Supporting Information

KOAc-promoted alkynylation of α-C–H bonds of ethers with alkynyl bromides under transition-metal-free conditions

Jiajun Zhang,[a] Pinhua Li,*[a] and Lei Wang*[a,b]

[a] Department of Chemistry, Huaibei Normal University, Huaibei, Anhui 235000, P R China

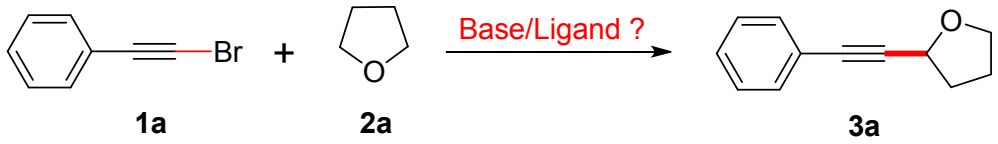
Tel: + 86-561-3802-069 Fax: + 86-561-3090-518 E-mail: leiwang@chnu.edu.cn

[b] State Key Laboratory of Organometallic Chemistry, Shanghai Institute of Organic Chemistry, Chinese Academy of Sciences, Shanghai 200032, P R China

Table of Contents for Supporting Information

1. Effect of base/ligand on the model reaction (TS1)

Table S1 Effect of base/ligand on the model reaction.[a]

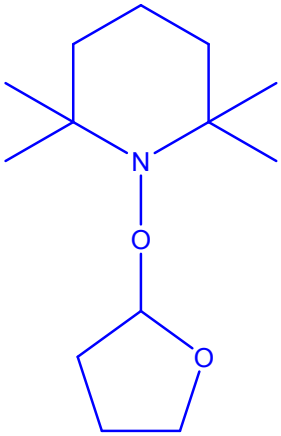


1a **2a** **3a**

Entry	Base	Ligand	Yield (%)[b]
1	KO^{t}Bu	L-Proline	34
2	KO^{t}Bu	TMEDA	NR
3	KO^{t}Bu	1,10-Phen	NR
4	KO^{t}Bu	Bipy	29
5	KO^{t}Bu	8-HQ	NR
6	KO^{t}Bu	Dppf	NR
7	NaO^{t}Bu	L-Proline	11
8	NaO^{t}Bu	TMEDA	NR
9	NaO^{t}Bu	1,10-Phen	NR
10	NaO^{t}Bu	Bipy	NR
11	NaO^{t}Bu	8-HQ	NR
12	NaO^{t}Bu	Dppf	NR
13	LiO^{t}Bu	L-Proline	18
14	LiO^{t}Bu	TMEDA	NR
15	LiO^{t}Bu	1,10-Phen	NR
16	LiO^{t}Bu	Bipy	NR
17	LiO^{t}Bu	8-HQ	NR
18	LiO^{t}Bu	Dppf	NR

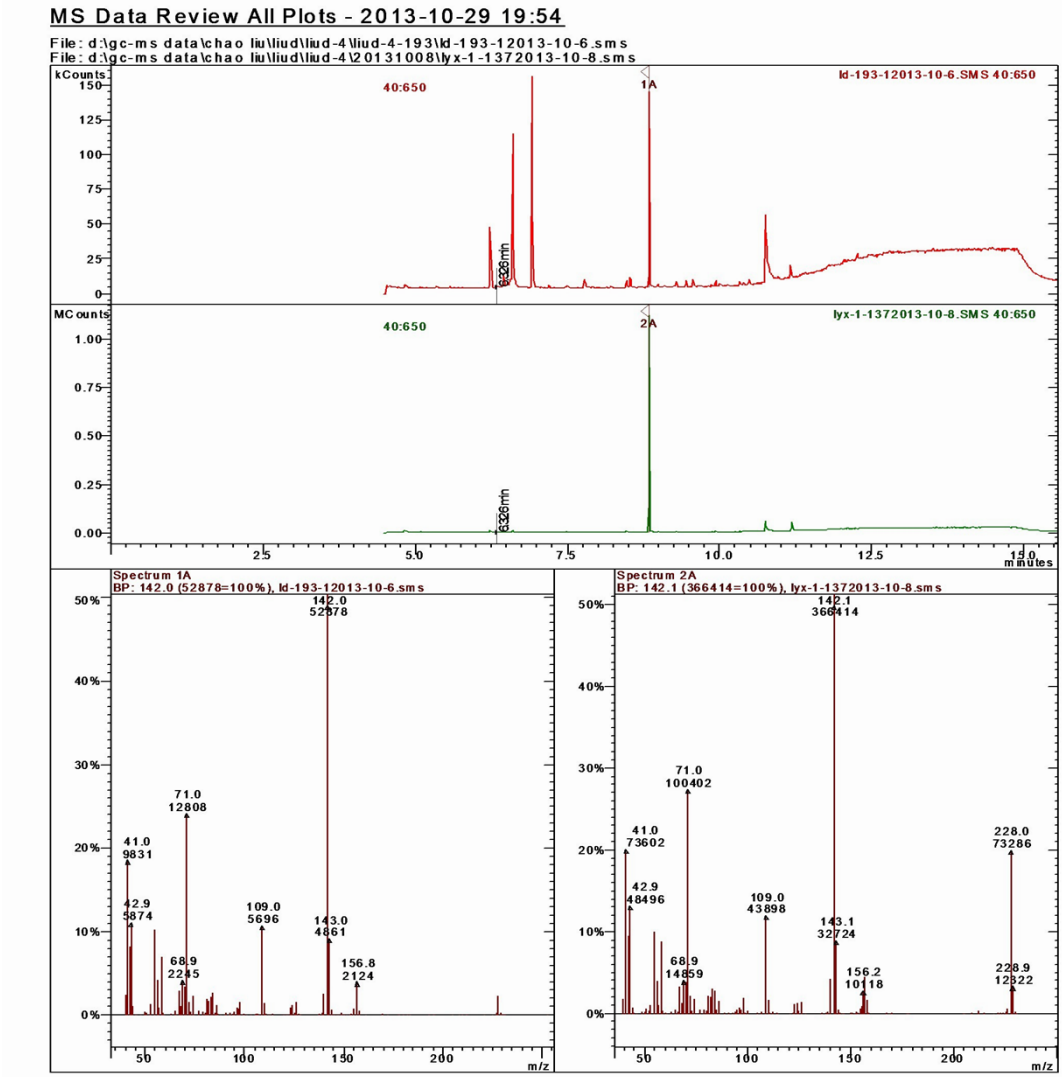
[a] Reaction conditions: **1a** (0.30 mmol), **2a** (2.0 mL, excess), base (0.60 mmol), ligand (0.15 mmol) at 150 °C for 12 h. [b] Isolated yield. TMEDA = *N,N,N',N'*-tetramethylethylenediamine. 1,10-Phen = 1,10-phenanthroline. Bipy = 2,2'-bipyridine. 8-HQ = 8-hydroxyquinoline. Dppf = 1,1'-bis(diphenylphosphino)ferrocene.

2. GC-MS of the TEMPO with tetrahydrofuran and phenylethynyl bromide under the standard reaction conditions

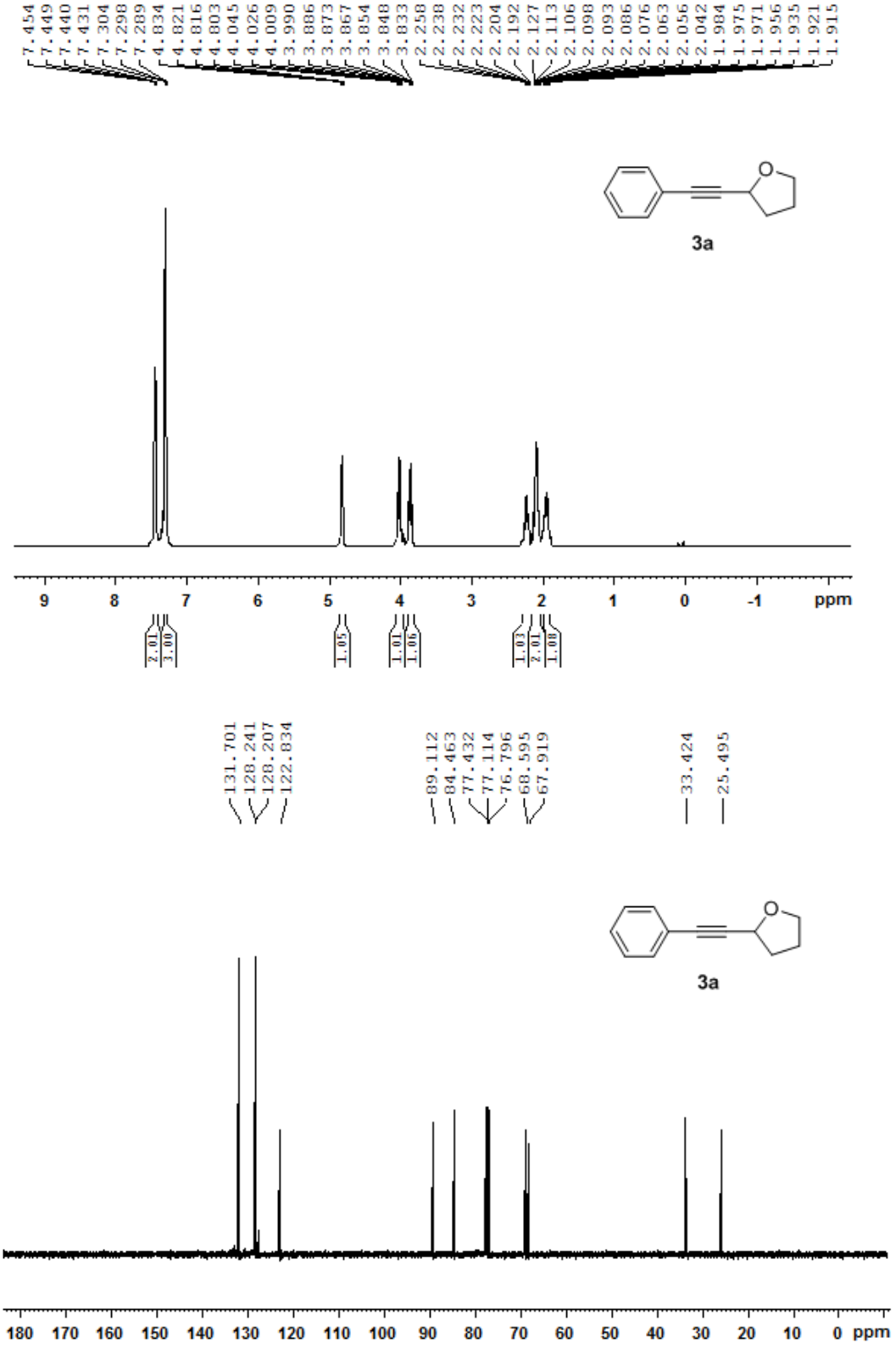


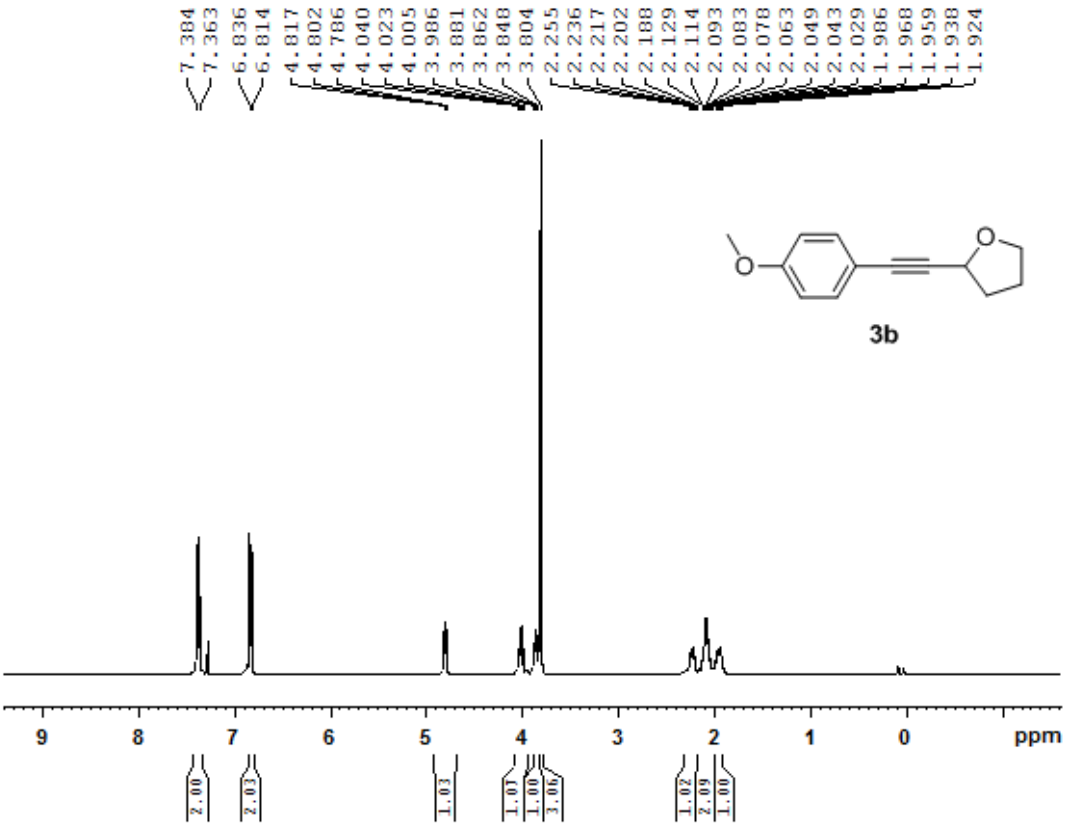
Chemical Formula: $C_{13}H_{25}NO_2$

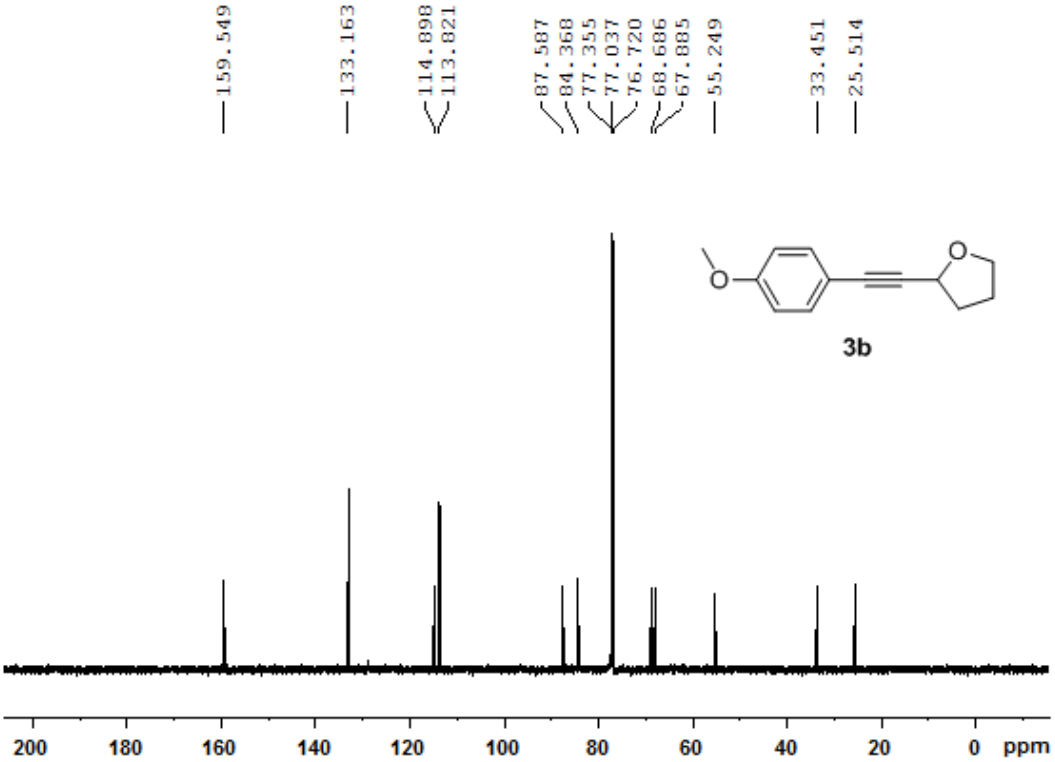
$[M+H]^+ = C_{13}H_{26}NO_2$
Exact Mass: 228.20
Molecular Weight: 228.35
m/z: 228.20 (100.0%), 229.20 (14.4%), 230.20 (1.4%)
Elemental Analysis: C, 68.38; H, 11.48; N, 6.13; O, 14.01

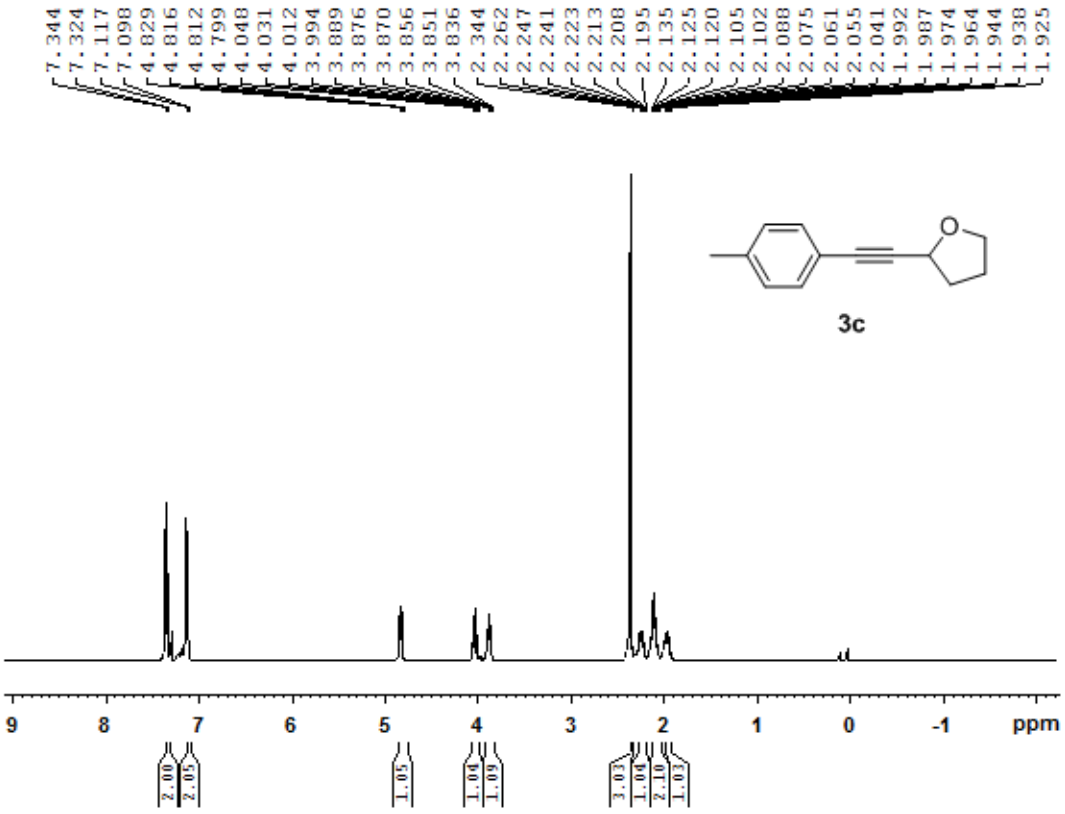


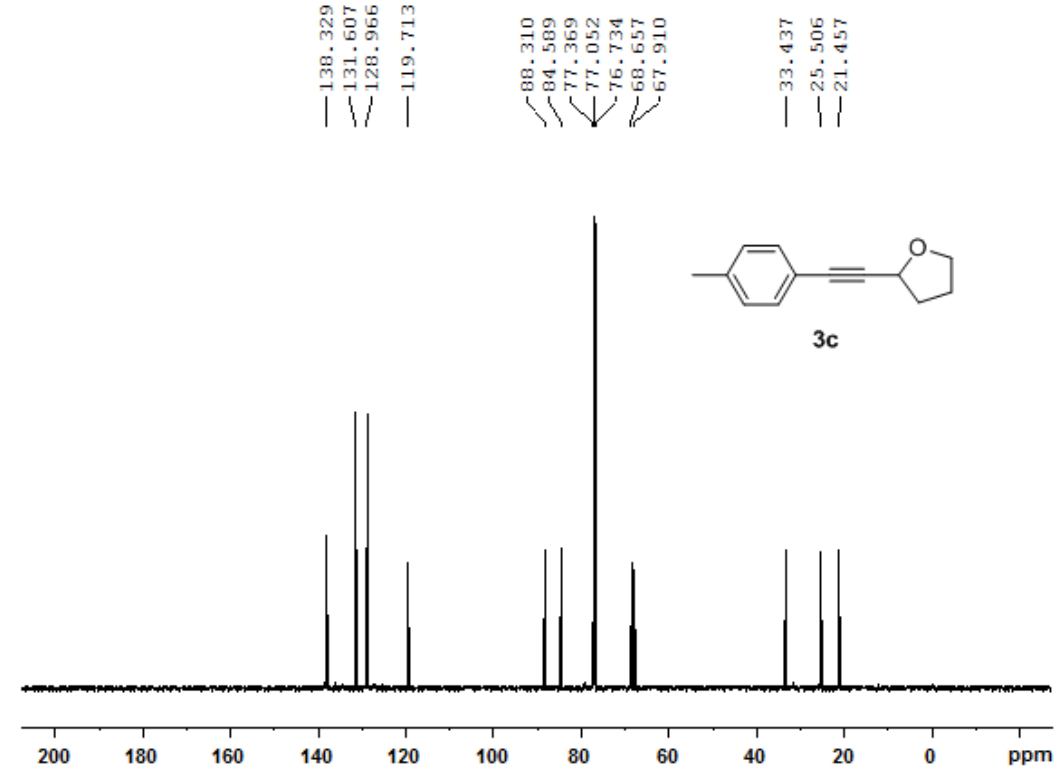
3. ^{1}H NMR and ^{13}C NMR spectra of the products

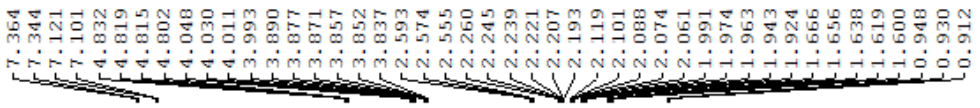


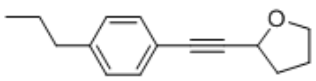

7.384
7.363
6.836
6.814
4.817
4.802
4.786
4.040
4.023
4.005
3.986
3.881
3.862
3.848
3.804
2.255
2.236
2.217
2.202
2.188
2.129
2.114
2.093
2.083
2.078
2.063
2.049
2.043
2.029
1.986
1.968
1.959
1.938
1.924
O
O
3b
9
8
7
6
5
4
3
2
1
0
ppm
2.00
2.03
1.01
1.07
1.00
3.06
1.02
2.09
1.00


159.549
133.163
114.898
113.821
87.587
84.368
77.355
77.037
76.720
68.686
67.885
55.249
33.451
25.514
O
O
3b
200
180
160
140
120
100
80
60
40
20
0
ppm

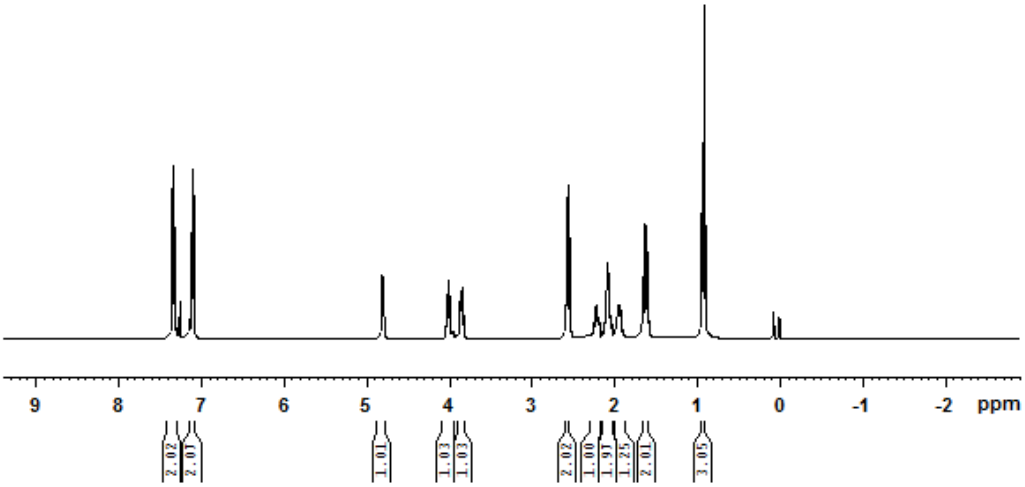

7.344
7.324
7.117
7.098
4.829
4.816
4.812
4.799
4.048
4.031
4.012
3.994
3.889
3.876
3.870
3.856
3.851
3.836
2.344
2.262
2.247
2.241
2.223
2.213
2.208
2.195
2.135
2.125
2.120
2.105
2.102
2.088
2.075
2.061
2.055
2.041
1.992
1.987
1.974
1.964
1.944
1.938
1.925
3c
9
8
7
6
5
4
3
2
1
0
-1
ppm
2.00
2.05
1.05
1.04
1.09
3.03
1.04
2.10
1.01

