Electronic Supplementary Material (ESI) for Chemical Science.

Supporting information for:

Facile Access to Functionalized Chiral Secondary Benzylic Boronic Esters via Catalytic Asymmetric Hydroboration

NMR Spectra

Suman Chakrabarty,[a] Hector Palencia,[b] Martha D. Morton,[a,c] Ryan O. Carr,[a] and James M. Takacs*[a,c]

[a] Department of Chemistry, University of Nebraska–Lincoln, Lincoln, Nebraska 68588–0304, United States.
[b] Department of Chemistry, University of Nebraska–Kearney, Kearney, Nebraska 68849, United States.
[c] Nebraska Center for Integrated Biomolecular Communication (NCIBC), University of Nebraska–Lincoln, Lincoln, Nebraska 68588–0304, United States.

Corresponding author e-mail: jtakacs1@unl.edu

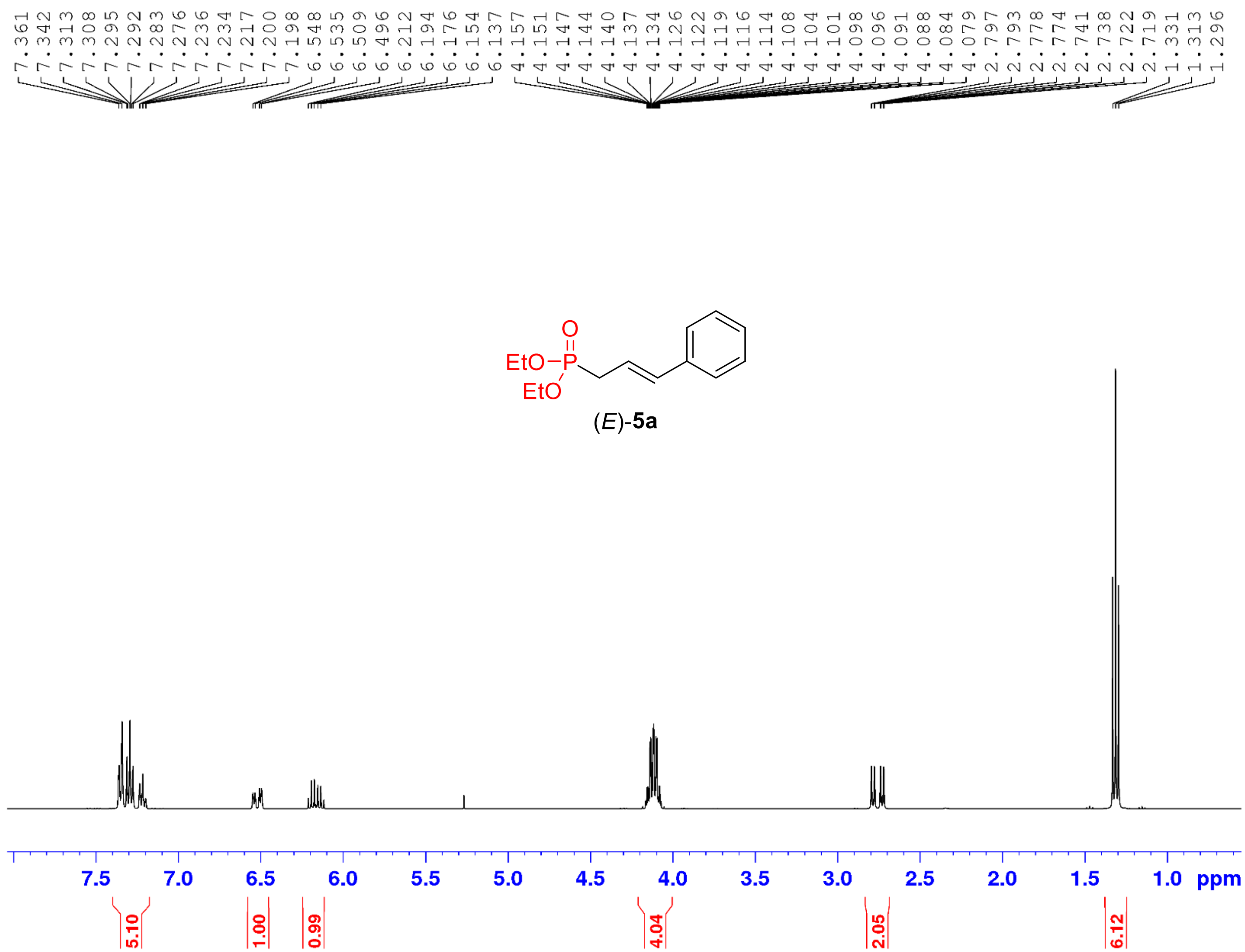
Table of Contents
(Hyperlinked: Click to navigate)

(*E*)-5a 5
(*Z*)-5a 11
(*E*)-5b 17
(*E*)-5c 23
(*E*)-5d 30
(*E*)-5e 37
(*E*)-5f 43
(*E*)-5g 49
(*E*)-5h 55
(*E*)-5i 61
(*E*)-5j 67
(*E*)-5k 73
(*E*)-5l 79
(*E*)-5m 85
(*E*)-5n 91
(*E*)-5o 97
(*E*)-5p 103
(*E*)-5q 109
(*E*)-5r 115
(*E*)-5s 121
(*E*)-5t 127

(*E*)-5u 133
9a 192
9b 228
9c 240
9d 247
9e 254
9f 260
9g 266
9h 272
9i 278
9j 284
9k 290
9l 296
9m 302
9n 308
9o 314
9p 320
9q 326
9r 332
9s 337
9t 343
9u 349
10a 384
11 198

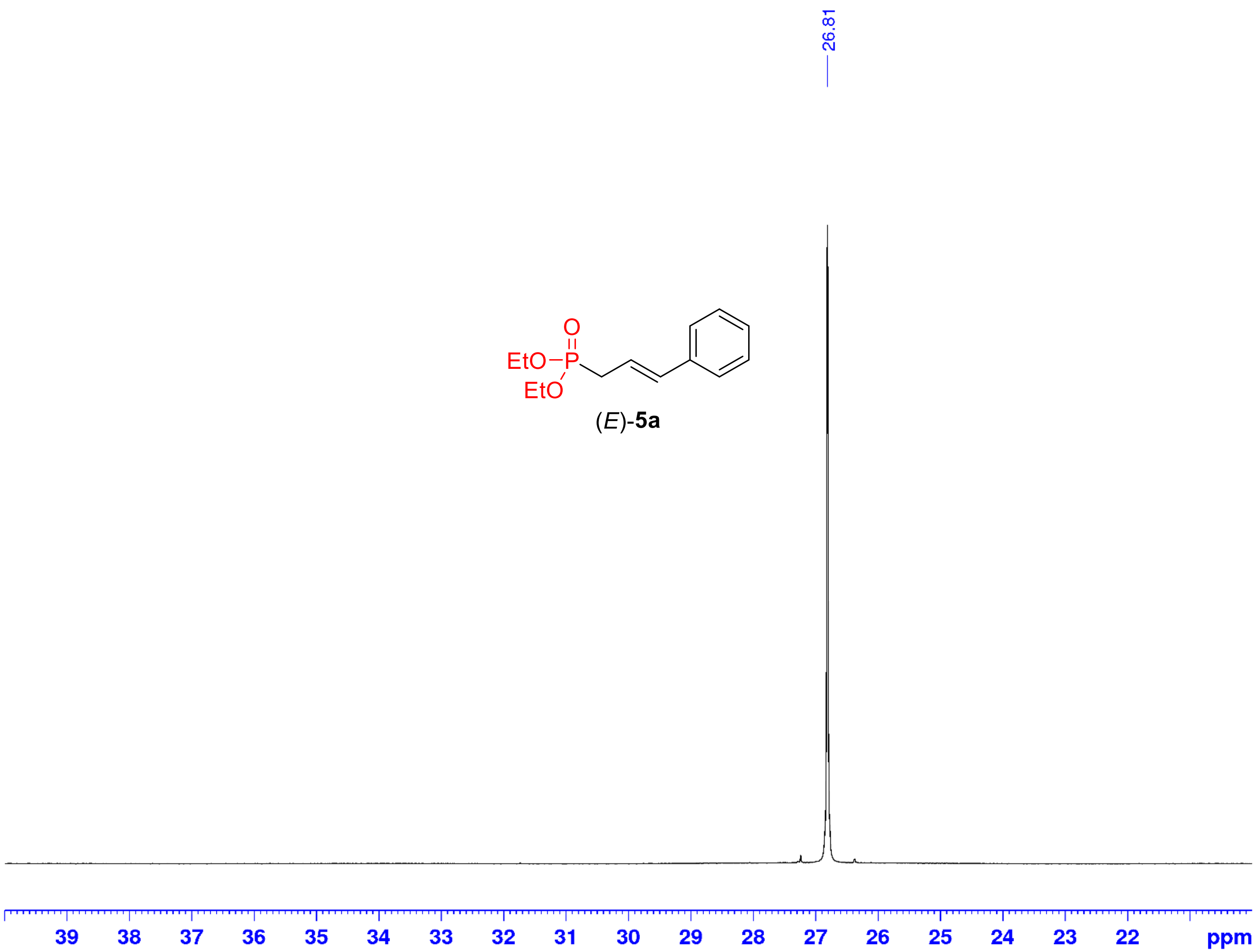
12 204
13 210
14 216
15 222
16 228
(*E*)-17 139
(*E*)-19 144
(*Z*)-19 150
(*E*)-21 153
(*E*)-22 159
(*E*)-23 165
(*E*)-24 177
(*E*)-25 183
(*E*)-31 171
34 355
35 360
36 366
37 372
38 378
Mixture of **9a** and **2-*d*-9a** obtained via CAHB/Oxidation of (*E*)-**5a** with pinBD 391
Mixture of **9a**, diastereomeric **2-*d*-9a** and **2,2-d_2-9a** obtained via CAHB/Oxidation of (*Z*)-**5a** with pinBD 394
Ligand B1 189

(*E*)-5a: ^{1}H NMR



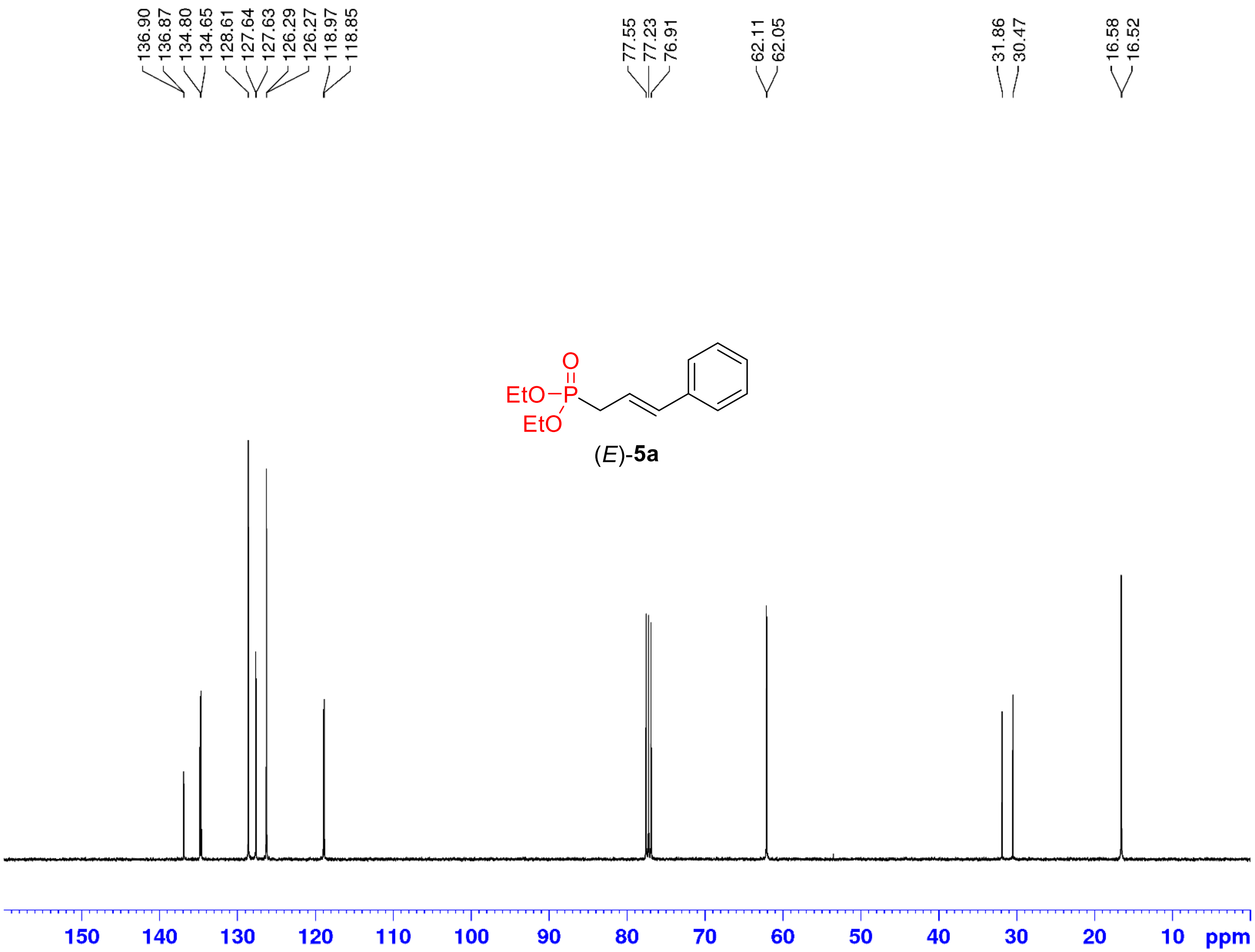
7.361
7.342
7.313
7.308
7.295
7.292
7.283
7.276
7.236
7.234
7.217
7.200
7.198
6.548
6.535
6.509
6.496
6.212
6.194
6.176
6.154
6.137
4.157
4.151
4.147
4.144
4.140
4.137
4.134
4.126
4.122
4.119
4.116
4.114
4.108
4.104
4.101
4.098
4.096
4.091
4.088
4.084
4.079
2.797
2.793
2.778
2.774
2.741
2.738
2.722
2.719
1.331
1.313
1.296
O
EtO
P
EtO
(E)-5a
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
5.10
1.00
0.99
4.04
2.05
6.12

(*E*)-5a: ^{31}P NMR



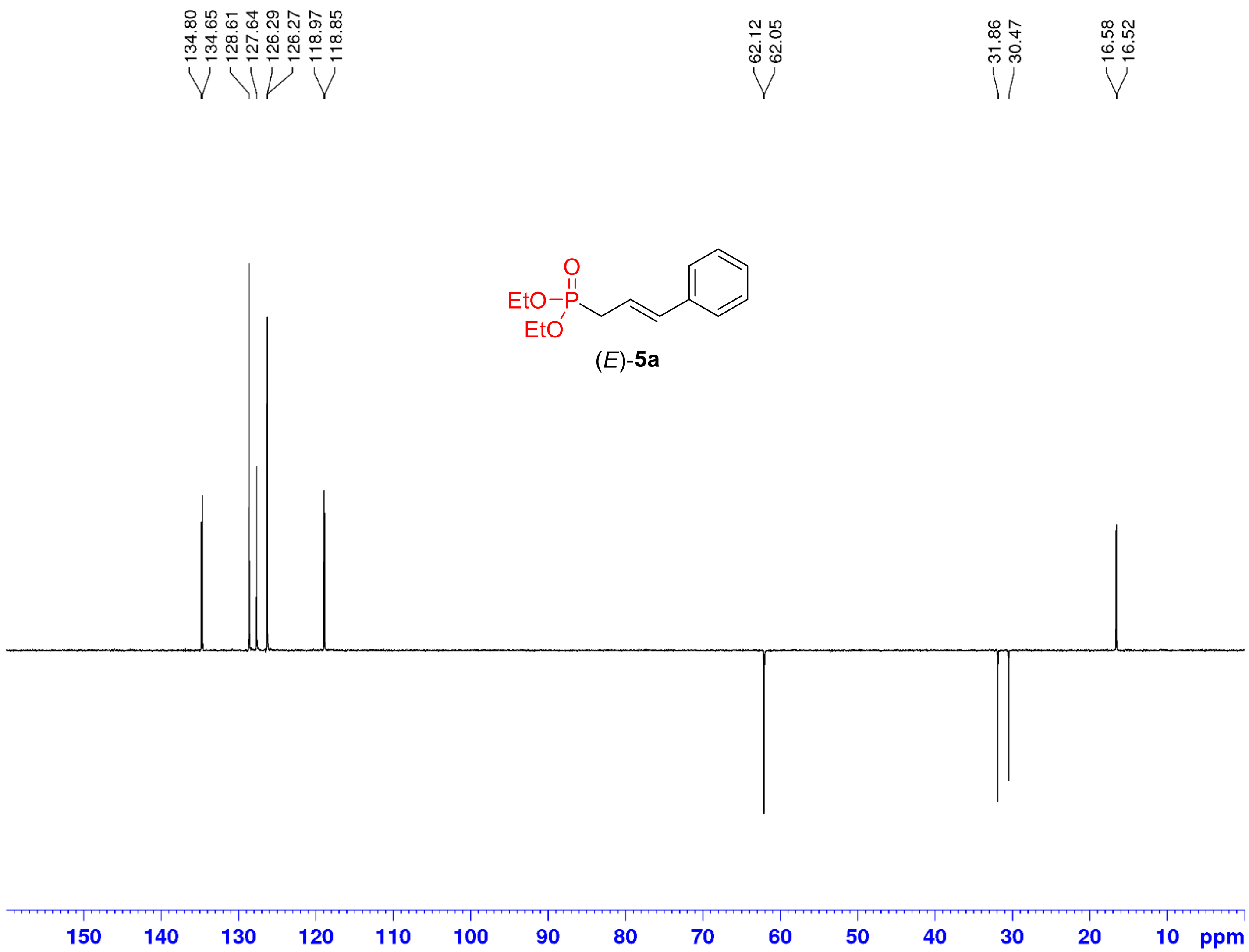
26.81
EtO
O
P
EtO
(*E*)-**5a**
39
38
37
36
35
34
33
32
31
30
29
28
27
26
25
24
23
22
ppm

(*E*)-5a: ^{13}C NMR

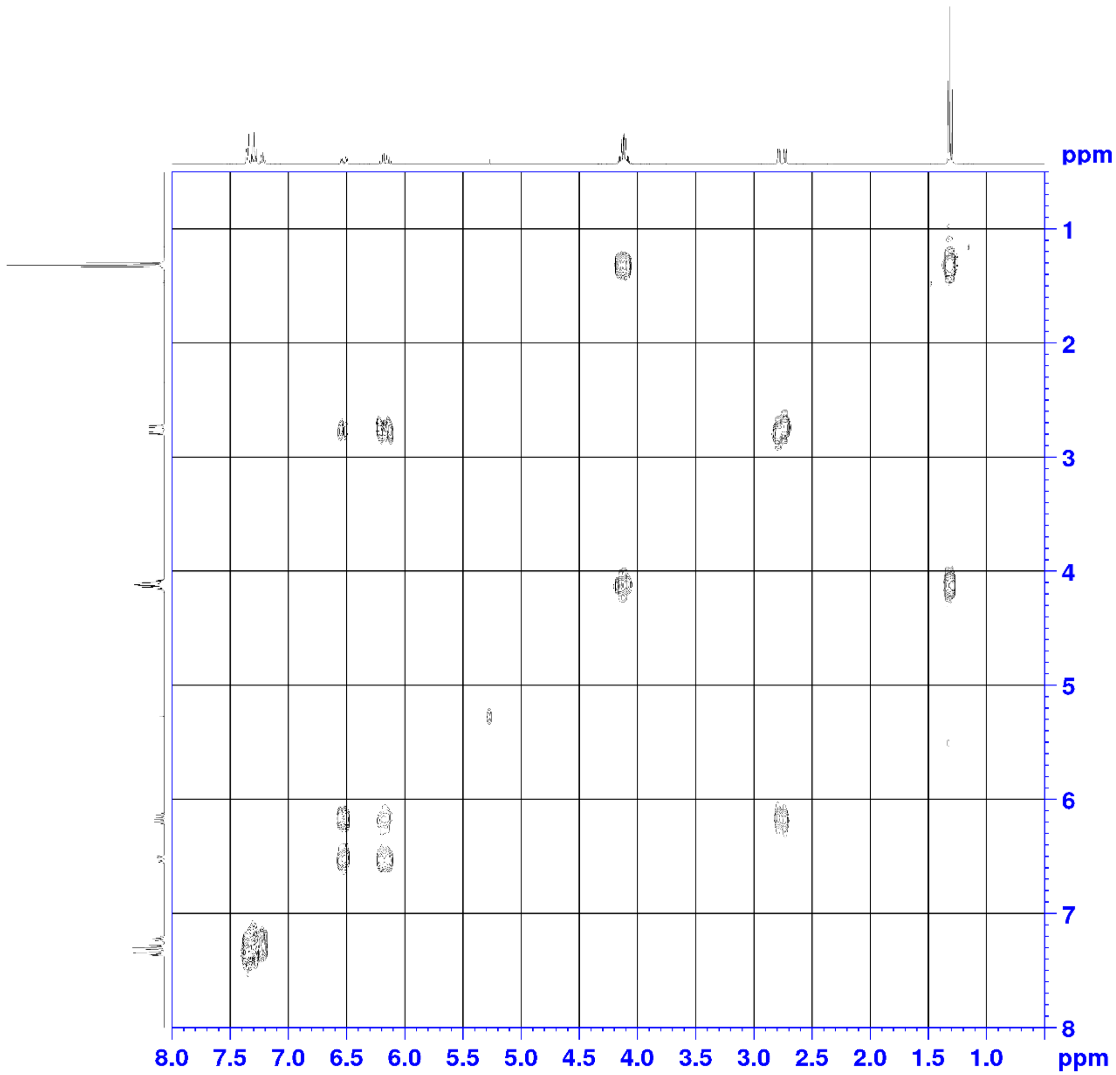


136.90
136.87
134.80
134.65
128.61
127.64
127.63
126.29
126.27
118.97
118.85
77.55
77.23
76.91
62.11
62.05
31.86
30.47
16.58
16.52
O
EtO
P
EtO
(*E*)-**5a**
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

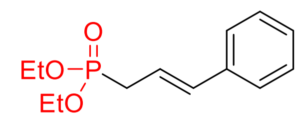
(*E*)-5a: ^{13}C DEPT-135



(*E*)-5a: COSY



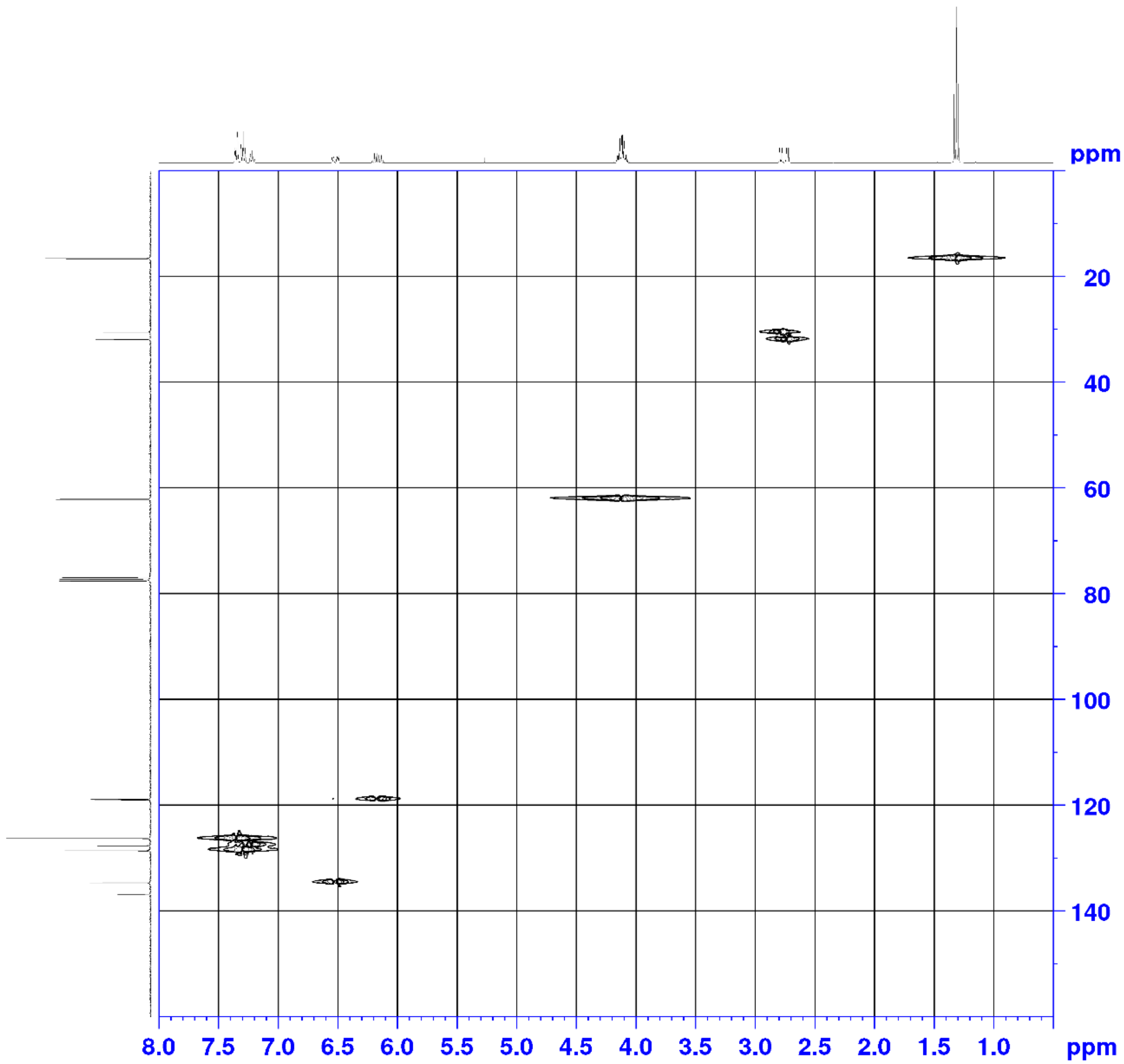
ppm
1
2
3
4
5
6
7
8
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

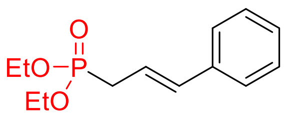


O
EtO
P
EtO

(*E*)-**5a**

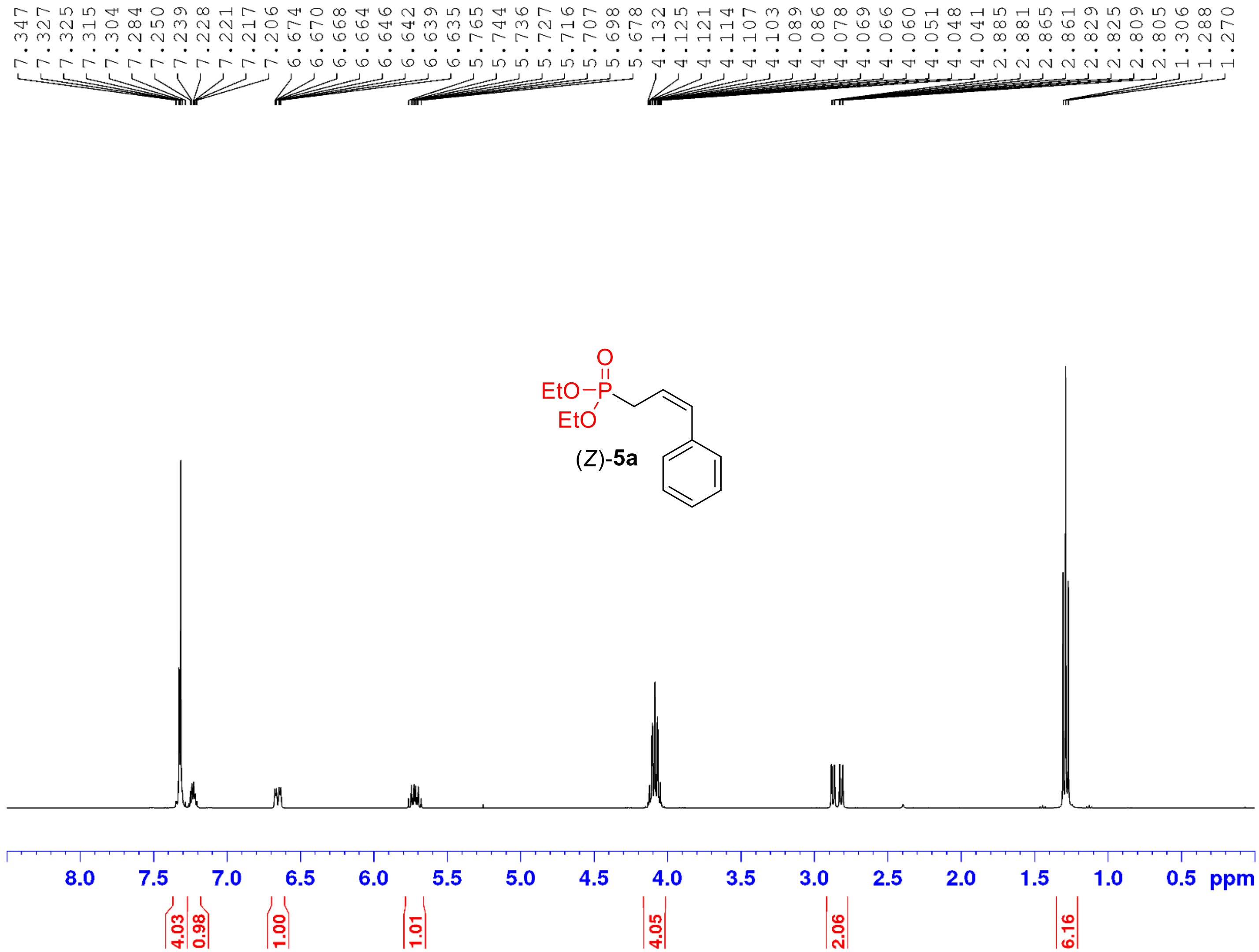
(*E*)-5a: ^{13}C-HSQC



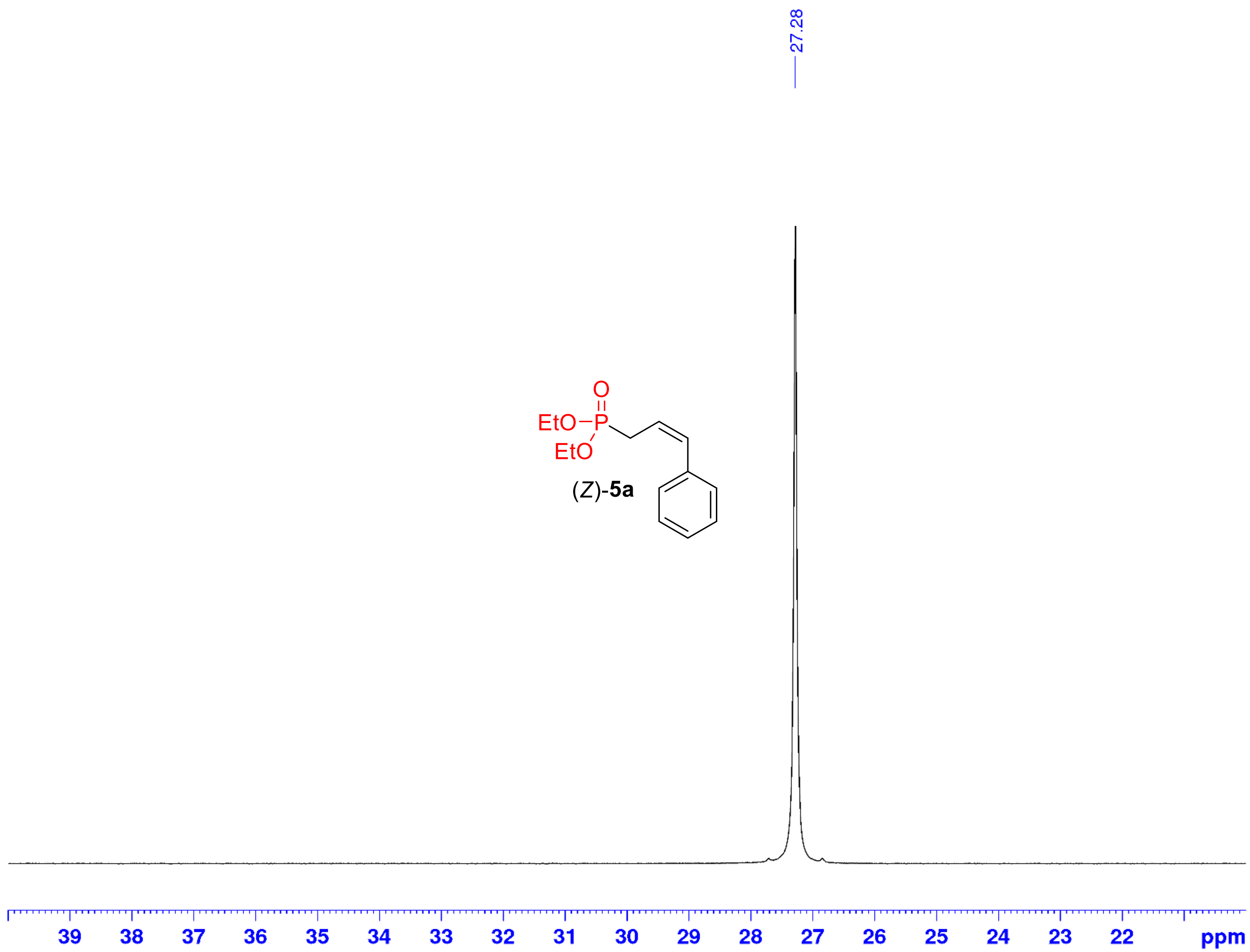


(*E*)-**5a**

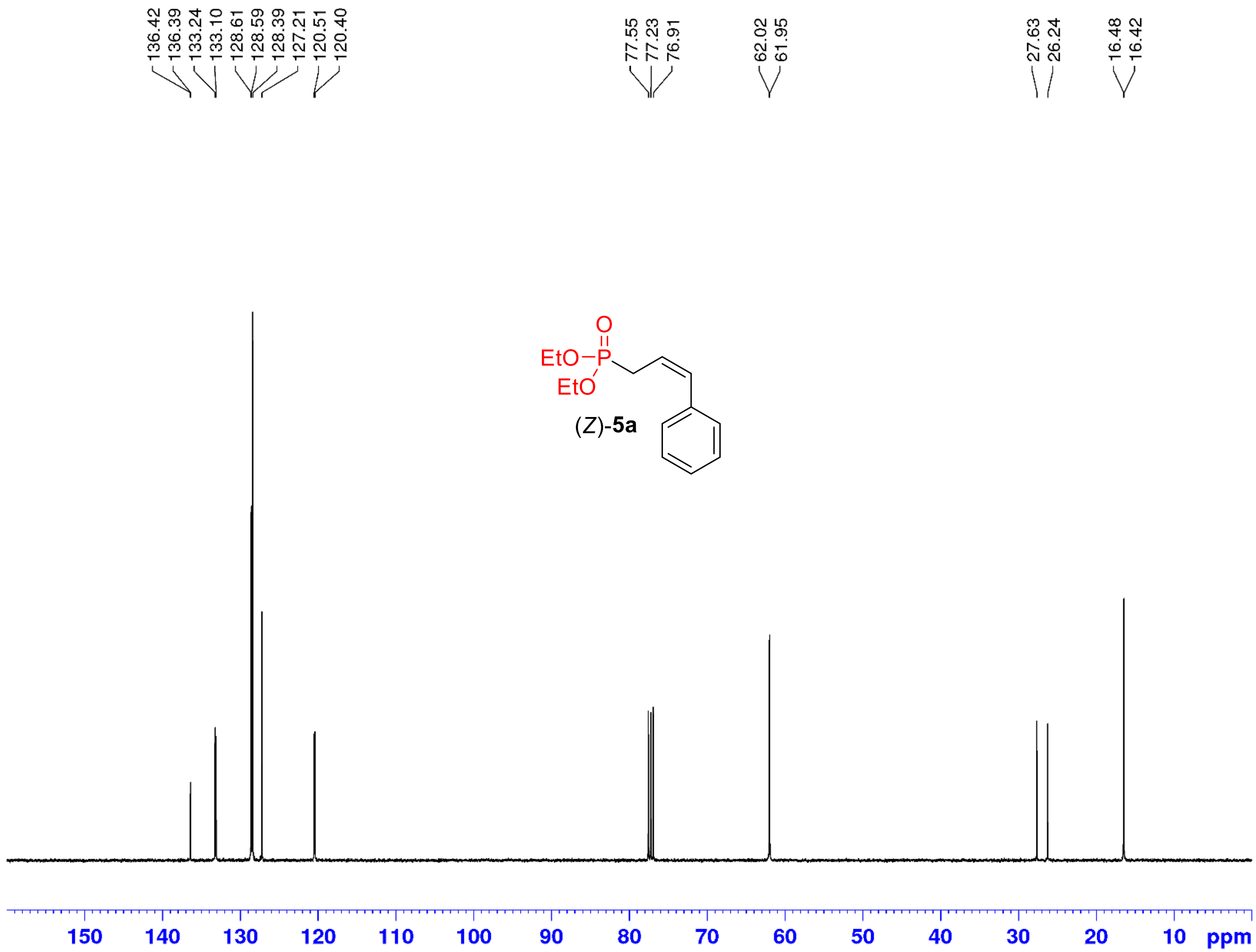
(*Z*)-5a: ^{1}H NMR


7.347
7.327
7.325
7.315
7.304
7.284
7.250
7.239
7.228
7.221
7.217
7.206
6.674
6.670
6.668
6.664
6.646
6.642
6.639
6.635
5.765
5.744
5.736
5.727
5.716
5.707
5.698
5.678
4.132
4.125
4.121
4.114
4.107
4.103
4.089
4.086
4.078
4.069
4.066
4.060
4.051
4.048
4.041
2.885
2.881
2.865
2.861
2.829
2.825
2.809
2.805
1.306
1.288
1.270
O
EtO
P
EtO
(Z)-5a
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
ppm
4.03
0.98
1.00
1.01
4.05
2.06
6.16

(*Z*)-5a: ^{31}P NMR

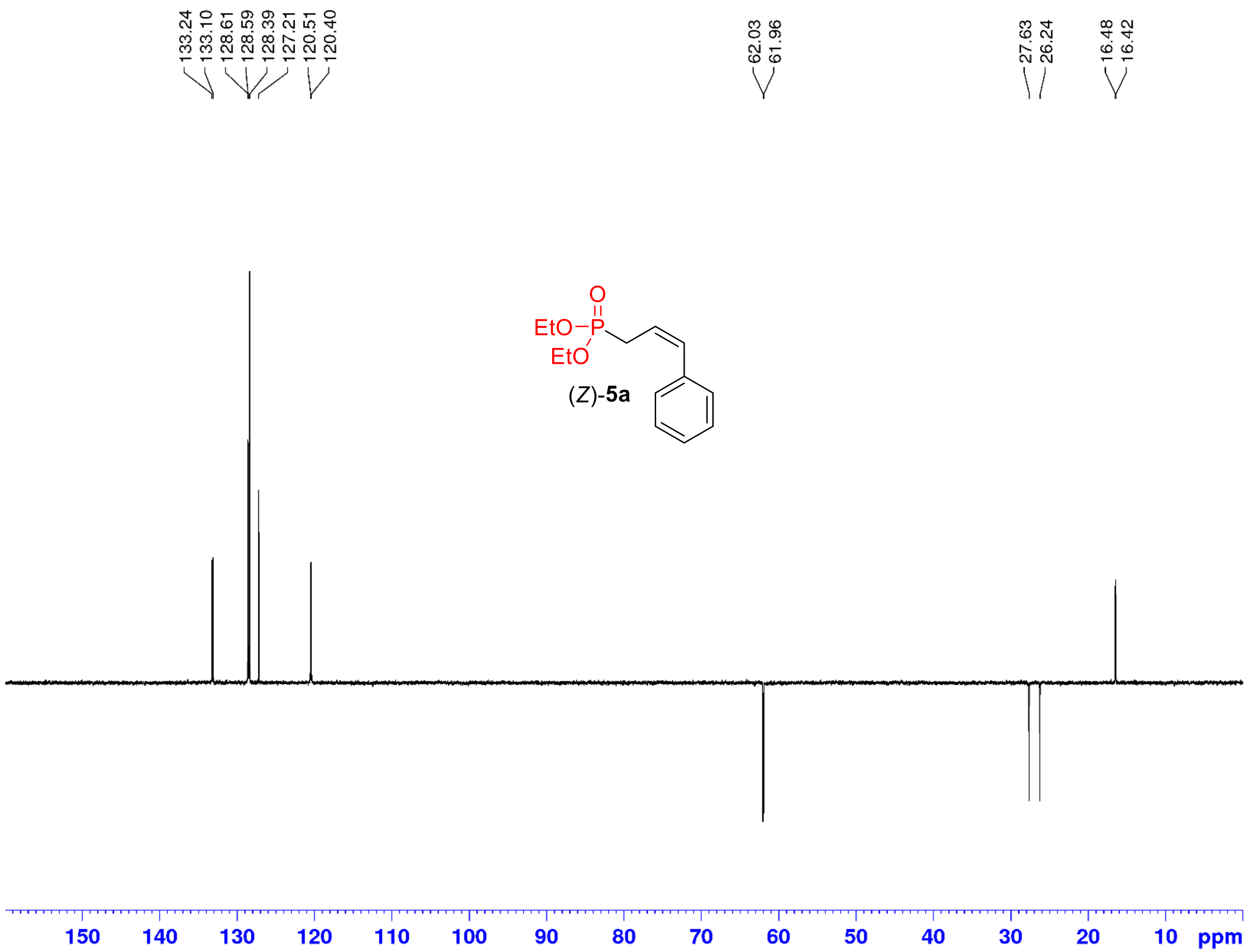

27.28
EtO
EtO
O
P
(*Z*)-**5a**
39
38
37
36
35
34
33
32
31
30
29
28
27
26
25
24
23
22
ppm

(Z)-5a: ^{13}C NMR

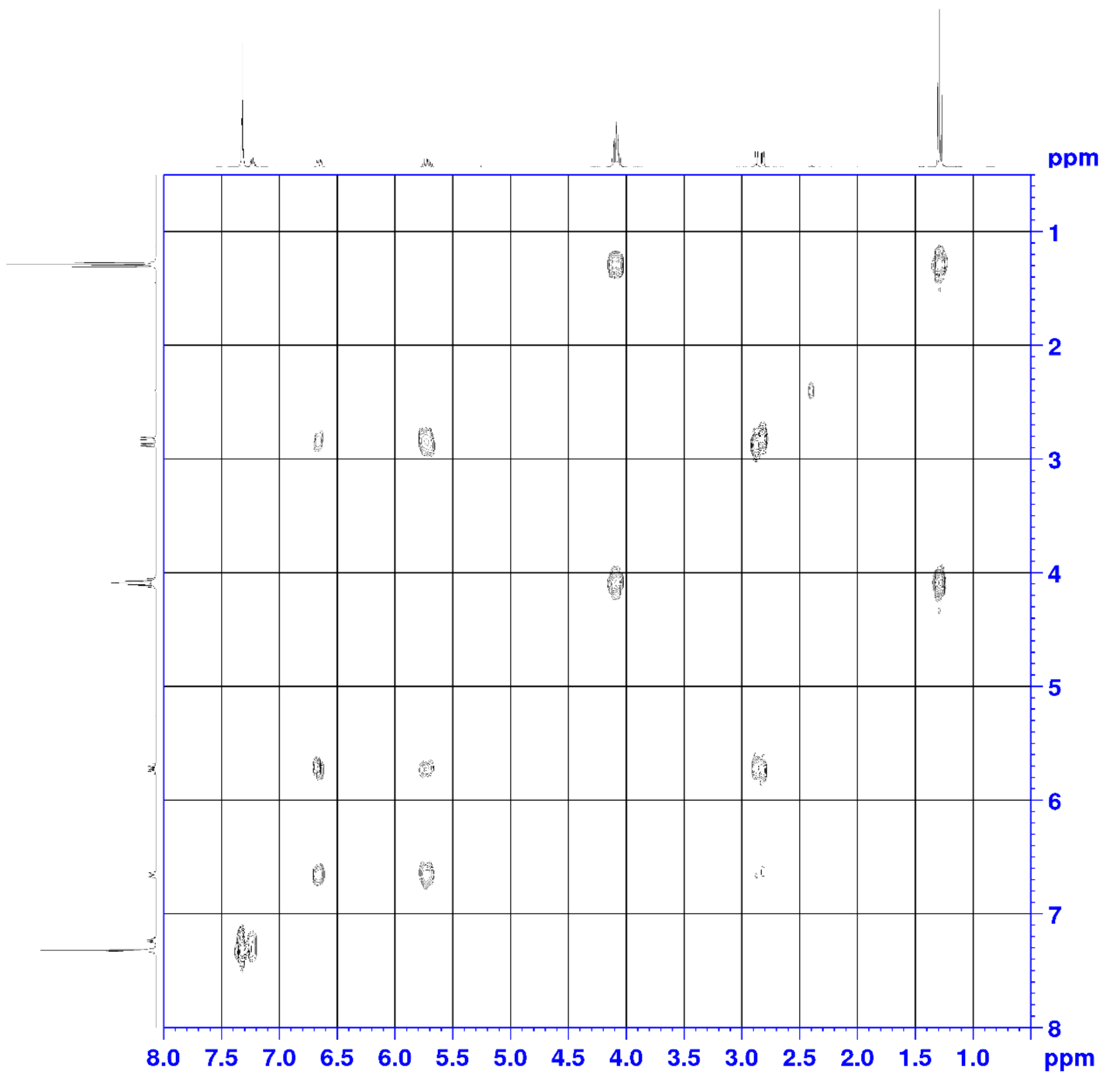


136.42
136.39
133.24
133.10
128.61
128.59
128.39
127.21
120.51
120.40
77.55
77.23
76.91
62.02
61.95
27.63
26.24
16.48
16.42
EtO
P
O
EtO
(Z)-5a
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

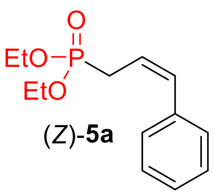
(*Z*)-5a: ^{13}C DEPT135



(Z)-5a: COSY



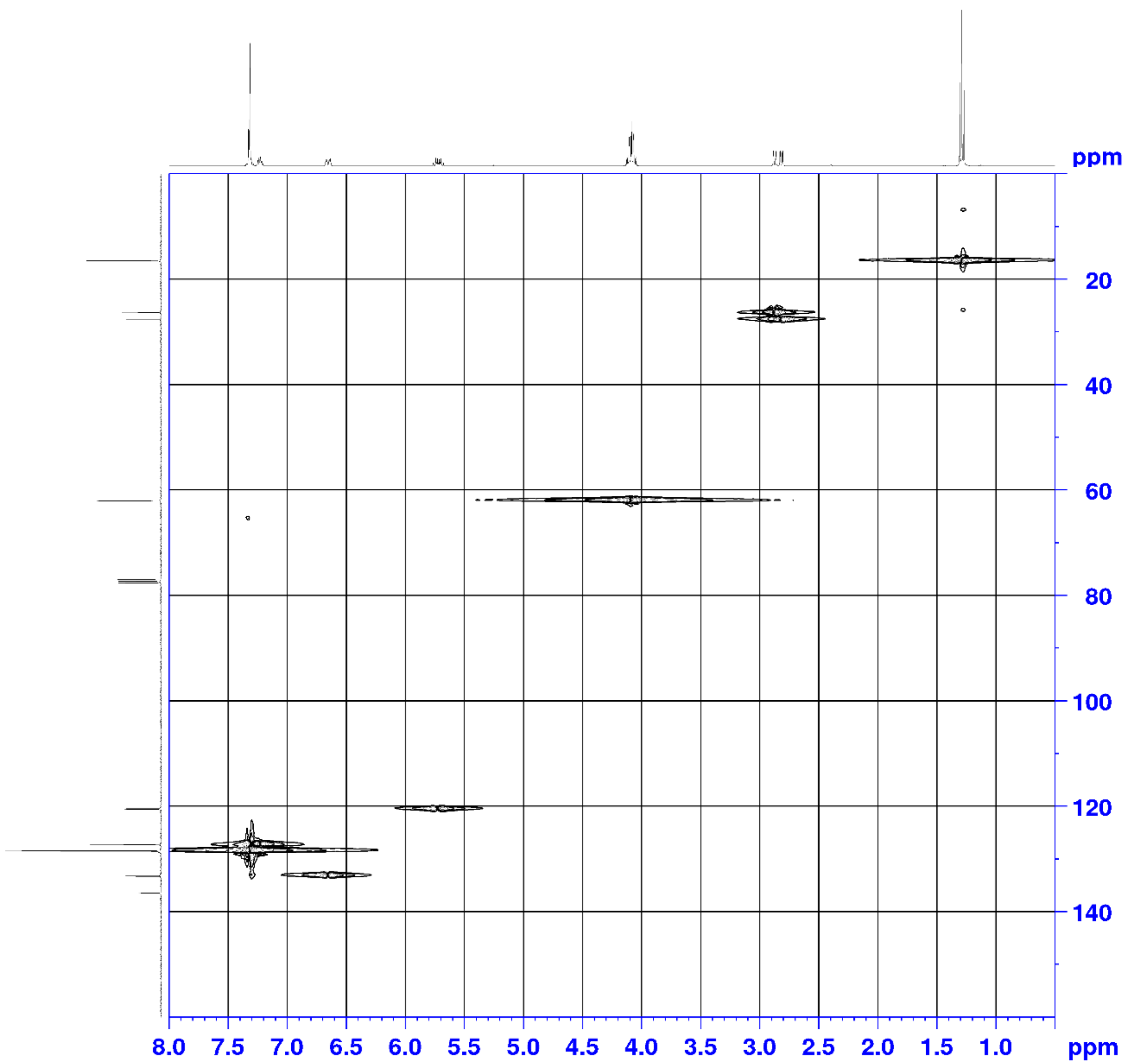
ppm
1
2
3
4
5
6
7
8
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

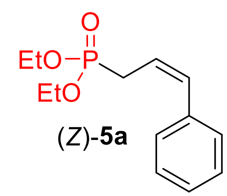


O
EtO
P
EtO

(Z)-**5a**

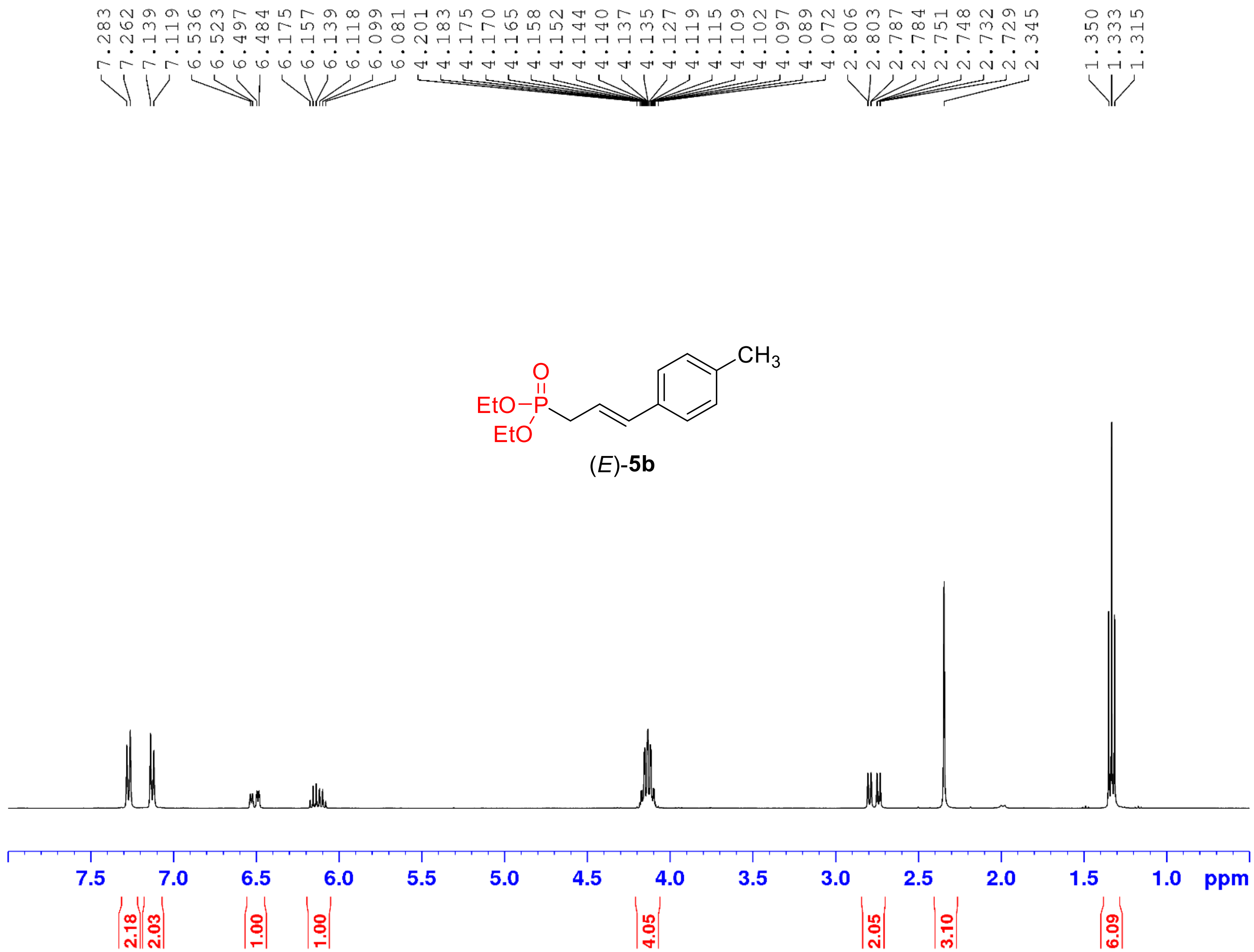
(*Z*)-5a: ^{13}C-HSQC



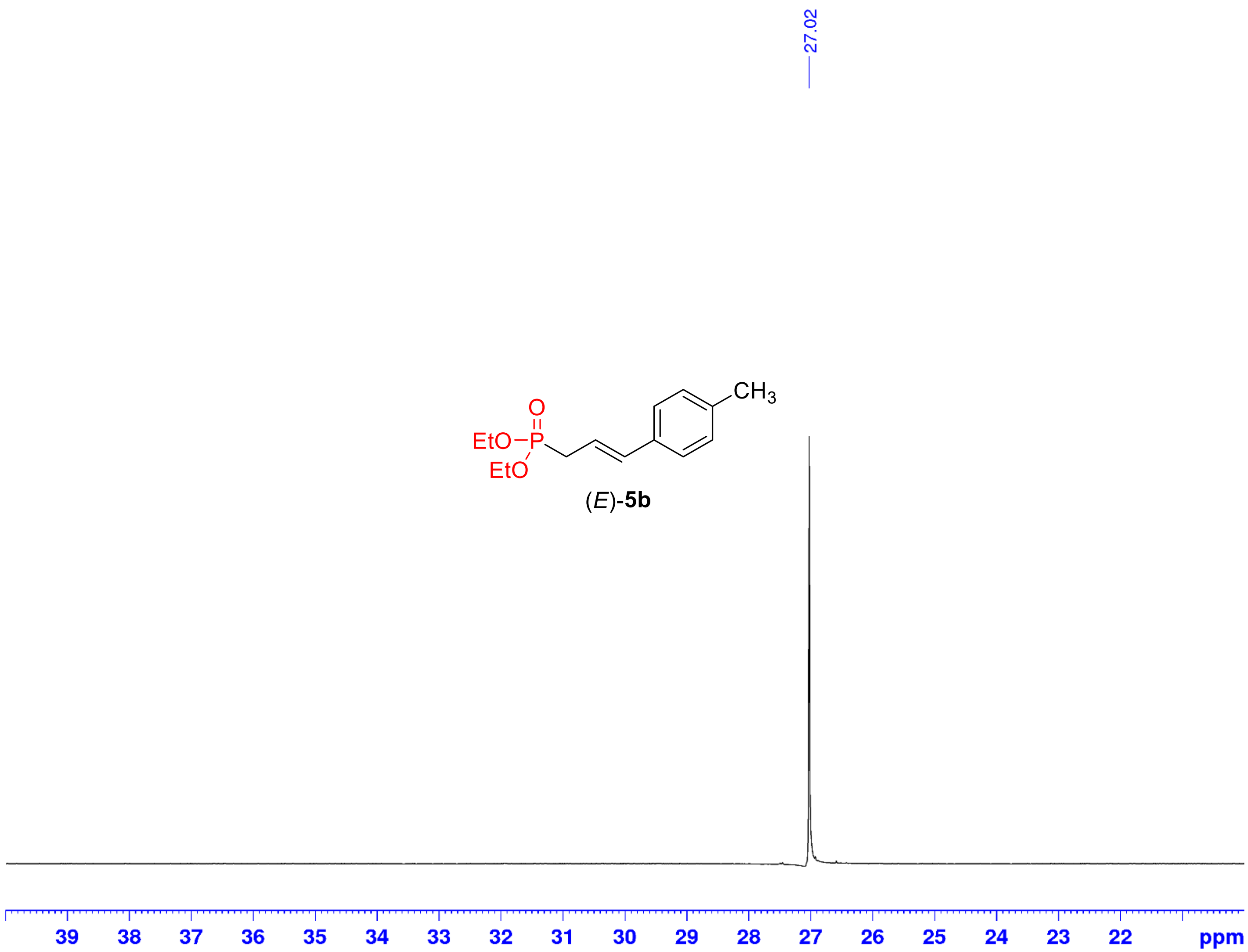


(*Z*)-**5a**

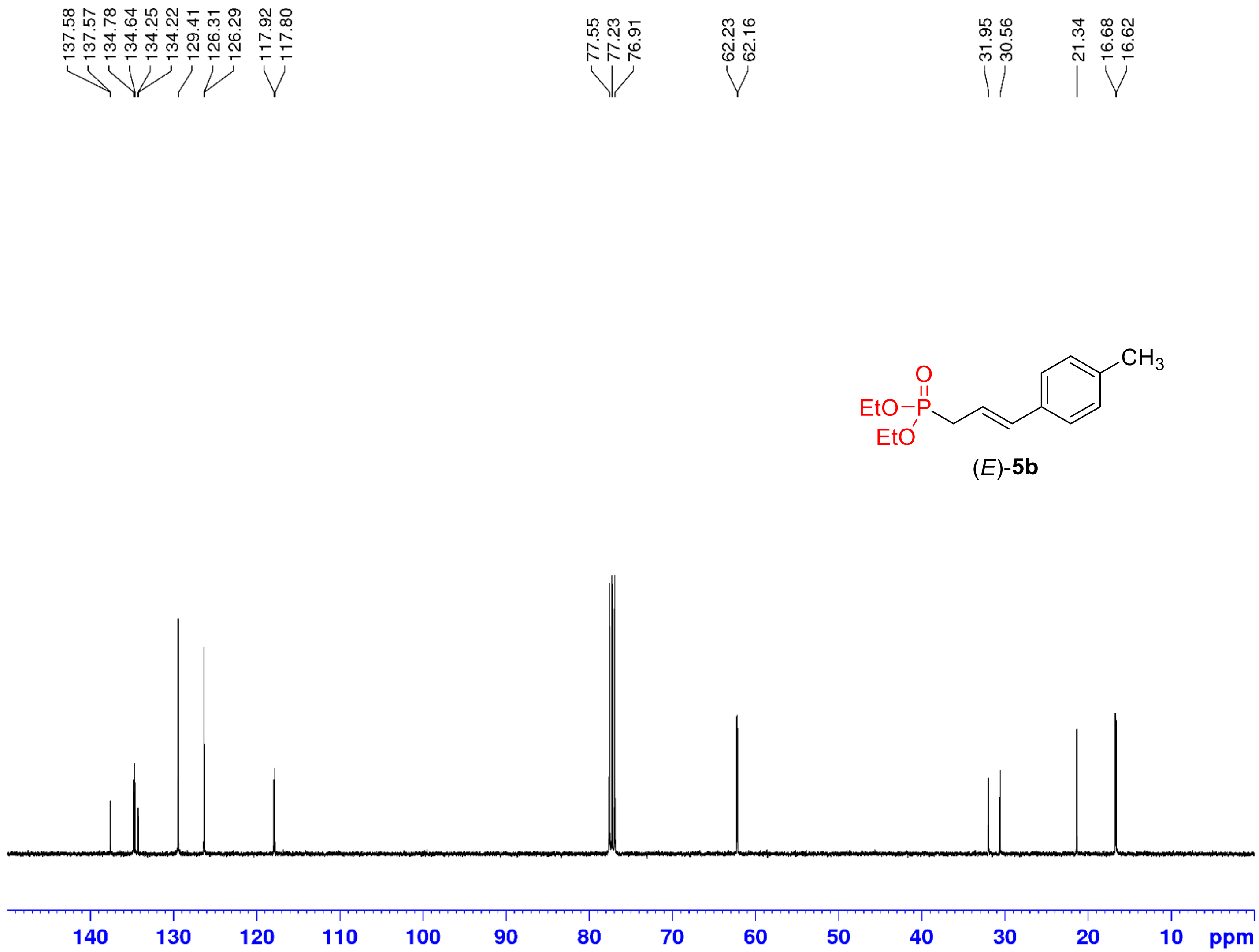
(*E*)-5b: ¹H NMR


7.283
7.262
7.139
7.119
6.536
6.523
6.497
6.484
6.175
6.157
6.139
6.118
6.099
6.081
4.201
4.183
4.175
4.170
4.165
4.158
4.152
4.144
4.140
4.137
4.135
4.127
4.119
4.115
4.109
4.102
4.097
4.089
4.072
2.806
2.803
2.787
2.784
2.751
2.748
2.732
2.729
2.345
1.350
1.333
1.315
CH3
O
EtO
P
EtO
(E)-5b
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
2.18
2.03
1.00
1.00
4.05
2.05
3.10
6.09

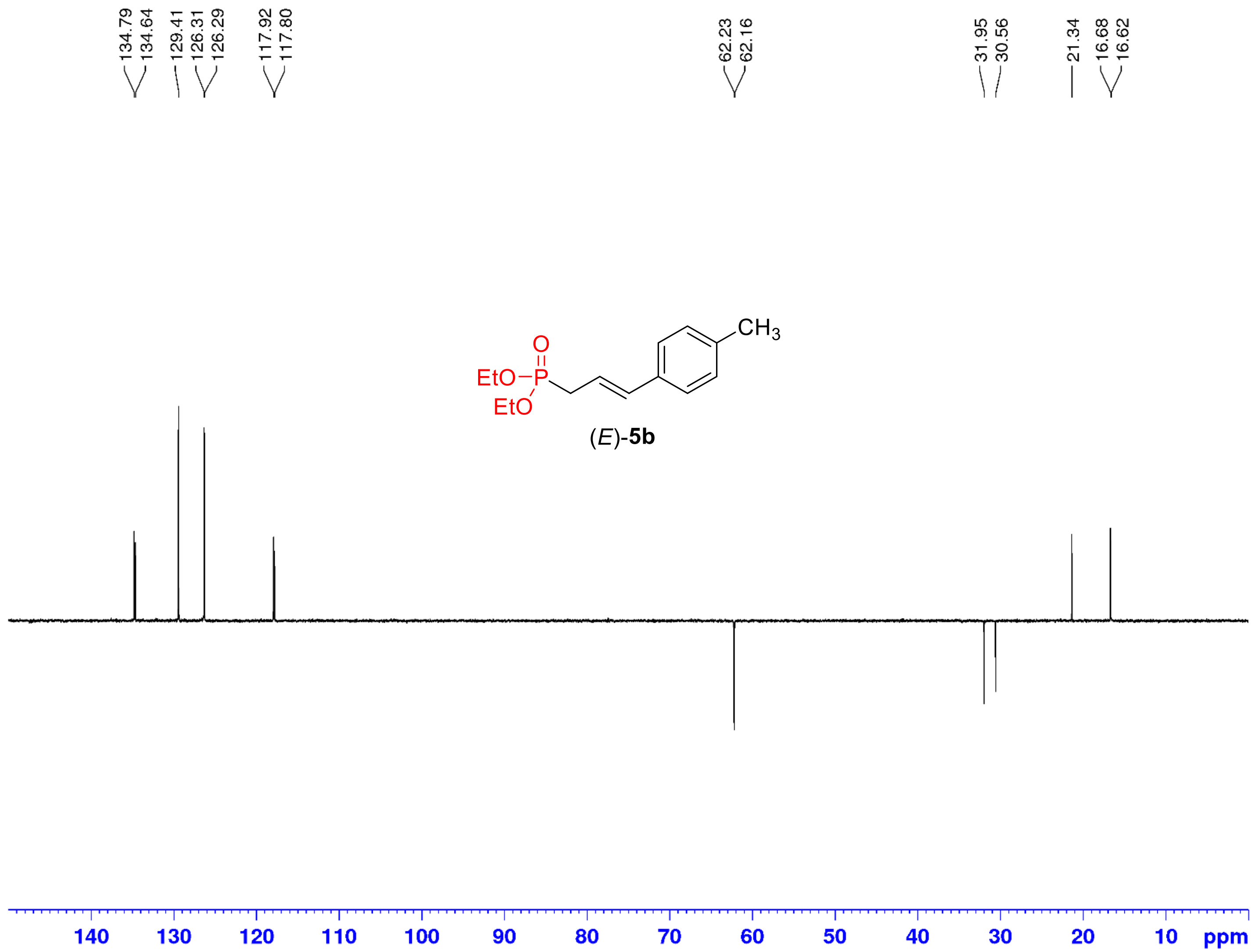
(*E*)-5b: ^{31}P NMR


27.02
EtO
EtO
O
P
CH3
(E)-5b
39
38
37
36
35
34
33
32
31
30
29
28
27
26
25
24
23
22
ppm

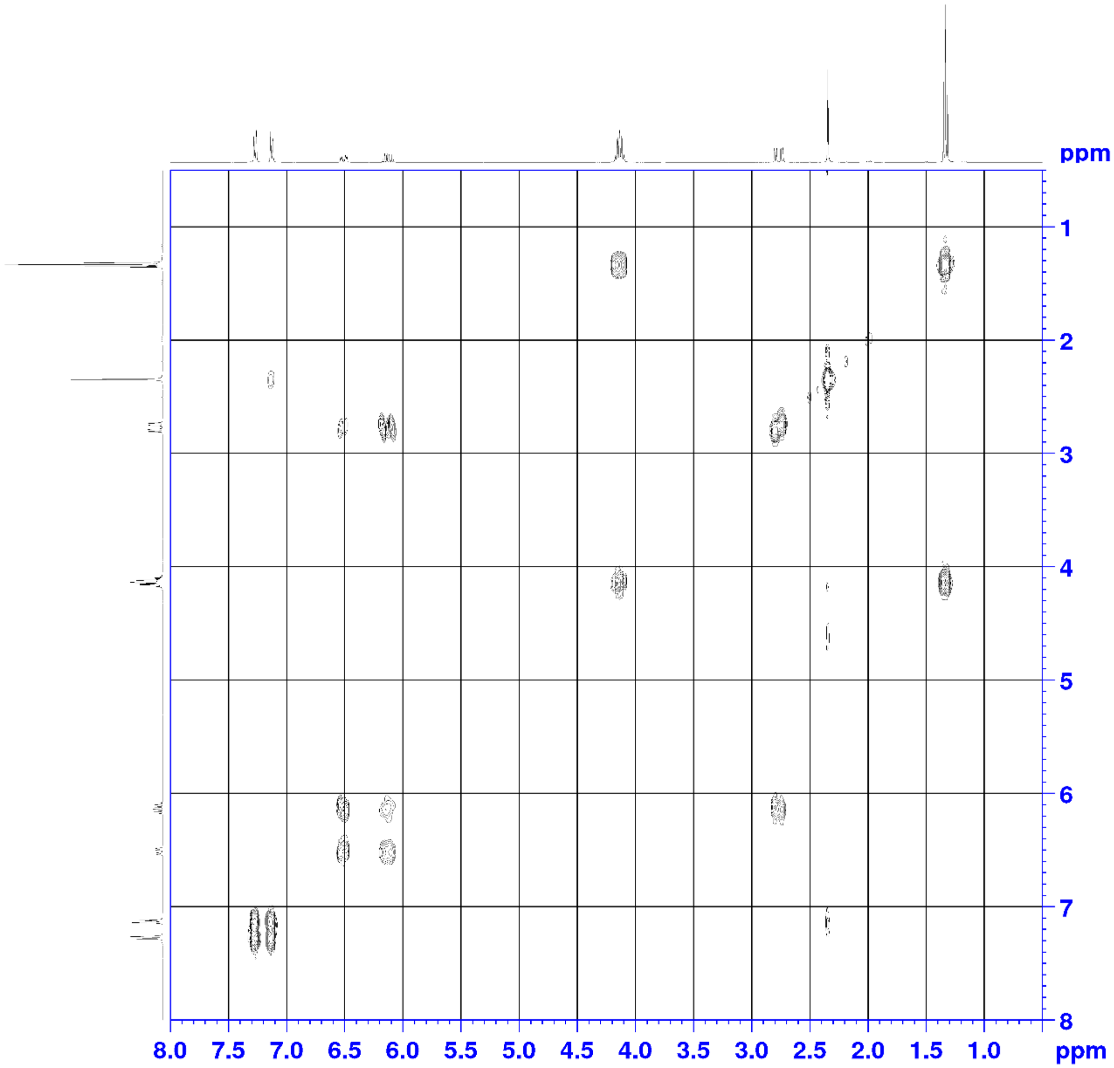
(E)-5b: ^{13}C NMR

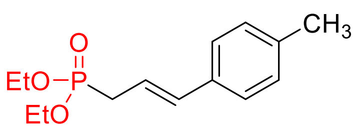

137.58
137.57
134.78
134.64
134.25
134.22
129.41
126.31
126.29
117.92
117.80
77.55
77.23
76.91
62.23
62.16
31.95
30.56
21.34
16.68
16.62
EtO
EtO
O
P
CH3
(E)-5b
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

(*E*)-5b: ^{13}C-DEPT135



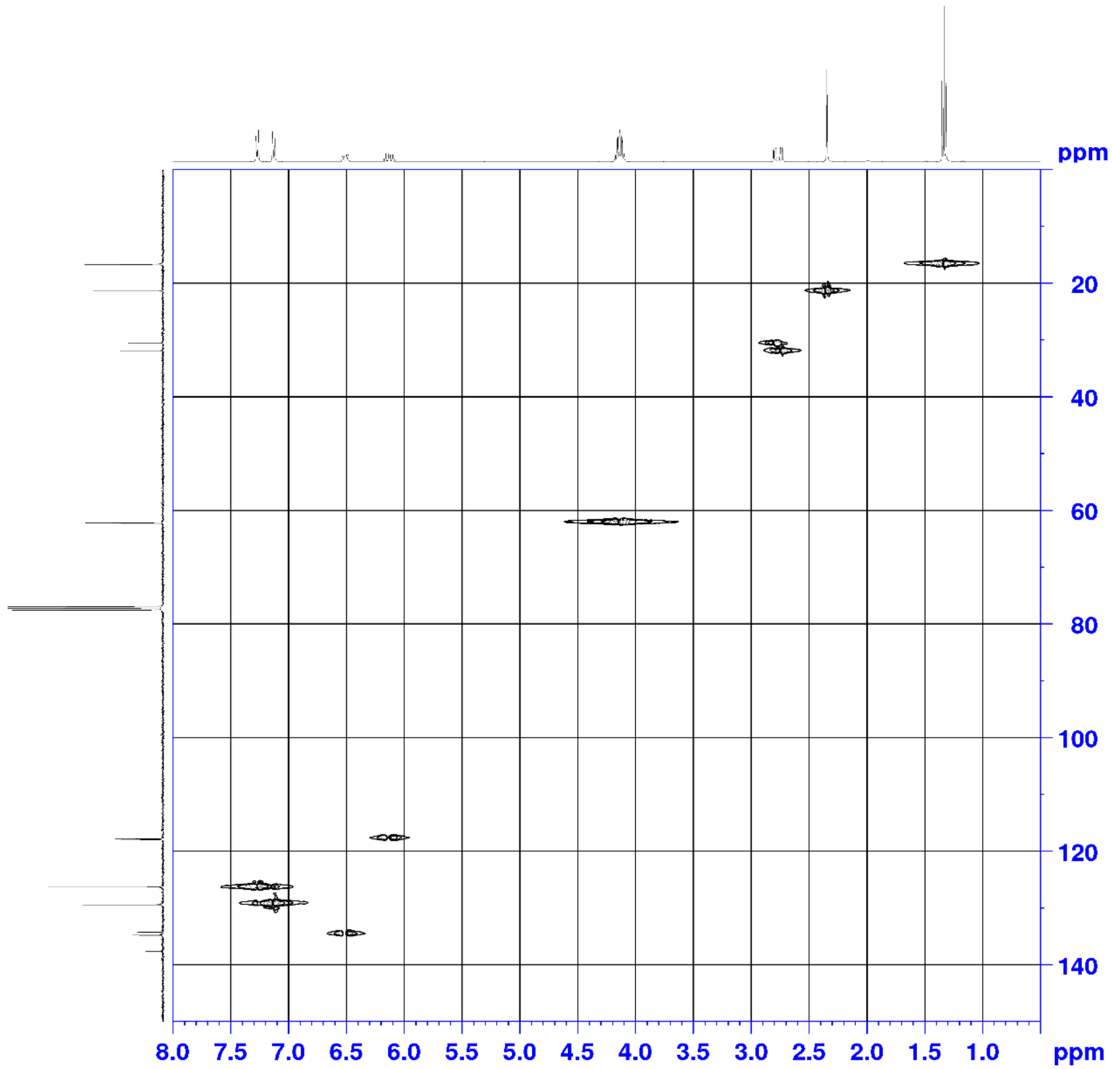
(*E*)-5b: COSY

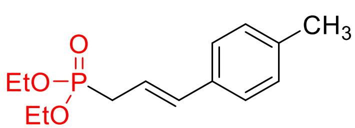




(*E*)-**5b**

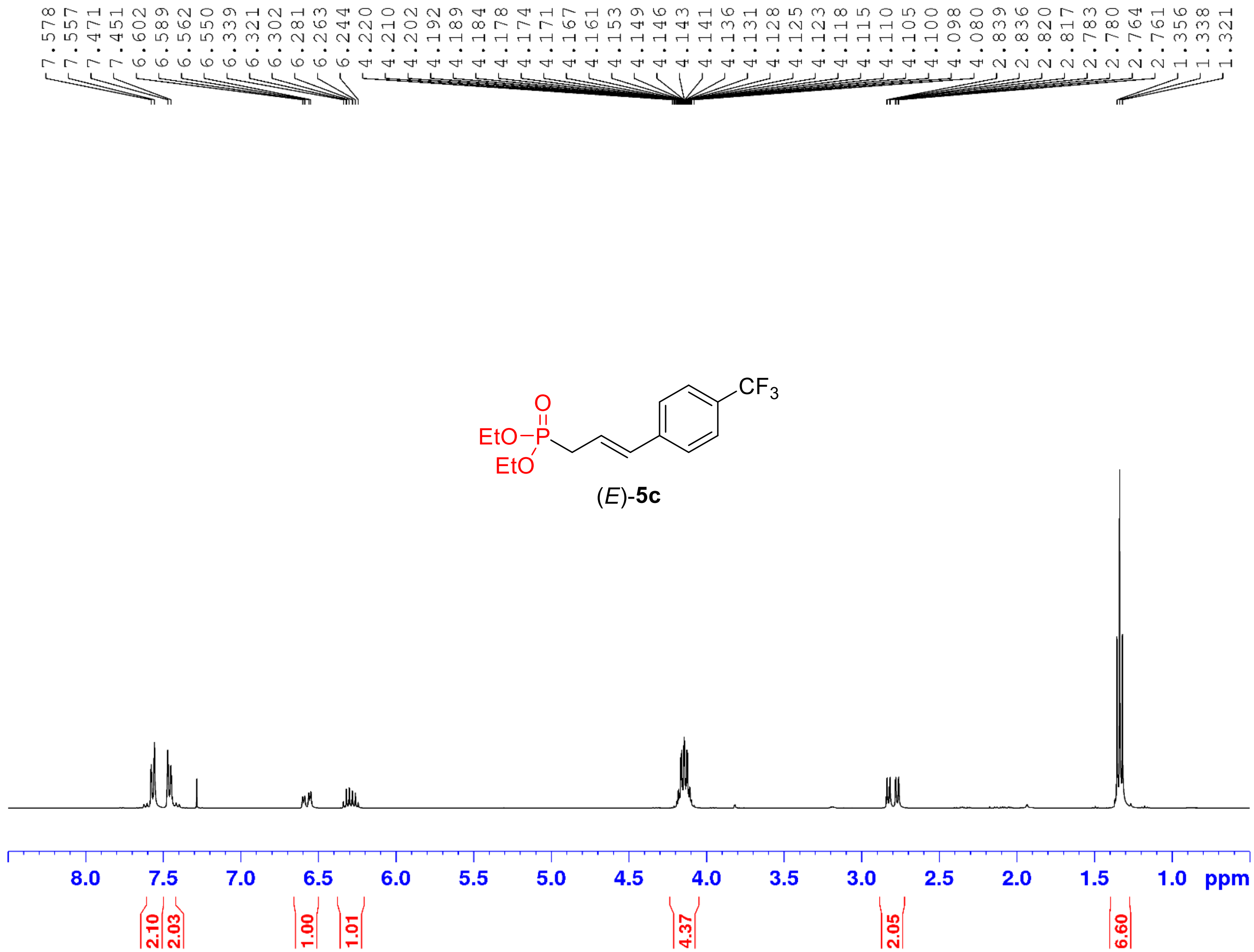
(*E*)-5b: ^{13}C HSQC



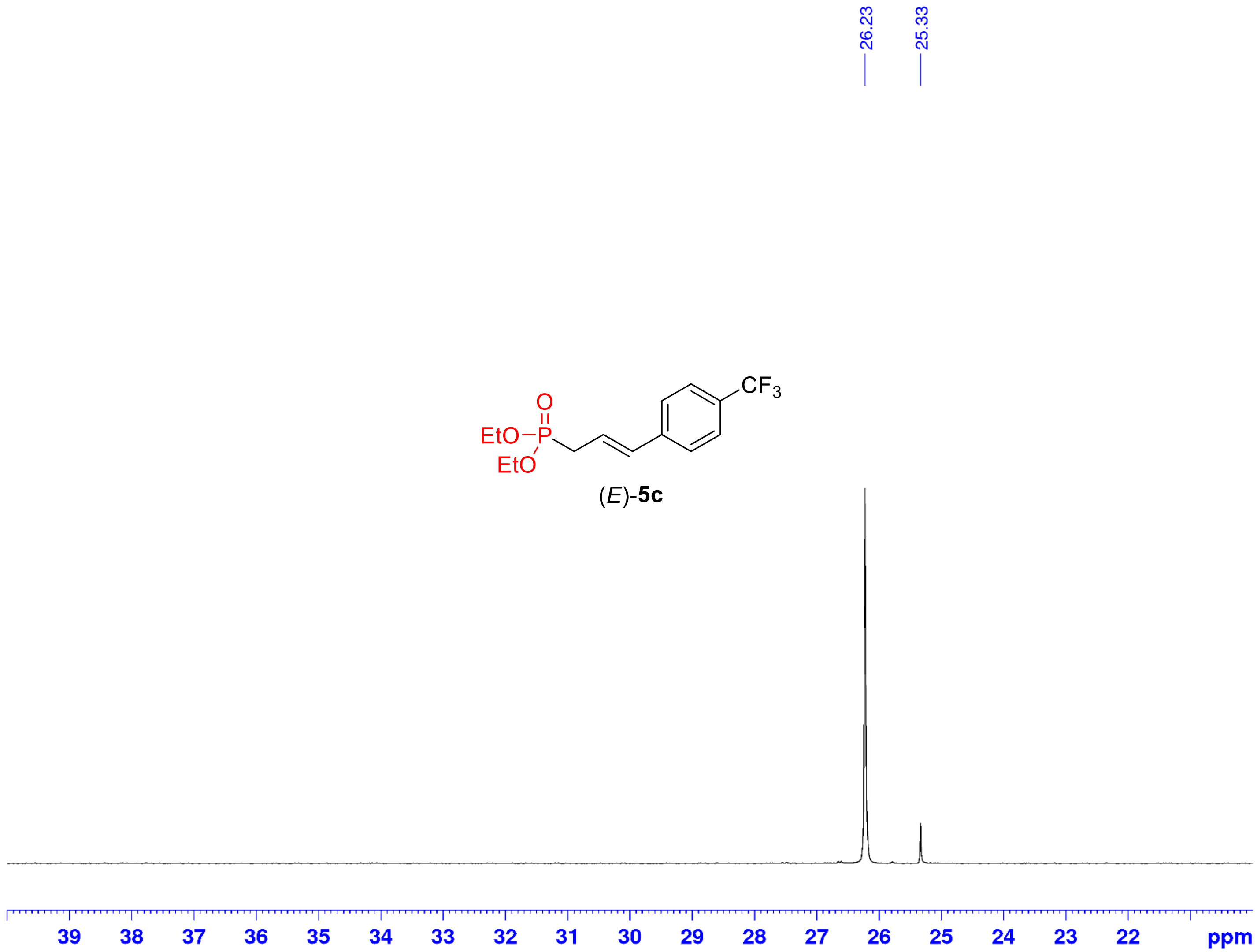


(*E*)-**5b**

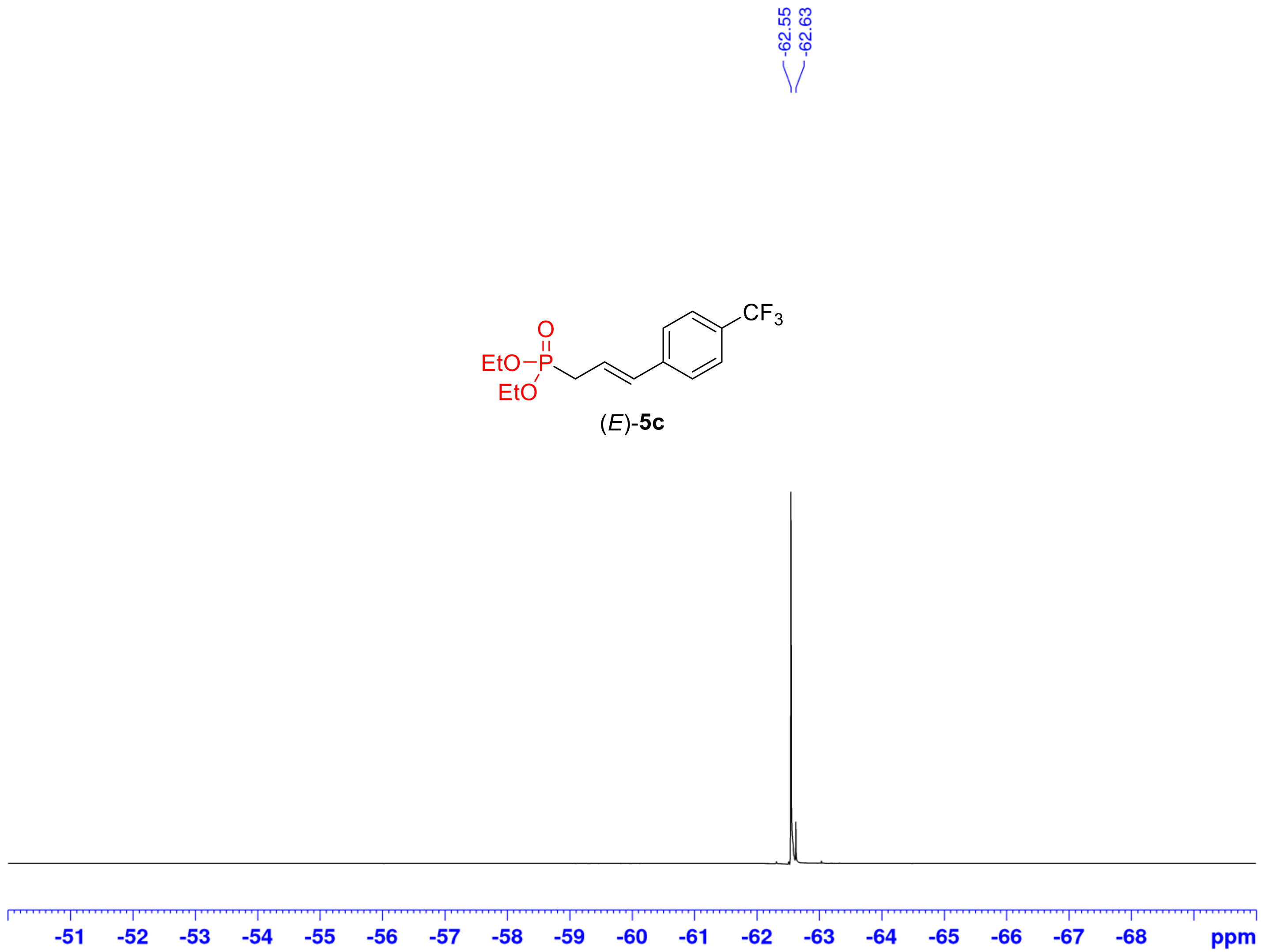
(*E*)-5c: ¹H NMR


7.578
7.557
7.471
7.451
6.602
6.589
6.562
6.550
6.339
6.321
6.302
6.281
6.263
6.244
4.220
4.210
4.202
4.192
4.189
4.184
4.178
4.174
4.171
4.167
4.161
4.153
4.149
4.146
4.143
4.141
4.136
4.131
4.128
4.125
4.123
4.118
4.115
4.110
4.105
4.100
4.098
4.080
2.839
2.836
2.820
2.817
2.783
2.780
2.764
2.761
1.356
1.338
1.321
O
EtO
P
EtO
CF3
(E)-5c
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
ppm
2.10
2.03
1.00
1.01
4.37
2.05
6.60

(*E*)-5c: ^{31}P NMR

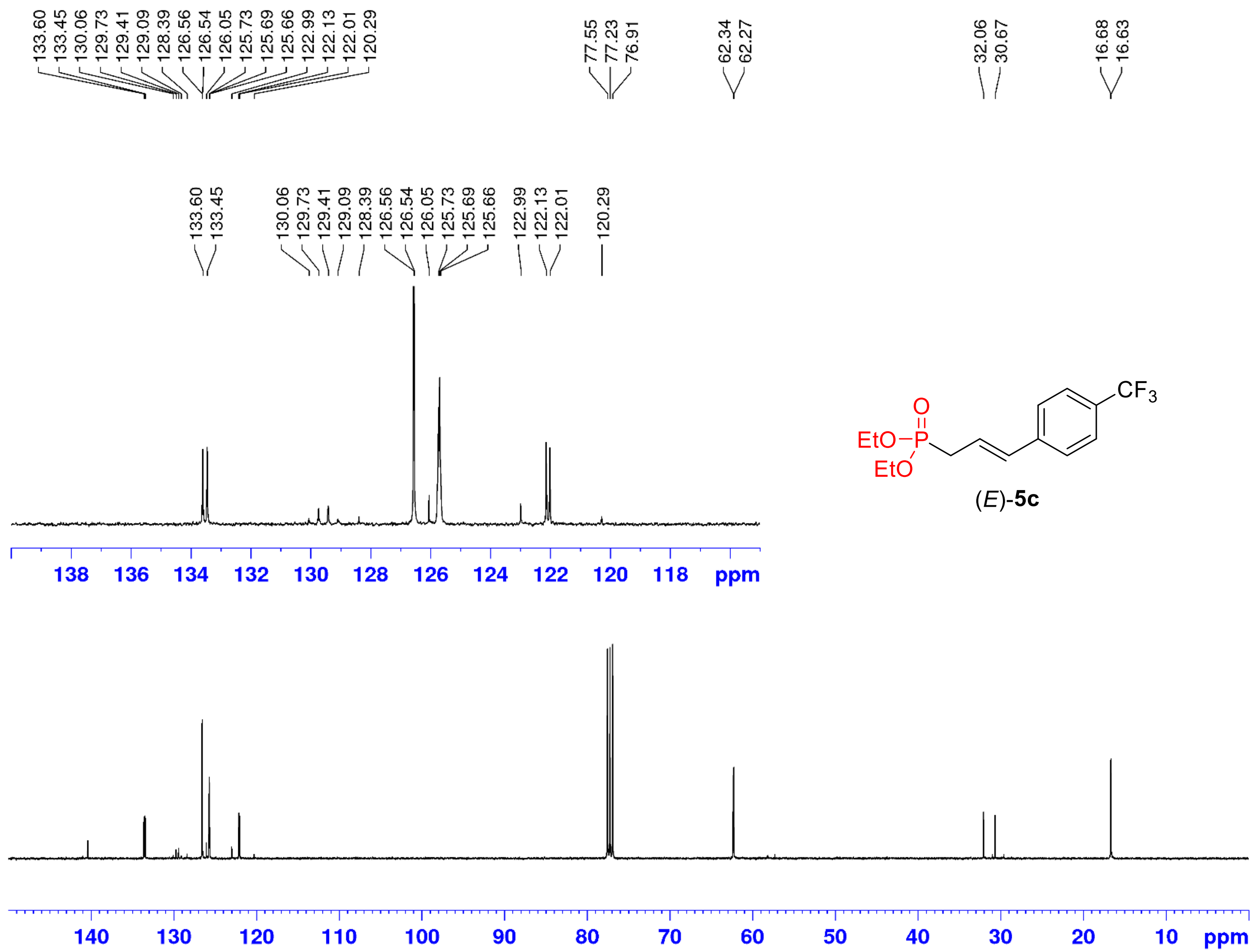

26.23
25.33
O
EtO
P
EtO
CF3
(E)-5c
39
38
37
36
35
34
33
32
31
30
29
28
27
26
25
24
23
22
ppm

(*E*)-5c: ^{19}F NMR



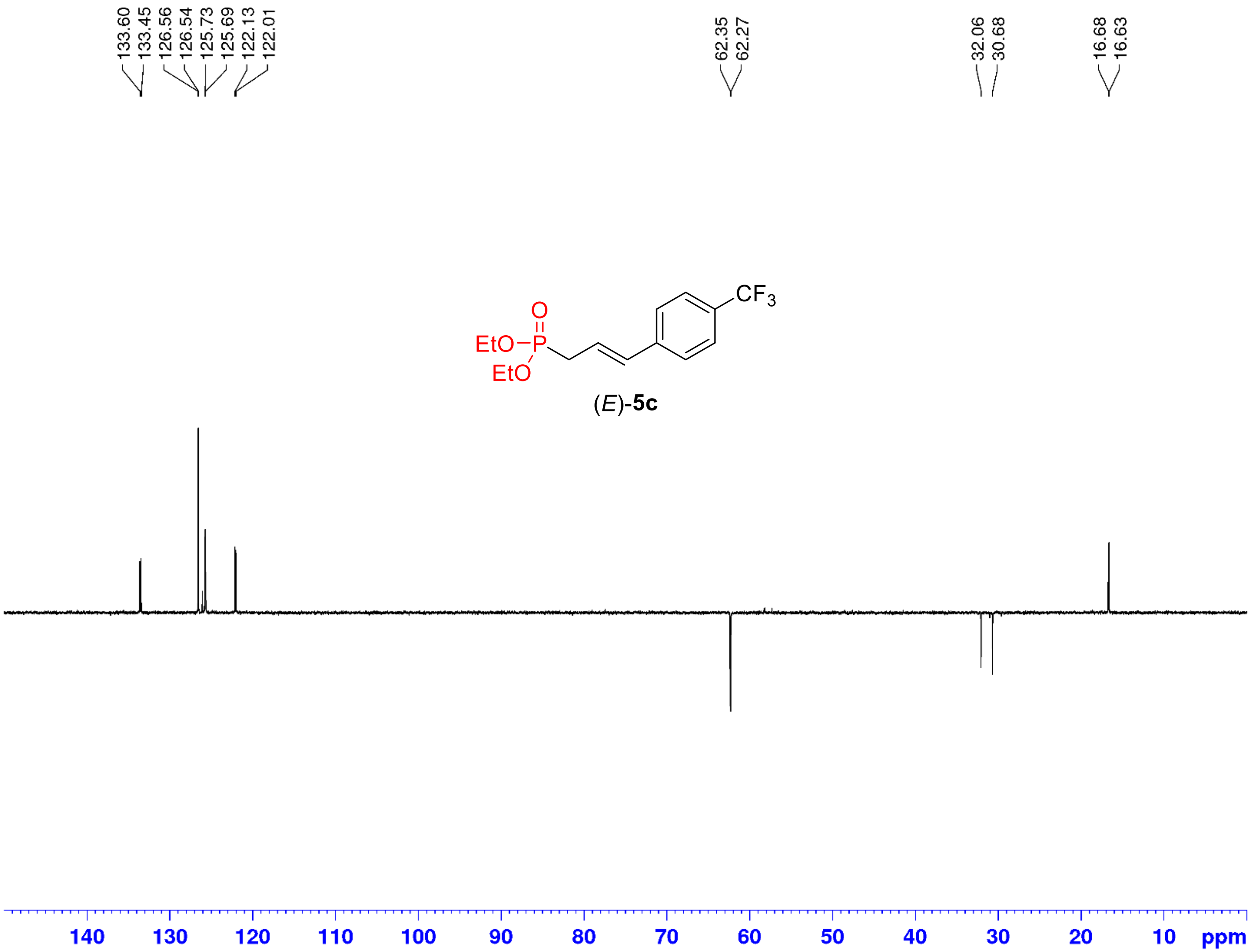
-62.55
-62.63
CF3
O
EtO
P
EtO
(E)-5c
-51 -52 -53 -54 -55 -56 -57 -58 -59 -60 -61 -62 -63 -64 -65 -66 -67 -68
ppm

(*E*)-5c: ^{13}C NMR

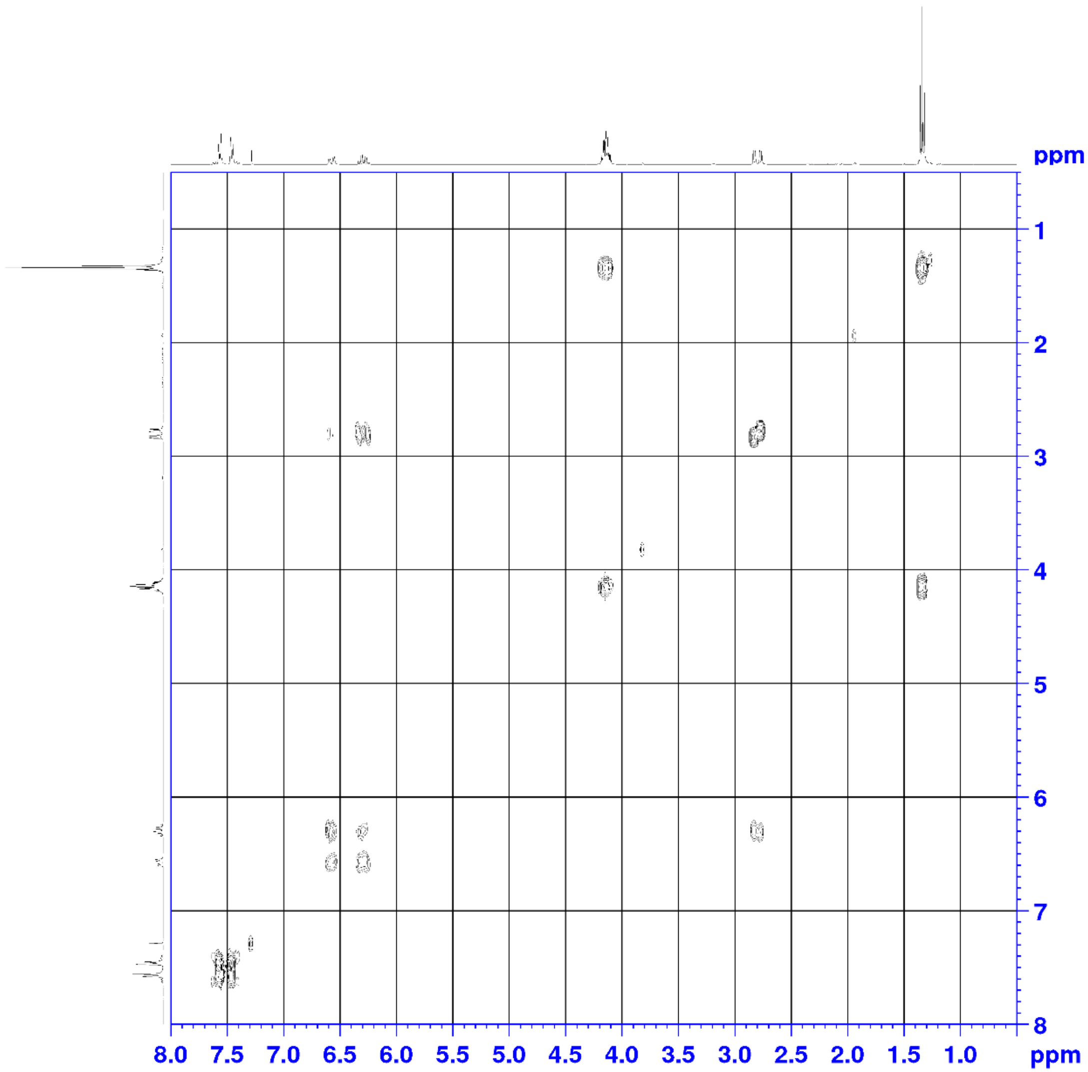


133.60
133.45
130.06
129.73
129.41
129.09
128.39
126.56
126.54
126.05
125.73
125.69
125.66
122.99
122.13
122.01
120.29
77.55
77.23
76.91
62.34
62.27
32.06
30.67
16.68
16.63
EtO
EtO
O
P
CF3
(E)-5c
138
136
134
132
130
128
126
124
122
120
118
ppm
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

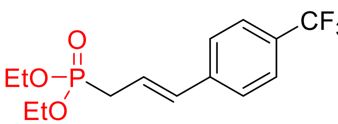
(*E*)-5c: ^{13}C DEPT135



(*E*)-5c: COSY



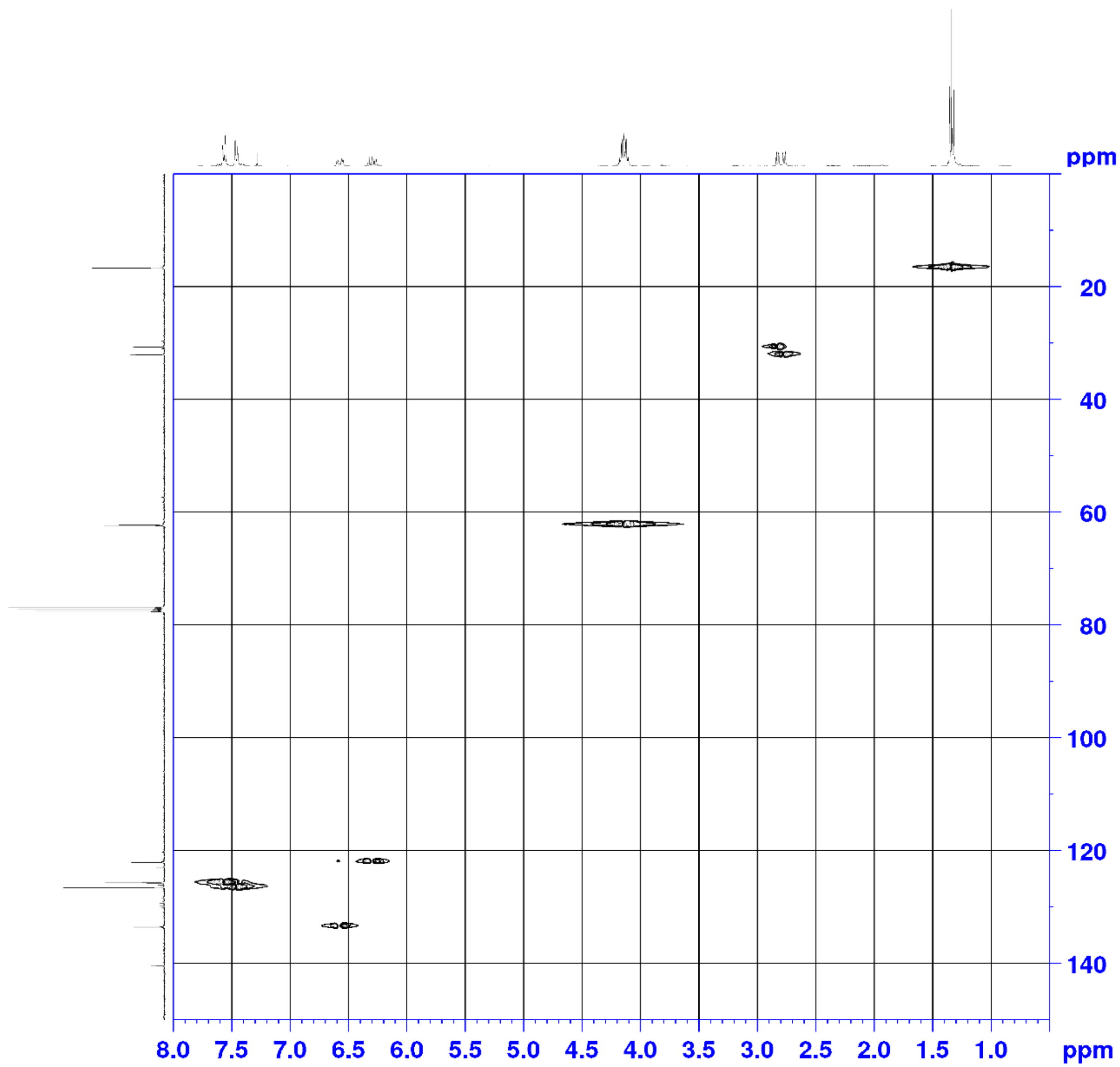
ppm
1
2
3
4
5
6
7
8
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

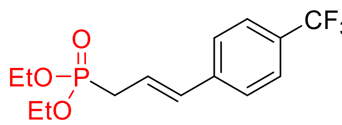


CF3
O
EtO
P
EtO

(*E*)-**5c**

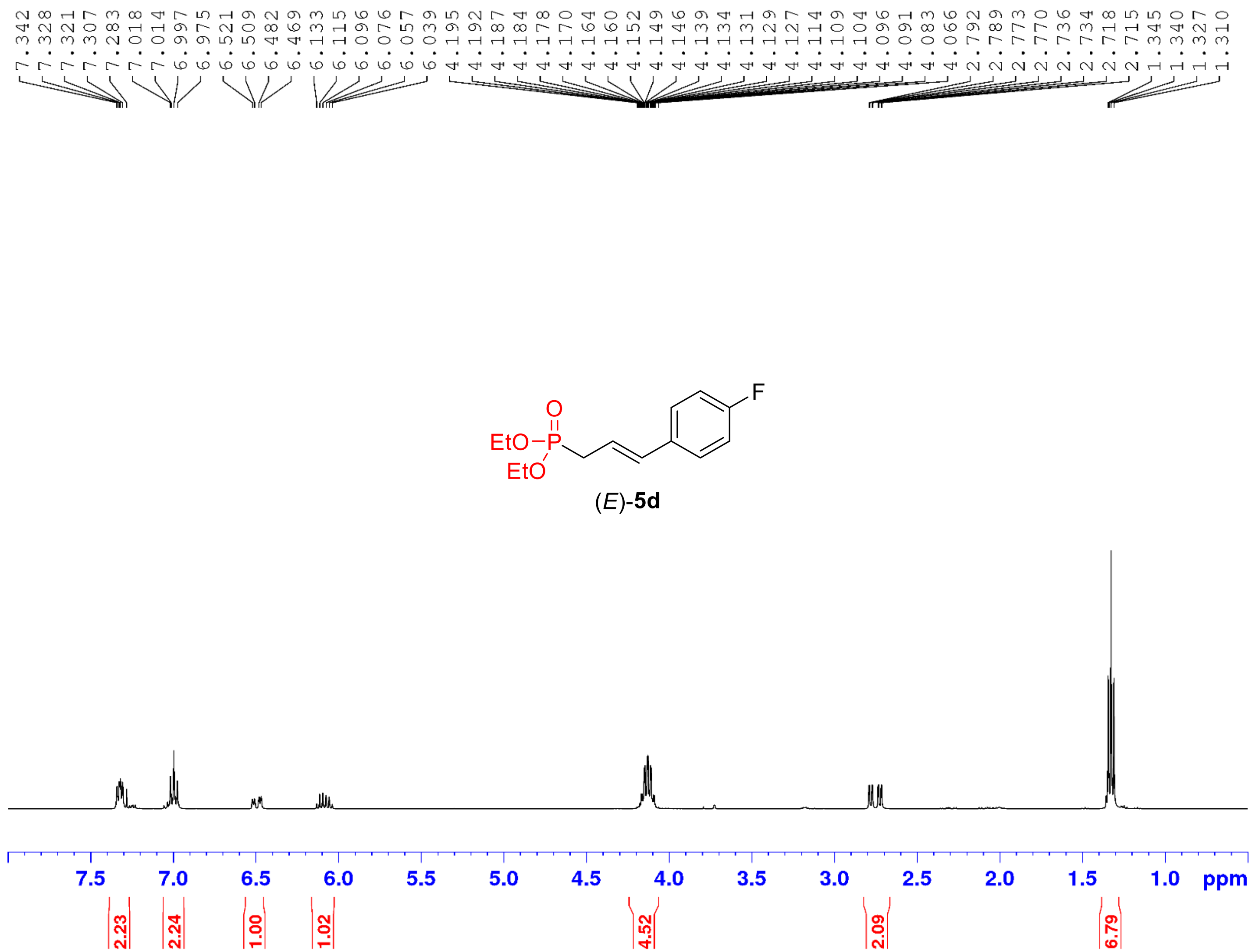
(*E*)-5c: ^{13}C-HSQC





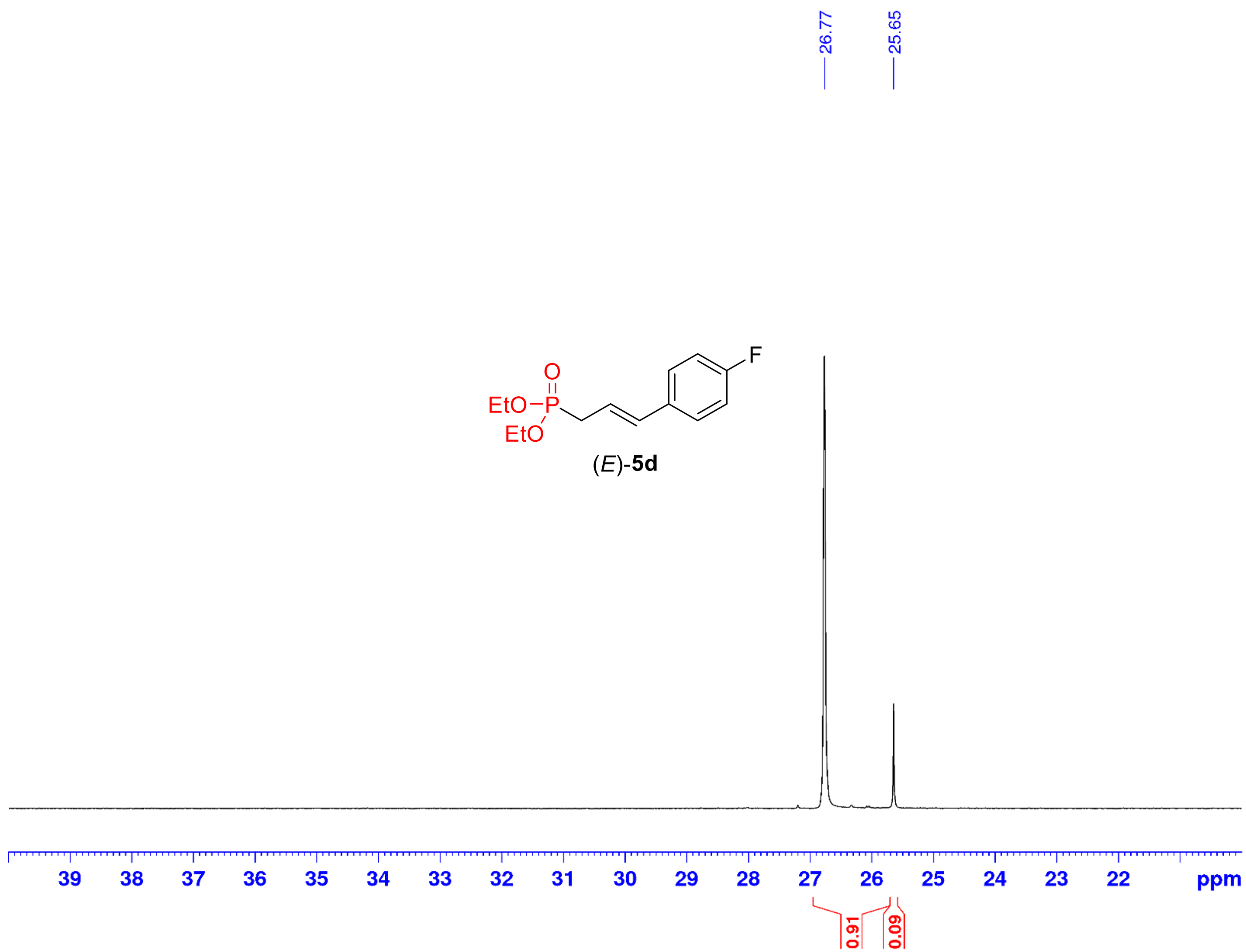
(*E*)-**5c**

(*E*)-5d: [1]H NMR

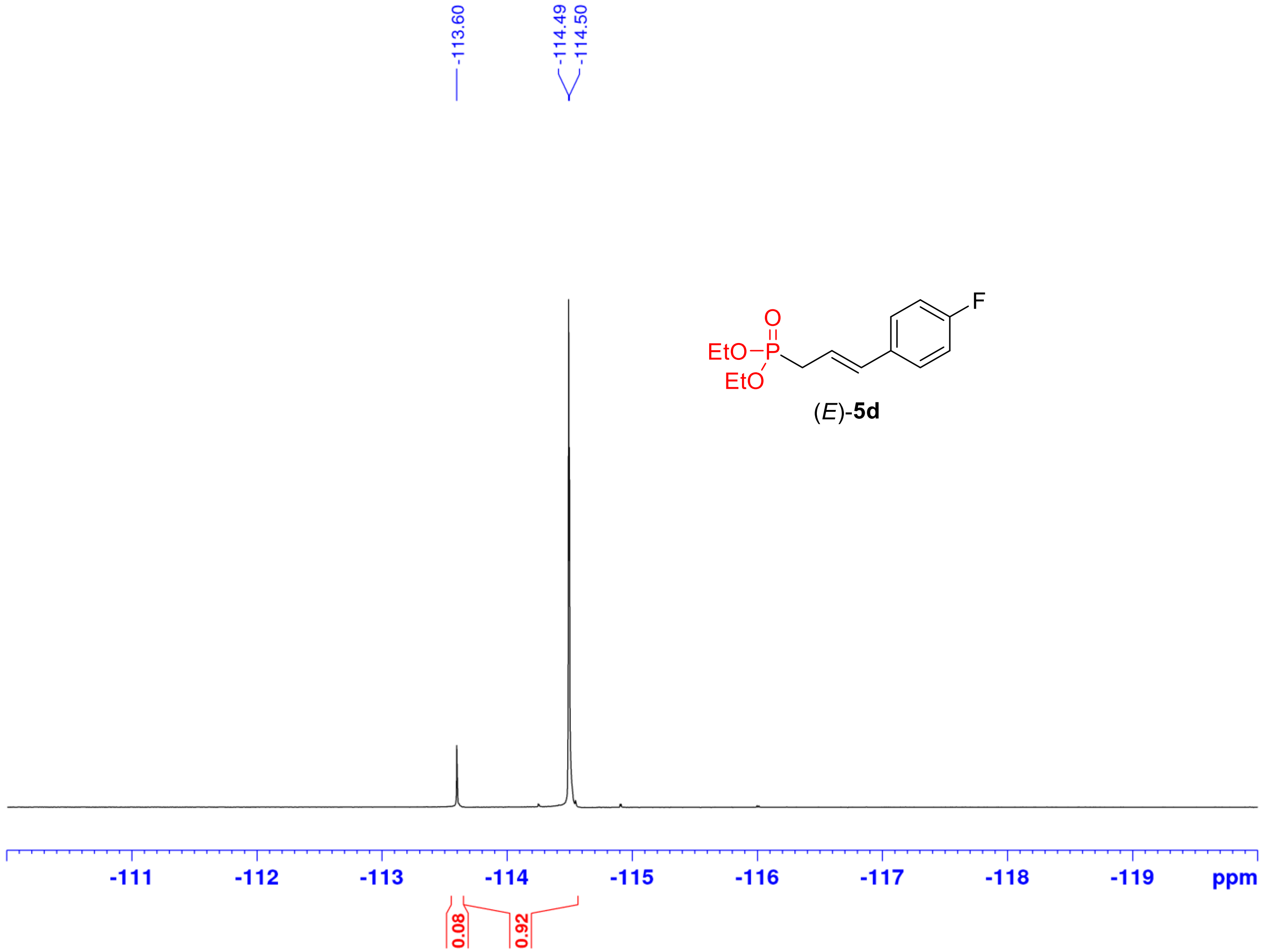


7.342
7.328
7.321
7.307
7.283
7.018
7.014
6.997
6.975
6.521
6.509
6.482
6.469
6.133
6.115
6.096
6.076
6.057
6.039
4.195
4.192
4.187
4.184
4.178
4.170
4.164
4.160
4.152
4.149
4.146
4.139
4.134
4.131
4.129
4.127
4.114
4.109
4.104
4.096
4.091
4.083
4.066
2.792
2.789
2.773
2.770
2.736
2.734
2.718
2.715
1.345
1.340
1.327
1.310
(*E*)-**5d**
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
2.23
2.24
1.00
1.02
4.52
2.09
6.79

(*E*)-5d: ^{31}P NMR

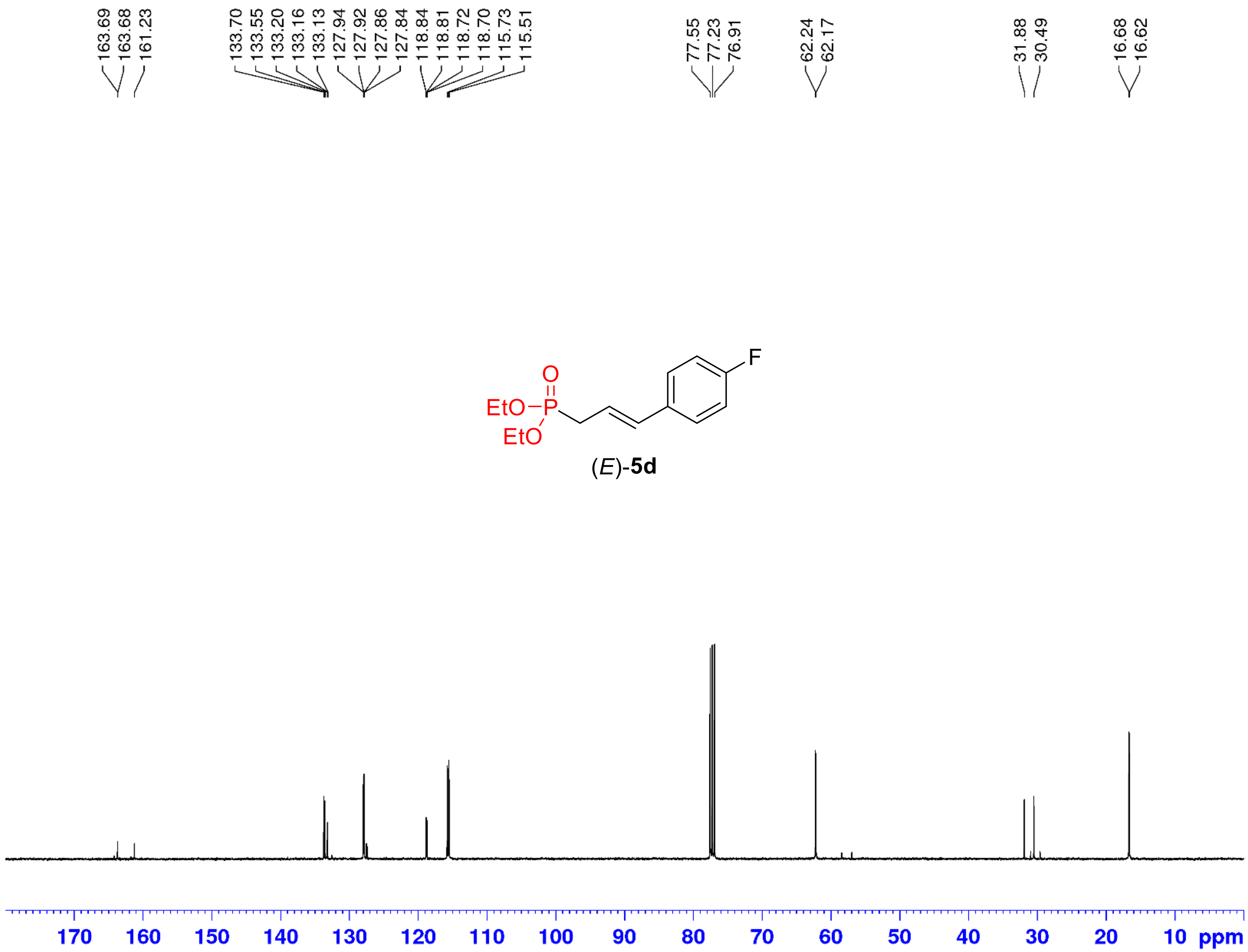

26.77
25.65
O
EtO
P
EtO
F
(E)-5d
39
38
37
36
35
34
33
32
31
30
29
28
27
26
25
24
23
22
ppm
0.91
0.09

(*E*)-5d: ^{19}F NMR

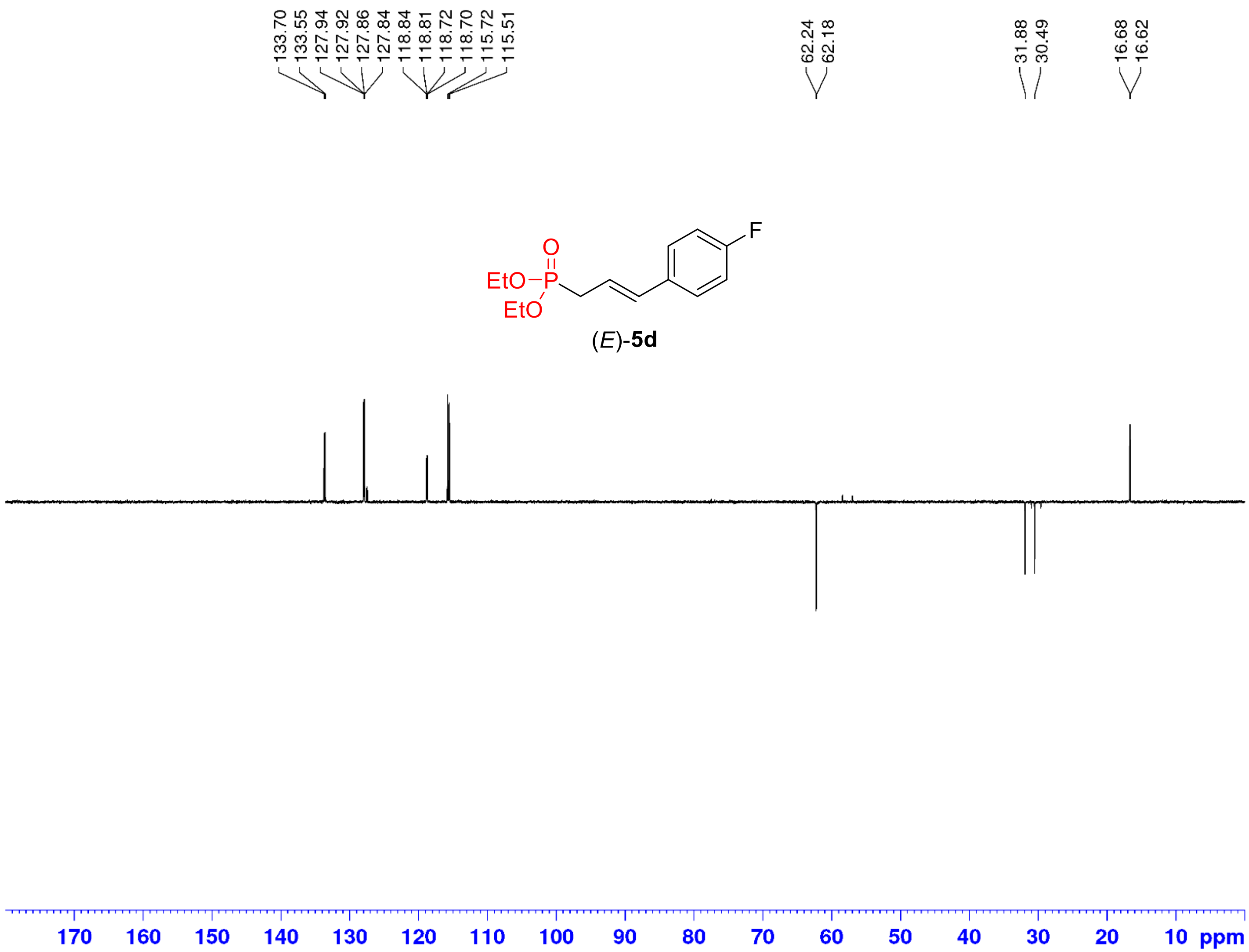


-113.60
-114.49
-114.50
O
EtO
P
EtO
F
(*E*)-**5d**
-111
-112
-113
-114
-115
-116
-117
-118
-119
ppm
0.08
0.92

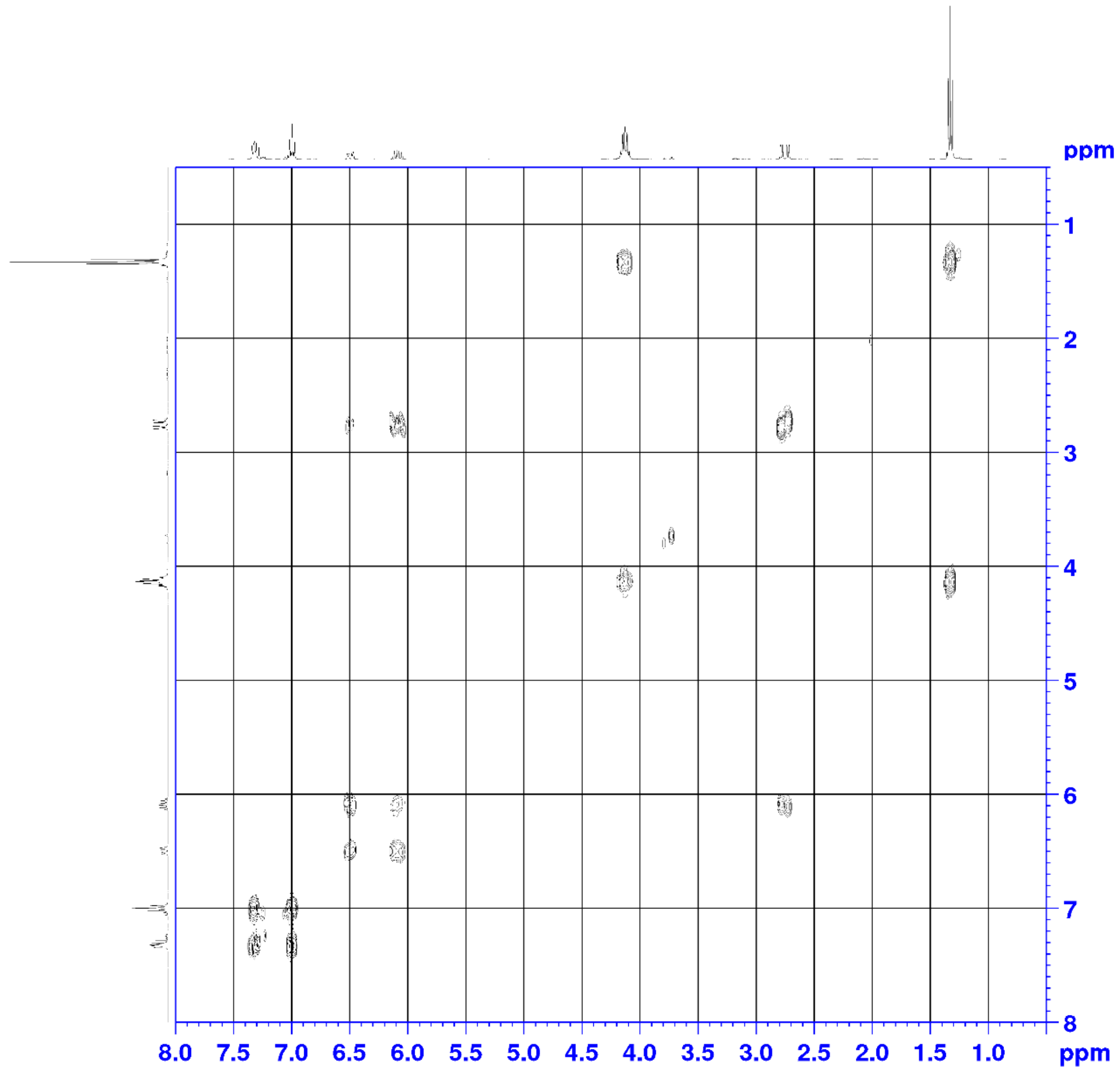
(*E*)-5d: ^{13}C NMR

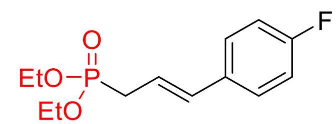

163.69
163.68
161.23
133.70
133.55
133.20
133.16
133.13
127.94
127.92
127.86
127.84
118.84
118.81
118.72
118.70
115.73
115.51
77.55
77.23
76.91
62.24
62.17
31.88
30.49
16.68
16.62
O
EtO
P
EtO
F
(E)-5d
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

(*E*)-5d: ^{13}C DEPT135



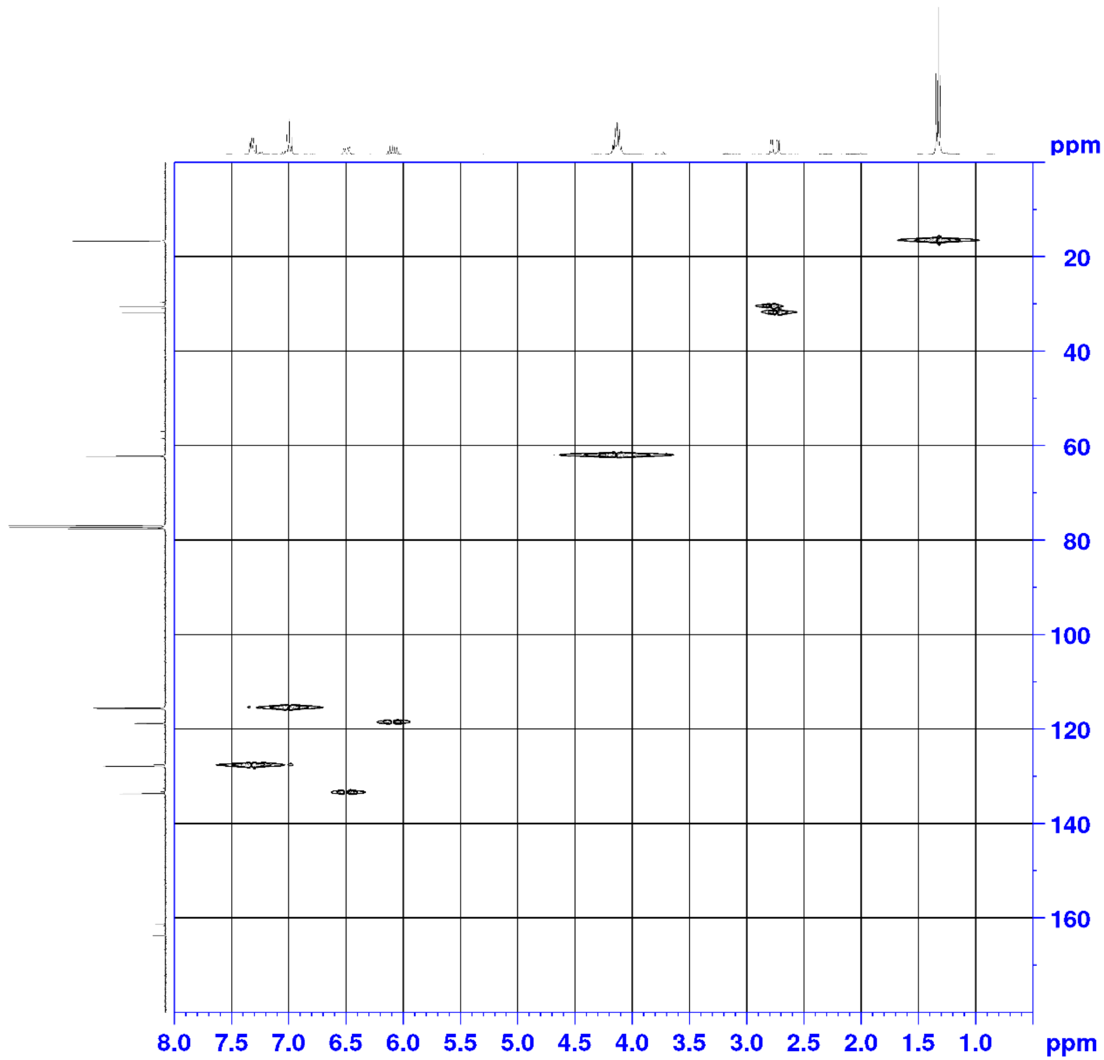
(*E*)-5d: COSY

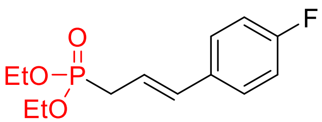

ppm
1
2
3
4
5
6
7
8
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0
ppm


O
EtO
P
EtO
F

(*E*)-**5d**

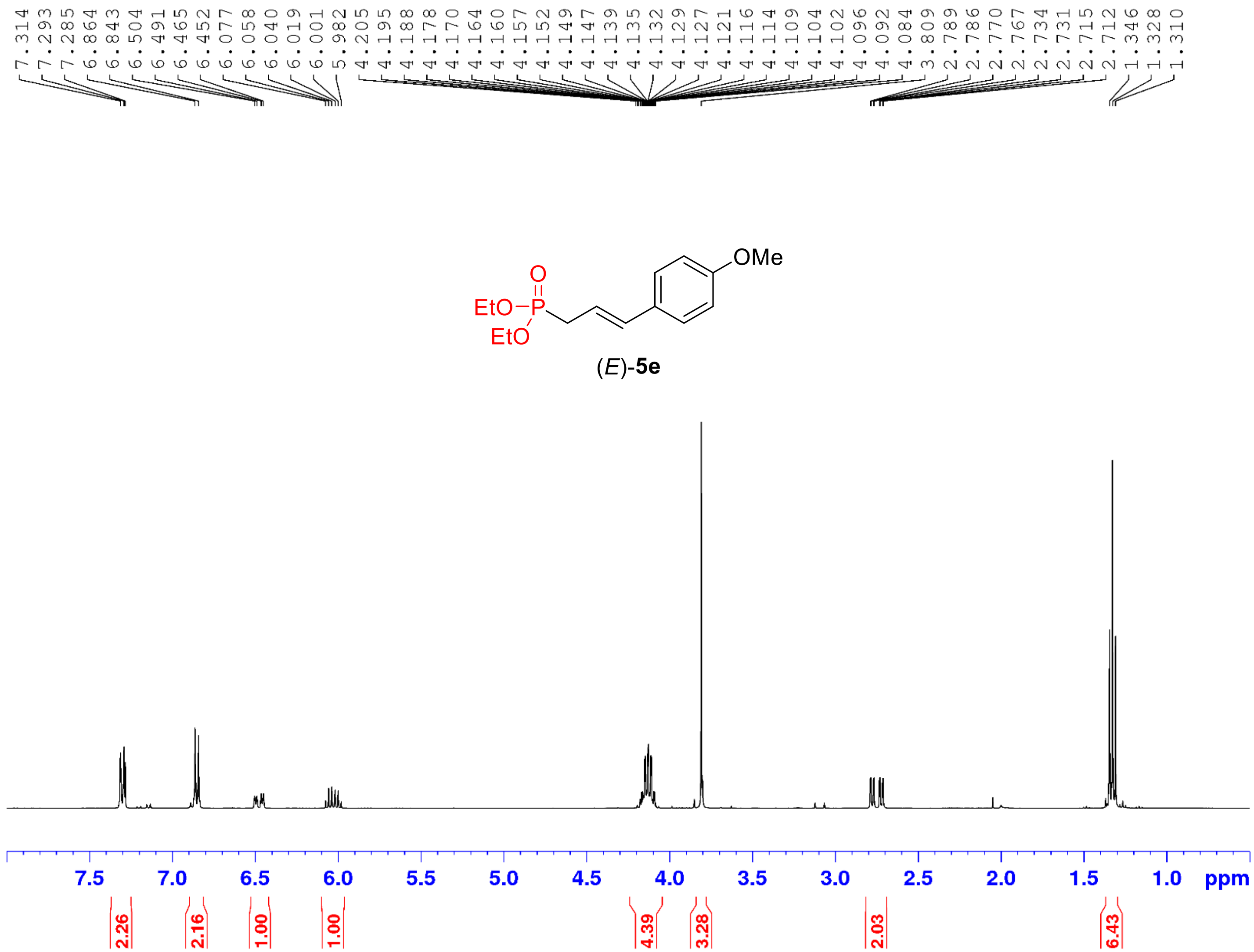
(*E*)-5d: ^{13}C-HSQC





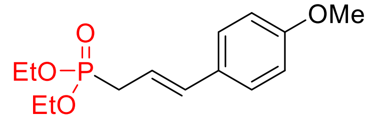
(*E*)-**5d**

(E)-5e: ^{1}H NMR

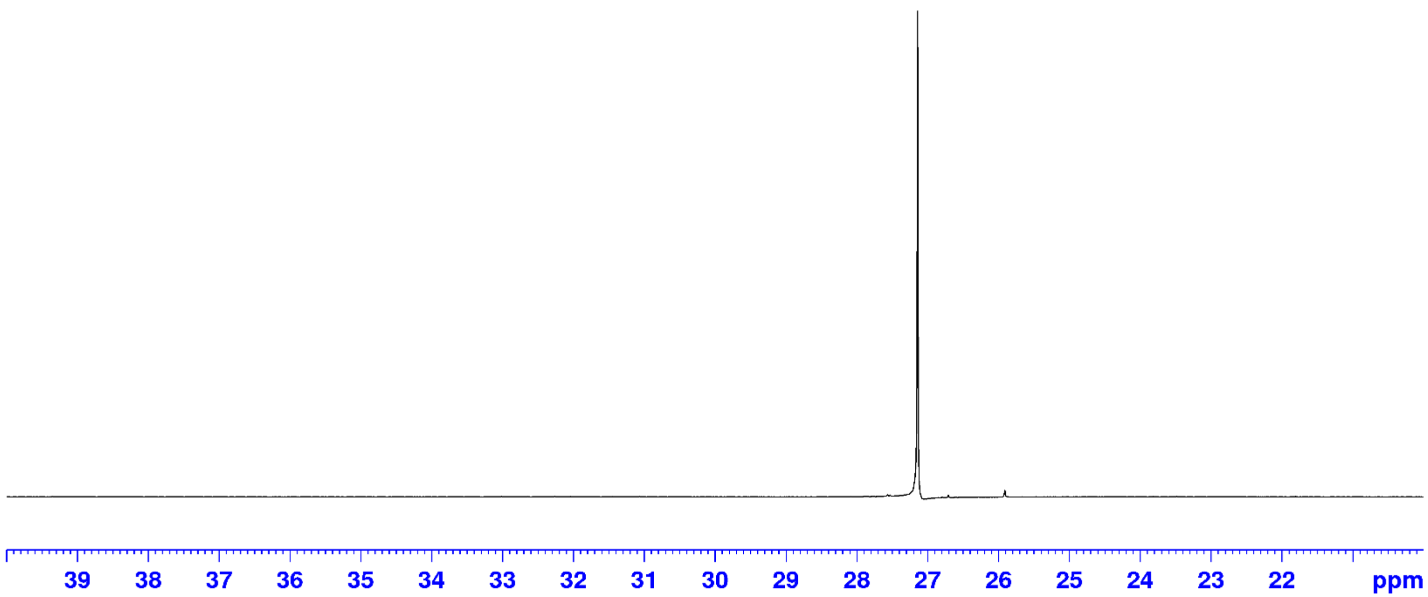


7.314
7.293
7.285
6.864
6.843
6.504
6.491
6.465
6.452
6.077
6.058
6.040
6.019
6.001
5.982
4.205
4.195
4.188
4.178
4.170
4.164
4.160
4.157
4.152
4.149
4.147
4.139
4.135
4.132
4.129
4.127
4.121
4.116
4.114
4.109
4.104
4.102
4.096
4.092
4.084
3.809
2.789
2.786
2.770
2.767
2.734
2.731
2.715
2.712
1.346
1.328
1.310
O
EtO
P
EtO
OMe
(E)-5e
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
2.26
2.16
1.00
1.00
4.39
3.28
2.03
6.43

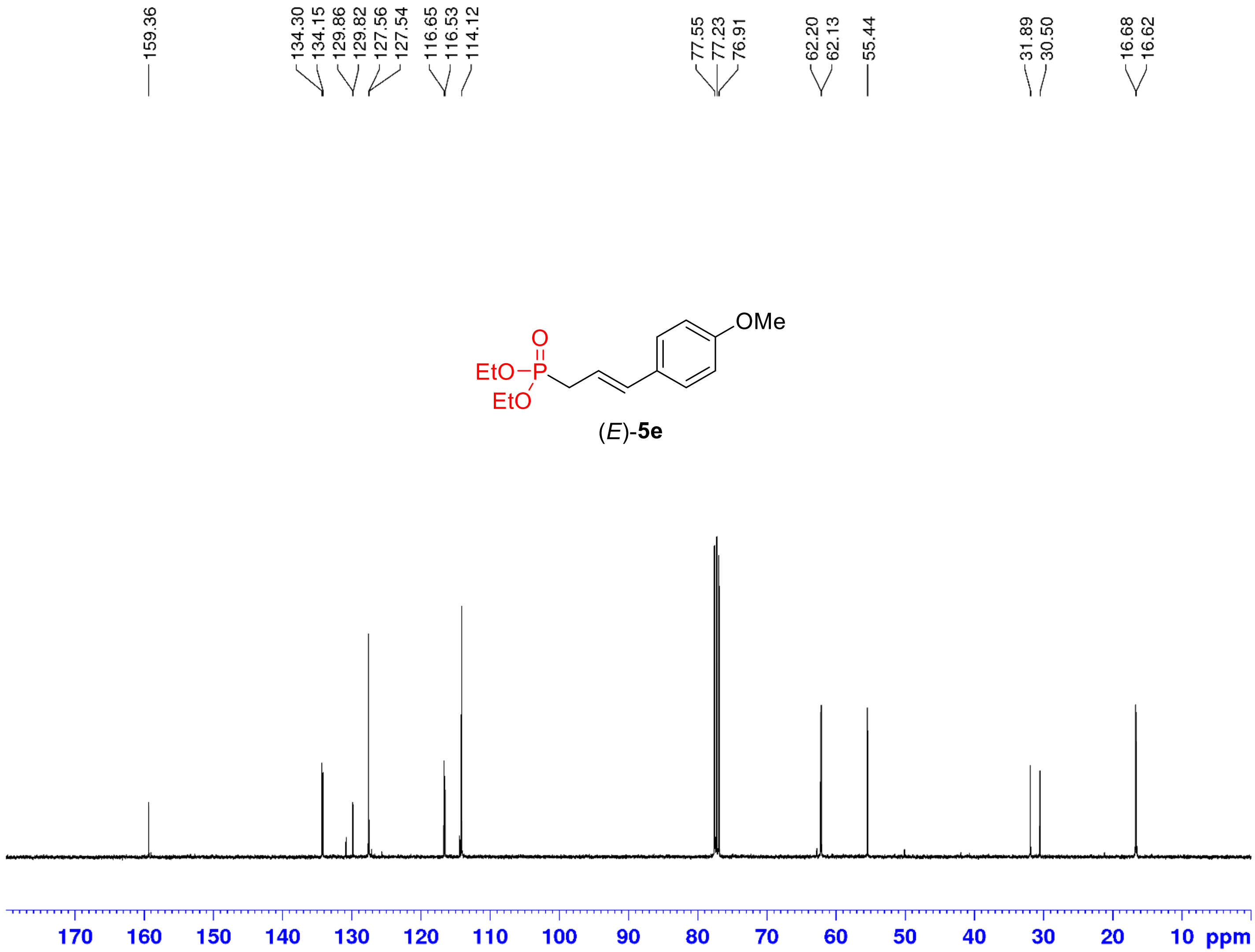
(*E*)-5e: ^{31}P NMR



(*E*)-**5e**

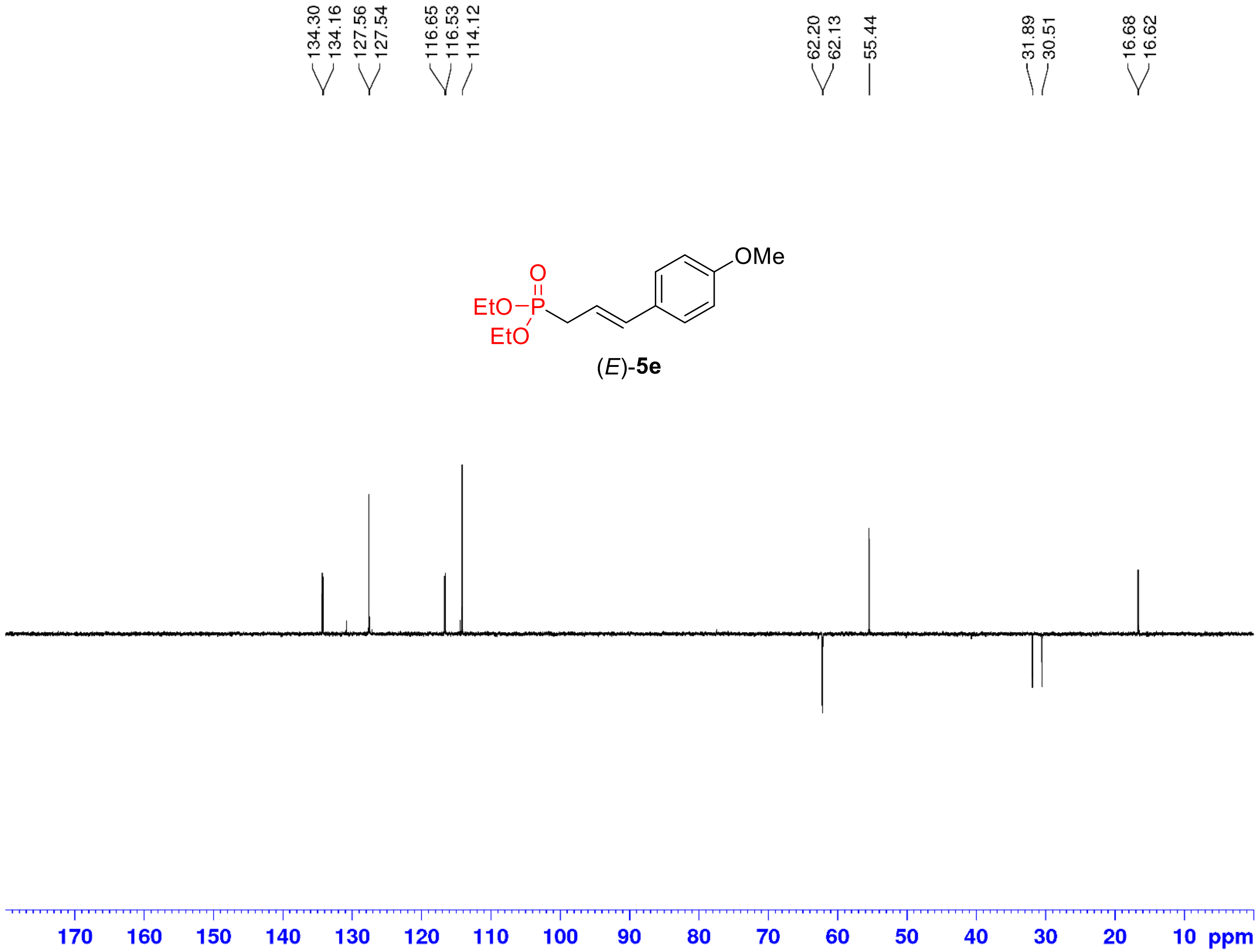


(*E*)-5e: ^{13}C NMR

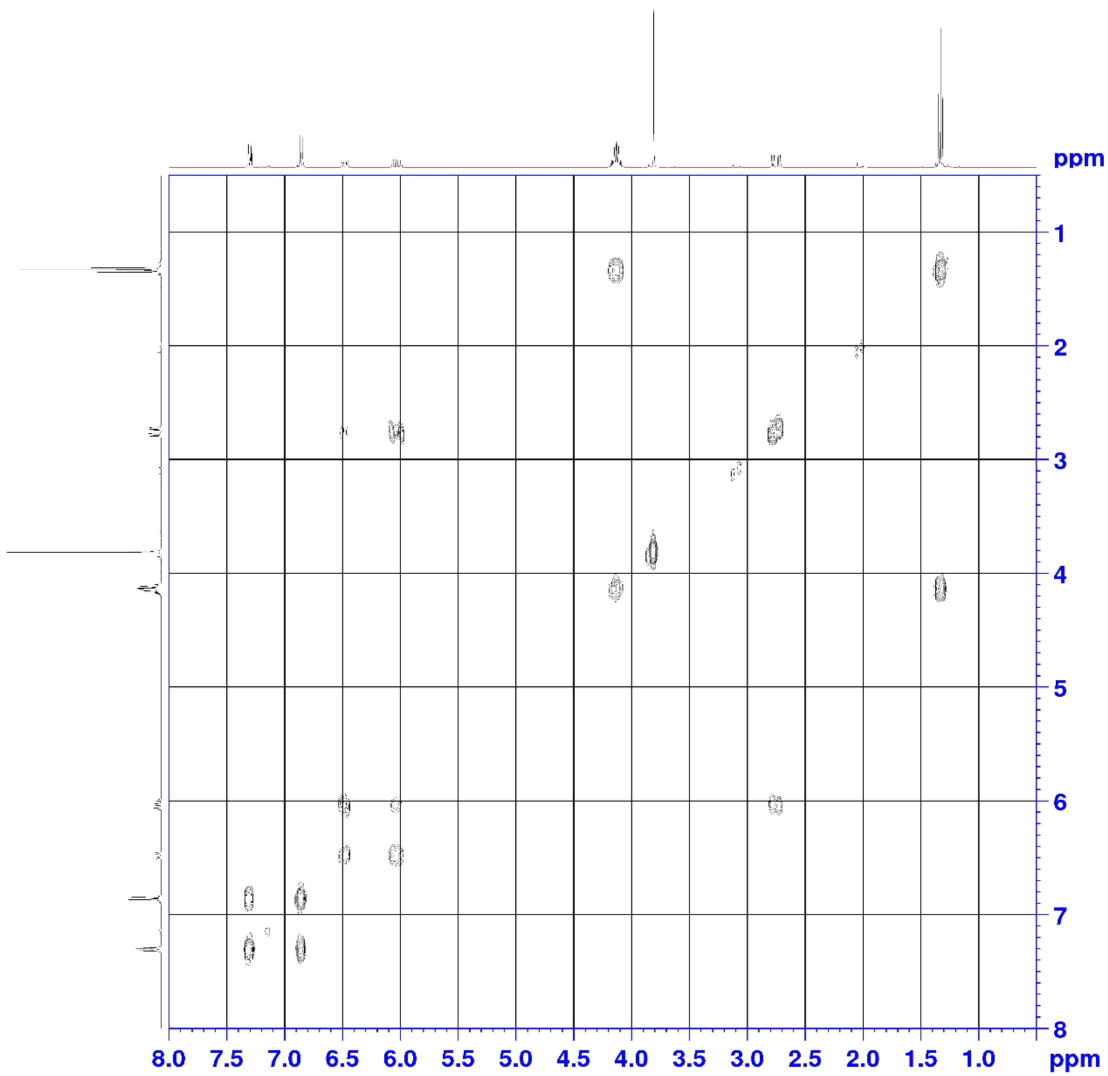


159.36
134.30
134.15
129.86
129.82
127.56
127.54
116.65
116.53
114.12
77.55
77.23
76.91
62.20
62.13
55.44
31.89
30.50
16.68
16.62
OMe
O
EtO
P
EtO
(E)-5e
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

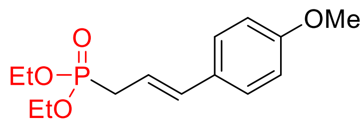
(*E*)-5e: ^{13}C DEPT135



(*E*)-5e: COSY



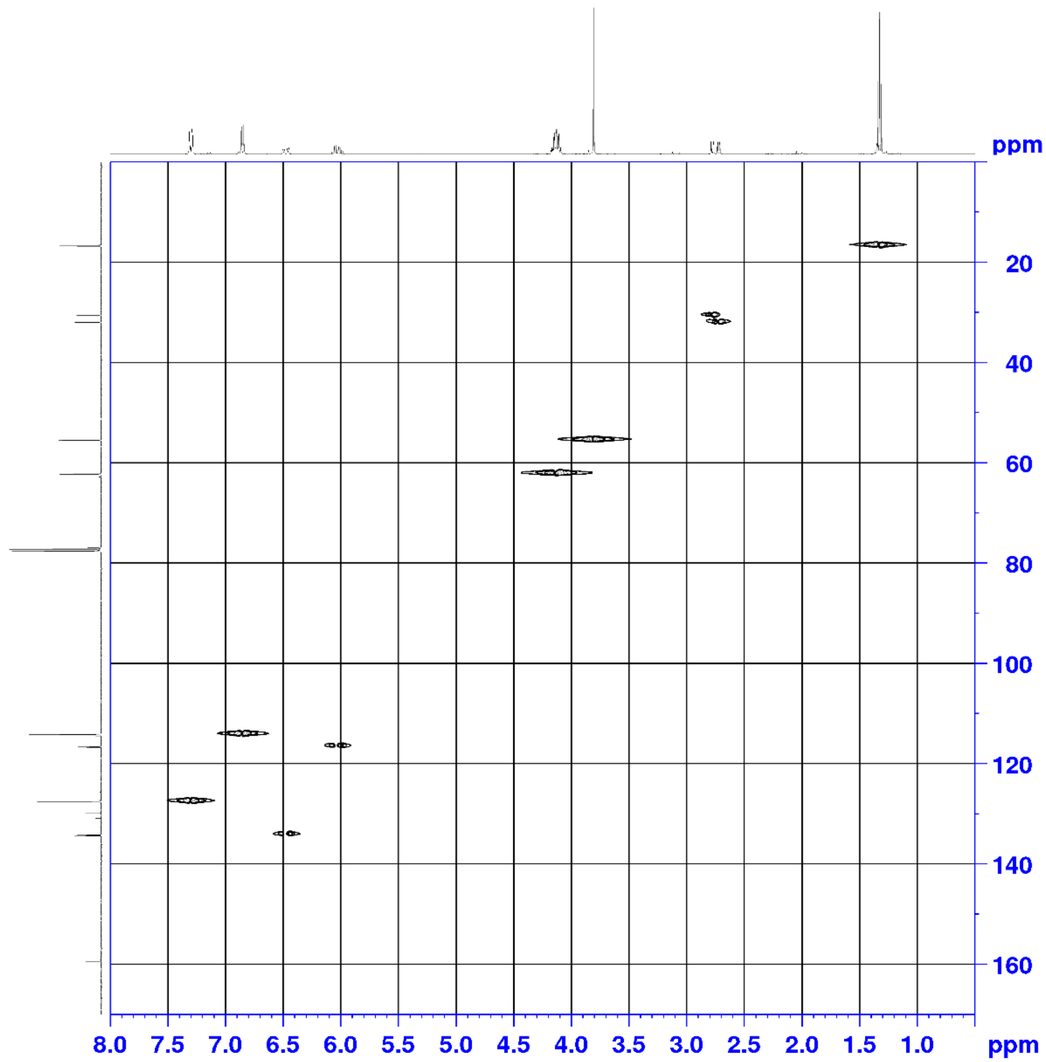
ppm
1
2
3
4
5
6
7
8
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

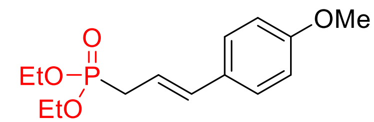


OMe
O
EtO
P
EtO

(*E*)-**5e**

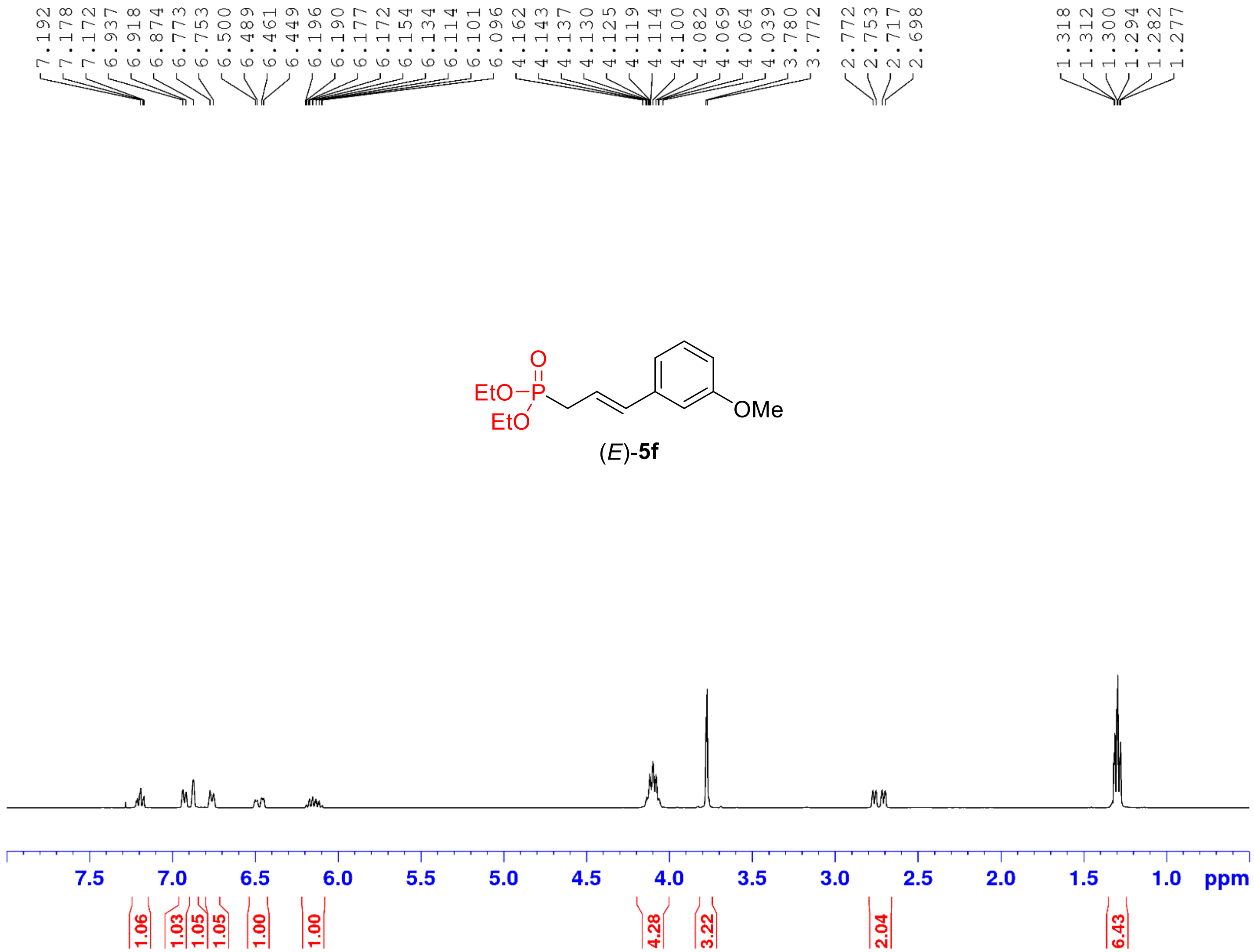
(*E*)-5e: ^{13}C-HSQC





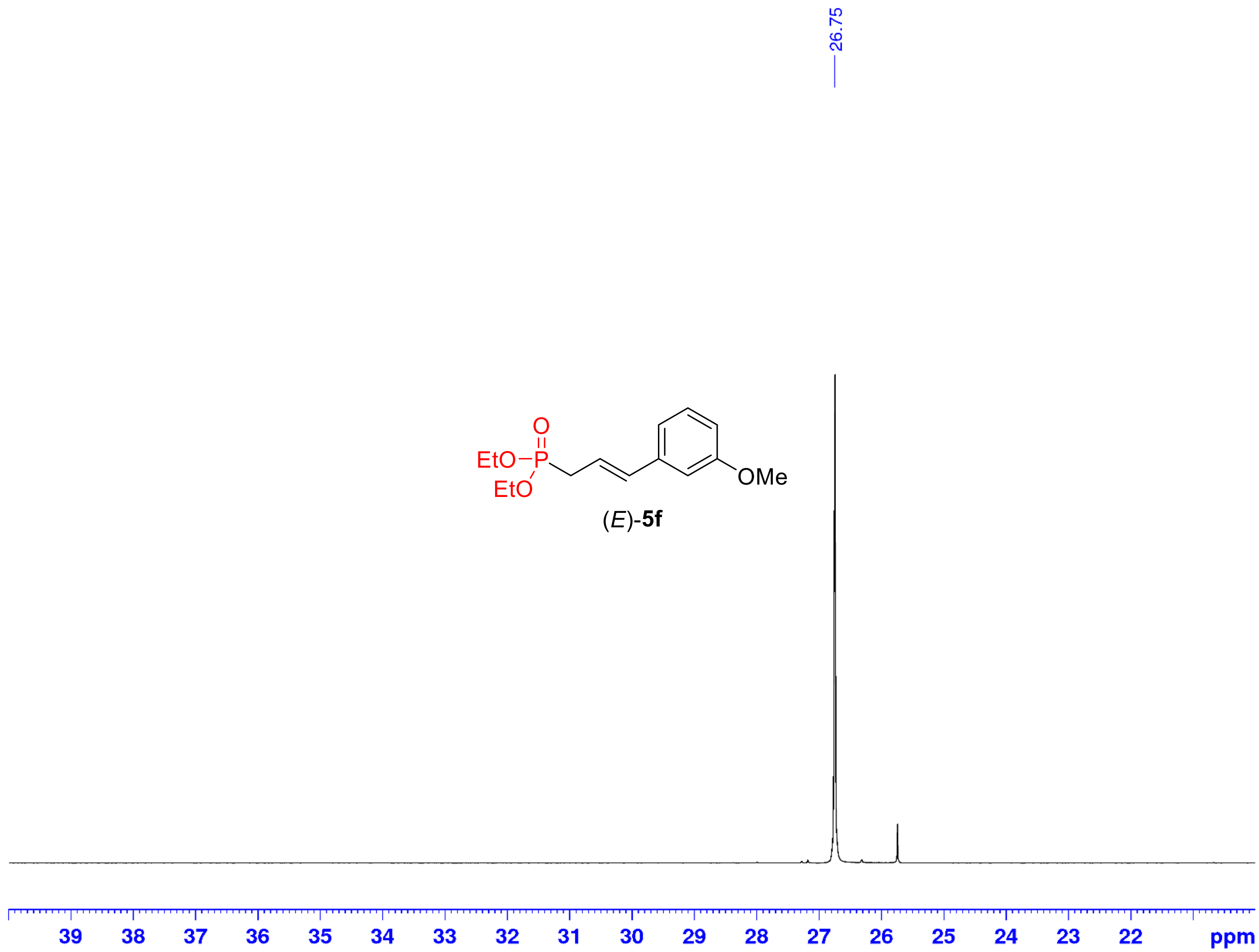
(*E*)-**5e**

(*E*)-5f: ^{1}H NMR

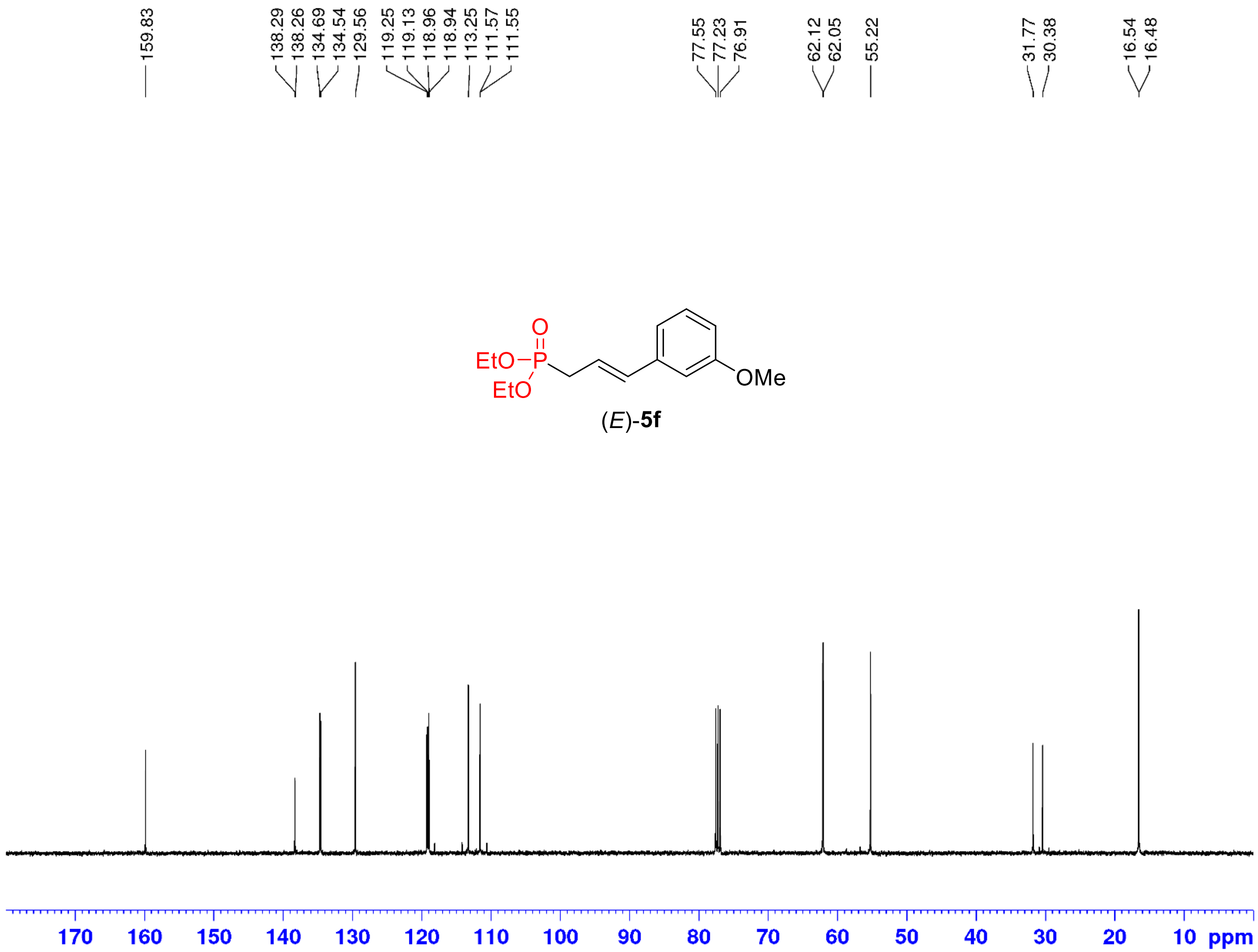


(*E*)-**5f**

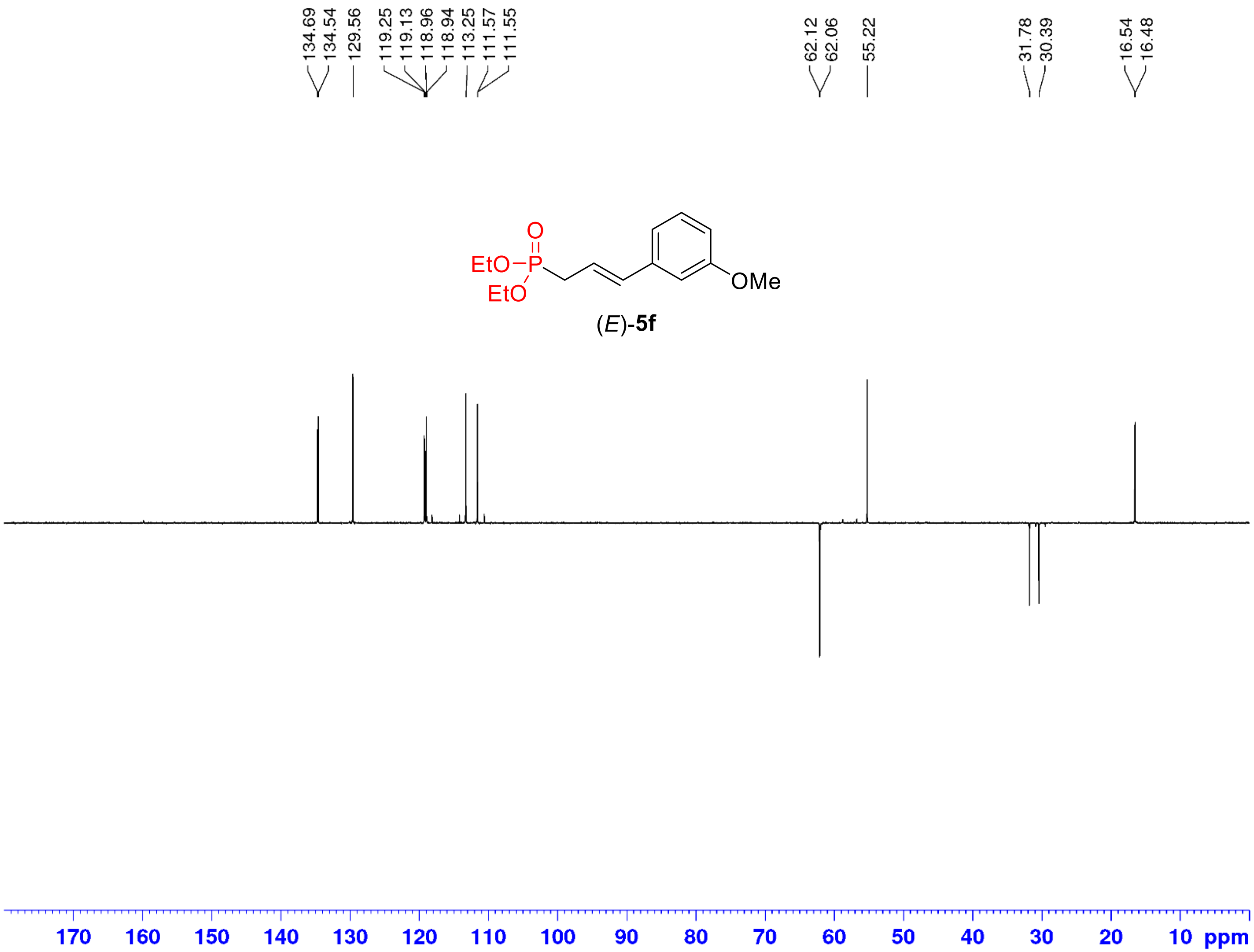
(*E*)-5f: ^{31}P NMR


26.75
EtO
EtO
O
P
OMe
(*E*)-**5f**
39
38
37
36
35
34
33
32
31
30
29
28
27
26
25
24
23
22
ppm

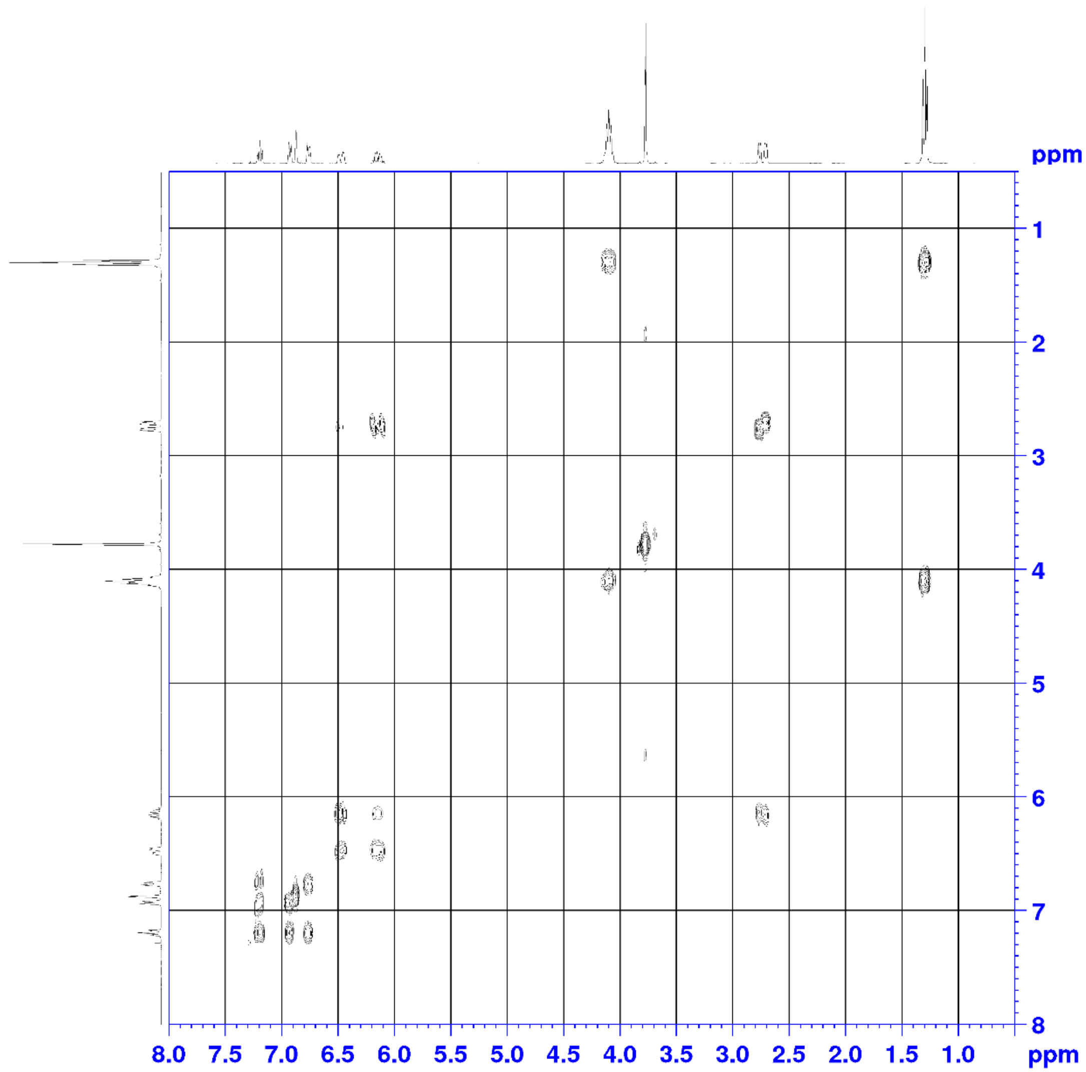
(*E*)-5f: ^{13}C NMR


159.83
138.29
138.26
134.69
134.54
129.56
119.25
119.13
118.96
118.94
113.25
111.57
111.55
77.55
77.23
76.91
62.12
62.05
55.22
31.77
30.38
16.54
16.48
O
EtO
P
EtO
OMe
(E)-5f
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

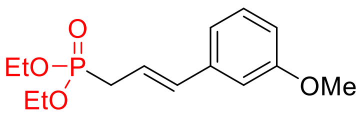
(*E*)-5f: ^{13}C-DEPT135



(*E*)-5f: COSY



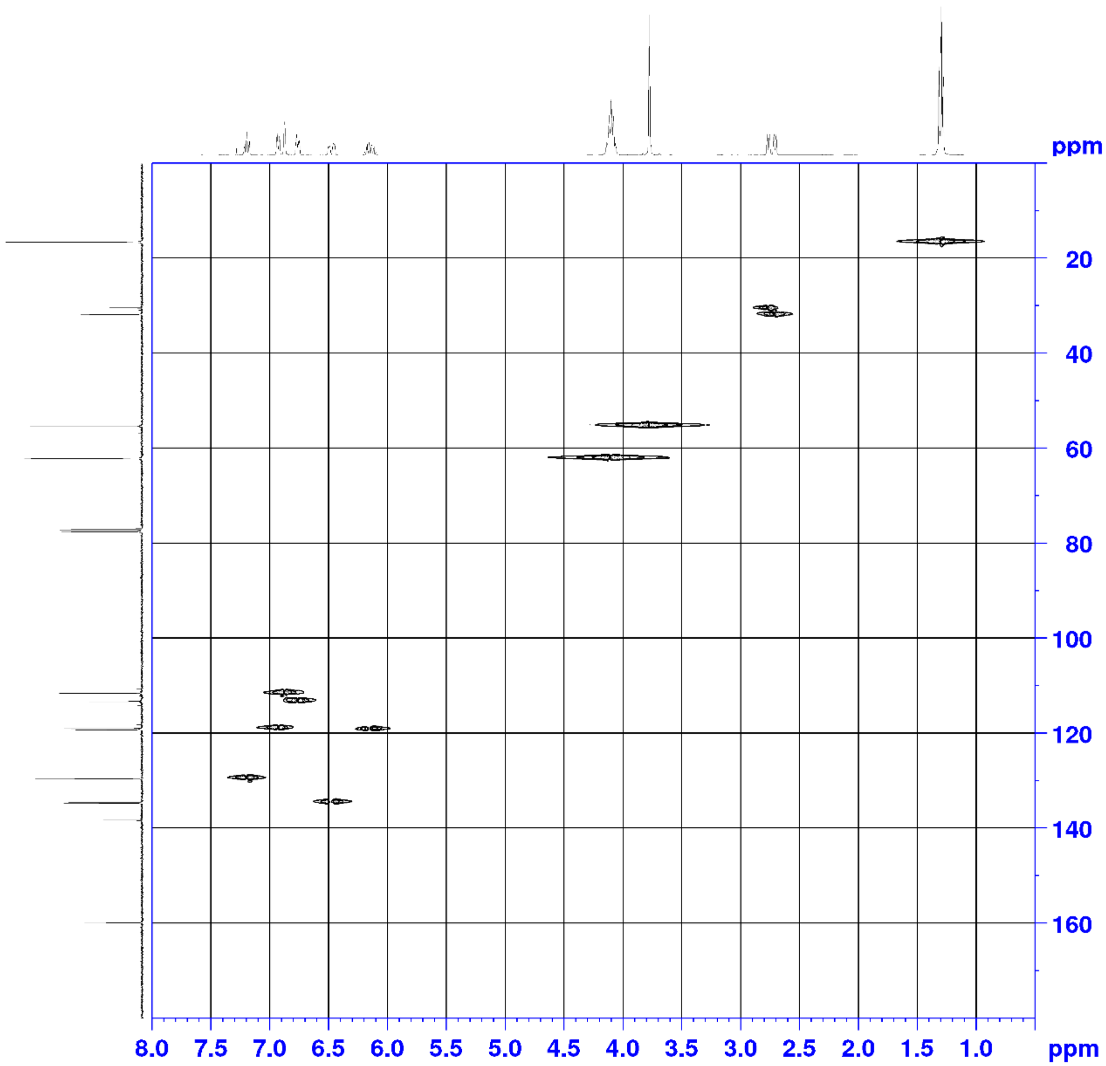
ppm
1
2
3
4
5
6
7
8
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

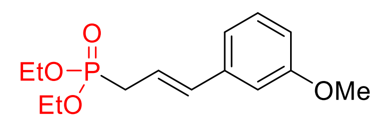


O
EtO
P
EtO
OMe

(*E*)-**5f**

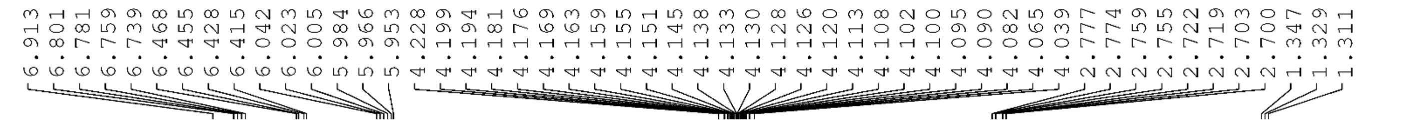
(*E*)-5f: ^{13}C-HSQC

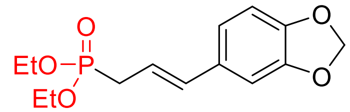




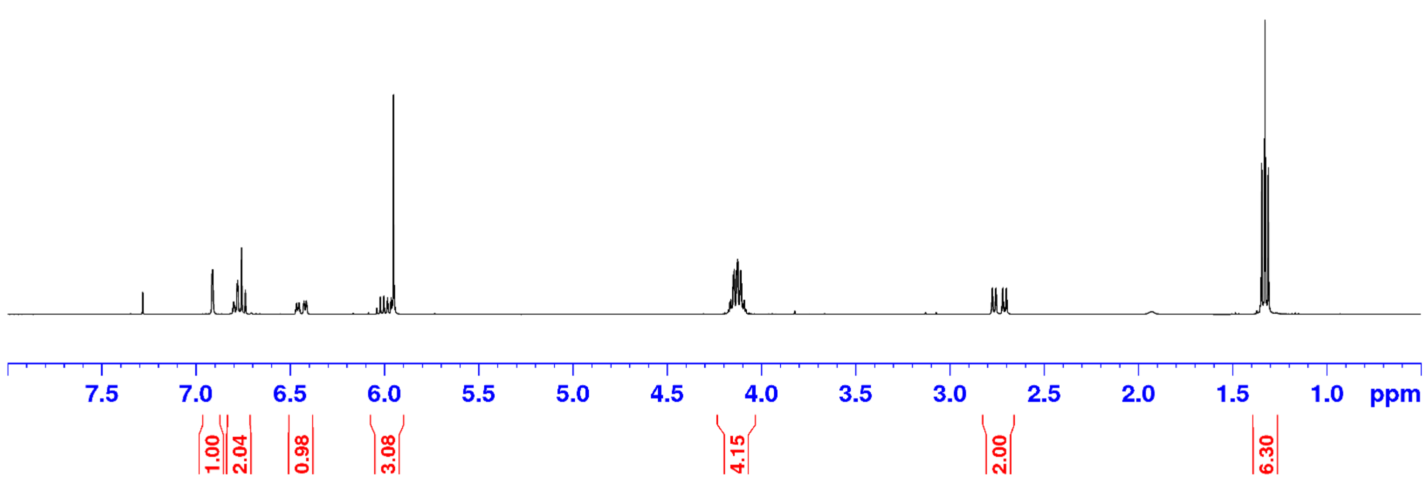
(*E*)-**5f**

(*E*)-5g: ^{1}H NMR

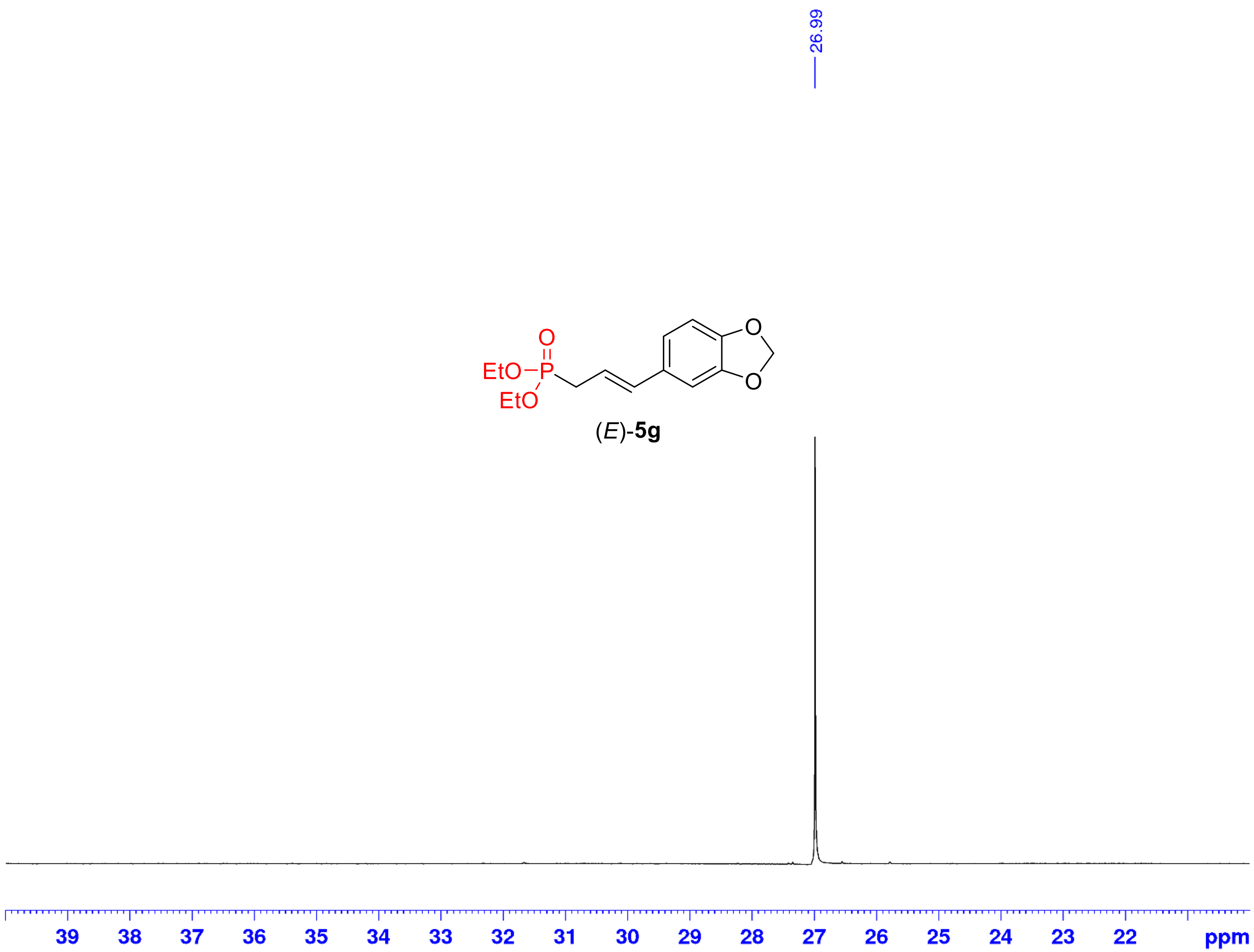




(*E*)-**5g**

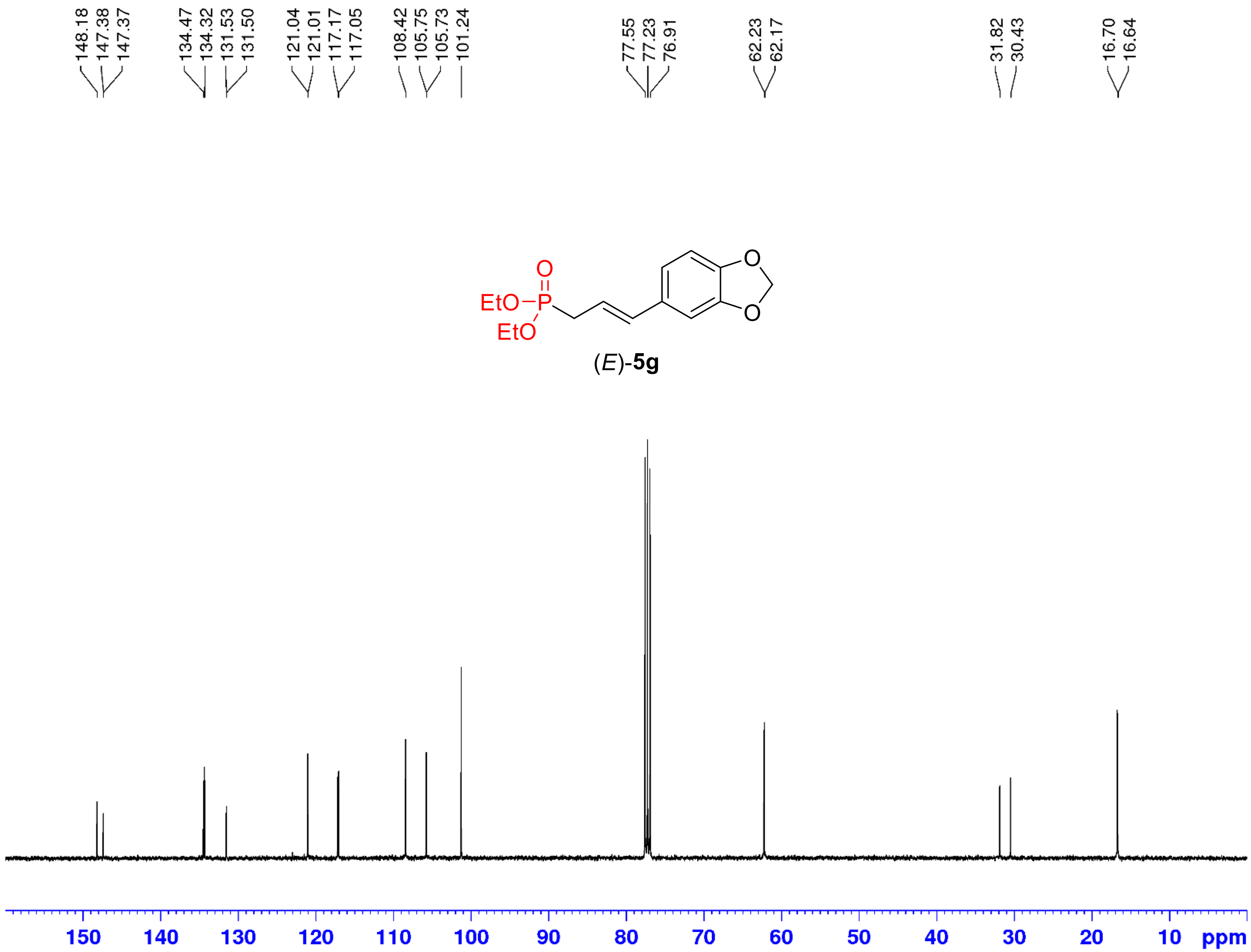


(*E*)-5g: ^{31}P NMR

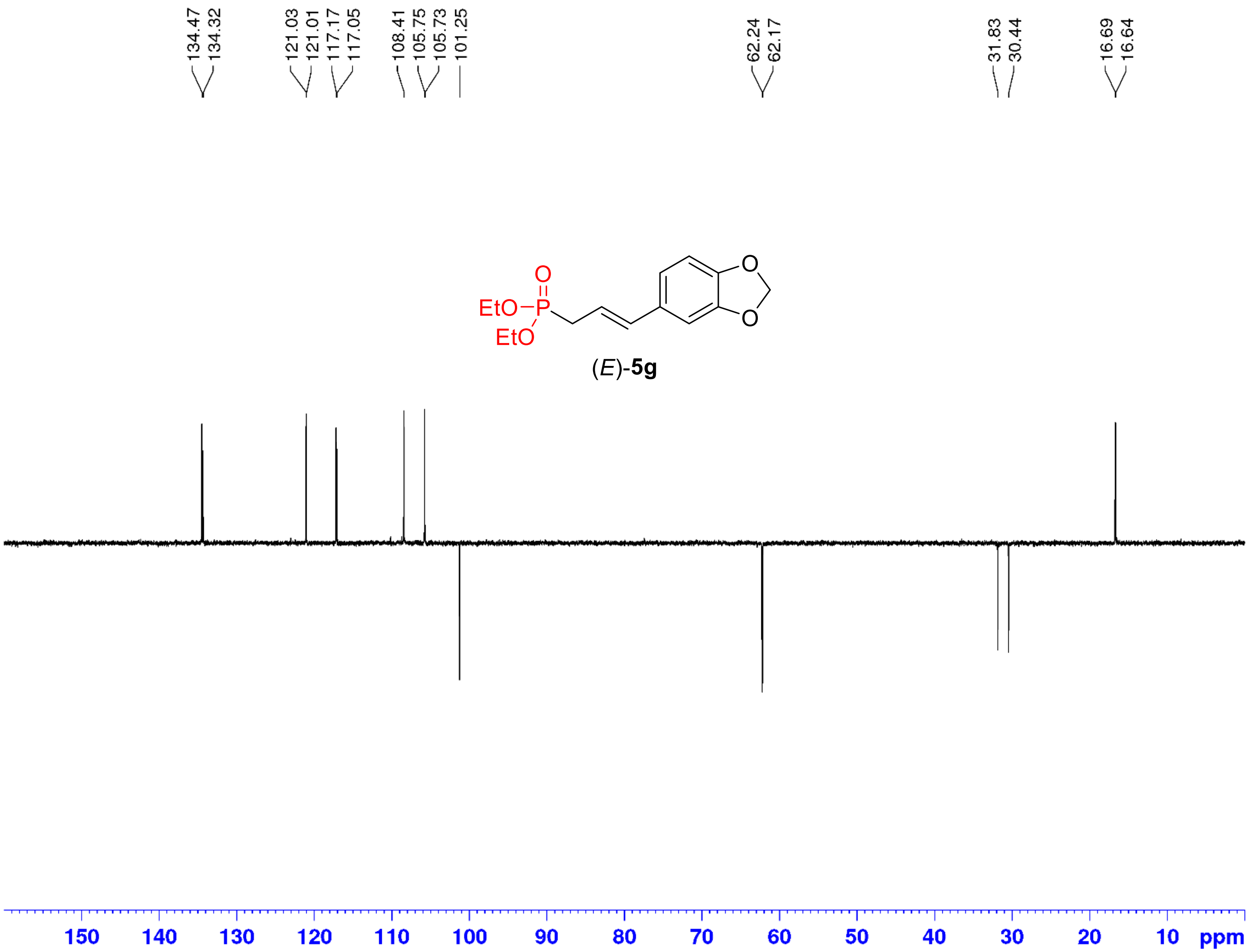


26.99
EtO
EtO
O
P
O
O
(E)-5g
39
38
37
36
35
34
33
32
31
30
29
28
27
26
25
24
23
22
ppm

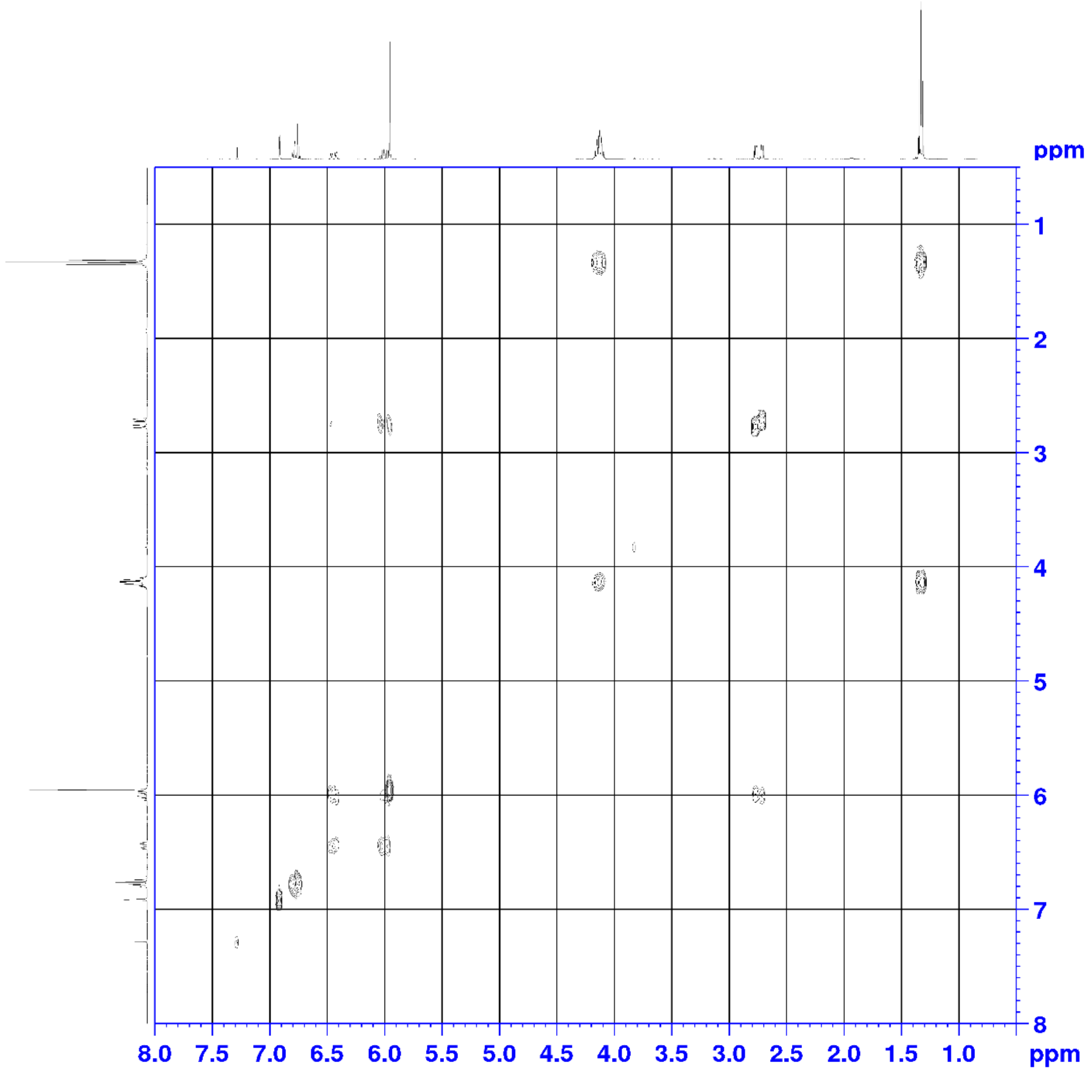
(*E*)-5g: ^{13}C NMR


148.18
147.38
147.37
134.47
134.32
131.53
131.50
121.04
121.01
117.17
117.05
108.42
105.75
105.73
101.24
77.55
77.23
76.91
62.23
62.17
31.82
30.43
16.70
16.64
EtO
EtO
P
O
O
O
(E)-5g
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

