Supporting Information for

One-Pot Synthesis of Carbazoles from Cyclohexanones and Aryl hydrazine Chlorides under Metal-Free Condntions

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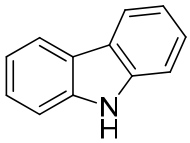
General information:

All experiments were carried out under an atmosphere of oxygen. Flash column chromatography was performed over silica gel 48-75 μm. ^{1}H NMR and ^{13}C NMR spectra were recorded on Bruker-AV (400 and 100 MHz, respectively) instrument internally referenced to $SiMe_4$ or chloroform signals. MS analyses were performed on an Agilent 5975 GC-MS instrument (EI). High-resolution mass spectra were recorded at the Center for Mass Spectrometry, Peking University. The structures of known compounds were further corroborated by comparing their ^{1}H NMR data and MS data with those of literature. All reagents were used as received from commercial sources without further purification.

General procedure: (3a):

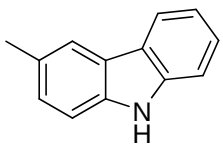
A 25 mL oven-dried reaction vessel was charged with cyclohexanone (**1a**, 20.7 μL, 0.2 mmol), phenylhydrazine hydrochloride (**2a**, 43.4 mg, 0.3 mmol) and reflushed with oxygen (1 atm). *N*-methyl-2-pyrrolidone (0.4 mL) was added to the sealed reaction vessel by syringe. The resulting solution was stirred at 140 ^{o}C for 24 h. After cooling to room temperature the volatiles were removed under vacuum and the residue was purified by column chromatography (silica gel, petroleum ether/ethyl acetate = 5:1) to give **3a** as pale yellow solid; yield: 24.4 mg (73%).

Carbazole (3a, CAS: 86-74-8) [1]



^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 8.09-8.06 (m, 3H), 7.43-7.40 (m, 4H), 7.26-7.23 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 139.6, 125.8, 123.4, 120.3, 119.5, 110.6; MS (EI) m/z (%)**:** 167 (100), 139, 113, 83, 75.

3-Methyl-carbazole (3b, CAS: 4630-20-0) [1]

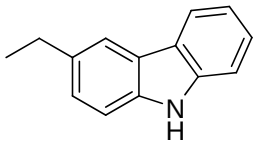


The reaction was conducted with 4-methylcyclohexanone (**1b**, 24.4 μL, 0.2 mmol), phenylhydrazine hydrochloride (**2a**, 43.4 mg, 0.3 mmol). The residue was purified by column

chromatography (silica gel, petroleum ether/ethyl acetate = 5:1) to give **3b** as pale yellow solid; yield: 27.2 mg (75%).

^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 8.04 (d, J = 7.6 Hz, 1H), 7.95 (s, 1H), 7.88 (s, 1H), 7.41-7.20 (m, 5H), 2.53 (s, 3H);^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 139.9, 137.8, 128.8, 127.2, 125.7, 123.6, 123.3, 120.3,120.2, 119.3, 110.6, 110.3, 21.4; MS (EI) m/z (%)：181 (100), 152, 127, 90, 77.

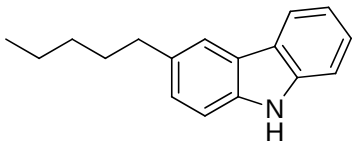
3-Ethyl-carbazole (3c, CAS: 5599-49-5) [1]



The reaction was conducted with 4-ethylcyclohexanone (**1c**, 28.2 μL, 0.2 mmol), phenylhydrazine hydrochloride (**2a**, 43.4 mg, 0.3 mmol). The residue was purified by column chromatography (silica gel, petroleum ether/ethyl acetate = 5:1) to give **3c** as pale yellow solid; yield: 28.5 mg (73%).

^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 8.06 (d, J = 7.8 Hz, 1H), 7.96-7.90 (m, 2H), 7.40-7.34 (m, 3 H), 7.28-7.20 (m, 2H), 2.83 (q, J = 7.5 Hz, 2H), 1.34 (t, J = 7.6 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 139.6, 138.0, 135.6, 126.2, 125.6, 123.6, 123.4, 120.2, 119.3, 119.0, 110.6, 110.4, 29.0, 16.4; MS (EI) m/z (%)： 195, 180 (100), 167, 152, 139, 90.

3-Pentyl-carbazole (3d)

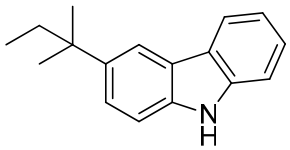


The reaction was conducted with 4-pentylcyclohexanone (**1d**, 37.8 μL, 0.2 mmol), phenylhydrazine hydrochloride (**2a**, 43.4 mg, 0.3 mmol). The residue was purified by column chromatography (silica gel, petroleum ether/ethyl acetate = 5:1) to give **3d** as pale yellow solid; yield: 35.1 mg (74%).

^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 8.05 (d, J = 7.6 Hz, 1H), 7.95-7.87 (m, 2H), 7.40-7.33 (m, 3 H), 7.26-7.20 (m, 2H), 2.78 (q, J = 7.6 Hz, 2H), 1.72-7.70 (m, 2H), 1.37 (m, 4H), 0.91 (m, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 139.9, 138.0, 134.2,126.6, 125.6, 123.6, 123.4, 120.2,

119.6, 119.2, 110.5, 110.2, 36.0, 32.0, 31.6, 22.6, 14.0; MS (EI) m/z (%)：237, 217, 204, 191, 180 (100), 152; HRMS calcd. for : $C_{17}H_{19}N$ $[M]^+$ 237.1512, found 237.1511.

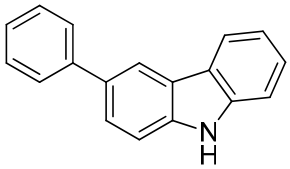
3-(*tert*-Pentyl)- carbazole (3e)



The reaction was conducted with 4-(*tert*-pentyl)cyclohexanone (**1e**, 36.6 μL, 0.2 mmol), phenylhydrazine hydrochloride (**2a**, 43.4 mg, 0.3 mmol). The residue was purified by column chromatography (silica gel, petroleum ether/ethyl acetate = 5:1) to give **3e** as pale yellow solid; yield: 36.5 mg (74%).

^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 8.08 (d, J = 7.6 Hz, 1H), 8.02 (s, 1H), 7.95 (s, 1H), 7.44-7.35 (m, 4H), 7.26-7.20 (m, 1H), 1.76 (q, J = 7.2 Hz, 2H), 1.41 (s, 6H), 0.72 (t, J = 7.4 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 140.8, 140.0, 137.7, 125.5, 124.4, 123.7, 123.2, 120.1, 119.2, 117.3, 110.6, 110.0, 37.9, 37.4, 29.1, 9.3; MS (EI) m/z (%)：237, 222, 208 (100), 191, 180, 167; HRMS calcd. for : $C_{17}H_{20}N$ $[M+1]^+$ 238.1590, found 238.1589.

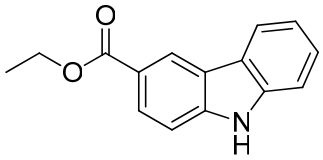
3-Phenyl-carbazole (3f, CAS: 103012-26-6) [2]



The reaction was conducted with 4-phenylcyclohexanone (**1f**, 34.8 mg, 0.2 mmol), phenylhydrazine hydrochloride (**2a**, 43.4 mg, 0.3 mmol). The residue was purified by column chromatography (silica gel, petroleum ether/ethyl acetate = 5:1) to give **3f** as pale yellow solid; yield: 33.0 mg (68%).

^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 8.29 (s, 1H), 8.14-8.08 (m, 2H), 7.73-7.67 (m, 3H), 7.50-7.42 (m, 5H), 7.36-7.33 (m, 1H), 7.26 (s, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 142.2, 140.1, 139.1, 133.2, 128.7, 127.3, 126.5, 126.1, 125.5, 124.0, 123.6, 120.4, 119.6, 118.9, 110.8, 110.7 MS (EI) m/z (%)：243 (100), 227, 213, 139, 120.

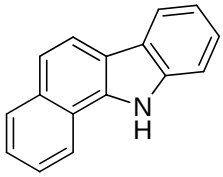
Ethyl carbazole-3-carboxylate (3g, CAS: 51035-14-4) [3]



The reaction was conducted with ethyl 4-oxocyclohexanecarboxylate (**1g**, 31.9 μL, 0.2 mmol), phenylhydrazine hydrochloride (**2a**, 43.4 mg, 0.3 mmol). The residue was purified by column chromatography (silica gel, petroleum ether/ethyl acetate = 5:1) to give **3g** as pale yellow solid; yield: 33.9 mg (71%).

^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 8.82 (s, 1H), 8.29 (s, 1H), 8.15-8.14 (m, 2H), 7.47-7.43 (m, 3H), 7.32-7.26 (m, 1H), 4.44 (q, J = 7.2 Hz, 2H), 1.46 (t, J = 7.0 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 167.5, 142.3, 140.0, 127.5, 126.5, 123.4, 123.2, 122.8, 121.9, 120.7, 120.3, 110.9, 110.1, 60.7, 14.5; MS (EI) m/z (%)：239, 224, 211, 194 (100), 166, 139.

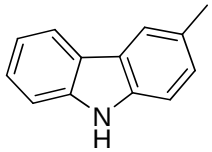
1,2-Benzocarbazole (3j, CAS: 239-01-0) [2]



The reaction was conducted with 3,4-dihydronaphthalen-1-one (**1j**, 26.7 μL, 0.2 mmol), phenylhydrazine hydrochloride (**2a**, 43.4 mg, 0.3 mmol). The residue was purified by column chromatography (silica gel, petroleum ether/ethyl acetate = 5:1) to give **3j** as pale yellow solid; yield: 34.7 mg (80%).

^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 8.76 (s, 1H), 8.14-7.99 (m, 4H), 7.66-7.50 (m, 4H), 7.44-7.41 (m, 1H), 7.32-7.24 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 138.6, 134.9, 132.5, 129.1, 125.6, 125.2, 124.9, 124.3, 121.2, 120.5, 120.3, 120.0, 119.9, 119.3, 118.6, 111.1.MS (EI) m/z (%)：217 (100), 189, 163, 108, 94.

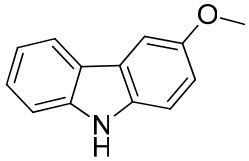
3-Methyl-carbazole (3k, CAS: 4630-20-0) [1]



The reaction was conducted with cyclohexanone (**1a**, 20.7 μL, 0.2 mmol), *p*-tolylhydrazine hydrochloride (**2b**, 47.4 mg, 0.3 mmol). The residue was purified by column chromatography (silica gel, petroleum ether/ethyl acetate = 5:1) to give **3k** as pale yellow solid; yield: 21.7 mg (63%).

^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 8.04 (d, J = 7.6 Hz, 1H), 7.95 (s, 1H), 7.88 (s, 1H), 7.41-7.20 (m, 5H), 2.53 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 139.9, 137.8, 128.8, 127.2, 125.7, 123.6, 123.3, 120.3, 120.2, 119.3, 110.6, 110.3, 21.4; MS (EI) m/z (%)**:** 181 (100), 152, 127, 90, 77.

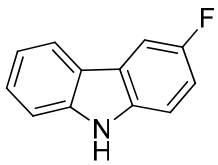
3-Methoxy-carbazole (3l, CAS: 18992-85-3) [1]



The reaction was conducted with cyclohexanone (**1a**, 20.7 μL, 0.2 mmol), (4-methoxyphenyl)hydrazine hydrochloride (**2c**, 52.4 mg, 0.3 mmol). The residue was purified by column chromatography (silica gel, petroleum ether/ethyl acetate = 4:1) to give **3l** as pale yellow solid; yield: 24.0 mg (61%).

^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 8.04 (d, J = 7.6 Hz, 1H), 7.92 (s, 1H), 7.56 (s, 1H), 7.41 (s, 1H), 7.34 (d, J = 8.8 Hz, 1H), 7.26-7.21 (m, 2H), 7.08-7.06 (m, 1H), 3.94 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 154.0, 140.4, 134.5, 125.8, 123.9, 123.4, 120.3, 119.1, 115.1, 111.3, 110.8, 103.4, 56.2; MS (EI) m/z (%)**:** 197, 182 (100), 154, 139, 127, 98.