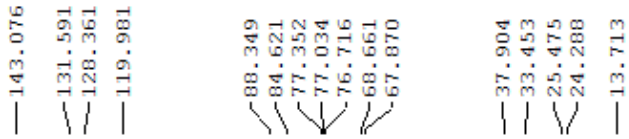

138.329
131.607
128.966
119.713
88.310
84.589
77.369
77.052
76.734
68.657
67.910
33.437
25.506
21.457
3c
200
180
160
140
120
100
80
60
40
20
0
ppm

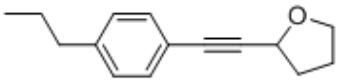

7.364
7.344
7.121
7.101
4.832
4.819
4.815
4.802
4.048
4.030
4.011
3.993
3.890
3.877
3.871
3.857
3.852
3.837
2.593
2.574
2.555
2.260
2.245
2.239
2.221
2.207
2.193
2.119
2.101
2.088
2.074
2.061
1.991
1.974
1.963
1.943
1.924
1.666
1.656
1.638
1.619
1.600
0.948
0.930
0.912



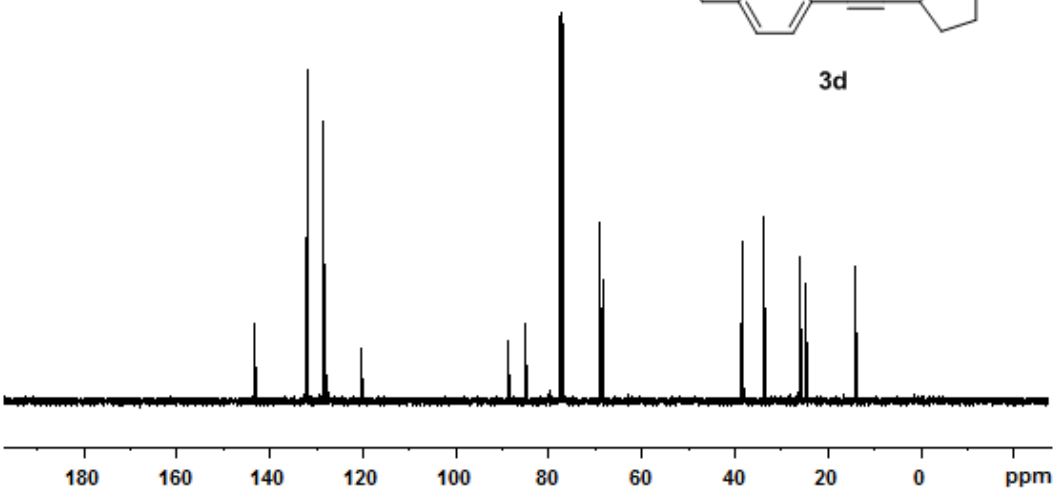
3d

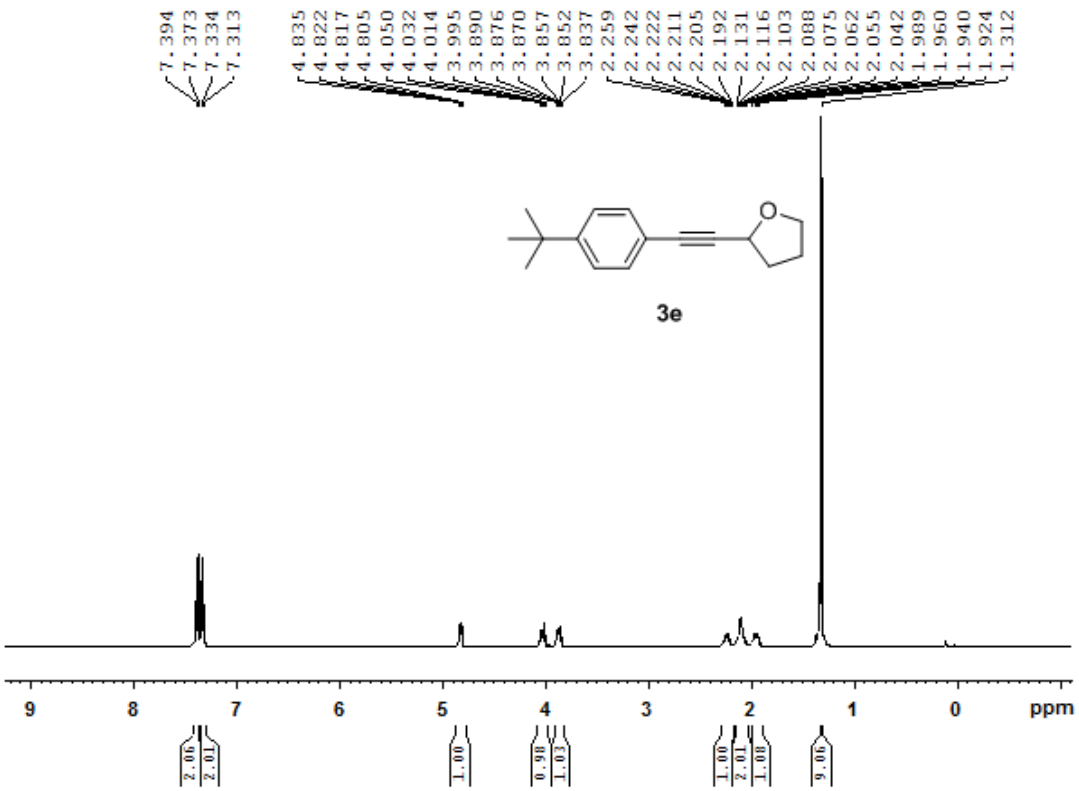

9
8
7
6
5
4
3
2
1
0
-1
-2
ppm
2.02
2.03
1.01
1.03
1.03
2.02
1.00
1.97
1.25
2.01
3.05

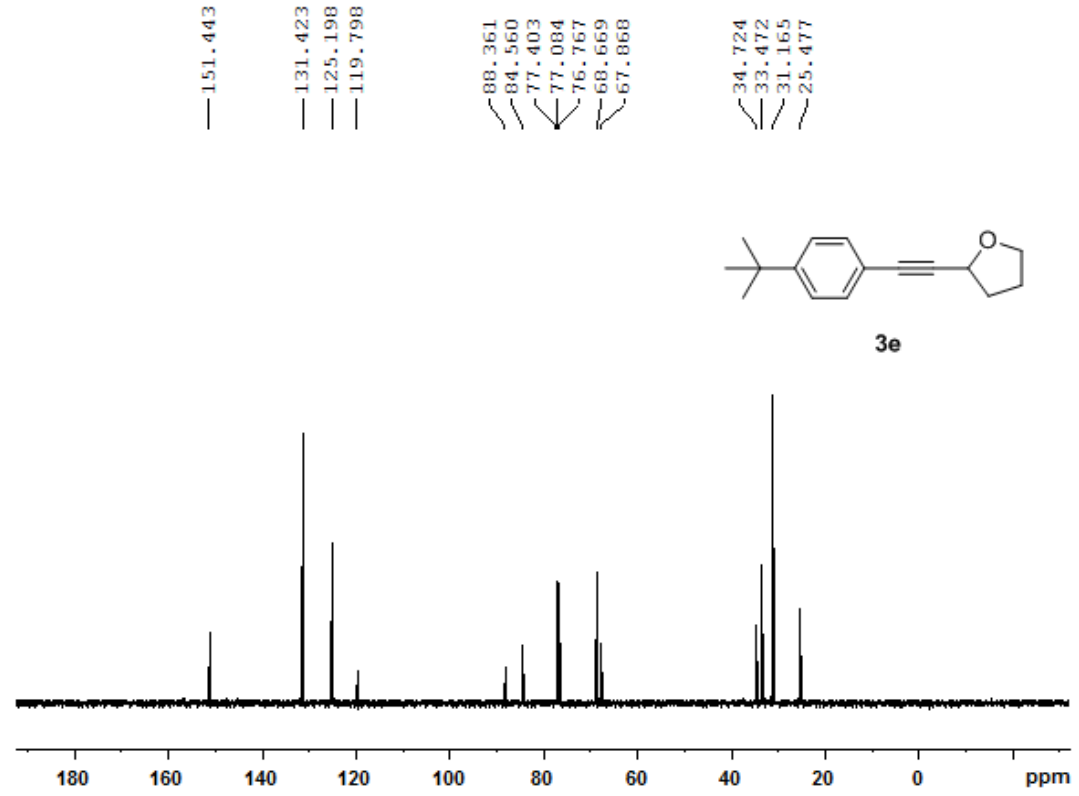

143.076
131.591
128.361
119.981
88.349
84.621
77.352
77.034
76.716
68.661
67.870
37.904
33.453
25.475
24.288
13.713

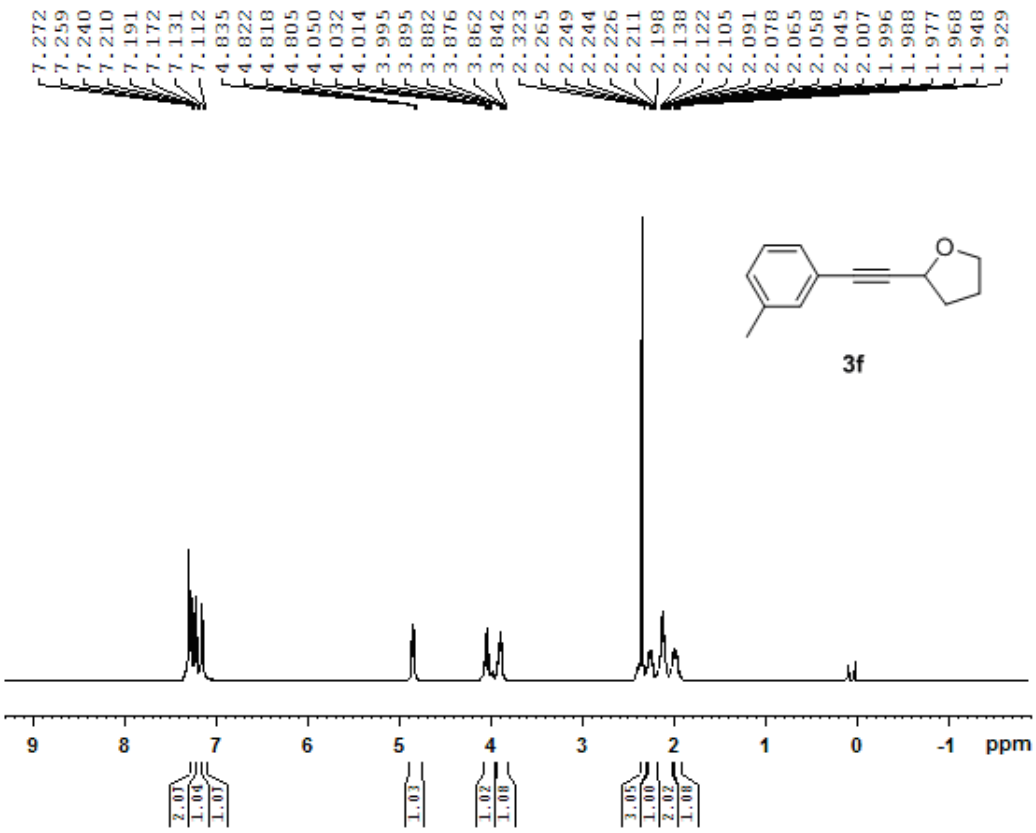


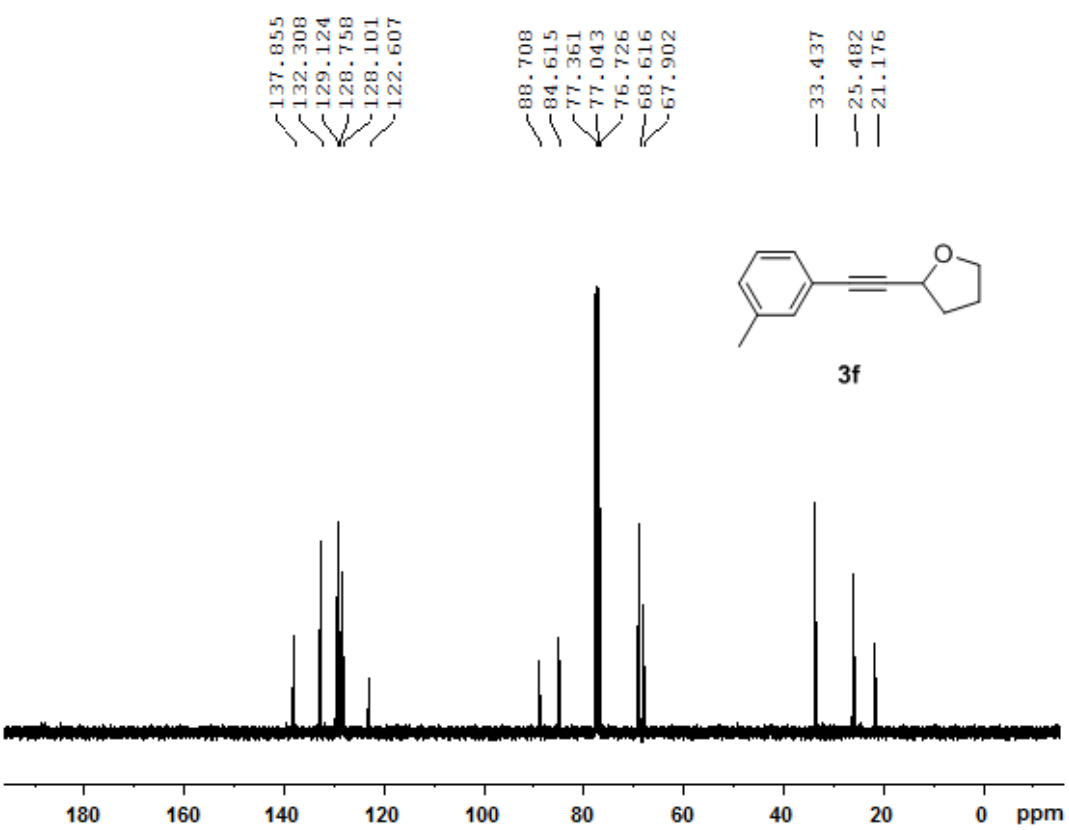
3d

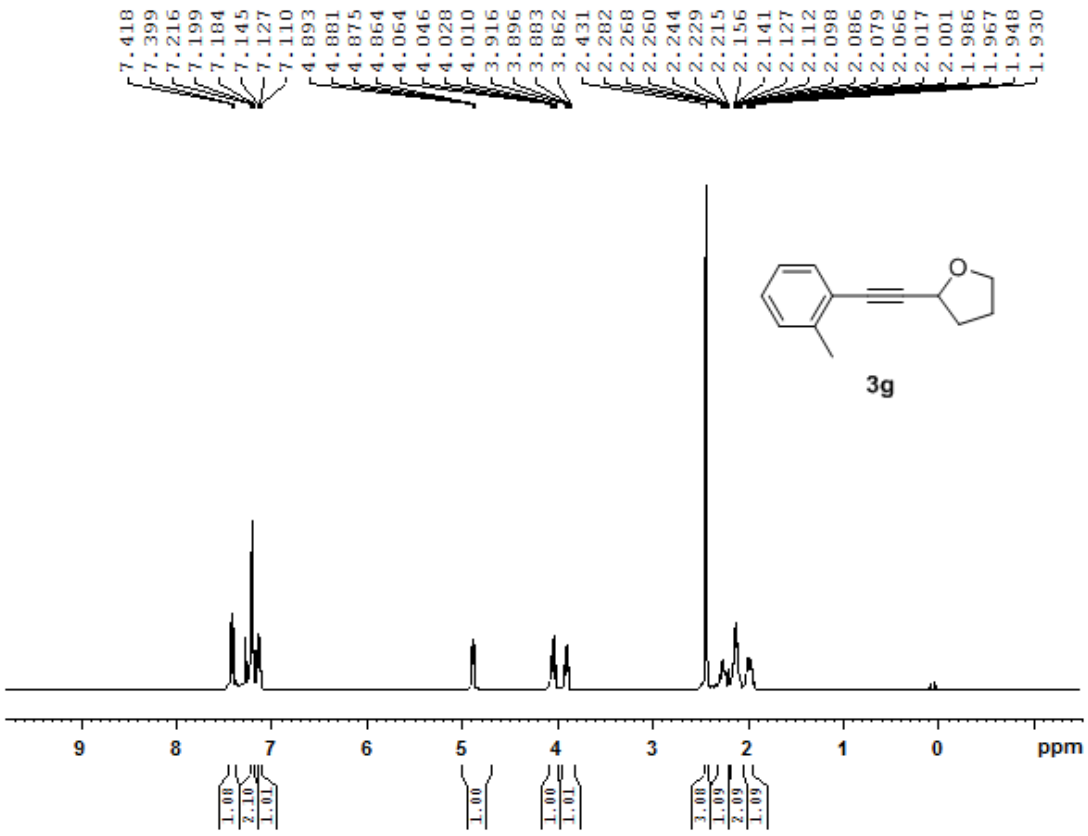

180
160
140
120
100
80
60
40
20
0
ppm

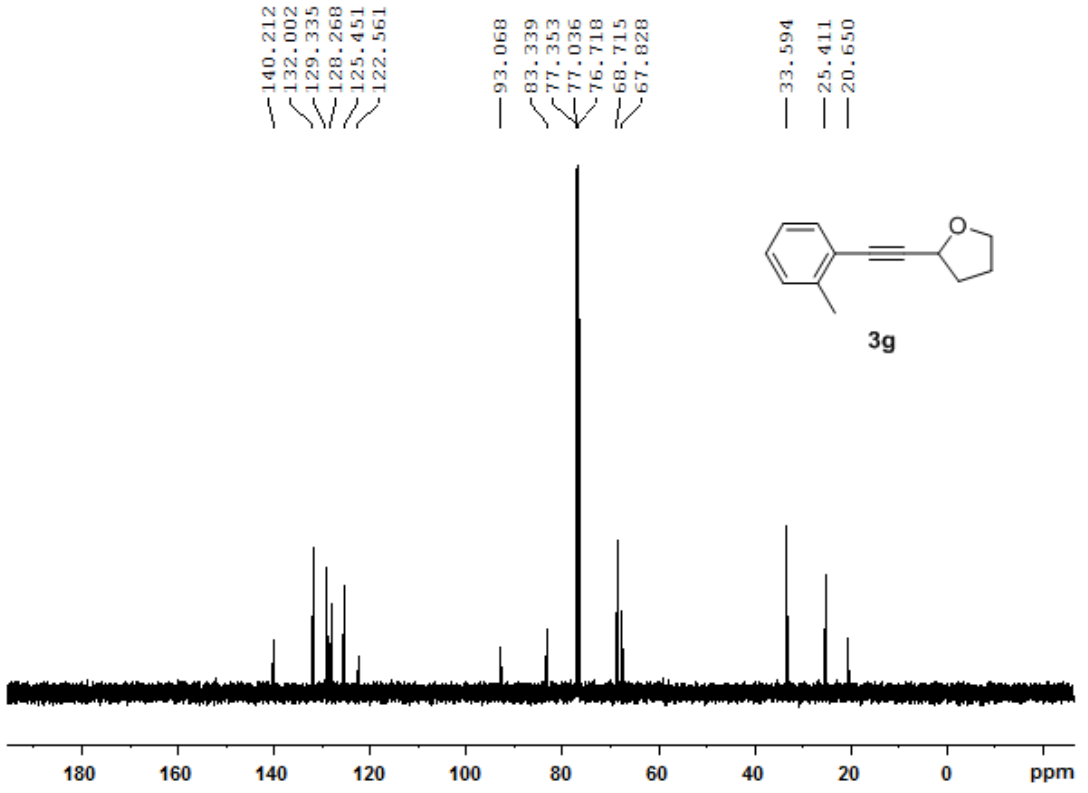

7.394
7.373
7.334
7.313
4.835
4.822
4.817
4.805
4.050
4.032
4.014
3.995
3.890
3.876
3.870
3.857
3.852
3.837
2.259
2.242
2.222
2.211
2.205
2.192
2.131
2.116
2.103
2.088
2.075
2.062
2.055
2.042
1.989
1.960
1.940
1.924
1.312
3e
9
8
7
6
5
4
3
2
1
0
ppm
2.06
2.01
1.00
0.98
1.03
1.00
2.01
1.08
9.06

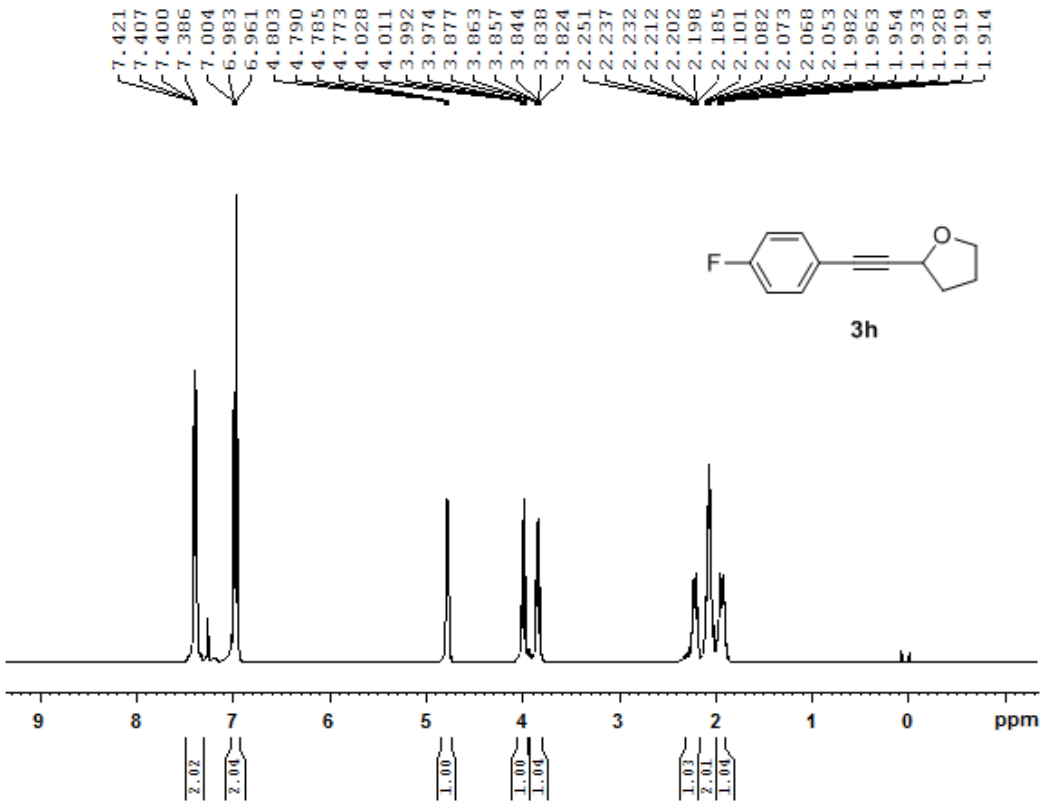

151.443
131.423
125.198
119.798
88.361
84.560
77.403
77.084
76.767
68.669
67.868
34.724
33.472
31.165
25.477
3e
180
160
140
120
100
80
60
40
20
0
ppm

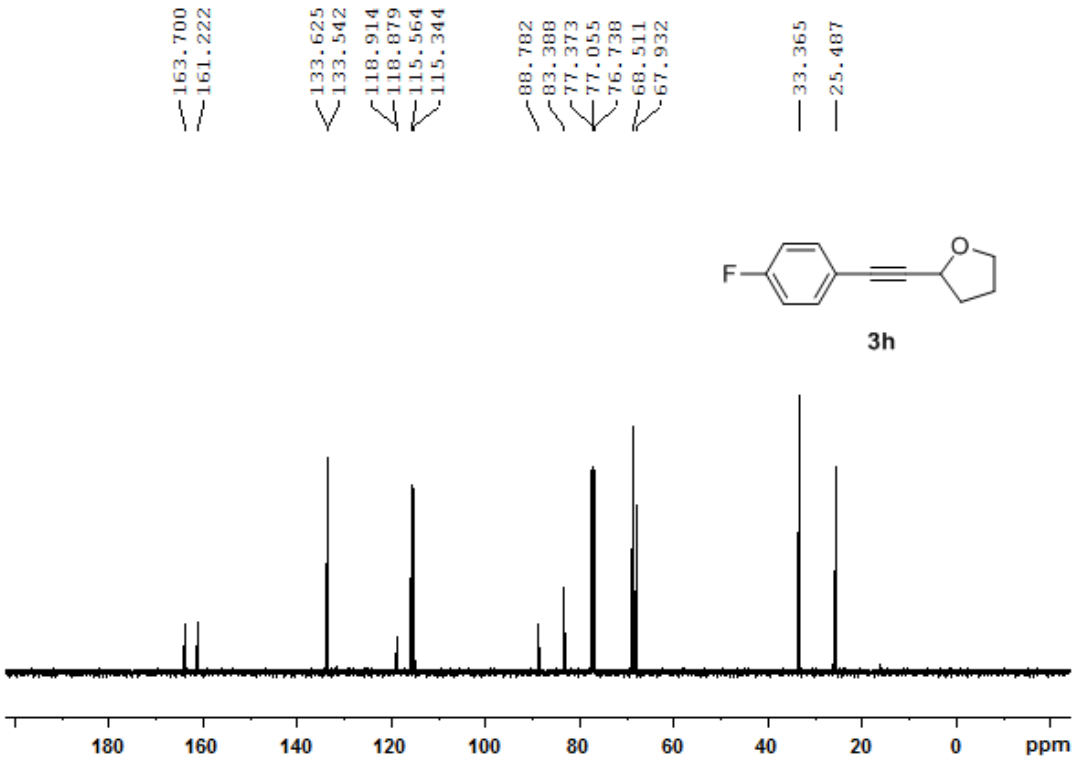

7.272
7.259
7.240
7.210
7.191
7.172
7.131
7.112
4.835
4.822
4.818
4.805
4.050
4.032
4.014
3.995
3.895
3.882
3.876
3.862
3.842
2.323
2.265
2.249
2.244
2.226
2.211
2.198
2.138
2.122
2.105
2.091
2.078
2.065
2.058
2.045
2.007
1.996
1.988
1.977
1.968
1.948
1.929
3f
9
8
7
6
5
4
3
2
1
0
-1
ppm
2.07
1.04
1.01
1.03
1.02
1.08
3.05
1.00
2.02
1.08

