Electronic Supplementary Material (ESI) for RSC Advances

Copper-catalyzed Synthesis of Benzazoles *via* Aerobic Oxidative Condensation of *o*-Amino/Mercaptan/Hydroxyanilines with Benzylamines

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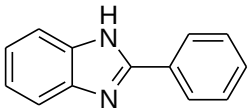
Representative experimental procedure: synthesis of 3a: To a 10 mL test-tube was added $CuBr_2$ (0.44 mg, 1 mol%), *o*-phenylenediamine **2a** (0.2 mmol), benzylamine **1a** (0.3 mmol) and toluene (2 mL). The reaction mixture was stirred at 100°C under air for 24 h. After cooling to room temperature, the resulting mixture was filtered through a short path of silica gel, eluting with ethyl acetate. The volatile compounds were removed in vacuo and the residue was purified by flash column chromatography on silica gel (eluent: hexane/ethyl acetate 5:1). Unless other indicated, the experiments in Tables 2 and 3 were carried out analogously. All products were purified by column chromatography and characterized by NMR spectroscopy and standard mass spectrometry.

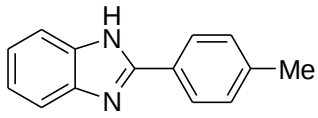
Spectral data for the products

2-phenyl-1H-benzo[d]imidazole(3a) [1]**:**



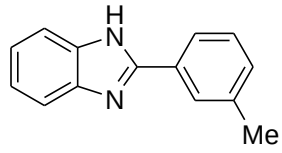
IR (film) 698, 825, 1026, 1159, 1259, 1405, 1456, 1595, 1630, 2931, 2959 cm^{-1}; ^1H NMR (300 MHz, DMSO-d_6) δ 12.92 (br s, 1H), 8.20 (d, *J*=7.2 Hz, 2H), 7.67 (m, 1H), 7.55-7.43 (m, 4H), 7.20-7.18 (m, 2H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 151.9, 144.5, 135.6, 130.8, 130.5, 129.6, 127.1, 123.2, 122.4, 119.5, 111.9; EI-MS (m/z, relative intensity): 194 (M^+, 100), 166 (9), 104 (6), 90 (8), 77 (7).

2-*p*-tolyl-1H-benzo[d]imidazole(3b) [1]**:**



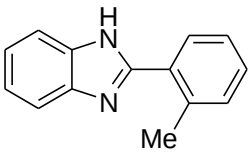
IR (film) 687, 762, 1070, 1432, 1495, 1546, 1653, 2923 cm^{-1}; ^{1}H NMR (300 MHz, DMSO-d_6) δ 12.82 (br s, 1H), 8.07 (d, *J*=8.1 Hz, 2H), 7.64-7.50 (m, 2H), 7.32 (d, *J*=8.1 Hz, 2H), 7.17 (d, *J*=7.5 Hz, 2H), 2.35(s, 3H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 152.0, 144.5, 140.2, 135.6, 130.1, 128.1, 127.1, 122.9, 122.2, 119.4, 111.9, 21.8; EI-MS (m/z, relative intensity): 208 (M^+, 100), 192 (4), 116 (6), 103 (10), 91 (7).

2-*m*-tolyl-1H-benzo[d]imidazole(3c)[2]:



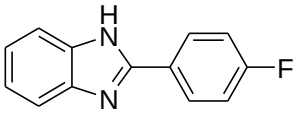
IR (film) 703, 806, 1110, 1145, 1407, 1462, 1546, 1601, 1632, 2924 cm^{-1}; ^{1}H NMR (300 MHz, DMSO-d_6) δ 12.86 (br s, 1H), 8.03 (s, 1H), 7.97 (d, *J*=7.5 Hz, 1H), 7.59 (m, 2H), 7.40 (t, *J*=7.5 Hz, 1H), 7.26 (d, *J*=7.5 Hz, 1H), 7.26-7.17 (m, 2H), 2.39(s, 3H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 152.0, 144.5, 138.8, 135.6, 131.1, 130.8, 129.5, 127.7, 124.3, 122.9, 119.4, 122.0, 21.9; EI-MS (m/z, relative intensity): 208 (M^+, 100), 192(4), 180 (4), 116 (4), 103 (13), 91 (8).

2-*o*-tolyl-1H-benzo[d]imidazole(3d)[3]:



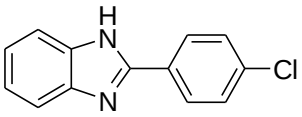
IR (film) 713, 760, 876, 1050, 1462, 1528, 1623, 1671, 2926 cm^{-1}; ^{1}H NMR (300 MHz, DMSO-d_6) δ 12.62 (br s, 1H), 7.76-7.73 (m, 1H), 7.68 (m, 1H), 7.53 (m, 1H), 7.36-7.30 (m, 3H), 7.21-7.19 (m, 2H), 2.62(s, 3H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 152.6, 144.4, 137.7, 135.5, 131.9, 130.8, 130.1, 129.9, 126.6, 122.9, 122.0, 119.6, 111.9, 21.9; EI-MS (m/z, relative intensity): 208 (M^+, 80), 207(100), 180 (4), 116 (3), 103 (13), 91 (7).

2-(4-fluorophenyl)-1H-benzo[d]imidazole(3e)[4]:



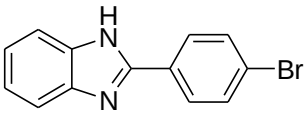
IR (film) 703, 973, 1145, 1310, 1408, 1467, 1537, 1630, 2924 cm^{-1}; ^{1}H NMR (300 MHz, DMSO-d_6) δ 12.91 (br s, 1H), 8.24 (d, *J*=8.7 Hz, 1H), 8.22 (d, *J*=8.7 Hz, 1H), 7.63-7.55 (m, 2H), 7.37 (t, *J*=8.7 Hz, 2H), 7.20-7.17 (m, 2H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 163.7 (d, *J*=245.9Hz), 151.0, 129.4, 129.3, 127.5, 123.1, 122.4, 110.5, 116.7, 116.5, 111.9; EI-MS (m/z, relative intensity): 212 (M^+, 100), 180 (7), 122 (5), 106 (7), 91 (8), 64(9).

2-(4-chlorophenyl)-1H-benzo[d]imidazole(3f)[1]:



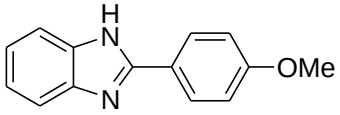
IR (film) 550, 694, 774, 1061, 1386, 1443, 1528, 1623, 2914 cm^{-1}; ^1H NMR (300 MHz, DMSO-d_6) δ 12.97 (br s, 1H), 8.18 (d, *J*=8.7 Hz, 2H), 7.60-7.57 (m, 4H), 7.21-7.17 (m, 2H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 150.8, 144.3, 137.4, 135.6, 135.1, 129.7, 128.8, 123.2, 122.6, 119.4, 112.1; EI-MS (m/z, relative intensity): 228 (M^+, 100) (Cl^{35}), 230 (M^+, 30) (Cl^{37}), 193 (20), 166 (7), 138(3), 90 (7), 63(10).

2-(4-bromophenyl)-1H-benzo[d]imidazole(3g)[5]:



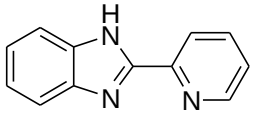
IR (film) 564, 698, 777, 1025, 1159, 1269, 1405, 1433, 1456, 1595, 2931 cm^{-1};^1H NMR (300 MHz, DMSO-d_6) δ 12.98 (br s, 1H), 8.11 (d, *J*=8.4 Hz, 2H), 7.73 (d, *J*=8.4 Hz, 2H), 7.66-7.52 (m, 2H), 7.19 (d, *J*=7.8 Hz, 2H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 150.8, 144.3, 135.6, 132.6, 130.1, 129.0, 123.9, 123.4, 122.5, 119.6, 112.1; EI-MS (m/z, relative intensity): 272 (M^+, 100) (Br^{79}), 274 (M^+, 94) (Br^{81}), 193 (65), 166 (16), 90 (20), 63 (17).

2-(4-methoxyphenyl)-1H-benzo[d]imidazole(3h)[1]:



IR (film) 747, 1030, 1177, 1249, 1436, 1496, 1583, 1612, 2968 cm^{-1}; ^1H NMR (300 MHz, DMSO-d_6) δ 12.73 (br s, 1H), 8.12 (d, *J*=8.7 Hz, 2H), 7.61-7.49 (m, 2H), 7.17-7.14 (m, 2H), 7.08 (d, *J*=8.7 Hz, 2H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 161.2, 151.9, 144.5, 135.6, 128.7, 123.4, 122.7, 122.4, 119.2, 115.0, 111.7; EI-MS (m/z, relative intensity): 224 (M^+, 100), 209 (57), 181 (45), 112 (7), 90 (8), 77 (9).

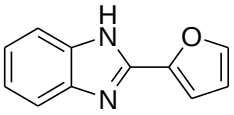
2-(pyridin-2-yl)-1H-benzo[d]imidazole(3i)[1]:



IR (film) 744, 1011, 1427, 1447, 1489, 1551, 1595, 1664, 1694, 1792, 2912 cm^{-1}; ^1H NMR (300 MHz, DMSO-d_6) δ 13.09 (br s, 1H), 8.70 (d, *J*=7.8 Hz, 1H), 8.31 (d, *J*=8.1 Hz, 1H), 7.97 (t, *J*=7.8 Hz, 1H), 7.68 (d, *J*=7.5 Hz, 1H), 7.50 (q, *J*=7.5 Hz, 2H), 7.25-7.15 (m, 2H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 151.3, 149.9, 149.2, 144.6, 138.1, 135.5, 125.5, 123.7, 122.5, 122.1, 119.9, 112.7; EI-MS (m/z, relative intensity): 195 (M^+, 100), 167 (17),

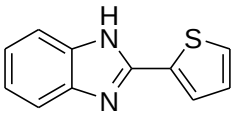
105 (6), 78 (12).

2-(furan-2-yl)-1H-benzo[d]imidazole(3j)[6]:



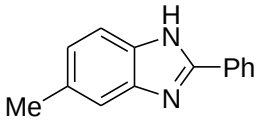
IR (film) 874, 1265, 1406, 1546, 1633, 1653, 2922 cm^{-1}; ^1H NMR (300 MHz, DMSO-d_6) δ 12.88 (br s, 1H), 7.92 (s, 1H), 7.59-7.42 (m, 2H), 7.18-7.17 (m, 3H), 6.17 (s, 1H); ^{13}C NMR (75 MHz, DMSO-d_6) δ146.3, 145.1, 145.0, 144.3, 122.8, 119.4, 112.8, 111.9, 111.1; EI-MS (m/z, relative intensity): 184 (M^+, 100), 155 (26), 129 (12), 91 (10).

2-(thiophen-2-yl)-1H-benzo[d]imidazole(3k)[6]:



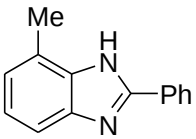
IR (film) 764, 831, 965, 1273, 1429, 1447, 1471, 1490, 1602, 2911 cm^{-1}; ^1H NMR (300 MHz, DMSO-d_6) δ 12.91 (br s, 1H), 7.82 (d, *J*=3.6 Hz, 1H), 7.70 (d, *J*=4.8 Hz, 1H), 7.56-7.50 (m, 2H), 7.22-7.16 (m, 3H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 147.7, 135.3, 134.4, 129.3, 128.9, 127.3, 123.1, 122.4, 119.1, 111.7; EI-MS (m/z, relative intensity): 200 (M^+, 100), 156 (6), 96 (8), 62 (10).

5-methyl-2-phenyl-1H-benzo[d]imidazole(3l)[1]:



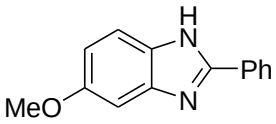
IR (film) 687, 762, 804, 961, 1069, 1431, 1475, 1505, 1634, 2919 cm^{-1}; ^1H NMR (300 MHz, DMSO-d_6) δ 12.78 (br s, 1H), 8.18 (d, *J*=7.2 Hz, 2H), 7.54-7.33 (m, 5H), 7.01 (d, *J*=8.1 Hz, 1H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 151.6, 142.6, 142.6, 135.8, 132.5, 131.0, 129.5, 126.9, 124.5, 124.0, 119.1, 111.7, 22.2; EI-MS (m/z, relative intensity): 208 (M^+, 100), 104 (26), 77 (22).

7-methyl-2-phenyl-1H-benzo[d]imidazole(3m)[7];mixture of tautomers



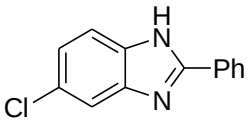
IR (film) 699, 742, 859, 1025, 1101, 1271, 1417, 1441, 1469, 1541, 1634, 2721, 2914 cm^{-1}; ^1H NMR (300 MHz, DMSO-d_6) δ 12.80 (br s, 0.5H), 12.54 (br s, 0.4H), 8.25-8.14 (m, 2H), 7.55-7.31 (m, 4H), 7.11-6.96 (m, 2H), 2.59-2.56 (m, 3H), ; ^{13}C NMR (75 MHz, DMSO-d_6) δ 156.5, 155.7, 148.9, 148.6, 139.9, 135.7, 134.9, 134.2, 134.1, 133.7, 132.1, 131.8, 128.5, 127.8, 127.8, 127.2, 127.1, 126.7, 121.7, 114.2, 22.7, 22.3; EI-MS (m/z, relative intensity): 208 (M^+, 100), 104 (28), 77 (24), 51 (9).

5-methoxy-2-phenyl-1H-benzo[d]imidazole(3n)[5]:



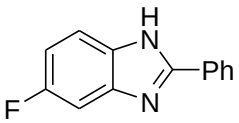
IR (film) 741, 764, 975, 1108, 1274, 1402, 1443, 1528, 1590, 1623, 2791, 2878 cm^{-1}; ^{1}H NMR (300 MHz, DMSO-d_6) δ 12.76 (br s, 1H), 8.14 (d, *J*=7.2 Hz, 2H), 7.53-7.40 (m, 4H), 7.09 (m, 1H), 6.84 (d, *J*=8.7 Hz, 1H), 3.79 (s, 3H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 161.2, 156.2, 156.2, 135.7, 134.8, 134.2, 131.5, 117.1, 117.0, 61.0; EI-MS (m/z, relative intensity): 224 (M^+, 100), 209 (97), 181 (38), 154 (22), 127 (12), 104 (7), 77 (16), 51 (13).

5-chloro-2-phenyl-1H-benzo[d]imidazole(3o)[1]:



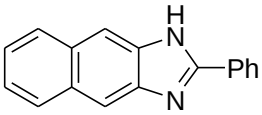
IR (film) 703, 749, 1131, 1405, 1458, 1563, 1632, 1655, 2932 cm^{-1}; ^{1}H NMR (300 MHz, DMSO-d_6) δ 13.10 (br s, 1H), 8.17 (d, *J*=6.9 Hz, 2H), 7.65-7.43 (m, 5H), 7.22-7.18 (m, 1H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 158.0, 135.5, 135.1, 134.3, 132.0, 127.7; EI-MS (m/z, relative intensity): 228 (M^+, 100) (Cl^{35}), 230 (M^+, 29) (Cl^{37}), 192 (10), 166 (8), 90 (8), 63 (13).

5-fluoro-2-phenyl-1H-benzo[d]imidazole(3p)[2]:



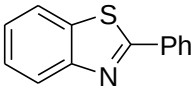
IR (film) 619, 687, 762, 961, 1071, 1249, 1432, 1474, 1505, 1632, 2922 cm^{-1}; ^{1}H NMR (300 MHz, DMSO-d_6) δ 13.03 (br s, 1H), 8.17 (d, *J*=7.5 Hz, 2H), 7.58-7.39 (m, 5H), 7.08-7.01 (m, 1H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 159.3 (d, *J*=234.0Hz), 153.3, 130.6, 129.6, 128.6, 127.1, 110.9, 110.6; EI-MS (m/z, relative intensity): 212 (M^+, 100), 184 (8), 106 (9), 82 (10).

2-phenyl-1H-naphtho[2,3-d]imidazole(3q)[7]:



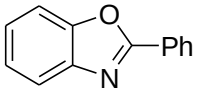
IR (film) 653, 816, 872, 1143, 1441, 1474, 1569, 1655, 2953 cm^{-1}; ^{1}H NMR (300 MHz, DMSO-d_6) δ 12.99 (br s, 1H), 8.33 (d, *J*=6.9 Hz, 2H), 8.23-7.98 (m, 4H), 7.61-7.50 (m, 3H), 7.36-7.33 (m, 2H); ^{13}C NMR (75 MHz, DMSO-d_6) δ 156.0, 145.0, 136.6, 131.3, 130.8, 130.5, 129.6, 127.8, 127.7, 124.2, 123.8, 115.9, 107.3; ESI-MS m/z 244.9 $(M+H)^+$.