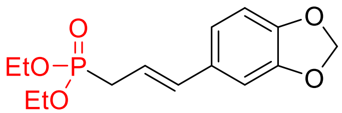
(*E*)-5g: ^{13}C DEPT135



(*E*)-5g: COSY



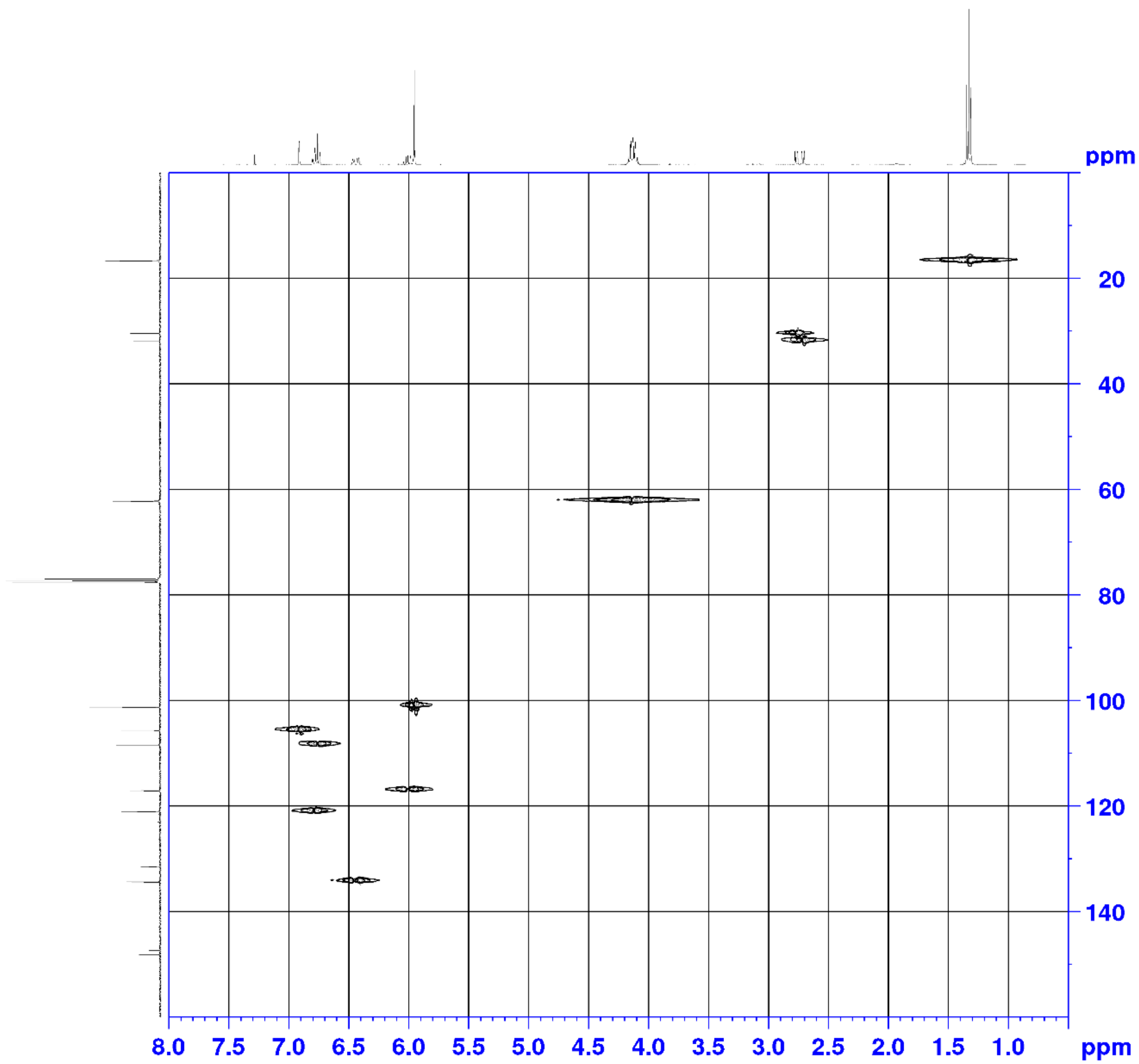
ppm
1
2
3
4
5
6
7
8
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0
ppm

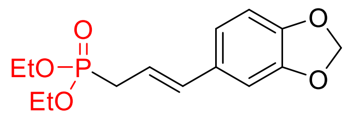


O
EtO
P
EtO
O
O

(*E*)-**5g**

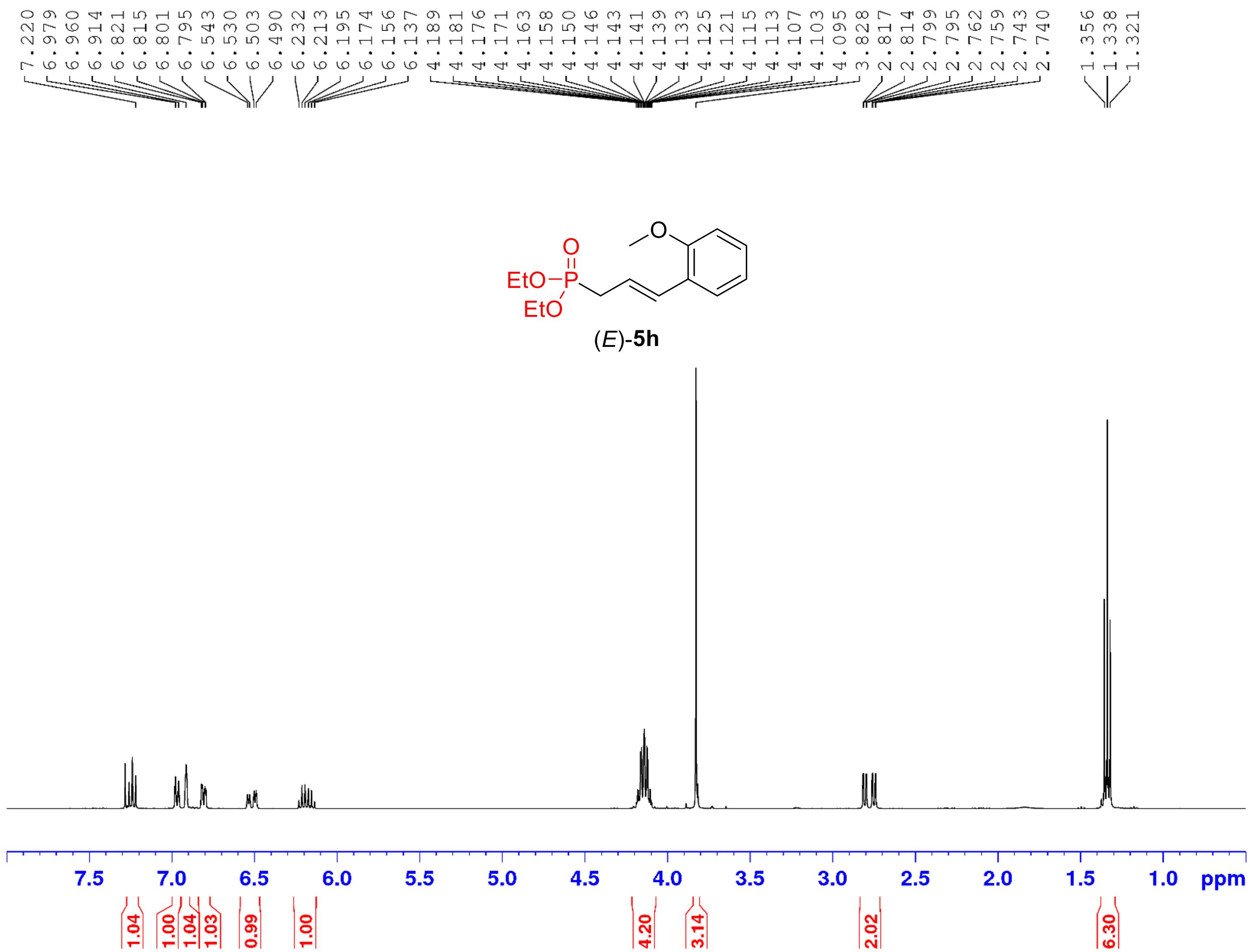
(*E*)-5g: ^{13}C-HSQC



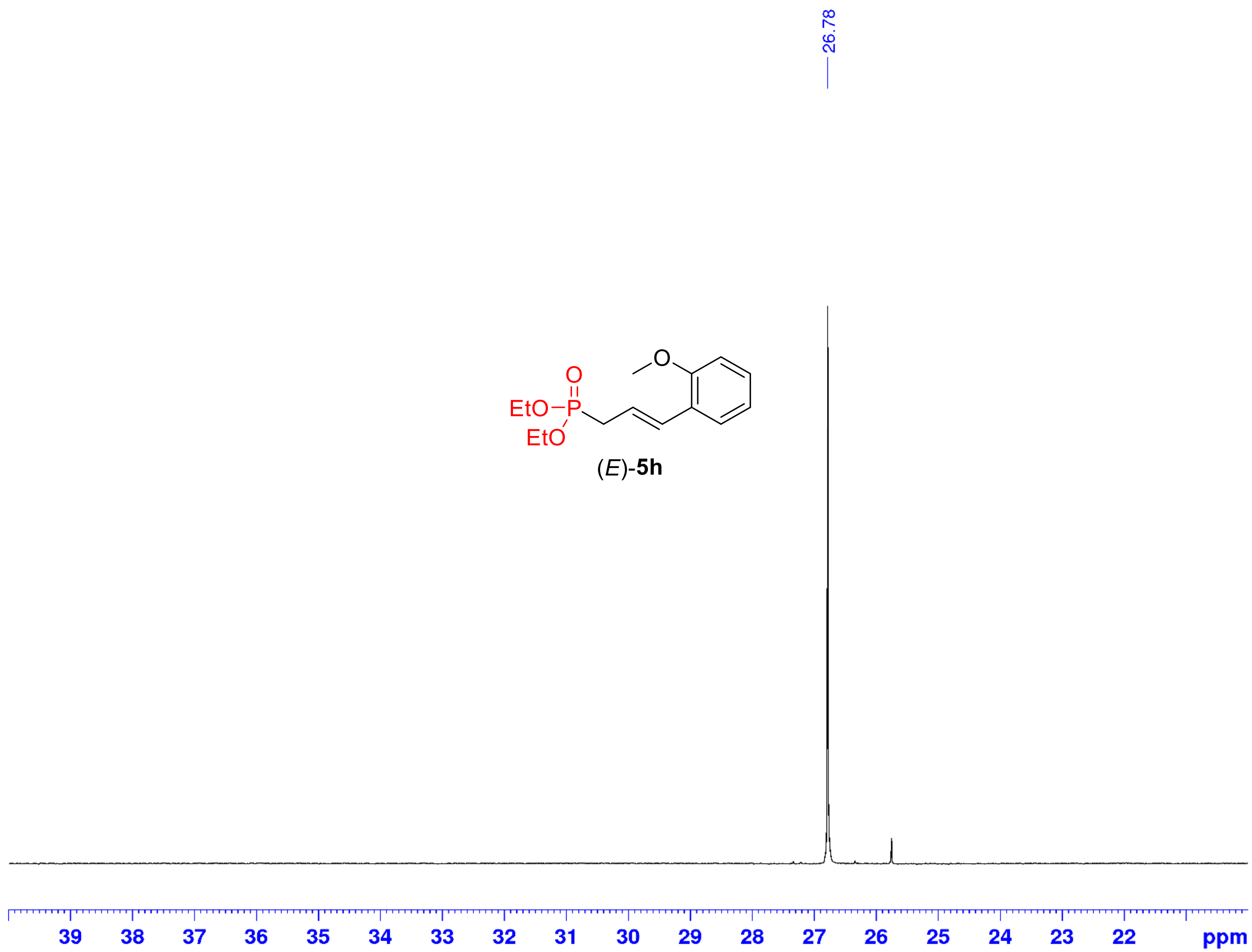


(*E*)-**5g**

(*E*)-5h: ^{1}H NMR

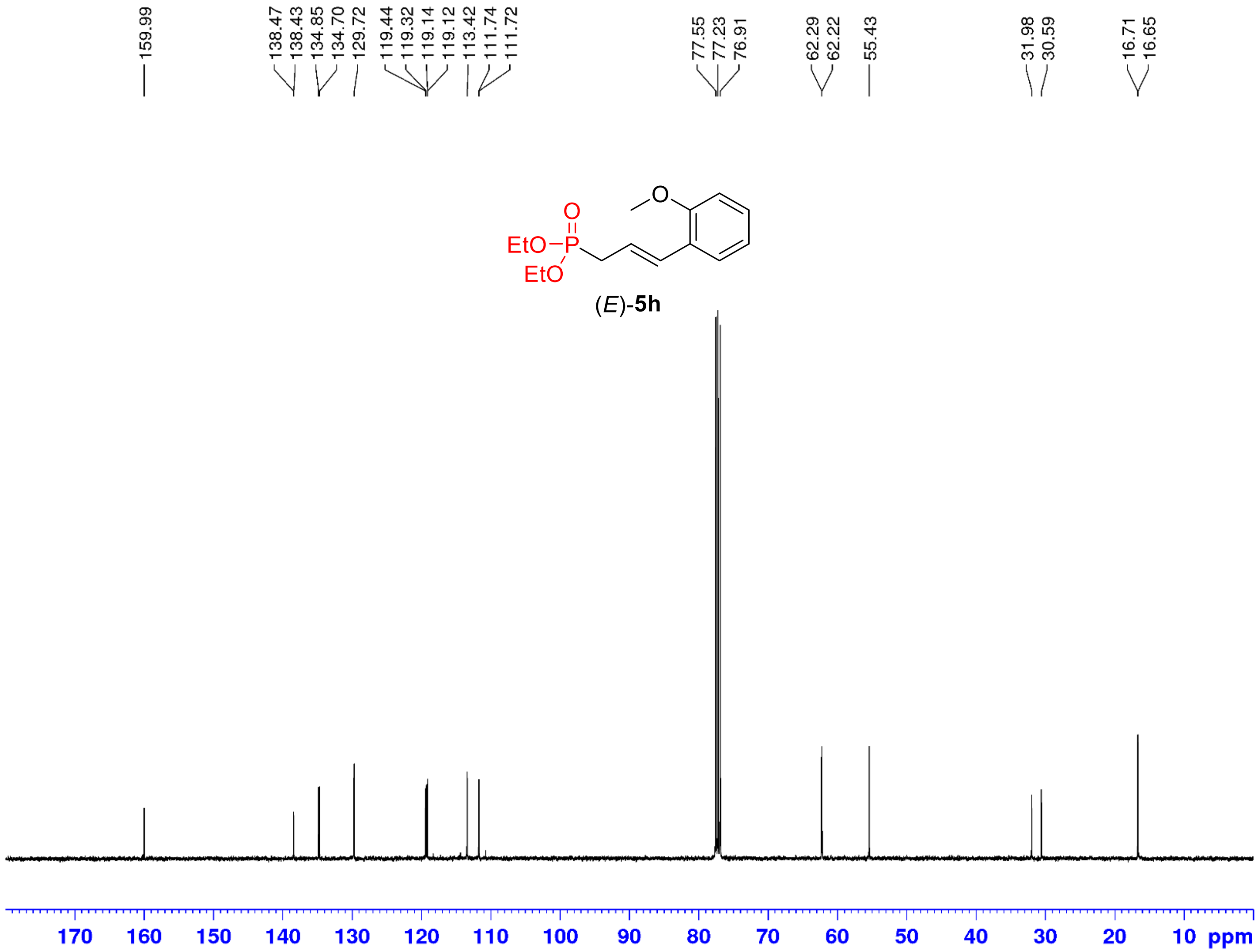

7.220
6.979
6.960
6.914
6.821
6.815
6.801
6.795
6.543
6.530
6.503
6.490
6.232
6.213
6.195
6.174
6.156
6.137
4.189
4.181
4.176
4.171
4.163
4.158
4.150
4.146
4.143
4.141
4.139
4.133
4.125
4.121
4.115
4.113
4.107
4.103
4.095
3.828
2.817
2.814
2.799
2.795
2.762
2.759
2.743
2.740
1.356
1.338
1.321
O
O
EtO
P
EtO
(*E*)-**5h**
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
1.04
1.00
1.04
1.03
0.99
1.00
4.20
3.14
2.02
6.30

(*E*)-5h: ^{31}P NMR



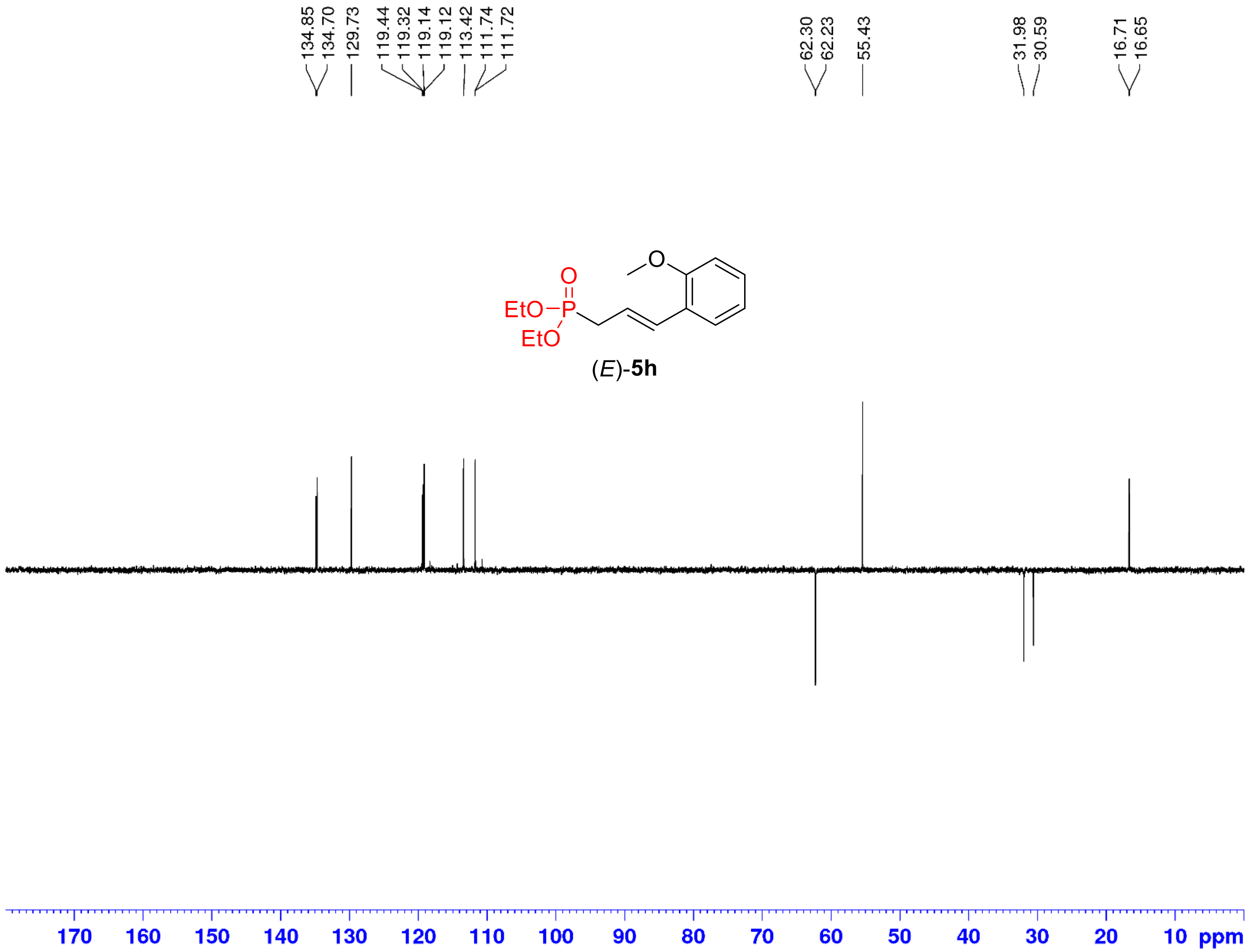
26.78
O
O
EtO
P
EtO
(*E*)-**5h**
39 38 37 36 35 34 33 32 31 30 29 28 27 26 25 24 23 22
ppm

(*E*)-5h: ^{13}C NMR

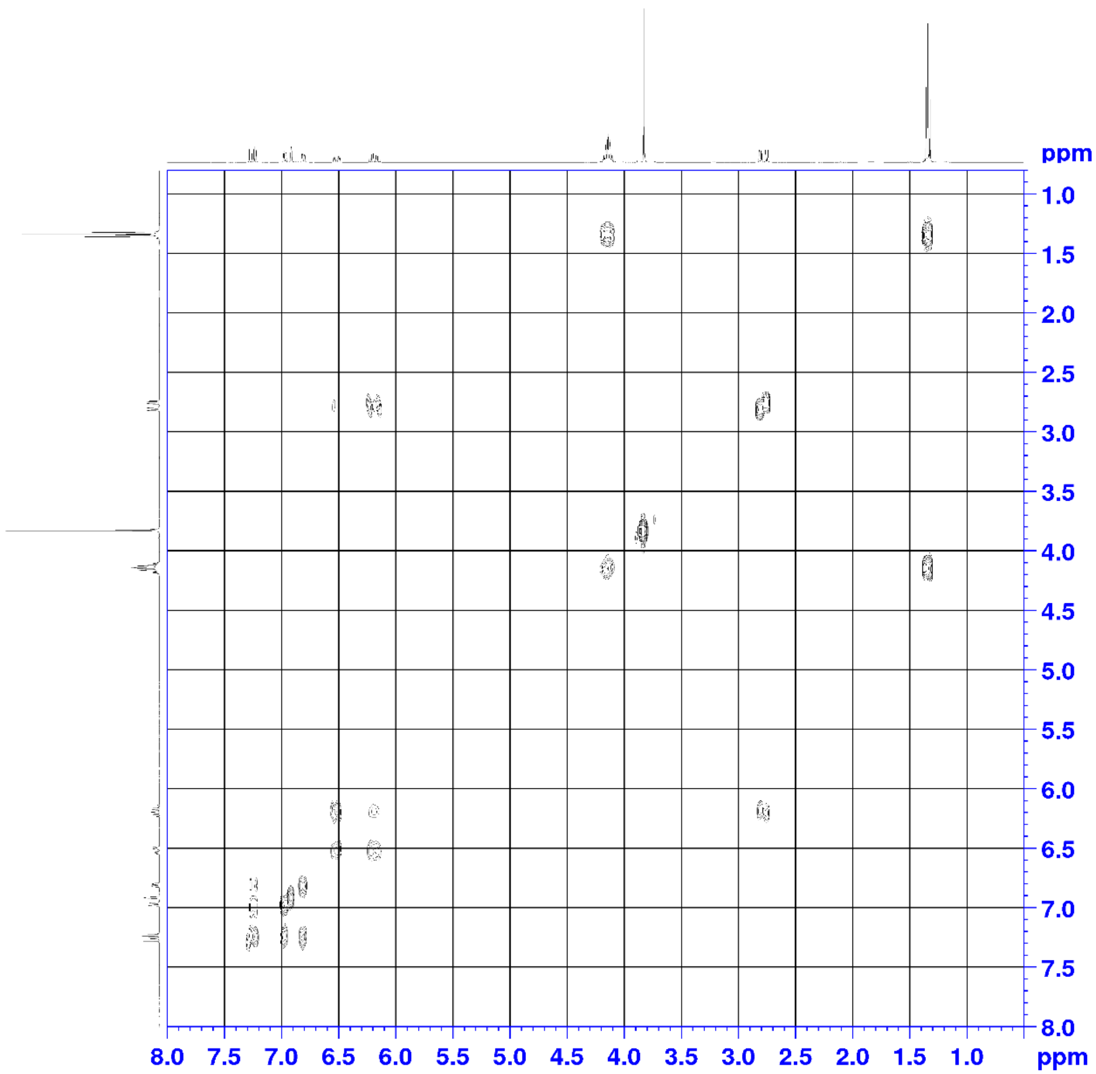


159.99
138.47
138.43
134.85
134.70
129.72
119.44
119.32
119.14
119.12
113.42
111.74
111.72
77.55
77.23
76.91
62.29
62.22
55.43
31.98
30.59
16.71
16.65
O
O
EtO
P
EtO
(E)-5h
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

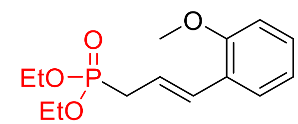
(*E*)-5h: ^{13}C DEPT135



(*E*)-5h: COSY



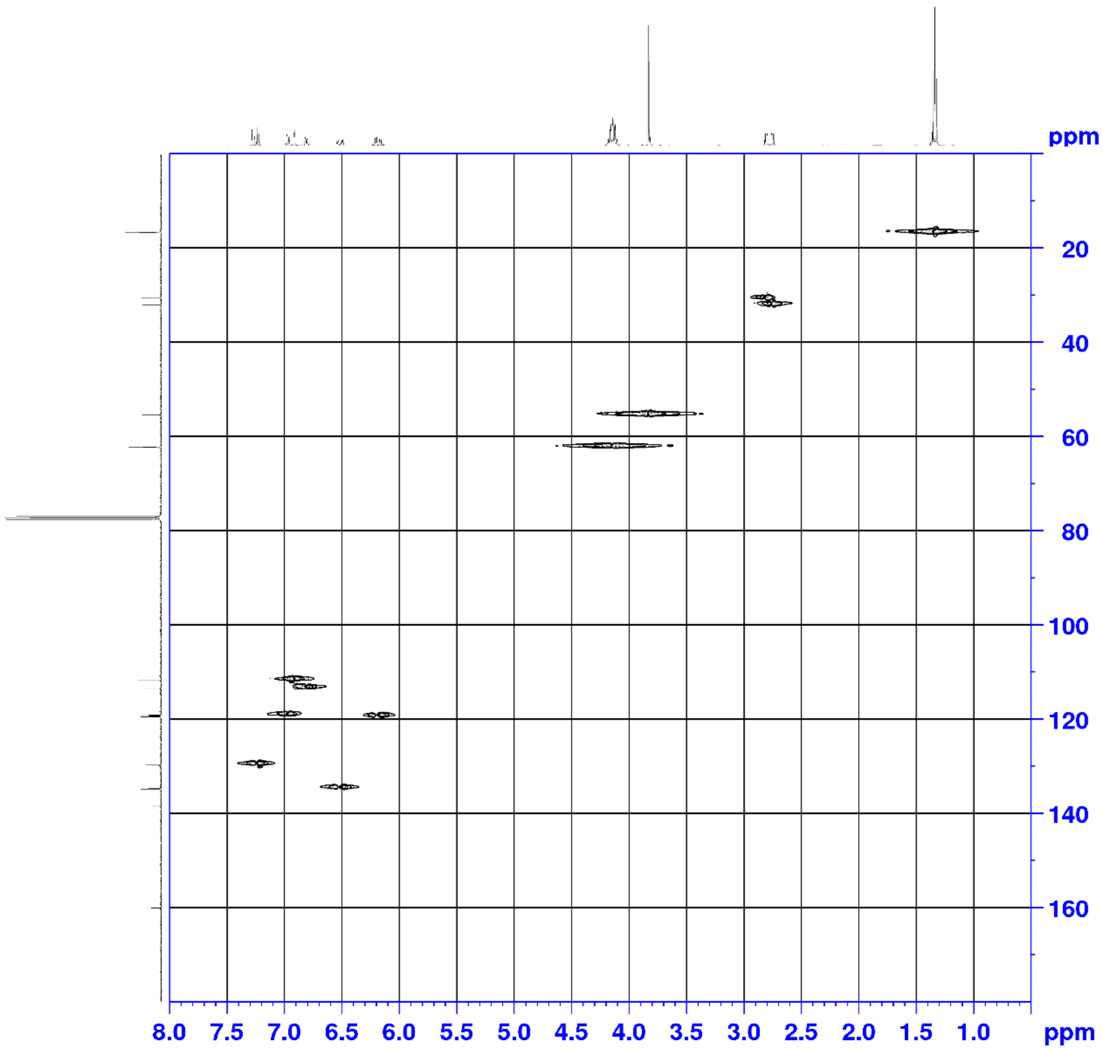
ppm
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0
ppm

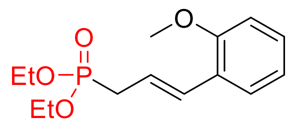


O
O
EtO
P
EtO

(*E*)-**5h**

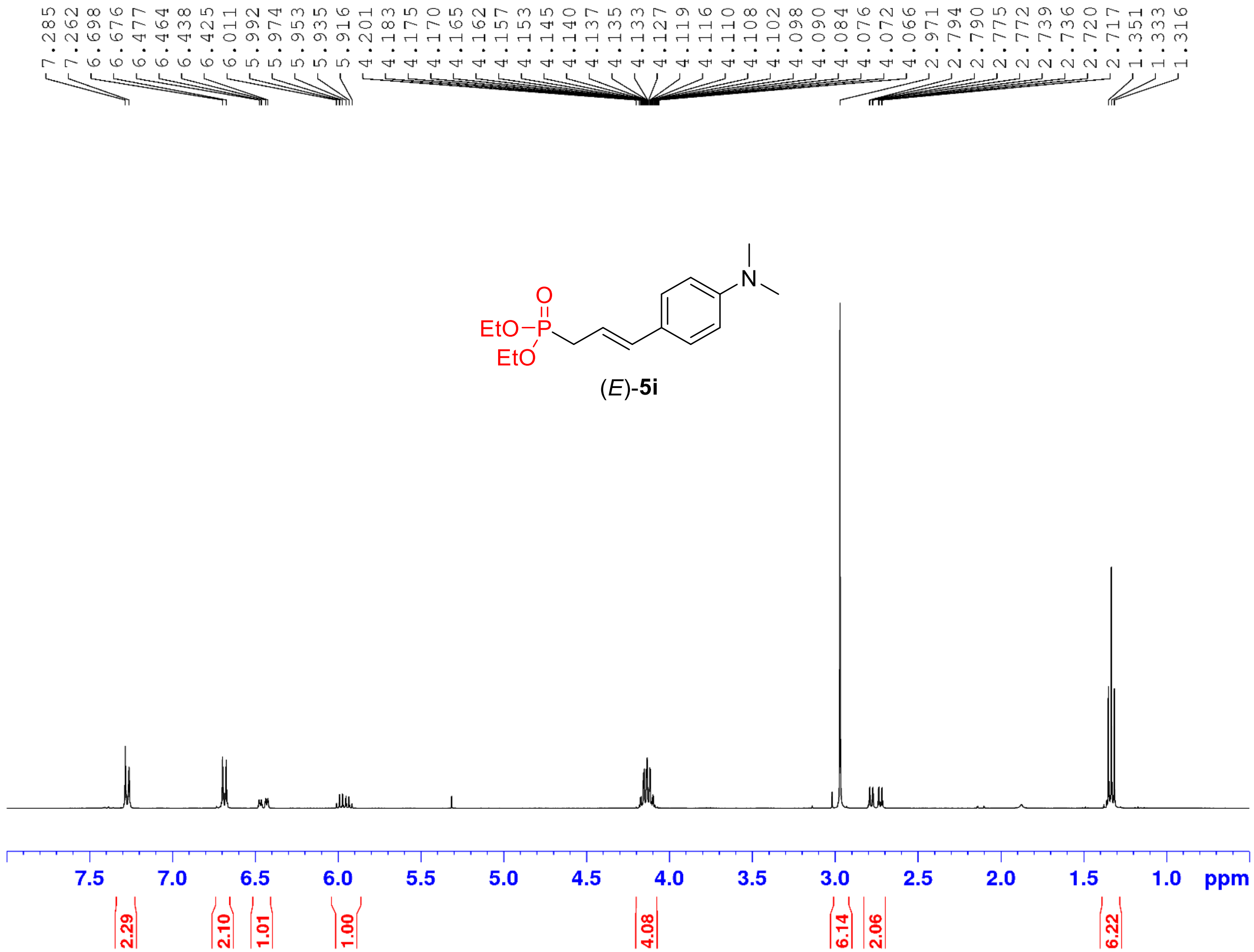
(*E*)-5h: [13]C-HSQC



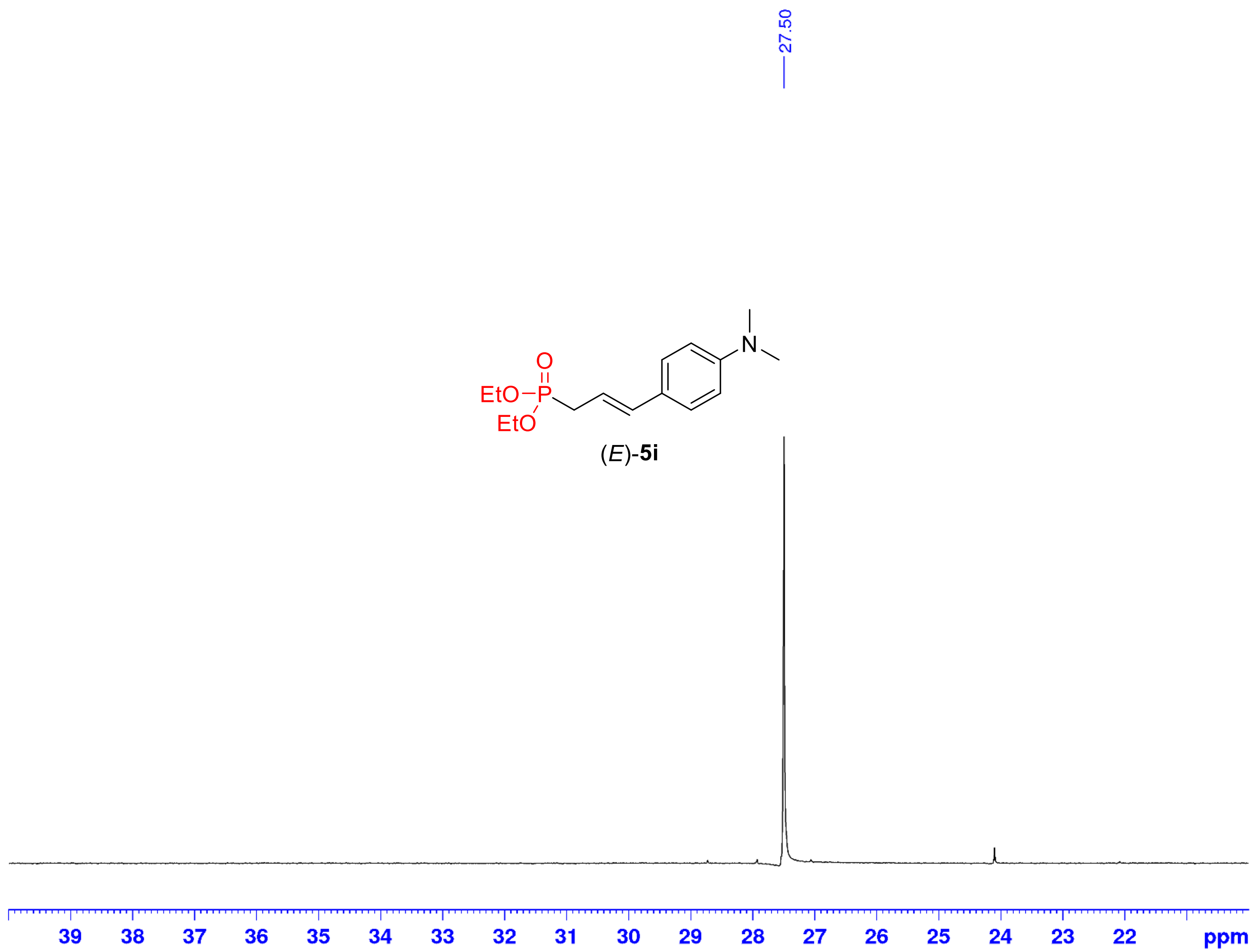


(*E*)-**5h**

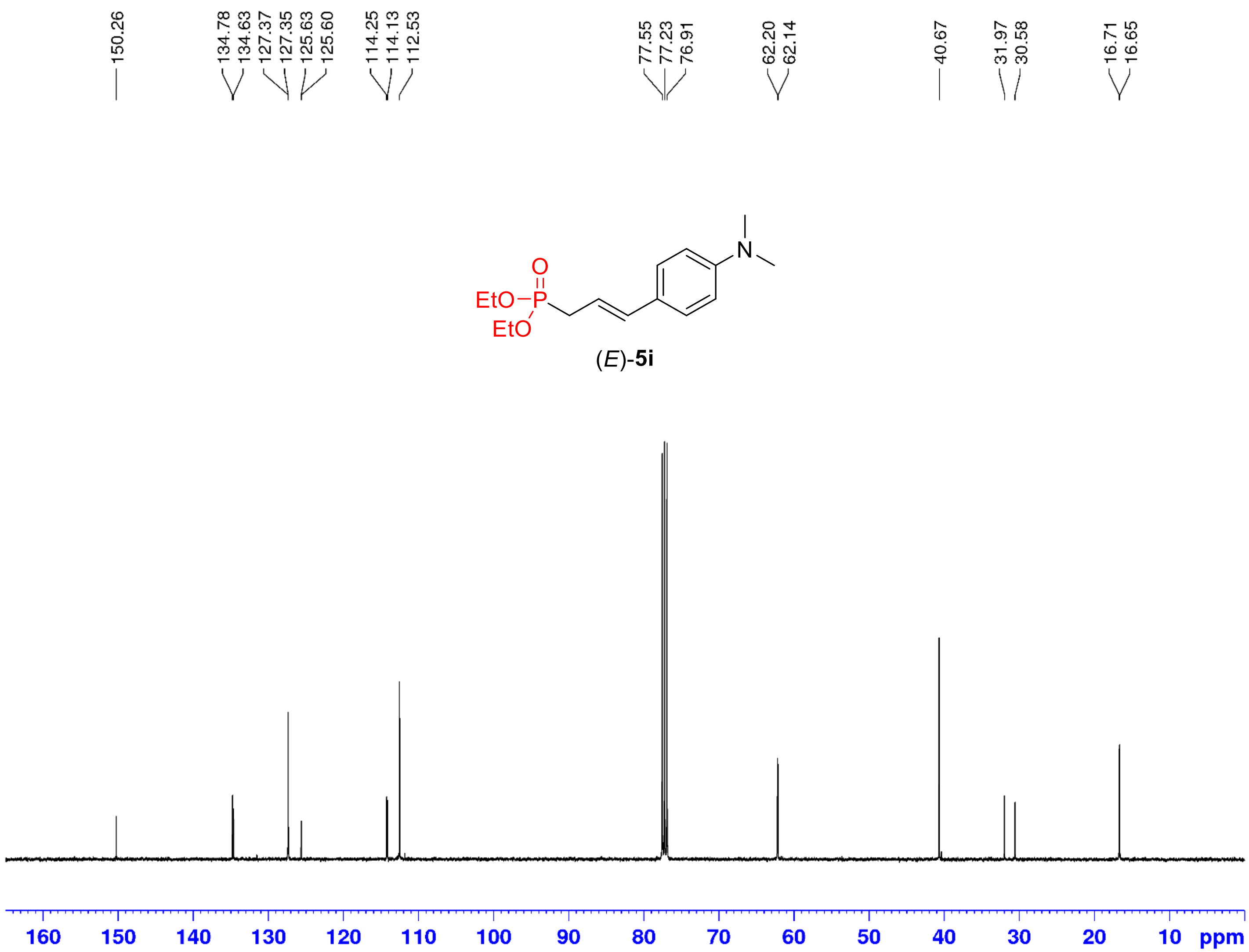
(*E*)-5i: ^{1}H NMR


7.285
7.262
6.698
6.676
6.477
6.464
6.438
6.425
6.011
5.992
5.974
5.953
5.935
5.916
4.201
4.183
4.175
4.170
4.165
4.162
4.157
4.153
4.145
4.140
4.137
4.135
4.133
4.127
4.119
4.116
4.110
4.108
4.102
4.098
4.090
4.084
4.076
4.072
4.066
2.971
2.794
2.790
2.775
2.772
2.739
2.736
2.720
2.717
1.351
1.333
1.316
O
EtO
P
EtO
N
(E)-5i
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
2.29
2.10
1.01
1.00
4.08
6.14
2.06
6.22

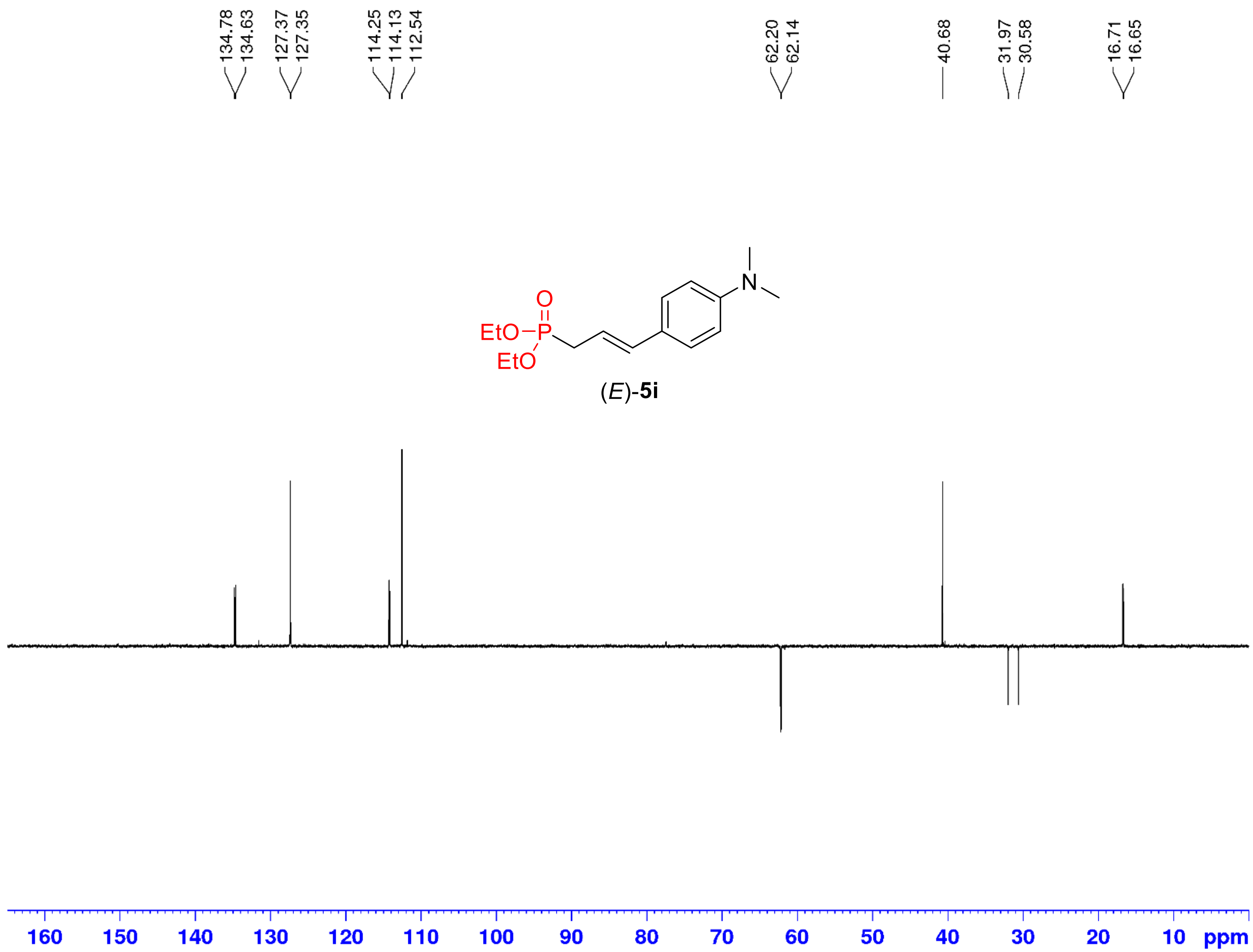
(*E*)-5i: ^{31}P NMR


27.50
O
EtO
P
EtO
N
(E)-5i
39
38
37
36
35
34
33
32
31
30
29
28
27
26
25
24
23
22
ppm

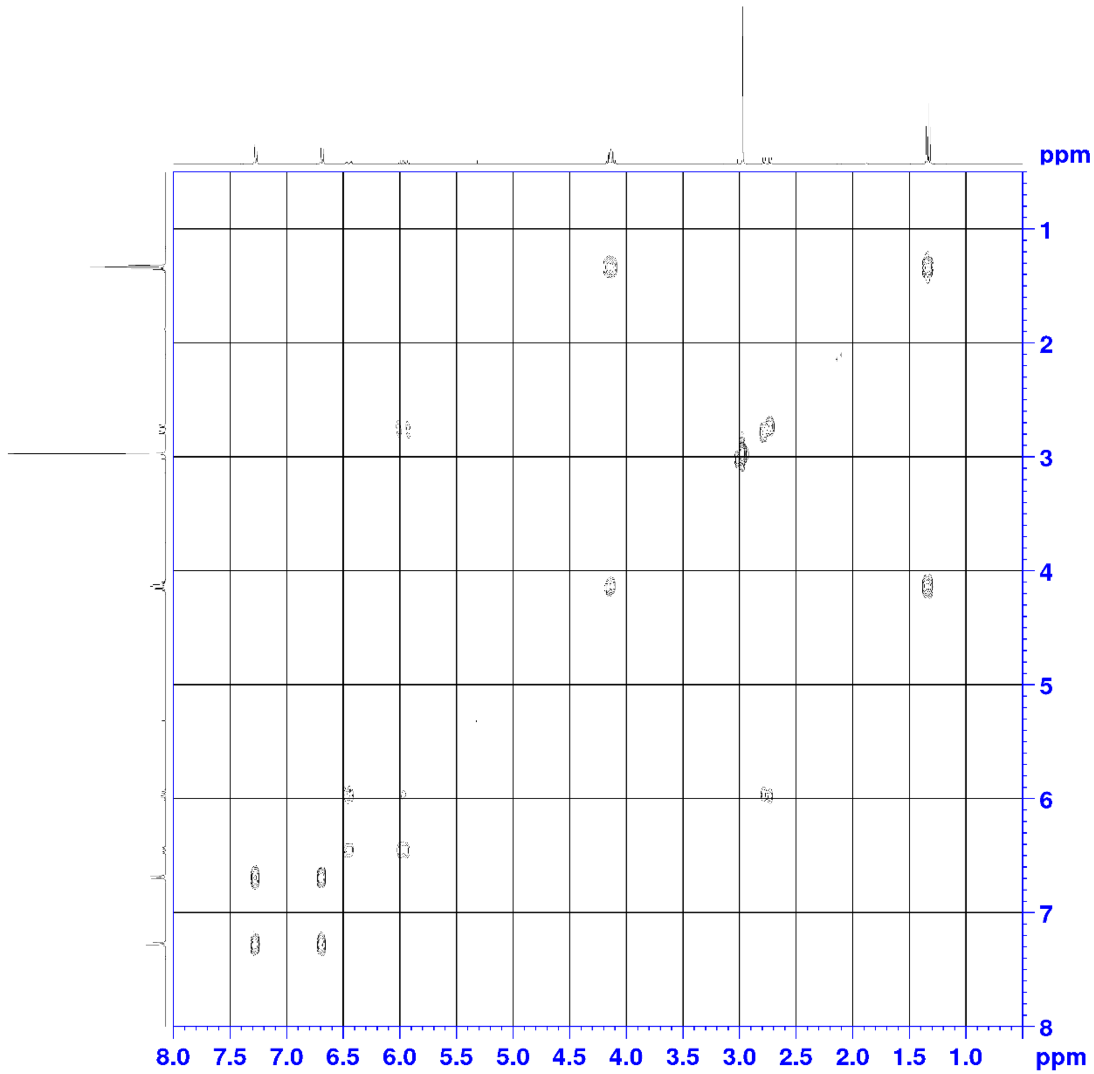
(*E*)-5i: ^{13}C NMR


150.26
134.78
134.63
127.37
127.35
125.63
125.60
114.25
114.13
112.53
77.55
77.23
76.91
62.20
62.14
40.67
31.97
30.58
16.71
16.65
O
EtO
P
EtO
N
(E)-5i
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

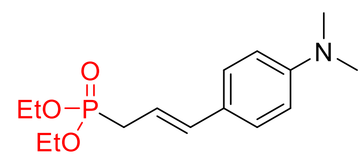
(*E*)-5i: ^{13}C DEPT135



(*E*)-5i: COSY



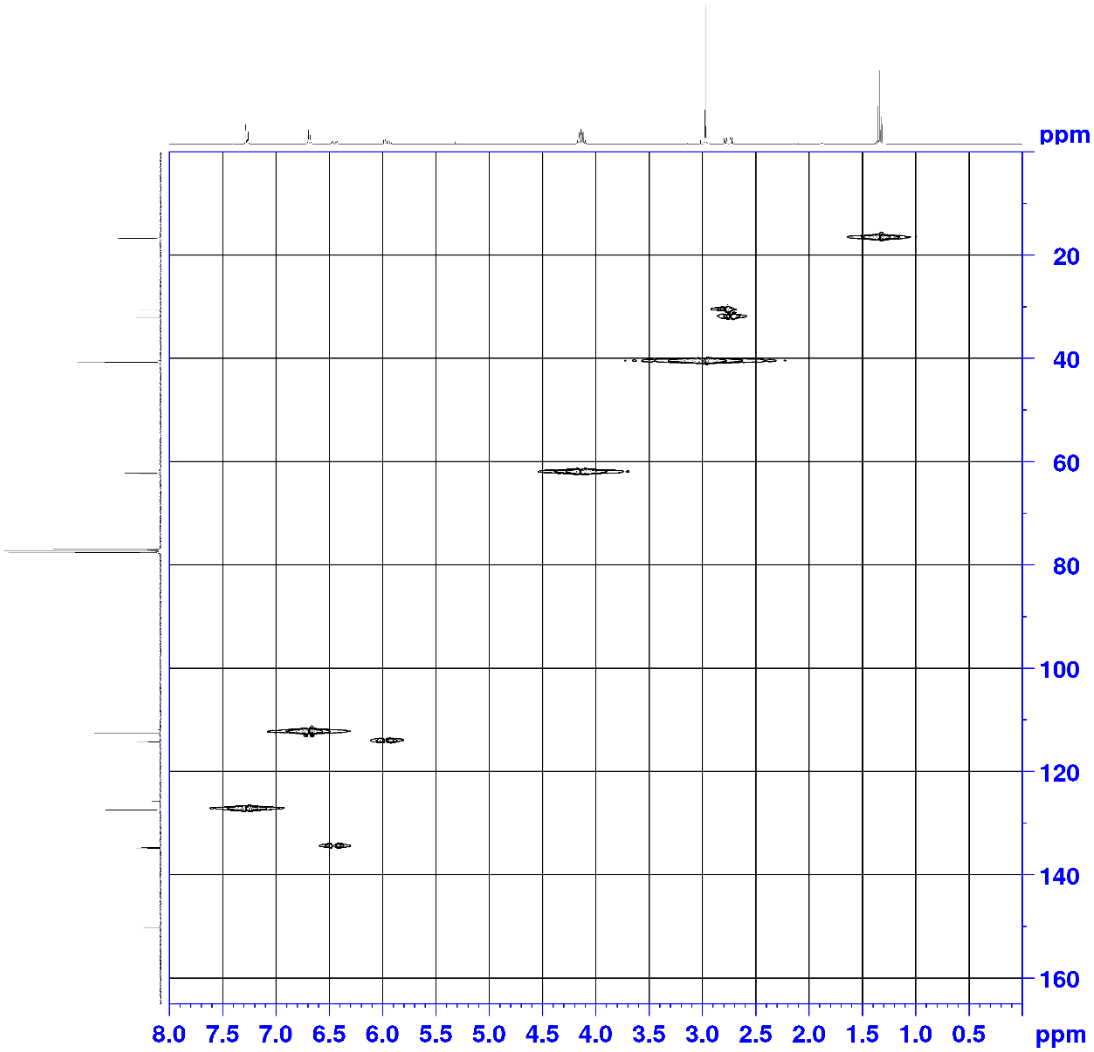
ppm
1
2
3
4
5
6
7
8
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

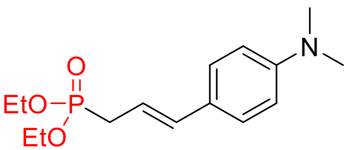


O
EtO
P
EtO
N

(*E*)-**5i**

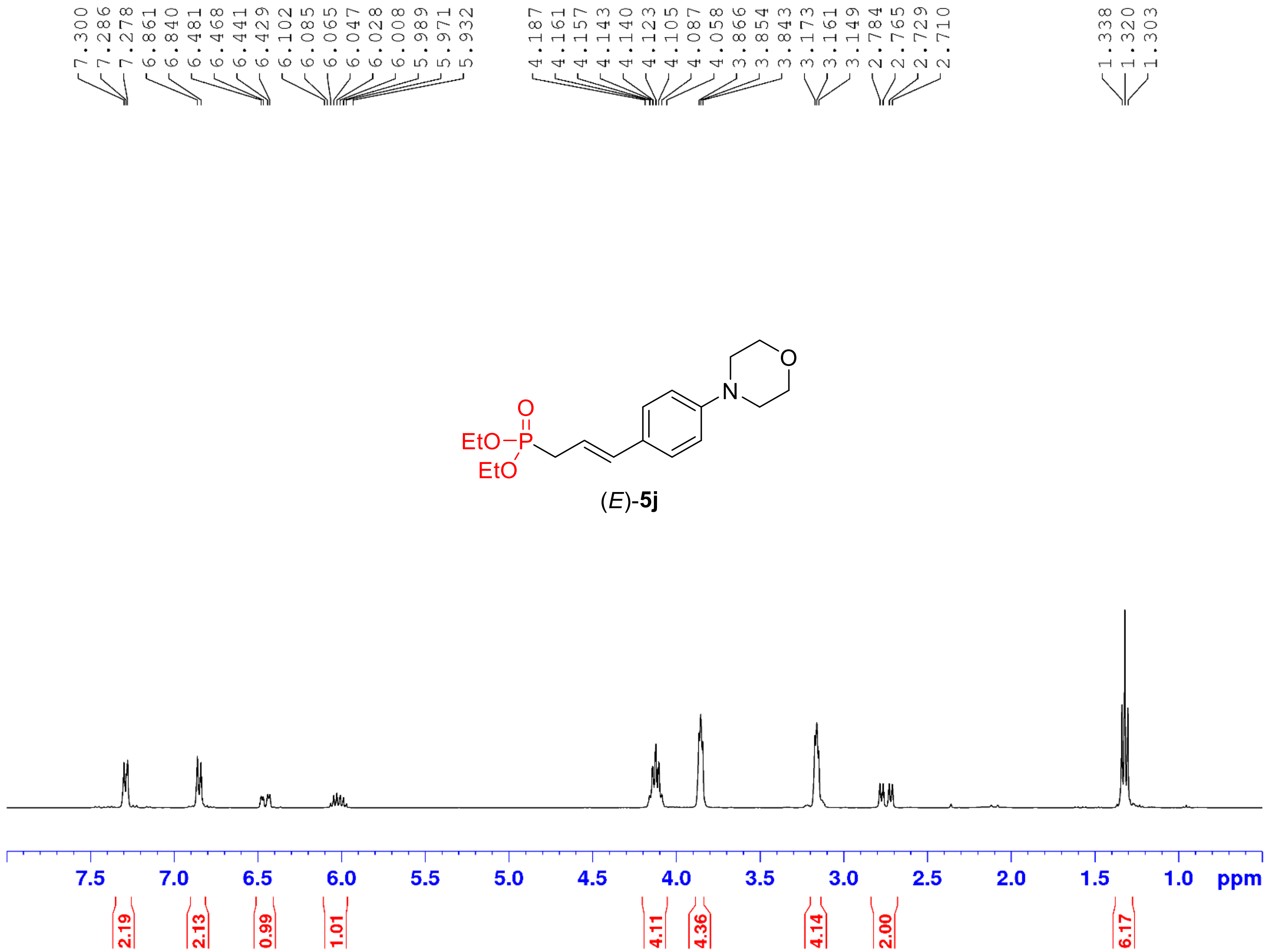
(*E*)-5i: ^{13}C HSQC



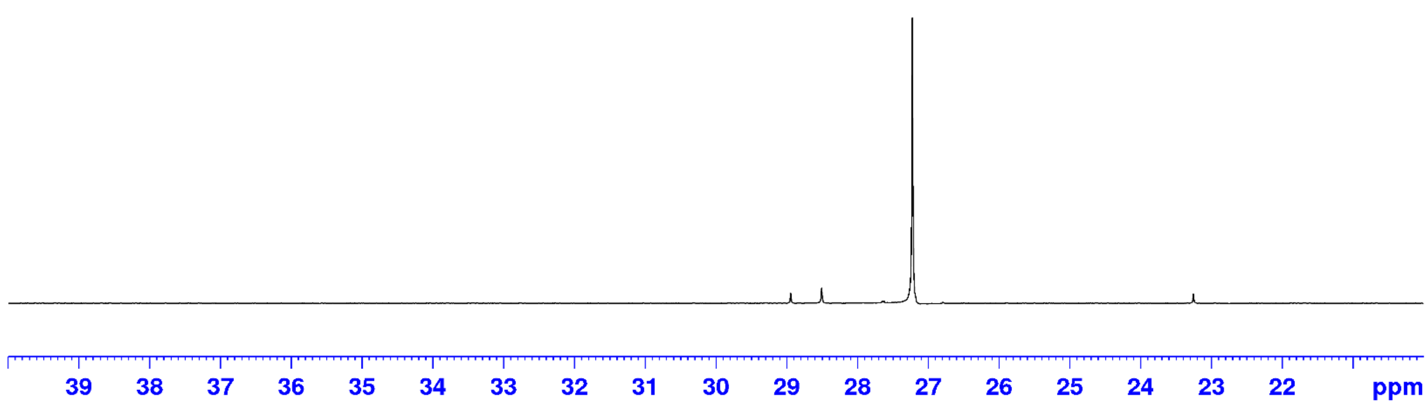


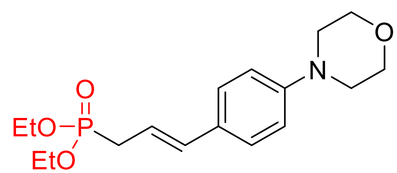
(*E*)-**5i**

(*E*)-5j: ^{1}H NMR


O
EtO
P
EtO
N
O
(*E*)-**5j**
7.300
7.286
7.278
6.861
6.840
6.481
6.468
6.441
6.429
6.102
6.085
6.065
6.047
6.028
6.008
5.989
5.971
5.932
4.187
4.161
4.157
4.143
4.140
4.123
4.105
4.087
4.058
3.866
3.854
3.843
3.173
3.161
3.149
2.784
2.765
2.729
2.710
1.338
1.320
1.303
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
2.19
2.13
0.99
1.01
4.11
4.36
4.14
2.00
6.17

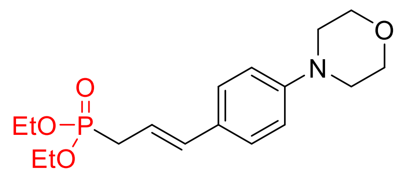
(*E*)-5j: ^{31}P NMR



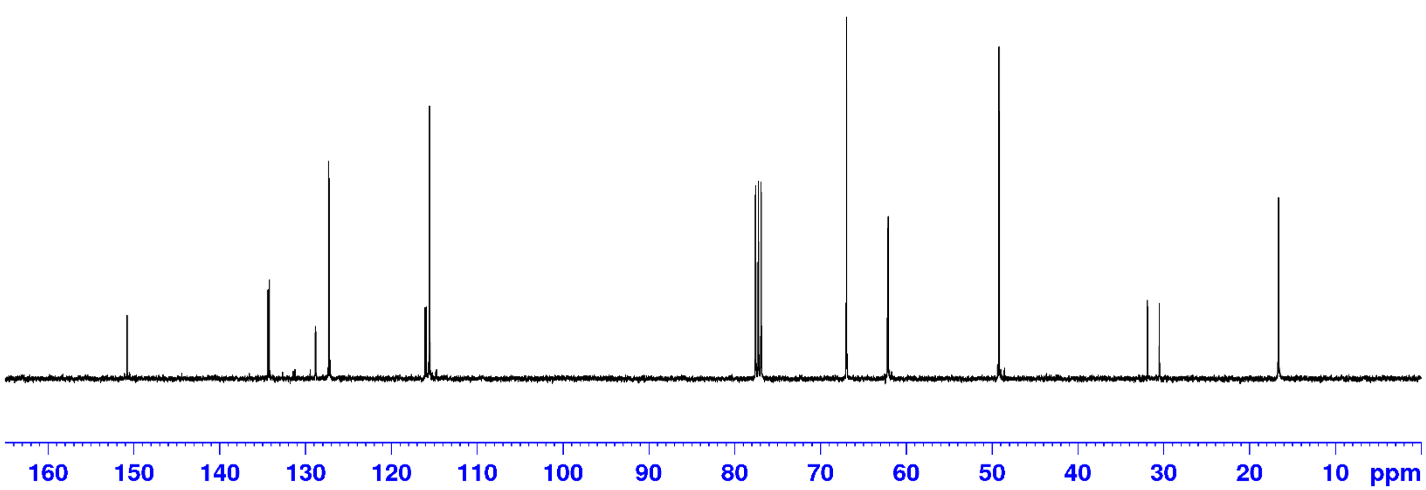


(*E*)-**5j**

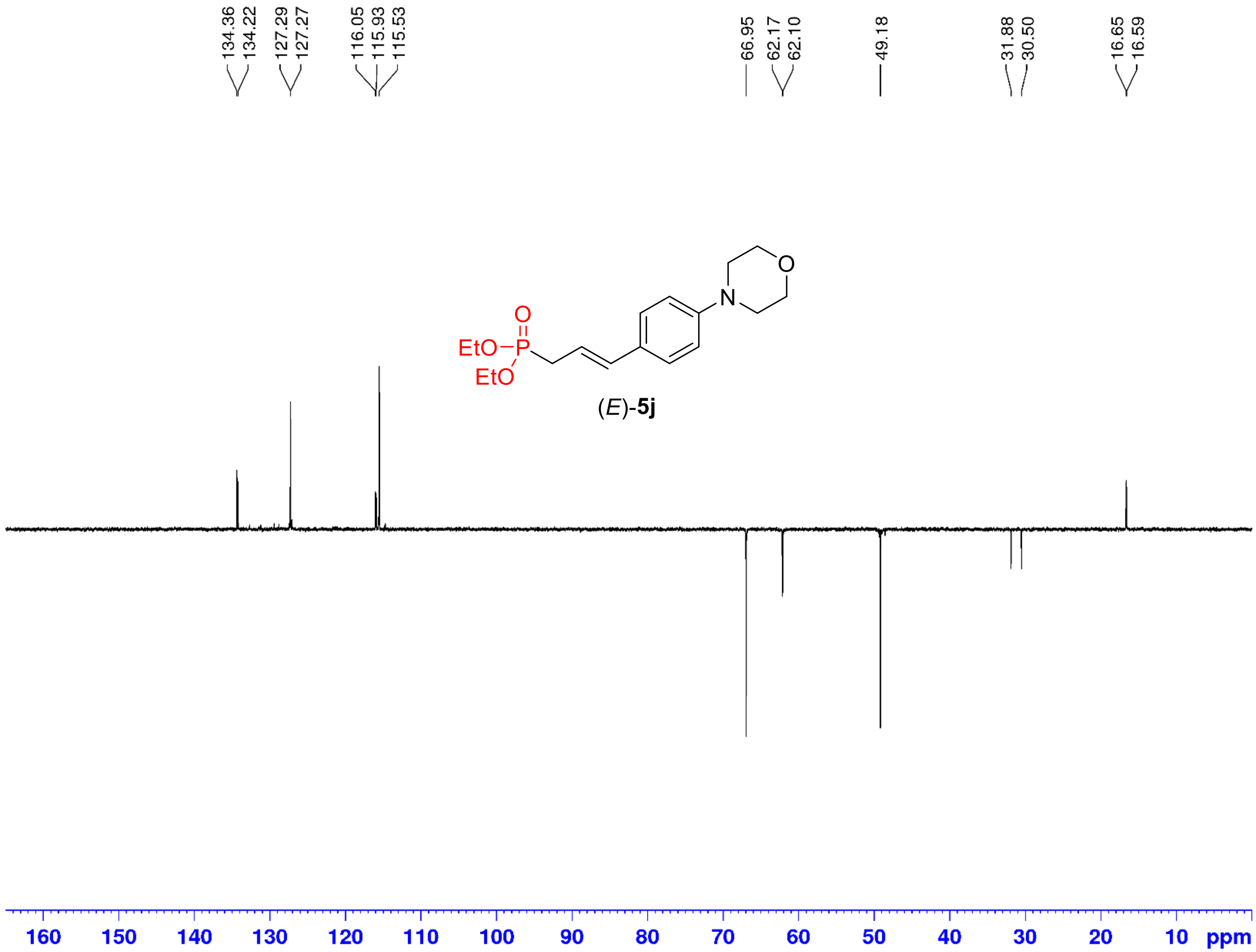
(*E*)-5j: ^{13}C NMR



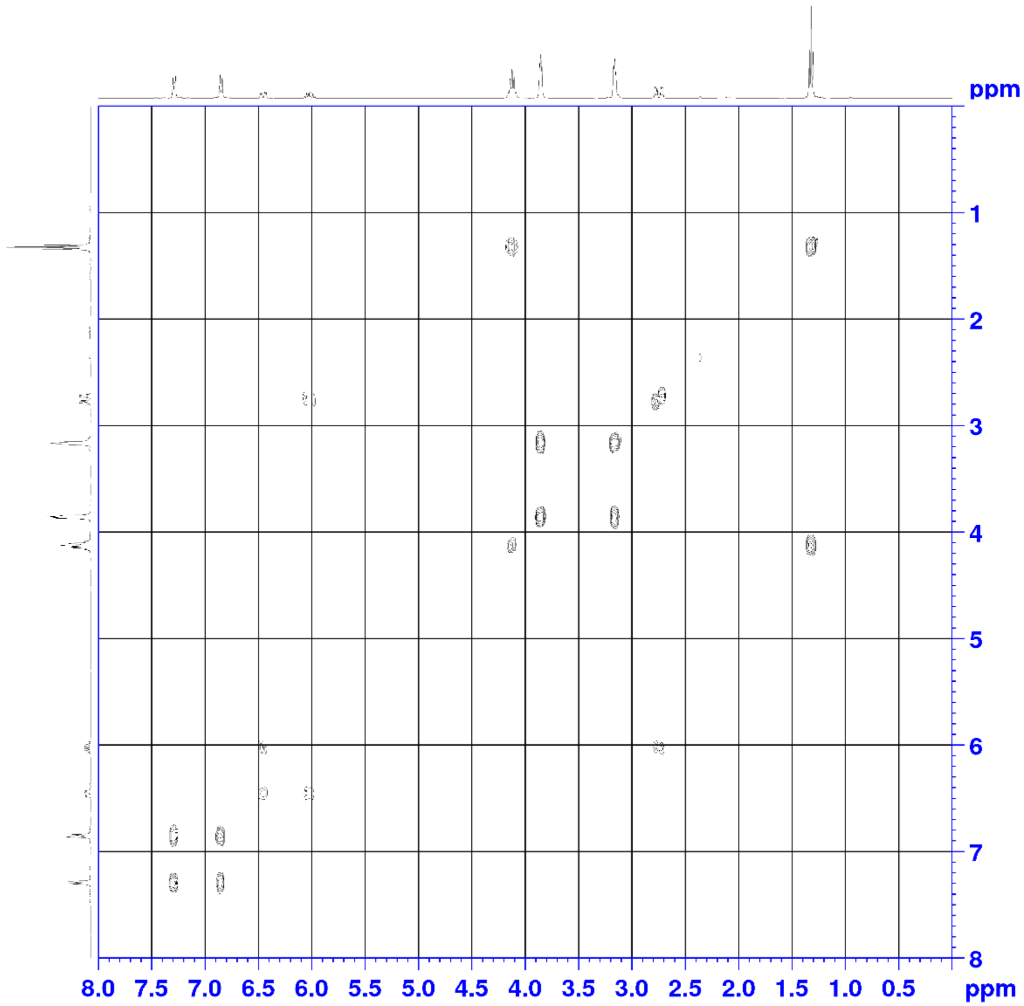
(*E*)-**5j**

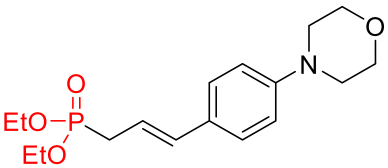


(*E*)-5j: ^{13}C DEPT135



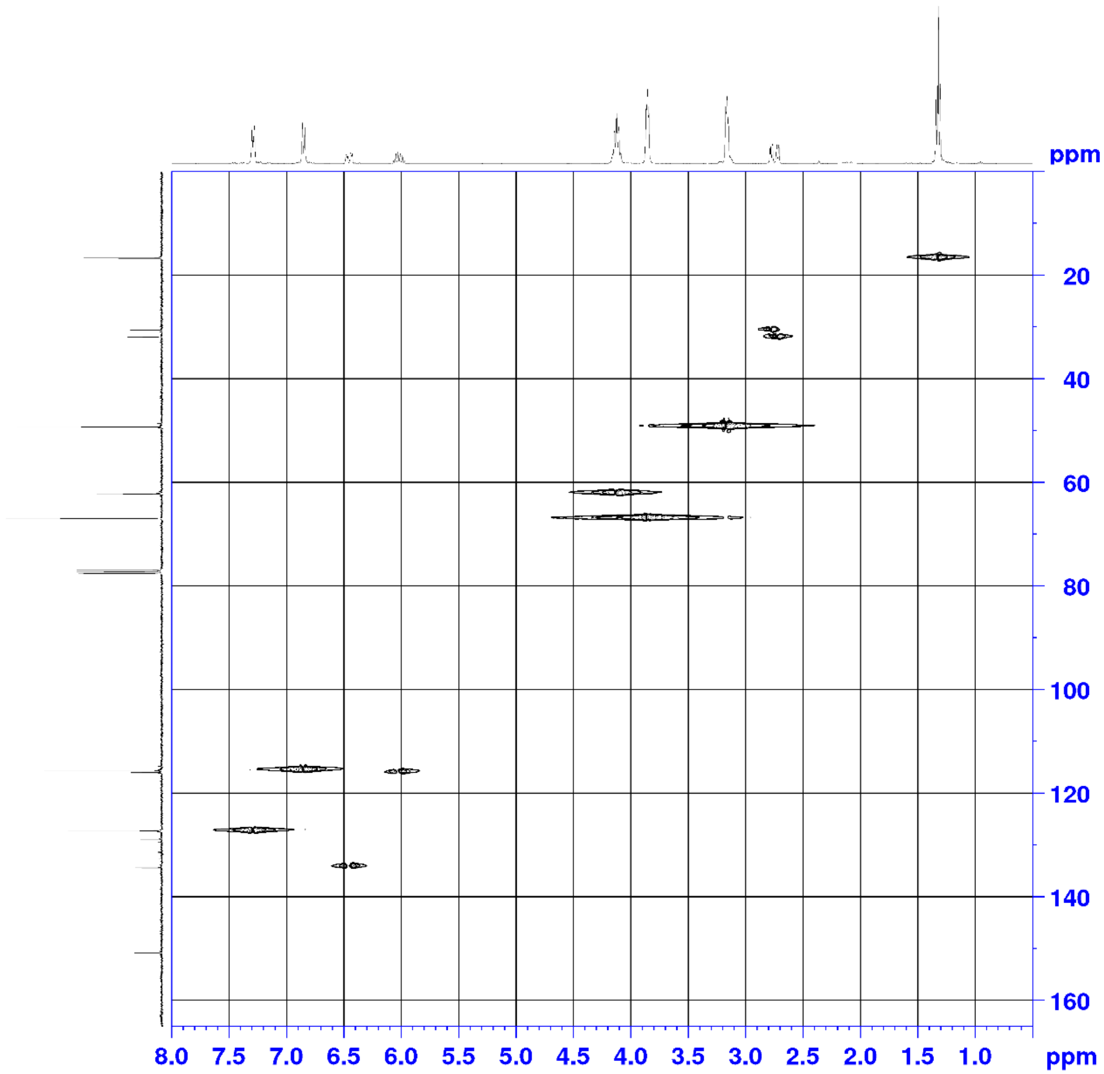
(*E*)-5j: COSY

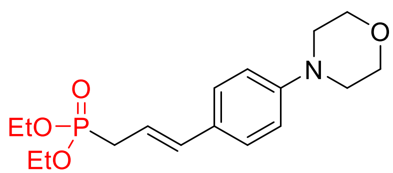

ppm
1
2
3
4
5
6
7
8
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
ppm


O
EtO
P
EtO
N
O

(*E*)-**5j**

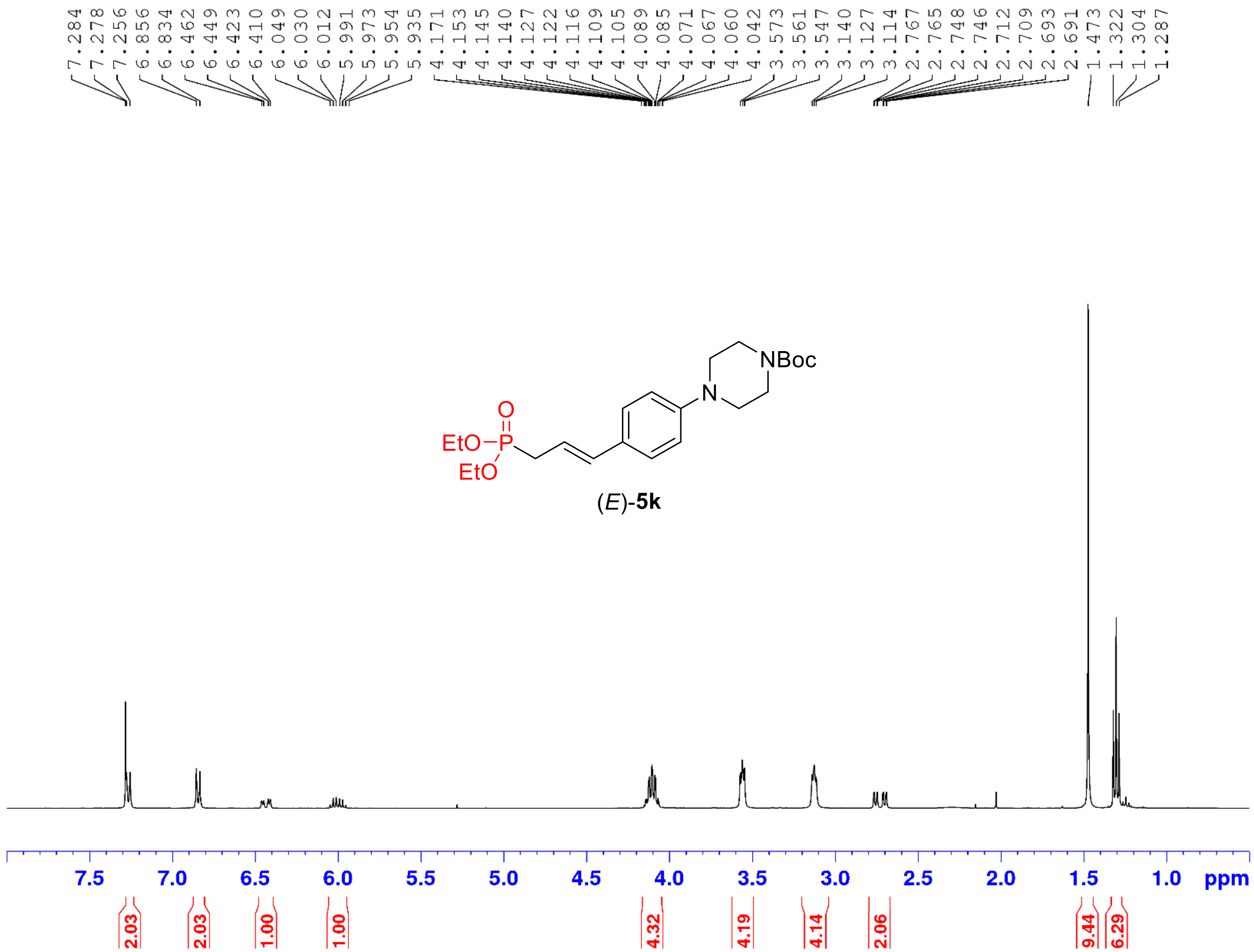
(*E*)-5j: ^{13}C HSQC





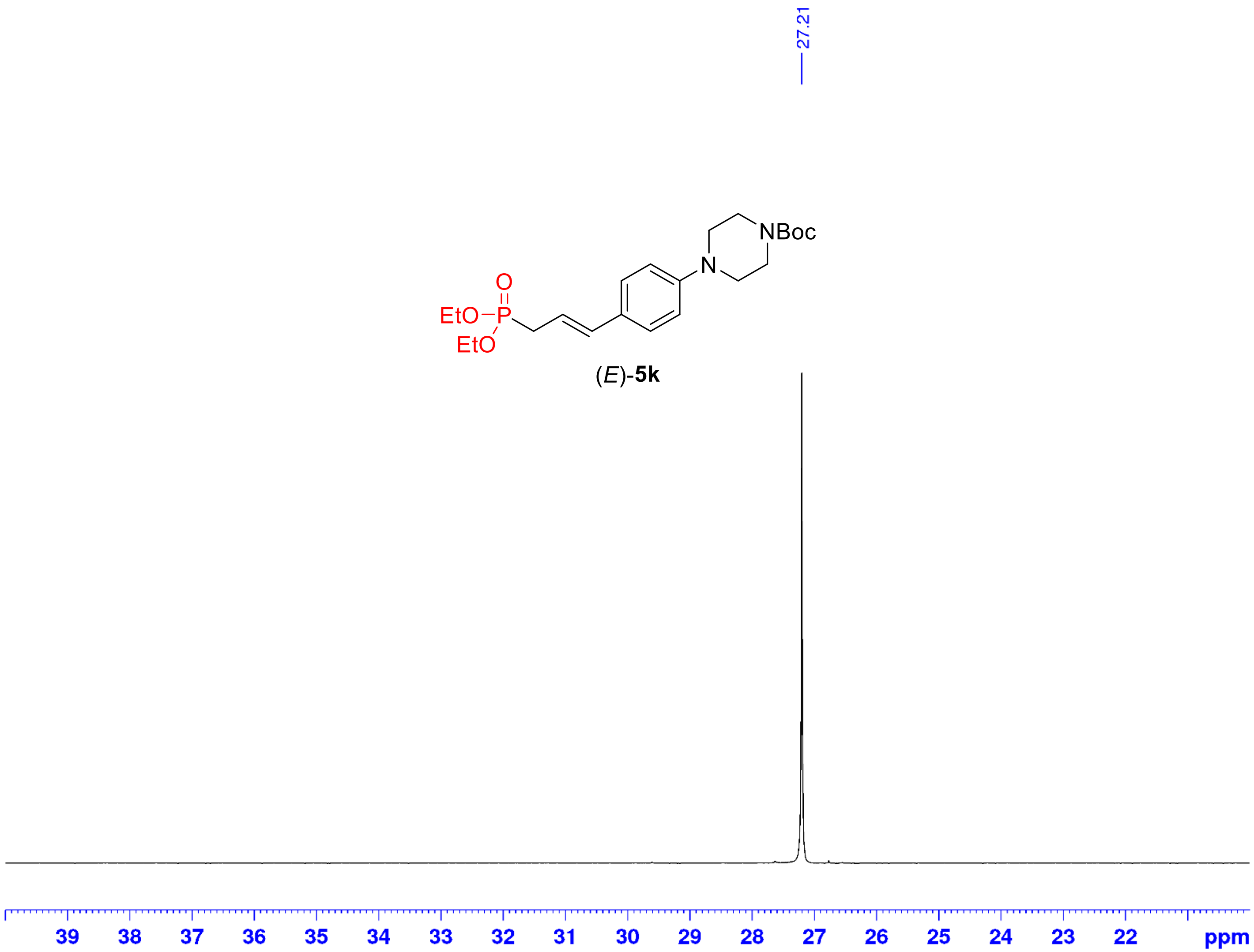
(*E*)-**5j**

(*E*)-5k: ^{1}H NMR



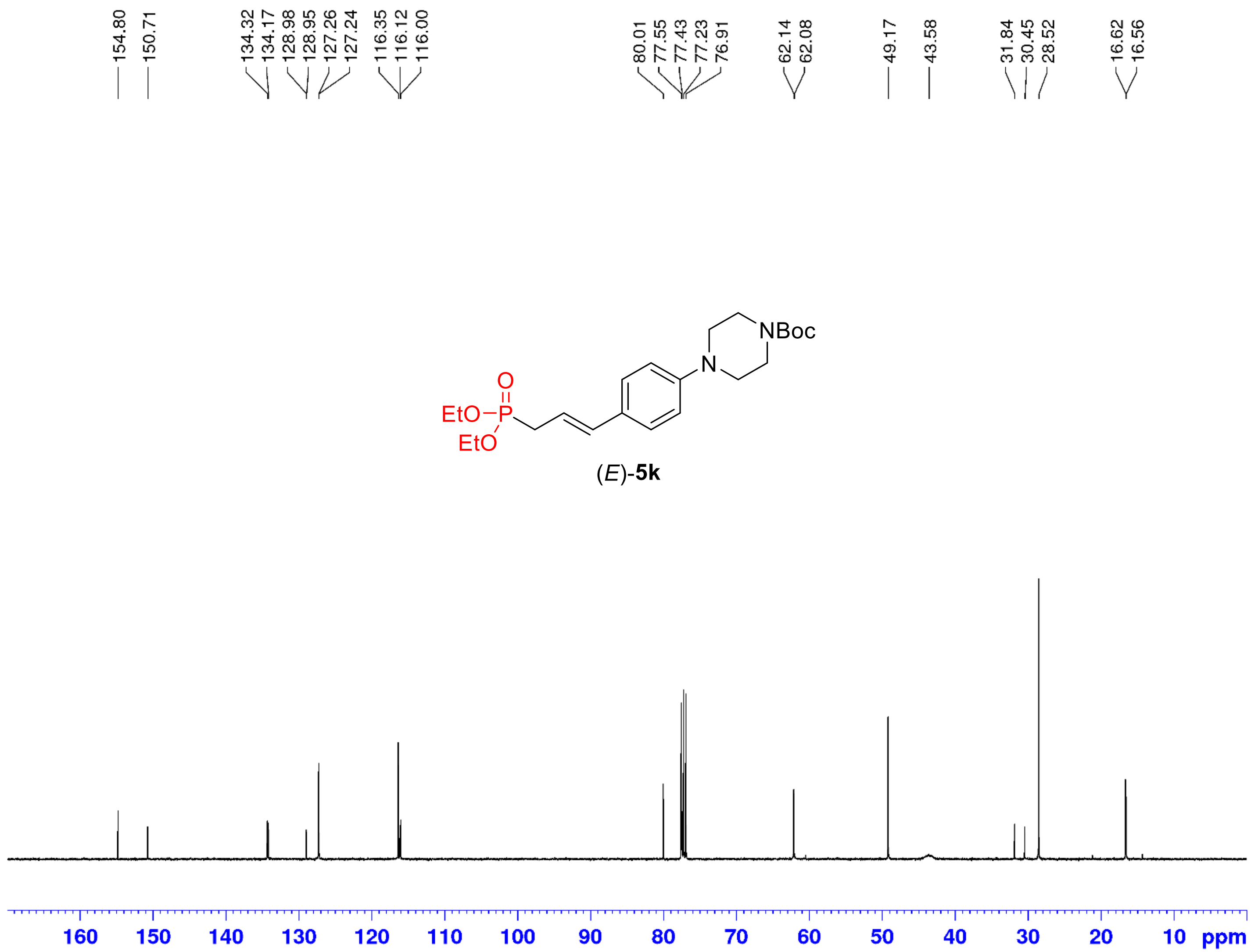
7.284
7.278
7.256
6.856
6.834
6.462
6.449
6.423
6.410
6.049
6.030
6.012
5.991
5.973
5.954
5.935
4.171
4.153
4.145
4.140
4.127
4.122
4.116
4.109
4.105
4.089
4.085
4.071
4.067
4.060
4.042
3.573
3.561
3.547
3.140
3.127
3.114
2.767
2.765
2.748
2.746
2.712
2.709
2.693
2.691
1.473
1.322
1.304
1.287
NBoc
N
O
EtO
P
EtO
(E)-5k
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
2.03
2.03
1.00
1.00
4.32
4.19
4.14
2.06
9.44
6.29

(*E*)-5k: ^{31}P NMR



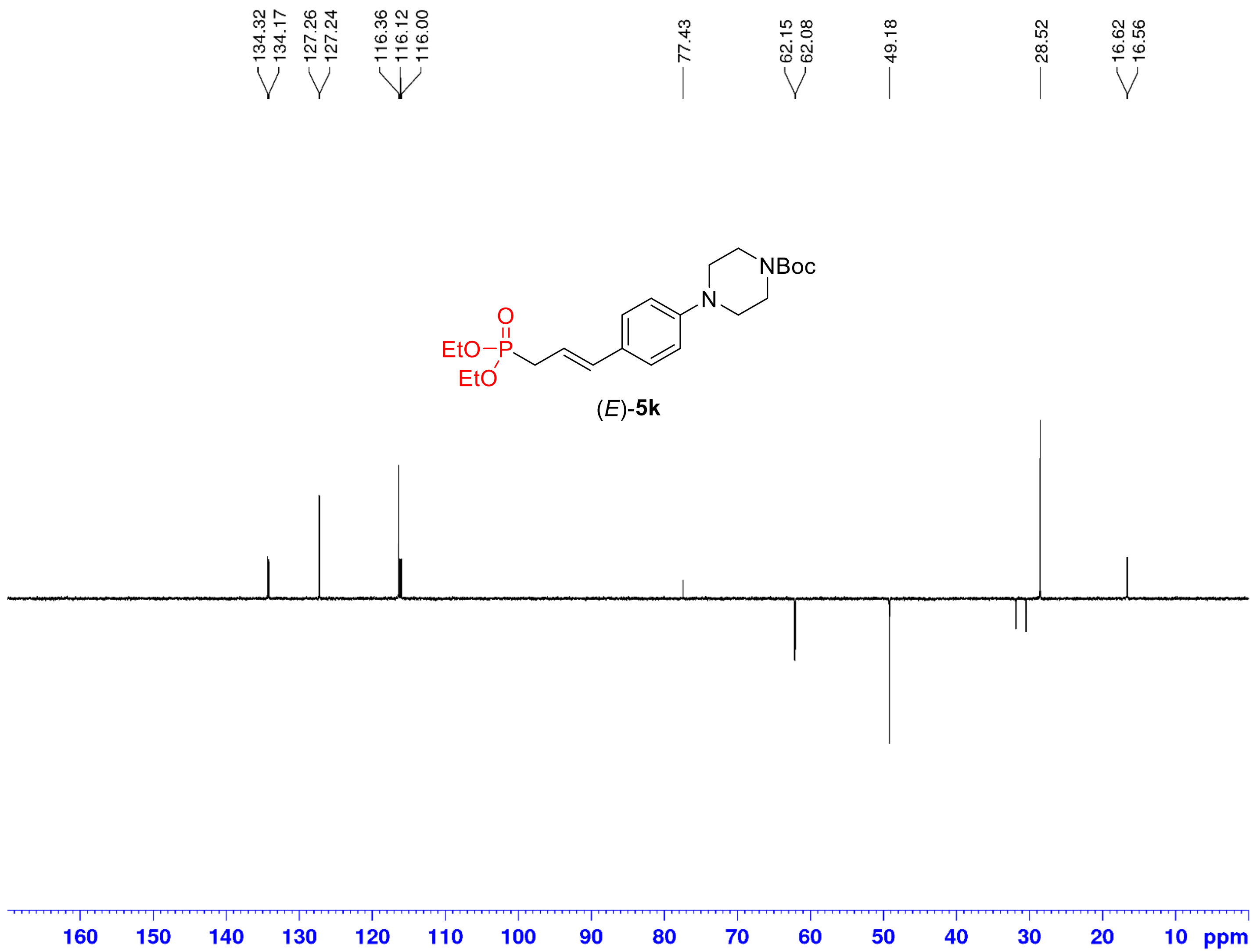
27.21
O
EtO
P
EtO
N
NBoc
(*E*)-**5k**
39
38
37
36
35
34
33
32
31
30
29
28
27
26
25
24
23
22
ppm

(*E*)-5k: ^{13}C NMR

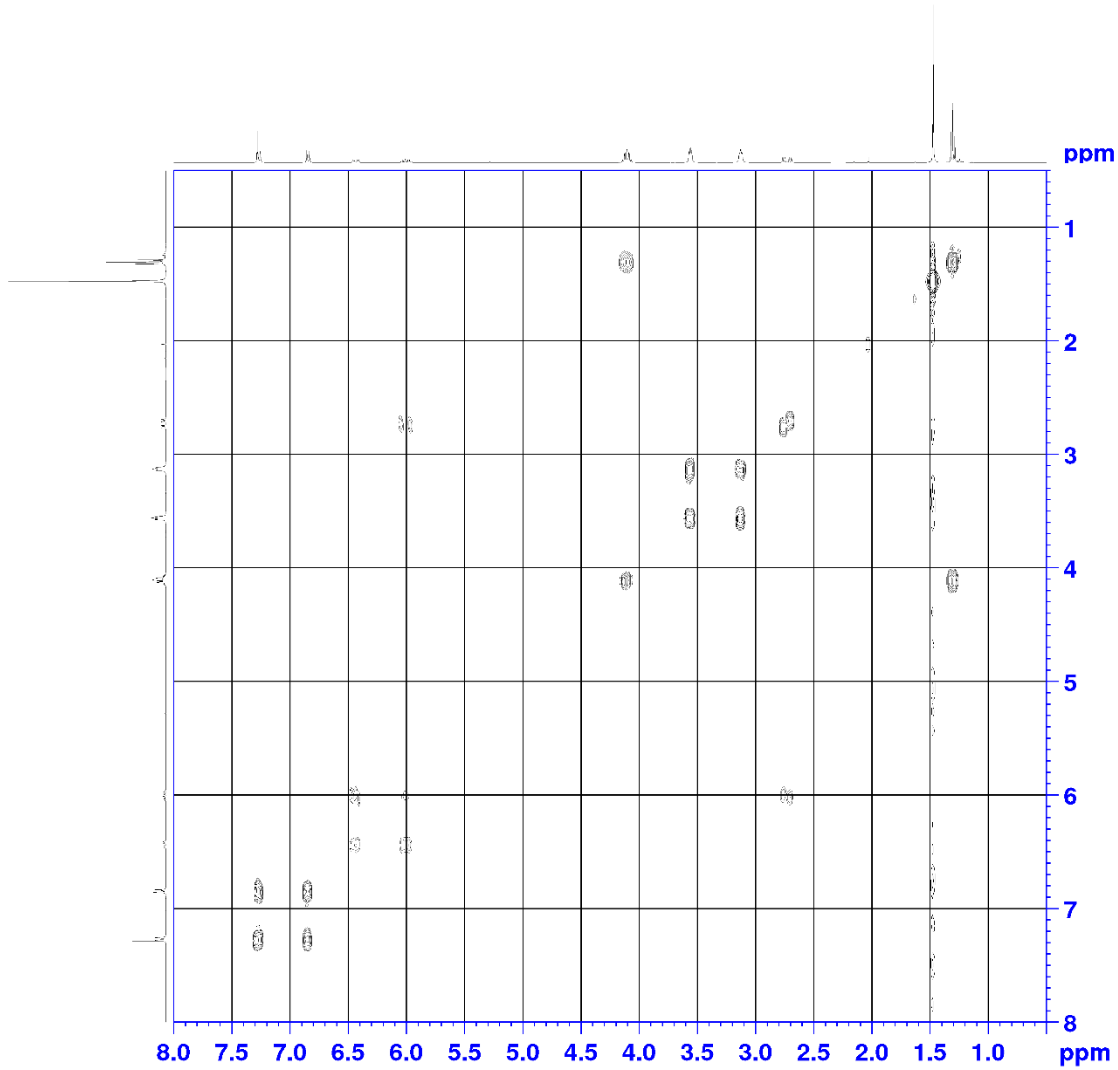


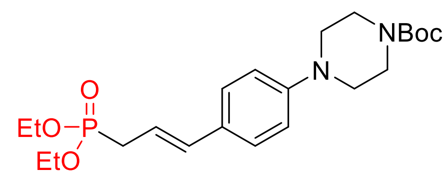
154.80
150.71
134.32
134.17
128.98
128.95
127.26
127.24
116.35
116.12
116.00
80.01
77.55
77.43
77.23
76.91
62.14
62.08
49.17
43.58
31.84
30.45
28.52
16.62
16.56
NBoc
N
O
EtO
P
EtO
(*E*)-**5k**
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

(*E*)-5k: ^{13}C DEPT135



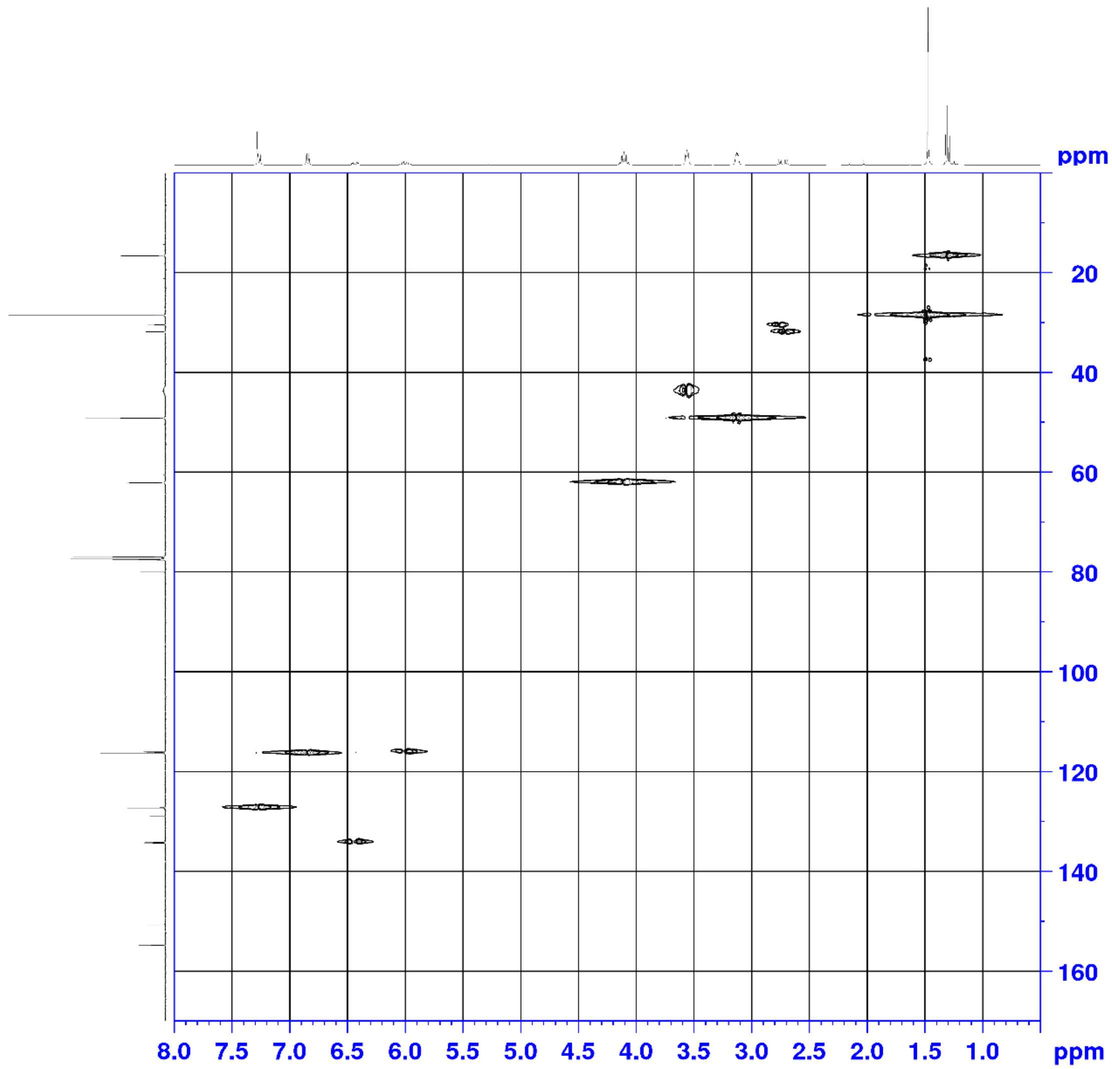
(*E*)-5k: COSY

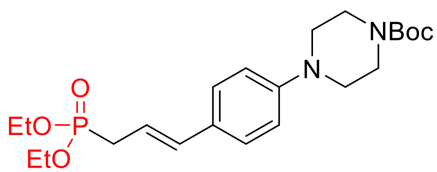

ppm
1
2
3
4
5
6
7
8
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm


NBoc
N
O
EtO
P
EtO

(*E*)-**5k**

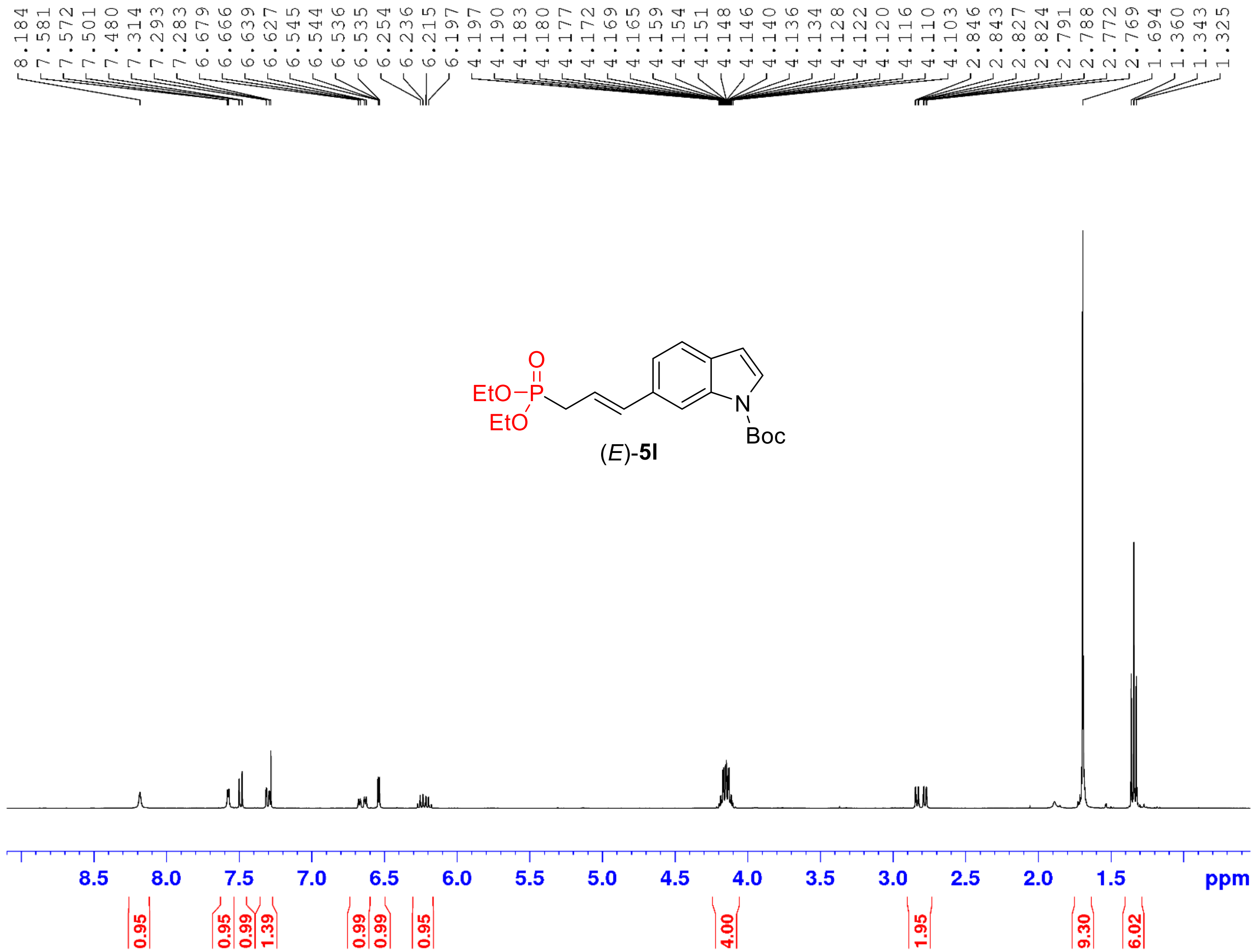
(*E*)-5k: ^{13}C HSQC



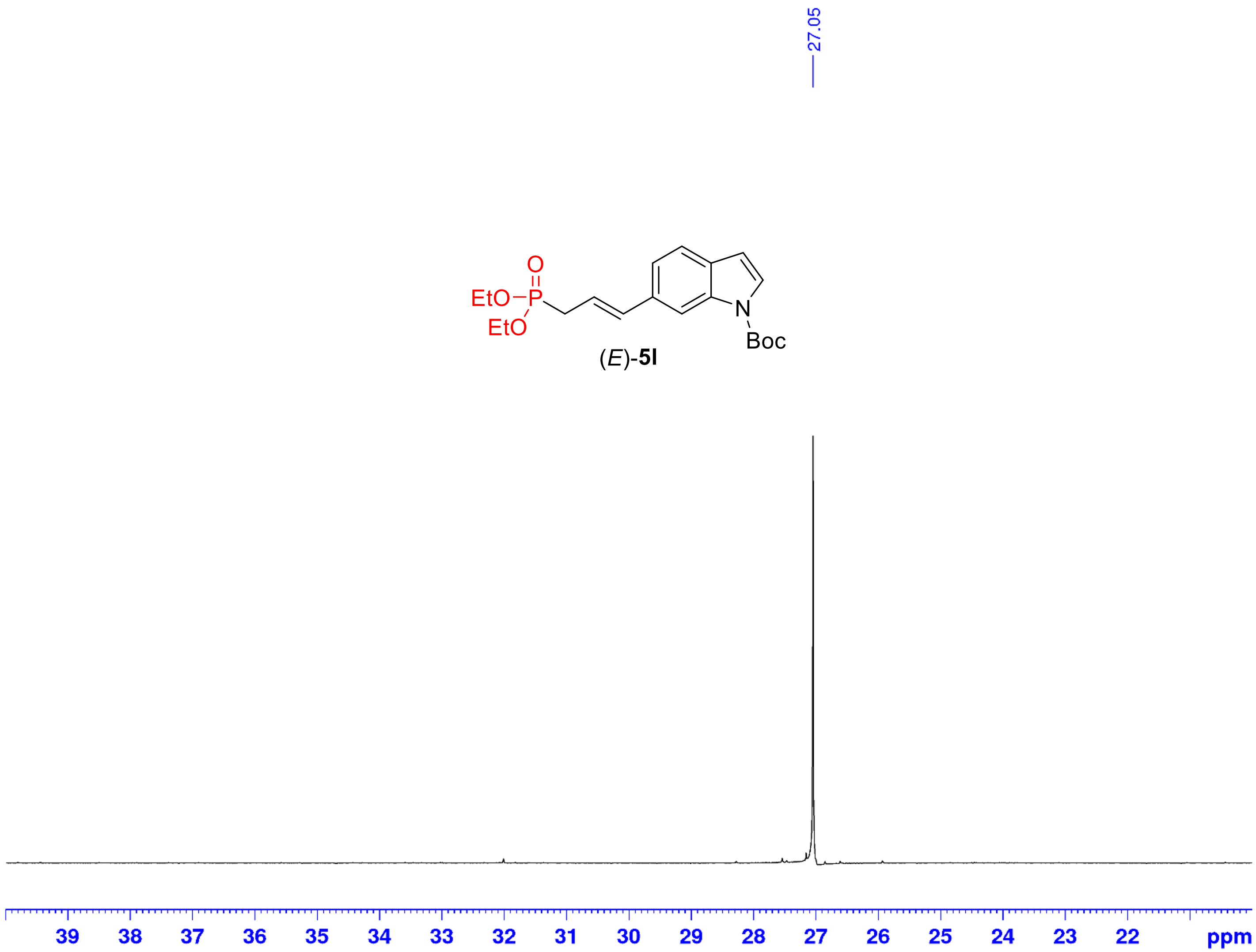


(*E*)-**5k**

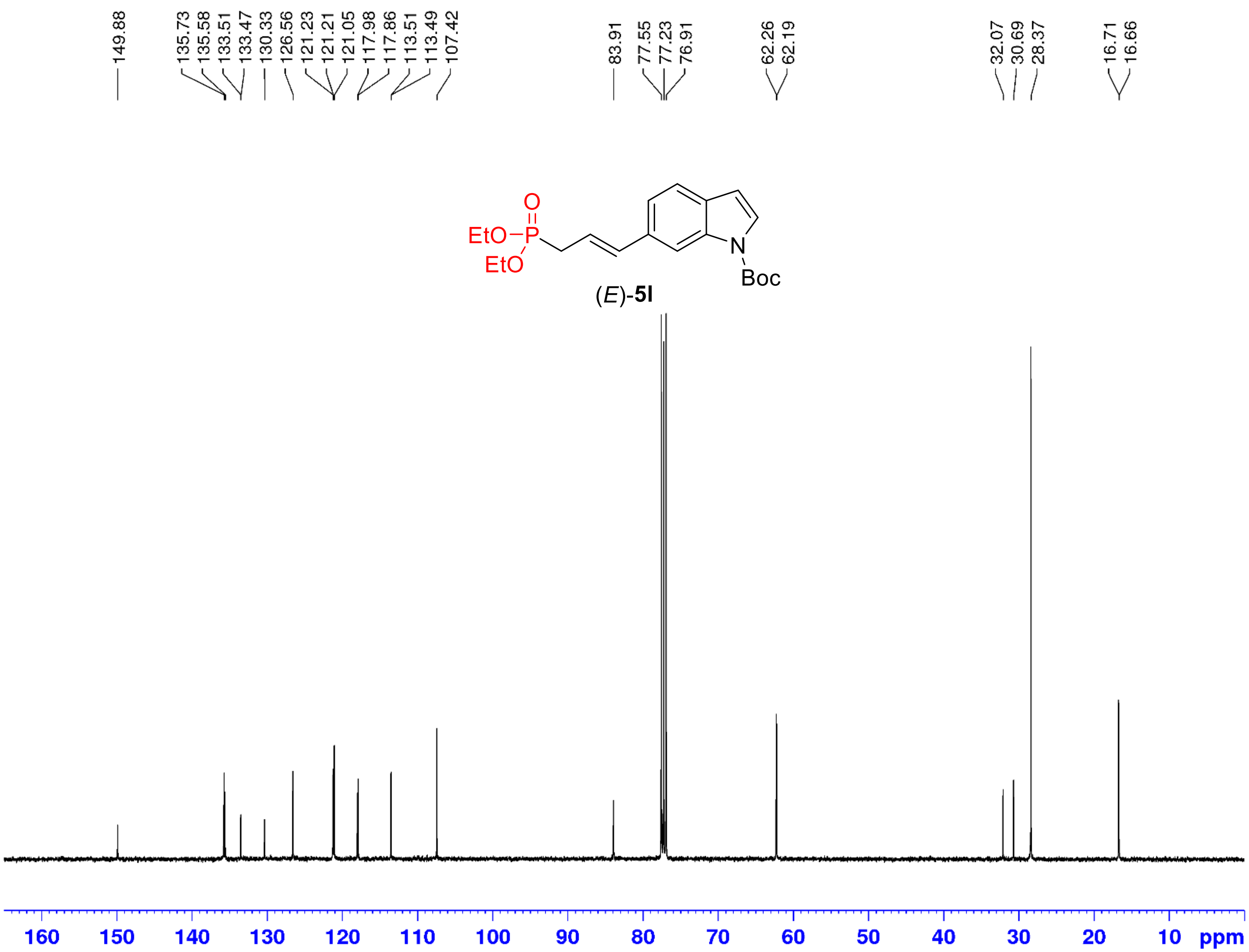
(*E*)-5l: ^{1}H NMR


8.184
7.581
7.572
7.501
7.480
7.314
7.293
7.283
6.679
6.666
6.639
6.627
6.545
6.544
6.536
6.535
6.254
6.236
6.215
6.197
4.197
4.190
4.183
4.180
4.177
4.172
4.169
4.165
4.159
4.154
4.151
4.148
4.146
4.140
4.136
4.134
4.128
4.122
4.120
4.116
4.110
4.103
2.846
2.843
2.827
2.824
2.791
2.788
2.772
2.769
1.694
1.360
1.343
1.325
EtO
EtO
O
P
N
Boc
(E)-5l
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
ppm
0.95
0.95
0.99
1.39
0.99
0.99
0.95
4.00
1.95
9.30
6.02

(*E*)-5l: ^{31}P NMR

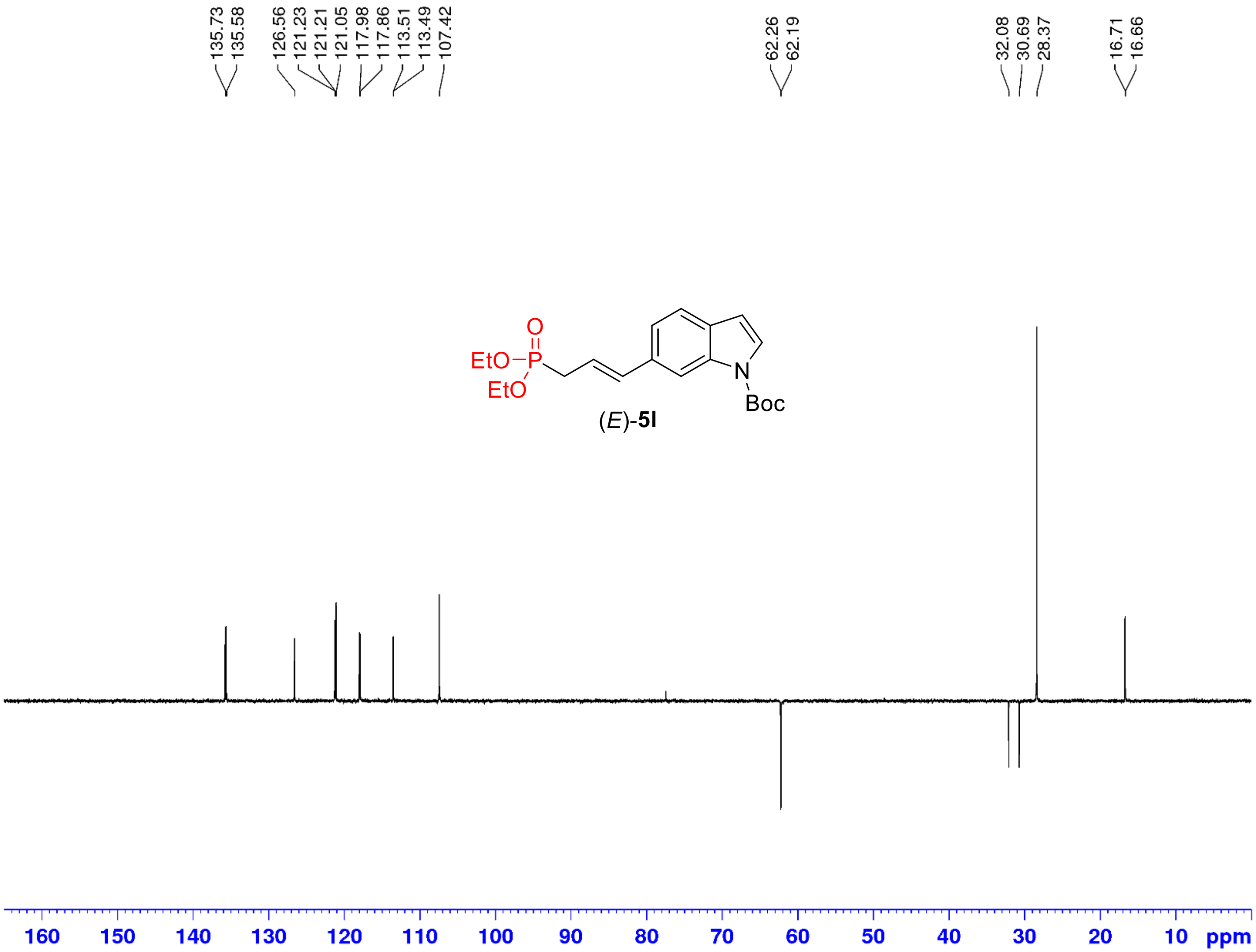

27.05
EtO
EtO
O
P
N
Boc
(E)-5l
39
38
37
36
35
34
33
32
31
30
29
28
27
26
25
24
23
22
ppm

(*E*)-5l: ^{13}C NMR

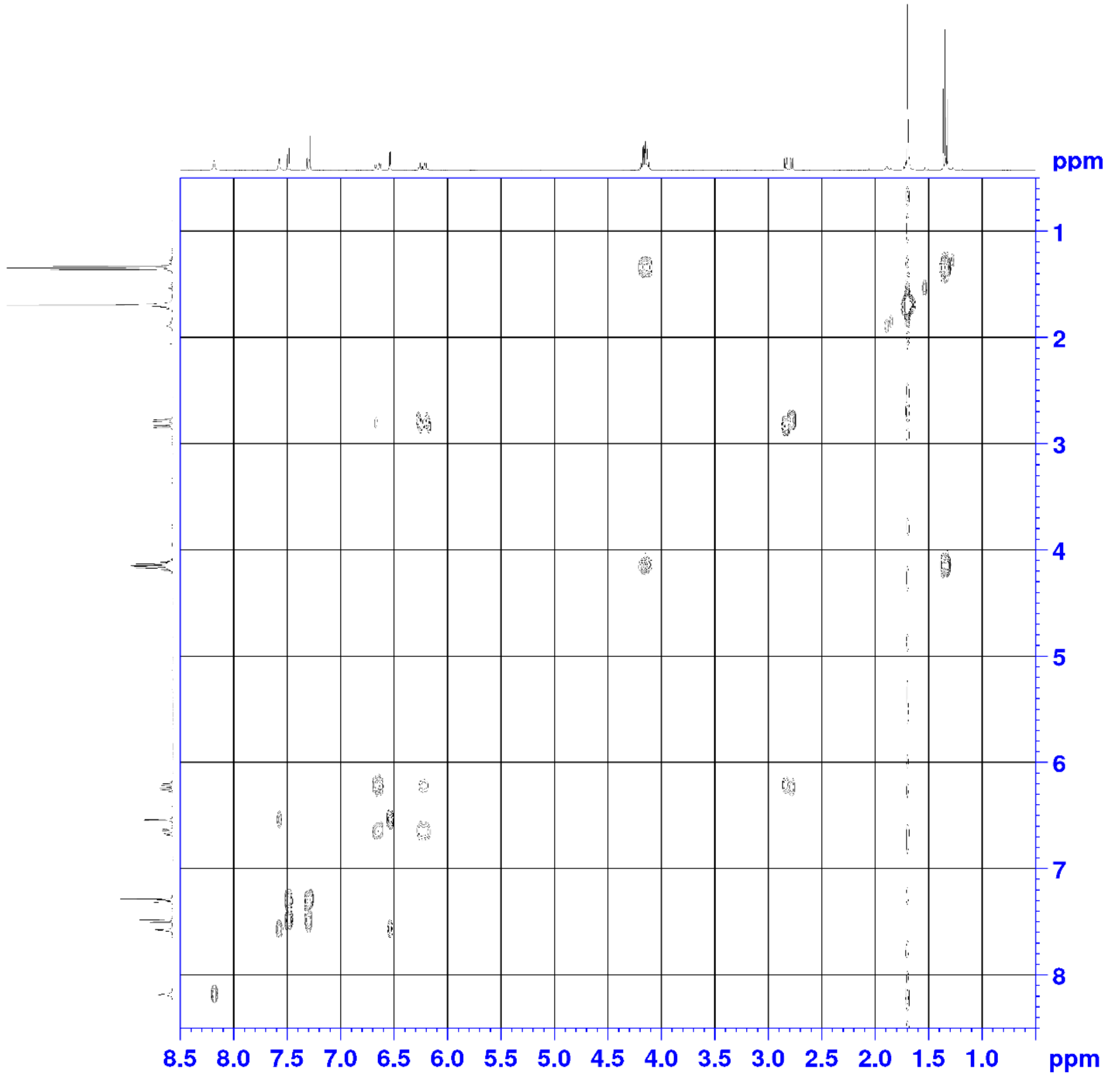


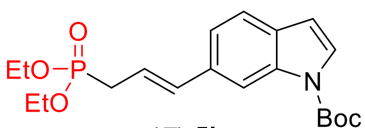
149.88
135.73
135.58
133.51
133.47
130.33
126.56
121.23
121.21
121.05
117.98
117.86
113.51
113.49
107.42
83.91
77.55
77.23
76.91
62.26
62.19
32.07
30.69
28.37
16.71
16.66
O
EtO
P
EtO
N
Boc
(*E*)-**5l**
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

(*E*)-5l: ^{13}C DEPT135



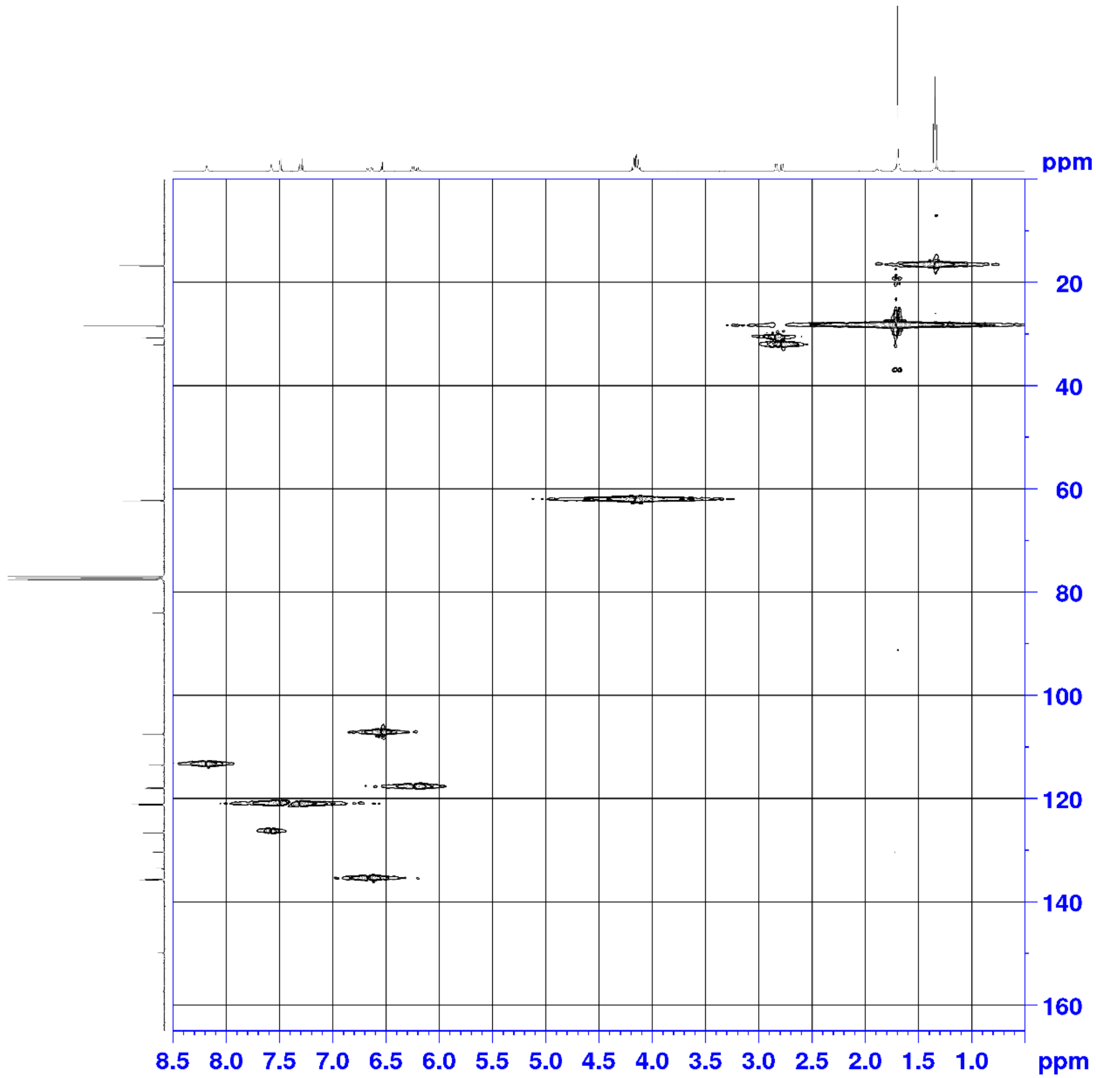
(*E*)-5l: COSY

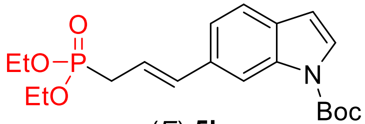

ppm
1
2
3
4
5
6
7
8
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm


O
EtO
P
EtO
N
Boc

(*E*)-**5l**

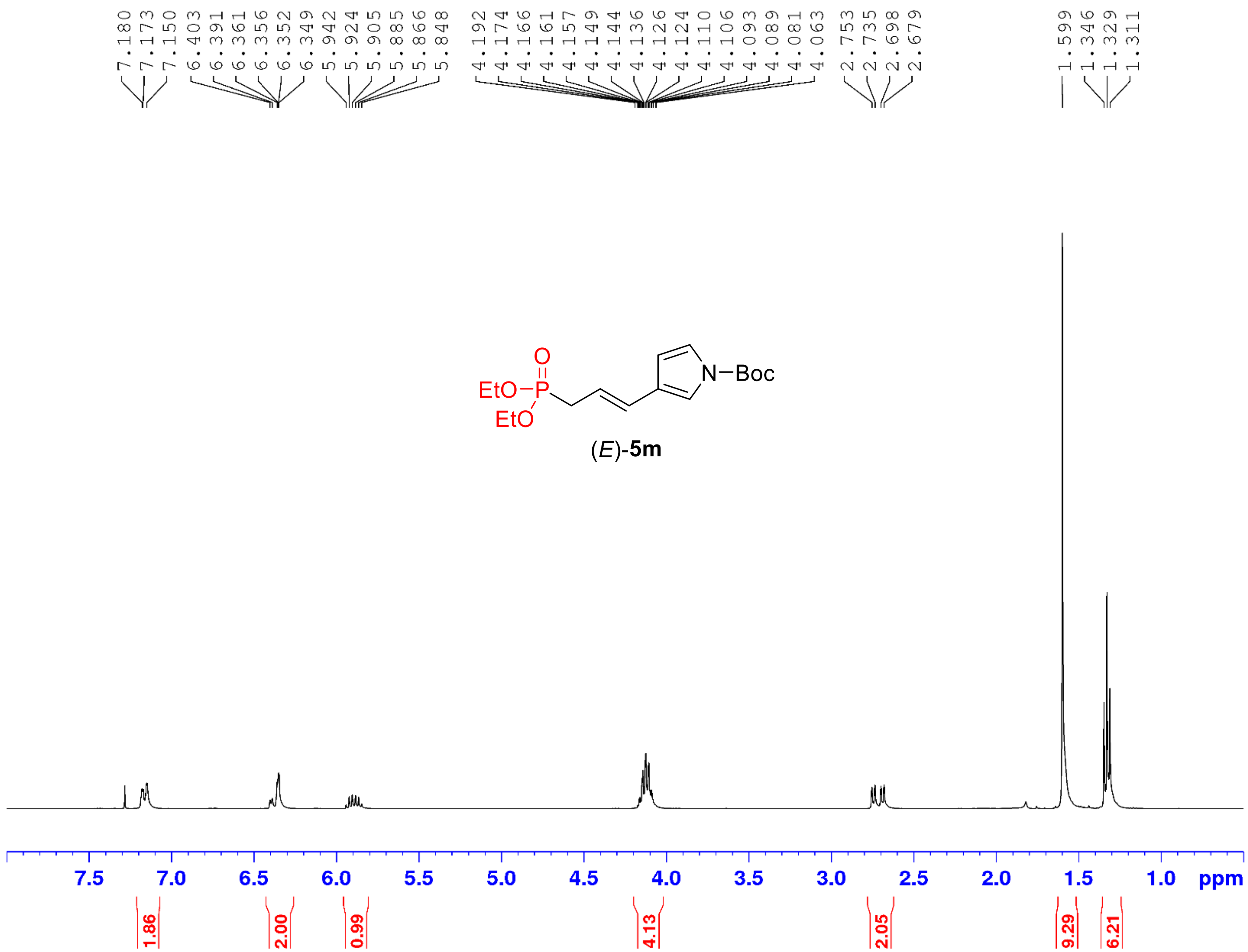
(*E*)-5l: ^{13}C HSQC





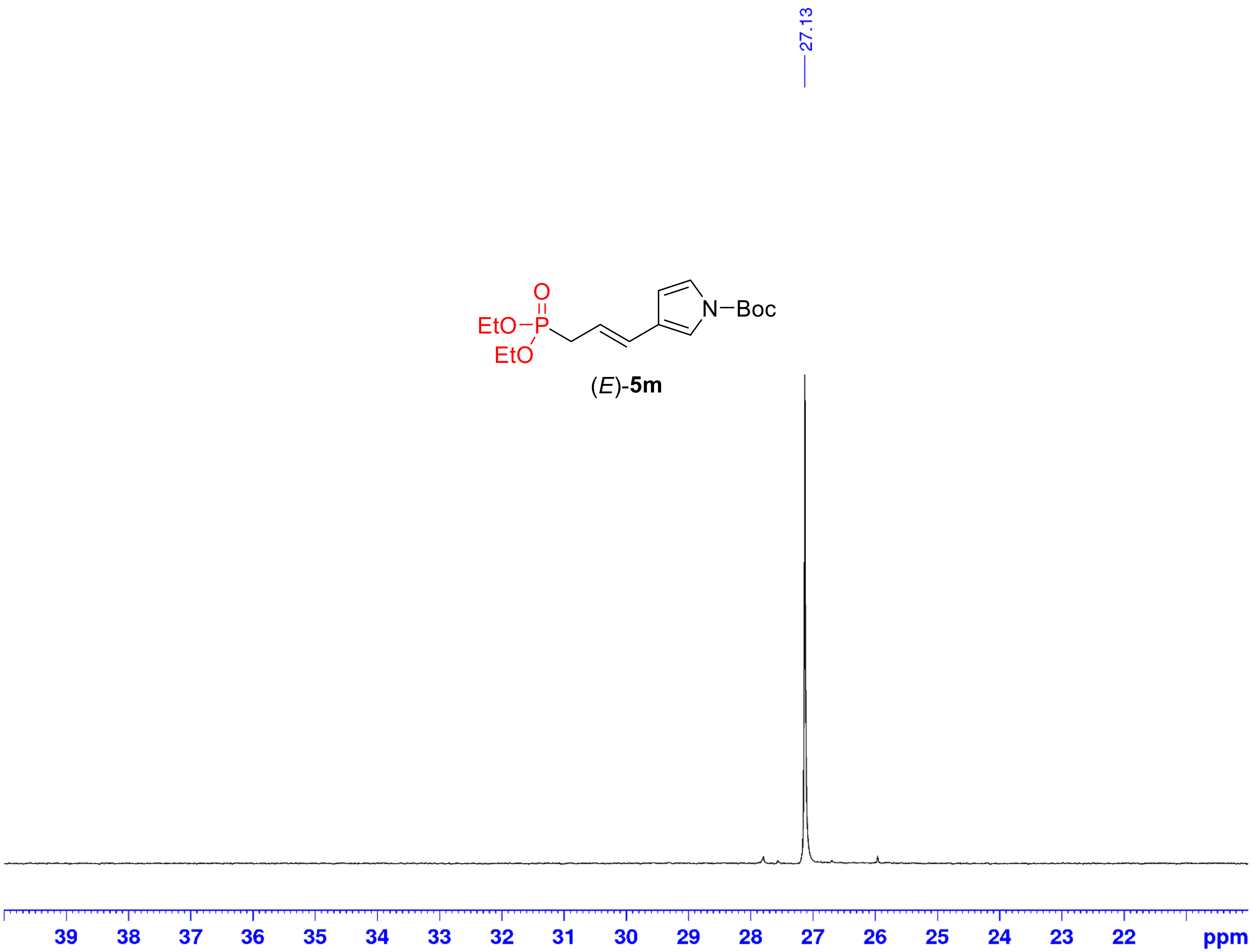
(*E*)-**5l**

(*E*)-5m: ^{1}H NMR

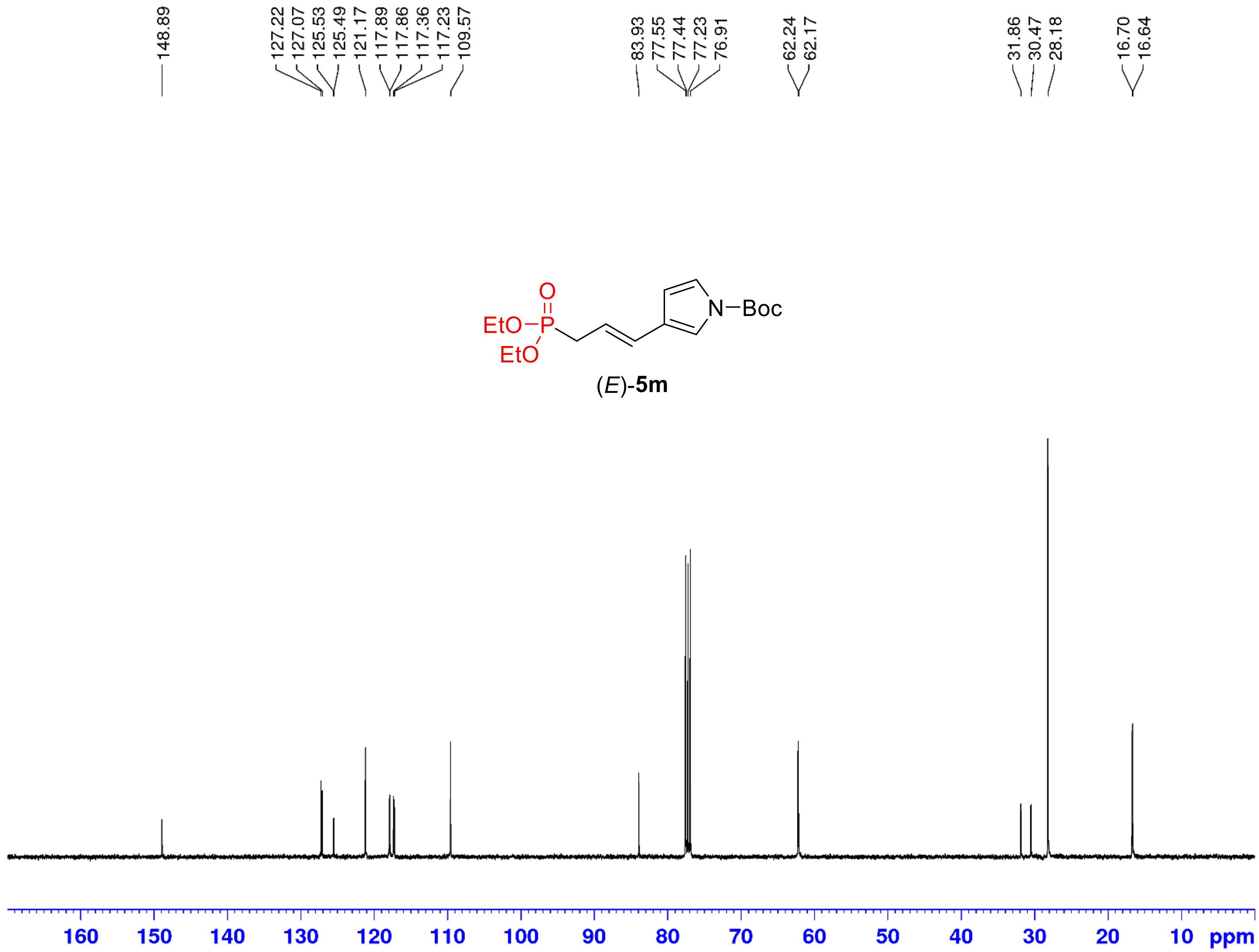


7.180
7.173
7.150
6.403
6.391
6.361
6.356
6.352
6.349
5.942
5.924
5.905
5.885
5.866
5.848
4.192
4.174
4.166
4.161
4.157
4.149
4.144
4.136
4.126
4.124
4.110
4.106
4.093
4.089
4.081
4.063
2.753
2.735
2.698
2.679
1.599
1.346
1.329
1.311
O
EtO
P
EtO
N
Boc
(E)-5m
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
1.86
2.00
0.99
4.13
2.05
9.29
6.21

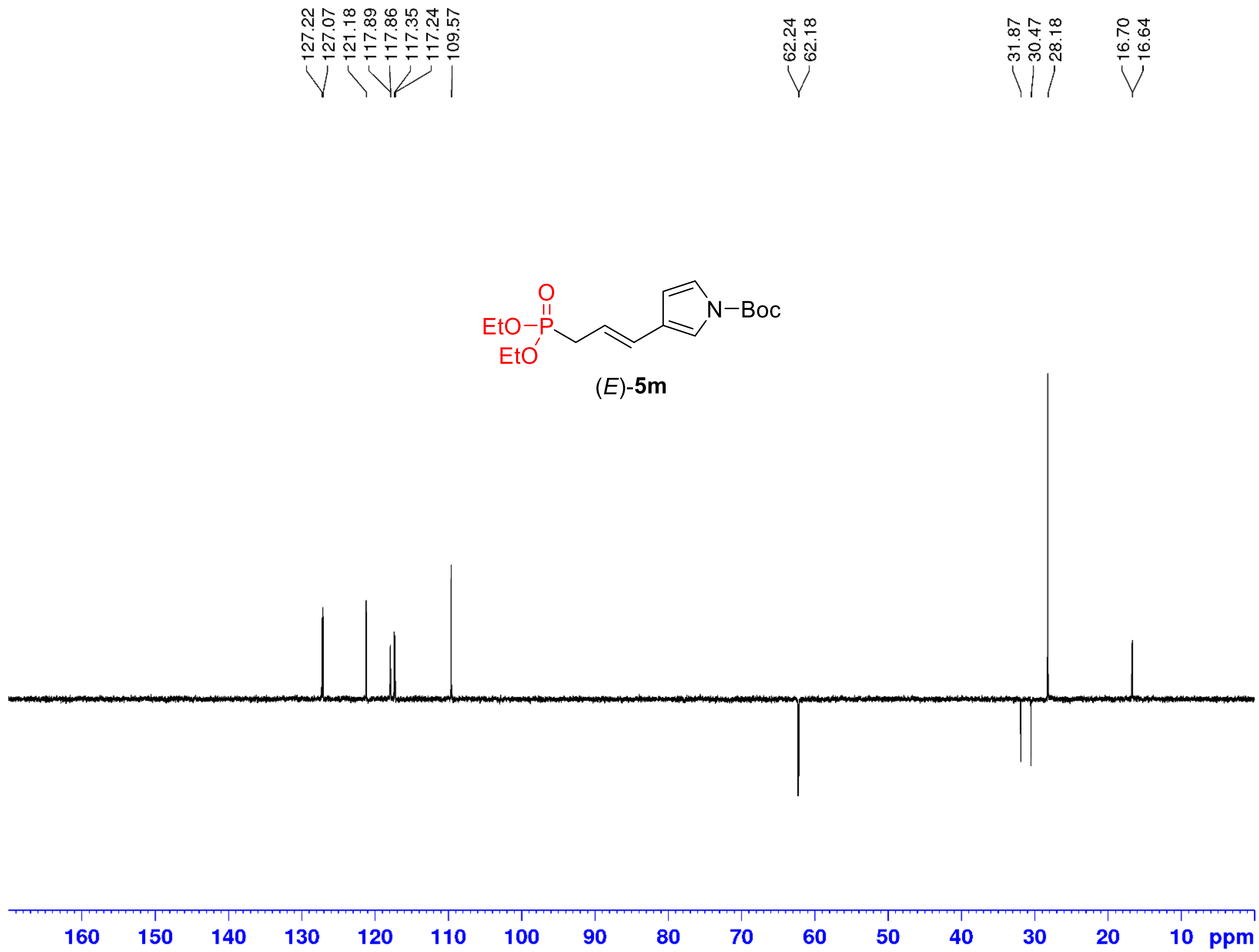
(*E*)-5m: ^{31}P NMR


27.13
EtO
EtO
O
P
N
Boc
(*E*)-**5m**
39
38
37
36
35
34
33
32
31
30
29
28
27
26
25
24
23
22
ppm

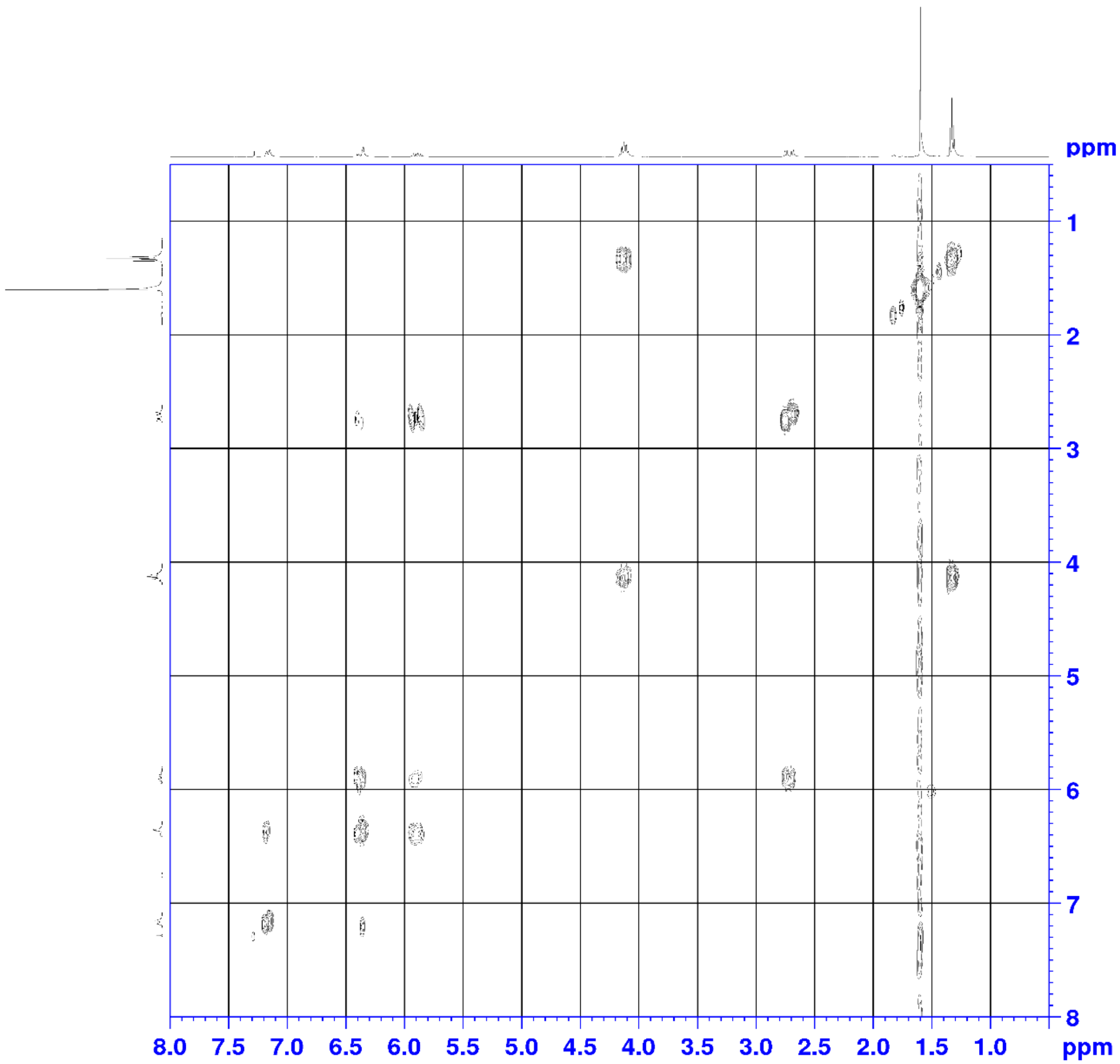
(*E*)-5m: ^{13}C NMR

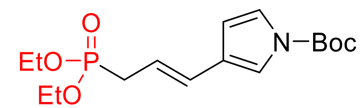

148.89
127.22
127.07
125.53
125.49
121.17
117.89
117.86
117.36
117.23
109.57
83.93
77.55
77.44
77.23
76.91
62.24
62.17
31.86
30.47
28.18
16.70
16.64
O
EtO
P
EtO
N
Boc
(*E*)-**5m**
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

(*E*)-5m: ^{13}C DEPT135



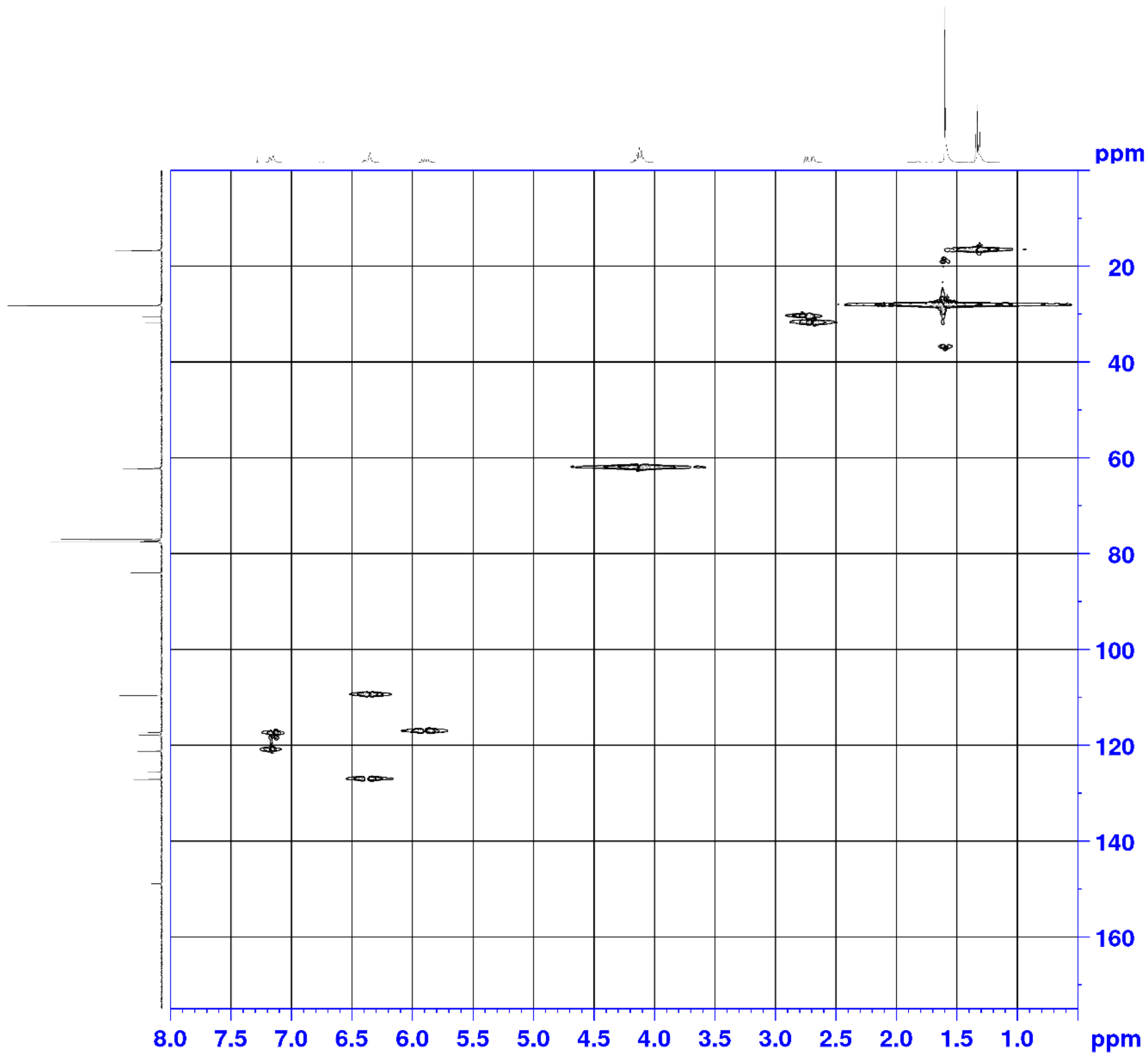
(*E*)-5m: COSY

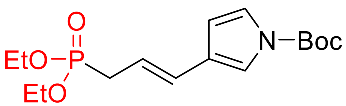

ppm
1
2
3
4
5
6
7
8
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm


O
EtO
P
EtO
N
Boc

(*E*)-**5m**

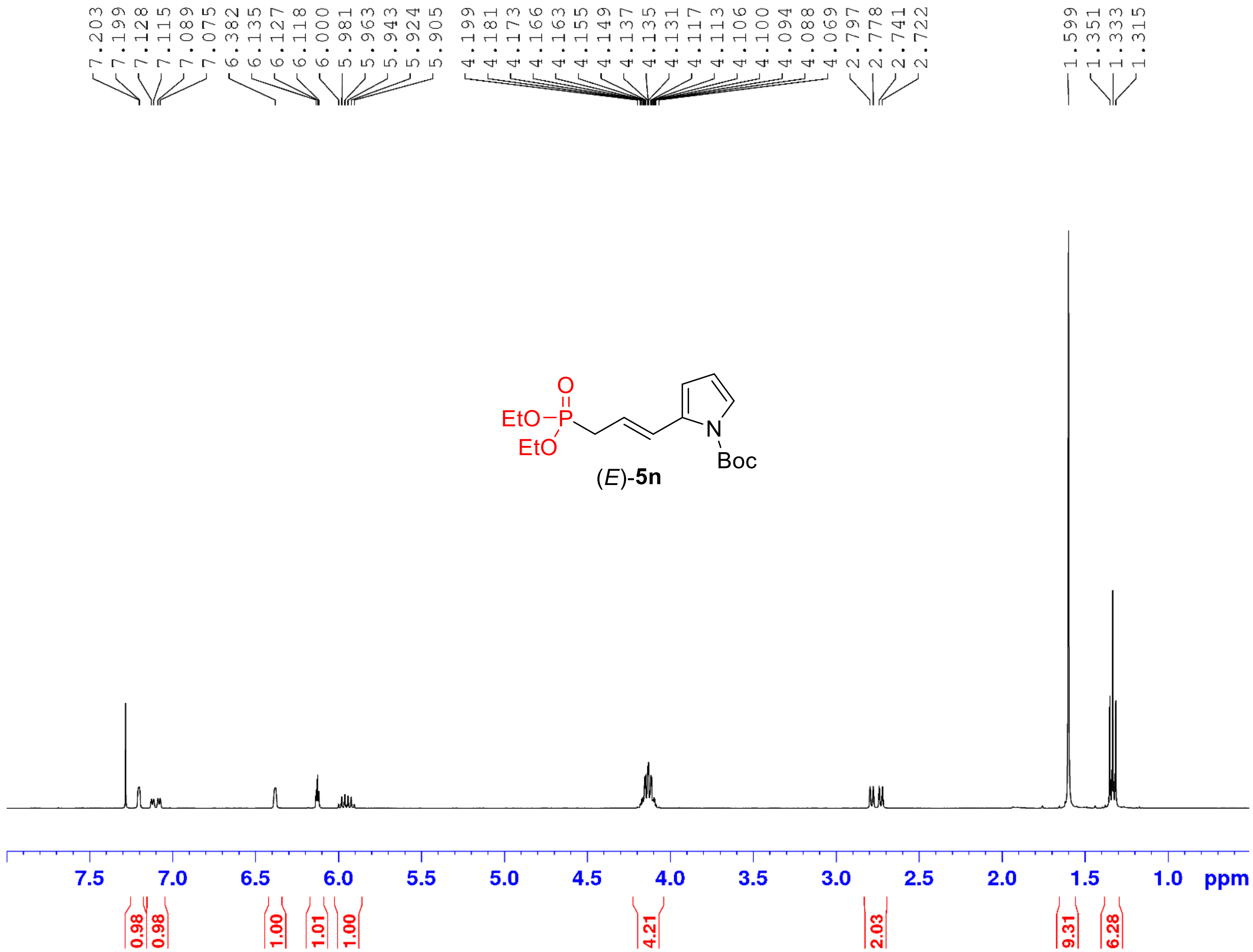
(*E*)-5m: ^{13}C-HSQC



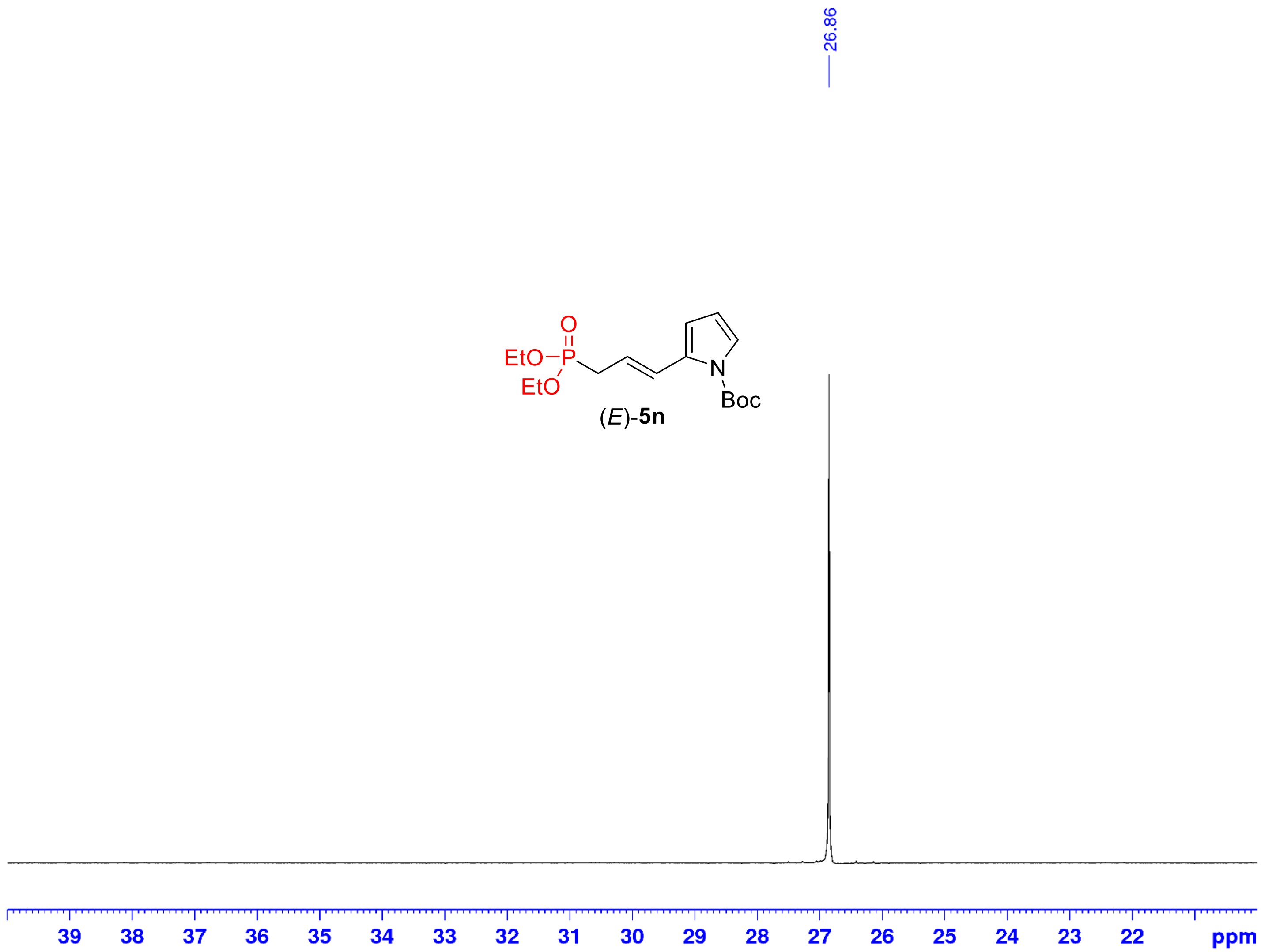


(*E*)-**5m**

(*E*)-5n: ^{1}H NMR

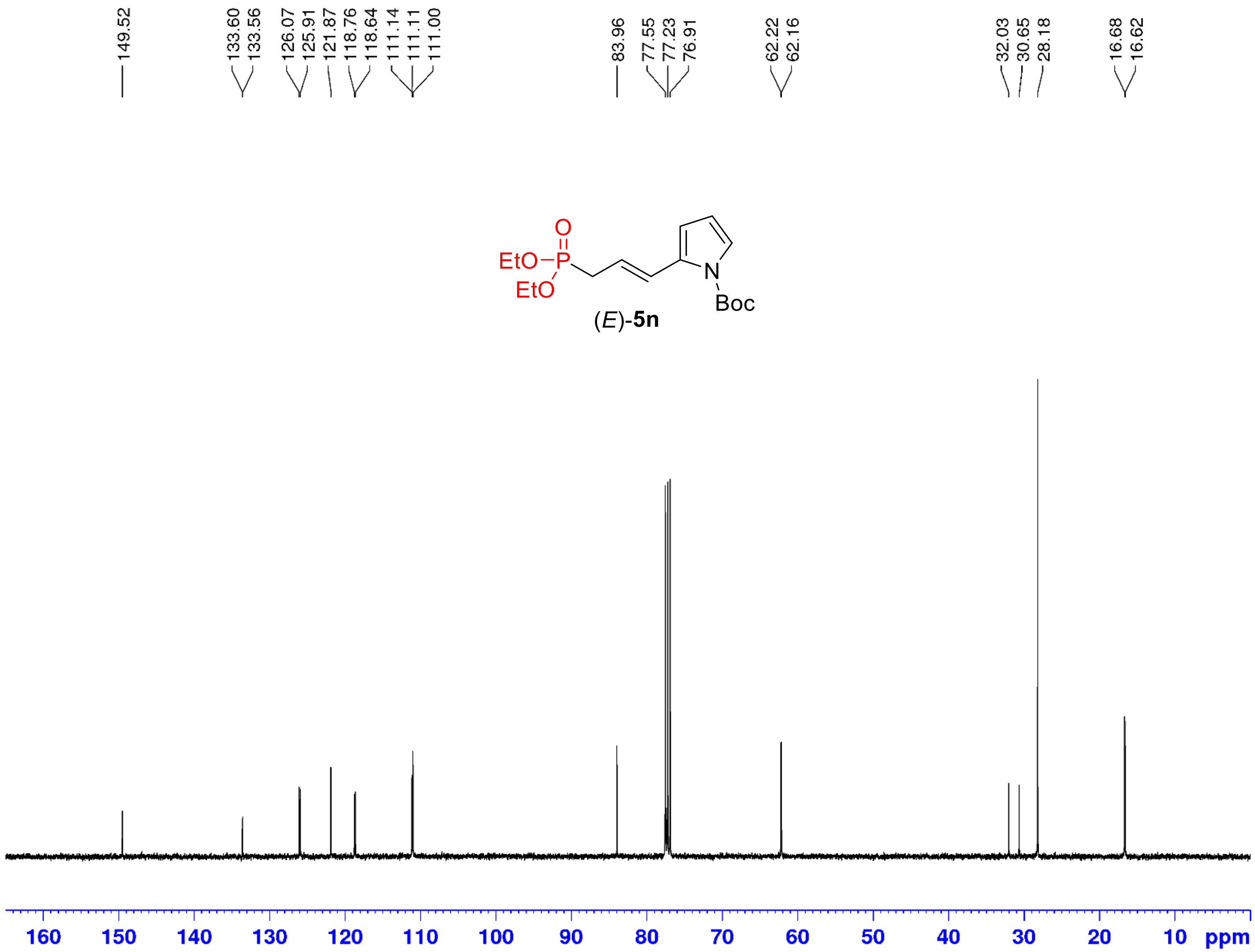

7.203
7.199
7.128
7.115
7.089
7.075
6.382
6.135
6.127
6.118
6.000
5.981
5.963
5.943
5.924
5.905
4.199
4.181
4.173
4.166
4.163
4.155
4.149
4.137
4.135
4.131
4.117
4.113
4.106
4.100
4.094
4.088
4.069
2.797
2.778
2.741
2.722
1.599
1.351
1.333
1.315
EtO
EtO
O
P
N
Boc
(E)-5n
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
0.98
0.98
1.00
1.01
1.00
4.21
2.03
9.31
6.28

(*E*)-5n: ^{31}P NMR



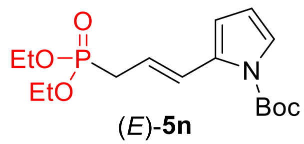
26.86
O
EtO
P
EtO
N
Boc
(E)-5n
39 38 37 36 35 34 33 32 31 30 29 28 27 26 25 24 23 22
ppm

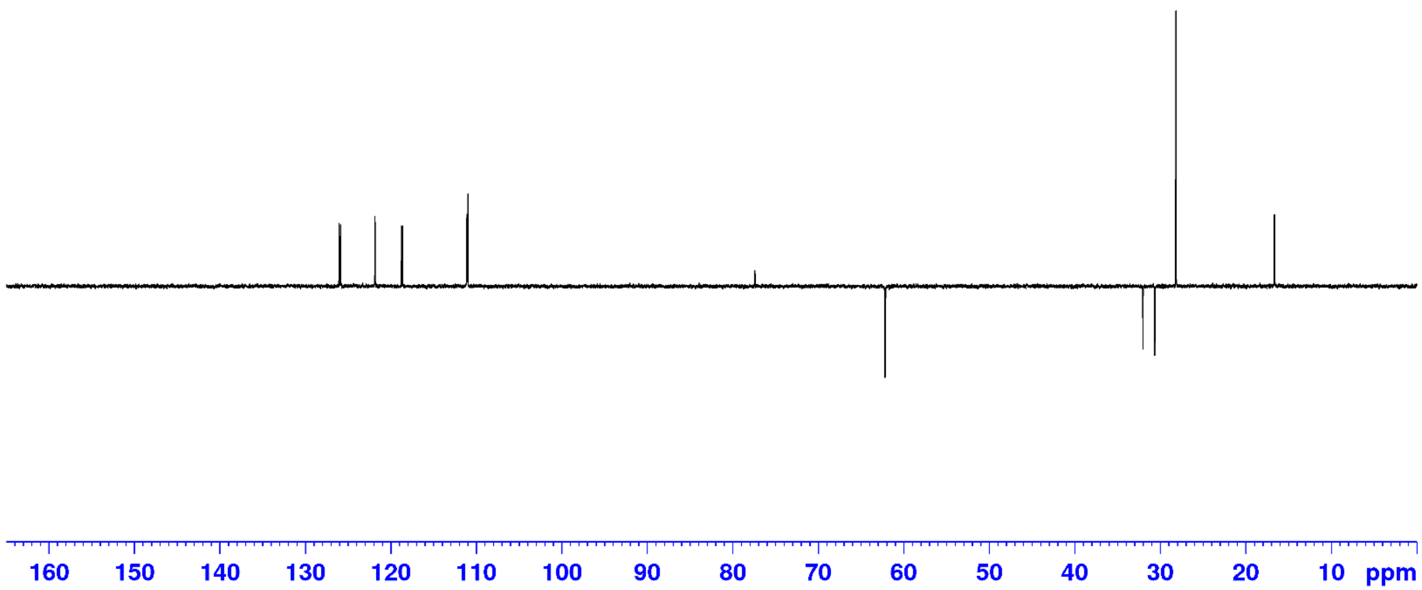
(*E*)-5n: ^{13}C NMR



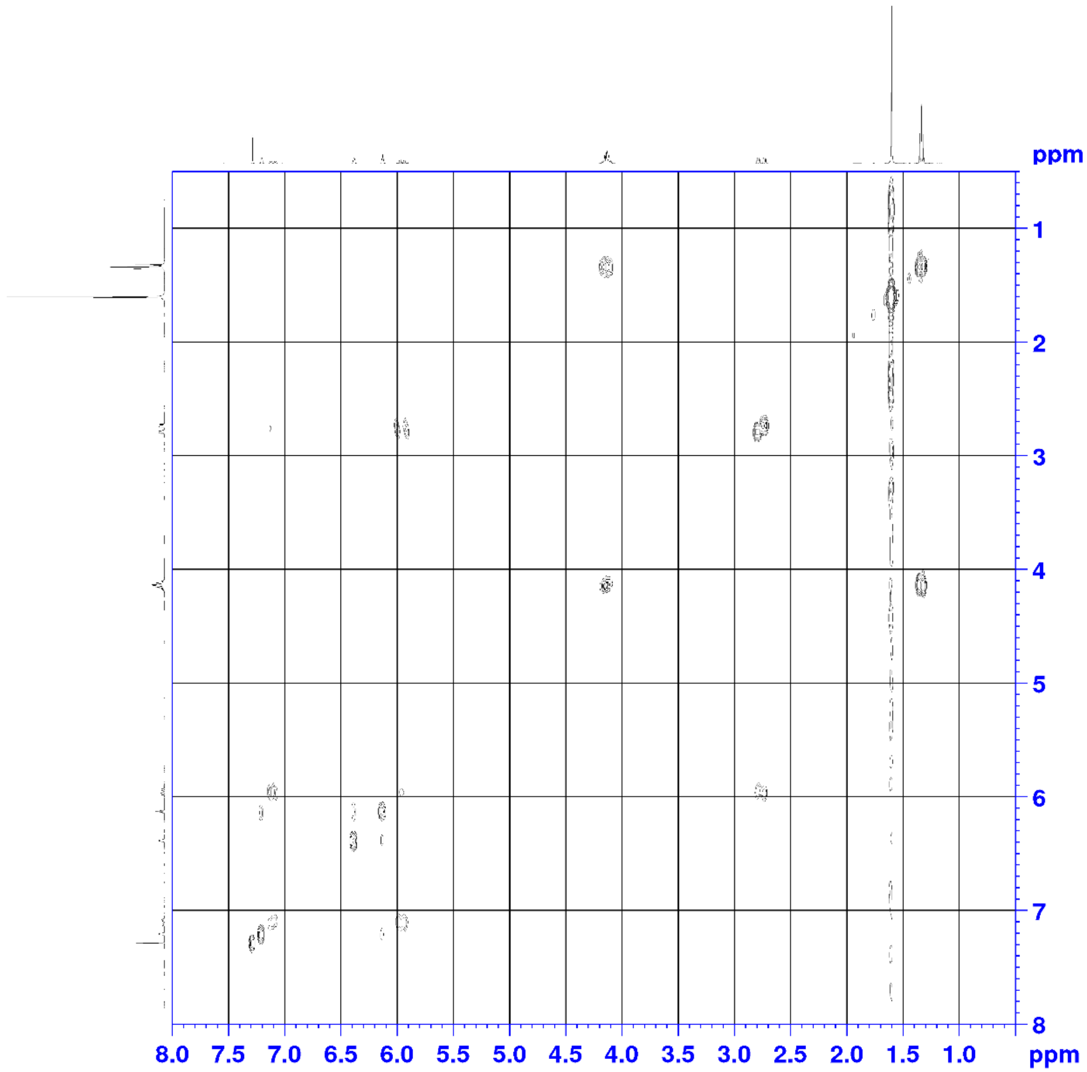
149.52
133.60
133.56
126.07
125.91
121.87
118.76
118.64
111.14
111.11
111.00
83.96
77.55
77.23
76.91
62.22
62.16
32.03
30.65
28.18
16.68
16.62
O
EtO
P
EtO
N
Boc
(*E*)-**5n**
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

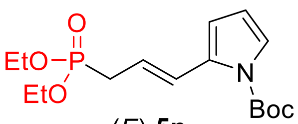
(E)-5n: ^{13}C DEPT135





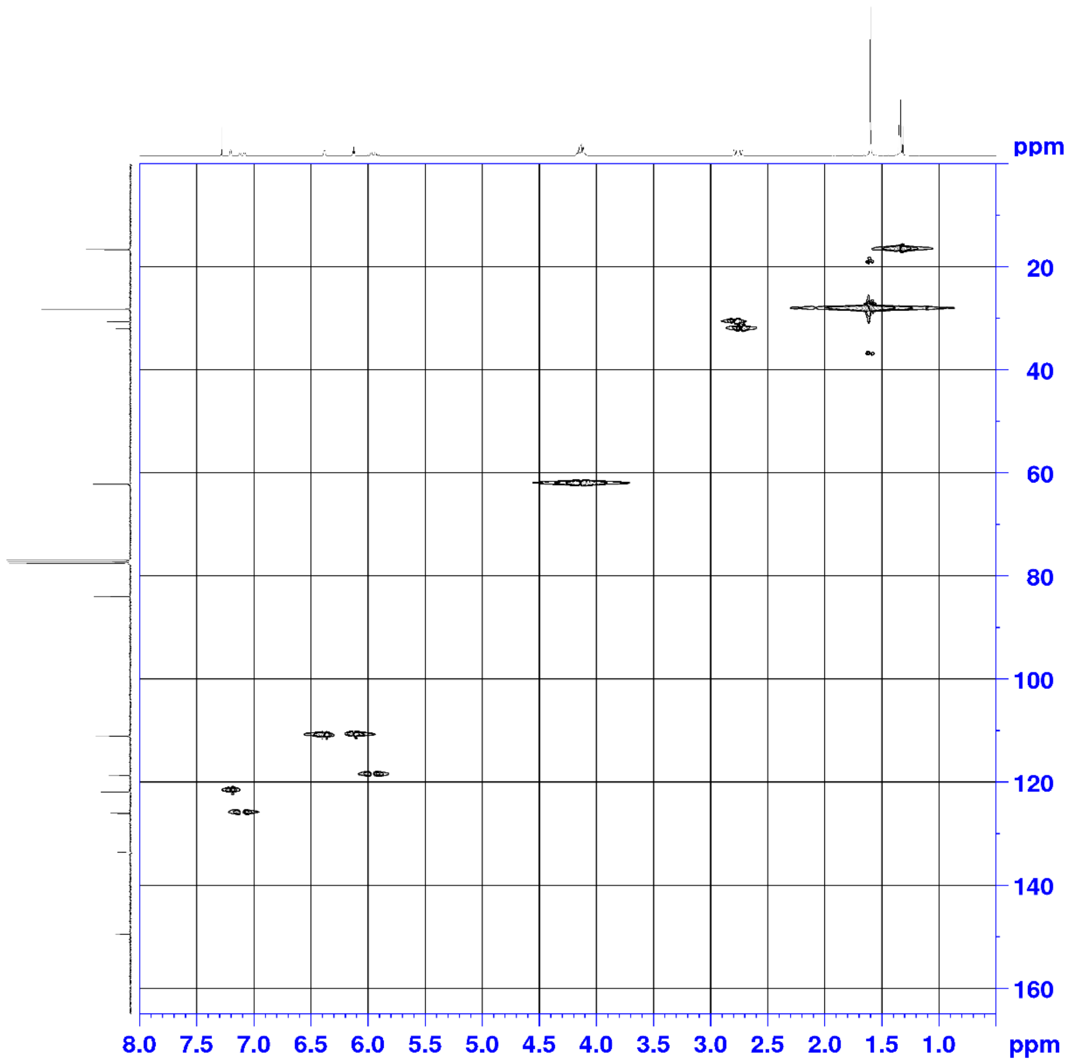
(*E*)-5n: COSY

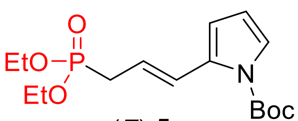

ppm
1
2
3
4
5
6
7
8
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm


O
EtO
P
EtO
N
Boc

(*E*)-**5n**

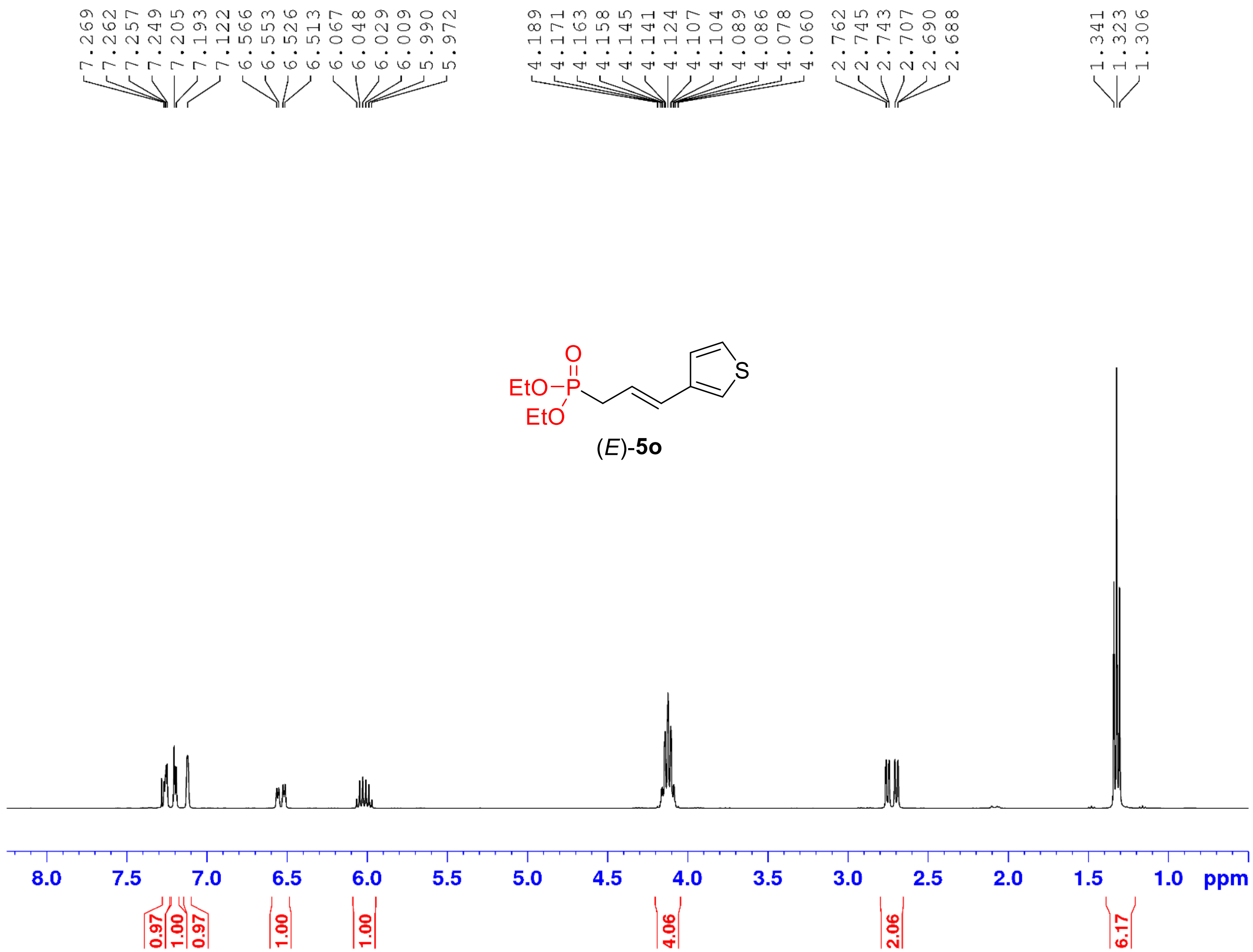
(*E*)-5n: ^{13}C HSQC



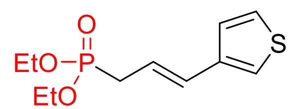


(*E*)-**5n**

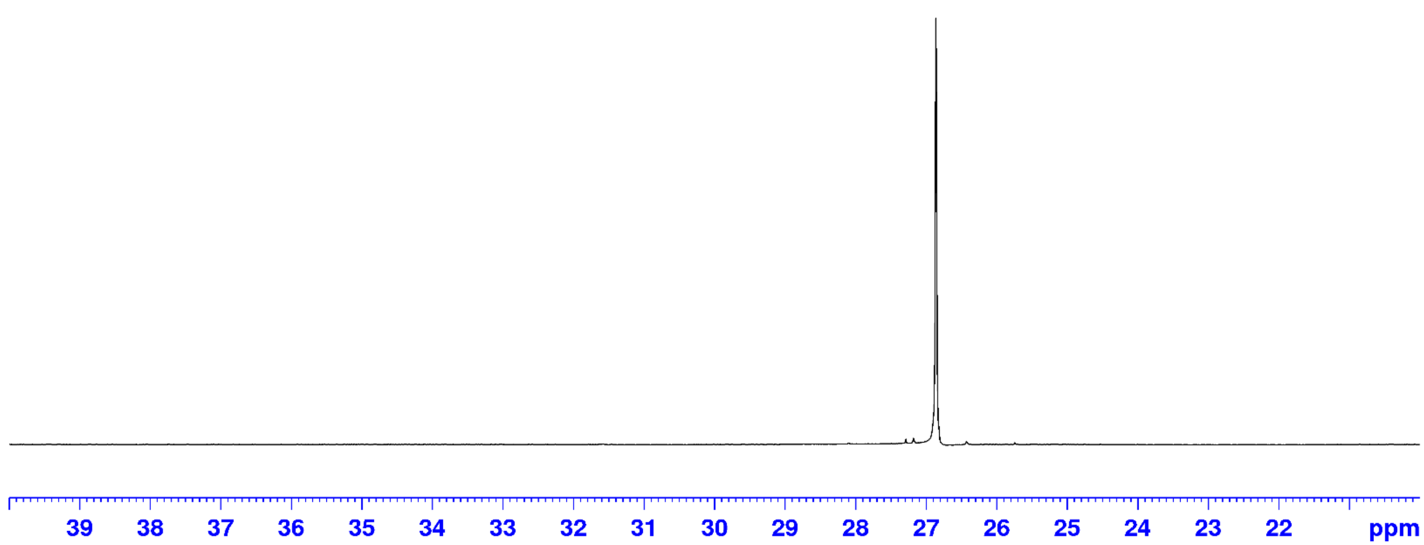
(*E*)-5o: ^{1}H NMR


7.269
7.262
7.257
7.249
7.205
7.193
7.122
6.566
6.553
6.526
6.513
6.067
6.048
6.029
6.009
5.990
5.972
4.189
4.171
4.163
4.158
4.145
4.141
4.124
4.107
4.104
4.089
4.086
4.078
4.060
2.762
2.745
2.743
2.707
2.690
2.688
1.341
1.323
1.306
O
EtO
P
EtO
S
(E)-5o
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
ppm
0.97
1.00
0.97
1.00
1.00
4.06
2.06
6.17

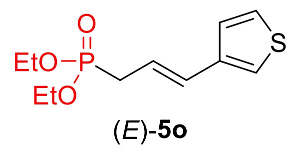
(*E*)-5o: ^{31}P NMR

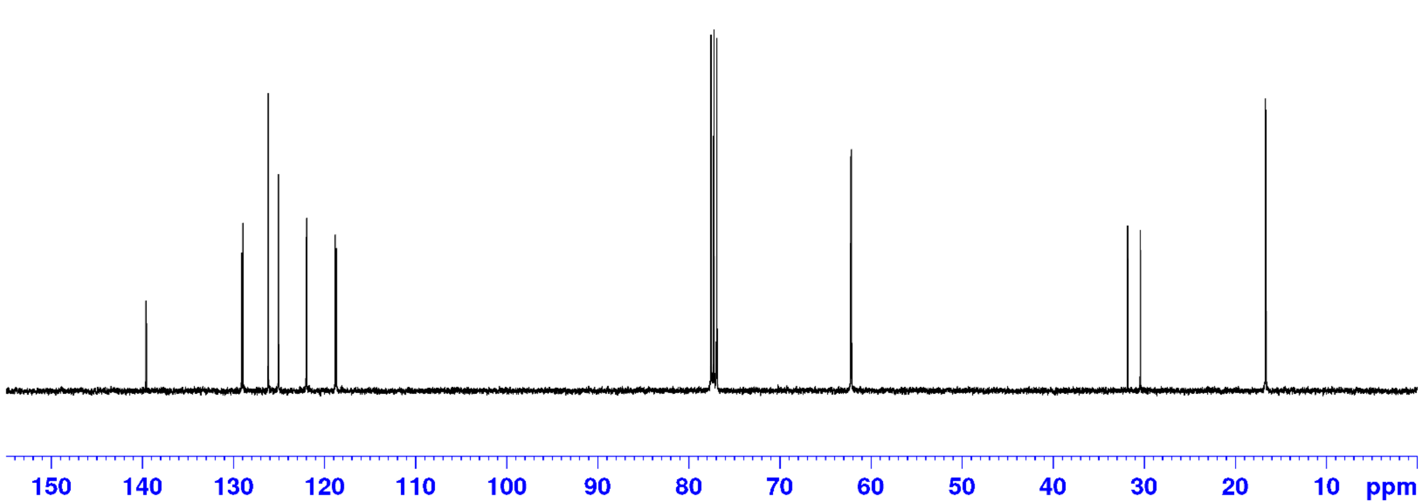


(*E*)-**5o**

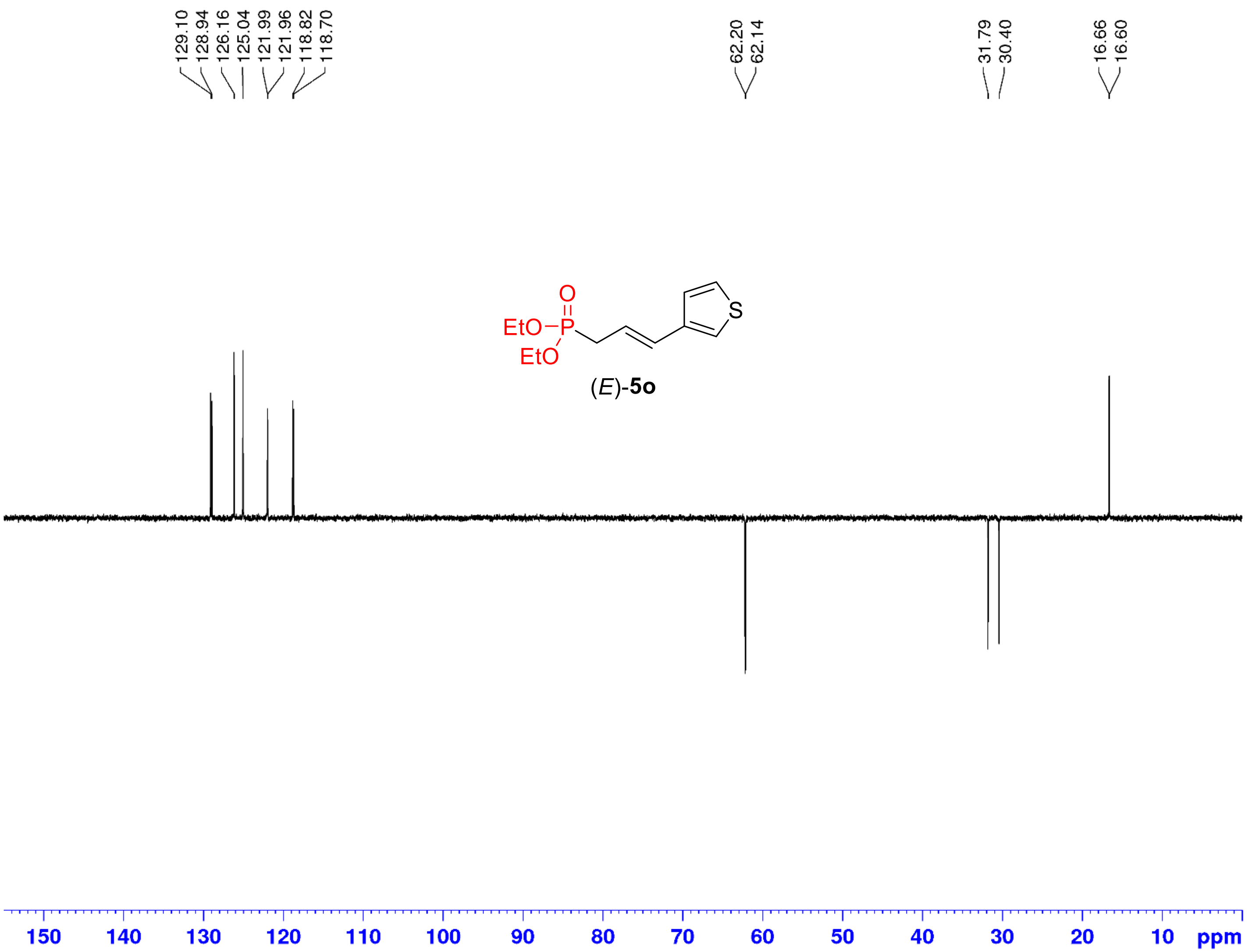


(*E*)-5o: ^{13}C NMR

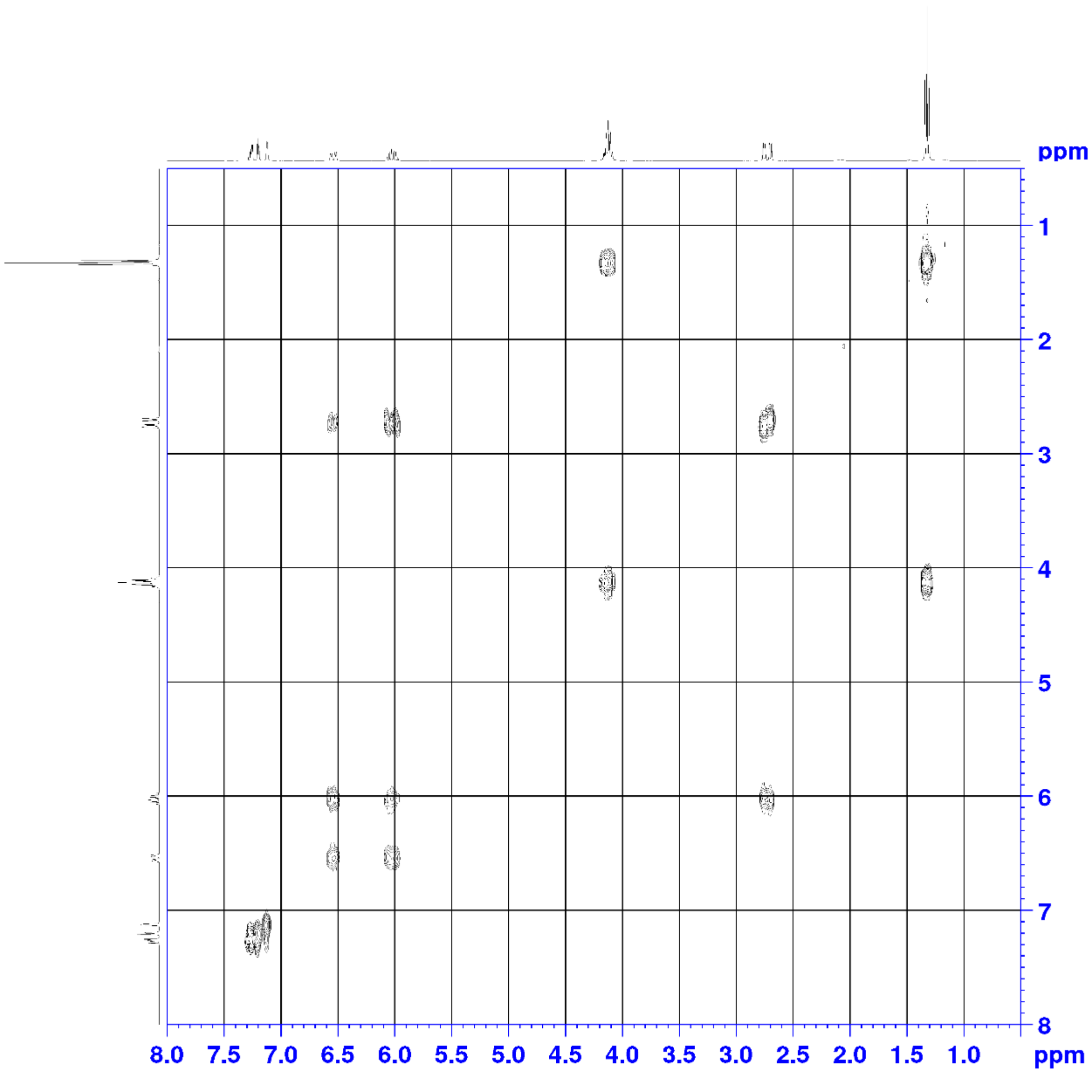


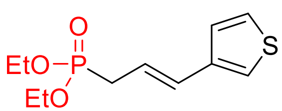

139.59
139.55
129.09
128.95
126.16
125.05
121.99
121.96
118.82
118.70
77.55
77.23
76.91
62.20
62.13
31.79
30.40
16.66
16.60
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