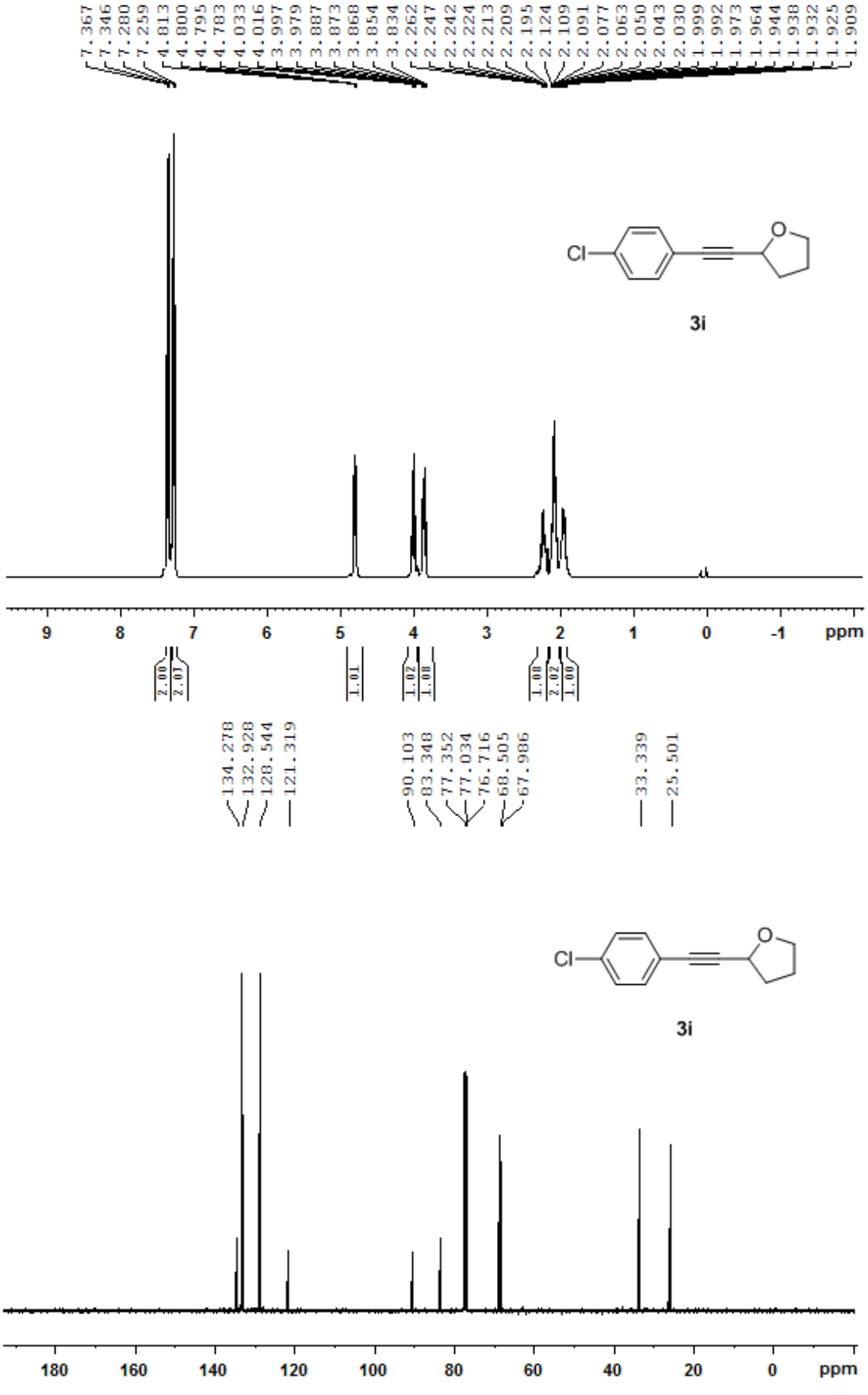

137.855
132.308
129.124
128.758
128.101
122.607
88.708
84.615
77.361
77.043
76.726
68.616
67.902
33.437
25.482
21.176
3f
180
160
140
120
100
80
60
40
20
0
ppm

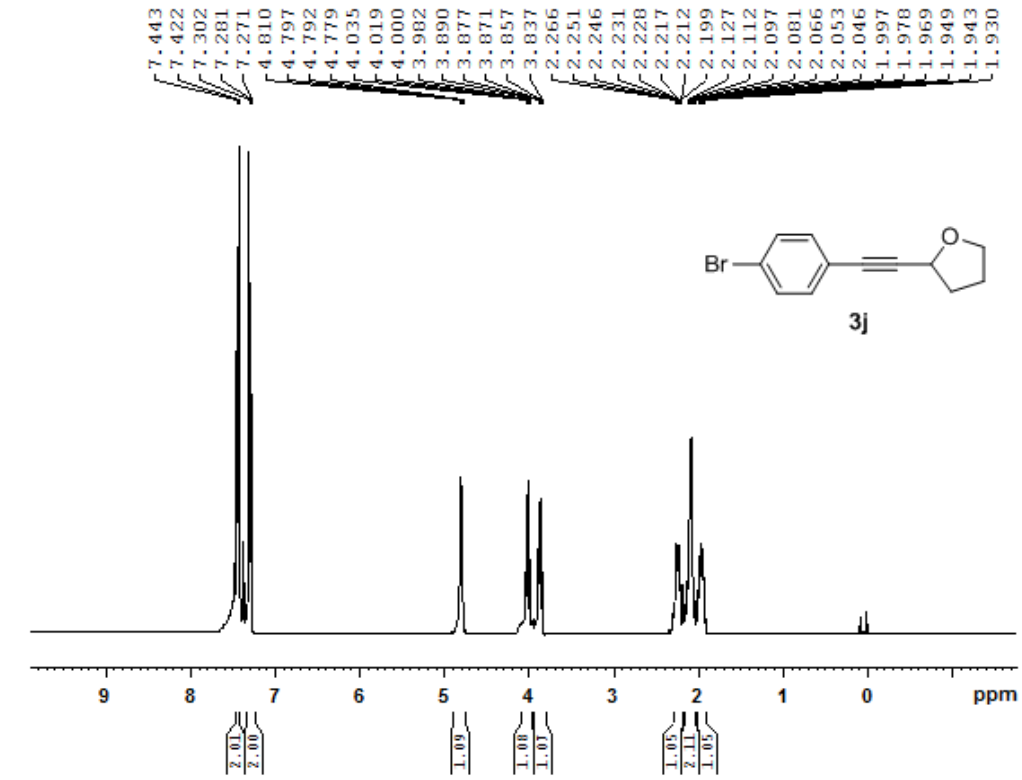

7.418
7.399
7.216
7.199
7.184
7.145
7.127
7.110
4.893
4.881
4.875
4.864
4.064
4.046
4.028
4.010
3.916
3.896
3.883
3.862
2.431
2.282
2.268
2.260
2.244
2.229
2.215
2.156
2.141
2.127
2.112
2.098
2.086
2.079
2.066
2.017
2.001
1.986
1.967
1.948
1.930
3g
9
8
7
6
5
4
3
2
1
0
ppm
1.00
2.10
1.01
1.00
1.00
1.01
3.08
1.09
2.09
1.09

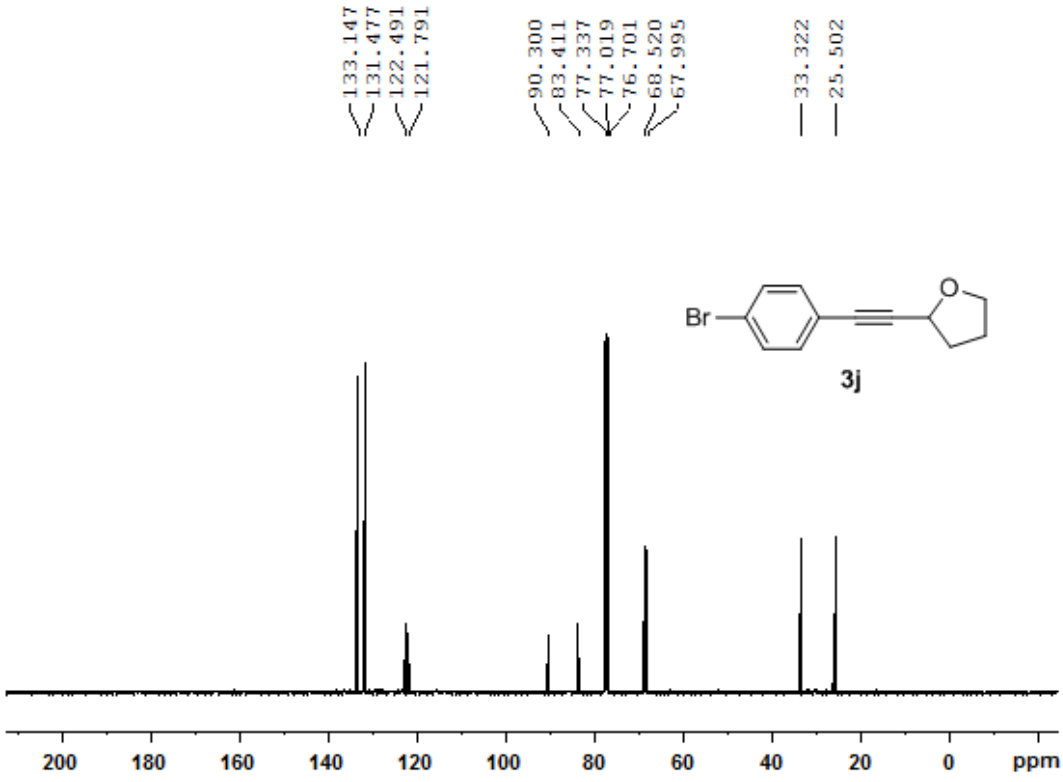

140.212
132.002
129.335
128.268
125.451
122.561
93.068
83.339
77.353
77.036
76.718
68.715
67.828
33.594
25.411
20.650
3g
180
160
140
120
100
80
60
40
20
0
ppm

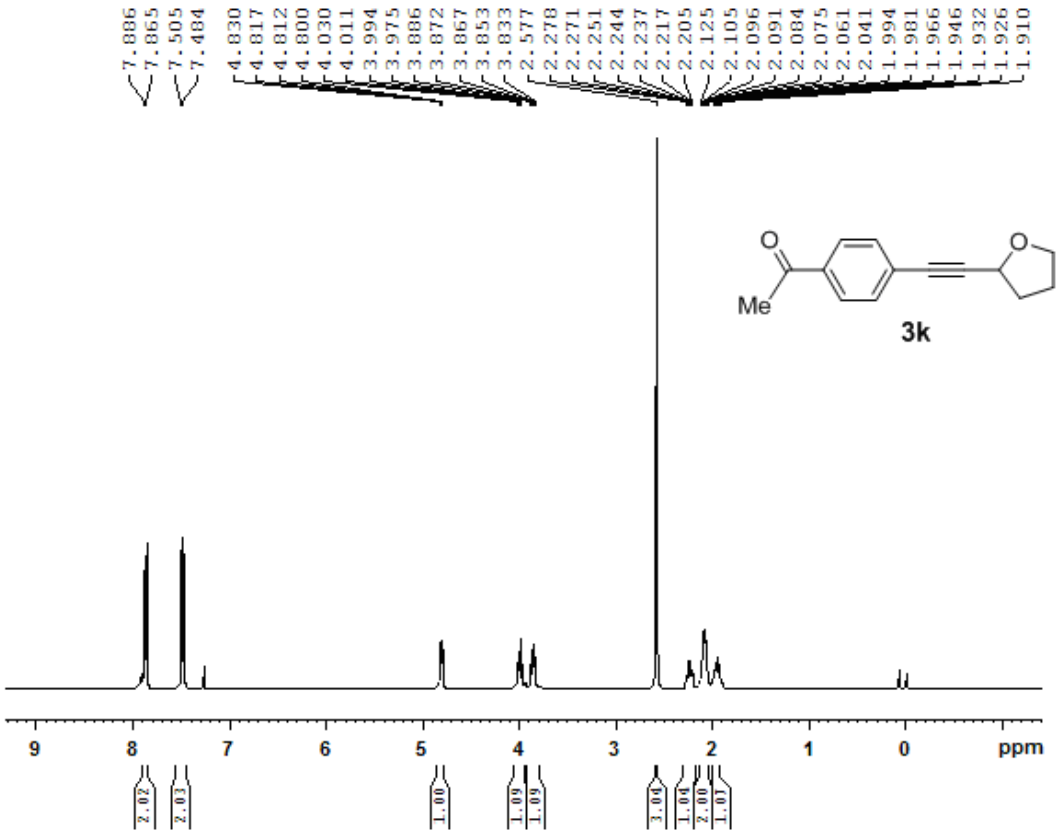

7.421
7.407
7.400
7.386
7.004
6.983
6.961
4.803
4.790
4.785
4.773
4.028
4.011
3.992
3.974
3.877
3.863
3.857
3.844
3.838
3.824
2.251
2.237
2.232
2.212
2.202
2.198
2.185
2.101
2.082
2.073
2.068
2.053
1.982
1.963
1.954
1.933
1.928
1.919
1.914
F
O
3h
9
8
7
6
5
4
3
2
1
0
ppm
2.02
2.04
1.00
1.00
1.04
1.03
2.01
1.04

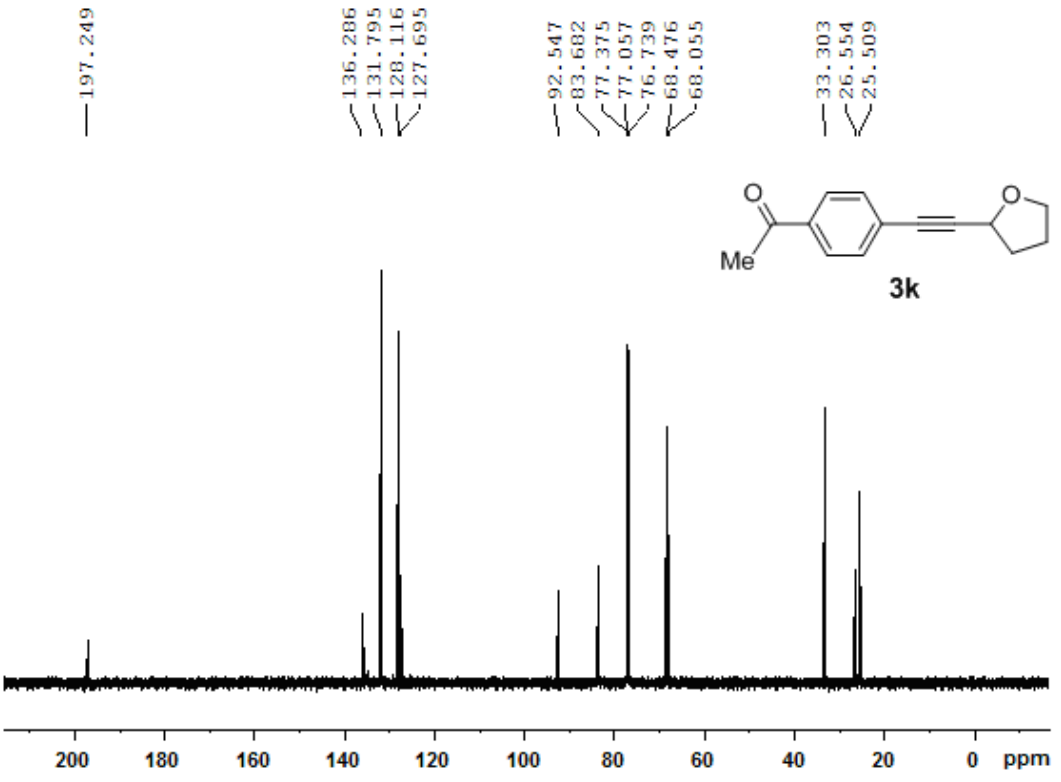

163.700
161.222
133.625
133.542
118.914
118.879
115.564
115.344
88.782
83.388
77.373
77.055
76.738
68.511
67.932
33.365
25.487
F
O
3h
180
160
140
120
100
80
60
40
20
0
ppm

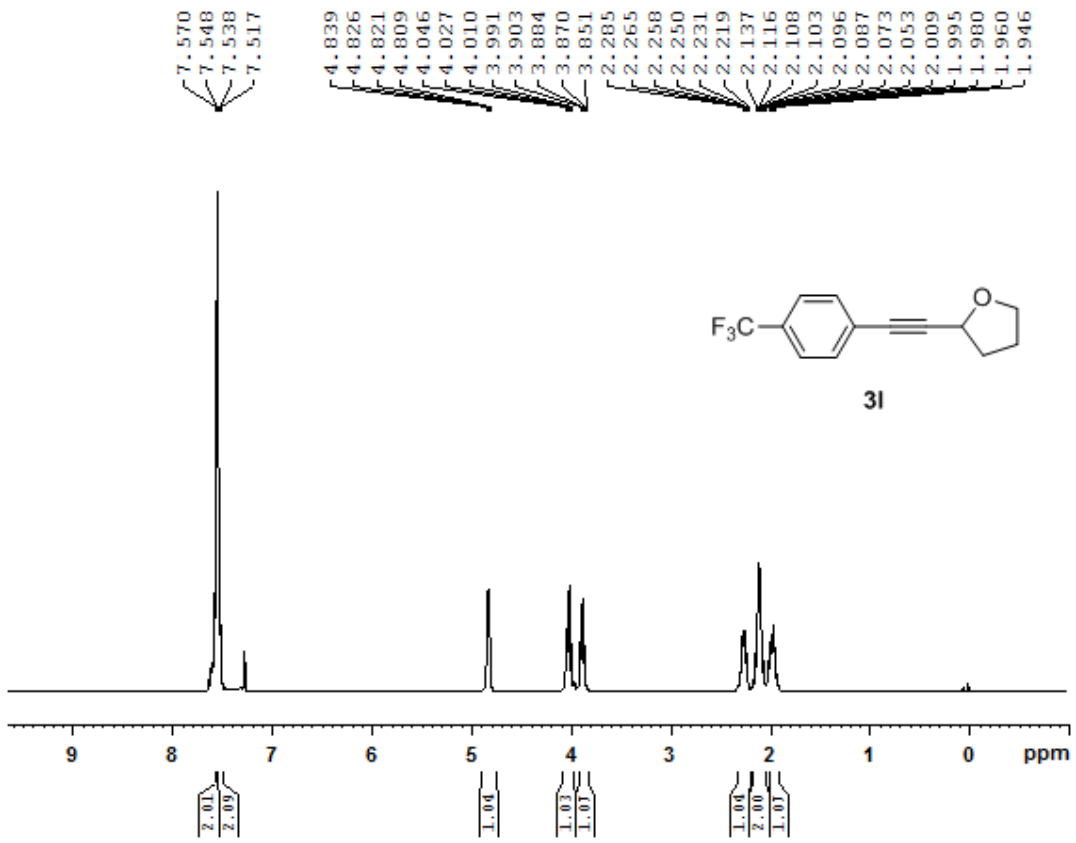

7.367
7.346
7.280
7.259
4.813
4.800
4.795
4.783
4.033
4.016
3.997
3.979
3.887
3.873
3.868
3.854
3.834
2.262
2.247
2.242
2.224
2.213
2.209
2.195
2.124
2.109
2.091
2.077
2.063
2.050
2.043
2.030
1.999
1.992
1.973
1.964
1.944
1.938
1.932
1.925
1.909
Cl
O
3i
9
8
7
6
5
4
3
2
1
0
-1
ppm
2.00
2.07
1.01
1.02
1.08
1.08
2.02
1.00
134.278
132.928
128.544
121.319
90.103
83.348
77.352
77.034
76.716
68.505
67.986
33.339
25.501
Cl
O
3i
180
160
140
120
100
80
60
40
20
0
ppm

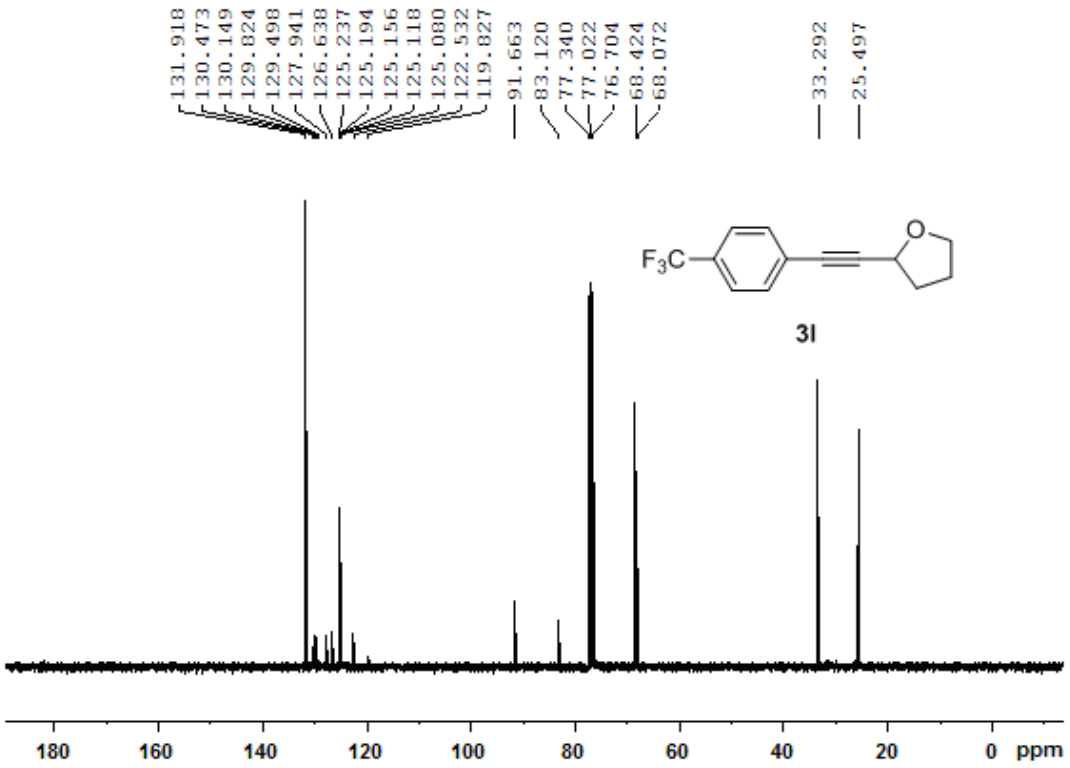

7.443
7.422
7.302
7.281
7.271
4.810
4.797
4.792
4.779
4.035
4.019
4.000
3.982
3.890
3.877
3.871
3.857
3.837
2.266
2.251
2.246
2.231
2.228
2.217
2.212
2.199
2.127
2.112
2.097
2.081
2.066
2.053
2.046
1.997
1.978
1.969
1.949
1.943
1.930
Br
O
3j
9
8
7
6
5
4
3
2
1
0
ppm
2.01
2.00
1.09
1.08
1.07
1.05
2.11
1.05

