Preparation of tetrasubstituted 3-phosphonopyrroles through hydroamination: scope and limitations

Wouter Debrouwer, Thomas S.A. Heugebaert, Christian V. Stevens*

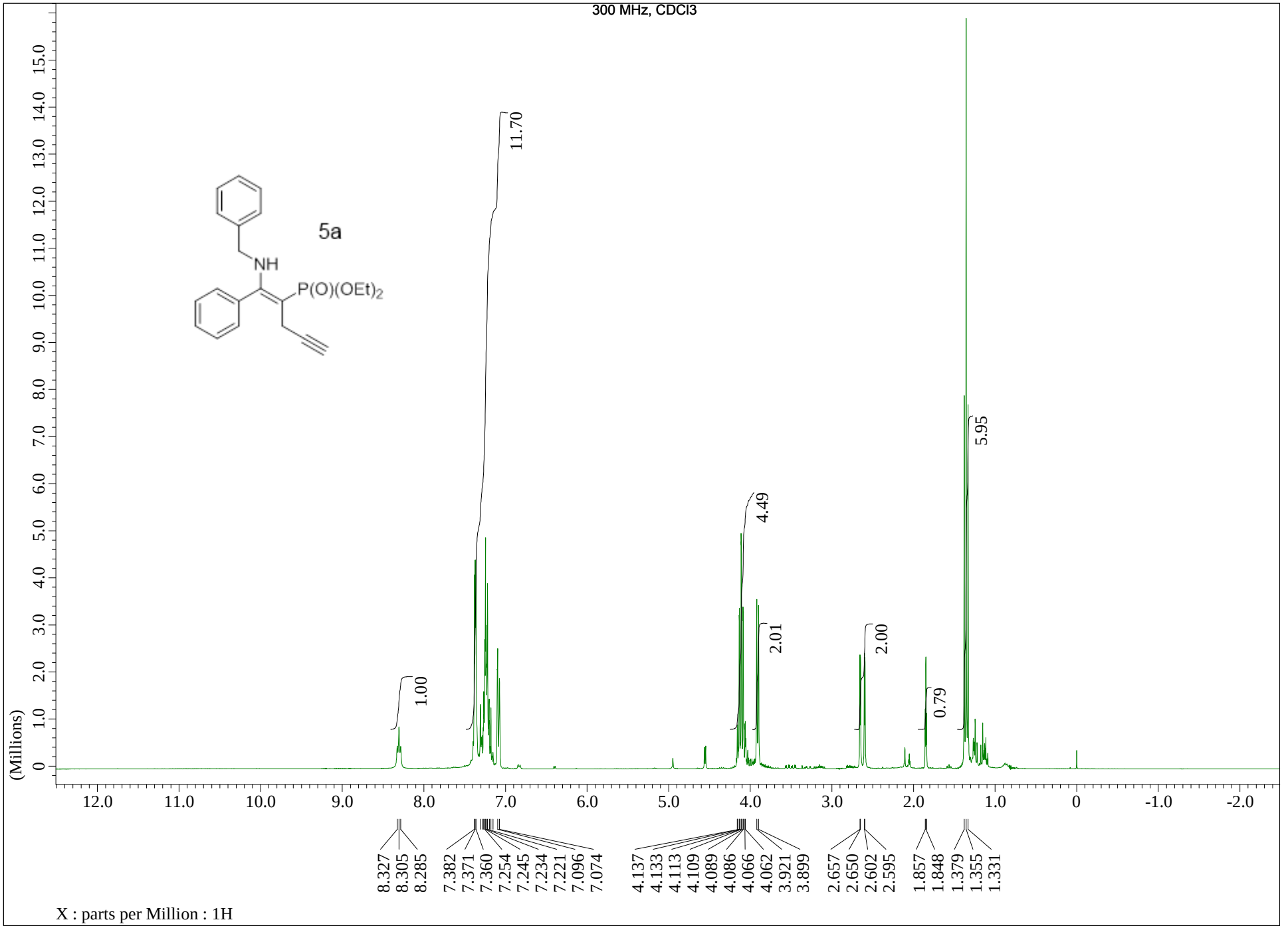
SynBioC Research Group, Department of Sustainable Organic Chemistry and Technology, Faculty of Bioscience Engineering, Ghent University, Coupure Links 653, B-9000 Gent, Belgium.

* E-mail: chris.stevens@ugent.be

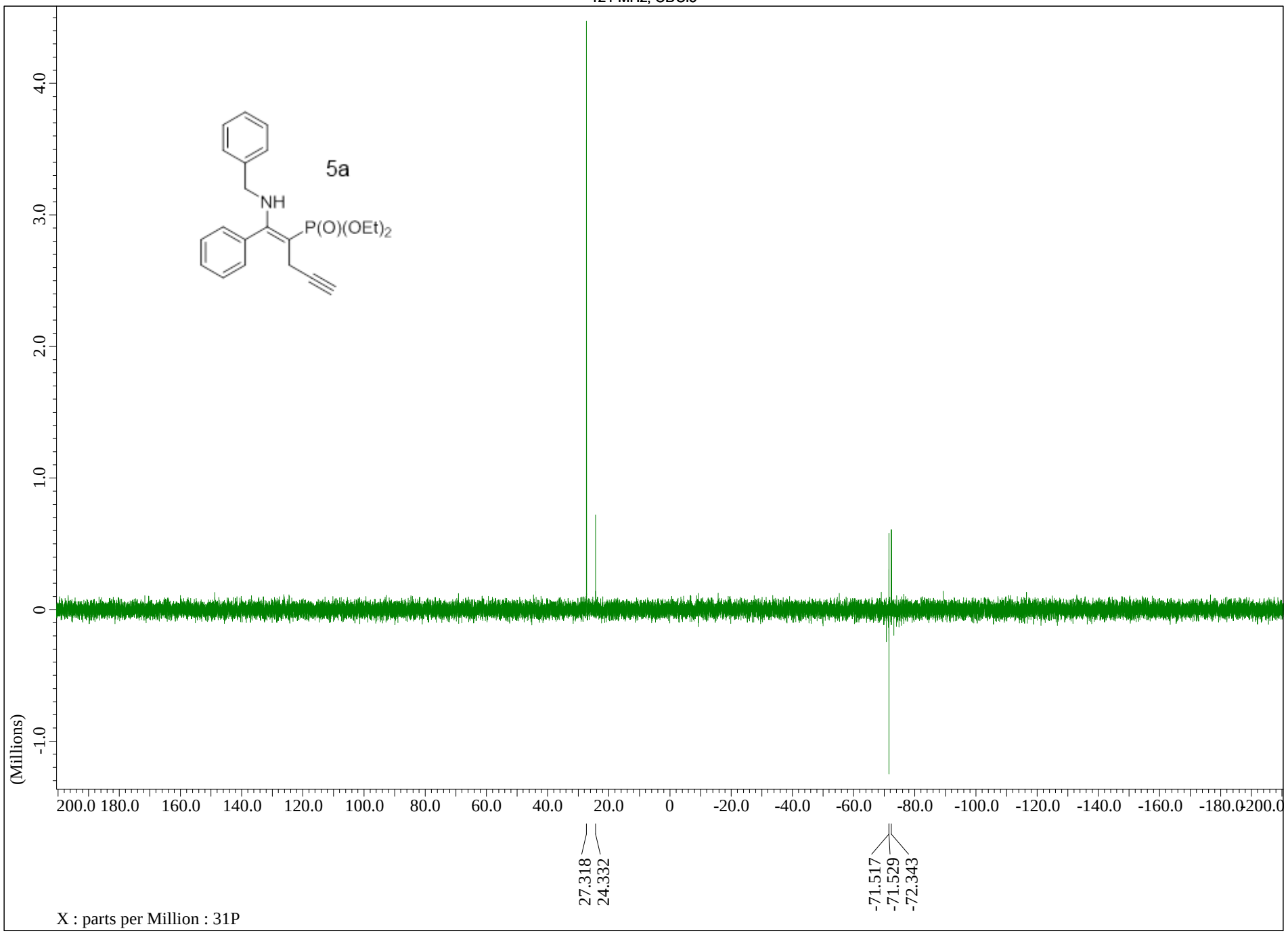
Supporting Information

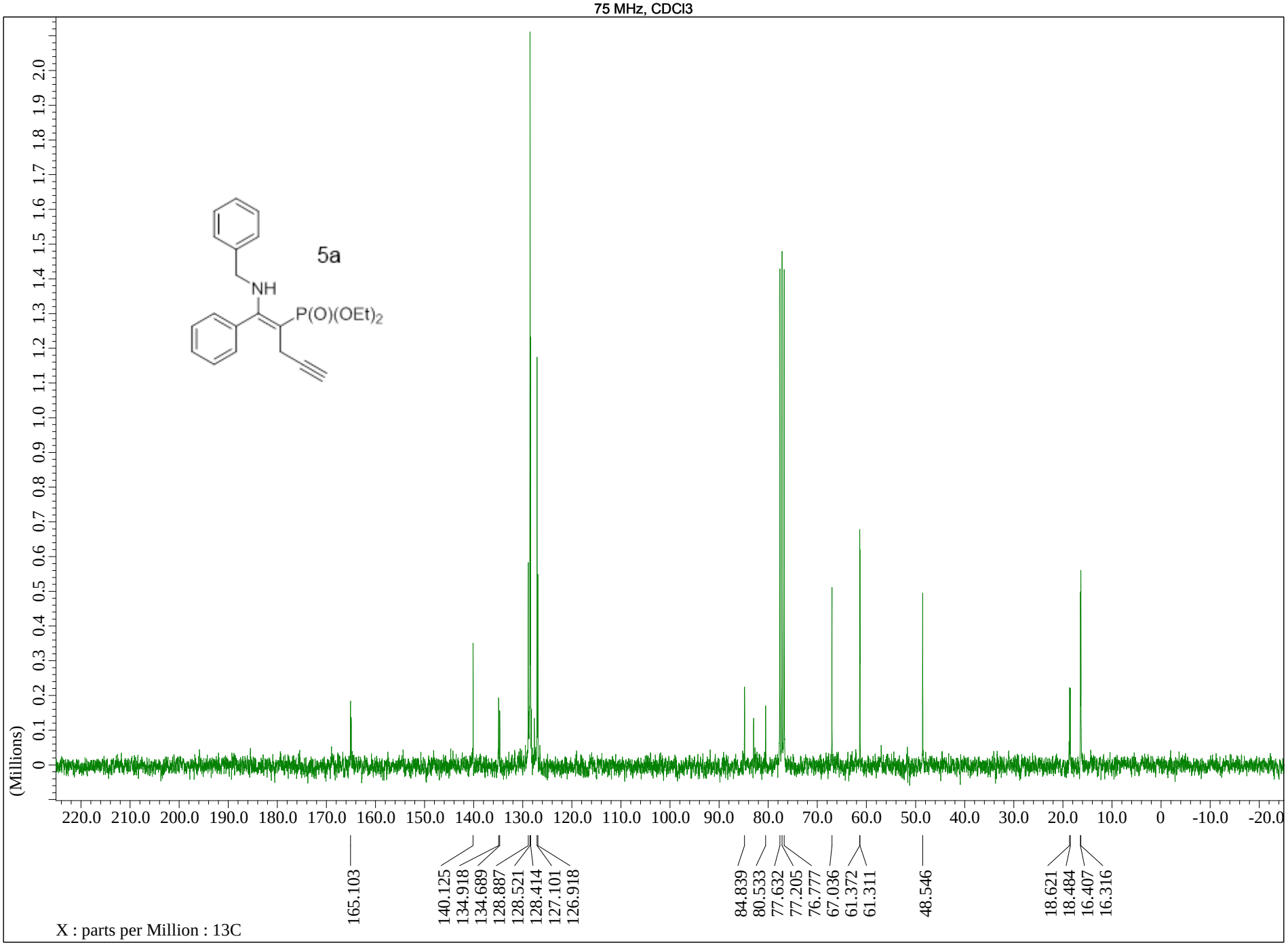
Table of contents.....SI1

^{1}H, ^{13}C, ^{31}P, ^{19}F NMR spectra and HPLC chromatograms of new compounds..SI2-SI95

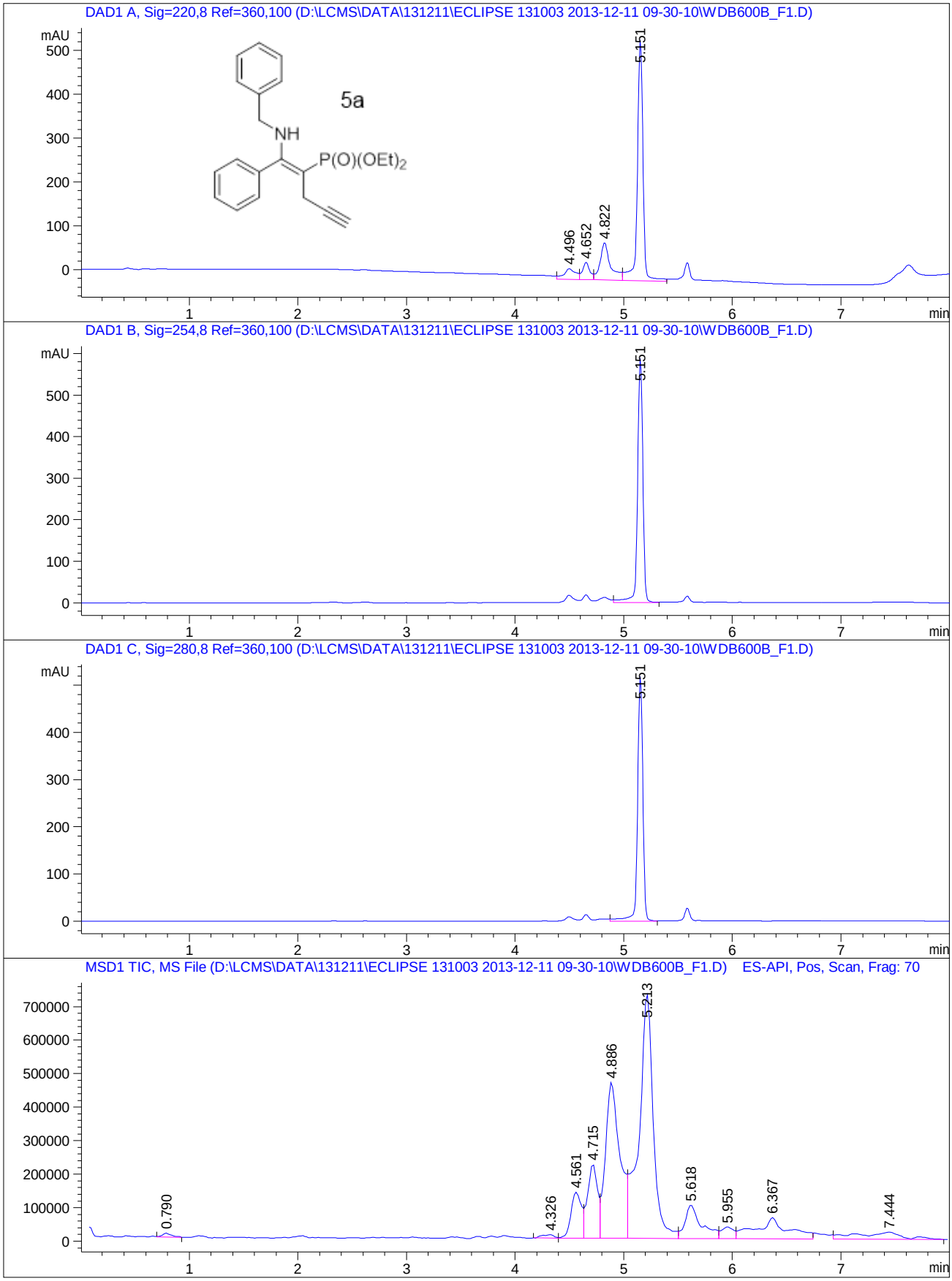

300 MHz, CDCl3
5a
NH
P(O)(OEt)2
11.70
4.49
2.01
2.00
1.00
0.79
5.95
(Millions)
8.327
8.305
8.285
7.382
7.371
7.360
7.254
7.245
7.234
7.221
7.096
7.074
4.137
4.133
4.113
4.109
4.089
4.086
4.066
4.062
3.921
3.899
2.657
2.650
2.602
2.595
1.857
1.848
1.379
1.355
1.331
X : parts per Million : 1H

121 MHz, CDCl3

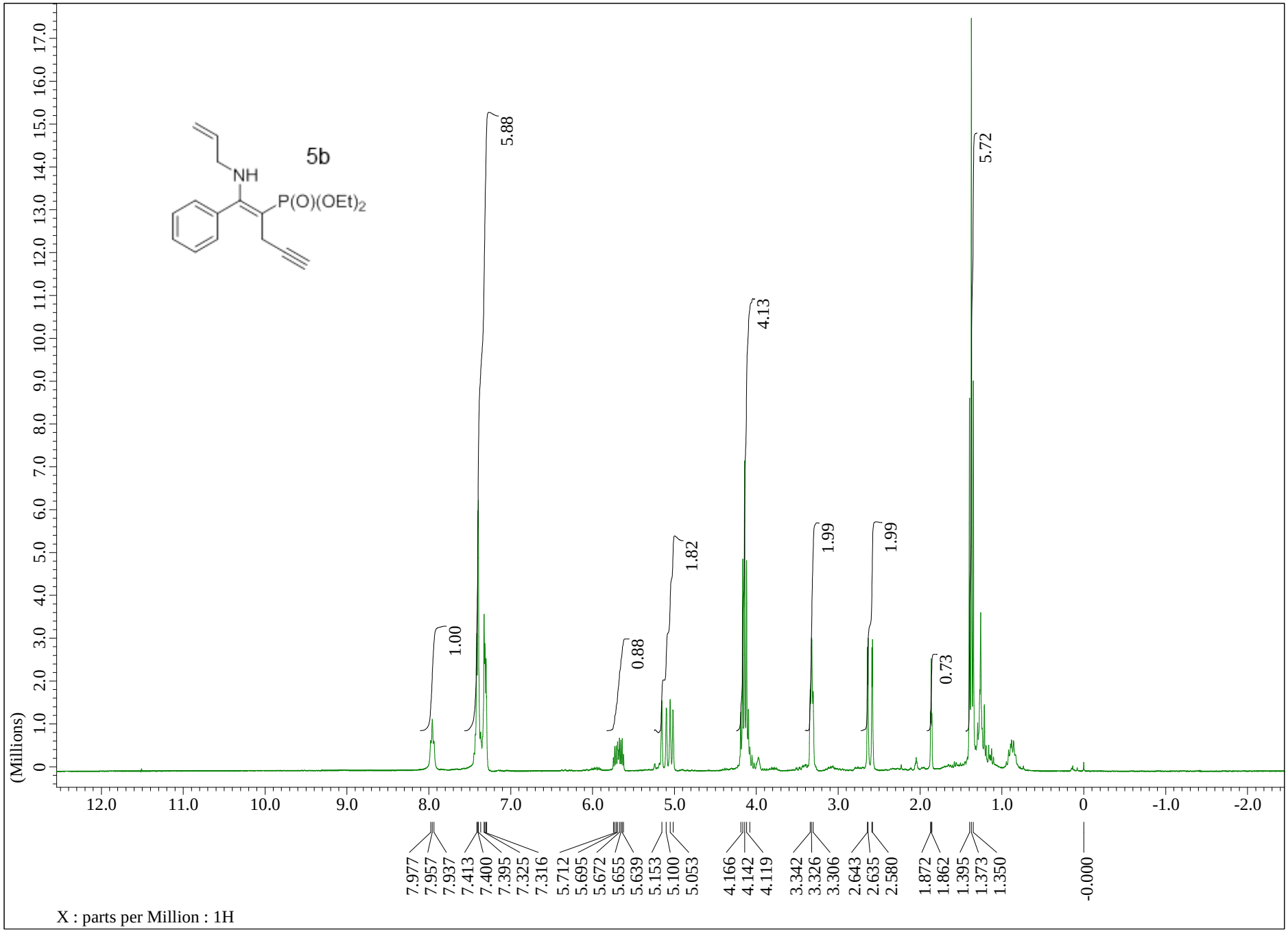


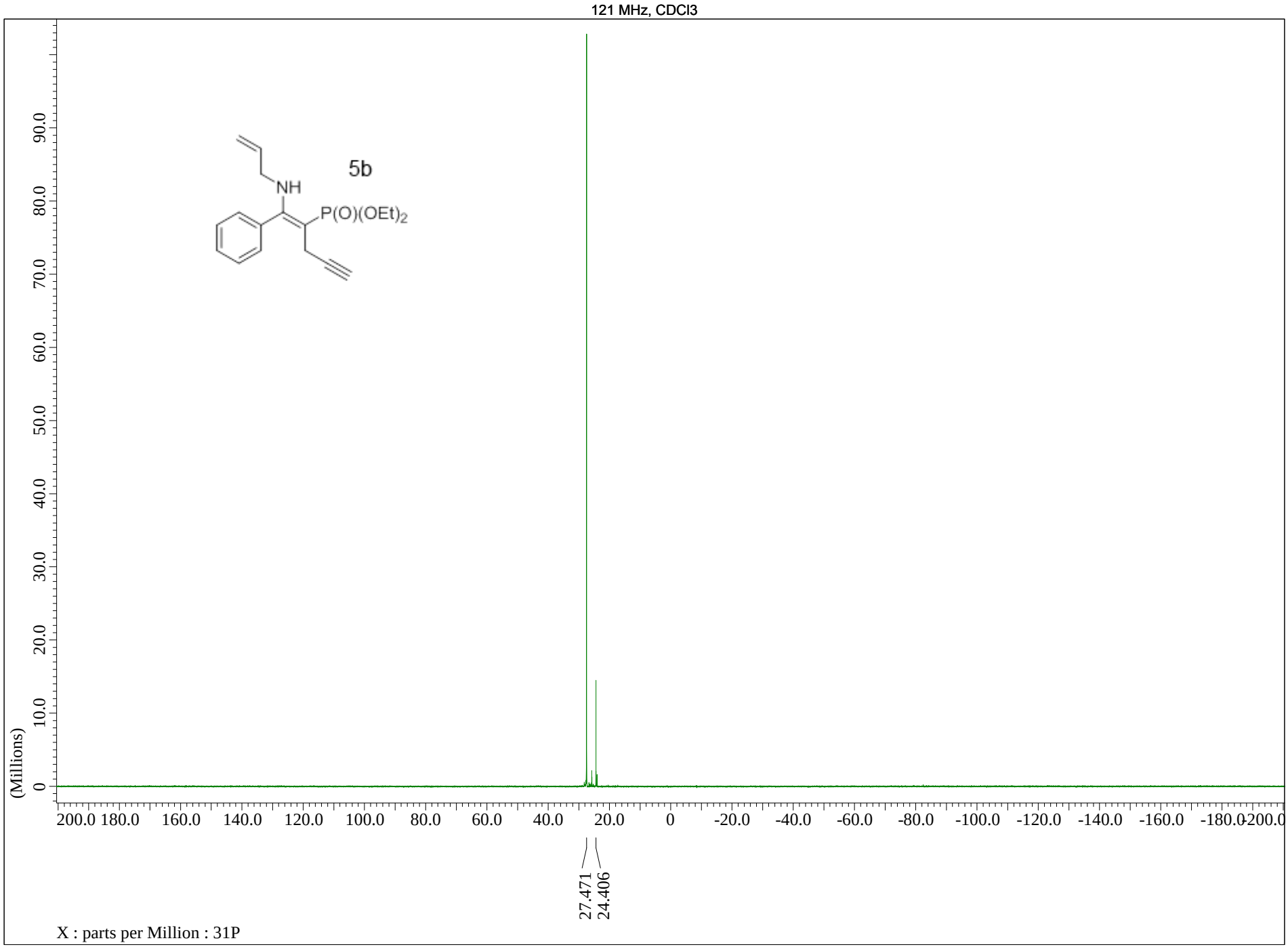

75 MHz, CDCl3
5a
NH
P(O)(OEt)2
(Millions)
0 0.1 0.2 0.3 0.4 0.5 0.6 0.7 0.8 0.9 1.0 1.1 1.2 1.3 1.4 1.5 1.6 1.7 1.8 1.9 2.0
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0 -20.0
165.103
140.125
134.918
134.689
128.887
128.521
128.414
127.101
126.918
84.839
80.533
77.632
77.205
76.777
67.036
61.372
61.311
48.546
18.621
18.484
16.407
16.316
X : parts per Million : 13C

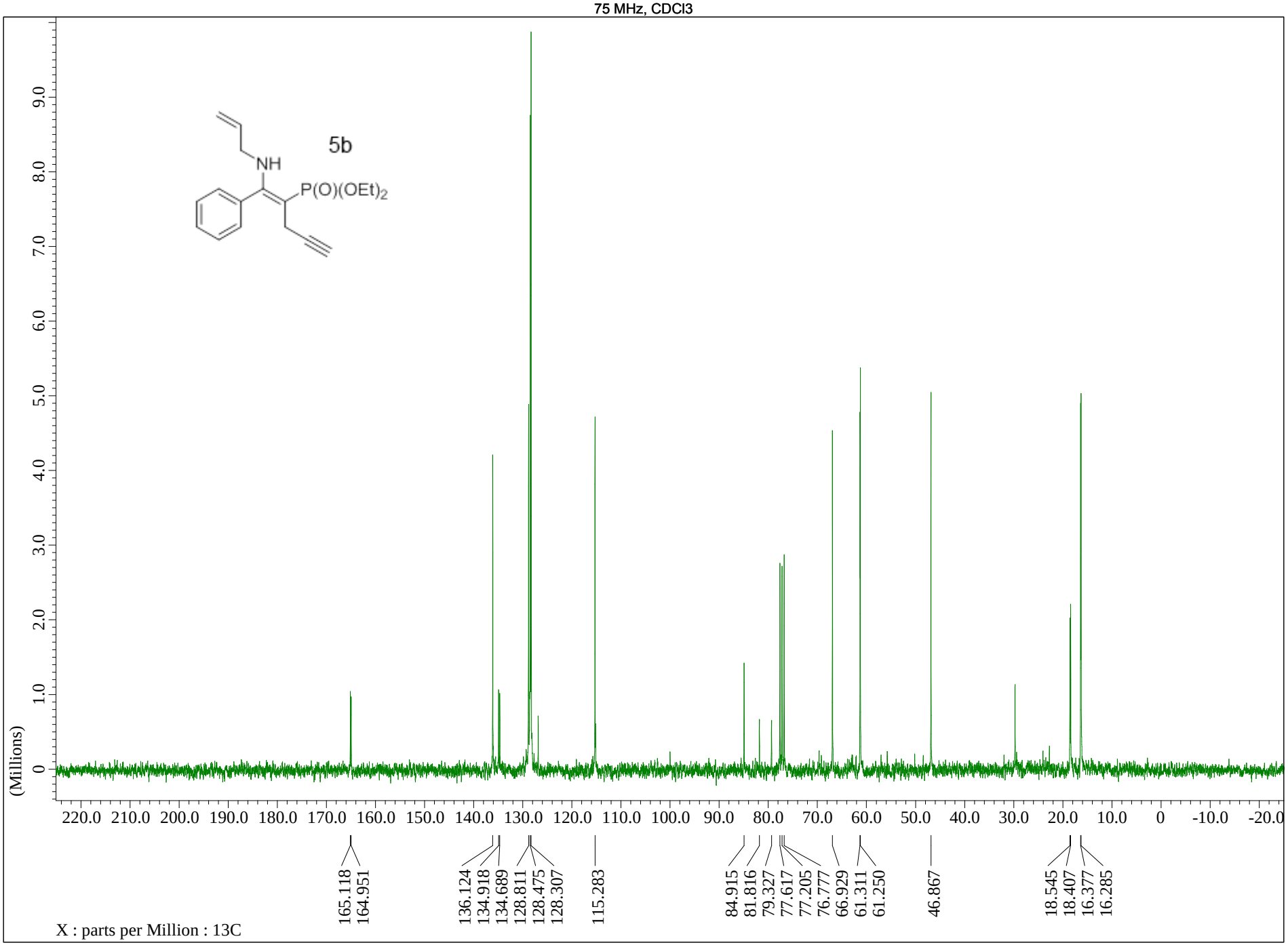
Current Chromatogram(s)



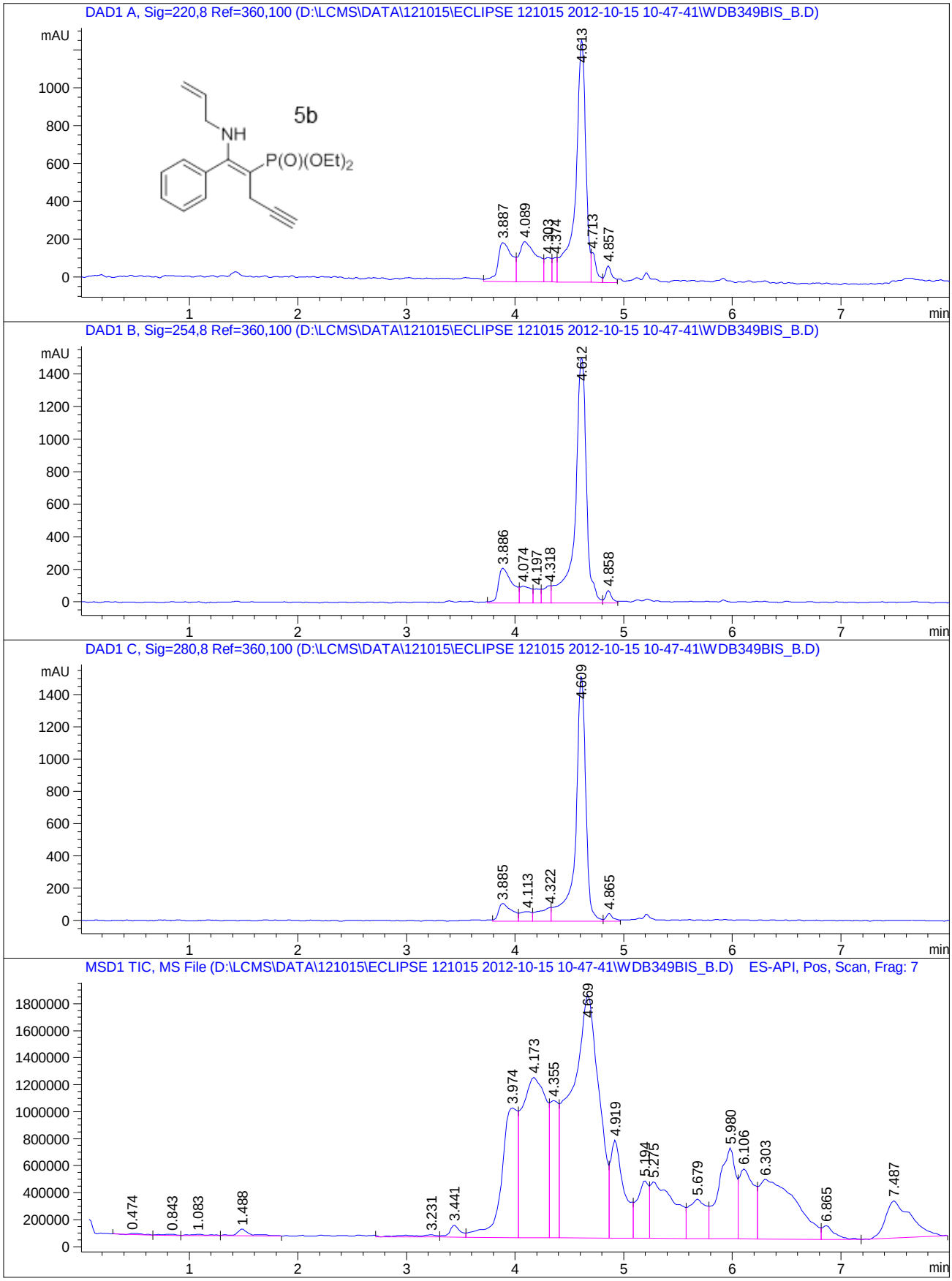
300 MHz, CDCl3

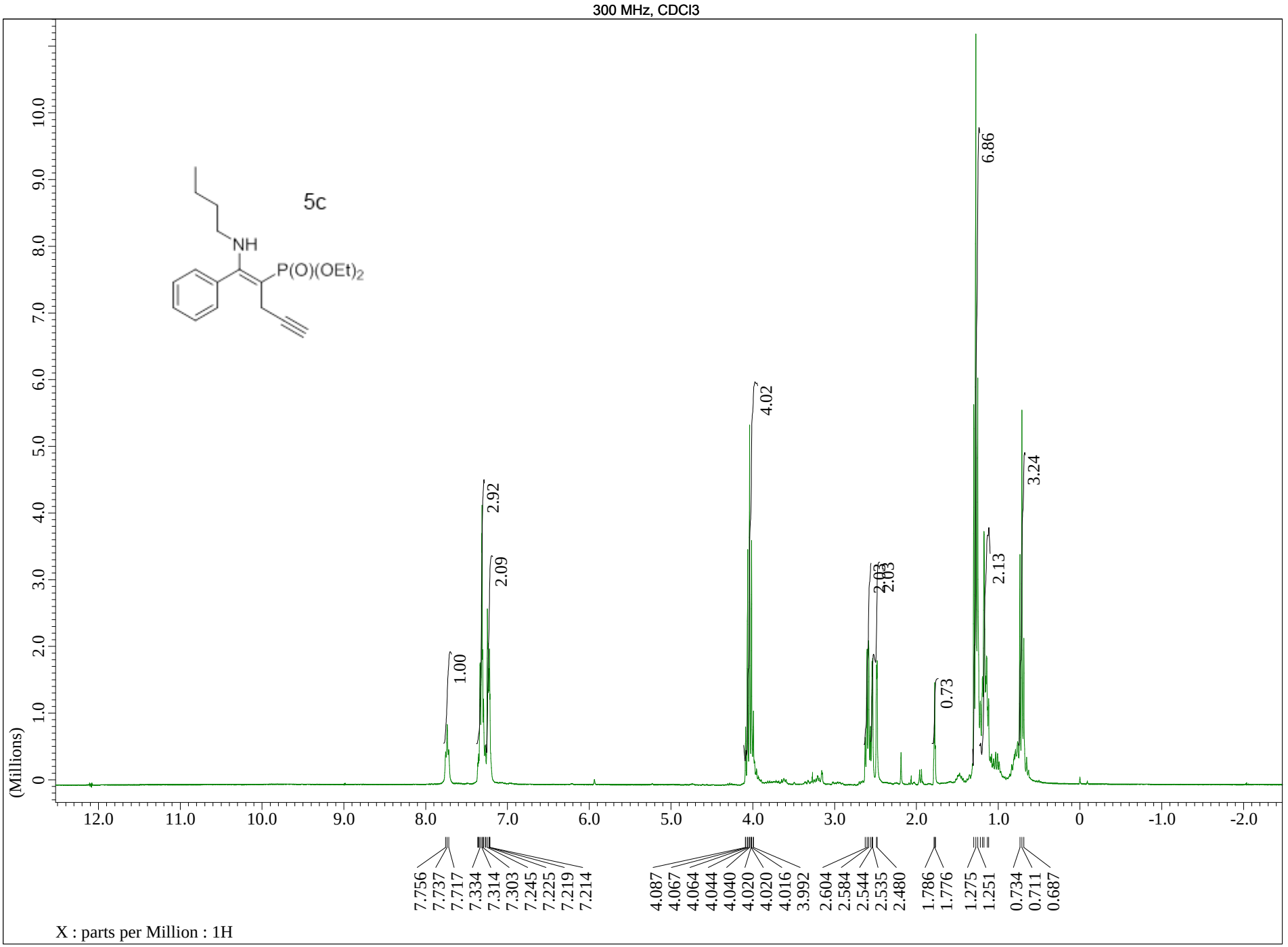



121 MHz, CDCl3
5b
NH
P(O)(OEt)2
(Millions)
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
200.0 180.0 160.0 140.0 120.0 100.0 80.0 60.0 40.0 20.0 0 -20.0 -40.0 -60.0 -80.0 -100.0 -120.0 -140.0 -160.0 -180.0 -200.0
27.471
24.406
X : parts per Million : 31P

