

Supplementary Information

CuI incorporated cobalt ferrite nanoparticle as magnetically separable catalyst for oxidative amidation reaction

Mintu Maan Dutta, Hrishikesh Talukdar, Prodeep Phukan*

Department of Chemistry, Gauhati University, Guwahati -781014, Assam, India

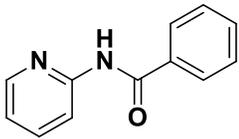
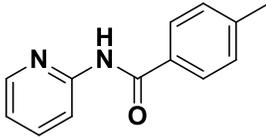
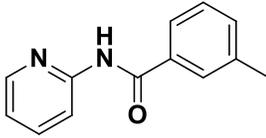
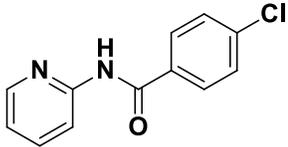
E-mail: pphukan@gauhati.ac.in

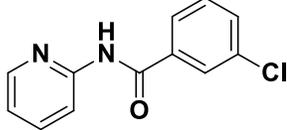
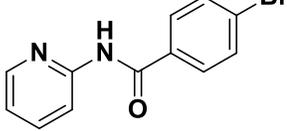
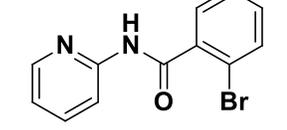
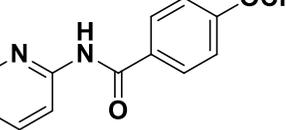
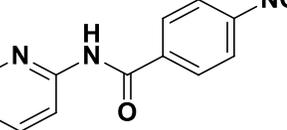
Contents:-

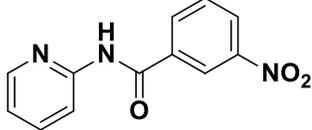
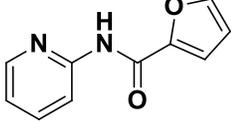
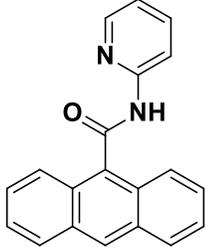
Spectroscopic Data	S2-S4
References	S4
¹ H and ¹³ C Spectra of Compounds	S5-S17

Experimental Data:

¹H NMR and ¹³C spectra were recorded in Bruker Advance 300 MHz instrument. Chemical shifts are given in δ units relative to the tetramethylsilane (TMS) signal as an internal reference in CDCl₃. Coupling constants (*J*) are reported in hertz (Hz). Silica gel (230-400 mesh) was used for column chromatography.

SI No.	NMR Data	Structure
1	<p><i>N</i>-(pyridin-2-yl)benzamide (3a): [1]</p> <p>White crystalline solid, ^1H NMR (300 MHz, CDCl_3) δ: 9.29 (s,1H), 8.41 (d, 8.4Hz, 1H), 8.11 (d, 4.2Hz, 1H), 7.93 (d, 2H, 7.2Hz), 7.77-7.71 (m, 1H), 7.57-7.44 (m, 3H), 7.03-6.99 (m, 1H); ^{13}C NMR (75 MHz, CDCl_3) δ: 166.0, 151.7, 147.7, 138.5, 134.3, 132.1, 128.7, 119.8, 114.3.</p>	
2	<p>4-methyl-<i>N</i>-(pyridin-2-yl)benzamide (3b): [1]</p> <p>White Solid, ^1H NMR (300 MHz, CDCl_3) δ: 8.74 (s, 1H), 8.40 (d, 8.4Hz, 1H), 8.27 (d, 1H, 3.9Hz), 7.84 (d, 7.8Hz, 2H), 7.31 (d, 7.8Hz, 2H), 7.78-7.73 (m, 1H), 7.30 (d, 8.4Hz, 1H), 2.43 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ: 165.7, 151.6, 147.8, 142.9, 138.5, 131.3, 129.5, 127.2, 119.8, 114.1, 21.5.</p>	
3	<p>3-methyl-<i>N</i>-(pyridin-2-yl)benzamide (3c): [2]</p> <p>White solid, ^1H NMR (300 MHz, CDCl_3) δ: 9.76 (s, 1H), 8.45 (d, 1H, 8.4Hz), 8.11 (d, 1H, 4.8Hz), 7.78-7.74 (m, 3H), 7.36-7.33 (m, 2H), 7.01 (t, 1H, 6.9 Hz), 2.38 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ: 166.3, 151.8, 147.1, 138.6, 138.3, 134.0, 132.8, 128.1, 124.5, 119.6, 114.6, 21.9.</p>	
4	<p>4-chloro-<i>N</i>-(pyridin-2-yl)benzamide (3d): [1]</p> <p>White crystalline solid, ^1H NMR (300 MHz, CDCl_3) δ: 9.76 (s,1H), 8.37 (d, 8.4.Hz, 1H), 7.99 (d, 3.9Hz, 1H), 7.86 (d, 8.4Hz,2H), 7.71 (t, 8.4Hz, 1H), 7.38 (d, 2H, 8.4Hz), 6.99 (t, 1H, 5.1 Hz); ^{13}C NMR (75 MHz, CDCl_3) δ: 165.1, 151.6, 147.5, 138.5, 138.3, 132.7, 128.8, 119.9, 114.6.</p>	

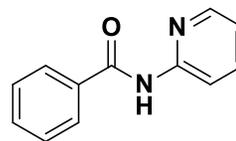
5	<p>3-chloro-<i>N</i>-(pyridin-2-yl)benzamide (3e): [4]</p> <p>White solid, ^1H NMR (300 MHz, CDCl_3) δ: 8.94 (s, 1H), 8.38 (d, 8.1Hz, 1H), 8.25 (d, 3.6Hz, 1H), 7.89 (d, 2H, 8.4Hz), 7.77 (t, 1H, 7.5Hz), 7.47 (d, 2H, 8.4Hz), 7.09 (t, 6.6Hz, 1H); ^{13}C NMR (75 MHz, CDCl_3) δ: 164.7, 151.4, 147.7, 138.6, 132.5, 129.1, 128.7, 120.1, 114.3.</p>	
6	<p>4-bromo-<i>N</i>-(pyridin-2-yl)benzamide (3f): [1]</p> <p>White crystalline solid, ^1H NMR (300 MHz, CDCl_3) δ: 9.42 (s, 1H), 8.38 (d, 8.4Hz, 1H), 8.10 (d, 3.9Hz, 1H), 7.81-7.72 (m, 3H), 7.58 (d, 8.4Hz, 2H), 7.04 (t, 6.3Hz, 1H); ^{13}C NMR (75 MHz, CDCl_3) δ: 165.1, 151.5, 147.6, 138.6, 133.1, 131.9, 128.9, 126.9, 120.0, 114.5.</p>	
7	<p>2-bromo-<i>N</i>-(pyridin-2-yl)benzamide (3g): [1]</p> <p>White crystalline solid, ^1H NMR (300 MHz, CDCl_3) δ: 10.60 (s, 1H), 8.39 (d, 8.1Hz, 1H), 7.69 (t, 7.5Hz, 1H), 7.57-7.52 (m, 2H), 7.40-7.29 (m, 3H), 6.86-6.82 (m, 1H); ^{13}C NMR (75 MHz, CDCl_3) δ: 166.6, 151.6, 146.9, 138.6, 138.0, 133.3, 131.3, 129.0, 127.5, 119.8, 119.5, 114.7</p>	
8	<p>4-methoxy-<i>N</i>-(pyridin-2-yl)benzamide (3h): [1]</p> <p>Yellow solid, ^1H NMR (300 MHz, CDCl_3) δ: 9.52 (s, 1H), 8.41 (d, 8.4 Hz, 1H), 8.17 (s, 1H), 7.94 (d, 8.7Hz, 2H), 7.76-7.71 (m, 1H), 7.03-6.99 (m, 1H), 6.93 (d, 8.7Hz, 2H), 3.83 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3) δ: 165.6, 162.6, 151.9, 147.2, 138.6, 131.8, 129.4, 126.2, 119.5, 114.5, 113.8, 113.4, 55.3.</p>	
9	<p>4-nitro-<i>N</i>-(pyridin-2-yl)benzamide (3i): [3]</p> <p>White solid, ^1H NMR (300 MHz, CDCl_3) δ: 11.20 (s, 1H), 8.40-8.16 (m, 6H), 7.86 (t, 8.1Hz, 1H), 7.21-7.17 (m, 1H); ^{13}C NMR (75 MHz, CDCl_3) δ: 164.7, 151.9, 149.3, 148.2, 139.9, 138.4, 129.7, 123.5, 120.4, 114.9.</p>	

10	<p>3-nitro-<i>N</i>-(pyridin-2-yl)benzamide (3j): [4]</p> <p>Pale yellow solid, ¹H NMR (300 MHz, CDCl₃) δ: 9.76 (s, 1H), 8.77 (s, 1H), 8.37-8.22 (m, 4H), 8.14 (d, 3.6Hz, 1H), 7.79-7.55 (m, 3H), 7.06 (t, 6Hz, 1H); ¹³C NMR (75 MHz, CDCl₃) δ: 163.7, 151.2, 147.7, 138.7, 139.9, 133.3, 129.9, 126.5, 122.5, 120.4, 114.7.</p>	
11	<p><i>N</i>-(pyridin-2-yl)furan-2-carboxamide (3k): [1]</p> <p>White solid, ¹H NMR (300 MHz, CDCl₃) δ: 8.83 (s, 1H), 8.33 (d, 8.7Hz, 2H), 7.77-7.74 (m, 1H), 7.54 (s, 1H), 7.28 (d, 3.3Hz, 1H), 7.09-7.05 (m, 1H), 6.58-6.57 (m, 1H); ¹³C NMR (75 MHz, CDCl₃) δ: 156.2, 150.9, 147.9, 144.7, 138.4, 119.9, 115.9, 114.1, 112.6.</p>	
12	<p><i>N</i>-(pyridin-2-yl) anthracene-9-carboxamide (3l):</p> <p>Yellow crystal, ¹H NMR (300 MHz, CDCl₃) δ: 9.88 (s,1H), 8.54 (d, 2H, 6Hz), 8.13 (d, 2H, 8.1Hz), 8.03 (d, 8.1Hz, 2H), 7.71-7.65 (m, 1H), 7.57-7.47 (m, 4H), 7.21 (d, 4.2Hz, 1H), 6.78-6.74 (m, 1H); ¹³C NMR (75 MHz, CDCl₃) δ: 168.4, 151.4, 147.2, 138.4, 130.9, 128.7, 128.5, 127.9, 127.2, 126.9, 125.6, 124.7, 119.8, 114.3. HRMS (ES) m/z 299.1188 [M+H, 100].</p>	

