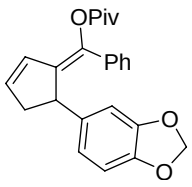
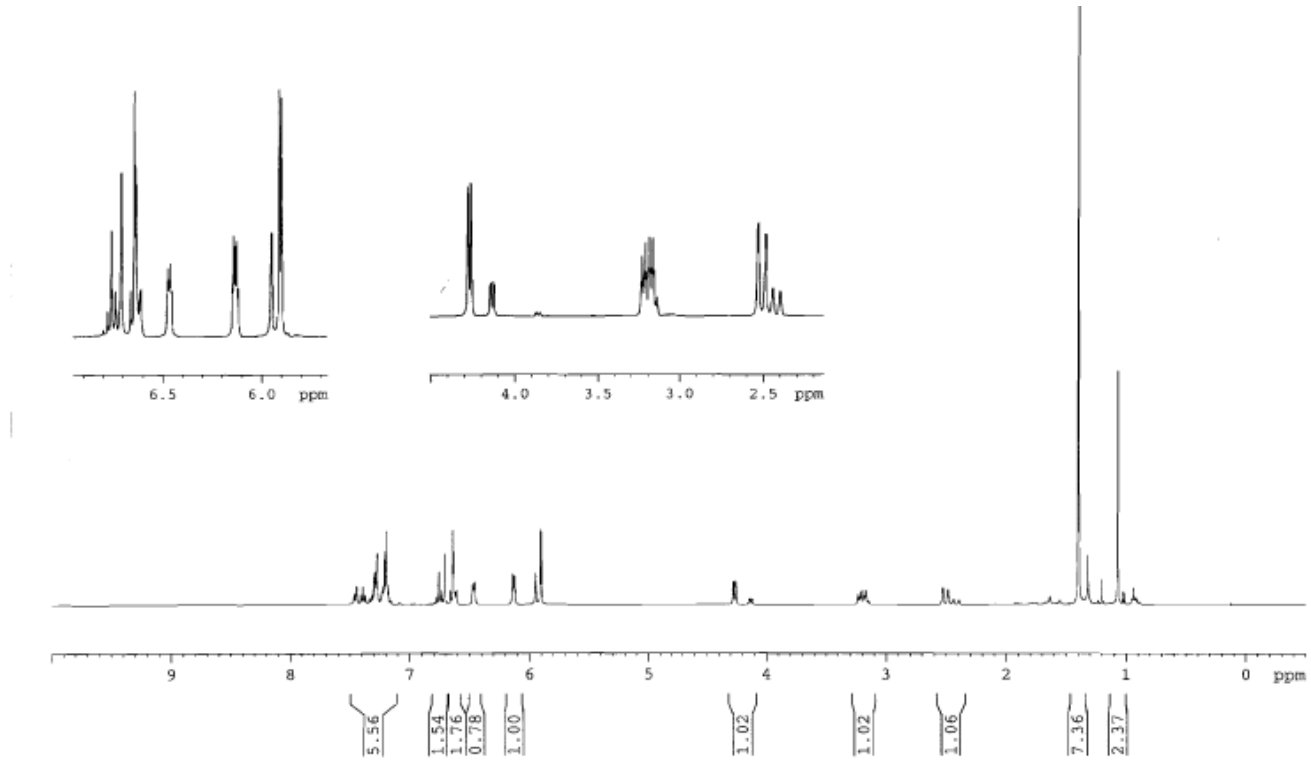
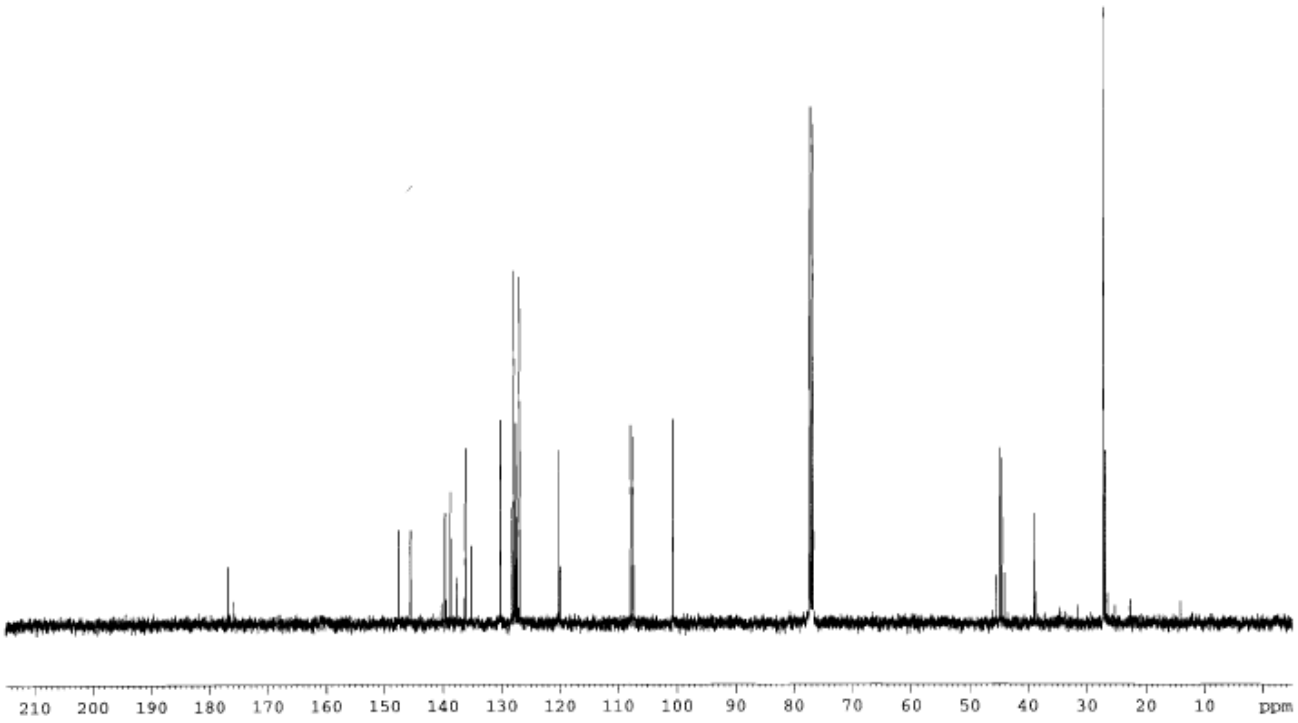
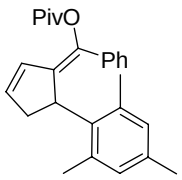
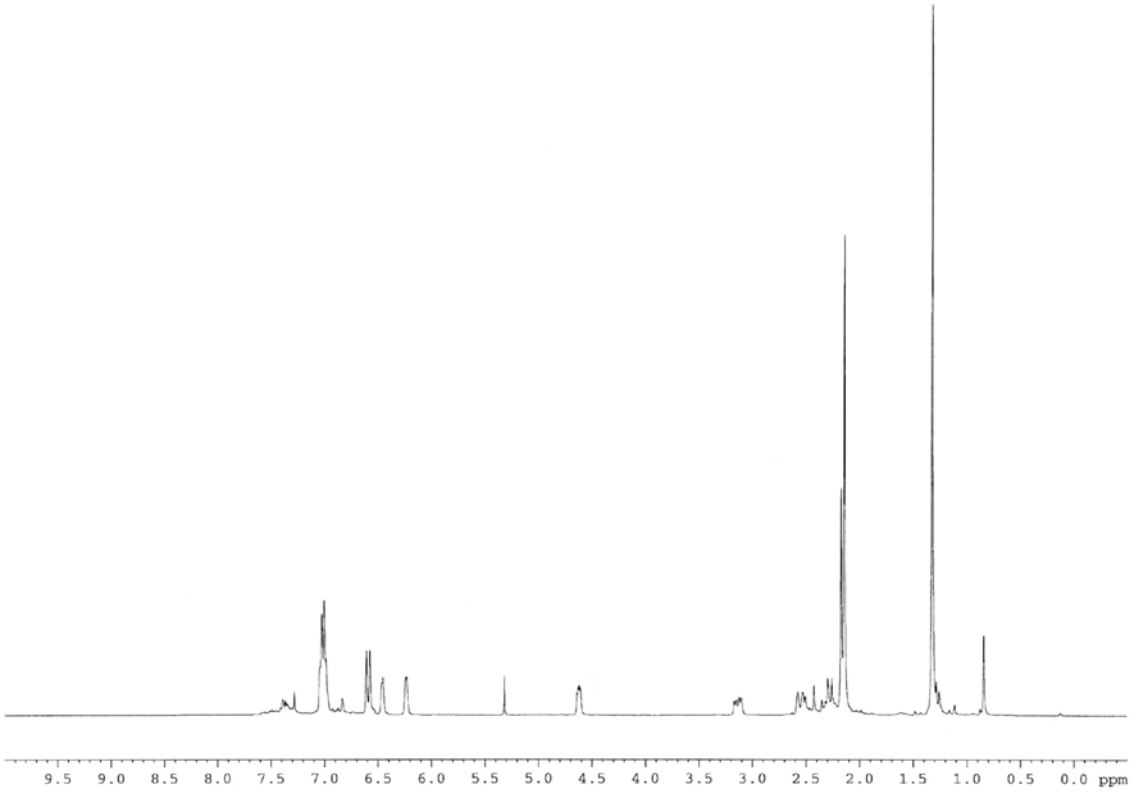
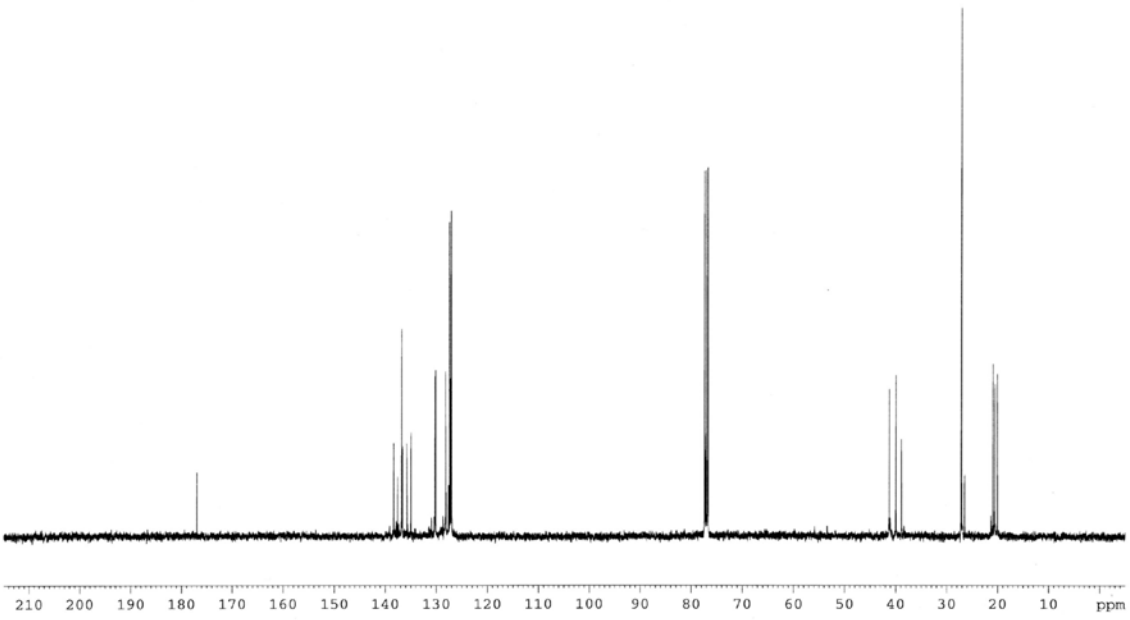

OPiv
Ph
O
O

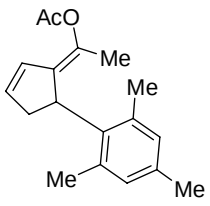

6.5
6.0 ppm
4.0
3.5
3.0
2.5 ppm
9
8
7
6
5
4
3
2
1
0 ppm
5.56
1.54
1.76
0.78
1.00
1.02
1.02
1.06
7.36
2.37

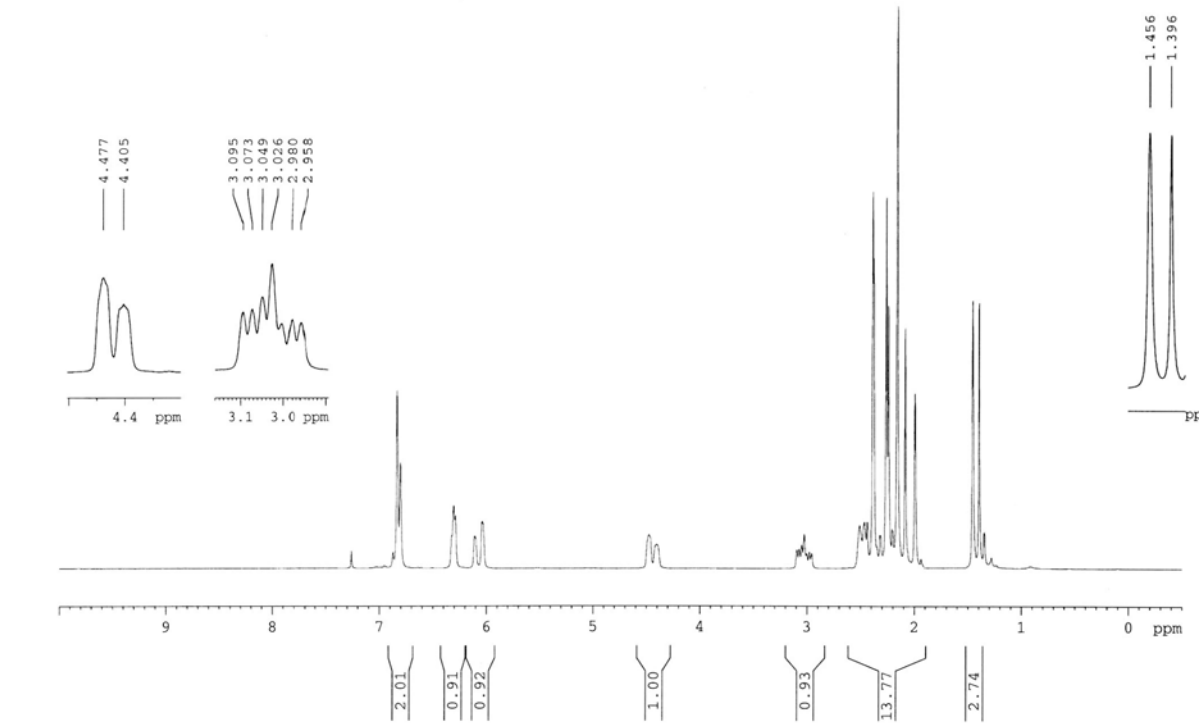

210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

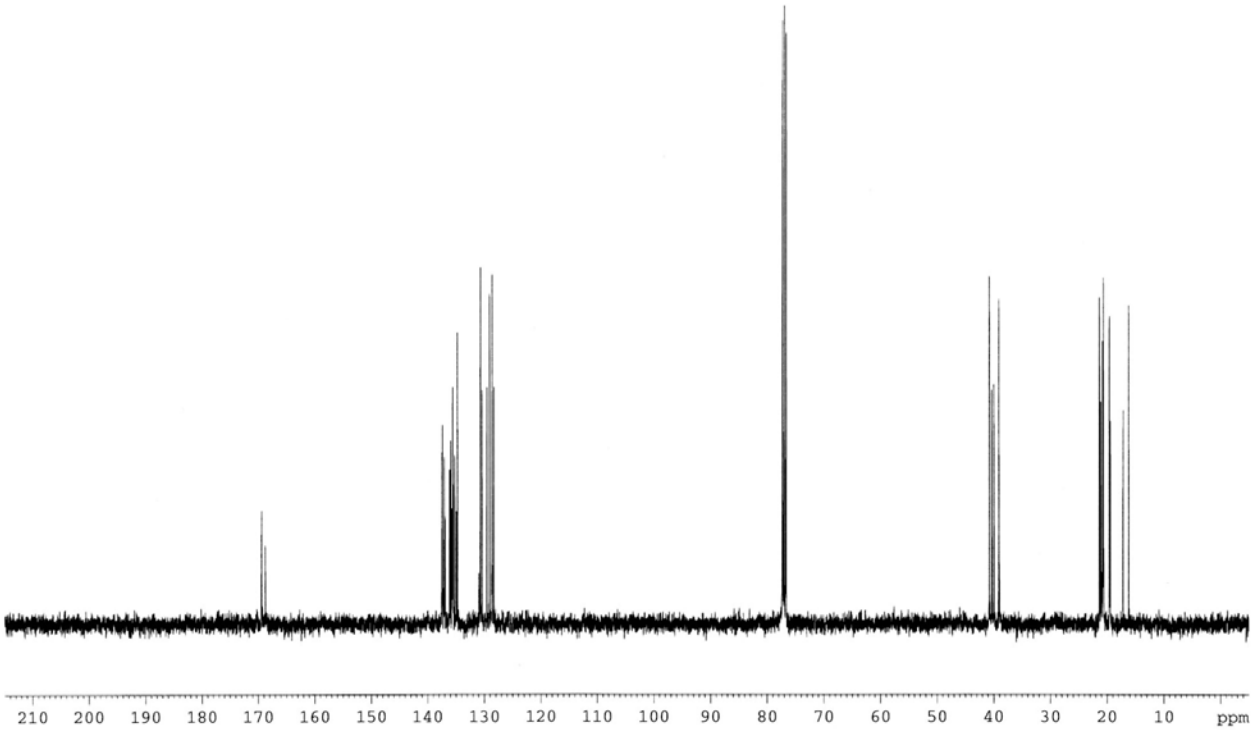

PivO
Ph

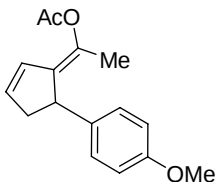

9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm

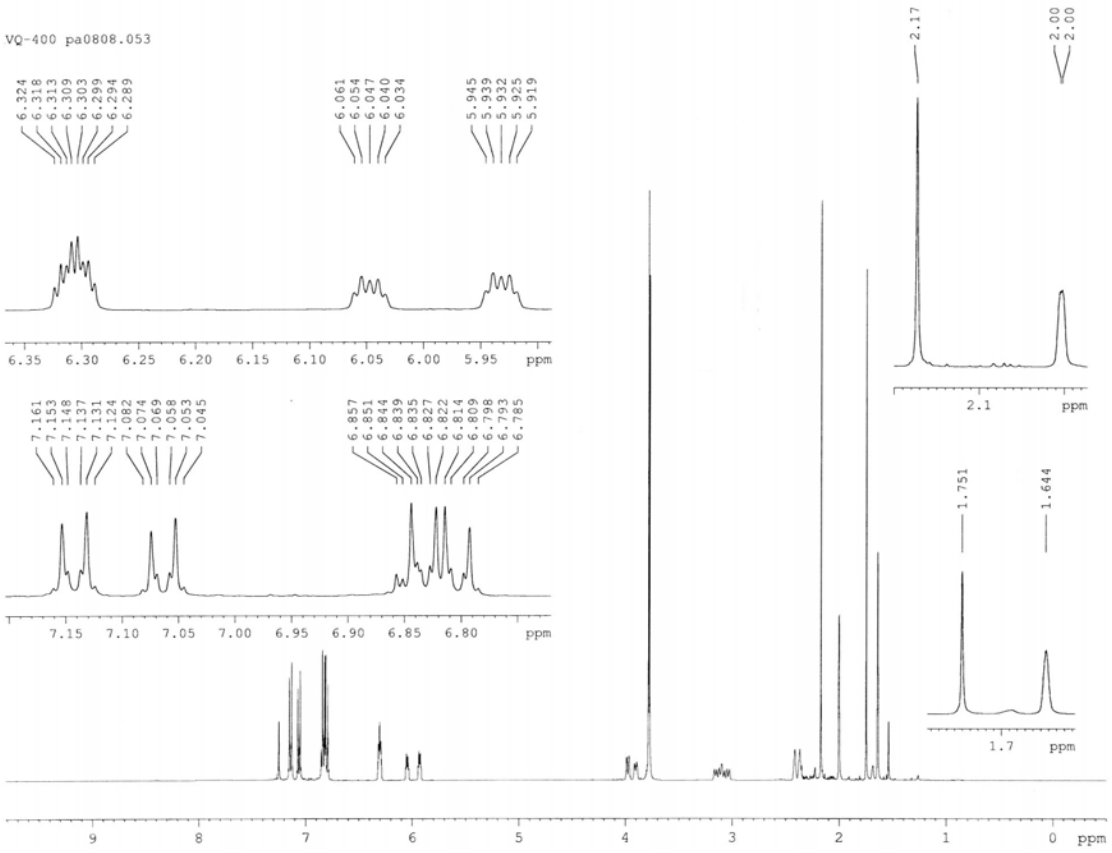

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

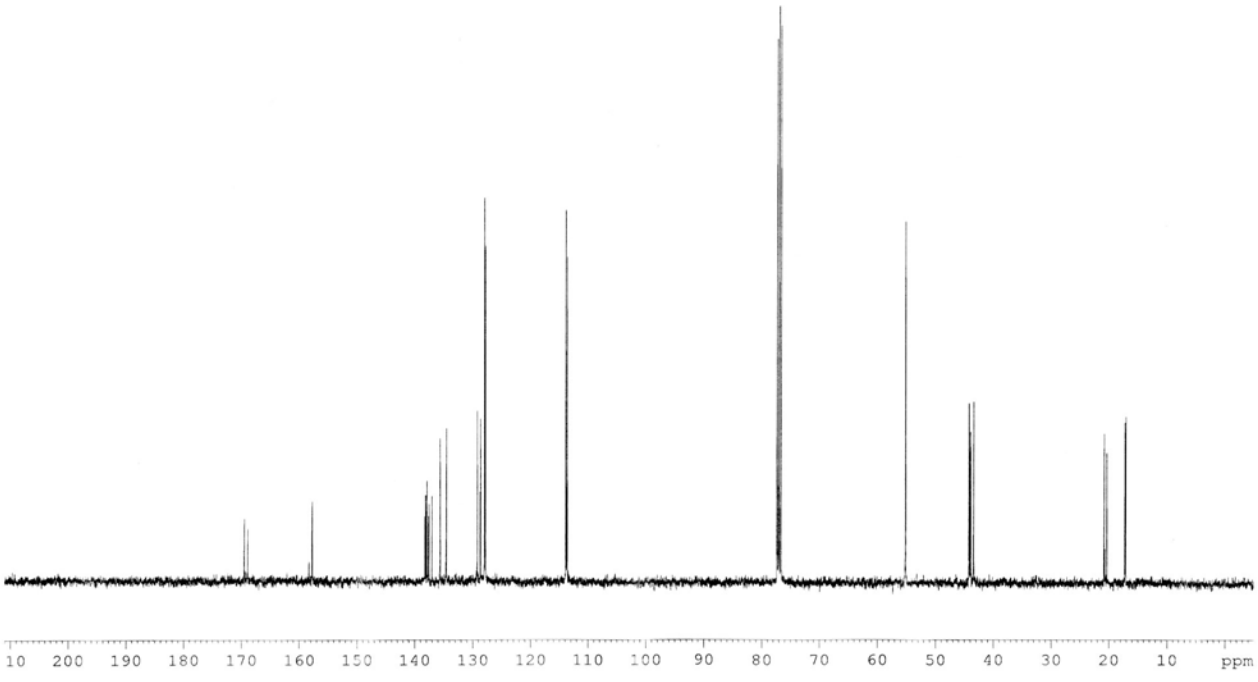

AcO
Me
Me
Me
Me

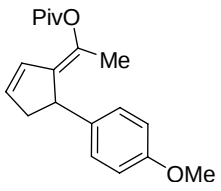

4.477
4.405
3.095
3.073
3.049
3.026
2.980
2.958
1.456
1.396
4.4 ppm
3.1 3.0 ppm
9 8 7 6 5 4 3 2 1 0 ppm
2.01
0.91
0.92
1.00
0.93
13.77
2.74