(*E*)-5o: ^{13}C DEPT135



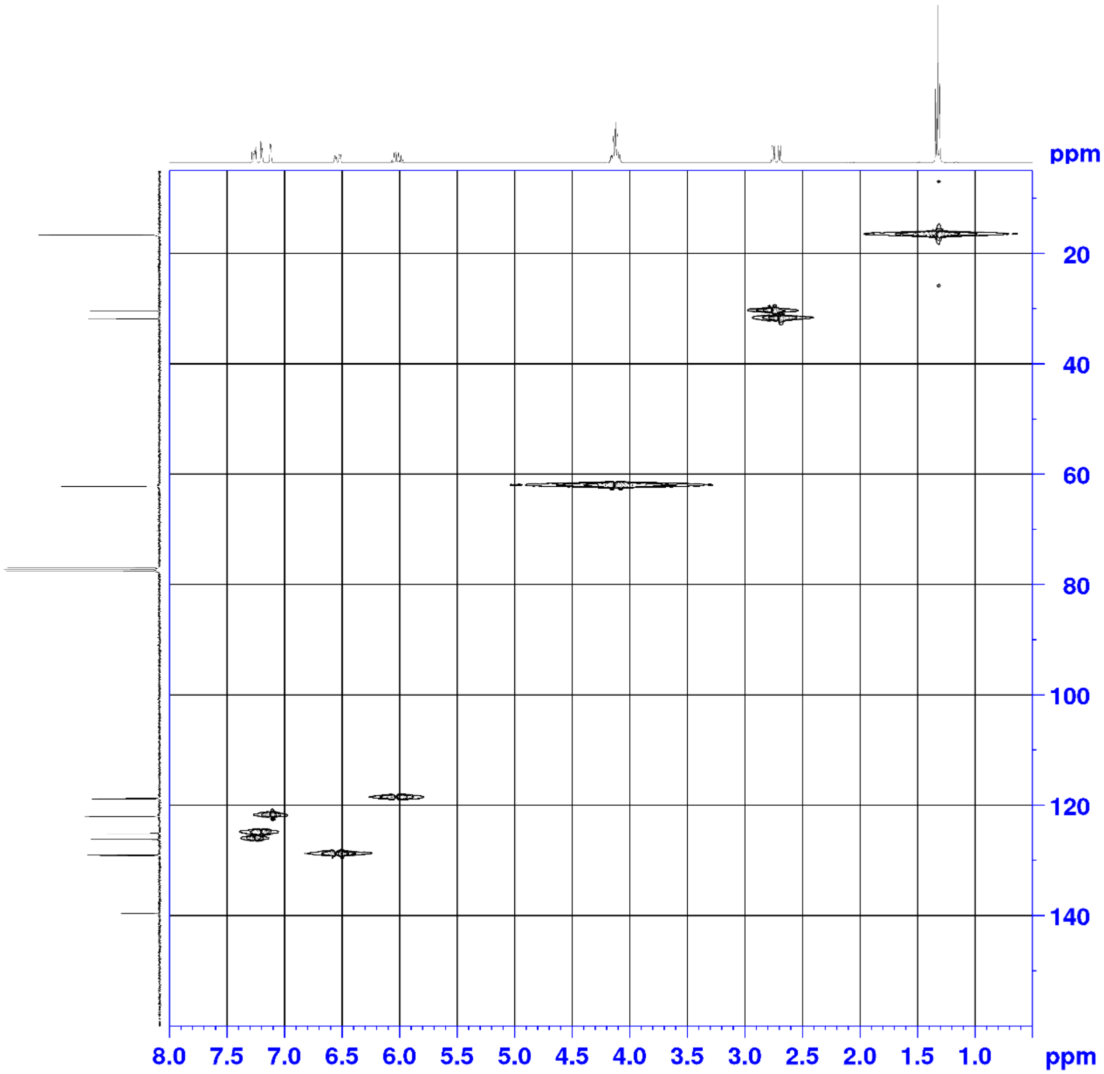
(*E*)-5o: COSY

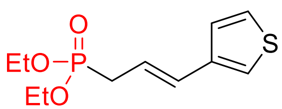

ppm
1
2
3
4
5
6
7
8
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm


O
EtO
P
EtO
S

(*E*)-**5o**

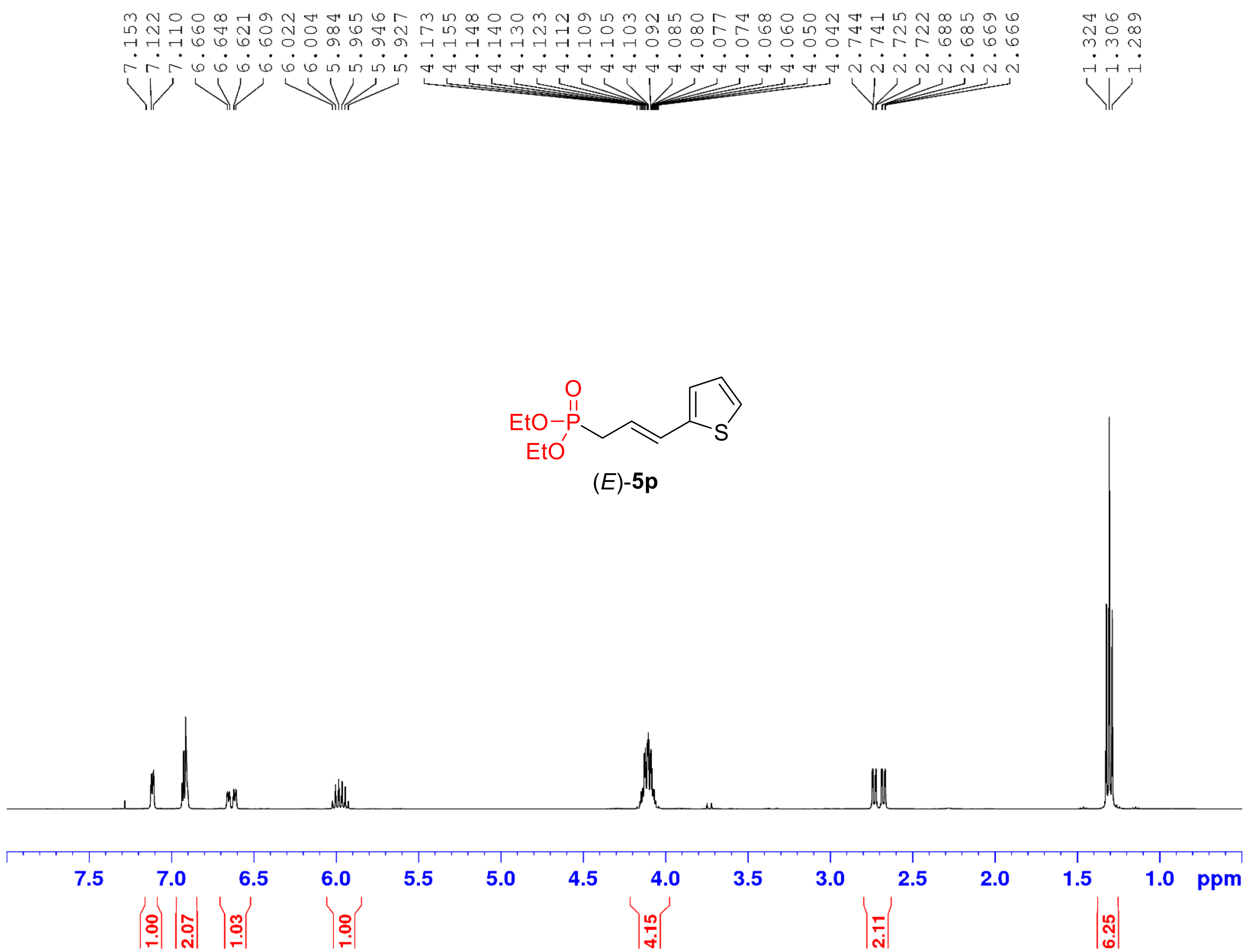
(*E*)-5o: ^{13}C HSQC





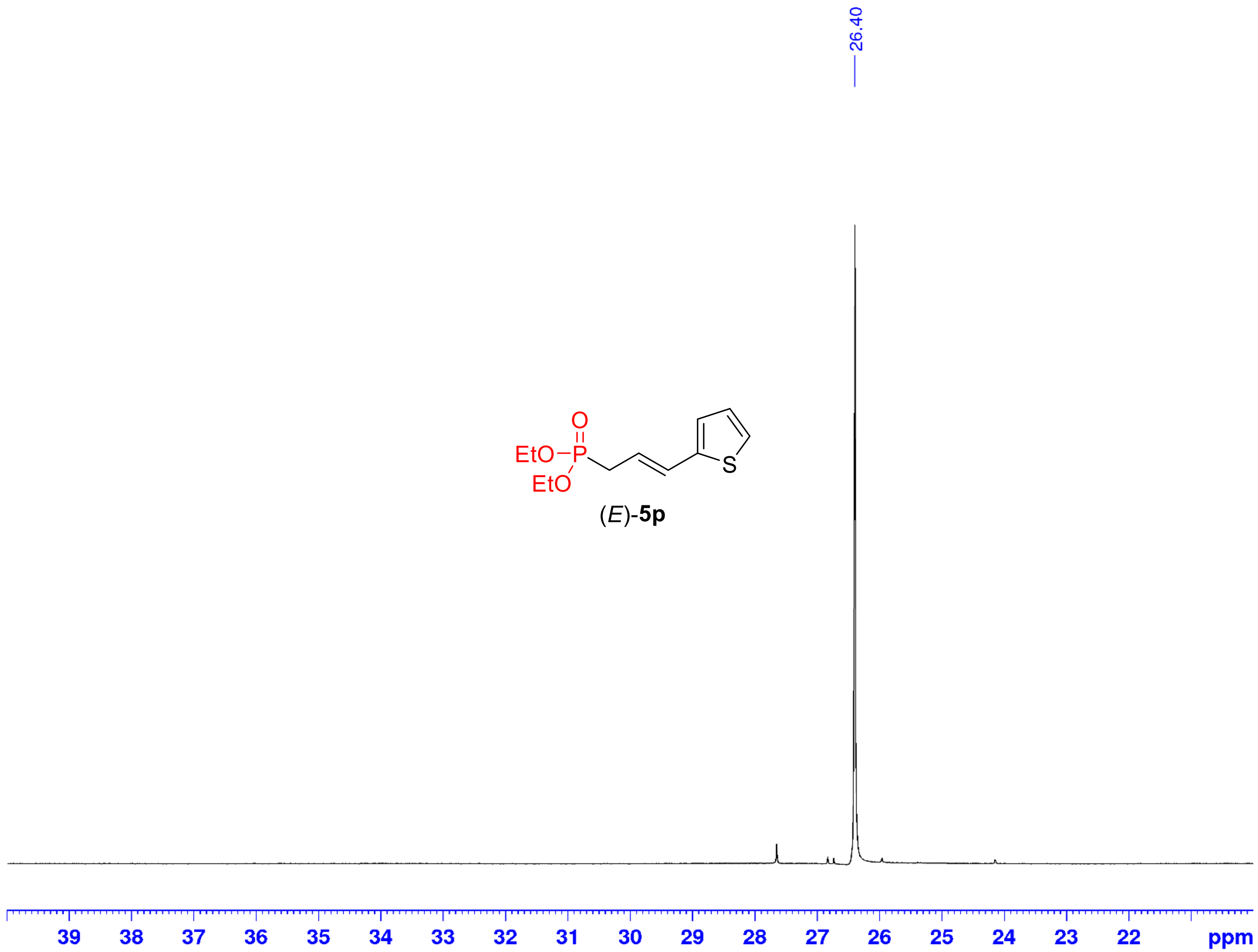
(*E*)-**5o**

(*E*)-5p: ^{1}H NMR

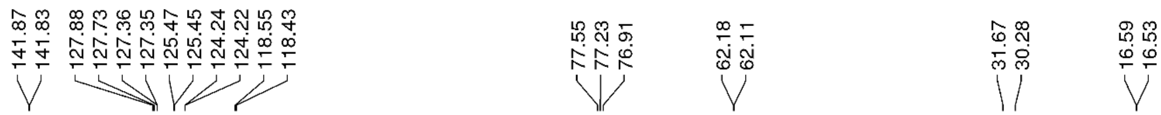


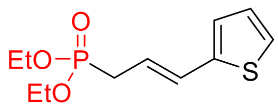
7.153
7.122
7.110
6.660
6.648
6.621
6.609
6.022
6.004
5.984
5.965
5.946
5.927
4.173
4.155
4.148
4.140
4.130
4.123
4.112
4.109
4.105
4.103
4.092
4.085
4.080
4.077
4.074
4.068
4.060
4.050
4.042
2.744
2.741
2.725
2.722
2.688
2.685
2.669
2.666
1.324
1.306
1.289
O
EtO
P
EtO
S
(E)-5p
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
1.00
2.07
1.03
1.00
4.15
2.11
6.25

(*E*)-5p: ^{31}P NMR

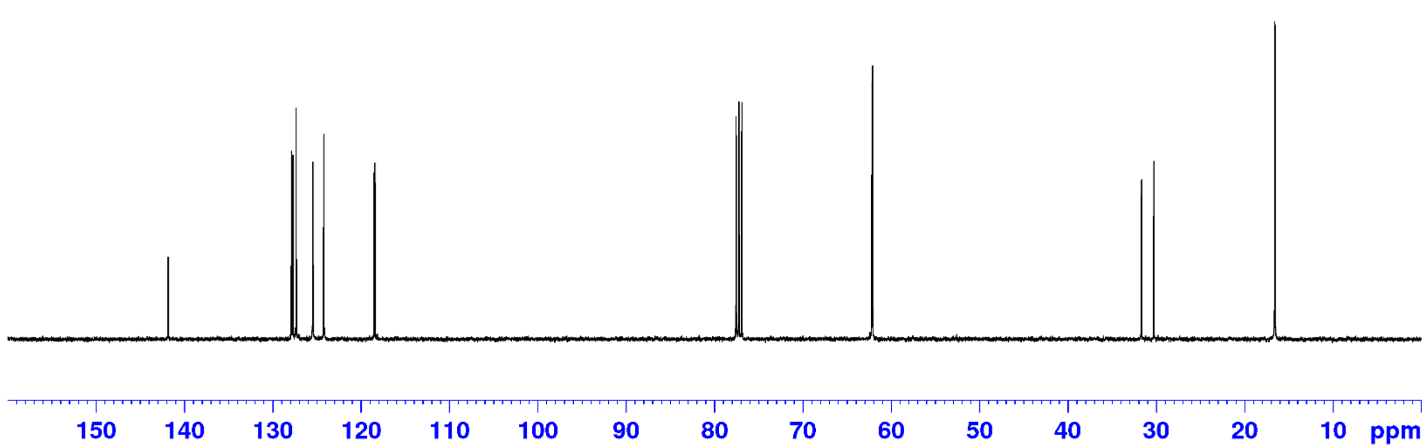

26.40
EtO
EtO
O
P
S
(E)-5p
39
38
37
36
35
34
33
32
31
30
29
28
27
26
25
24
23
22
ppm

(*E*)-5p: ^{13}C NMR

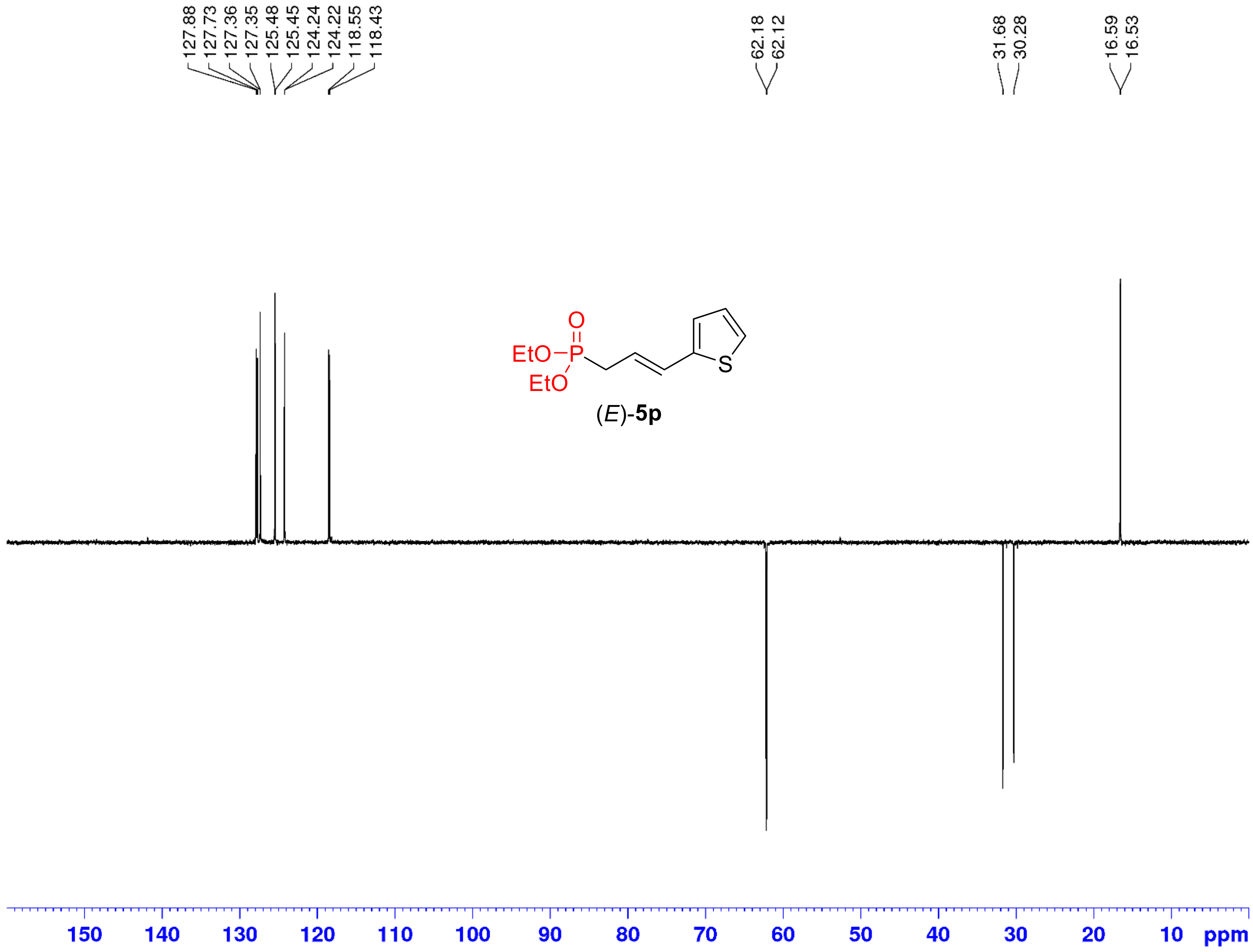




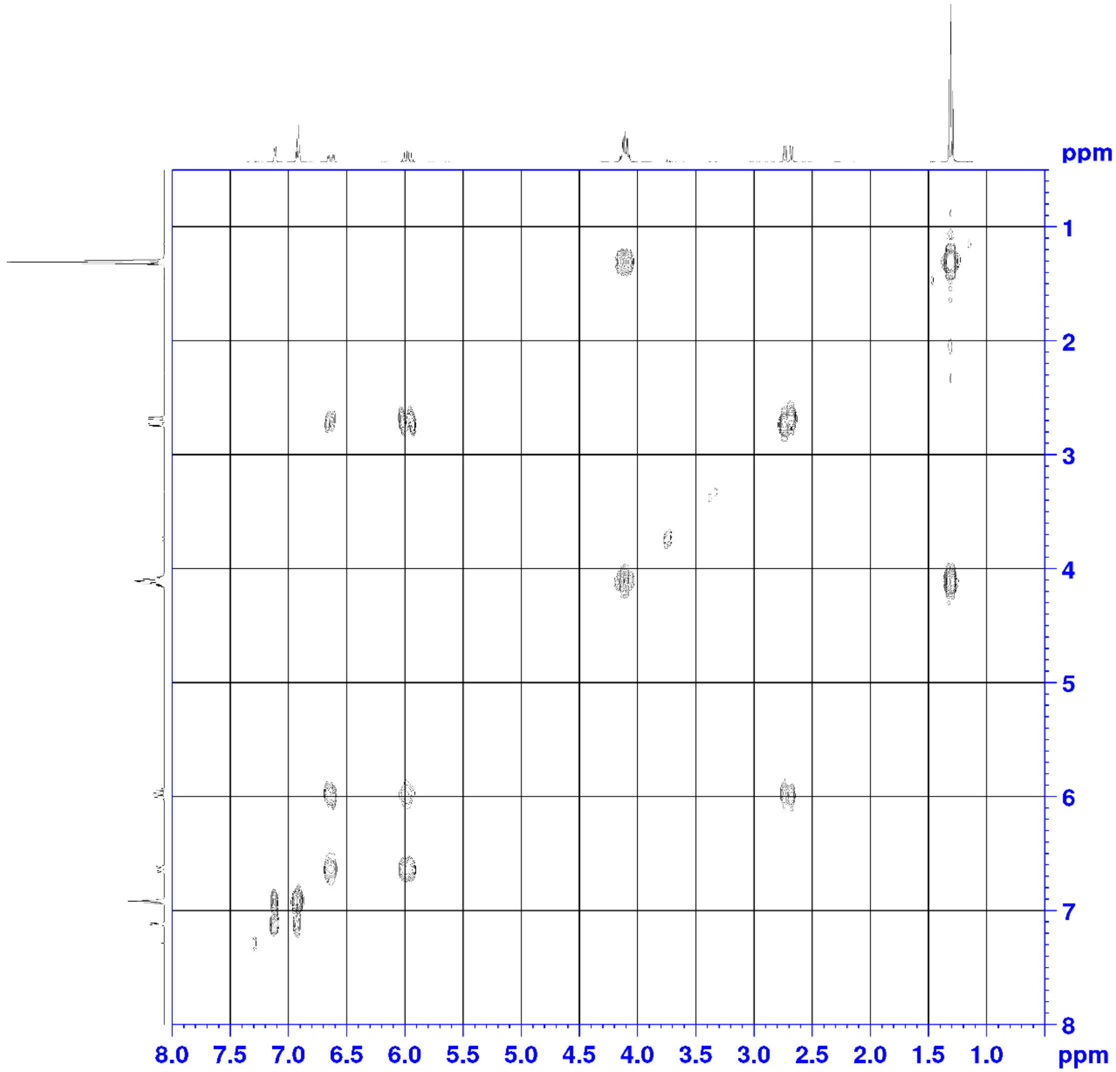
(*E*)-**5p**

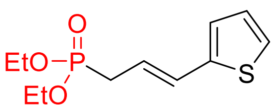


(E)-5p: ^{13}C DEPT135



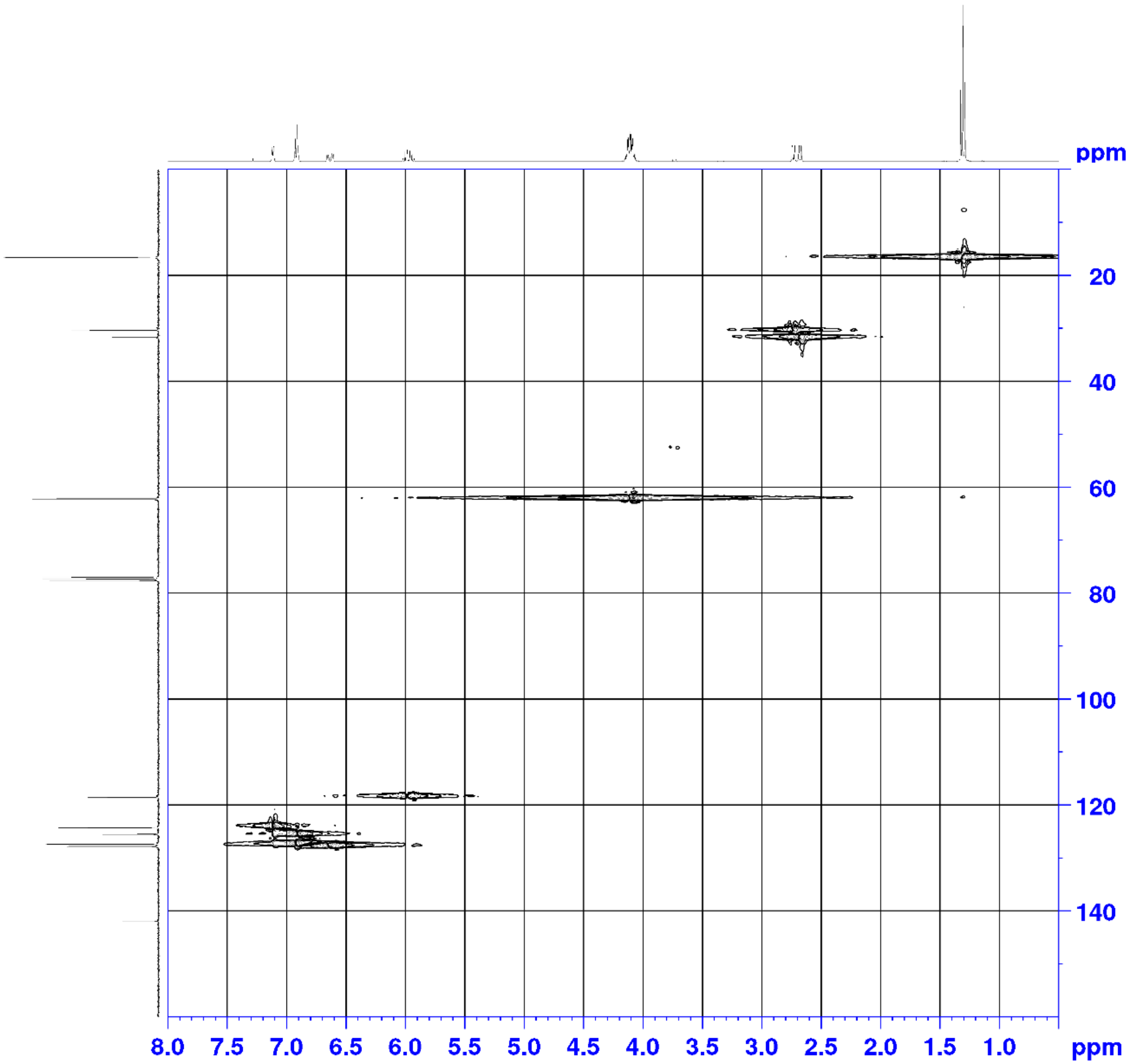
(*E*)-5p: COSY

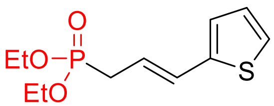

ppm
1
2
3
4
5
6
7
8
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0
ppm


O
EtO
P
EtO
S

(*E*)-**5p**

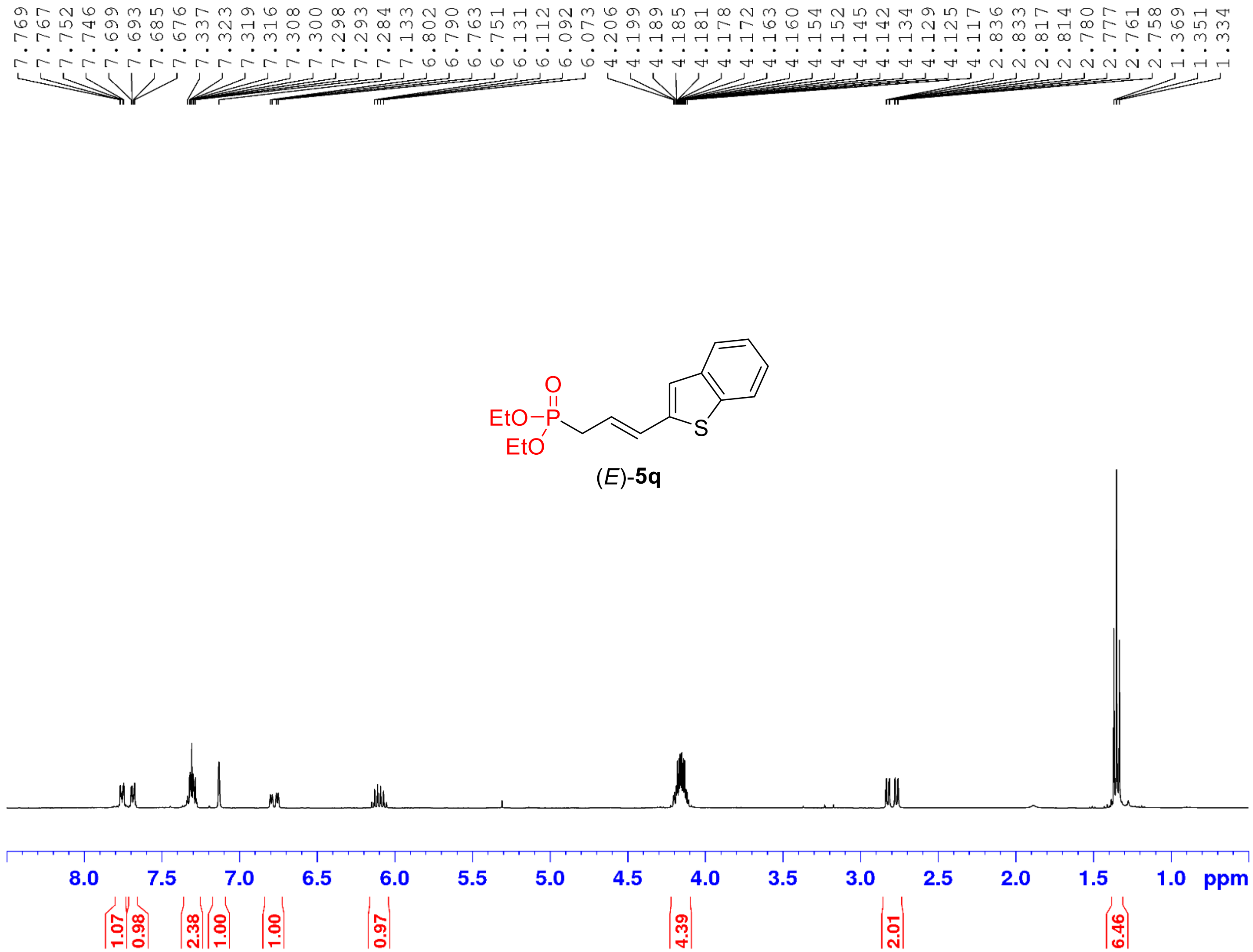
(*E*)-5p: ^{13}C HSQC





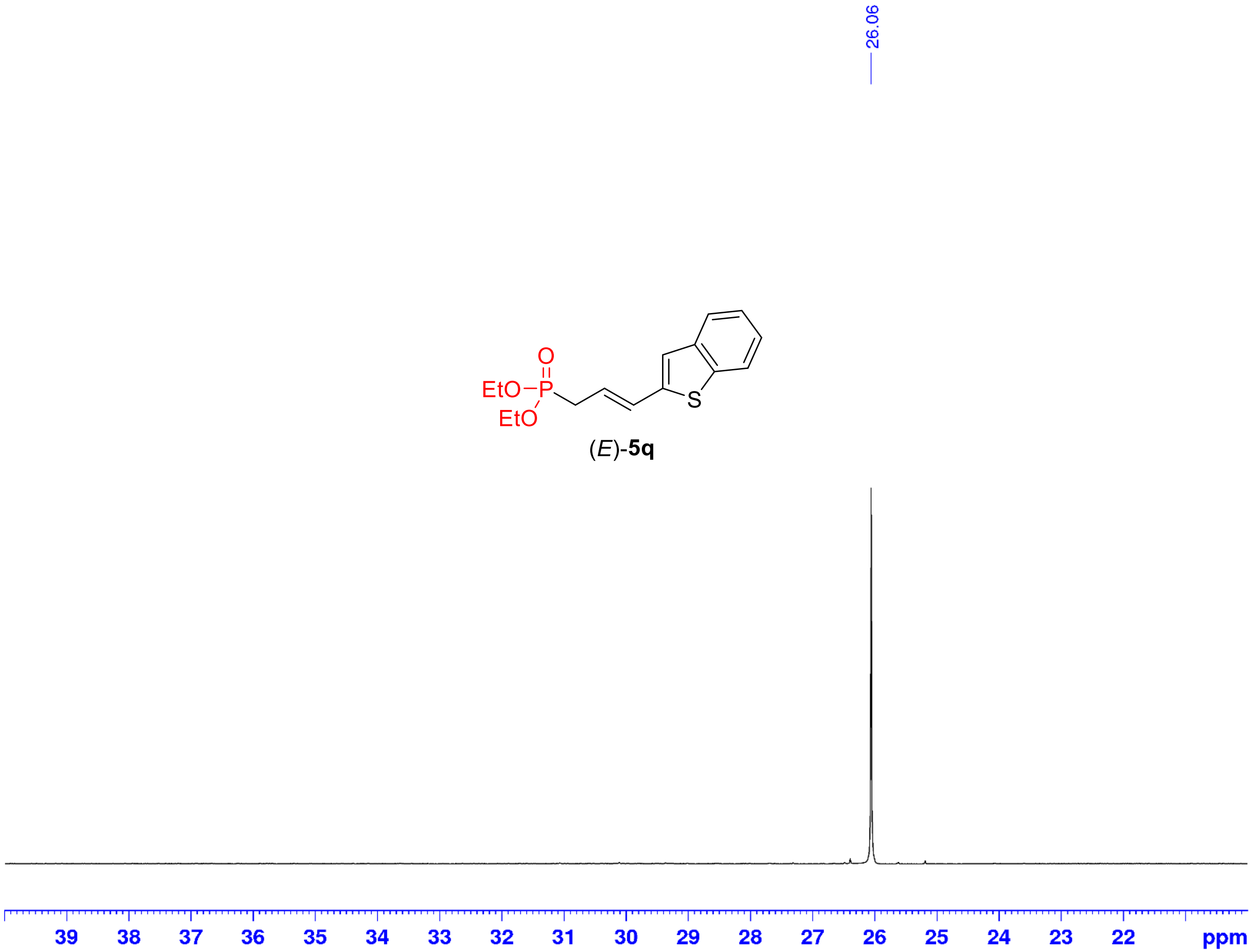
(*E*)-**5p**

(*E*)-5q: ^{1}H NMR

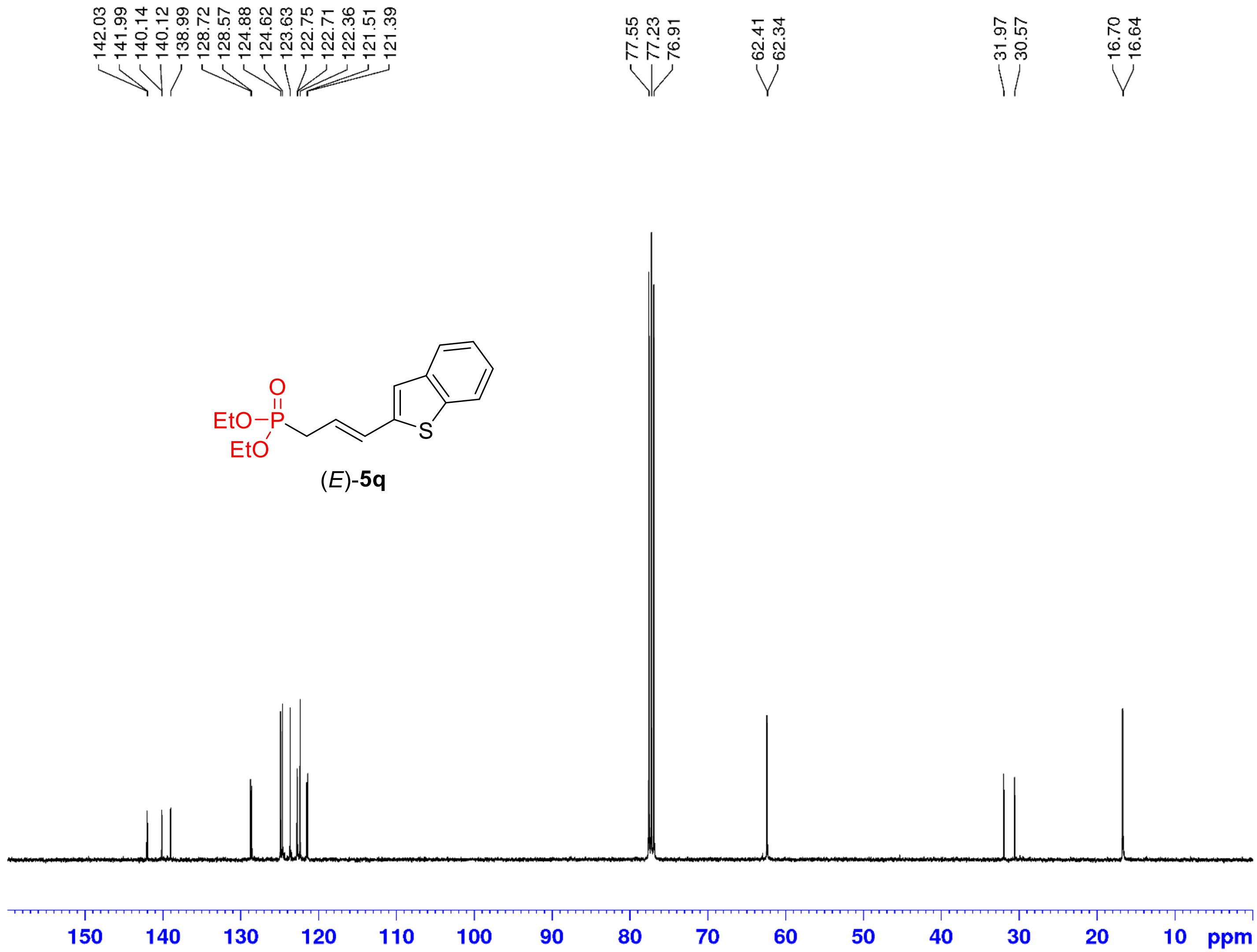


7.769
7.767
7.752
7.746
7.699
7.693
7.685
7.676
7.337
7.323
7.319
7.316
7.308
7.300
7.298
7.293
7.284
7.133
6.802
6.790
6.763
6.751
6.131
6.112
6.092
6.073
4.206
4.199
4.189
4.185
4.181
4.178
4.172
4.163
4.160
4.154
4.152
4.145
4.142
4.134
4.129
4.125
4.117
2.836
2.833
2.817
2.814
2.780
2.777
2.761
2.758
1.369
1.351
1.334
O
EtO
P
EtO
S
(*E*)-**5q**
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
ppm
1.07
0.98
2.38
1.00
1.00
0.97
4.39
2.01
6.46

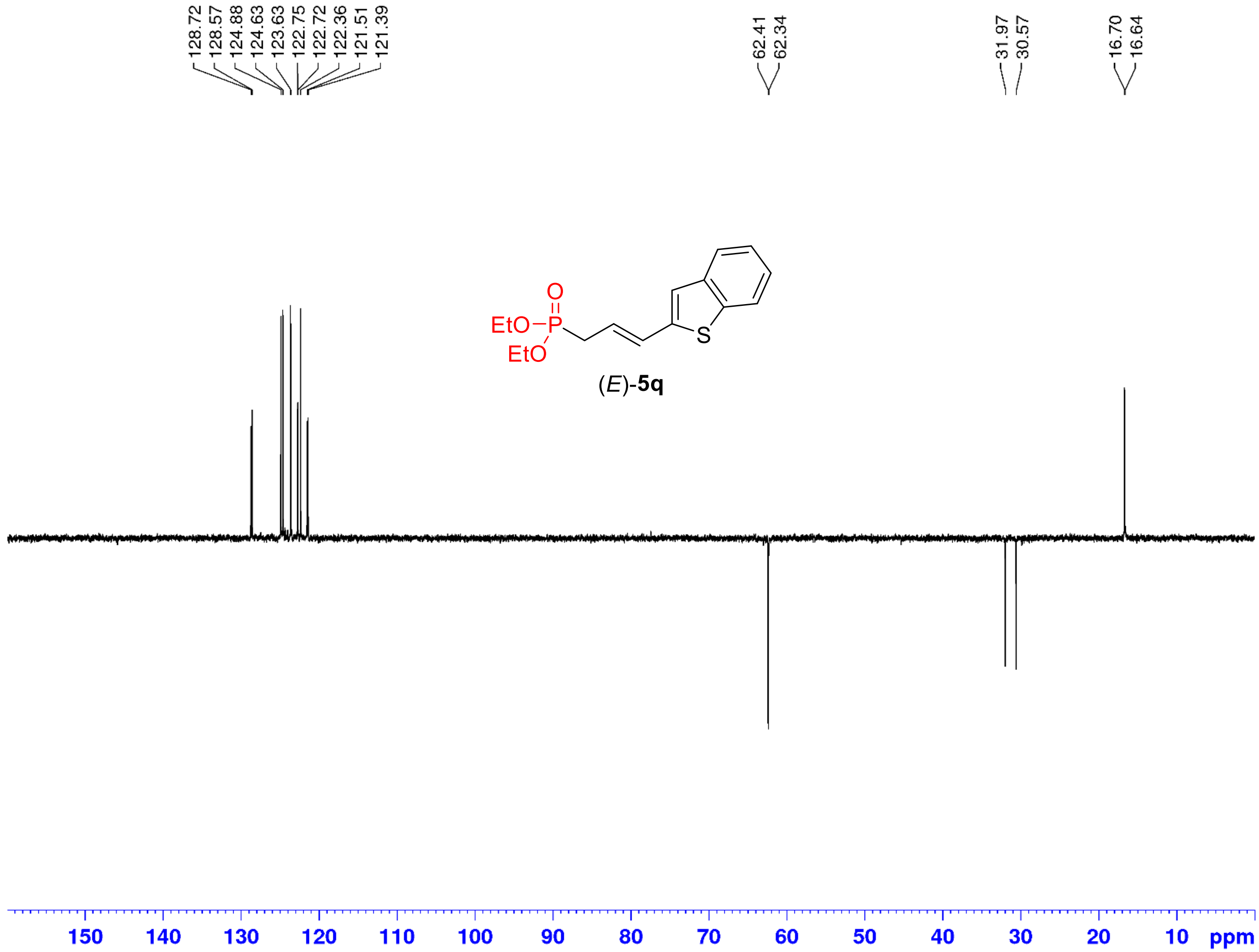
(*E*)-5q: ^{31}P NMR



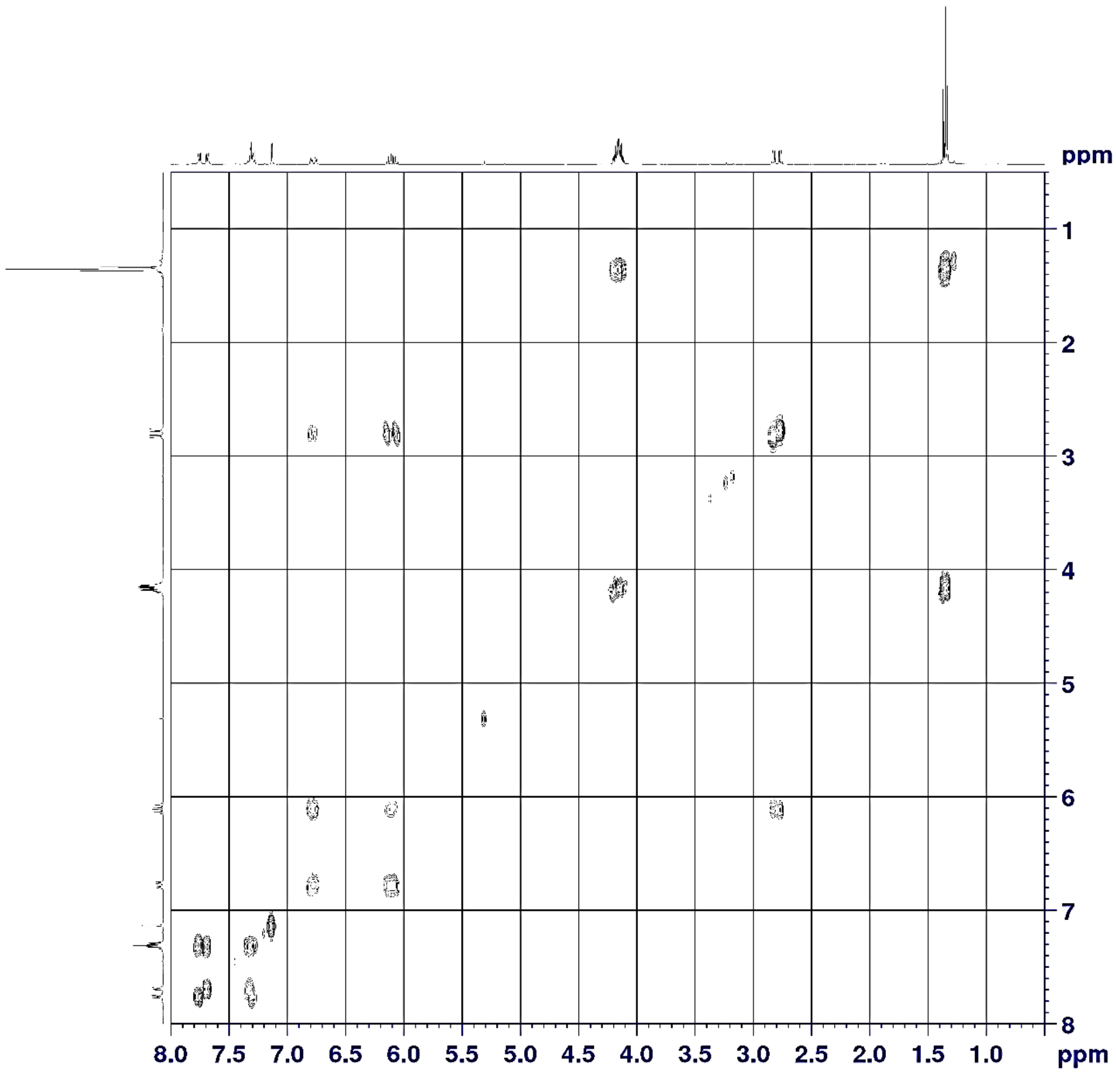
(*E*)-5q: ^{13}C NMR

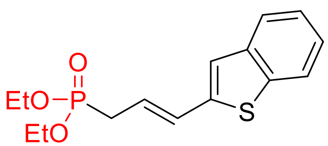

142.03
141.99
140.14
140.12
138.99
128.72
128.57
124.88
124.62
123.63
122.75
122.71
122.36
121.51
121.39
77.55
77.23
76.91
62.41
62.34
31.97
30.57
16.70
16.64
O
EtO
P
EtO
S
(E)-5q
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

(*E*)-5q: ^{13}C DEPT135



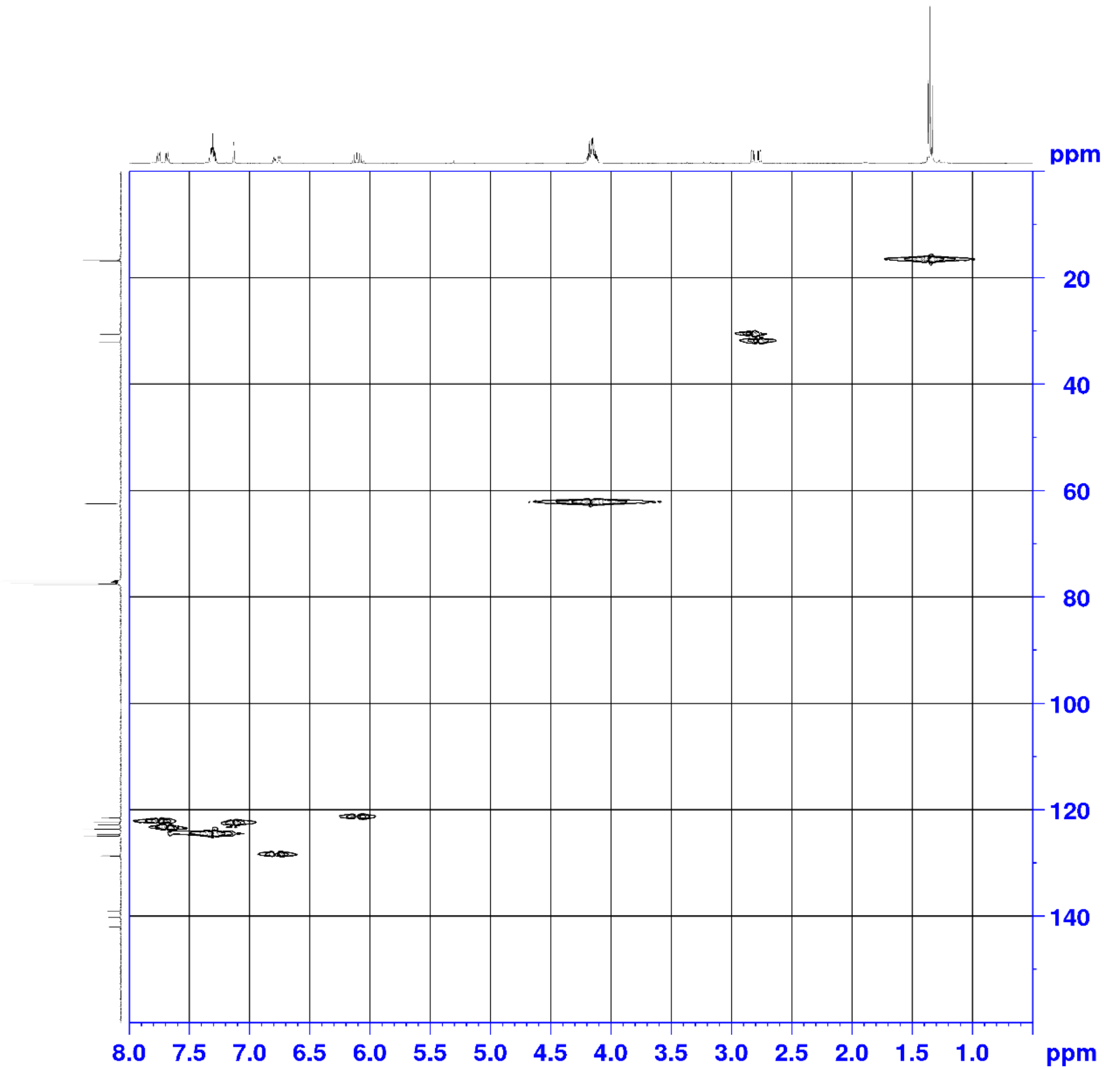
(*E*)-5q: COSY

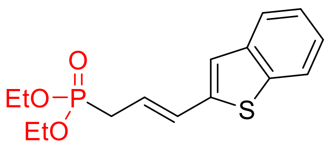

ppm
1
2
3
4
5
6
7
8
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm


O
EtO
P
EtO
S

(*E*)-**5q**

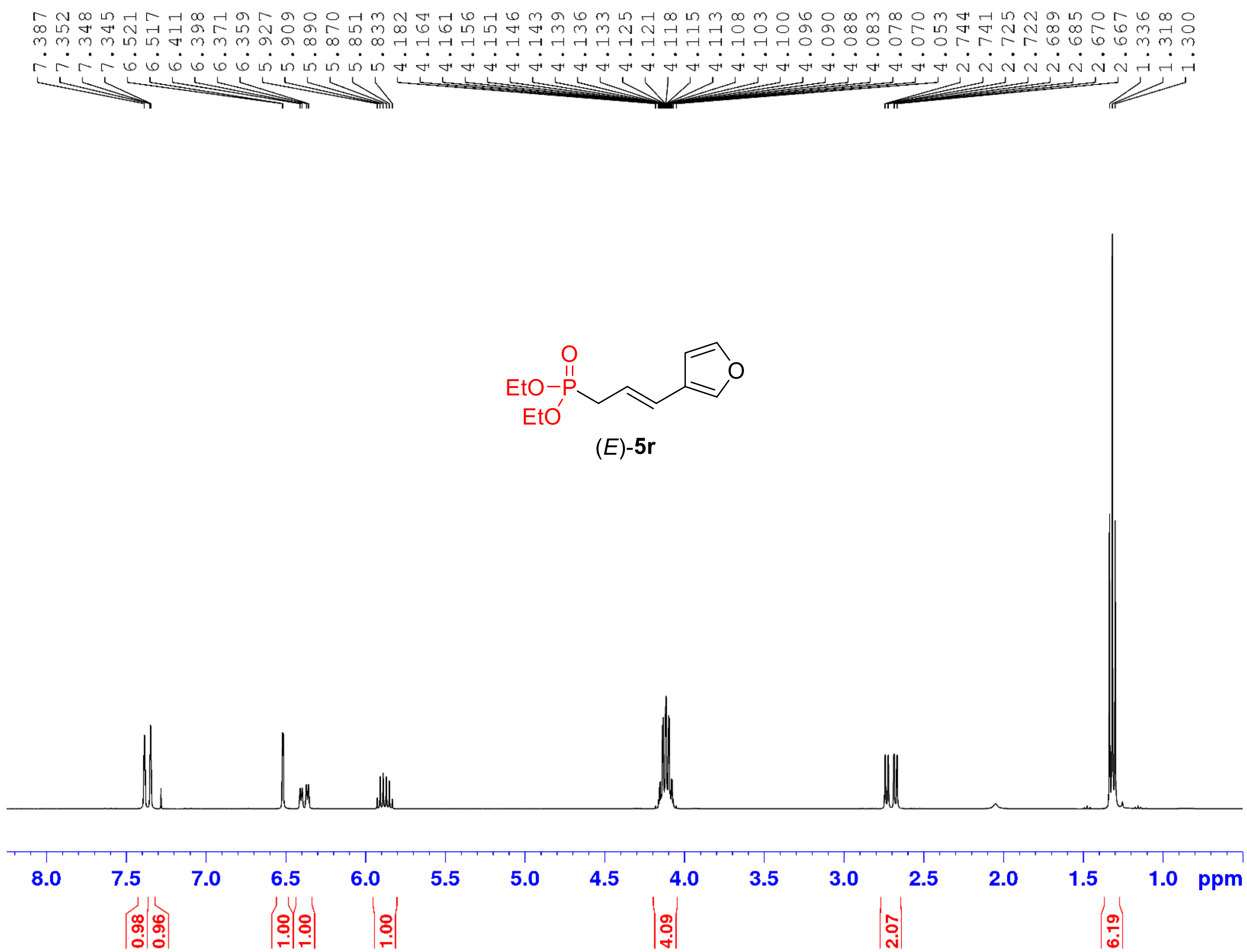
(*E*)-5q: ^{13}C HSQC





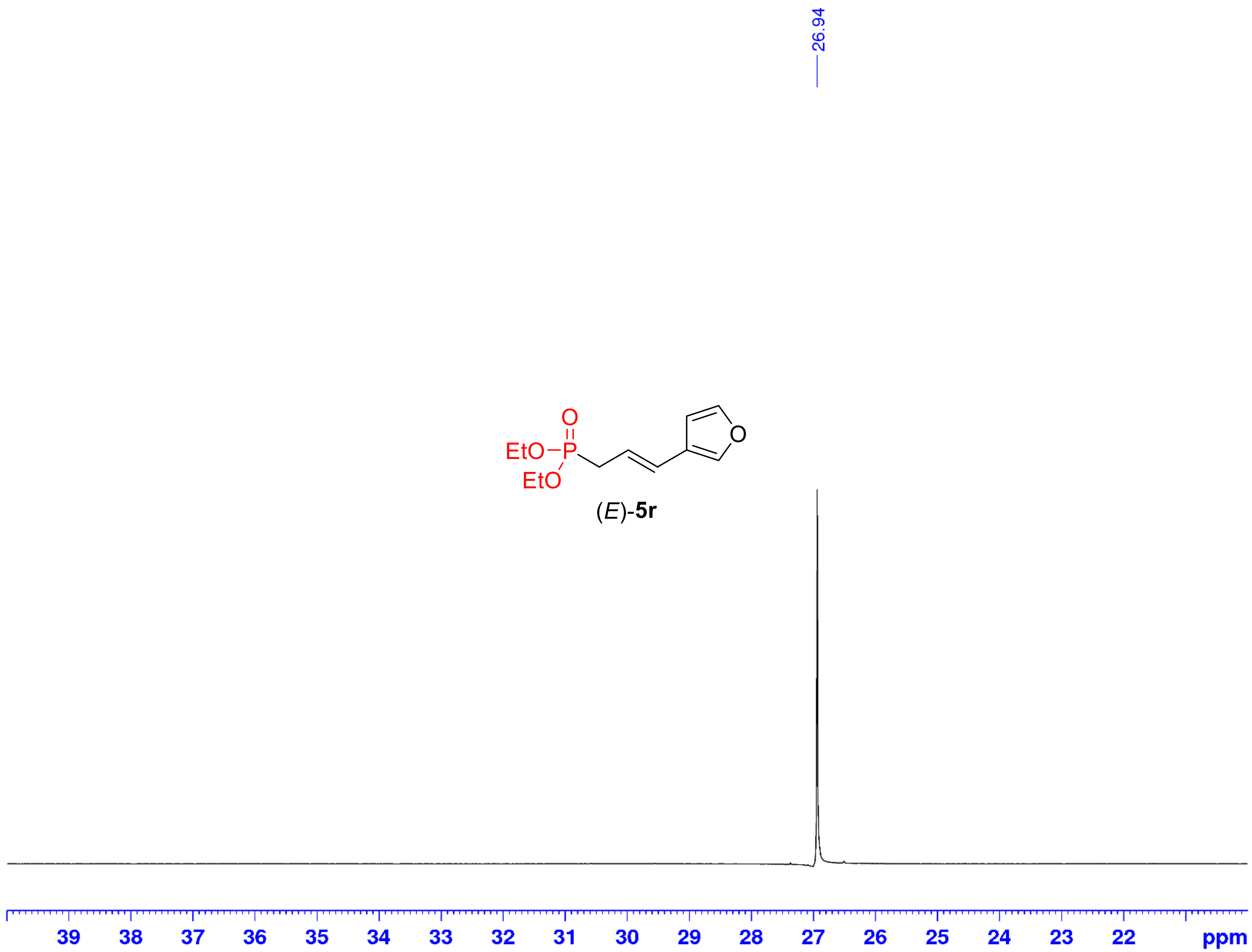
(*E*)-**5q**

(*E*)-5r: ^{1}H NMR

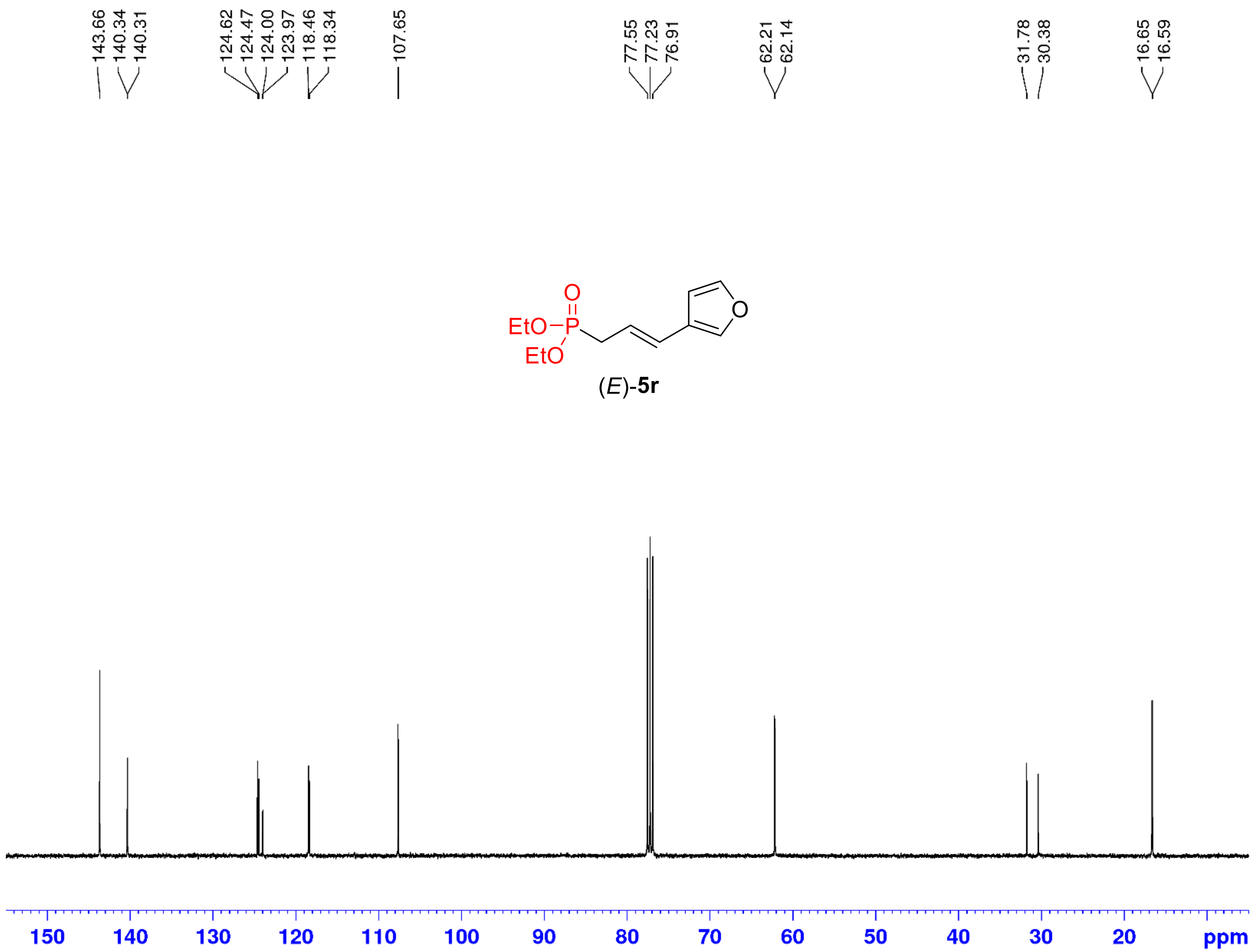


7.387
7.352
7.348
7.345
6.521
6.517
6.411
6.398
6.371
6.359
5.927
5.909
5.890
5.870
5.851
5.833
4.182
4.164
4.161
4.156
4.151
4.146
4.143
4.139
4.136
4.133
4.125
4.121
4.118
4.115
4.113
4.108
4.103
4.100
4.096
4.090
4.088
4.083
4.078
4.070
4.053
2.744
2.741
2.725
2.722
2.689
2.685
2.670
2.667
1.336
1.318
1.300
O
EtO
P
EtO
O
(E)-5r
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
ppm
0.98
0.96
1.00
1.00
1.00
4.09
2.07
6.19

(*E*)-5r: ^{31}P NMR

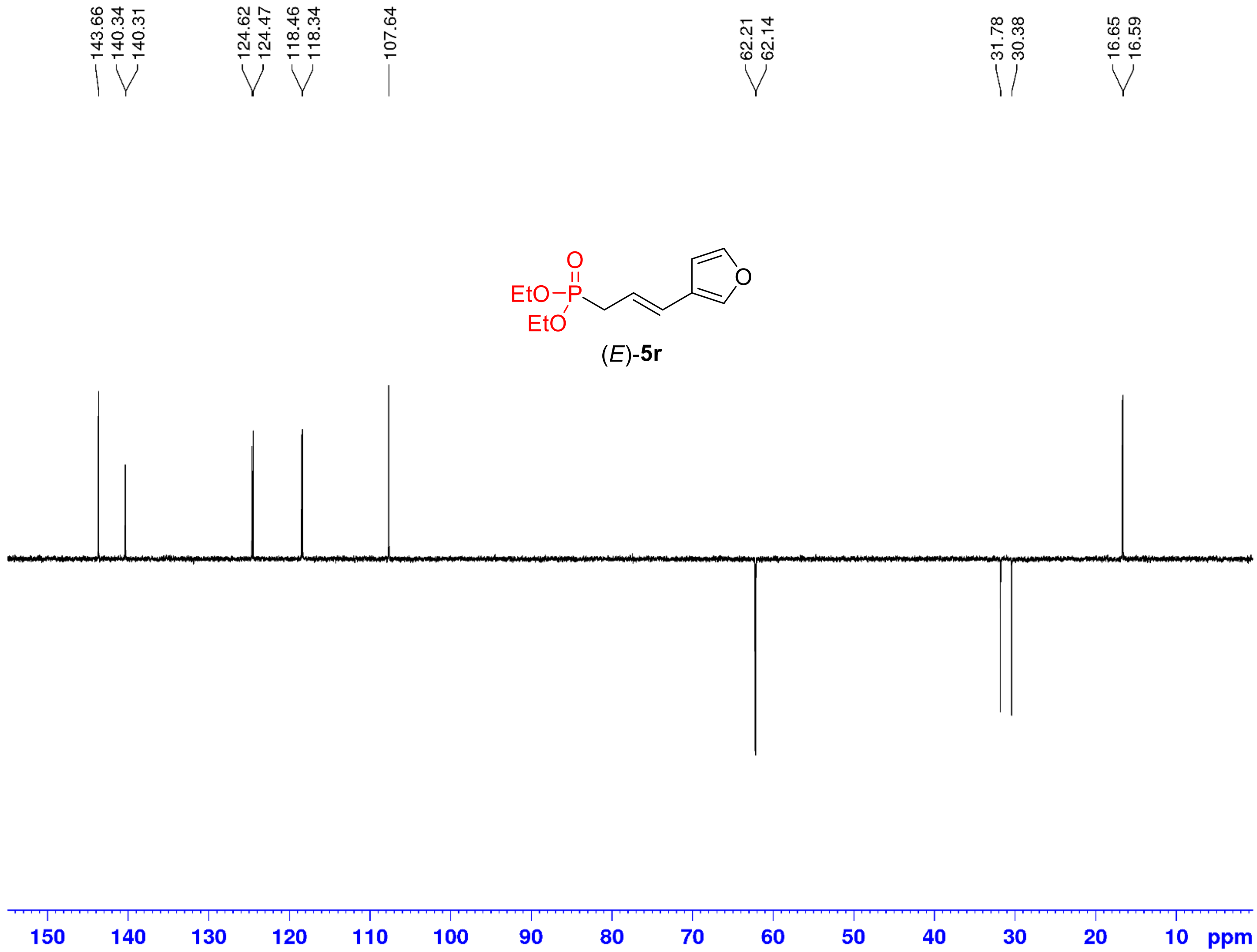

26.94
EtO
EtO
O
P
O
(E)-5r
39
38
37
36
35
34
33
32
31
30
29
28
27
26
25
24
23
22
ppm

(*E*)-5r: ^{13}C NMR

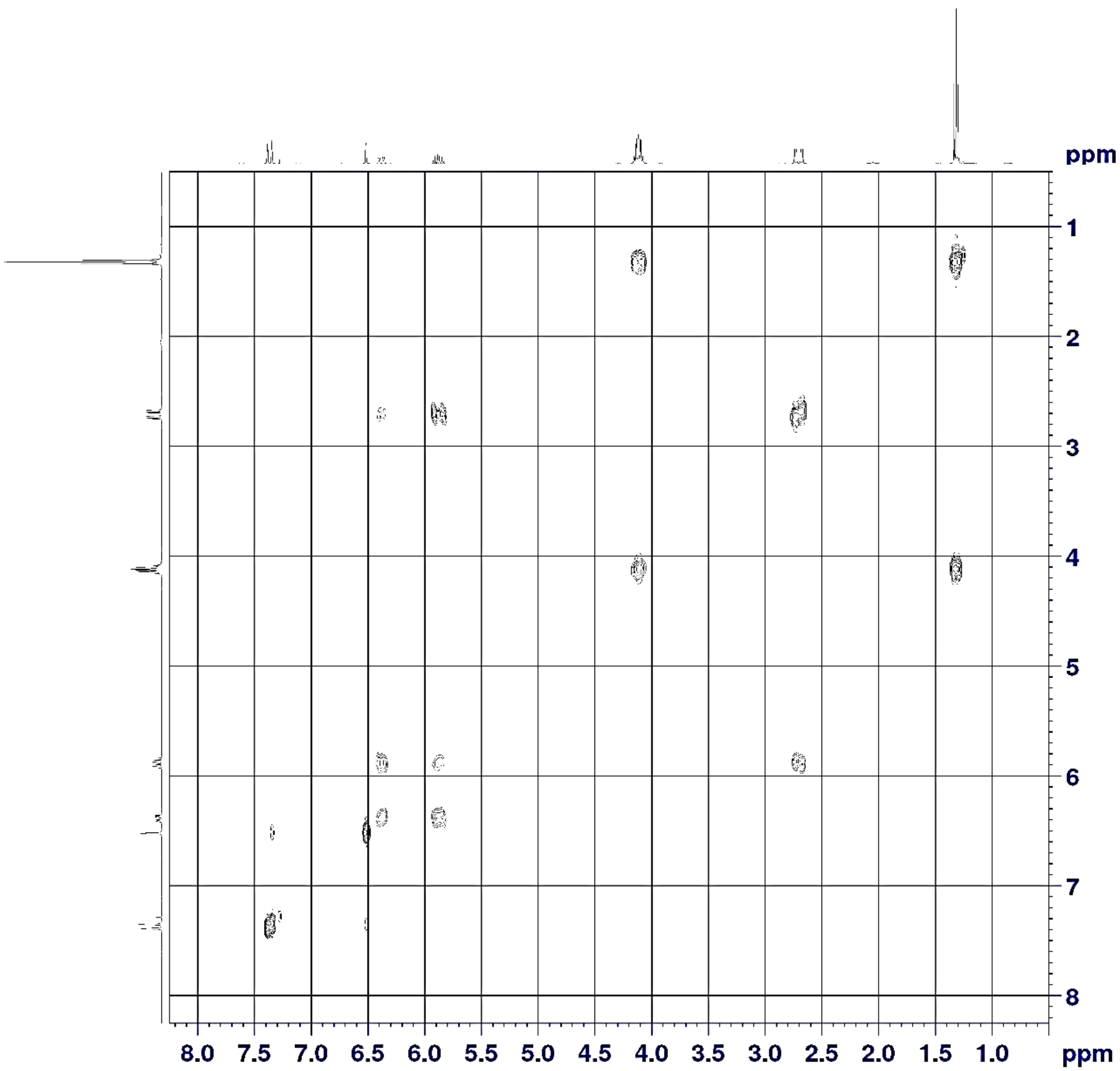


143.66
140.34
140.31
124.62
124.47
124.00
123.97
118.46
118.34
107.65
77.55
77.23
76.91
62.21
62.14
31.78
30.38
16.65
16.59
EtO
EtO
O
P
O
(*E*)-**5r**
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

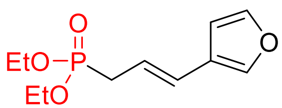
(E)-5r: ^{13}C DEPT135



(*E*)-5r: COSY



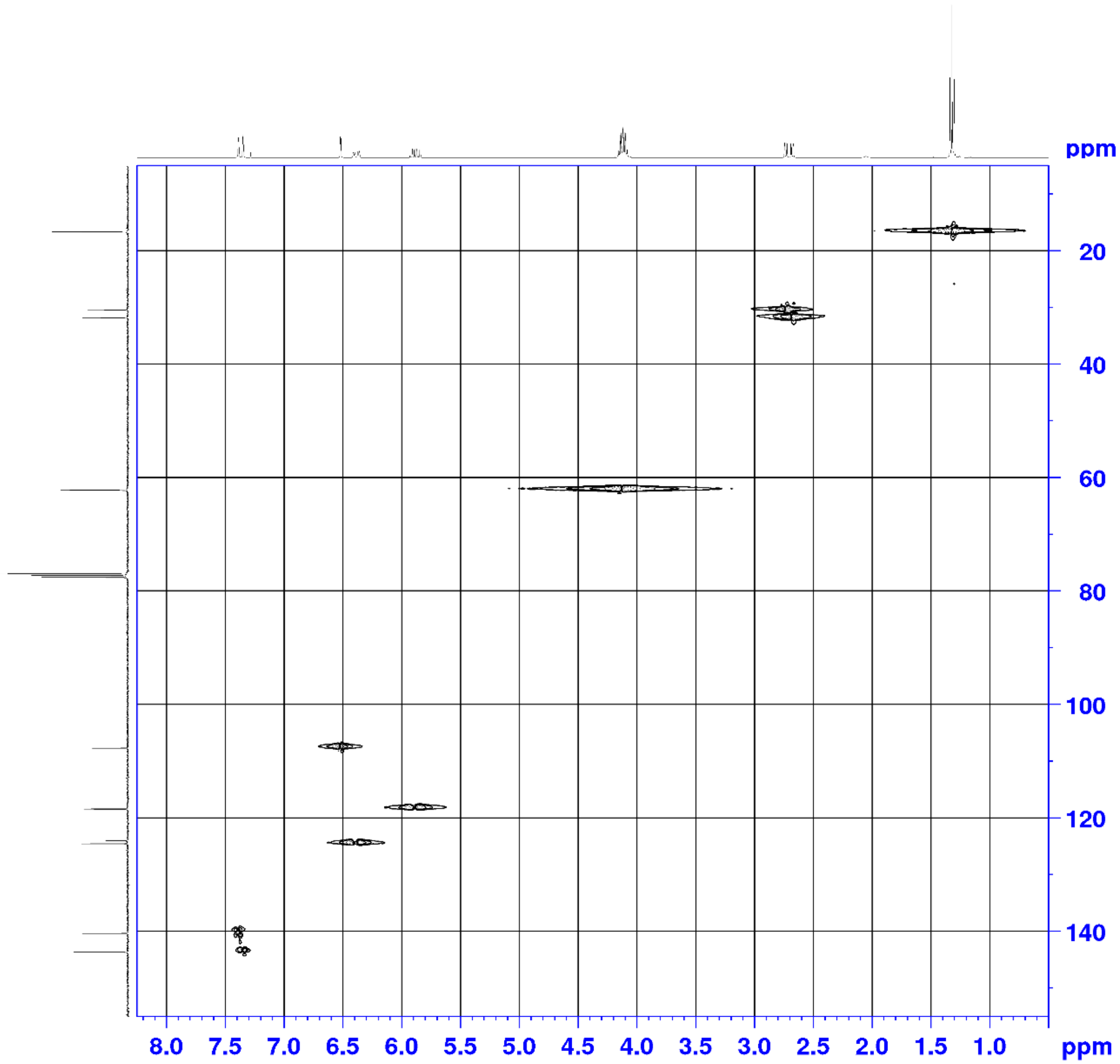
ppm
1
2
3
4
5
6
7
8
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

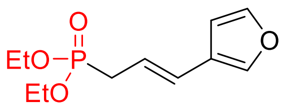


O
EtO
P
EtO
O

(*E*)-**5r**

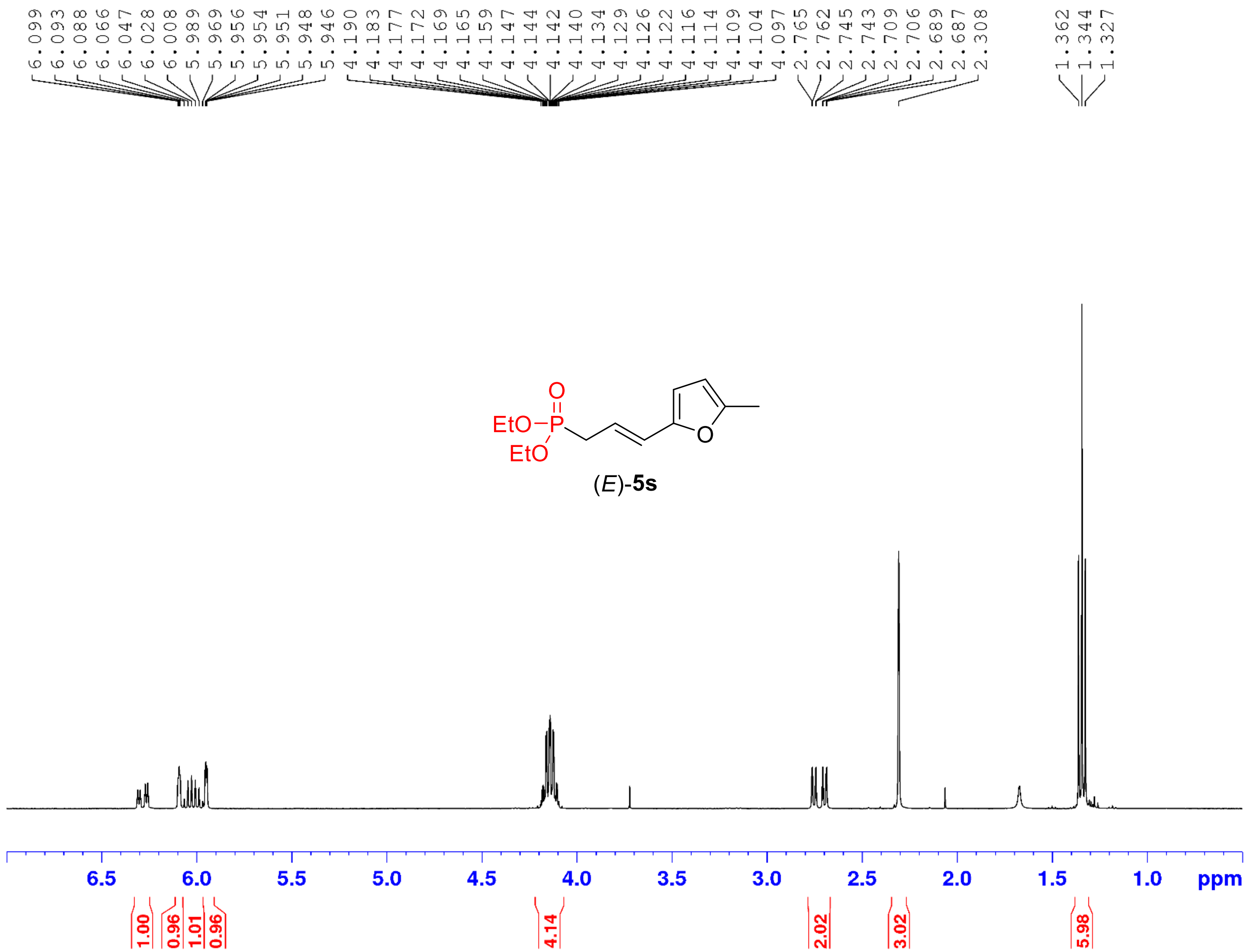
(*E*)-5r: ^{13}C HSQC



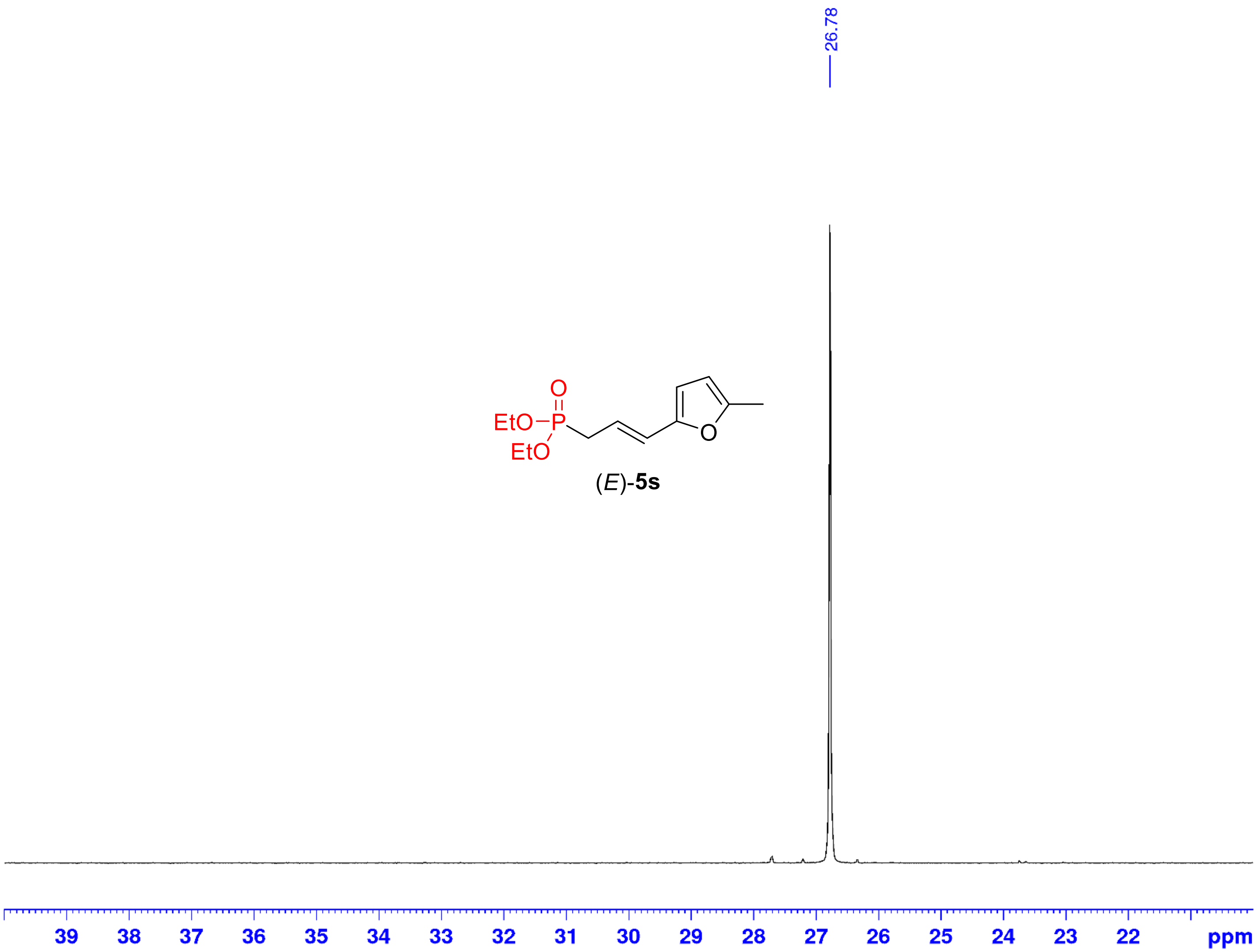


(*E*)-**5r**

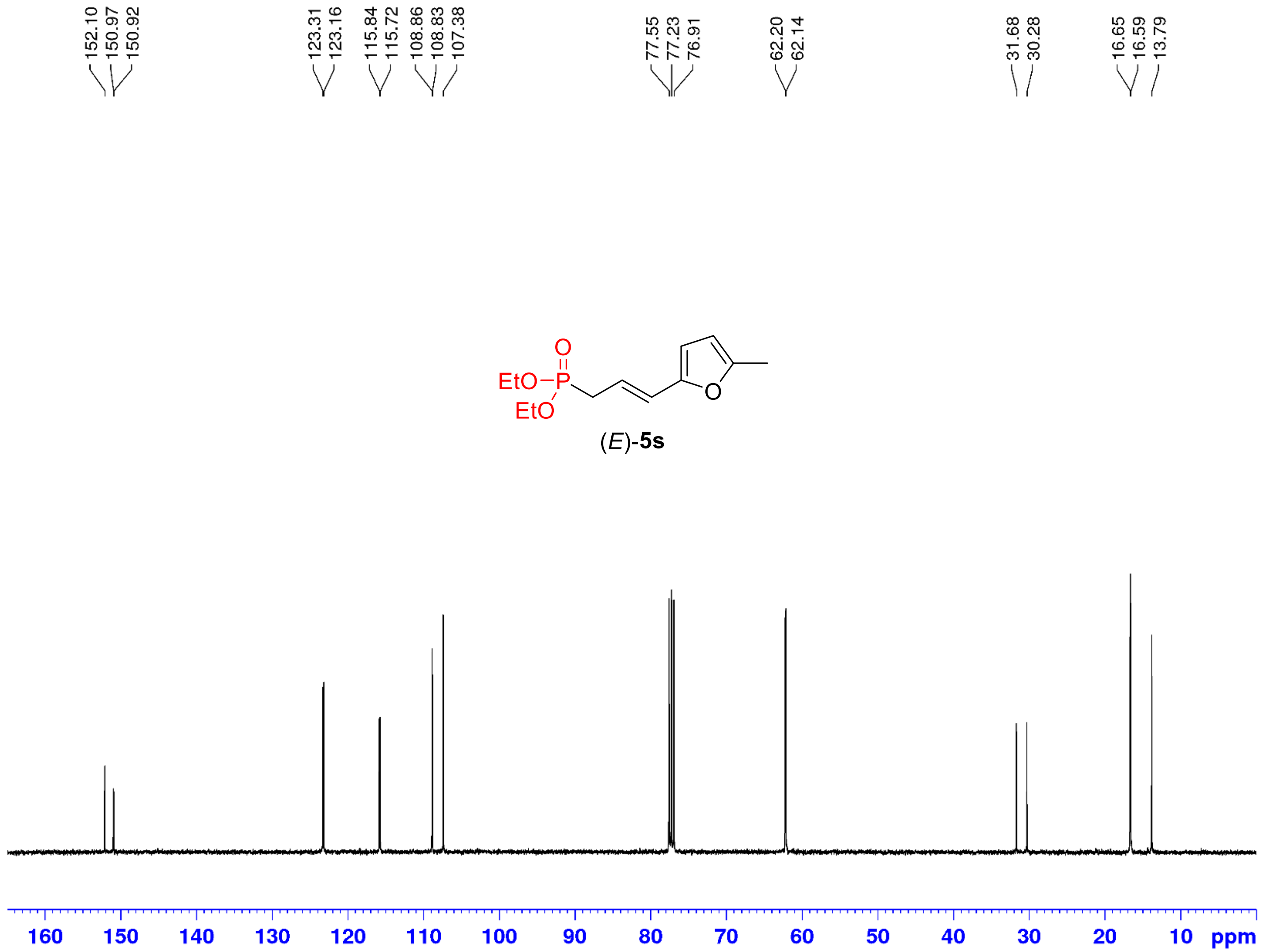
(*E*)-5s: ^{1}H NMR



(*E*)-5s: ^{31}P NMR

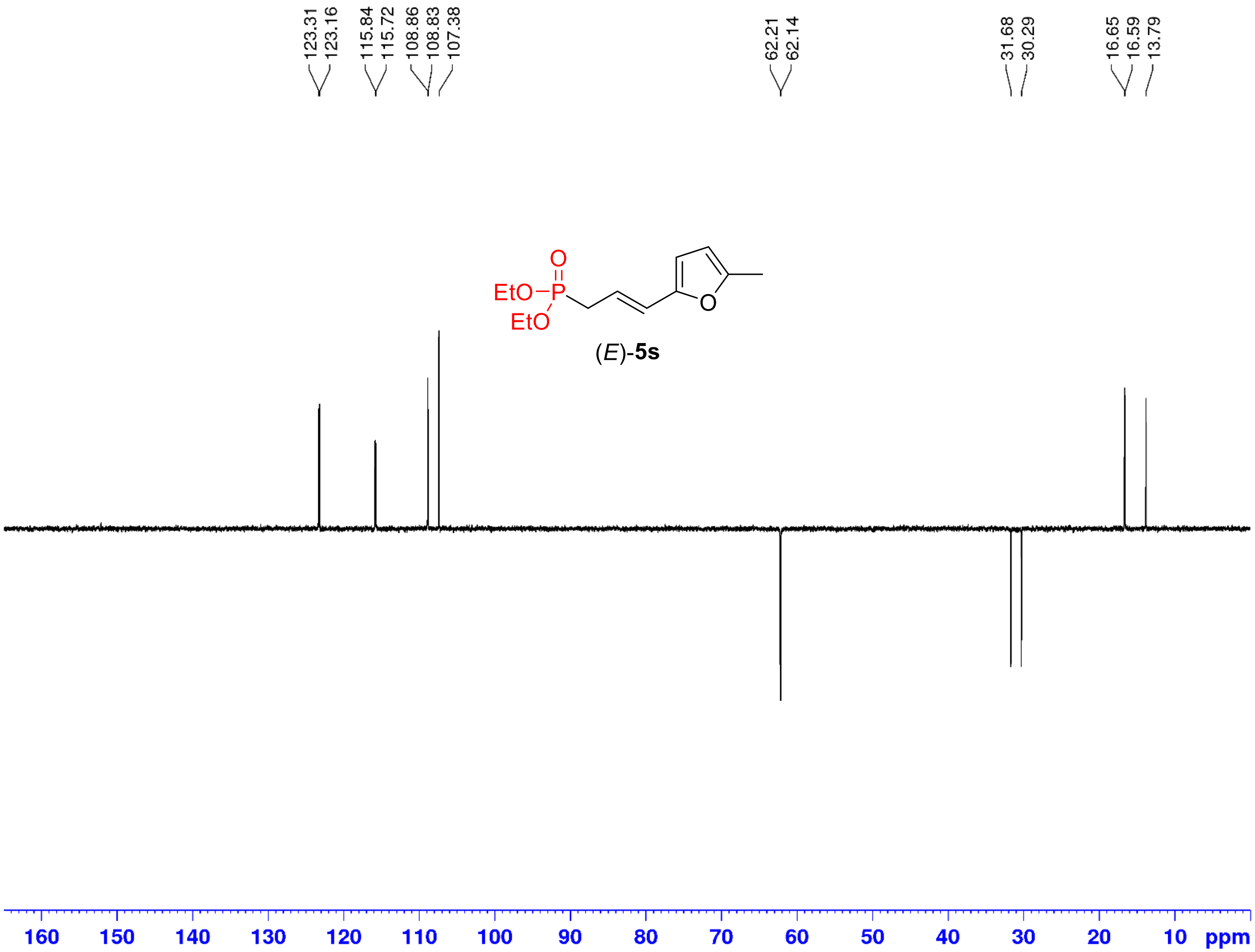

26.78
EtO
EtO
O
P
O
(E)-5s
39
38
37
36
35
34
33
32
31
30
29
28
27
26
25
24
23
22
ppm

(*E*)-5s: ^{13}C NMR

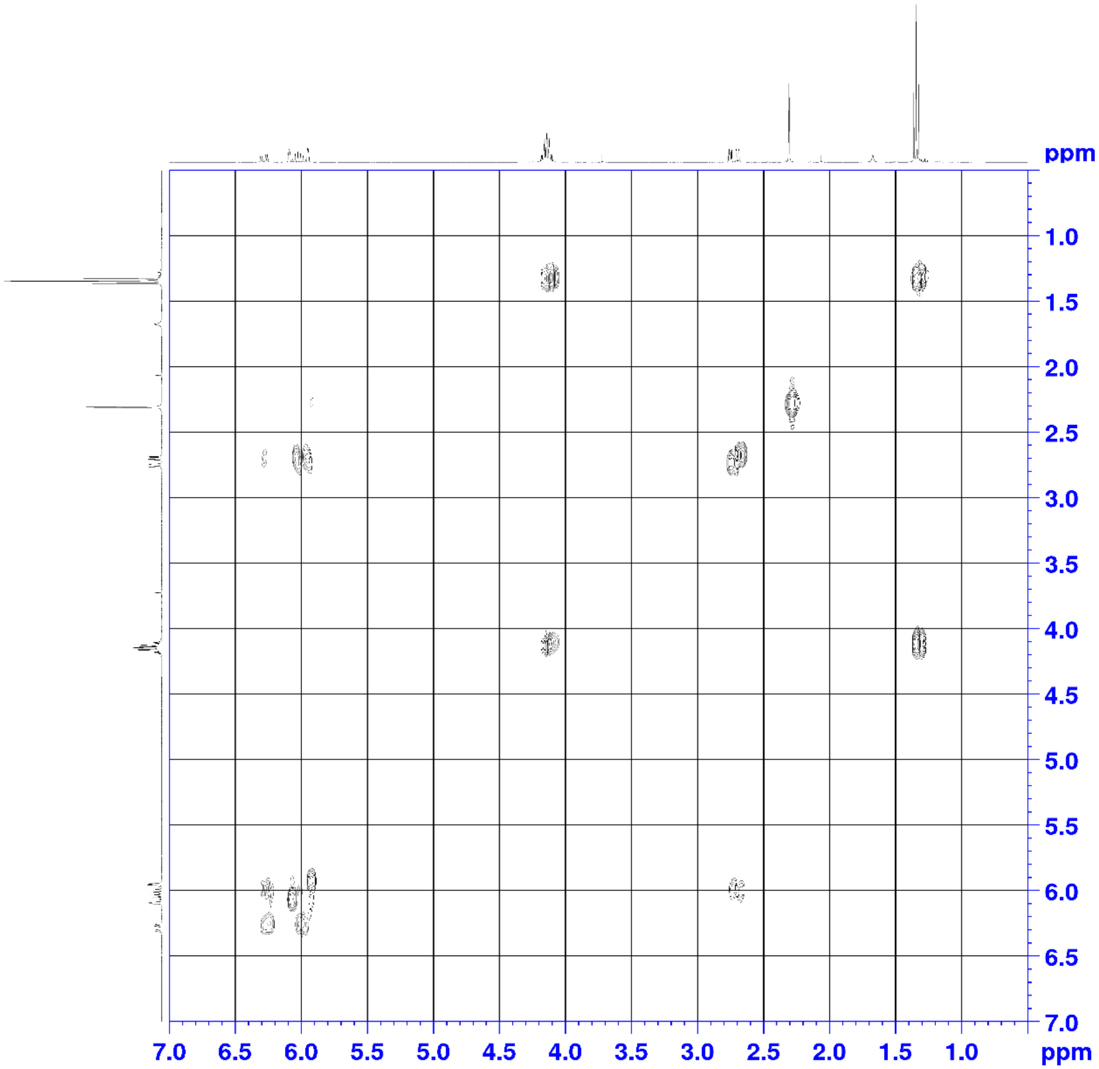


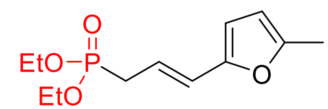
(*E*)-**5s**

(*E*)-5s: ^{13}C DEPT135



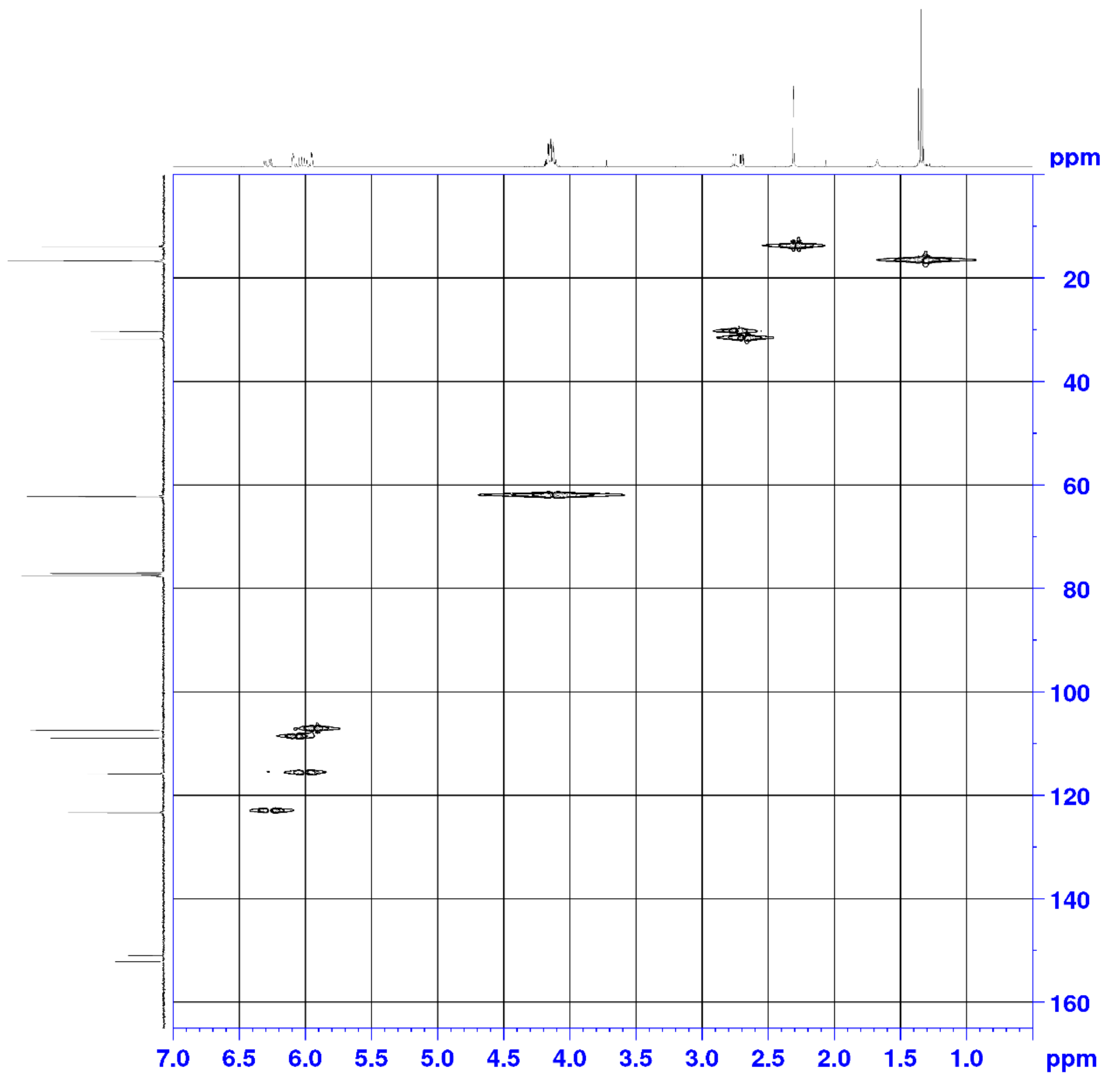
(*E*)-5s: COSY

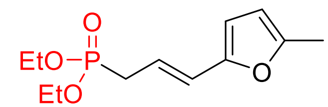

ppm
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0
ppm


O
EtO
P
EtO
O

(*E*)-**5s**

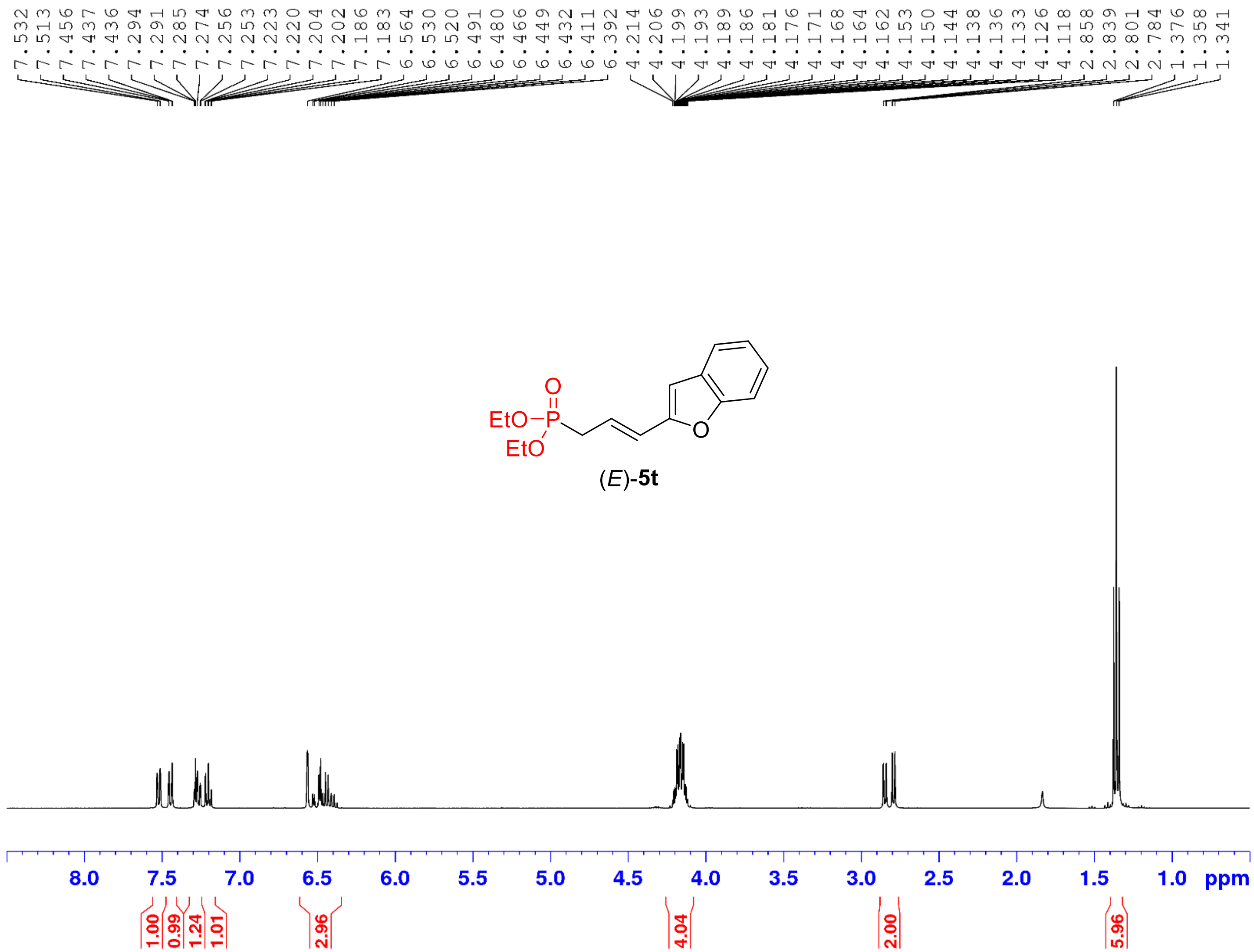
(*E*)-5s: ^{13}C-HSQC





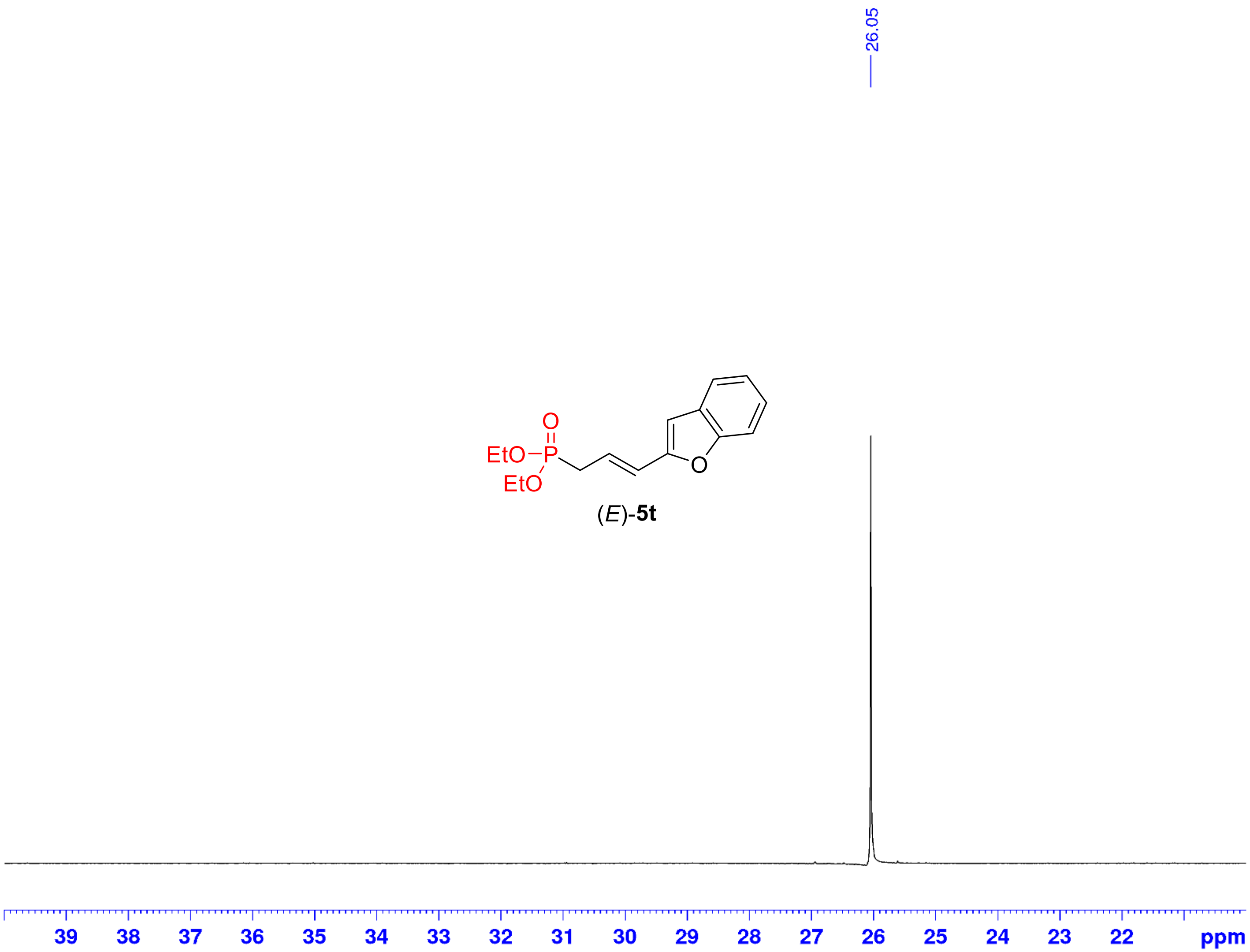
(*E*)-**5s**

(*E*)-5t: ^{1}H NMR



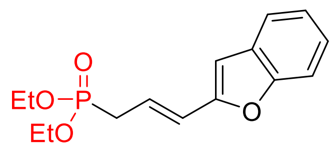
7.532
7.513
7.456
7.437
7.436
7.294
7.291
7.285
7.274
7.256
7.253
7.223
7.220
7.204
7.202
7.186
7.183
6.564
6.530
6.520
6.491
6.480
6.466
6.449
6.432
6.411
6.392
4.214
4.206
4.199
4.193
4.189
4.186
4.181
4.176
4.171
4.168
4.164
4.162
4.153
4.150
4.144
4.138
4.136
4.133
4.126
4.118
2.858
2.839
2.801
2.784
1.376
1.358
1.341
EtO
EtO
P
O
O
(*E*)-**5t**
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
ppm
1.00
0.99
1.24
1.01
2.96
4.04
2.00
5.96

(*E*)-5t: ^{31}P NMR

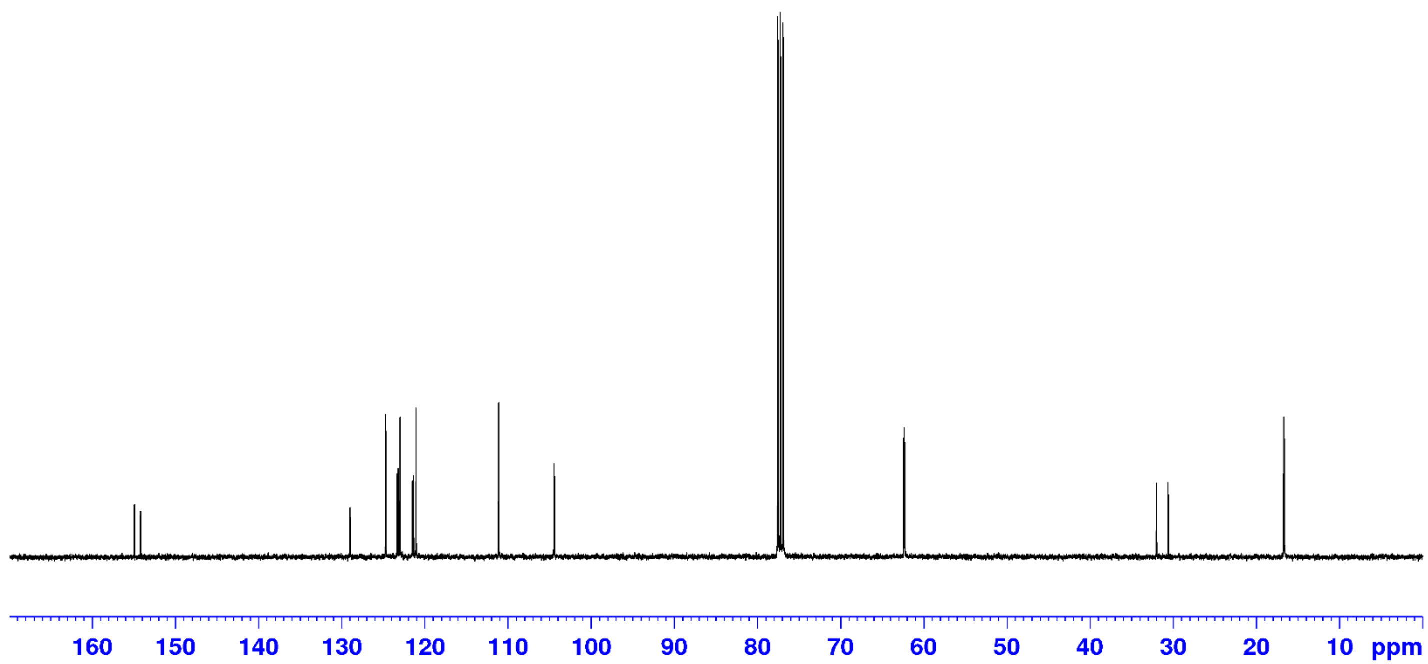


(*E*)-**5t**

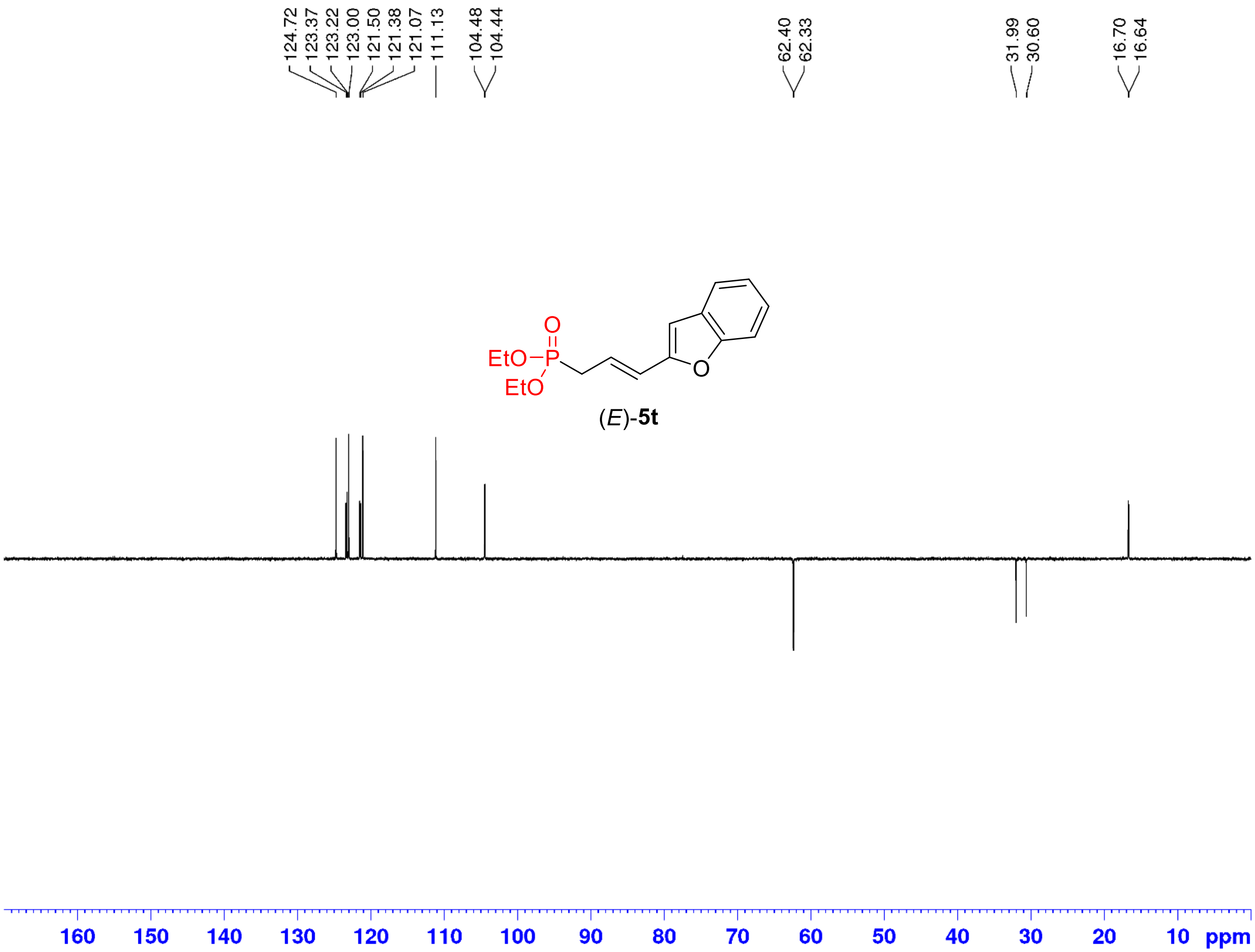
(*E*)-5t: ^{13}C NMR



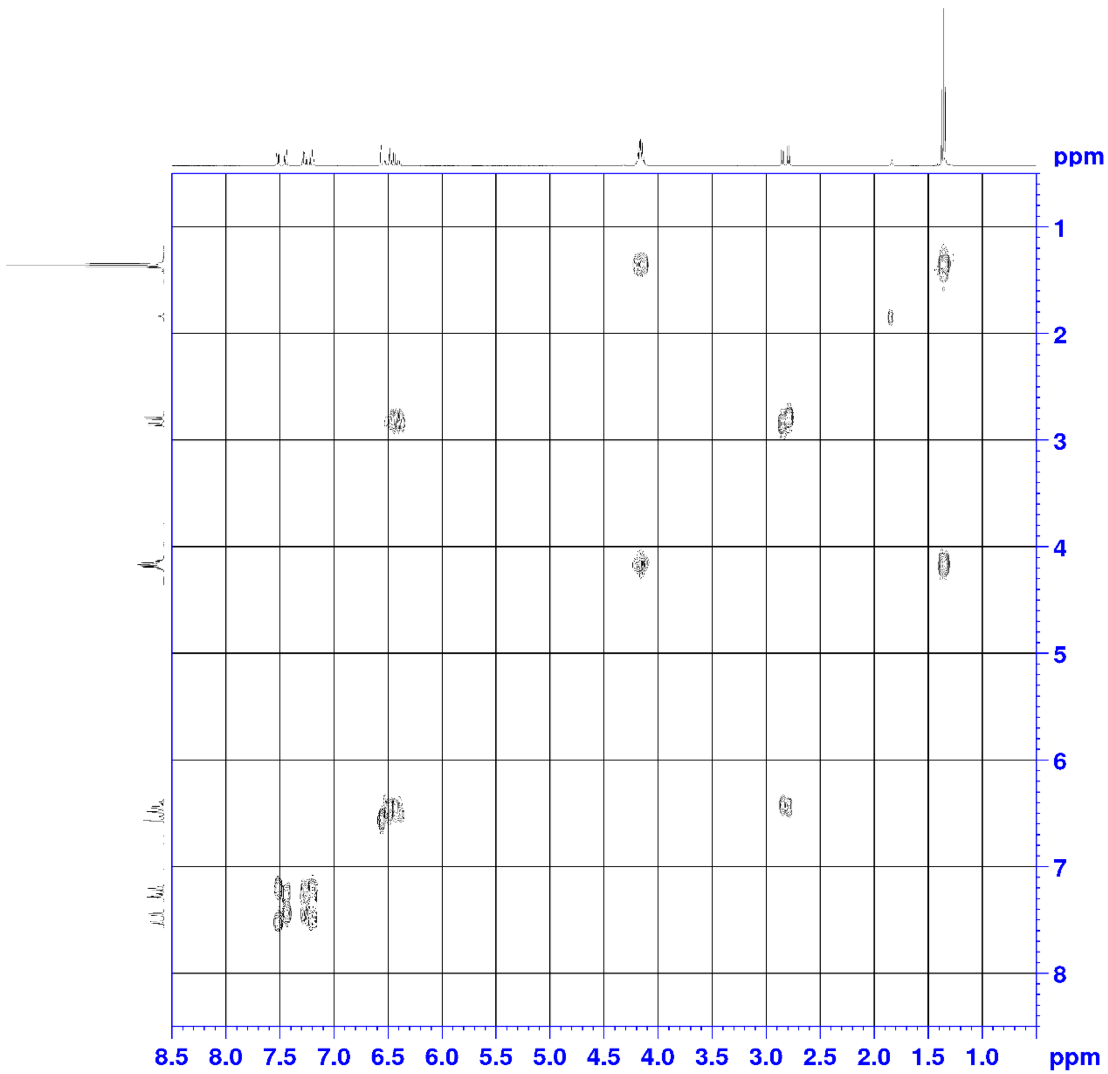
(*E*)-**5t**



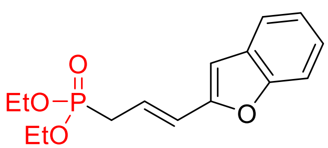
(E)-5t: ^{13}C DEPT135



(*E*)-5t: COSY



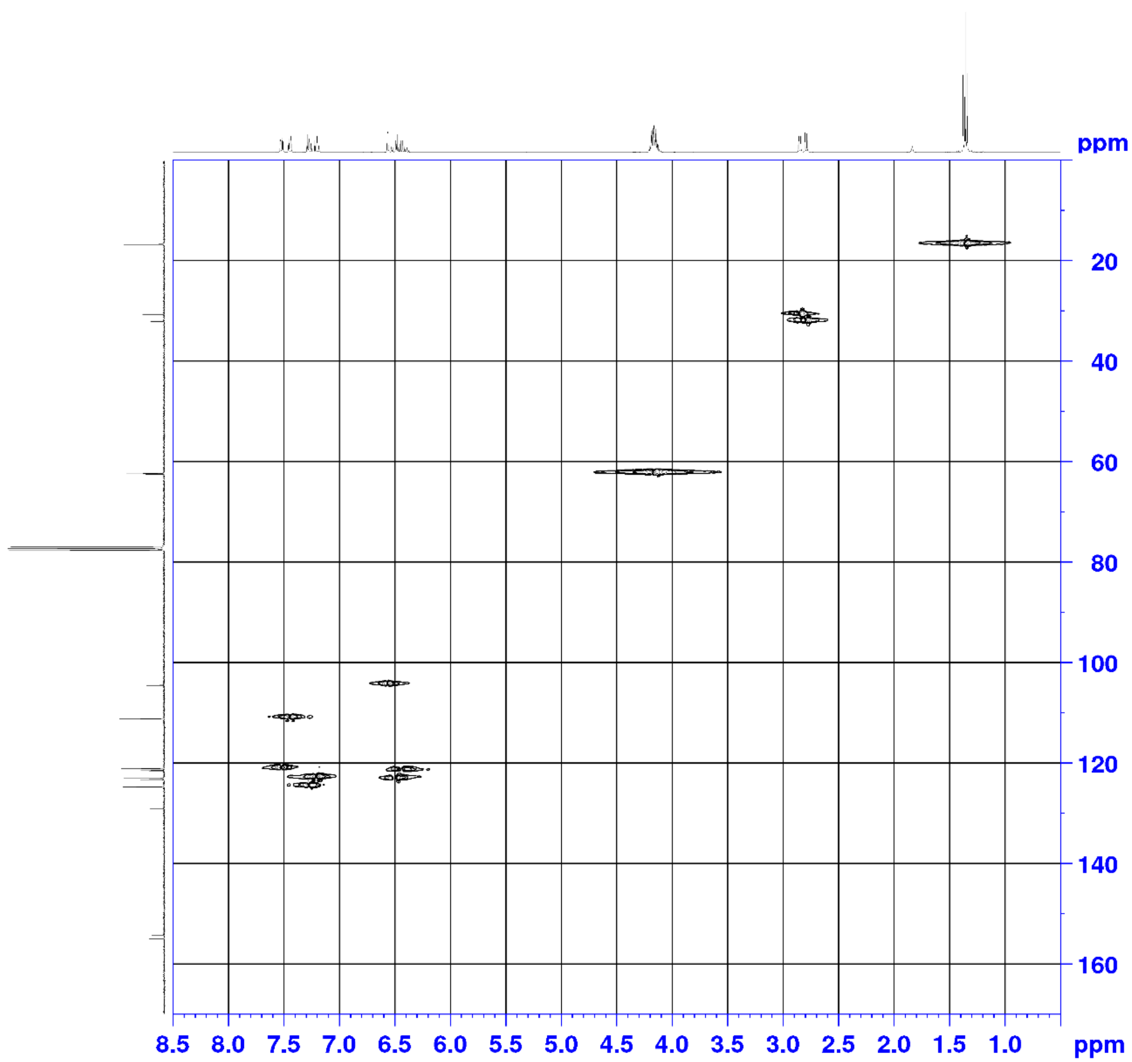
ppm
1
2
3
4
5
6
7
8
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

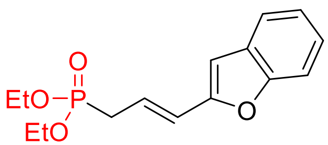


O
EtO
P
EtO
O

(*E*)-**5t**

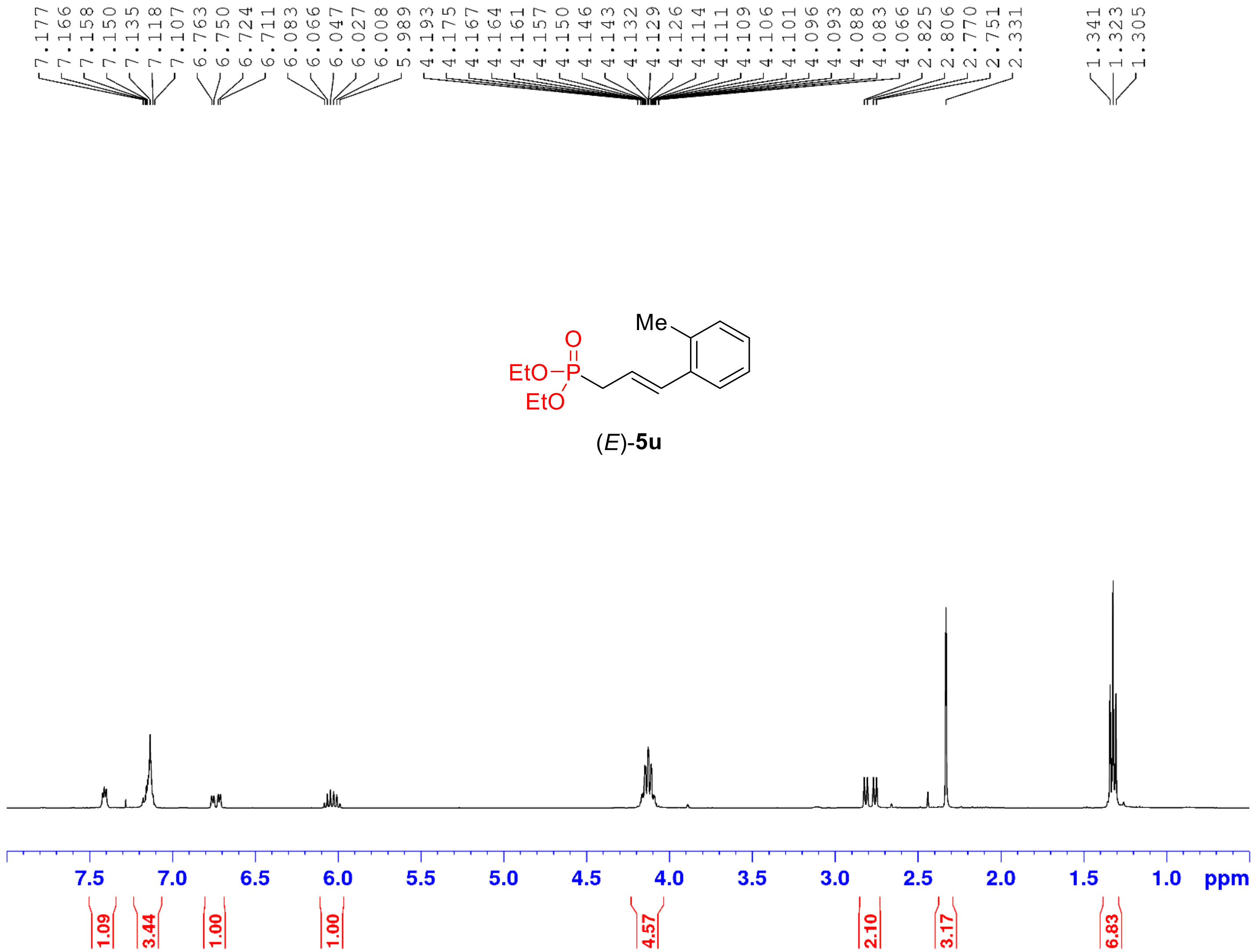
(*E*)-5t: ^{13}C HSQC



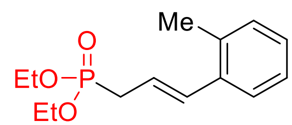


(*E*)-**5t**

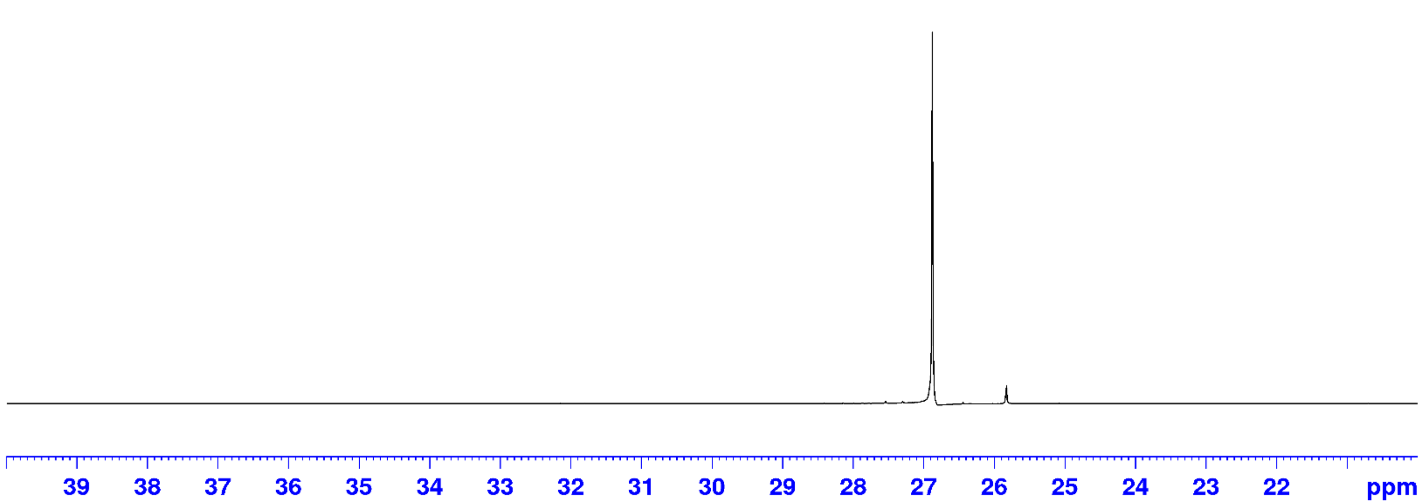
(*E*)-5u: ^{1}H NMR


7.177
7.166
7.158
7.150
7.135
7.118
7.107
6.763
6.750
6.724
6.711
6.083
6.066
6.047
6.027
6.008
5.989
4.193
4.175
4.167
4.164
4.161
4.157
4.150
4.146
4.143
4.132
4.129
4.126
4.114
4.111
4.109
4.106
4.101
4.096
4.093
4.088
4.083
4.066
2.825
2.806
2.770
2.751
2.331
1.341
1.323
1.305
Me
O
EtO
P
EtO
(E)-5u
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
1.09
3.44
1.00
1.00
4.57
2.10
3.17
6.83

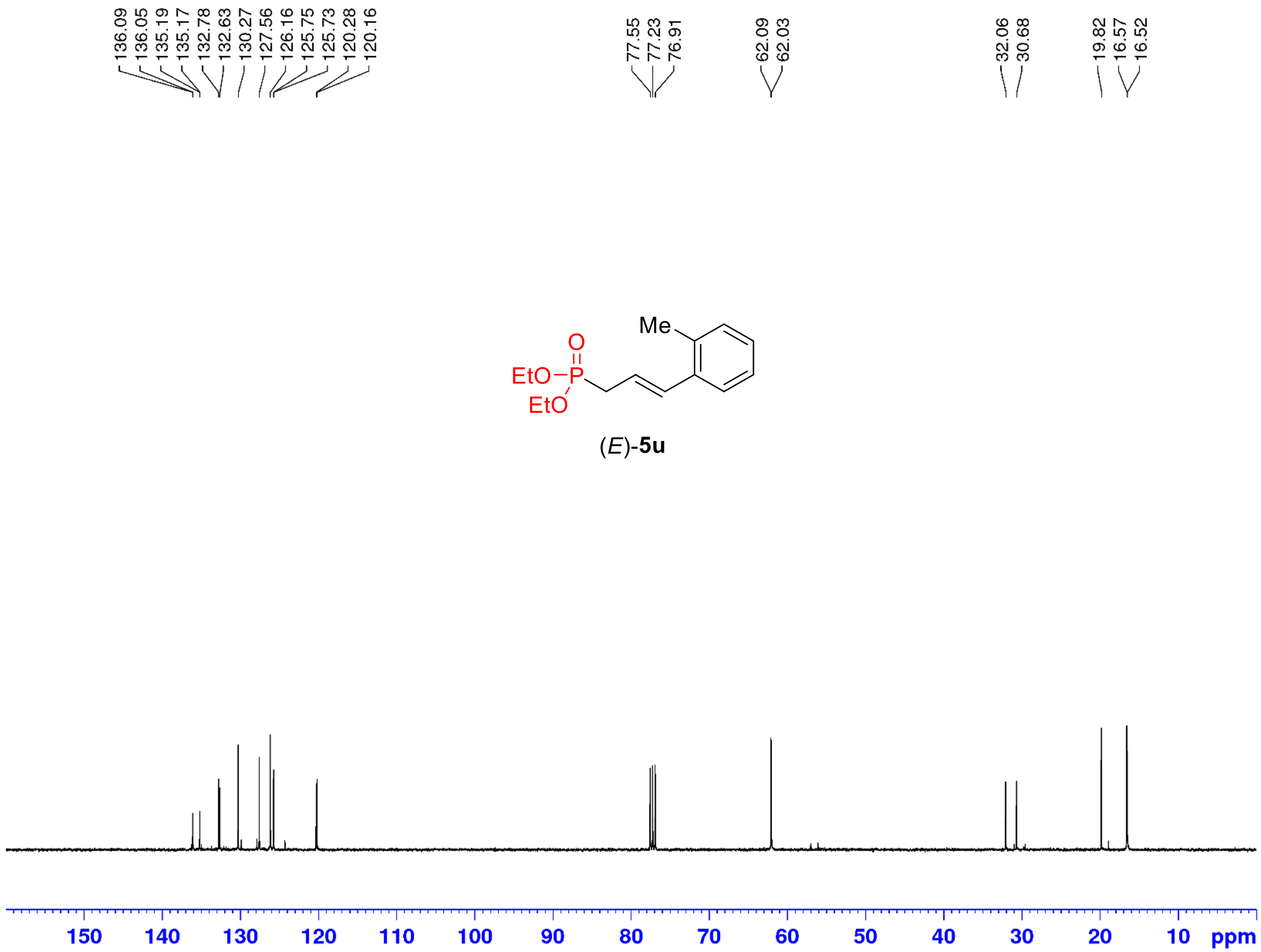
(*E*)-5u: ^{31}P NMR



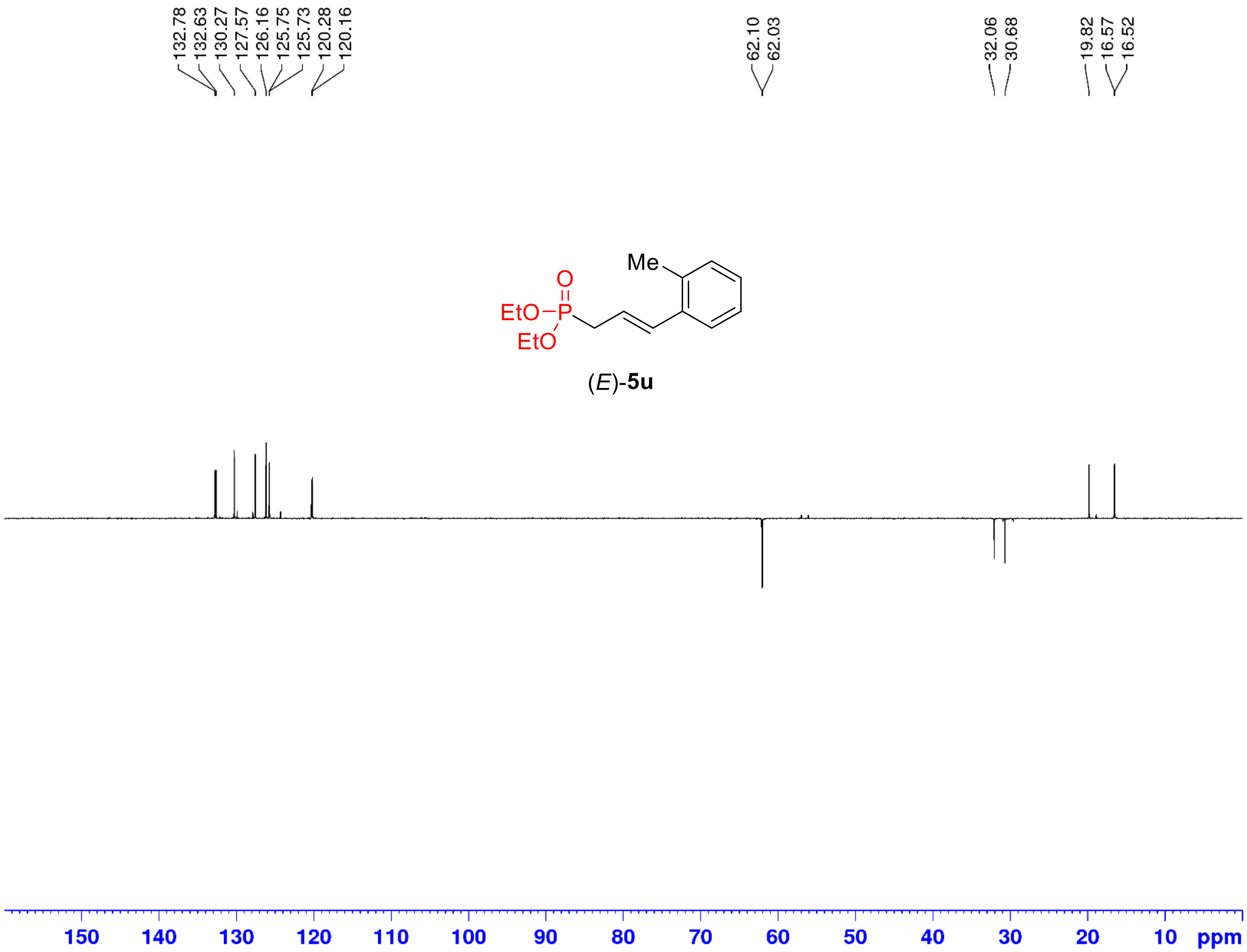
(*E*)-**5u**



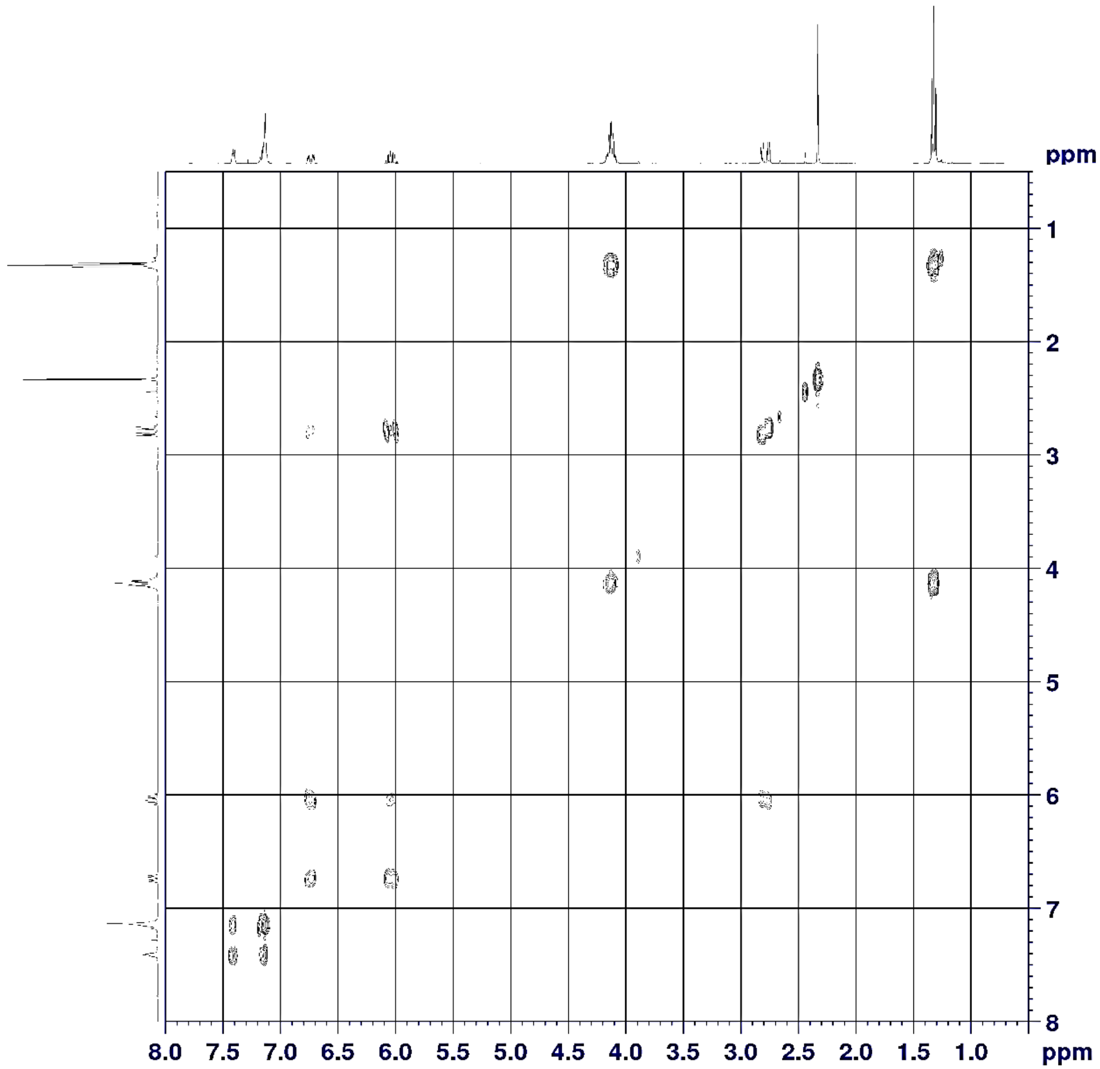
(*E*)-5u: ^{13}C NMR

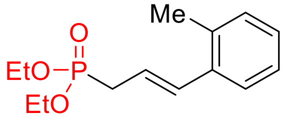

136.09
136.05
135.19
135.17
132.78
132.63
130.27
127.56
126.16
125.75
125.73
120.28
120.16
77.55
77.23
76.91
62.09
62.03
32.06
30.68
19.82
16.57
16.52
Me
O
EtO
P
EtO
(E)-5u
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

(*E*)-5u: ^{13}C DEPT135



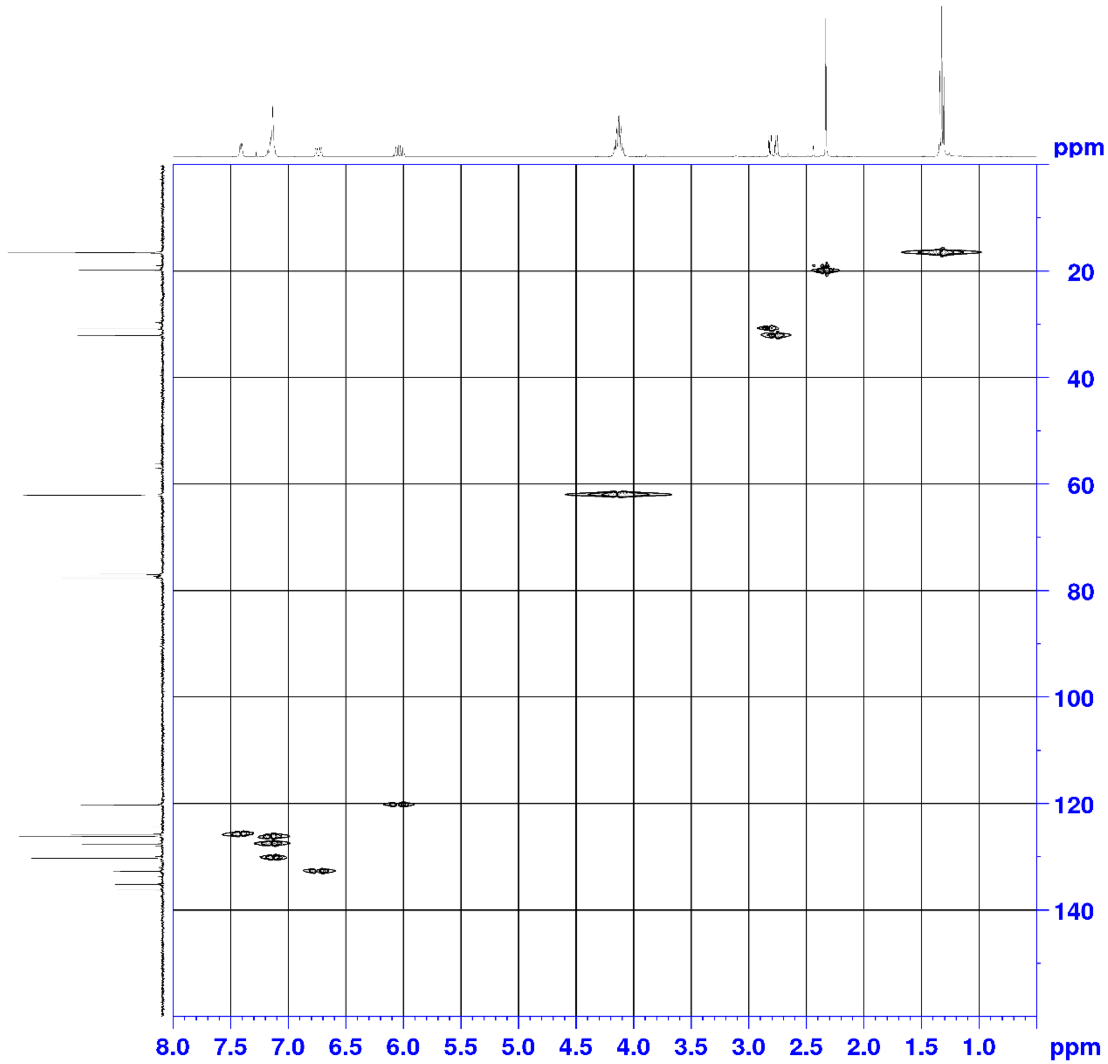
(*E*)-5u: COSY

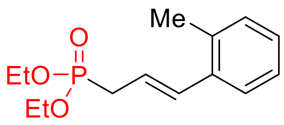

ppm
1
2
3
4
5
6
7
8
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm


Me
O
EtO
P
EtO

(*E*)-**5u**

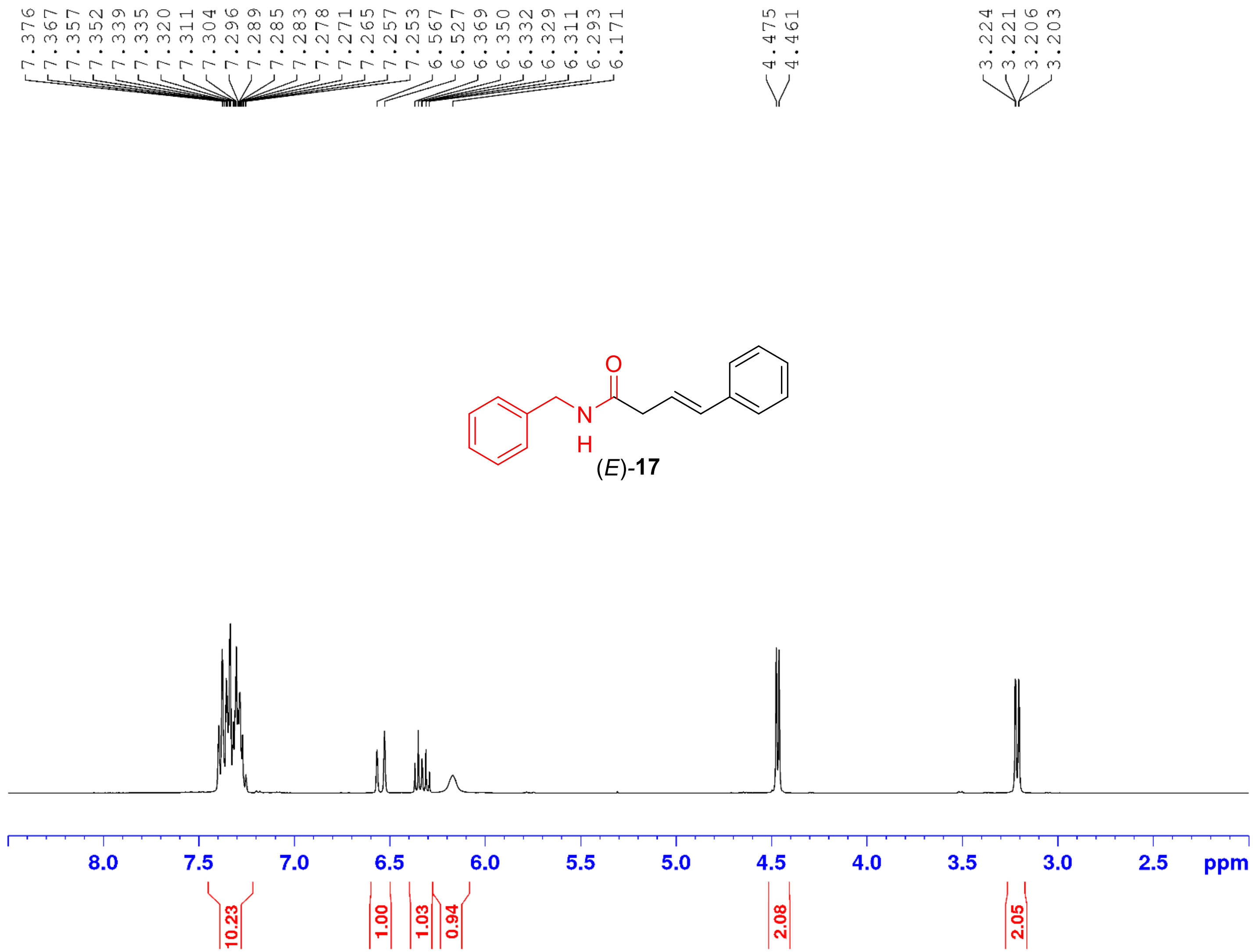
(*E*)-5u: ^{13}C HSQC



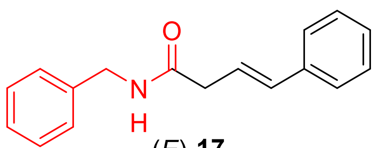


(*E*)-**5u**

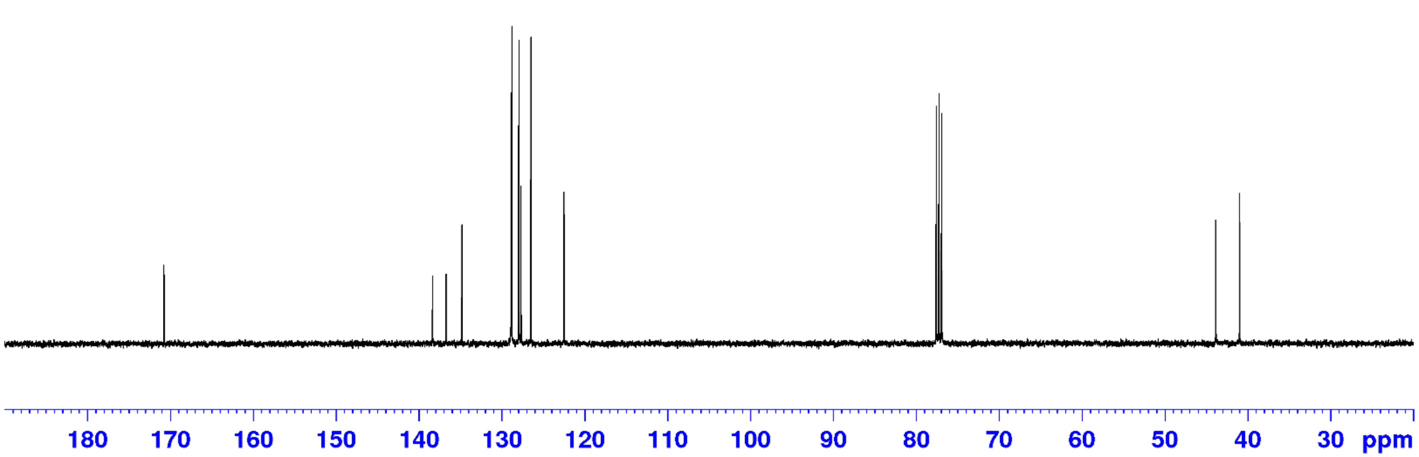
(*E*)-17: ^{1}H NMR


7.376
7.367
7.357
7.352
7.339
7.335
7.320
7.311
7.304
7.296
7.289
7.285
7.283
7.278
7.271
7.265
7.257
7.253
6.567
6.527
6.369
6.350
6.332
6.329
6.311
6.293
6.171
4.475
4.461
3.224
3.221
3.206
3.203
O
N
H
(E)-17
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
ppm
10.23
1.00
1.03
0.94
2.08
2.05

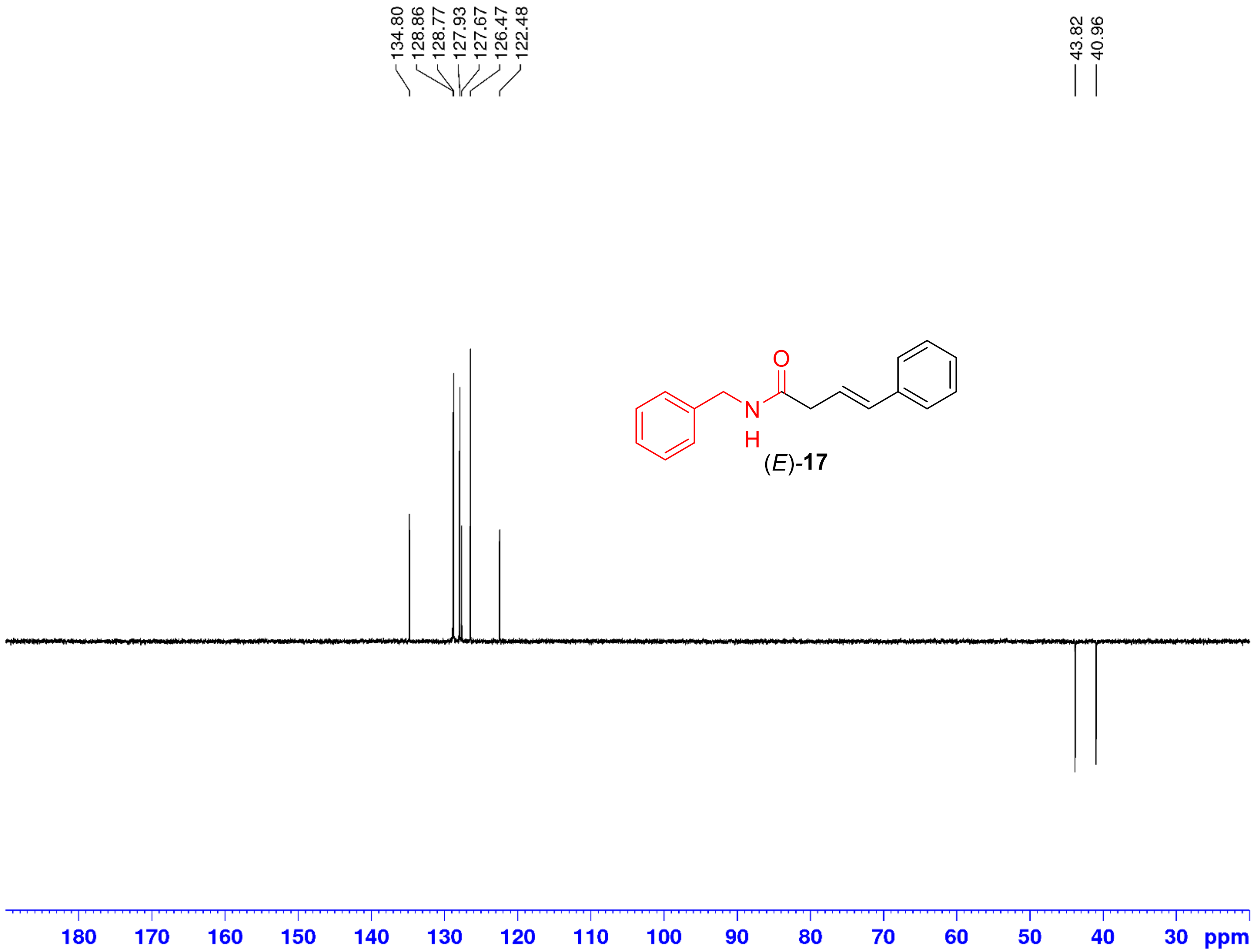
(*E*)-17: ^{13}C NMR



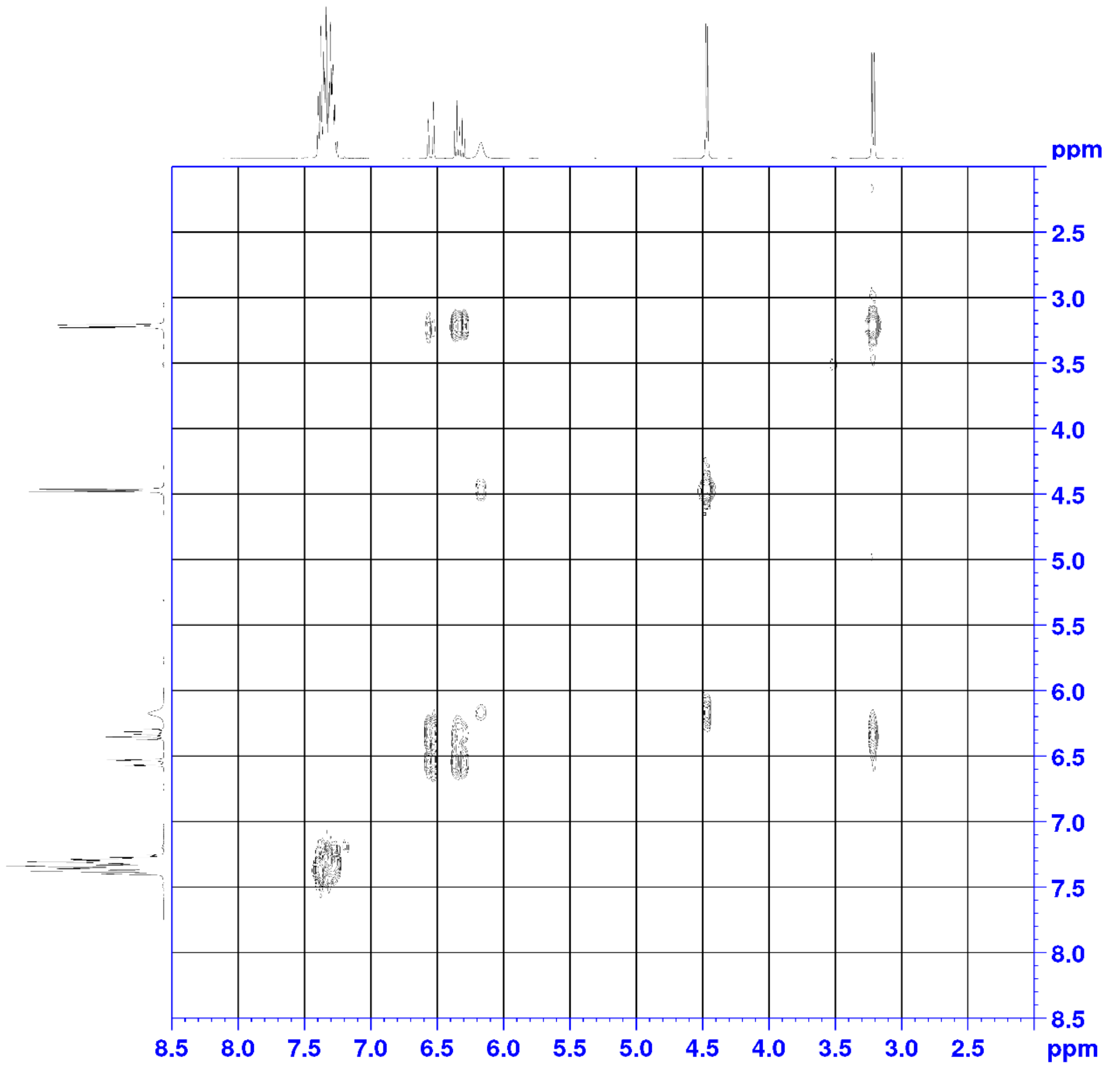
(*E*)-**17**

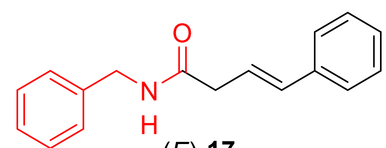


(*E*)-17: ^{13}C DEPT135



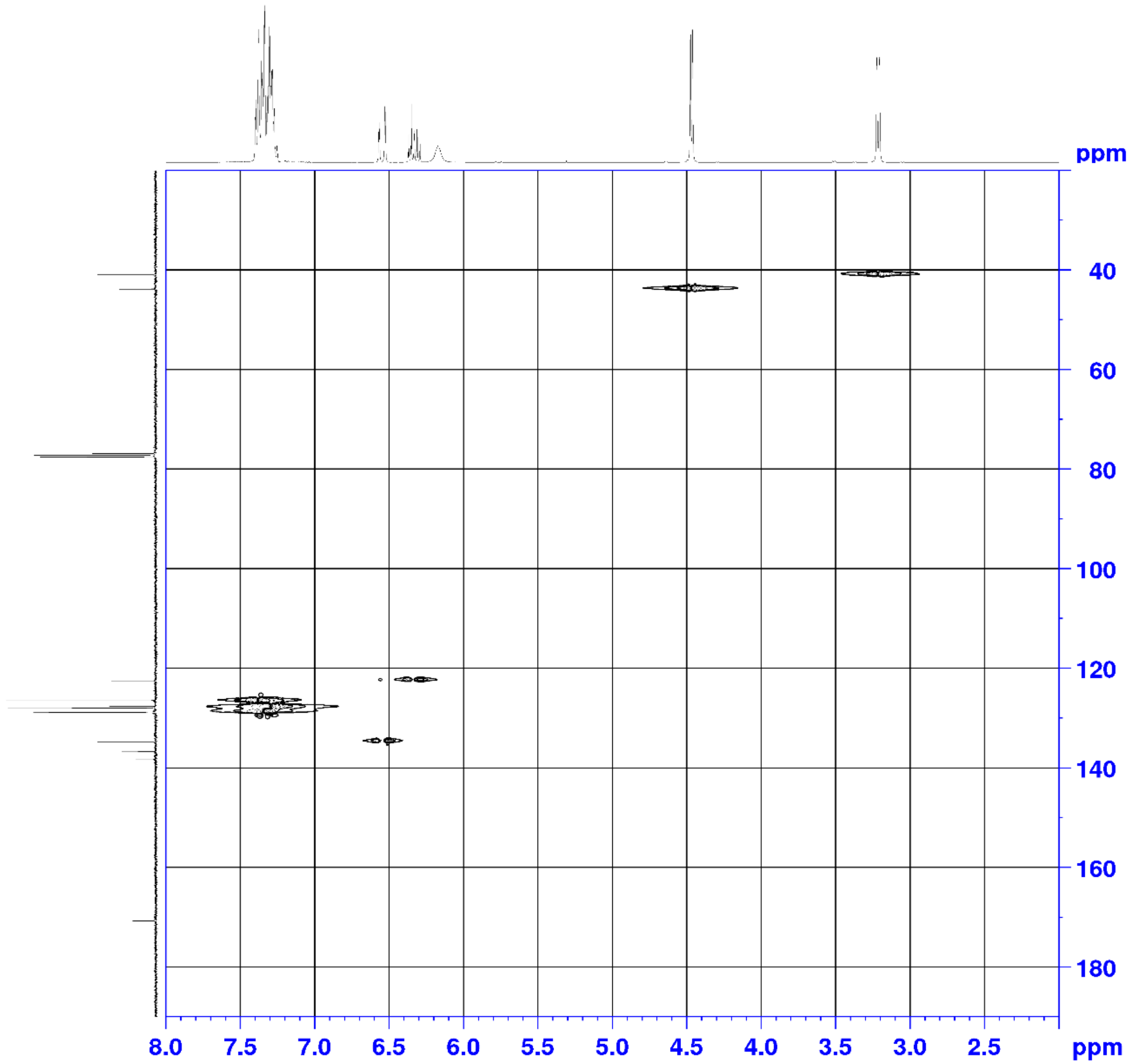
(*E*)-17: COSY

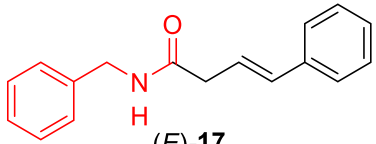

ppm
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8.5
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
ppm


O
N
H

(*E*)-**17**

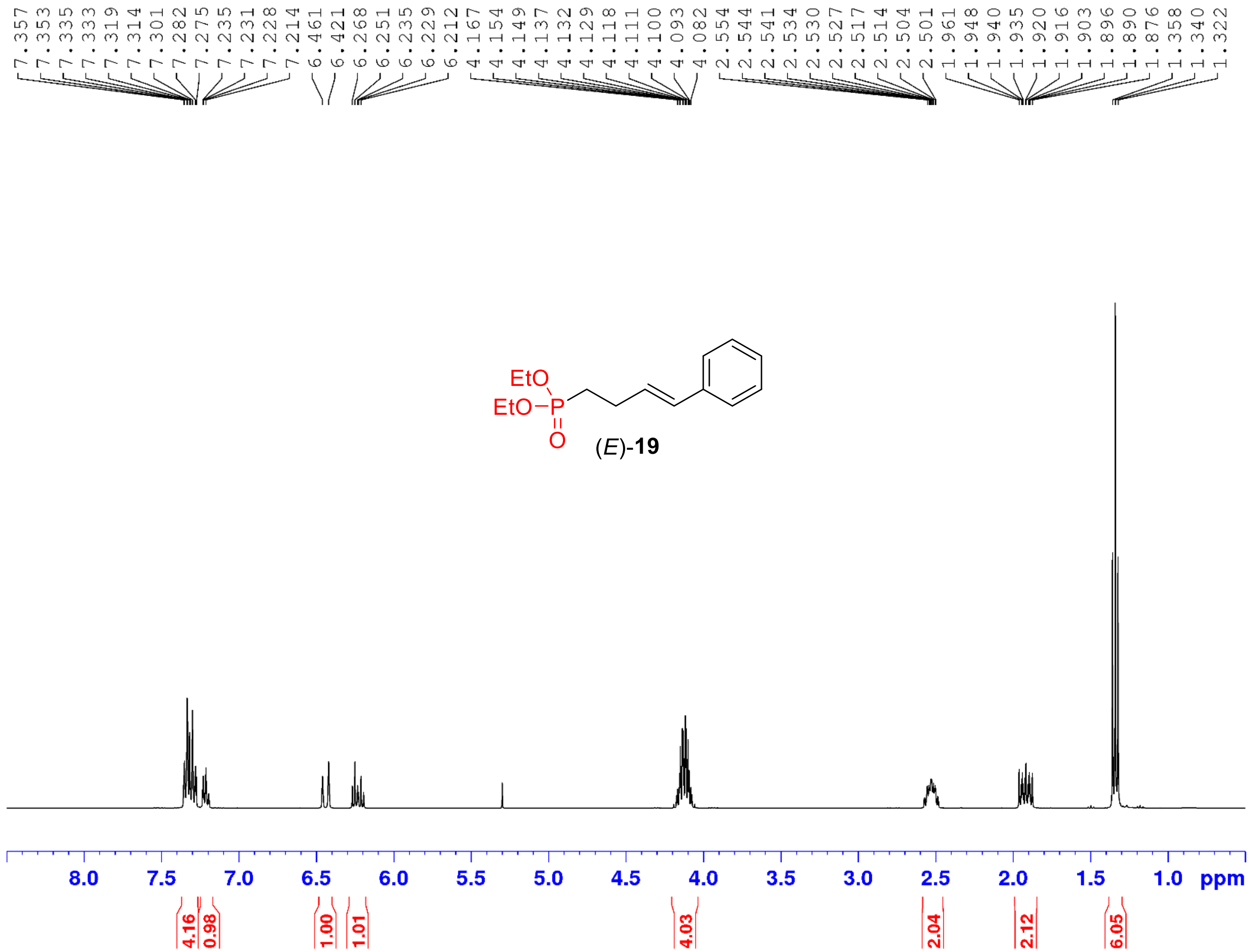
(*E*)-17: ^{13}C HSQC





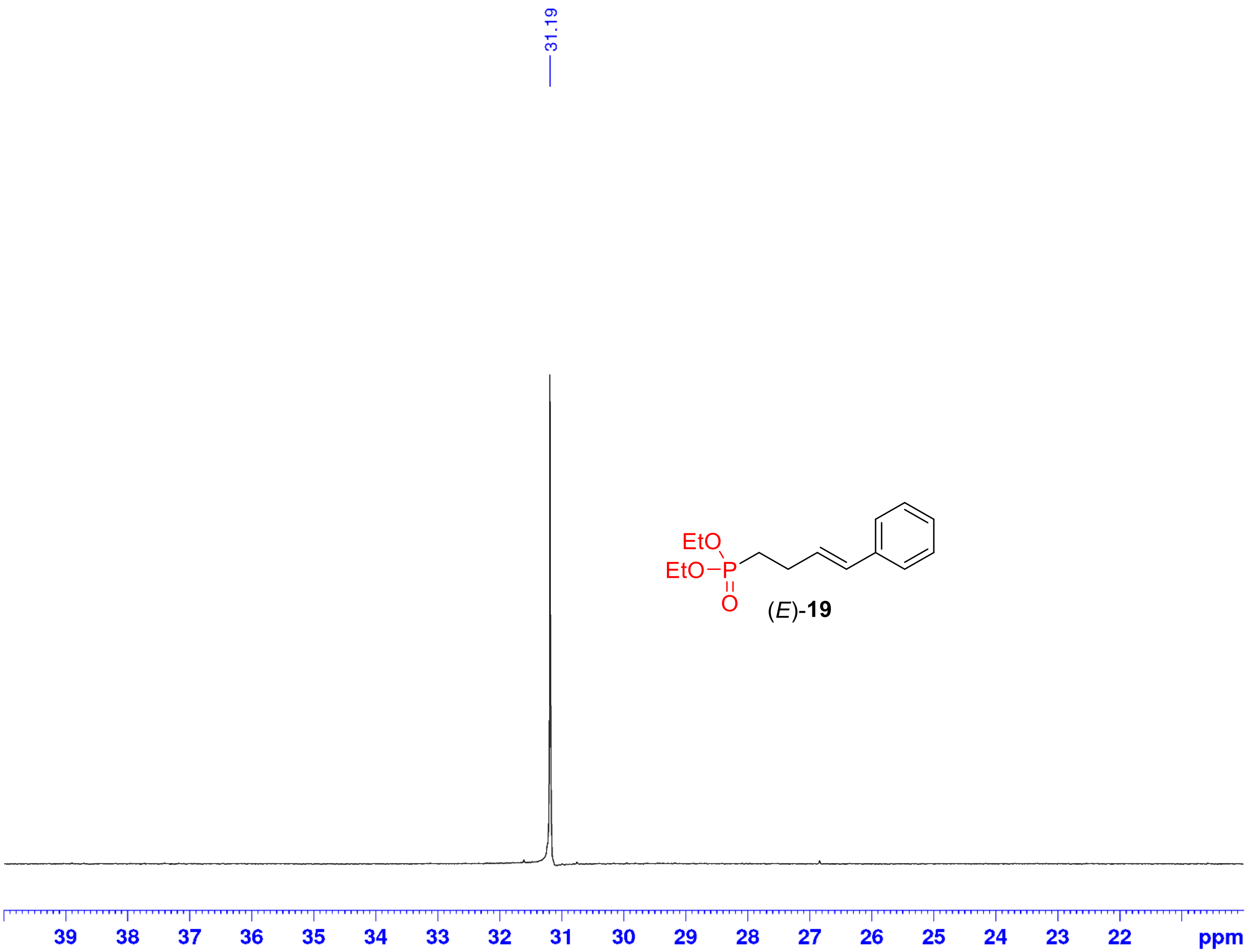
(*E*)-**17**

(*E*)-19: ^{1}H NMR

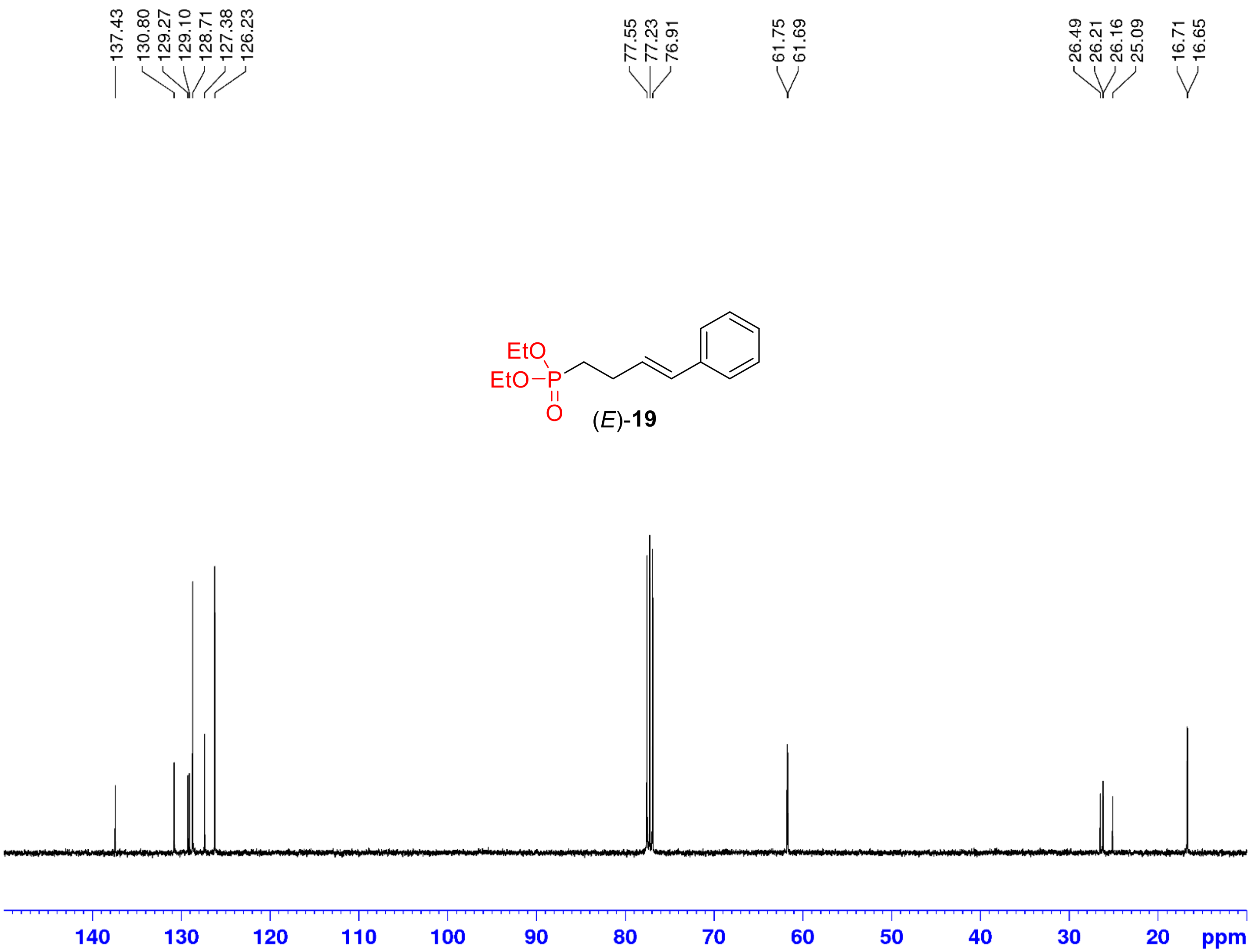


7.357
7.353
7.335
7.333
7.319
7.314
7.301
7.282
7.275
7.235
7.231
7.228
7.214
6.461
6.421
6.268
6.251
6.235
6.229
6.212
4.167
4.154
4.149
4.137
4.132
4.129
4.118
4.111
4.100
4.093
4.082
2.554
2.544
2.541
2.534
2.530
2.527
2.517
2.514
2.504
2.501
1.961
1.948
1.940
1.935
1.920
1.916
1.903
1.896
1.890
1.876
1.358
1.340
1.322
EtO
EtO
P
O
(E)-19
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
ppm
4.16
0.98
1.00
1.01
4.03
2.04
2.12
6.05

(*E*)-19: ^{31}P NMR

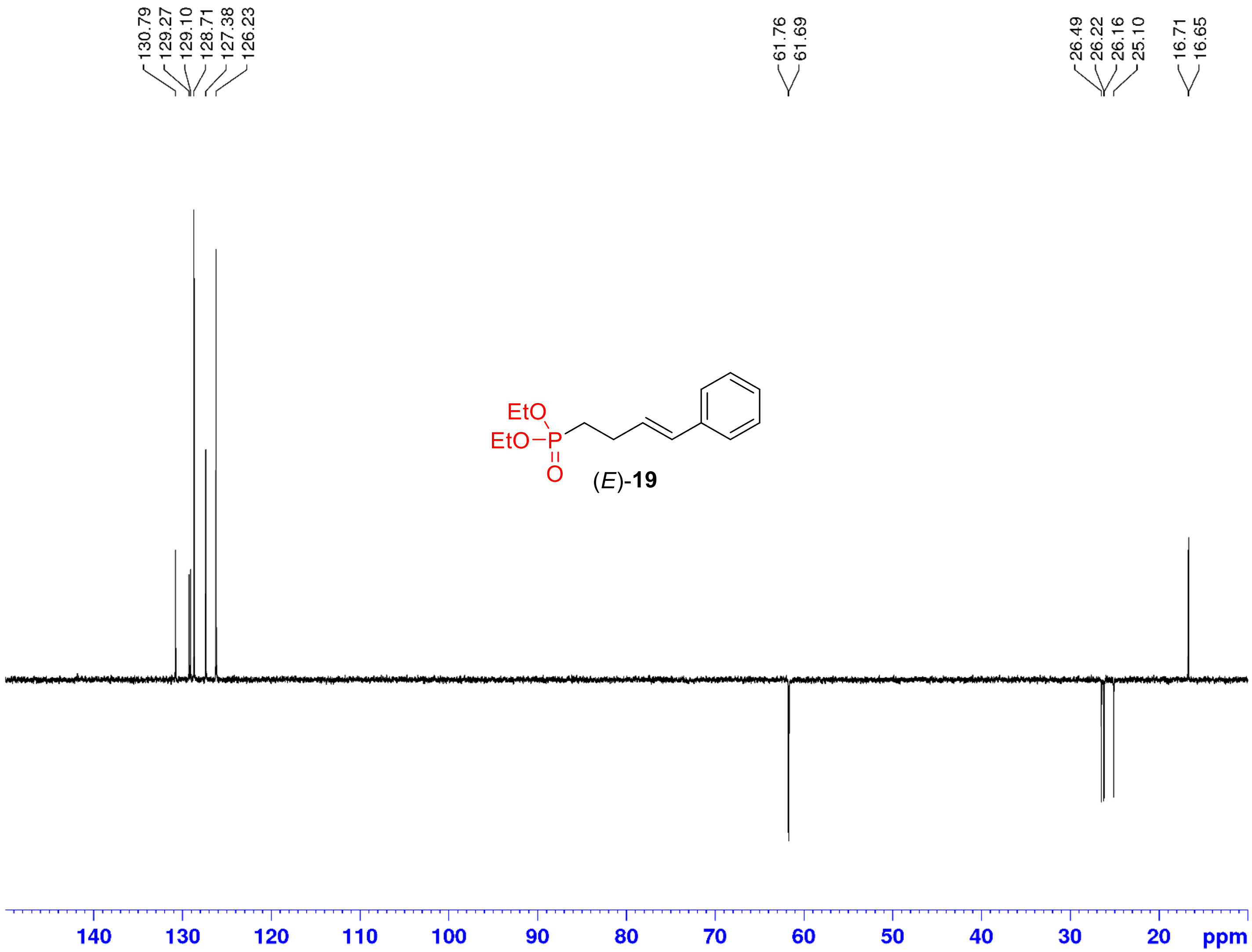

31.19
EtO
EtO
P
O
(E)-19
39
38
37
36
35
34
33
32
31
30
29
28
27
26
25
24
23
22
ppm

(*E*)-19: ^{13}C NMR

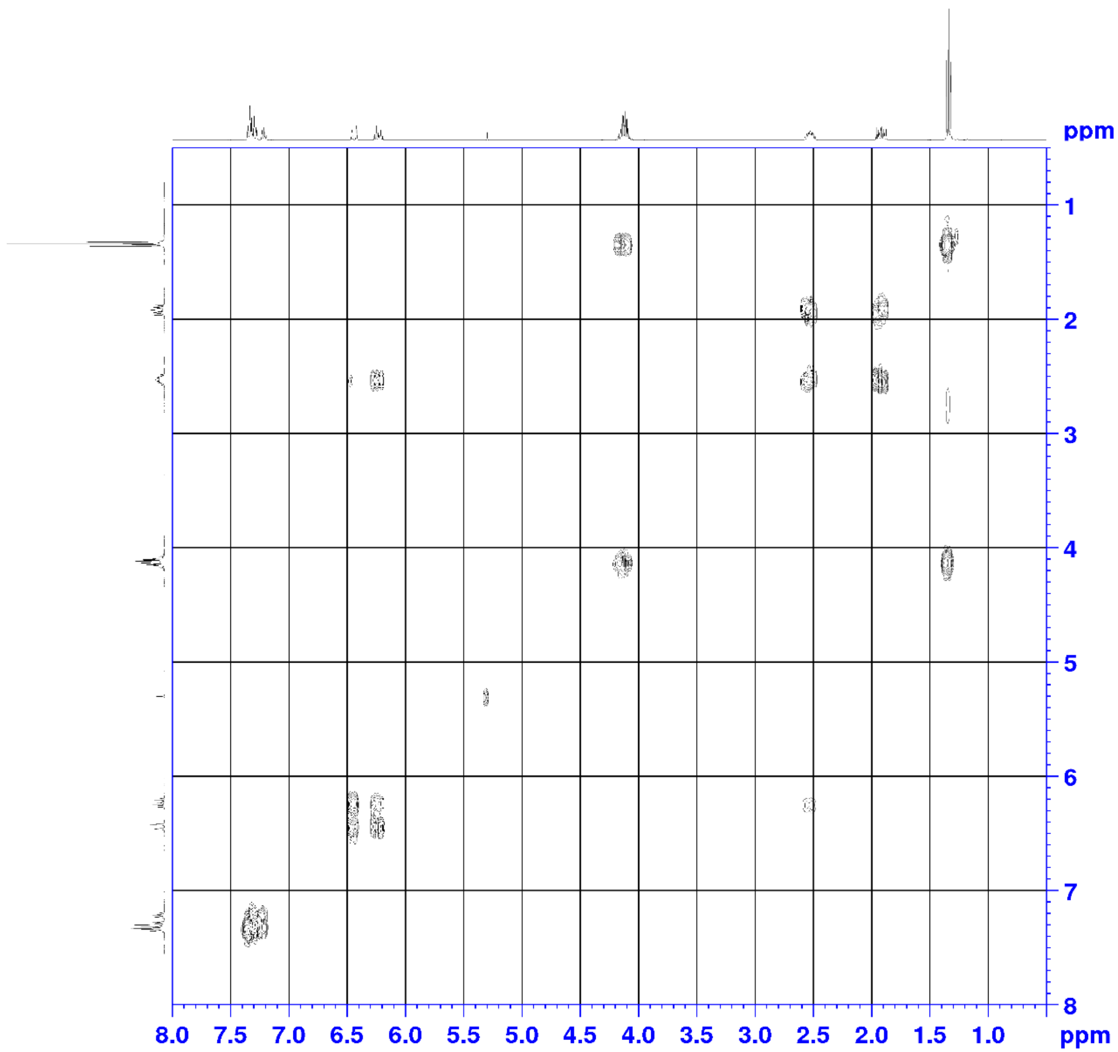


137.43
130.80
129.27
129.10
128.71
127.38
126.23
77.55
77.23
76.91
61.75
61.69
26.49
26.21
26.16
25.09
16.71
16.65
EtO
EtO
P
O
(E)-19
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

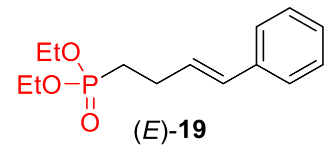
(*E*)-19: ^{13}C DEPT135



(*E*)-19: COSY



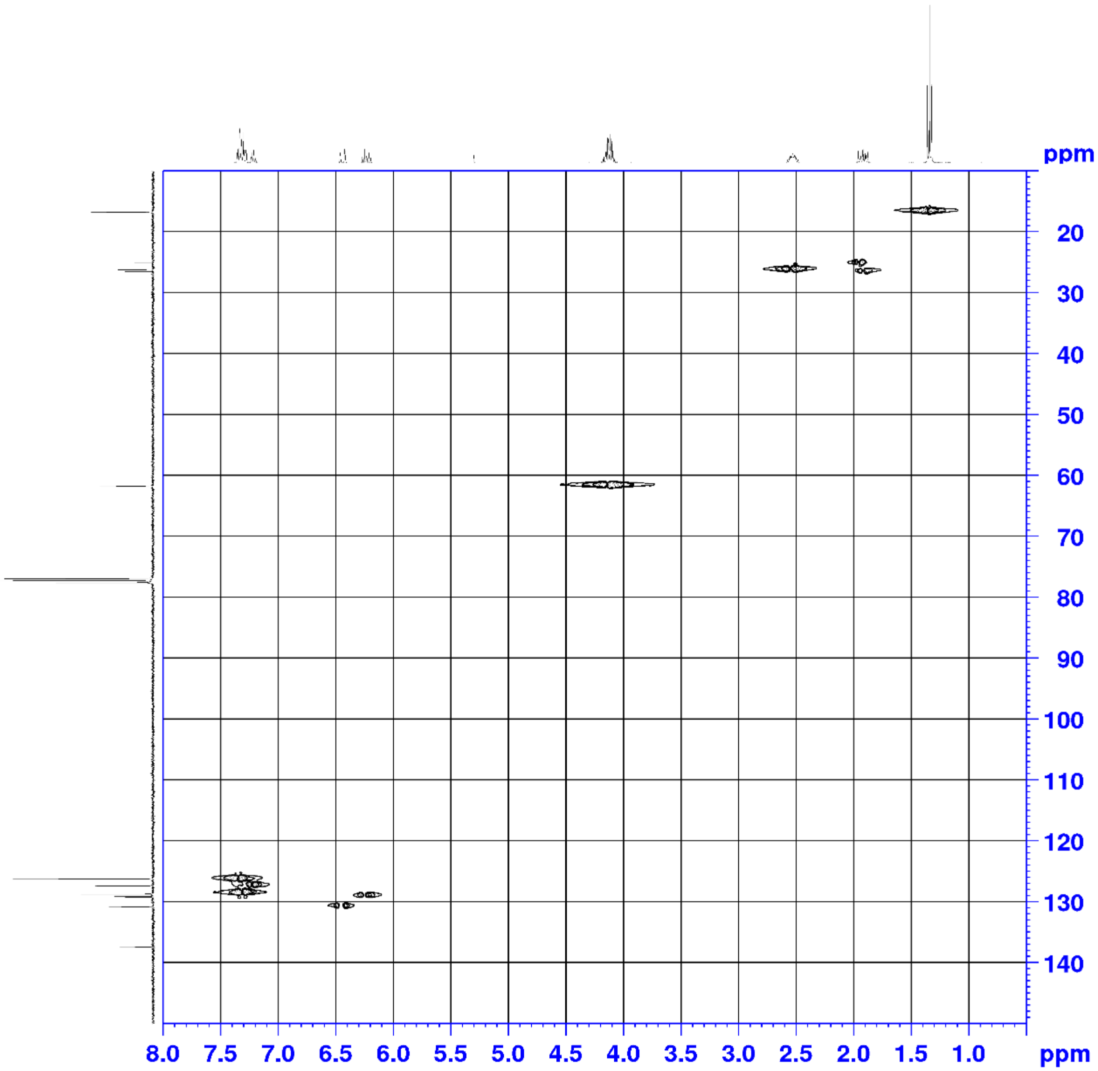
ppm
1
2
3
4
5
6
7
8
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

