KOH-promoted cascade C-Cl bond activation and amidation of trichloromethyl aromatic compounds with formamides in water

Chenyu Wang,[a] Yan Wang, Jianglong Wu, Qian Hu, Hui Luo, ZhongJie Wang, Yingxin Wang, Dian*J*un Li[a] *, Jun Liang[a] *, Jinhui Yang[a] *

[a] School of Chemistry and Chemical Engineering, Ningxia University, Yinchuan, PR China. E-mail: yang_Jh@nxu.edu.cn

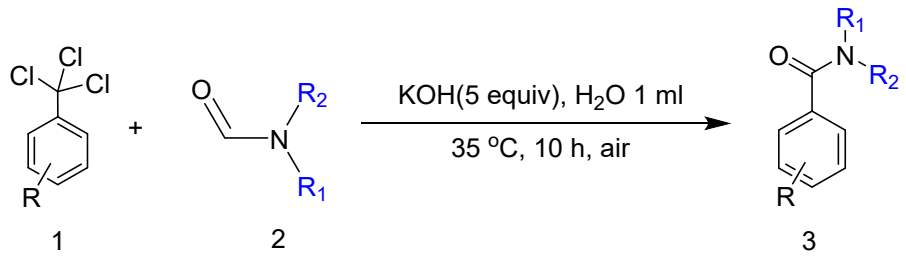
Supporting Imformation

Table of contents **Page**

1. General information

Unless otherwise noted, all reactions were carried out in quartz tubes under air atmosphere. ^{1}H NMR spectra were recorded on a Bruker AVANCE III 400 spectrometer at room temperature. Chemical shifts (ppm) were referenced to tetramethylsilane (TMS, δ = 0 ppm) in $CDCl_3$ as an internal standard. ^{13}C NMR spectra and ^{19}F NMR spectra were obtained by the same NMR spectrometer and were calibrated with $CDCl_3$ (δ = 77.00 ppm). Data for ^{1}H NMR were reported as follows: chemical shifts (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet or unresolved, br s = broad singlet), coupling constant (Hz) and integration. Data for ^{13}C NMR were reported in terms of chemical shift and multiplicity where appropriate. High-Resolution Mass Spectrometry (HRMS) were performed on a Thermo Fisher LTQ Orbit rap XL. Melting points were measured on SGW X-4 melting point apparatus and uncorrected. Anhydrous solvents were from J&K Scientific Ltd and dried by standard procedures. All other commercially available reagents were from Innochem Chemicals and used as received. Flash chromatography was carried out with silica gel (200-300 mesh). Analytical TLC was performed with silica gel GF254 plates, and the products were visualized by UV detection. All commercially available chemicals were used as received without further purification. Formamide substrates are mostly purchased directly from reagent platforms, The formamides used in 3h and 3q were prepared according to the literature [1, 2].

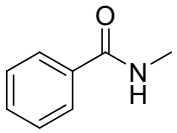
2. General procedure for synthesis of compound 3:



1 2 3

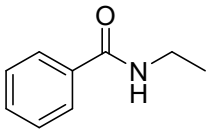
To a 10 mL quartz tube was charged with trichloromethyl aromatic compounds **1** (0.3 mmol, 1.0 equiv.), formamid compounds 2 (1.2 mmol, 4.0 equiv.), KOH (1.5 mmol, 5.0 equiv.) and H_2O (1 mL). Then the mixture was stirred for 10 h under air atmosphere at 35 ℃. After completion, the mixture was extracted with dichloromethane (10 mL × 3). The combined organic layers were dried over Na_2SO_4, and concentrated under reduced pressure. The residue was purified by chromatography on silica gel, eluting with the mixture of ethyl acetate/petroleum ether to give product(s) **3**.

3. Spectroscopic data for the products 3



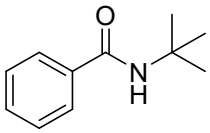
N-methylbenzamide (3a)

White solid; 40 mg, 99% yield, m. p.: 80−81 °C; **^{1}H NMR (400 MHz, Chloroform-d)** δ 7.77 (s, 2H), 7.40 (m, 3H), 6.53(s, 1H), 2.99 (s, 3H); **^{13}C NMR (101 MHz, Chloroform-d)** δ 168.3, 134.5, 131.3, 128.5, 126.8, 26.8. **HRMS (ESI)**: m/z calcd for C_8H_9NO [M + H^+]: 136.0757, found: 136.0755.