2-phenylbenzo[d]thiazole(3r)[1]:



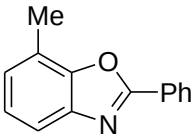
IR (film) 831, 965, 1090, 1225, 1429, 1490, 1602, 3023 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 8.09 (d, *J*=6.6 Hz, 2H), 7.88 (d, *J*=7.8 Hz, 3H), 7.52-7.48 (m, 4H), 7.38 (d, *J*=7.5 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.2, 154.3, 135.3, 133.8, 131.1, 129.2, 127.8, 126.5, 125.4, 123.5, 121.8; EI-MS (m/z, relative intensity): 211 (M^+, 100), 184 (6), 108 (27), 82 (8), 69 (13).

2-phenylbenzo[d]oxazole(3s)[1]:



IR (film) 683, 746, 935, 1050, 1421, 1586, 1647, 3051 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 8.26 (d, *J*=5.1 Hz, 2H), 7.77 (t, *J*=3.0 Hz, 1H), 7.59-7.53 (m, 4H), 7.36-7.34 (t, *J*=3.0 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 163.2, 150.9, 142.3, 131.7, 129.1, 127.8, 127.4, 125.3, 124.7, 120.2, 110.7; EI-MS (m/z, relative intensity): 196 (M^+, 14), 195 (100), 167 (24), 92 (8), 77 (10), 63 (18).

7-methyl-2-phenylbenzo[d]oxazole(3t)[8]:



IR (film) 613, 699, 859, 1025, 1417, 1541, 1634, 3047 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 8.24-8.21 (m, 2H), 7.63 (d, *J*=8.1 Hz, 1H), 7.52-7.47 (m, 2H), 7.38 (s, 1H), 7.16 (d, *J*=8.1 Hz, 1H), 2.52 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 162.7, 151.2, 140.1, 131.6, 131.4, 129.0, 128.8, 127.6, 125.9, 119.5, 110.9, 22.2; EI-MS (m/z, relative intensity): 210 (M^+, 14), 209 (100), 180 (14), 106 (26), 78 (43), 51 (13).

Reference

1, T. B. Nguyen, L. Ermolenko, W. A. Dean and A. Al-Mourabit, *Org. Lett.* 2012, **14**, 5948.

2, M. Shen and T. G. Driver, *Org. Lett.* 2008, **15**, 3367.

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4823.

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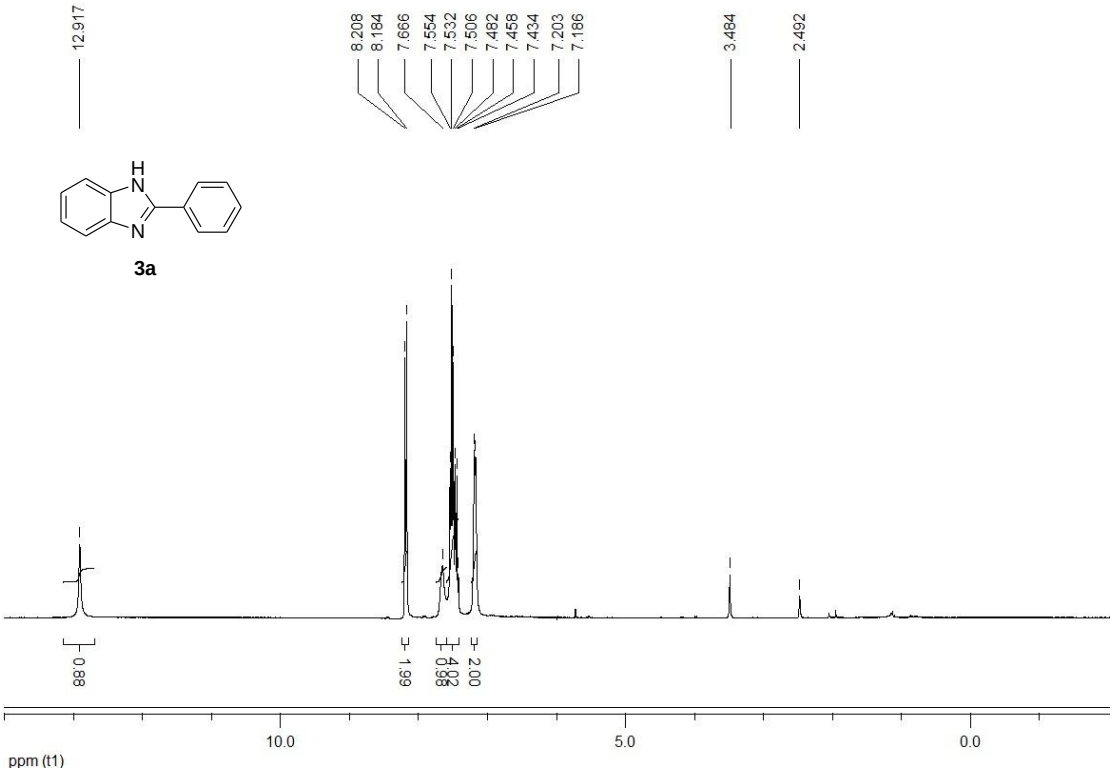
^{1}H NMR and ^{13}C NMR spectra



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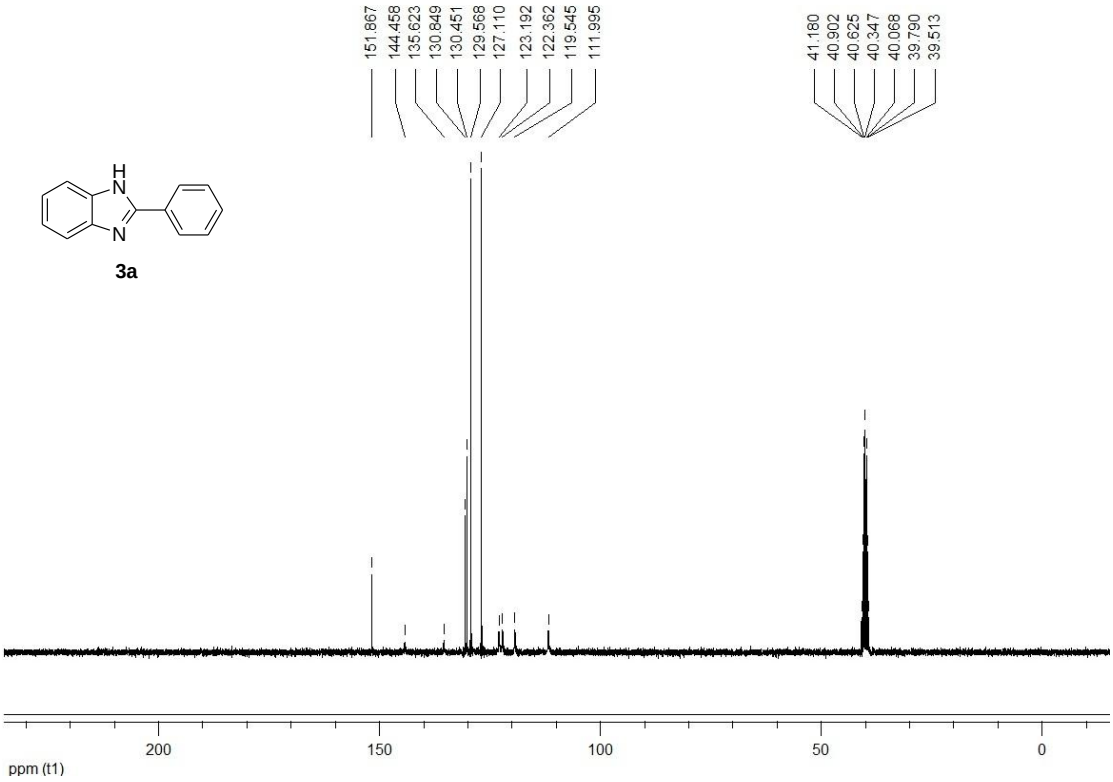

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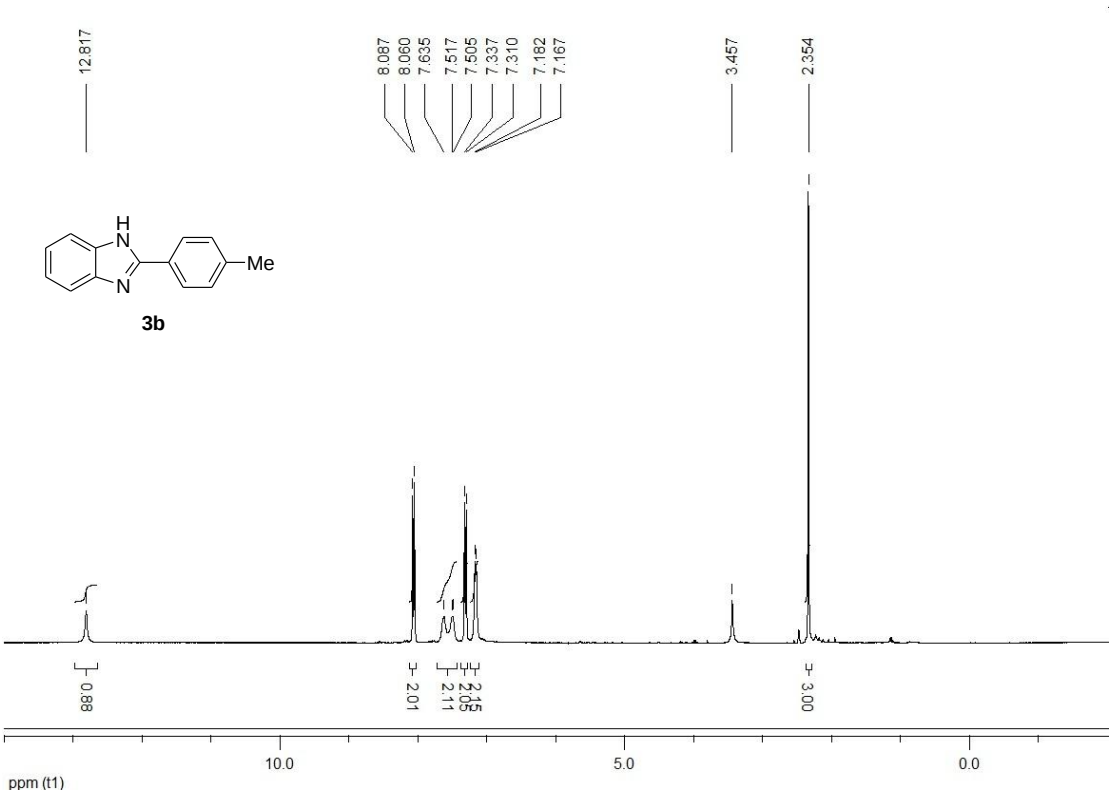

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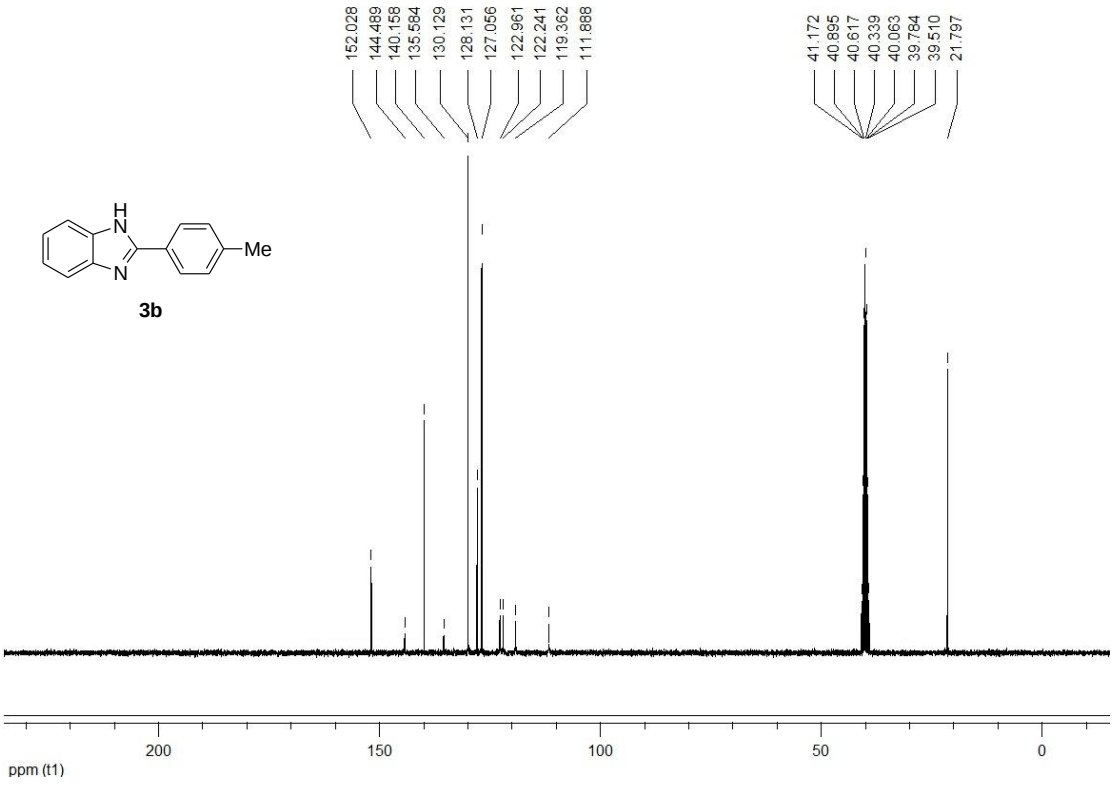