3-Fluoro-carbazole (3m, CAS: 391-45-7) [1]

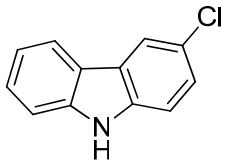


The reaction was conducted with cyclohexanone (**1a**, 20.7 μL, 0.2 mmol), (4-fluorophenyl)hydrazine hydrochloride (**2d**, 48.6 mg, 0.3 mmol). The residue was purified by

column chromatography (silica gel, petroleum ether/ethyl acetate = 5:1) to give **3m** as pale yellow solid; yield: 29.6 mg (80%).

^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 8.03-8.01 (m, 2H), 7.73 (d, J = 7.6 Hz, 1H), 7.43 (s, 2H), 7.36-7.33 (m, 1H), 7.26-7.23 (m, 1H), 7.18-7.14 (m,1H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 157.6 (J = 235.0 Hz), 140.6, 135.8, 126.4, 124.0 (J = 9.7 Hz), 123.2 (J = 3.8 Hz), 120.6, 119.5, 113.6 (J = 25.5 Hz), 111.1 (J = 9.0 Hz), 110.9, 106.0 (J = 23.7 Hz); MS (EI) m/z (%)：185 (100), 164, 157, 131, 92.

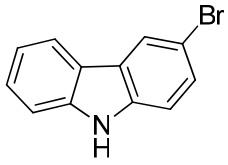
3-Chloro-carbazole (3n, CAS: 2732-25-4) [1]



The reaction was conducted with cyclohexanone (**1a**, 20.7 μL, 0.2 mmol), (4-chlorophenyl)hydrazine hydrochloride (**2e**, 53.4 mg, 0.3 mmol). The residue was purified by column chromatography (silica gel, petroleum ether/ethyl acetate = 5:1) to give **3n** as pale yellow solid; yield: 30.2 mg (75%).

^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 8.07-8.02 (m, 3H), 7.44 (s, 2H), 7.38-7.33 (m, 2H), 7.26 (s,1H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 140.1, 137.8, 126.6, 125.9, 125.0, 124.7, 122.6, 120.5, 120.1, 119.9, 111.5, 110.8; MS (EI) m/z (%)：201 (100), 174, 166, 139, 113, 82.

3-Bromo-carbazole (3o, CAS: 1592-95-6) [4]

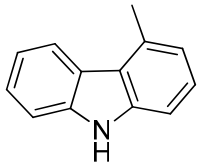


The reaction was conducted with cyclohexanone (**1a**, 20.7 μL, 0.2 mmol), (4-bromophenyl)hydrazine hydrochloride (**2f**, 66.6 mg, 0.3 mmol). The residue was purified by column chromatography (silica gel, petroleum ether/ethyl acetate = 5:1) to give **3o** as pale yellow solid; yield: 33.3 mg (68%).

^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 8.19 (s, 1H), 8.08-8.02 (m, 2H), 7.51-7.44 (m, 3H), 7.32-7.26 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 139.9, 138.1, 128.6, 126.6, 125.3, 123.1,

122.5, 120.5, 119.9, 112.3, 112.0, 110.8; MS (EI) m/z (%)：247 (100), 166, 139, 123, 113, 82.

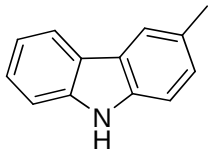
4-Methyl-carbazole (3p, CAS: 6510-65-2) [1]



The reaction was conducted with cyclohexanone (**1a**, 20.7 μL, 0.2 mmol), *o*-tolylhydrazine hydrochloride (**2g**, 47.4 mg, 0.3 mmol). The residue was purified by column chromatography (silica gel, petroleum ether/ethyl acetate = 5:1) to give **3p** as pale yellow solid; yield: 15.2 mg (42%).

^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 8.07 (d, J = 7.6 Hz, 1H), 7.98-7.93 (m, 2H), 7.48-7.40 (m, 2H), 7.26-7.23 (m, 2H), 7.19-7.15 (m, 1H), 2.58 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, pp m): δ 139.5, 138.9, 126.4, 125.8, 125.7, 124.0, 123.0, 120.5, 119.6, 119.5, 118.0, 110.7, 16. 8; MS (EI) m/z (%)：MS (EI) m/z (%)：197, 182 (100), 154, 139, 127, 98.

3-Methyl-carbazole (3r, CAS: 4630-20-0) [1]



The reaction was conducted with cyclohexanone (**1a**, 20.7 μL, 0.2 mmol), phenylhydrazine sulfate (**2i**, 61.8 mg, 0.3 mmol). The residue was purified by column chromatography (silica gel, petroleum ether/ethyl acetate = 5:1) to give **3r** as pale yellow solid; yield: 26.1 mg (72%).

^{1}H NMR ($CDCl_3$, 400 MHz, ppm): δ 8.04 (d, J = 7.6 Hz, 1H), 7.95 (s, 1H), 7.88 (s, 1H), 7.41-7.20 (m, 5H), 2.53 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, ppm): δ 139.9, 137.8, 128.8, 127.2, 125.7, 123.6, 123.3, 120.3, 120.2, 119.3, 110.6, 110.3, 21.4; MS (EI) m/z (%)：181 (100), 152, 127, 90, 77.

References:

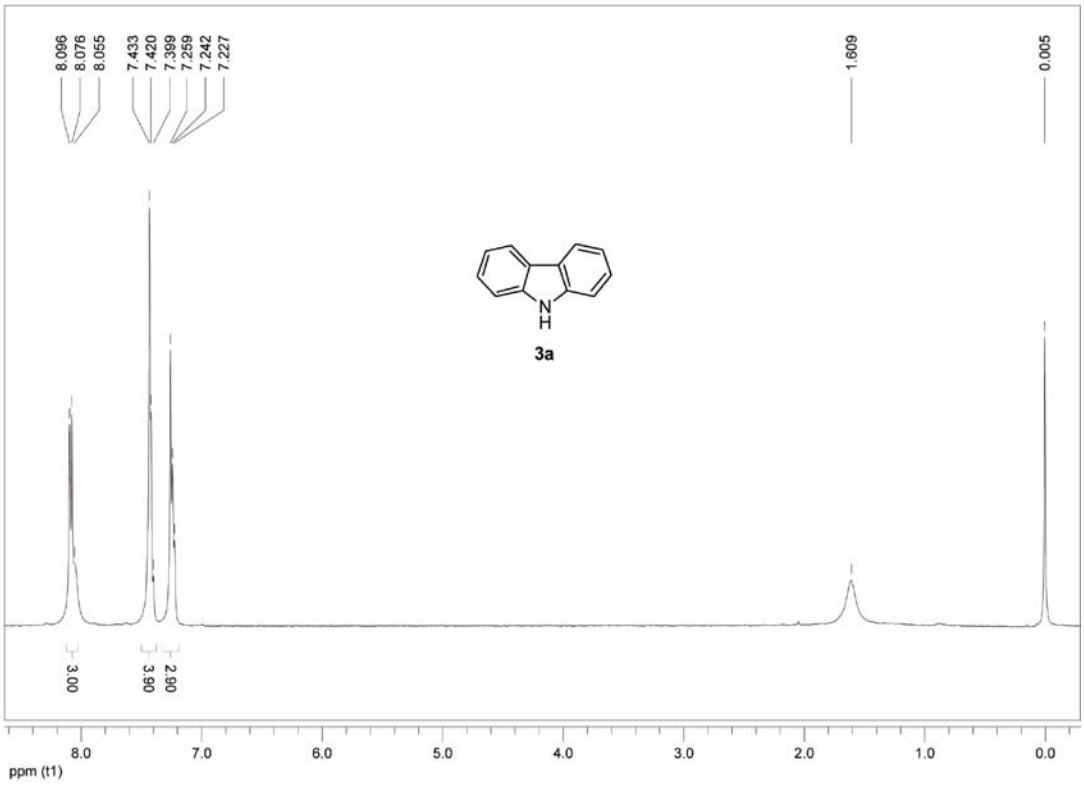
[1] Stokes, B. J.; Jovanovic, B.; Dong, H.; Richert, K. J.; Riell, R. D.; Driver, T. G. *J. Org. Chem.* **2009** , *74*, 3225.

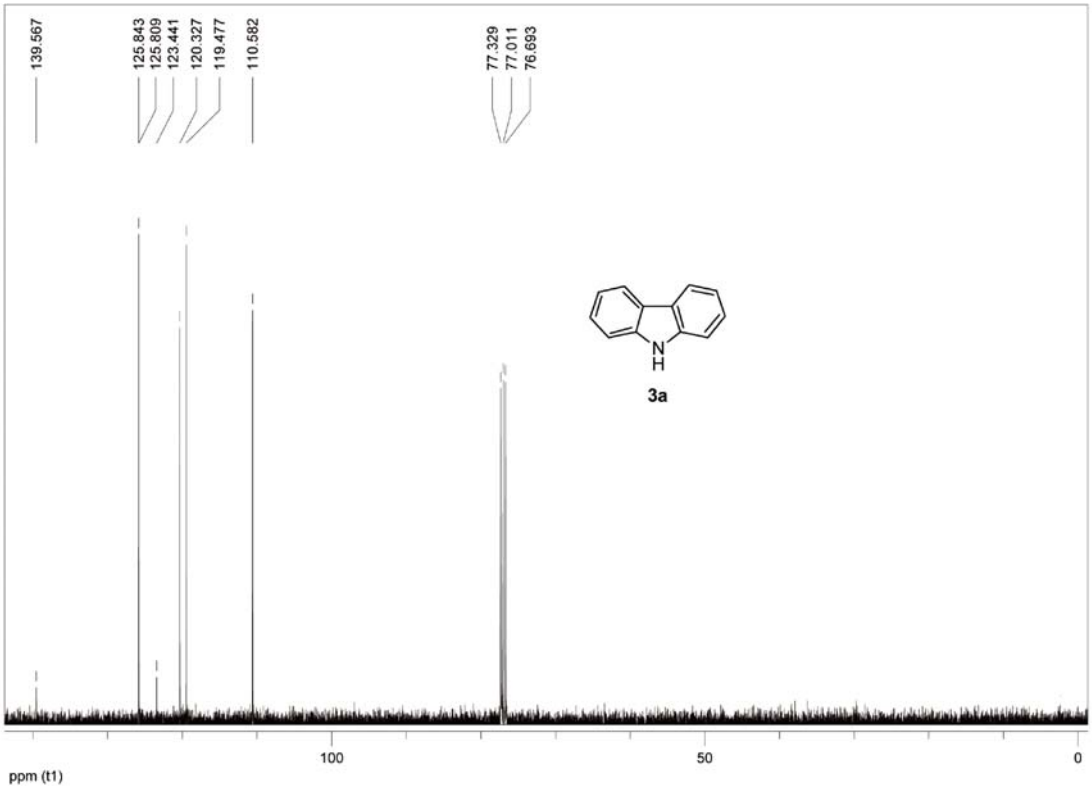
[2] Buden, M. E.; Vaillard, V. A.; Martin, S. E.; Rossi, R. A. *J. Org. Chem.* **2009**, *74*, 4490.

[3] Yang, W.; Zhou, J.; Wang, B.; Ren, H. *Chem-Eur. J*. **2011**, *17*, 13665.