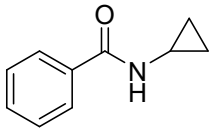
N-ethylbenzamide (3b)

White solid; 44 mg, 99% yield, m. p.: 70−71 °C; d; **^{1}H NMR (400 MHz, Chloroform-d)** δ 7.76 (d, *J* = 7.6 Hz, 2H), 7.44 (t, *J* = 7.2 Hz, 1H), 7.36 (t, *J* = 7.2 Hz, 2H), 6.55 (s, 1H), 3.48 – 3.42 (m, 2H), 1.21 (t, *J* = 7.2 Hz, 3H); **^{13}C NMR (101 MHz, Chloroform-d)** δ 167.5, 134.7, 131.1, 128.4, 126.8, 34.8, 14.8, 14.7. **HRMS (ESI)**: m/z calcd for $C_9H_{15}NO$ [M + H^+]: 150.0913, found: 150.0911.



N-(tert-butyl)benzamide (3c)

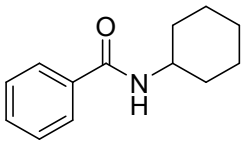
White solid; 38 mg, 71% yield, m. p.: 172−174 °C; **^{1}H NMR (400 MHz, Chloroform-d)** δ 7.70 (d, *J* = 7.6 Hz, 2H), 7.43 (t, *J* = 7.2 Hz, 1H), 7.37 (t, *J* = 7.2 Hz, 2H), 6.04 (s, 1H), 1.45 (s, 9H) ; **^{13}C NMR (101 MHz, Chloroform-d)** δ 166.8, 135.8, 130.9, 128.3, 126.6, 51.5, 28.7. **HRMS (ESI)**: m/z calcd for $C_{11}H_{15}NO$ [M + H^+]: 178.1226, found: 178.1224.



N-cyclopropylbenzamide (3d)

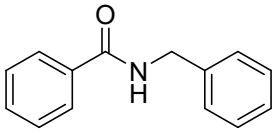
Colorless oil; 43 mg, 90% yield, R_f = 0.3 (petroleum ether : EtOAc = 10 : 1); **^{1}H NMR (400 MHz, Chloroform-d)** δ 7.74 (d, *J* = 7.2 Hz, 2H), 7.45 (t, *J* = 7.2 Hz, 1H), 7.37 (t, *J* = 7.6 Hz, 2H), 6.61 (s, 1H), 2.89 – 2.84(m, 1H), 0.81 (t, *J* = 6.0 Hz, 2H), 0.63 – 0.61 (m, 2H).; **^{13}C NMR (101 MHz, Chloroform-d)** δ 169.1, 134.2, 131.1,

128.1, 126.9 , 23.1, 23.0, 6.3. **HRMS (ESI)**: m/z calcd for $C_{10}H_{11}NO$ [M + H^+]: 162.0913, found: 162.0911.



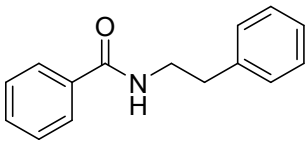
N-cyclohexylbenzamide (3e)

Colorless crystals; 54 mg, 89% yield, m. p.: 145−146 °C; **^{1}H NMR (400 MHz, Chloroform-d)** δ 7.74 (d, *J* = 7.6 Hz, 2H), 7.43 (t, *J* = 7.2 Hz, 1H), 7.35 (t, *J* = 7.2 Hz, 2H), 6.36 (s, 1H), 3.93 – 3.91 (m, 1H), 1.96 (d, *J* = 10 Hz, 2H), 1.72 – 1.69 (m, 2H), 1.62 – 1.59 (m, 1H), 1.40 – 1.12 (m, 5H); **^{13}C NMR (101 MHz, Chloroform-d)** δ 166.6, 134.9, 131.0, 128.3, 126.8, 48.6, 33.0, 25.4, 24.8. **HRMS (ESI)**: m/z calcd for C13H17NO [M + H^+]: 204.1383, found: 204.1379.



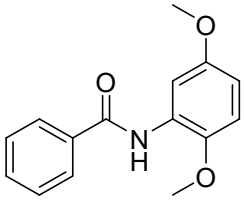
N-benzylbenzamide (3f)

White solid; 50 mg, 80% yield, m. p.: 102−105 °C; **^{1}H NMR (400 MHz, Chloroform-d)** δ 7.77 (d, *J* = 7.2 Hz, 2H), 7.44 (t, *J* = 7.2 Hz, 1H), 7.34 (t, *J* = 7.6 Hz, 2H), 7.29 – 7.23 (m, 5H), 7.01 (s, 1H), 4.55 (d, *J* = 5.6 Hz, 2H); **^{13}C NMR (101 MHz, Chloroform-d)** δ 167.4, 138.2, 134.2, 131.3, 128.5, 128.4, 127.7, 127.3, 126.9, 43.8. **HRMS (ESI)**: m/z calcd for $C_{14}H_{13}NO$ [M + H^+]: 212.1070, found: 212.1067.