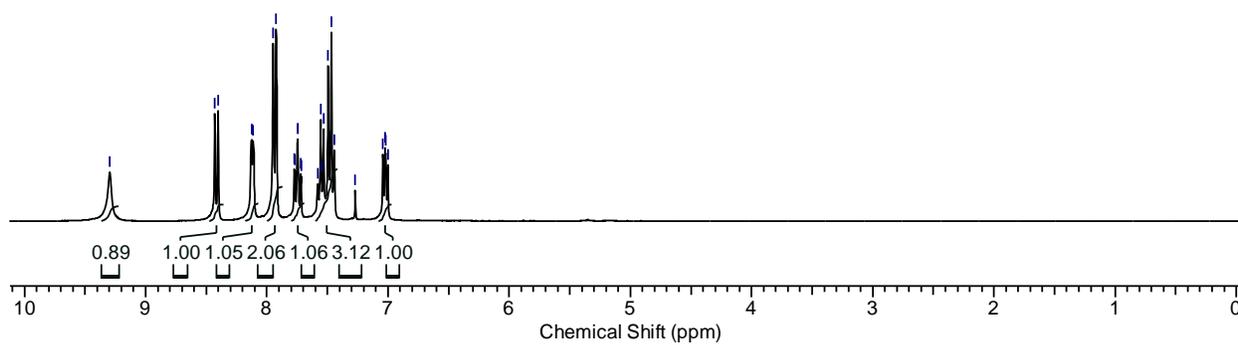
References

1. S. Yang, H. Yan, X. Ren, X. Shi, J. Li, Y. Wang and G. Huang, *Tetrahedron*, 2013, **69**, 6431.
2. K. Yan, D. Yang, W. Wei, G. Li, M. Sun, Q. Zhang, L. Tian and H. Wang, *RSC Adv.*, 2015, **5**, 100102.
3. E. S. Devi, A. Alanthadka, A. Tamilselvi, S. Nagarajan, V. Sridharana and C. U. Maheswari, *Org. Biomol. Chem.*, 2016, **14**, 8228.
4. V. Pappula, C. Ravi, S. Samanta and S. Adimurthy, *ChemistrySelect*, 2017, **2**, 5887.

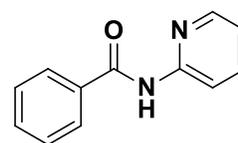
9.296
8.430
8.403
8.126
8.112
7.947
7.923
7.746
7.743
7.556
7.532
7.494
7.468
7.041
7.025
7.020



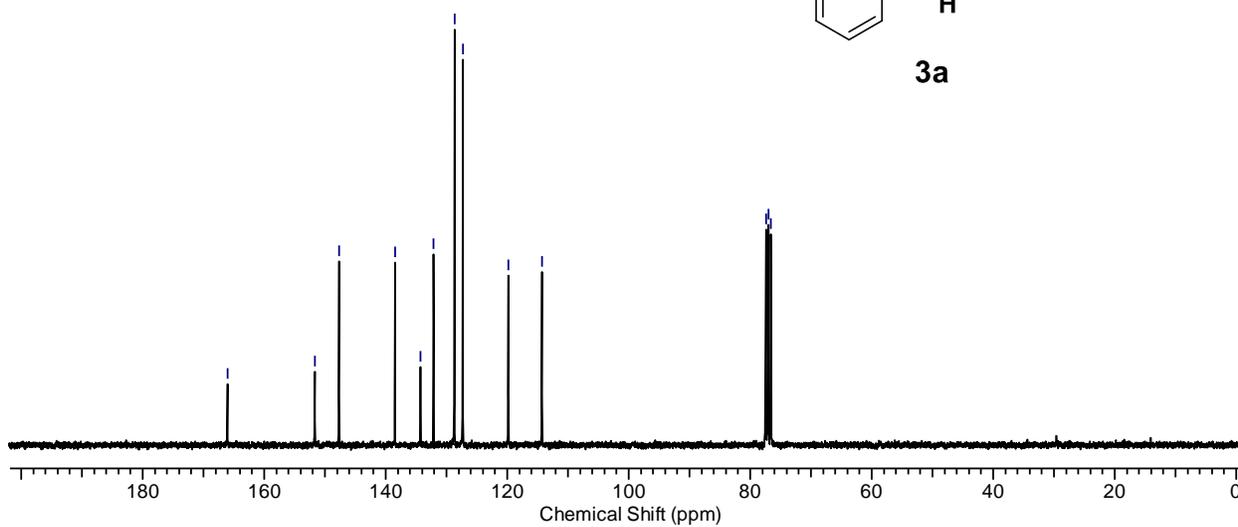
3a

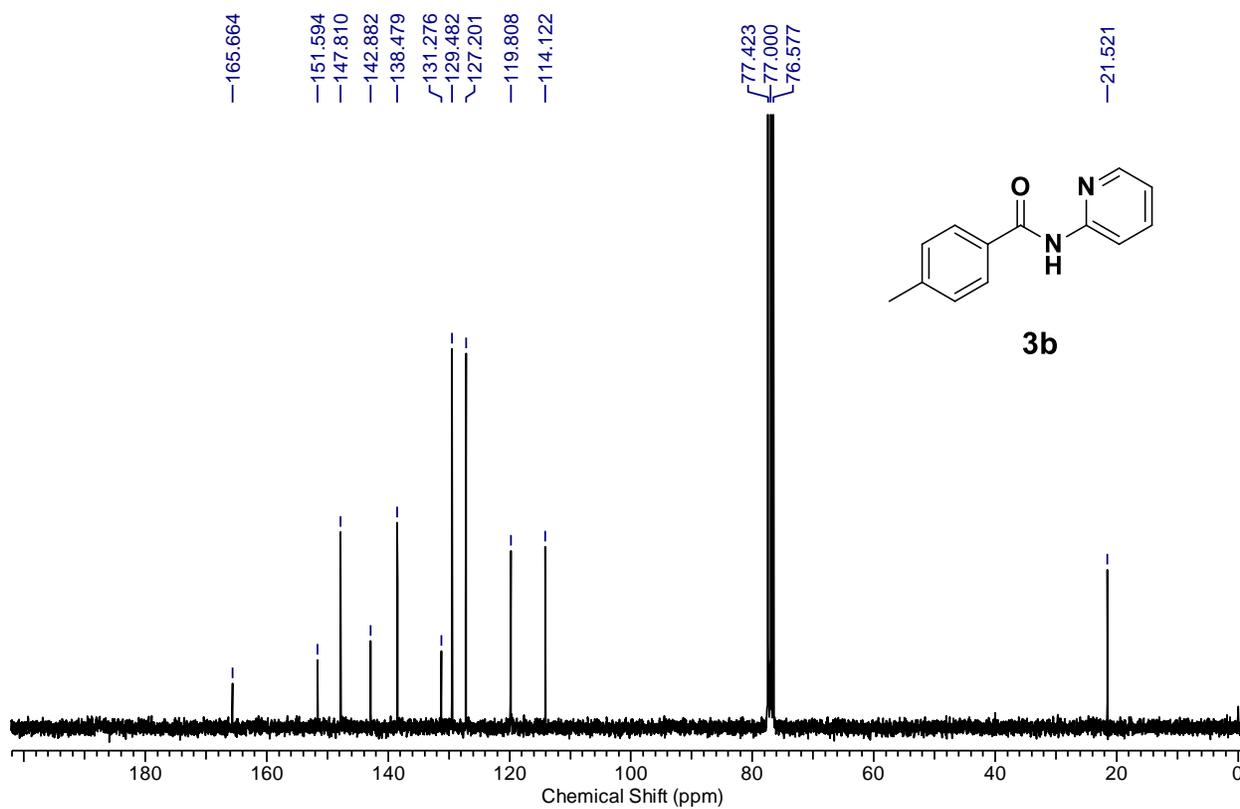
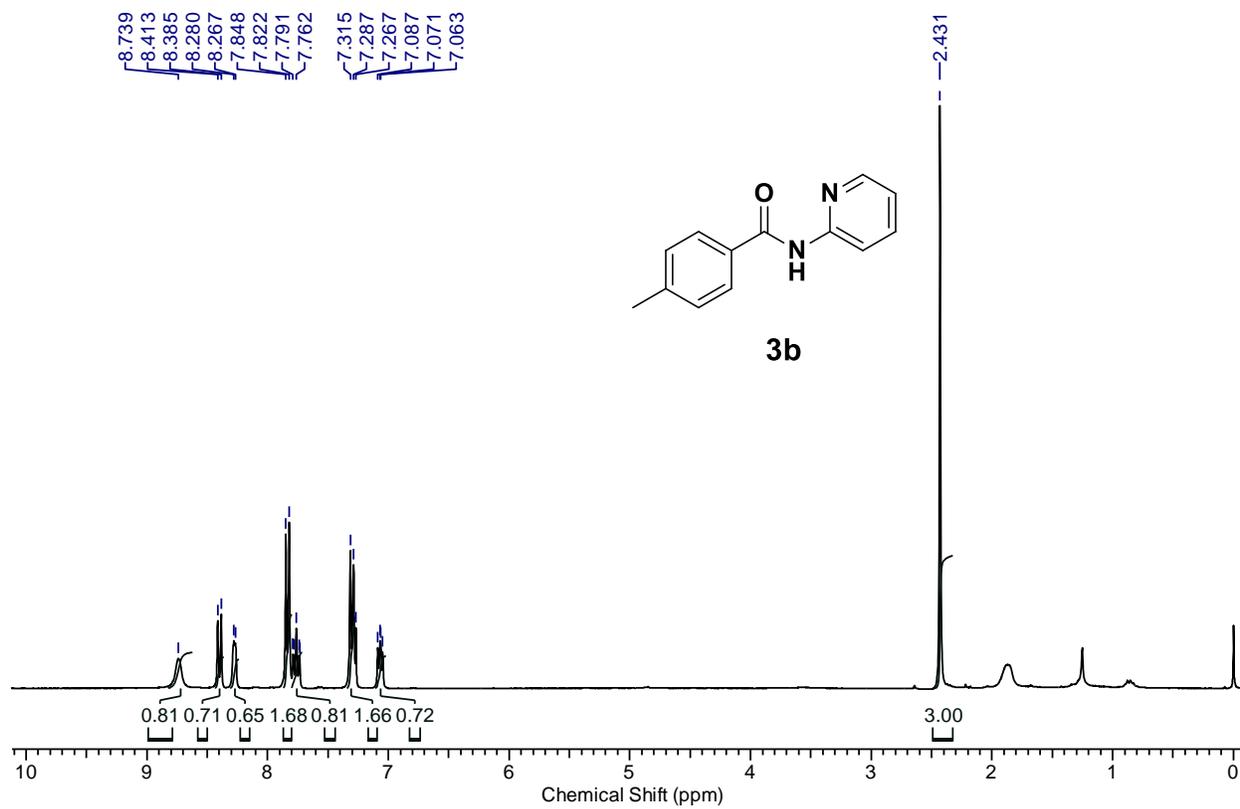