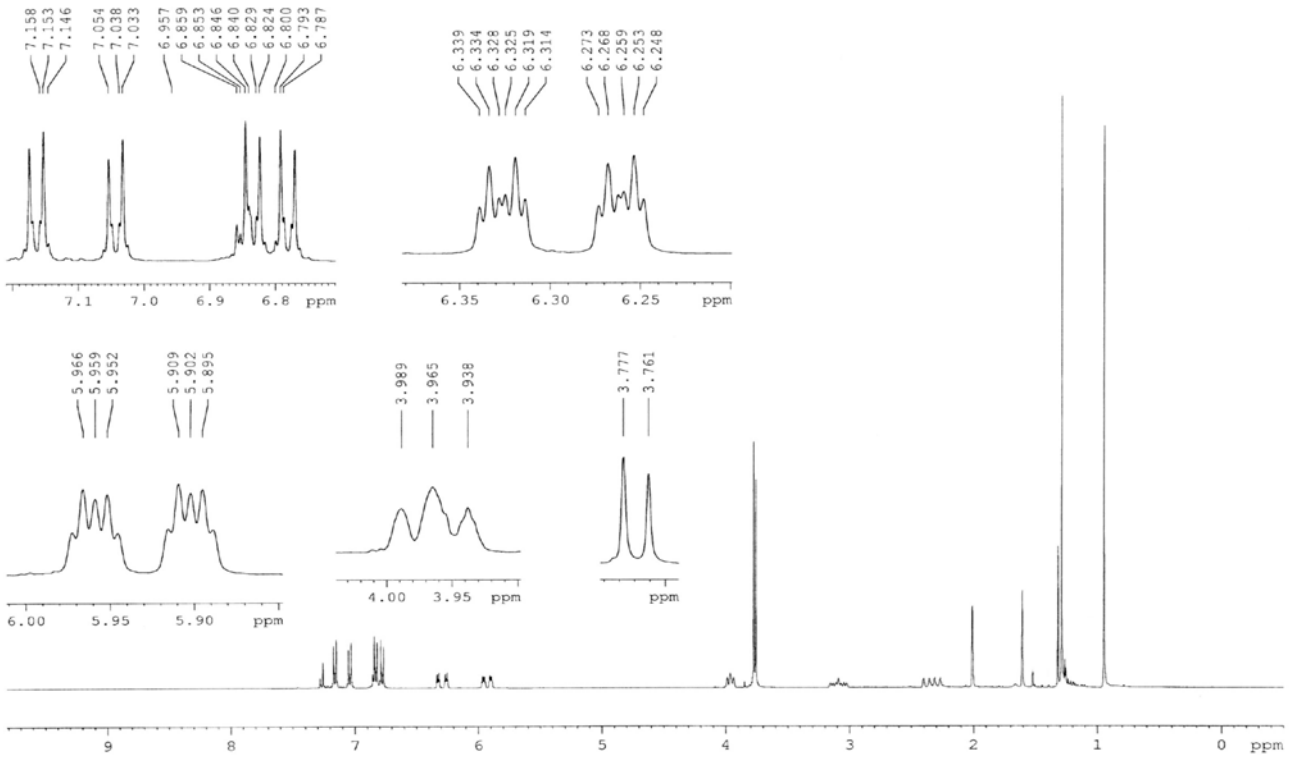

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

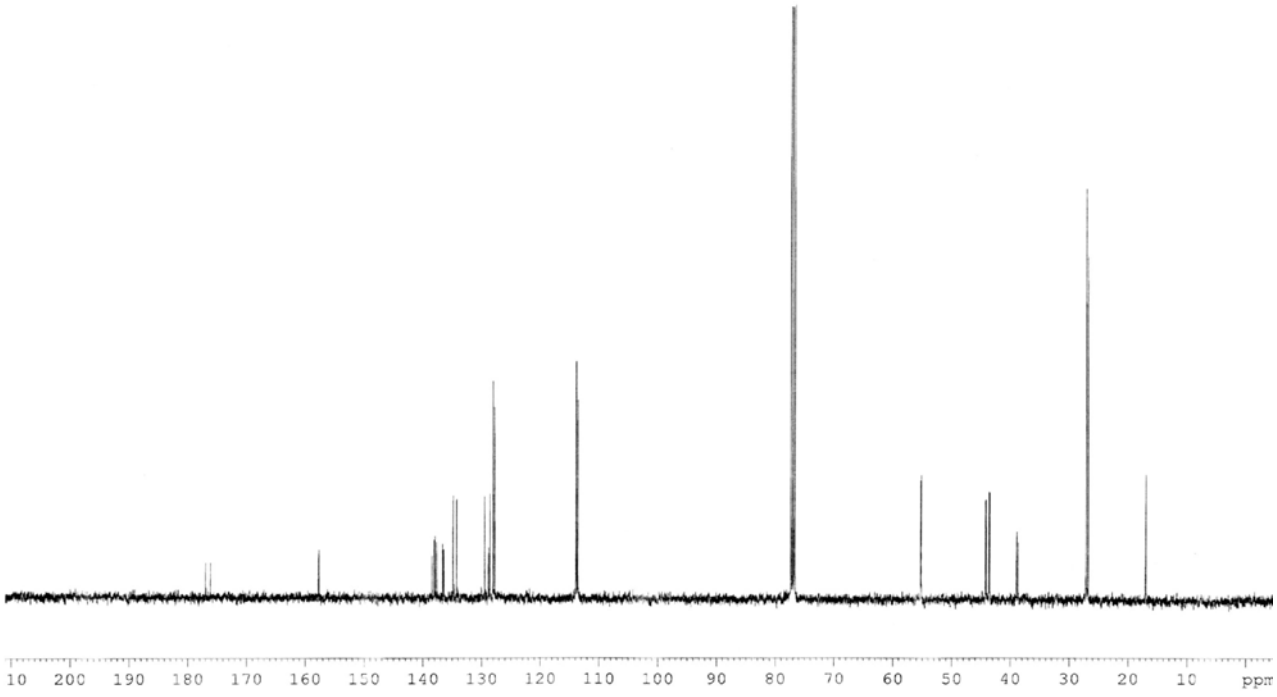

AcO
Me
OMe

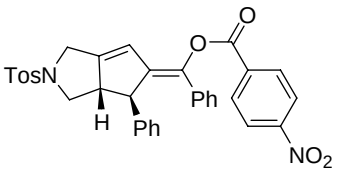

VQ-400 pa0808.053
6.324
6.318
6.313
6.309
6.303
6.299
6.294
6.289
6.061
6.054
6.047
6.040
6.034
5.945
5.939
5.932
5.925
5.919
2.17
2.00
2.00
7.161
7.153
7.148
7.137
7.131
7.124
7.082
7.074
7.069
7.058
7.053
7.045
6.857
6.851
6.844
6.839
6.835
6.827
6.822
6.814
6.809
6.798
6.793
6.785
1.751
1.644
ppm

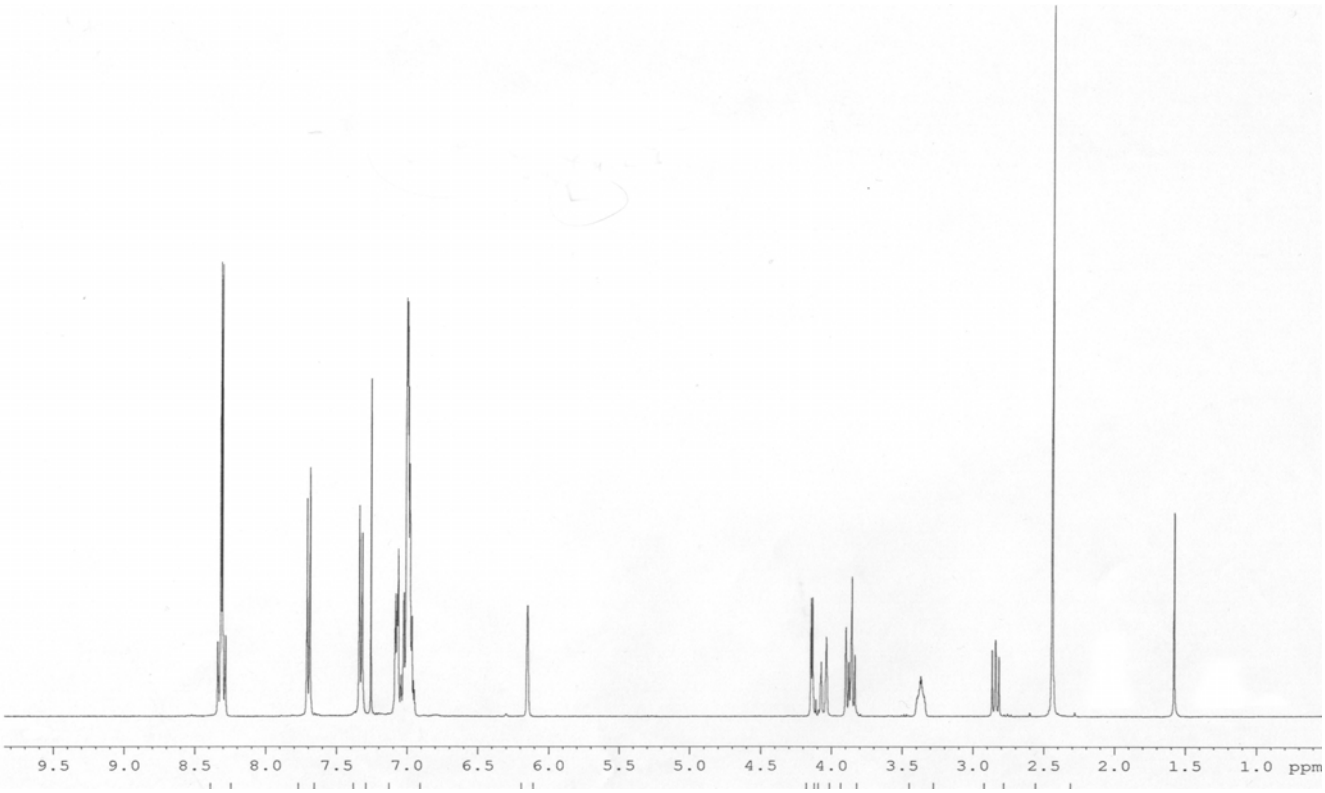

ppm

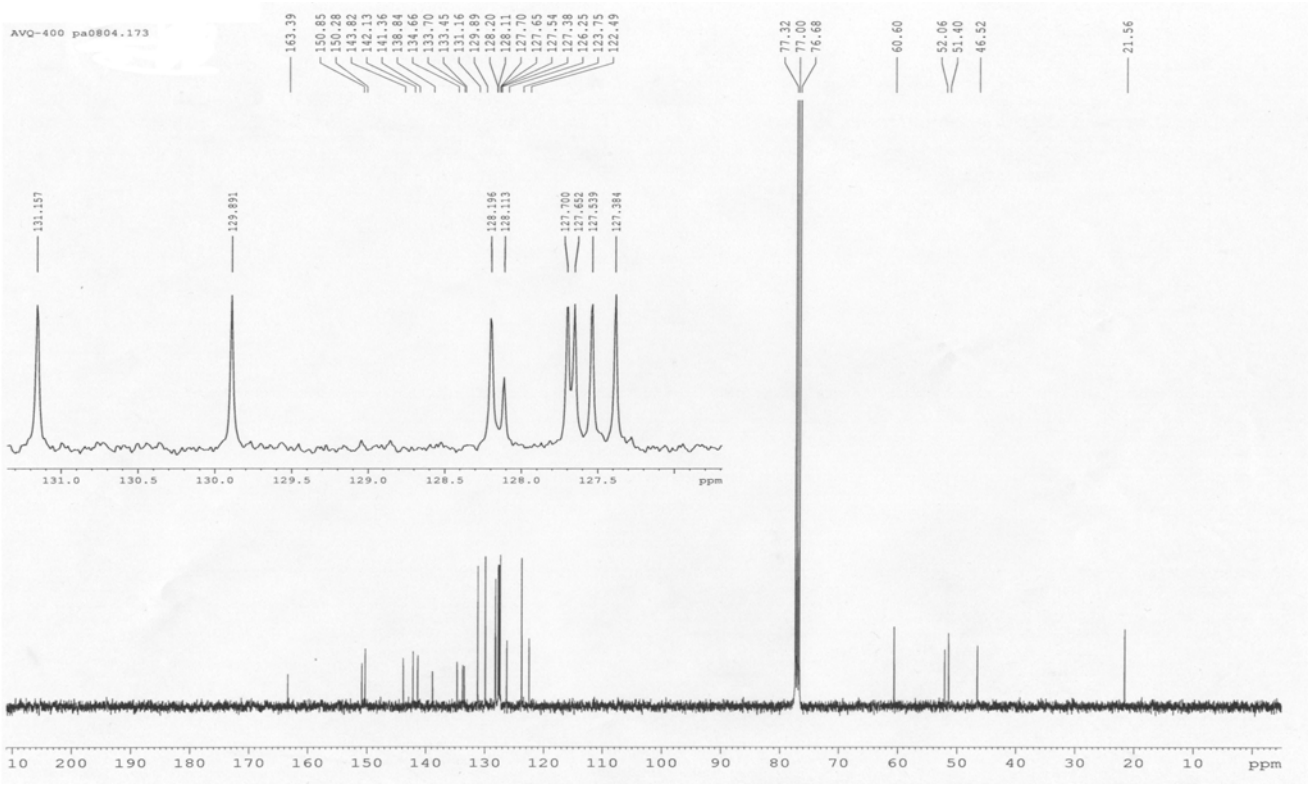

PivO
Me
OMe

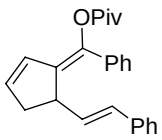

7.158
7.153
7.146
7.054
7.038
7.033
6.957
6.859
6.853
6.846
6.840
6.829
6.824
6.800
6.793
6.787
6.339
6.334
6.328
6.325
6.319
6.314
6.273
6.268
6.259
6.253
6.248
5.966
5.959
5.952
5.909
5.902
5.895
3.989
3.965
3.938
3.777
3.761
ppm

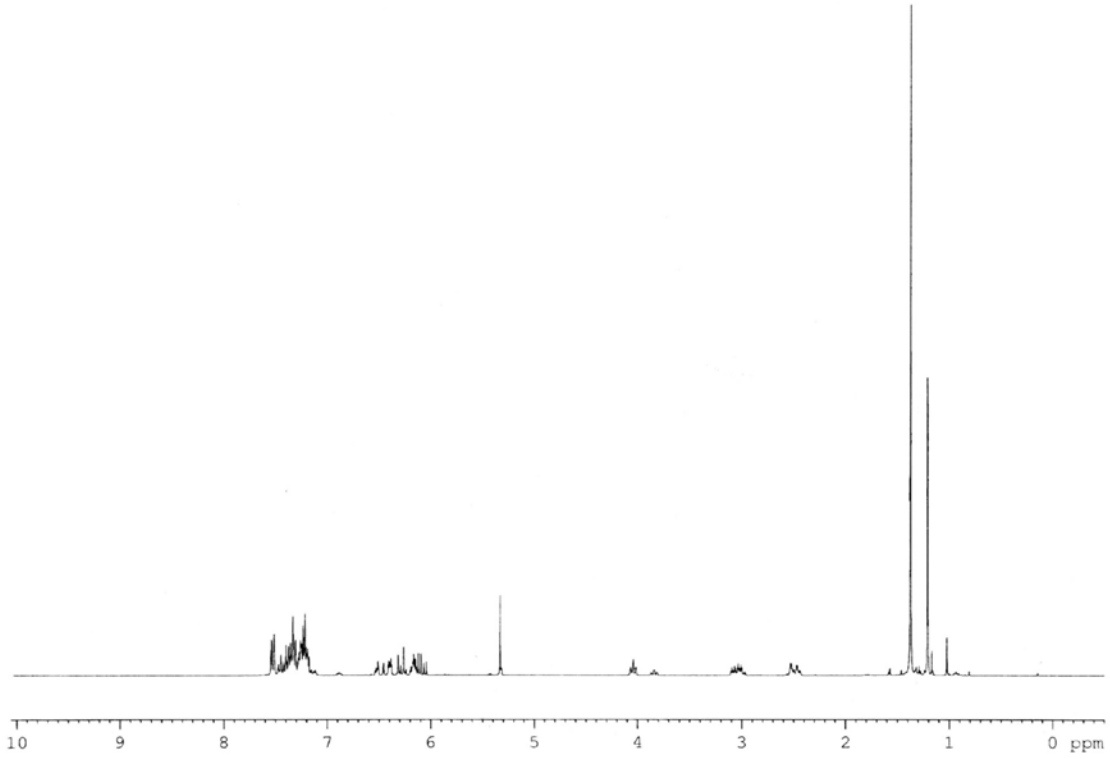

ppm

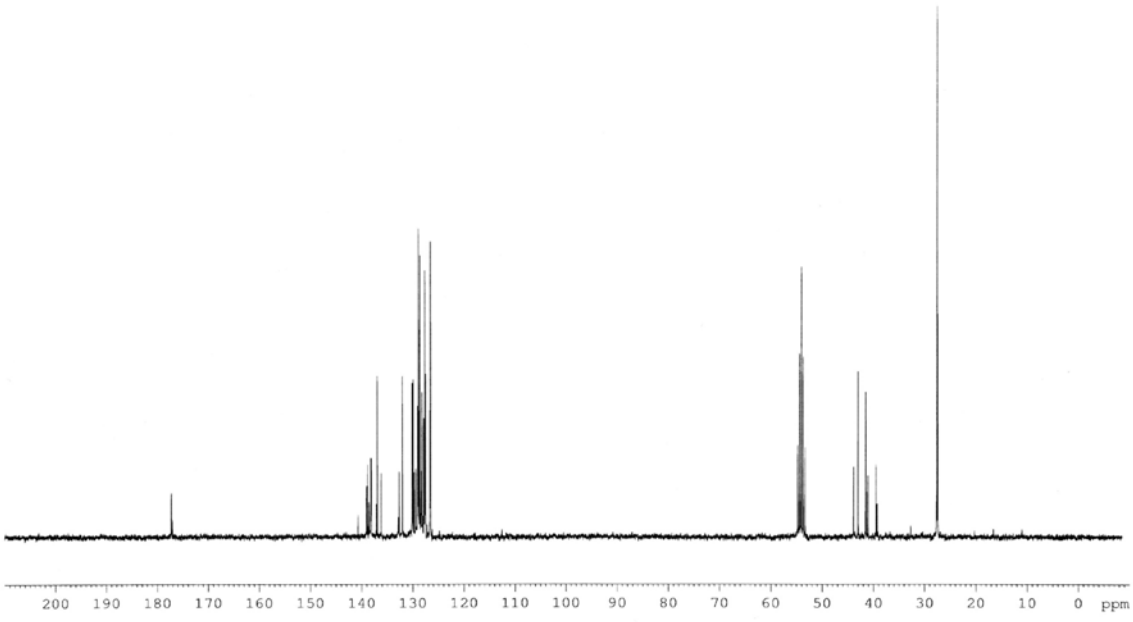

TosN
H
Ph
Ph
O
O
NO2

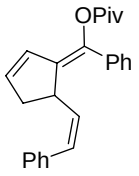

9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm

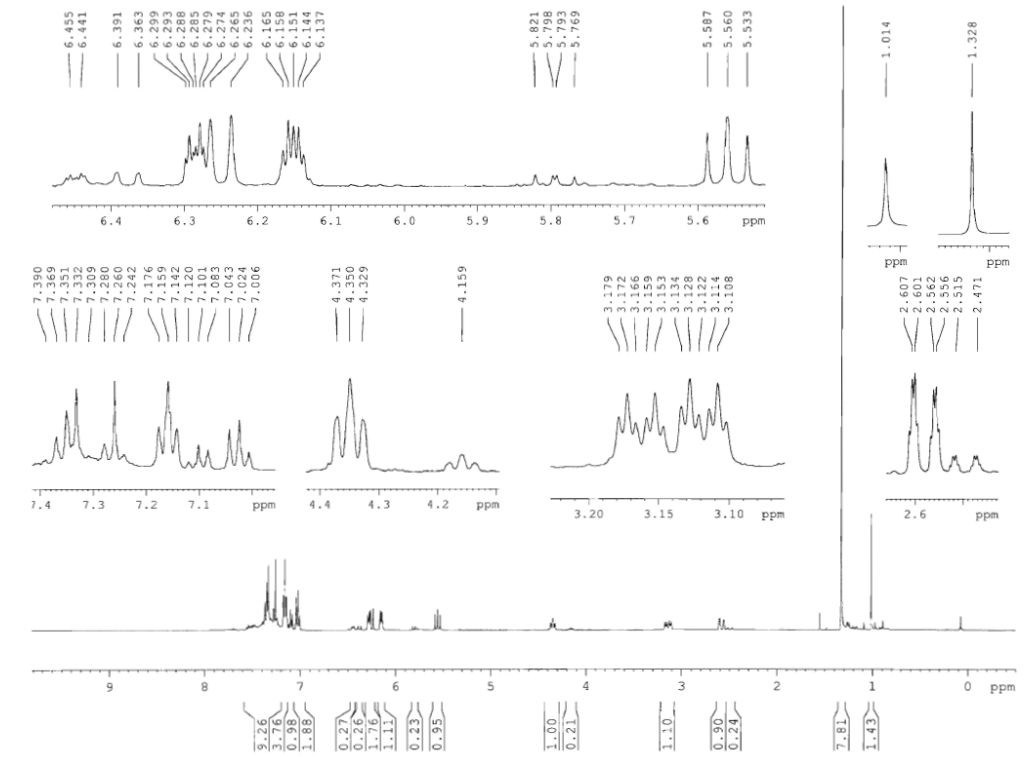

AVQ-400 pa0804.173
163.39
150.85
150.28
143.82
142.13
141.36
138.84
134.66
133.70
133.45
131.16
129.89
128.20
128.11
127.70
127.65
127.54
127.38
126.25
123.75
122.49
77.32
77.00
76.68
60.60
52.06
51.40
46.52
21.56
131.157
129.891
128.196
128.113
127.700
127.602
127.559
127.384
131.0 130.5 130.0 129.5 129.0 128.5 128.0 127.5 ppm
10 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