N-phenethylbenzamide (3g)

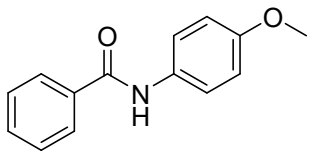
white solid; 49 mg, 72% yield, m. p.: 132−136 °C; **^{1}H NMR (400 MHz, DMSO-d6)** δ 7.69 (t, *J* = 8 Hz, 2H), 7.47 – 7.39 (m, 1H), 7.37 (t, *J* = 8 Hz, 2H), 7.30 (t, *J* = 7.2Hz, 2H), 7.24 – 7.20 (m, 3H), 6.48 (s, 1H), 3.70 – 3.65 (m, 2H) ,2.91 (t, *J* = 7.2 Hz, 2H); **^{13}C NMR (101 MHz, DMSO-d6)** δ 167.5, 138.8, 134.5, 131.3, 128.7, 128.6, 128.4, 126.8, 126.4, 41.1, 35.6. **HRMS (ESI)**: m/z calcd for $C_{15}H_{15}NO$ [M + H^+]: 226.1226, found: 226.1221.



N-(2,5-dimethoxyphenyl)benzamide (3h)[1]

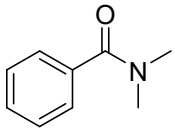
White solid; 22 mg, 28% yield, m. p.: 141 −145 °C; **^{1}H NMR (400 MHz,**

Chloroform-d) δ 8.60 (s, 1H), 8.30 (s, 1H), 7.89 (s, 2H), 7.49 (s, 3H), 6.81 (s, 1H), 6.61 (s, 1H), 3.86 (s, 3H) , 3.81 (s, 3H); **^{13}C NMR (101 MHz, Chloroform-d)** δ 164.9, 153.7, 142.2, 134.9, 131.6, 128.6, 128.2, 126.8, 110.4, 108.4, 105.8, 56.1, 56.0, 55.6, 55.5. **HRMS (ESI)**: m/z calcd for $C_{15}H_{15}NO_3$ [M + H^+]: 258.1125, found: 258.1122.



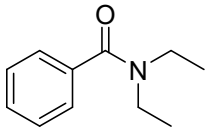
N-(4-methoxyphenyl)benzamide (3i)

Grayish solid; 22 mg, 32% yield, m. p.: 171−174 °C; **^{1}H NMR (400 MHz, DMSO-d6)** δ 10.15 (s, 1H), 7.96 (d, *J* = 6.8 Hz, 2H), 7.79 (d, *J* = 9.2 Hz, 2H), 7.60 – 7.56 (m, 1H), 7.52 (t, *J* = 7.2 Hz, 2H), 6.96 – 6.92 (m, 2H), 3.75 (s, 3H); **^{13}C NMR (101 MHz, DMSO-d6)** δ 165.6, 156.0, 135.5, 132.7, 131.8, 128.8, 128.0, 122.4, 114.2, 55.7, 55.6. **HRMS (ESI)**: m/z calcd for $C_{14}H_{13}NO_2$ [M + H^+]: 228.1019, found: 228.1016.



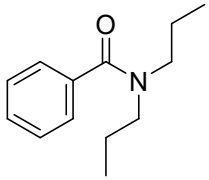
N,N-dimethylbenzamide (3J)

Yellow oil; 44mg, 98% yield, R_f = 0.3 (petroleum ether : EtOAc = 2 : 1); **^{1}H NMR (400 MHz, Chloroform-d)** δ 7.30 – 7.29 (m, 5H), 3.00 (s, 3H), 2.85 (s, 3H).; **^{13}C NMR (101 MHz, Chloroform-d)** δ 171.4, 136.1, 129.2, 128.1, 126.8, 39.3, 35.0. **HRMS (ESI)**: m/z calcd for $C_9H_{11}NO$ [M + H^+]: 150.0913, found: 150.0911.



N,N-diethylbenzamide (3k)

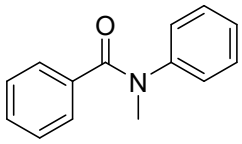
Yellow oil; 52 mg, 98% yield, R_f = 0.2 (petroleum ether : EtOAc = 5 : 1); **^{1}H NMR (400 MHz, Chloroform-d)** δ 7.37 (s, 5H), 3.54 (s, 2H), 3.26 – 3.25 (m, 2H), 1.24 (s, 3H),1.12 – 1.10 (m, 3H); **^{13}C NMR (101 MHz, Chloroform-d)** δ 171.7, 137.4, 128.9, 128.3, 126.4, 50.6, 46.2, 21.9, 20.7; **HRMS (ESI)**: m/z calcd for $C_{11}H_{15}NO$ [M + H^+]: 178.1226, found: 178.1223.



