Supplemental Material for

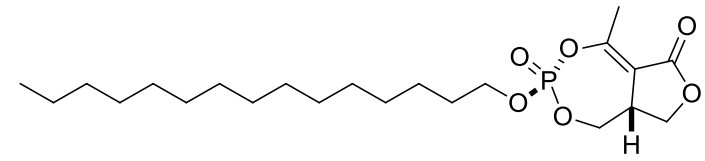
Rat Hormone Sensitive Lipase Inhibition by Cyclipostins and Their Analogs

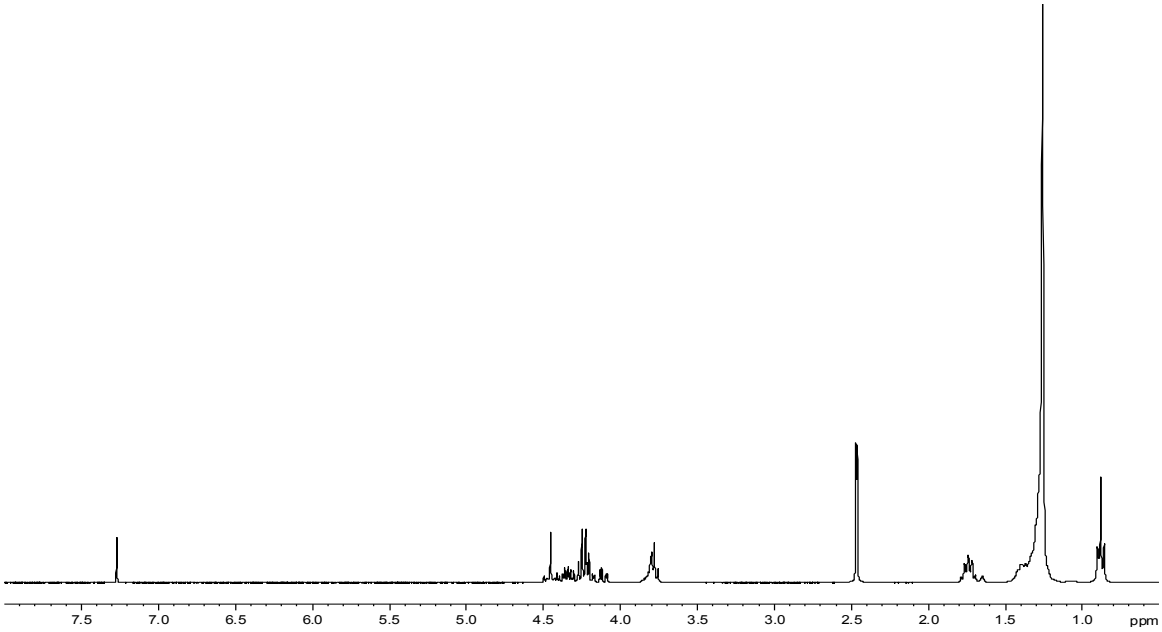
Elena Vasilieva, Supratik Dutta, Raj Malla, Benjamin Martin, Christopher D. Spilling, and Cynthia M. Dupureur*

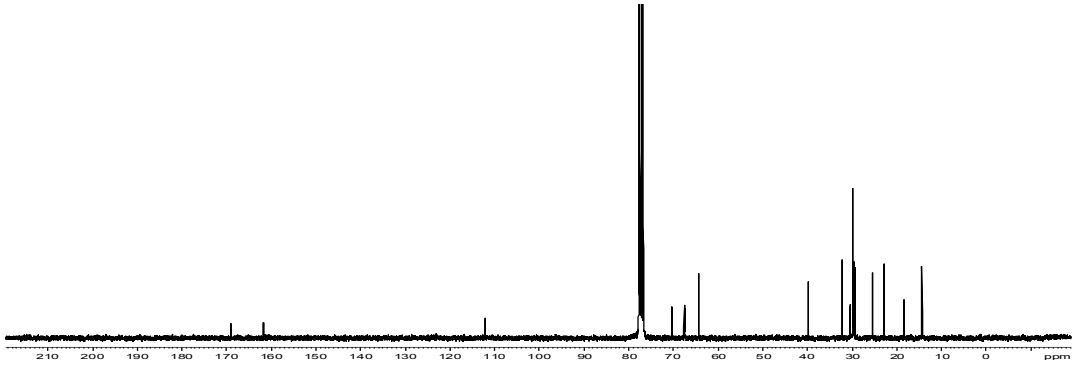
Department of Chemistry & Biochemistry and the Center for Nanoscience, University of Missouri St. Louis, St. Louis, MO 63121

