Supporting information

Metal-free Oxidative amidation of aldehydes with aminopyridines employing aqueous hydrogen peroxide

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1. General Information

All chemicals were purchased from Sigma-Aldrich, Alfa Aesar, Finar and Avra Synthesis, Pvt. Ltd. India and used as received. ACME silica gel (100–200 mesh) was used for column chromatography and thin-layer chromatography was performed on Merck-pre-coated silica gel 60-F_{254} plates. All the other chemicals and solvents were obtained from commercial sources and purified using standard methods.

^{1}H NMR spectra was recorded on Bruker 300 MHz spectrometer. ^{13}C NMR spectra were recorded on Bruker 75 MHz spectrometer. ^{1}H NMR chemical shifts are expressed in parts per million (δ) downfield from tetramethylsilane (with the $CHCl_3$ and DMSO peak around 7.26 ppm 2.5 ppm used as standard respectively). ^{13}C NMR chemical shifts are expressed in parts per million (δ) downfield from tetramethylsilane (with the central peak of $CHCl_3$ and DMSO around 77.2 ppm and 39.5 ppm used as standard respectively). All ^{13}C spectra were measured with complete proton decoupling. NMR coupling constants (J) are reported in Hertz (Hz), and splitting patterns are indicated as follows: br, broad; s, singlet; d, doublet; dd, doublet of doublet; ddd, doublet of doublet of doublet; dt, doublet of triplet; t, triplet; q, quartet; m, multiplet.

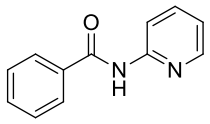
2. Experimental section:

(i) General Procedure for synthesis of substituted *N*-(pyridin-2-yl)benzamide

A mixture of benzaldehyde (0.106g, 1mmol), 2-aminopyridine (0.0941g, 1mmol) in water (2 mL) and Hydrogen peroxide (3 equiv.) was stirred at 80 °C for 4 h. Progress of the reaction was monitored by TLC. After the completion of the reaction, the mixture was extracted with ethyl acetate. The extract was washed with brine solution and dried over anhydrous Na_2SO_4. Removal of the solvent under vacuum afforded the crude product, which was purified by column chromatography using hexane/ethyl acetate mixture.

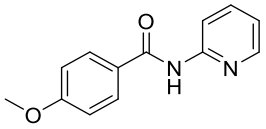
3. Spectroscopic data for the products:

1. *N*-(pyridin-2-yl)benzamide (3a)



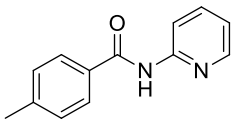
Isolated yield = 91%, 180 mg, white crystalline solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.83 (s, 1H), 8.41 (dt, *J* = 8.4, 0.9 Hz, 1H), 8.25 (m, 1H), 7.99 – 7.90 (m, 2H), 7.82 – 7.72 (m, 1H), 7.62 – 7.45 (m, 3H), 7.07 (m, 1H).^{13}C NMR (75 MHz, $CDCl_3$) δ 165.9, 151.7, 147.8, 138.5, 134.3, 132.2, 128.8, 127.3, 119.9, 114.3. Physical and spectral properties of this material were identical to those previously reported in literature.[1]

2. 4-methoxy-*N*-(pyridin-2-yl)benzamide (3b)



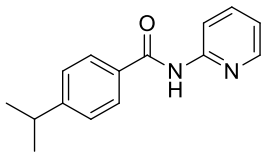
Isolated yield = 78%, 224 mg, white solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.84 (s, 1H), 8.31 (d, *J* = 8.4 Hz, 1H), 8.15 (s, 1H), 7.87 – 7.79 (m, 2H), 7.71 – 7.62 (m, 1H), 6.98-6.96 (m, 1H), 6.92 – 6.84 (m, 2H), 3.79 (s, 3H).^{13}C NMR (75 MHz, $CDCl_3$) δ 165.60, 162.72, 152.03, 147.67, 138.45, 129.37, 126.51, 119.59, 114.43, 113.90, 55.43.Physical and spectral properties of this material were identical to those previously reported in literature.[1]

3. 4-methyl-*N*-(pyridin-2-yl)benzamide (3c)



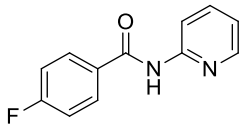
Isolated yield = 76%, 161 mg, white solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.65 (s, 1H), 8.39 (m, 1H), 8.28 (m, 1H), 7.87 – 7.80 (m, 2H), 7.79 – 7.71 (m, 1H), 7.30 (d, *J* = 7.9 Hz, 2H), 7.06 (m, 1H), 2.43 (s, 3H).^{13}C NMR (75 MHz, $CDCl_3$) δ 166.0, 151.9, 147.8, 142.7, 138.4, 131.5, 129.4, 127.4, 119.7, 114.4, 21.5. Physical and spectral properties of this material were identical to those previously reported in literature.[1]

4. 4-isopropyl-*N*-(pyridin-2-yl)benzamide (3d)



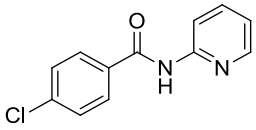
Isolated yield = 82%, 196 mg, white solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.69 (s, 1H), 8.40 (d, *J* = 8.4 Hz, 1H), 8.27 (dd, *J* = 4.9, 1.0 Hz, 1H), 7.92 – 7.82 (m, 2H), 7.82 – 7.68 (m, 1H), 7.35 (d, *J* = 8.5 Hz, 2H), 7.06 (m, 1H), 2.98 (m, 1H), 1.29 (d, *J* = 6.9 Hz, 6H).^{13}C NMR (75 MHz, $CDCl_3$) δ 166.1, 153.5, 151.9, 147.8, 138.5, 127.5, 126.8, 119.7, 114.4, 34.1, 23.7. HRMS (ESI): calcd for $C_{15}H_{16}N_2O$, m/z 263.2901($[M+Na]^+$); found, m/z 263.1180. Physical and spectral properties of this material were identical to those previously reported in literature.[7]

5. 4-fluoro-*N*-(pyridin-2-yl)benzamide (3e)



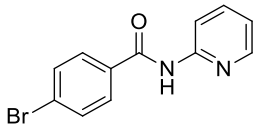
Isolated yield = 80%, 172 mg, white solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.78 (s, 1H), 8.37 (dt, *J* = 8.4, 0.9 Hz, 1H), 8.28 – 8.15 (m, 1H), 7.95 (m, 2H), 7.77 (m, 1H), 7.22 – 6.98 (m, 3H).^{13}C NMR (75 MHz, $CDCl_3$) δ 166.8, 165.1, 163.4, 151.8, 147.7, 138.6, 130.6, 130.5, 129.9, 129.8, 119.9, 116.0, 115.7, 114.5. HRMS (ESI): calcd for $C_{12}H_9FN_2O$ m/z 239.2008 ($[M+Na]^+$); found, m/z 239.0441. Physical and spectral properties of this material were identical to those previously reported in literature.[7]

6. 4-chloro-*N*-(pyridin-2-yl)benzamide (3f)



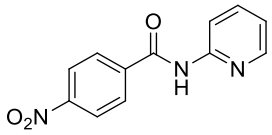
Isolated yield = 85%, 197 mg, white crystalline solid. ^{1}HNMR (300 MHz, $CDCl_3$) δ 8.88 (s, 1H), 8.42 – 8.29 (m, 1H), 8.23(d, *J* = 3.2 Hz, 1H), 7.93 – 7.82 (m, 2H), 7.81 – 7.69 (m, 1H), 7.51 – 7.38 (m, 2H), 7.08 (dd, *J* = 6.4, 5.0 Hz, 1H).^{13}C NMR (75 MHz, $CDCl_3$) δ 165.2, 151.7, 147.7, 138.6, 138.4, 132.8, 128.9, 120.0, 114.7.Physical and spectral properties of this material were identical to those previously reported in literature.[1]

7. 4-bromo-*N*-(pyridin-2-yl)benzamide (3g)



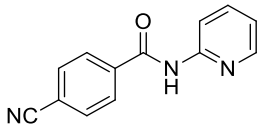
Isolated yield = 82%, 227 mg, white crystalline solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.86 (s, 1H), 8.37 (dd, *J* = 8.4, 0.8 Hz, 1H), 8.26 – 8.12 (m, 1H), 7.86 – 7.69 (m, 3H), 7.67 – 7.50 (m, 2H), 7.08 (m, 1H).^{13}C NMR (75 MHz, $CDCl_3$) δ 165.3, 151.7, 147.7, 138.6, 133.3, 131.9, 129.1, 127.0, 120.0, 114.6. Physical and spectral properties of this material were identical to those previously reported in literature.[1]

8. 4-nitro-*N*-(pyridin-2-yl)benzamide (3h)



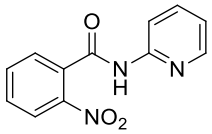
Isolated yield = 49%, 119 mg, white solid. ^{1}H NMR (300 MHz, $CDCl_3$+ DMSO) δ 10.61 (s, 1H), 8.32 (m, 6H), 7.80 (d, *J* = 7.2 Hz, 1H), 7.13 (s, 1H).^{13}C NMR (75 MHz, $CDCl_3$ + DMSO) δ 164.4, 151.8, 149.2, 147.8, 139.8, 138.0, 129.5, 123.2, 120.0, 114.8. Physical and spectral properties of this material were identical to those previously reported in literature.[2]

9. 4-cyano-*N*-(pyridin-2-yl)benzamide (3i)



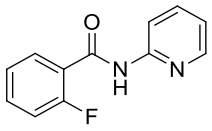
Isolated yield = 48%, 107 mg, white solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.84 (s, 1H), 8.37 (dd, *J* = 8.4, 0.8 Hz, 1H), 8.28 (m, 1H), 8.09 – 7.98 (m, 2H), 7.86 – 7.72 (m, 3H), 7.12 (m, 1H).^{13}C NMR (75 MHz, $CDCl_3$+ DMSO) δ 164.3, 151.4, 147.8, 138.4, 138.3, 132.4, 128.3, 120.3, 117.9, 115.4, 114.7. HRMS (ESI): calcd for $C_{13}H_9N_3O$ m/z 224.238 ($[M+H]^+$); found, m/z 224.0668. Physical and spectral properties of this material were identical to those previously reported in literature.[7]

10. 2-nitro-*N*-(pyridin-2-yl)benzamide (3j)



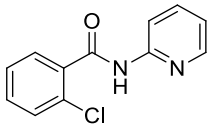
Isolated yield = 42%, 102 mg, white solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.79 (s, 1H), 8.32 (d, *J* = 8.1 Hz, 1H), 8.10 (dd, *J* = 8.0, 0.7 Hz, 1H), 7.81 – 7.56 (m, 5H), 7.00 – 6.92 (m, 1H).^{13}C NMR (75 MHz, $CDCl_3$) δ 165.3, 151.6, 146.6, 146.2, 138.9, 134.0, 132.7, 130.7, 128.8, 124.5, 120.0, 115.4. HRMS (ESI): calcd for $C_{12}H_9N_3O_3$ m/z 266.2079 ($[M+Na]^+$); found, m/z 266.0501.

11. 2-fluoro-*N*-(pyridin-2-yl)benzamide (3k)



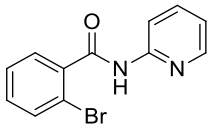
Isolated yield = 78%, 168 mg, white solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.11 (d, *J* = 11.4 Hz, 1H), 8.44 – 8.23 (m, 2H), 8.15 (m, 1H), 7.83 – 7.68 (m, 1H), 7.61 – 7.46 (m, 1H), 7.32 (m, 1H), 7.20 (m, 1H), 7.09 (m, 1H).^{13}C NMR (75 MHz, $CDCl_3$) δ 162.0, 161.8, 161.7, 158.7, 151.4, 148.0, 138.4, 134.1, 133.9, 131.9, 131.8, 125.0, 124.9, 121.5, 121.3, 120.1, 116.5, 116.2, 114.6. Physical and spectral properties of this material were identical to those previously reported in literature.[1]

12. 2-chloro-*N*-(pyridin-2-yl)benzamide (3l)



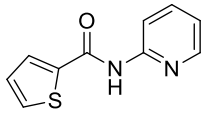
Isolated yield = 80%, 185 mg, white crystalline solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.43 (s, 1H), 8.39 (d, *J* = 8.4 Hz, 1H), 7.94 (m, 1H), 7.81 – 7.66 (m, 2H), 7.50 – 7.30 (m, 3H), 7.00 (m, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 165.7, 151.7, 147.2, 138.6, 135.8, 131.5, 130.3, 129.5, 127.1, 119.9, 114.7. Physical and spectral properties of this material were identical to those previously reported in literature.[1]

13. 2-bromo-*N*-(pyridin-2-yl)benzamide (3m)



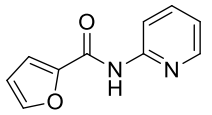
Isolated yield = 81%, 224 mg, white crystalline solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.32 (s, 1H), 8.38 (d, *J* = 8.3 Hz, 1H), 7.97 – 7.88 (m, 1H), 7.80 – 7.70 (m, 1H), 7.62 (m, 2H), 7.45 – 7.31 (m, 2H), 6.99 (m, 1H).^{13}C NMR (75 MHz, $CDCl_3$) δ 166.7, 151.7, 147.1, 138.6, 138.2, 133.4, 131.4, 129.1, 127.6, 119.9, 119.6, 114.8. Physical and spectral properties of this material were identical to those previously reported in literature.[1]

14. *N*-(pyridin-2-yl)thiophene-2-carboxamide(3n)



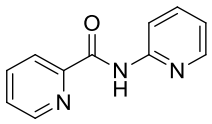
Isolated yield = 89%, 181 mg, white solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.59 (s, 1H), 8.37 – 8.25 (m, 2H), 7.81 – 7.71 (m, 1H), 7.68 (dd, *J* = 3.8, 1.0 Hz, 1H), 7.59 (dd, *J* = 5.0, 1.0 Hz, 1H), 7.18 – 7.11 (m, 1H), 7.11 – 7.03 (m, 1H).^{13}C NMR (75 MHz, $CDCl_3$) δ 160.3, 151.5, 147.7, 139.1, 138.6, 131.6, 129.0, 127.9, 119.9, 114.6. Physical and spectral properties of this material were identical to those previously reported in literature.[2]

15. *N*-(pyridin-2-yl)furan-2-carboxamide (3o)



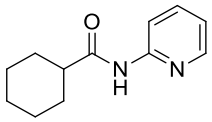
Isolated yield = 80%, 150 mg, white solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.80 (s, 1H), 8.46 – 8.28 (m, 2H), 7.84 – 7.71 (m, 1H), 7.62 – 7.52 (m, 1H), 7.35 – 7.25 (m, 1H), 7.19 – 7.03 (m, 1H), 6.58 (dd, *J* = 3.5, 1.7 Hz, 1H).^{13}C NMR (75 MHz, $CDCl_3$) δ 156.2, 151.1, 148.0, 147.3, 144.7, 138.4, 119.9, 115.9, 114.2, 112.6. Physical and spectral properties of this material were identical to those previously reported in literature.[1]

16. *N*-(pyridin-2-yl)picolinamide (3p)



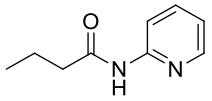
Isolated yield = 84%, 167 mg, white crystalline solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.54 (s, 1H), 8.40 (d, *J* = 8.4 Hz, 1H), 7.76 – 7.66 (m, 1H), 7.65 – 7.55 (m, 1H), 7.46 (s, 1H), 7.41 – 7.28 (m, 3H), 6.86 (dd, *J* = 6.5, 5.7 Hz, 1H).^{13}C NMR (75 MHz, $CDCl_3$) δ 162.5, 151.1, 149.2, 148.2, 138.2, 137.5, 126.7, 122.4, 119.8, 113.9. Physical and spectral properties of this material were identical to those previously reported in literature.[3]

17. *N*-(pyridin-2-yl)cyclohexanecarboxamide (3q)



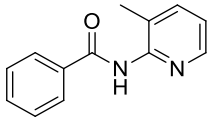
Isolated yield = 76%, 155 mg, white solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.26 (m, 3H), 7.70 (m, 1H), 7.03 (m, 1H), 2.26 (tt, *J* = 11.7, 3.5 Hz, 1H), 1.96 (d, *J* = 13.7 Hz, 2H), 1.83 (dd, *J* = 9.3, 3.3 Hz, 2H), 1.74 – 1.65 (m, 1H), 1.54 (m, 2H), 1.37 – 1.20 (m, 3H).^{13}C NMR (75 MHz, $CDCl_3$) δ 175.2, 152.0, 147.4, 138.5, 119.5, 114.5, 46.2, 29.4, 25.5. HRMS (ESI): calcd for $C_{12}H_{16}N_2O$ m/z 227.2566 ($[M+Na]^+$); found, m/z 227.114.

18. *N*-(pyridin-2-yl)butyramide (3r)



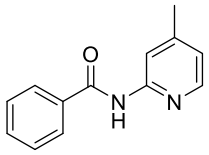
Isolated yield = 42%, 68 mg, colourless oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.79 (s, 1H), 8.37 – 8.17 (m, 2H), 7.78 – 7.67 (m, 1H), 7.04 (m, 1H), 2.37 (t, *J* = 7.4 Hz, 2H), 1.88 – 1.67 (m, 2H), 0.99 (t, *J* = 7.4 Hz, 3H).^{13}C NMR (75 MHz, $CDCl_3$) δ172.0, 151.8, 147.5, 138.5, 119.6, 114.4, 39.5, 18.8, 13.7. Physical and spectral properties of this material were identical to those previously reported in literature.[2]

19. *N*-(3-methylpyridin-2-yl)benzamide (3s)



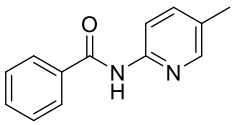
Isolated yield = 68%, 144 mg, whites solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.77 (s, 1H), 8.30 (d, *J* = 8.5 Hz, 1H), 8.06 (s, 1H), 7.97 – 7.87 (m, 2H), 7.63 – 7.45 (m, 4H), 2.30 (s, 3H).^{13}C NMR (75 MHz, $CDCl_3$) δ 165.7, 149.4, 147.7, 139.1, 134.4, 132.1, 129.3, 128.8, 127.2, 113.7, 17.8. Physical and spectral properties of this material were identical to those previously reported in literature.[4]

20. *N*-(4-methylpyridin-2-yl)benzamide (3t)



Isolated yield = 86%, 182 mg, white crystalline solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.69 (s, 1H), 8.25 (d, *J* = 0.7 Hz, 1H), 8.12 (d, *J* = 5.1 Hz, 1H), 7.97 – 7.83 (m, 2H), 7.61 – 7.41 (m, 3H), 6.94 – 6.83 (m, 1H), 2.41 (s, 3H).^{13}C NMR (75 MHz, $CDCl_3$) δ 166.2, 151.9, 150.0, 147.3, 134.6, 132.0, 128.7, 127.4, 121.0, 115.0,21.4. Physical and spectral properties of this material were identical to those previously reported in literature.[1]

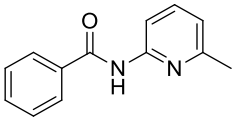
21. *N*-(5-methylpyridin-2-yl)benzamide (3u)



Isolated yield = 90%, 190 mg, white crystalline solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.97 (s, 1H), 8.30 (d, *J* = 8.5 Hz, 1H), 8.05 – 7.88 (m, 3H), 7.62 – 7.45 (m, 4H), 2.28 (s,

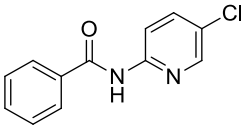
3H).^{13}C NMR (75 MHz, $CDCl_3$) δ 166.2, 149.7, 147.7, 139.0, 134.7, 131.9, 129.1, 128.7, 127.4, 113.9, 17.8. Physical and spectral properties of this material were identical to those previously reported in literature.[1]

22. *N*-(6-methylpyridin-2-yl)benzamide (3v)



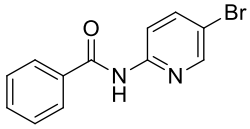
Isolated yield = 92%, 195 mg, white solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ8.58 (s, 1H), 8.20 (d, *J* = 8.3 Hz, 1H), 7.98 – 7.90 (m, 2H), 7.71 – 7.62 (m, 1H), 7.61 – 7.43 (m, 3H), 6.94 (d, *J* = 7.5 Hz, 1H), 2.47 (s, 3H).^{13}C NMR (75 MHz, $CDCl_3$) δ 165.9, 156.9, 151.0, 138.7, 134.4, 132.1, 128.7, 127.3, 119.4, 111.1, 23.8. Physical and spectral properties of this material were identical to those previously reported in literature.[1]

23. *N*-(5-chloropyridin-2-yl)benzamide (3w)



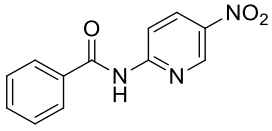
Isolated yield = 87%, 201 mg, white crystalline solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.94 (s, 1H), 8.39 (d, *J* = 8.9 Hz, 1H), 8.11 (d, *J* = 2.5 Hz, 1H), 7.99 – 7.86 (m, 2H), 7.70 (dd, *J* = 8.9, 2.6 Hz, 1H), 7.65 – 7.44 (m, 3H).^{13}C NMR (75 MHz, $CDCl_3$) δ 166.1, 150.1, 146.4, 138.1, 134.1, 132.4, 128.8, 127.4, 126.7, 115.1. Physical and spectral properties of this material were identical to those previously reported in literature.[2]

24. *N*-(5-bromopyridin-2-yl)benzamide (3x)



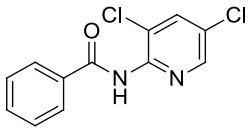
Isolated yield = 93%, 257 mg, white crystalline solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ8.64 (s, 1H), 8.35 (dd, *J* = 9.8, 1.1 Hz, 2H), 7.93 – 7.83 (m, 3H), 7.63 – 7.56 (m, 1H), 7.54 – 7.48 (m, 2H).^{13}C NMR (75 MHz, $CDCl_3$) δ 166.0, 150.4, 148.7, 140.9, 134.1, 132.4, 128.8, 127.3, 115.5, 114.7. Physical and spectral properties of this material were identical to those previously reported in literature.[5]

25. *N*-(5-nitropyridin-2-yl)benzamide (3y)



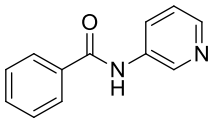
Isolated yield = 55%, 133 mg, white solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.16 (dd, *J* = 2.5, 0.7 Hz, 1H), 8.97 (s, 1H), 8.62 – 8.50 (m, 2H), 7.99 – 7.88 (m, 2H), 7.63 (dd, *J* = 5.0, 3.6 Hz, 1H), 7.58 – 7.48 (m, 2H).^{13}C NMR (75 MHz, $CDCl_3$+DMSO) δ 164.3, 151.4, 147.8, 138.4, 138.3, 132.4, 128.3, 120.3, 117.9, 115.4, 114.7. Physical and spectral properties of this material were identical to those previously reported in literature.[5]

26. *N*-(3,5-dichloropyridin-2-yl)benzamide (3z)



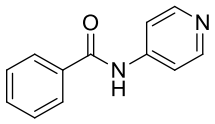
Isolated yield = 82%, 218 mg, white crystalline solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.39 (d, *J* = 2.3 Hz, 2H), 7.97 – 7.89 (m, 2H), 7.80 (d, *J* = 2.3 Hz, 1H), 7.60 (m, 1H), 7.56 – 7.46 (m, 2H).^{13}C NMR (75 MHz, $CDCl_3$) δ 164.9, 146.3, 145.6, 137.6, 133.7, 132.6, 128.9, 128.1, 127.6, 123.3. HRMS (ESI): calcd for $C_{12}H_8Cl_2N_2O$ m/z 289.1323 ($[M+Na]^+$); found, m/z 288.9961.