N,N-dipropylbenzamide (3l)

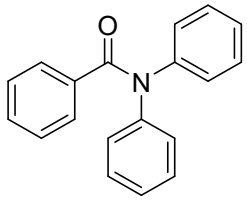
Yellow solid; 26 mg, 43% yield, m. p.: 44−46 °C; **^{1}H NMR (400 MHz, Chloroform-**

d) δ 7.36 (s, 5H), 3.45 (s, 2H), 3.15 (s, 2H), 1.68 (s, 2H), 1.51 (s, 2H), 0.97 (s, 3H), 0.73 (s, 3H); **^{13}C NMR (101 MHz, Chloroform-d)** δ171.7, 137.4, 128.9, 128.3, 126.4, 50.6, 46.2, 21.9, 20.7, 11.4, 11.0. **HRMS (ESI)**: m/z calcd for $C_{13}H_{19}NO$ [M + H^+]: 206.1539, found: 206.1536.



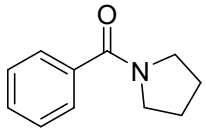
N-methyl-N-phenylbenzamide (3m)

White solid; 51 mg, 80% yield, m. p. 160 −164 °C; **^{1}H NMR (400 MHz, Chloroform-d)** δ 7.29 (s, 2H), 7.21 – 7.14 (m, 6H), 7.03 (s, 2H), 3.45 (s, 3H); **^{13}C NMR (101 MHz, Chloroform-d)** δ 170.5, 144.7, 135.7, 129.4, 129.0, 128.5, 127.5, 126.7, 126.3, 38.23.**HRMS (ESI)**: m/z calcd for $C_{14}H_{13}NO$ [M + H^+]: 212.1070, found: 212.1068.



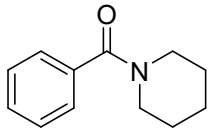
N,N-diphenylbenzamide（3n）

Yellow solid; 63 mg, 77% yield, m. p.: 170−173 °C; **^{1}H NMR (400 MHz, Chloroform-d)** δ 7.95 (d, *J* = 5.6 Hz, 6H), 7.44 (s, 9H); **^{13}C NMR (101 MHz, Chloroform-d)** δ 144.2, 130.3, 128.3, 125.5, 97.7.**HRMS (ESI)**: m/z calcd for $C_{19}H_{15}NO$ [M + H^+]: 274.1226, found: 274.1225.



phenyl(pyrrolidin-1-yl)methanone (3o)

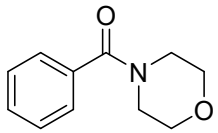
Colorless oil; 51 mg, 97% yield, R_f = 0.3 (petroleum ether : EtOAc = 5 : 1); **^{1}H NMR (400 MHz, Chloroform-d)** δ 7.46 (t, *J* = 3.6 Hz, 2H), 7.33 (t, *J* = 4.4 Hz, 3H), 3.59 (s, 2H), 3.36 (s, 2H), 1.88 (d, *J* = 6 Hz, 2H), 1.81 (d, *J* = 5.6 Hz, 2H); **^{13}C NMR (101 MHz, Chloroform-d)** δ 169.57, 137.2, 129.6, 128.1, 127.0, 49.5, 46.1, 26.3, 24.4. **HRMS (ESI)**: m/z calcd for $C_{11}H_{13}NO$ [M + H^+]: 176.1070, found: 176.1066.



phenyl(piperidin-1-yl)methanone (3p)

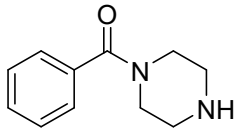
Yellow solid; 52 mg, 93% yield, m. p.: 48−50 °C; **^{1}H NMR (400 MHz, Chloroform-**

d) δ 7.32 (s, 5H), 3.65 (s, 2H), 3.27 (s, 2H), 1.60 (s, 4H) , 1.44 (s, 2H); **^{13}C NMR (101 MHz, Chloroform-d)** δ 169.9, 136.2, 129.0, 128.0, 126.4, 48.4, 42.7, 26.2, 25.3, 24.2. **HRMS (ESI)**: m/z calcd for $C_{12}H_{15}NO$ [M + H^+]: 190.1226, found: 190.1223.