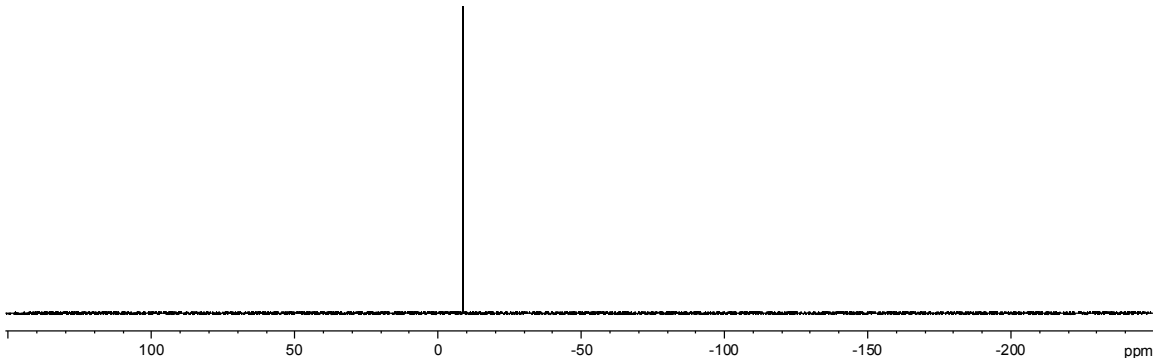
Supporting Information

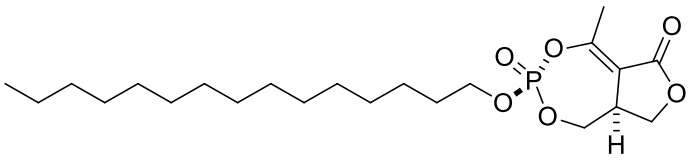
Table of contents

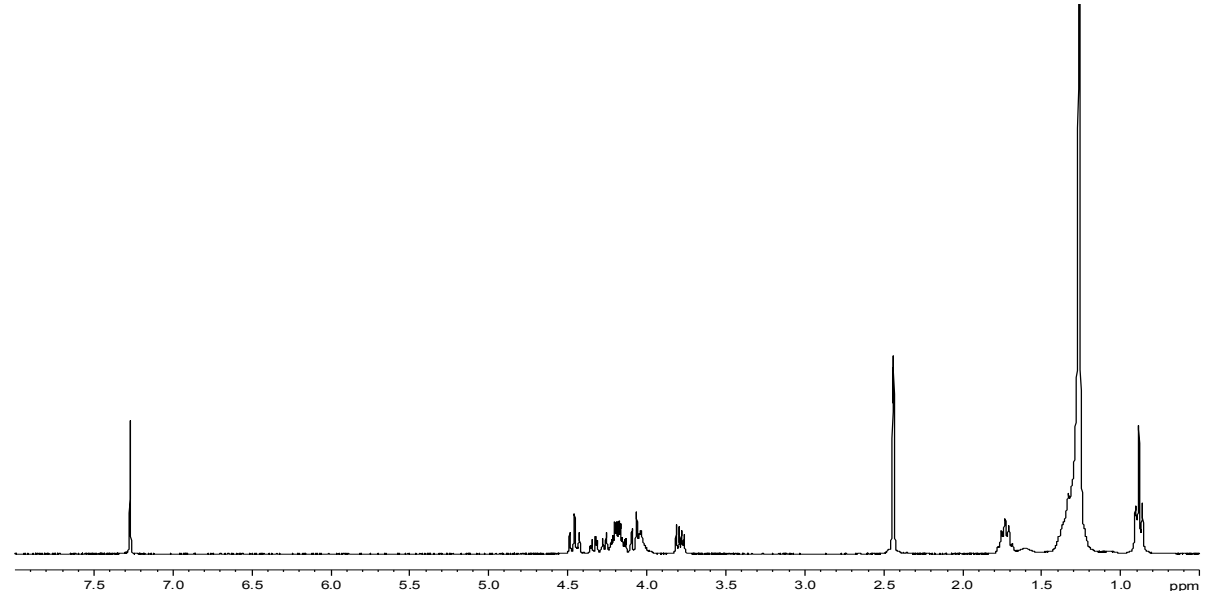

O
O
O
P
O
O
O
O
H

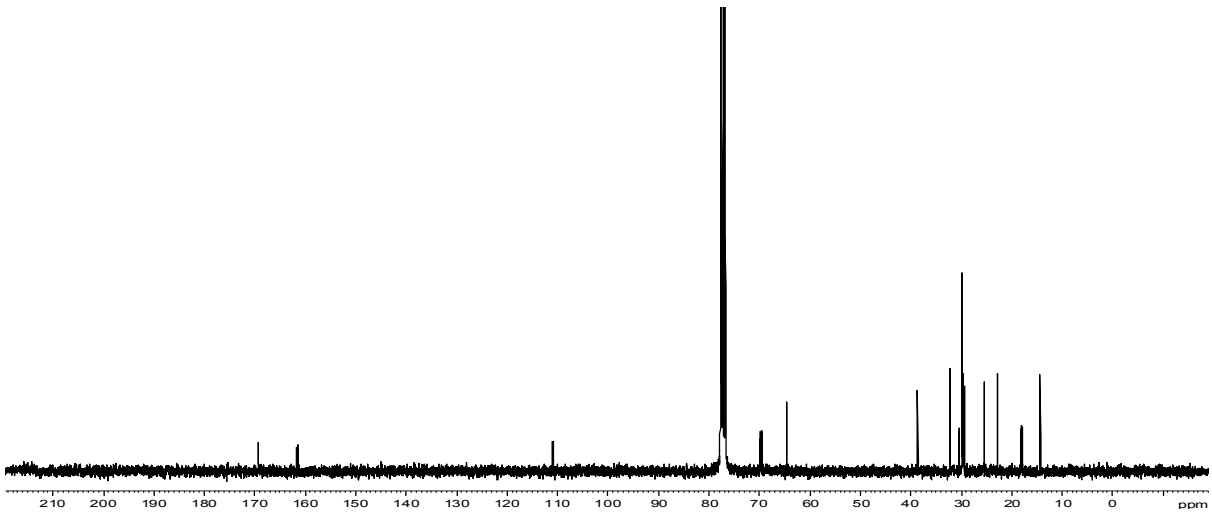

7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

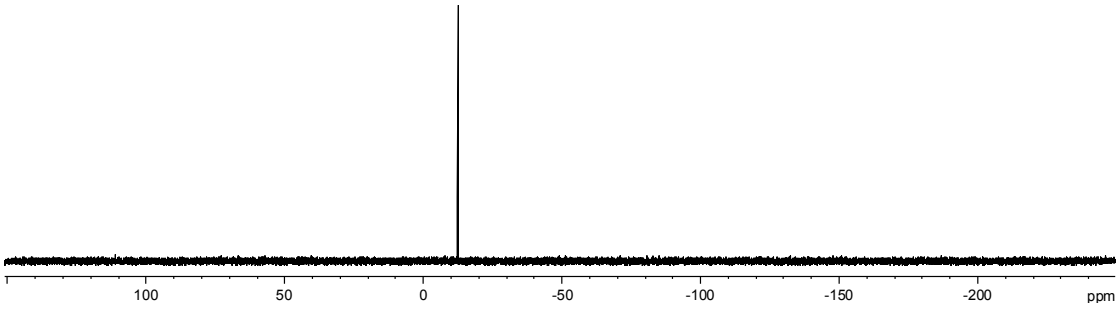

210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

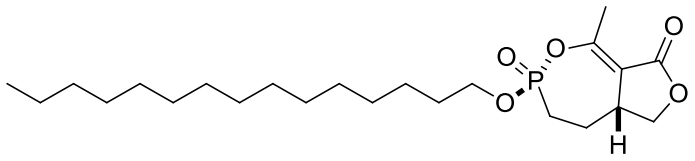

100
50
0
-50
-100
-150
-200
ppm

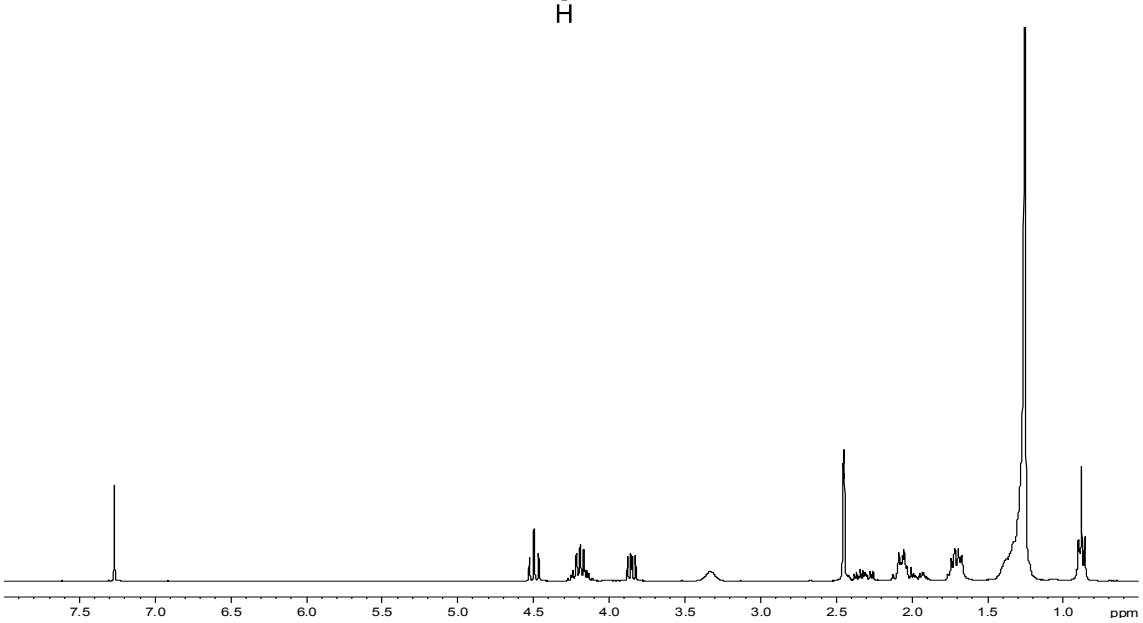

O
O
O
P
O
O
O
O
H

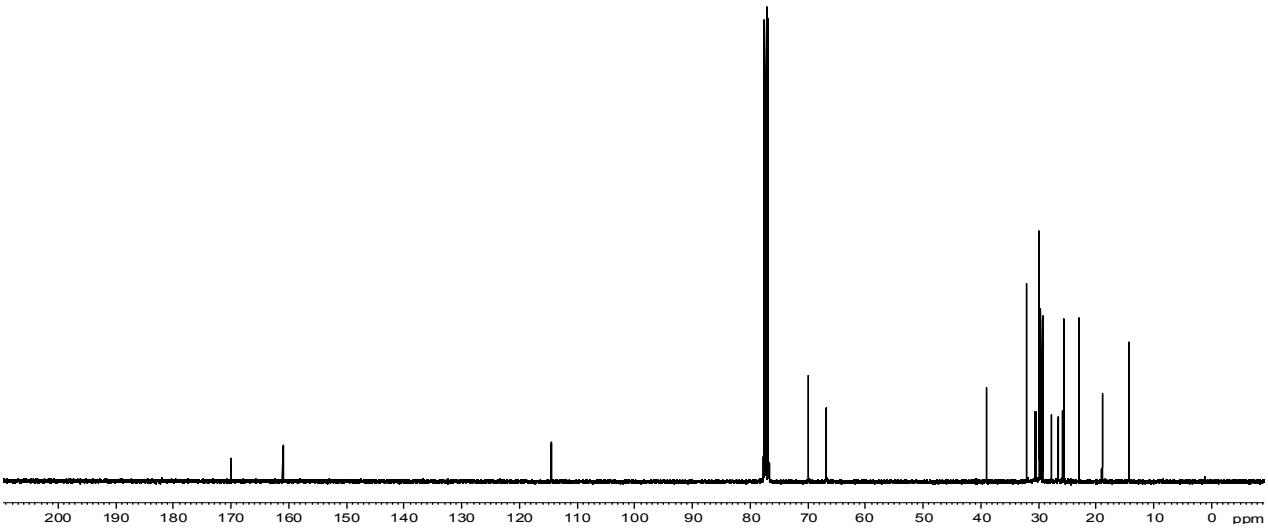