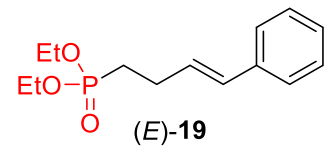


EtO
EtO
P
O

(*E*)-**19**

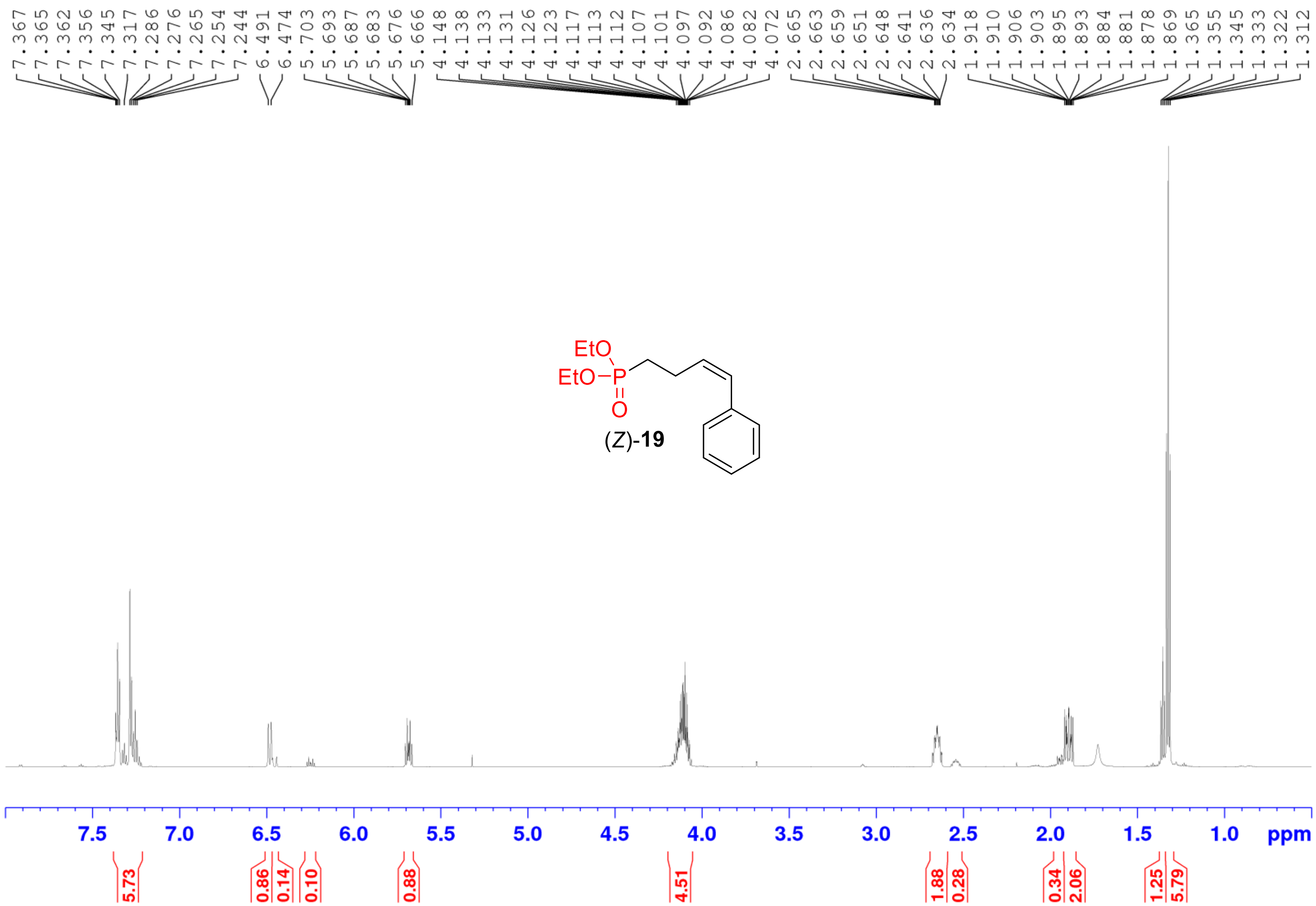
(*E*)-19: ^{13}C HSQC





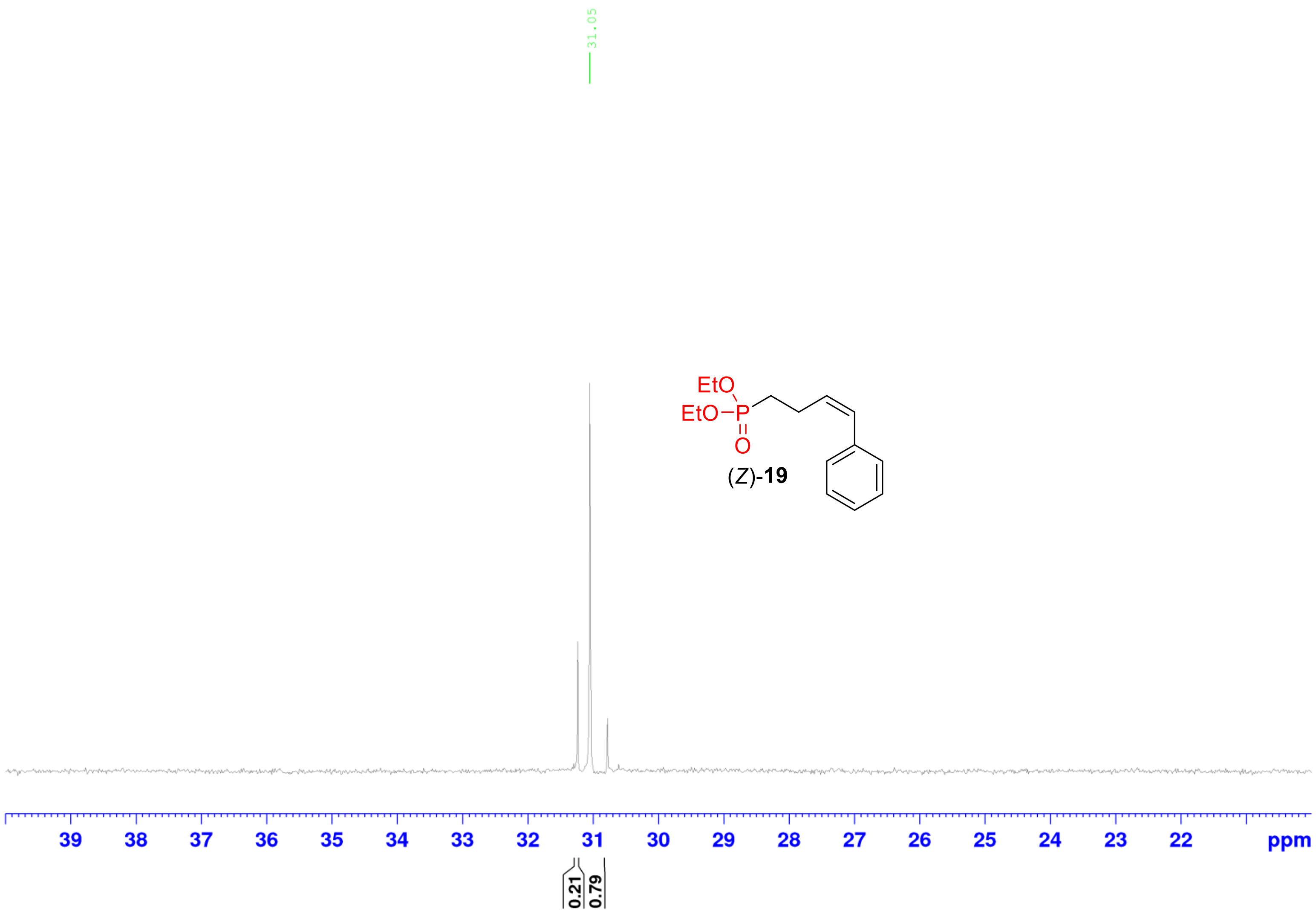
(*E*)-**19**

(*Z*)-19: ^{1}H NMR



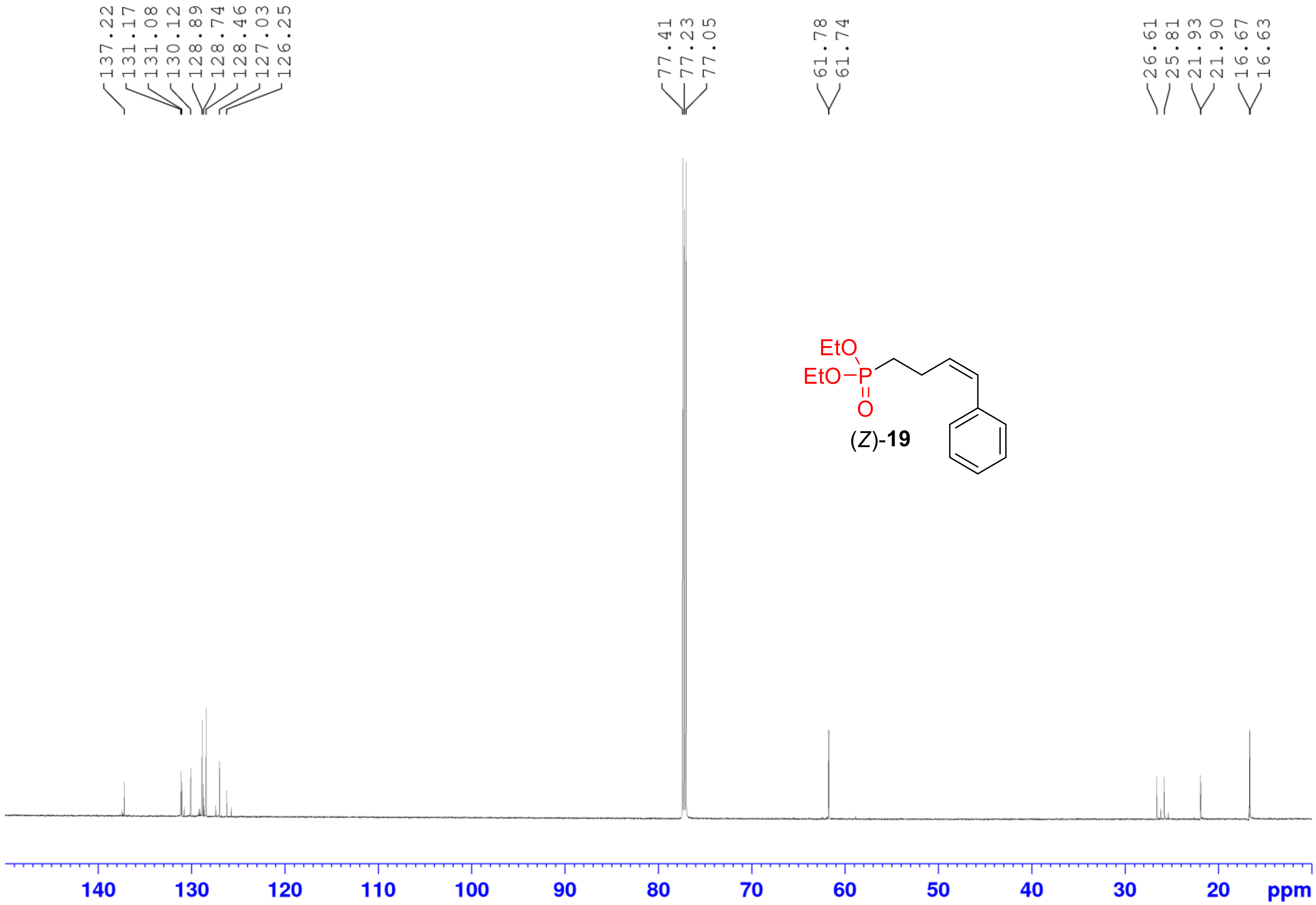
7.367
7.365
7.362
7.356
7.345
7.317
7.286
7.276
7.265
7.254
7.244
6.491
6.474
5.703
5.693
5.687
5.683
5.676
5.666
4.148
4.138
4.133
4.131
4.126
4.123
4.117
4.113
4.112
4.107
4.101
4.097
4.092
4.086
4.082
4.072
2.665
2.663
2.659
2.651
2.648
2.641
2.636
2.634
1.918
1.910
1.906
1.903
1.895
1.893
1.884
1.881
1.878
1.869
1.365
1.355
1.345
1.333
1.322
1.312
EtO
EtO
P
O
(*Z*)-**19**
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
5.73
0.86
0.14
0.10
0.88
4.51
1.88
0.28
0.34
2.06
1.25
5.79

(*Z*)-19: ^{31}P NMR

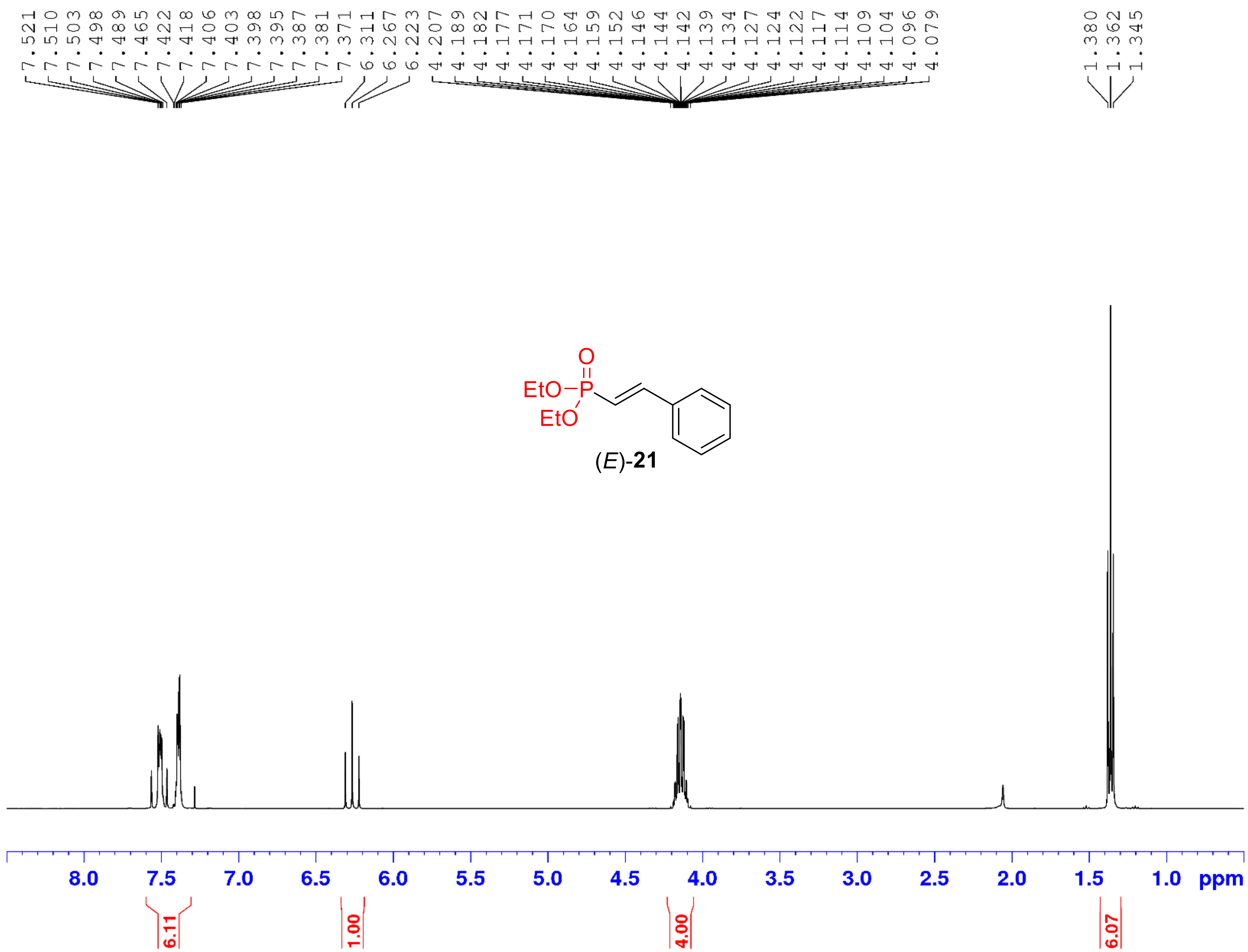


31.05
EtO
EtO
P
O
(Z)-19
39
38
37
36
35
34
33
32
31
30
29
28
27
26
25
24
23
22
ppm
0.21
0.79

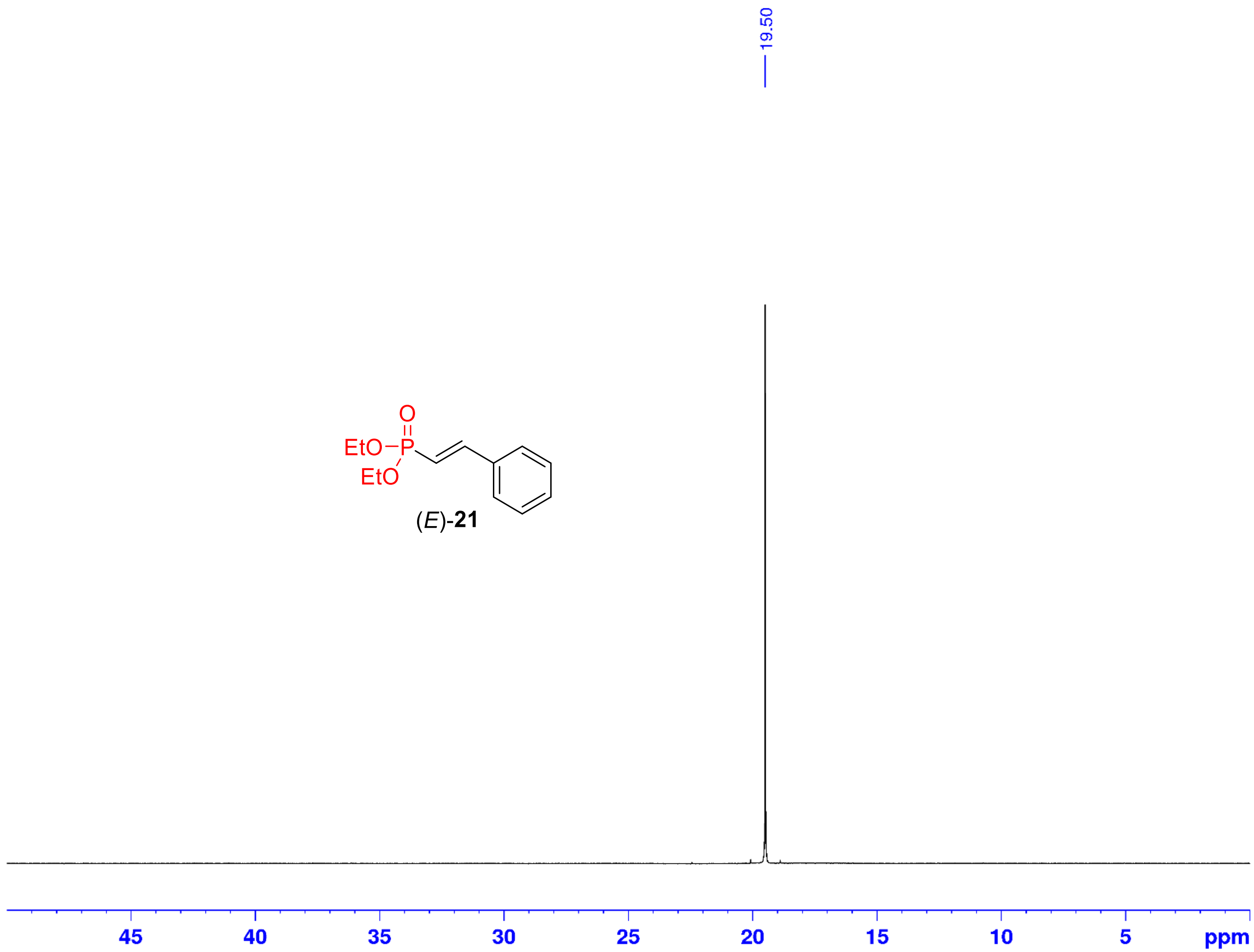
(*Z*)-19: ^{13}C NMR


137.22
131.17
131.08
130.12
128.89
128.74
128.46
127.03
126.25
77.41
77.23
77.05
61.78
61.74
26.61
25.81
21.93
21.90
16.67
16.63
EtO
EtO
P
O
(Z)-19
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

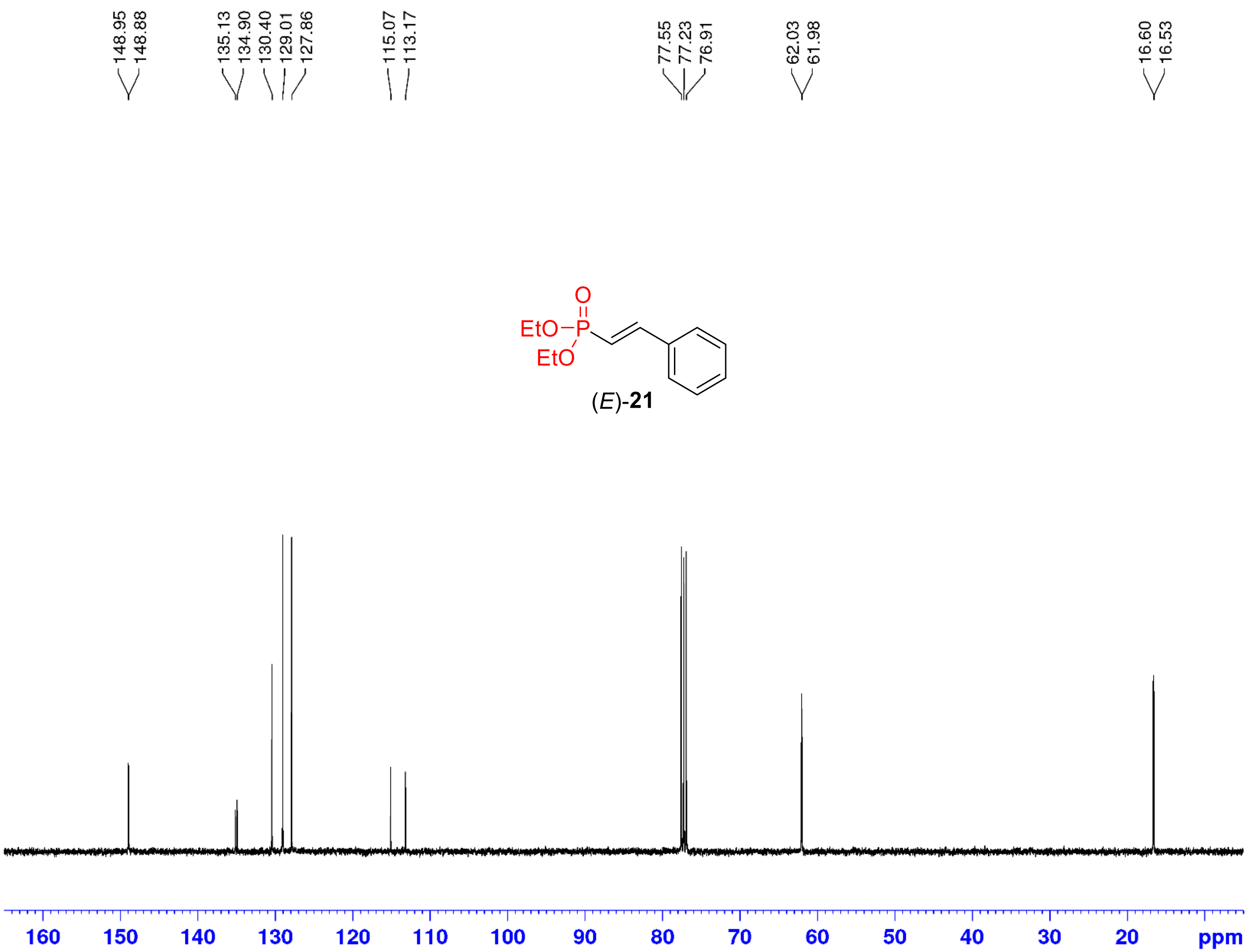
(*E*)-21: ^{1}H NMR


7.521 7.510 7.503 7.498 7.489 7.465 7.422 7.418 7.406 7.403 7.398 7.395 7.387 7.381 7.371
6.311 6.267 6.223
4.207 4.189 4.182 4.177 4.171 4.170 4.164 4.159 4.152 4.146 4.144 4.142 4.139 4.134 4.127 4.124 4.122 4.117 4.114 4.109 4.104 4.096 4.079
1.380 1.362 1.345
O
EtO
P
EtO
(E)-21
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 ppm
6.11
1.00
4.00
6.07

(*E*)-21: ^{31}P NMR

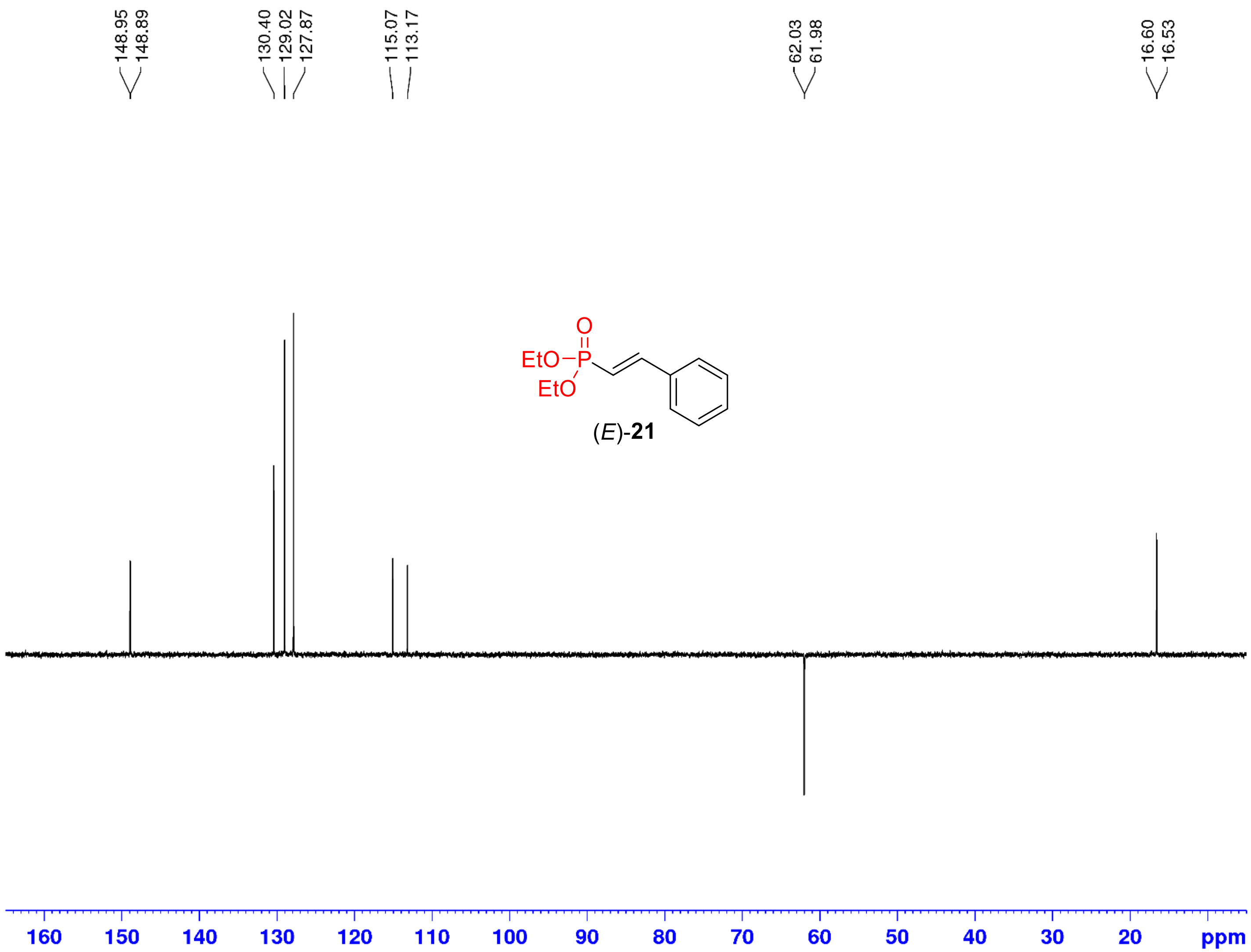


(*E*)-21: ^{13}C NMR

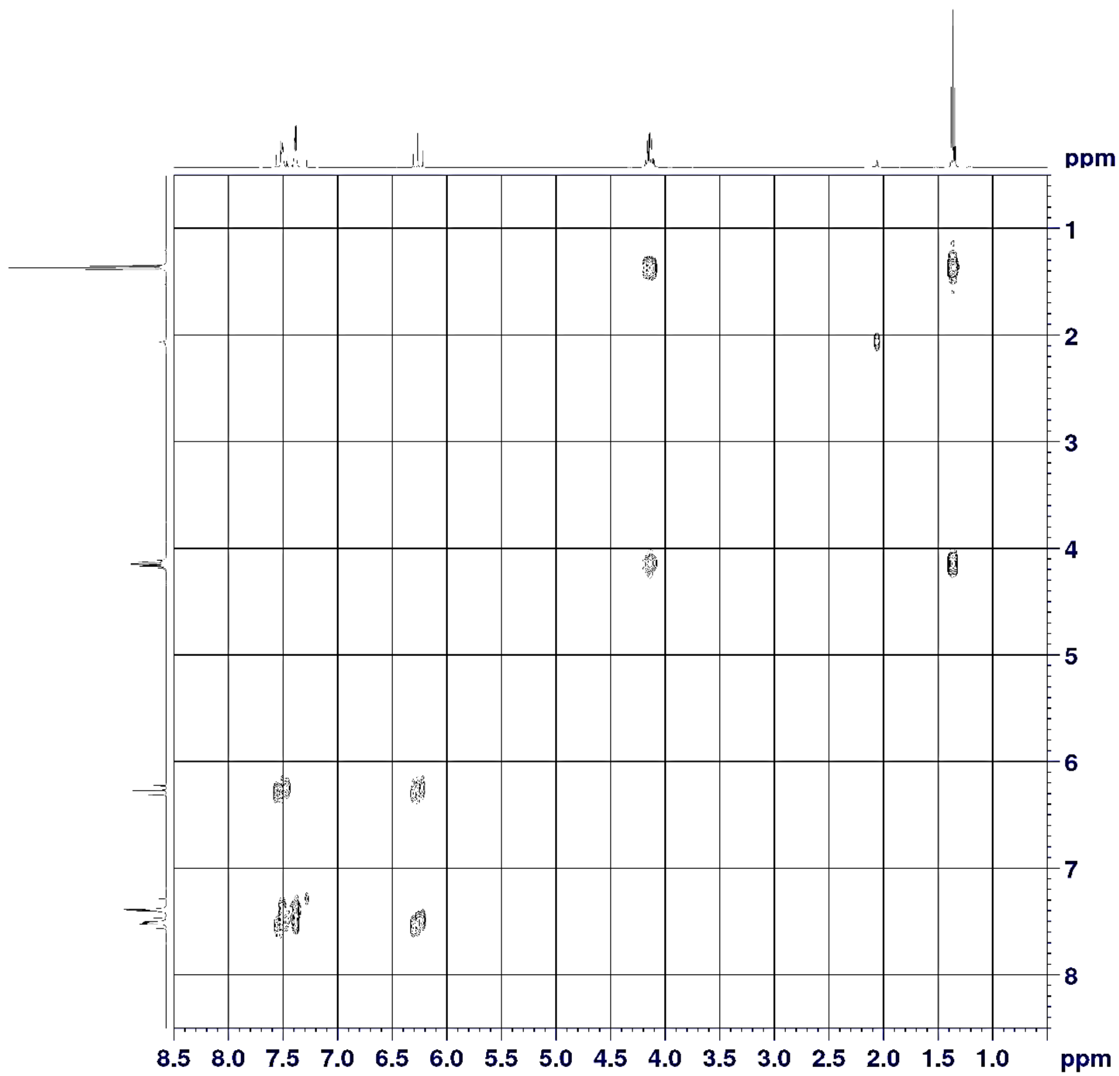


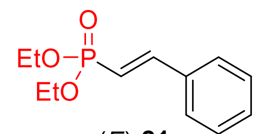
148.95
148.88
135.13
134.90
130.40
129.01
127.86
115.07
113.17
77.55
77.23
76.91
62.03
61.98
16.60
16.53
O
EtO
P
EtO
(*E*)-**21**
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

(*E*)-21: ^{13}C DEPT135



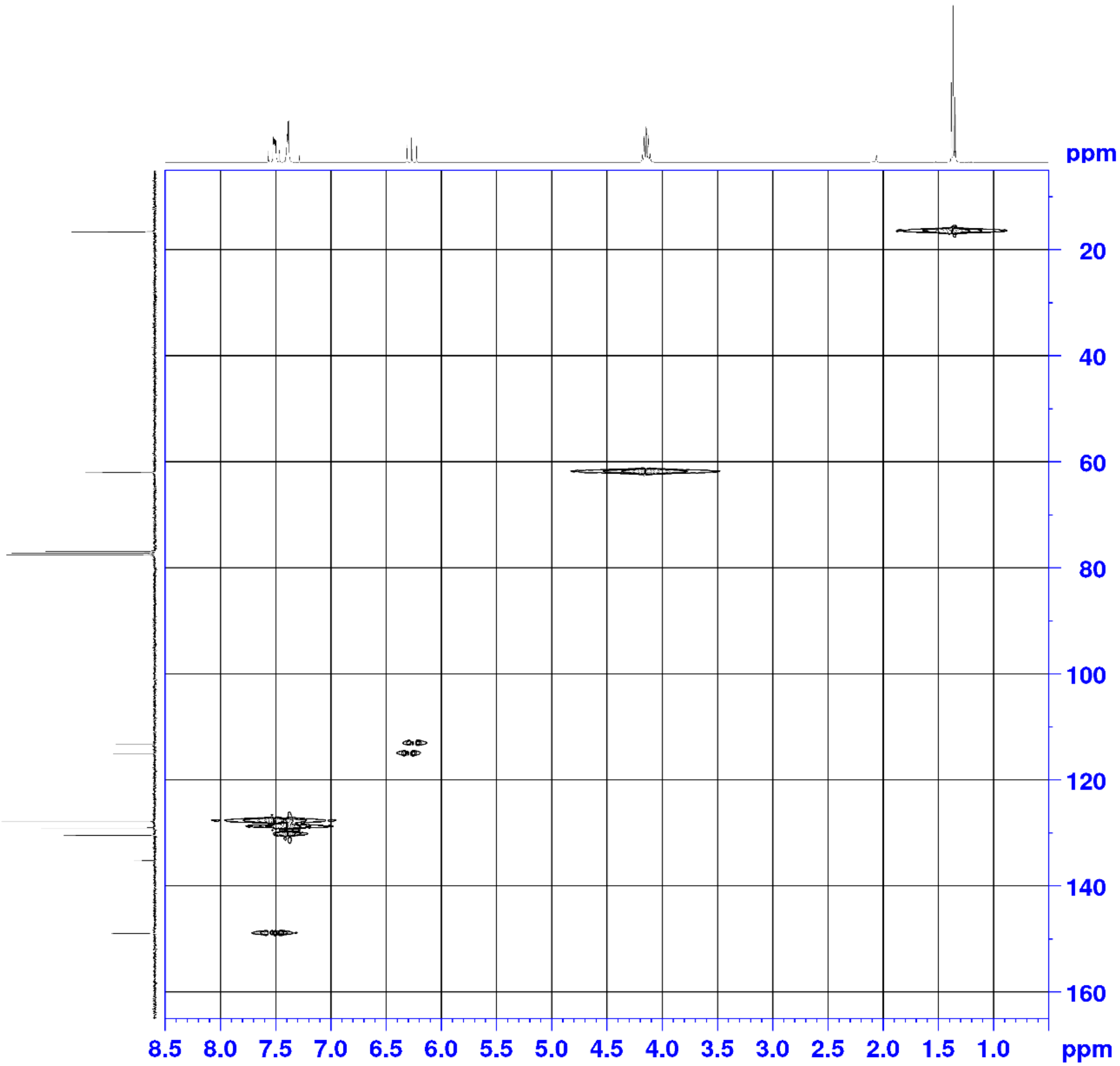
(*E*)-21: COSY

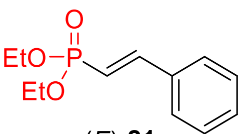

ppm
1
2
3
4
5
6
7
8
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0
ppm


O
EtO
P
EtO

(*E*)-**21**

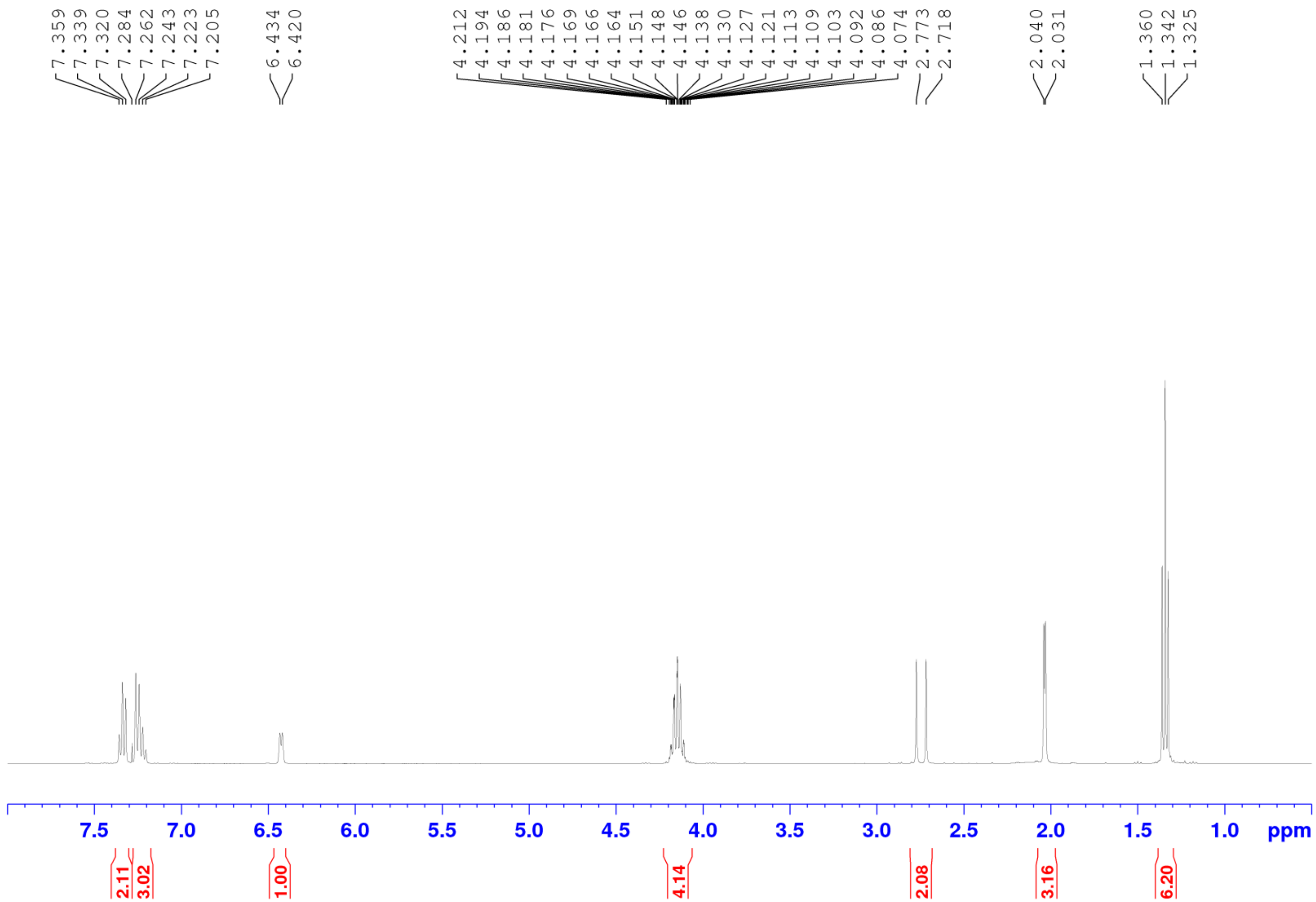
(*E*)-21: ^{13}C HSQC





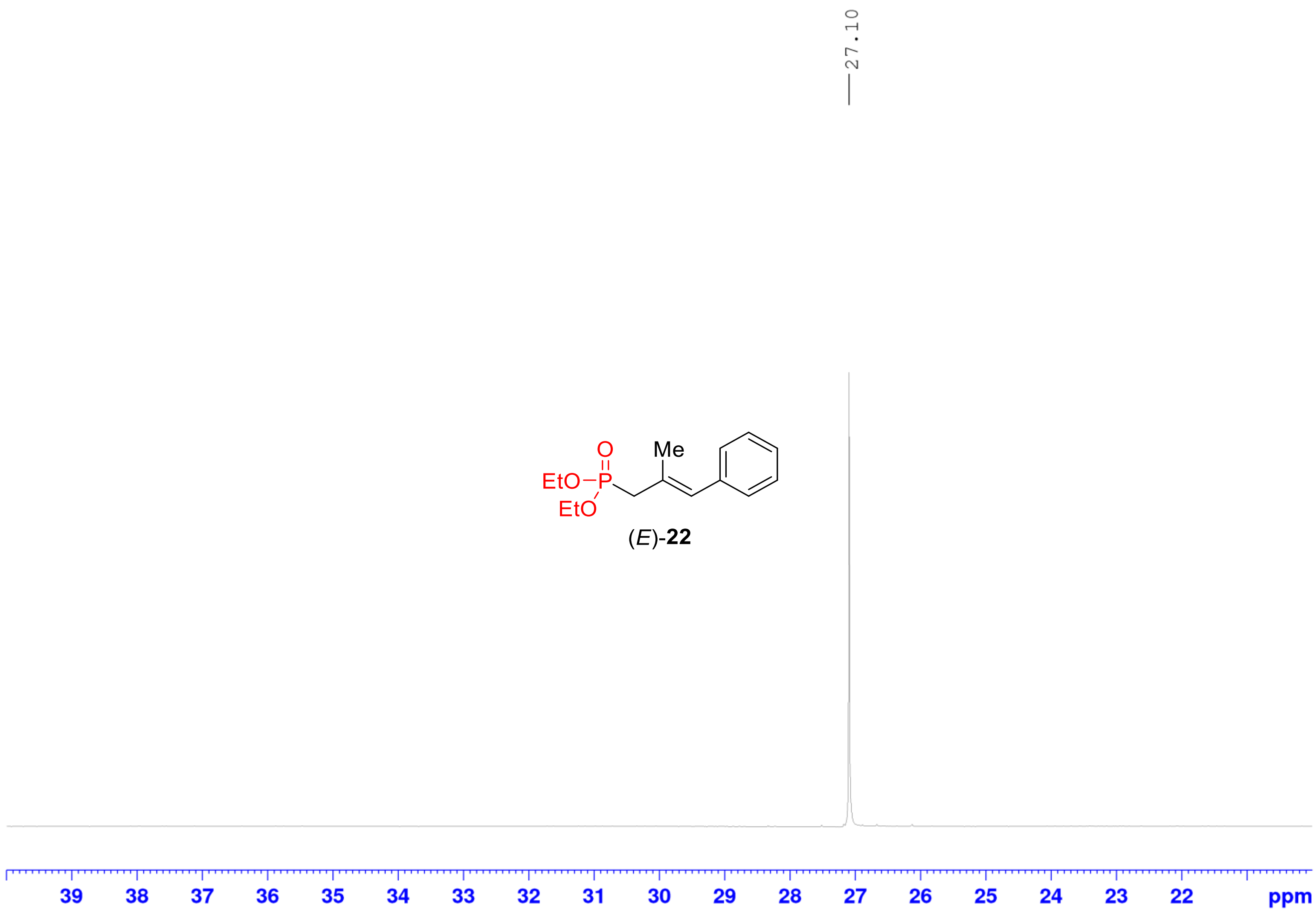
(*E*)-**21**

(*E*)-22: [1]H NMR



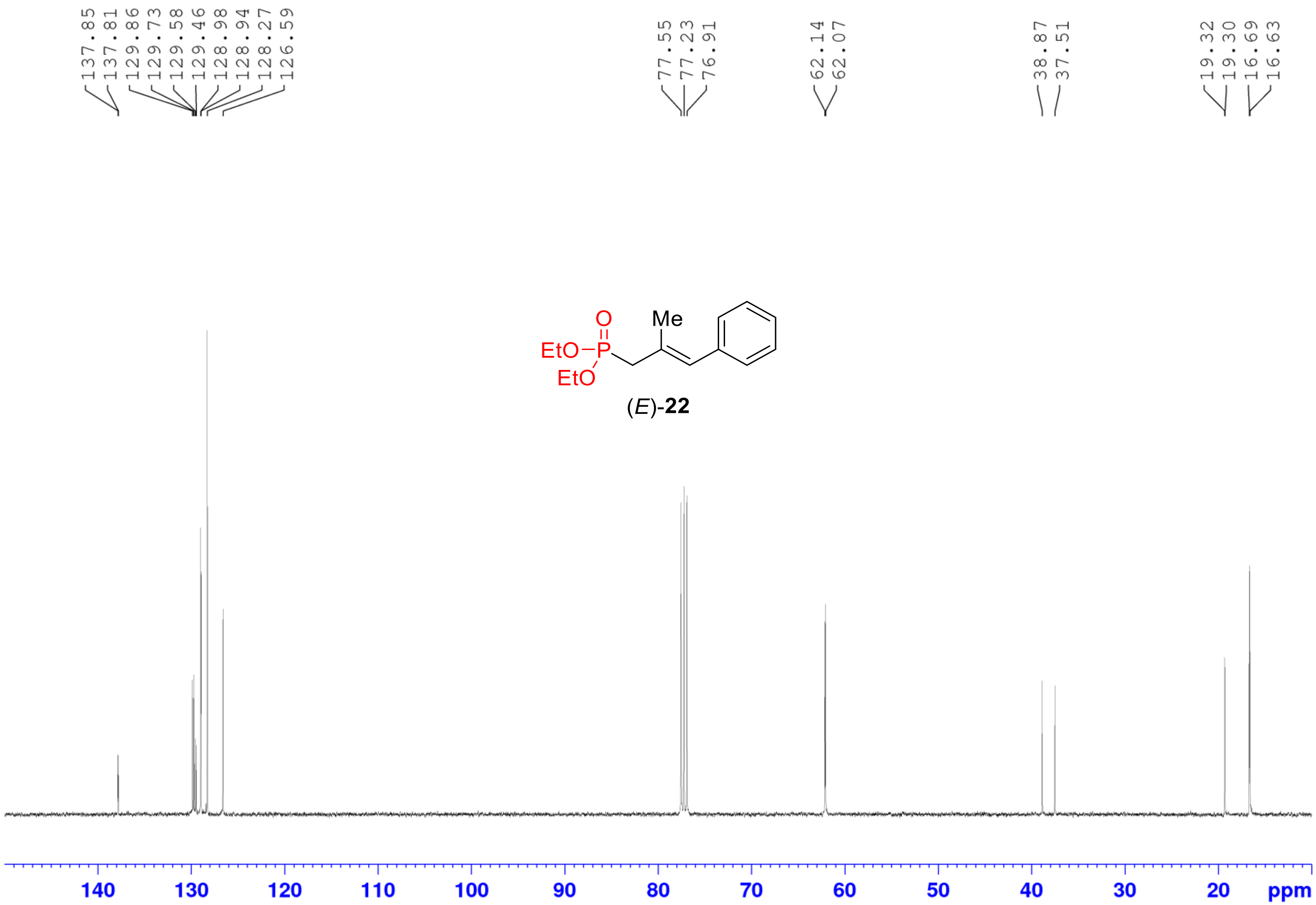
7.359
7.339
7.320
7.284
7.262
7.243
7.223
7.205
6.434
6.420
4.212
4.194
4.186
4.181
4.176
4.169
4.166
4.164
4.151
4.148
4.146
4.138
4.130
4.127
4.121
4.113
4.109
4.103
4.092
4.086
4.074
2.773
2.718
2.040
2.031
1.360
1.342
1.325
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
2.11
3.02
1.00
4.14
2.08
3.16
6.20

(*E*)-22: ^{31}P NMR

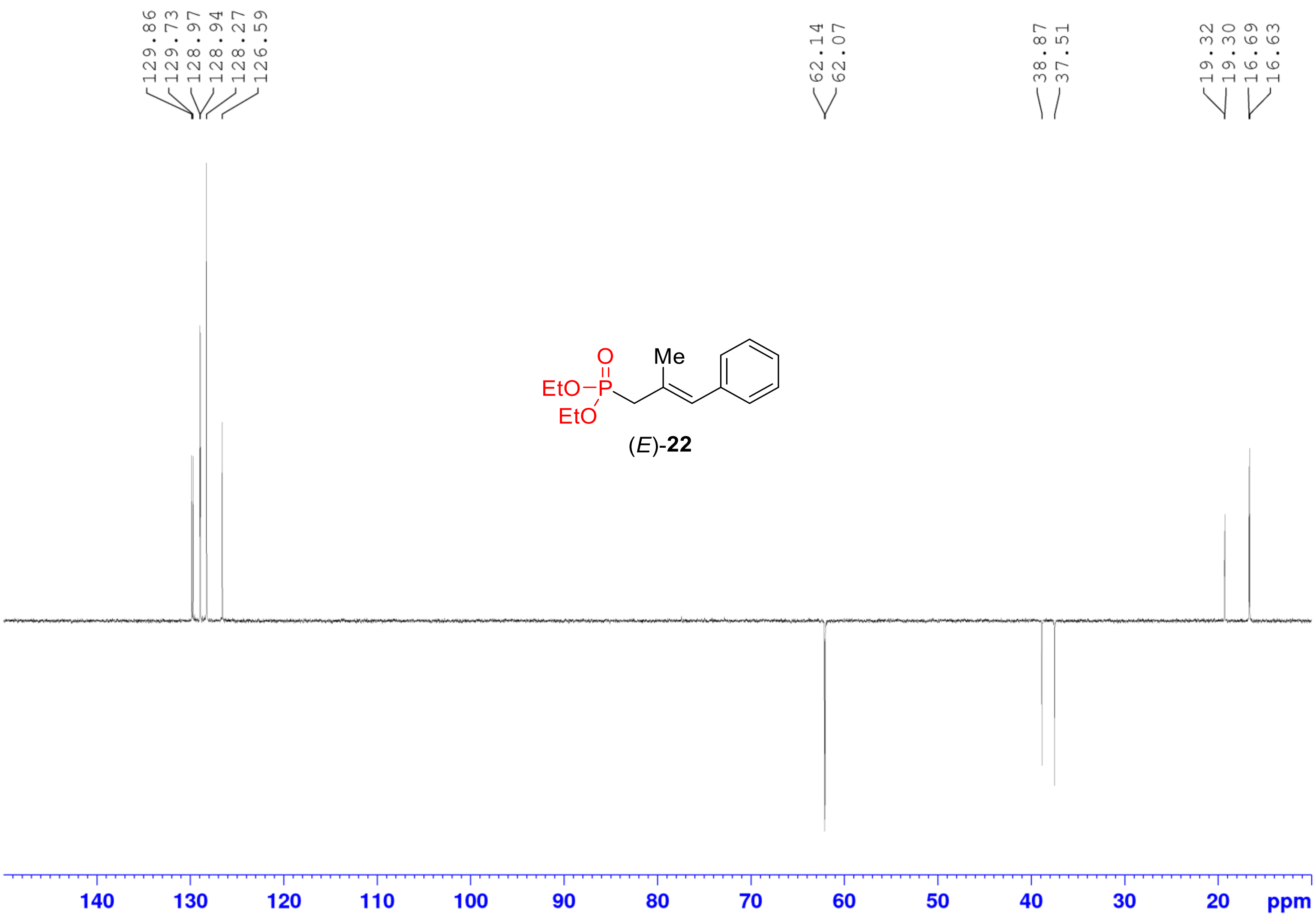


27.10
O
EtO
P
EtO
Me
(*E*)-**22**
39
38
37
36
35
34
33
32
31
30
29
28
27
26
25
24
23
22
ppm

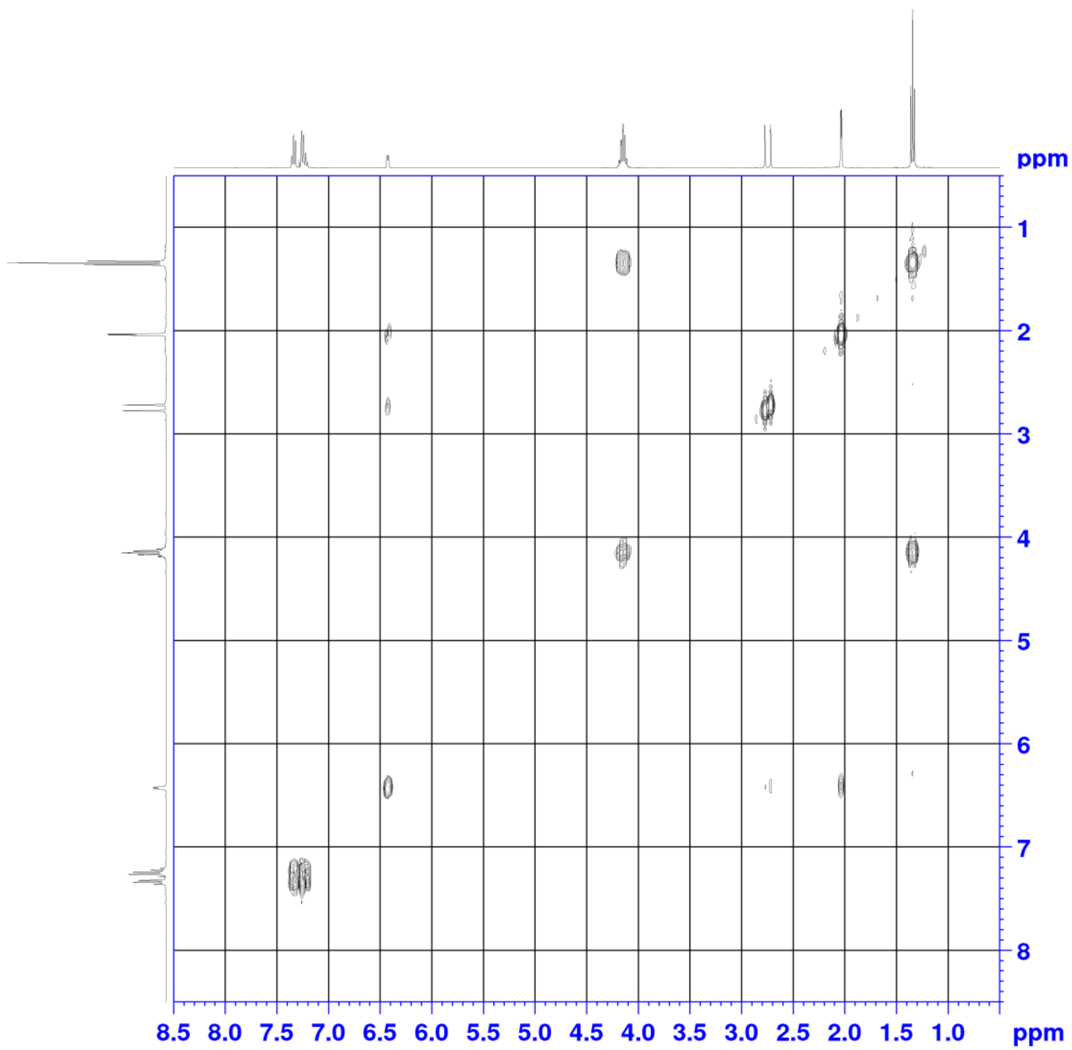
(*E*)-22: ^{13}C NMR

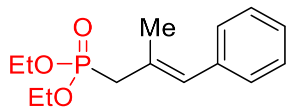

137.85
137.81
129.86
129.73
129.58
129.46
128.98
128.94
128.27
126.59
77.55
77.23
76.91
62.14
62.07
38.87
37.51
19.32
19.30
16.69
16.63
O
Me
EtO
P
EtO
(E)-22
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

(*E*)-22: ^{13}C DEPT135



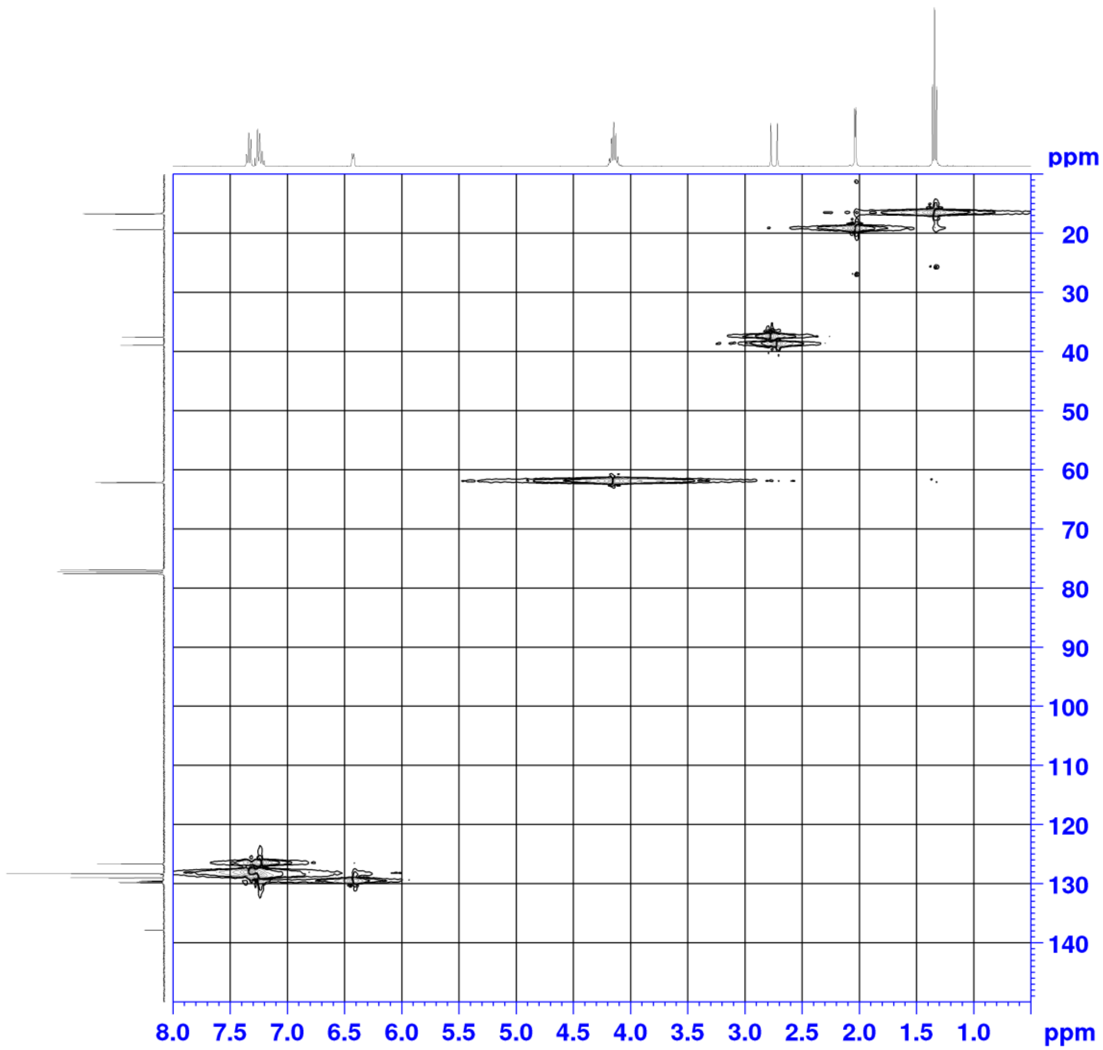
(*E*)-22: COSY

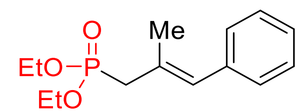

ppm
1
2
3
4
5
6
7
8
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0
ppm


O
Me
EtO
P
EtO

(*E*)-**22**

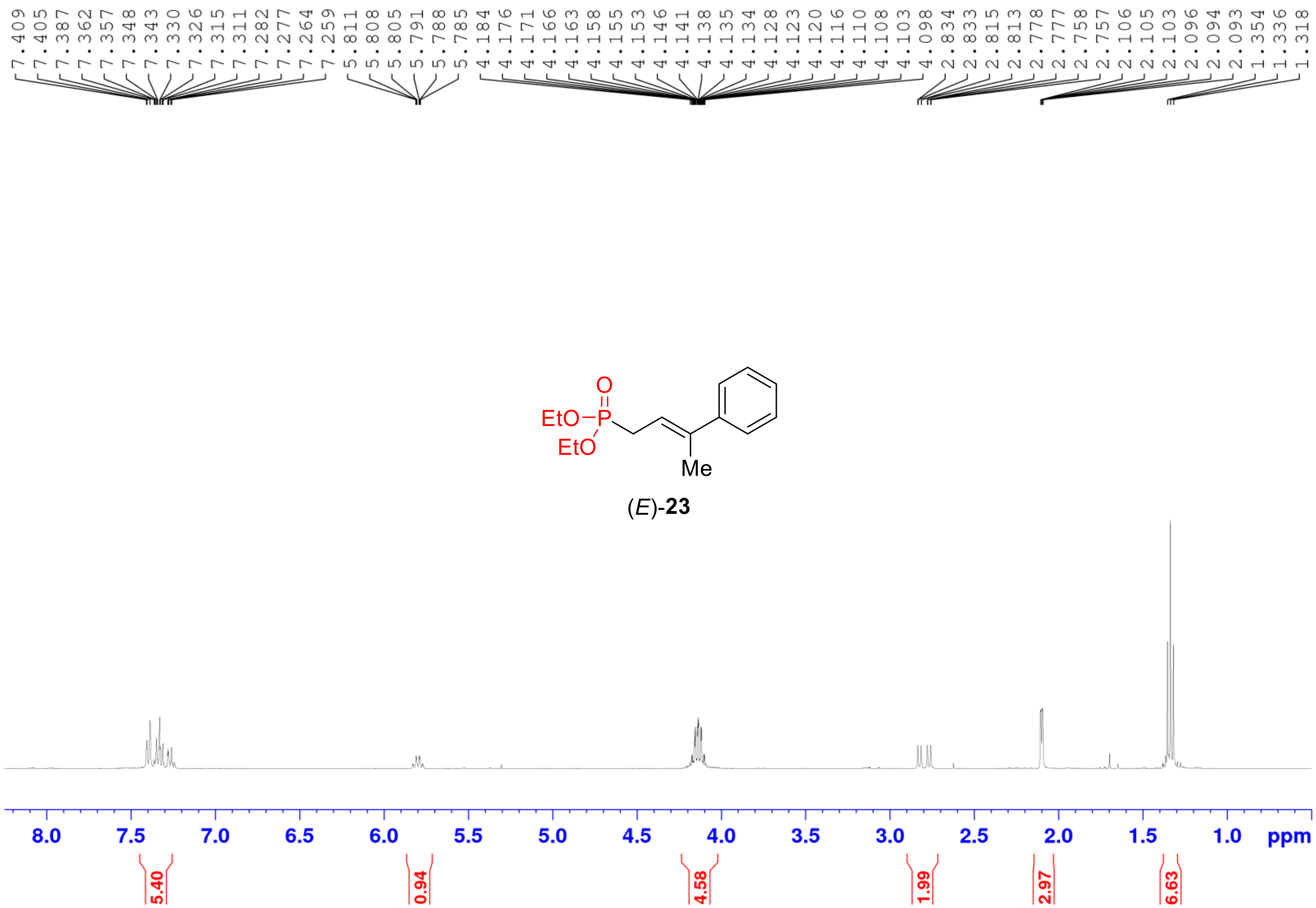
(*E*)-22: ^{13}C HSQC



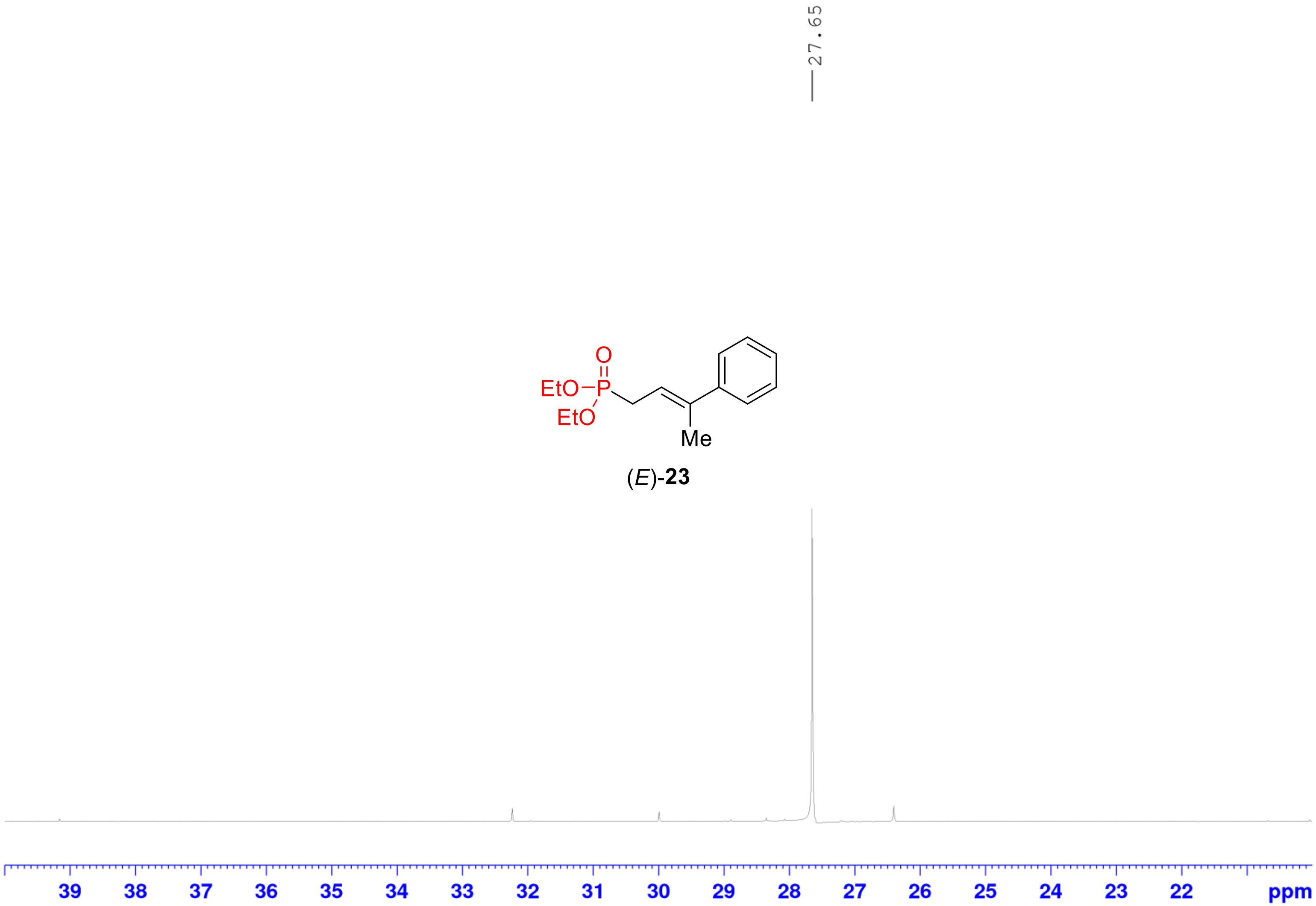


(*E*)-**22**

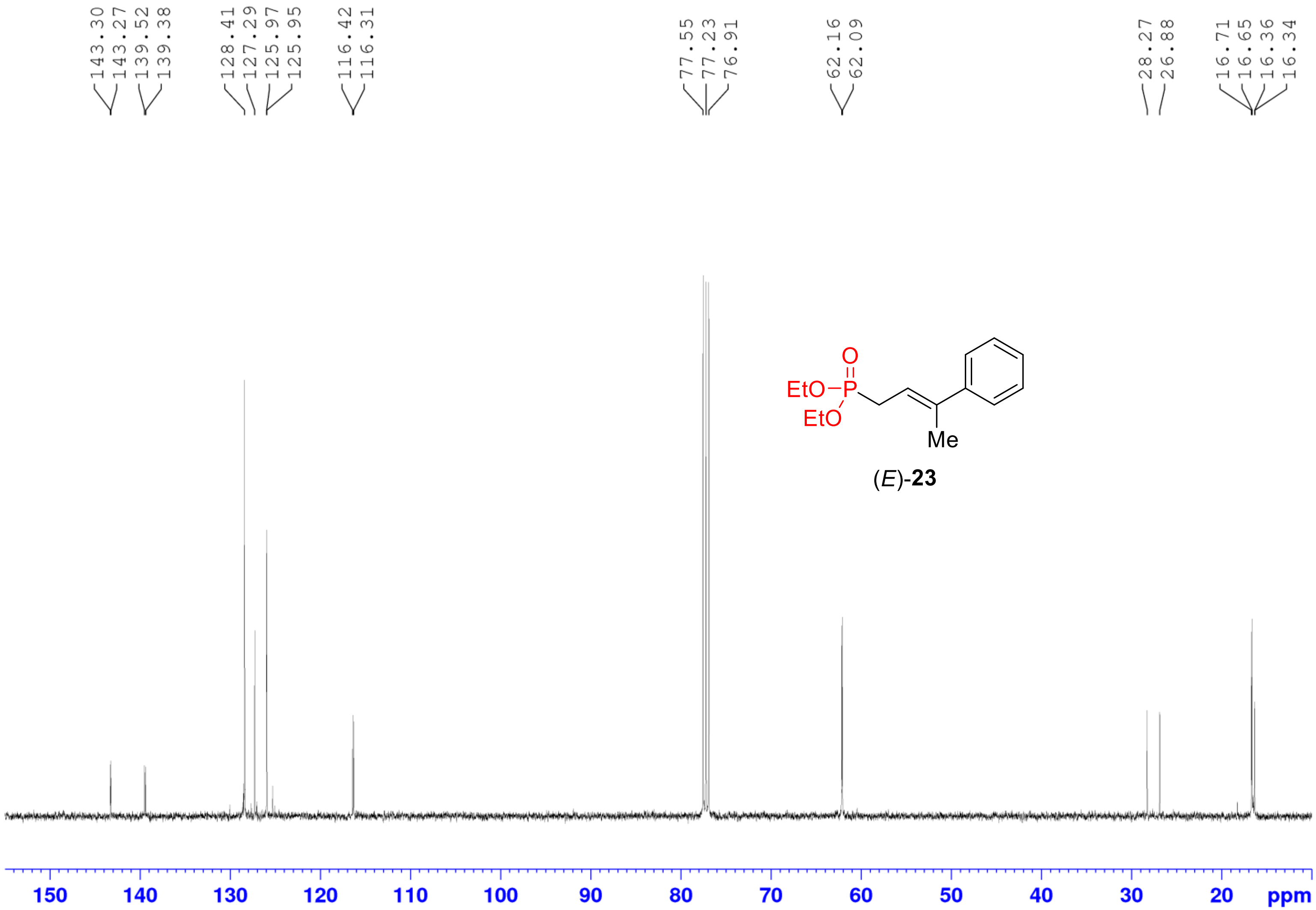
(*E*)-23: ^{1}H NMR


7.409 7.405 7.387 7.362 7.357 7.348 7.343 7.330 7.326 7.315 7.311 7.282 7.277 7.264 7.259
5.811 5.808 5.805 5.791 5.788 5.785
4.184 4.176 4.171 4.166 4.163 4.158 4.155 4.153 4.146 4.141 4.138 4.135 4.134 4.128 4.123 4.120 4.116 4.110 4.108 4.103 4.098
2.834 2.833 2.815 2.813 2.778 2.777 2.758 2.757
2.106 2.105 2.103 2.096 2.094 2.093
1.354 1.336 1.318
O
EtO
P
EtO
Me
(E)-23
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 ppm
5.40
0.94
4.58
1.99
2.97
6.63

(*E*)-23: ^{31}P NMR

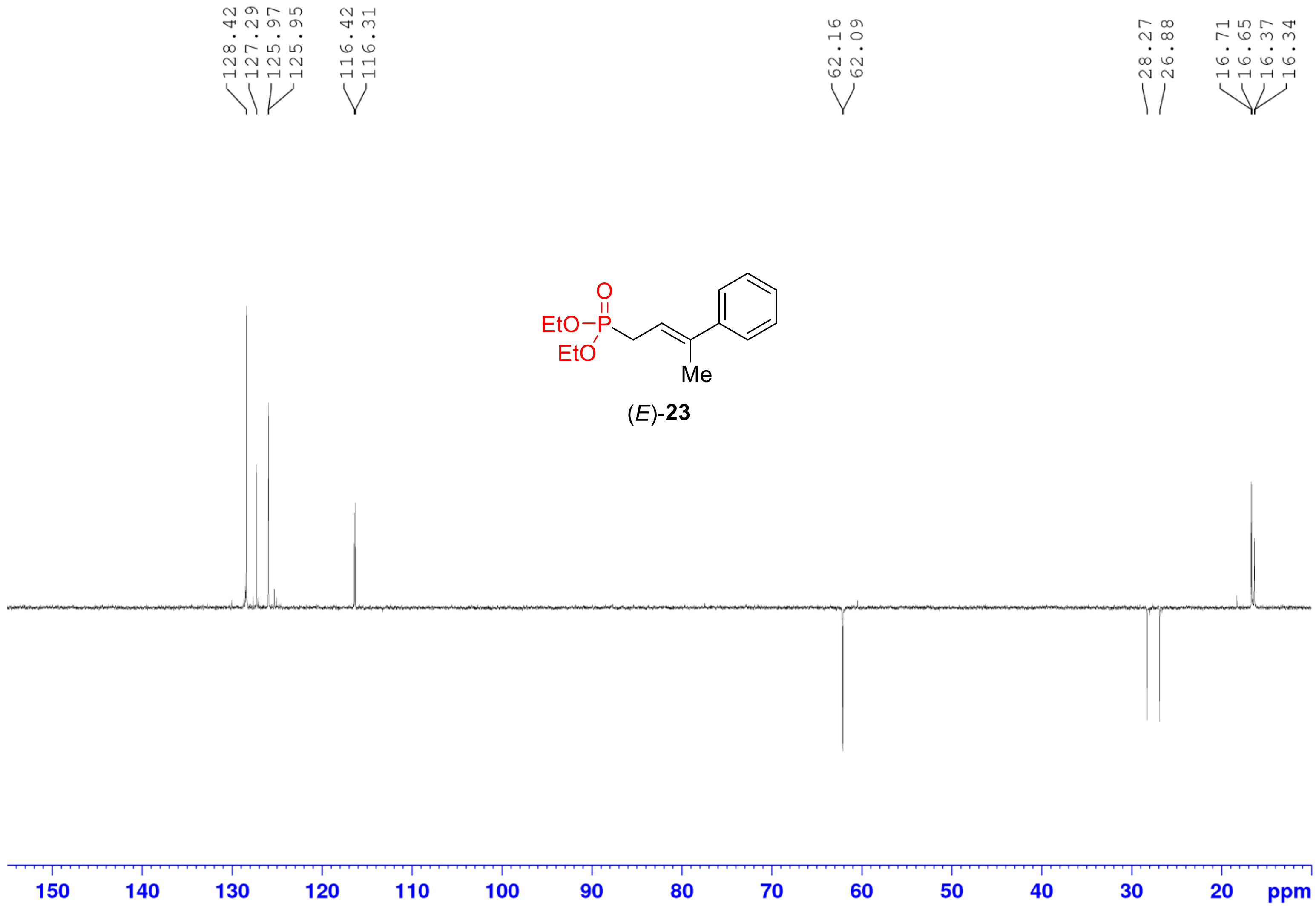

27.65
O
EtO–P
EtO
Me
(E)-23
39 38 37 36 35 34 33 32 31 30 29 28 27 26 25 24 23 22 ppm

(*E*)-23: ¹³C NMR

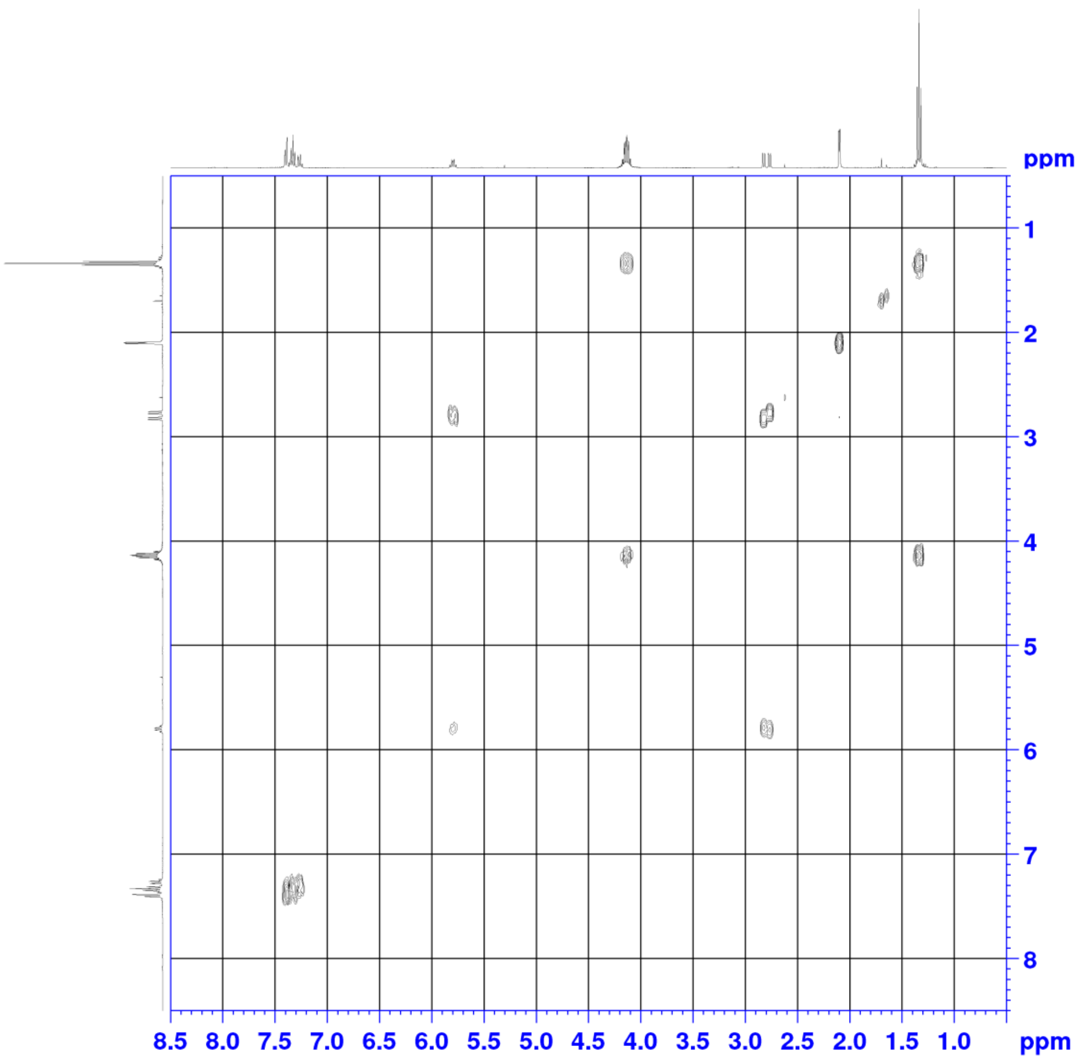


143.30
143.27
139.52
139.38
128.41
127.29
125.97
125.95
116.42
116.31
77.55
77.23
76.91
62.16
62.09
28.27
26.88
16.71
16.65
16.36
16.34
O
EtO
P
EtO
Me
(E)-23
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

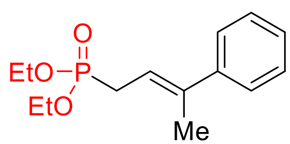
(*E*)-23: ^{13}C DEPT135



(*E*)-23: COSY



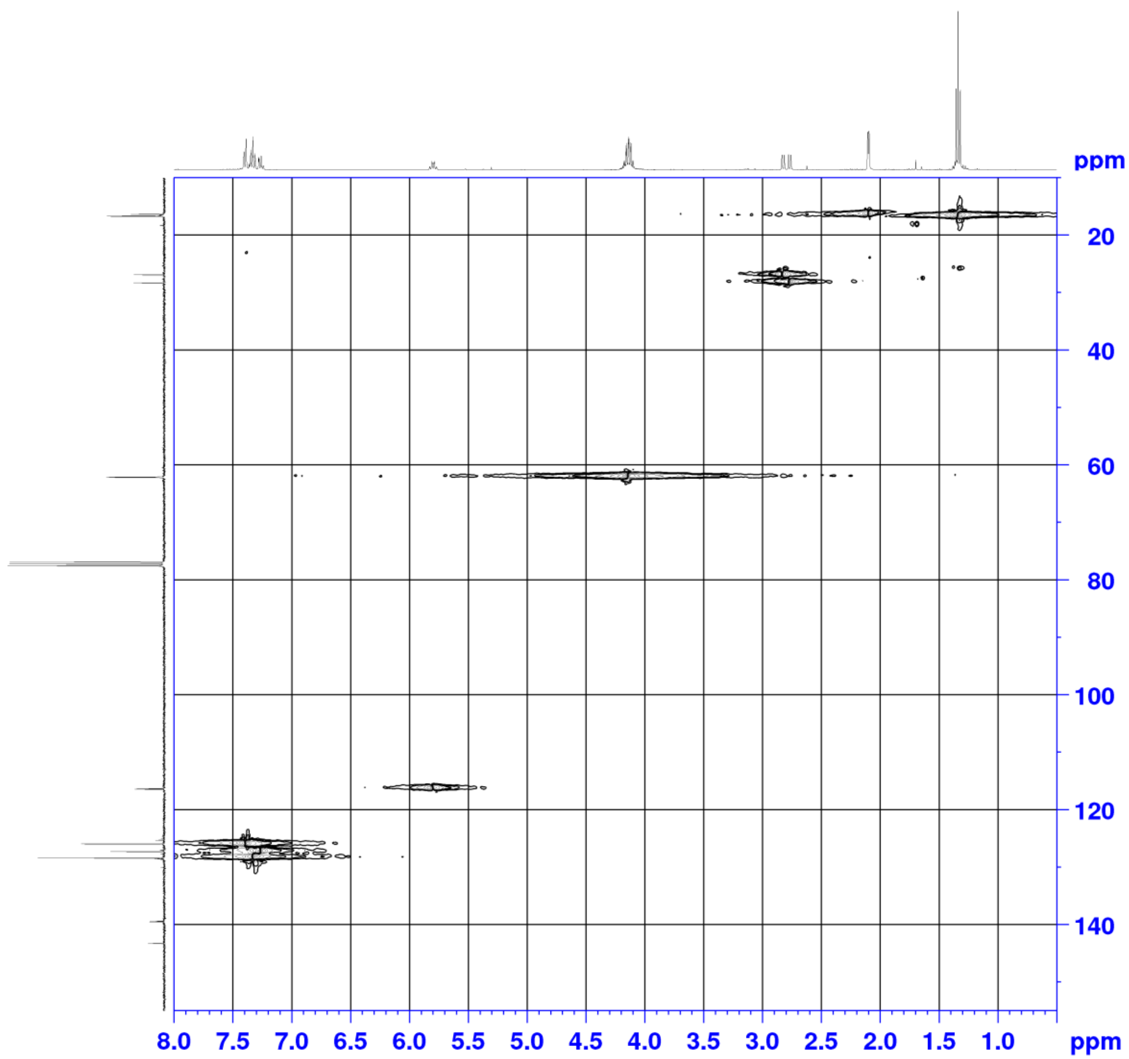
ppm
1
2
3
4
5
6
7
8
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

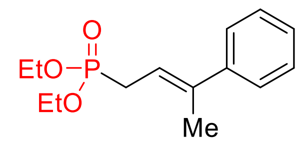


O
EtO
P
EtO
Me

(*E*)-**23**

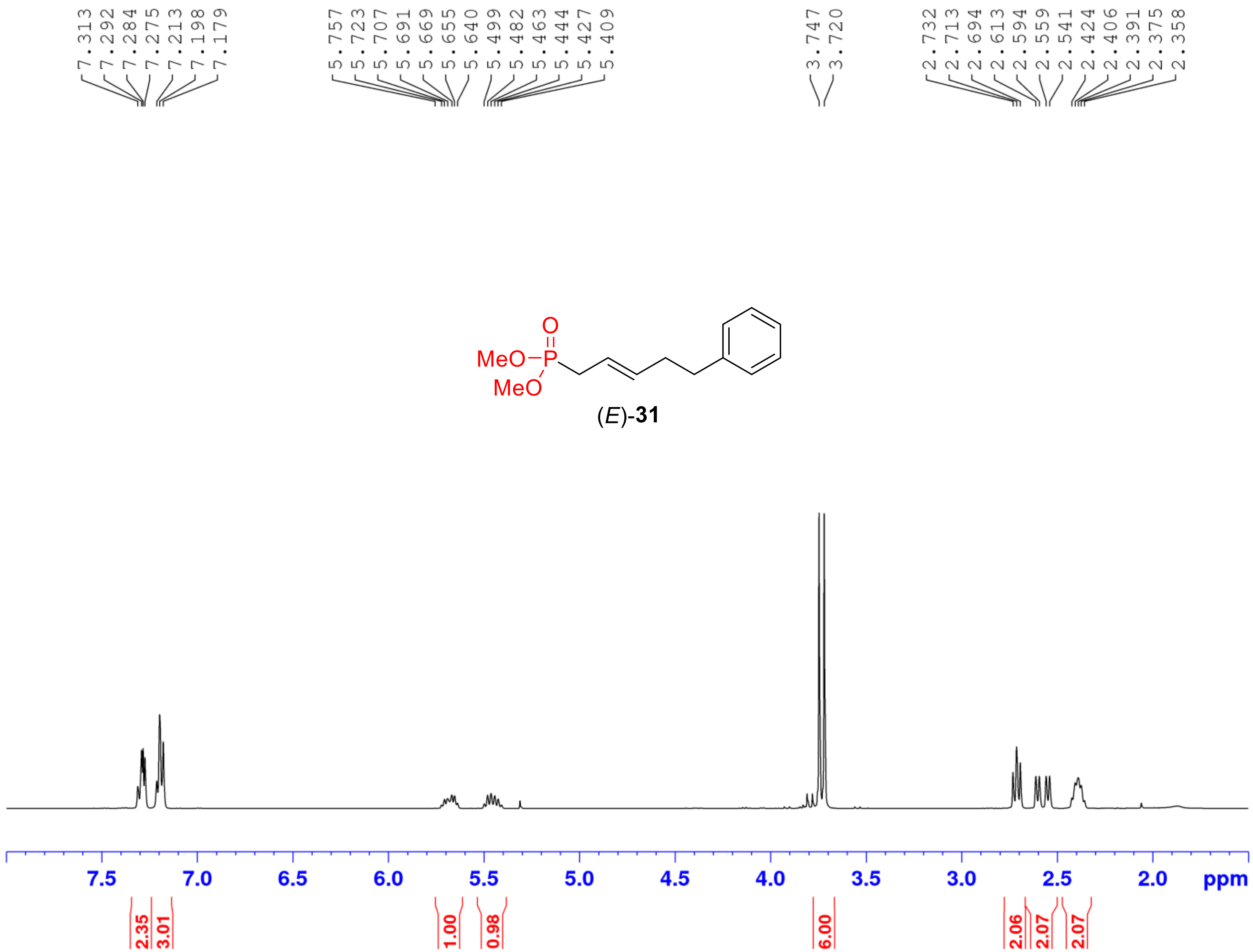
(*E*)-23: ^{13}C HSQC



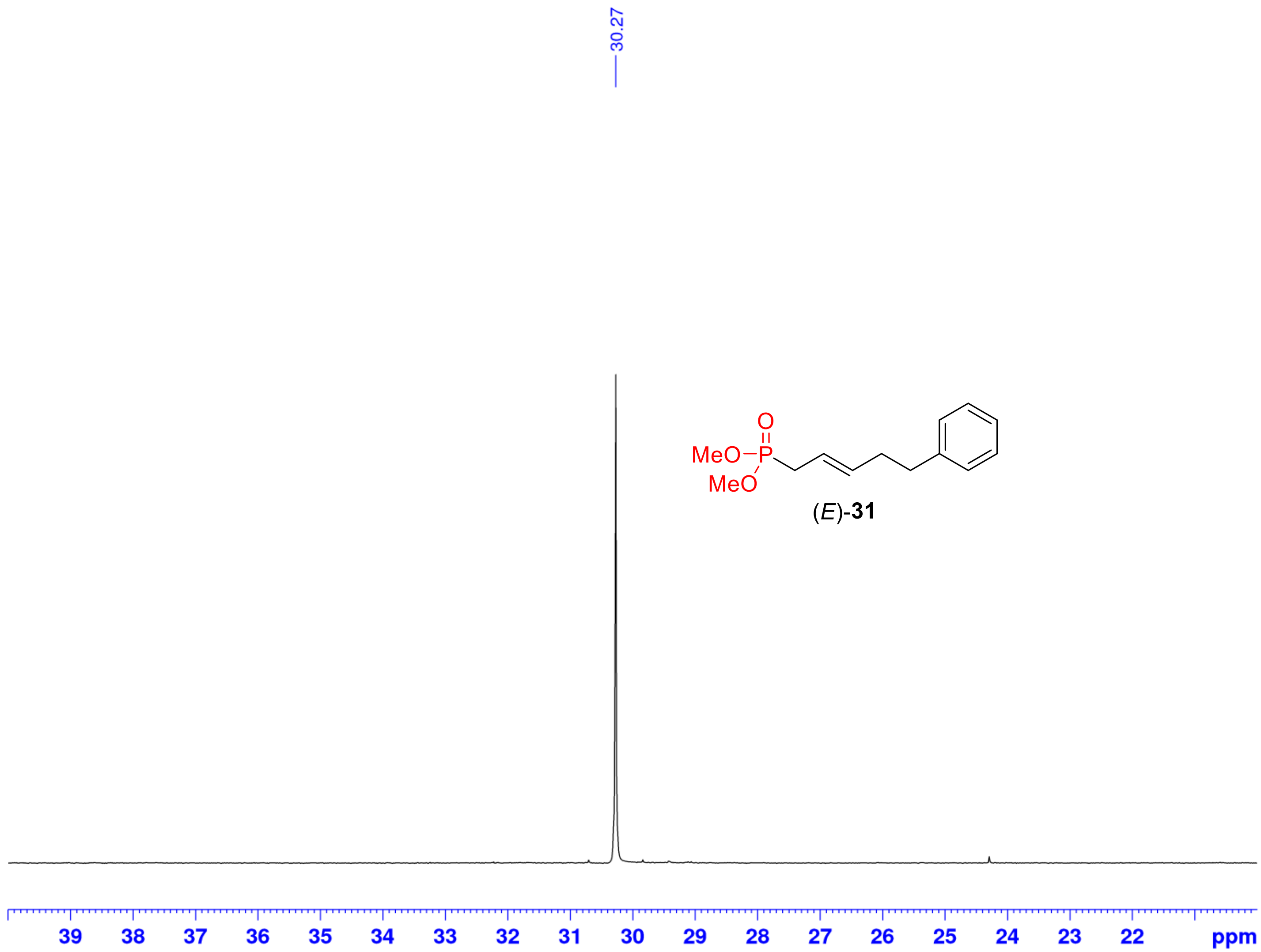


(*E*)-**23**

(*E*)-31: ^{1}H NMR

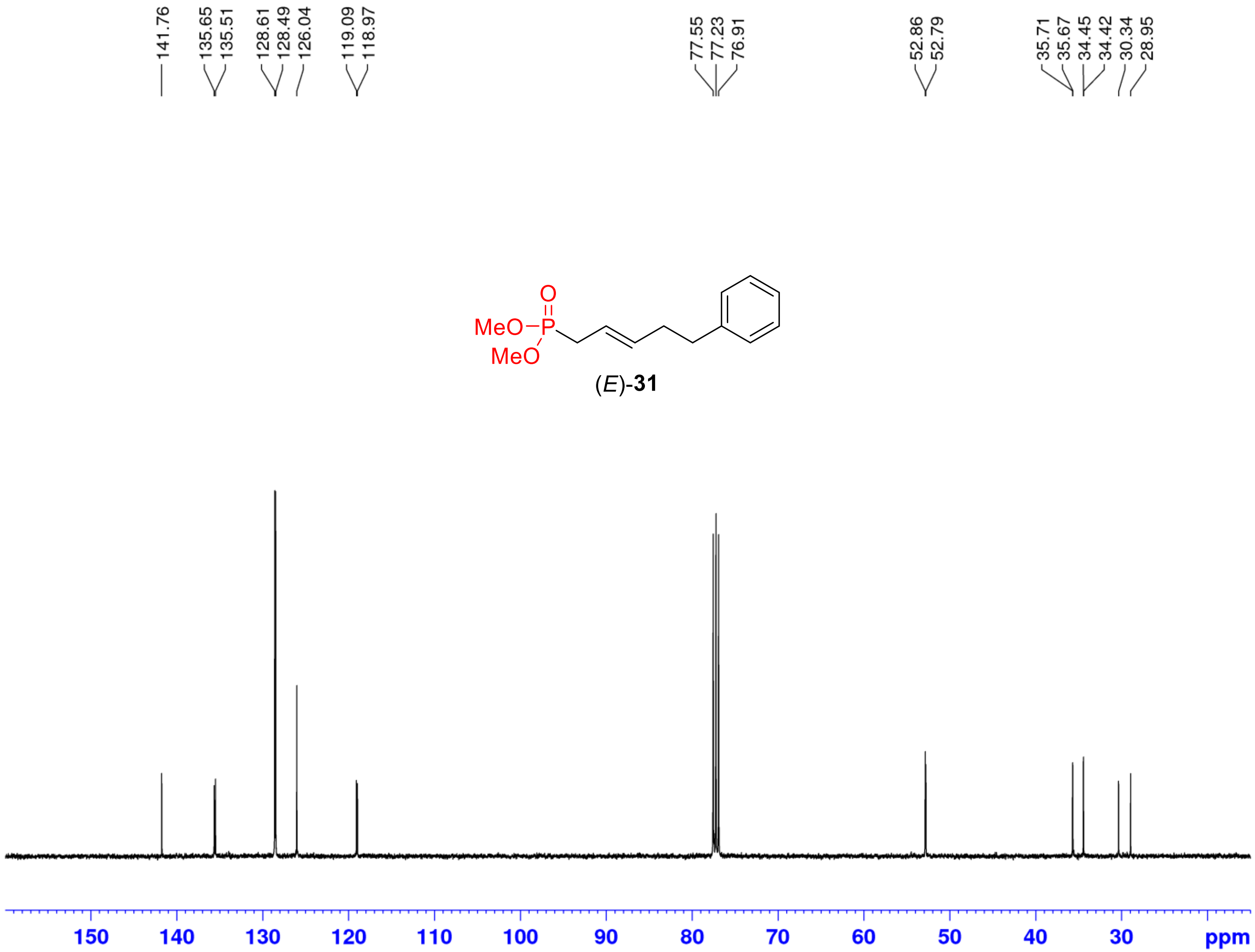

7.313
7.292
7.284
7.275
7.213
7.198
7.179
5.757
5.723
5.707
5.691
5.669
5.655
5.640
5.499
5.482
5.463
5.444
5.427
5.409
3.747
3.720
2.732
2.713
2.694
2.613
2.594
2.559
2.541
2.424
2.406
2.391
2.375
2.358
O
MeO
P
MeO
(*E*)-**31**
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
ppm
2.35
3.01
1.00
0.98
6.00
2.06
2.07
2.07

(*E*)-31: ^{31}P NMR



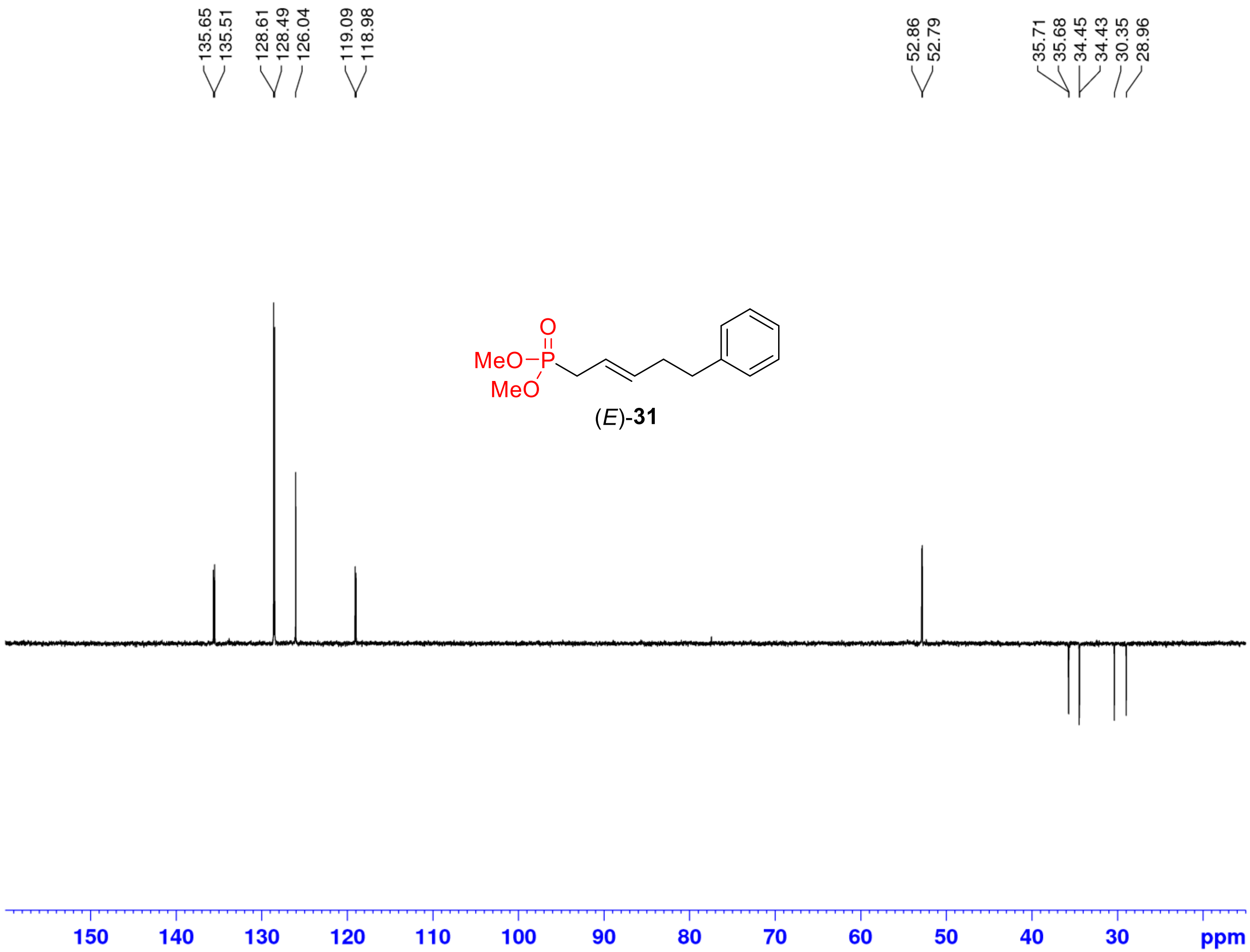
30.27
MeO
MeO
O
P
(E)-31
39
38
37
36
35
34
33
32
31
30
29
28
27
26
25
24
23
22
ppm

(*E*)-31: ^{13}C NMR

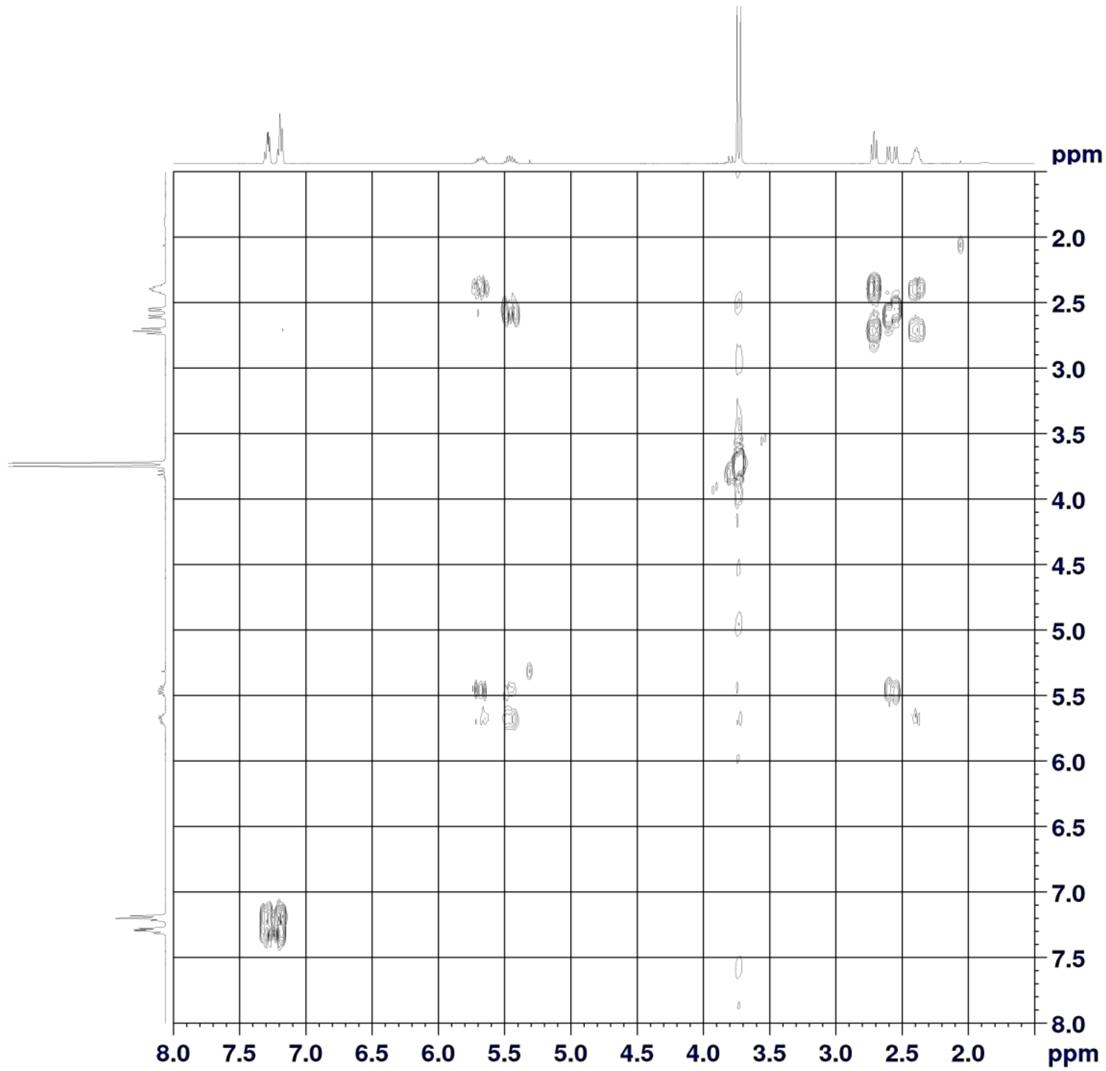


(E)-31
MeO
MeO
O
P
141.76
135.65
135.51
128.61
128.49
126.04
119.09
118.97
77.55
77.23
76.91
52.86
52.79
35.71
35.67
34.45
34.42
30.34
28.95
150
140
130
120
110
100
90
80
70
60
50
40
30
ppm

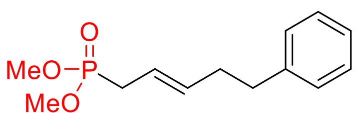
(*E*)-31: ^{13}C DEPT135



(*E*)-31: COSY



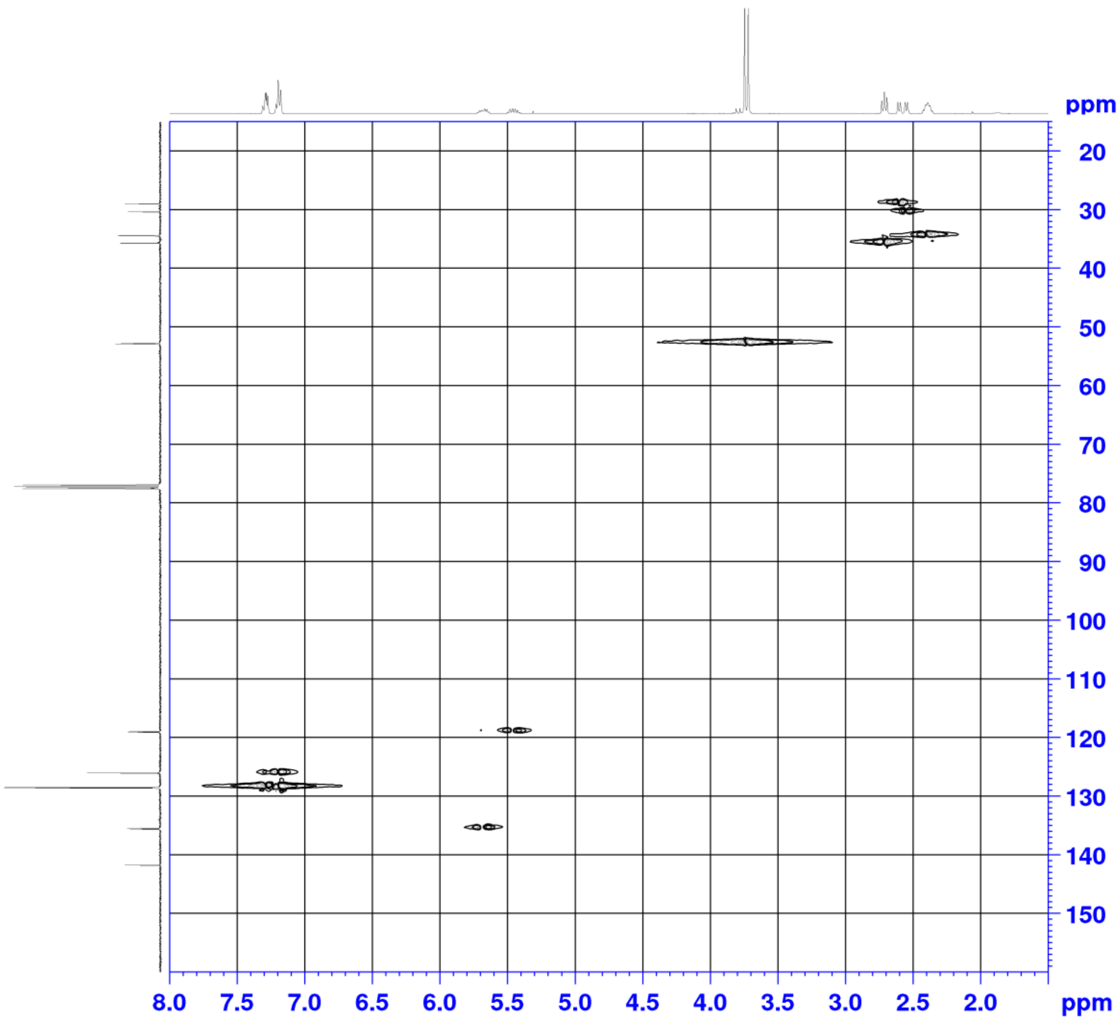
ppm
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
ppm

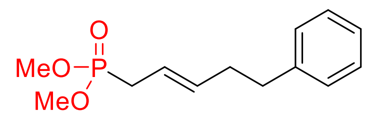


O
MeO
P
MeO

(*E*)-**31**

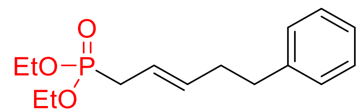
(*E*)-31: ^{13}C HSQC



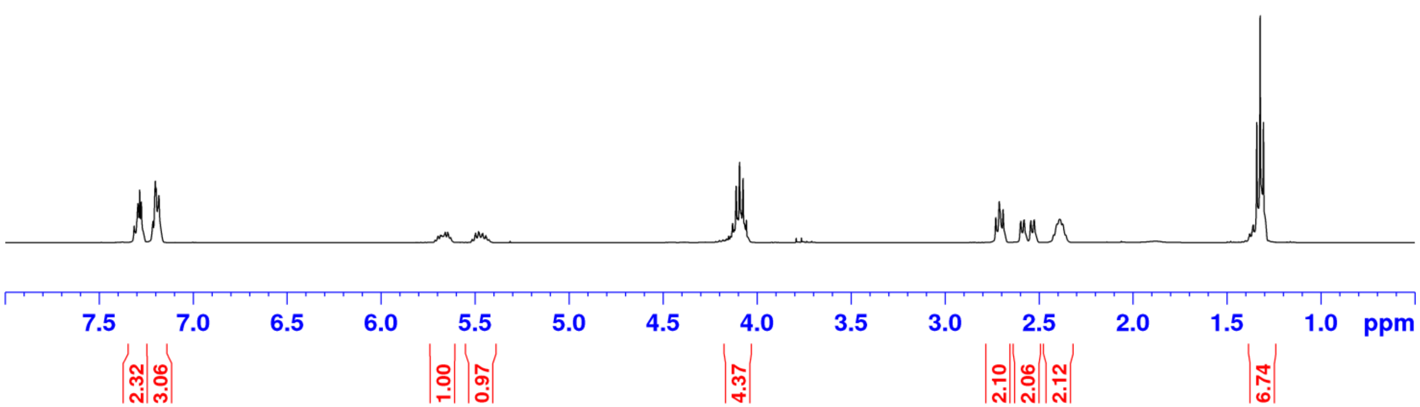


(*E*)-**31**

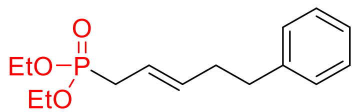
(*E*)-24: [1]H NMR



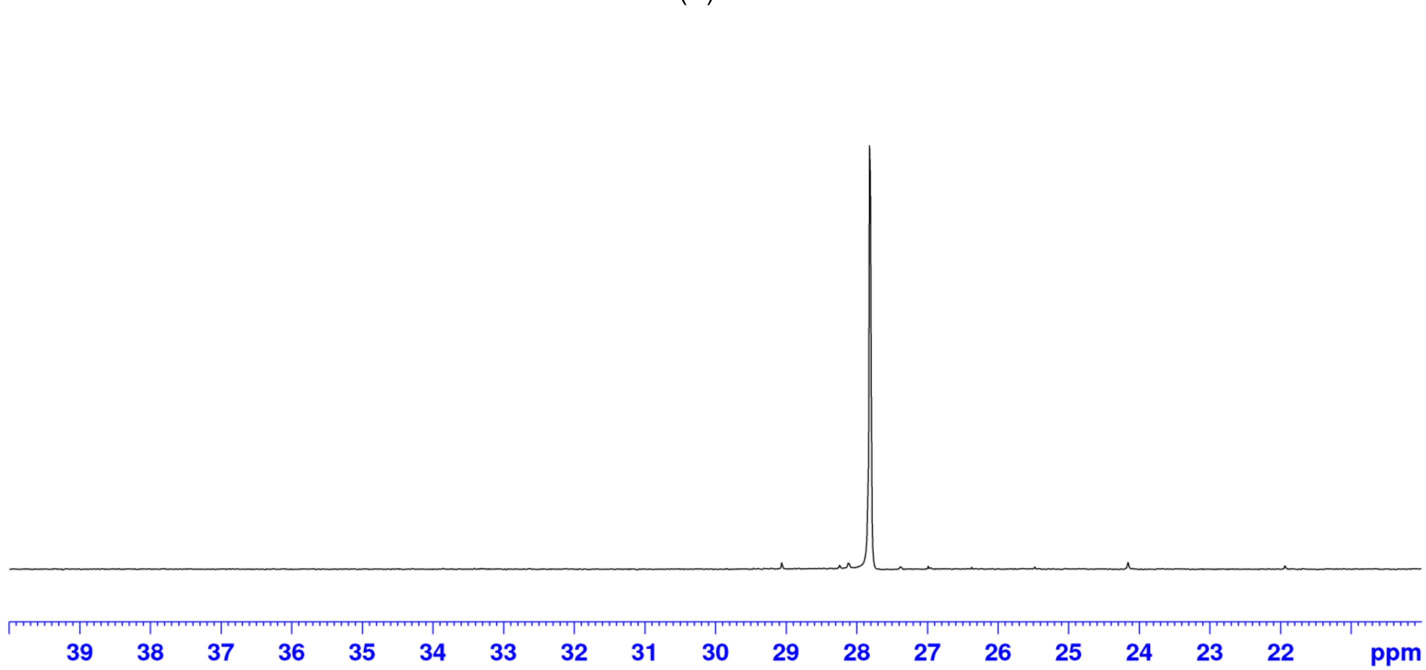
(*E*)-**24**



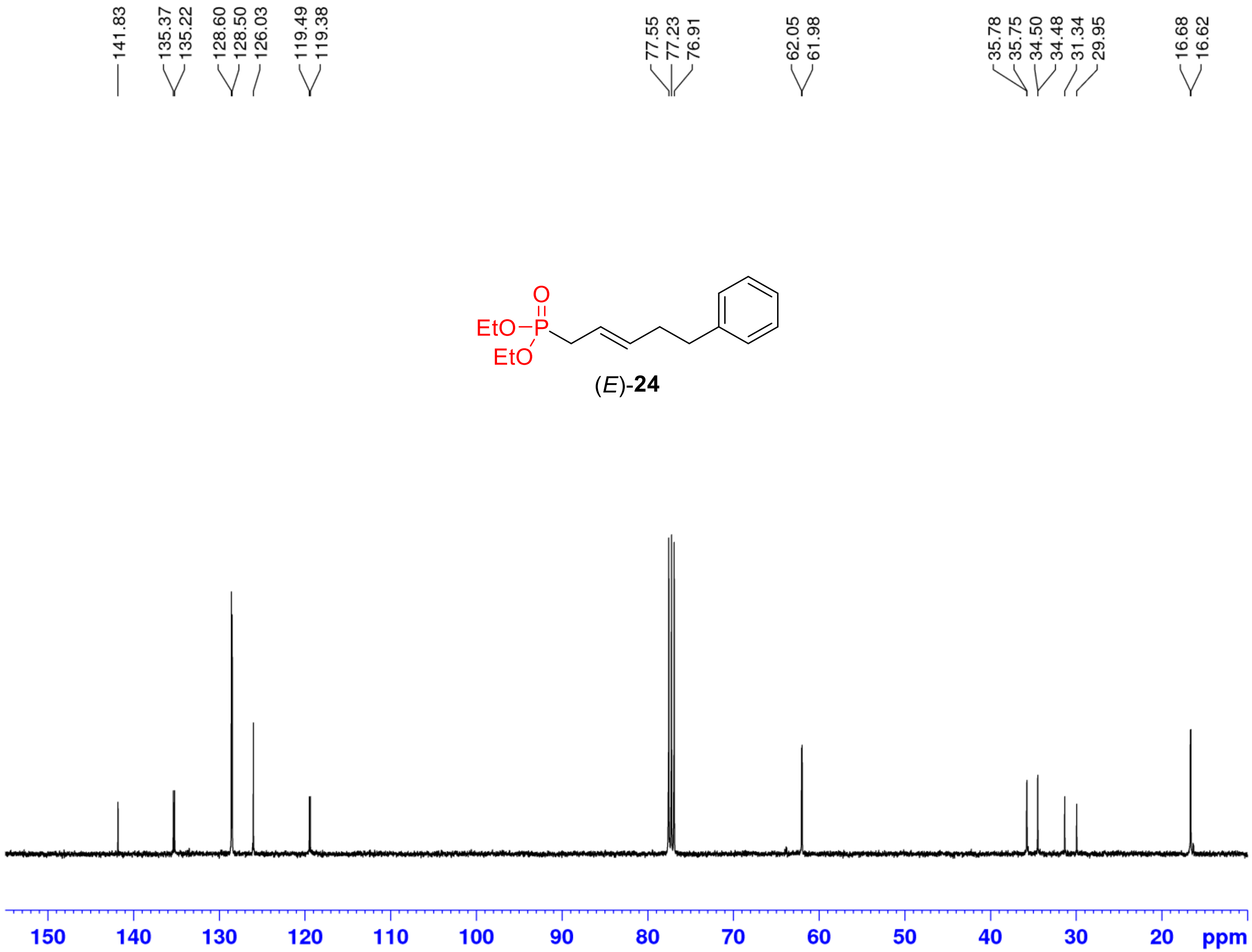
(*E*)-24: ^{31}P NMR



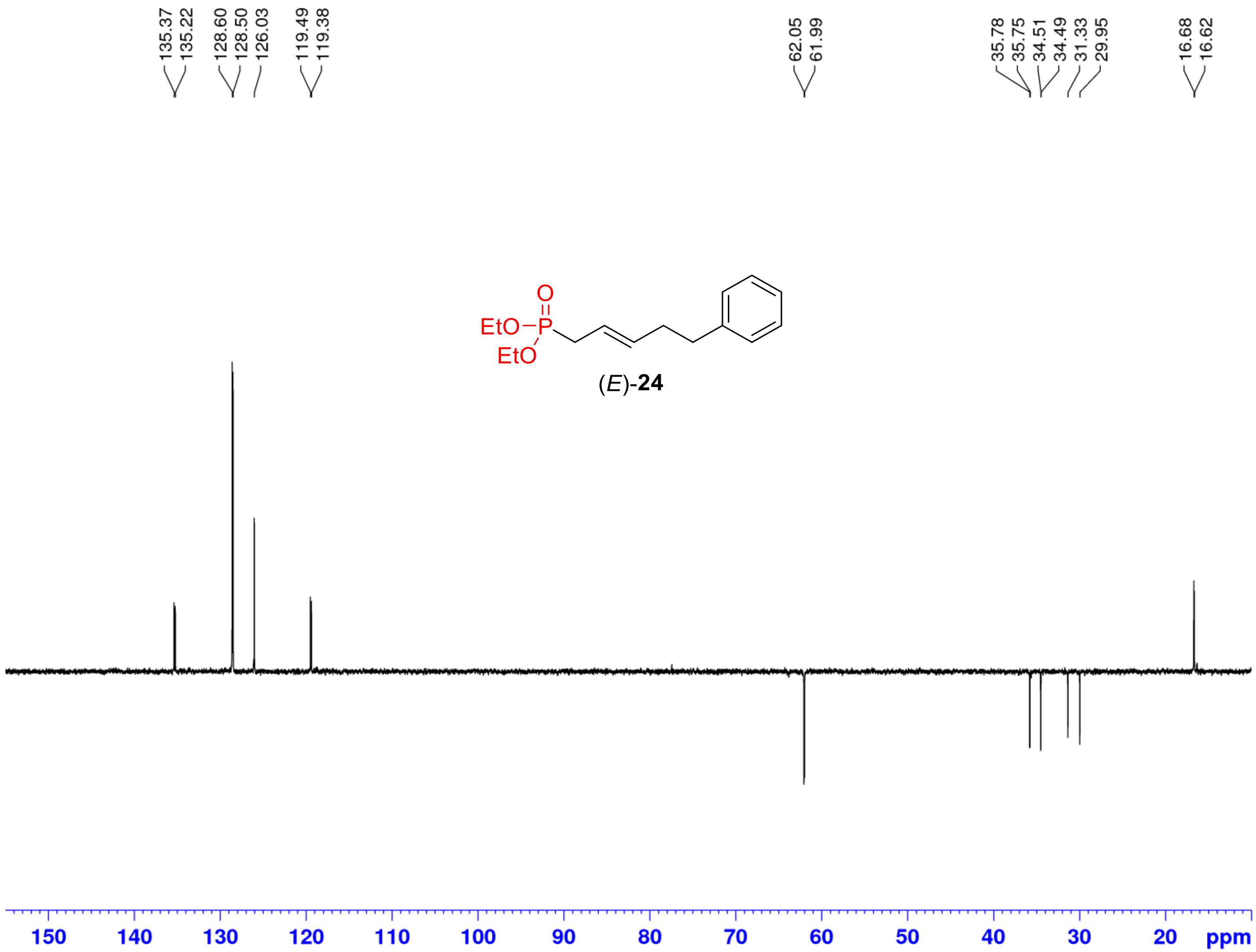
(*E*)-**24**



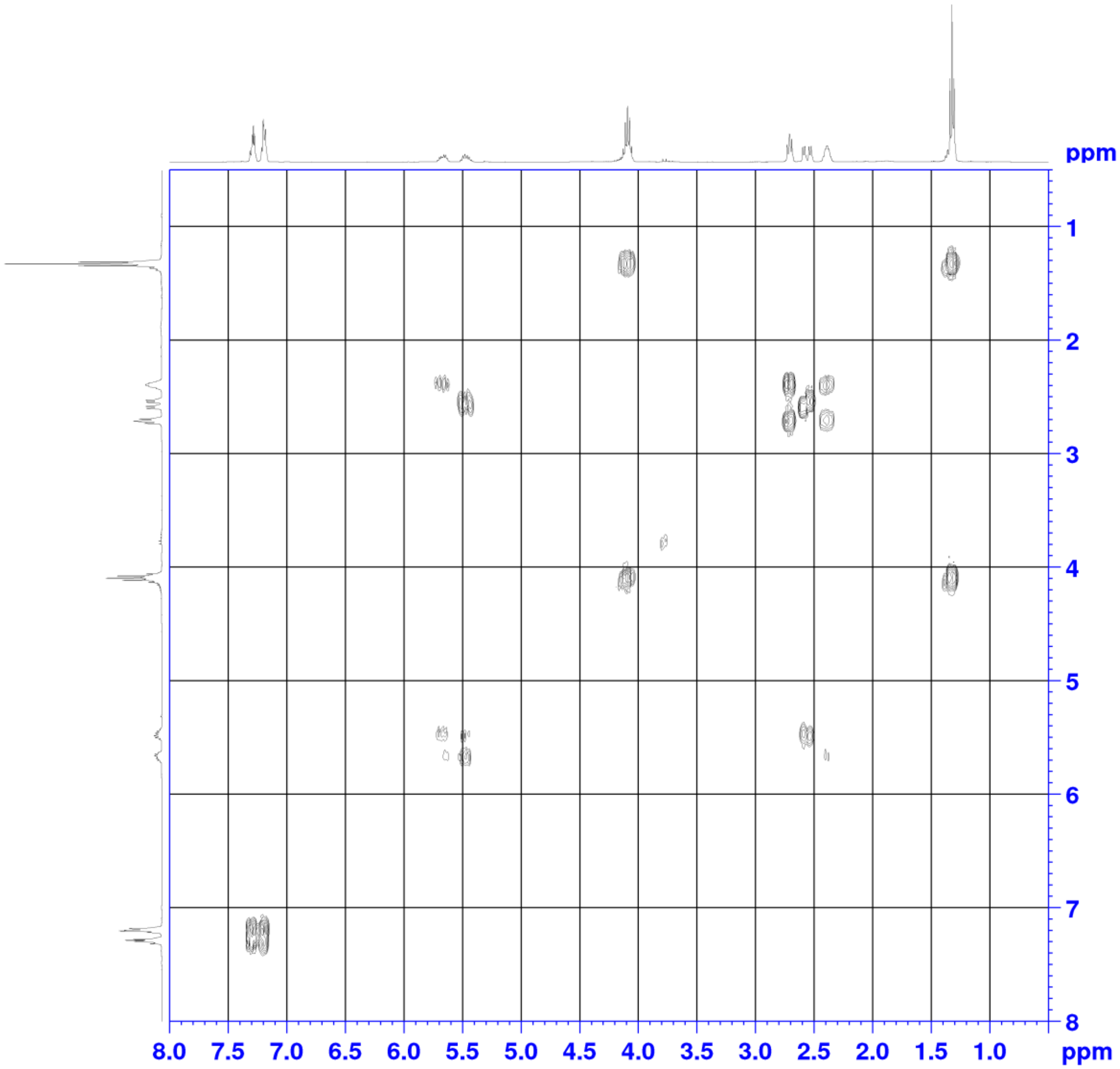
(*E*)-24: ^{13}C NMR

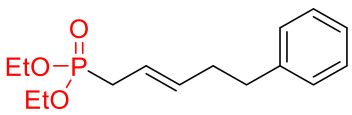

141.83
135.37
135.22
128.60
128.50
126.03
119.49
119.38
77.55
77.23
76.91
62.05
61.98
35.78
35.75
34.50
34.48
31.34
29.95
16.68
16.62
O
EtO
P
EtO
(E)-24
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

(*E*)-24: ^{13}C DEPT135



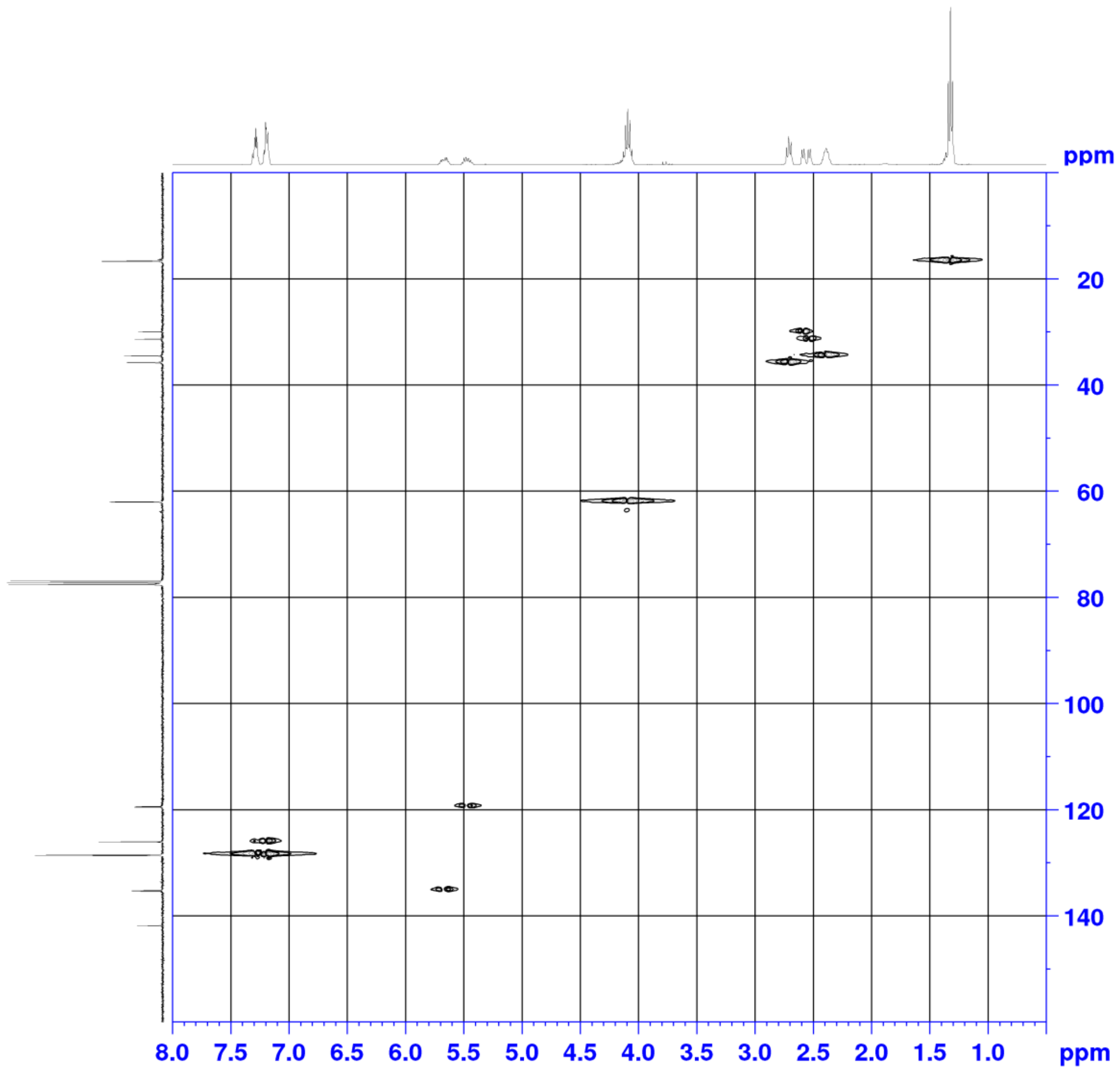
(*E*)-24: COSY

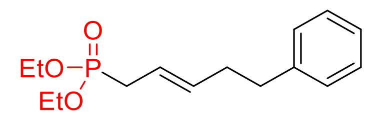

ppm
1
2
3
4
5
6
7
8
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0
ppm


O
EtO
P
EtO

(*E*)-**24**

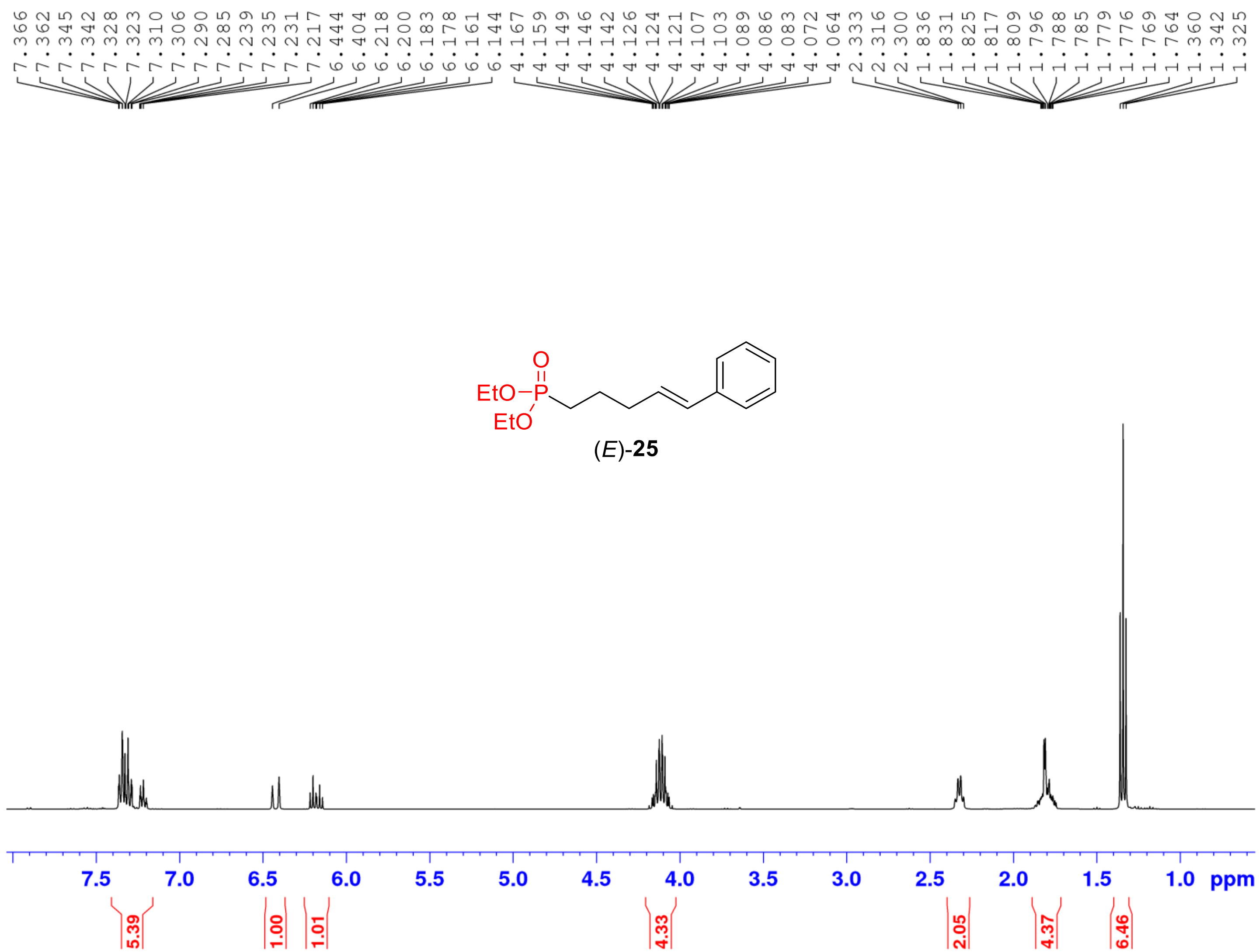
(*E*)-24: ^{13}C HSQC





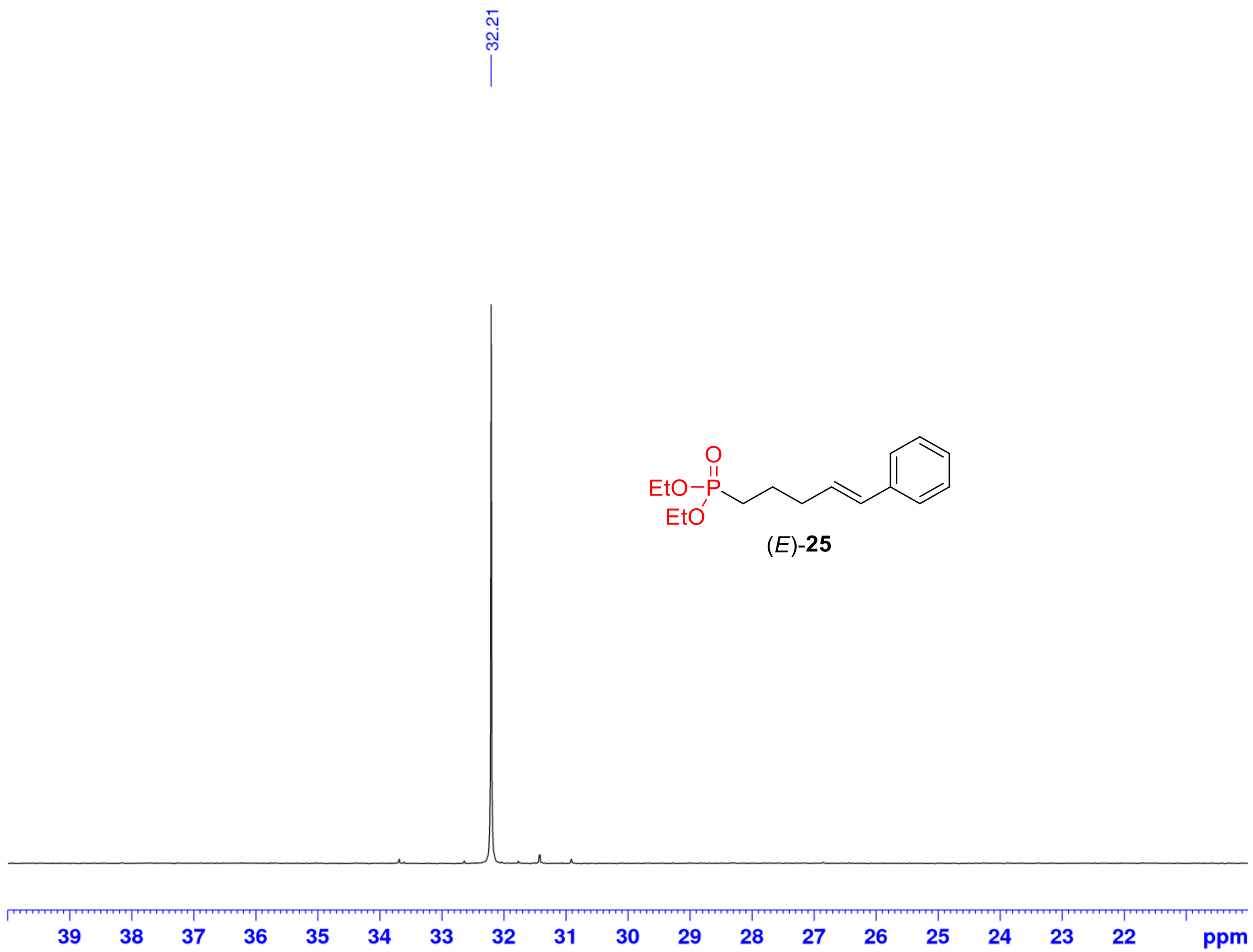
(*E*)-**24**

(*E*)-25: ^{1}H NMR

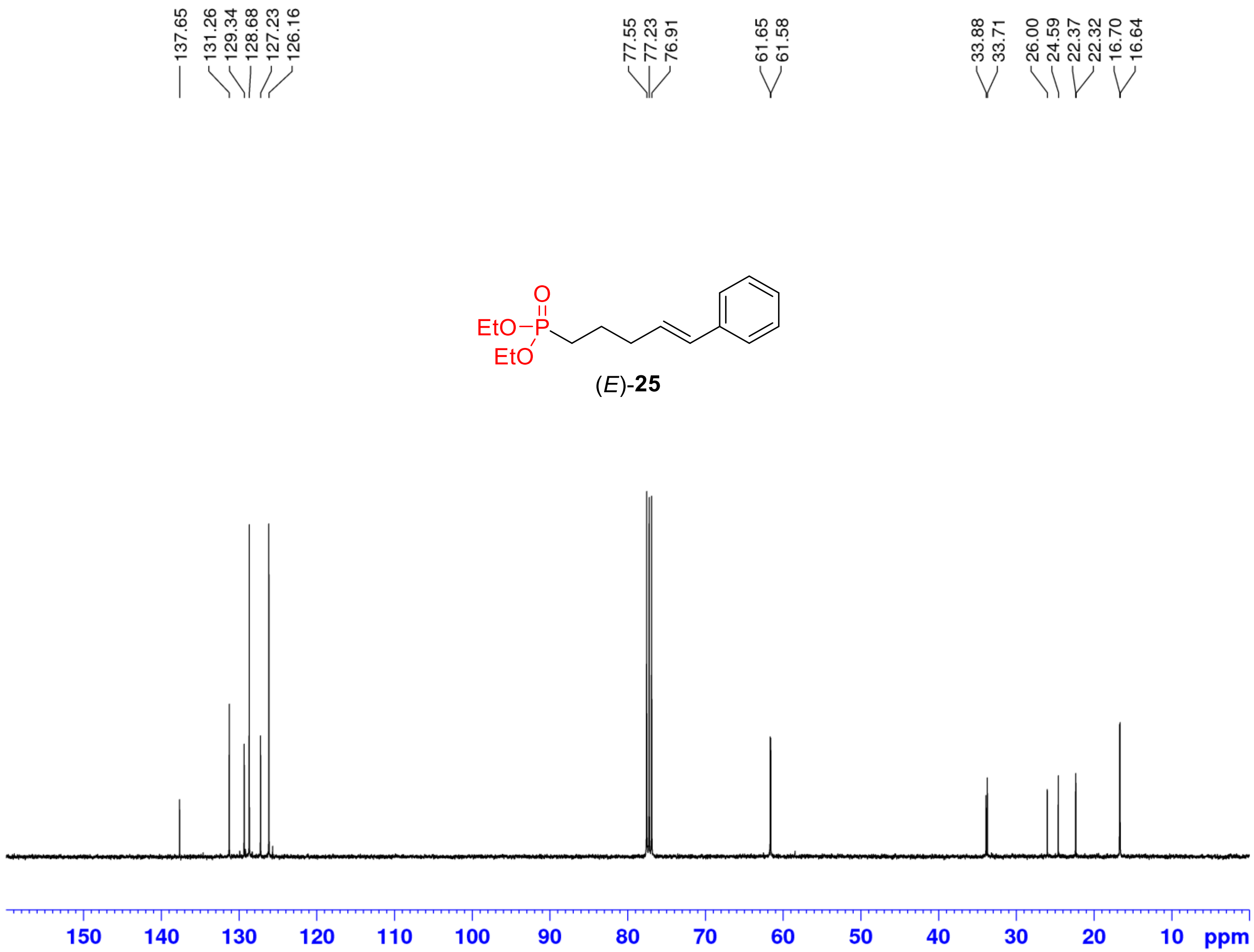


7.366 7.362 7.345 7.342 7.328 7.323 7.310 7.306 7.290 7.285 7.239 7.235 7.231 7.217 6.444 6.404 6.218 6.200 6.183 6.178 6.161 6.144 4.167 4.159 4.149 4.146 4.142 4.126 4.124 4.121 4.107 4.103 4.089 4.086 4.083 4.072 4.064 2.333 2.316 2.300 1.836 1.831 1.825 1.817 1.809 1.796 1.788 1.785 1.779 1.776 1.769 1.764 1.360 1.342 1.325
EtO
EtO
P
O
(E)-25
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
5.39
1.00
1.01
4.33
2.05
4.37
6.46

(*E*)-25: ^{31}P NMR

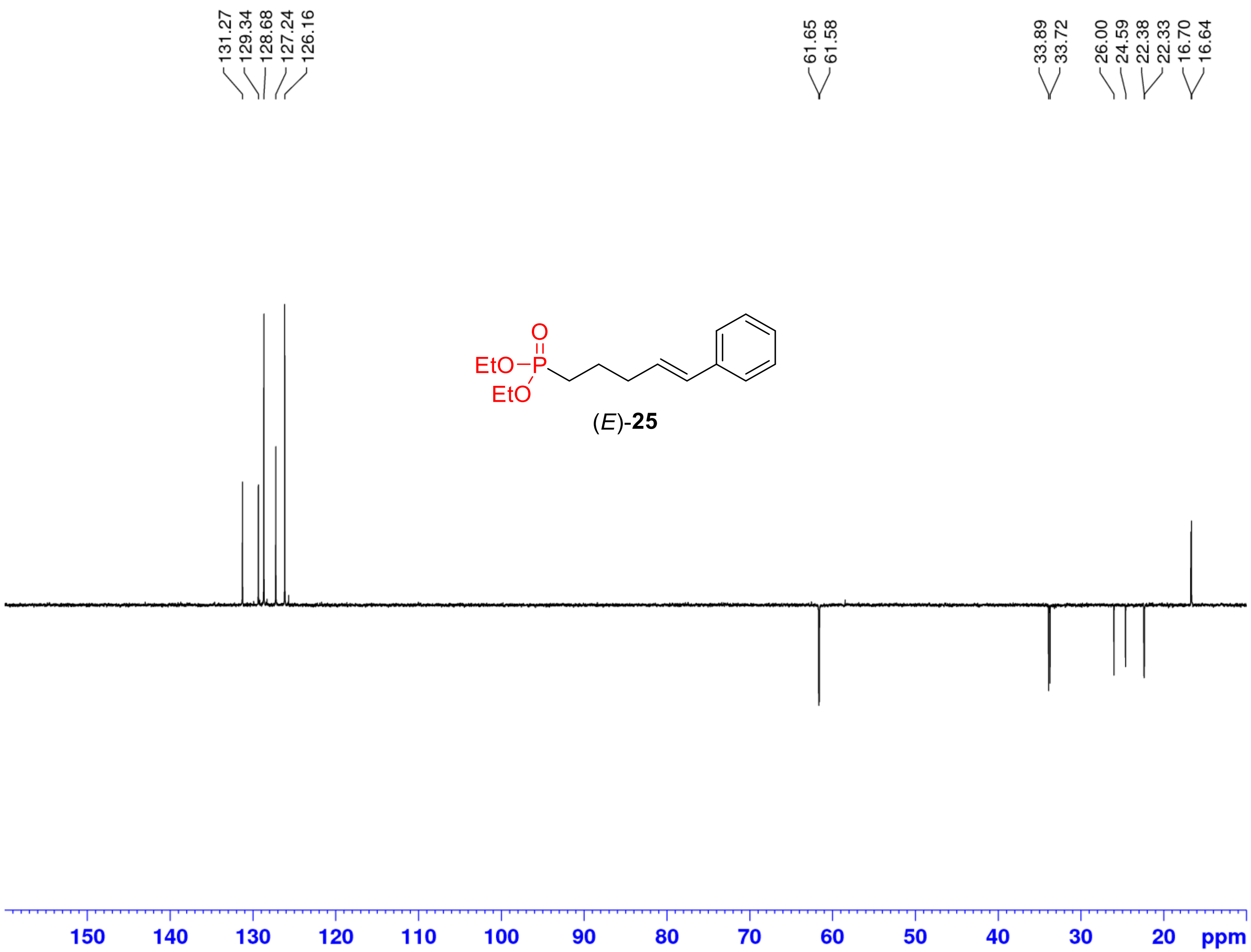

32.21
EtO
EtO
O
P
(E)-25
39
38
37
36
35
34
33
32
31
30
29
28
27
26
25
24
23
22
ppm

(*E*)-25: ^{13}C NMR

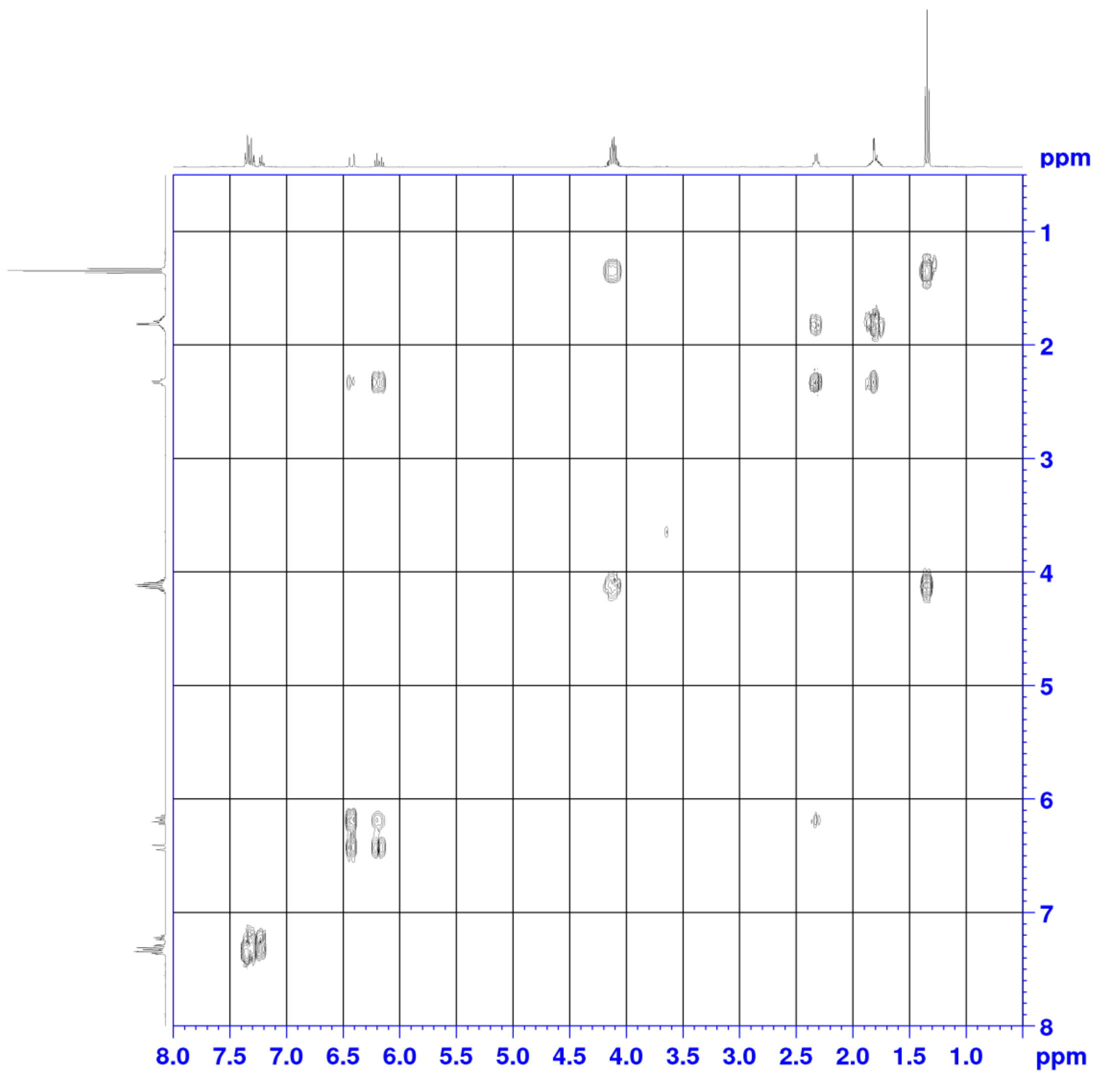


137.65
131.26
129.34
128.68
127.23
126.16
77.55
77.23
76.91
61.65
61.58
33.88
33.71
26.00
24.59
22.37
22.32
16.70
16.64
O
EtO
P
EtO
(E)-25
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

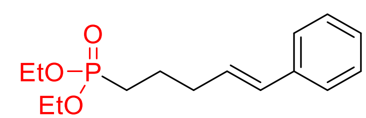
(*E*)-25: ^{13}C DEPT135



(*E*)-25: COSY



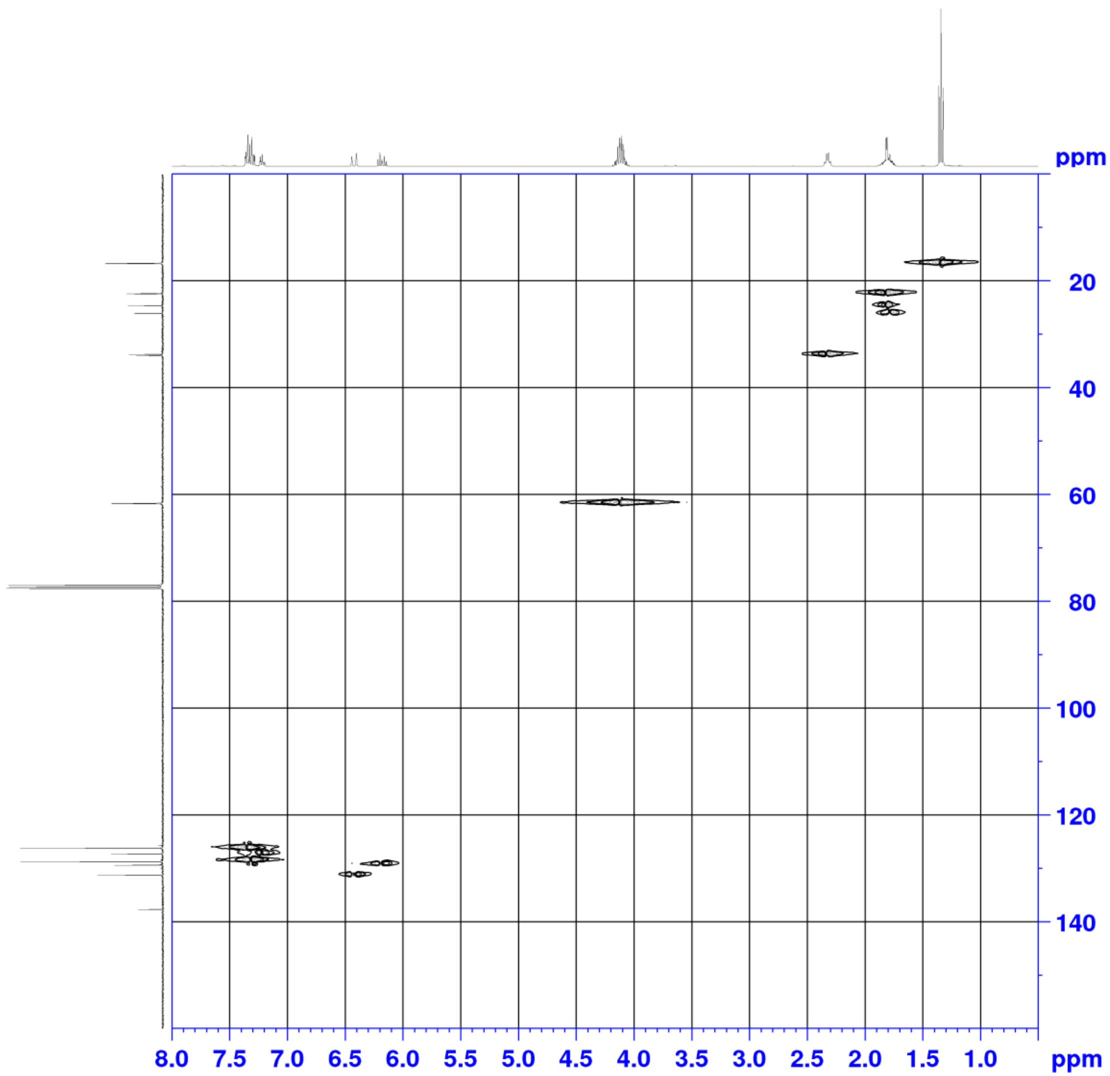
ppm
1
2
3
4
5
6
7
8
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

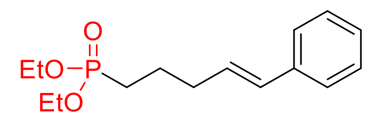


O
EtO
P
EtO

(*E*)-**25**

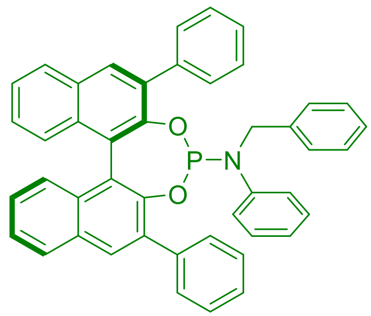
(*E*)-25: ^{13}C HSQC



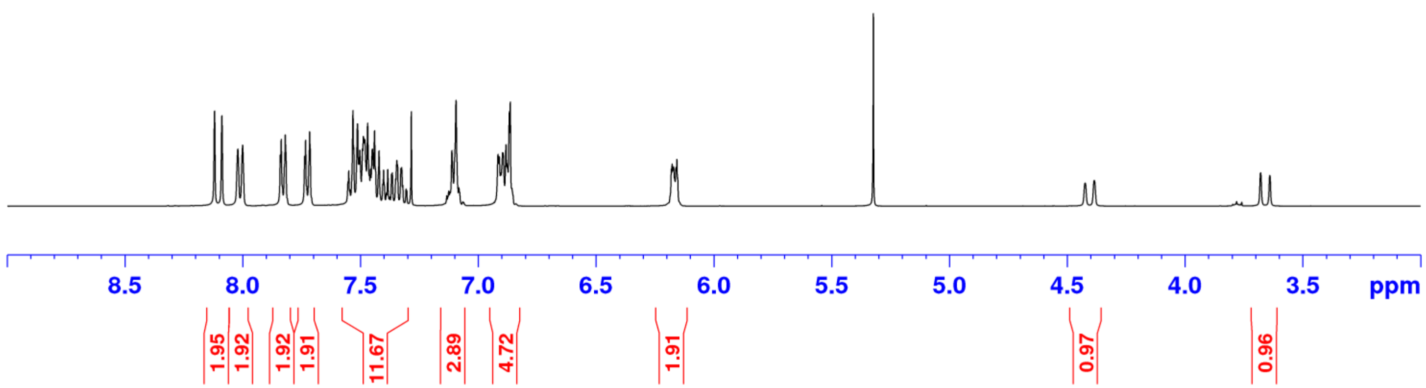


(*E*)-**25**

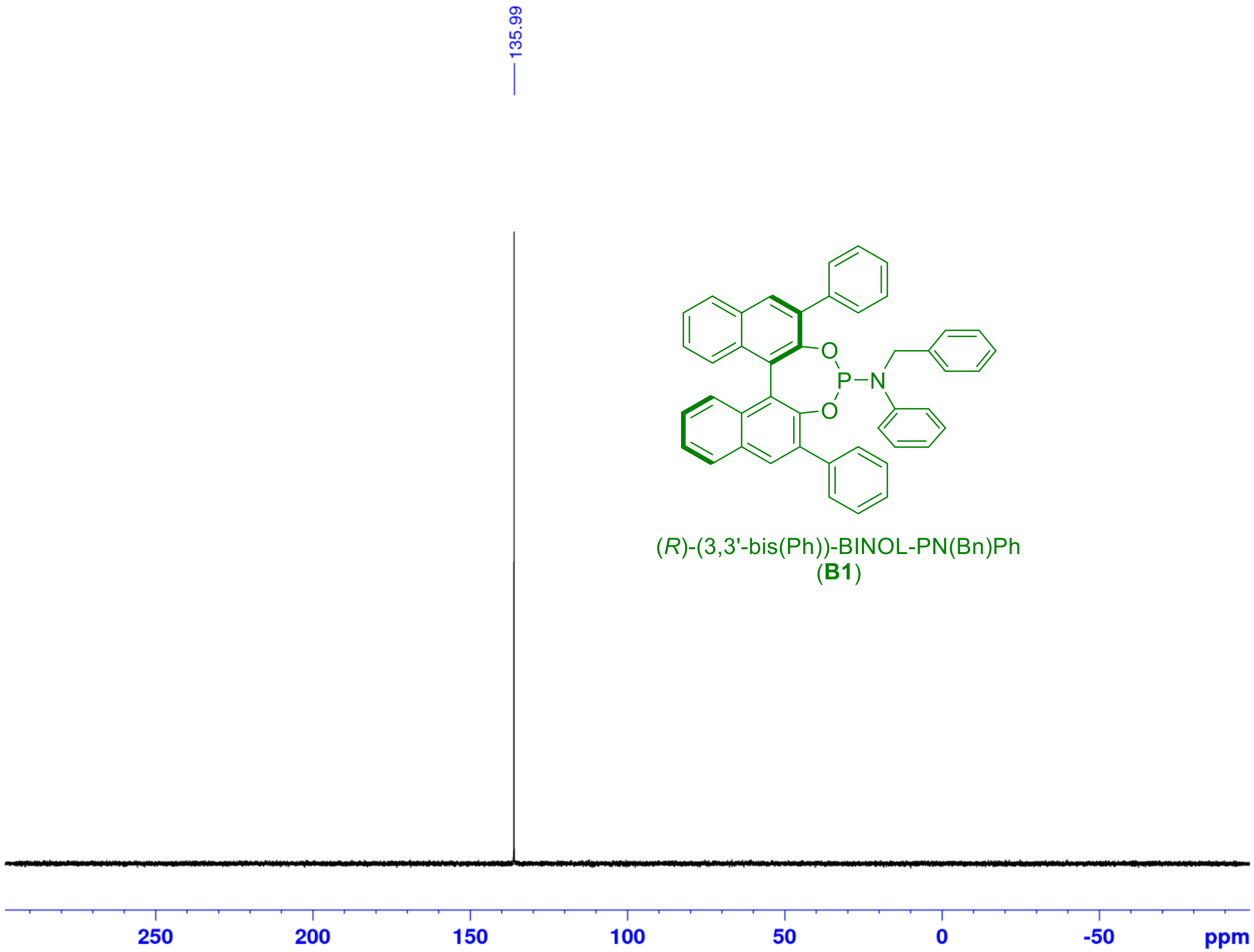
Ligand B1: ^{1}H NMR



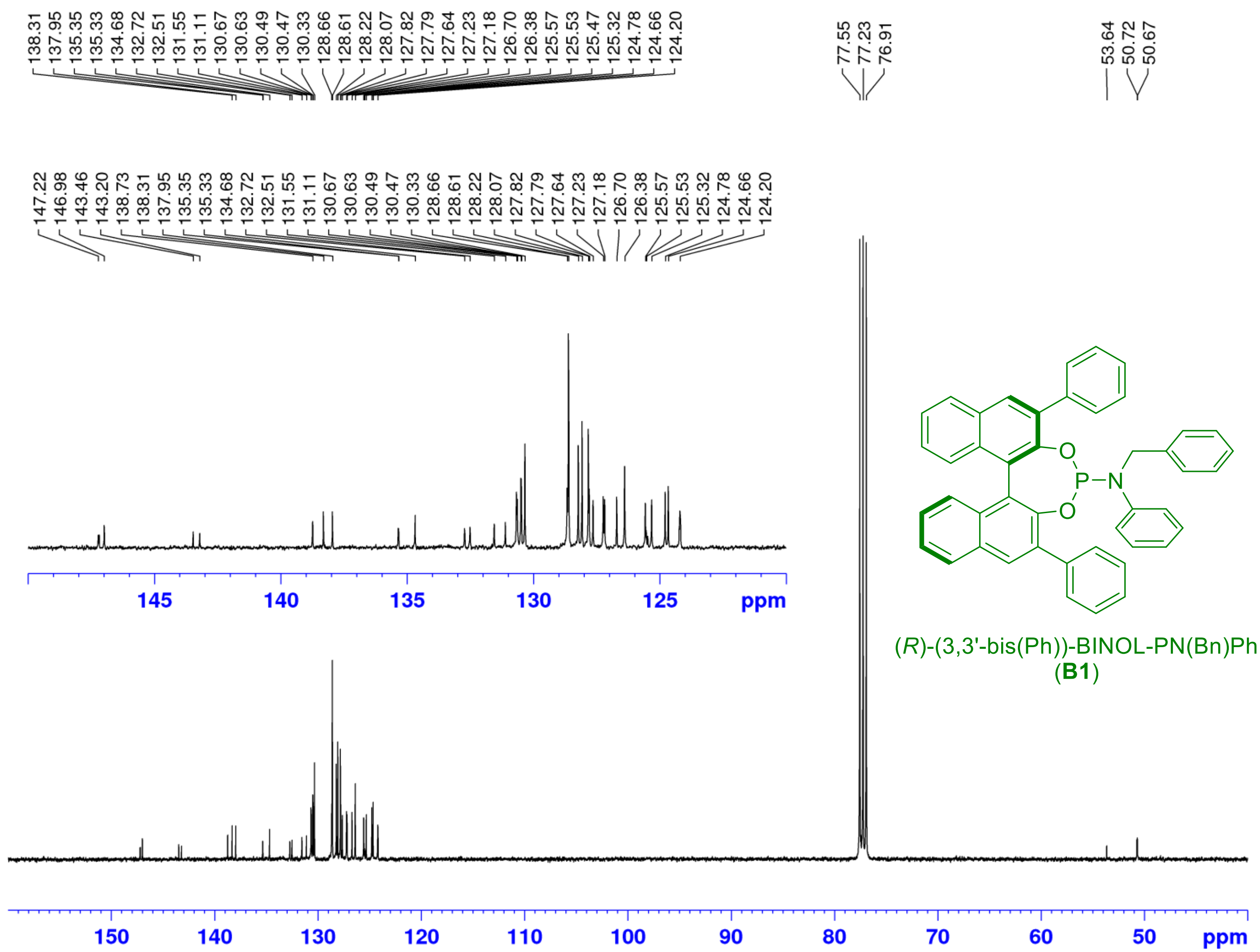
(*R*)-(3,3'-bis(Ph))-BINOL-PN(Bn)Ph
(**B1**)



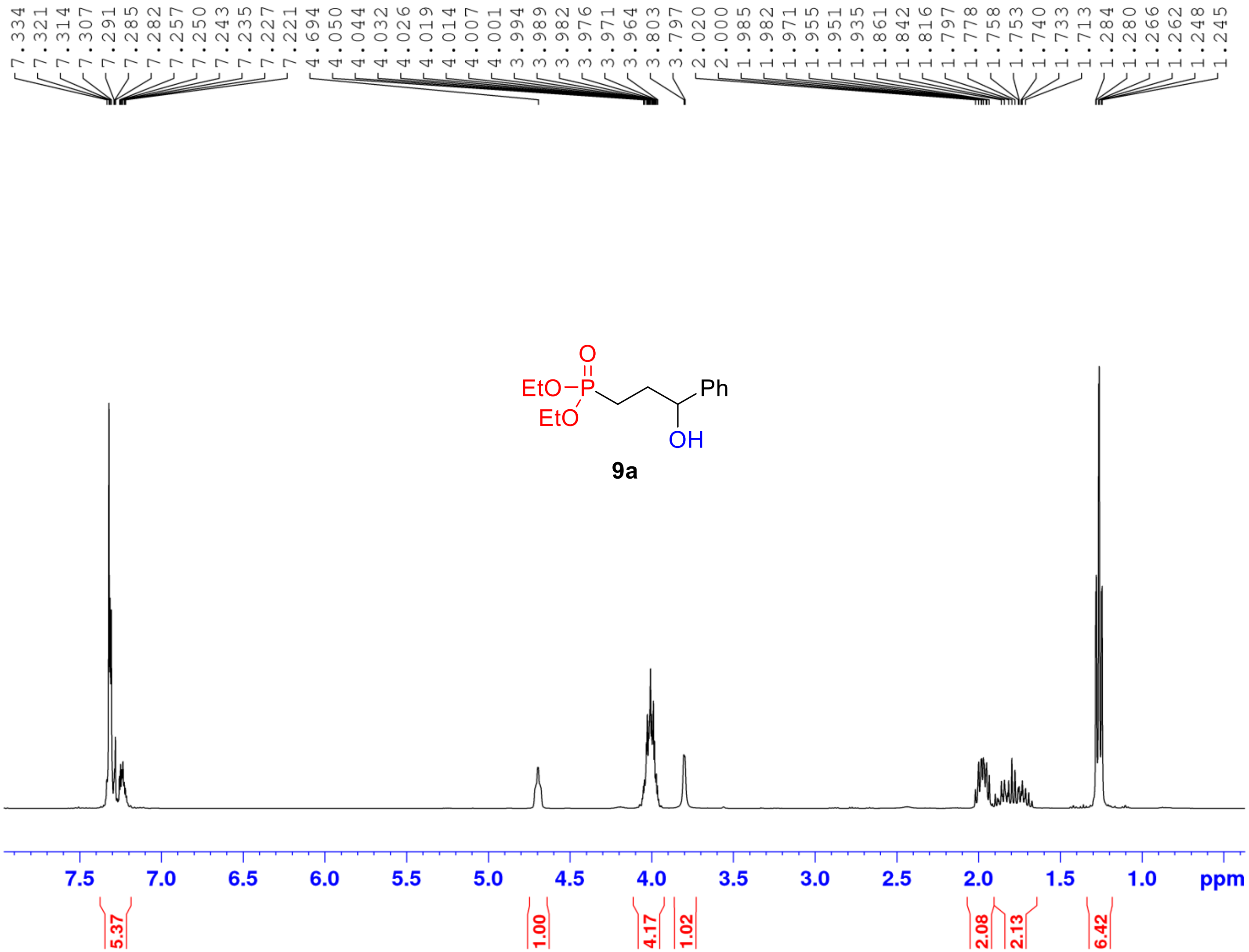
Ligand B1: ^{31}P NMR



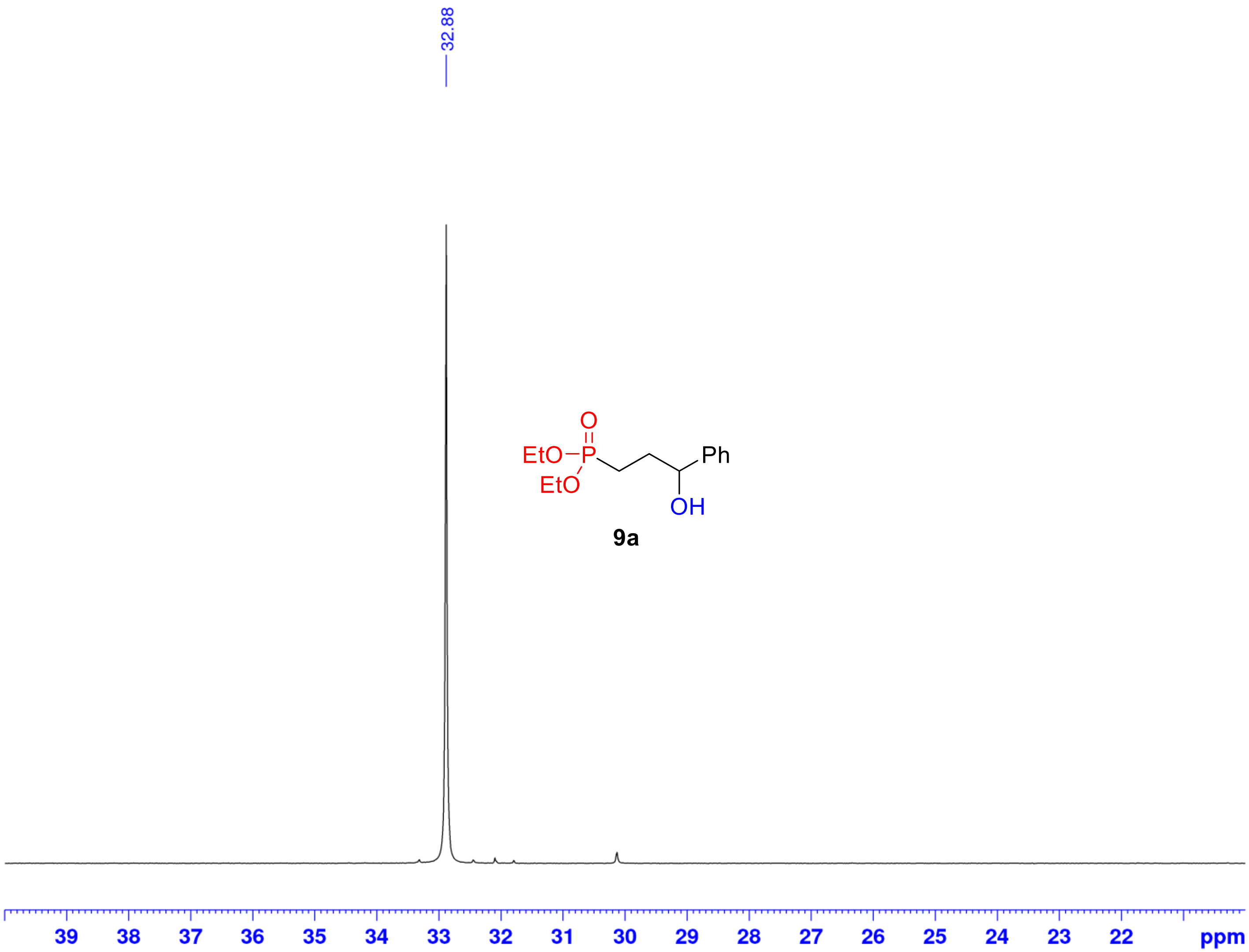
Ligand B1: ^{13}C NMR



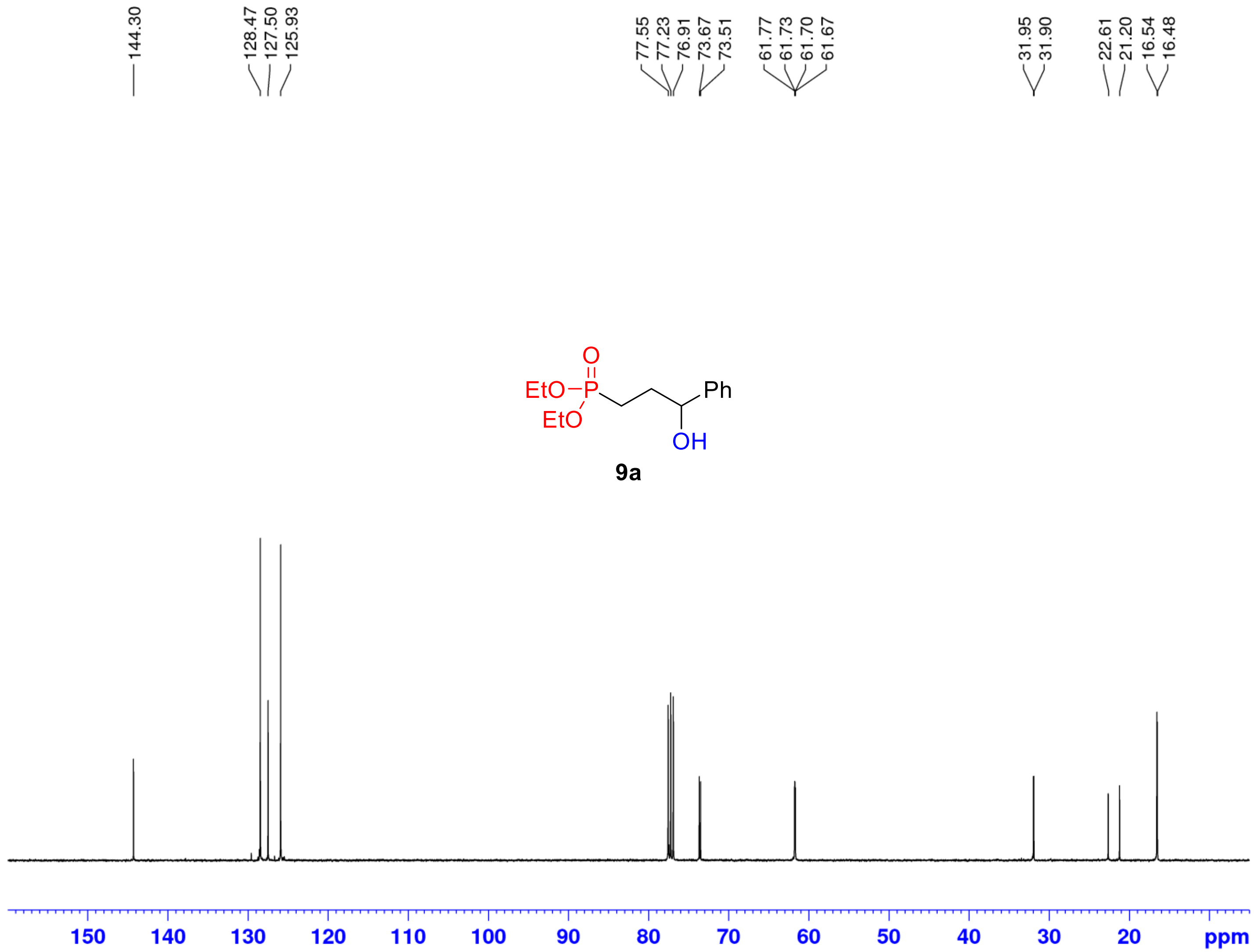
9a: ¹H NMR


7.334 7.321 7.314 7.307 7.291 7.285 7.282 7.257 7.250 7.243 7.235 7.227 7.221 4.694 4.050 4.044 4.032 4.026 4.019 4.014 4.007 4.001 3.994 3.989 3.982 3.976 3.971 3.964 3.803 3.797 2.020 2.000 1.985 1.982 1.971 1.955 1.951 1.935 1.861 1.842 1.816 1.797 1.778 1.758 1.753 1.740 1.733 1.713 1.284 1.280 1.266 1.262 1.248 1.245
O
EtO
P
EtO
Ph
OH
9a
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
5.37 1.00 4.17 1.02 2.08 2.13 6.42

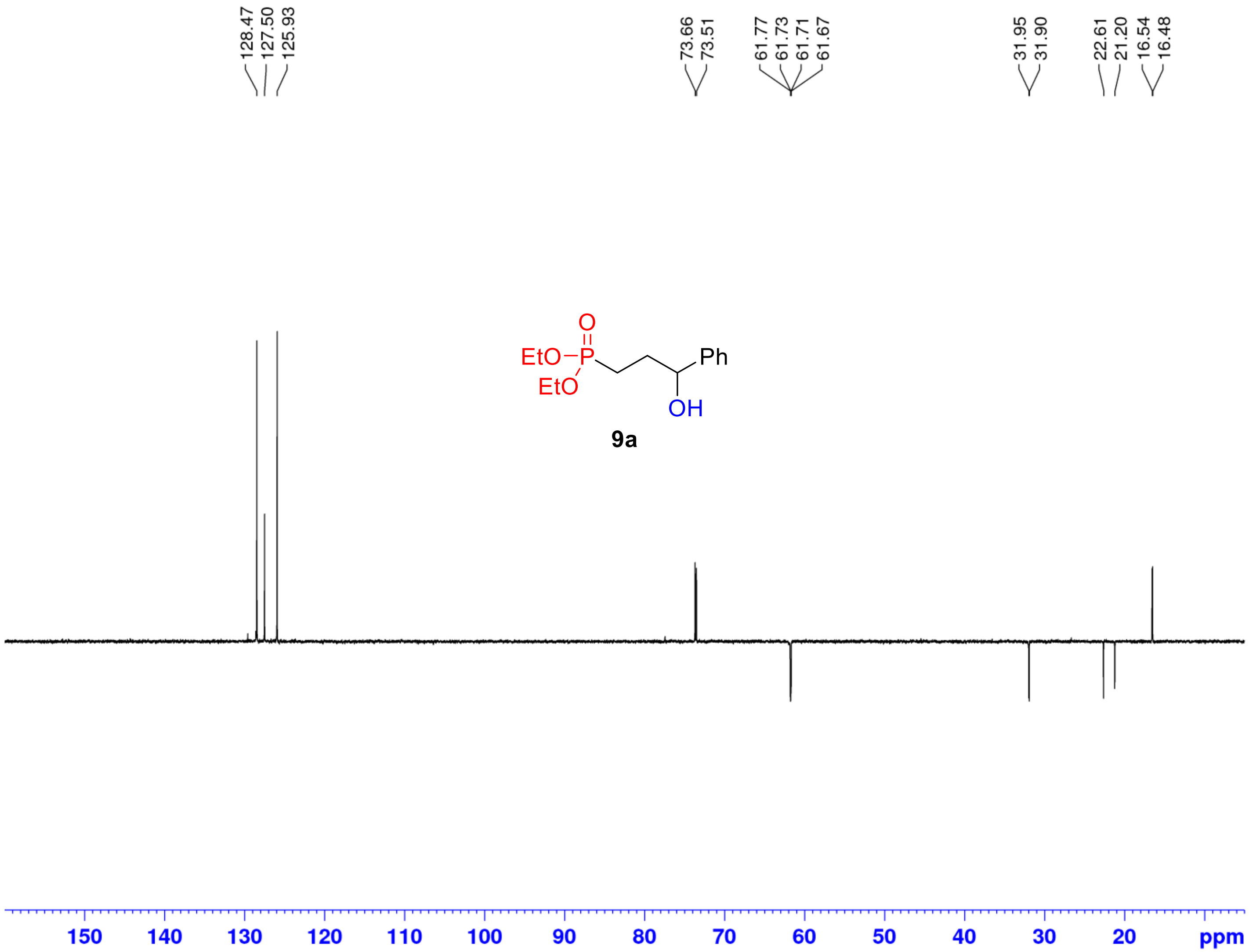
9a: ^{31}P NMR



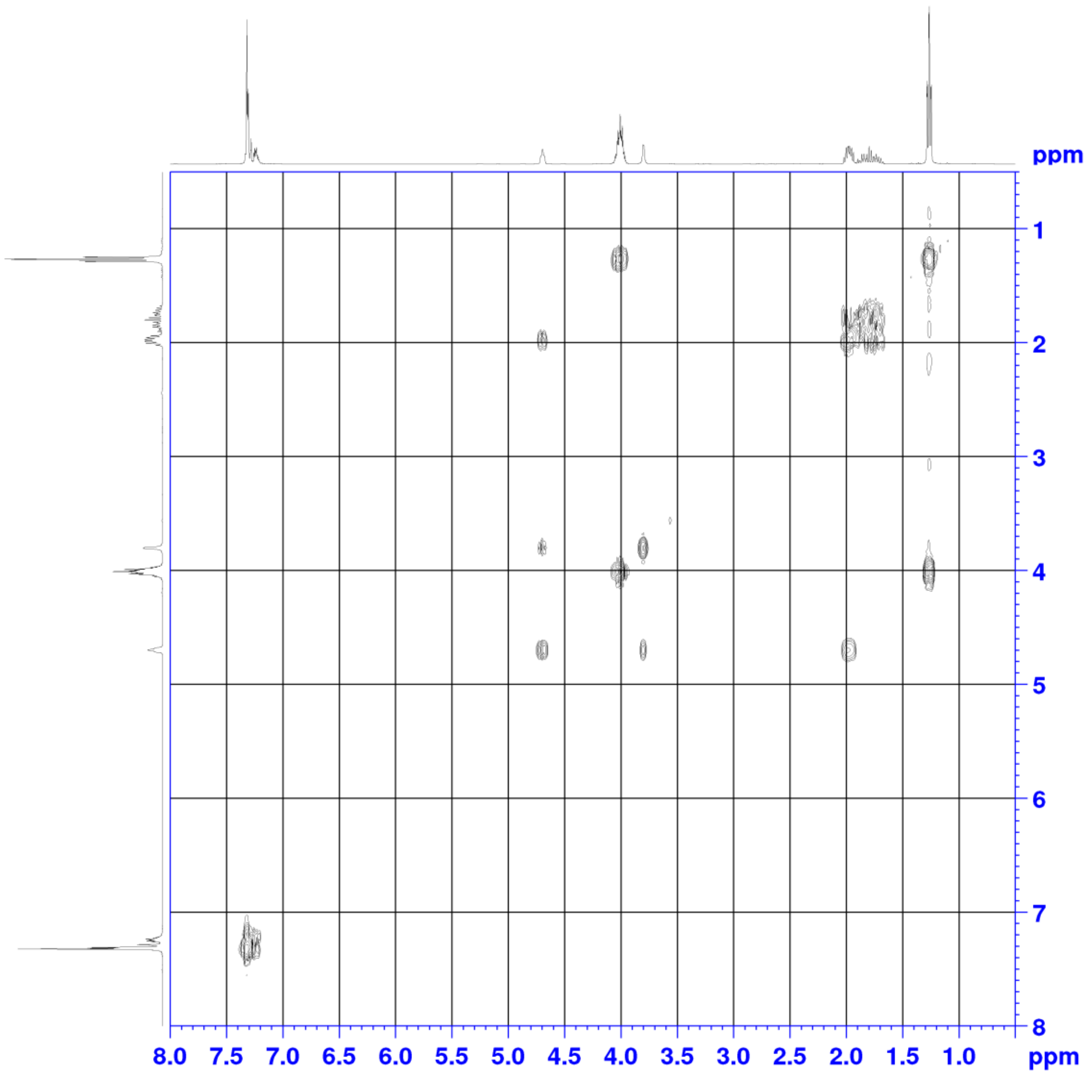
9a: ^{13}C NMR


144.30
128.47
127.50
125.93
77.55
77.23
76.91
73.67
73.51
61.77
61.73
61.70
61.67
31.95
31.90
22.61
21.20
16.54
16.48
O
EtO
P
EtO
Ph
OH
9a
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

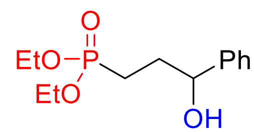
9a: ^{13}C DEPT135



9a: COSY



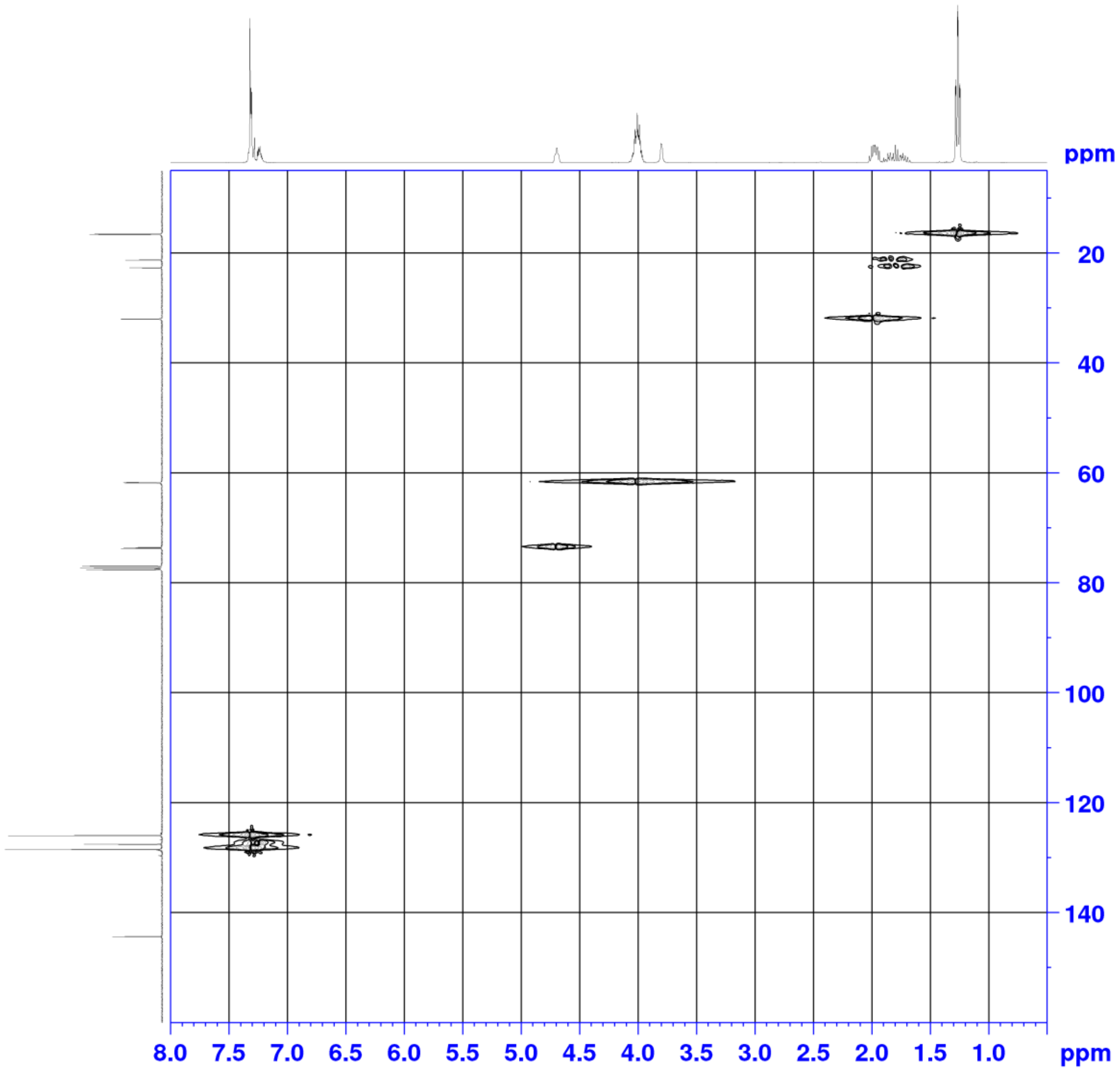
ppm
1
2
3
4
5
6
7
8
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

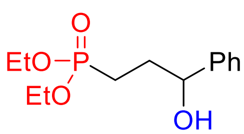


O
EtO
P
EtO
Ph
OH

9a

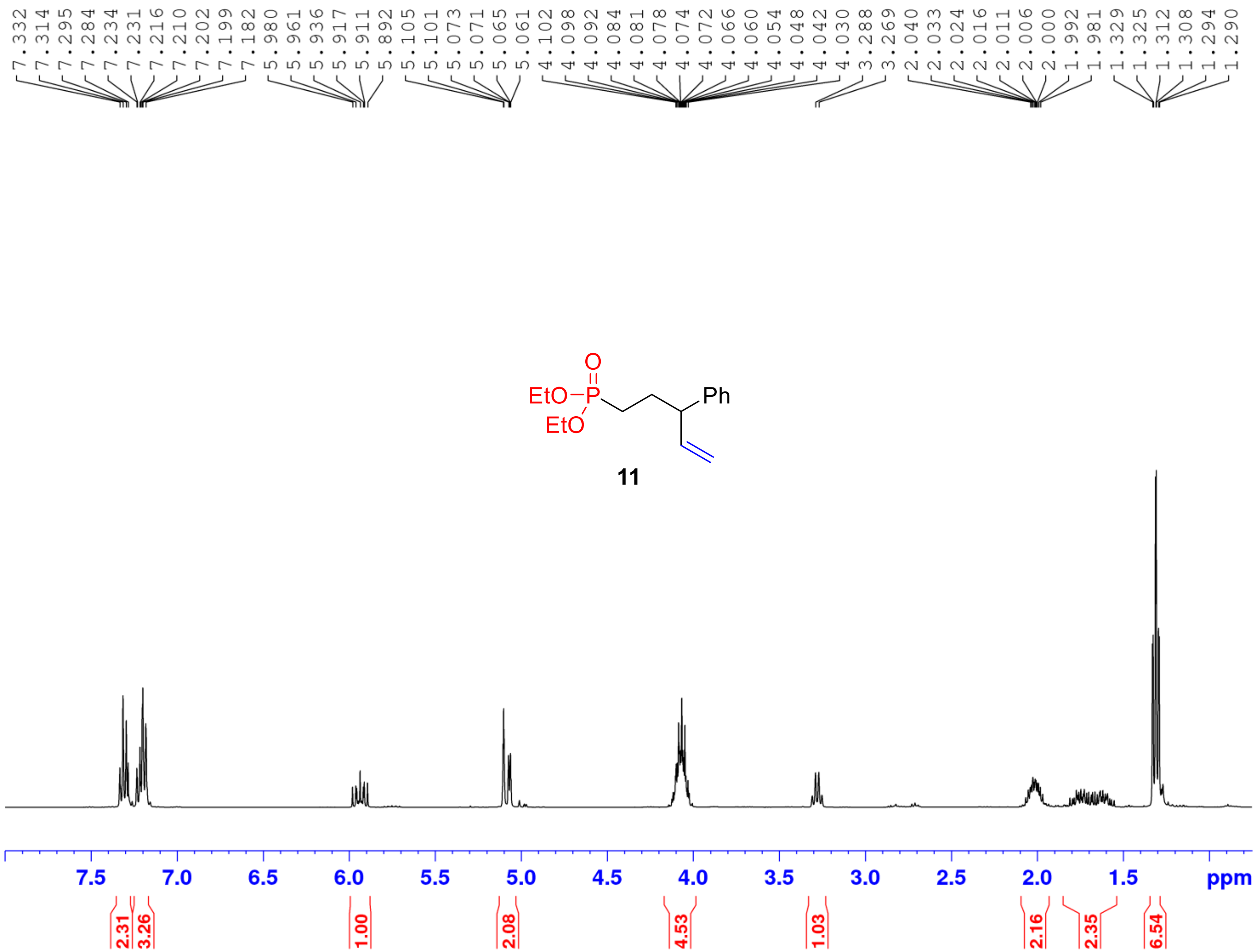
9a: ^{13}C HSQC





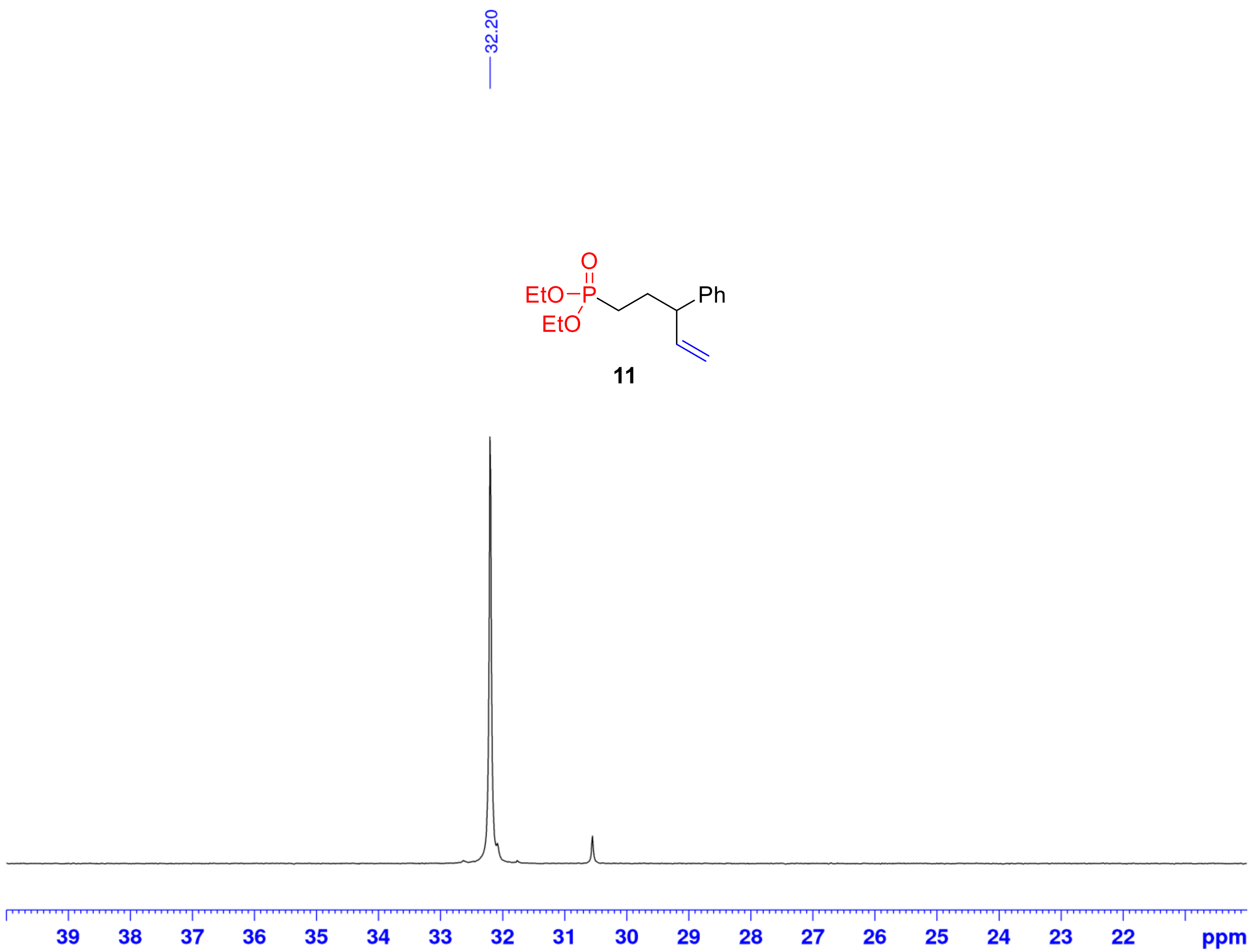
9a

11: ^{1}H NMR



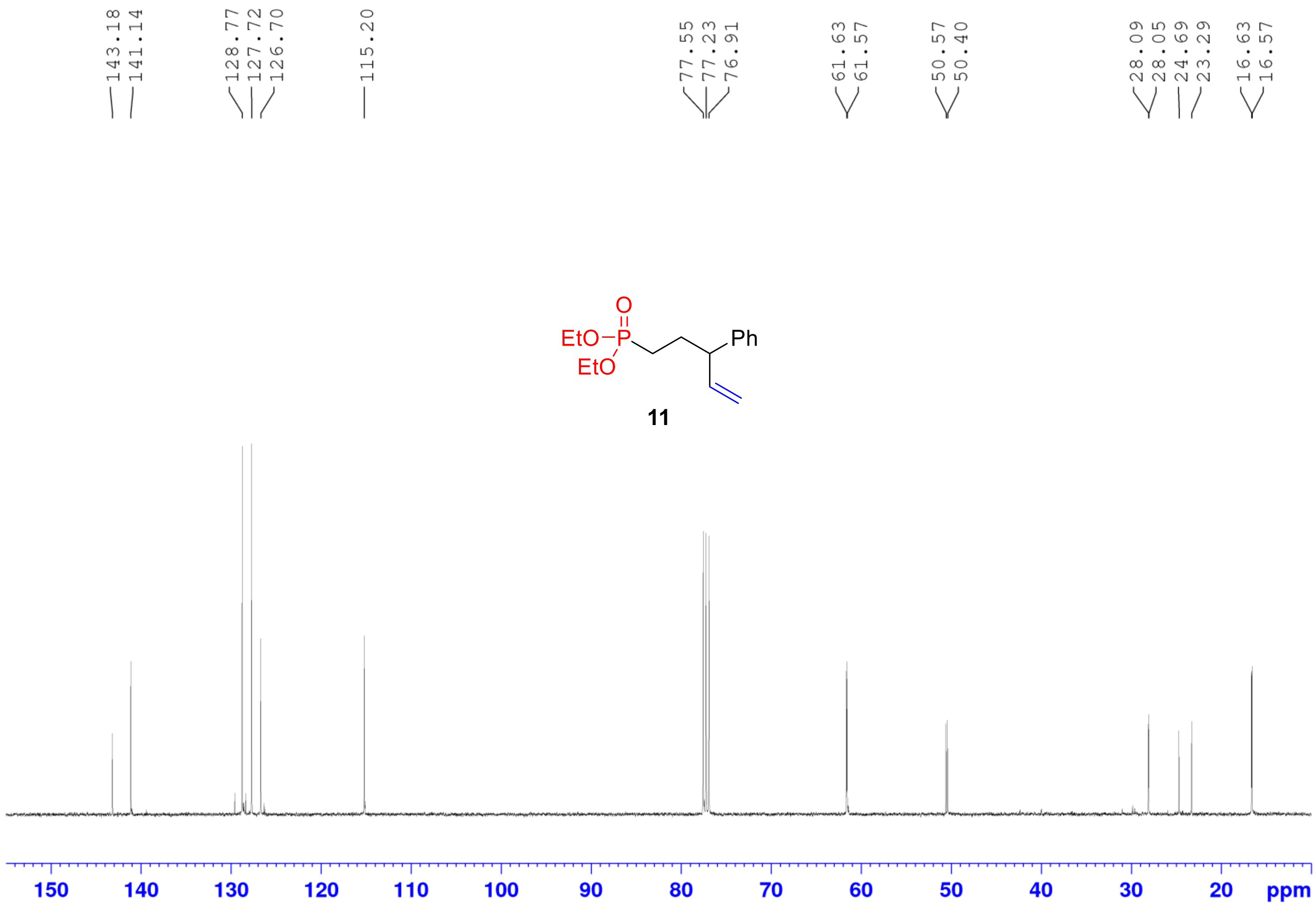
7.332 7.314 7.295 7.284 7.234 7.231 7.216 7.210 7.202 7.199 7.182 5.980 5.961 5.936 5.917 5.911 5.892 5.105 5.101 5.073 5.071 5.065 5.061 4.102 4.098 4.092 4.084 4.081 4.078 4.074 4.072 4.066 4.060 4.054 4.048 4.042 4.030 3.288 3.269 2.040 2.033 2.024 2.016 2.011 2.006 2.000 1.992 1.981 1.329 1.325 1.312 1.308 1.294 1.290
O
EtO
P
EtO
Ph
11
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 ppm
2.31 3.26 1.00 2.08 4.53 1.03 2.16 2.35 6.54