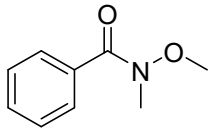
morpholino(phenyl)methanone (3q)[2]

White solid; 42 mg, 74% yield, m. p.: 62−68 °C; **^{1}H NMR (400 MHz, Chloroform-d)** δ 7.47-7.45 (m, 2H), 7.34-7.32 (m, 3H), 3.76 (s, 4H), 3.62 (s, 2H), 3.44 (s, 2H); **^{13}C NMR (101 MHz, Chloroform-d)** δ 170.4, 135.2, 129.8, 128.5, 127.0, 66.8. **HRMS (ESI)**: m/z calcd for $C_{11}H_{13}NO_2$ [M + H^+]: 192.1019, found: 192.1016.



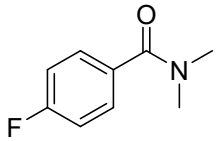
phenyl(piperazin-1-yl)methanone (3r)

White solid; 47 mg, 82% yield, m. p.: 62−65 °C; **^{1}H NMR (400 MHz, Chloroform-d)** δ 3.64 (s, 5H), 3.71 (s, 2H), 3.35 (s, 2H), 2.90(s, 2H), 2.76 (s, 2H), 1.78 (s, 1H); **^{13}C NMR (101 MHz, Chloroform-d)** δ 170.0, 135.5, 129.2, 128.1, 126.6, 48.6, 46.1, 45.6, 42.8. **HRMS (ESI)**: m/z calcd for $C_{11}H_{14}N_2O$ [M + H^+]: 191.1179, found: 191.1177.



N-methoxy-N-methylbenzamide (3s)

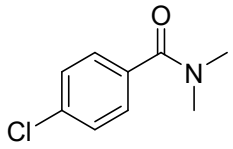
White solid; 41 mg, 83% yield, m. p.: 69−70 °C; **^{1}H NMR (400 MHz, Chloroform-d)** δ 7.63 (d, *J* = 7.2 Hz, 2H), 7.41 – 7.34 (m, 3H), 3.50 (s, 3H), 3.31 (s, 3H); **^{13}C NMR (101 MHz, Chloroform-d)** δ 169.7, 133.9, 130.4, 127.9, 127.8, 60.8, 60.8, 33.6. **HRMS (ESI)**: m/z calcd for $C_9H_{11}NO_2$ [M + H^+]: 166.0862, found: 166.0860.



4-fluoro-N,N-dimethylbenzamide (3t)

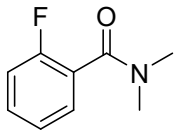
Yellow oil; 36 mg, 71% yield, R_f = 0.2 (petroleum ether : EtOAc = 2 : 1); **^{1}H NMR (400 MHz, Chloroform-d)** δ 7.33-7.31 (m, 1H), 7.25-7.20 (m, 3H), 3.07 (s, 3H), 2.79 (s, 3H); **^{13}C NMR (101 MHz, Chloroform-d)** δ 169.4, 136.3, 130.0, 129.5, 127.7, 127.1, 38.0, 34.6. **^{19}F NMR (376 MHz, Chloroform-d)** δ-110.73. **HRMS (ESI)**: m/z

calcd for $C_9H_{10}FNO$ [M + H^+]: 168.0819, found: 168.0820.



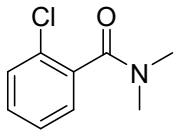
4-chloro-N,N-dimethylbenzamide (3u)

Yellow oil; 23 mg, 42% yield, R_f = 0.2 (petroleum ether : EtOAc = 2 : 1); **^{1}H NMR (400 MHz, Chloroform-d)** δ7.36 – 7.32 (m, 4H), 3.07 (s, 3H), 2.94 (s, 3H); **^{13}C NMR (101 MHz, Chloroform-d)** δ 170.4, 135.4, 134.5, 128.5, 39.4, 35.3. **HRMS (ESI)**: m/z calcd for $C_9H_{10}ClNO$ [M + H^+]: 184.0524, found: 184.0521.



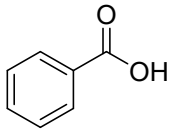
2-fluoro-N,N-dimethylbenzamide (3v)

Yellow oil; 33 mg, 67% yield, R_f = 0.2 (petroleum ether : EtOAc = 2 : 1); **^{1}H NMR (400 MHz, Chloroform-d)** δ 7.36 – 7.33 (m, 2H), 7.16 (t, *J* = 7.6 Hz, 1H), 7.05 (t, *J* = 8.8 Hz, 1H), 3.09 (s, 3H), 2.90 (s, 3H); **^{13}C NMR (101 MHz, Chloroform-d)** δ 166.7, 159.3, 156.8, 131.1, 131.0, 128.9, 128.9, 124.6, 124.5, 124.5, 124.4, 115.7, 115.5, 38.2, 34.8; **^{19}F NMR (376 MHz, Chloroform-d)** δ-116.20. **HRMS (ESI)**: m/z calcd for $C_9H_{10}FNO$ [M + H^+]: 168.0819, found: 168.0816.



