C
O
O
220
210
200
190
180
170
160
150
140
130
120
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90
80
70
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50
40
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20
10
0
f1 (ppm)

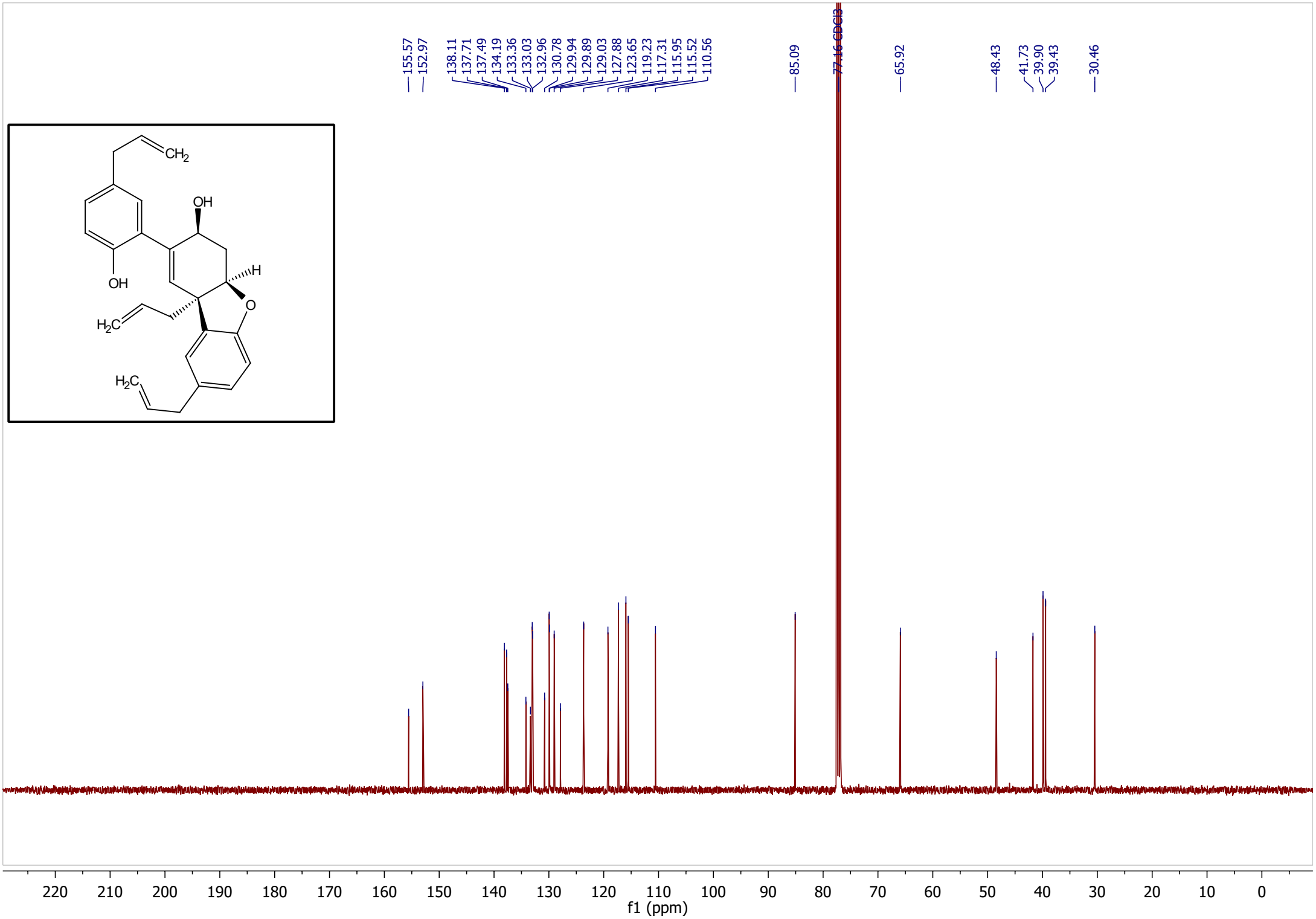


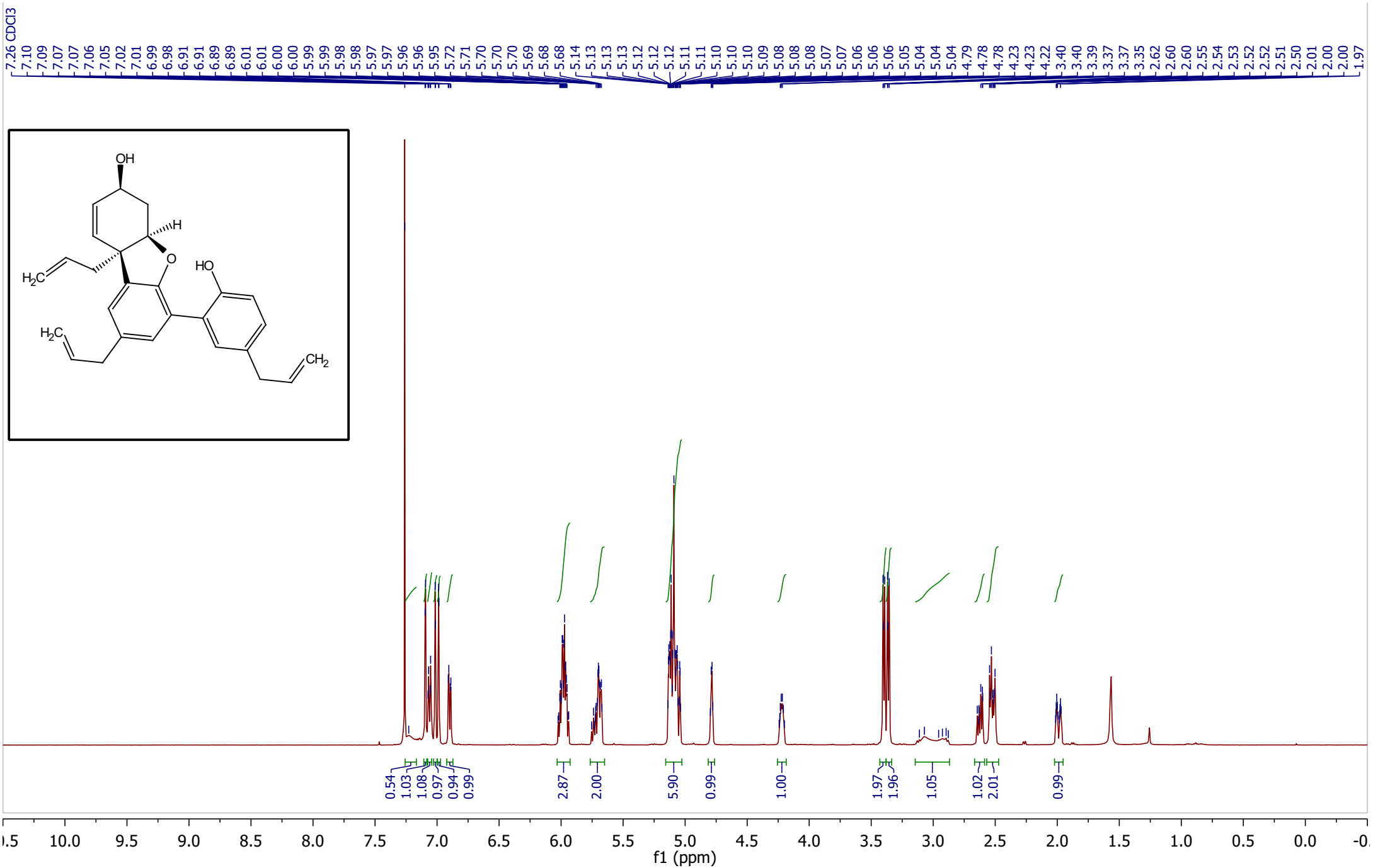
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.14 (dd, *J* = 8.3, 2.3 Hz, 2H), 7.07 (d, *J* = 2.3 Hz, 2H), 6.91 (d, *J* = 8.3 Hz, 2H), 6.00 (ddt, *J* = 16.8, 10.0, 6.7 Hz, 2H), 5.14 – 5.03 (m, 4H), 3.76 (s, 6H), 3.37 (ddd, *J* = 6.7, 1.5, 1.5 Hz, 4H).


155.64
137.98
131.90
131.74
128.56
127.99
115.62
111.30
77.16 CDCl3
56.03
39.55
H2C
H3C
O
O
CH3
CH2
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

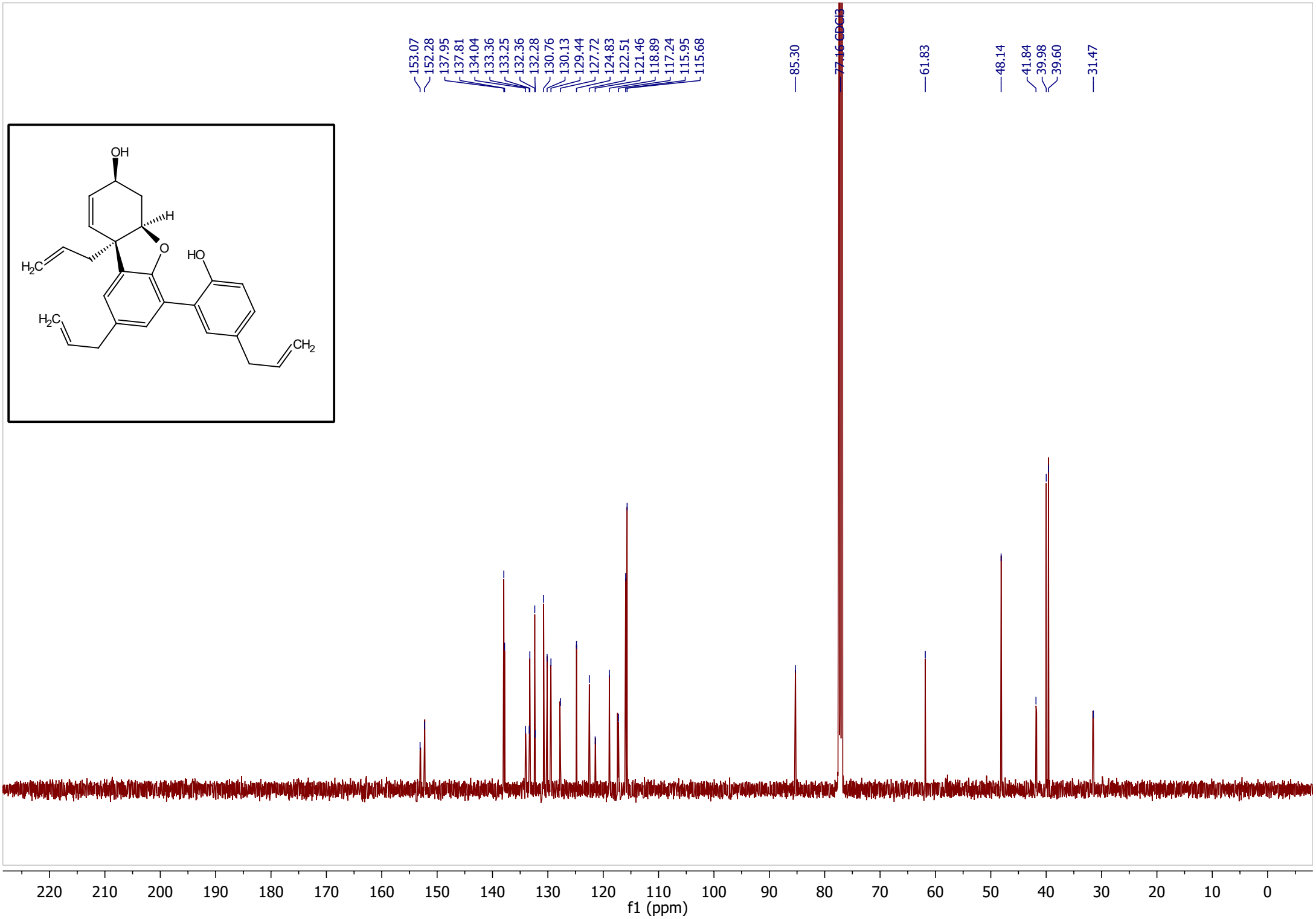


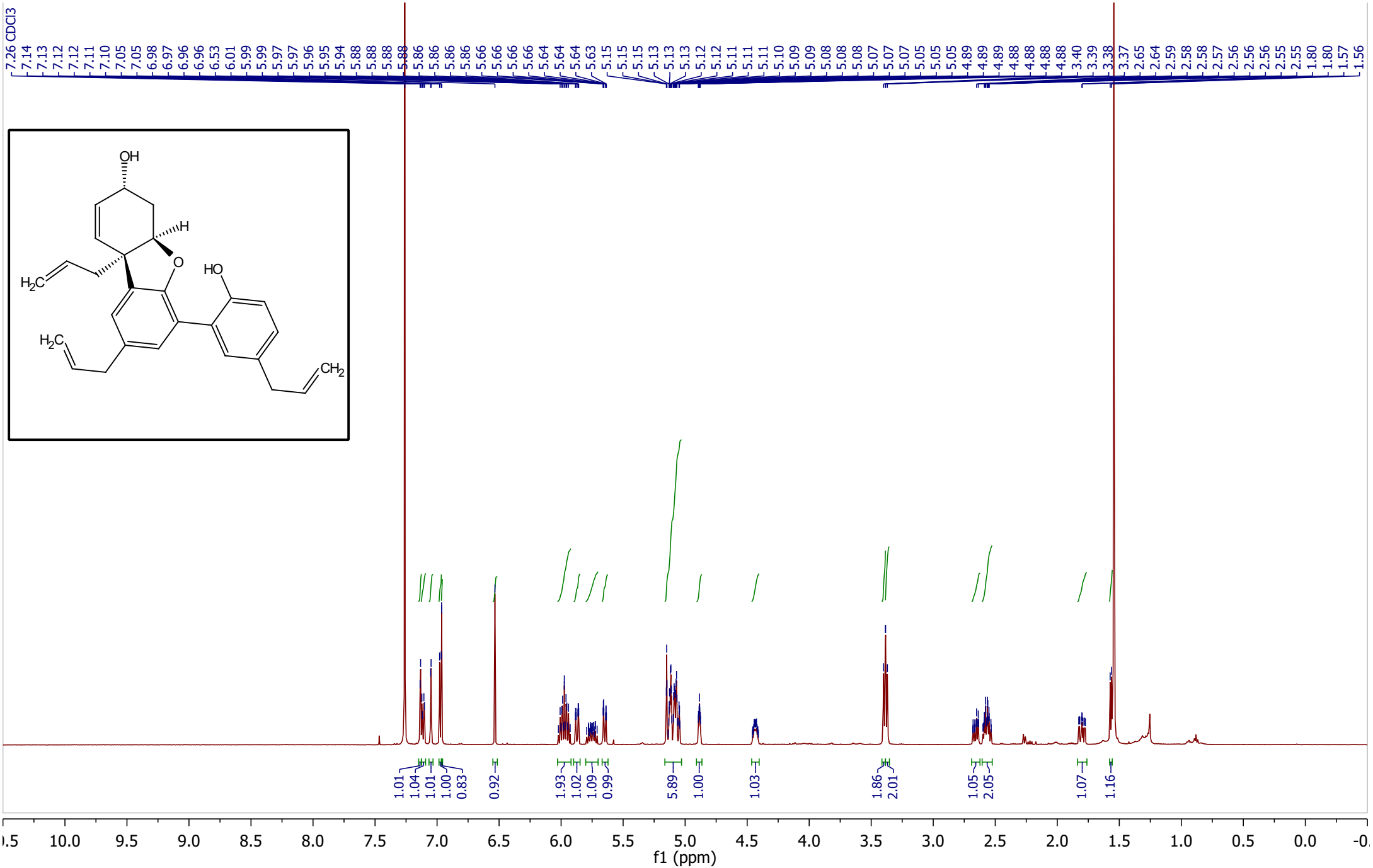
1H NMR (500 MHz, Chloroform-d) δ 9.33 (s, 1H), 7.02 (d, J = 1.8 Hz, 1H), 6.99 (dd, J = 8.2, 2.2 Hz, 1H), 6.98 (dd, J = 8.0, 1.8 Hz, 1H), 6.83 (d, J = 2.2 Hz, 1H), 6.79 (d, J = 8.2 Hz, 1H), 6.77 (d, J = 8.0 Hz, 1H), 6.01 – 5.87 (m, 2H), 5.78 (s, 1H), 5.83 – 5.71 (m, 1H), 5.21 – 5.15 (m, 2H), 5.11 – 5.02 (m, 4H), 4.83 – 4.78 (m, 1H), 4.42 – 4.35 (m, 1H), 4.07 (s, 1H), 3.32 (d, J = 7.1 Hz, 2H), 3.30 (d, J = 7.1 Hz, 2H), 2.79 (ddd, J = 15.5, 3.2, 2.1 Hz, 1H), 2.76 (dd, J = 14.1, 7.1 Hz, 1H), 2.61 (dd, J = 14.1, 7.1 Hz, 1H), 2.09 (ddd, J = 15.5, 4.1, 2.5 Hz, 1H).