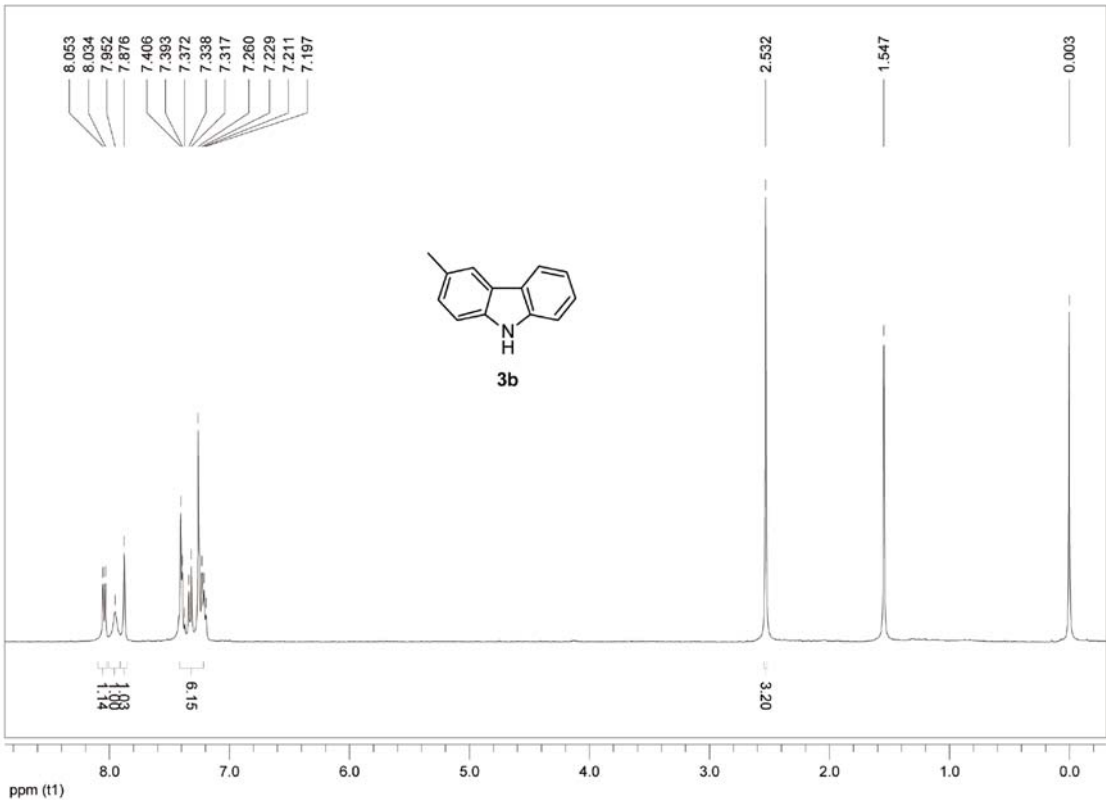
[4] Midya, A.; Yang, J.; Loh, K.; Xie, Z.; Wang, J.; Chen, Z.-K. *Chem. Commun.* **2010**, *46*, 2091.