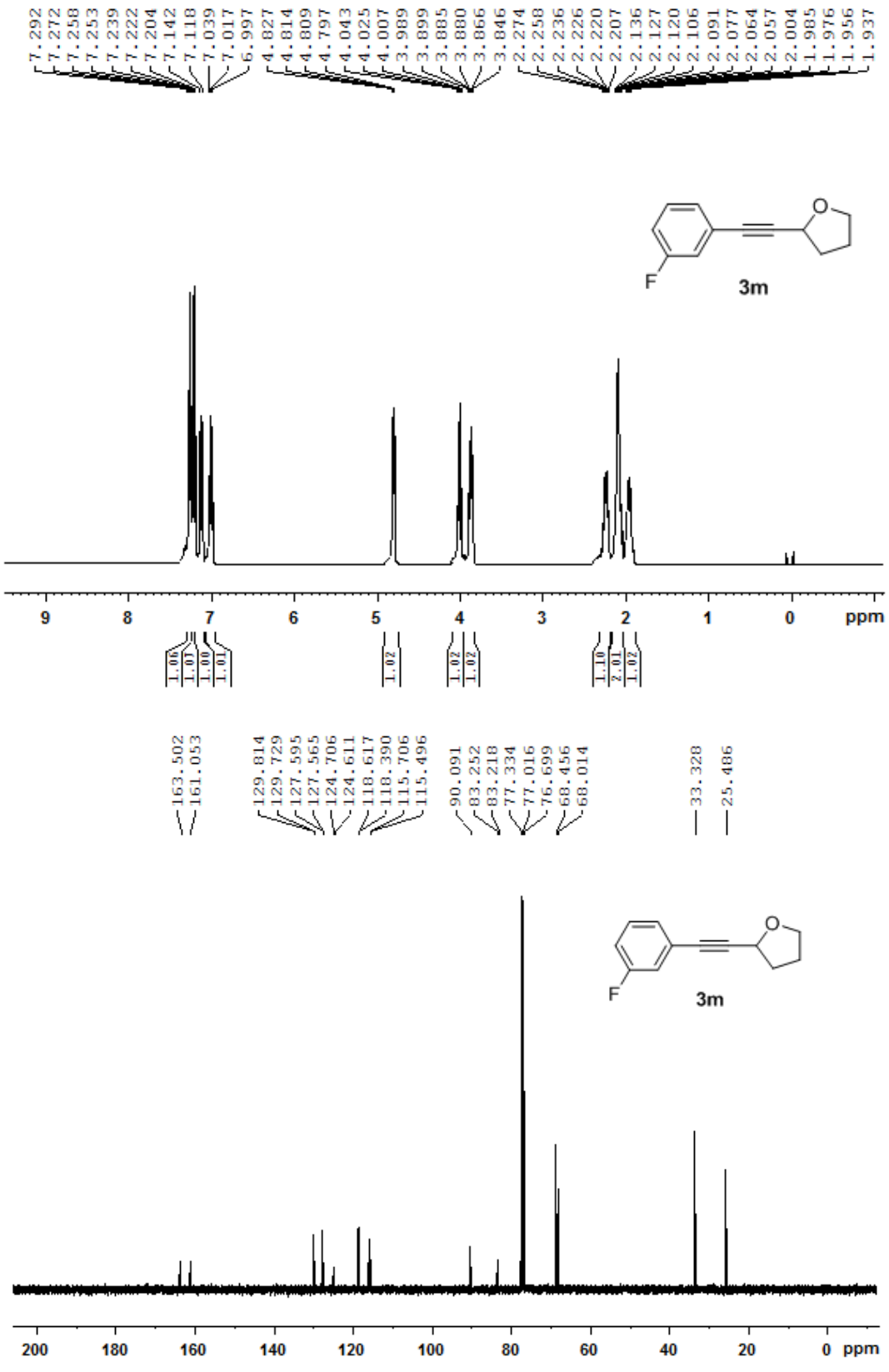

133.147
131.477
122.491
121.791
90.300
83.411
77.337
77.019
76.701
68.520
67.995
33.322
25.502
Br
O
3j
200
180
160
140
120
100
80
60
40
20
0
ppm

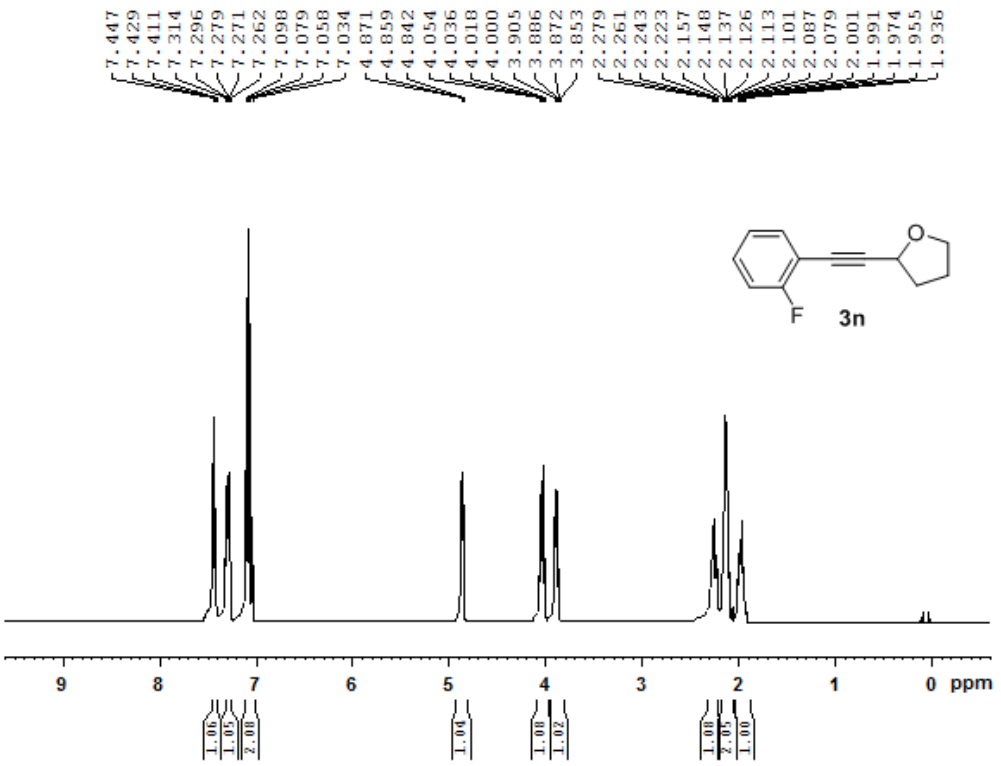

7.886
7.865
7.505
7.484
4.830
4.817
4.812
4.800
4.030
4.011
3.994
3.975
3.886
3.872
3.867
3.853
3.833
2.577
2.278
2.271
2.251
2.244
2.237
2.217
2.205
2.125
2.105
2.096
2.091
2.084
2.075
2.061
2.041
1.994
1.981
1.966
1.946
1.932
1.926
1.910
O
O
Me
3k
9
8
7
6
5
4
3
2
1
0
ppm
2.02
2.03
1.00
1.09
1.09
3.04
1.04
2.00
1.01

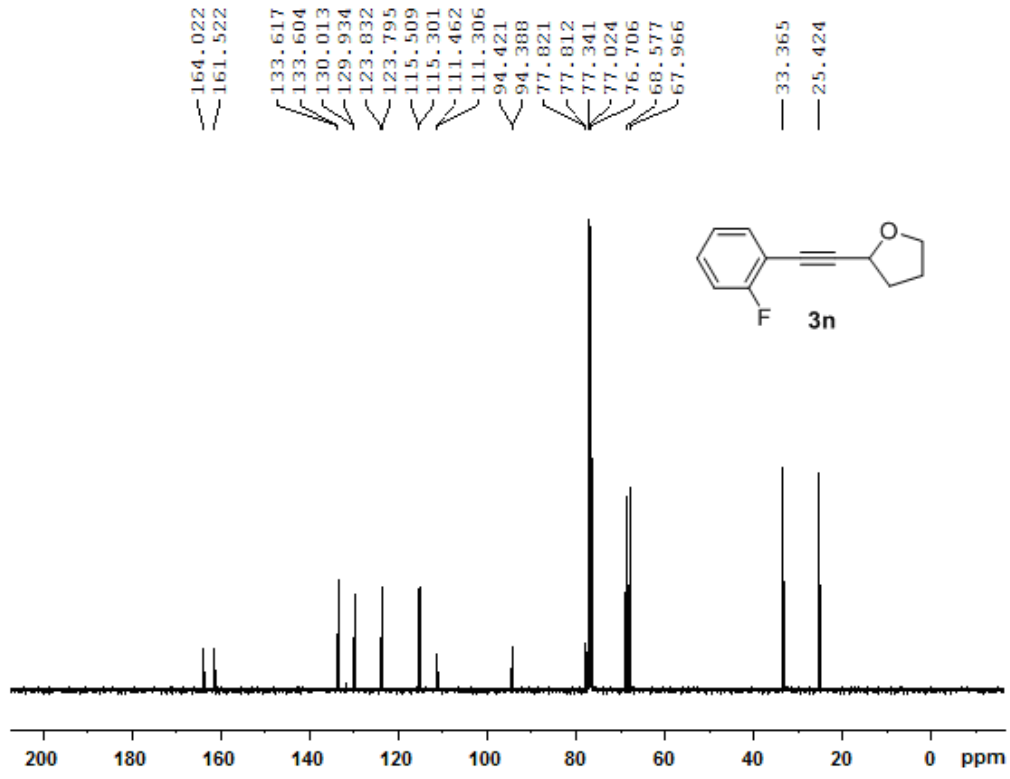

197.249
136.286
131.795
128.116
127.695
92.547
83.682
77.375
77.057
76.739
68.476
68.055
33.303
26.554
25.509
O
O
Me
3k
200
180
160
140
120
100
80
60
40
20
0
ppm

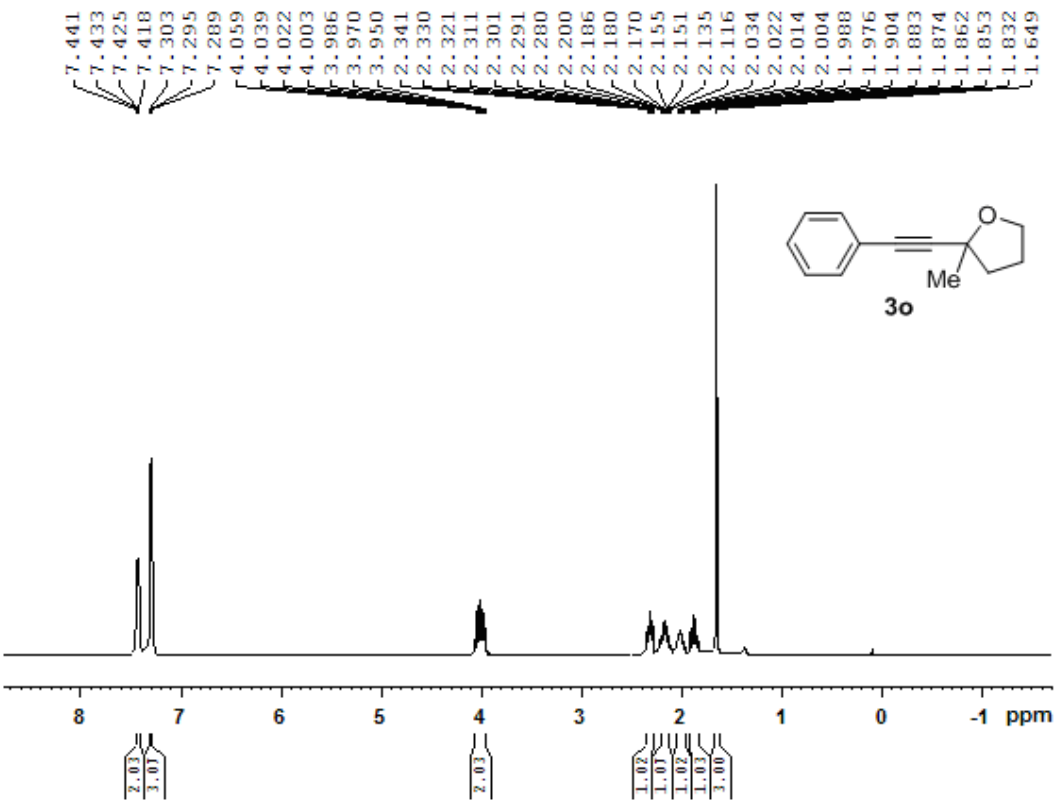

7.570
7.548
7.538
7.517
4.839
4.826
4.821
4.809
4.046
4.027
4.010
3.991
3.903
3.884
3.870
3.851
2.285
2.265
2.258
2.250
2.231
2.219
2.137
2.116
2.108
2.103
2.096
2.087
2.073
2.053
2.009
1.995
1.980
1.960
1.946
F3C
O
3l
9
8
7
6
5
4
3
2
1
0
ppm
2.01
2.09
1.04
1.03
1.07
1.04
2.00
1.07

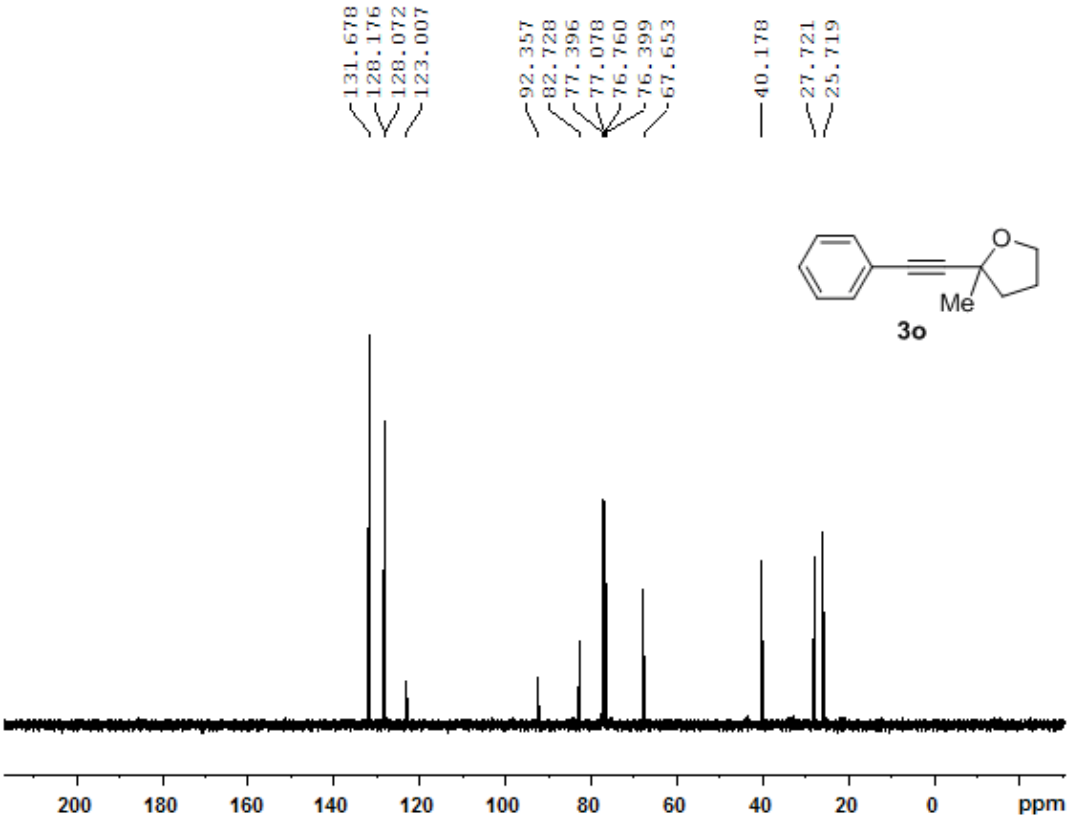

131.918
130.473
130.149
129.824
129.498
127.941
126.638
125.237
125.194
125.156
125.118
125.080
122.532
119.827
91.663
83.120
77.340
77.022
76.704
68.424
68.072
33.292
25.497
F3C
O
3l
180
160
140
120
100
80
60
40
20
0
ppm

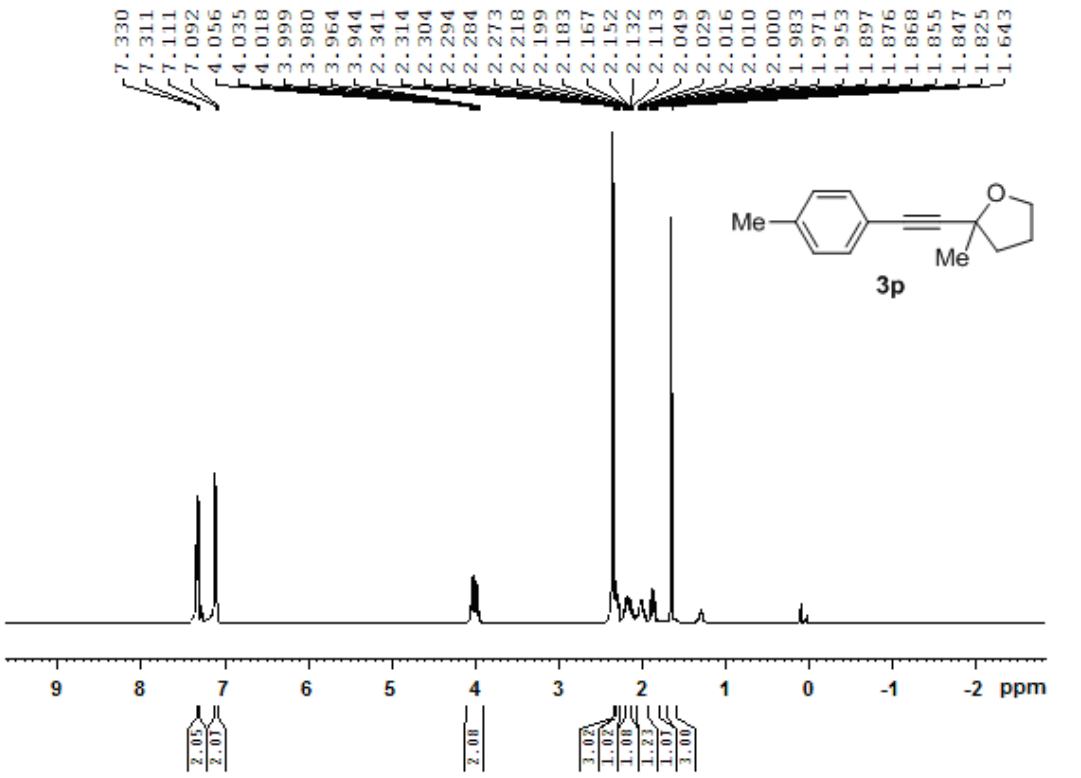

7.292
7.272
7.258
7.253
7.239
7.222
7.204
7.142
7.118
7.039
7.017
6.997
4.827
4.814
4.809
4.797
4.043
4.025
4.007
3.989
3.899
3.885
3.880
3.866
3.846
2.274
2.258
2.236
2.226
2.220
2.207
2.136
2.127
2.120
2.106
2.091
2.077
2.064
2.057
2.004
1.985
1.976
1.956
1.937
F
O
3m
9
8
7
6
5
4
3
2
1
0
ppm
1.06
1.07
1.00
1.01
1.02
1.02
1.02
1.10
2.01
1.02
163.502
161.053
129.814
129.729
127.595
127.565
124.706
124.611
118.617
118.390
115.706
115.496
90.091
83.252
83.218
77.334
77.016
76.699
68.456
68.014
33.328
25.486
F
O
3m
200
180
160
140
120
100
80
60
40
20
0
ppm

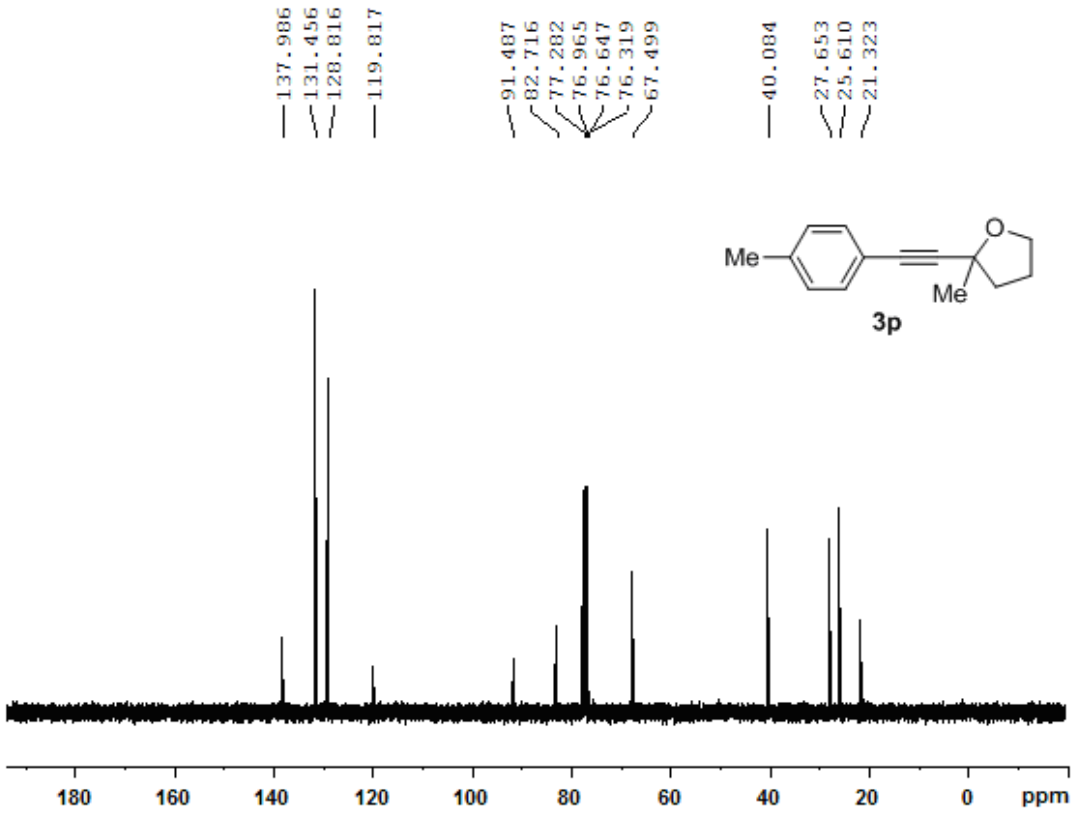

7.447
7.429
7.411
7.314
7.296
7.279
7.271
7.262
7.098
7.079
7.058
7.034
4.871
4.859
4.842
4.054
4.036
4.018
4.000
3.905
3.886
3.872
3.853
2.279
2.261
2.243
2.223
2.157
2.148
2.137
2.126
2.113
2.101
2.087
2.079
2.001
1.991
1.974
1.955
1.936
O
F
3n
9
8
7
6
5
4
3
2
1
0 ppm
1.06
1.05
2.08
1.04
1.08
1.02
1.08
2.05
1.00

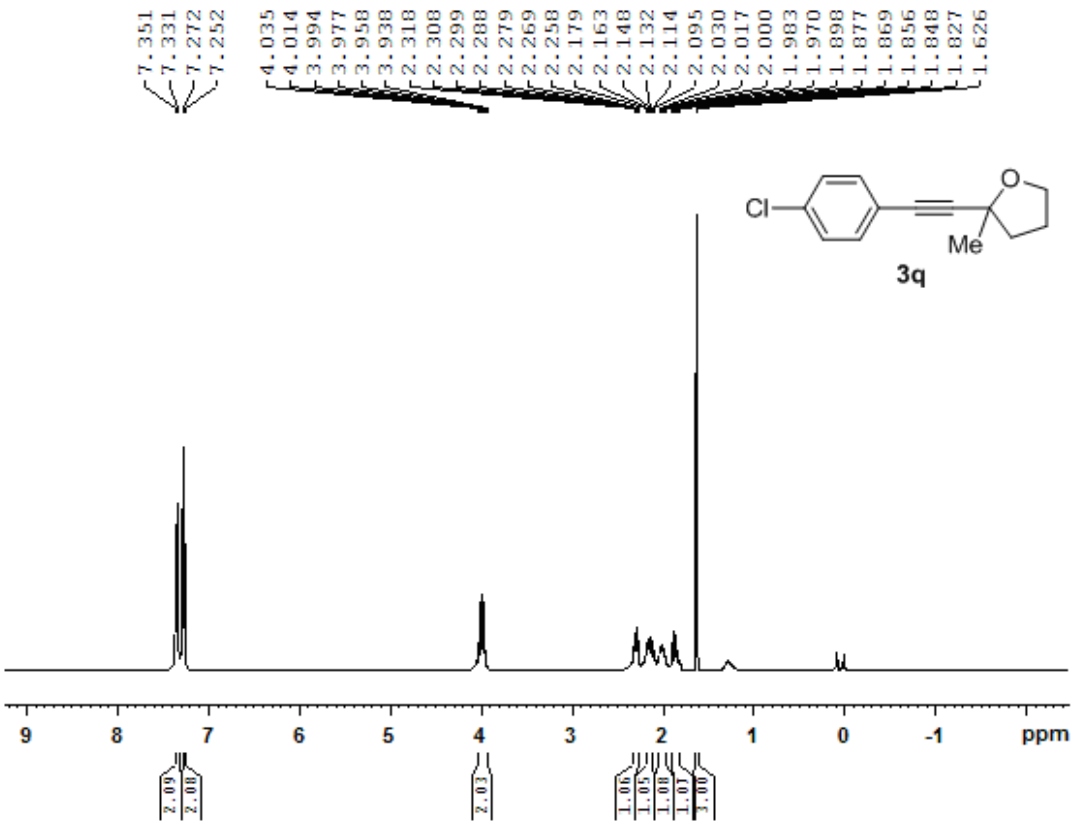

164.022
161.522
133.617
133.604
130.013
129.934
123.832
123.795
115.509
115.301
111.462
111.306
94.421
94.388
77.821
77.812
77.341
77.024
76.706
68.577
67.966
33.365
25.424
O
F
3n
200
180
160
140
120
100
80
60
40
20
0 ppm