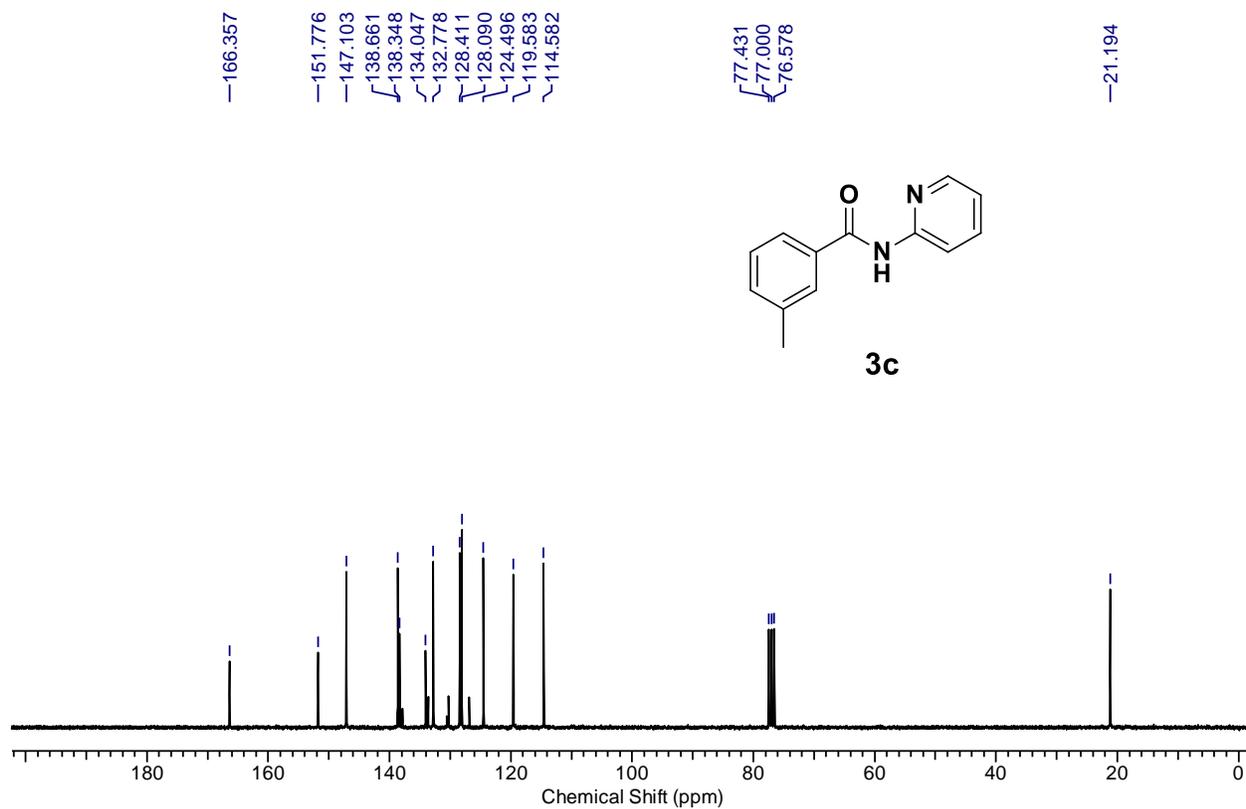
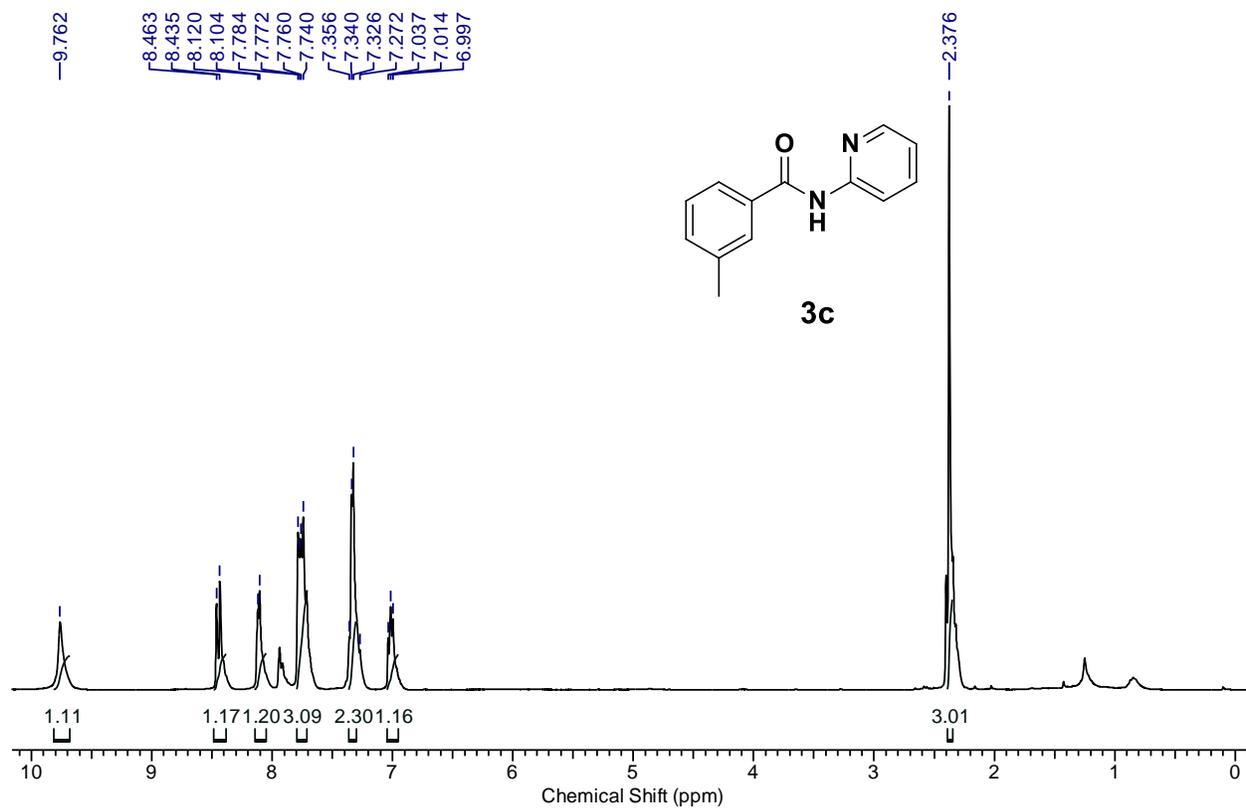
166.023
151.661
147.680
138.465
134.274
132.123
128.689
127.282
119.788
114.306
77.424
77.001
76.579