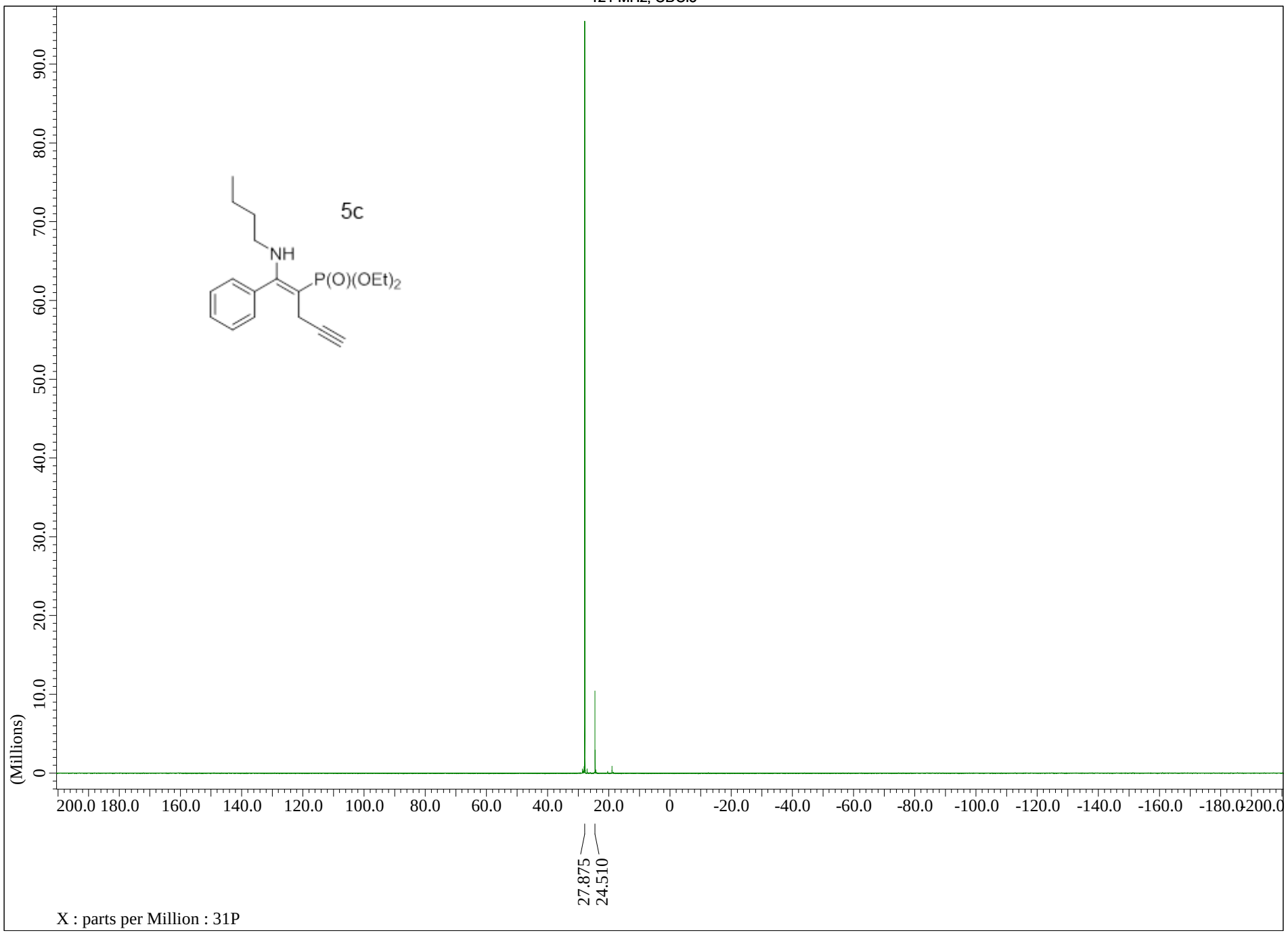

75 MHz, CDCl3
5b
NH
P(O)(OEt)2
(Millions)
0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0 -20.0
165.118
164.951
136.124
134.918
134.689
128.811
128.475
128.307
115.283
84.915
81.816
79.327
77.617
77.205
76.777
66.929
61.311
61.250
46.867
18.545
18.407
16.377
16.285
X : parts per Million : 13C

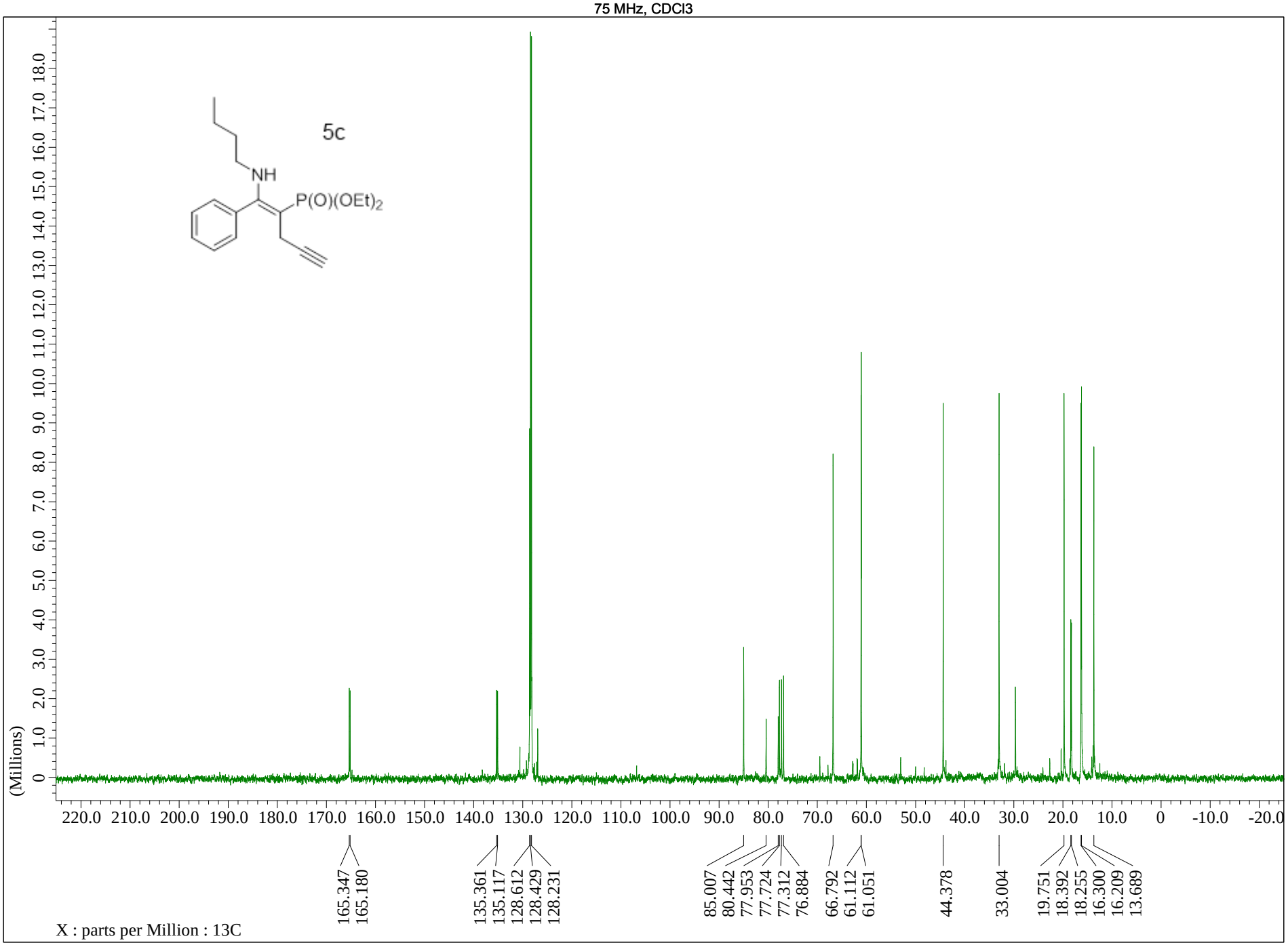
Current Chromatogram(s)



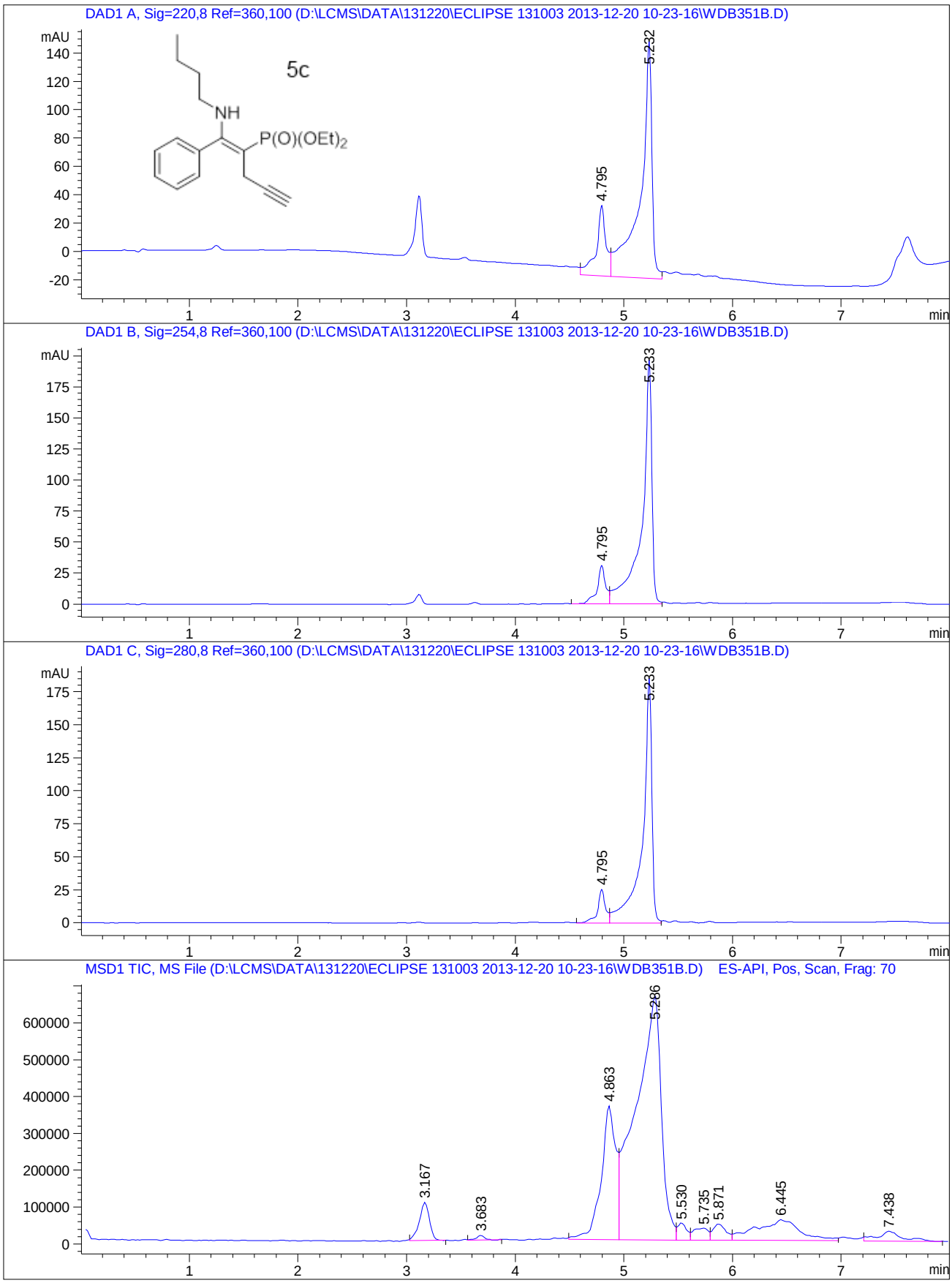

300 MHz, CDCl3
5c
NH
P(O)(OEt)2
(Millions)
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
-1.0
-2.0
1.00
2.92
2.09
4.02
2.03
2.03
0.73
6.86
2.13
3.24
7.756
7.737
7.717
7.334
7.314
7.303
7.245
7.225
7.219
7.214
4.087
4.067
4.064
4.044
4.040
4.020
4.020
4.016
3.992
2.604
2.584
2.544
2.535
2.480
1.786
1.776
1.275
1.251
0.734
0.711
0.687
X : parts per Million : 1H

121 MHz, CDCl3

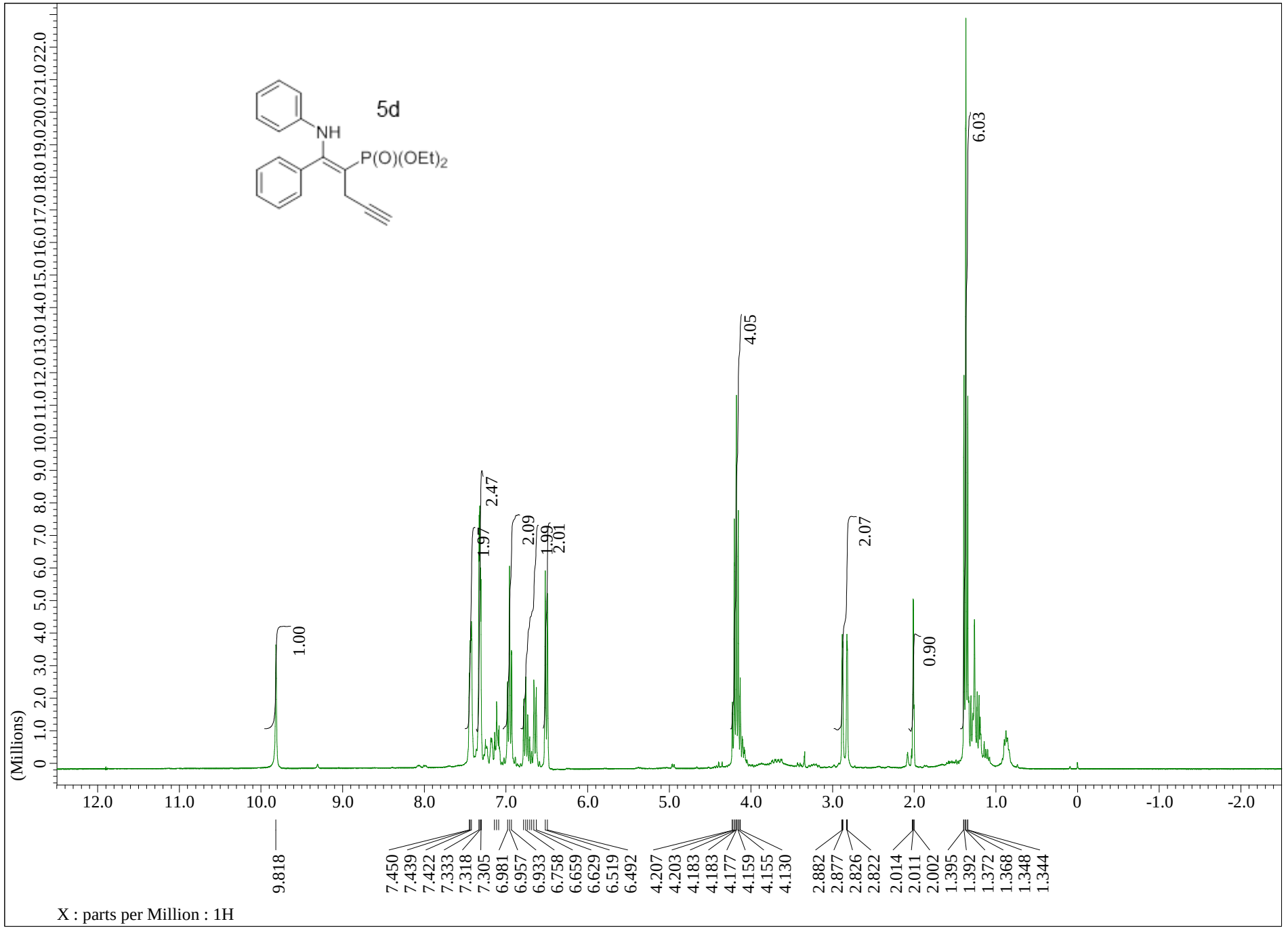



75 MHz, CDCl3
5c
NH
P(O)(OEt)2
(Millions)
0 1.0 2.0 3.0 4.0 5.0 6.0 7.0 8.0 9.0 10.0 11.0 12.0 13.0 14.0 15.0 16.0 17.0 18.0
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0 -20.0
165.347
165.180
135.361
135.117
128.612
128.429
128.231
85.007
80.442
77.953
77.724
77.312
76.884
66.792
61.112
61.051
44.378
33.004
19.751
18.392
18.255
16.300
16.209
13.689
X : parts per Million : 13C

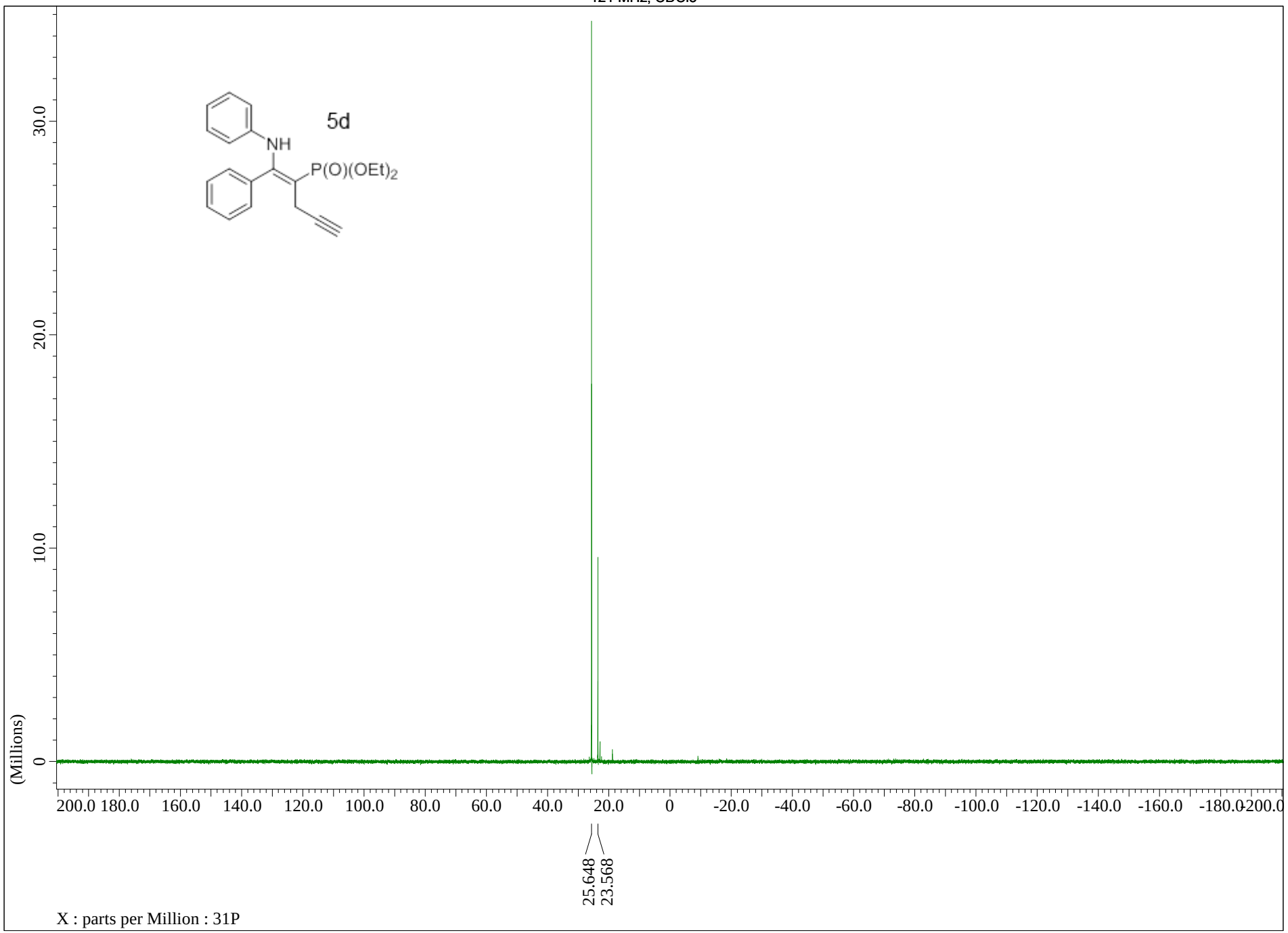
Current Chromatogram(s)

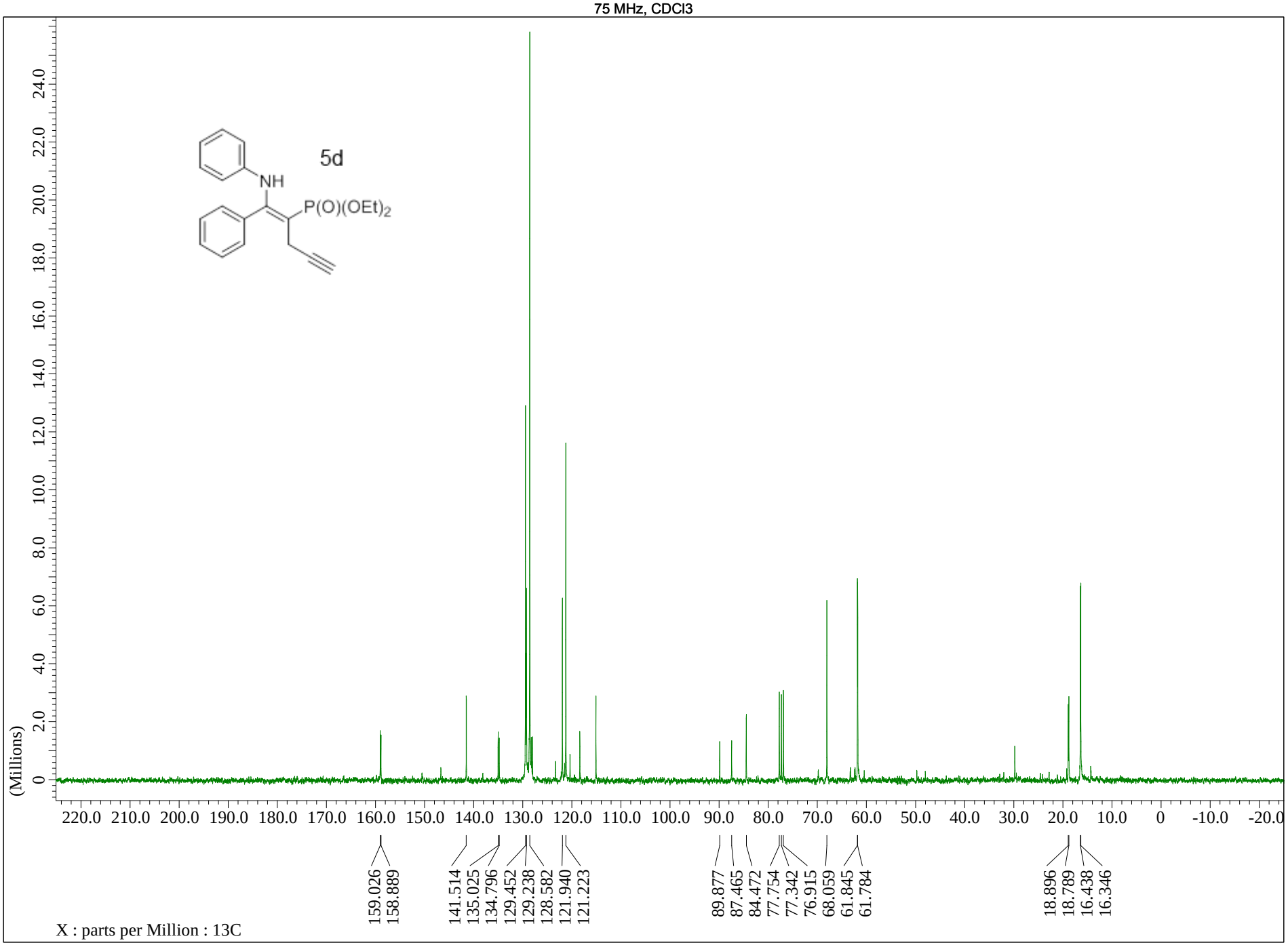


300 MHz, CDCl3

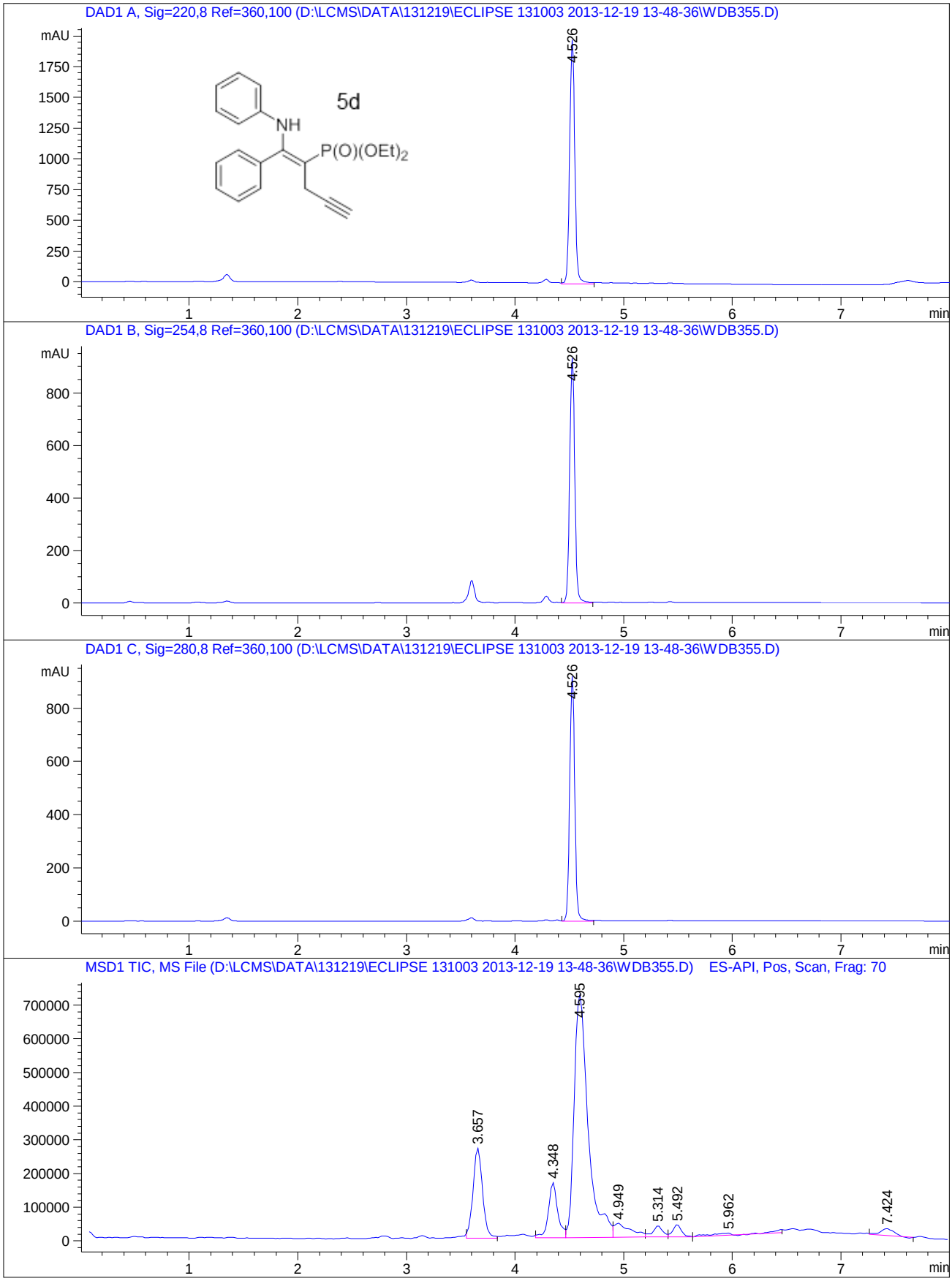


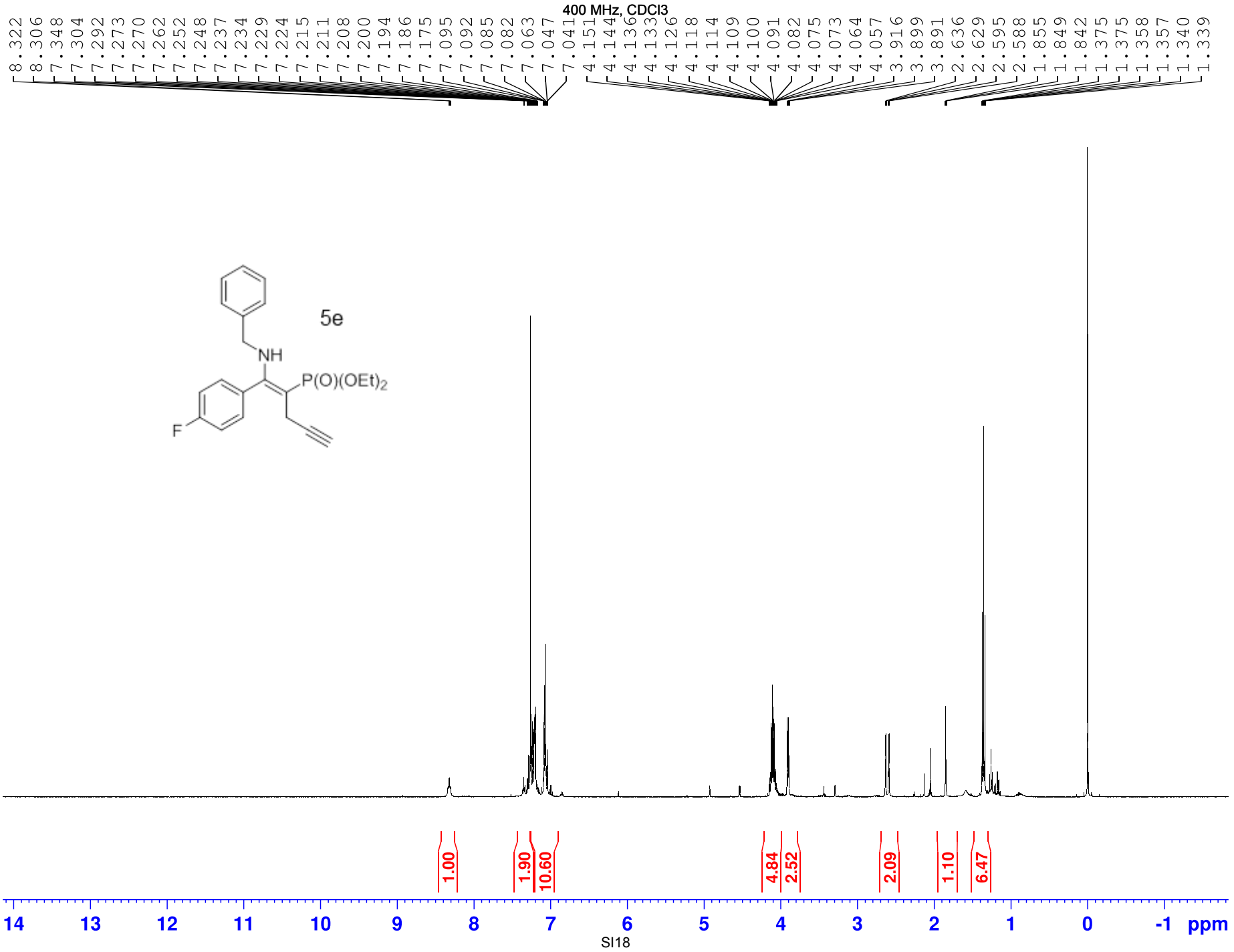
121 MHz, CDCl3




75 MHz, CDCl3
5d
NH
P(O)(OEt)2
(Millions)
0
2.0
4.0
6.0
8.0
10.0
12.0
14.0
16.0
18.0
20.0
22.0
24.0
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0 -20.0
159.026
158.889
141.514
135.025
134.796
129.452
129.238
128.582
121.940
121.223
89.877
87.465
84.472
77.754
77.342
76.915
68.059
61.845
61.784
18.896
18.789
16.438
16.346
X : parts per Million : 13C

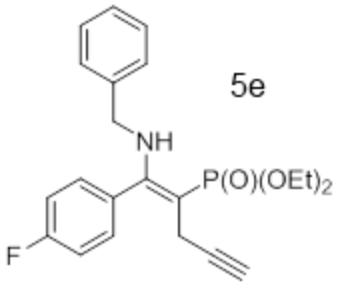
Current Chromatogram(s)




400 MHz, CDCl3
8.322
8.306
7.348
7.304
7.292
7.273
7.270
7.262
7.252
7.248
7.237
7.234
7.229
7.224
7.215
7.211
7.208
7.200
7.194
7.186
7.175
7.095
7.092
7.085
7.082
7.063
7.047
7.041
4.151
4.144
4.136
4.133
4.126
4.118
4.114
4.109
4.100
4.091
4.082
4.075
4.073
4.064
4.057
3.916
3.899
3.891
2.636
2.629
2.595
2.588
1.855
1.849
1.842
1.375
1.375
1.358
1.357
1.340
1.339
5e
NH
P(O)(OEt)2
F
1.00
1.90
10.60
4.84
2.52
2.09
1.10
6.47
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm

162 MHz, CDCl3

26.23
23.45

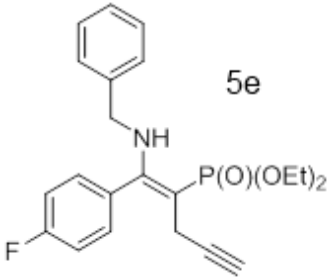


5e

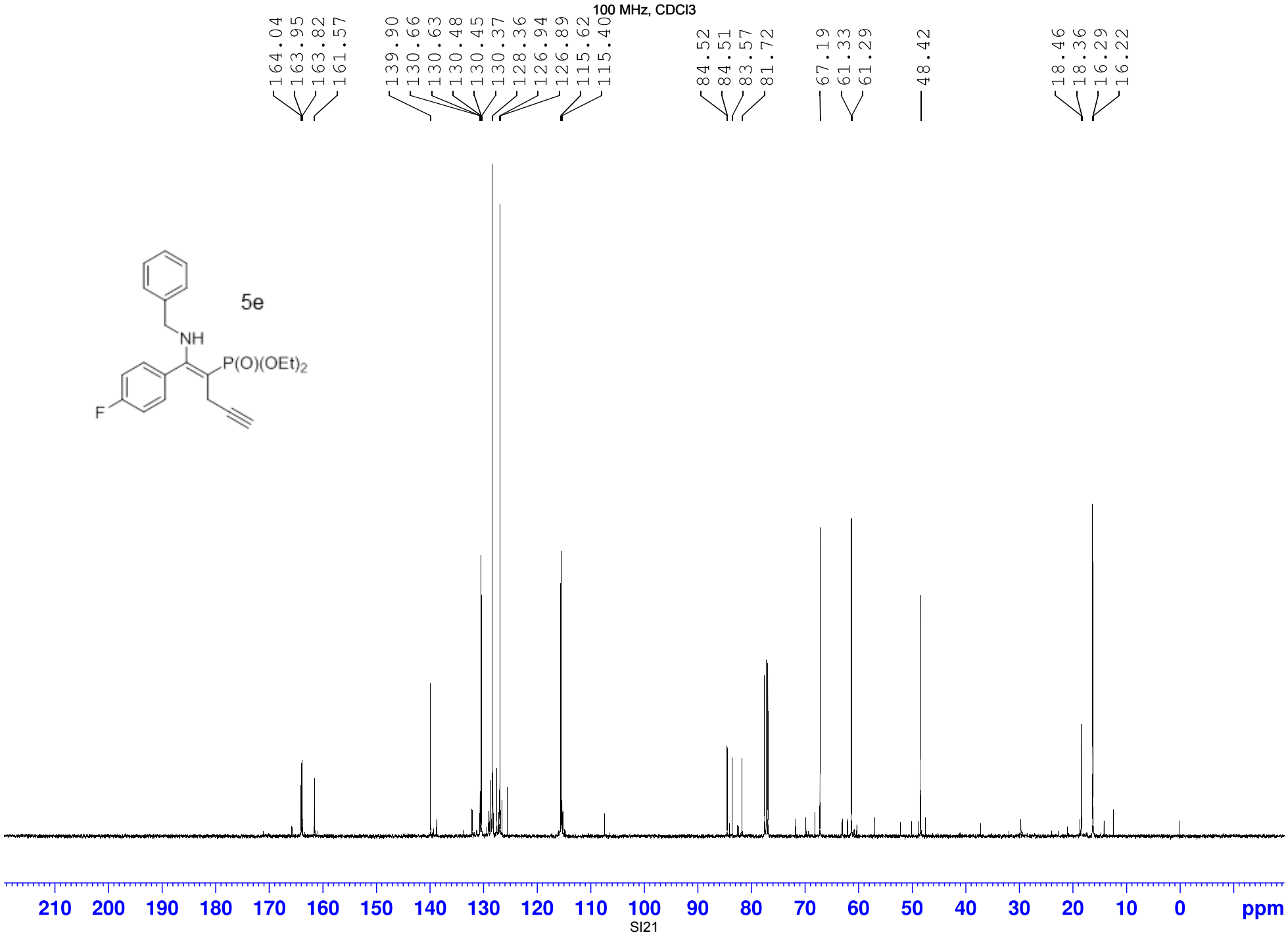
100 50 0 -50 -100 -150 -200 ppm

376 MHz, CDCl3

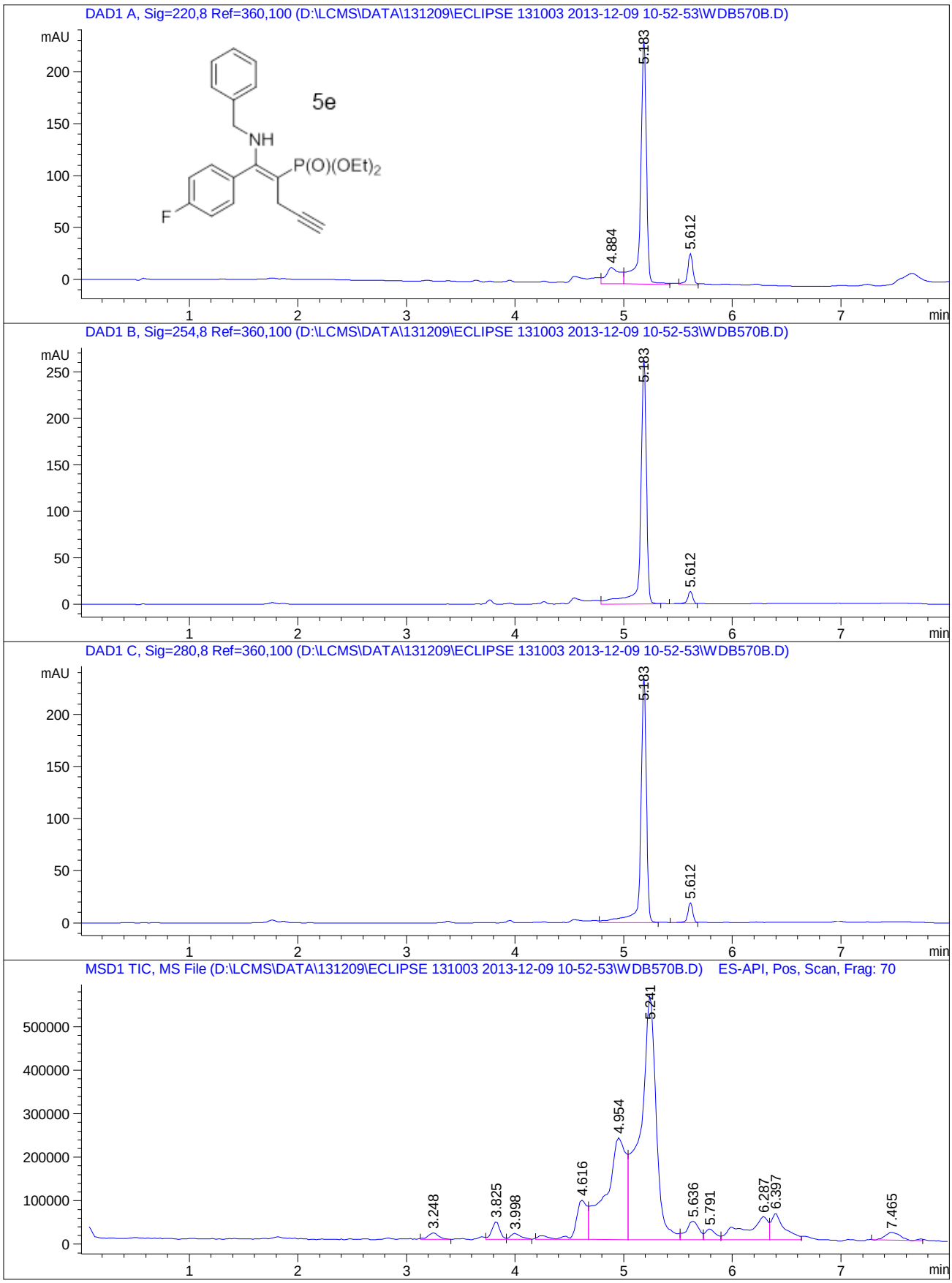
-111.99
-112.00
-112.01
-112.01
-112.02
-112.04
-112.05
-112.06
-112.08
-112.09
-112.10
-112.12
-112.12
-114.45