27. *N*-(pyridin-3-yl)benzamide (3aa)



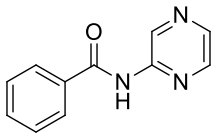
Isolated yield = 78%, 154 mg, white crystalline solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.67 (d, *J* = 2.5 Hz, 1H), 8.57 – 8.23 (m, 3H), 8.05 – 7.85 (m, 2H), 7.68 – 7.44 (m, 3H), 7.31 (dd, *J* = 8.3, 4.8 Hz, 1H).^{13}C NMR (75 MHz, $CDCl_3$) δ 167.1, 145.0, 141.9, 135.5, 134.2, 132.1, 128.6, 128.3, 127.4, 123.8. Physical and spectral properties of this material were identical to those previously reported in literature.[6]

28. *N*-(pyridin-4-yl)benzamide (3ab)

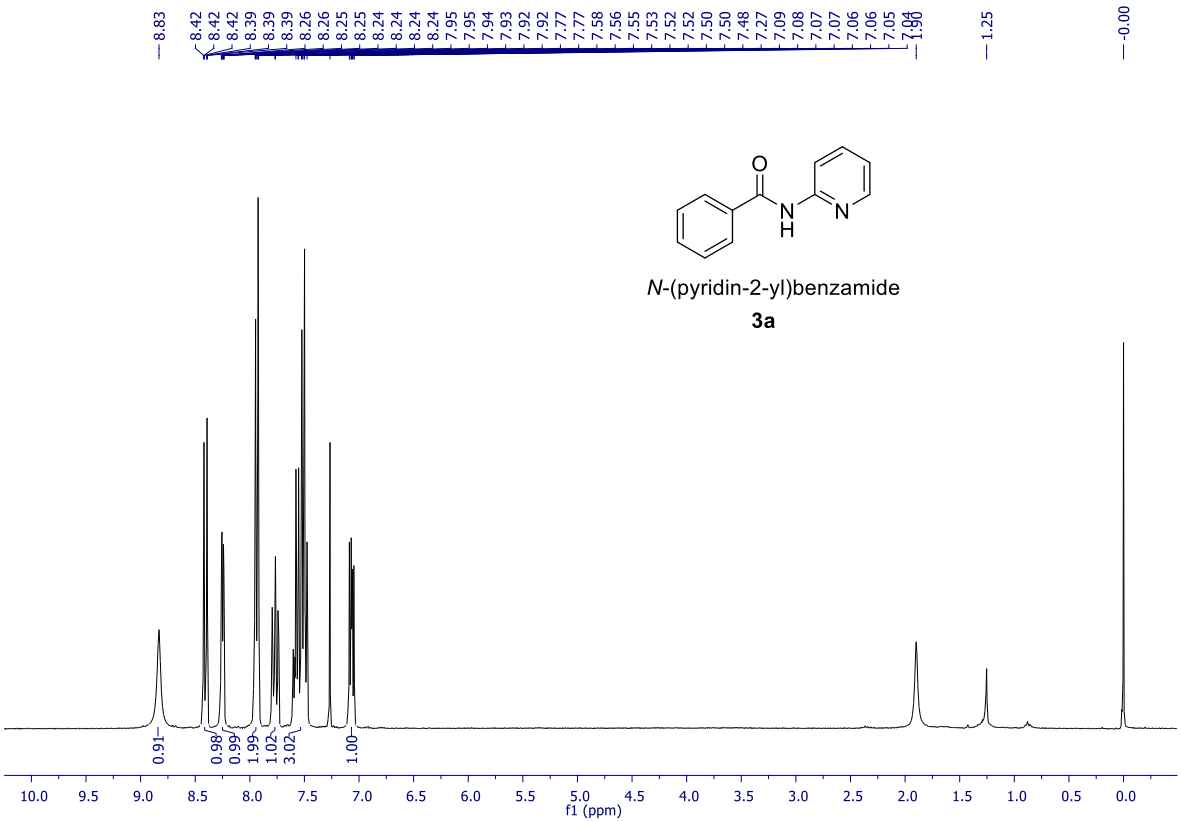


Isolated yield = 79%, 156 mg, white crystalline solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.56 (d, *J* = 6.2 Hz, 2H), 8.01 (s, 1H), 7.92 – 7.85 (m, 2H), 7.62 (dd, *J* = 5.0, 1.6 Hz, 3H), 7.53 (d, *J* = 8.0 Hz, 2H).^{13}C NMR (75 MHz, $CDCl_3$+DMSO) δ 165.9, 149.1, 145.2, 133.6, 130.8, 127.2, 126.8, 113.2. Physical and spectral properties of this material were identical to those previously reported in literature.[6]

29. *N*-(pyrazin-2-yl)benzamide (3ac)

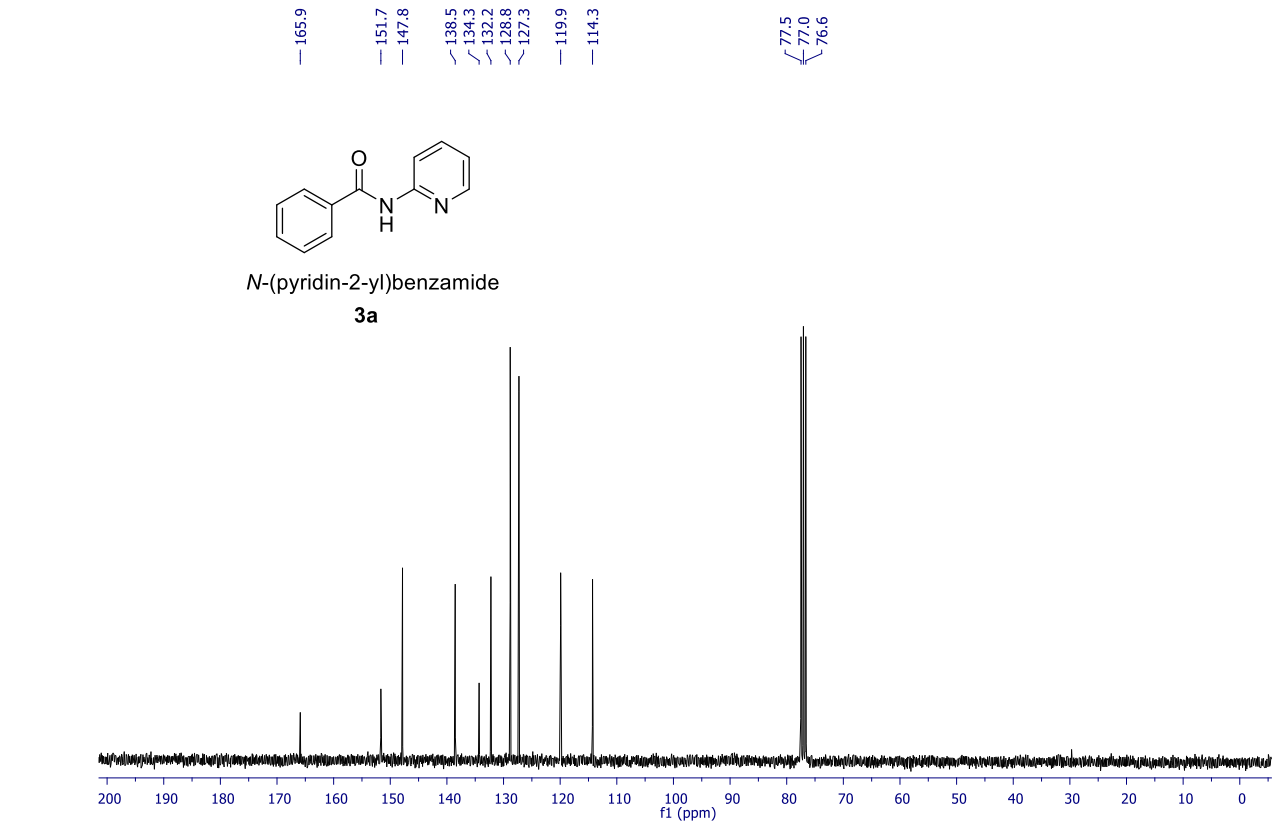


Isolated yield = 62%, 123 mg, white crystalline solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.73 (d, *J* = 1.5 Hz, 1H), 8.58 (s, 1H), 8.39 (d, *J* = 2.6 Hz, 1H), 8.27 (dd, *J* = 2.5, 1.6 Hz, 1H), 7.99 – 7.91 (m, 2H), 7.65 – 7.58 (m, 1H), 7.57 – 7.50 (m, 2H).^{13}C NMR (75 MHz, $CDCl_3$) δ ^{13}C NMR (75 MHz, $CDCl_3$) δ 165.6, 148.4, 142.1, 140.4, 137.3, 133.4, 132.7, 129.0, 127.4. Physical and spectral properties of this material were identical to those previously reported in literature.[2]



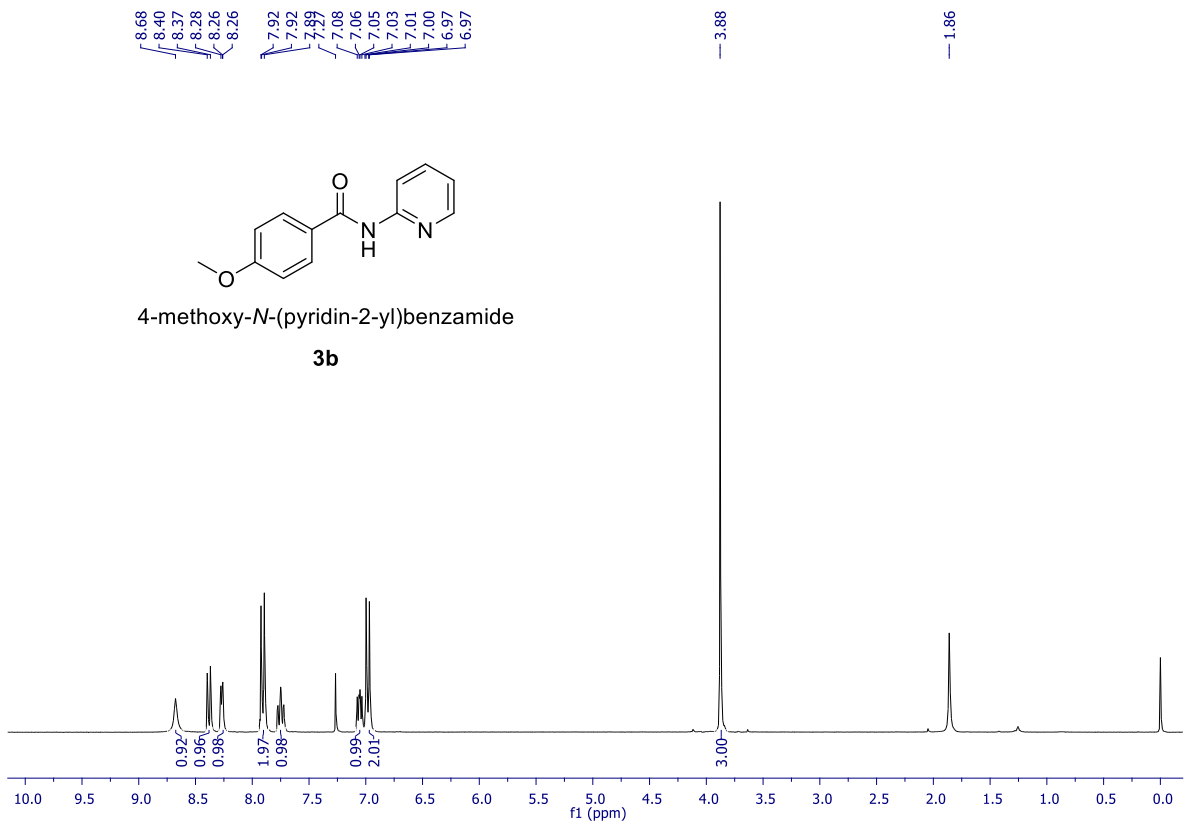
N-(pyridin-2-yl)benzamide

3a



N-(pyridin-2-yl)benzamide

3a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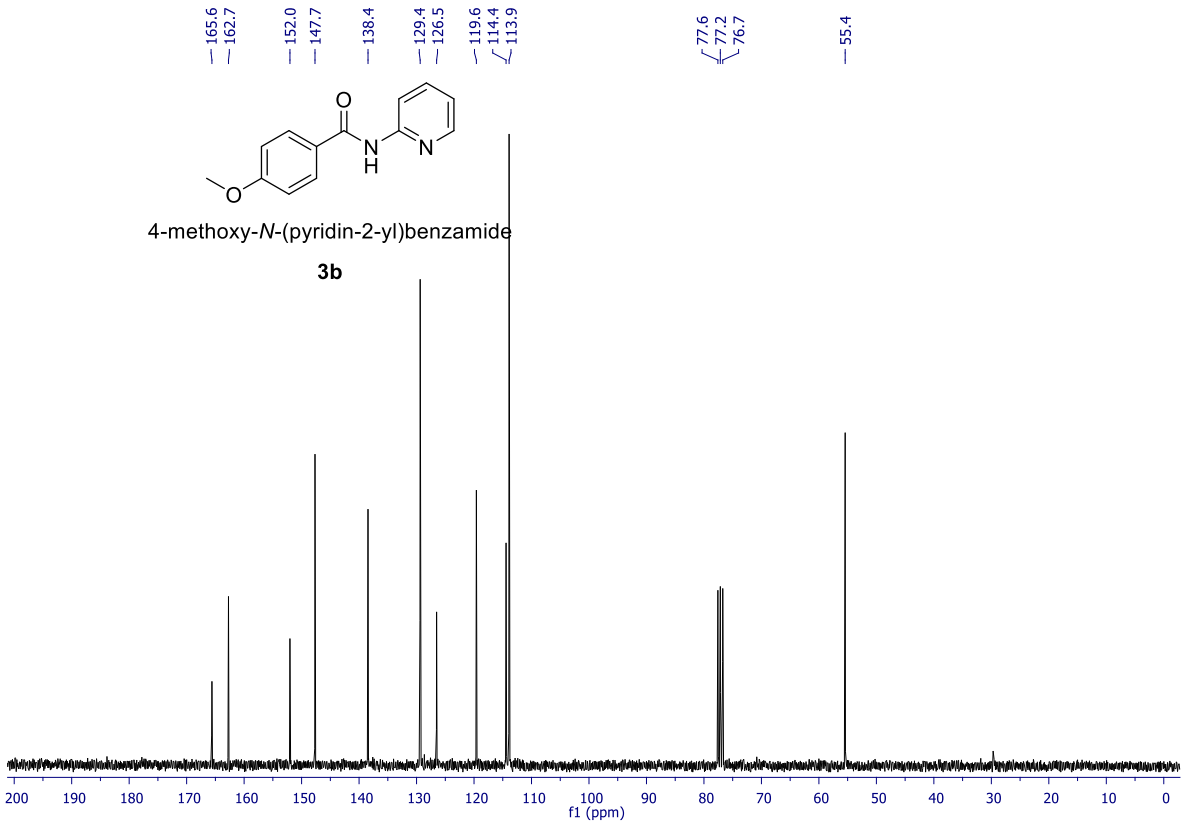
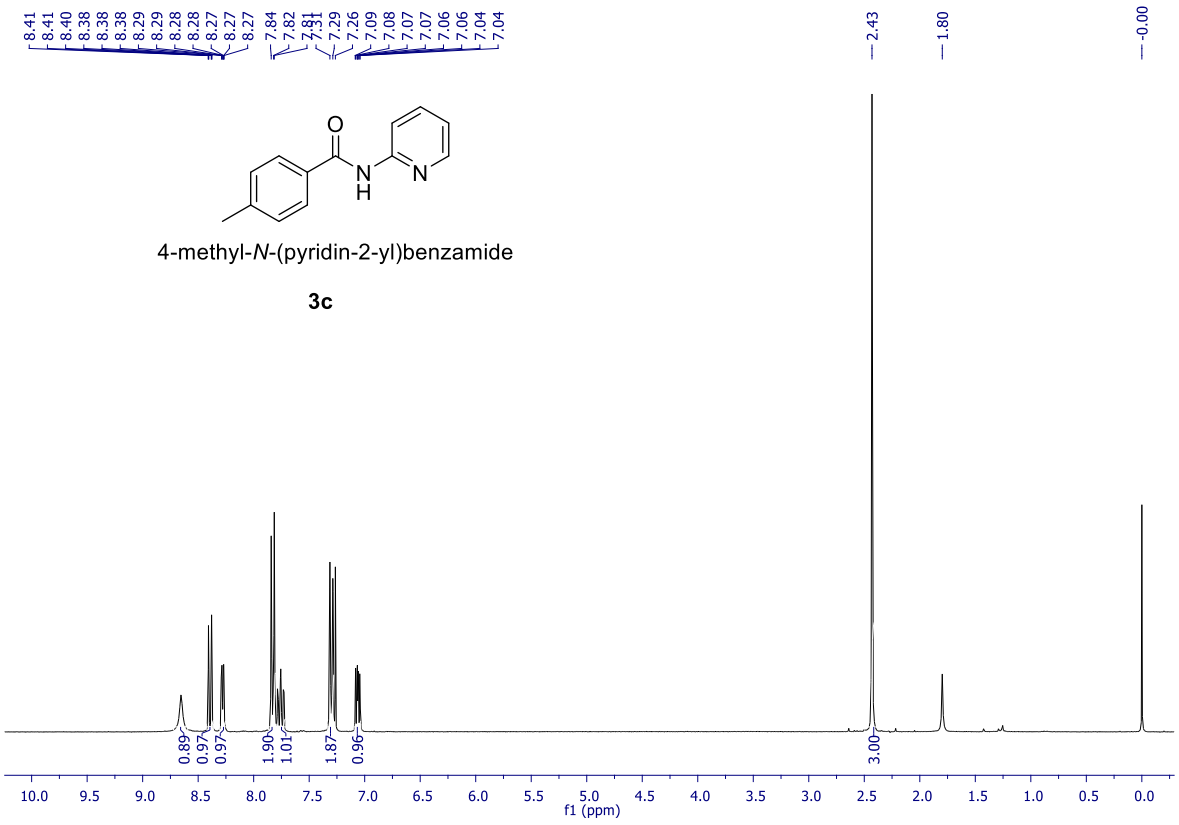
8.40
8.37
8.28
8.26
8.26
7.92
7.92
7.89
7.27
7.08
7.06
7.05
7.03
7.01
7.00
6.97
6.97
3.88
1.86
O
N
H
N
O
4-methoxy-N-(pyridin-2-yl)benzamide
3b
0.92
0.96
0.98
1.97
0.98
0.99
2.01
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


165.6
162.7
152.0
147.7
138.4
129.4
126.5
119.6
114.4
113.9
77.6
77.2
76.7
55.4
O
N
H
N
O
4-methoxy-N-(pyridin-2-yl)benzamide
3b
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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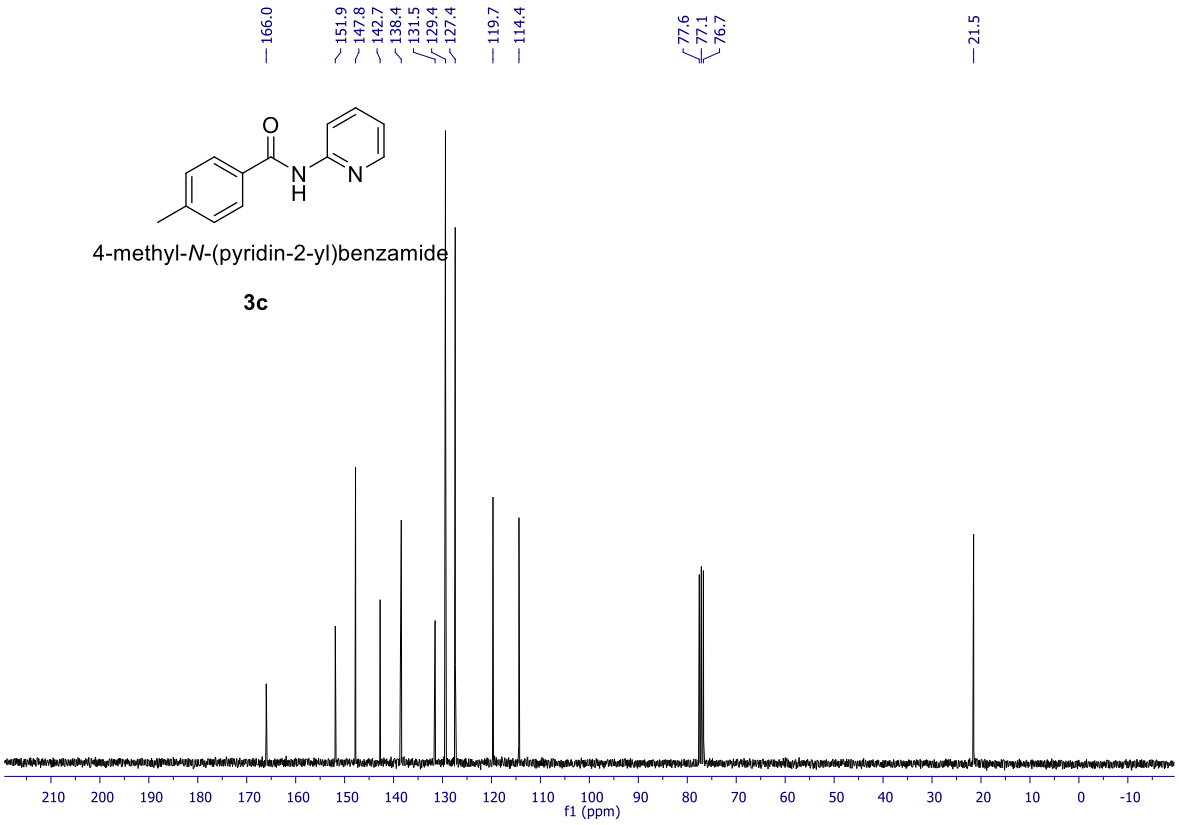
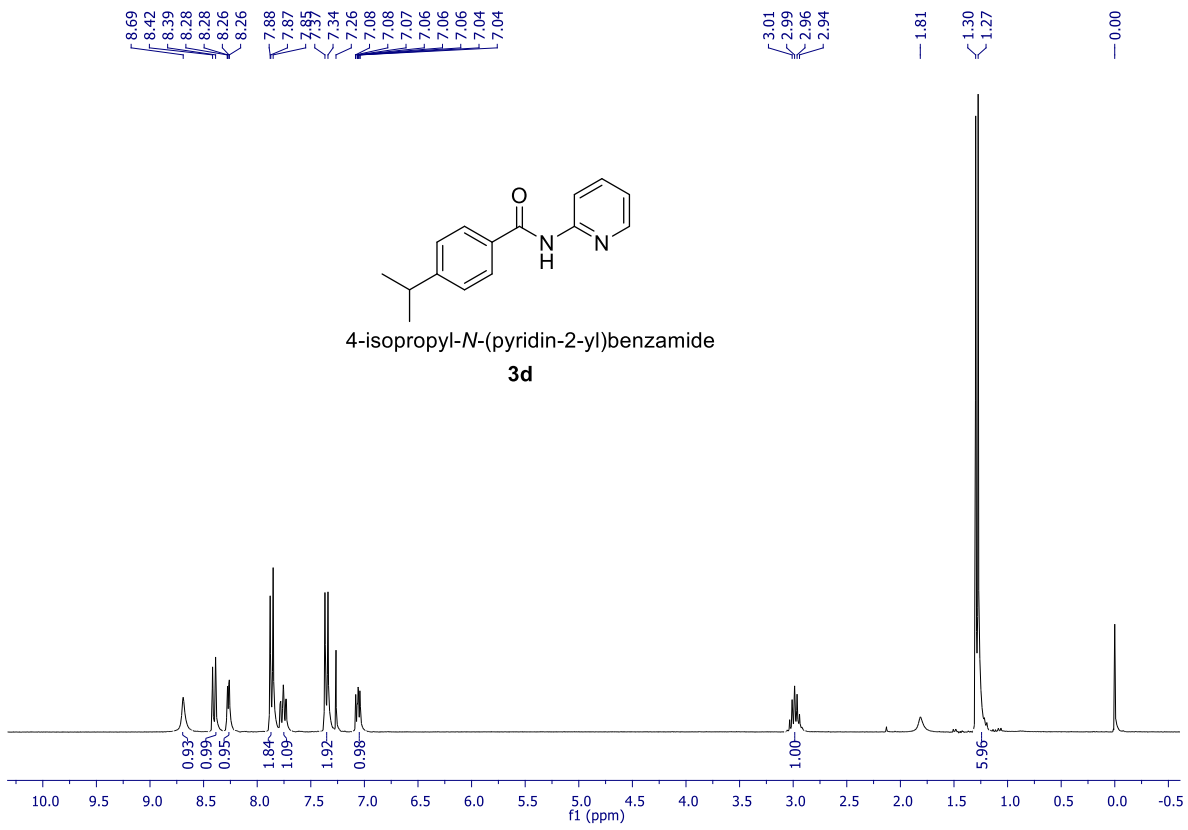
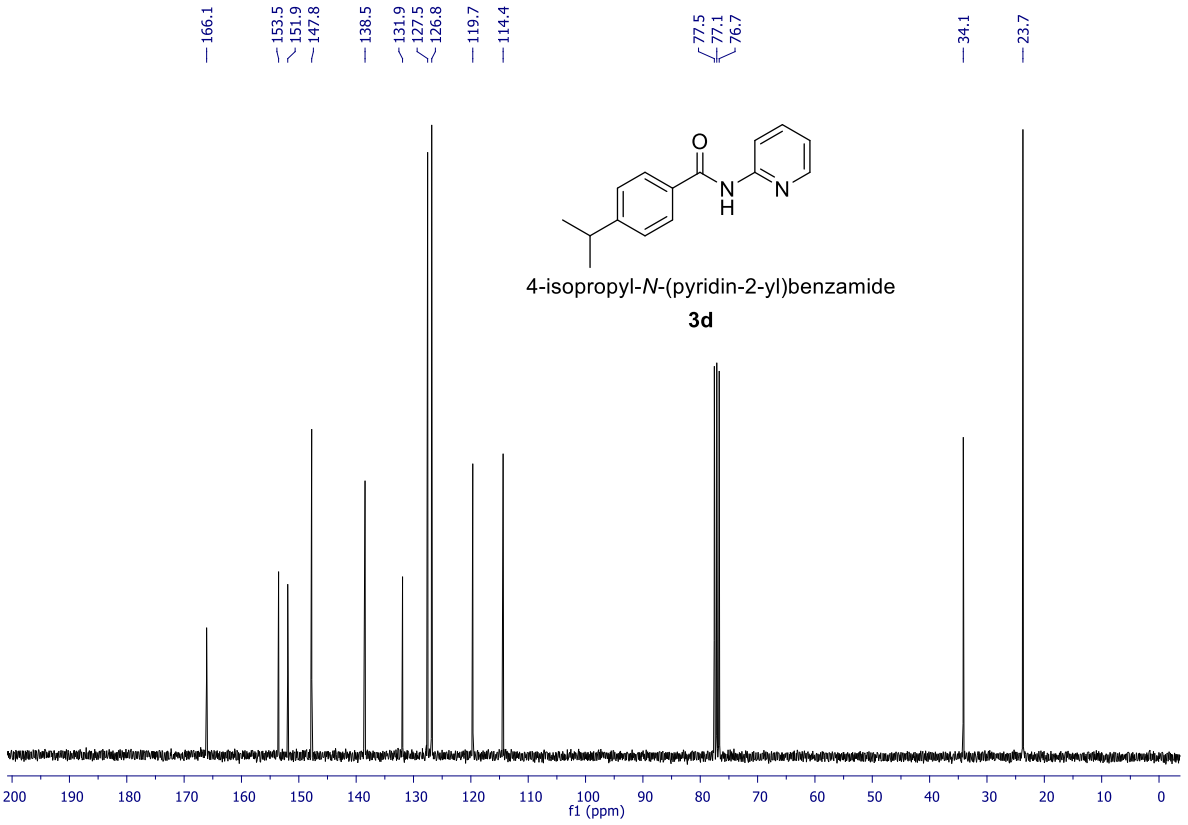
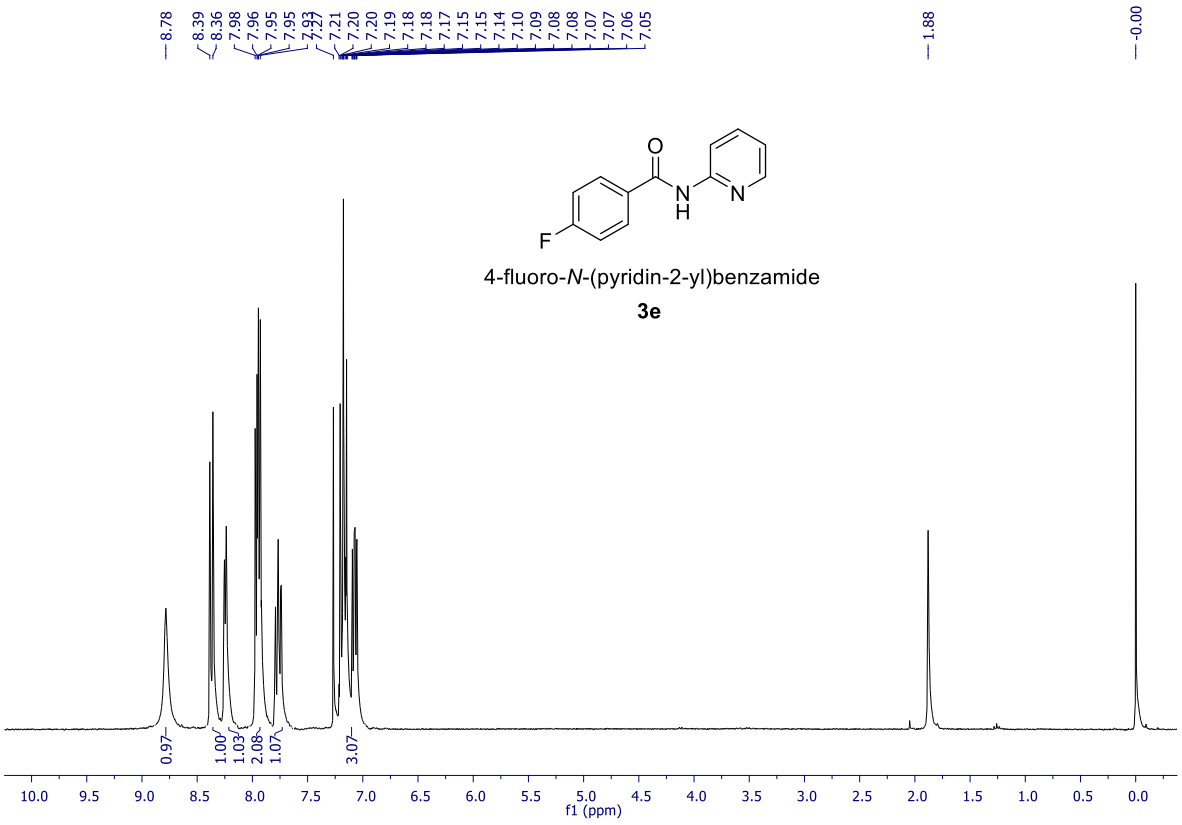
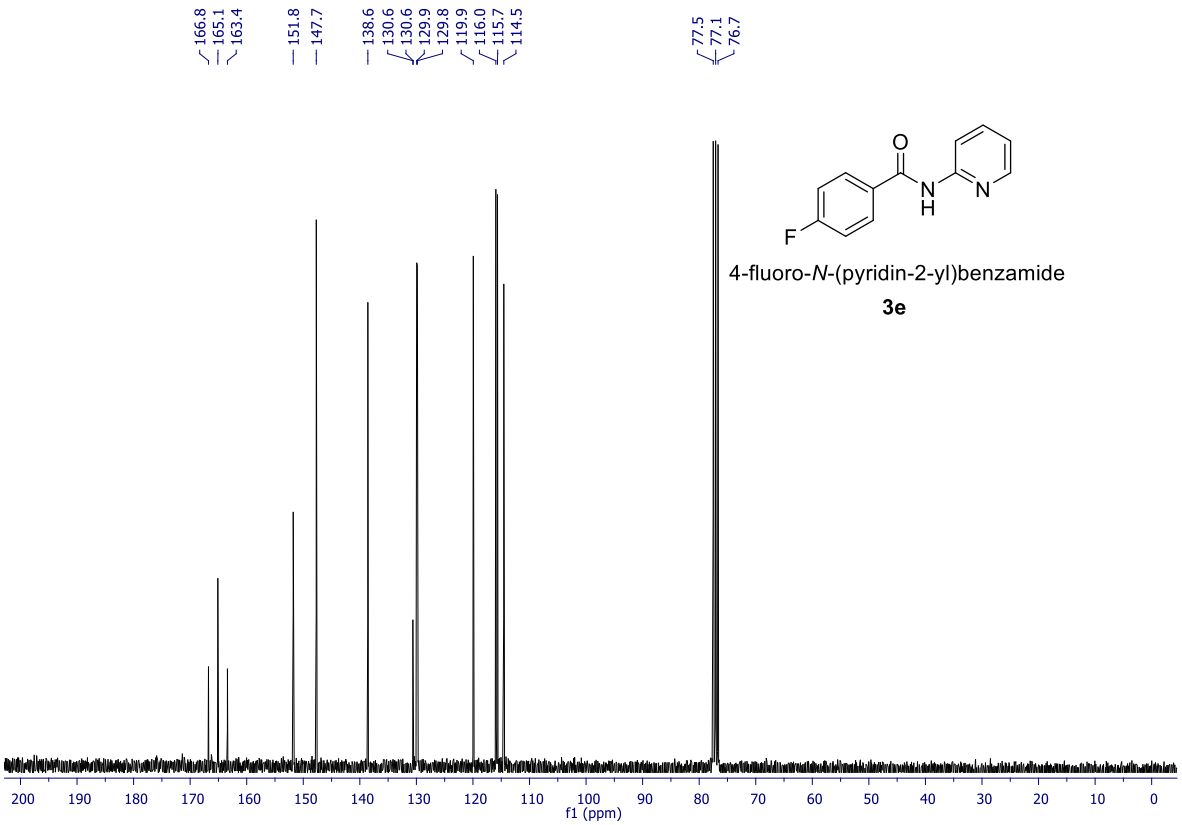
8.41
8.40
8.38
8.38
8.38
8.29
8.29
8.28
8.28
8.27
8.27
8.27
7.84
7.82
7.81
7.31
7.29
7.26
7.09
7.08
7.07
7.07
7.06
7.06
7.04
7.04
2.43
1.80
-0.00
O
N
H
N
4-methyl-N-(pyridin-2-yl)benzamide
3c
0.89
0.97
0.97
1.90
1.01
1.87
0.96
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