2-chloro-N,N-dimethylbenzamide (3w)

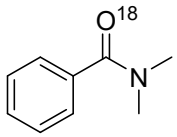
Yellow oil; 22 mg, 40% yield, R_f = 0.2 (petroleum ether : EtOAc = 2 : 1); **^{1}H NMR (400 MHz, Chloroform-d)** δ 7.41 – 7.37(m, 2H), 7.07-7.02 (m, 2H), 3.06 (s, 3H), 2.95 (s, 3H); **^{13}C NMR (101 MHz, Chloroform-d)** δ 170.5, 164.4, 161.9, 129.3, 129.2, 115.4, 115.2, 39.5, 35.4. **HRMS (ESI)**: m/z calcd for $C_9H_{10}ClNO$ [M + H^+]: 184.0524, found: 184.0525.



benzoic acid

White solid; 28 mg, 23% yield, m. p.: 121−125 °C; **^{1}H NMR (400 MHz, Chloroform-d)** δ 12.86 (s, 1H), 8.16 – 8.14 (d, *J* = 7.2 Hz, 2H), 7.63(t, *J* = 7.2 Hz, 1H), 7.49 (t, *J* = 7.6 Hz, 2H); **^{13}C NMR (101 MHz, Chloroform-d)** δ 172.5, 133.8, 130.2, 129.3, 128.5; **HRMS (ESI)**: m/z calcd for $C_7H_6O_2$ [M + H^+]: 123.0441, found:

123.0440.



N,N-dimethylbenzamide (3Ja)
Yellow oil; 44mg, 98% yield, R_f = 0.3 (petroleum ether : EtOAc = 2 : 1); **HRMS (ESI)**: m/z calcd for $C_9H_{11}NO^{18}$ [M + H^+]: 152.0957, found: 152.0956.

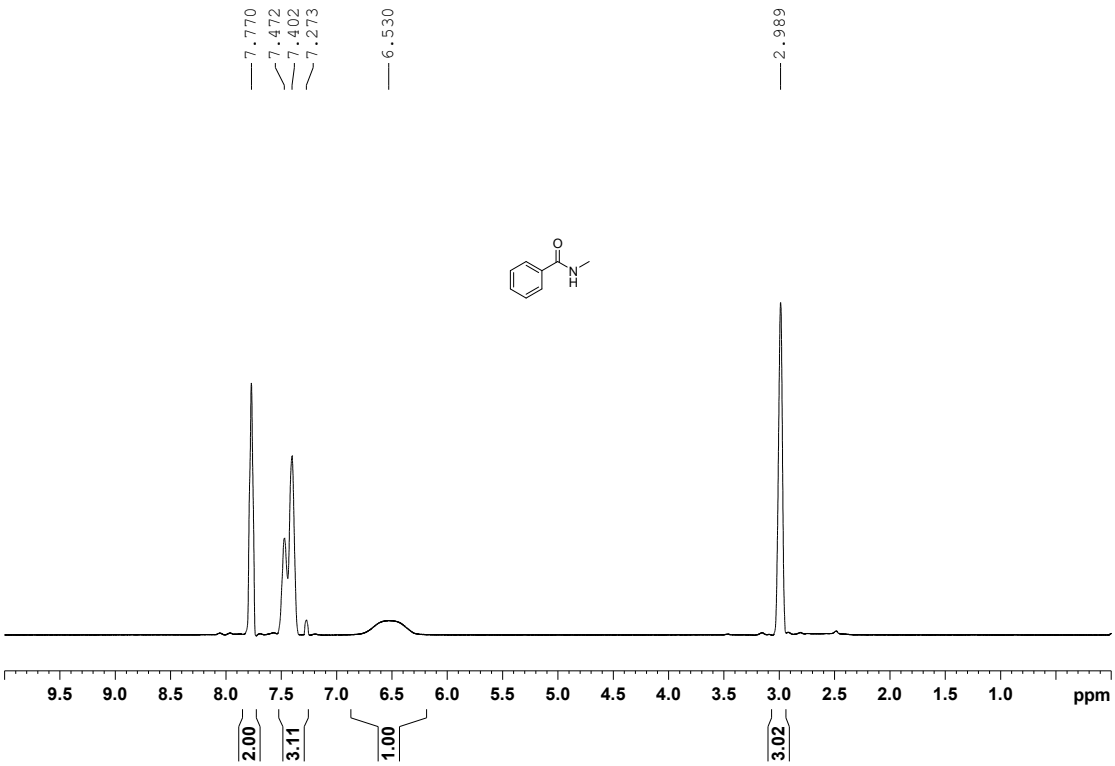
4. References

[1] K. H. Chiang, S. H. Lu, W. P. Yen, N. Uramaru, W. S. Tseng, T. W. Chang, F. F. Wong, *Heteroat Chem*, **2016**, 27(4), 235-242.