155.57
152.97
138.11
137.71
137.49
134.19
133.36
133.03
132.96
130.78
129.94
129.89
129.03
127.88
123.65
119.23
117.31
115.95
115.52
110.56
85.09
77.16 CDCl3
65.92
48.43
41.73
39.90
39.43
30.46
OH
OH
H
O
CH2
H2C
H2C
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

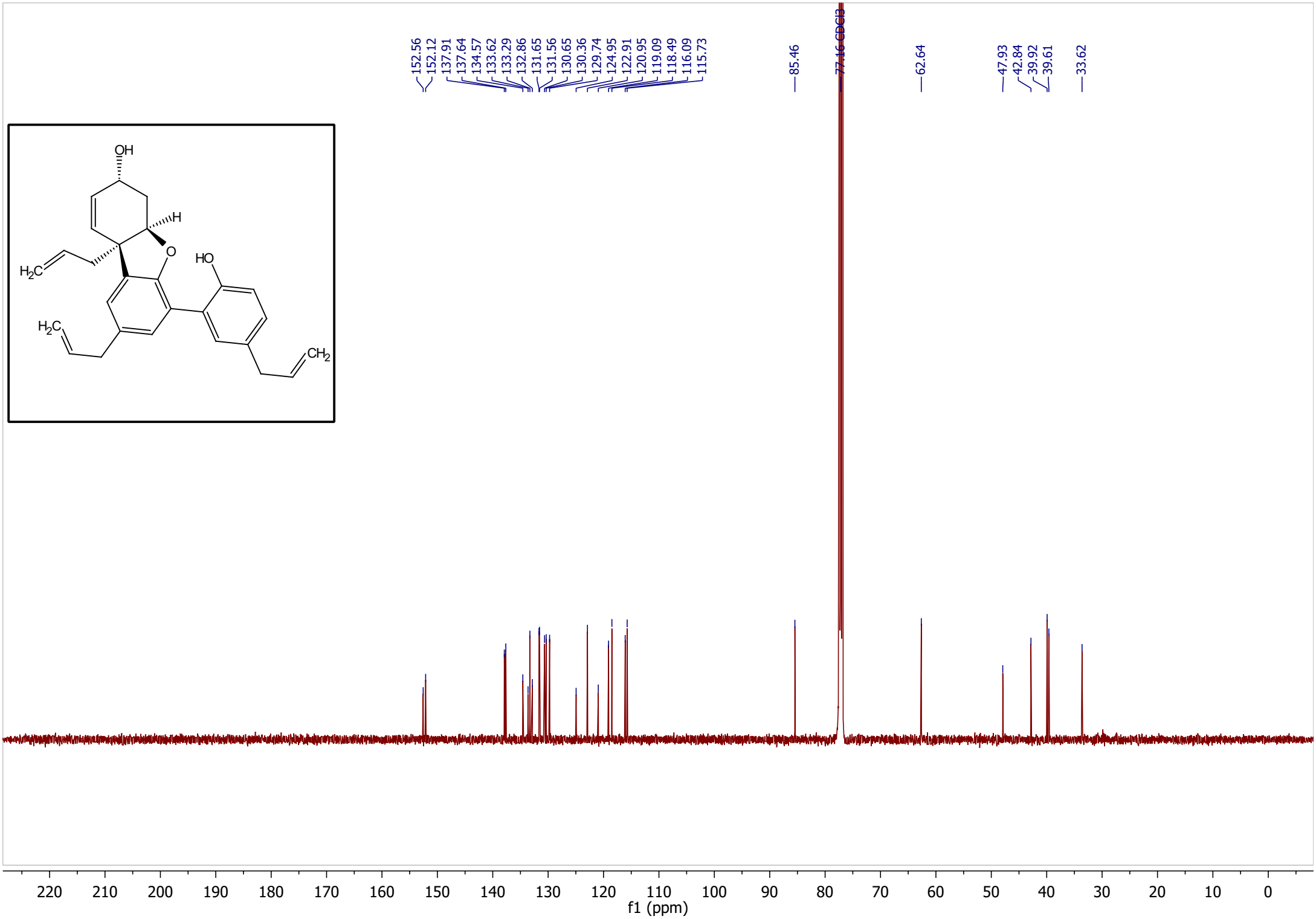


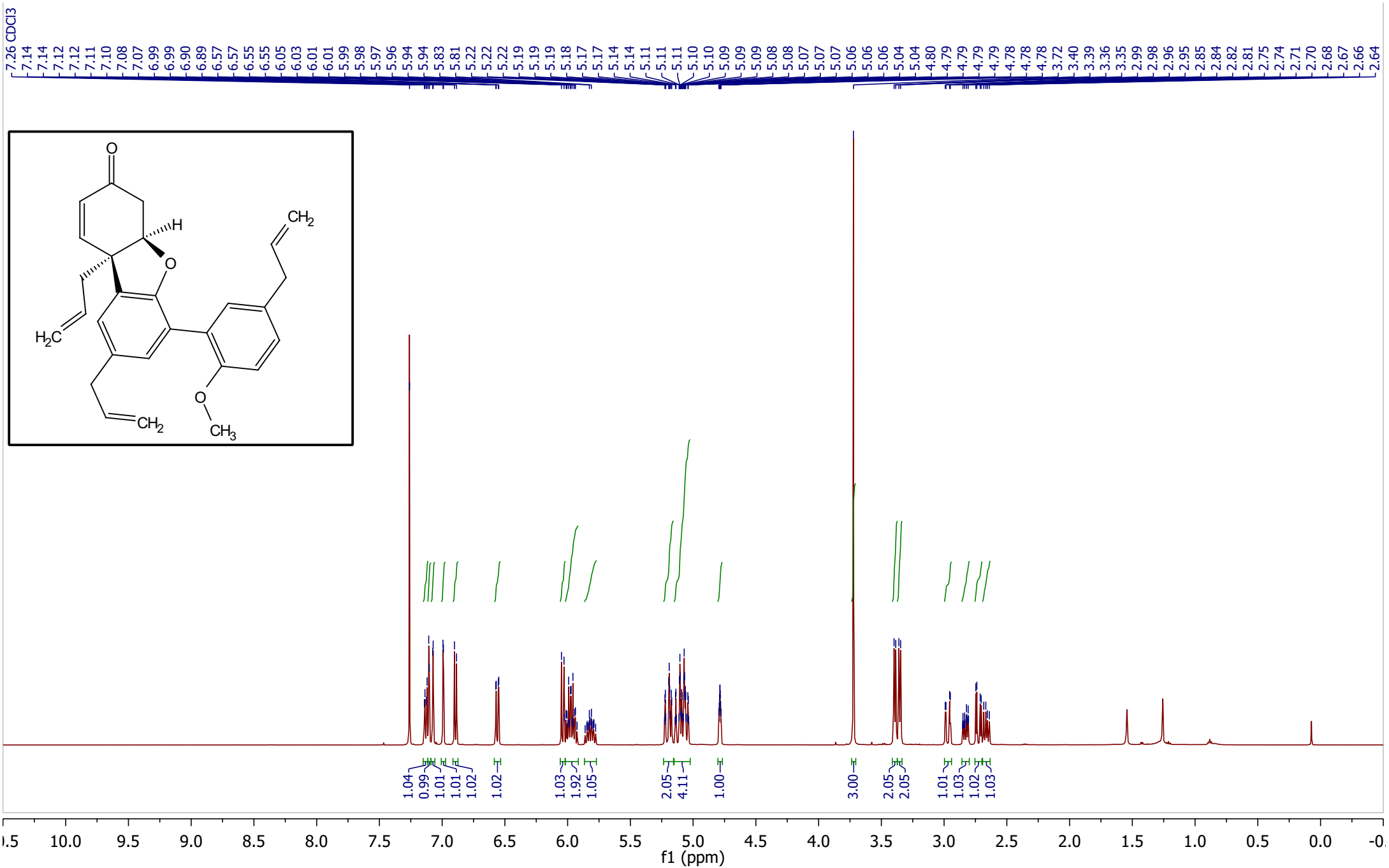
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.27 – 7.17 (m, 1H), 7.09 (d, *J* = 2.3 Hz, 1H), 7.06 (dd, *J* = 8.2, 2.3 Hz, 1H), 7.01 (d, *J* = 1.7 Hz, 1H), 6.98 (d, *J* = 1.7 Hz, 1H), 6.90 (d, *J* = 8.2 Hz, 1H), 5.98 (d, *J* = 10.0 Hz, 1H), 6.04 – 5.92 (m, 2H), 5.77 – 5.65 (m, 1H), 5.68 (d, *J* = 10.0 Hz, 1H), 5.15 – 5.03 (m, 6H), 4.81 – 4.76 (m, 1H), 4.25 – 4.18 (m, 1H), 3.40 (d, *J* = 6.6 Hz, 2H), 3.36 (d, *J* = 6.6 Hz, 2H), 3.15 – 2.85 (m, 1H), 2.62 (dddd, *J* = 14.2, 7.0, 1.4, 1.4 Hz, 1H), 2.57 – 2.49 (m, 2H), 1.98 (ddd, *J* = 15.1, 4.7, 2.9 Hz, 1H).


153.07
152.28
137.95
137.81
134.04
133.36
133.25
132.36
132.28
130.76
130.13
129.44
127.72
124.83
122.51
121.46
118.89
117.24
115.95
115.68
85.30
77.16 CDCl3
61.83
48.14
41.84
39.98
39.60
31.47
OH
H
O
HO
H2C
H2C
CH2
220
210
200
190
180
170
160
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100
90
80
70
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50
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30
20
10
0
f1 (ppm)

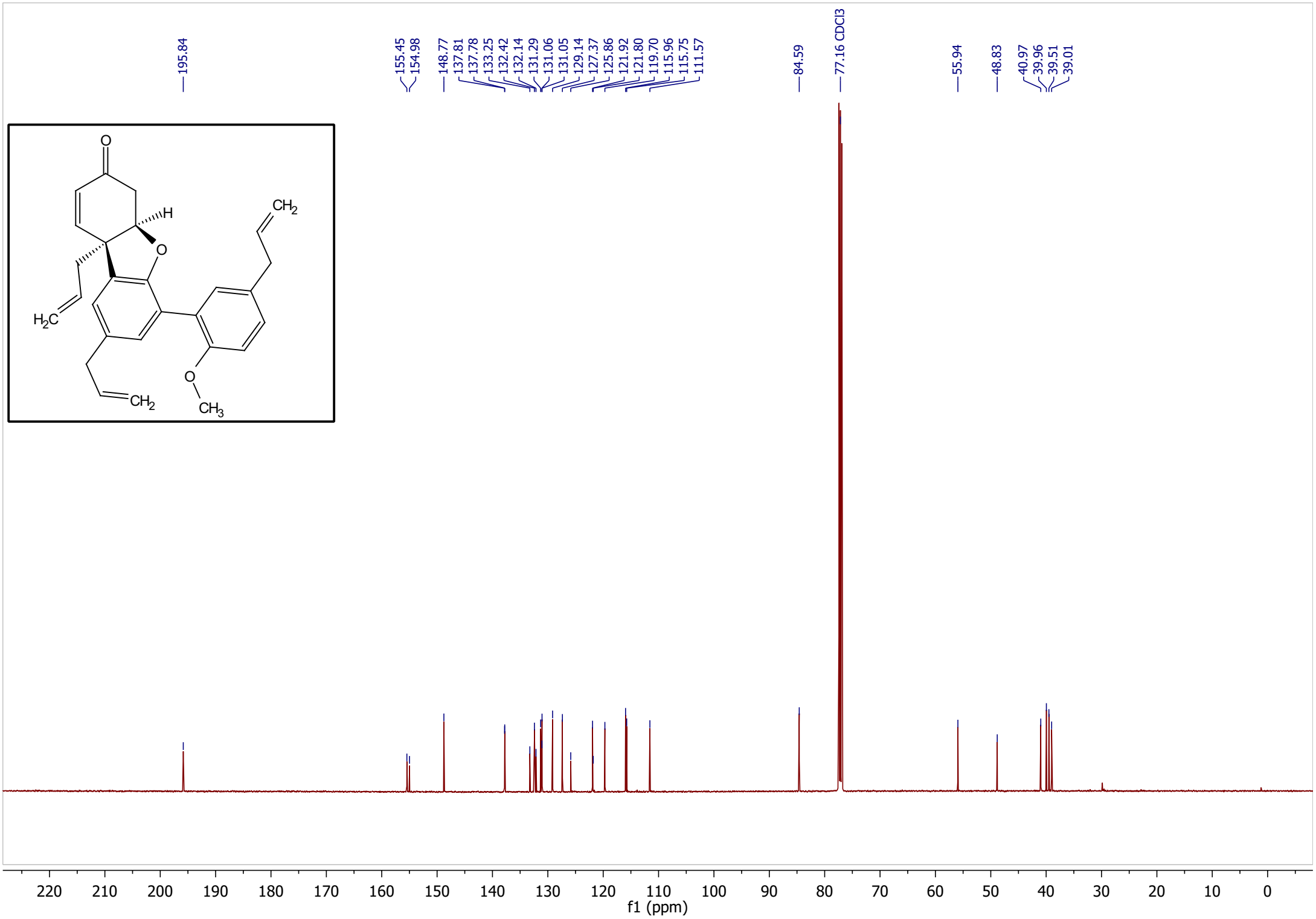


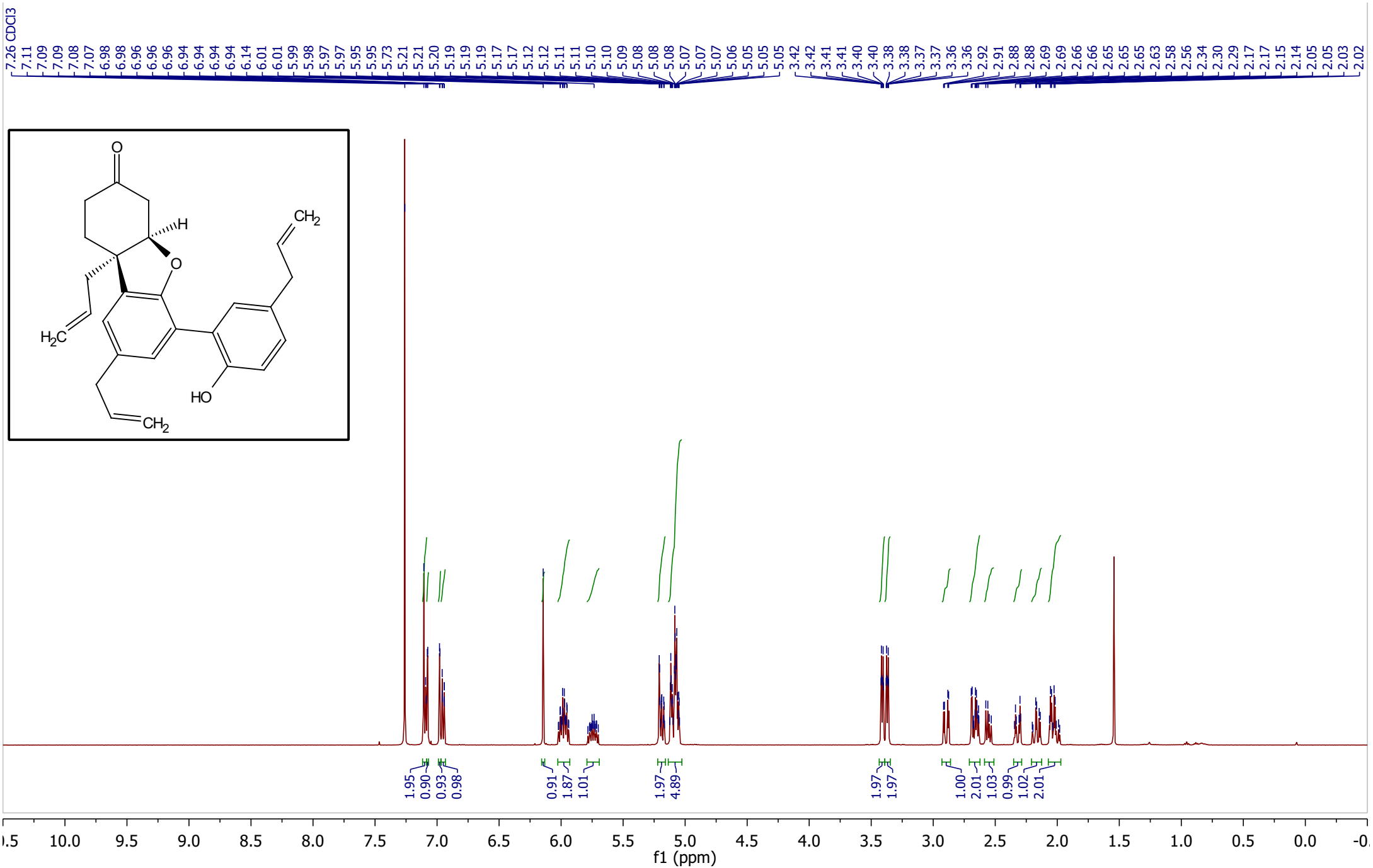
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.13 (d, *J* = 2.2 Hz, 1H), 7.11 (dd, *J* = 8.2, 2.2 Hz, 1H), 7.05 (d, *J* = 1.6 Hz, 1H), 6.97 (d, *J* = 8.2 Hz, 2H), 6.96 (d, *J* = 1.6 Hz, 1H), 6.53 (s, 1H), 5.97 (dddd, *J* = 16.8, 10.0, 6.8, 6.8 Hz, 2H), 5.87 (ddd, *J* = 10.1, 2.2, 1.1 Hz, 1H), 5.80 – 5.70 (m, 1H), 5.65 (ddd, *J* = 10.1, 1.9, 1.1 Hz, 1H), 5.16 – 5.03 (m, 7H), 4.88 (ddd, *J* = 4.4, 3.3, 0.9 Hz, 1H), 4.48 – 4.39 (m, 1H), 3.39 (d, *J* = 6.8 Hz, 2H), 3.38 (d, *J* = 6.8 Hz, 2H), 2.66 (dddd, *J* = 14.3, 6.7, 1.3, 1.3 Hz, 1H), 2.61 – 2.51 (m, 2H), 1.80 (ddd, *J* = 13.9, 9.5, 3.2 Hz, 1H), 1.57 (d, *J* = 5.8 Hz, 1H).