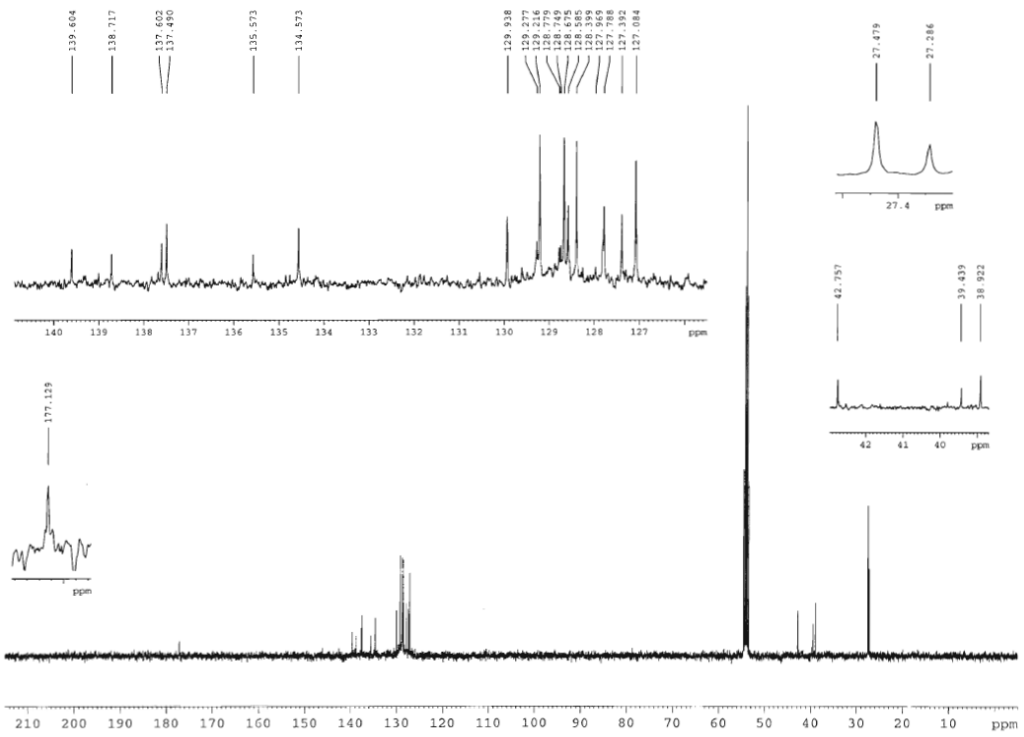

OPiv
Ph
Ph

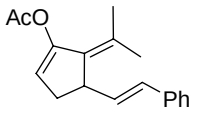

10
9
8
7
6
5
4
3
2
1
0 ppm

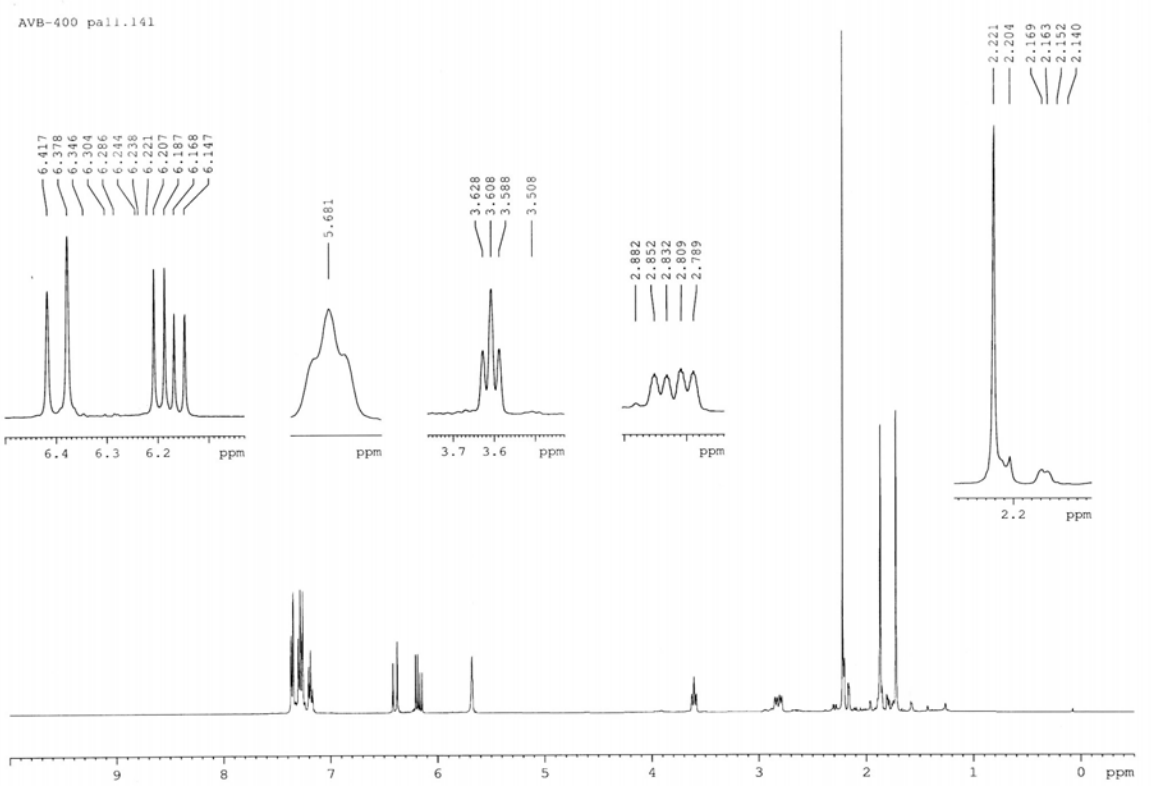

200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm

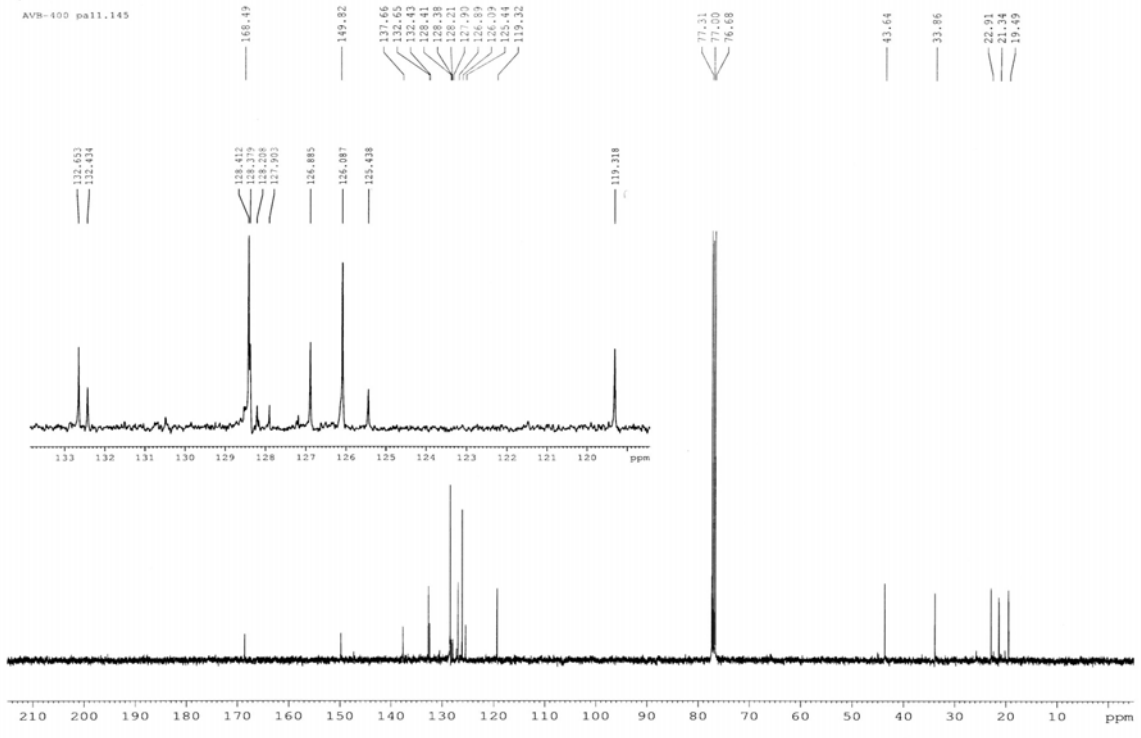

OPiv
Ph
Ph



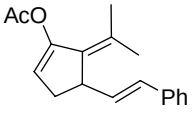


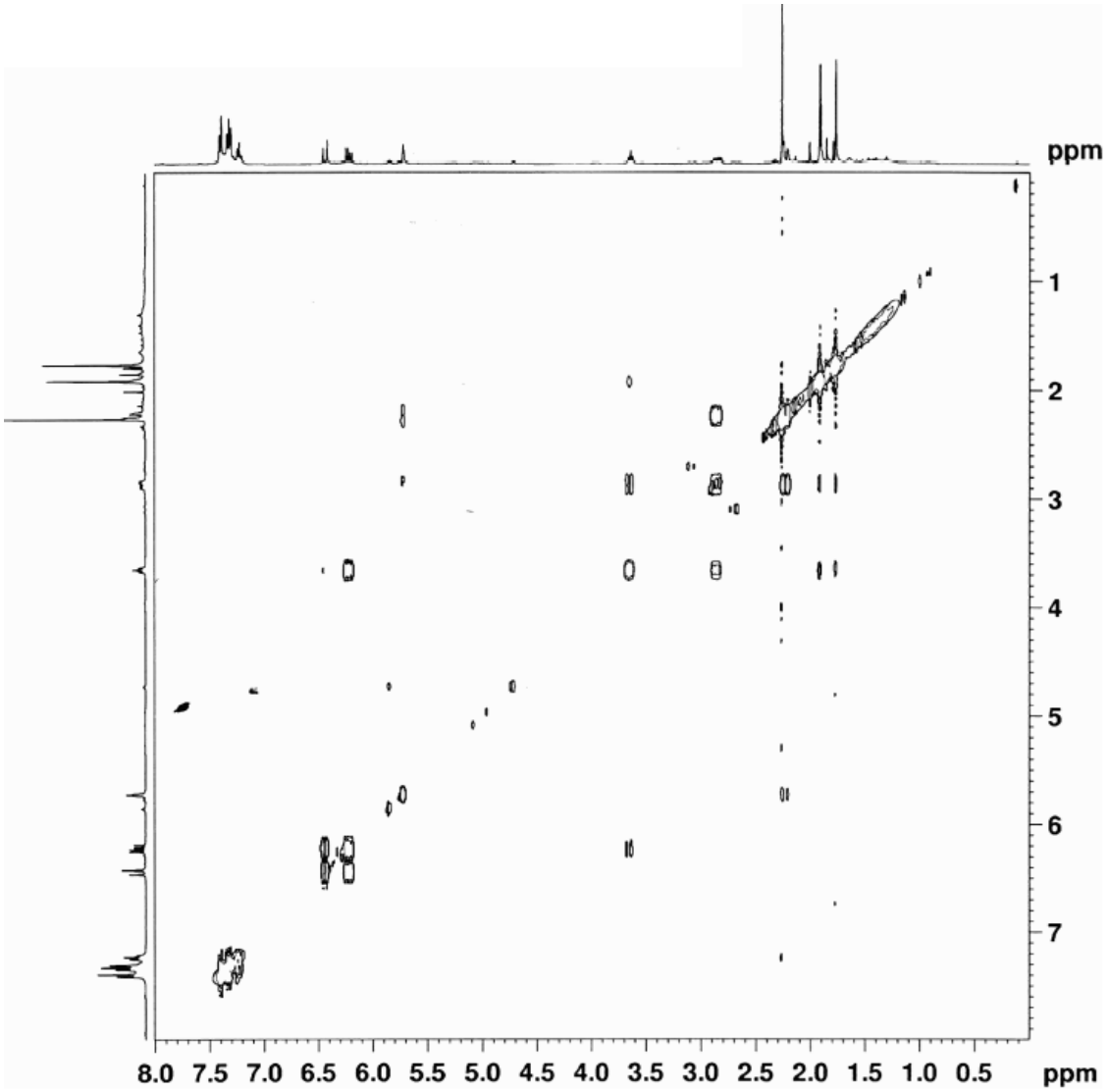

AcO
Ph


AVB-400 pa11.141
6.417
6.378
6.346
6.304
6.286
6.244
6.238
6.221
6.207
6.187
6.168
6.147
5.681
3.628
3.608
3.588
3.508
2.882
2.852
2.832
2.809
2.789
2.221
2.204
2.169
2.163
2.152
2.140
ppm

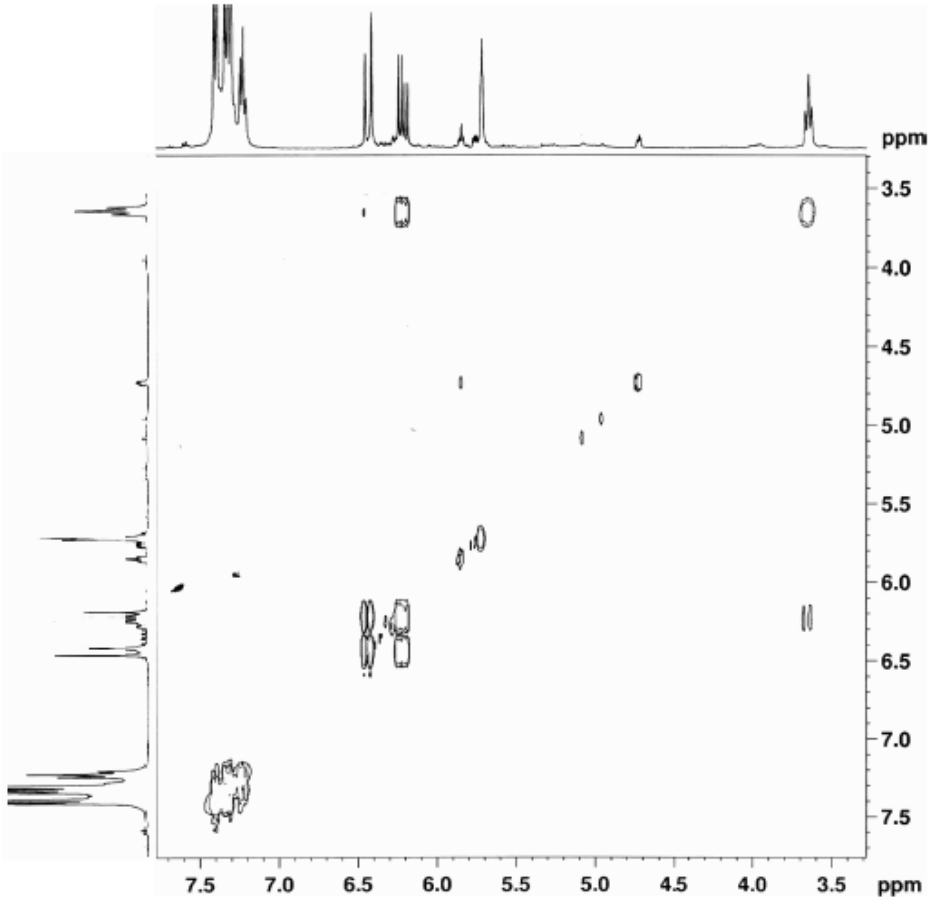

AVB-400 pa11.145
168.49
149.82
137.66
132.65
132.43
128.41
128.38
128.21
127.90
126.89
126.09
125.44
119.32
77.31
77.00
76.68
43.64
33.86
22.91
21.34
19.49
ppm