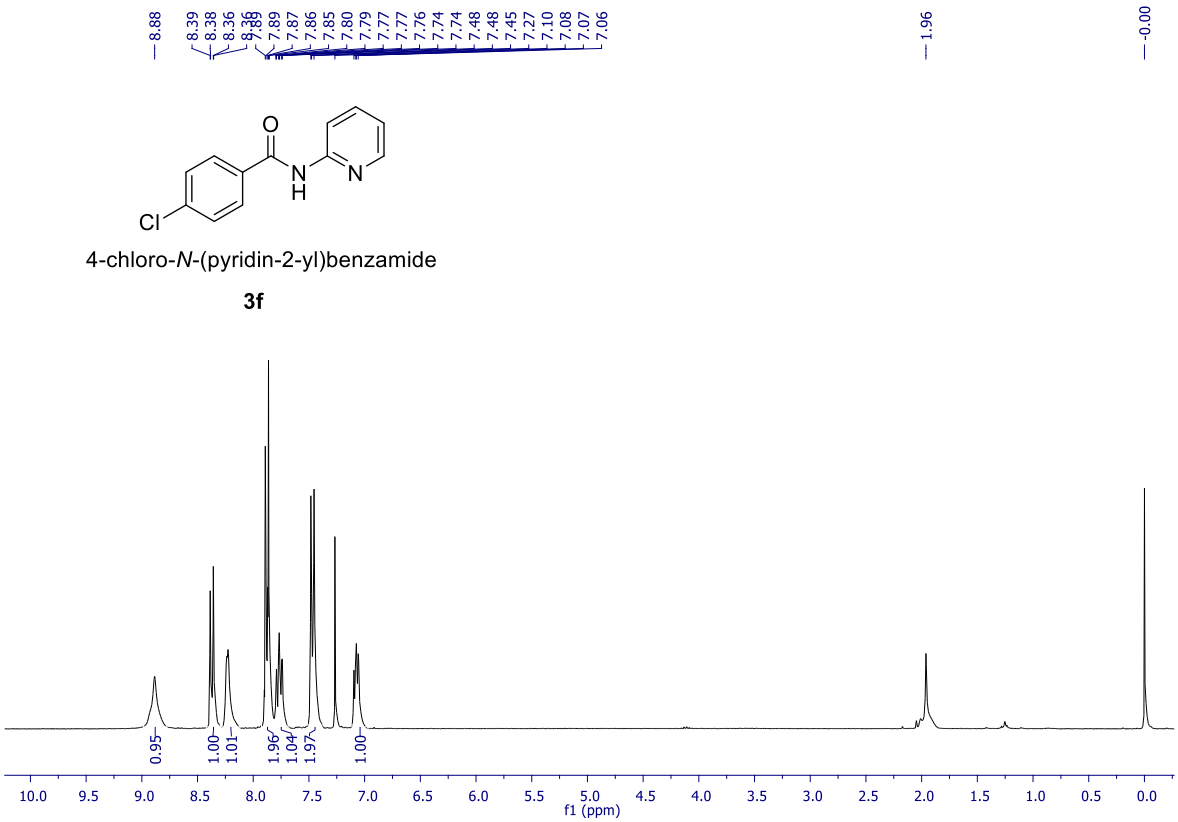

166.0
151.9
147.8
142.7
138.4
131.5
129.4
127.4
119.7
114.4
77.6
77.1
76.7
21.5
O
N
H
N
4-methyl-N-(pyridin-2-yl)benzamide
3c
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


8.69
8.42
8.39
8.28
8.28
8.26
8.26
7.88
7.87
7.35
7.34
7.26
7.08
7.08
7.07
7.06
7.06
7.06
7.04
7.04
3.01
2.99
2.96
2.94
1.81
1.30
1.27
0.00
O
N
H
N
4-isopropyl-N-(pyridin-2-yl)benzamide
3d
0.93
0.99
0.95
1.84
1.09
1.92
0.98
1.00
5.96
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 0.0 -0.5
f1 (ppm)


166.1
153.5
151.9
147.8
138.5
131.9
127.5
126.8
119.7
114.4
77.5
77.1
76.7
34.1
23.7
O
N
H
N
4-isopropyl-N-(pyridin-2-yl)benzamide
3d
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

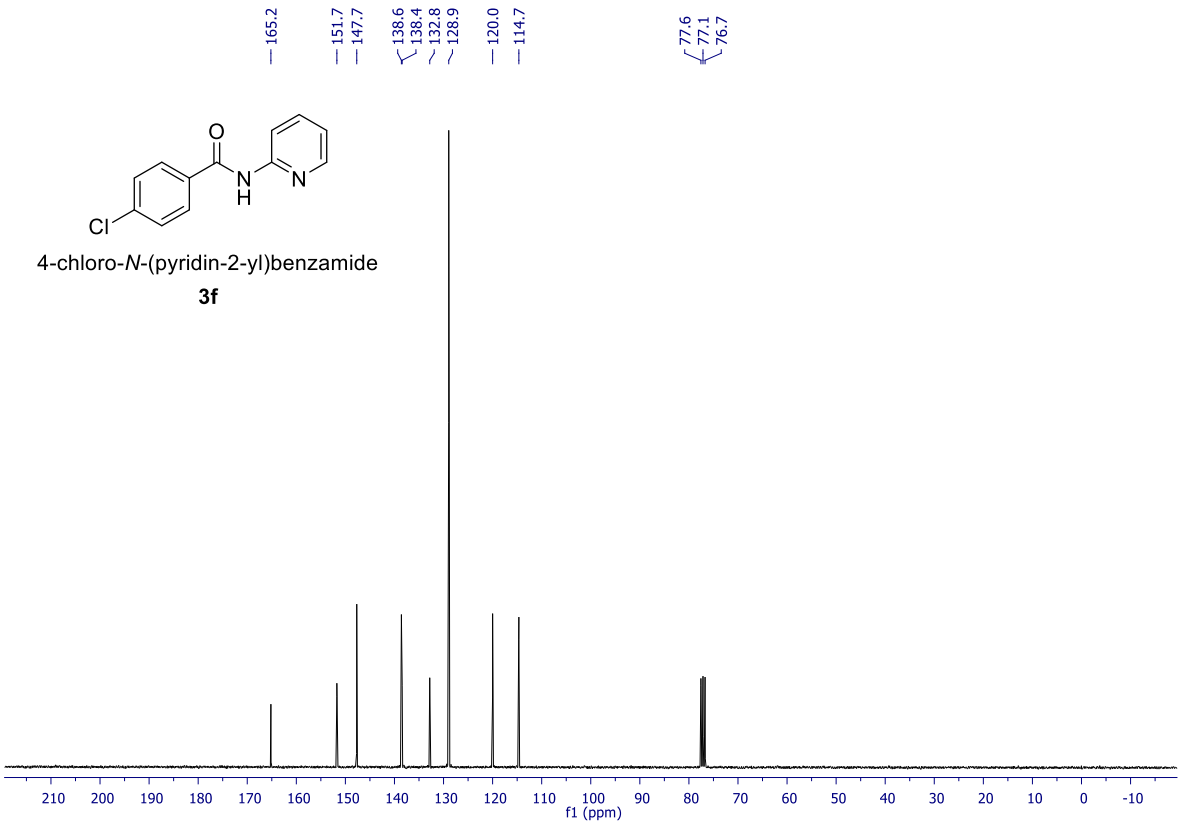

8.78
8.39
8.36
7.98
7.96
7.95
7.95
7.93
7.27
7.21
7.20
7.20
7.19
7.18
7.18
7.17
7.15
7.15
7.14
7.10
7.09
7.08
7.08
7.07
7.07
7.06
7.05
1.88
-0.00
O
N
H
N
F
4-fluoro-*N*-(pyridin-2-yl)benzamide
3e
0.97
1.00
1.03
2.08
1.07
3.07
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


166.8
165.1
163.4
151.8
147.7
138.6
130.6
130.6
129.9
129.8
119.9
116.0
115.7
114.5
77.5
77.1
76.7
O
N
H
N
F
4-fluoro-*N*-(pyridin-2-yl)benzamide
3e
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