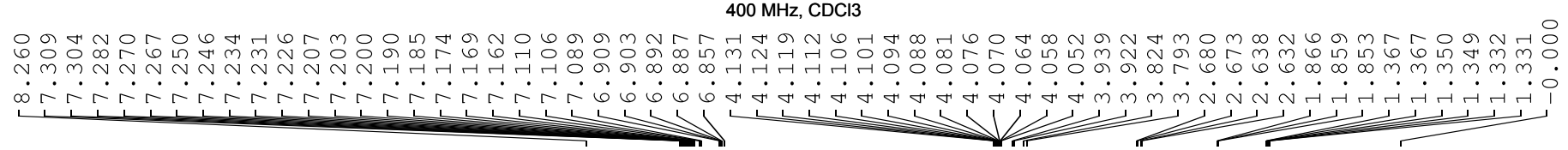
5e

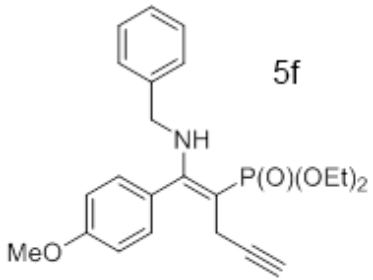

100 MHz, CDCl3
164.04
163.95
163.82
161.57
139.90
130.66
130.63
130.48
130.45
130.37
128.36
126.94
126.89
115.62
115.40
84.52
84.51
83.57
81.72
67.19
61.33
61.29
48.42
18.46
18.36
16.29
16.22
5e
NH
P(O)(OEt)2
F
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

Current Chromatogram(s)



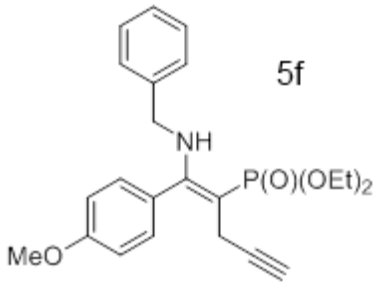
400 MHz, CDCl3





162 MHz, CDCl3

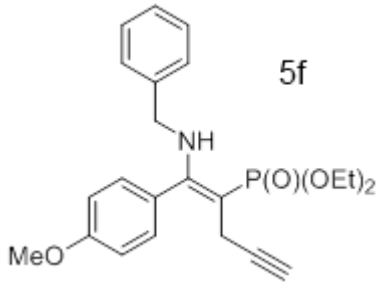
26.82
23.91



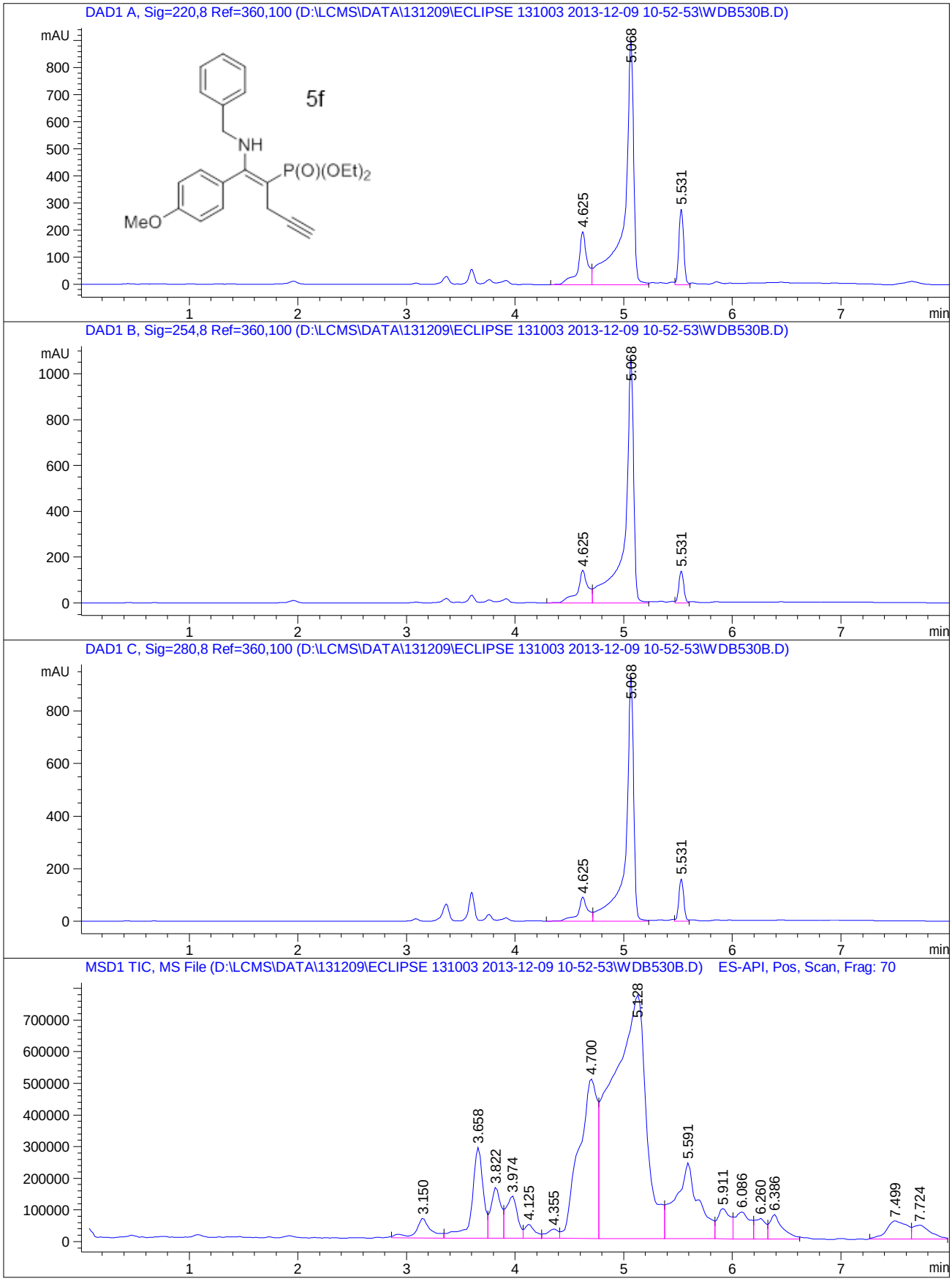
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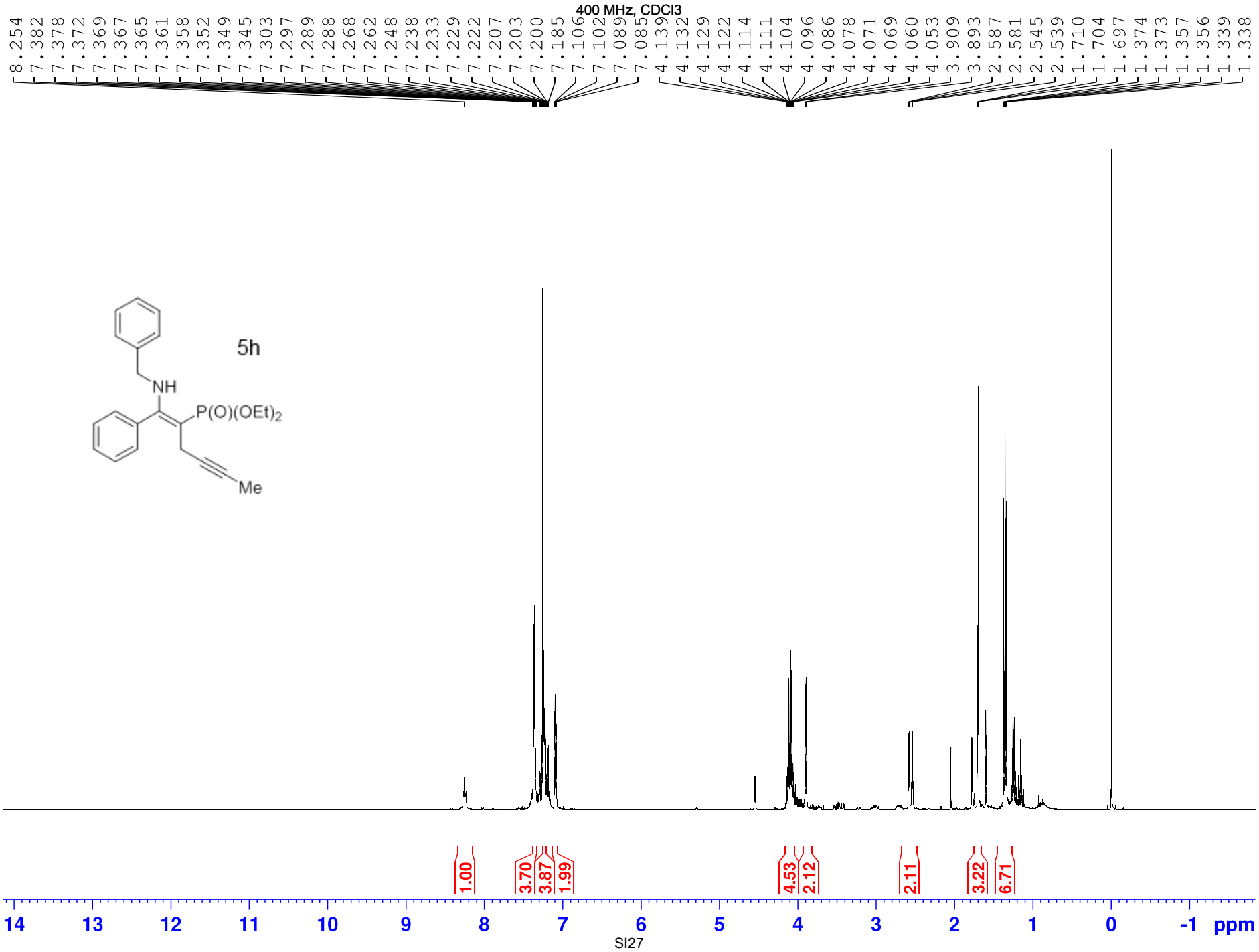
100 MHz, CDCl3

164.97
164.84
159.85
140.08
129.77
128.27
127.00
126.77
126.72
113.76
113.72
84.86
84.85
82.99
81.15
67.10
61.15
61.11
55.09
48.48
18.62
18.52
16.29
16.22



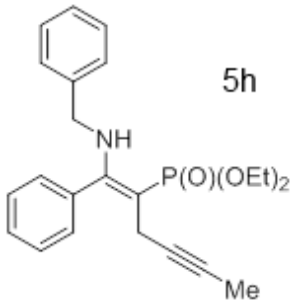
Current Chromatogram(s)




400 MHz, CDCl3
8.254
7.382
7.378
7.372
7.369
7.367
7.365
7.361
7.358
7.352
7.349
7.345
7.303
7.297
7.289
7.288
7.268
7.262
7.248
7.238
7.233
7.229
7.222
7.207
7.203
7.200
7.185
7.106
7.102
7.089
7.085
4.139
4.132
4.129
4.122
4.114
4.111
4.104
4.096
4.086
4.078
4.071
4.069
4.060
4.053
3.909
3.893
2.587
2.581
2.545
2.539
1.710
1.704
1.697
1.374
1.373
1.357
1.356
1.339
1.338
5h
NH
P(O)(OEt)2
Me
1.00
3.70
3.87
1.99
4.53
2.12
2.11
3.22
6.71
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm

162 MHz, CDCl3

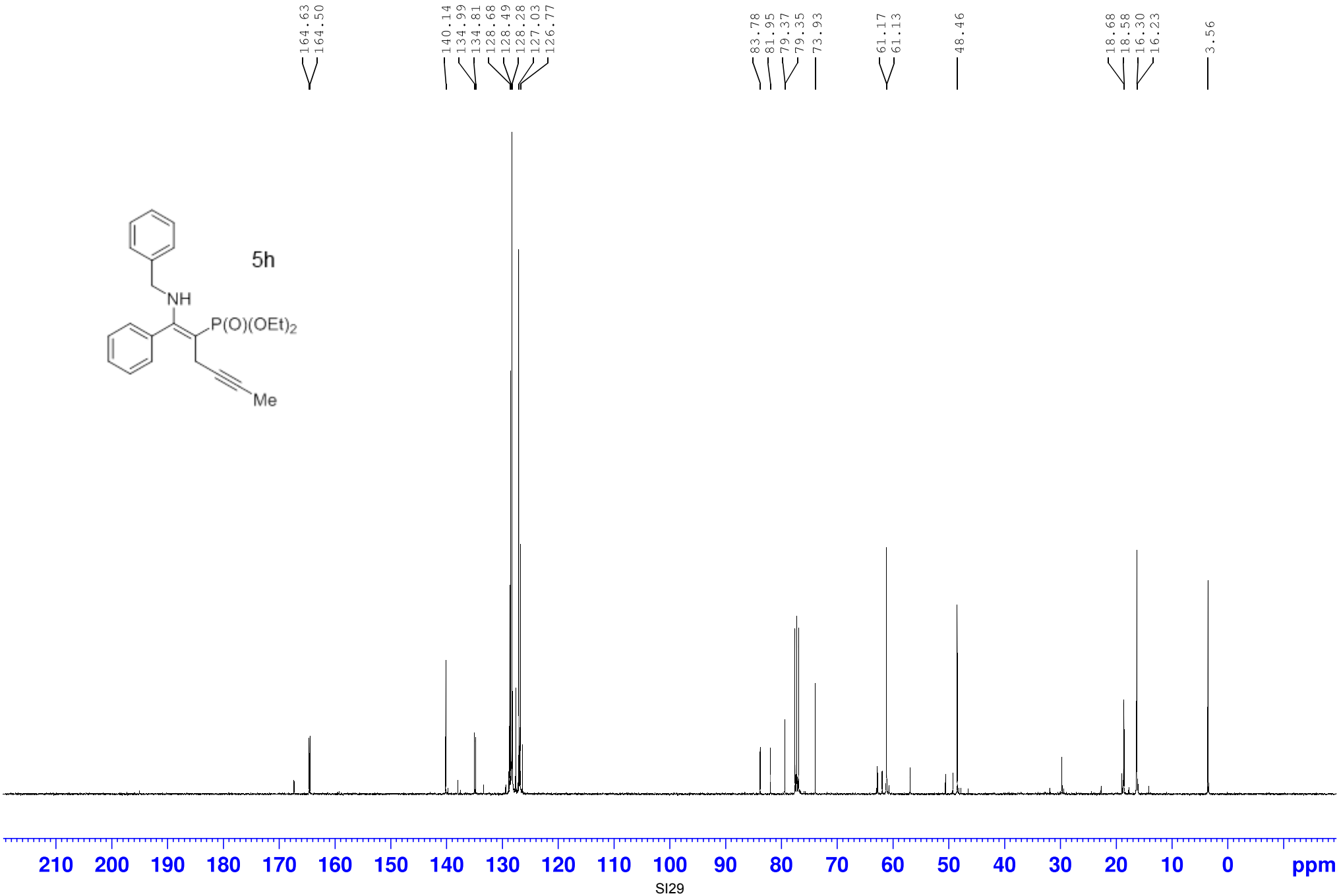
27.17
24.27



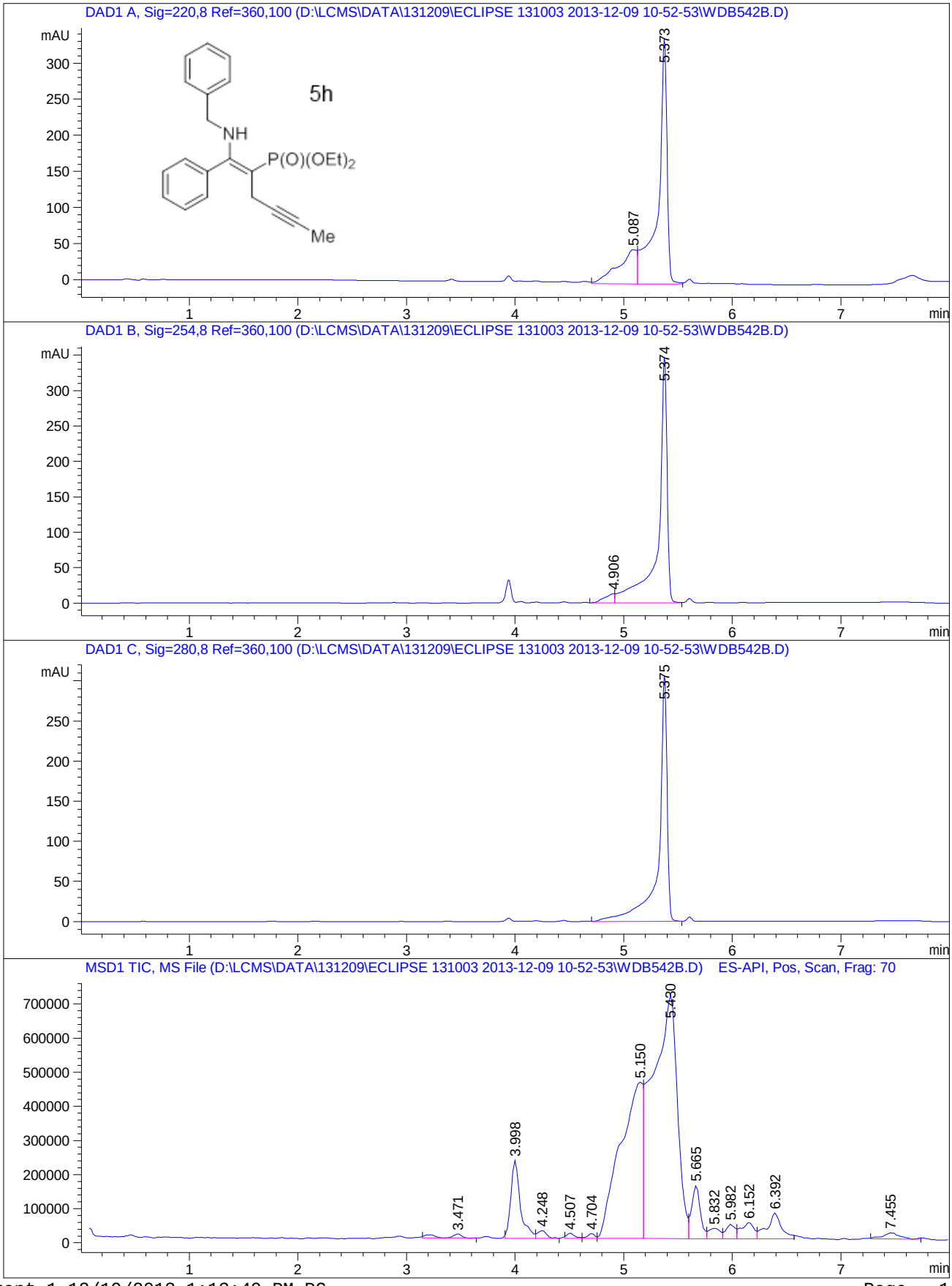
5h

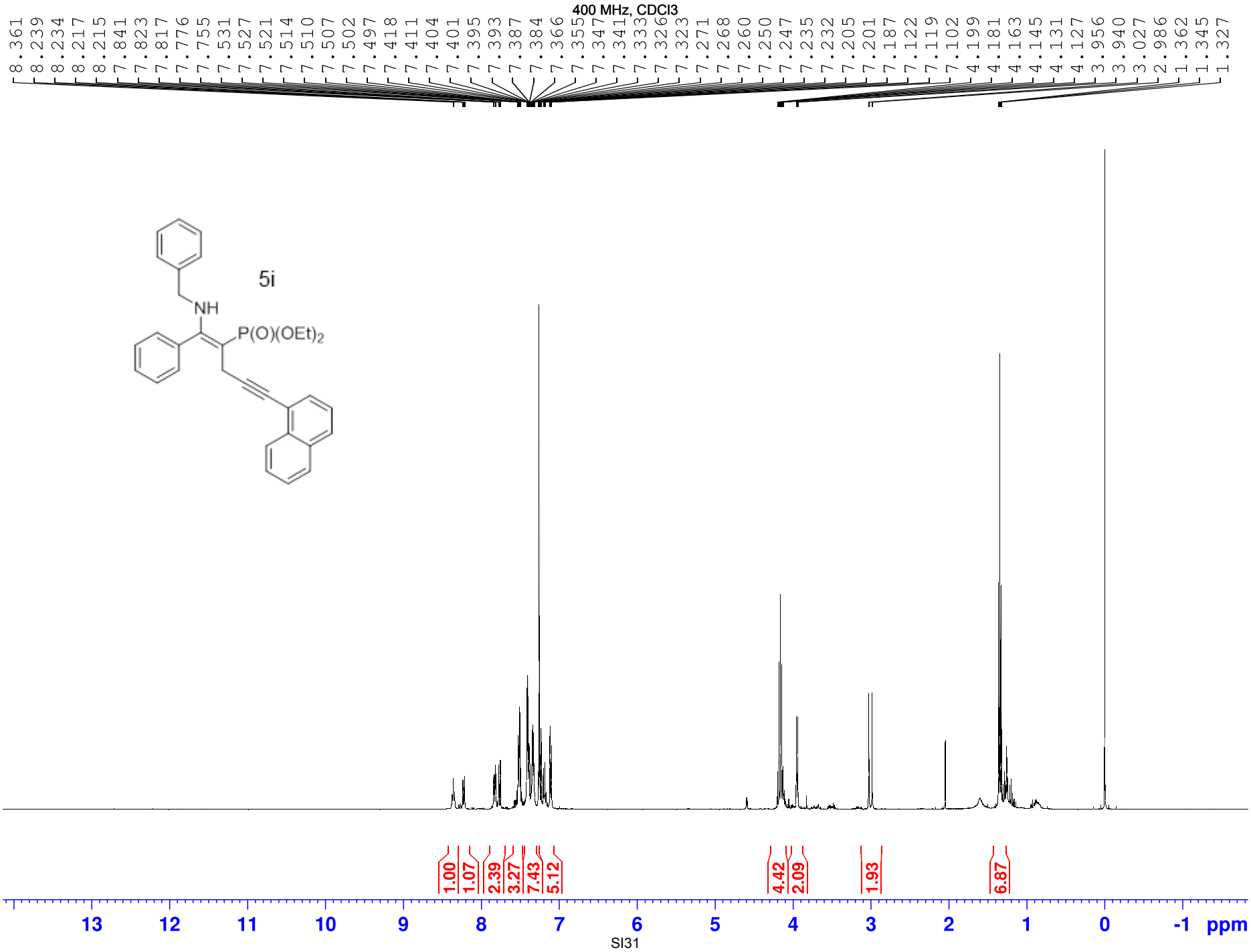
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100 MHz, CDCl3



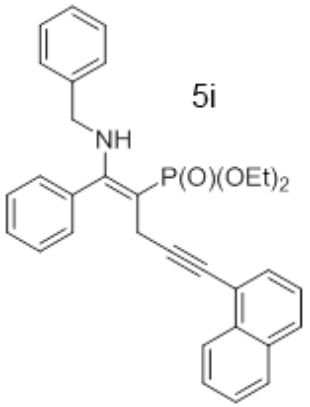
Current Chromatogram(s)




400 MHz, CDCl3
8.361
8.239
8.234
8.217
8.215
7.841
7.823
7.817
7.776
7.755
7.531
7.527
7.521
7.514
7.510
7.507
7.502
7.497
7.418
7.411
7.404
7.401
7.395
7.393
7.387
7.384
7.366
7.355
7.347
7.341
7.333
7.326
7.323
7.271
7.268
7.260
7.250
7.247
7.235
7.232
7.205
7.201
7.187
7.122
7.119
7.102
4.199
4.181
4.163
4.145
4.131
4.127
3.956
3.940
3.027
2.986
1.362
1.345
1.327
5i
NH
P(O)(OEt)2
1.00
1.07
2.39
3.27
7.43
5.12
4.42
2.09
1.93
6.87
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm

162 MHz, CDCl3

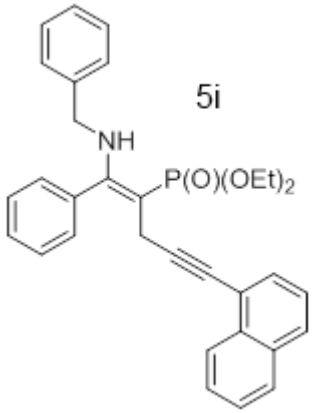
26.96
23.88



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

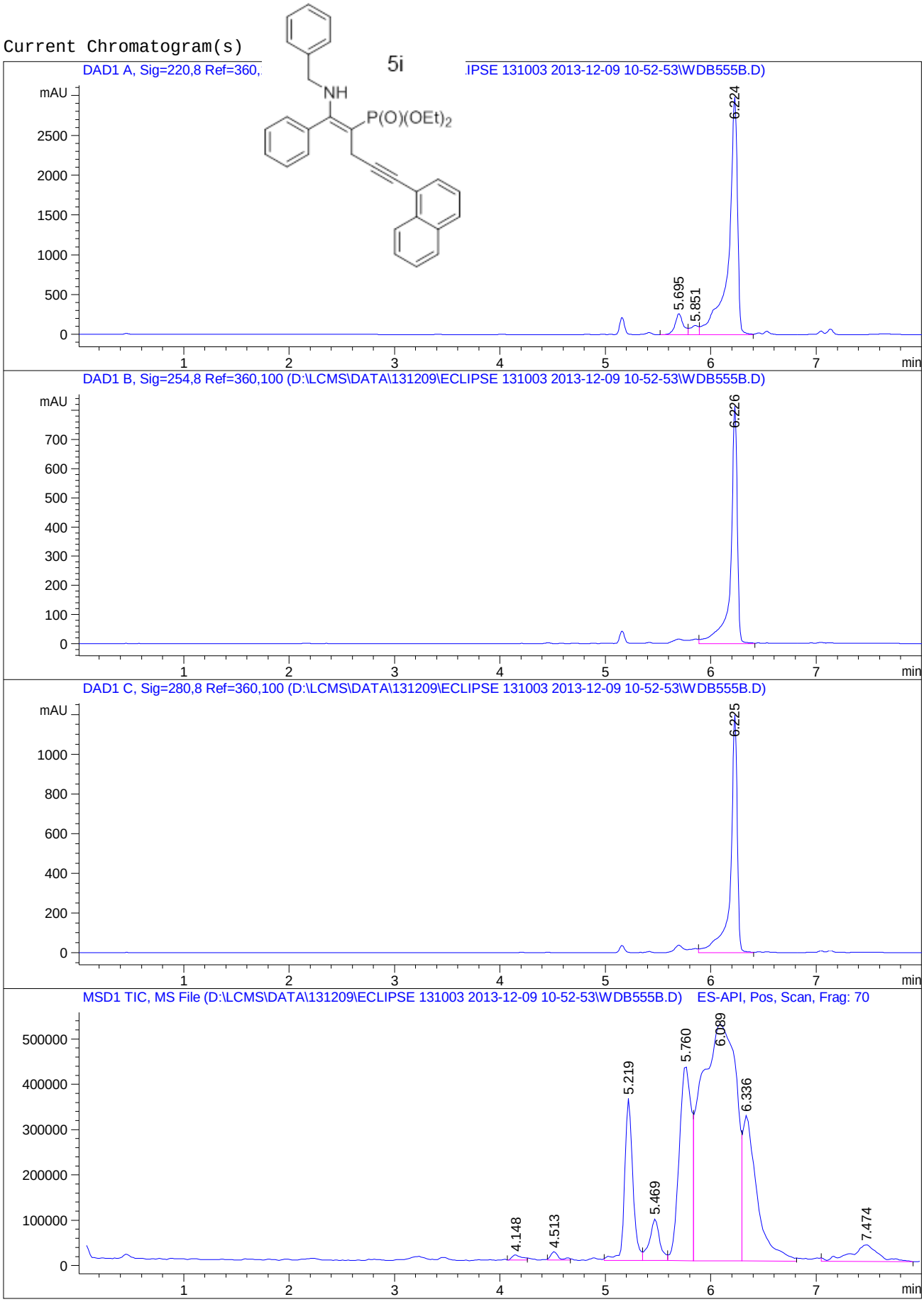
100 MHz, CDCl3

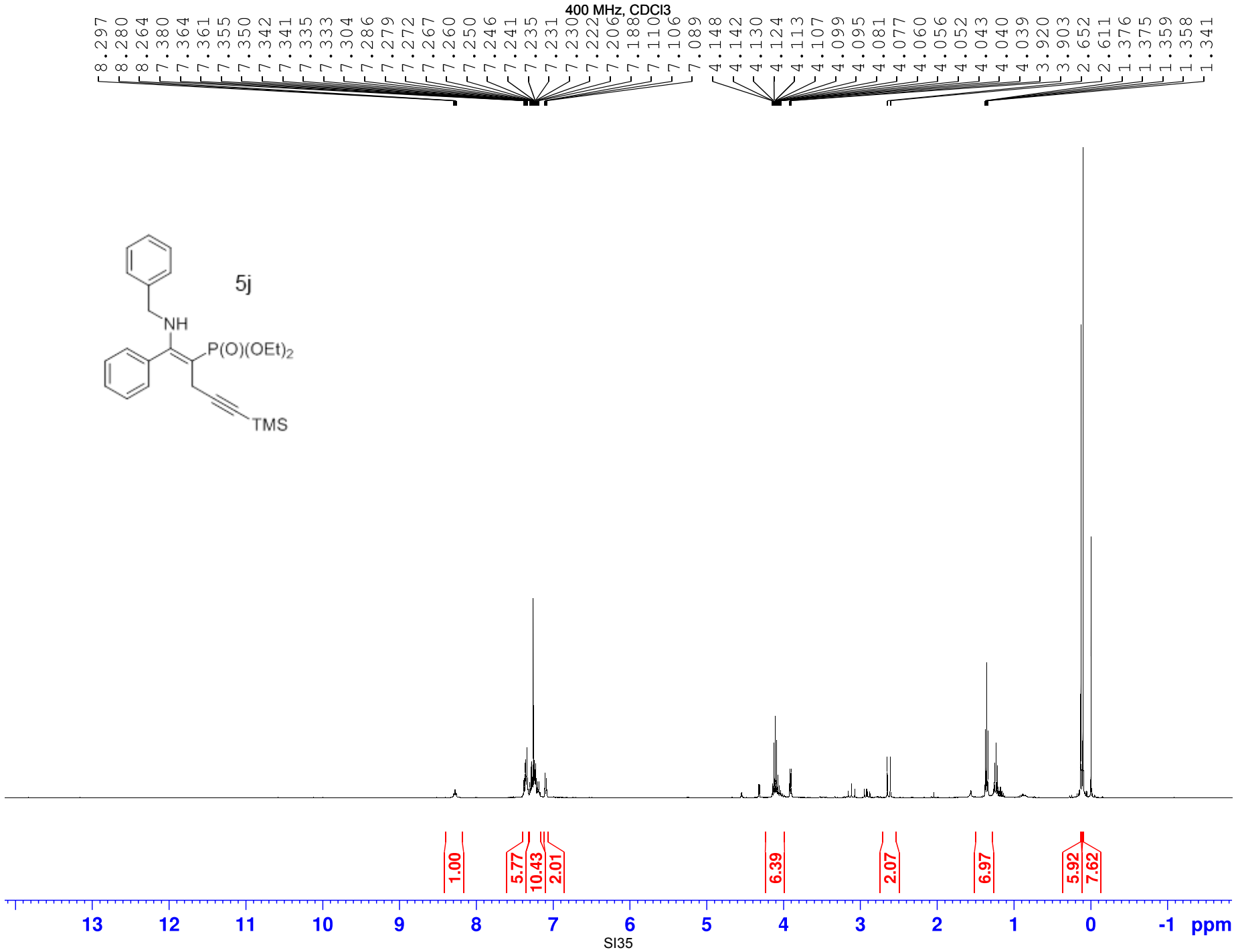
165.24
165.12
140.15
135.02
134.85
133.67
133.29
129.81
128.96
128.62
128.61
128.46
128.30
127.93
127.09
126.97
126.53
126.43
126.35
125.35
122.09
95.84
95.82
83.11
81.26
77.44
61.49
61.44
48.58
19.81
19.71
16.55
16.48



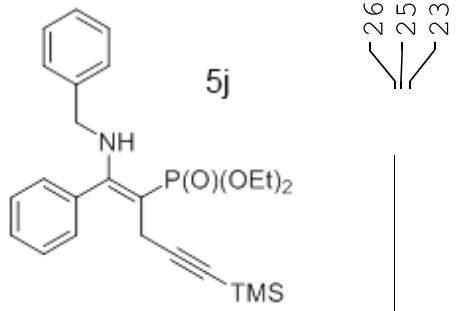
5i

Current Chromatogram(s)