^{1}H NMR and ^{13}C NMR spectra for all compounds

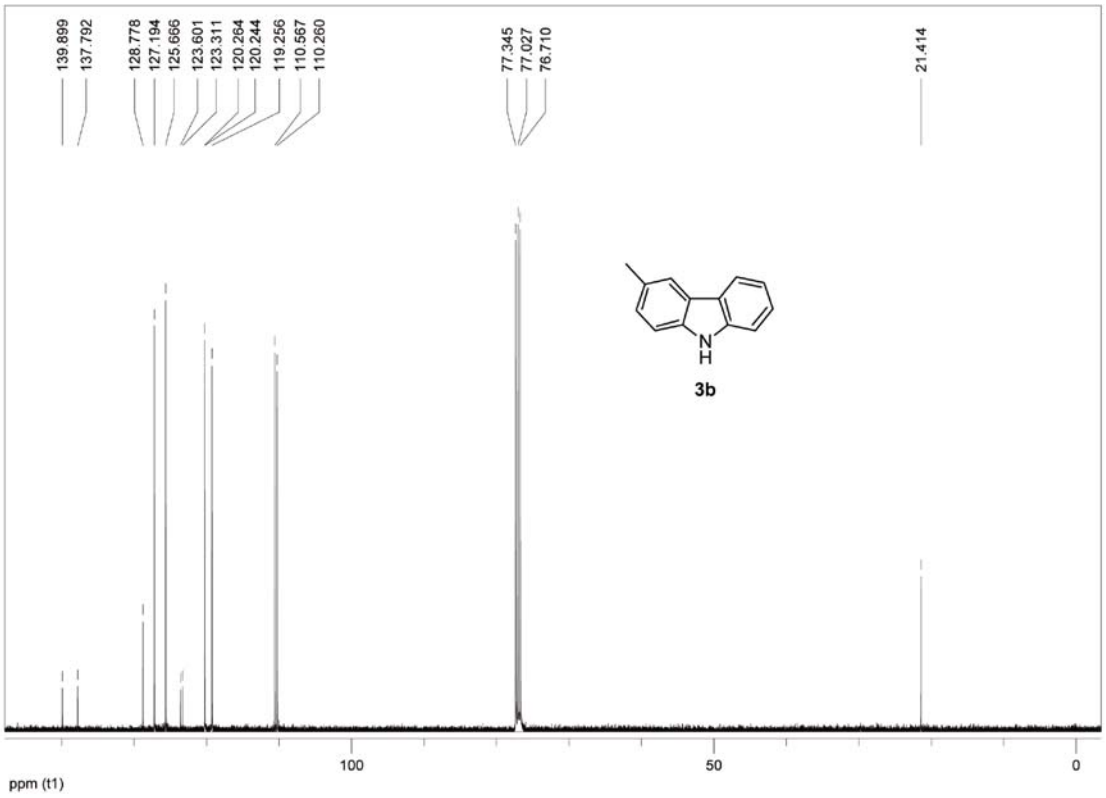




3a



3b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21.414
3b
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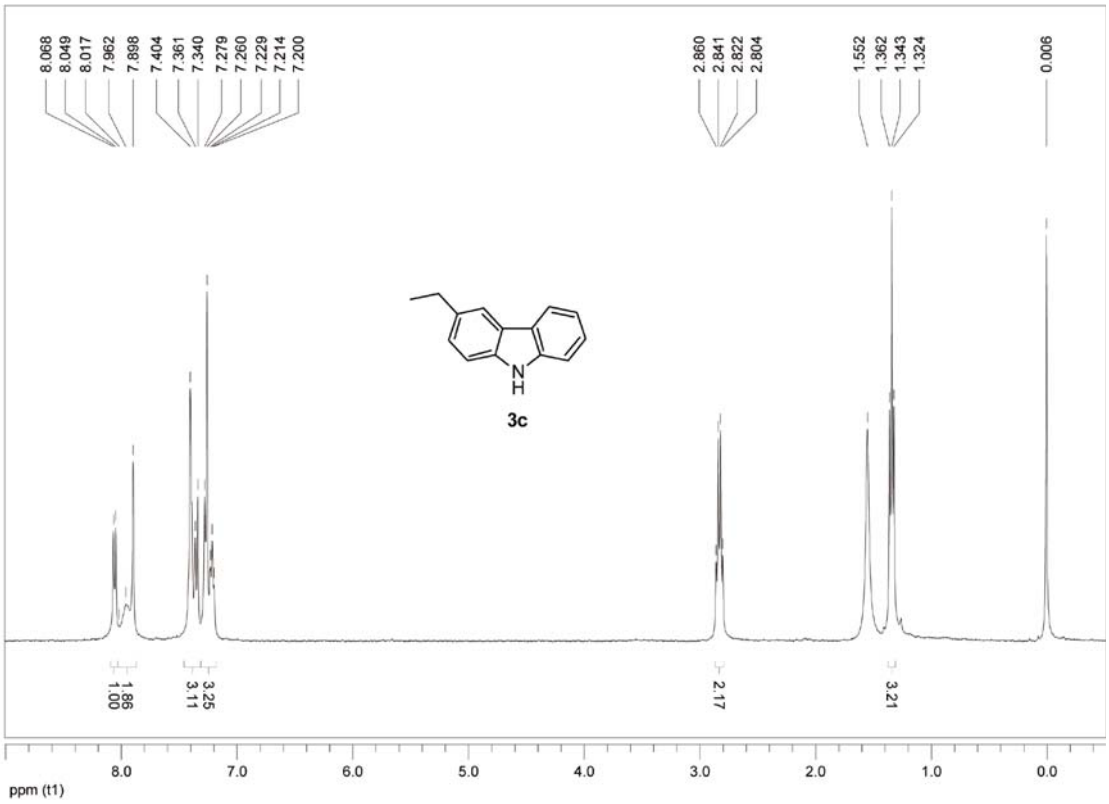
50
0
ppm (t1)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7.260
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7.200
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2.841
2.822
2.804
1.552
1.362
1.343
1.324
0.006
3c
1.00
1.89
3.11
3.25
2.17
3.21
8.0
7.0
6.0
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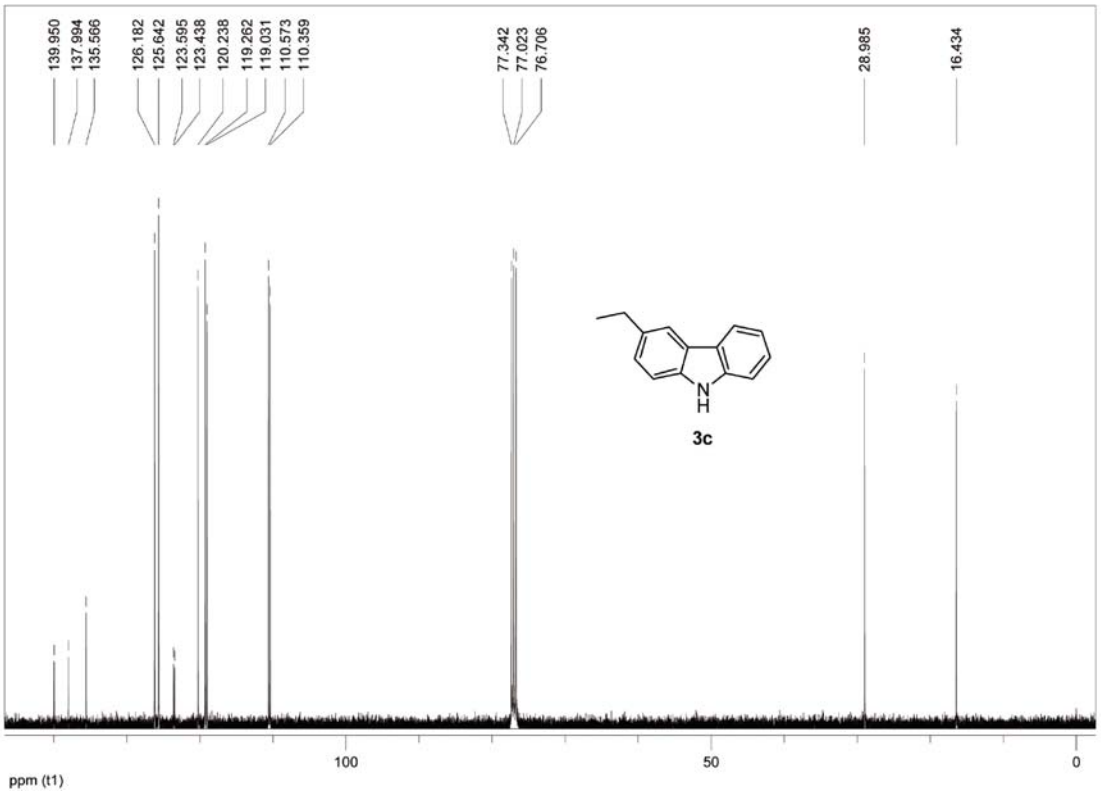
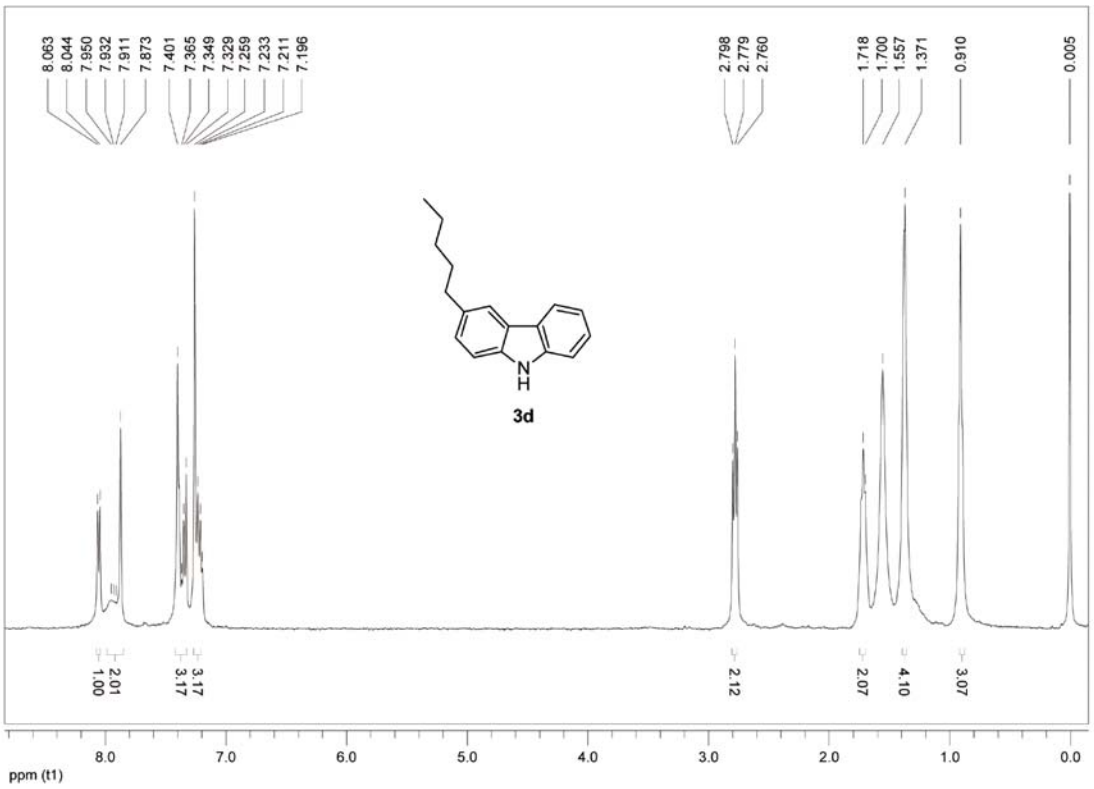
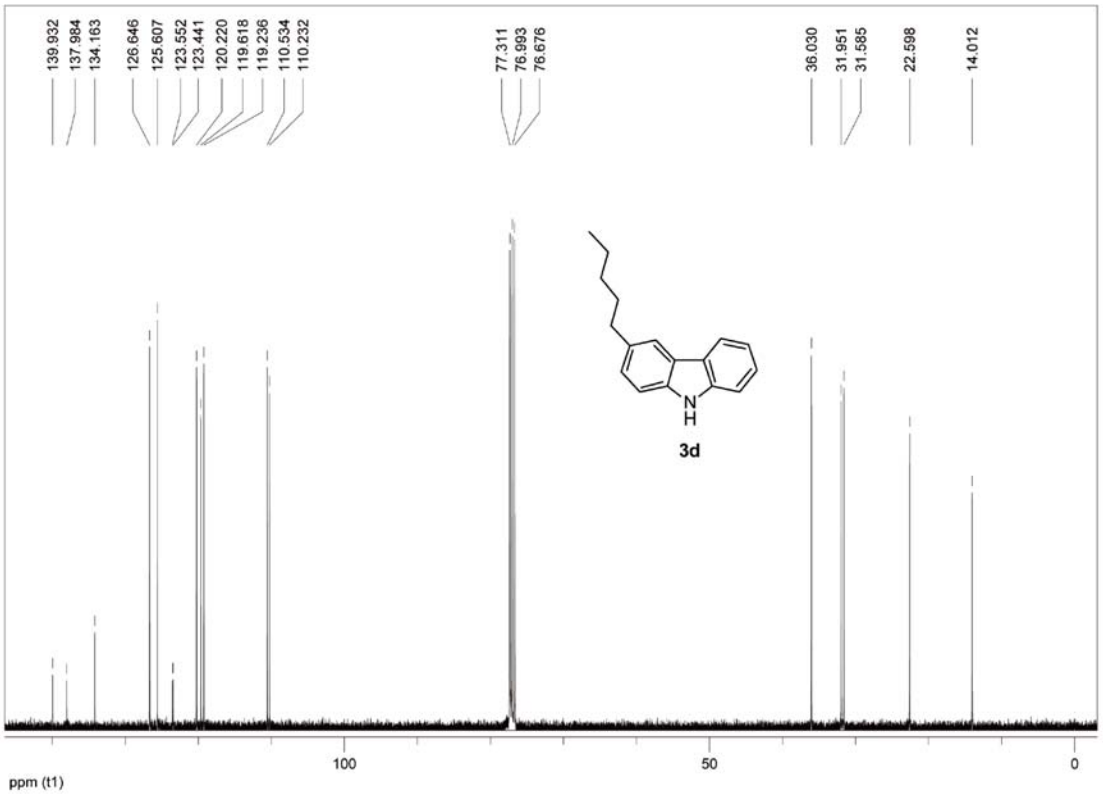
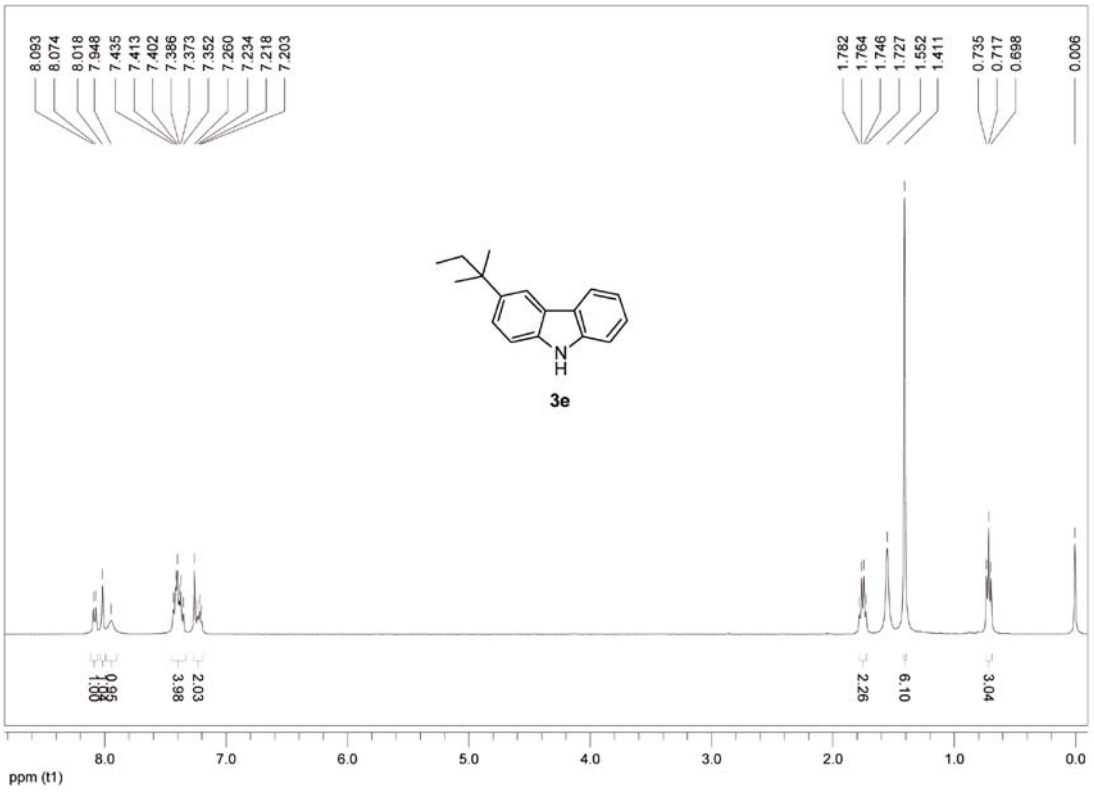
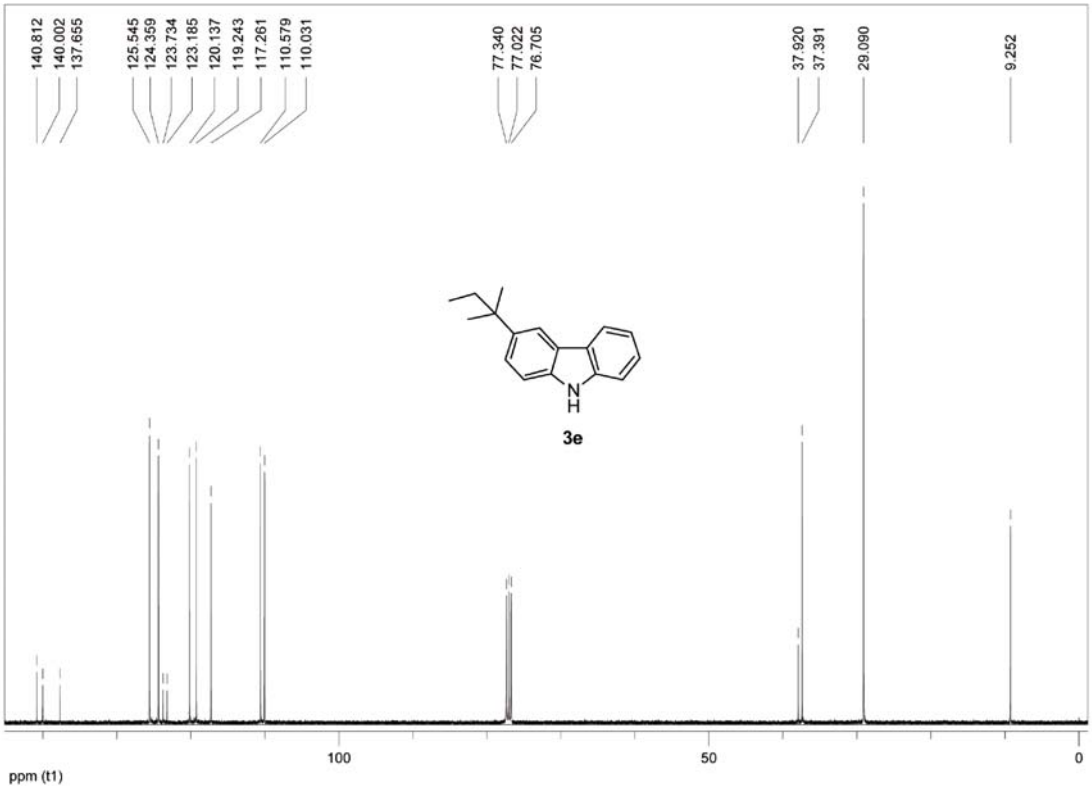
4.0
3.0
2.0
1.0
0.0
ppm (t1)