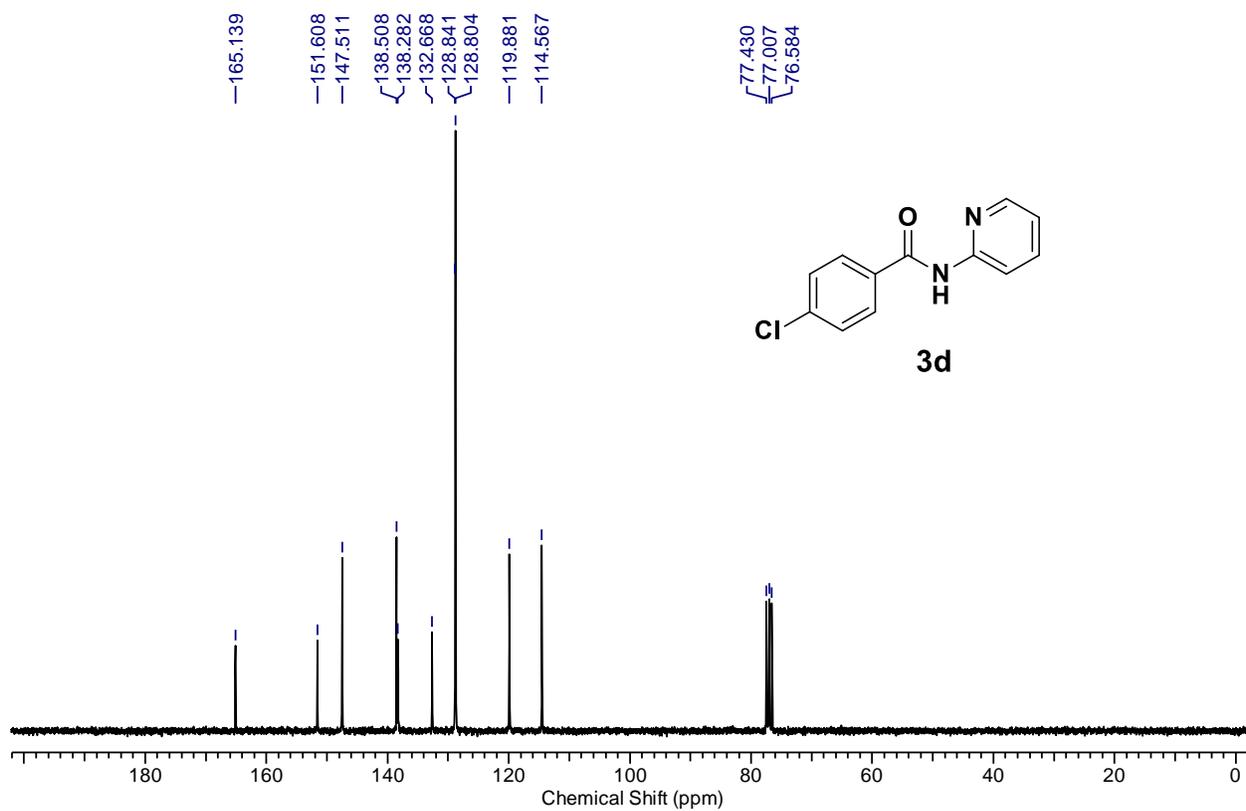
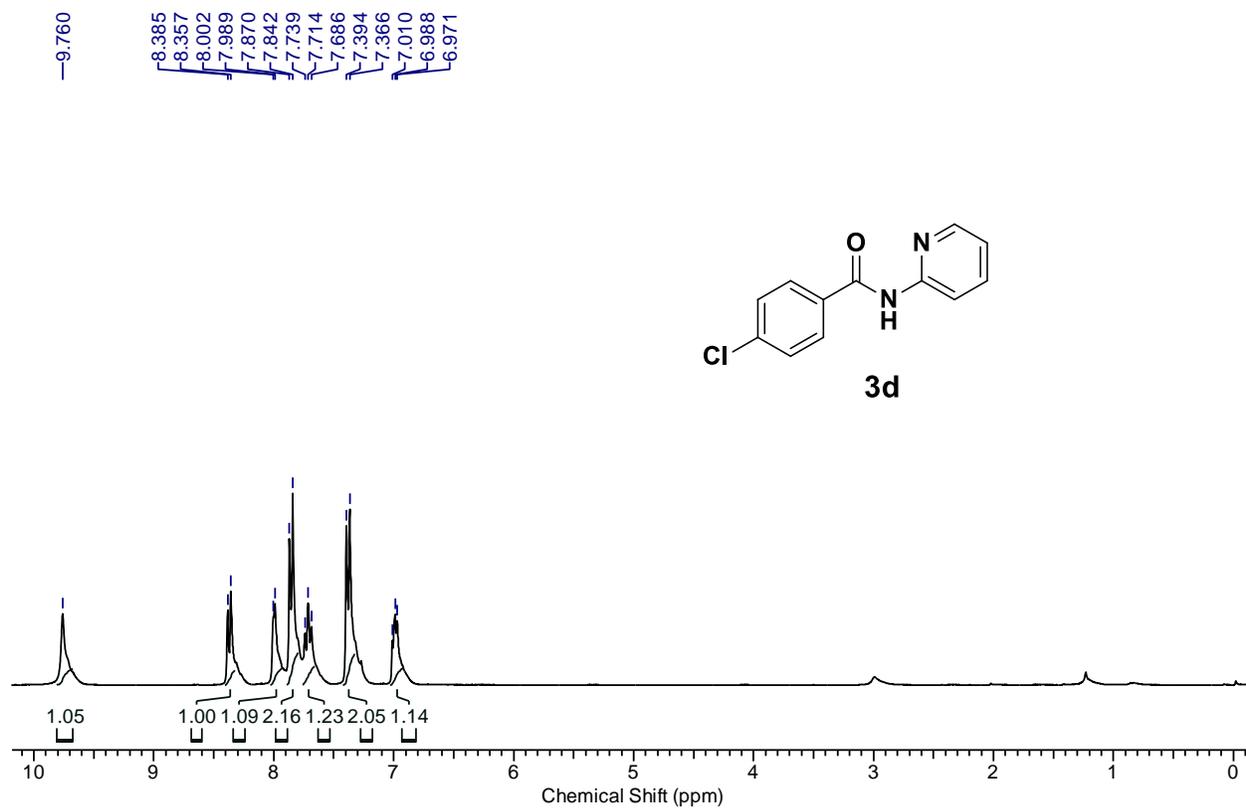


3a

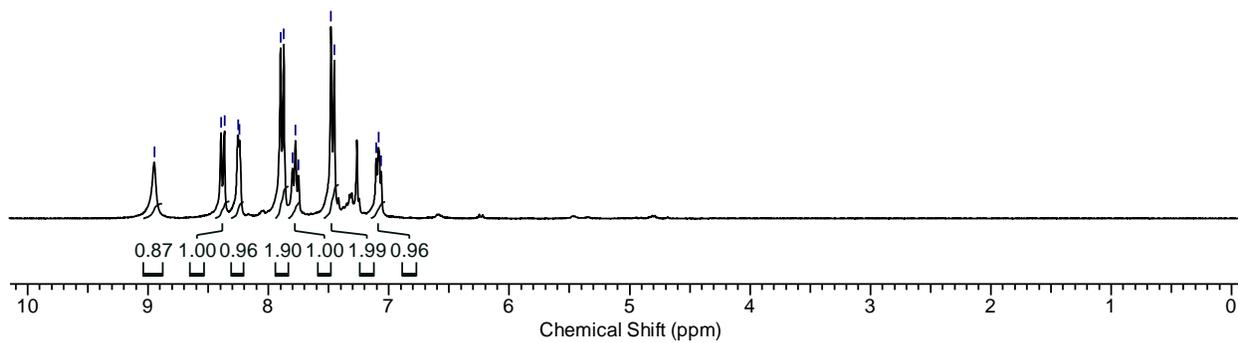
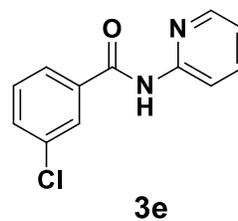






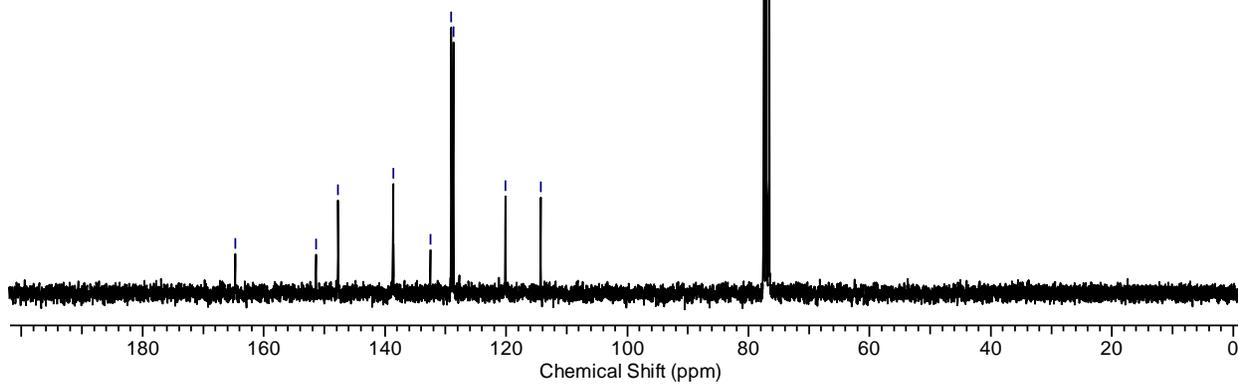
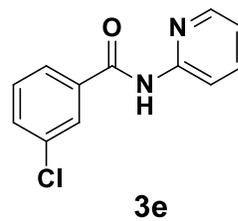


8.948
8.393
8.366
8.252
8.240
7.900
7.872
7.801
7.774
7.749
7.481
7.453
7.105
7.086
7.064

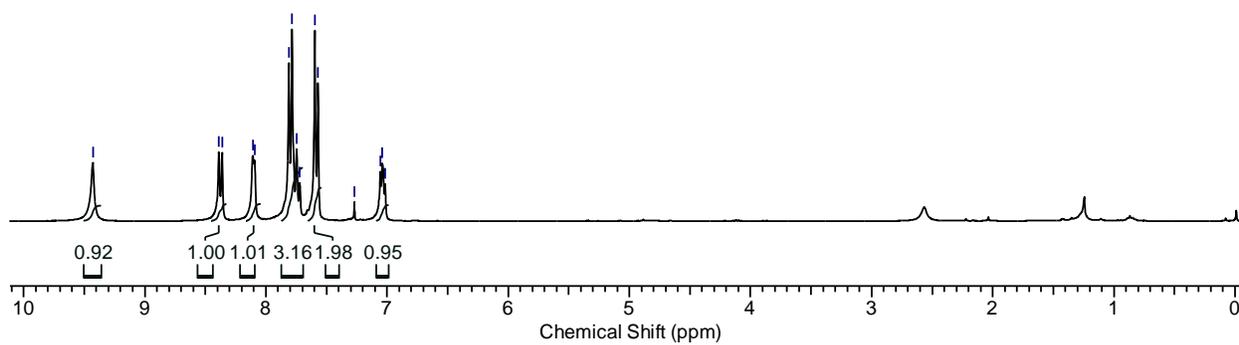
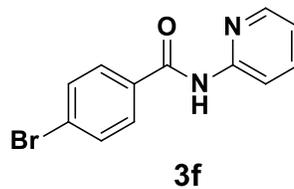