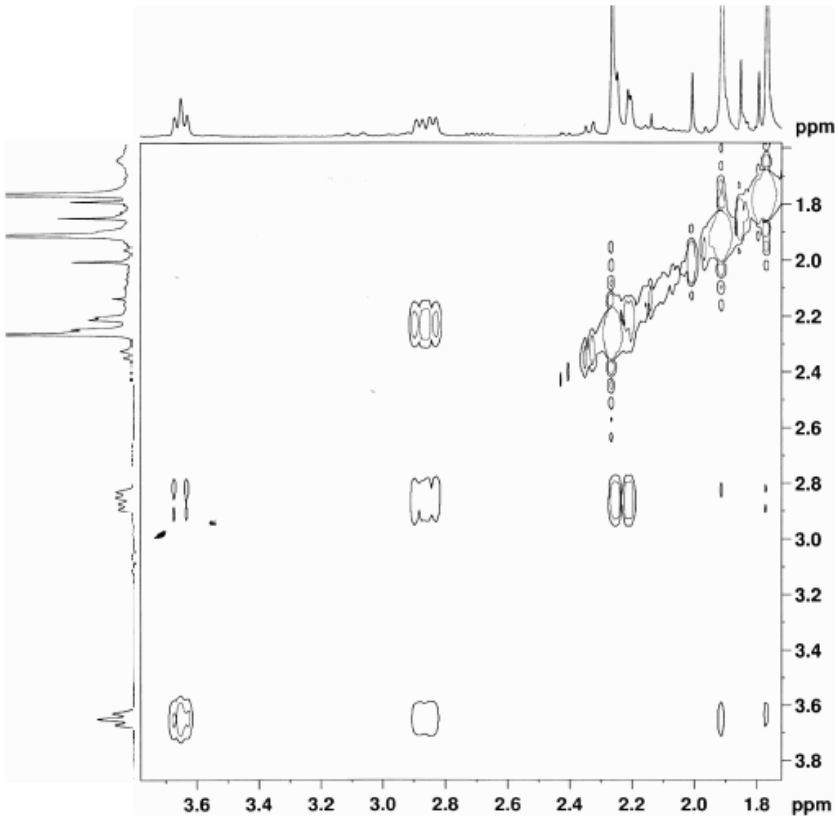
2D studies on cyclopentene 36: COSY



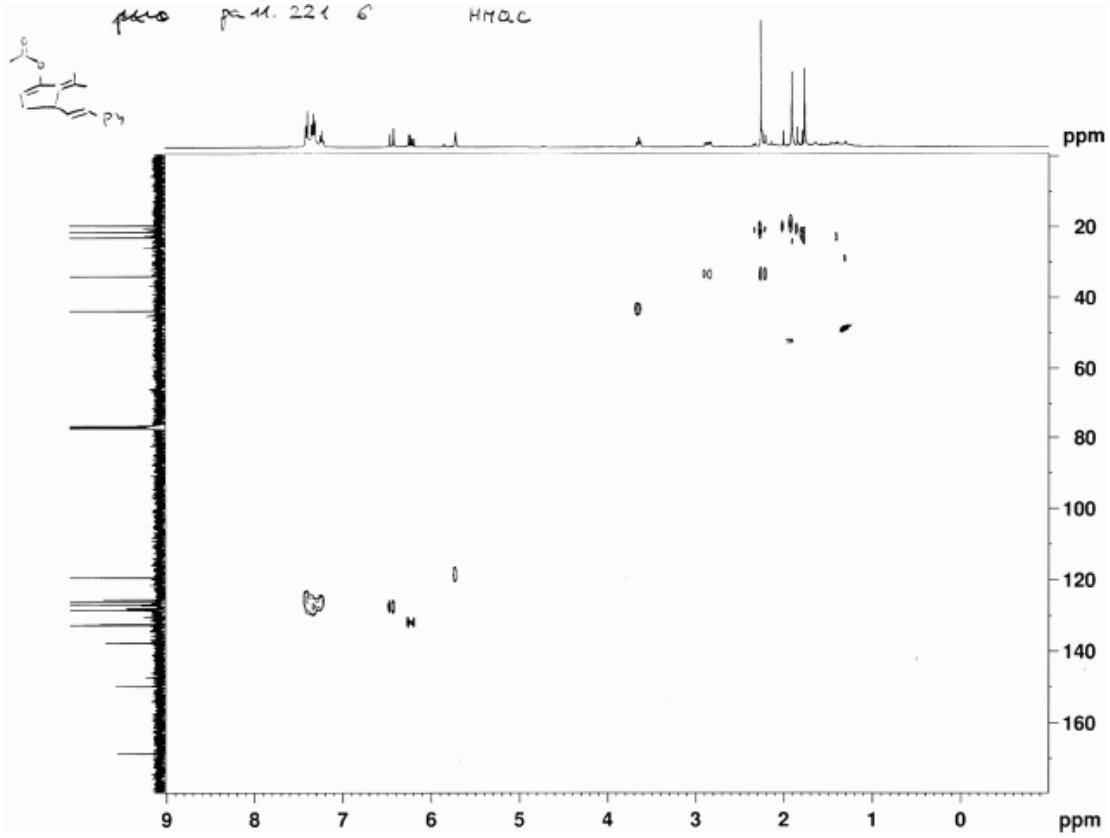


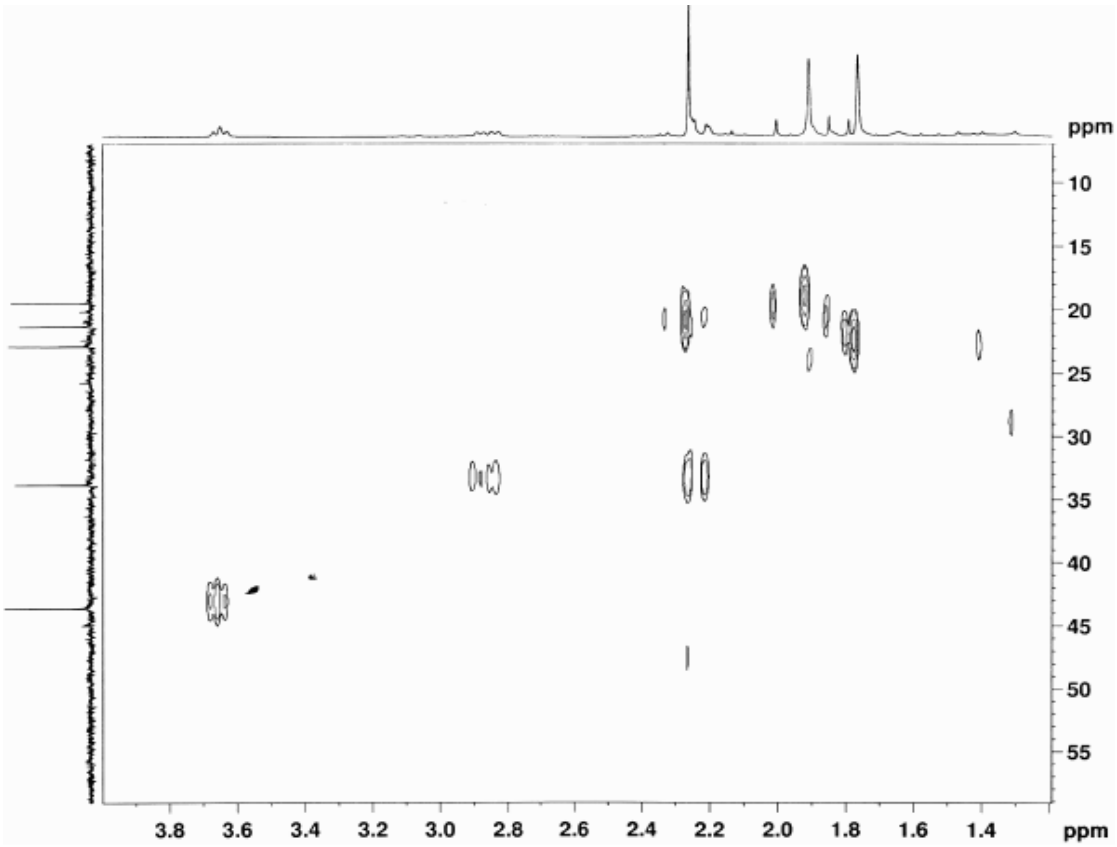
2D studies on cyclopentene 36: COSY

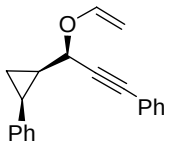


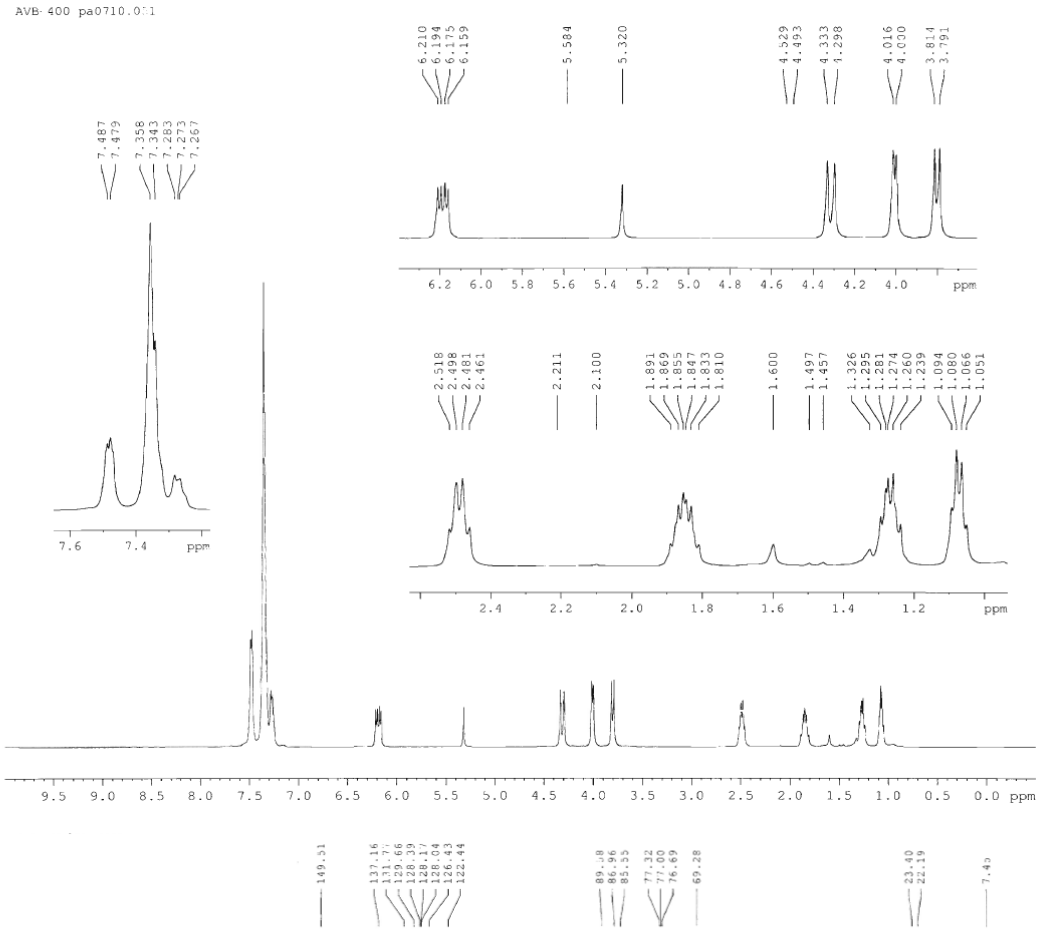


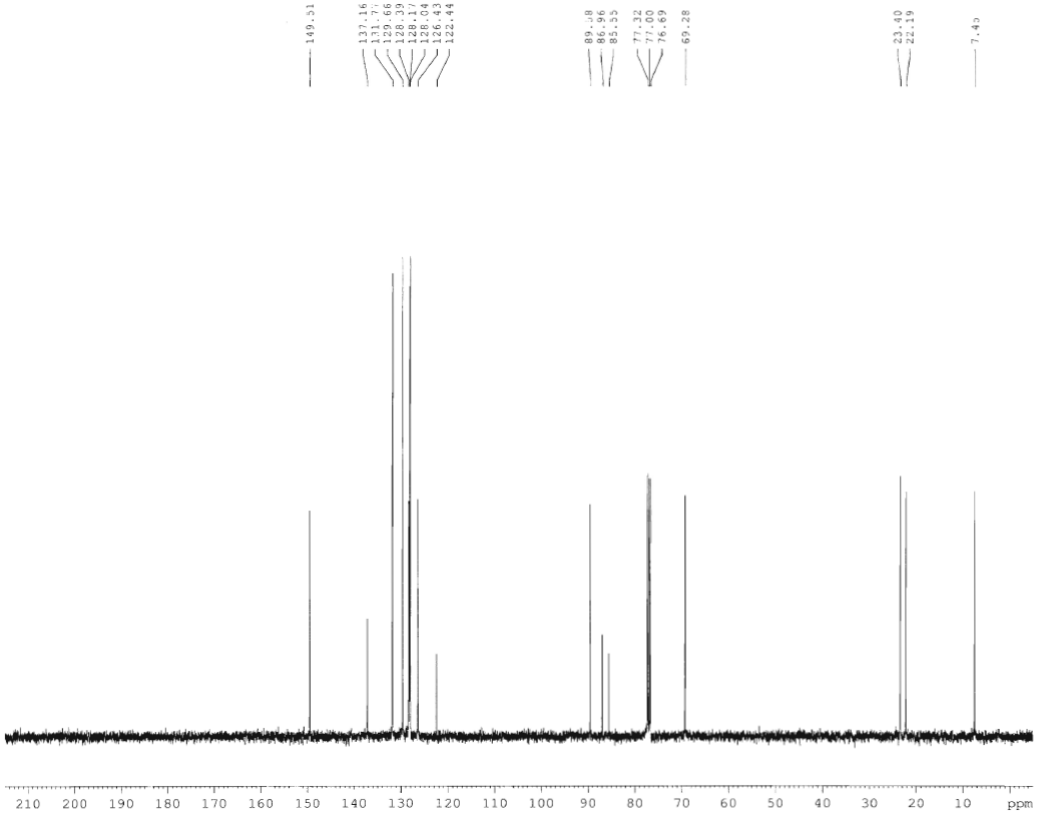
2D studies on cyclopentene 36: HMQC

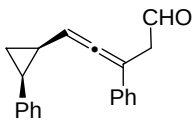


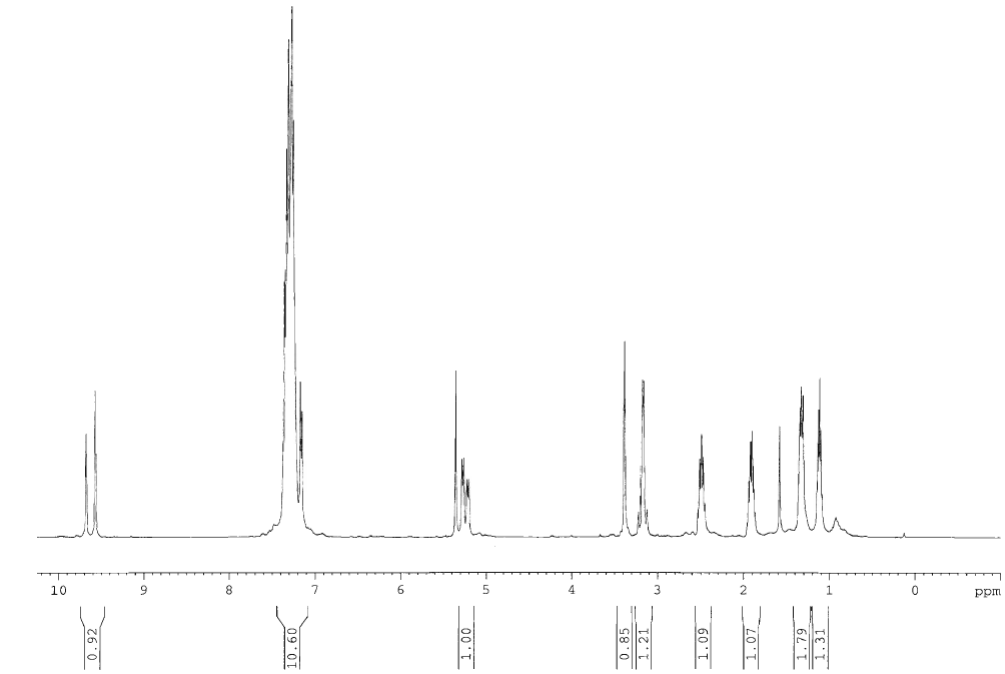


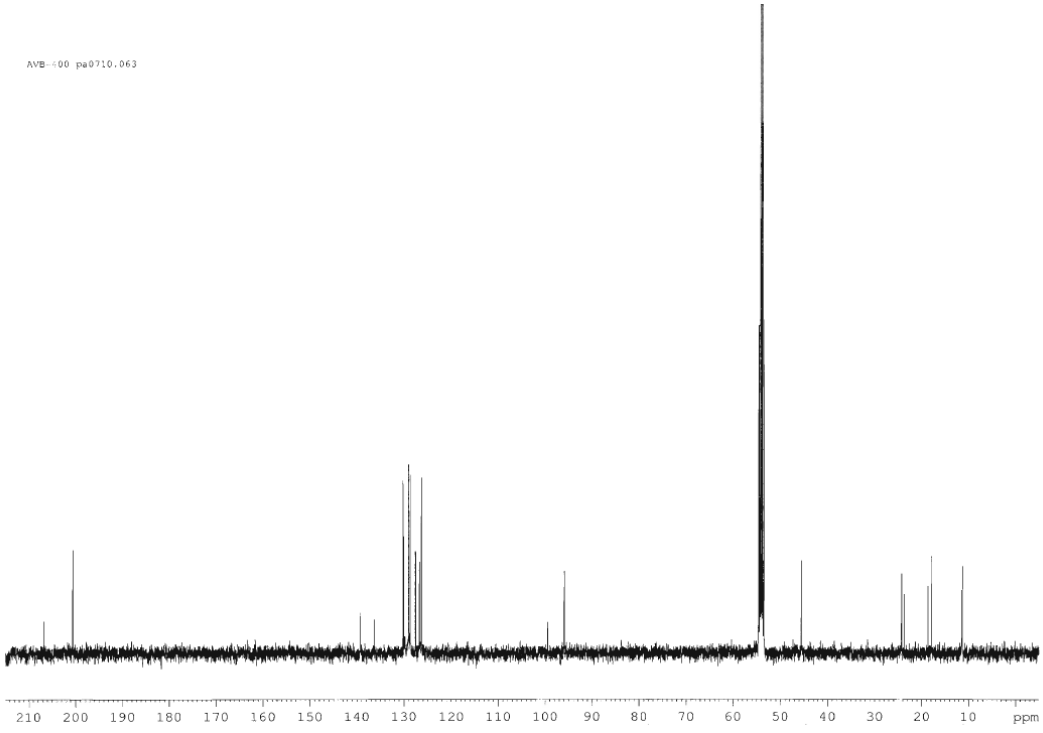

O
Ph
Ph

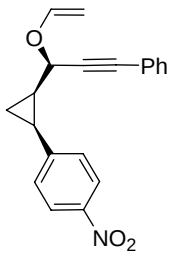

AVB-400 pa0710.011
6.210
6.194
6.175
6.159
5.584
5.320
4.529
4.493
4.333
4.298
4.016
4.000
3.814
3.791
7.487
7.479
7.358
7.343
7.283
7.273
7.267
2.518
2.498
2.481
2.461
2.211
2.100
1.891
1.869
1.855
1.847
1.833
1.810
1.600
1.497
1.457
1.326
1.295
1.281
1.274
1.260
1.239
1.094
1.080
1.066
1.051
ppm

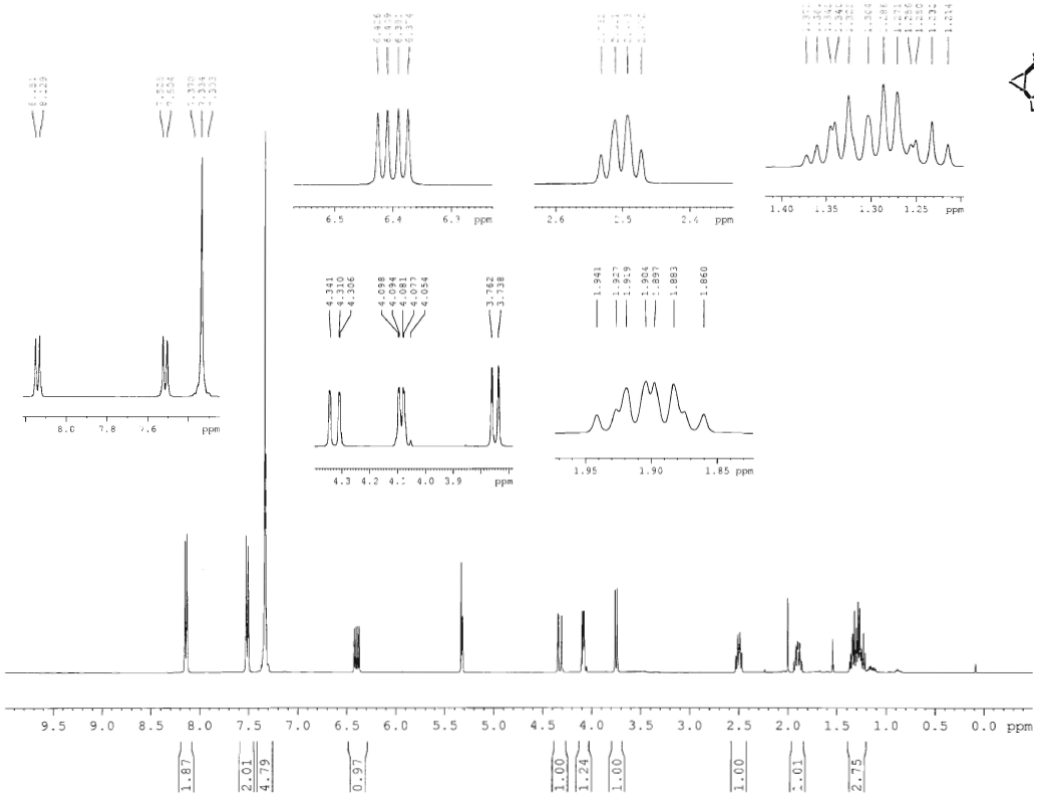

149.31
137.16
131.77
129.66
128.39
128.17
128.04
126.43
122.44
89.48
86.96
85.55
77.32
77.00
76.69
69.28
23.40
22.19
7.43
ppm

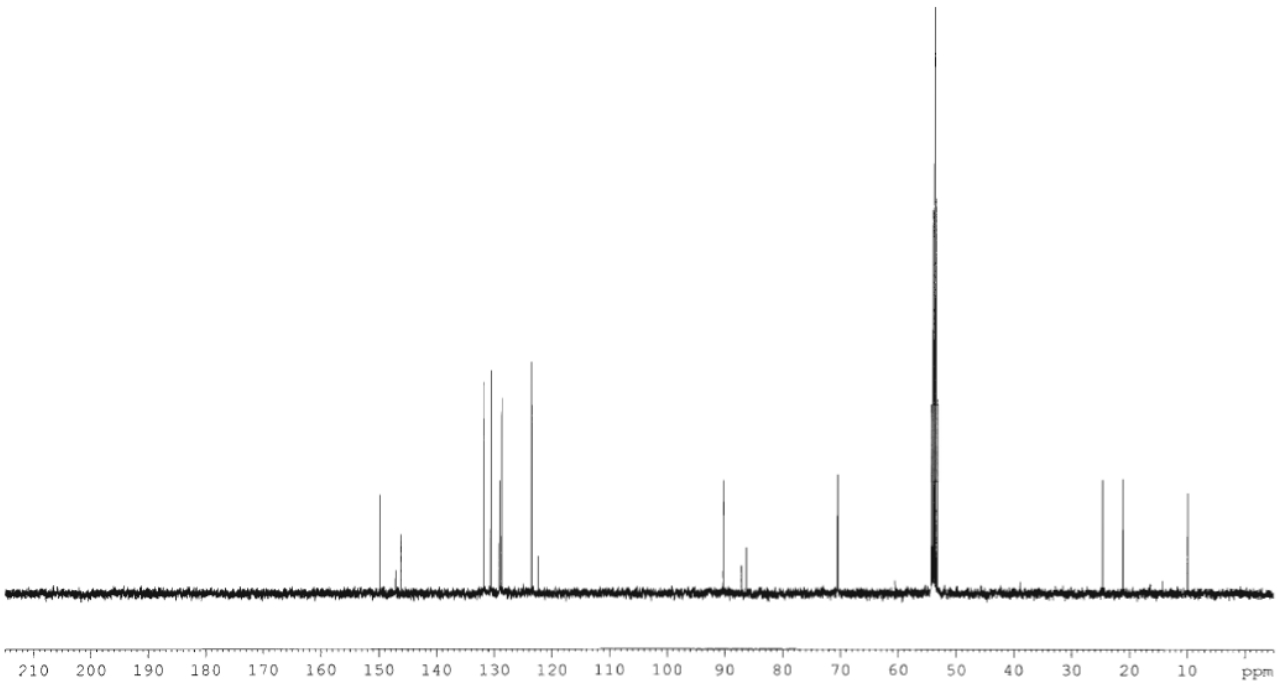

CHO
Ph
Ph

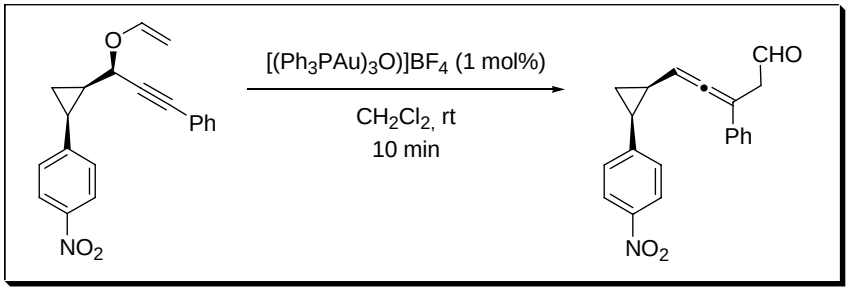

10
9
8
7
6
5
4
3
2
1
0
ppm
0.92
10.60
1.00
0.85
1.21
1.09
1.07
1.79
1.31

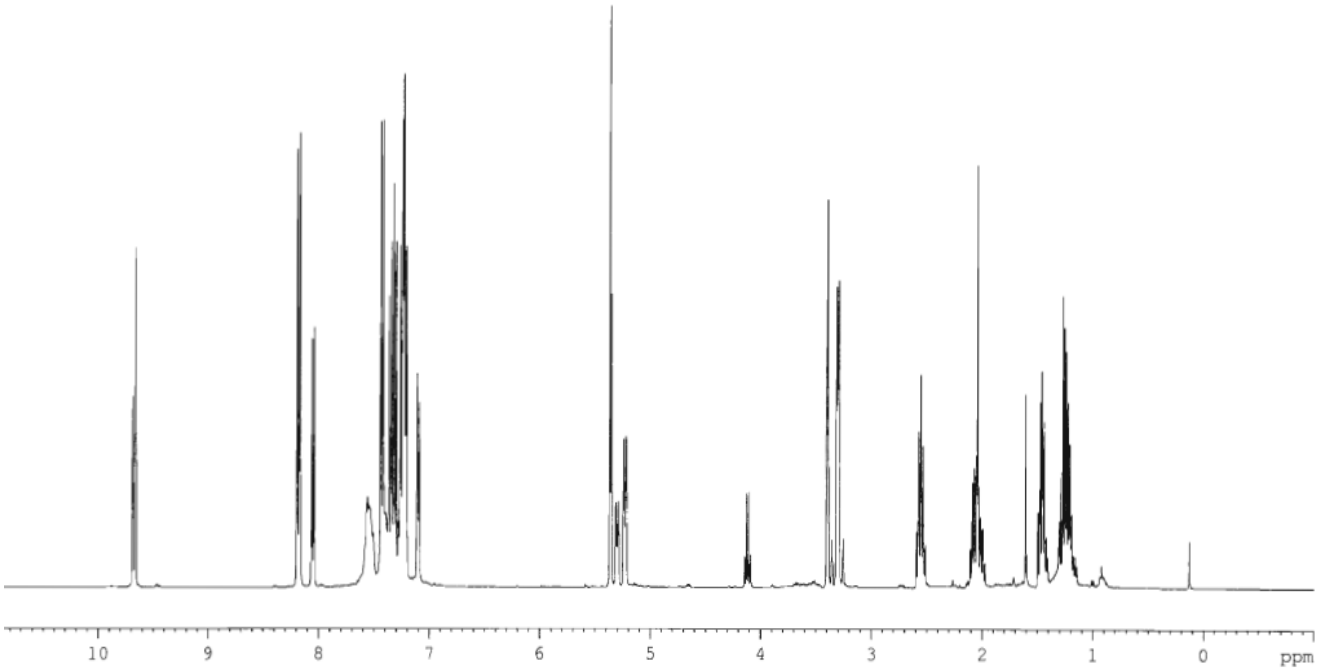

AVB-400 pa0710.063
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

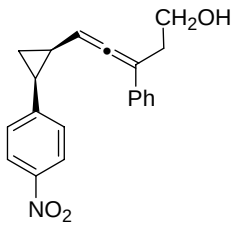

O
Ph
NO2

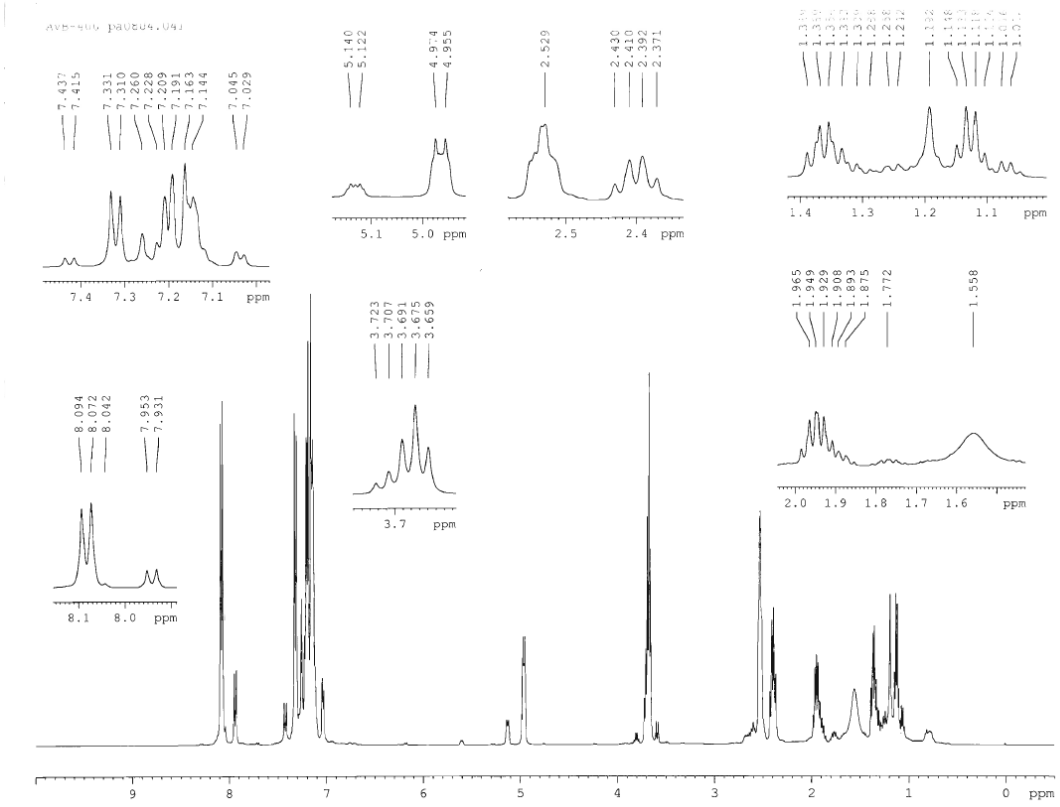

9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.87
2.01
4.79
0.97
1.00
1.24
1.00
1.00
3.01
2.75