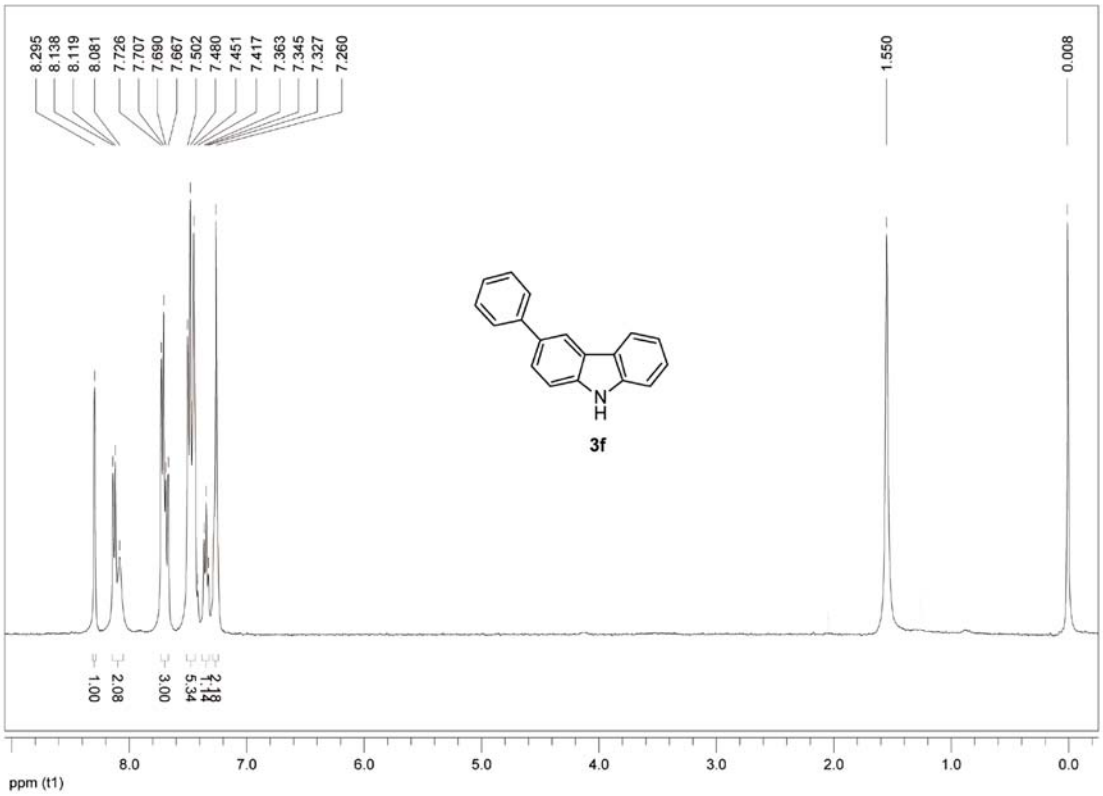

139.950
137.994
135.566
126.182
125.642
123.595
123.438
120.238
119.262
119.031
110.573
110.359
77.342
77.023
76.706
28.985
16.434
3c
100
50
0
ppm (t1)

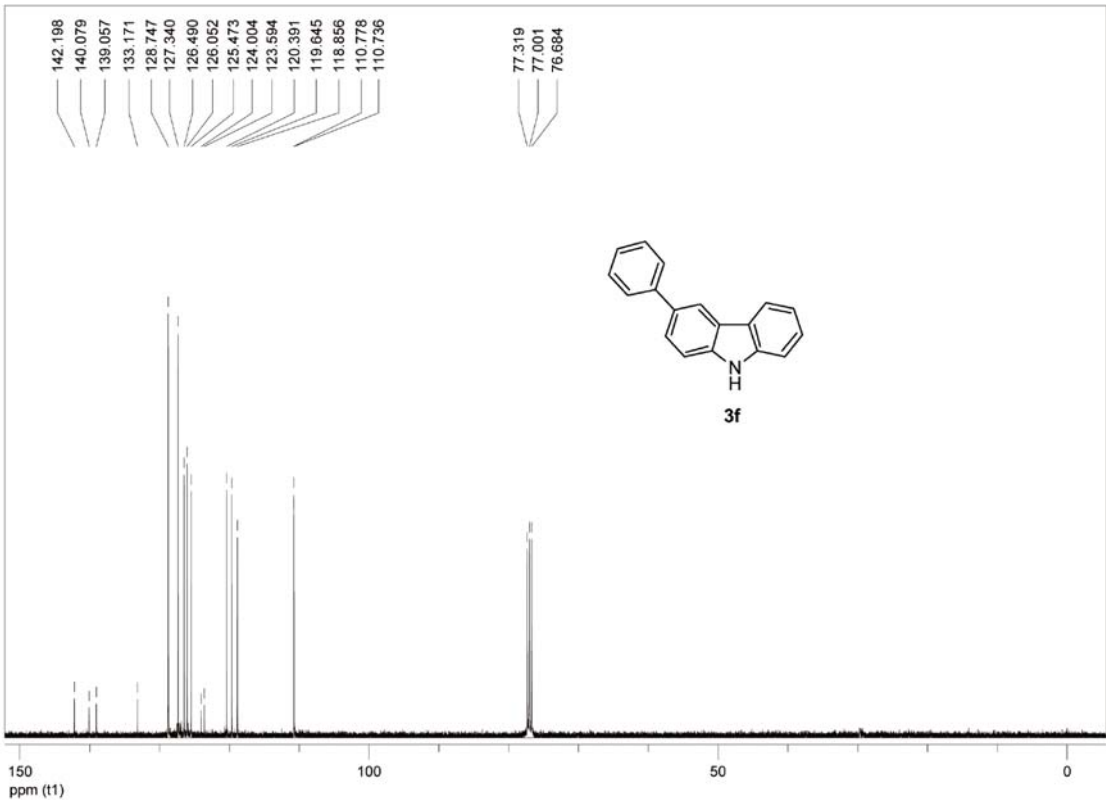

8.063
8.044
7.950
7.932
7.911
7.873
7.401
7.365
7.349
7.329
7.259
7.233
7.211
7.196
2.798
2.779
2.760
1.718
1.700
1.557
1.371
0.910
0.005
3d
1.00
2.01
3.17
3.17
2.12
2.07
4.10
3.07
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


139.932
137.984
134.163
126.646
125.607
123.552
123.441
120.220
119.618
119.236
110.534
110.232
77.311
76.993
76.676
36.030
31.951
31.585
22.598
14.012
3d
100
50
0
ppm (t1)

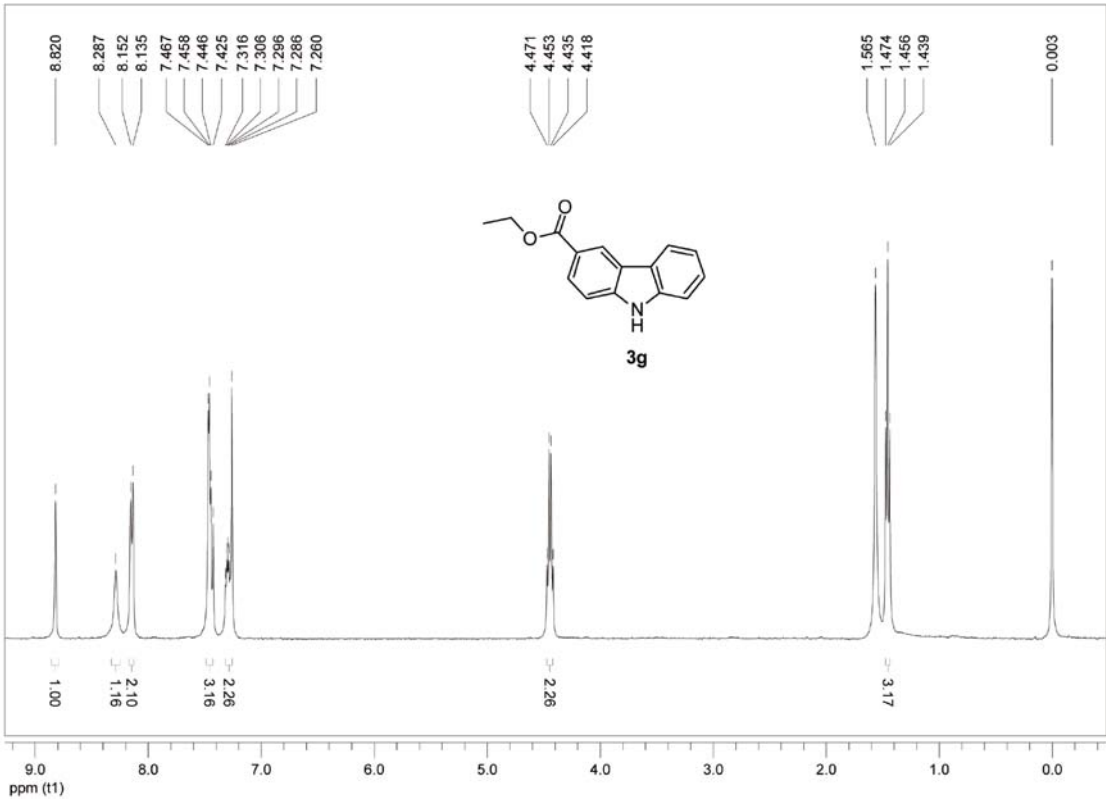

8.093
8.074
8.018
7.948
7.435
7.413
7.402
7.386
7.373
7.352
7.260
7.234
7.218
7.203
1.782
1.764
1.746
1.727
1.552
1.411
0.735
0.717
0.698
0.006
3e
1.00
0.96
3.98
2.03
2.26
6.10
3.04
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


140.812
140.002
137.655
125.545
124.359
123.734
123.185
120.137
119.243
117.261
110.579
110.031
77.340
77.022
76.705
37.920
37.391
29.090
9.252
N
H
3e
100
50
0
ppm (t1)

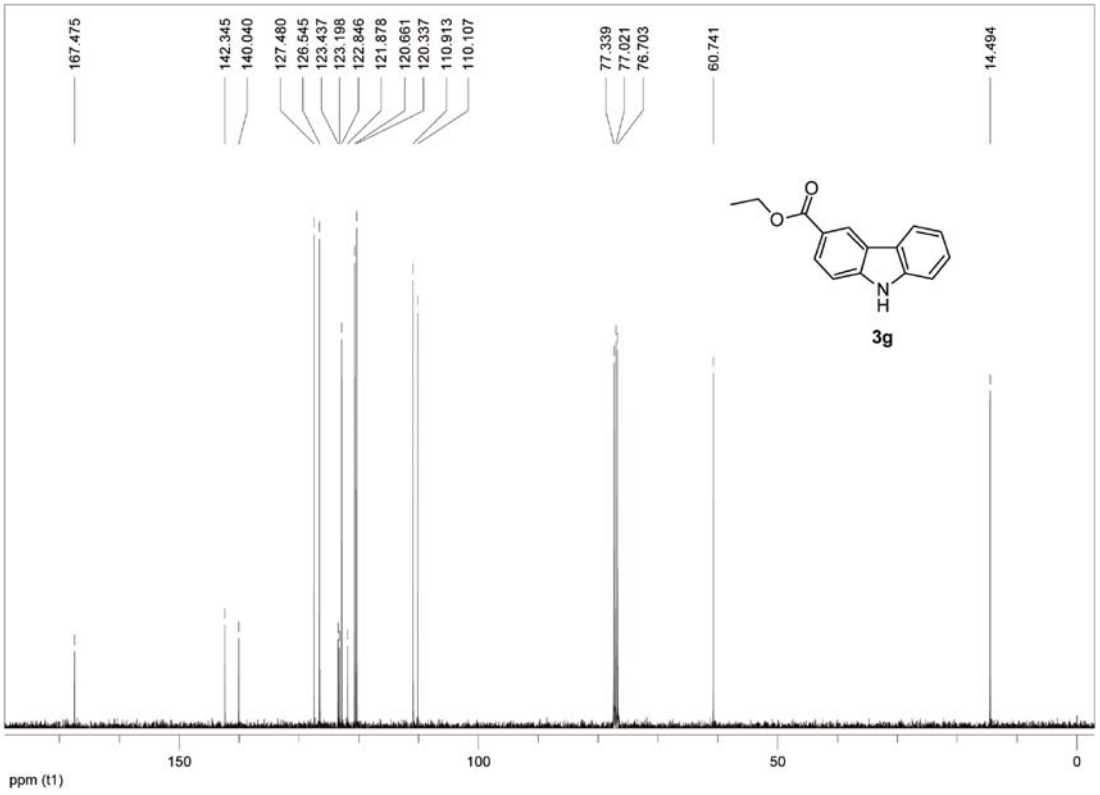

8.295
8.138
8.119
8.081
7.726
7.707
7.690
7.667
7.502
7.480
7.451
7.417
7.363
7.345
7.327
7.260
1.550
0.008
1.00
2.08
3.00
5.34
2.18
N
H
3f
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

