RSC Advances

(Electronic Supplementary Information)

Functionalization of Fe_3O_4@SiO_2 nanoparticles with Cu(I)-thiosemicarbazone complex as a robust and efficient heterogeneous nanocatalyst for N-arylation of N-heterocycles with aryl halides

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1-phenylpyrimidine-2,4(1H,3H)-dione (**3a**)

Column chromatography on silica gel (EtOAc:*n*-hexane, 2:1) afforded the product as a white solid; yield: 67%; mp 241-243 °C (Lit.[1] 242-244 °C); ^{1}H NMR (250 MHz, DMSO-d_6): δ_{ppm} = 5.37 (d, *J* = 7.7 Hz, 1H, C(5)-H of uracil), 7.28-7.47 (complex, 4H, aryl, C(6)-H of uracil), 7.76 (d, *J* = 7.2 Hz, 1H, C(6)-H, uracil), 11.16 (s, 1H, N(3)-H of uracil); ^{13}C NMR (62.5 MHz, DMSO-d_6): δ_{ppm} = 102.2, 121.9, 125.4, 128.4, 132.2, 143.6, 153.8, 161.4; IR (KBr): 3170, 3045, 1732, 1695, 1601, 1448 cm^{-1}.

1-(4-methoxyphenyl)pyrimidine-2,4(1H,3H)-dione (**3b**)

Column chromatography on silica gel (EtOAc:*n*-hexane, 2:1) afforded the product as a white solid; yield: 65%; mp 227-229 °C (Lit.[1] 228-229 °C); ^{1}H NMR (250 MHz, DMSO-d_6): δ_{ppm} = 3.70 (s, 3H, OCH_3), 5.54 (d, *J* = 7.8 Hz, 1H, C(5)-H of uracil), 6.84 (d, *J* = 8.6 Hz, 2H, aryl), 7.14 (d, *J* = 8.6 Hz, 2H, aryl), 7.69 (d, *J* = 7.8 Hz, 1H, C(6)-H of uracil), 11.17 (s, 1H, N(3)-H of uracil); ^{13}C NMR (62.5 MHz, DMSO-d_6): δ_{ppm} = 57.7, 102.8, 115.5, 123.4, 125.4, 143.8, 152.4, 157.4, 162.0; IR (KBr): 3151, 3010, 2953, 1729, 1685, 1510, 1449, 1242 cm^{-1}.

5-methyl-1-phenylpyrimidine-2,4(1H,3H)-dione (**3c**)

Column chromatography on silica gel (EtOAc:*n*-hexane, 2:1) afforded the product as a white solid; yield: 59%; mp 199-201 °C (Lit.[1] 198-200 °C); ^{1}H NMR (250 MHz, DMSO-d_6): δ_{ppm} = 1.96 (s, 3H, $=CCH_3$), 7.54-7.61 (m, 2H, aryl), 7.68-7.74 (m, 1H, aryl), 8.00-8.03 (m, 2H, aryl), 8.10 (s, 1H, C(6)-H of thymine), 11.17 (s, 1H, N(3)-H of thymine); ^{13}C NMR (62.5 MHz, DMSO-d_6): δ_{ppm} = 14.6, 111.6, 122.7, 125.0, 129.5, 132.7, 134.9, 153.2, 164.8; IR (KBr): 3185, 3072, 2958, 1726, 1701, 1598, 1469 cm^{-1}.

5-methyl-1-p-tolylpyrimidine-2,4(1H,3H)-dione (**3d**)

Column chromatography on silica gel (EtOAc:*n*-hexane, 2:1) afforded the product as a white solid; yield: 54%; mp 217-219 °C (Lit.[1] 216-217 °C); ^{1}H NMR (250 MHz, DMSO-d_6): δ_{ppm} = 2.05 (s, 3H, $=CCH_3$), 2.72 (s, 3H, OCH_3), 6.89 (d, *J* = 8.8 Hz, 2H, aryl), 7.60 (d, *J* = 9.0 Hz, 2H, aryl), 8.48 (s, 1H, C(6)-H of thymine), 11.86 (s, 1H, N(3)-H of thymine); ^{13}C NMR (62.5 MHz, DMSO-d_6): δ_{ppm}

= 16.4, 25.5, 112.8, 122.9, 128.5, 129.5, 135.9, 136.9, 154.6, 164.3; IR (KBr): 3178, 3065, 2969, 1723, 1697, 1602, 1479 cm^{-1}.

1,3-dimethyl-7-phenyl-1H-purine-2,6(3H,7H)-dione (**3e**)

Column chromatography on silica gel (EtOAc:*n*-hexane, 2:1) afforded the product as a white solid; yield: 77%; mp 196-198 °C (Lit.[2] 195.5 °C); ^{1}H NMR (250 MHz, $CDCl_3$): δ_{ppm} = 3.33 (s, 3H, N(1)-CH_3), 3.59 (s, 3H, N(1)-CH_3), 7.43 (br s, 5H, aryl), 7.67 (s, 1H, C(8)-H of theophylline); ^{13}C NMR (62.5 MHz, $CDCl_3$): δ_{ppm} = 27.7, 29.7, 105.95 126.1, 128.1, 129.1, 136.0, 141.4, 150.3, 152.4, 156.7; IR (KBr): 3047, 2978, 1715, 1701, 1654, 1474 cm^{-1}.

7-(4-methoxyphenyl)-1,3-dimethyl-1H-purine-2,6(3H,7H)-dione (**3f**)

Column chromatography on silica gel (EtOAc:*n*-hexane, 2:1) afforded the product as a white foam (Lit.[3]); yield: 74%; ^{1}H NMR (250 MHz, $CDCl_3$): δ_{ppm} = 3.32 (s, 3H, N(1)-CH_3), 3.58 (s, 3H, N(1)-CH_3), 3.79 (s, 3H, OCH_3), 6.92 (d, *J* = 9.0 Hz, 2H, aryl), 7.33 (d, *J* = 9.0 Hz, 2H, aryl), 7.62 (s, 1H, C(8)-H of theophylline); ^{13}C NMR (62.5 MHz, $CDCl_3$): δ_{ppm} = 28.1, 29.9, 55.3, 107.2, 114.9, 125.7, 127.9, 142.7, 150.5, 152.7, 155.6, 161.7; IR (KBr): 3061, 2947, 1719, 1702, 1659, 1468, 1236 cm^{-1}.

3,7-dimethyl-1-phenyl-1H-purine-2,6(3H,7H)-dione (**3g**)

Column chromatography on silica gel (EtOAc:*n*-hexane, 2:1) afforded the product as a white solid; yield: 78%; mp 218-220 °C (Lit.[4] 218.5 °C); ^{1}H NMR (250 MHz, $CDCl_3$): δ_{ppm} = 3.61 (s, 3H, N(3)-CH_3), 3.94 (s, 3H, N(7)-CH_3), 7.22-7.26 (m, 2H, aryl), 7.44-7.50 (m, 3H, aryl), 7.55 (s, 1H, C(8)-H of theobromine); ^{13}C NMR (62.5 MHz, $CDCl_3$): δ_{ppm} = 27.2, 31.2, 106.4, 123.2, 125.8, 129.2, 130.4, 146.5, 148.1, 150.7, 165.8; IR (KBr): 3115, 2949, 1815, 1701, 1653, 1605, 1448 cm^{-1}.

1-(4-methoxyphenyl)-3,7-dimethyl-1H-purine-2,6(3H,7H)-dione (**3h**)

Column chromatography on silica gel (EtOAc:*n*-hexane, 2:1) afforded the product as a white solid; yield: 76%; mp 181-183 °C (Lit.[4] 182.5 °C); ^{1}H NMR (250 MHz, $CDCl_3$): δ_{ppm} = 3.62 (s, 3H, N(3)-CH_3), 3.82 (s, 3H, N(7)-CH_3), 3.95 (s, 3H, OCH_3), 6.97-7.02 (m, 2H, aryl), 7.12-7.16 (m, 2H, aryl), 7.53 (s, 1H, C(8)-H of theobromine); ^{13}C NMR (62.5 MHz, $CDCl_3$): δ_{ppm} = 28.3, 30.2, 53.9, 105.4,

115.1, 122.9, 124.2, 144.9, 149.2, 151.3, 157.5, 164.7; IR (KBr): 3130, 2979, 2839, 1869, 1708, 1663, 1602, 1469, 1243 cm^{-1}.

1-(4-methoxyphenyl)-1H-indole (**3i**)

Column chromatography on silica gel (EtOAc:*n*-hexane, 1:1) afforded the product as a white solid; yield: 88%; mp 56-58 °C (Lit.[5] 57-59 °C); ^{1}H NMR (250 MHz, $CDCl_3$): δ_{ppm} = 3.71 (s, 3H, OCH_3), 6.58 (br s, 1H, C(3)-H of indole), 6.73 (d, *J* = 7.5 Hz, 2H, aryl), 6.93 (d, *J* = 5.0 Hz, 2H, aryl), 7.09m (d, *J* = 5.0 Hz, 2H, aryl), 7.19 (br s, 2H, aryl), 7.83 (br s, 1H, C(2)-H of indole); ^{13}C NMR (62.5 MHz, $CDCl_3$): δ_{ppm} = 55.5, 102.4, 110.5, 114.5, 120.2, 121.6, 122.5, 126.3, 128.6, 129.3, 133.1, 136.8, 158.8; IR (KBr): 3050, 2975, 1669, 1597, 1463, 1238 cm^{-1}.

1-(2-methoxyphenyl)-1H-indole (**3j**)

Column chromatography on silica gel (EtOAc:*n*-hexane, 1:1) afforded the product as a colorless oil (Lit.[5]); yield: 73%; ^{1}H NMR (250 MHz, $CDCl_3$): δ_{ppm} = 3.72 (s, 3H, OCH_3), 6.67-6.74 (complex, 5H, aryl, C(3)-H of indole), 7.12-7.19 (m, 4H, aryl), 7.60 (br s, 1H, C(2)-H of indole); ^{13}C NMR (62.5 MHz, $CDCl_3$): δ_{ppm} = 56.1, 102.6, 110.1, 114.5, 119.1, 120.4, 121.8, 122.5, 123.2, 125.9, 127.0, 128.6, 130.6, 144.5, 146.2; IR (film): 3079, 2959, 1664, 1590, 1470, 1245 cm^{-1}.

1-phenyl-1H-benzo[d]imidazole (**3k**)

Column chromatography on silica gel (EtOAc:*n*-hexane, 1:1) afforded the product as a white solid; yield: 92%; mp 91-93 °C (Lit.[6] 94-95 °C); ^{1}H NMR (250 MHz, $CDCl_3$): δ_{ppm} = 7.11-7.28 (m, 5H, aryl), 7.48-7.63 (m, 4H, aryl), 8.31 (s, 1H, C(2)-H of benzimidazole); ^{13}C NMR (62.5 MHz, $CDCl_3$): δ_{ppm} = 110.9, 120.5, 122.6, 123.4, 124.5, 128.1, 129.1, 133.7, 137.2, 142.1, 144.9; IR (KBr): 3015, 1645,1600, 1497 cm^{-1}.

1-(naphthalen-1-yl)-1H-imidazole (**3l**)

Column chromatography on silica gel (EtOAc:*n*-hexane, 1:1) afforded the product as a creamy solid; yield: 82%; mp 62-64 °C (Lit.[7] 63-64 °C); ^{1}H NMR (250 MHz, $CDCl_3$): δ_{ppm} = 6.68-6.76 (m, 2H, aryl), 6.85 (s, 1H, C(5)-H of imidazole), 7.09-7.11 (m, 2H, aryl), 7.12 (s, 1H, C(4)-H of imidazole), 7.22-7.25 (m, 2H, aryl), 7.41-7.52 (complex, 2H, aryl, C(2)-H of imidazole); ^{13}C NMR

(62.5 MHz, $CDCl_3$): δ_{ppm} = 119.3, 121.8, 122.5, 123.9, 125.4, 126.8, 127.7, 128.7, 129.2, 130.1, 133.3, 135.0, 139.3; IR (KBr): 3052, 1684, 1580, 1468 cm^{-1}.

1-phenyl-1H-imidazole (**3m**)

Column chromatography on silica gel (EtOAc:*n*-hexane, 2:1) afforded the product as a colorless liquid (Lit.[8]); yield: 93%; ^1H NMR (250 MHz, $CDCl_3$): δ_{ppm} = 6.96 (s, 1H, C(5)-H of imidazole), 7.14 (s, 1H, C(4)-H of imidazole), 7.55-7.74 (complex, 4H, aryl, C(2)-H of imidazole), 8.03-8.06 (m, 2H, aryl); ^{13}C NMR (62.5 MHz, $CDCl_3$): δ_{ppm} = 118.3, 121.2, 127.2, 128.8, 129.6, 135.3, 137.3; IR (film): 3077, 1675, 1617, 1500, 1457 cm^{-1}.

1-(4-methoxyphenyl)-1H-imidazole (**3n**)

Column chromatography on silica gel (EtOAc:*n*-hexane, 2:1) afforded the product as a white solid; yield: 91%; mp 54-56 °C (Lit.[8] 55-57 °C); ^1H NMR (250 MHz, $CDCl_3$): δ_{ppm} = 3.85 (s, 3H, OCH_3), 6.92 (s, 1H, C(5)-H of imidazole), 7.08-7.15 (complex, 3H, aryl, C(4)-H of imidazole), 7.59 (s, 1H, C(2)-H of imidazole), 8.02-8.06 (s, 2H, aryl); ^{13}C NMR (62.5 MHz, $CDCl_3$): δ_{ppm} = 56.9, 115.1, 118.6, 123.4, 129.5, 130.8, 135.8, 159.6; IR (KBr): 3058, 2972, 1679, 1602, 1482, 1229 cm^{-1}.