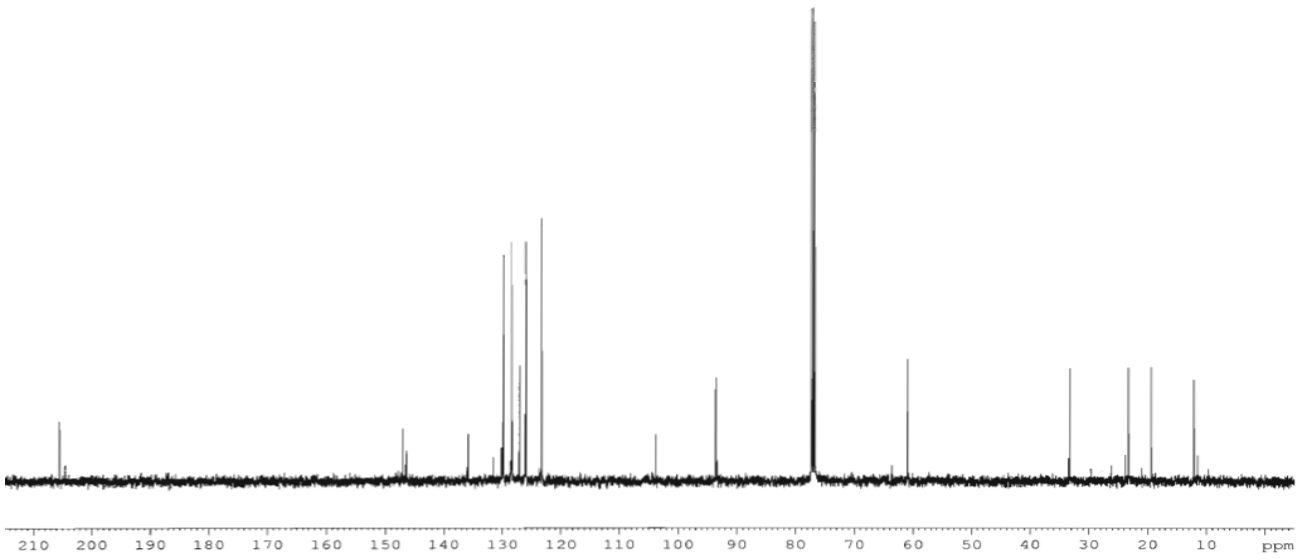

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

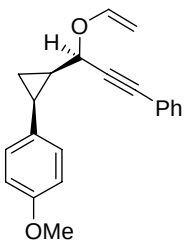

O
Ph
NO2
[(Ph3PAu)3O)]BF4 (1 mol%)
CH2Cl2, rt
10 min
CHO
Ph
NO2

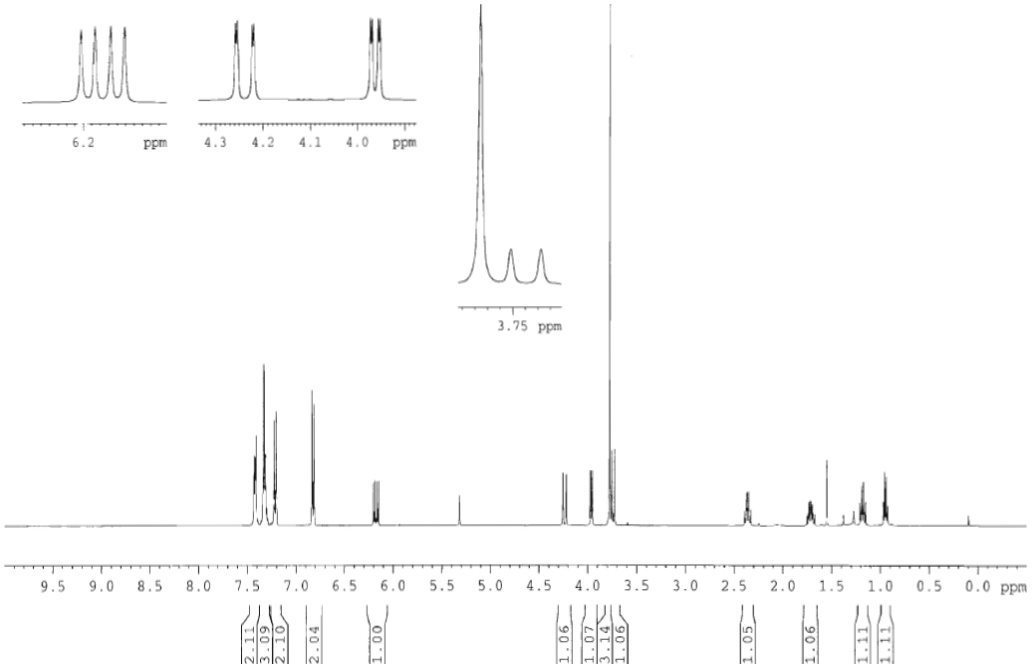

10
9
8
7
6
5
4
3
2
1
0
ppm

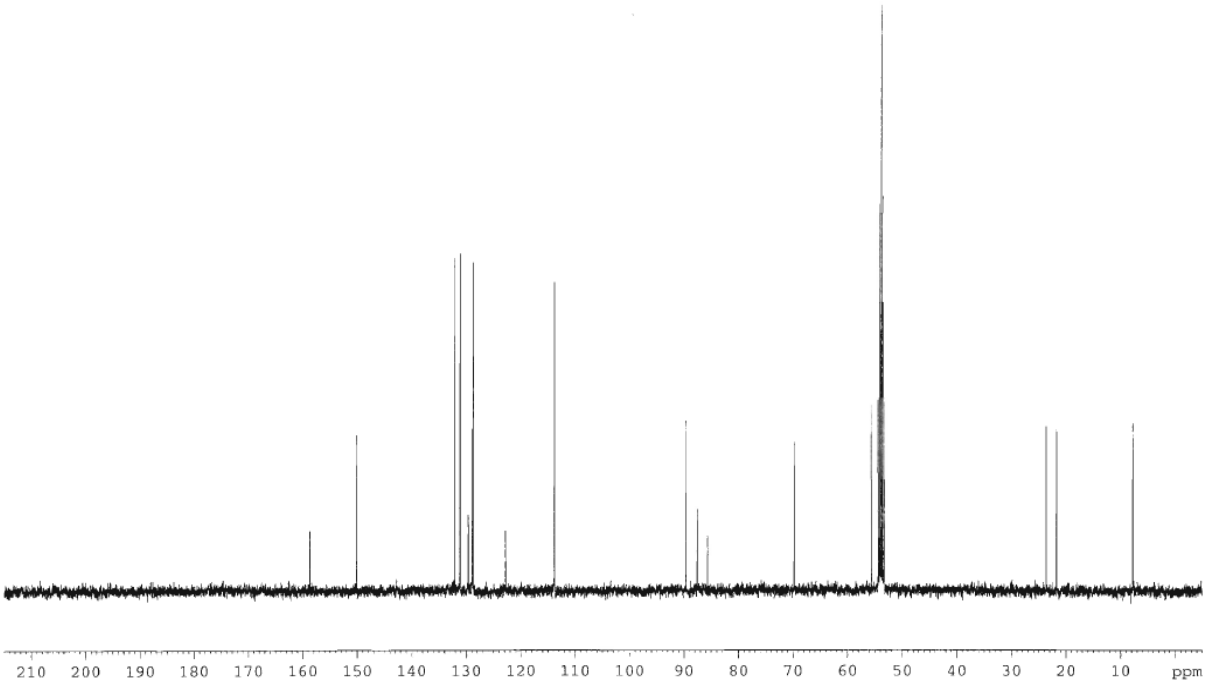

CH2OH
Ph
NO2

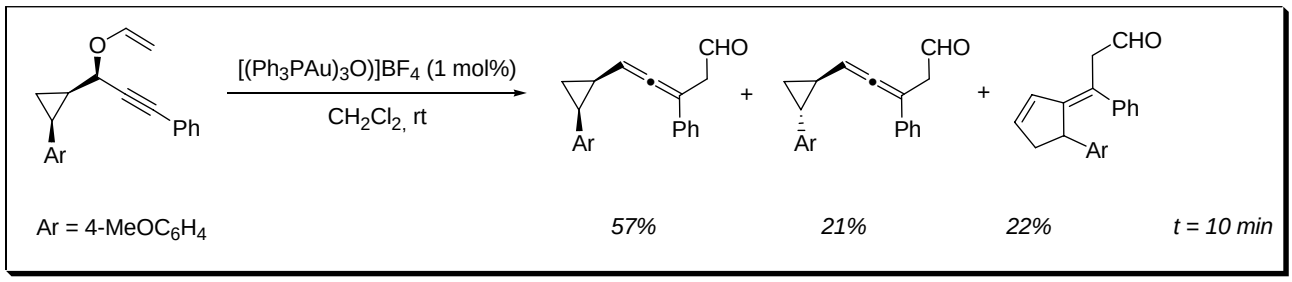

7.437
7.415
7.331
7.310
7.260
7.228
7.209
7.191
7.163
7.144
7.045
7.029
5.140
5.122
4.974
4.955
2.529
2.430
2.410
2.392
2.371
1.965
1.949
1.929
1.908
1.893
1.875
1.772
1.558
3.723
3.707
3.691
3.675
3.659
8.094
8.072
8.042
7.953
7.931
ppm

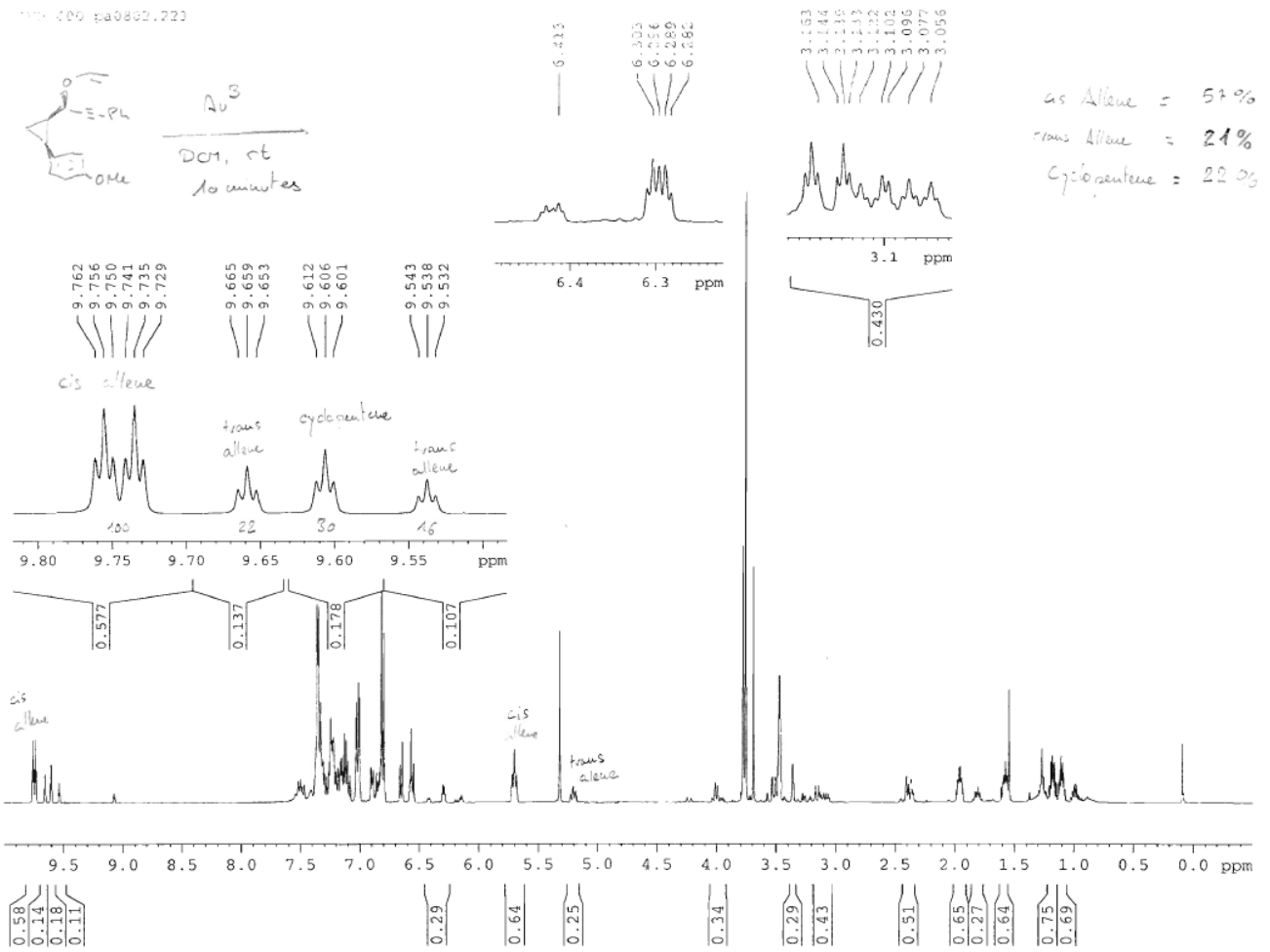

ppm

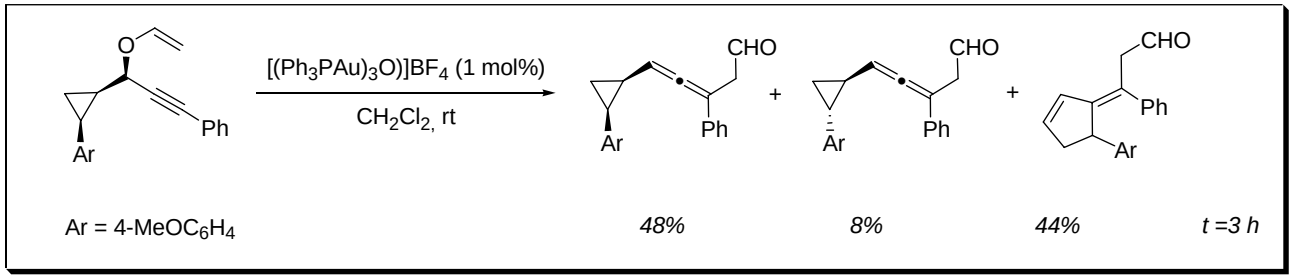

H
O
Ph
OMe


6.2 ppm
4.3 4.2 4.1 4.0 ppm
3.75 ppm
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.11
3.09
2.10
2.04
1.00
1.06
1.07
3.14
1.06
1.05
1.06
1.11
1.11

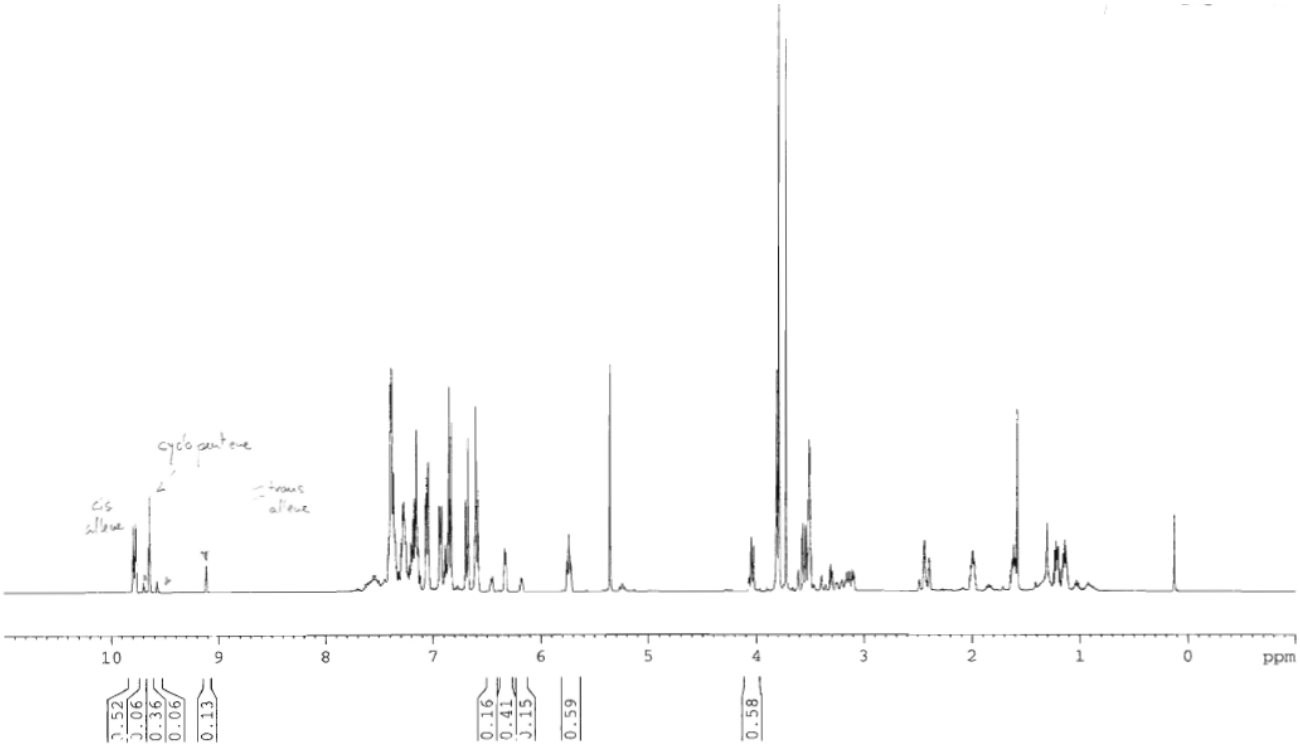

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

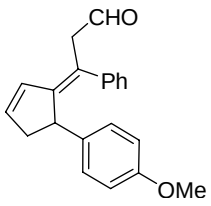

O
Ph
Ar
[(Ph3PAu)3O)]BF4 (1 mol%)
CH2Cl2, rt
CHO
Ar
Ph
+
CHO
Ar
Ph
+
CHO
Ph
Ar
Ar = 4-MeOC6H4
57%
21%
22%
t = 10 min

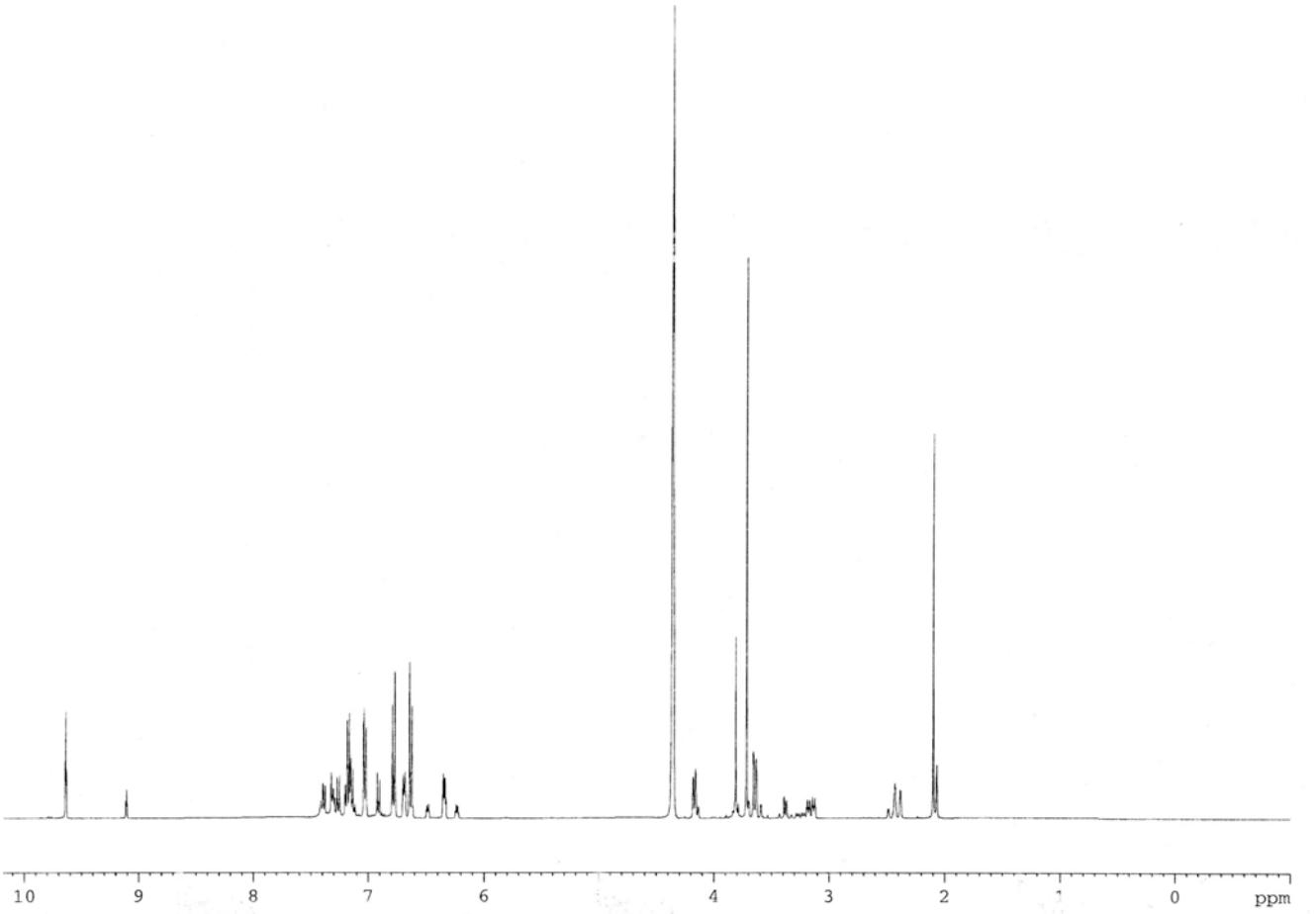

cis allene
trans allene
cyclopentene
trans allene
DCM, rt
10 minutes
cis allene = 57 %
trans allene = 21 %
Cyclopentene = 22 %
ppm

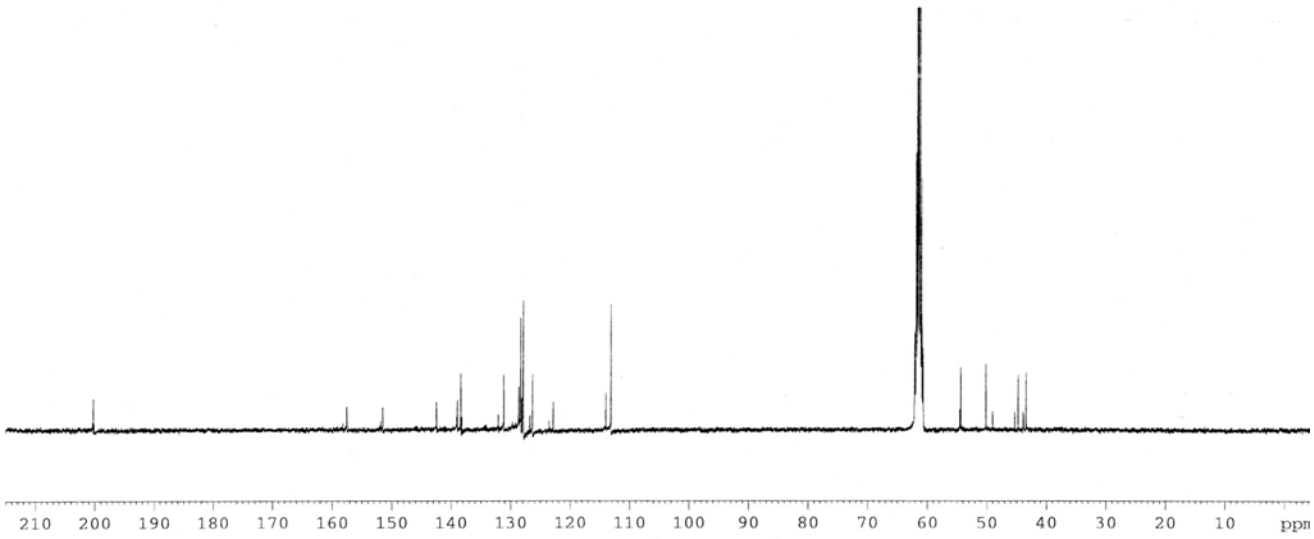


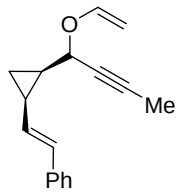
Ar = 4-$MeOC_6H_4$ *48%* *8%* *44%* *t =3 h*