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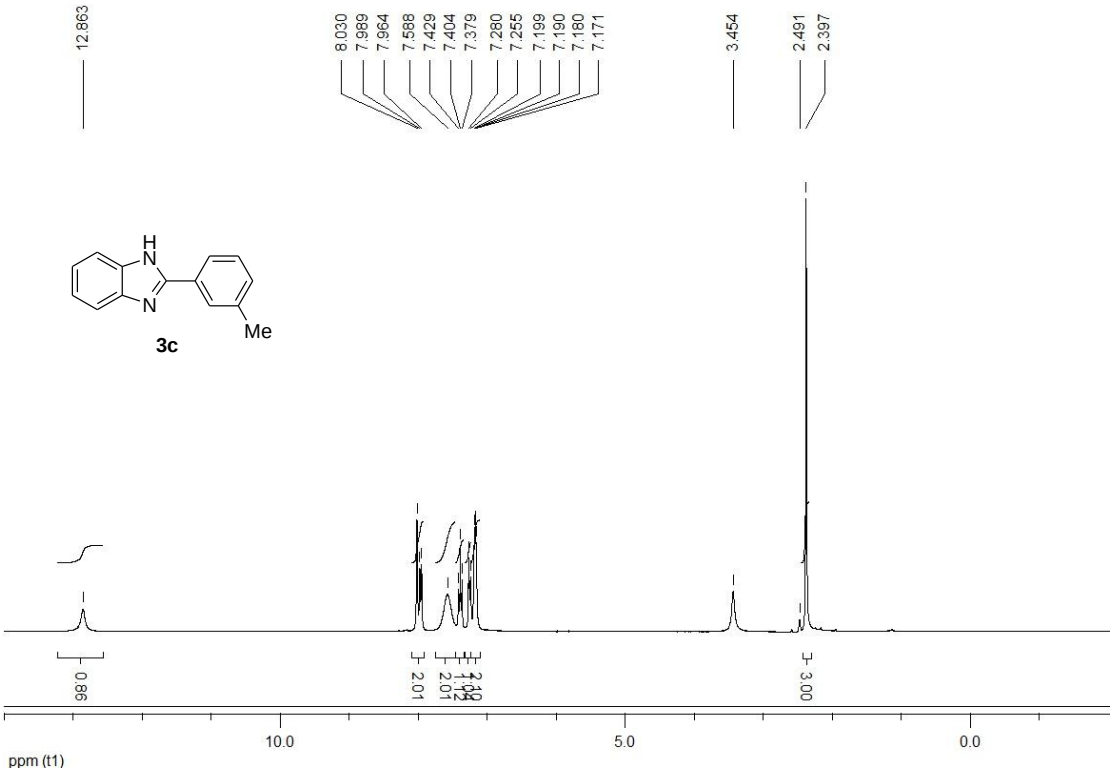
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7.379
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7.255
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7.180
7.171
3.454
2.491
2.397
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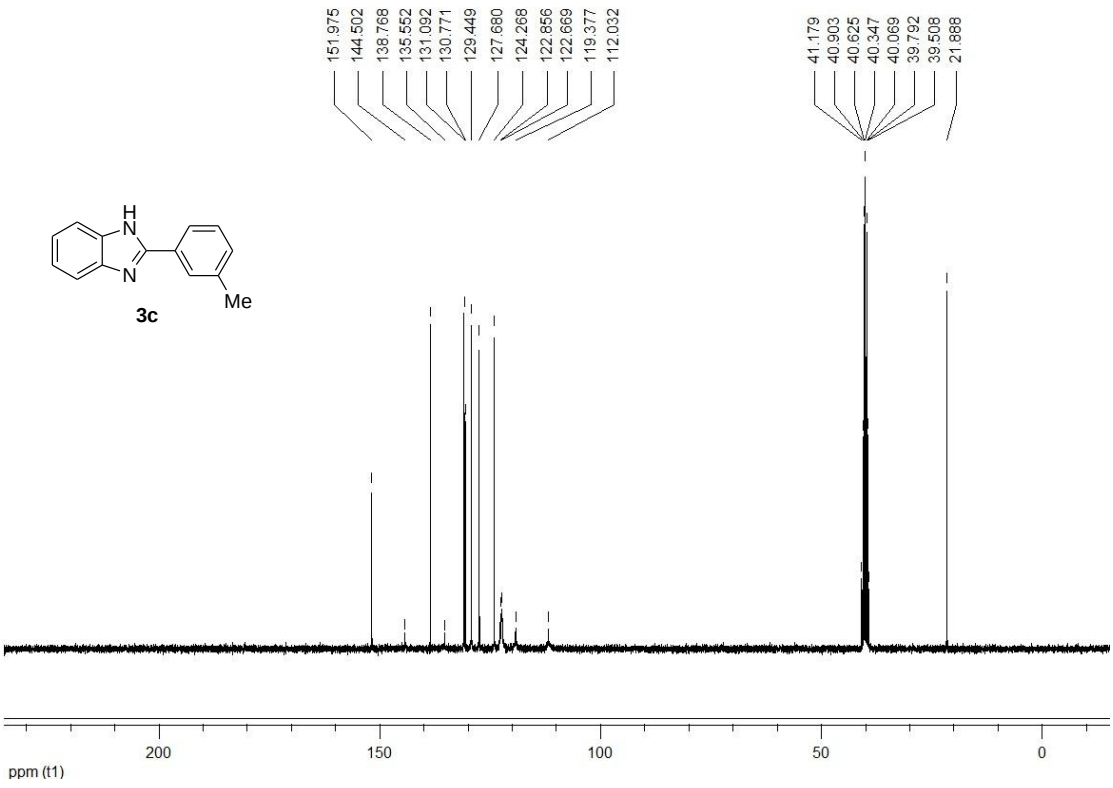
N
N
Me
3c
0.86
2.01
2.01
2.10
3.00
10.0
5.0
0.0
ppm (t1)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144.502
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129.449
127.680
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122.856
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119.377
112.032
41.179
40.903
40.625
40.347
40.069
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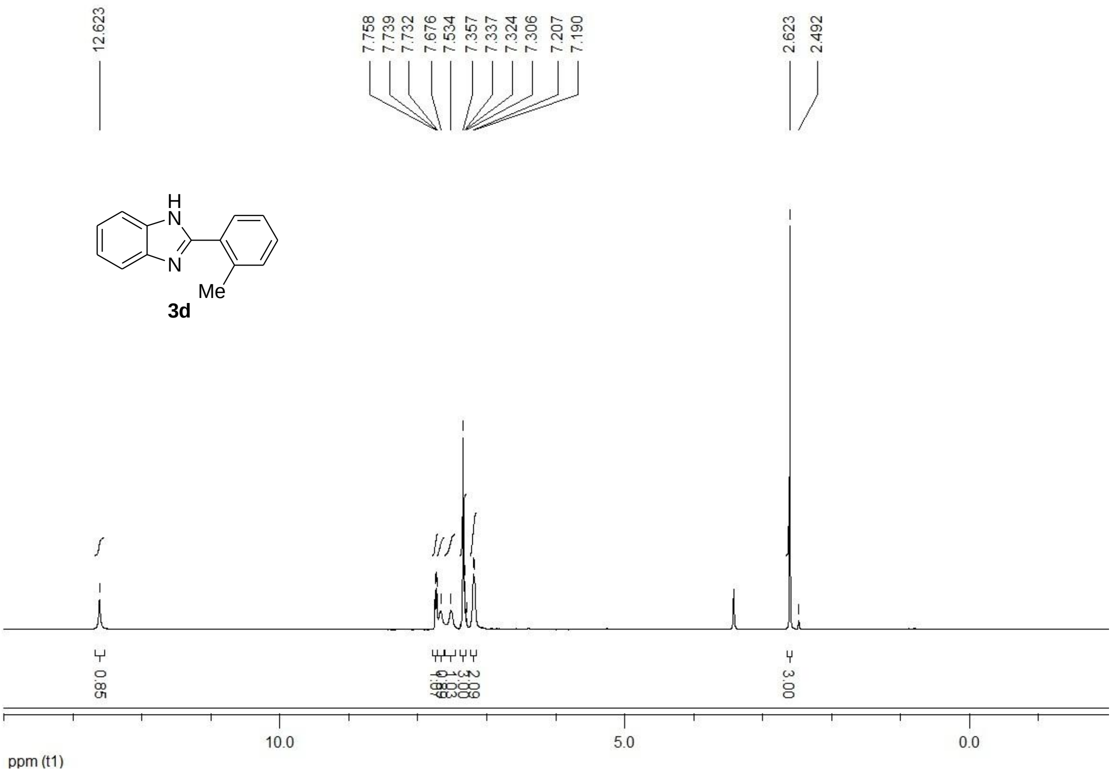
39.508
21.888
H
N
N
Me
3c
200
150
100
50
0
ppm (t1)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7.739
7.732
7.676
7.534
7.357
7.337
7.324
7.306
7.207
7.190
2.623
2.492
H
N
N
Me
3d
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0.89
1.03
3.00
2.09
3.00
10.0
5.0
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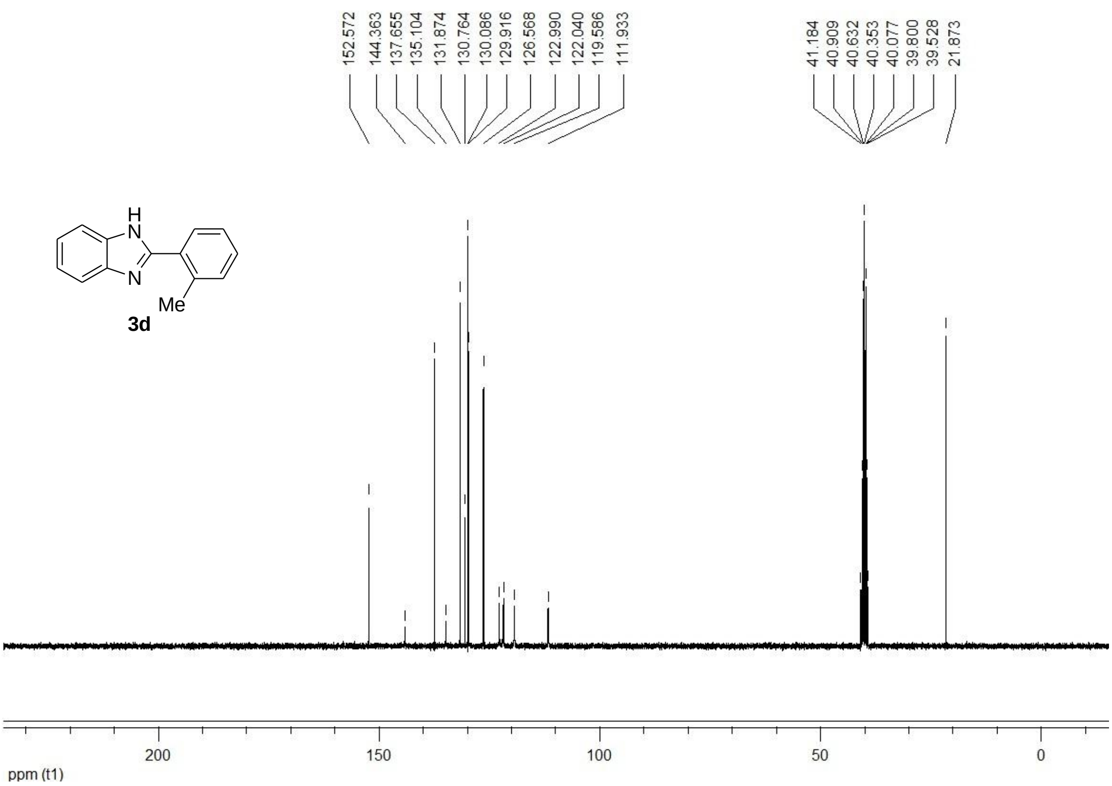
ppm (t1)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144.363
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130.764
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129.916
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119.586
111.933
41.184
40.909
40.632
40.353
40.077
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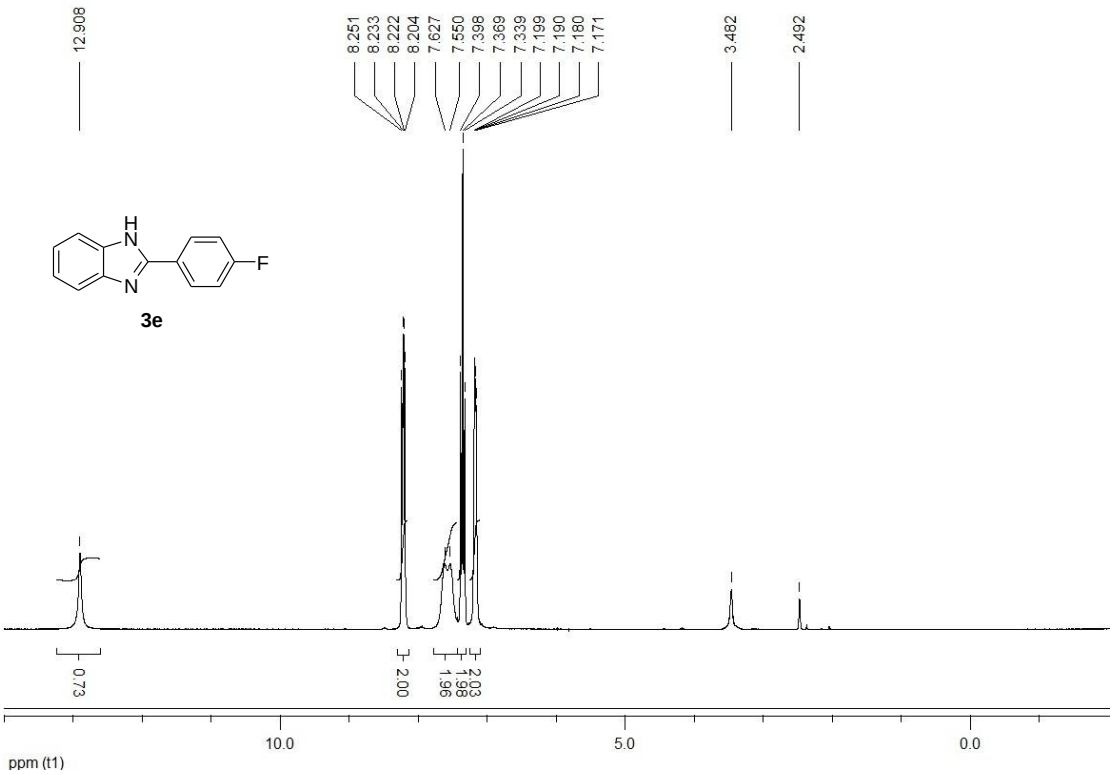
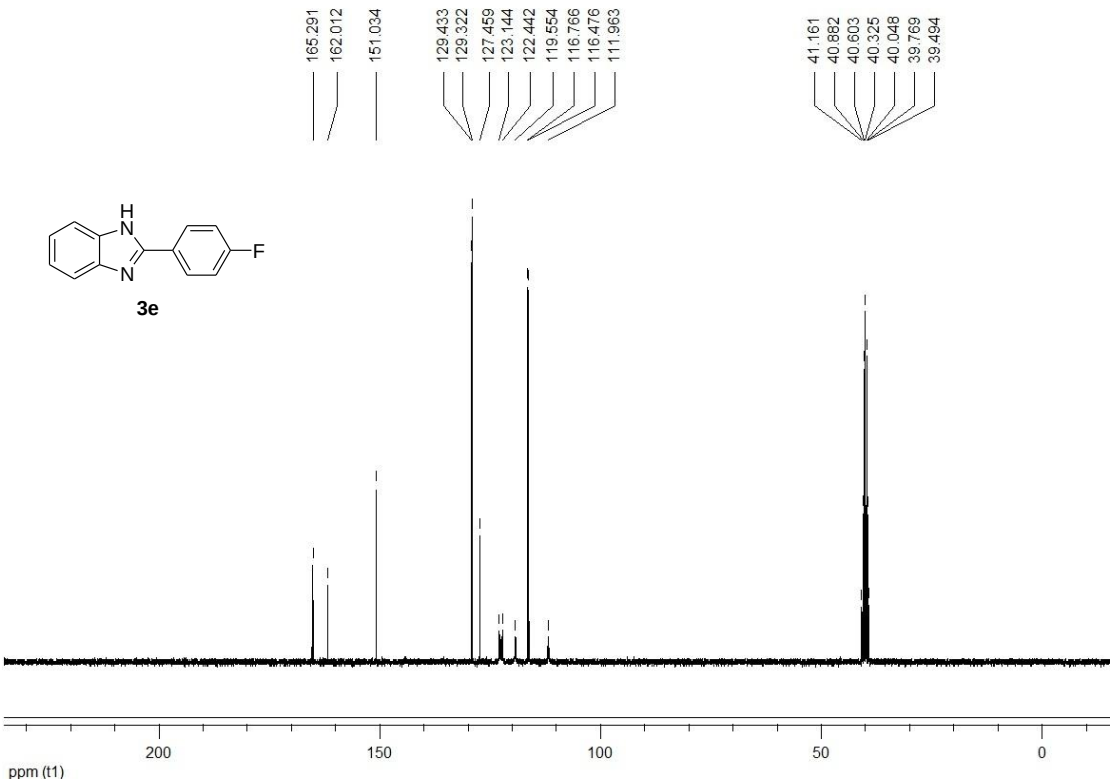
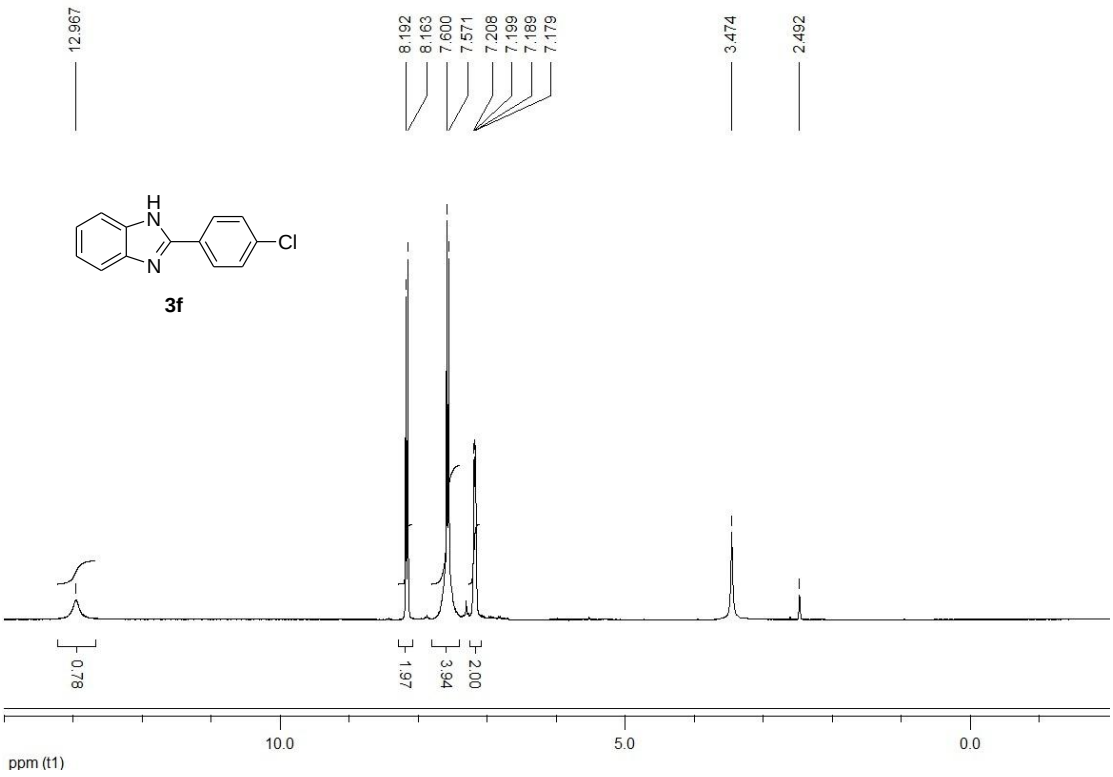
39.528
21.873
H
N
N
Me
3d
200
150
100
50
0
ppm (t1)







