Synthesis of Solandelactone F, Constanolactone A, Advanced Intermediate Towards Solandelactone E from a Common Synthetic Intermediate

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Supplementary Information

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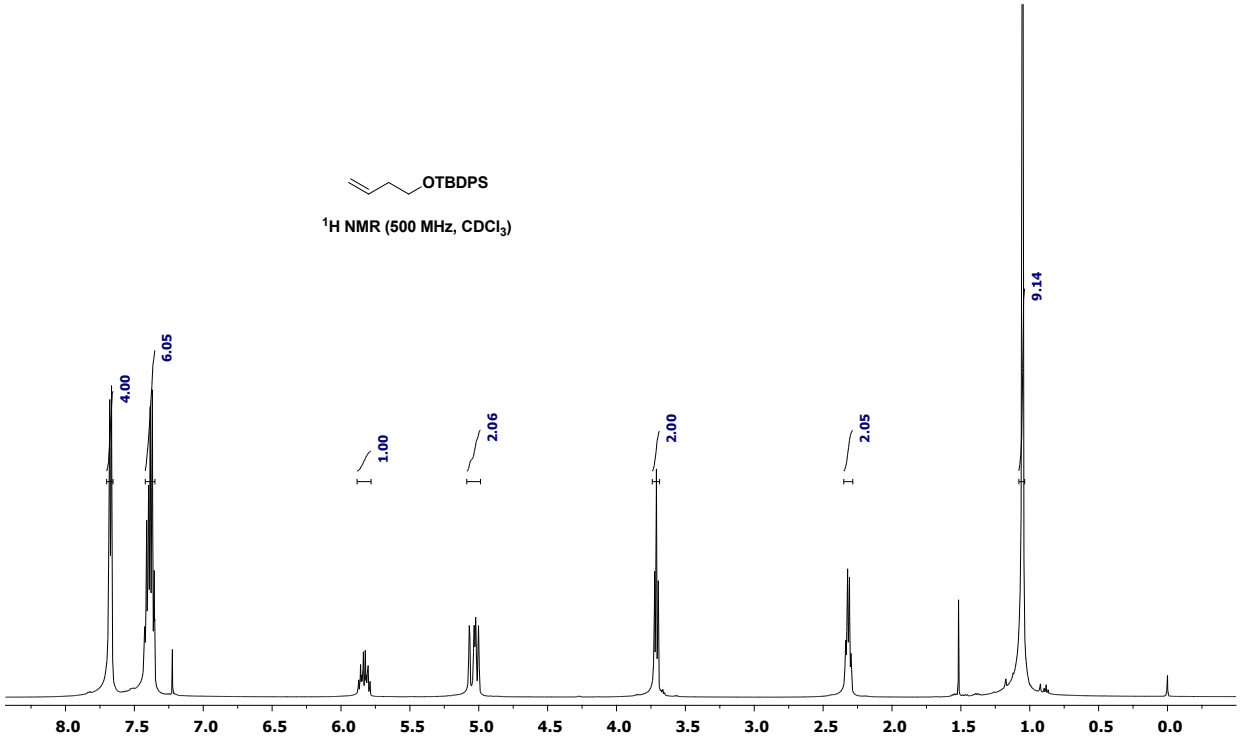
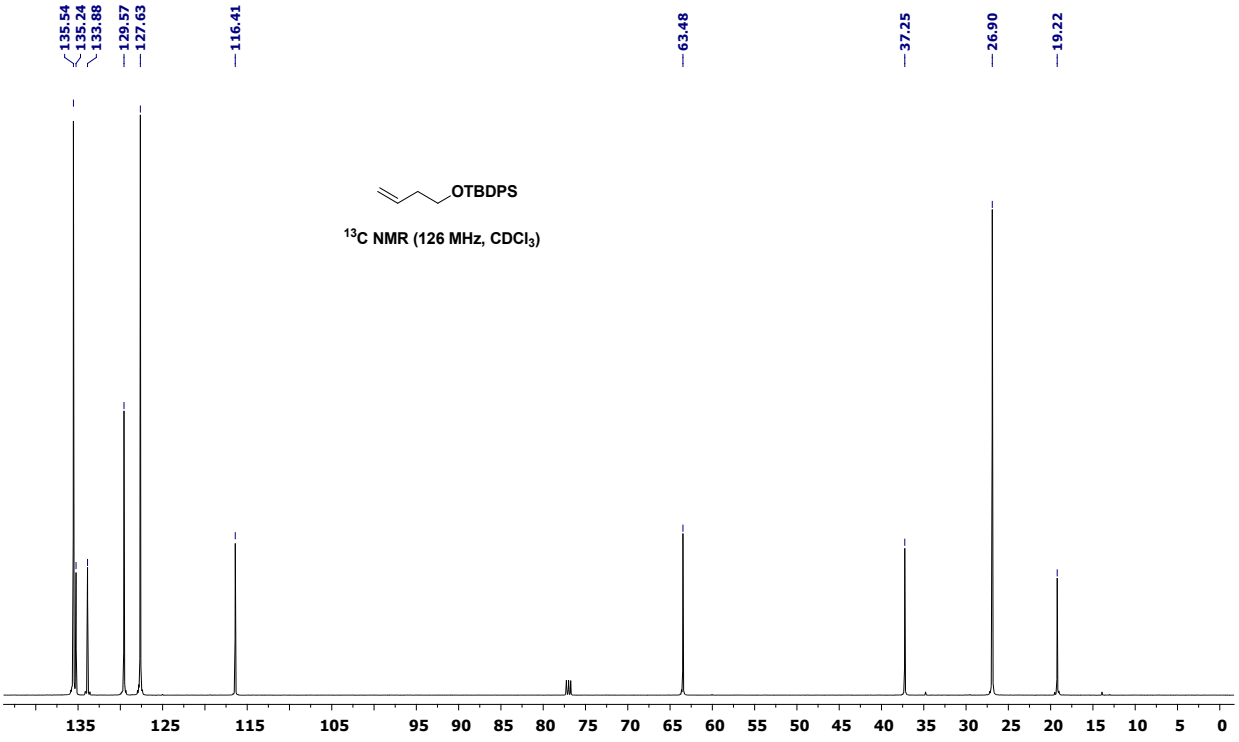
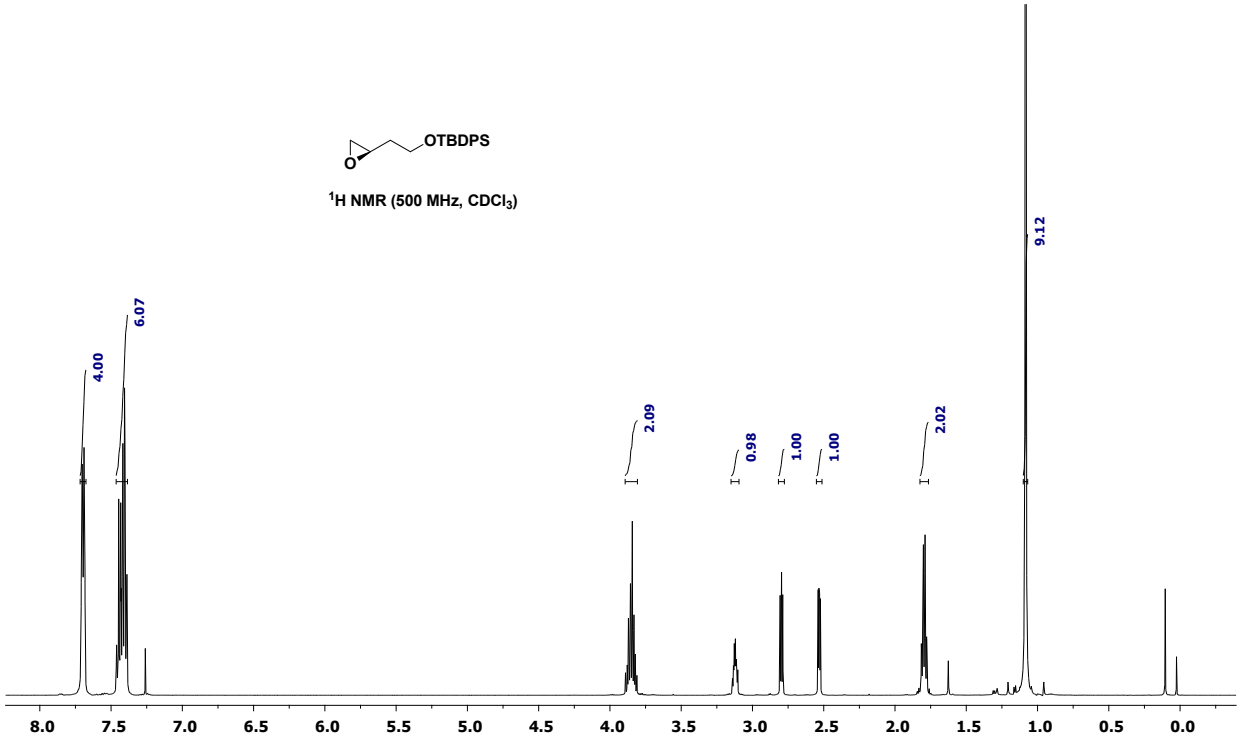
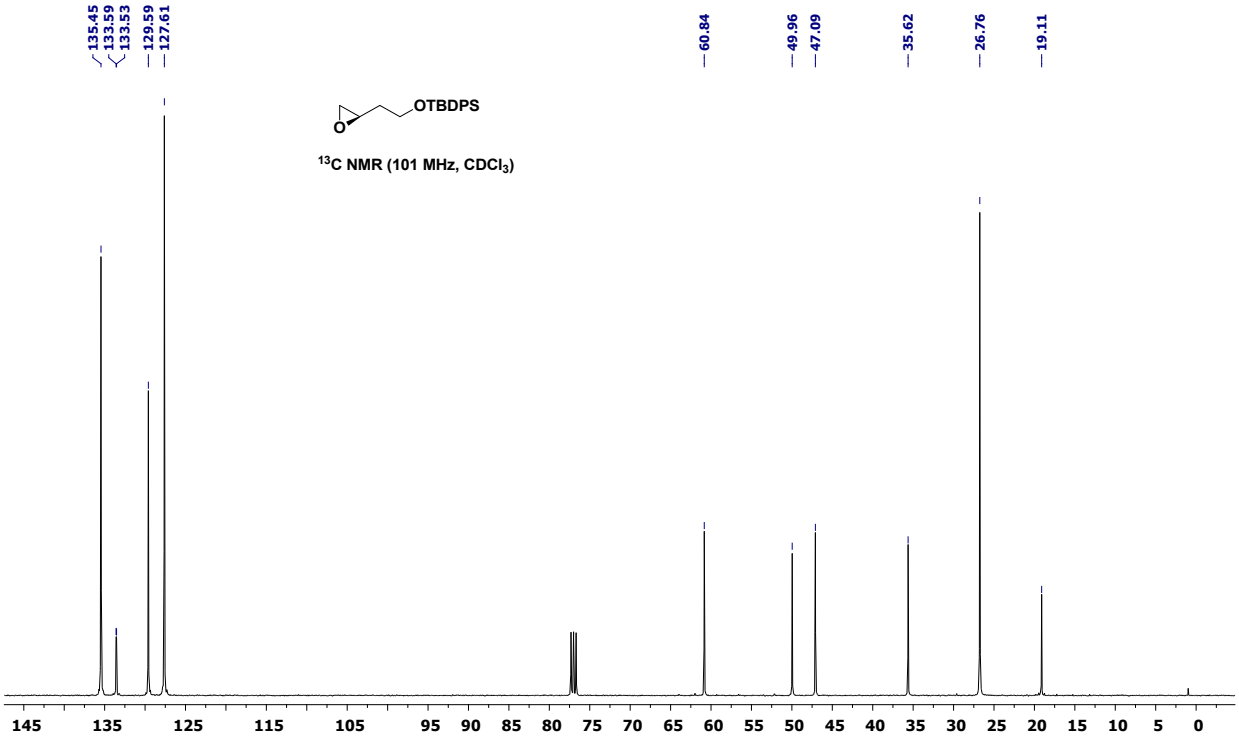
(But-3-en-1-yloxy) (*tert*-butyl)diphenylsilane (V):



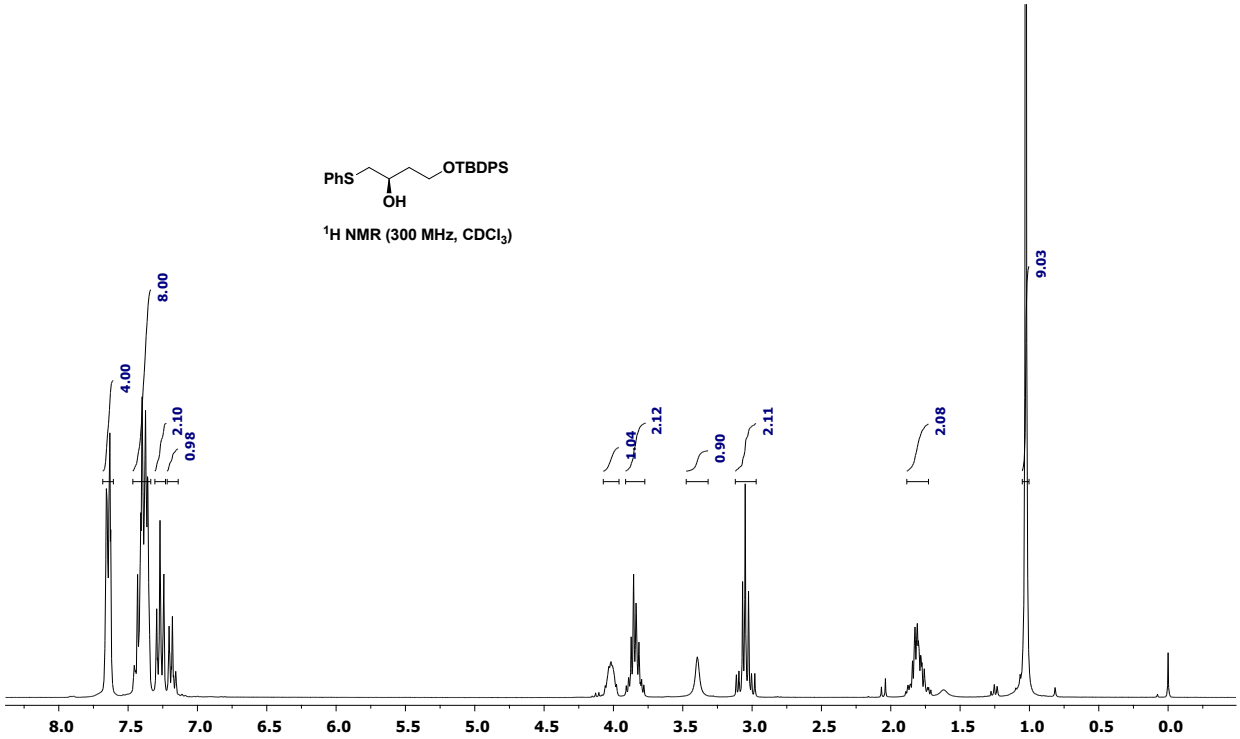


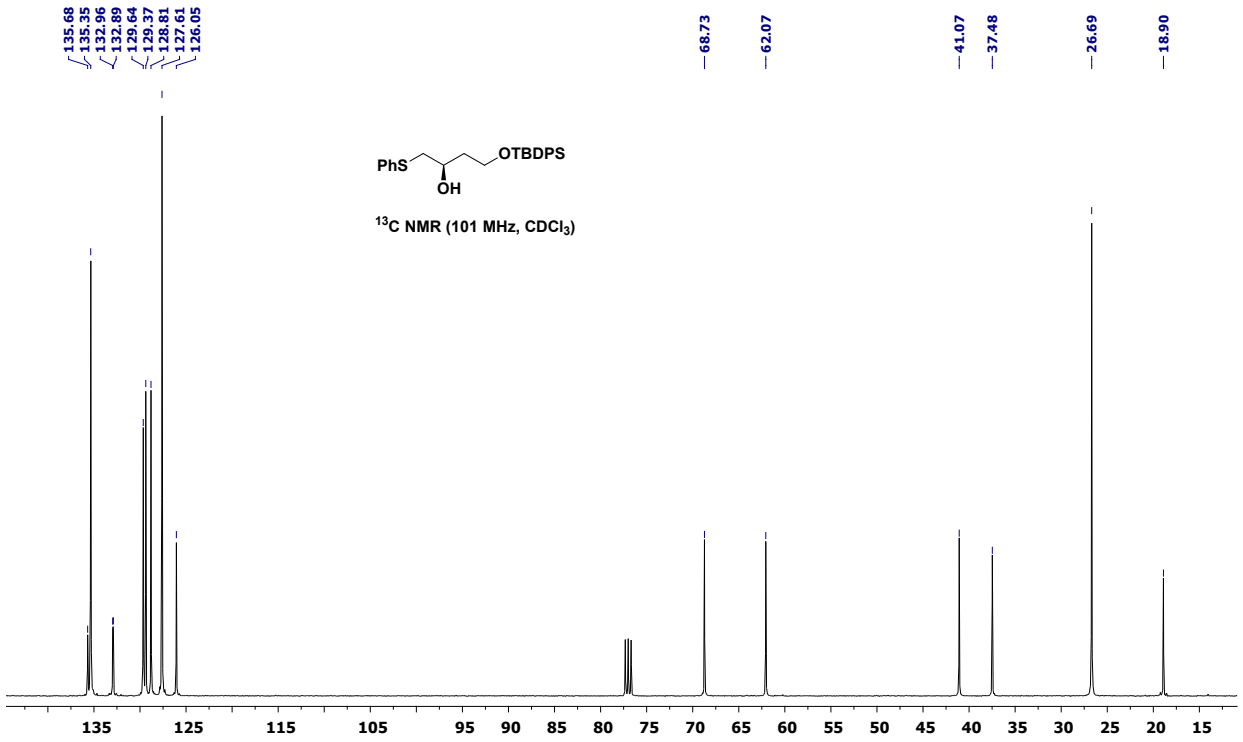
Compound 12:


OTBDPS
O
^{1}H NMR (500 MHz, CDCl$_3$)
4.00
6.07
2.09
0.98
1.00
1.00
2.02
9.12
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

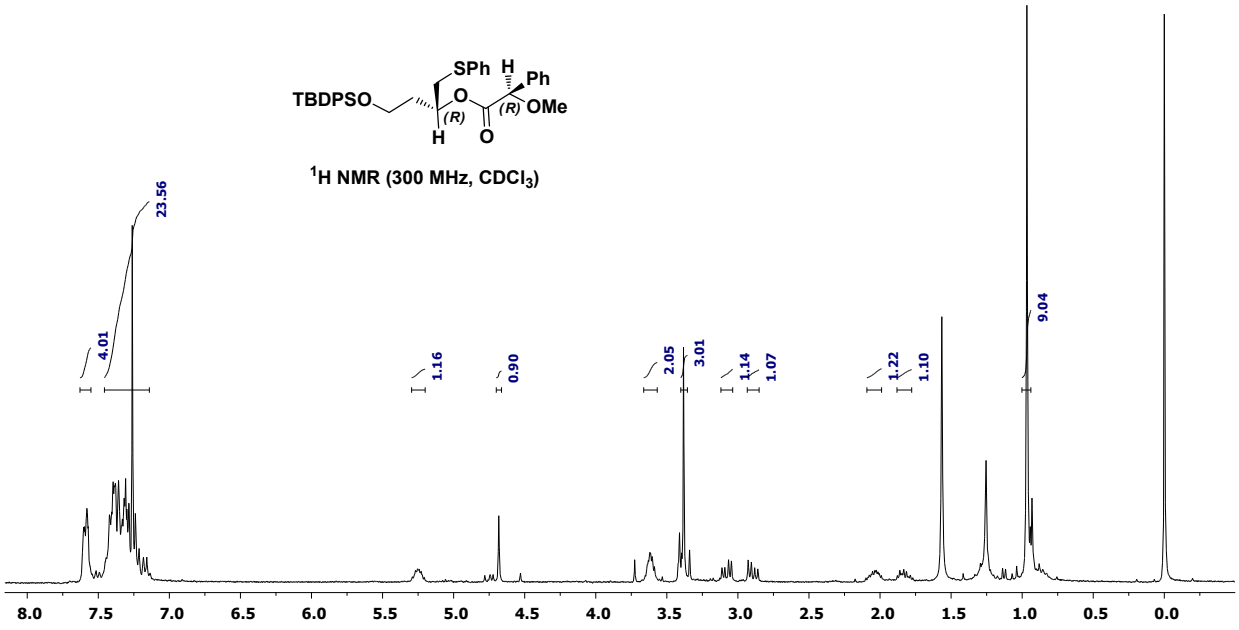

135.45
133.59
133.53
129.59
127.61
60.84
49.96
47.09
35.62
26.76
19.11
OTBDPS
O
^{13}C NMR (101 MHz, CDCl$_3$)
145
135
125
115
105
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0

Compound 13:

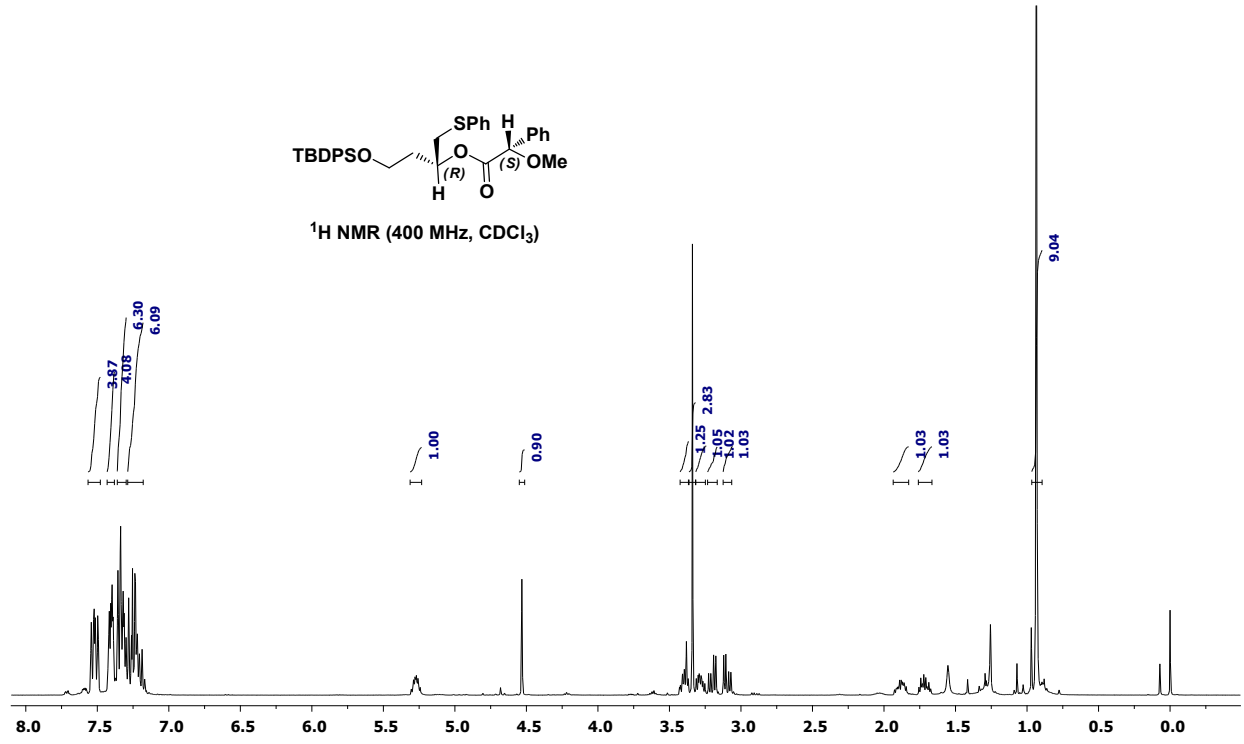

PhS
OTBDPS
OH
^{1}H NMR (300 MHz, CDCl$_3$)
4.00
8.00
2.10
0.98
1.04
2.12
0.90
2.11
2.08
9.03
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0


135.68
135.35
132.96
132.89
129.64
129.37
128.81
127.61
126.05
68.73
62.07
41.07
37.48
26.69
18.90
PhS
OTBDPS
OH
^{13}C NMR (101 MHz, CDCl$_3$)
135 125 115 105 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15

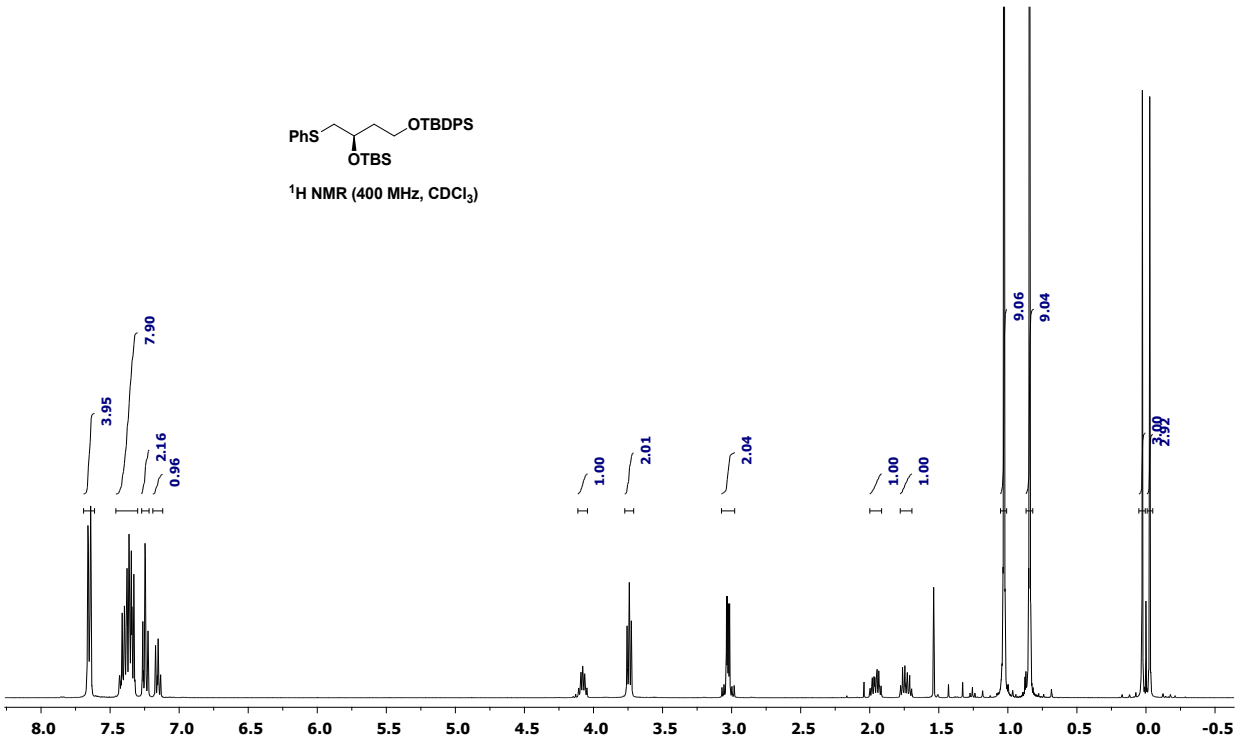
(*R*)-Mandelate ester of compound 13:

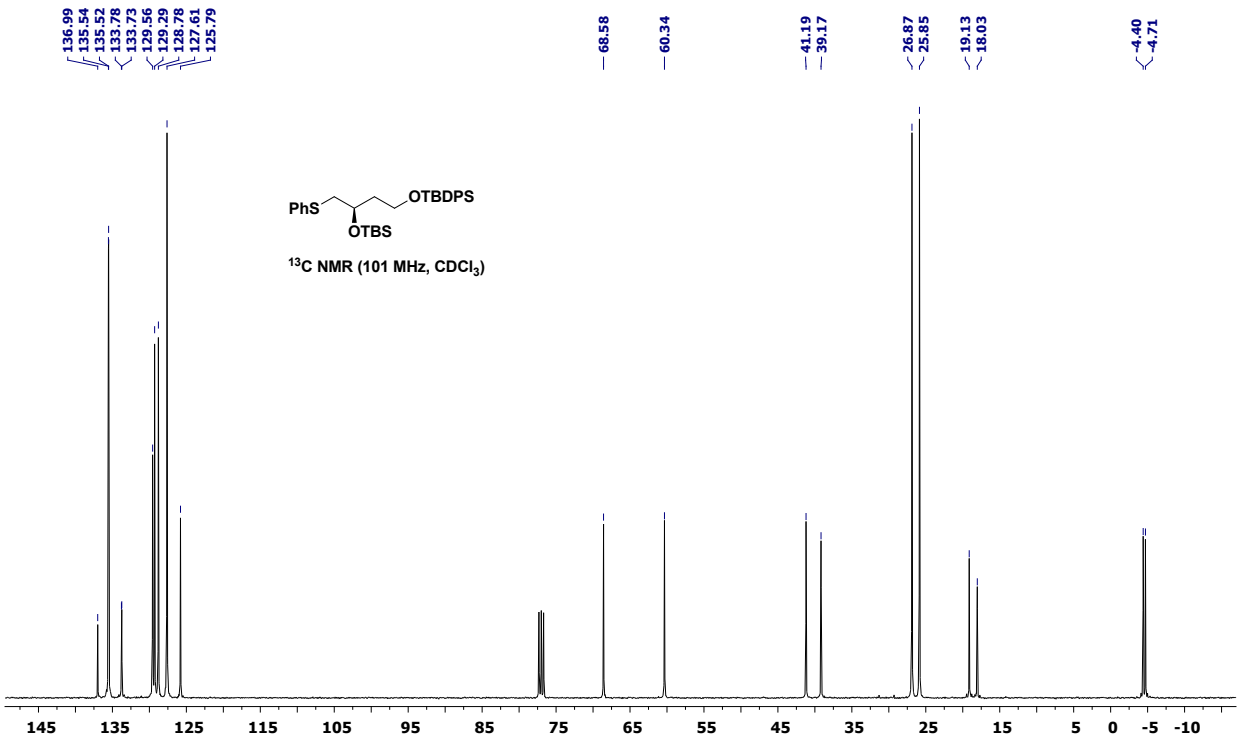


(*S*)-Mandelate ester of compound 13:

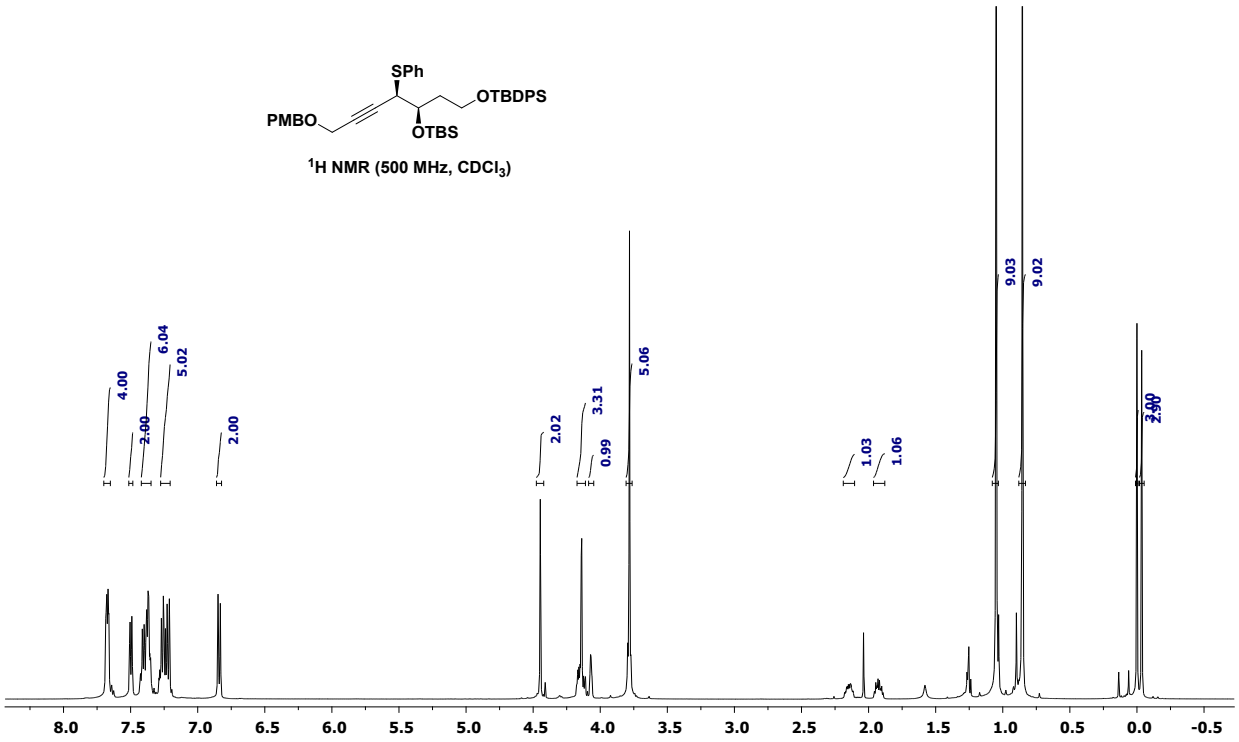


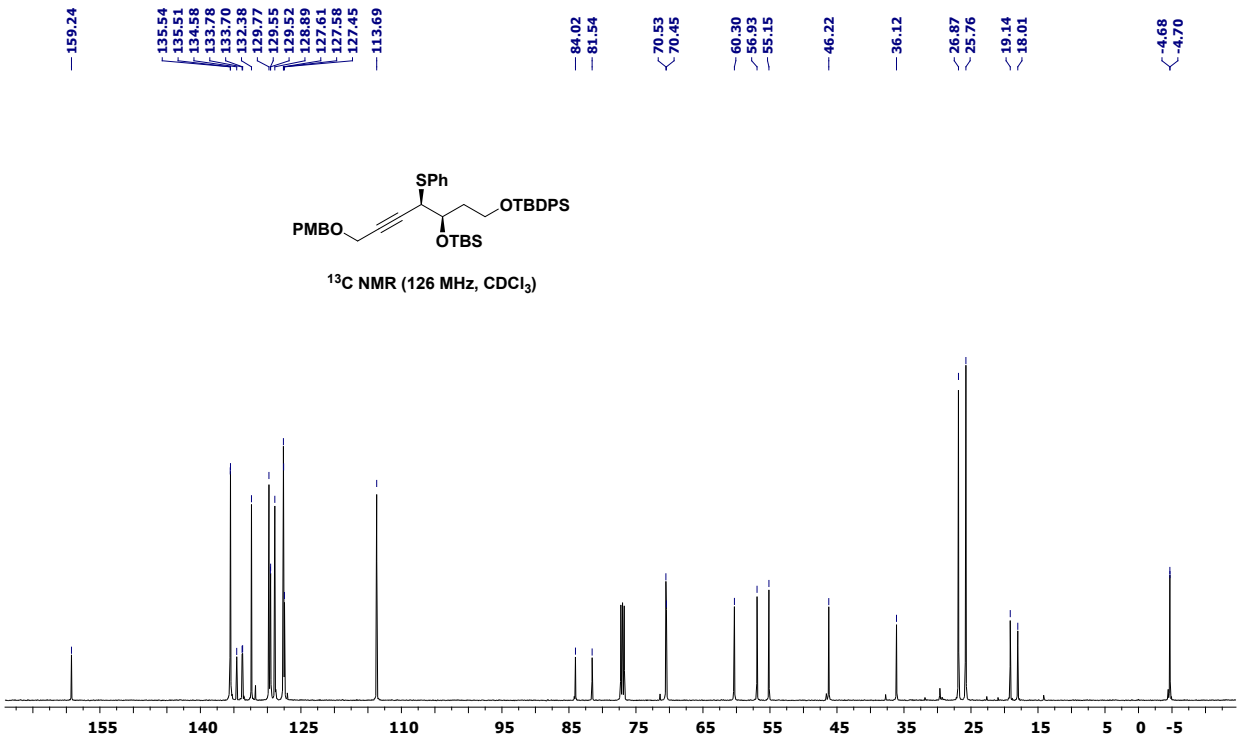
Compound 14:


PhS
OTBDPS
OTBS
^{1}H NMR (400 MHz, CDCl$_3$)
3.95
7.90
2.16
0.96
1.00
2.01
2.04
1.00
1.00
9.06
9.04
3.00
2.92
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

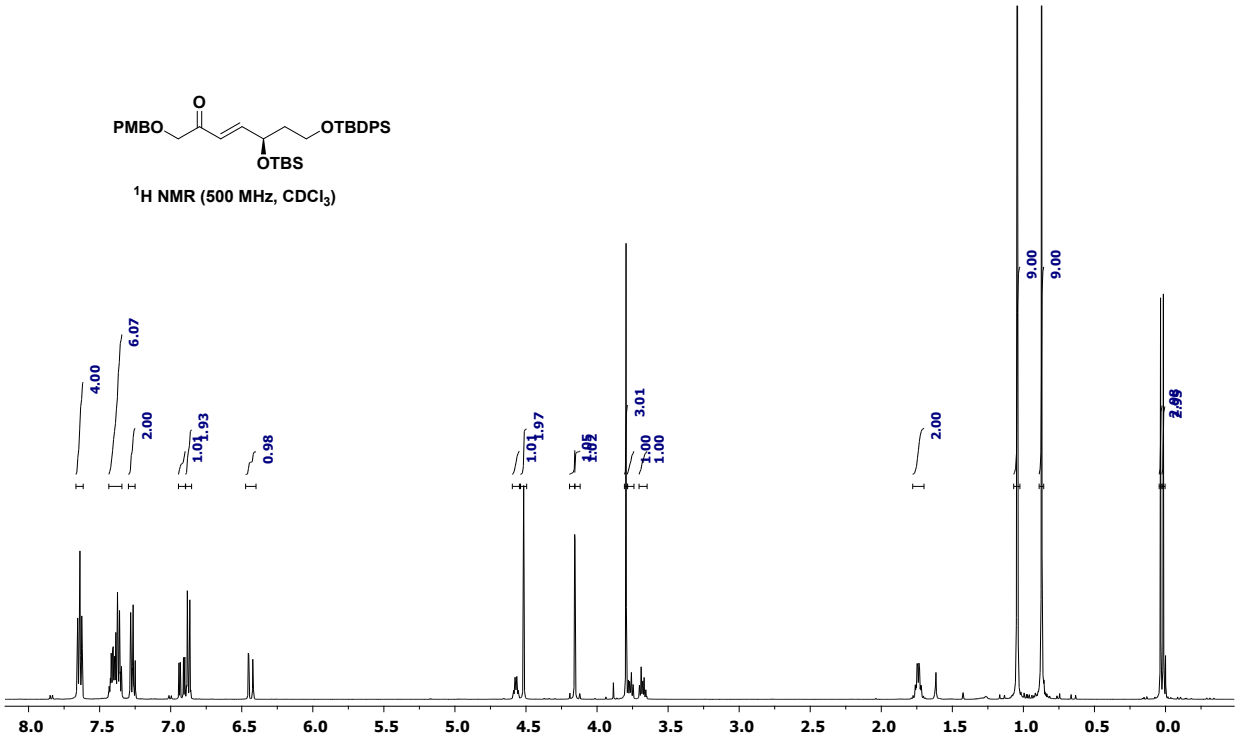

136.99
135.54
135.52
133.78
133.73
129.56
129.29
128.78
127.61
125.79
68.58
60.34
41.19
39.17
26.87
25.85
19.13
18.03
-4.40
-4.71
PhS
OTBDPS
OTBS
^{13}C NMR (101 MHz, CDCl$_3$)
145 135 125 115 105 95 85 75 65 55 45 35 25 15 5 0 -5 -10