152.56
152.12
137.91
137.64
134.57
133.62
133.29
132.86
131.65
131.56
130.65
130.36
129.74
124.95
122.91
120.95
119.09
118.49
116.09
115.73
85.46
77.16 CDCl3
62.64
47.93
42.84
39.92
39.61
33.62
OH
H
O
HO
H2C
H2C
CH2
220
210
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f1 (ppm)

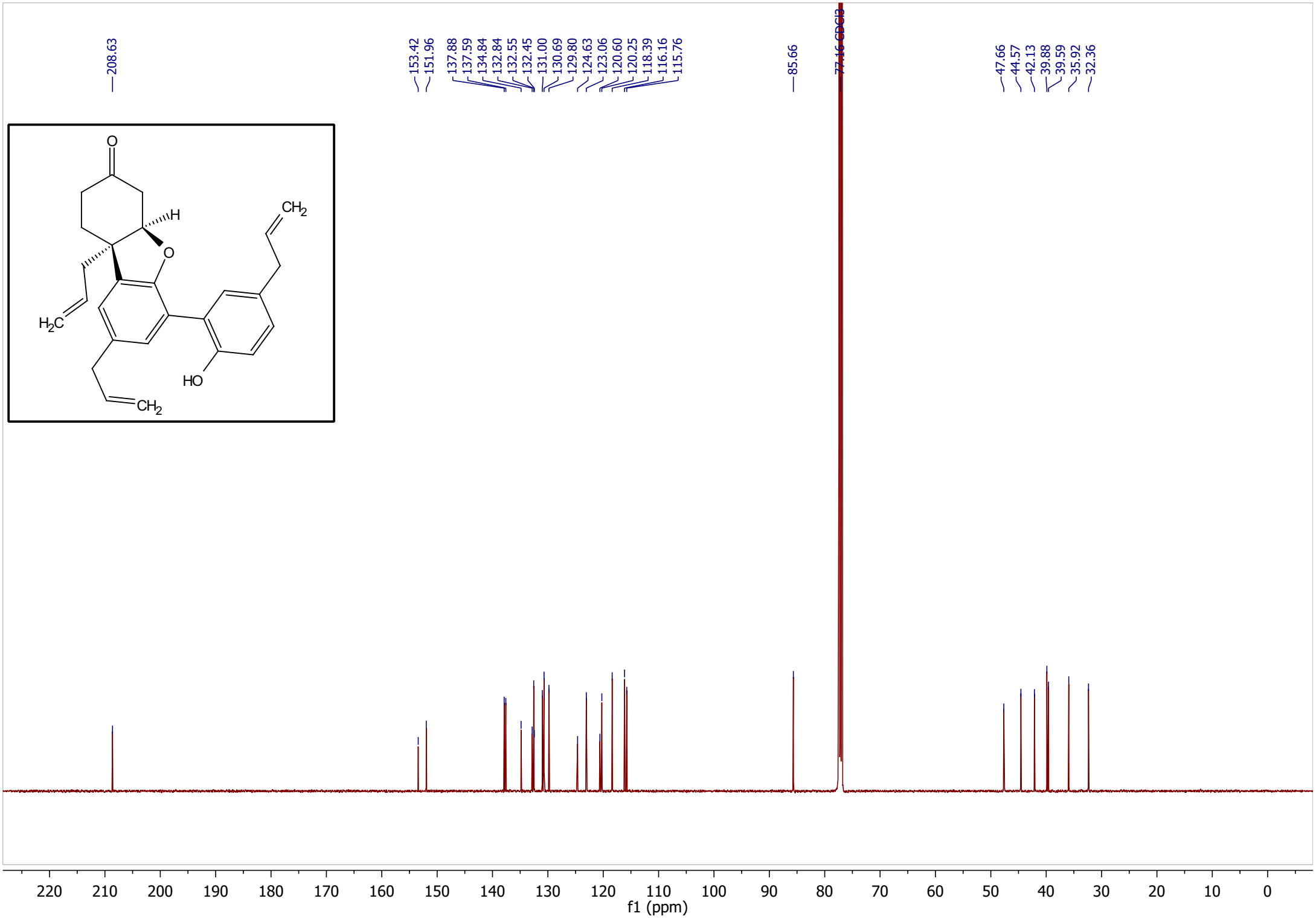


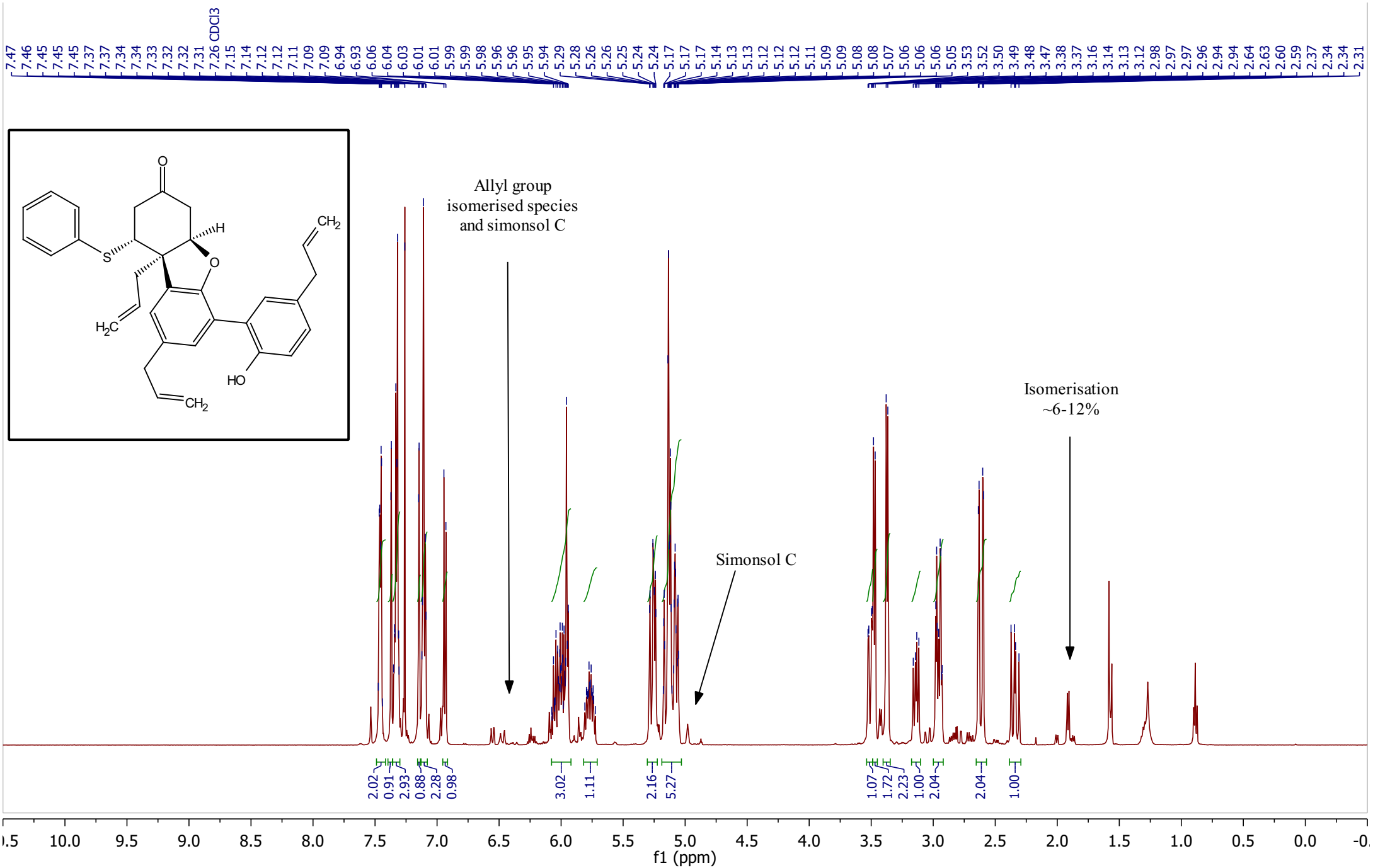
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.13 (dd, *J* = 8.3, 2.3 Hz, 1H), 7.10 (d, *J* = 2.3 Hz, 1H), 7.07 (d, *J* = 1.7 Hz, 1H), 6.99 (d, *J* = 1.7 Hz, 1H), 6.89 (d, *J* = 8.3 Hz, 1H), 6.56 (dd, *J* = 10.2, 1.8 Hz, 1H), 6.04 (d, *J* = 10.2 Hz, 1H), 5.97 (dddd, *J* = 16.8, 9.9, 6.8, 6.8 Hz, 2H), 5.82 (dddd, *J* = 16.8, 10.0, 8.1, 6.6 Hz, 1H), 5.23 – 5.16 (m, 2H), 5.15 – 5.03 (m, 4H), 4.79 (ddd, *J* = 4.1, 3.0, 1.8 Hz, 1H), 3.72 (s, 3H), 3.39 (d, *J* = 6.8 Hz, 2H), 3.35 (d, *J* = 6.8 Hz, 2H), 2.97 (dd, *J* = 17.5, 3.0 Hz, 1H), 2.83 (dddd, *J* = 14.2, 6.6, 1.4, 1.4 Hz, 1H), 2.73 (dd, *J* = 17.5, 4.1 Hz, 1H), 2.66 (dd, *J* = 14.2, 8.1 Hz, 1H).


195.84
155.45
154.98
148.77
137.81
137.78
133.25
132.42
132.14
131.29
131.06
131.05
129.14
127.37
125.86
121.92
121.80
119.70
115.96
115.75
111.57
84.59
77.16 CDCl3
55.94
48.83
40.97
39.96
39.51
39.01
O
H
O
CH2
H2C
CH2
O
CH3
220
210
200
190
180
170
160
150
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130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

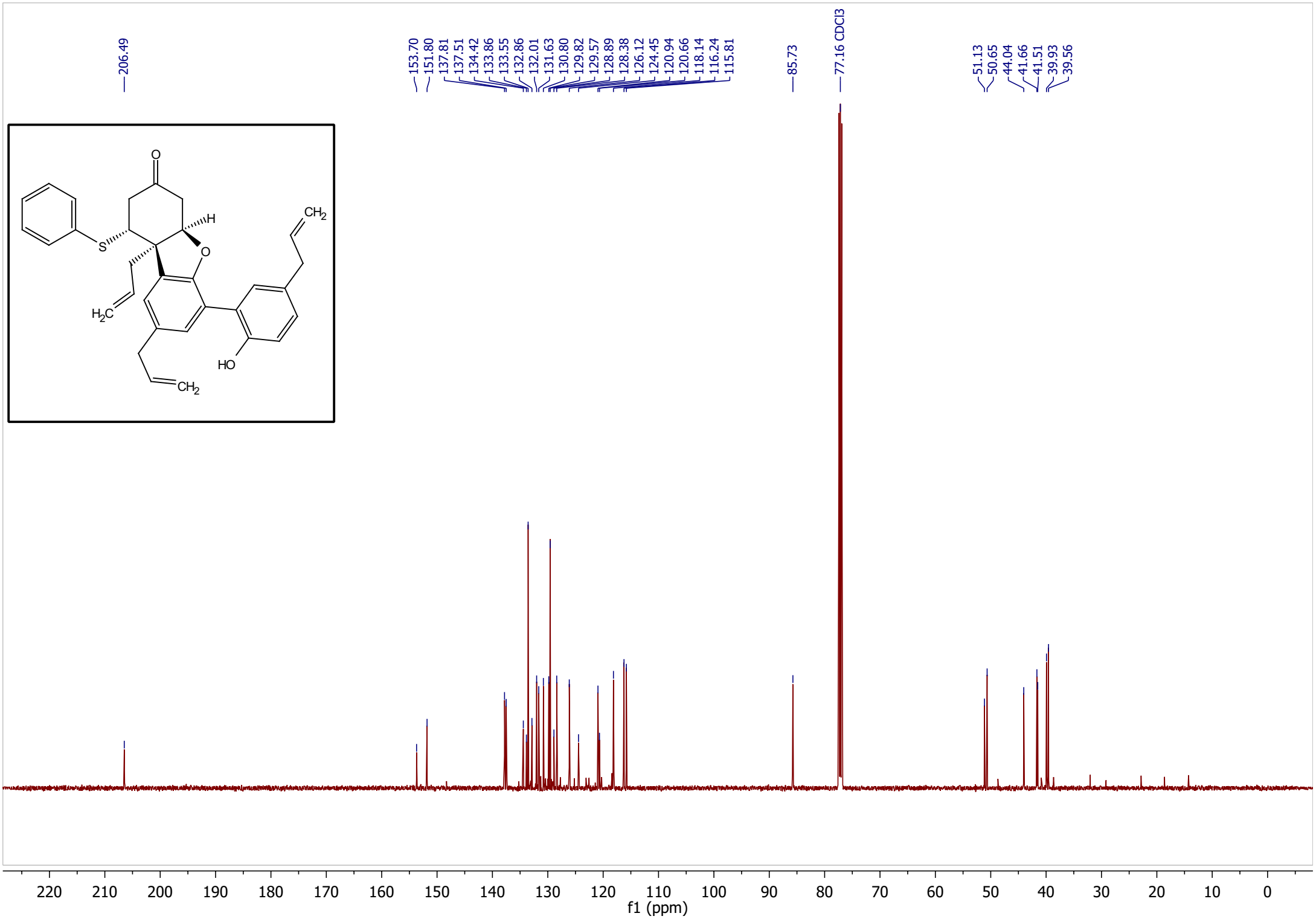


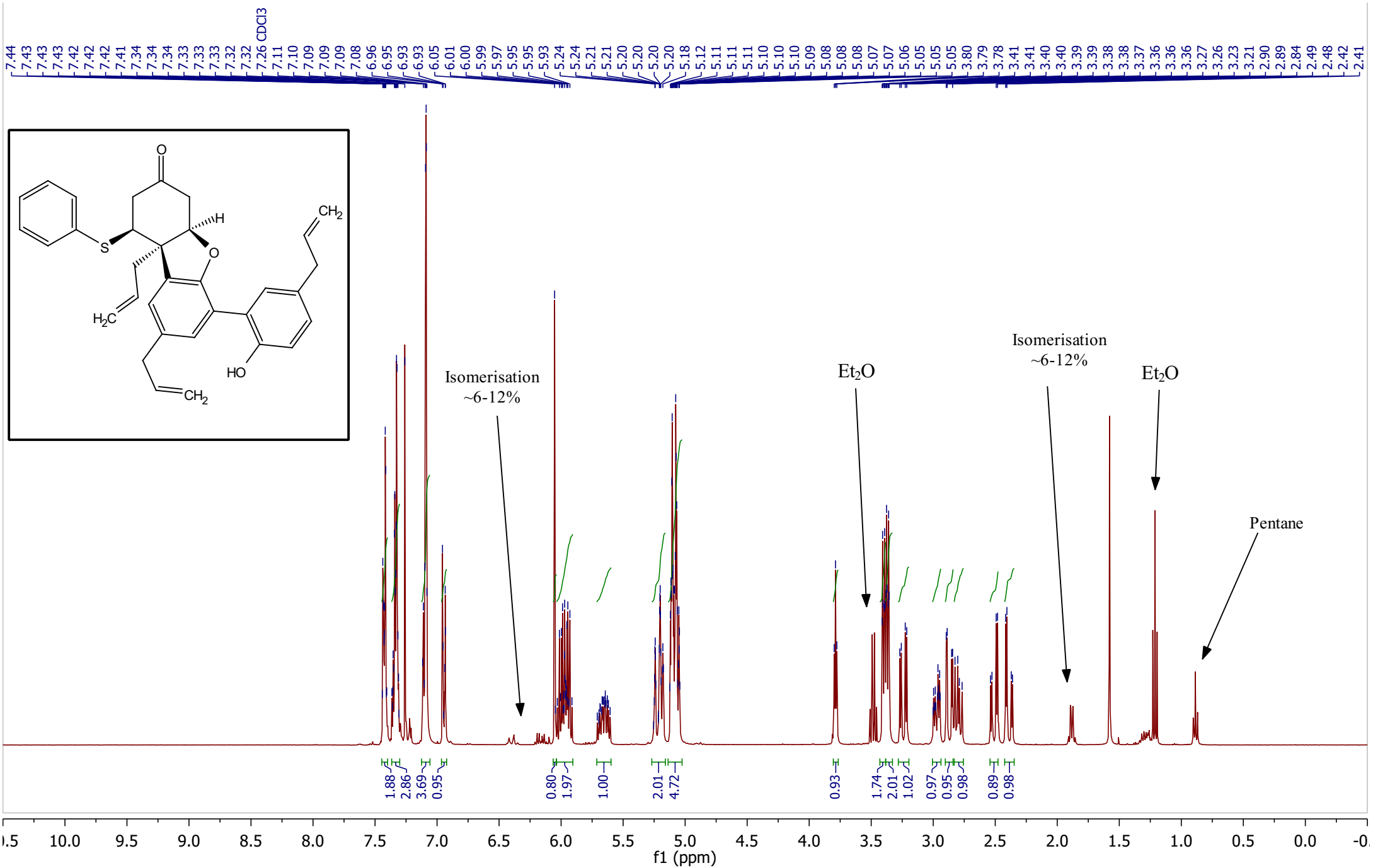
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.11 – 7.08 (m, 2H), 7.08 (d, *J* = 1.8 Hz, 1H), 6.98 (d, *J* = 1.8 Hz, 1H), 6.98 – 6.92 (m, 1H), 6.14 (s, 1H), 6.03 – 5.93 (m, 2H), 5.74 (dddd, *J* = 16.8, 10.5, 8.2, 6.6 Hz, 1H), 5.22 – 5.16 (m, 2H), 5.13 – 5.04 (m, 5H), 3.41 (ddd, *J* = 6.8, 1.5, 1.5 Hz, 2H), 3.37 (ddd, *J* = 6.7, 1.5, 1.5 Hz, 2H), 2.90 (dd, *J* = 17.1, 3.3 Hz, 1H), 2.67 (dd, *J* = 17.1, 3.7 Hz, 1H), 2.66 (dddd, *J* = 14.0, 6.6, 1.2, 1.2 Hz, 1H), 2.55 (dd, *J* = 14.0, 8.2 Hz, 1H), 2.35 – 2.29 (m, 1H), 2.22 – 2.13 (m, 1H), 2.08 – 1.97 (m, 2H).


208.63
153.42
151.96
137.88
137.59
134.84
132.84
132.55
132.45
131.00
130.69
129.80
124.63
123.06
120.60
120.25
118.39
116.16
115.76
85.66
77.16 CDCl3
47.66
44.57
42.13
39.88
39.59
35.92
32.36
O
H
O
CH2
H2C
HO
CH2
220
210
200
190
180
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120
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100
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80
70
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40
30
20
10
0
f1 (ppm)