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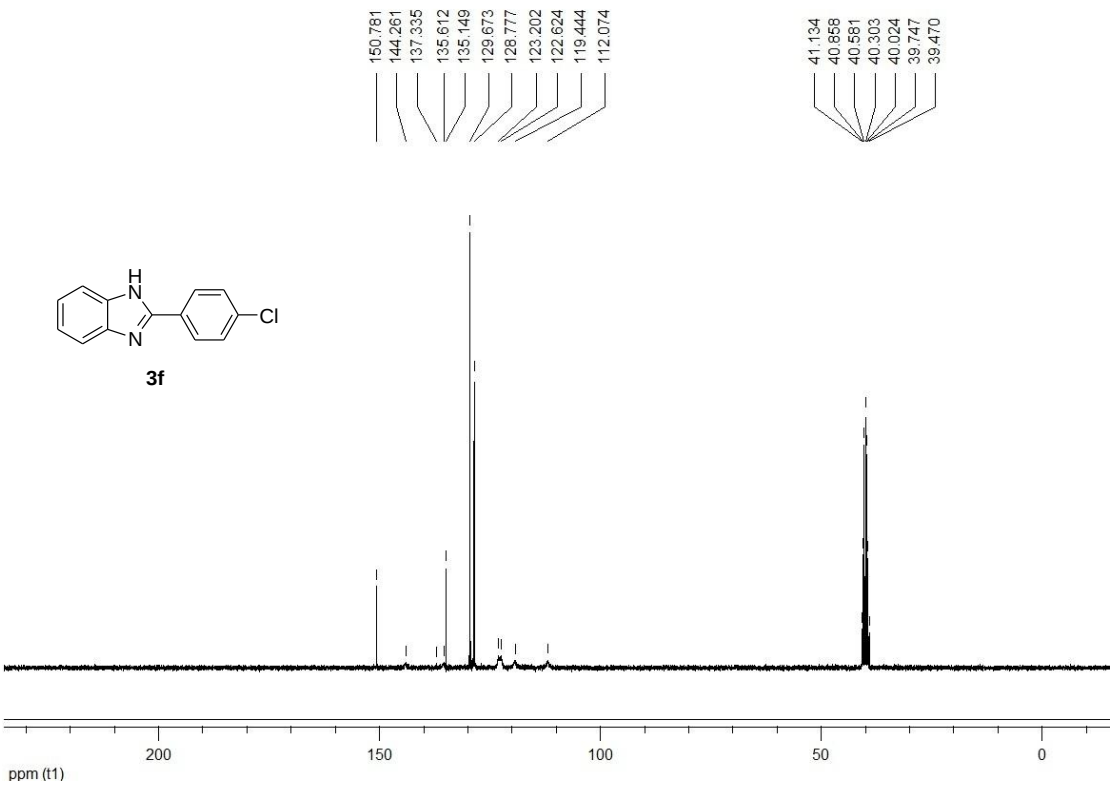

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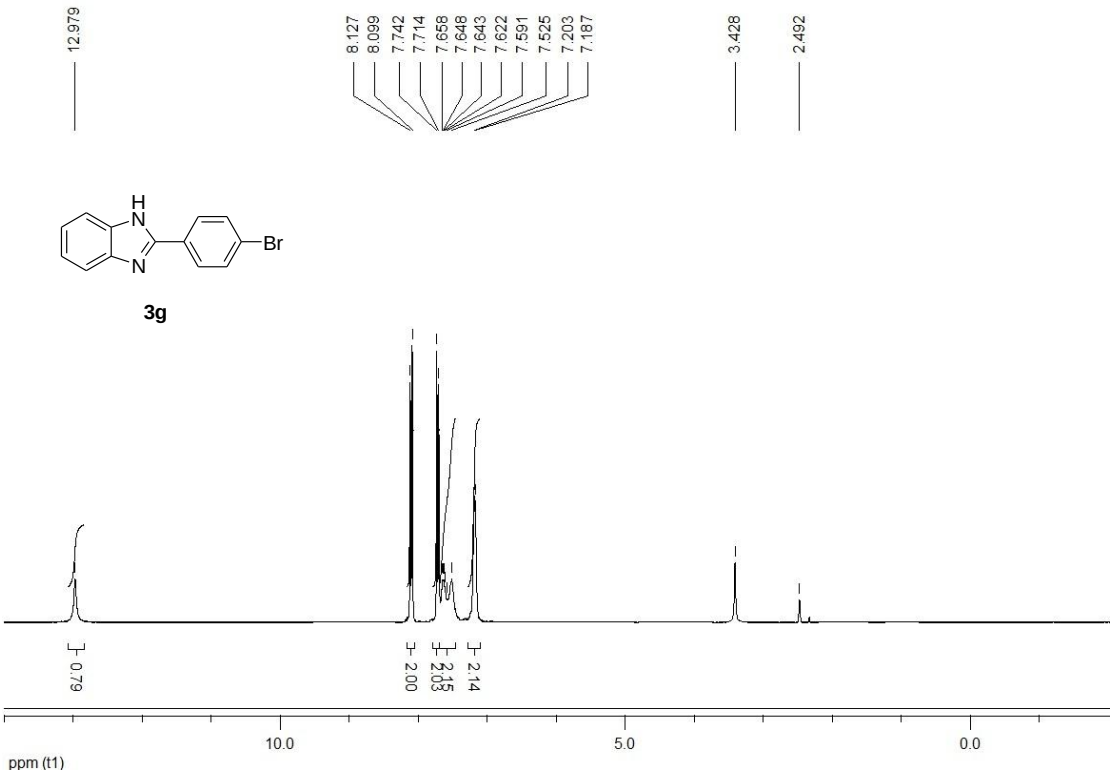
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8.099
7.742
7.714
7.658
7.648
7.643
7.622
7.591
7.525
7.203
7.187
3.428
2.492
H
N
N
Br
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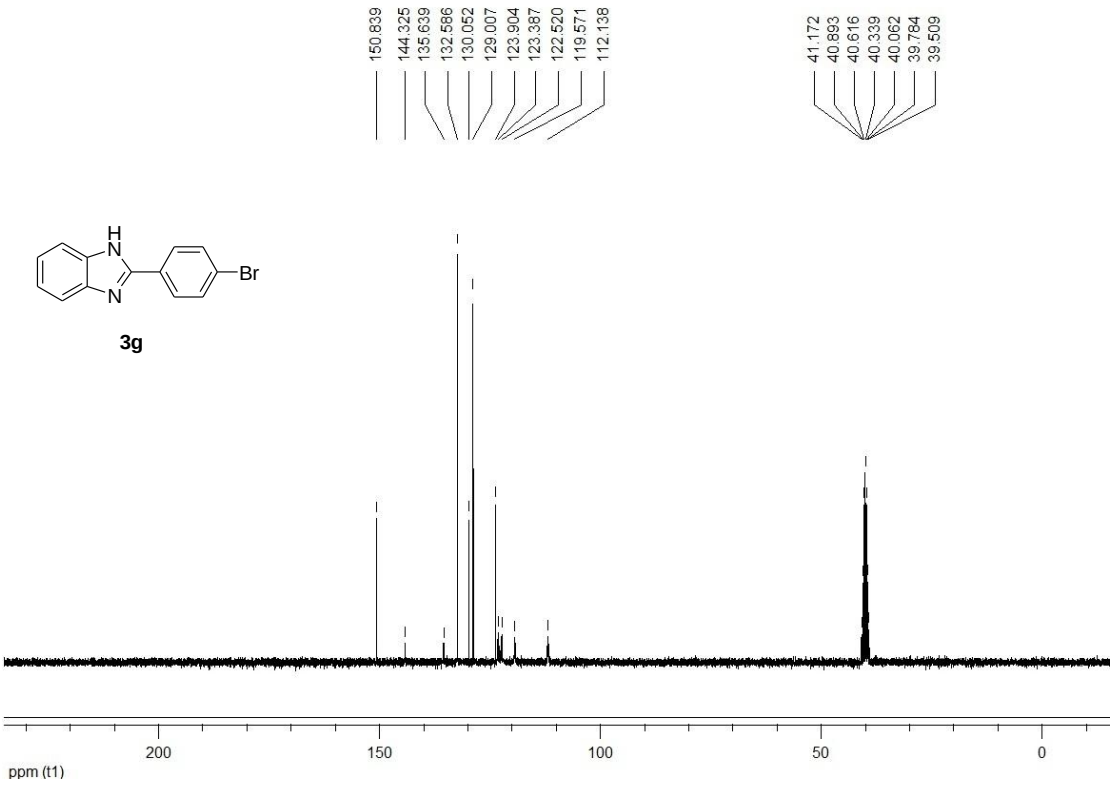
2.00
2.15
2.03
2.14
10.0
5.0
0.0
ppm (t1)

3g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135.639
132.586
130.052
129.007
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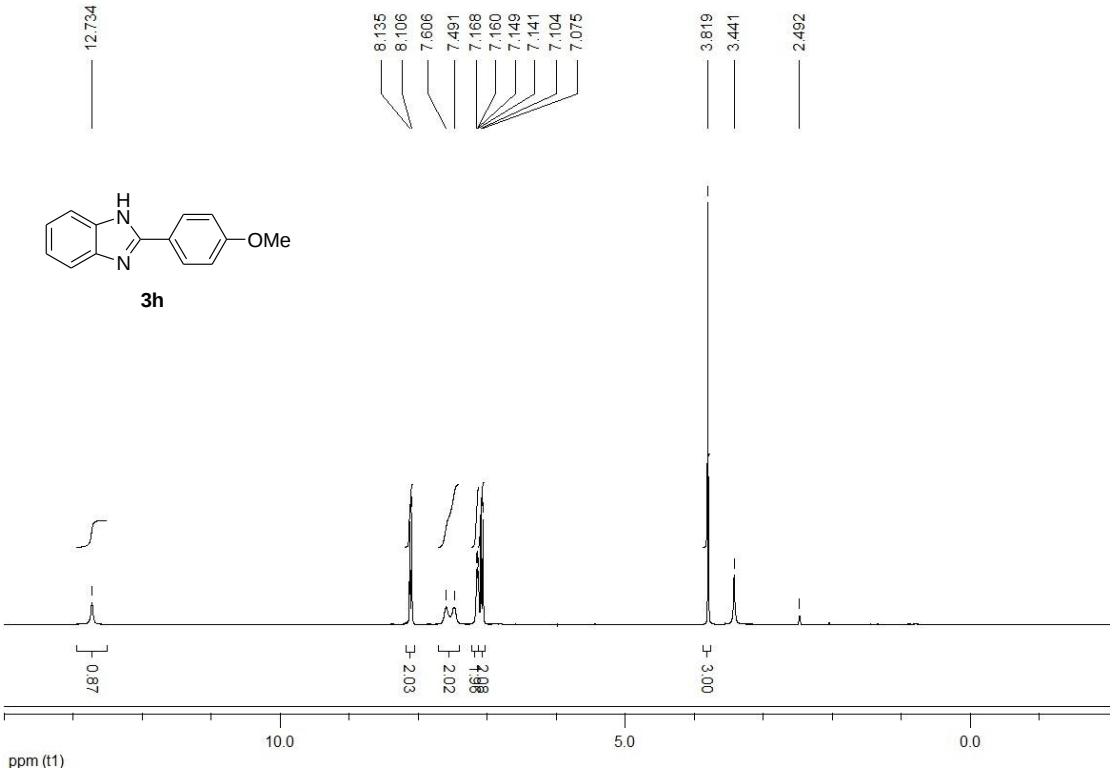
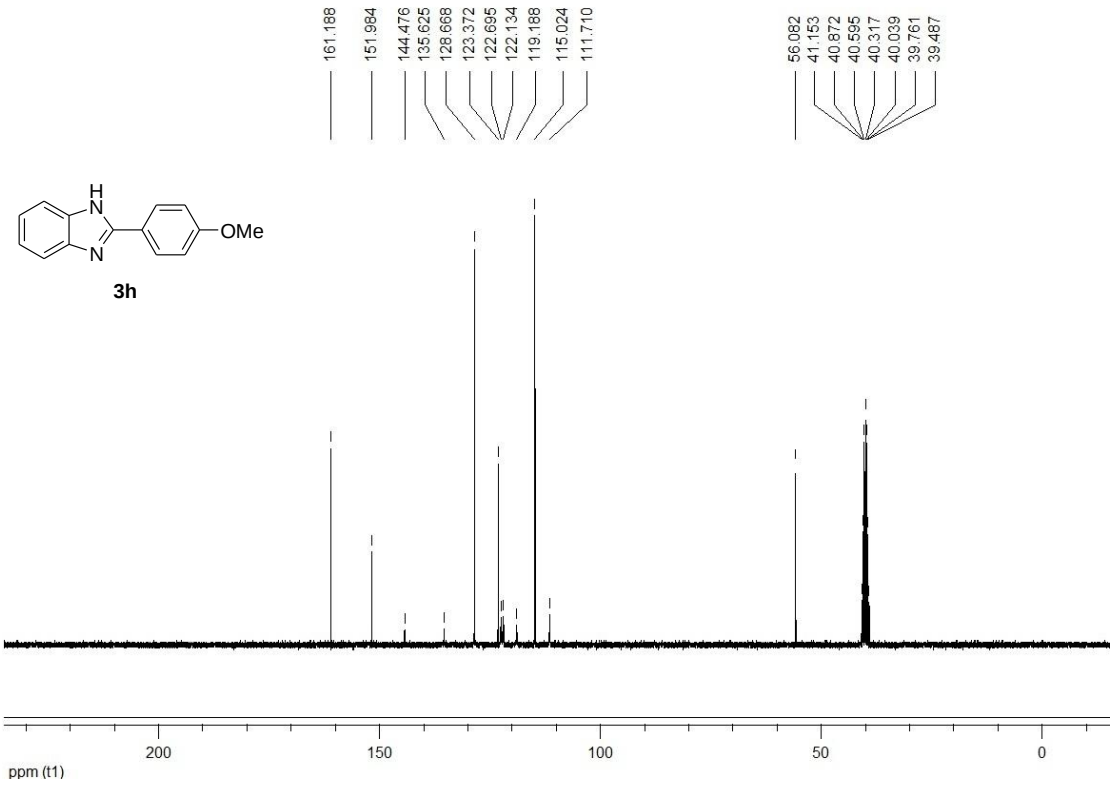
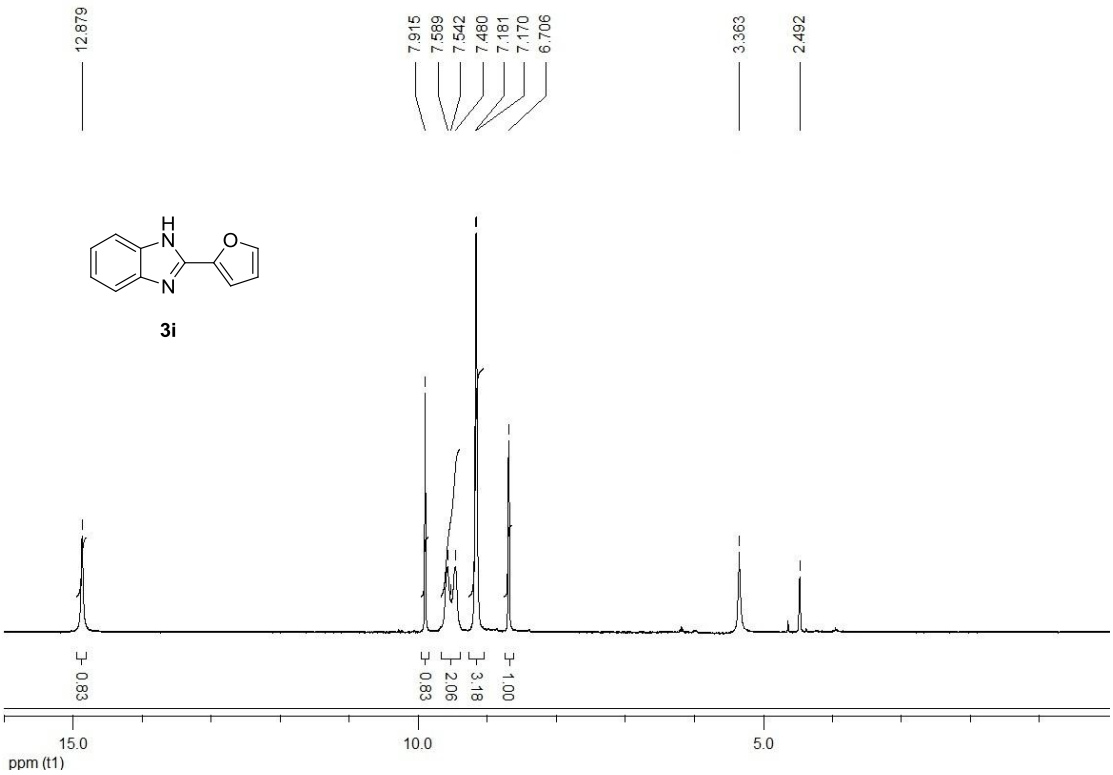
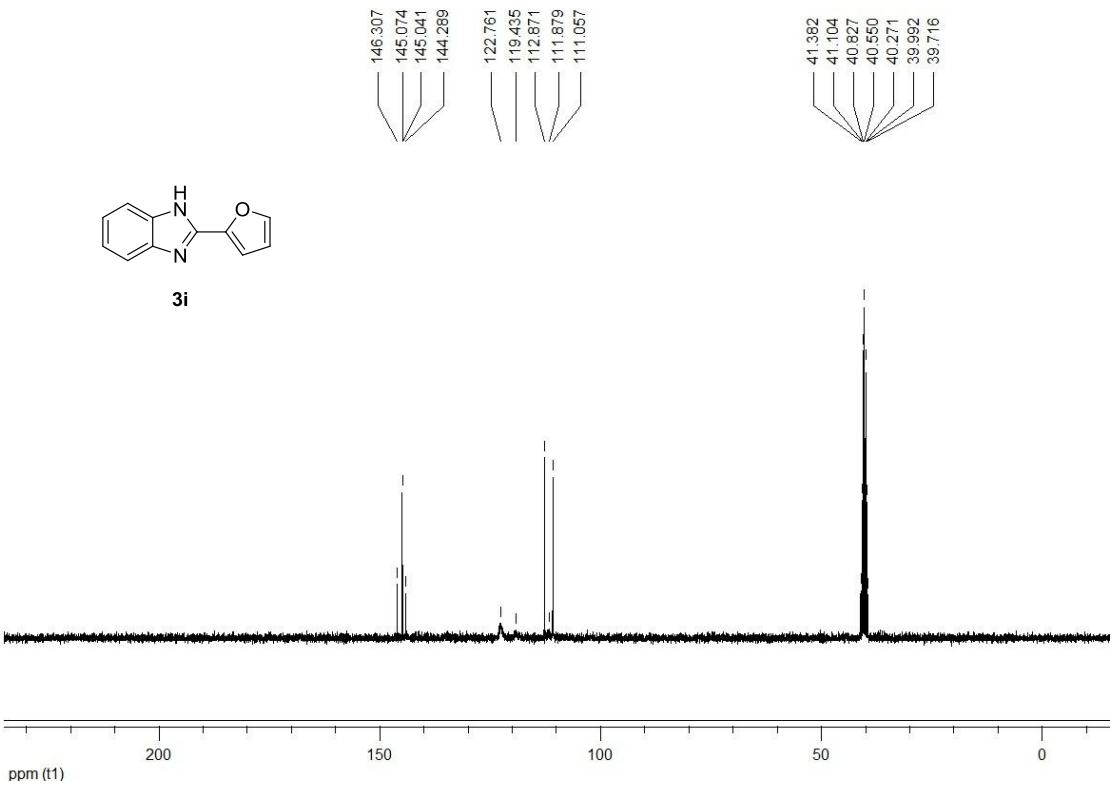
122.520
119.571
112.138
41.172
40.893
40.616
40.339
40.062
39.784
39.509
H
N
N
Br
200
150
100
50
0
ppm (t1)