164.731
151.368
147.745
138.646
132.523
129.067
128.695
120.107
114.319

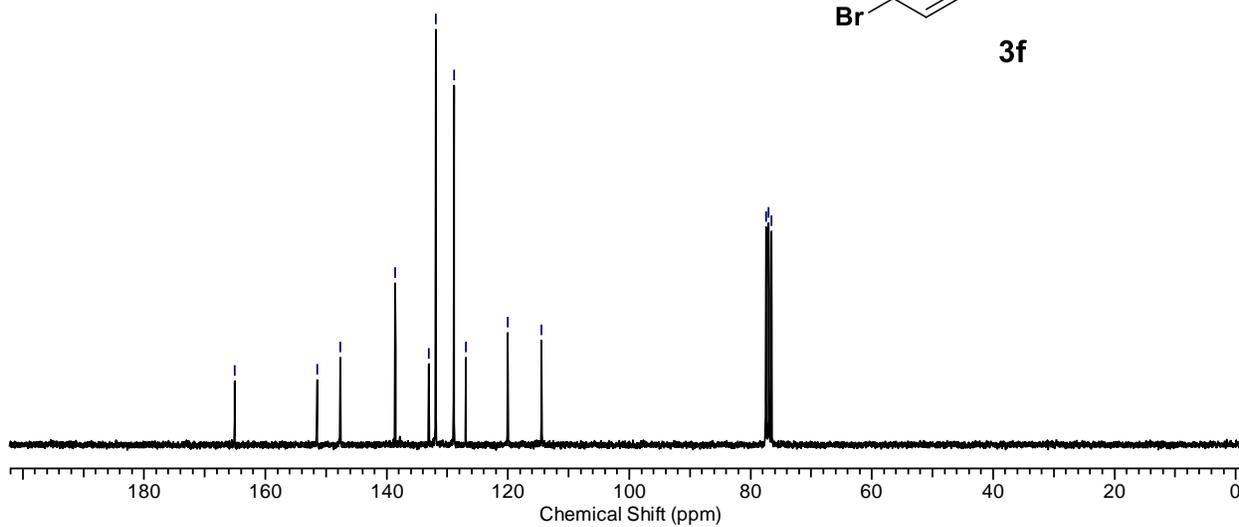
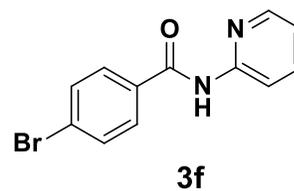
77.423
77.000
76.577

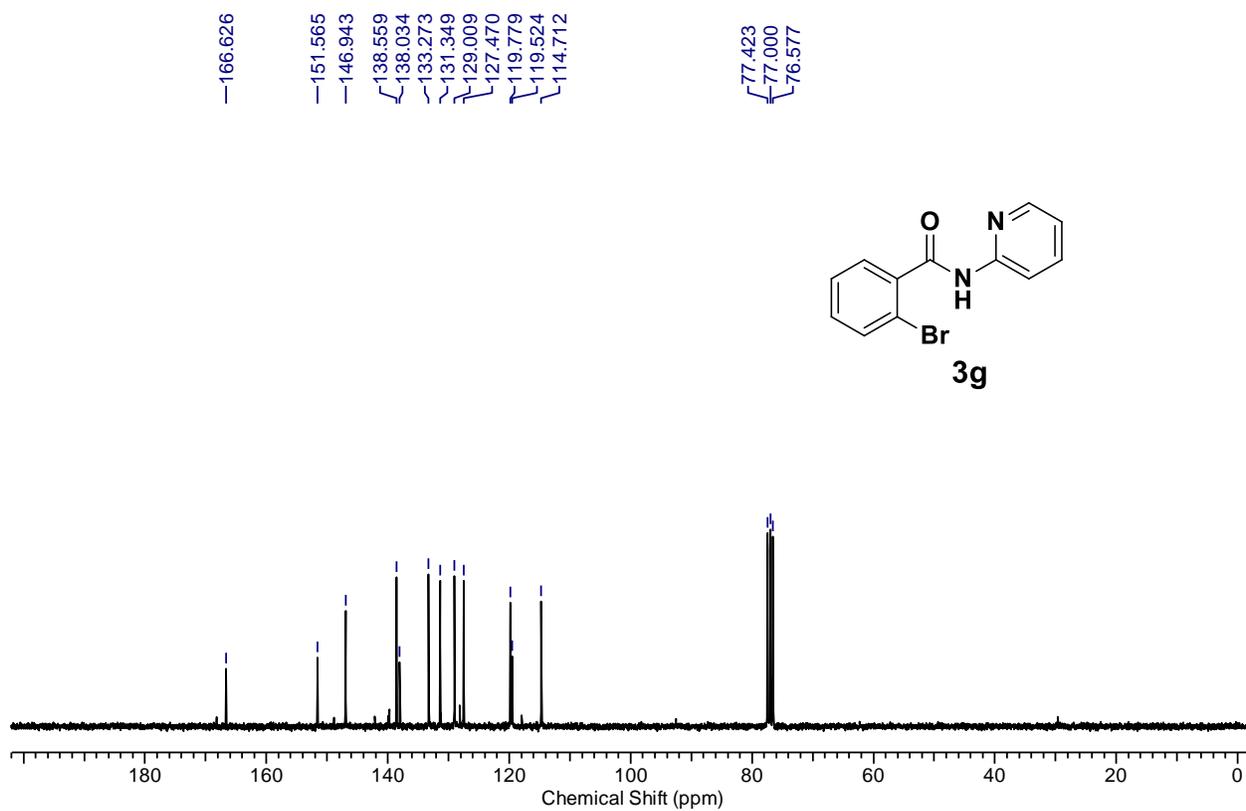
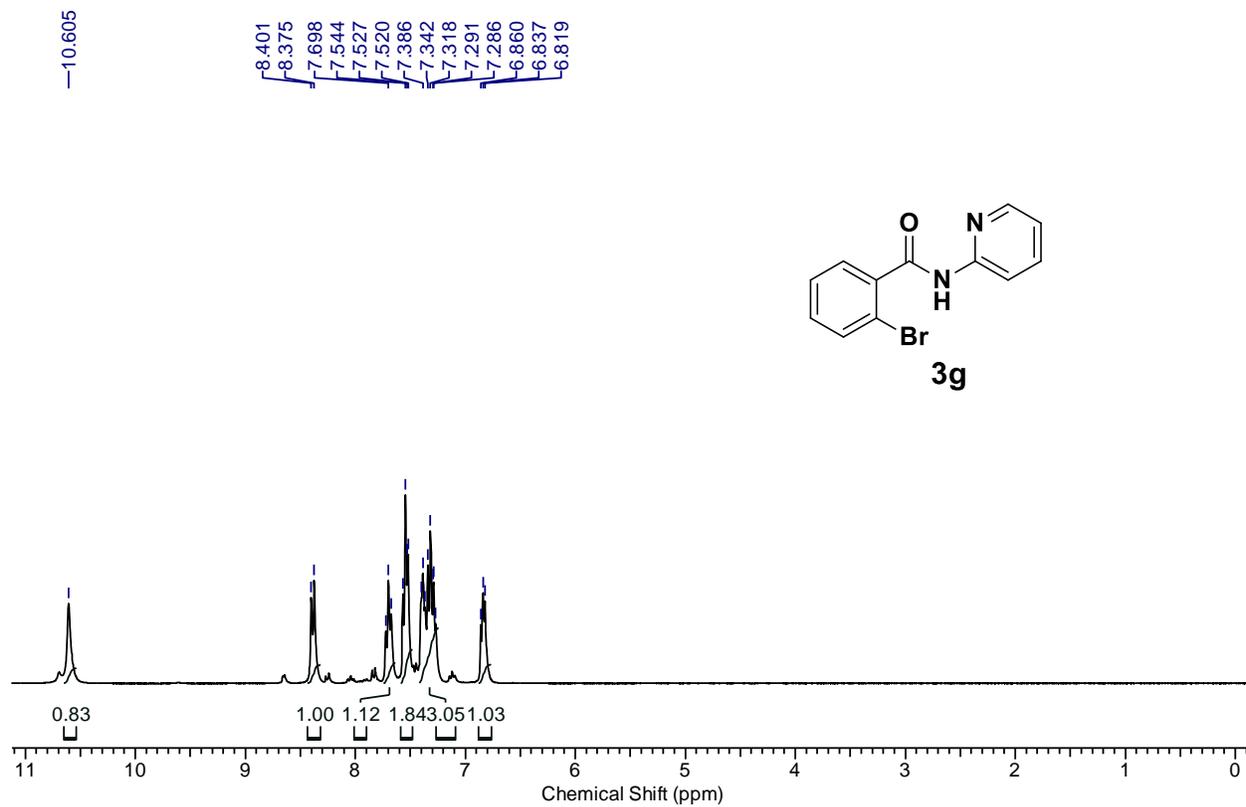