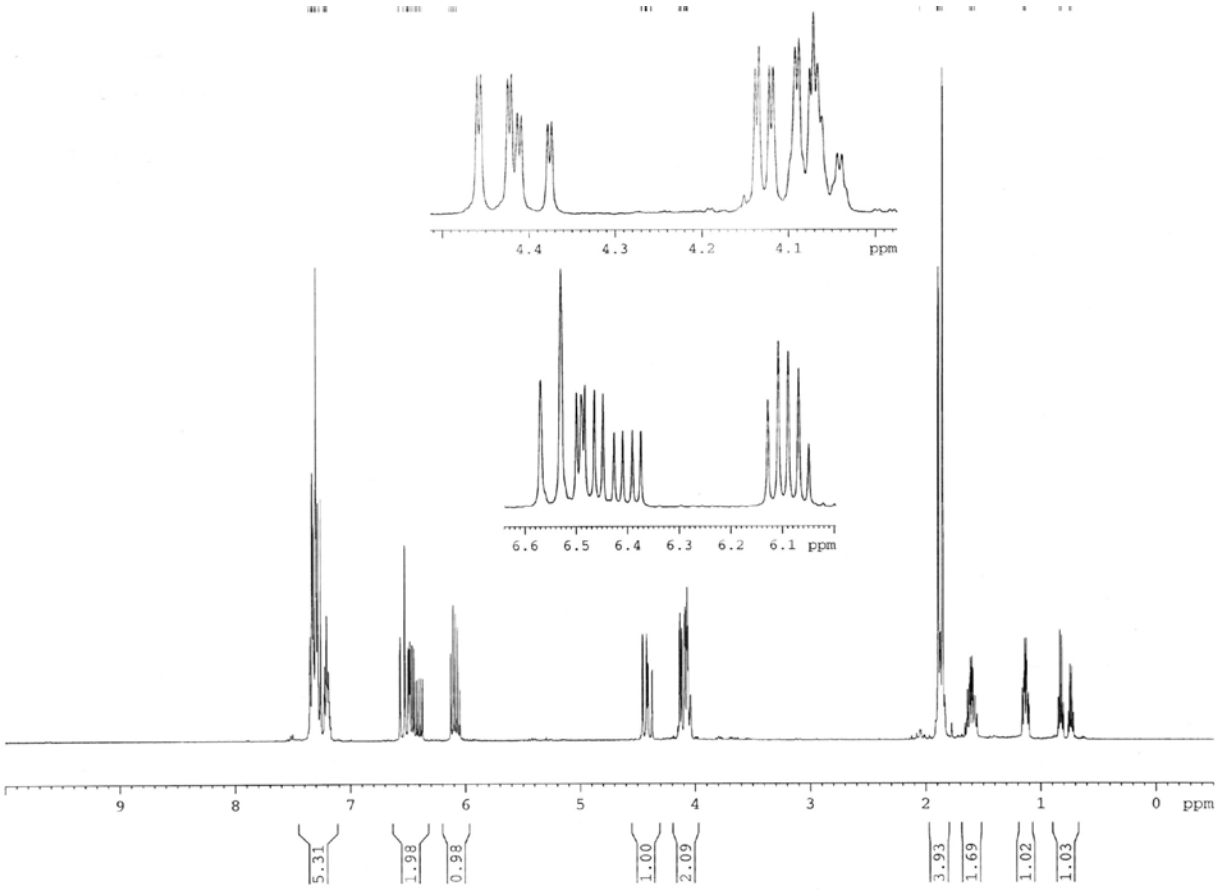


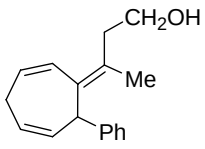

CHO
Ph
OMe

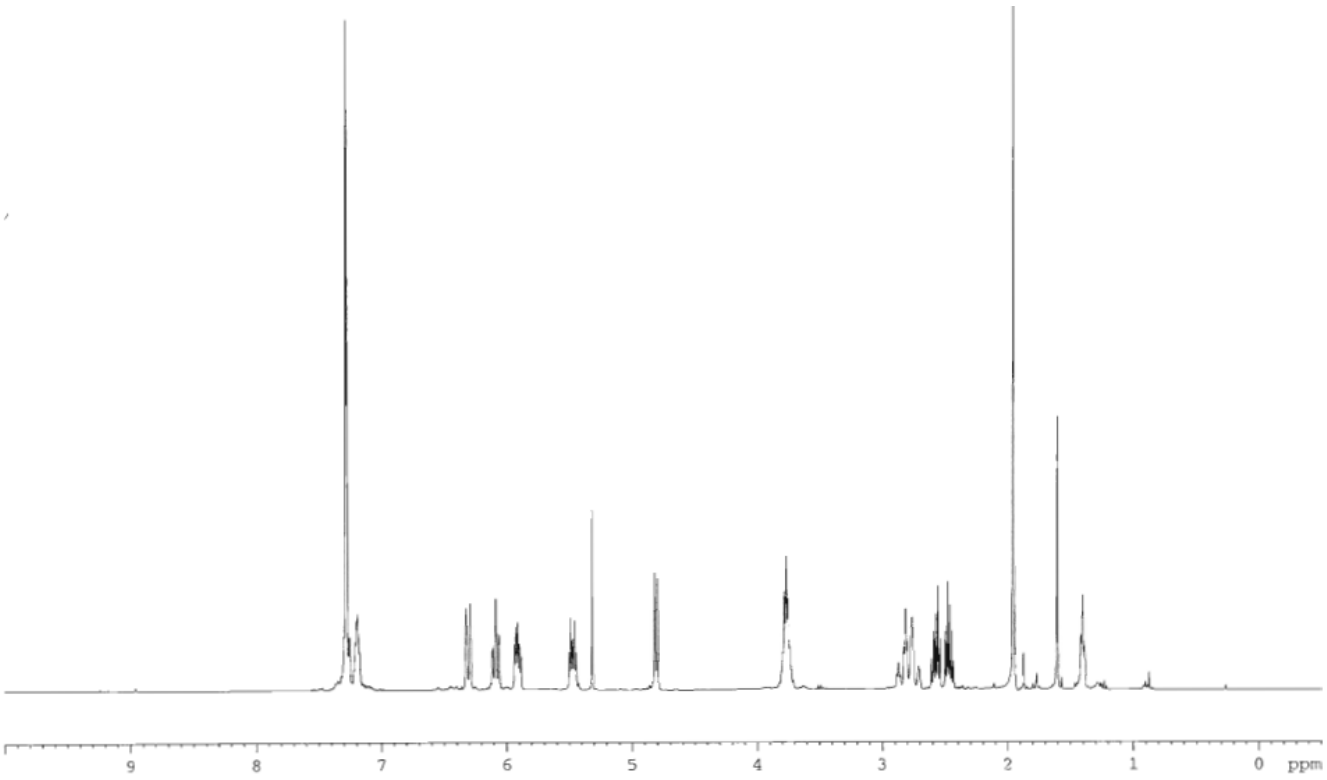

10
9
8
7
6
4
3
2
0
ppm

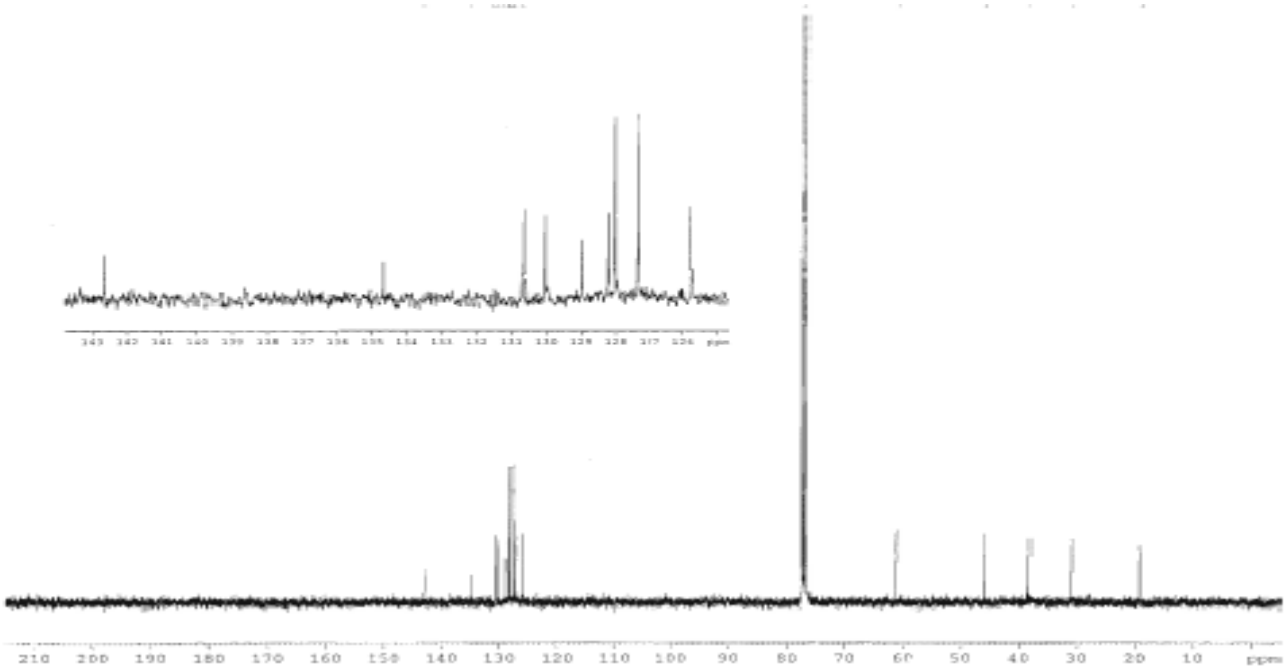

210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

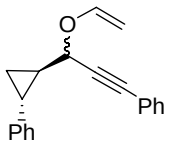

O
Me
Ph

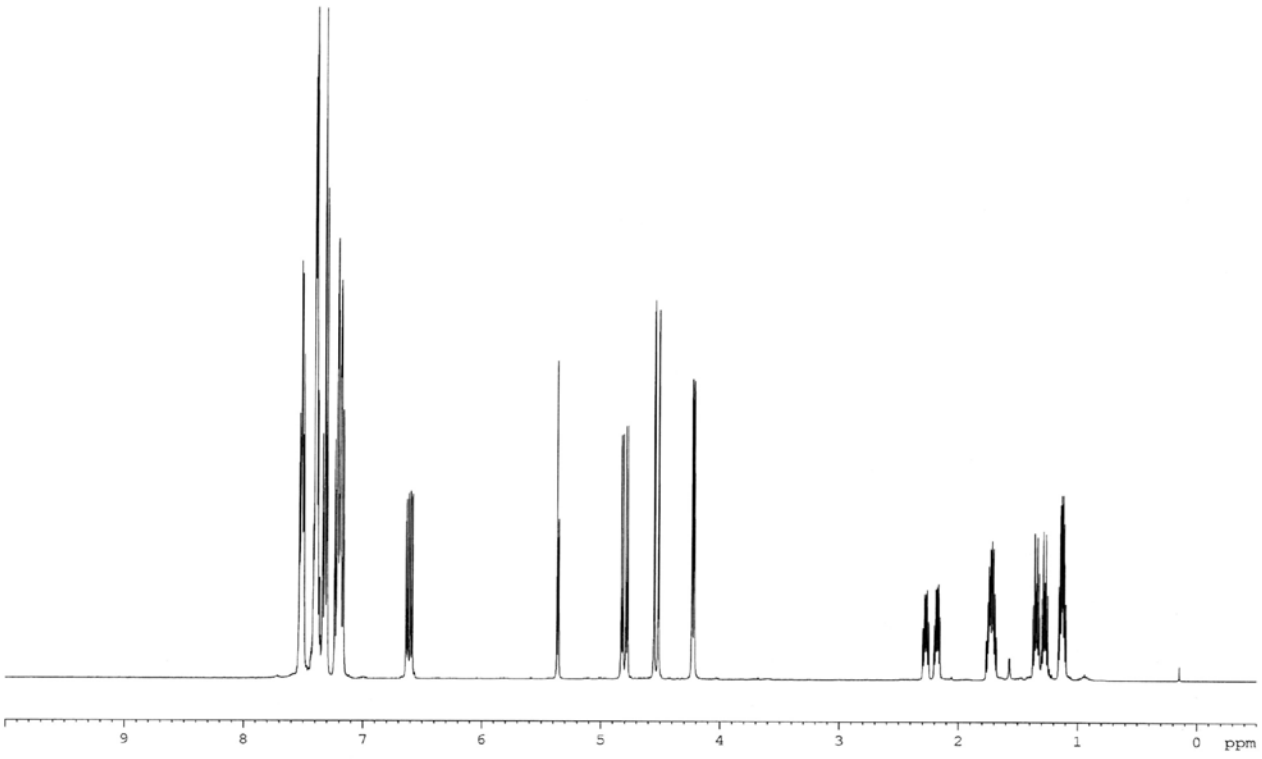

4.4
4.3
4.2
4.1
ppm
6.6
6.5
6.4
6.3
6.2
6.1
ppm
9
8
7
6
5
4
3
2
1
0
ppm
5.31
1.98
0.98
1.00
2.09
3.93
1.69
1.02
1.03

