Effect of electronic enrichment of NHCs on the catalytic activity of [Pd(NHC)(acac)Cl] in Buchwald-Hartwig coupling

Gaëtan Le Duc, Sebastien Meiries, Steven P. Nolan*

† EaStCHEM School of Chemistry, University of St. Andrews, St. Andrews, Fife, KY16 9ST, UK.

SUPPORTING INFORMATION

General information

Reagents, reactants and solvents were purchased and used as received unless otherwise stated in the text. Some aryl halides were prepared using literature procedures. LiHMDS, KO^{t}Bu, anhydrous 1,4-dioxane, anhydrous toluene and anhydrous dimethoxyethane (DME) were used as received and stored in a glovebox. KO^{t}Am was prepared by evaporation of the solvent of a commercial solution *in vacuo* and stored under nitrogen. Commercially available $Pd(acac)_2$ was obtained from UMICORE. Gas chromatography was performed on an Agilent Technologies 7890A GC-System equipped with a 7683B series injector and a 7683 series autosampler. Analytical thin-layer chromatography (TLC) was carried out using Merck silica gel 60 F254 glass-backed plates and visualized under UV light (254 and/or 366 nm). Flash column chromatography was performed on silica gel with a 60 Å pore diameter and 40–63 μm particle size. ^{1}H, ^{13}C and ^{19}F nuclear magnetic resonance (NMR) spectra were recorded on a Bruker-300 MHz spectrometer at ambient temperature in $CDCl_3$ without TMSCl as an internal standard. Elemental analyses were performed at London Metropolitan University (166-220 Holloway Road, London, N7 8DB, U.K.).

General Procedure for Buchwald-Hartwig Cross Coupling of Aryl Halides with Anilines

In a glovebox, a glass vial equipped with a stirring bar was charged with KO^{t}Am (0.55 mmole) and sealed with a screw cap fitted with a septum. The vial was then loaded with the neat aniline (0.55 mmole) and the aryl halide (0.50 mmole) outside the glovebox. Finally, a pre-made solution of the precatalyst in anhydrous solvent (prepared in the glovebox) was injected at room temperature under nitrogen and the reaction mixture was stirred and heated until completion, as indicated by GC. Then, water was added to the reaction mixture and the organic layer was extracted with Et_2O, dried over magnesium sulfate and the solvent was evaporated *in vacuo*. The product was purified by flash chromatography on silica gel.

(*N*-4-fluorophenyl)-4'-methoxy-aniline (Table 3, Entry 1). The general procedure yielded (103 mg, 95 %) of the pure arylamine as beige solid. Data were in full agreement with those reported in literature.[1] **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.01 (d, *J* = 8.8, 2H), 6.95-6.83 (m, 6H), 5.38 (bs, 1H), 3.79 (3H, s). **^{13}C NMR (100 MHz, $CDCl_3$)** δ 157.1 (d, *J* = 238 Hz), 155.0, 141.09, 136.5, 121.2, 117.7 (d, *J* = 8 Hz), 115.8 (d, *J* = 22 Hz), 114.7, 55.6. **^{19}F NMR (282 MHz, $CDCl_3$)** δ -124.7.

4-fluoro-*N*-(2,6-dimethylphenyl)aniline (Table 3, Entry 2). The general procedure yielded (98 mg, 91 %) of the pure arylamine as a white solid. Data were in full agreement with those reported in literature.[2] **^{1}H NMR (400 MHz, $CDCl_3$)** δ 7.14-7.05 (m, 3H), 6.86 (tt, *J* = 8.8 Hz, 2.3 Hz, 2H), 6.47 (m, 2H), 5.20 (bs, 1H), 2.21 (s, 6H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 156.3 (d, *J* = 240 Hz, C-F), 142.4, 138.5, 135.4, 128.6, 125.7, 115.6 (d, *J* = 22 Hz), 114.5 (d, *J* = 7 Hz), 18.3. **^{19}F NMR (282 MHz, $CDCl_3$)** δ – 126.7.

(*N*-4-fluoro)-2-methoxy-aniline (Table 3, Entry 3) The general procedure yielded (104 mg, 96 %) of the pure arylamine as a yellow oil. **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.17-7.09 (m, 3H), 7 (t, *J* = 8.3 Hz, 2H),

6.91-6.82 (m, 3H), 6.08 (bs, 1H), 3.91 (s, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 158.0 (d, *J* = 238 Hz), 156.6, 147.9, 138.6 (d, *J* = 2 Hz), 133.8, 121.3 (d, *J* = 8 Hz), 120.9, 119.6, 115.9 (d, *J* = 22 Hz), 113.7, 110.5, 55.6. **^{19}F NMR (282 MHz, $CDCl_3$)** δ -122.4.

2-fluoro-*N*-(2,6-dimethylphenyl)aniline (Table 3, Entry 4). The general procedure yielded (99 mg, 92 %) of the pure arylamine as a white solid. Data were in full agreement with those reported in literature.[3] **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.19-7.16 (m, 3H), 7.10 (ddd, *J* = 11.9 Hz, 8.7 Hz, 1.4 Hz, 1H), 6.90 (ddd, *J* = 15.6 Hz, 2.1 Hz, 1.0 Hz, 1H), 6.68 (m, 1H) 6.25 (ddd, *J* = 9.4 Hz, 7.9 Hz, 1.6 Hz, 1H), 5.38 (bs, 1H), 2.27 (s, 6H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 151.5 (d, *J* = 237 Hz), 137.2, 136.4, 134.6 (d, *J* = 11 Hz), 128.6, 126.3, 124.4 (d, *J* = 3 Hz), 117.4 (d, *J* = 7 Hz), 114.7 (d, *J* = 18 Hz), 113.2 (d, *J* = 3 Hz), 18.2. **^{19}F NMR (282 MHz, $CDCl_3$)** δ -137.0.

(N-dimethoxyphenyl)-N-methyl-aniline (Table 3, Entry 5). The general procedure yielded (112 mg, 92 %) of the pure arylamine as white solid. Data were in full agreement with those reported in literature.[4] **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.22 (t, *J* = 8.3 Hz, 1H), 7.14 (m, 2H), 6.67 (dt, *J* = 7.3 Hz, 1 Hz, 3H), 6.62 (d, *J* = 8.4 Hz, 2H), 6.54 (m, 2H), 3.74 (s, 6H), 3.19 (s, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 158.0, 149.0, 128.7, 127.4, 124.2, 116.1, 111.7, 105.0, 56.0, 38.1.

(*N*-2,6-dimethoxyphenyl)-2,6-dimethyl-aniline (Table 3, Entry 6)**.** The general procedure yielded (102 mg, 79 %) of the pure arylamine as a white solid. Data were in full agreement with those reported in literature.[4] **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.00-6.90 (m, 3H), 6.78 (t, *J* = 8.4 Hz, 1H), 6.56 (d, *J* = 8.1 Hz, 2H), 4.65 (bs, 1H), 3.64 (s, 6H), 2.17 (s, 6H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 149.5, 141.5, 133.9, 127.5, 125.8, 123.5, 118.2, 105.7, 56.3, 18.8.

***N*-(2,6-dimethoxyphenyl)aniline** (Table 3, Entry 7). The general procedure yielded (85 mg, 74 %) of the pure arylamine as a white solid. Data were in full agreement with those reported in literature.[5] **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.20-7.07 (m, 3H), 6.83 (t, *J* = 7.5Hz, 1H), 6, 74 (d, *J* = 8.4Hz, 2H), 6.64 (d, *J* = 8.4 Hz, 2H), 4.80 (bs, 1H) 3.82 (s, 6H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 154.0, 145.4, 128.5, 124.3, 119.7, 115.1, 104.4, 55.8.

(*N*-4-methoxyphenyl)-3-trifluoromethyl-aniline (Table 3, Entry 8)**.** The general procedure yielded (122 mg, 91 %) of the pure arylamine as a brown solid. Data were in full agreement with those reported in literature.[6] **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.30 (t, *J* = 8.0 Hz, 1H), 7.10 (d, *J* = 8.5 Hz, 2H), 7.00 (m, 3H), 6.92 (dt, *J* = 8.5 Hz, *J* = 2.5Hz, 2H), 5.61 (bs, 1H), 3.84 (s, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 152.2, 146.0, 134.3, 131.7 (q, *J* = 31 Hz), 129.8, 124.2 (q, *J* = 270 Hz), 123.5, 117.9, 115.6 (q, *J* = 4 Hz), 114.9, 111.3 (q, *J* = 4Hz), 55.6. **^{19}F NMR (282 MHz, $CDCl_3$)** δ – 63.2.

(*N*-2-methoxyphenyl)-3-trifluoromethyl-aniline (Table 3, Entry 9)**.** The general procedure yielded (118 mg, 88 %) of the pure arylamine as colourless oil. Data were in full agreement with those reported in literature.[7] **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.39-7.29 (m, 4H), 7.15 (d, *J* = 7.5 Hz, 1H), 6.98-6.89 (m, 3H), 6.25 (bs, 1H), 3.90 (s, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 148.9, 143.6, 131.5 (q, *J* = 32 Hz), 131.4, 129.7, 124.1 (q, *J* = 271 Hz), 121.3, 120.8, 120.1, 116.9 (q, *J* = 4 Hz), 116.0, 113.8 (q, *J* = 4 Hz), 110.8, 55.5. **^{19}F NMR (282 MHz, $CDCl_3$)** δ – 63.3.

(*N*-4-fluorophenyl)-2,6-dimethoxy-aniline (Table 3**,** Entry 10)**.** The general procedure yielded (110 mg, 90 %) of the pure arylamine as a white solid. **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.11 (t, *J* = 7.3 Hz, 1H), 6.88 (m, 2H), 6.68-6.63 (m, 2H), 6.63 (d, *J* = 8.3 Hz, 2H), 3.83 (s, 6H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 157.0

(d, *J* = 234 Hz, C-F), 153.6, 141.4, 124.2, 120.0, 116.7 (d, J = 7.5 Hz), 114.9 (d, *J* = 22 Hz), 104.4, 55.8. **^{19}F NMR (282 MHz, $CDCl_3$)** δ = -126.2. **Anal. Calcd for $C_{14}H_{14}FNO_2$**: C, 68.00; H, 5.71; N, 5.66. Found: C, 68.05; H, 5.76; N, 5.67.

(*N*-2,4,6-trimethoxyphenyl)-2,6-dimethyl-aniline (Table 3, Entry 11). The general procedure yielded (119 mg, 83%) of the pure arylamine as a white solid. **^{1}H NMR (300 MHz, $CDCl_3$)** δ 6.95-6.79 (m, 3H), 6.16 (s, 2H), 4.86 (bs, 1H), 3.79 (s, 3H), 3.65 (s, 6H), 2.12 (s, 6H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 154.6, 152.2, 142.8, 131.4, 127.9, 121.9, 118.6, 92.1, 56.2, 55.6, 18.7**. Anal. Calcd** for $C_{17}H_{21}NO_3$: C, 71.06; H, 7.37; N, 4.87. Found: C, 71.13; H, 7.37; N, 4.87.

N-Methyl-N-2-tolylaniline (Table 4, Entry 2). The general procedure yielded (90 mg, 91 %) of the pure arylamine as colourless oil. Data were in full agreement with those reported in literature.[8] **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.30-7.13 (m, 6H) 6.71 (t, *J* = 7.2 Hz, 1H), 6.53 (d, *J* = 7.8 Hz, 2H), 3.22 (s, 3H), 2.15 (s, 3H)**. ^{13}C NMR (75 MHz, $CDCl_3$)** δ 149.1, 146.8, 136.8, 131.4, 129.0, 128.3, 127.5, 116.4, 112.8, 39.0, 17.8.

N-Methyl-N-3-tolylaniline (Table 4, Entry 3). The general procedure yielded (83 mg, 84%) of the pure arylamine as yellow oil. Data were in full agreement with those reported in literature.[8] **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.65-7.60 (m, 2H), 7.52 (t, J = 7.8 Hz, 1H), 7.37-7.27 (m, 3H), 7.21-7.41 (m, 3H), 3.65 (s, 3H), 2.66 (s, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 149.1, 149.0, 139.0, 129.1, 129.0, 122.3, 121.4, 120.9, 120.1, 117.9, 40.2, 21.5.

N-Methyl-N-4-tolylaniline (Table 4, Entry 4). The general procedure yielded (83 mg, 84%) of the pure arylamine as yellow oil. Data were in full agreement with those reported in literature.[9] **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.33-7.28 (m, 2H), 7.19 (d, *J* = 8.4 Hz, 2H), 7.07 (d, J = 8.4 Hz, 2H), 7.02-6.92 (m, 3H), 3.36 (s, 3H), 2.40 (s, 3H)**. ^{13}C NMR (75 MHz, $CDCl_3$)** δ 149.3, 146.5, 132.0, 129.9, 129.0, 122.5, 119.8, 118.2, 40.3, 20.7.

4-methoxy-N-Methyl-N-phenylaniline (Table 4, Entry 5). The general procedure yielded (89 mg, 83%) of the pure arylamine as yellow oil. Data were in full agreement with those reported in literature.[10] **^{1}H NMR (300 MHz, $CDCl_3$)** δ 7.62-7.68 (m, 2H), 7.53-7.56 (m, 2H), 7.33-7.36 (m, 2H), 7.21-7.26 (m, 3H), 4.26 (s, 3H), 3.71 (s, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 156.2, 149.7, 142.15, 128.9, 126.1, 118.3, 115.7, 114.7, 55.4, 40.4

1. Fors, B. P.; Watson, D. A.; Biscoe, M. R.; Buchwald, S. L. *J. Am. Chem. Soc.*, **2008**, *130*, 13552-13554.

2. Seipel, K. R.; Platt, Z. H.; Nguyen, M., Holland, A. W. *J. Org. Chem.*, **2008**, *73*, 4291-4294.

3. Liang, L.,-C.; Huang, M.-H.; Hung, C.-H. *Inorg. Chem.*, **2004**, *43*, 2166-2174.

4. Meiries S.; Le Duc G.; Chartoire A.; Collado A.; Speck K.; Athukorla Arachchige K. S.; Slawin A. M. Z.; Nolan S. P. *Chem. Eur. J.* DOI: 10.1002/chem.201302471.

5. Ishii, H.; Sugiura, T.; Akiyama, Y.; Ichikawa, Y.; Watanabee, T.; Murakami, Y. *Chem. Pharm. Bull*, **1990**, *38*, 2118-2126.