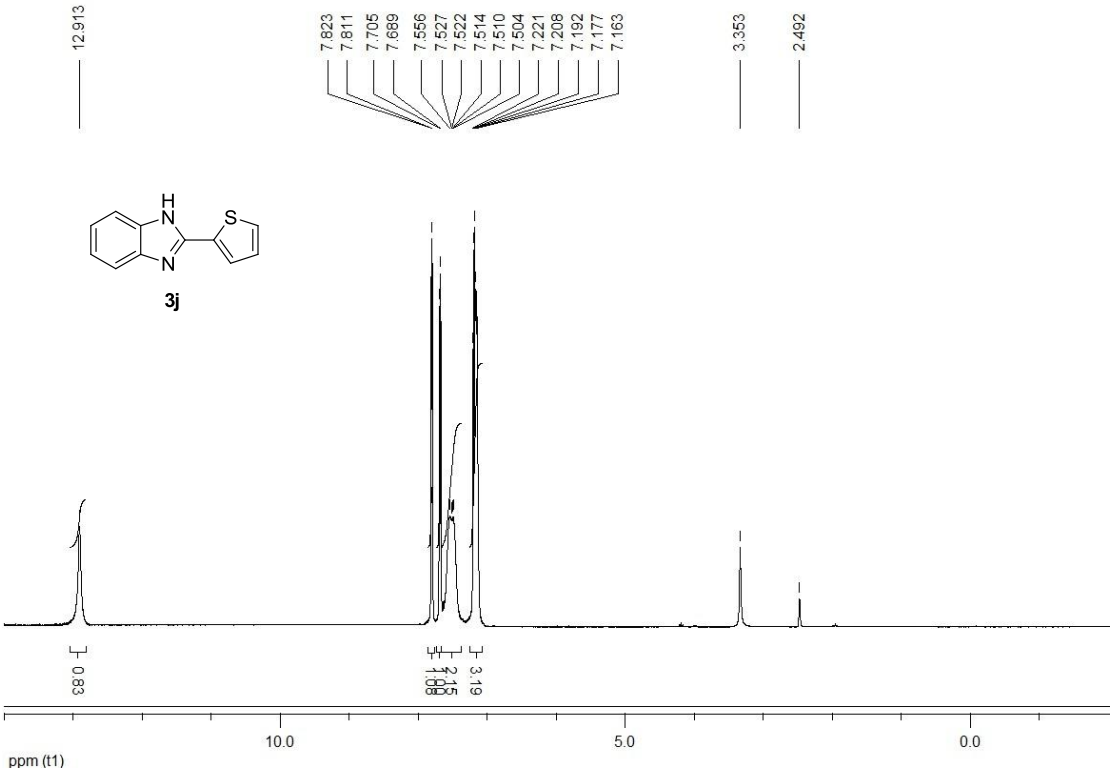
3g







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7.556
7.527
7.522
7.514
7.510
7.504
7.221
7.208
7.192
7.177
7.163
3.353
2.492
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10.0
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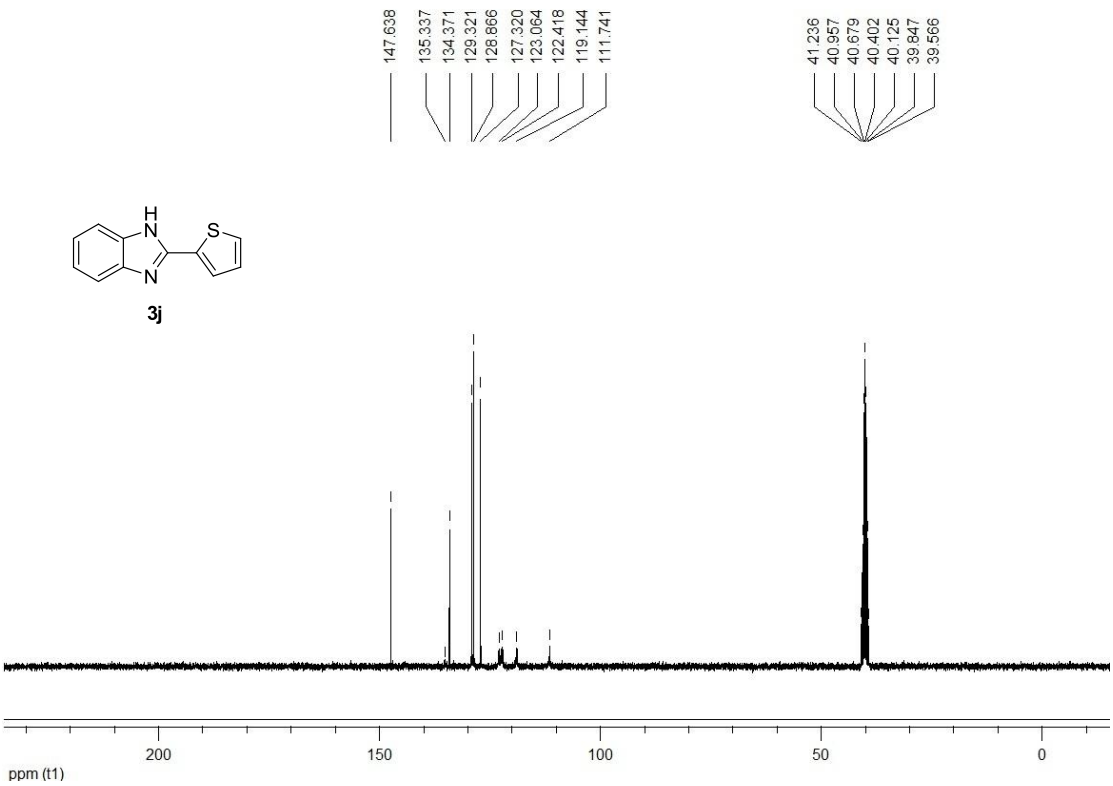
0.0
ppm (t1)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127.320
123.064
122.418
119.144
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40.125
39.847
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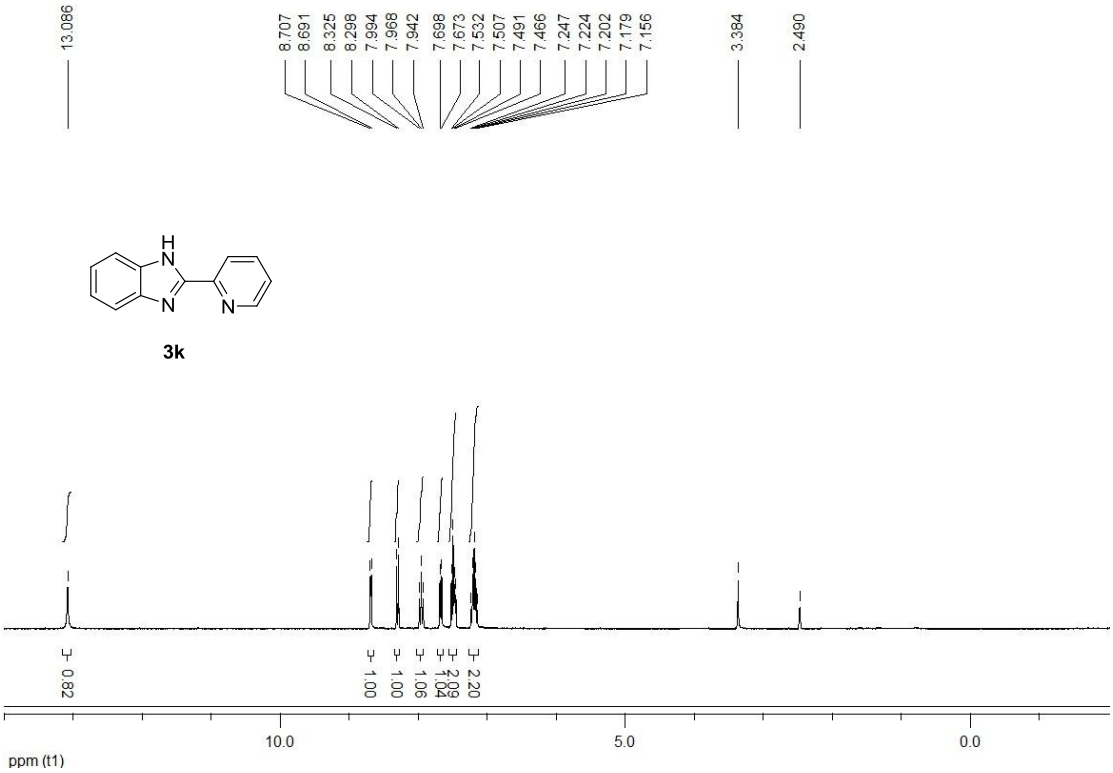
100
50
0
ppm (t1)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7.994
7.968
7.942
7.698
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7.532
7.507
7.491
7.466
7.247
7.224
7.202
7.179
7.156
3.384
2.490
H
N
N
N
3k
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1.00
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2.09
1.04
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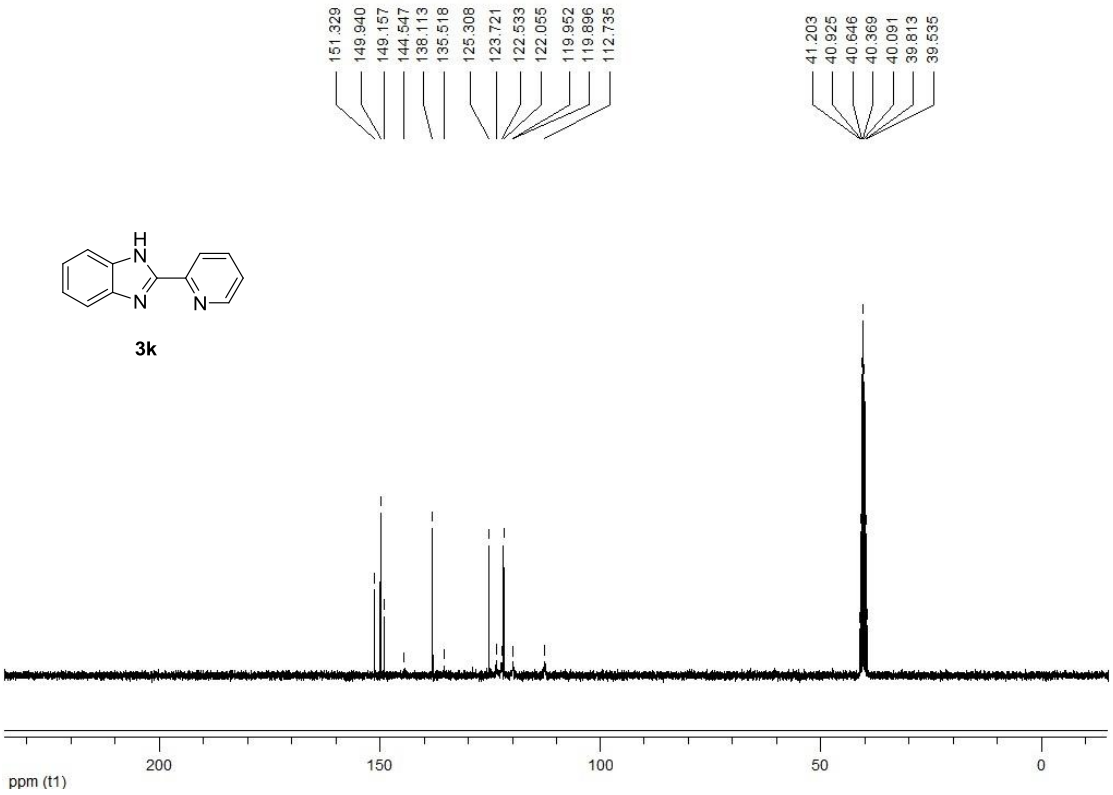
ppm (t1)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112.735
41.203
40.925
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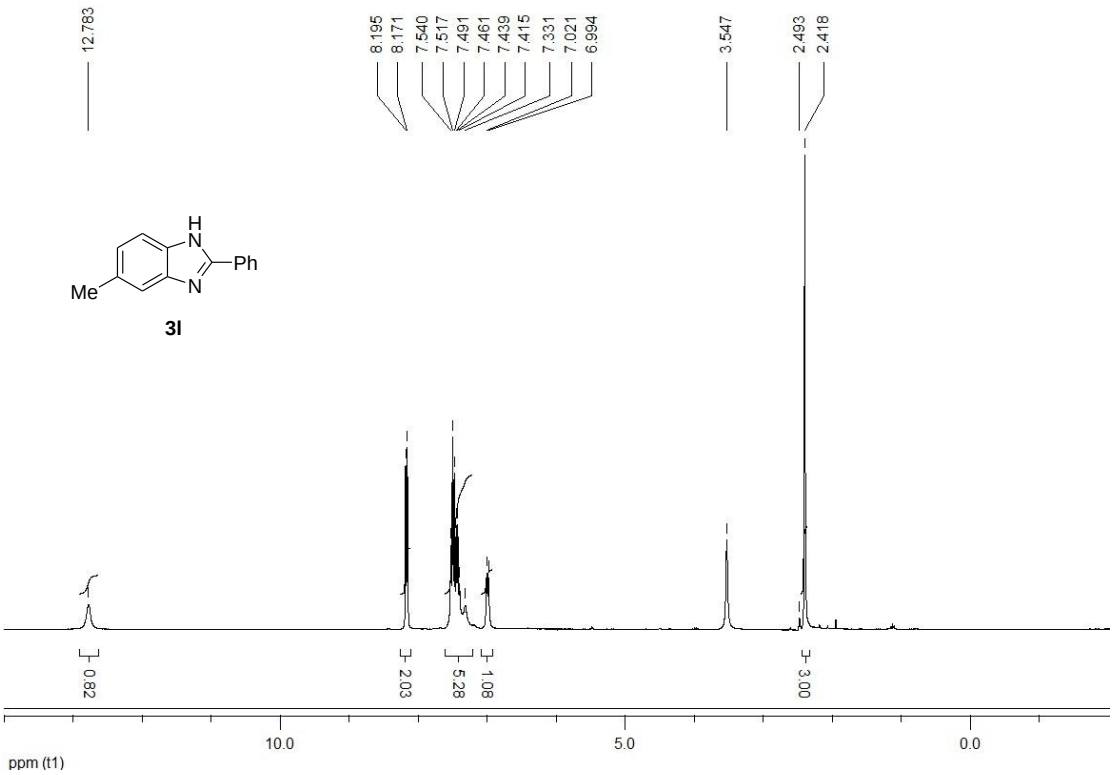
N
N
3k
200
150
100
50
0
ppm (t1)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7.439
7.415
7.331
7.021
6.994
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2.493
2.418
H
N
Ph
Me
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1.08
3.00
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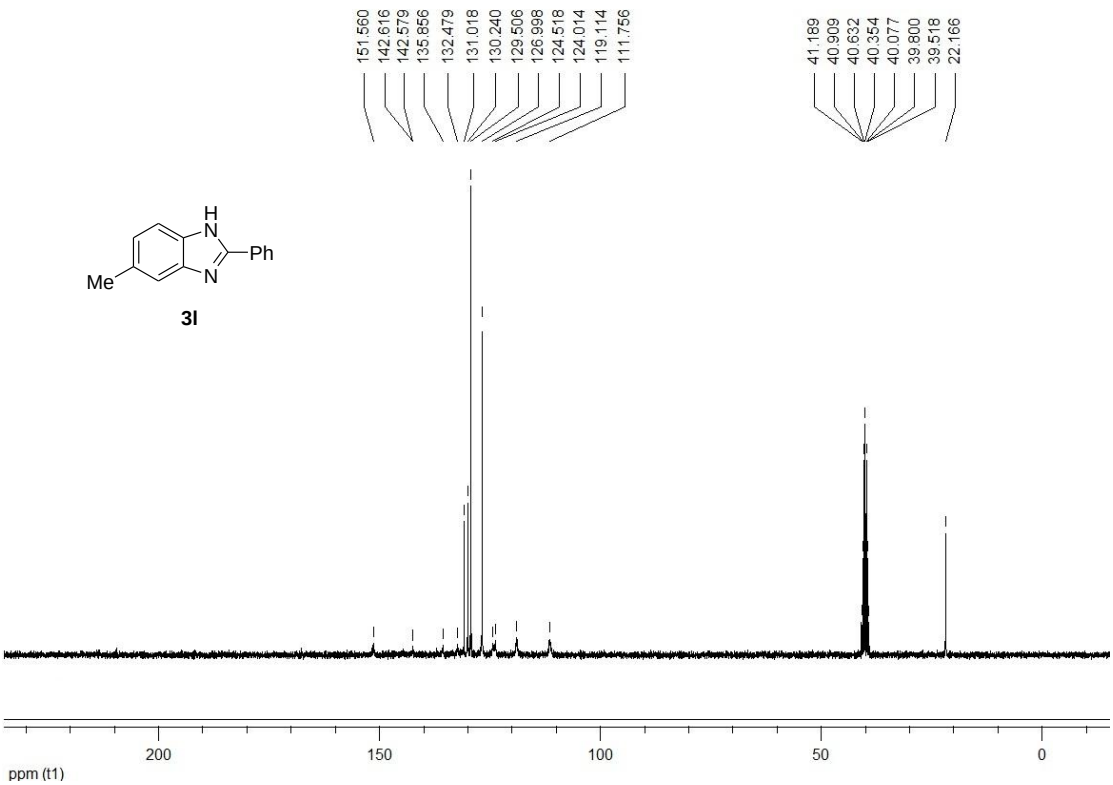
5.0
0.0
ppm (t1)