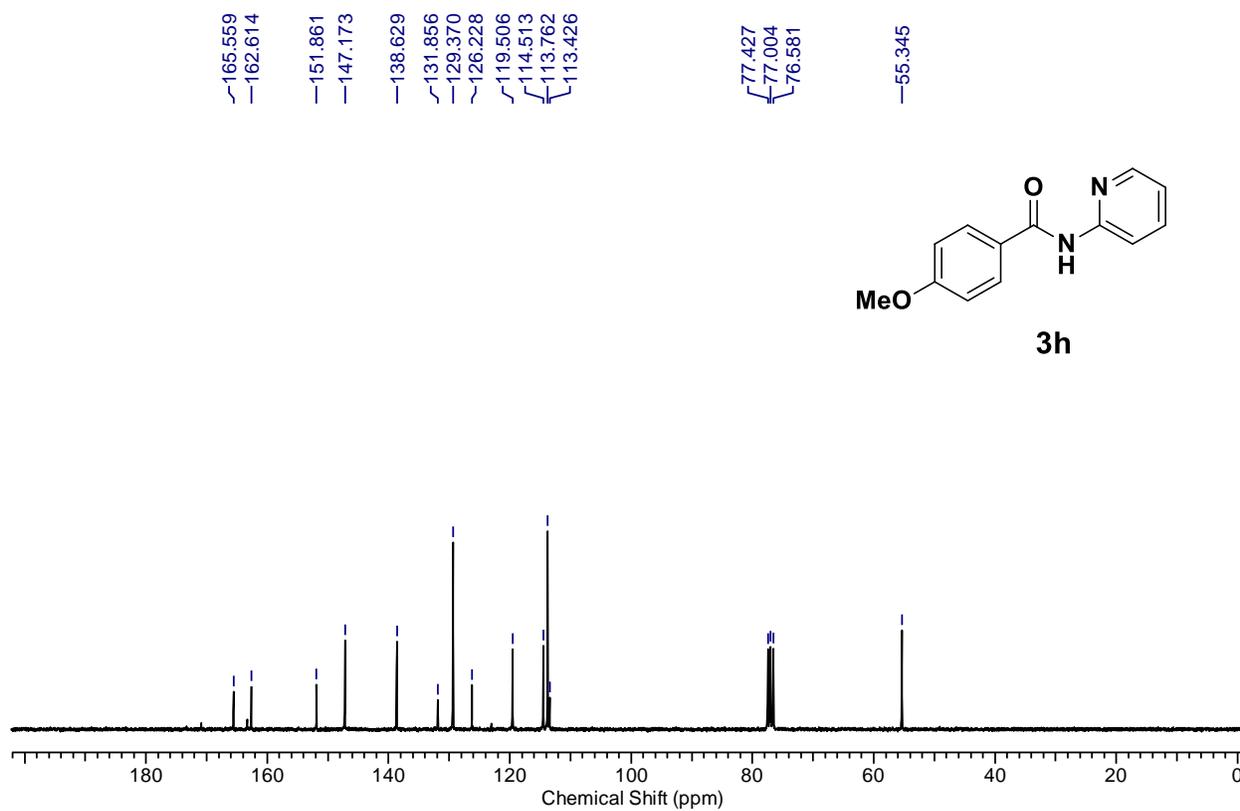
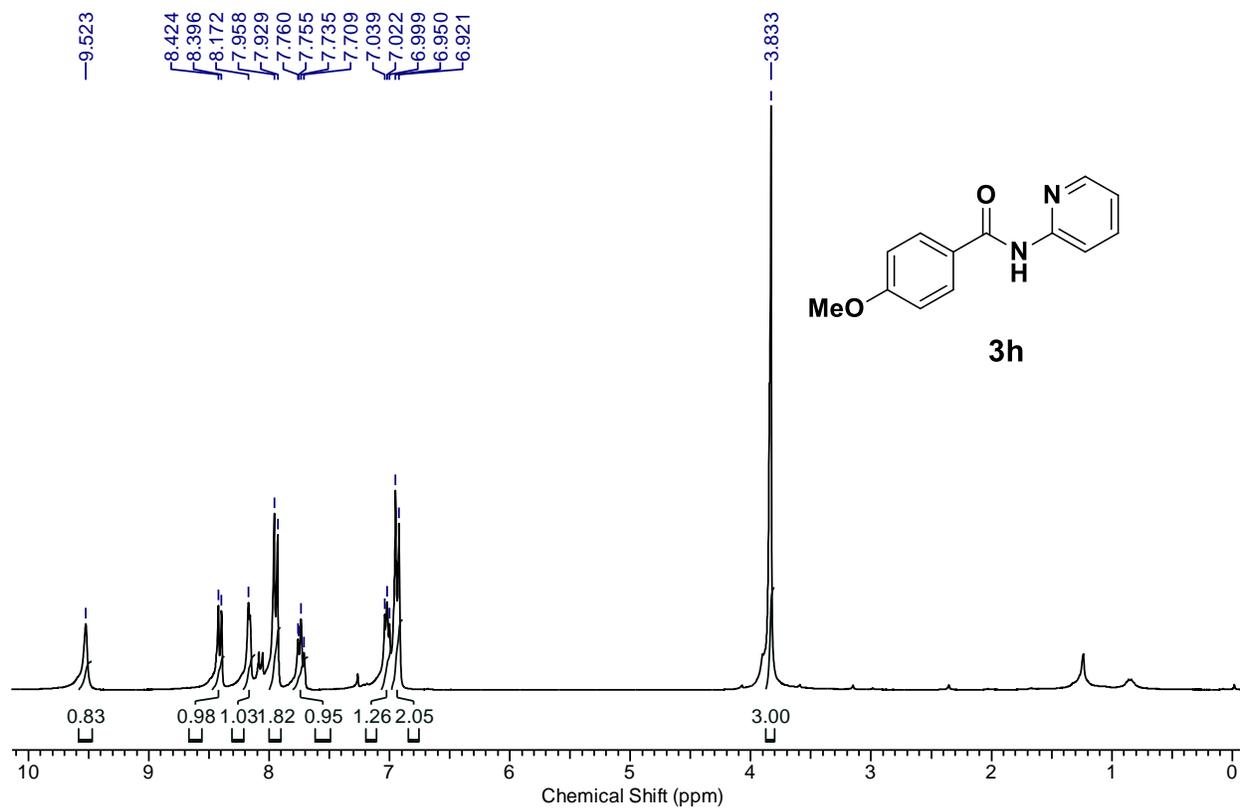
9.428
8.390
8.362
8.107
8.094
7.811
7.783
7.746
7.721
7.597
7.569
7.270
7.056
7.038
7.017

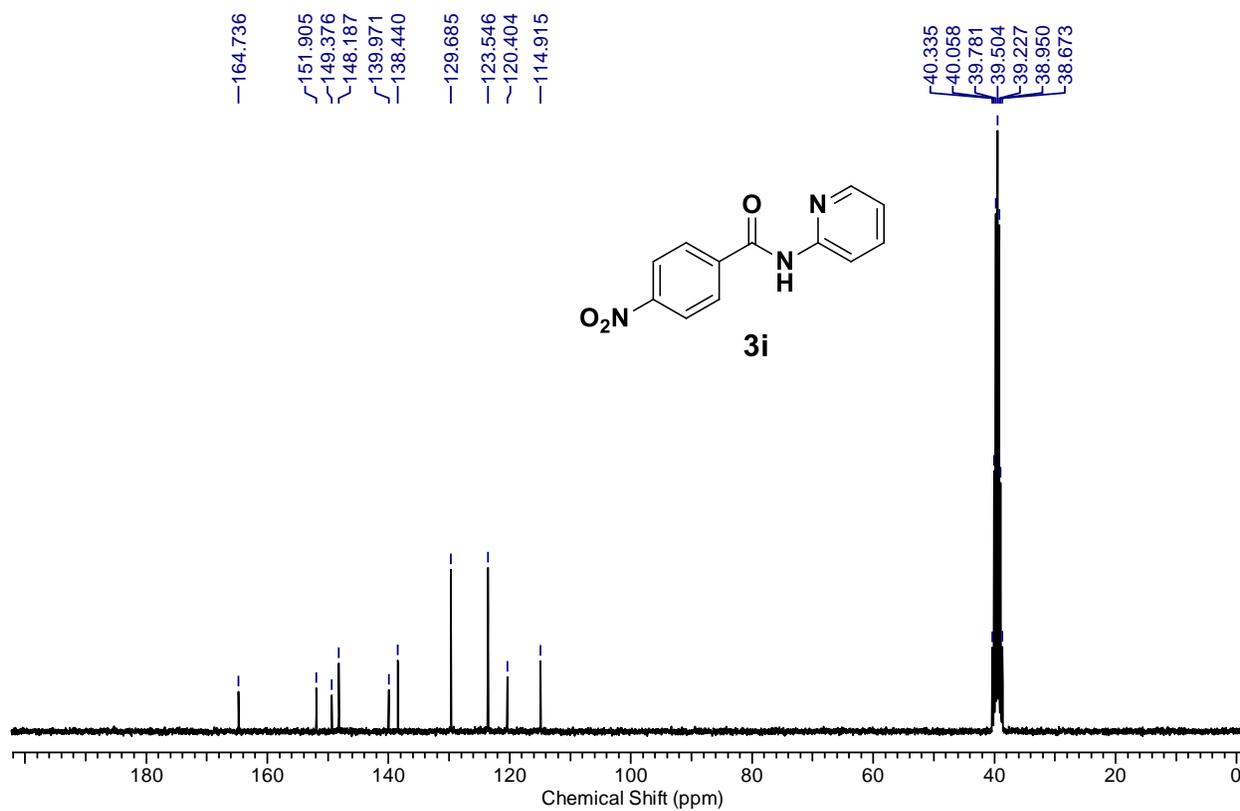
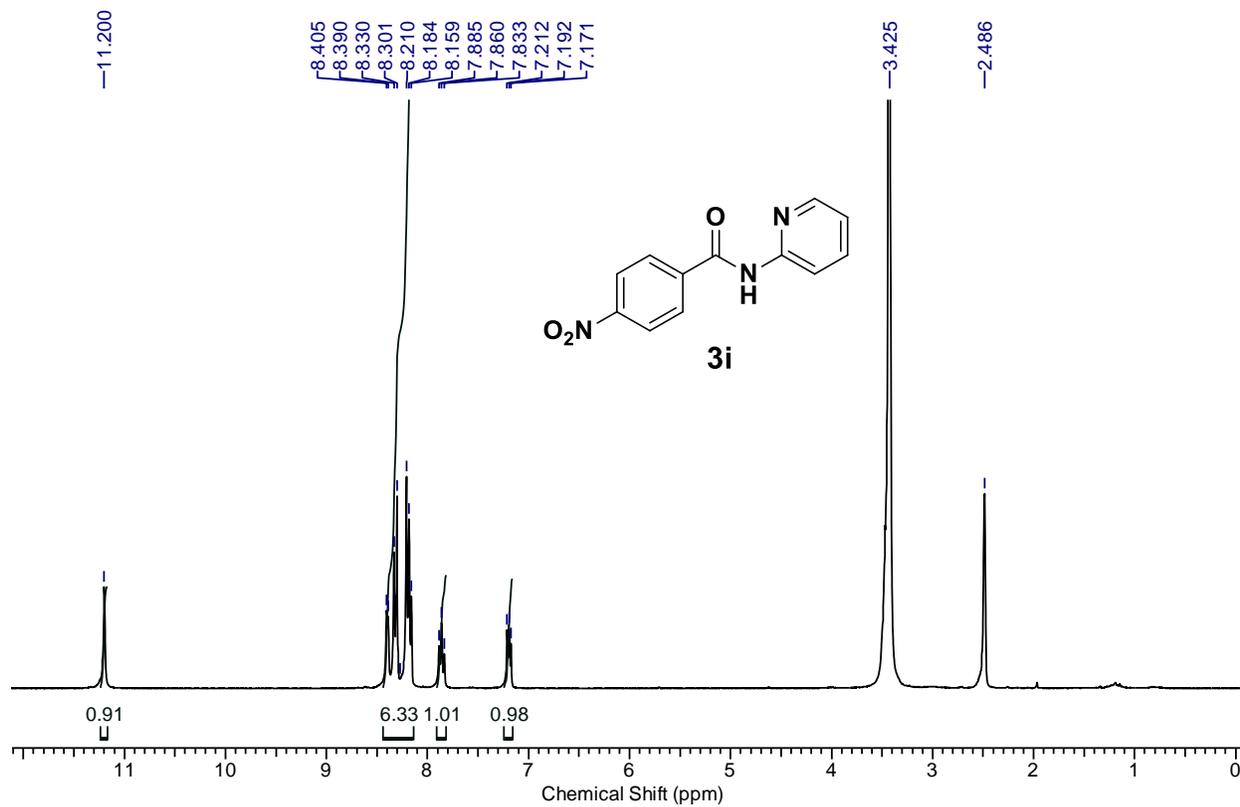