1,2-diphenyl-1H-imidazole (**3o**)

Column chromatography on silica gel (EtOAc:*n*-hexane, 1:1) afforded the product as a white solid; yield: 79%; mp 86-88 °C (Lit.[7] 88-89 °C); ^1H NMR (250 MHz, $CDCl_3$): δ_{ppm} = 6.91 (d, J = 1.0 Hz, 1H, C(5)-H of imidazole), 7.09 (d, J = 1.0 Hz, 1H, C(4)-H of imidazole), 7.21-7.30 (m, 5H, aryl), 7.42-7.45 (m, 3H, aryl), 7.55-7.58 (m, 5H, aryl); ^{13}C NMR (62.5 MHz, $CDCl_3$): δ_{ppm} = 115.9, 123.5, 125.7, 127.3, 128.1, 128.9, 129.2, 129.8, 130.8, 137.4, 144.8; IR (KBr): 3078, 1693, 1517, 1479 cm^{-1}.

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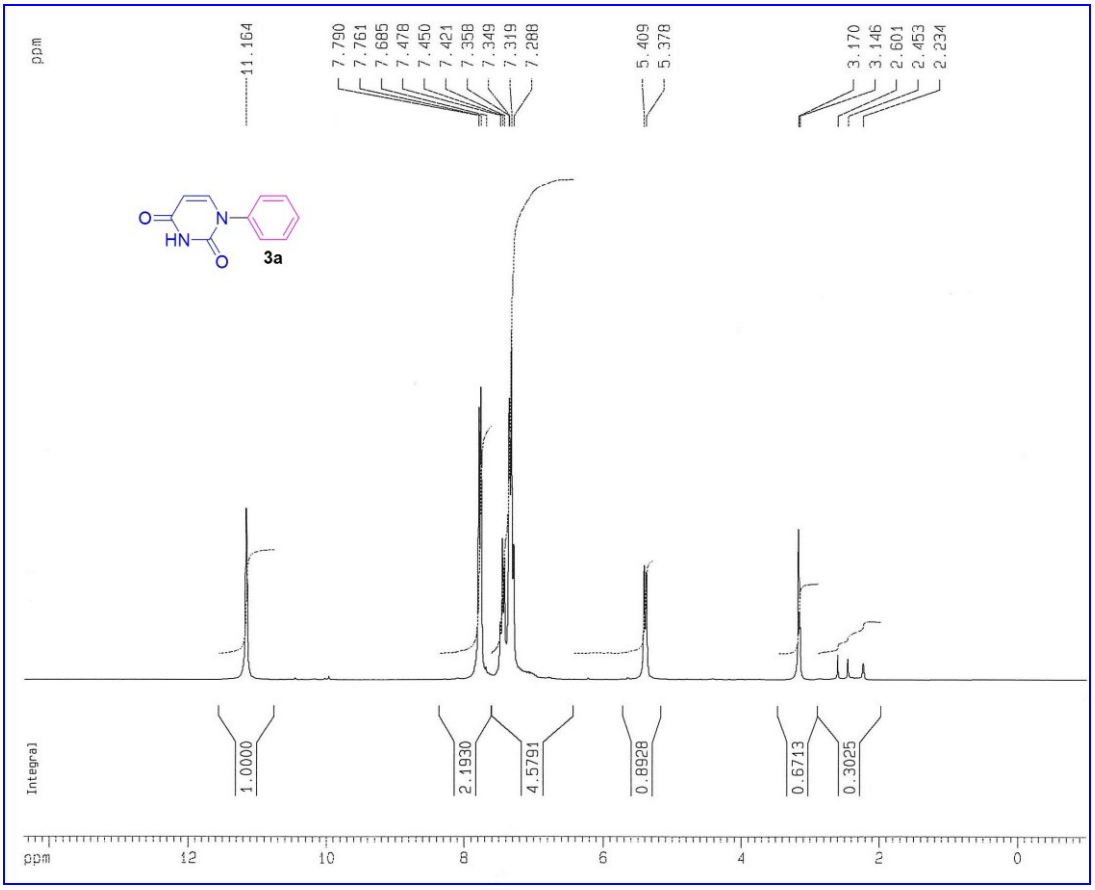