3l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124.518
124.014
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111.756
41.189
40.909
40.632
40.354
40.077
39.800
39.518
22.166
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N
Ph
Me
N
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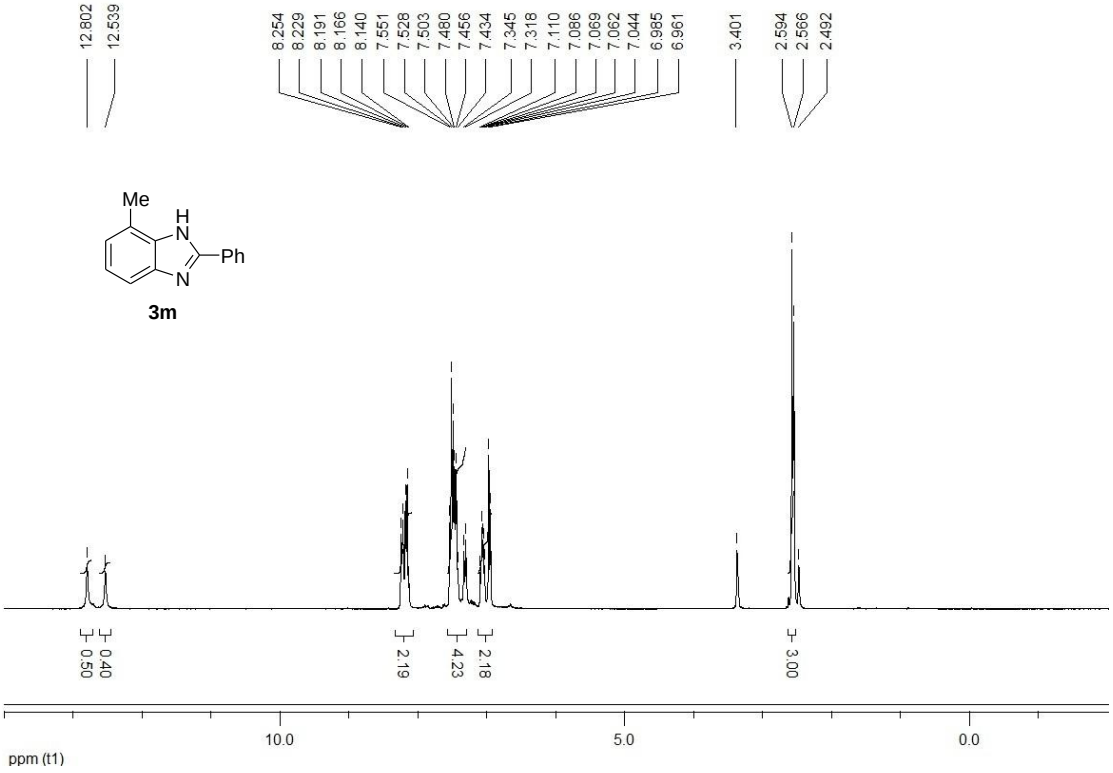
150
100
50
0
ppm (t1)

3l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8.191
8.166
8.140
7.551
7.528
7.503
7.480
7.456
7.434
7.345
7.318
7.110
7.086
7.069
7.062
7.044
6.985
6.961
3.401
2.594
2.566
2.492
Me
H
N
Ph
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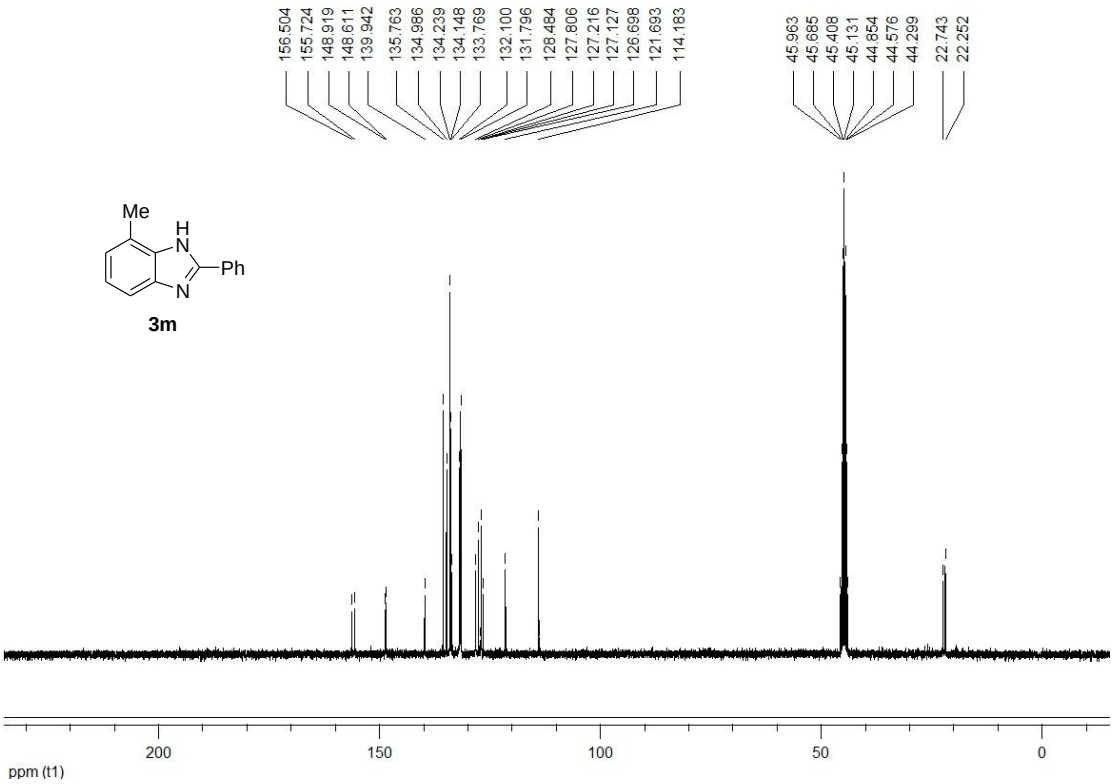
ppm (t1)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134.986
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134.148
133.769
132.100
131.796
128.484
127.806
127.216
127.127
126.698
121.693
114.183
45.963
45.685
45.408
45.131
44.854
44.576
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22.252
Me
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N
Ph
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3m
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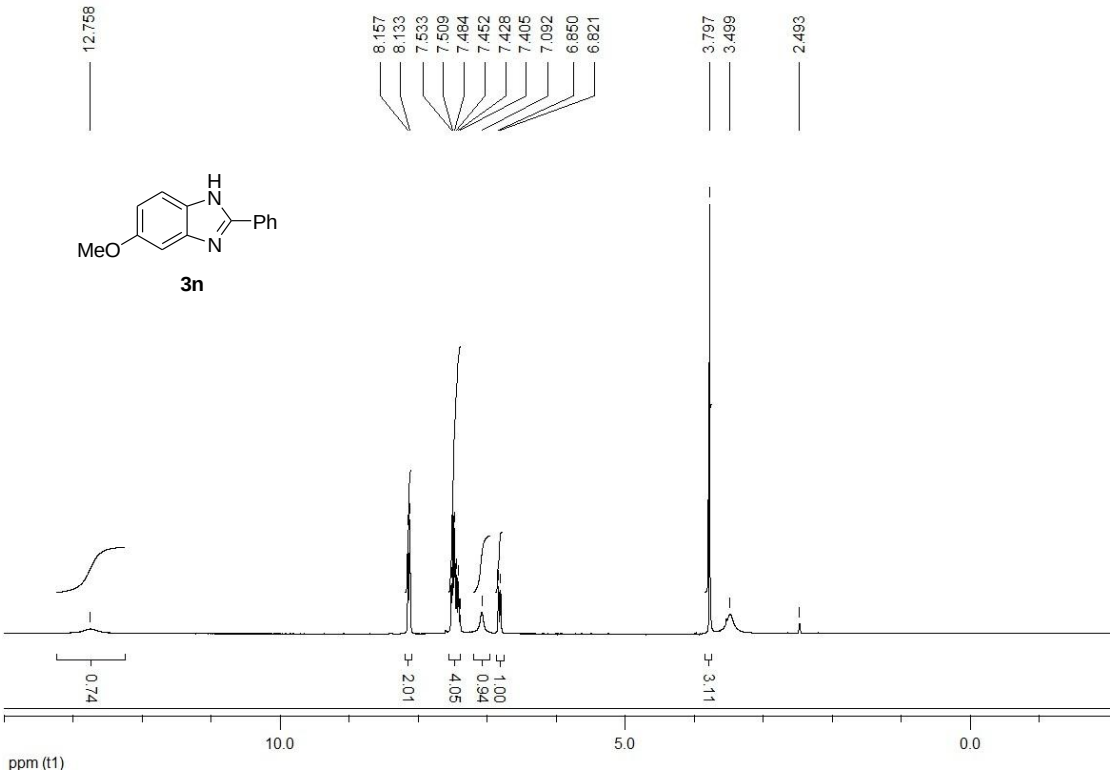
100
50
0
ppm (t1)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7.533
7.509
7.484
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7.428
7.405
7.092
6.850
6.821
3.797
3.499
2.493
H
N
Ph
MeO
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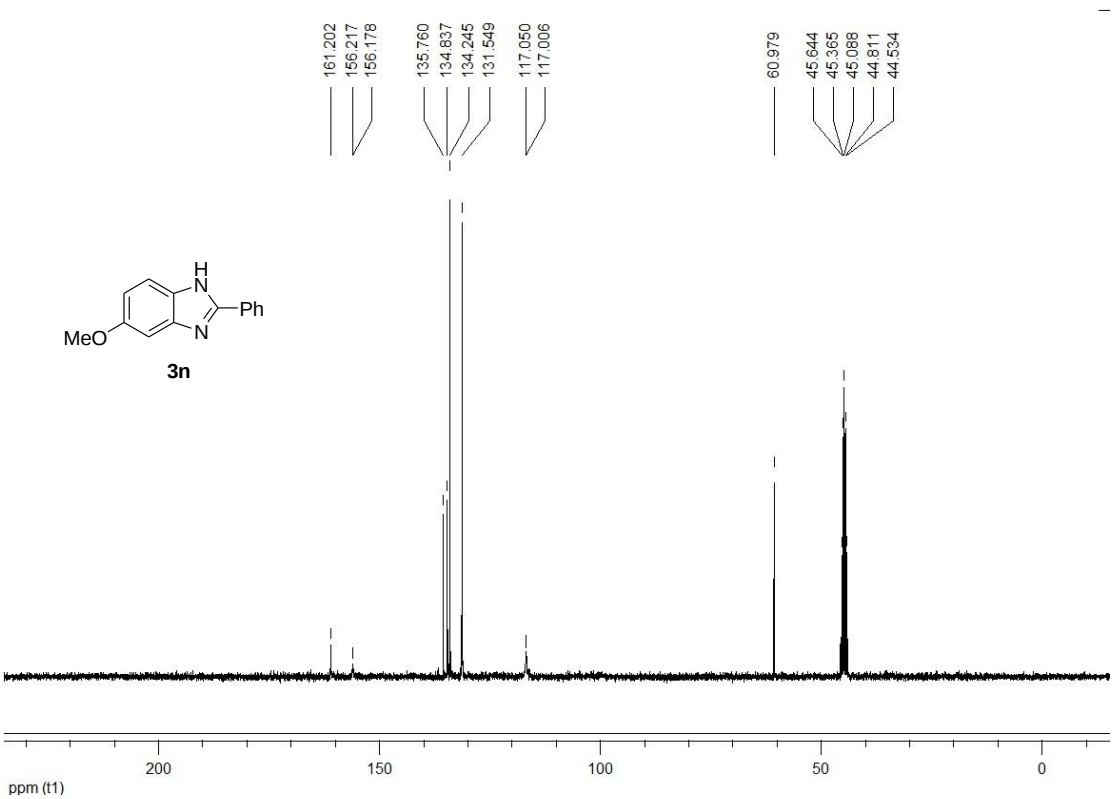
ppm (t1)