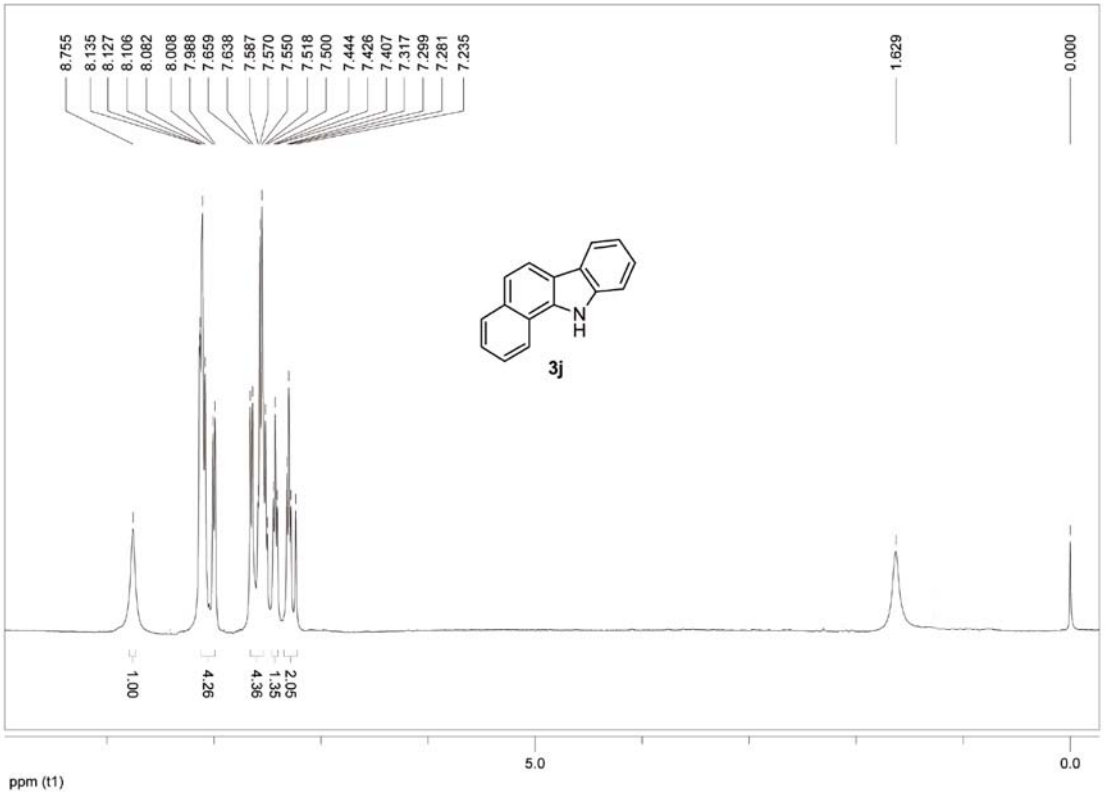

142.198
140.079
139.057
133.171
128.747
127.340
126.490
126.052
125.473
124.004
123.594
120.391
119.645
118.856
110.778
110.736
77.319
77.001
76.684
N
H
150
ppm (t1)
100
50
0

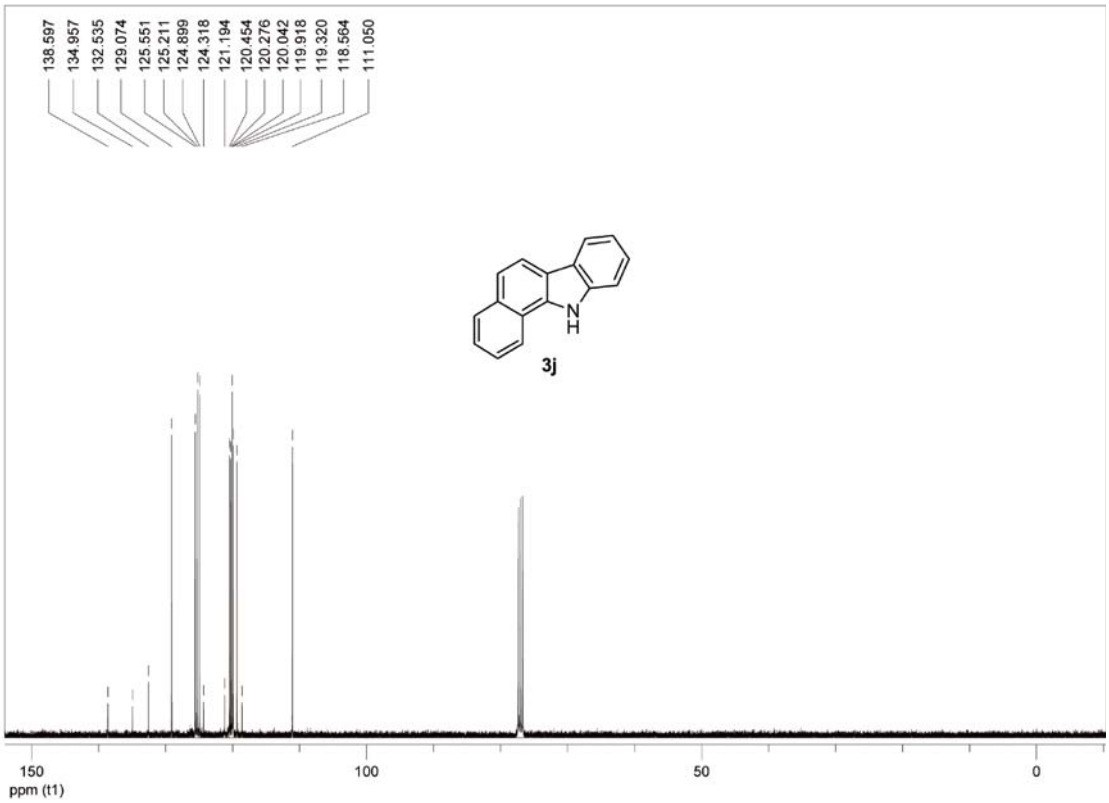
3f

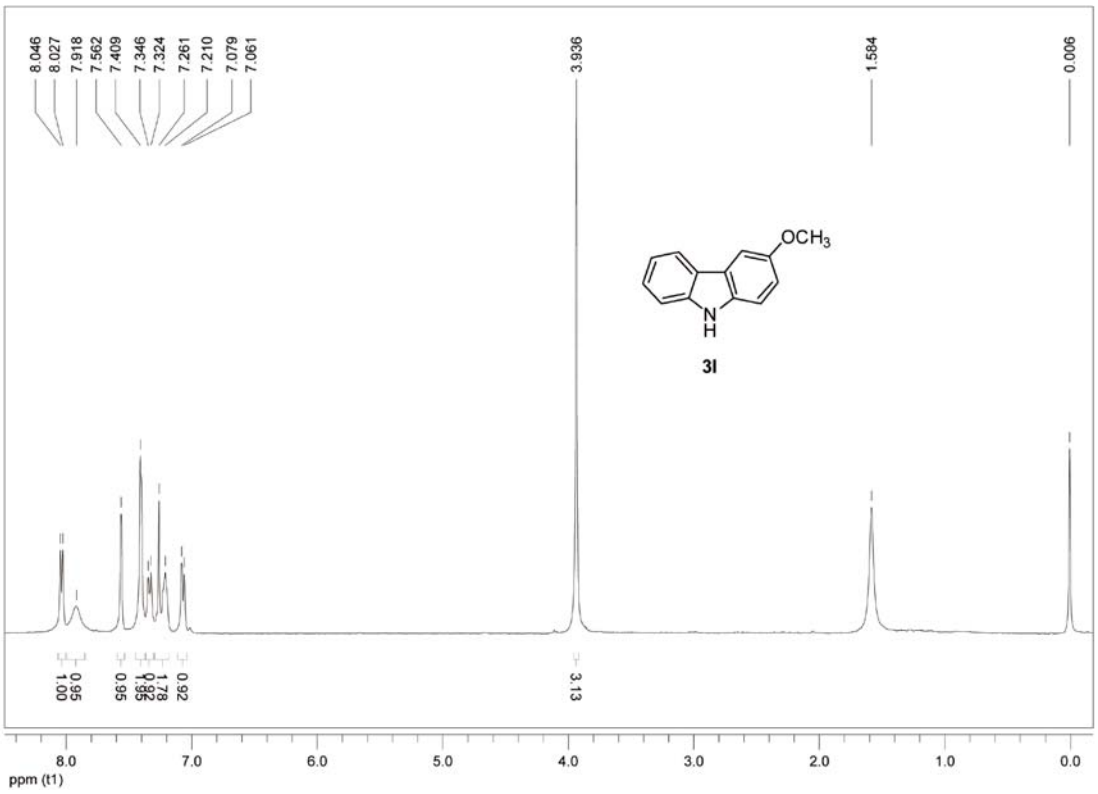

8.820
8.287
8.152
8.135
7.467
7.458
7.446
7.425
7.316
7.306
7.296
7.286
7.260
4.471
4.453
4.435
4.418
1.565
1.474
1.456
1.439
0.003
O
O
N
H
1.00
1.16
2.10
3.16
2.26
2.26
3.17
9.0
ppm (t1)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0