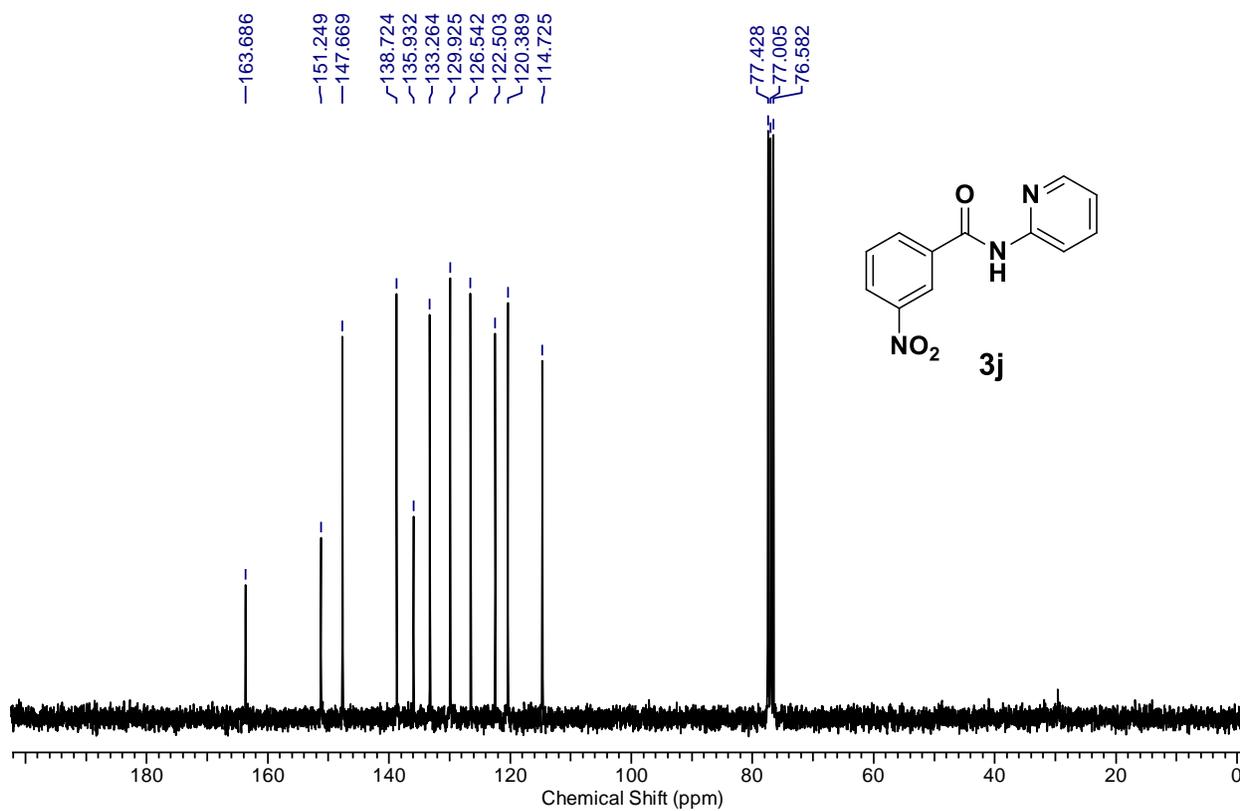
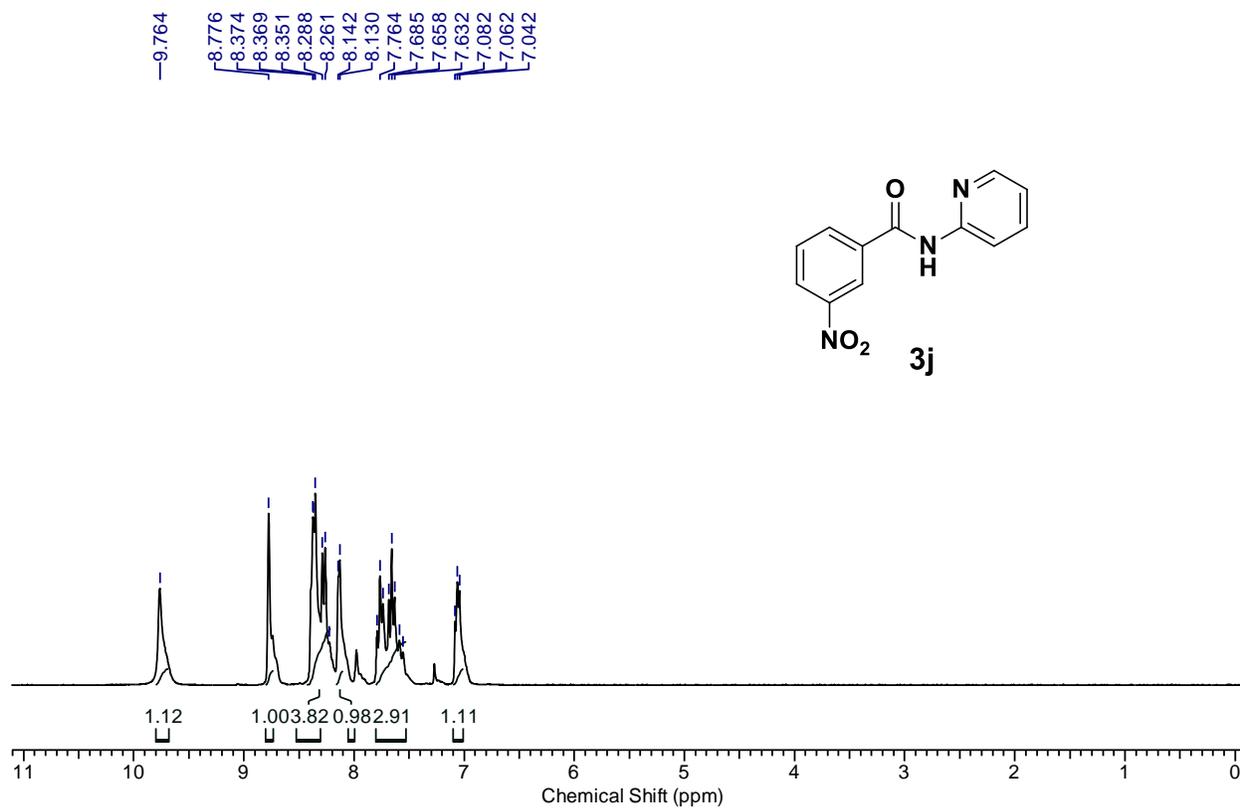
165.077
151.488
147.646
138.569
133.080
131.899
128.924
126.934
120.008
114.468
77.426
77.003
76.580



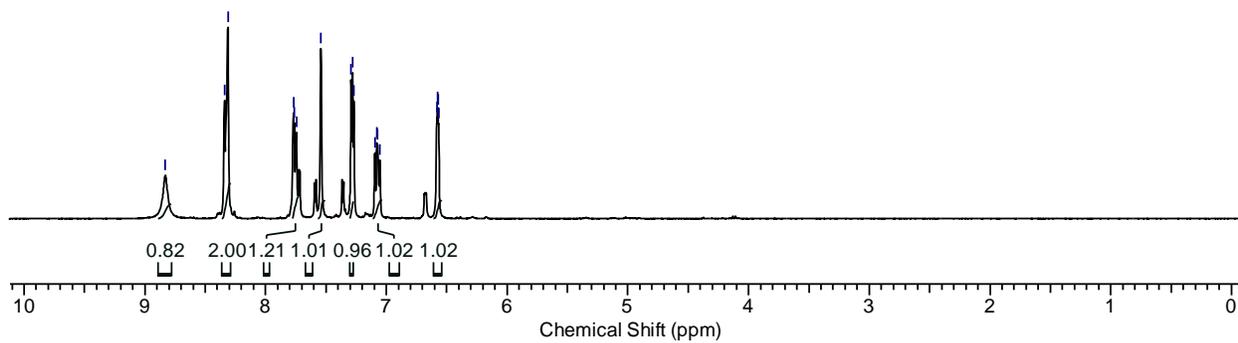
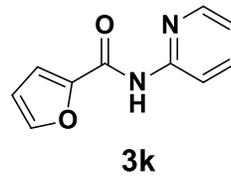




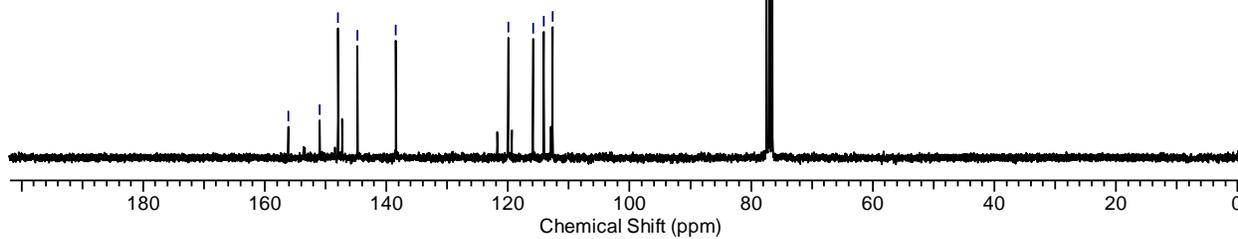
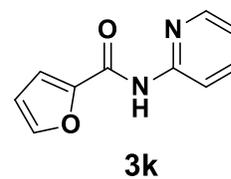