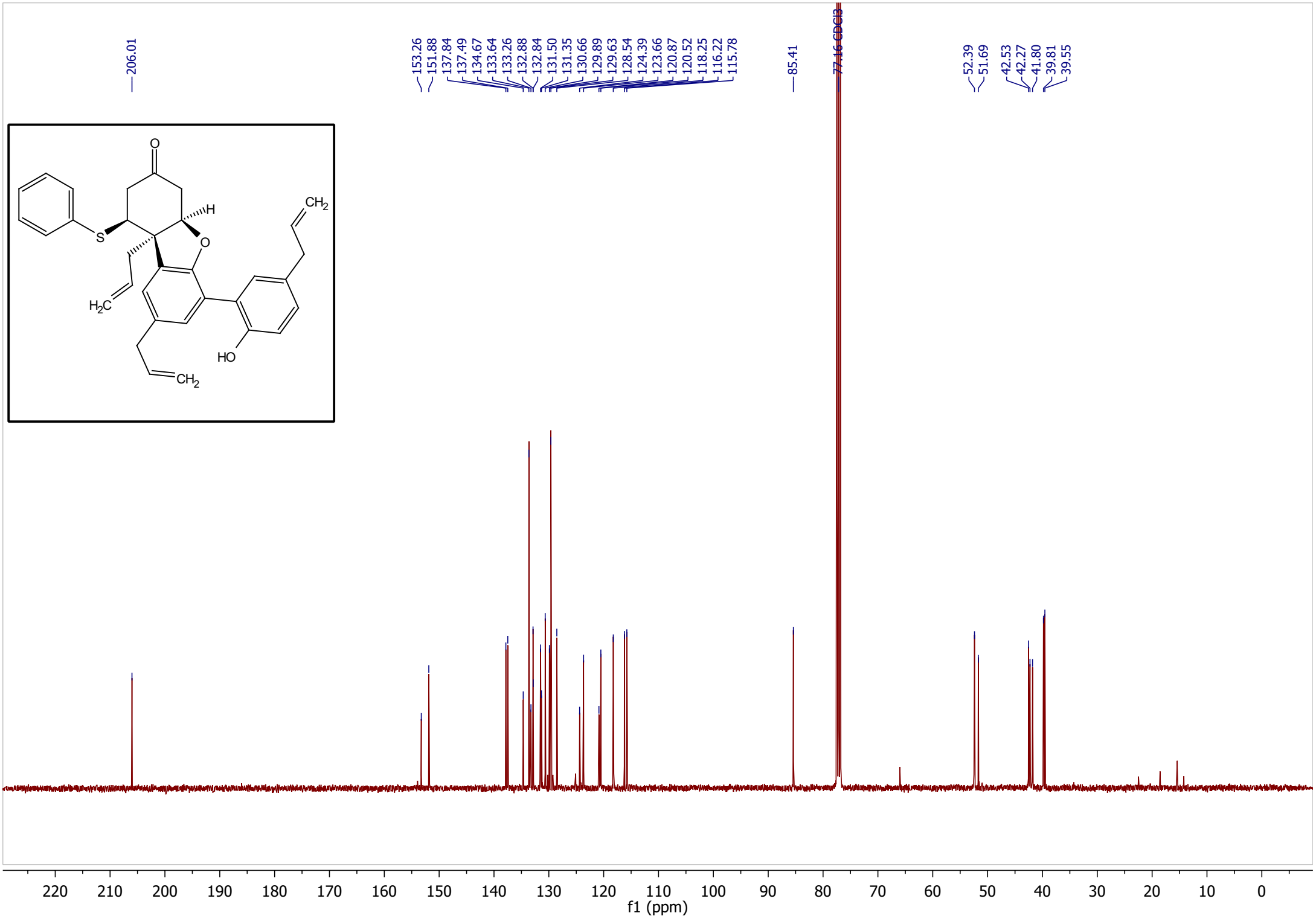


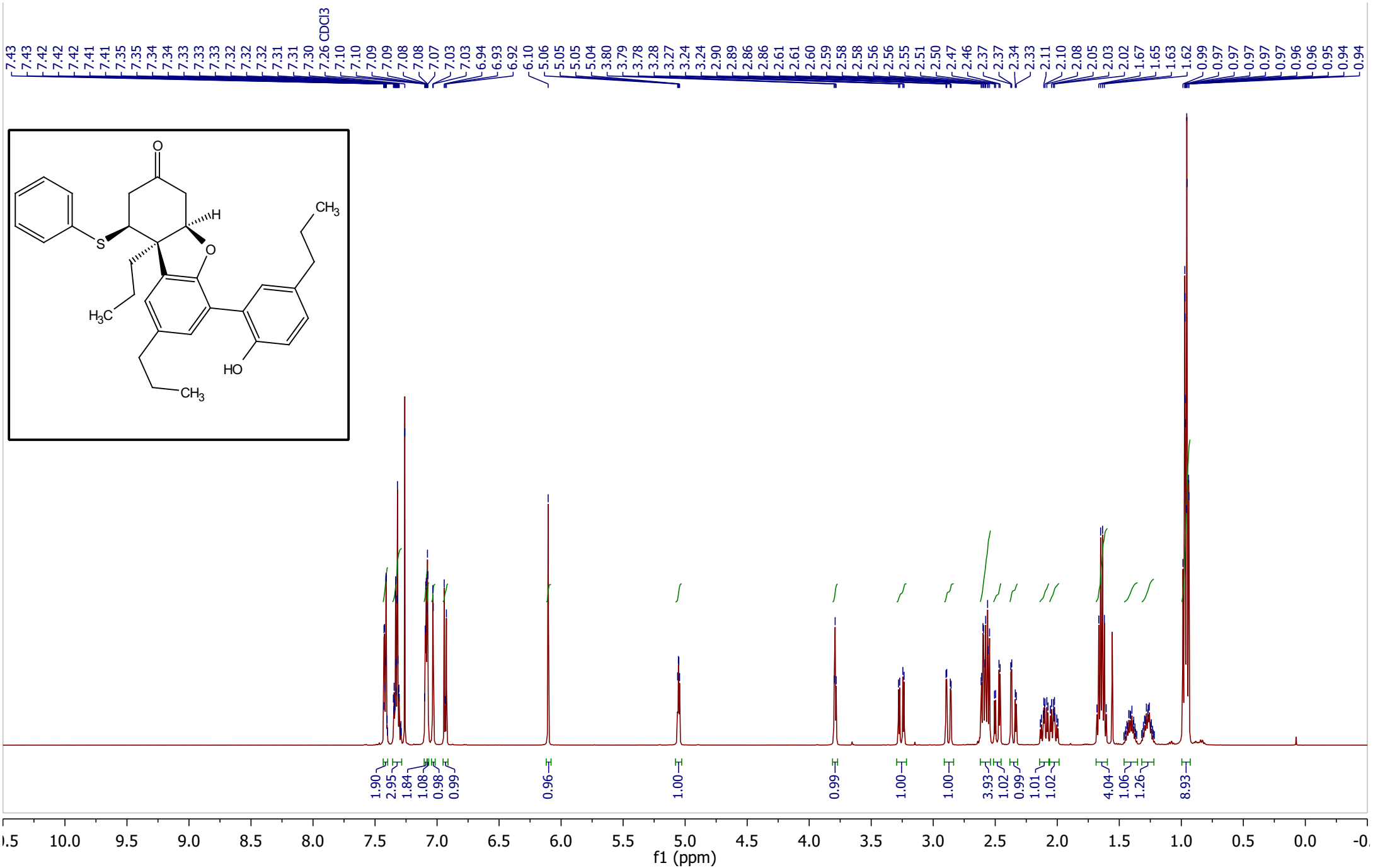
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.48 – 7.43 (m, 2H), 7.37 (d, *J* = 1.8 Hz, 1H), 7.35 – 7.30 (m, 3H), 7.15 (d, *J* = 1.8 Hz, 1H), 7.13 – 7.08 (m, 2H), 6.94 (d, *J* = 8.1 Hz, 1H), 6.09 – 5.92 (m, 3H), 5.77 (dddd, *J* = 16.7, 10.3, 8.5, 6.4 Hz, 1H), 5.30 – 5.23 (m, 2H), 5.18 – 5.04 (m, 5H), 3.51 (dd, *J* = 13.8, 3.3 Hz, 1H), 3.47 (d, *J* = 6.8 Hz, 2H), 3.37 (d, *J* = 6.7 Hz, 2H), 3.14 (dd, *J* = 14.1, 8.5 Hz, 1H), 2.99 – 2.93 (m, 2H), 2.66 – 2.55 (m, 2H), 2.34 (dd, *J* = 18.1, 13.8 Hz, 1H).


206.49
153.70
151.80
137.81
137.51
134.42
133.86
133.55
132.86
132.01
131.63
130.80
129.82
129.57
128.89
128.38
126.12
124.45
120.94
120.66
118.14
116.24
115.81
85.73
77.16 CDCl3
51.13
50.65
44.04
41.66
41.51
39.93
39.56
O
H
S
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CH2
H2C
HO
CH2
220
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200
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10
0
f1 (ppm)

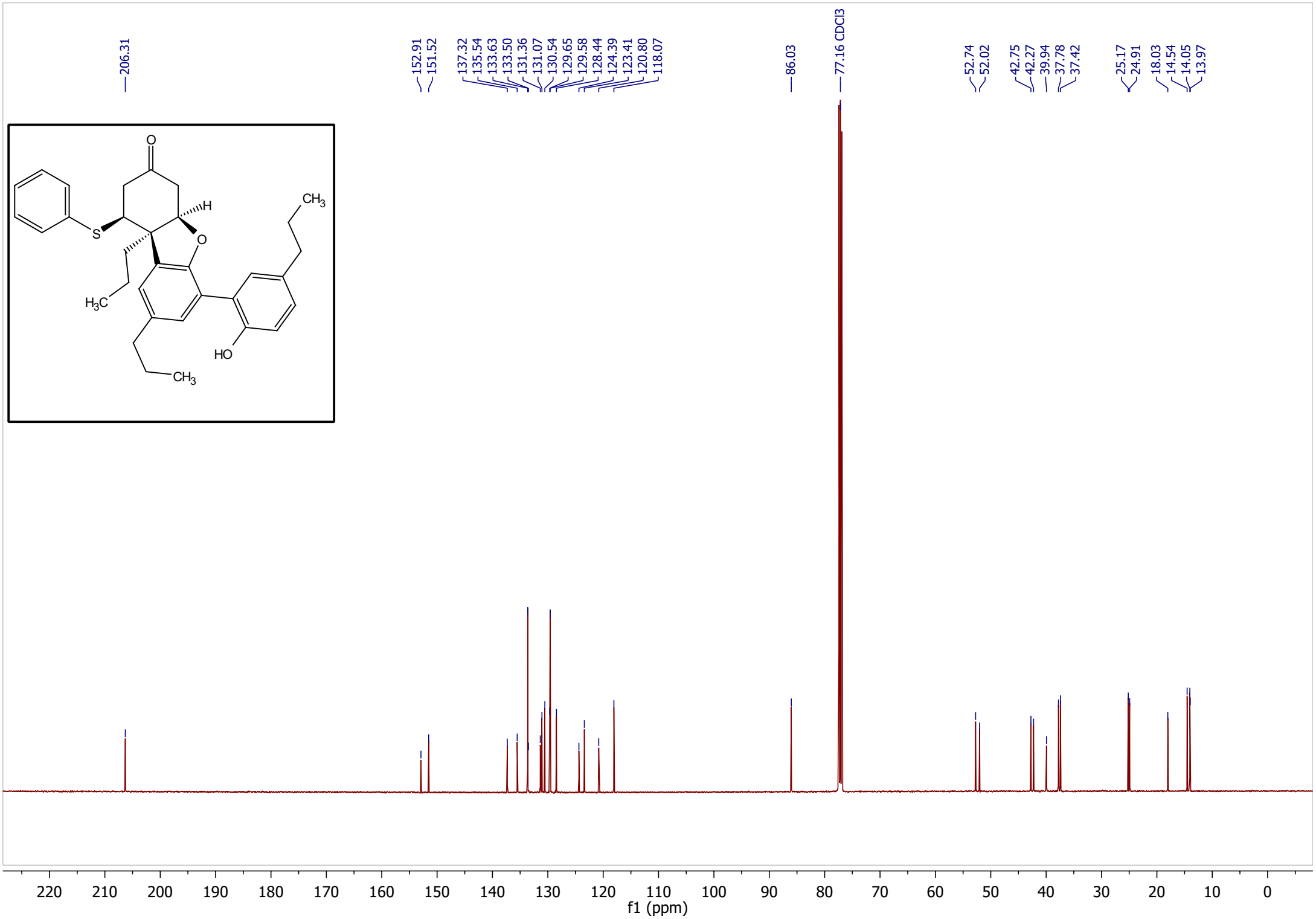


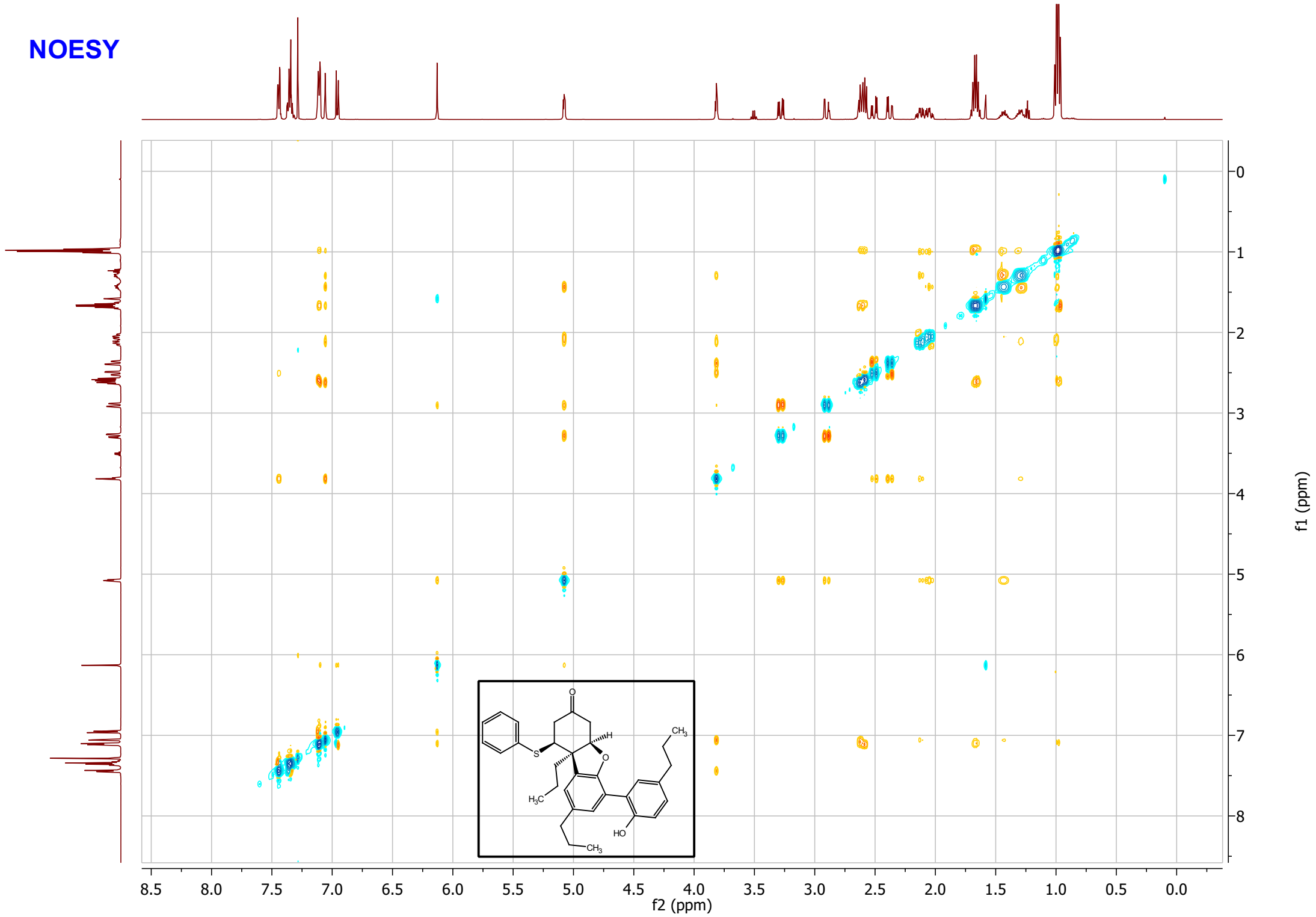
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.45 – 7.40 (m, 2H), 7.37 – 7.31 (m, 3H), 7.12 – 7.07 (m, 4H), 6.96 – 6.92 (m, 1H), 6.05 (s, 1H), 6.04 – 5.89 (m, 2H), 5.66 (dddd, *J* = 17.1, 10.1, 8.8, 5.6 Hz, 1H), 5.26 – 5.16 (m, 2H), 5.13 – 5.03 (m, 5H), 3.79 (dd, *J* = 4.6, 4.0 Hz, 1H), 3.40 (ddd, *J* = 6.7, 1.5, 1.5 Hz, 2H), 3.37 (ddd, *J* = 6.7, 1.5, 1.5 Hz, 2H), 3.24 (dd, *J* = 17.5, 4.9 Hz, 1H), 2.97 (dddd, *J* = 14.0, 5.6, 1.5, 1.5 Hz, 1H), 2.87 (dd, *J* = 17.5, 3.0 Hz, 1H), 2.80 (dd, *J* = 14.0, 8.8 Hz, 1H), 2.51 (dd, *J* = 18.1, 4.6 Hz, 1H), 2.39 (dd, *J* = 18.1, 4.0 Hz, 1H).


206.01
153.26
151.88
137.84
137.49
134.67
133.64
133.26
132.88
132.84
131.50
131.35
130.66
129.89
129.63
128.54
124.39
123.66
120.87
120.52
118.25
116.22
115.78
85.41
77.16 CDCl3
52.39
51.69
42.53
42.27
41.80
39.81
39.55
O
H
S
O
CH2
H2C
HO
CH2
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0
f1 (ppm)

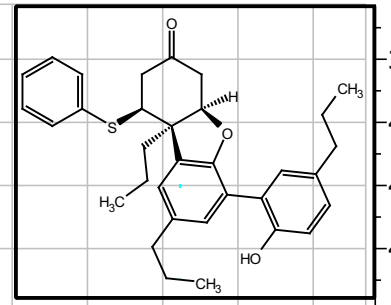


^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.44 – 7.40 (m, 2H), 7.36 – 7.29 (m, 3H), 7.11 – 7.08 (m, 2H), 7.08 (d, *J* = 1.8 Hz, 1H), 7.03 (d, *J* = 1.8 Hz, 1H), 6.95 – 6.92 (m, 1H), 6.10 (s, 1H), 5.05 (dd, *J* = 4.8, 3.0 Hz, 1H), 3.79 (dd, *J* = 4.5, 3.9 Hz, 1H), 3.26 (dd, *J* = 17.5, 4.8 Hz, 1H), 2.88 (dd, *J* = 17.5, 3.0 Hz, 1H), 2.63 – 2.53 (m, 4H), 2.48 (dd, *J* = 18.2, 4.5 Hz, 1H), 2.35 (dd, *J* = 18.2, 3.9 Hz, 1H), 2.11 (ddd, *J* = 13.9, 12.1, 4.6 Hz, 1H), 2.02 (ddd, *J* = 13.9, 11.9, 4.6 Hz, 1H), 1.70 – 1.59 (m, 4H), 1.48 – 1.34 (m, 1H), 1.34 – 1.20 (m, 1H), 0.97 (dd, *J* = 7.2, 7.2 Hz, 3H), 0.96 (dd, *J* = 7.3, 7.3 Hz, 3H), 0.95 (dd, *J* = 7.3, 7.3 Hz, 3H).