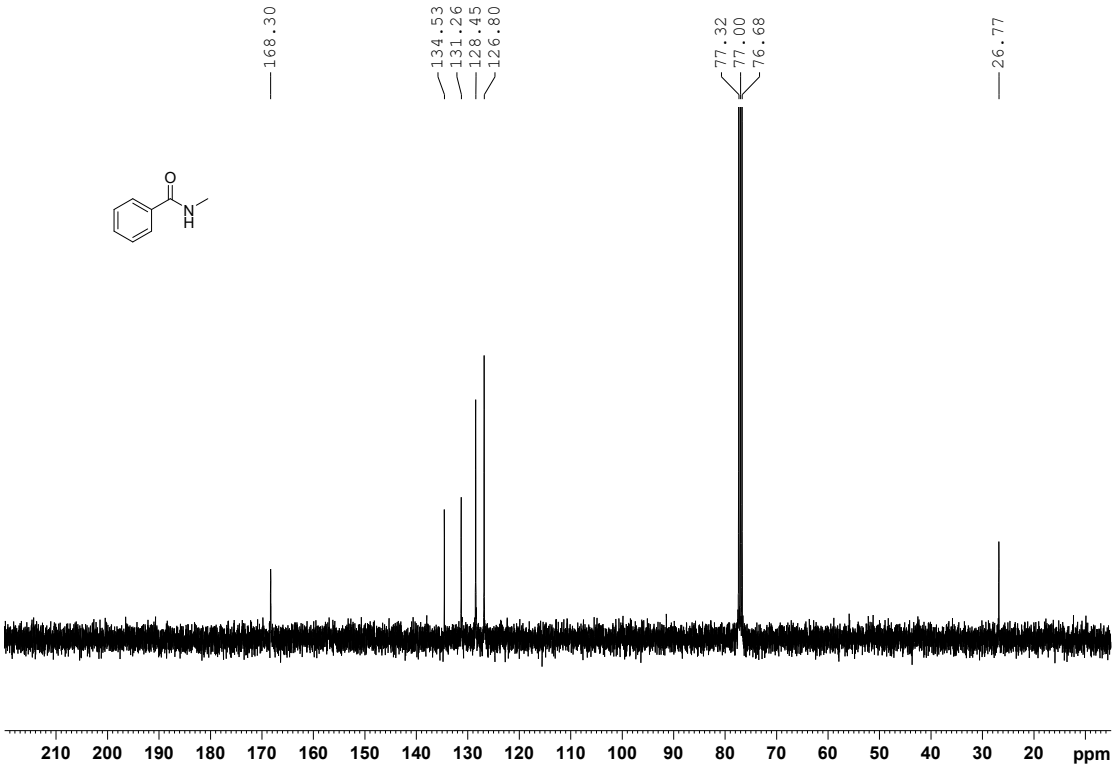
[2] C. D. Gomes, O. Jacquet. C. Villiers, P. Thuery, M. Ephritikhine, T. Cantat, *Angew. Chem. Int.* **2012,** 51, 187-190.