6. Biscoe M. R.; Fors, B. P.; Buchwald, S. L. *J. Am. Chem. Soc.*, **2008**, *130*, 6686-6687. Nokamura, Y.; Ilies, L.; Nakamura, E. *Org. Lett.*, **2011**, *13*, 5998-6001.

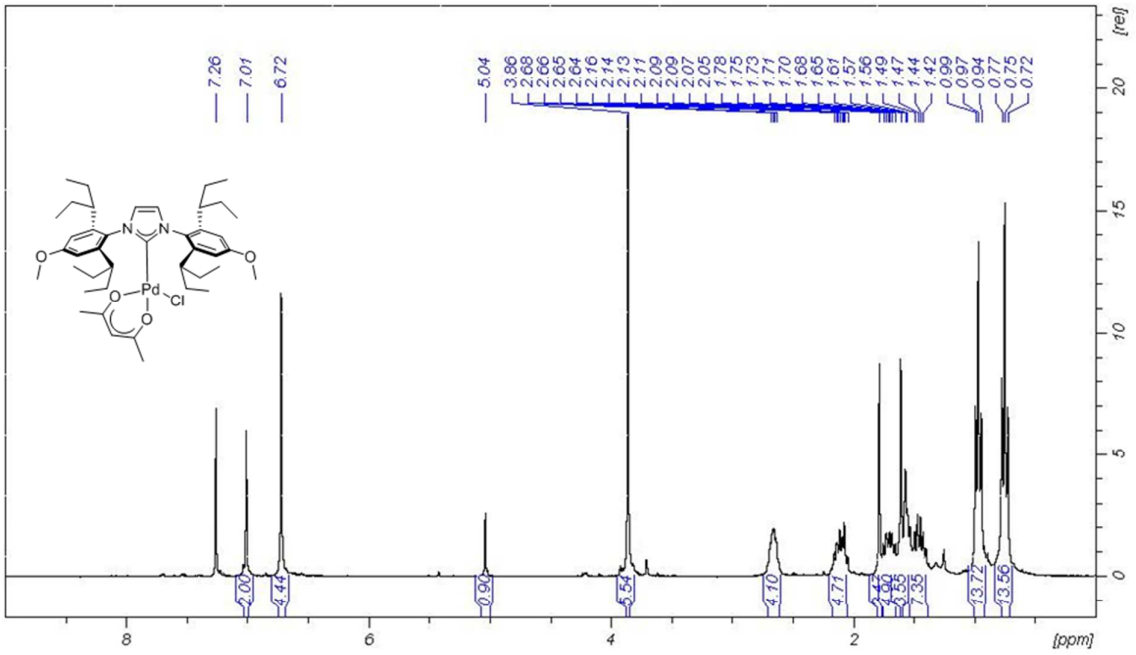
7. Reddy, C. V.; Kingston, J. V.; Verkade, J. G. *J. Org. Chem.*, **2008**, *73*, 3047-3062.

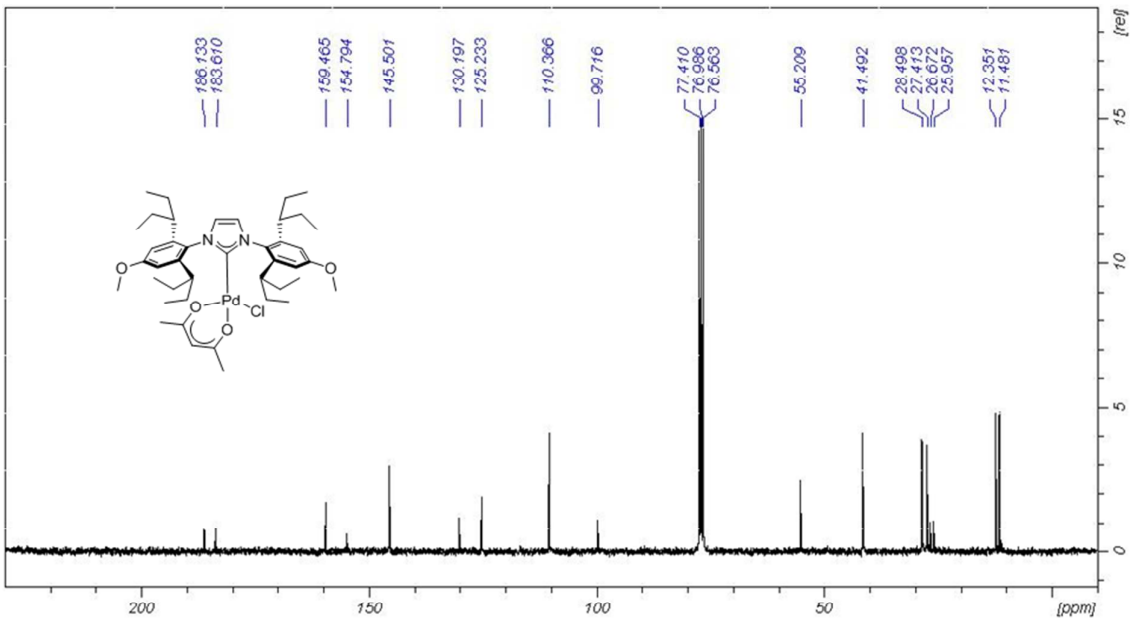
8. Rataboul F., Zapf A., Jackstell R., Harkal S., Riermeier T., Monsees A., Dingerdissen U., Beller M. *Chem. Eur. J.*, **2004**, *10*, 2983-2990.

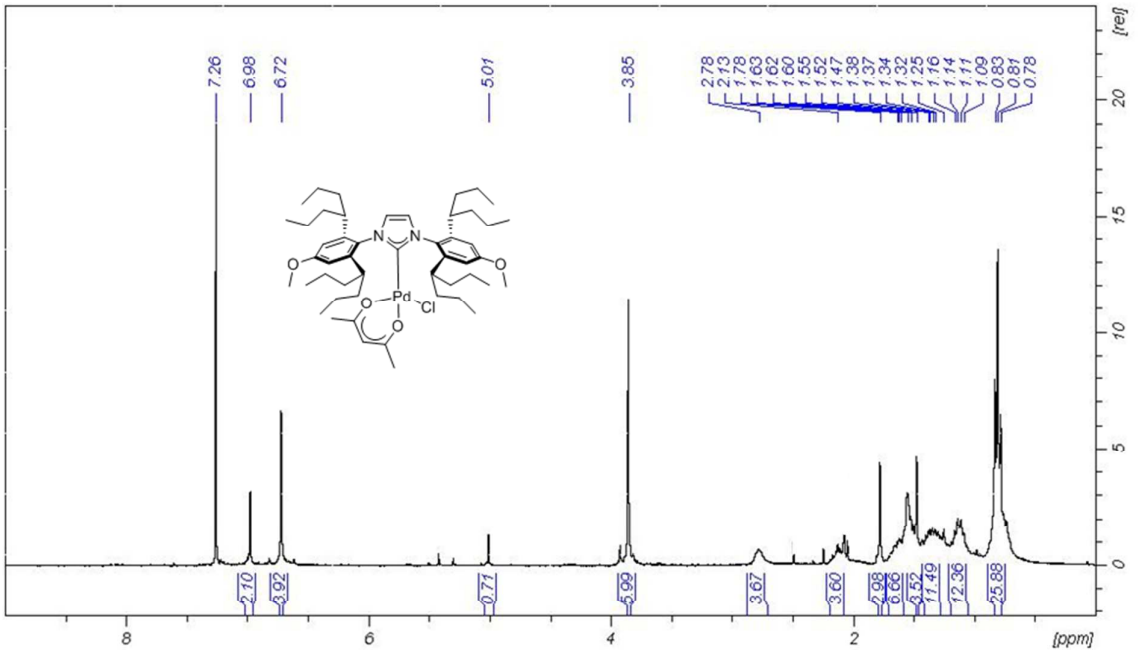
9. Park S.-E. , Beom Kang S., Jung K-J. , Won J-E. , Lee S.-G,Yoon Y-J. Synthesis, **2009**, *5*, 815 – 823.

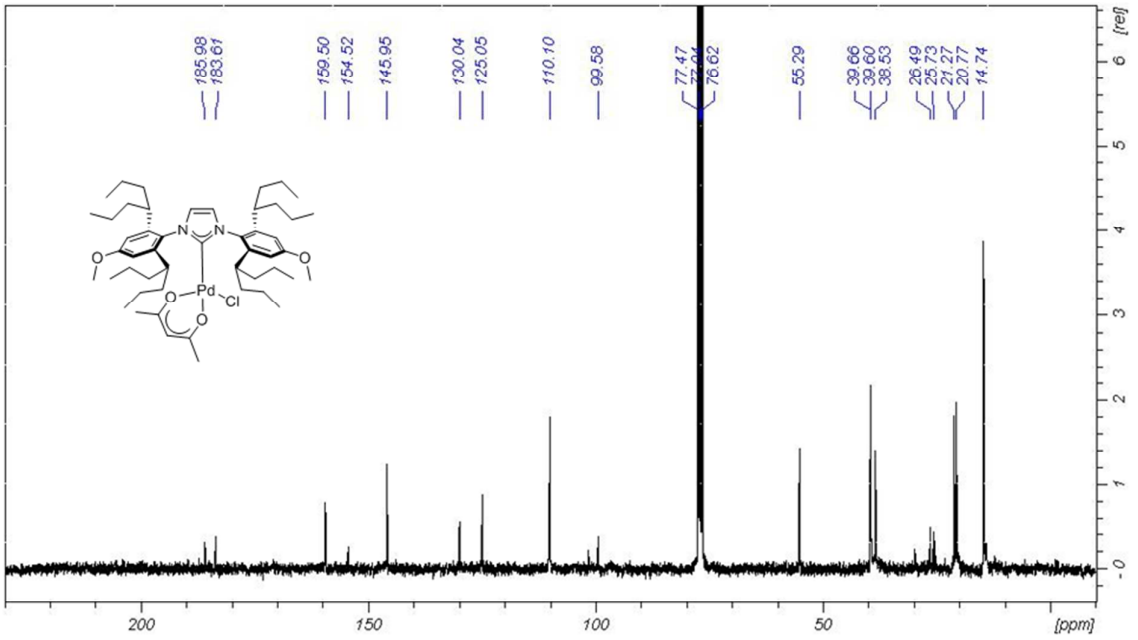
10. (a) MacNeil S. L., Wilson B. J., Snieckus V. *Org. Lett.*, **2006**, *8*, 1133–1136. (b) Carroll M. A., Wood R. A. *Tetrahedron*, **2007**, 11349–11354.

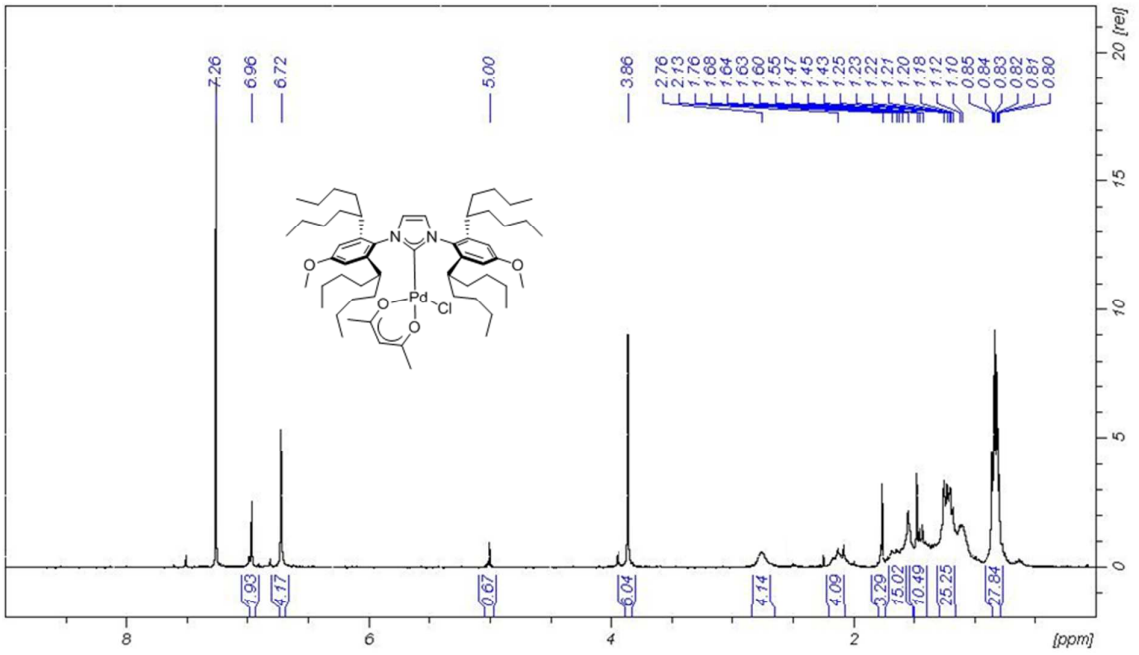
^{1}H NMR and ^{13}C NMR Spectra of Pd(ITent^{OMe})(acac)Cl complexes