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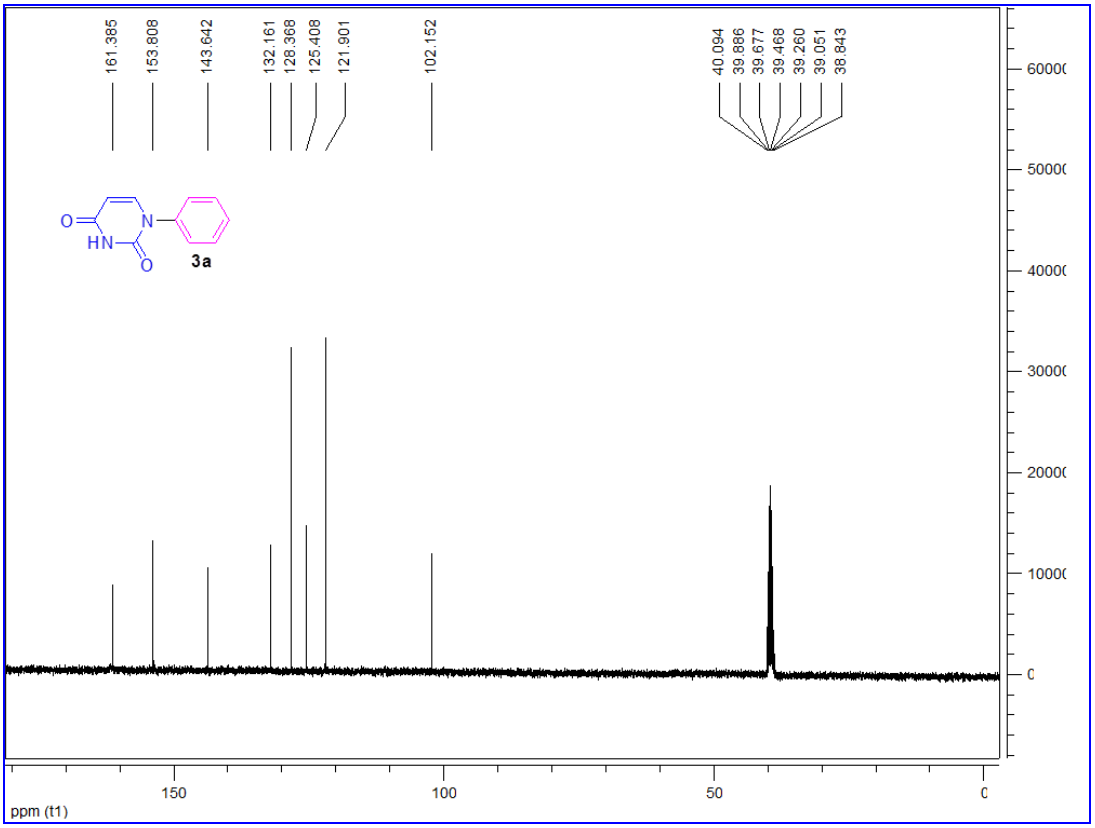
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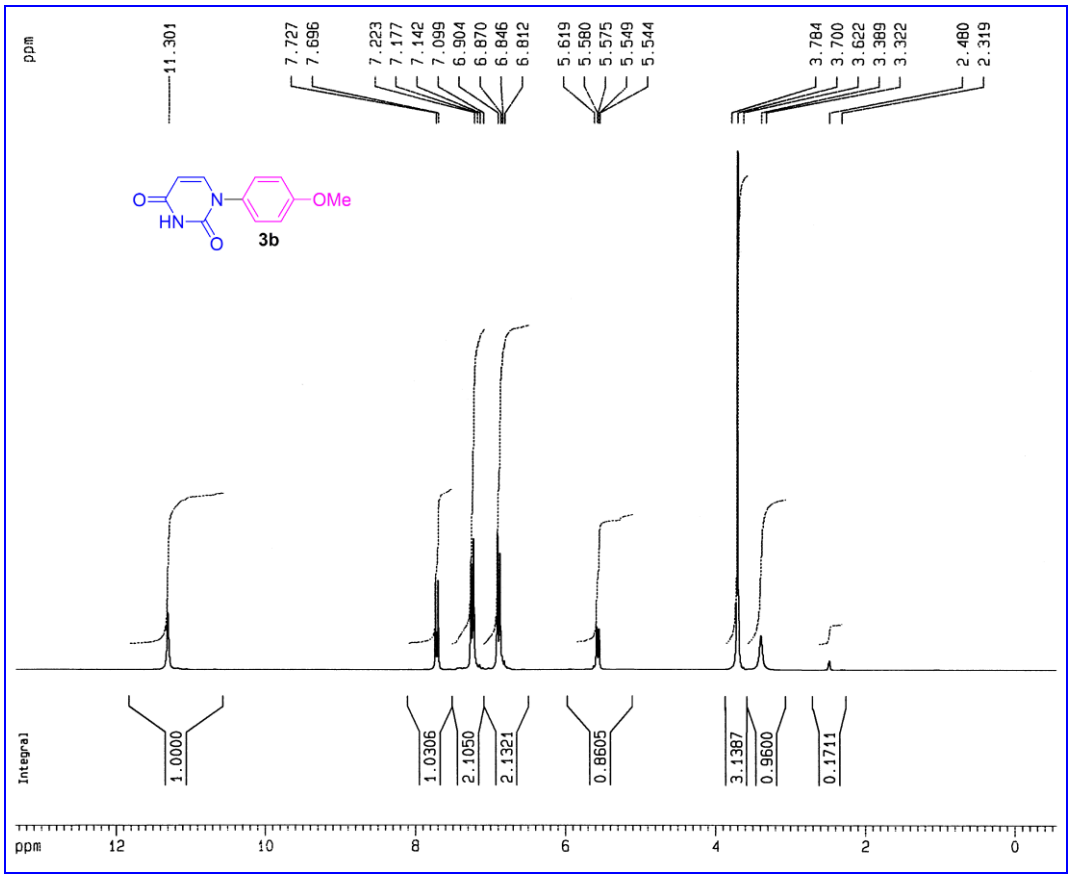
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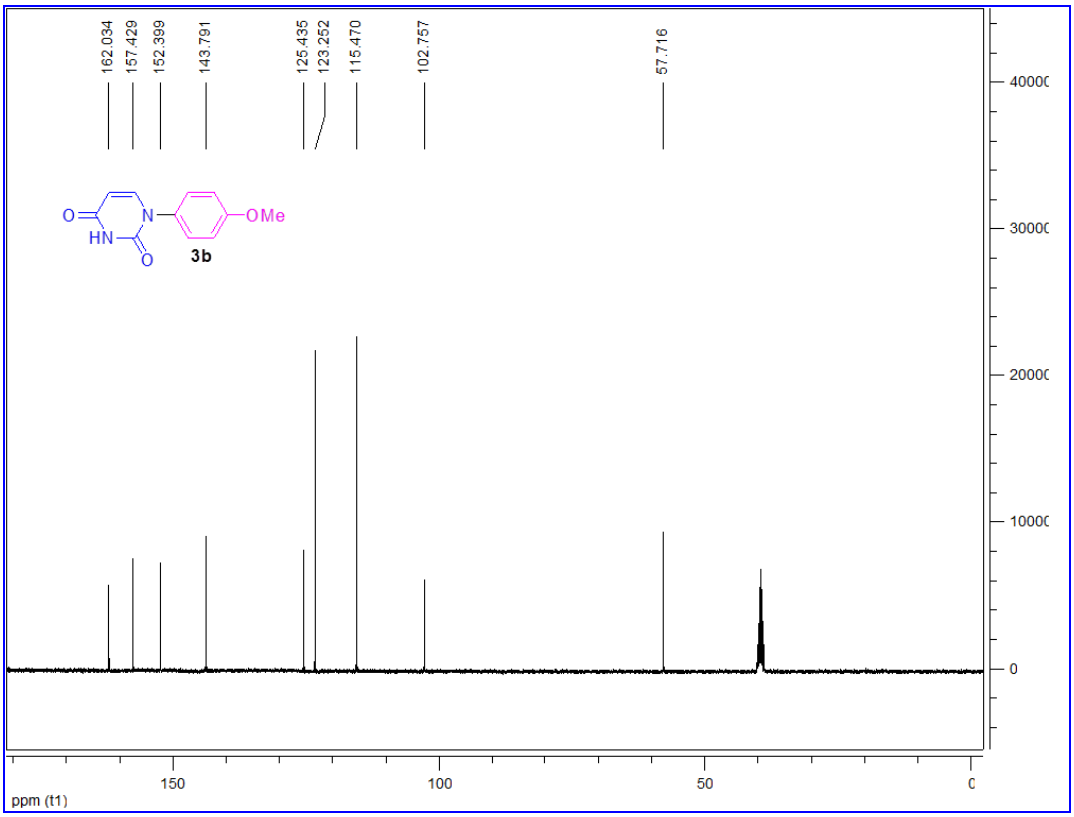
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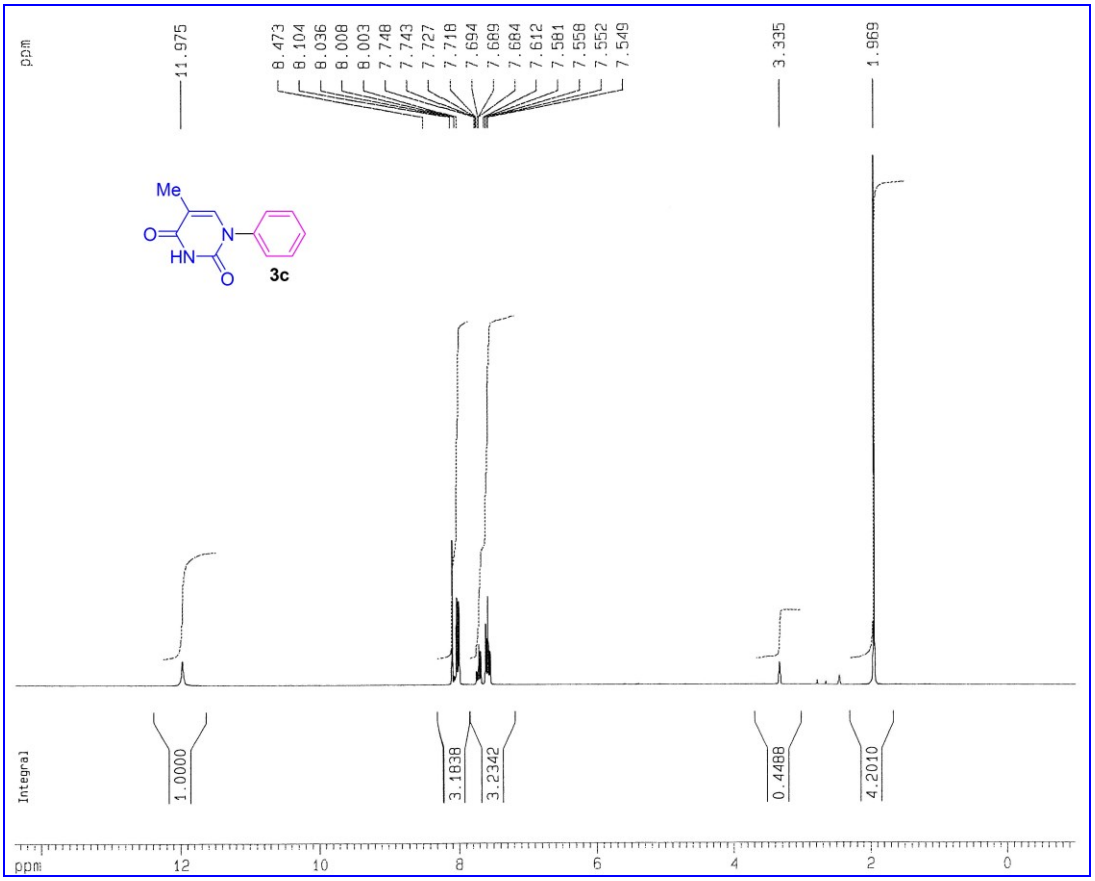
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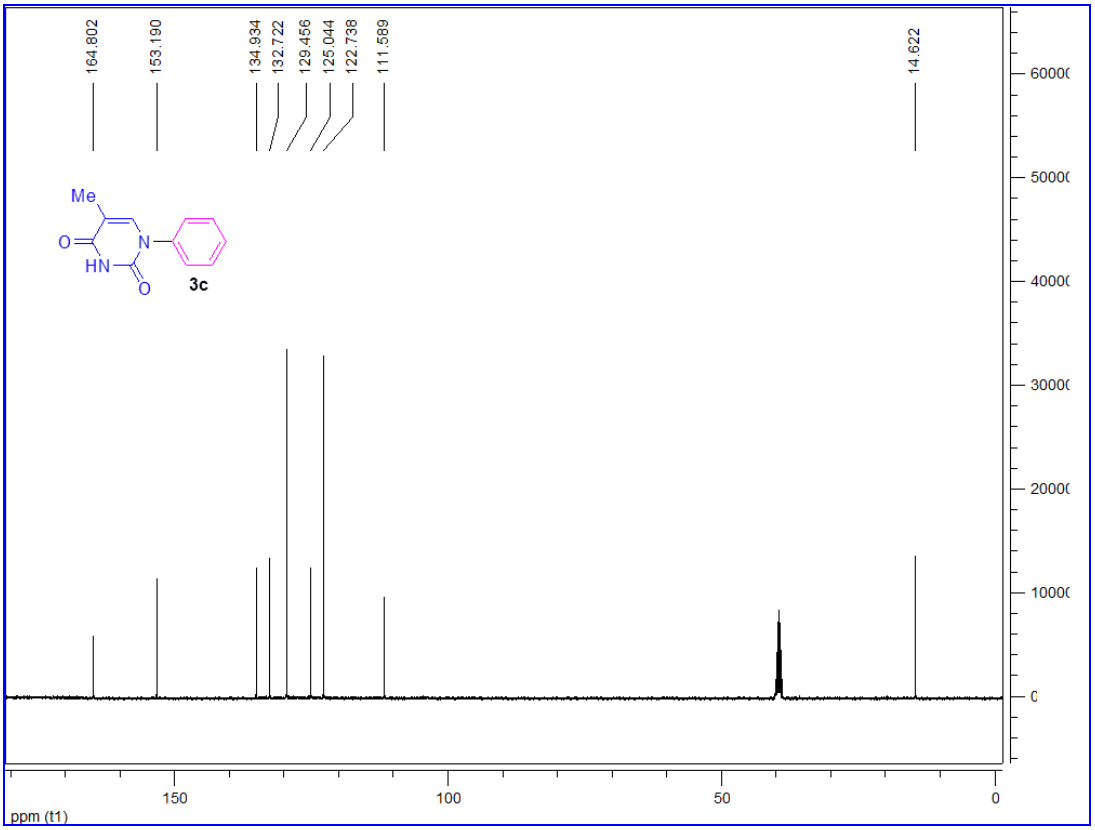
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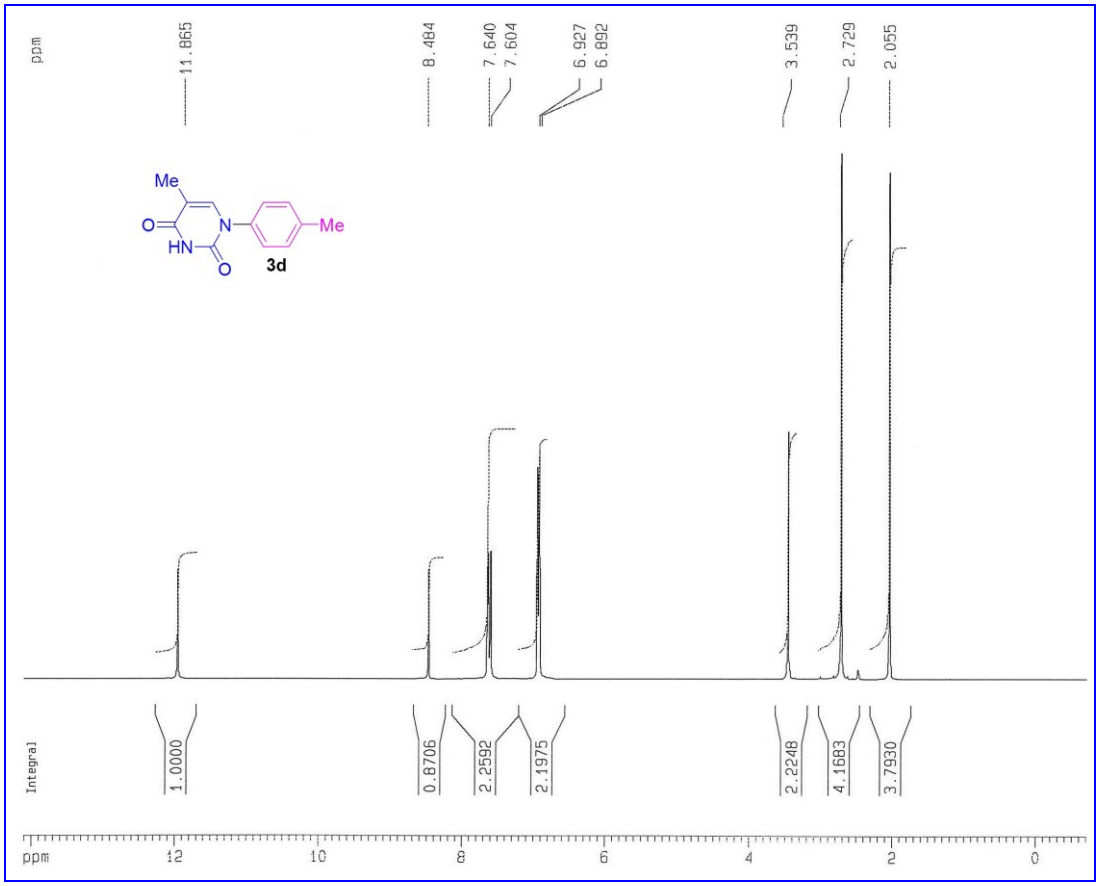
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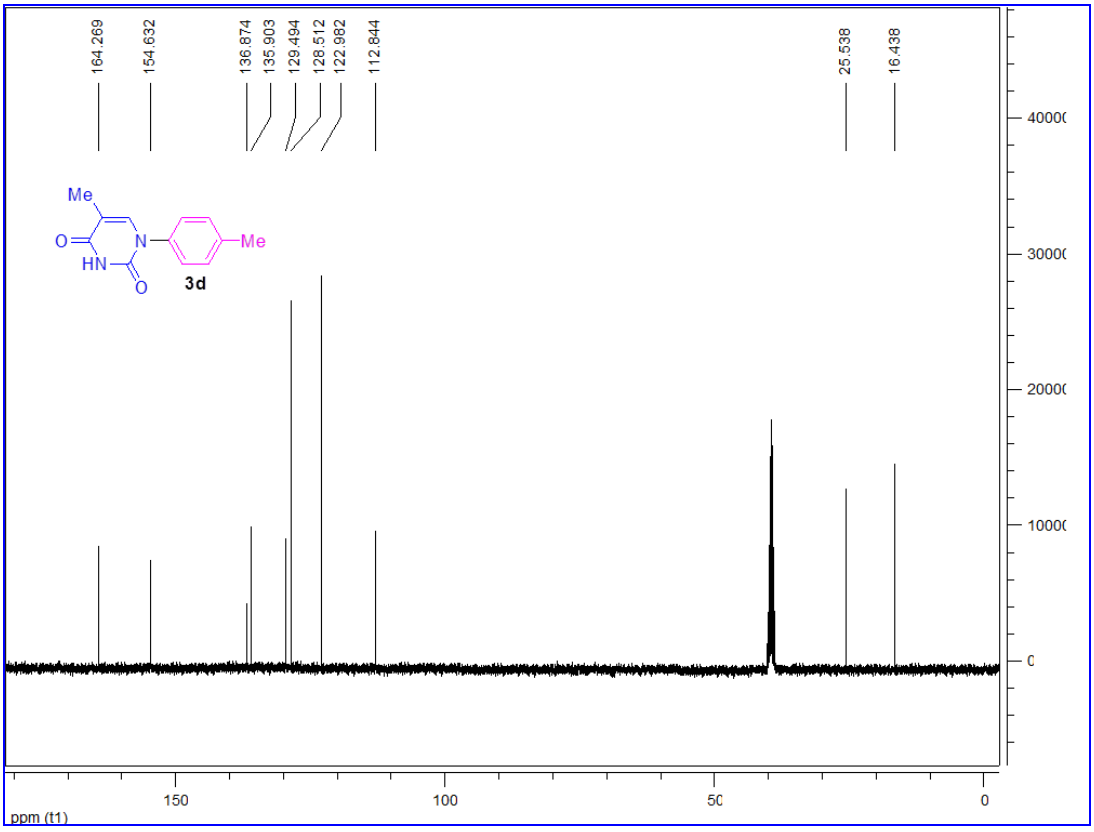
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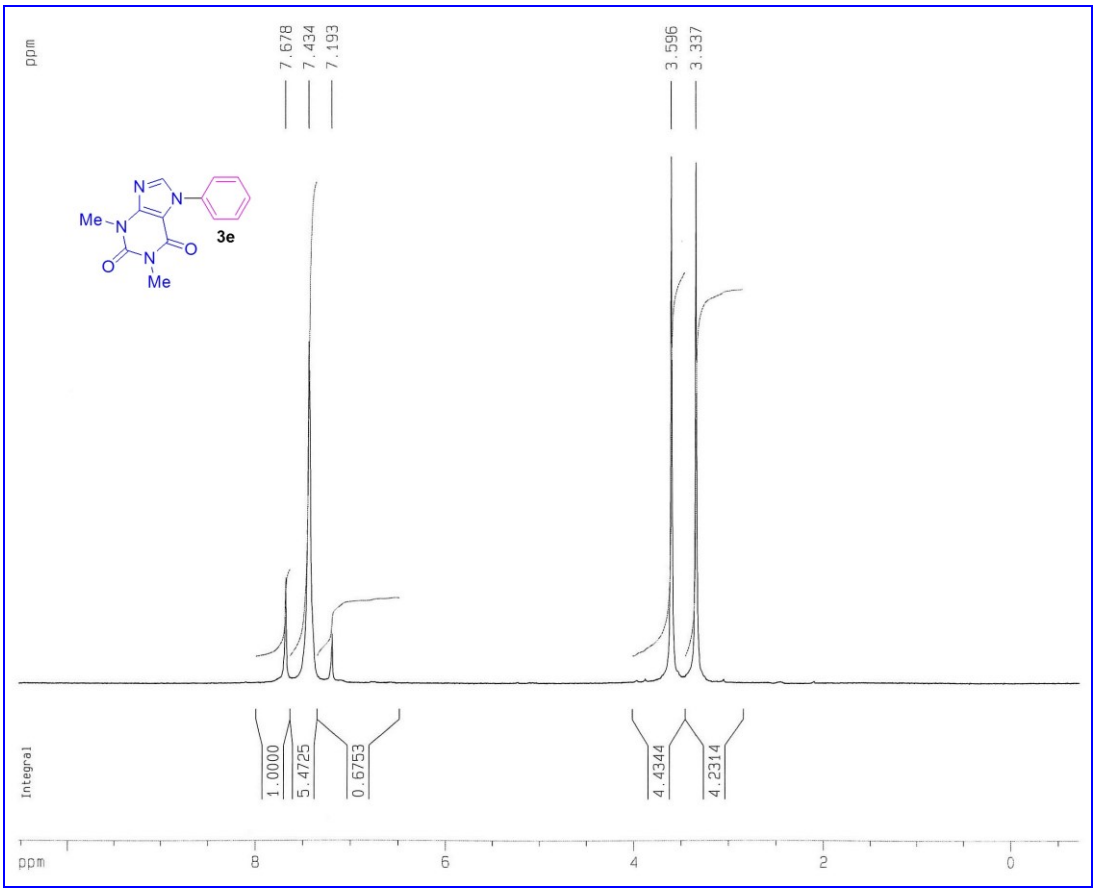