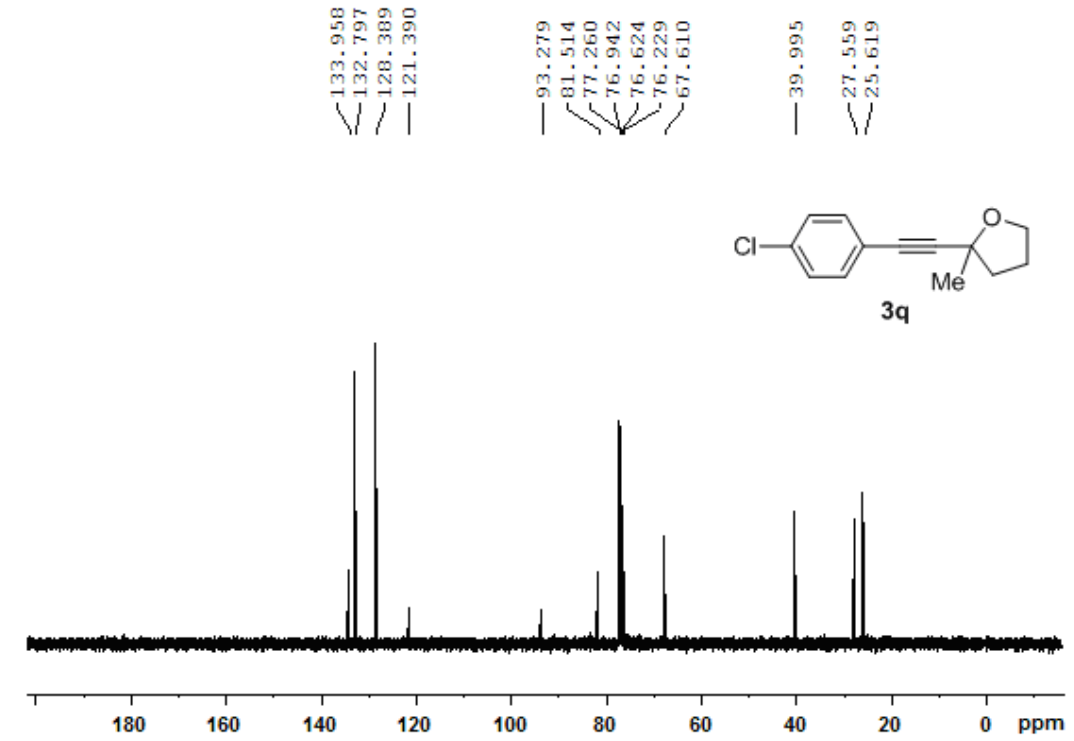

7.441
7.433
7.425
7.418
7.303
7.295
7.289
4.059
4.039
4.022
4.003
3.986
3.970
3.950
2.341
2.330
2.321
2.311
2.301
2.291
2.280
2.200
2.186
2.180
2.170
2.155
2.151
2.135
2.116
2.034
2.022
2.014
2.004
1.988
1.976
1.904
1.883
1.874
1.862
1.853
1.832
1.649
O
Me
3o
8
7
6
5
4
3
2
1
0
-1
ppm
2.03
3.01
2.03
1.02
1.01
1.02
1.03
3.00

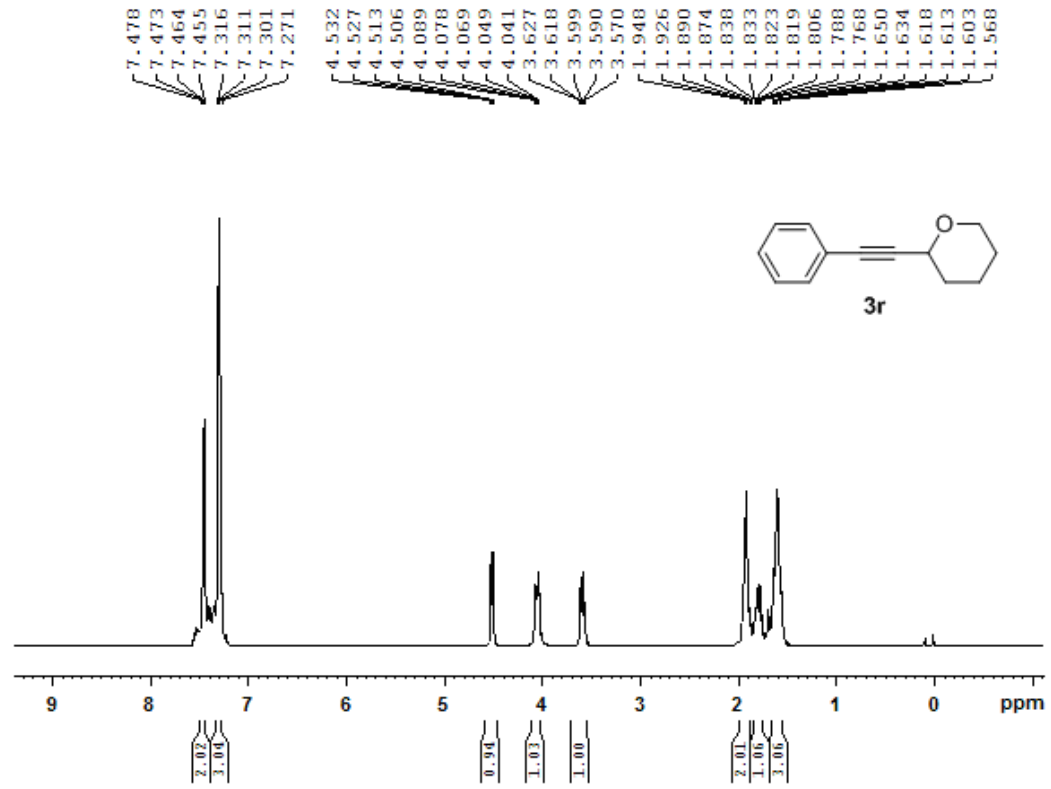

131.678
128.176
128.072
123.007
92.357
82.728
77.396
77.078
76.760
76.399
67.653
40.178
27.721
25.719
O
Me
3o
200
180
160
140
120
100
80
60
40
20
0
ppm

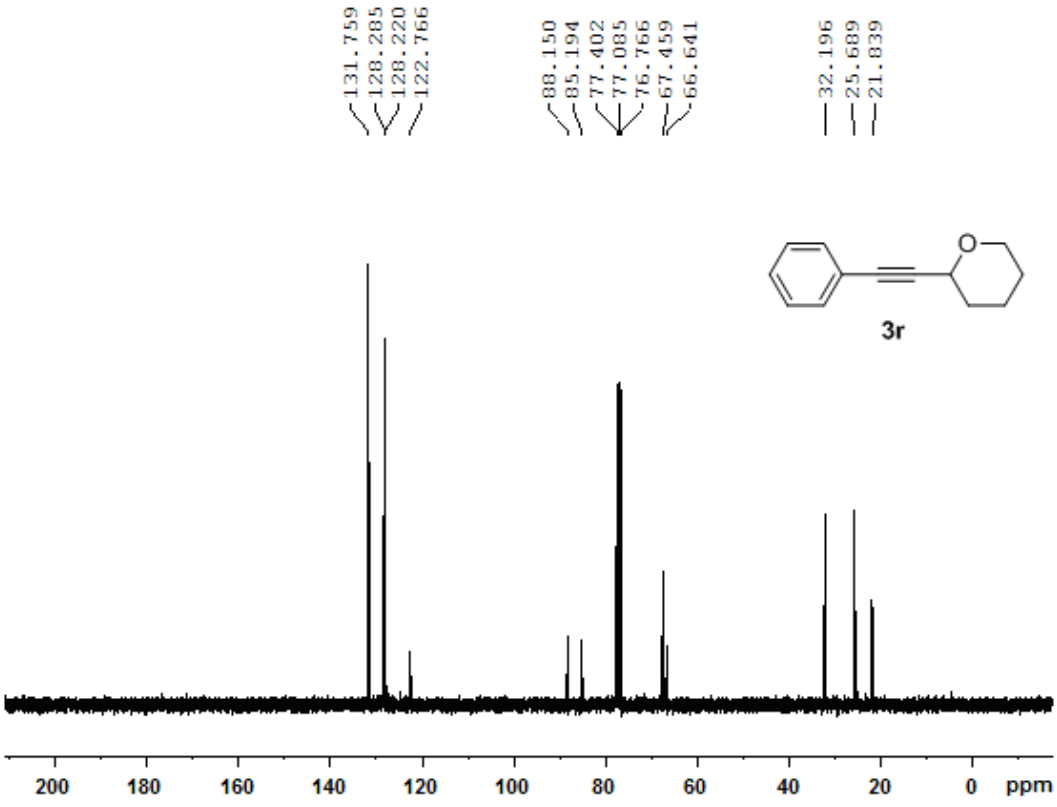

7.330
7.311
7.111
7.092
4.056
4.035
4.018
3.999
3.980
3.964
3.944
2.341
2.314
2.304
2.294
2.284
2.273
2.218
2.199
2.183
2.167
2.152
2.132
2.113
2.049
2.029
2.016
2.010
2.000
1.983
1.971
1.953
1.897
1.876
1.868
1.855
1.847
1.825
1.643
Me
O
Me
3p
9
8
7
6
5
4
3
2
1
0
-1
-2
ppm
2.05
2.07
2.08
3.02
1.02
1.08
1.23
1.07
3.00

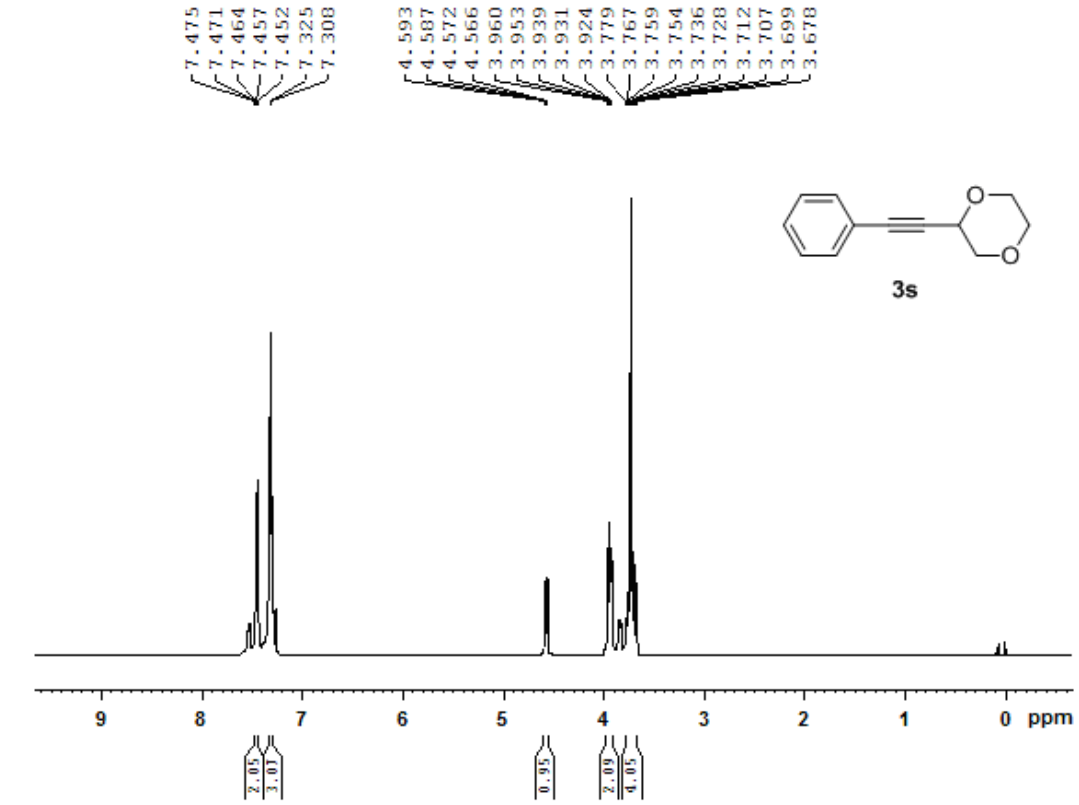

137.986
131.456
128.816
119.817
91.487
82.716
77.282
76.965
76.647
76.319
67.499
40.084
27.653
25.610
21.323
Me
O
Me
3p
180
160
140
120
100
80
60
40
20
0
ppm

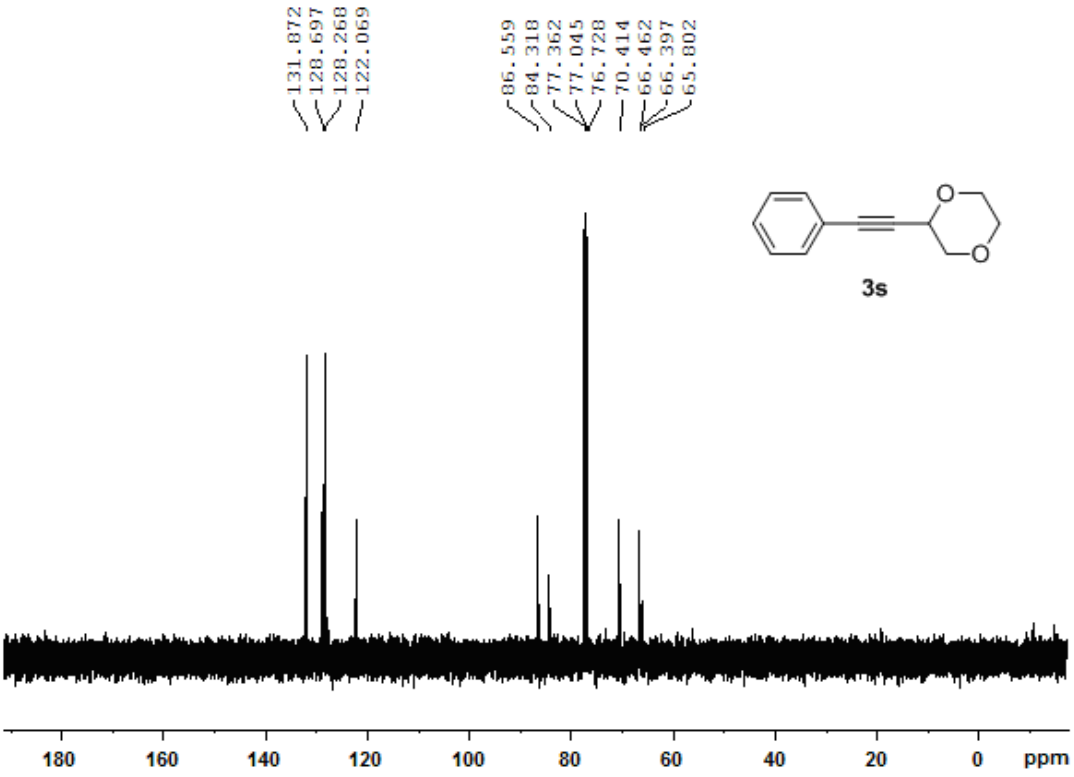

7.351
7.331
7.272
7.252
4.035
4.014
3.994
3.977
3.958
3.938
2.318
2.308
2.299
2.288
2.279
2.269
2.258
2.179
2.163
2.148
2.132
2.114
2.095
2.030
2.017
2.000
1.983
1.970
1.898
1.877
1.869
1.856
1.848
1.827
1.626
Cl
O
Me
3q
9
8
7
6
5
4
3
2
1
0
-1
ppm
2.09
2.08
2.03
1.06
1.05
1.08
1.07
3.00

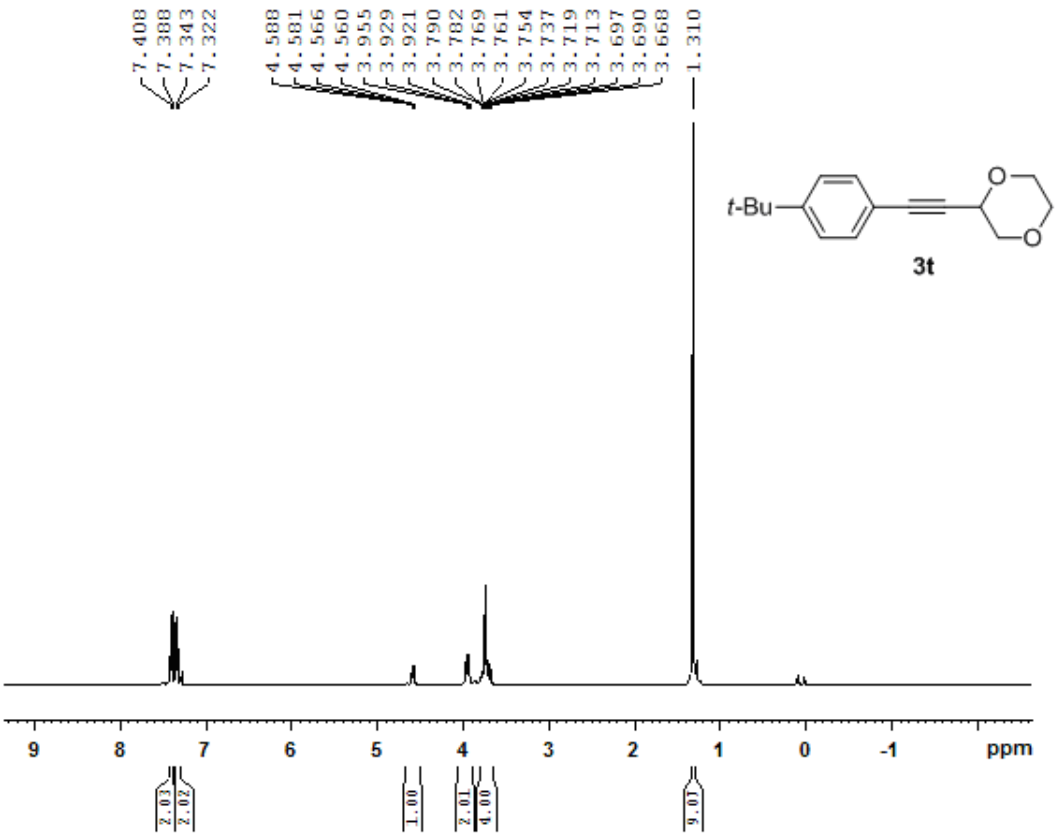

133.958
132.797
128.389
121.390
93.279
81.514
77.260
76.942
76.624
76.229
67.610
39.995
27.559
25.619
Cl
O
Me
3q
180
160
140
120
100
80
60
40
20
0
ppm

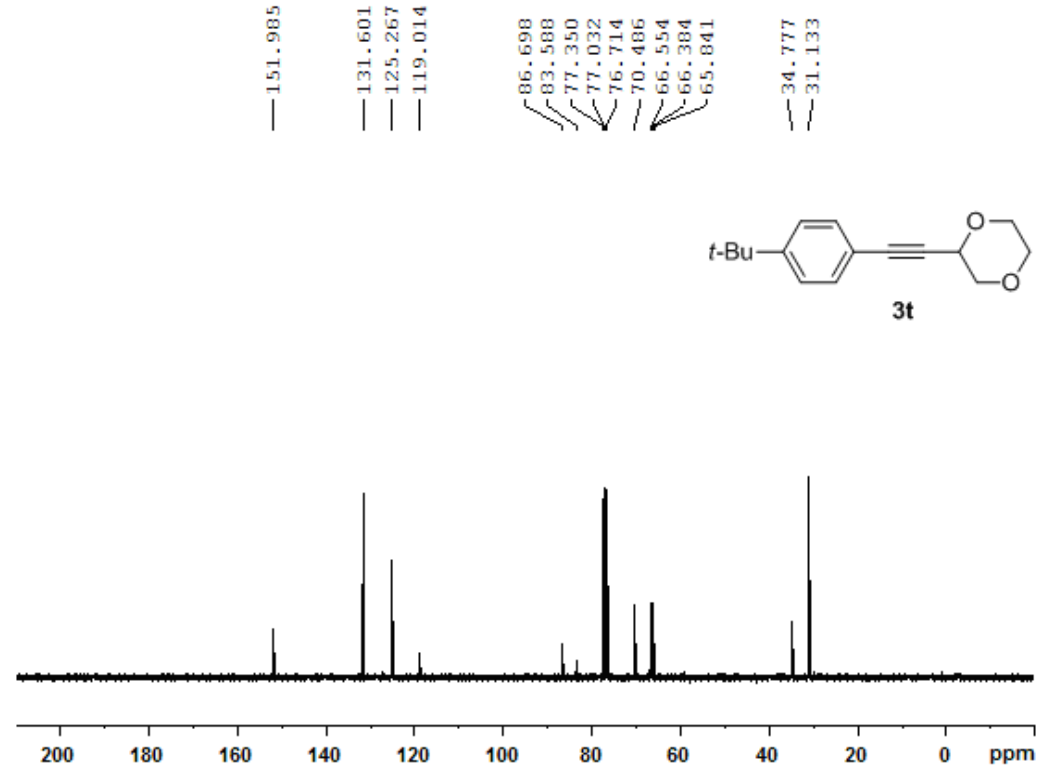

7.478
7.473
7.464
7.455
7.316
7.311
7.301
7.271
4.532
4.527
4.513
4.506
4.089
4.078
4.069
4.049
4.041
3.627
3.618
3.599
3.590
3.570
1.948
1.926
1.890
1.874
1.838
1.833
1.823
1.819
1.806
1.788
1.768
1.650
1.634
1.618
1.613
1.603
1.568
O
3r
9
8
7
6
5
4
3
2
1
0
ppm
2.02
3.04
0.94
1.03
1.00
2.01
1.06
3.06

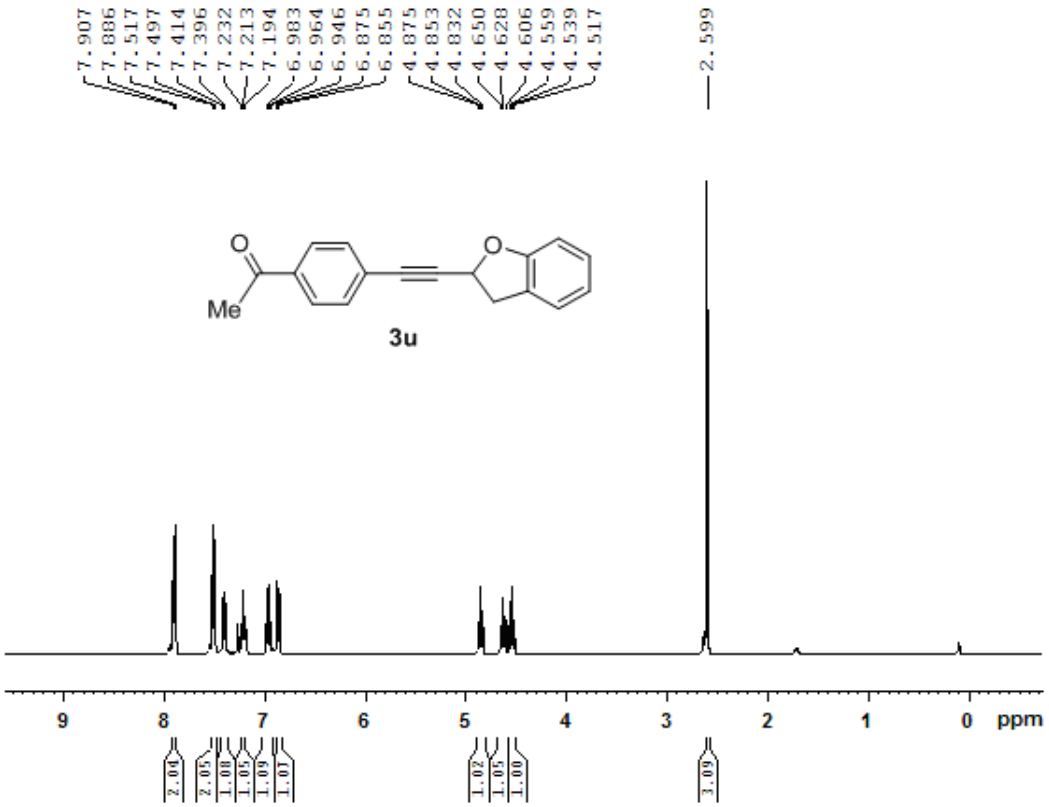

131.759
128.285
128.220
122.766
88.150
85.194
77.402
77.085
76.766
67.459
66.641
32.196
25.689
21.839
O
3r
200
180
160
140
120
100
80
60
40
20
0
ppm