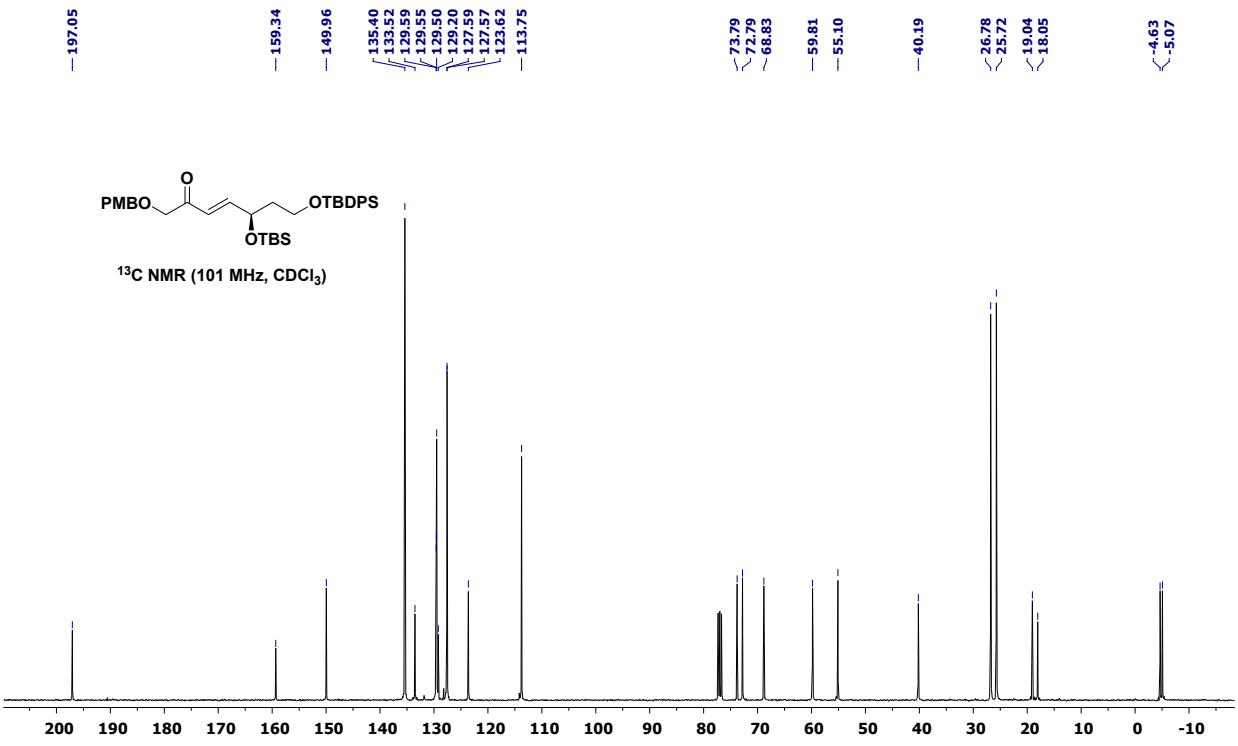
Compound 17:


SPh
OTBDPS
PMBO
OTBS
^{1}H NMR (500 MHz, CDCl$_3$)
4.00
2.00
6.04
5.02
2.00
2.02
3.31
0.99
5.06
1.03
1.06
9.03
9.02
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

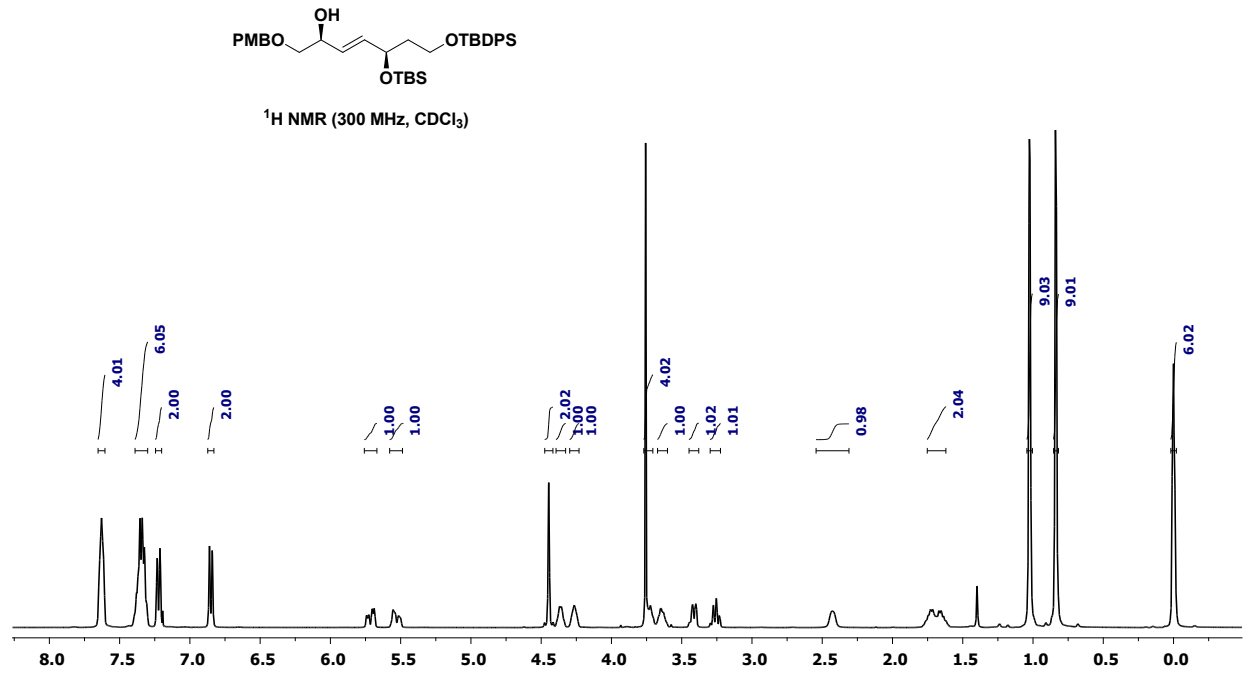

SPh
OTBDPS
PMBO
OTBS
^{13}C NMR (126 MHz, CDCl$_3$)
159.24
135.54
135.51
134.58
133.78
133.70
132.38
129.77
129.55
129.52
128.89
127.61
127.58
127.45
113.69
84.02
81.54
70.53
70.45
60.30
56.93
55.15
46.22
36.12
26.87
25.76
19.14
18.01
-4.68
-4.70
155
140
125
110
95
85
75
65
55
45
35
25
15
5
0
-5

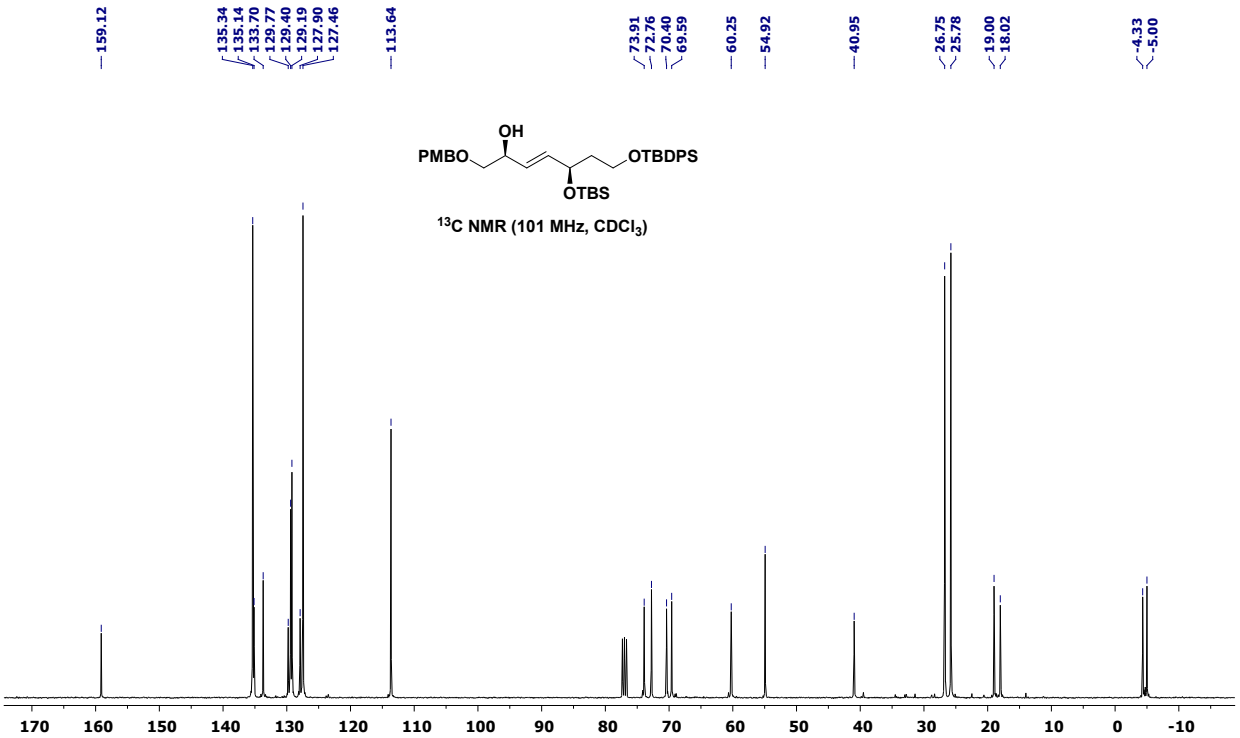
Compound 18:


PMBO
O
OTBDPS
OTBS
^{1}H NMR (500 MHz, CDCl$_3$)
4.00
6.07
2.00
1.01
1.93
0.98
1.01
1.97
1.05
1.02
3.01
1.00
1.00
2.00
9.00
9.00
2.00
2.00
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

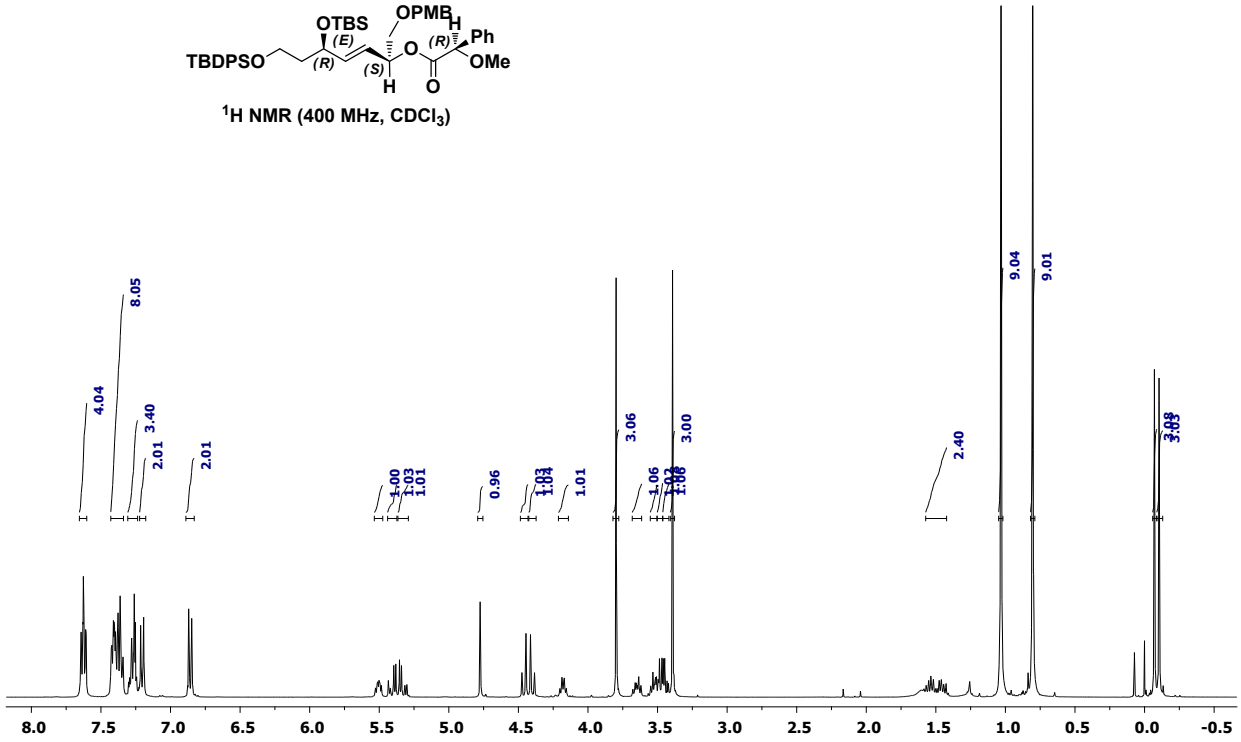

197.05
159.34
149.96
135.40
133.52
129.59
129.55
129.50
129.20
127.59
127.57
123.62
113.75
73.79
72.79
68.83
59.81
55.10
40.19
26.78
25.72
19.04
18.05
-4.63
-5.07
PMBO
O
OTBDPS
OTBS
^{13}C NMR (101 MHz, CDCl$_3$)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10

Compound 19:

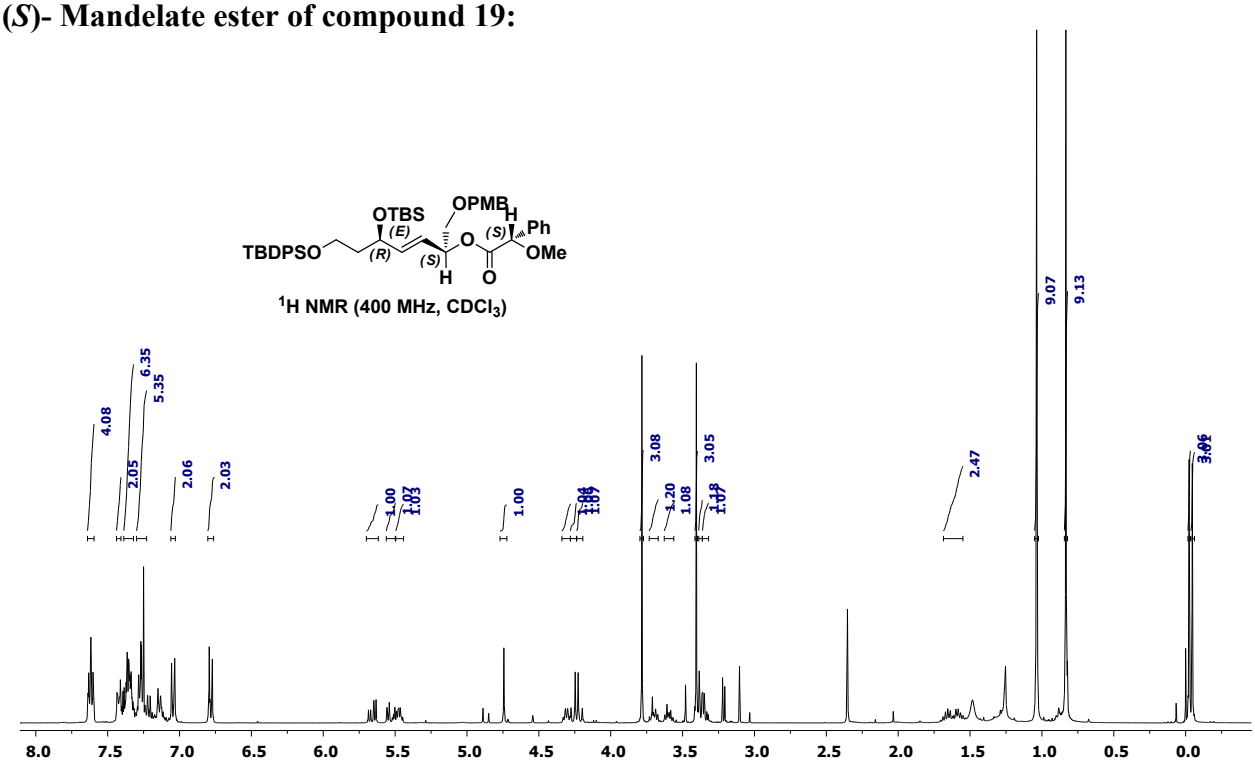

OH
PMBO
OTBDPS
OTBS
^{1}H NMR (300 MHz, CDCl$_3$)
4.01
6.05
2.00
2.00
1.00
1.00
2.02
1.00
1.00
4.02
1.00
1.02
1.01
0.98
2.04
9.03
9.01
6.02
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0


159.12
135.34
135.14
133.70
129.77
129.40
129.19
127.90
127.46
113.64
73.91
72.76
70.40
69.59
60.25
54.92
40.95
26.75
25.78
19.00
18.02
-4.33
-5.00
OH
PMBO
OTBDPS
OTBS
^{13}C NMR (101 MHz, CDCl$_3$)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10

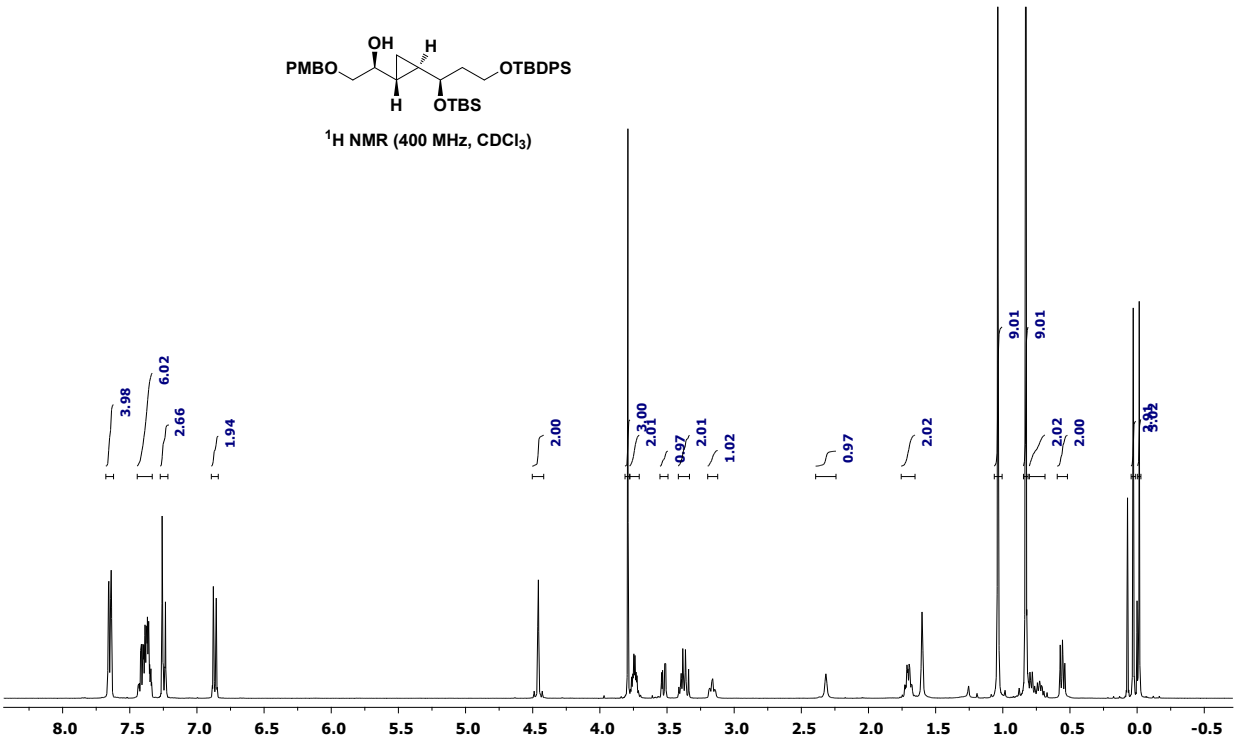
(*R*)- Mandelate ester of compound 19:

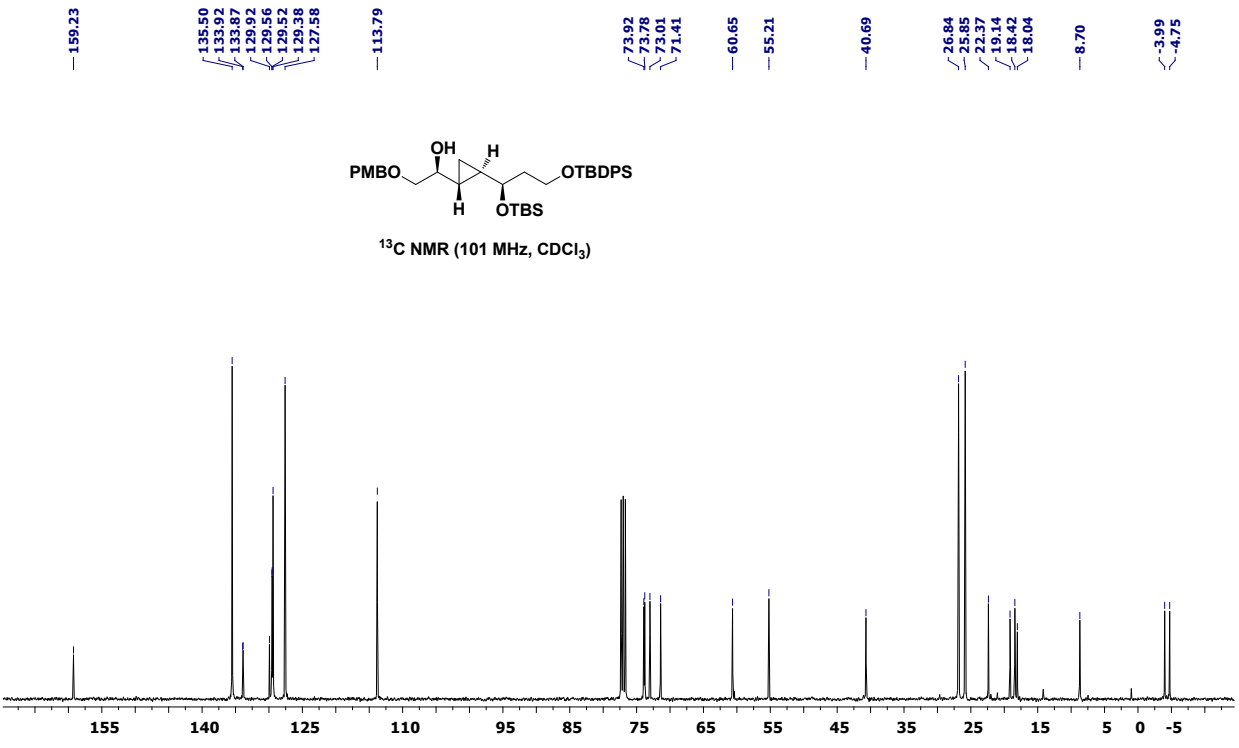


(*S*)- Mandelate ester of compound 19:

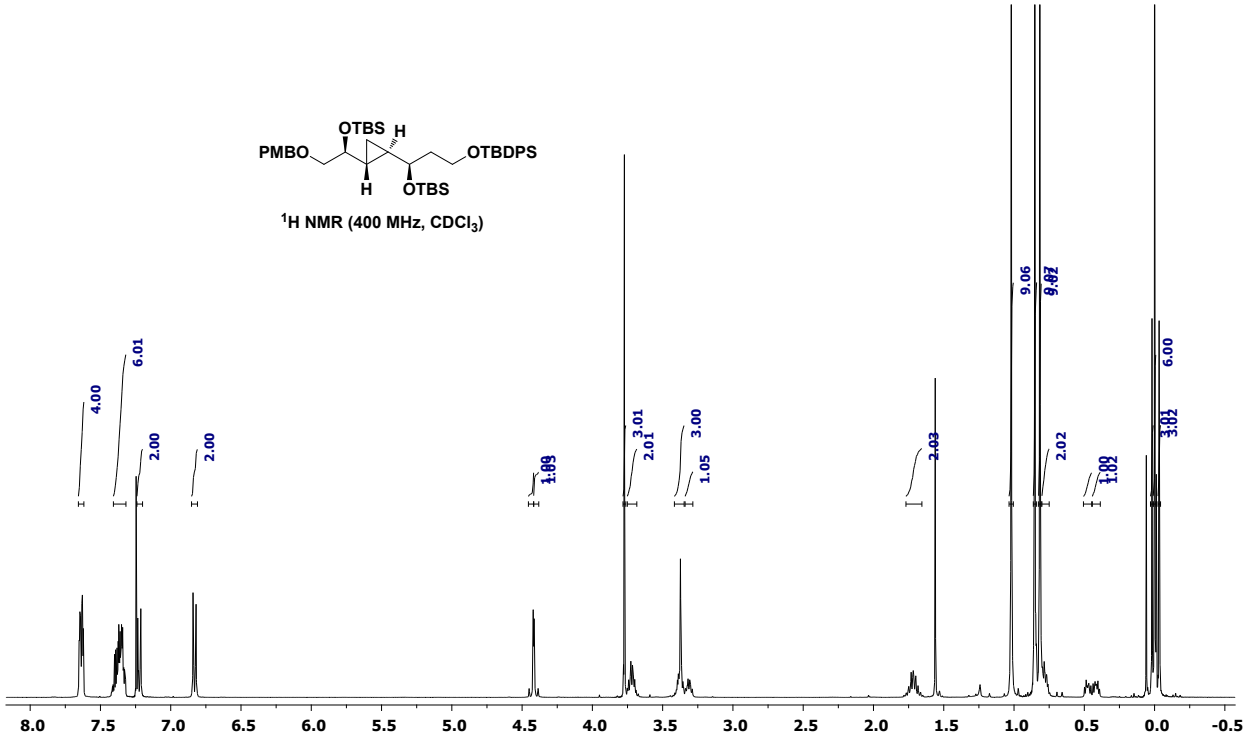


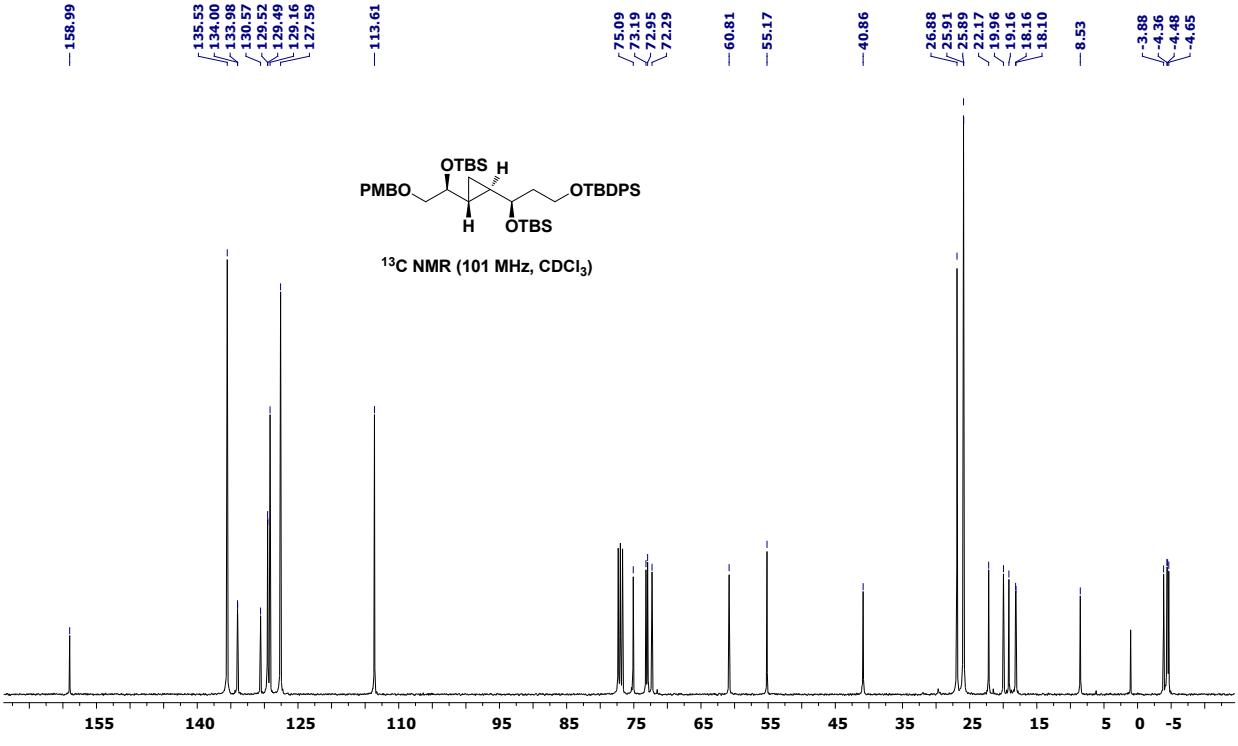
Compound 20:


OH
H
PMBO
OTBDPS
H
OTBS
^{1}H NMR (400 MHz, CDCl$_3$)
3.98
6.02
2.66
1.94
2.00
3.00
2.01
0.97
2.01
1.02
0.97
2.02
9.01
9.01
2.02
2.00
3.01
3.02
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

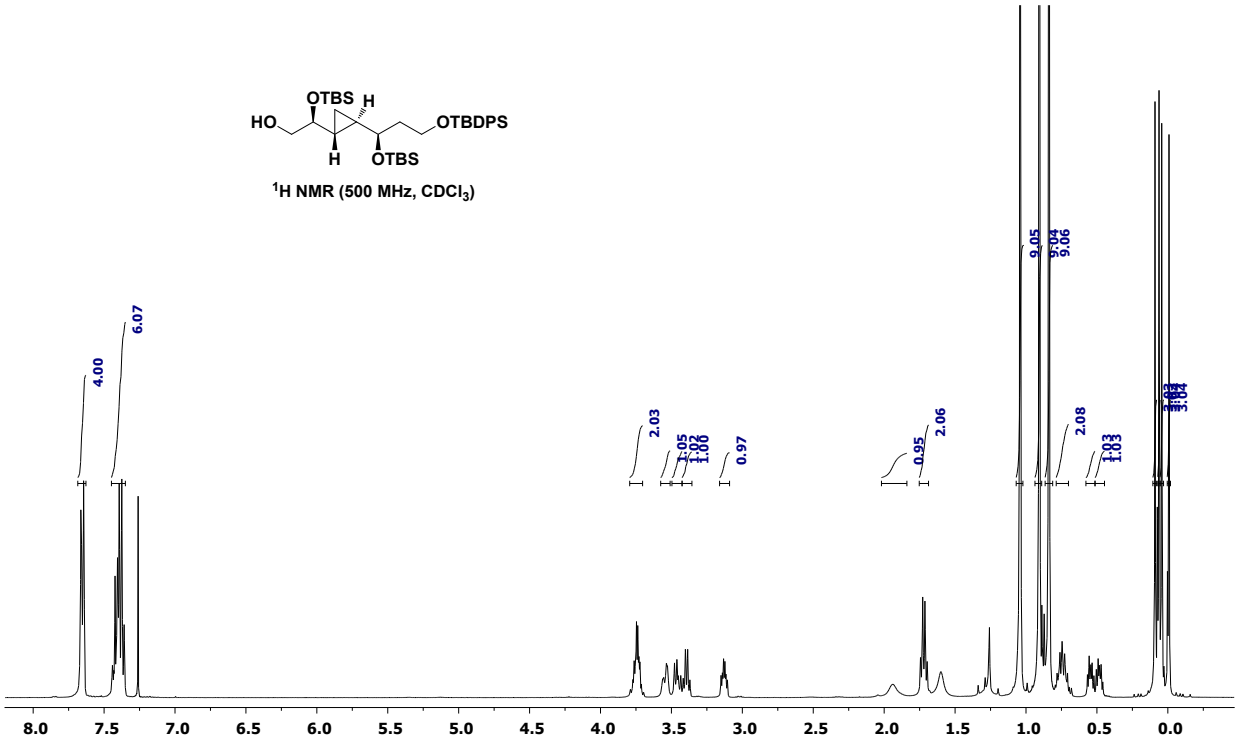

159.23
135.50
133.92
133.87
129.92
129.56
129.52
129.38
127.58
113.79
73.92
73.78
73.01
71.41
60.65
55.21
40.69
26.84
25.85
22.37
19.14
18.42
18.04
8.70
-3.99
-4.75
OH
H
PMBO
OTBDPS
H
OTBS
^{13}C NMR (101 MHz, CDCl$_3$)
155
140
125
110
95
85
75
65
55
45
35
25
15
5
0
-5

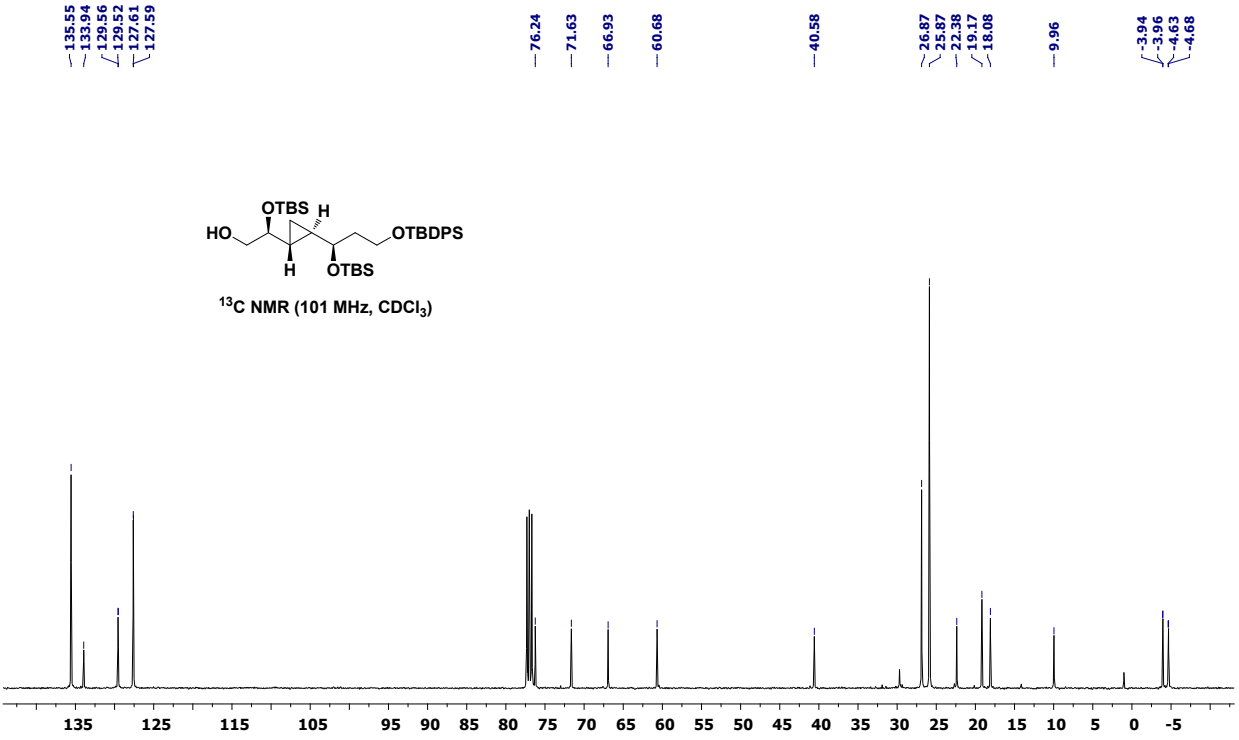
Compound 21:


OTBS H
PMBO
OTBDPS
H
OTBS
^{1}H NMR (400 MHz, CDCl$_3$)
4.00
6.01
2.00
2.00
1.09
1.09
3.01
2.01
3.00
1.05
2.03
9.06
9.07
9.07
2.02
1.09
1.02
6.00
3.01
3.02
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

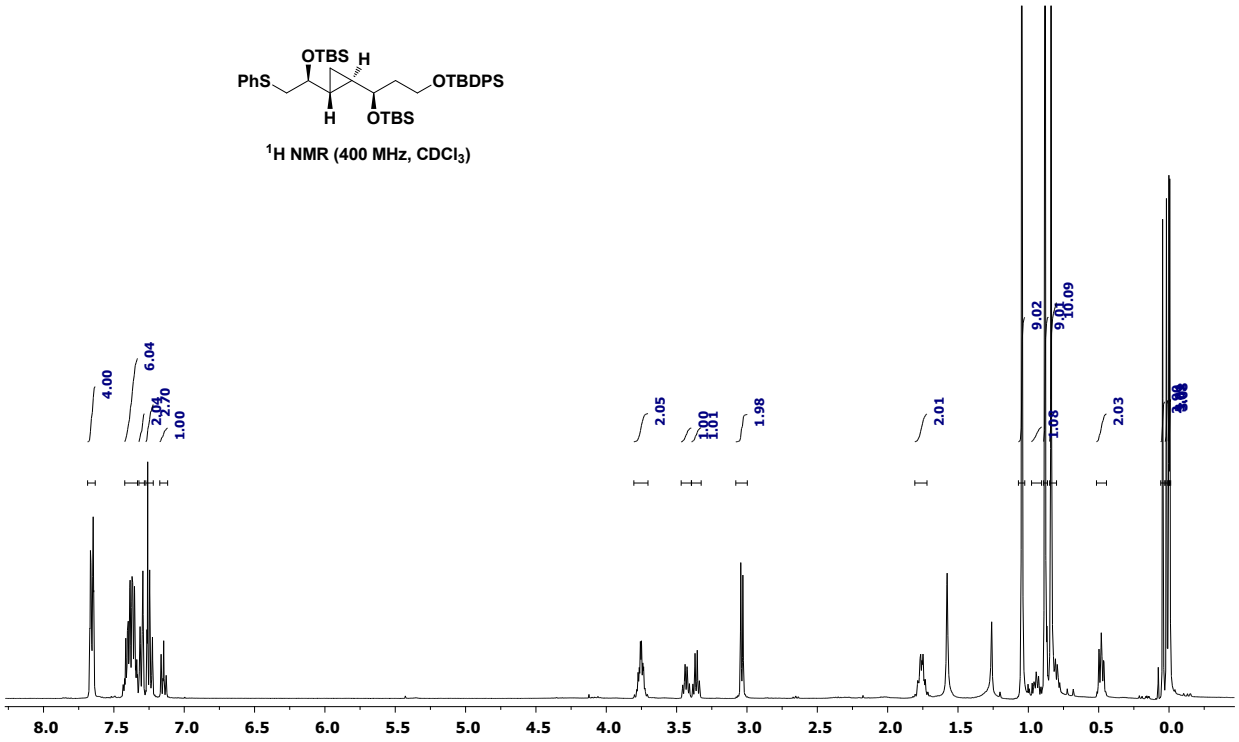

158.99
135.53
134.00
133.98
130.57
129.52
129.49
129.16
127.59
113.61
75.09
73.19
72.95
72.29
60.81
55.17
40.86
26.88
25.91
25.89
22.17
19.96
19.16
18.16
18.10
8.53
-3.88
-4.36
-4.48
-4.65
OTBS H
PMBO
OTBDPS
H
OTBS
^{13}C NMR (101 MHz, CDCl$_3$)
155
140
125
110
95
85
75
65
55
45
35
25
15
5
0
-5

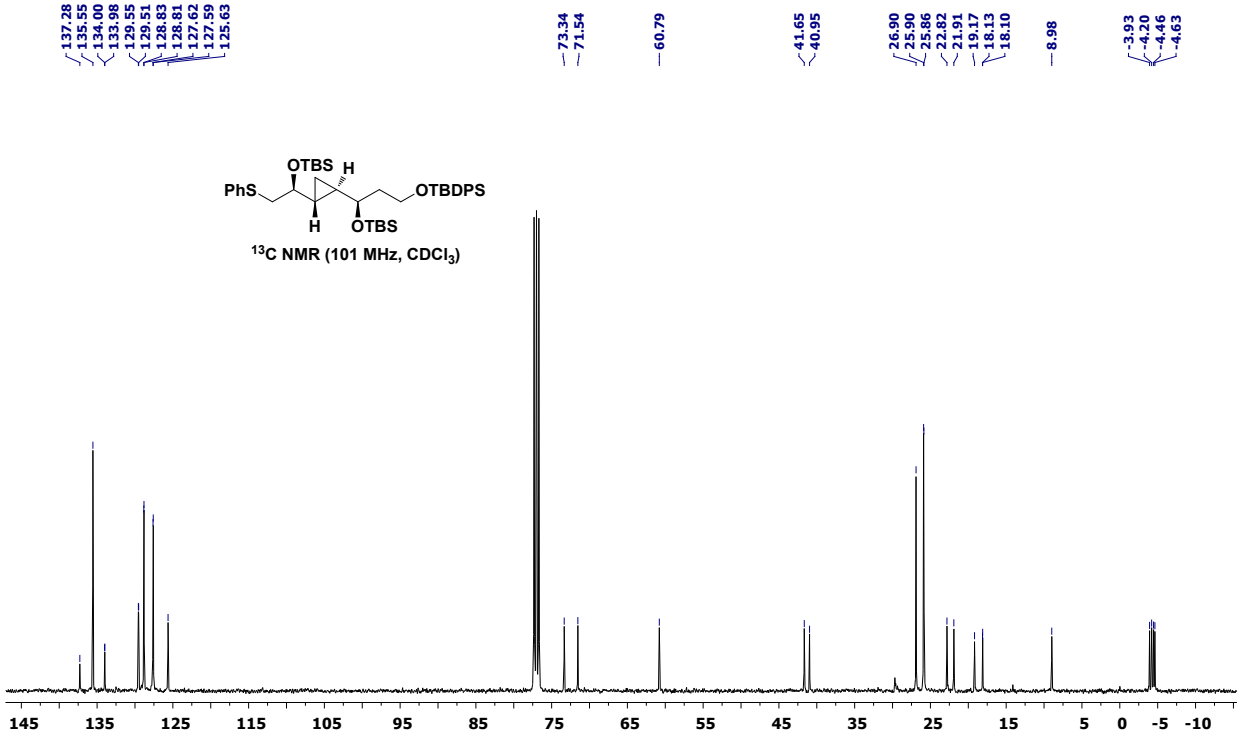
Compound 22:


OTBS
H
HO
OTBDPS
H
OTBS
^{1}H NMR (500 MHz, CDCl$_3$)
4.00
6.07
2.03
2.05
1.02
1.00
0.97
0.95
2.06
9.05
9.04
9.06
2.08
1.03
1.03
3.02
3.03
3.04
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

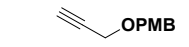

OTBS
H
HO
OTBDPS
H
OTBS
^{13}C NMR (101 MHz, CDCl$_3$)
135.55
133.94
129.56
129.52
127.61
127.59
76.24
71.63
66.93
60.68
40.58
26.87
25.87
22.38
19.17
18.08
9.96
-3.94
-3.96
-4.63
-4.68
135
125
115
105
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0
-5

Compound 23:

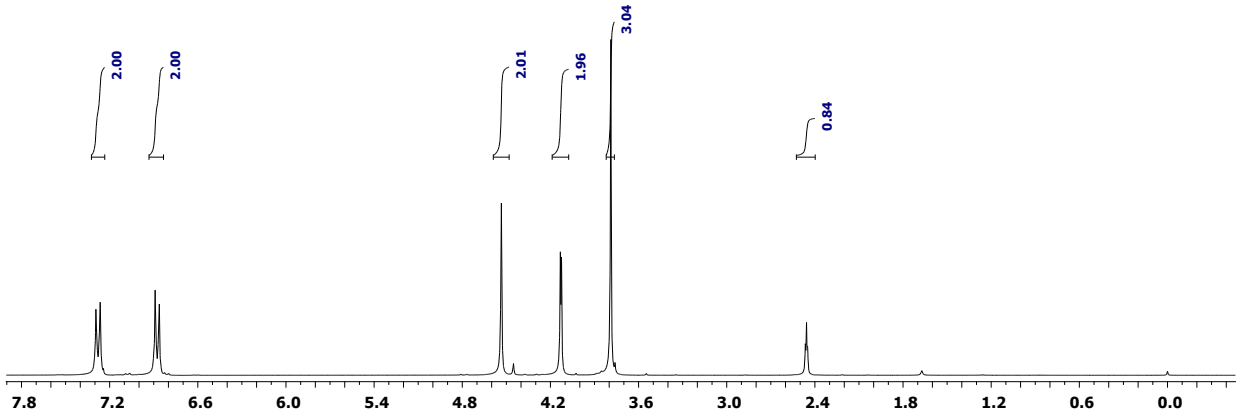

OTBS H
PhS
OTBDPS
H OTBS
^{1}H NMR (400 MHz, CDCl$_3$)
4.00
6.04
2.04
2.00
1.00
2.05
2.00
2.01
1.98
2.01
9.02
9.01
10.09
1.08
2.03
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0


137.28
135.55
134.00
133.98
129.55
129.51
128.83
128.81
127.62
127.59
125.63
73.34
71.54
60.79
41.65
40.95
26.90
25.90
25.86
22.82
21.91
19.17
18.13
18.10
8.98
-3.93
-4.20
-4.46
-4.63
OTBS H
PhS
OTBDPS
H OTBS
^{13}C NMR (101 MHz, CDCl$_3$)
145 135 125 115 105 95 85 75 65 55 45 35 25 15 5 0 -5 -10

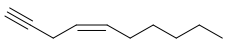
Compound 16:



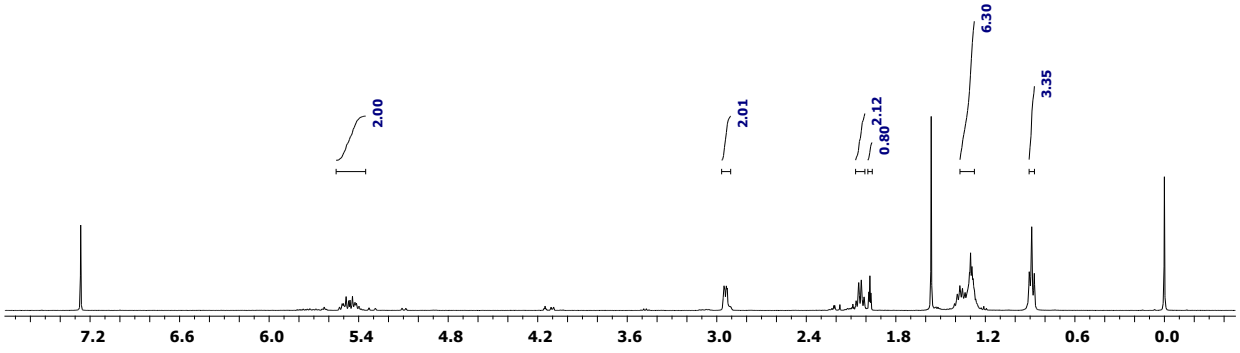
^{1}H NMR (300 MHz, $CDCl_3$)



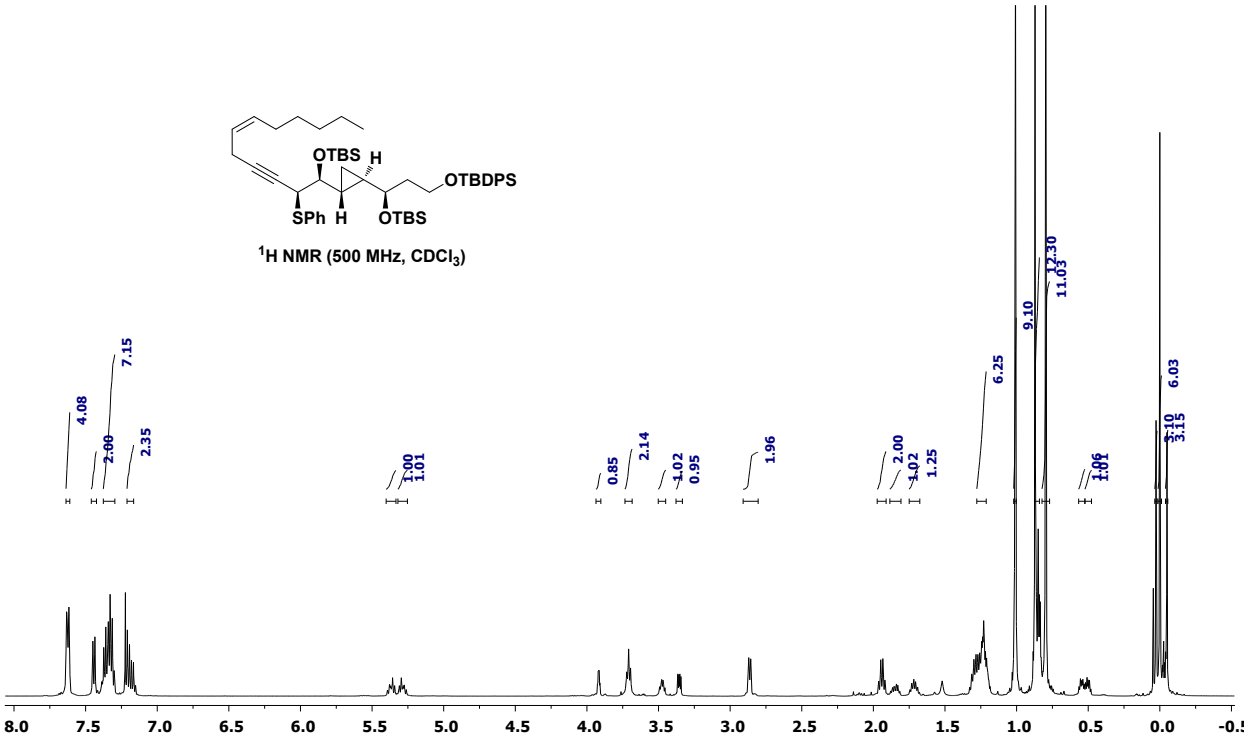
Compound 24:

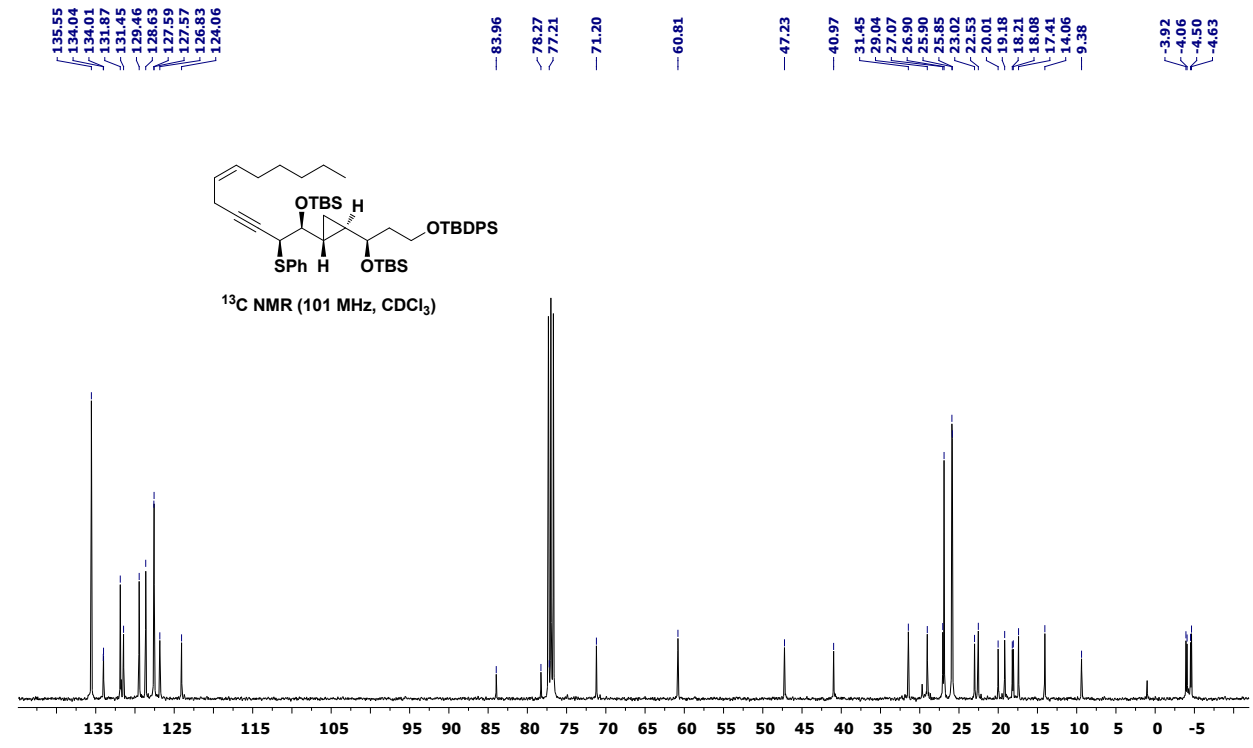


^{1}H NMR (400 MHz, $CDCl_3$)

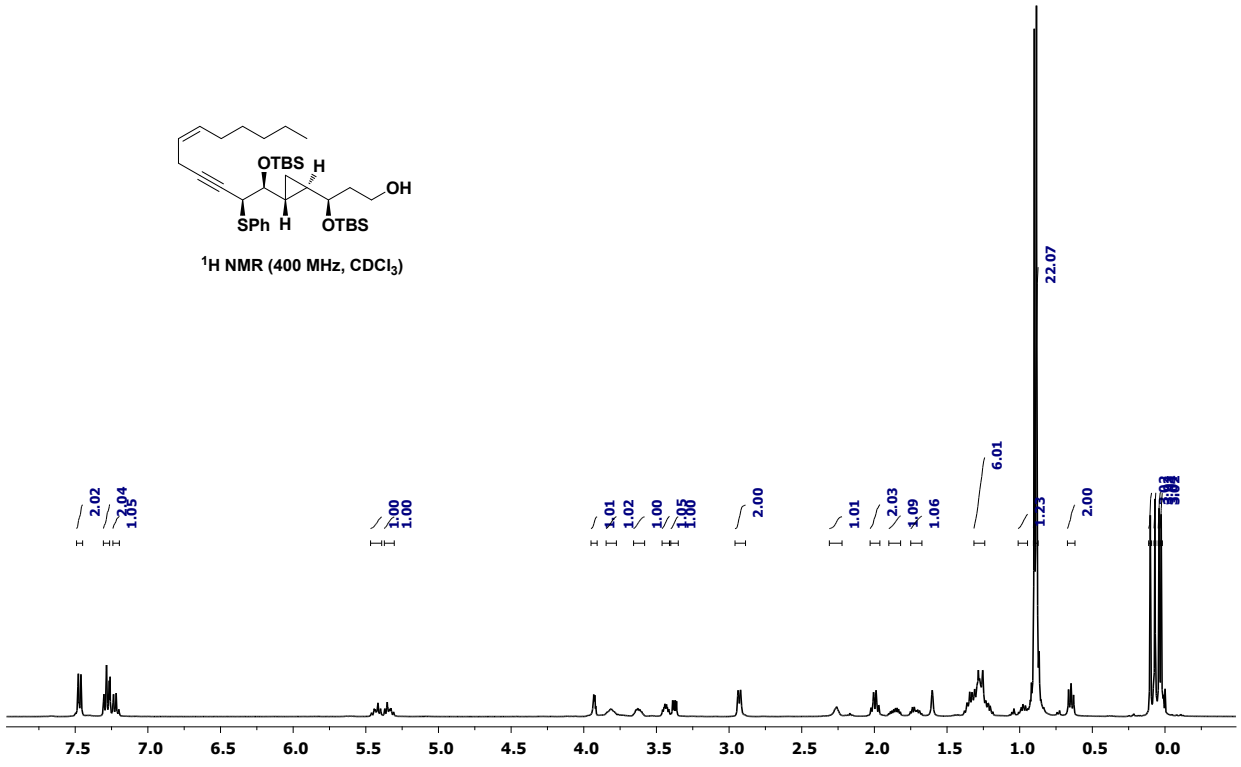


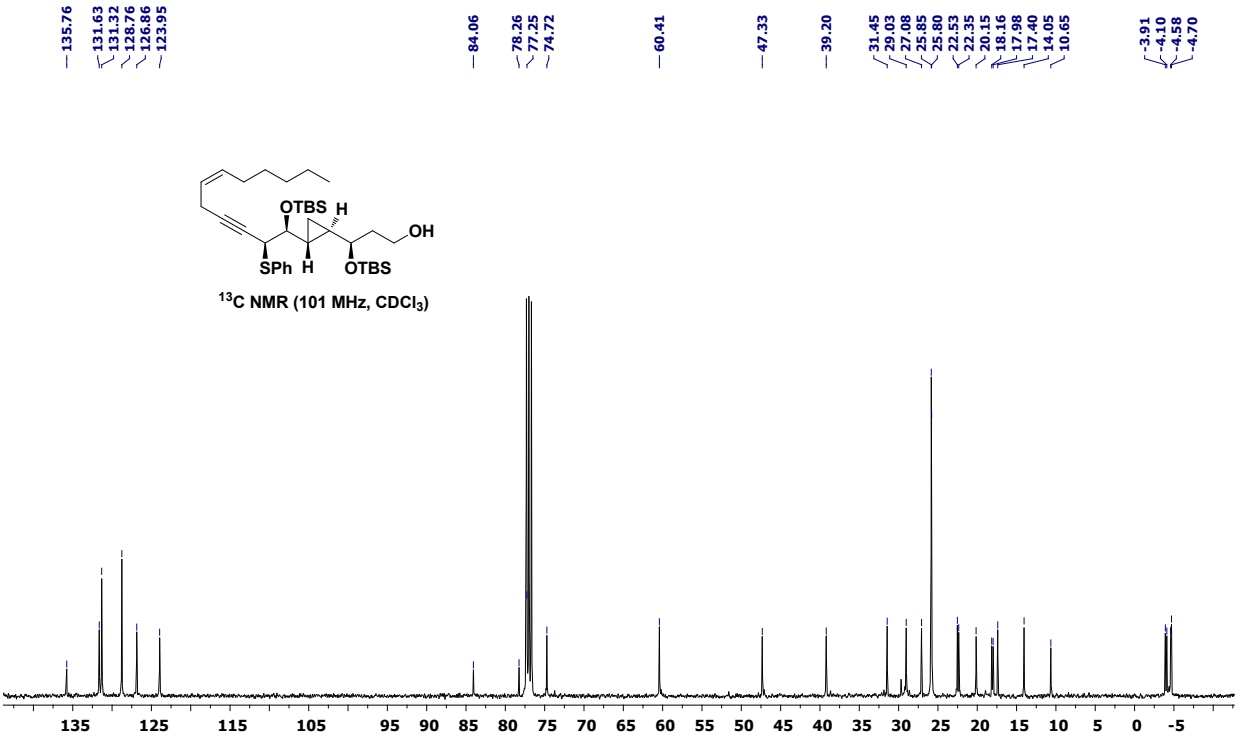
Compound 25:


OTBS H
OTBDPS
SPh H OTBS
^{1}H NMR (500 MHz, CDCl$_3$)
4.08
2.00
7.15
2.35
1.00
1.01
0.85
2.14
1.02
0.95
1.96
2.00
1.02
1.25
6.25
9.10
12.30
11.03
1.06
1.01
6.03
3.10
3.15
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

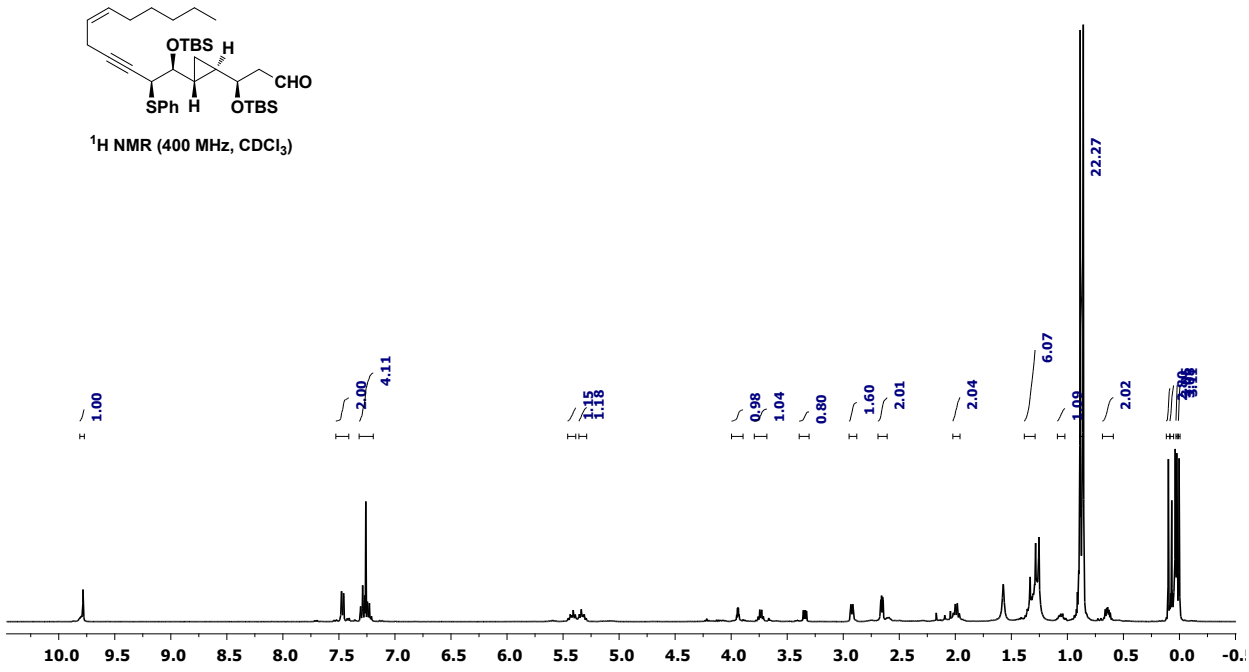

135.55
134.04
134.01
131.87
131.45
129.46
128.63
127.59
127.57
126.83
124.06
83.96
78.27
77.21
71.20
60.81
47.23
40.97
31.45
29.04
27.07
26.90
25.90
25.85
23.02
22.53
20.01
19.18
18.21
18.08
17.41
14.06
9.38
-3.92
-4.06
-4.50
-4.63
OTBS H
OTBDPS
SPh H OTBS
^{13}C NMR (101 MHz, CDCl$_3$)
135 125 115 105 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0 -5

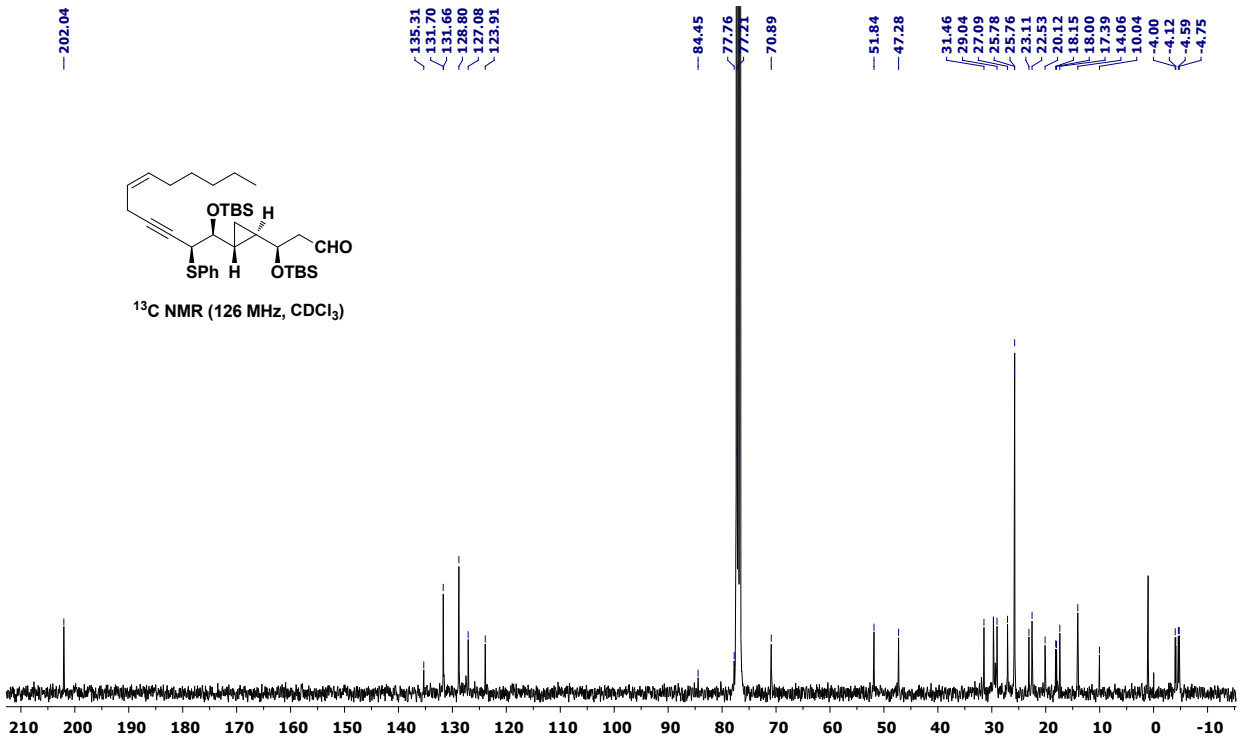
Compound 26:


OTBS H
OH
SPh H OTBS
^{1}H NMR (400 MHz, CDCl$_3$)
2.02
2.04
1.05
1.00
1.00
1.01
1.02
1.00
1.05
1.00
2.00
1.01
2.03
1.09
1.06
6.01
22.07
1.23
2.00
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

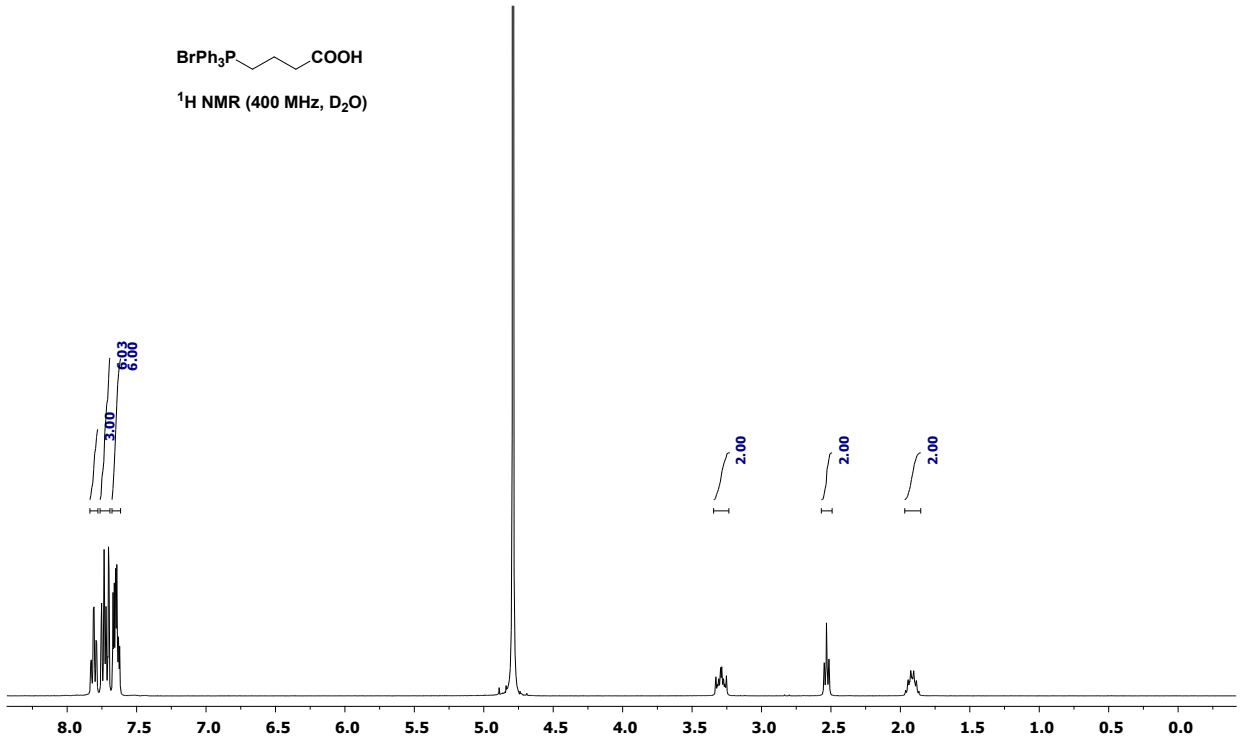

135.76
131.63
131.32
128.76
126.86
123.95
84.06
78.26
77.25
74.72
60.41
47.33
39.20
31.45
29.03
27.08
25.85
25.80
22.53
22.35
20.15
18.16
17.98
17.40
14.05
10.65
-3.91
-4.10
-4.58
-4.70
OTBS H
OH
SPh H OTBS
^{13}C NMR (101 MHz, CDCl$_3$)
135
125
115
105
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0
-5

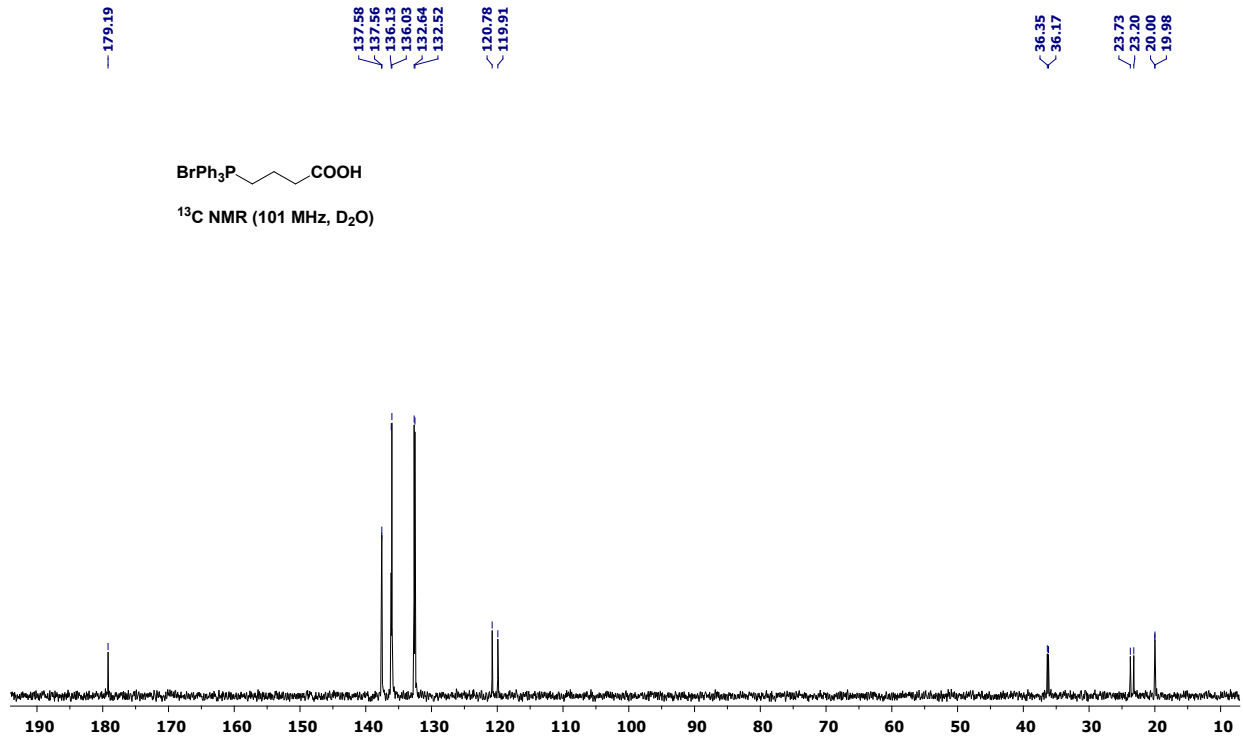
Compound 27:


OTBS H
CHO
SPh H OTBS
^{1}H NMR (400 MHz, CDCl$_3$)
1.00
2.00
4.11
1.15
1.18
0.98
1.04
0.80
1.60
2.01
2.04
6.07
1.09
22.27
2.02
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

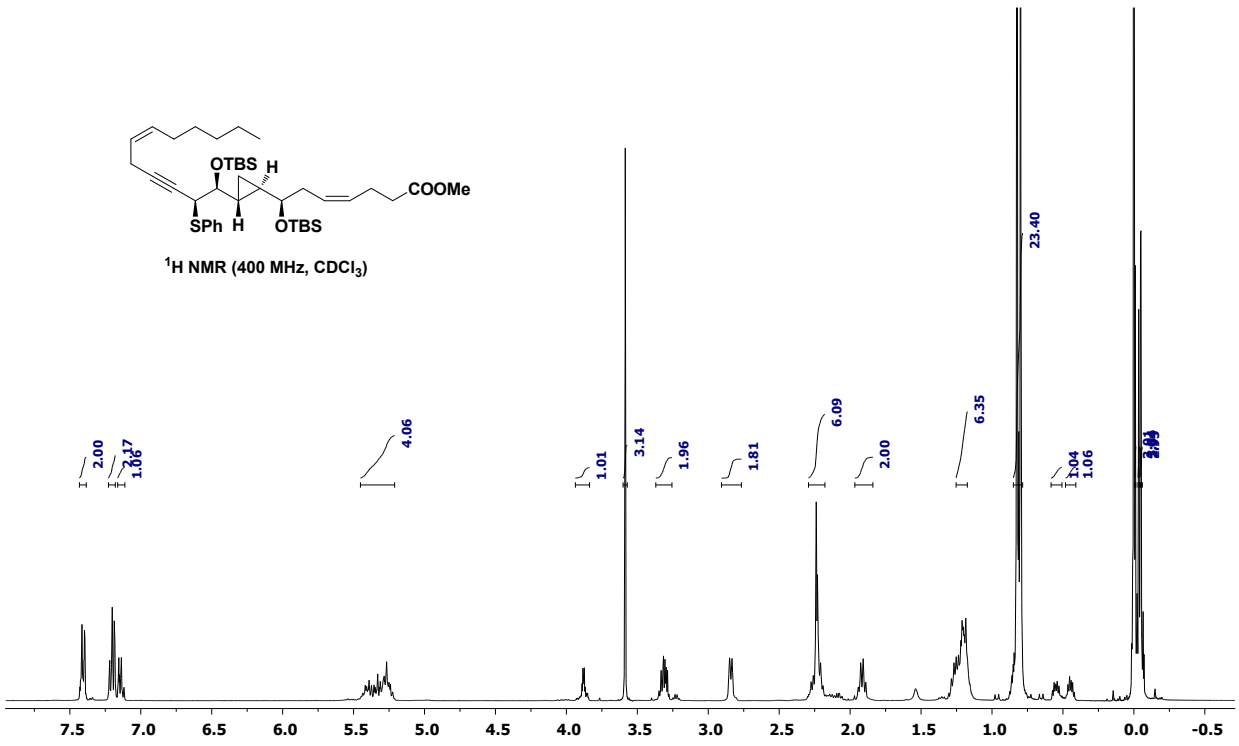

202.04
135.31
131.70
131.66
128.80
127.08
123.91
84.45
77.76
77.21
70.89
51.84
47.28
31.46
29.04
27.09
25.78
25.76
23.11
22.53
20.12
18.15
18.00
17.39
14.06
10.04
-4.00
-4.12
-4.59
-4.75
OTBS H
CHO
SPh H OTBS
^{13}C NMR (126 MHz, CDCl$_3$)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

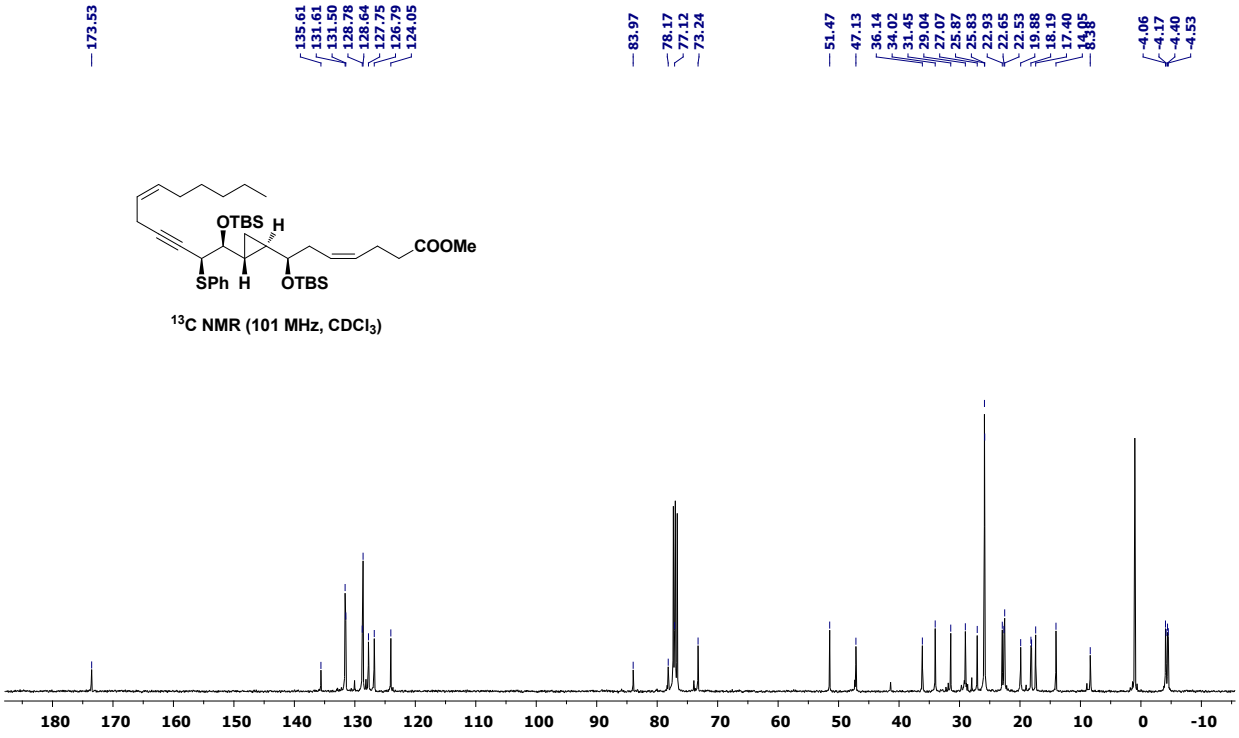
Compound 28:


BrPh3P COOH
1H NMR (400 MHz, D2O)
6.03
6.00
3.00
2.00
2.00
2.00
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

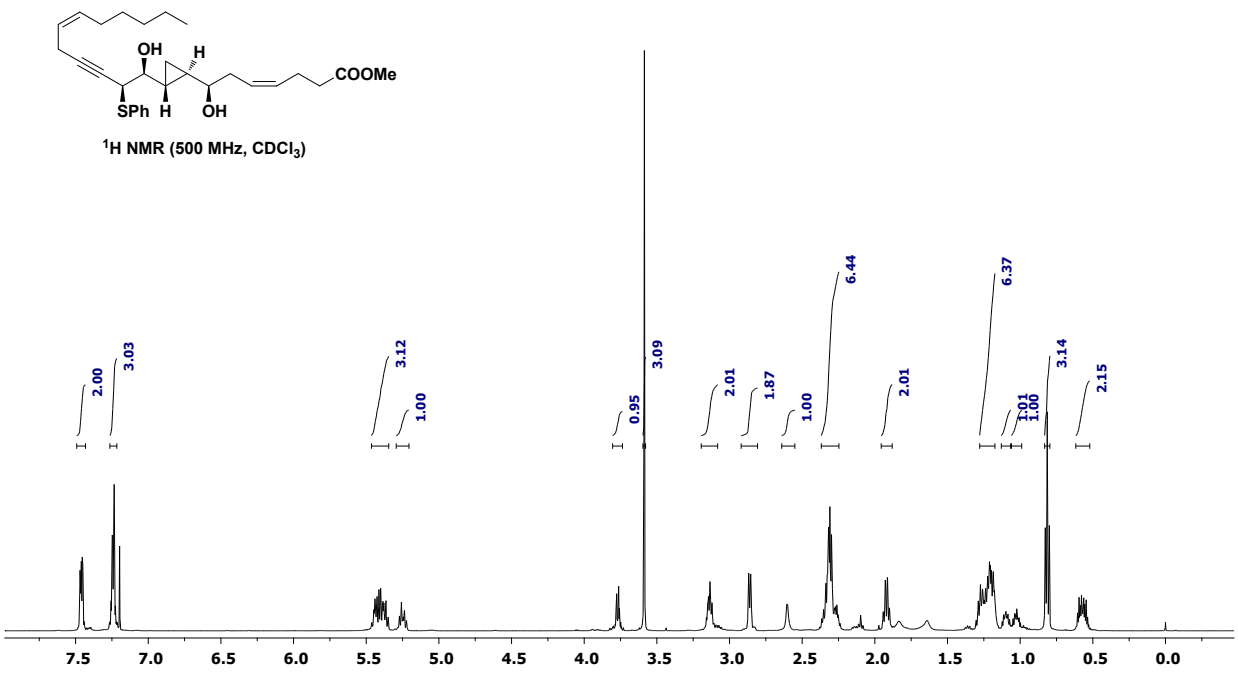

179.19
137.58
137.56
136.13
136.03
132.64
132.52
120.78
119.91
36.35
36.17
23.73
23.20
20.00
19.98
BrPh3P COOH
13C NMR (101 MHz, D2O)
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10

Compound 29:

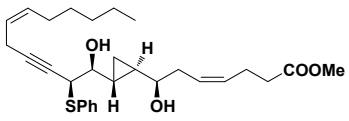

OTBS H
COOMe
SPh H OTBS
^{1}H NMR (400 MHz, CDCl$_3$)
2.00
2.17
1.06
4.06
1.01
3.14
1.96
1.81
6.09
2.00
6.35
23.40
1.04
1.06
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5


OTBS H
COOMe
SPh H OTBS
^{13}C NMR (101 MHz, CDCl$_3$)
173.53
135.61
131.61
131.50
128.78
128.64
127.75
126.79
124.05
83.97
78.17
77.12
73.24
51.47
47.13
36.14
34.02
31.45
29.04
27.07
25.87
25.83
22.93
22.65
22.53
19.88
18.19
17.40
14.05
-4.06
-4.17
-4.40
-4.53
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