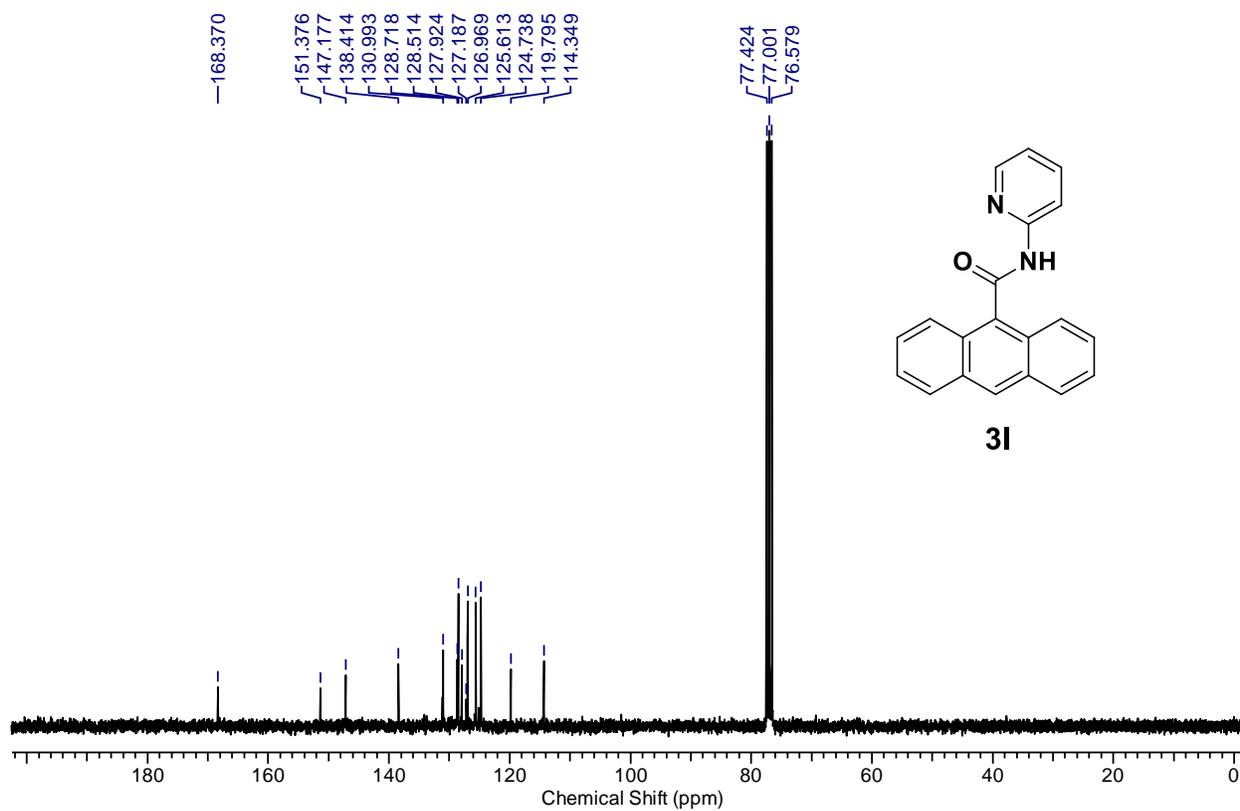
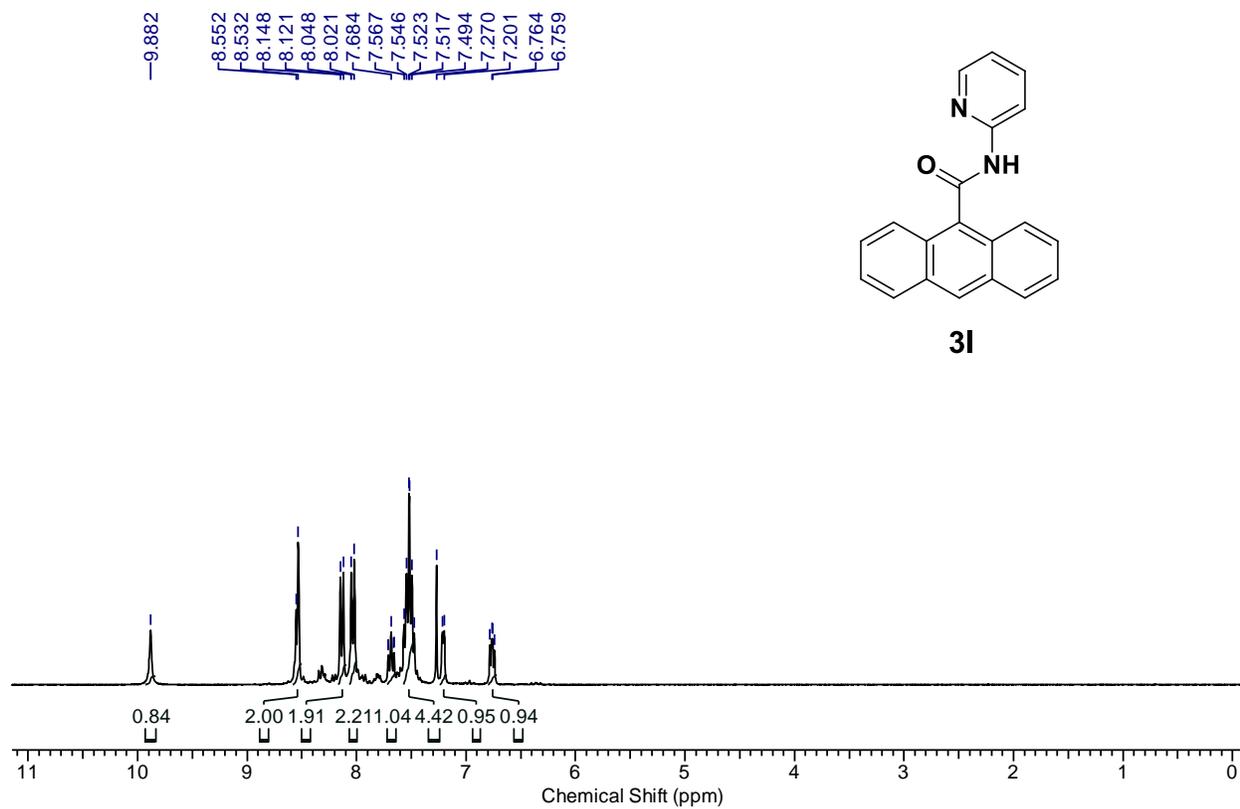


8.830
8.341
8.312
7.770
7.762
7.744
7.541
7.291
7.280
7.270
7.079
7.072
6.582
6.577
6.572
6.567

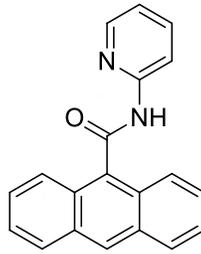
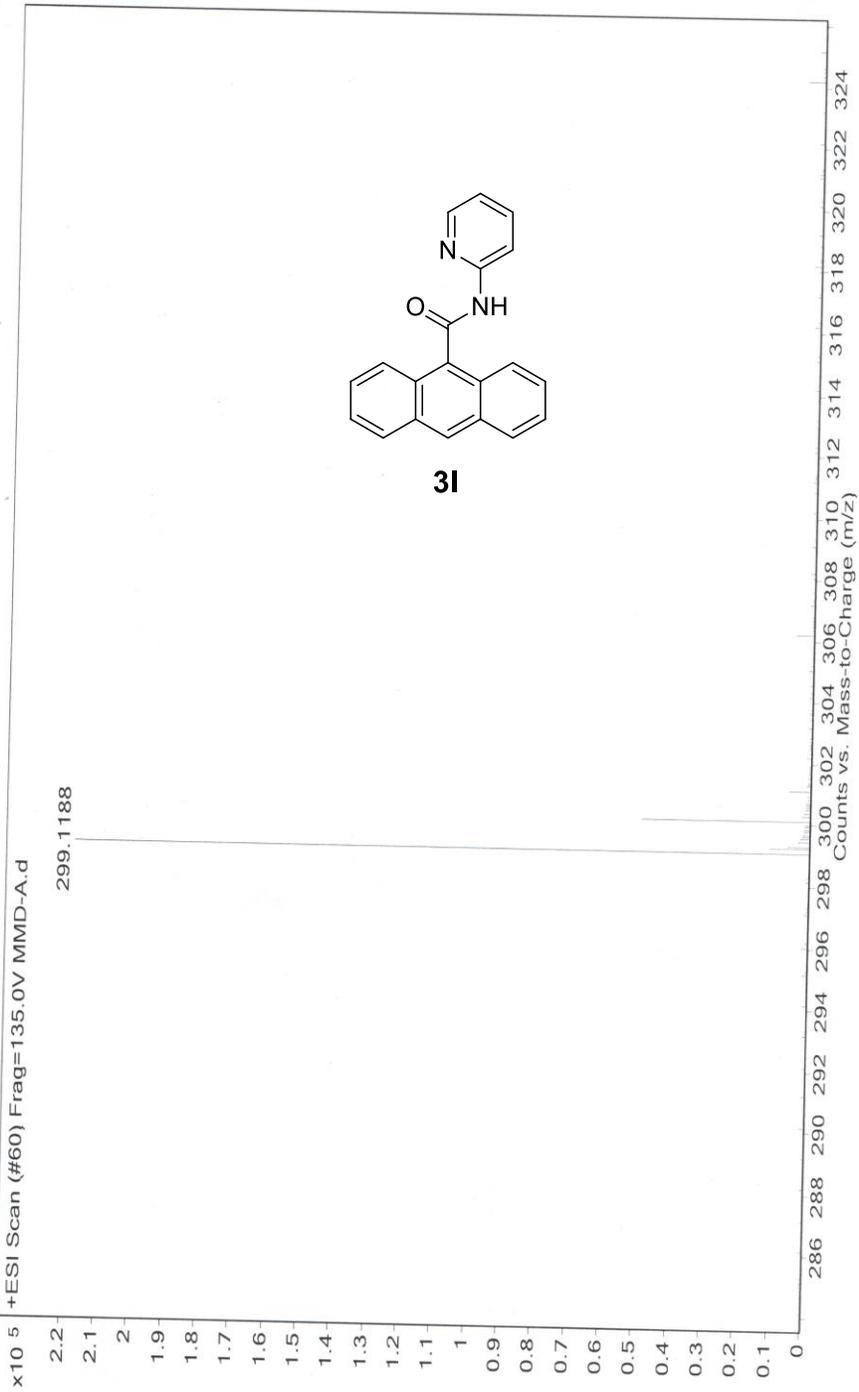


156.160
150.933
147.944
144.751
138.444
119.949
115.896
114.124
112.644
77.425
77.002
76.579





Sample Name MMD-A
Inj Vol -1
Data Filename MMD-A.d
Position Vial 1
InjPosition
ACQ Method
Instrument Name QTOF
SampleType Sample
Comment
User Name
IRM Calibration Status Success
Acquired Time 8/30/2018 4:45:27 PM



31