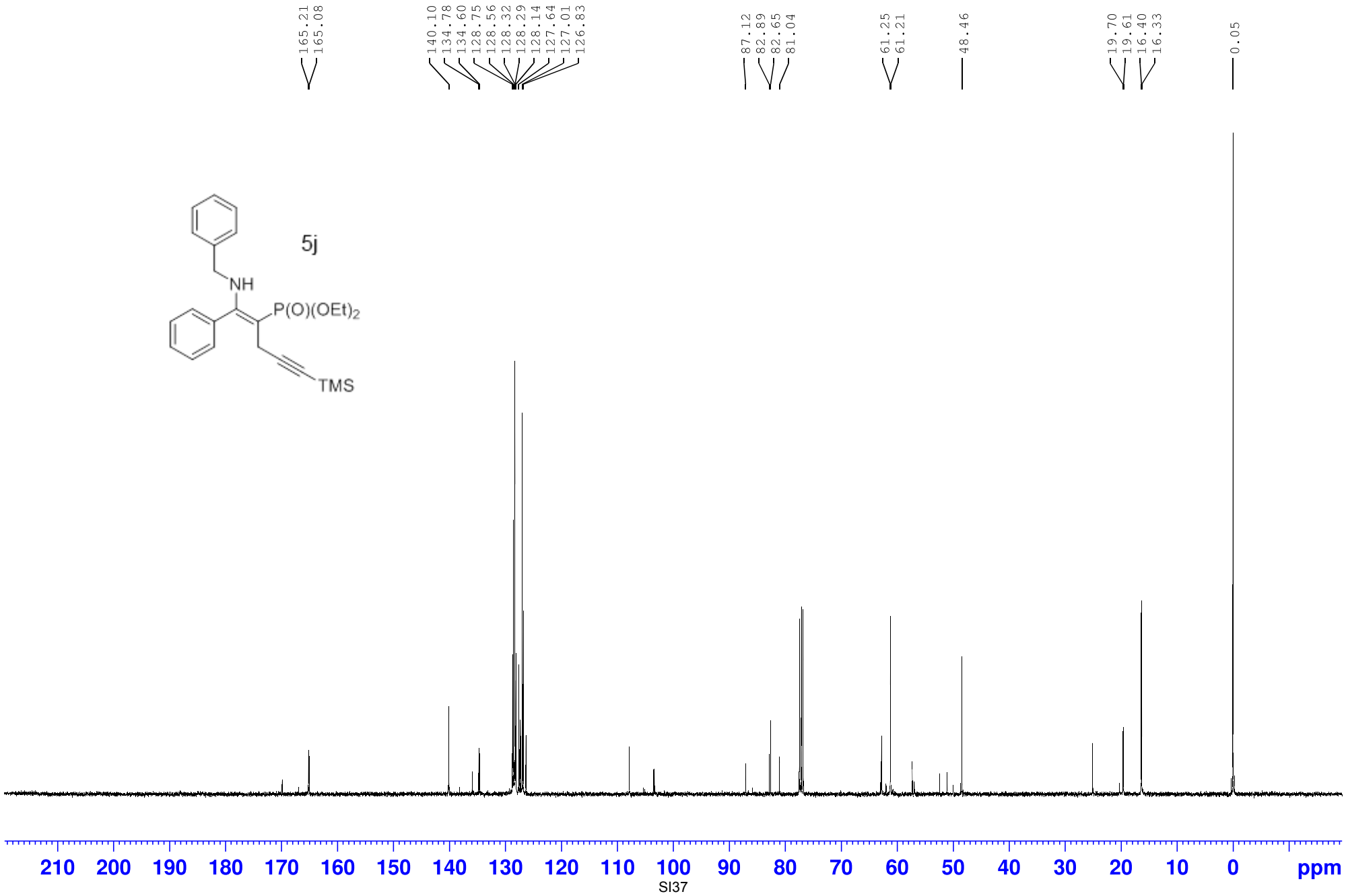

400 MHz, CDCl3
8.297
8.280
8.264
7.380
7.364
7.361
7.355
7.350
7.342
7.341
7.335
7.333
7.304
7.286
7.279
7.272
7.267
7.260
7.250
7.246
7.241
7.235
7.231
7.230
7.222
7.206
7.188
7.110
7.106
7.089
4.148
4.142
4.130
4.124
4.113
4.107
4.099
4.095
4.081
4.077
4.060
4.056
4.052
4.043
4.040
4.039
3.920
3.903
2.652
2.611
1.376
1.375
1.359
1.358
1.341
5j
NH
P(O)(OEt)2
TMS
1.00
5.77
10.43
2.01
6.39
2.07
6.97
5.92
7.62
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm

162 MHz, CDCl3

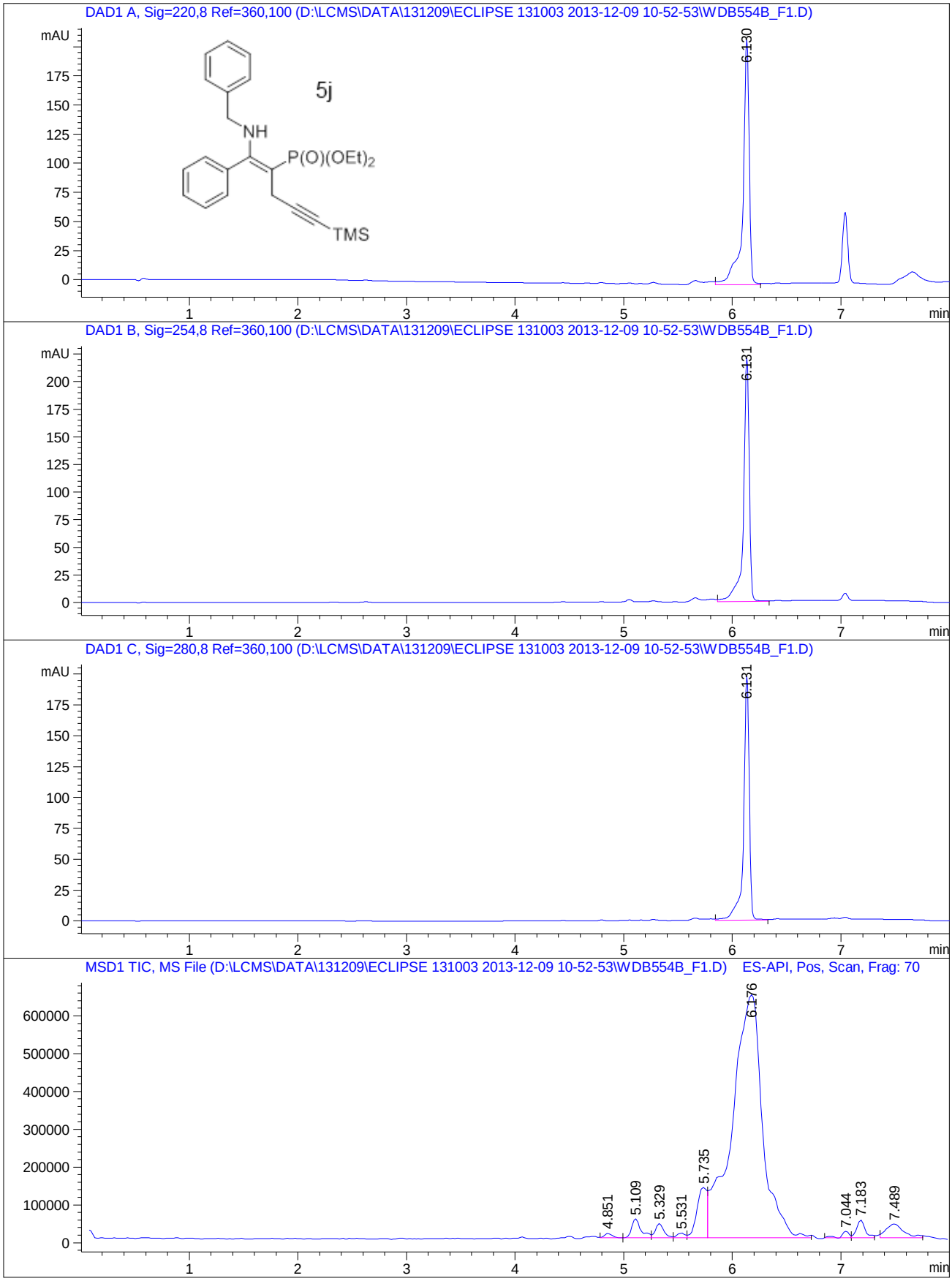


26.83
25.61
23.74

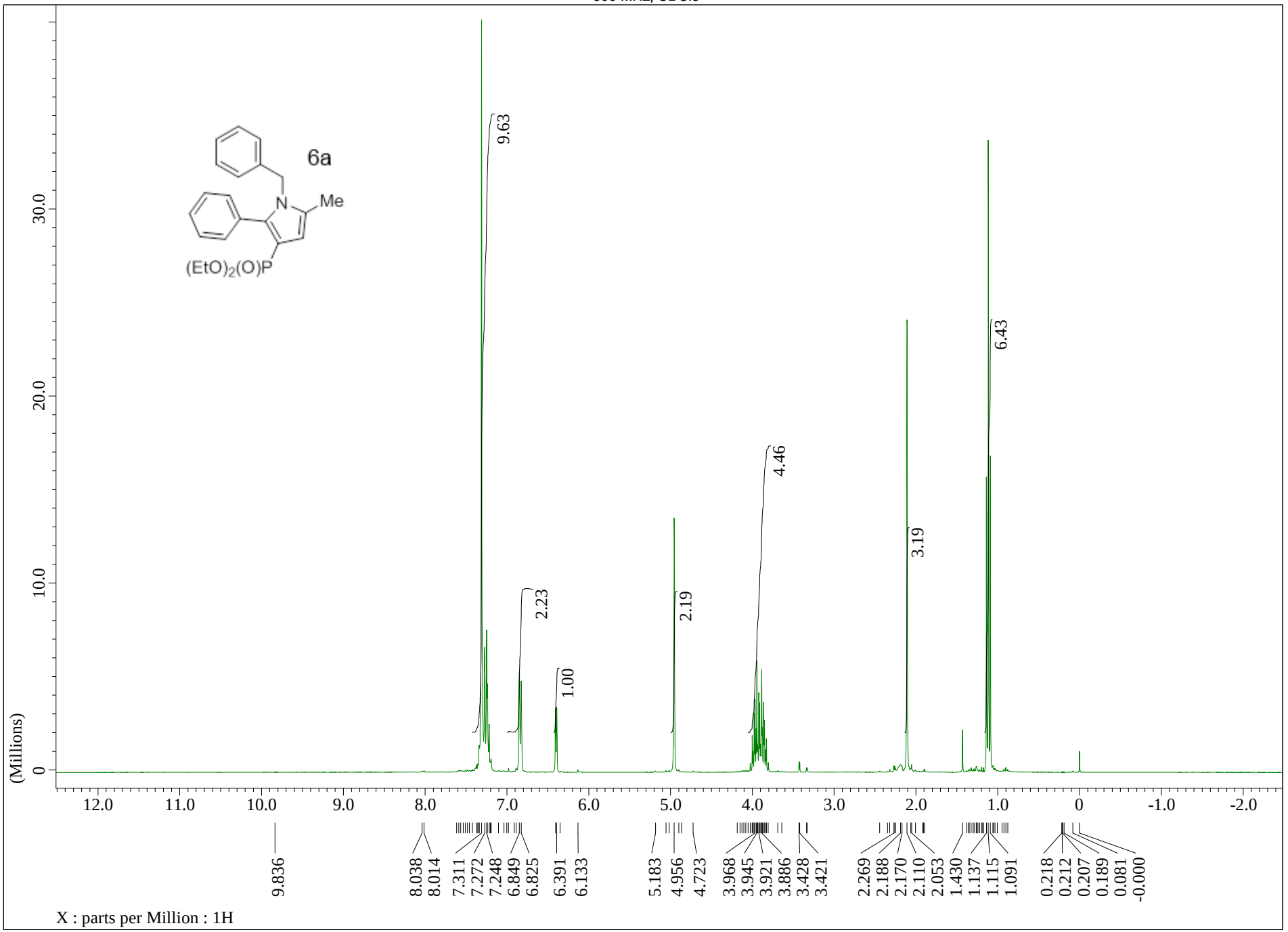
100 MHz, CDCl3



Current Chromatogram(s)

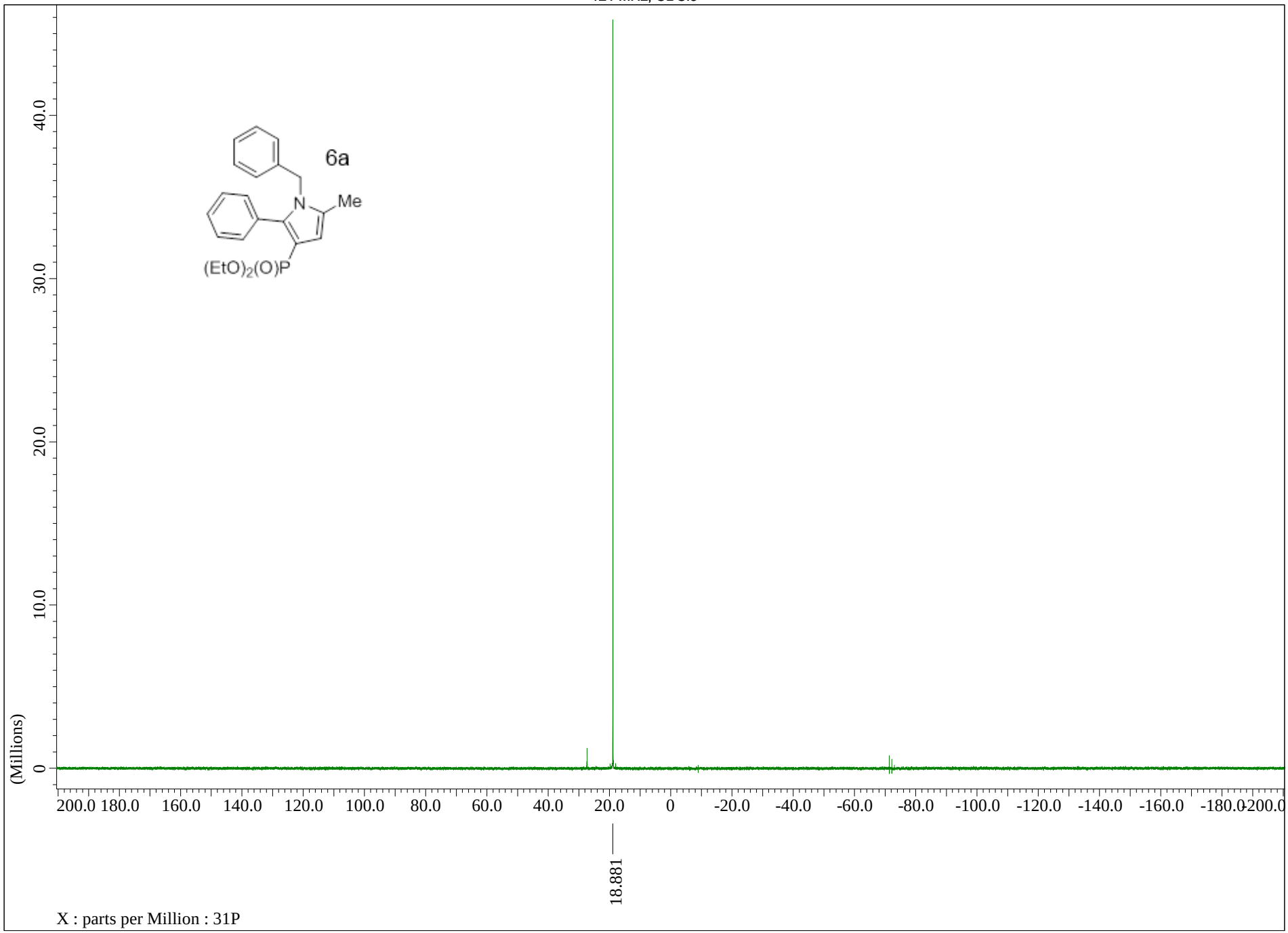


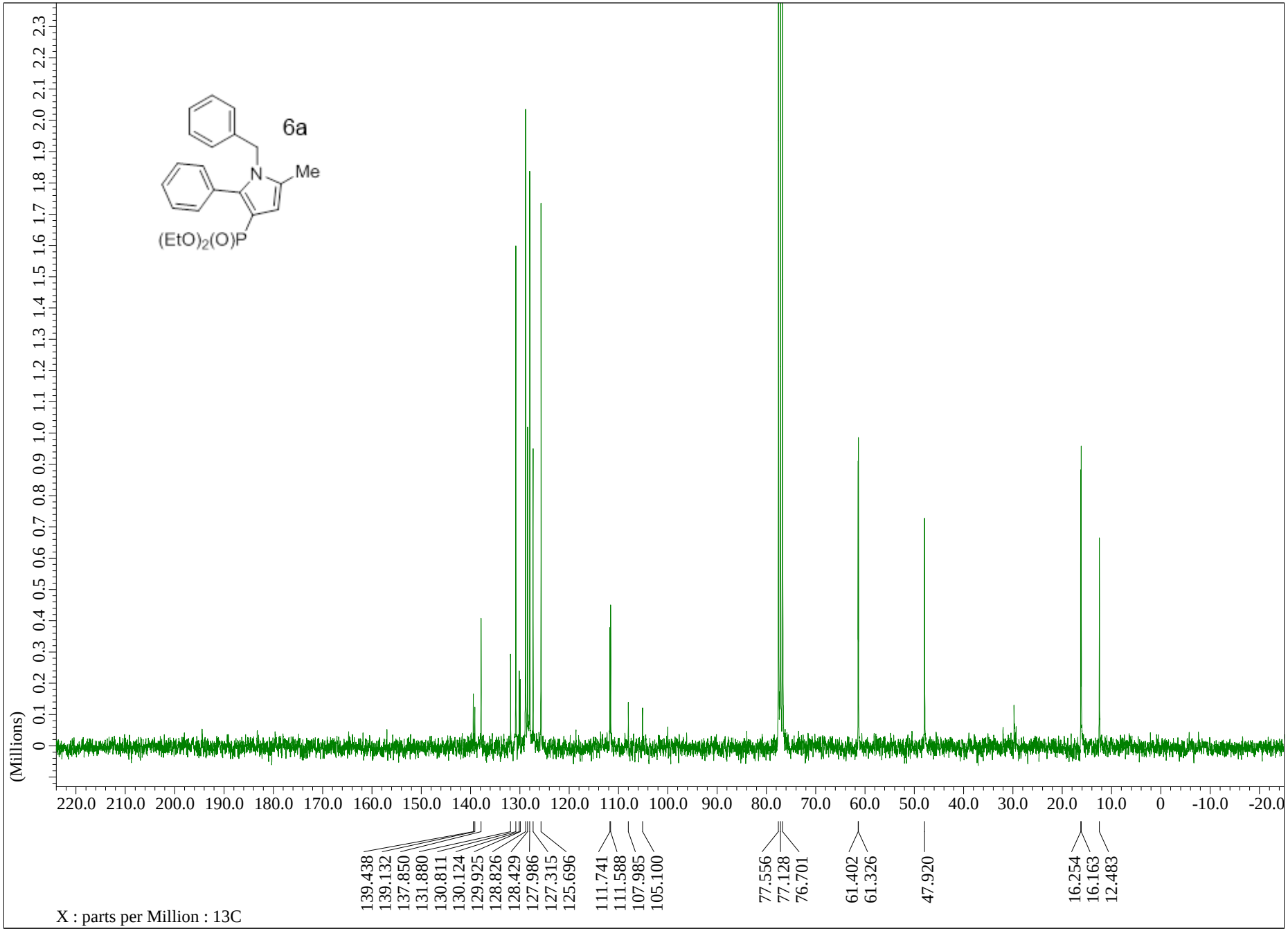
300 MHz, CDCl3



X : parts per Million : 1H

121 MHz, CDCl3

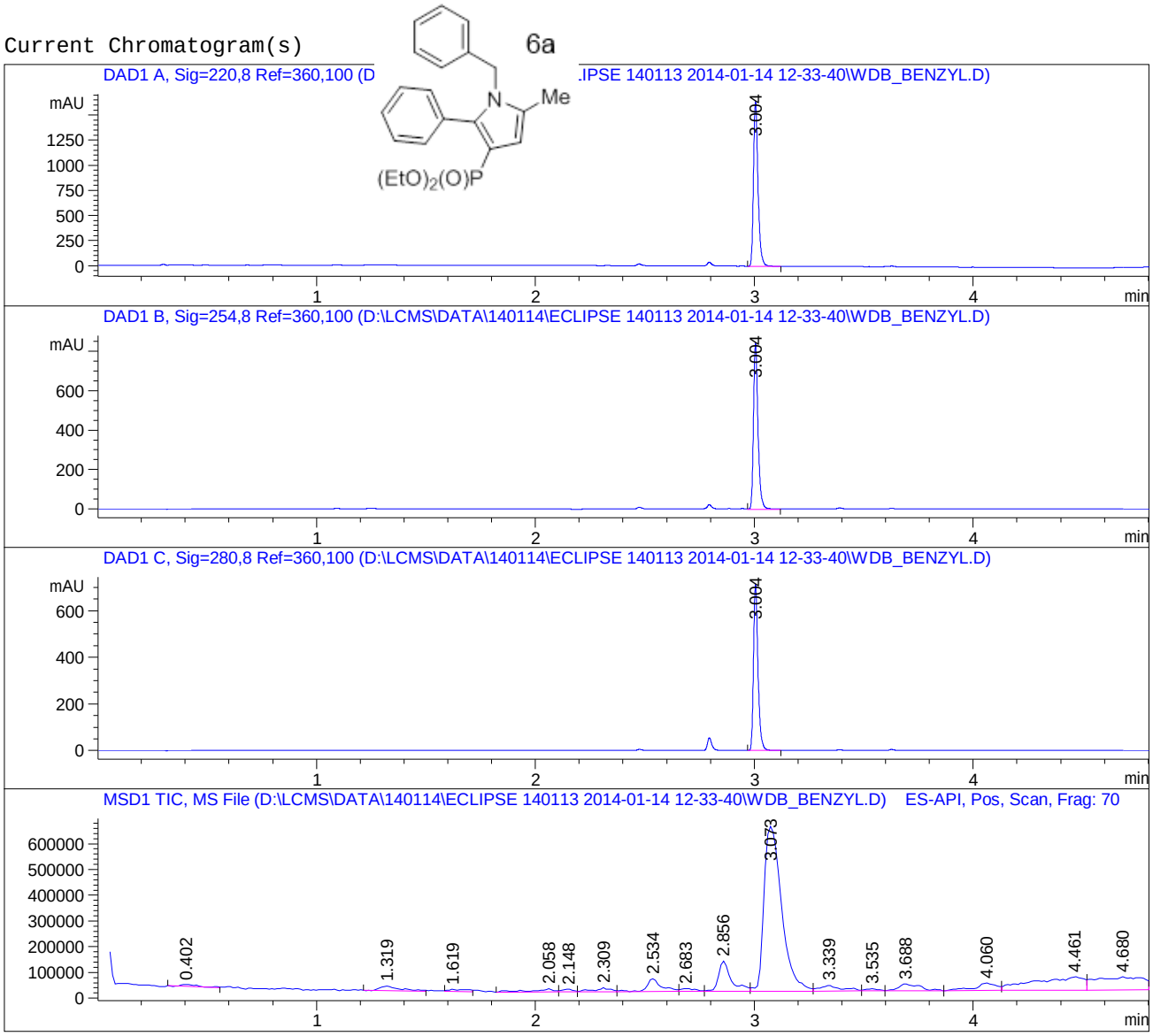


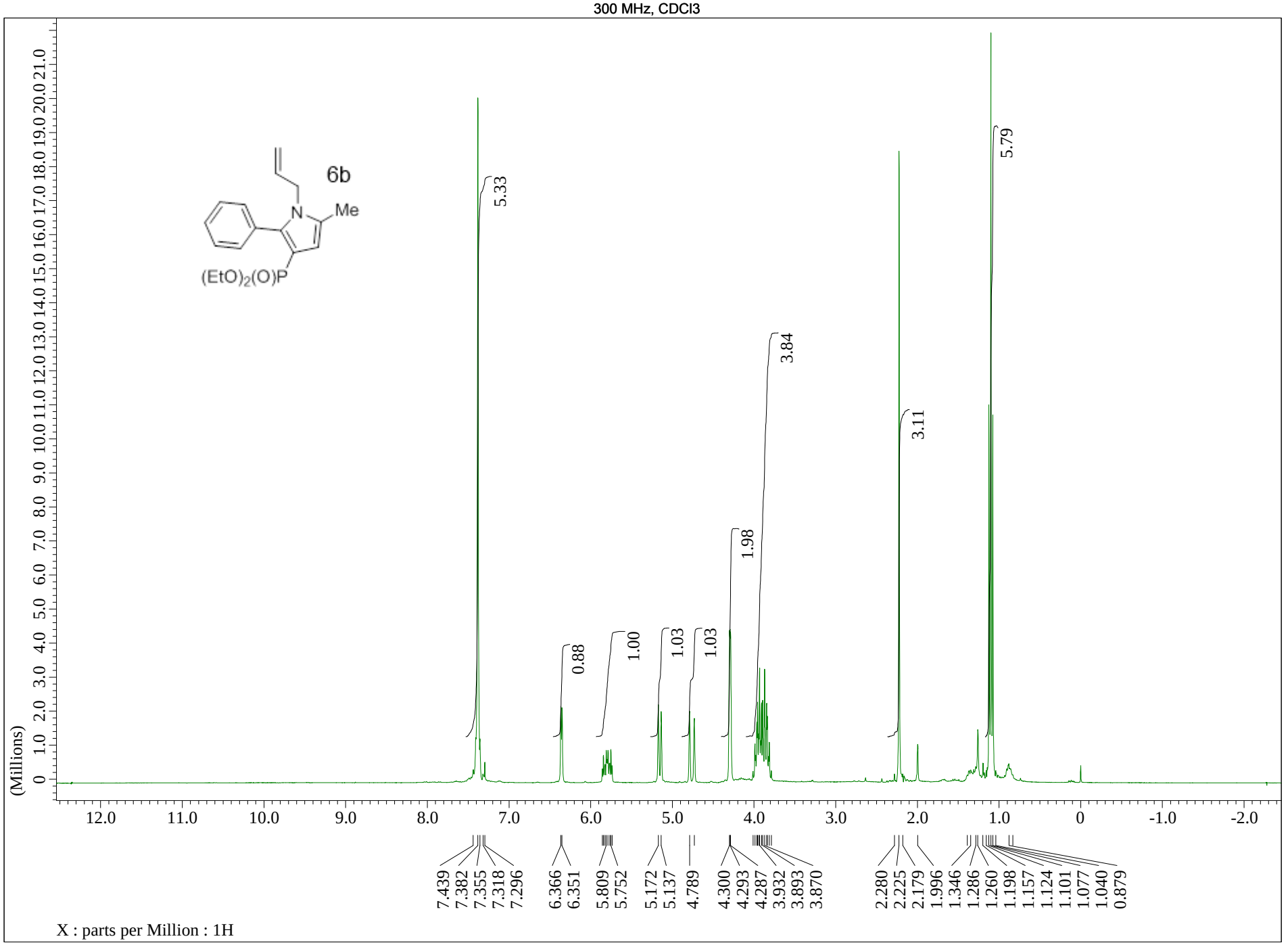

6a
N
Me
(EtO)2(O)P
(Millions)
0 0.1 0.2 0.3 0.4 0.5 0.6 0.7 0.8 0.9 1.0 1.1 1.2 1.3 1.4 1.5 1.6 1.7 1.8 1.9 2.0 2.1 2.2 2.3
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0 -20.0
139.438
139.132
137.850
131.880
130.811
130.124
129.925
128.826
128.429
127.986
127.315
125.696
111.741
111.588
107.985
105.100
77.556
77.128
76.701
61.402
61.326
47.920
16.254
16.163
12.483
X : parts per Million : 13C

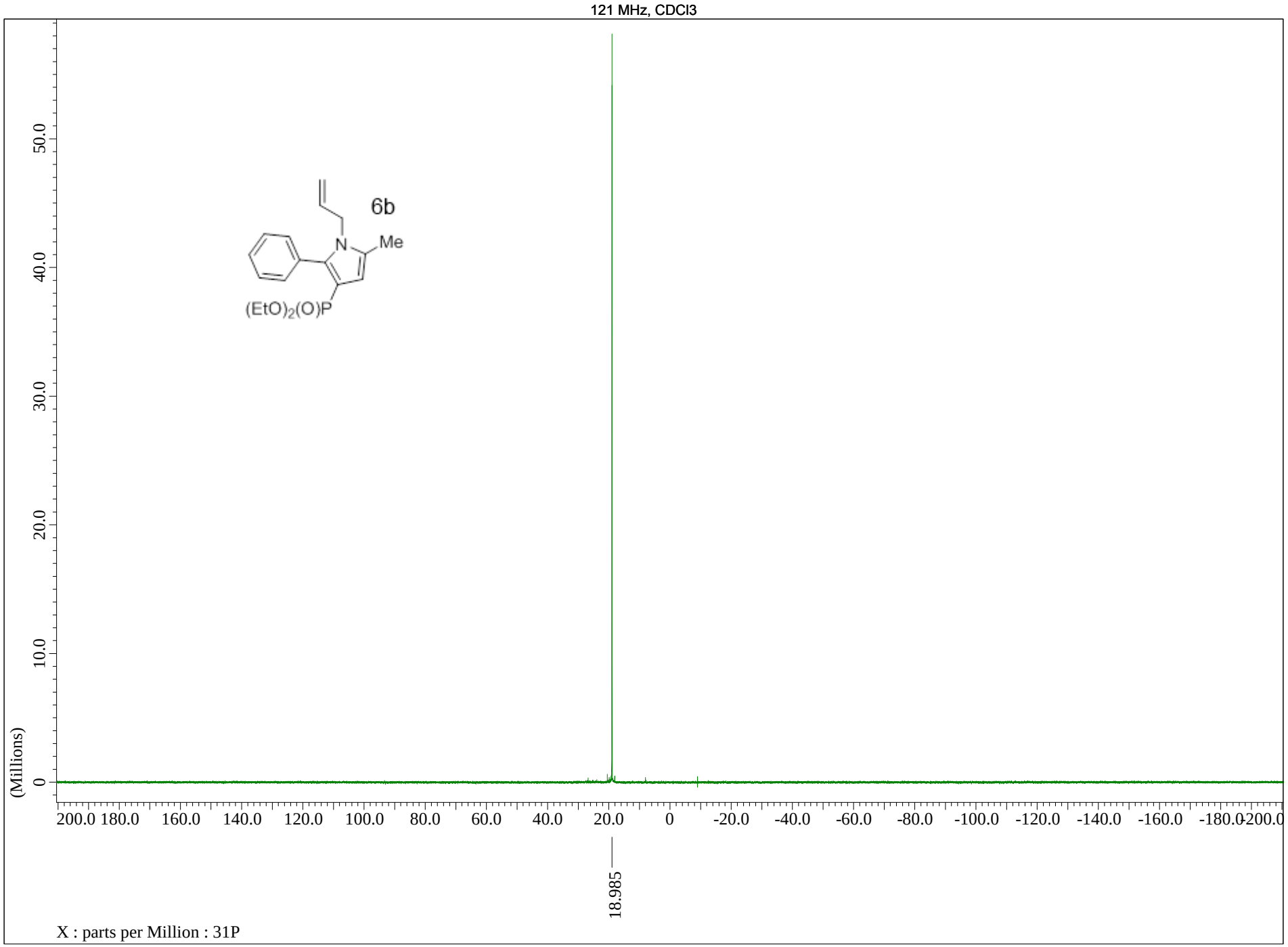
Print of window 38: Current Chromatogram(s)
Data File : D:\LCMS\DATA\140114\ECLIPSE 140113 2014-01-14 12-33-40\WDB_BENZYL.D
Sample Name : WDB_benzyl

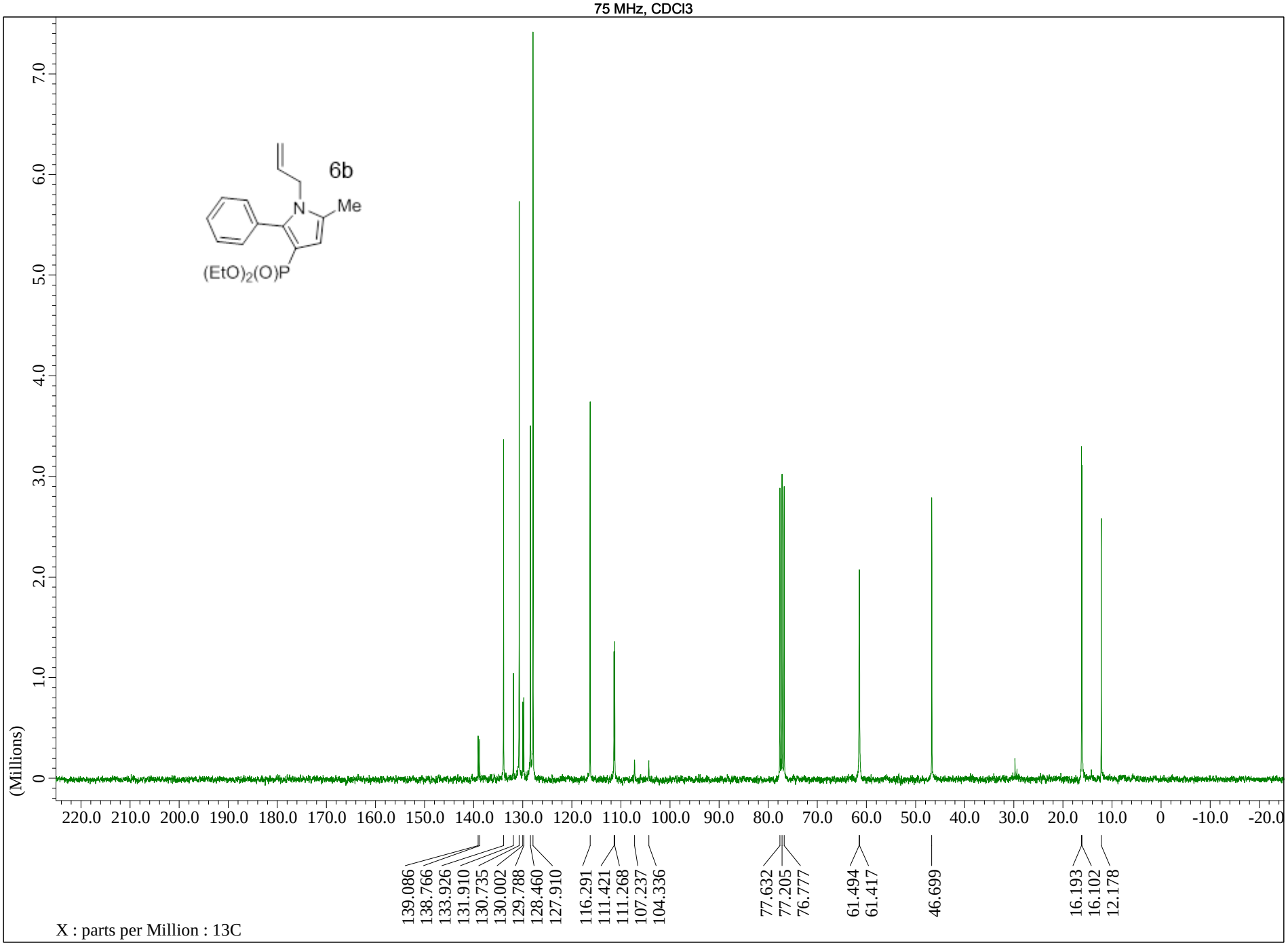
Acq. Operator : PC Seq. Line : 27
Acq. Instrument : Instrument 1 Location : Vial 64
Injection Date : 1/14/2014 3:20:49 PM Inj : 1
Inj Volume : 2.0 µl
Acq. Method : D:\LCMS\DATA\140114\ECLIPSE 140113 2014-01-14 12-33-40\ASCENTIS CH3CN LCMS
Last changed : 1/13/2014 4:31:43 PM by PC
Analysis Method : D:\LCMS\METHODS\NEG ASCENTIS CH3CN LCMS11L.M
Last changed : 1/14/2014 1:45:24 PM by PC
(modified after loading)
Method Info : Ascentis column method for Synthesis samples

Current Chromatogram(s)




300 MHz, CDCl3
6b
N
Me
(EtO)2(O)P
(Millions)
0 1.0 2.0 3.0 4.0 5.0 6.0 7.0 8.0 9.0 10.0 11.0 12.0 13.0 14.0 15.0 16.0 17.0 18.0 19.0 20.0 21.0
5.33
0.88
1.00
1.03
1.03
1.98
3.84
3.11
5.79
12.0 11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0 -1.0 -2.0
7.439 7.382 7.355 7.318 7.296
6.366 6.351
5.809 5.752
5.172 5.137
4.789
4.300 4.293 4.287 3.932 3.893 3.870
2.280 2.225 2.179 1.996 1.346 1.286 1.260 1.198 1.157 1.124 1.101 1.077 1.040 0.879
X : parts per Million : 1H


121 MHz, CDCl3
6b
N
Me
(EtO)2(O)P
(Millions)
50.0
40.0
30.0
20.0
10.0
0
200.0 180.0 160.0 140.0 120.0 100.0 80.0 60.0 40.0 20.0 0 -20.0 -40.0 -60.0 -80.0 -100.0 -120.0 -140.0 -160.0 -180.0 -200.0
18.985
X : parts per Million : 31P