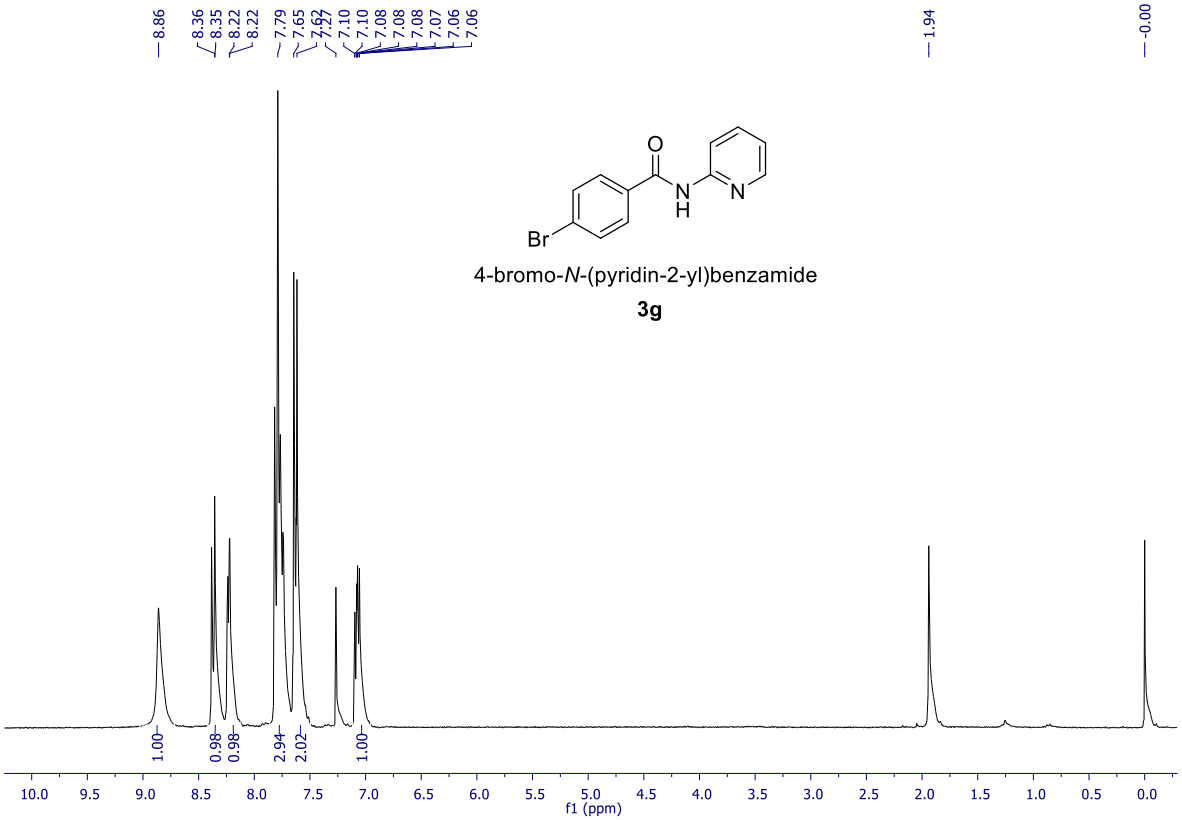
4-chloro-*N*-(pyridin-2-yl)benzamide

3f



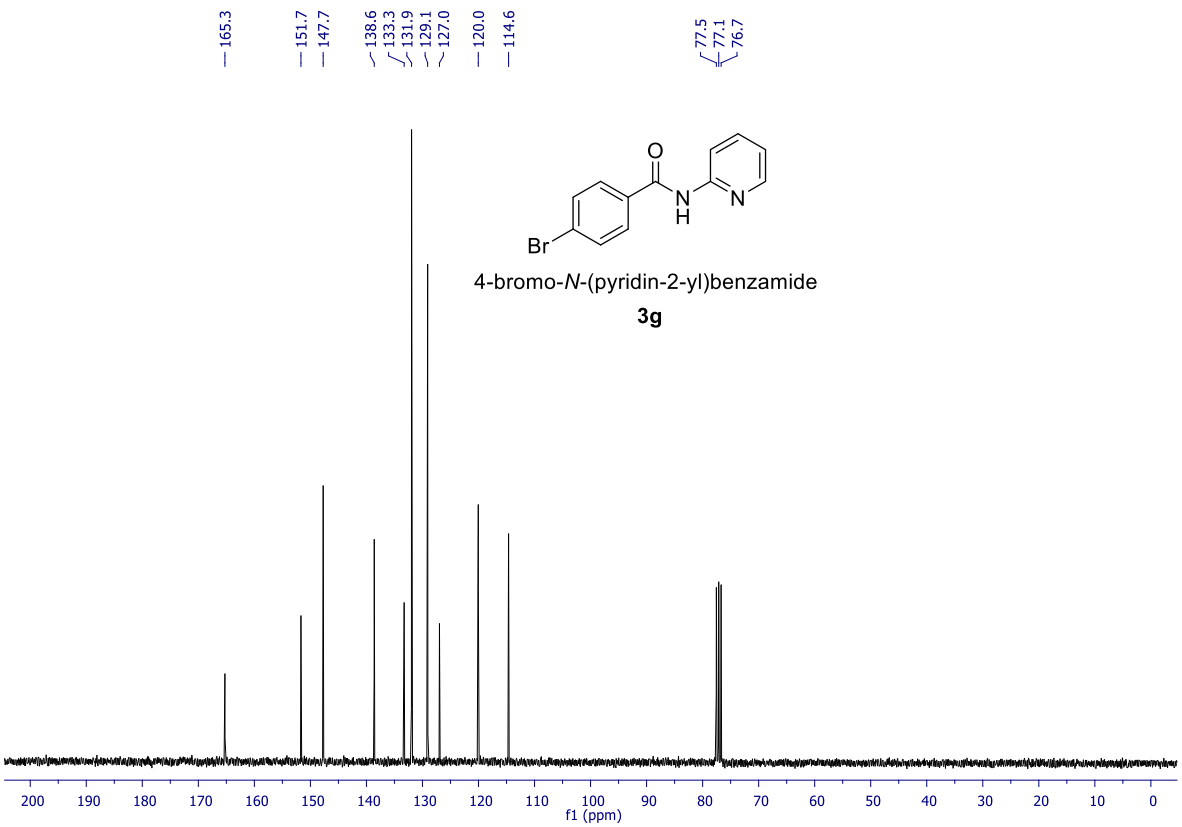
4-chloro-*N*-(pyridin-2-yl)benzamide

3f



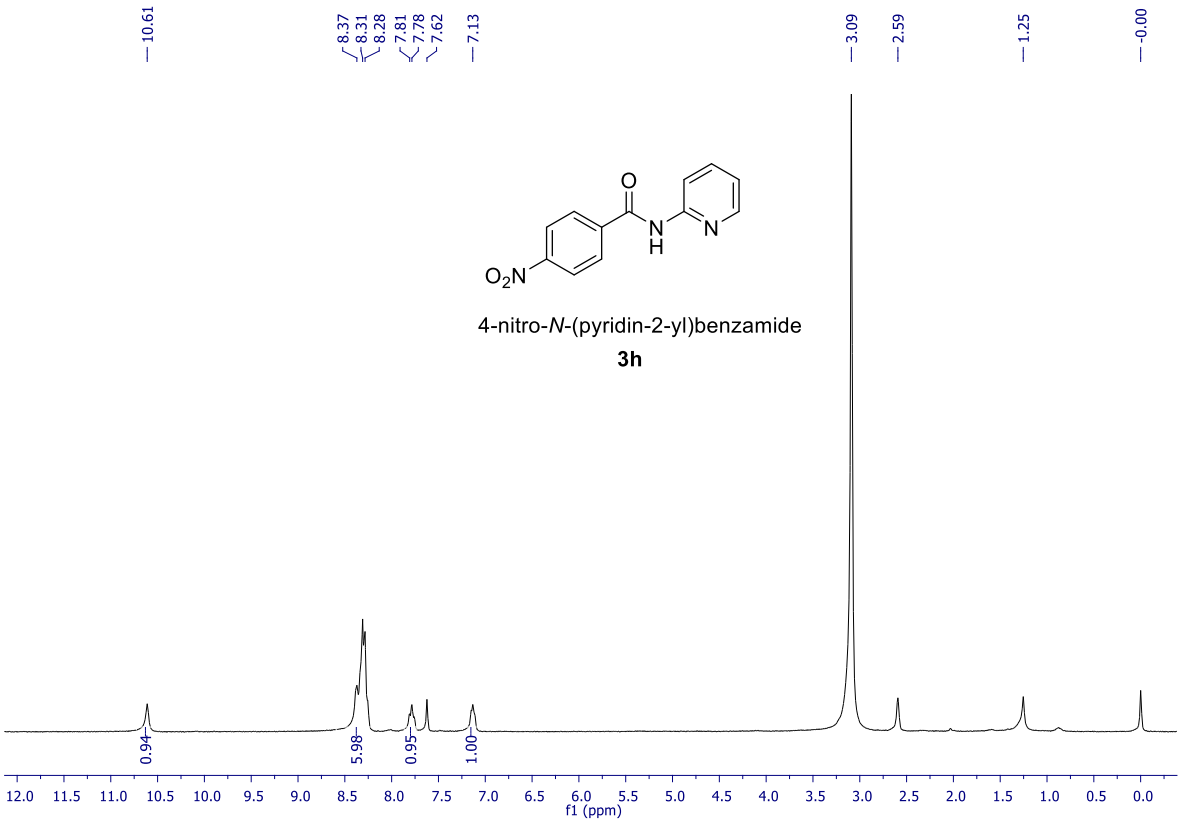
4-bromo-*N*-(pyridin-2-yl)benzamide

3g



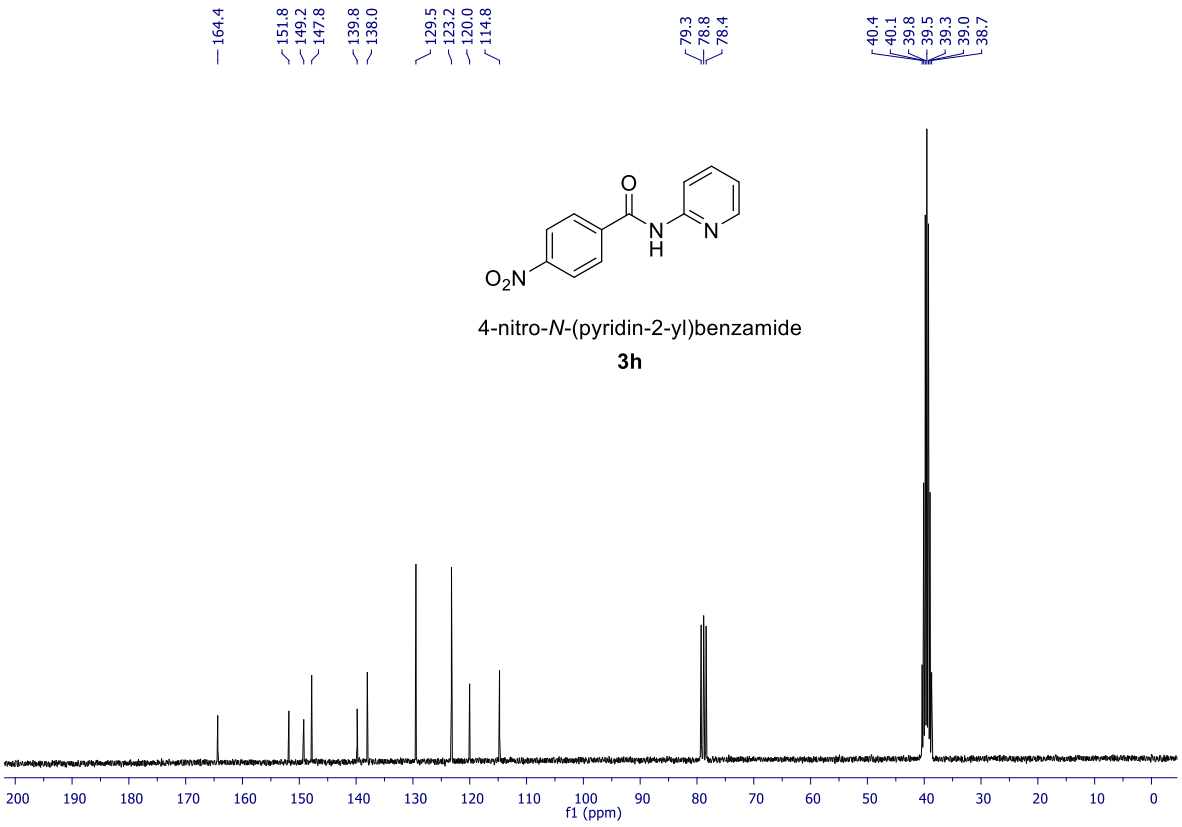
4-bromo-*N*-(pyridin-2-yl)benzamide

3g



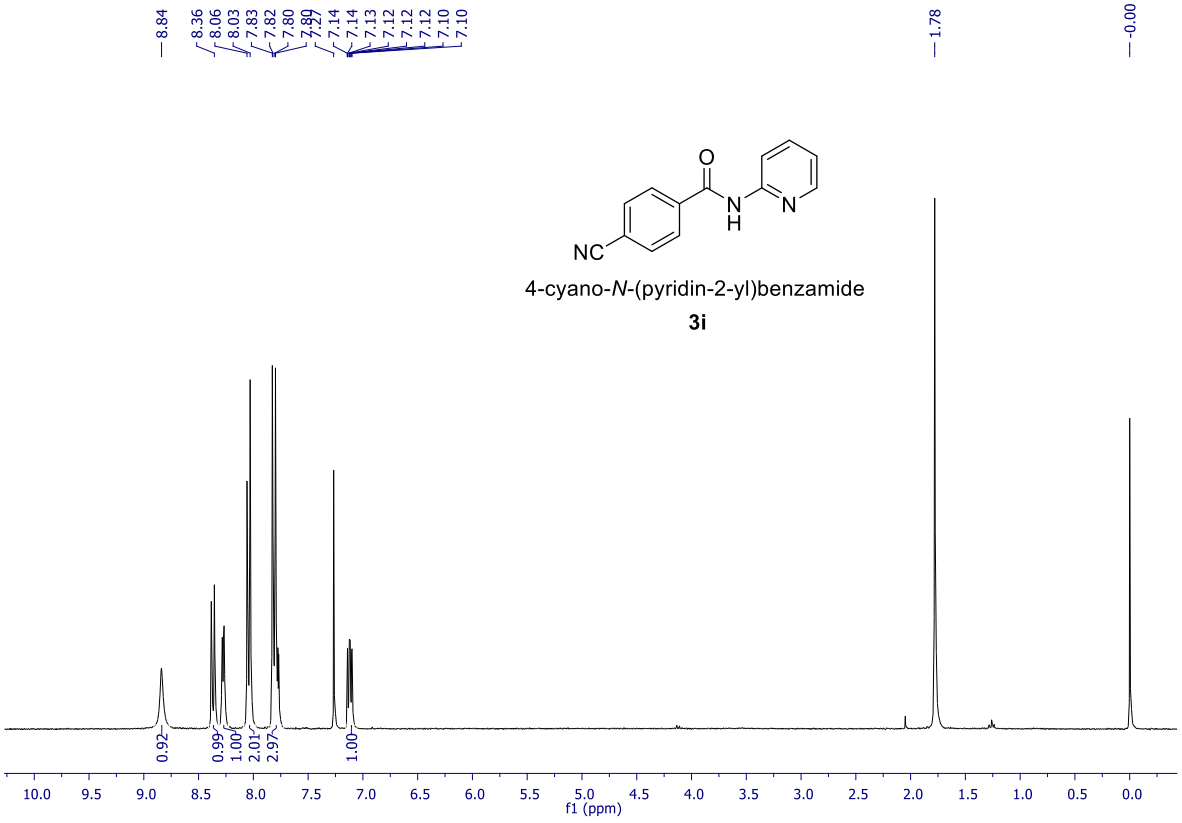
4-nitro-*N*-(pyridin-2-yl)benzamide

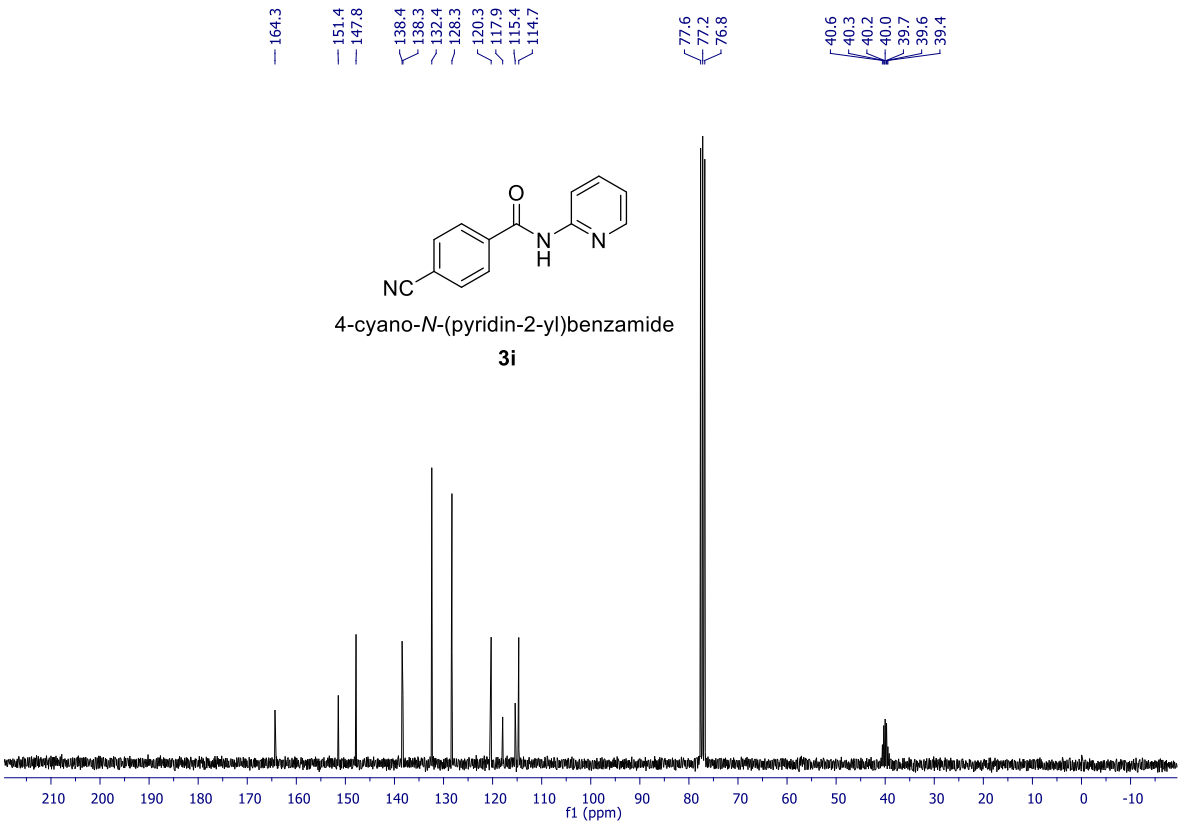
3h

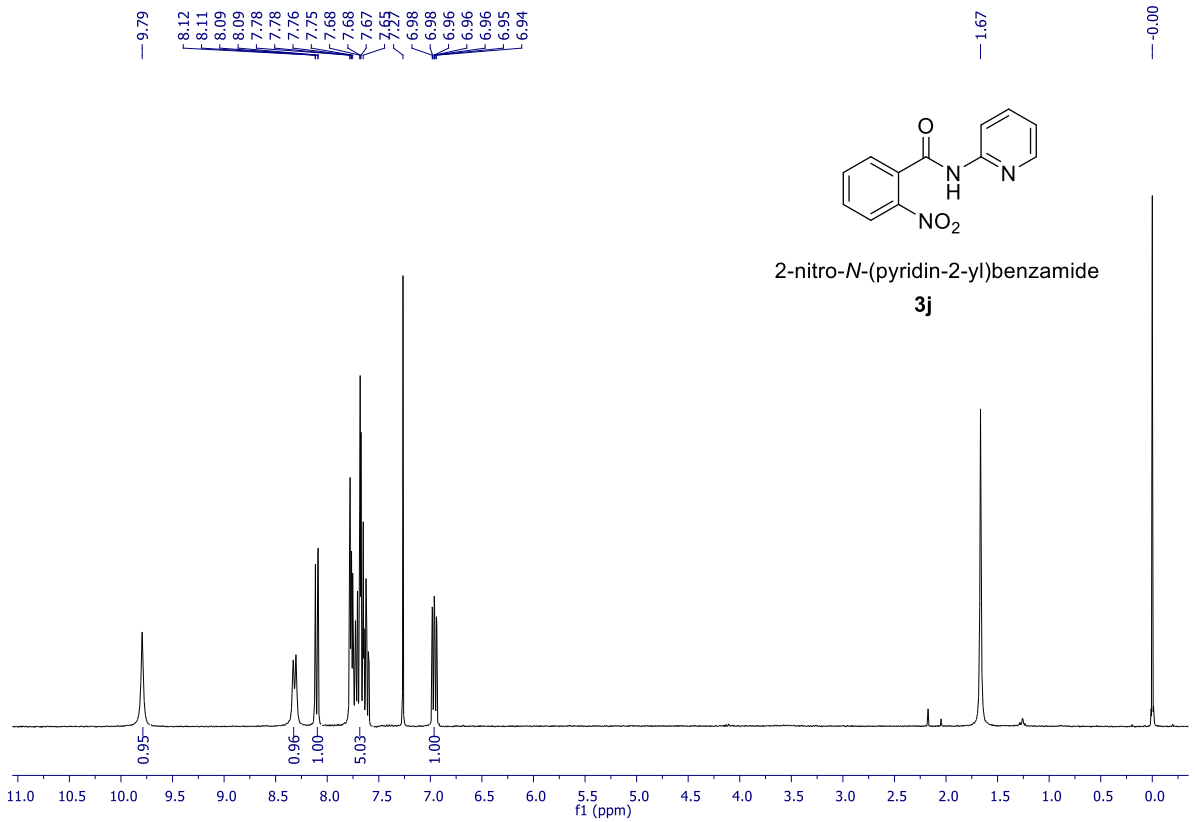


4-nitro-*N*-(pyridin-2-yl)benzamide

3h

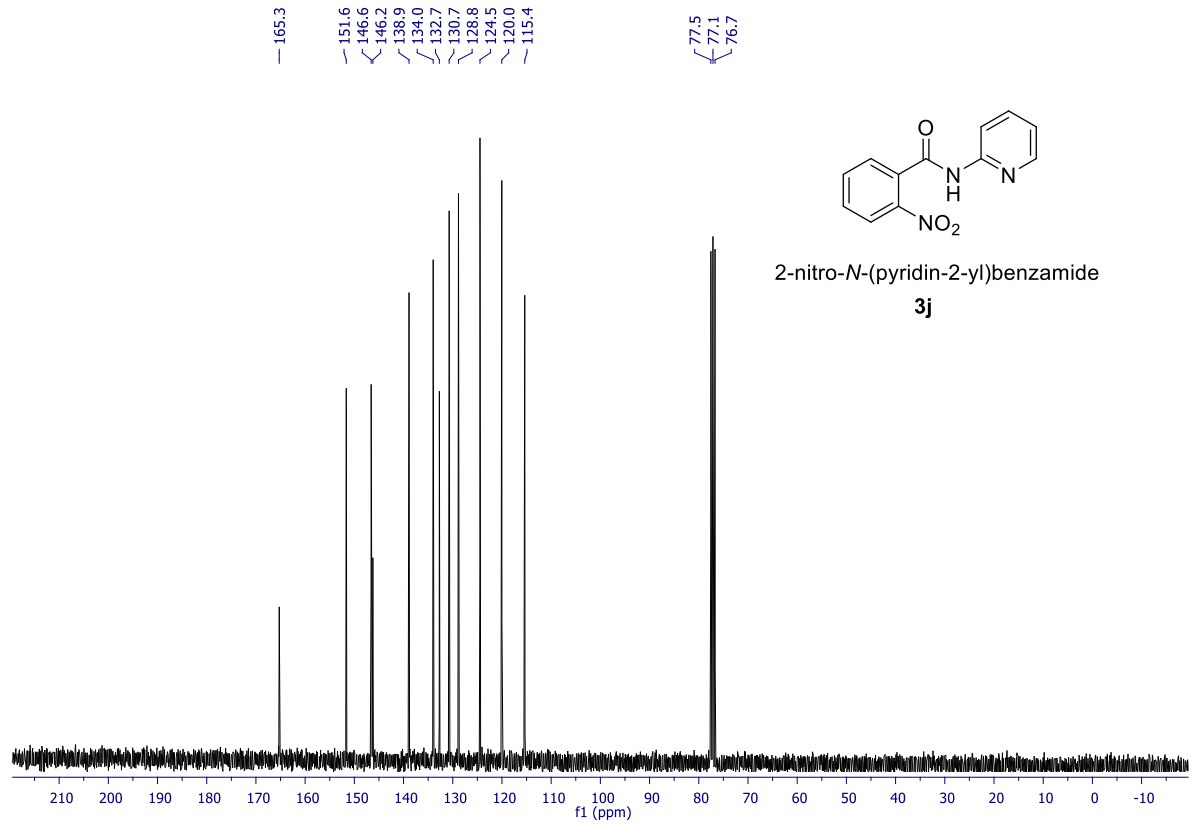

8.84
8.36
8.06
8.03
7.83
7.82
7.80
7.27
7.14
7.13
7.12
7.12
7.12
7.10
7.10
1.78
-0.00
O
N
H
N
NC
4-cyano-N-(pyridin-2-yl)benzamide
3i
0.92
0.99
1.00
2.01
2.97
1.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


164.3
151.4
147.8
138.4
138.3
132.4
128.3
120.3
117.9
115.4
114.7
77.6
77.2
76.8
40.6
40.3
40.2
40.0
39.7
39.6
39.4
O
N
H
N
NC
4-cyano-N-(pyridin-2-yl)benzamide
3i
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)



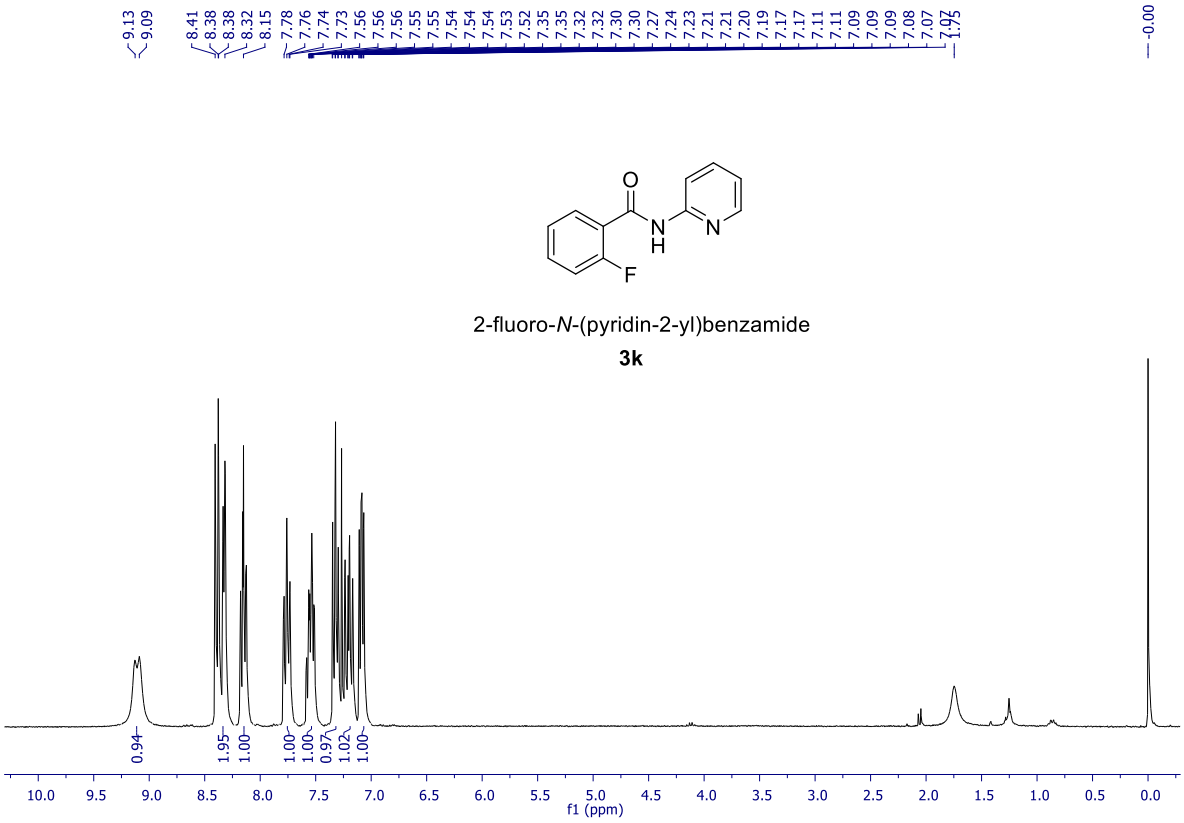
2-nitro-*N*-(pyridin-2-yl)benzamide

3j



2-nitro-*N*-(pyridin-2-yl)benzamide

3j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7.78
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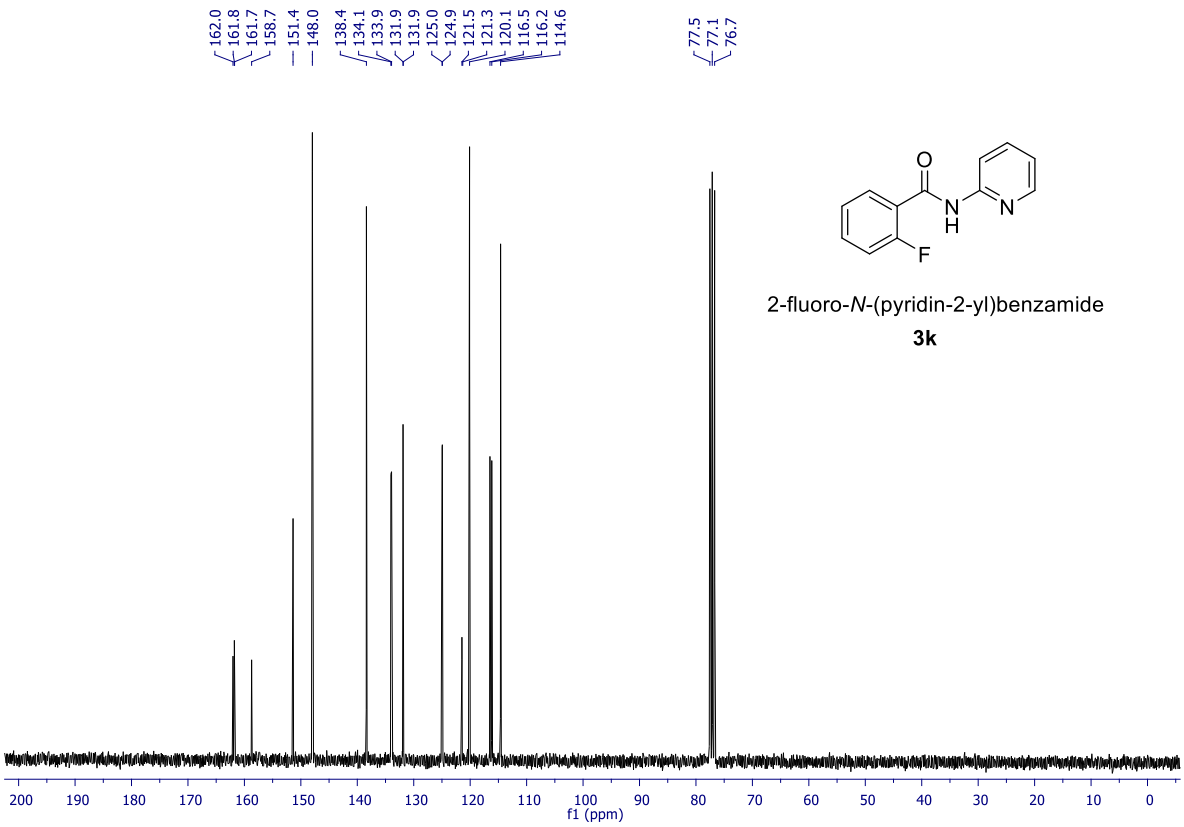
7.56
7.56
7.56
7.55
7.55
7.54
7.54
7.54
7.53
7.52
7.35
7.35
7.32
7.32
7.30
7.30
7.27
7.24
7.23
7.21
7.21
7.20
7.19
7.17
7.17
7.11
7.11
7.09
7.09
7.09
7.08
7.07
7.07
1.95
-0.00
O
N
H
N
F
2-fluoro-N-(pyridin-2-yl)benzamide
3k
0.94
1.95
1.00
1.00
1.00
0.97
1.02
1.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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161.7
158.7
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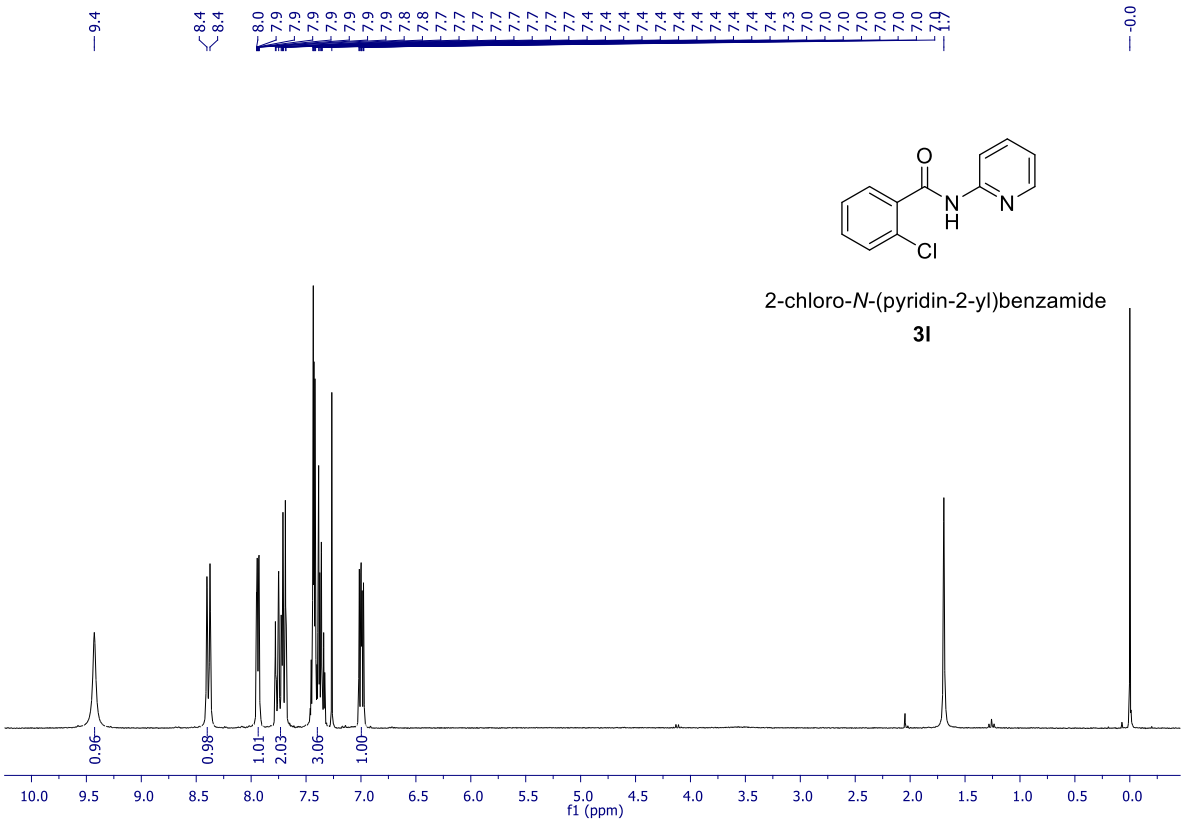
138.4
134.1
133.9
131.9
131.9
125.0
124.9
121.5
121.3
120.1
116.5
116.2
114.6
77.5
77.1
76.7
O
N
H
N
F
2-fluoro-N-(pyridin-2-yl)benzamide
3k
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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8.4
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7.9
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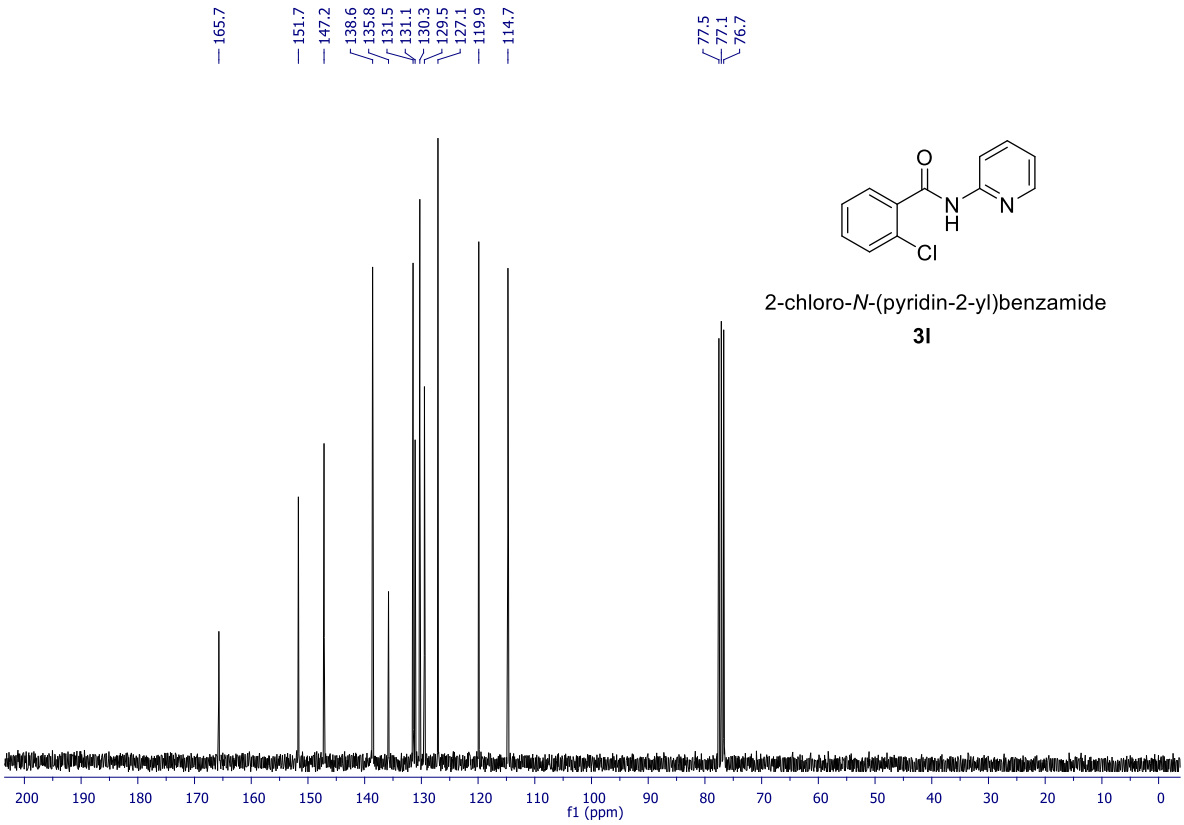
7.8
7.8
7.7
7.7
7.7
7.7
7.7
7.7
7.7
7.4
7.4
7.4
7.4
7.4
7.4
7.4
7.4
7.4
7.3
7.0
7.0
7.0
7.0
7.0
7.0
7.0
1.9
1.7
0.0
O
N
H
N
Cl
2-chloro-*N*-(pyridin-2-yl)benzamide
3l
0.96
0.98
1.01
2.03
3.06
1.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