7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

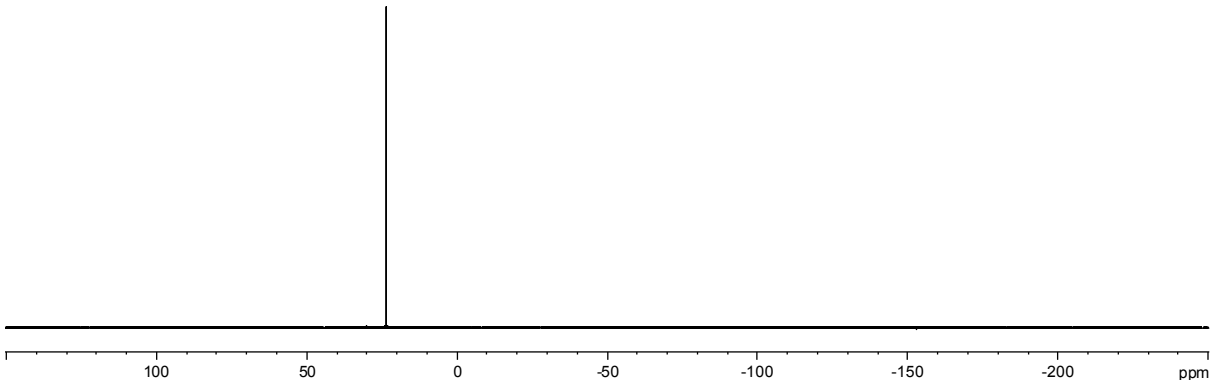

210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

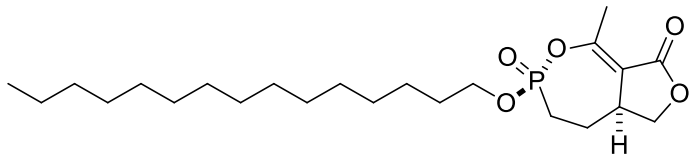

100
50
0
-50
-100
-150
-200
ppm

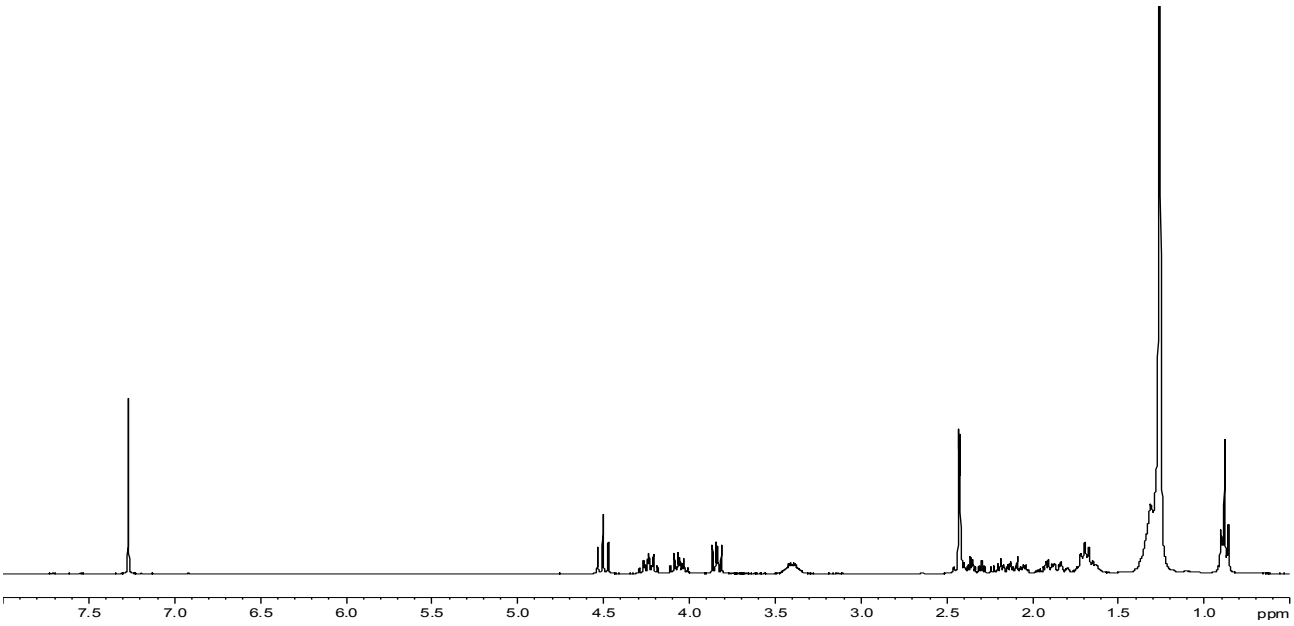

O
O
O
P
O
O
H

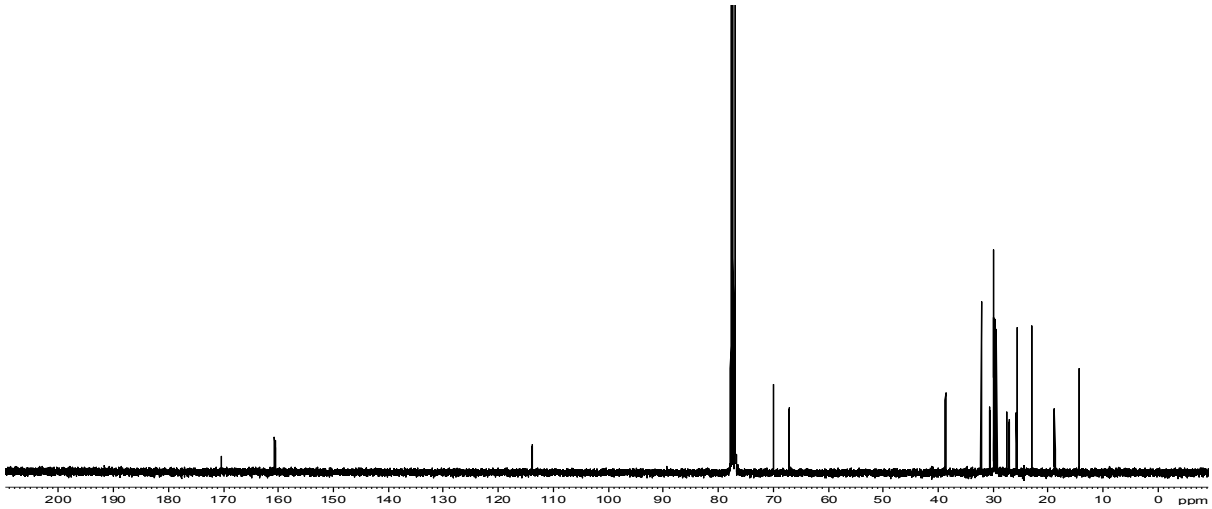

7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

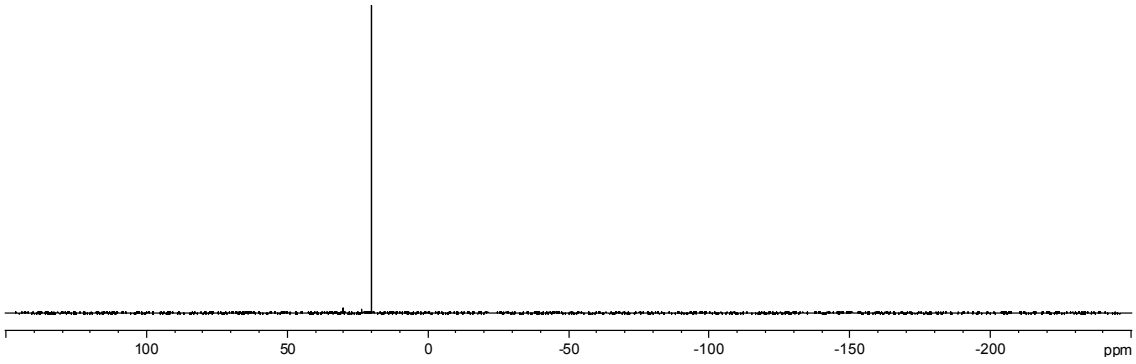

200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

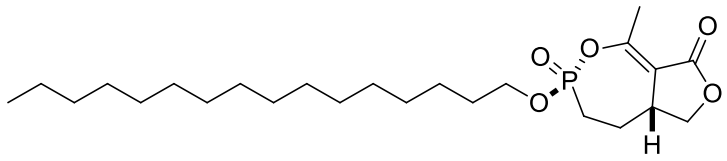

100
50
0
-50
-100
-150
-200
ppm

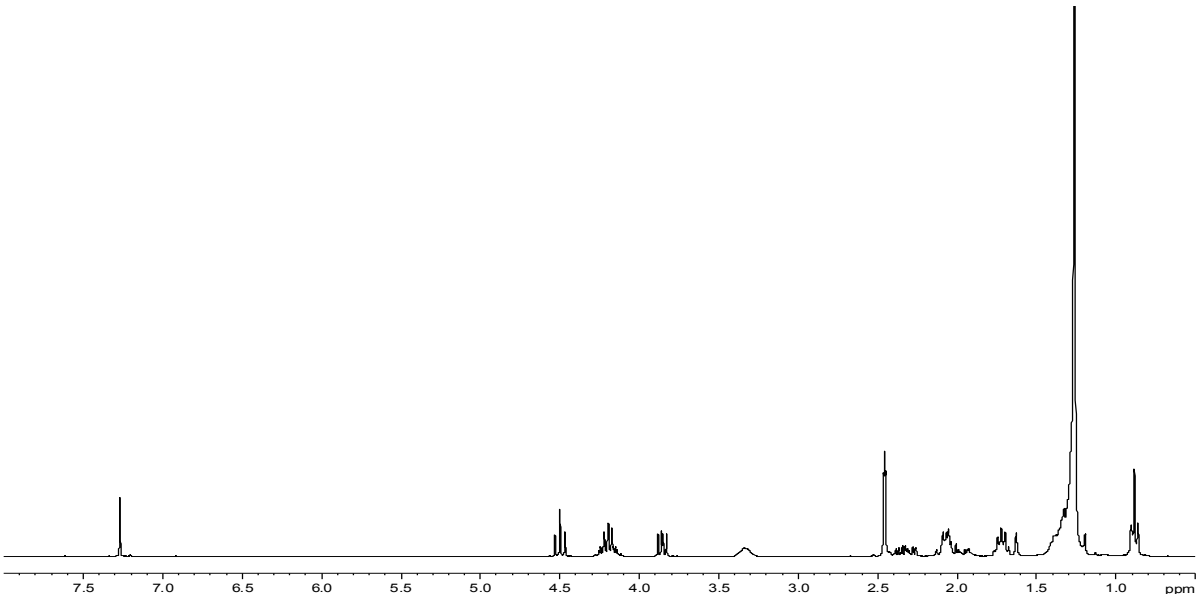