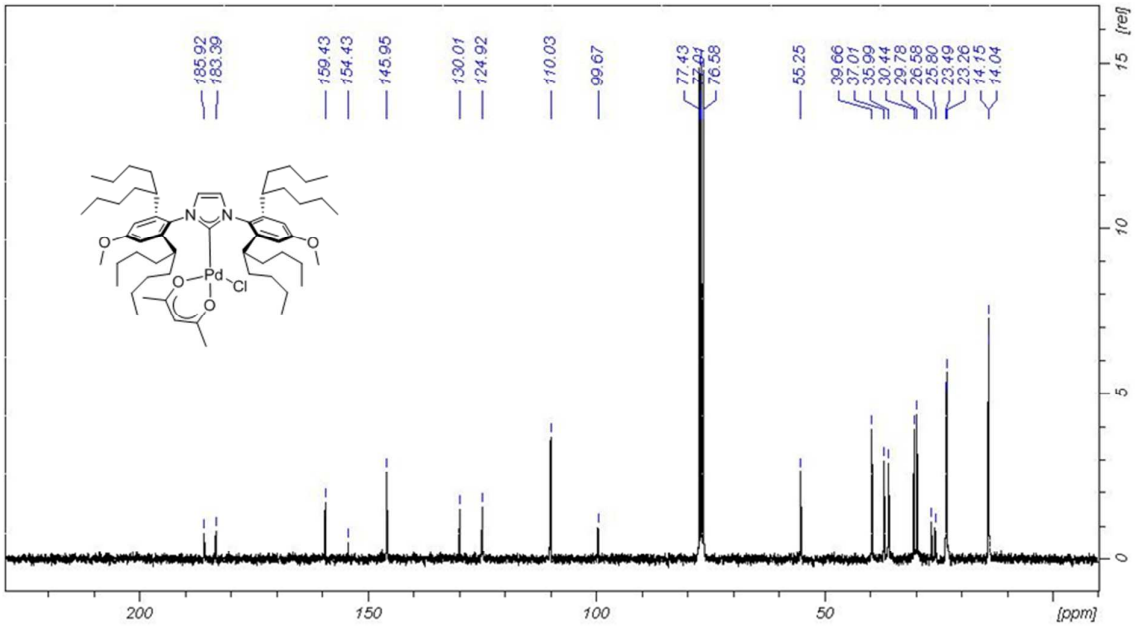




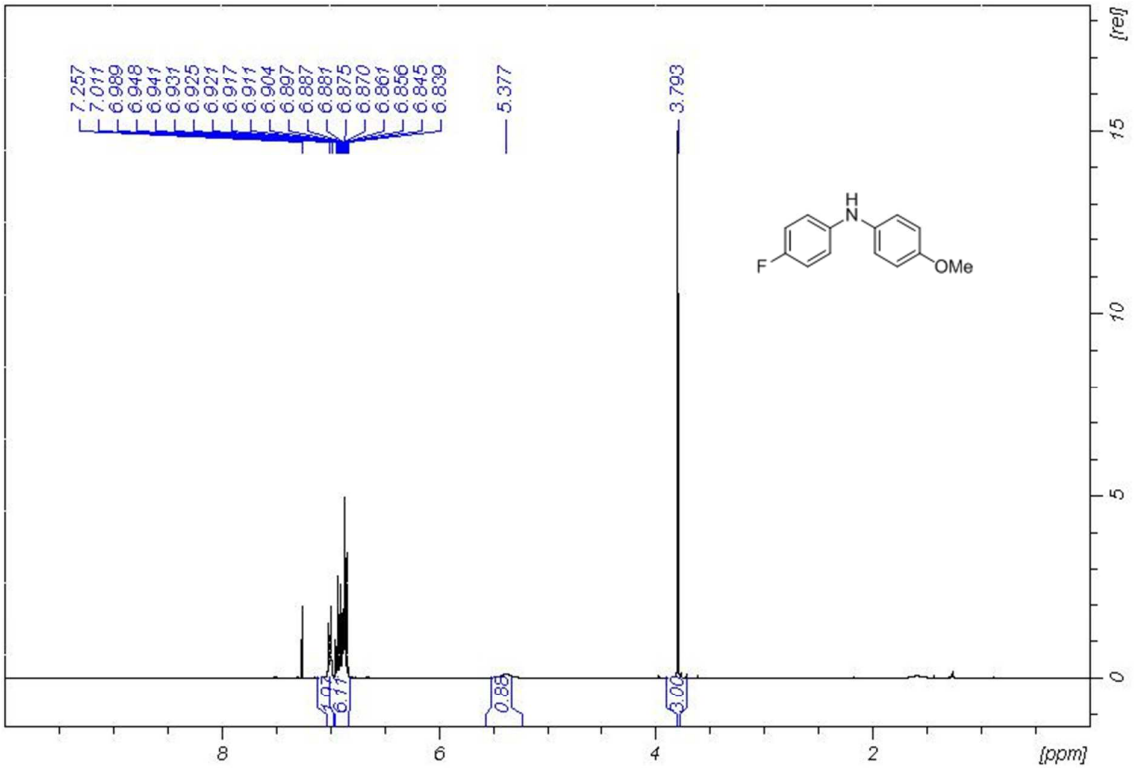

7.26
6.98
6.72
5.01
3.85
2.78
2.13
1.78
1.63
1.62
1.60
1.55
1.52
1.47
1.38
1.37
1.34
1.32
1.25
1.16
1.14
1.11
1.09
0.83
0.81
0.78
2.10
3.92
0.71
5.99
3.67
3.60
2.98
6.66
3.52
11.49
12.06
25.88
[rel]
[ppm]

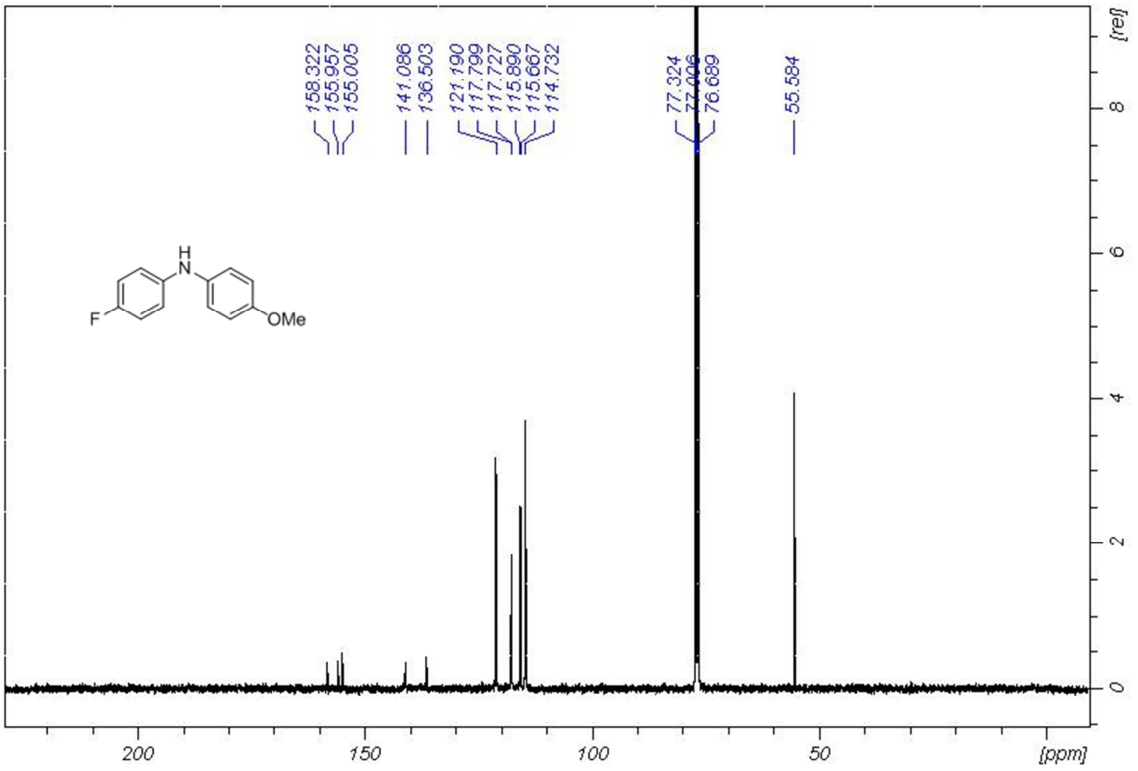

185.98
183.61
159.50
154.52
145.95
130.04
125.05
110.10
99.58
77.47
77.04
76.62
55.29
39.66
39.60
38.53
26.49
25.73
21.27
20.77
14.74
[rel]
[ppm]

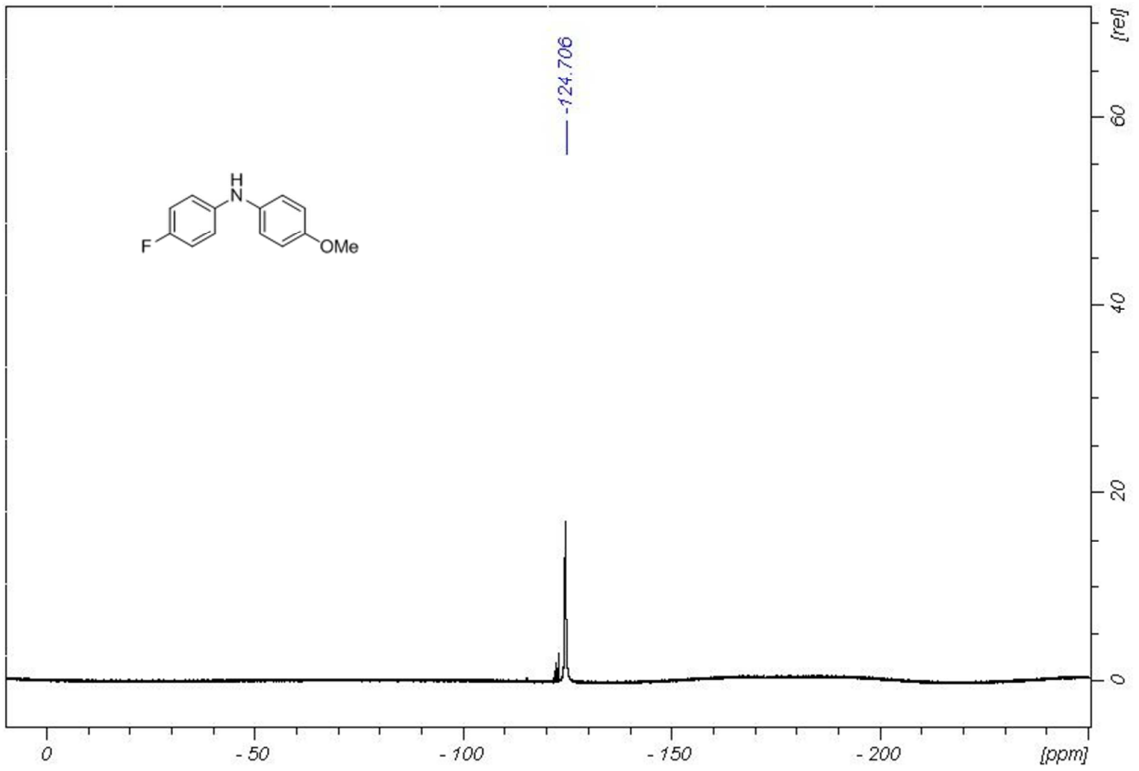

7.26
6.96
6.72
5.00
3.86
2.76
2.13
1.76
1.68
1.64
1.63
1.60
1.55
1.47
1.45
1.43
1.25
1.23
1.22
1.21
1.20
1.18
1.12
1.10
0.85
0.84
0.83
0.82
0.81
0.80
1.93
4.17
0.67
6.04
4.14
4.09
3.29
15.02
10.49
25.25
27.84
[ppm]
[rel]

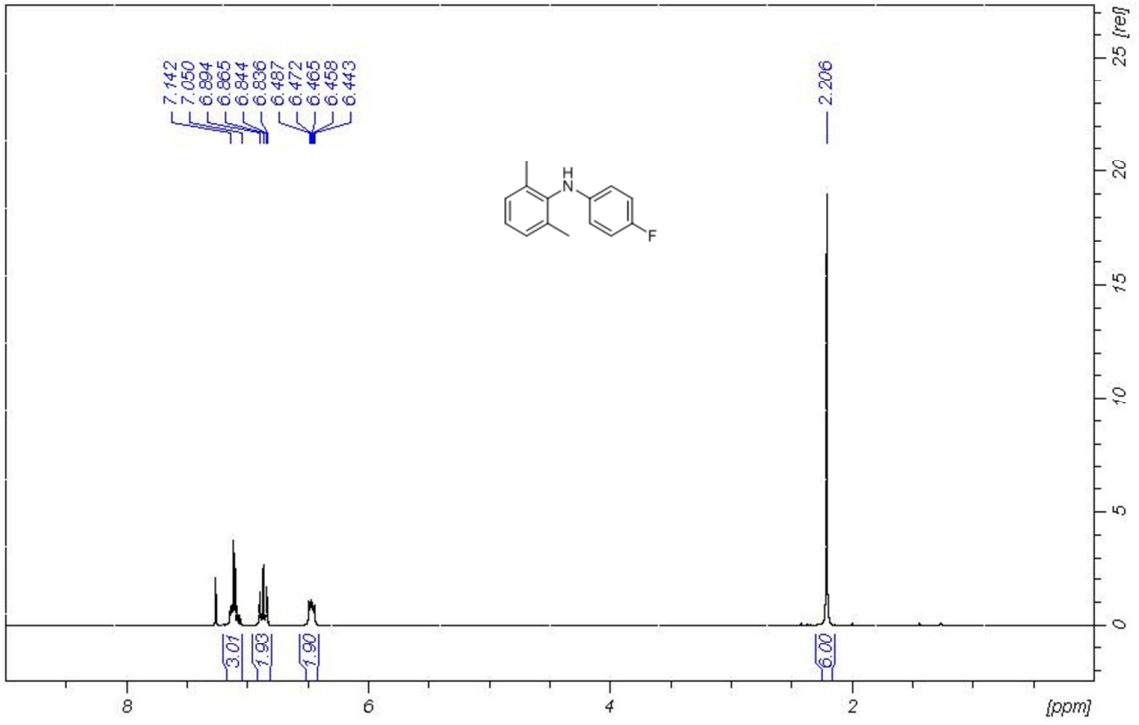

185.92
183.39
159.43
154.43
145.95
130.01
124.92
110.03
99.67
77.43
77.04
76.58
55.25
39.66
37.01
35.99
30.44
29.78
26.58
25.80
23.49
23.26
14.15
14.04
[ppm]
[rel]