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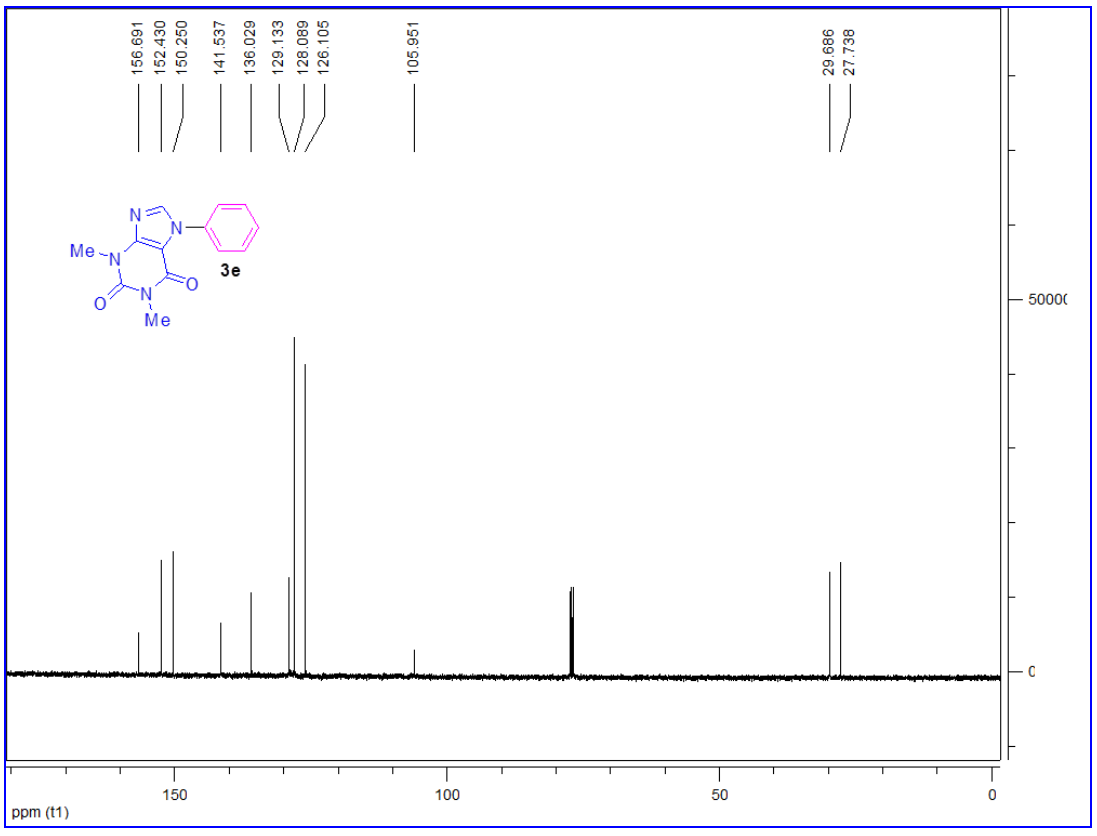
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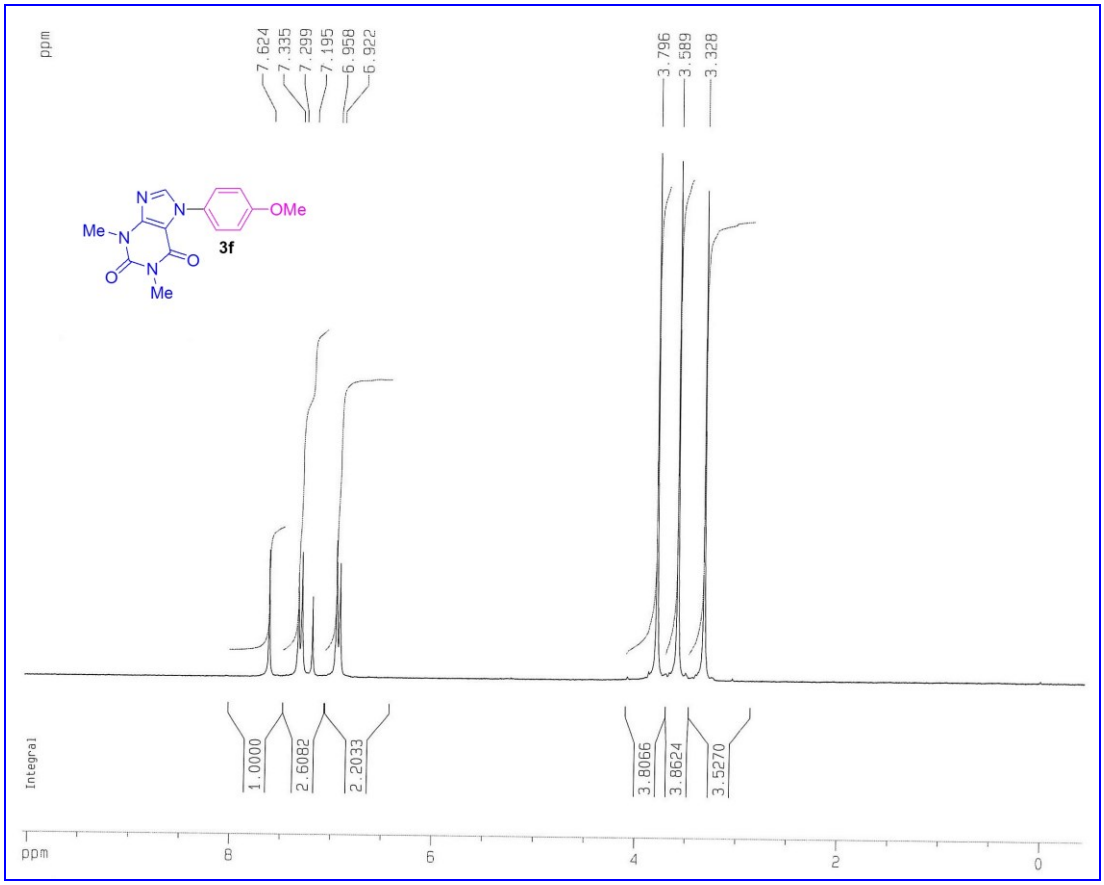
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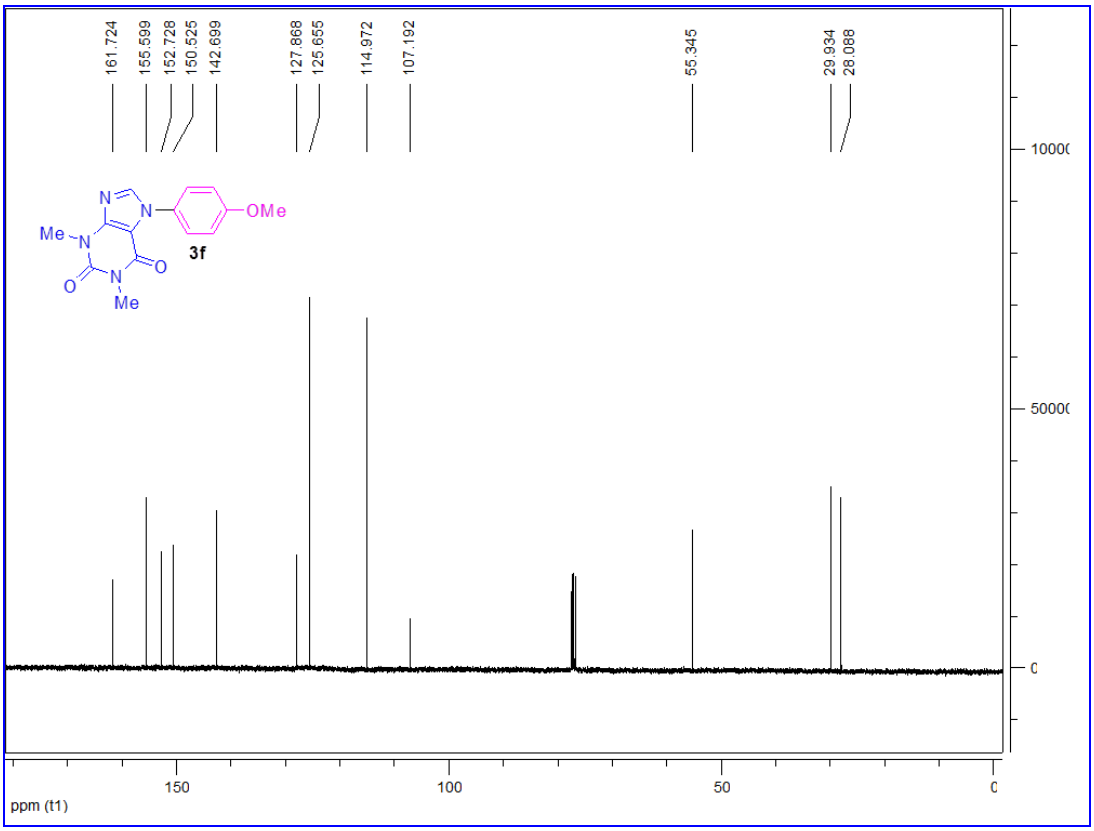
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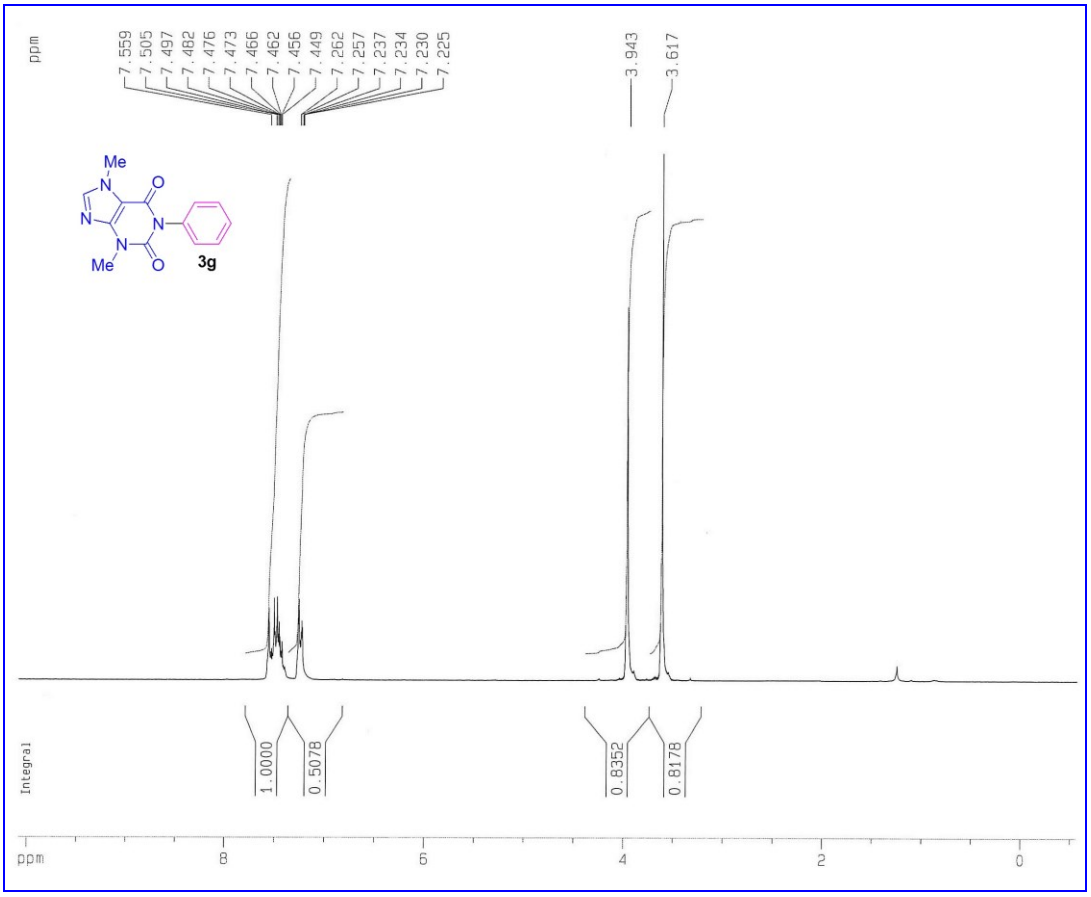
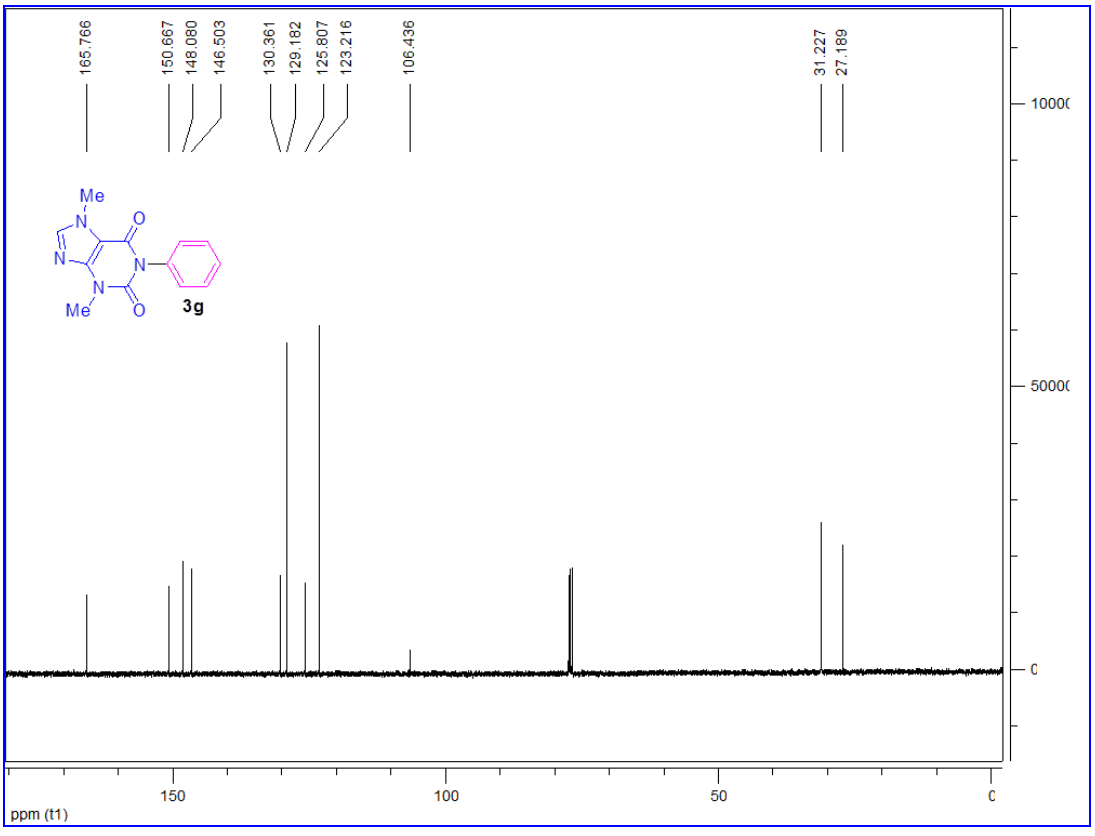
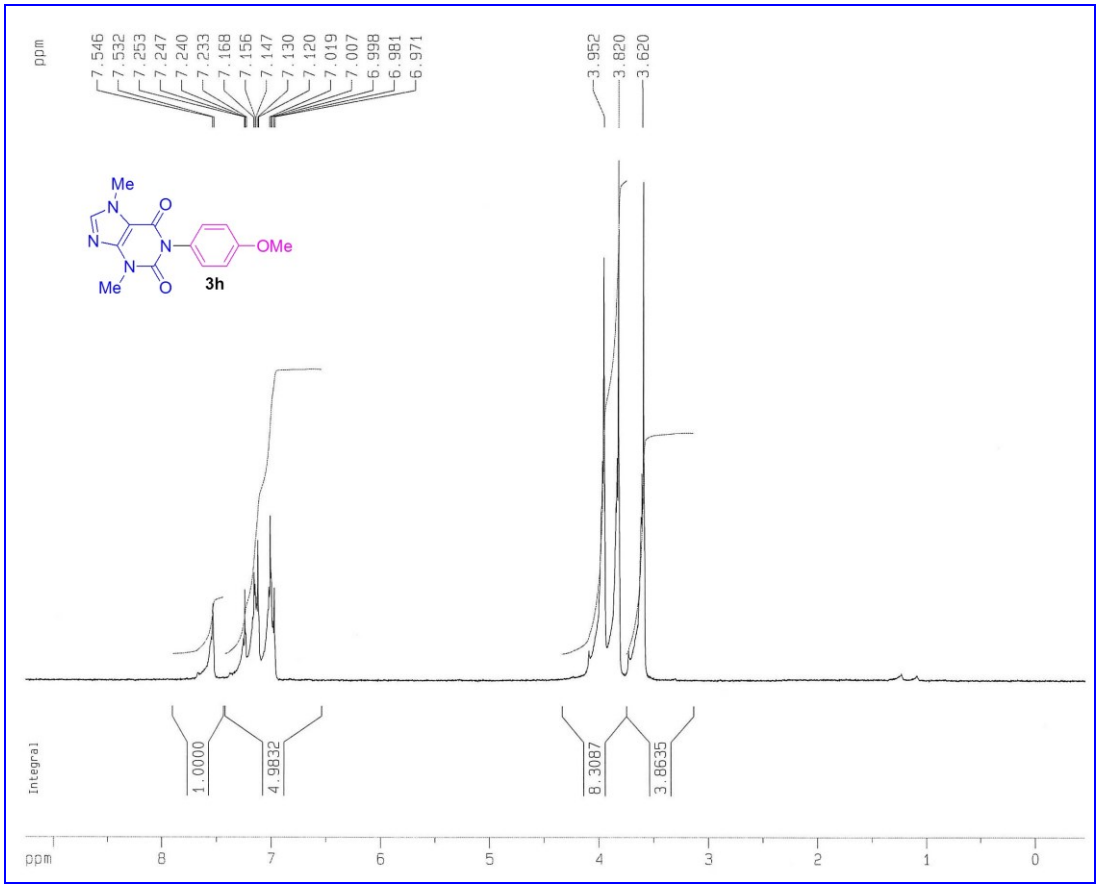
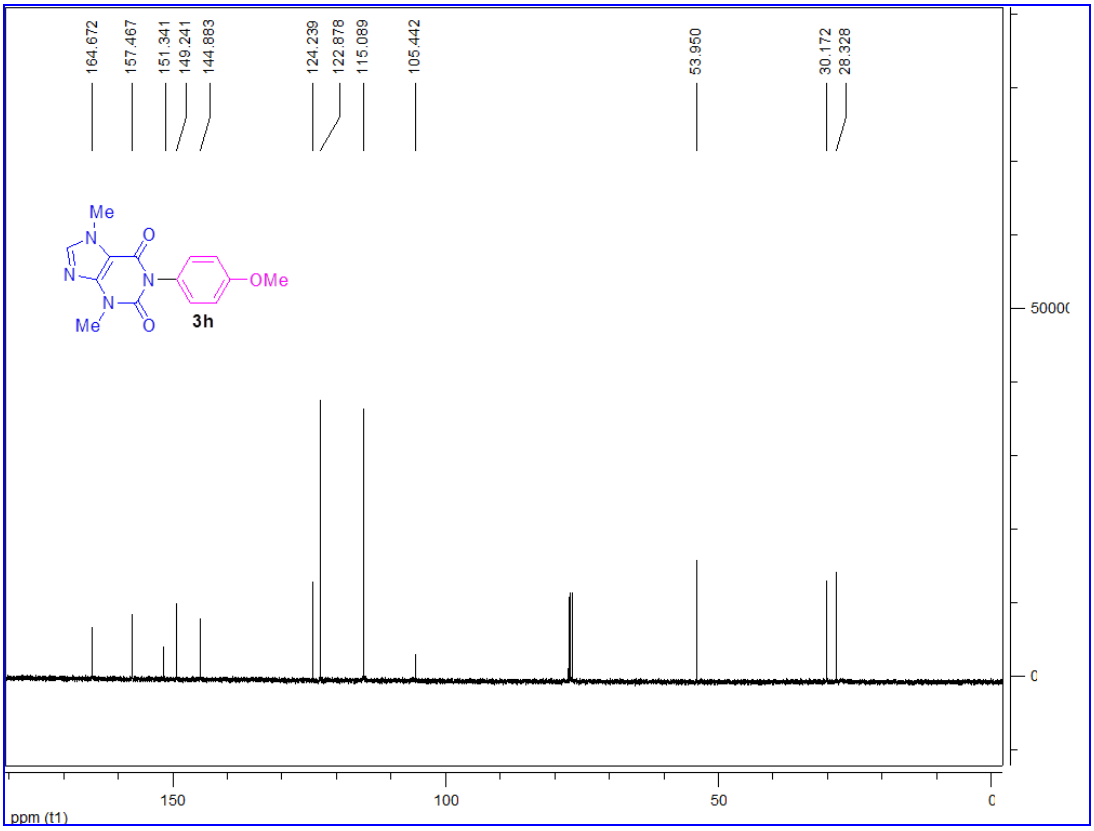
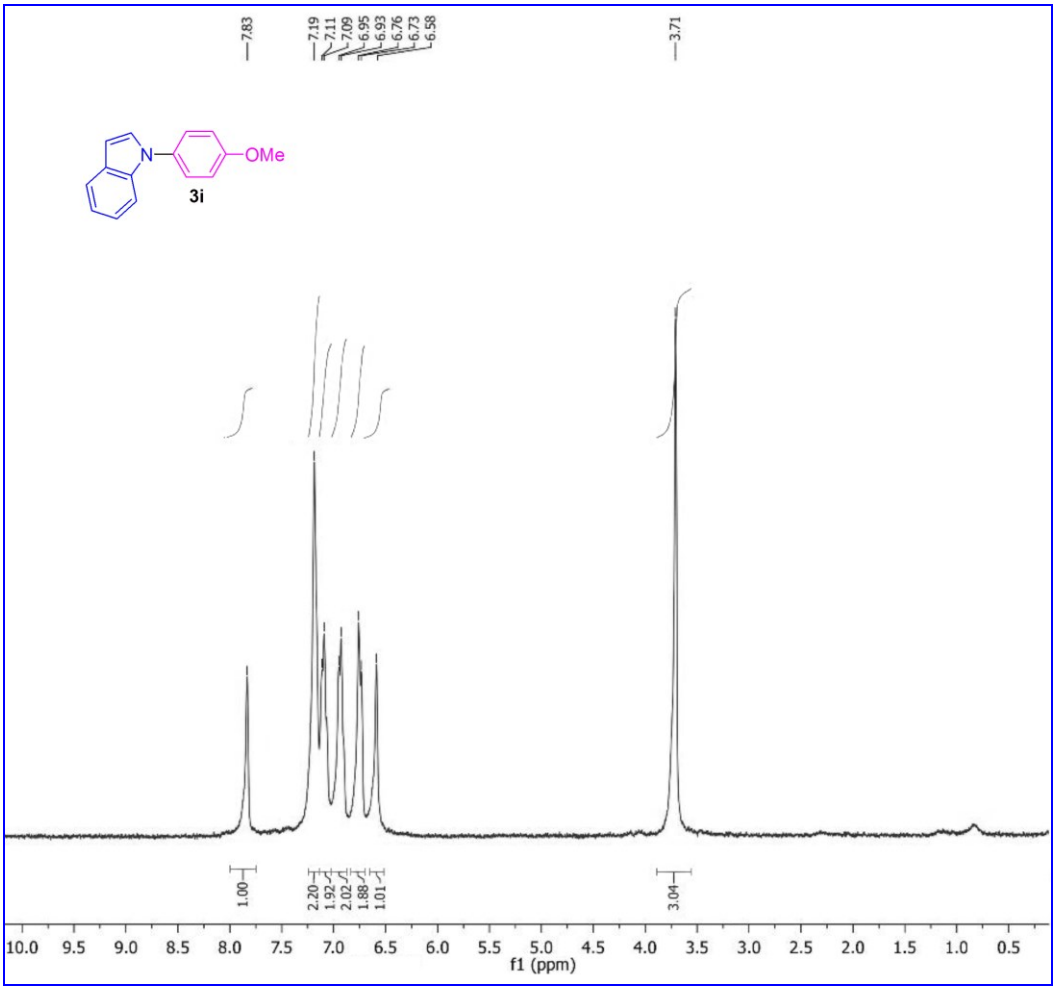
114.972
107.192
55.345
29.934
28.088
Me
N
N
OMe
3f
O
N
O
Me
10000
5000
0
150
100
50
0
ppm (t1)