3n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156.178
135.760
134.837
134.245
131.549
117.050
117.006
60.979
45.644
45.365
45.088
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44.534
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N
Ph
MeO
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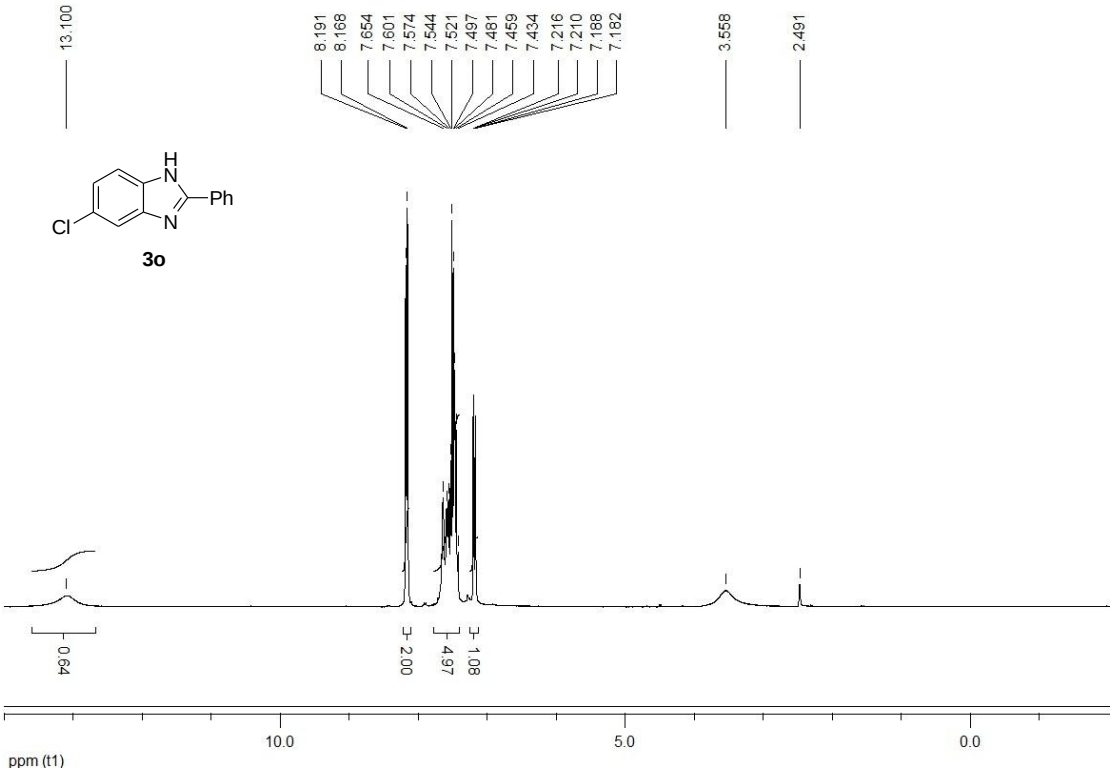
150
100
50
0
ppm (t1)

3n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7.654
7.601
7.574
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7.521
7.497
7.481
7.459
7.434
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7.210
7.188
7.182
3.558
2.491
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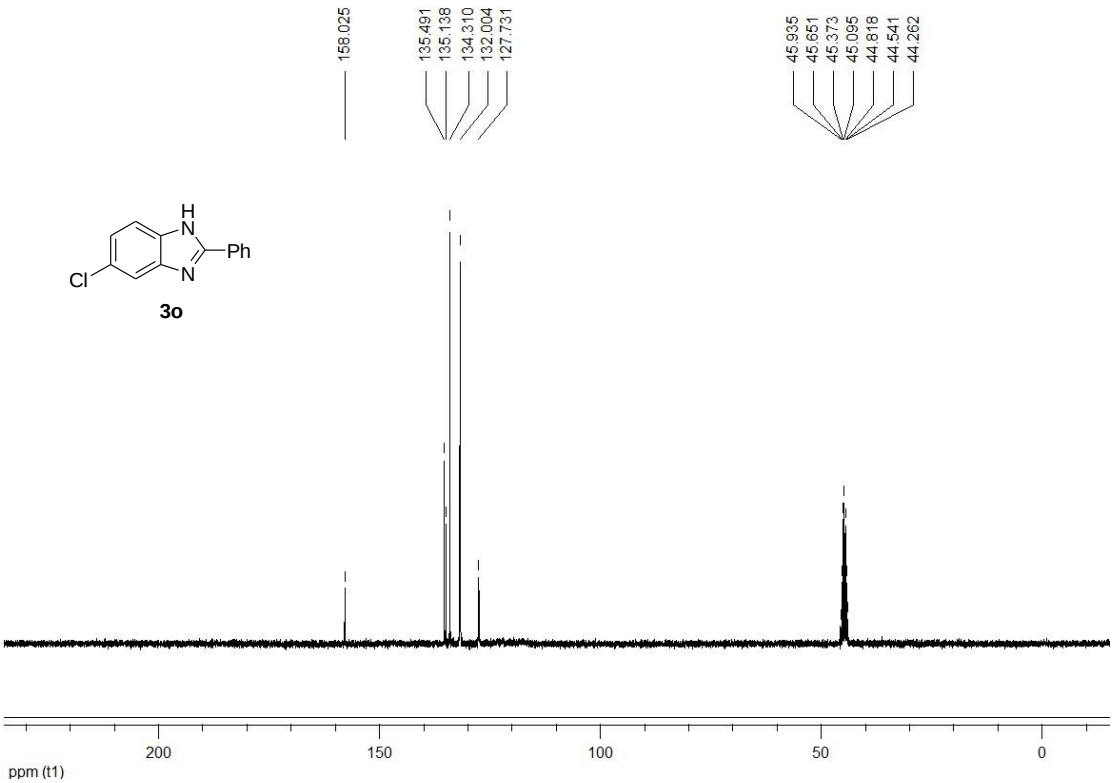
ppm (t1)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45.651
45.373
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44.262
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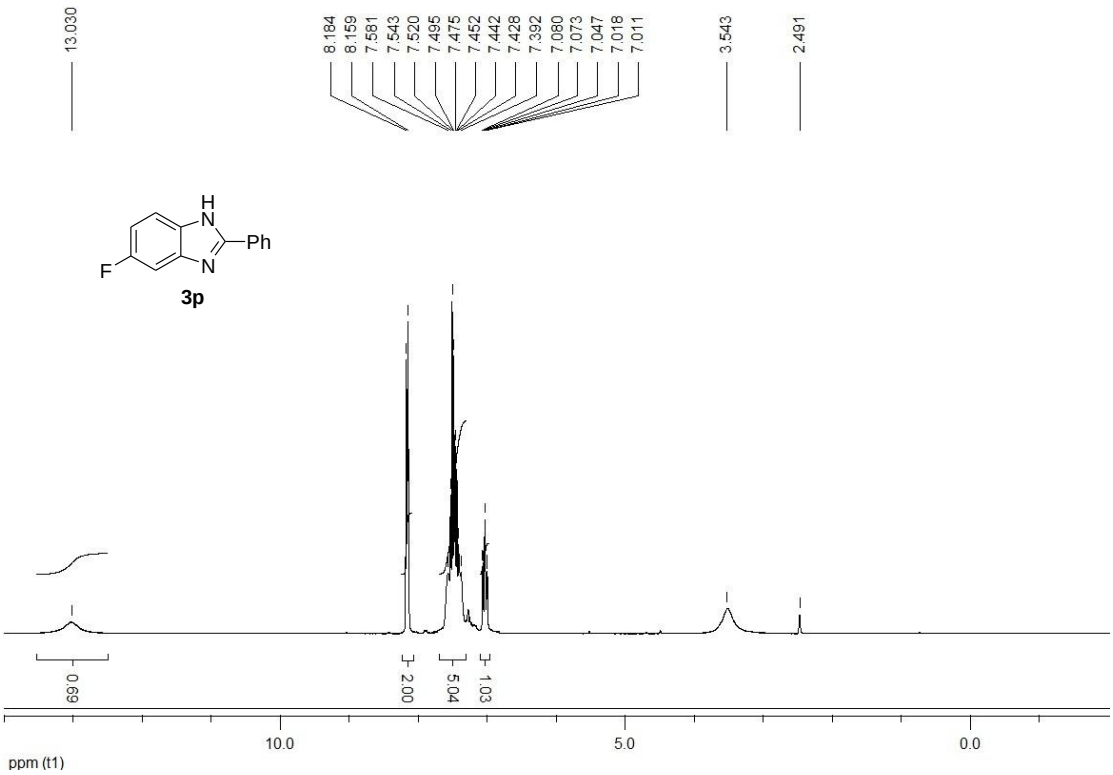
N
Ph
Cl
N
3o
200
150
100
50
0
ppm (t1)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8.159
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7.495
7.475
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7.442
7.428
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7.080
7.073
7.047
7.018
7.011
3.543
2.491
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Ph
F
N
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5.0
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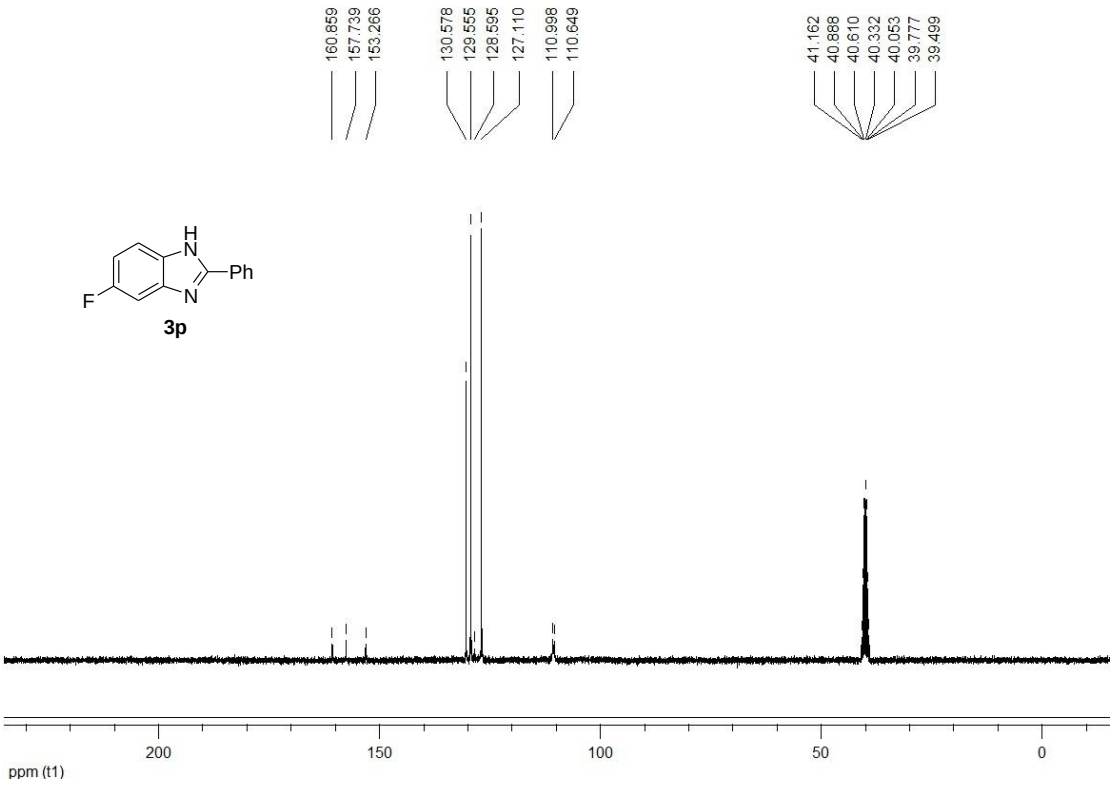
ppm (t1)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130.578
129.555
128.595
127.110
110.998
110.649
41.162
40.888
40.610
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39.499
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N
Ph
F
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3p
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150
100
50
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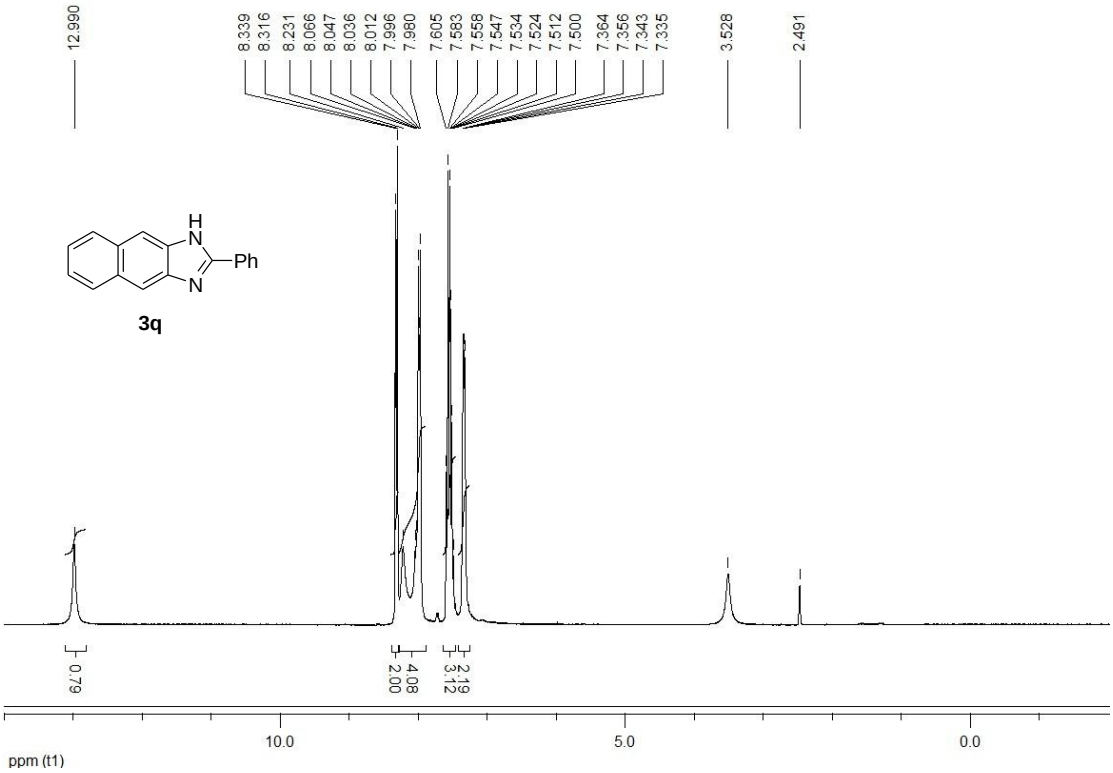
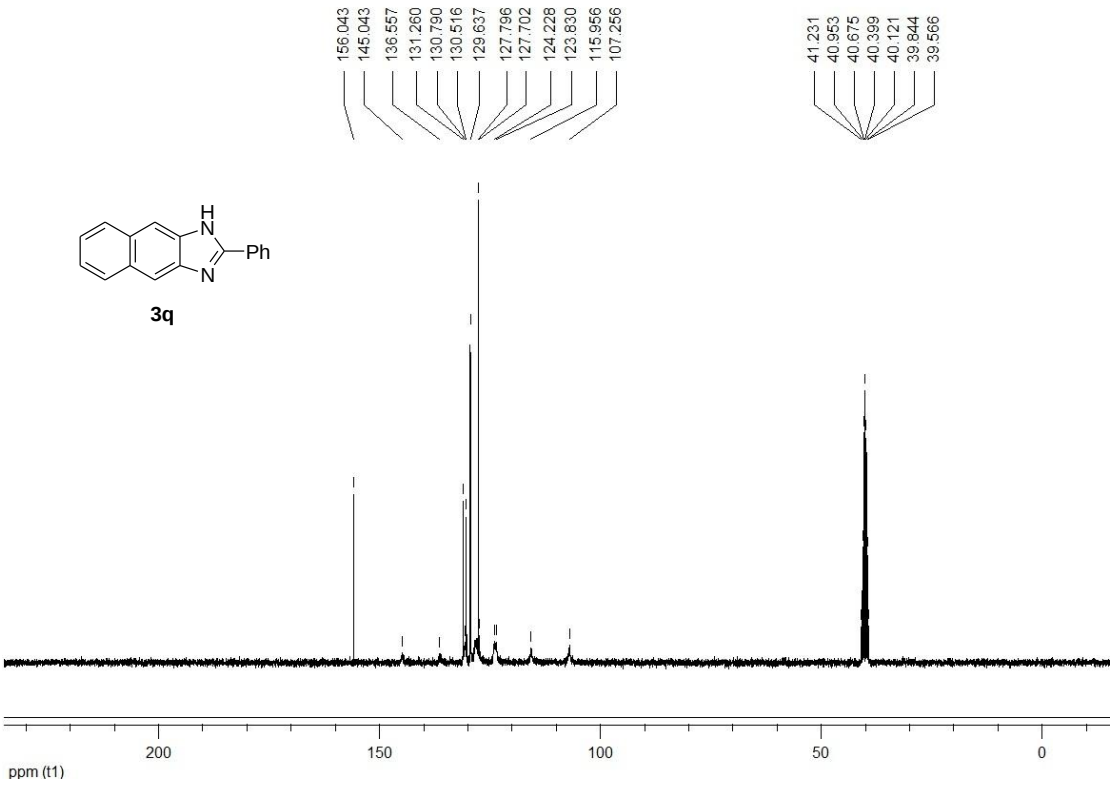
ppm (t1)