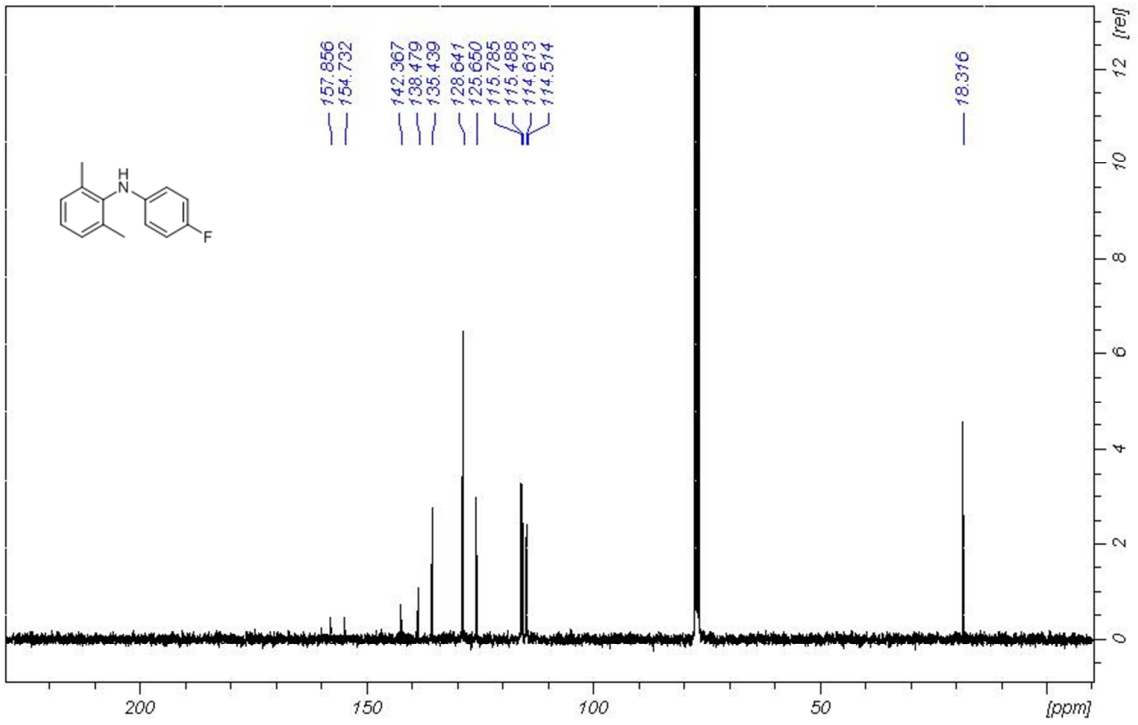
^{1}H NMR, ^{13}C NMR and ^{19}F NMR Spectra of arylamination products

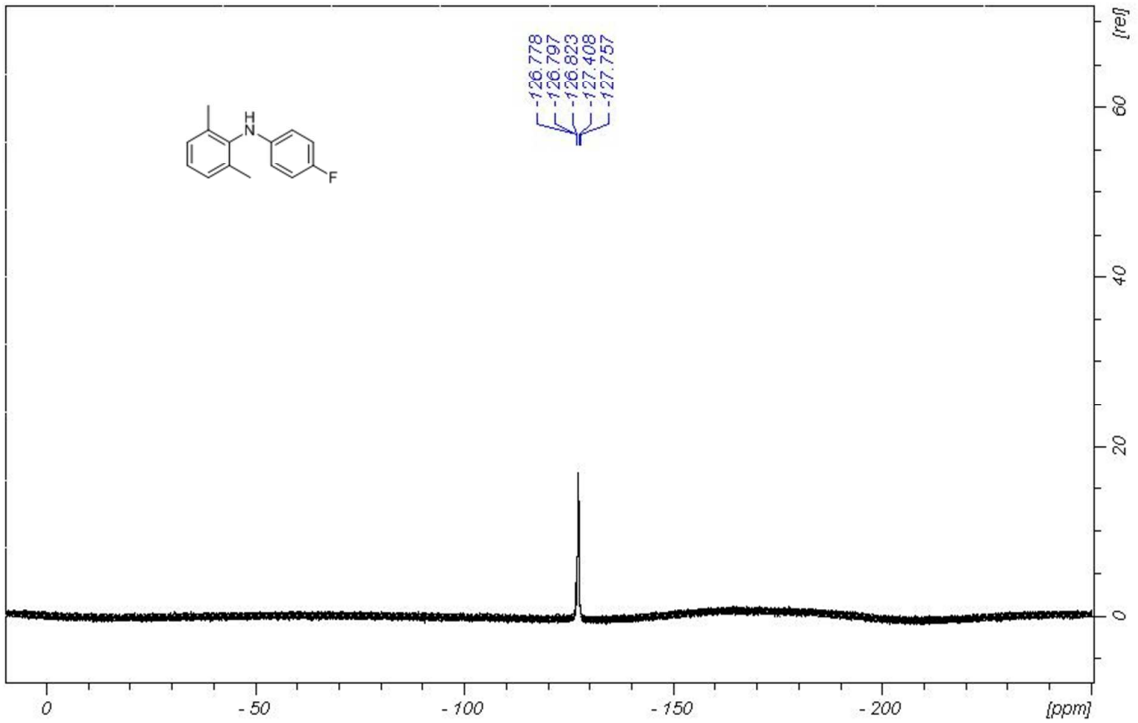


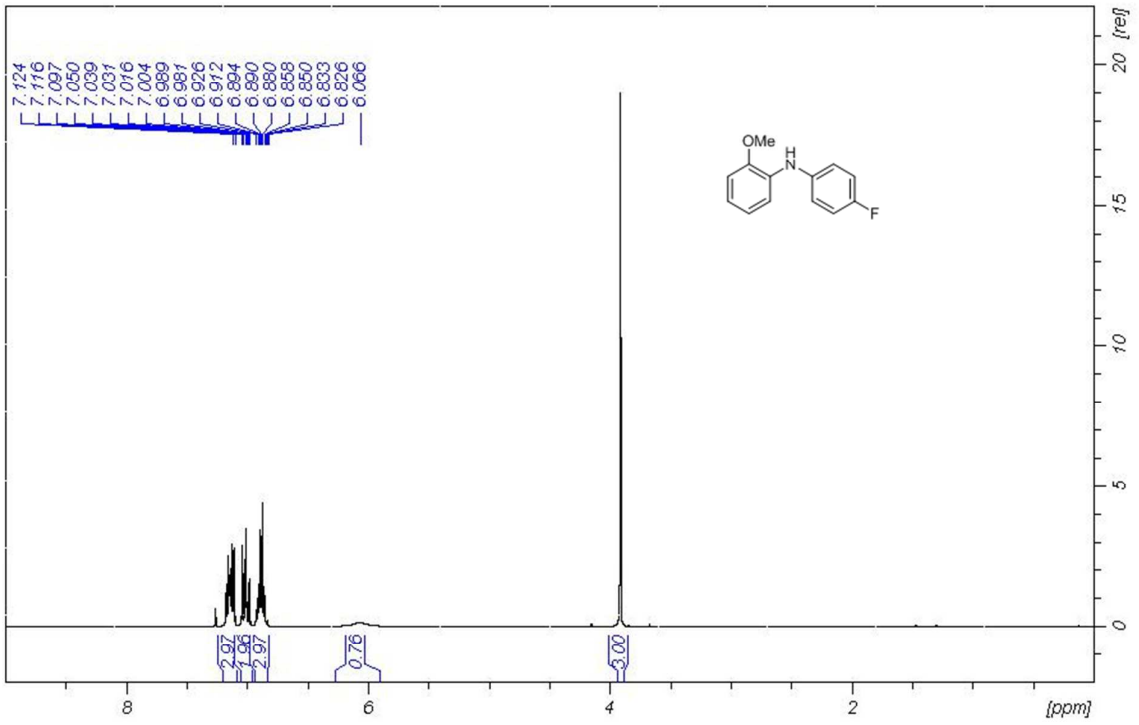


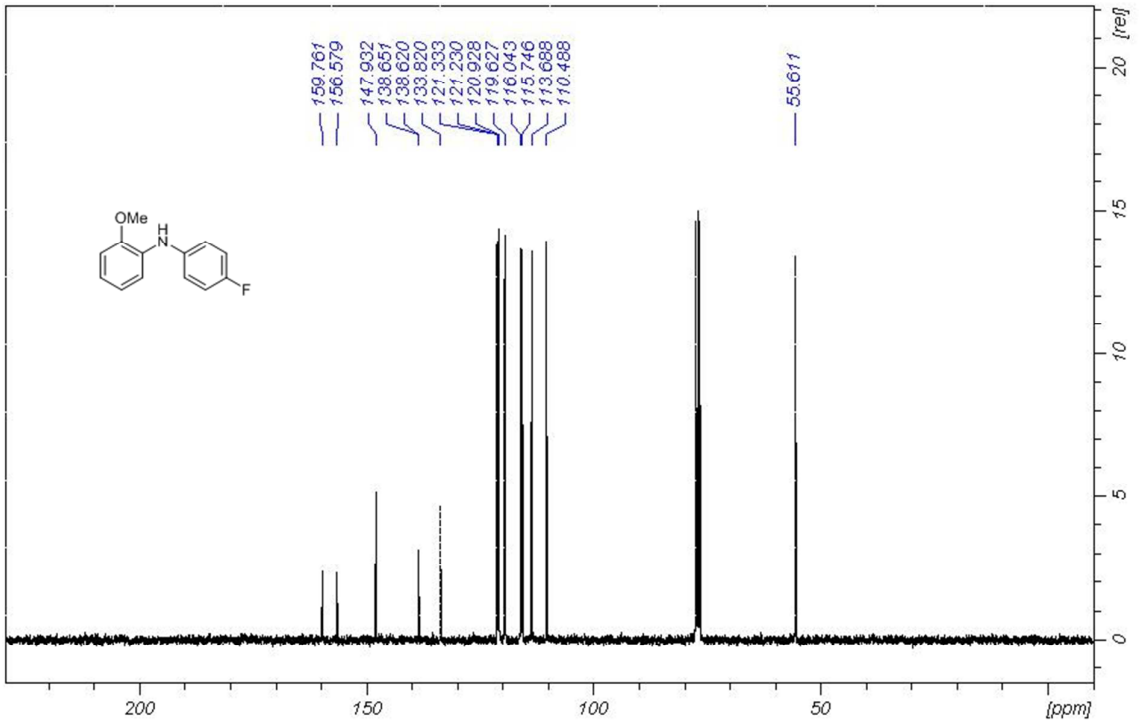

-124.706
H
N
F
OMe
[ref]
60
40
20
0
0
- 50
- 100
- 150
- 200
[ppm]

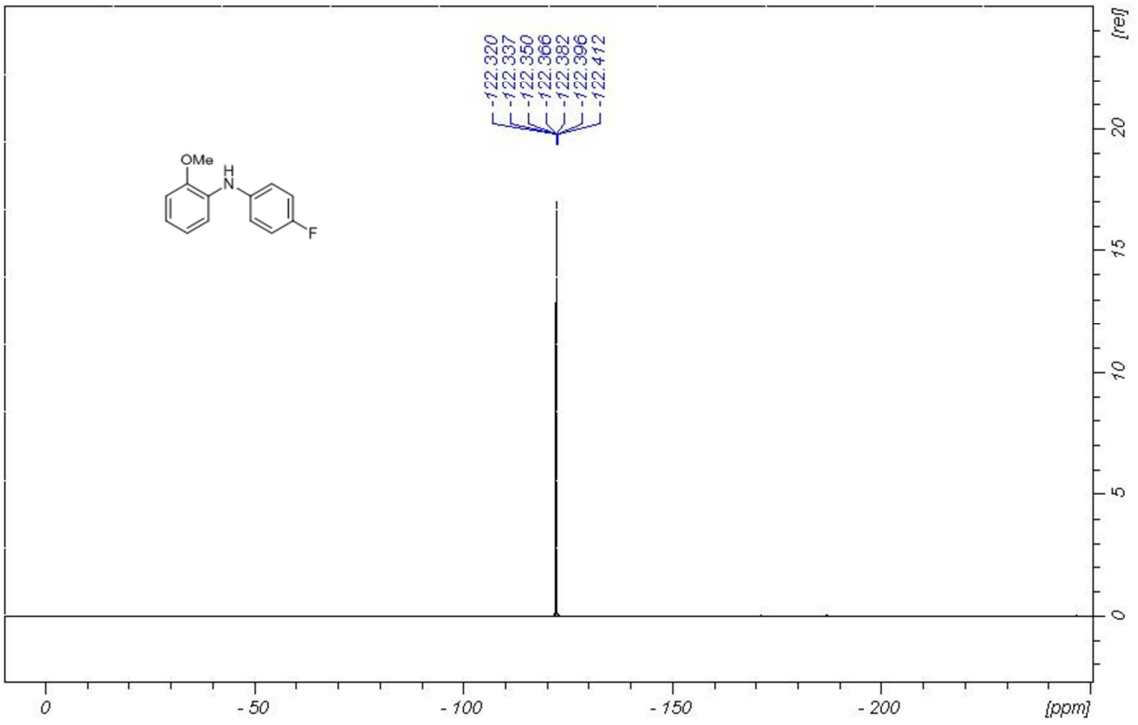

7.142
7.050
6.894
6.865
6.844
6.836
6.487
6.472
6.465
6.458
6.443
2.206
H
N
F
3.01
1.93
1.90
6.00
[ref]
25
20
15
10
5
0
8
6
4
2
[ppm]

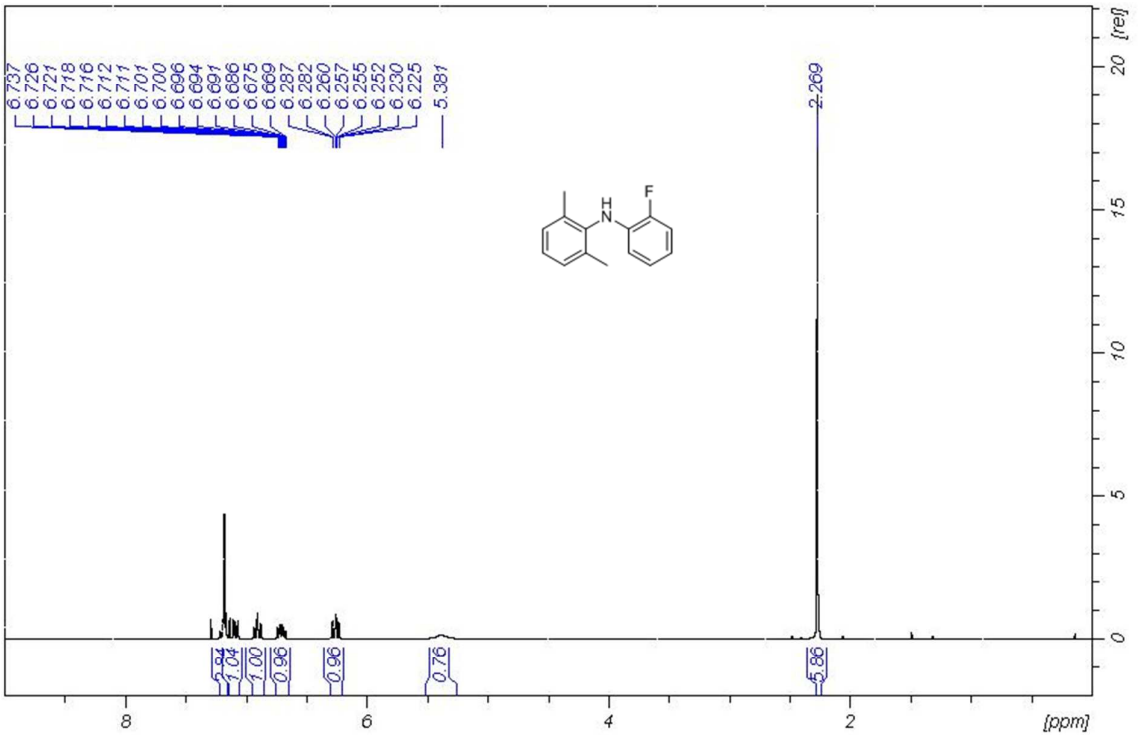

157.856
154.732
142.367
138.479
135.439
128.641
125.650
115.785
115.488
114.613
114.514
18.316
[rel]
200
150
100
50
[ppm]