206.31
152.91
151.52
137.32
135.54
133.63
133.50
131.36
131.07
130.54
129.65
129.58
128.44
124.39
123.41
120.80
118.07
86.03
77.16 CDCl3
52.74
52.02
42.75
42.27
39.94
37.78
37.42
25.17
24.91
18.03
14.54
14.05
13.97
O
H
S
O
CH3
H3C
HO
CH3
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f1 (ppm)

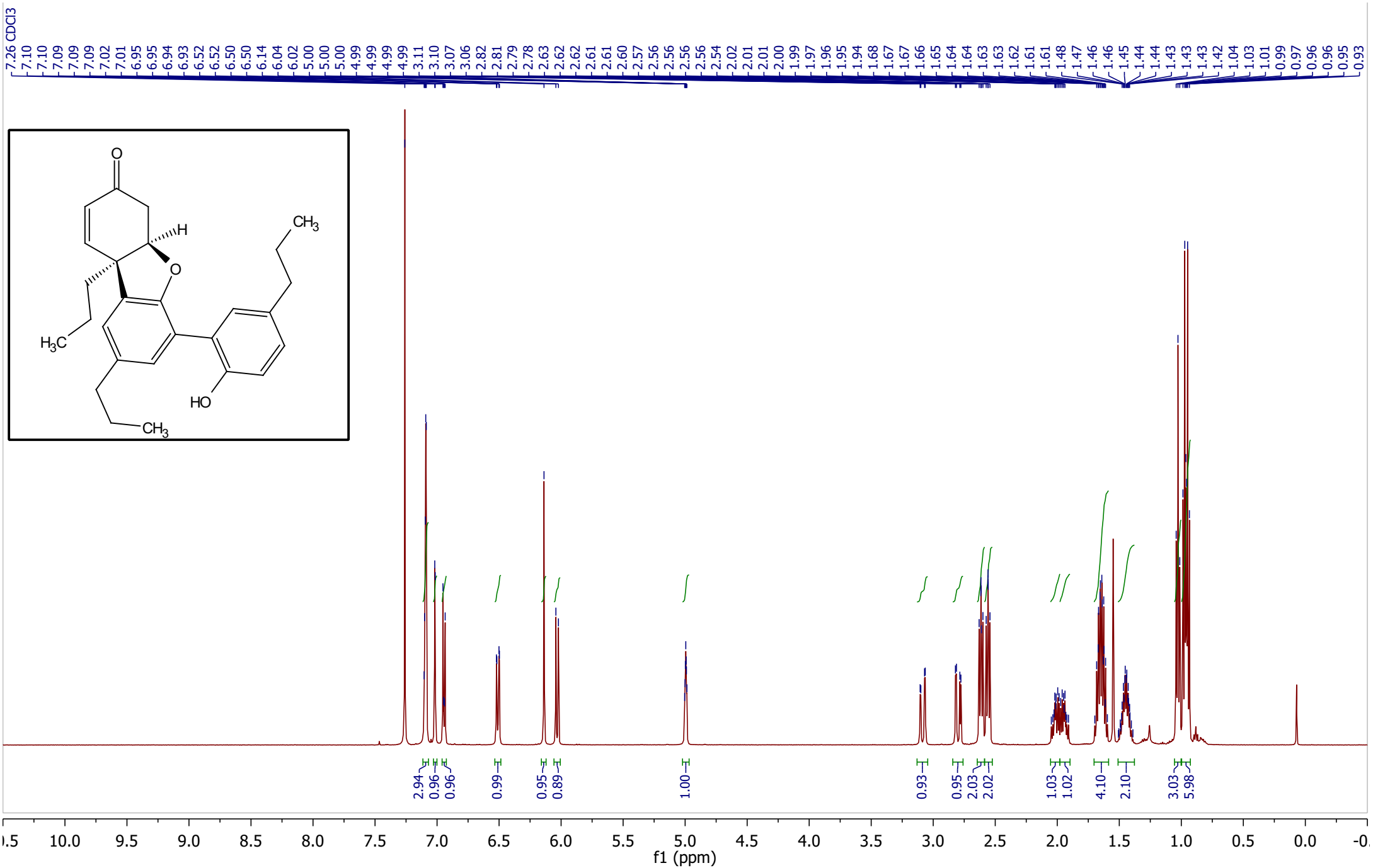

NOESY
f2 (ppm)
f1 (ppm)
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
0
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4
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6
7
8
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H
S
O
H3C
CH3
HO
CH3

NOESY

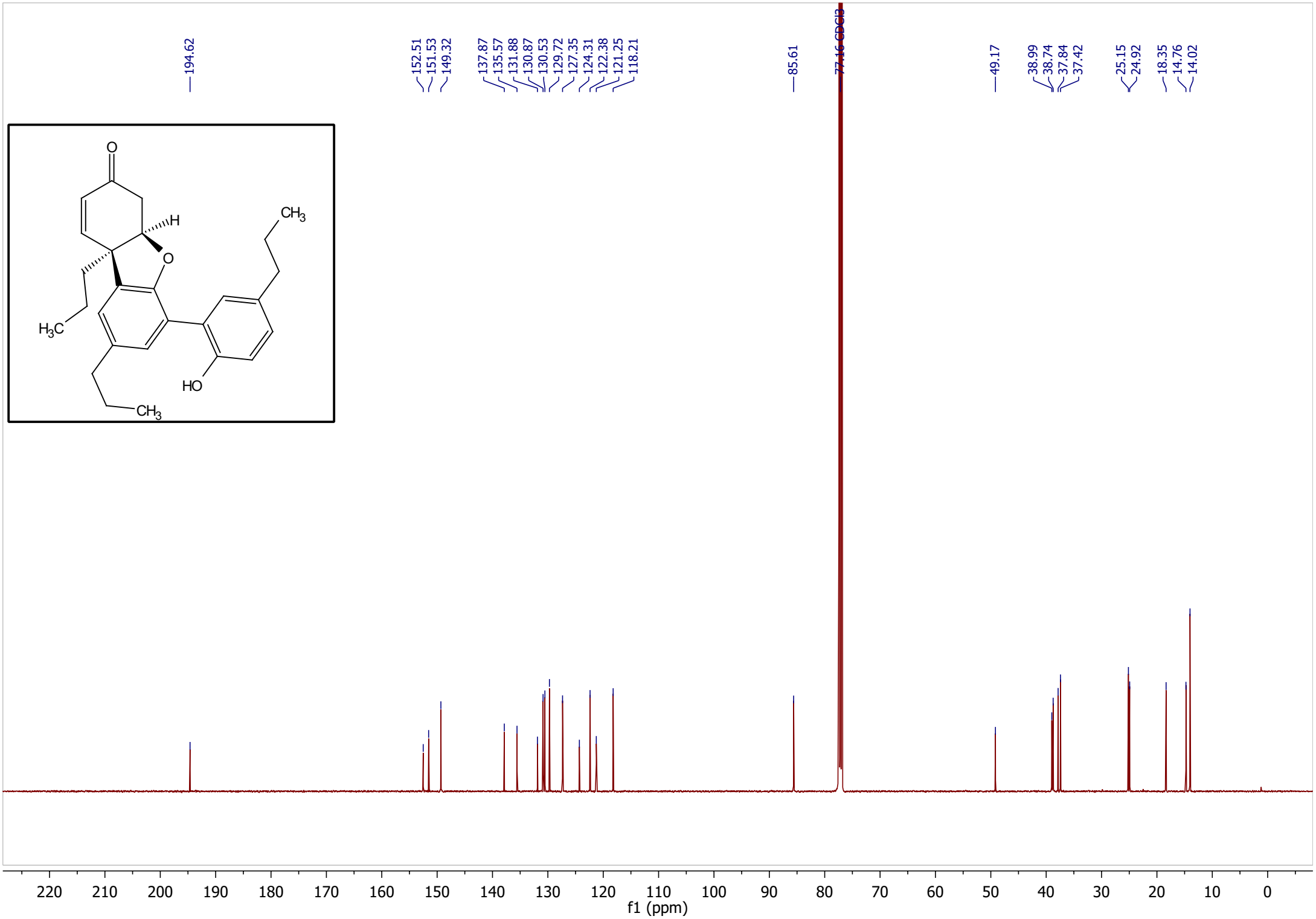


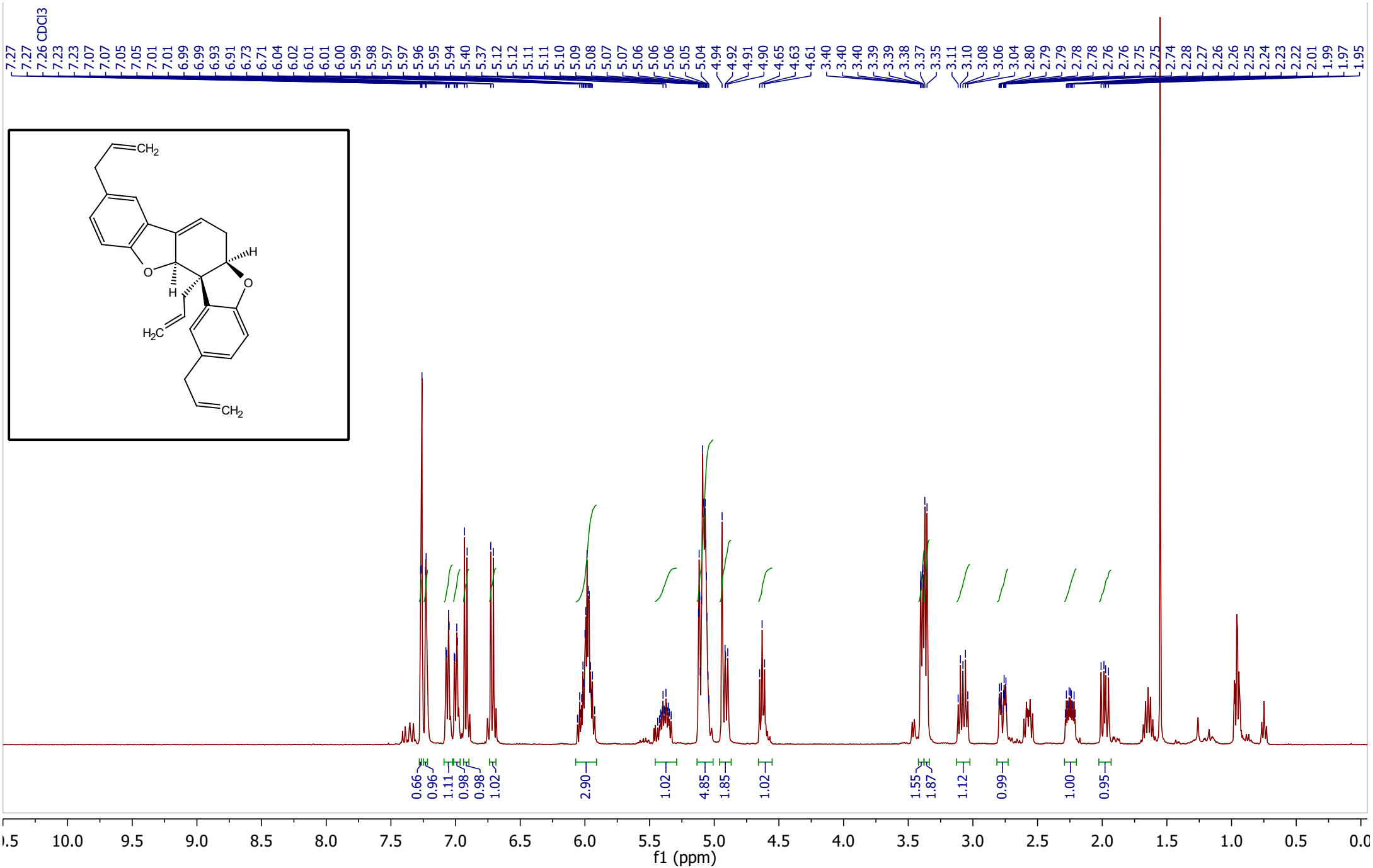
f2 (ppm)

f1 (ppm)

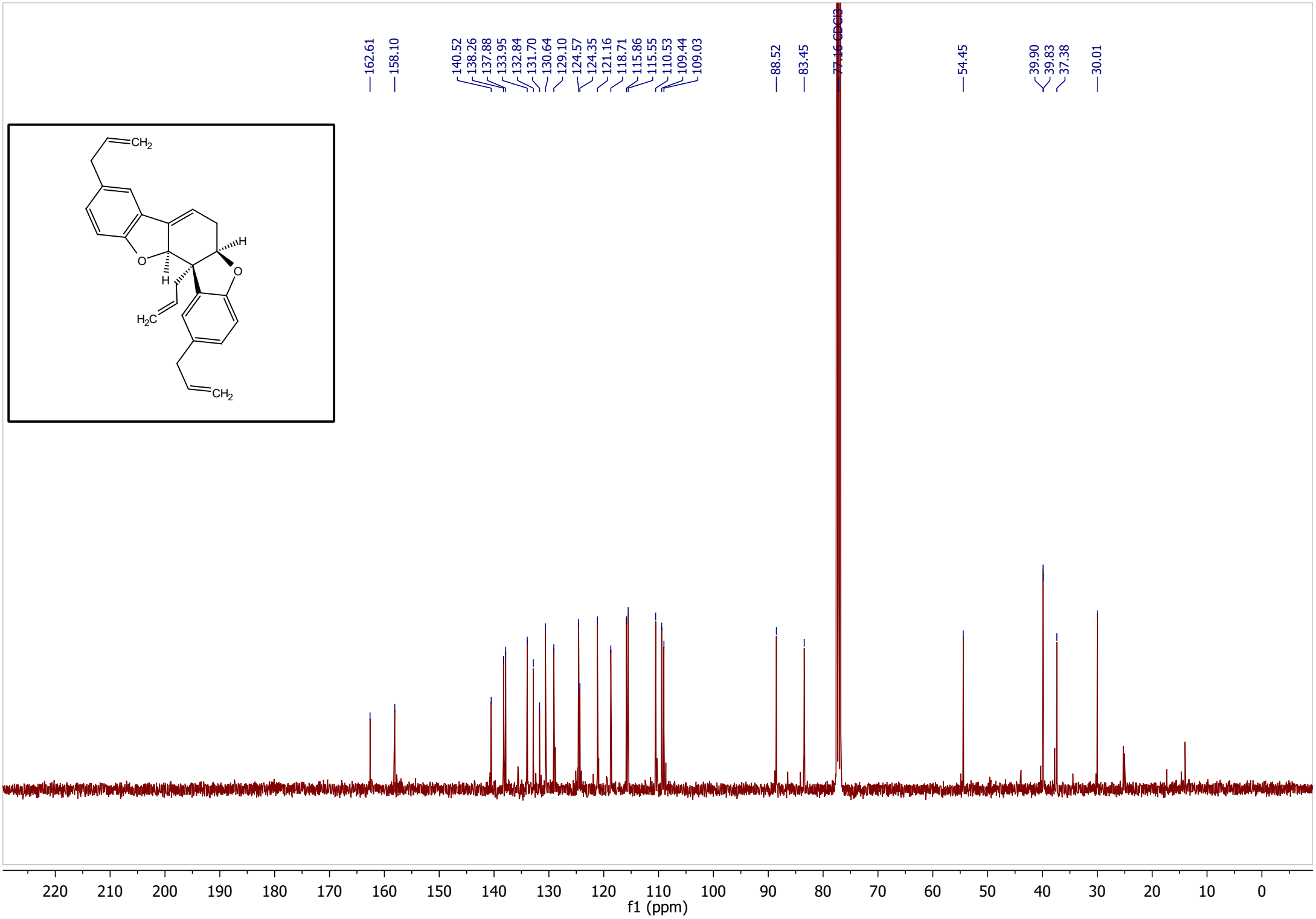


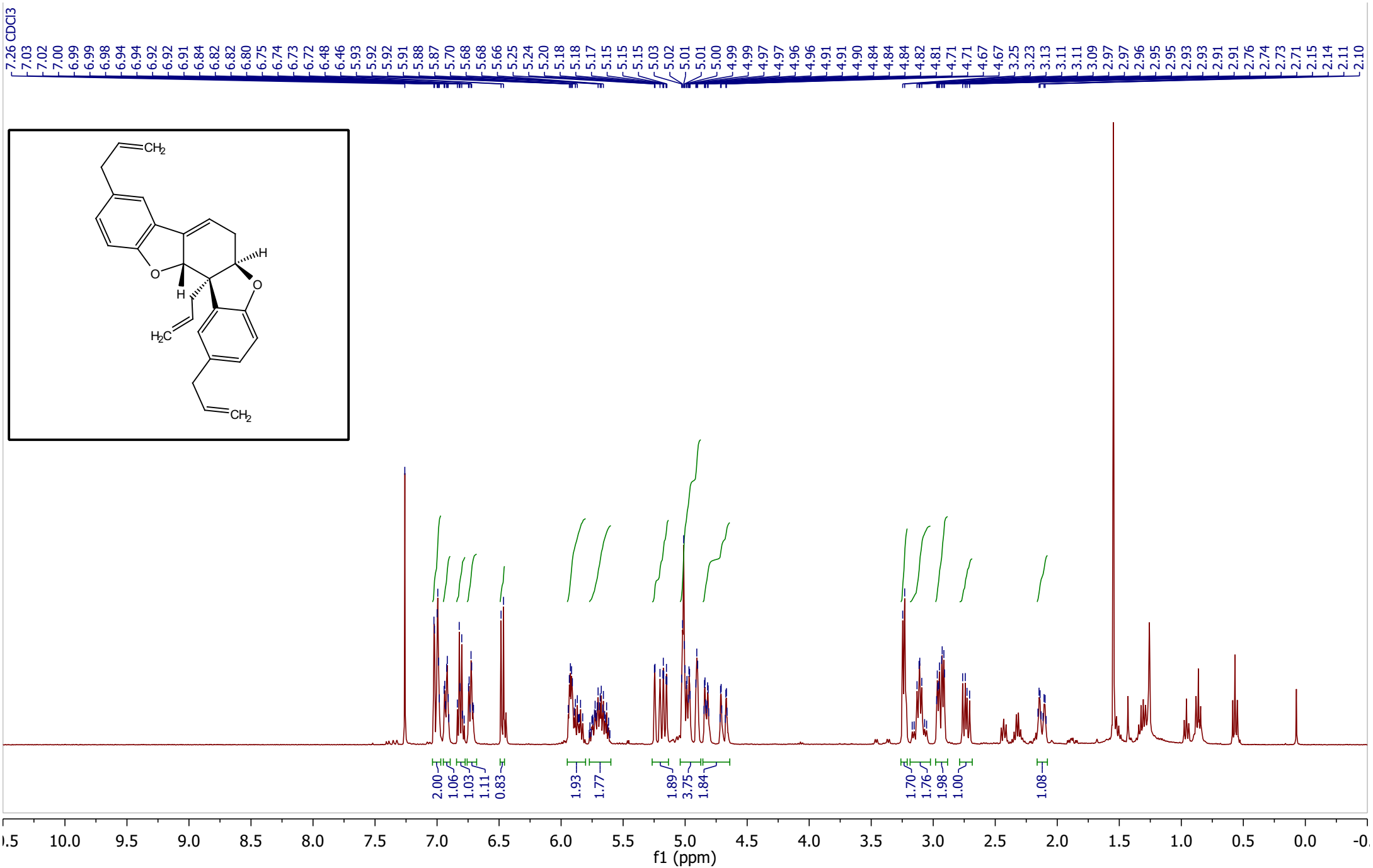
^{1}H NMR (500 MHz, Chloroform-*d*) δ 7.12 – 7.07 (m, 3H), 7.02 (d, *J* = 1.8 Hz, 1H), 6.97 – 6.92 (m, 1H), 6.51 (dd, *J* = 10.2, 1.8 Hz, 1H), 6.14 (s, 1H), 6.03 (d, *J* = 10.2 Hz, 1H), 4.99 (ddd, *J* = 4.2, 2.9, 1.8 Hz, 1H), 3.08 (dd, *J* = 17.8, 2.9 Hz, 1H), 2.80 (dd, *J* = 17.8, 4.2 Hz, 1H), 2.61 (dd, *J* = 8.2, 7.1 Hz, 2H), 2.56 (dd, *J* = 8.2, 7.1 Hz, 2H), 2.02 (ddd, *J* = 14.0, 11.4, 5.7 Hz, 1H), 1.94 (ddd, *J* = 14.0, 11.2, 6.2 Hz, 1H), 1.70 – 1.59 (m, 4H), 1.51 – 1.38 (m, 2H), 1.03 (dd, *J* = 7.3, 7.3 Hz, 3H), 0.97 (dd, *J* = 7.4, 7.4 Hz, 3H), 0.95 (dd, *J* = 7.3, 7.3 Hz, 3H).