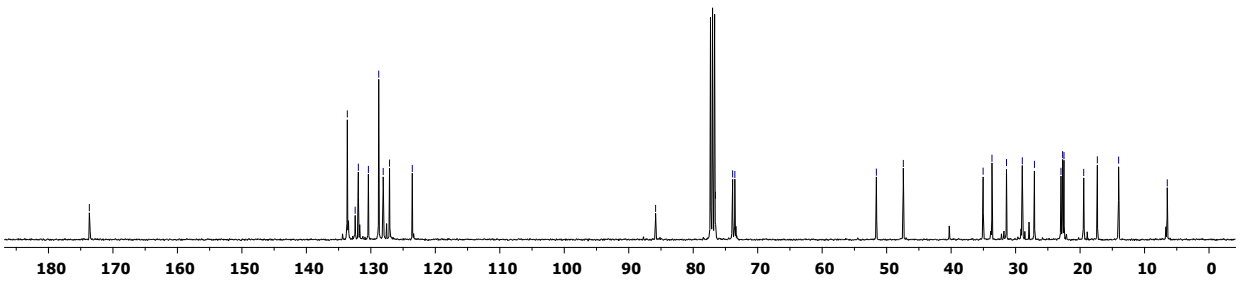
Compound 30:



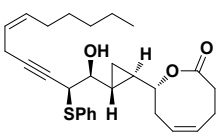
^{1}H NMR (500 MHz, $CDCl_3$)



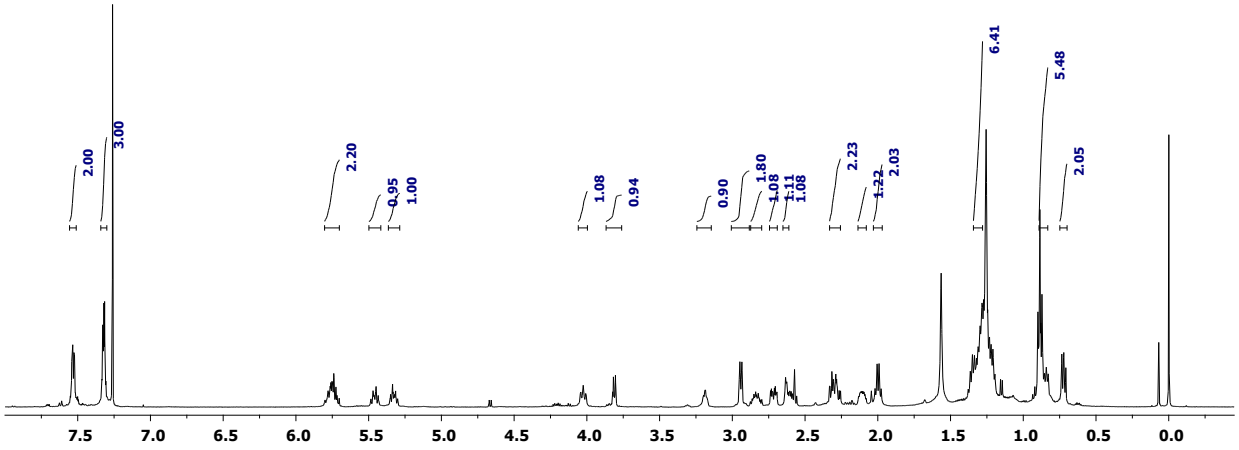
^{13}C NMR (101 MHz, $CDCl_3$)

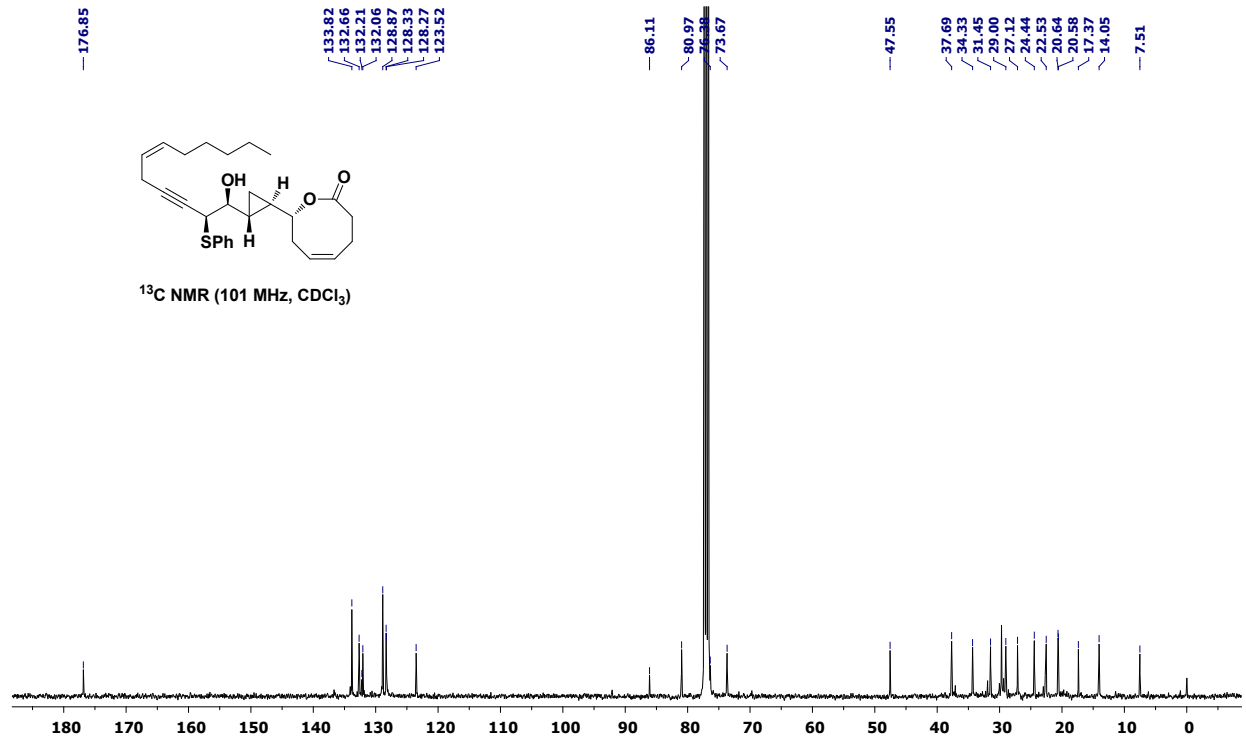


Compound 31:



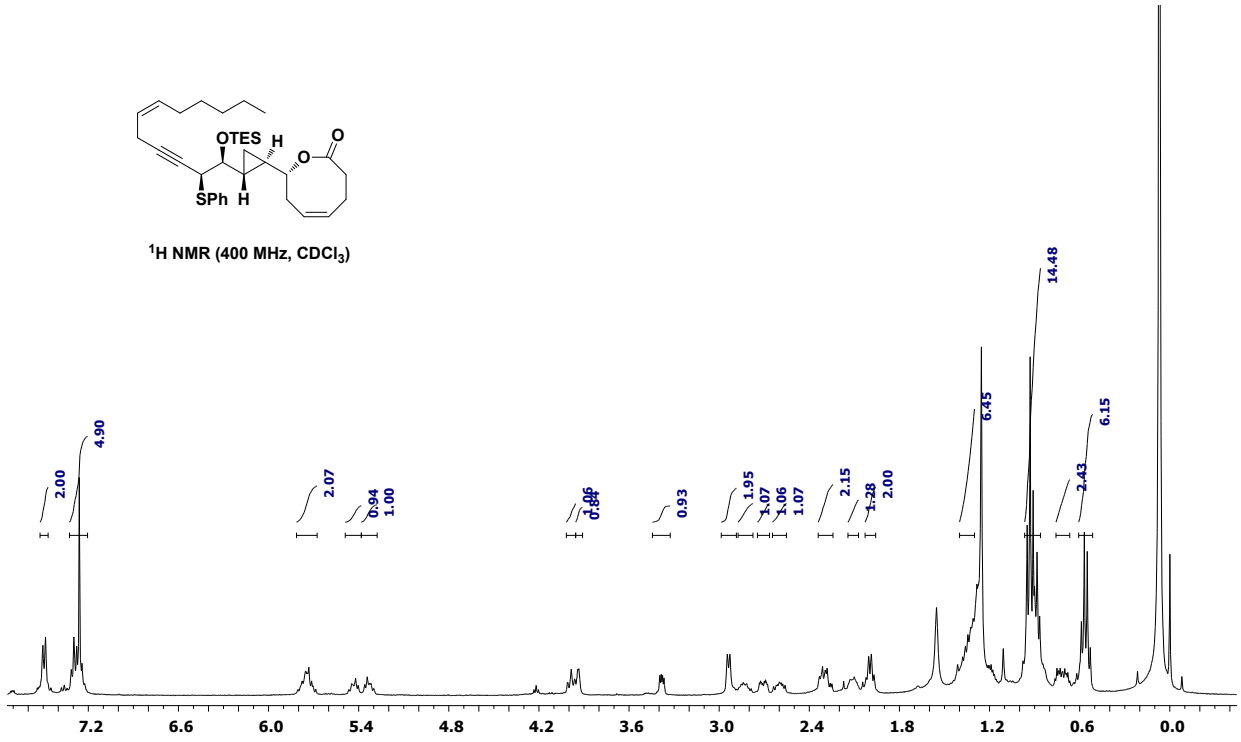
^{1}H NMR (500 MHz, $CDCl_3$)

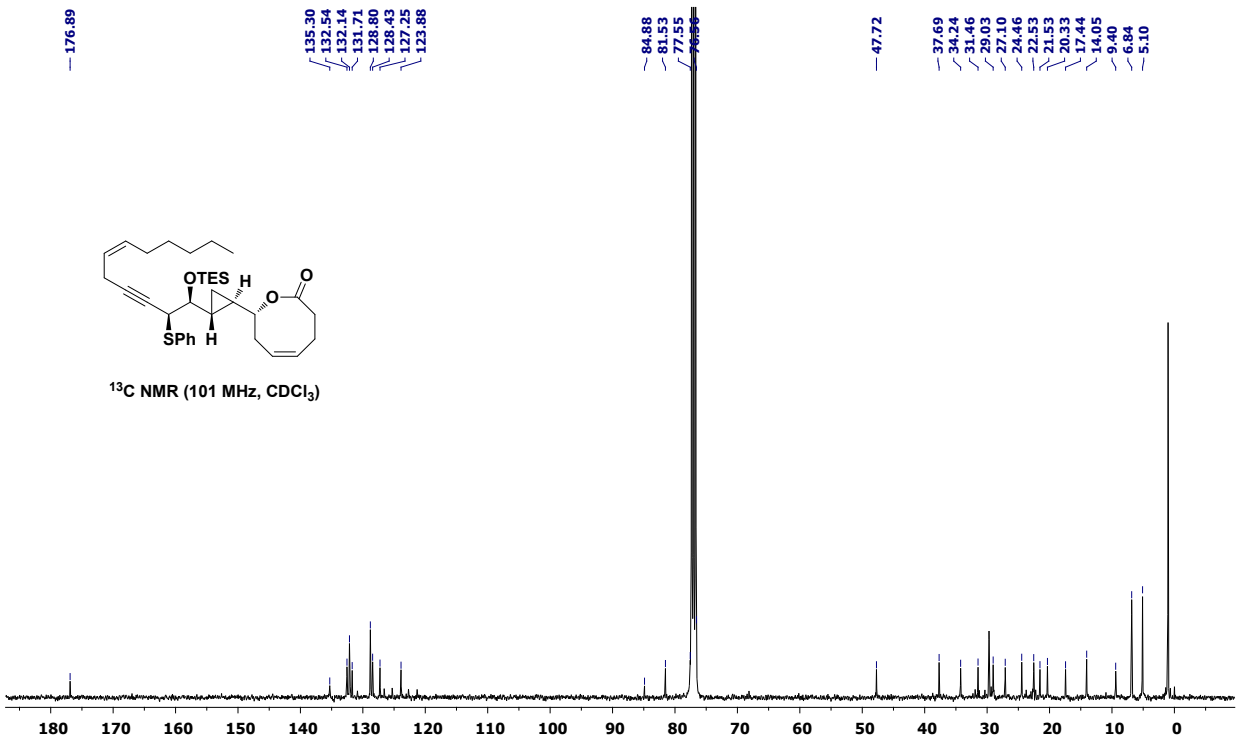




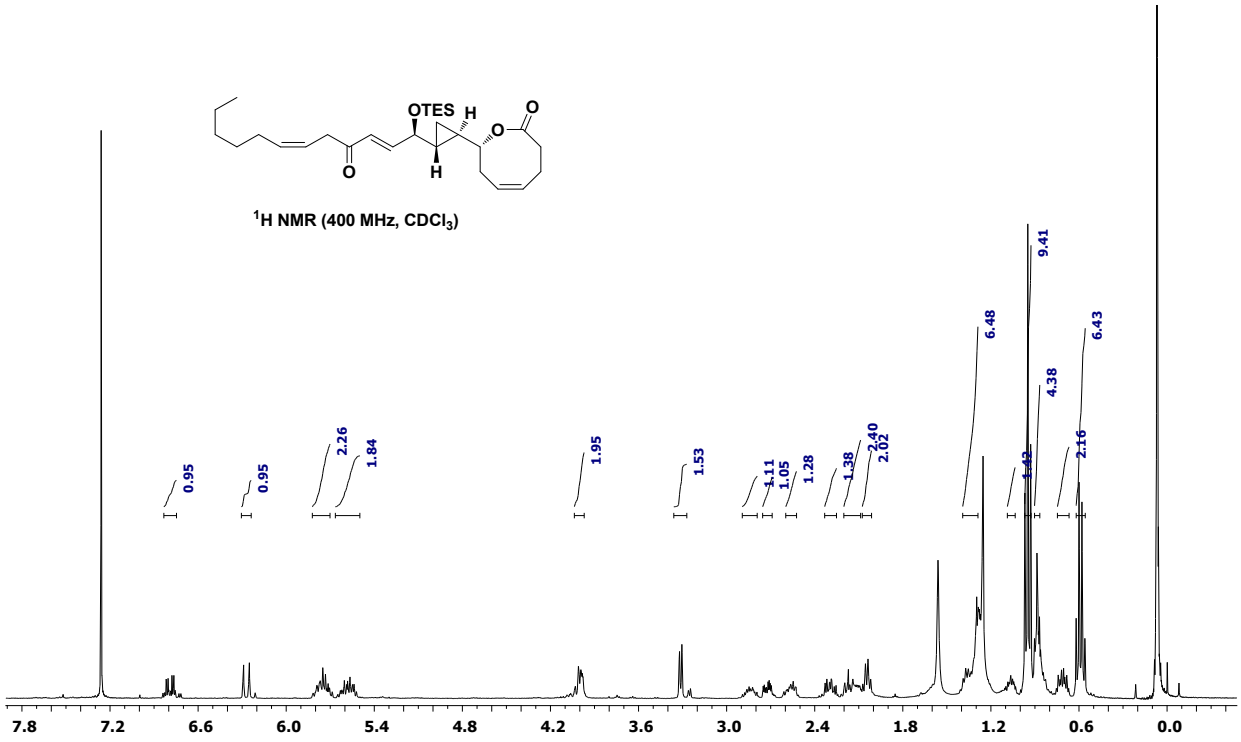
^{13}C NMR (101 MHz, $CDCl_3$)

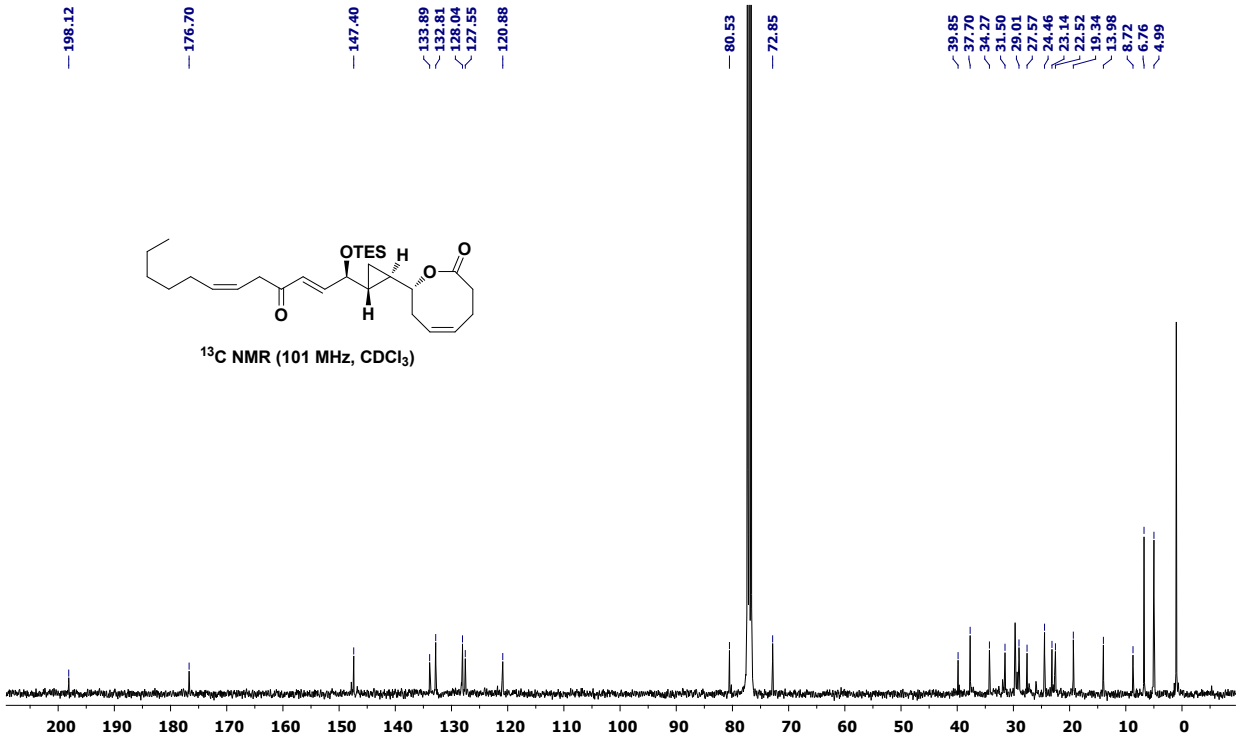
Compound 32:


OTES
H
O
O
SPh
H
^{1}H NMR (400 MHz, CDCl$_3$)
2.00
4.90
2.07
0.94
1.00
0.93
1.95
1.07
1.06
1.07
2.15
1.28
2.00
6.45
14.48
6.15
2.43
7.2
6.6
6.0
5.4
4.8
4.2
3.6
3.0
2.4
1.8
1.2
0.6
0.0

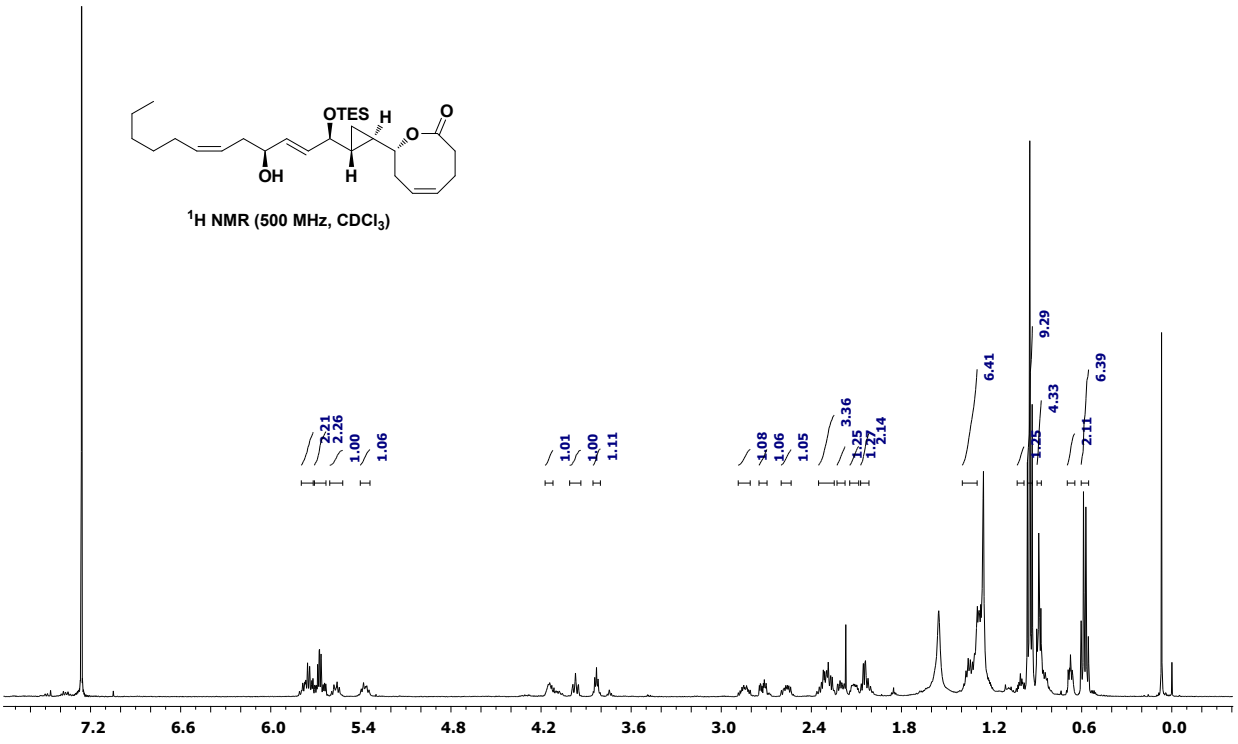

176.89
135.30
132.54
132.14
131.71
128.80
128.43
127.25
123.88
84.88
81.53
77.55
47.72
37.69
34.24
31.46
29.03
27.10
24.46
22.53
21.53
20.33
17.44
14.05
9.40
6.84
5.10
OTES
H
O
O
SPh
H
^{13}C NMR (101 MHz, CDCl$_3$)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

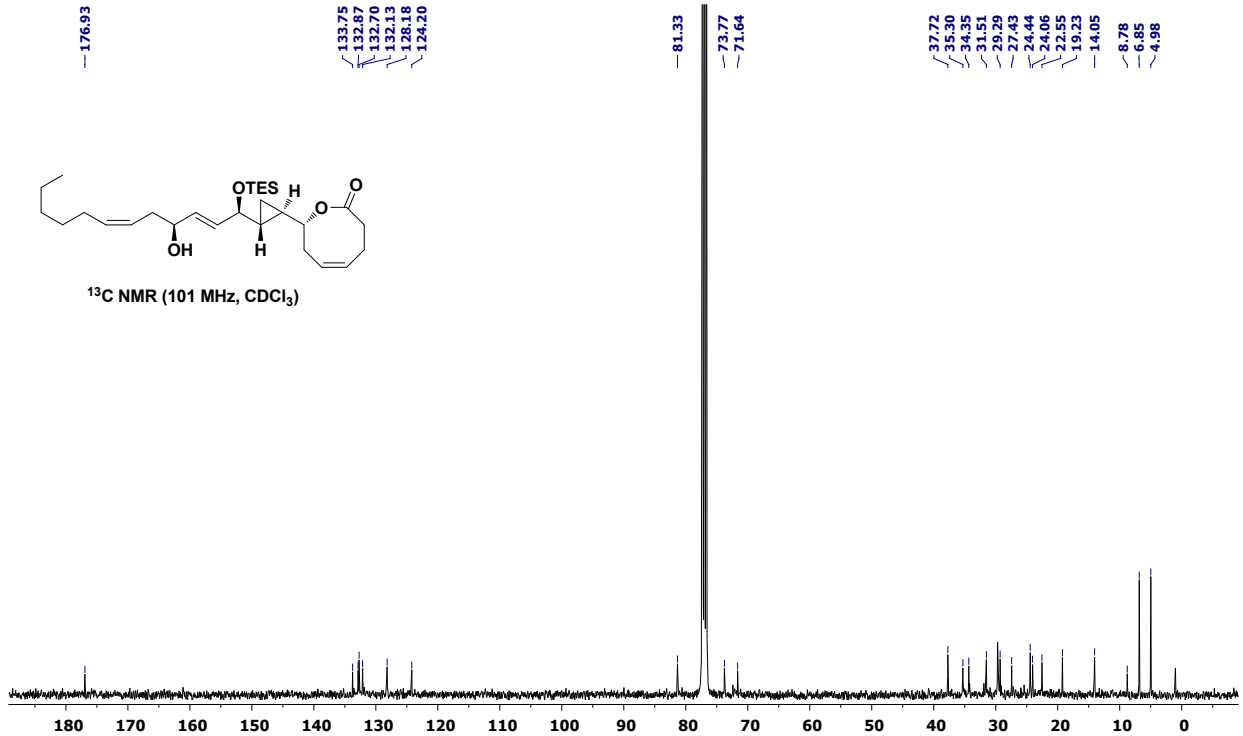
Compound 33:


OTES
H
O
O
O
H
^{1}H NMR (400 MHz, $CDCl_3$)
0.95
0.95
2.26
1.84
1.95
1.53
1.11
1.05
1.28
1.38
2.40
2.02
6.48
9.41
1.42
4.38
6.43
2.16
7.8
7.2
6.6
6.0
5.4
4.8
4.2
3.6
3.0
2.4
1.8
1.2
0.6
0.0

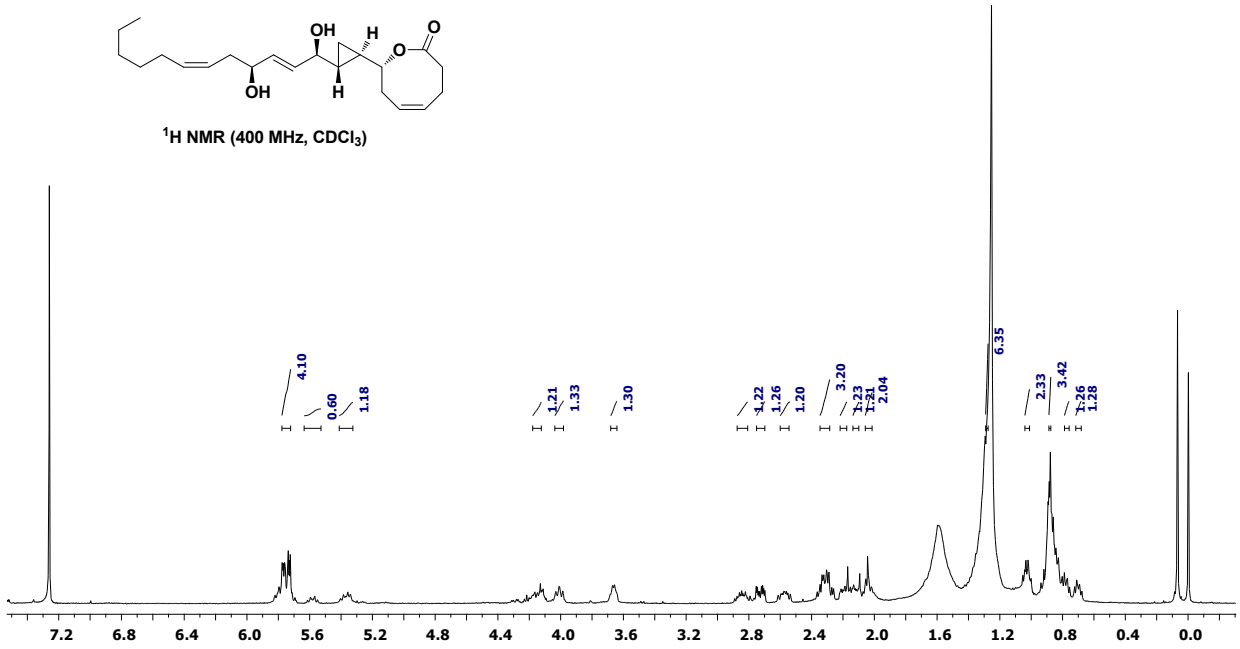

198.12
176.70
147.40
133.89
132.81
128.04
127.55
120.88
80.53
72.85
39.85
37.70
34.27
31.50
29.01
27.57
24.46
23.14
22.52
19.34
13.98
8.72
6.76
4.99
OTES
H
O
O
O
H
^{13}C NMR (101 MHz, $CDCl_3$)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

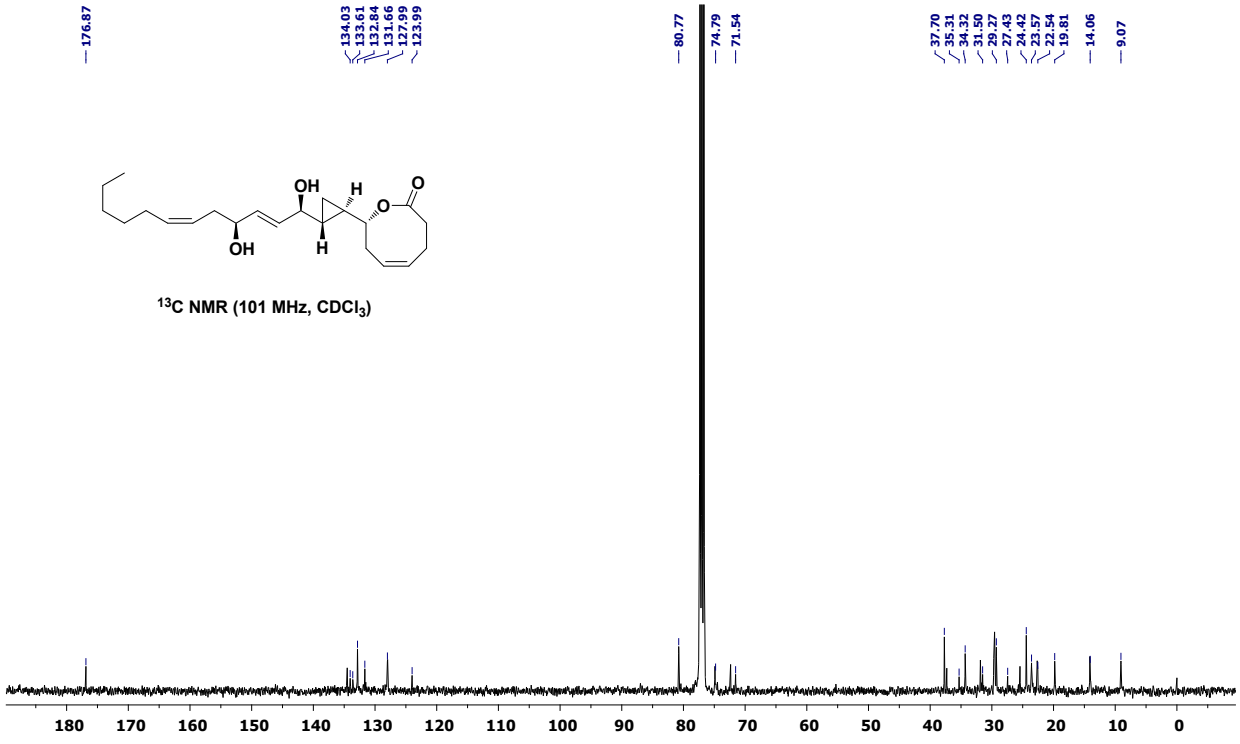
Compound 34:


OTES
H
O
O
OH
H
^{1}H NMR (500 MHz, CDCl$_3$)
2.21
2.26
1.00
1.06
1.01
1.00
1.11
1.08
1.06
1.05
3.36
3.25
1.27
2.14
6.41
9.29
4.33
1.25
2.11
6.39
7.2
6.6
6.0
5.4
4.8
4.2
3.6
3.0
2.4
1.8
1.2
0.6
0.0

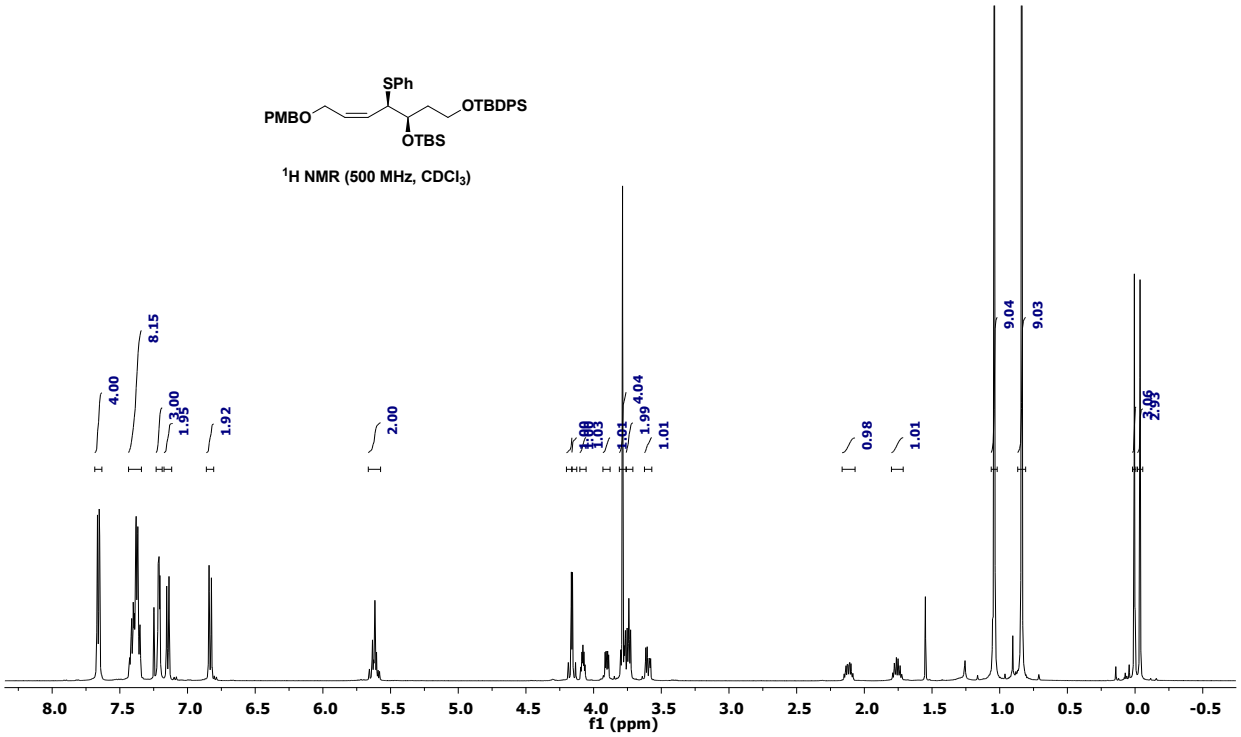

176.93
133.75
132.87
132.70
132.13
128.18
124.20
81.33
73.77
71.64
37.72
35.30
34.35
31.51
29.29
27.43
24.44
24.06
22.55
19.23
14.05
8.78
6.85
4.98
OTES
H
O
O
OH
H
^{13}C NMR (101 MHz, CDCl$_3$)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

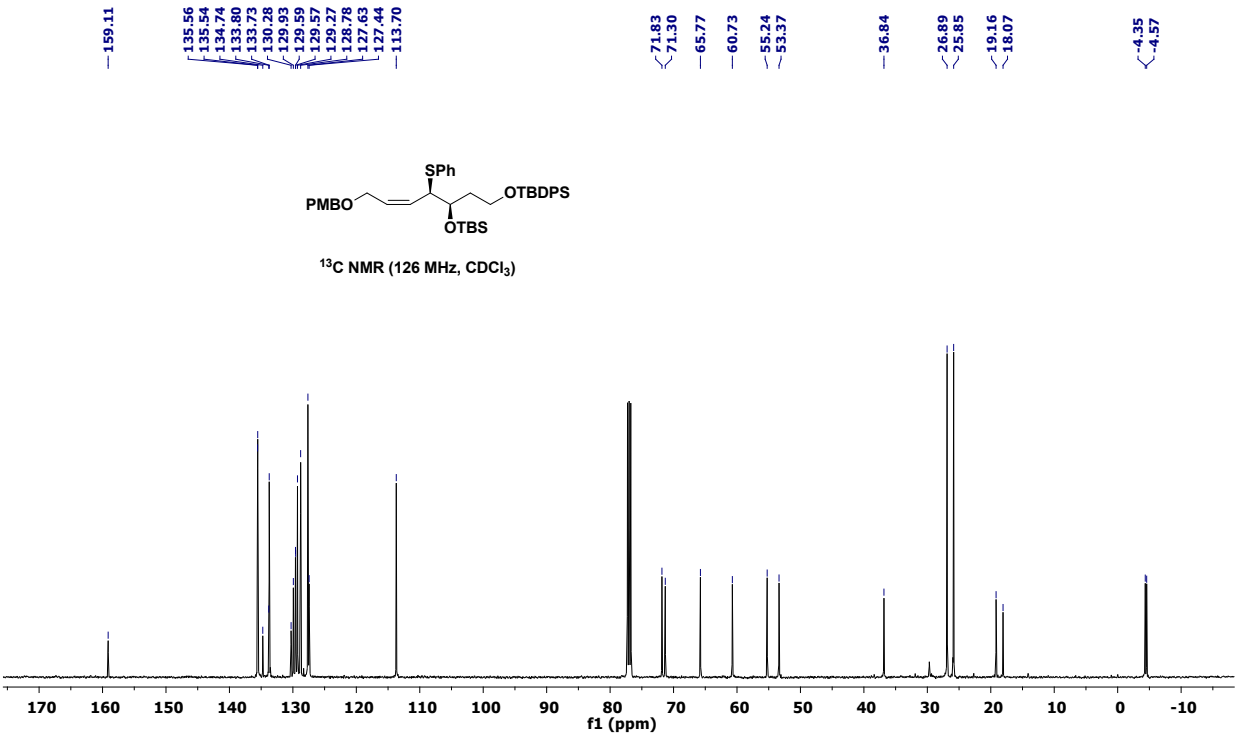
Compound 2:


OH
H
O
O
OH
H
^{1}H NMR (400 MHz, CDCl$_3$)
4.10
0.60
1.18
1.21
1.33
1.30
1.22
1.26
1.20
3.20
1.23
1.31
2.04
6.35
2.33
3.42
1.26
1.28
7.2
6.8
6.4
6.0
5.6
5.2
4.8
4.4
4.0
3.6
3.2
2.8
2.4
2.0
1.6
1.2
0.8
0.4
0.0

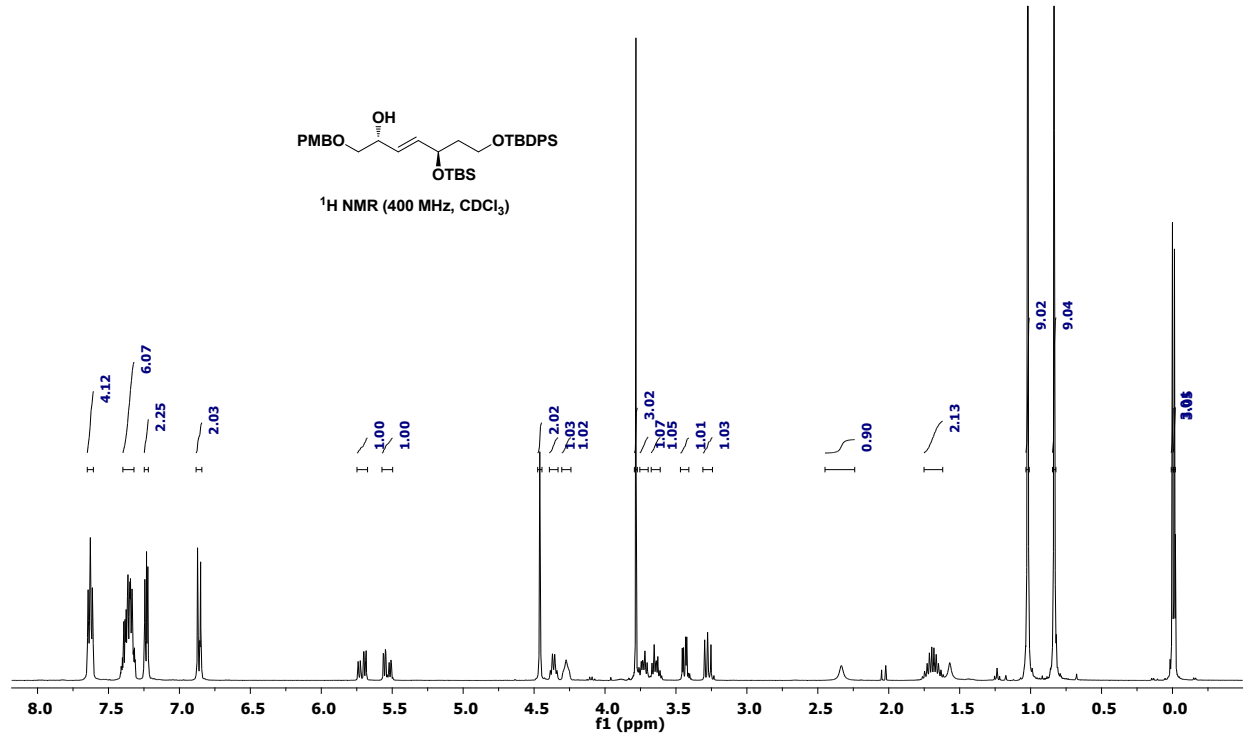

176.87
134.03
133.61
132.84
131.66
127.99
123.99
80.77
74.79
71.54
37.70
35.31
34.32
31.50
29.27
27.43
24.42
23.57
22.54
19.81
14.06
9.07
OH
H
O
O
OH
H
^{13}C NMR (101 MHz, CDCl$_3$)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