11: ^{31}P NMR

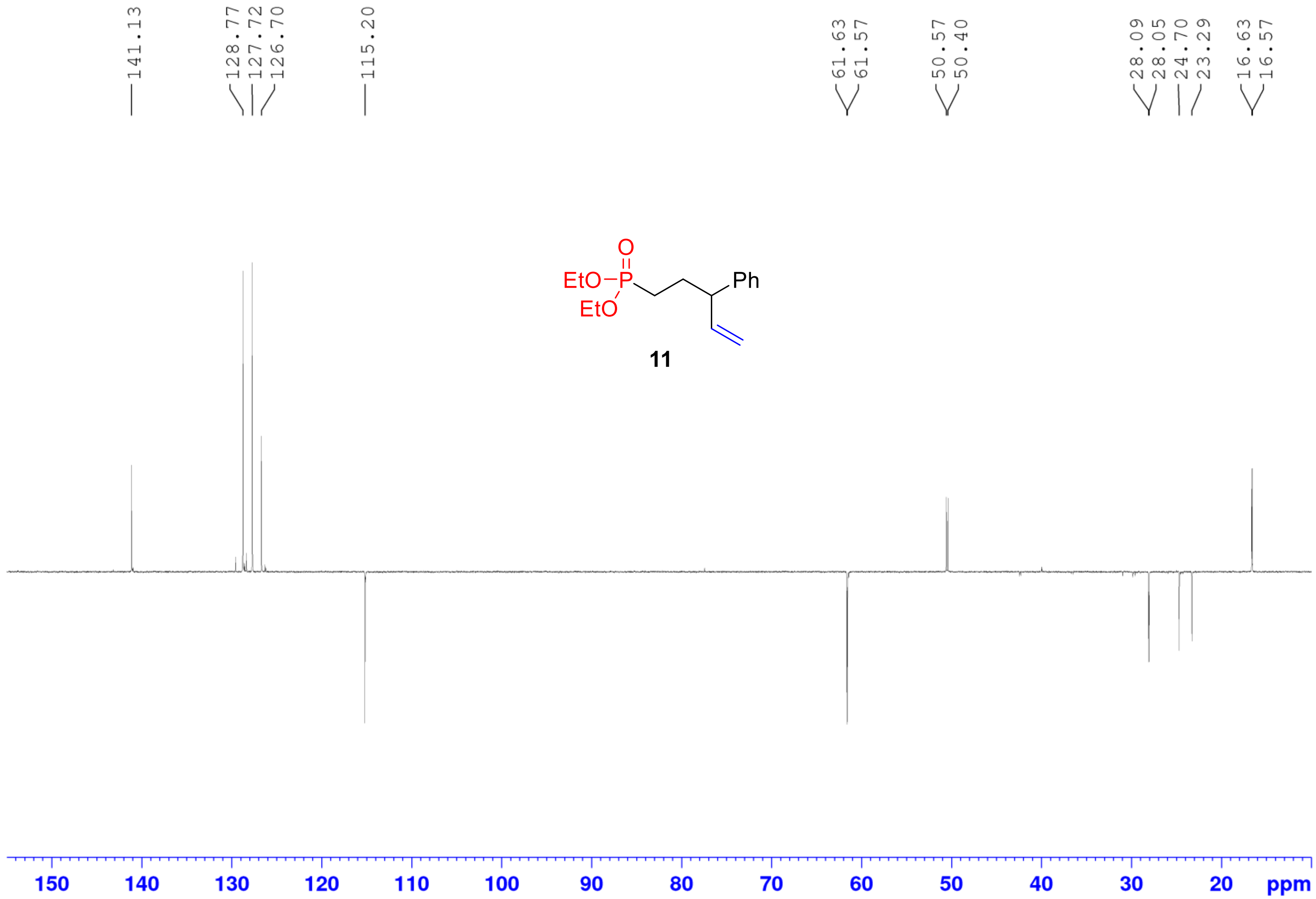


32.20
EtO
EtO
O
P
Ph
11
39
38
37
36
35
34
33
32
31
30
29
28
27
26
25
24
23
22
ppm

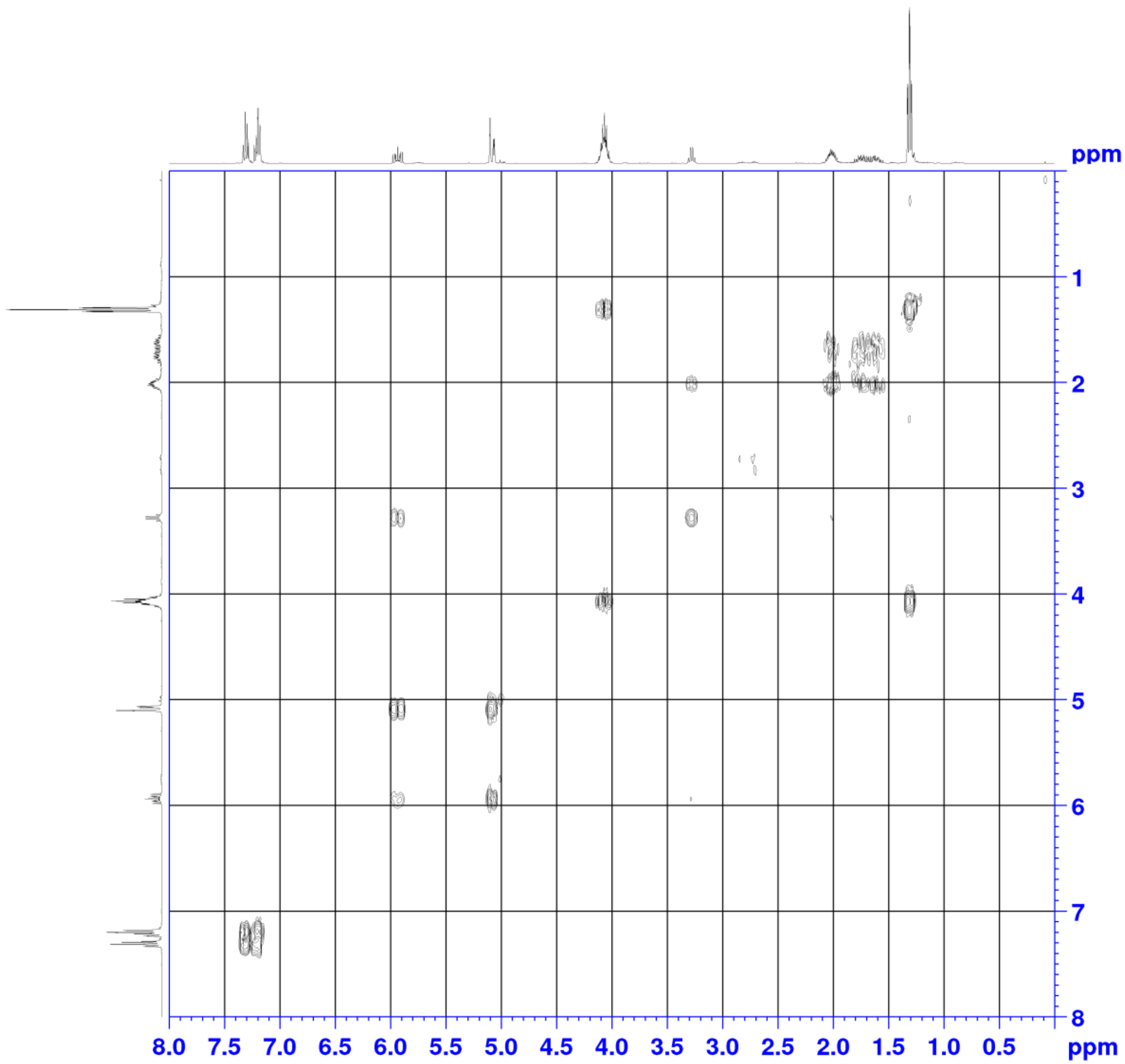
11: ^{13}C NMR


143.18
141.14
128.77
127.72
126.70
115.20
77.55
77.23
76.91
61.63
61.57
50.57
50.40
28.09
28.05
24.69
23.29
16.63
16.57
O
EtO
P
EtO
Ph
11
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

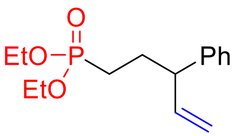
11: ^{13}C DEPT135



11: COSY



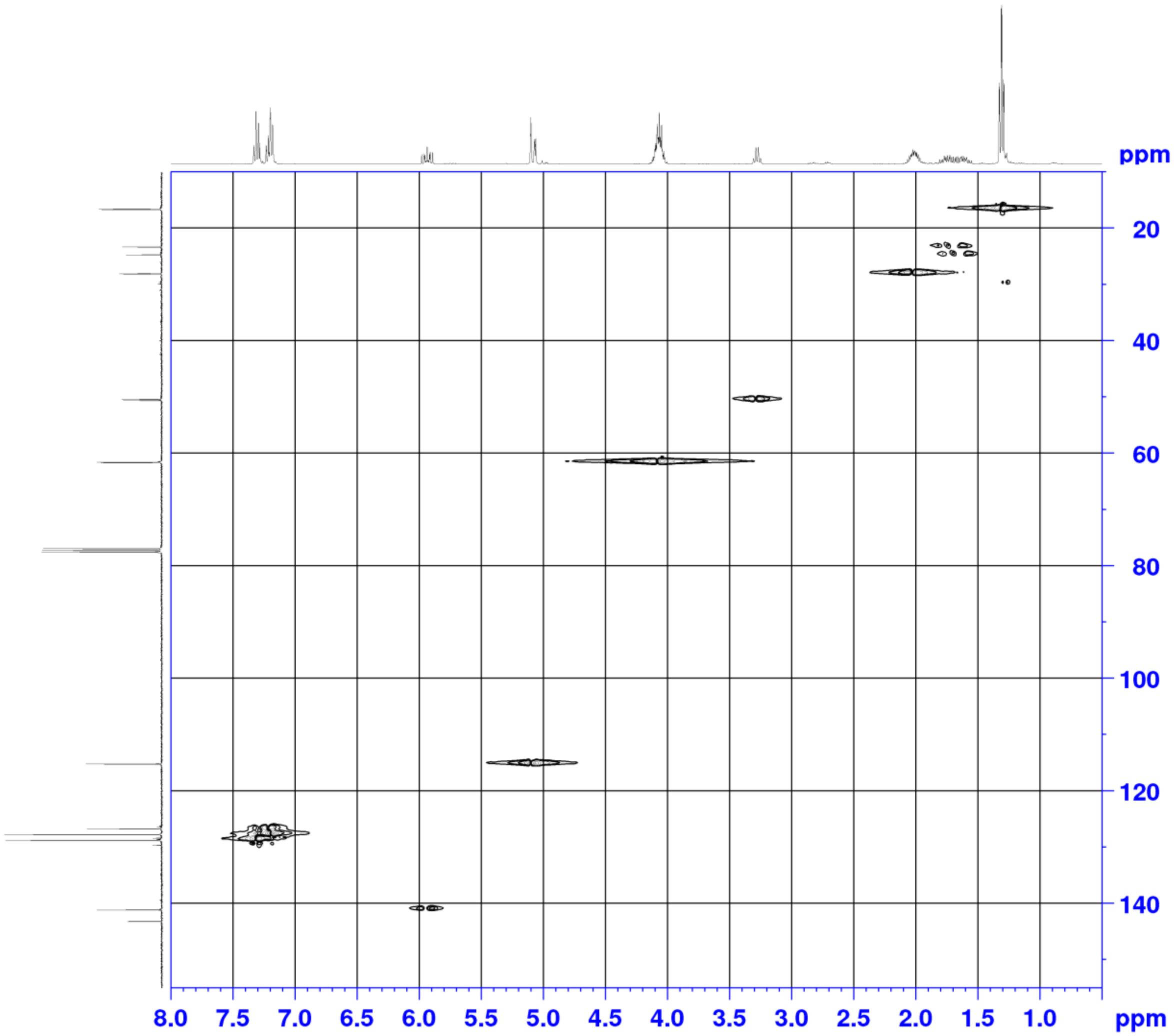
ppm
1
2
3
4
5
6
7
8
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm

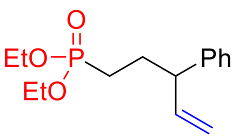


O
EtO
P
Ph
EtO

11

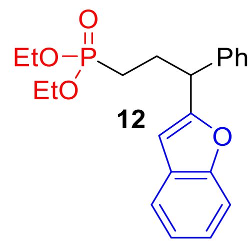
11: ^{13}C HSQC

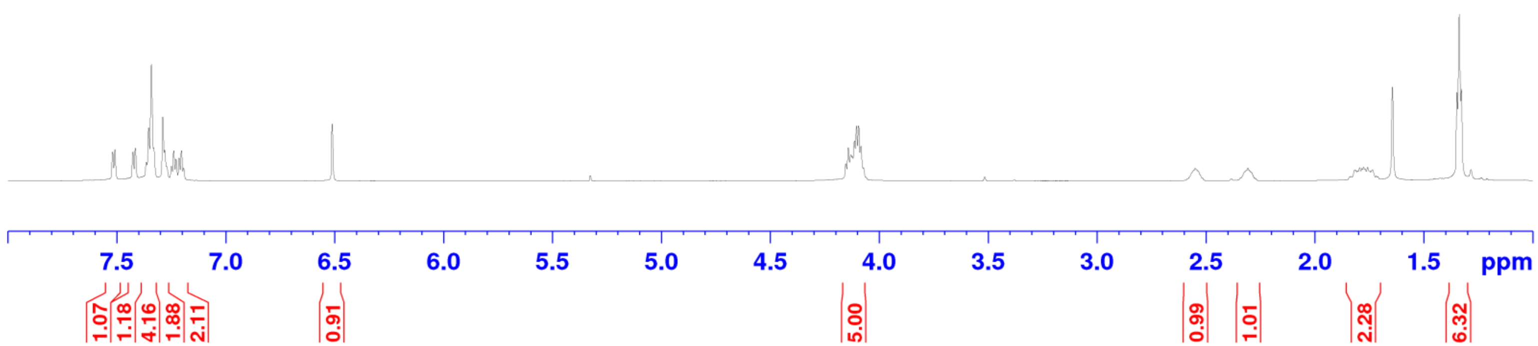




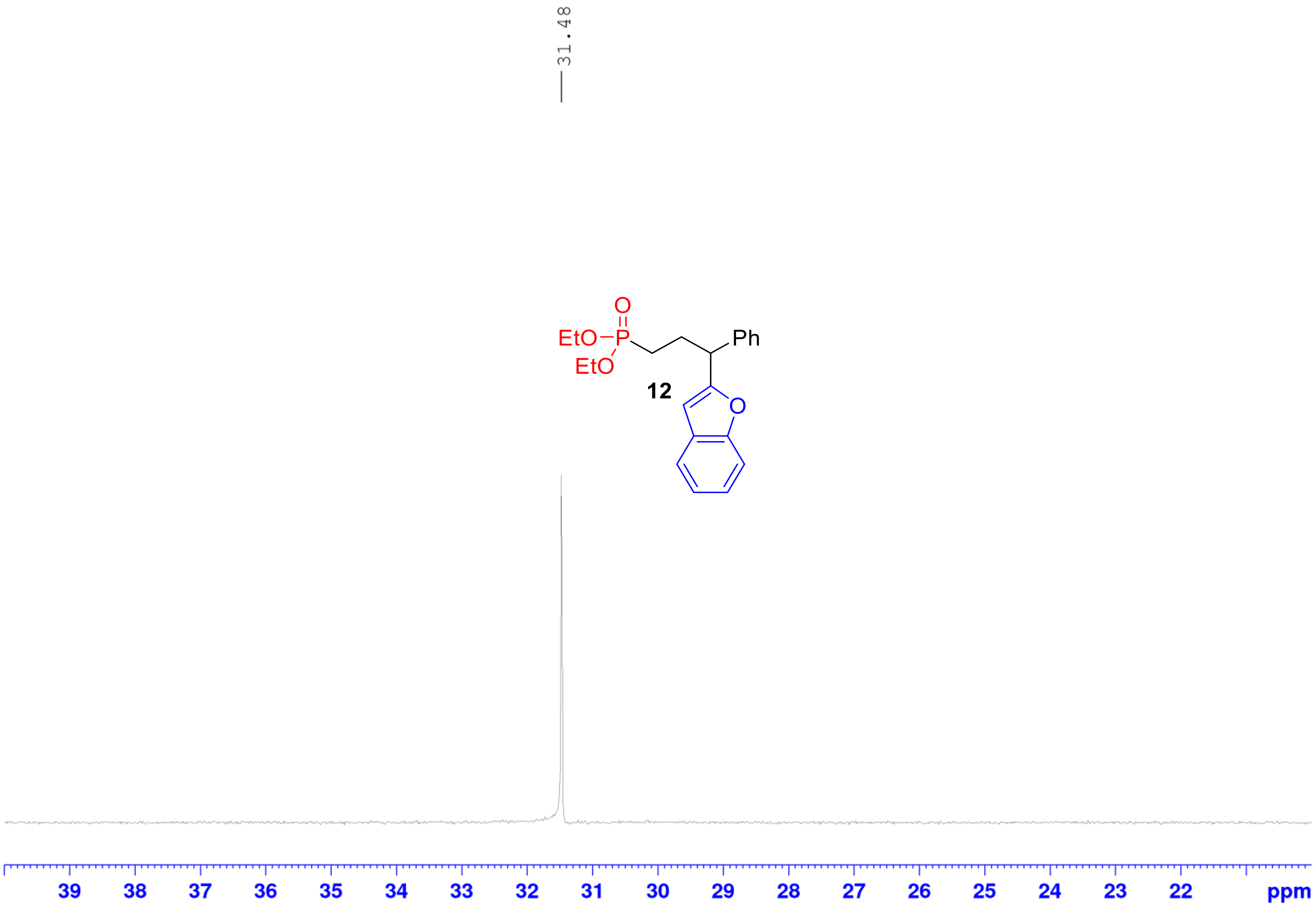
11

12: ^{1}H NMR


EtO
EtO
P
O
Ph
O
12

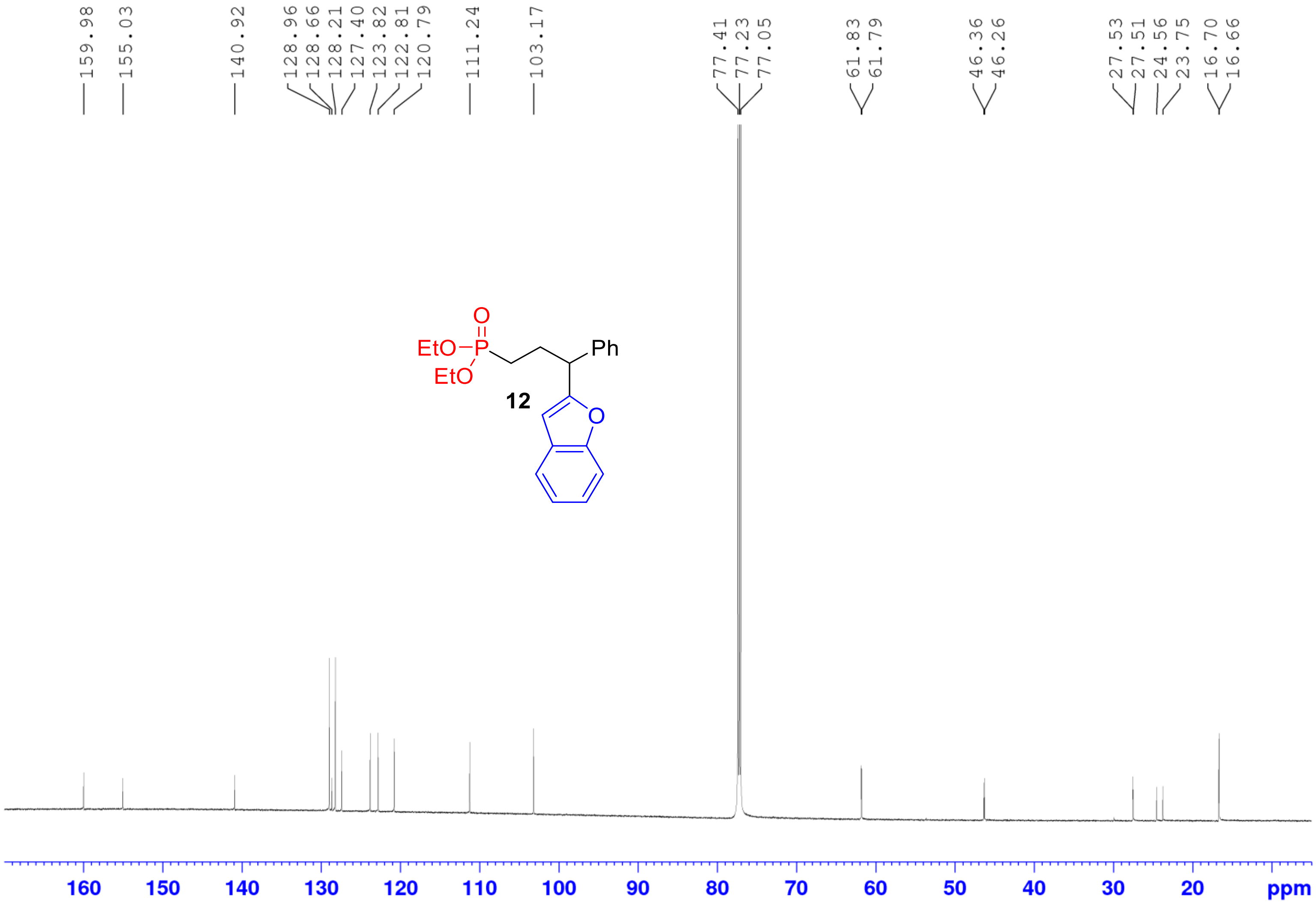

7.416
7.365
7.355
7.342
7.331
7.289
7.281
7.250
7.239
7.228
7.214
7.204
7.194
6.512
4.153
4.142
4.131
4.125
4.114
4.104
4.094
4.084
4.072
2.549
2.308
1.835
1.815
1.805
1.794
1.784
1.777
1.769
1.758
1.735
1.719
1.347
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
ppm
1.07
1.18
4.16
1.88
2.11
0.91
5.00
0.99
1.01
2.28
6.32

12: ^{31}P NMR



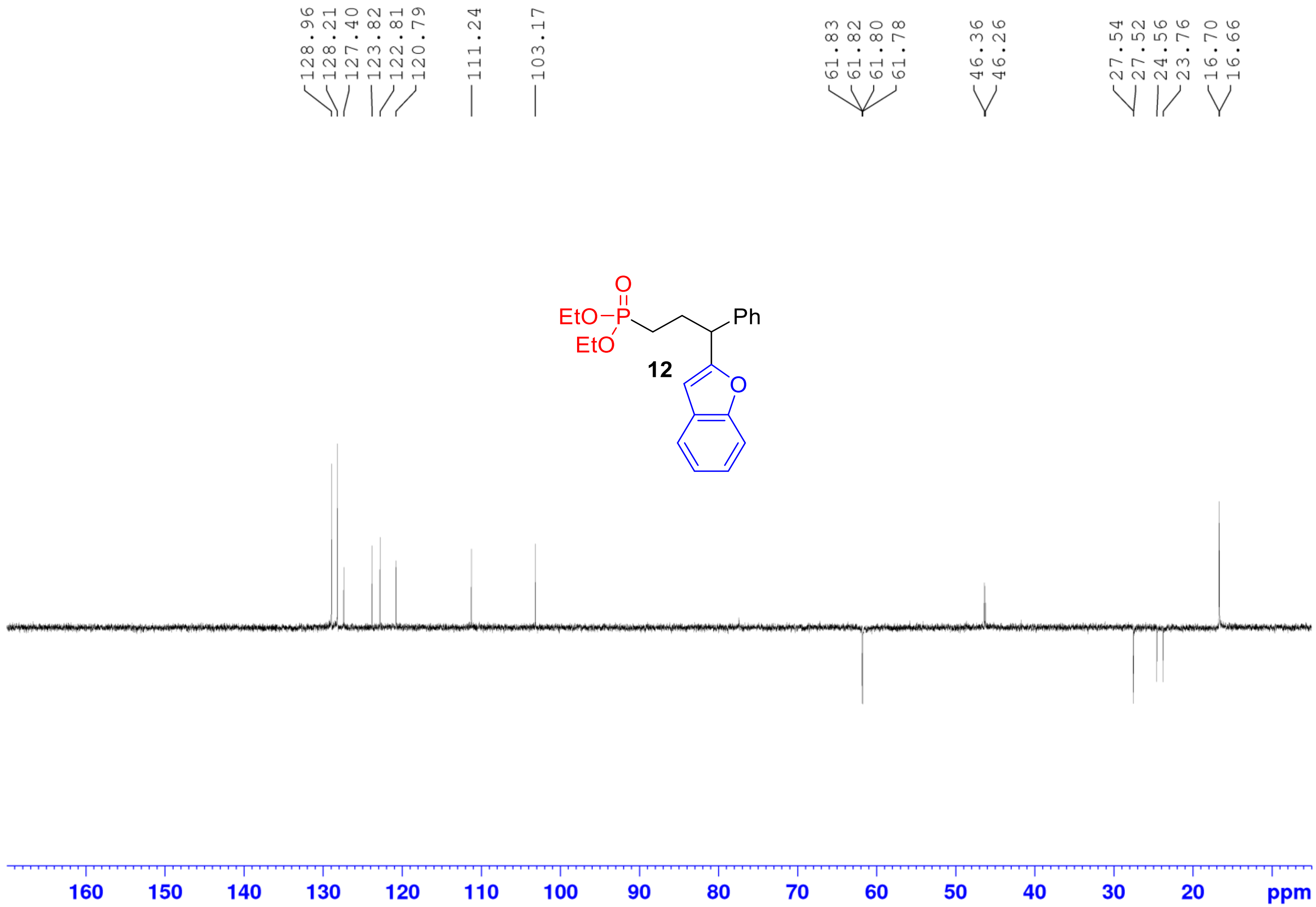
31.48
EtO
EtO
P
O
Ph
O
12
39
38
37
36
35
34
33
32
31
30
29
28
27
26
25
24
23
22
ppm

12: ^{13}C NMR

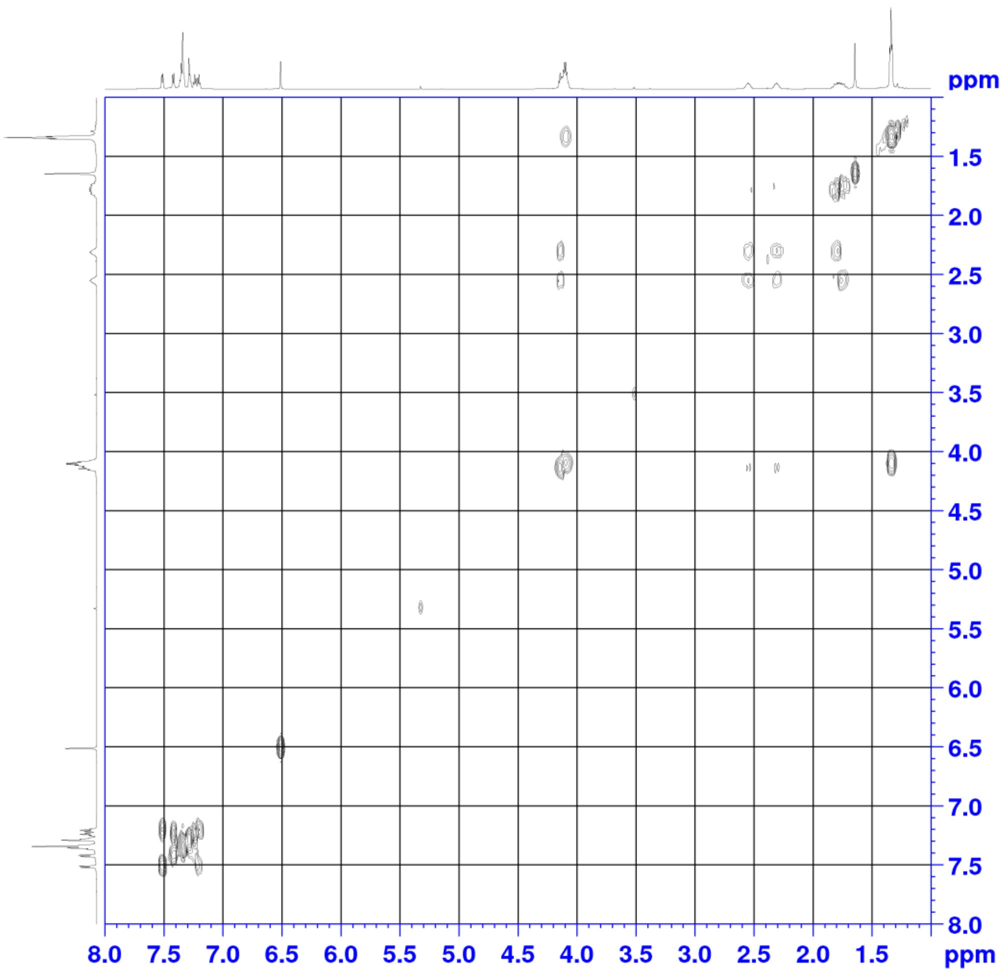


159.98
155.03
140.92
128.96
128.66
128.21
127.40
123.82
122.81
120.79
111.24
103.17
77.41
77.23
77.05
61.83
61.79
46.36
46.26
27.53
27.51
24.56
23.75
16.70
16.66
O
EtO
P
EtO
Ph
12
O
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

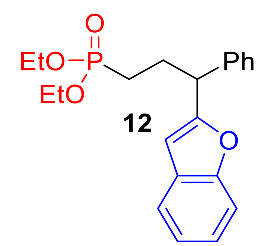
12: ^{13}C DEPT135



12: COSY



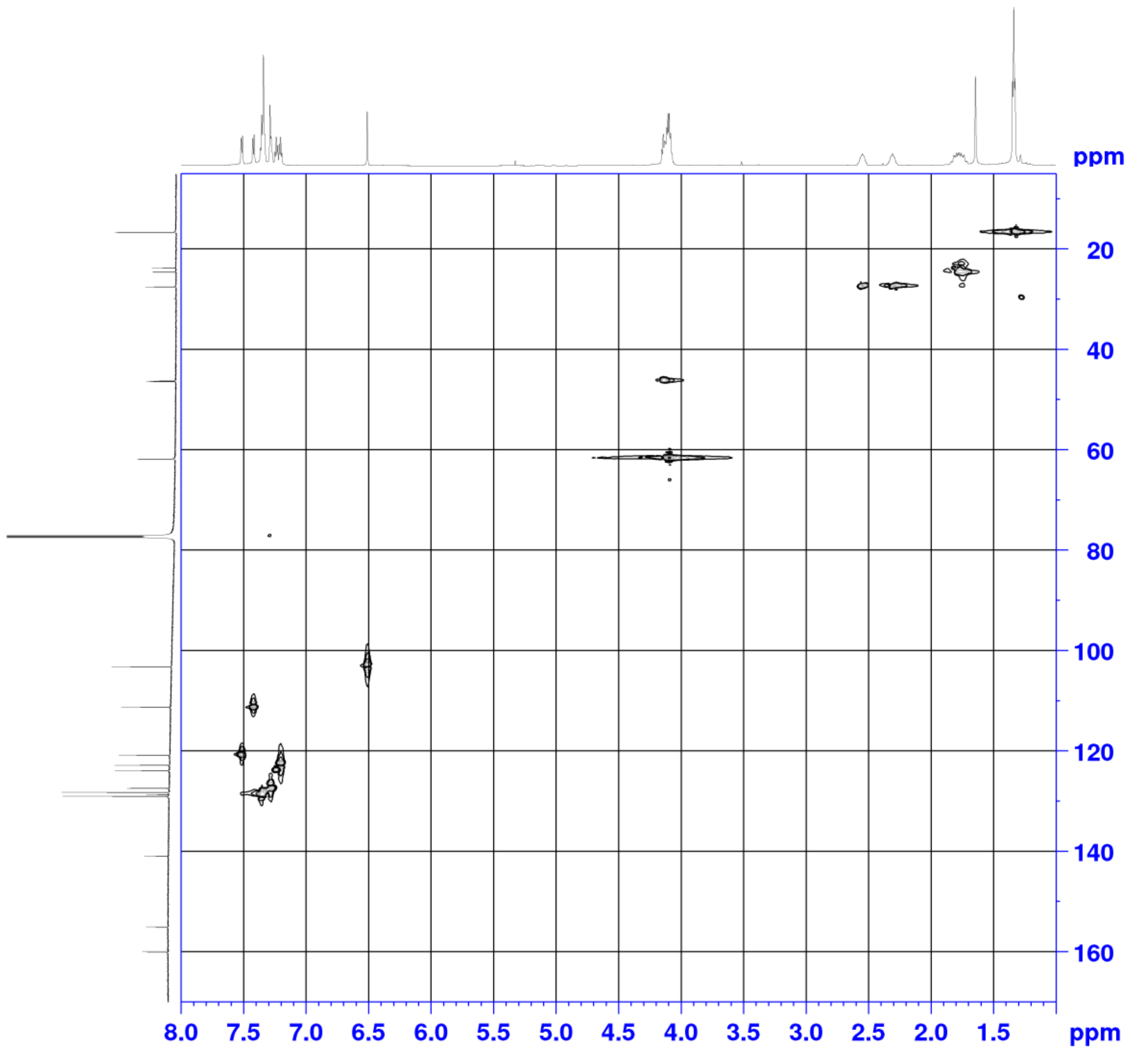
ppm
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
ppm

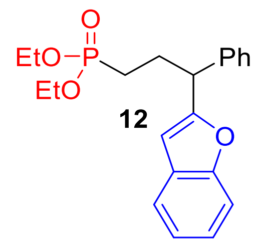


O
EtO
P
EtO
Ph
O

12

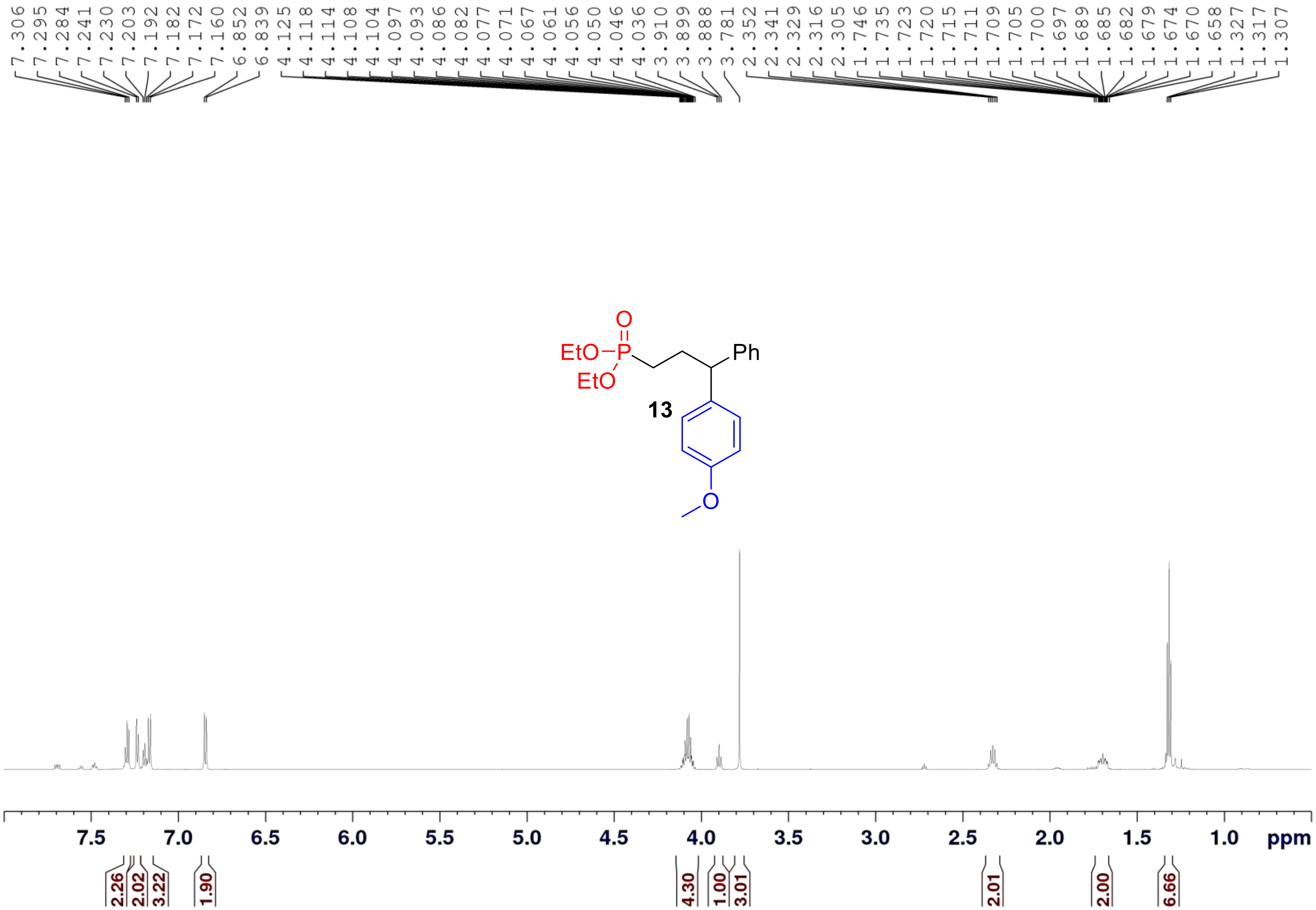
12: ^{13}C HSQC



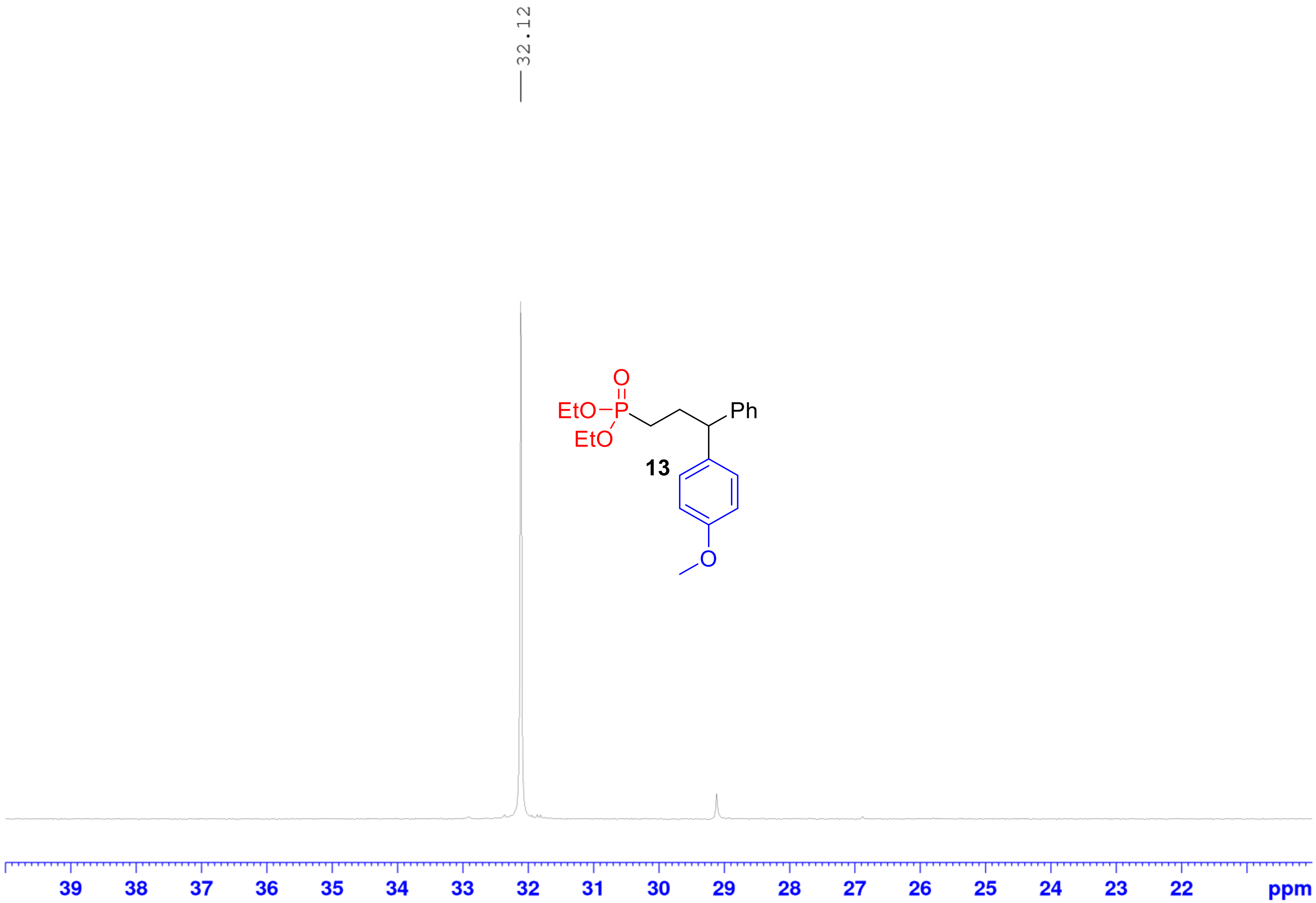


12

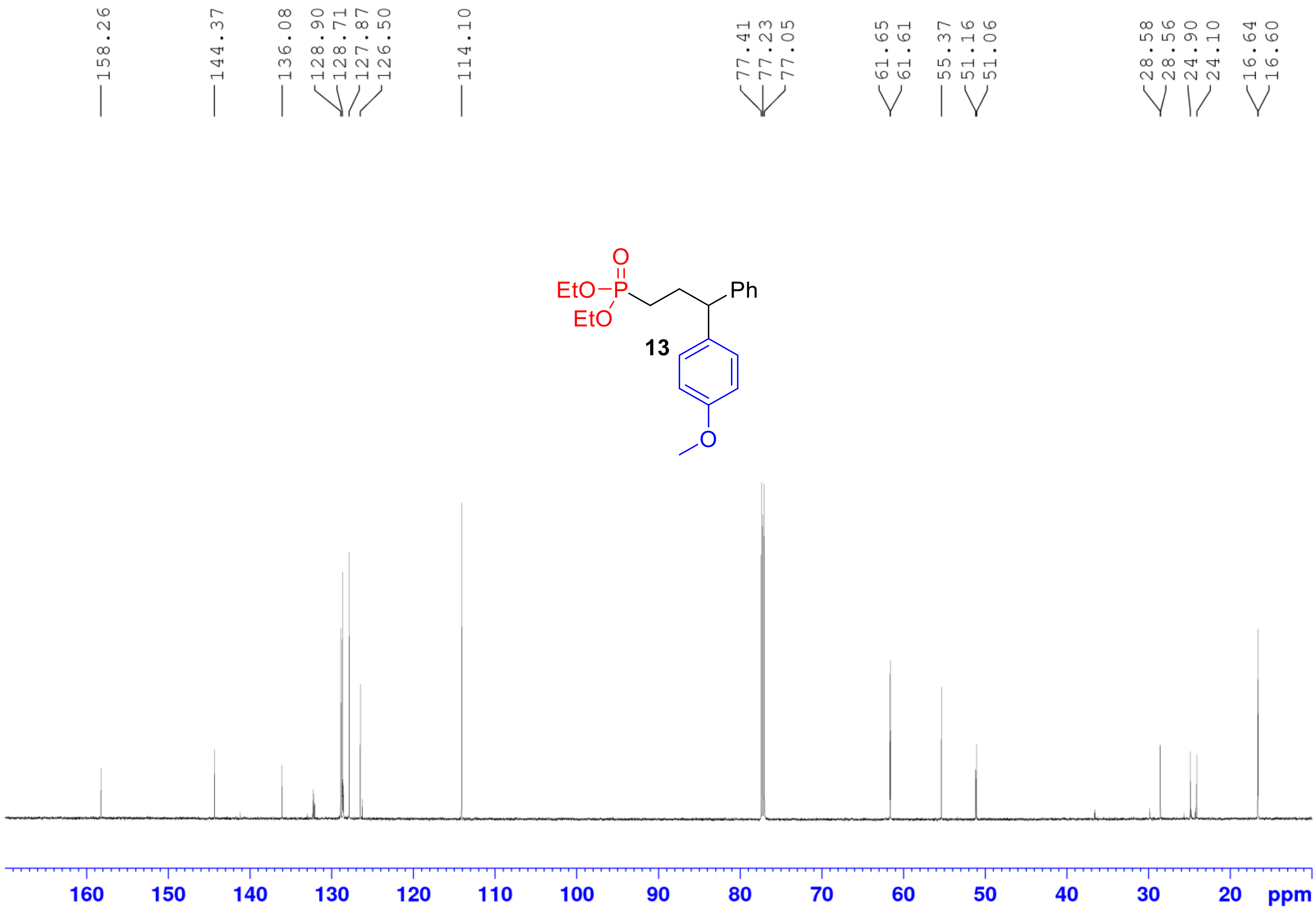
13: ^{1}H NMR


7.306
7.295
7.284
7.241
7.230
7.203
7.192
7.182
7.172
7.160
6.852
6.839
4.125
4.118
4.114
4.108
4.104
4.097
4.093
4.086
4.082
4.077
4.071
4.067
4.061
4.056
4.050
4.046
4.036
3.910
3.899
3.888
3.781
2.352
2.341
2.329
2.316
2.305
1.746
1.735
1.723
1.720
1.715
1.711
1.709
1.705
1.700
1.697
1.689
1.685
1.682
1.679
1.674
1.670
1.658
1.327
1.317
1.307
O
EtO
P
Ph
EtO
13
O
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
2.26
2.02
3.22
1.90
4.30
1.00
3.01
2.01
2.00
6.66

13: ^{31}P NMR

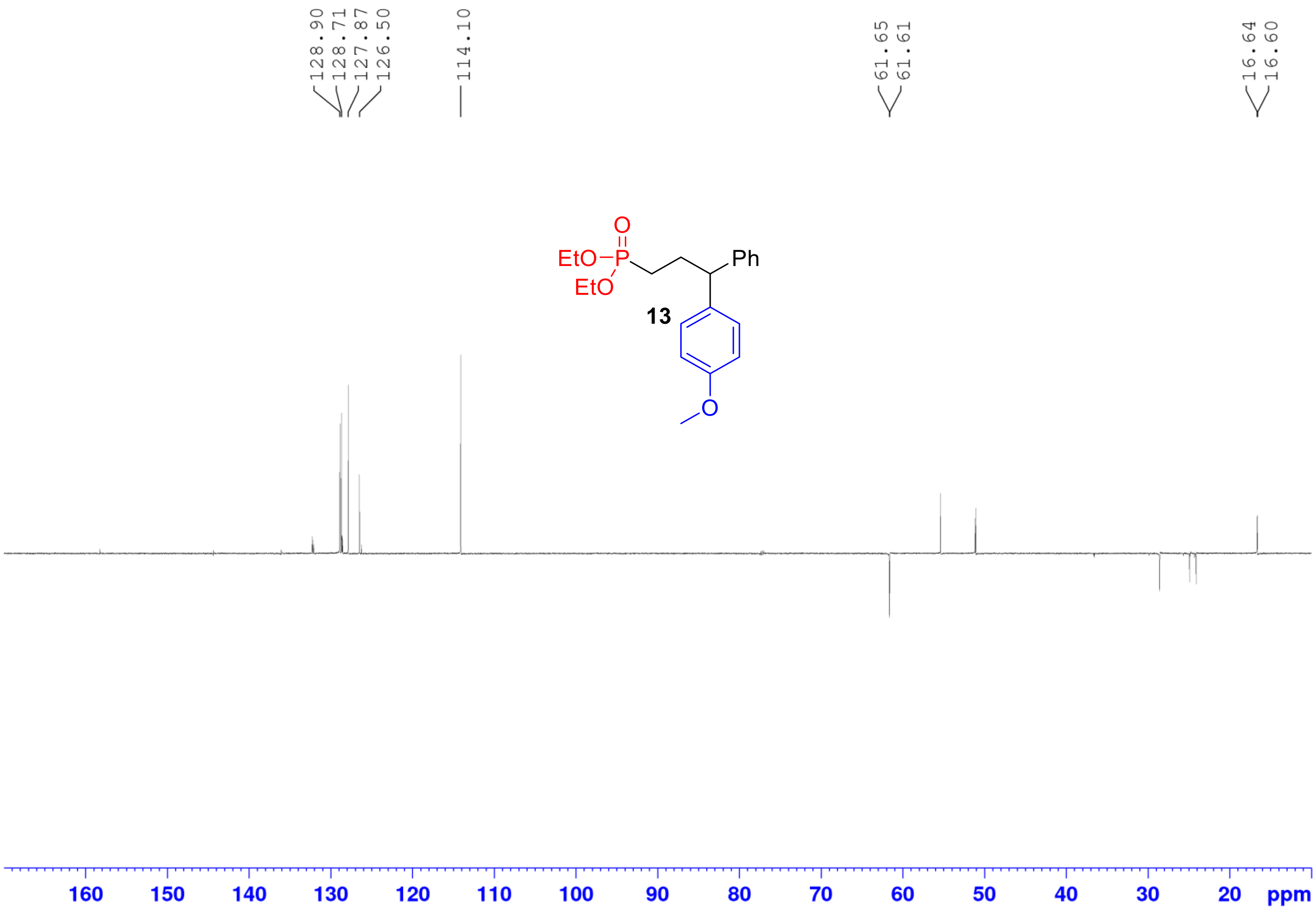

32.12
EtO
EtO
O
P
Ph
O
13
39
38
37
36
35
34
33
32
31
30
29
28
27
26
25
24
23
22
ppm

13: ^{13}C NMR

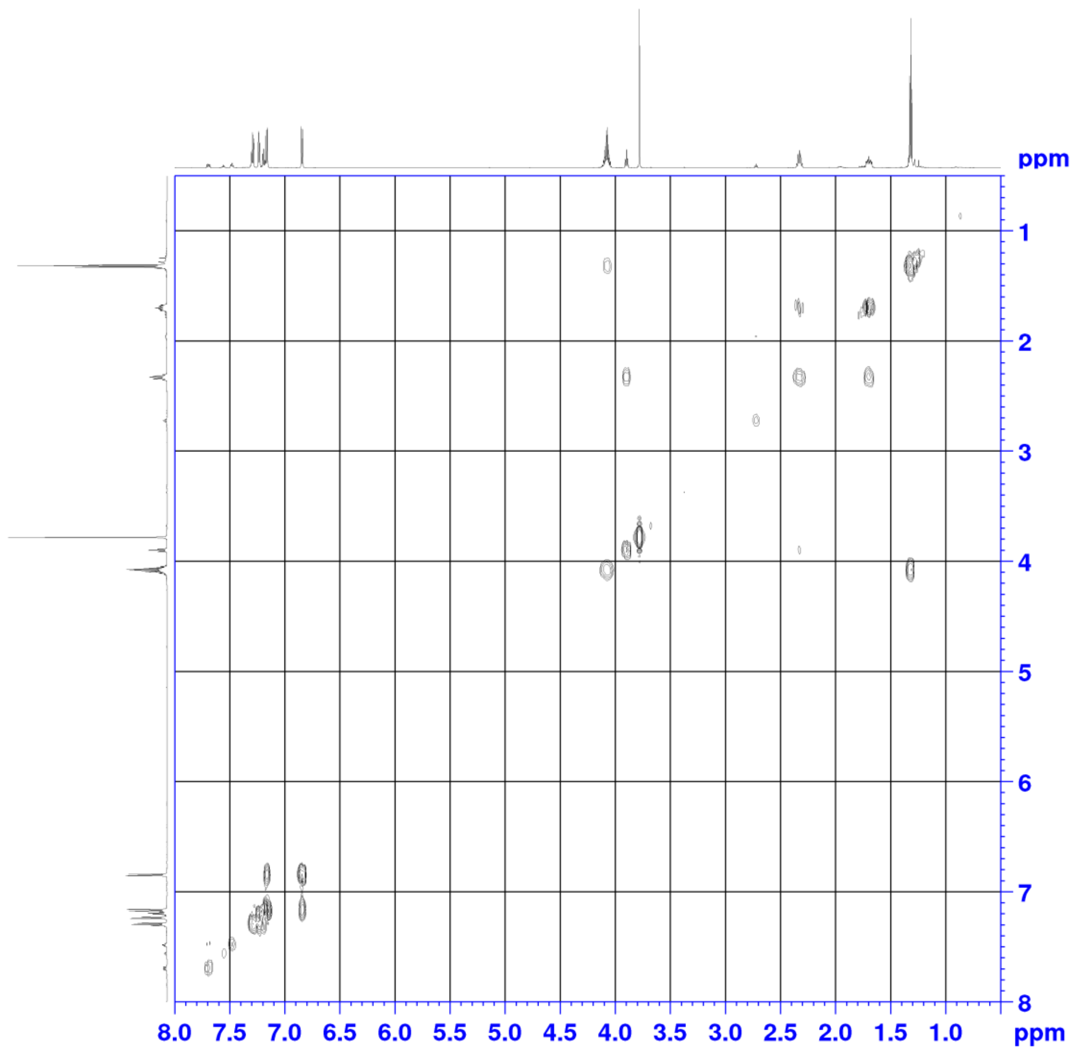


158.26
144.37
136.08
128.90
128.71
127.87
126.50
114.10
77.41
77.23
77.05
61.65
61.61
55.37
51.16
51.06
28.58
28.56
24.90
24.10
16.64
16.60
EtO
P
O
EtO
Ph
13
O
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

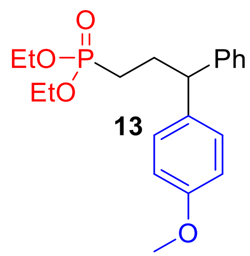
13: ^{13}C DEPT135



13: COSY



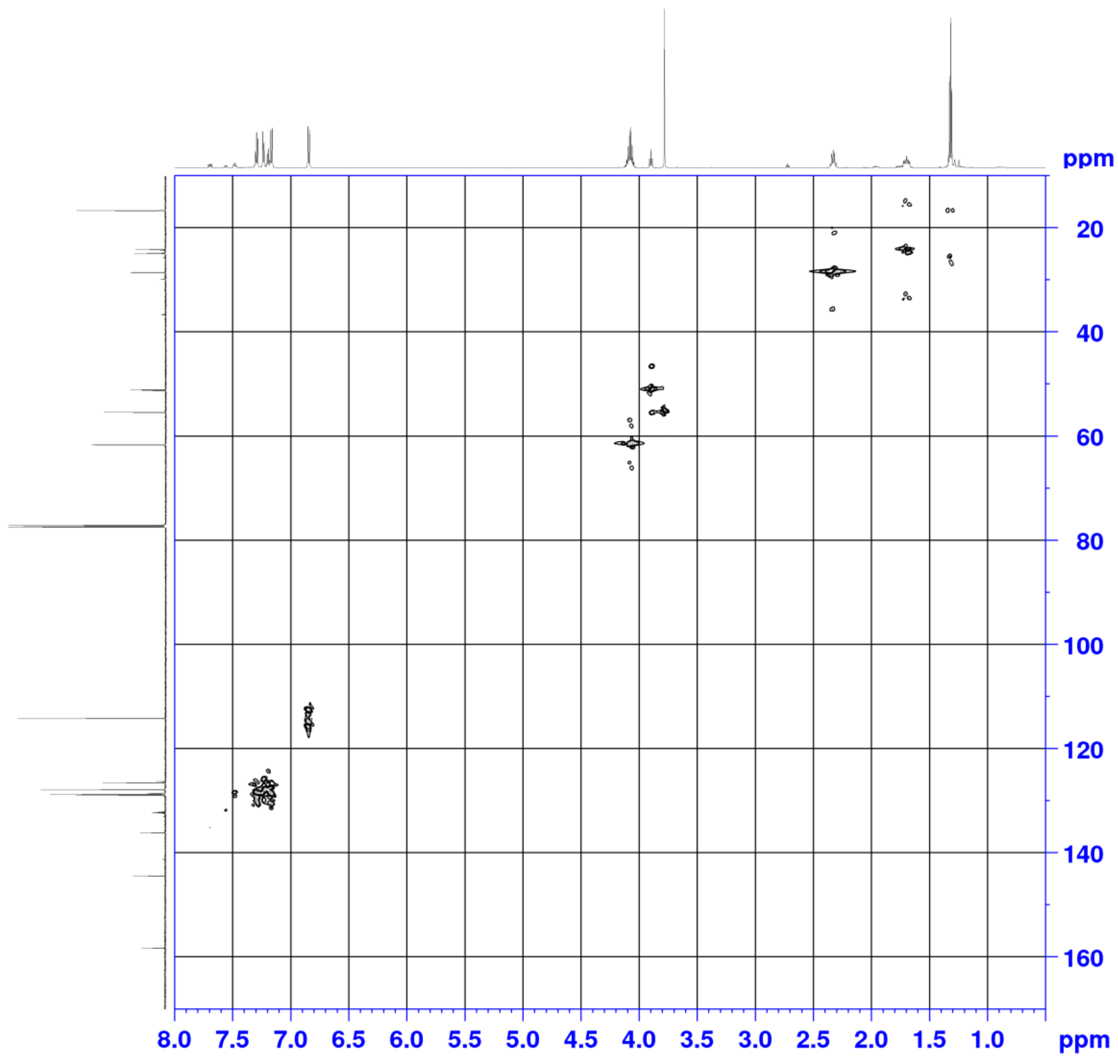
ppm
1
2
3
4
5
6
7
8
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

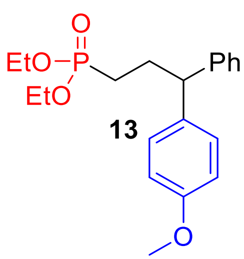


O
EtO
P
EtO
Ph
O

13

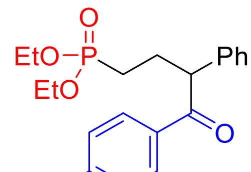
13: ^{13}C HSQC





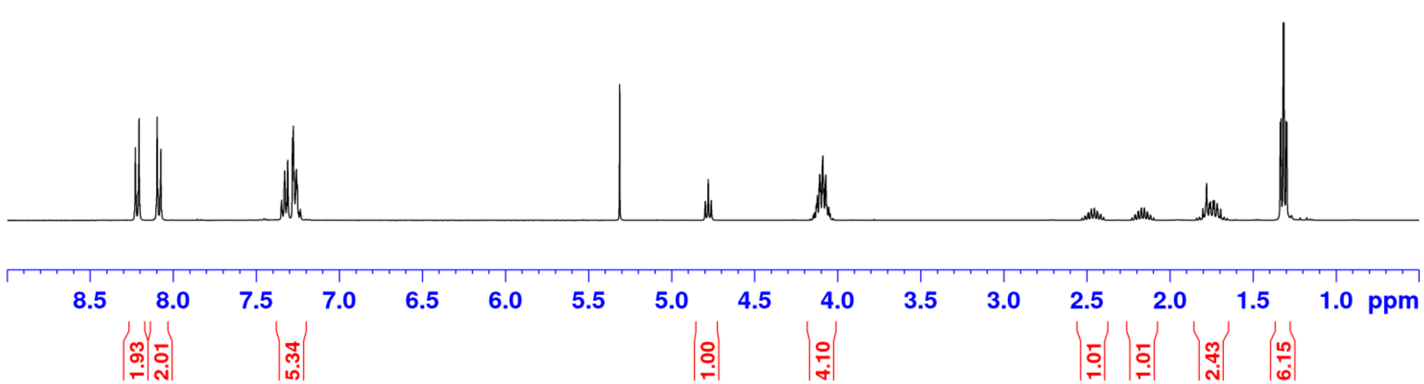
13

14: ^{1}H NMR



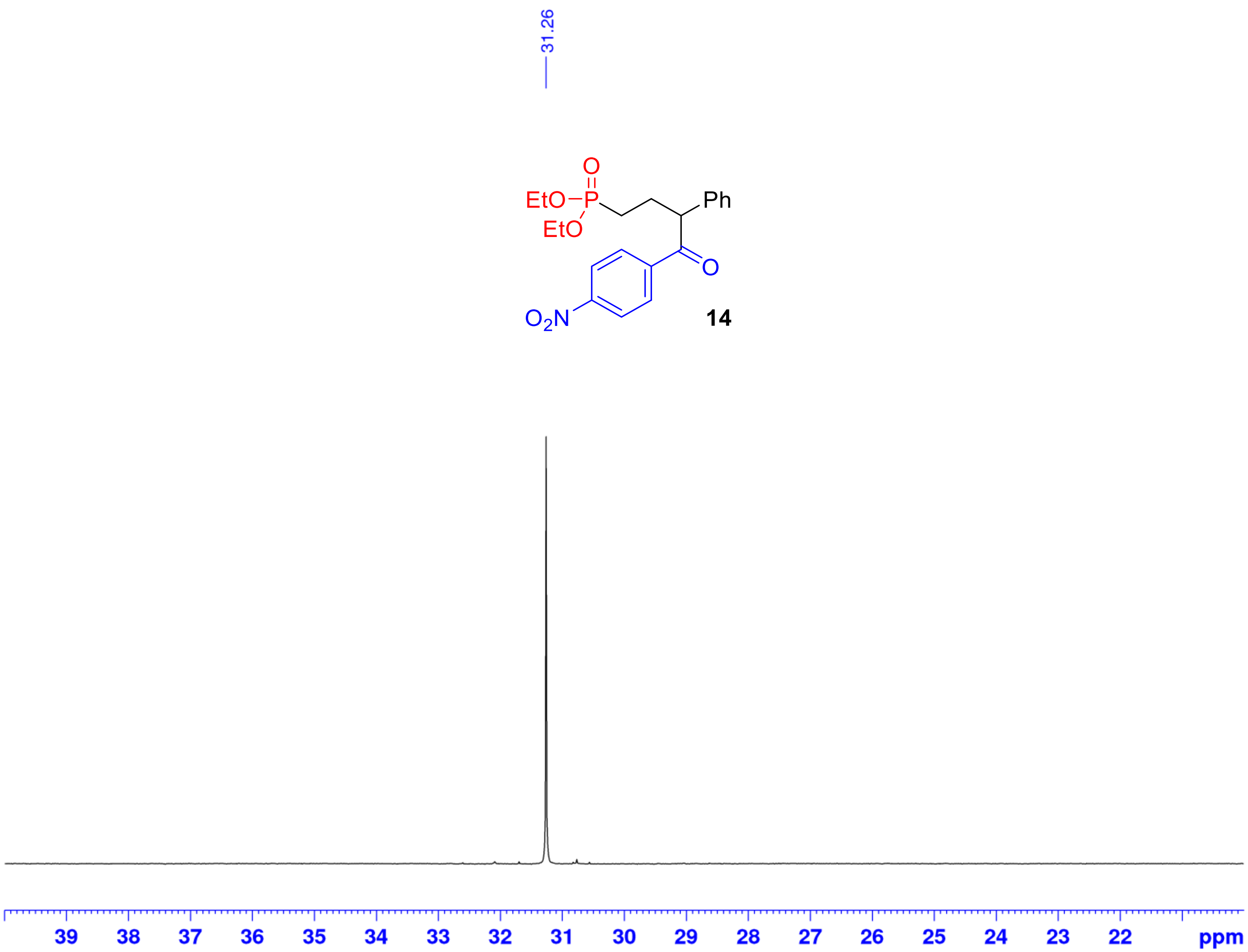
EtO
EtO
P
O
Ph
O
O_2N

14



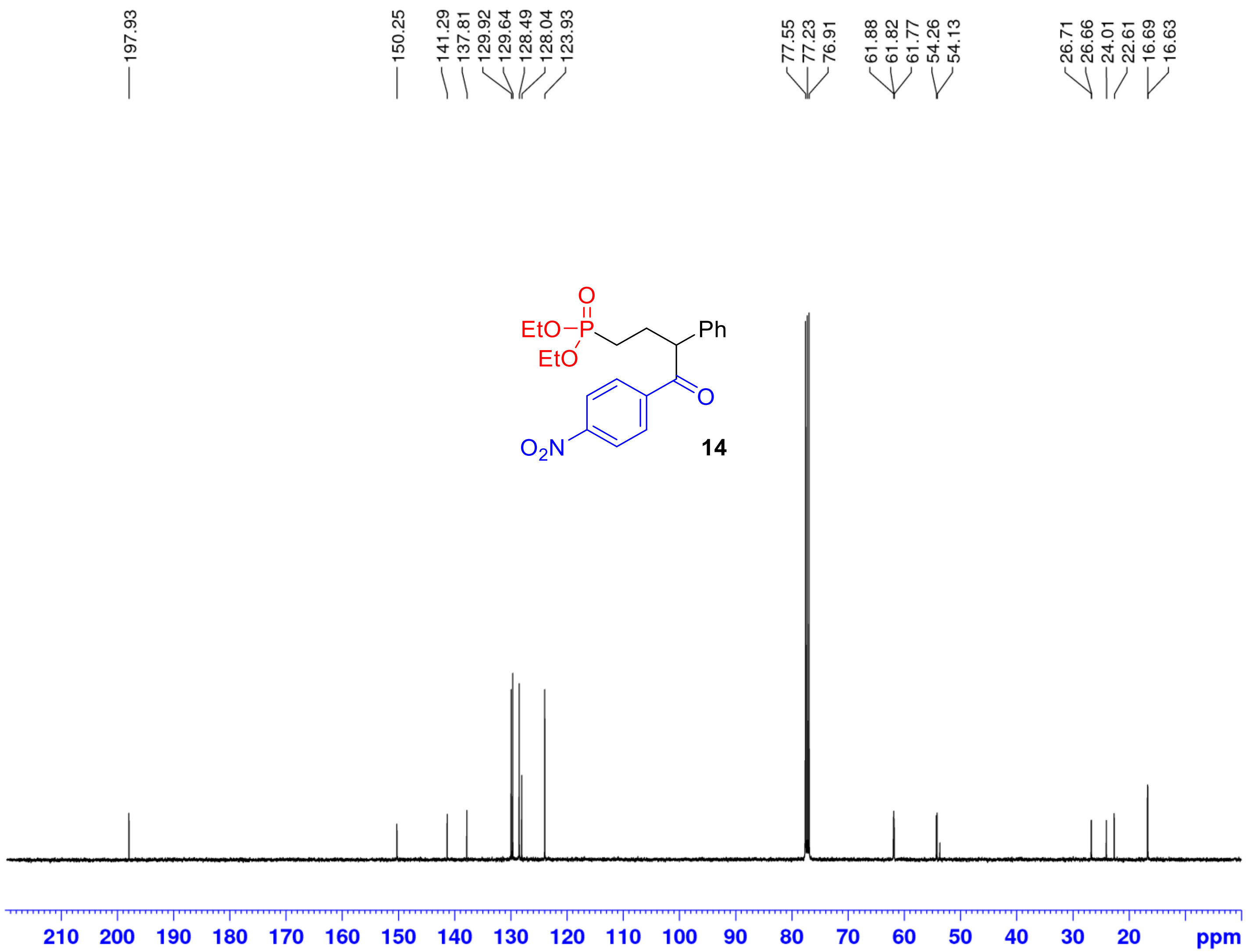
8.229
8.207
8.098
8.076
7.351
7.348
7.330
7.328
7.318
7.312
7.283
7.278
7.273
7.260
7.253
4.798
4.780
4.762
4.131
4.128
4.123
4.113
4.110
4.108
4.106
4.103
4.095
4.092
4.090
4.085
4.075
4.072
4.067
4.065
4.058
4.055
2.456
2.173
2.155
1.802
1.785
1.780
1.763
1.757
1.740
1.734
1.717
1.712
1.336
1.332
1.319
1.314
1.301
1.297
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
ppm
1.93
2.01
5.34
1.00
4.10
1.01
1.01
2.43
6.15

14: ^{31}P NMR



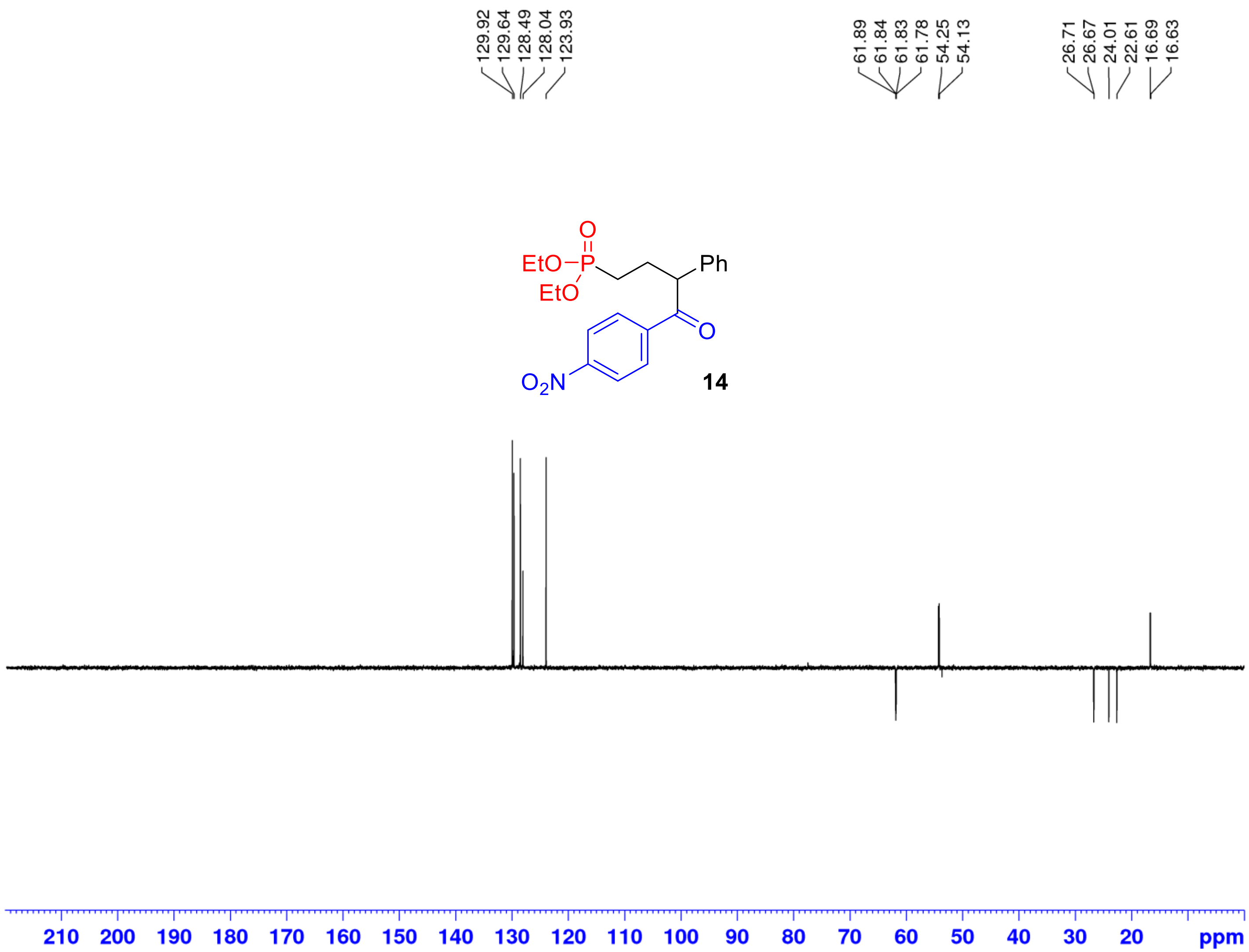
31.26
O
EtO
P
EtO
Ph
O
O2N
14
39
38
37
36
35
34
33
32
31
30
29
28
27
26
25
24
23
22
ppm

14: ^{13}C NMR

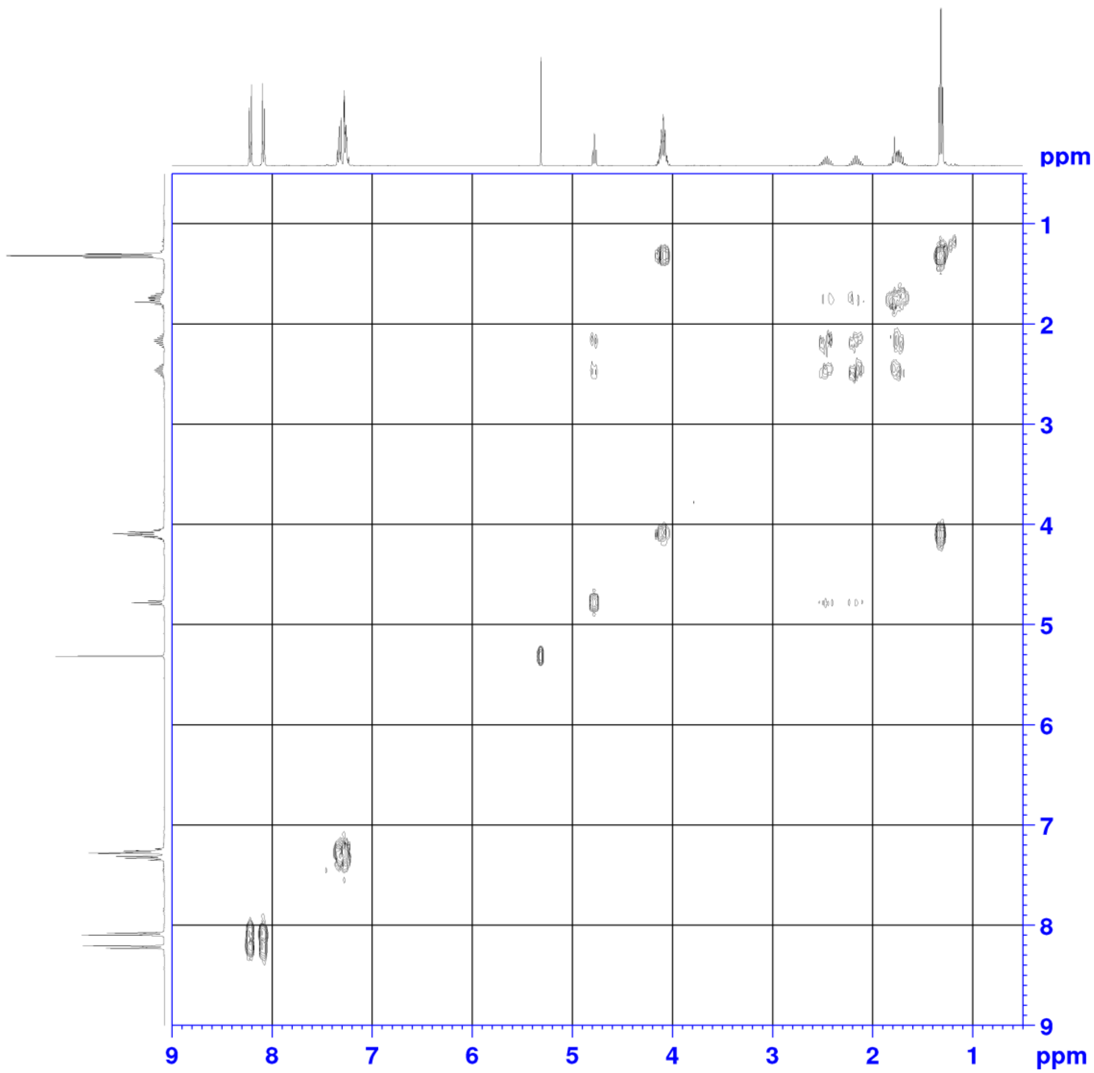


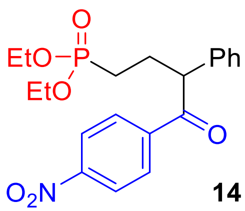
197.93
150.25
141.29
137.81
129.92
129.64
128.49
128.04
123.93
77.55
77.23
76.91
61.88
61.82
61.77
54.26
54.13
26.71
26.66
24.01
22.61
16.69
16.63
O
EtO
P
Ph
EtO
O
O2N
14
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

14: ^{13}C DEPT135



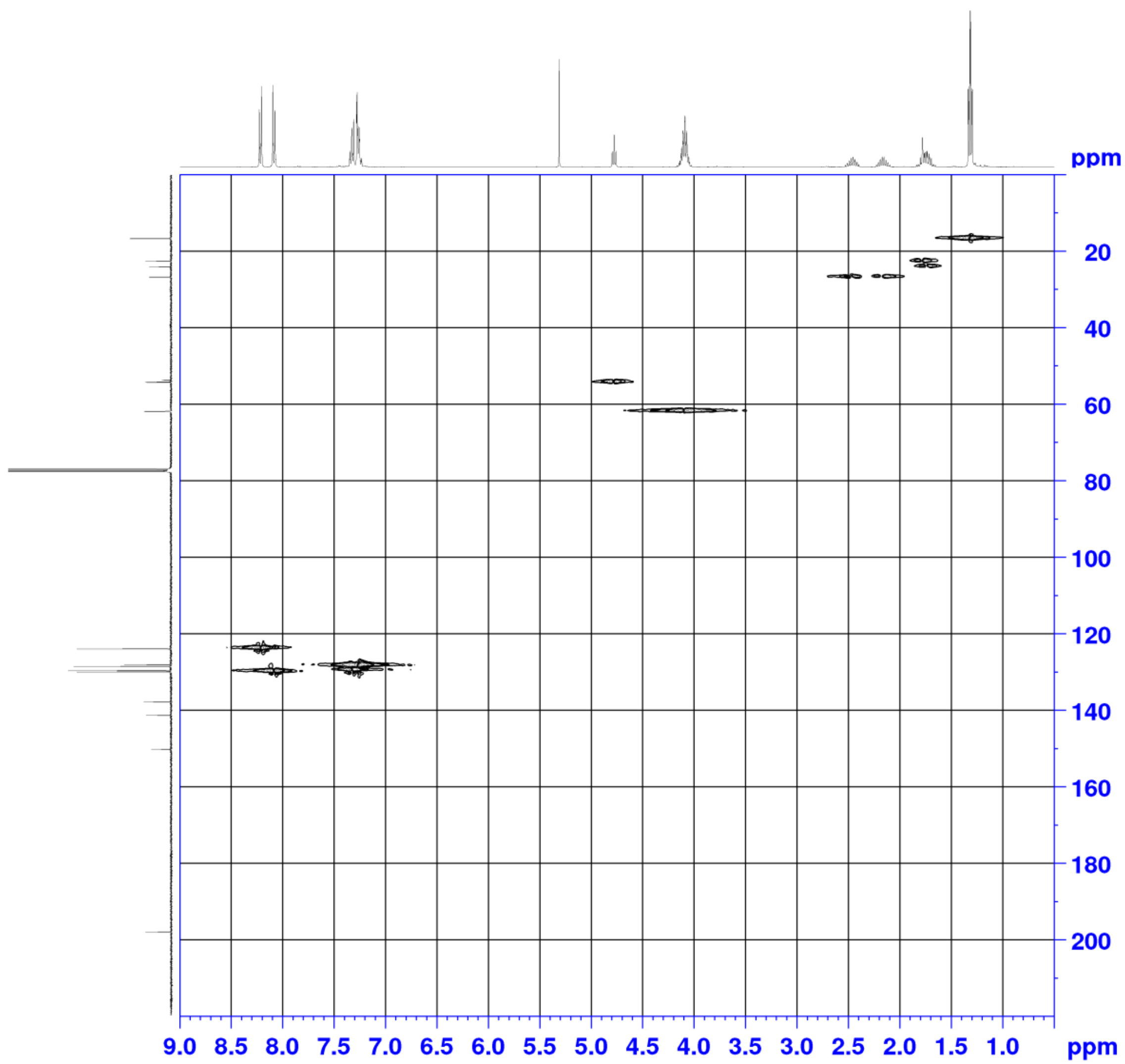
14: COSY

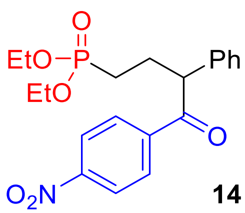




14

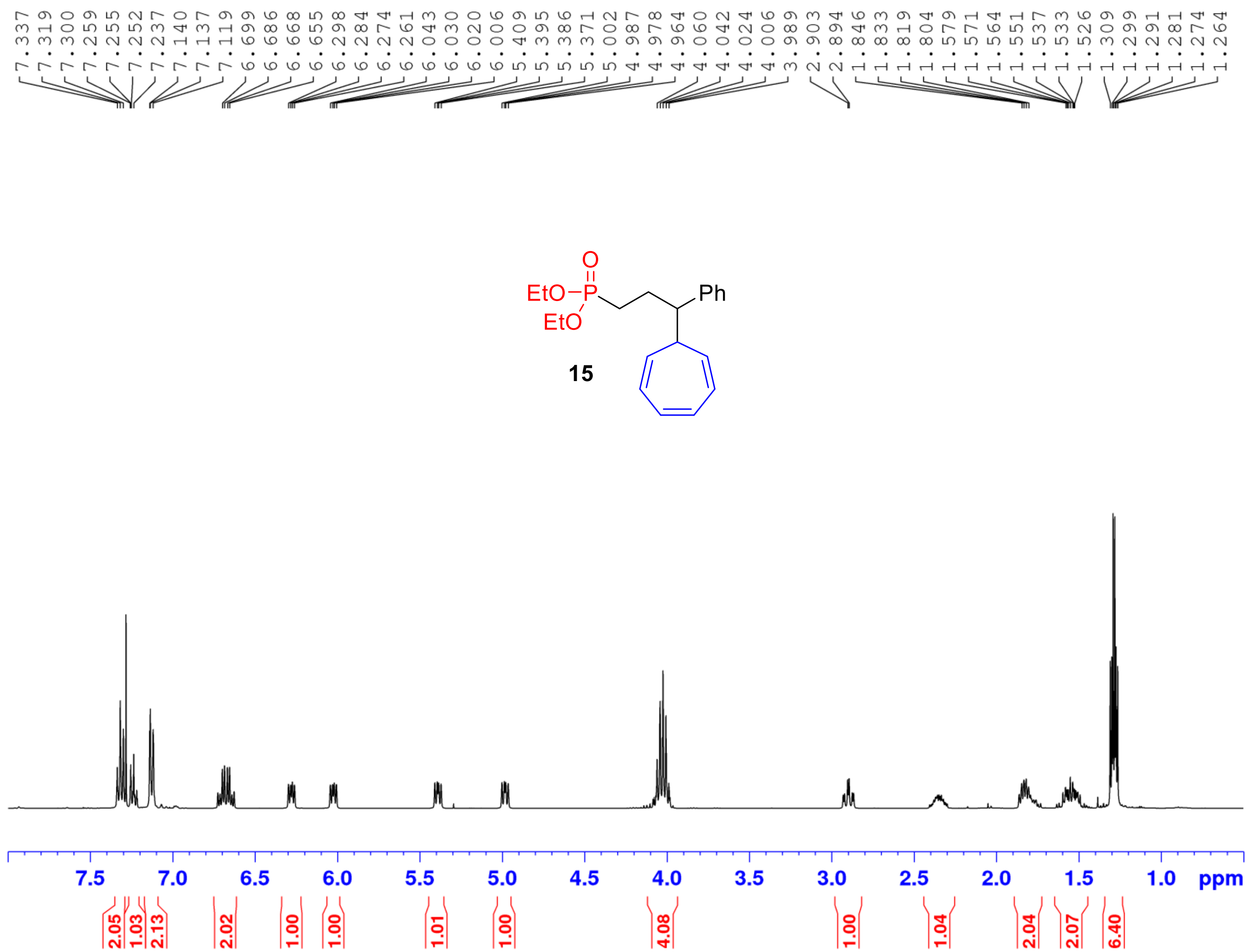
14: ^{13}C HSQC





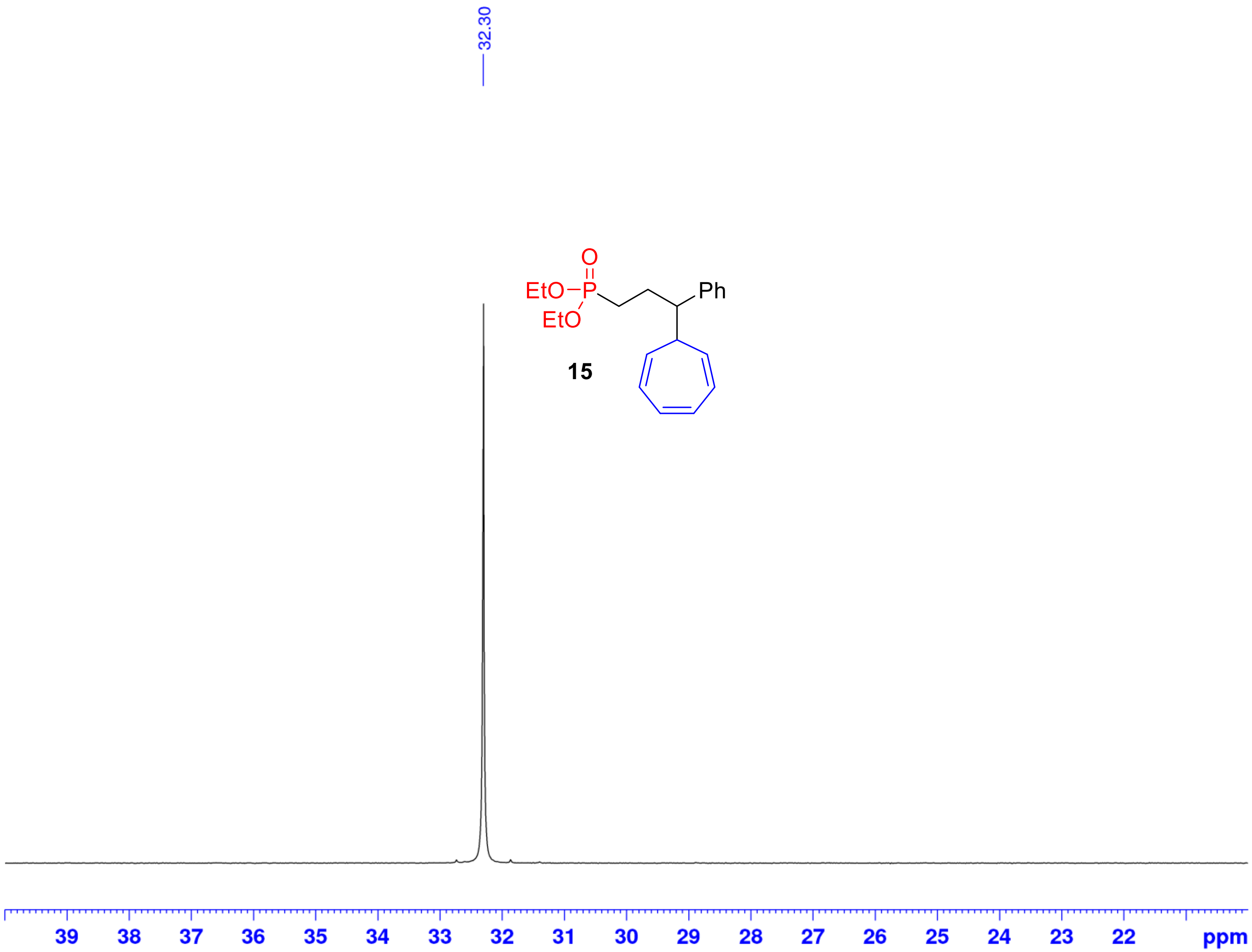
14

15: ^{1}H NMR

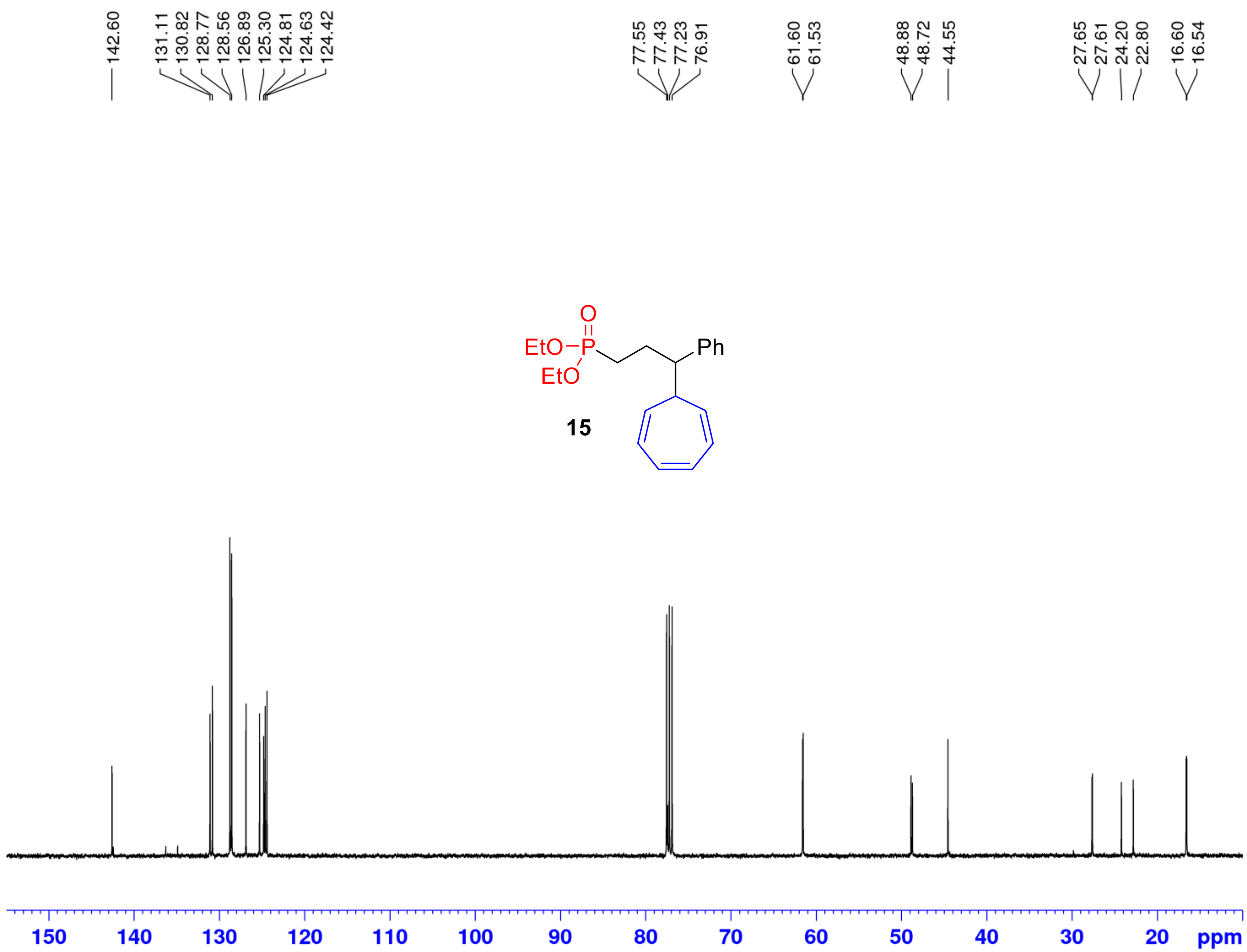


7.337 7.319 7.300 7.259 7.255 7.252 7.237 7.140 7.137 7.119 6.699 6.686 6.668 6.655 6.298 6.284 6.274 6.261 6.043 6.030 6.020 6.006 5.409 5.395 5.386 5.371 5.002 4.987 4.978 4.964 4.060 4.042 4.024 4.006 3.989 2.903 2.894 1.846 1.833 1.819 1.804 1.579 1.571 1.564 1.551 1.537 1.533 1.526 1.309 1.299 1.291 1.281 1.274 1.264
EtO
EtO
O
P
Ph
15
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
2.05 1.03 2.13 2.02 1.00 1.00 1.01 1.00 4.08 1.00 1.04 2.04 2.07 6.40

15: ^{31}P NMR

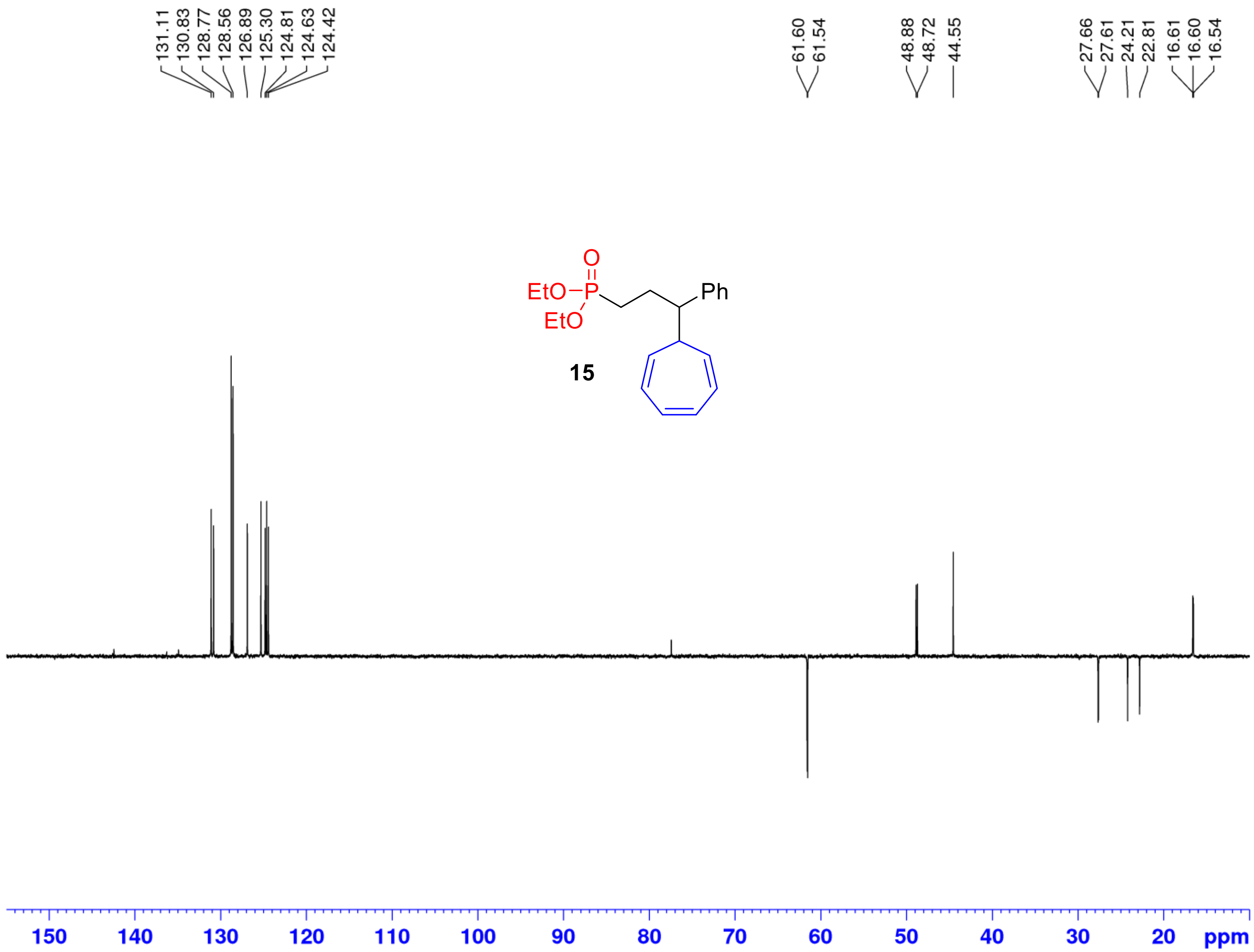

32.30
O
EtO
P
EtO
Ph
15
39
38
37
36
35
34
33
32
31
30
29
28
27
26
25
24
23
22
ppm

15: ^{13}C NMR

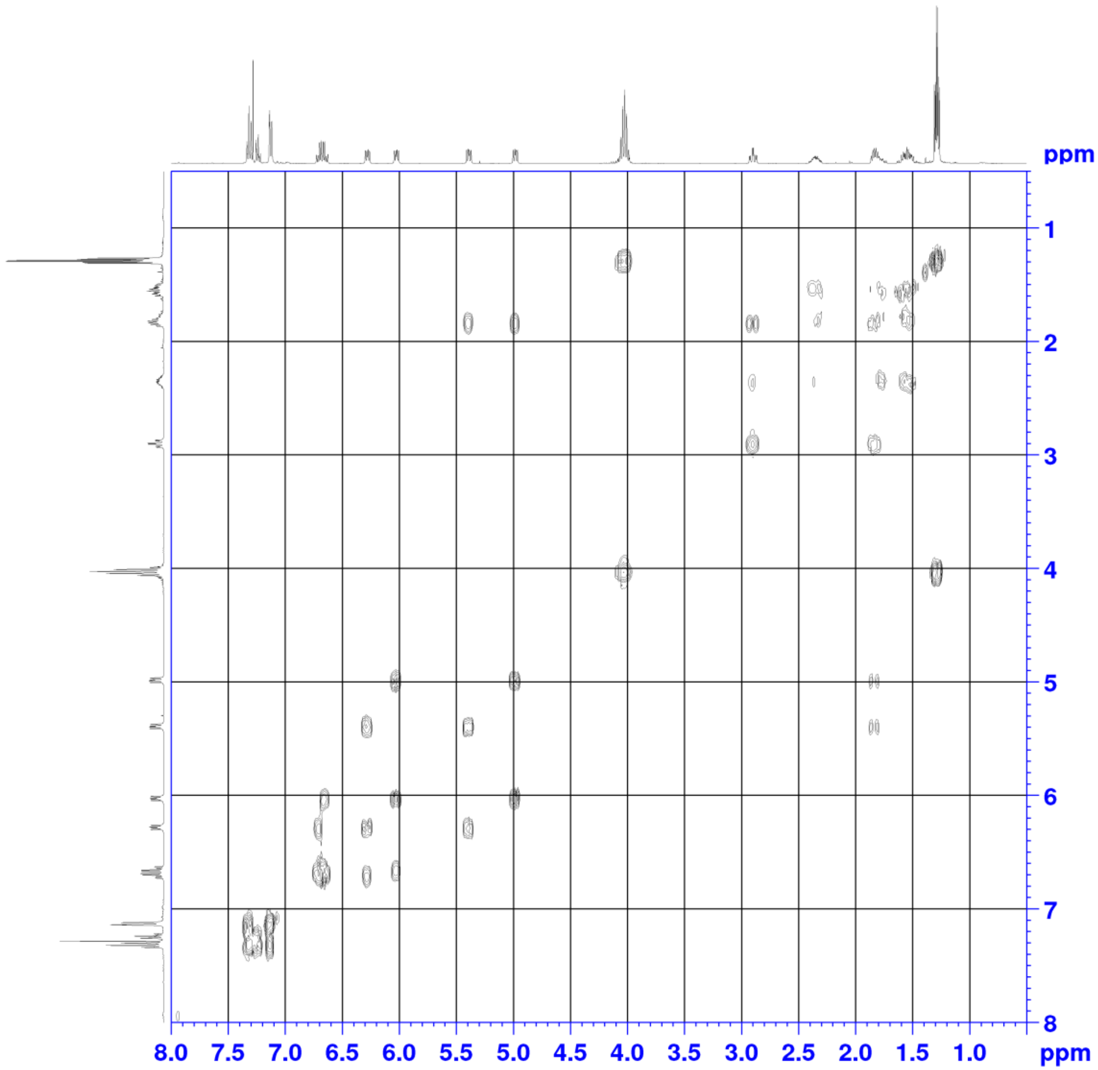


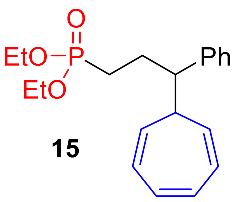
142.60
131.11
130.82
128.77
128.56
126.89
125.30
124.81
124.63
124.42
77.55
77.43
77.23
76.91
61.60
61.53
48.88
48.72
44.55
27.65
27.61
24.20
22.80
16.60
16.54
O
EtO
P
EtO
Ph
15
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

15: ^{13}C DEPT135



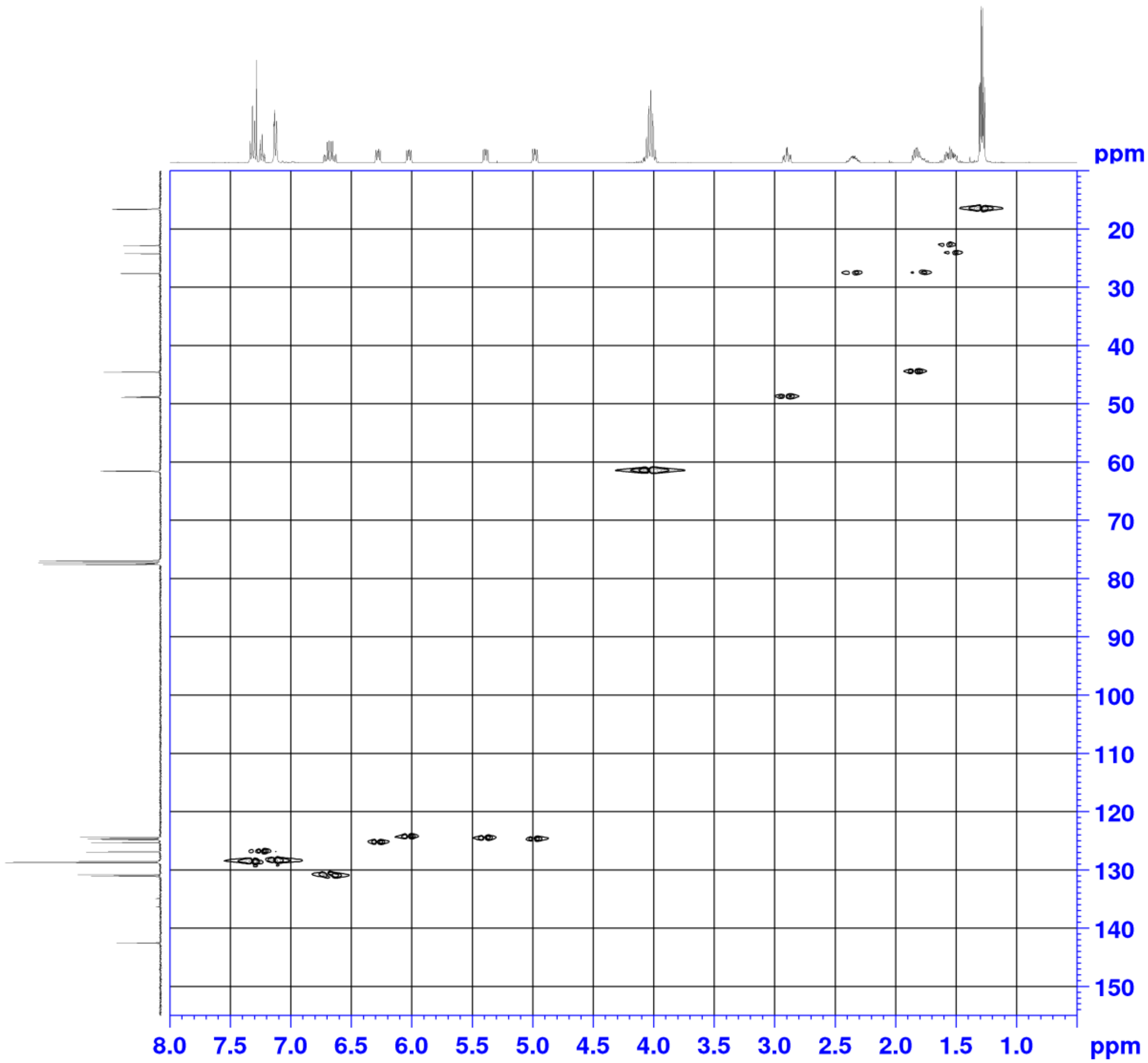
15: COSY

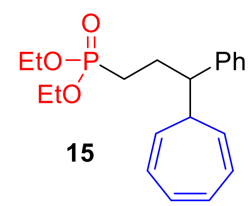

ppm
1
2
3
4
5
6
7
8
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0
ppm


O
EtO
P
Ph
EtO

15

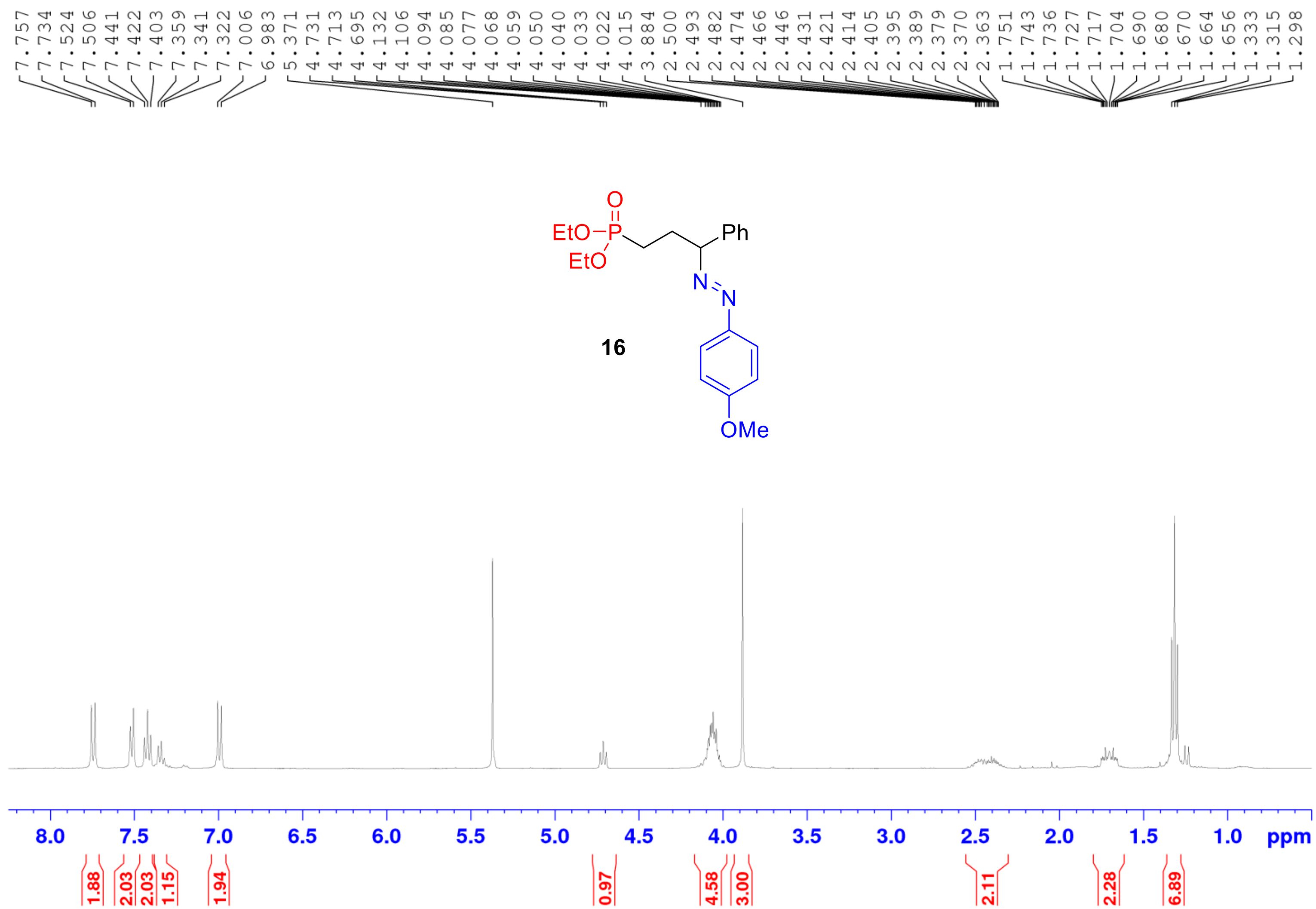
15: ^{13}C HSQC





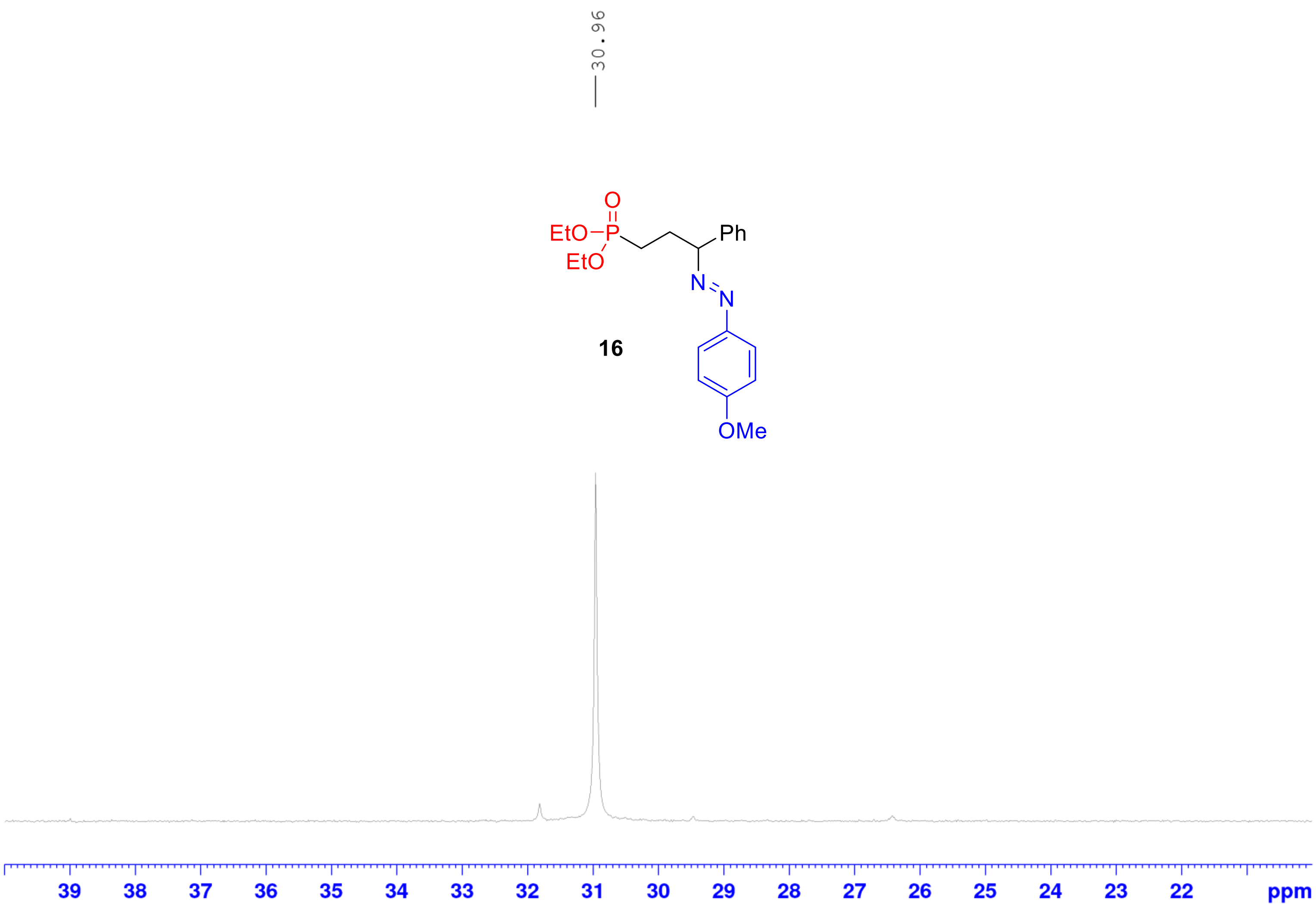
15

16: ^{1}H NMR



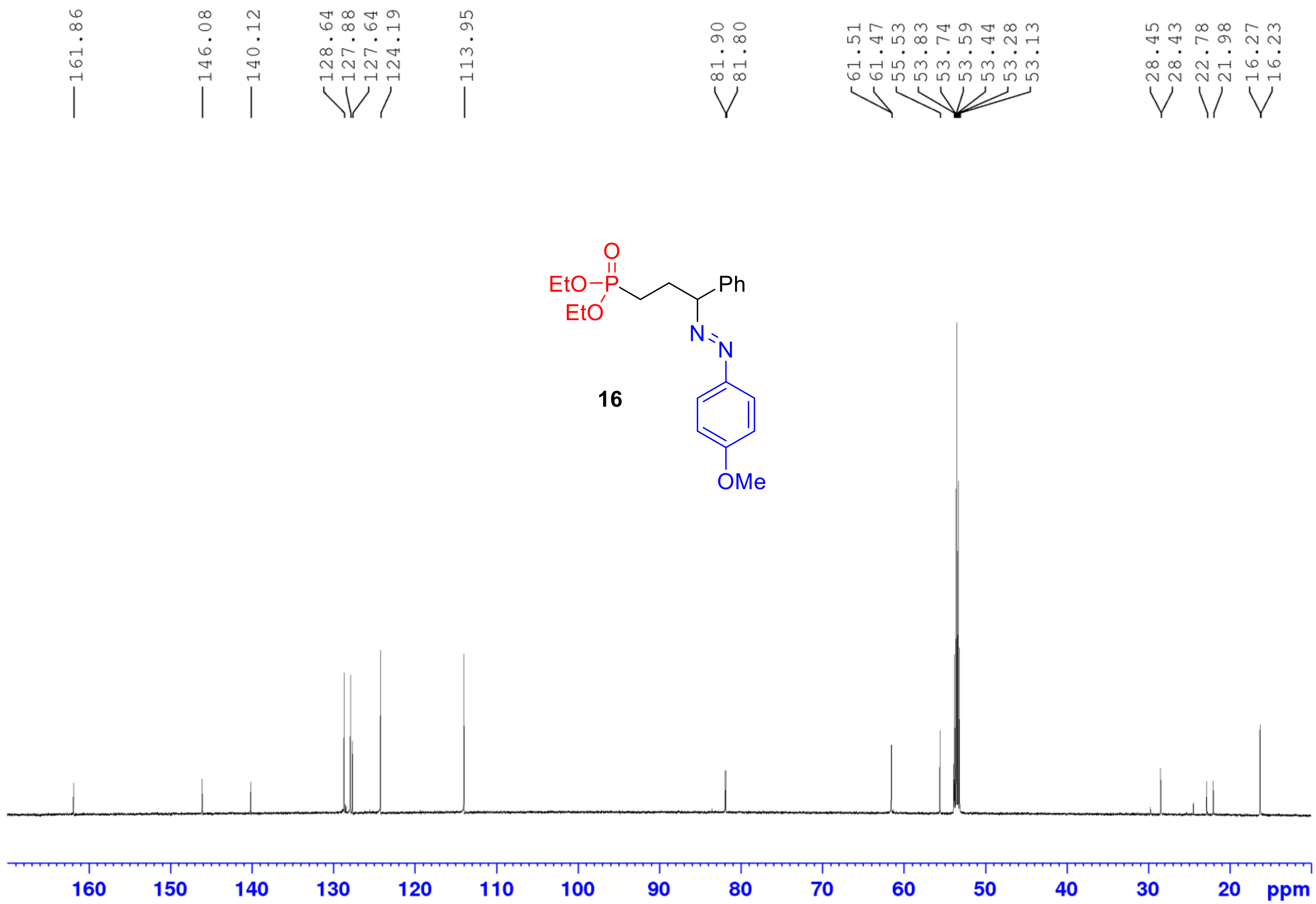
7.757 7.734 7.524 7.506 7.441 7.422 7.403 7.359 7.341 7.322 7.006 6.983 5.371 4.731 4.713 4.695 4.132 4.106 4.094 4.085 4.077 4.068 4.059 4.050 4.040 4.033 4.022 4.015 3.884 2.500 2.493 2.482 2.474 2.466 2.446 2.431 2.421 2.414 2.405 2.395 2.389 2.379 2.370 2.363 1.751 1.743 1.736 1.727 1.717 1.704 1.690 1.680 1.670 1.664 1.656 1.333 1.315 1.298
O
EtO
P
EtO
Ph
N
N
16
OMe
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 ppm
1.88 2.03 2.03 1.15 1.94 0.97 4.58 3.00 2.11 2.28 6.89

16: ^{31}P NMR

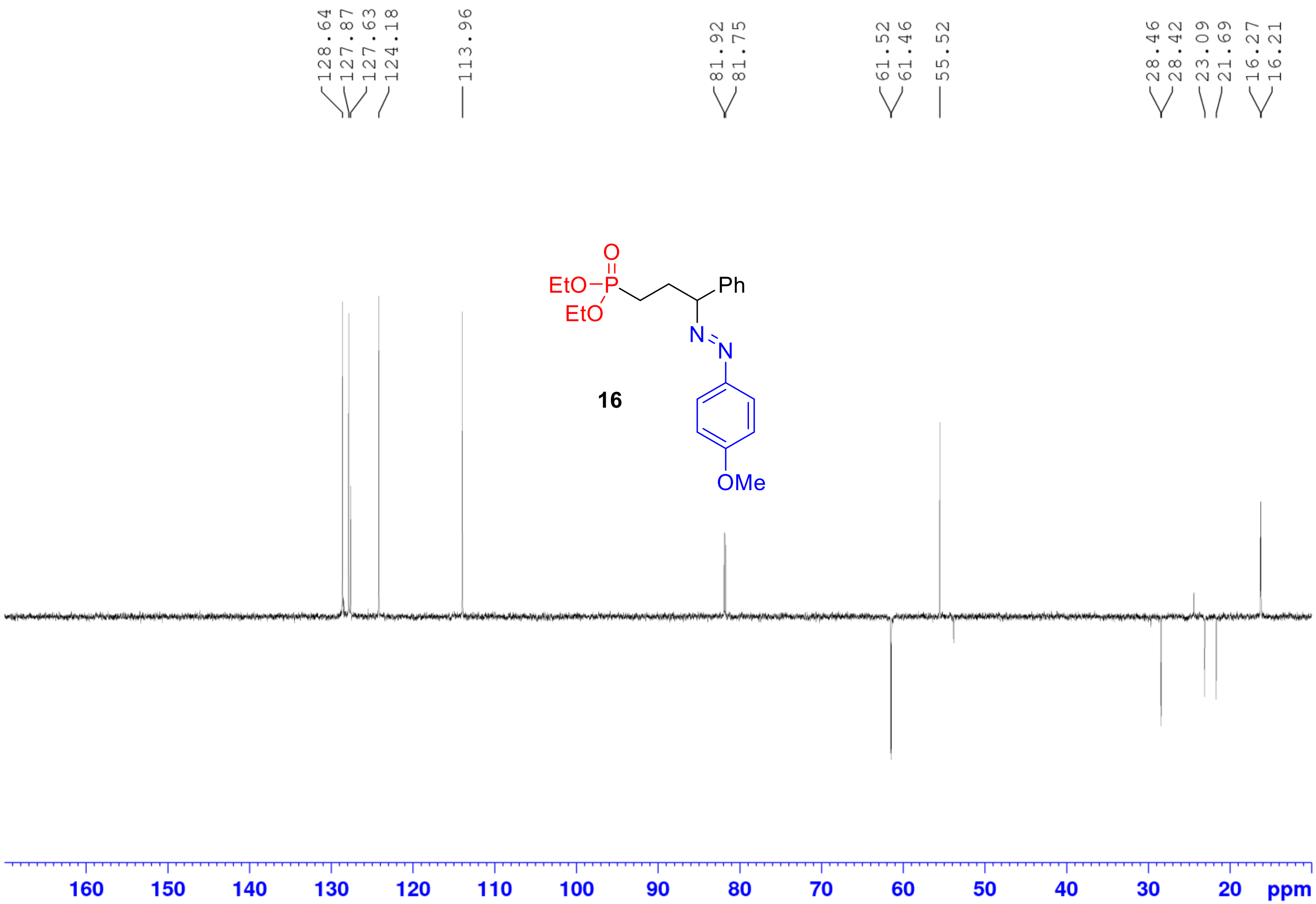


30.96
EtO
P
O
EtO
Ph
N
N
OMe
16
39
38
37
36
35
34
33
32
31
30
29
28
27
26
25
24
23
22
ppm

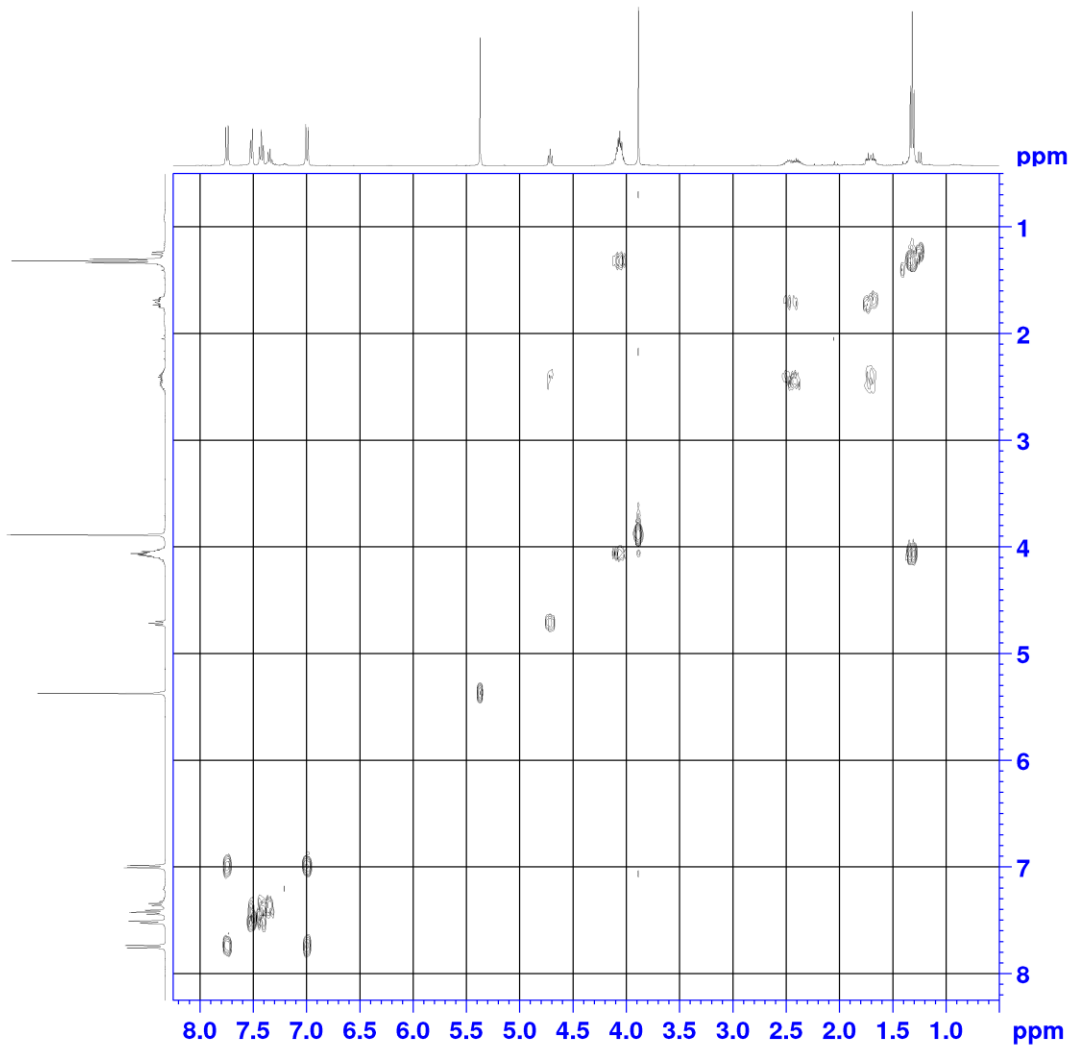
16: ^{13}C NMR

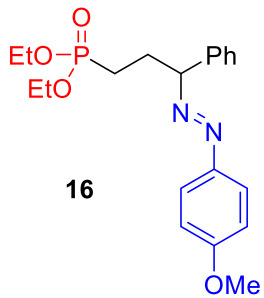

161.86
146.08
140.12
128.64
127.88
127.64
124.19
113.95
81.90
81.80
61.51
61.47
55.53
53.83
53.74
53.59
53.44
53.28
53.13
28.45
28.43
22.78
21.98
16.27
16.23
O
EtO
P
EtO
Ph
N
N
16
OMe
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

16: ^{13}C DEPT135



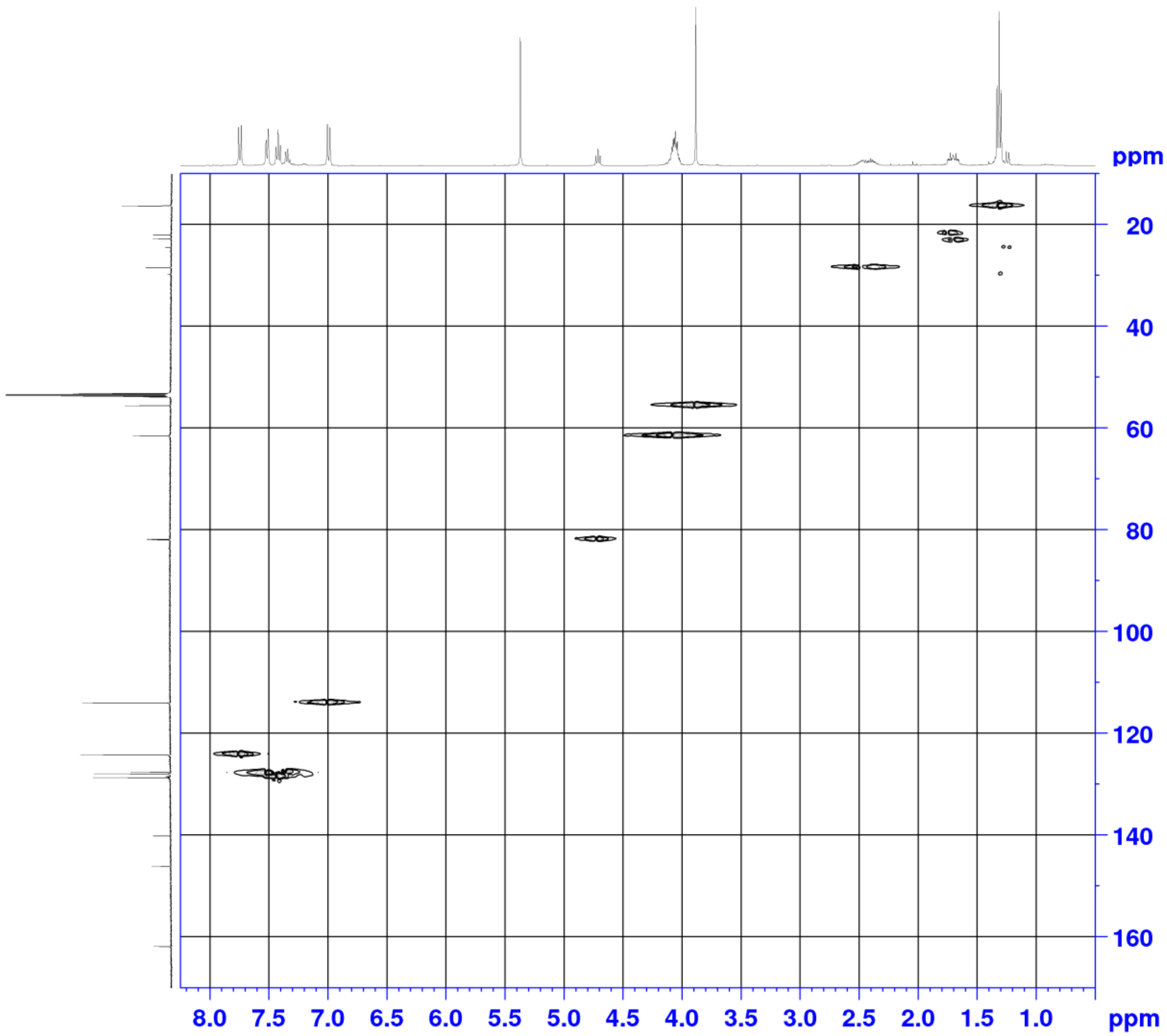
16: COSY

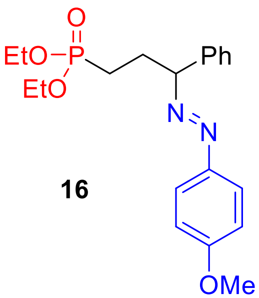

ppm
1
2
3
4
5
6
7
8
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0
ppm


O
EtO
P
EtO
Ph
N
N
OMe

16

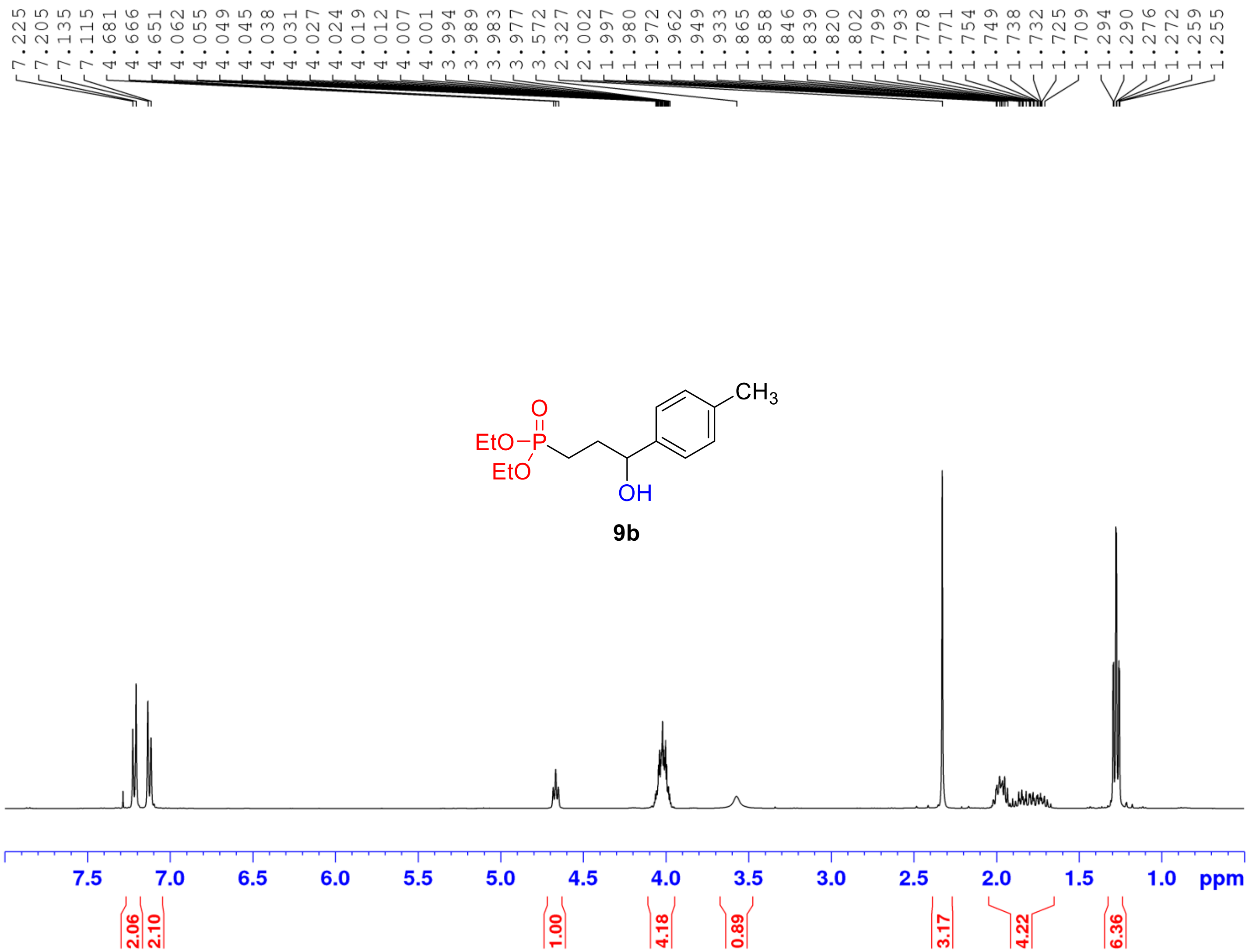
16: ^{13}C HSQC





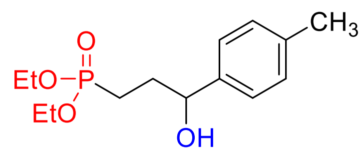
16

9b: [1]H NMR



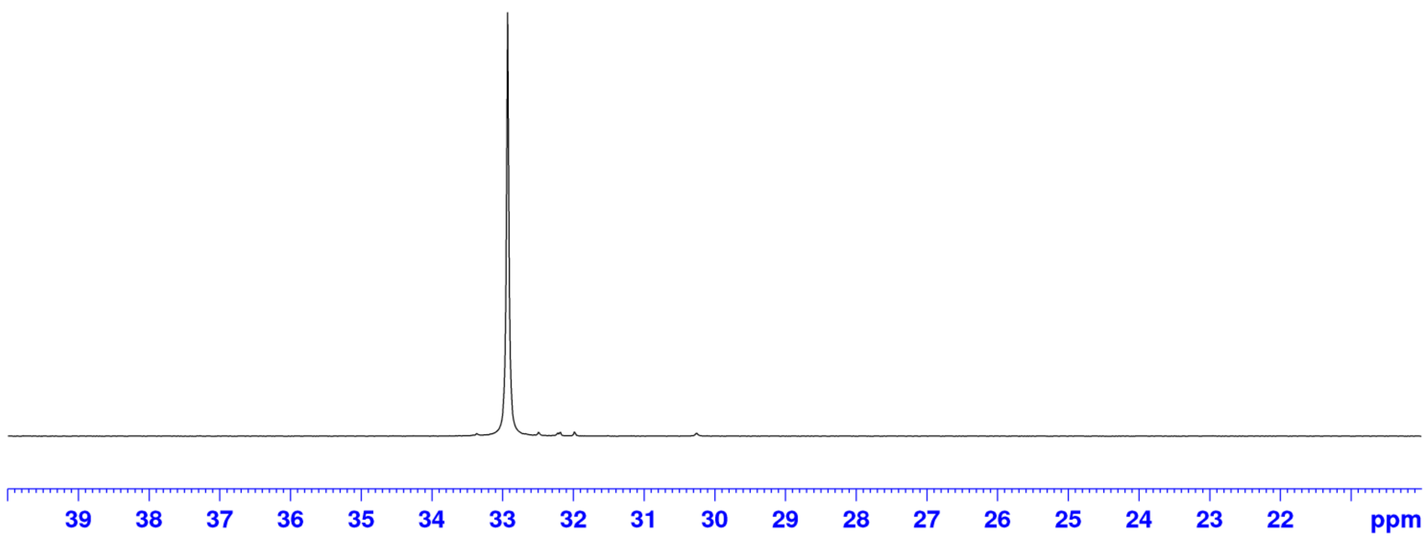
7.225
7.205
7.135
7.115
4.681
4.666
4.651
4.062
4.055
4.049
4.045
4.038
4.031
4.027
4.024
4.019
4.012
4.007
4.001
3.994
3.989
3.983
3.977
3.572
2.327
2.002
1.997
1.980
1.972
1.962
1.949
1.933
1.865
1.858
1.846
1.839
1.820
1.802
1.799
1.793
1.778
1.771
1.754
1.749
1.738
1.732
1.725
1.709
1.294
1.290
1.276
1.272
1.259
1.255
CH3
O
EtO
P
EtO
OH
9b
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
2.06
2.10
1.00
4.18
0.89
3.17
4.22
6.36

9b: ^{31}P NMR



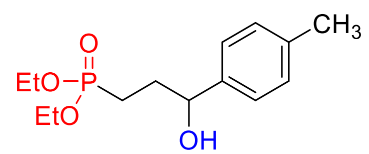
O
EtO
P
EtO
OH
CH3

9b

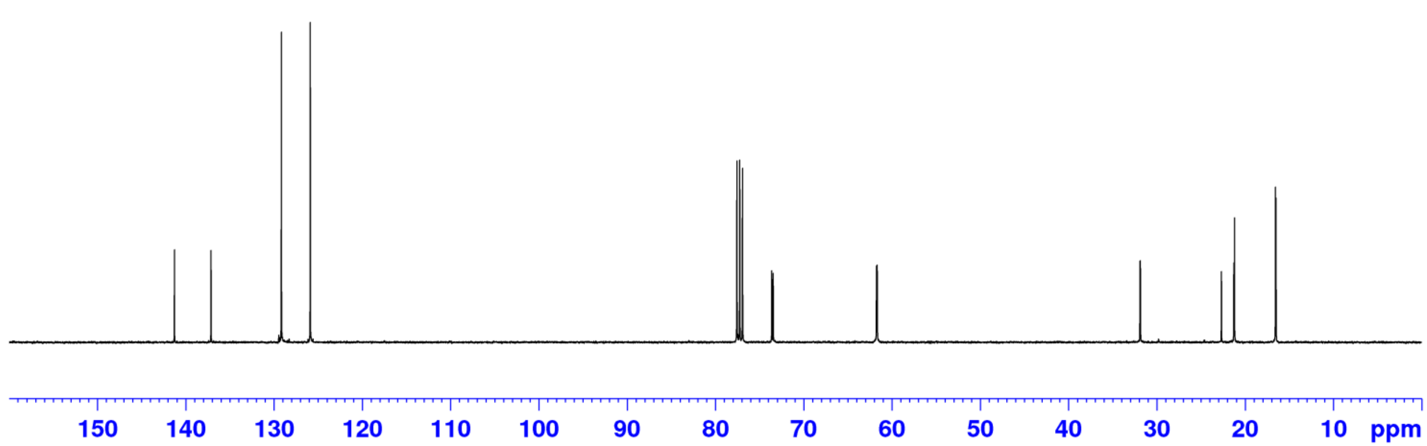


32.93
39
38
37
36
35
34
33
32
31
30
29
28
27
26
25
24
23
22
ppm

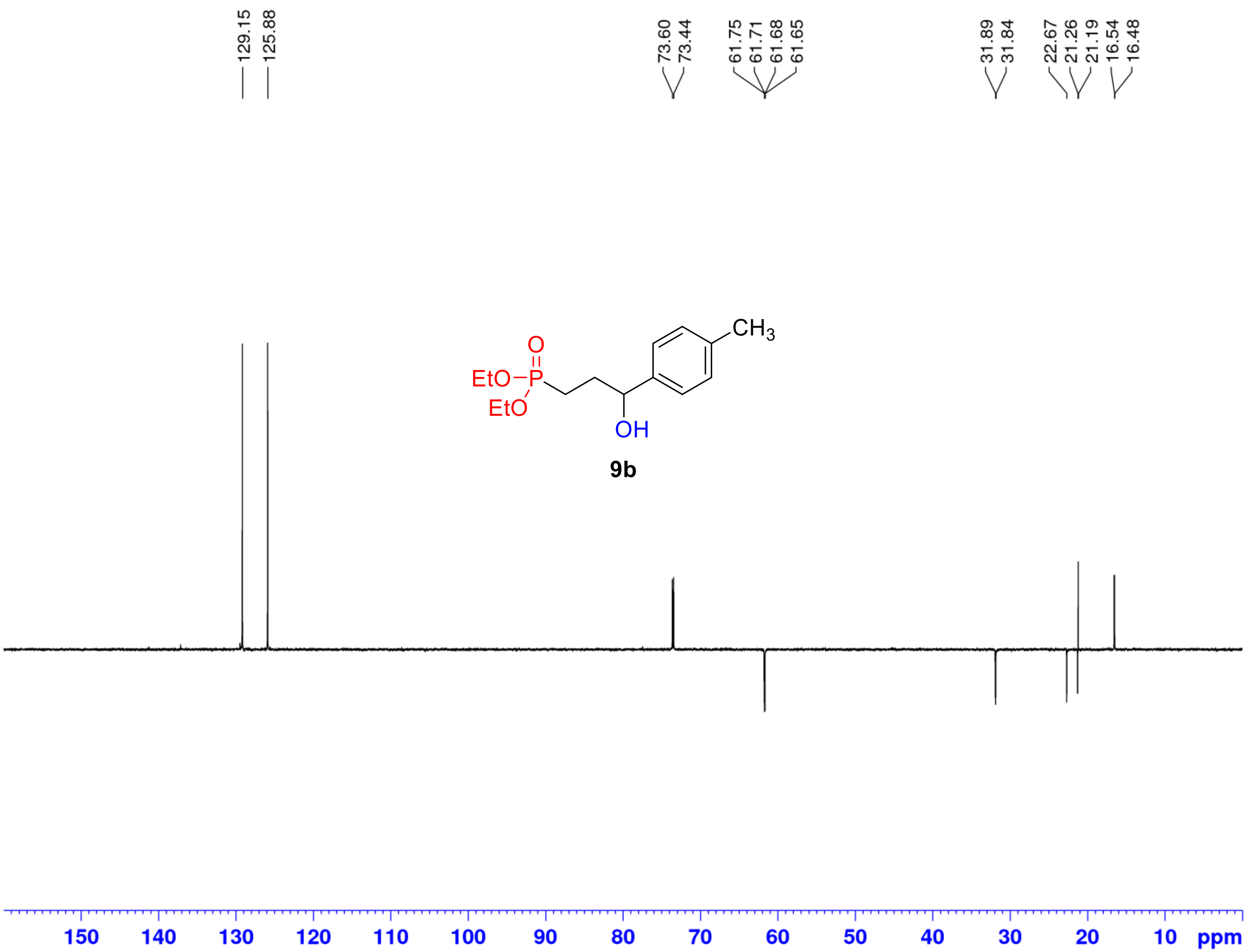
9b: ^{13}C NMR


EtO
EtO
O
P
OH
CH3

9b

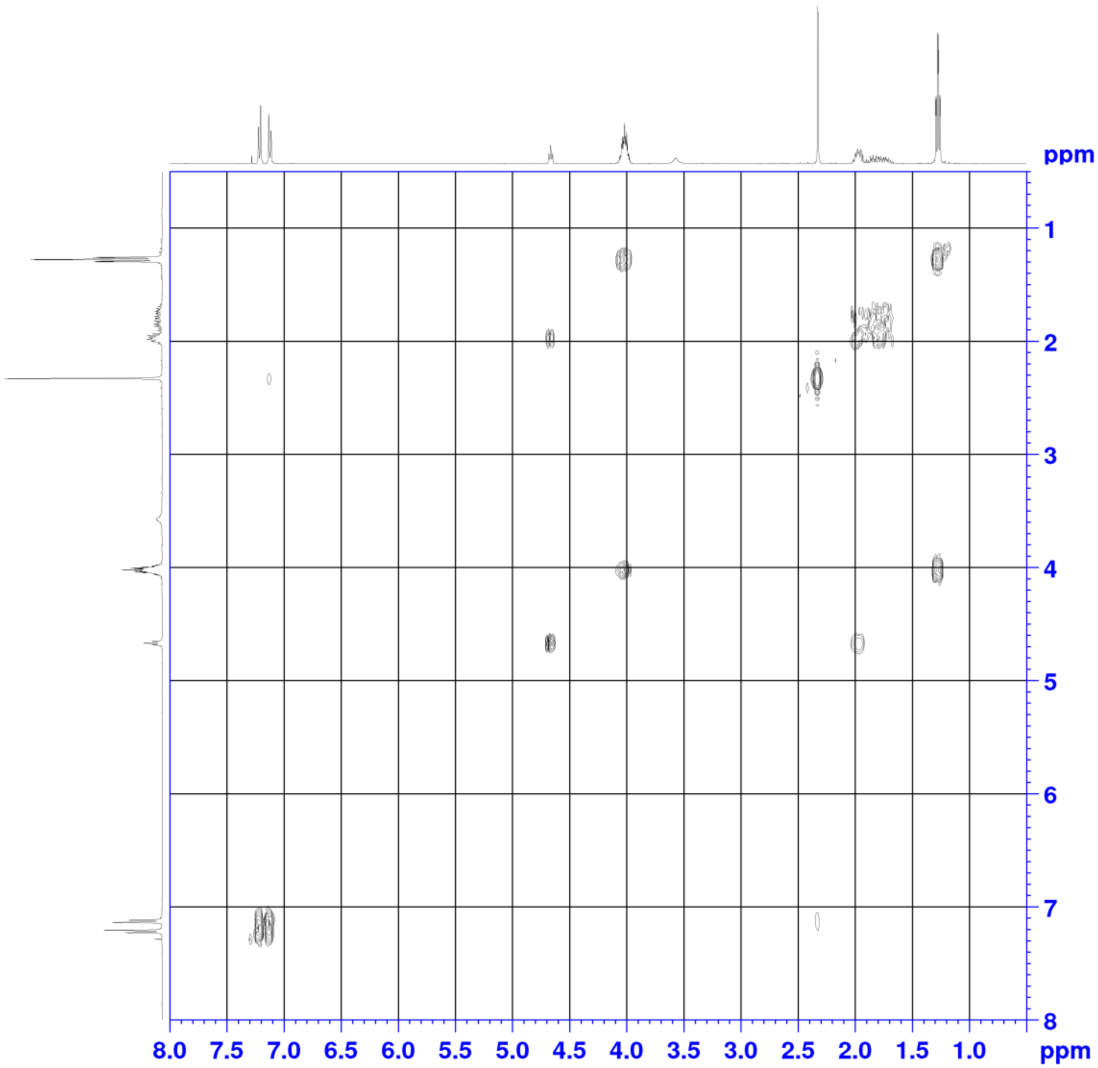

141.27
137.12
129.14
125.88
77.55
77.23
76.91
73.60
73.44
61.74
61.71
61.68
61.65
31.88
31.84
22.66
21.25
21.19
16.54
16.48
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

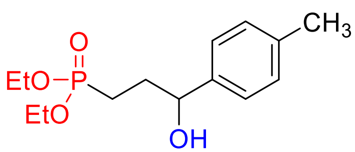
9b: ^{13}C DEPT135



9b

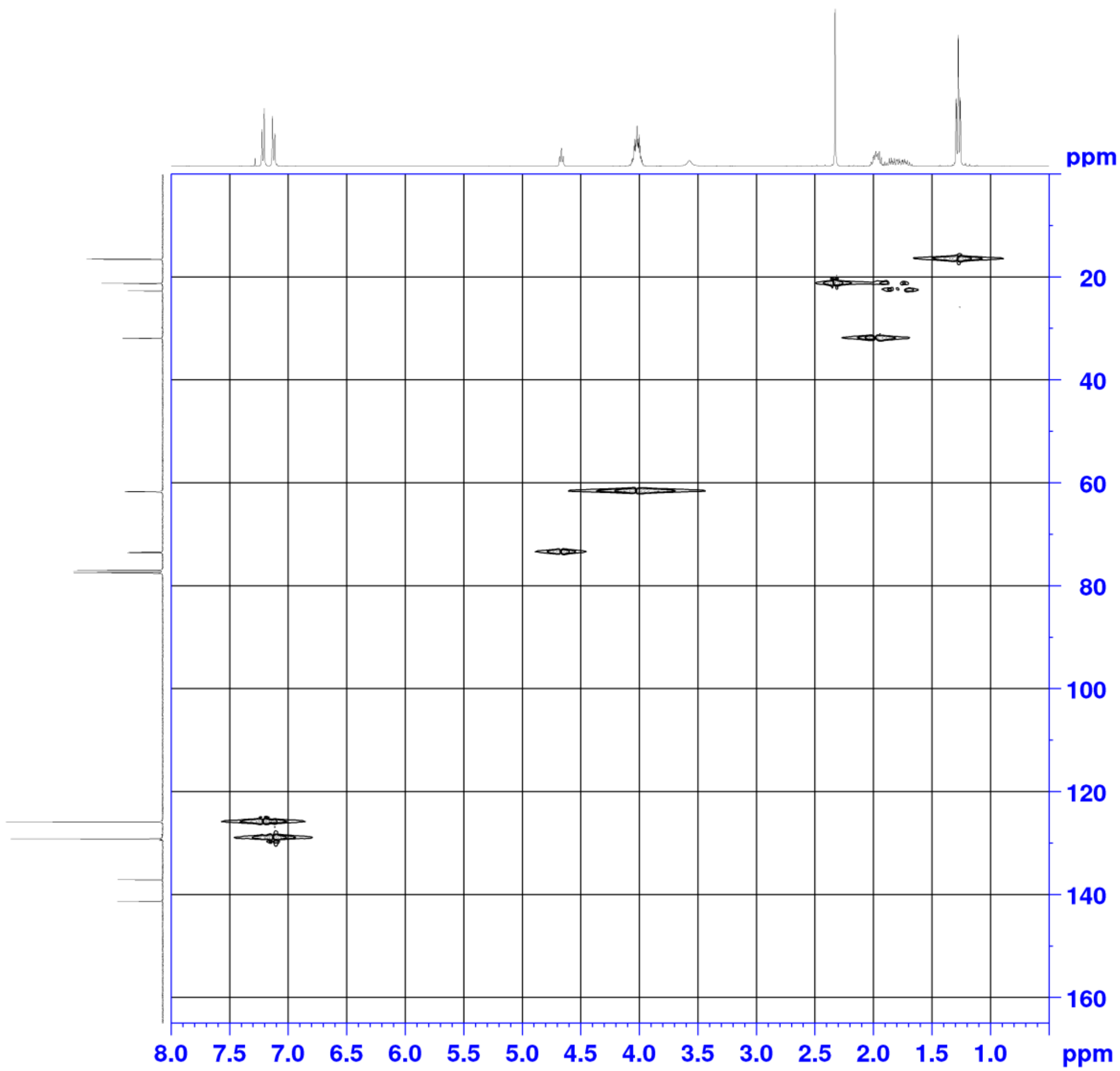
9b: COSY

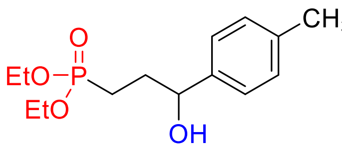

ppm
1
2
3
4
5
6
7
8
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0
ppm


O
EtO
P
EtO
OH
CH_3

9b

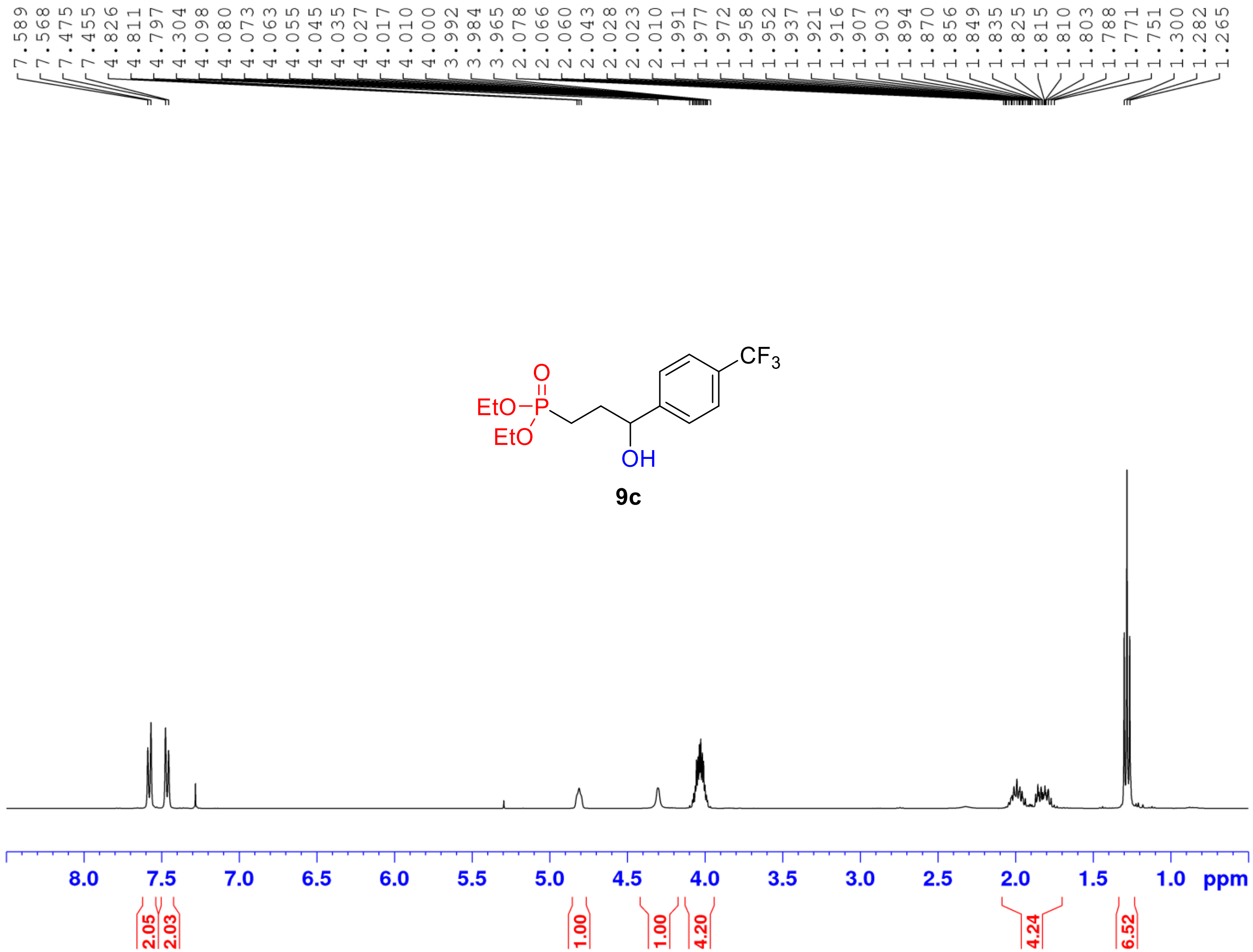
9b: ^{13}C HSQC



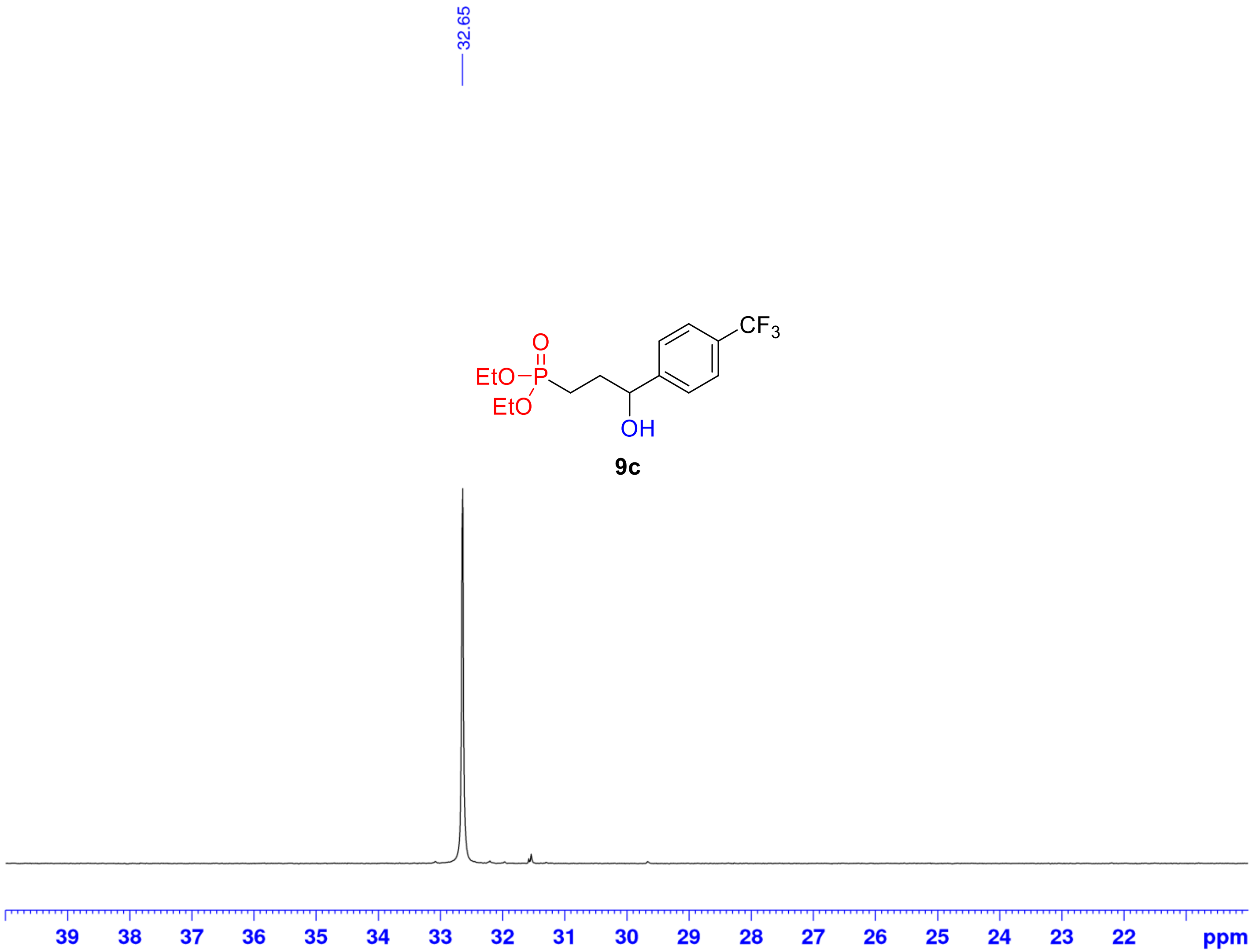


9b

9c: ^{1}H NMR

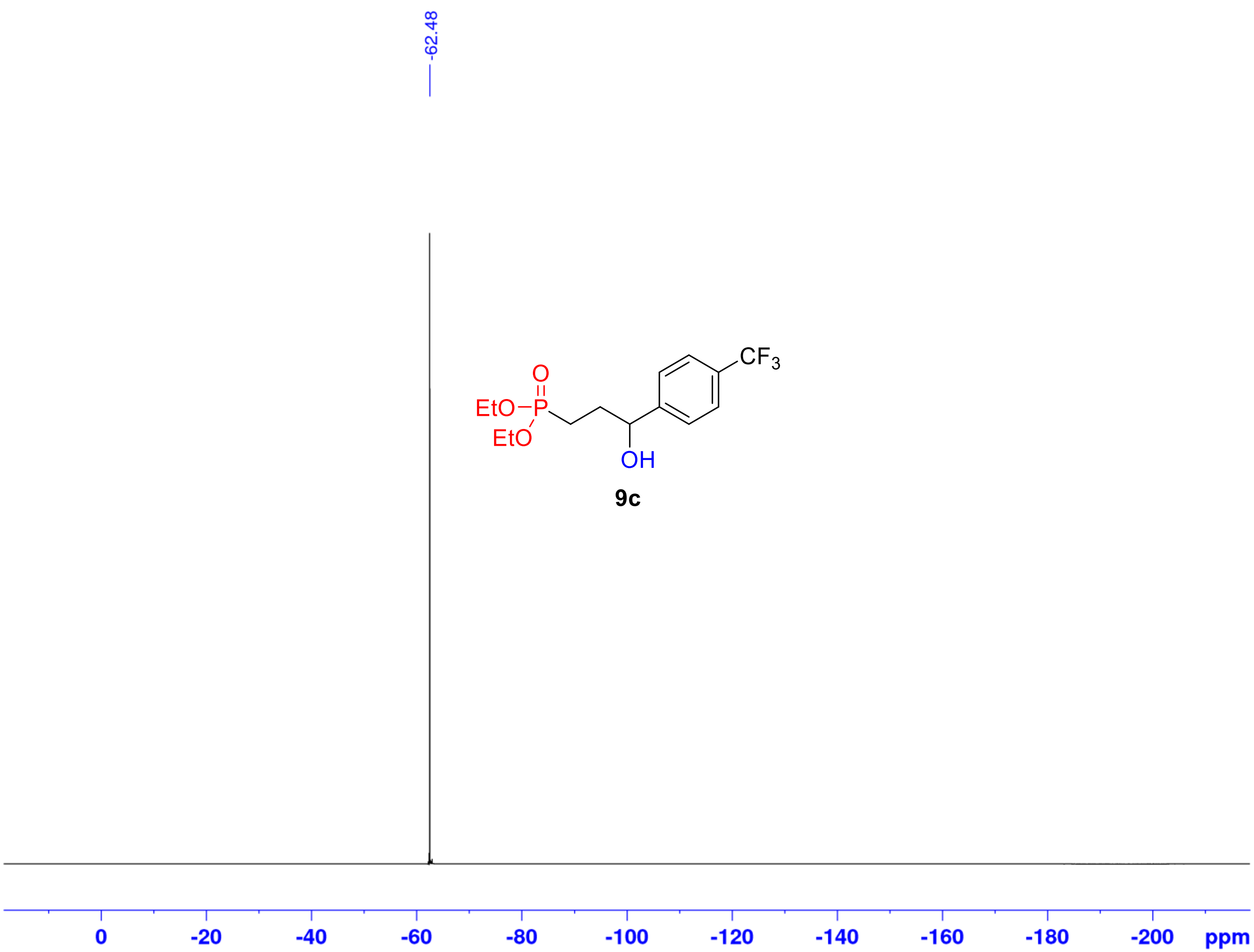

7.589
7.568
7.475
7.455
4.826
4.811
4.797
4.304
4.098
4.080
4.073
4.063
4.055
4.045
4.035
4.027
4.017
4.010
4.000
3.992
3.984
3.965
2.078
2.066
2.060
2.043
2.028
2.023
2.010
1.991
1.977
1.972
1.958
1.952
1.937
1.921
1.916
1.907
1.903
1.894
1.870
1.856
1.849
1.835
1.825
1.815
1.810
1.803
1.788
1.771
1.751
1.300
1.282
1.265
CF3
O
EtO
P
EtO
OH
9c
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
ppm
2.05
2.03
1.00
1.00
4.20
4.24
6.52

9c: ^{31}P NMR



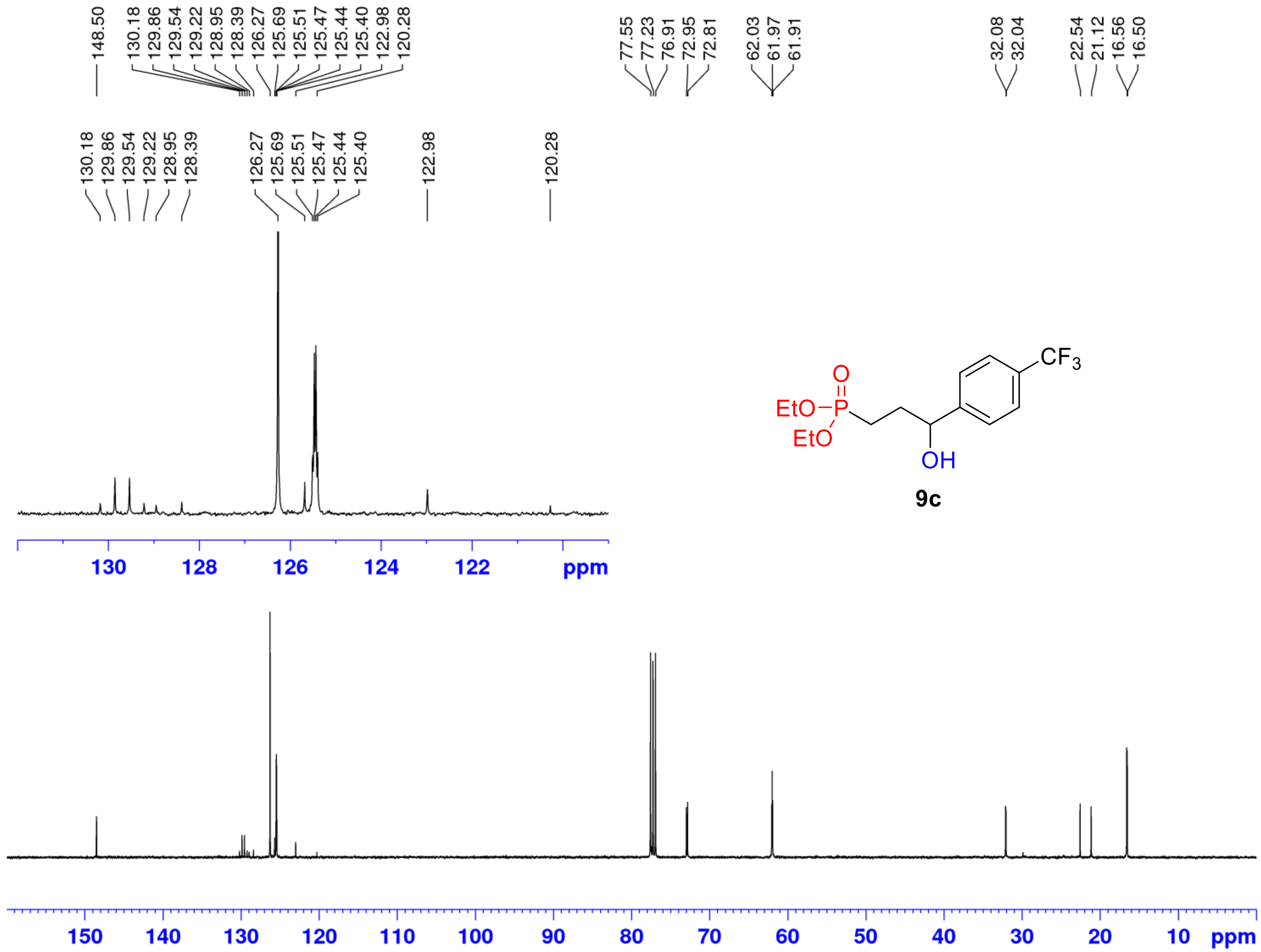
32.65
O
EtO–P
EtO
OH
CF3
9c
39 38 37 36 35 34 33 32 31 30 29 28 27 26 25 24 23 22
ppm

9c: ^{19}F NMR



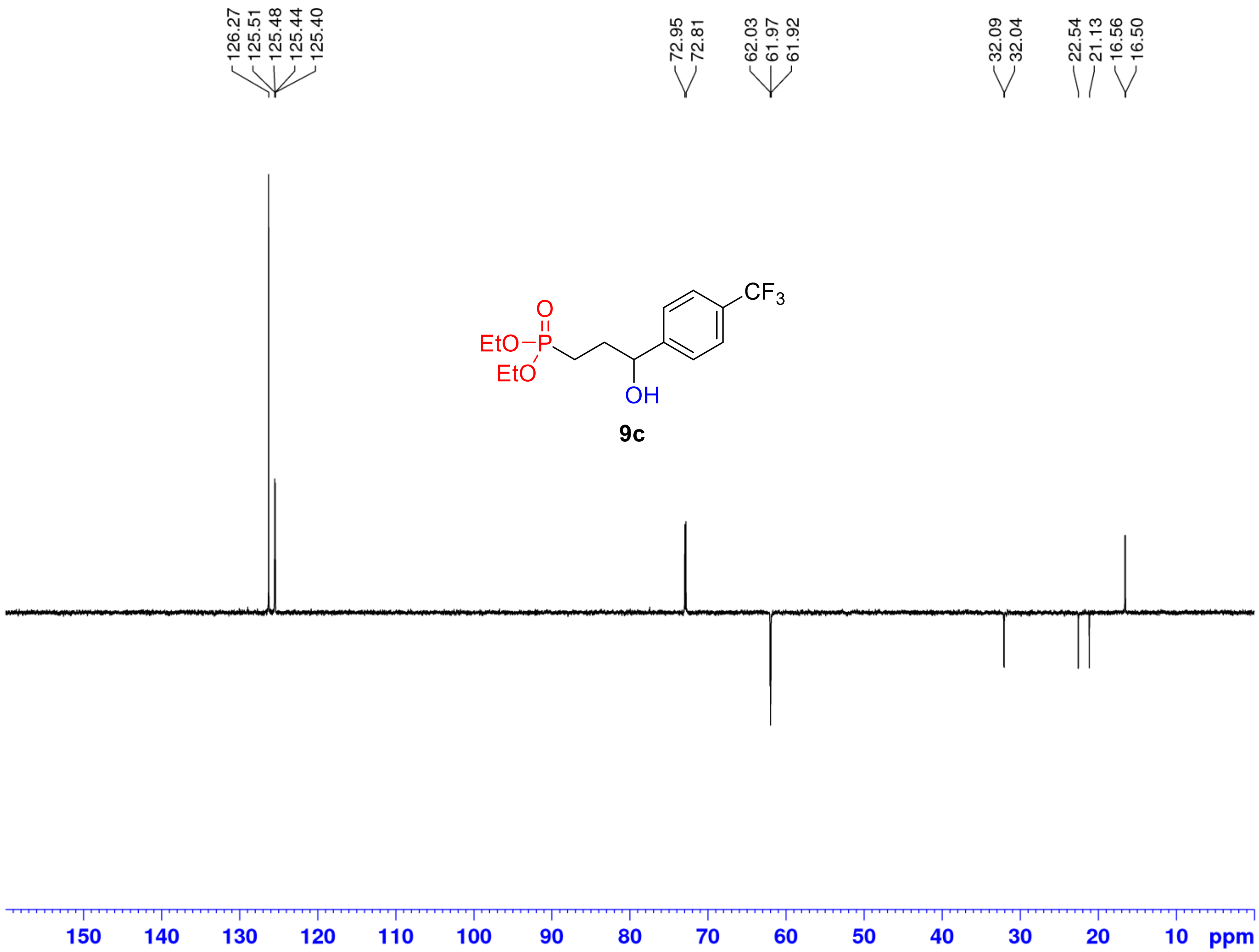
9c

9c: ^{13}C NMR

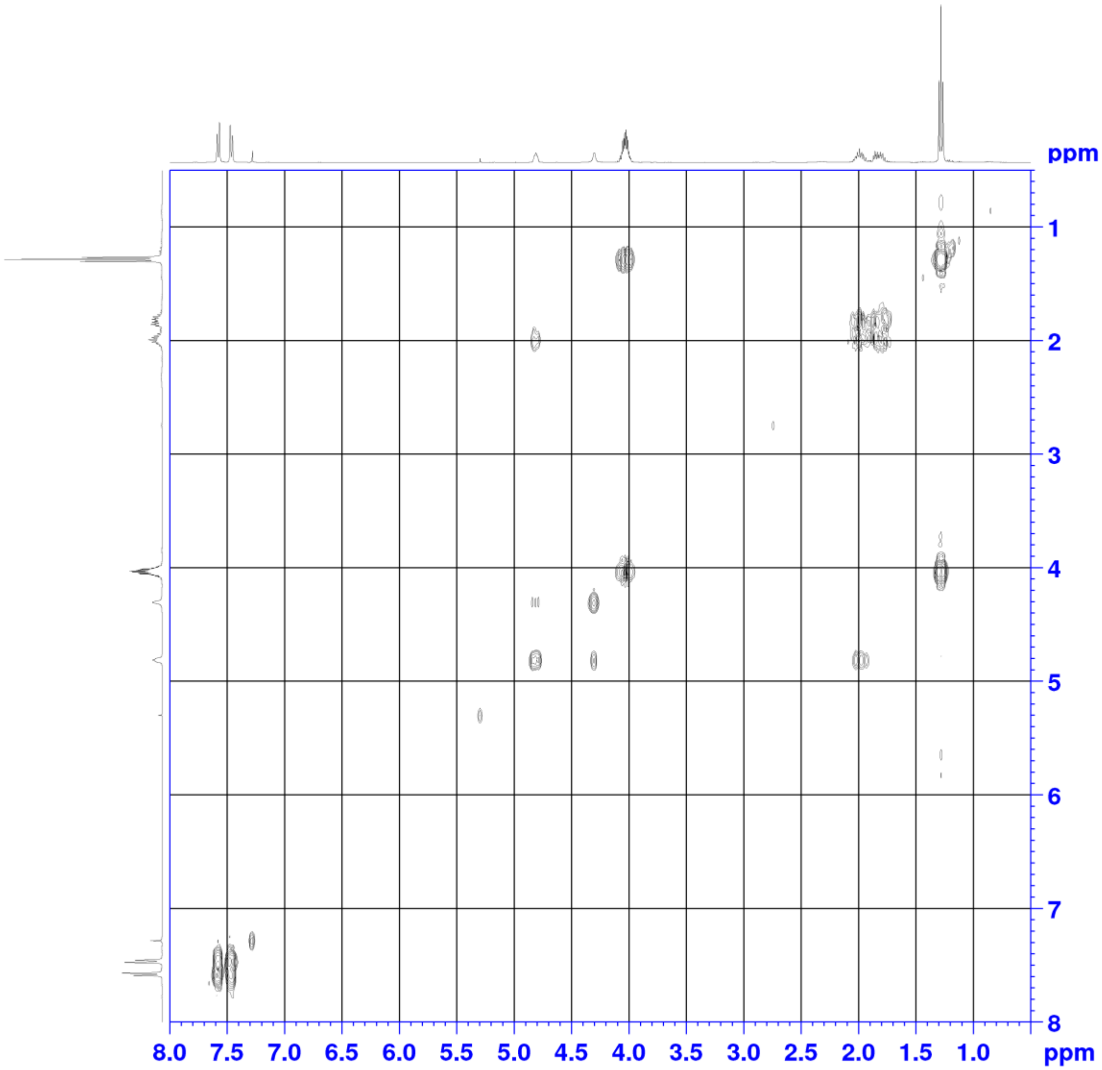

148.50
130.18
129.86
129.54
129.22
128.95
128.39
126.27
125.69
125.51
125.47
125.44
125.40
122.98
120.28
77.55
77.23
76.91
72.95
72.81
62.03
61.97
61.91
32.08
32.04
22.54
21.12
16.56
16.50
130.18
129.86
129.54
129.22
128.95
128.39
126.27
125.69
125.51
125.47
125.44
125.40
122.98
120.28
130
128
126
124
122
ppm
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm
O
EtO
P
EtO
OH
CF3

9c

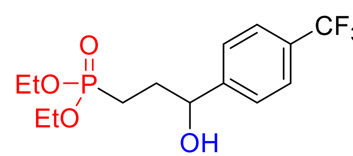
9c: ^{13}C DEPT135



9c: COSY



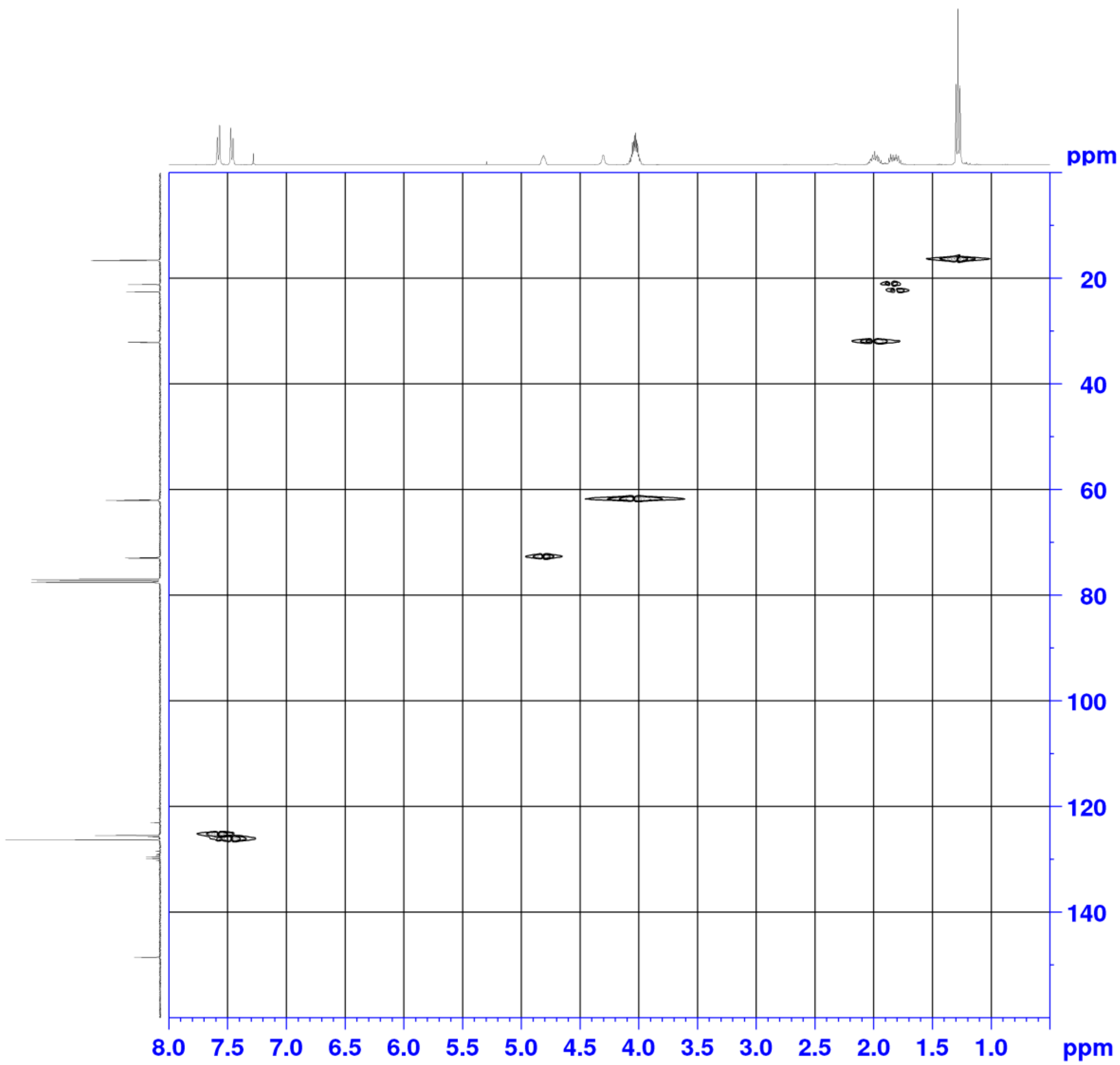
ppm
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
1
2
3
4
5
6
7
8

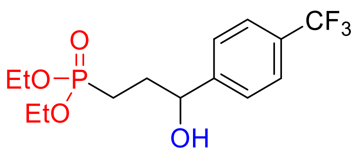


O
EtO
P
EtO
OH
CF_3

9c

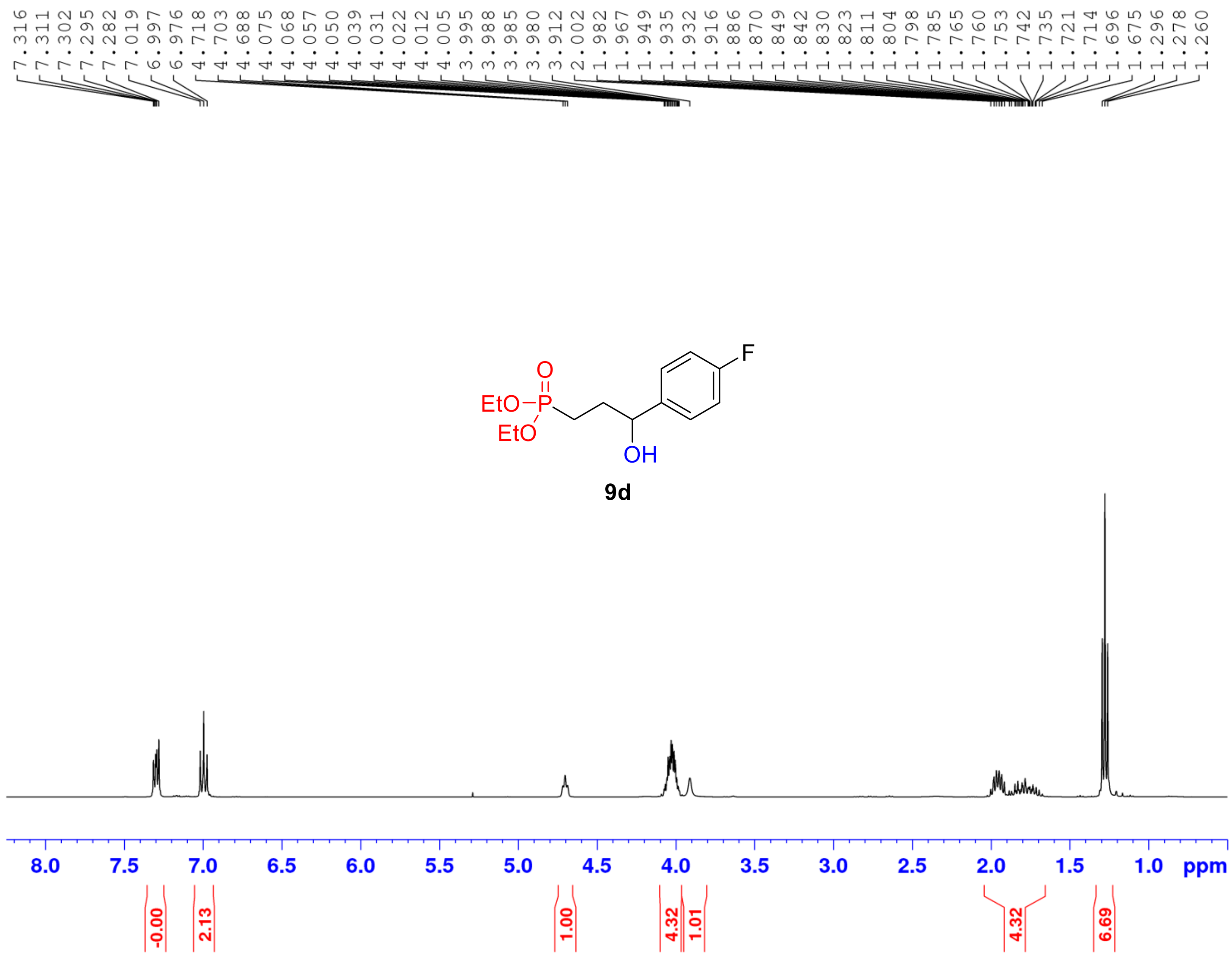
9c: ^{13}C HSQC



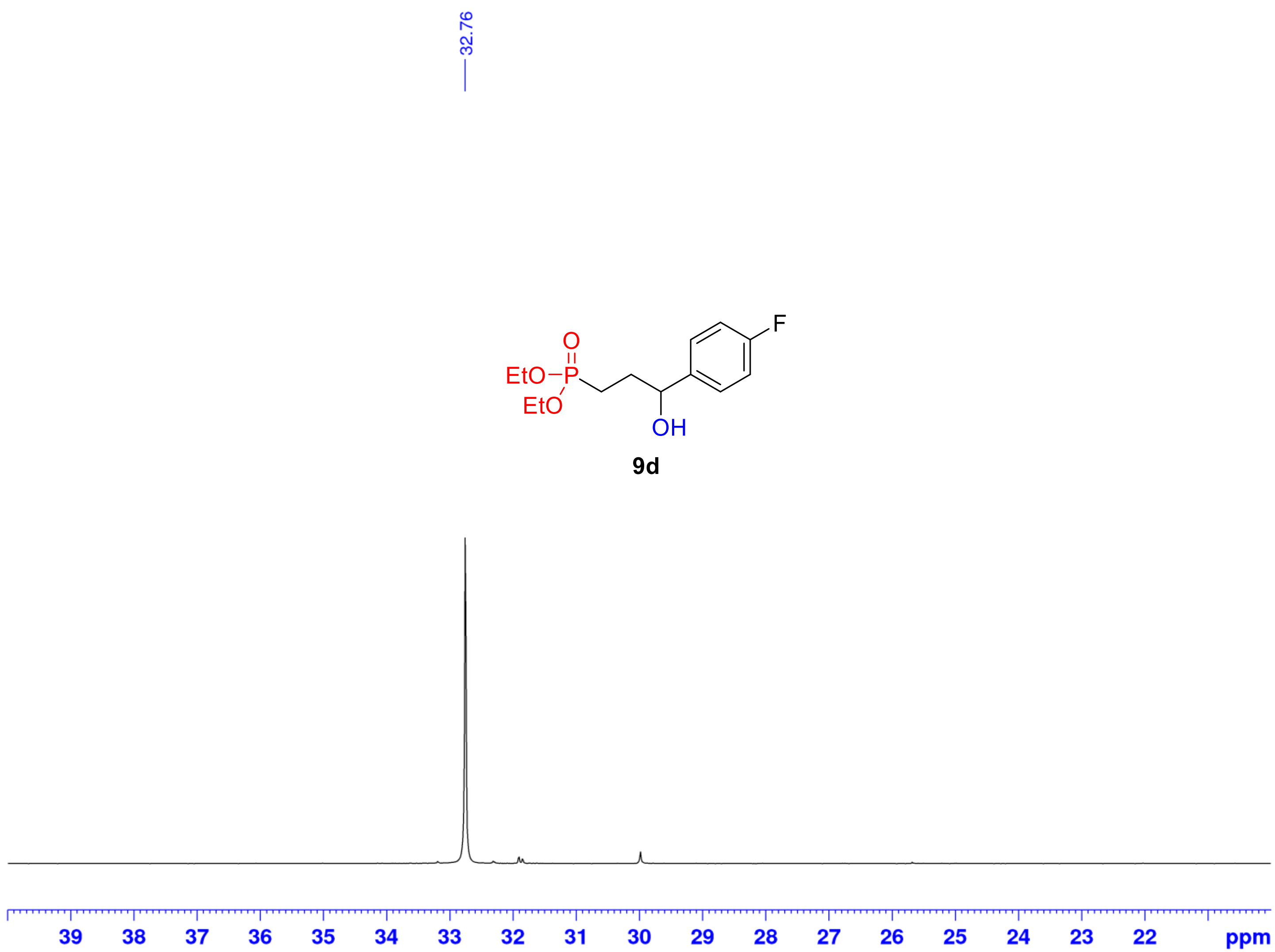


9c

9d: ^{1}H NMR

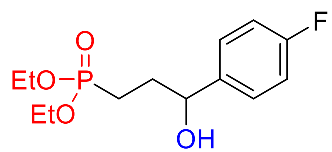

7.316 7.311 7.302 7.295 7.282 7.019 6.997 6.976 4.718 4.703 4.688 4.075 4.068 4.057 4.050 4.039 4.031 4.022 4.012 4.005 3.995 3.988 3.985 3.980 3.912 2.002 1.982 1.967 1.949 1.935 1.932 1.916 1.886 1.870 1.849 1.842 1.830 1.823 1.811 1.804 1.798 1.785 1.765 1.760 1.753 1.742 1.735 1.721 1.714 1.696 1.675 1.296 1.278 1.260
O
EtO
P
EtO
F
OH
9d
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 ppm
-0.00 2.13 1.00 4.32 1.01 4.32 6.69