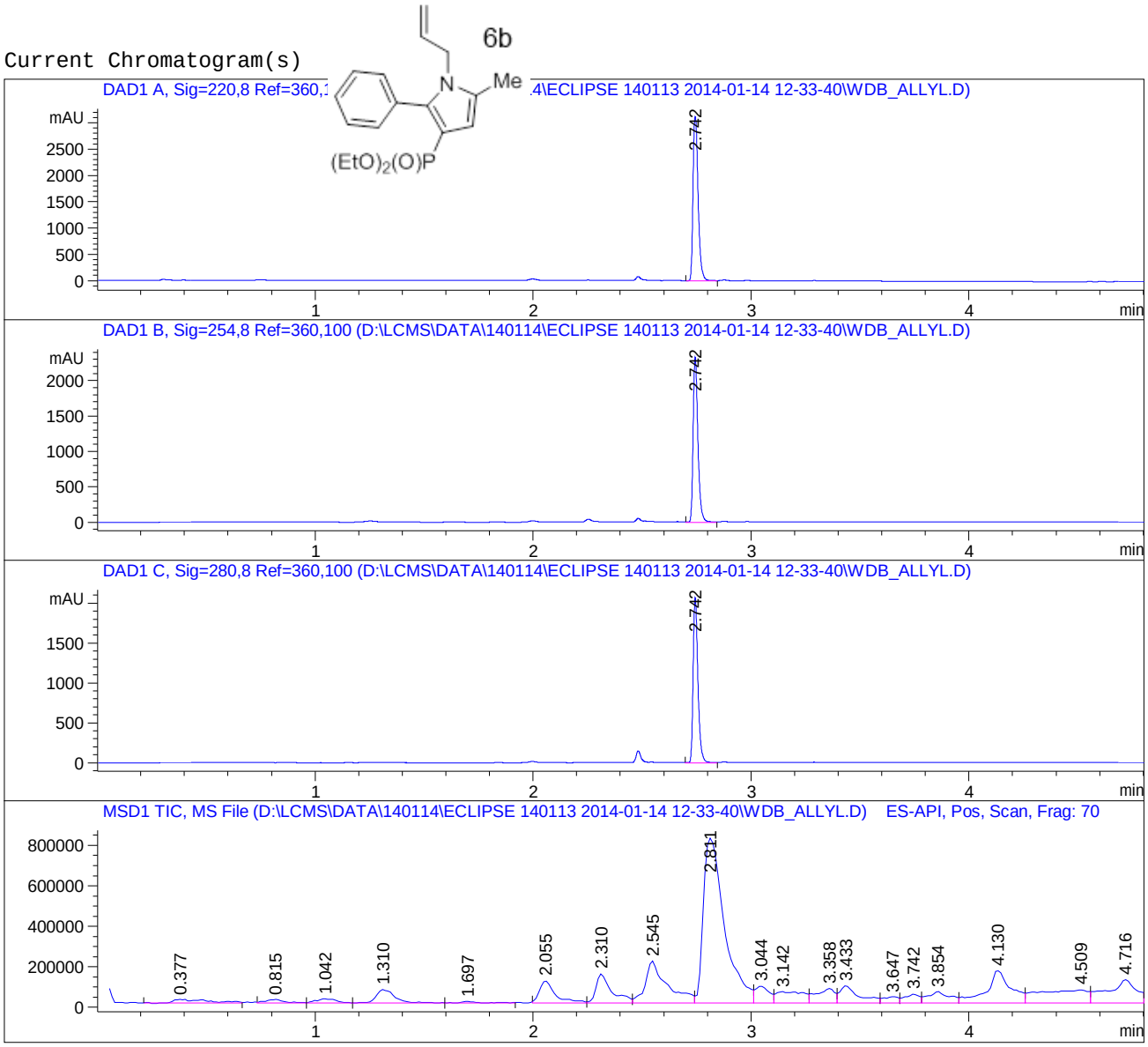

75 MHz, CDCl3
6b
N
Me
(EtO)2(O)P
(Millions)
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0 -20.0
139.086
138.766
133.926
131.910
130.735
130.002
129.788
128.460
127.910
116.291
111.421
111.268
107.237
104.336
77.632
77.205
76.777
61.494
61.417
46.699
16.193
16.102
12.178
X : parts per Million : 13C

Print of window 38: Current Chromatogram(s)
Data File : D:\LCMS\DATA\140114\ECLIPSE 140113 2014-01-14 12-33-40\WDB_ALLYL.D
Sample Name : WDB_allyl

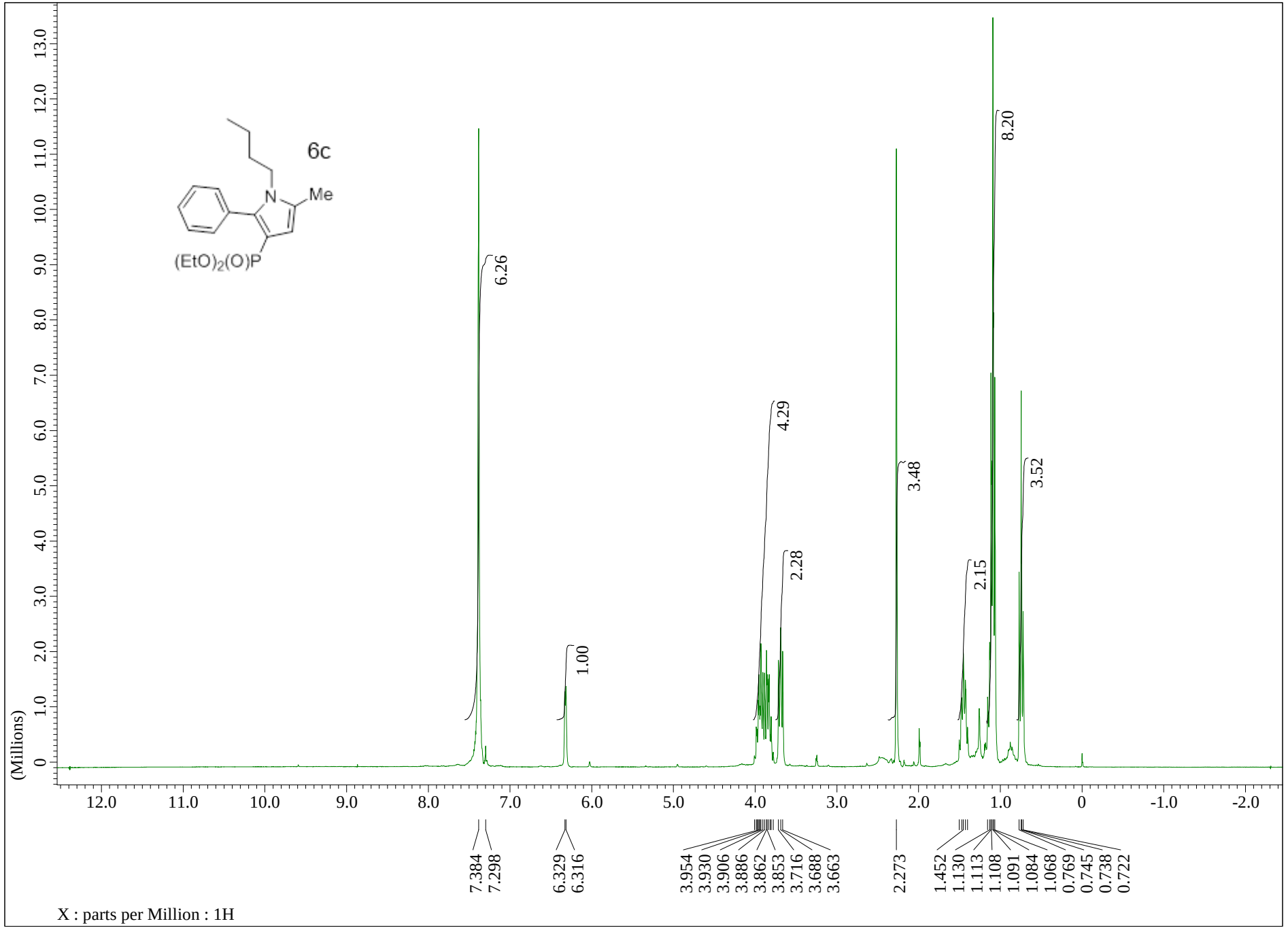
Acq. Operator	: PC	Seq. Line	: 29
Acq. Instrument	: Instrument 1	Location	: Vial 66
Injection Date	: 1/14/2014 3:32:46 PM	Inj	: 1
		Inj Volume	: 2.0 µl

Acq. Method : D:\LCMS\DATA\140114\ECLIPSE 140113 2014-01-14 12-33-40\ASCENTIS CH3CN LCMS
Last changed : 1/13/2014 4:31:43 PM by PC
Analysis Method : D:\LCMS\METHODS\NEG ASCENTIS CH3CN LCMS11L.M
Last changed : 1/14/2014 1:45:24 PM by PC
(modified after loading)
Method Info : Ascentis column method for Synthesis samples

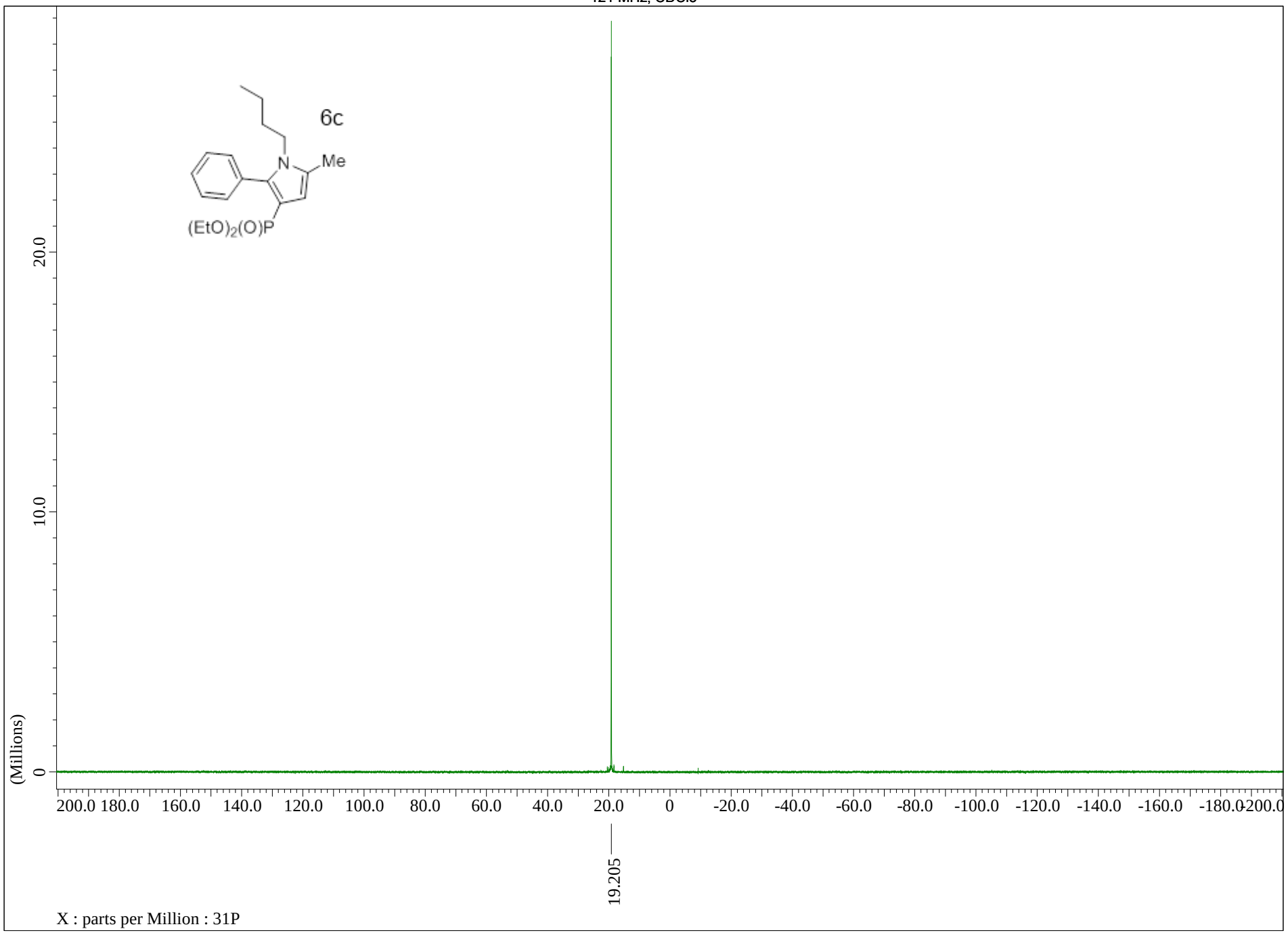
Current Chromatogram(s)

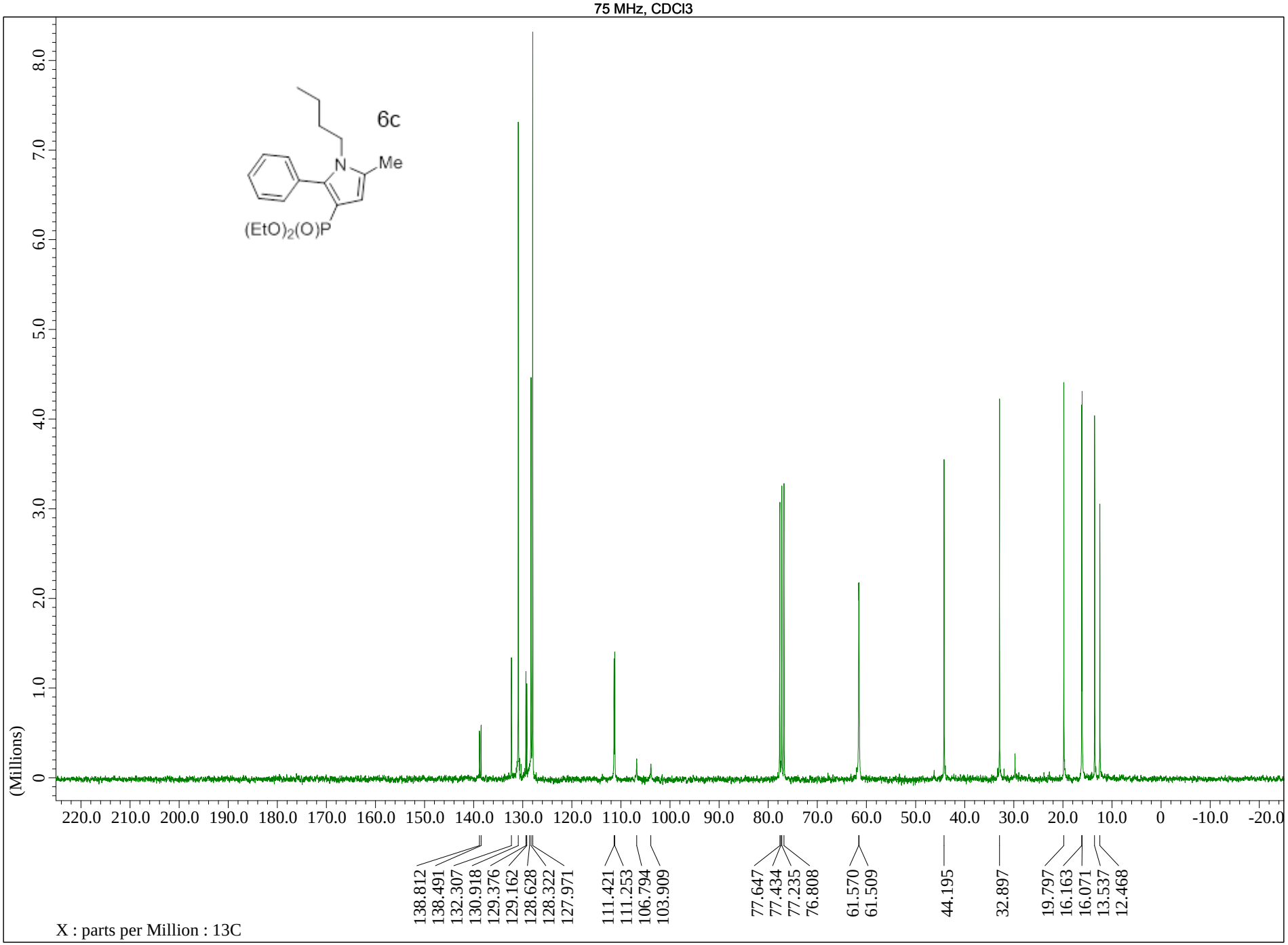


300 MHz, CDCl3



121 MHz, CDCl3



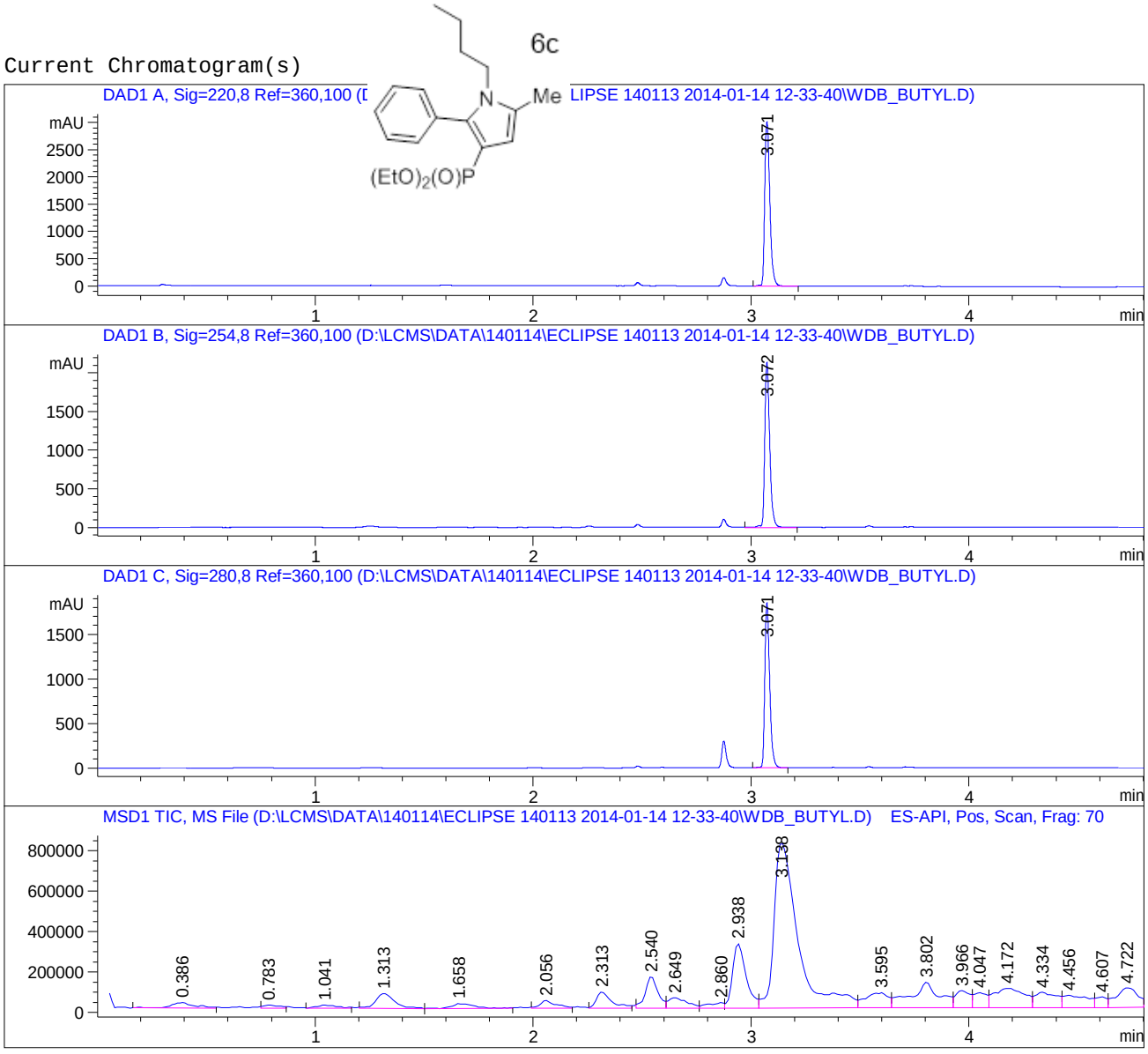

75 MHz, CDCl3
6c
N
Me
(EtO)2(O)P
(Millions)
0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0 -20.0
138.812
138.491
132.307
130.918
129.376
129.162
128.628
128.322
127.971
111.421
111.253
106.794
103.909
77.647
77.434
77.235
76.808
61.570
61.509
44.195
32.897
19.797
16.163
16.071
13.537
12.468
X : parts per Million : 13C

Print of window 38: Current Chromatogram(s)
Data File : D:\LCMS\DATA\140114\ECLIPSE 140113 2014-01-14 12-33-40\WDB_BUTYL.D
Sample Name : WDB_butyl

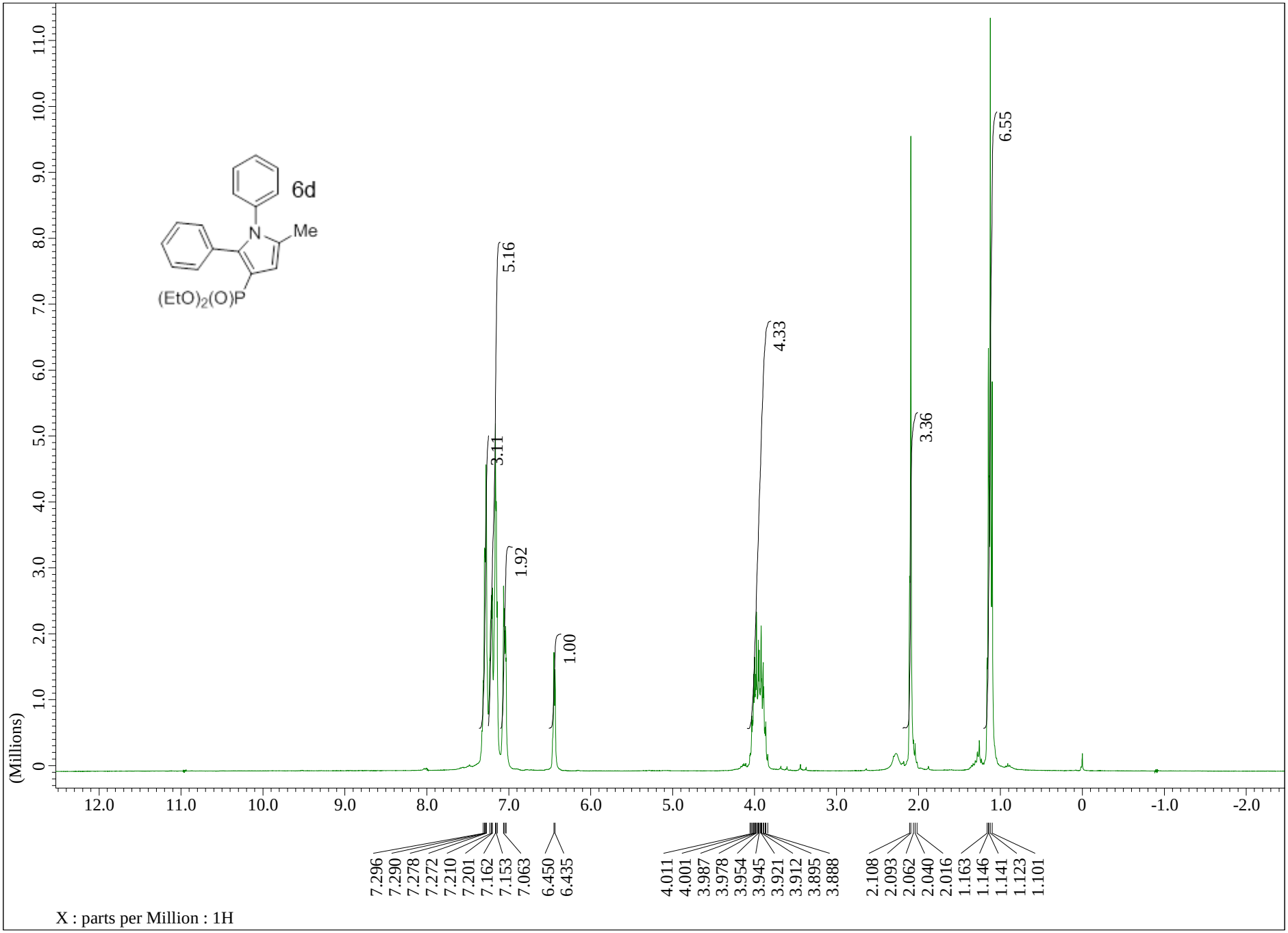
Acq. Operator	: PC	Seq. Line	: 28
Acq. Instrument	: Instrument 1	Location	: Vial 65
Injection Date	: 1/14/2014 3:26:48 PM	Inj	: 1
		Inj Volume	: 2.0 µl

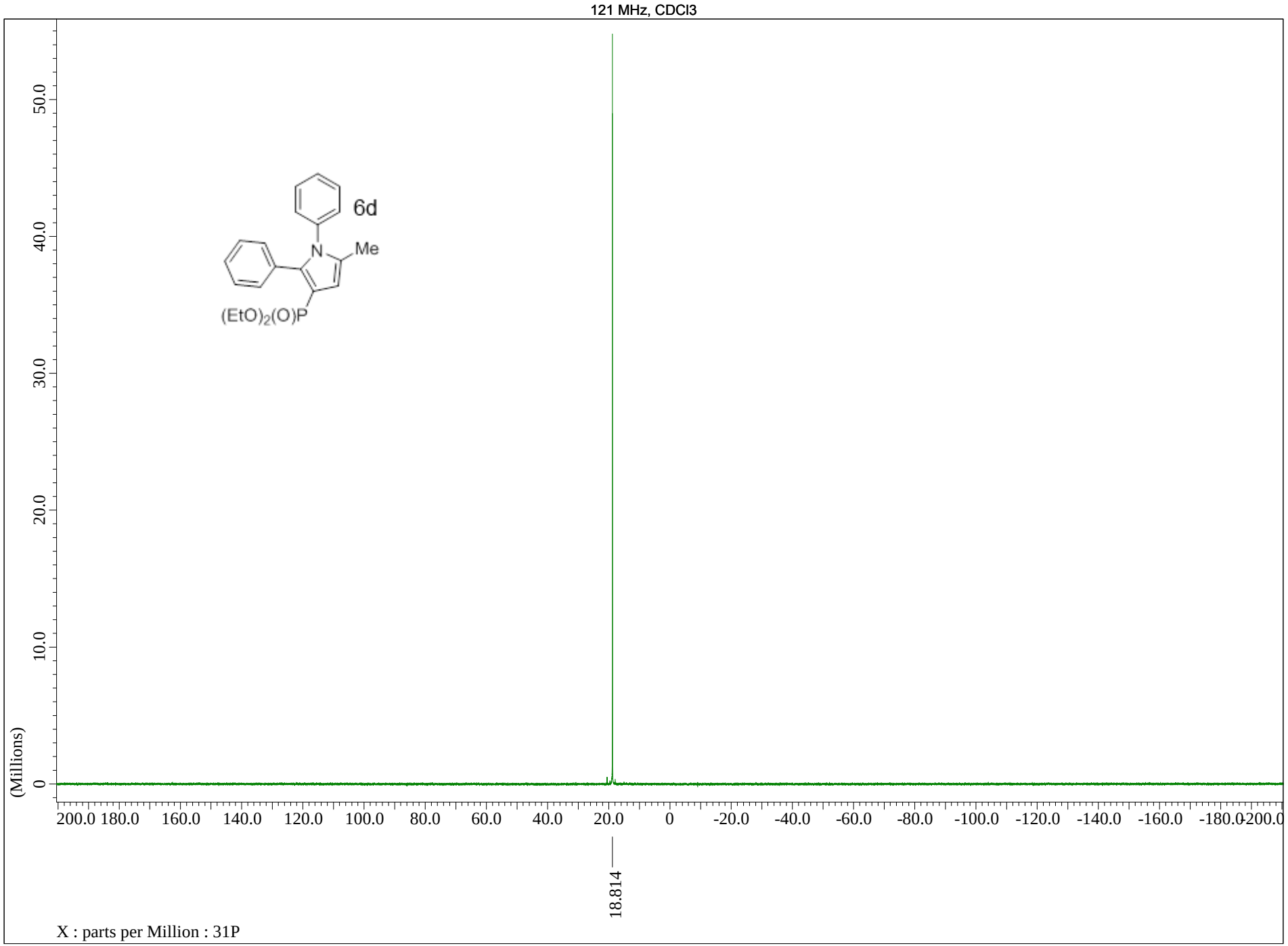
Acq. Method : D:\LCMS\DATA\140114\ECLIPSE 140113 2014-01-14 12-33-40\ASCENTIS CH3CN LCMS
Last changed : 1/13/2014 4:31:43 PM by PC
Analysis Method : D:\LCMS\METHODS\NEG ASCENTIS CH3CN LCMS11L.M
Last changed : 1/14/2014 1:45:24 PM by PC
(modified after loading)
Method Info : Ascentis column method for Synthesis samples

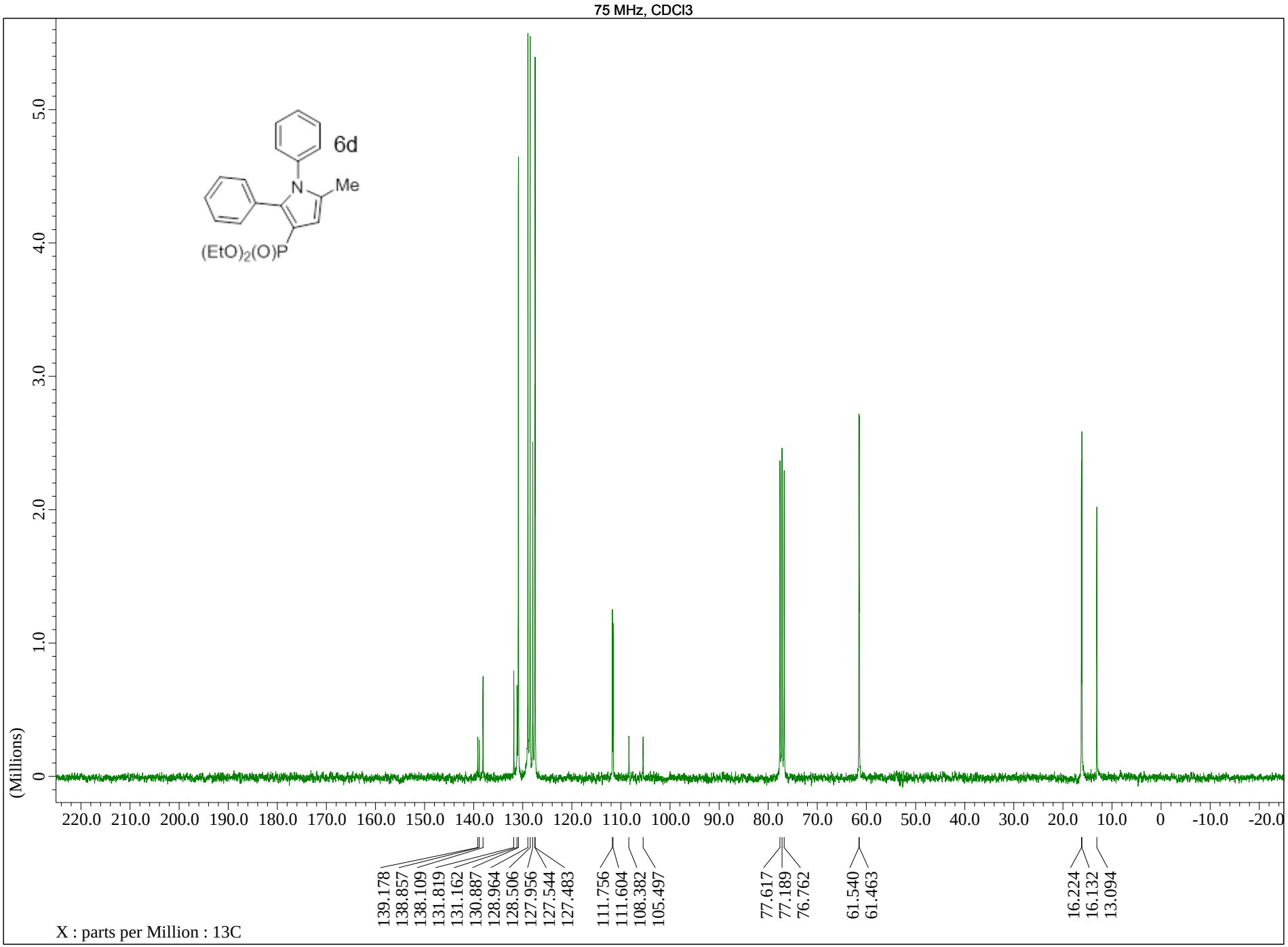
Current Chromatogram(s)



300 MHz, CDCl3



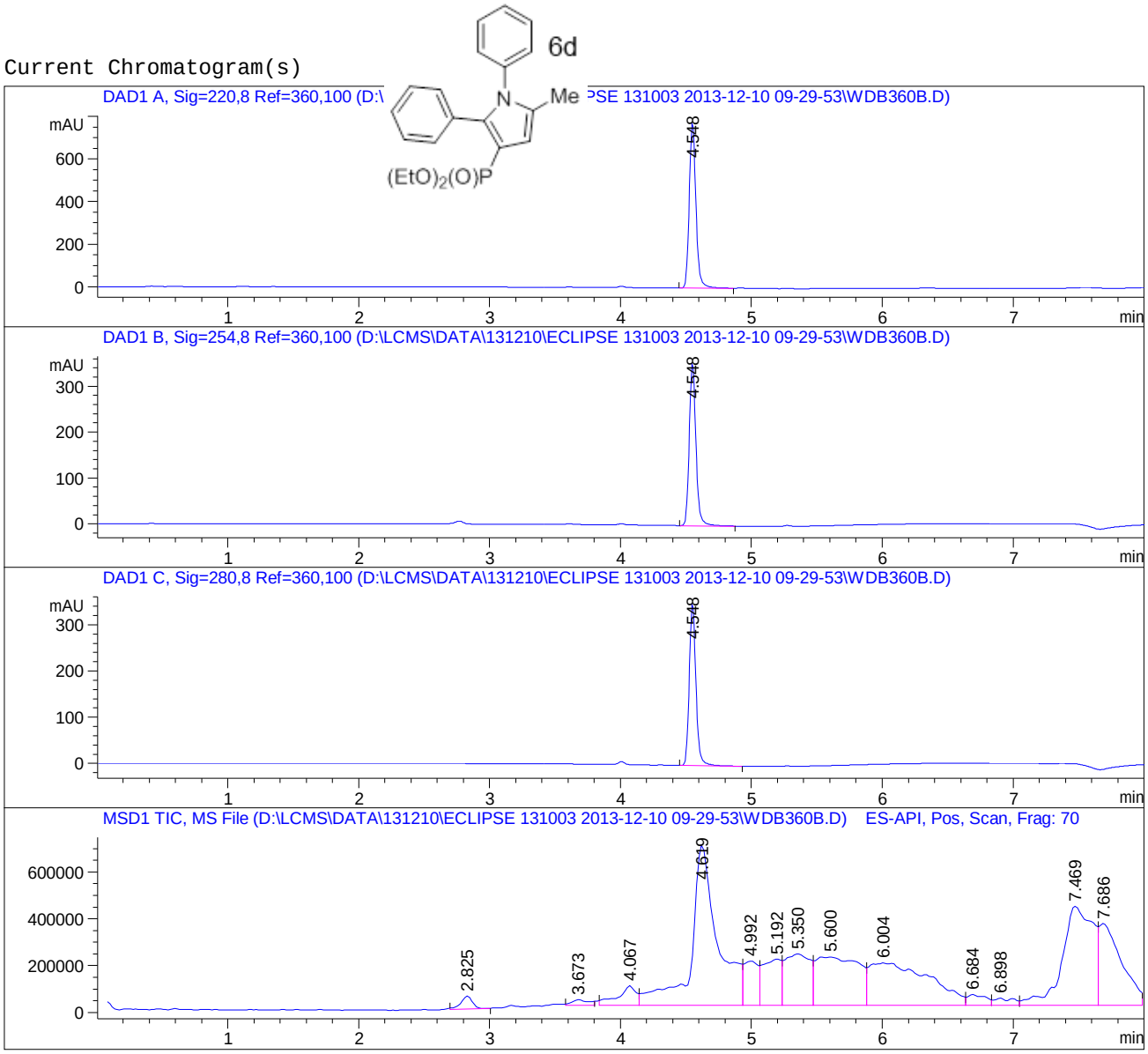

121 MHz, CDCl3
6d
N
Me
(EtO)2(O)P
(Millions)
50.0
40.0
30.0
20.0
10.0
0
200.0 180.0 160.0 140.0 120.0 100.0 80.0 60.0 40.0 20.0 0 -20.0 -40.0 -60.0 -80.0 -100.0 -120.0 -140.0 -160.0 -180.0 -200.0
18.814
X : parts per Million : 31P