O
O
O
P
O
O
H

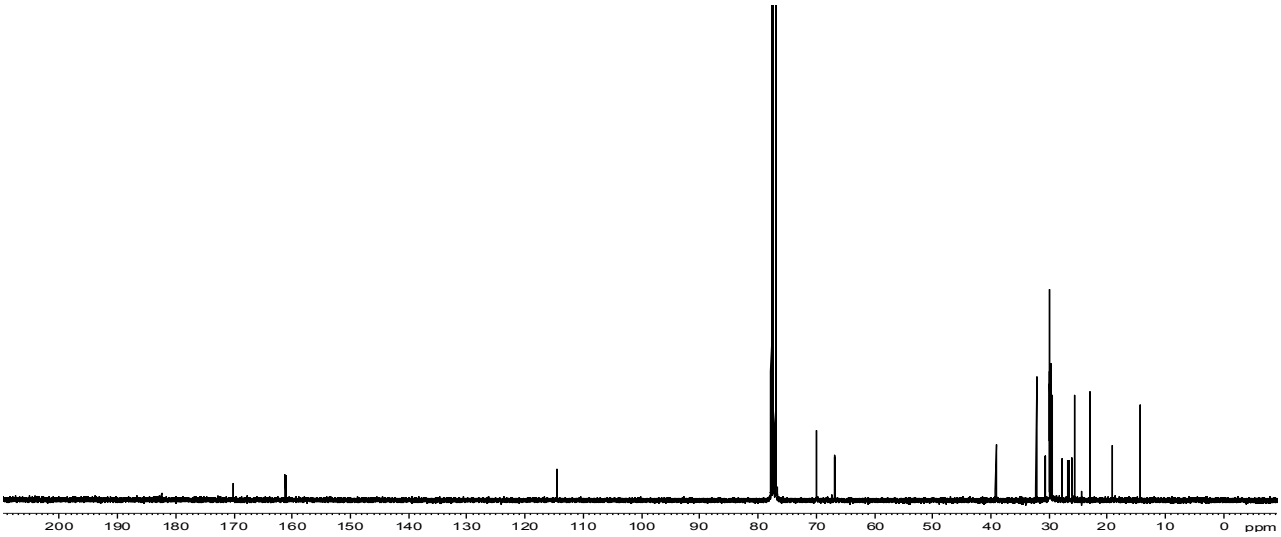

7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

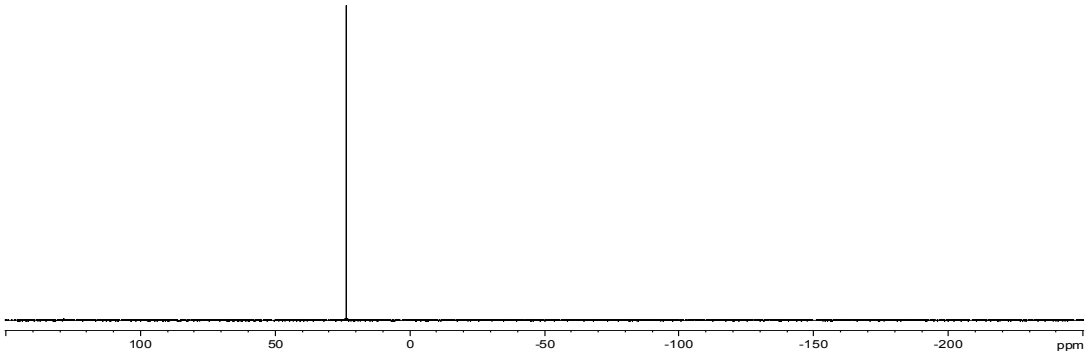

200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

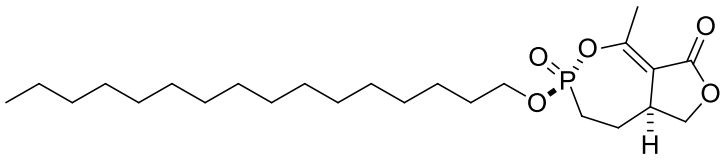

100
50
0
-50
-100
-150
-200
ppm

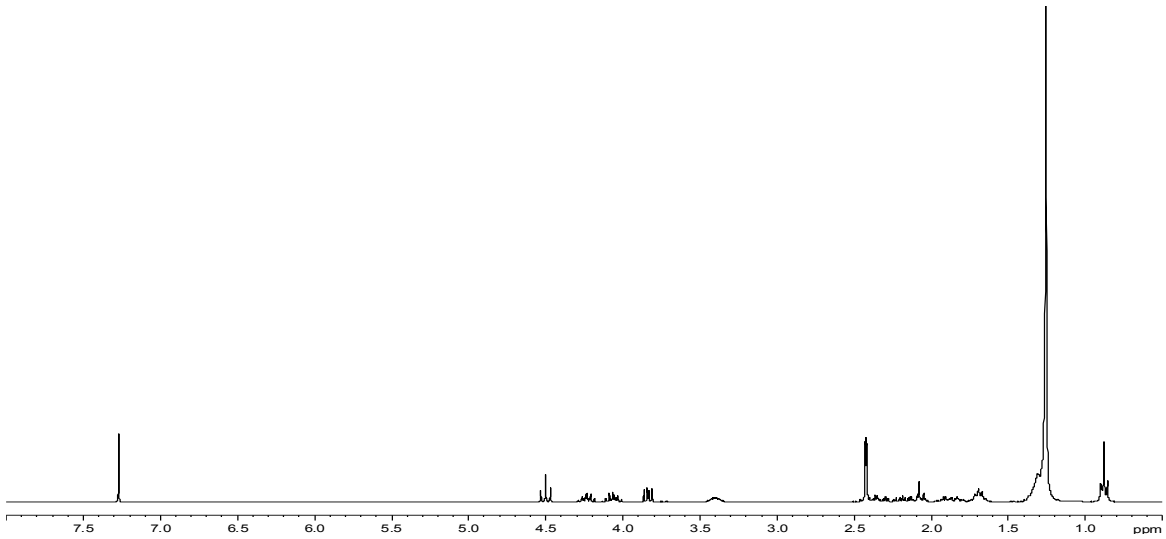

O
O
O
P
O
O
O
H

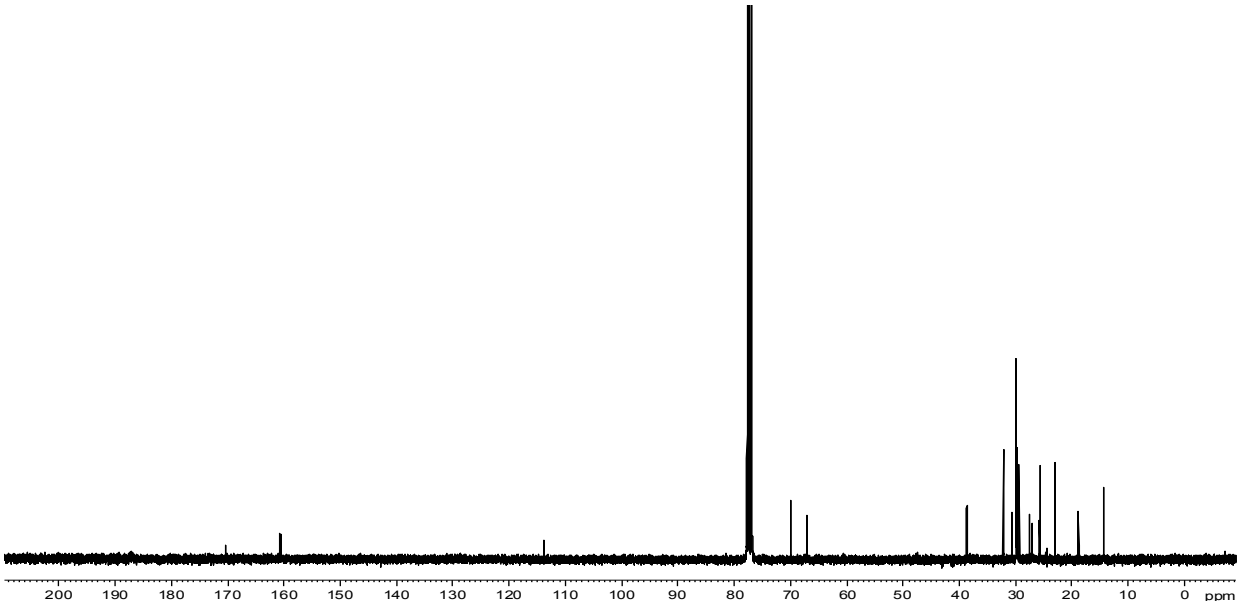

7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

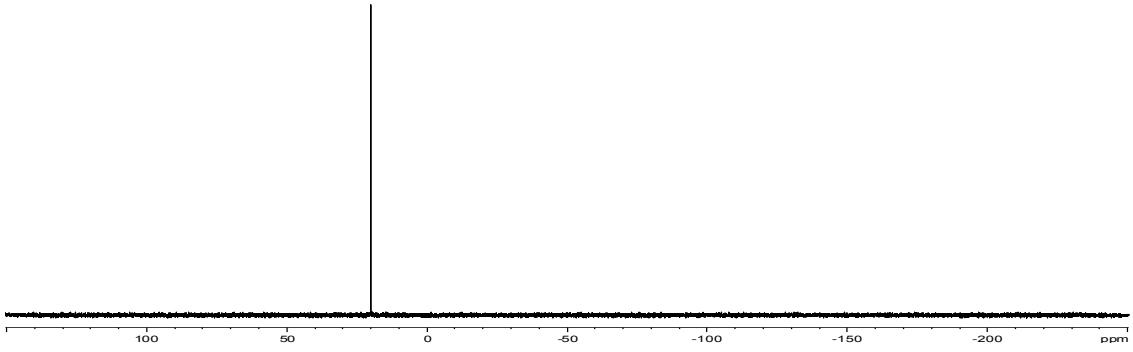

200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