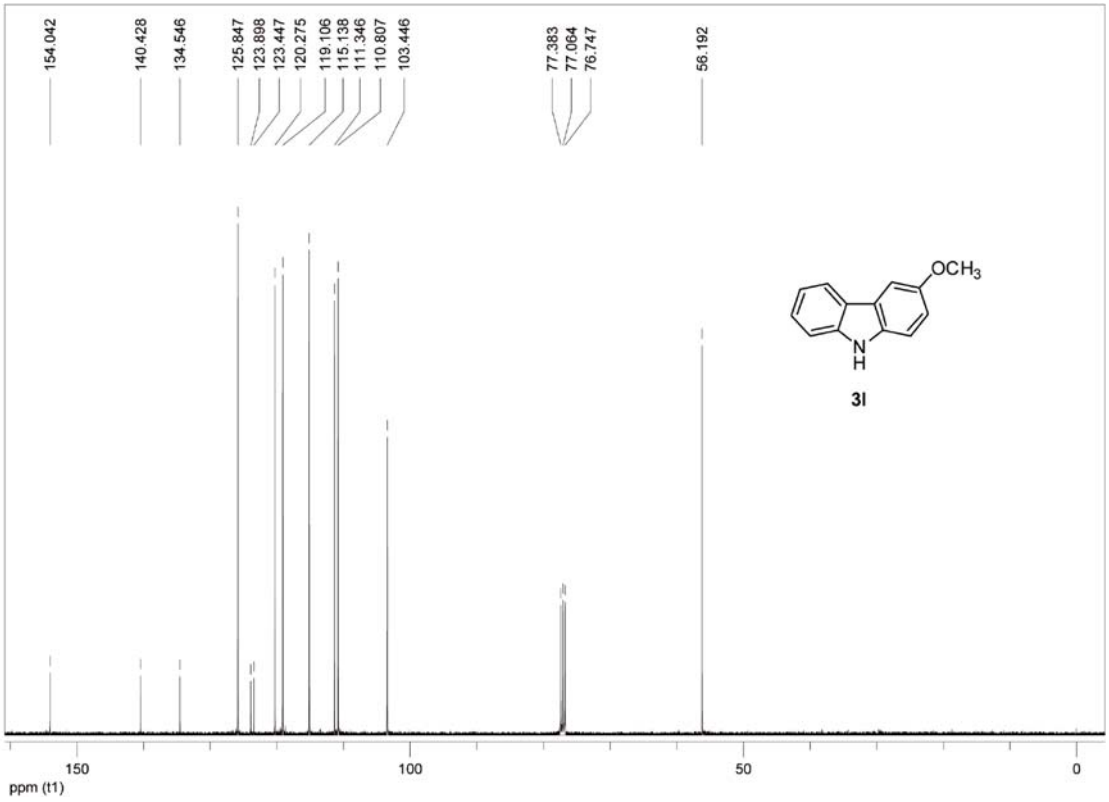
3g

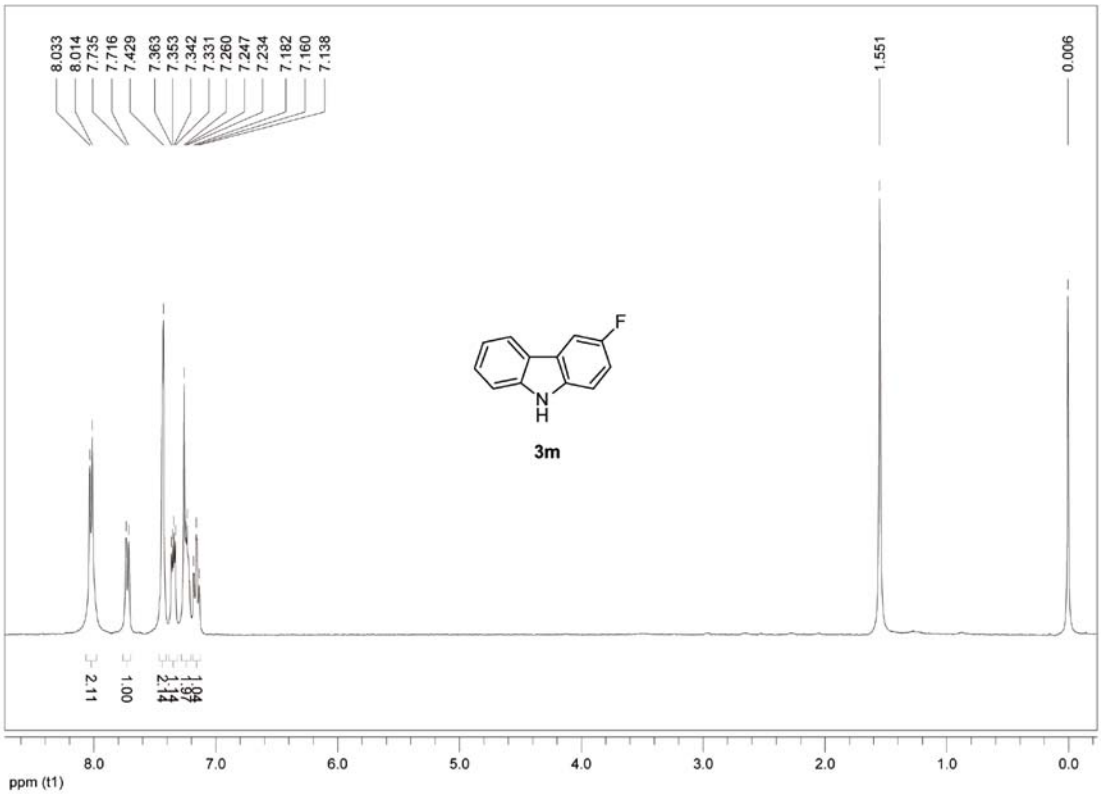

167.475
142.345
140.040
127.480
126.545
123.437
123.198
122.846
121.878
120.661
120.337
110.913
110.107
77.339
77.021
76.703
60.741
14.494
O
O
N
H
3g
150
100
50
0
ppm (t1)

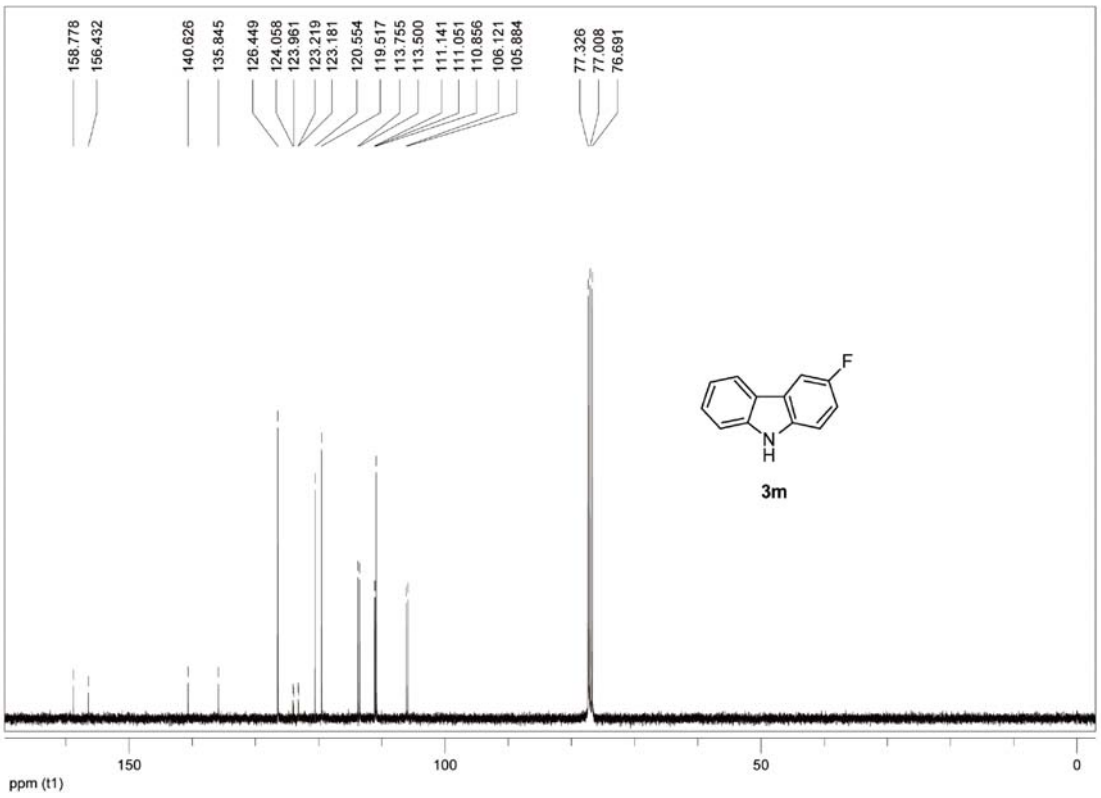

8.755
8.135
8.127
8.106
8.082
8.008
7.988
7.659
7.638
7.587
7.570
7.550
7.518
7.500
7.444
7.426
7.407
7.317
7.299
7.281
7.235
1.629
0.000
N
H
3j
1.00
4.26
4.36
1.35
2.90
5.0
0.0
ppm (t1)

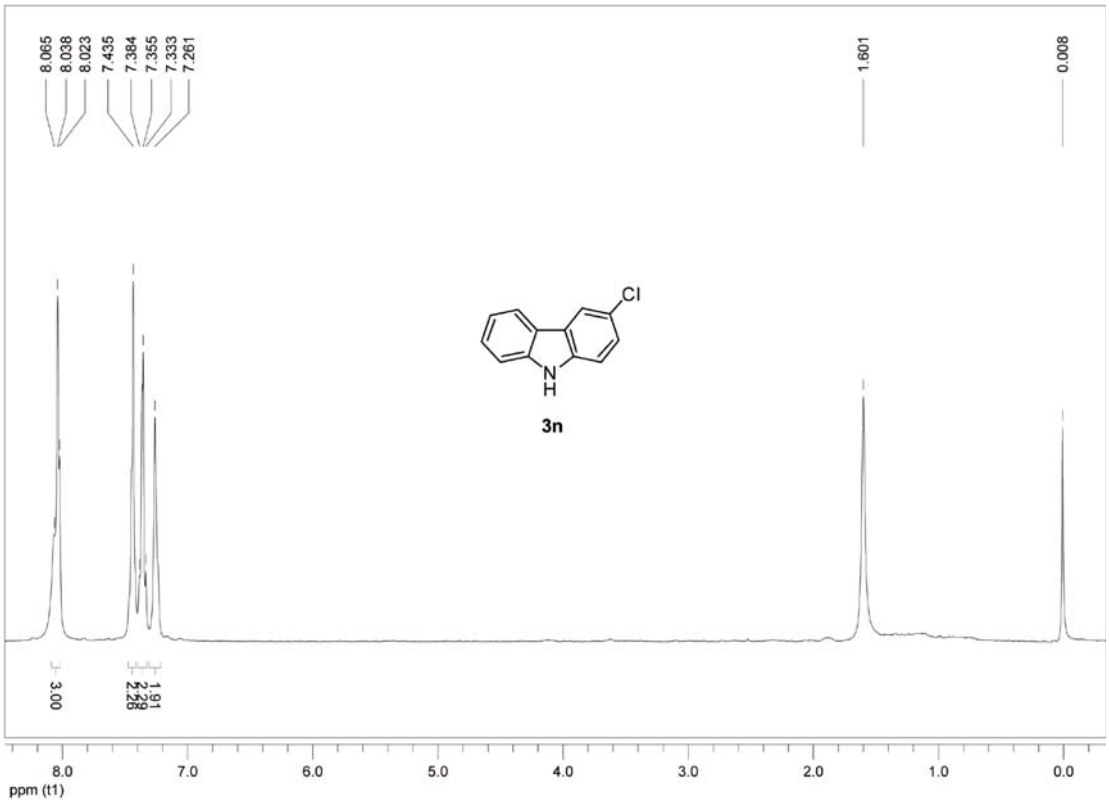

138.597
134.957
132.535
129.074
125.551
125.211
124.899
124.318
121.194
120.454
120.276
120.042
119.918
119.320
118.564
111.050
N
H
3j
150
ppm (t1)
100
50
0

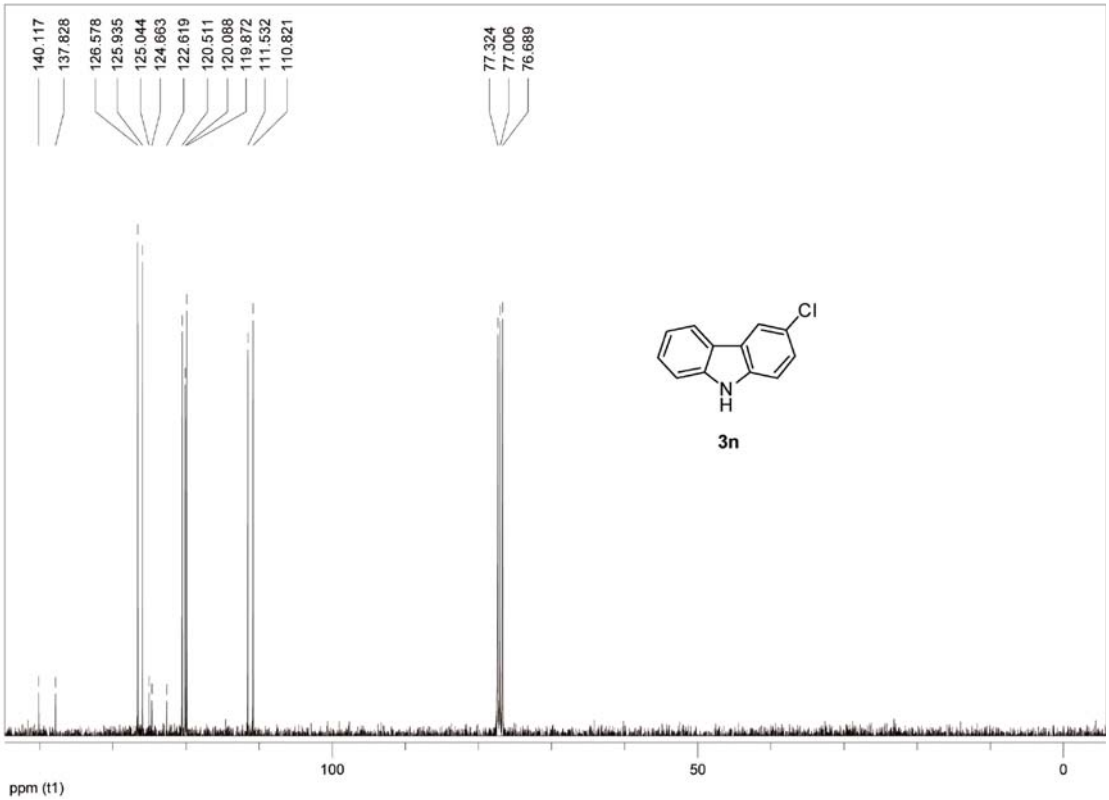

8.046
8.027
7.918
7.562
7.409
7.346
7.324
7.261
7.210
7.079
7.061
3.936
1.584
0.006
OCH3
N
H
3l
0.96
1.00
0.95
1.78
0.92
3.13
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)