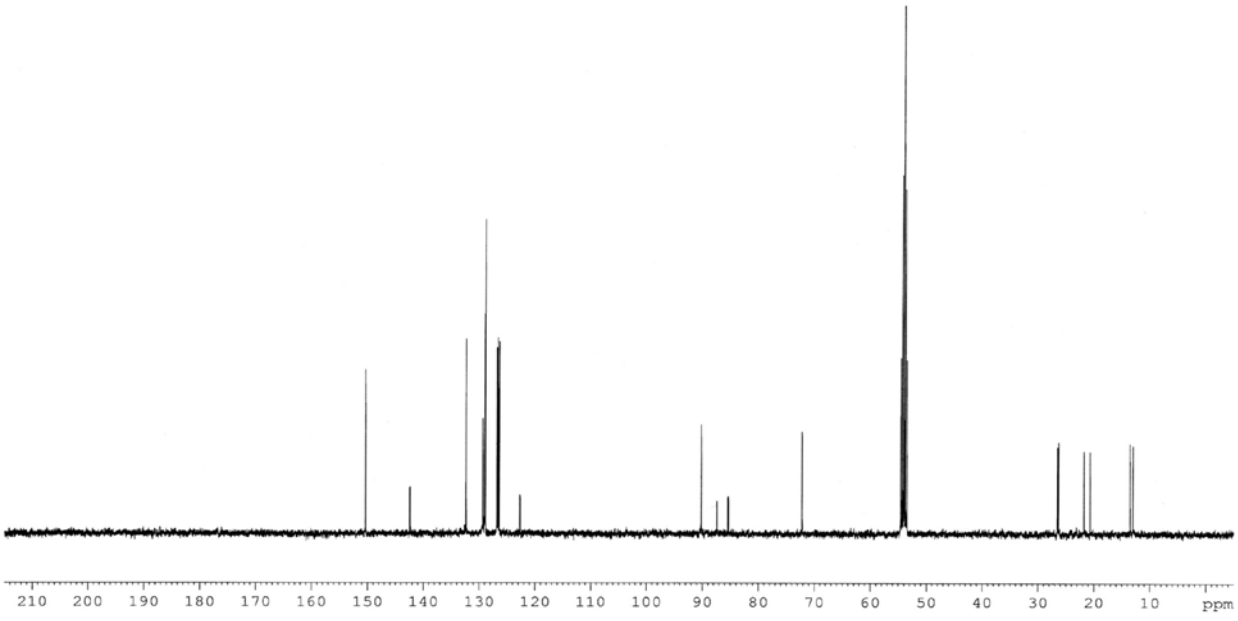

CH2OH
Me
Ph

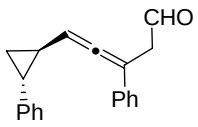

9
8
7
6
5
4
3
2
1
0
ppm

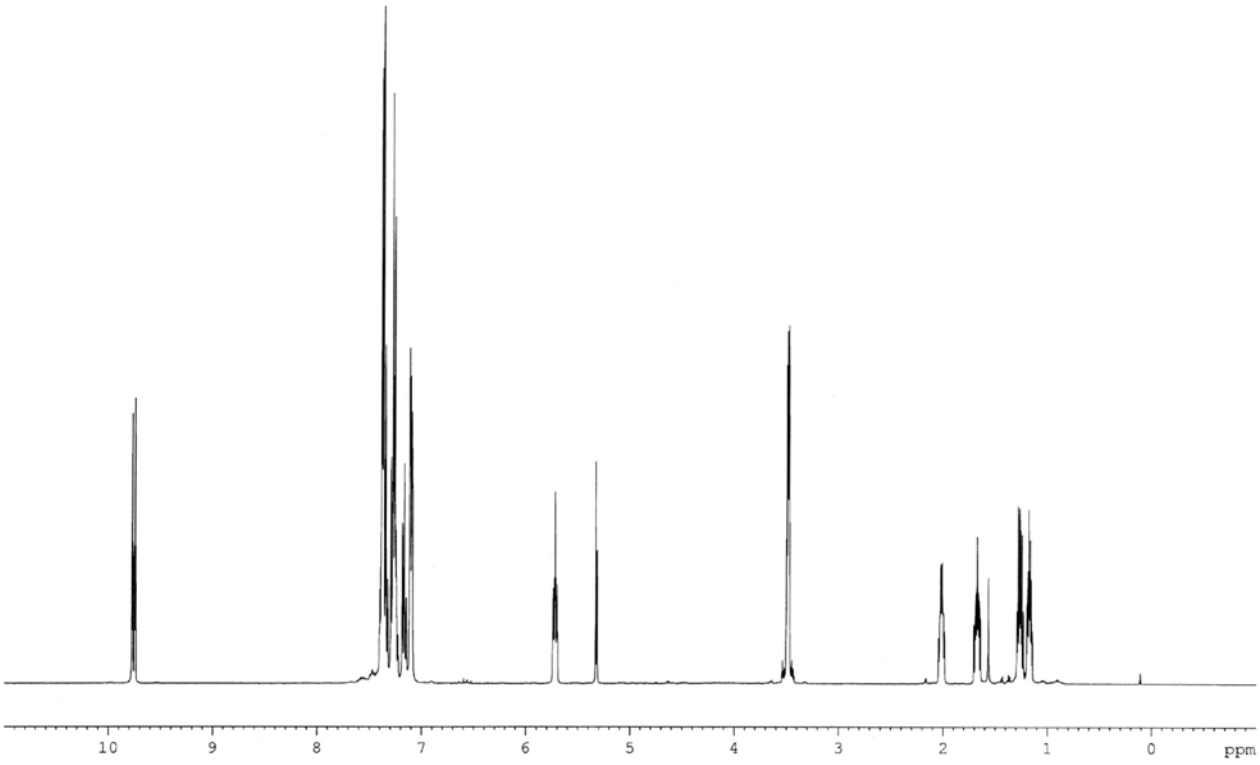

210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

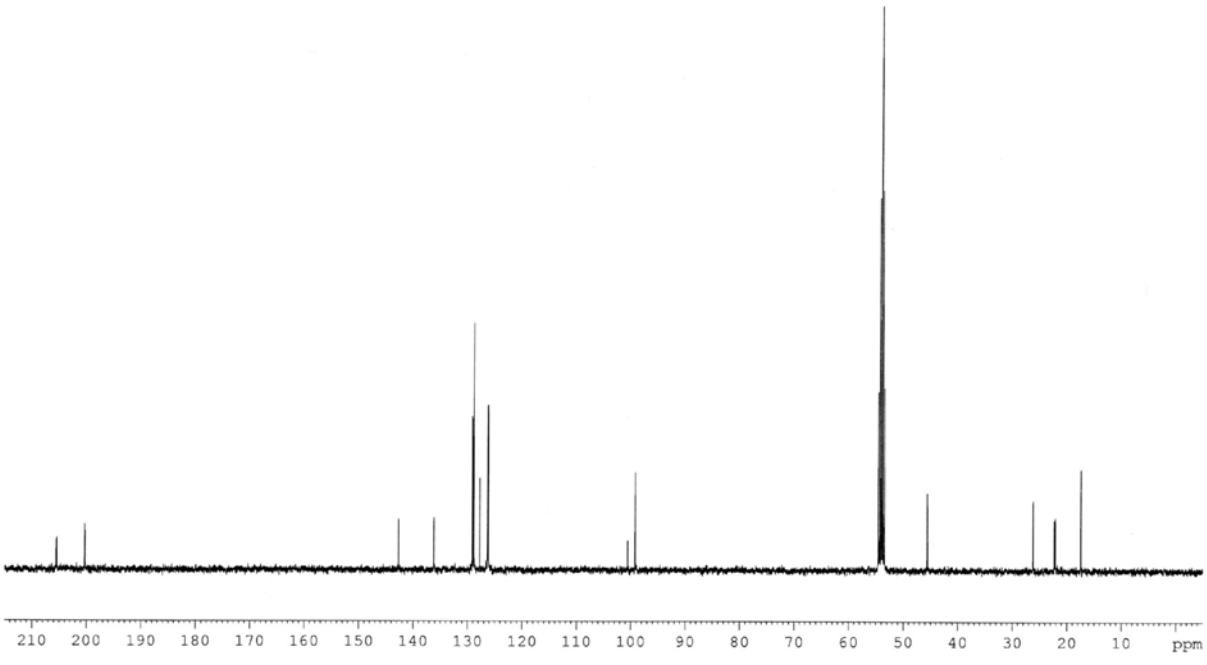

O
Ph
Ph

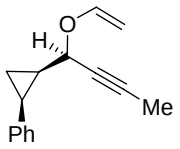

9
8
7
6
5
4
3
2
1
0
ppm

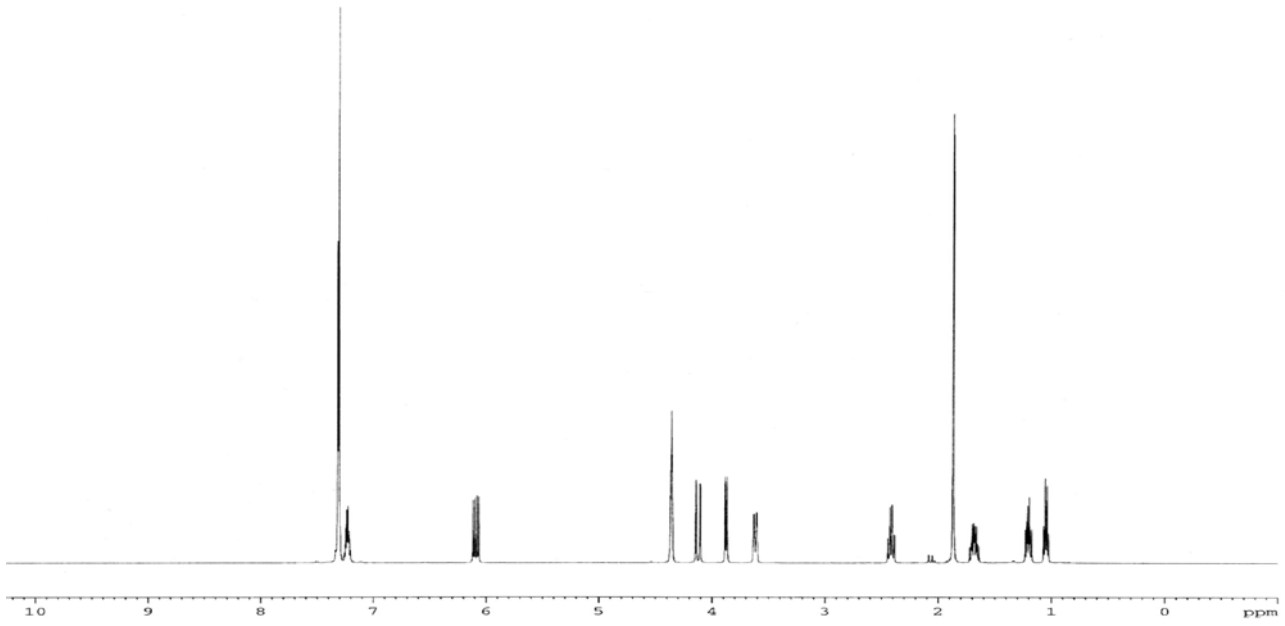

210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

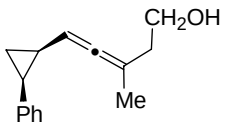

CHO
Ph
Ph

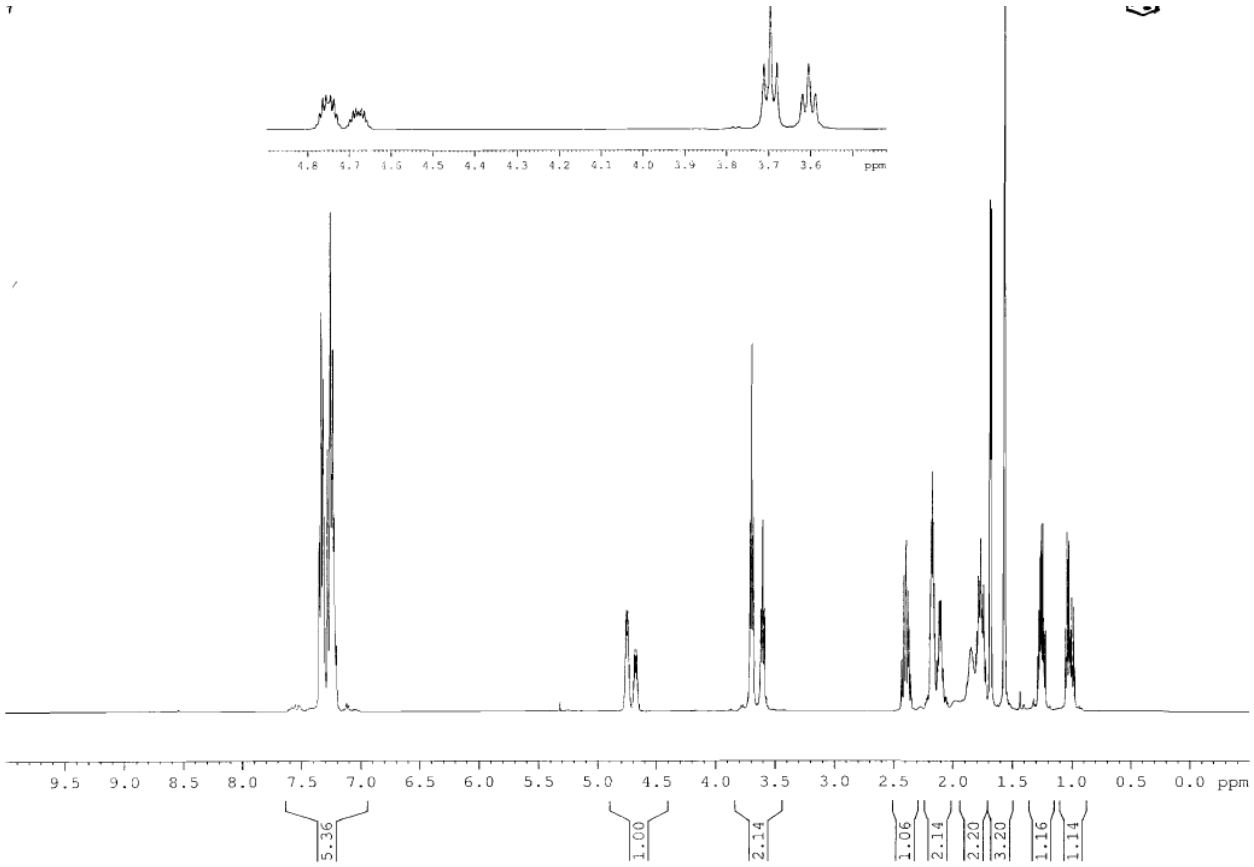

10
9
8
7
6
5
4
3
2
1
0
ppm

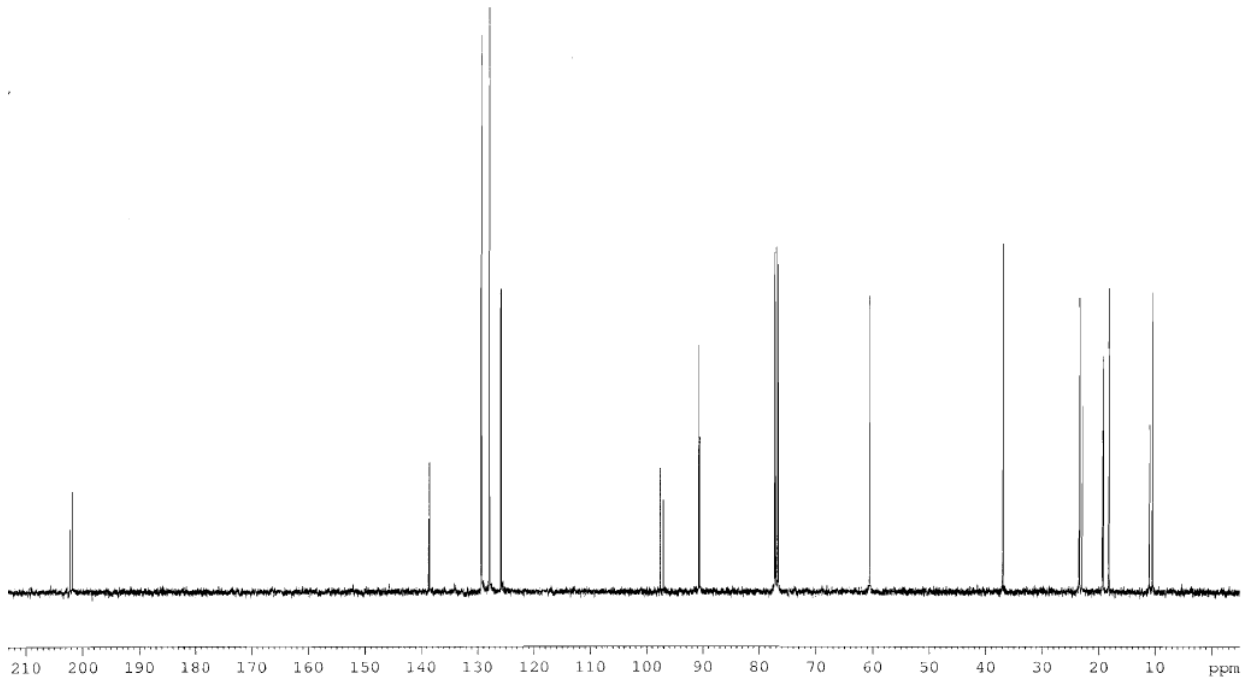

210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

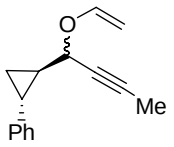

O
H
Me
Ph

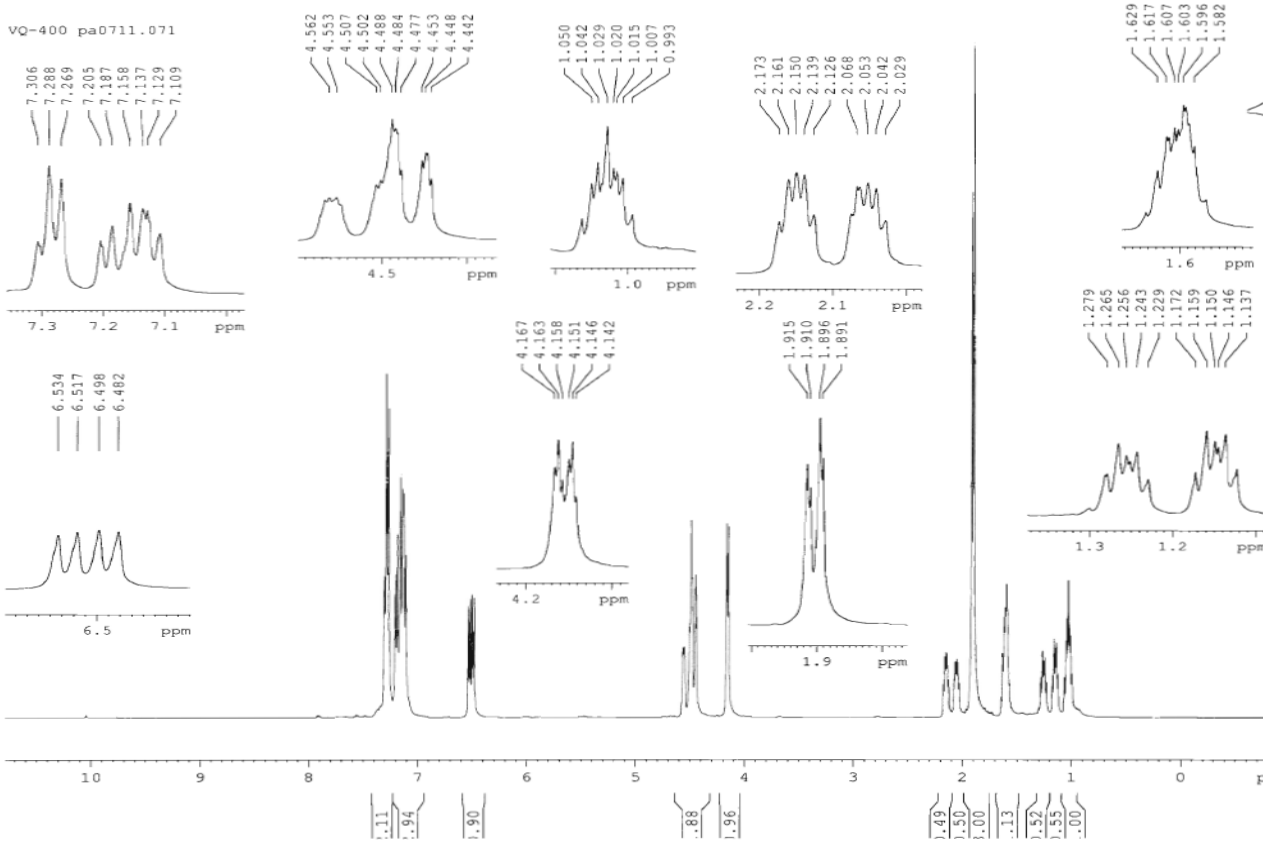

10
9
8
7
6
5
4
3
2
1
0
ppm

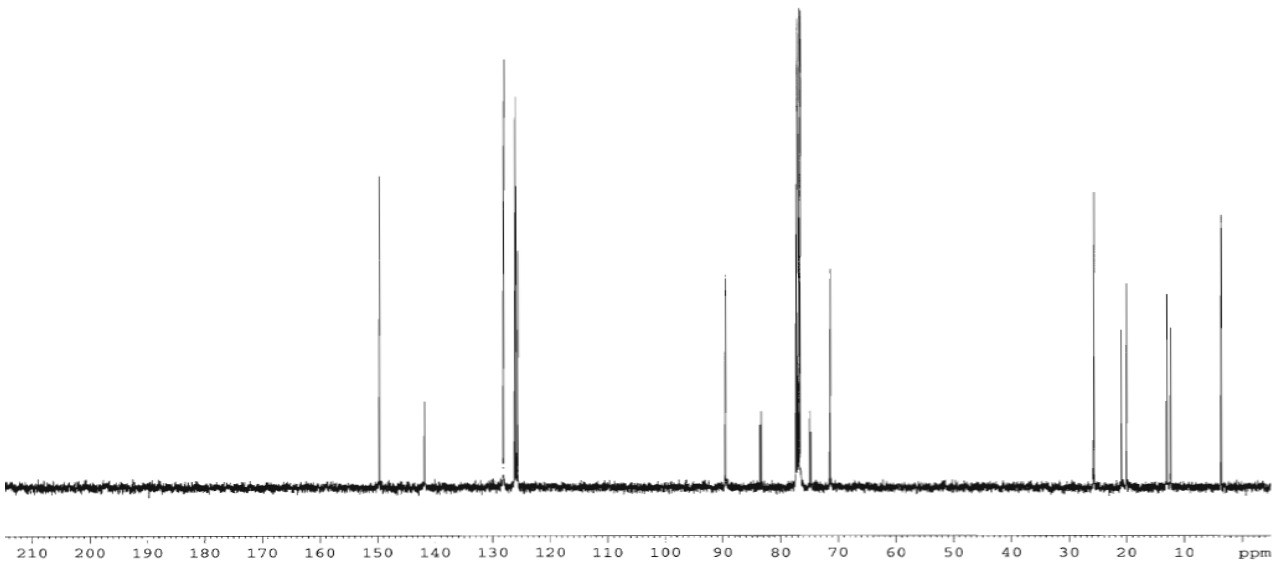

CH2OH
Ph
Me

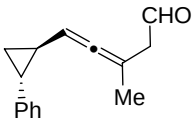

4.8 4.7 4.6 4.5 4.4 4.3 4.2 4.1 4.0 3.9 3.8 3.7 3.6 ppm
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
5.36
1.00
2.14
1.06
2.14
2.20
3.20
1.16
1.14

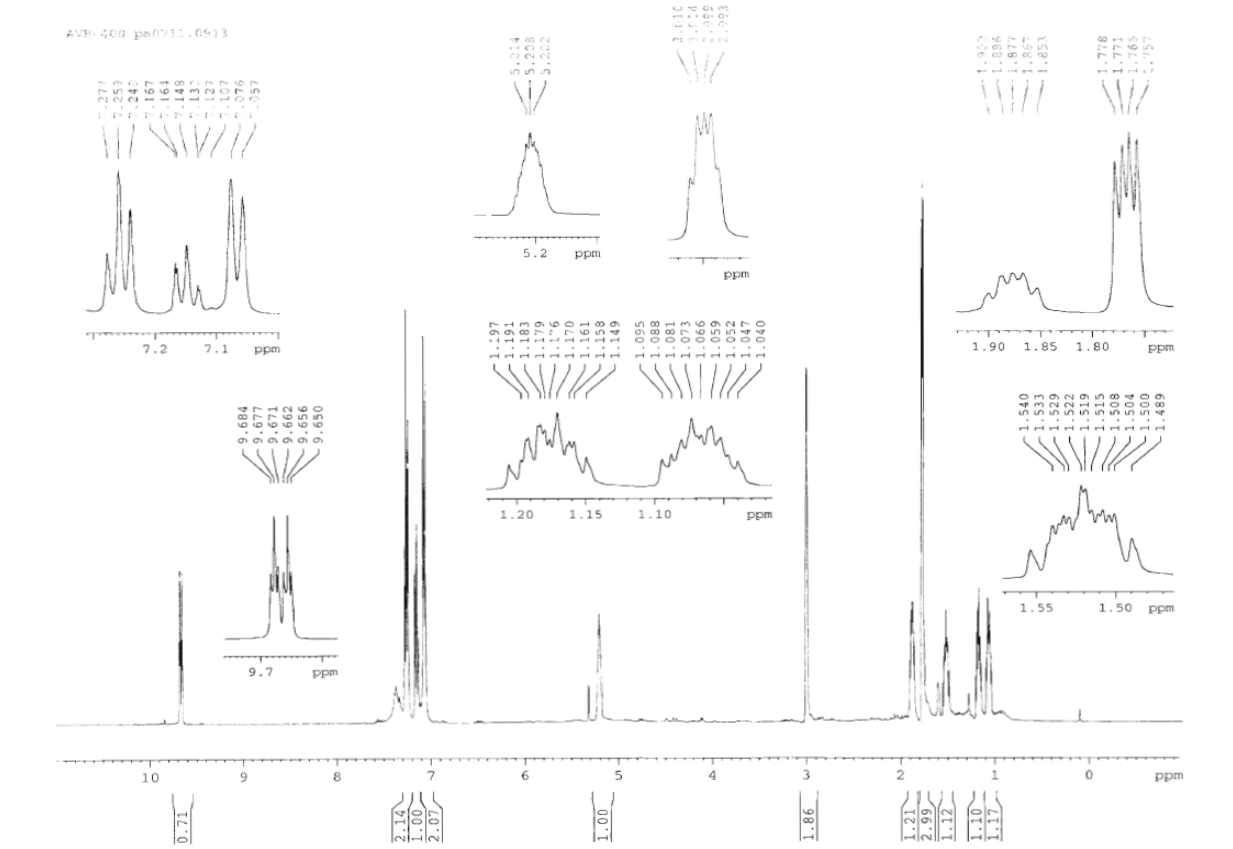

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

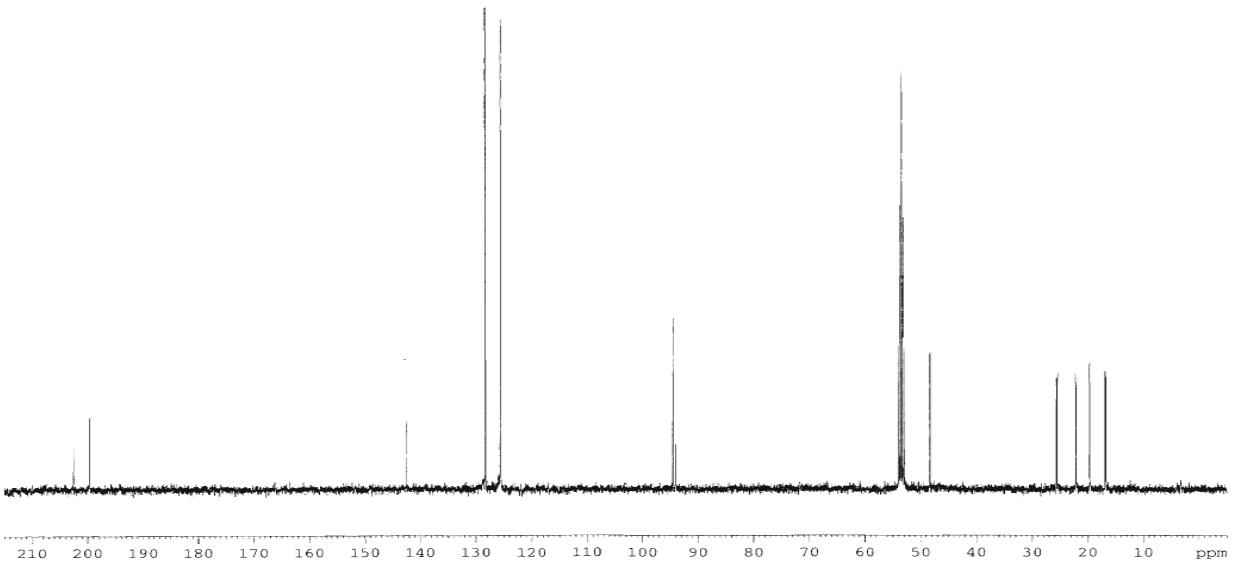

O
Me
Ph

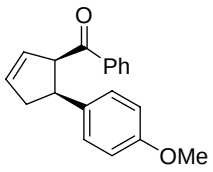

VQ-400 pa0711.071
7.306
7.288
7.269
7.205
7.187
7.158
7.137
7.129
7.109
4.562
4.553
4.507
4.502
4.488
4.484
4.477
4.453
4.448
4.442
1.050
1.042
1.029
1.020
1.015
1.007
0.993
2.173
2.161
2.150
2.139
2.126
2.068
2.053
2.042
2.029
1.629
1.617
1.607
1.603
1.596
1.582
6.534
6.517
6.498
6.482
4.167
4.163
4.158
4.151
4.146
4.142
1.915
1.910
1.896
1.891
1.279
1.265
1.256
1.243
1.229
1.172
1.159
1.150
1.146
1.137
ppm