194.62
152.51
151.53
149.32
137.87
135.57
131.88
130.87
130.53
129.72
127.35
124.31
122.38
121.25
118.21
85.61
77.16 CDCl3
49.17
38.99
38.74
37.84
37.42
25.15
24.92
18.35
14.76
14.02
O
H
O
CH3
H3C
HO
CH3
220
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200
190
180
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10
0
f1 (ppm)

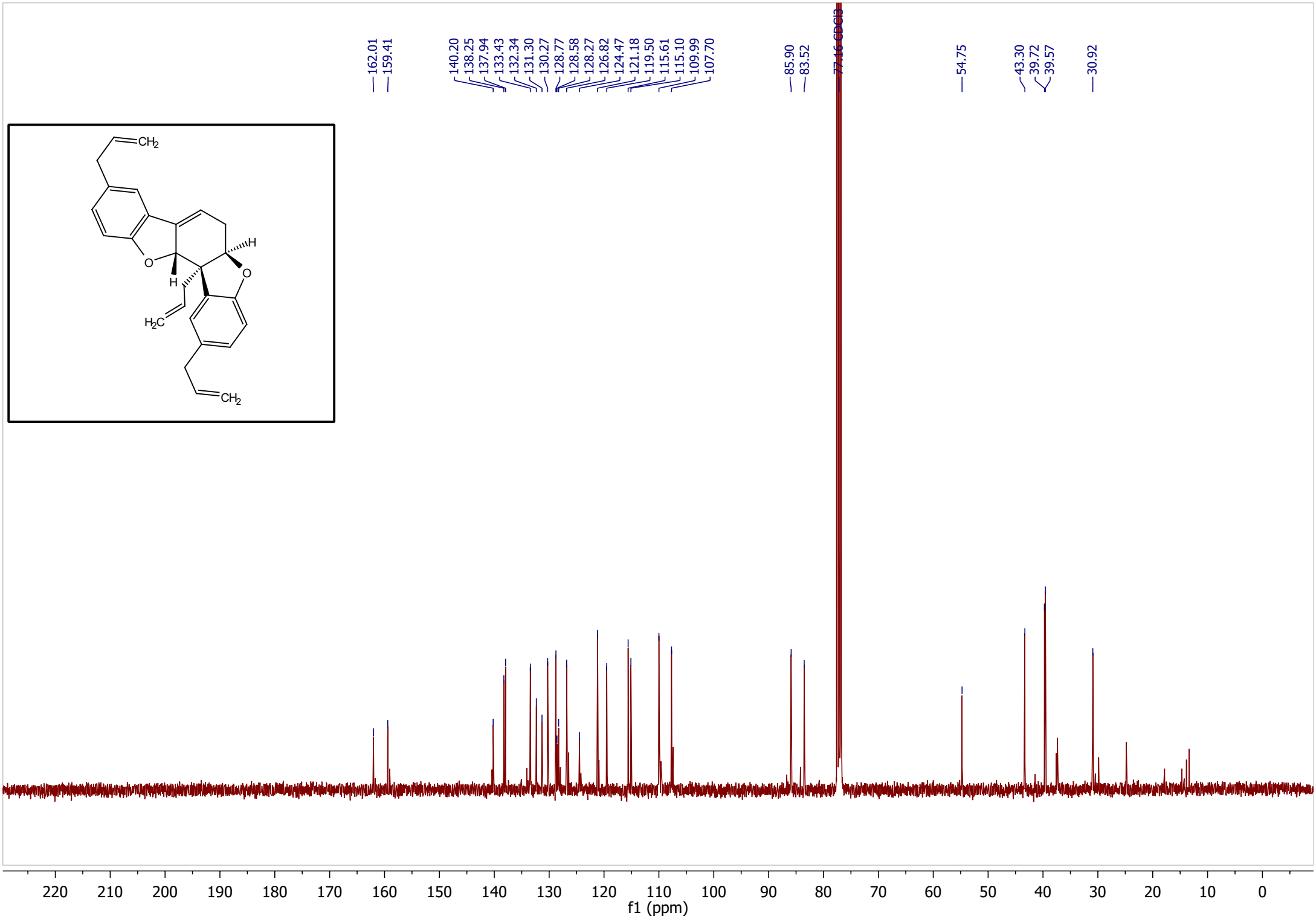


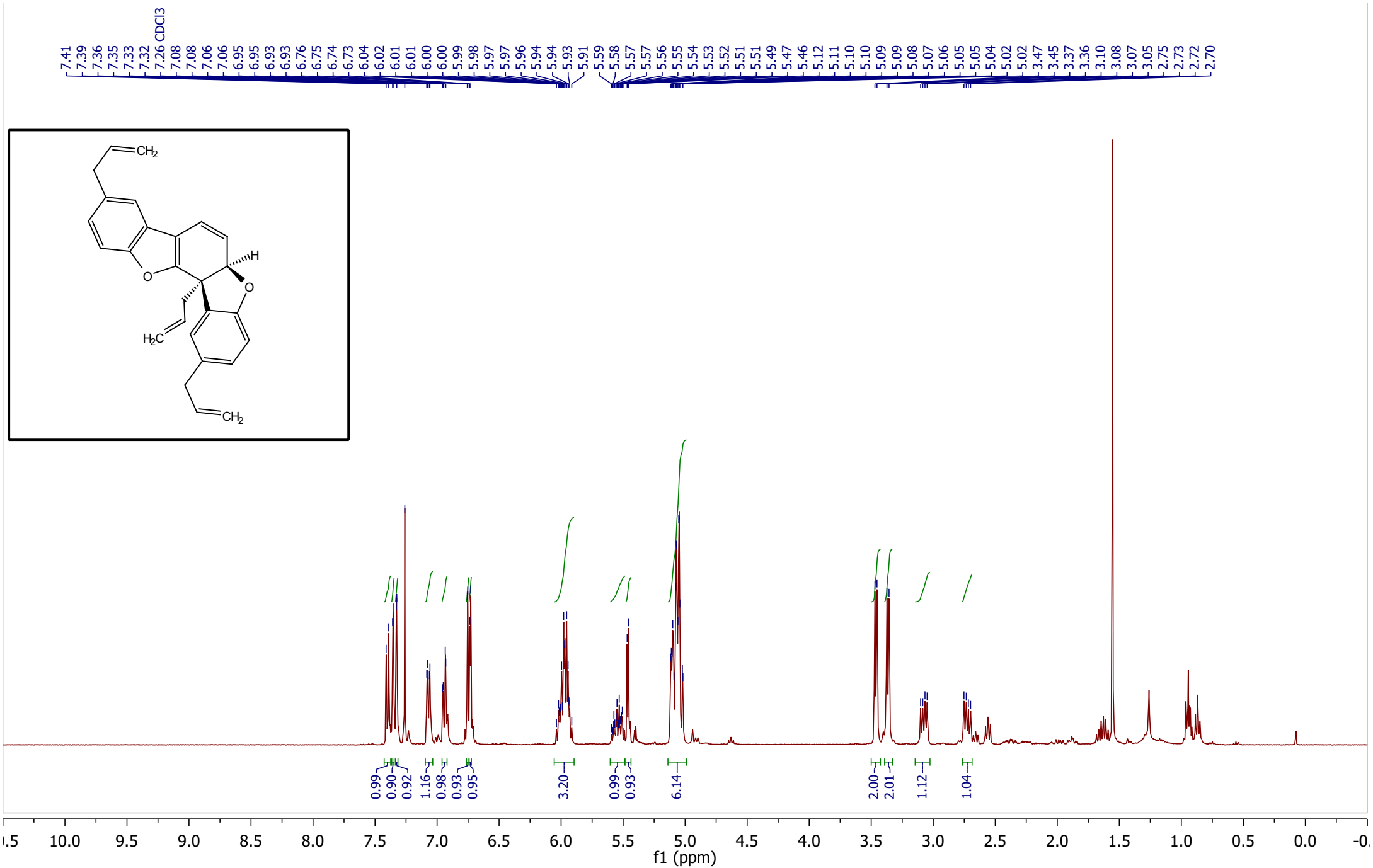
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.27 (d, *J* = 1.8 Hz, 1H), 7.23 (d, *J* = 1.9 Hz, 1H), 7.06 (dd, *J* = 8.2, 1.9 Hz, 1H), 7.00 (dd, *J* = 8.2, 1.8 Hz, 1H), 6.92 (d, *J* = 8.2 Hz, 1H), 6.72 (d, *J* = 8.2 Hz, 1H), 6.08 – 5.90 (m, 3H), 5.45 – 5.31 (m, 1H), 5.14 – 5.03 (m, 5H), 4.96 – 4.87 (m, 2H), 4.63 (dd, *J* = 7.7, 7.7 Hz, 1H), 3.39 (d, *J* = 6.6 Hz, 2H), 3.36 (d, *J* = 6.8 Hz, 2H), 3.14 – 3.01 (m, 1H), 2.82 – 2.73 (m, 1H), 2.31 – 2.19 (m, 1H), 2.02 – 1.91 (m, 1H).


162.61
158.10
140.52
138.26
137.88
133.95
132.84
131.70
130.64
129.10
124.57
124.35
121.16
118.71
115.86
115.55
110.53
109.44
109.03
88.52
83.45
77.16 CDCl3
54.45
39.90
39.83
37.38
30.01
CH2
H
O
O
H
H2C
CH2
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

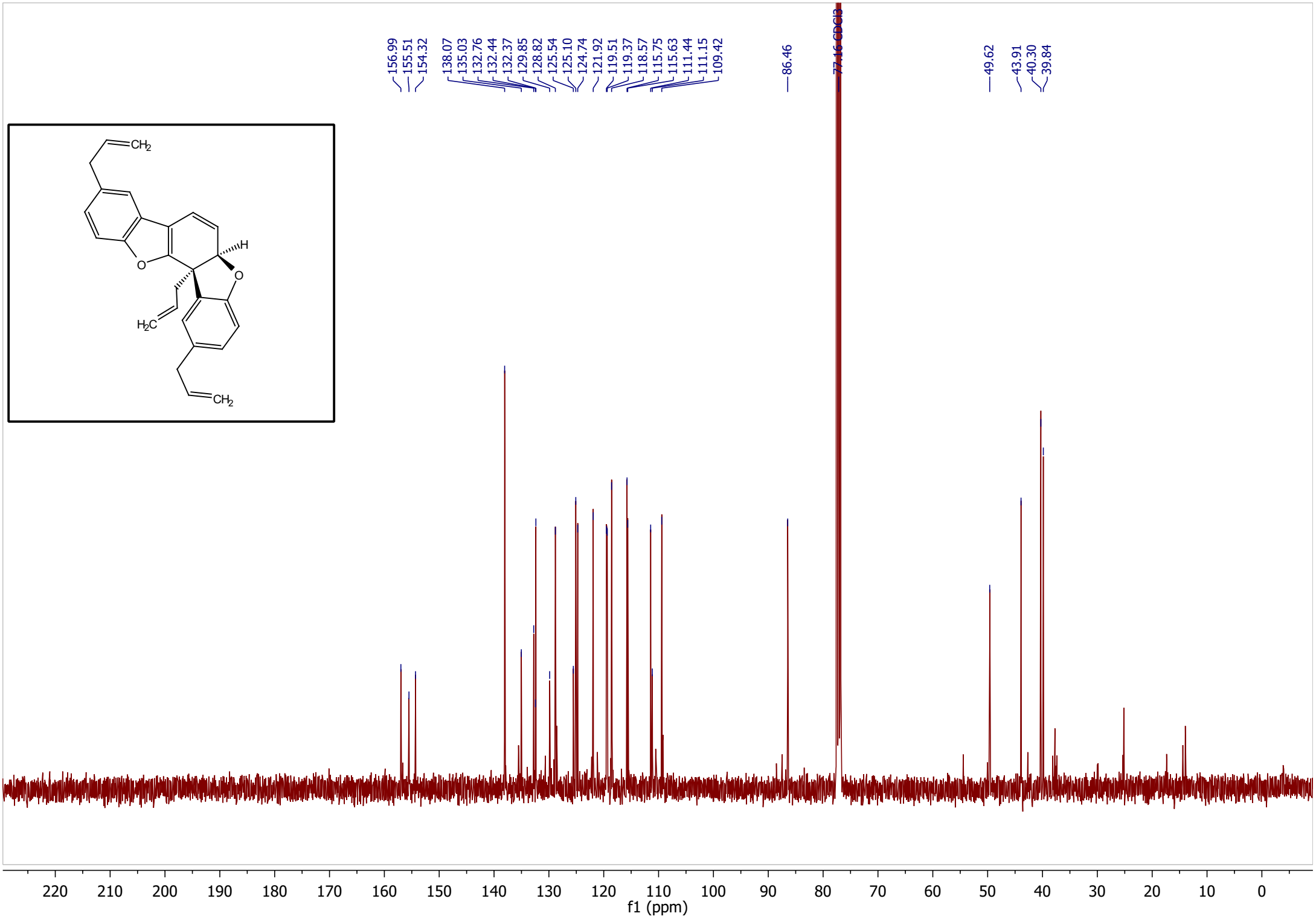


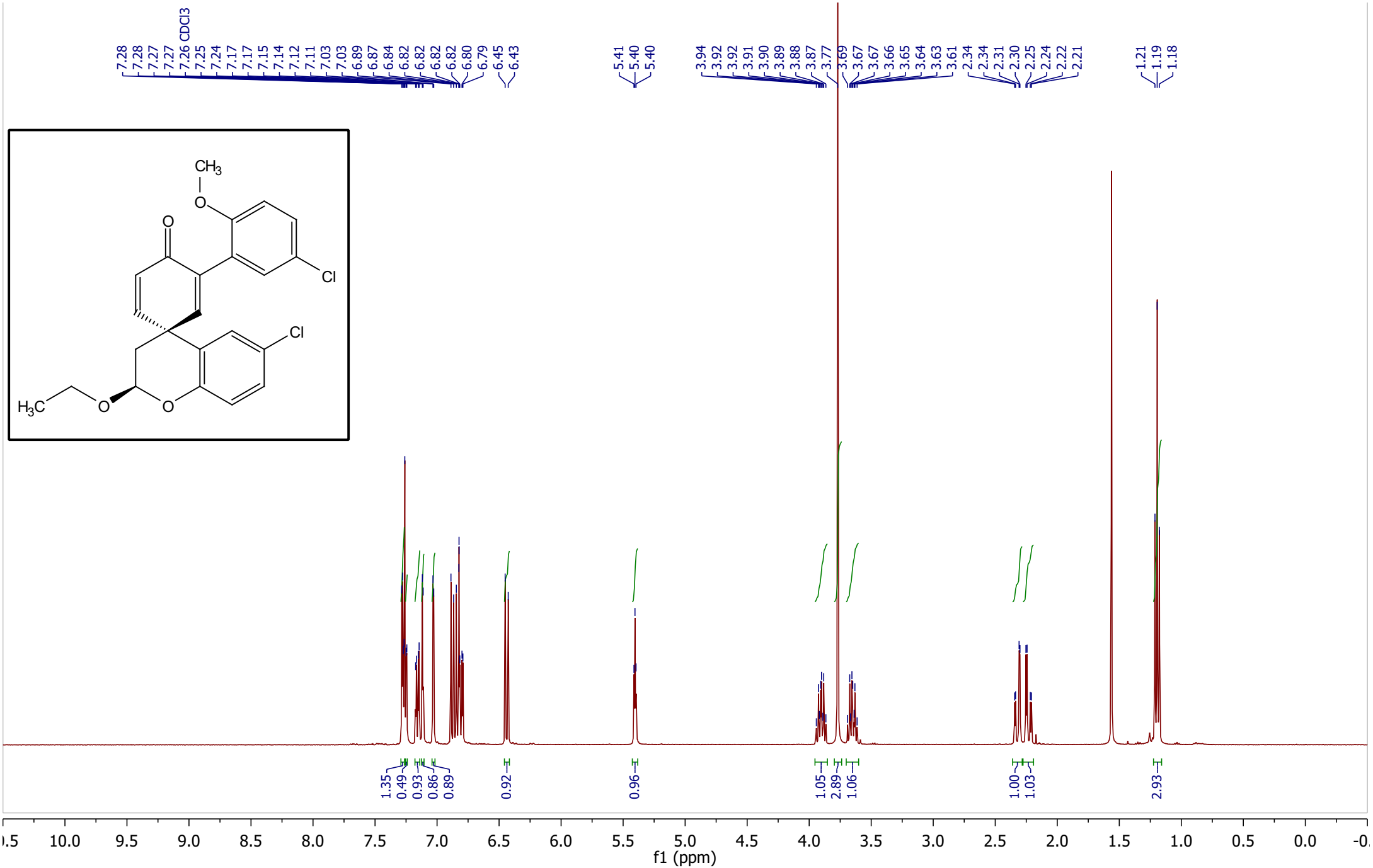
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.04 – 6.97 (m, 2H), 6.96 – 6.90 (m, 1H), 6.85 – 6.77 (m, 1H), 6.75 – 6.69 (m, 1H), 6.47 (d, *J* = 8.0 Hz, 1H), 5.96 – 5.81 (m, 2H), 5.79 – 5.60 (m, 2H), 5.26 – 5.12 (m, 2H), 5.05 – 4.88 (m, 4H), 4.86 – 4.65 (m, 2H), 3.24 (d, *J* = 6.4 Hz, 2H), 3.19 – 3.03 (m, 2H), 2.99 – 2.89 (m, 2H), 2.73 (dd, *J* = 14.0, 8.5 Hz, 1H), 2.16 – 2.08 (m, 1H).