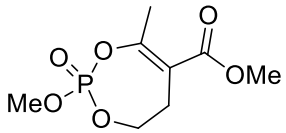

100
50
0
-50
-100
-150
-200
ppm


O
O
O
P
O
O
O
H

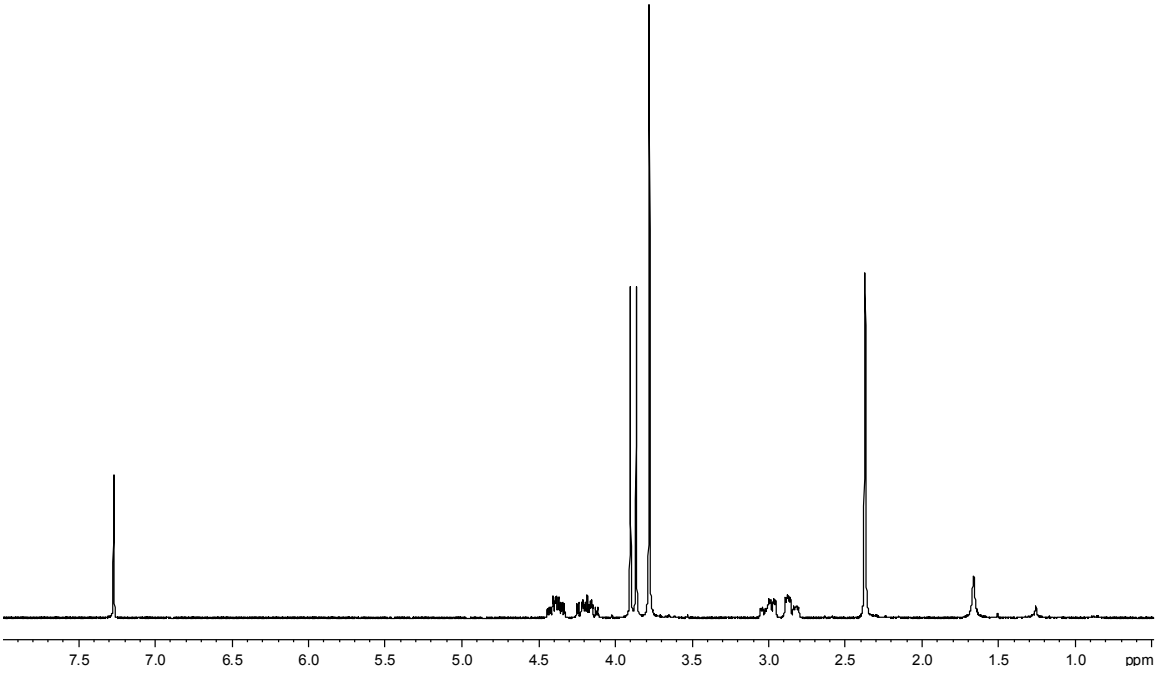

7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

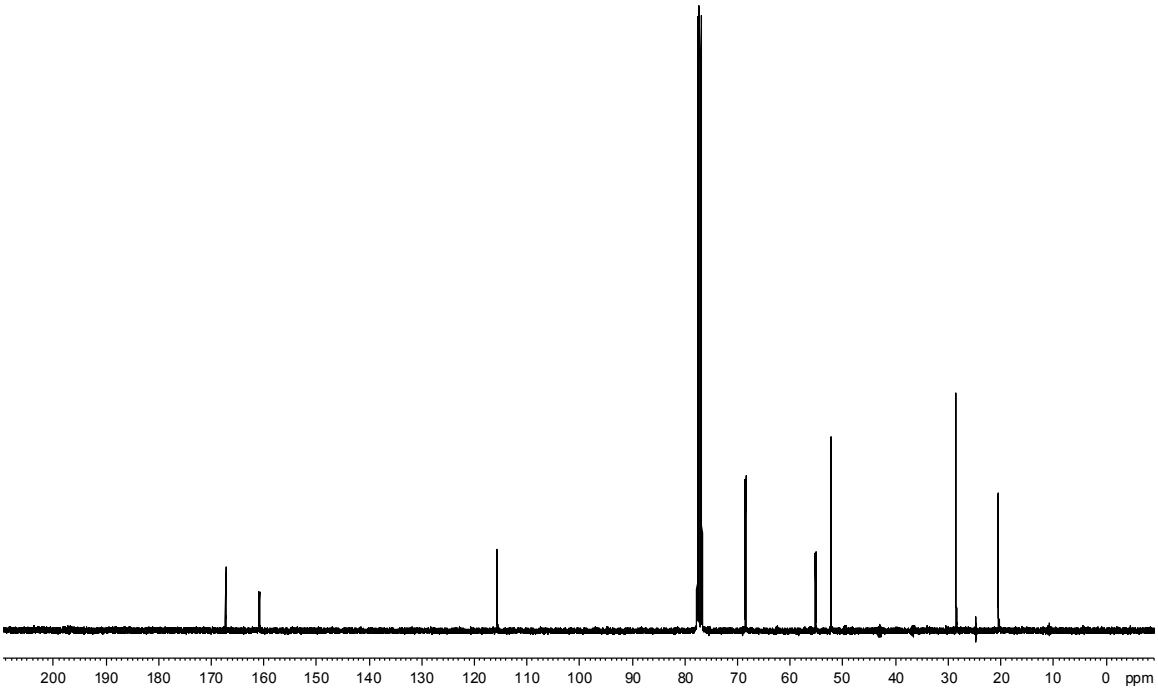

200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

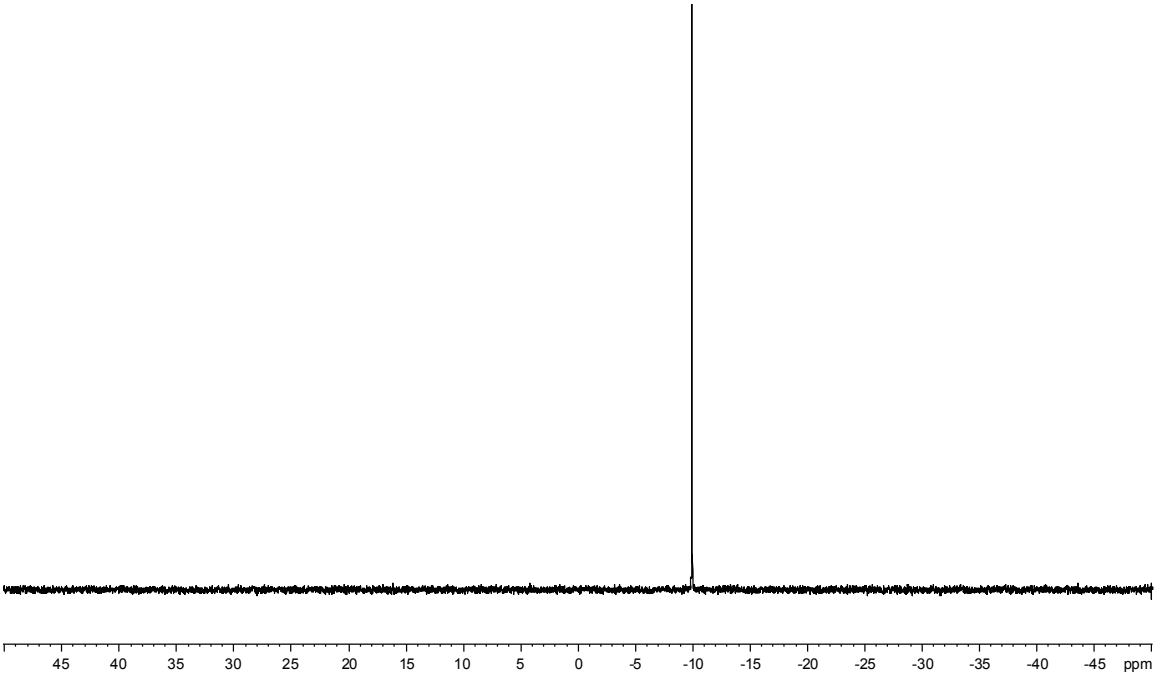

100
50
0
-50
-100
-150
-200
ppm

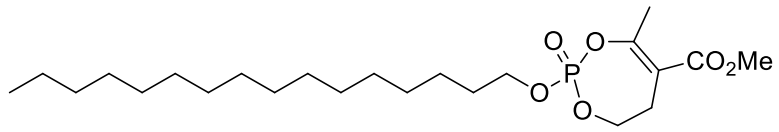


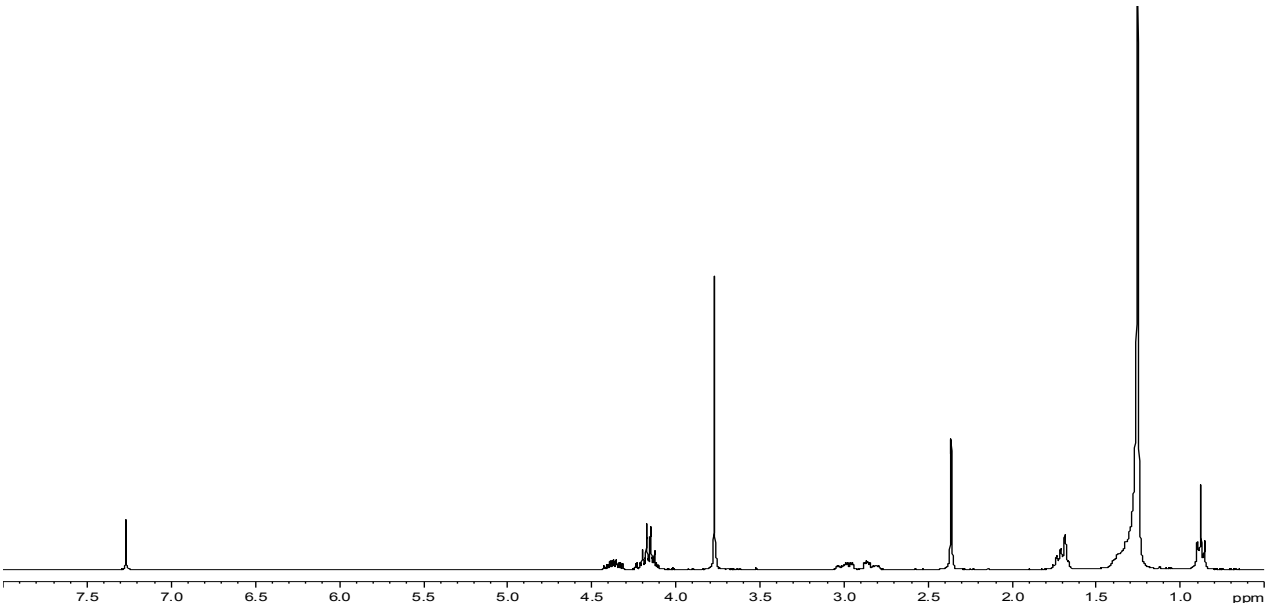
methyl 2-methoxy-4-methyl-6,7-dihydro-1,3,2-dioxaphosphepine-5-carboxylate 2-oxide (YY). IR (neat, NaCl) 2960, 2917, 1718, 1652 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.39 (1H, m), 4.18 (1H, m), 3.88 (3H, d, J_{HP} = 11.5 Hz), 3.78 (3H, s), 3.00 (1H, ddd, J_{HH} = 15.7, 9.4, 3.7 Hz), 2.85 (1H, ddd, J_{HH} = 15.8, 6.4, 3.4 Hz); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 167.2 (d, J_{CP} = 2.0 Hz), 161.0 (d, J_{CP} = 9.6 Hz), 115.7 (d, J_{CP} = 3.5 Hz), 68.5 (d, J_{CP} = 6.6 Hz), 55.1 (d, $J_{C,P}$ = 5.5 Hz), 52.2, 28.4, 20.4 (d, $J_{C,P}$ = 4.1 Hz), $^{31}P\{^{1}H$ NMR (121.4 MHz, $CDCl_3$) δ -9.94; HRMS (FAB, NBA, MH^+) calcd for $C_8H_{14}O_6P$: 237.0528, found 237.0528.