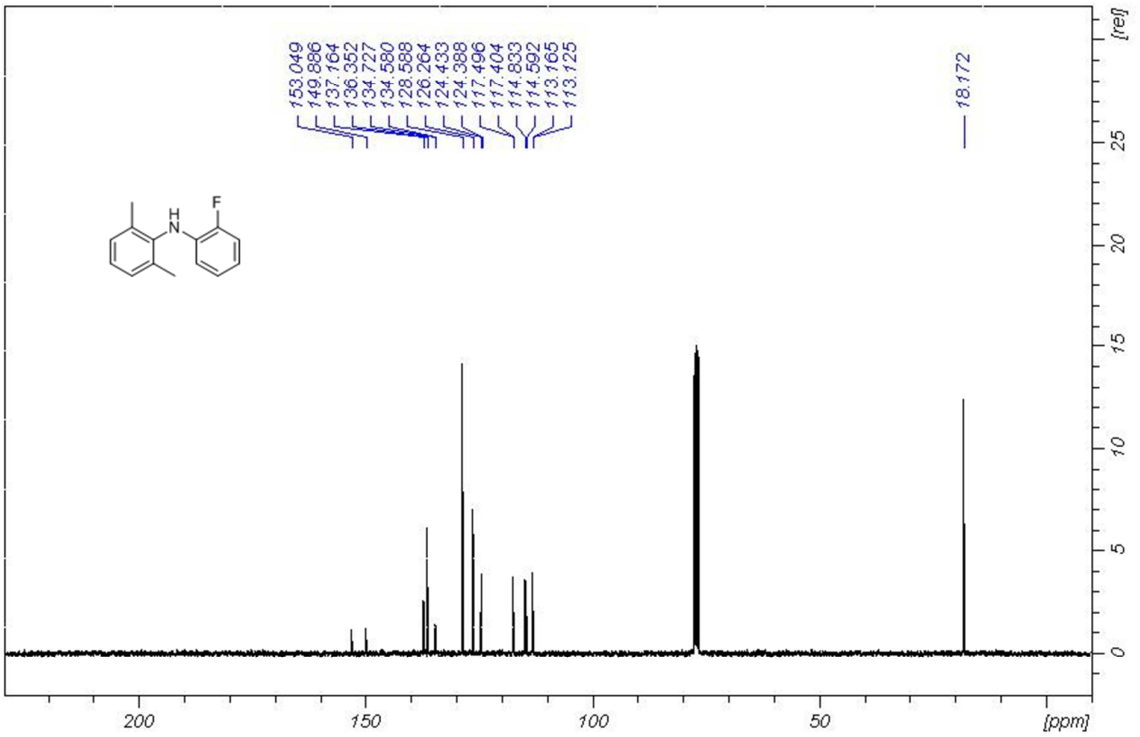

126.778
126.797
126.823
127.408
127.757
[rel]
0
- 50
- 100
- 150
- 200
[ppm]

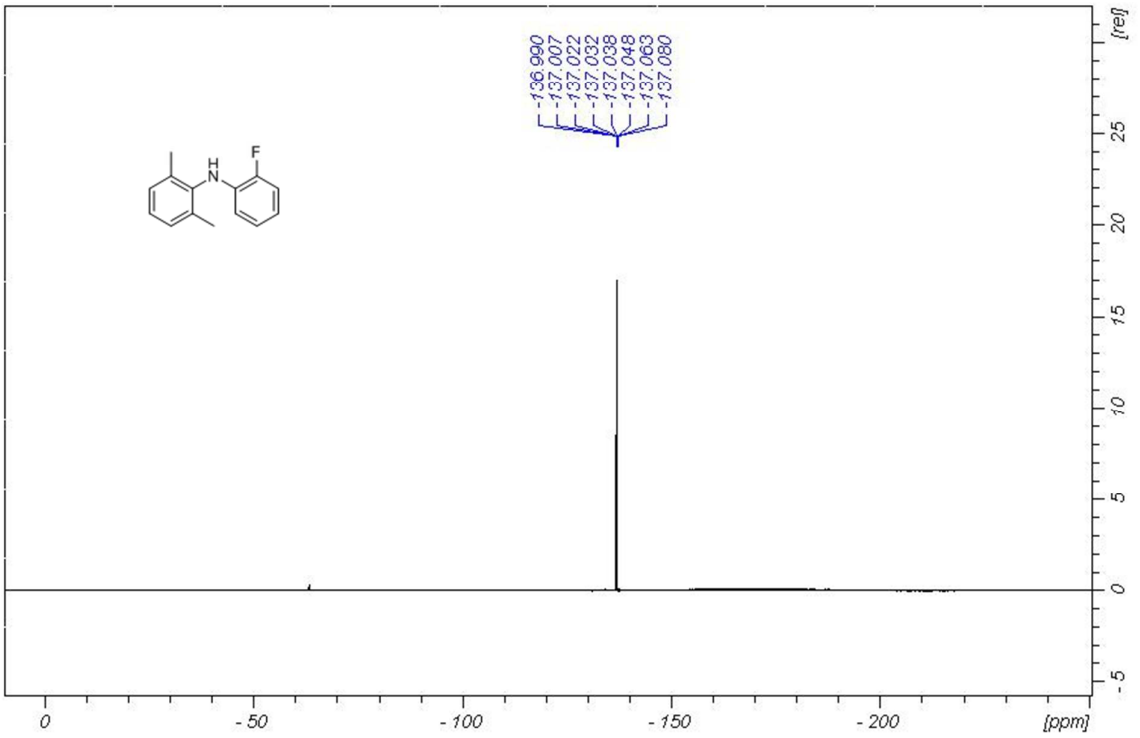

7.124
7.116
7.097
7.050
7.039
7.031
7.016
7.004
6.989
6.981
6.926
6.912
6.894
6.890
6.880
6.858
6.850
6.833
6.826
6.066
OMe
H
N
F
2.07
1.96
2.97
0.76
3.00
8
6
4
2
[ppm]
0
5
10
15
20
[rel]

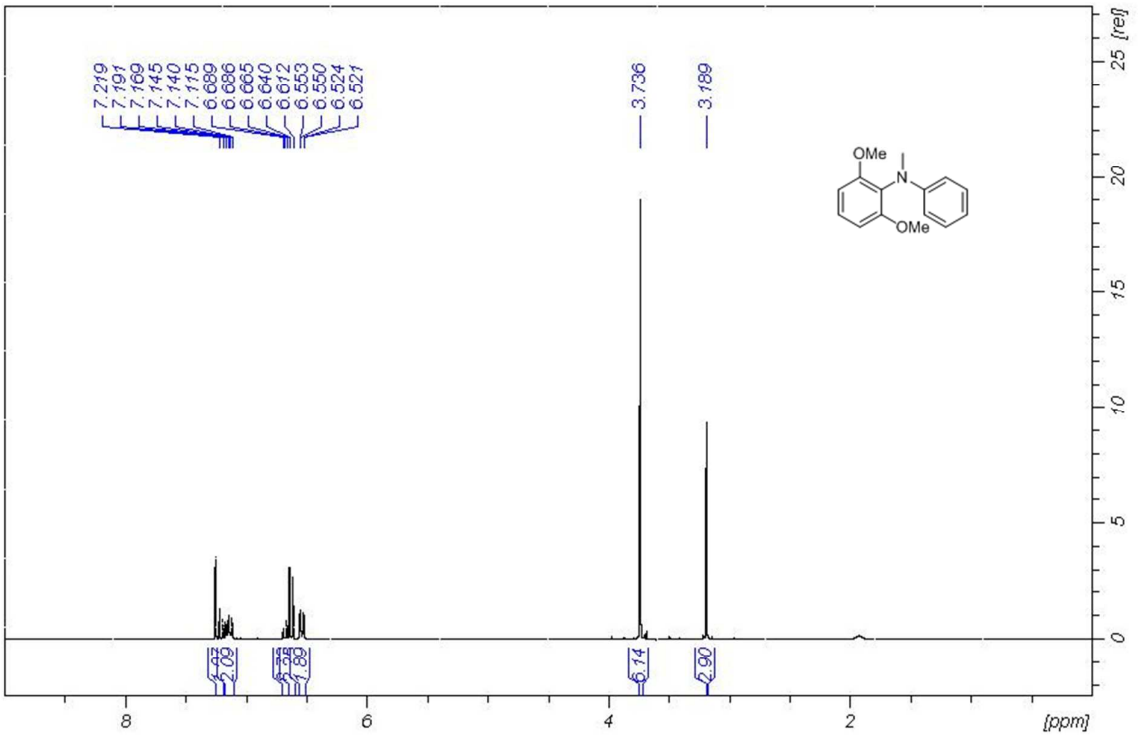

159.761
156.579
147.932
138.651
138.620
133.820
121.333
121.230
120.928
119.627
116.043
115.746
113.688
110.488
55.611
OMe
H
N
F
200
150
100
50
[ppm]
0
5
10
15
20
[rel]

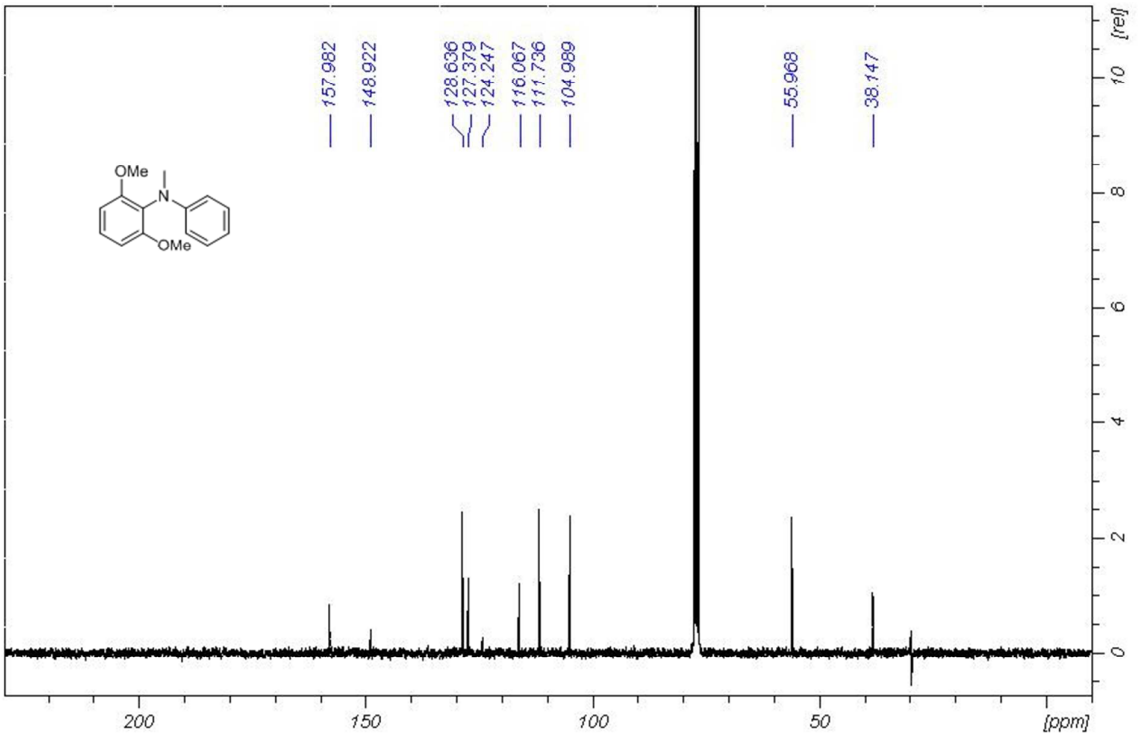

-122.320
-122.337
-122.350
-122.366
-122.382
-122.396
-122.412
OMe
H
N
F
[rel]
0
5
10
15
20
0
- 50
- 100
- 150
- 200
[ppm]

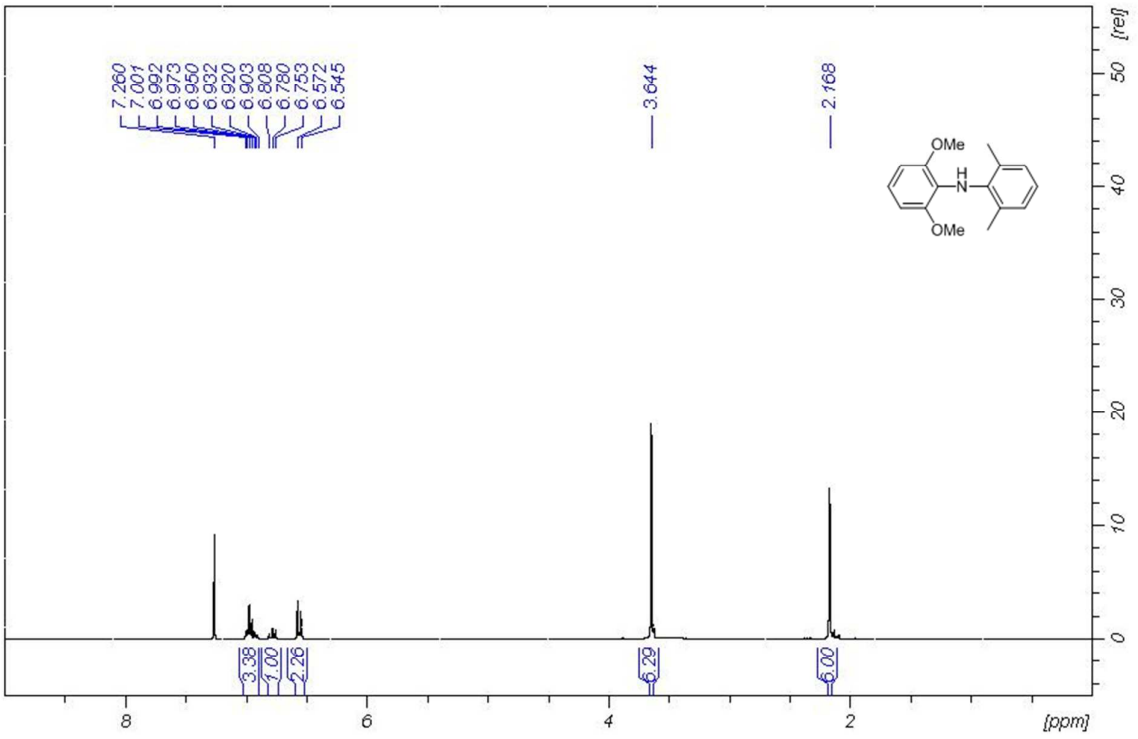

6.737
6.726
6.721
6.718
6.716
6.712
6.711
6.701
6.700
6.696
6.694
6.691
6.686
6.675
6.669
6.287
6.282
6.260
6.257
6.255
6.252
6.230
6.225
5.381
2.269
H
N
F
2.04
1.04
1.00
0.96
0.96
0.76
5.86
[rel]
0
5
10
15
20
8
6
4
2
[ppm]