162.01
159.41
140.20
138.25
137.94
133.43
132.34
131.30
130.27
128.77
128.58
128.27
126.82
124.47
121.18
119.50
115.61
115.10
109.99
107.70
85.90
83.52
77.16 CDCl3
54.75
43.30
39.72
39.57
30.92
CH2
H
O
O
H
H2C
CH2
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

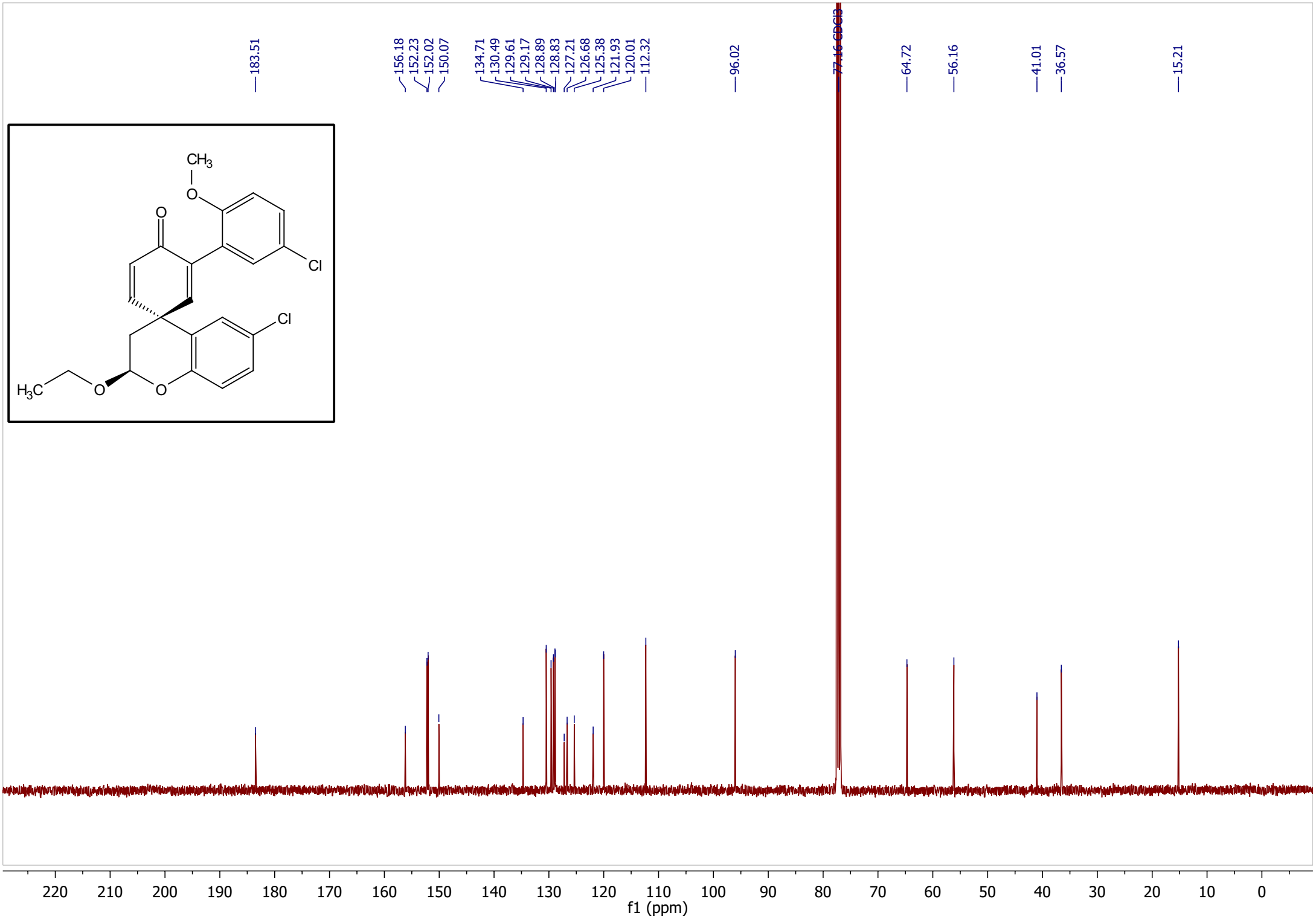


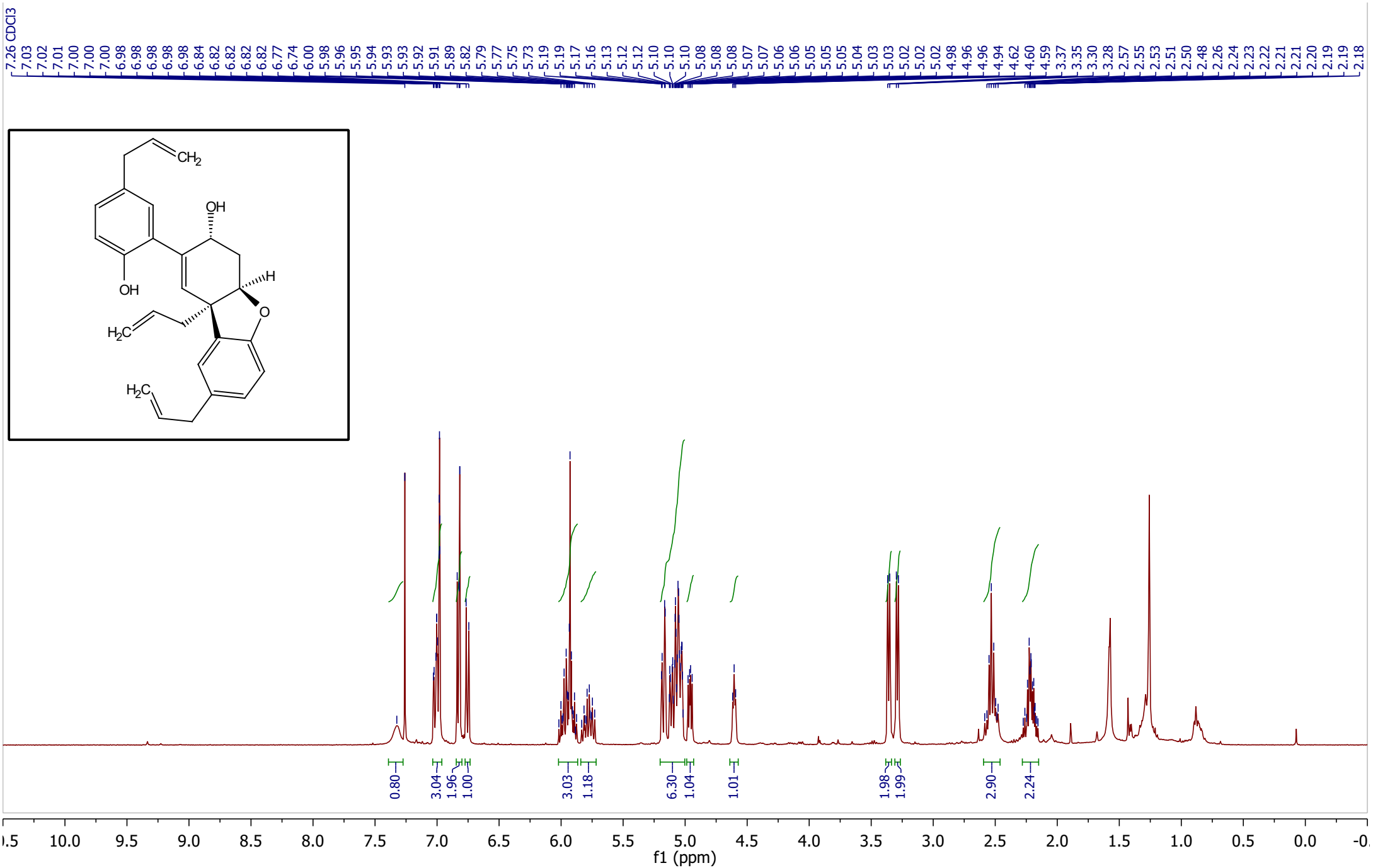
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.40 (d, *J* = 8.4 Hz, 1H), 7.36 (d, *J* = 1.8 Hz, 1H), 7.33 (d, *J* = 1.8 Hz, 1H), 7.07 (dd, *J* = 8.4, 1.8 Hz, 1H), 6.94 (dd, *J* = 8.0, 1.8 Hz, 1H), 6.75 (d, *J* = 8.0 Hz, 1H), 6.74 (d, *J* = 9.6 Hz, 1H), 5.97 (d, *J* = 9.6 Hz, 1H), 6.06 – 5.89 (m, 2H), 5.60 – 5.49 (m, 1H), 5.46 (d, *J* = 4.5 Hz, 1H), 5.14 – 4.99 (m, 6H), 3.46 (d, *J* = 6.6 Hz, 2H), 3.36 (d, *J* = 6.7 Hz, 2H), 3.08 (dd, *J* = 14.1, 7.0 Hz, 1H), 2.73 (dd, *J* = 14.1, 7.4 Hz, 1H).


156.99
155.51
154.32
138.07
135.03
132.76
132.44
132.37
129.85
128.82
125.54
125.10
124.74
121.92
119.51
119.37
118.57
115.75
115.63
111.44
111.15
109.42
86.46
77.16 CDCl3
49.62
43.91
40.30
39.84
CH2
H
O
O
H2C
CH2
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

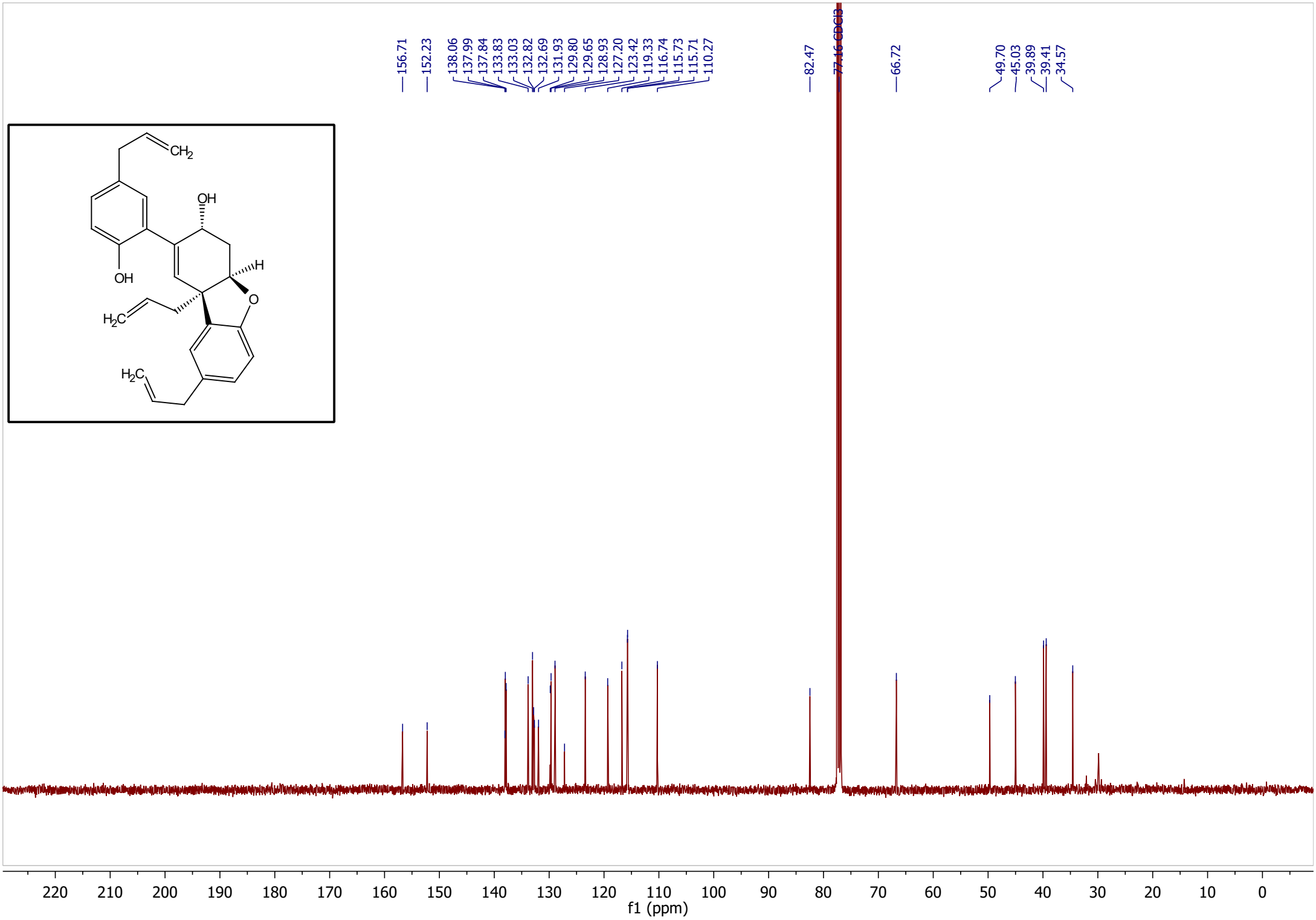


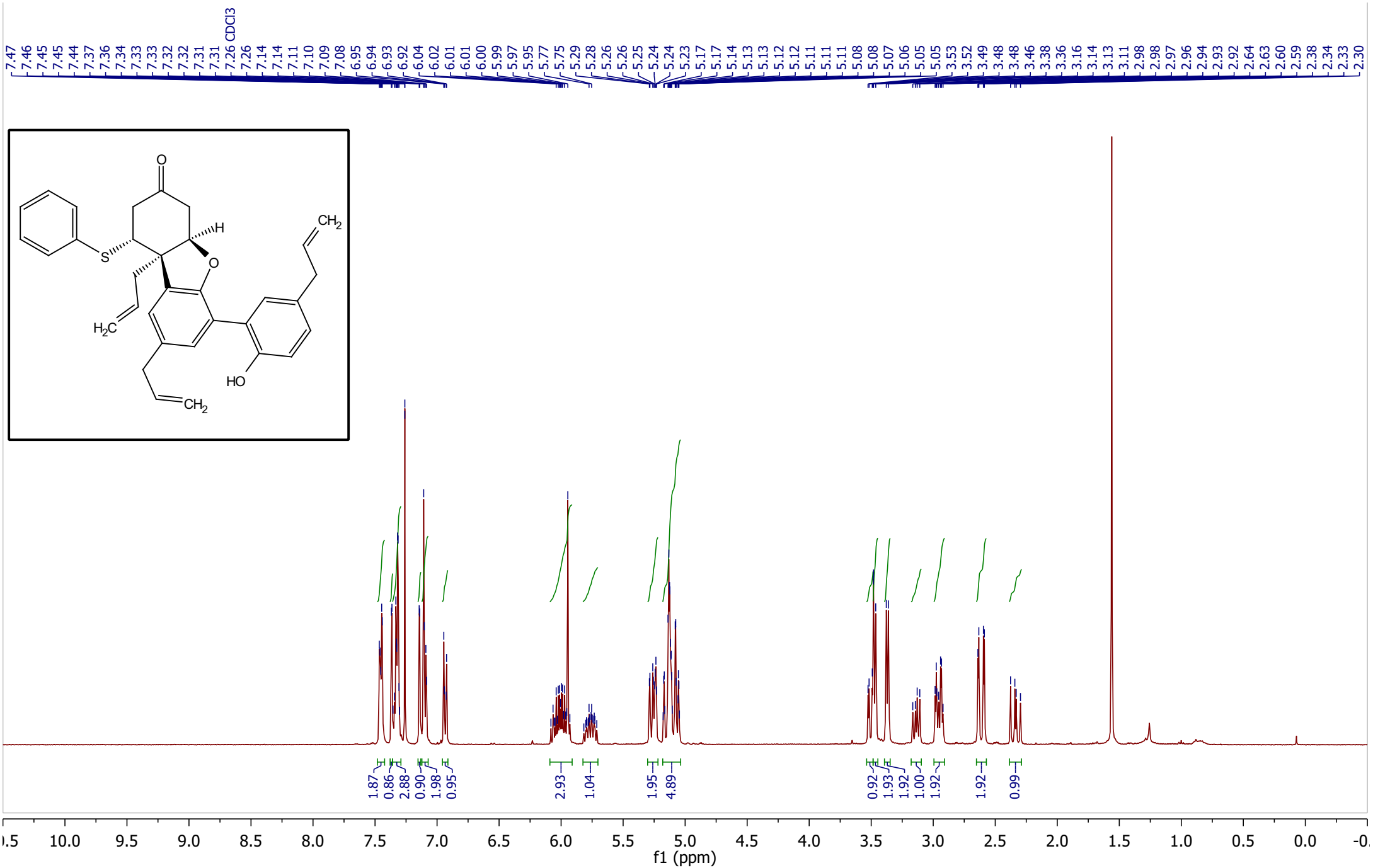
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.28 (d, *J* = 3.0 Hz, 1H), 7.26 (dd, *J* = 8.7, 2.7 Hz, 1H), 7.16 (dd, *J* = 8.7, 2.5 Hz, 1H), 7.12 (d, *J* = 2.7 Hz, 1H), 7.03 (d, *J* = 2.5 Hz, 1H), 6.88 (d, *J* = 8.7 Hz, 1H), 6.83 (d, *J* = 8.7 Hz, 1H), 6.81 (dd, *J* = 9.9, 3.0 Hz, 1H), 6.44 (d, *J* = 9.9 Hz, 1H), 5.40 (dd, *J* = 3.7, 2.9 Hz, 1H), 3.90 (dq, *J* = 9.6, 7.1 Hz, 1H), 3.77 (s, 3H), 3.65 (dq, *J* = 9.6, 7.1 Hz, 1H), 2.32 (dd, *J* = 14.1, 2.9 Hz, 1H), 2.23 (dd, *J* = 14.1, 3.7 Hz, 1H), 1.19 (dd, *J* = 7.1, 7.1 Hz, 3H).