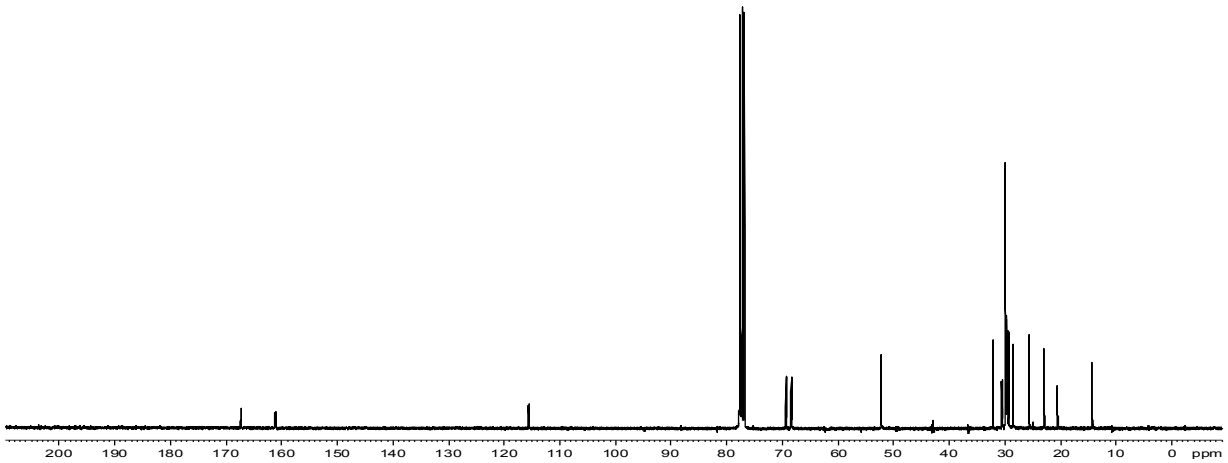


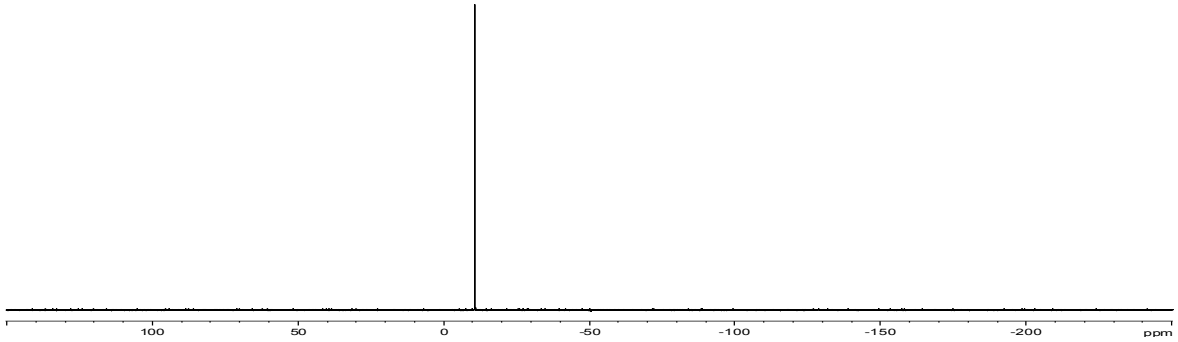

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

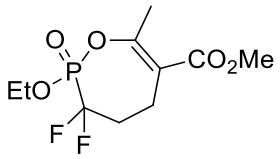

45 40 35 30 25 20 15 10 5 0 -5 -10 -15 -20 -25 -30 -35 -40 -45 ppm


O
O
O
P
O
O
CO2Me

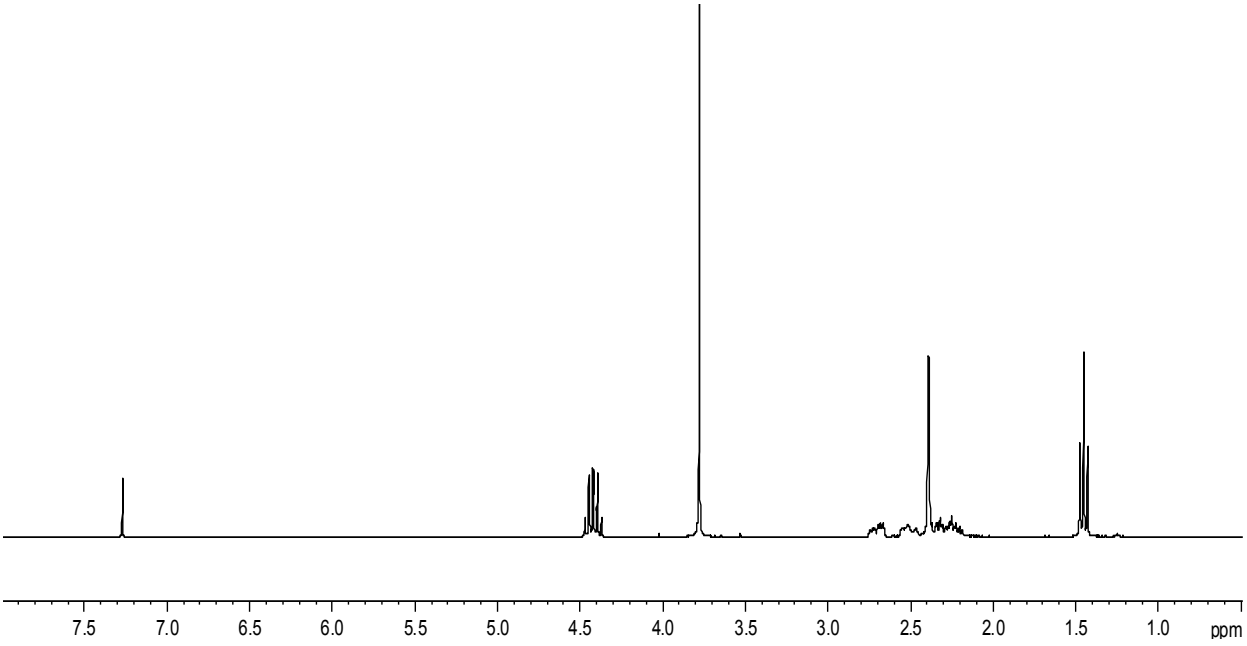

7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

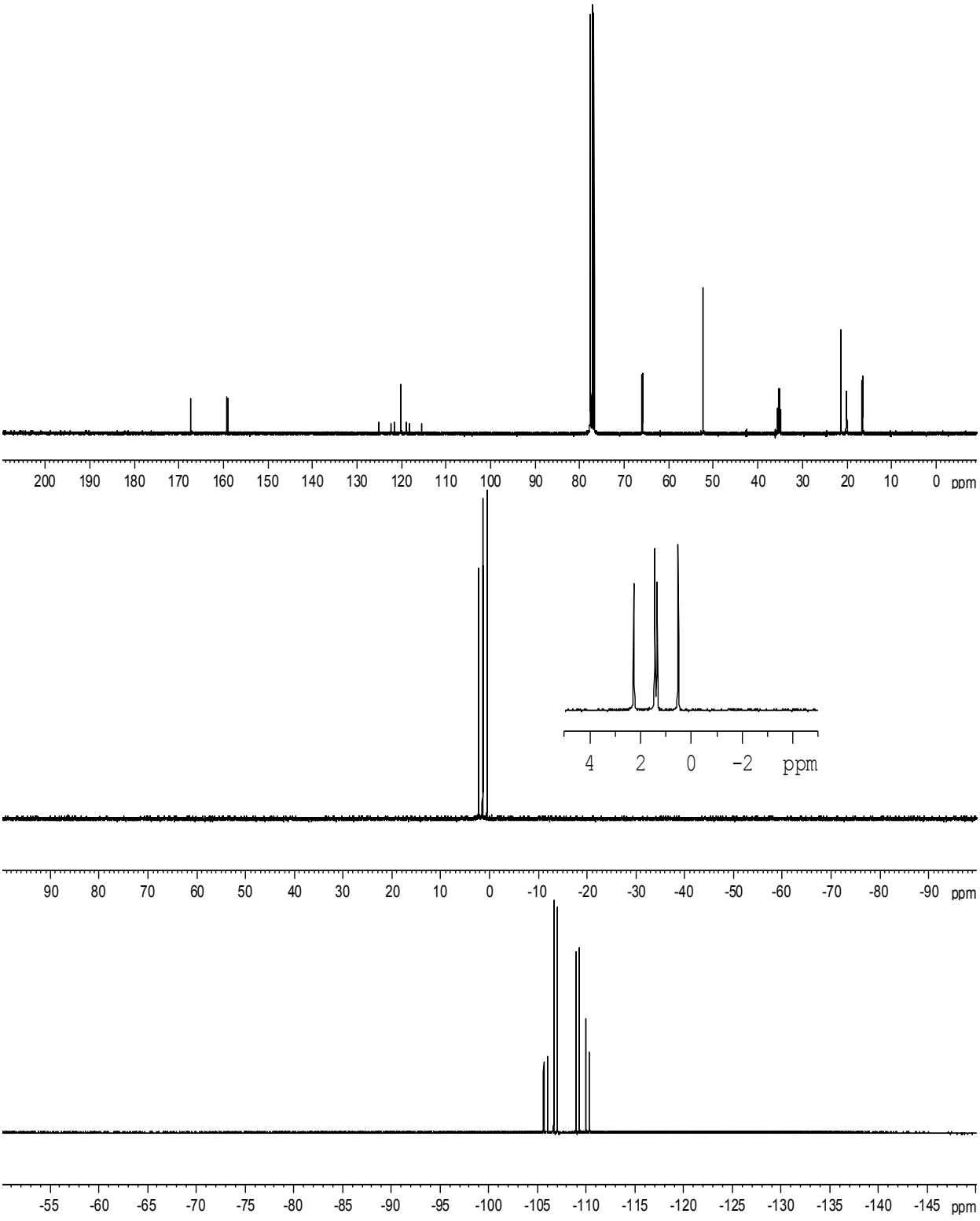

200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

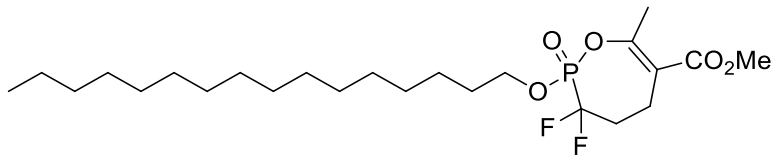

100
50
0
-50
-100
-150
-200
ppm

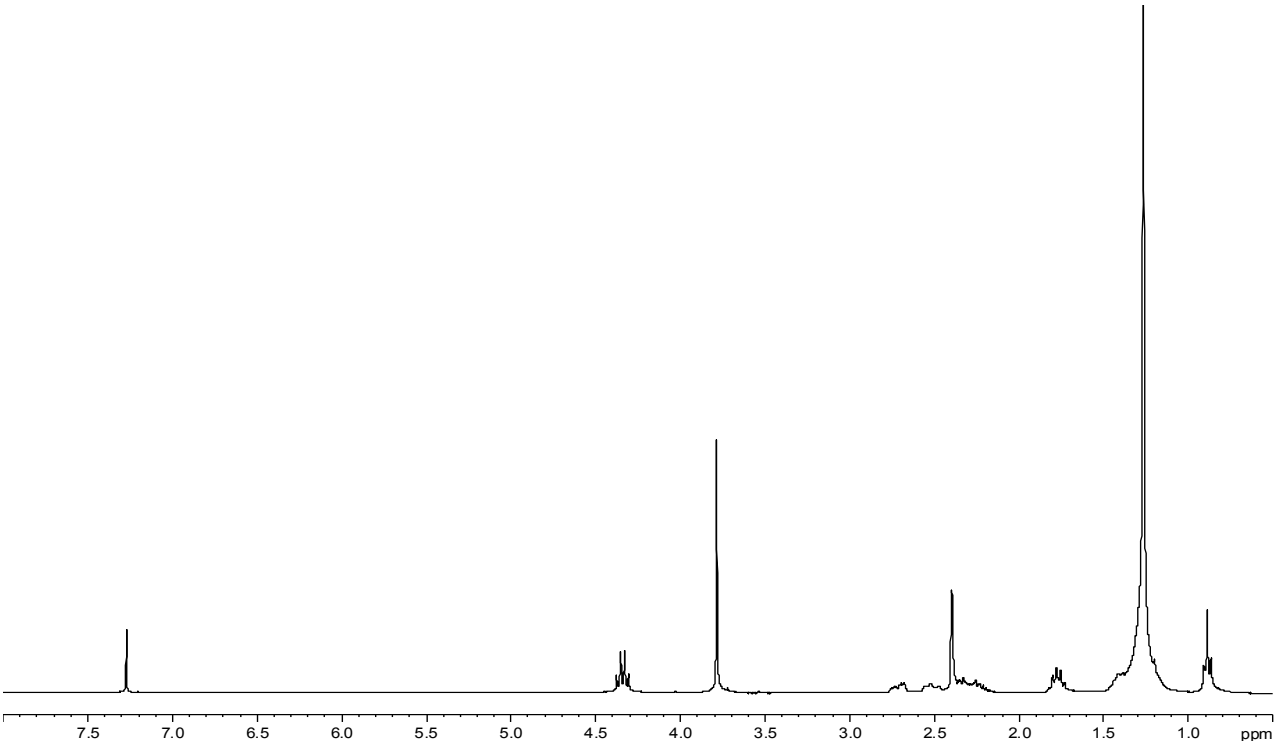


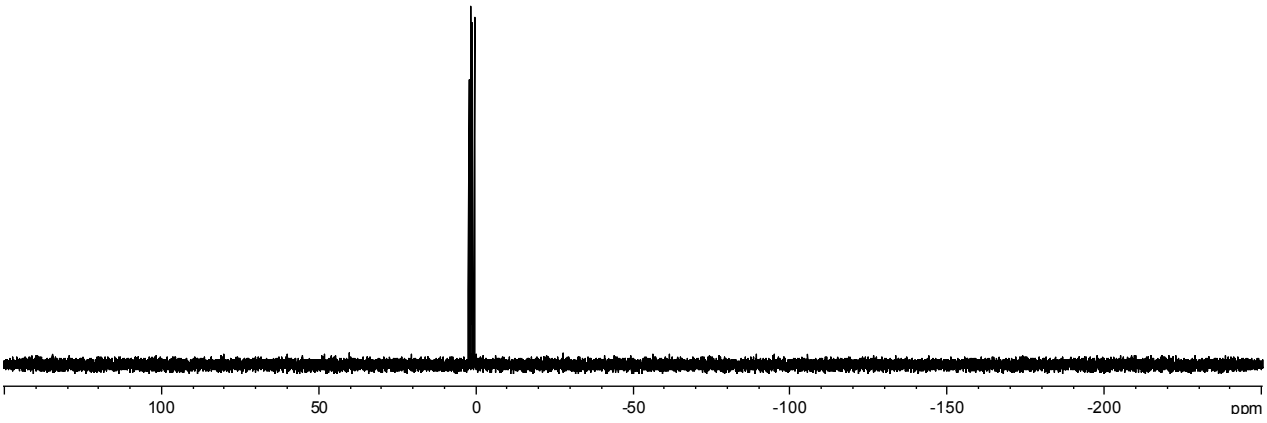
Methyl 2-ethoxy-3,3-difluoro-7-methyl-2,3,4,5-tetrahydro-1,2-oxaphosphepine-6-carboxylate 2-oxide (XX). IR (neat, NaCl, 2956, 2925, 1719, 1655 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.43 (2H, dq, J_{HP} = 8.4 Hz, J_{HF} = 7.1 Hz), 3.78 (3H, s), 2.65–2.77 (1H, m), 2.46–2.58 (1H, m), 2.40 (3H, s), 2.14–2.42 (2H, m), 1.45 (3H, t, J_{HH} = 7.1 Hz); ^{13}C NMR (75.4 MHz, $CDCl_3$) δ 