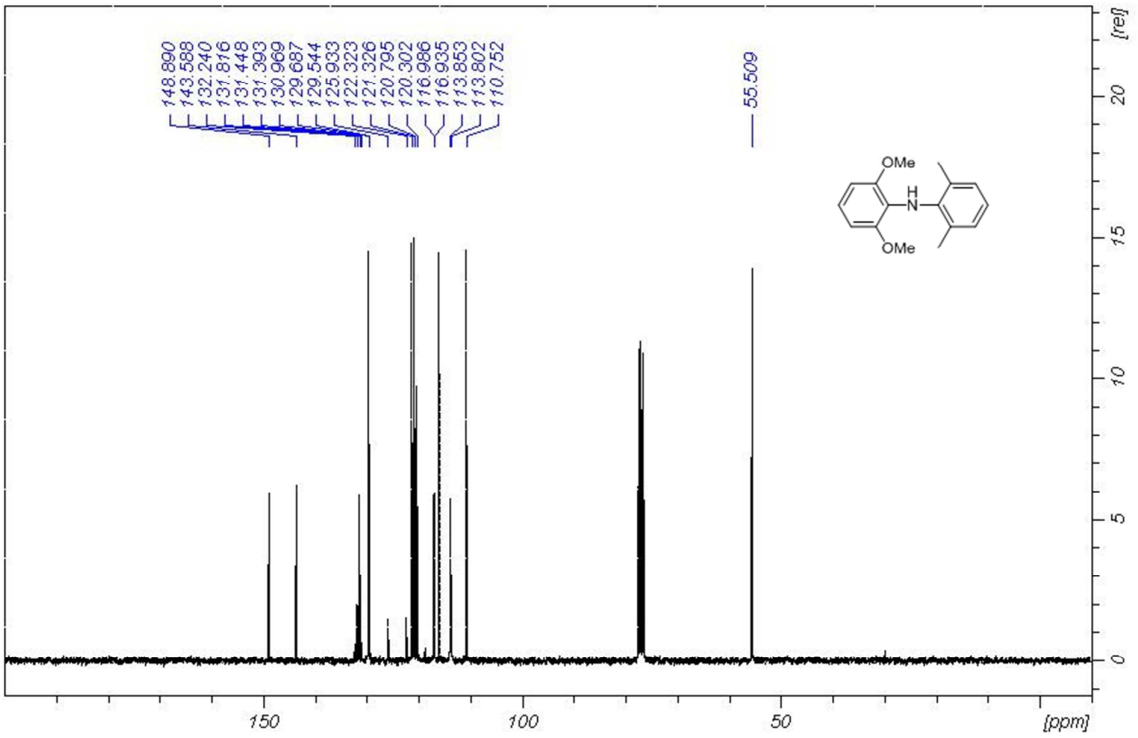

153.049
149.886
137.164
136.352
134.727
134.580
128.588
126.264
124.433
124.388
117.496
117.404
114.833
114.592
113.165
113.125
18.172
F
H
N
200
150
100
50
[ppm]
[rel]

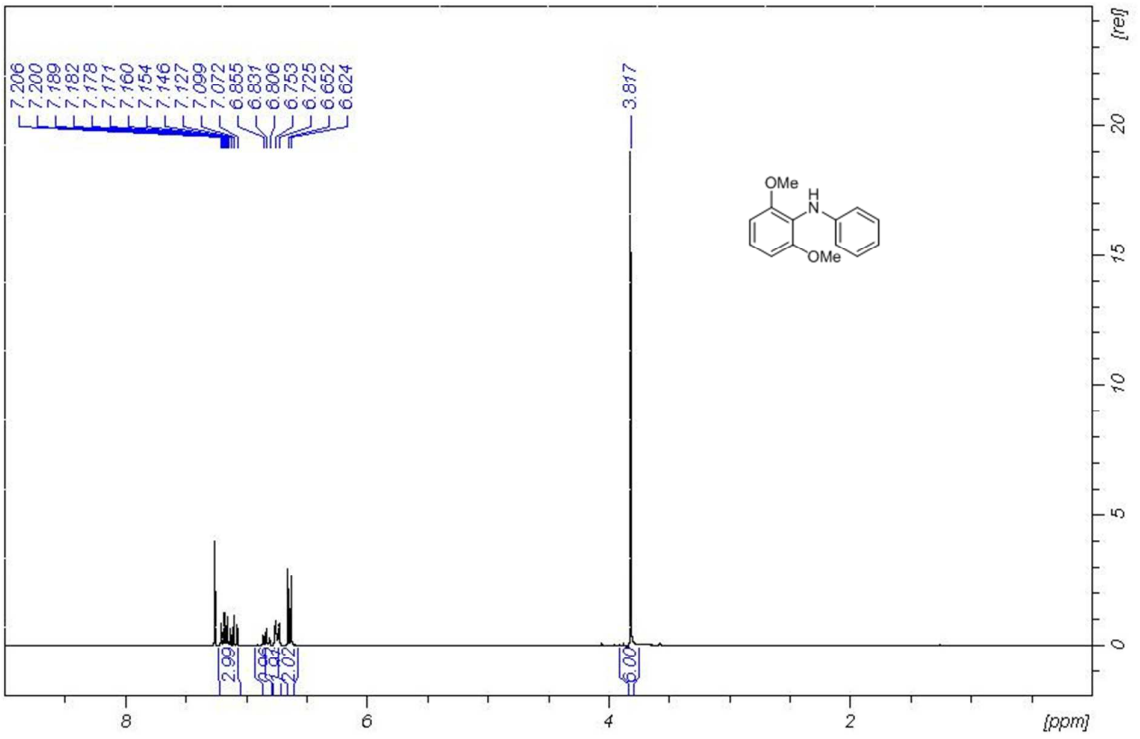

136.990
137.007
137.022
137.032
137.038
137.048
137.063
137.080
F
H
N
0
- 50
- 100
- 150
- 200
[ppm]
[rel]

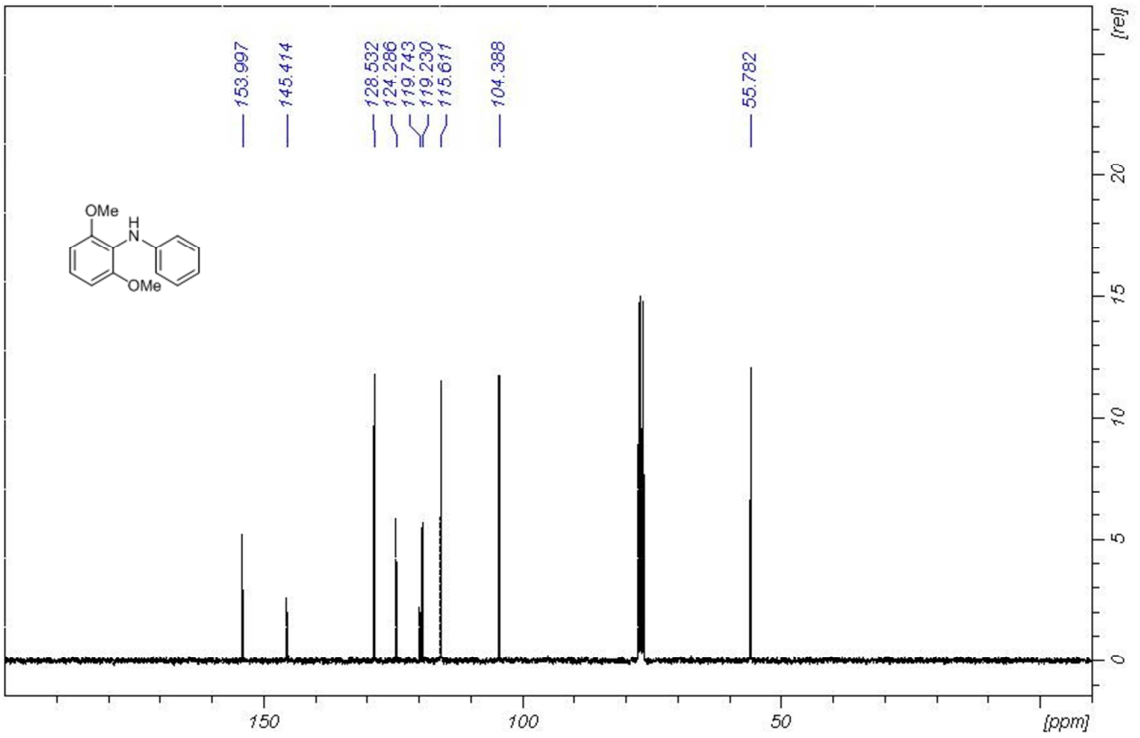

7.219
7.191
7.169
7.145
7.140
7.115
6.689
6.686
6.665
6.640
6.612
6.553
6.550
6.524
6.521
3.736
3.189
OMe
N
OMe
2.05
2.09
6.14
2.90
8
6
4
2
[ppm]
25
20
15
10
5
0
[rel]

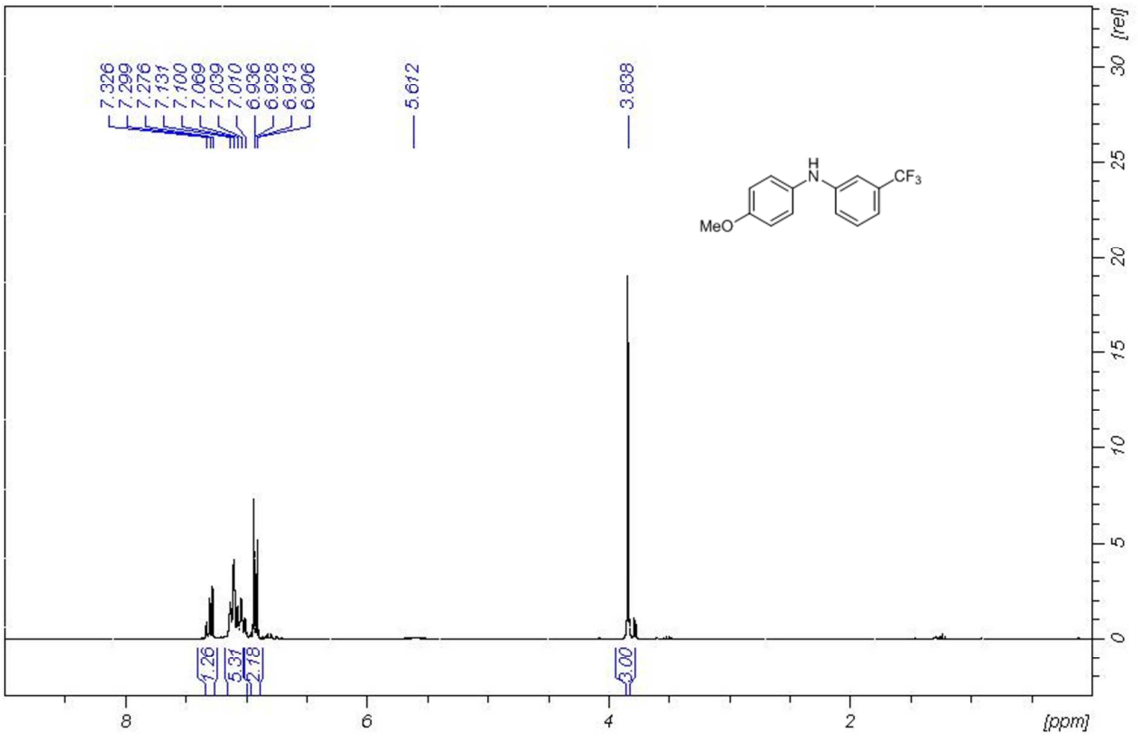

157.982
148.922
128.636
127.379
124.247
116.067
111.736
104.989
55.968
38.147
OMe
N
OMe
200
150
100
50
[ppm]
10
8
6
4
2
0
[rel]