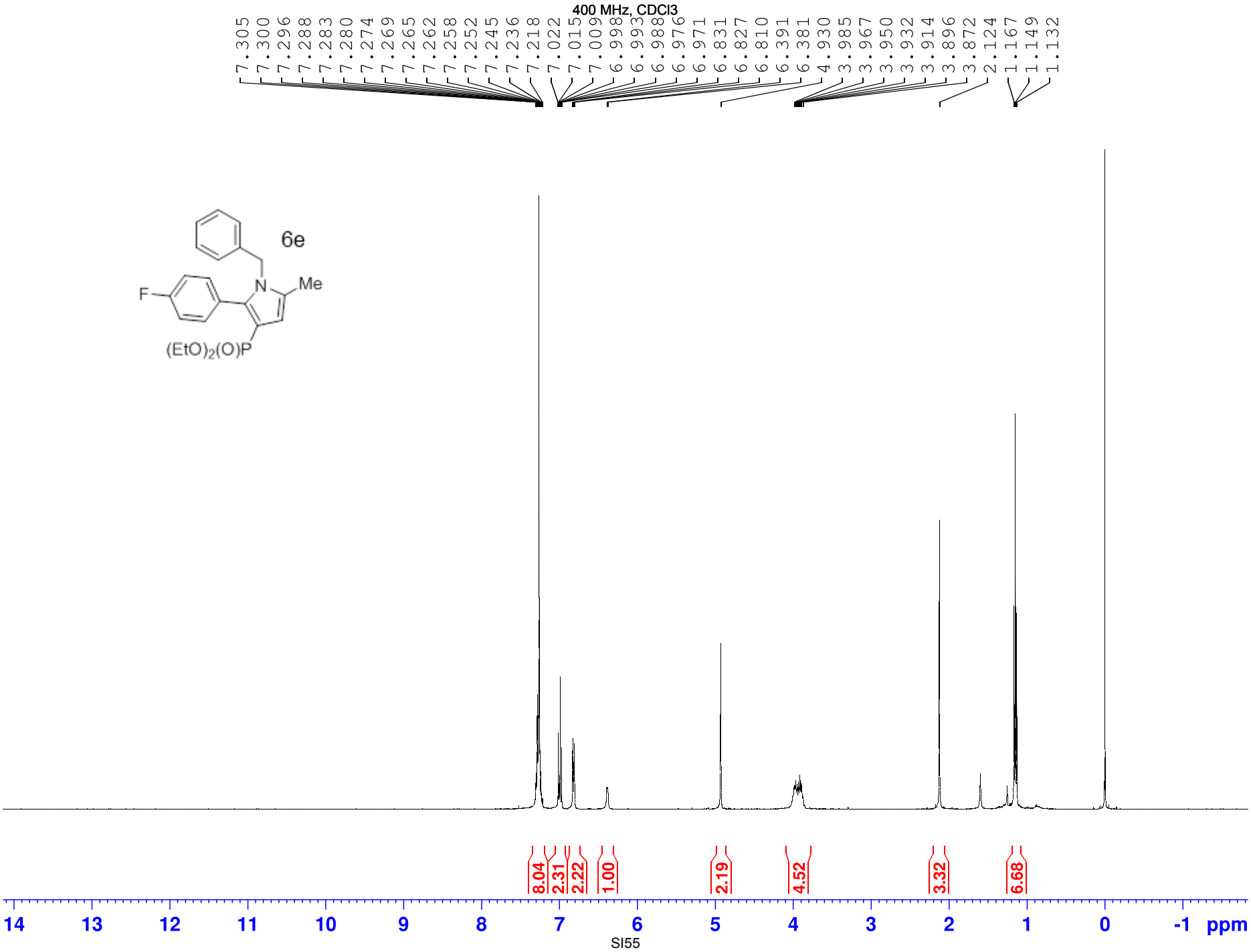

75 MHz, CDCl3
6d
N
Me
(EtO)2(O)P
(Millions)
5.0
4.0
3.0
2.0
1.0
0
220.0 210.0 200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 -10.0 -20.0
139.178
138.857
138.109
131.819
131.162
130.887
128.964
128.506
127.956
127.544
127.483
111.756
111.604
108.382
105.497
77.617
77.189
76.762
61.540
61.463
16.224
16.132
13.094
X : parts per Million : 13C

Print of window 38: Current Chromatogram(s)
Data File : D:\LCMS\DATA\131210\ECLIPSE 131003 2013-12-10 09-29-53\WDB360B.D
Sample Name : WDB360b

Acq. Operator : PC Seq. Line : 5
Acq. Instrument : Instrument 1 Location : Vial 4
Injection Date : 12/10/2013 10:11:35 AM Inj : 1
Inj Volume : 2.0 µl
Acq. Method : D:\LCMS\DATA\131210\ECLIPSE 131003 2013-12-10 09-29-53\ECLIPSE CH3CN LCMS1
Last changed : 9/11/2012 4:01:11 PM by PC
Analysis Method : D:\LCMS\METHODS\NEG ASCENTIS CH3CN LCMS11L.M
Last changed : 1/14/2014 1:45:24 PM by PC
(modified after loading)
Method Info : Ascentis column method for Synthesis samples

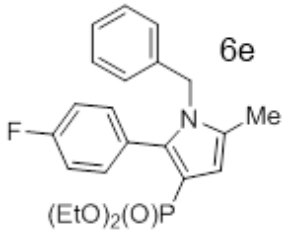
Current Chromatogram(s)

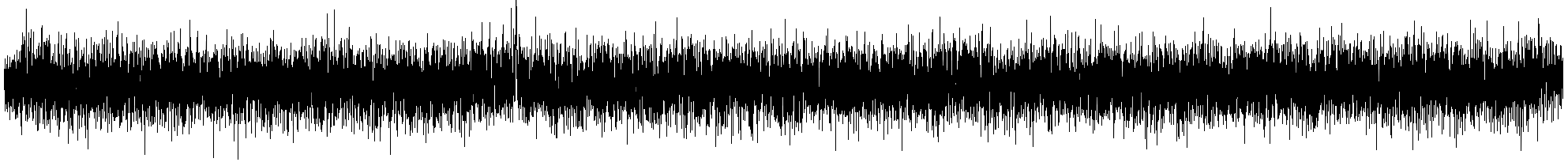



400 MHz, CDCl3
7.305
7.300
7.296
7.288
7.283
7.280
7.274
7.269
7.265
7.262
7.258
7.252
7.245
7.236
7.218
7.022
7.015
7.009
6.998
6.993
6.988
6.976
6.971
6.831
6.827
6.810
6.391
6.381
4.930
3.985
3.967
3.950
3.932
3.914
3.896
3.872
2.124
1.167
1.149
1.132
6e
F
N
Me
(EtO)2(O)P
8.04
2.31
2.22
1.00
2.19
4.52
3.32
6.68
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm

162 MHz, CDCl3

17.92

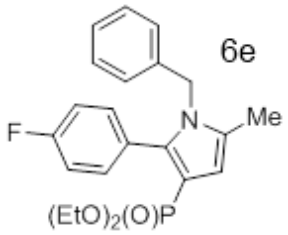




100 50 0 -50 -100 -150 -200 ppm

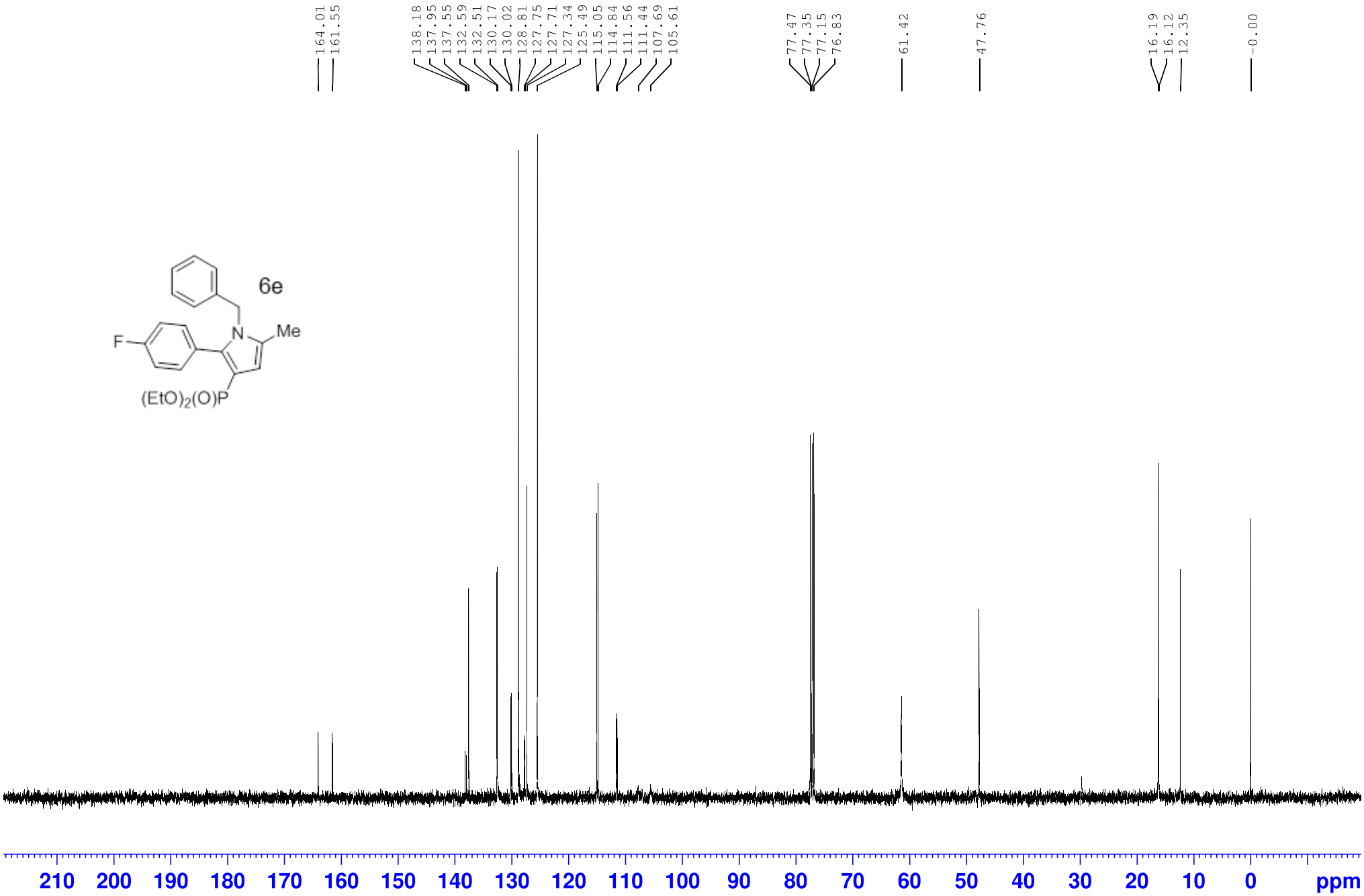
376 MHz, CDCl3

-113.01



0 -20 -40 -60 -80 -100 -120 -140 -160 -180 -200 ppm

100 MHz, CDCl3

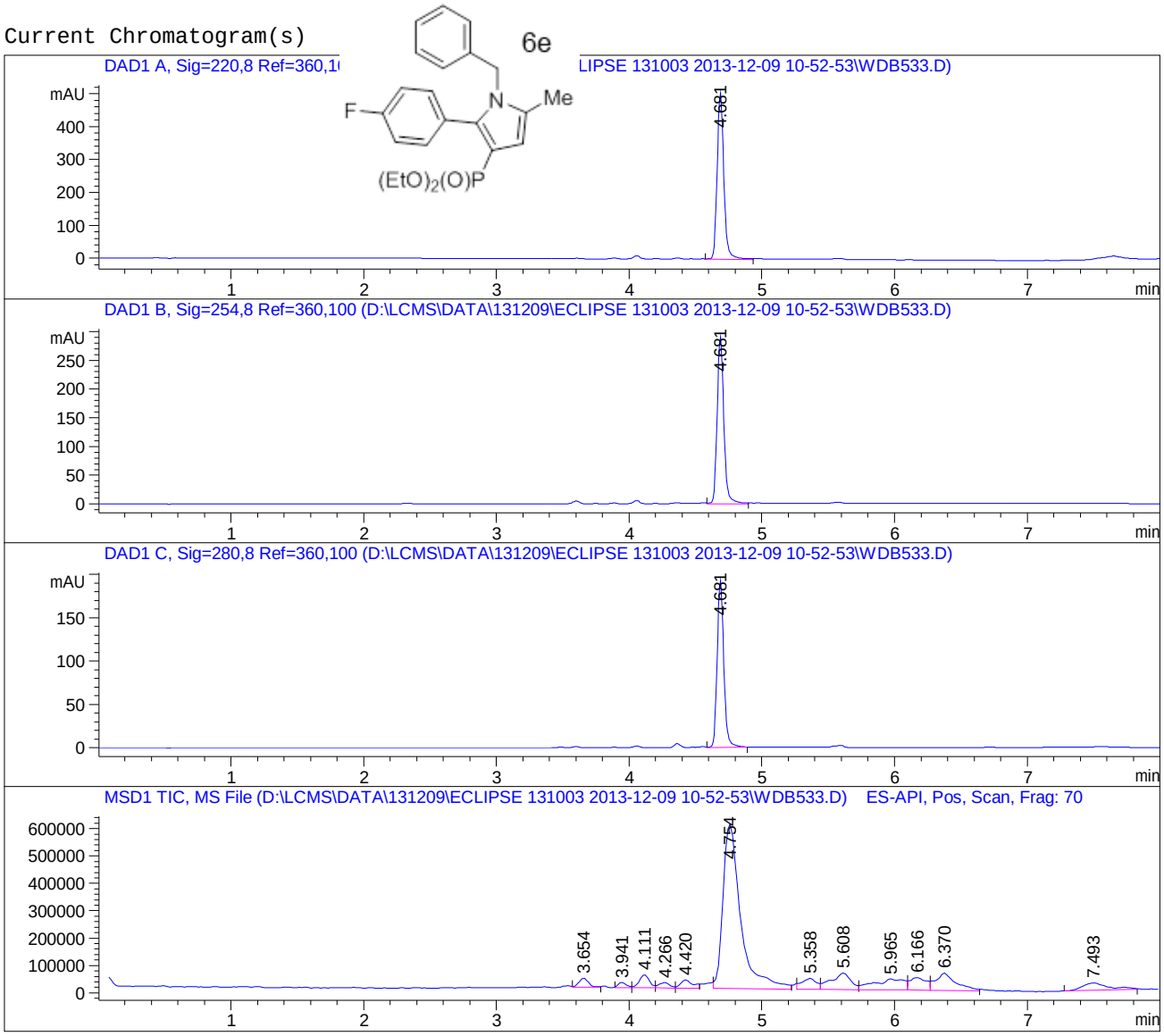


Print of window 38: Current Chromatogram(s)
Data File : D:\LCMS\DATA\131209\ECLIPSE 131003 2013-12-09 10-52-53\WDB533.D
Sample Name : WDB533

Acq. Operator	: PC	Seq. Line	: 21
Acq. Instrument	: Instrument 1	Location	: Vial 85
Injection Date	: 12/9/2013 2:16:30 PM	Inj	: 1
		Inj Volume	: 2.0 µl

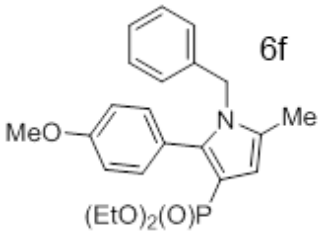
Acq. Method : D:\LCMS\DATA\131209\ECLIPSE 131003 2013-12-09 10-52-53\ECLIPSE CH3CN LCMS1
Last changed : 9/11/2012 4:01:11 PM by PC
Analysis Method : D:\LCMS\METHODS\NEG ASCENTIS CH3CN LCMS11L.M
Last changed : 1/14/2014 1:45:24 PM by PC
(modified after loading)
Method Info : Ascentis column method for Synthesis samples

Current Chromatogram(s)



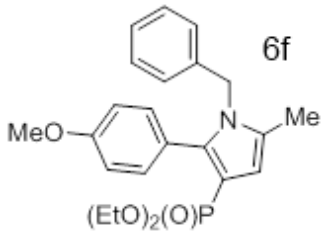
400 MHz, CDCl3

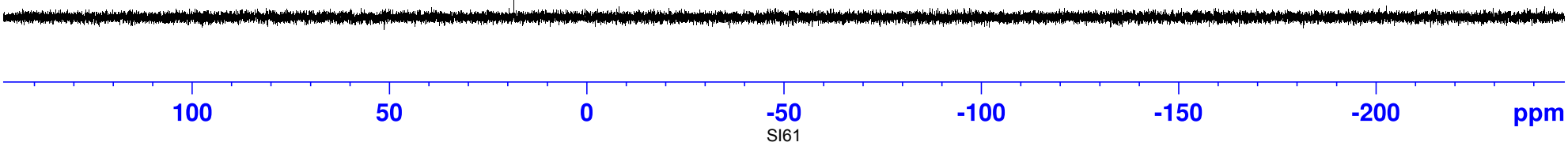
7.280
7.276
7.262
7.246
7.239
7.234
7.228
7.222
7.217
6.853
6.849
6.844
6.838
6.832
6.827
6.821
6.376
6.374
6.365
6.363
4.943
3.982
3.975
3.957
3.940
3.918
3.901
3.899
3.881
3.790
2.103
1.601
1.168
1.167
1.150
1.149
1.132
1.131
-0.000



162 MHz, CDCl3

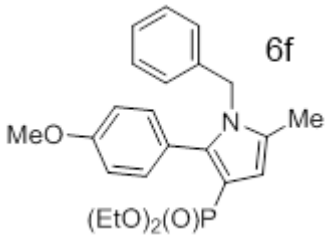
18.45





100 MHz, CDCl3

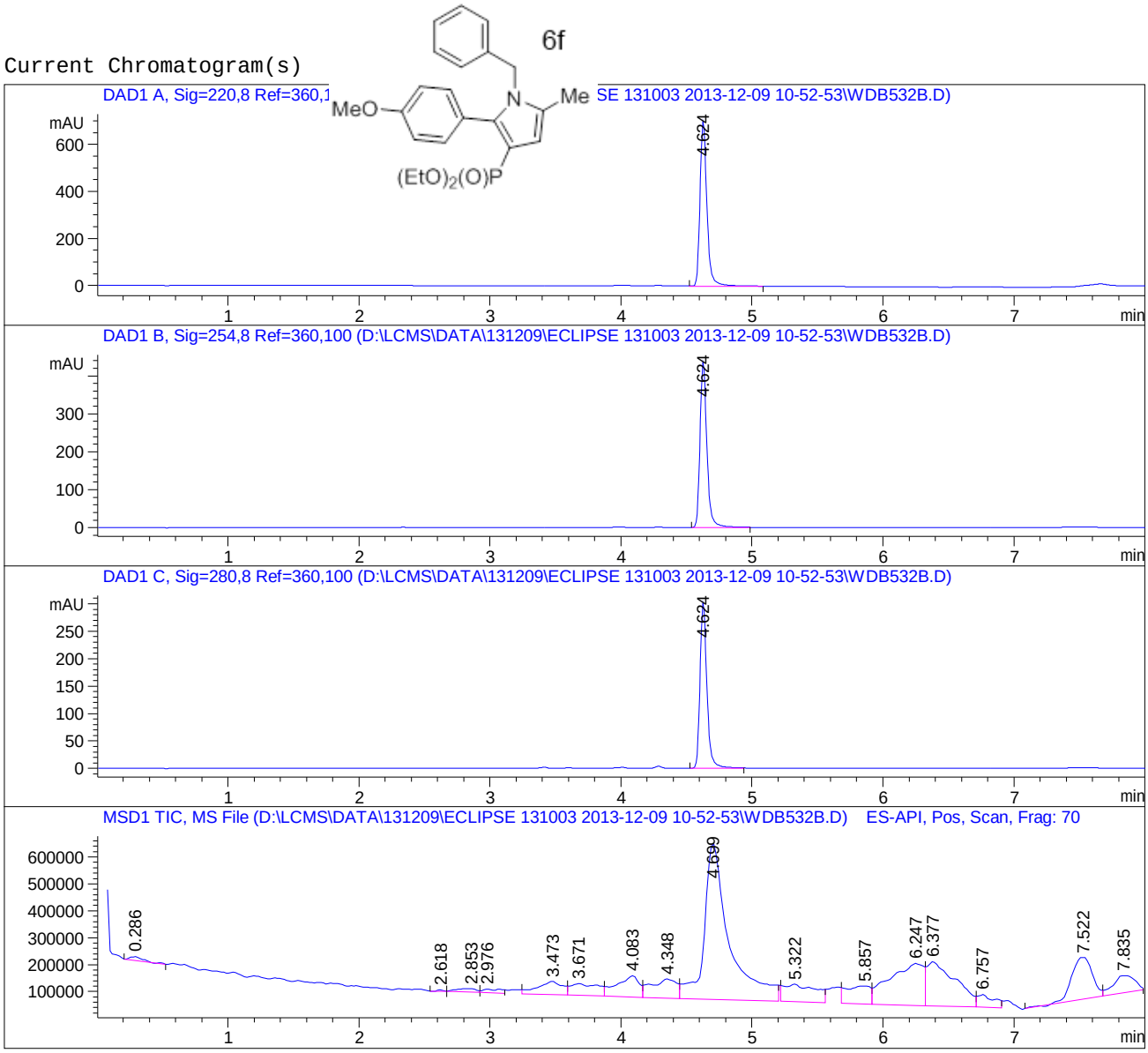
159.63
139.26
139.03
137.85
131.93
129.77
129.62
128.74
127.21
125.58
123.94
113.34
111.44
111.32
107.14
104.97
77.44
77.12
76.80
61.31
61.25
55.22
47.72
16.24
16.16
12.41



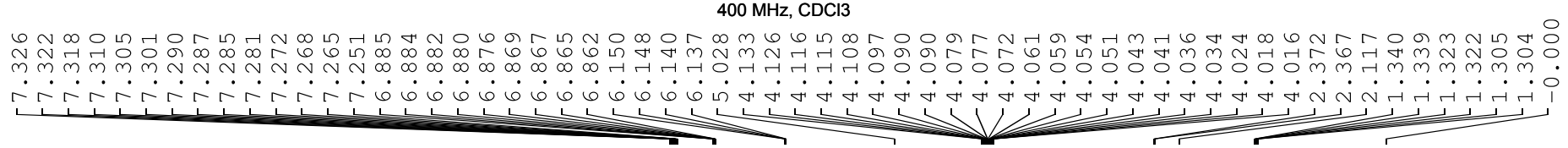
Print of window 38: Current Chromatogram(s)
Data File : D:\LCMS\DATA\131209\ECLIPSE 131003 2013-12-09 10-52-53\WDB532B.D
Sample Name : WDB532b

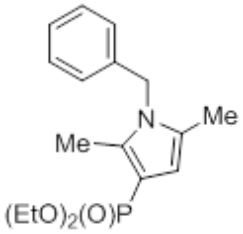
Acq. Operator : PC Seq. Line : 8
Acq. Instrument : Instrument 1 Location : Vial 81
Injection Date : 12/9/2013 12:12:59 PM Inj : 1
Inj Volume : 2.0 µl
Acq. Method : D:\LCMS\DATA\131209\ECLIPSE 131003 2013-12-09 10-52-53\ECLIPSE CH3CN LCMS1
Last changed : 9/11/2012 4:01:11 PM by PC
Analysis Method : D:\LCMS\METHODS\NEG ASCENTIS CH3CN LCMS11L.M
Last changed : 1/14/2014 1:45:24 PM by PC
(modified after loading)
Method Info : Ascentis column method for Synthesis samples

Current Chromatogram(s)



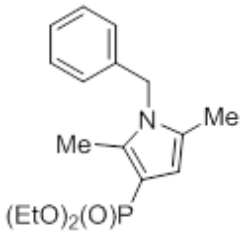
400 MHz, CDCl3



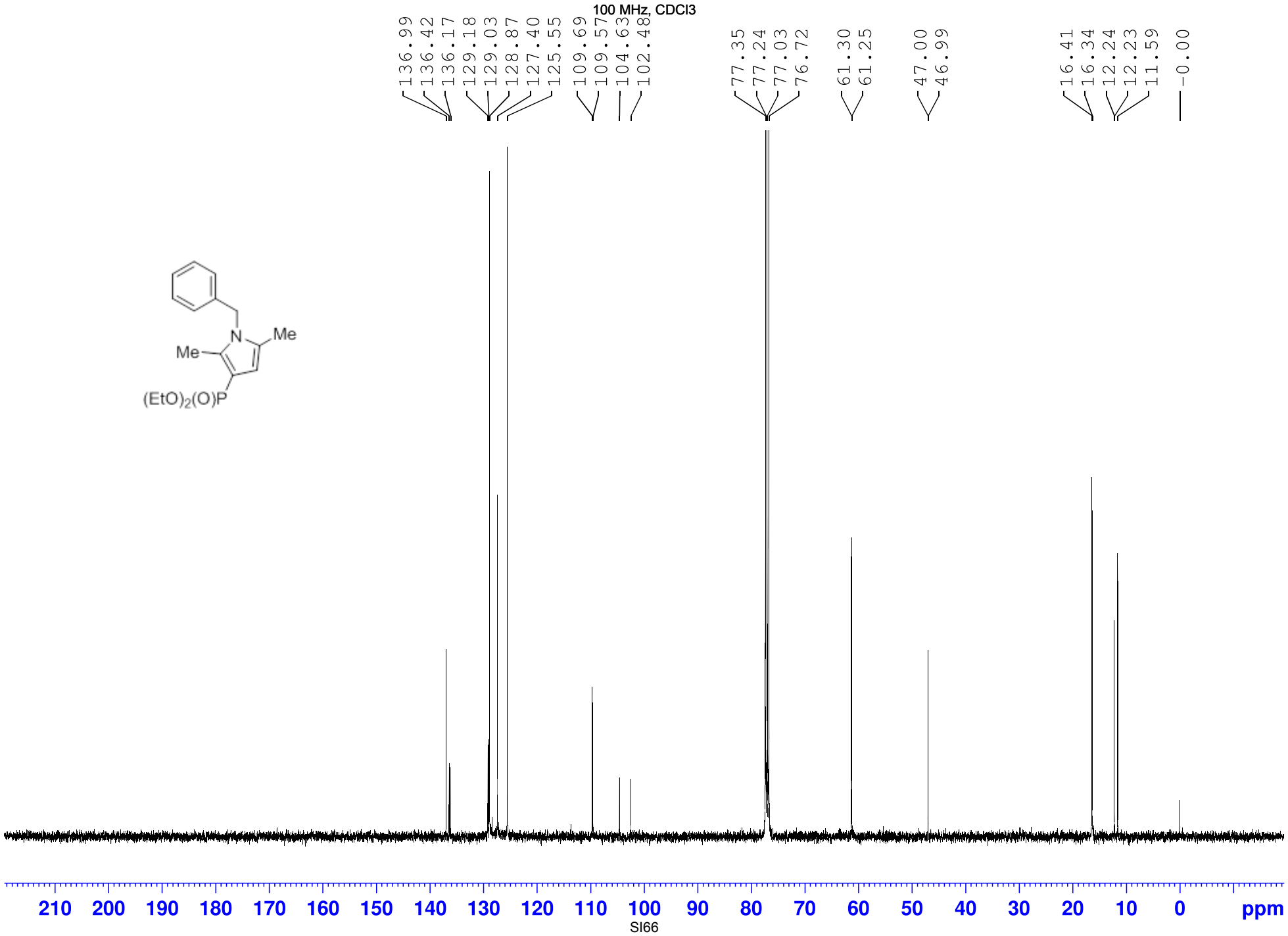


162 MHz, CDCl3

19.77



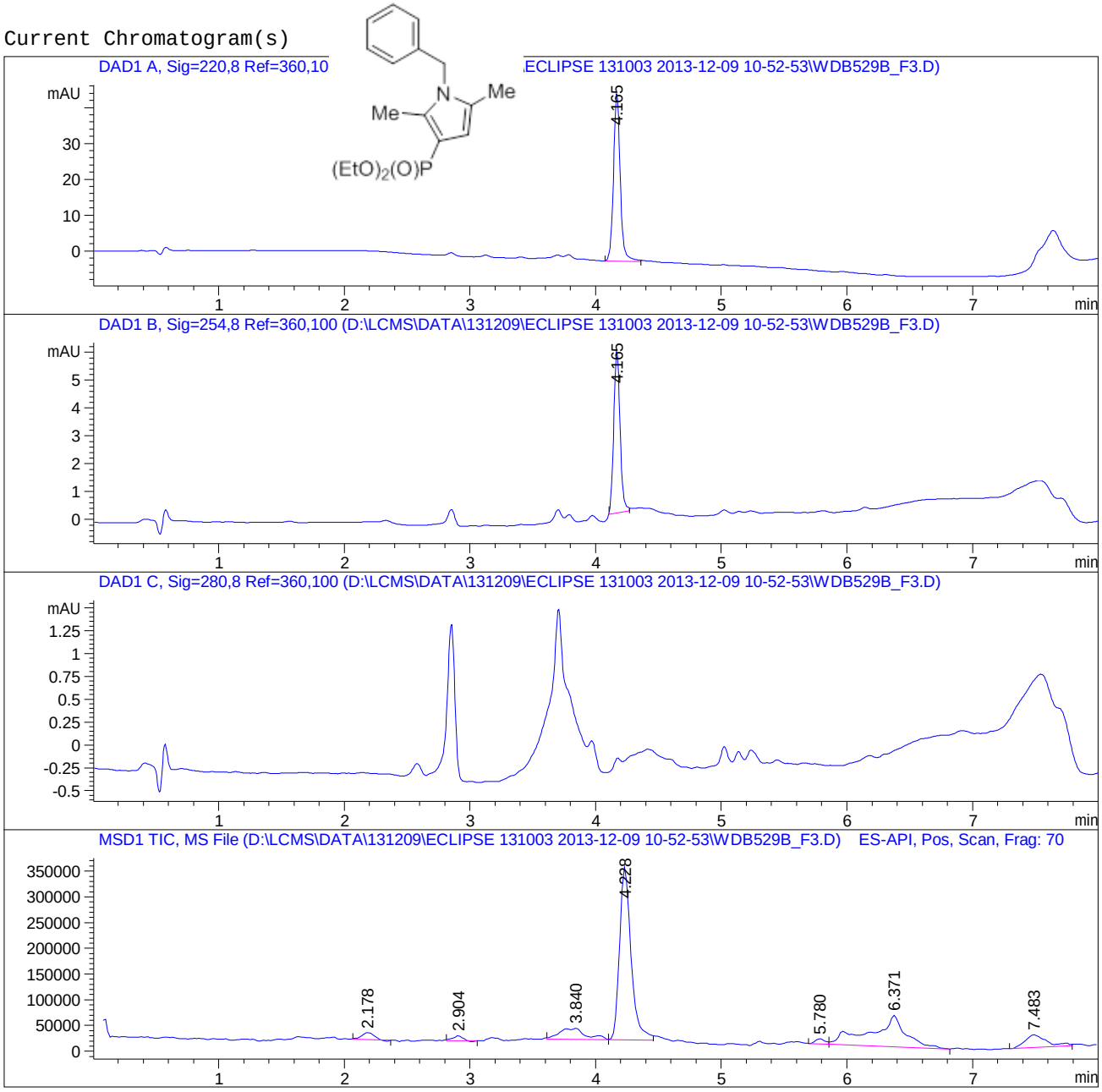
100 50 0 -50 -100 -150 -200 ppm

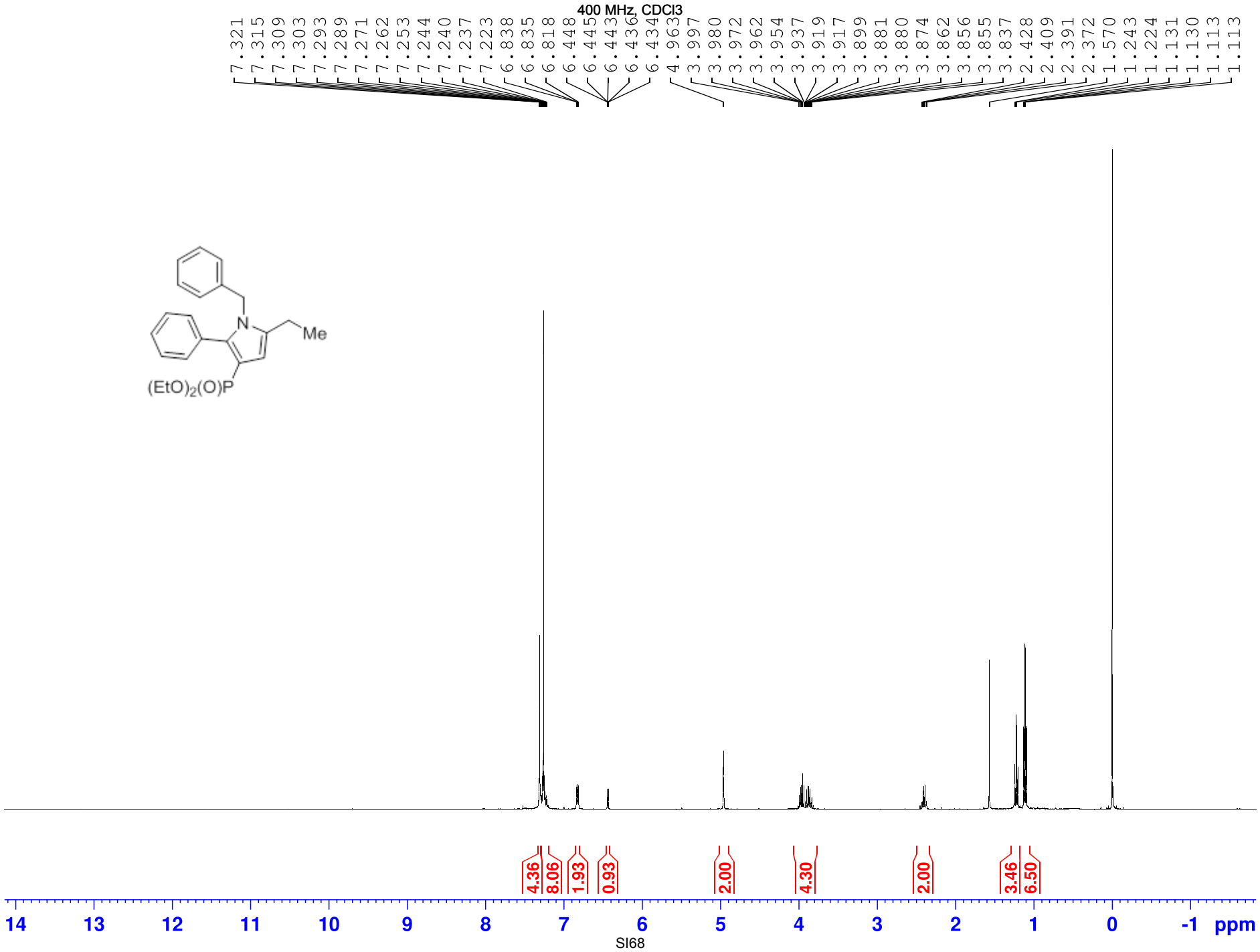

100 MHz, CDCl3
136.99
136.42
136.17
129.18
129.03
128.87
127.40
125.55
109.69
109.57
104.63
102.48
77.35
77.24
77.03
76.72
61.30
61.25
47.00
46.99
16.41
16.34
12.24
12.23
11.59
-0.00
N
Me
Me
(EtO)2(O)P
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

Print of window 38: Current Chromatogram(s)
Data File : D:\LCMS\DATA\131209\ECLIPSE 131003 2013-12-09 10-52-53\WDB529B_F3.D
Sample Name : WDB529b_f3

Acq. Operator : PC Seq. Line : 20
Acq. Instrument : Instrument 1 Location : Vial 84
Injection Date : 12/9/2013 2:06:54 PM Inj : 1
Inj Volume : 2.0 µl
Acq. Method : D:\LCMS\DATA\131209\ECLIPSE 131003 2013-12-09 10-52-53\ECLIPSE CH3CN LCMS1
Last changed : 9/11/2012 4:01:11 PM by PC
Analysis Method : D:\LCMS\METHODS\NEG ASCENTIS CH3CN LCMS11L.M
Last changed : 1/14/2014 1:45:24 PM by PC
(modified after loading)
Method Info : Ascentis column method for Synthesis samples

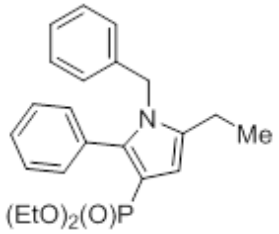
Current Chromatogram(s)