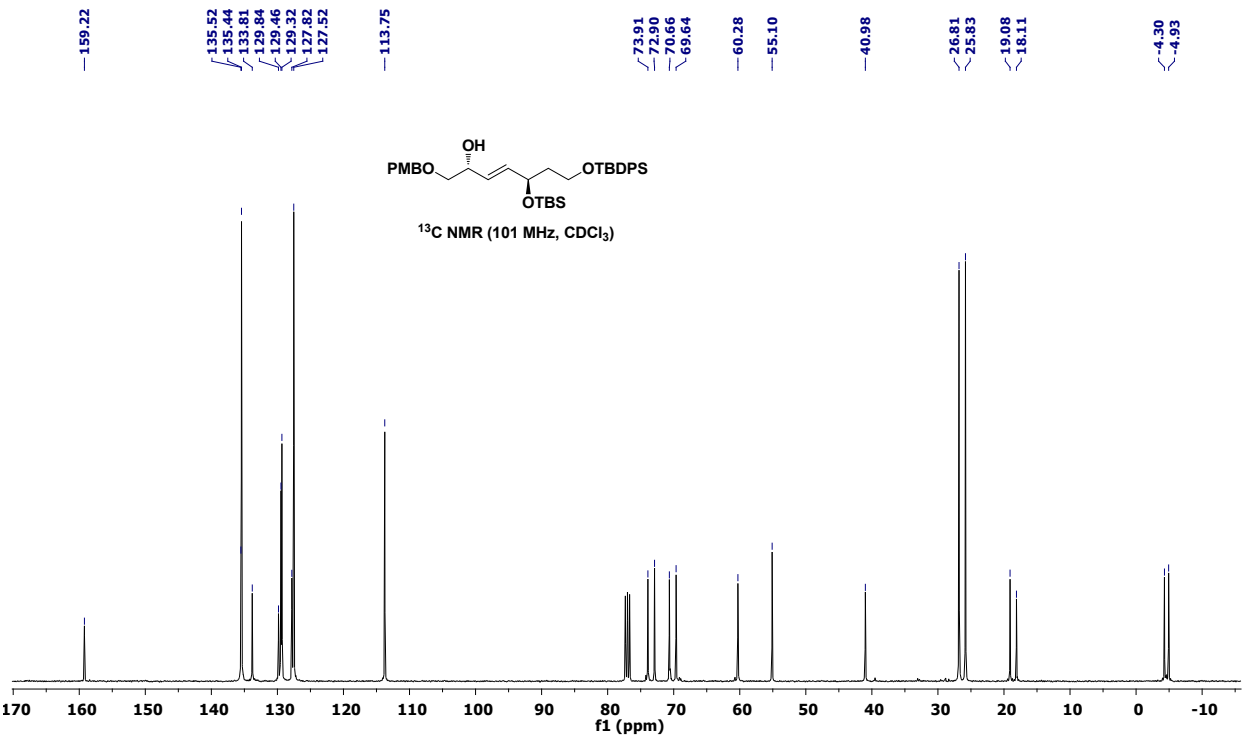
Compound 35:


SPh
PMBO
OTBDPS
OTBS
^{1}H NMR (500 MHz, CDCl$_3$)
4.00
8.15
3.00
1.95
1.92
2.00
1.01
1.99
4.04
1.01
0.98
1.01
9.04
9.03
2.06
2.05
f1 (ppm)

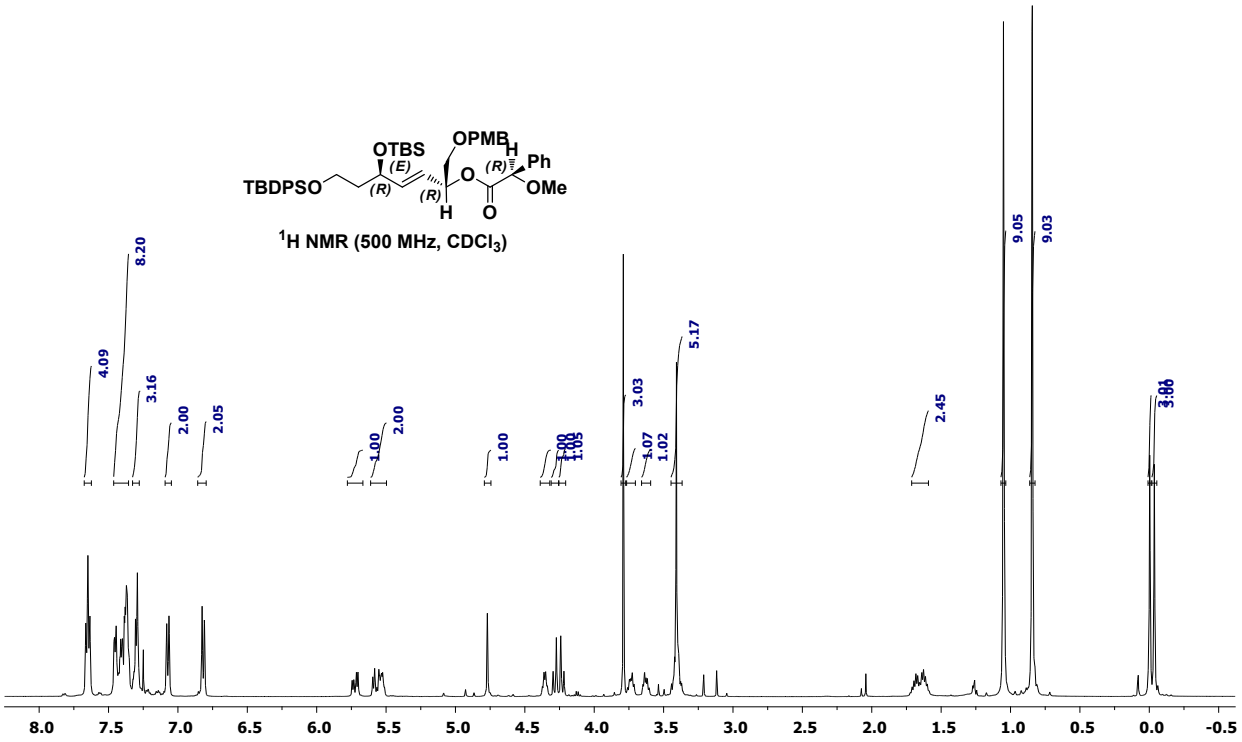

159.11
135.56
135.54
134.74
133.80
133.73
130.28
129.93
129.59
129.57
129.27
128.78
127.63
127.44
113.70
71.83
71.30
65.77
60.73
55.24
53.37
36.84
26.89
25.85
19.16
18.07
-4.35
-4.57
SPh
PMBO
OTBDPS
OTBS
^{13}C NMR (126 MHz, CDCl$_3$)
f1 (ppm)

Compound 36:

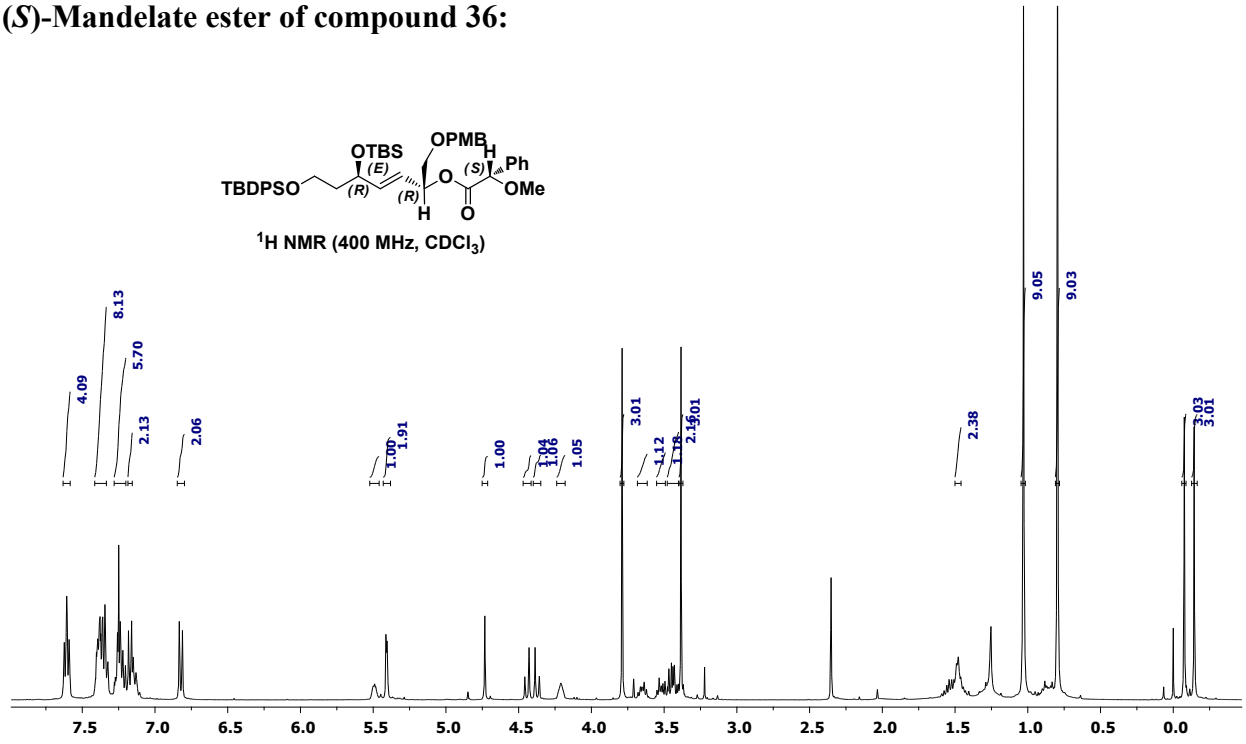

OH
PMBO
OTBDPS
OTBS
^{1}H NMR (400 MHz, CDCl$_3$)
4.12
6.07
2.25
2.03
1.00
1.00
2.02
1.03
1.02
3.02
1.07
1.05
1.01
1.03
0.90
2.13
9.02
9.04
f1 (ppm)


159.22
135.52
135.44
133.81
129.84
129.46
129.32
127.82
127.52
113.75
73.91
72.90
70.66
69.64
60.28
55.10
40.98
26.81
25.83
19.08
18.11
-4.30
-4.93
OH
PMBO
OTBDPS
OTBS
^{13}C NMR (101 MHz, CDCl$_3$)
f1 (ppm)

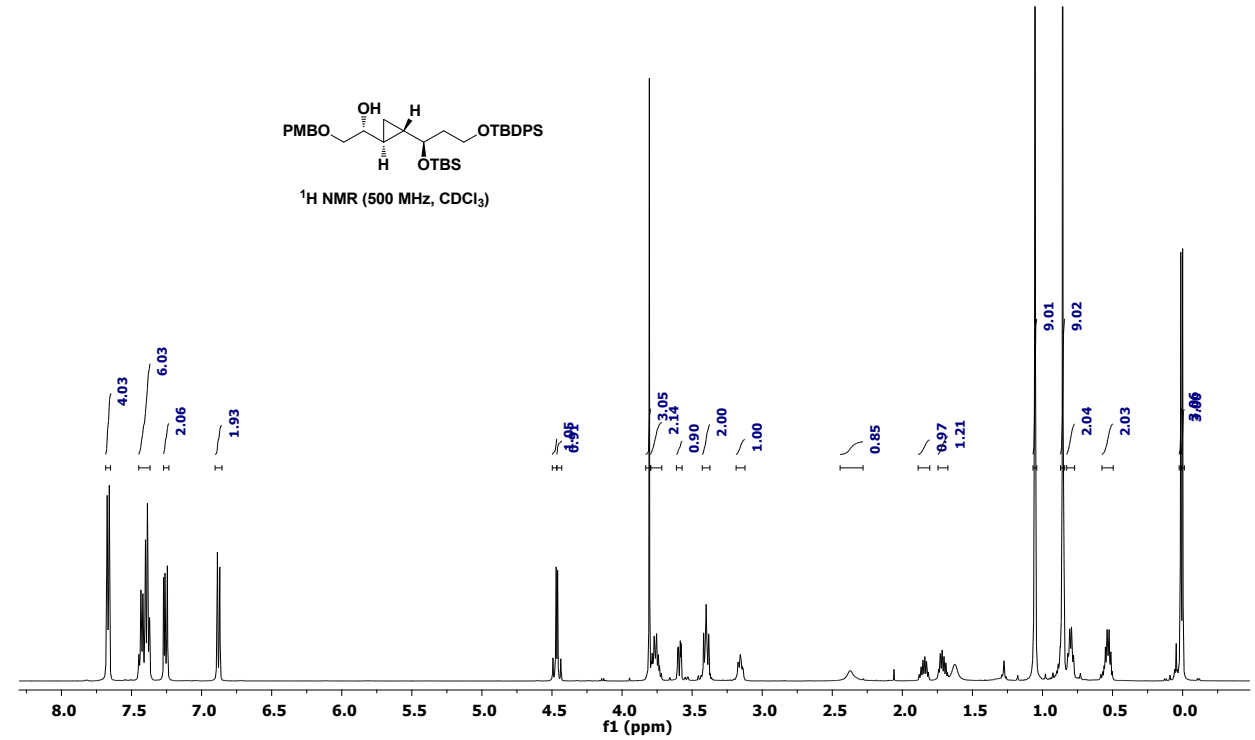
(*R*)-Mandelate ester of compound 36:

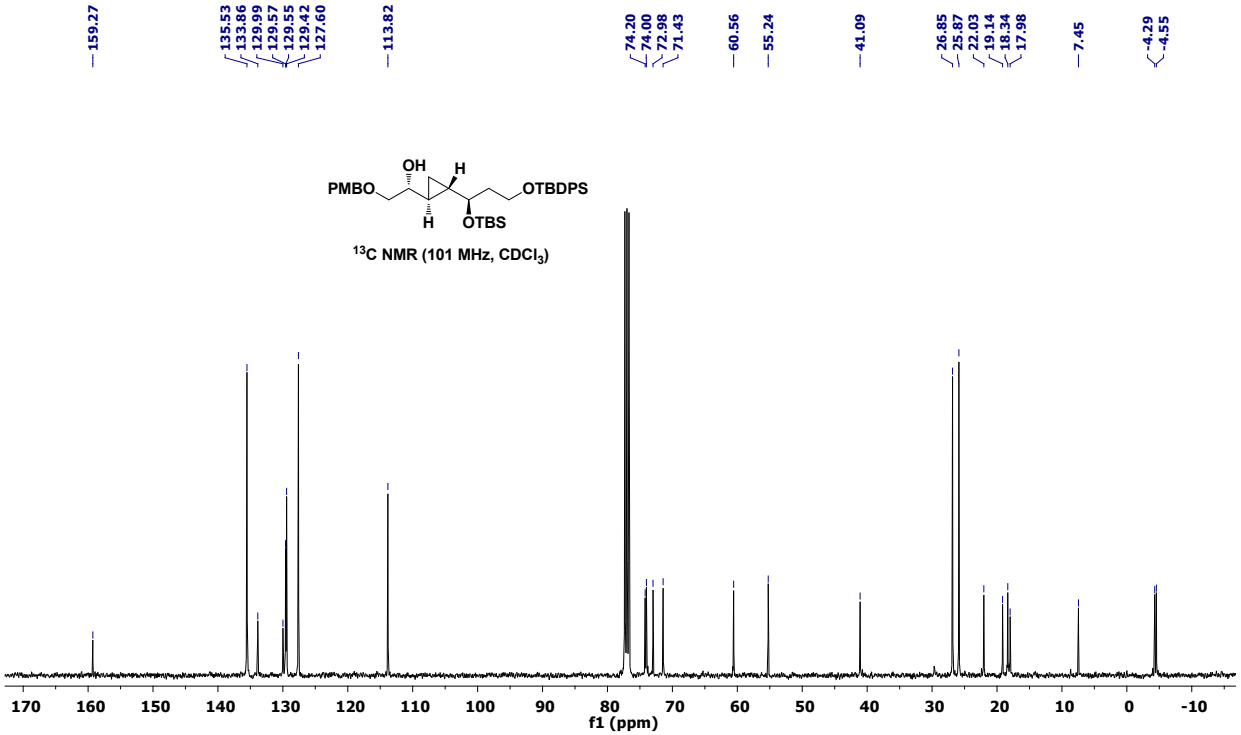


(*S*)-Mandelate ester of compound 36:

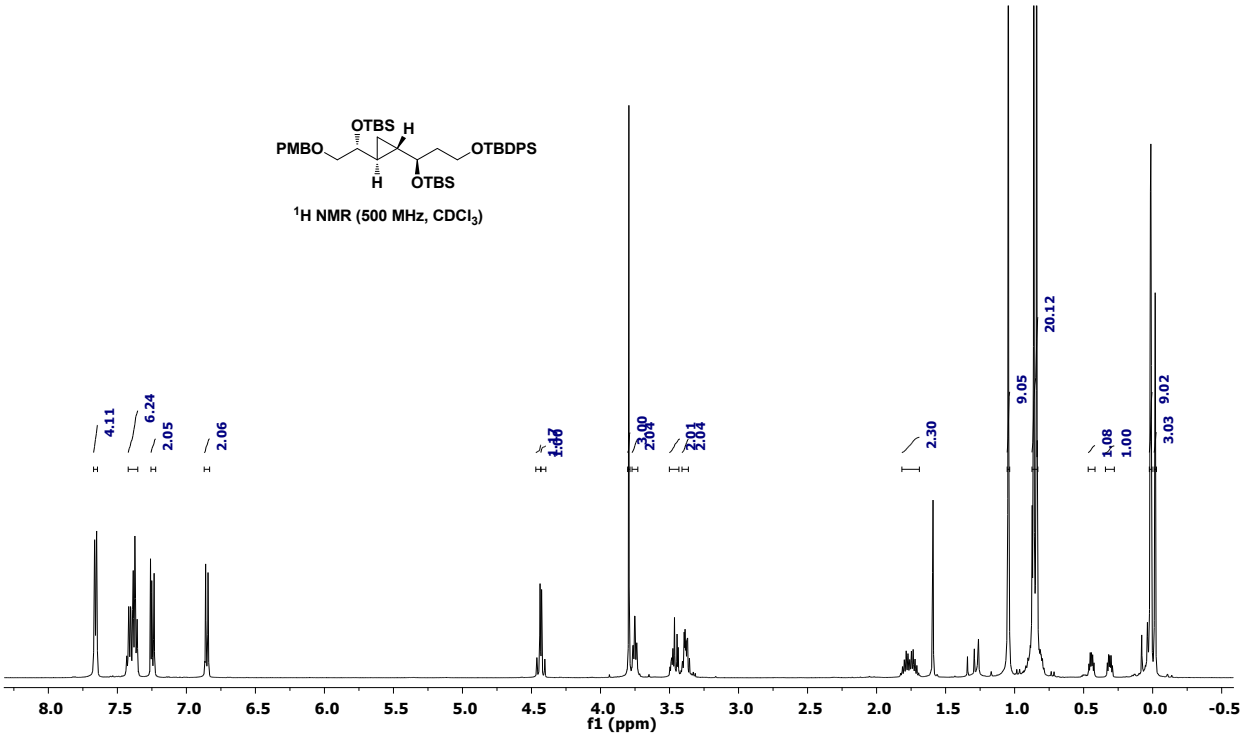


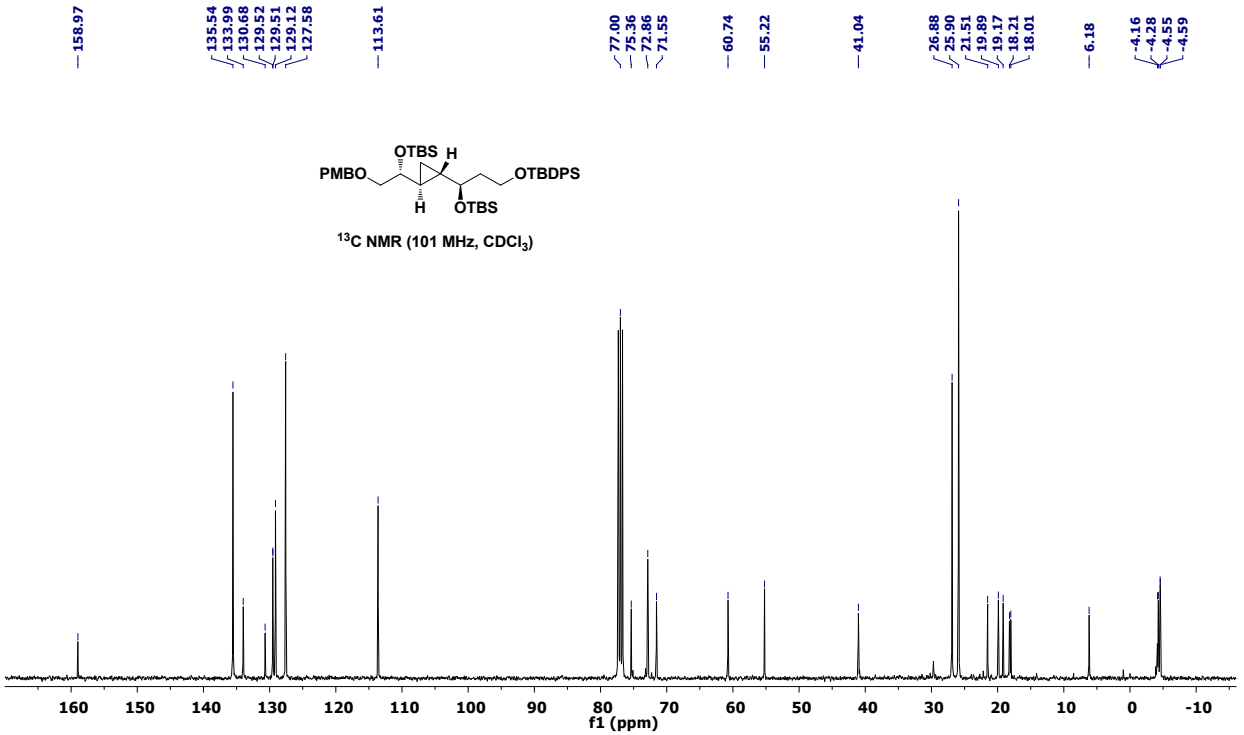
Compound 37:


OH
H
PMBO
OTBDPS
H
OTBS
^{1}H NMR (500 MHz, CDCl$_3$)
4.03
6.03
2.06
1.93
3.05
2.14
0.90
2.00
1.00
0.85
0.97
1.21
9.01
9.02
2.04
2.03
f1 (ppm)

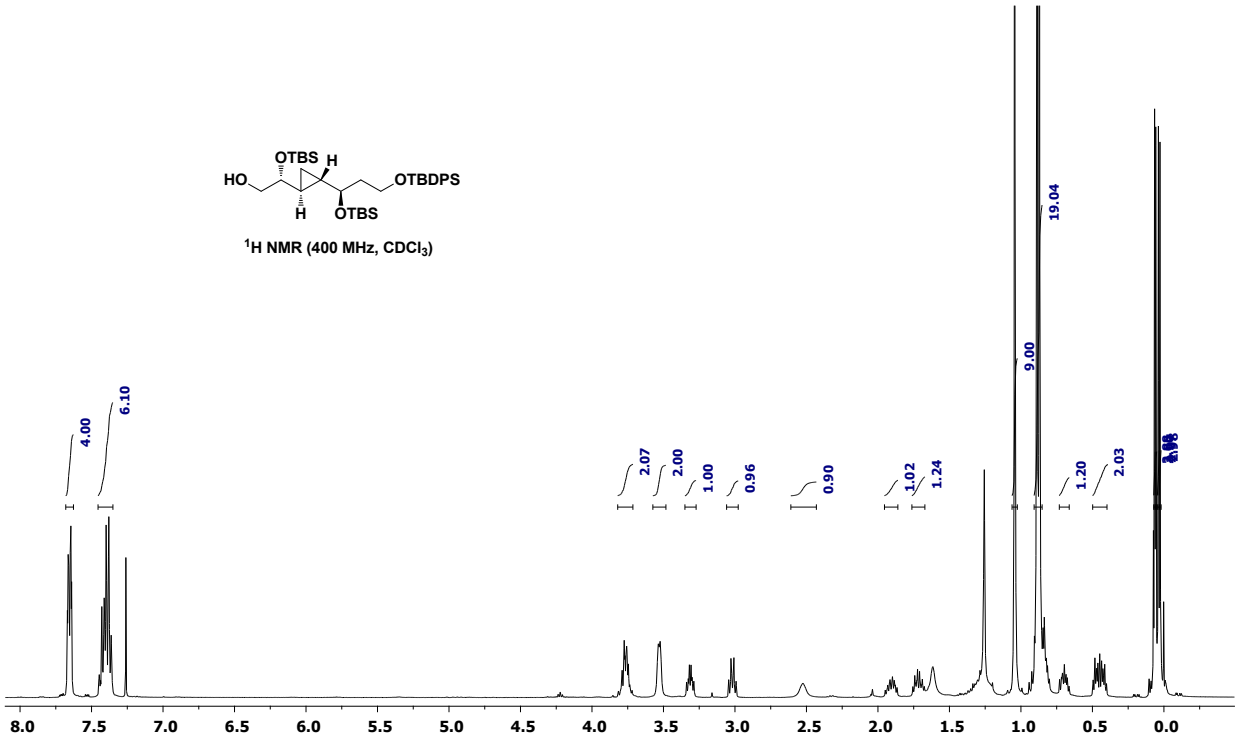

159.27
135.53
133.86
129.99
129.57
129.55
129.42
127.60
113.82
74.20
74.00
72.98
71.43
60.56
55.24
41.09
26.85
25.87
22.03
19.14
18.34
17.98
7.45
-4.29
-4.55
OH
H
PMBO
OTBDPS
H
OTBS
^{13}C NMR (101 MHz, CDCl$_3$)
f1 (ppm)

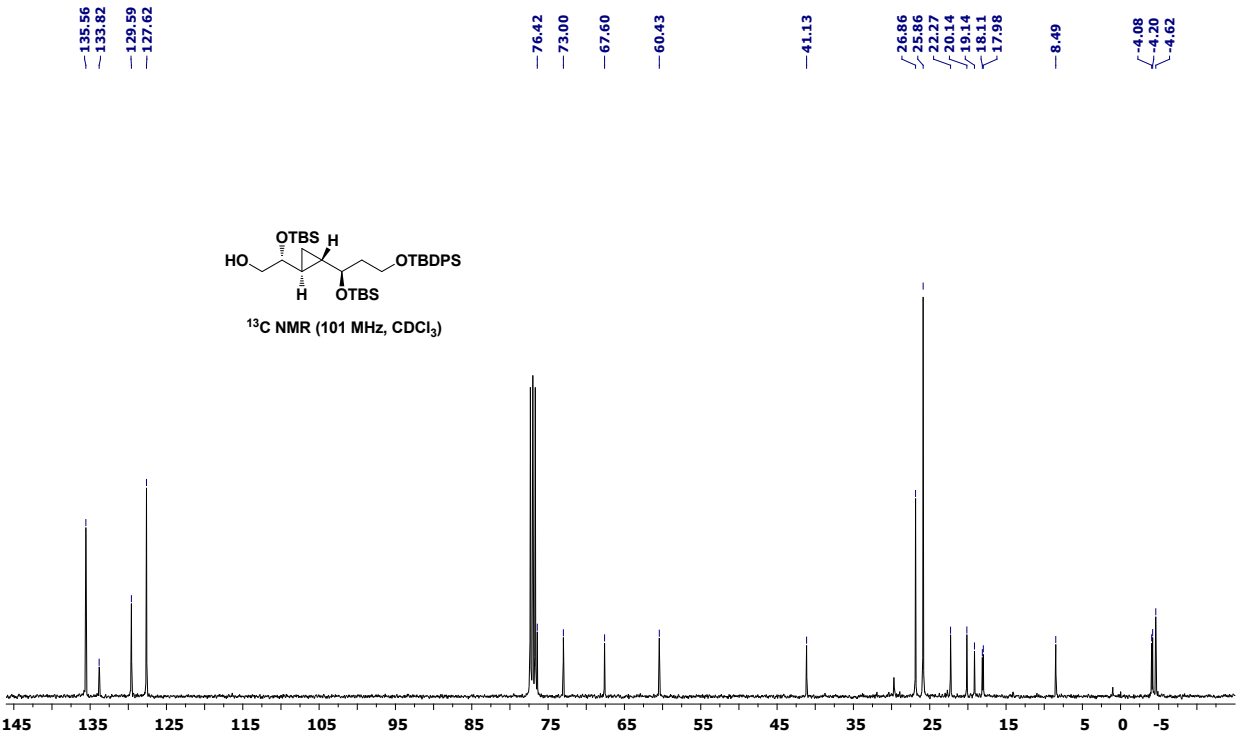
Compound 38:


OTBS H
PMBO
OTBDPS
H
OTBS
^{1}H NMR (500 MHz, CDCl$_3$)
4.11
6.24
2.05
2.06
2.30
9.05
20.12
1.08
1.00
9.02
3.03
f1 (ppm)

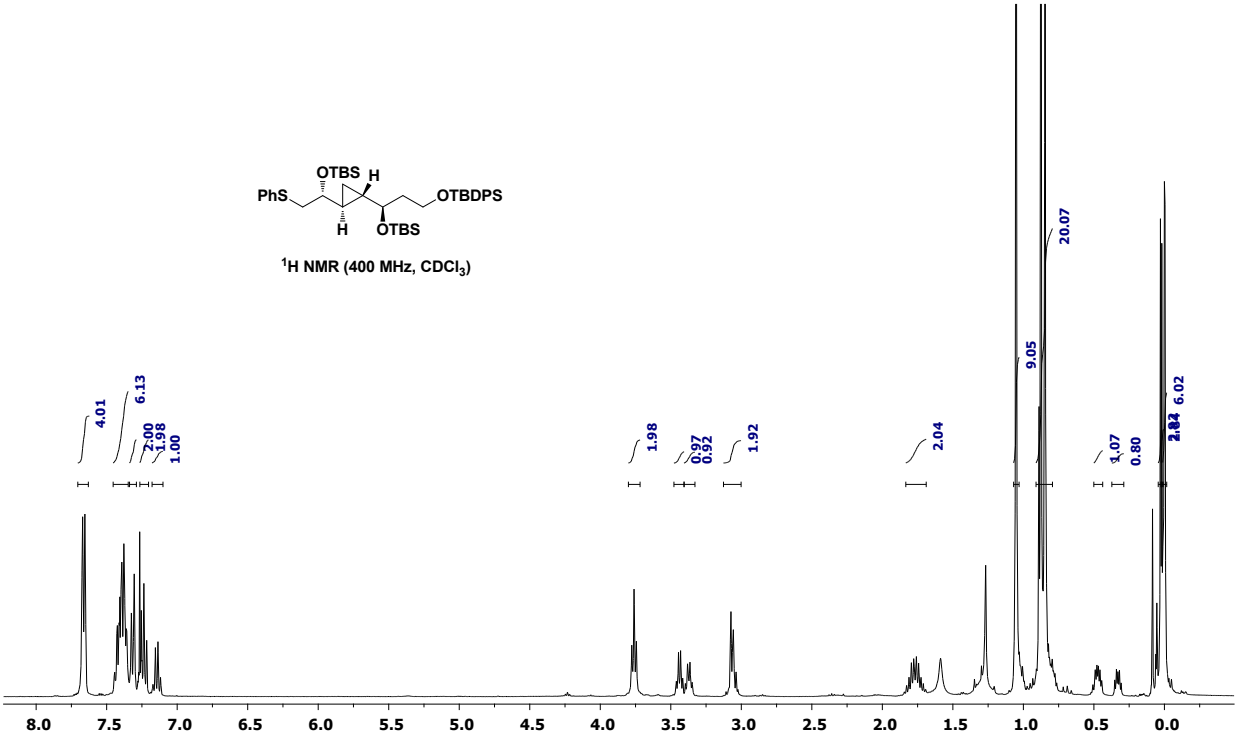

158.97
135.54
133.99
130.68
129.52
129.51
129.12
127.58
113.61
77.00
75.36
72.86
71.55
60.74
55.22
41.04
26.88
25.90
21.51
19.89
19.17
18.21
18.01
6.18
-4.16
-4.28
-4.55
-4.59
OTBS H
PMBO
OTBDPS
H
OTBS
^{13}C NMR (101 MHz, CDCl$_3$)
f1 (ppm)

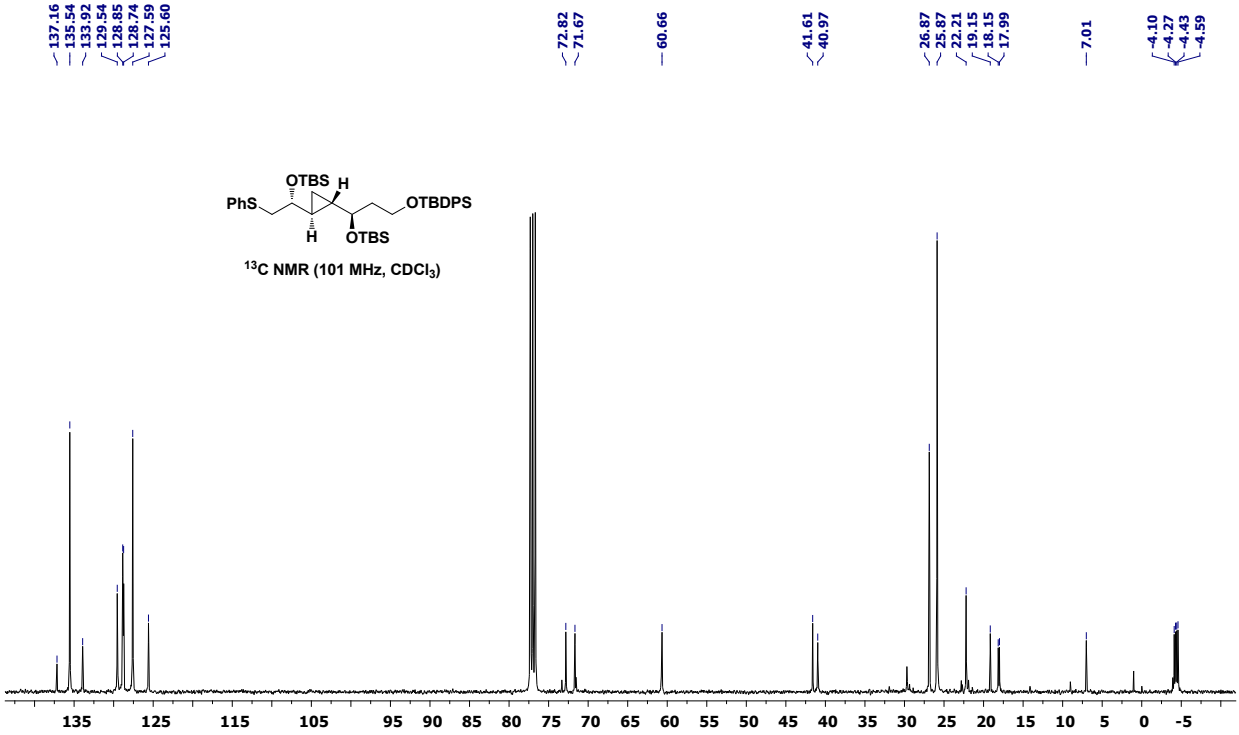
Compound 39:


OTBS H
HO
OTBDPS
H
OTBS
^{1}H NMR (400 MHz, CDCl$_3$)
4.00
6.10
2.07
2.00
1.00
0.96
0.90
1.02
1.24
9.00
19.04
1.20
2.03
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

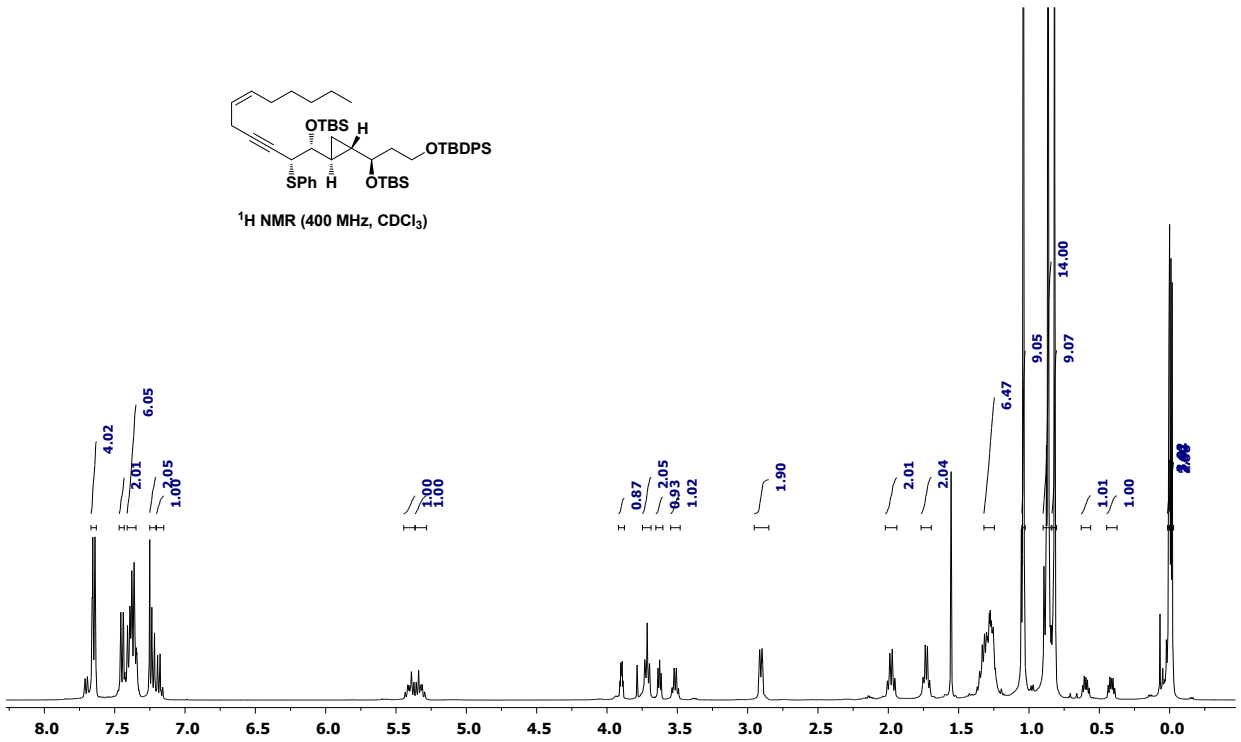

OTBS H
HO
OTBDPS
H
OTBS
^{13}C NMR (101 MHz, CDCl$_3$)
135.56
133.82
129.59
127.62
76.42
73.00
67.60
60.43
41.13
26.86
25.86
22.27
20.14
19.14
18.11
17.98
8.49
-4.08
-4.20
-4.62
145
135
125
115
105
95
85
75
65
55
45
35
25
15
5
0
-5