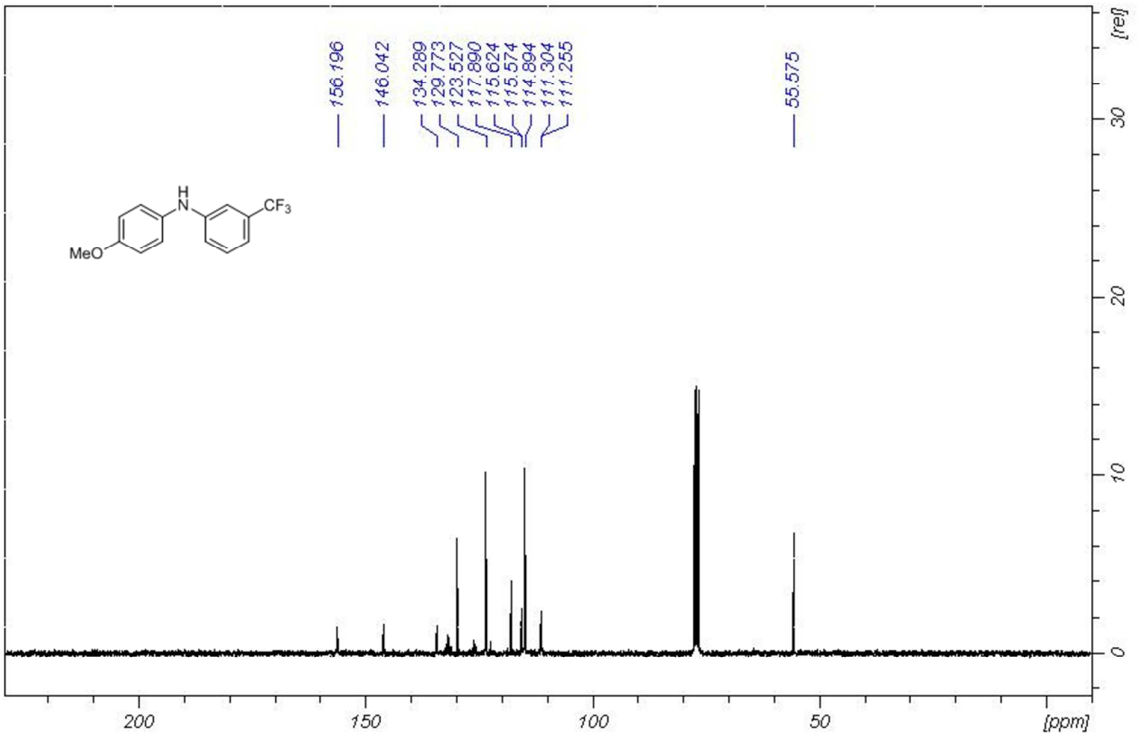

7.260
7.001
6.992
6.973
6.950
6.932
6.920
6.903
6.808
6.780
6.753
6.572
6.545
3.644
2.168
OMe
H
N
OMe
3.38
1.00
2.26
6.29
6.00
8
6
4
2
[ppm]
[rel]

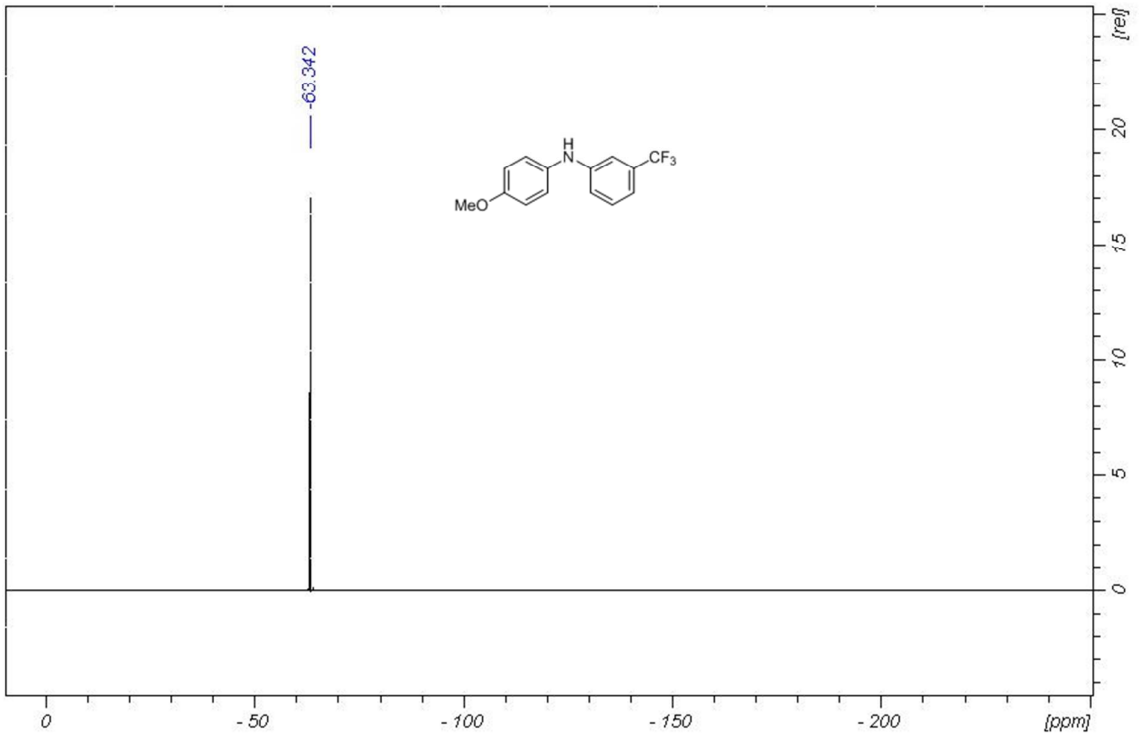

148.890
143.588
132.240
131.816
131.448
131.393
130.969
129.687
129.544
125.933
122.323
121.326
120.795
120.302
116.986
116.935
113.853
113.802
110.752
55.509
OMe
H
N
OMe
150
100
50
[ppm]
[rel]

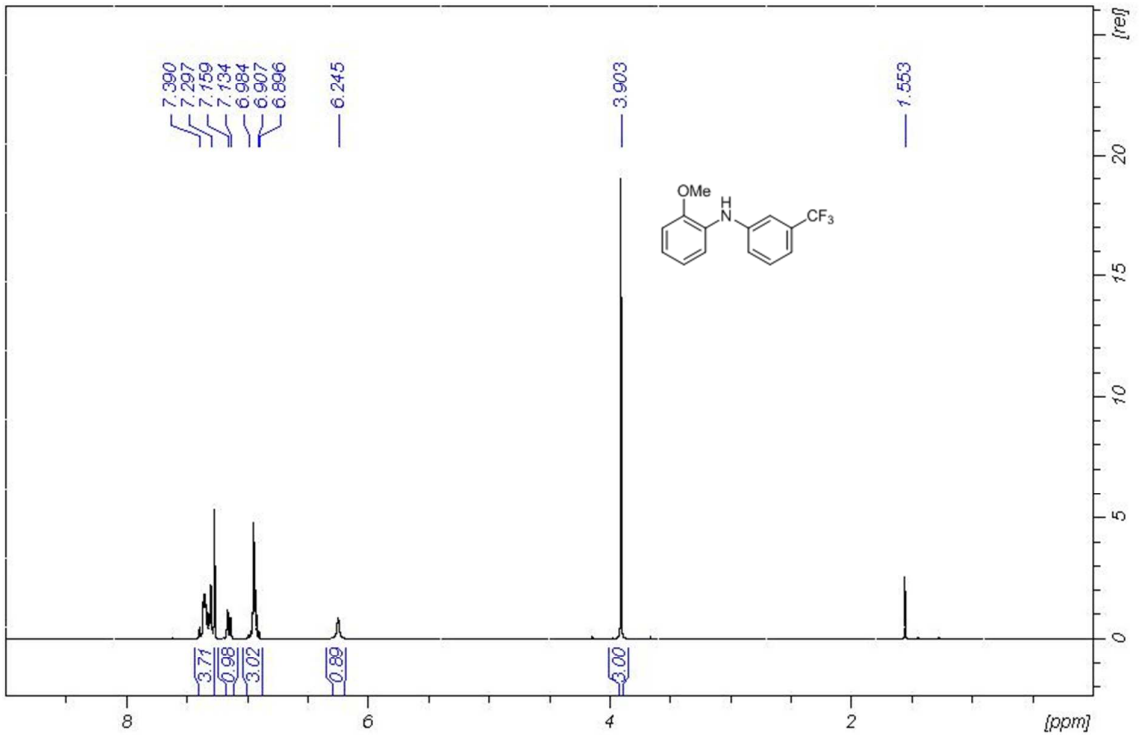

7.206
7.200
7.189
7.182
7.178
7.171
7.160
7.154
7.146
7.127
7.099
7.072
6.855
6.831
6.806
6.753
6.725
6.652
6.624
3.817
OMe
H
N
OMe
2.99
0.95
1.04
2.02
6.00
8
6
4
2
[ppm]
[rel]

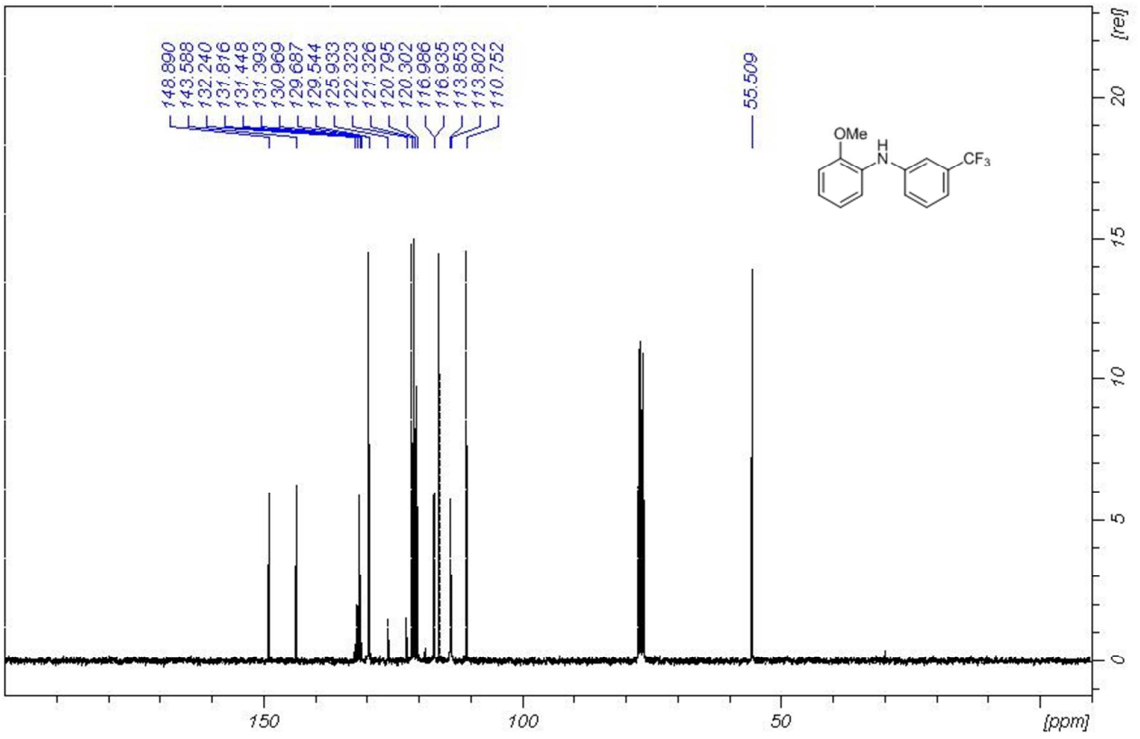

153.997
145.414
128.532
124.286
119.743
119.230
115.611
104.388
55.782
OMe
H
N
OMe
150
100
50
[ppm]
[rel]

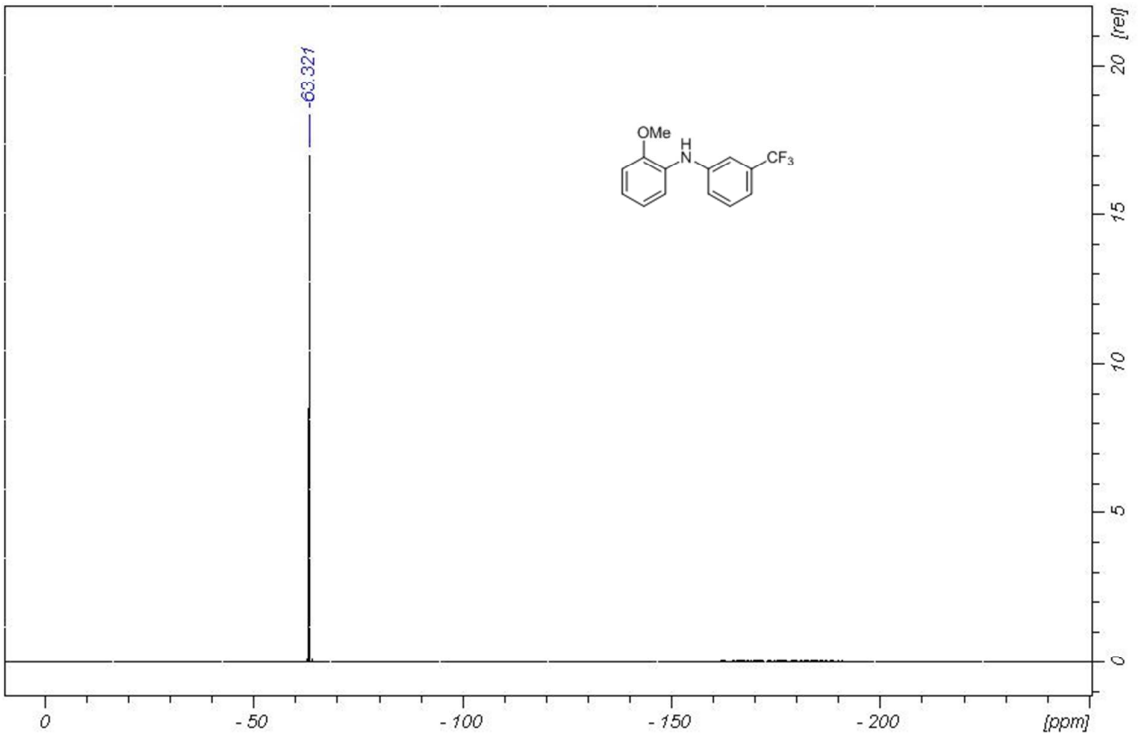

7.326
7.299
7.276
7.131
7.100
7.069
7.039
7.010
6.936
6.928
6.913
6.906
5.612
3.838
MeO
CF3
1.26
5.31
2.18
3.00
8
6
4
2
[ppm]
[rel]