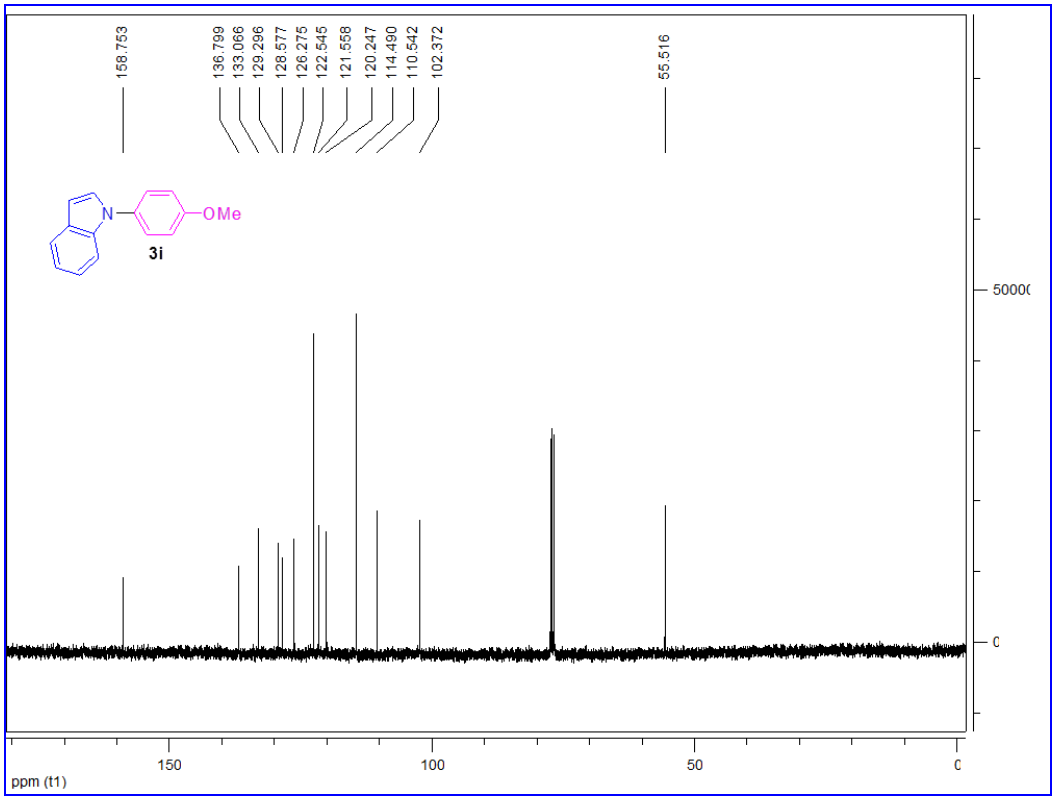

ppm
7.559
7.505
7.497
7.482
7.476
7.473
7.466
7.462
7.456
7.449
7.262
7.257
7.237
7.234
7.230
7.225
3.943
3.617
Me
N
O
N
N
N
Me
O
3g
Integral
1.0000
0.5078
0.8352
0.8178
ppm
8
6
4
2
0