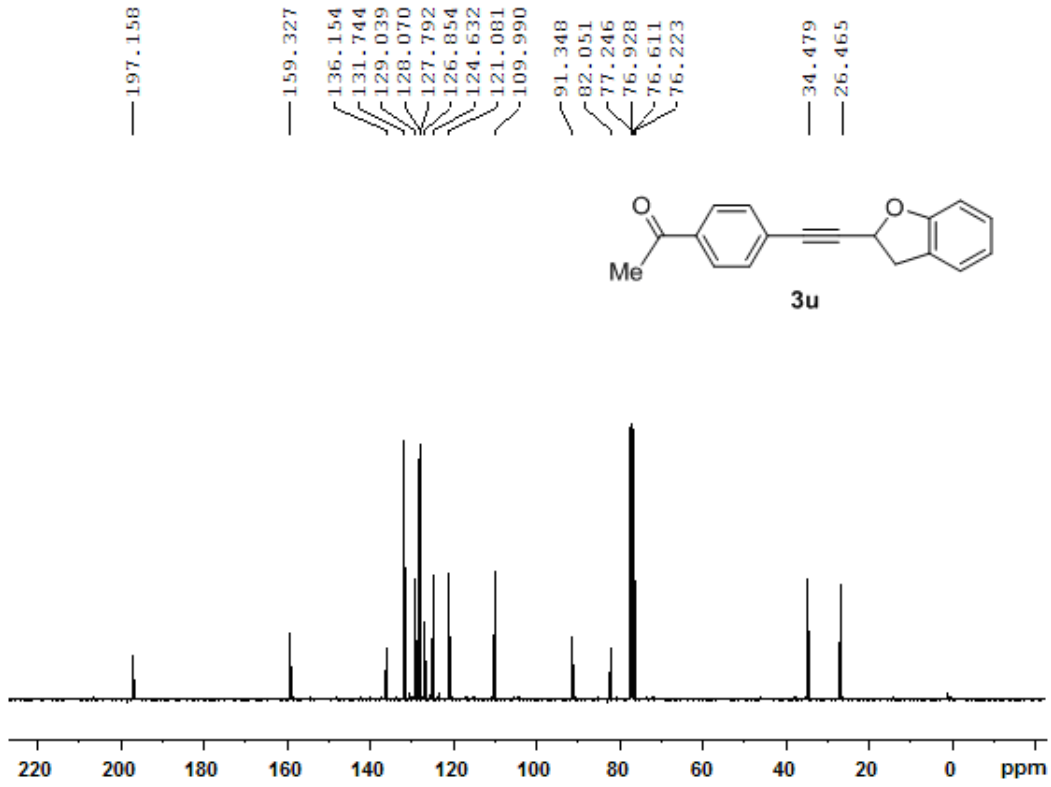

7.475
7.471
7.464
7.457
7.452
7.325
7.308
4.593
4.587
4.572
4.566
3.960
3.953
3.939
3.931
3.924
3.779
3.767
3.759
3.754
3.736
3.728
3.712
3.707
3.699
3.678
3s
9
8
7
6
5
4
3
2
1
0
ppm
2.05
3.07
0.95
2.09
4.05

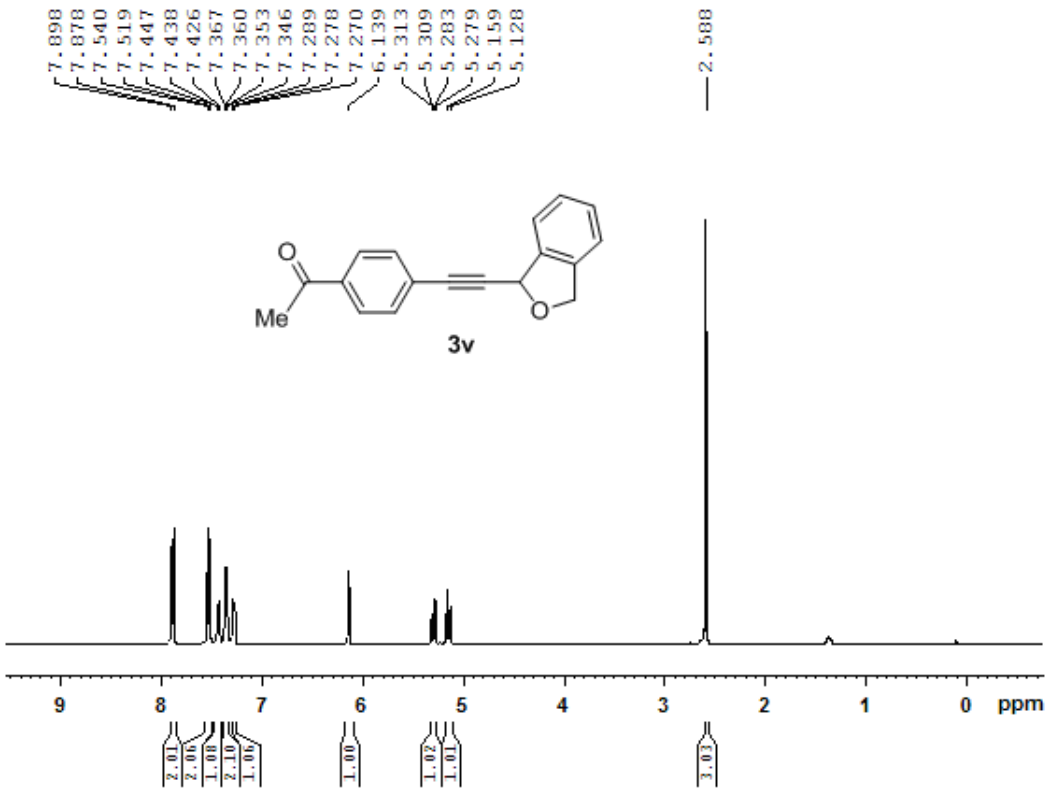

131.872
128.697
128.268
122.069
86.559
84.318
77.362
77.045
76.728
70.414
66.462
66.397
65.802
3s
180
160
140
120
100
80
60
40
20
0
ppm

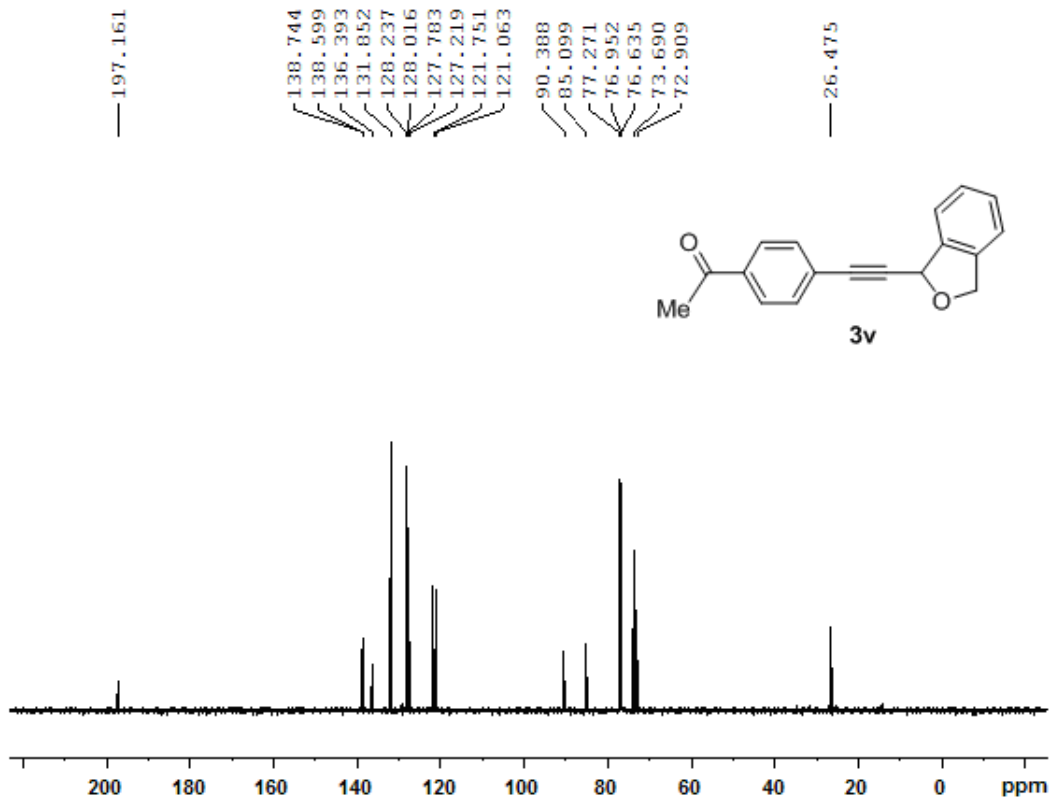

7.408
7.388
7.343
7.322
4.588
4.581
4.566
4.560
3.955
3.929
3.921
3.790
3.782
3.769
3.761
3.754
3.737
3.719
3.713
3.697
3.690
3.668
1.310
t-Bu
3t
9
8
7
6
5
4
3
2
1
0
-1
ppm
2.03
2.02
1.00
2.01
4.00
9.01

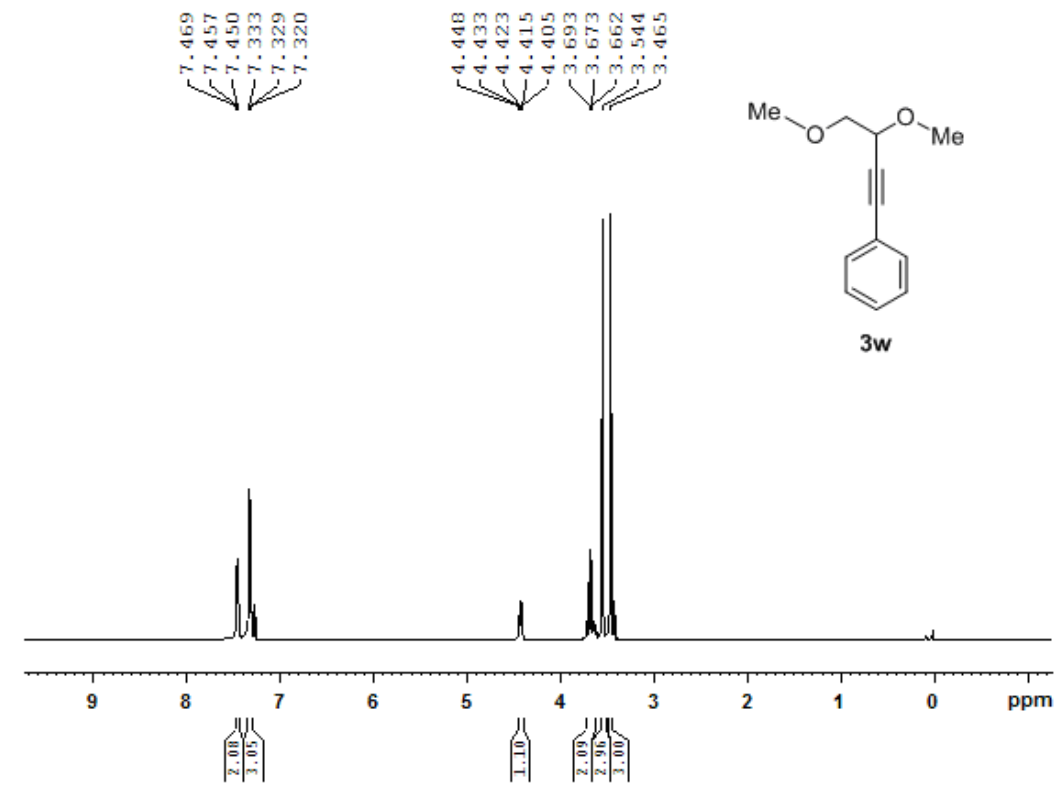

151.985
131.601
125.267
119.014
86.698
83.588
77.350
77.032
76.714
70.486
66.554
66.384
65.841
34.777
31.133
t-Bu
3t
200
180
160
140
120
100
80
60
40
20
0
ppm

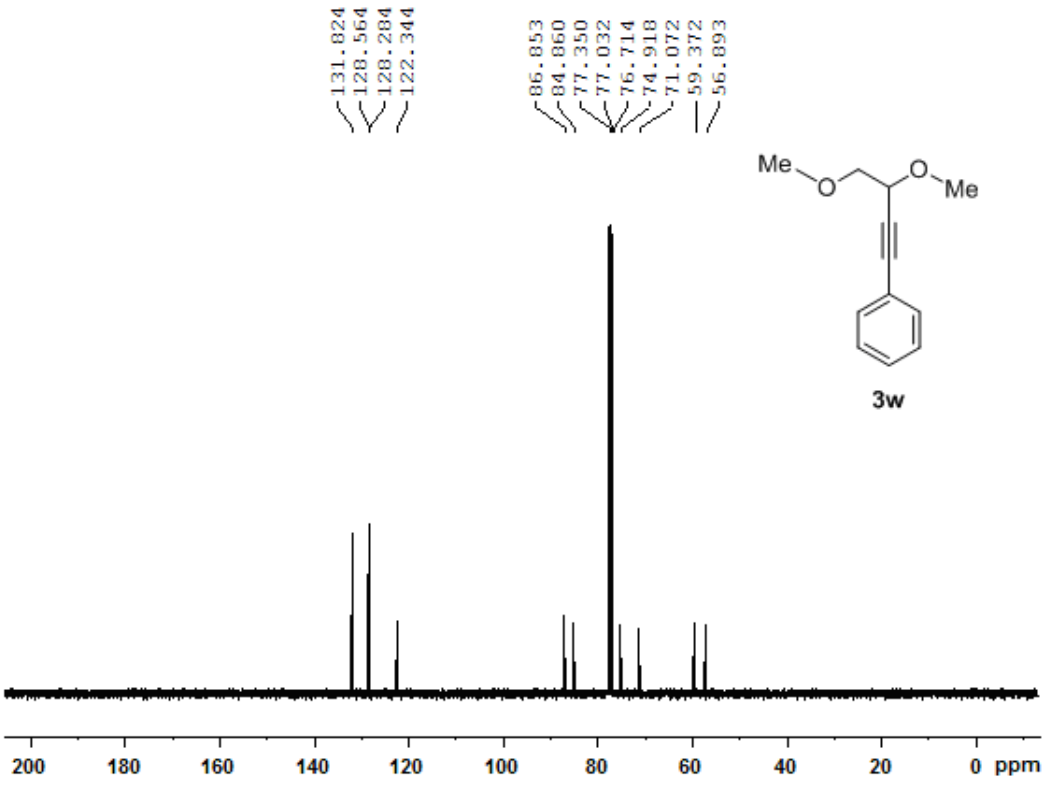

7.907
7.886
7.517
7.497
7.414
7.396
7.232
7.213
7.194
6.983
6.964
6.946
6.875
6.855
4.875
4.853
4.832
4.650
4.628
4.606
4.559
4.539
4.517
2.599
O
O
Me
3u
9
8
7
6
5
4
3
2
1
0
ppm
2.04
2.05
1.08
1.05
1.09
1.07
1.02
1.05
1.00
3.09

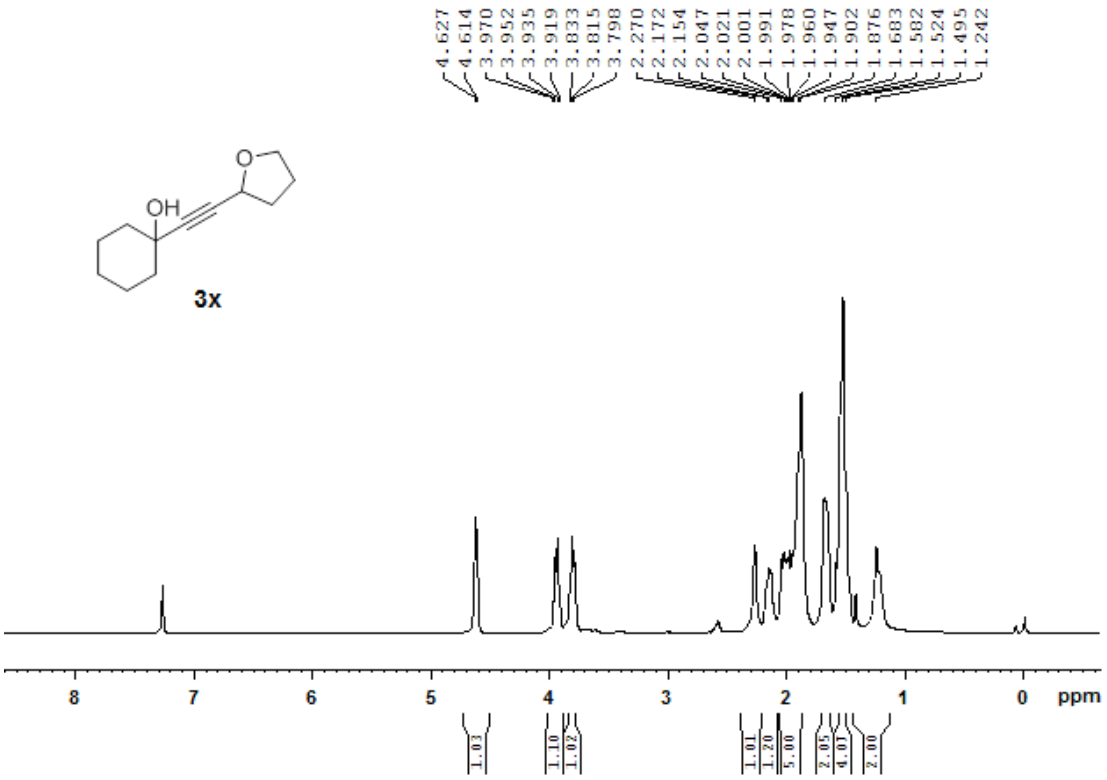

197.158
159.327
136.154
131.744
129.039
128.070
127.792
126.854
124.632
121.081
109.990
91.348
82.051
77.246
76.928
76.611
76.223
34.479
26.465
O
O
Me
3u
220
200
180
160
140
120
100
80
60
40
20
0
ppm

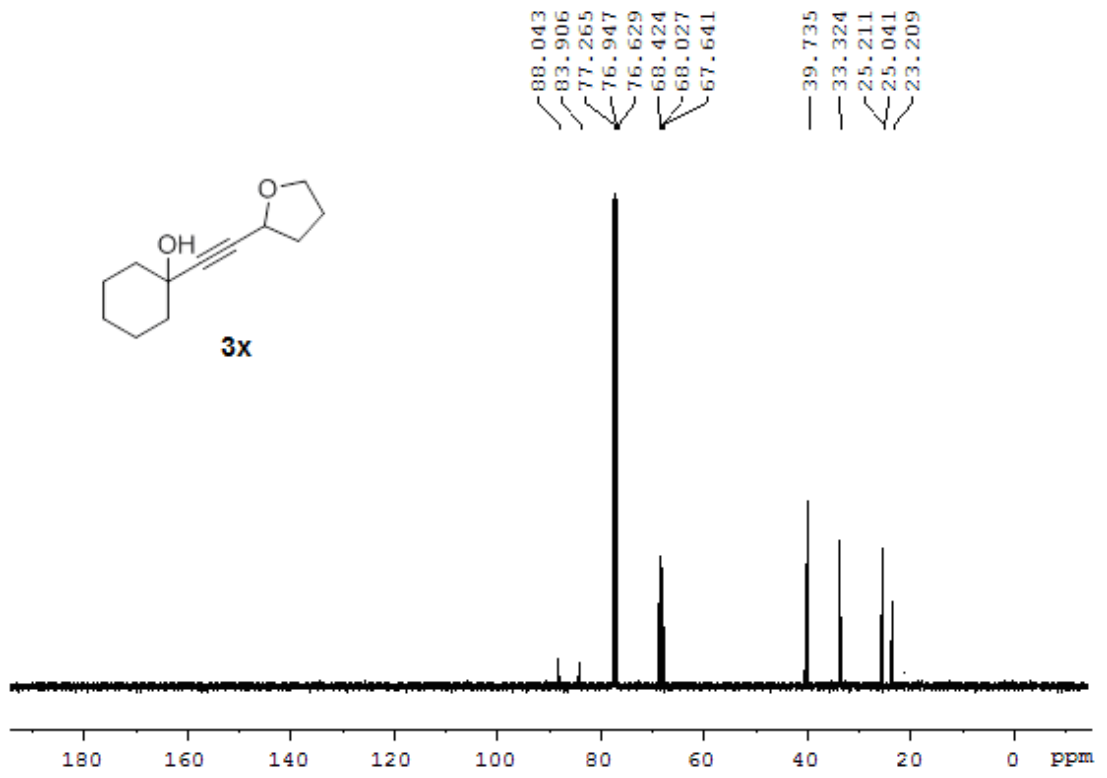

7.898
7.878
7.540
7.519
7.447
7.438
7.426
7.367
7.360
7.353
7.346
7.289
7.278
7.270
6.139
5.313
5.309
5.283
5.279
5.159
5.128
2.588
O
Me
O
3v
9
8
7
6
5
4
3
2
1
0
ppm
2.01
2.01
1.08
2.10
1.06
1.00
1.02
1.01
3.03

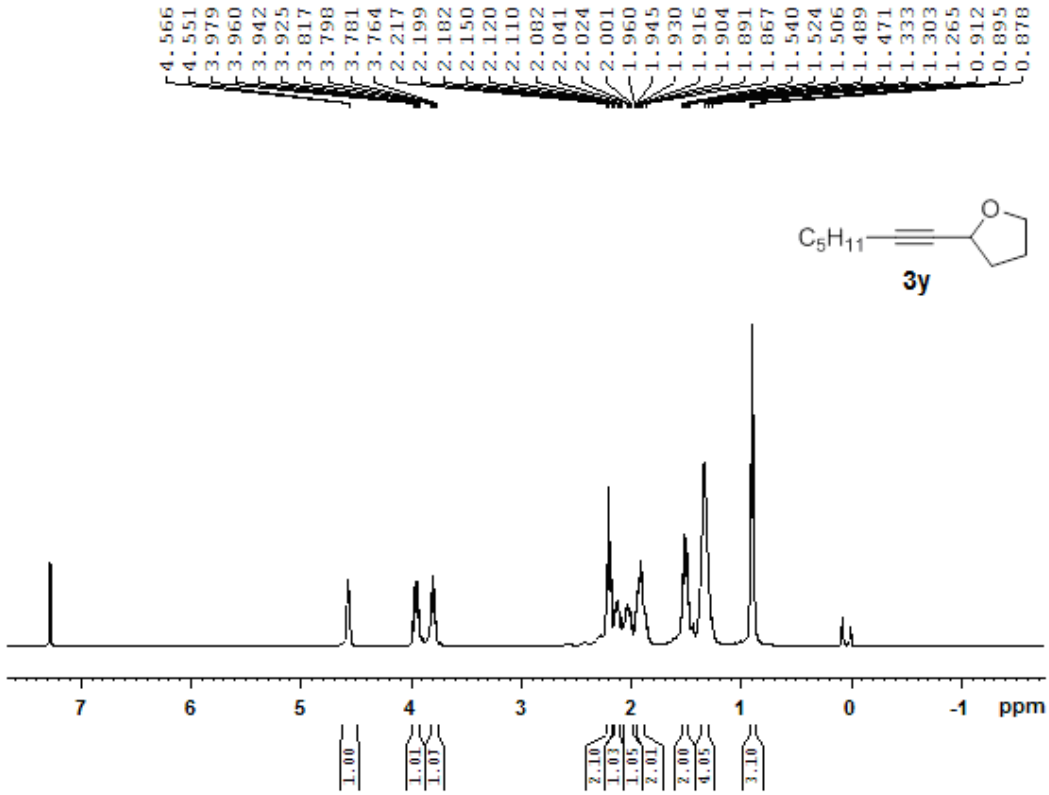

197.161
138.744
138.599
136.393
131.852
128.237
128.016
127.783
127.219
121.751
121.063
90.388
85.099
77.271
76.952
76.635
73.690
72.909
26.475
O
Me
O
3v
200
180
160
140
120
100
80
60
40
20
0
ppm

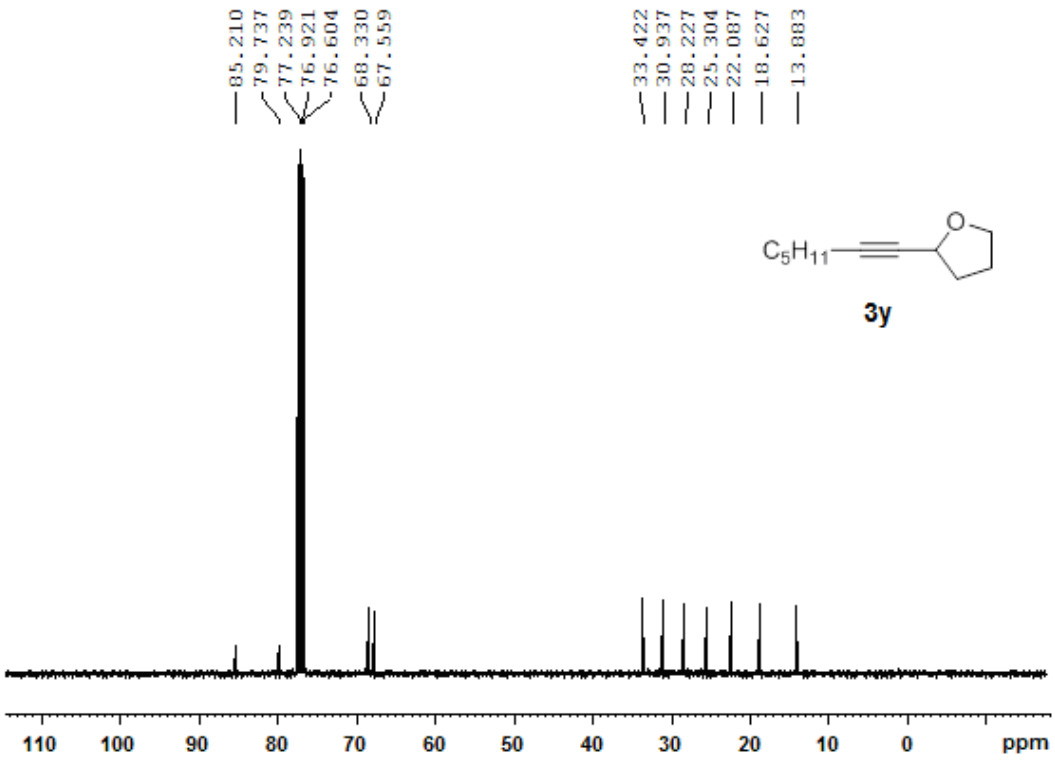

7.469
7.457
7.450
7.333
7.329
7.320
4.448
4.433
4.423
4.415
4.405
3.693
3.673
3.662
3.544
3.465
Me
O
O
Me
3w
9
8
7
6
5
4
3
2
1
0
ppm
2.08
3.05
1.10
2.09
2.96
3.00