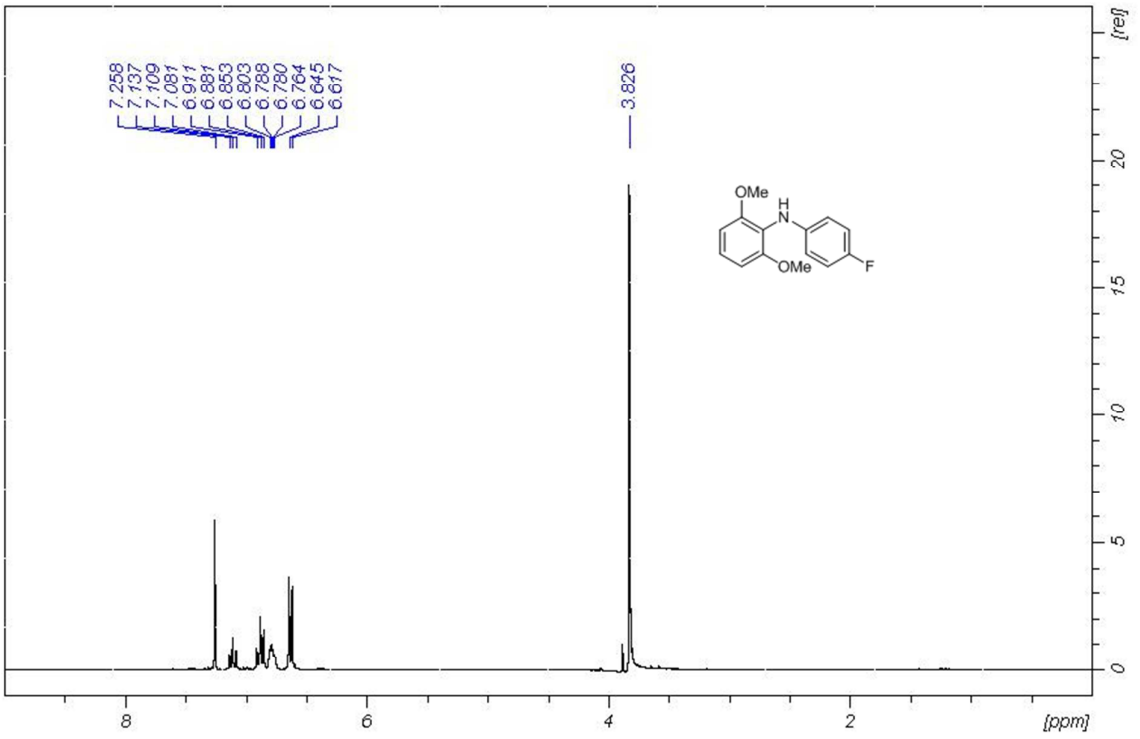

156.196
146.042
134.289
129.773
123.527
117.890
115.624
115.574
114.894
111.304
111.255
55.575
MeO
CF3
200
150
100
50
[ppm]
[rel]

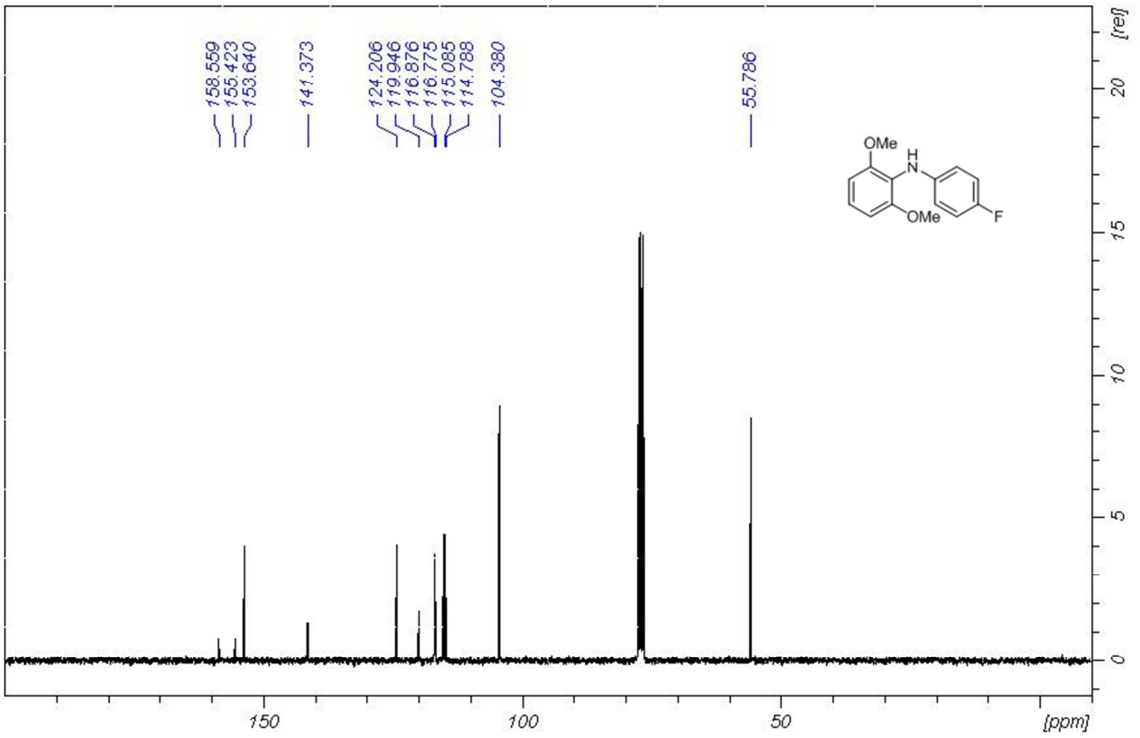

-63.342
MeO
H
N
CF3
[rel]
20
15
10
5
0
0
- 50
- 100
- 150
- 200
[ppm]

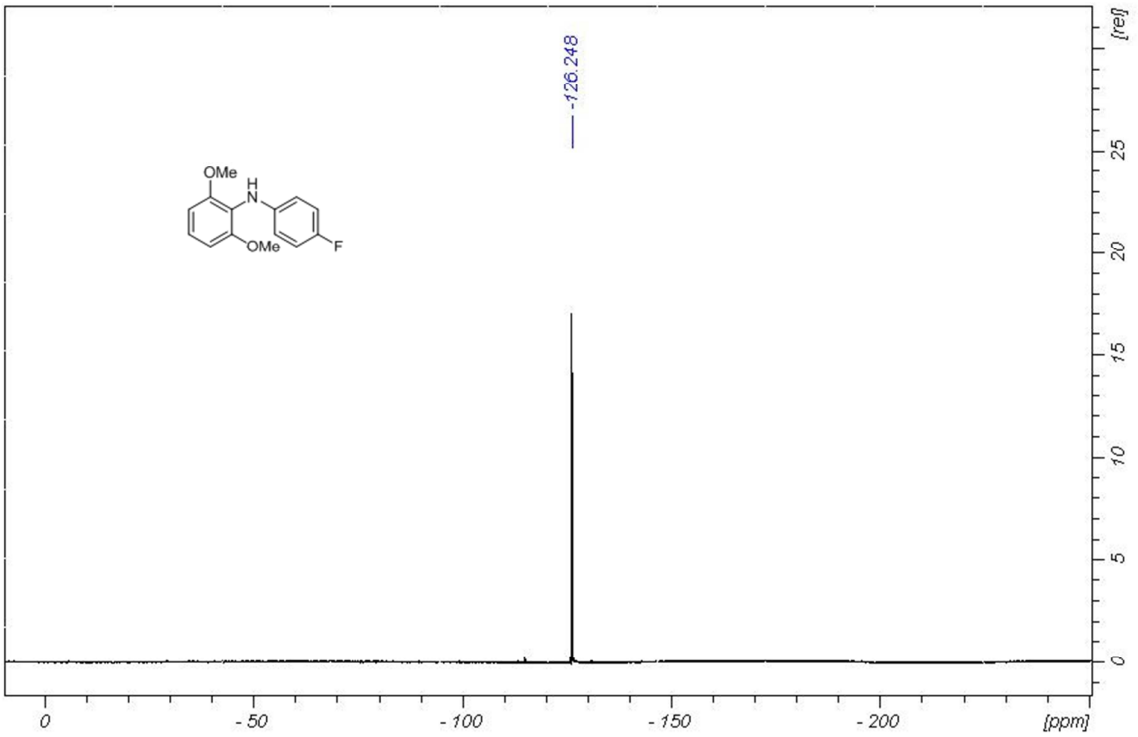

7.390
7.297
7.159
7.134
6.984
6.907
6.896
6.245
3.903
1.553
OMe
H
N
CF3
3.71
0.98
3.02
0.89
3.00
[rel]
20
15
10
5
0
8
6
4
2
[ppm]

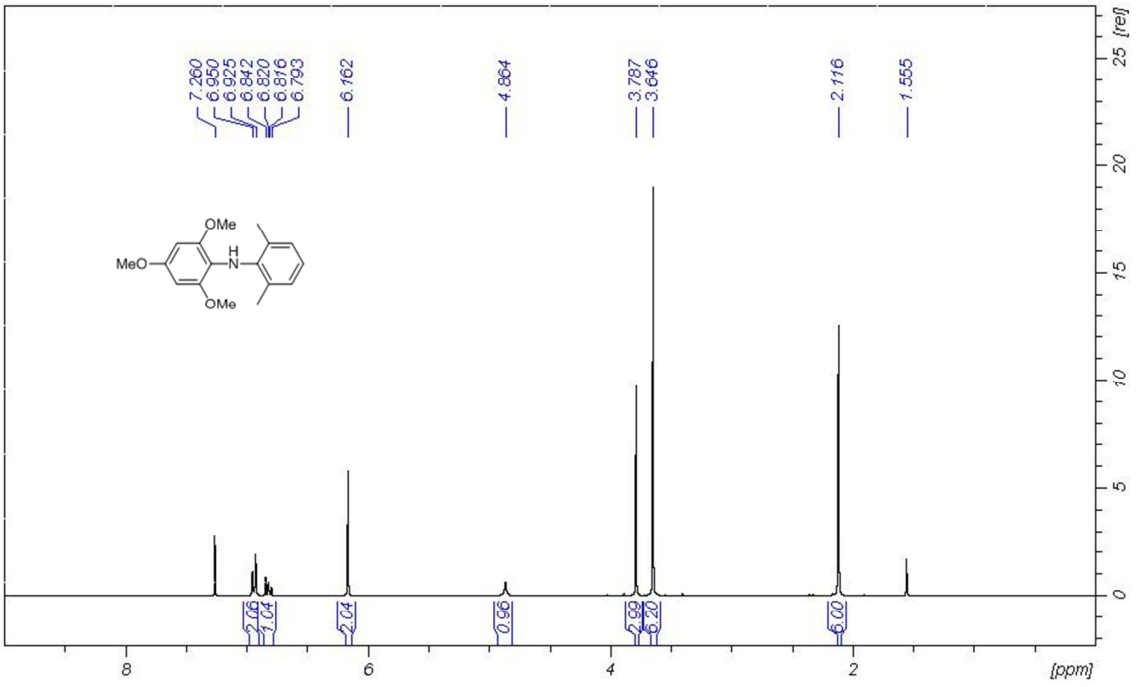

148.890
143.588
132.240
131.816
131.448
131.393
130.969
129.687
129.544
125.933
122.323
121.326
120.795
120.302
116.986
116.935
113.853
113.802
110.752
55.509
OMe
H
N
CF3
150
100
50
[ppm]
[rel]

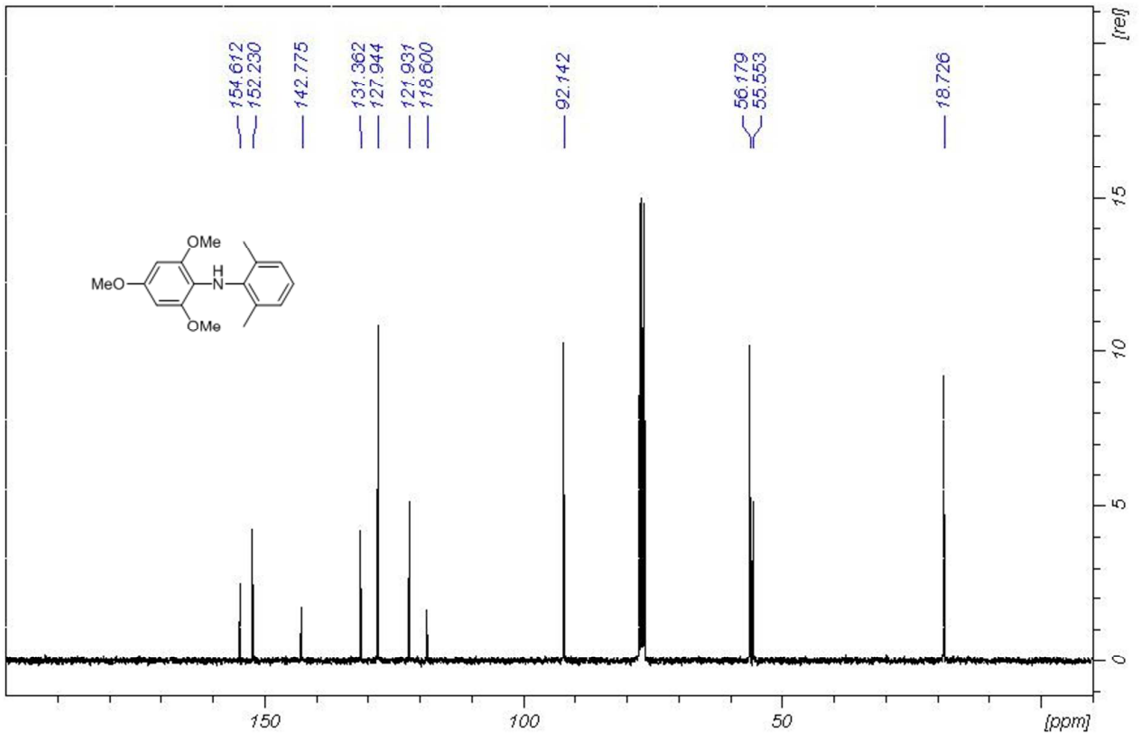

-63.321
OMe
H
N
CF3
0
- 50
- 100
- 150
- 200
[ppm]
[rel]


7.258
7.137
7.109
7.081
6.911
6.881
6.853
6.803
6.788
6.780
6.764
6.645
6.617
3.826
OMe
H
N
OMe
F
[ref]
[ppm]


158.559
155.423
153.640
141.373
124.206
119.946
116.876
116.775
115.085
114.788
104.380
55.786
OMe
H
N
OMe
F
[ref]
[ppm]


-126.248
OMe
H
N
OMe
F
[rel]
25
20
15
10
5
0
0
- 50
- 100
- 150
- 200
[ppm]


7.260
6.950
6.925
6.842
6.820
6.816
6.793
6.162
4.864
3.787
3.646
2.116
1.555
OMe
MeO
H
N
OMe
2.06
1.04
2.04
0.96
3.00
3.20
6.00
[rel]
25
20
15
10
5
0
8
6
4
2
[ppm]


154.612
152.230
142.775
131.362
127.944
121.931
118.600
92.142
56.179
55.553
18.726
OMe
MeO
H
N
OMe
[rel]
15
10
5
0
150
100
50
[ppm]