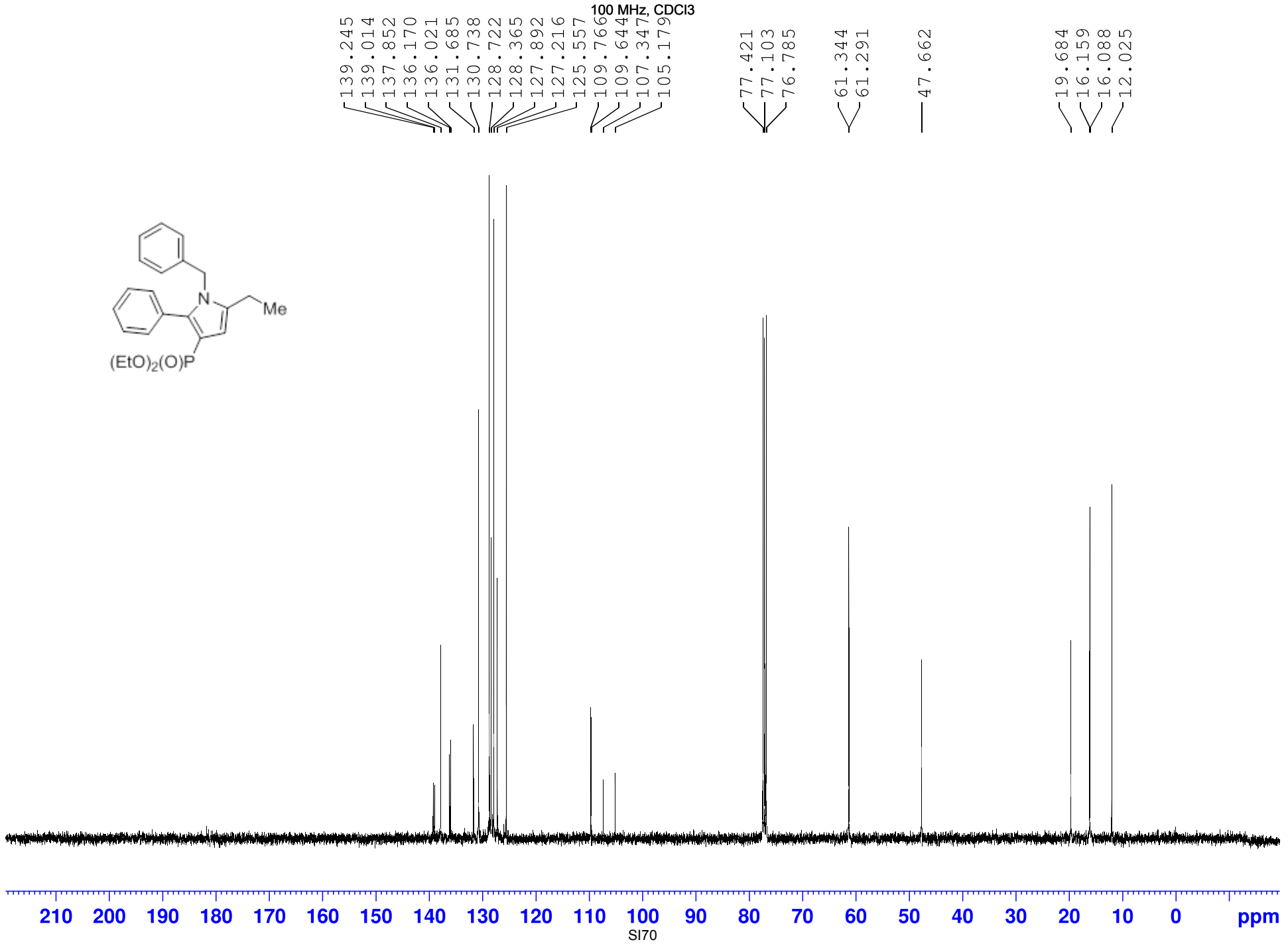

400 MHz, CDCl3
7.321
7.315
7.309
7.303
7.293
7.289
7.271
7.262
7.253
7.244
7.240
7.237
7.223
6.838
6.835
6.818
6.448
6.445
6.443
6.436
6.434
4.963
3.997
3.980
3.972
3.962
3.954
3.937
3.919
3.917
3.899
3.881
3.880
3.874
3.862
3.856
3.855
3.837
2.428
2.409
2.391
2.372
1.570
1.243
1.224
1.131
1.130
1.113
1.113
N
Me
(EtO)2(O)P
4.36
8.06
1.93
0.93
2.00
4.30
2.00
3.46
6.50
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm

162 MHz, CDCl3

18.53



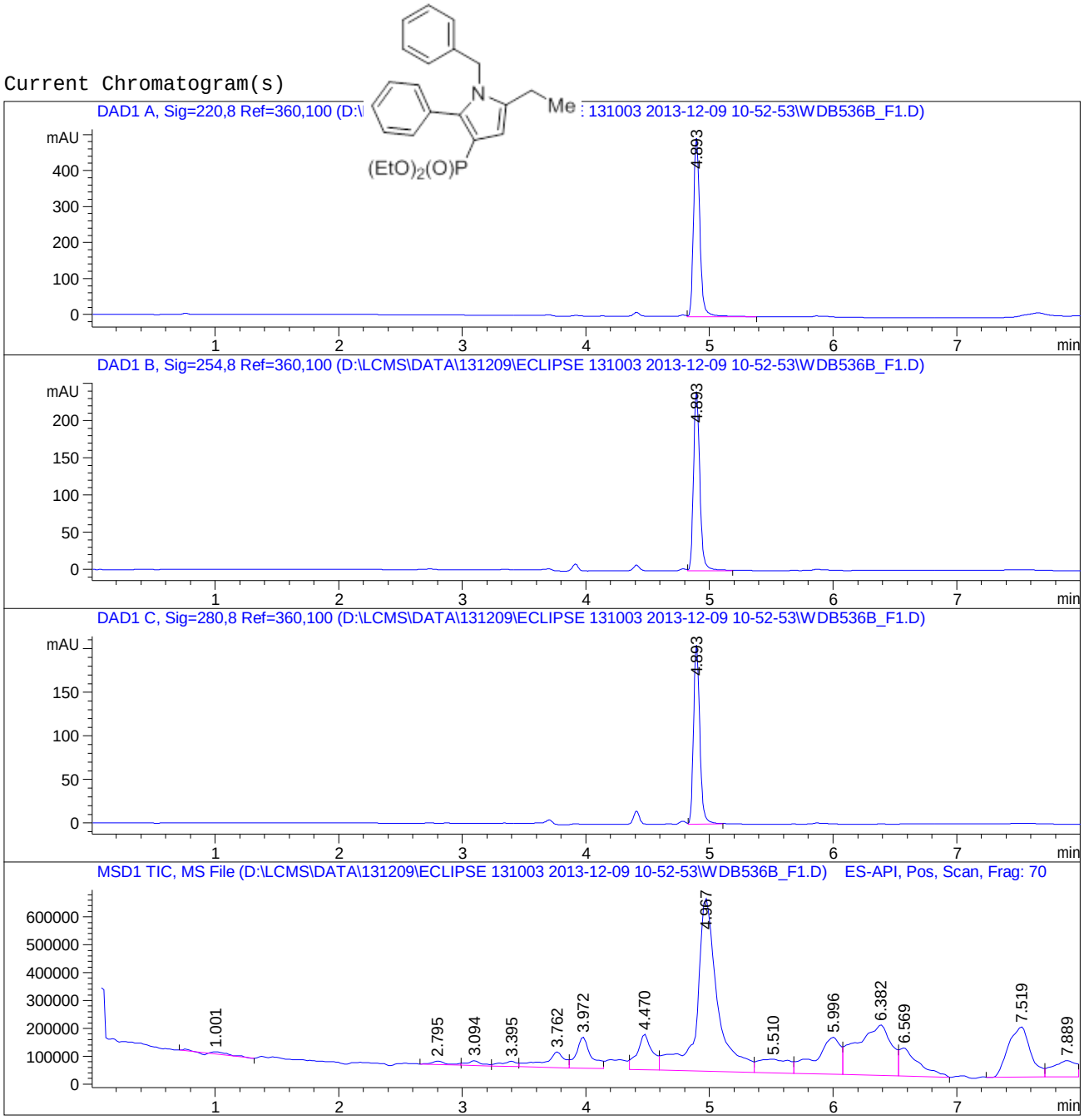
100 50 0 -50 -100 -150 -200 ppm

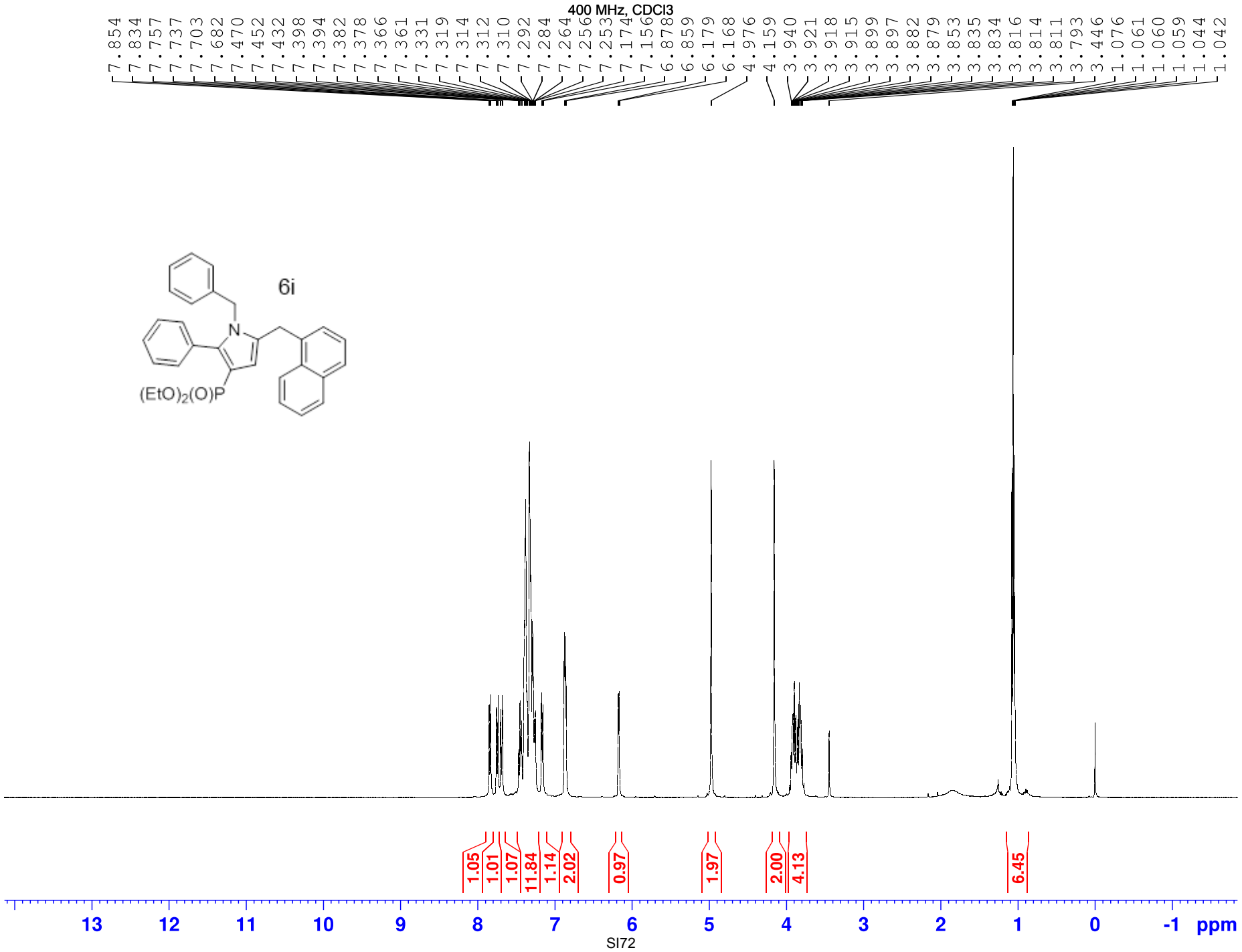

100 MHz, CDCl3
139.245
139.014
137.852
136.170
136.021
131.685
130.738
128.722
128.365
127.892
127.216
125.557
109.766
109.644
107.347
105.179
77.421
77.103
76.785
61.344
61.291
47.662
19.684
16.159
16.088
12.025
N
Me
(EtO)2(O)P
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

Print of window 38: Current Chromatogram(s)
Data File : D:\LCMS\DATA\131209\ECLIPSE 131003 2013-12-09 10-52-53\WDB536B_F1.D
Sample Name : WDB536b_f1

Acq. Operator : PC Seq. Line : 9
Acq. Instrument : Instrument 1 Location : Vial 82
Injection Date : 12/9/2013 12:22:32 PM Inj : 1
Inj Volume : 2.0 µl
Acq. Method : D:\LCMS\DATA\131209\ECLIPSE 131003 2013-12-09 10-52-53\ECLIPSE CH3CN LCMS1
Last changed : 9/11/2012 4:01:11 PM by PC
Analysis Method : D:\LCMS\METHODS\NEG ASCENTIS CH3CN LCMS11L.M
Last changed : 1/14/2014 1:45:24 PM by PC
(modified after loading)
Method Info : Ascentis column method for Synthesis samples

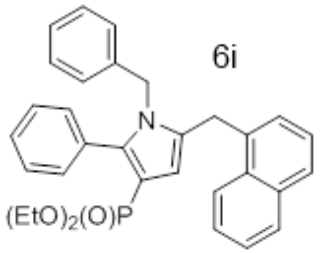
Current Chromatogram(s)



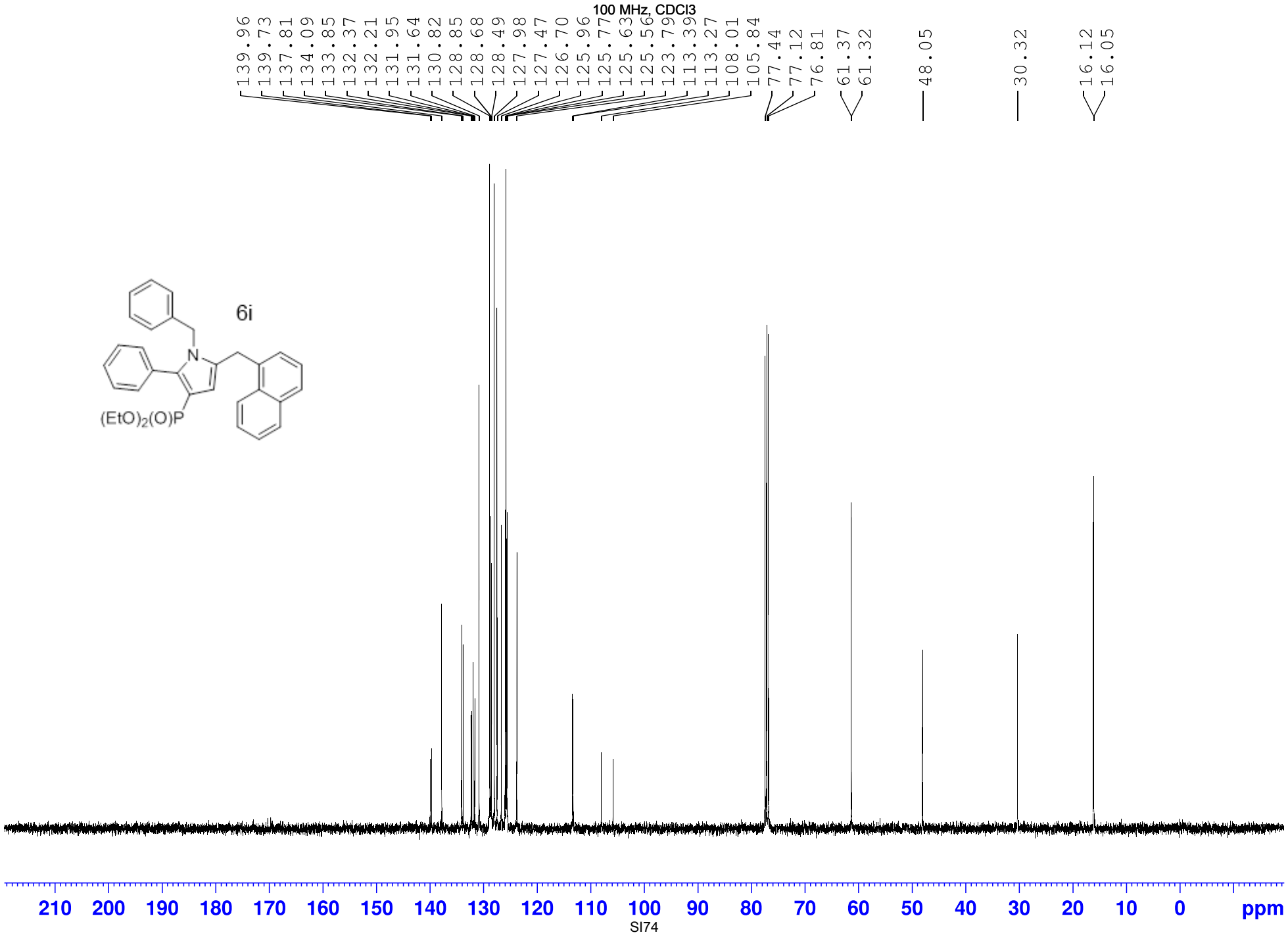

400 MHz, CDCl3
7.854
7.834
7.757
7.737
7.703
7.682
7.470
7.452
7.432
7.398
7.394
7.382
7.378
7.366
7.361
7.331
7.319
7.314
7.312
7.310
7.292
7.284
7.264
7.256
7.253
7.174
7.156
6.8788
6.859
6.179
6.168
4.976
4.159
3.940
3.921
3.918
3.915
3.899
3.897
3.882
3.879
3.853
3.835
3.834
3.816
3.814
3.811
3.793
3.446
1.076
1.061
1.060
1.059
1.044
1.042
6i
N
(EtO)2(O)P
1.05
1.01
1.07
11.84
1.14
2.02
0.97
1.97
2.00
4.13
6.45
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm

162 MHz, CDCl3

18.06



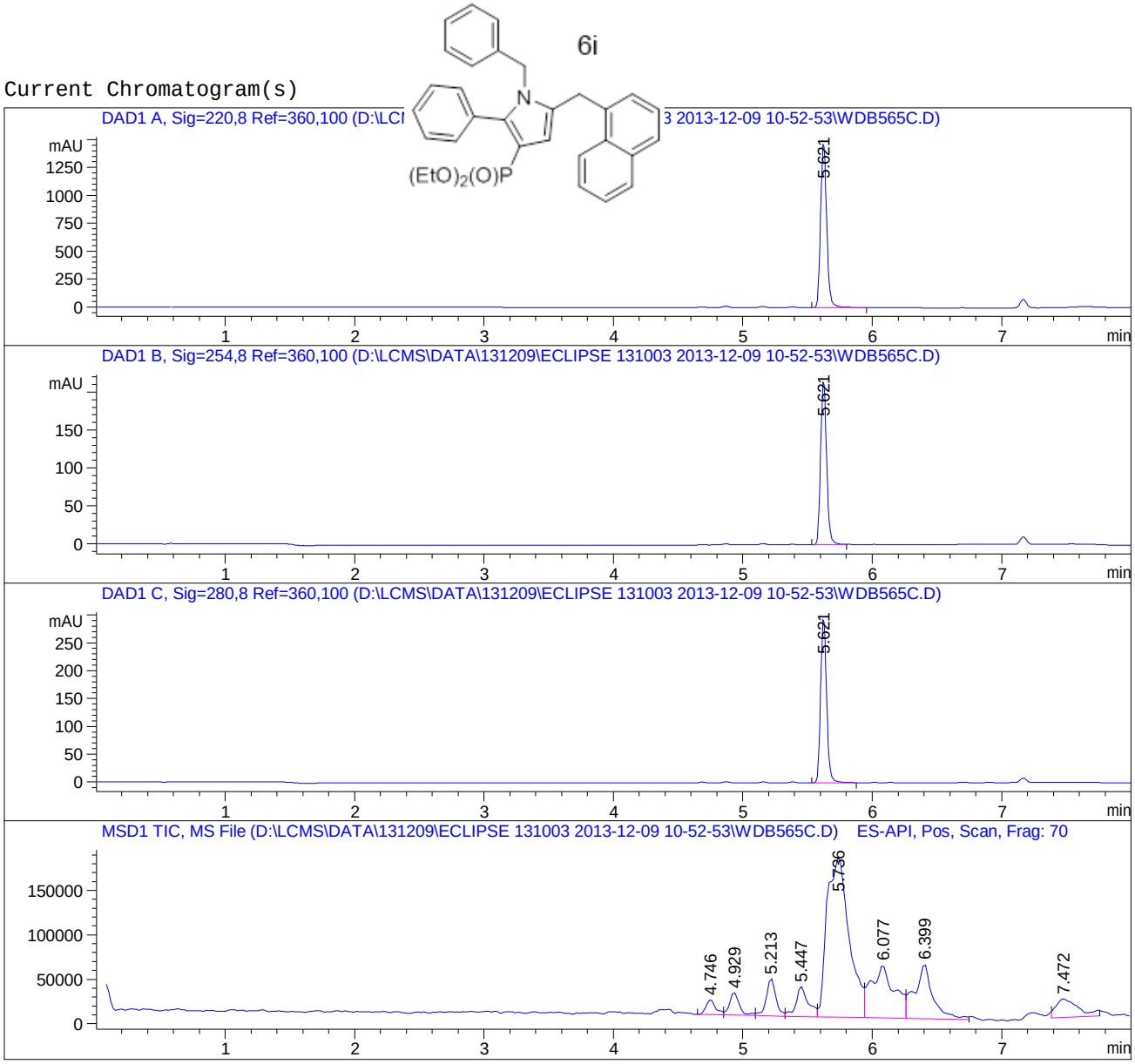
100 50 0 -50 -100 -150 -200 ppm


100 MHz, CDCl3
139.96
139.73
137.81
134.09
133.85
132.37
132.21
131.95
131.64
130.82
128.85
128.68
128.49
127.98
127.47
126.70
125.96
125.77
125.63
125.56
123.79
113.39
113.27
108.01
105.84
77.44
77.12
76.81
61.37
61.32
48.05
30.32
16.12
16.05
6i
N
(EtO)2(O)P
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

Print of window 38: Current Chromatogram(s)
Data File : D:\LCMS\DATA\131209\ECLIPSE 131003 2013-12-09 10-52-53\WDB565C.D
Sample Name : WDB565c

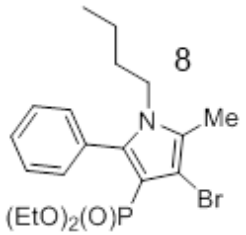
Acq. Operator : PC Seq. Line : 32
Acq. Instrument : Instrument 1 Location : Vial 85
Injection Date : 12/9/2013 4:01:27 PM Inj : 1
Inj Volume : 2.0 µl
Acq. Method : D:\LCMS\DATA\131209\ECLIPSE 131003 2013-12-09 10-52-53\ECLIPSE CH3CN LCMS1
Last changed : 9/11/2012 4:01:11 PM by PC
Analysis Method : D:\LCMS\METHODS\NEG ASCENTIS CH3CN LCMS11L.M
Last changed : 1/14/2014 1:45:24 PM by PC
(modified after loading)
Method Info : Ascentis column method for Synthesis samples

Current Chromatogram(s)



400 MHz, CDCl3

7.412
7.408
7.404
7.403
7.401
7.398
7.395
7.392
7.389
7.387
7.326
7.322
7.319
7.315
7.313
7.312
7.311
7.310
7.307
7.305
7.302
7.263
3.996
3.978
3.971
3.960
3.953
3.935
3.860
3.843
3.840
3.835
3.823
3.818
3.815
3.798
3.639
3.624
3.620
3.616
3.600
2.279
2.278
1.425
1.406
1.143
1.141
1.134
1.129
1.125
1.124
1.115
1.107
1.106
1.096
1.078
0.757
0.739
0.721
-0.000

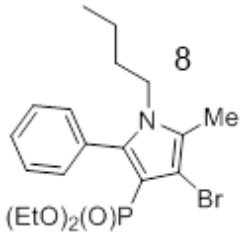


3.31
2.11
2.26
2.39
2.13
3.00
2.28
8.80
3.47

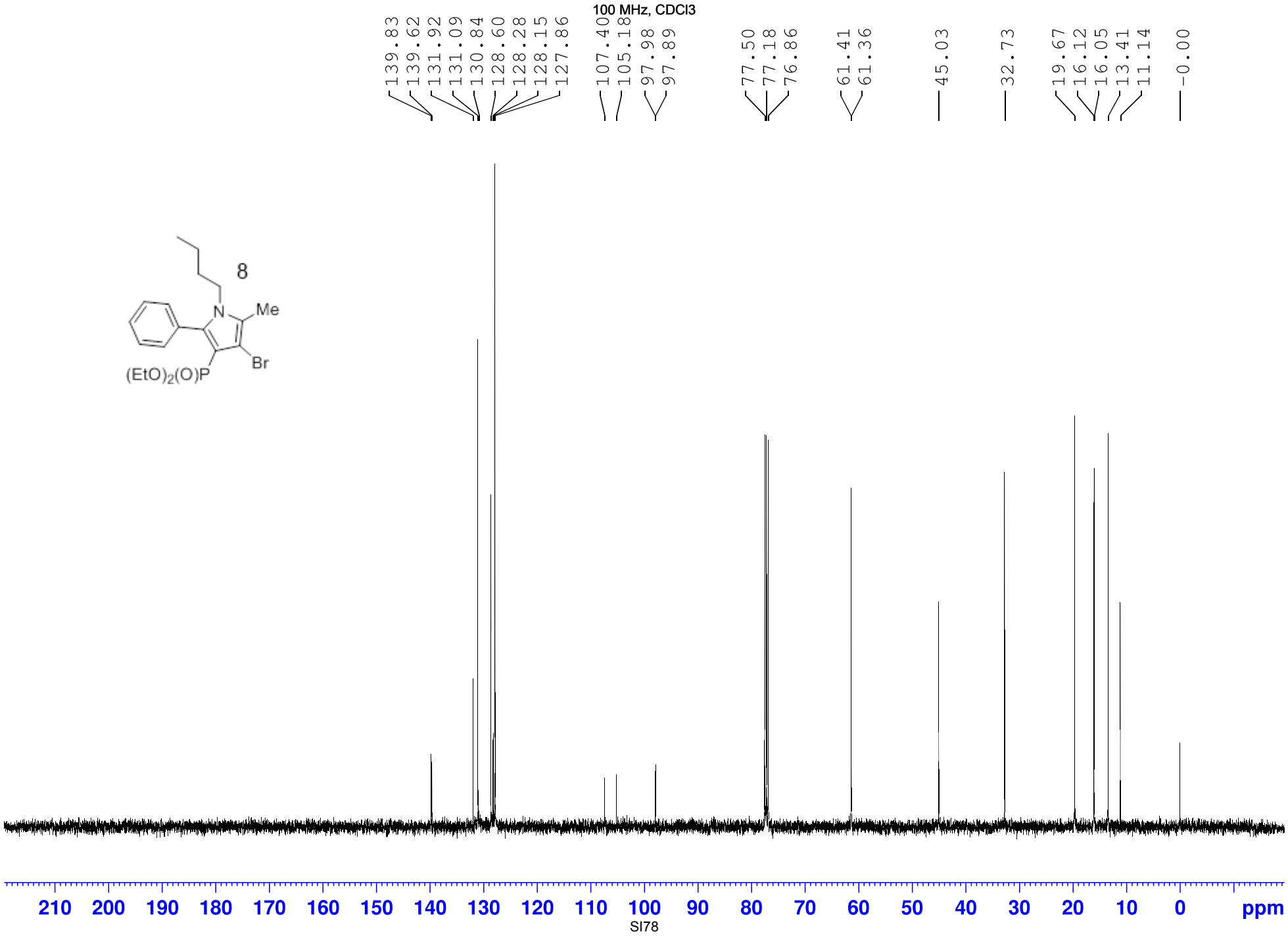
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 ppm

162 MHz, CDCl3

14.12



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

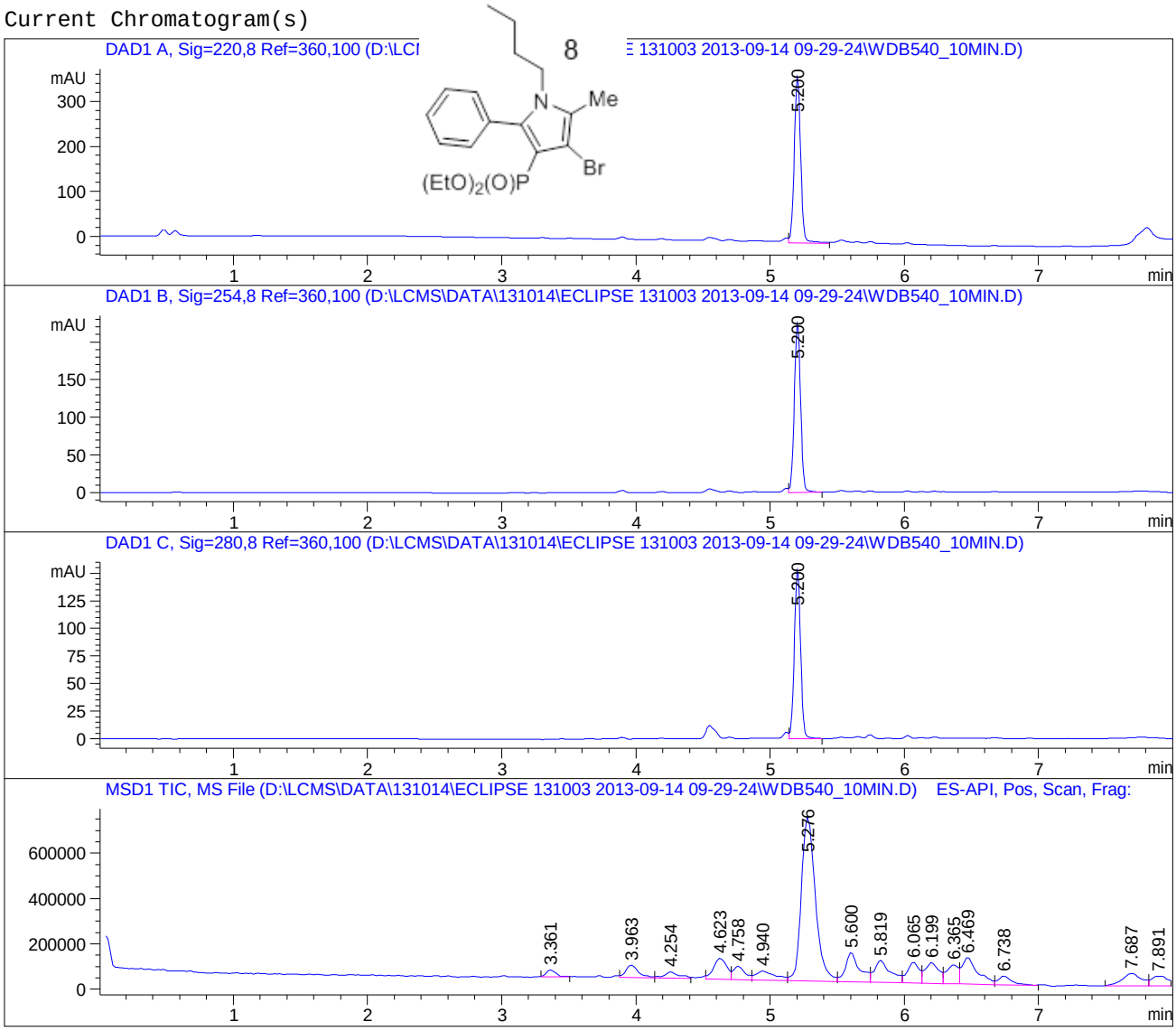

100 MHz, CDCl3
139.83
139.62
131.92
131.09
130.84
128.60
128.28
128.15
127.86
107.40
105.18
97.98
97.89
77.50
77.18
76.86
61.41
61.36
45.03
32.73
19.67
16.12
16.05
13.41
11.14
-0.00
8
N
Me
Br
(EtO)2(O)P
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

Print of window 38: Current Chromatogram(s)
Data File : D:\LCMS\DATA\131014\ECLIPSE 131003 2013-09-14 09-29-24\WDB540_10MIN.D
Sample Name : WDB540_10min

Acq. Operator	: PC	Seq. Line	: 2
Acq. Instrument	: Instrument 1	Location	: Vial 1
Injection Date	: 9/14/2013 9:42:45 AM	Inj	: 1
		Inj Volume	: 2.0 µl

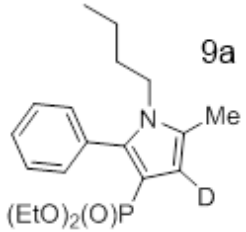
Acq. Method : D:\LCMS\DATA\131014\ECLIPSE 131003 2013-09-14 09-29-24\ECLIPSE CH3CN LCMS1
Last changed : 9/11/2012 4:01:11 PM by PC
Analysis Method : D:\LCMS\METHODS\NEG ASCENTIS CH3CN LCMS11L.M
Last changed : 1/14/2014 1:45:24 PM by PC
(modified after loading)
Method Info : Ascentis column method for Synthesis samples

Current Chromatogram(s)



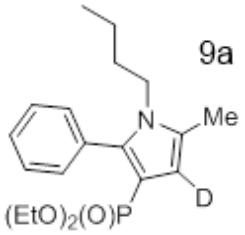
400 MHz, CDCl3

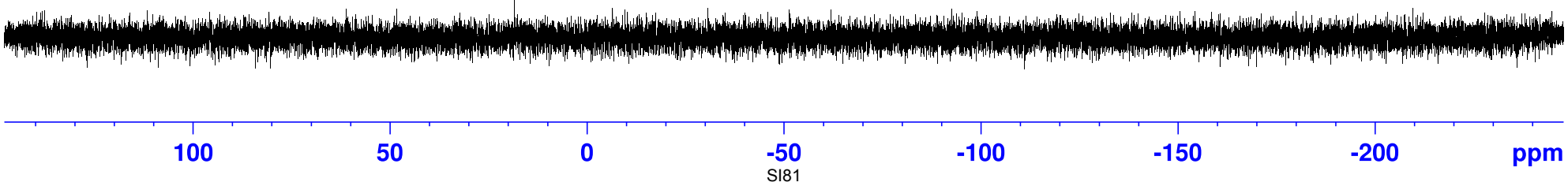
7.405 7.399 7.392 7.387 7.383 7.375 7.368 7.364 7.260 3.949 3.941 3.931 3.923 3.913 3.906 3.895 3.888 3.875 3.870 3.857 3.850 3.839 3.838 3.832 3.820 3.814 3.813 3.802 3.795 3.701 3.682 3.662 2.273 1.487 1.468 1.464 1.454 1.448 1.442 1.433 1.430 1.425 1.419 1.410 1.255 1.146 1.127 1.116 1.115 1.108 1.098 1.097 1.090 1.081 1.080 1.072 0.766 0.748 0.729 -0.000