167.3 (d, J_{CP} = 2.0 Hz), 159.2 (d, J_{CP} = 8.0 Hz), 120.3 (td, J_{CF} = 261.4 Hz, J_{CP} = 205 Hz), 120.2 (d, J_{CP} = 6.0 Hz), 65.9 (d, J_{CP} = 7.1 Hz), 52.3, 35.3 (td, J_{CF} = 20.6 Hz, J_{CP} = 11.6 Hz), 21.4, 20.1 (dd, J_{CF} = 8.8 Hz, 3.8 Hz), 16.5 (d, J_{CP} = 5.5 Hz); $^{31}P\{^{1}H\}$ NMR (121.4 MHz, $CDCl_3$) δ 1.4 (dd, J_{PF} 111.4, 100.9 Hz); $^{19}F\{^{1}H\}$ NMR (282.2 MHz, $CDCl_3$) δ -109.6 (dd, J_{FF} = 290.5 Hz, J_{FP} = 100.8 Hz), -106.3 (dd, J_{FF} = 290.5 Hz, J_{FP} = 111.5 Hz); HRMS (FAB, NBA, MH^+) calcd for $C_{10}H_{15}F_2O_5P$: 285.0703, found 285.0708.

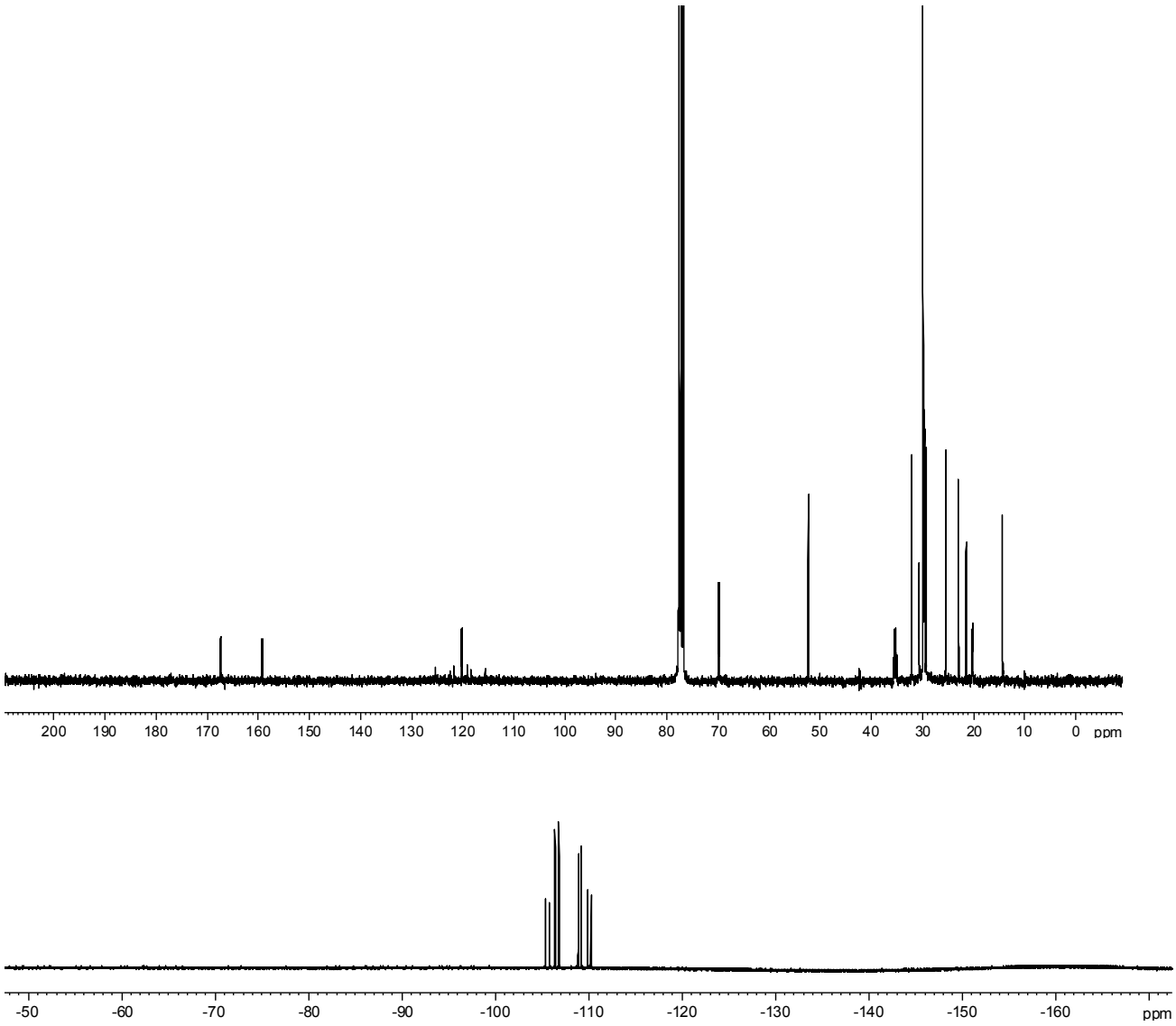



200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm
4 2 0 −2 ppm
90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 ppm
-55 -60 -65 -70 -75 -80 -85 -90 -95 -100 -105 -110 -115 -120 -125 -130 -135 -140 -145 ppm


O
O
P
O
F
F
CO2Me


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
ppm


100
50
0
-50
-100
-150
-200
ppm


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