9d: ^{31}P NMR

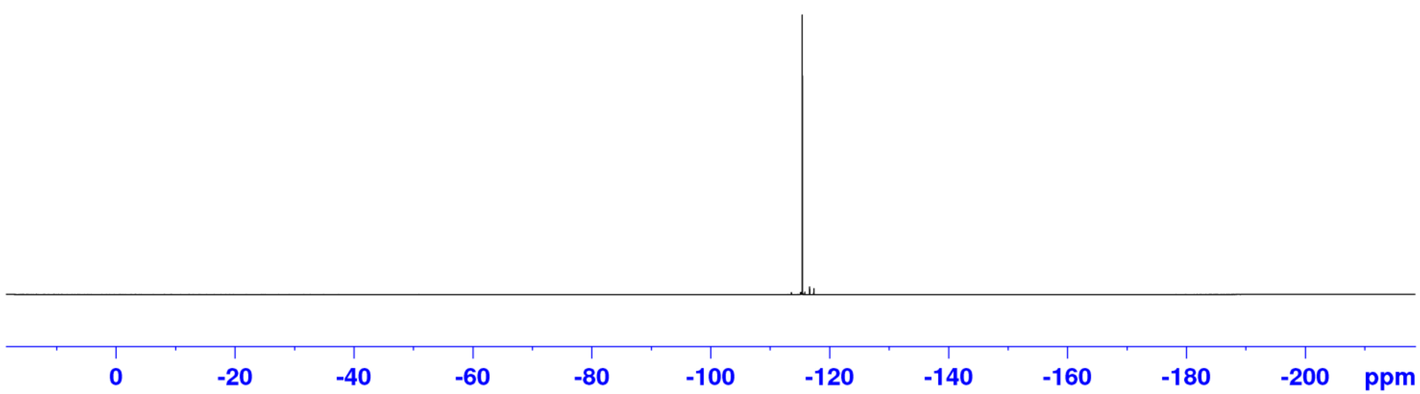


32.76
EtO
EtO
P
O
OH
F
9d
39
38
37
36
35
34
33
32
31
30
29
28
27
26
25
24
23
22
ppm

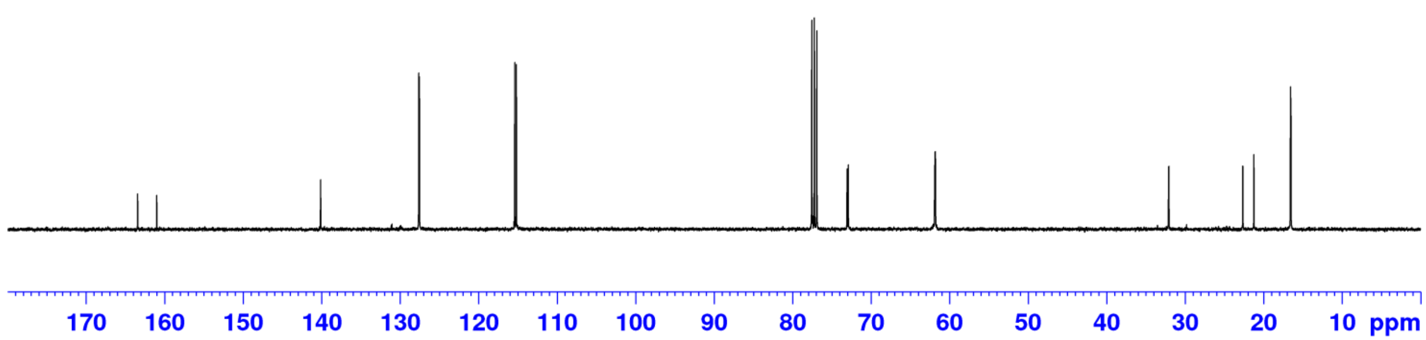
9d: ^{19}F NMR

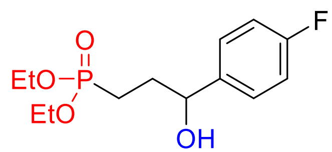


9d



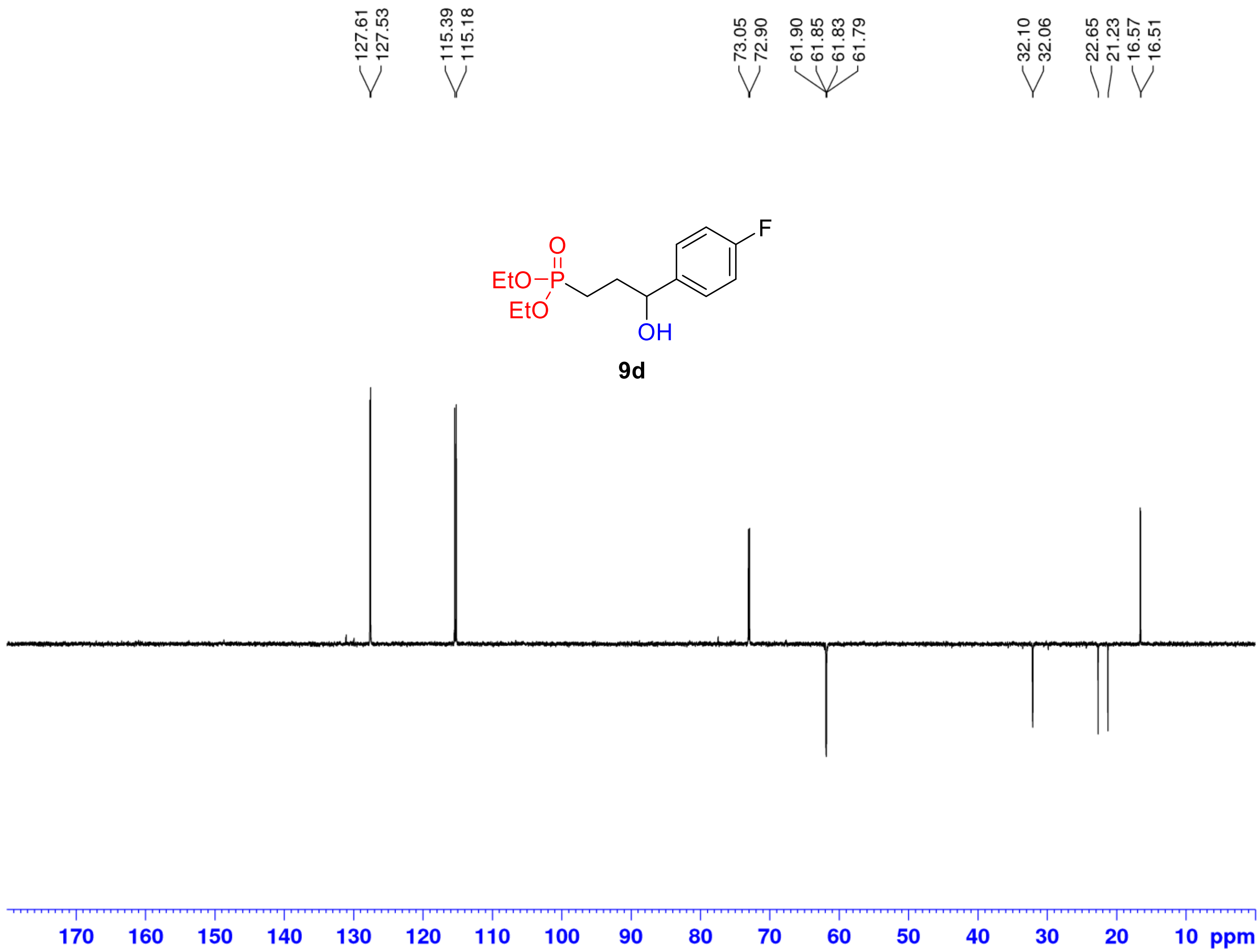
9d: ^{13}C NMR


163.43
161.00
140.13
140.10
127.61
127.53
115.39
115.18
77.55
77.23
76.91
73.05
72.90
61.89
61.85
61.83
61.79
32.10
32.05
22.64
21.23
16.57
16.51
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

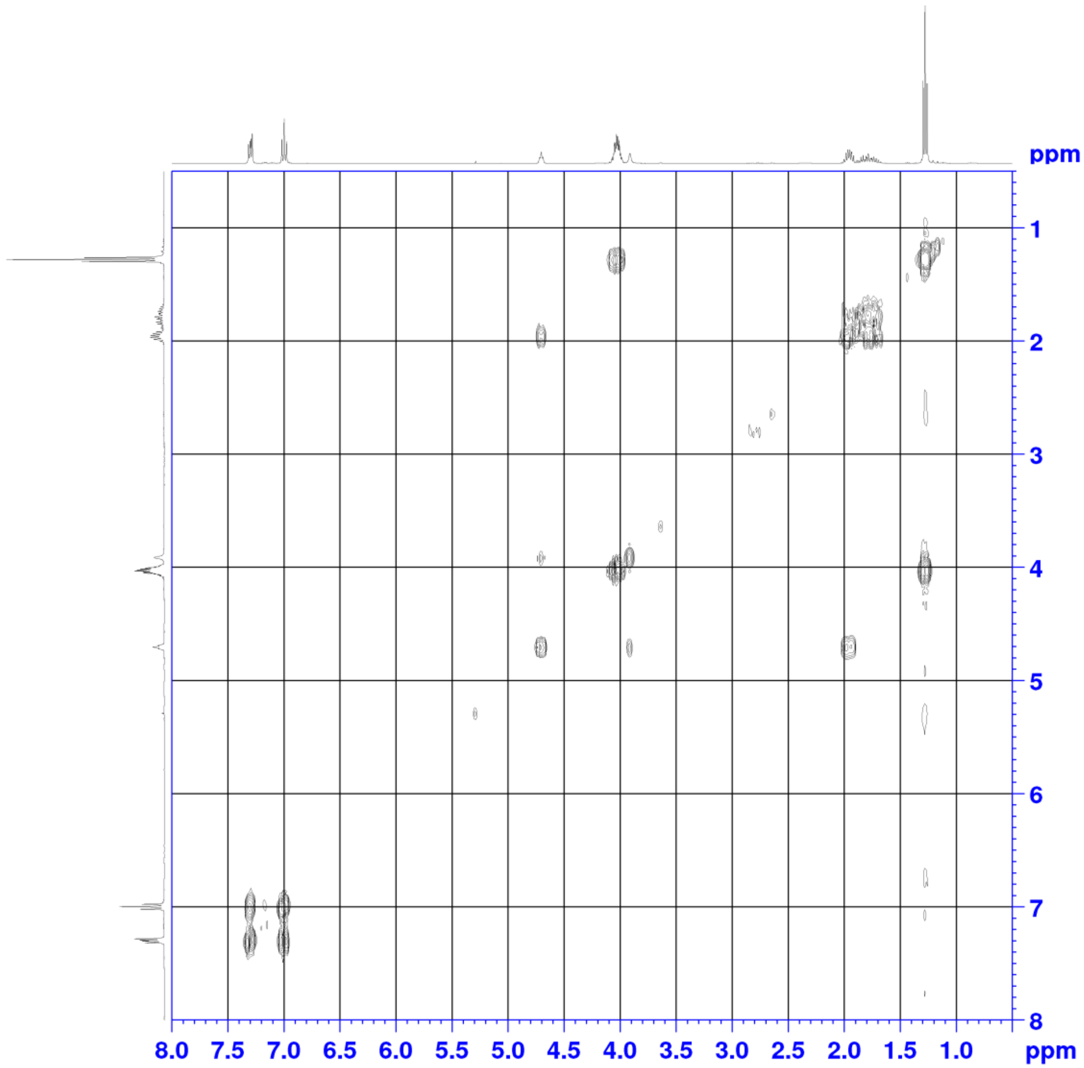

O
EtO
P
EtO
OH
F

9d

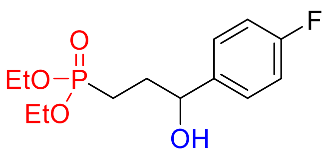
9d: ^{13}C DEPT135



9d: COSY



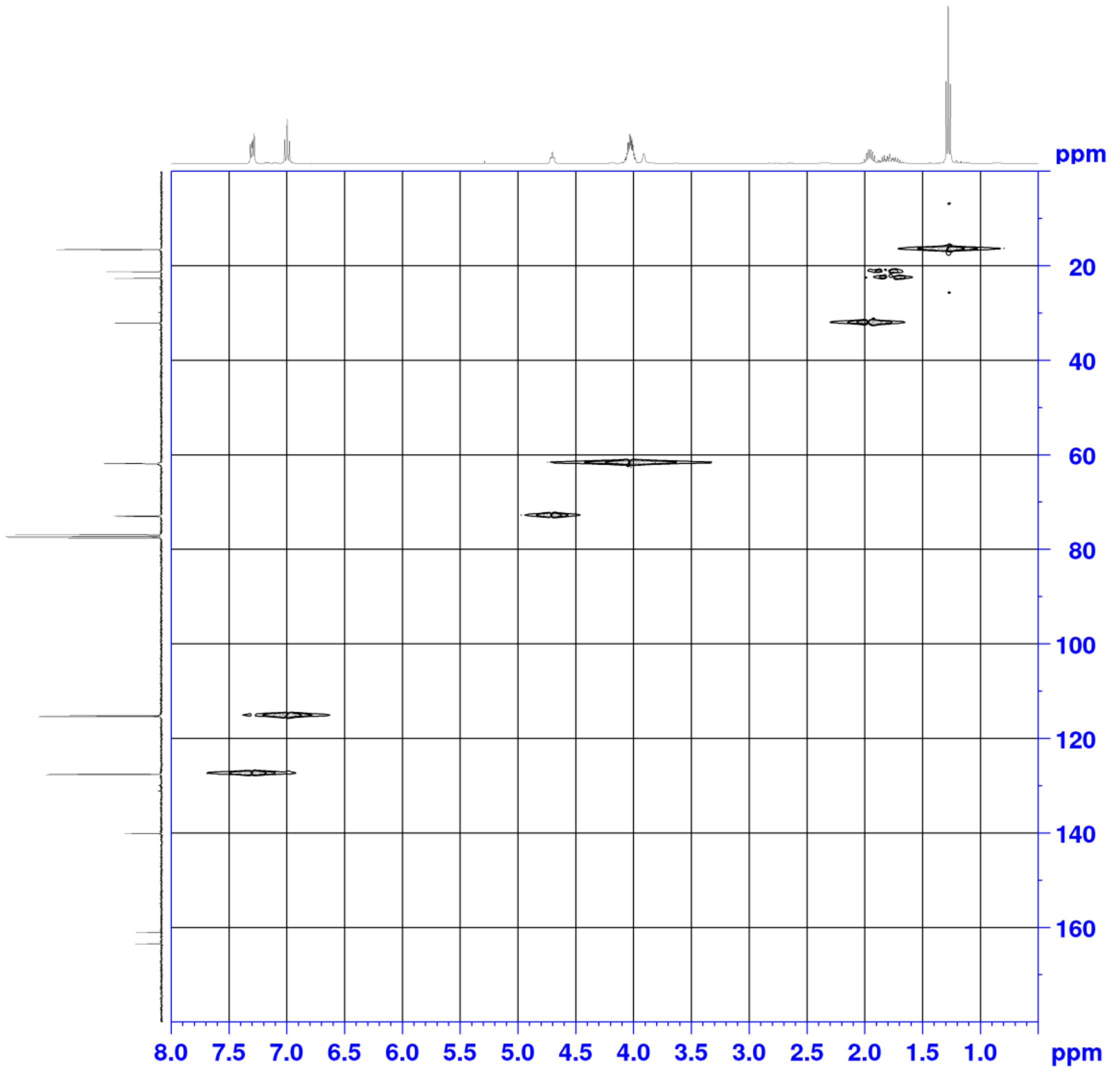
ppm
1
2
3
4
5
6
7
8
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

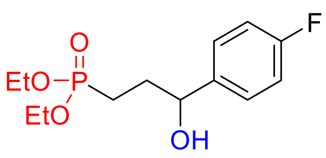


O
EtO
P
EtO
OH
F

9d

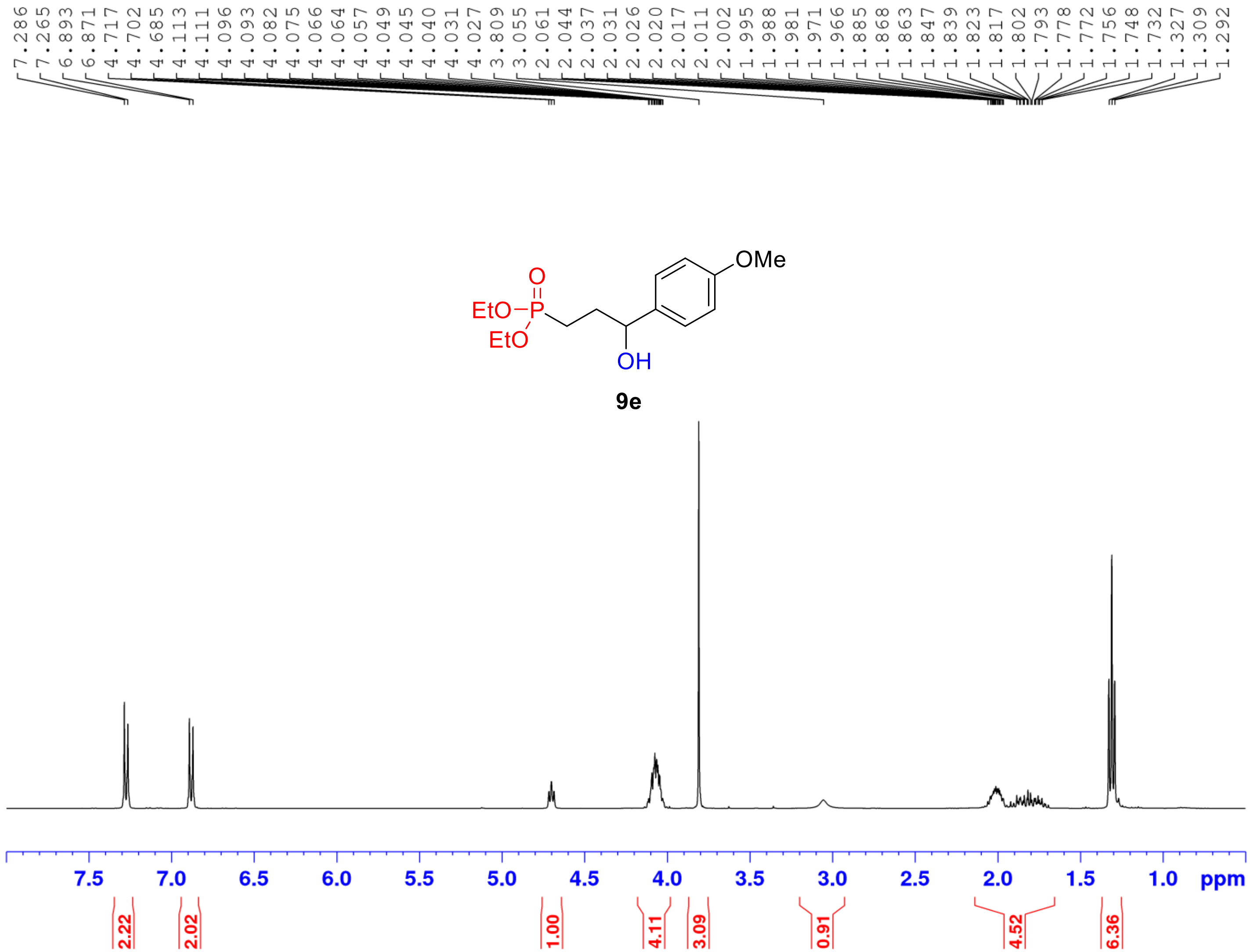
9d: ^{13}C HSQC



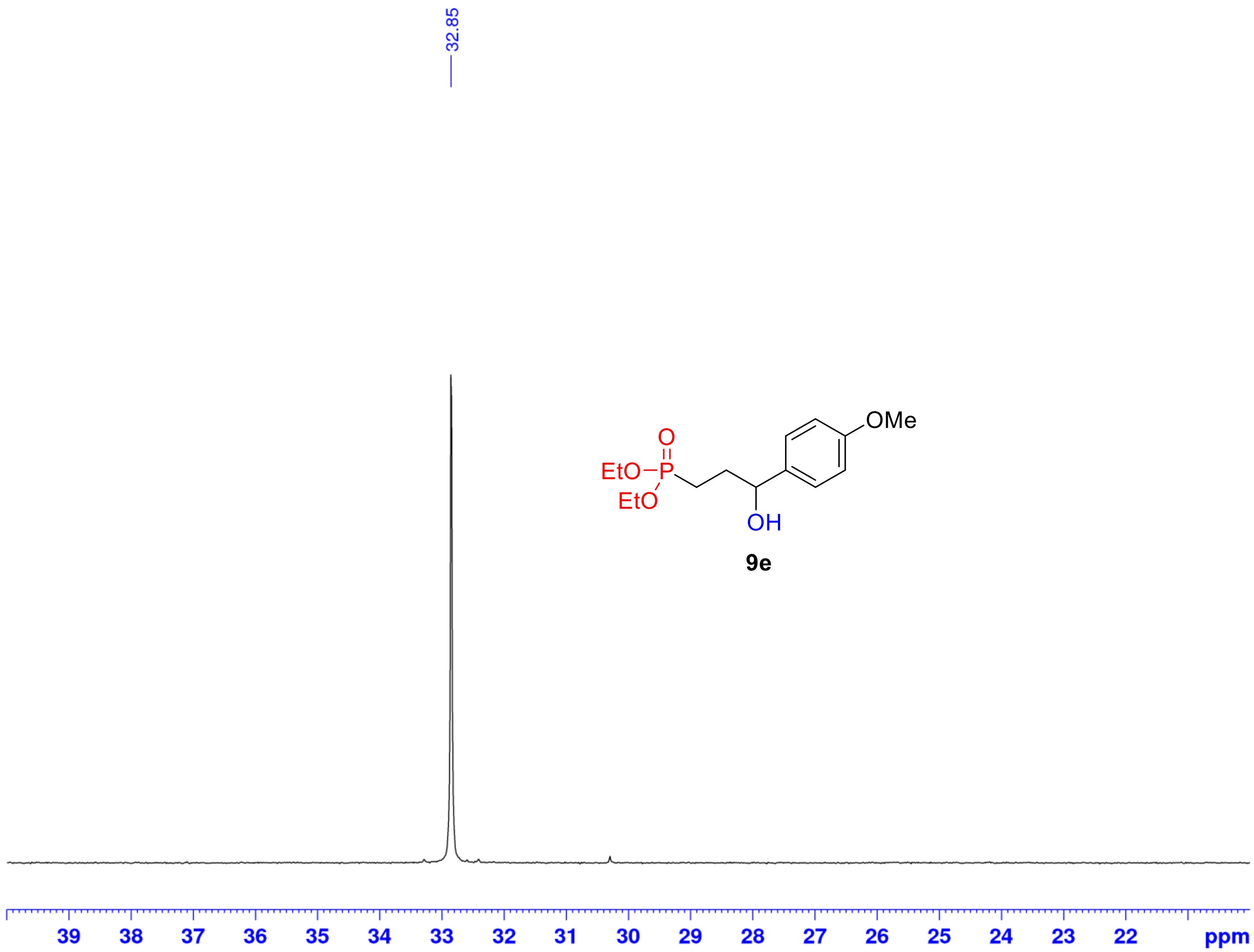


9d

9e: ^{1}H NMR

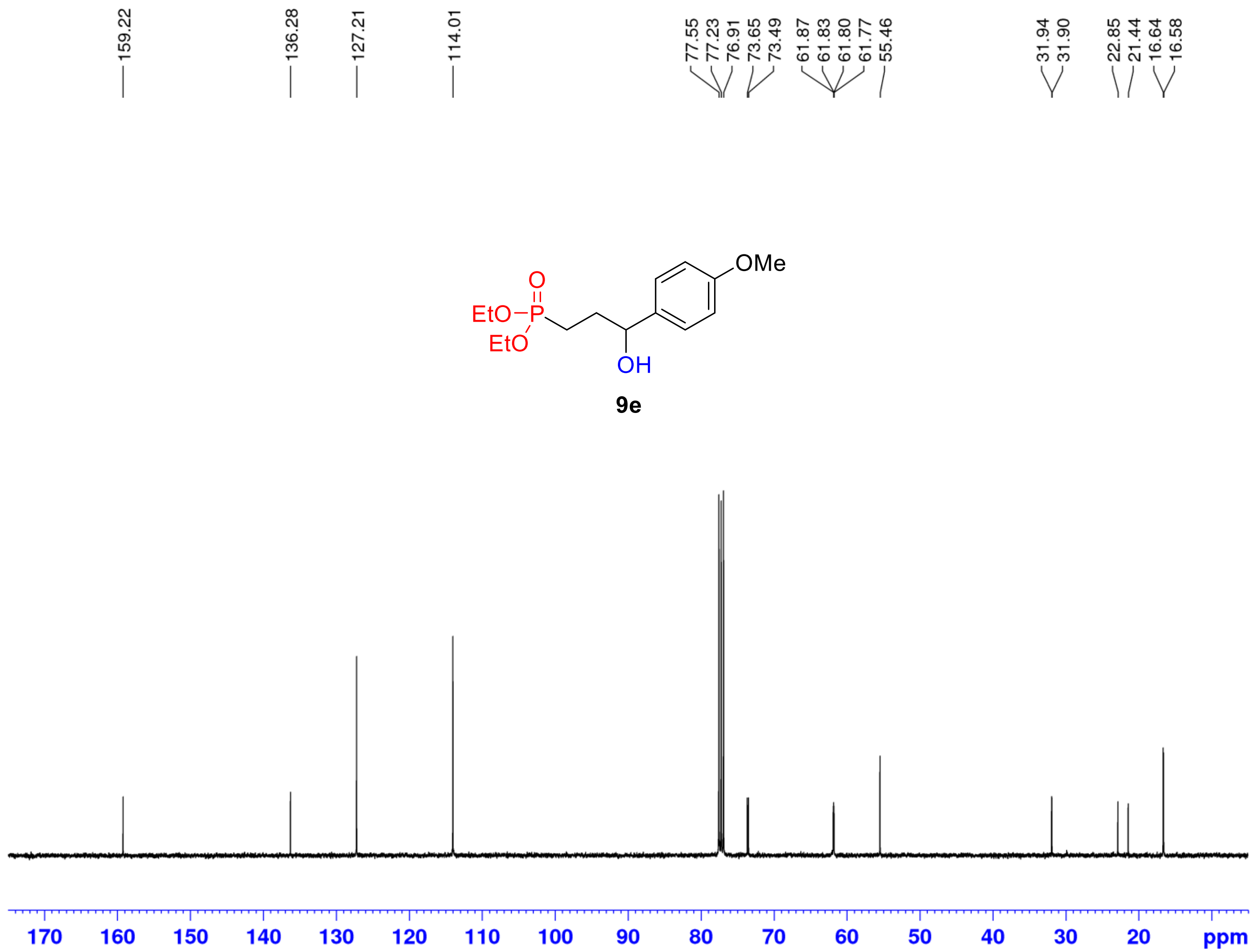

7.286
7.265
6.893
6.871
4.717
4.702
4.685
4.113
4.111
4.096
4.093
4.082
4.075
4.066
4.064
4.057
4.049
4.045
4.040
4.031
4.027
3.809
3.055
2.061
2.044
2.037
2.031
2.026
2.020
2.017
2.011
2.002
1.995
1.988
1.981
1.971
1.966
1.885
1.868
1.863
1.847
1.839
1.823
1.817
1.802
1.793
1.778
1.772
1.756
1.748
1.732
1.327
1.309
1.292
OMe
O
EtO
P
EtO
OH
9e
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
2.22
2.02
1.00
4.11
3.09
0.91
4.52
6.36

9e: ^{31}P NMR



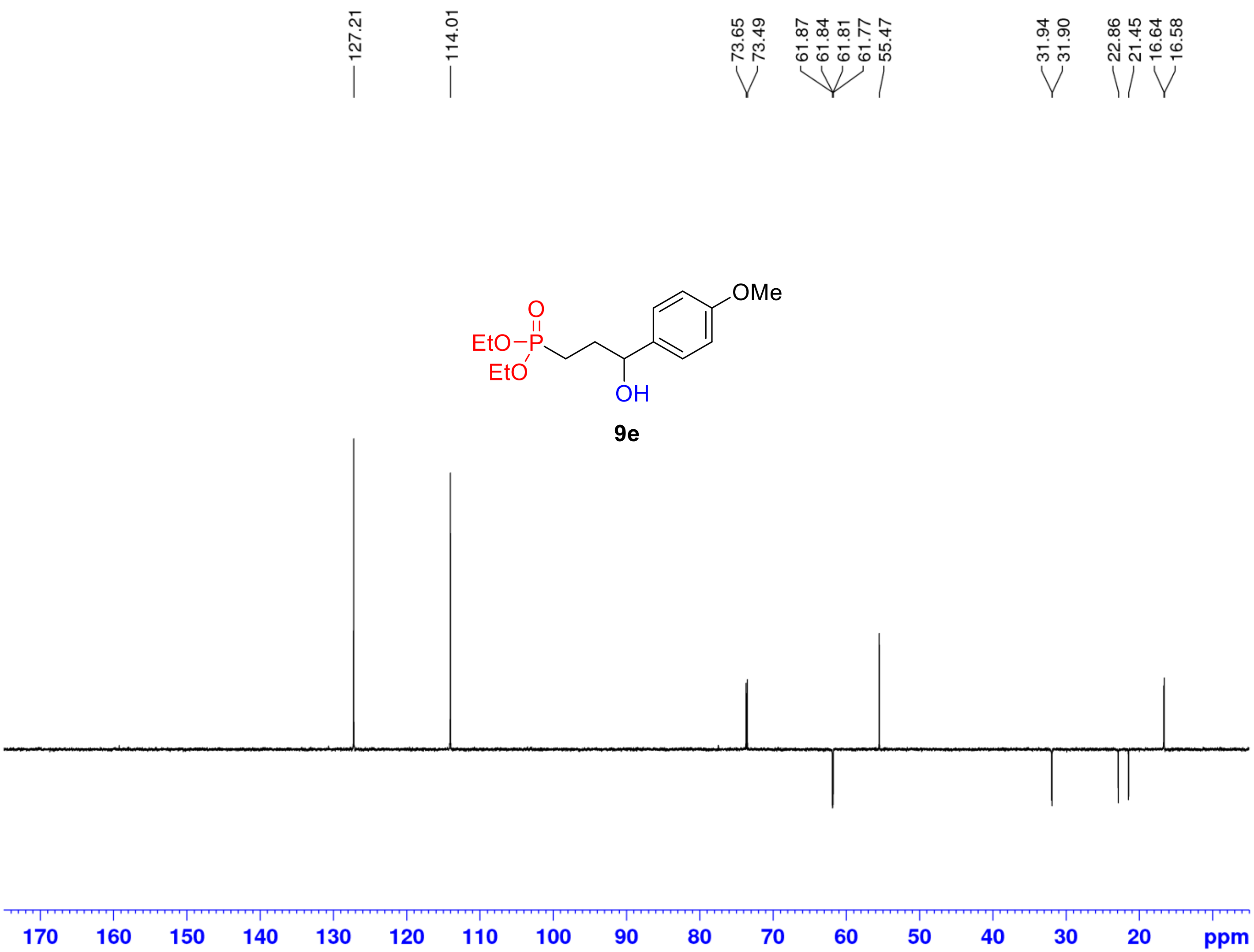
32.85
OMe
O
EtO
P
EtO
OH
9e
39
38
37
36
35
34
33
32
31
30
29
28
27
26
25
24
23
22
ppm

9e: ^{13}C NMR

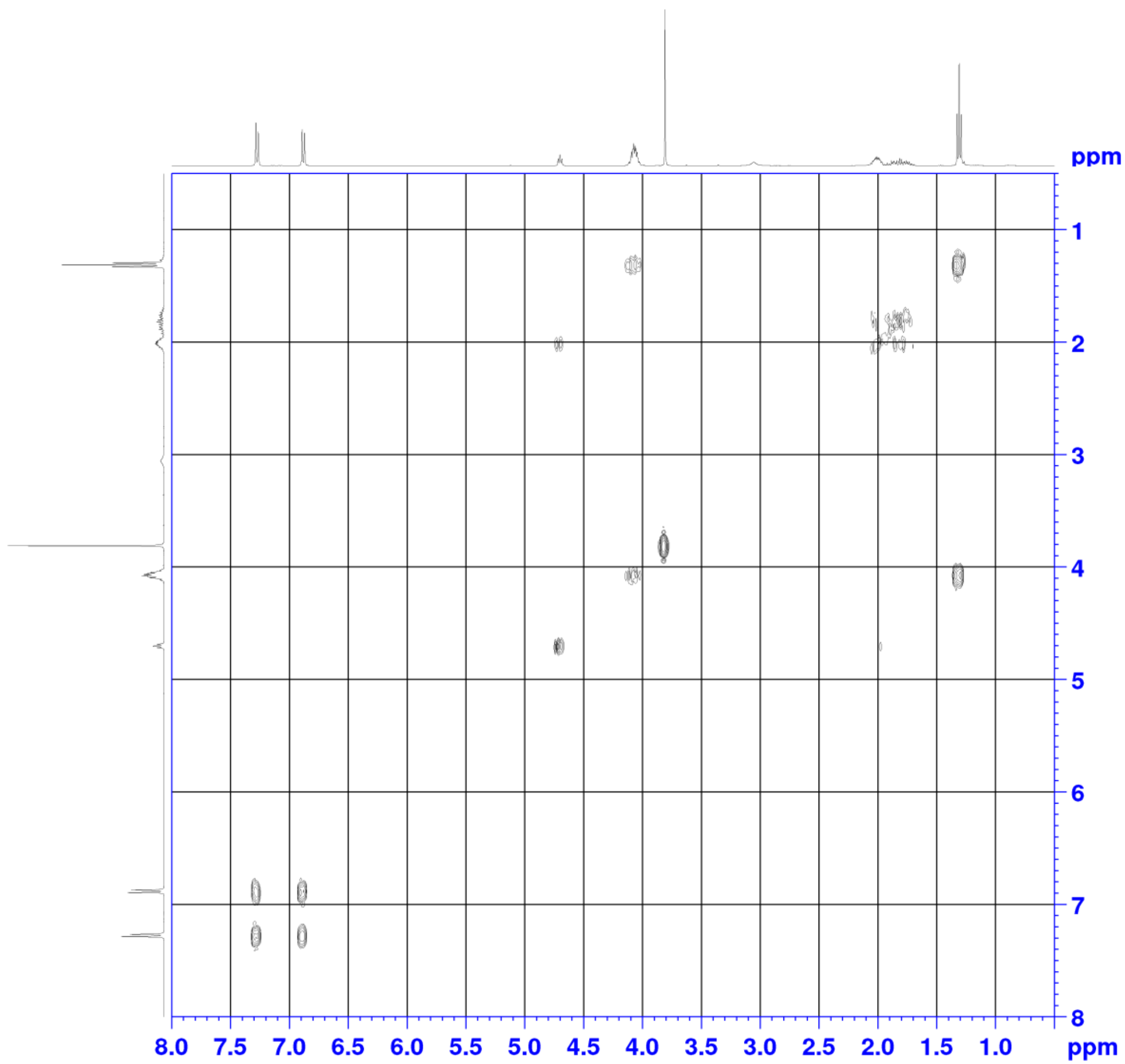


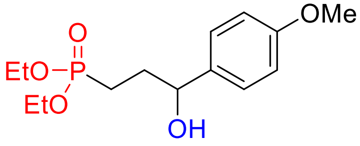
159.22
136.28
127.21
114.01
77.55
77.23
76.91
73.65
73.49
61.87
61.83
61.80
61.77
55.46
31.94
31.90
22.85
21.44
16.64
16.58
OMe
O
EtO
P
EtO
OH
9e
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

9e: ^{13}C DEPT135



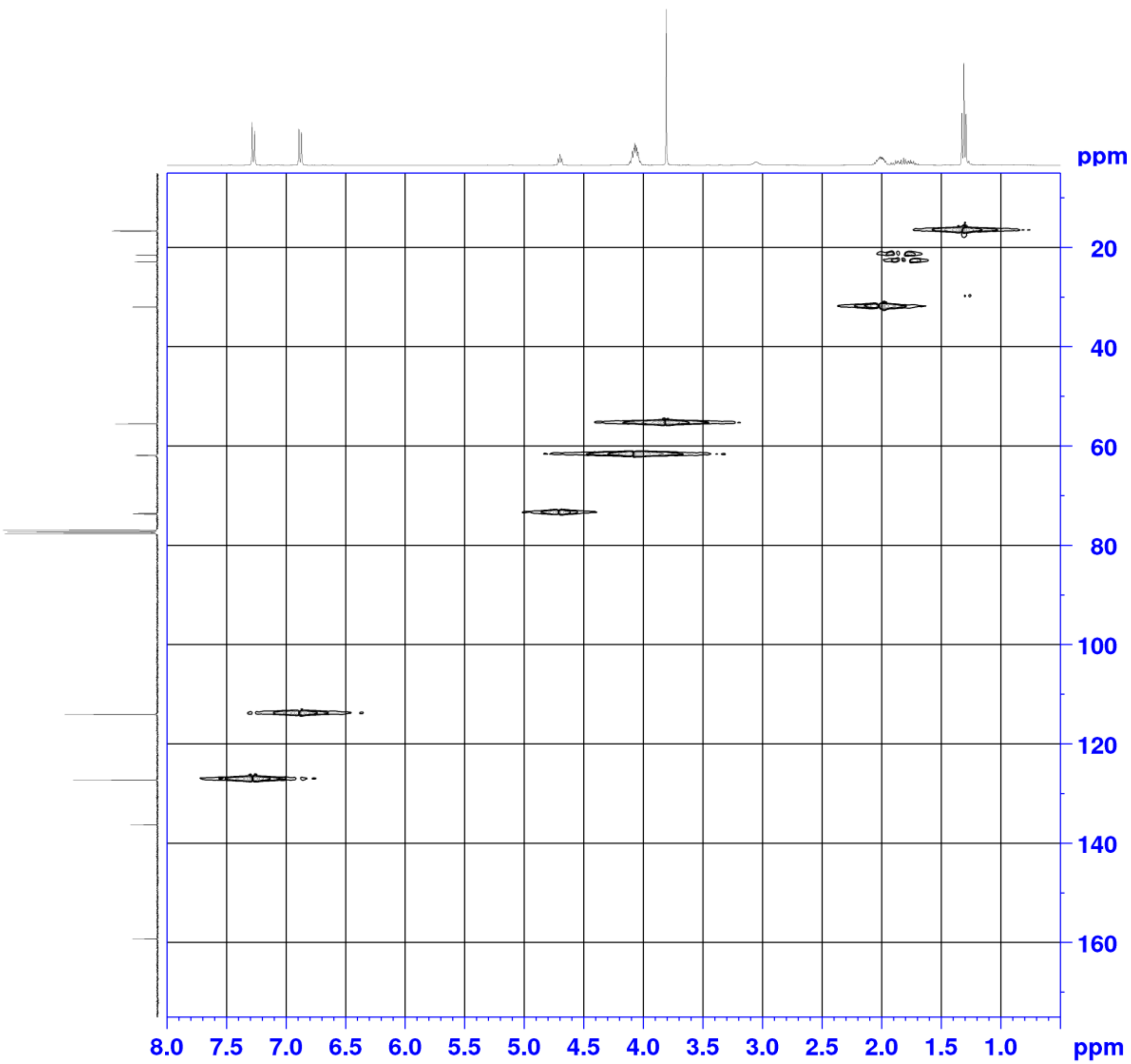
9e: COSY

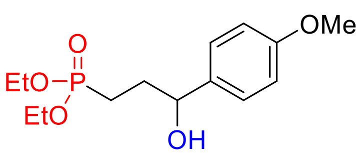

ppm
1
2
3
4
5
6
7
8
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0
ppm


O
EtO
P
EtO
OMe
OH

9e

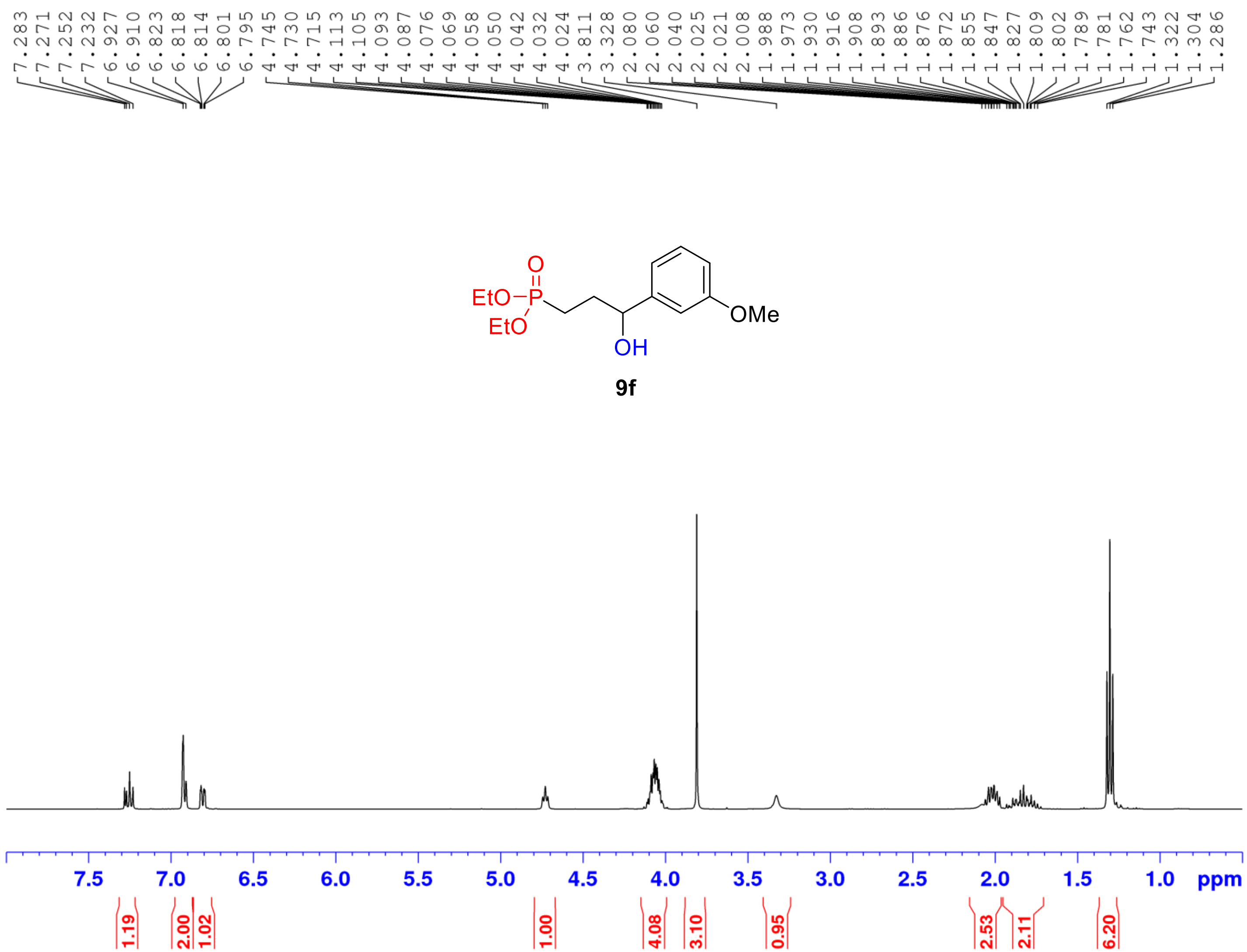
9e: ^{13}C HSQC





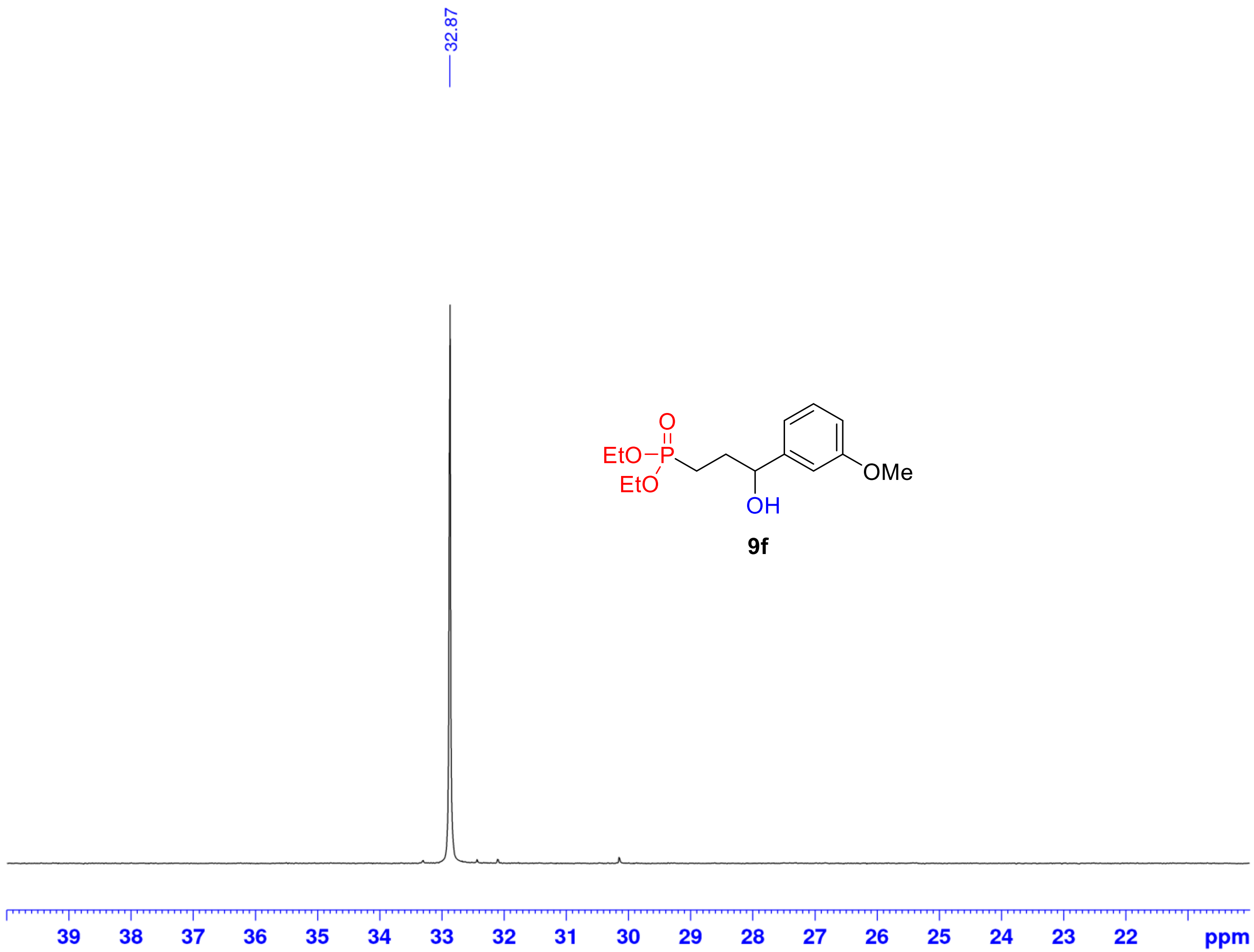
9e

9f: [1]H NMR



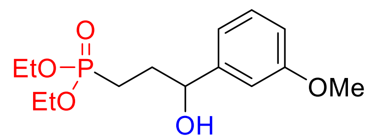
7.283
7.271
7.252
7.232
6.927
6.910
6.823
6.818
6.814
6.801
6.795
4.745
4.730
4.715
4.113
4.105
4.093
4.087
4.076
4.069
4.058
4.050
4.042
4.032
4.024
3.811
3.328
2.080
2.060
2.040
2.025
2.021
2.008
1.988
1.973
1.930
1.916
1.908
1.893
1.886
1.876
1.872
1.855
1.847
1.827
1.809
1.802
1.789
1.781
1.762
1.743
1.322
1.304
1.286
O
EtO
P
EtO
OH
OMe
9f
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
1.19
2.00
1.02
1.00
4.08
3.10
0.95
2.53
2.11
6.20

9f: ^{31}P NMR

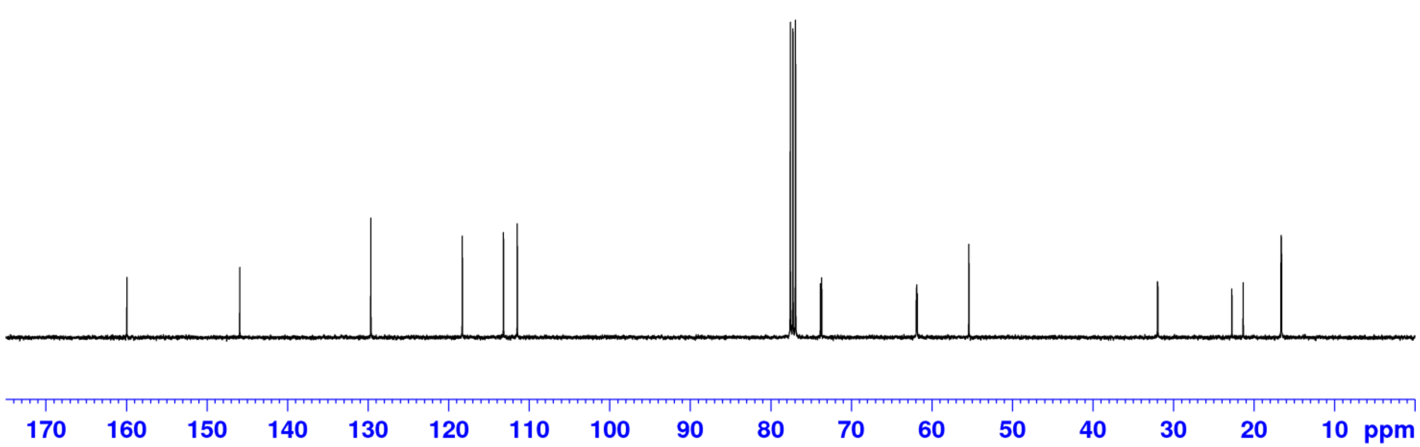


32.87
EtO
EtO
O
P
OH
OMe
9f
39 38 37 36 35 34 33 32 31 30 29 28 27 26 25 24 23 22
ppm

9f: ^{13}C NMR

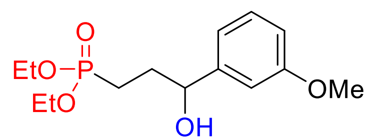


9f

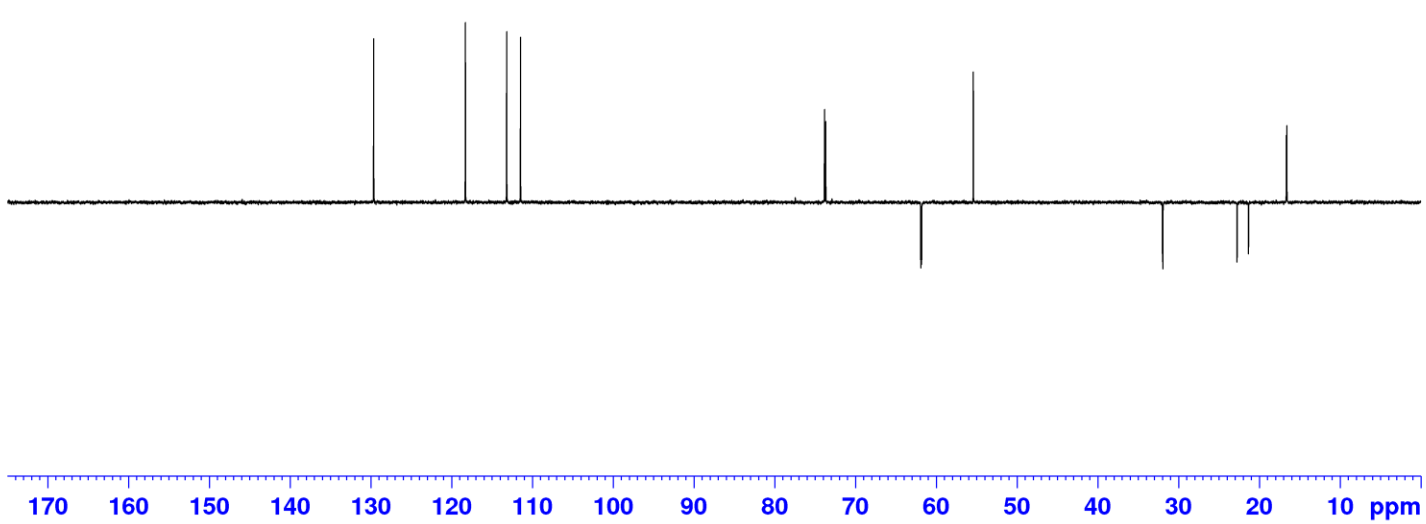

159.93
145.92
129.64
118.29
113.17
111.46
77.55
77.23
76.91
73.82
73.67
61.90
61.86
61.84
61.79
55.39
31.96
31.92
22.73
21.32
16.63
16.57
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

9f: ^{13}C DEPT135

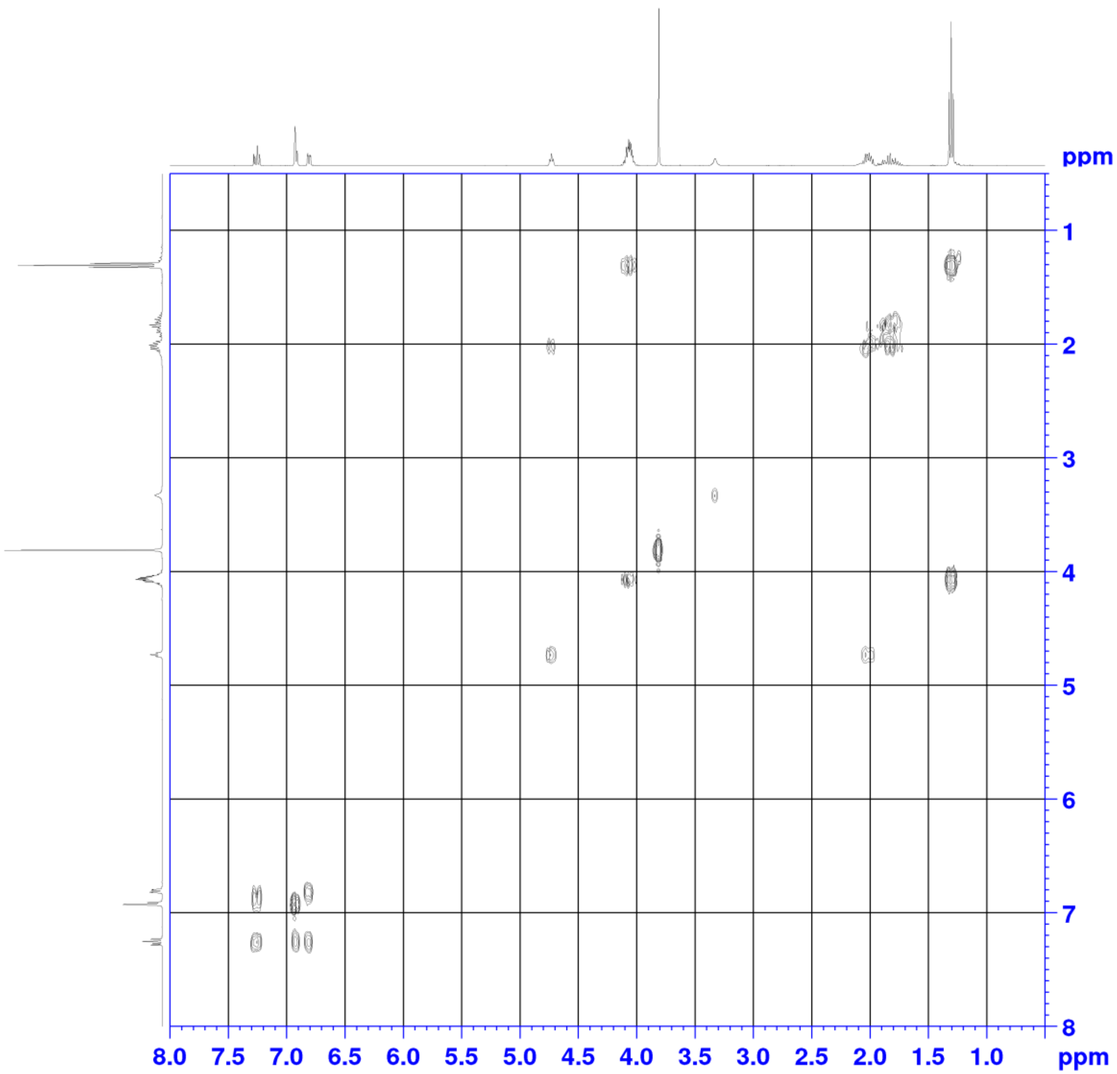
129.64
118.29
113.17
111.46
73.82
73.67
61.90
61.86
61.84
61.80
55.39
31.96
31.92
22.73
21.32
16.63
16.57



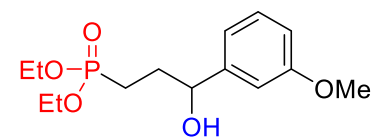
9f



9f: COSY



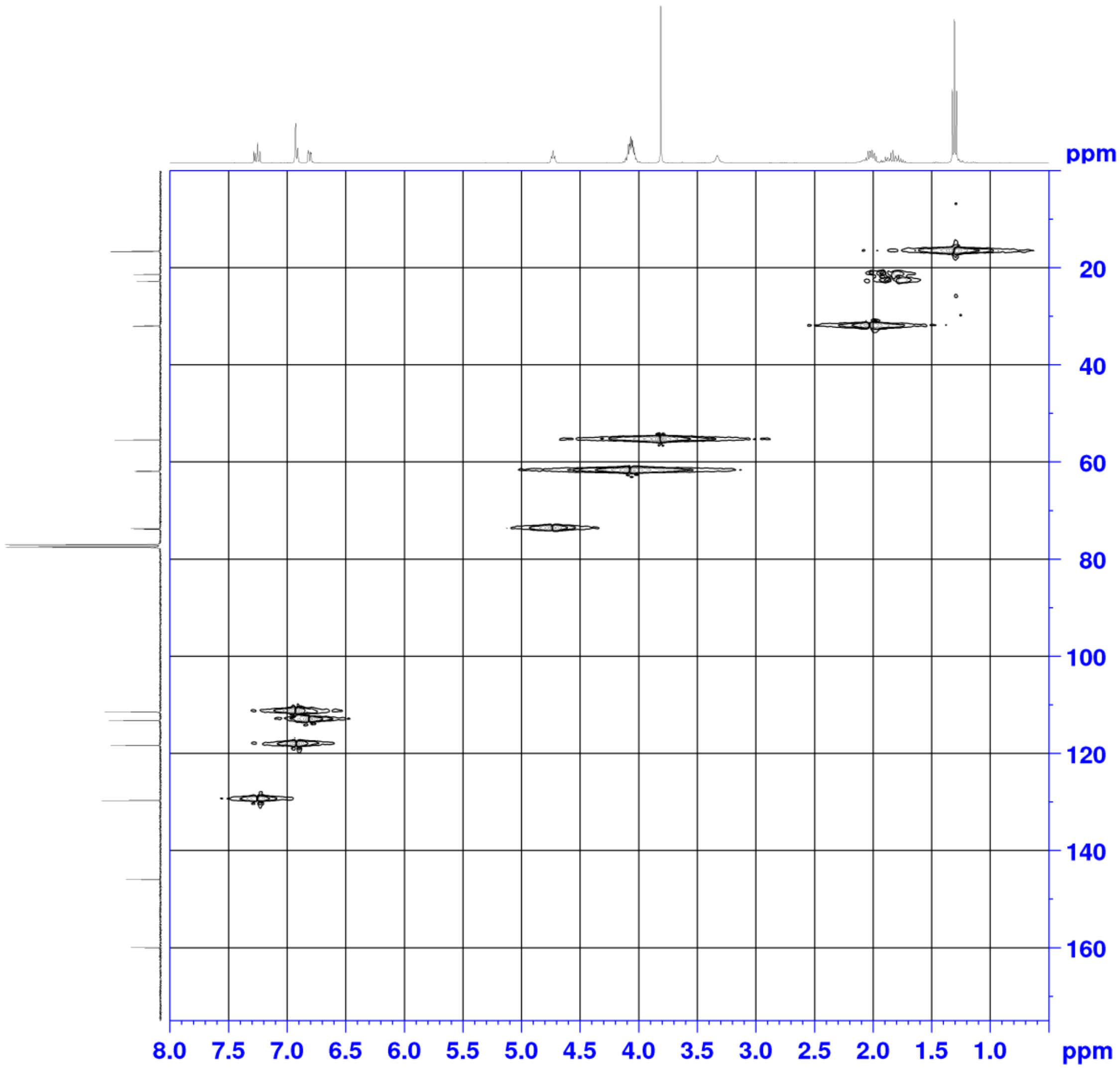
ppm
1
2
3
4
5
6
7
8
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

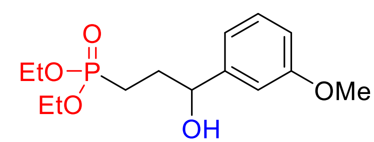


O
EtO
P
EtO
OH
OMe

9f

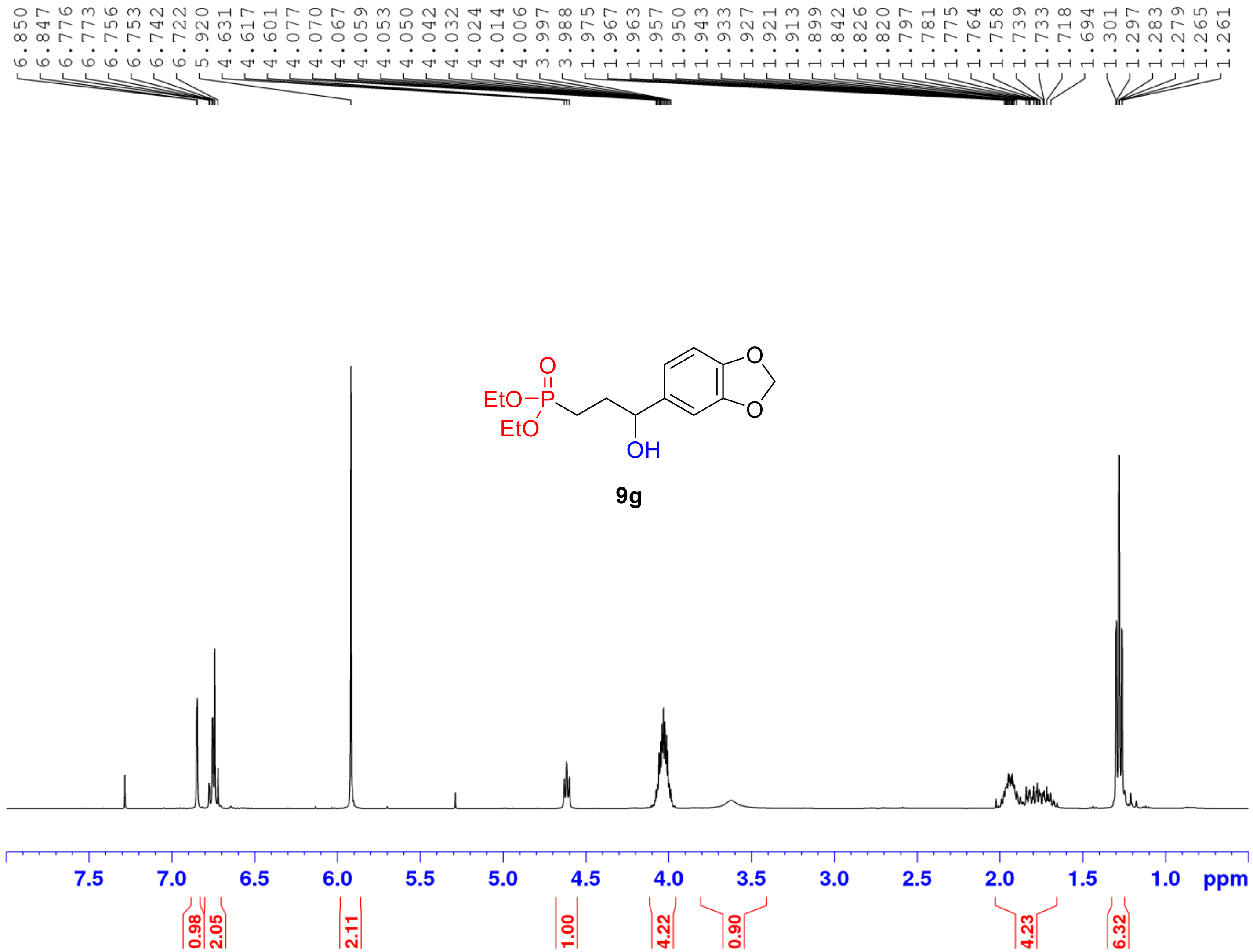
9f: ^{13}C HSQC



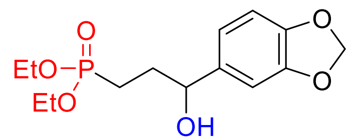


9f

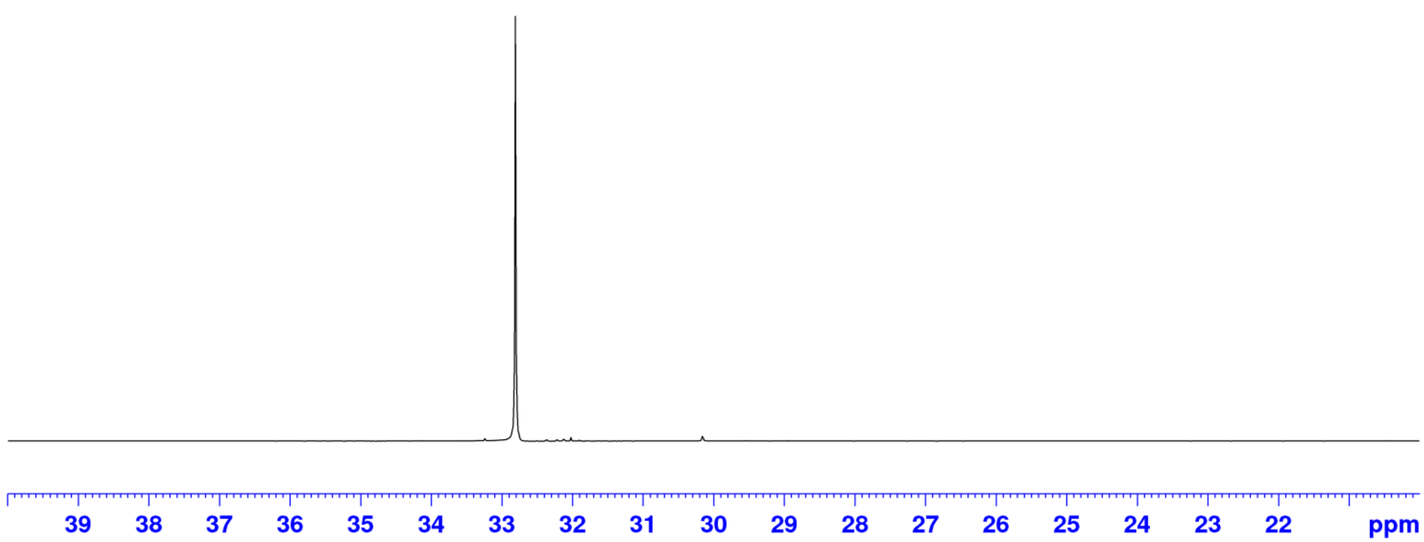
9g: ^{1}H NMR


6.850
6.847
6.776
6.773
6.756
6.753
6.742
6.722
5.920
4.631
4.617
4.601
4.077
4.070
4.067
4.059
4.053
4.050
4.042
4.032
4.024
4.014
4.006
3.997
3.988
1.975
1.967
1.963
1.957
1.950
1.943
1.933
1.927
1.921
1.913
1.899
1.842
1.826
1.820
1.797
1.781
1.775
1.764
1.758
1.739
1.733
1.718
1.694
1.301
1.297
1.283
1.279
1.265
1.261
EtO
EtO
O
P
O
O
OH
9g
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
0.98
2.05
2.11
1.00
4.22
0.90
4.23
6.32

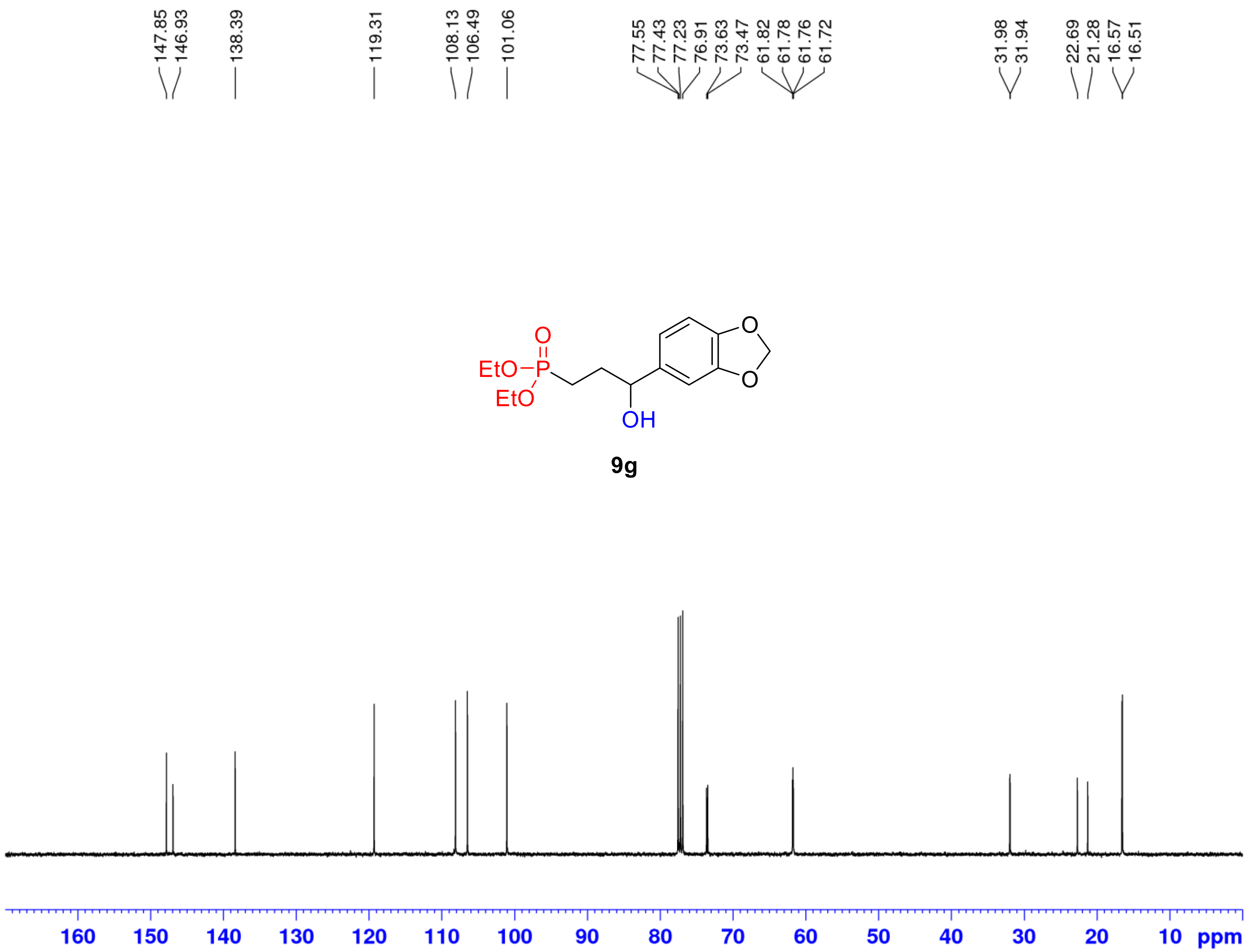
9g: ^{31}P NMR



9g

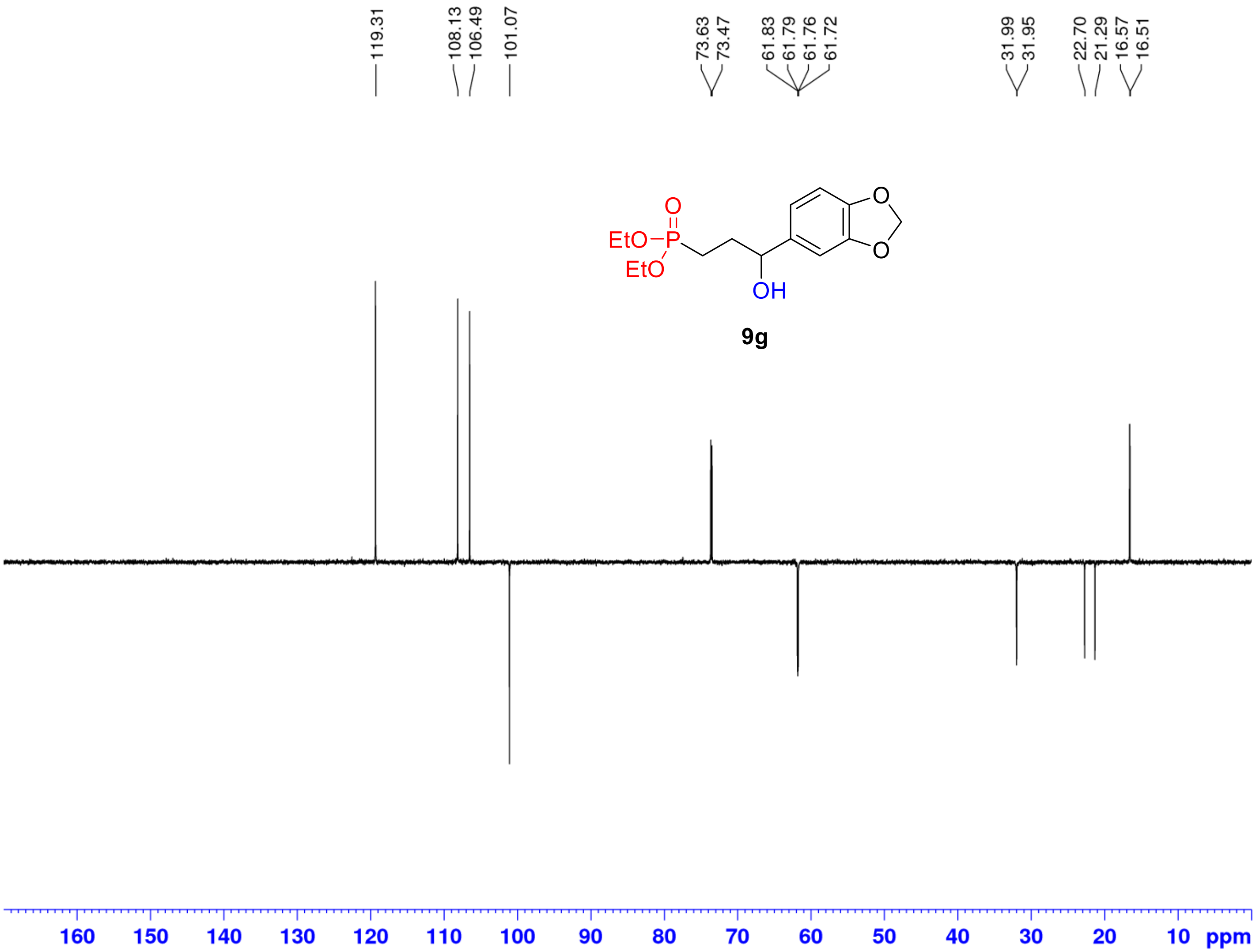


9g: ^{13}C NMR

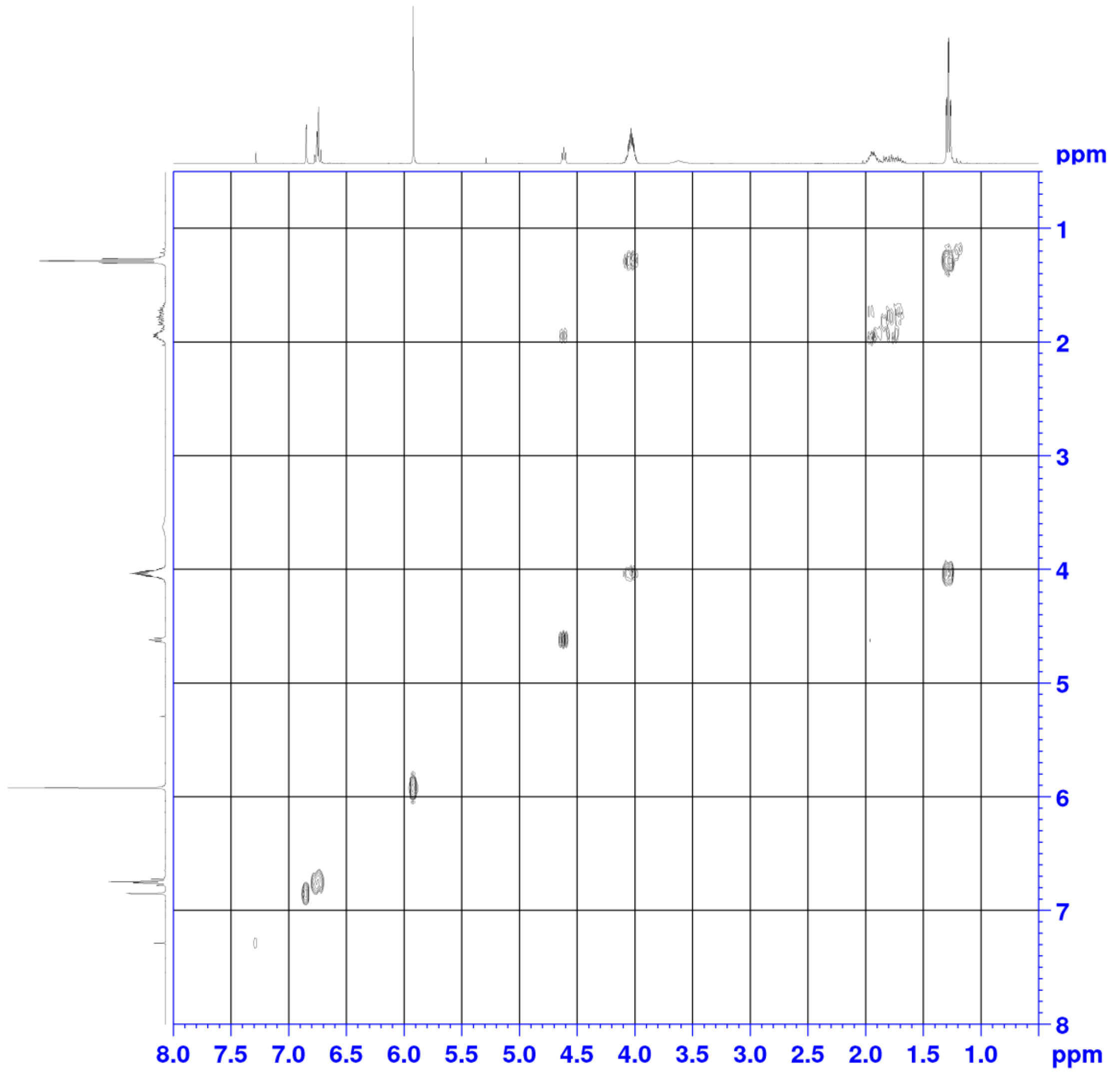


147.85
146.93
138.39
119.31
108.13
106.49
101.06
77.55
77.43
77.23
76.91
73.63
73.47
61.82
61.78
61.76
61.72
31.98
31.94
22.69
21.28
16.57
16.51
O
EtO
P
EtO
OH
O
O
9g
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

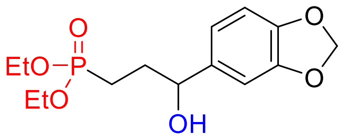
9g: ^{13}C DEPT135



9g: COSY



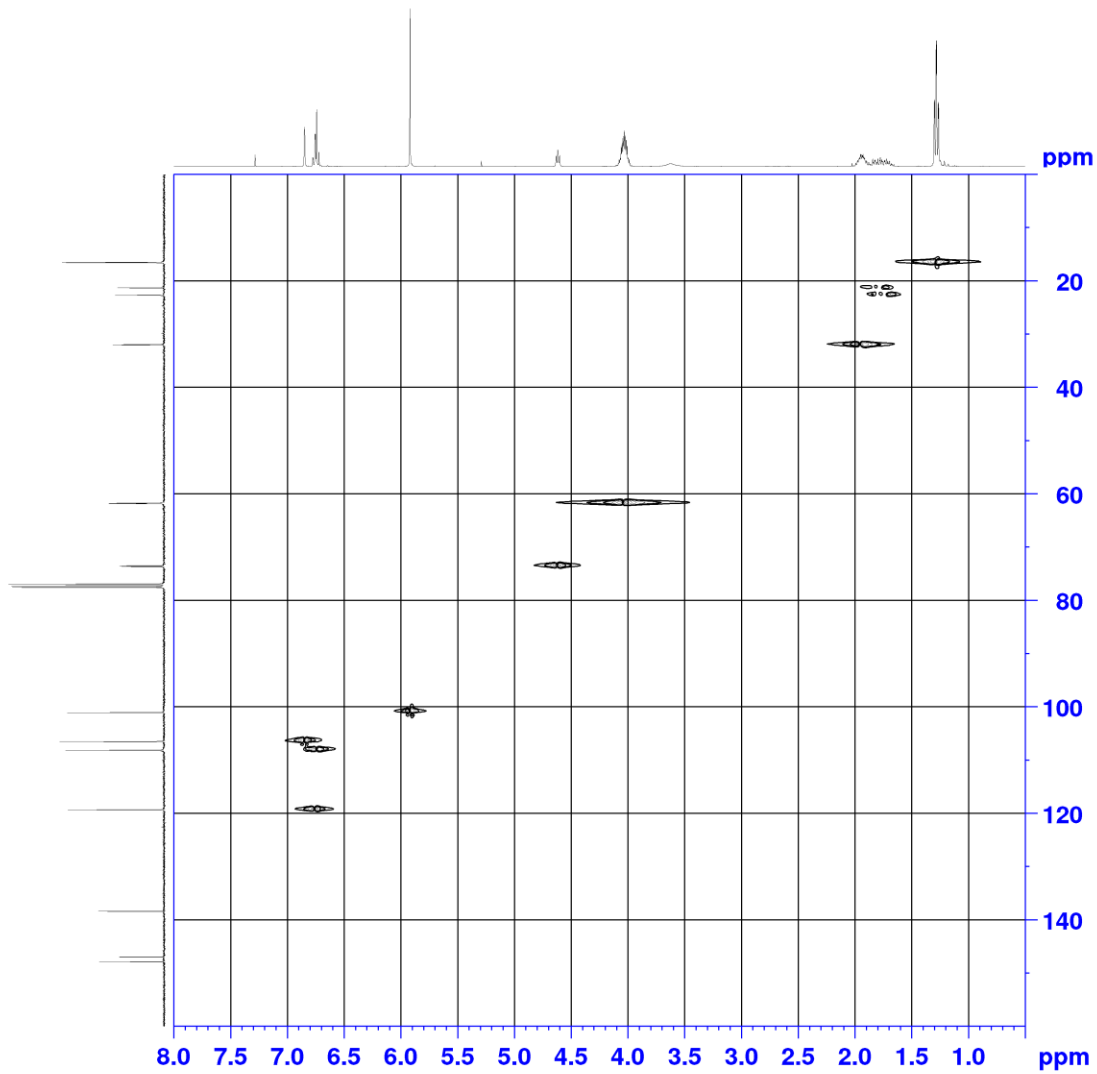
ppm
1
2
3
4
5
6
7
8
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

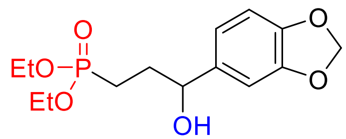


O
EtO
P
EtO
OH
O
O

9g

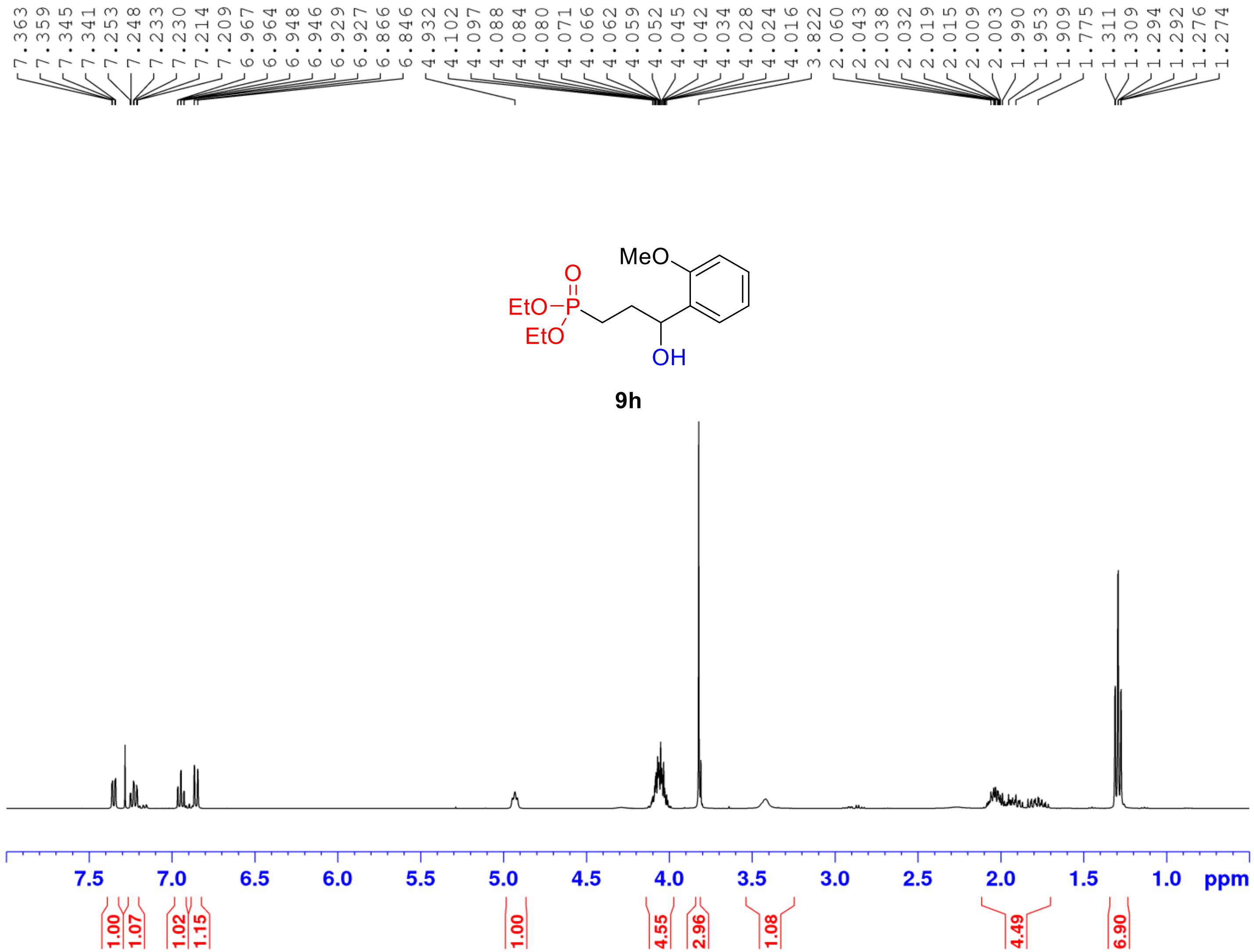
9g: ^{13}C HSQC





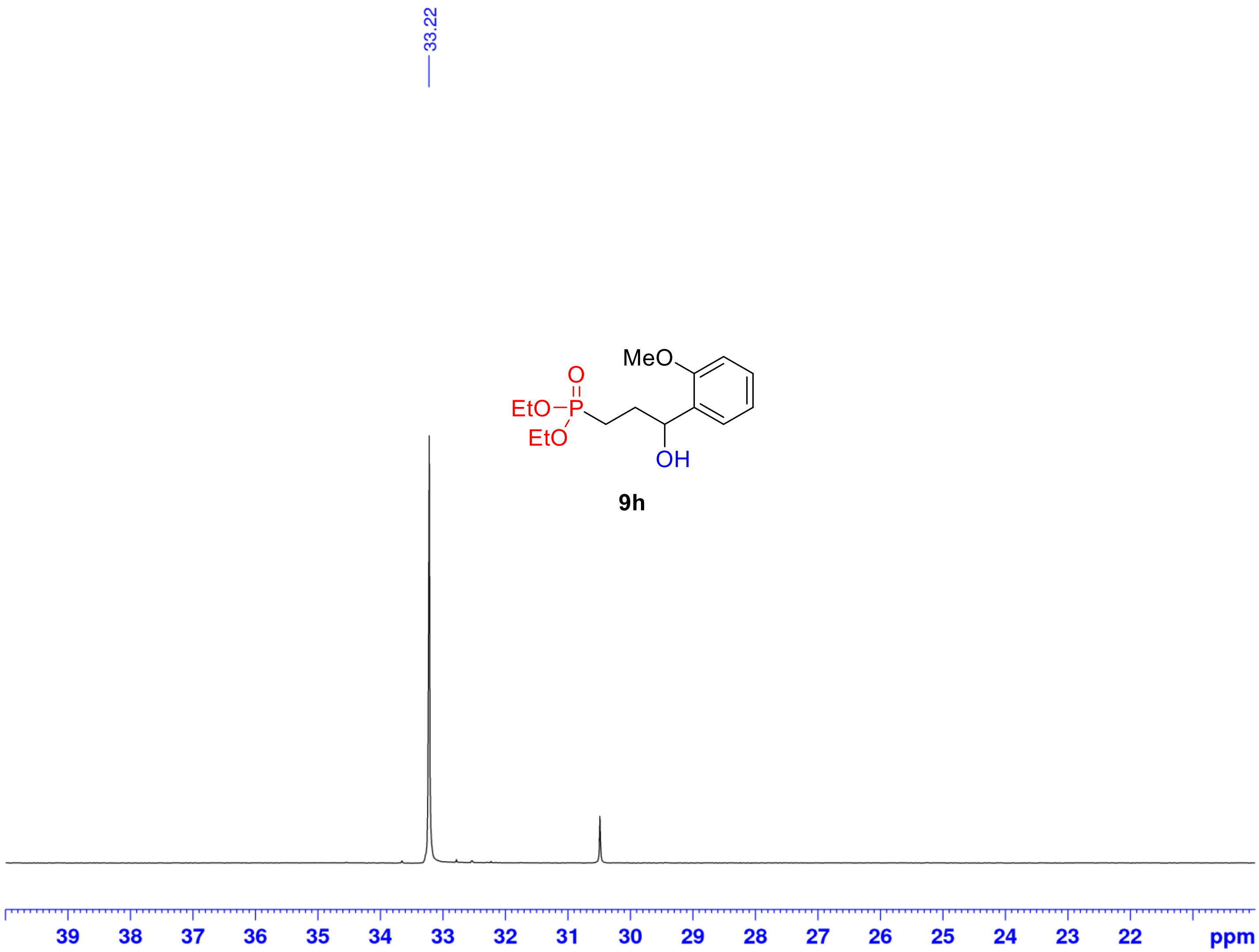
9g

9h: ^{1}H NMR



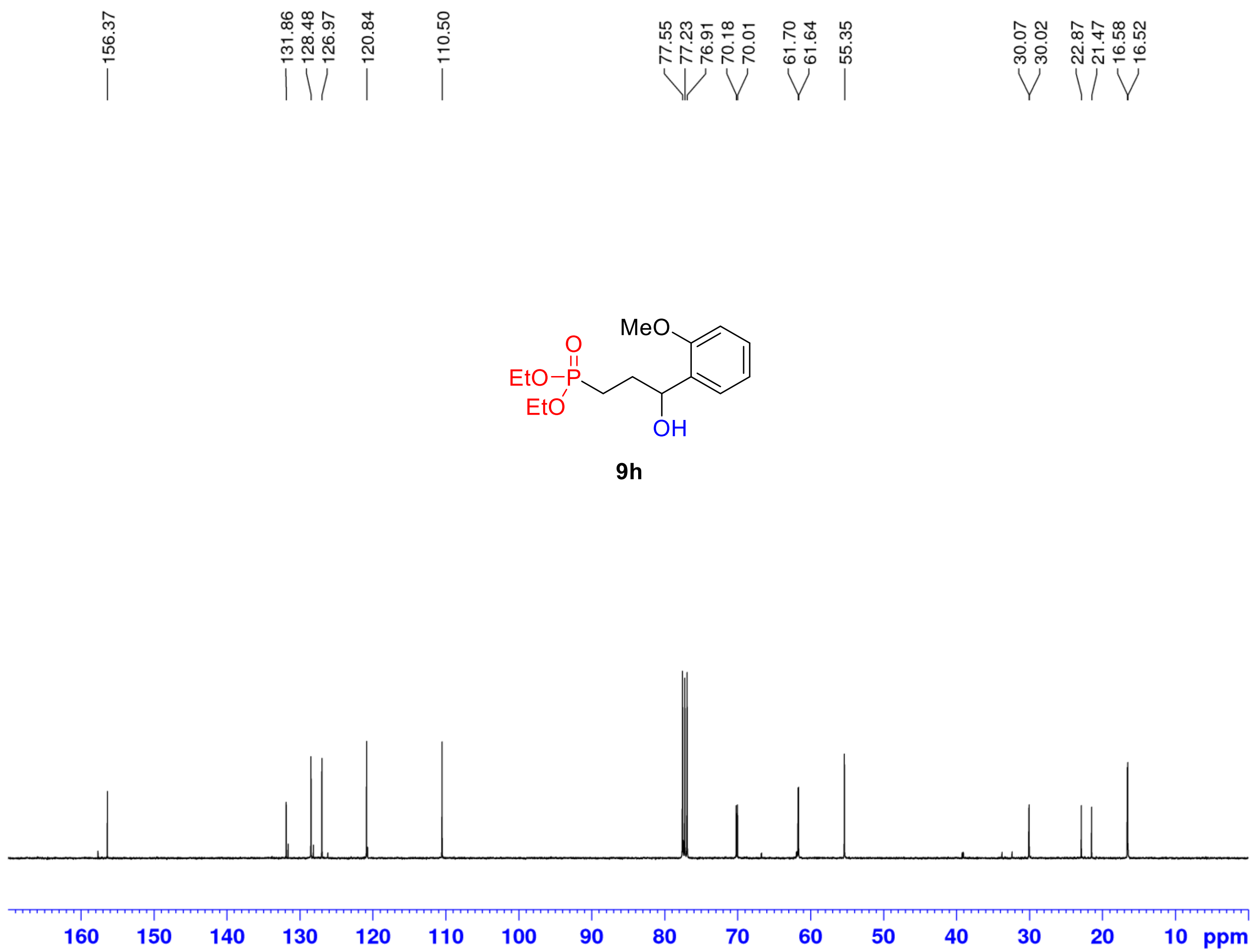
7.363 7.359 7.345 7.341 7.253 7.248 7.233 7.230 7.214 7.209 6.967 6.964 6.948 6.946 6.929 6.927 6.866 6.846 4.932 4.102 4.097 4.088 4.084 4.080 4.071 4.066 4.062 4.059 4.052 4.045 4.042 4.034 4.028 4.024 4.016 3.822 2.060 2.043 2.038 2.032 2.019 2.015 2.009 2.003 1.990 1.953 1.909 1.775 1.311 1.309 1.294 1.292 1.276 1.274
MeO
O
EtO
P
EtO
OH
9h
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
1.00 1.07 1.02 1.15 1.00 4.55 2.96 1.08 4.49 6.90

9h: ^{31}P NMR


33.22
39
38
37
36
35
34
33
32
31
30
29
28
27
26
25
24
23
22
ppm
MeO
O
EtO
P
EtO
OH

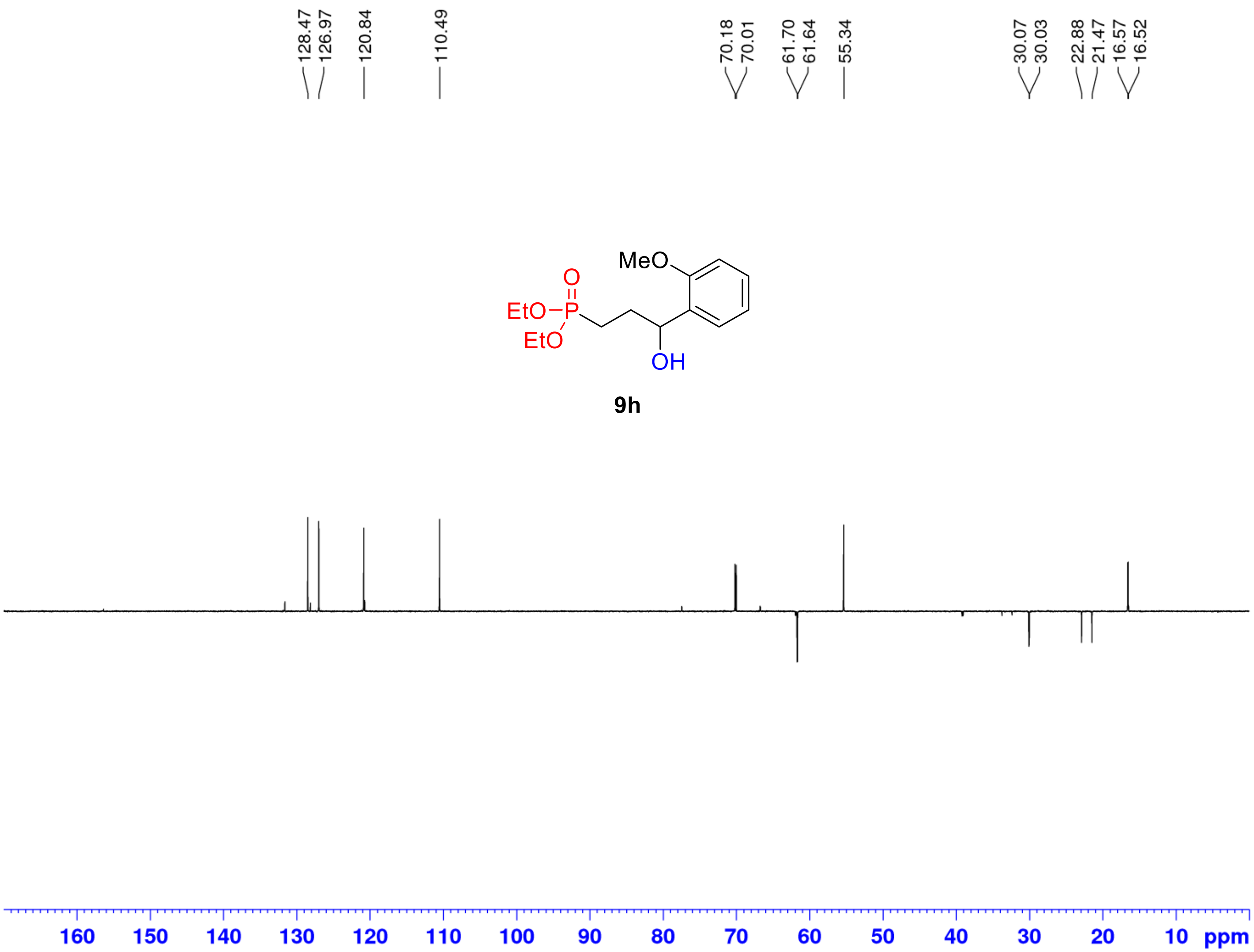
9h

9h: ^{13}C NMR

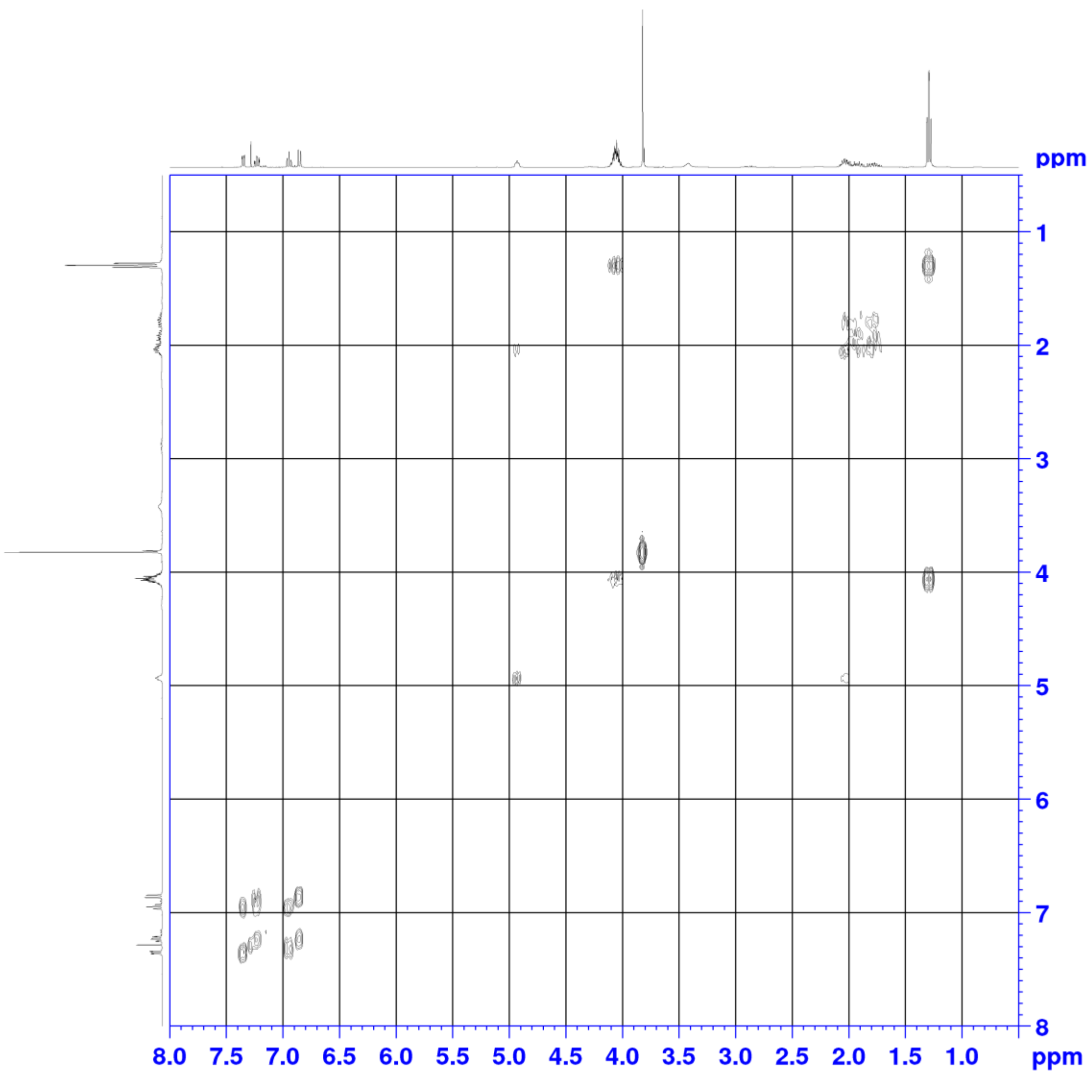


156.37
131.86
128.48
126.97
120.84
110.50
77.55
77.23
76.91
70.18
70.01
61.70
61.64
55.35
30.07
30.02
22.87
21.47
16.58
16.52
MeO
O
EtO
P
EtO
OH
9h
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

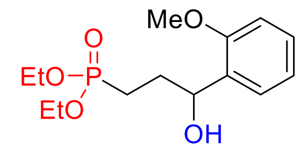
9h: ^{13}C DEPT135



9h: COSY



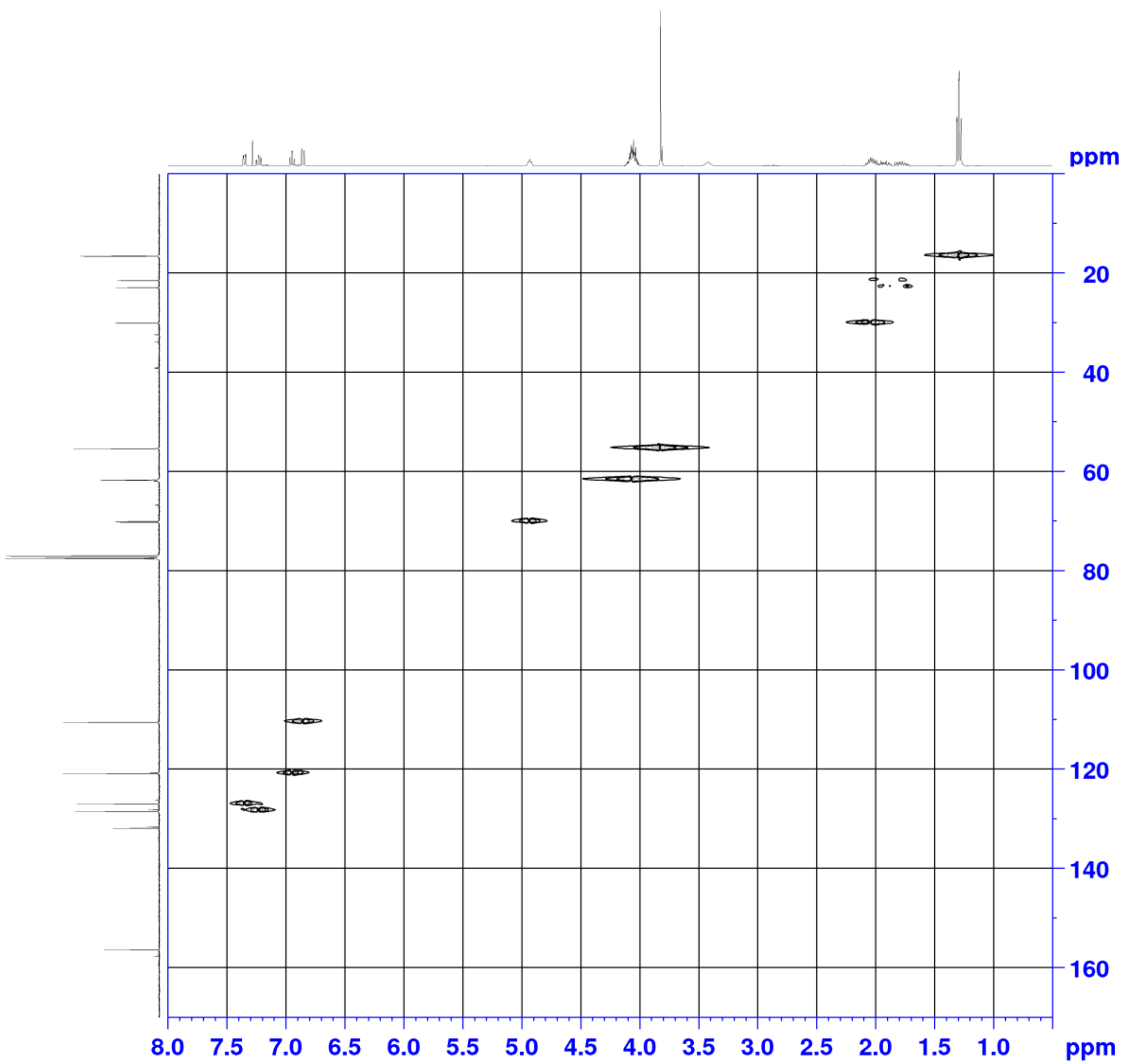
ppm
1
2
3
4
5
6
7
8
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

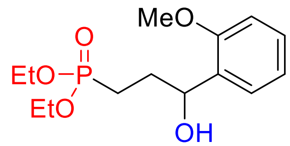


MeO
O
EtO
P
EtO
OH

9h

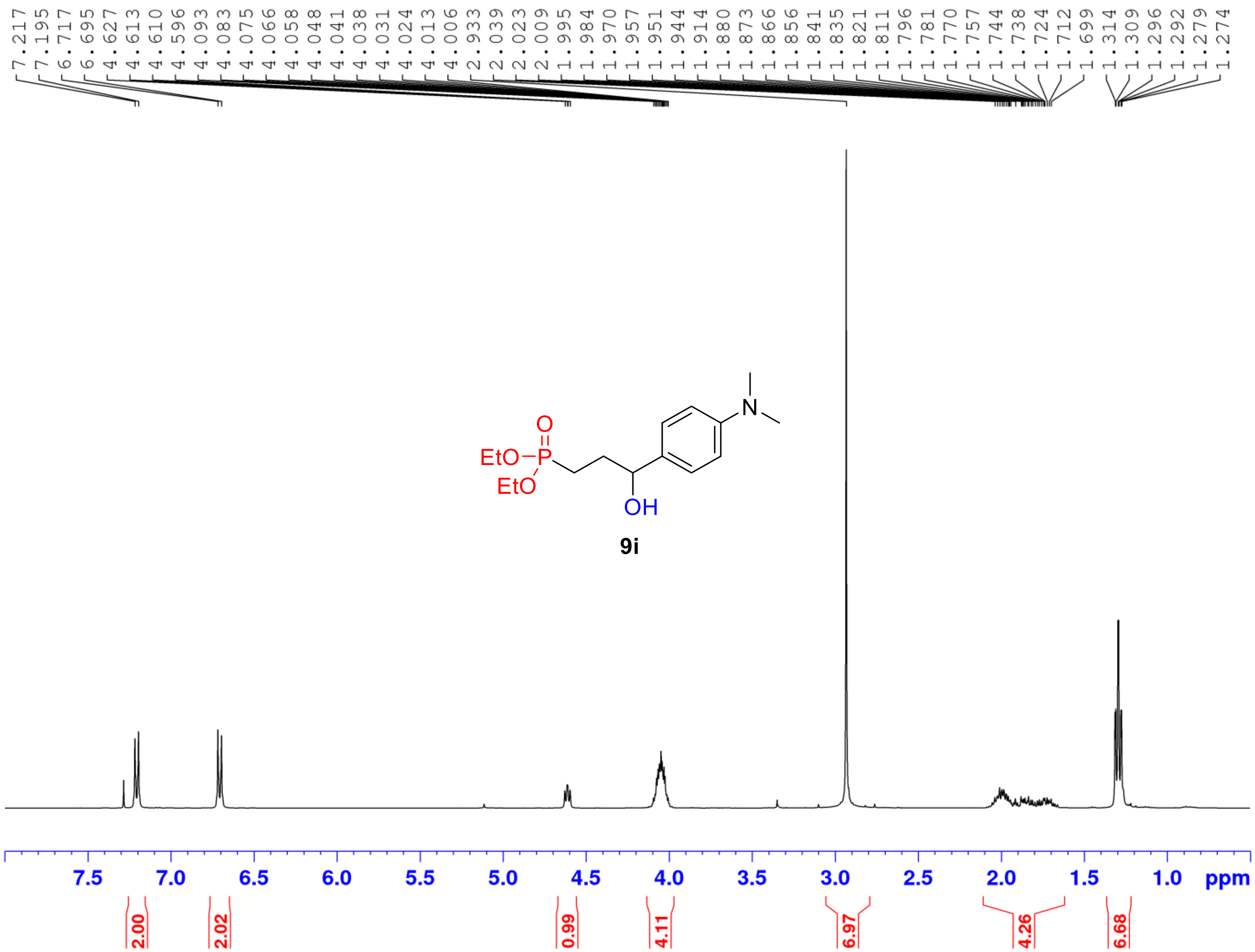
9h: ^{13}C HSQC





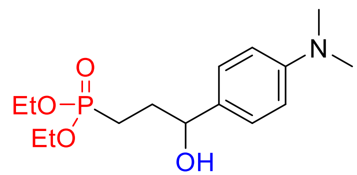
9h

9i: ^{1}H NMR

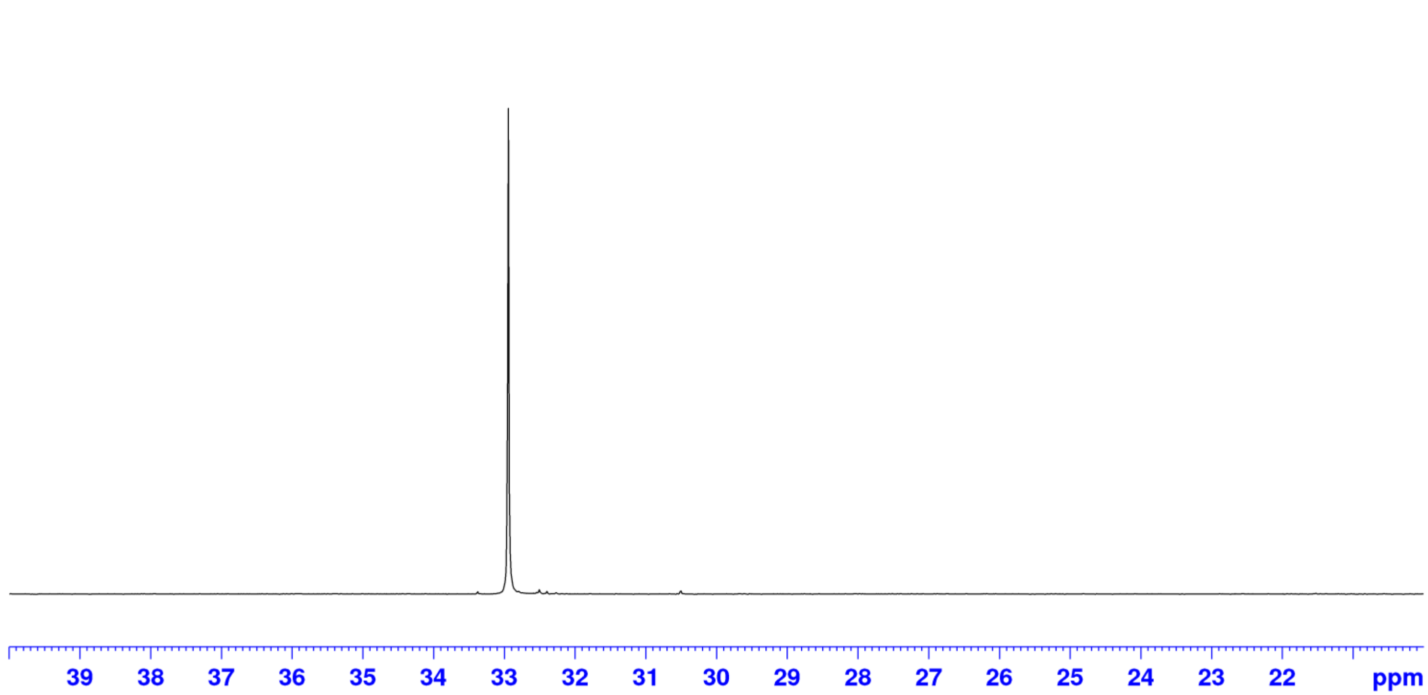


7.217
7.195
6.717
6.695
4.627
4.613
4.610
4.596
4.093
4.083
4.075
4.066
4.058
4.048
4.041
4.038
4.031
4.024
4.013
4.006
2.933
2.039
2.023
2.009
1.995
1.984
1.970
1.957
1.951
1.944
1.914
1.880
1.873
1.866
1.856
1.841
1.835
1.821
1.811
1.796
1.781
1.770
1.757
1.744
1.738
1.724
1.712
1.699
1.314
1.309
1.296
1.292
1.279
1.274
EtO
EtO
O
P
N
OH
9i
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
2.00
2.02
0.99
4.11
6.97
4.26
6.68

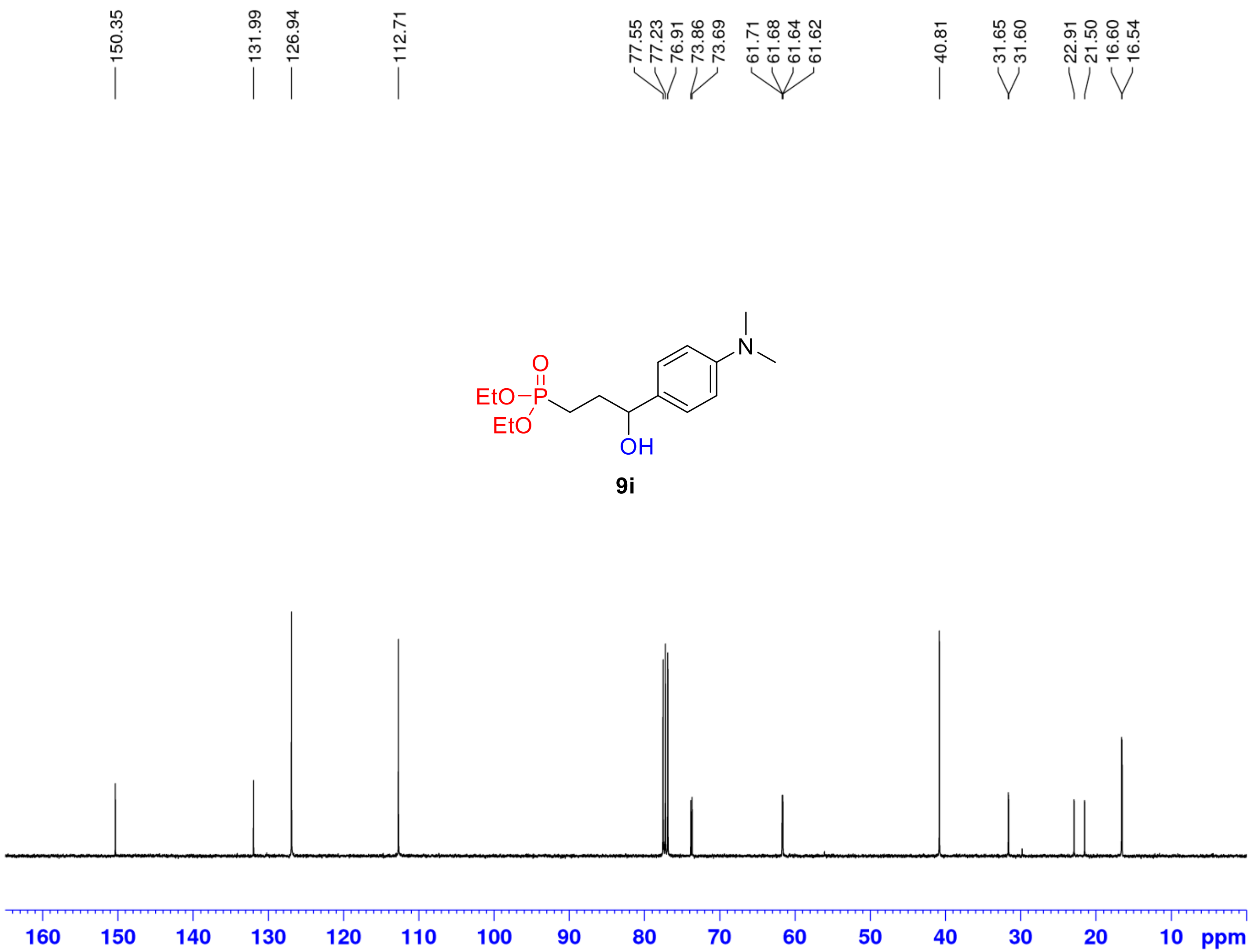
9i: ^{31}P NMR



9i

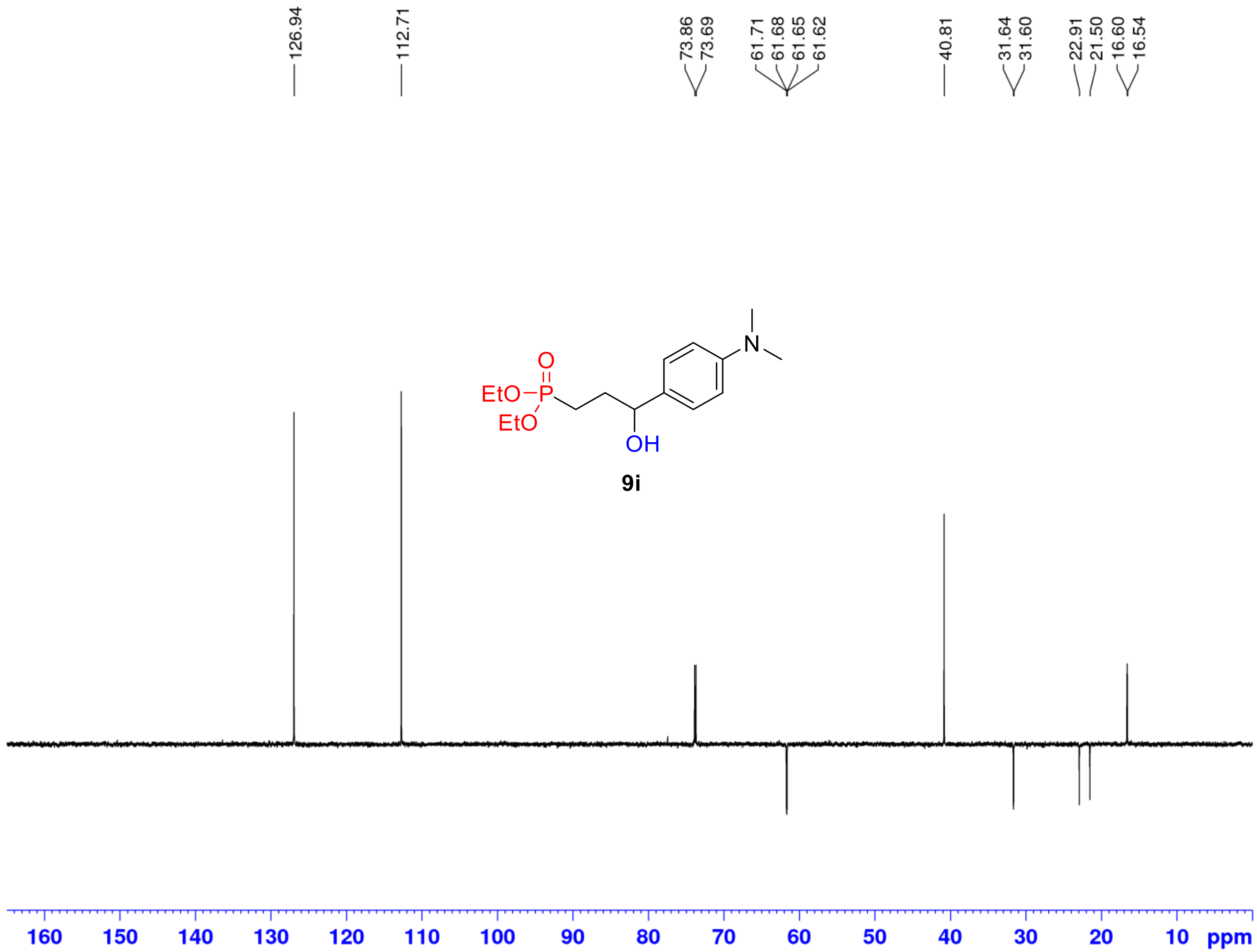


9i: ^{13}C NMR

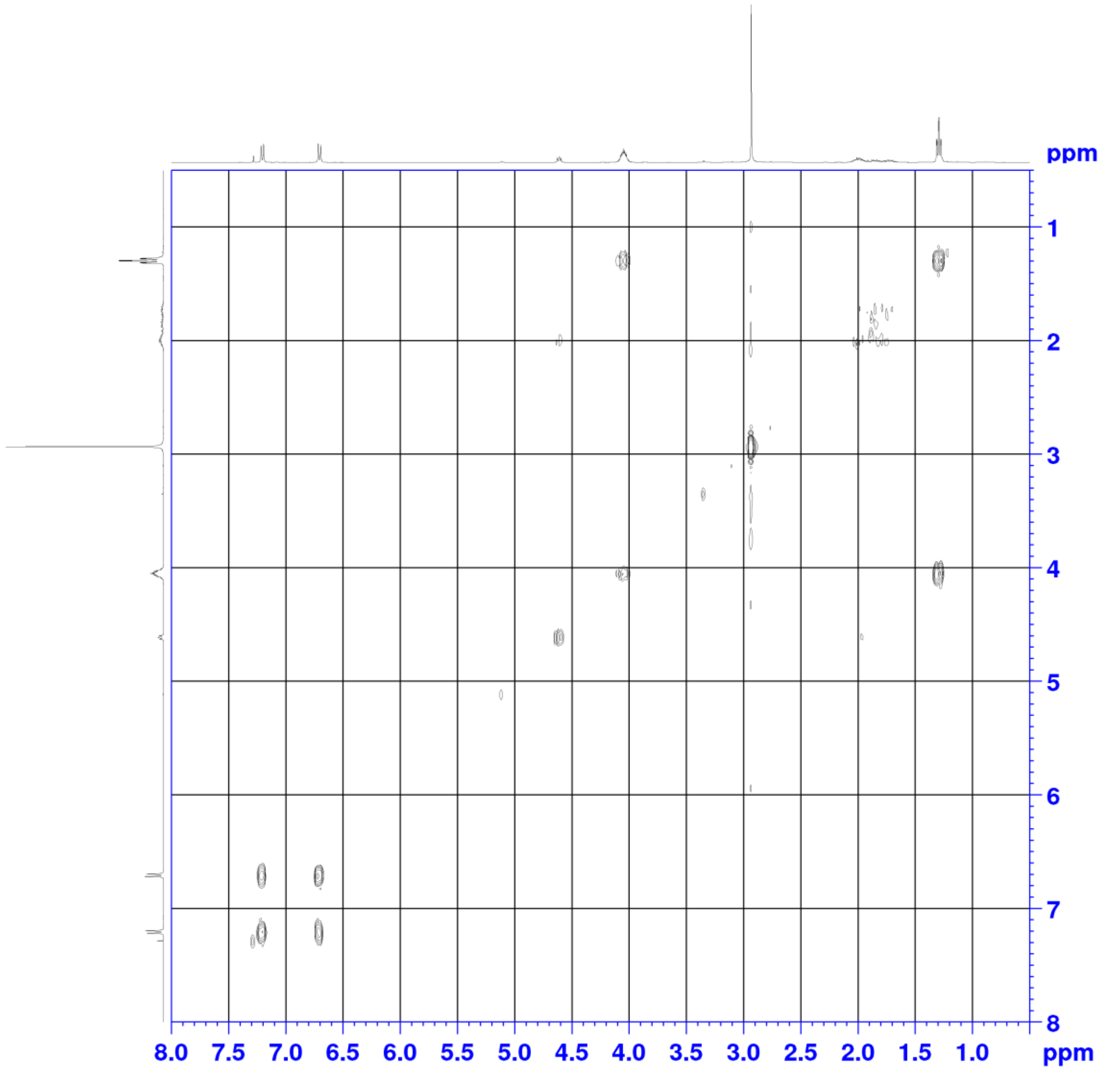


150.35
131.99
126.94
112.71
77.55
77.23
76.91
73.86
73.69
61.71
61.68
61.64
61.62
40.81
31.65
31.60
22.91
21.50
16.60
16.54
O
EtO
P
EtO
OH
N
9i
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

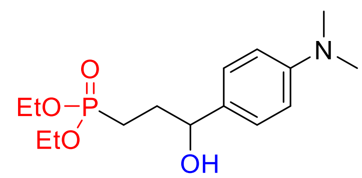
9i: ^{13}C DEPT135



9i: COSY



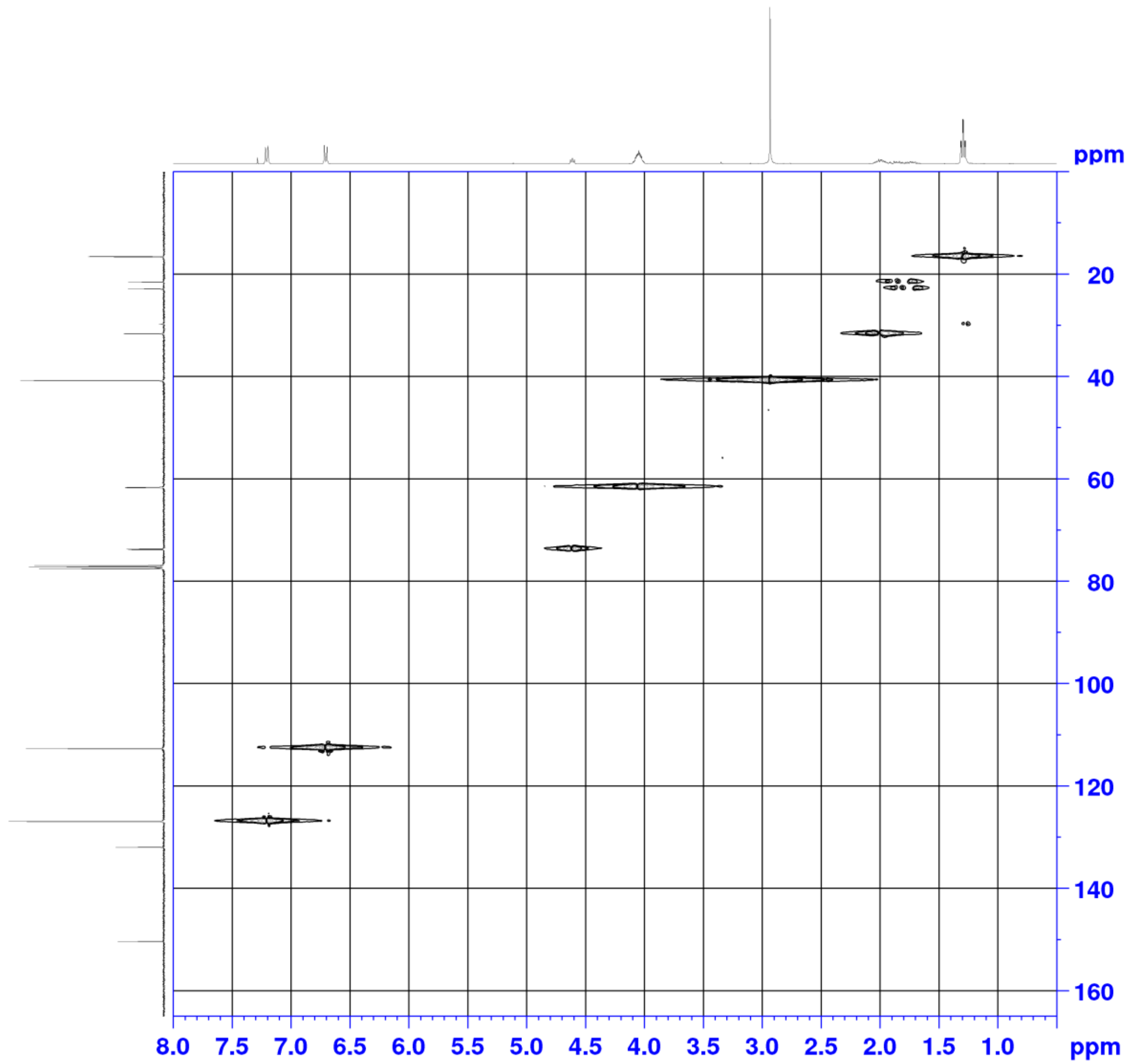
ppm
1
2
3
4
5
6
7
8
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

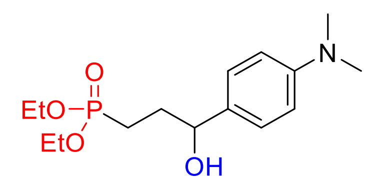


O
EtO
P
EtO
OH
N

9i

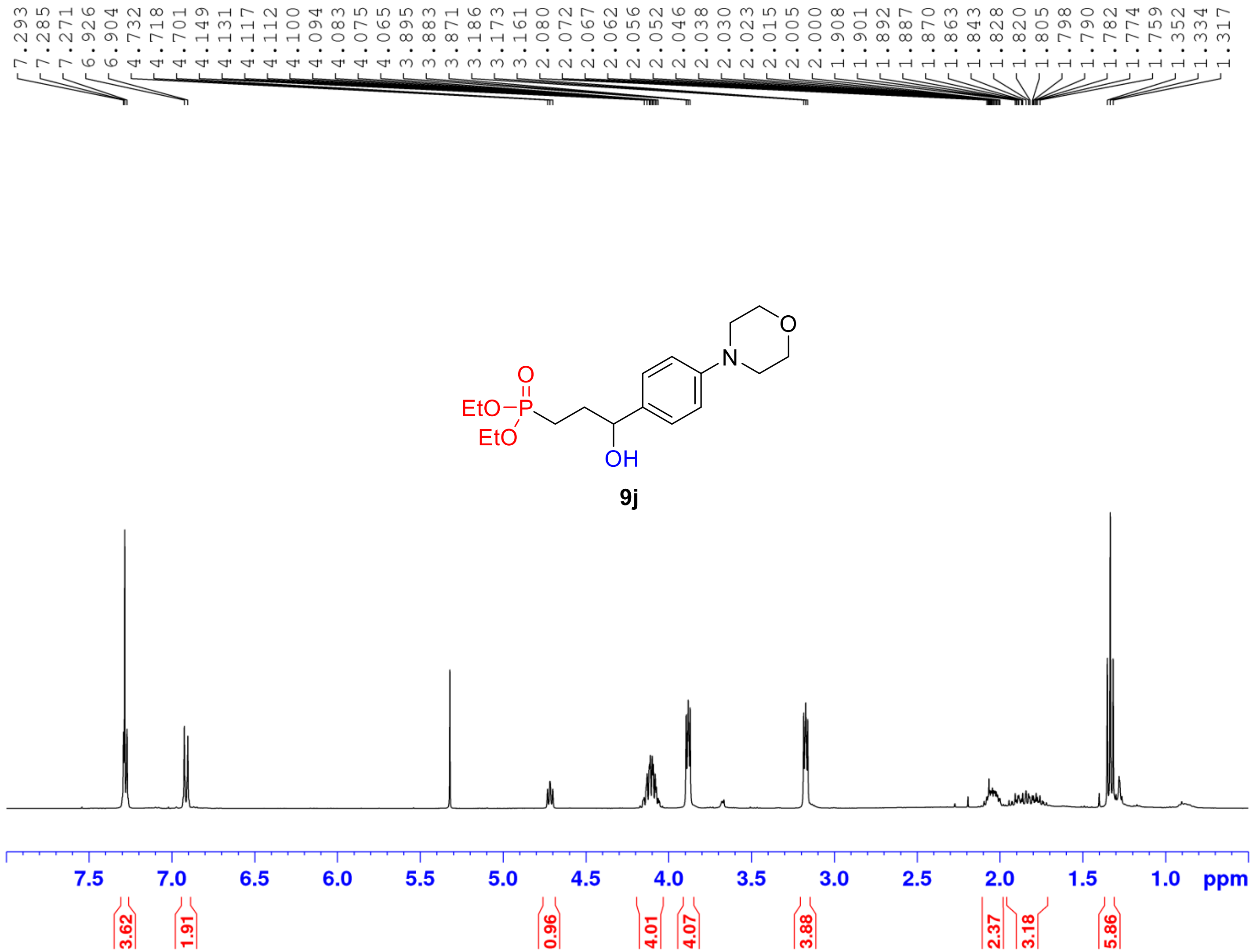
9i: ^{13}C HSQC





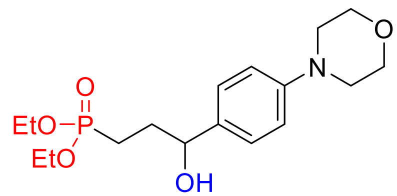
9i

9j: ^{1}H NMR



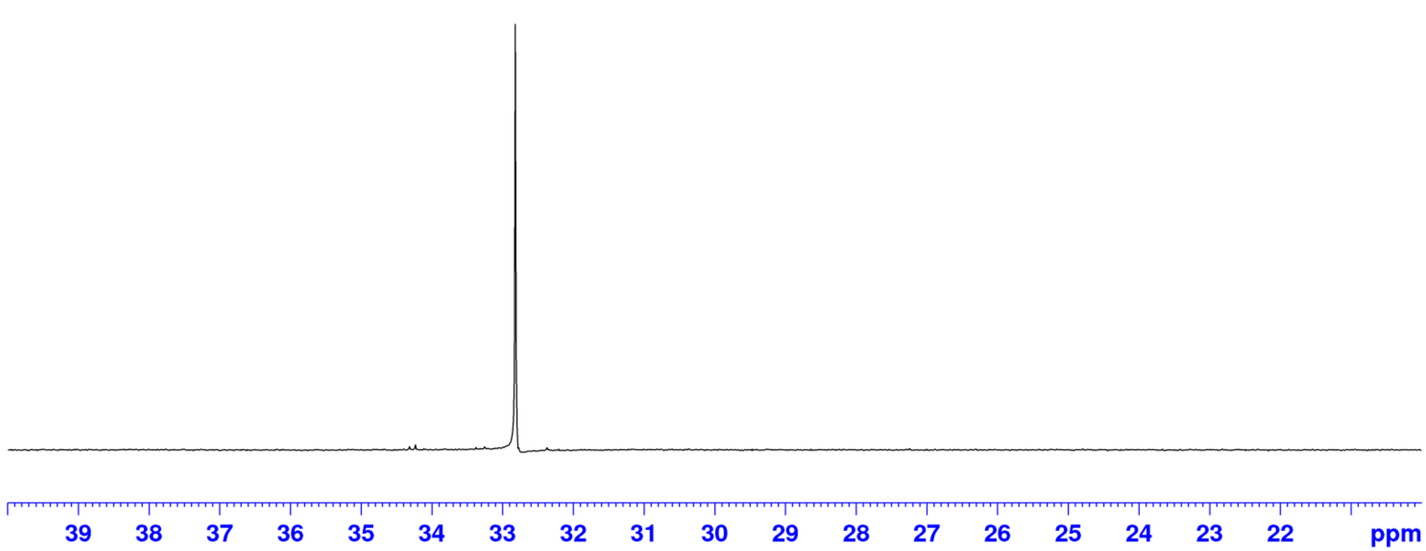
7.293
7.285
7.271
6.926
6.904
4.732
4.718
4.701
4.149
4.131
4.117
4.112
4.100
4.094
4.083
4.075
4.065
3.895
3.883
3.871
3.186
3.173
3.161
2.080
2.072
2.067
2.062
2.056
2.052
2.046
2.038
2.030
2.023
2.015
2.005
2.000
1.908
1.901
1.892
1.887
1.870
1.863
1.843
1.828
1.820
1.805
1.798
1.790
1.782
1.774
1.759
1.352
1.334
1.317
EtO
EtO
P
O
OH
N
O
9j
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
3.62
1.91
0.96
4.01
4.07
3.88
2.37
3.18
5.86

9j: ^{31}P NMR



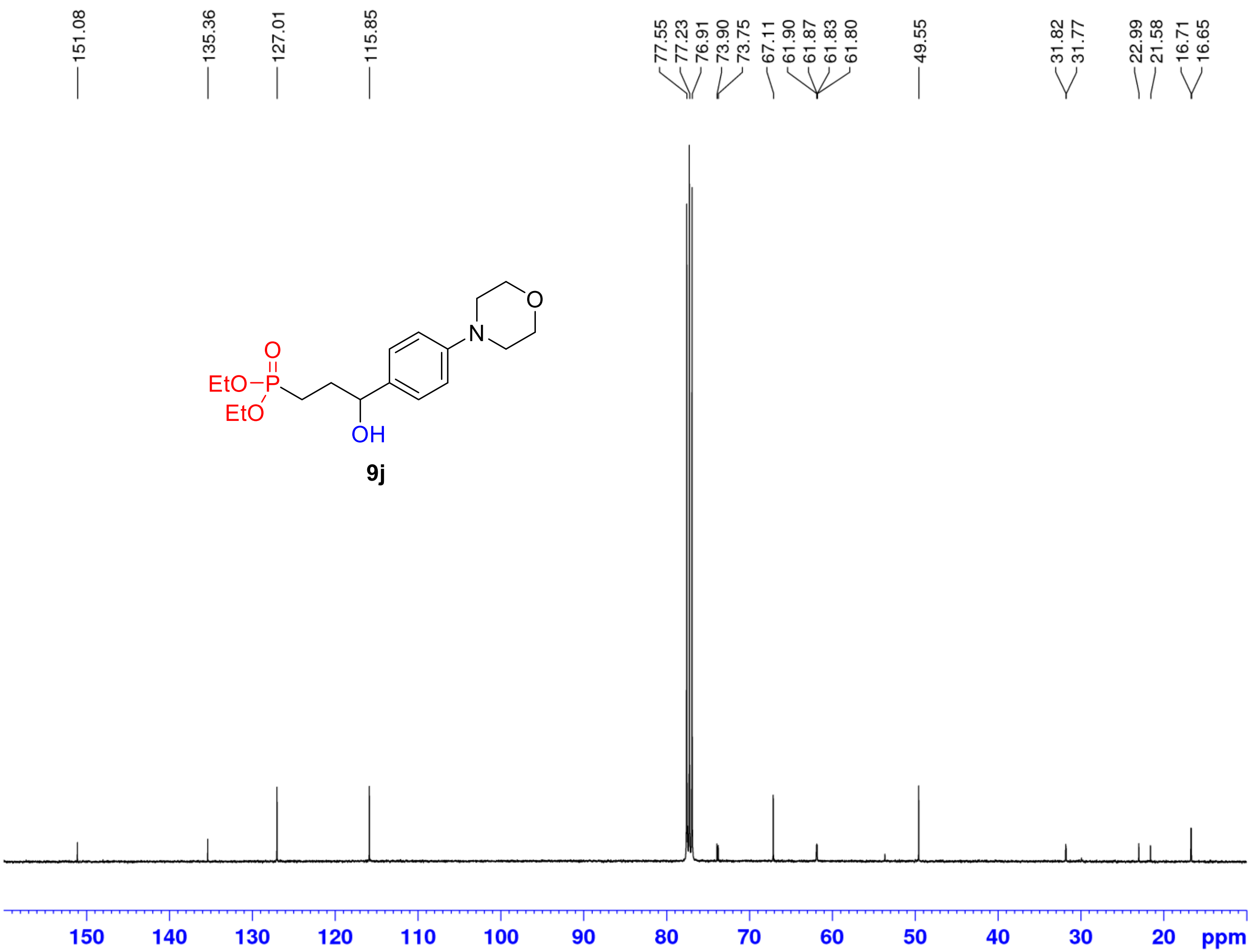
EtO
EtO
OH

9j

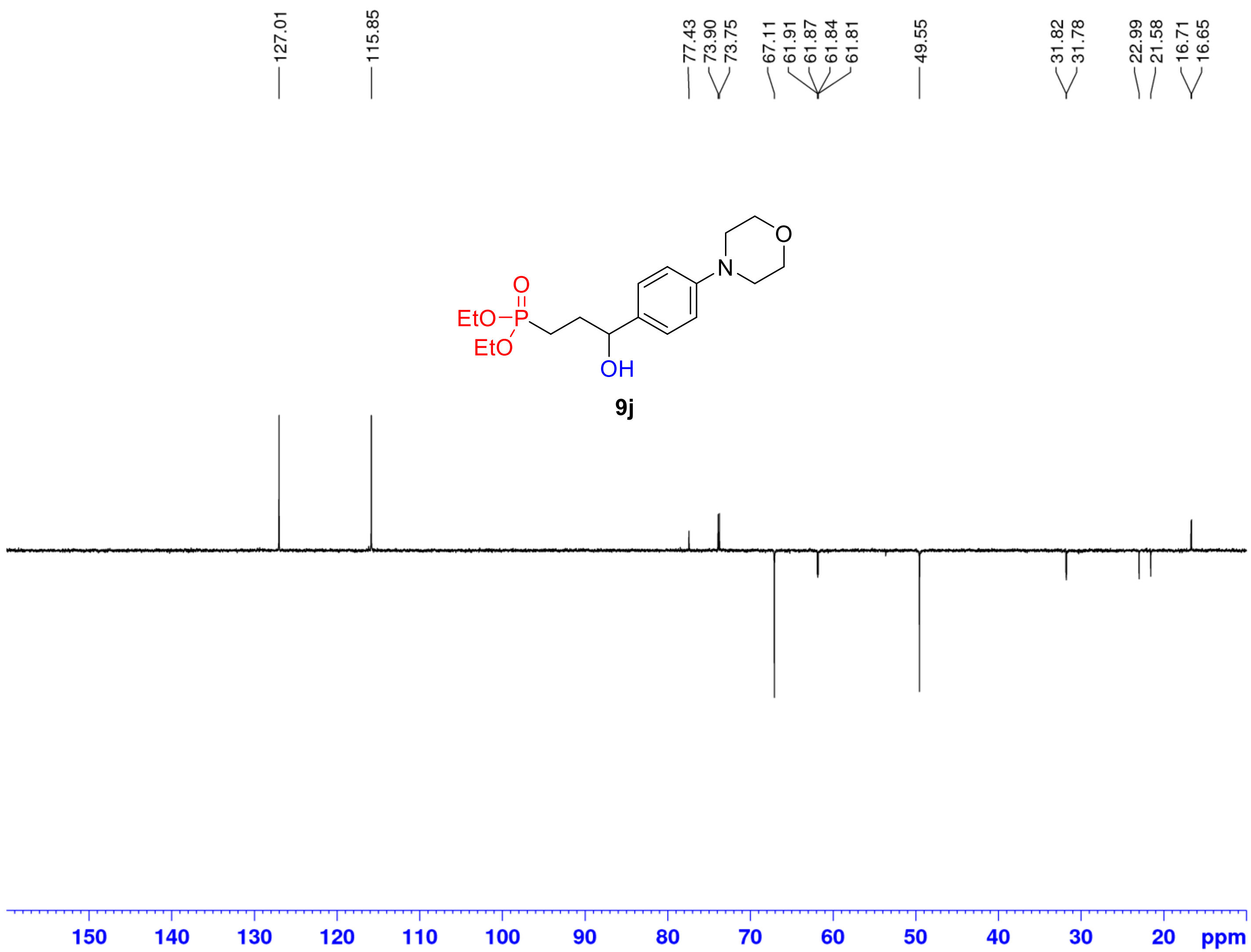


32.82
39
38
37
36
35
34
33
32
31
30
29
28
27
26
25
24
23
22
ppm

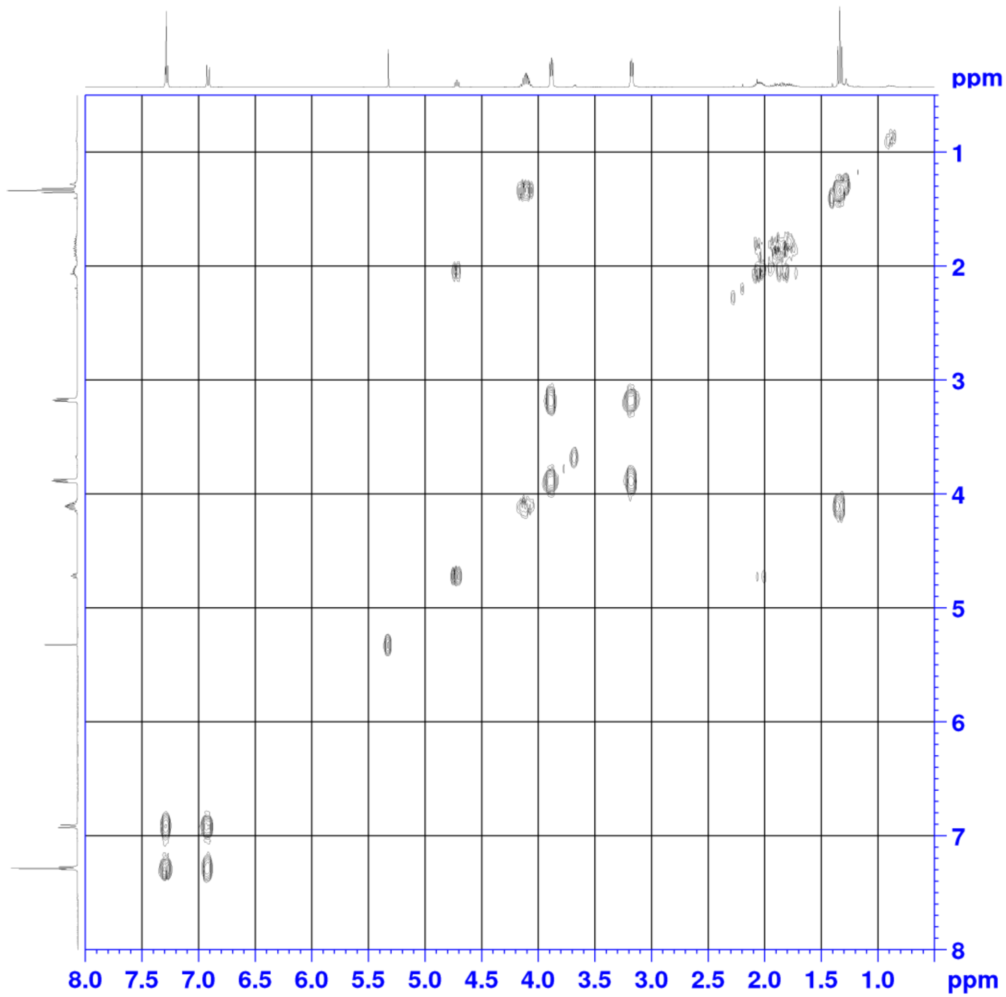
9j: ^{13}C NMR


151.08
135.36
127.01
115.85
77.55
77.23
76.91
73.90
73.75
67.11
61.90
61.87
61.83
61.80
49.55
31.82
31.77
22.99
21.58
16.71
16.65
EtO
EtO
O
P
OH
N
O
9j
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

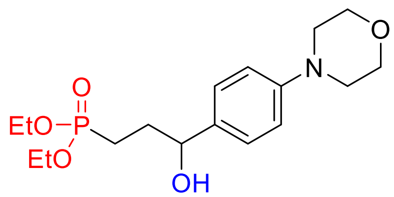
9j: ^{13}C DEPT135



9j: COSY



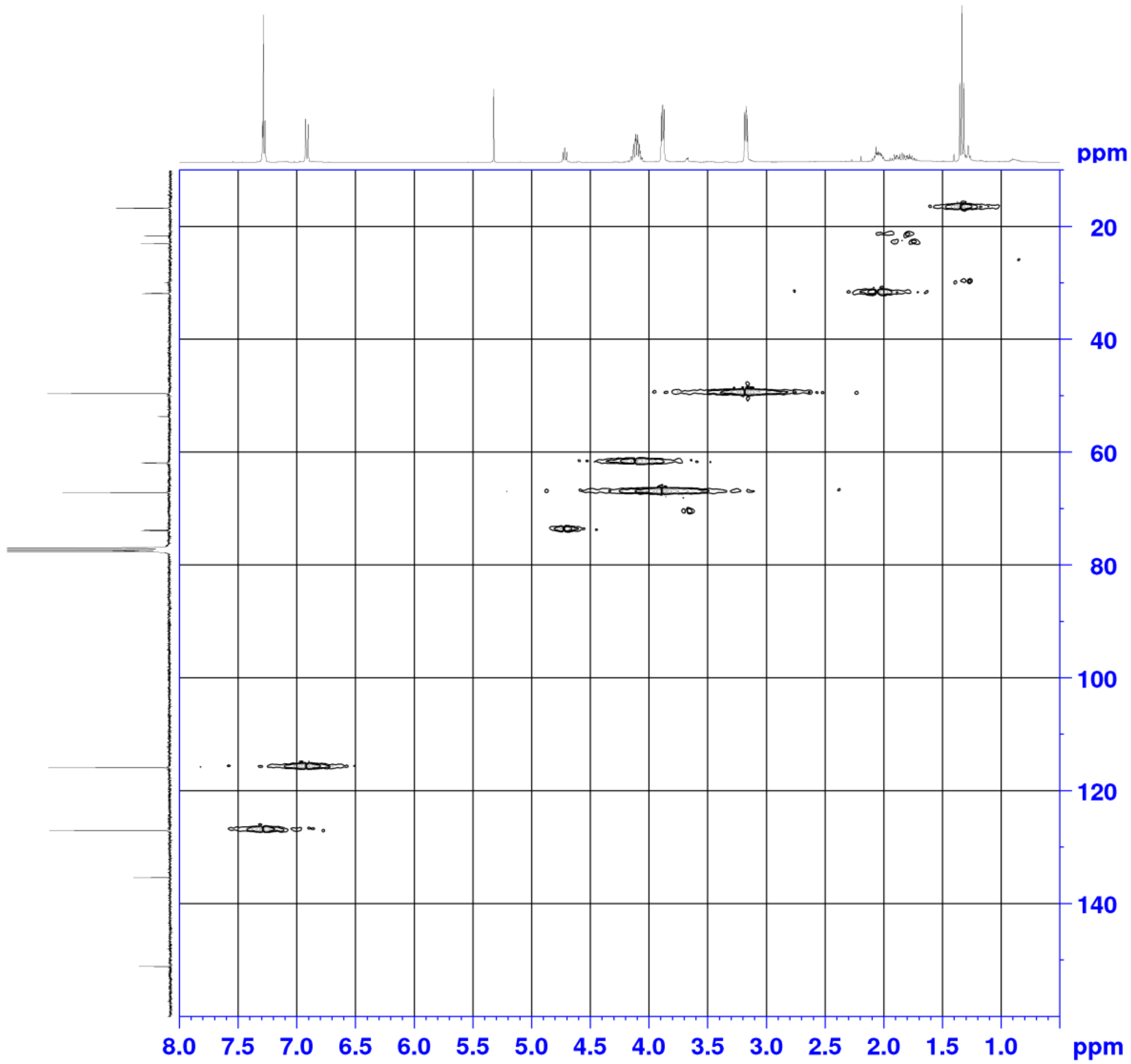
ppm
1
2
3
4
5
6
7
8
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

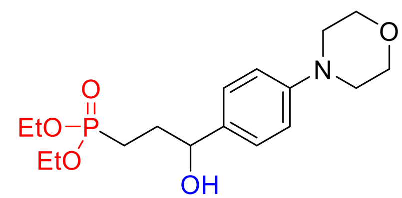


O
EtO
P
EtO
OH
N
O

9j

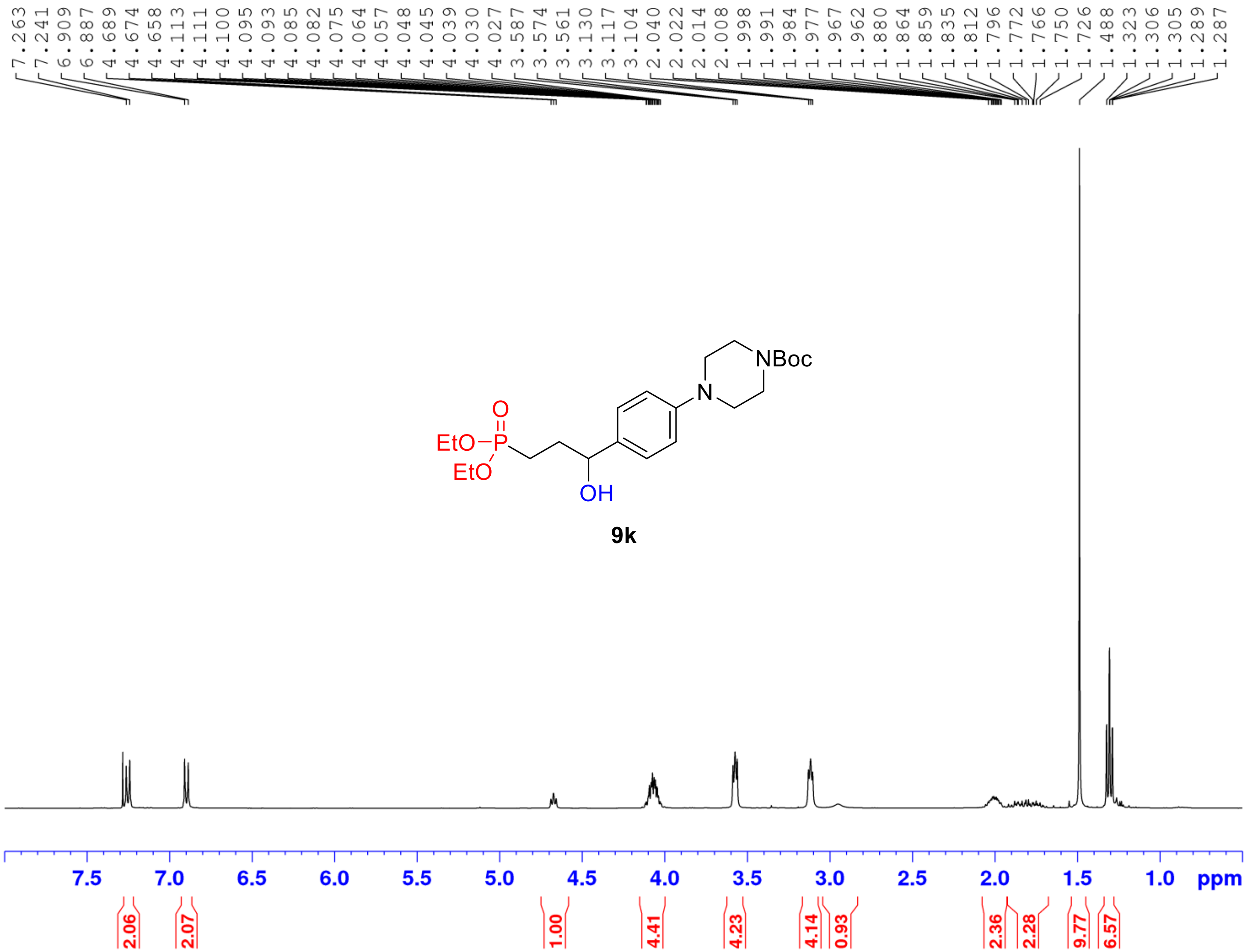
9j: ^{13}C HSQC



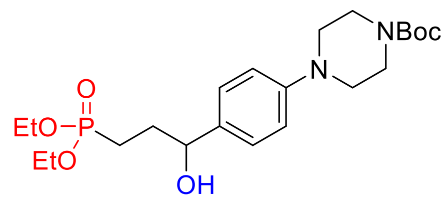


9j

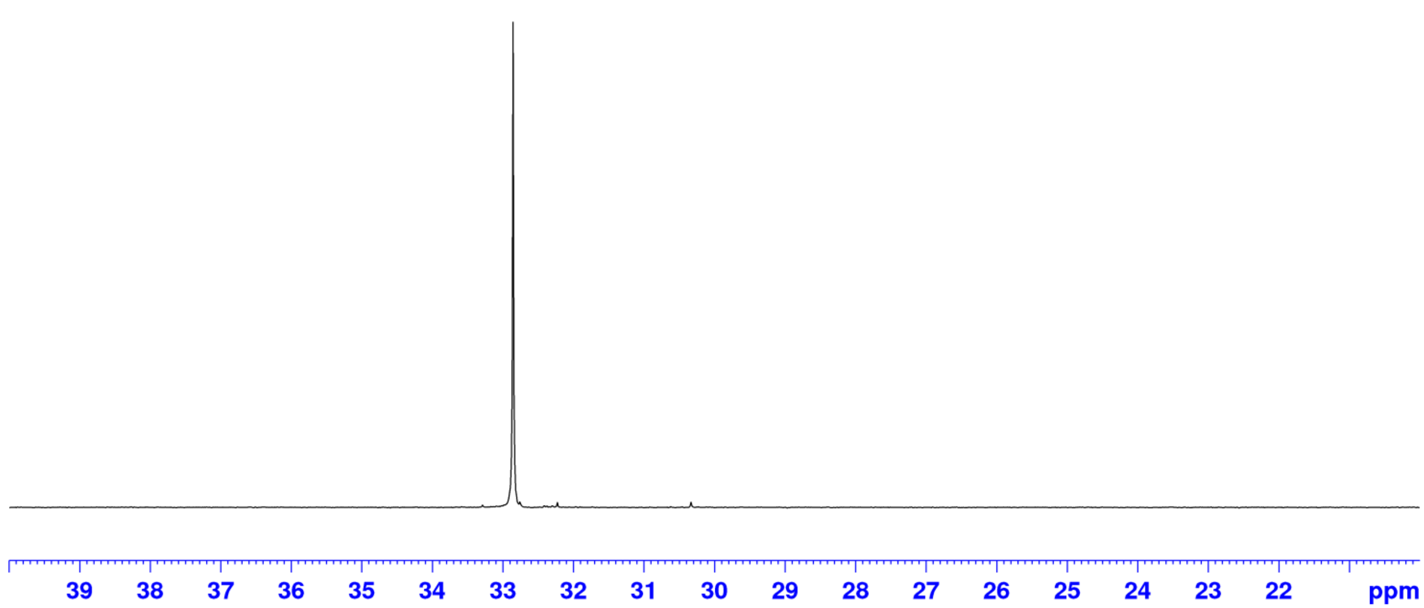
9k: ^{1}H NMR



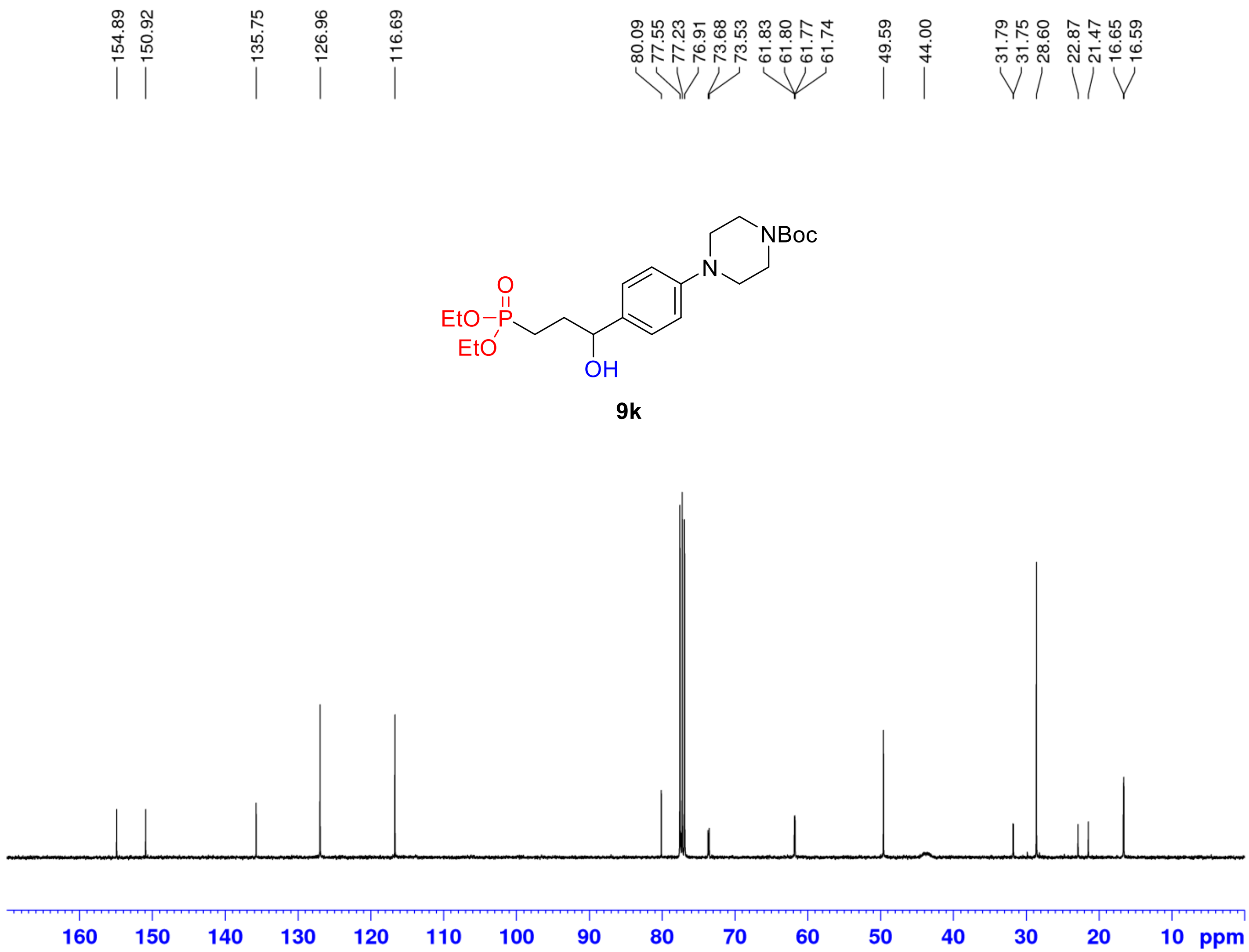
9k: ^{31}P NMR



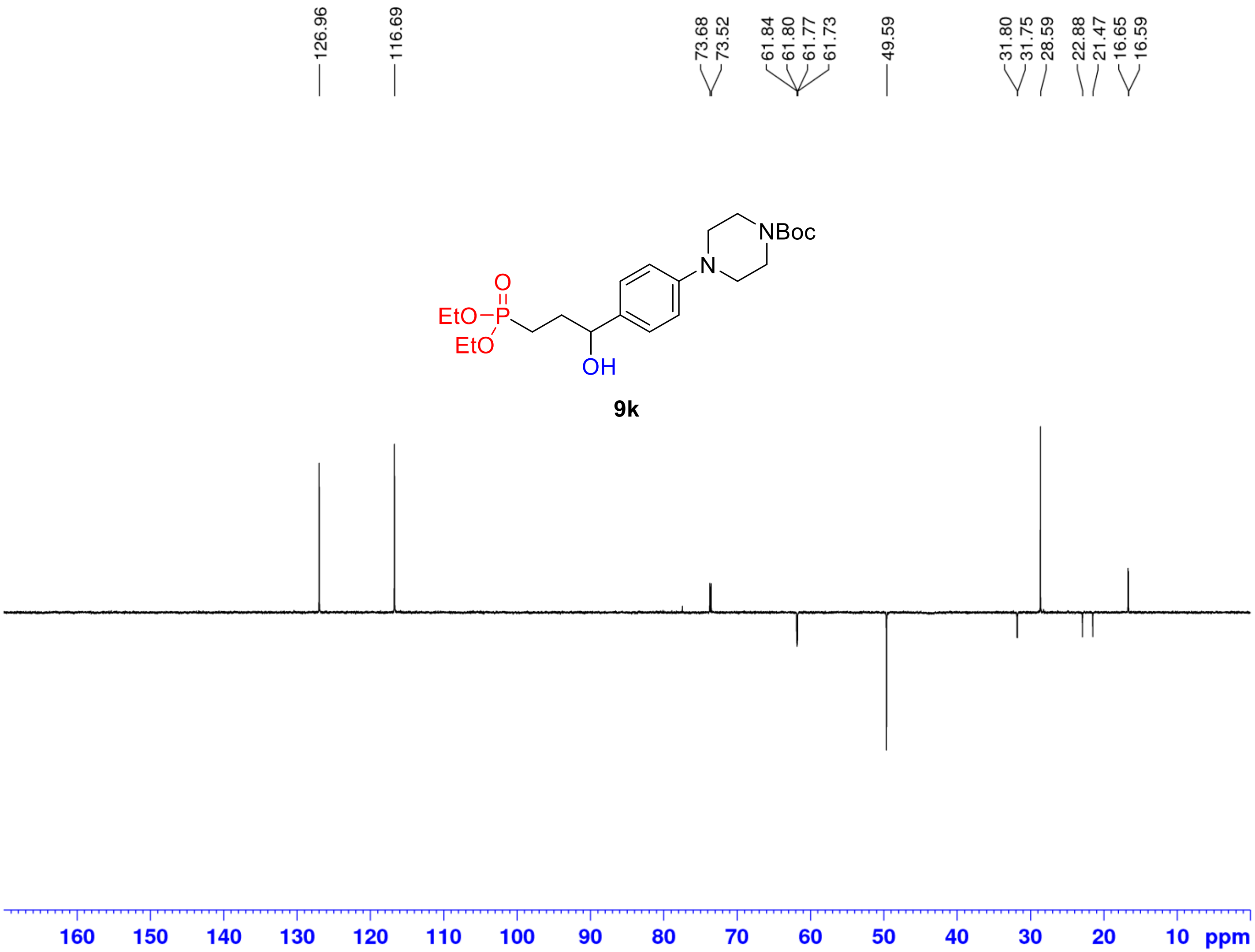
9k



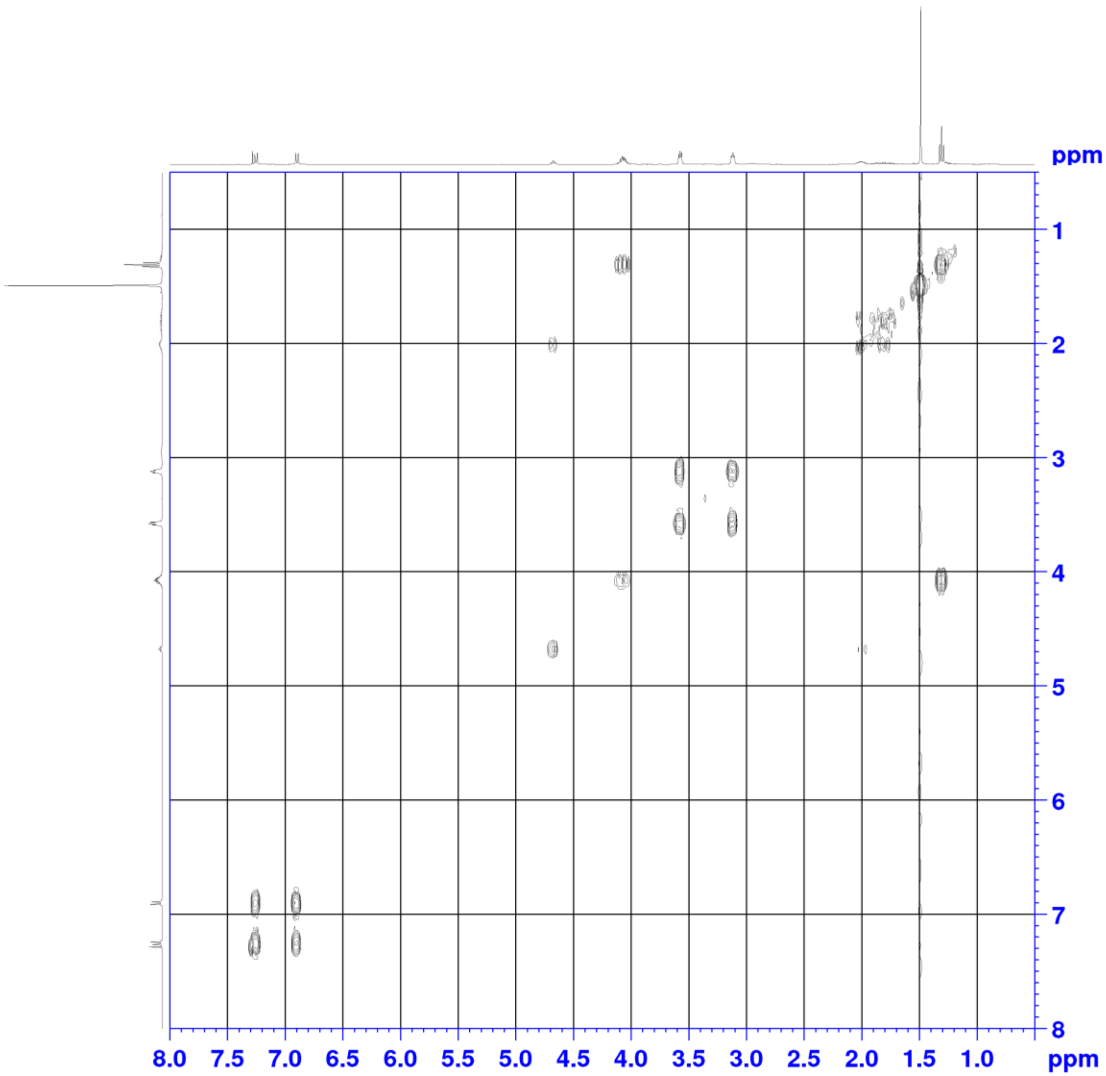
9k: ^{13}C NMR


154.89
150.92
135.75
126.96
116.69
80.09
77.55
77.23
76.91
73.68
73.53
61.83
61.80
61.77
61.74
49.59
44.00
31.79
31.75
28.60
22.87
21.47
16.65
16.59
NBoc
N
O
EtO
P
EtO
OH
9k
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

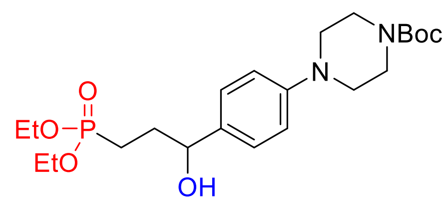
9k: ^{13}C DEPT135



9k: COSY



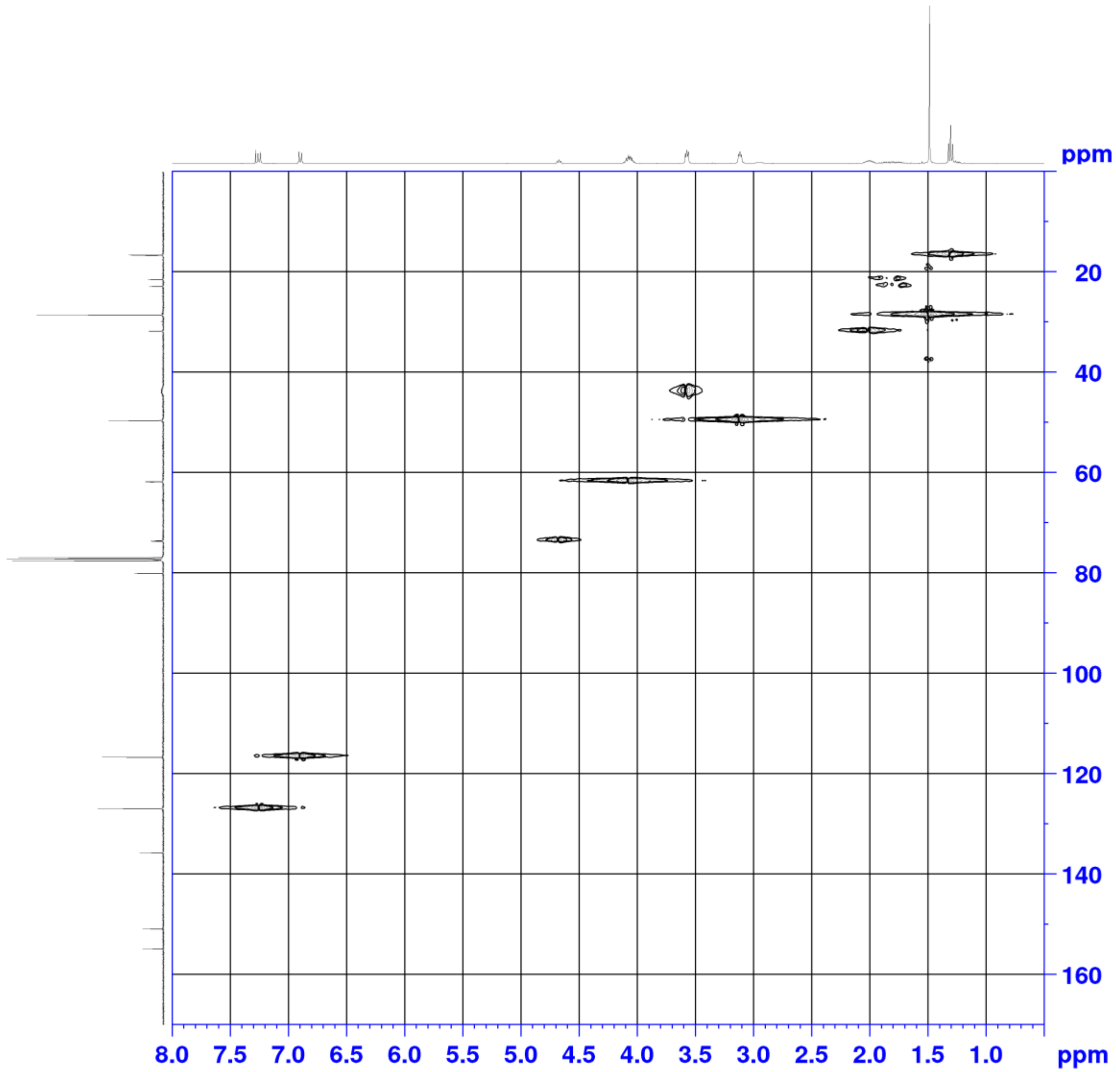
ppm
1
2
3
4
5
6
7
8
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

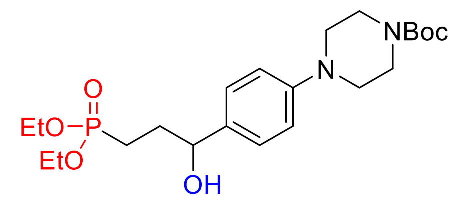


NBoc
N
O
EtO
P
EtO
OH

9k

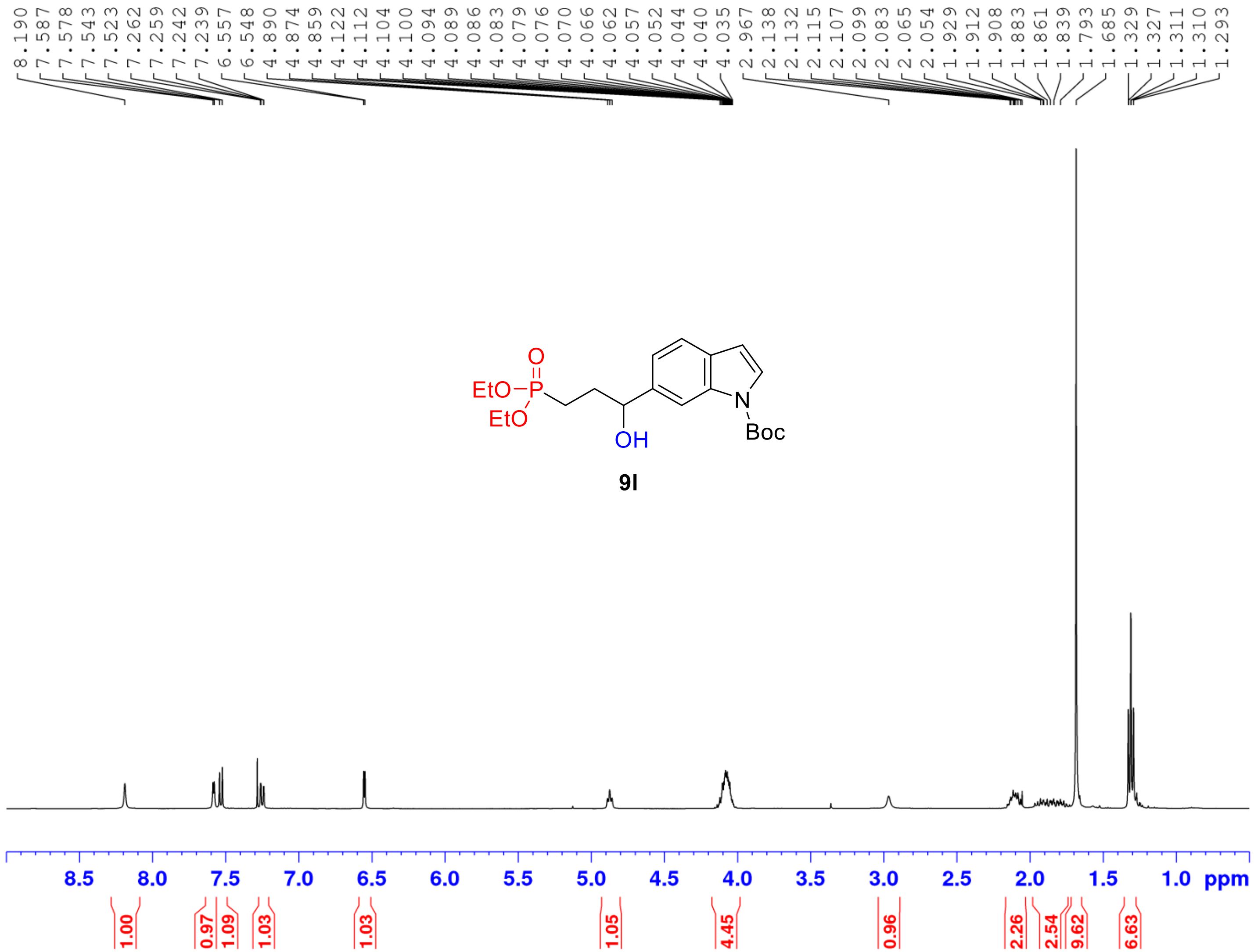
9k: ^{13}C HSQC





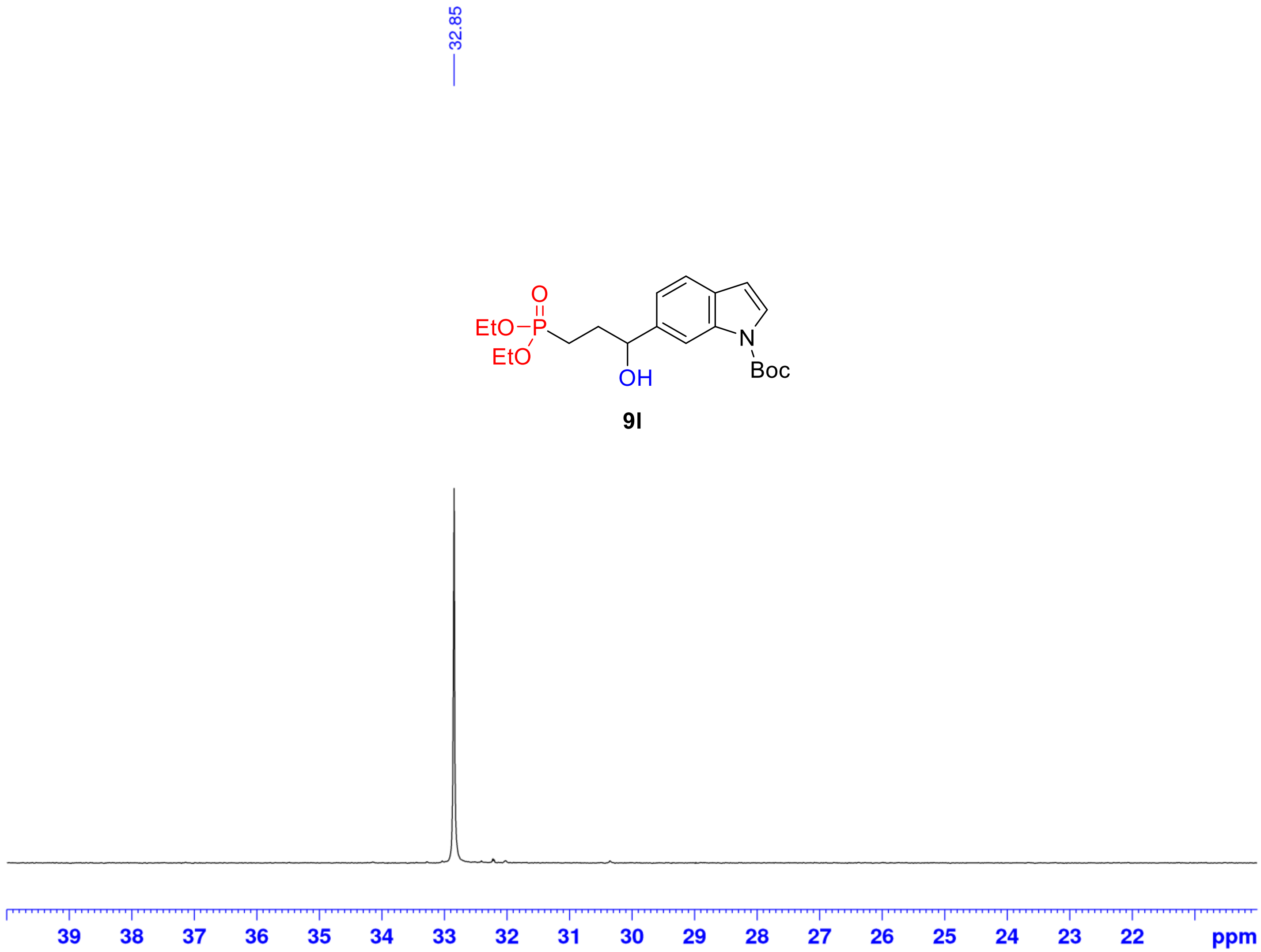
9k

9l: ^{1}H NMR



8.190
7.587
7.578
7.543
7.523
7.262
7.259
7.242
7.239
6.557
6.548
4.890
4.874
4.859
4.122
4.112
4.104
4.100
4.094
4.089
4.086
4.083
4.079
4.076
4.070
4.066
4.062
4.057
4.052
4.044
4.040
4.035
2.967
2.138
2.132
2.115
2.107
2.099
2.083
2.065
2.054
1.929
1.912
1.908
1.883
1.861
1.839
1.793
1.685
1.329
1.327
1.311
1.310
1.293
EtO
EtO
P
O
OH
N
Boc
9l
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
ppm
1.00
0.97
1.09
1.03
1.03
1.05
4.45
0.96
2.26
2.54
9.62
6.63

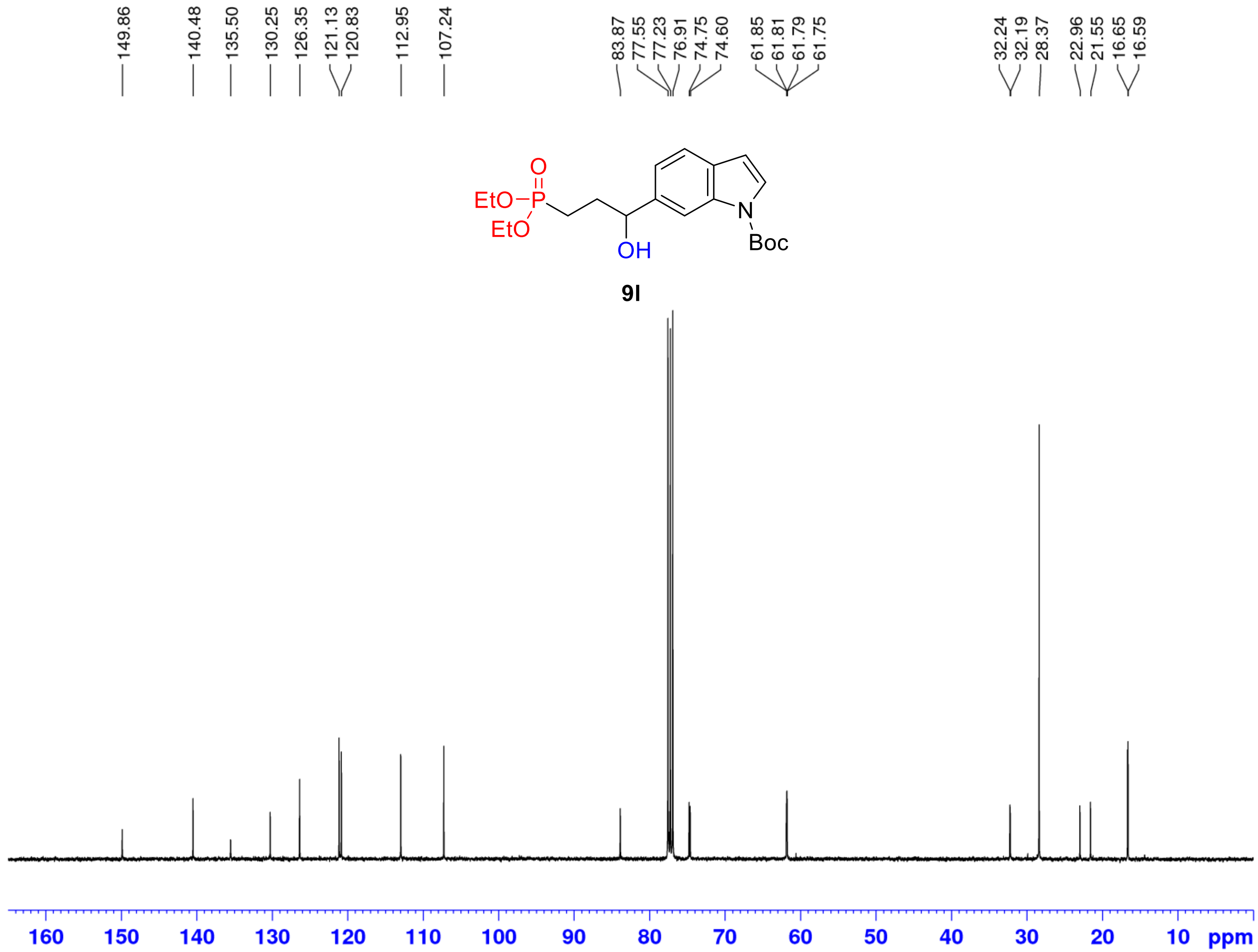
9l: ^{31}P NMR



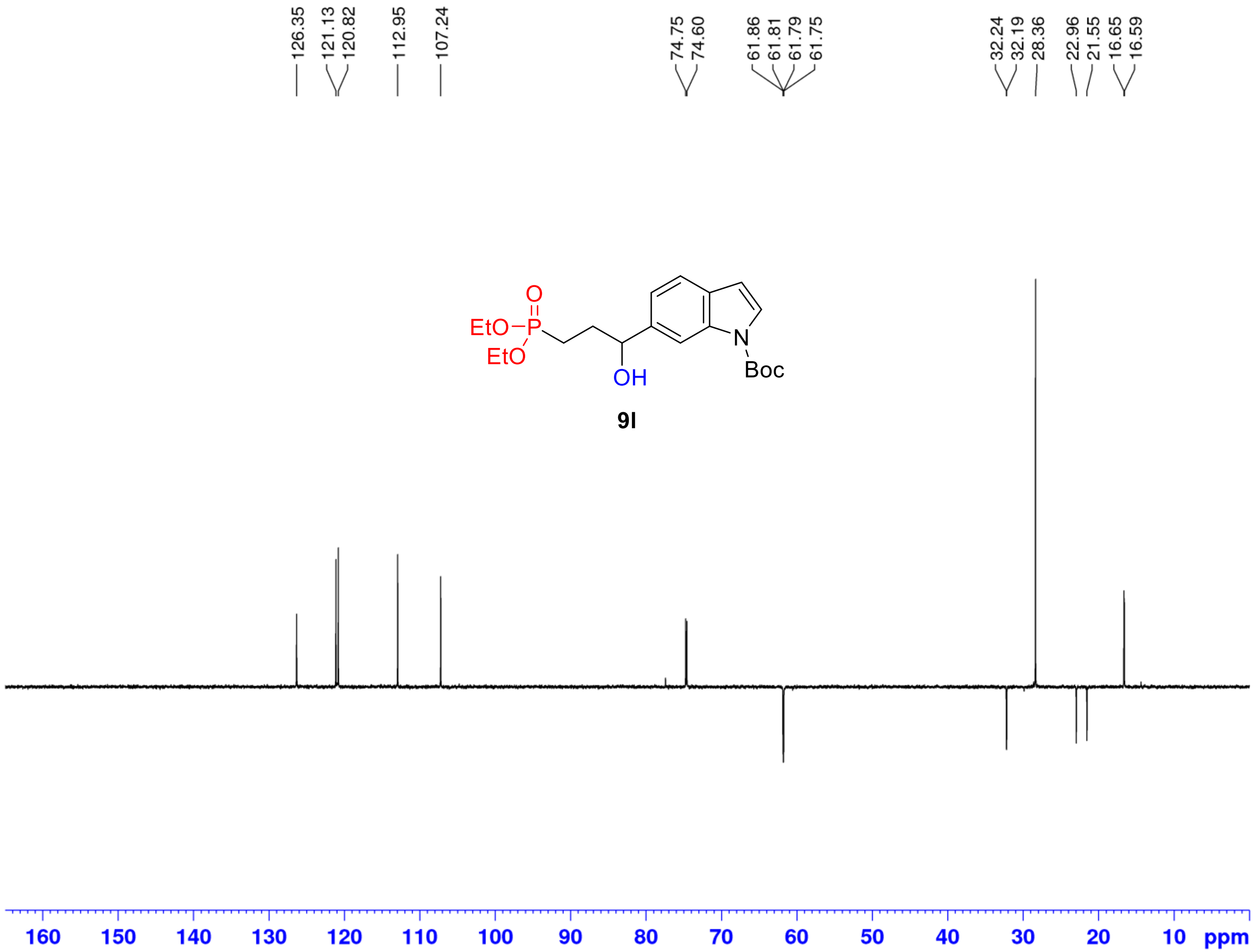
32.85
EtO
O
P
EtO
OH
N
Boc
39
38
37
36
35
34
33
32
31
30
29
28
27
26
25
24
23
22
ppm