165.7
151.7
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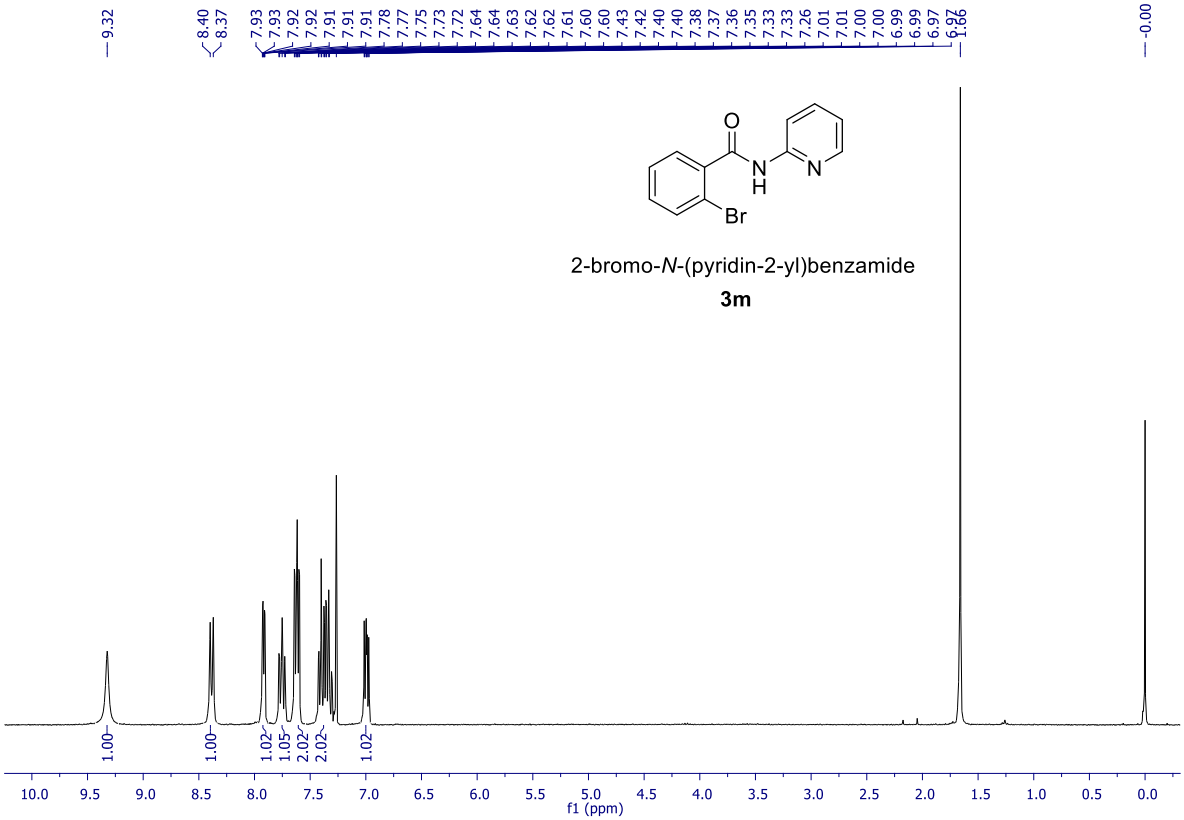
138.6
135.8
131.5
131.1
130.3
129.5
127.1
119.9
114.7
77.5
77.1
76.7
O
N
H
N
Cl
2-chloro-*N*-(pyridin-2-yl)benzamide
3l
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.75
7.73
7.72
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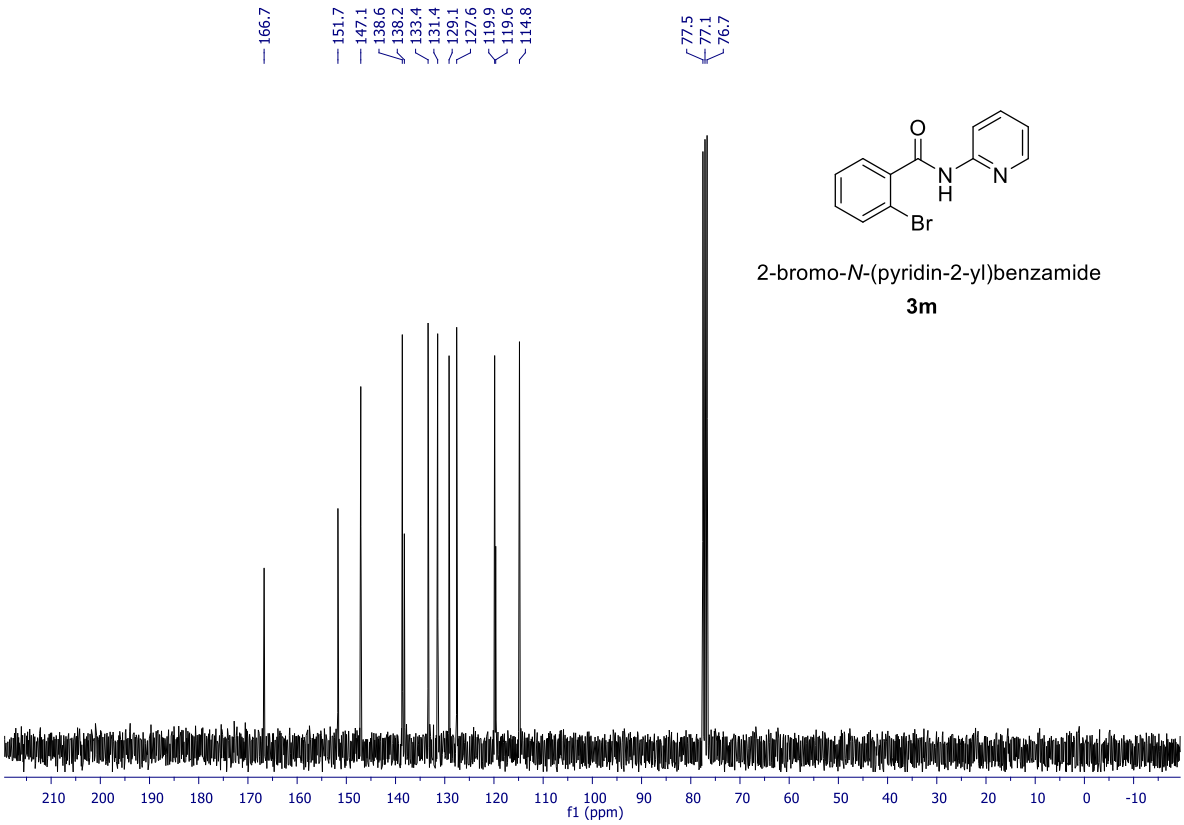
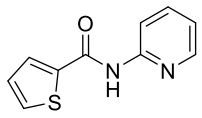
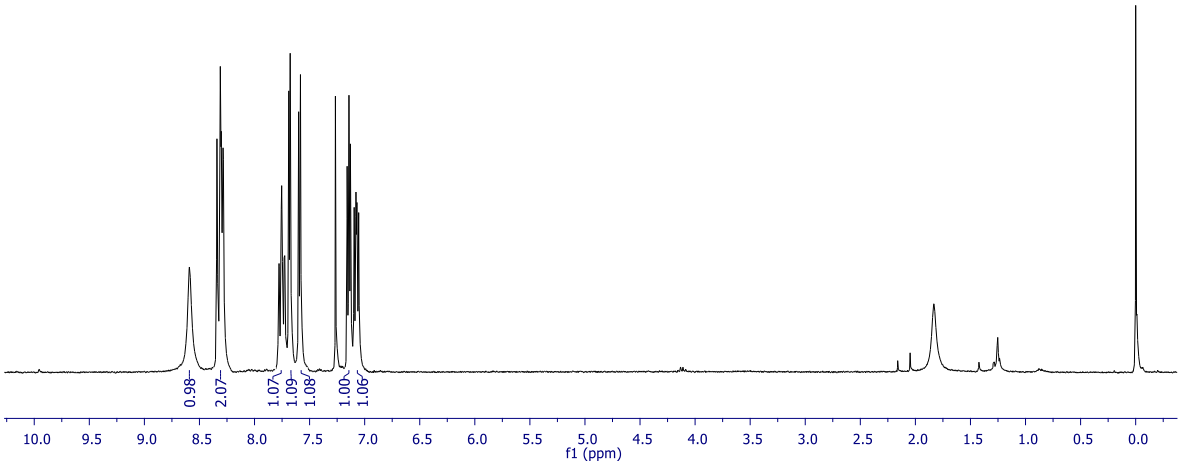
7.64
7.63
7.62
7.62
7.61
7.60
7.60
7.43
7.42
7.40
7.40
7.38
7.37
7.36
7.35
7.33
7.33
7.26
7.01
7.01
7.00
7.00
6.99
6.99
6.97
6.97
1.66
-0.00
O
N
H
N
Br
2-bromo-N-(pyridin-2-yl)benzamide
3m
1.00
1.00
1.02
1.05
2.02
2.02
1.02
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

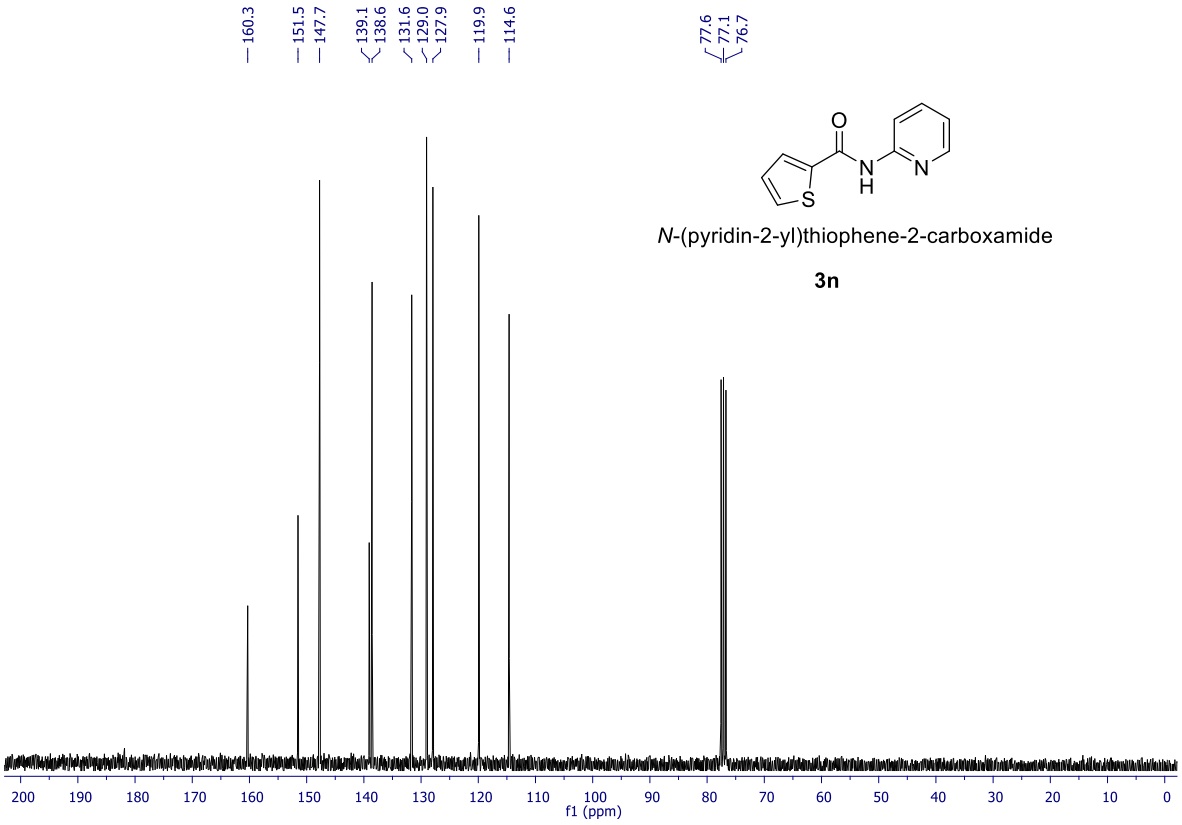

166.7
151.7
147.1
138.6
138.2
133.4
131.4
129.1
127.6
119.9
119.6
114.8
77.5
77.1
76.7
O
N
H
N
Br
2-bromo-N-(pyridin-2-yl)benzamide
3m
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)