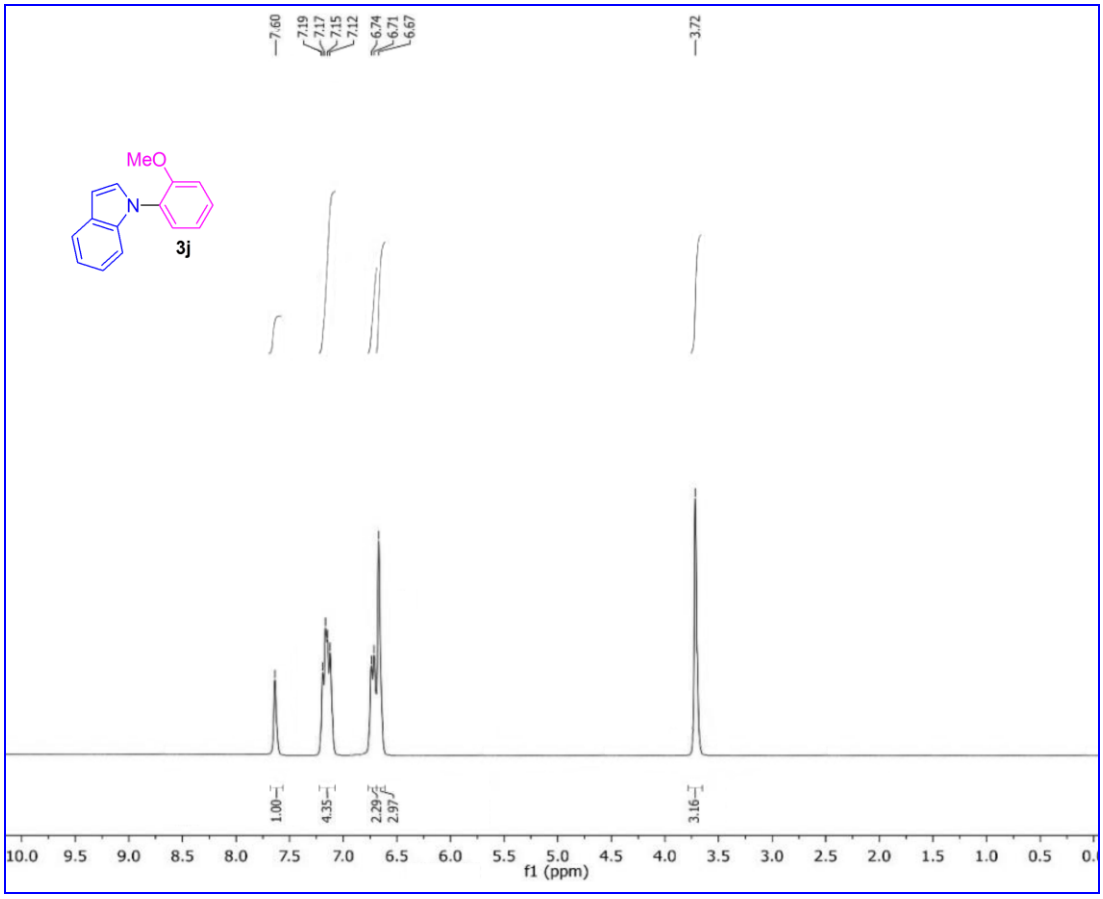

165.766
150.667
148.080
146.503
130.361
129.182
125.807
123.216
106.436
31.227
27.189
Me
N
O
N
N
N
Me
O
3g
10000
50000
0
150
100
50
0
ppm (t1)

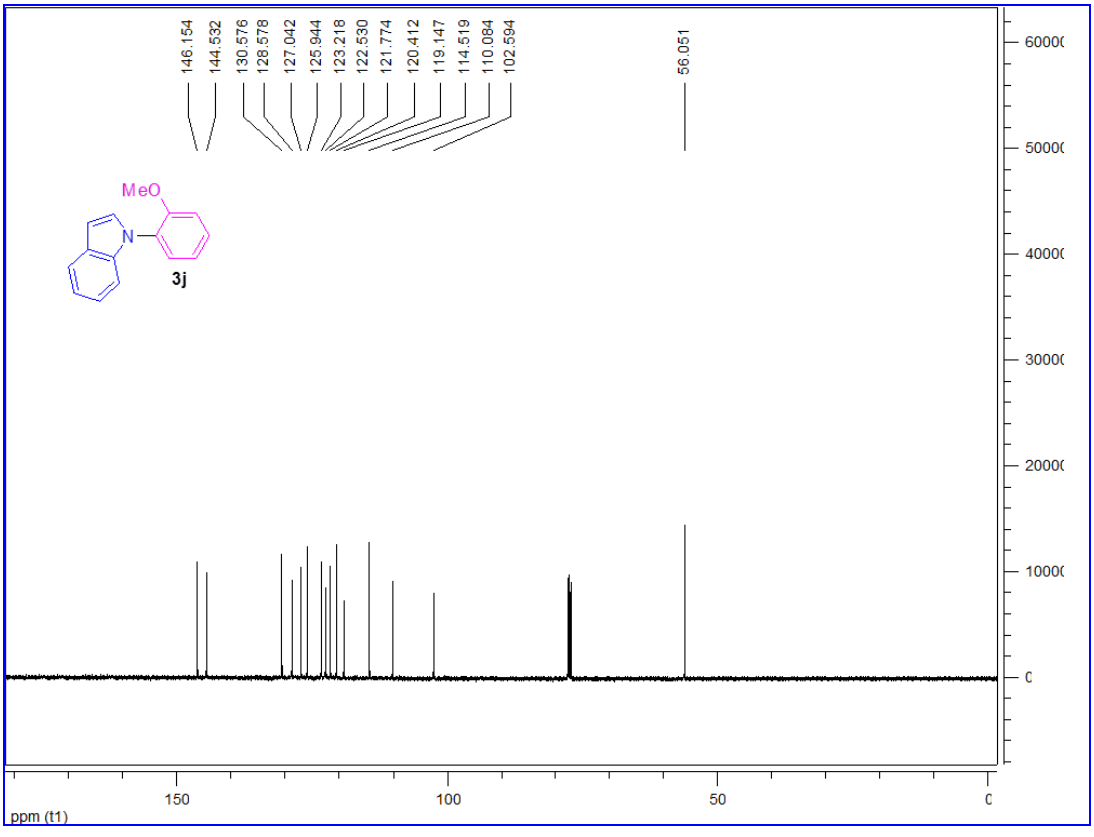

ppm
7.546
7.532
7.253
7.247
7.240
7.233
7.168
7.156
7.147
7.130
7.120
7.019
7.007
6.998
6.981
6.971
3.952
3.820
3.620
Me
N
O
N
OMe
N
Me
O
3h
Integral
1.0000
4.9832
8.3087
3.8635
ppm
8
7
6
5
4
3
2
1
0

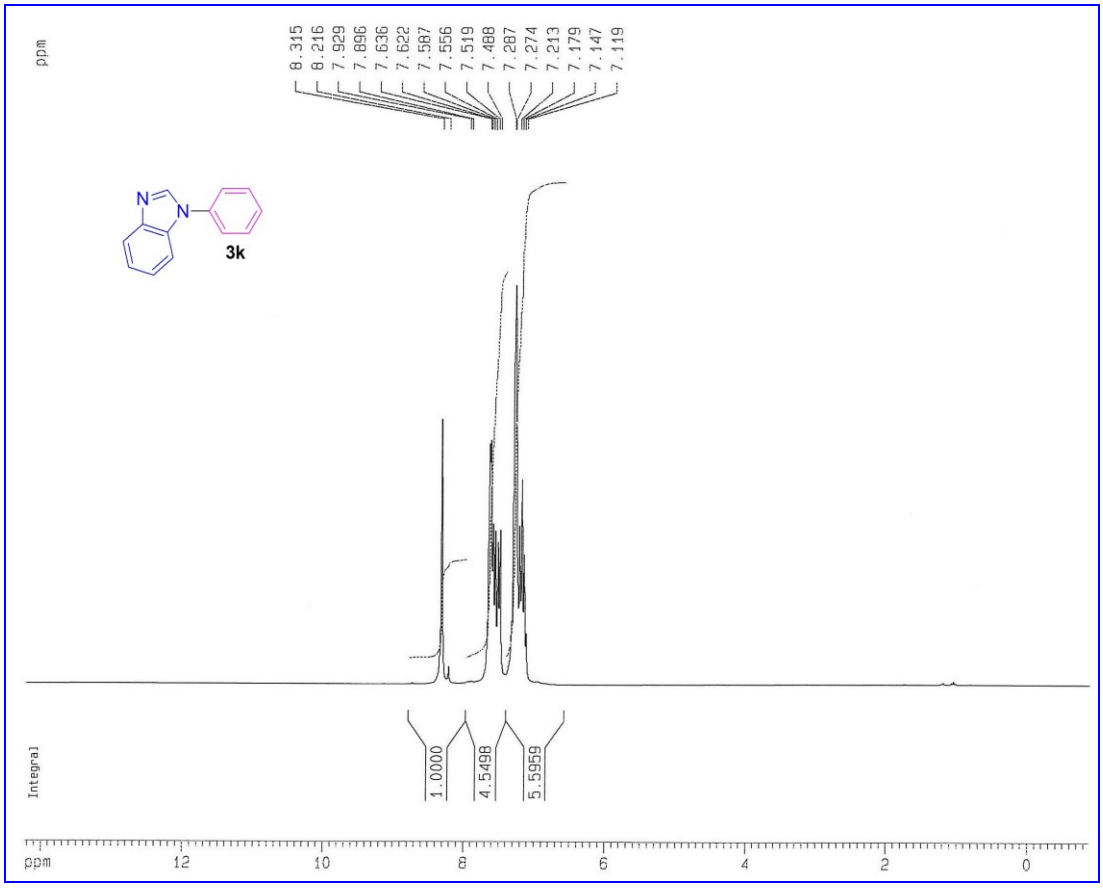

164.672
157.467
151.341
149.241
144.883
124.239
122.878
115.089
105.442
53.950
30.172
28.328
Me
N
O
N
OMe
N
Me
O
3h
50000
0
150
100
50
0
ppm (t1)

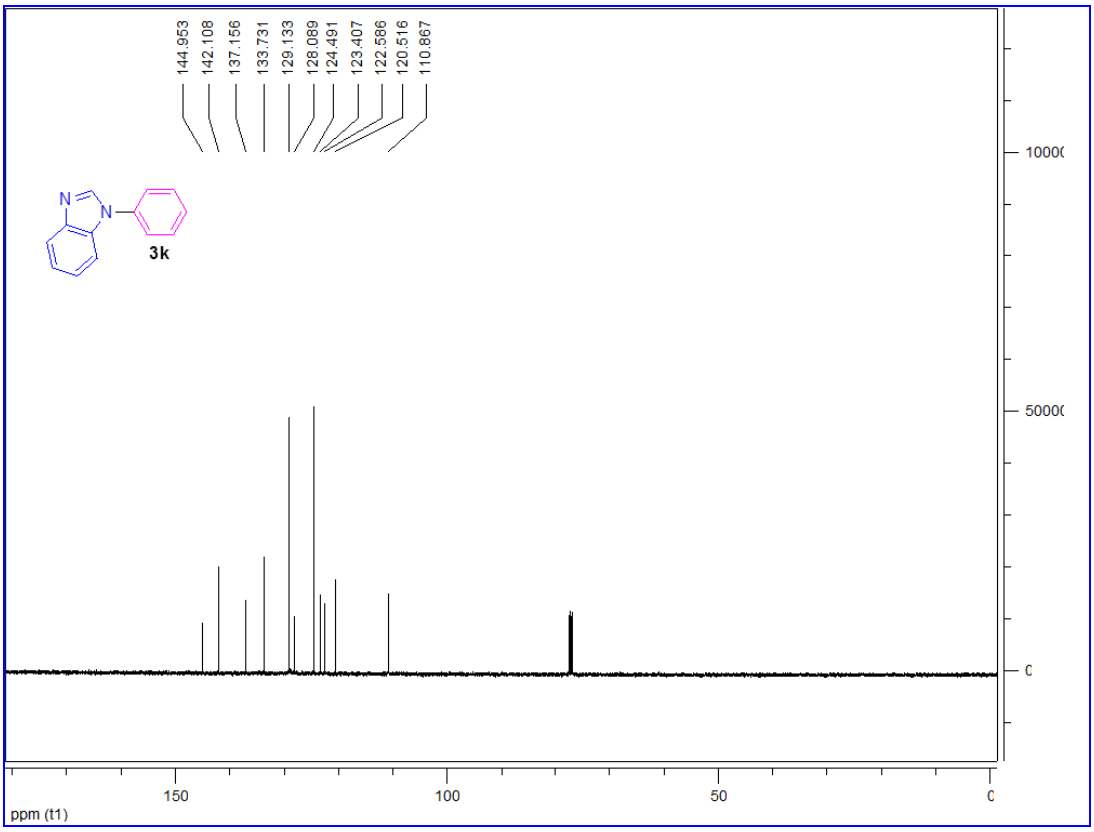

7.83
7.19
7.11
7.09
6.95
6.93
6.76
6.73
6.58
3.71
OMe
3i
1.00
2.20
1.92
2.02
1.88
1.01
3.04
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