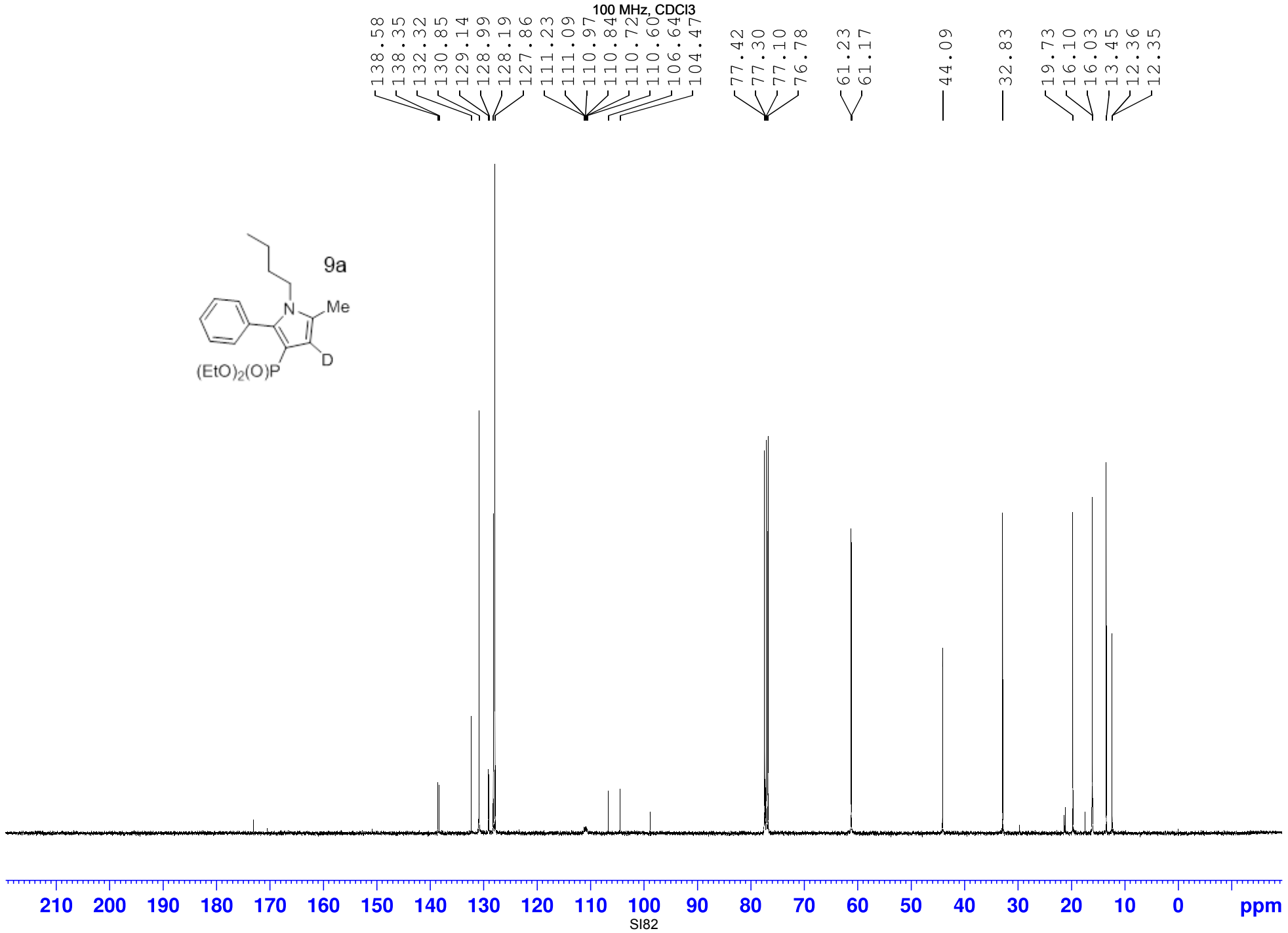


162 MHz, CDCl3

18.44



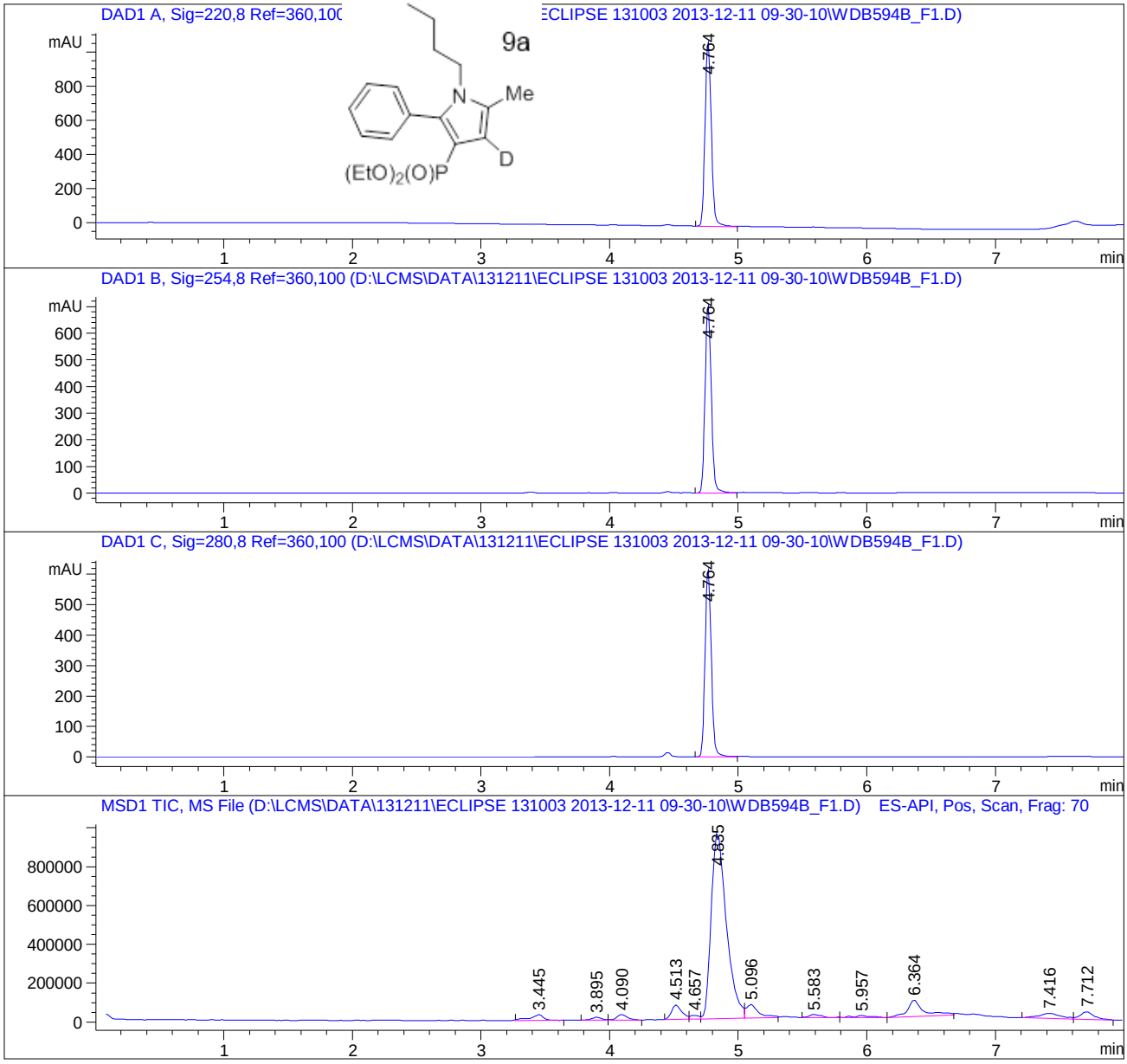


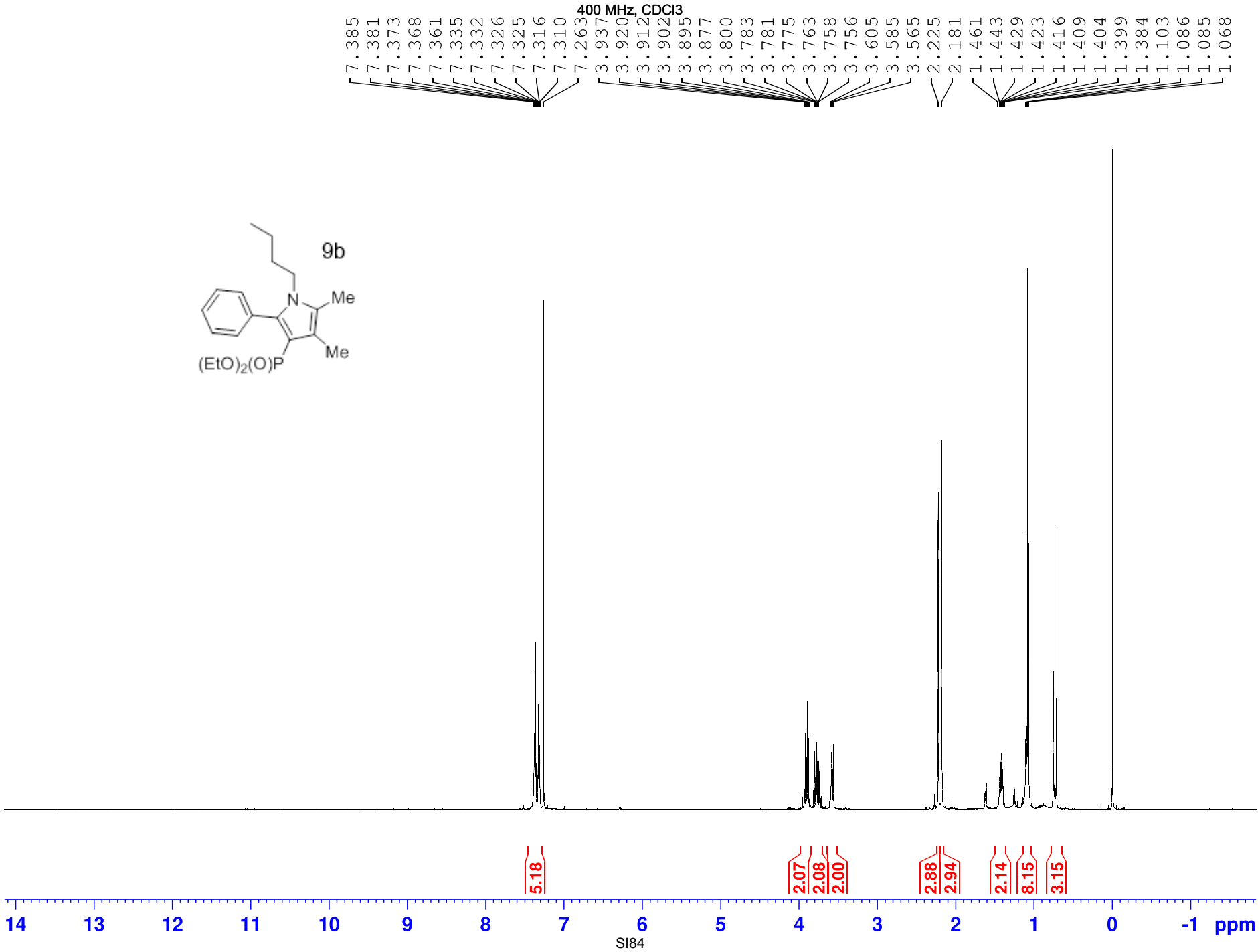

100 MHz, CDCl3
138.58
138.35
132.32
130.85
129.14
128.99
128.19
127.86
111.23
111.09
110.97
110.84
110.72
110.60
106.64
104.47
77.42
77.30
77.10
76.78
61.23
61.17
44.09
32.83
19.73
16.10
16.03
13.45
12.36
12.35
9a
N
Me
D
(EtO)2(O)P
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

Print of window 38: Current Chromatogram(s)
Data File : D:\LCMS\DATA\131211\ECLIPSE 131003 2013-12-11 09-30-10\WDB594B_F1.D
Sample Name : WDB594b_f1

Acq. Operator : PC Seq. Line : 3
Acq. Instrument : Instrument 1 Location : Vial 2
Injection Date : 12/11/2013 9:52:58 AM Inj : 1
Inj Volume : 2.0 µl
Acq. Method : D:\LCMS\DATA\131211\ECLIPSE 131003 2013-12-11 09-30-10\ECLIPSE CH3CN LCMS1
Last changed : 9/11/2012 4:01:11 PM by PC
Analysis Method : D:\LCMS\METHODS\NEG ASCENTIS CH3CN LCMS11L.M
Last changed : 1/14/2014 1:45:24 PM by PC
(modified after loading)
Method Info : Ascentis column method for Synthesis samples

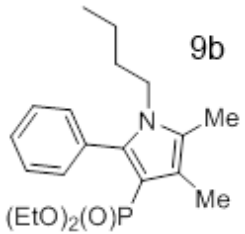
Current Chromatogram(s)



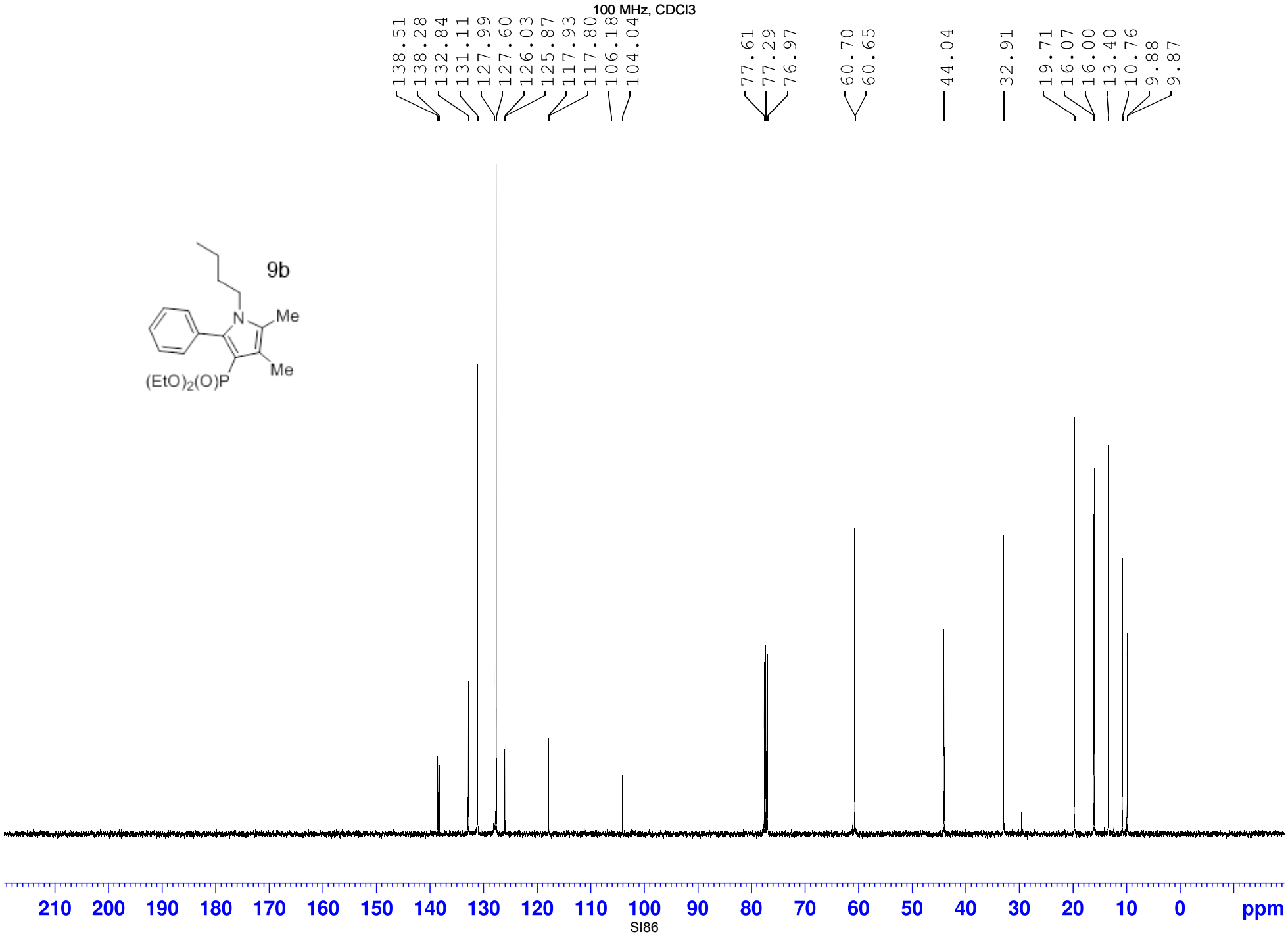

400 MHz, CDCl3
7.385
7.381
7.373
7.368
7.361
7.335
7.332
7.326
7.325
7.316
7.310
7.263
3.937
3.920
3.912
3.902
3.895
3.877
3.800
3.783
3.781
3.775
3.763
3.758
3.756
3.605
3.585
3.565
2.225
2.181
1.461
1.443
1.429
1.423
1.416
1.409
1.404
1.399
1.384
1.103
1.086
1.085
1.068
9b
N
Me
Me
(EtO)2(O)P
5.18
2.07
2.08
2.00
2.88
2.94
2.14
8.15
3.15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm

162 MHz, CDCl3

18.56



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

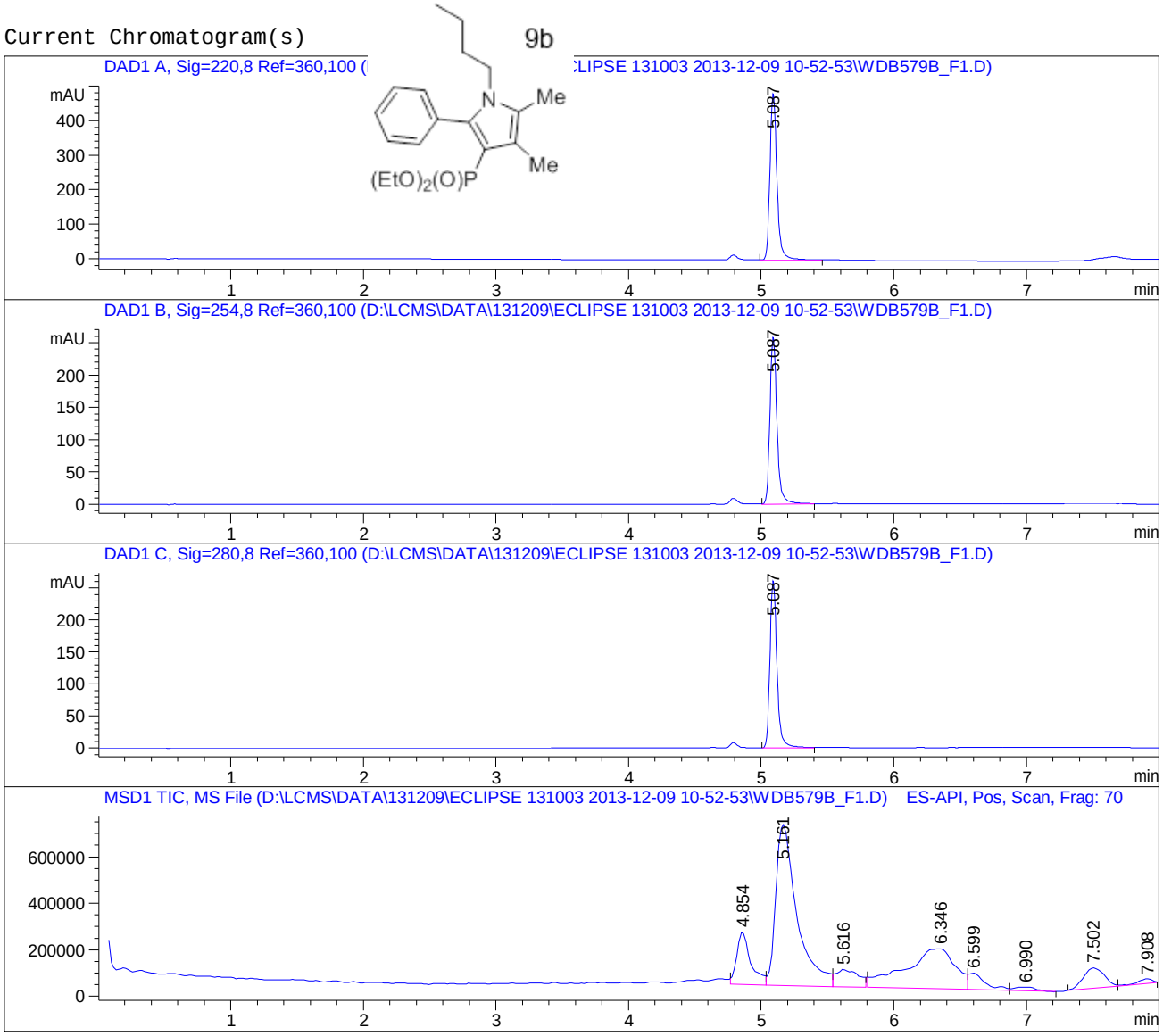

100 MHz, CDCl3
138.51
138.28
132.84
131.11
127.99
127.60
126.03
125.87
117.93
117.80
106.18
104.04
77.61
77.29
76.97
60.70
60.65
44.04
32.91
19.71
16.07
16.00
13.40
10.76
9.88
9.87
9b
N
Me
Me
(EtO)2(O)P
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

Print of window 38: Current Chromatogram(s)
Data File : D:\LCMS\DATA\131209\ECLIPSE 131003 2013-12-09 10-52-53\WDB579B_F1.D
Sample Name : WDB579b_f1

Acq. Operator	: PC	Seq. Line	: 10
Acq. Instrument	: Instrument 1	Location	: Vial 83
Injection Date	: 12/9/2013 12:32:06 PM	Inj	: 1
		Inj Volume	: 2.0 µl

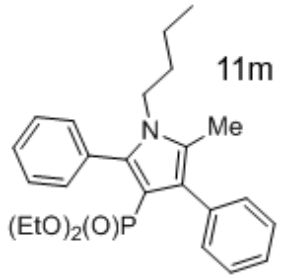
Acq. Method : D:\LCMS\DATA\131209\ECLIPSE 131003 2013-12-09 10-52-53\ECLIPSE CH3CN LCMS1
Last changed : 9/11/2012 4:01:11 PM by PC
Analysis Method : D:\LCMS\METHODS\NEG ASCENTIS CH3CN LCMS11L.M
Last changed : 1/14/2014 1:45:24 PM by PC
(modified after loading)
Method Info : Ascentis column method for Synthesis samples

Current Chromatogram(s)



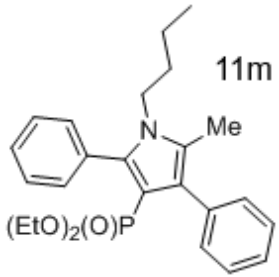
400 MHz, CDCl3

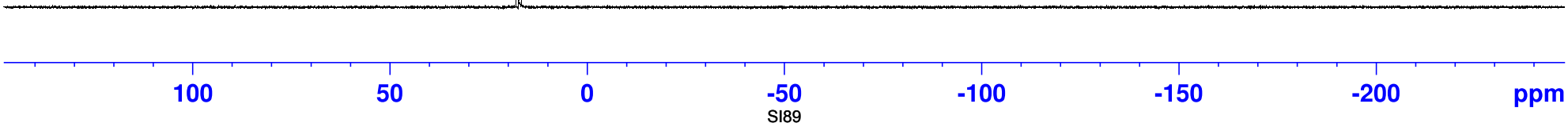
7.471 7.468 7.460 7.450 7.447 7.439 7.435 7.432 7.417 7.413 7.402 7.398 7.392 7.380 7.361 7.346 7.342 7.278 7.275 7.271 7.266 7.261 7.256 7.251 7.238 3.703 3.682 3.664 3.647 3.639 3.629 3.622 3.604 3.490 3.473 3.471 3.465 3.453 3.448 3.446 3.428 2.199 1.517 1.503 1.497 1.490 1.478 1.473 1.458 1.177 1.158 1.140 1.121 0.792 0.783 0.774 0.765 0.757 0.747 -0.000

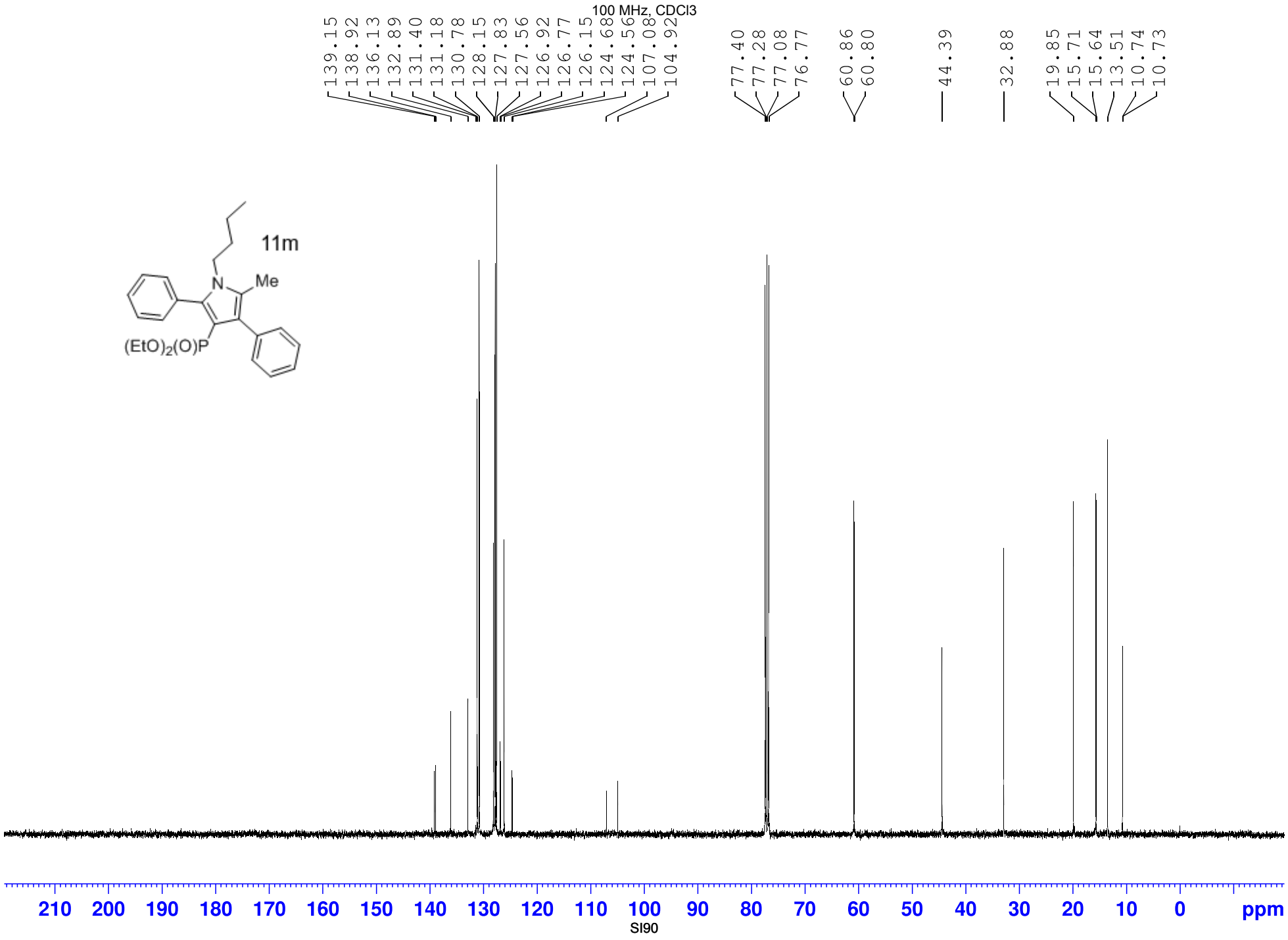


162 MHz, CDCl3

17.30





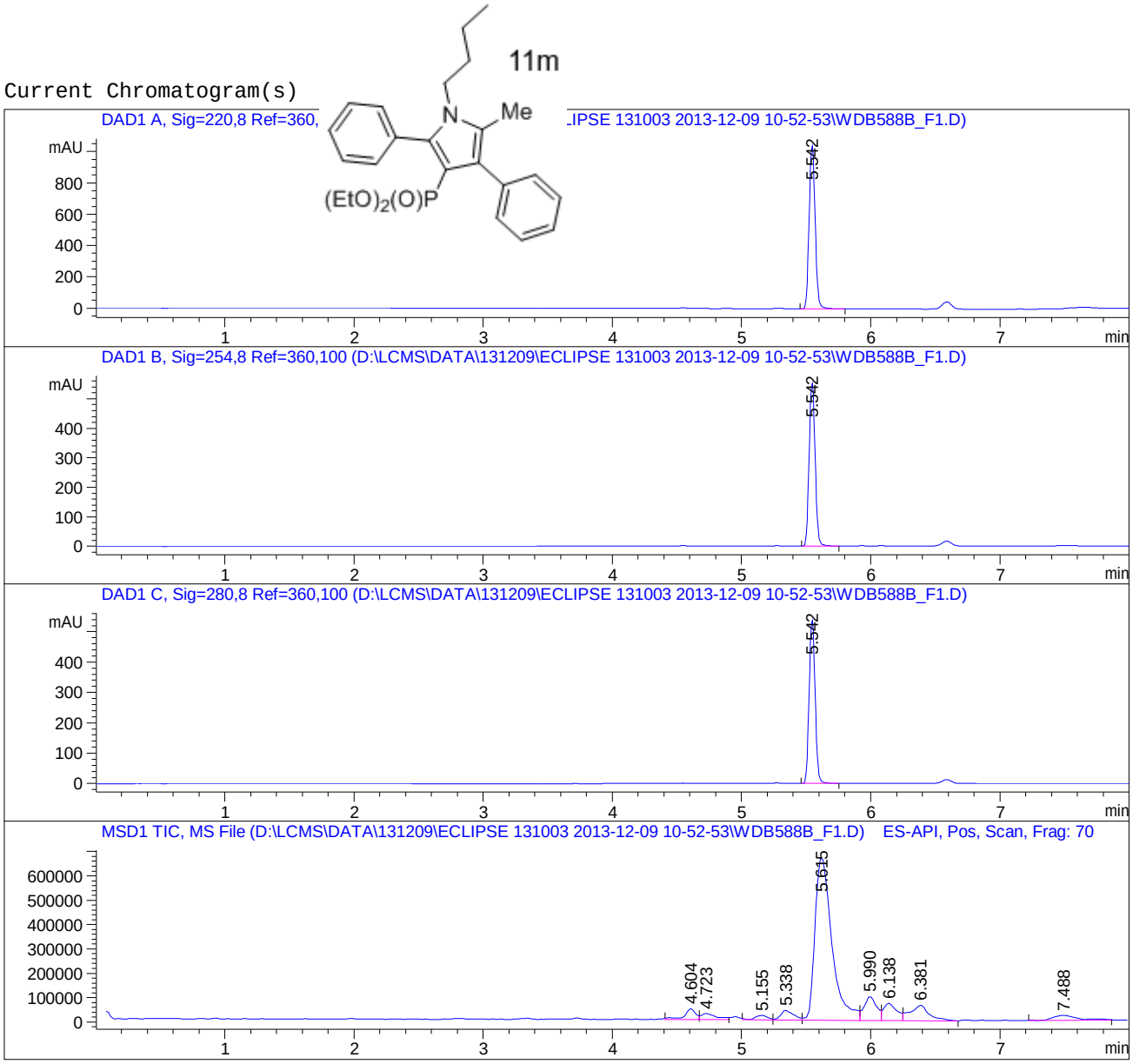

100 MHz, CDCl3
139.15
138.92
136.13
132.89
131.40
131.18
130.78
128.15
127.83
127.56
126.92
126.77
126.15
124.68
124.56
107.08
104.92
77.40
77.28
77.08
76.77
60.86
60.80
44.39
32.88
19.85
15.71
15.64
13.51
10.74
10.73
11m
N
Me
(EtO)2(O)P
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

Print of window 38: Current Chromatogram(s)
Data File : D:\LCMS\DATA\131209\ECLIPSE 131003 2013-12-09 10-52-53\WDB588B_F1.D
Sample Name : WDB588b_f1

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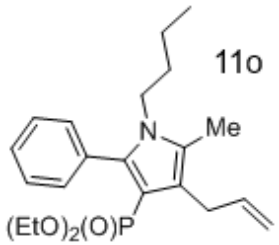
Acq. Operator : PC Seq. Line : 34
Acq. Instrument : Instrument 1 Location : Vial 86
Injection Date : 12/9/2013 4:20:38 PM Inj : 1
Inj Volume : 2.0 µl
Acq. Method : D:\LCMS\DATA\131209\ECLIPSE 131003 2013-12-09 10-52-53\ECLIPSE CH3CN LCMS1
Last changed : 9/11/2012 4:01:11 PM by PC
Analysis Method : D:\LCMS\METHODS\NEG ASCENTIS CH3CN LCMS11L.M
Last changed : 1/14/2014 1:45:24 PM by PC
(modified after loading)
Method Info : Ascentis column method for Synthesis samples

Current Chromatogram(s)



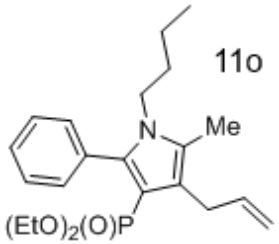
400 MHz, CDCl3

7.384 7.380 7.372 7.366 7.360 7.335 7.333 7.328 7.325 7.316 7.311 7.264 5.986 5.028 5.023 4.985 4.980 4.968 4.963 4.943 4.938 3.924 3.906 3.899 3.888 3.881 3.863 3.789 3.772 3.770 3.764 3.752 3.747 3.745 3.727 3.612 3.593 3.573 3.492 3.477 2.171 1.432 1.428 1.409 1.404 1.390 1.256 1.132 1.125 1.125 1.113 1.108 1.093 1.075 1.075 1.058 0.758 0.739 0.721 -0.000

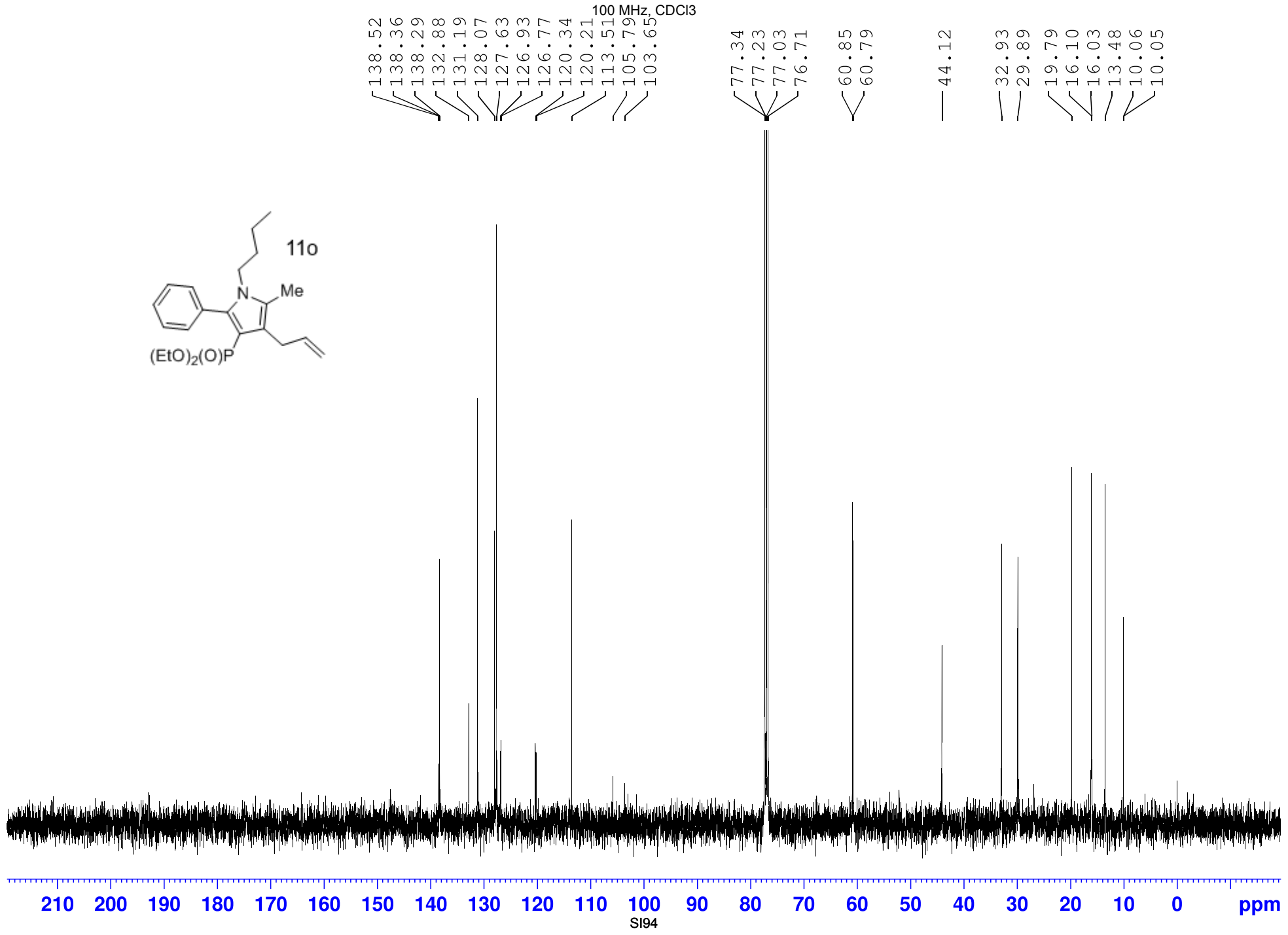


162 MHz, CDCl3

18.23



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


100 MHz, CDCl3
138.52
138.36
138.29
132.88
131.19
128.07
127.63
126.93
126.77
120.34
120.21
113.51
105.79
103.65
77.34
77.23
77.03
76.71
60.85
60.79
44.12
32.93
29.89
19.79
16.10
16.03
13.48
10.06
10.05
11o
N
Me
(EtO)2(O)P
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

Print of window 38: Current Chromatogram(s)
Data File : D:\LCMS\DATA\140303\ASCENTIS 06022014 LCMS 2014-03-03 13-05-52\WDB710B.D
Sample Name : WDB710b

Acq. Operator	: PC	Seq. Line	: 42
Acq. Instrument	: Instrument 1	Location	: Vial 86
Injection Date	: 3/3/2014 5:16:12 PM	Inj	: 1
		Inj Volume	: 2.0 µl
Acq. Method	: D:\LCMS\DATA\140303\ASCENTIS 06022014 LCMS 2014-03-03 13-05-52\ASCENTIS CH3CN LCMS		
Last changed	: 1/13/2014 4:31:43 PM by PC		
Analysis Method	: D:\LCMS\METHODS\COND ASCENTIS LCMS.M		
Last changed	: 1/14/2014 9:15:07 AM by PC		
Method Info	: Conditioning method Eclipse column		

Current Chromatogram(s)