N-(pyridin-2-yl)thiophene-2-carboxamide

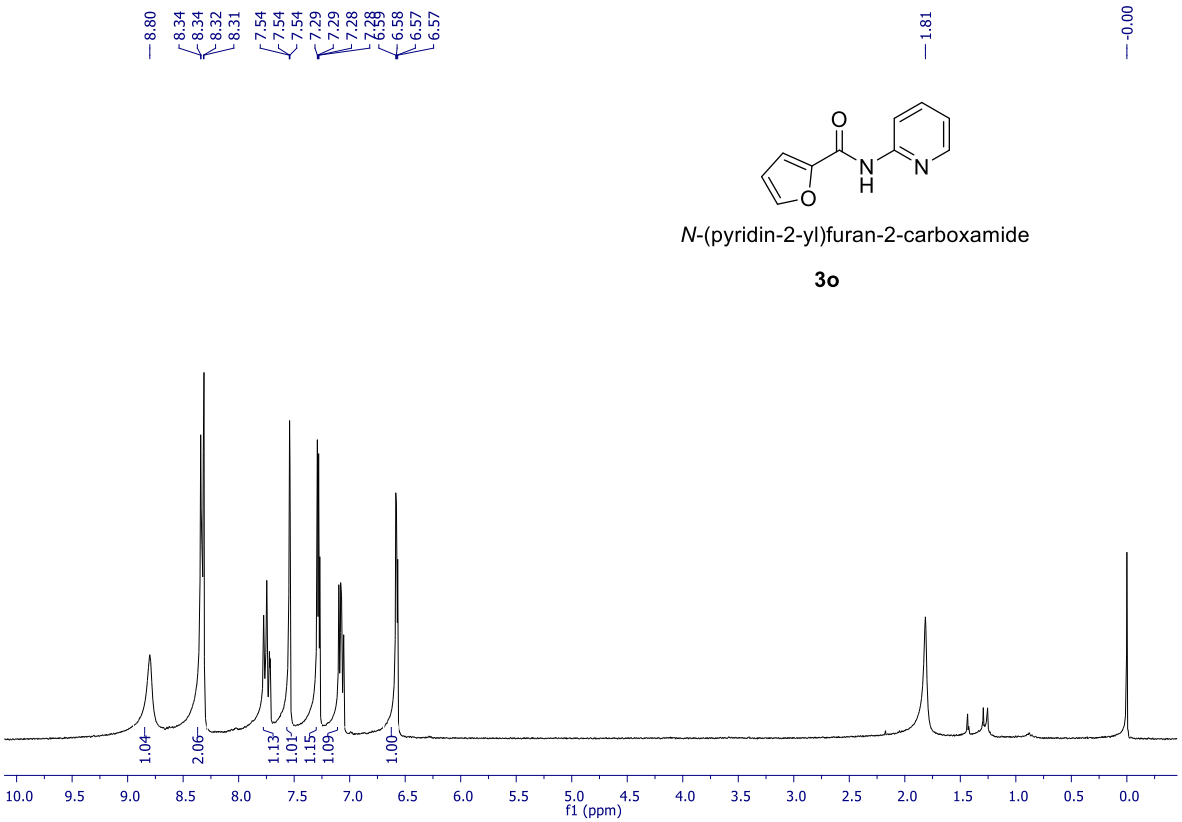
3n





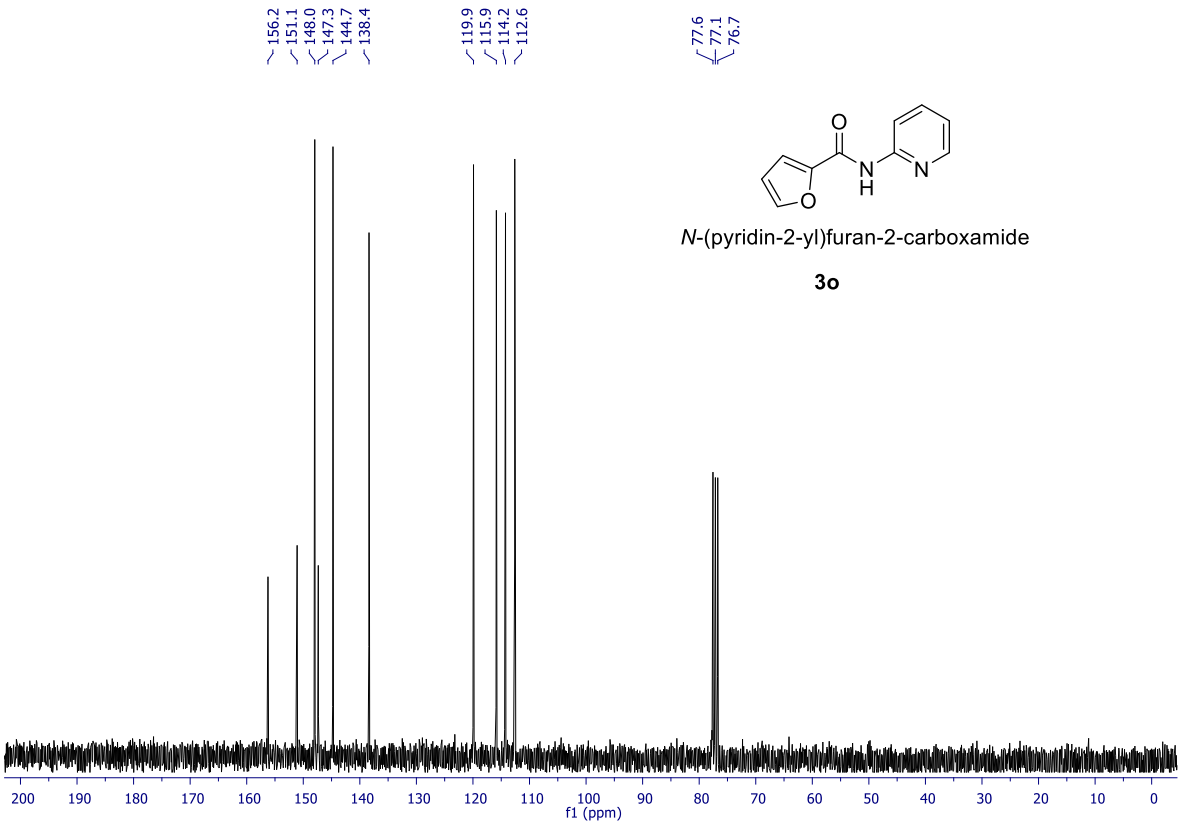
N-(pyridin-2-yl)thiophene-2-carboxamide

3n



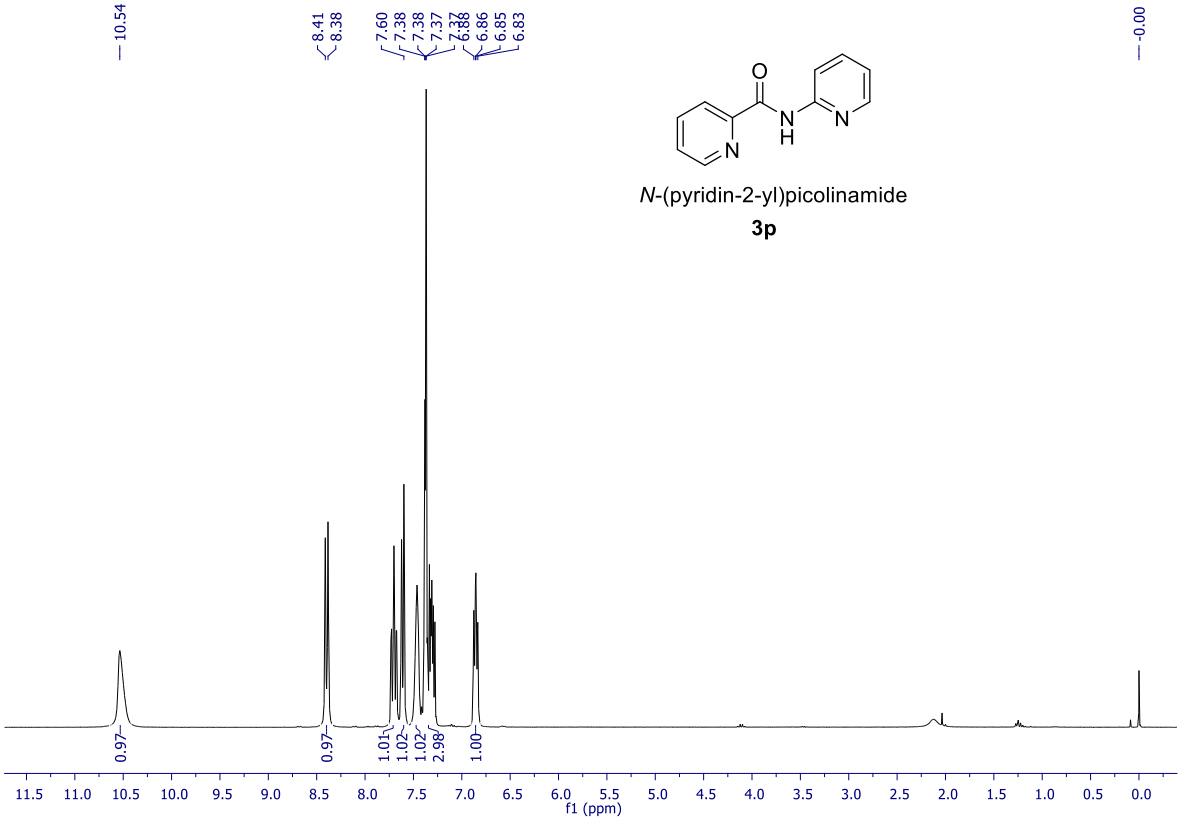
N-(pyridin-2-yl)furan-2-carboxamide

3o



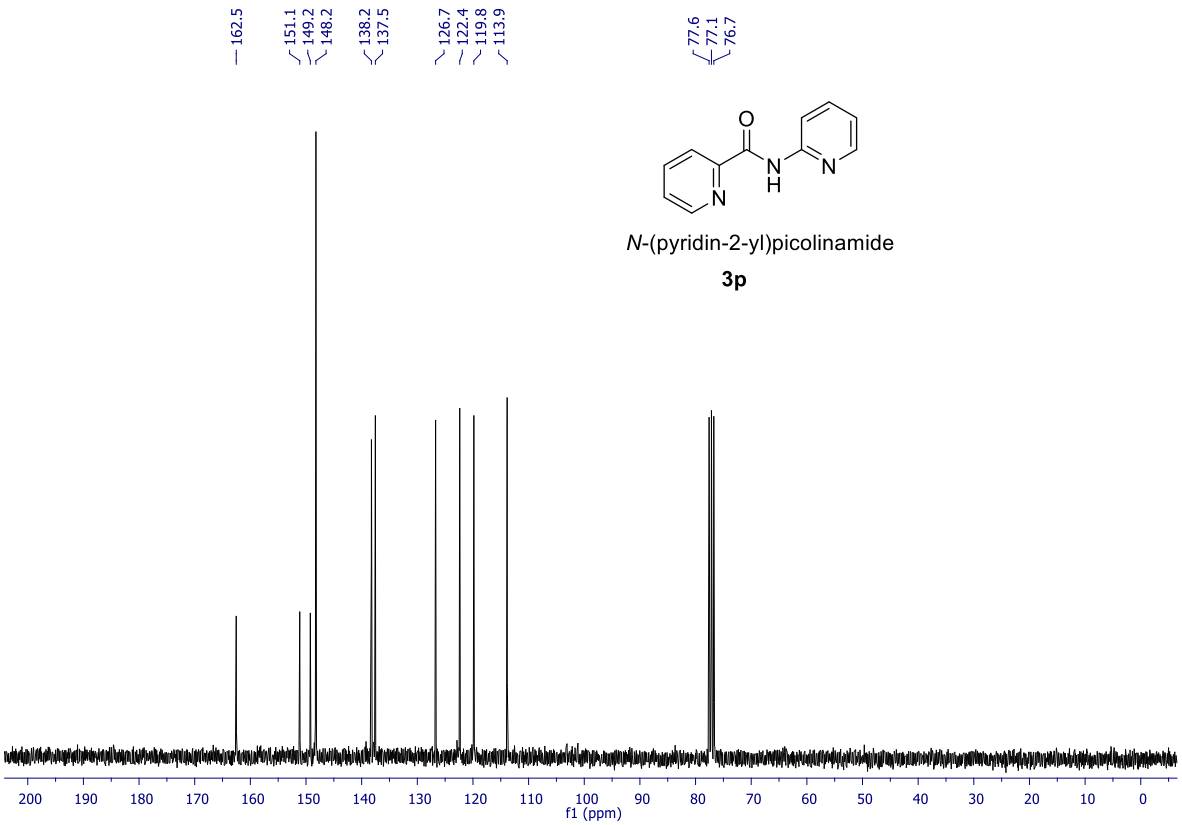
N-(pyridin-2-yl)furan-2-carboxamide

3o



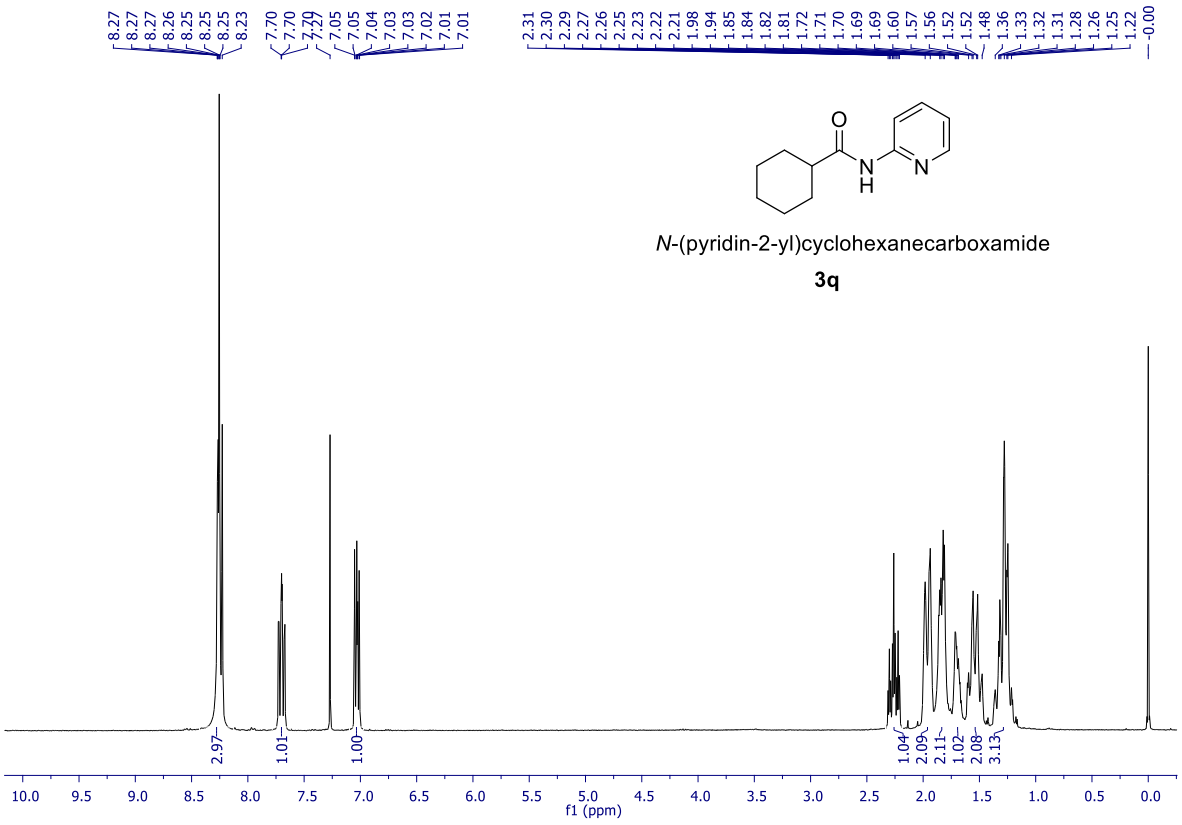
N-(pyridin-2-yl)picolinamide

3p



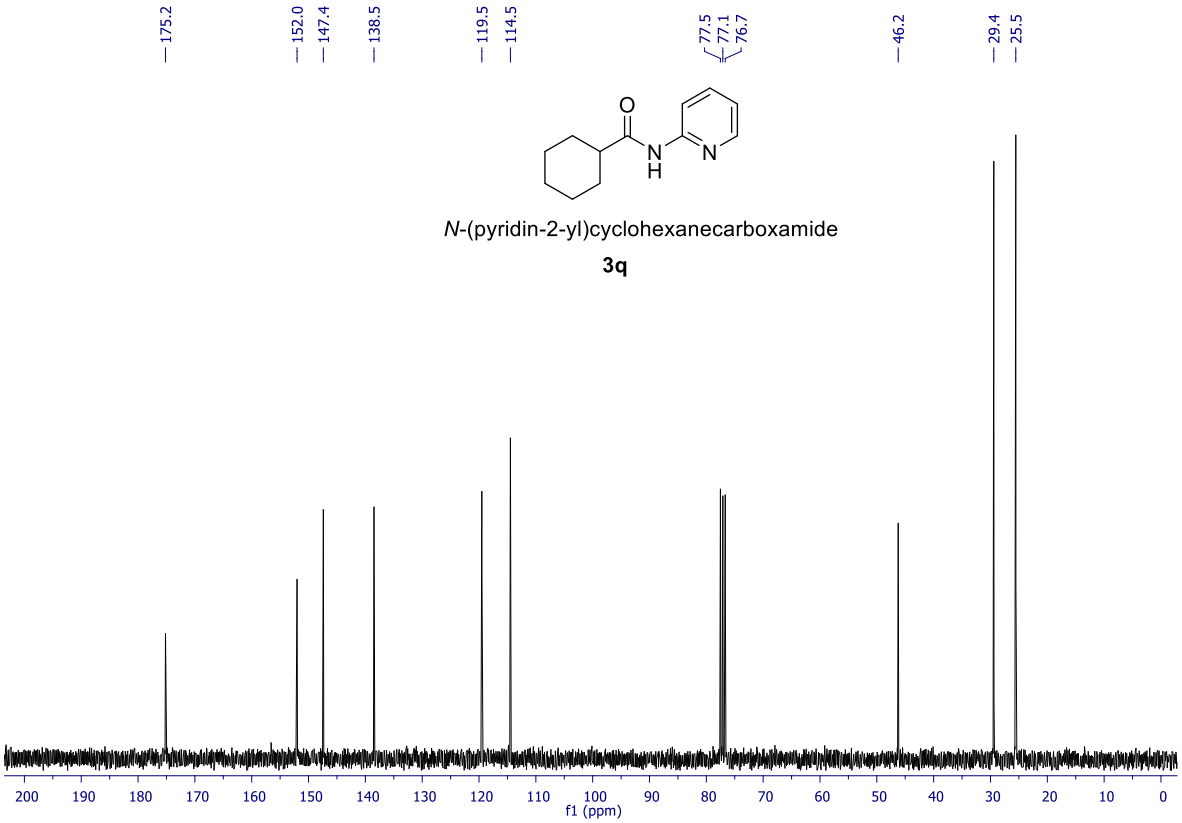
N-(pyridin-2-yl)picolinamide

3p



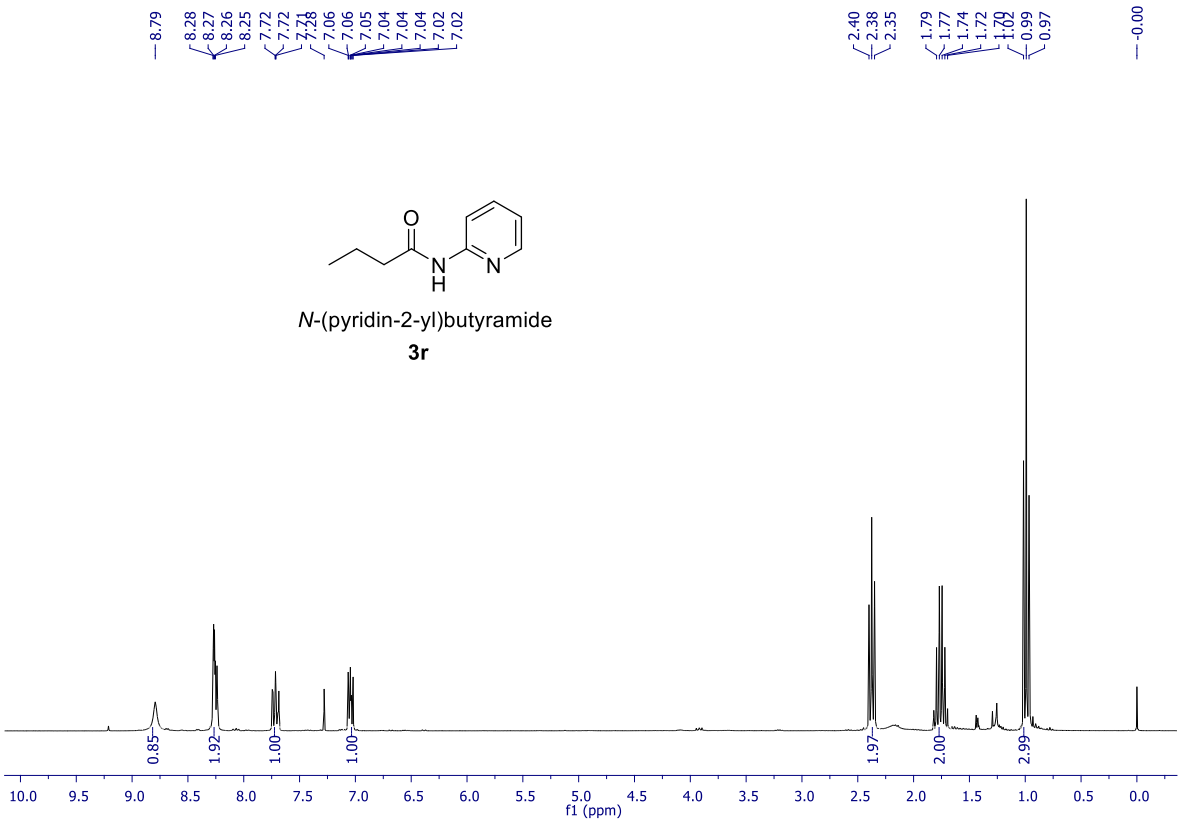
N-(pyridin-2-yl)cyclohexanecarboxamide

3q



N-(pyridin-2-yl)cyclohexanecarboxamide

3q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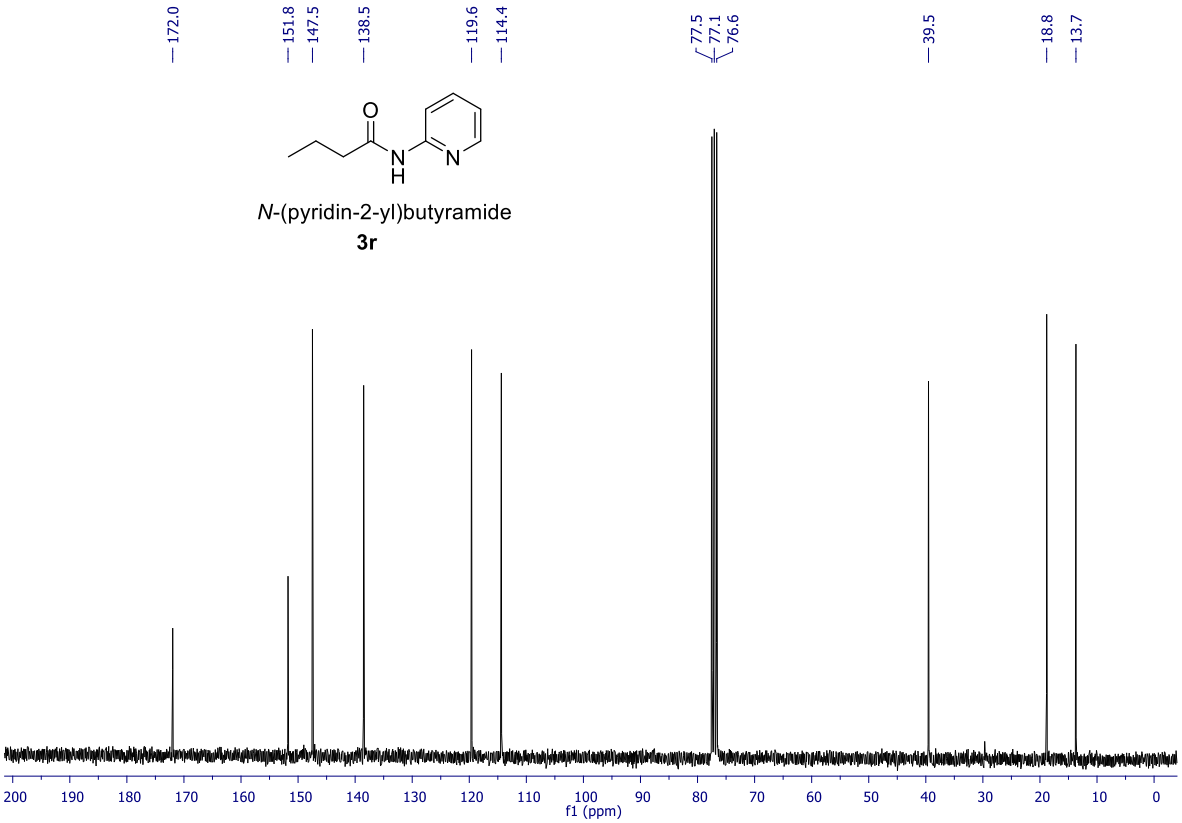
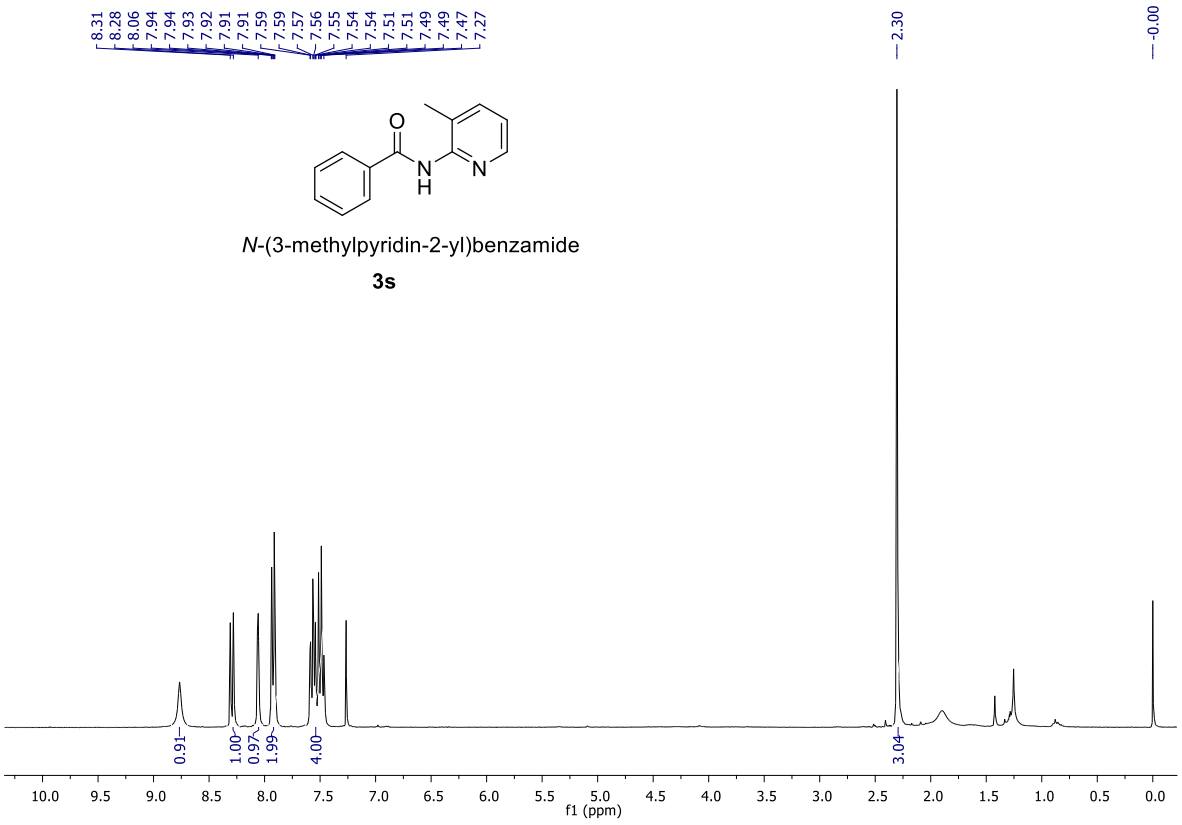
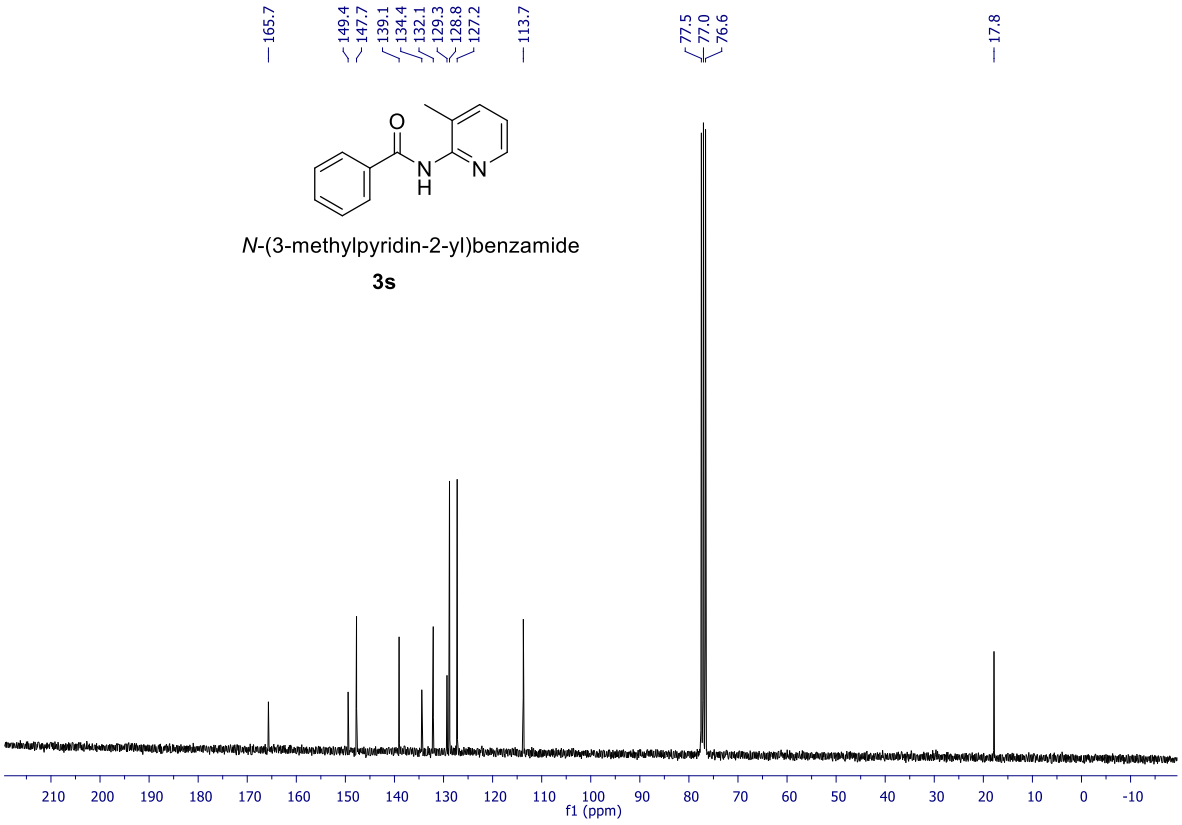
8.27
8.26
8.25
7.72
7.72
7.71
7.71
7.06
7.06
7.05
7.04
7.04
7.04
7.02
7.02
2.40
2.38
2.35
1.79
1.77
1.74
1.72
1.70
1.02
0.99
0.97
-0.00
O
N
H
N
N-(pyridin-2-yl)butyramide
3r
0.85
1.92
1.00
1.00
1.97
2.00
2.99
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


172.0
151.8
147.5
138.5
119.6
114.4
77.5
77.1
76.6
39.5
18.8
13.7
O
N
H
N
N-(pyridin-2-yl)butyramide
3r
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)