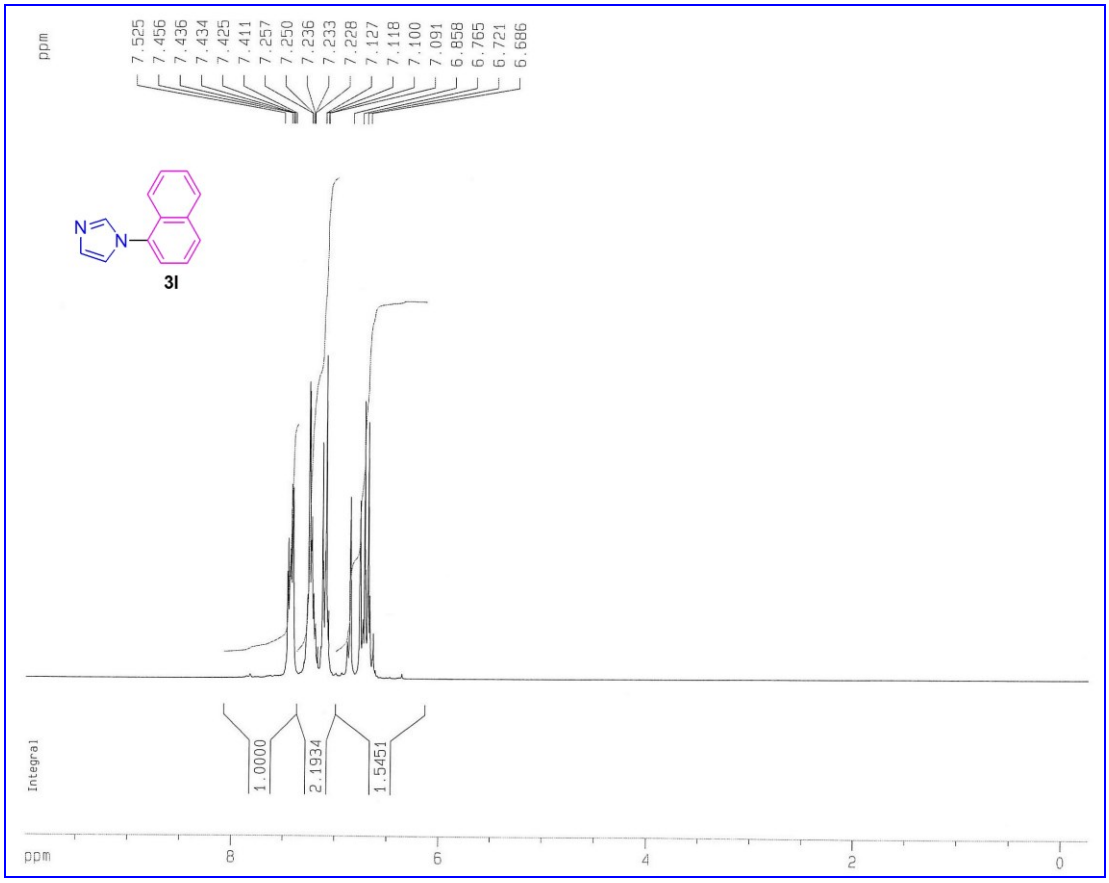

158.753
136.799
133.066
129.296
128.577
126.275
122.545
121.558
120.247
114.490
110.542
102.372
55.516
OMe
3i
150
100
50
ppm (t1)

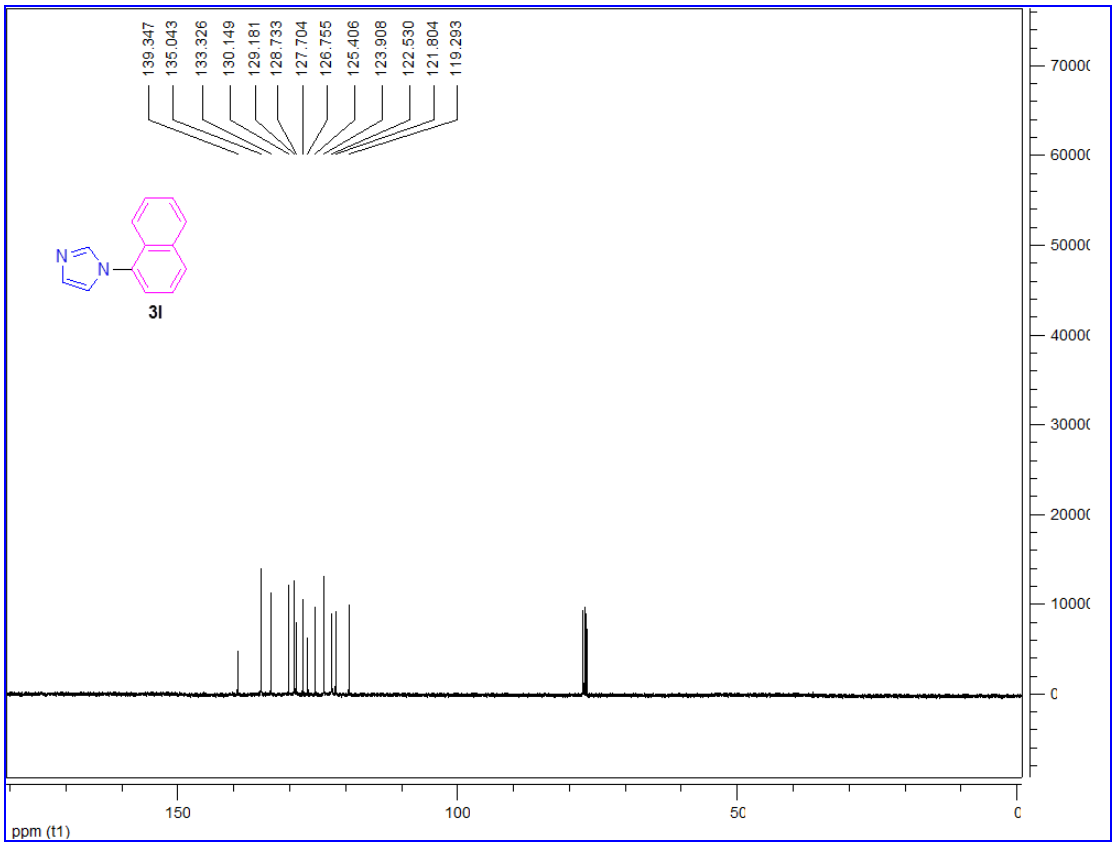

7.60
7.19
7.17
7.15
7.12
6.74
6.71
6.67
3.72
MeO
N
3j
1.00
4.35
2.29
2.97
3.16
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

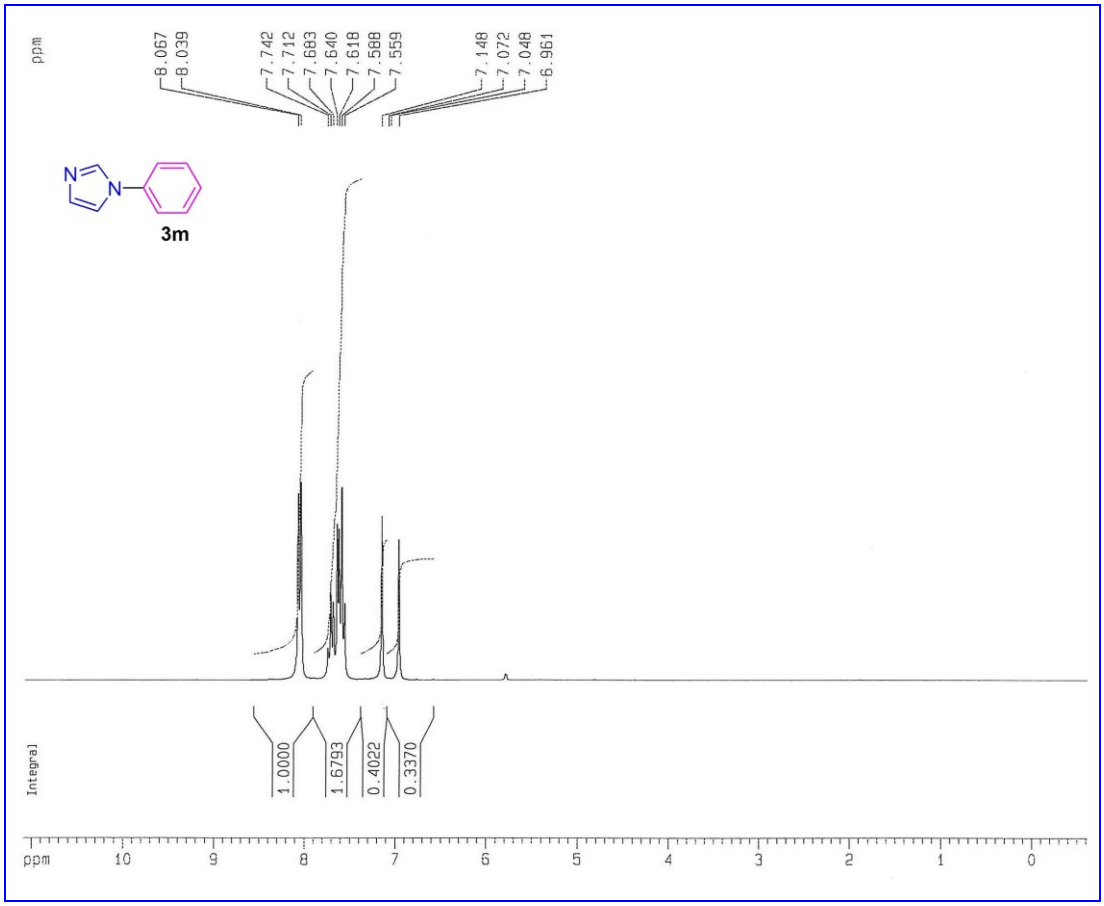

146.154
144.532
130.576
128.578
127.042
125.944
123.218
122.530
121.774
120.412
119.147
114.519
110.084
102.594
56.051
MeO
N
3j
6000
5000
4000
3000
2000
1000
0
150
100
50
0
ppm (t1)

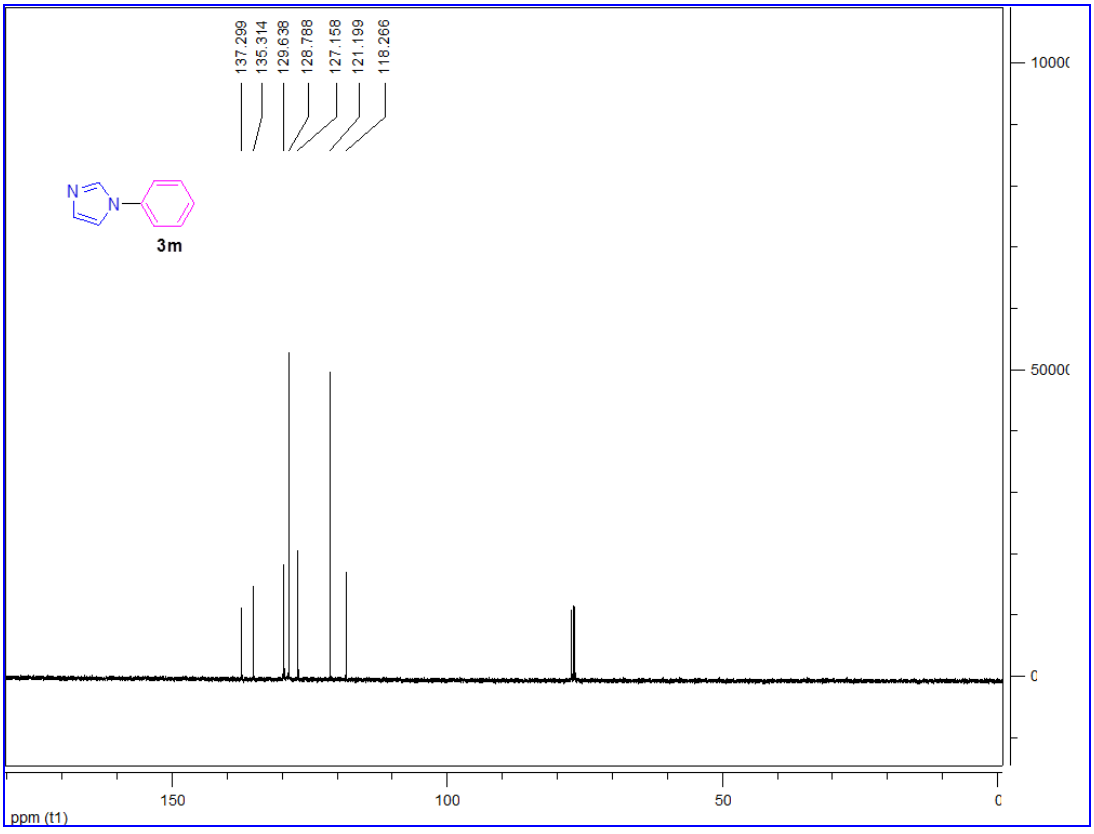

ppm
8.315
8.216
7.929
7.896
7.636
7.622
7.567
7.556
7.519
7.488
7.287
7.274
7.213
7.179
7.147
7.119
3k
Integral
1.0000
4.5498
5.5959
ppm
12
10
8
6
4
2
0