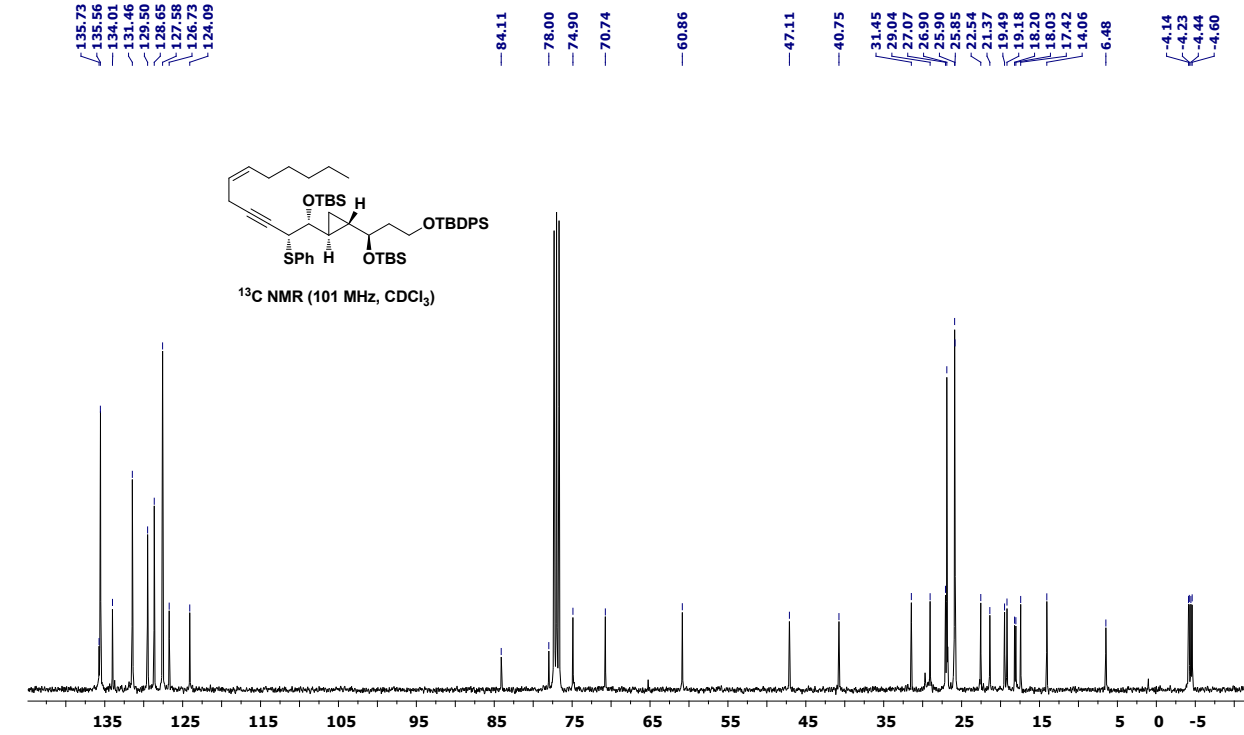
Compound 40:


OTBS H
PhS
OTBDPS
H
OTBS
^{1}H NMR (400 MHz, CDCl$_3$)
4.01
6.13
2.00
1.98
1.00
1.98
0.97
0.92
1.92
2.04
9.05
20.07
1.07
0.80
6.02
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

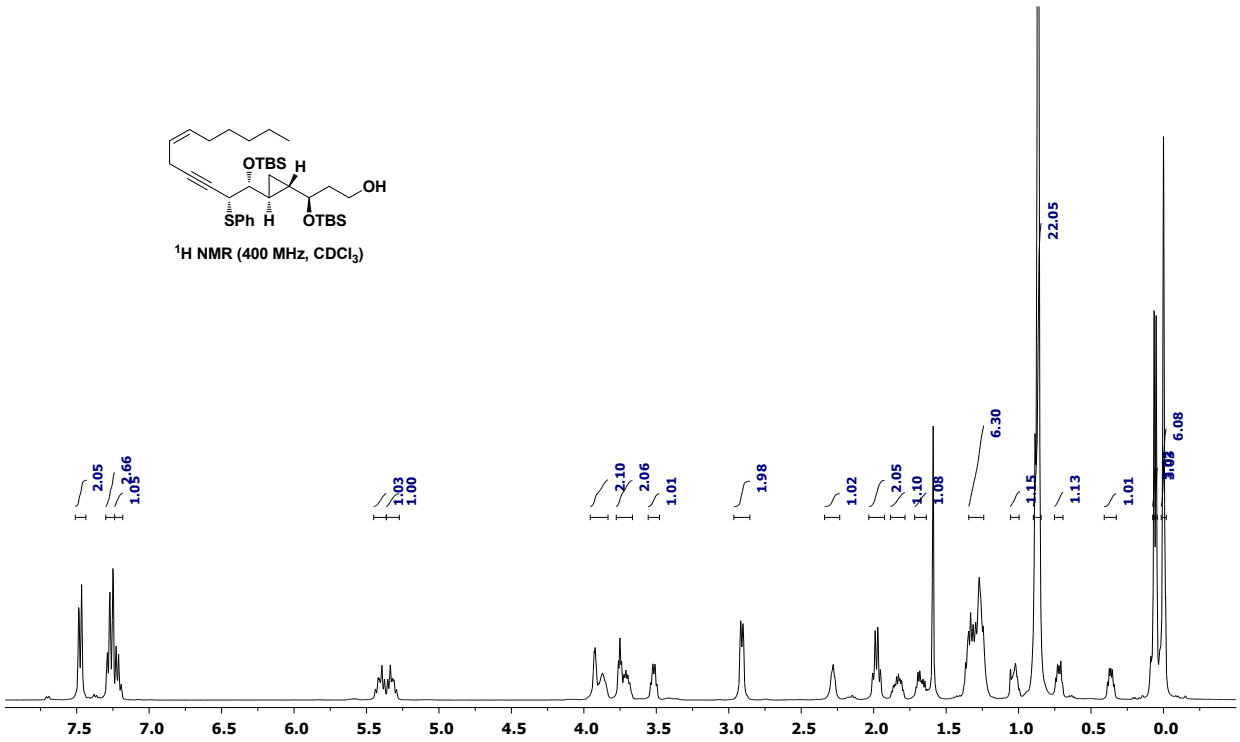

137.16
135.54
133.92
129.54
128.85
128.74
127.59
125.60
72.82
71.67
60.66
41.61
40.97
26.87
25.87
22.21
19.15
18.15
17.99
7.01
-4.10
-4.27
-4.43
-4.59
OTBS H
PhS
OTBDPS
H
OTBS
^{13}C NMR (101 MHz, CDCl$_3$)
135
125
115
105
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0
-5

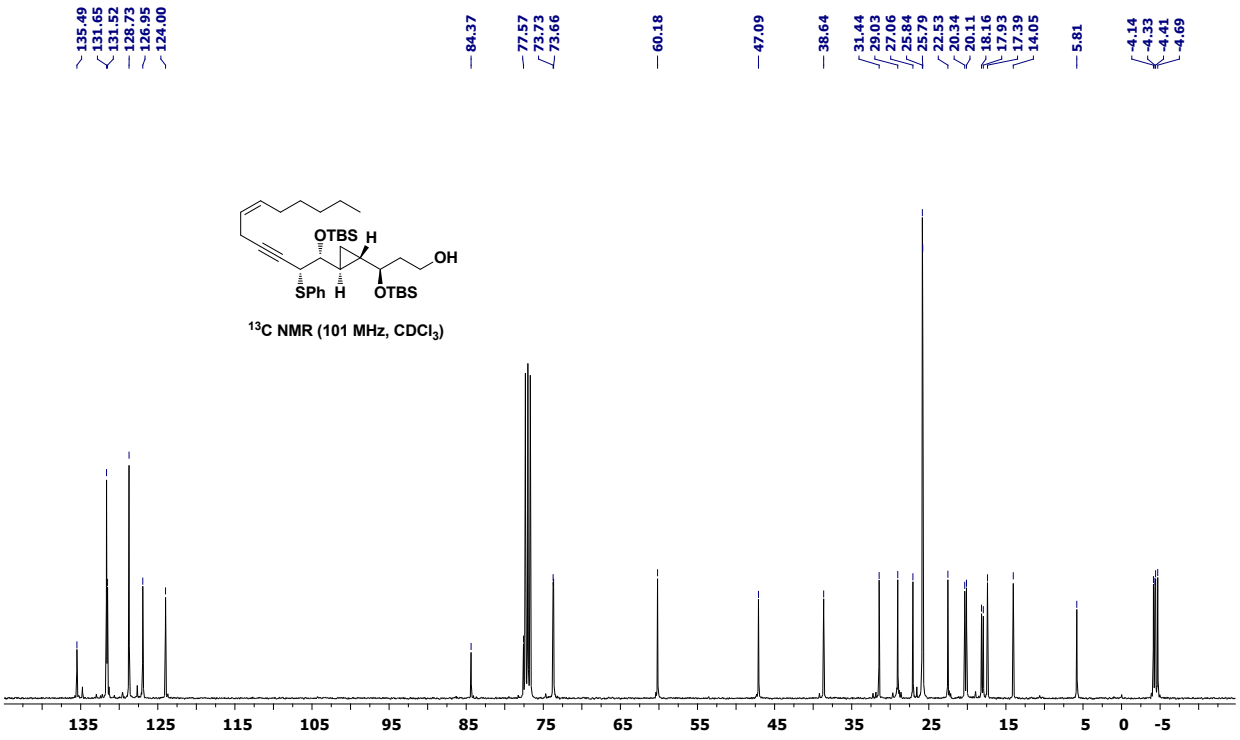
Compound 41:


OTBS H
OTBDPS
SPh H OTBS
^{1}H NMR (400 MHz, CDCl$_3$)
4.02
2.01
6.05
2.05
1.00
1.00
1.00
0.87
2.05
0.93
1.02
1.90
2.01
2.04
6.47
9.05
14.00
9.07
1.01
1.00
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

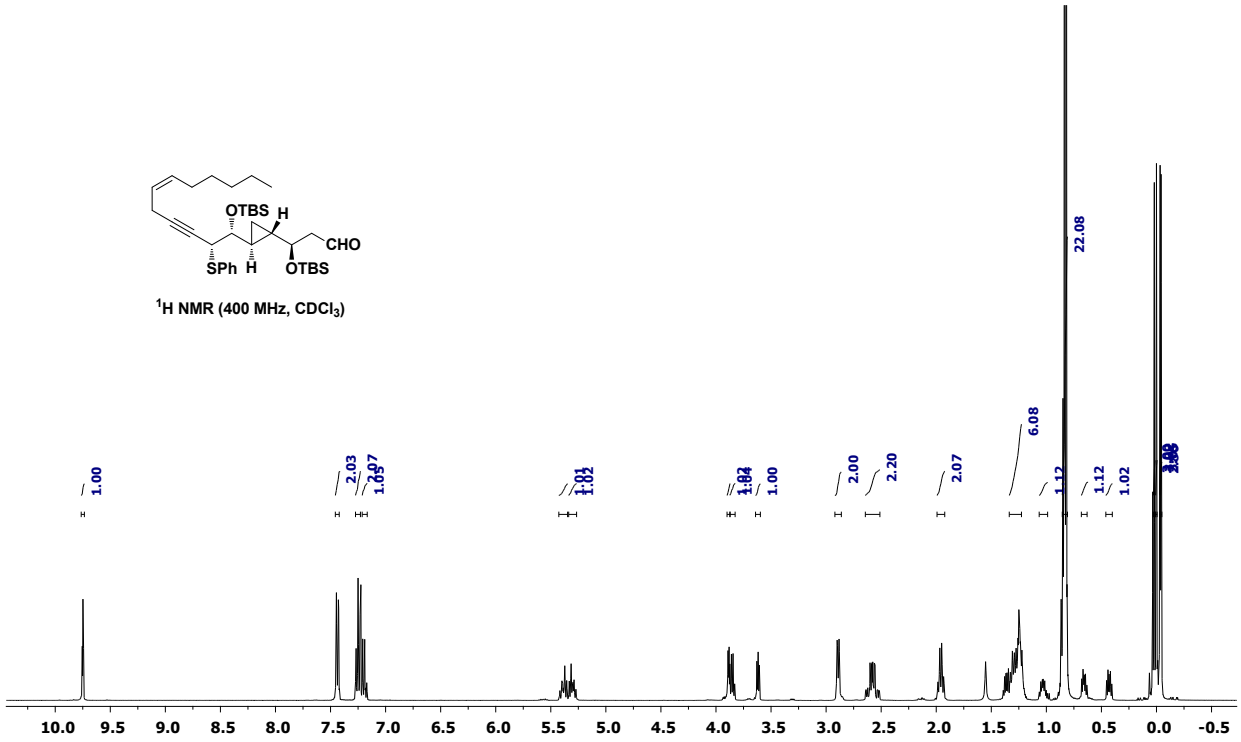

135.73
135.56
134.01
131.46
129.50
128.65
127.58
126.73
124.09
84.11
78.00
74.90
70.74
60.86
47.11
40.75
31.45
29.04
27.07
26.90
25.90
25.85
22.54
21.37
19.49
19.18
18.20
18.03
17.42
14.06
6.48
-4.14
-4.23
-4.44
-4.60
OTBS H
OTBDPS
SPh H OTBS
^{13}C NMR (101 MHz, CDCl$_3$)
135
125
115
105
95
85
75
65
55
45
35
25
15
5
0
-5

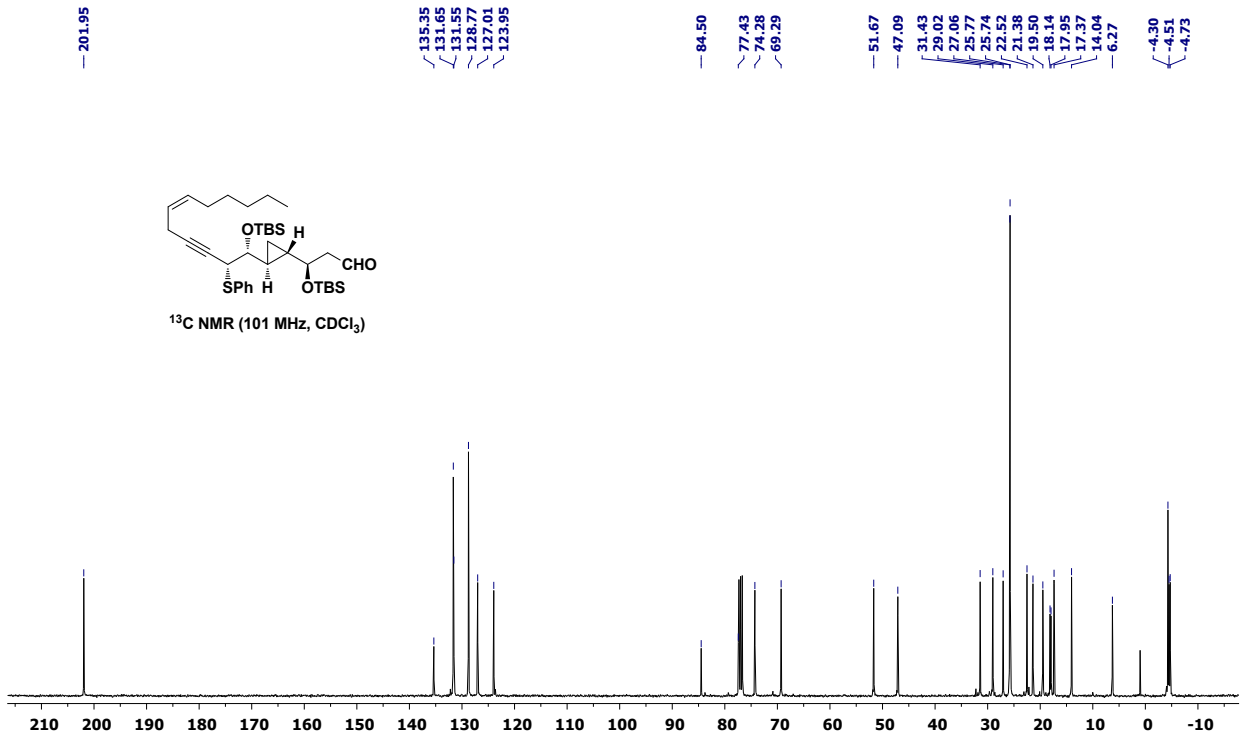
Compound 42:


OTBS H
OH
SPh H OTBS
^{1}H NMR (400 MHz, CDCl$_3$)
2.05
2.66
1.03
3.03
1.00
2.10
2.06
1.01
1.98
1.02
2.05
3.10
1.08
6.30
22.05
1.15
1.13
1.01
6.08
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

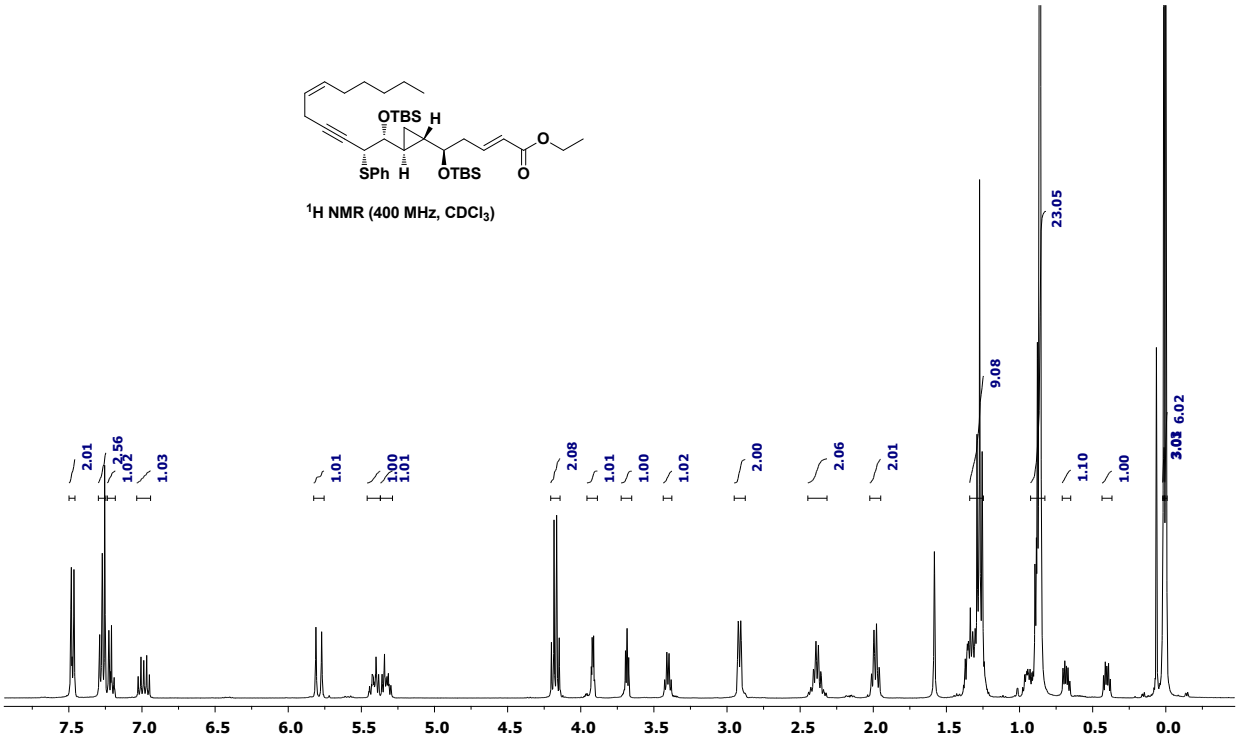

135.49
131.65
131.52
128.73
126.95
124.00
84.37
77.57
73.73
73.66
60.18
47.09
38.64
31.44
29.03
27.06
25.84
25.79
22.53
20.34
20.11
18.16
17.93
17.39
14.05
5.81
-4.14
-4.33
-4.41
-4.69
OTBS H
OH
SPh H OTBS
^{13}C NMR (101 MHz, CDCl$_3$)
135 125 115 105 95 85 75 65 55 45 35 25 15 5 0 -5

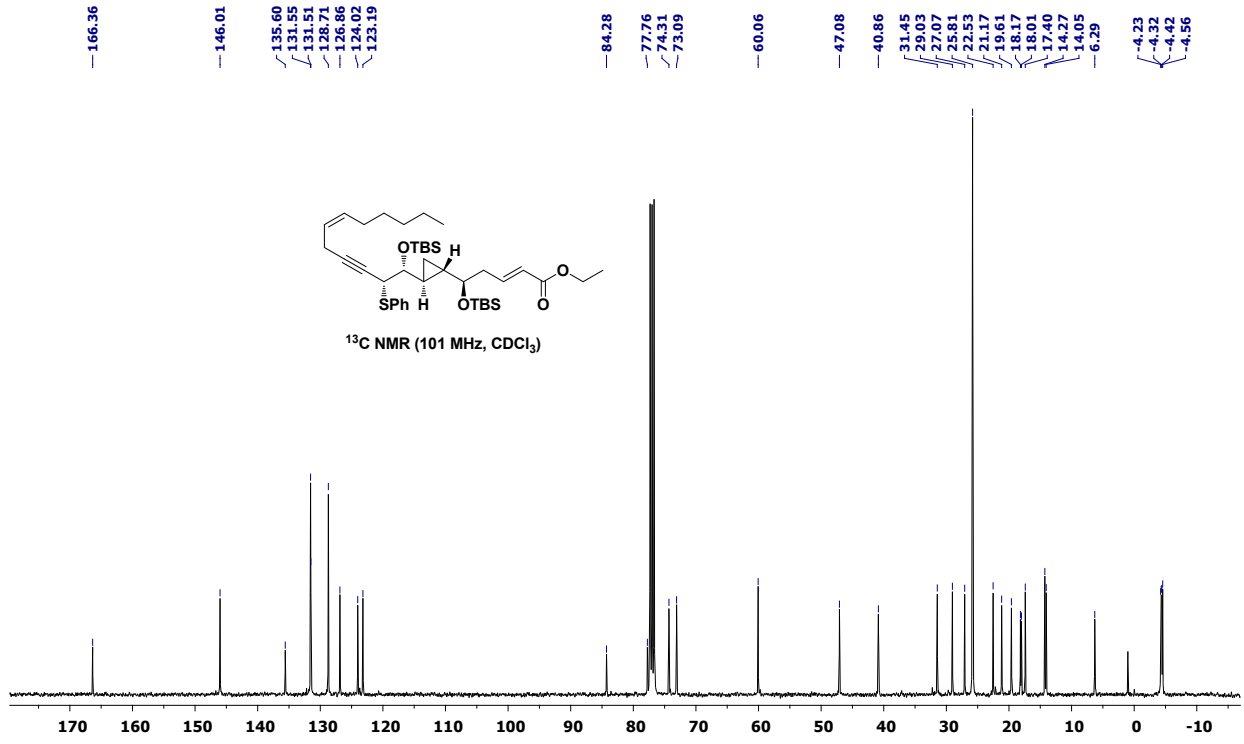
Compound 43:


OTBS H
CHO
SPh H OTBS
1H NMR (400 MHz, CDCl3)
1.00
2.03
2.07
1.05
1.01
1.02
1.03
1.04
1.00
2.00
2.20
2.07
6.08
1.12
22.08
1.12
1.02
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

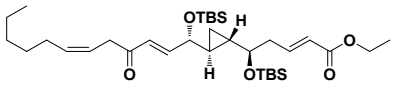

201.95
135.35
131.65
131.55
128.77
127.01
123.95
84.50
77.43
74.28
69.29
51.67
47.09
31.43
29.02
27.06
25.77
25.74
22.52
21.38
19.50
18.14
17.95
17.37
14.04
6.27
-4.30
-4.51
-4.73
OTBS H
CHO
SPh H OTBS
13C NMR (101 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

Compound 44:

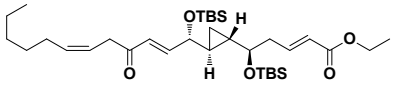

OTBS H
SPh H OTBS O
O
^{1}H NMR (400 MHz, CDCl$_3$)
2.01
2.56
1.02
1.03
1.01
1.00
1.01
2.08
1.01
1.00
1.02
2.00
2.06
2.01
9.08
23.05
1.10
1.00
6.02
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0


166.36
146.01
135.60
131.55
131.51
128.71
126.86
124.02
123.19
84.28
77.76
74.31
73.09
60.06
47.08
40.86
31.45
29.03
27.07
25.81
22.53
21.17
19.61
18.17
18.01
17.40
14.27
14.05
6.29
-4.23
-4.32
-4.42
-4.56
OTBS H
SPh H OTBS O
O
^{13}C NMR (101 MHz, CDCl$_3$)
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

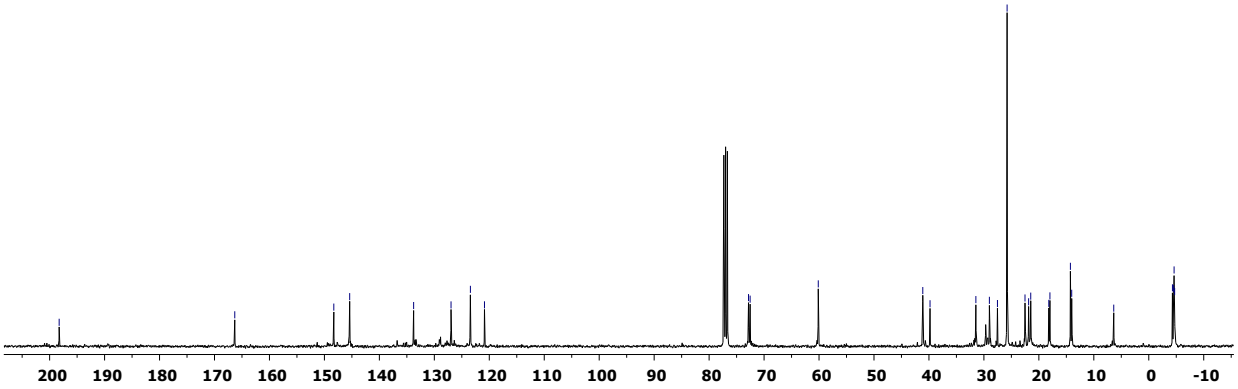
Compound 45:



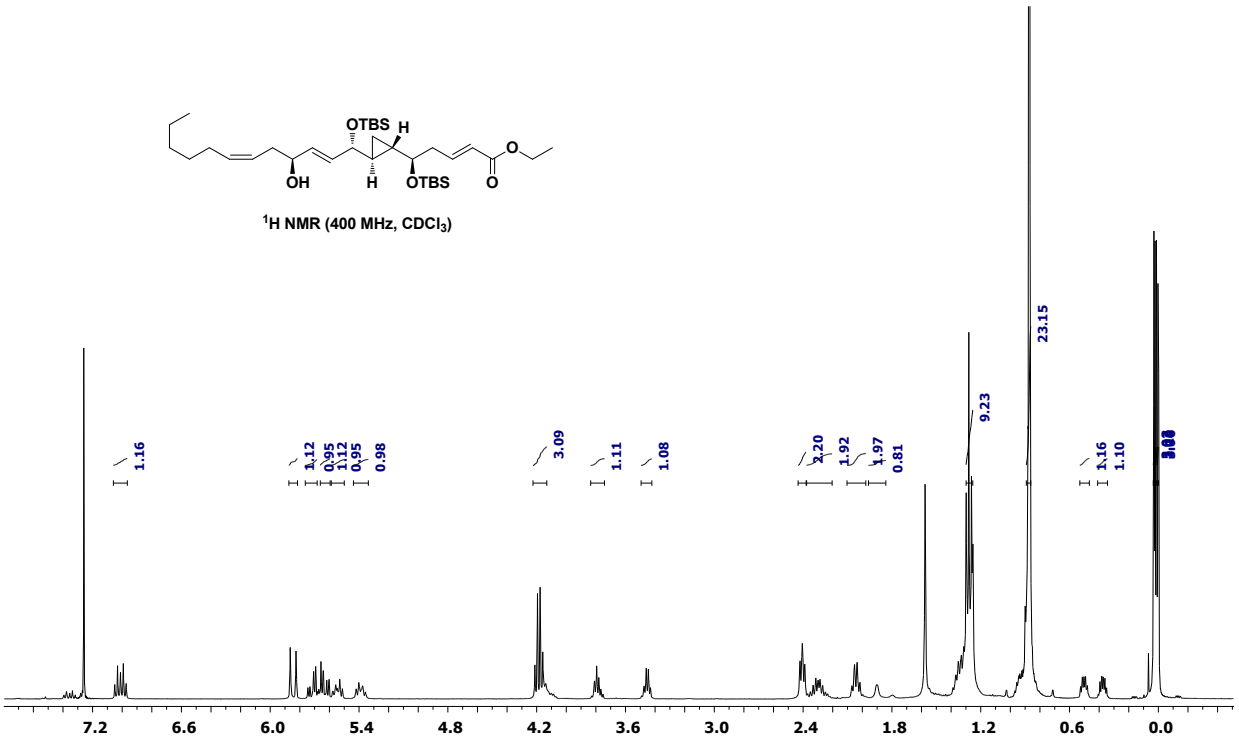
^{1}H NMR (400 MHz, $CDCl_3$)

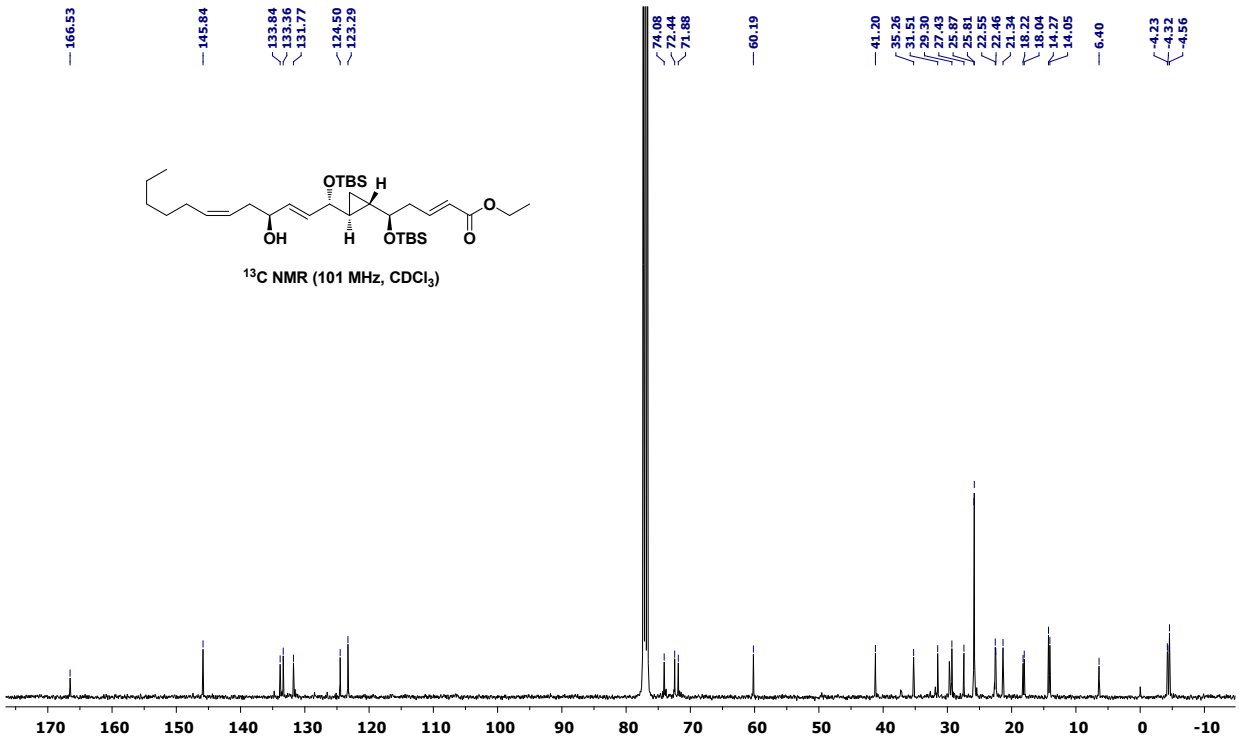


^{13}C NMR (101 MHz, $CDCl_3$)

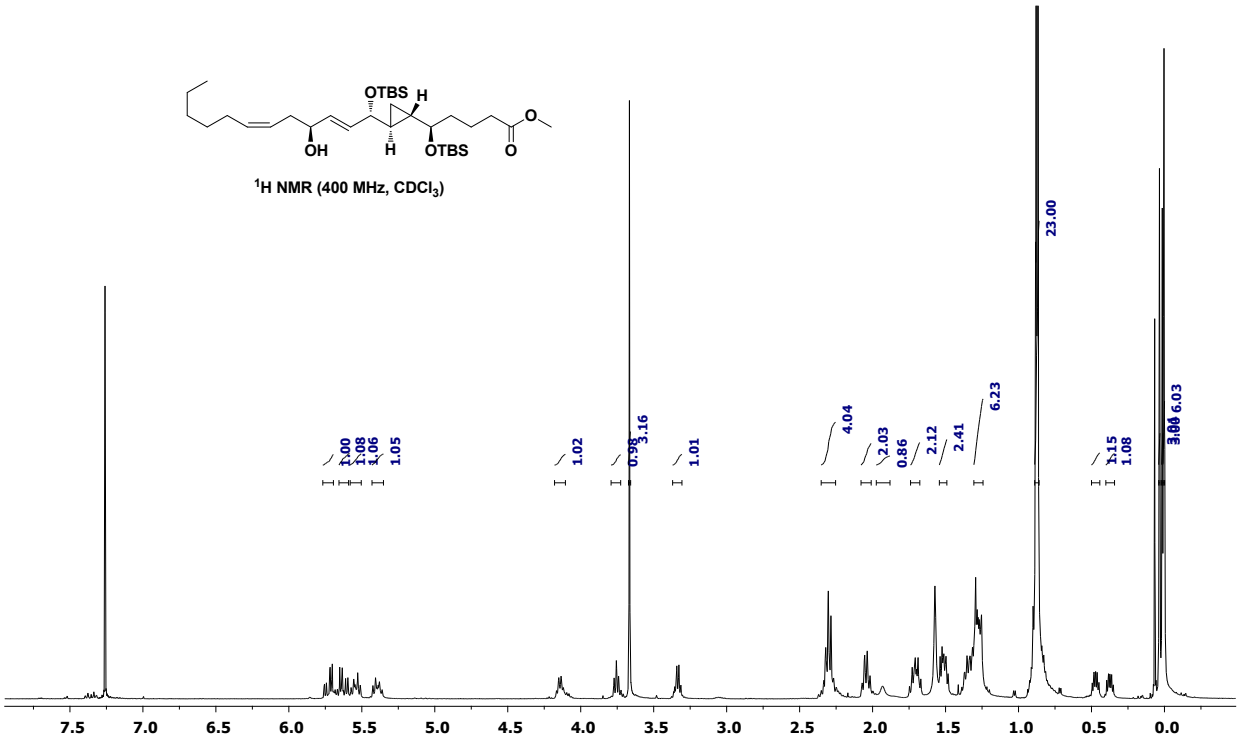


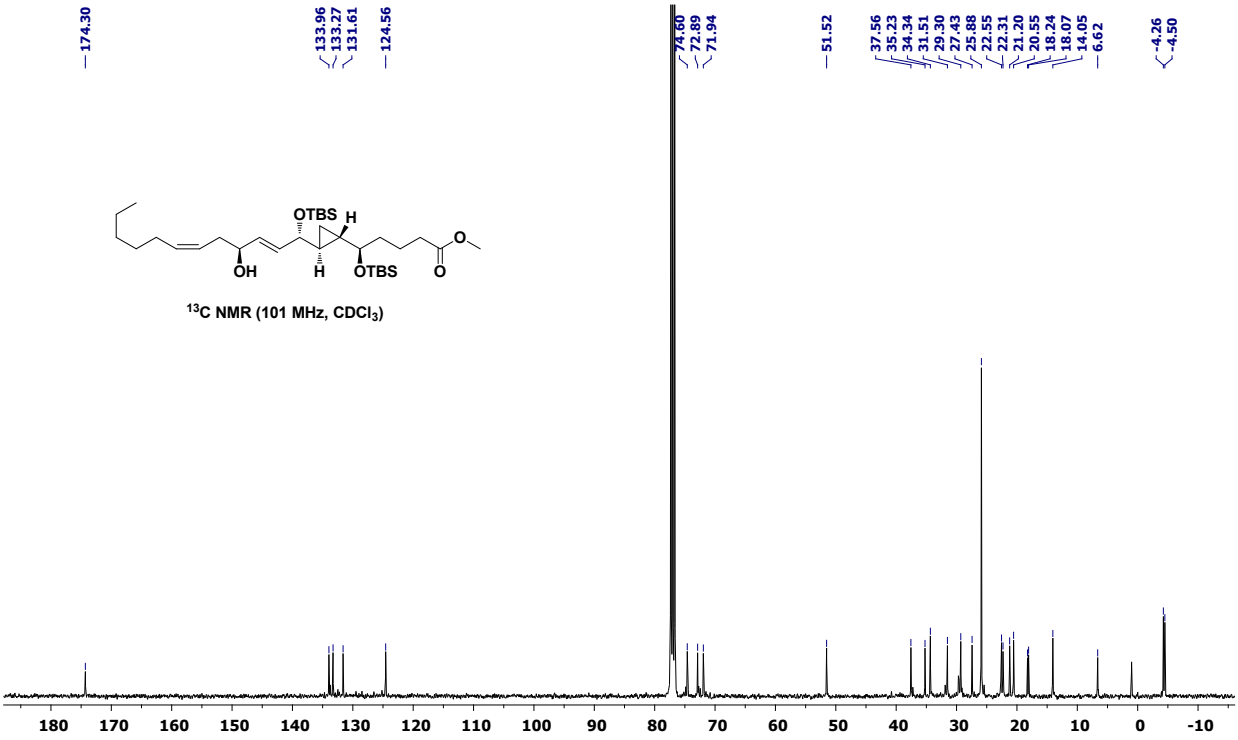
Compound 46:


OTBS H
O
OH
H
OTBS
O
^{1}H NMR (400 MHz, CDCl$_3$)
1.16
1.12
0.95
1.12
0.95
0.98
3.09
1.11
1.08
2.20
1.92
1.97
0.81
9.23
23.15
2.16
1.10
7.2
6.6
6.0
5.4
4.8
4.2
3.6
3.0
2.4
1.8
1.2
0.6
0.0

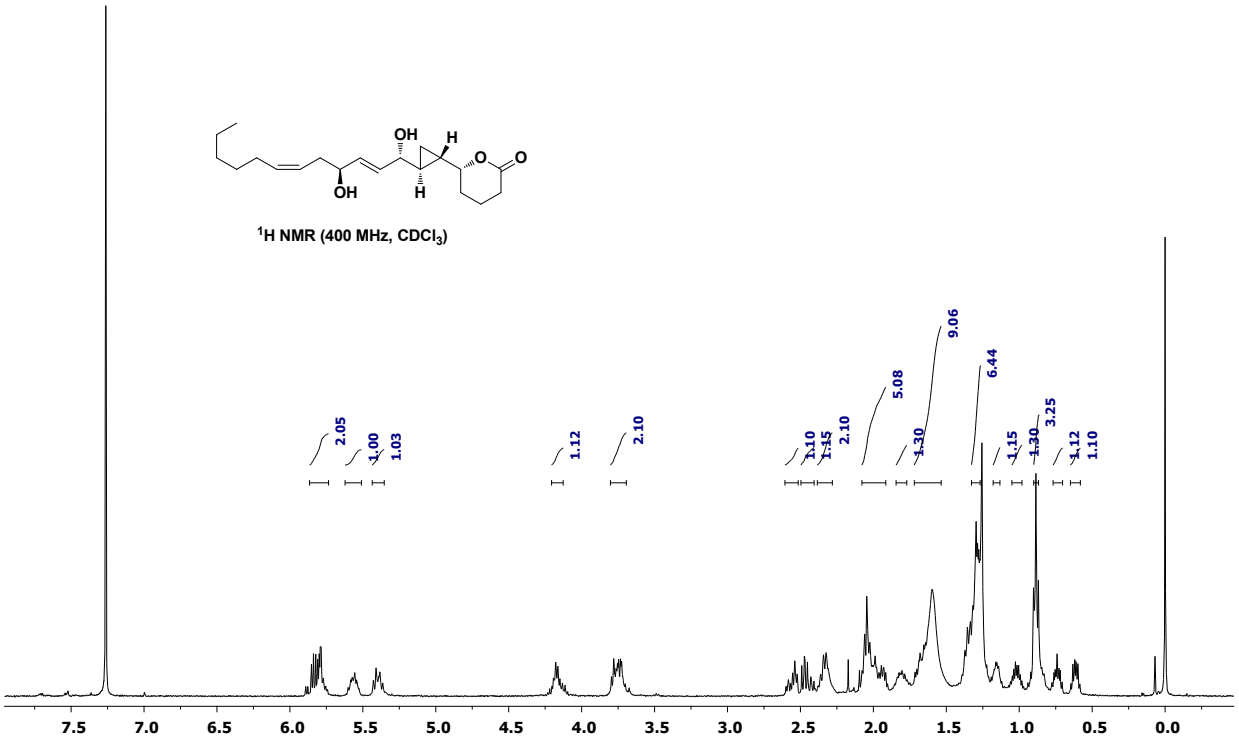

166.53
145.84
133.84
133.36
131.77
124.50
123.29
74.08
72.44
71.88
60.19
41.20
35.26
31.51
29.30
27.43
25.87
25.81
22.55
22.46
21.34
18.22
18.04
14.27
14.05
6.40
-4.23
-4.32
-4.56
OTBS H
O
OH
H
OTBS
O
^{13}C NMR (101 MHz, CDCl$_3$)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10