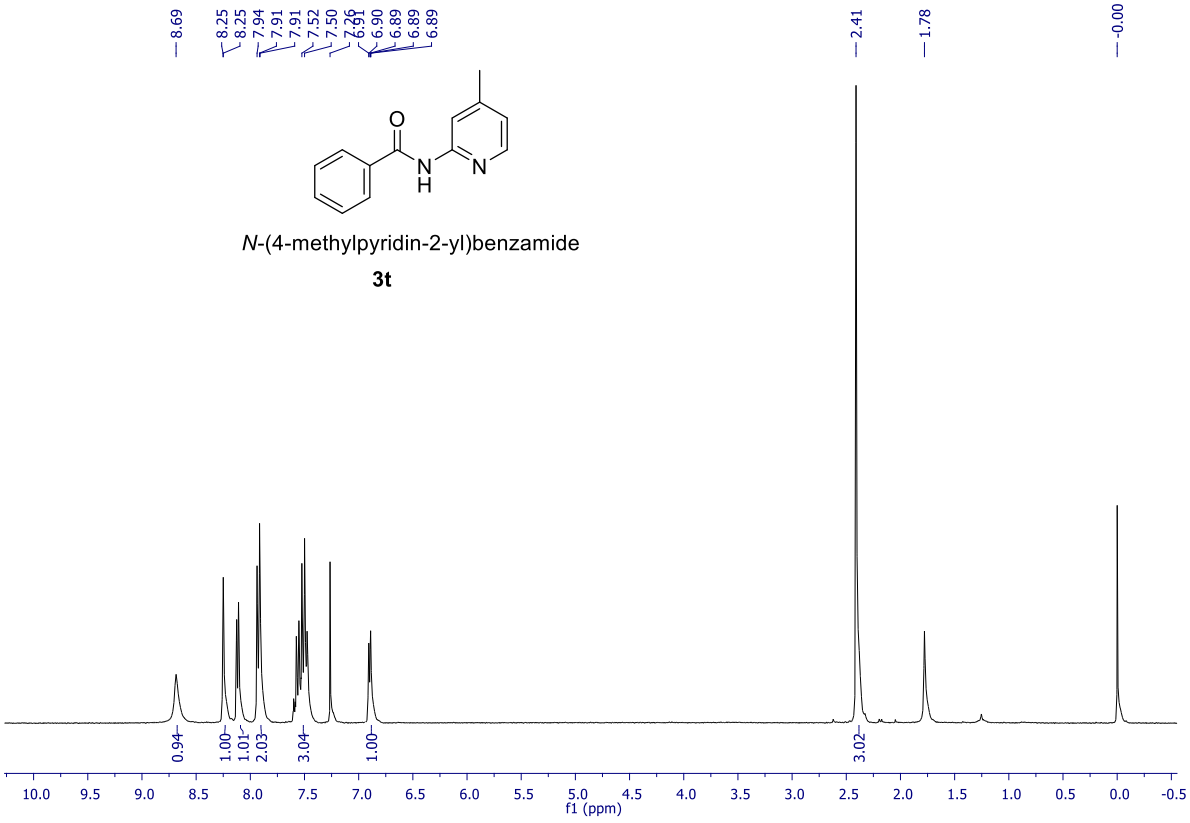
N-(3-methylpyridin-2-yl)benzamide

3s



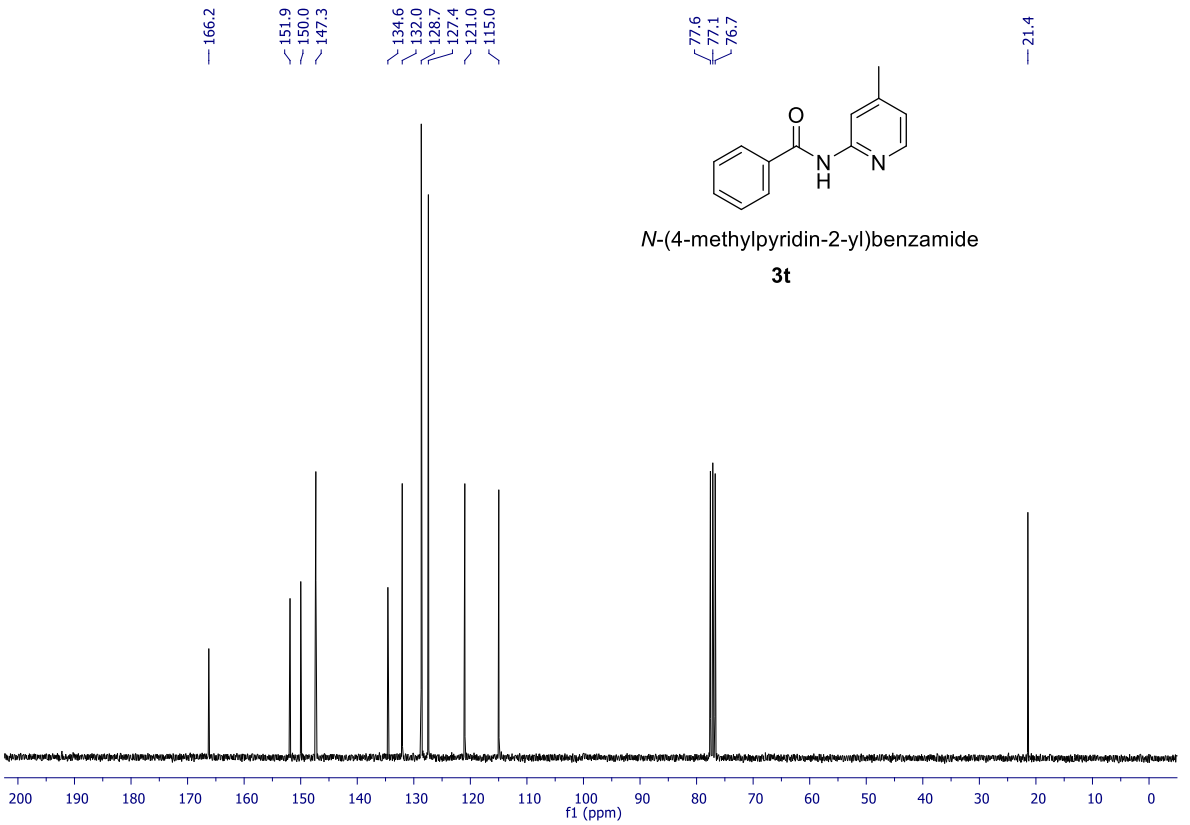
N-(3-methylpyridin-2-yl)benzamide

3s



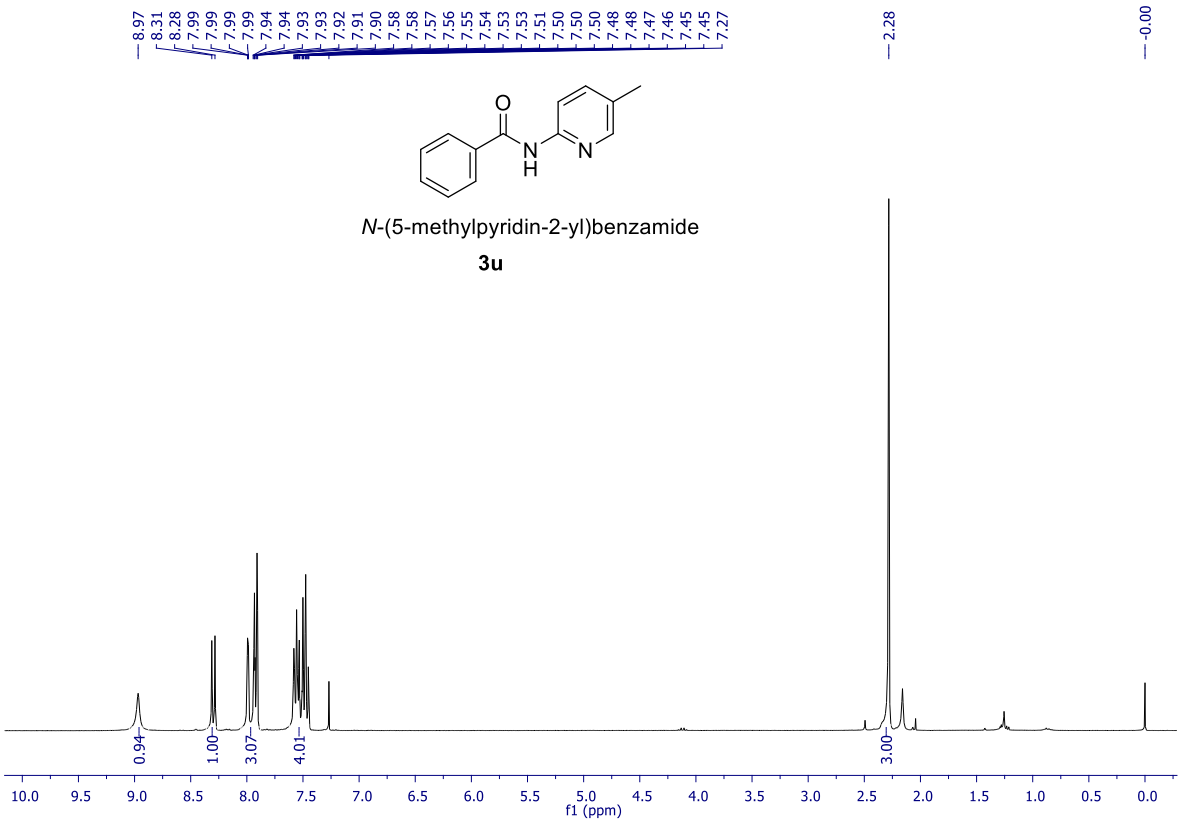
N-(4-methylpyridin-2-yl)benzamide

3t



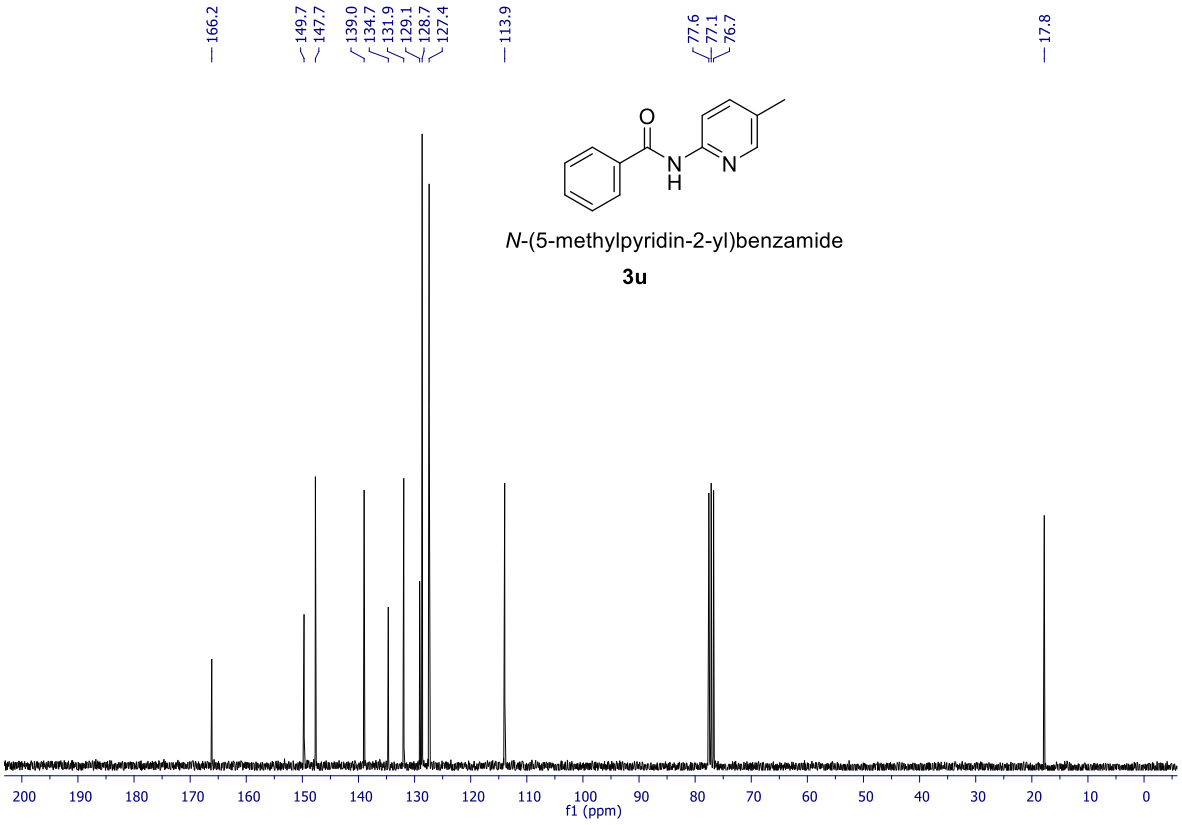
N-(4-methylpyridin-2-yl)benzamide

3t



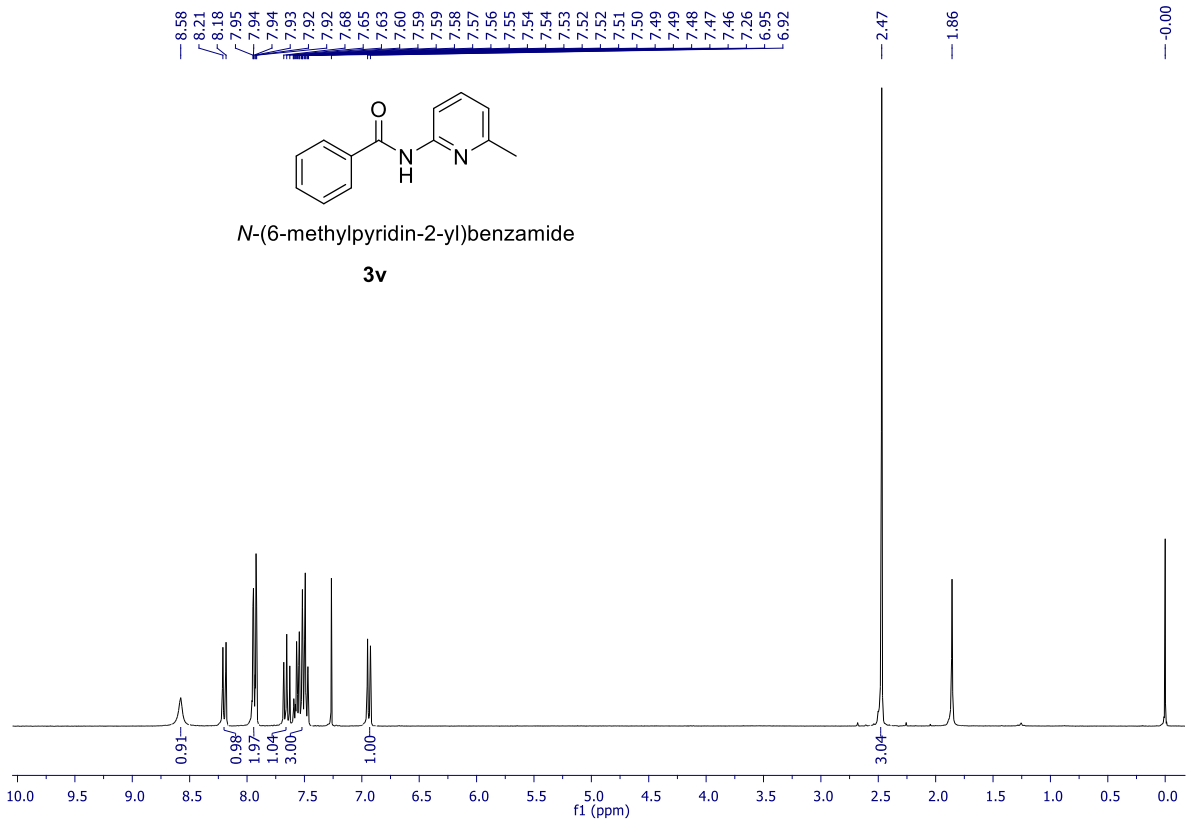
N-(5-methylpyridin-2-yl)benzamide

3u



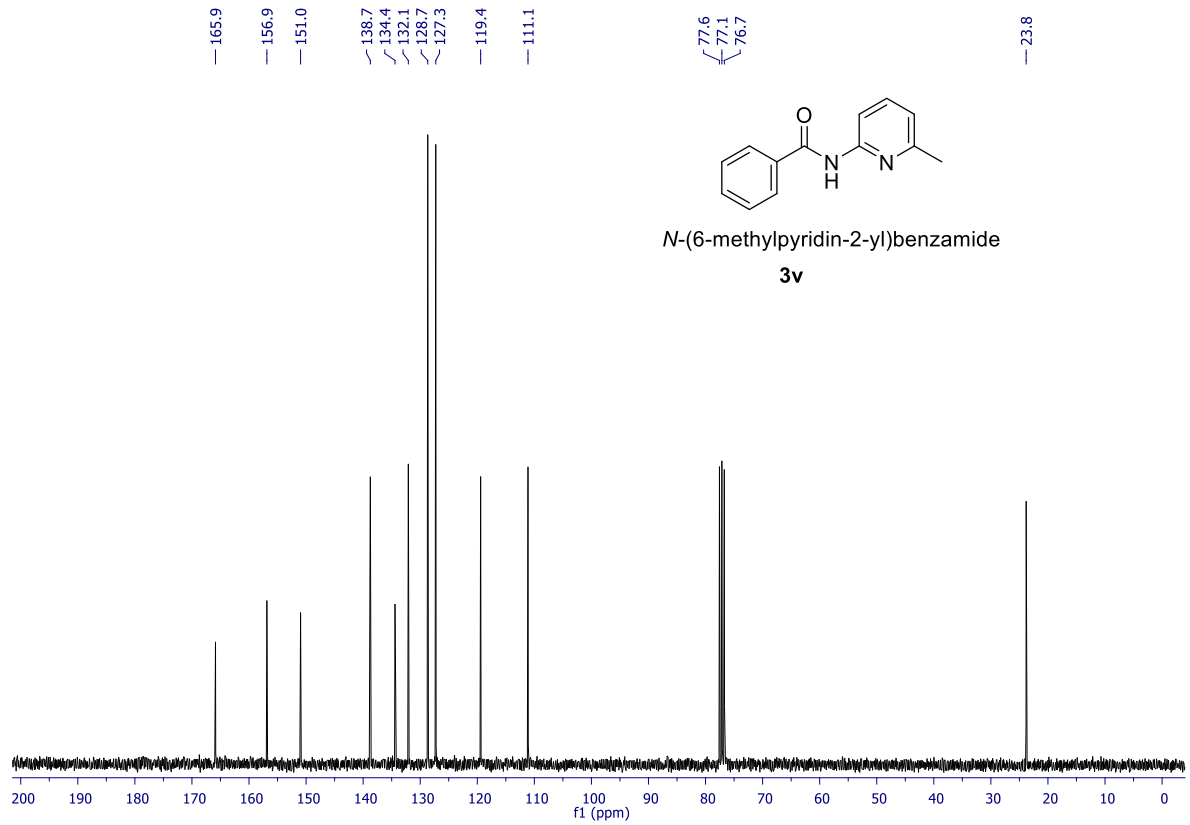
N-(5-methylpyridin-2-yl)benzamide

3u



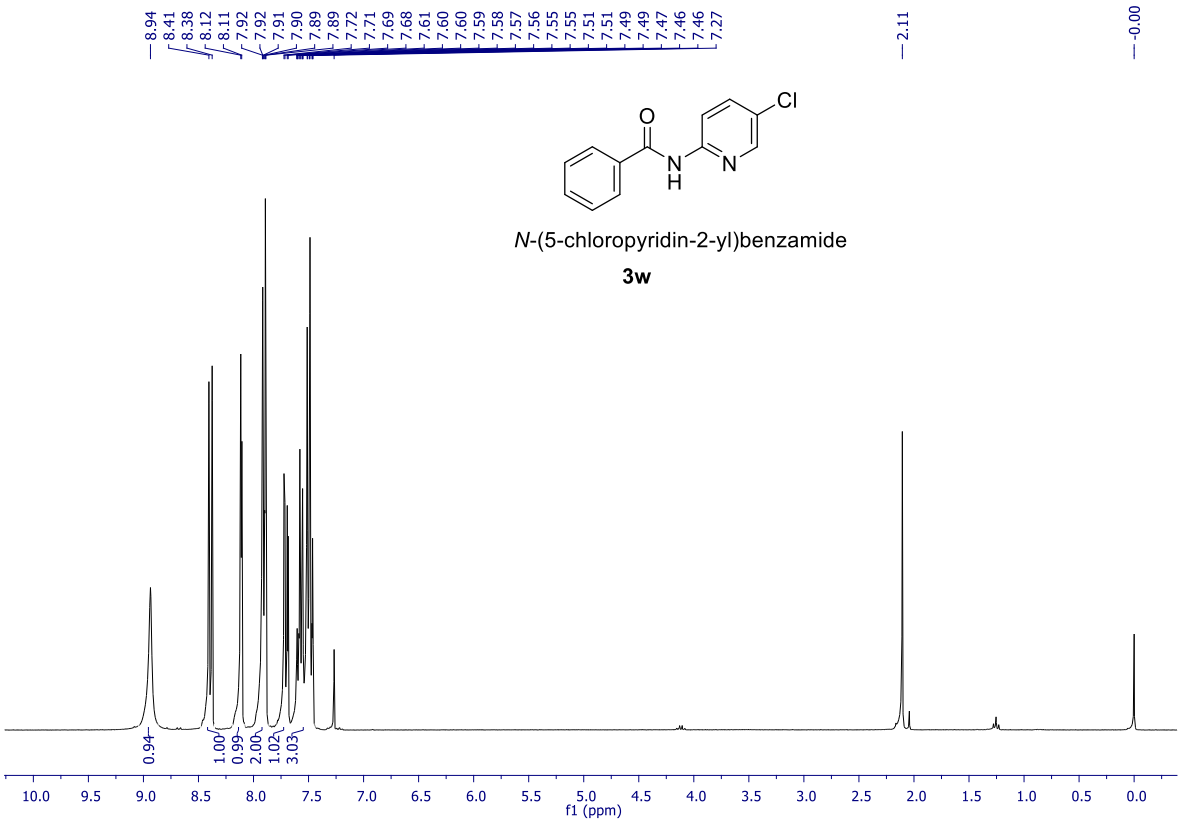
N-(6-methylpyridin-2-yl)benzamide

3v



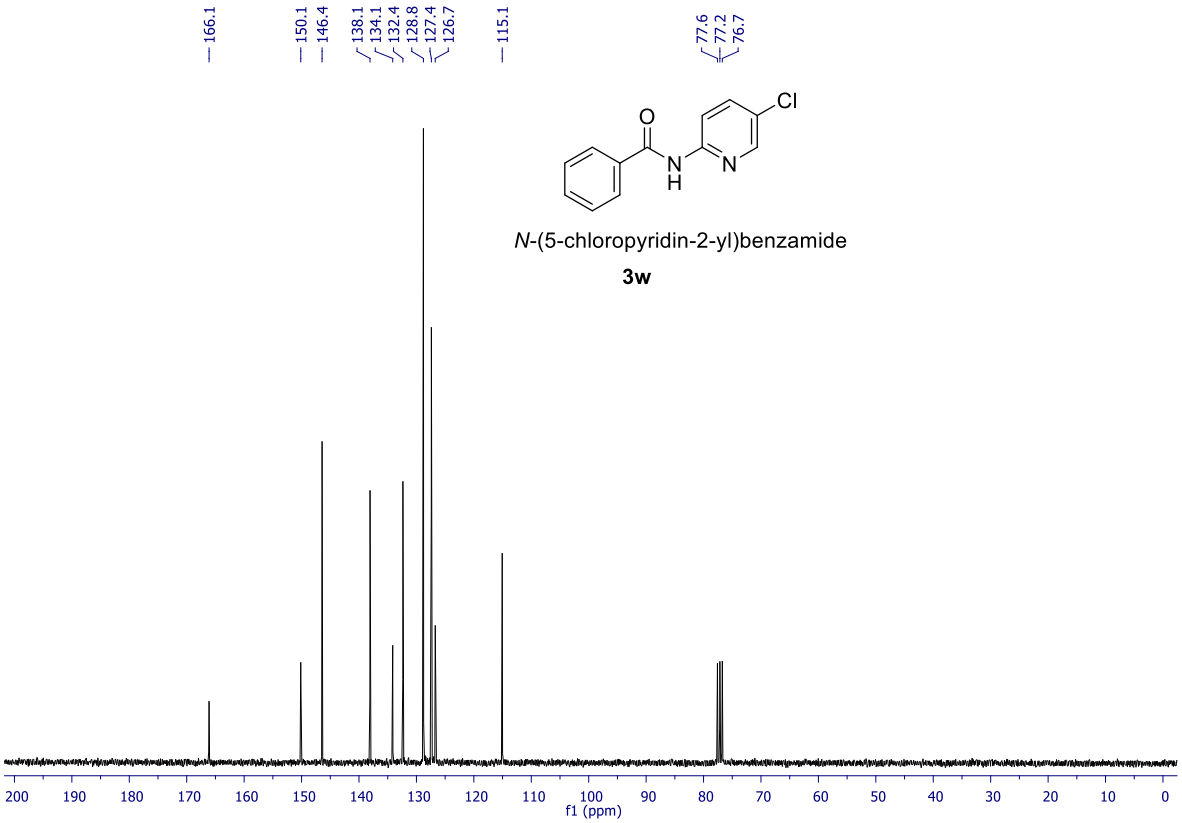
N-(6-methylpyridin-2-yl)benzamide

3v



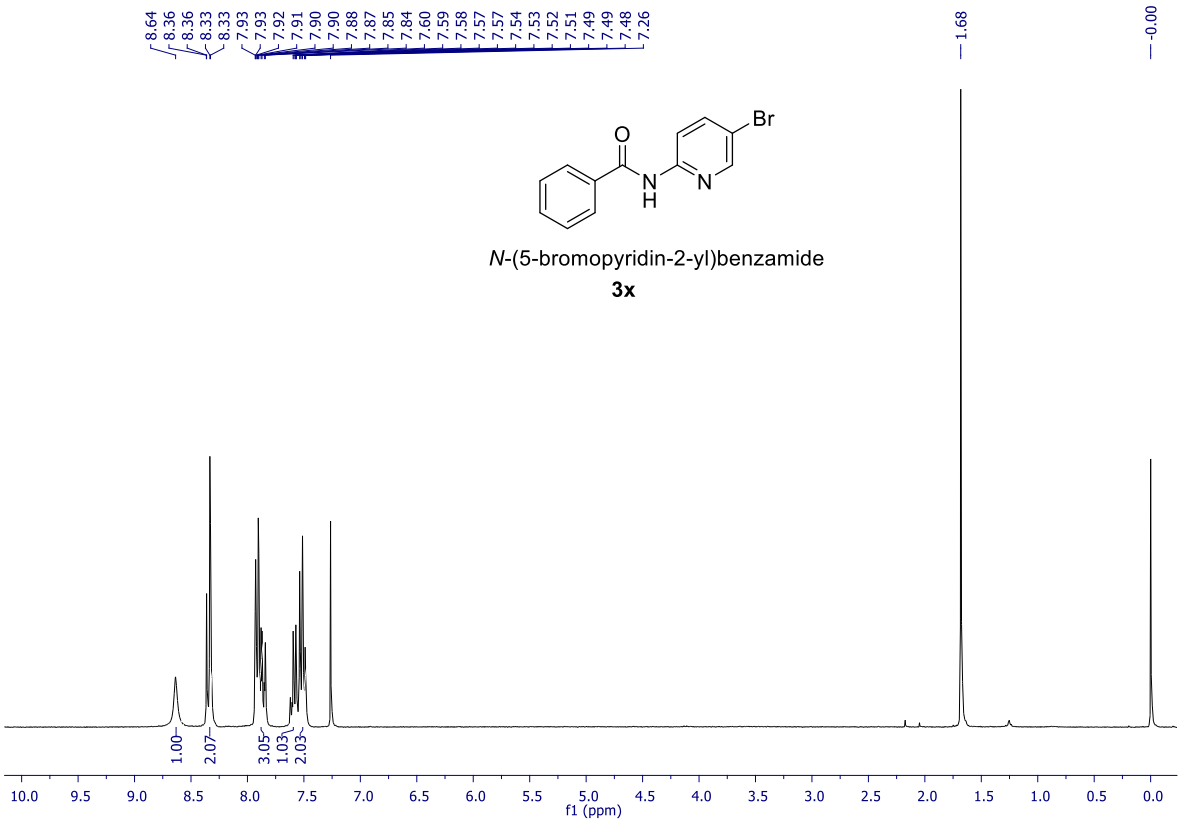
N-(5-chloropyridin-2-yl)benzamide

3w



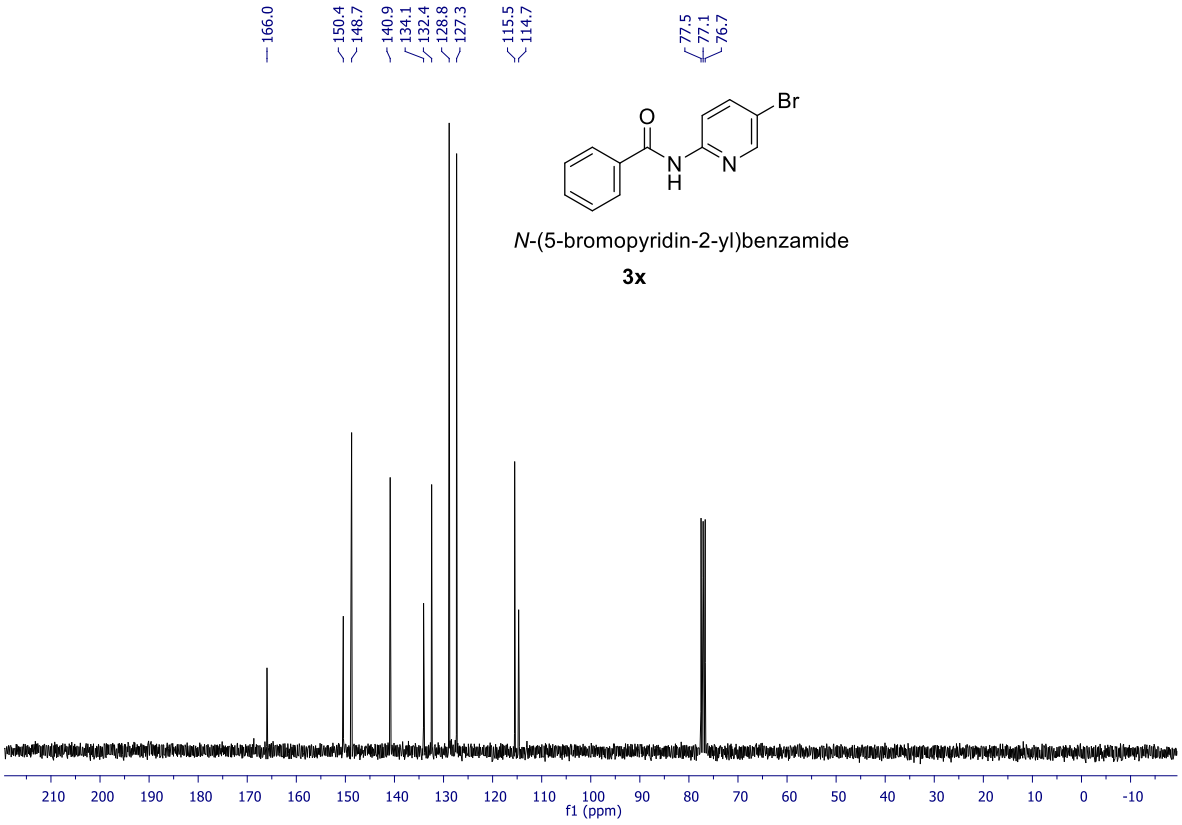
N-(5-chloropyridin-2-yl)benzamide

3w



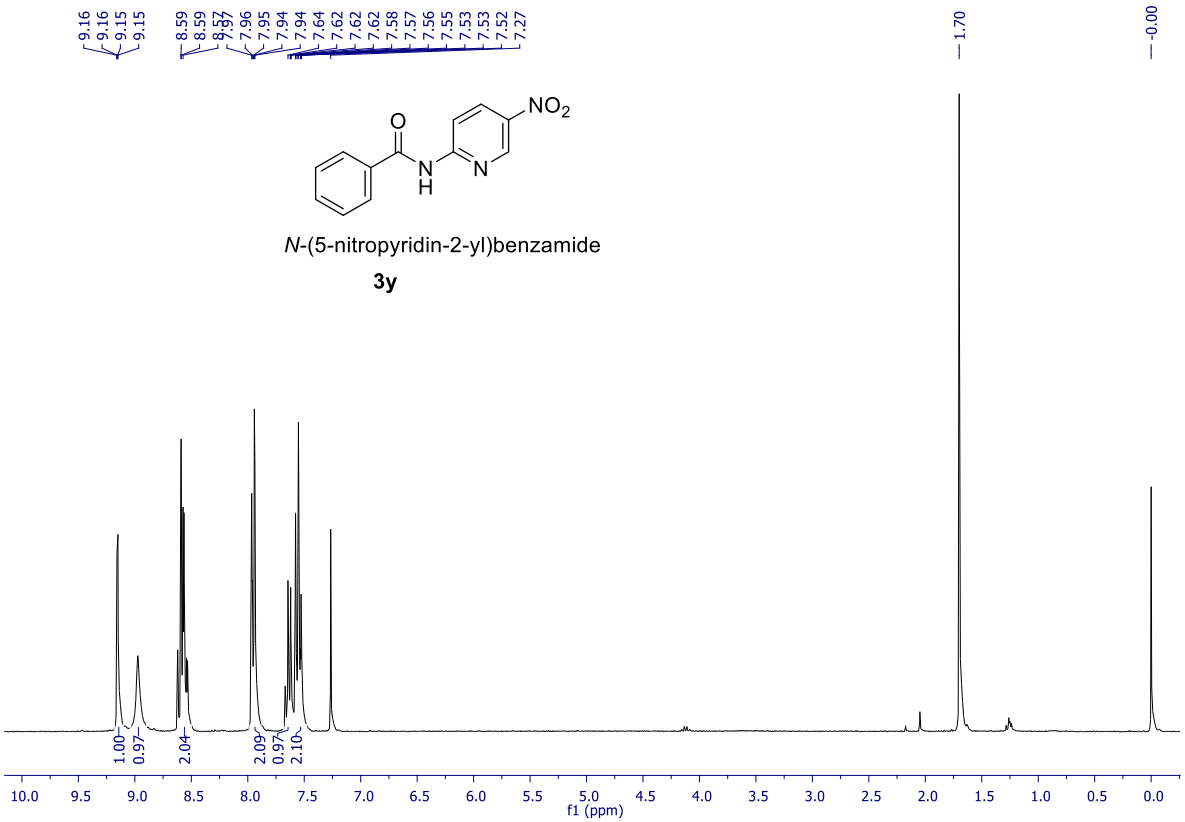
N-(5-bromopyridin-2-yl)benzamide

3x



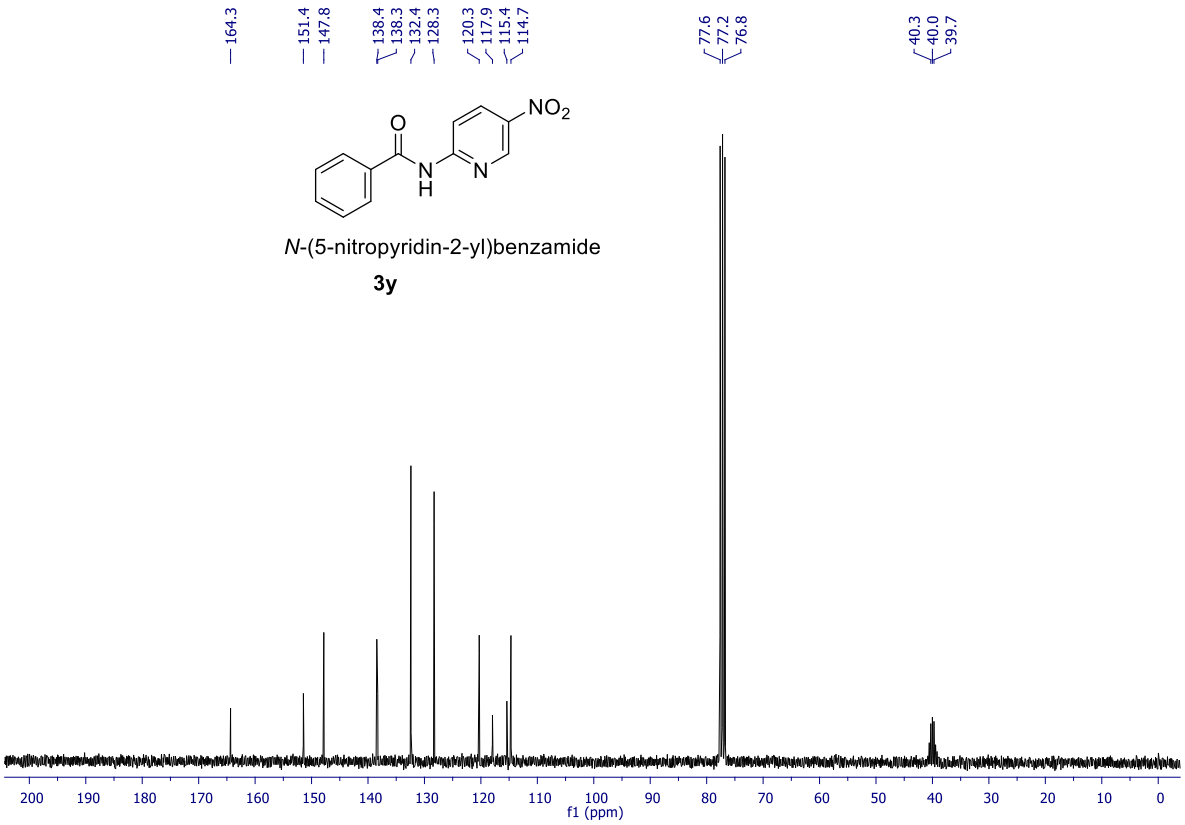
N-(5-bromopyridin-2-yl)benzamide

3x



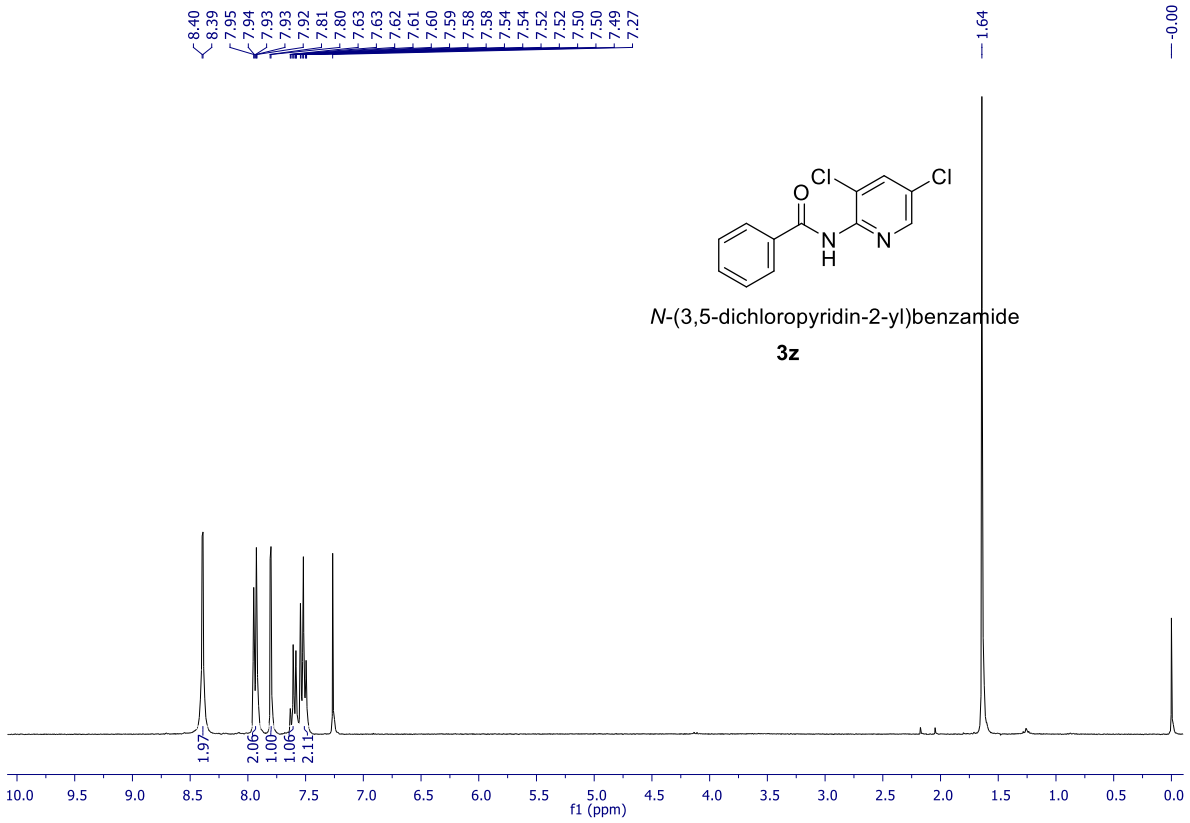