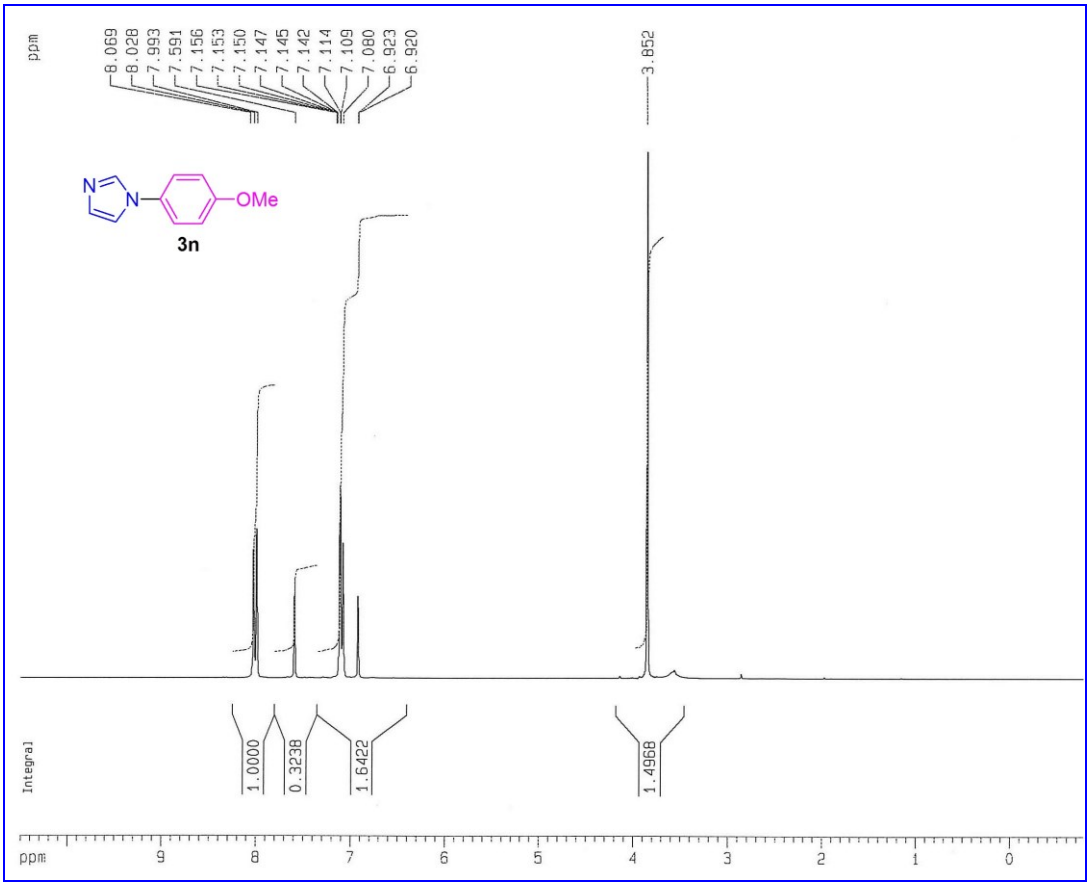

144.953
142.108
137.156
133.731
129.133
128.089
124.491
123.407
122.586
120.516
110.867
3k
1000
5000
0
150
100
50
0
ppm (t1)

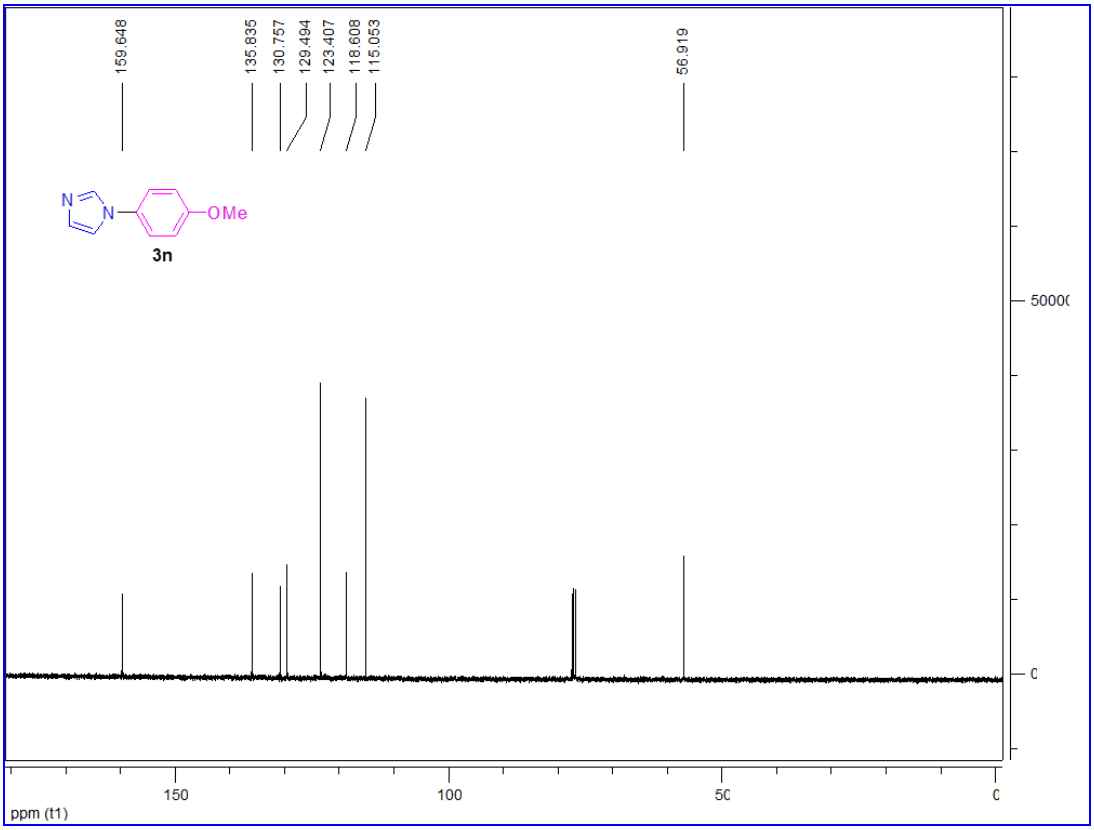

ppm
7.525
7.456
7.436
7.434
7.425
7.411
7.257
7.250
7.236
7.233
7.228
7.127
7.118
7.100
7.091
6.858
6.765
6.721
6.686
3l
Integral
1.0000
2.1934
1.5451
ppm
8
6
4
2
0

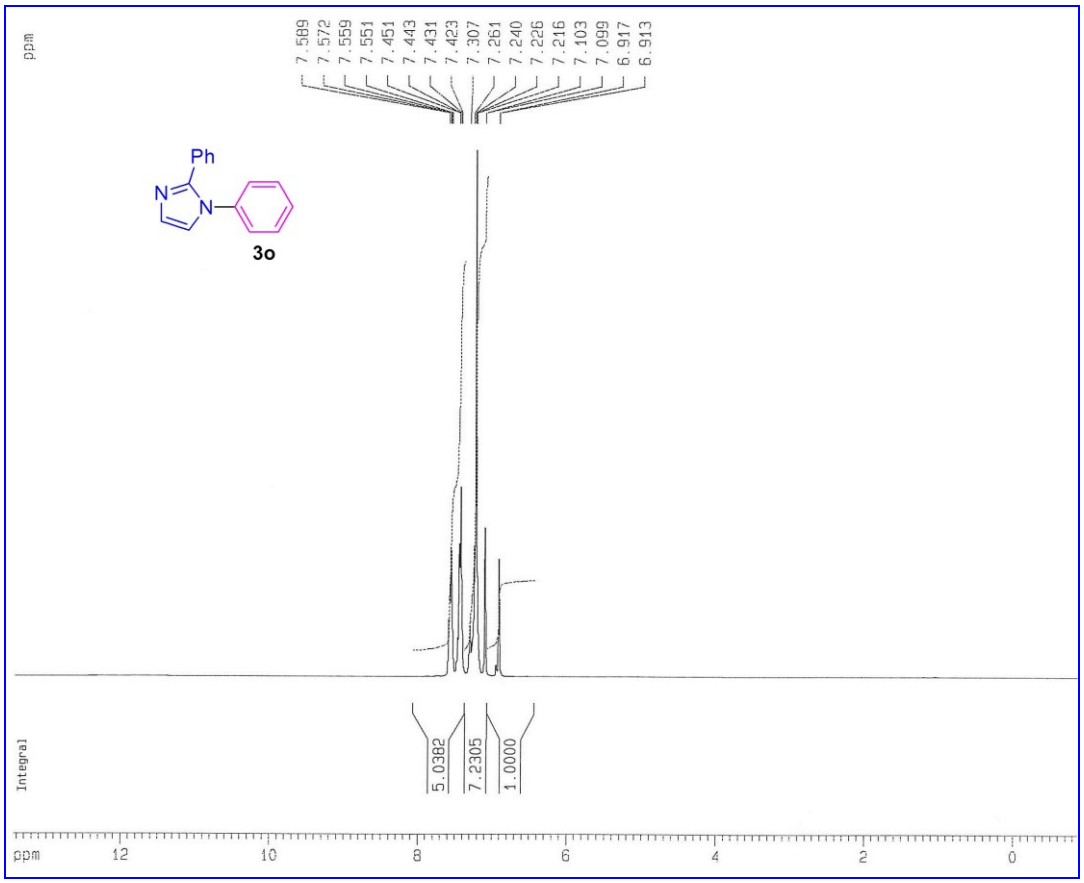

139.347
135.043
133.326
130.149
129.181
128.733
127.704
126.755
125.406
123.908
122.530
121.804
119.293
3l
7000
6000
5000
4000
3000
2000
1000
0
150
100
50
0
ppm (t1)

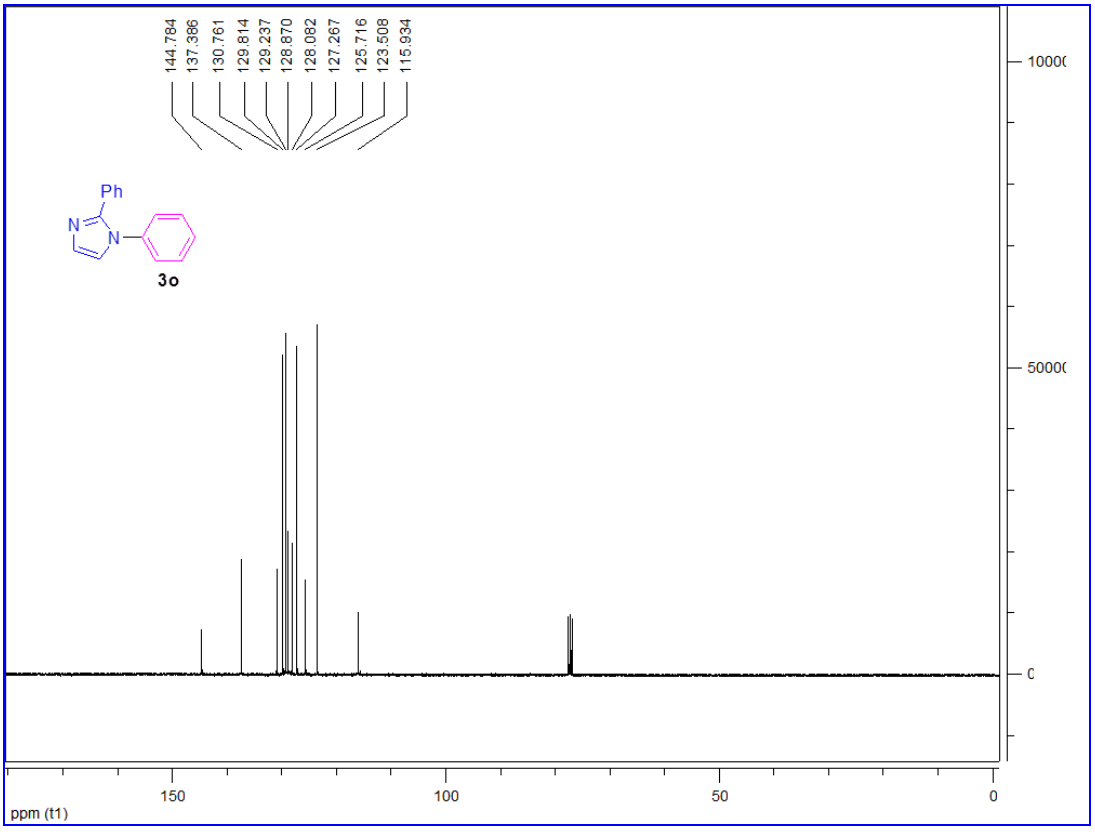

ppm
8.067
8.039
7.742
7.712
7.683
7.640
7.618
7.588
7.559
7.148
7.072
7.048
6.961
3m
Integral
1.0000
1.6793
0.4022
0.3370
ppm
10
9
8
7
6
5
4
3
2
1
0


137.299
135.314
129.638
128.788
127.158
121.199
118.266
3m
10000
50000
0
150
100
50
0
ppm (t1)


ppm
8.069
8.028
7.993
7.591
7.156
7.153
7.150
7.147
7.145
7.142
7.114
7.109
7.080
6.923
6.920
3.852
OMe
3n
Integral
1.0000
0.3238
1.6422
1.4968
ppm
9
8
7
6
5
4
3
2
1
0


159.648
135.835
130.757
129.494
123.407
118.608
115.053
56.919
OMe
3n
50000
0
150
100
50
0
ppm (t1)


ppm
7.569
7.572
7.559
7.551
7.451
7.443
7.431
7.423
7.307
7.261
7.240
7.226
7.216
7.103
7.099
6.917
6.913
Ph
N
N
3o
Integral
5.0382
7.2305
1.0000
ppm
12
10
8
6
4
2
0


144.784
137.386
130.761
129.814
129.237
128.870
128.082
127.267
125.716
123.508
115.934
Ph
N
N
3o
10000
50000
0
150
100
50
0
ppm (t1)