9l

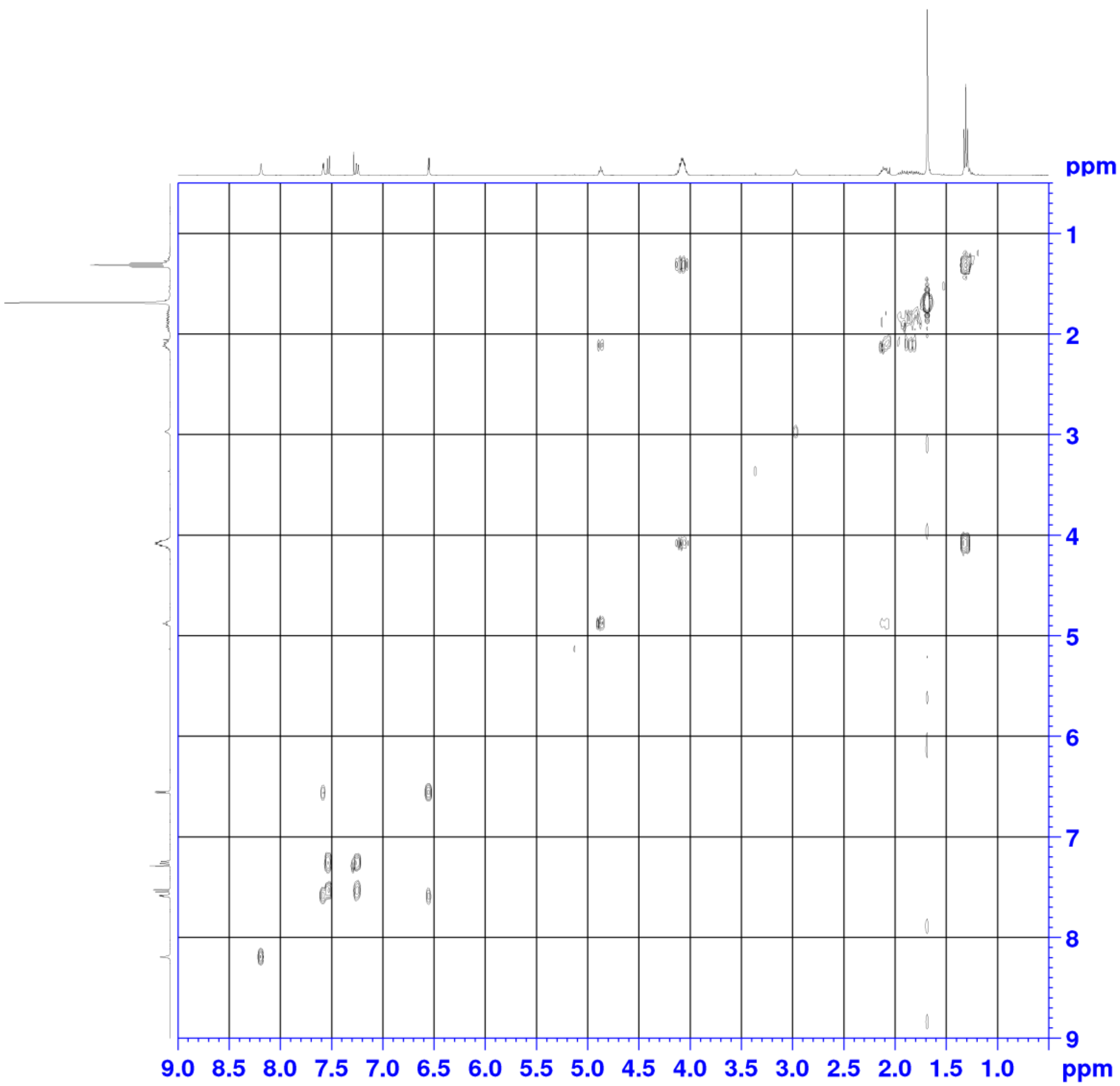
9l: ^{13}C NMR

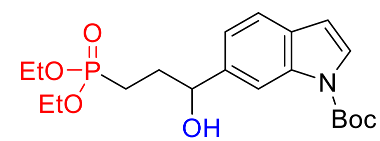

149.86
140.48
135.50
130.25
126.35
121.13
120.83
112.95
107.24
83.87
77.55
77.23
76.91
74.75
74.60
61.85
61.81
61.79
61.75
32.24
32.19
28.37
22.96
21.55
16.65
16.59
O
EtO
P
EtO
OH
N
Boc
9l
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

9l: ^{13}C DEPT135



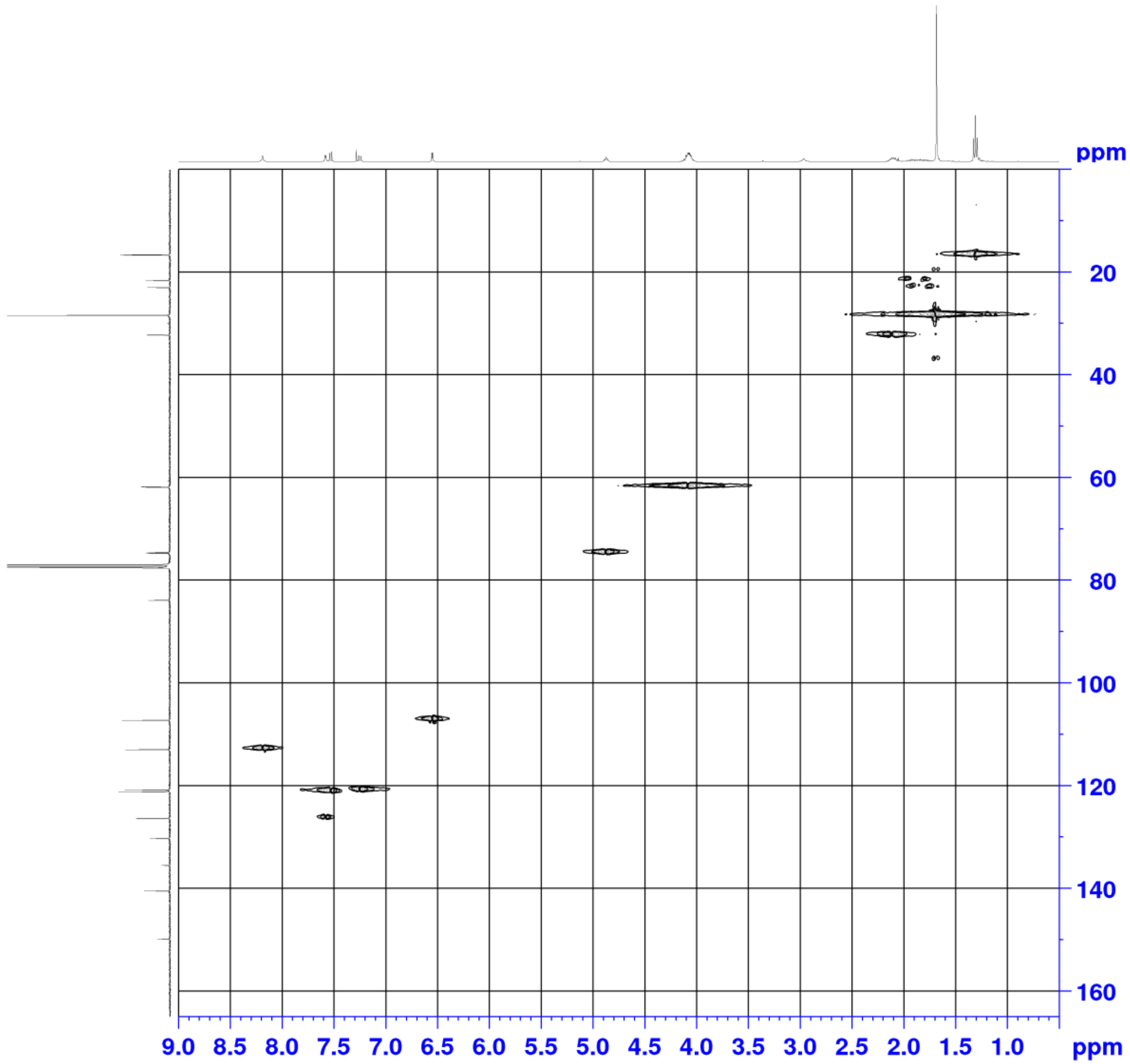
9l: COSY

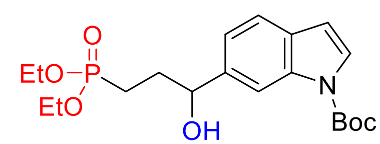

ppm
1
2
3
4
5
6
7
8
9
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0
ppm


O
EtO
P
EtO
OH
N
Boc

9l

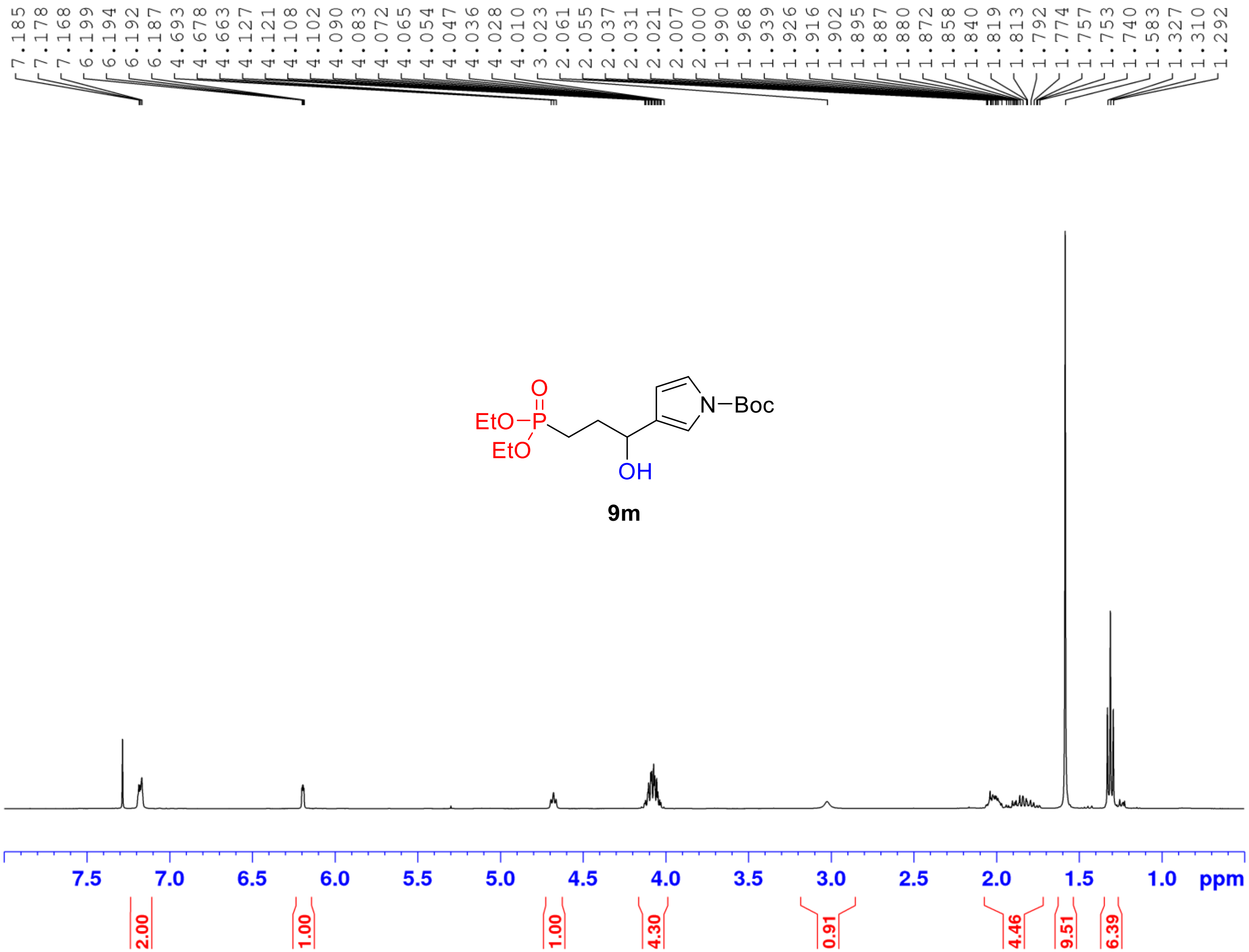
9l: ^{13}C HSQC





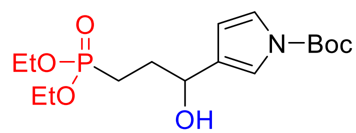
9l

9m: ^{1}H NMR



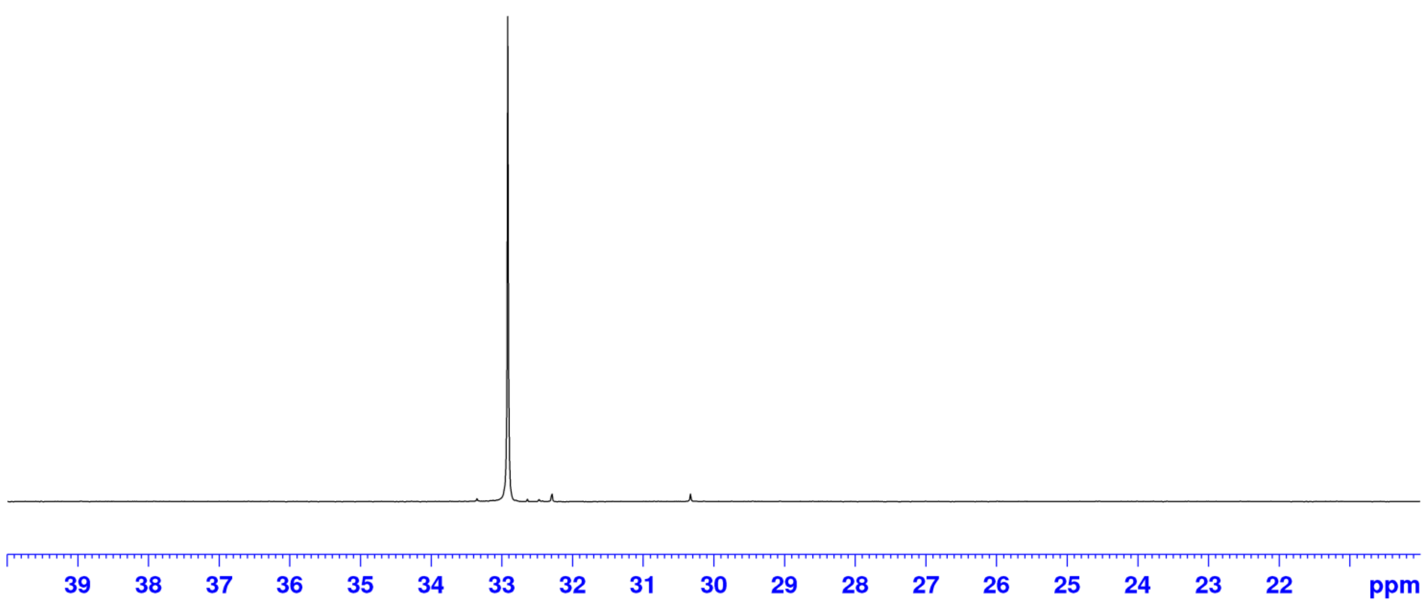
9m

9m: ^{31}P NMR



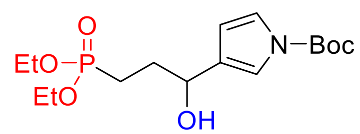
EtO
EtO
O
P
OH
N
Boc

9m

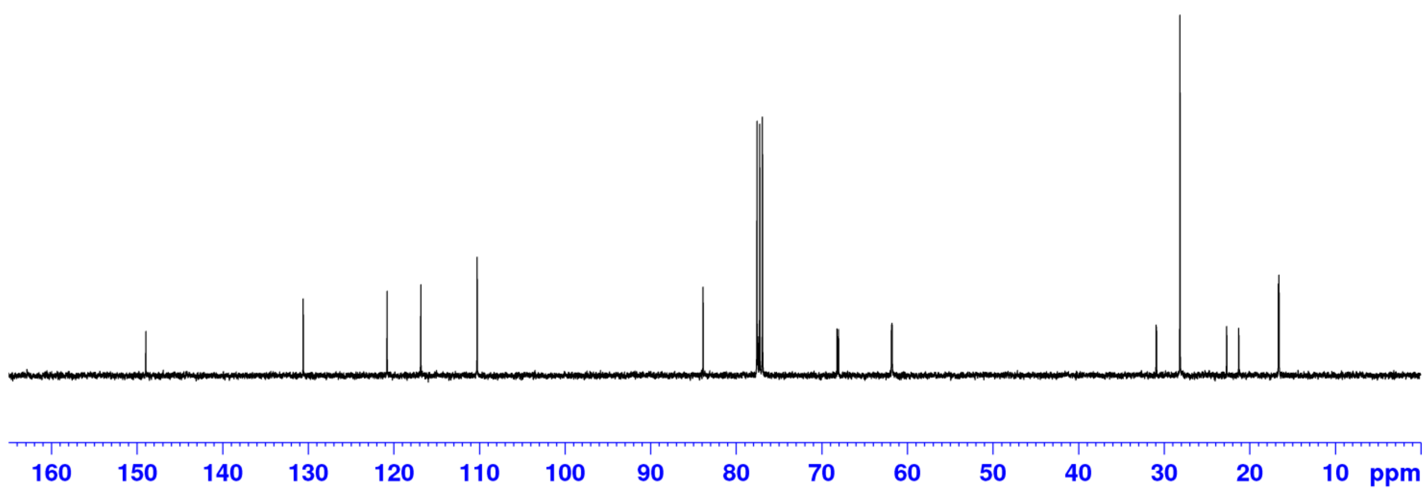


32.92
39
38
37
36
35
34
33
32
31
30
29
28
27
26
25
24
23
22
ppm

9m: ^{13}C NMR

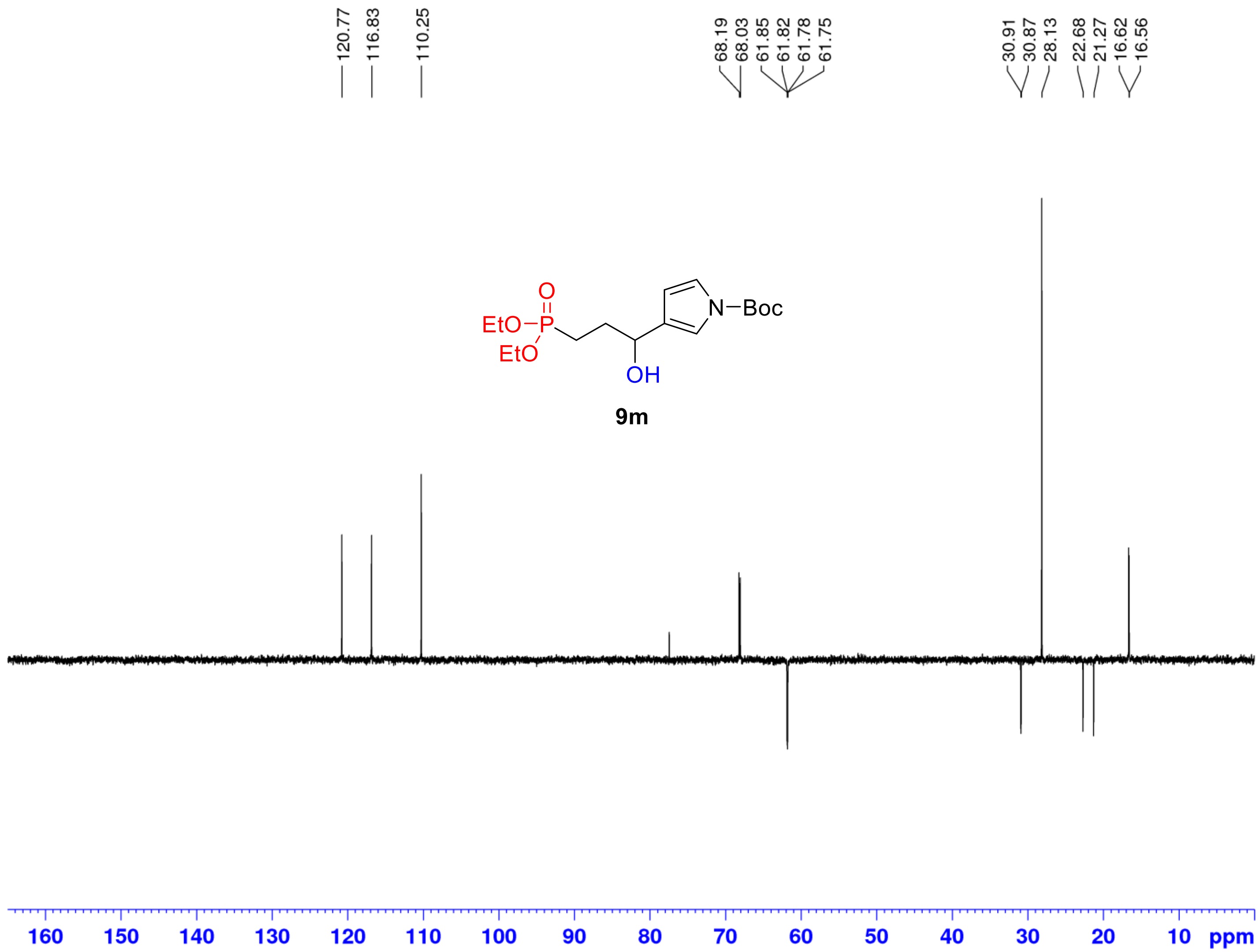


9m

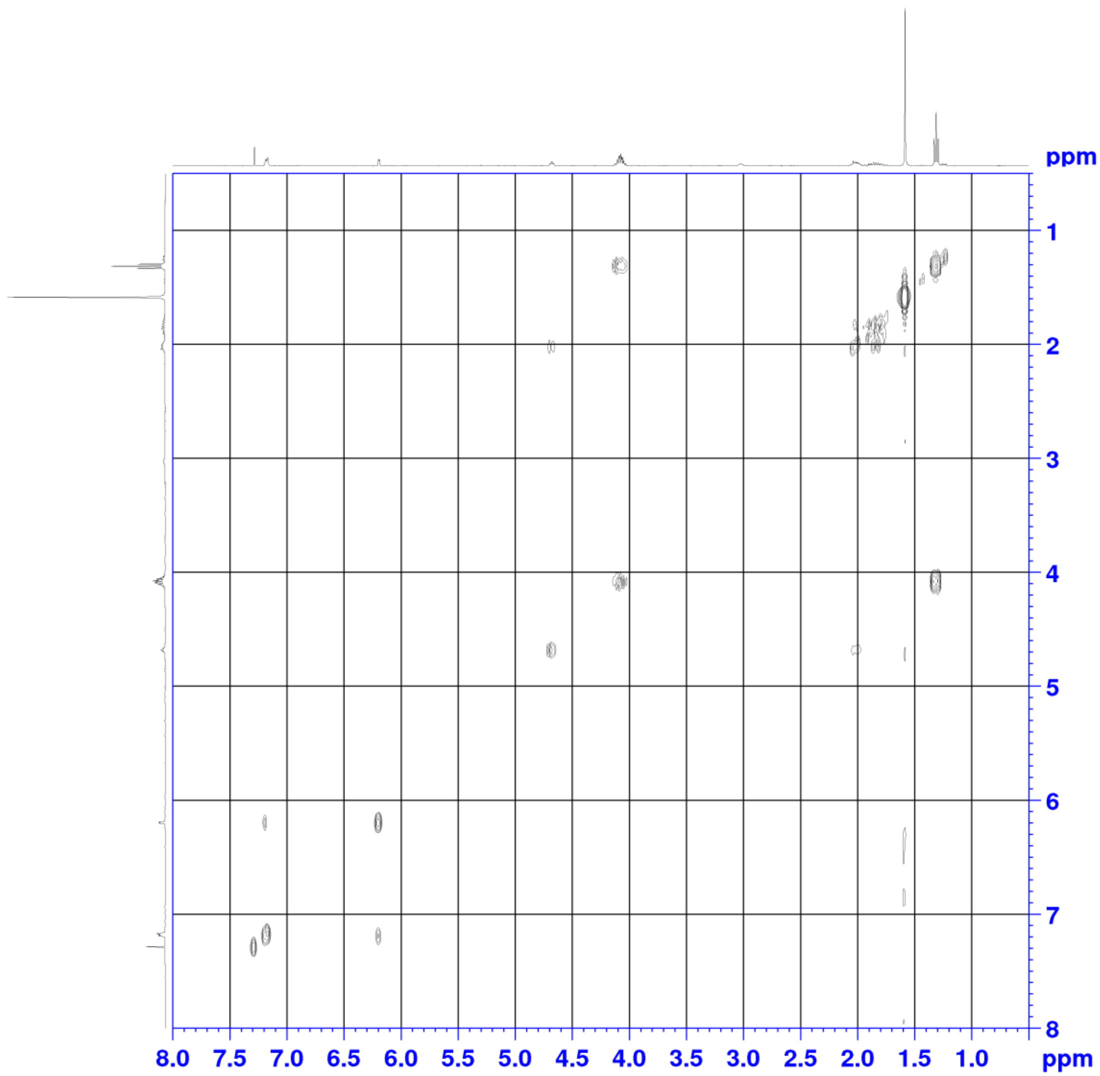


148.96
130.58
120.77
116.83
110.25
83.85
77.55
77.23
76.91
68.19
68.03
61.84
61.81
61.78
61.75
30.91
30.87
28.13
22.67
21.26
16.62
16.56
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

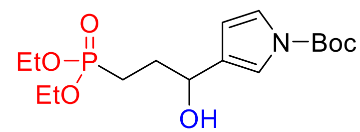
9m: ^{13}C DEPT135



9m: COSY



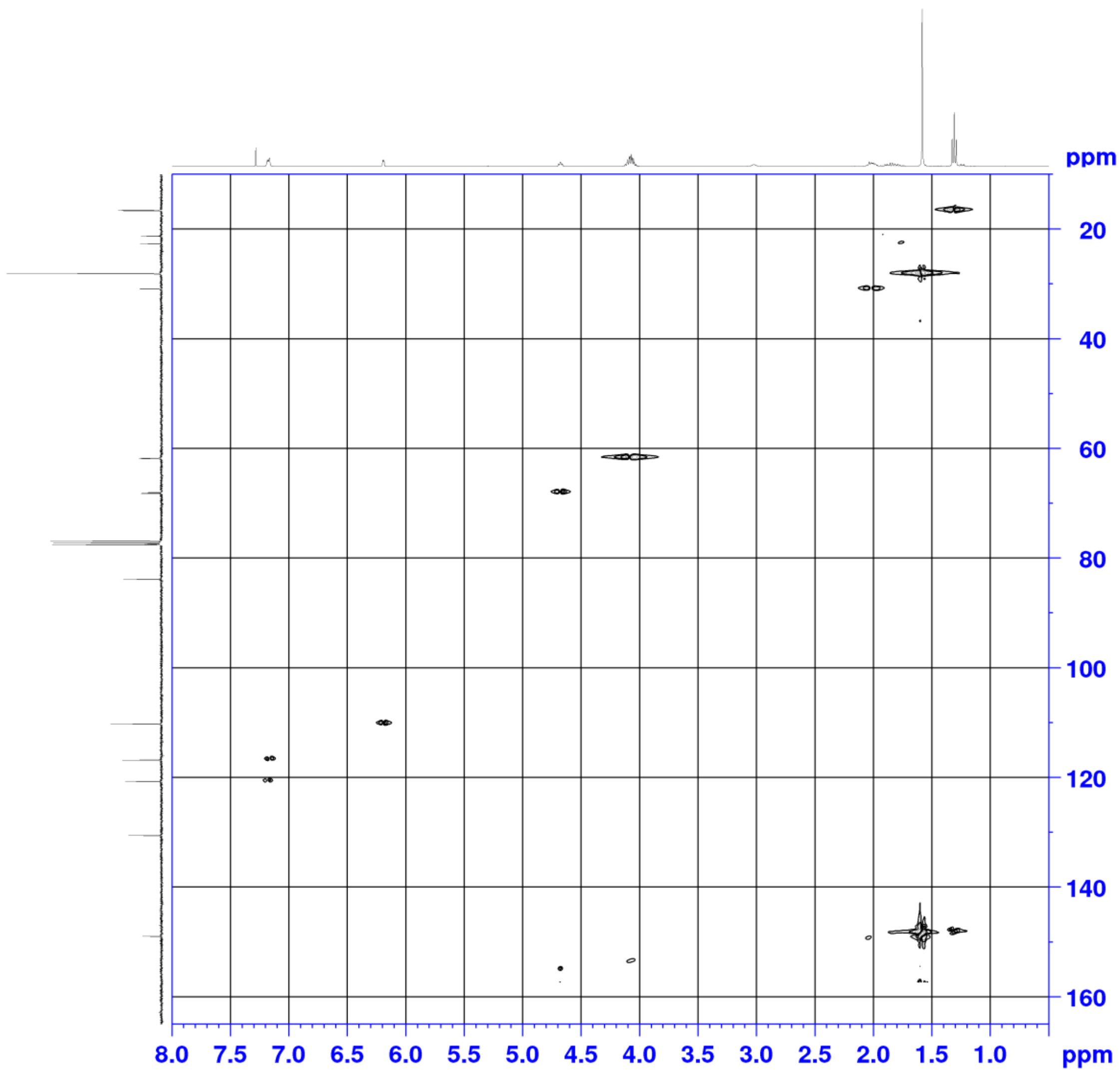
ppm
1
2
3
4
5
6
7
8
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

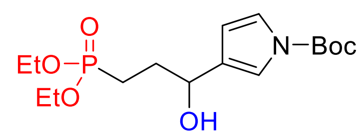


O
EtO
P
EtO
OH
N
Boc

9m

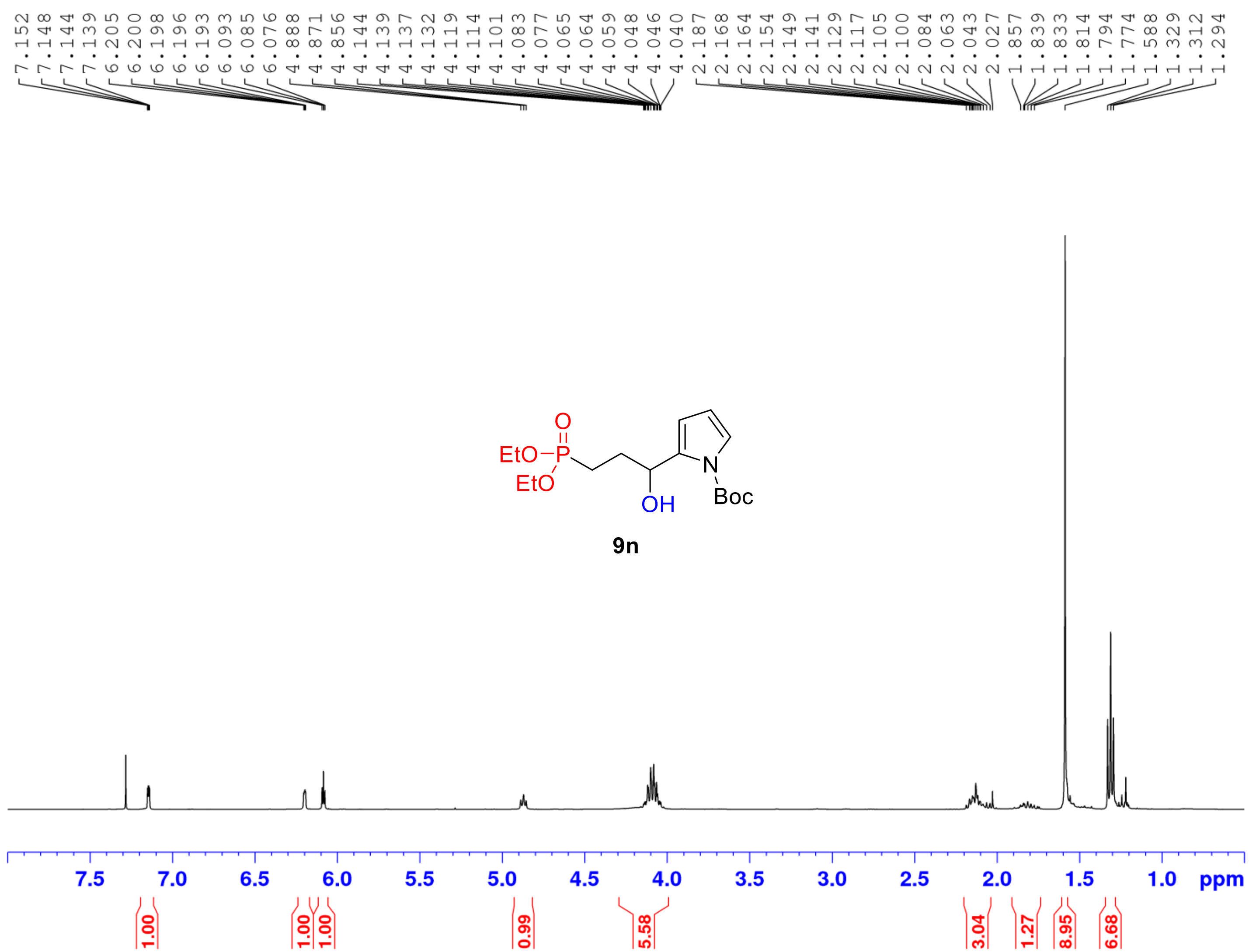
9m: ^{13}C HSQC



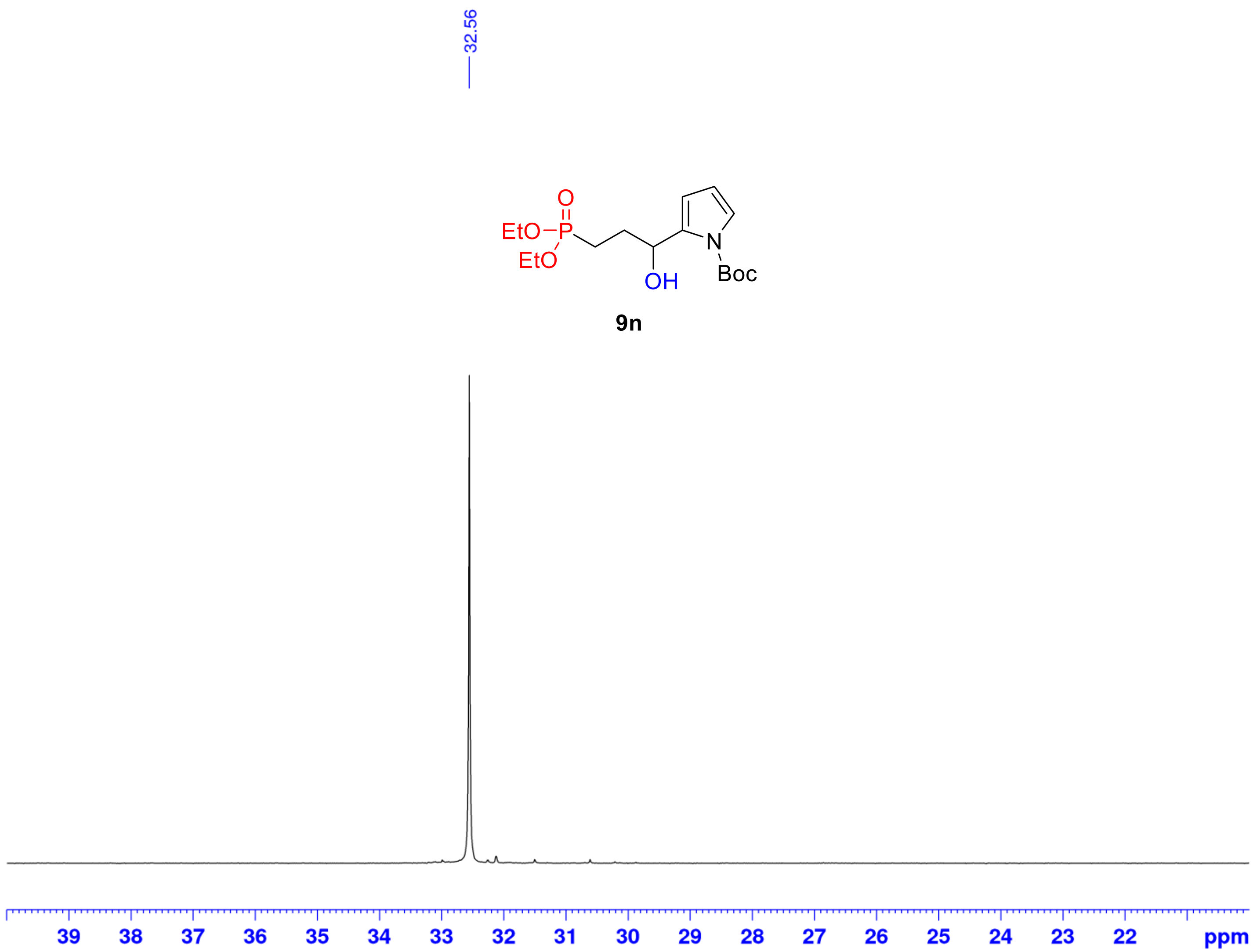


9m

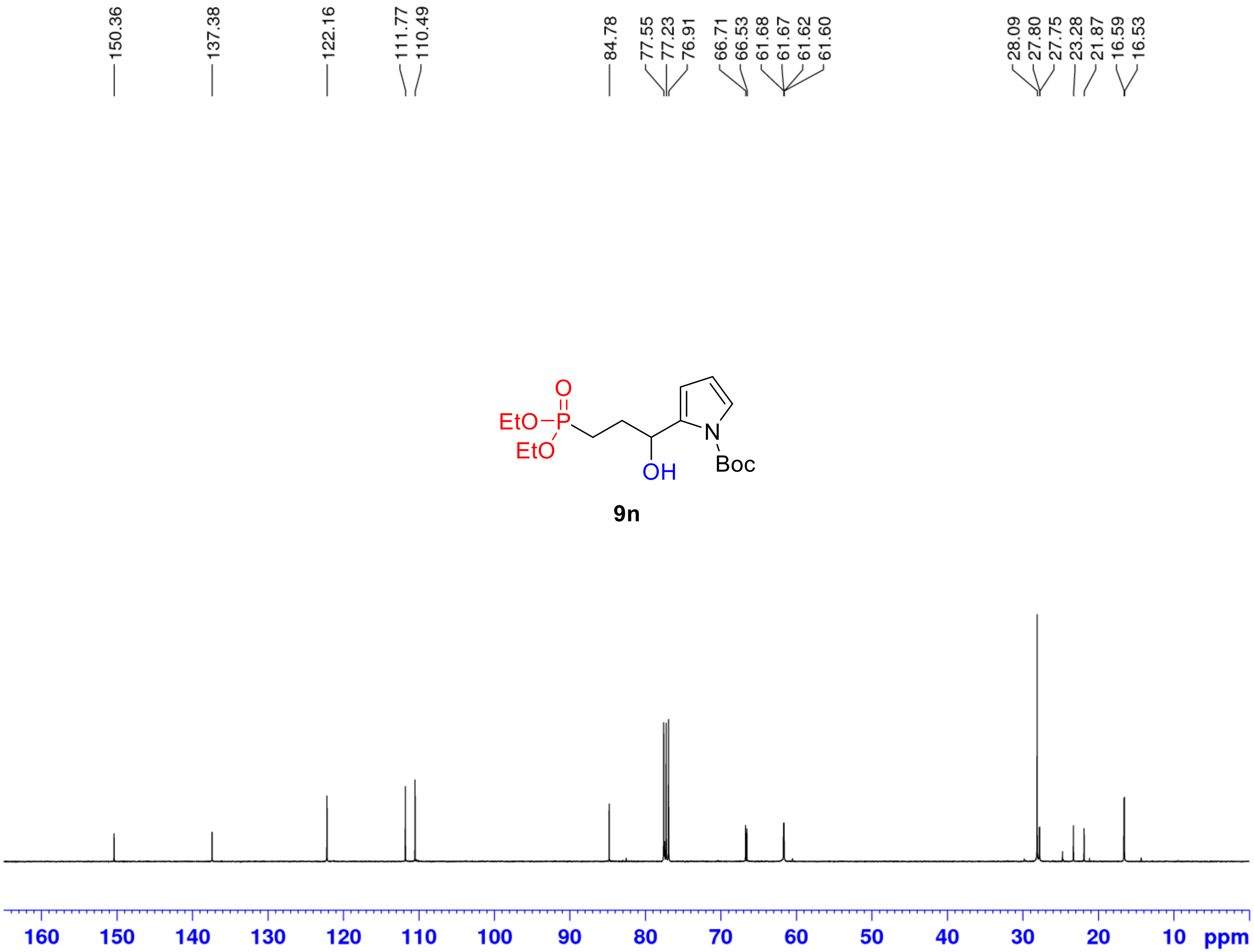
9n: ^{1}H NMR


7.152
7.148
7.144
7.139
6.205
6.200
6.198
6.196
6.193
6.093
6.085
6.076
4.888
4.871
4.856
4.144
4.139
4.137
4.132
4.119
4.114
4.101
4.083
4.077
4.065
4.064
4.059
4.048
4.046
4.040
2.187
2.168
2.164
2.154
2.149
2.141
2.129
2.117
2.105
2.100
2.084
2.063
2.043
2.027
1.857
1.839
1.833
1.814
1.794
1.774
1.588
1.329
1.312
1.294
EtO
P
O
EtO
OH
N
Boc
9n
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
1.00
1.00
1.00
0.99
5.58
3.04
1.27
8.95
6.68

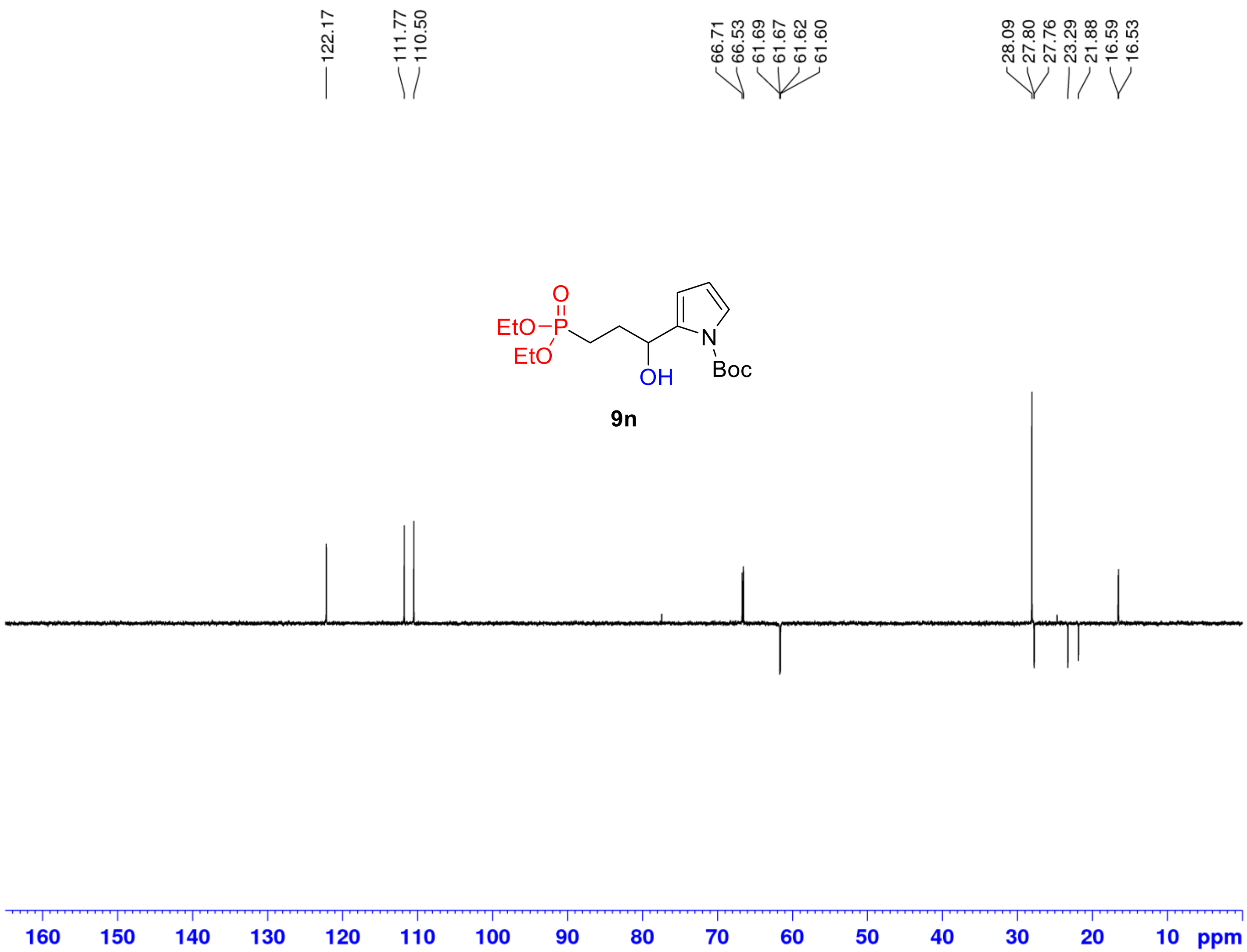
9n: ^{31}P NMR


32.56
EtO
EtO
O
P
N
Boc
OH
9n
39
38
37
36
35
34
33
32
31
30
29
28
27
26
25
24
23
22
ppm

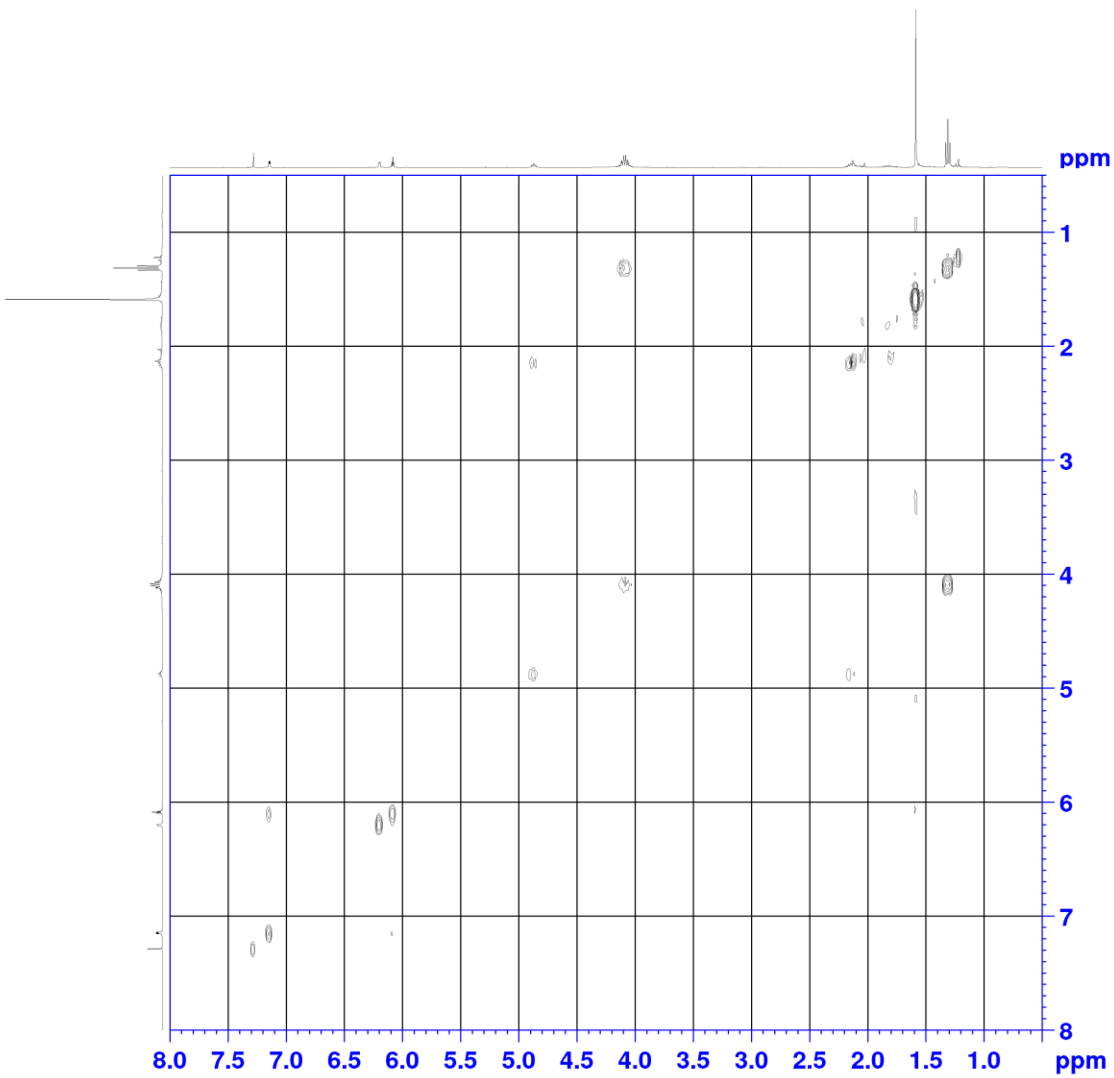
9n: ^{13}C NMR


150.36
137.38
122.16
111.77
110.49
84.78
77.55
77.23
76.91
66.71
66.53
61.68
61.67
61.62
61.60
28.09
27.80
27.75
23.28
21.87
16.59
16.53
EtO
EtO
P
O
OH
N
Boc
9n
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

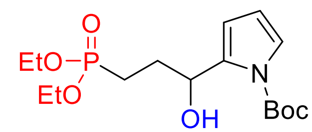
9n: ^{13}C DEPT135



9n: COSY



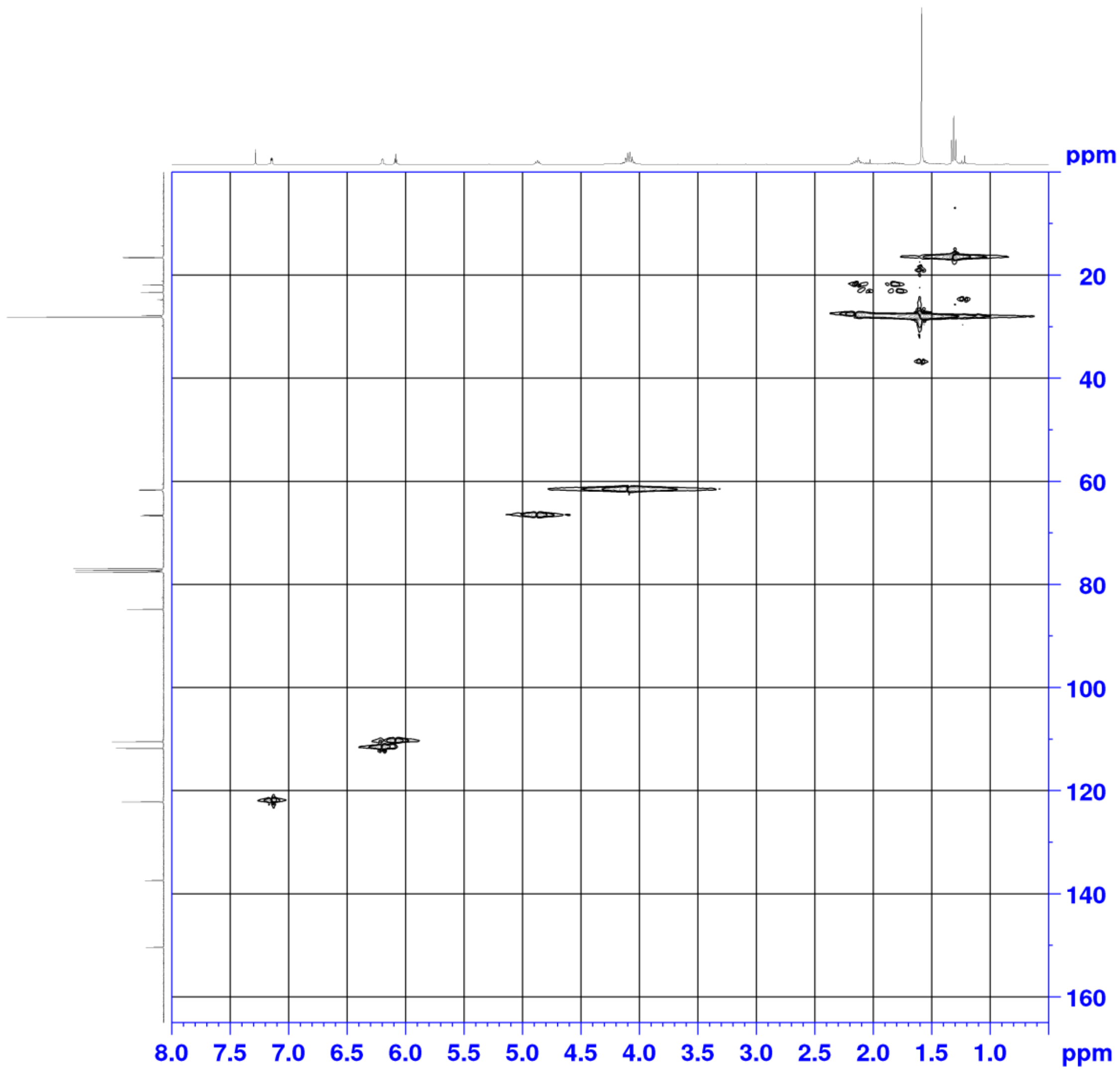
ppm
1
2
3
4
5
6
7
8
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

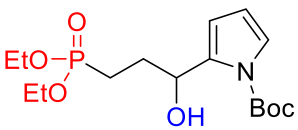


O
EtO
P
EtO
OH
N
Boc

9n

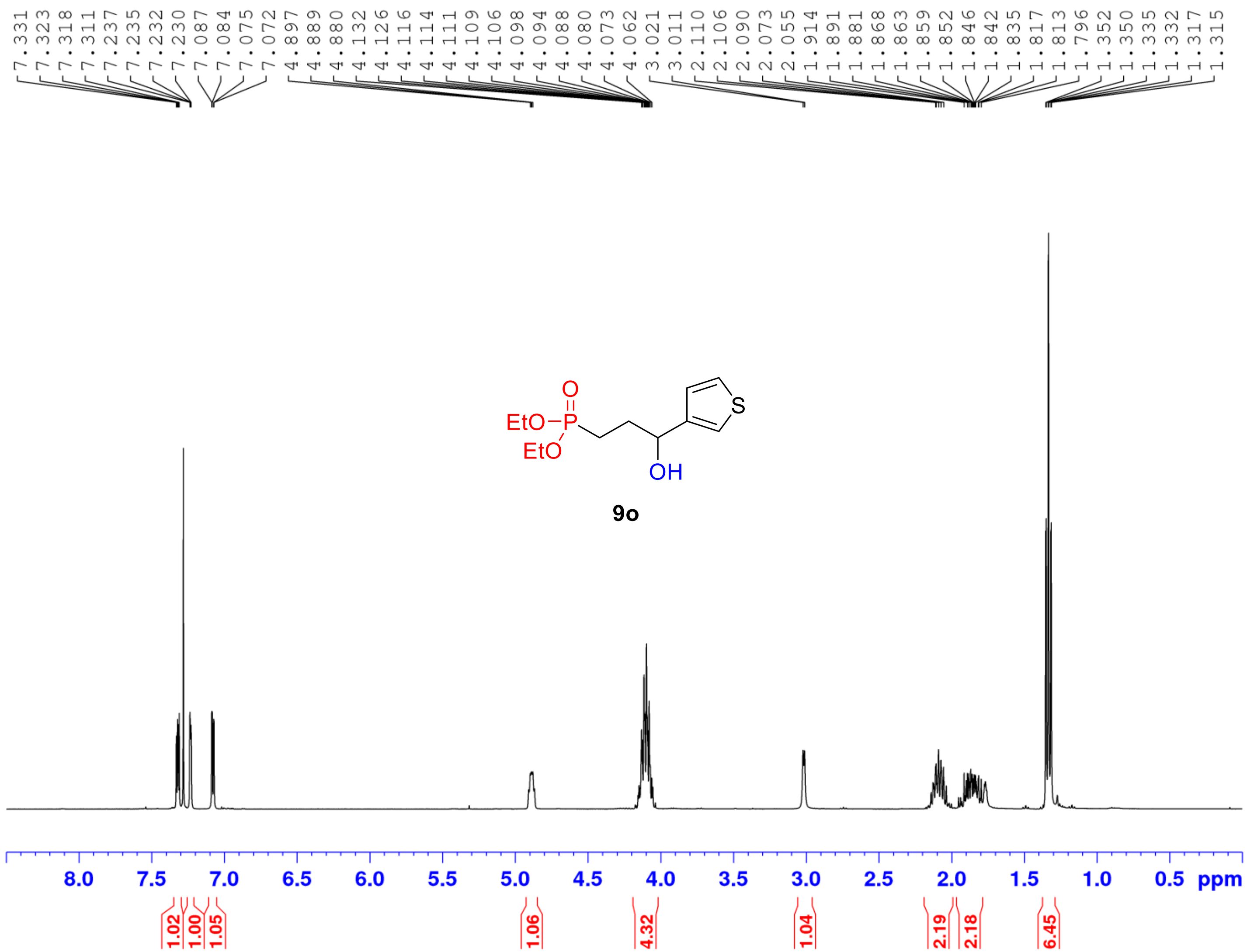
9n: ^{13}C HSQC





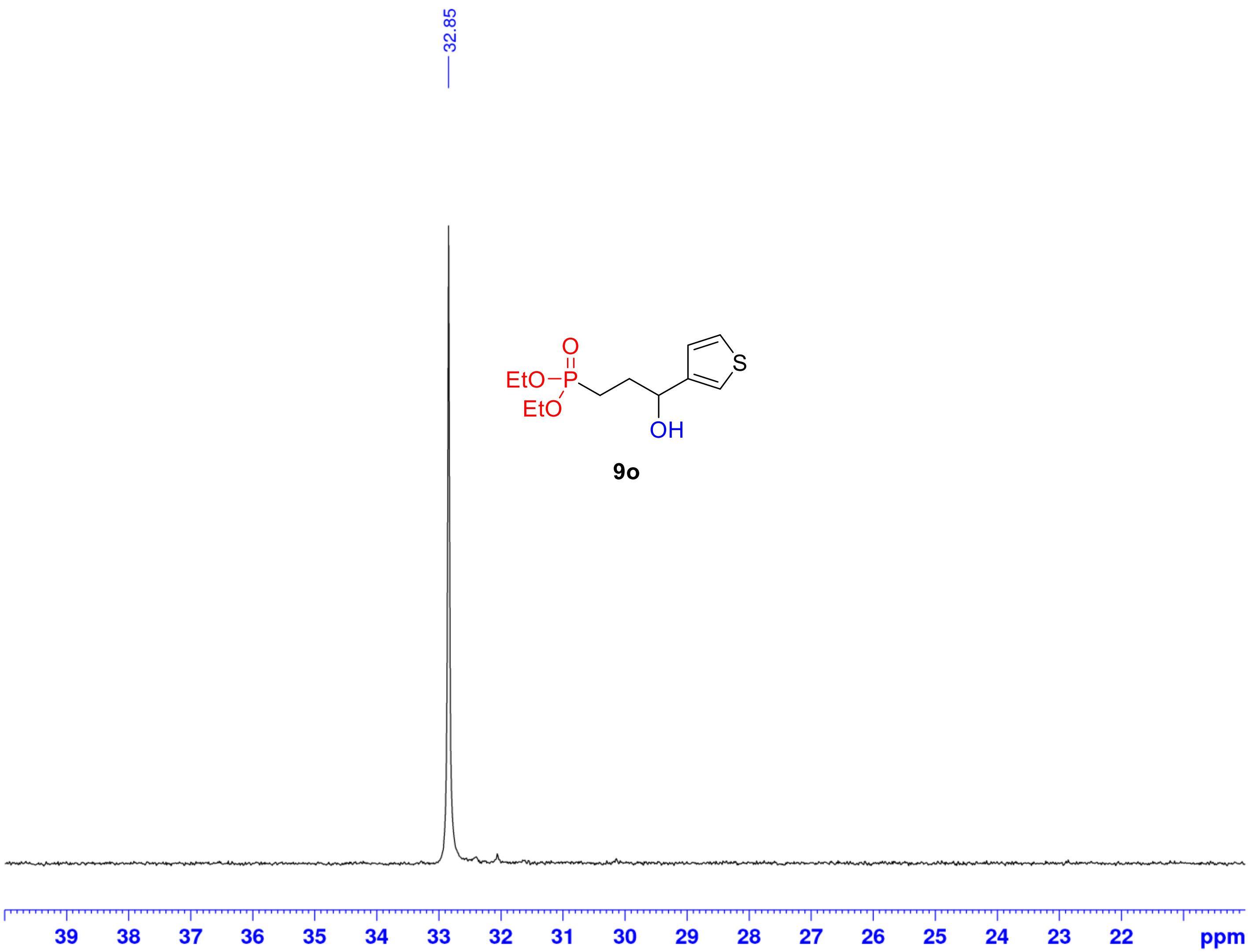
9n

9o: ^{1}H NMR

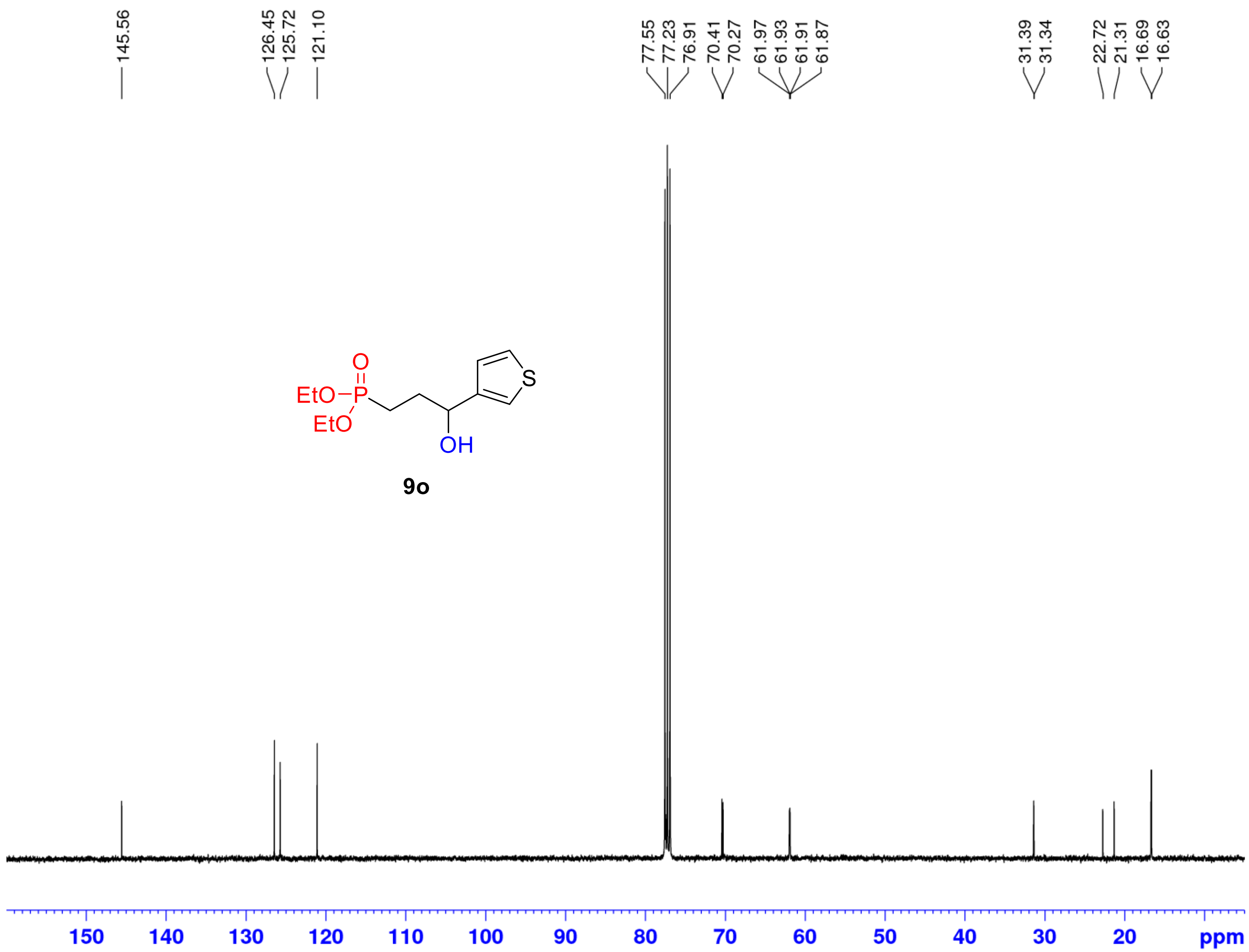


7.331
7.323
7.318
7.311
7.237
7.235
7.232
7.230
7.087
7.084
7.075
7.072
4.897
4.889
4.880
4.132
4.126
4.116
4.114
4.111
4.109
4.106
4.098
4.094
4.088
4.080
4.073
4.062
3.021
3.011
2.110
2.106
2.090
2.073
2.055
1.914
1.891
1.881
1.868
1.863
1.859
1.852
1.846
1.842
1.835
1.817
1.813
1.796
1.352
1.350
1.335
1.332
1.317
1.315
O
EtO
P
EtO
S
OH
9o
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
ppm
1.02
1.00
1.05
1.06
4.32
1.04
2.19
2.18
6.45

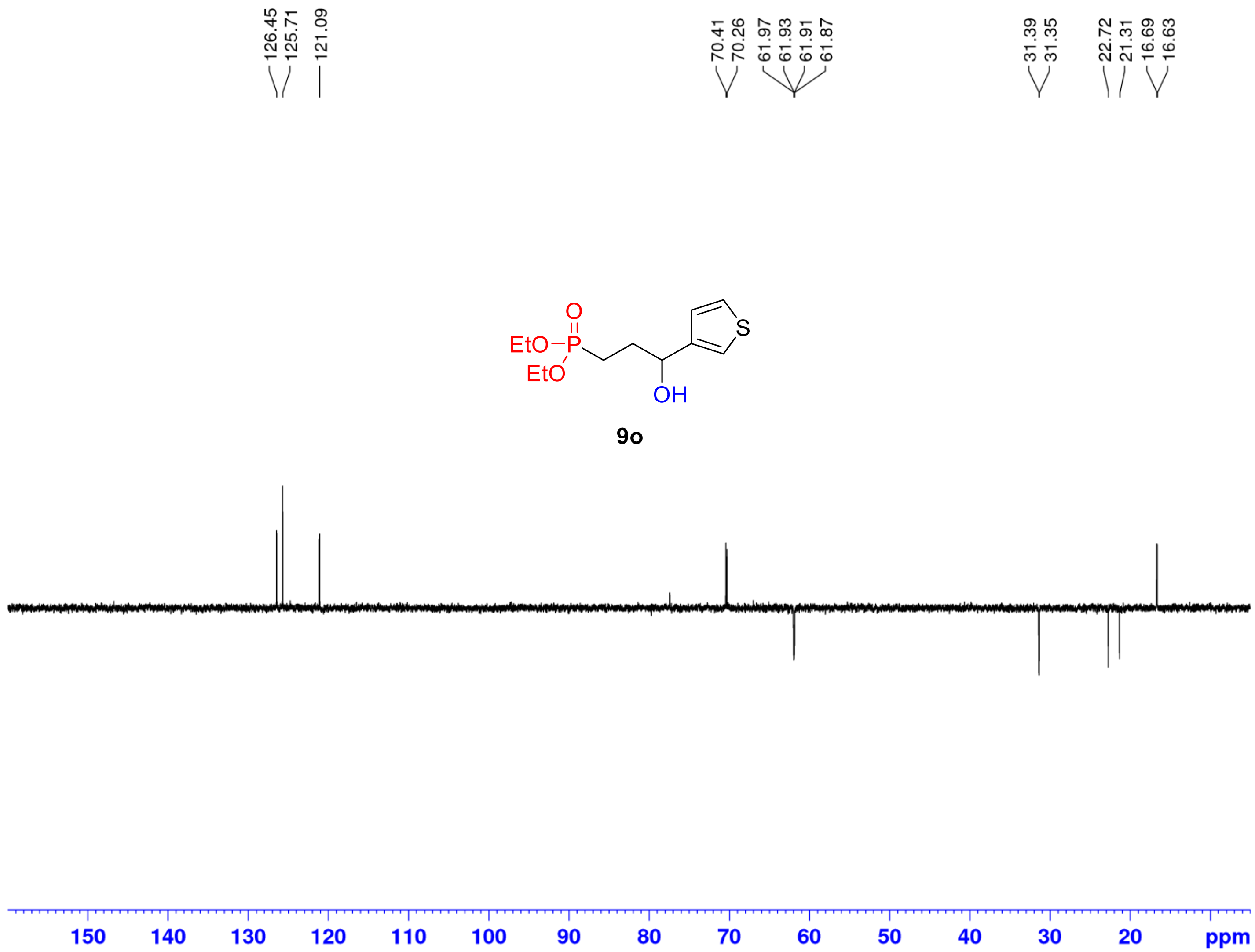
9o: ^{31}P NMR


32.85
O
EtO
P
EtO
S
OH
9o
39
38
37
36
35
34
33
32
31
30
29
28
27
26
25
24
23
22
ppm

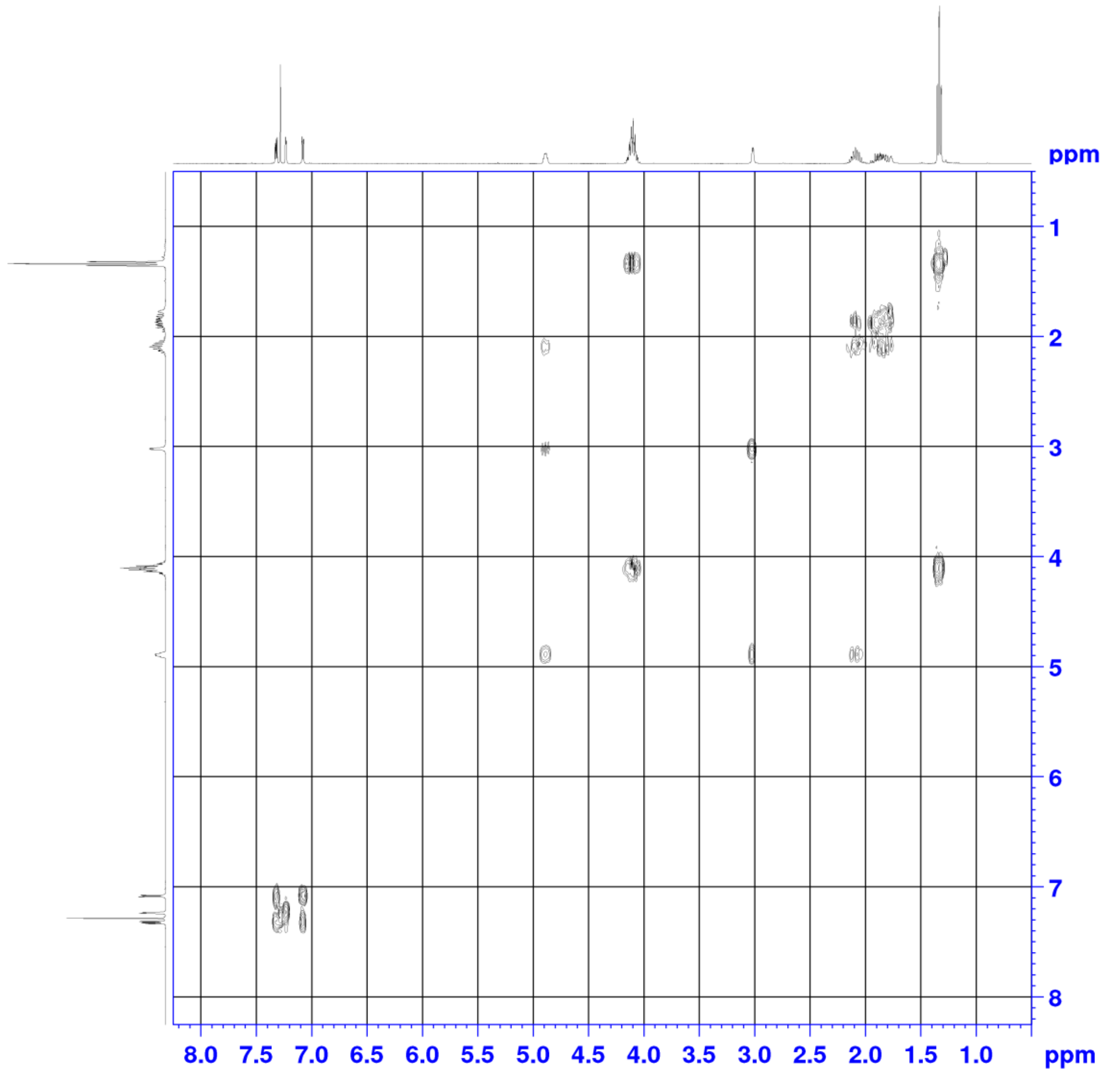
9o: ^{13}C NMR


145.56
126.45
125.72
121.10
77.55
77.23
76.91
70.41
70.27
61.97
61.93
61.91
61.87
31.39
31.34
22.72
21.31
16.69
16.63
O
EtO
P
EtO
S
OH
9o
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

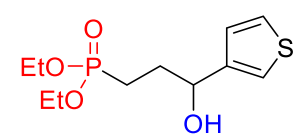
9o: ^{13}C DEPT135



9o: COSY



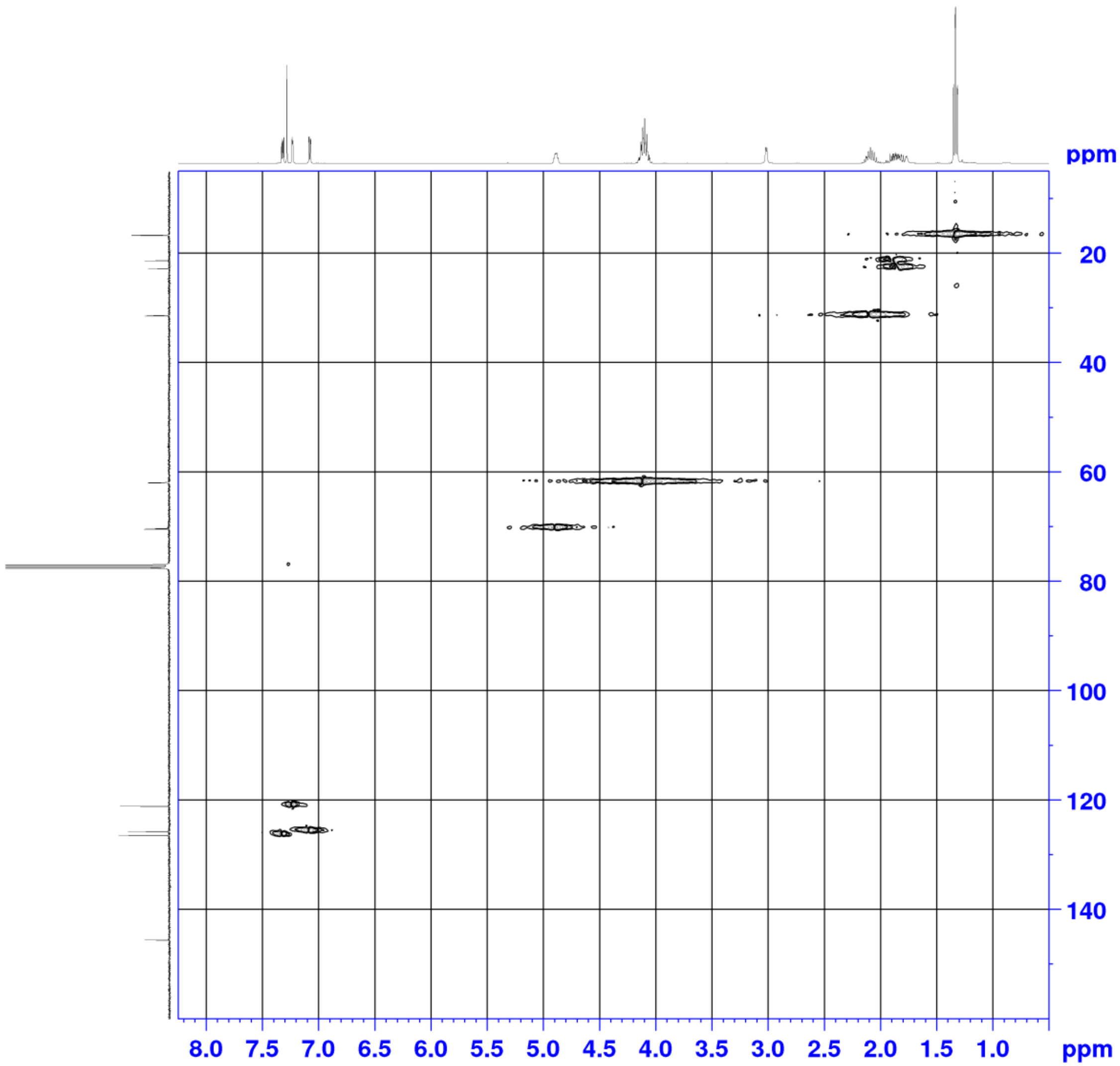
ppm
1
2
3
4
5
6
7
8
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

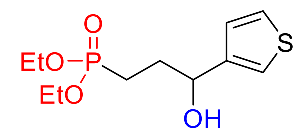


O
EtO
P
EtO
S
OH

9o

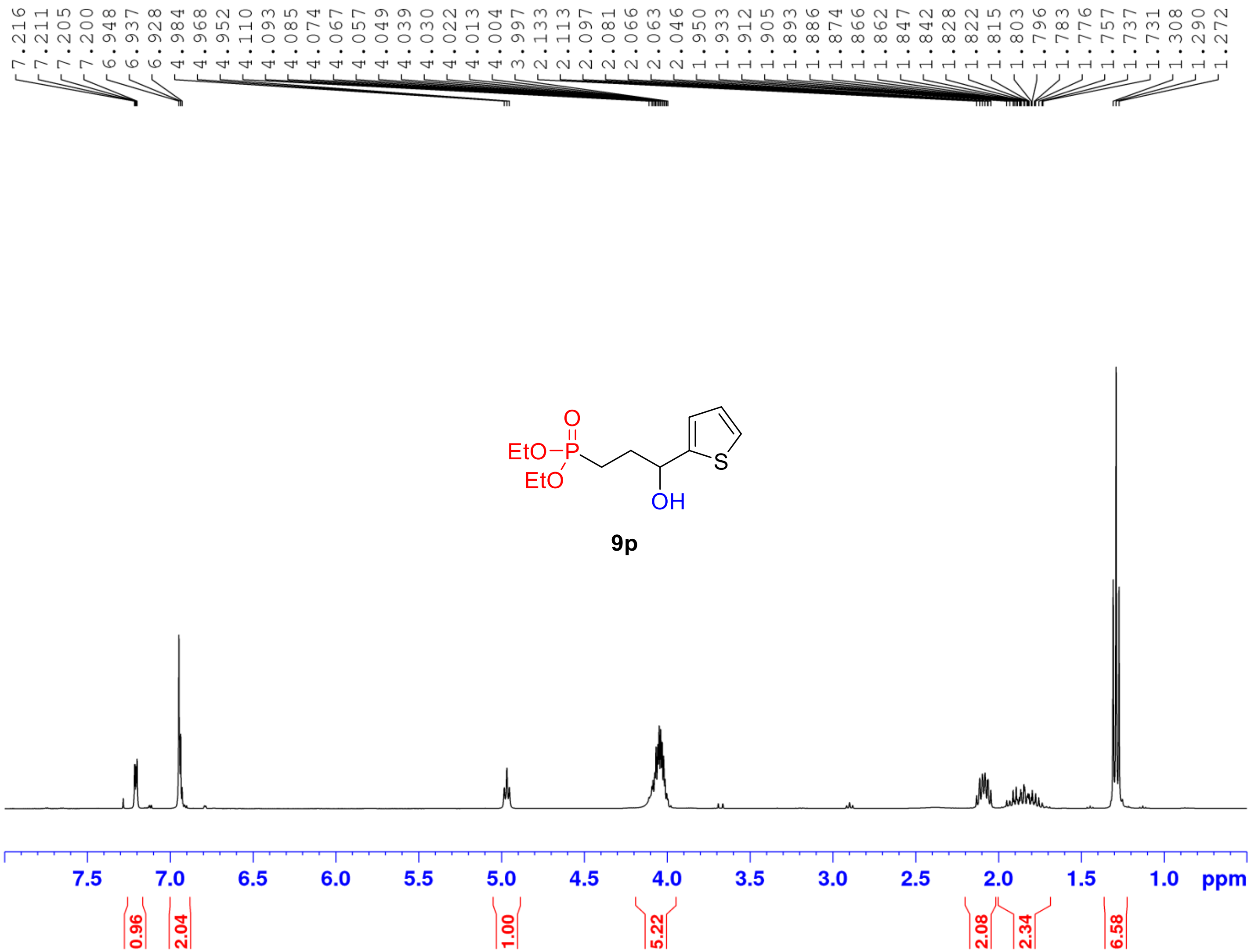
9o: ^{13}C HSQC





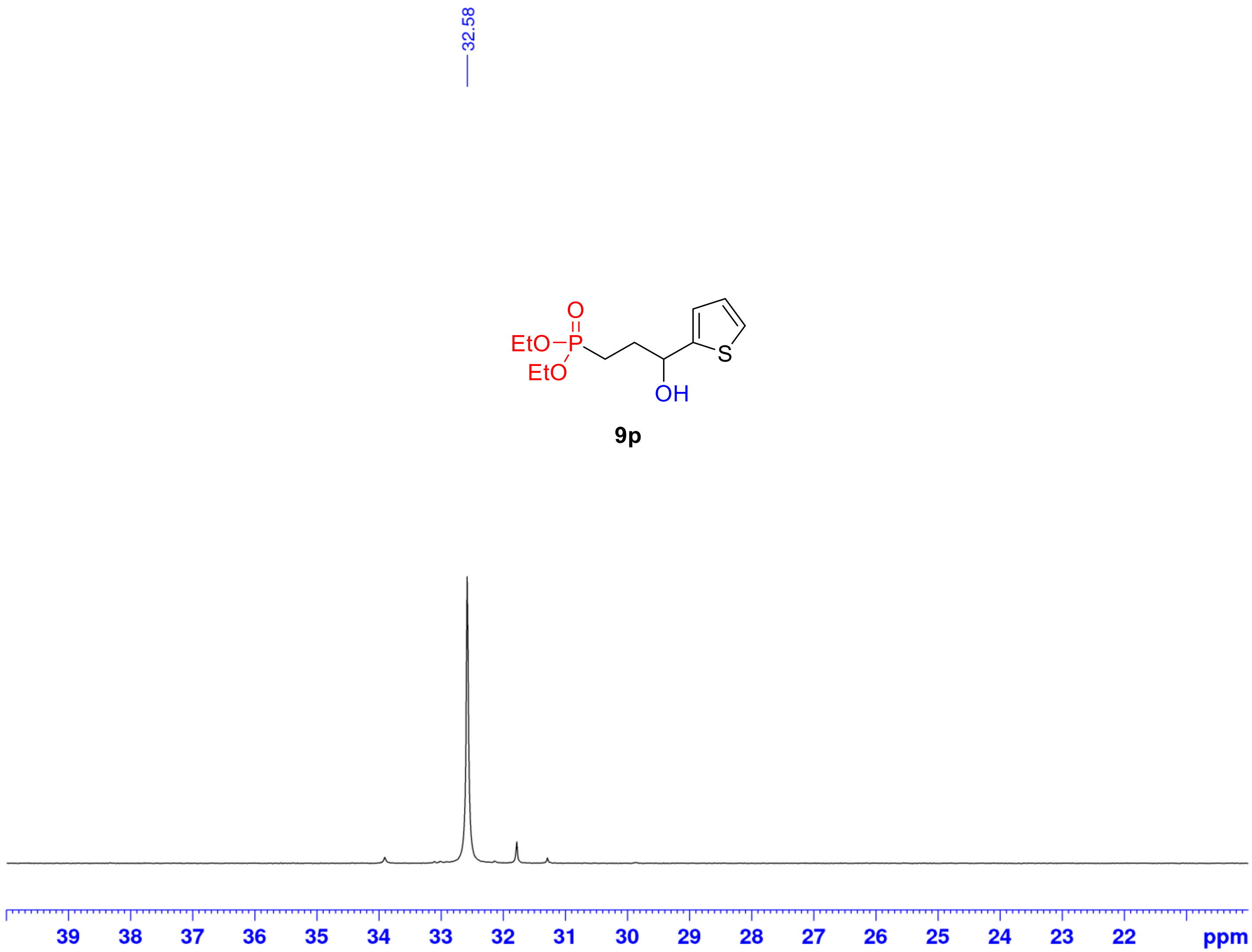
9o

9p: ^{1}H NMR



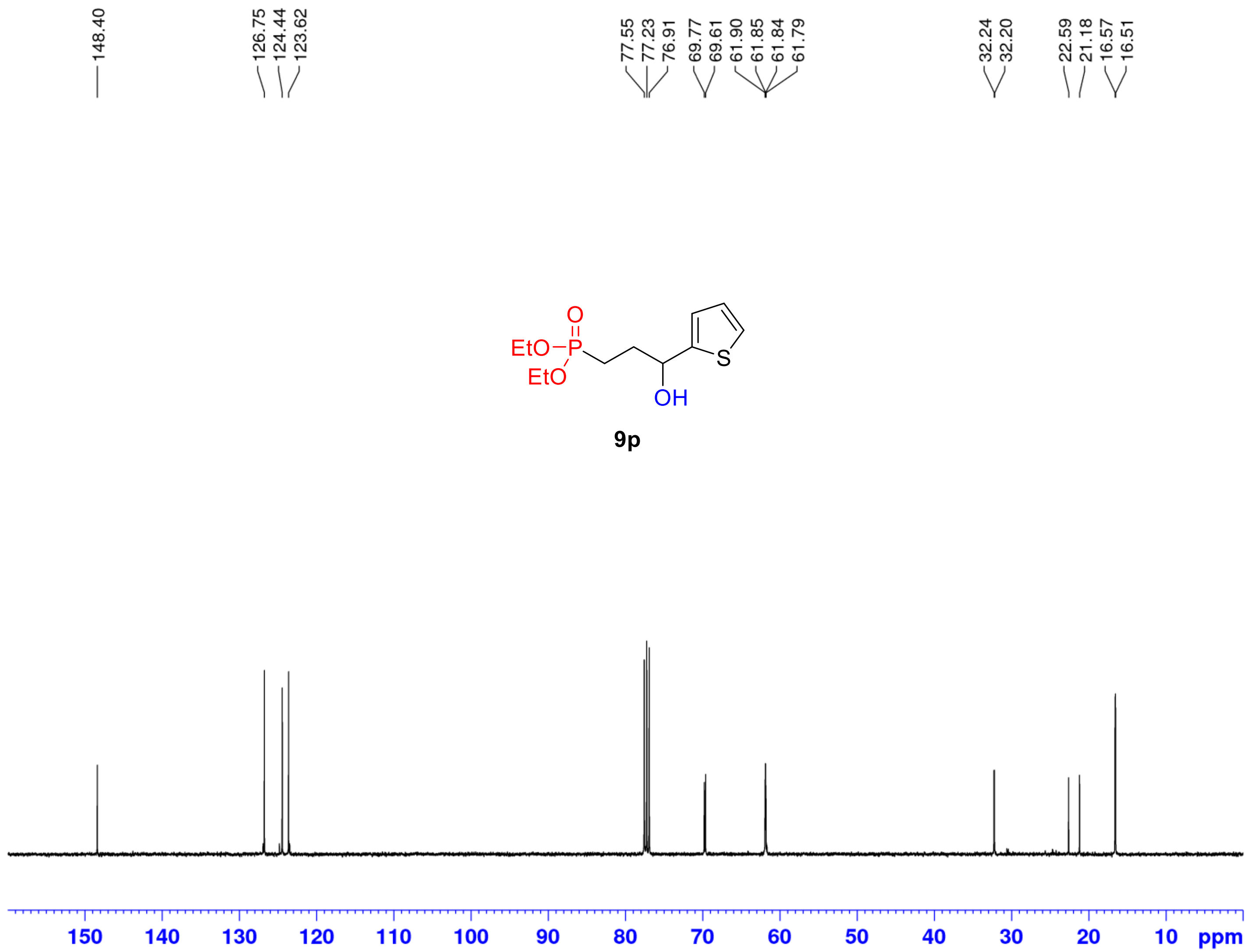
7.216
7.211
7.205
7.200
6.948
6.937
6.928
4.984
4.968
4.952
4.110
4.093
4.085
4.074
4.067
4.057
4.049
4.039
4.030
4.022
4.013
4.004
3.997
2.133
2.113
2.097
2.081
2.066
2.063
2.046
1.950
1.933
1.912
1.905
1.893
1.886
1.874
1.866
1.862
1.847
1.842
1.828
1.822
1.815
1.803
1.796
1.783
1.776
1.757
1.737
1.731
1.308
1.290
1.272
O
EtO
P
EtO
S
OH
9p
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
0.96
2.04
1.00
5.22
2.08
2.34
6.58

9p: ^{31}P NMR

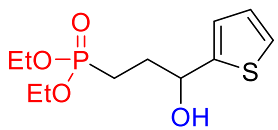


32.58
O
EtO
P
EtO
OH
S
9p
39 38 37 36 35 34 33 32 31 30 29 28 27 26 25 24 23 22
ppm

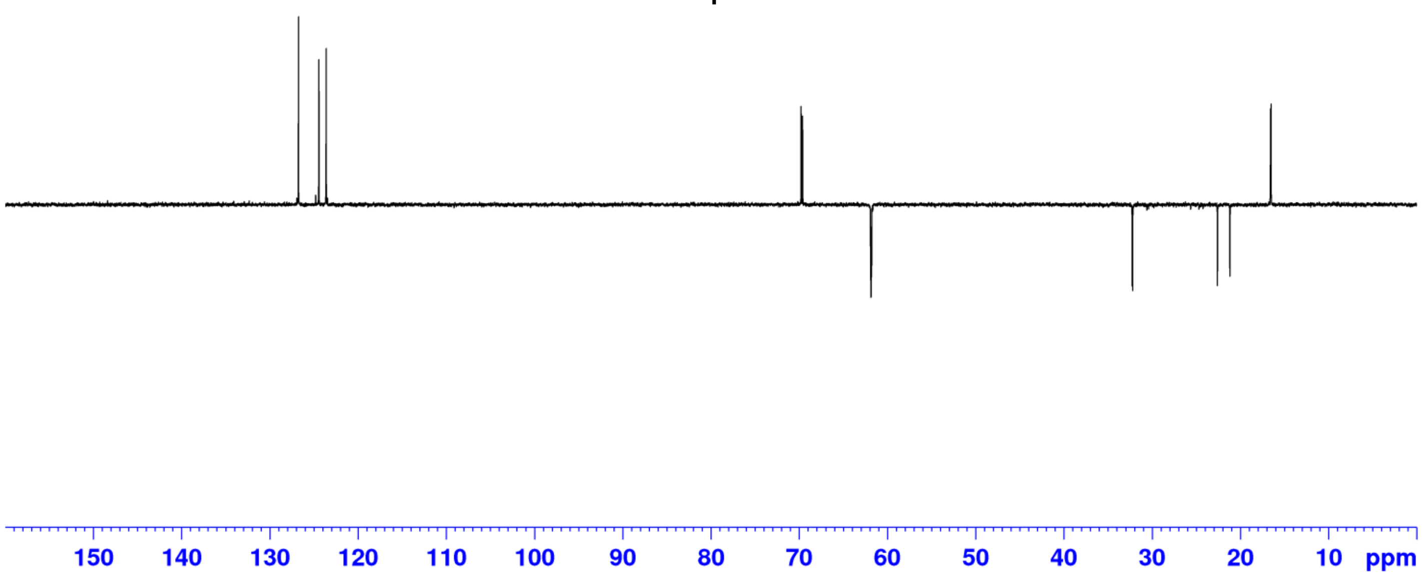
9p: ^{13}C NMR


148.40
126.75
124.44
123.62
77.55
77.23
76.91
69.77
69.61
61.90
61.85
61.84
61.79
32.24
32.20
22.59
21.18
16.57
16.51
O
EtO
P
EtO
S
OH
9p
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

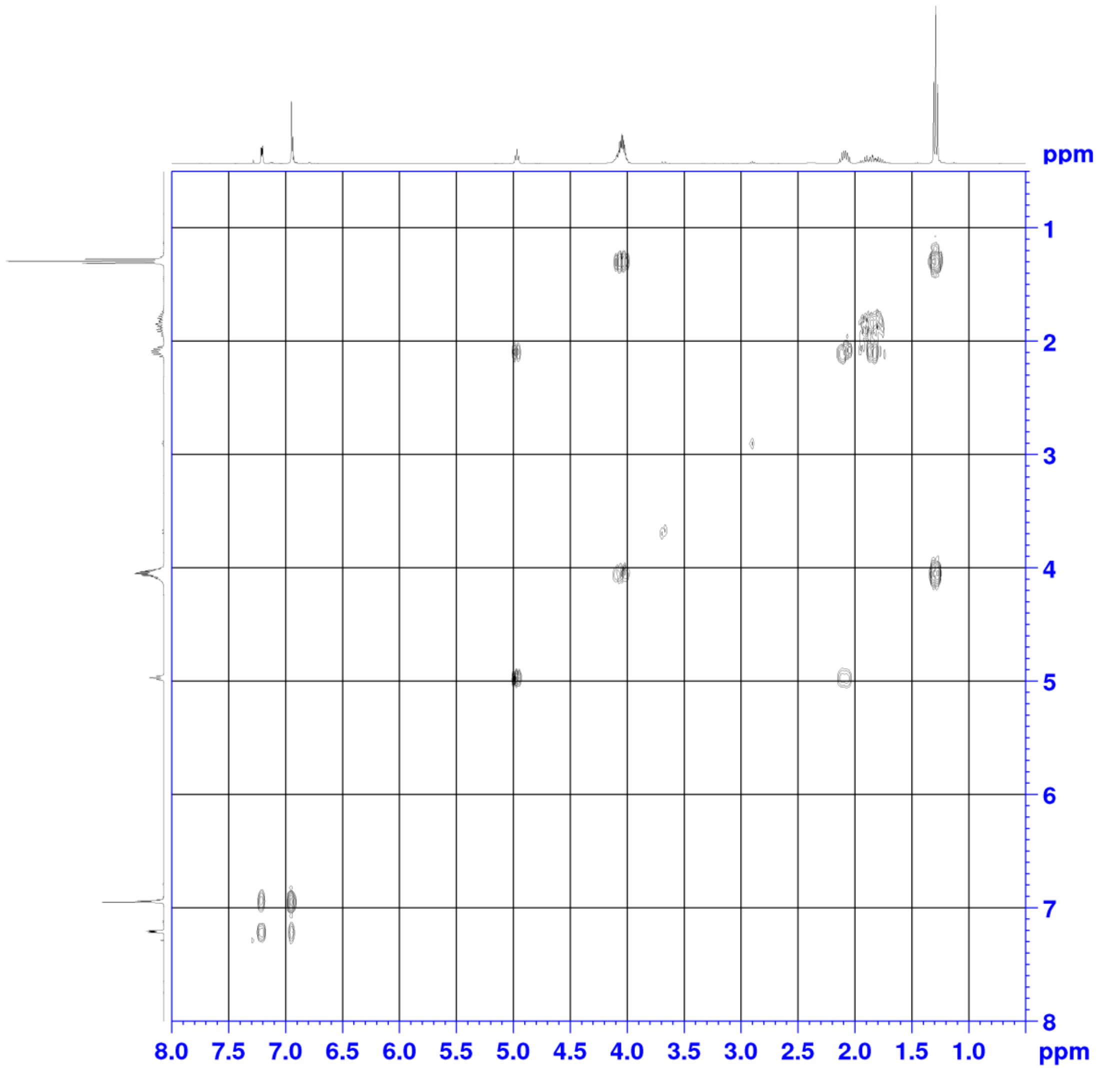
9p: ^{13}C DEPT135

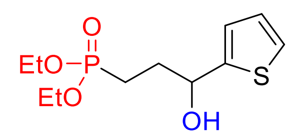


9p



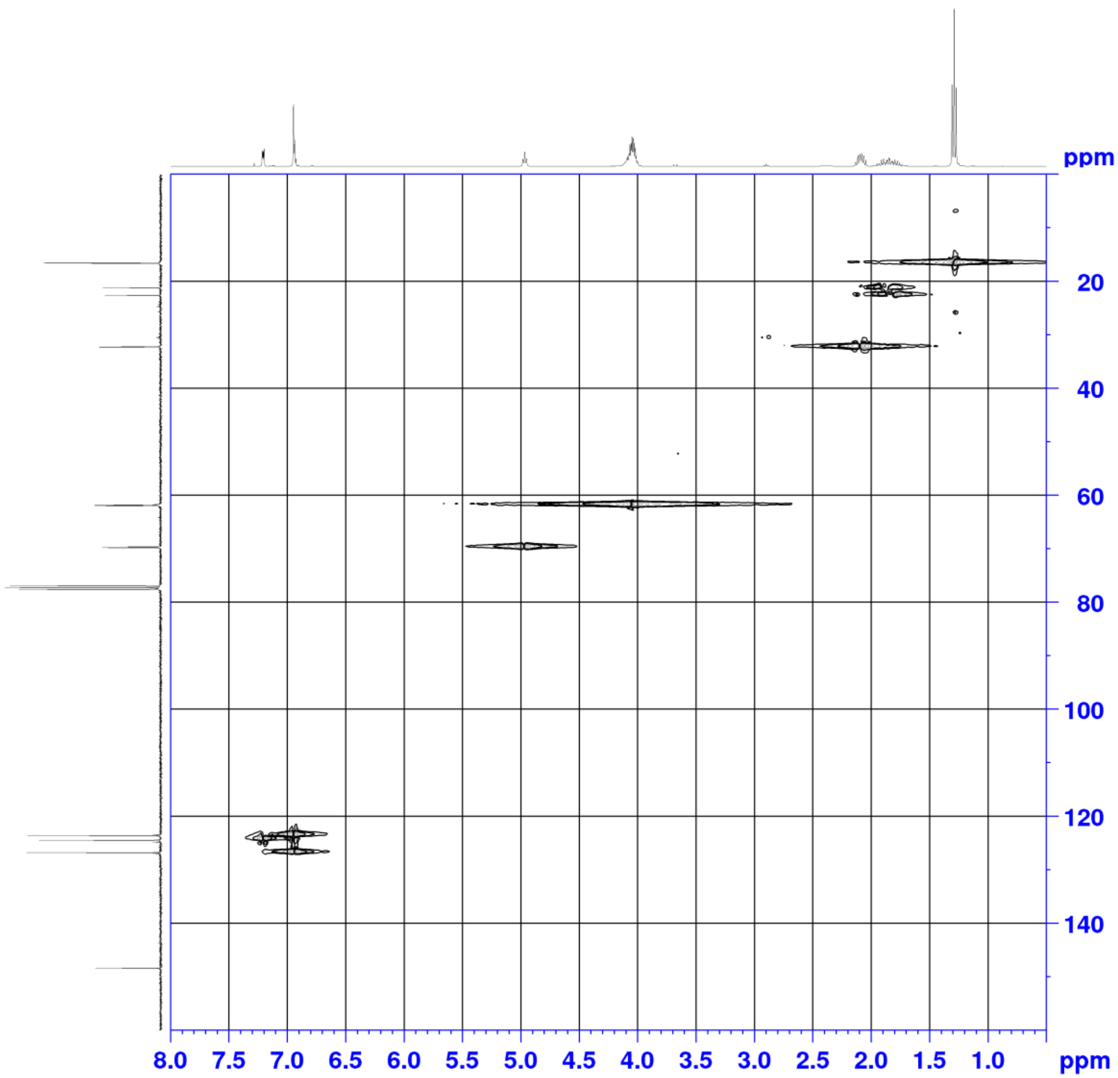
9p: COSY

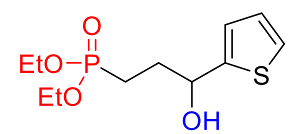




9p

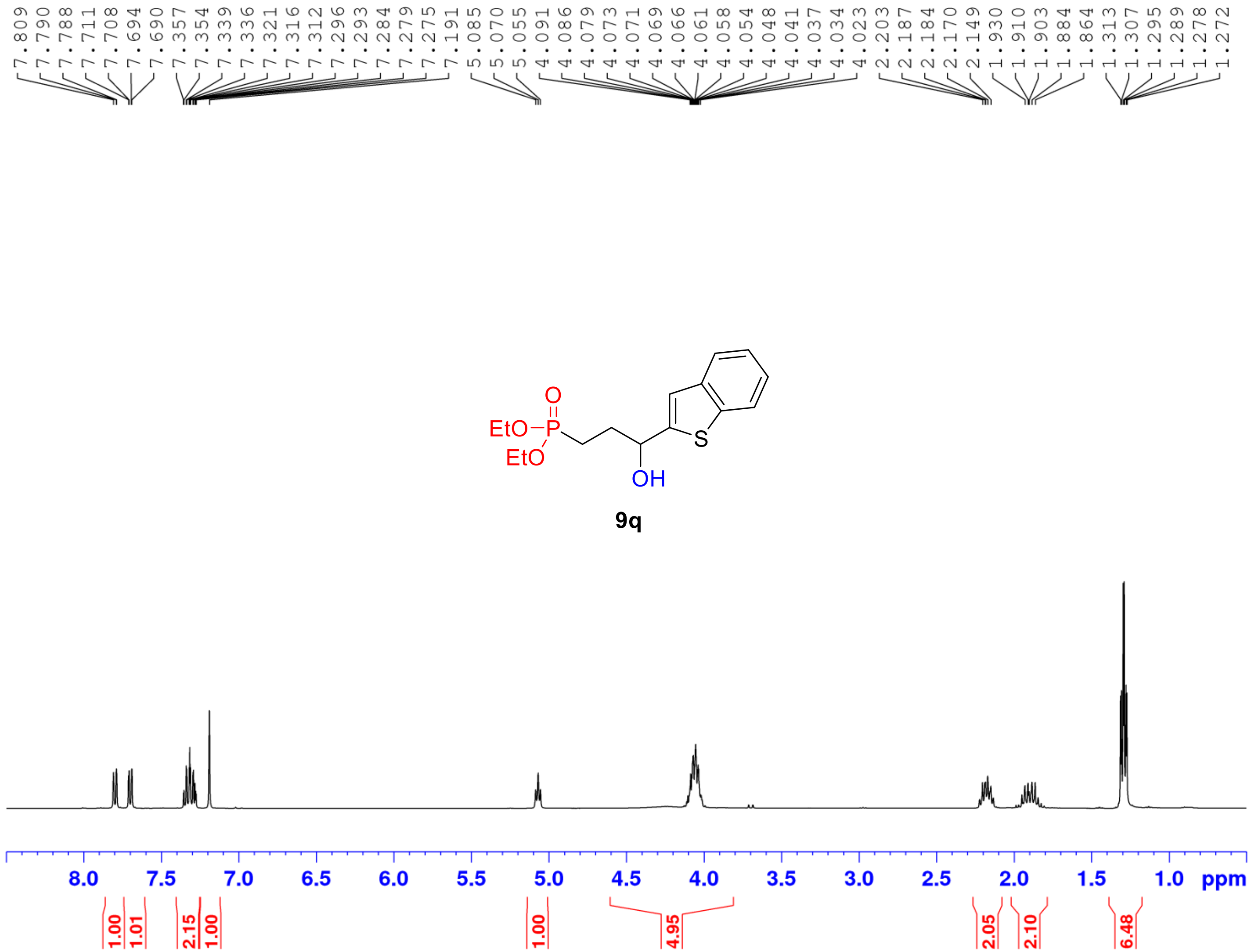
9p: ^{13}C HSQC





9p

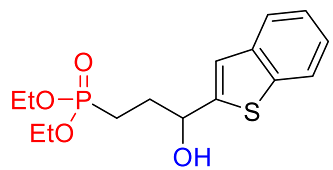
9q: ^{1}H NMR



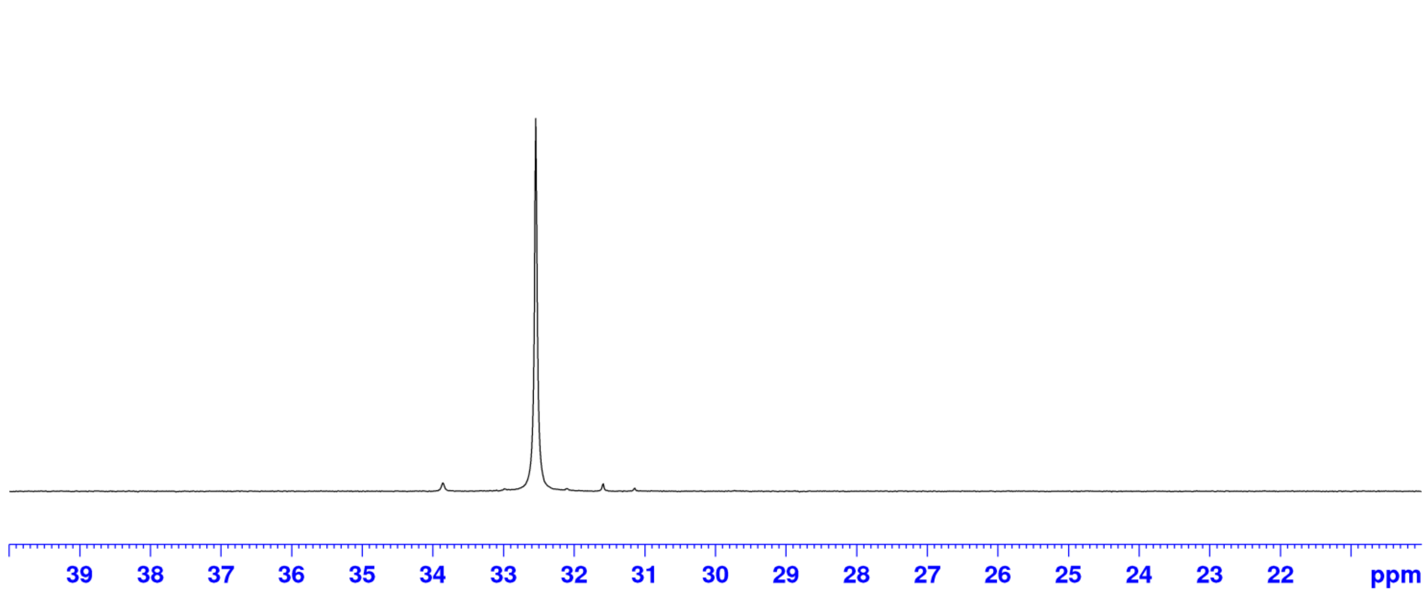
7.809
7.790
7.788
7.711
7.708
7.694
7.690
7.357
7.354
7.339
7.336
7.321
7.316
7.312
7.296
7.293
7.284
7.279
7.275
7.191
5.085
5.070
5.055
4.091
4.086
4.079
4.073
4.071
4.069
4.066
4.061
4.058
4.054
4.048
4.041
4.037
4.034
4.023
2.203
2.187
2.184
2.170
2.149
1.930
1.910
1.903
1.884
1.864
1.313
1.307
1.295
1.289
1.278
1.272
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
ppm
1.00
1.01
2.15
1.00
1.00
4.95
2.05
2.10
6.48
O
EtO
P
EtO
S
OH

9q

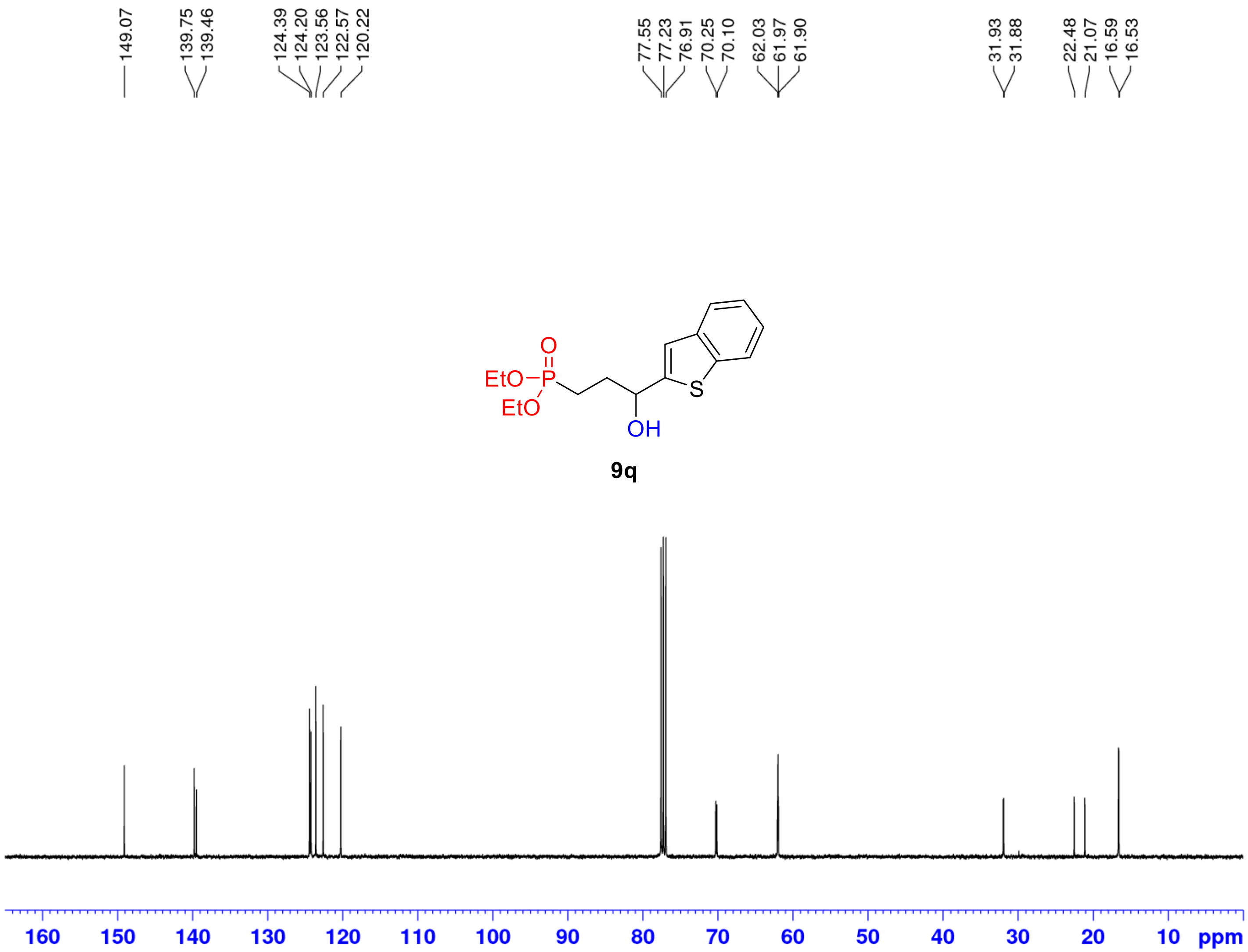
9q: ^{31}P NMR



9q

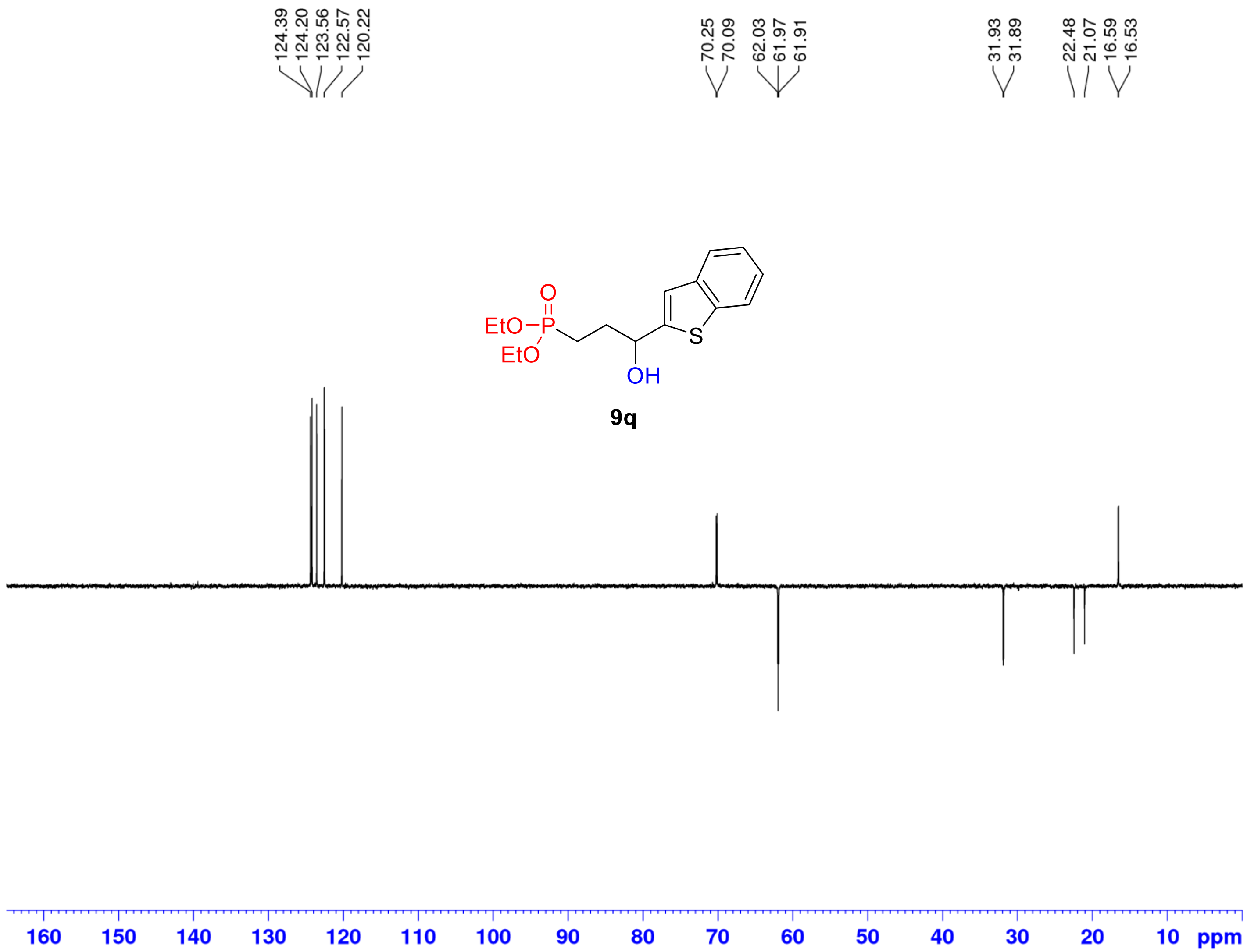


9q: ^{13}C NMR

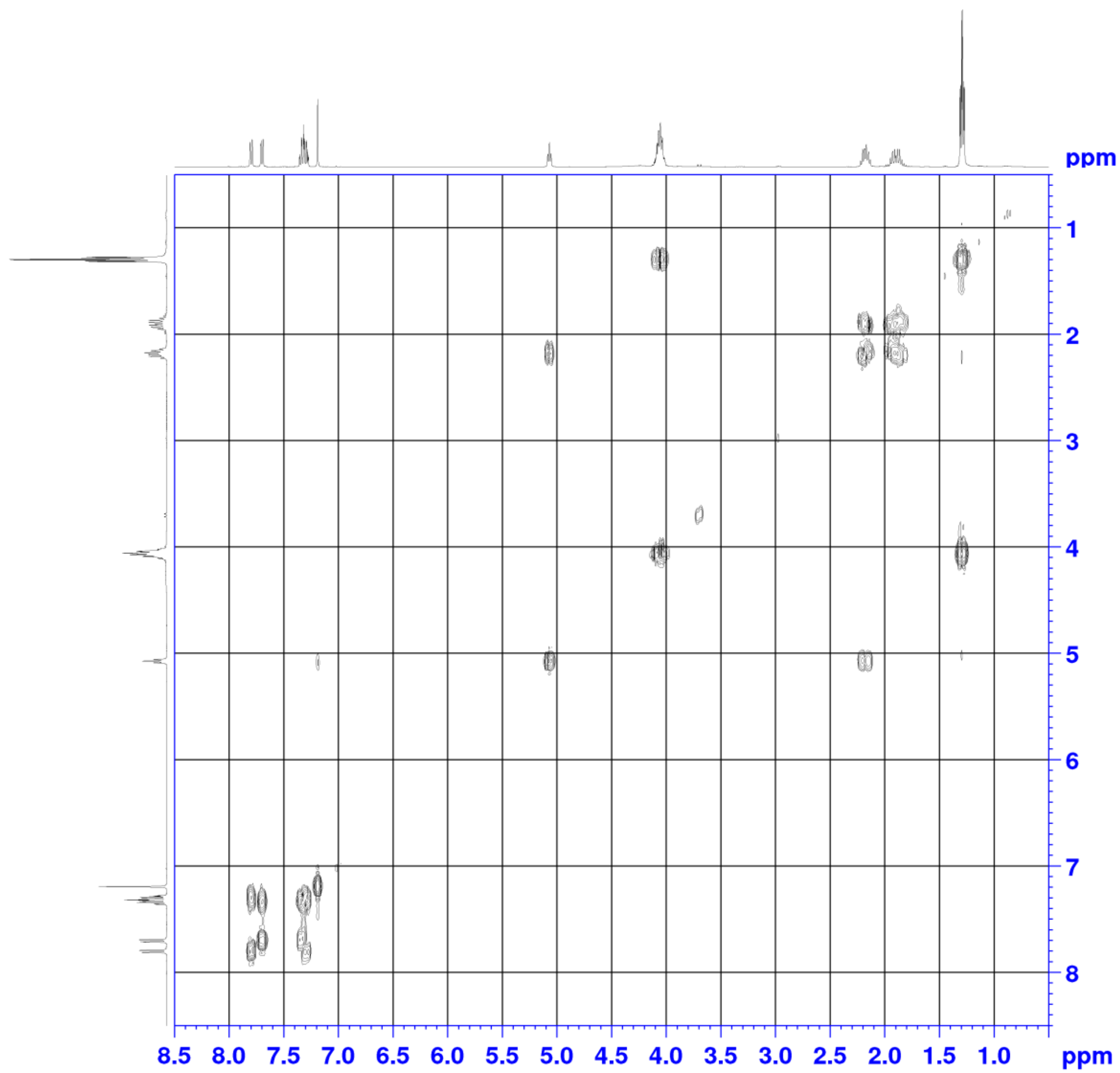


149.07
139.75
139.46
124.39
124.20
123.56
122.57
120.22
77.55
77.23
76.91
70.25
70.10
62.03
61.97
61.90
31.93
31.88
22.48
21.07
16.59
16.53
O
EtO
P
EtO
S
OH
9q
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

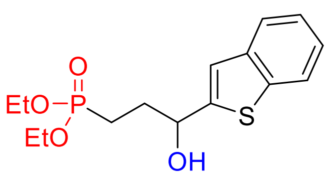
9q: ^{13}C DEPT135



9q: COSY



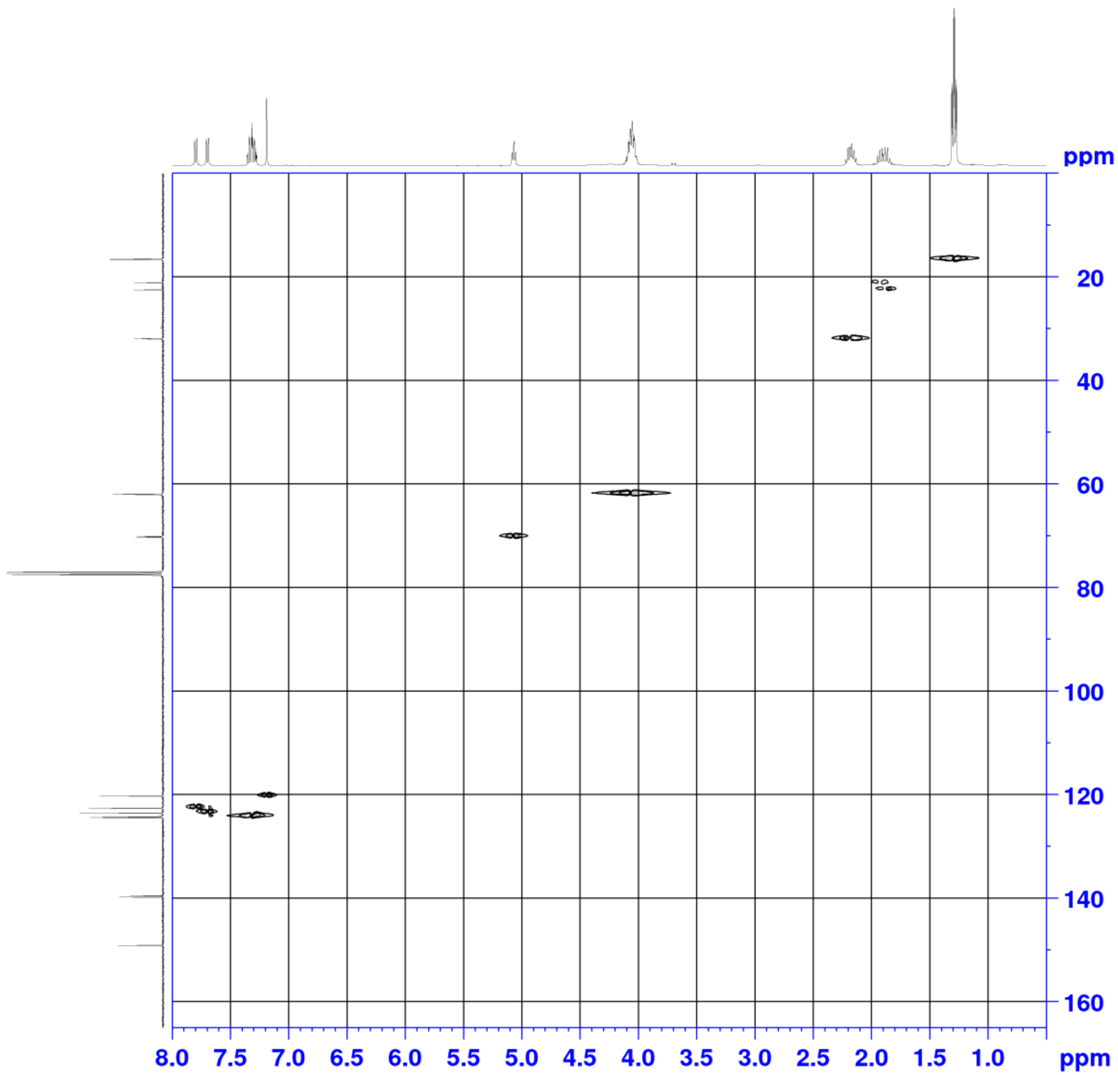
ppm
1
2
3
4
5
6
7
8
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

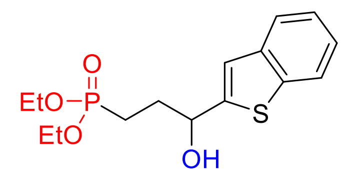


O
EtO
P
EtO
S
OH

9q

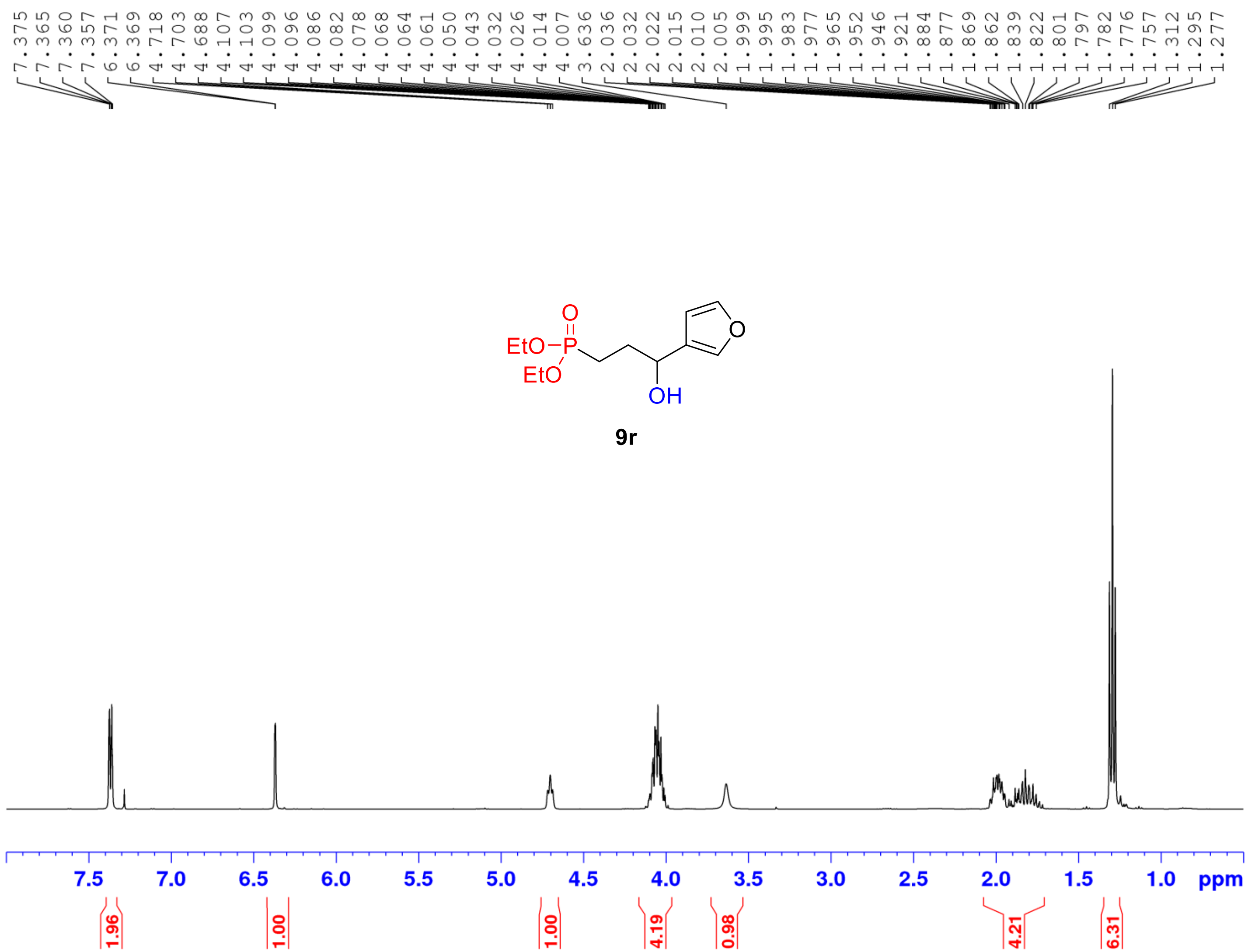
9q: ^{13}C HSQC



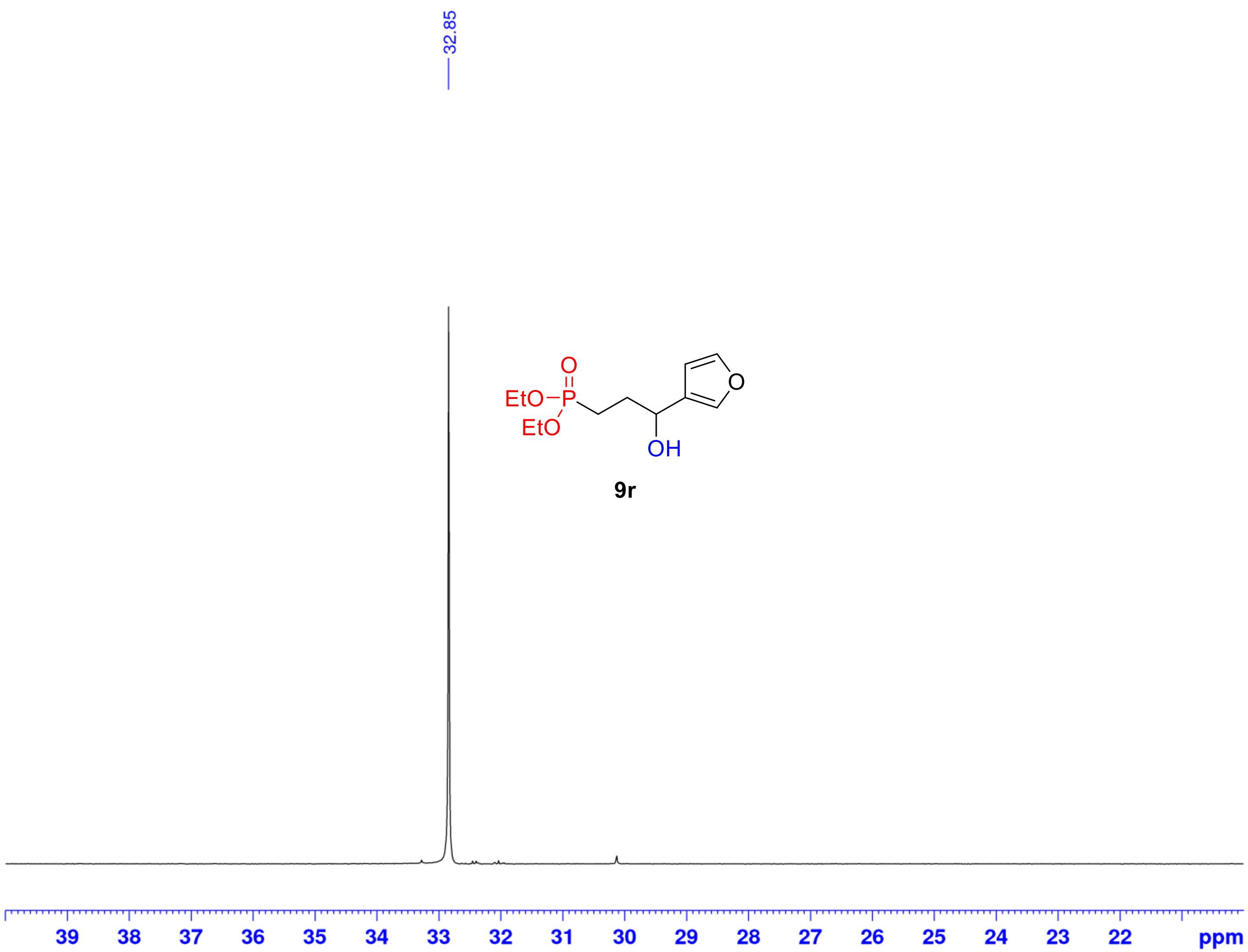


9q

9r: ^{1}H NMR

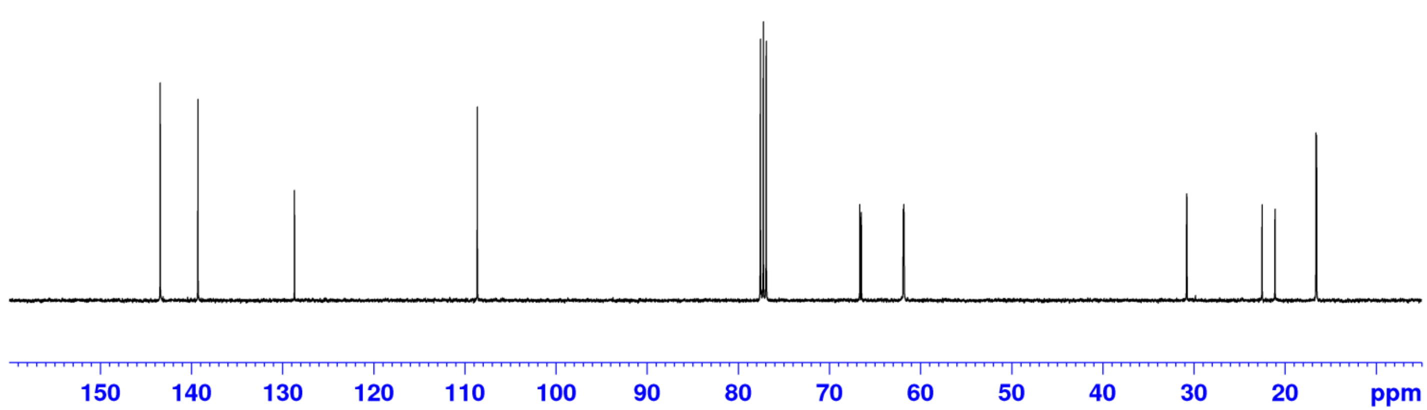

7.375
7.365
7.360
7.357
6.371
6.369
4.718
4.703
4.688
4.107
4.103
4.099
4.096
4.086
4.082
4.078
4.068
4.064
4.061
4.050
4.043
4.032
4.026
4.014
4.007
3.636
2.036
2.032
2.022
2.015
2.010
2.005
1.999
1.995
1.983
1.977
1.965
1.952
1.946
1.921
1.884
1.877
1.869
1.862
1.839
1.822
1.801
1.797
1.782
1.776
1.757
1.312
1.295
1.277
O
EtO
P
EtO
O
OH
9r
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
1.96
1.00
1.00
4.19
0.98
4.21
6.31

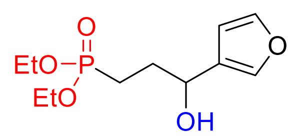
9r: ^{31}P NMR



32.85
O
EtO−P
EtO
O
OH
9r
39 38 37 36 35 34 33 32 31 30 29 28 27 26 25 24 23 22
ppm

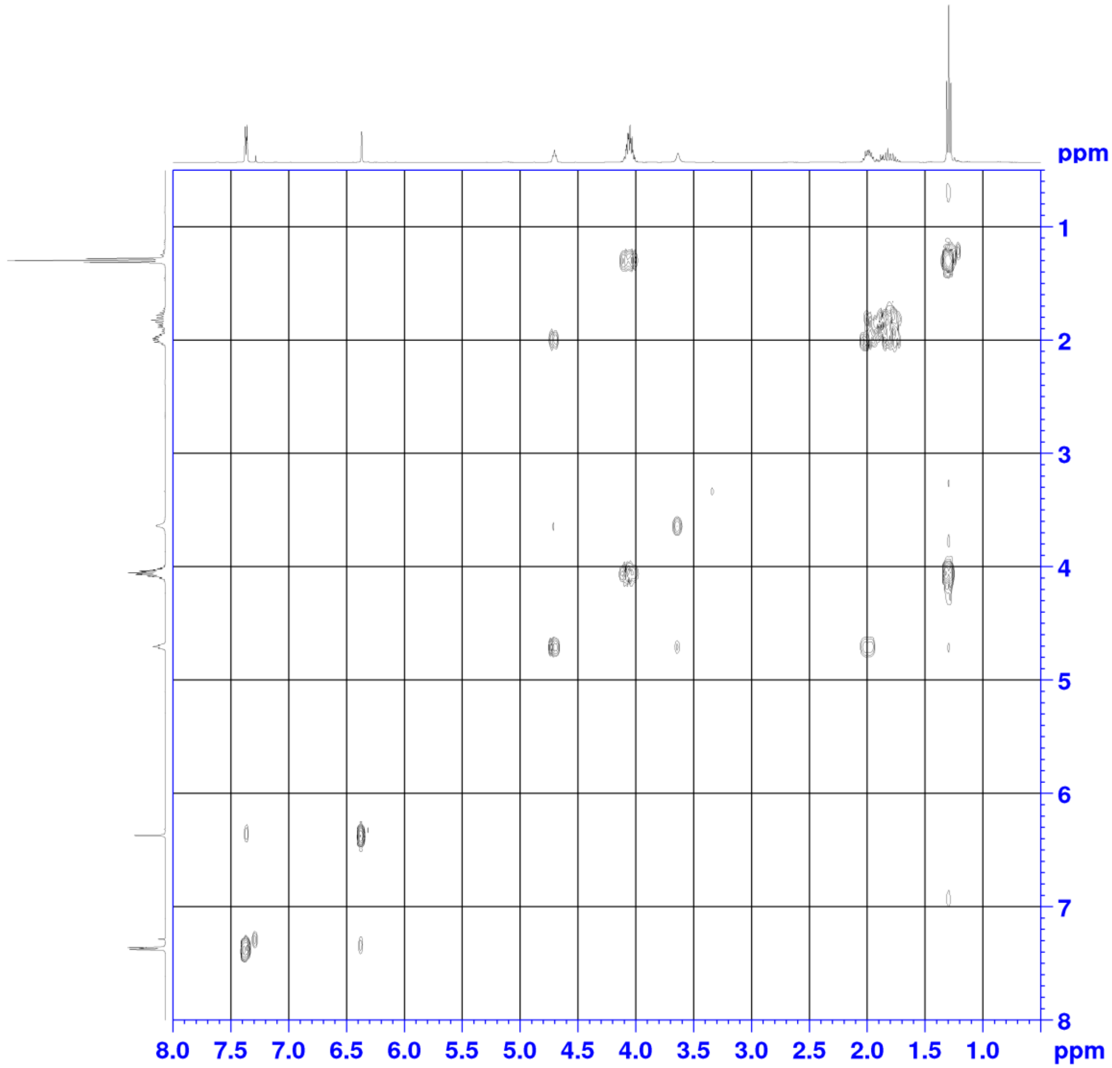
9r: ^{13}C DEPT135

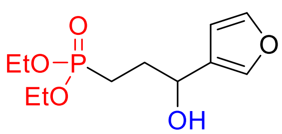




9r

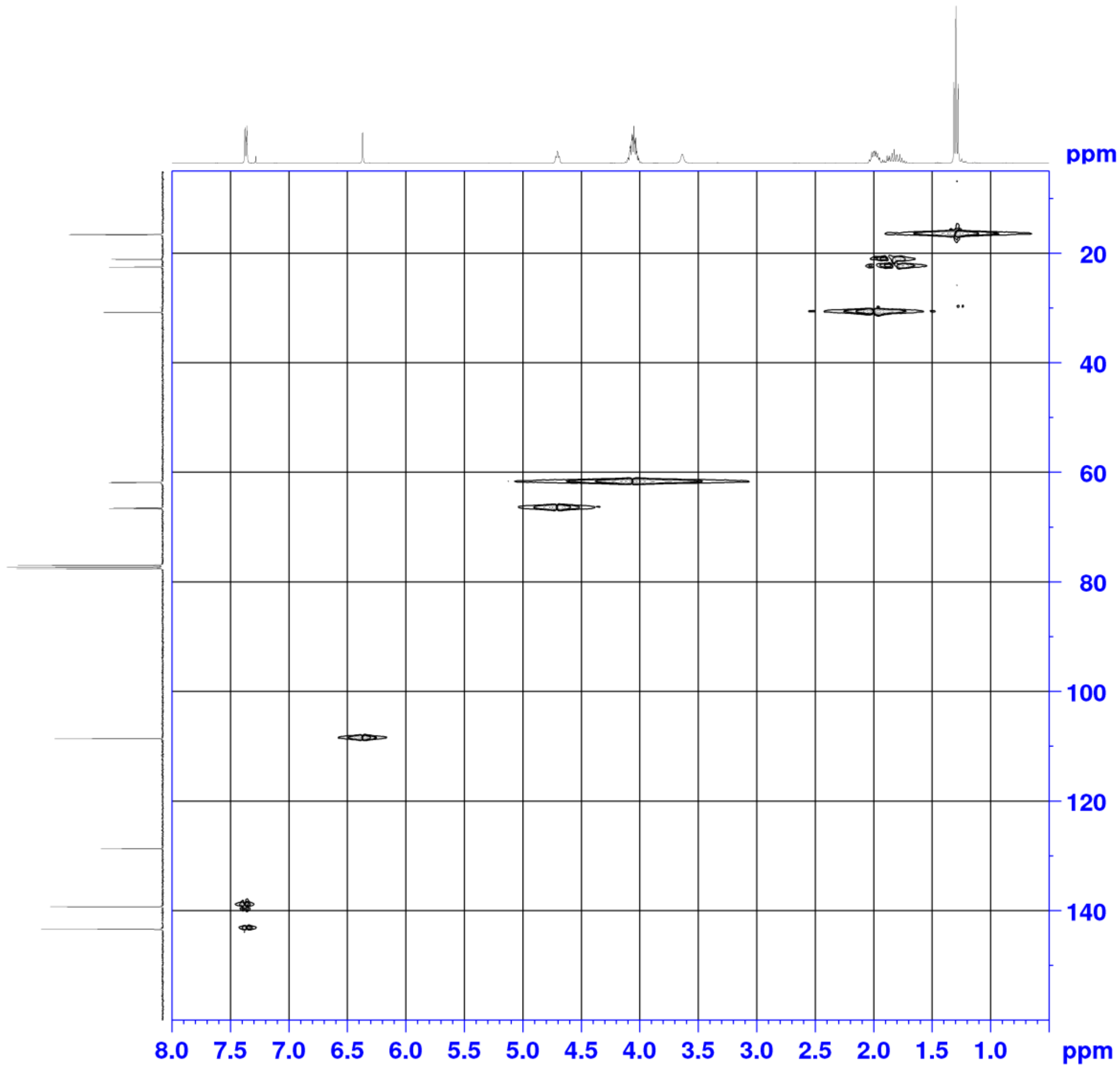
9r: COSY

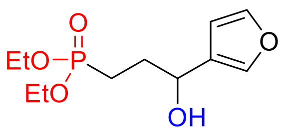

ppm
1
2
3
4
5
6
7
8
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0
ppm


O
EtO
P
EtO
O
OH

9r

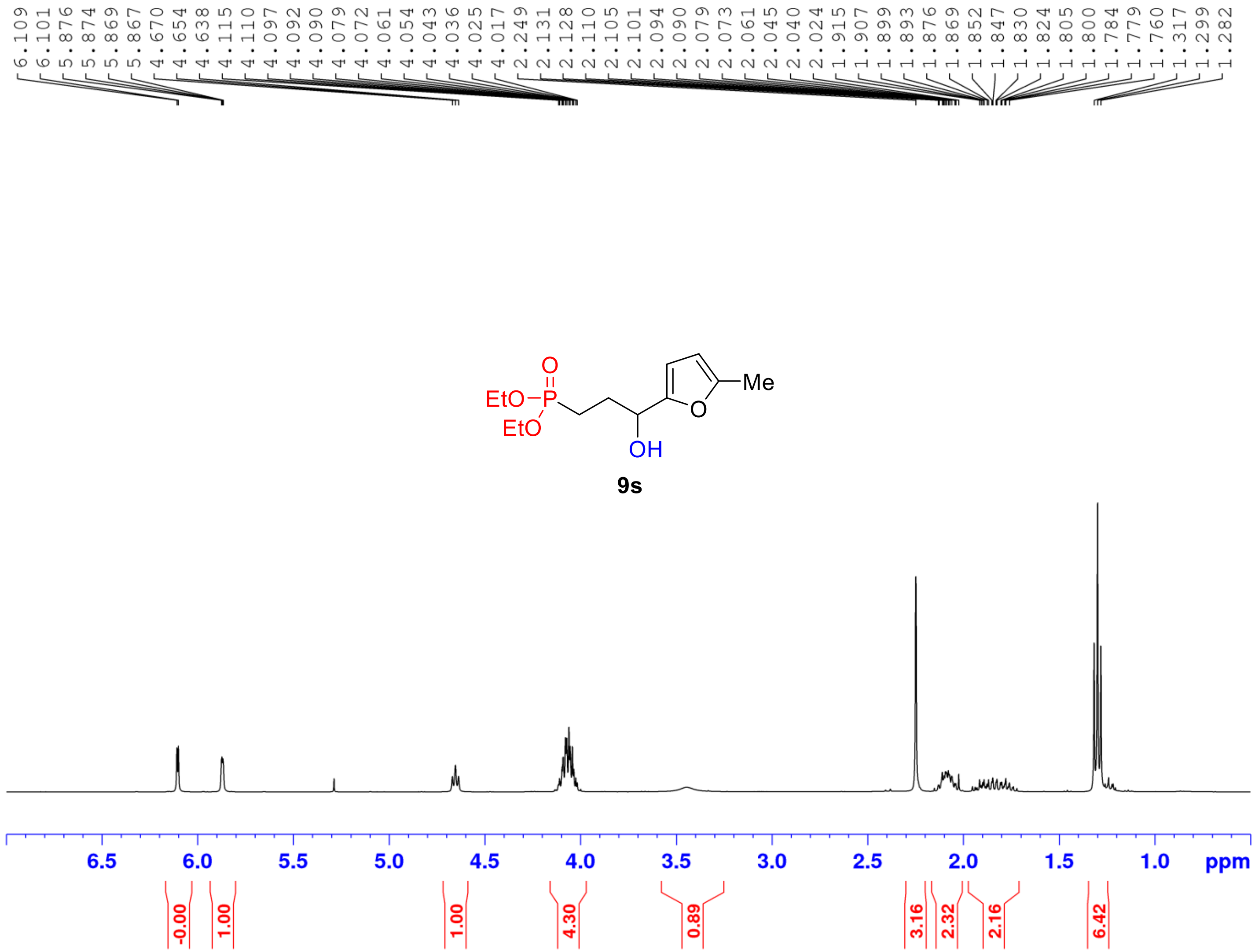
9r: ^{13}C HSQC





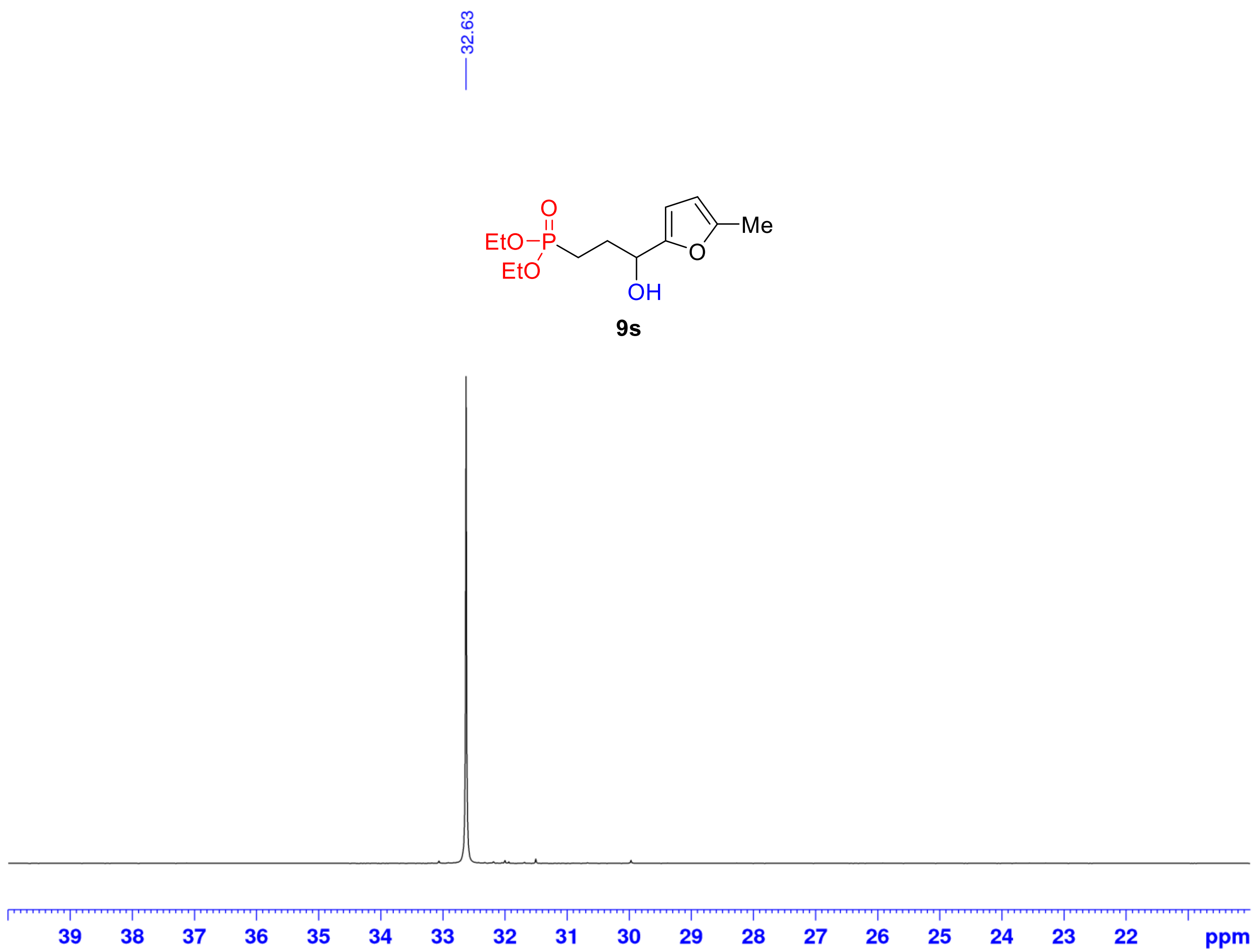
9r

9s: ^{1}H NMR



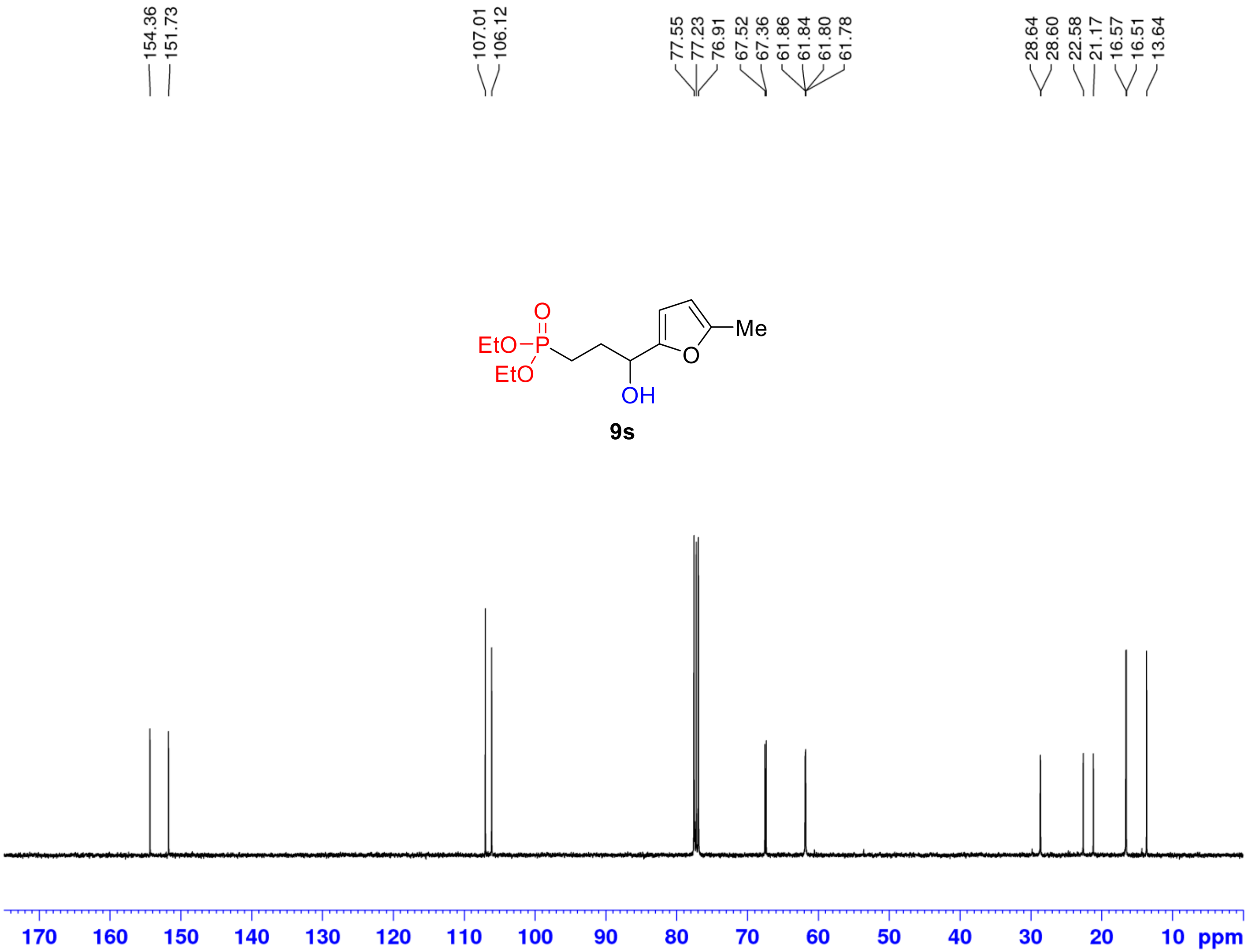
6.109
6.101
5.876
5.874
5.869
5.867
4.670
4.654
4.638
4.115
4.110
4.097
4.092
4.090
4.079
4.072
4.061
4.054
4.043
4.036
4.025
4.017
2.249
2.131
2.128
2.110
2.105
2.101
2.094
2.090
2.079
2.073
2.061
2.045
2.040
2.024
1.915
1.907
1.899
1.893
1.876
1.869
1.852
1.847
1.830
1.824
1.805
1.800
1.784
1.779
1.760
1.317
1.299
1.282
O
EtO
P
EtO
O
Me
OH
9s
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
-0.00
1.00
1.00
4.30
0.89
3.16
2.32
2.16
6.42

9s: ^{31}P NMR



32.63
EtO
EtO
O
P
OH
O
Me
9s
39 38 37 36 35 34 33 32 31 30 29 28 27 26 25 24 23 22 ppm

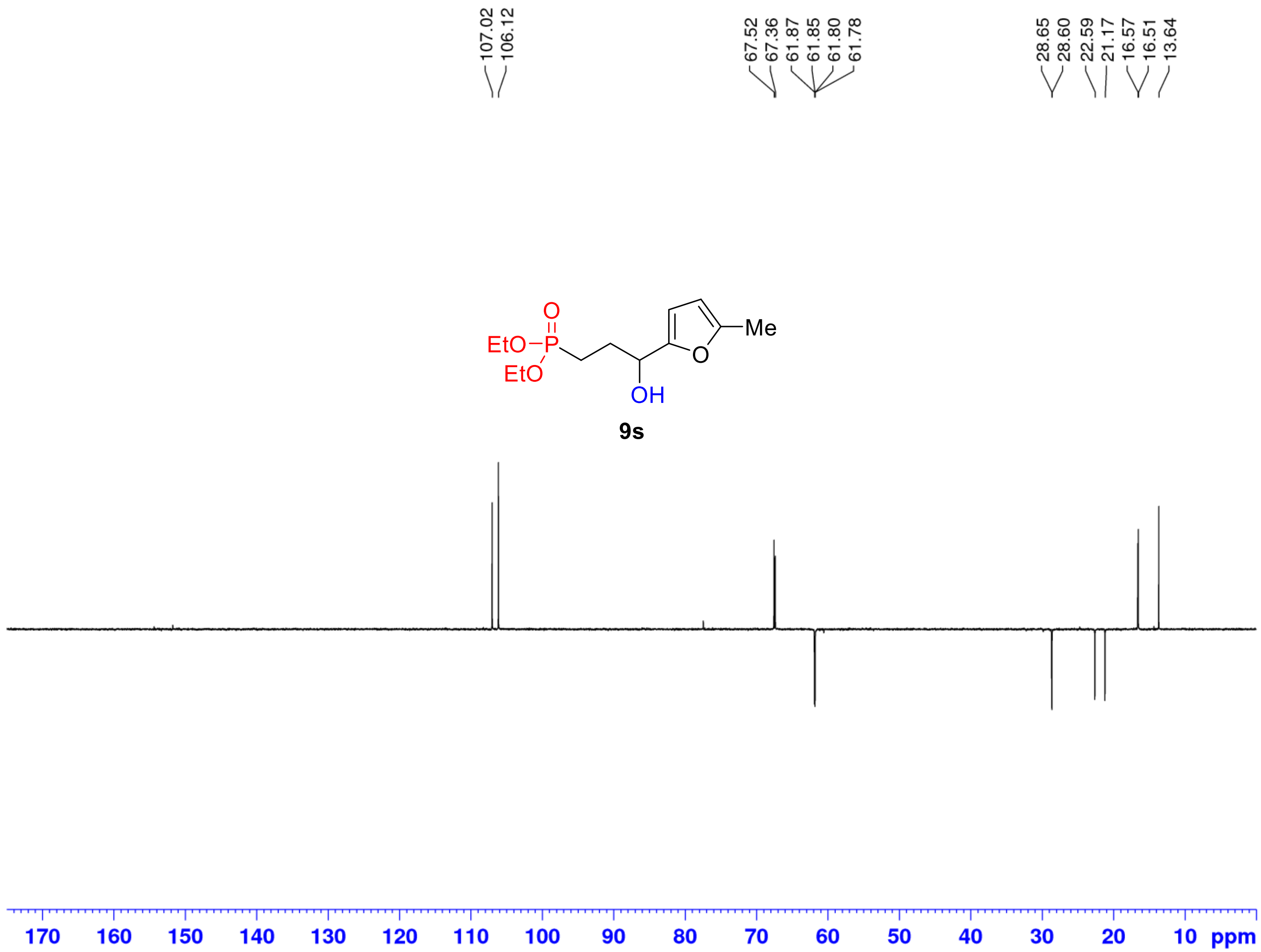
9s: ^{13}C NMR



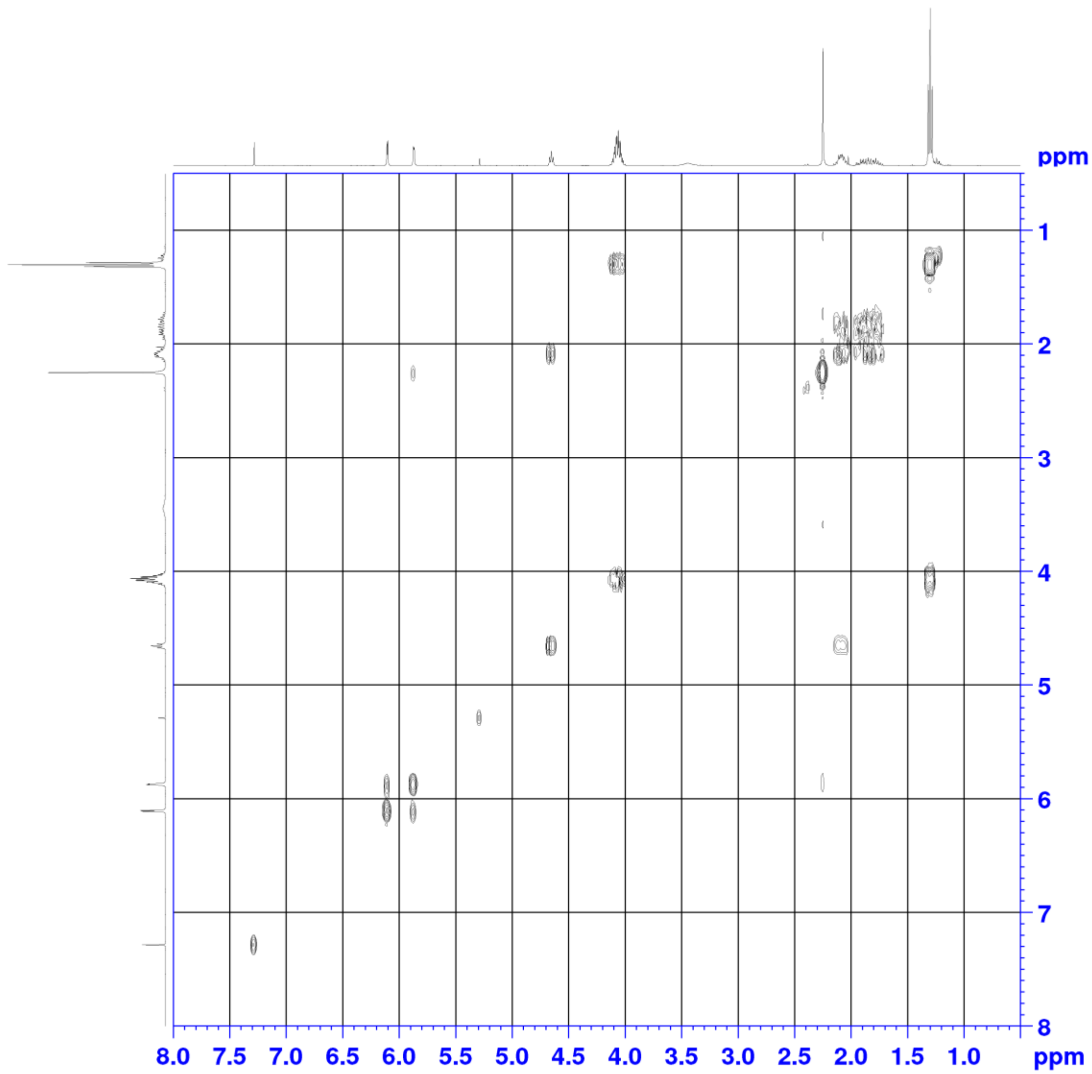
154.36
151.73
107.01
106.12
77.55
77.23
76.91
67.52
67.36
61.86
61.84
61.80
61.78
28.64
28.60
22.58
21.17
16.57
16.51
13.64
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm
EtO
EtO
P
O
OH
O
Me

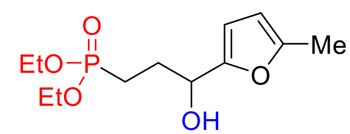
9s

9s: ^{13}C DEPT135



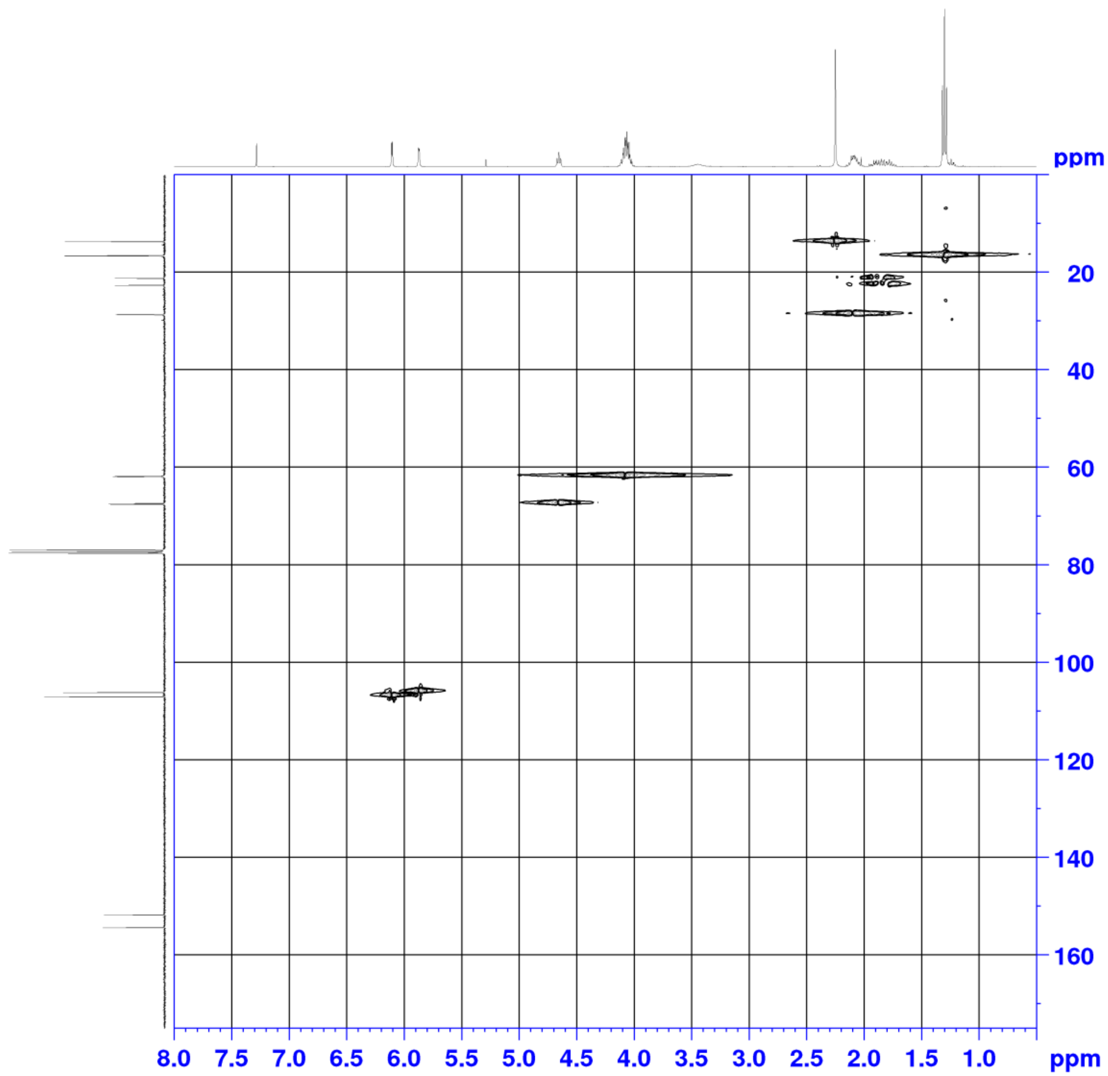
9s: COSY

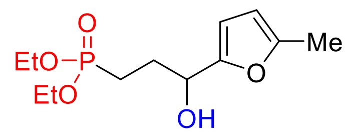

ppm
1
2
3
4
5
6
7
8
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0
ppm


O
EtO
P
EtO
OH
O
Me

9s

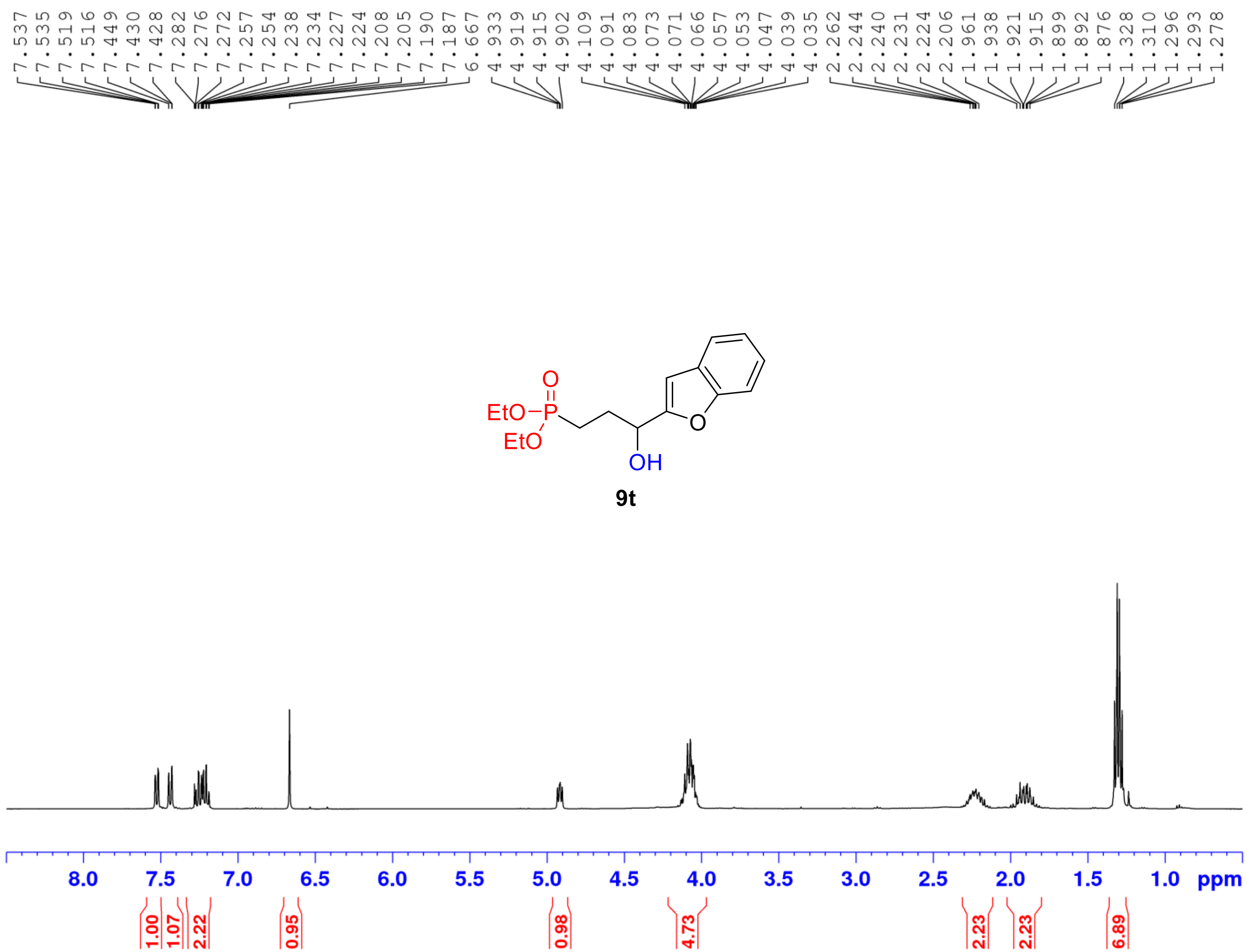
9s: ^{13}C HSQC





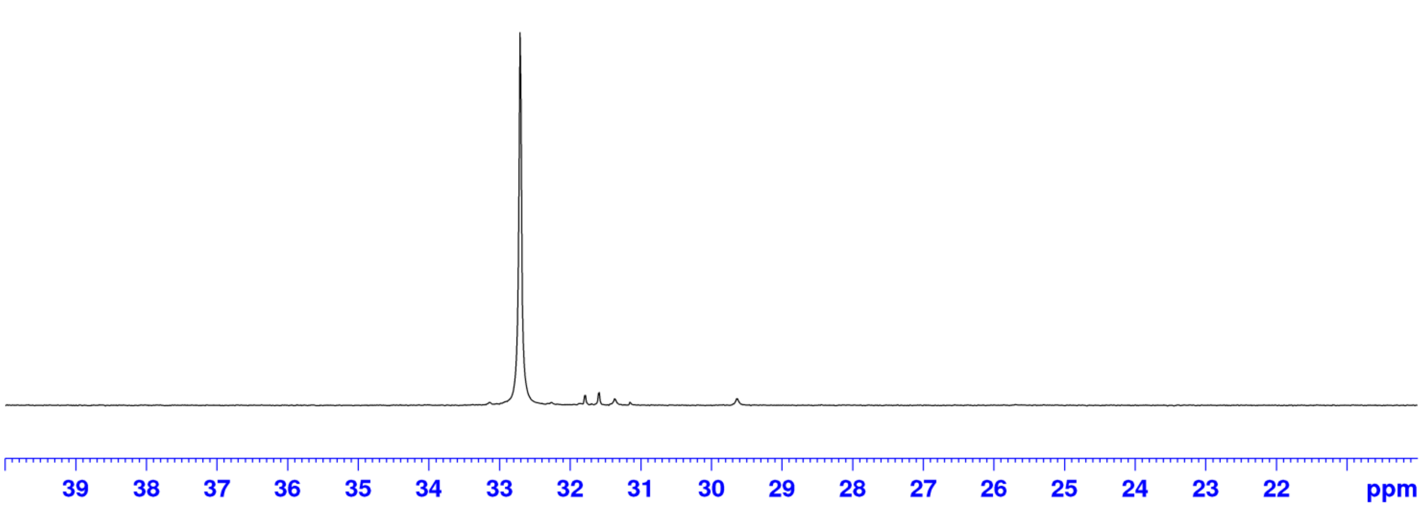
9s

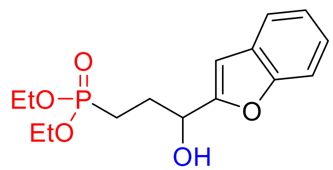
9t: ¹H NMR



7.537
7.535
7.519
7.516
7.449
7.430
7.428
7.282
7.276
7.272
7.257
7.254
7.238
7.234
7.227
7.224
7.208
7.205
7.190
7.187
6.667
4.933
4.919
4.915
4.902
4.109
4.091
4.083
4.073
4.071
4.066
4.057
4.053
4.047
4.039
4.035
2.262
2.244
2.240
2.231
2.224
2.206
1.961
1.938
1.921
1.915
1.899
1.892
1.876
1.328
1.310
1.296
1.293
1.278
EtO
EtO
P
O
O
OH
9t
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
ppm
1.00
1.07
2.22
0.95
0.98
4.73
2.23
2.23
6.89

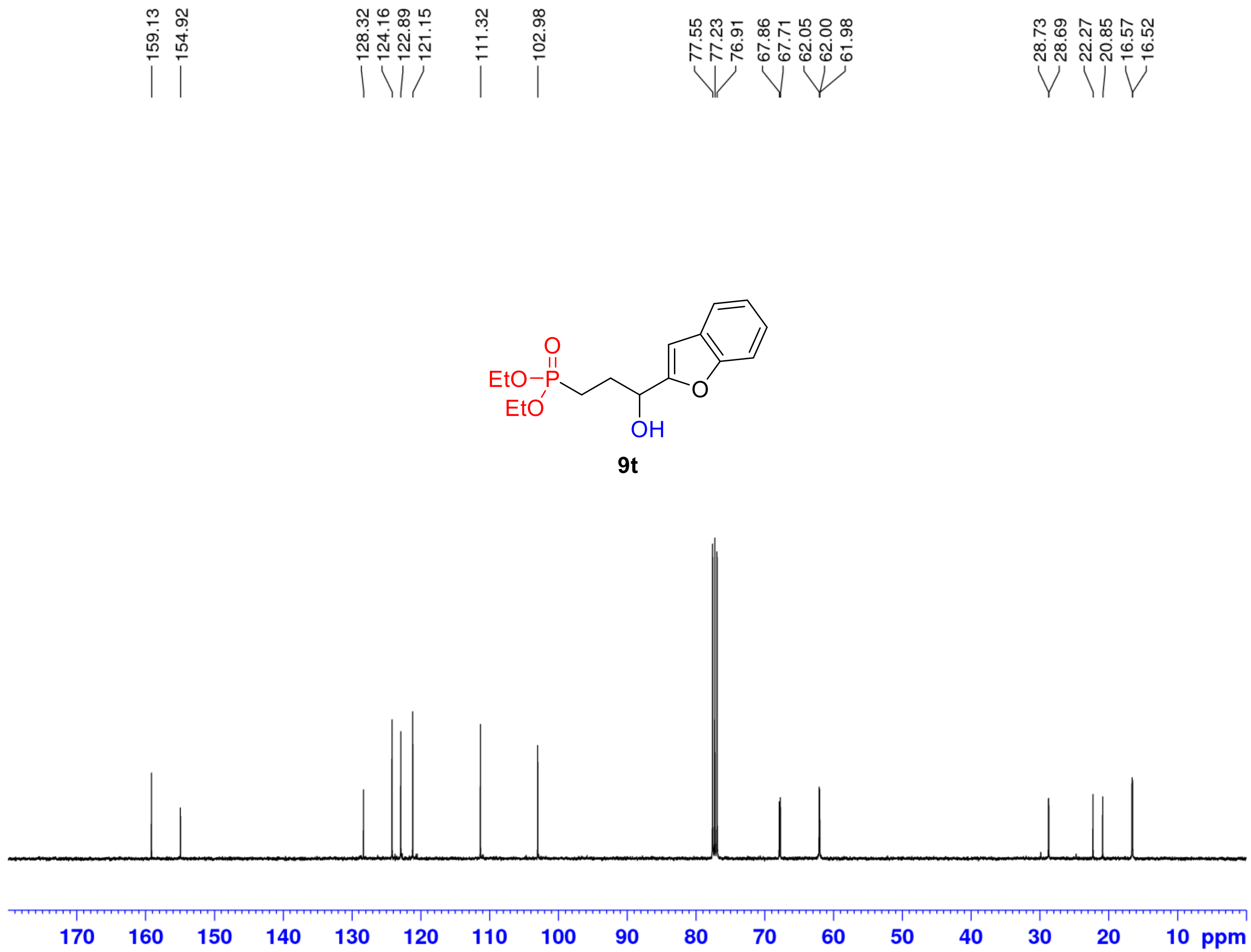
9t: ^{31}P NMR



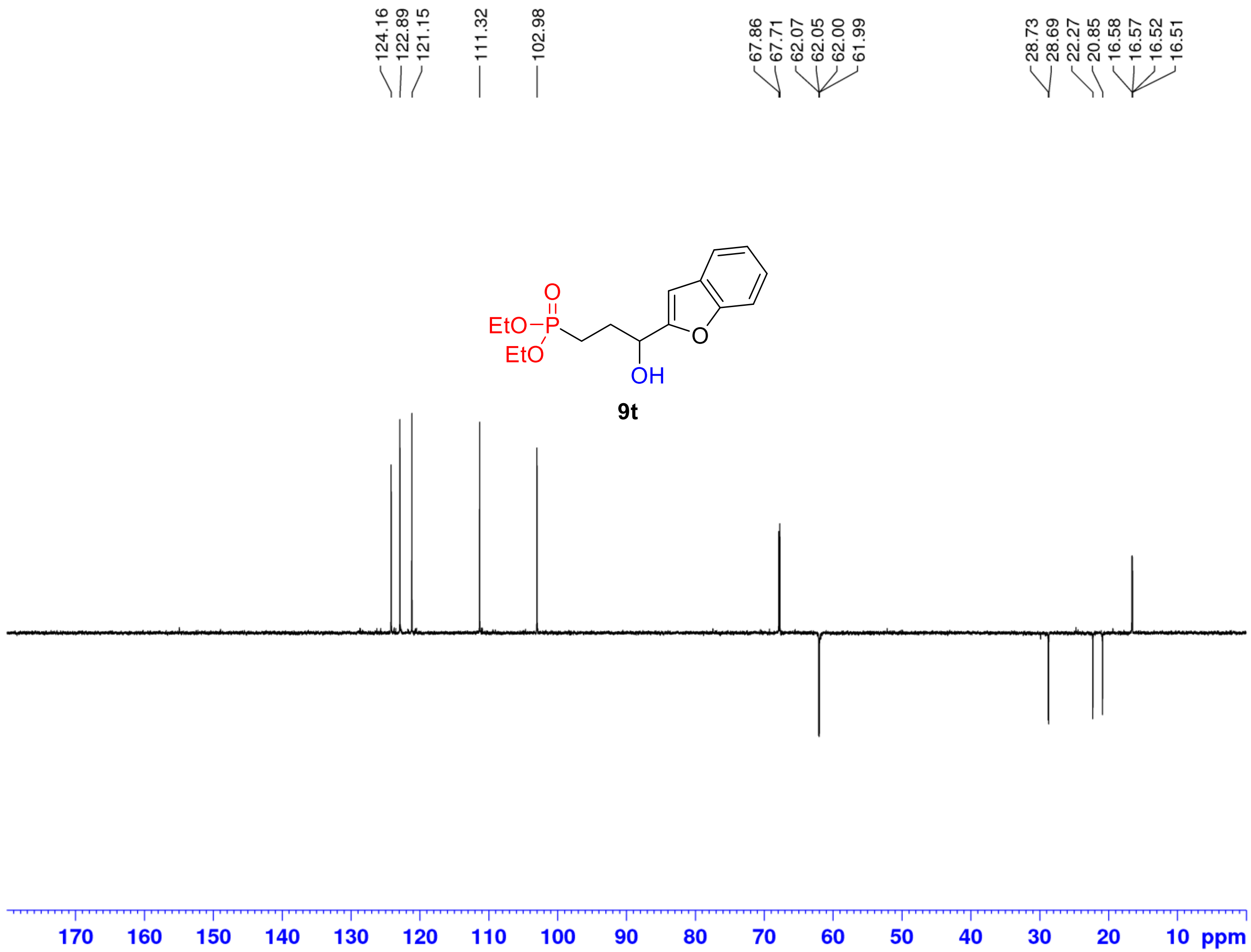


9t

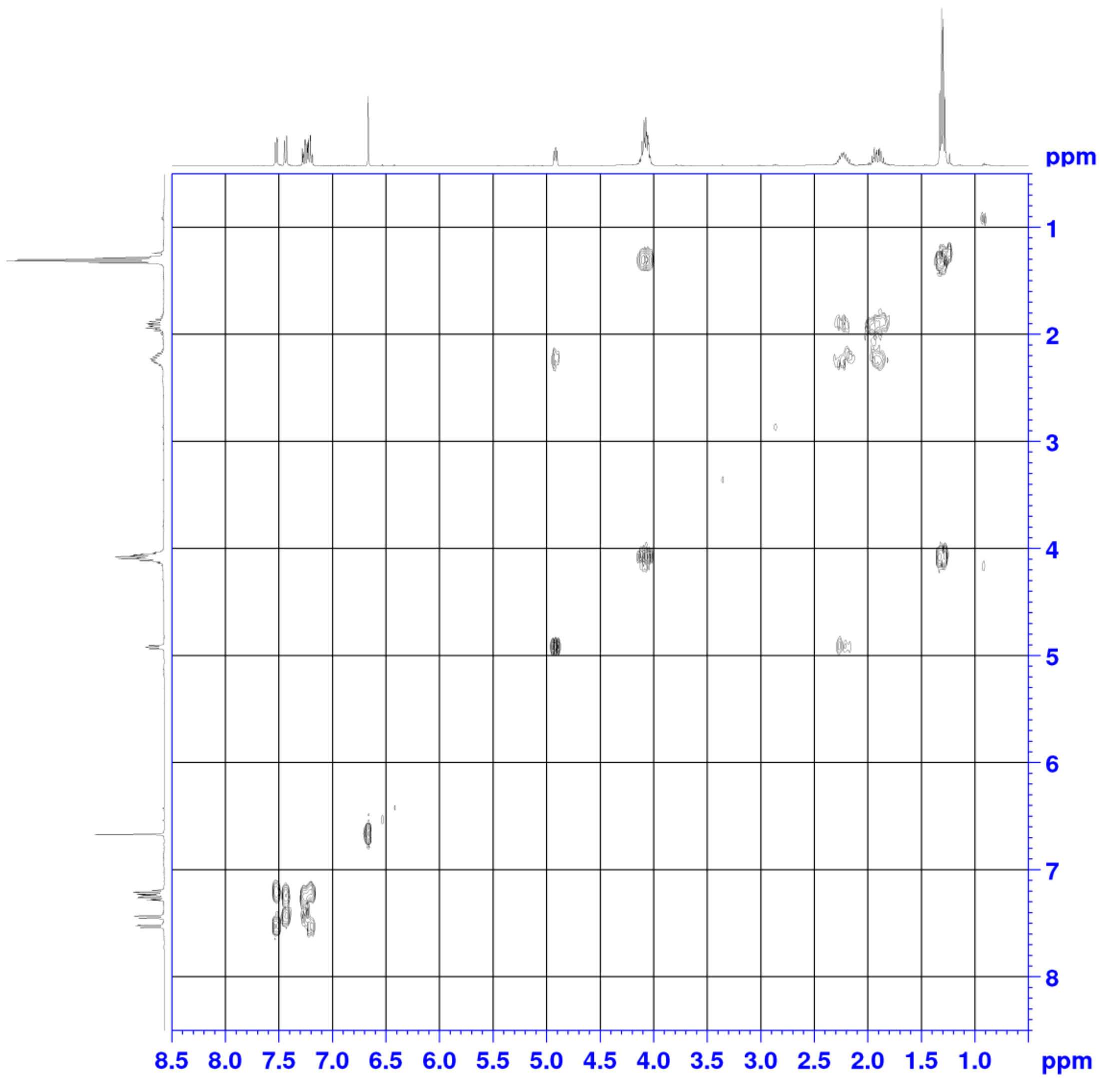
9t: ^{13}C NMR


159.13
154.92
128.32
124.16
122.89
121.15
111.32
102.98
77.55
77.23
76.91
67.86
67.71
62.05
62.00
61.98
28.73
28.69
22.27
20.85
16.57
16.52
O
EtO
P
EtO
O
OH
9t
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

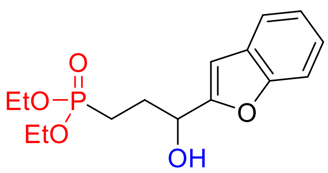
9t: ^{13}C DEPT135



9t: COSY



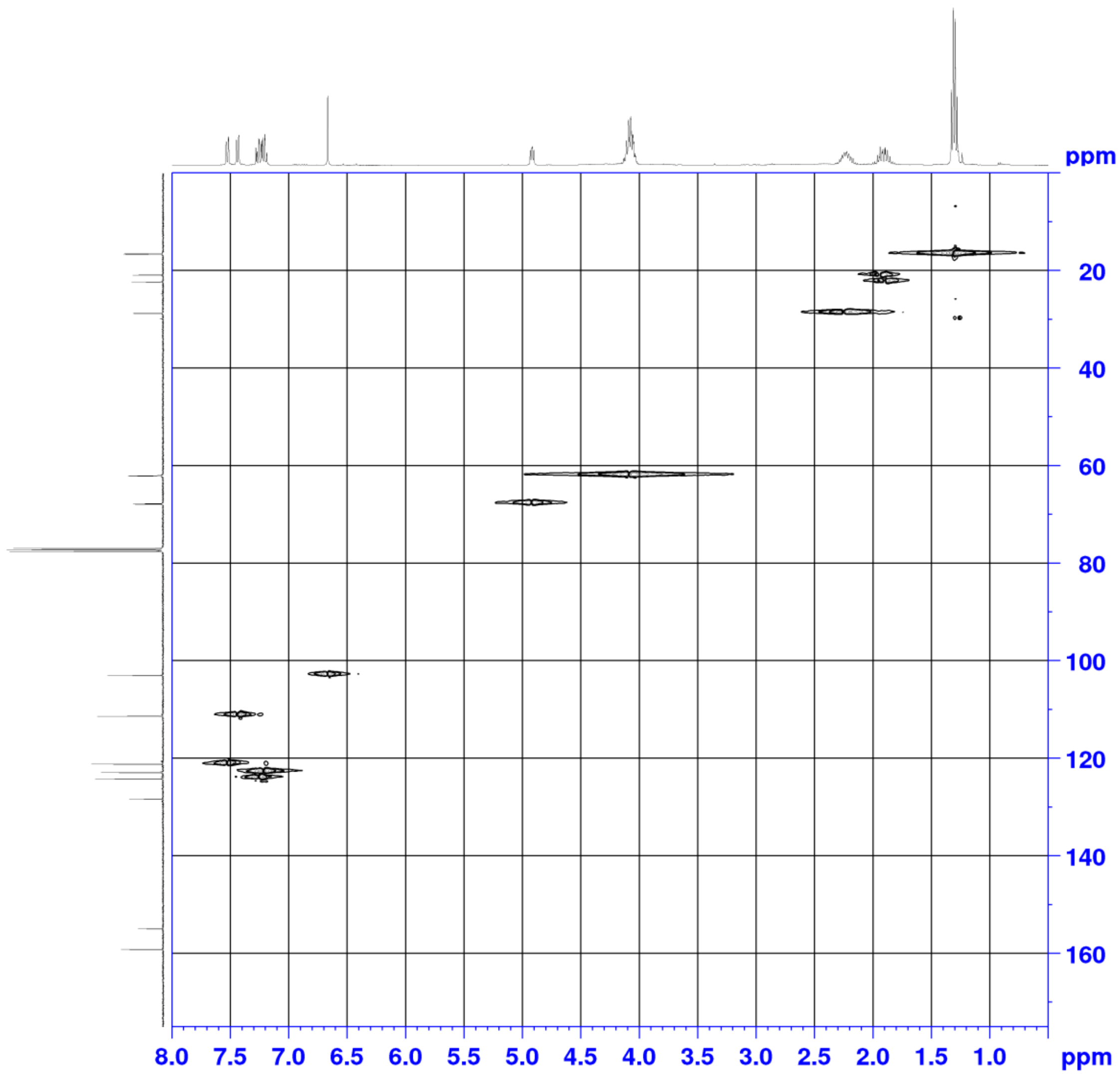
ppm
1
2
3
4
5
6
7
8
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm

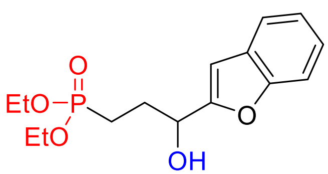


O
EtO
P
EtO
OH
O

9t

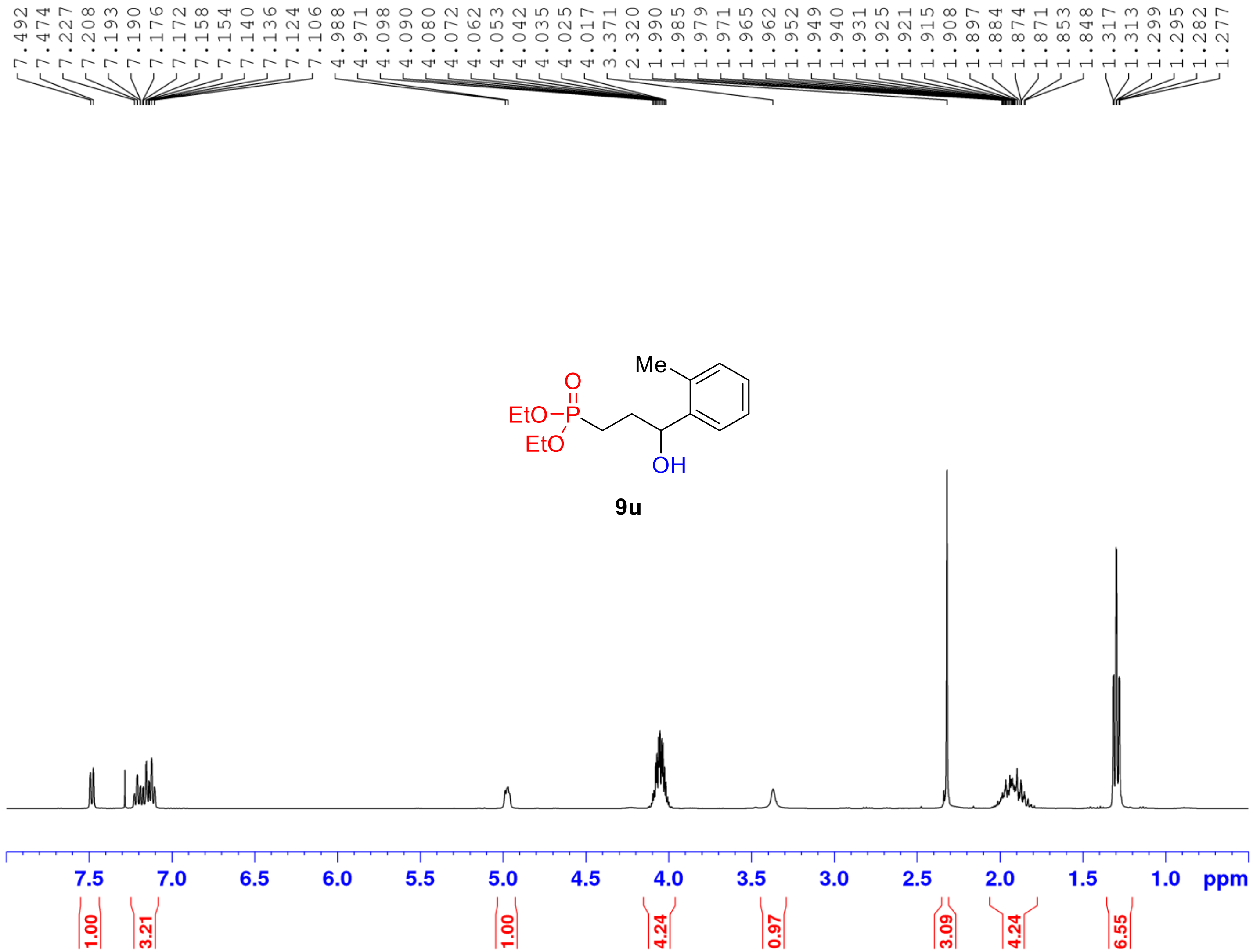
9t: ^{13}C HSQC



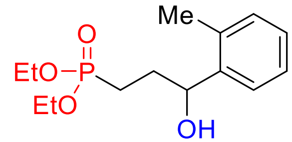


9t

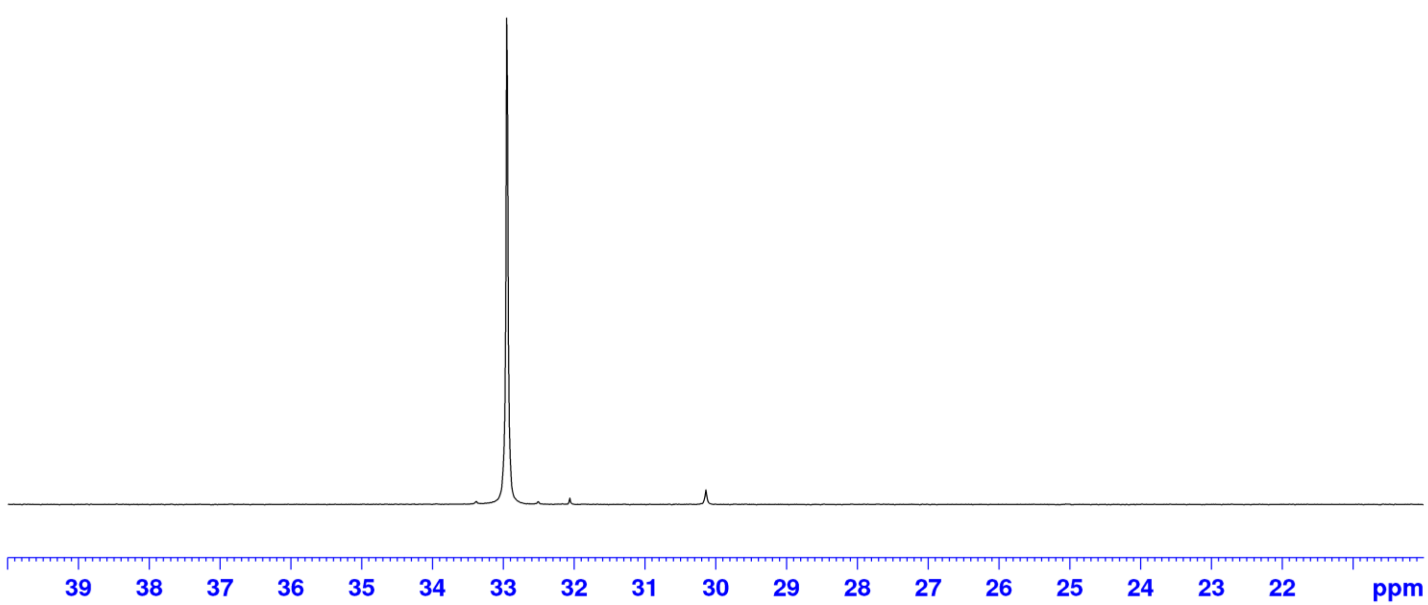
9u: ^{1}H NMR


7.492
7.474
7.227
7.208
7.193
7.190
7.176
7.172
7.158
7.154
7.140
7.136
7.124
7.106
4.988
4.971
4.098
4.090
4.080
4.072
4.062
4.053
4.042
4.035
4.025
4.017
3.371
2.320
1.990
1.985
1.979
1.971
1.965
1.962
1.952
1.949
1.940
1.931
1.925
1.921
1.915
1.908
1.897
1.884
1.874
1.871
1.853
1.848
1.317
1.313
1.299
1.295
1.282
1.277
Me
O
EtO
P
EtO
OH
9u
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
1.00
3.21
1.00
4.24
0.97
3.09
4.24
6.55

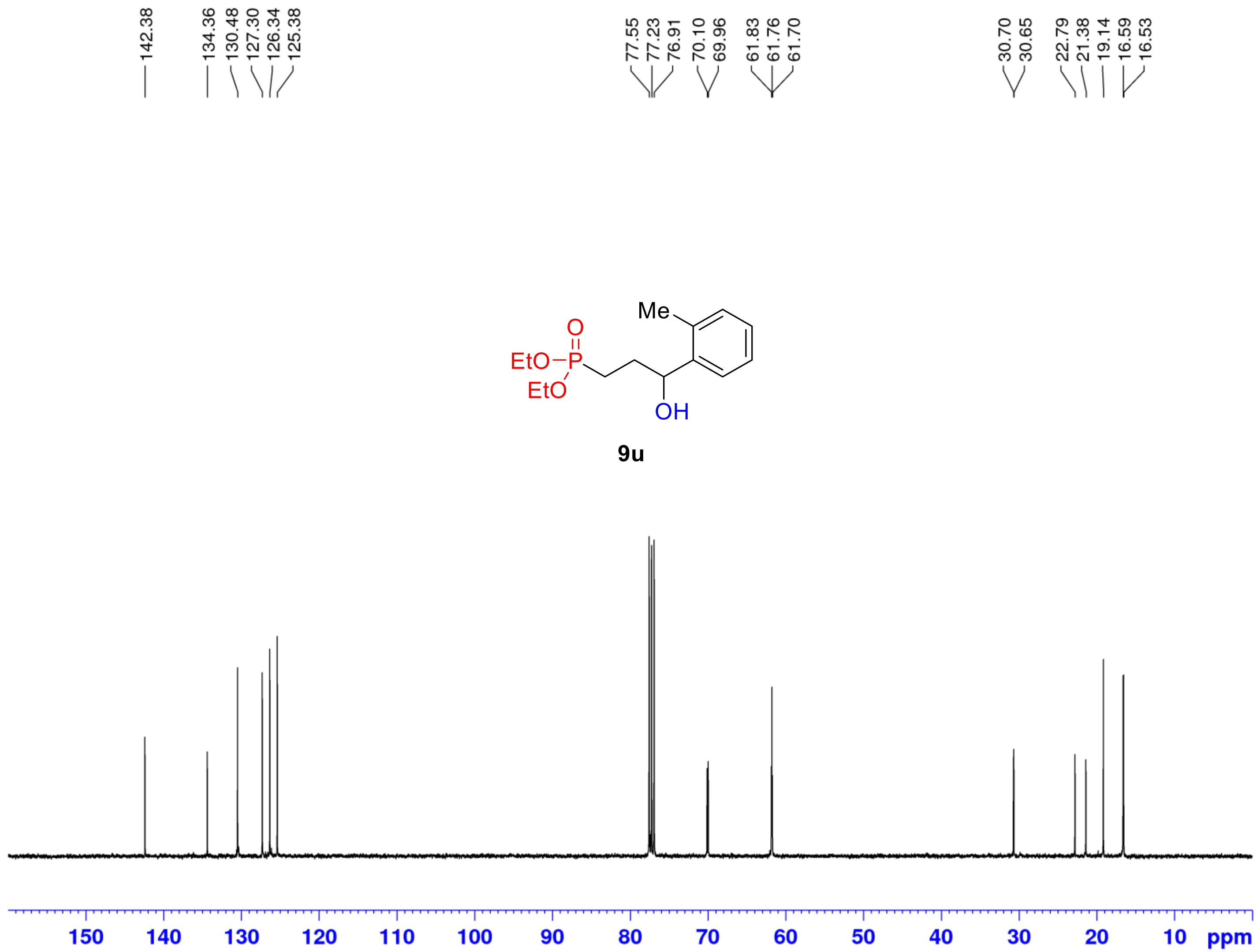
9u: ^{31}P NMR



9u

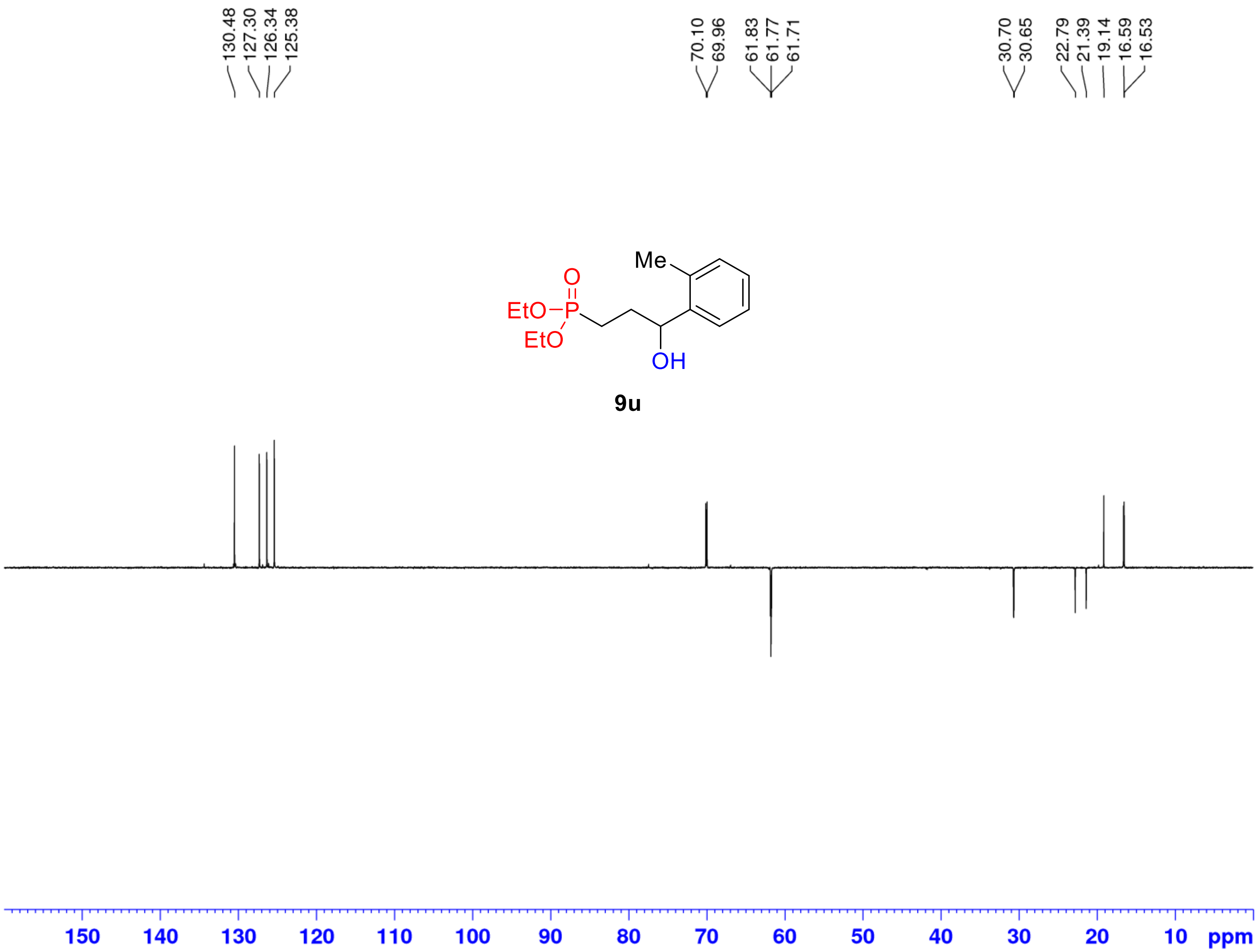


9u: ^{13}C NMR

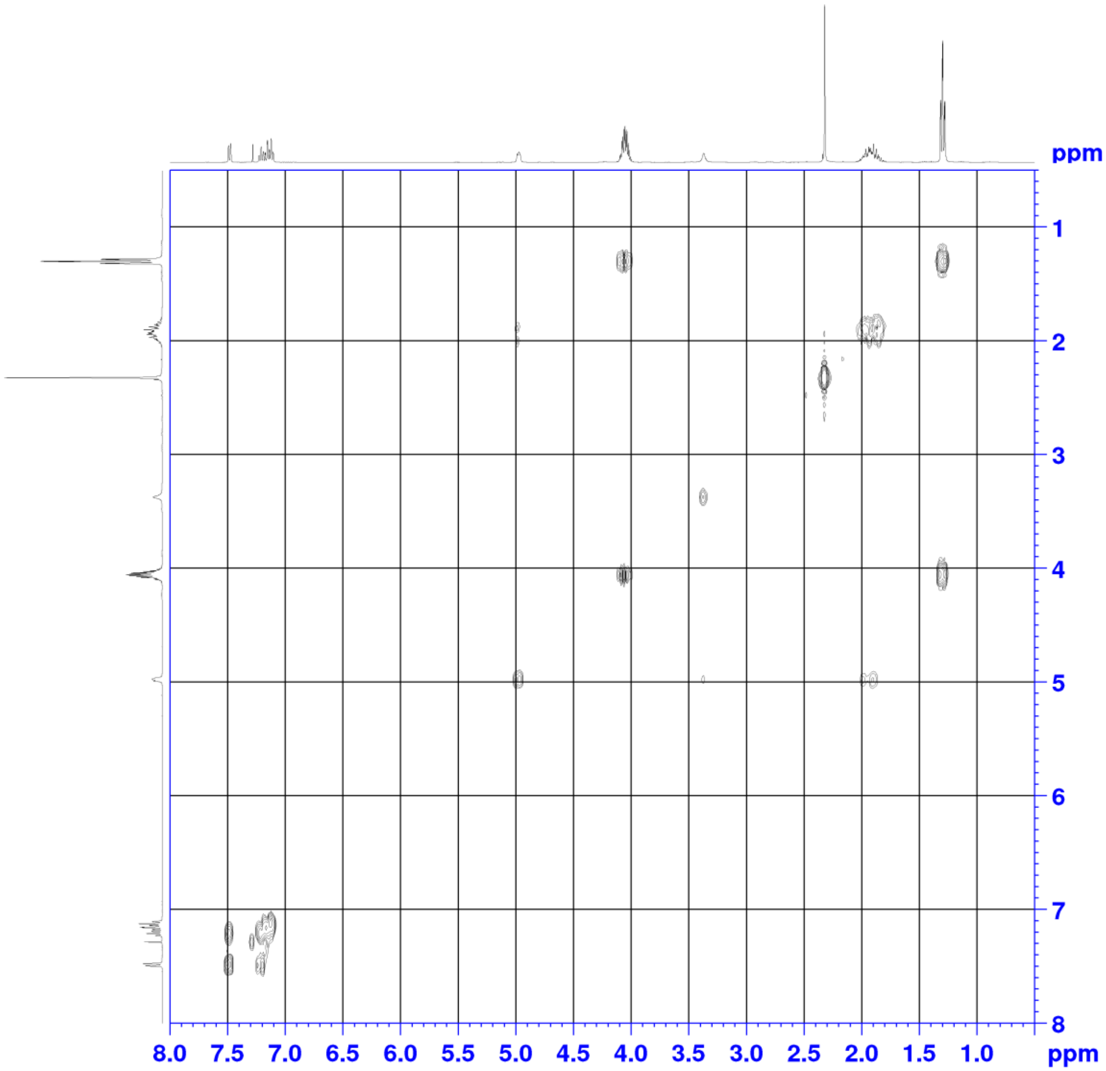


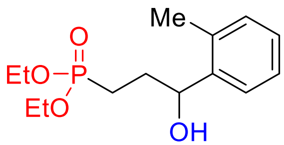
142.38
134.36
130.48
127.30
126.34
125.38
77.55
77.23
76.91
70.10
69.96
61.83
61.76
61.70
30.70
30.65
22.79
21.38
19.14
16.59
16.53
Me
O
EtO
P
EtO
OH
9u
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

9u: ^{13}C DEPT135



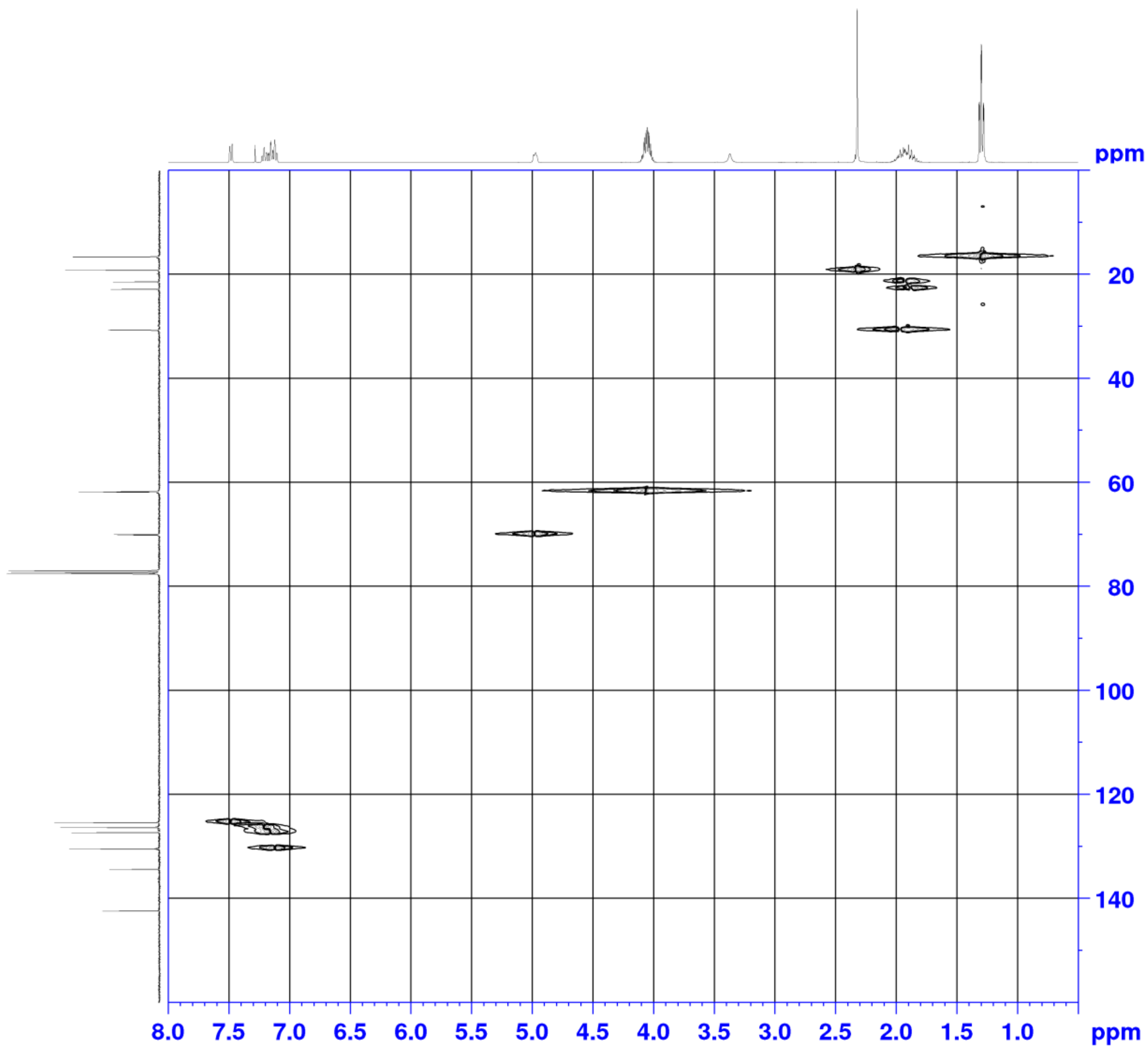
9u: COSY

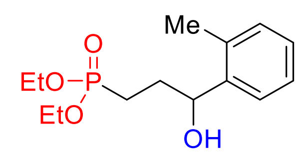

ppm
1
2
3
4
5
6
7
8
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0
ppm


Me
O
EtO
P
EtO
OH

9u

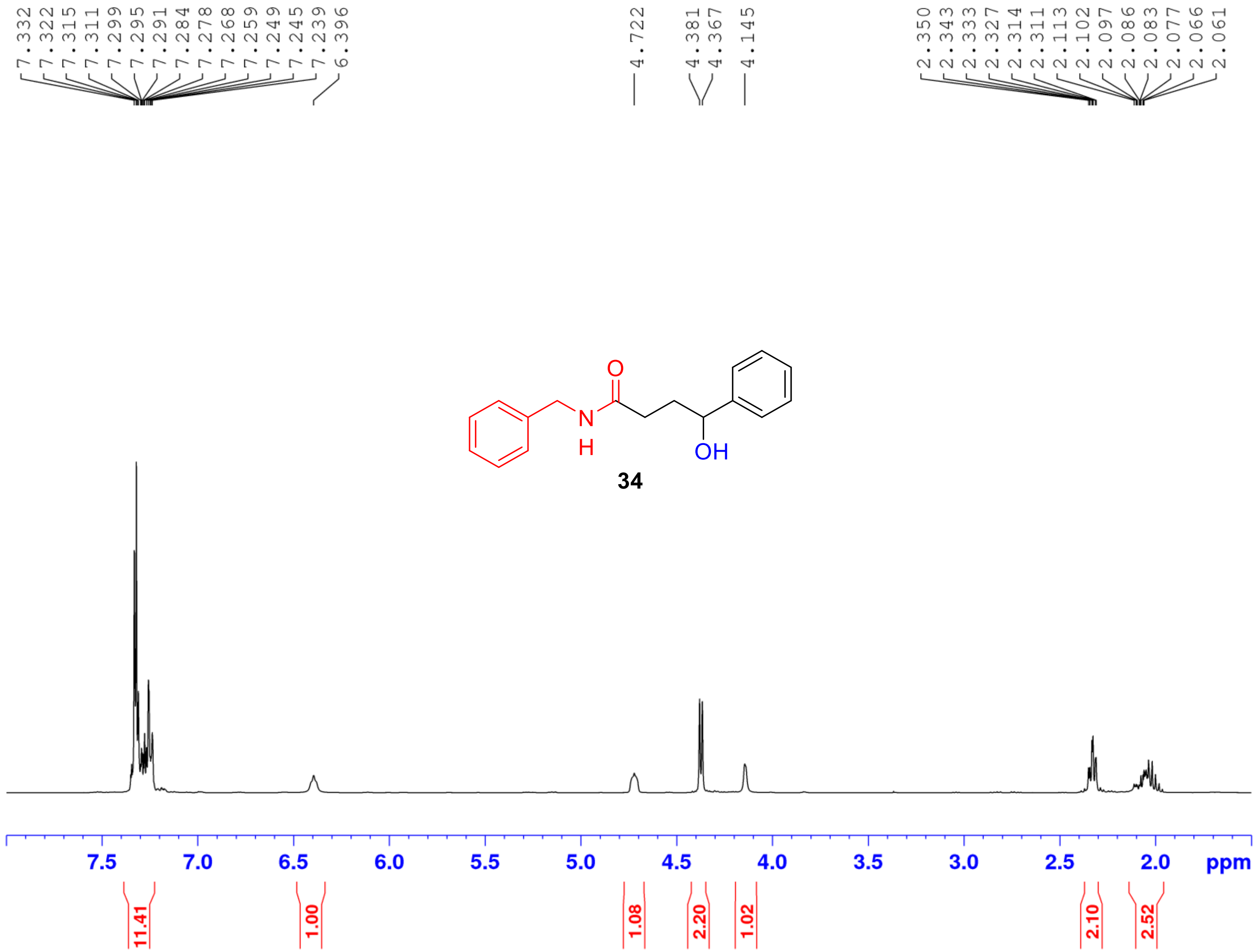
9u: ^{13}C HSQC



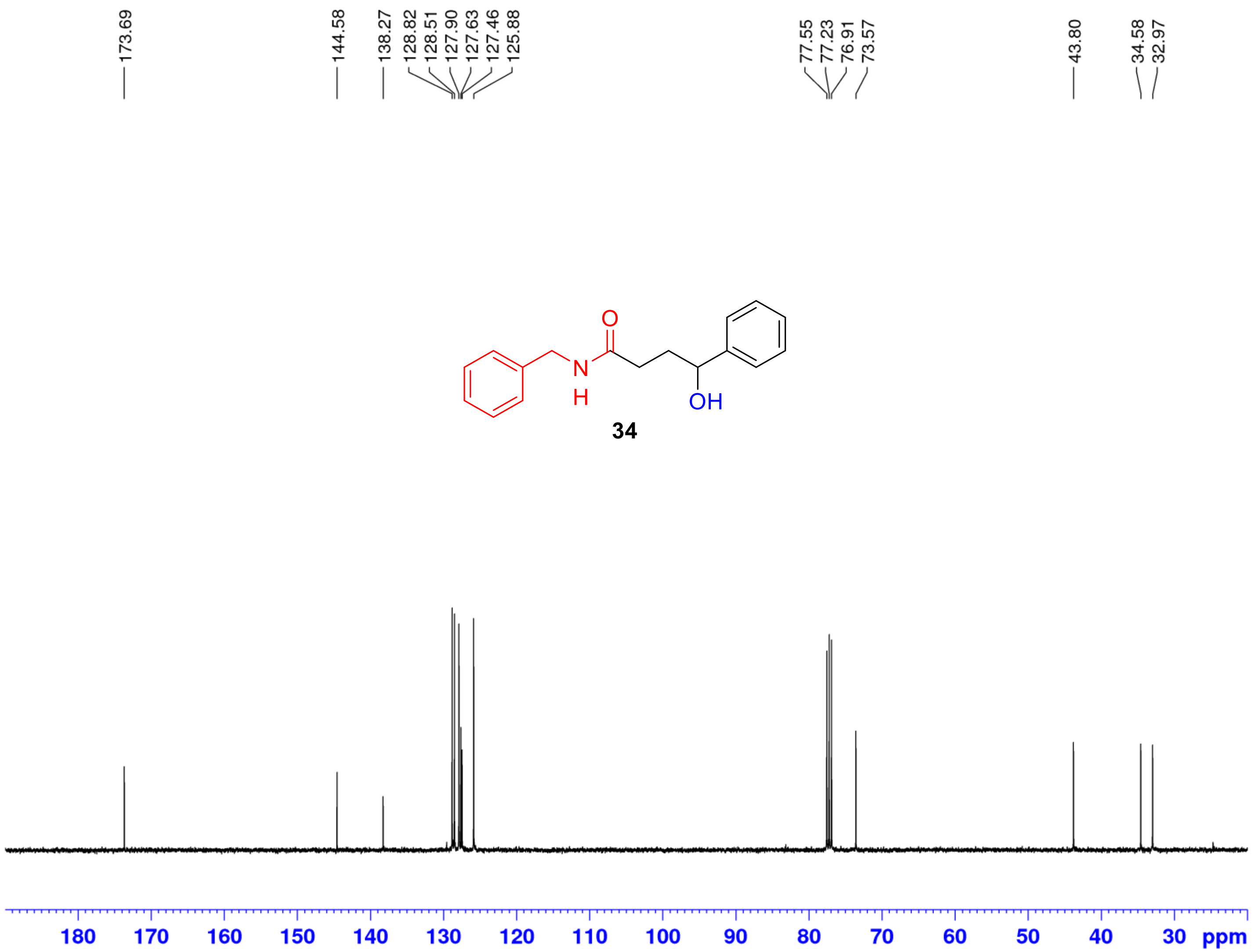


9u

34: ^{1}H NMR

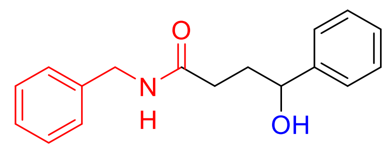

7.332
7.322
7.315
7.311
7.299
7.295
7.291
7.284
7.278
7.268
7.259
7.249
7.245
7.239
6.396
4.722
4.381
4.367
4.145
2.350
2.343
2.333
2.327
2.314
2.311
2.113
2.102
2.097
2.086
2.083
2.077
2.066
2.061
O
N
H
OH
34
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
ppm
11.41
1.00
1.08
2.20
1.02
2.10
2.52

34: ^{13}C NMR

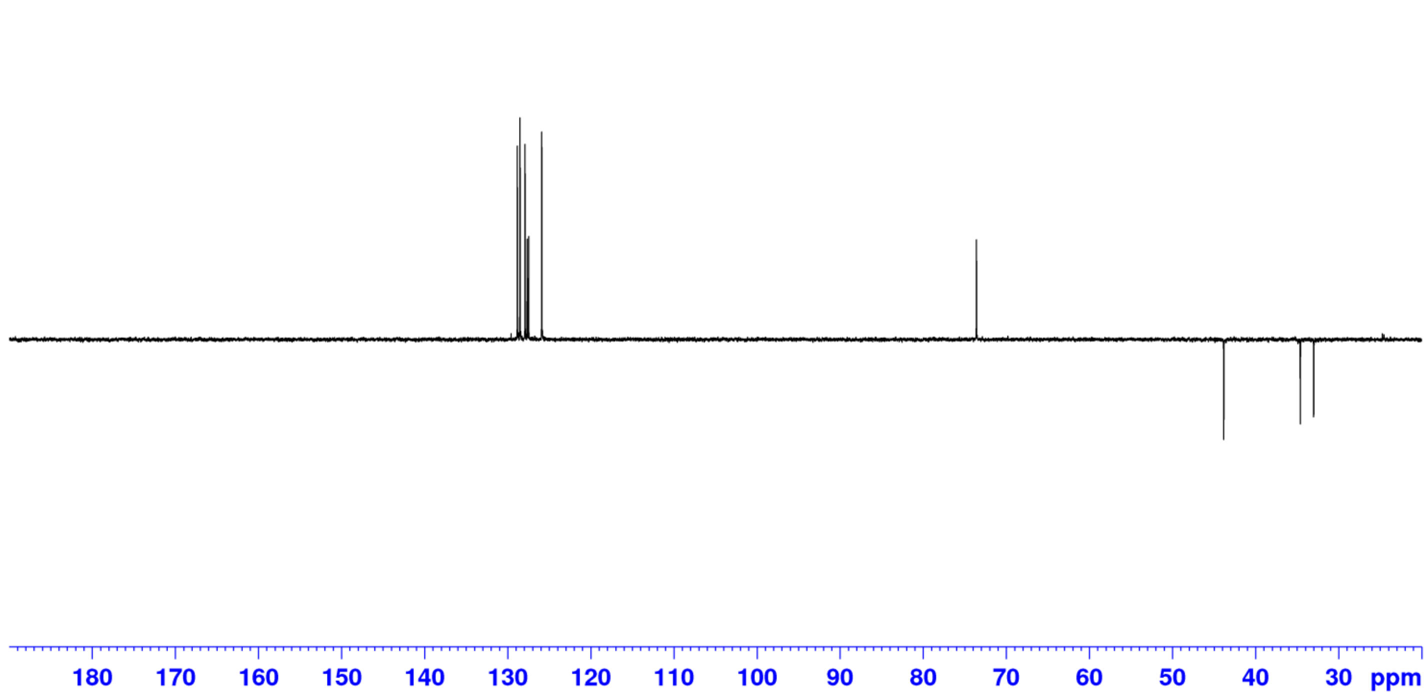


173.69
144.58
138.27
128.82
128.51
127.90
127.63
127.46
125.88
77.55
77.23
76.91
73.57
43.80
34.58
32.97
O
N
H
OH
34
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
ppm

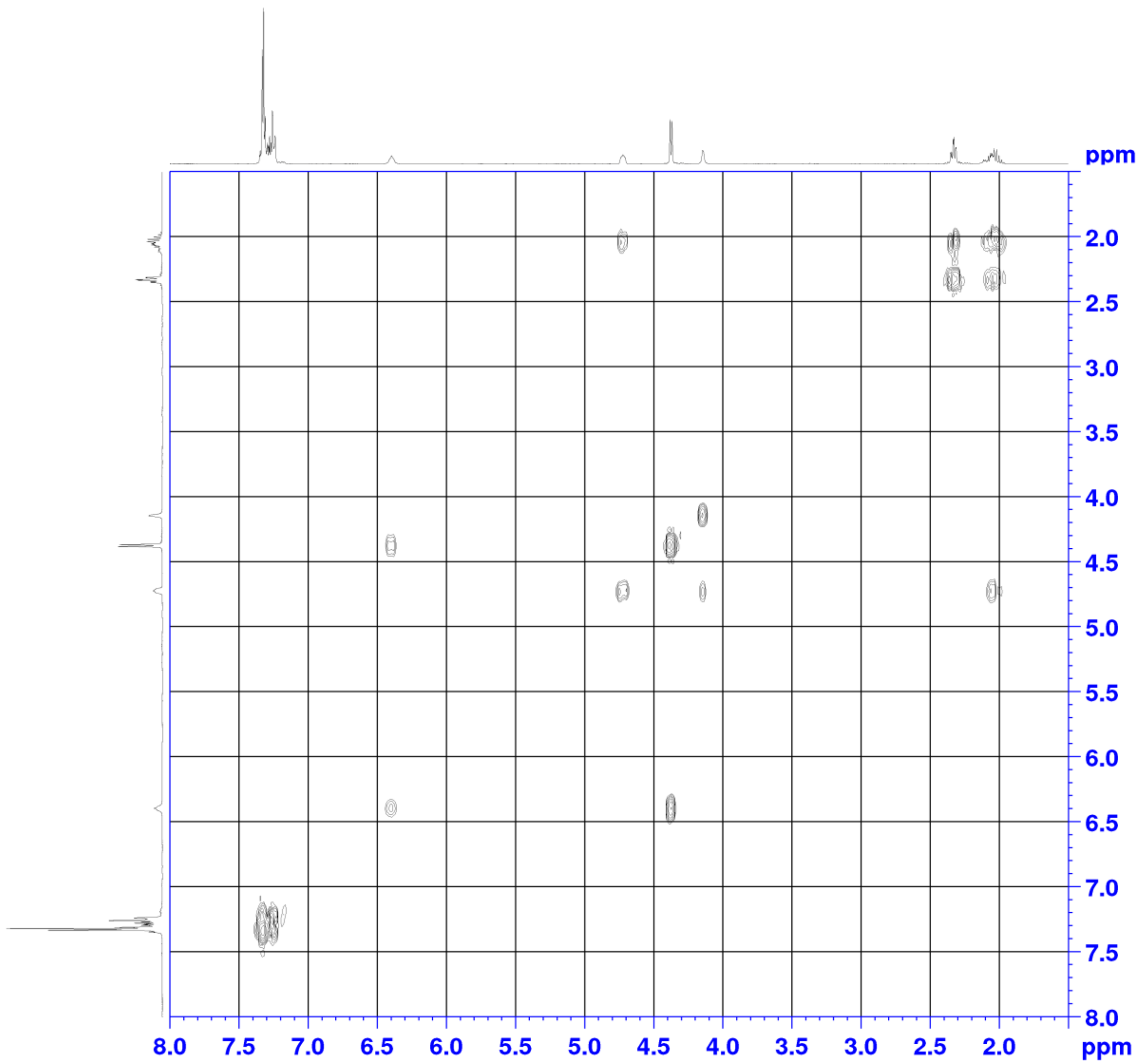
34: ^{13}C DEPT135

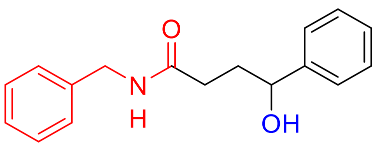


34



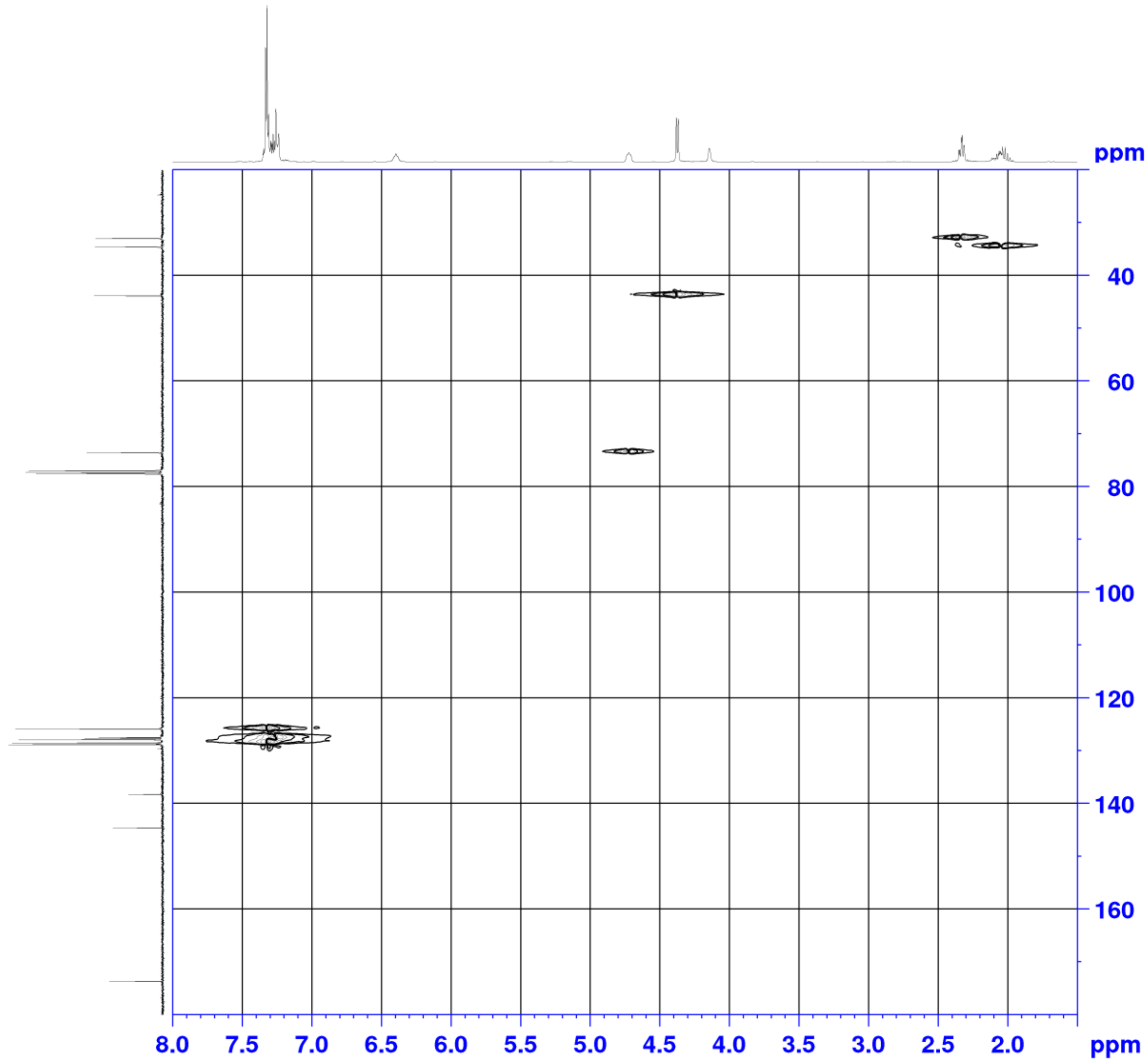
34: COSY

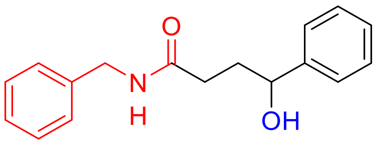

ppm
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0
ppm


O
N
H
OH

34

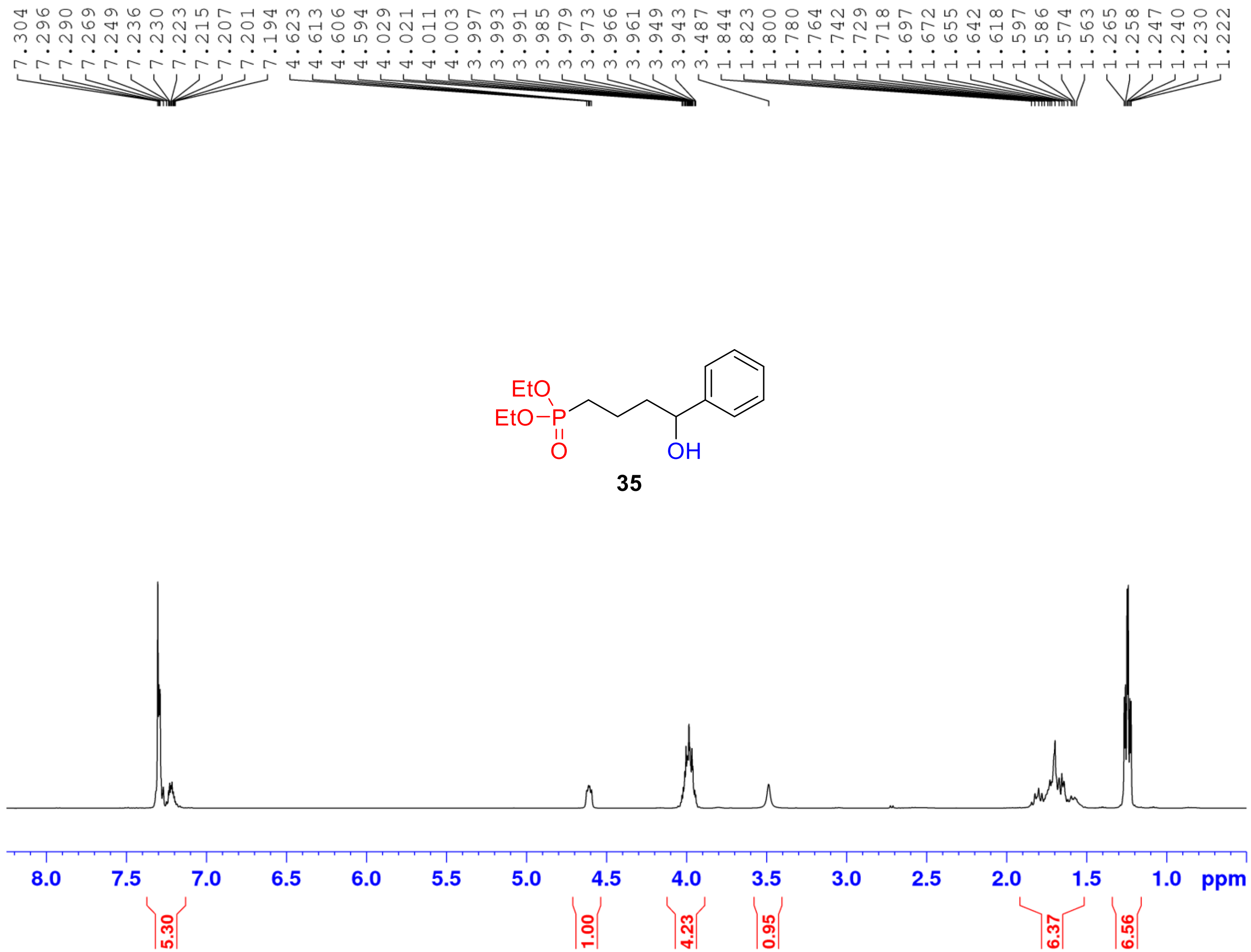
34: ^{13}C HSQC





34

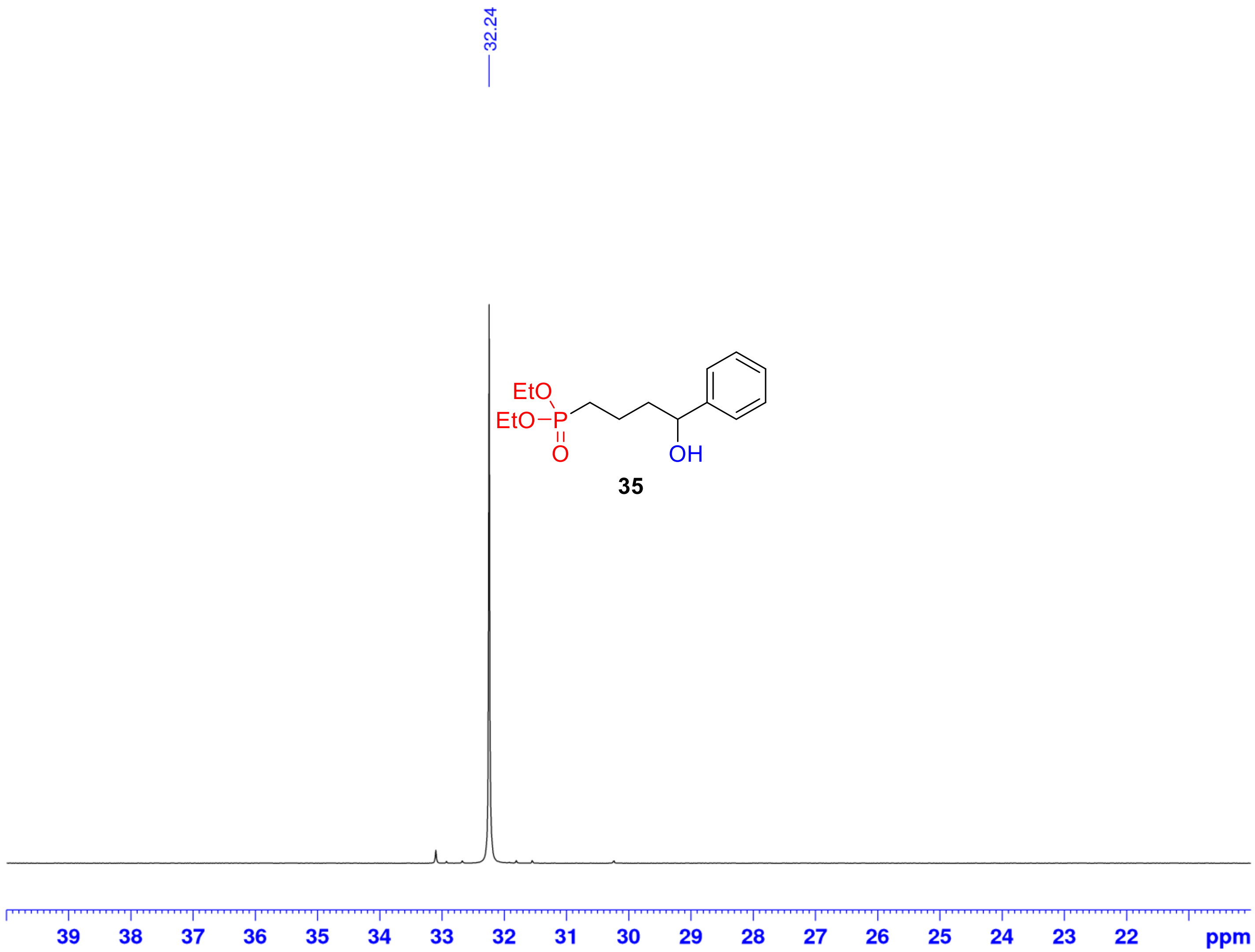
35: ^{1}H NMR



7.304
7.296
7.290
7.269
7.249
7.236
7.230
7.223
7.215
7.207
7.201
7.194
4.623
4.613
4.606
4.594
4.029
4.021
4.011
4.003
3.997
3.993
3.991
3.985
3.979
3.973
3.966
3.961
3.949
3.943
3.487
1.844
1.823
1.800
1.780
1.764
1.742
1.729
1.718
1.697
1.672
1.655
1.642
1.618
1.597
1.586
1.574
1.563
1.265
1.258
1.247
1.240
1.230
1.222
EtO
EtO
P
O
OH
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
ppm
5.30
1.00
4.23
0.95
6.37
6.56

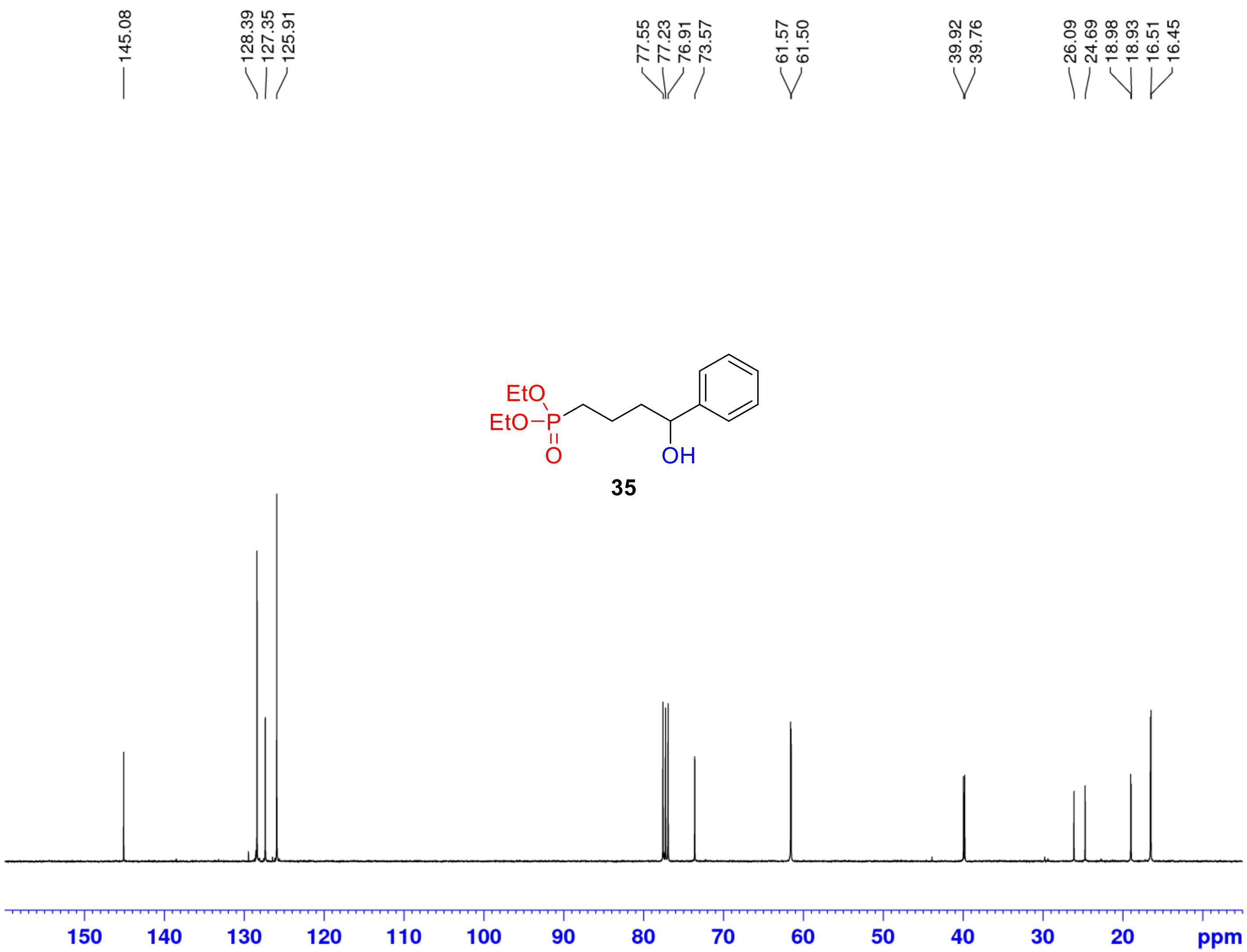
35

35: ^{31}P NMR



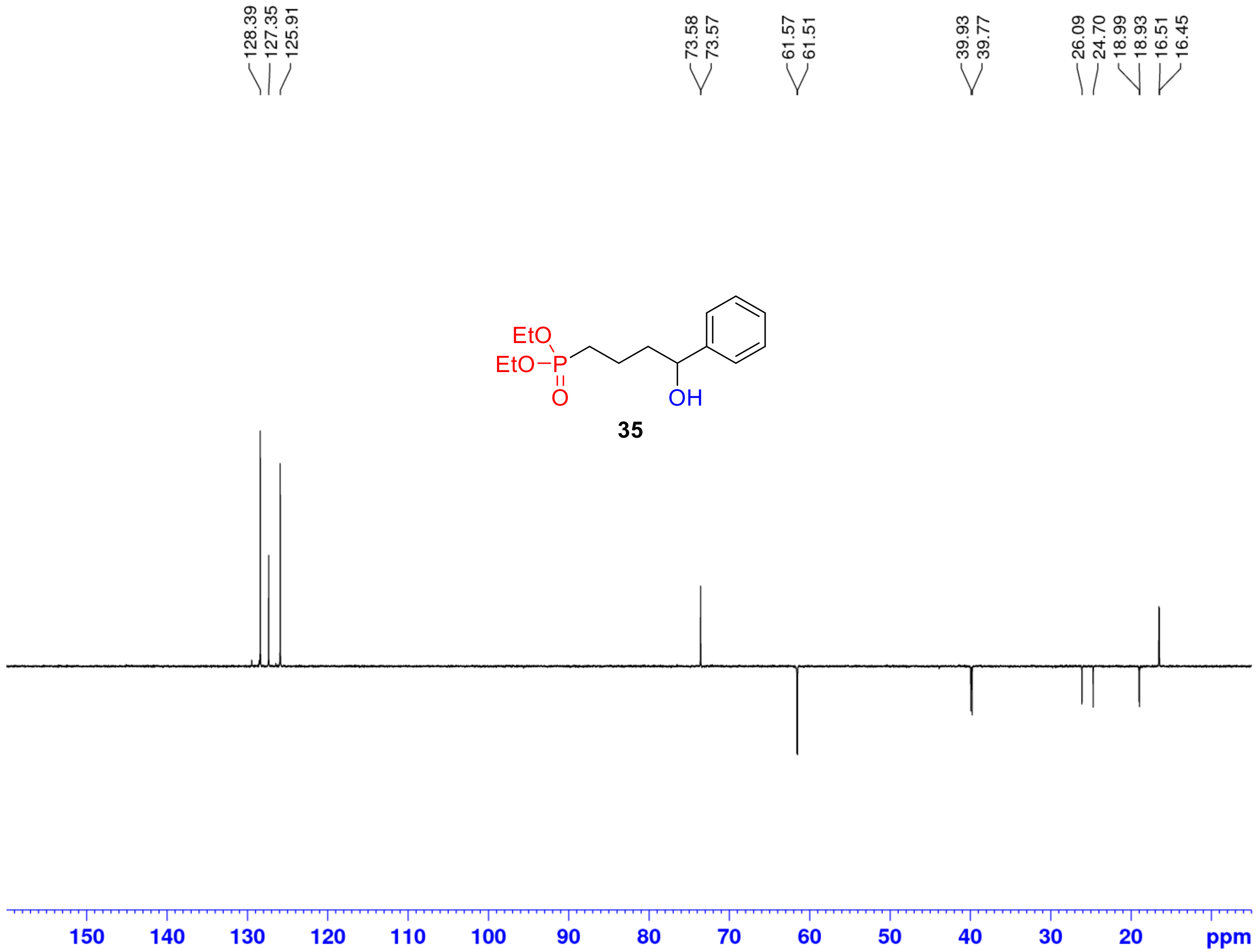
32.24
EtO
EtO
P
O
OH
35
39
38
37
36
35
34
33
32
31
30
29
28
27
26
25
24
23
22
ppm

35: ^{13}C NMR

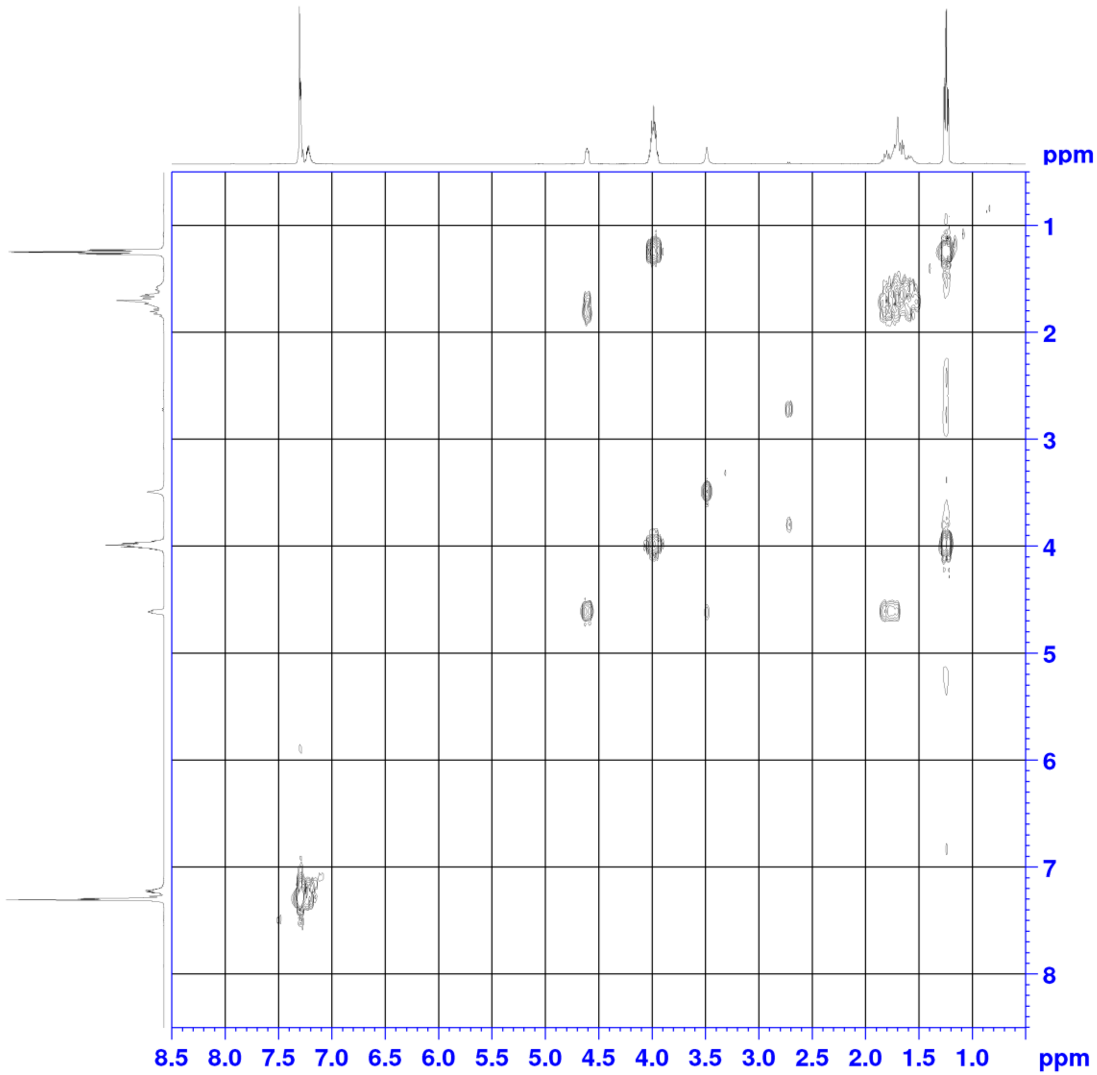


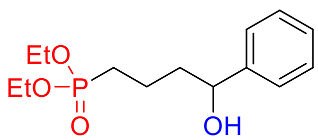
145.08
128.39
127.35
125.91
77.55
77.23
76.91
73.57
61.57
61.50
39.92
39.76
26.09
24.69
18.98
18.93
16.51
16.45
EtO
EtO
P
O
OH
35
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

35: ^{13}C DEPT135



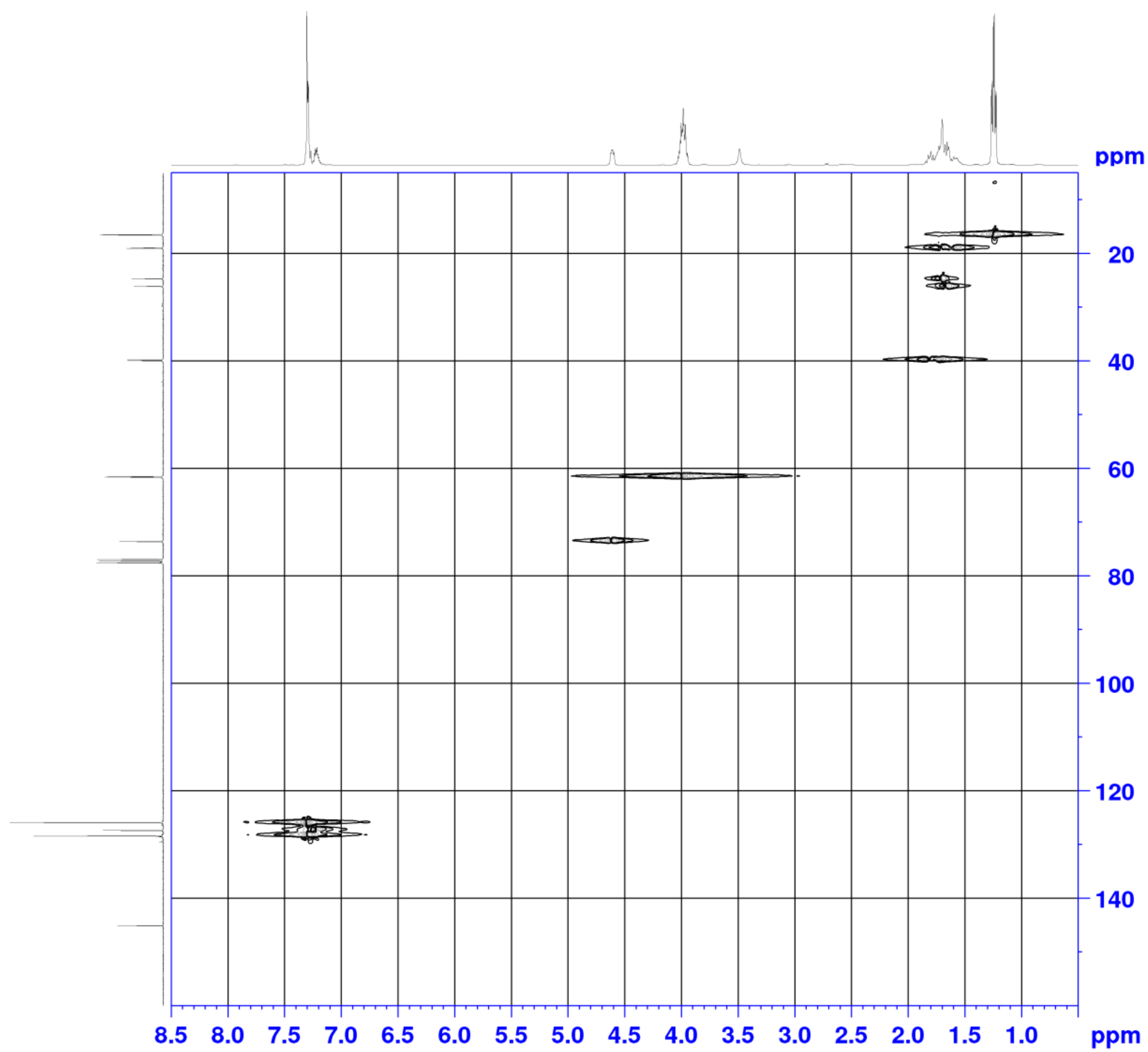
35: COSY

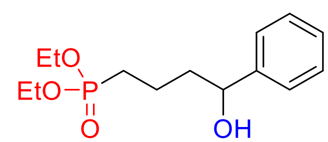

ppm
1
2
3
4
5
6
7
8
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0
ppm


EtO
EtO
P
O
OH

35

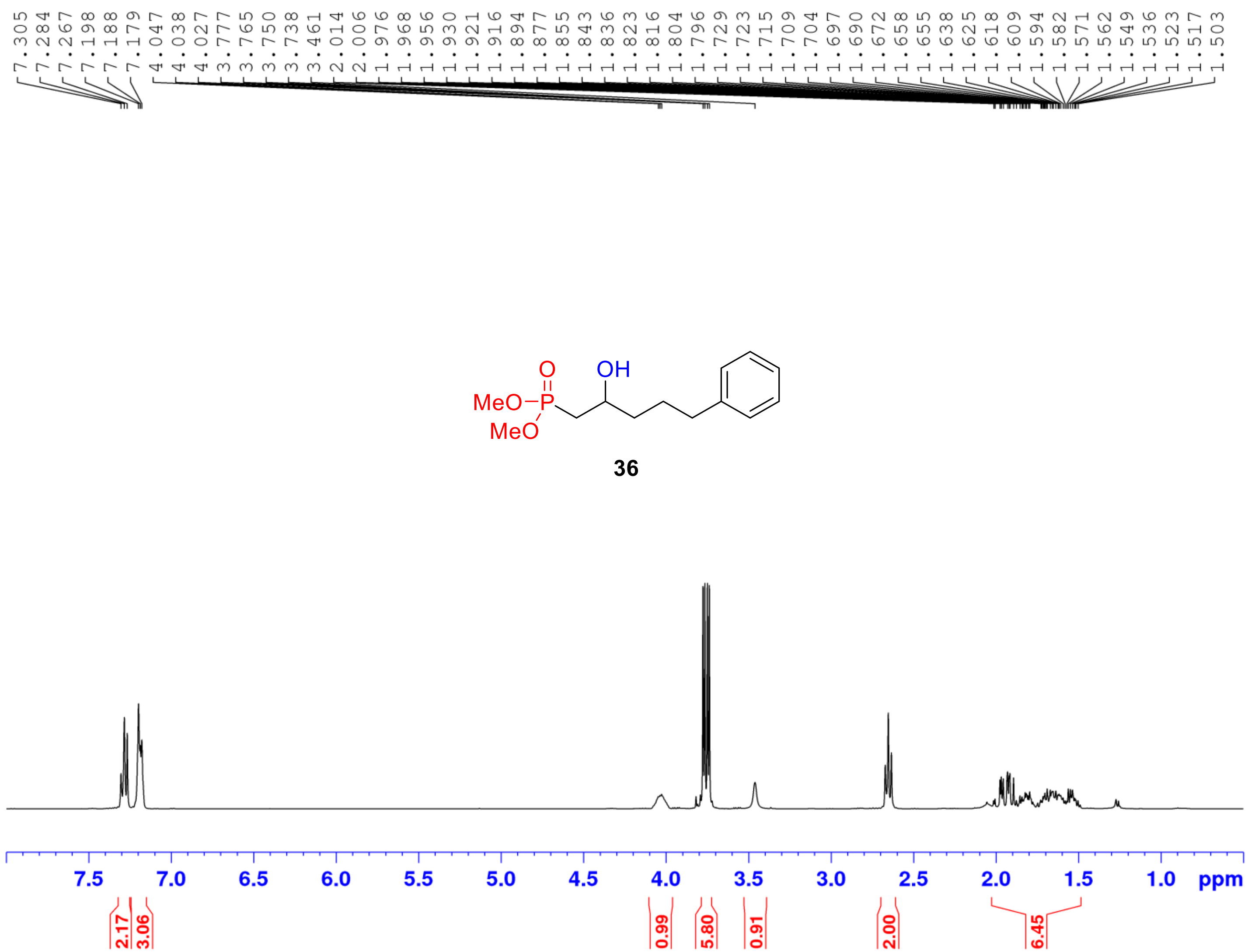
35: ^{13}C HSQC





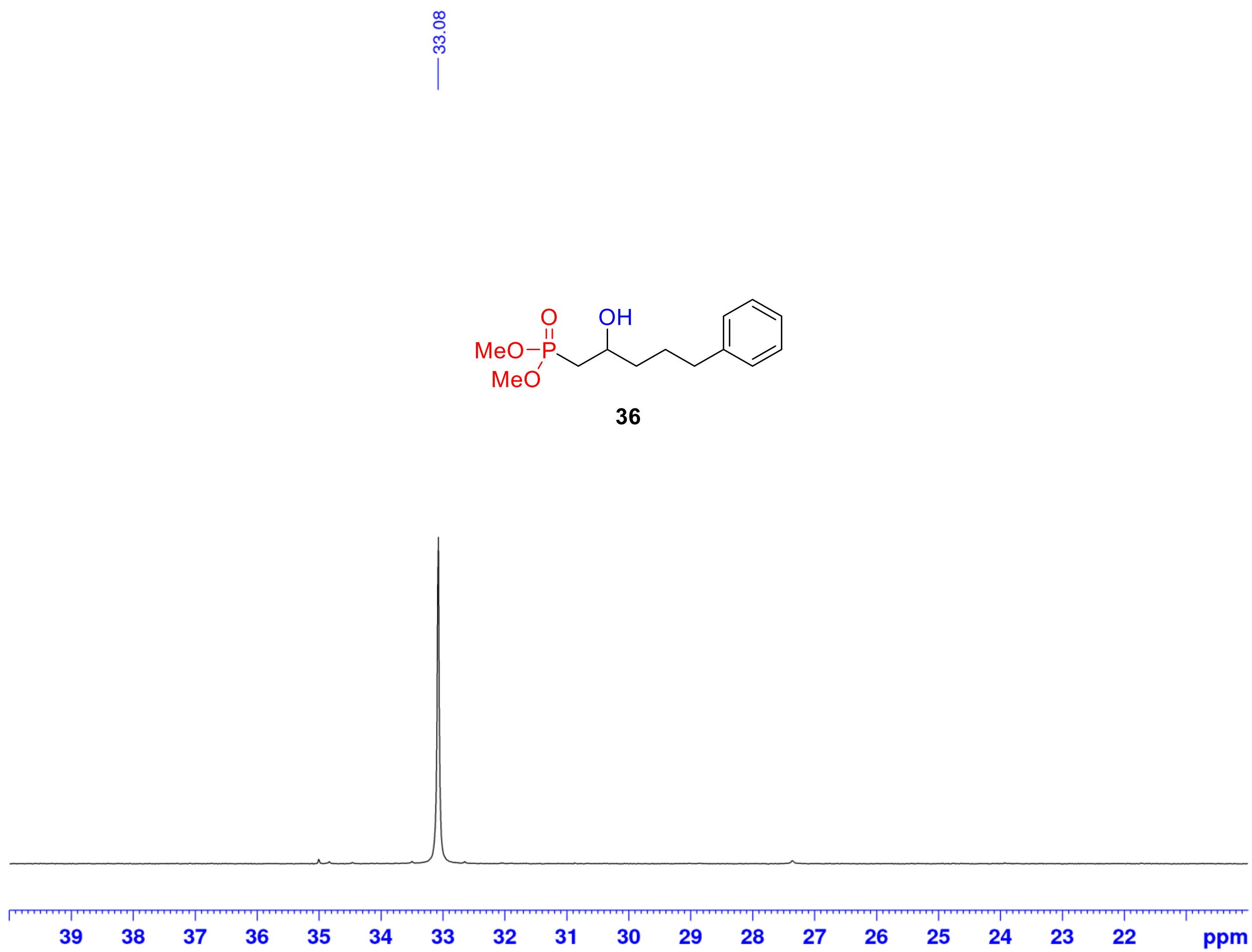
35

36: ^{1}H NMR

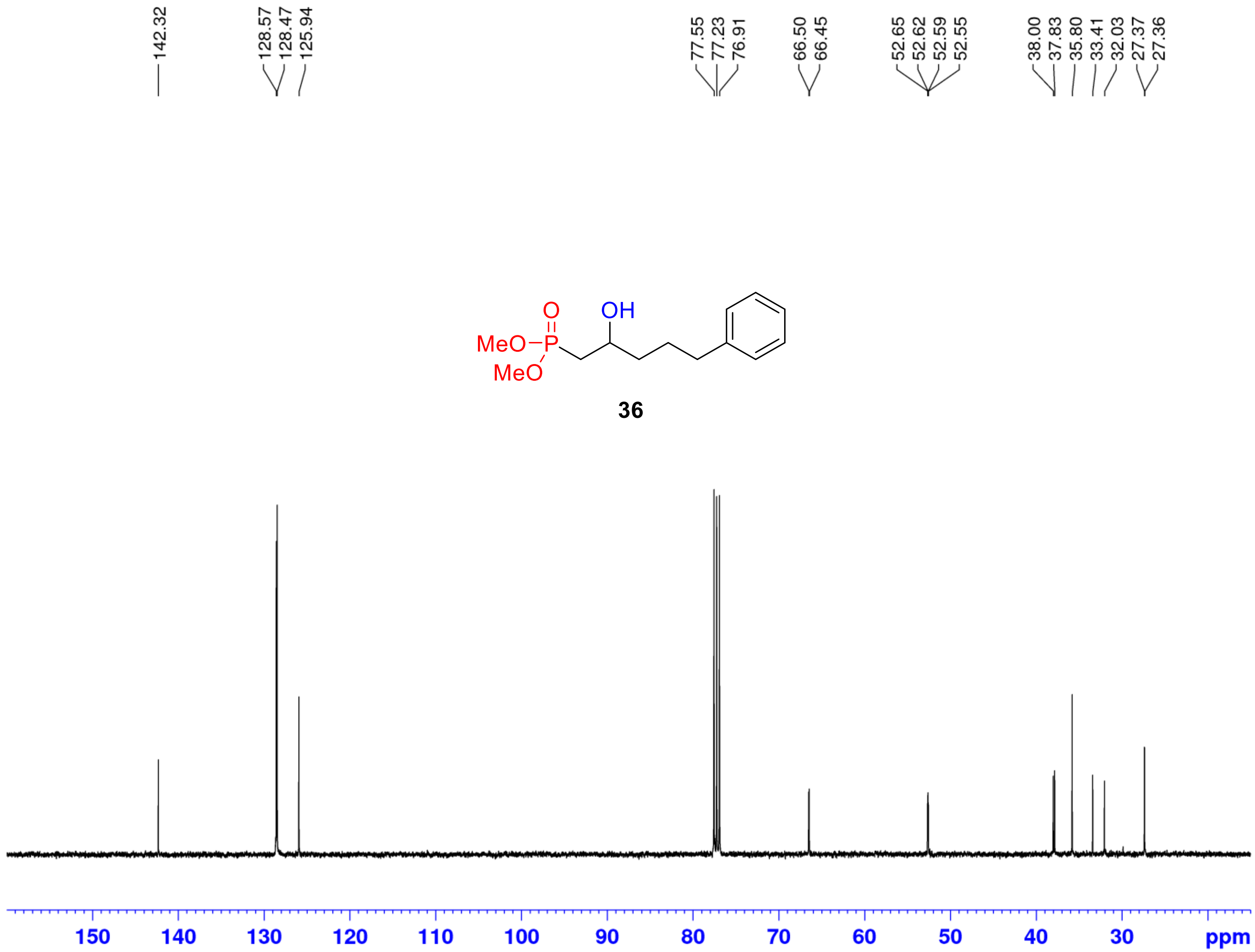


7.305
7.284
7.267
7.198
7.188
7.179
4.047
4.038
4.027
3.777
3.765
3.750
3.738
3.461
2.014
2.006
1.976
1.968
1.956
1.930
1.921
1.916
1.894
1.877
1.855
1.843
1.836
1.823
1.816
1.804
1.796
1.729
1.723
1.715
1.709
1.704
1.697
1.690
1.672
1.658
1.655
1.638
1.625
1.618
1.609
1.594
1.582
1.571
1.562
1.549
1.536
1.523
1.517
1.503
O
OH
MeO
P
MeO
36
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
2.17
3.06
0.99
5.80
0.91
2.00
6.45

36: ^{31}P NMR

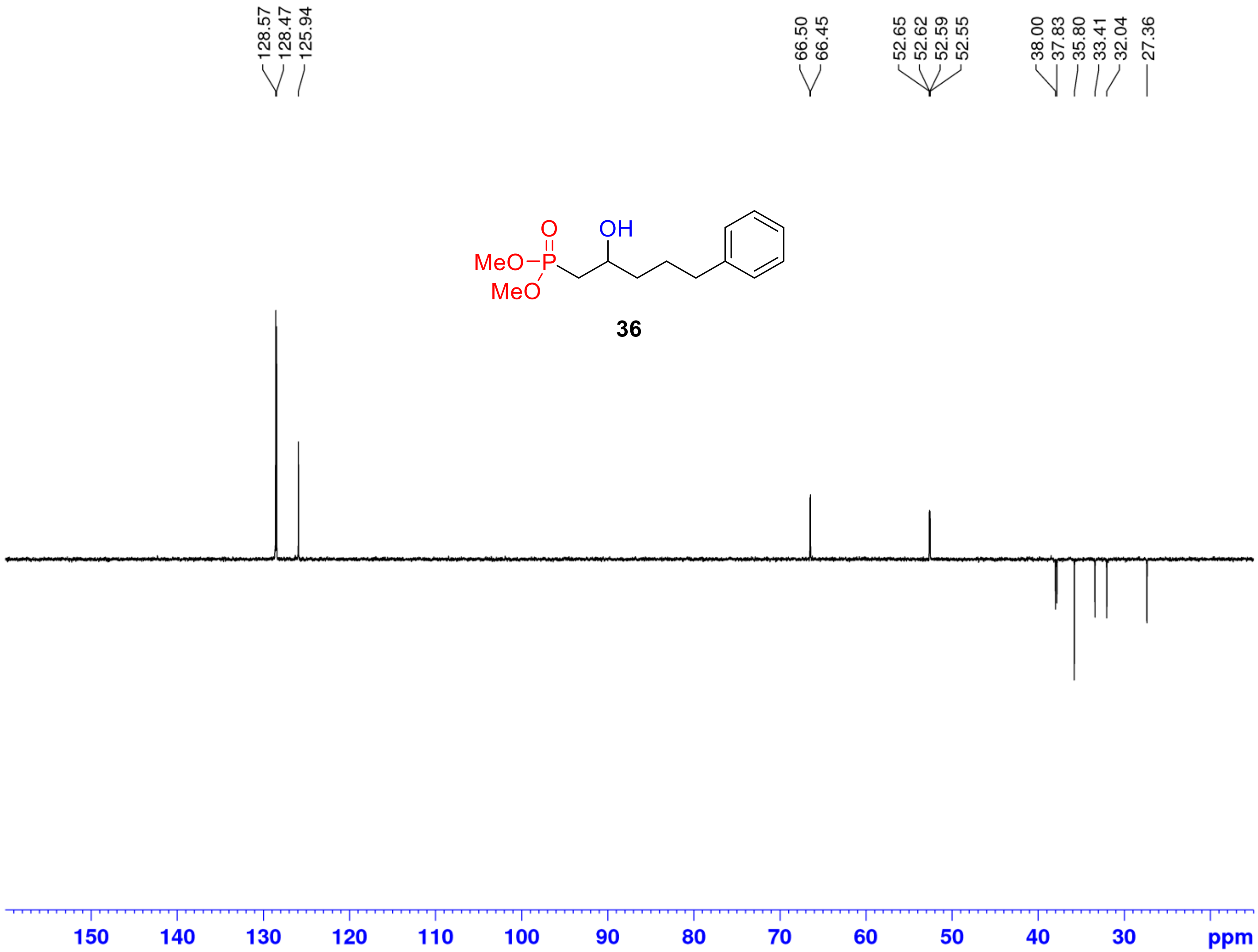

33.08
O
OH
MeO
P
MeO
36
39
38
37
36
35
34
33
32
31
30
29
28
27
26
25
24
23
22
ppm

36: ^{13}C NMR

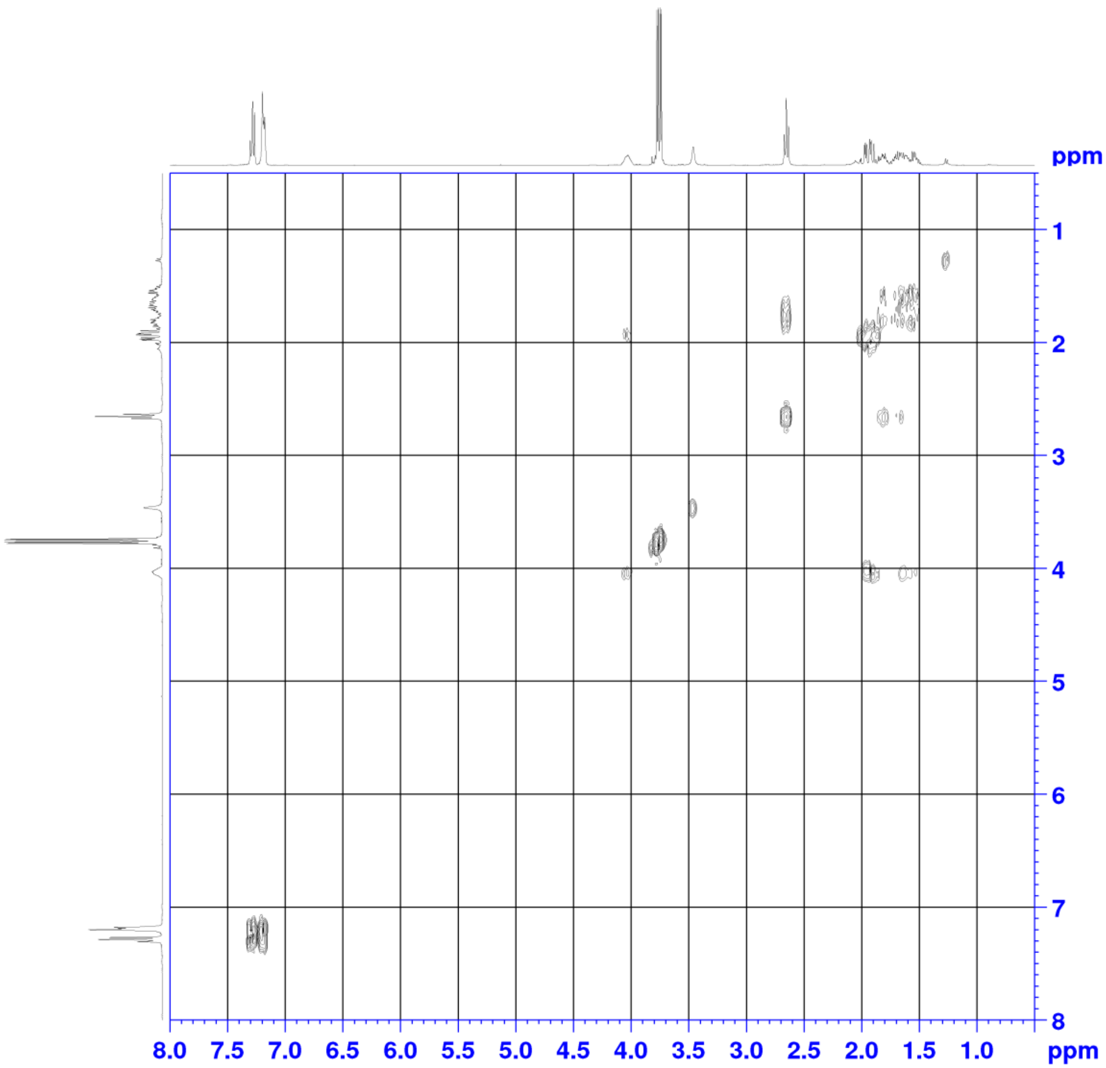


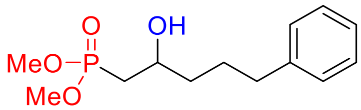
142.32
128.57
128.47
125.94
77.55
77.23
76.91
66.50
66.45
52.65
52.62
52.59
52.55
38.00
37.83
35.80
33.41
32.03
27.37
27.36
O
OH
MeO
P
MeO
36
150
140
130
120
110
100
90
80
70
60
50
40
30
ppm

36: ^{13}C DEPT135



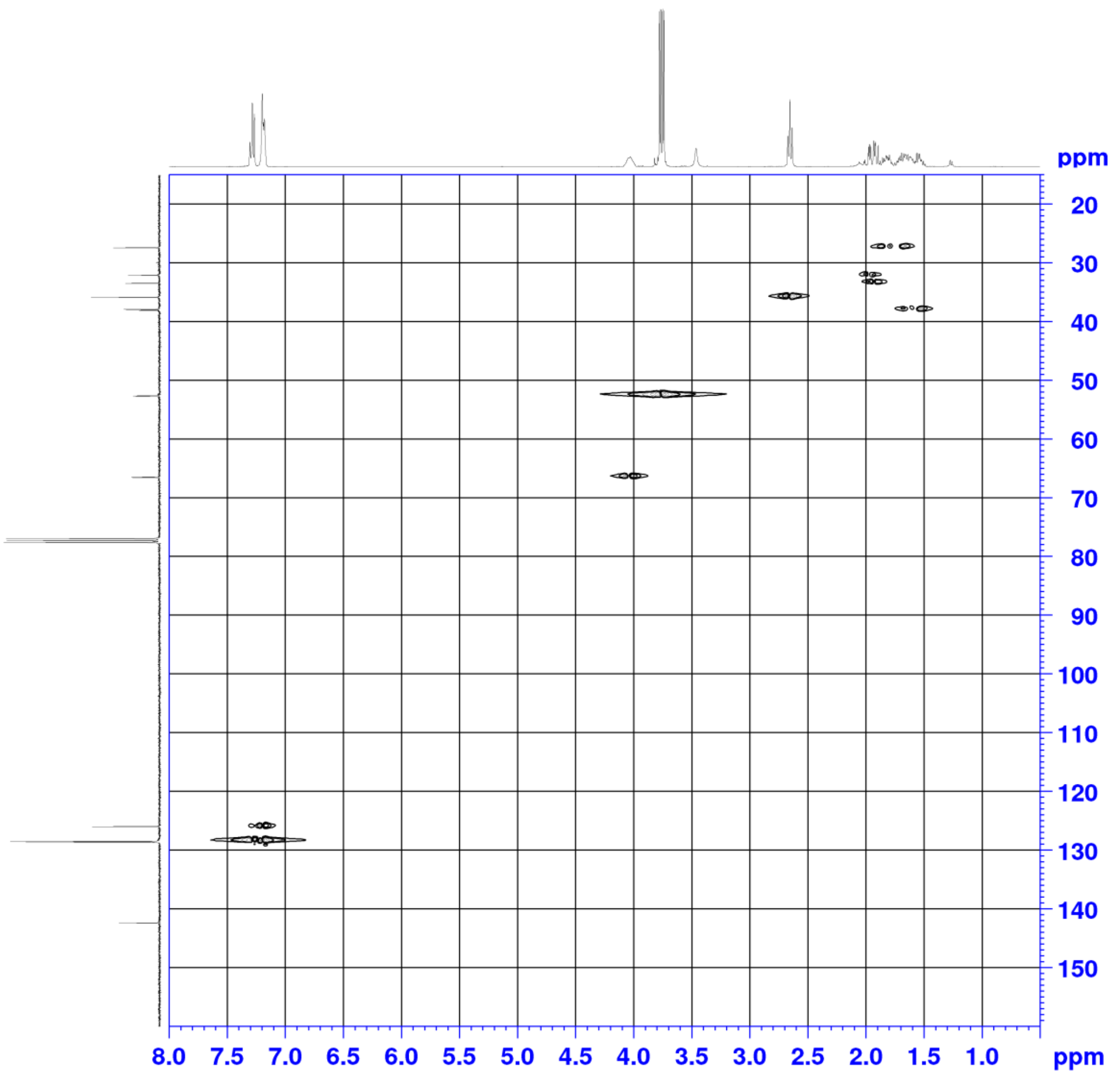
36: COSY

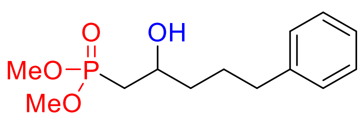

ppm
1
2
3
4
5
6
7
8
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm


O
OH
MeO
P
MeO

36

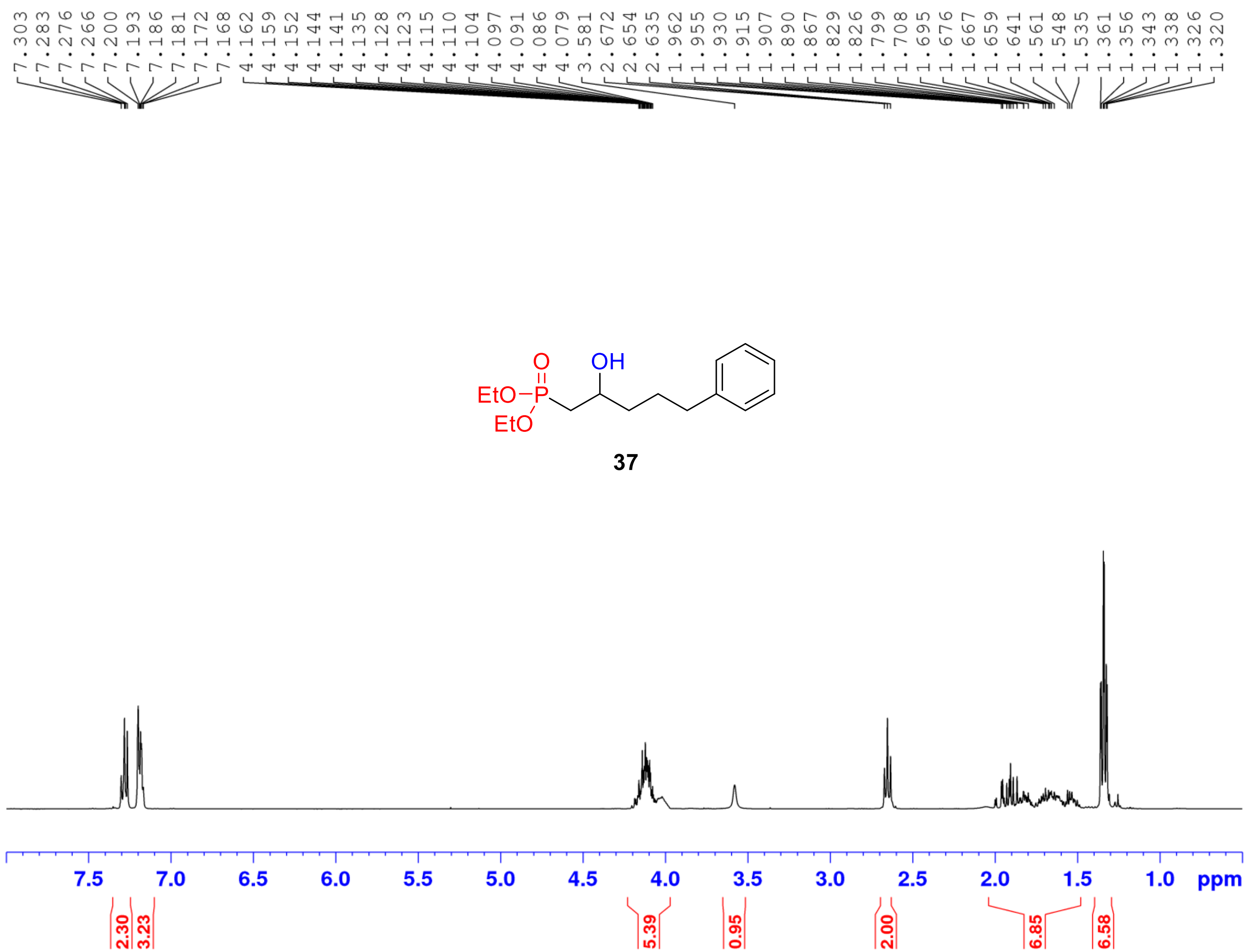
36: ^{13}C HSQC



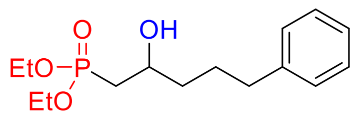


36

37: ¹H NMR

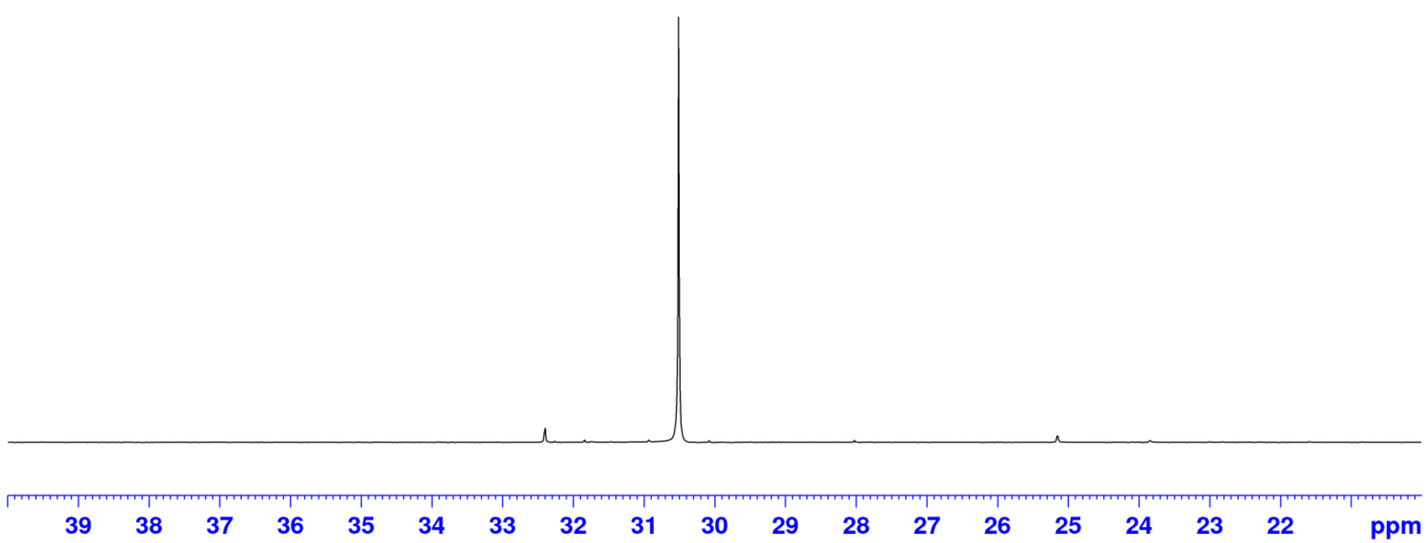

7.303
7.283
7.276
7.266
7.200
7.193
7.186
7.181
7.172
7.168
4.162
4.159
4.152
4.144
4.141
4.135
4.128
4.123
4.115
4.110
4.104
4.097
4.091
4.086
4.079
3.581
2.672
2.654
2.635
1.962
1.955
1.930
1.915
1.907
1.890
1.867
1.829
1.826
1.799
1.708
1.695
1.676
1.667
1.659
1.641
1.561
1.548
1.535
1.361
1.356
1.343
1.338
1.326
1.320
O
OH
EtO
P
EtO
37
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
2.30
3.23
5.39
0.95
2.00
6.85
6.58

37: ^{31}P NMR



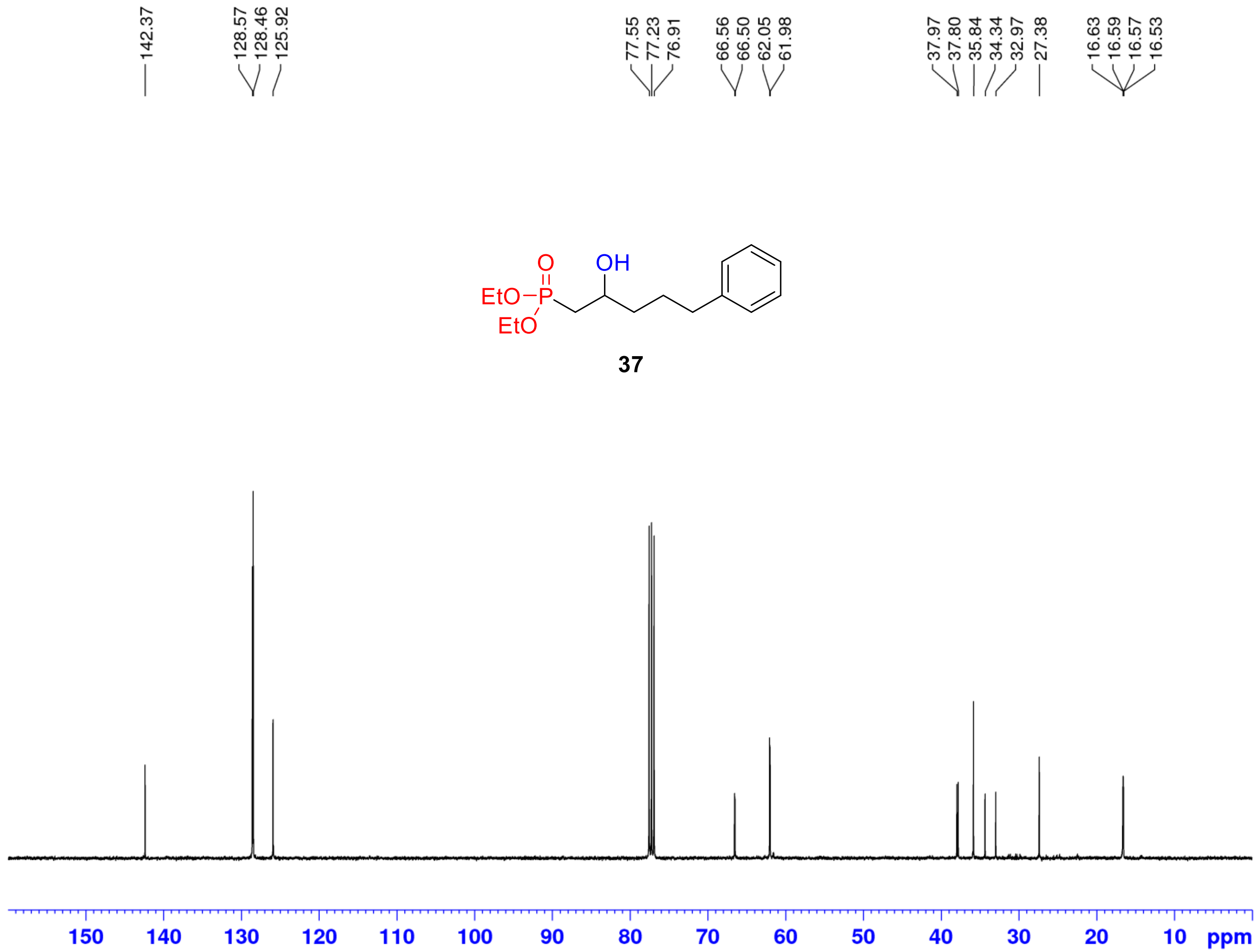
O
OH
EtO
P
EtO

37

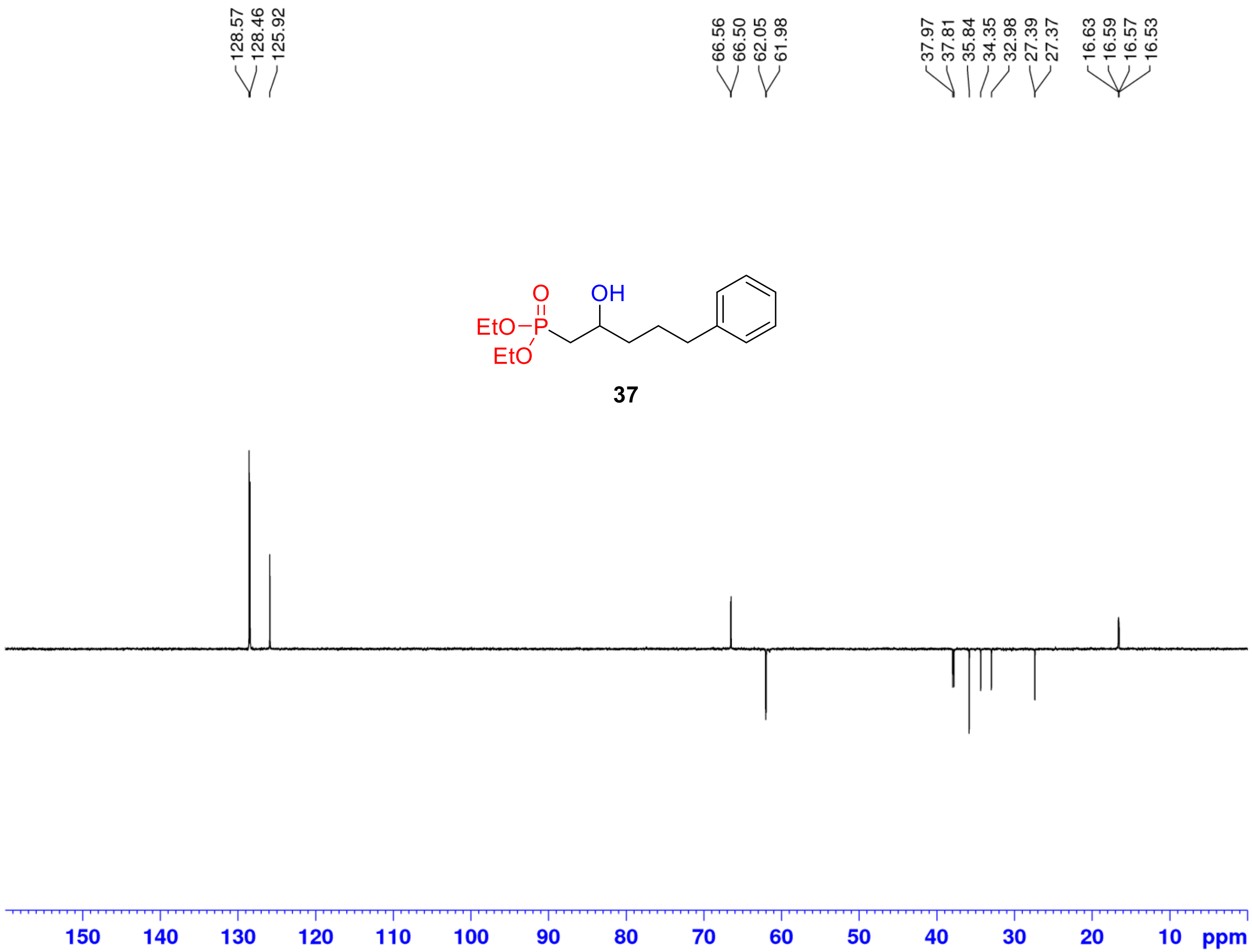


30.51
39
38
37
36
35
34
33
32
31
30
29
28
27
26
25
24
23
22
ppm

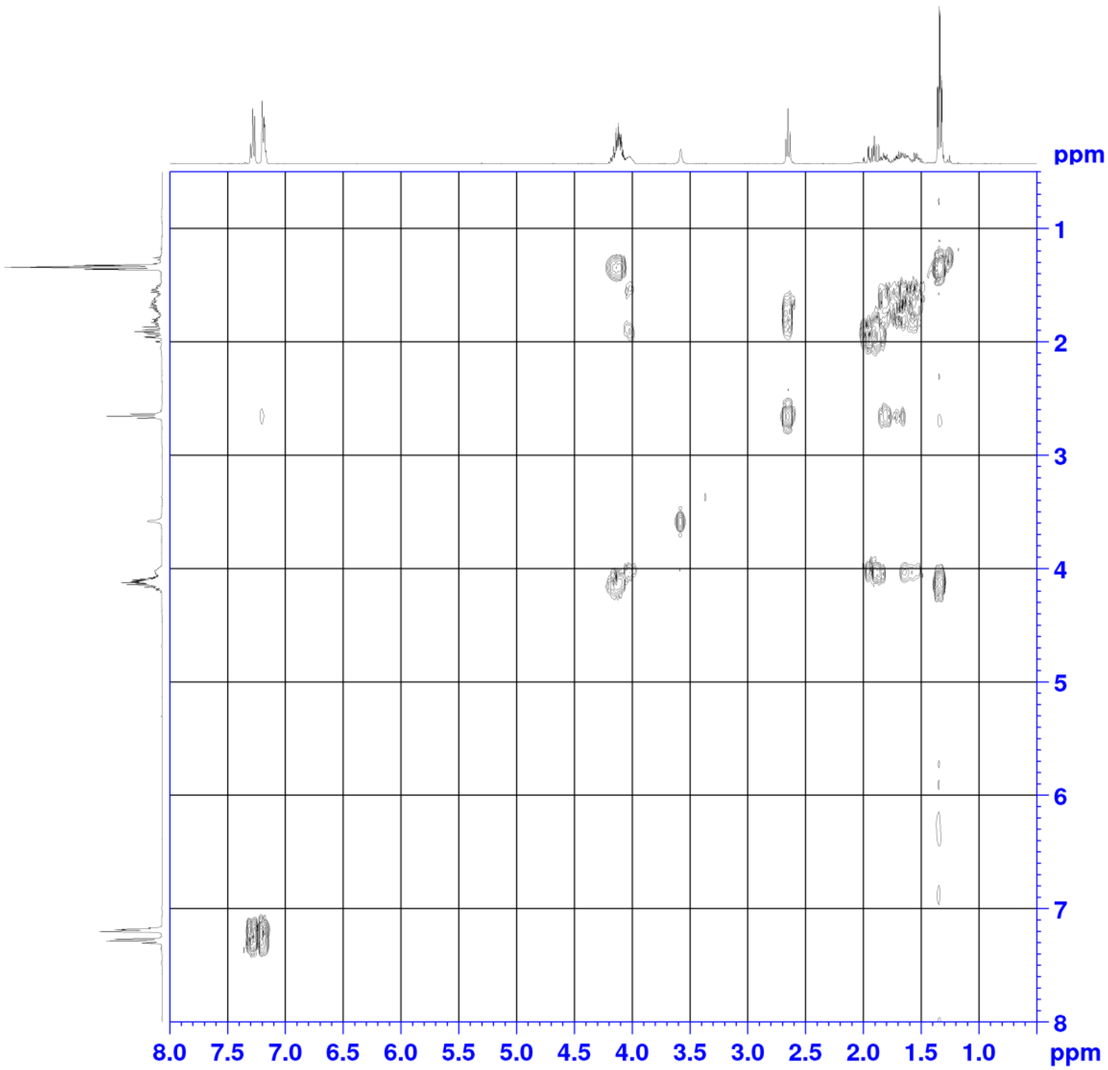
37: ^{13}C NMR

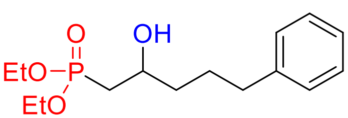

142.37
128.57
128.46
125.92
77.55
77.23
76.91
66.56
66.50
62.05
61.98
37.97
37.80
35.84
34.34
32.97
27.38
16.63
16.59
16.57
16.53
O
OH
EtO
P
EtO
37
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

37: ^{13}C DEPT135



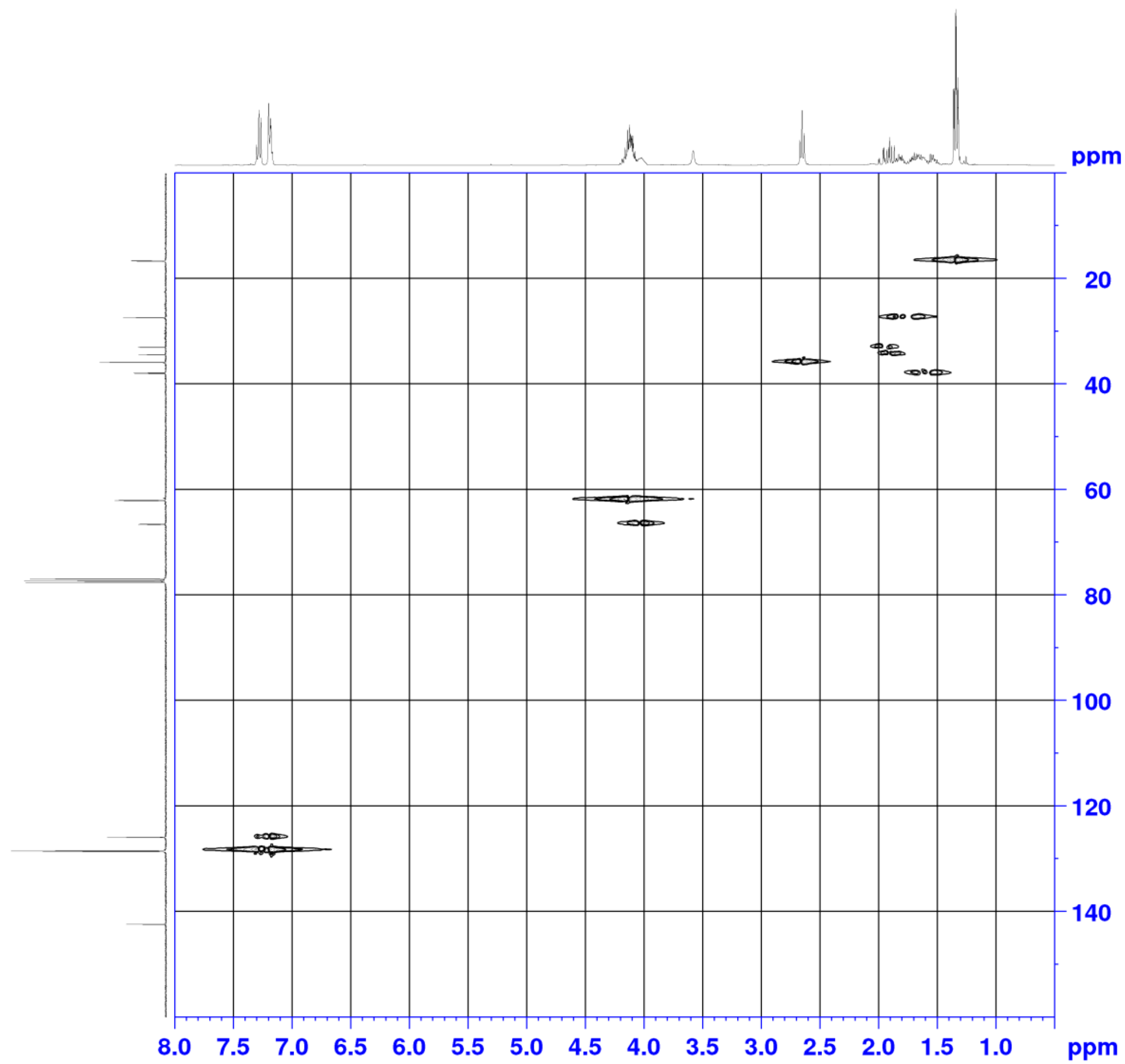
37: COSY

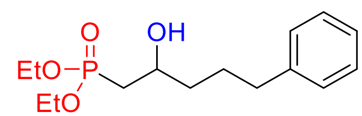

ppm
1
2
3
4
5
6
7
8
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm


O
OH
EtO
P
EtO

37

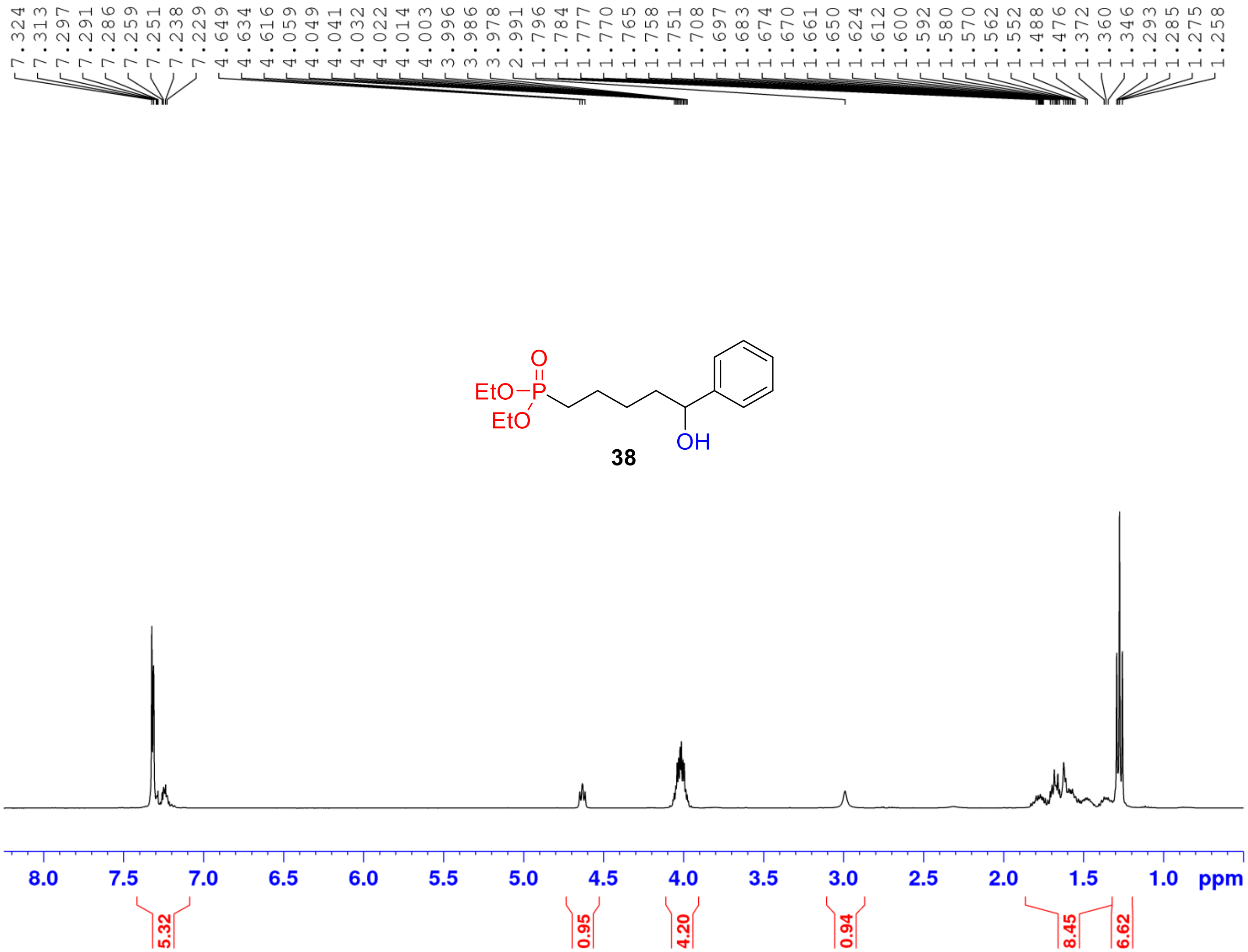
37: ^{13}C HSQC



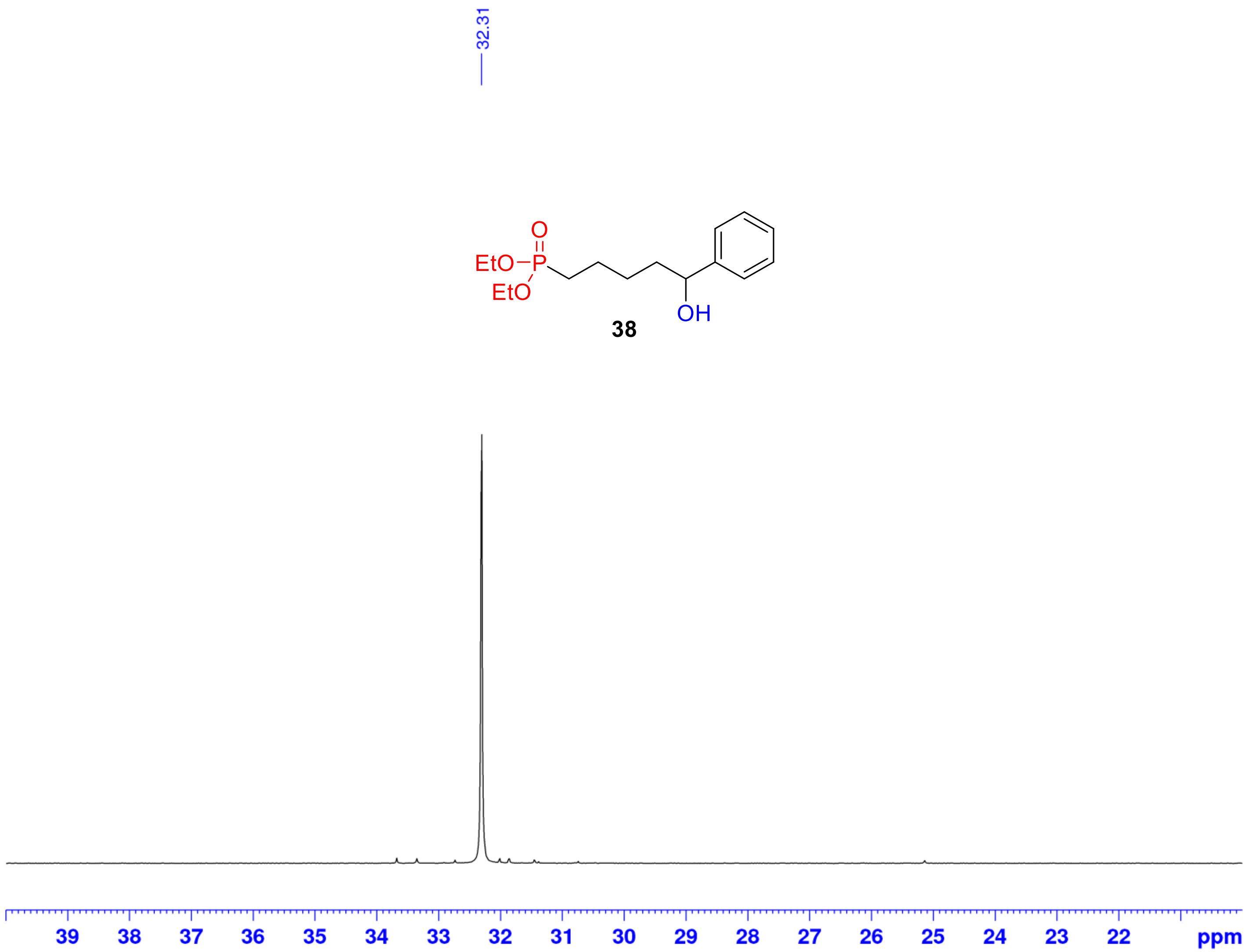


37

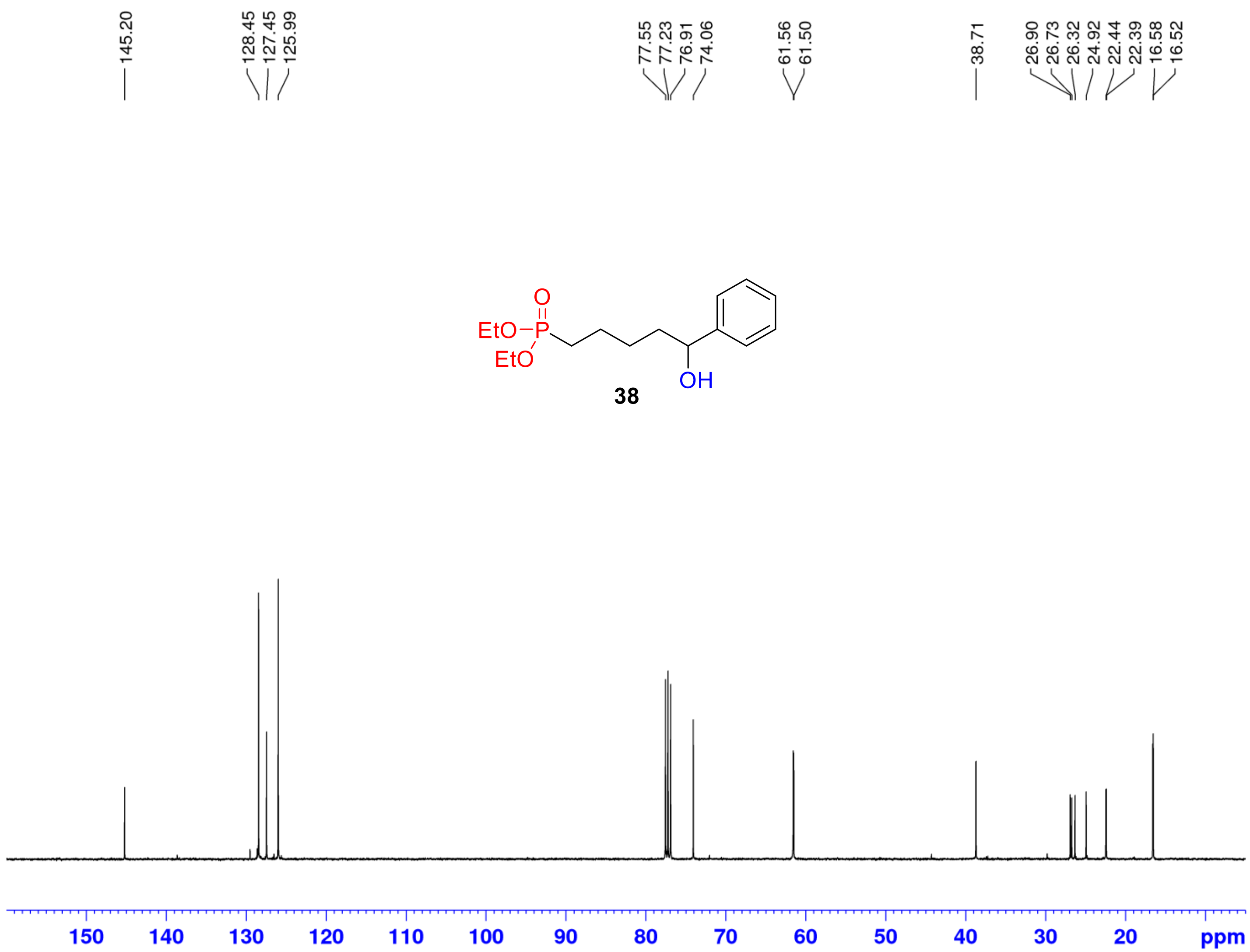
38: ^{1}H NMR


7.324
7.313
7.297
7.291
7.286
7.259
7.251
7.238
7.229
4.649
4.634
4.616
4.059
4.049
4.041
4.032
4.022
4.014
4.003
3.996
3.986
3.978
2.991
1.796
1.784
1.777
1.770
1.765
1.758
1.751
1.708
1.697
1.683
1.674
1.670
1.661
1.650
1.624
1.612
1.600
1.592
1.580
1.570
1.562
1.552
1.488
1.476
1.372
1.360
1.346
1.293
1.285
1.275
1.258
O
EtO
P
EtO
OH
38
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
ppm
5.32
0.95
4.20
0.94
8.45
6.62

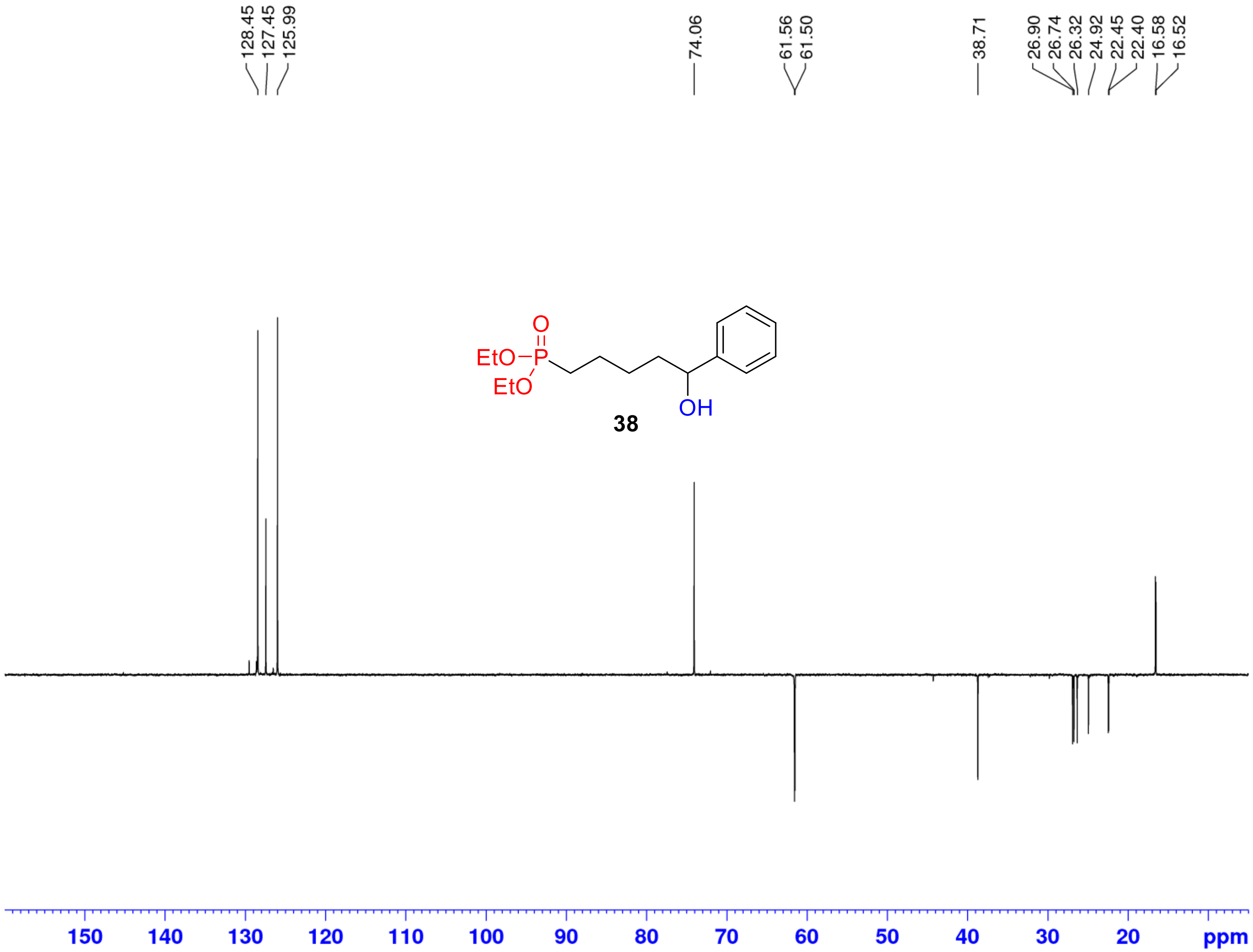
38: ^{31}P NMR


32.31
EtO
EtO
O
P
OH
38
39
38
37
36
35
34
33
32
31
30
29
28
27
26
25
24
23
22
ppm

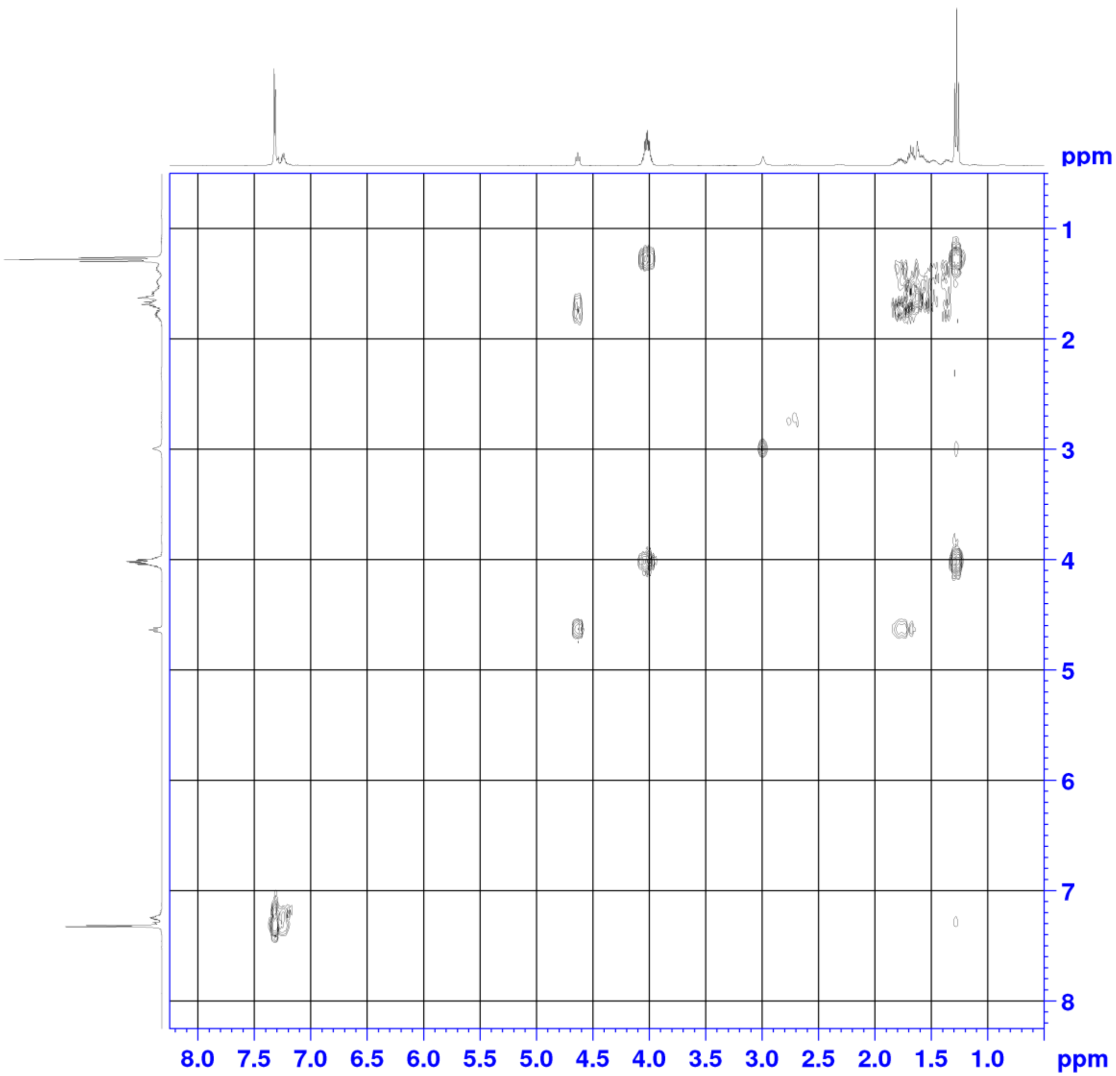
38: ^{13}C NMR


145.20
128.45
127.45
125.99
77.55
77.23
76.91
74.06
61.56
61.50
38.71
26.90
26.73
26.32
24.92
22.44
22.39
16.58
16.52
O
EtO
P
EtO
OH
38
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

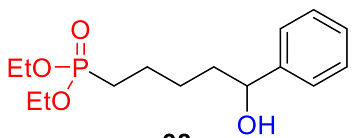
38: ^{13}C DEPT135



38: COSY



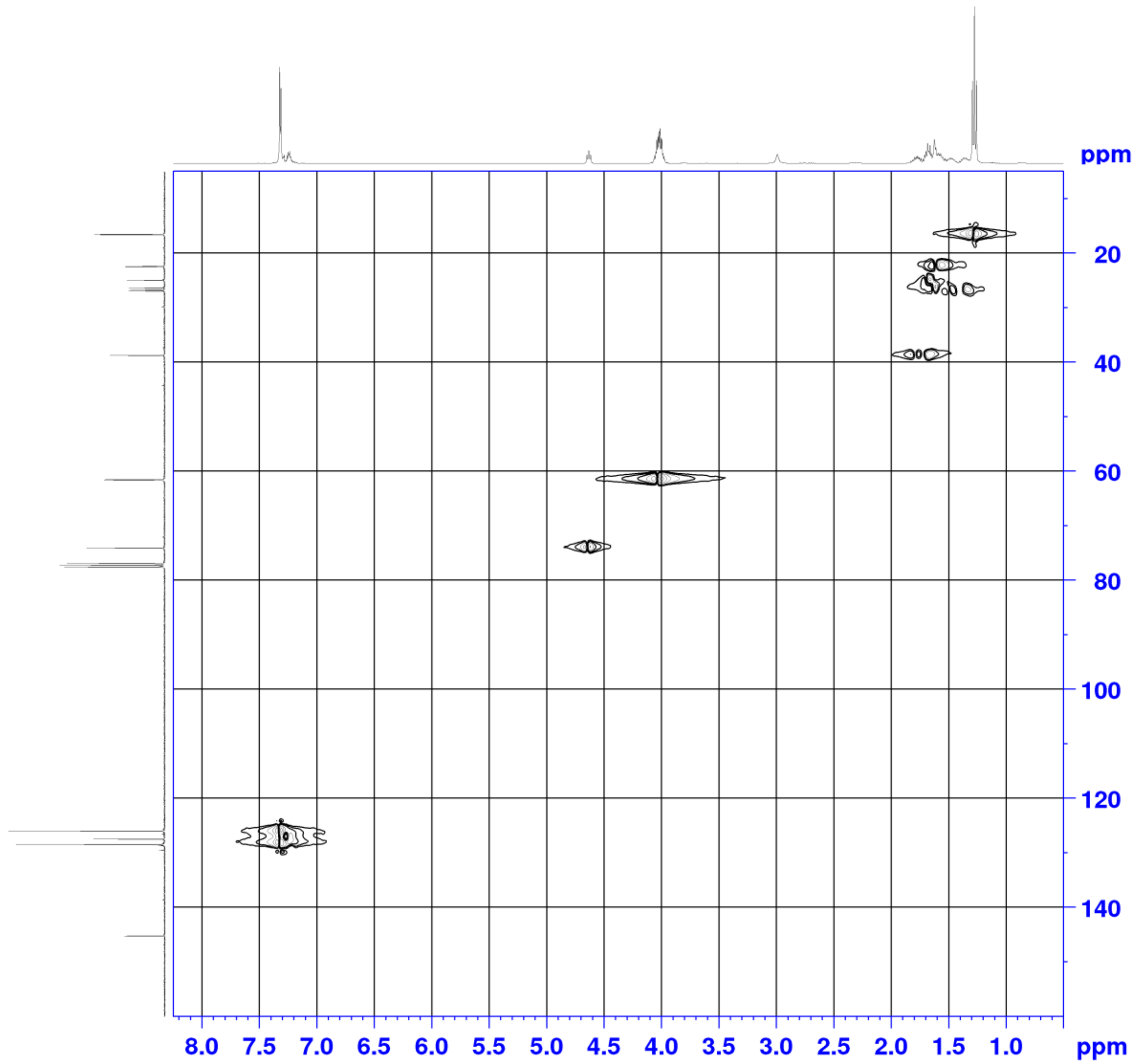
ppm
1
2
3
4
5
6
7
8
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0
ppm

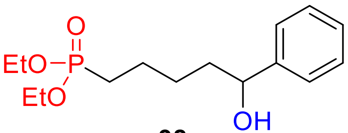


O
EtO
P
EtO
OH

38

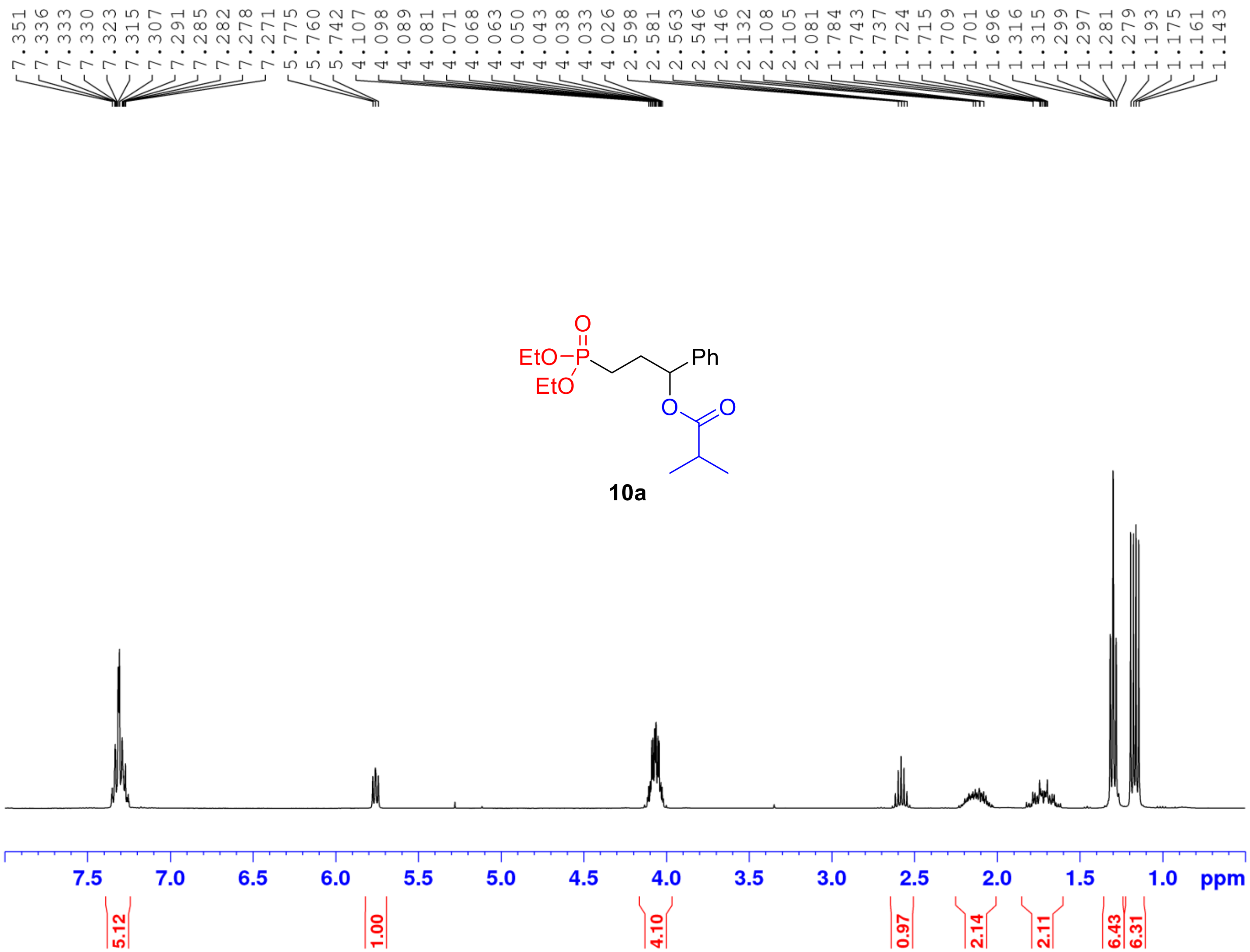
38: ^{13}C HSQC





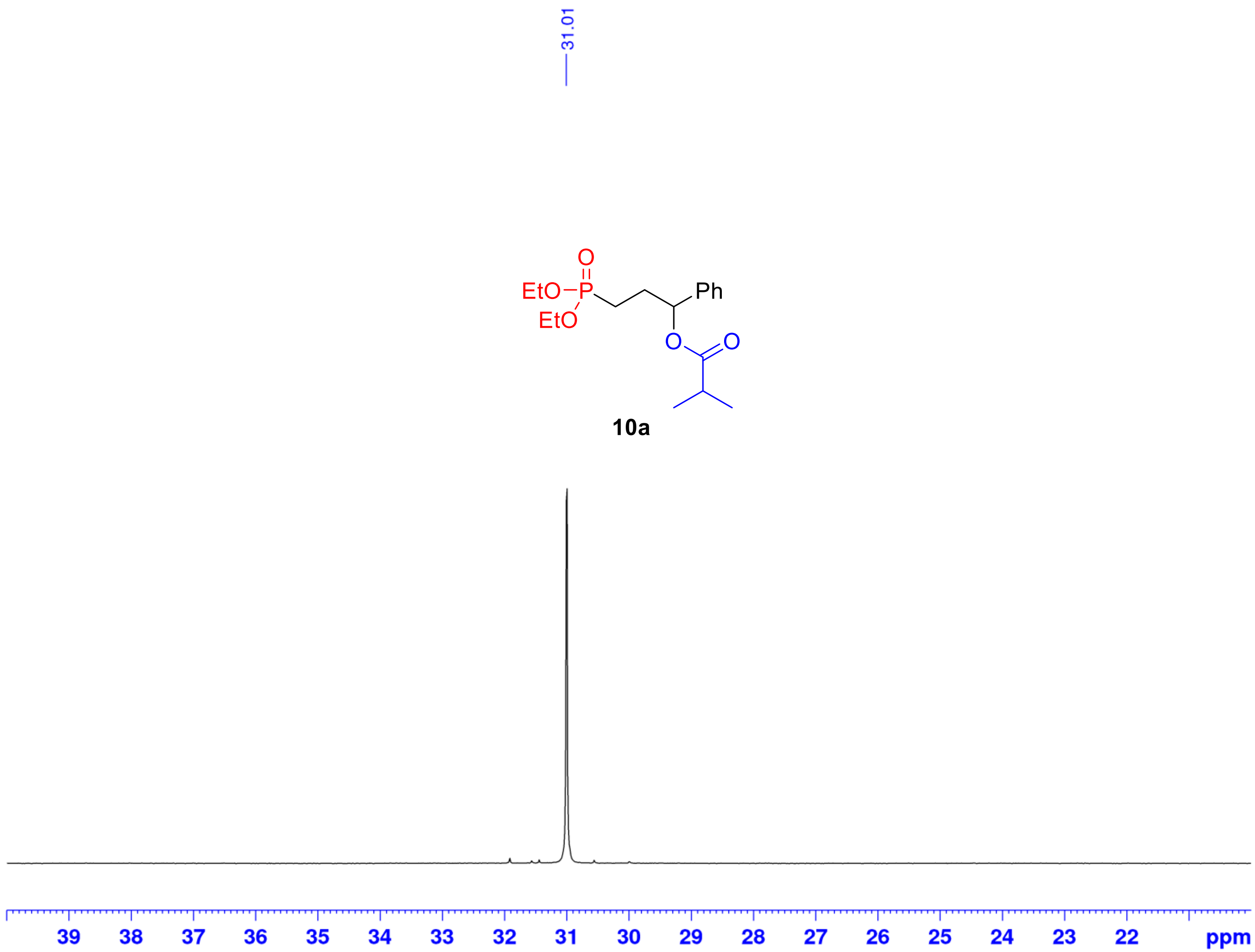
38

10a: ^{1}H NMR



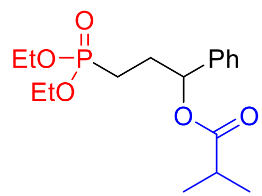
7.351
7.336
7.333
7.330
7.323
7.315
7.307
7.291
7.285
7.282
7.278
7.271
5.775
5.760
5.742
4.107
4.098
4.089
4.081
4.071
4.068
4.063
4.050
4.043
4.038
4.033
4.026
2.598
2.581
2.563
2.546
2.146
2.132
2.108
2.105
2.081
1.784
1.743
1.737
1.724
1.715
1.709
1.701
1.696
1.316
1.315
1.299
1.297
1.281
1.279
1.193
1.175
1.161
1.143
EtO
EtO
P
O
Ph
O
O
10a
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
5.12
1.00
4.10
0.97
2.14
2.11
6.43
6.31

10a: ^{31}P NMR

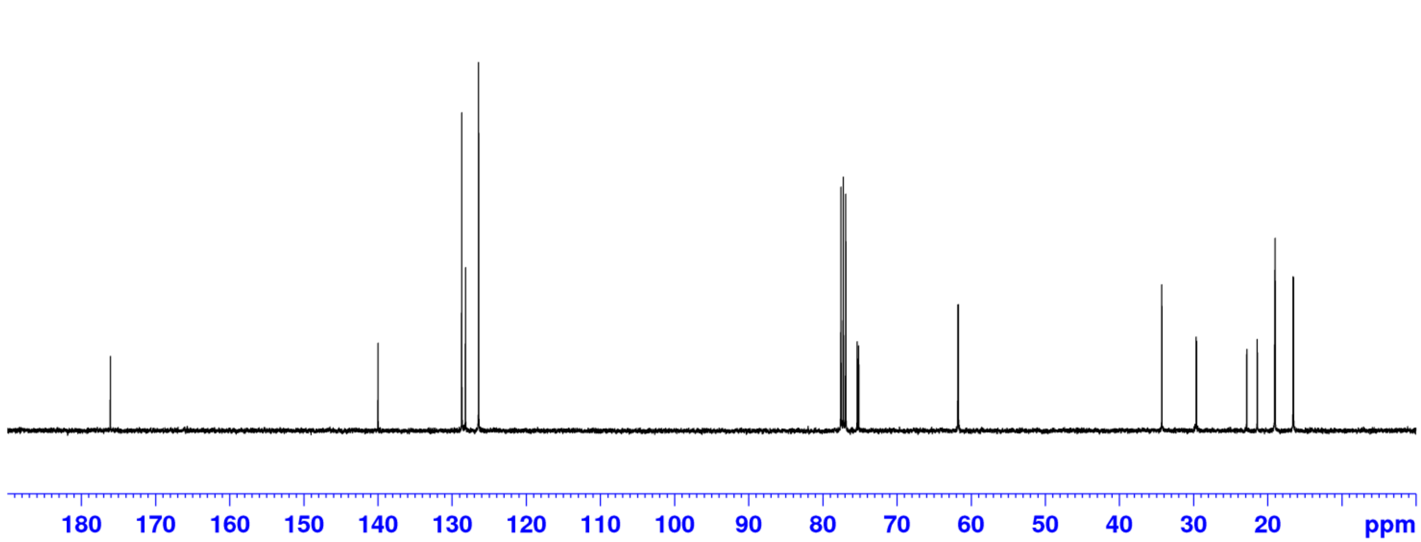


10a

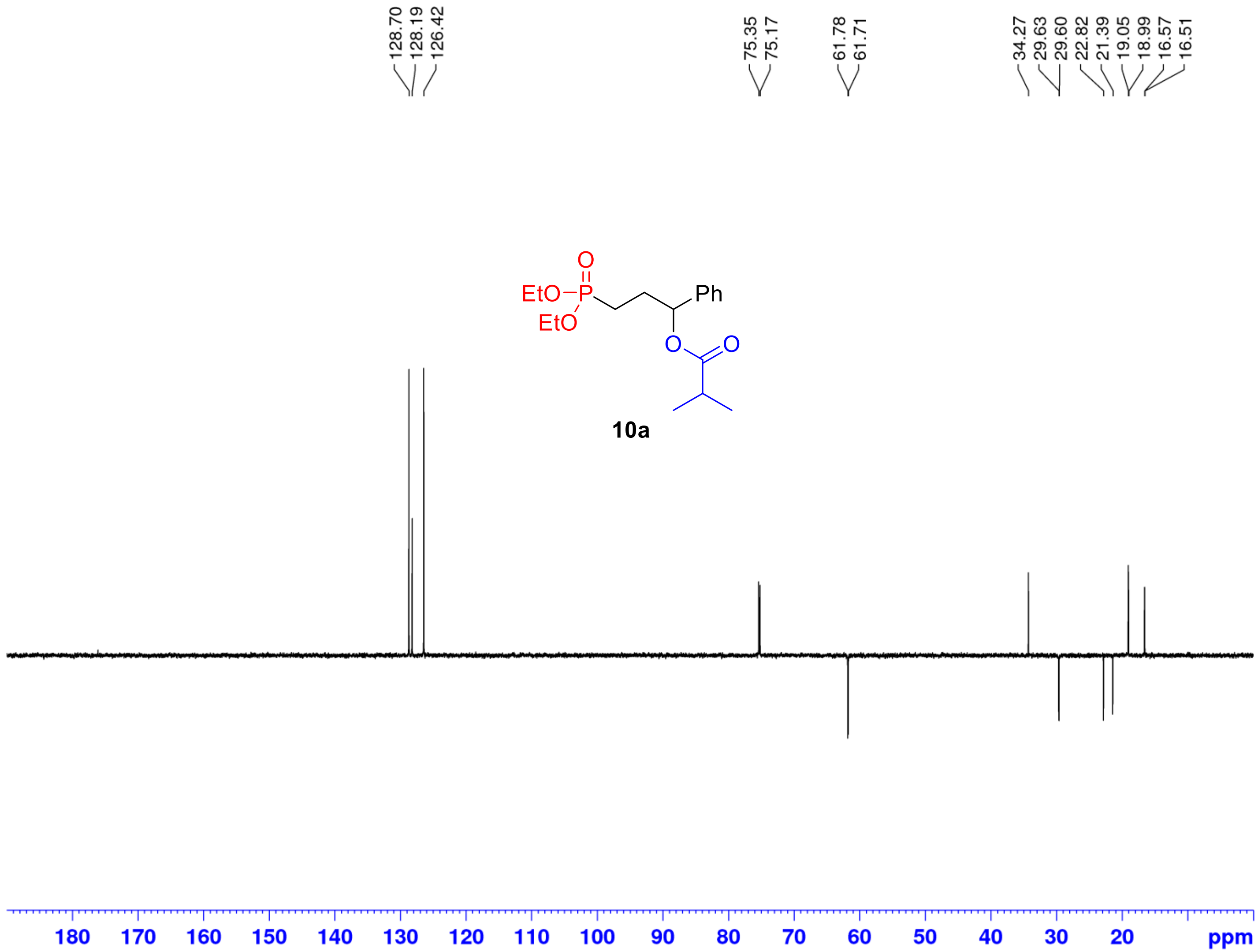
10a: ^{13}C NMR



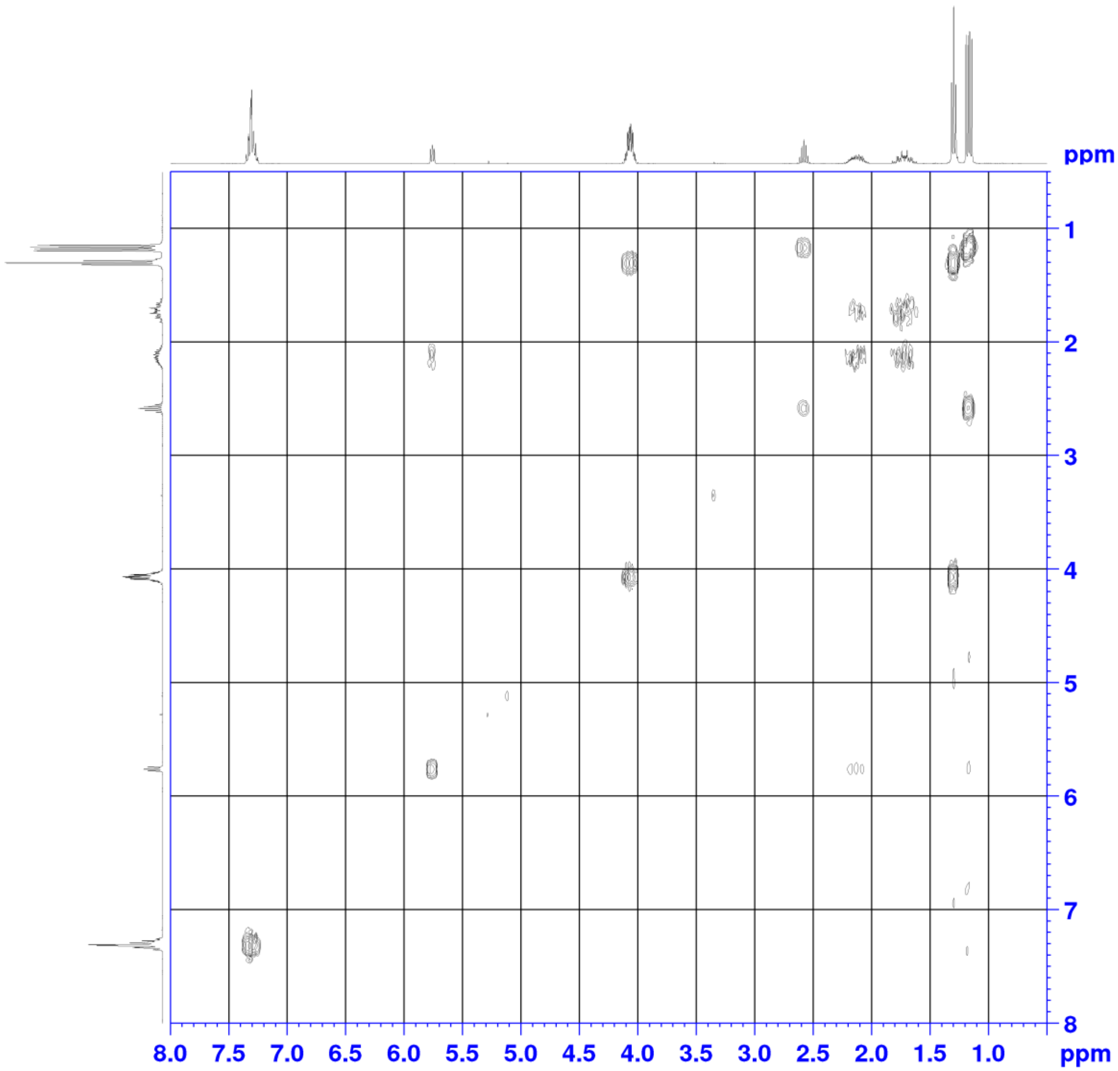
10a

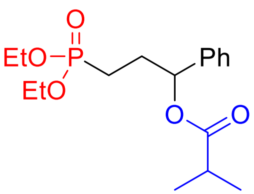


10a: ^{13}C DEPT135



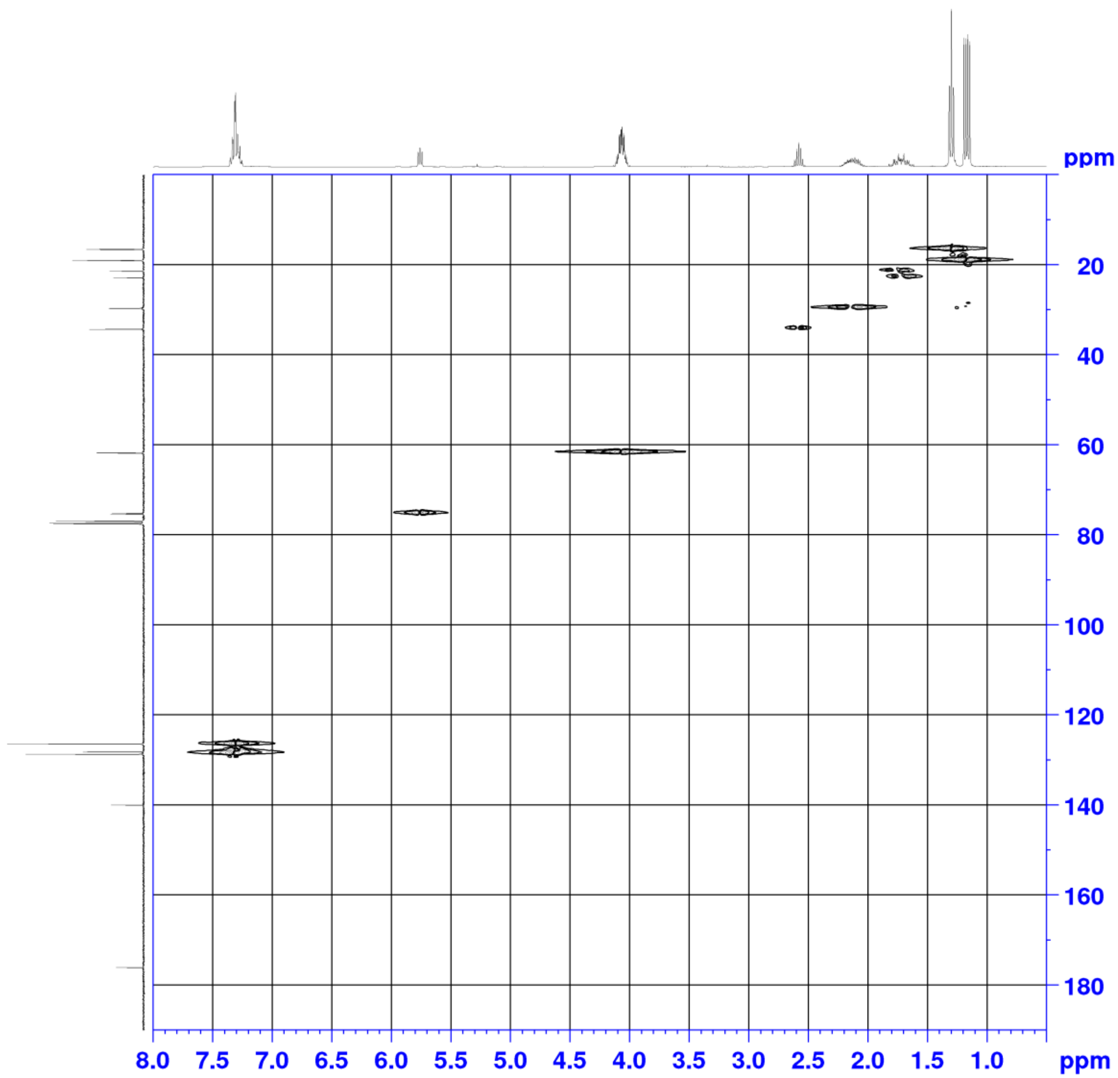
10a: COSY

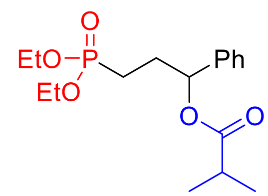

ppm
1
2
3
4
5
6
7
8
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0
ppm


O
EtO
P
EtO
Ph
O
O

10a

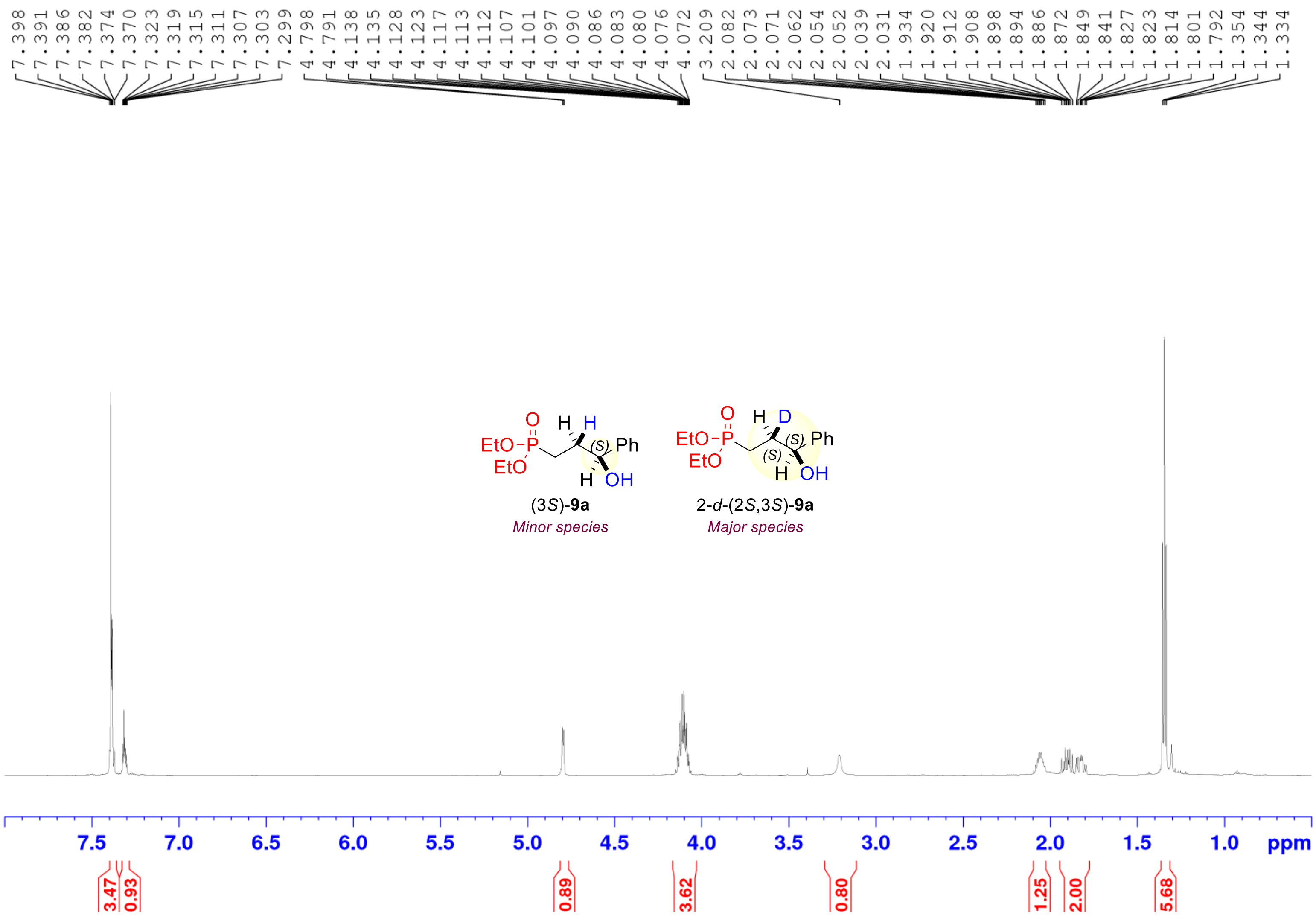
10a: ^{13}C HSQC



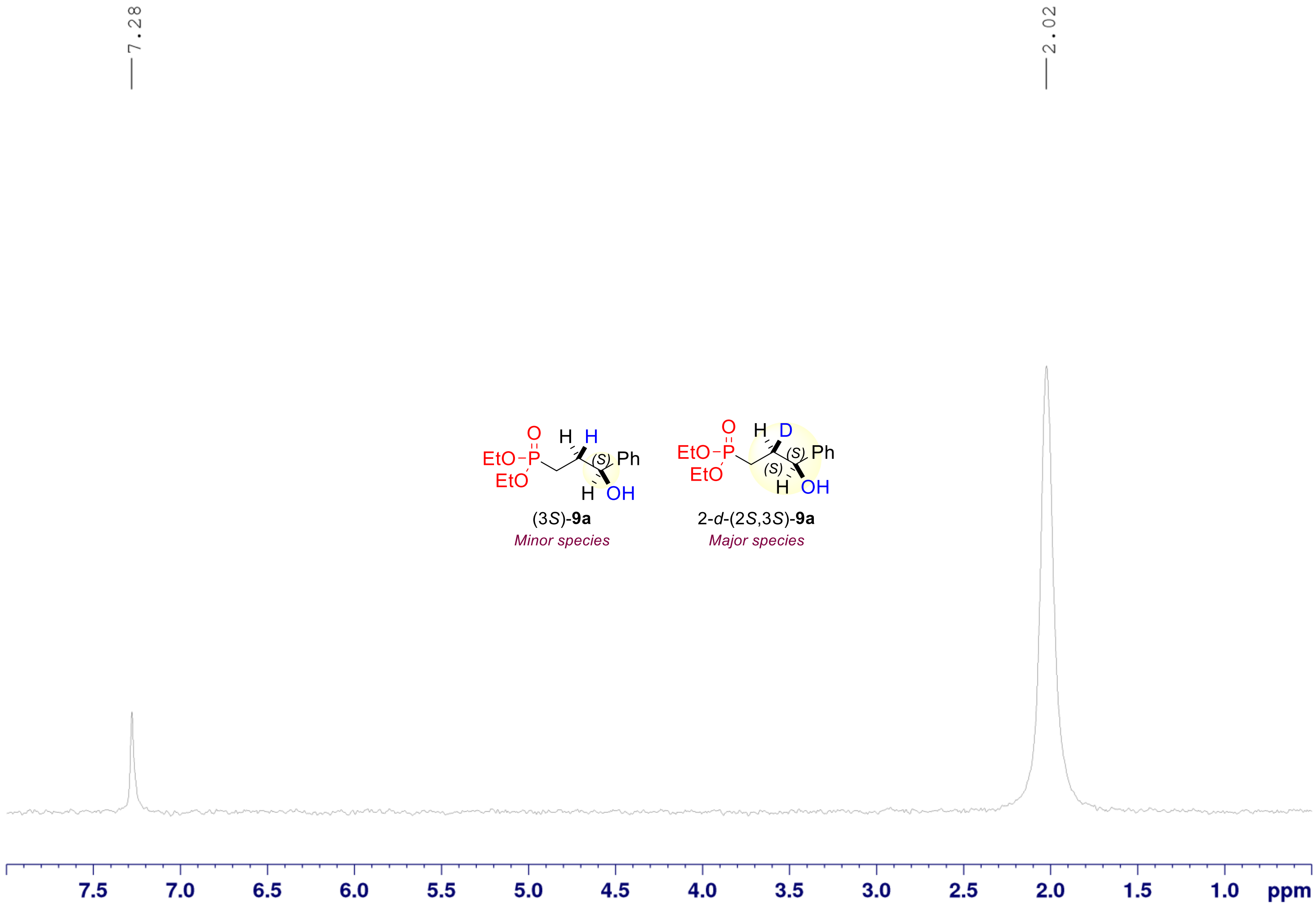


10a

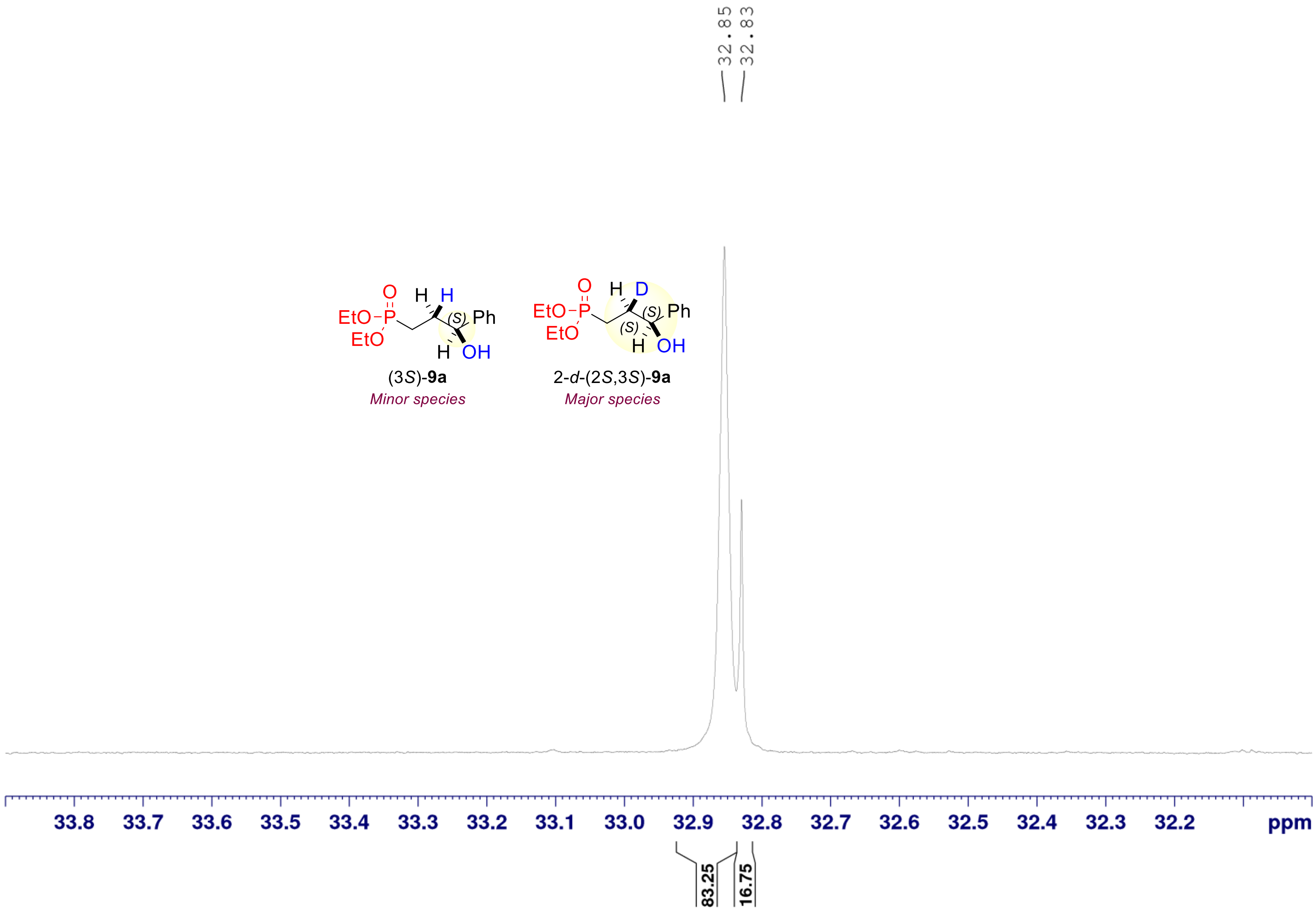
Mixture of **9a** and **2-*d*-9a** obtained via CAHB/Oxidation of **(*E*)-5a** with pinBD**: ^{1}H NMR**



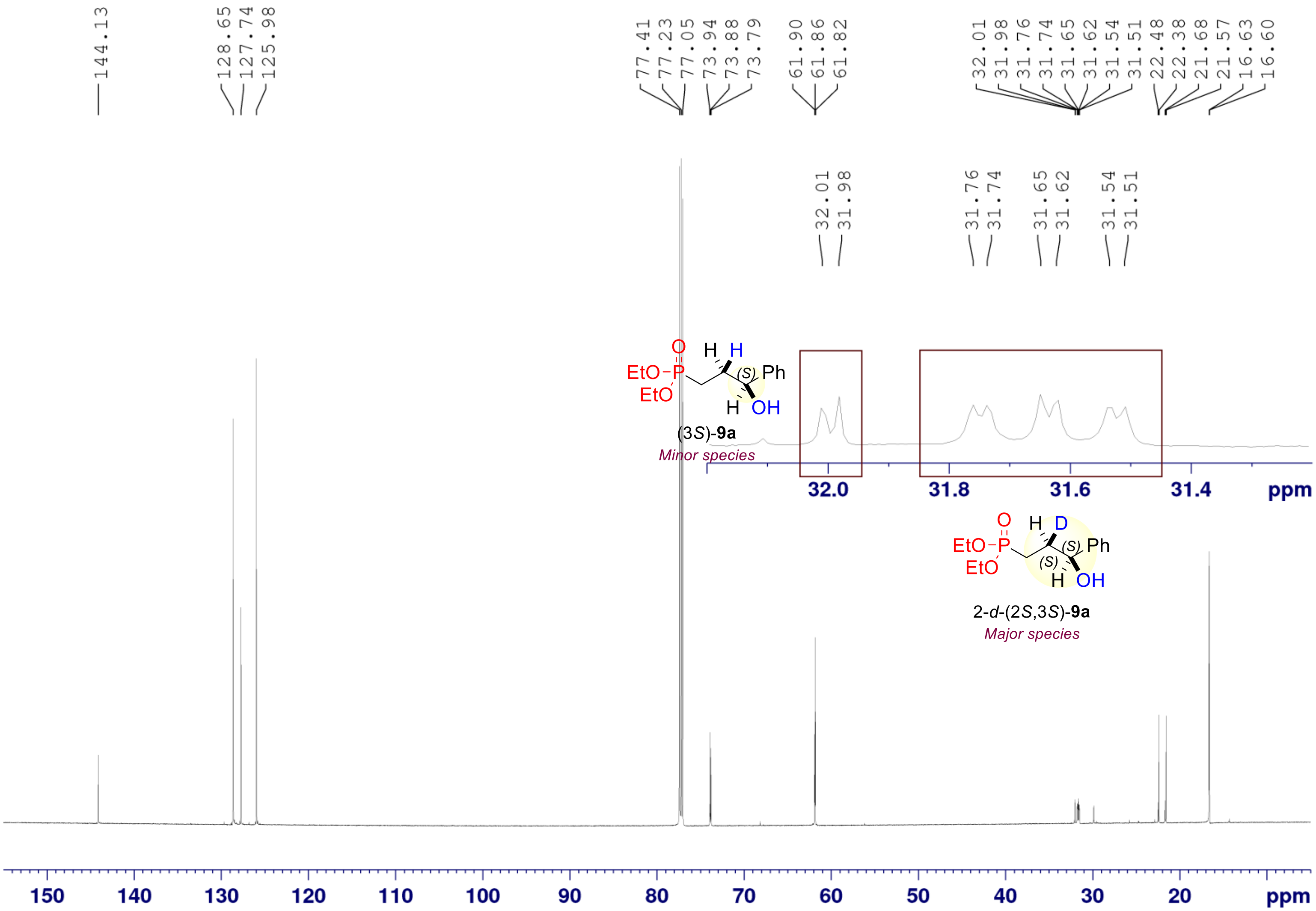
Mixture of **9a** and **2-*d*-9a** obtained via CAHB/Oxidation of (*E*)-**5a** with pinBD: **^{2}H NMR**
Note: Experiment carried out in $CHCl_3$. Peak at 7.28 is of trace $CDCl_3$.



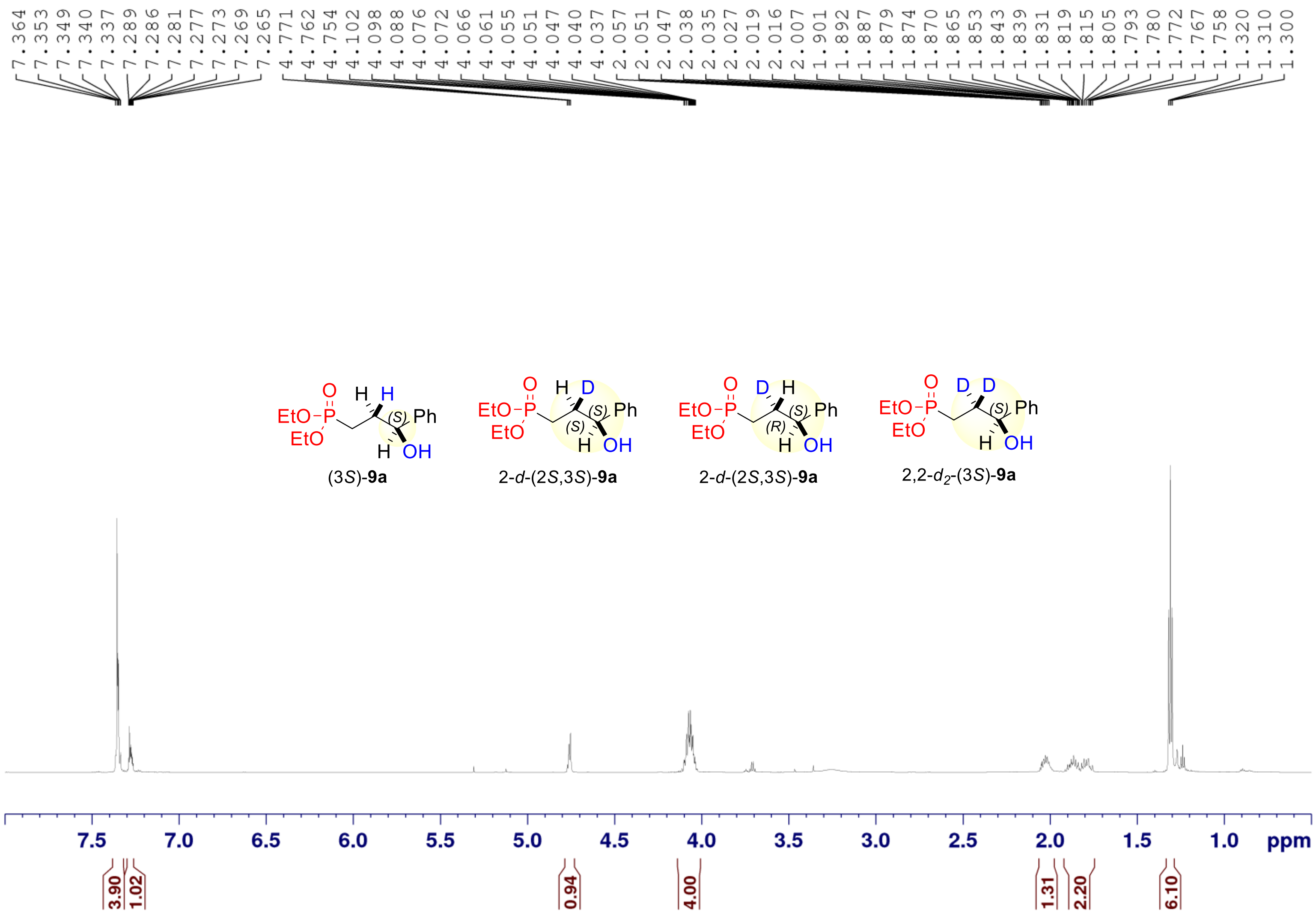
Mixture of **9a** and **2-*d*-9a** obtained via CAHB/Oxidation of (*E*)-**5a** with pinBD: **^{31}P NMR**
Note: Peak at 32.83 ppm is for **9a** and that of 32.85 ppm is for **2-*d*-9a**.



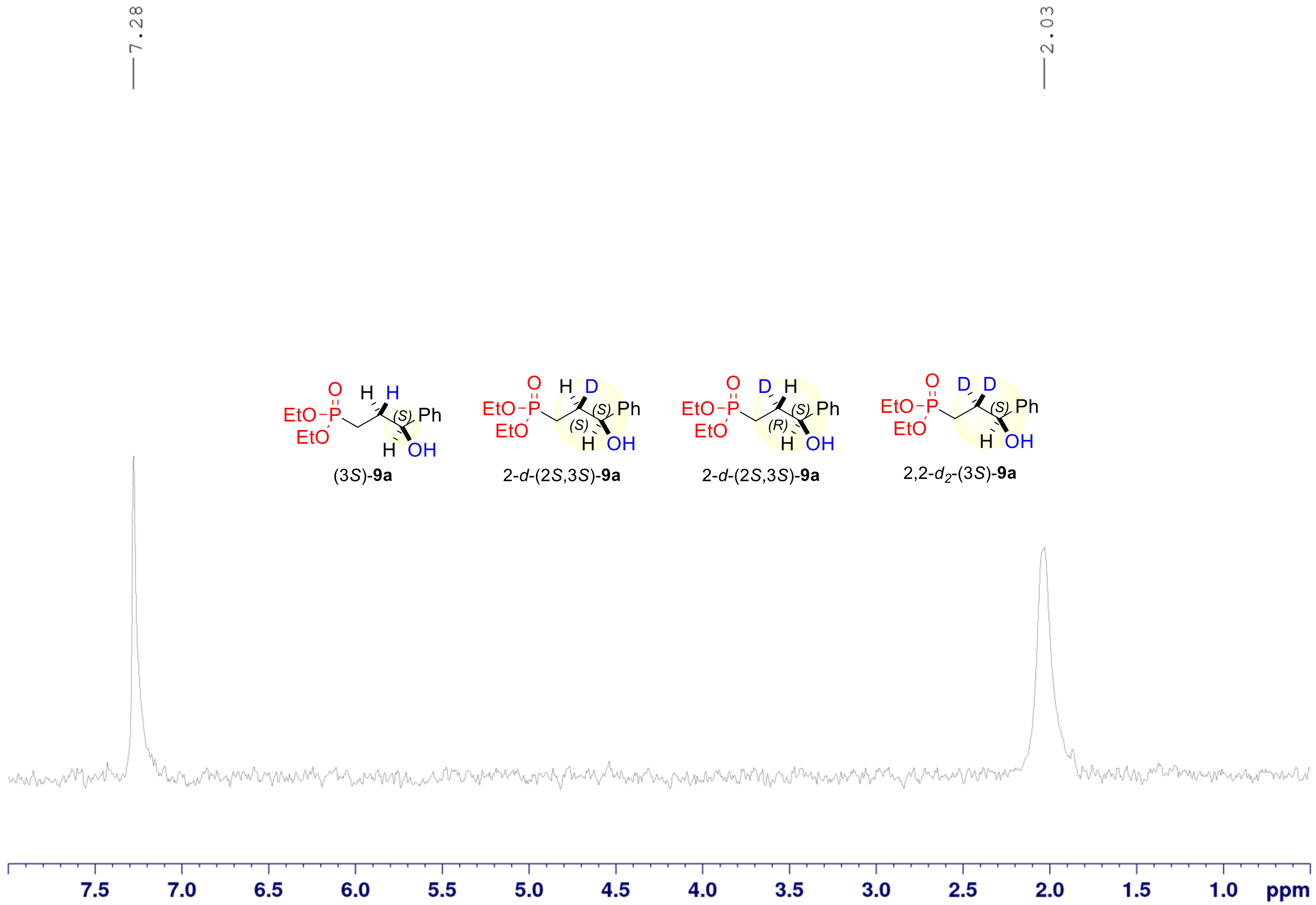
Mixture of **9a** and **2-*d*-9a** obtained via CAHB/Oxidation of (*E*)-**5a** with pinBD: ^{13}**C NMR**
Note: Peaks in boxes are for β-carbon. β-carbon of **2-*d*-9a** connected to deuterium appears as a triplet of doublets.



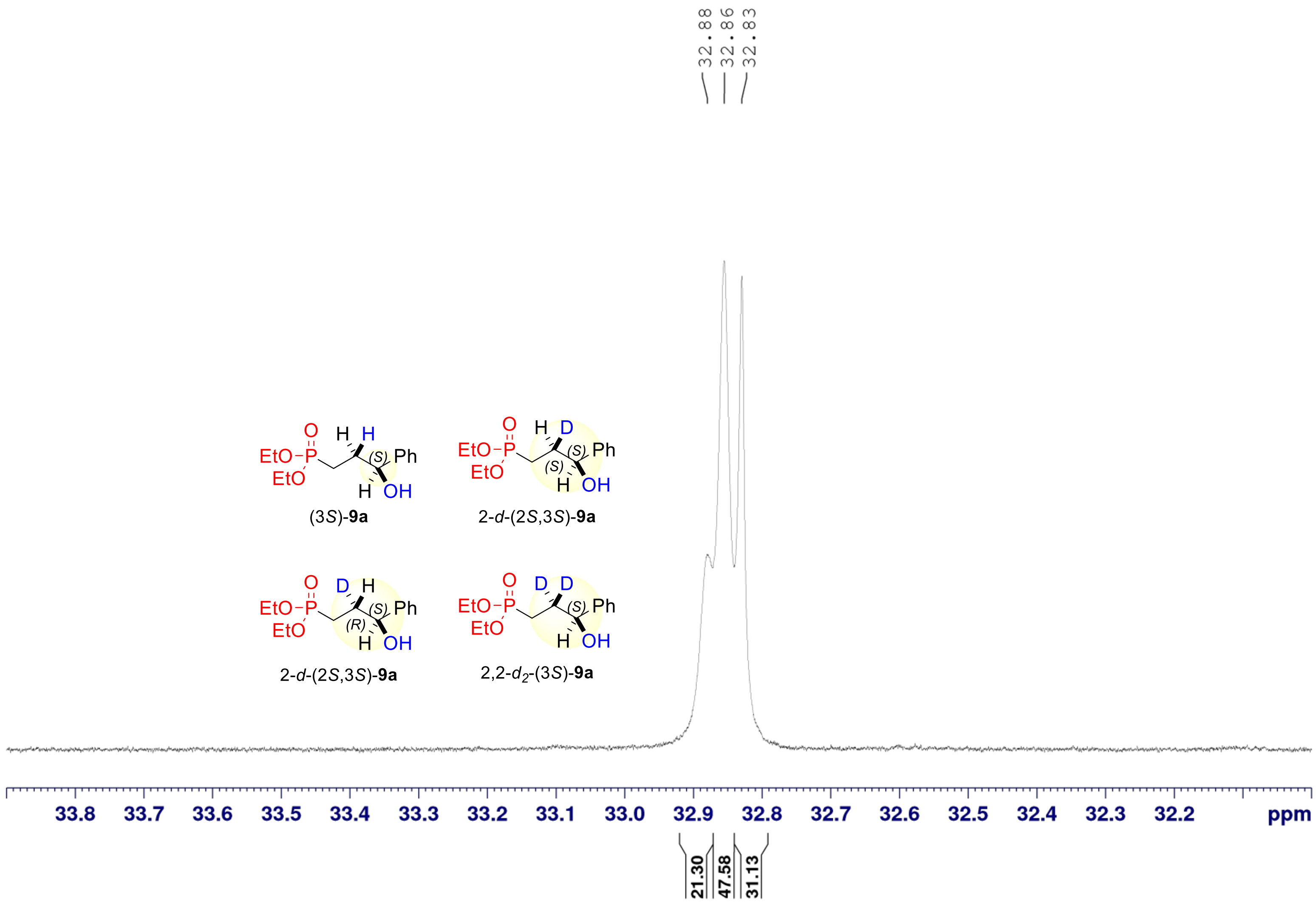
Mixture of **9a**, diastereomeric **2-*d*-9a** and **2,2-d_2-9a** obtained via CAHB/Oxidation of (*Z*)-**5a** with pinBD: **^{1}H NMR**



Mixture of **9a**, diastereomeric **2-*d*-9a** and **2,2-*d*$_2$-9a** obtained via CAHB/Oxidation of (*Z*)-**5a** with pinBD: **^{2}H NMR**
Note: Experiment carried out in $CHCl_3$. Peak at 7.28 is of trace $CDCl_3$.



Mixture of **9a**, diastereomeric **2-*d*-9a** and **2,2-*d*$_2$-9a** obtained via CAHB/Oxidation of (*Z*)-**5a** with pinBD: **^{31}P NMR**
Note: Peak at 32.83 ppm is for **9a**, and that of 32.86 ppm is for **2-*d*-9a** diastereomers and 32.88 ppm is for **2,2-*d*$_2$-9a**.



Mixture of **9a**, diastereomeric **2-*d*-9a** and **2,2-*d*$_2$-9a** obtained via CAHB/Oxidation of (*Z*)-**5a** with pinBD: ^{13}**C NMR**

Note: Peaks in boxes are for β-carbon. β-carbons of diastereomeric **2-*d*-9a**'s connected to deuterium appear as triplet of triplets (overlapping pair of triplet of doublets). **2,2-*d*$_2$-9a** is identified via HRMS only.