N-(5-nitropyridin-2-yl)benzamide

3y



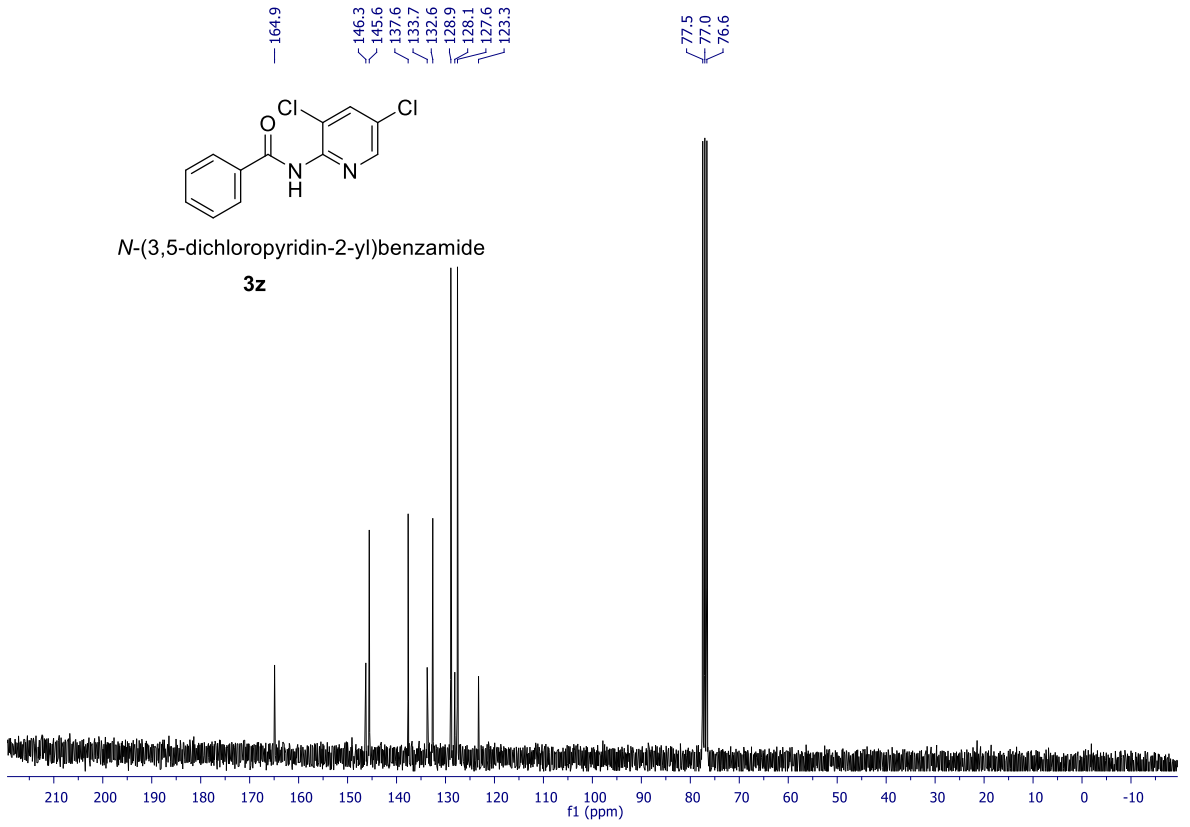
N-(5-nitropyridin-2-yl)benzamide

3y



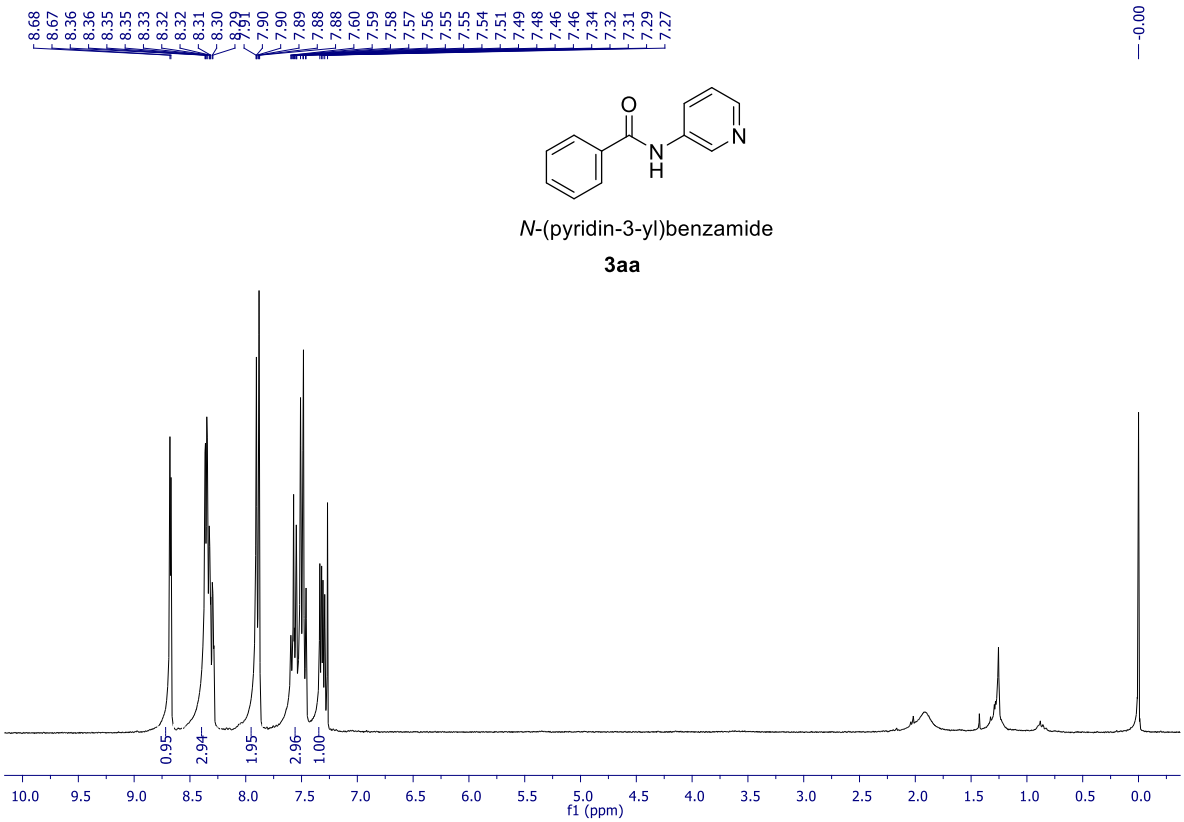
N-(3,5-dichloropyridin-2-yl)benzamide

3z



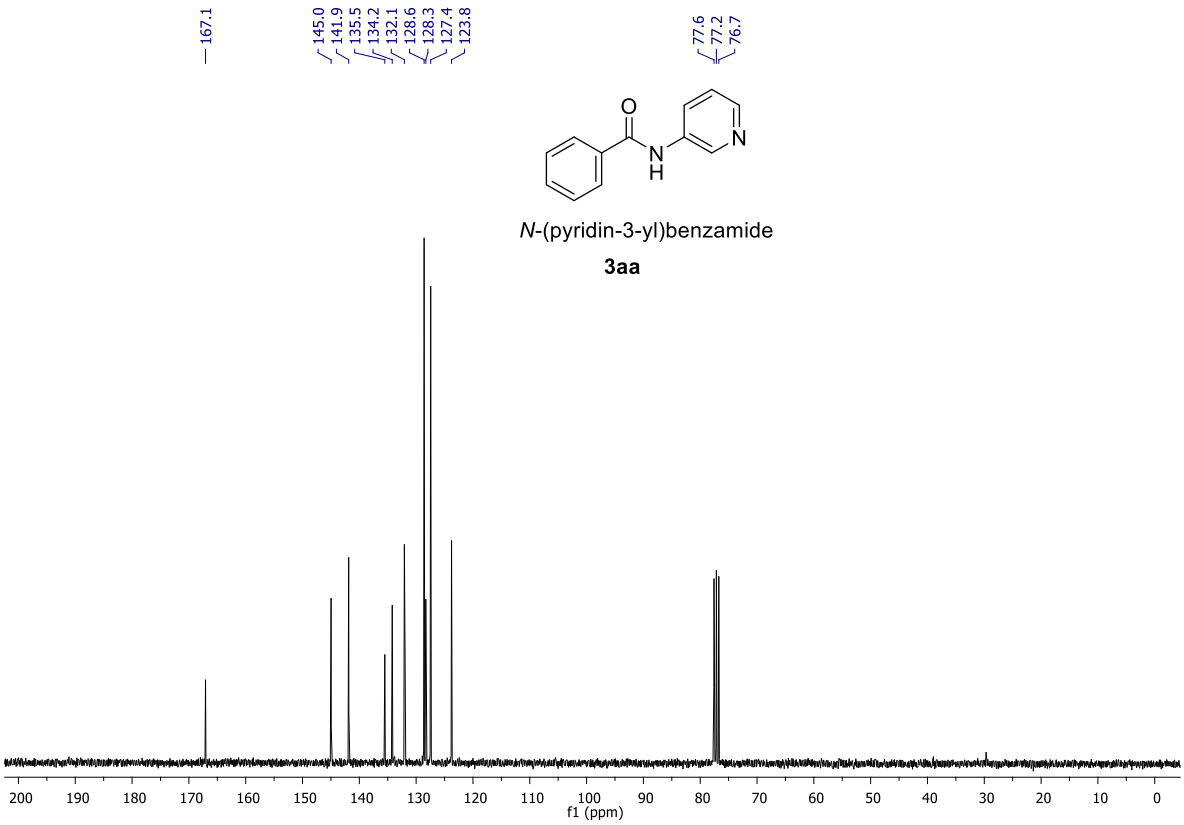
N-(3,5-dichloropyridin-2-yl)benzamide

3z



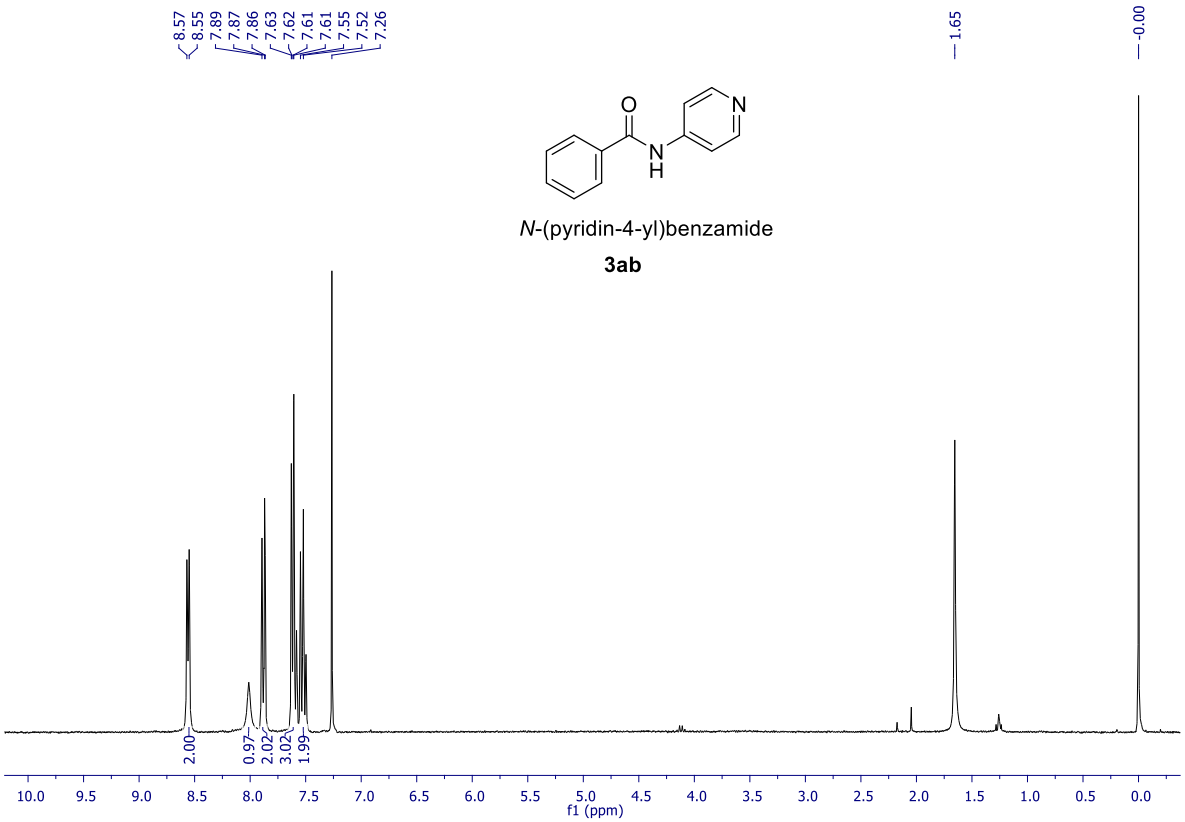
N-(pyridin-3-yl)benzamide

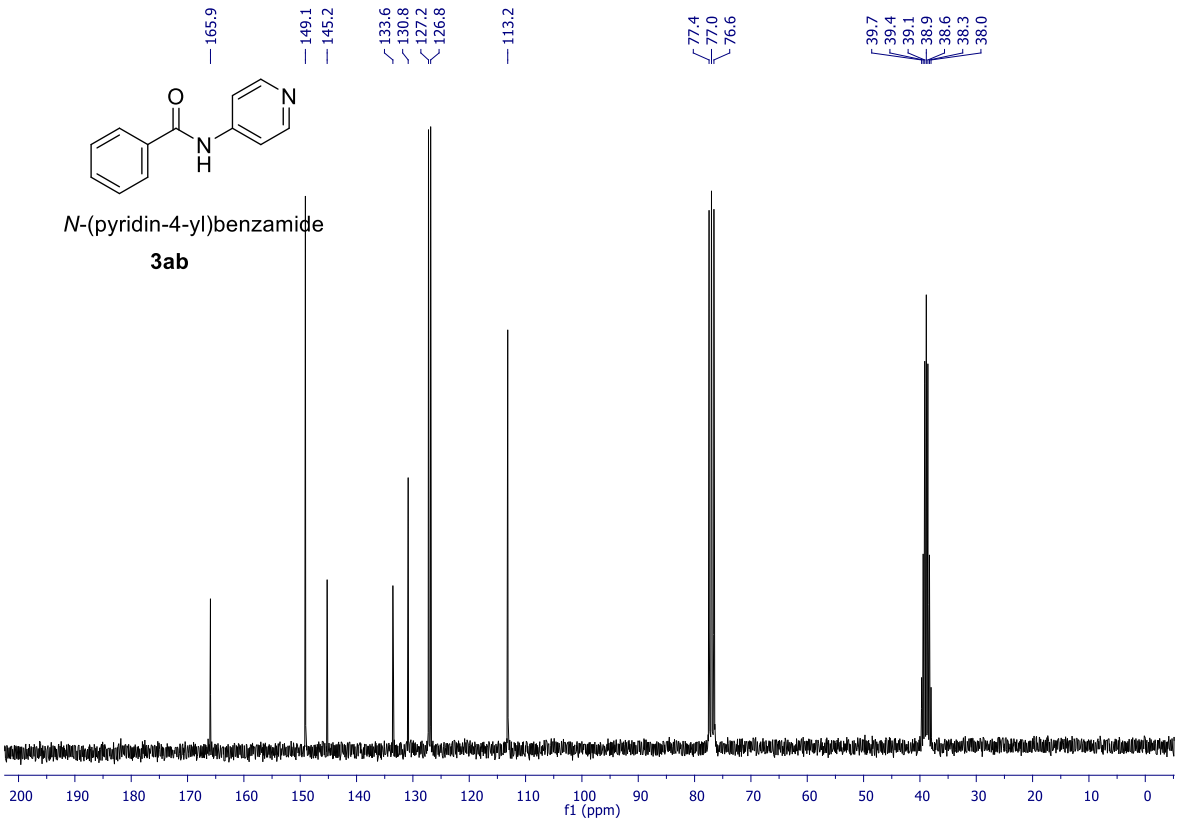
3aa

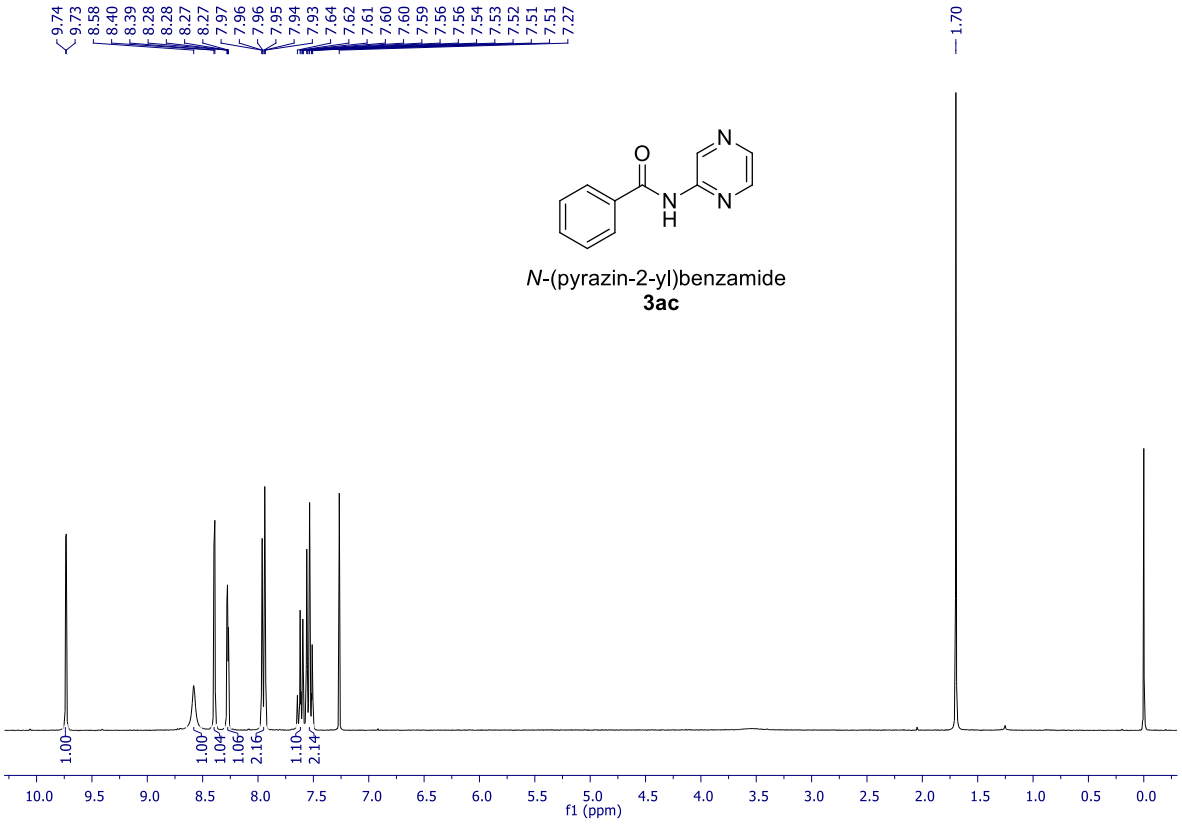


N-(pyridin-3-yl)benzamide

3aa

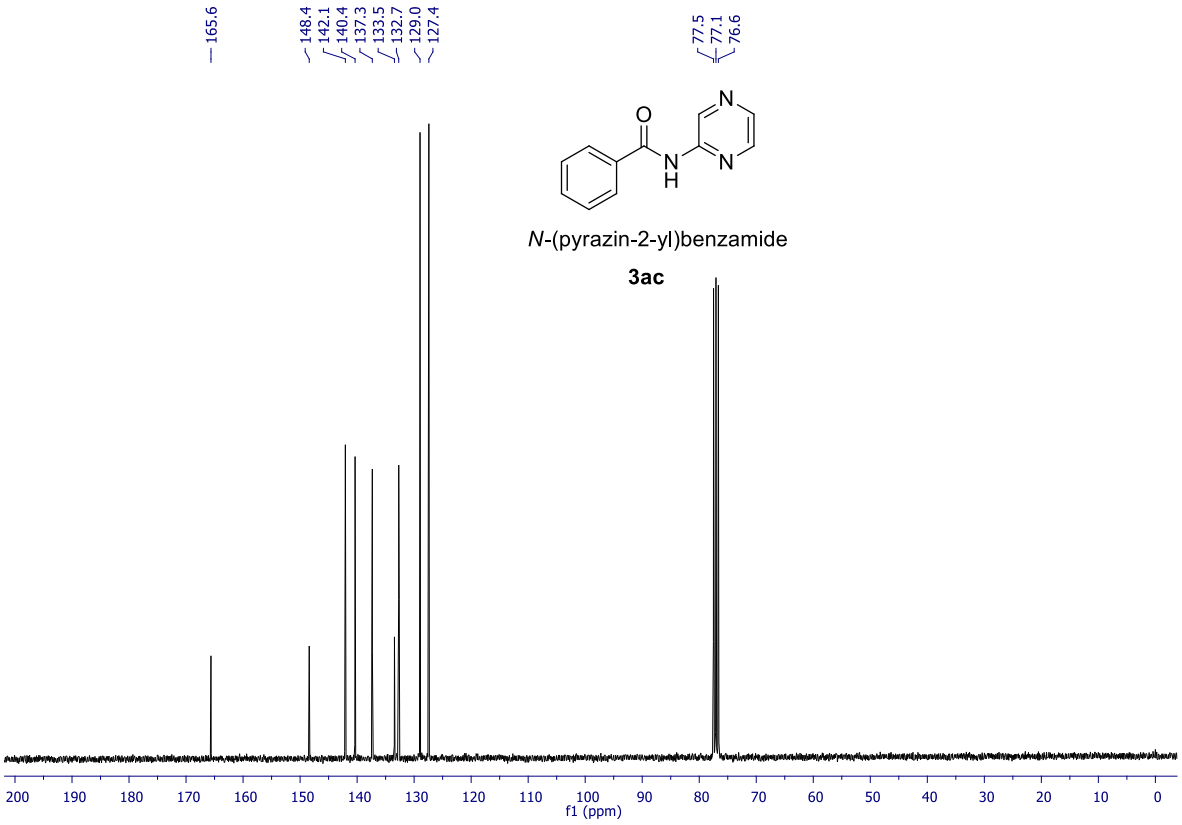

8.57
8.55
7.89
7.87
7.86
7.63
7.62
7.61
7.61
7.55
7.52
7.26
1.65
-0.00
O
N
N
H
N-(pyridin-4-yl)benzamide
3ab
2.00
0.97
2.02
3.02
1.99
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


165.9
149.1
145.2
133.6
130.8
127.2
126.8
113.2
77.4
77.0
76.6
39.7
39.4
39.1
38.9
38.6
38.3
38.0
O
N
N
H
N-(pyridin-4-yl)benzamide
3ab
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



N-(pyrazin-2-yl)benzamide

3ac



N-(pyrazin-2-yl)benzamide

3ac

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