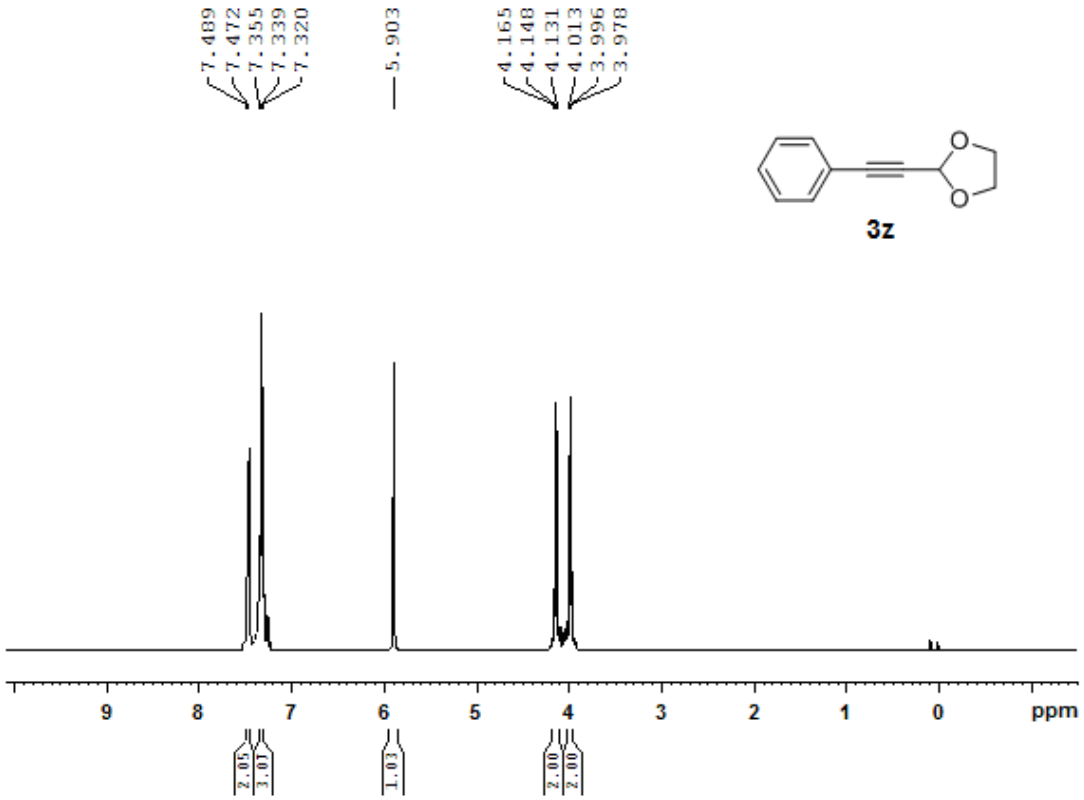

131.824
128.564
128.284
122.344
86.853
84.860
77.350
77.032
76.714
74.918
71.072
59.372
56.893
Me
O
O
Me
3w
200
180
160
140
120
100
80
60
40
20
0
ppm

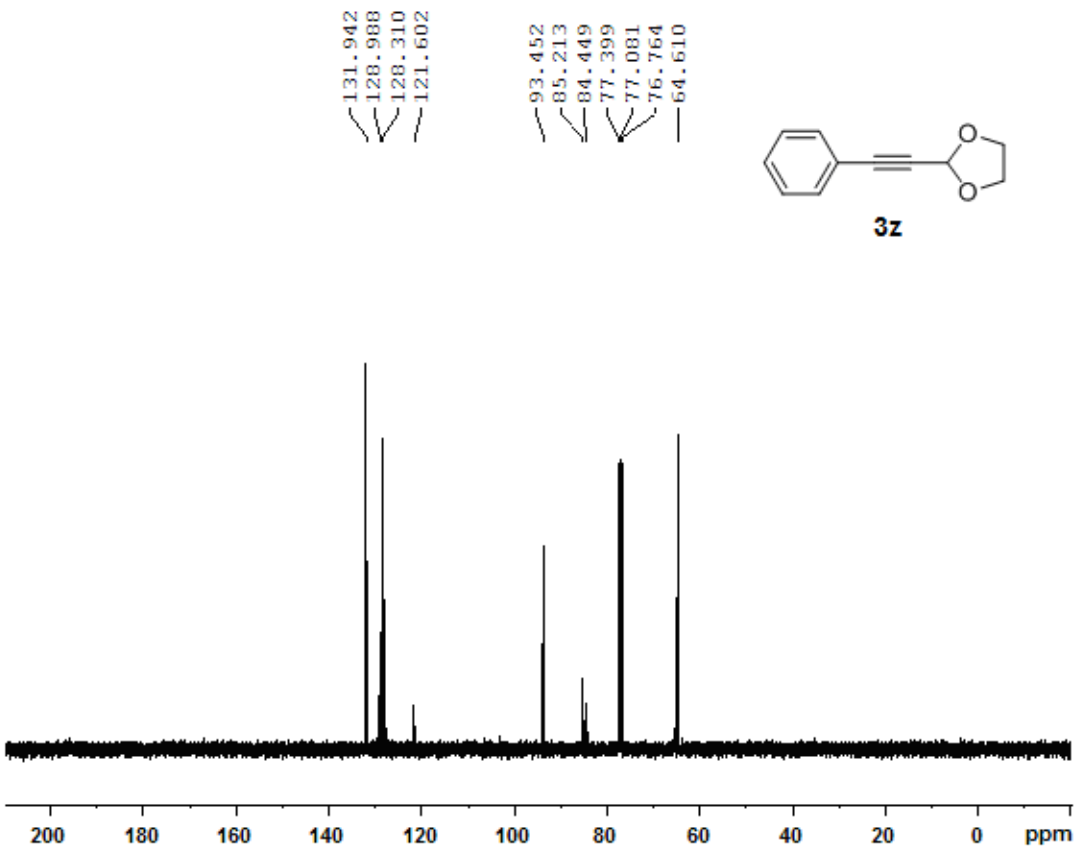

4.627
4.614
3.970
3.952
3.935
3.919
3.833
3.815
3.798
2.270
2.172
2.154
2.047
2.021
2.001
1.991
1.978
1.960
1.947
1.902
1.876
1.683
1.582
1.524
1.495
1.242
OH
O
3x
8
7
6
5
4
3
2
1
0
ppm
1.03
1.10
1.02
1.01
1.20
5.00
2.05
4.01
2.00

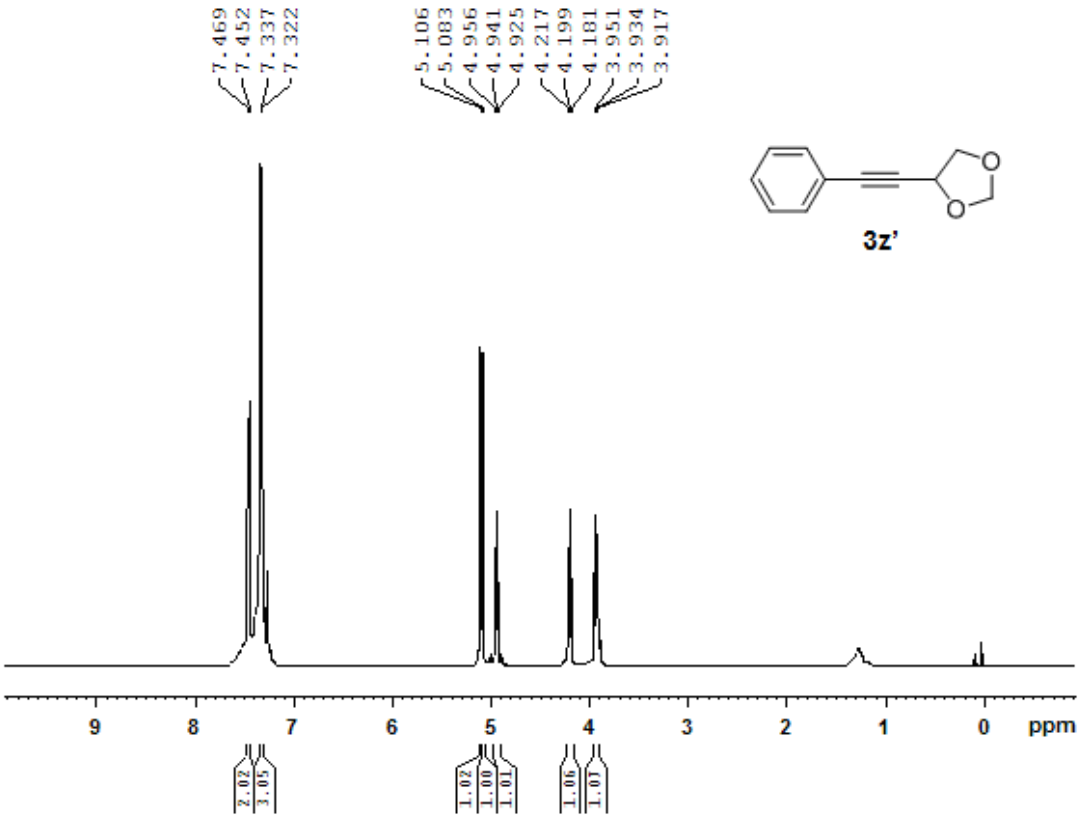

88.043
83.906
77.265
76.947
76.629
68.424
68.027
67.641
39.735
33.324
25.211
25.041
23.209
OH
O
3x
180
160
140
120
100
80
60
40
20
0
ppm

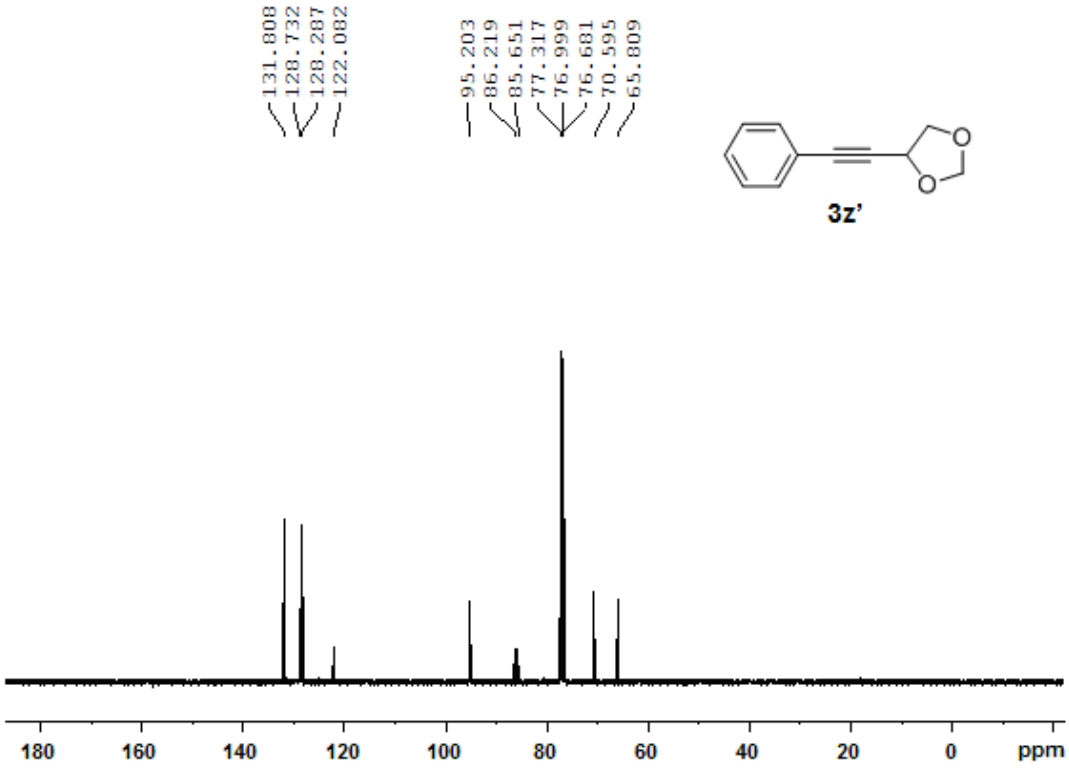

4.566
4.551
3.979
3.960
3.942
3.925
3.817
3.798
3.781
3.764
2.217
2.199
2.182
2.150
2.120
2.110
2.082
2.041
2.024
2.001
1.960
1.945
1.930
1.916
1.904
1.891
1.867
1.540
1.524
1.506
1.489
1.471
1.333
1.303
1.265
0.912
0.895
0.878
C5H11
O
3y
7
6
5
4
3
2
1
0
-1
ppm
1.00
1.01
1.01
2.10
1.03
1.05
2.01
2.00
4.05
3.10

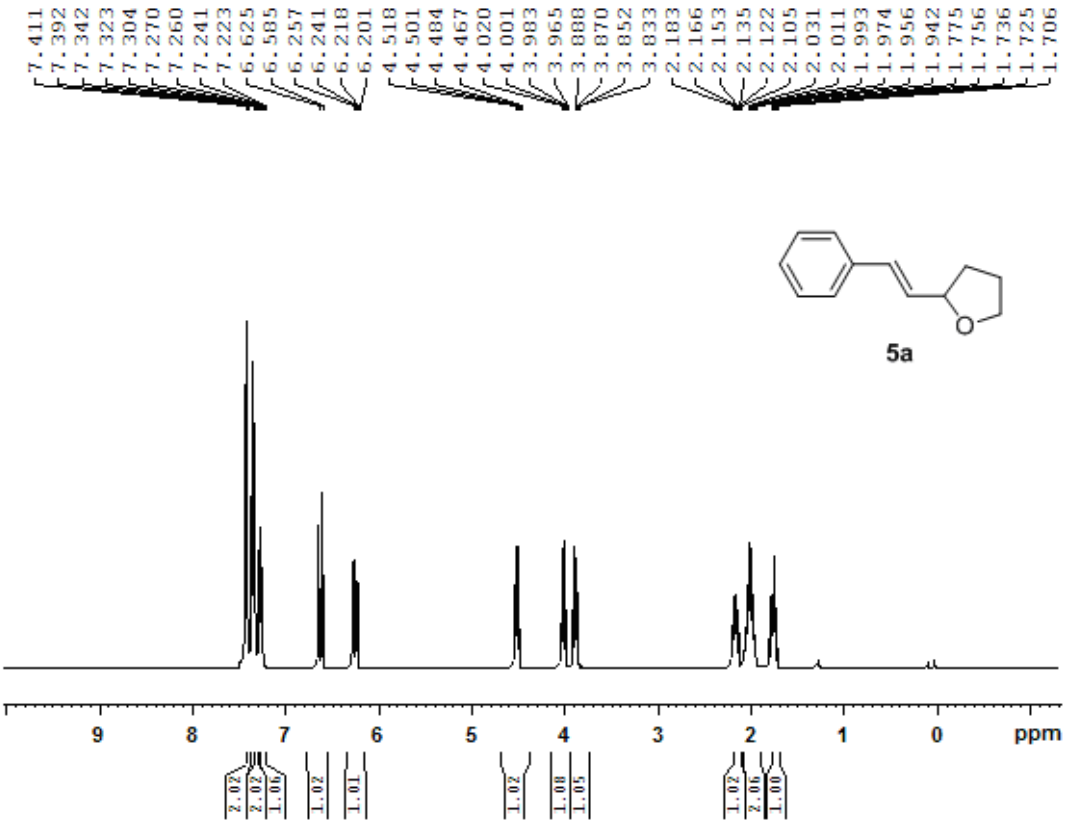

85.210
79.737
77.239
76.921
76.604
68.330
67.559
33.422
30.937
28.227
25.304
22.087
18.627
13.883
C5H11
O
3y
110
100
90
80
70
60
50
40
30
20
10
0
ppm

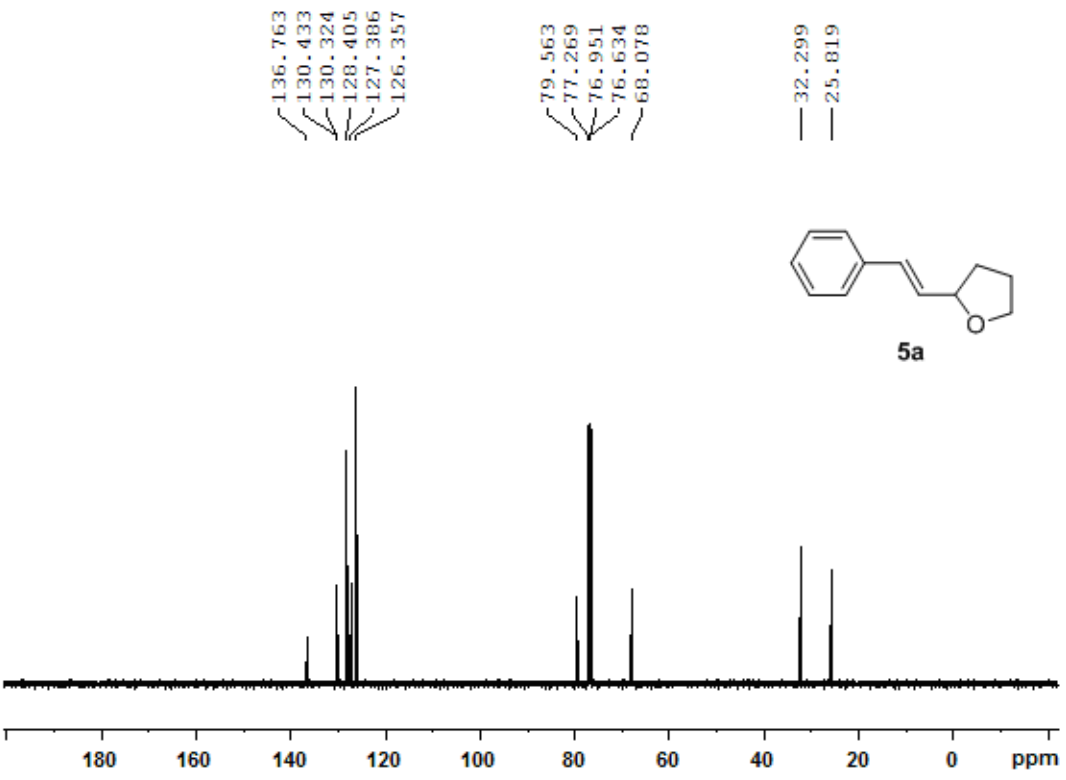

7.489
7.472
7.355
7.339
7.320
5.903
4.165
4.148
4.131
4.013
3.996
3.978
3z
9
8
7
6
5
4
3
2
1
0
ppm
2.05
3.01
1.03
2.00
2.00

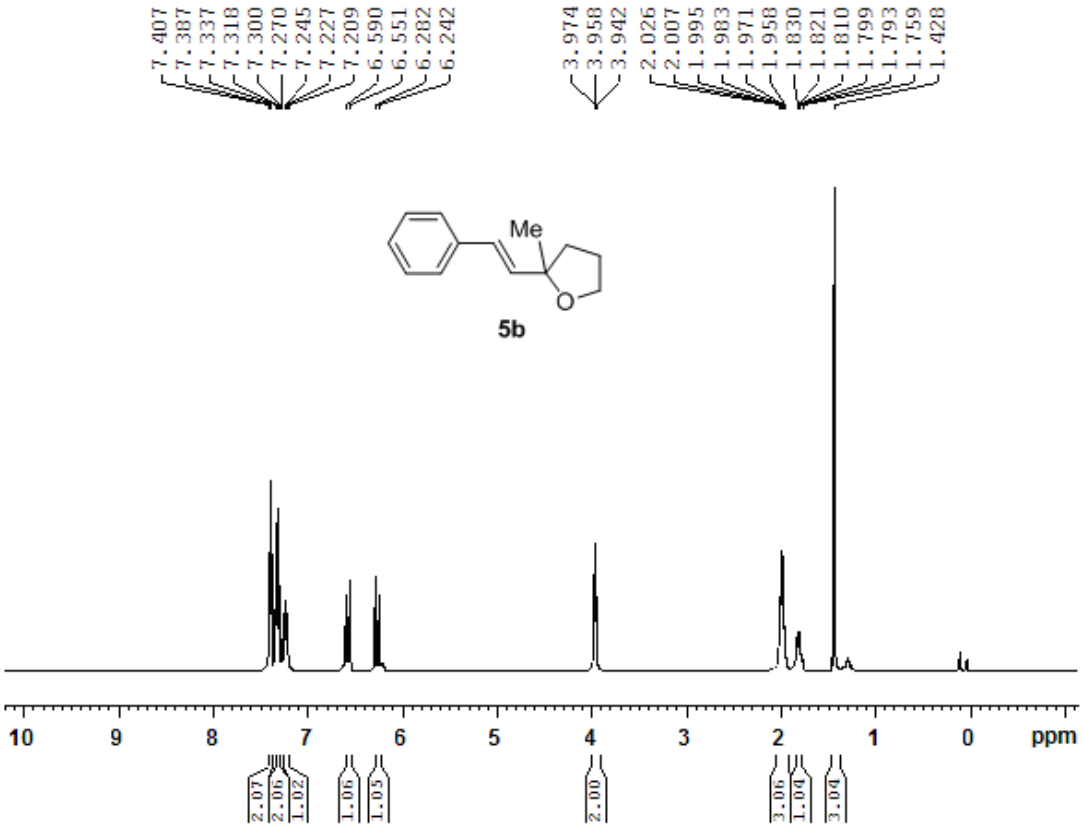

131.942
128.988
128.310
121.602
93.452
85.213
84.449
77.399
77.081
76.764
64.610
3z
200
180
160
140
120
100
80
60
40
20
0
ppm

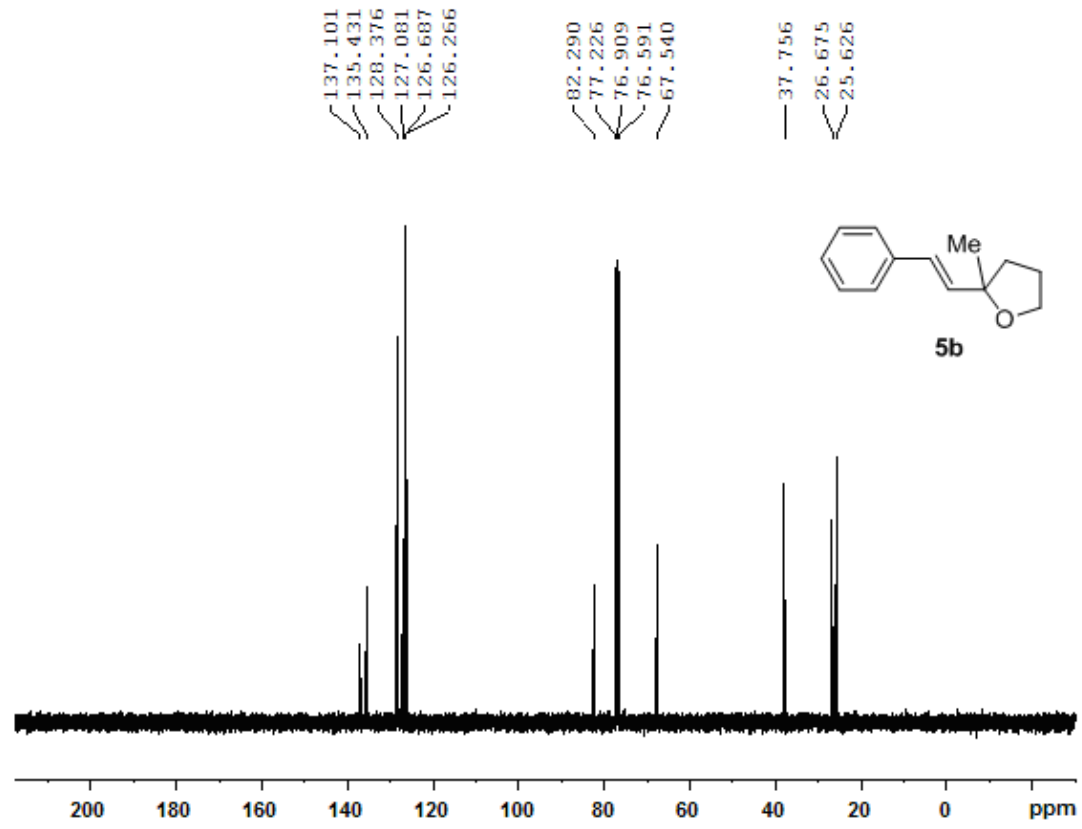

7.469
7.452
7.337
7.322
5.106
5.083
4.956
4.941
4.925
4.217
4.199
4.181
3.951
3.934
3.917
3z'
9
8
7
6
5
4
3
2
1
0
ppm
2.02
3.05
1.02
1.00
1.01
1.06
1.07