3q



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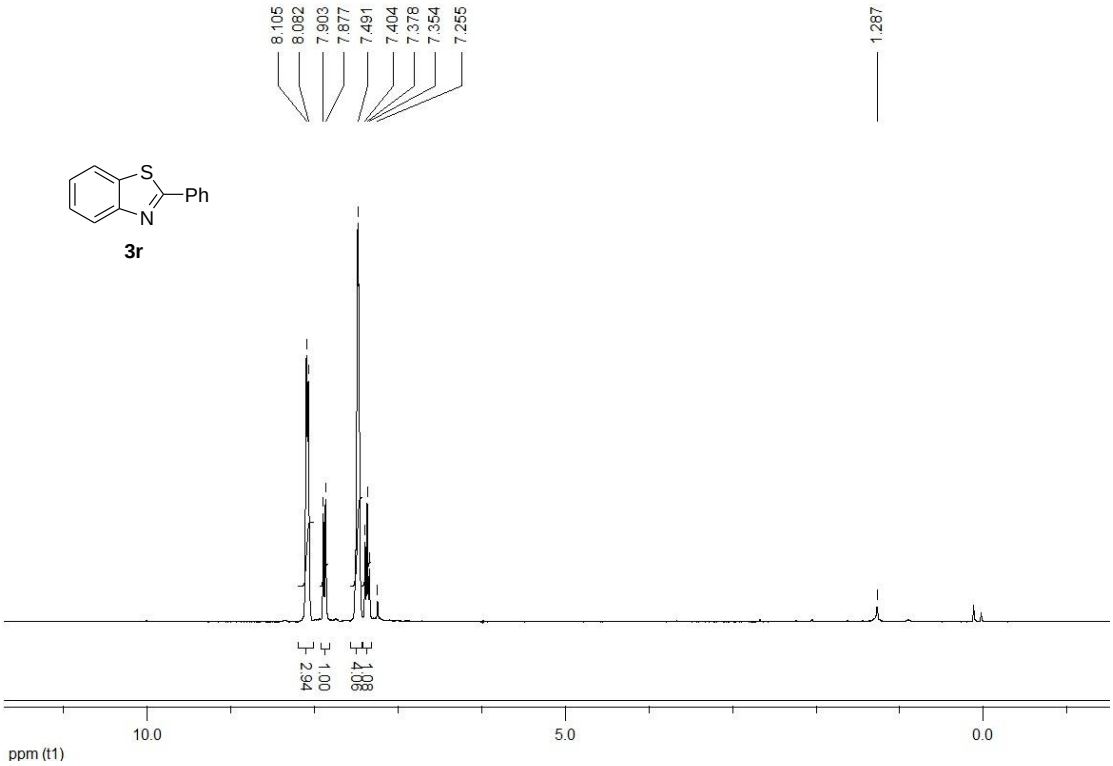

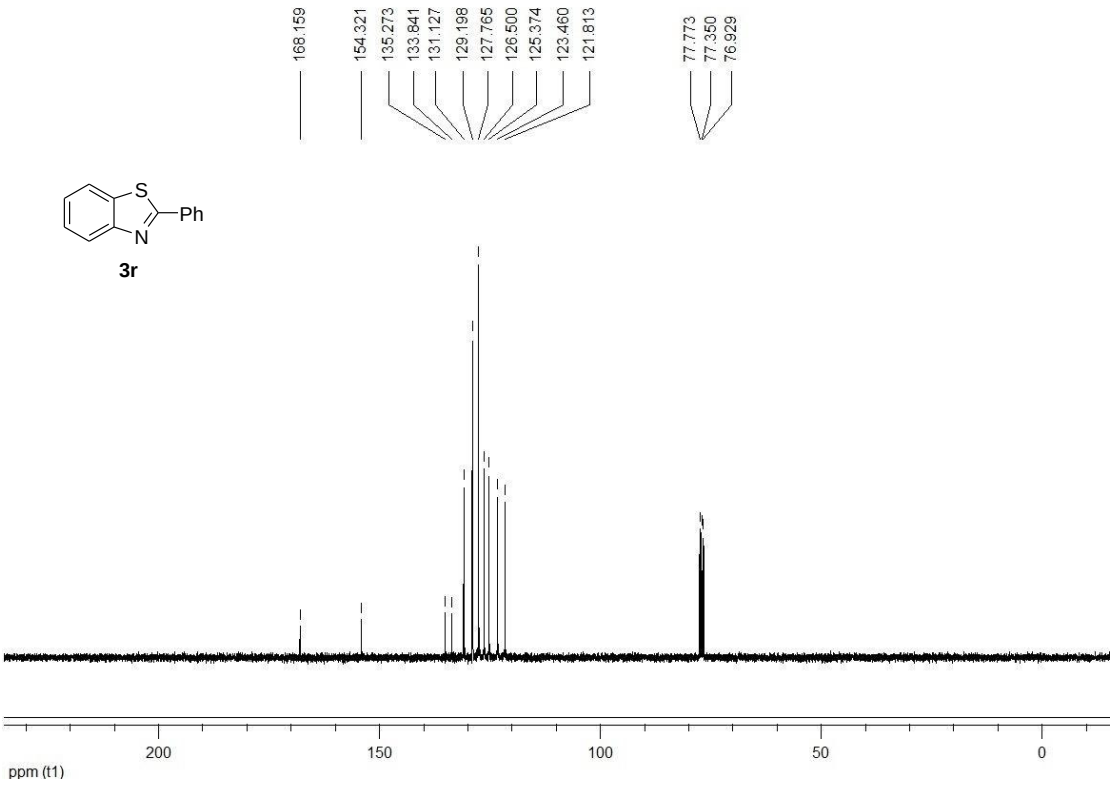

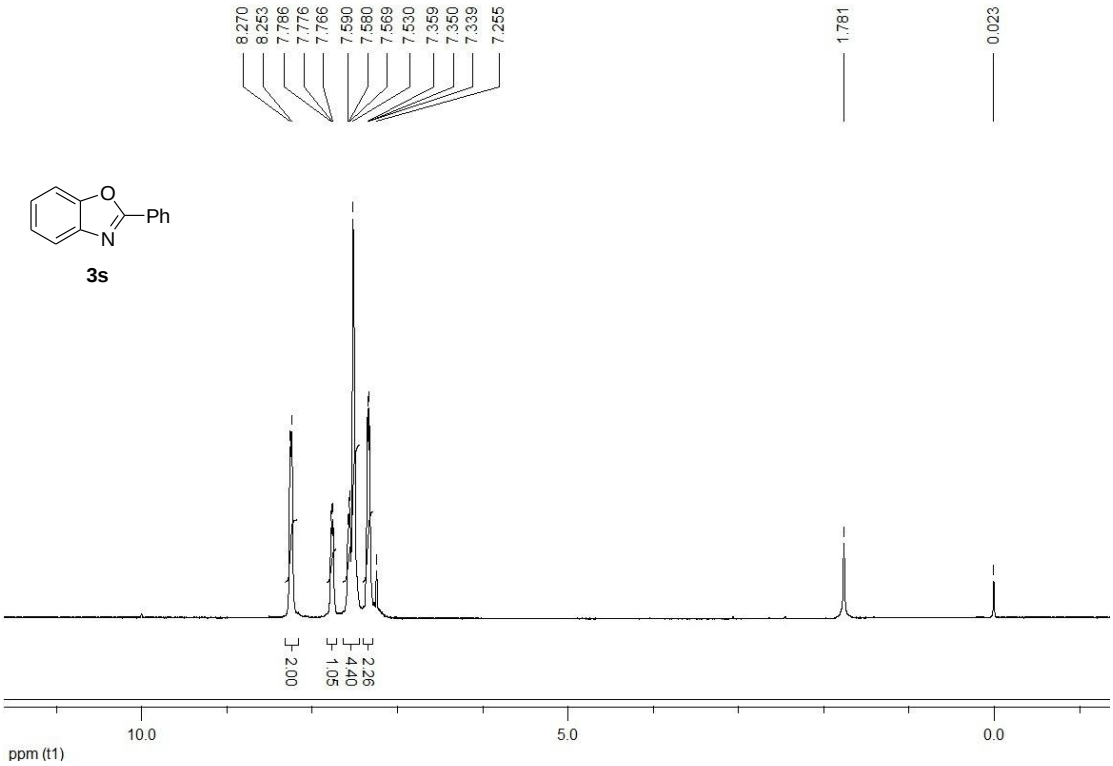
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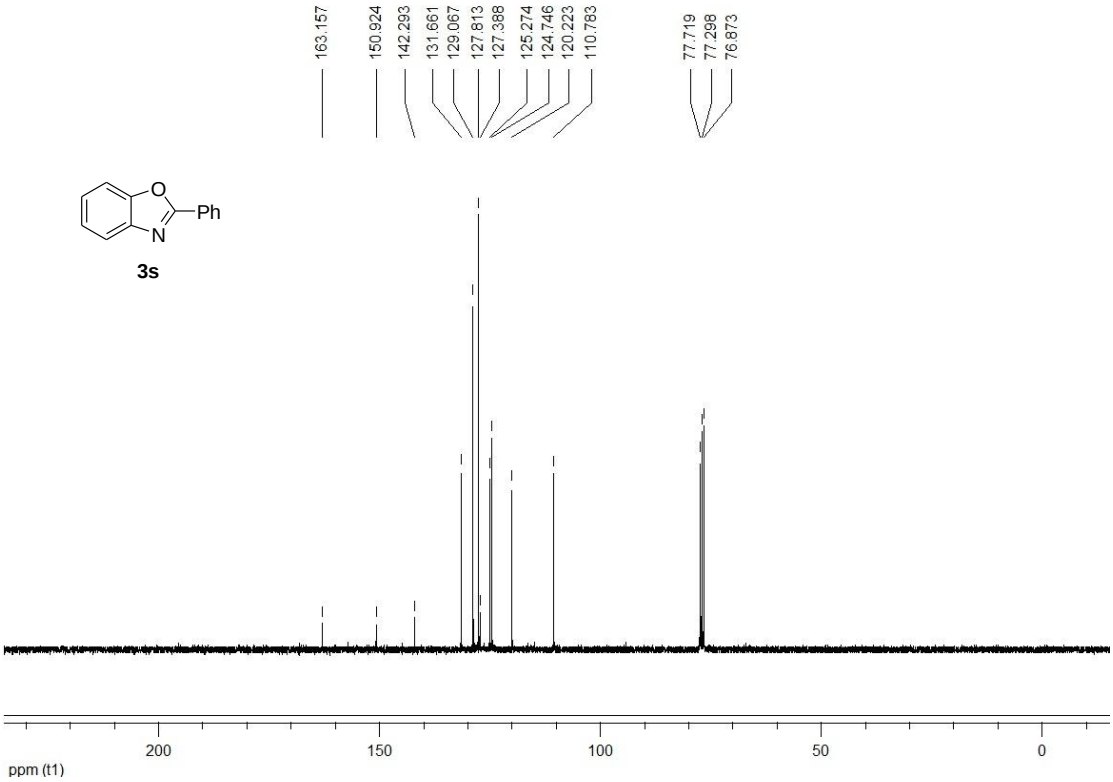
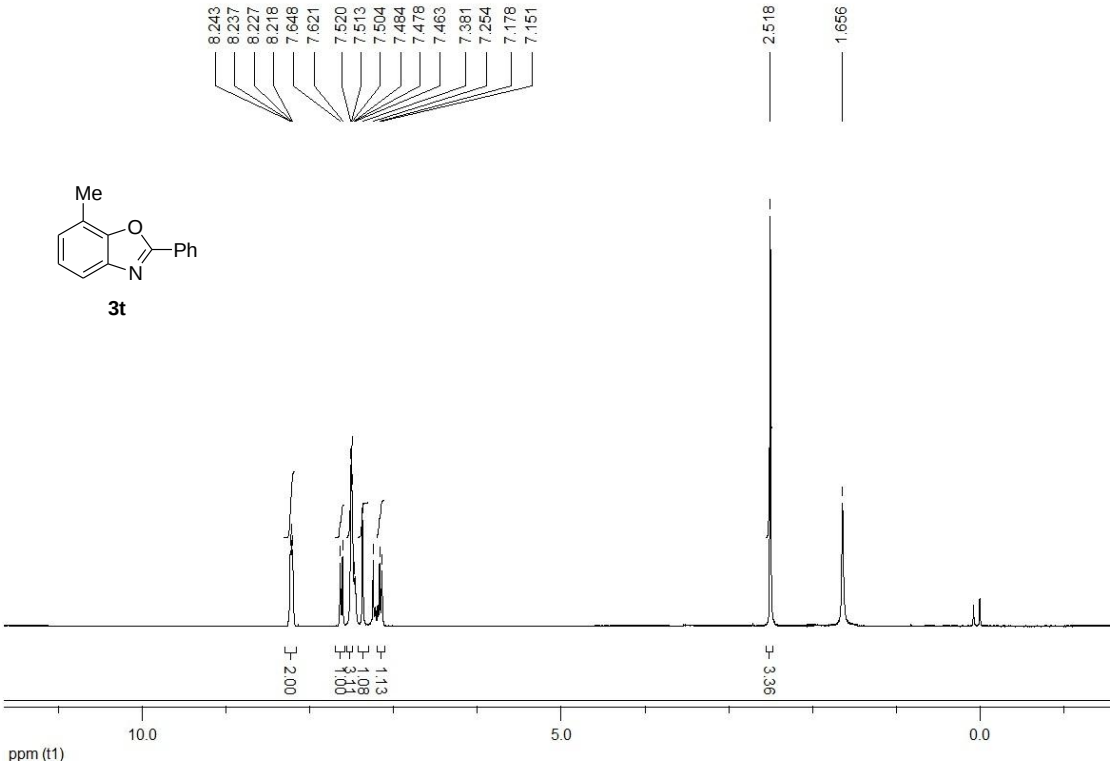
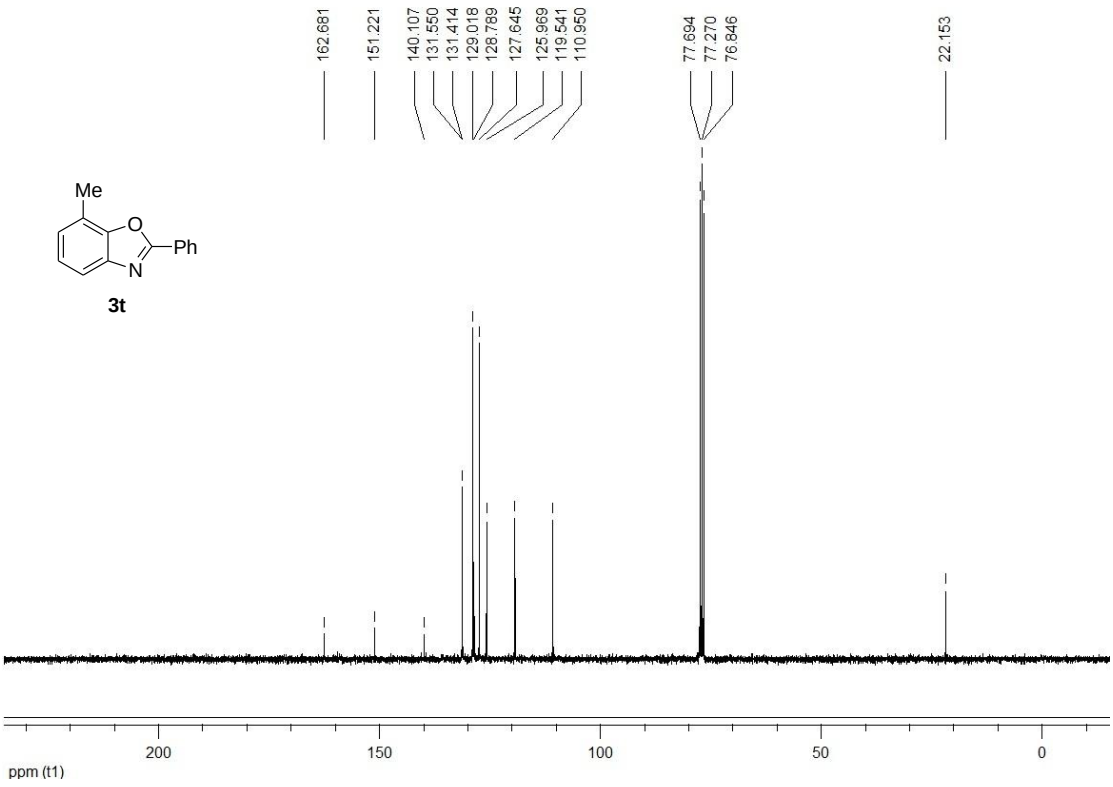
7.255
1.781
0.023
O
Ph
N
3s
2.00
1.05
1.40
2.26
10.0
5.0
0.0
ppm (t1)


163.157
150.924
142.293
131.661
129.067
127.813
127.388
125.274
124.746
120.223
110.783
77.719
77.298
76.873
O
Ph
N
3s
200
150
100
50
0
ppm (t1)


8.243
8.237
8.227
8.218
7.648
7.621
7.520
7.513
7.504
7.484
7.478
7.463
7.381
7.254
7.178
7.151
2.518
1.656
Me
O
Ph
N
3t
2.00
3.00
1.80
1.13
3.36
10.0
5.0
0.0
ppm (t1)


162.681
151.221
140.107
131.550
131.414
129.018
128.789
127.645
125.969
119.541
110.950
77.694
77.270
76.846
22.153
Me
O
Ph
N
3t
200
150
100
50
0
ppm (t1)