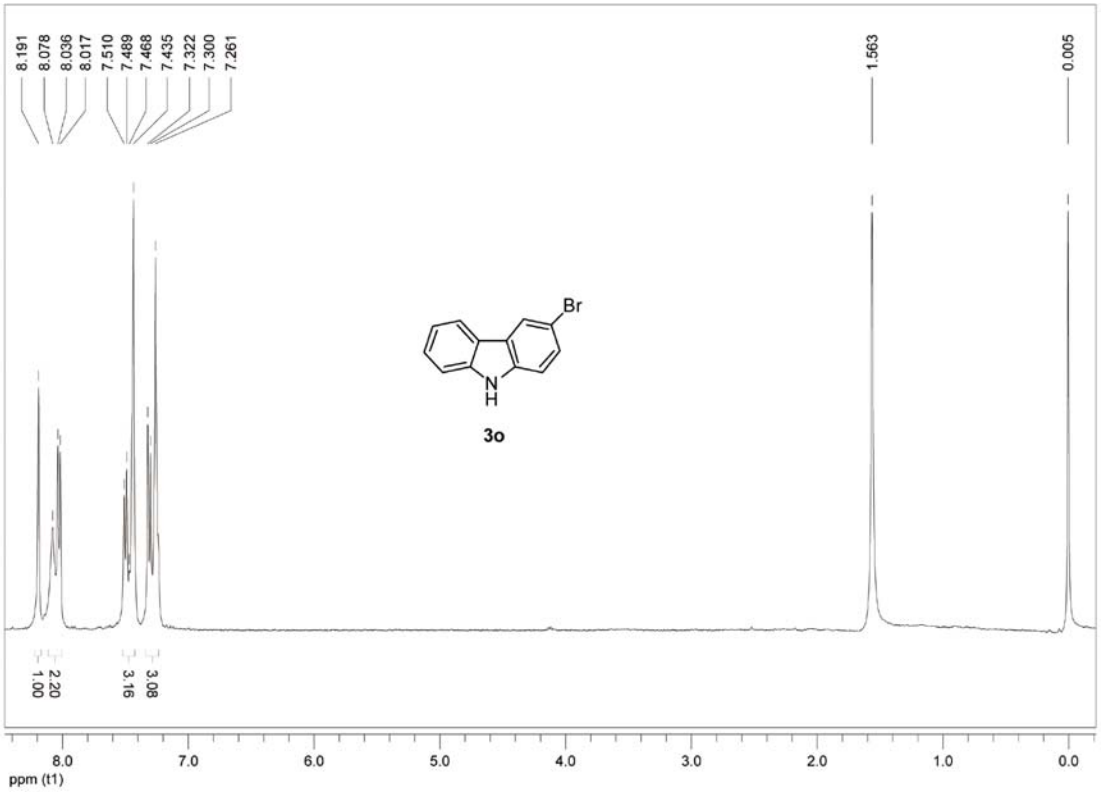

154.042
140.428
134.546
125.847
123.898
123.447
120.275
119.106
115.138
111.346
110.807
103.446
77.383
77.064
76.747
56.192
OCH3
N
H
3l
150
100
50
0
ppm (t1)

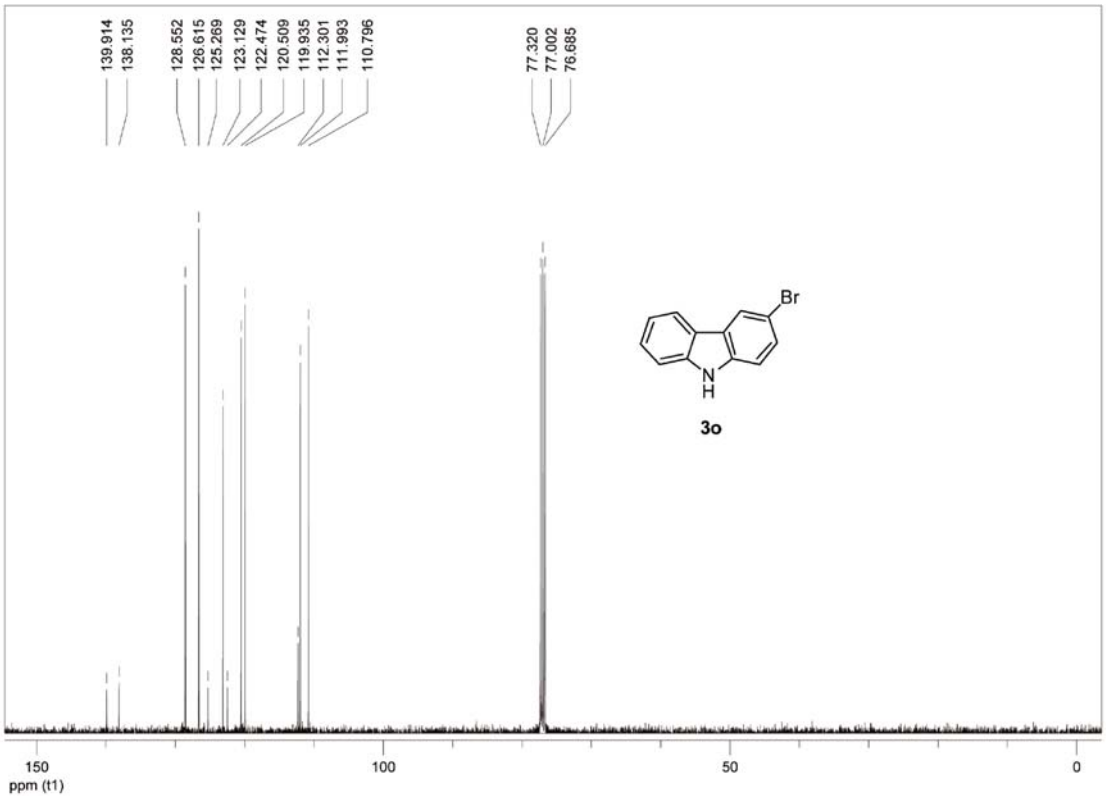


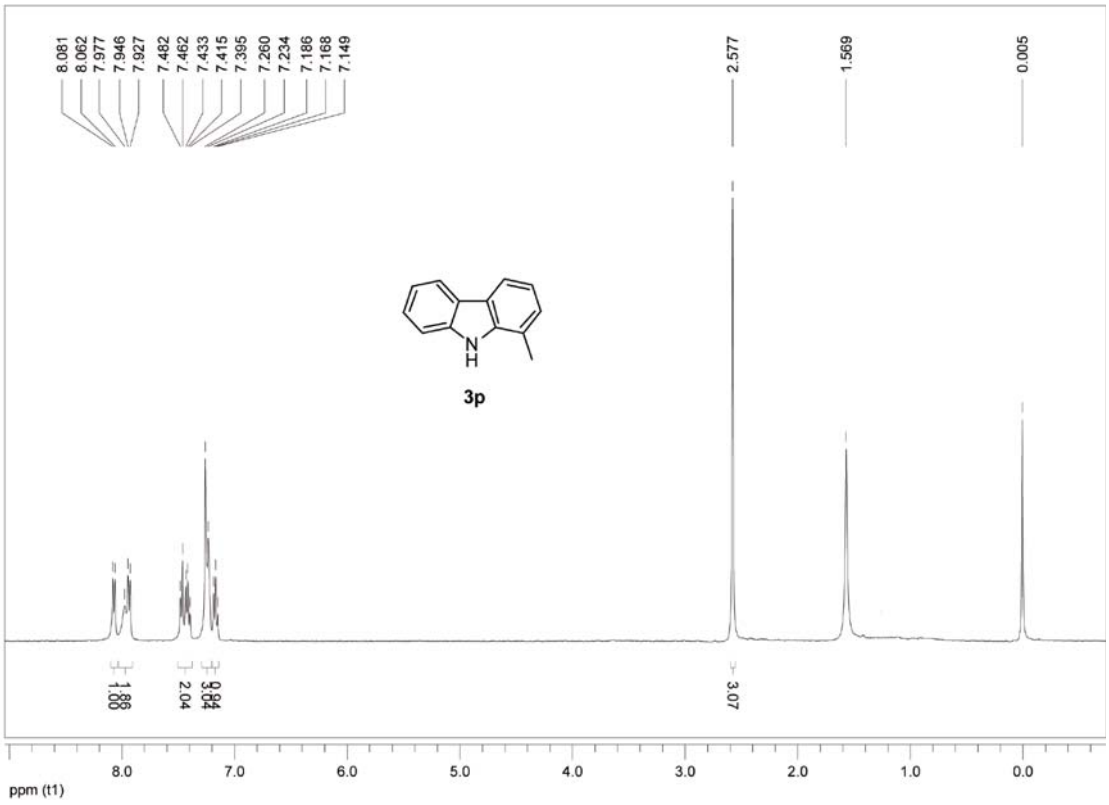


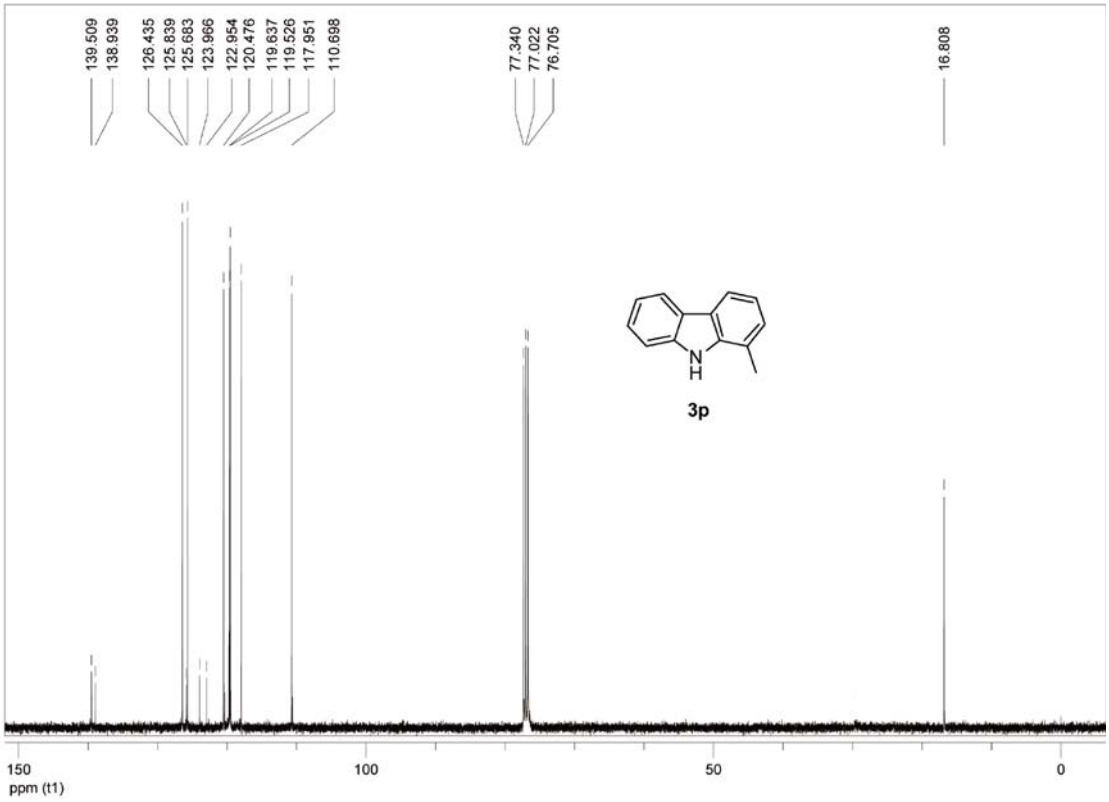





8.191
8.078
8.036
8.017
7.510
7.489
7.468
7.435
7.322
7.300
7.261
1.563
0.005
Br
N
H
3o
1.00
2.20
3.16
3.08
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


139.914
138.135
128.552
126.615
125.269
123.129
122.474
120.509
119.935
112.301
111.993
110.796
77.320
77.002
76.685
Br
N
H
3o
150
100
50
0
ppm (t1)


8.081
8.062
7.977
7.946
7.927
7.482
7.462
7.433
7.415
7.395
7.260
7.234
7.186
7.168
7.149
2.577
1.569
0.005
3p
1.00
1.98
2.04
3.04
3.07
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm (t1)


139.509
138.939
126.435
125.839
125.683
123.966
122.954
120.476
119.637
119.526
117.951
110.698
77.340
77.022
76.705
16.808
3p
150
100
50
0
ppm (t1)