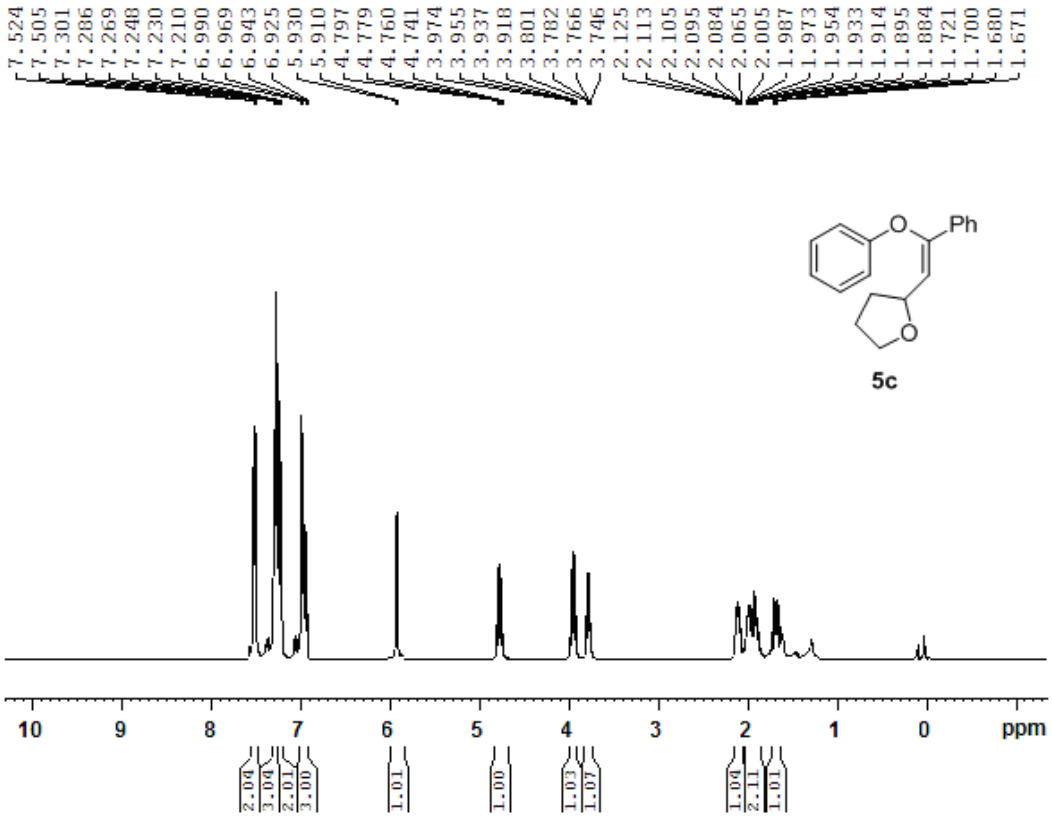

131.808
128.732
128.287
122.082
95.203
86.219
85.651
77.317
76.999
76.681
70.595
65.809
3z'
180
160
140
120
100
80
60
40
20
0
ppm

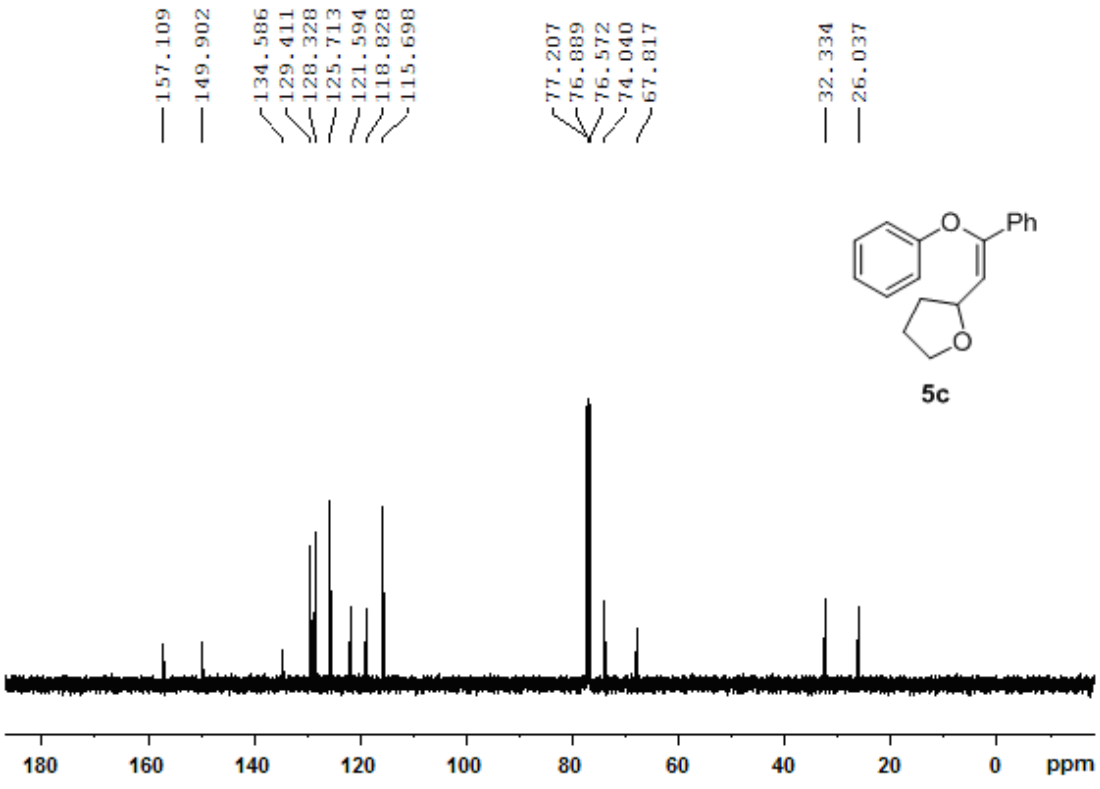

7.411
7.392
7.342
7.323
7.304
7.270
7.260
7.241
7.223
6.625
6.585
6.257
6.241
6.218
6.201
4.518
4.501
4.484
4.467
4.020
4.001
3.983
3.965
3.888
3.870
3.852
3.833
2.183
2.166
2.153
2.135
2.122
2.105
2.031
2.011
1.993
1.974
1.956
1.942
1.775
1.756
1.736
1.725
1.706
5a
9
8
7
6
5
4
3
2
1
0
ppm
2.07
2.02
1.06
1.02
1.01
1.02
1.08
1.05
1.02
2.06
1.00

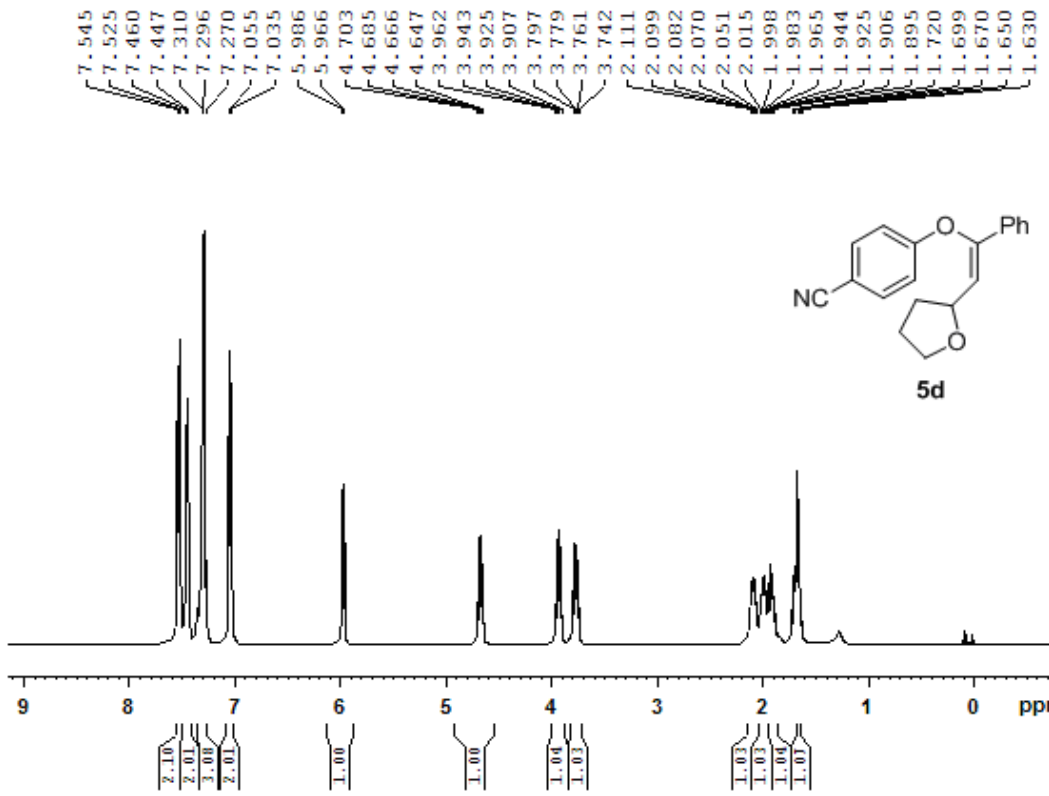

136.763
130.433
130.324
128.405
127.386
126.357
79.563
77.269
76.951
76.634
68.078
32.299
25.819
5a
180
160
140
120
100
80
60
40
20
0
ppm

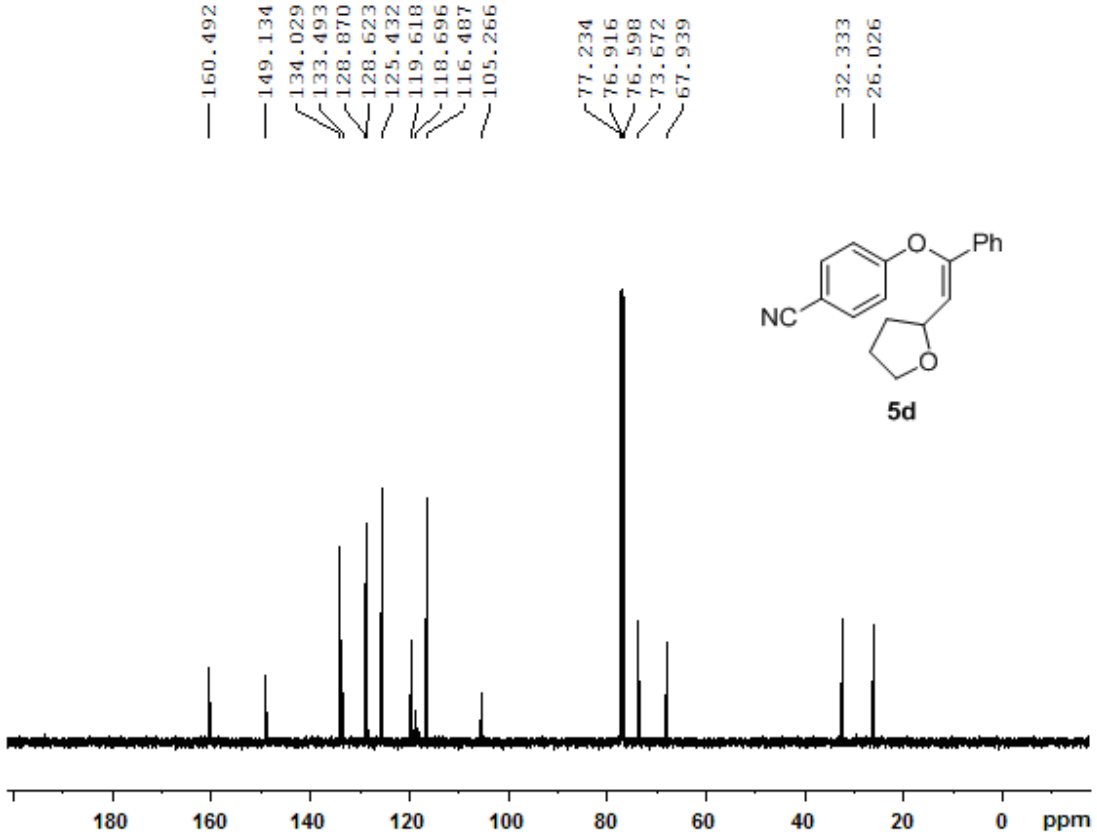

7.407
7.387
7.337
7.318
7.300
7.270
7.245
7.227
7.209
6.590
6.551
6.282
6.242
3.974
3.958
3.942
2.026
2.007
1.995
1.983
1.971
1.958
1.830
1.821
1.810
1.799
1.793
1.759
1.428
Me
O
5b
10
9
8
7
6
5
4
3
2
1
0
ppm
2.07
2.06
1.02
1.06
1.05
2.00
3.06
1.04
3.04

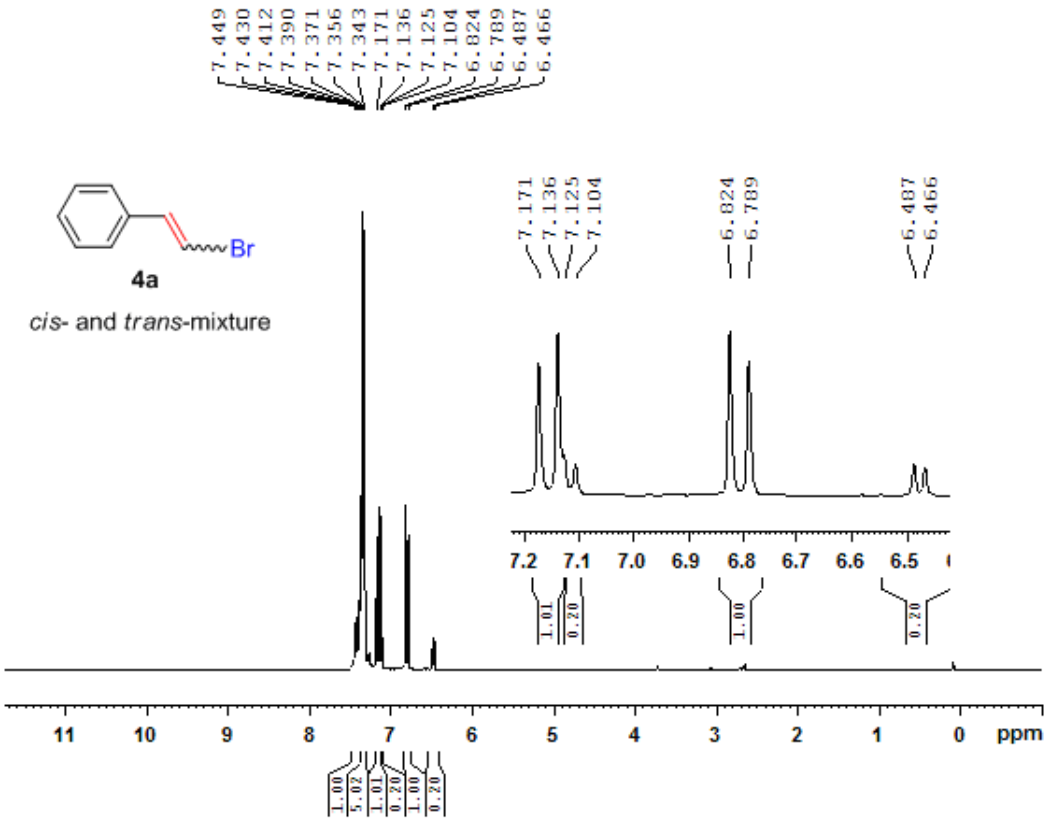

137.101
135.431
128.376
127.081
126.687
126.266
82.290
77.226
76.909
76.591
67.540
37.756
26.675
25.626
Me
O
5b
200
180
160
140
120
100
80
60
40
20
0
ppm

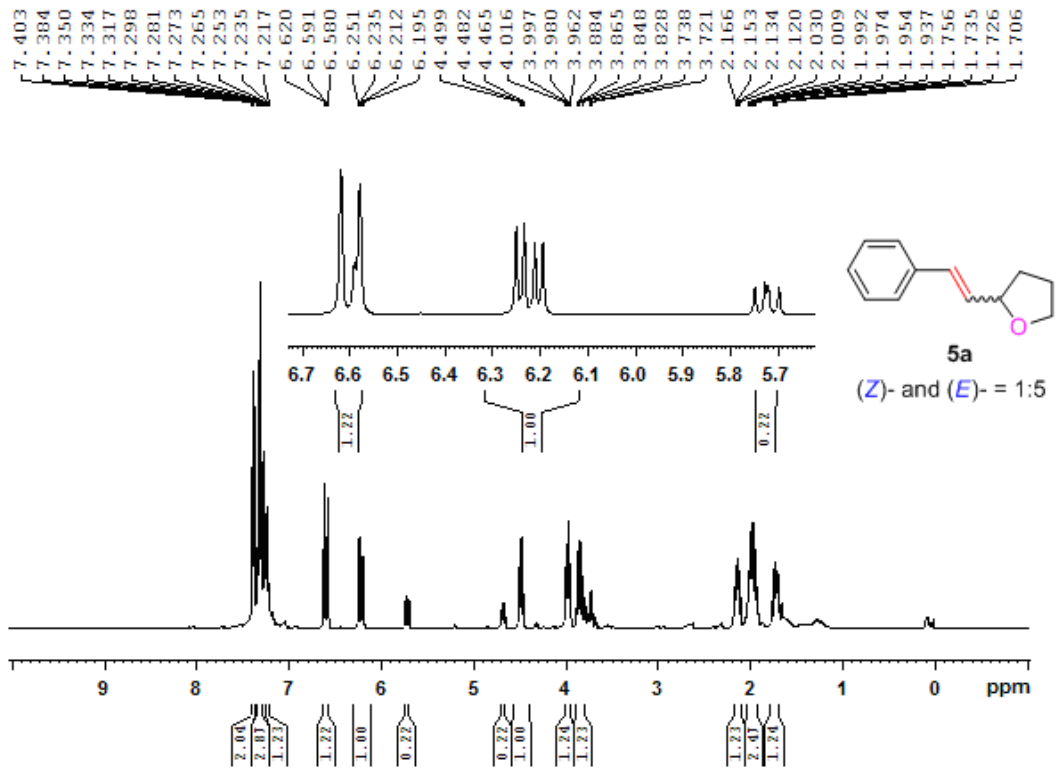

7.524
7.505
7.301
7.286
7.269
7.248
7.230
7.210
6.990
6.969
6.943
6.925
5.930
5.910
4.797
4.779
4.760
4.741
3.974
3.955
3.937
3.918
3.801
3.782
3.766
3.746
2.125
2.113
2.105
2.095
2.084
2.065
2.005
1.987
1.973
1.954
1.933
1.914
1.895
1.884
1.721
1.700
1.680
1.671
O
Ph
O
5c
10
9
8
7
6
5
4
3
2
1
0
ppm
2.04
3.04
2.01
3.00
1.01
1.00
1.03
1.07
1.04
2.11
1.01

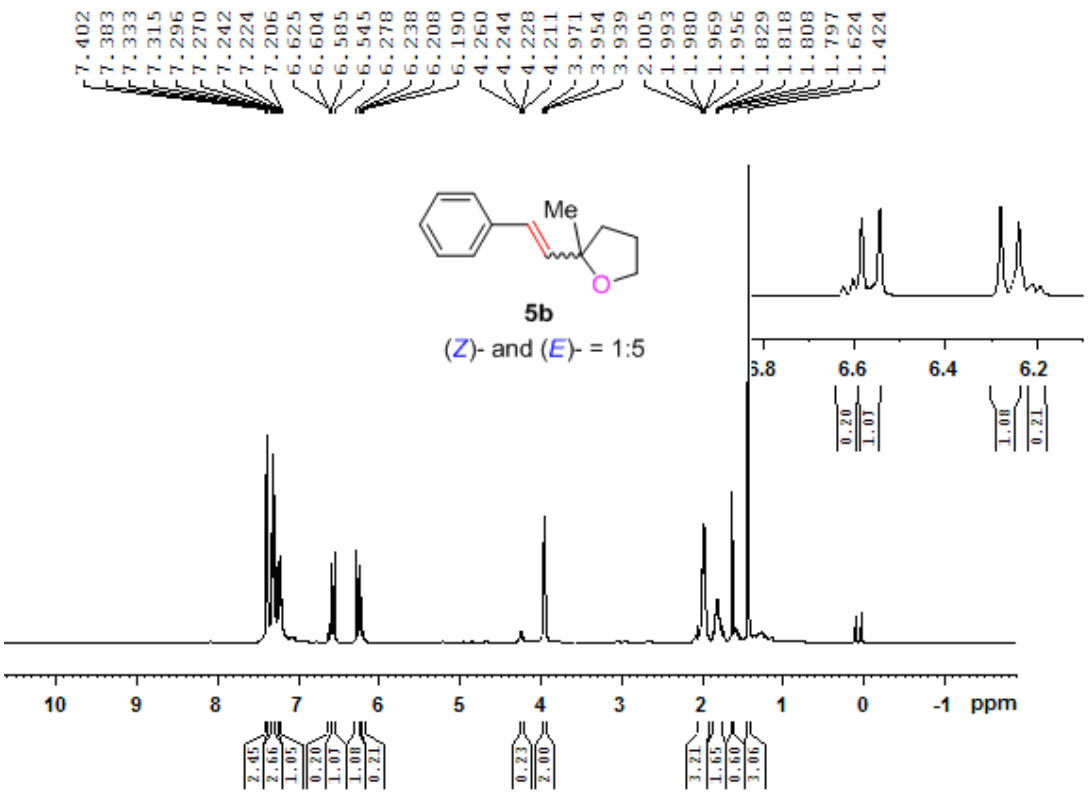

157.109
149.902
134.586
129.411
128.328
125.713
121.594
118.828
115.698
77.207
76.889
76.572
74.040
67.817
32.334
26.037
O
Ph
O
5c
180
160
140
120
100
80
60
40
20
0
ppm


7.545
7.525
7.460
7.447
7.310
7.296
7.270
7.055
7.035
5.986
5.966
4.703
4.685
4.666
4.647
3.962
3.943
3.925
3.907
3.797
3.779
3.761
3.742
2.111
2.099
2.082
2.070
2.051
2.015
1.998
1.983
1.965
1.944
1.925
1.906
1.895
1.720
1.699
1.670
1.650
1.630
O
Ph
NC
O
5d
9
8
7
6
5
4
3
2
1
0
ppm
2.10
2.01
3.08
2.01
1.00
1.00
1.04
1.03
1.03
1.03
1.04
1.07


160.492
149.134
134.029
133.493
128.870
128.623
125.432
119.618
118.696
116.487
105.266
77.234
76.916
76.598
73.672
67.939
32.333
26.026
O
Ph
NC
O
5d
180
160
140
120
100
80
60
40
20
0
ppm


7.449
7.430
7.412
7.390
7.371
7.356
7.343
7.171
7.136
7.125
7.104
6.824
6.789
6.487
6.466
Br
4a
cis- and trans-mixture
7.2
7.1
7.0
6.9
6.8
6.7
6.6
6.5
11
10
9
8
7
6
5
4
3
2
1
0
ppm


7.403
7.384
7.350
7.334
7.317
7.298
7.281
7.273
7.265
7.253
7.235
7.217
6.620
6.591
6.580
6.251
6.235
6.212
6.195
4.499
4.482
4.465
4.016
3.997
3.980
3.962
3.884
3.865
3.848
3.828
3.738
3.721
2.166
2.153
2.134
2.120
2.030
2.009
1.992
1.974
1.954
1.937
1.756
1.735
1.726
1.706
O
5a
(Z)- and (E)- = 1:5
6.7
6.6
6.5
6.4
6.3
6.2
6.1
6.0
5.9
5.8
5.7
9
8
7
6
5
4
3
2
1
0
ppm


7.402
7.383
7.333
7.315
7.296
7.270
7.242
7.224
7.206
6.625
6.604
6.585
6.545
6.278
6.238
6.208
6.190
4.260
4.244
4.228
4.211
3.971
3.954
3.939
2.005
1.993
1.980
1.969
1.956
1.829
1.818
1.808
1.797
1.624
1.424
Me
O
5b
(Z)- and (E)- = 1:5
6.8
6.6
6.4
6.2
0.20
1.07
1.08
0.21
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
2.41
2.66
1.05
0.20
1.07
1.08
0.21
0.23
2.00
3.21
1.65
0.60
3.06