183.51
156.18
152.23
152.02
150.07
134.71
130.49
129.61
129.17
128.89
128.83
127.21
126.68
125.38
121.93
120.01
112.32
96.02
77.16 CDCl3
64.72
56.16
41.01
36.57
15.21
CH3
O
O
Cl
Cl
H3C
O
O
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

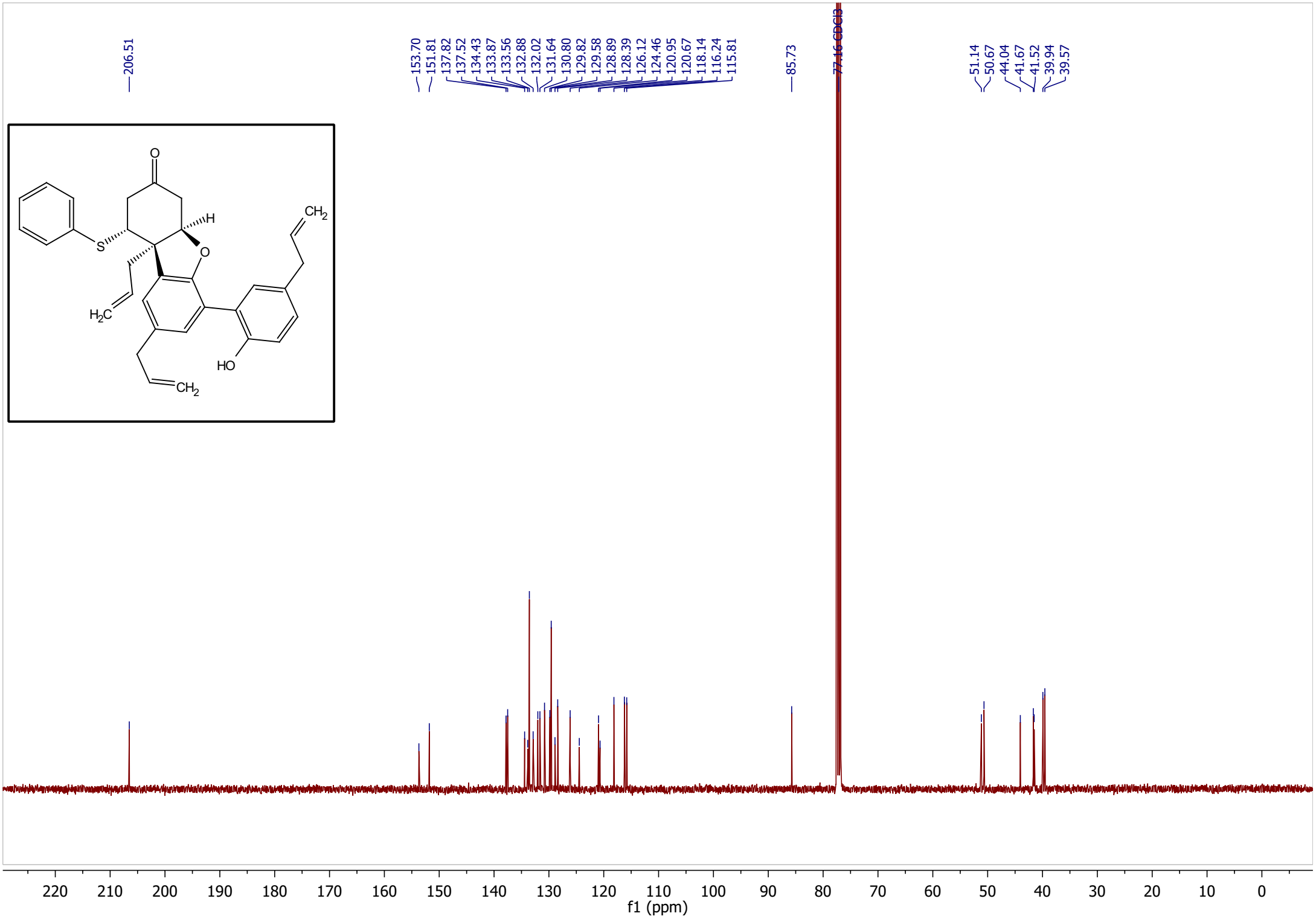


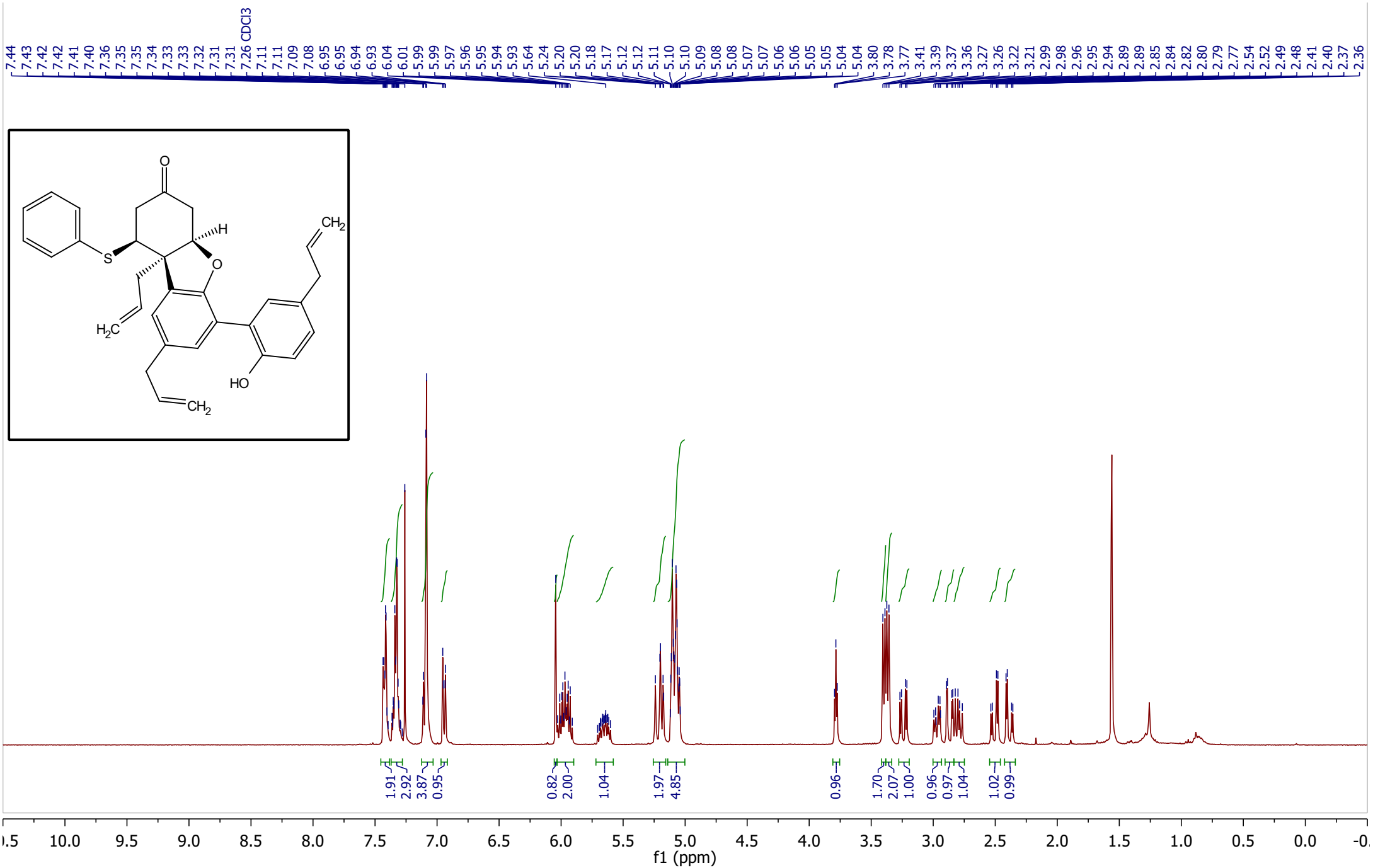
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.32 (s, 1H), 7.02 (dd, *J* = 8.3, 2.4 Hz, 1H), 6.99 (dd, *J* = 8.3, 2.2 Hz, 1H), 6.98 (d, *J* = 2.2 Hz, 1H), 6.83 (d, *J* = 8.3 Hz, 1H), 6.82 (d, *J* = 2.4 Hz, 1H), 6.75 (d, *J* = 8.3 Hz, 1H), 6.04 – 5.85 (m, 2H), 5.93 (s, 1H), 5.78 (dddd, *J* = 17.3, 10.1, 7.3 Hz, 1H), 5.21 – 5.00 (m, 6H), 4.96 (dd, *J* = 8.1, 5.1 Hz, 1H), 4.60 (dd, *J* = 4.8 Hz, 1H), 3.36 (d, *J* = 6.7 Hz, 2H), 3.29 (d, *J* = 6.7 Hz, 2H), 2.59 – 2.45 (m, 3H), 2.28 – 2.14 (m, 2H).


156.71
152.23
138.06
137.99
137.84
133.83
133.03
132.82
132.69
131.93
129.80
129.65
128.93
127.20
123.42
119.33
116.74
115.73
115.71
110.27
82.47
77.16 CDCl3
66.72
49.70
45.03
39.89
39.41
34.57
OH
OH
H
O
CH2
H2C
H2C
220
210
200
190
180
170
160
150
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130
120
110
100
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80
70
60
50
40
30
20
10
0
f1 (ppm)

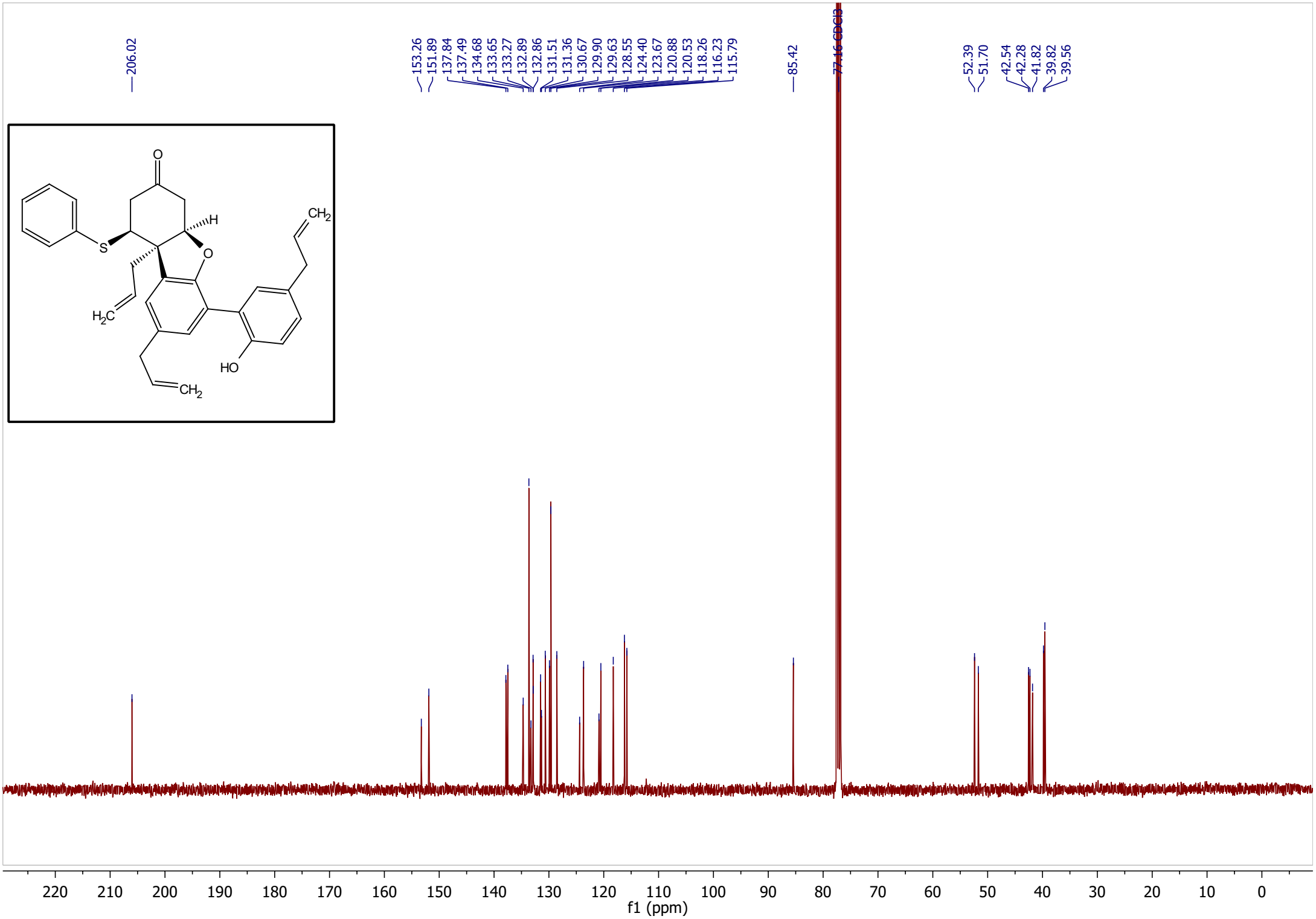


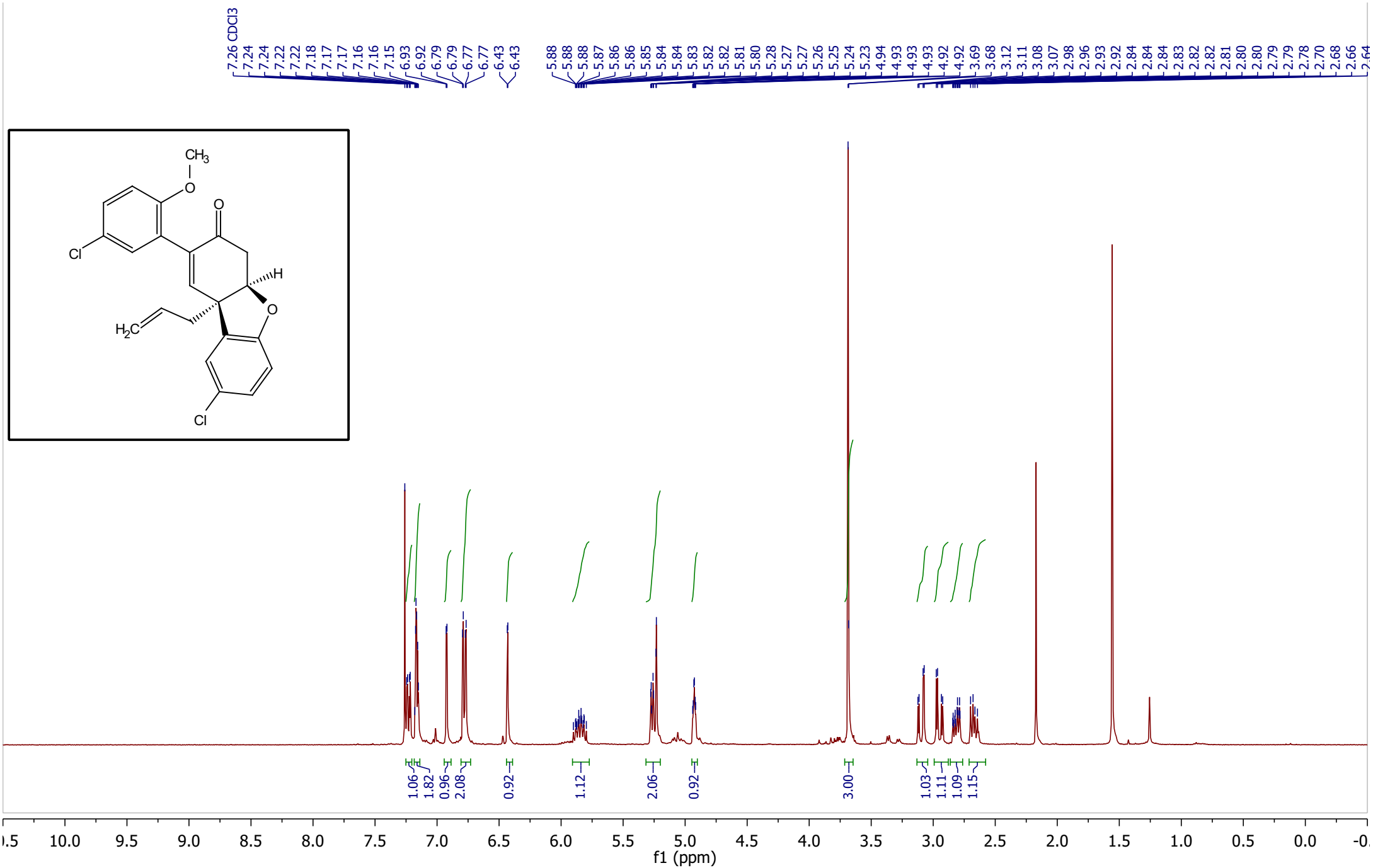
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.48 – 7.43 (m, 2H), 7.37 (d, *J* = 1.7 Hz, 1H), 7.36 – 7.30 (m, 3H), 7.14 (d, *J* = 1.7 Hz, 1H), 7.12 – 7.07 (m, 2H), 6.97 – 6.90 (m, 1H), 6.11 – 5.90 (m, 2H), 5.95 (s, 1H), 5.76 (dddd, *J* = 16.5, 10.0, 8.4, 6.3 Hz, 1H), 5.31 – 5.22 (m, 2H), 5.19 – 5.04 (m, 5H), 3.50 (dd, *J* = 13.9, 3.3 Hz, 1H), 3.47 (d, *J* = 7.0 Hz, 2H), 3.37 (d, *J* = 6.7 Hz, 2H), 3.14 (dd, *J* = 14.2, 8.4 Hz, 1H), 2.99 – 2.91 (m, 2H), 2.64 – 2.58 (m, 2H), 2.34 (dd, *J* = 18.1, 13.9 Hz, 1H).


206.51
153.70
151.81
137.82
137.52
134.43
133.87
133.56
132.88
132.02
131.64
130.80
129.82
129.58
128.89
128.39
126.12
124.46
120.95
120.67
118.14
116.24
115.81
85.73
77.16 CDCl3
51.14
50.67
44.04
41.67
41.52
39.94
39.57
O
H
S
O
CH2
H2C
HO
CH2
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.46 – 7.37 (m, 2H), 7.38 – 7.28 (m, 3H), 7.13 – 7.04 (m, 4H), 6.96 – 6.92 (m, 1H), 6.04 (s, 1H), 6.03 – 5.89 (m, 2H), 5.65 (dddd, *J* = 16.9, 10.1, 8.8, 5.5 Hz, 1H), 5.26 – 5.16 (m, 2H), 5.14 – 5.02 (m, 5H), 3.78 (dd, *J* = 4.6, 4.0 Hz, 1H), 3.40 (d, *J* = 6.7 Hz, 2H), 3.37 (d, *J* = 6.8 Hz, 2H), 3.24 (dd, *J* = 17.5, 4.9 Hz, 1H), 2.97 (dd, *J* = 14.1, 5.6 Hz, 1H), 2.87 (dd, *J* = 17.5, 3.0 Hz, 1H), 2.79 (dd, *J* = 14.1, 8.8 Hz, 1H), 2.51 (dd, *J* = 18.1, 4.6 Hz, 1H), 2.39 (dd, *J* = 18.1, 4.0 Hz, 1H).


206.02
153.26
151.89
137.84
137.49
134.68
133.65
133.27
132.89
132.86
131.51
131.36
130.67
129.90
129.63
128.55
124.40
123.67
120.88
120.53
118.26
116.23
115.79
85.42
77.16 CDCl3
52.39
51.70
42.54
42.28
41.82
39.82
39.56
O
H
S
O
CH2
H2C
HO
CH2
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.23 (dd, *J* = 8.7, 2.7 Hz, 1H), 7.18 – 7.14 (m, 2H), 6.92 (d, *J* = 2.7 Hz, 1H), 6.78 (d, *J* = 8.7 Hz, 1H), 6.78 (d, *J* = 9.1 Hz, 1H), 6.43 (d, *J* = 1.7 Hz, 1H), 5.85 (dddd, *J* = 17.0, 10.4, 8.0, 6.7 Hz, 1H), 5.29 – 5.21 (m, 2H), 4.93 (ddd, *J* = 4.2, 3.5, 1.7 Hz, 1H), 3.69 (s, 3H), 3.10 (dd, *J* = 16.6, 3.5 Hz, 1H), 2.95 (dd, *J* = 16.6, 4.2 Hz, 1H), 2.81 (dddd, *J* = 14.2, 6.7, 1.3, 1.3 Hz, 1H), 2.67 (dd, *J* = 14.2, 8.0 Hz, 1H).