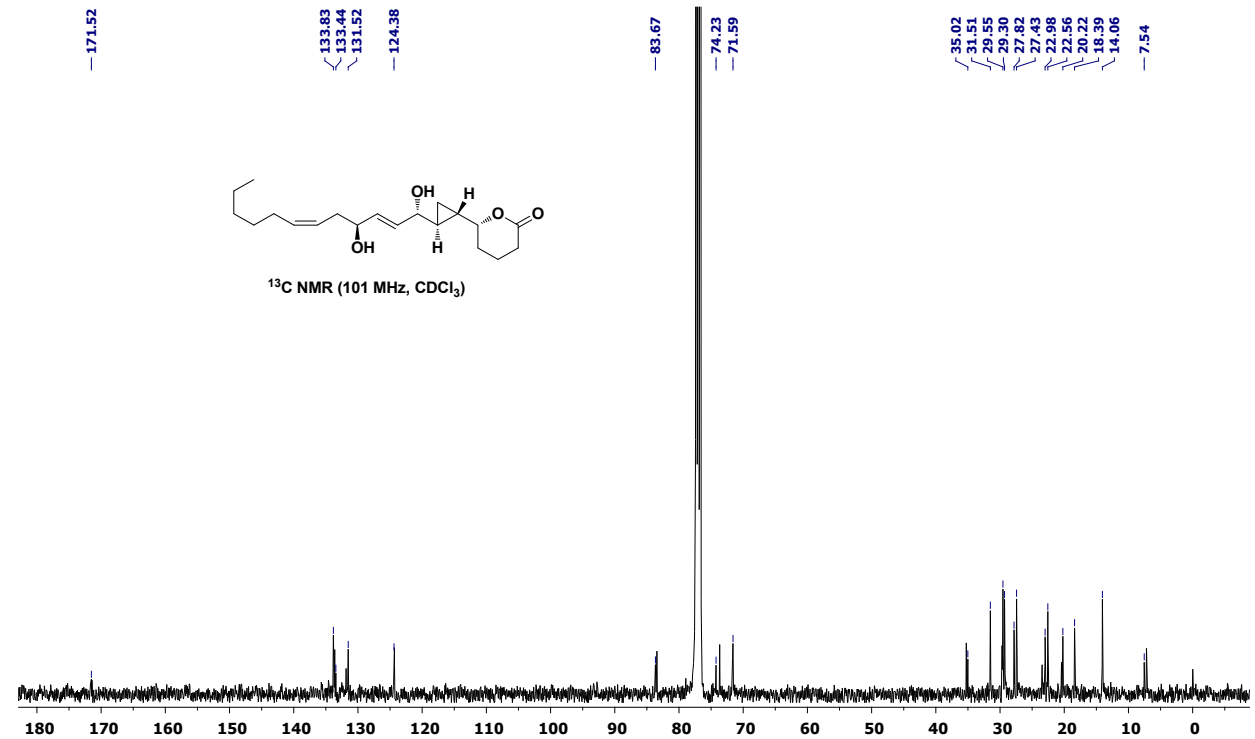
Compound 47:


OTBS H
O
OH
H
OTBS
O
^{1}H NMR (400 MHz, CDCl$_3$)
1.00
1.08
1.06
1.05
1.02
0.98
3.16
1.01
4.04
2.03
0.86
2.12
2.41
6.23
23.00
1.15
1.08
3.04
6.03
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

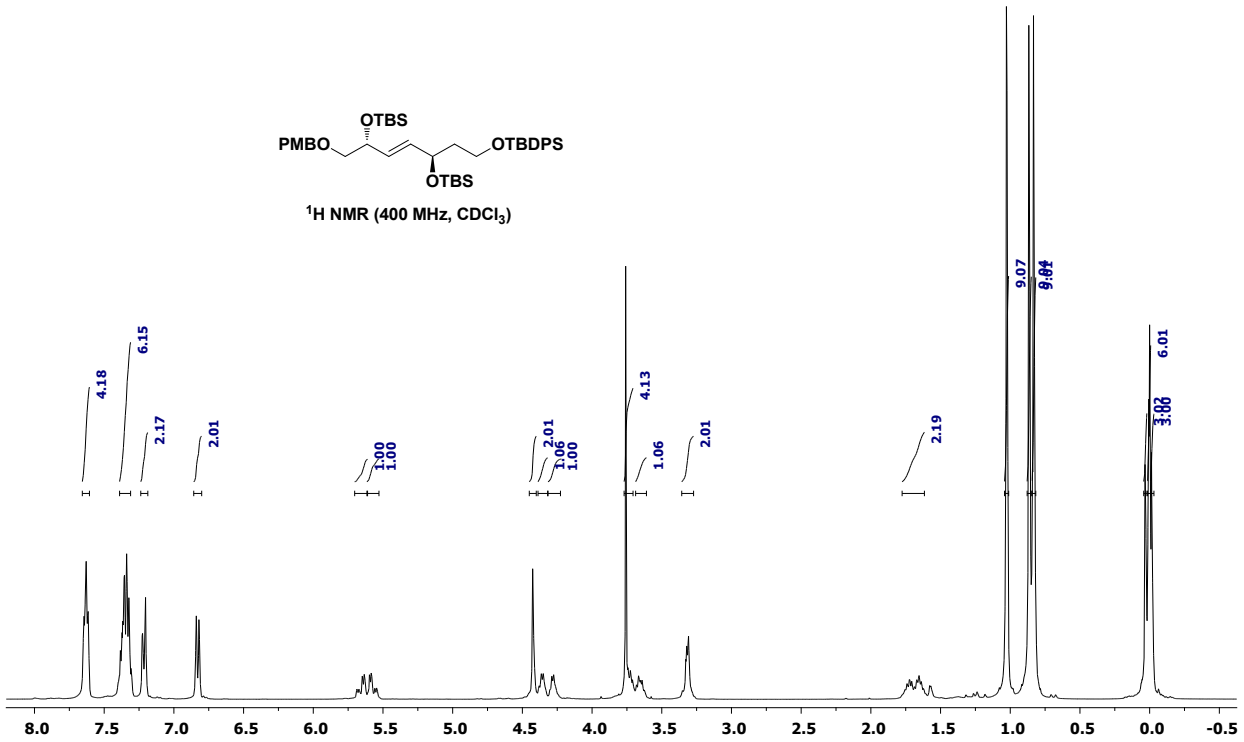

174.30
133.96
133.27
131.61
124.56
74.60
72.89
71.94
51.52
37.56
35.23
34.34
31.51
29.30
27.43
25.88
22.55
22.31
21.20
20.55
18.24
18.07
14.05
6.62
-4.26
-4.50
OTBS H
O
OH
H
OTBS
O
^{13}C NMR (101 MHz, CDCl$_3$)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10

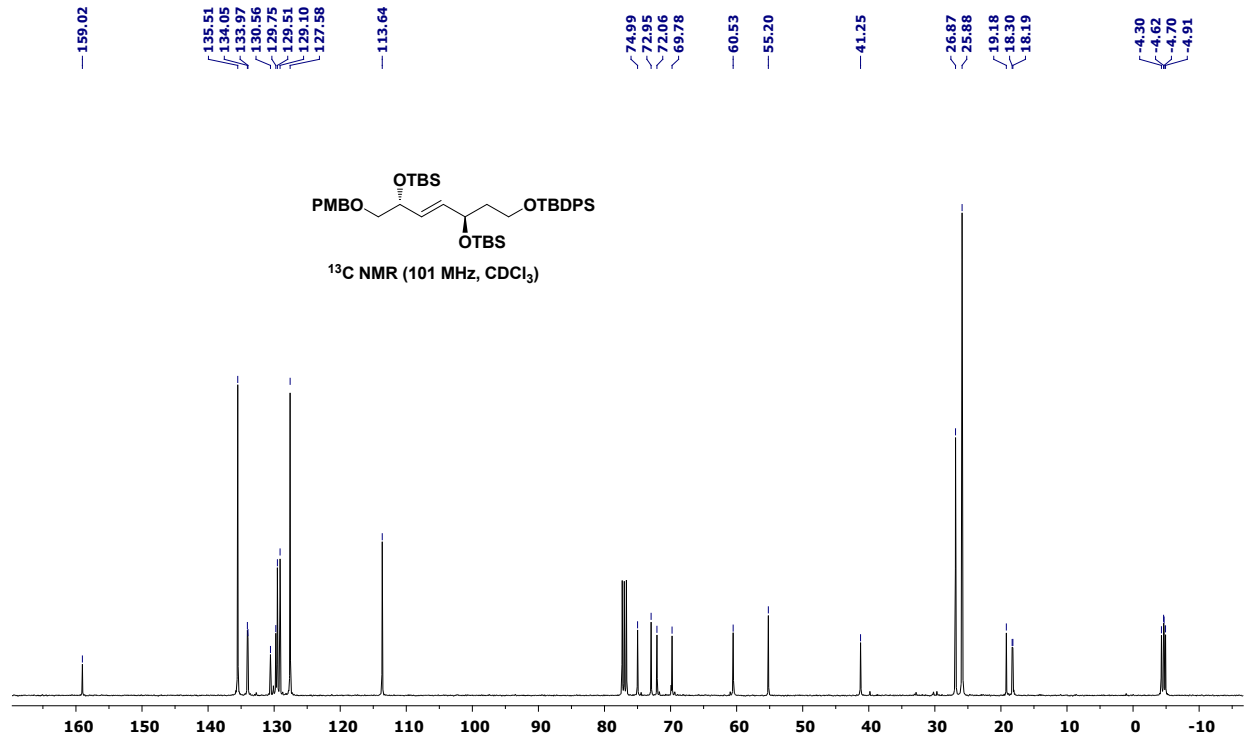
Compound 3:


OH
H
O
O
OH
H
^{1}H NMR (400 MHz, CDCl$_3$)
2.05
1.00
1.03
1.12
2.10
1.10
1.15
2.10
5.08
1.30
9.06
6.44
1.15
1.30
3.25
1.12
1.10
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

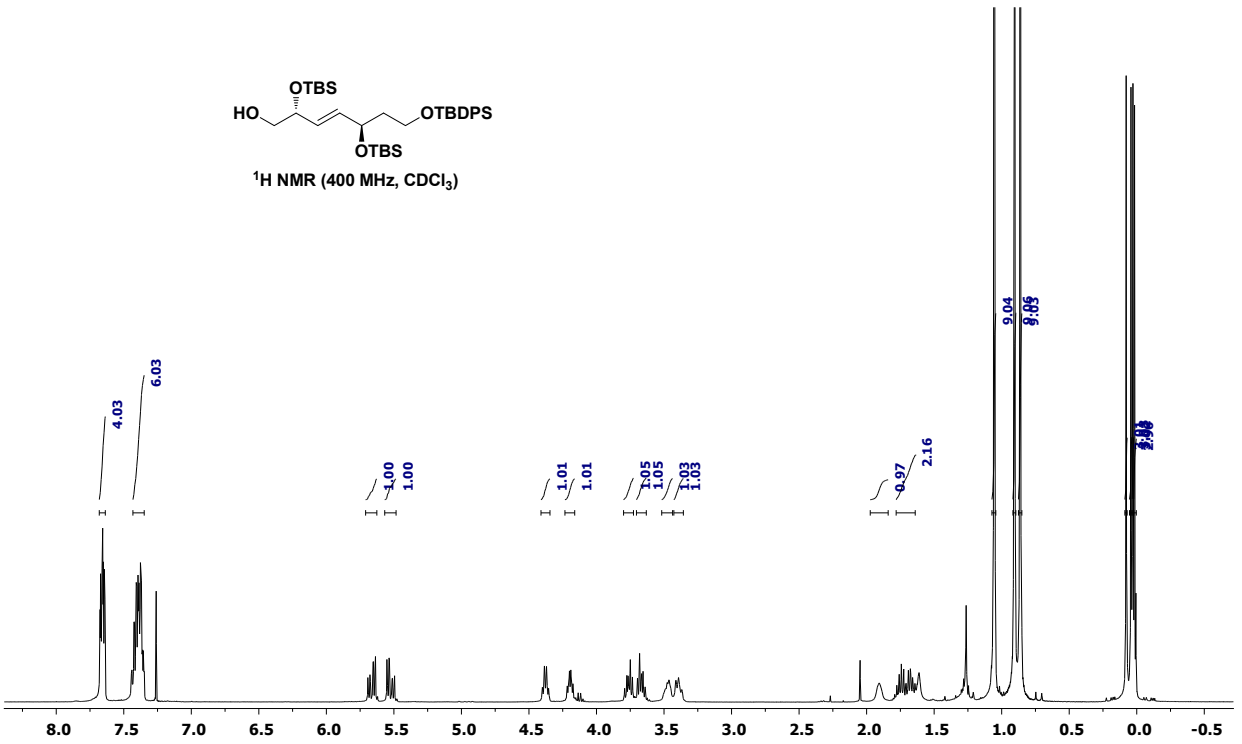

171.52
133.83
133.44
131.52
124.38
83.67
74.23
71.59
35.02
31.51
29.55
29.30
27.82
27.43
22.98
22.56
20.22
18.39
14.06
7.54
OH
H
O
O
OH
H
^{13}C NMR (101 MHz, CDCl$_3$)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0

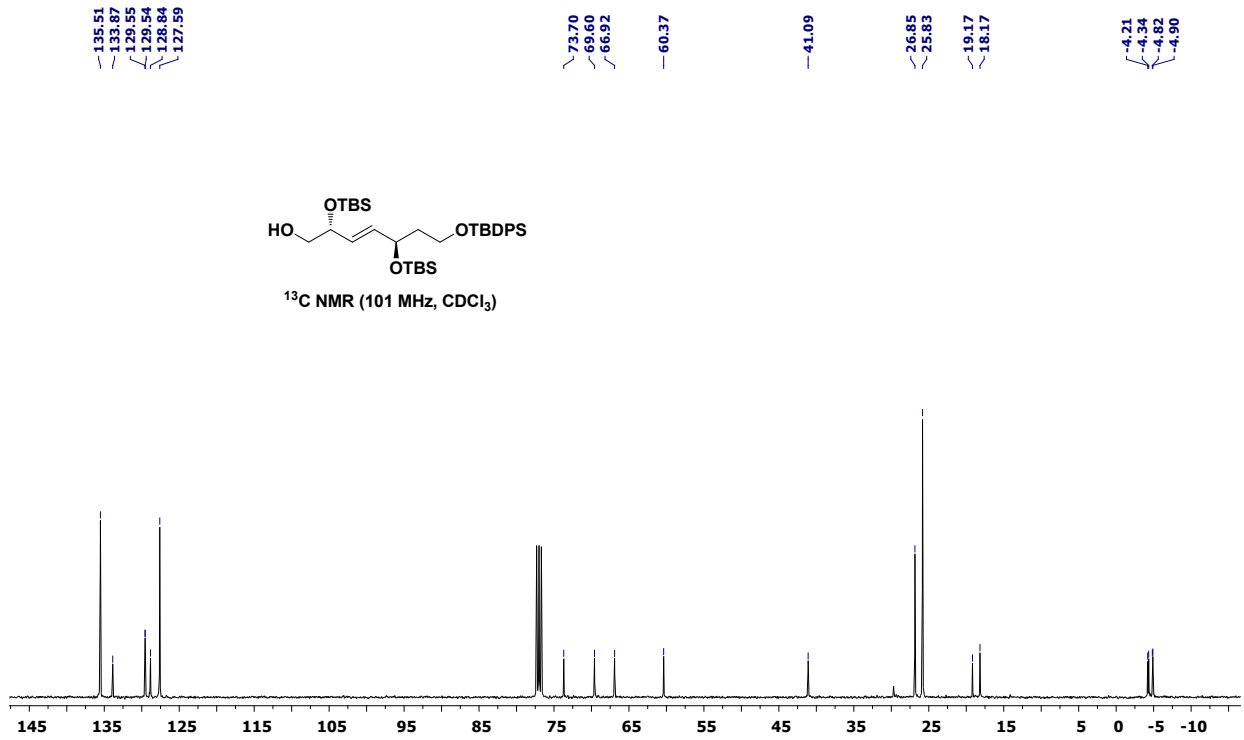
Compound 48:


OTBS
PMBO
OTBDPS
OTBS
^{1}H NMR (400 MHz, CDCl$_3$)
4.18
6.15
2.17
2.01
1.00
1.00
2.01
1.06
1.00
4.13
1.06
2.01
2.19
9.07
6.01
3.00

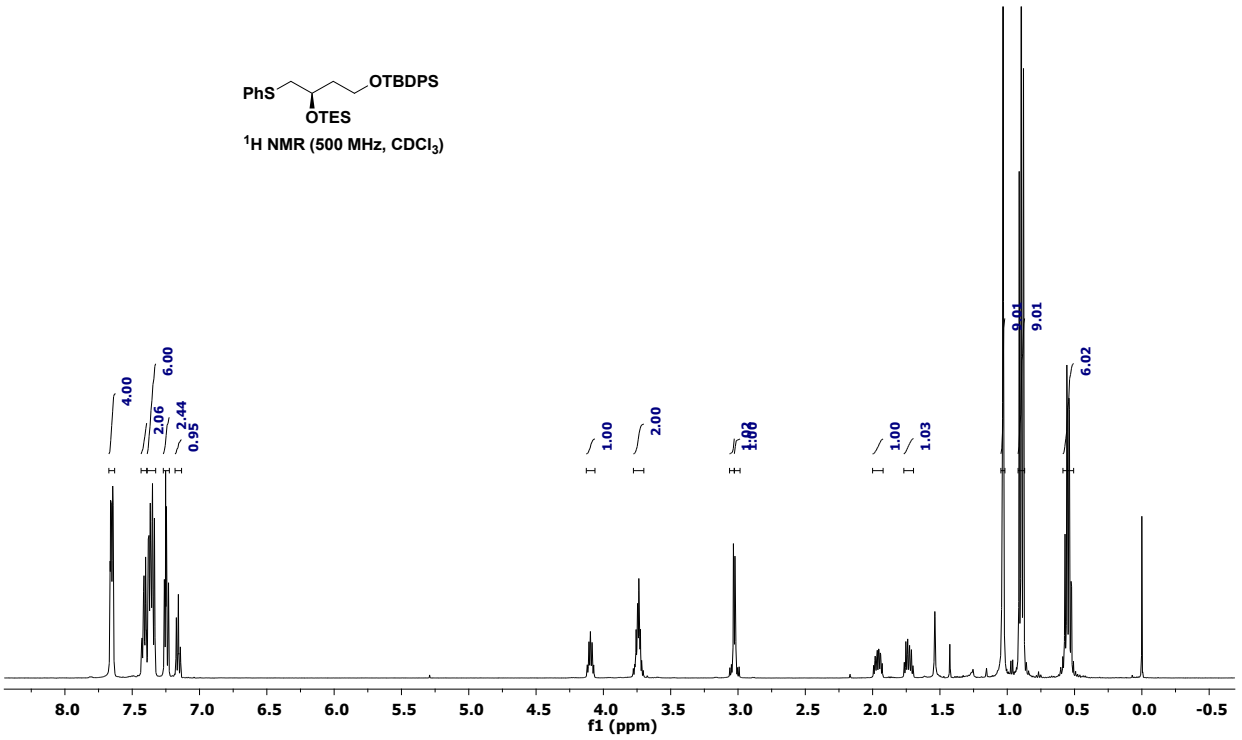

OTBS
PMBO
OTBDPS
OTBS
^{13}C NMR (101 MHz, CDCl$_3$)
159.02
135.51
134.05
133.97
130.56
129.75
129.51
129.10
127.58
113.64
74.99
72.95
72.06
69.78
60.53
55.20
41.25
26.87
25.88
19.18
18.30
18.19
-4.30
-4.62
-4.70
-4.91

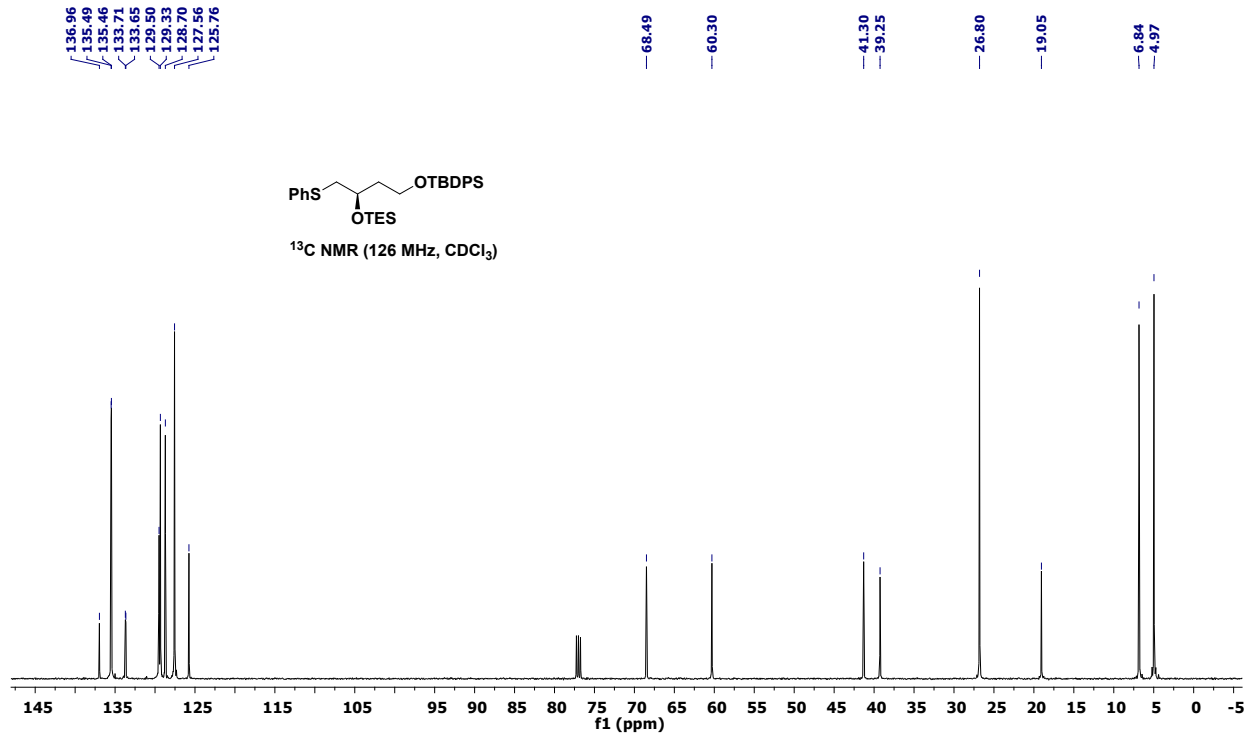
Compound 49:


OTBS
HO
OTBDPS
OTBS
^{1}H NMR (400 MHz, CDCl$_3$)
4.03
6.03
1.00
1.00
1.01
1.01
1.05
1.05
1.03
1.03
0.97
2.16
9.04
9.05
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

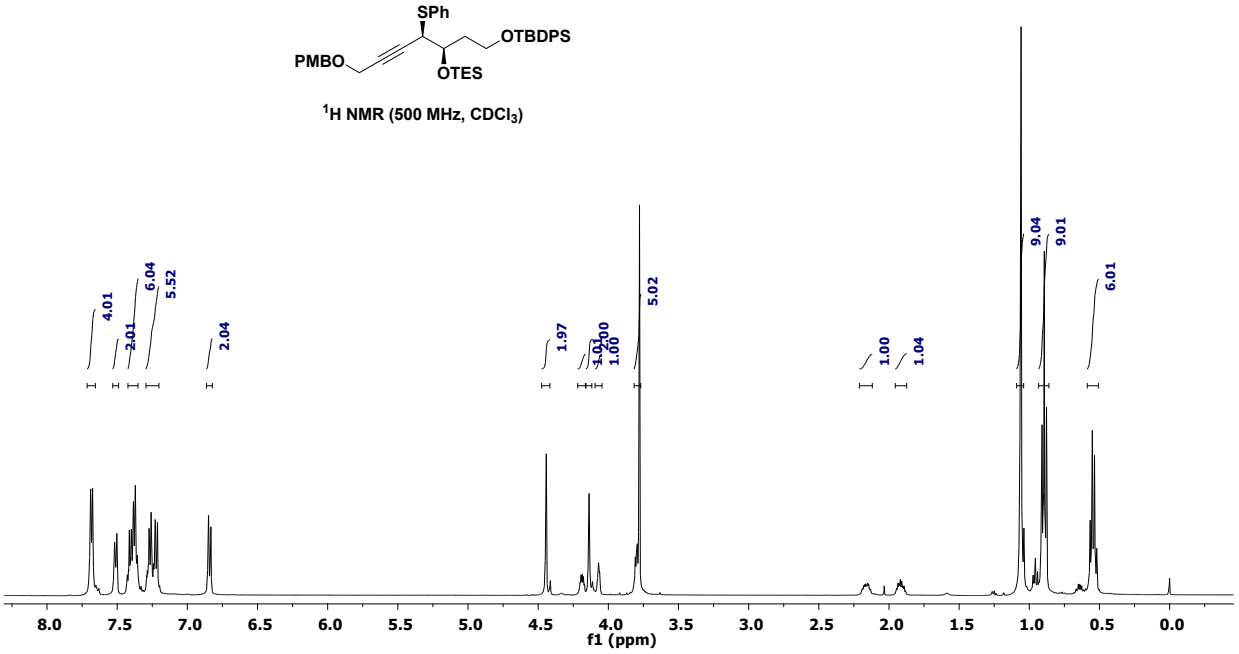

OTBS
HO
OTBDPS
OTBS
^{13}C NMR (101 MHz, CDCl$_3$)
135.51
133.87
129.55
129.54
128.84
127.59
73.70
69.60
66.92
60.37
41.09
26.85
25.83
19.17
18.17
-4.21
-4.34
-4.82
-4.90
145
135
125
115
105
95
85
75
65
55
45
35
25
15
5
0
-5
-10

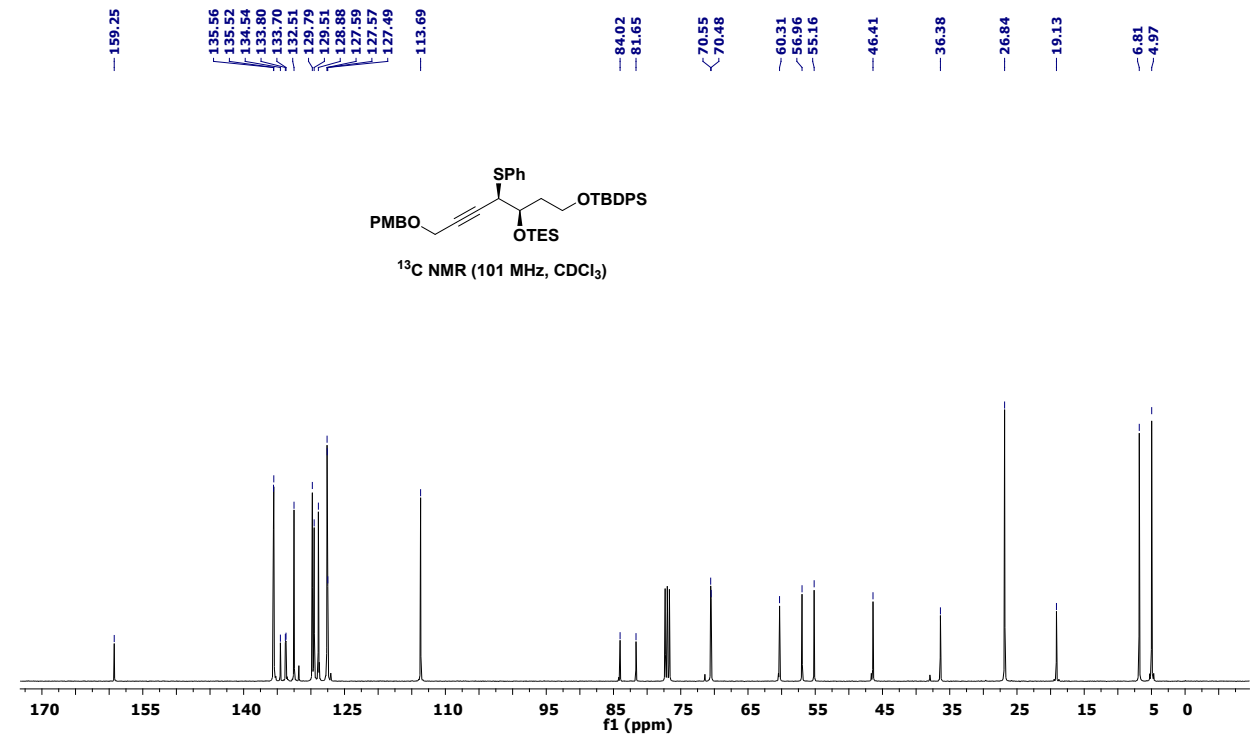
Compound 50:


PhS
OTBDPS
OTES
^{1}H NMR (500 MHz, CDCl$_3$)
4.00
2.06
6.00
2.44
0.95
1.00
2.00
1.00
1.03
9.01
9.01
6.02
f1 (ppm)

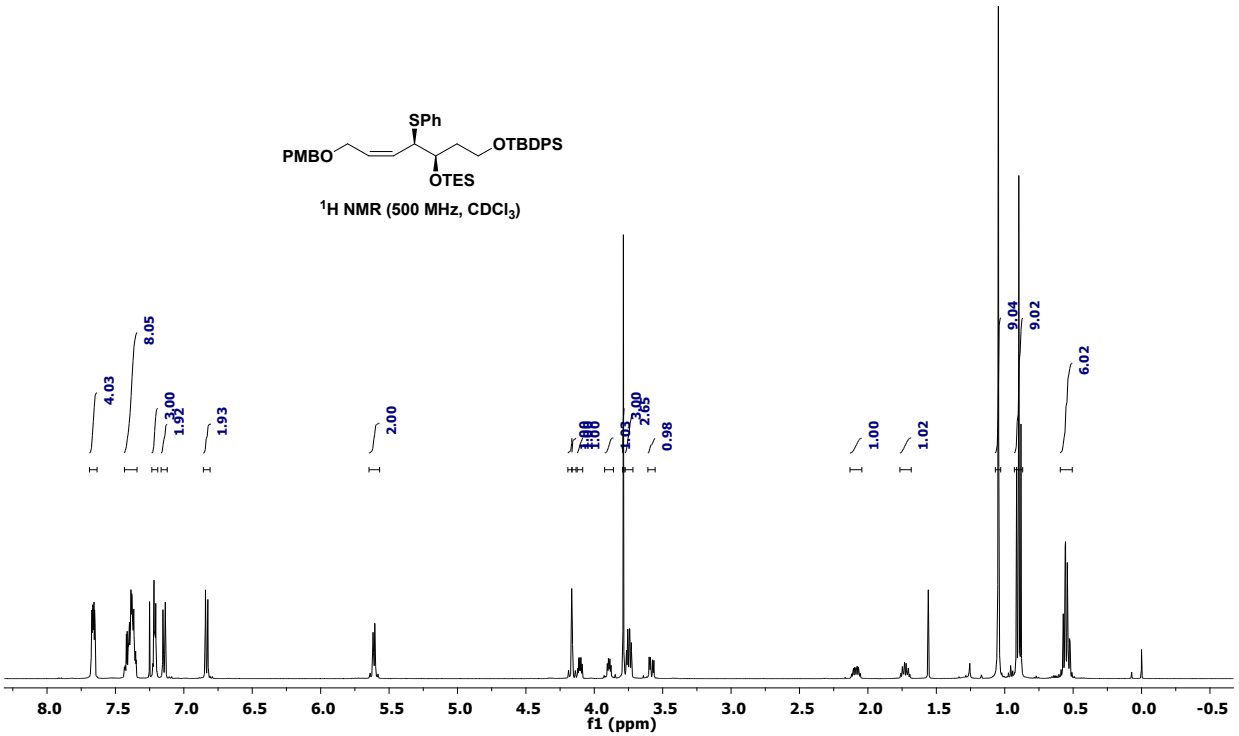

136.96
135.49
135.46
133.71
133.65
129.50
129.33
128.70
127.56
125.76
68.49
60.30
41.30
39.25
26.80
19.05
6.84
4.97
PhS
OTBDPS
OTES
^{13}C NMR (126 MHz, CDCl$_3$)
f1 (ppm)

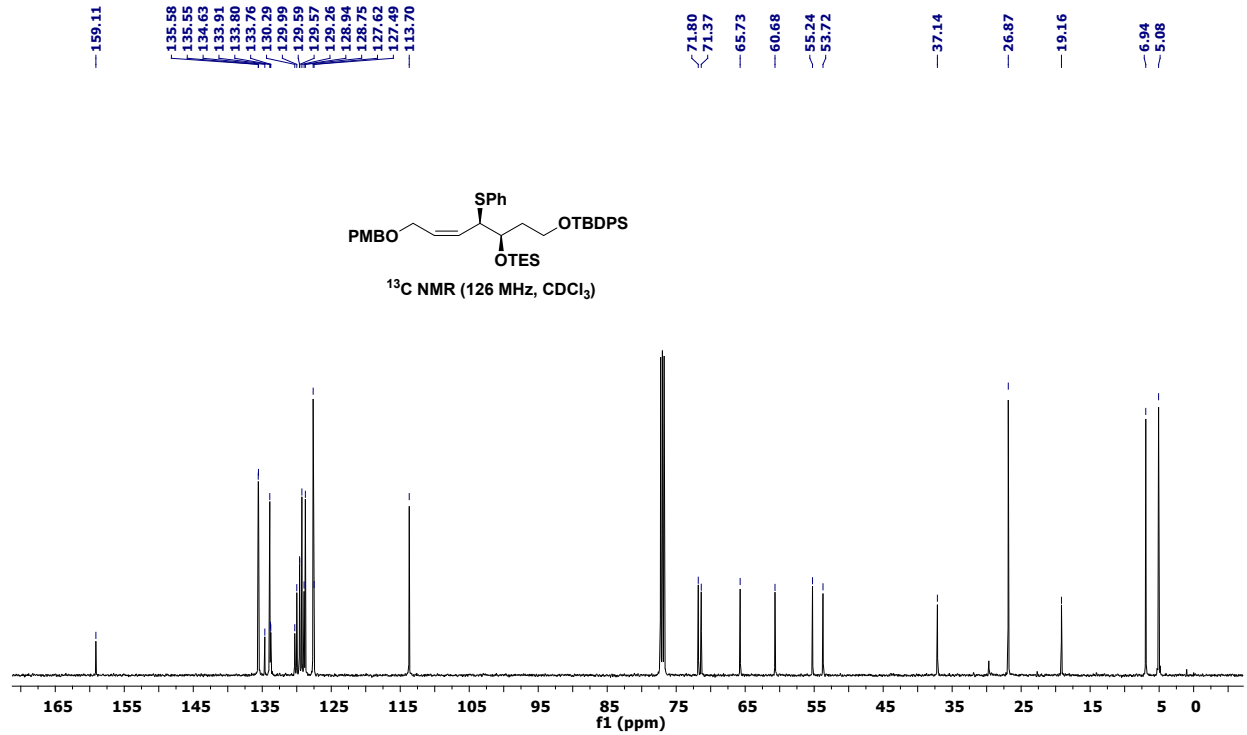
Compound 51:


SPh
OTBDPS
PMBO
OTES
^{1}H NMR (500 MHz, CDCl$_3$)
4.01
2.01
6.04
5.52
2.04
1.97
1.01
2.00
1.00
5.02
1.00
1.04
9.04
9.01
6.01
f1 (ppm)

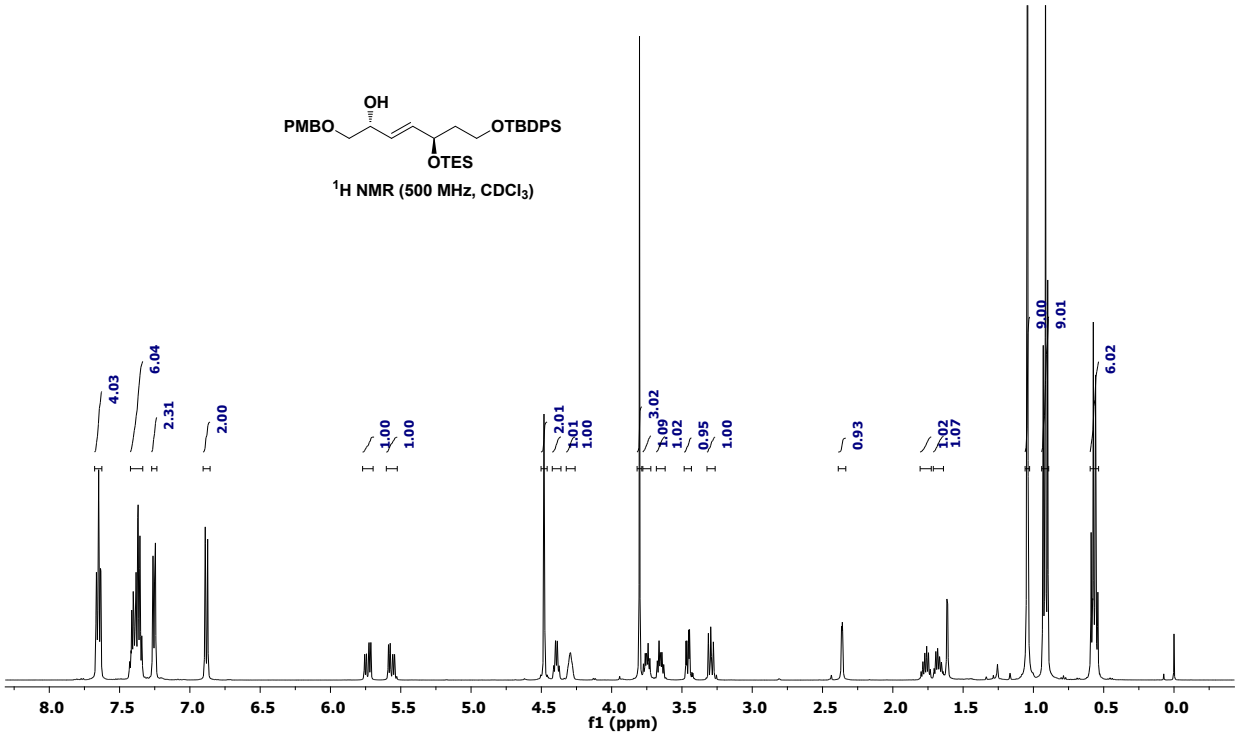

159.25
135.56
135.52
134.54
133.80
133.70
132.51
129.79
129.51
128.88
127.59
127.57
127.49
113.69
84.02
81.65
70.55
70.48
60.31
56.96
55.16
46.41
36.38
26.84
19.13
6.81
4.97
SPh
OTBDPS
PMBO
OTES
^{13}C NMR (101 MHz, CDCl$_3$)
f1 (ppm)

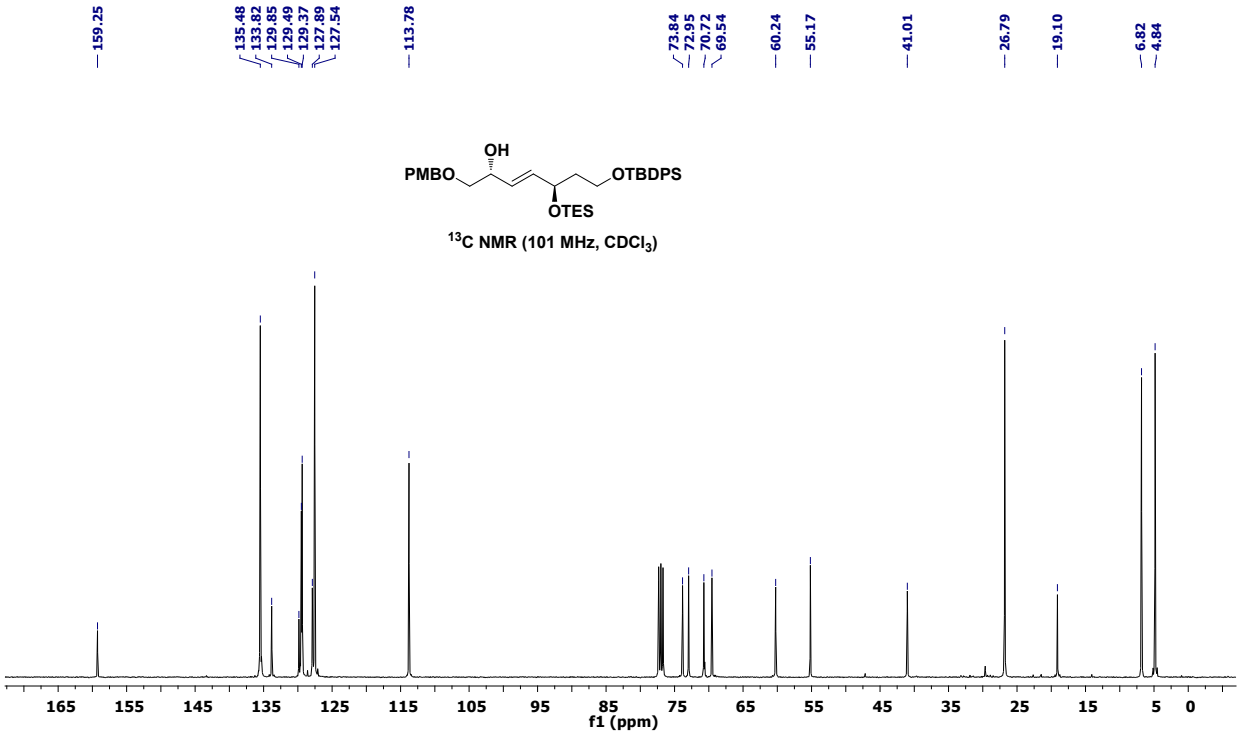
Compound 52:


SPh
PMBO
OTBDPS
OTES
^{1}H NMR (500 MHz, CDCl$_3$)
f1 (ppm)

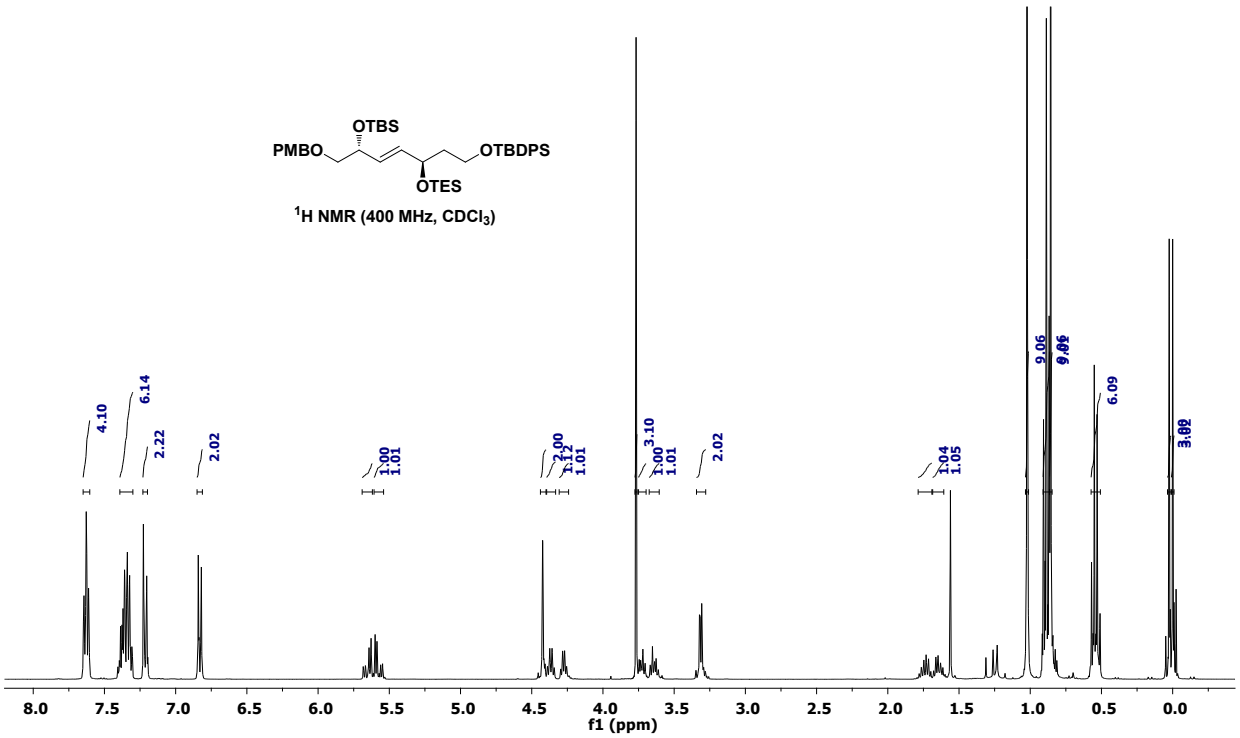

SPh
PMBO
OTBDPS
OTES
^{13}C NMR (126 MHz, CDCl$_3$)
f1 (ppm)

Compound 53:

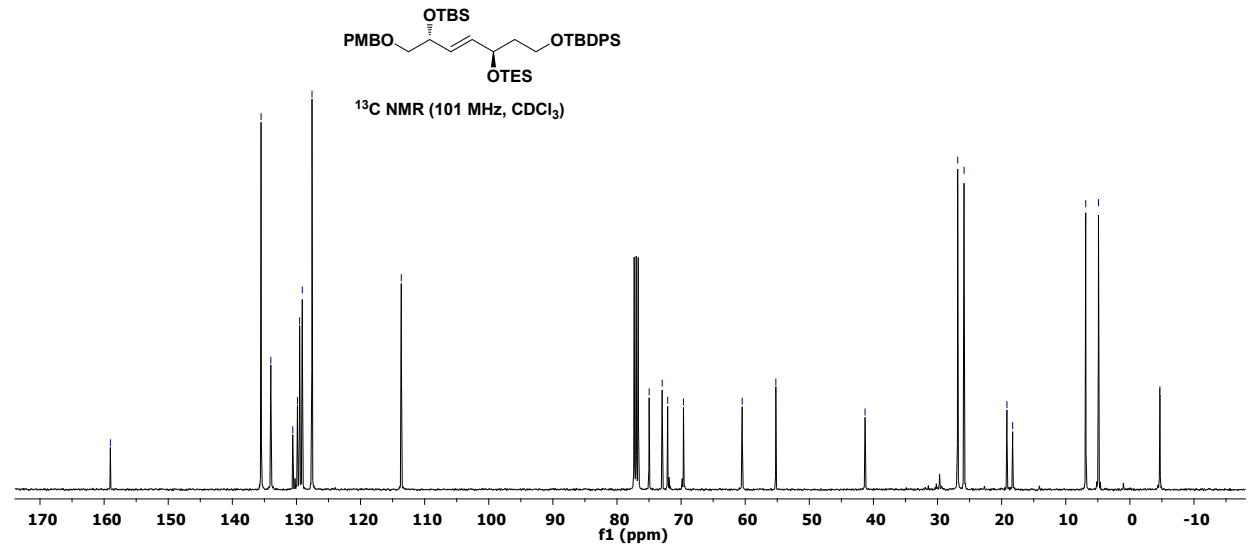

OH
PMBO
OTBDPS
OTES
^{1}H NMR (500 MHz, CDCl$_3$)
4.03
6.04
2.31
2.00
1.00
1.00
2.01
1.01
1.00
3.02
2.09
1.02
0.95
1.00
0.93
1.02
1.07
9.00
9.01
6.02
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


159.25
135.48
133.82
129.85
129.49
129.37
127.89
127.54
113.78
73.84
72.95
70.72
69.54
60.24
55.17
41.01
26.79
19.10
6.82
4.84
OH
PMBO
OTBDPS
OTES
^{13}C NMR (101 MHz, CDCl$_3$)
165 155 145 135 125 115 105 95 85 75 65 55 45 35 25 15 5 0
f1 (ppm)

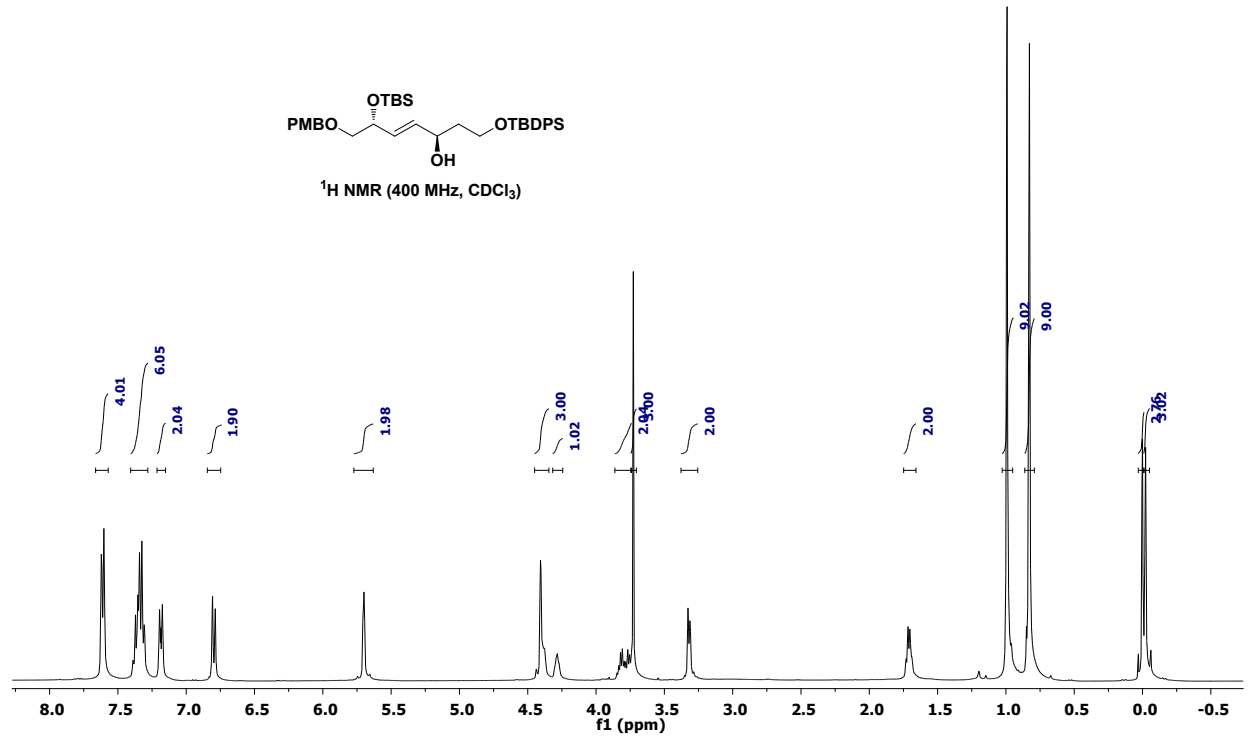
Compound 54:

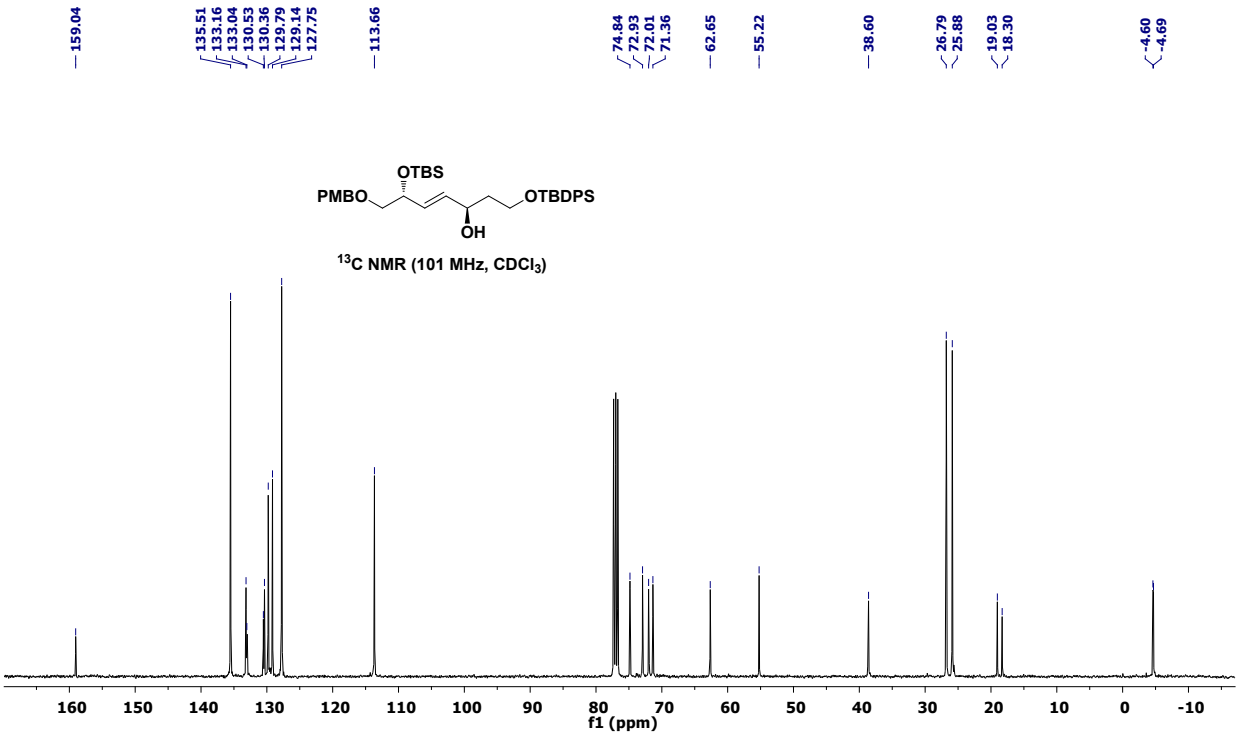

OTBS
PMBO
OTBDPS
OTES
^{1}H NMR (400 MHz, CDCl$_3$)
f1 (ppm)


159.03
135.52
133.99
130.57
129.84
129.50
129.09
127.57
113.65
74.98
72.95
72.09
69.62
60.47
55.22
41.30
26.85
25.86
19.18
18.28
6.88
4.89
-4.65
-4.69

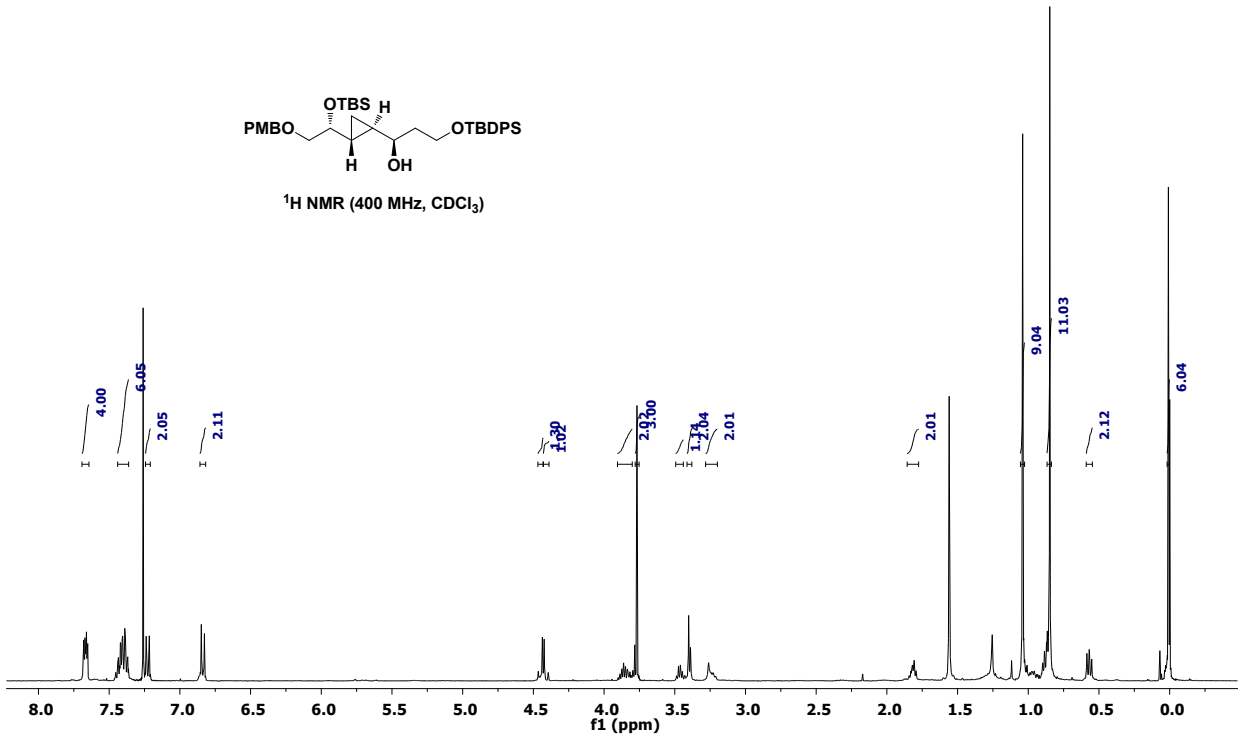

OTBS
PMBO
OTBDPS
OTES
^{13}C NMR (101 MHz, CDCl$_3$)
f1 (ppm)

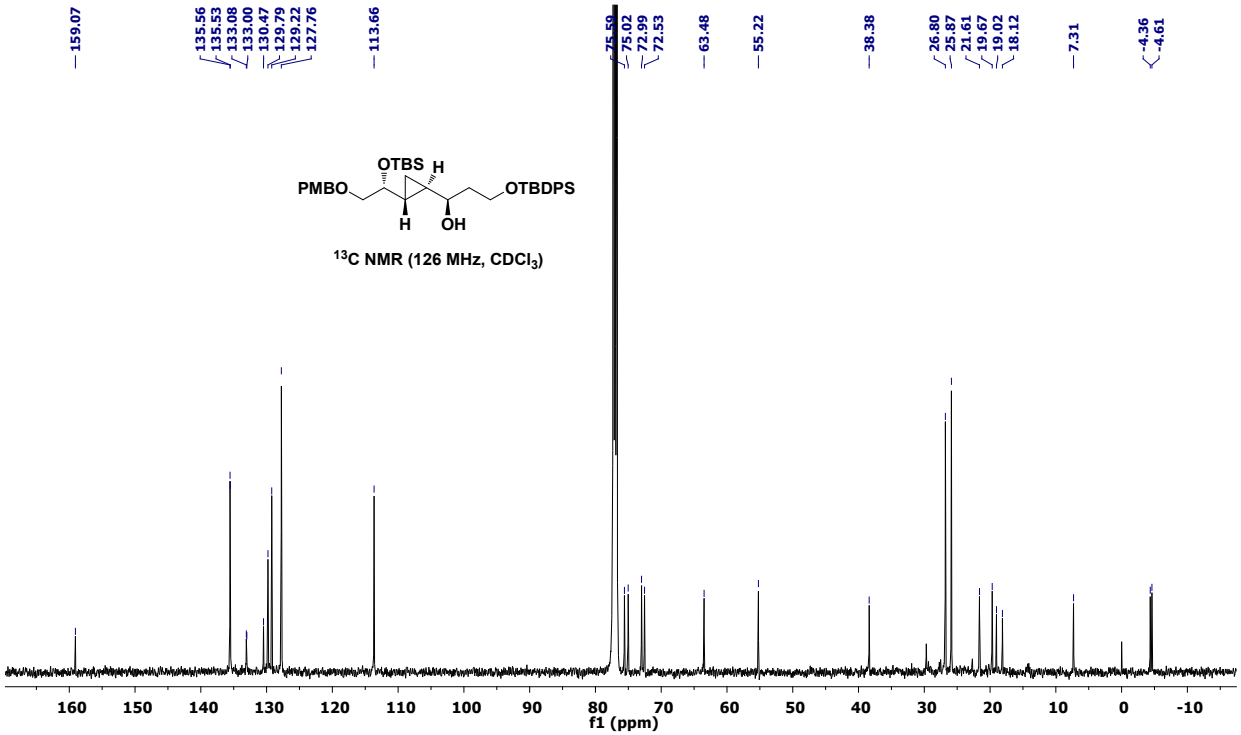
Compound 55:


OTBS
PMBO
OTBDPS
OH
^{1}H NMR (400 MHz, CDCl$_3$)
4.01
6.05
2.04
1.90
1.98
3.00
1.02
2.00
2.00
9.02
9.00
f1 (ppm)

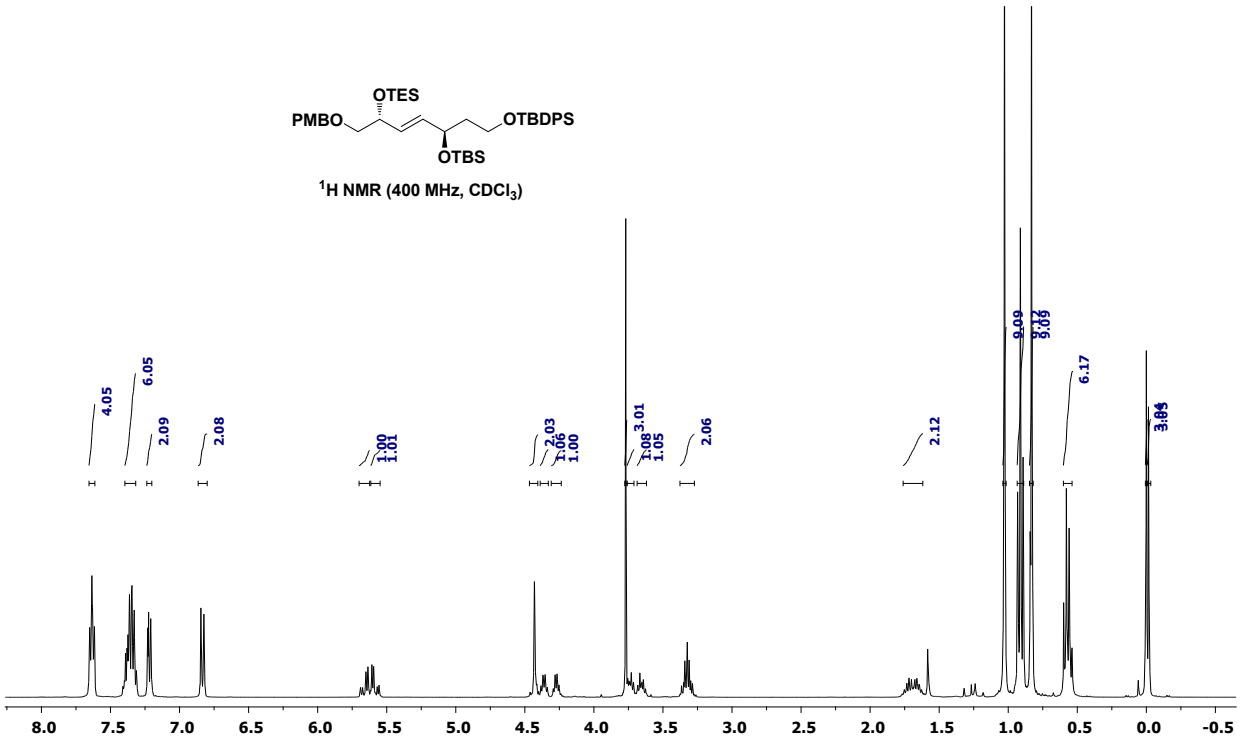

159.04
135.51
133.16
133.04
130.53
130.36
129.79
129.14
127.75
113.66
74.84
72.93
72.01
71.36
62.65
55.22
38.60
26.79
25.88
19.03
18.30
-4.60
-4.69
OTBS
PMBO
OTBDPS
OH
^{13}C NMR (101 MHz, CDCl$_3$)
f1 (ppm)

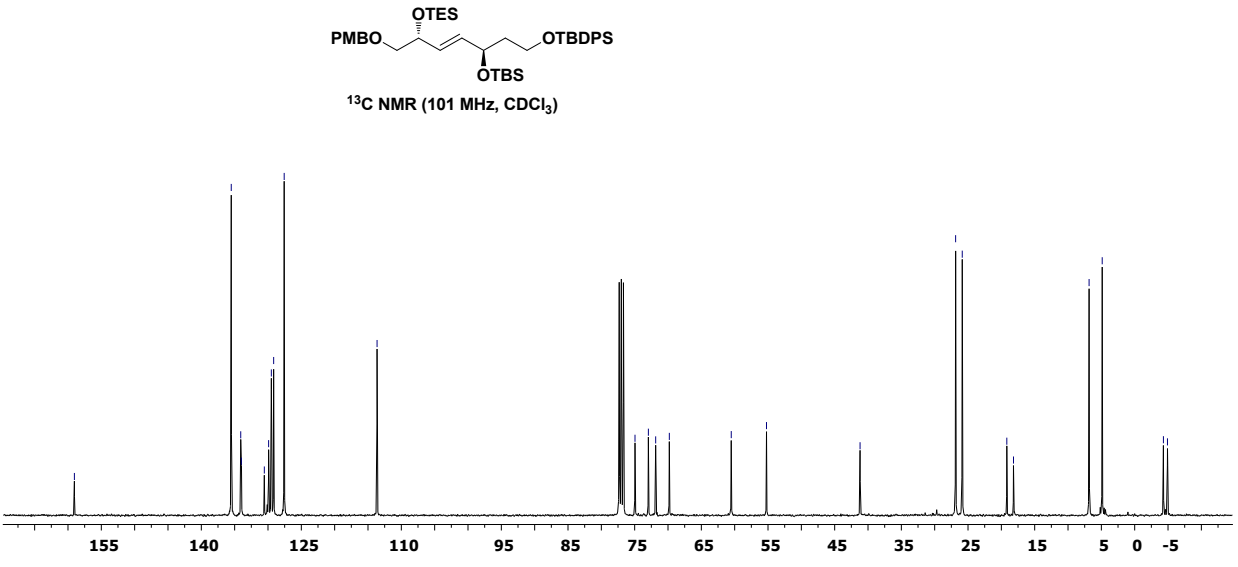
Compound 56:


OTBS H
PMBO
OTBDPS
H
OH
^{1}H NMR (400 MHz, CDCl$_3$)
4.00
6.05
2.05
2.11
1.30
1.02
2.02
2.00
1.14
1.04
2.01
2.01
9.04
11.03
2.12
6.04
f1 (ppm)

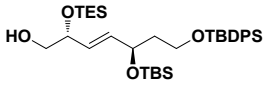

159.07
135.56
135.53
133.08
133.00
130.47
129.79
129.22
127.76
113.66
75.59
75.02
72.99
72.53
63.48
55.22
38.38
26.80
25.87
21.61
19.67
19.02
18.12
7.31
-4.36
-4.61
OTBS H
PMBO
OTBDPS
H
OH
^{13}C NMR (126 MHz, CDCl$_3$)
f1 (ppm)

Compound VII:

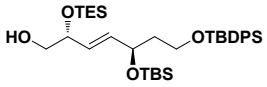




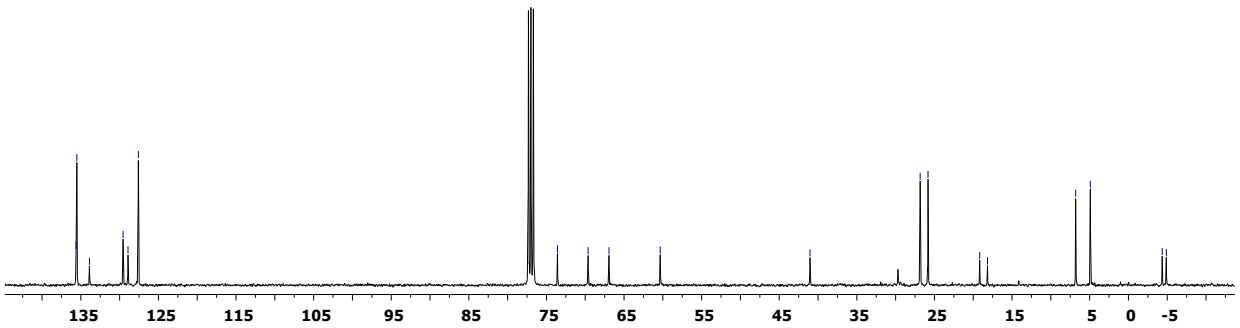
Compound VIII:



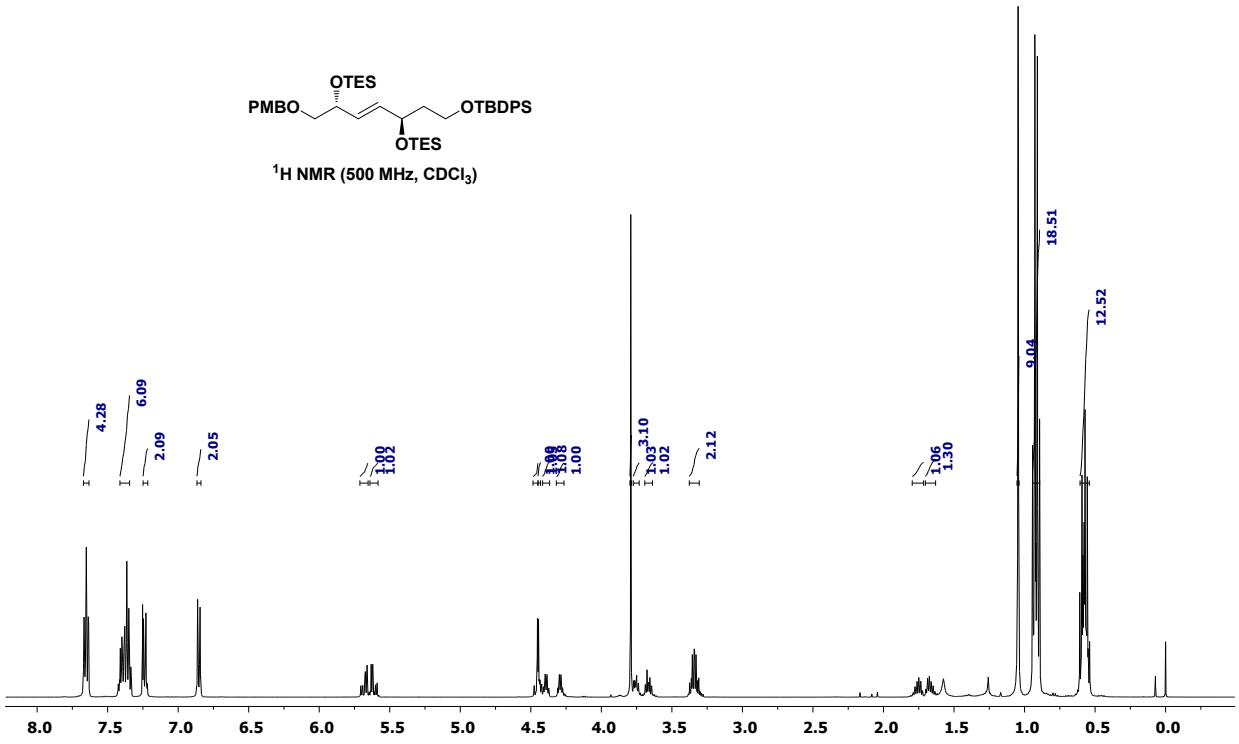
^{1}H NMR (400 MHz, $CDCl_3$)

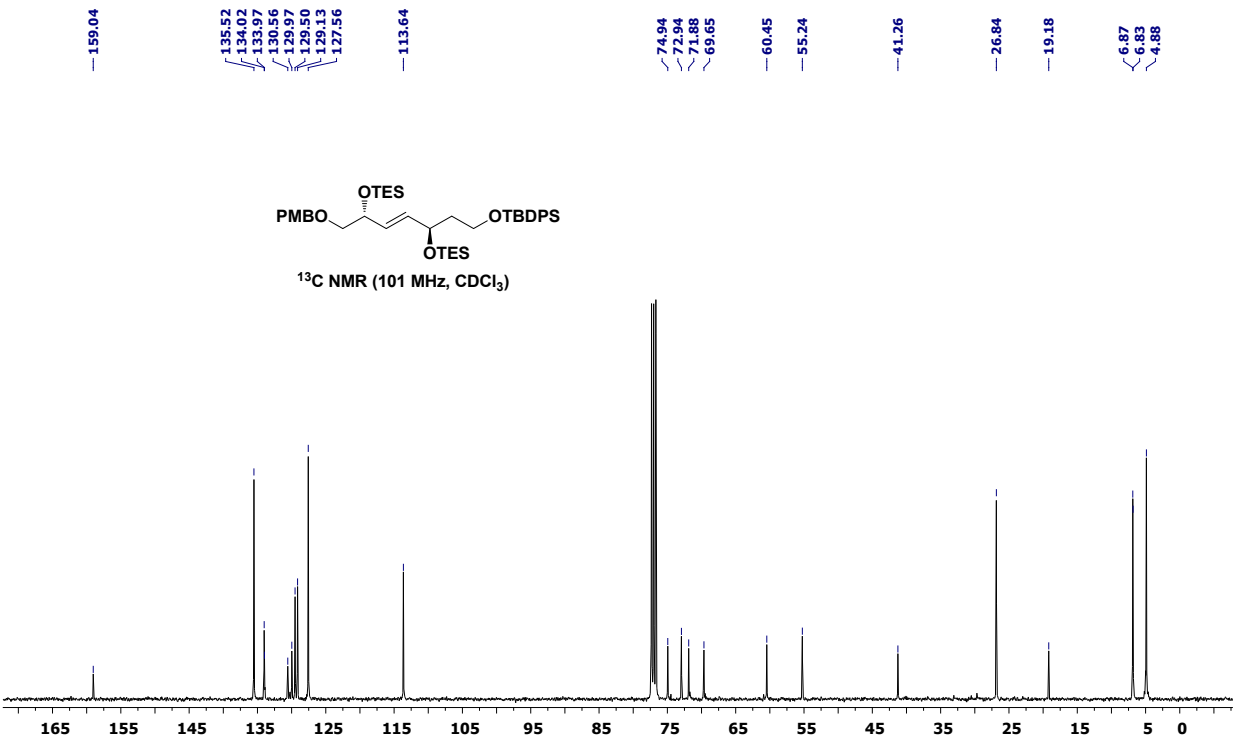


^{13}C NMR (101 MHz, $CDCl_3$)

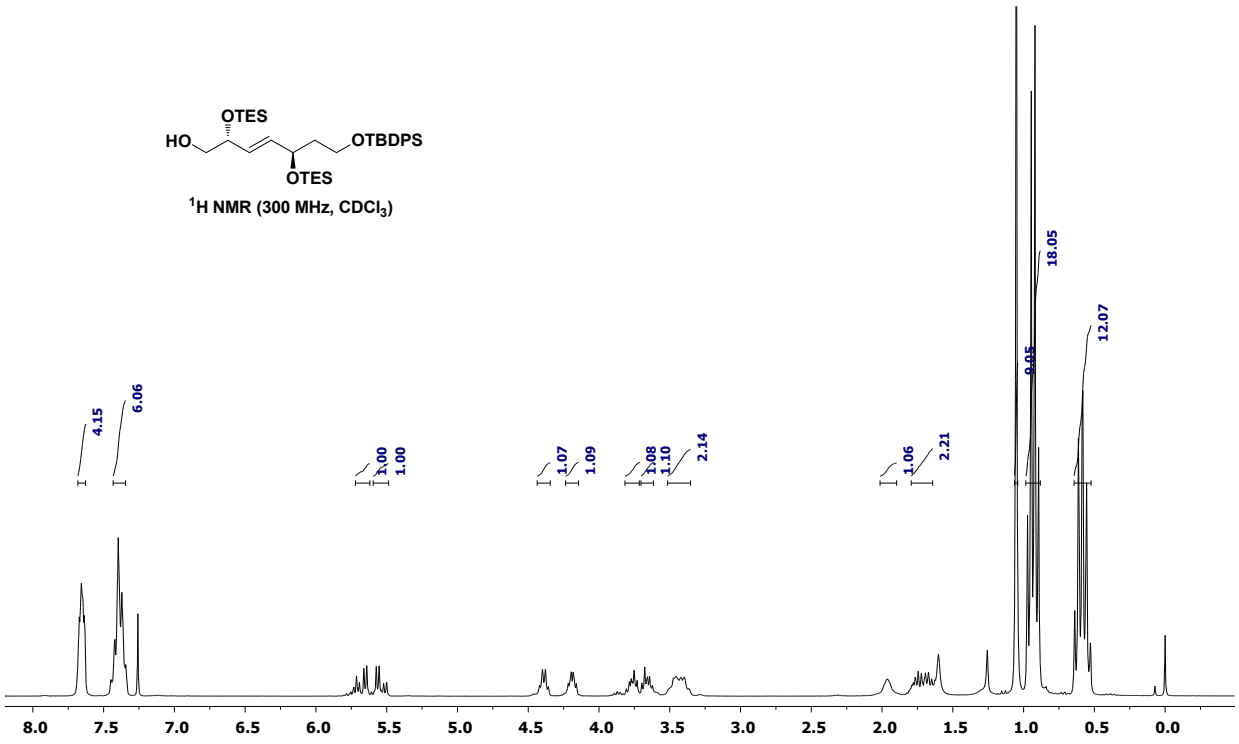


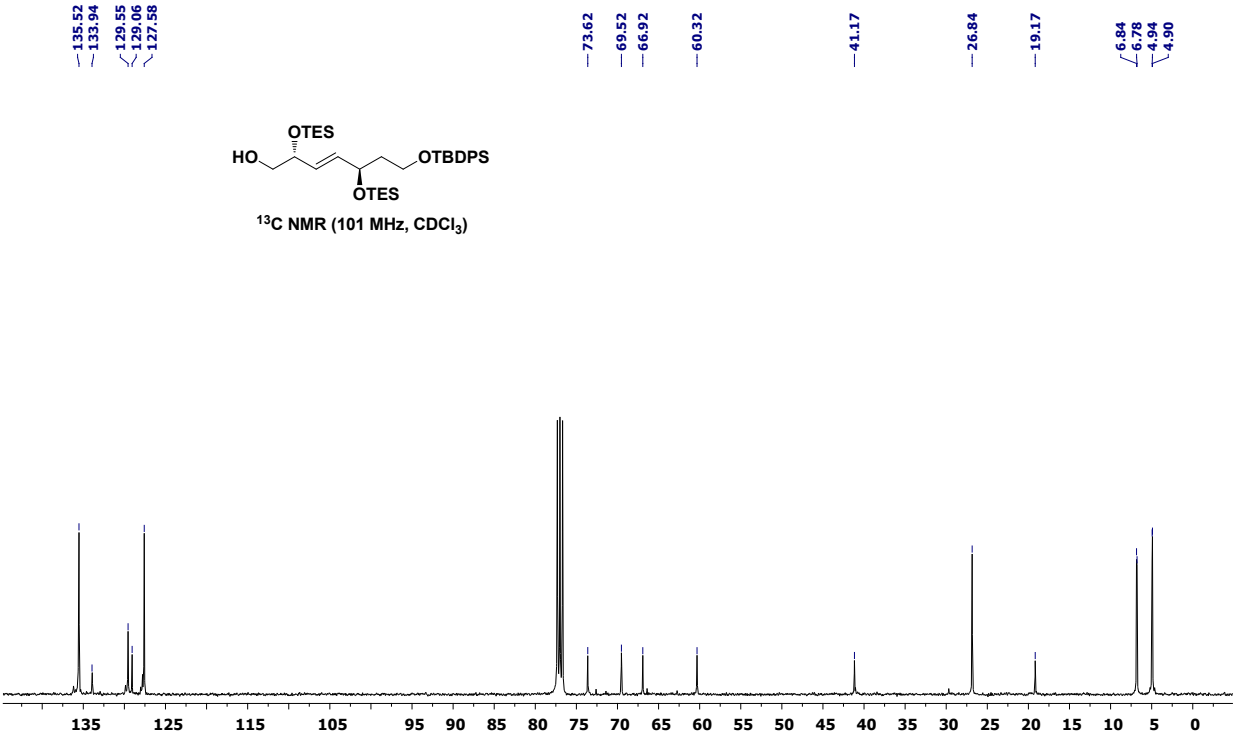
Compound IX:


OTES
PMBO
OTBDPS
OTES
^{1}H NMR (500 MHz, CDCl$_3$)
4.28
6.09
2.09
2.05
1.00
1.02
3.10
1.03
1.02
2.12
1.06
1.30
9.04
18.51
12.52
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


OTES
PMBO
OTBDPS
OTES
^{13}C NMR (101 MHz, CDCl$_3$)
159.04
135.52
134.02
133.97
130.56
129.97
129.50
129.13
127.56
113.64
74.94
72.94
71.88
69.65
60.45
55.24
41.26
26.84
19.18
6.87
6.83
4.88
165
155
145
135
125
115
105
95
85
75
65
55
45
35
25
15
5
0

Compound X:


OTES
HO
OTBDPS
OTES
^{1}H NMR (300 MHz, CDCl$_3$)
4.15
6.06
1.00
1.00
1.07
1.09
1.08
1.10
2.14
1.06
2.21
9.05
18.05
12.07
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


135.52
133.94
129.55
129.06
127.58
73.62
69.52
66.92
60.32
41.17
26.84
19.17
6.84
6.78
4.94
4.90
OTES
HO
OTBDPS
OTES
^{13}C NMR (101 MHz, CDCl$_3$)
135
125
115
105
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0