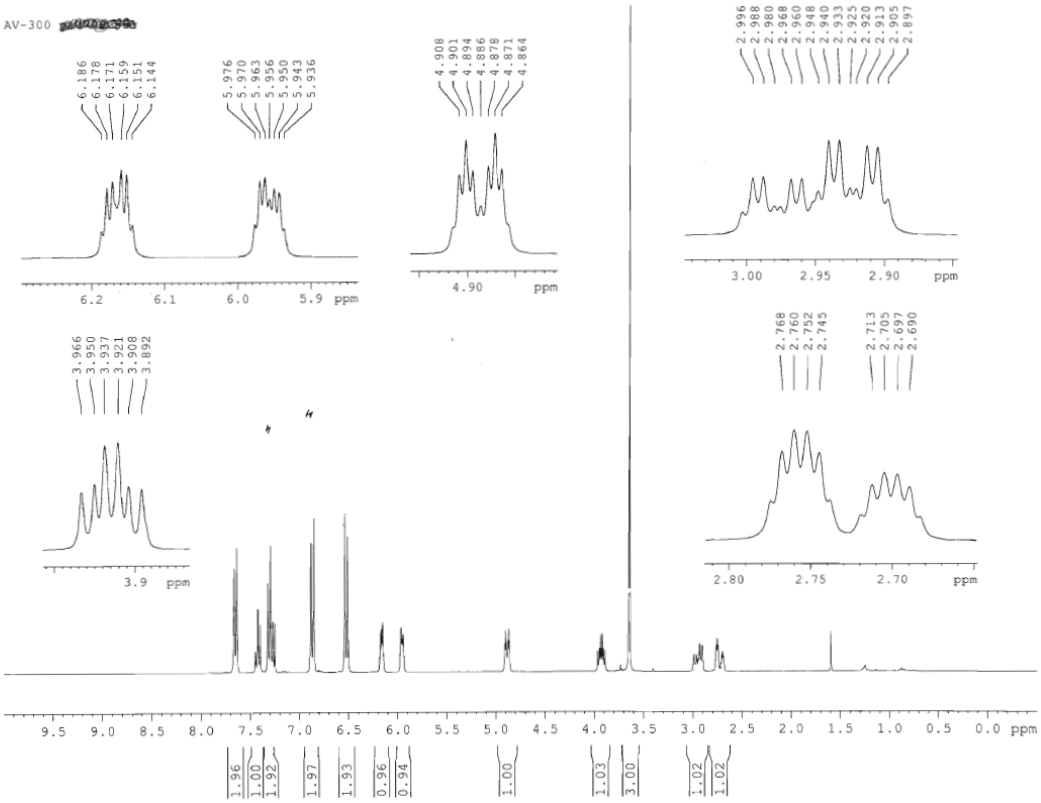

ppm

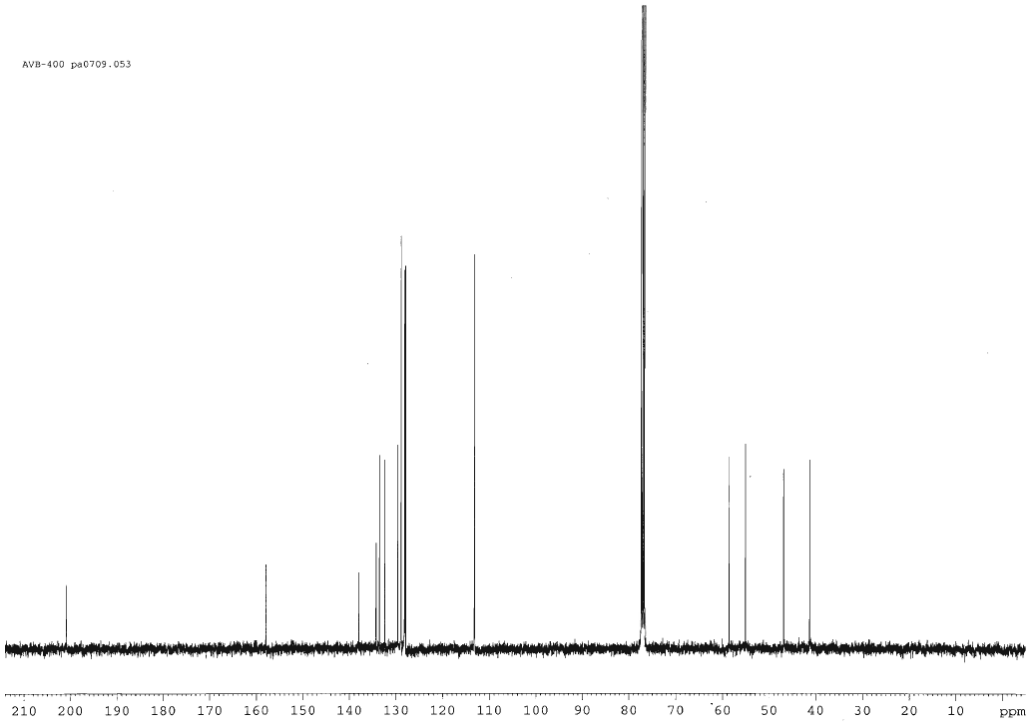

CHO
Me
Ph



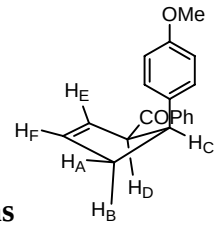


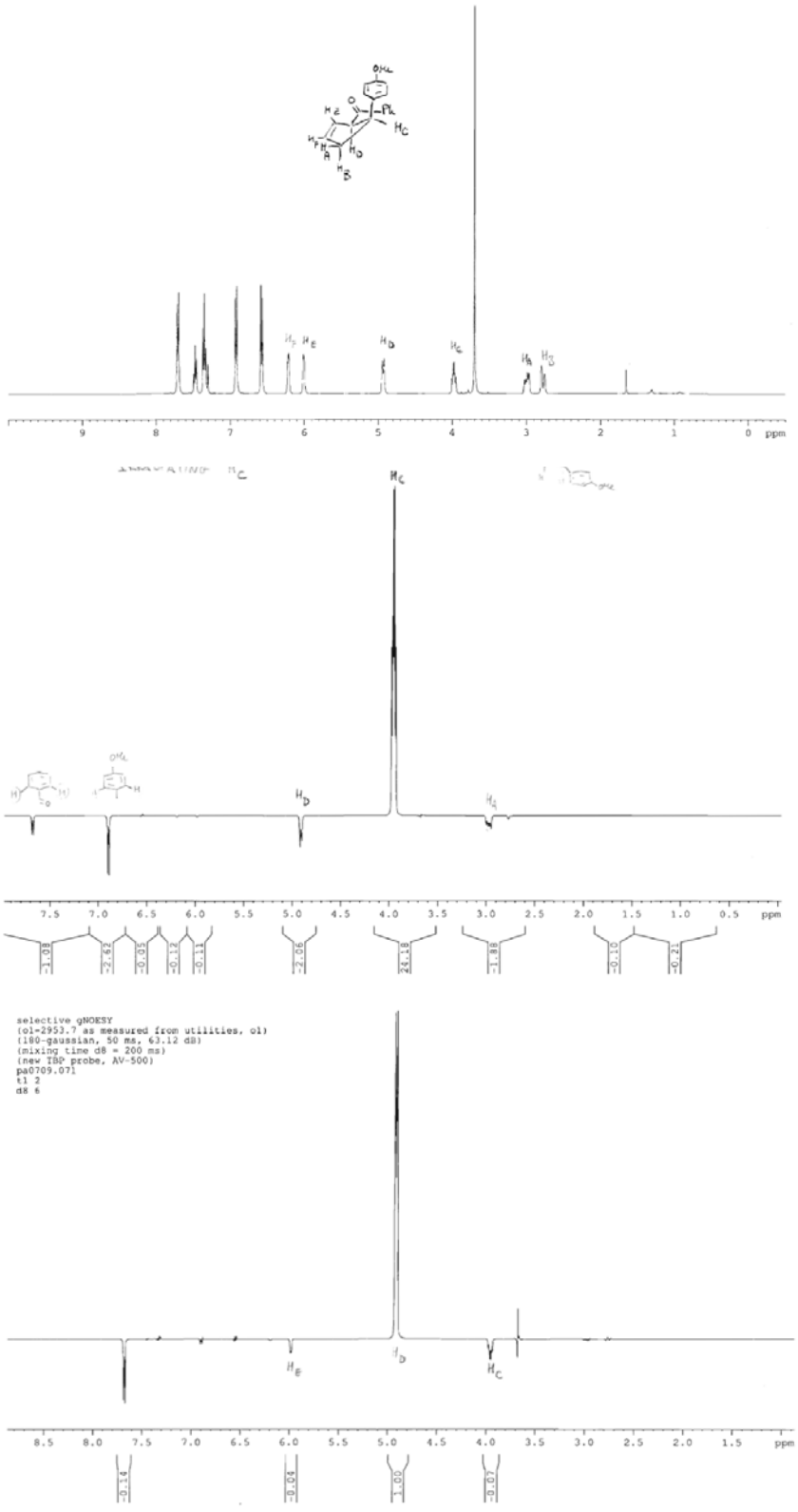

O
Ph
OMe

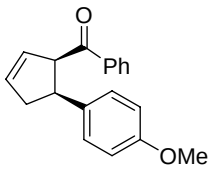

AV-300
6.186
6.178
6.171
6.159
6.151
6.144
5.976
5.970
5.963
5.956
5.950
5.943
5.936
4.908
4.901
4.894
4.886
4.878
4.871
4.864
2.996
2.988
2.980
2.968
2.960
2.948
2.940
2.933
2.925
2.920
2.913
2.905
2.897
3.966
3.950
3.937
3.921
3.908
3.892
2.768
2.760
2.752
2.745
2.713
2.705
2.697
2.690
6.2
6.1
6.0
5.9 ppm
4.90 ppm
3.00
2.95
2.90 ppm
3.9 ppm
2.80
2.75
2.70 ppm
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.96
1.00
1.92
1.97
1.93
0.96
0.94
1.00
1.03
3.00
1.02
1.02


AVB-400 pa0709.053
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

NOESY correlations

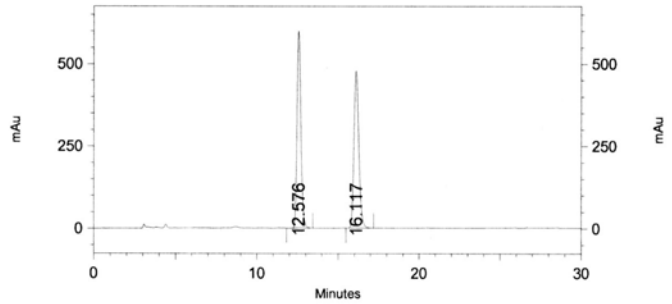






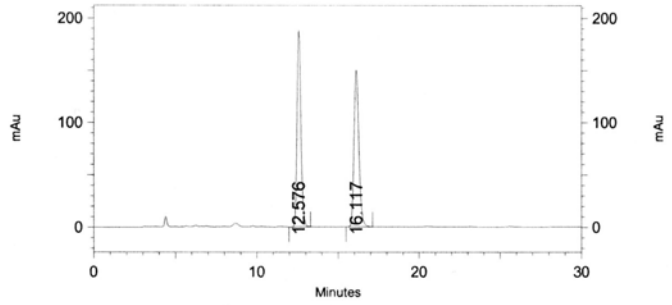
Sample ID: pa0709.081
Filename:
C:\EZStart\Projects\Default\Data\Pablo\2007\pa0709.081AD9703IP.metBeach9-7-2007 8-55-33 PM.dat
Method:
C:\EZStart\Projects\Default\Method\AD\AD9703IP.met
Injection volume: 5 uL

Description: {Data Description}



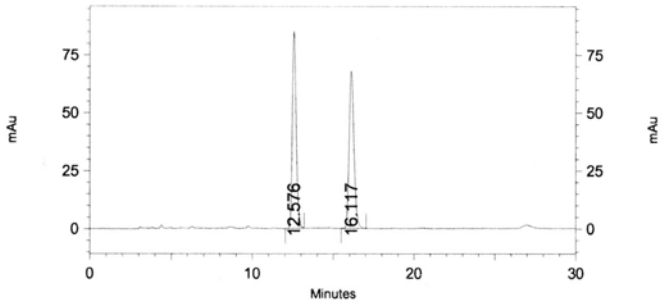
1: 230 nm, 4 nm Results

Retention Time	Area	Area Percent
12.576	9912233	49.915
16.117	9945909	50.085



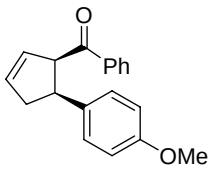
2: 254 nm, 4 nm Results

Retention Time	Area	Area Percent
12.576	3121421	49.990
16.117	3122714	50.010



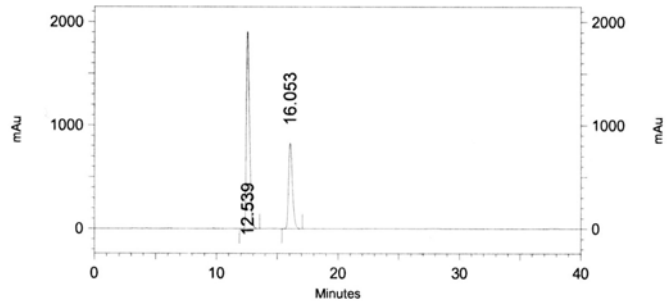
3: 280 nm, 4 nm Results

Retention Time	Area	Area Percent
12.576	1408537	49.978
16.117	1409757	50.022



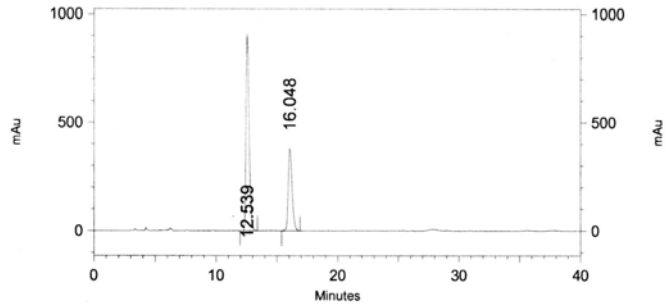
Sample ID: **pa0709151**
Filename:
C:\EZStart\Projects\Default\Data\Pablo\2007\pa0709151AD9703IP40min.metBeach9-14-2007 11-48-54 PM.dat Method:
C:\EZStart\Projects\Default\Method\Britt\AD9703IP40min.met
Injection volume: 5 uL

Description: {Data Description}



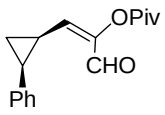
2: 254 nm, 4 nm Results

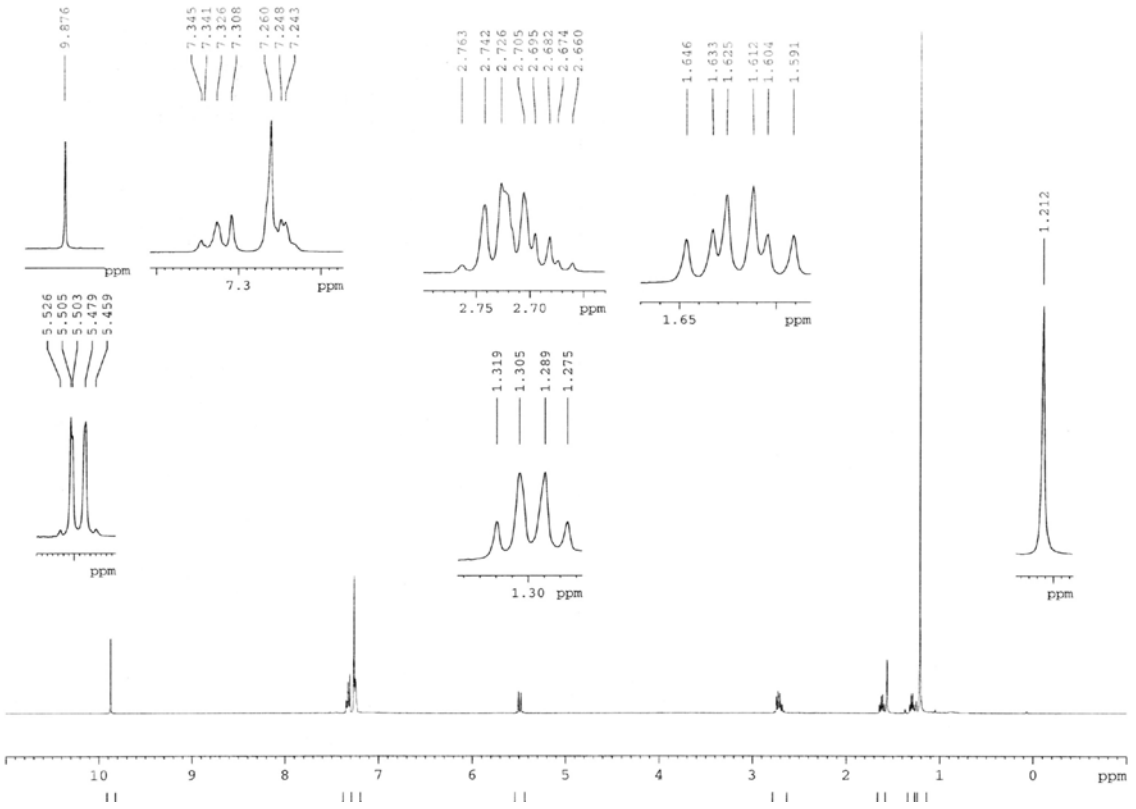
Retention Time	Area	Area Percent
12.539	35211258	66.063
16.053	18088312	33.937

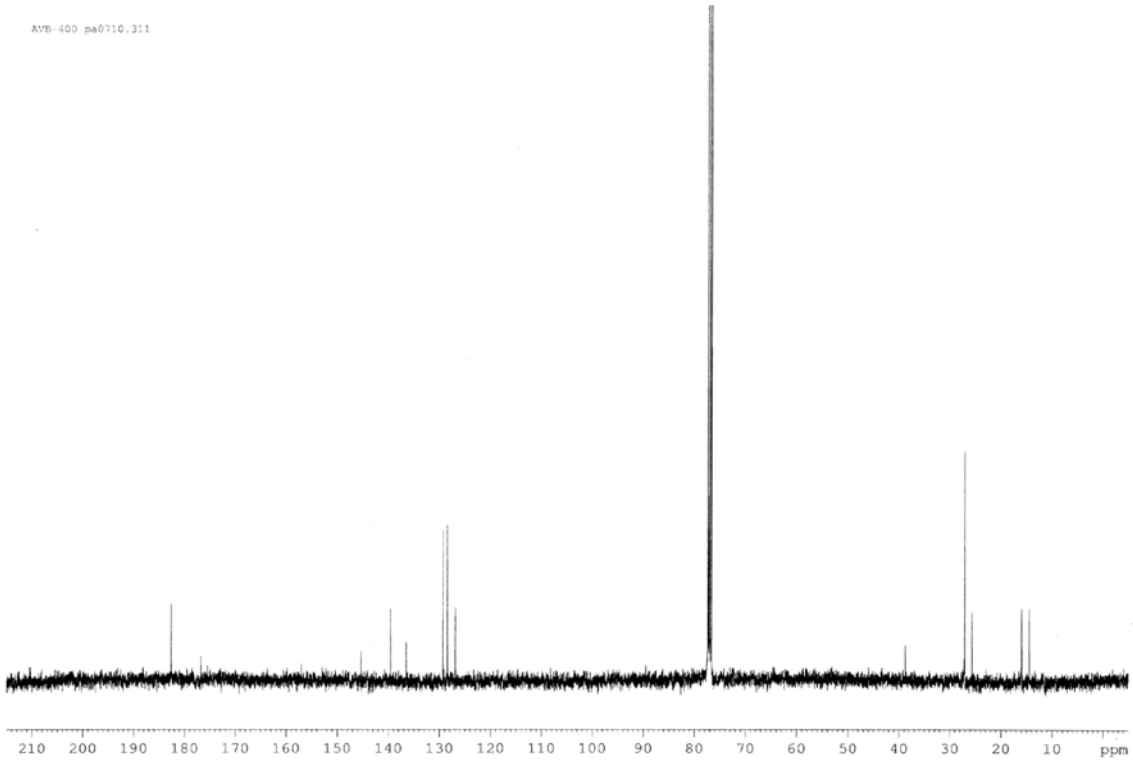


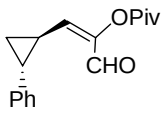
3: 280 nm, 4 nm Results

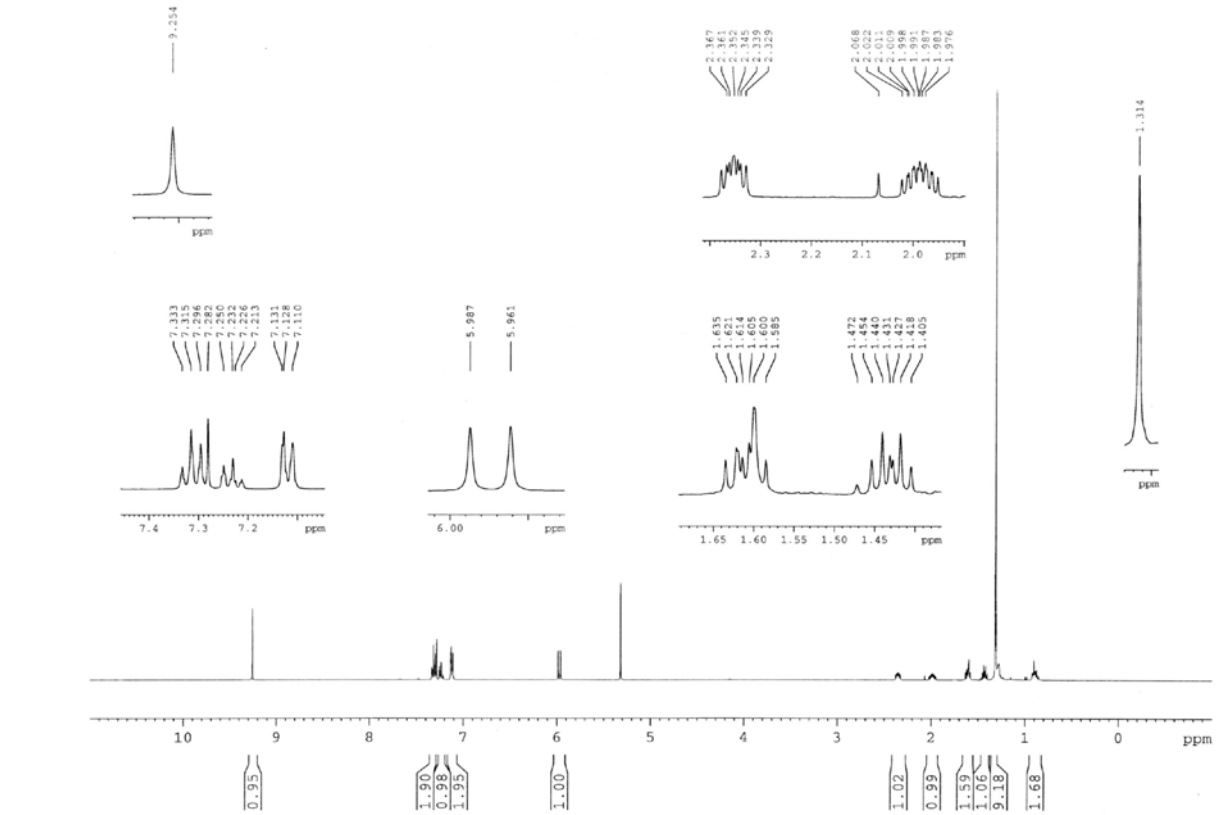
Retention Time	Area	Area Percent
12.539	16428854	66.250
16.048	8369252	33.750

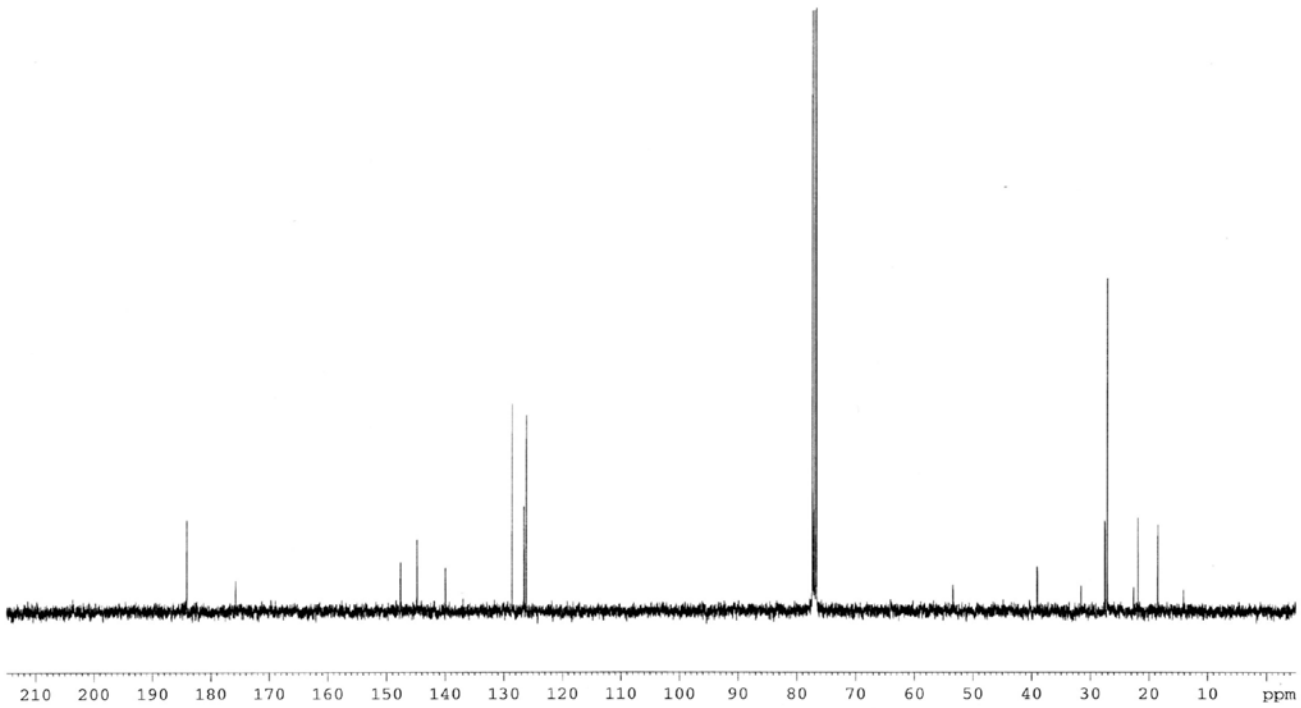

OPiv
CHO
Ph


9.876
7.345
7.341
7.326
7.308
7.260
7.248
7.243
2.763
2.742
2.726
2.705
2.695
2.682
2.674
2.660
1.646
1.633
1.625
1.612
1.604
1.591
1.212
5.526
5.505
5.503
5.479
5.459
1.319
1.305
1.289
1.275
7.3
2.75 2.70
1.65
1.30
ppm


AVB-400 pa0710.311
ppm


OPiv
CHO
Ph


9.254
7.333
7.315
7.296
7.282
7.262
7.252
7.232
7.226
7.213
7.131
7.128
7.110
5.987
5.961
2.367
2.361
2.352
2.345
2.339
2.329
2.068
2.022
2.011
2.009
1.998
1.991
1.987
1.983
1.976
1.635
1.621
1.614
1.605
1.600
1.595
1.472
1.454
1.440
1.431
1.427
1.418
1.405
1.314
ppm
0.95
1.90
0.98
1.95
1.00
1.02
0.99
1.59
1.06
9.18
1.68


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