5. NMR spectra

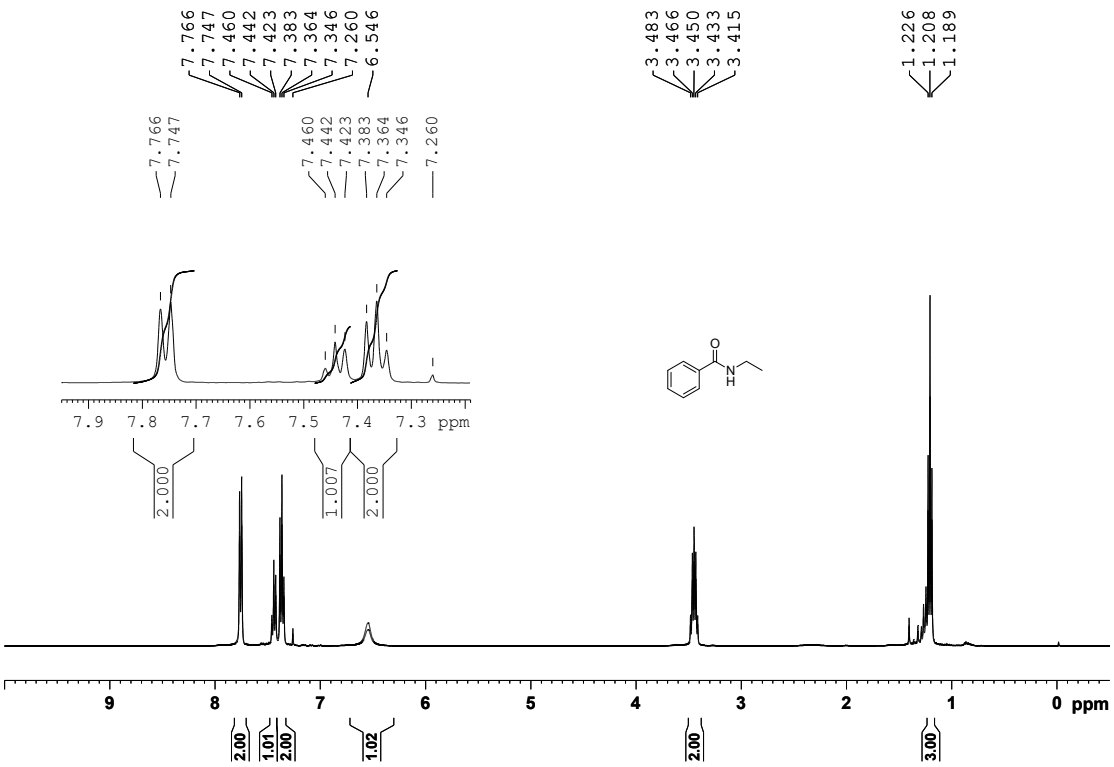
3a ^{1}H NMR (400 MHz, CDCl3)



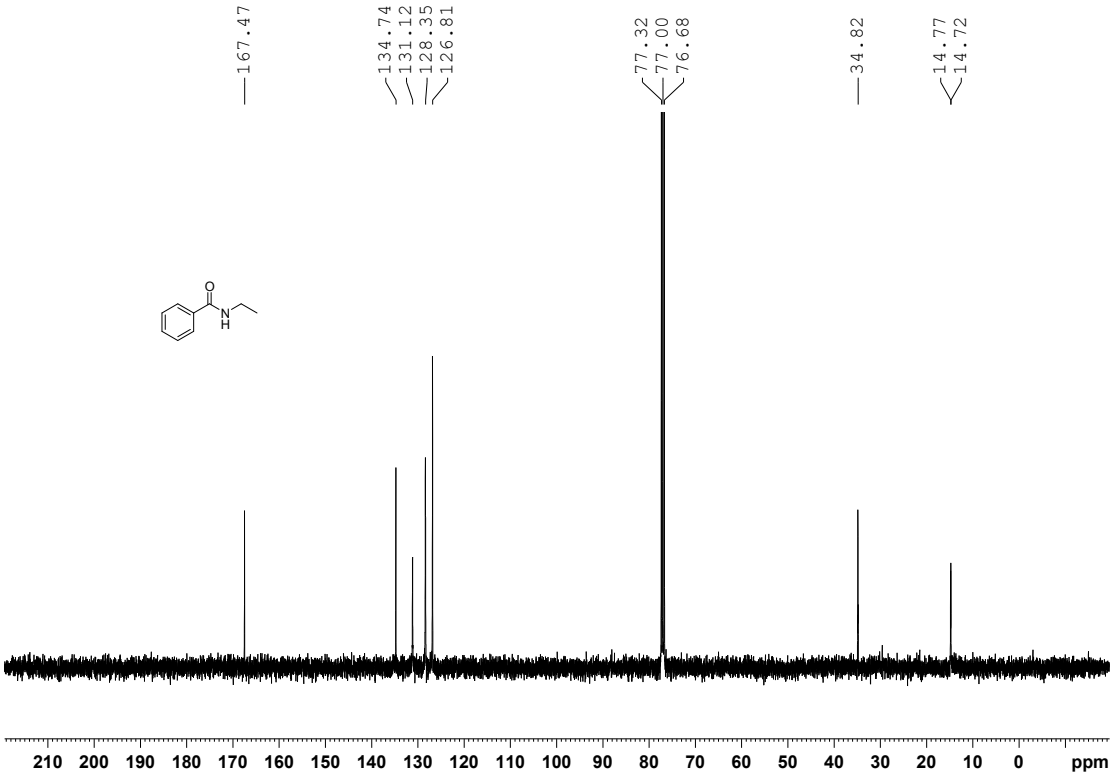
3a ^{13}C NMR (101 MHz, CDCl3)



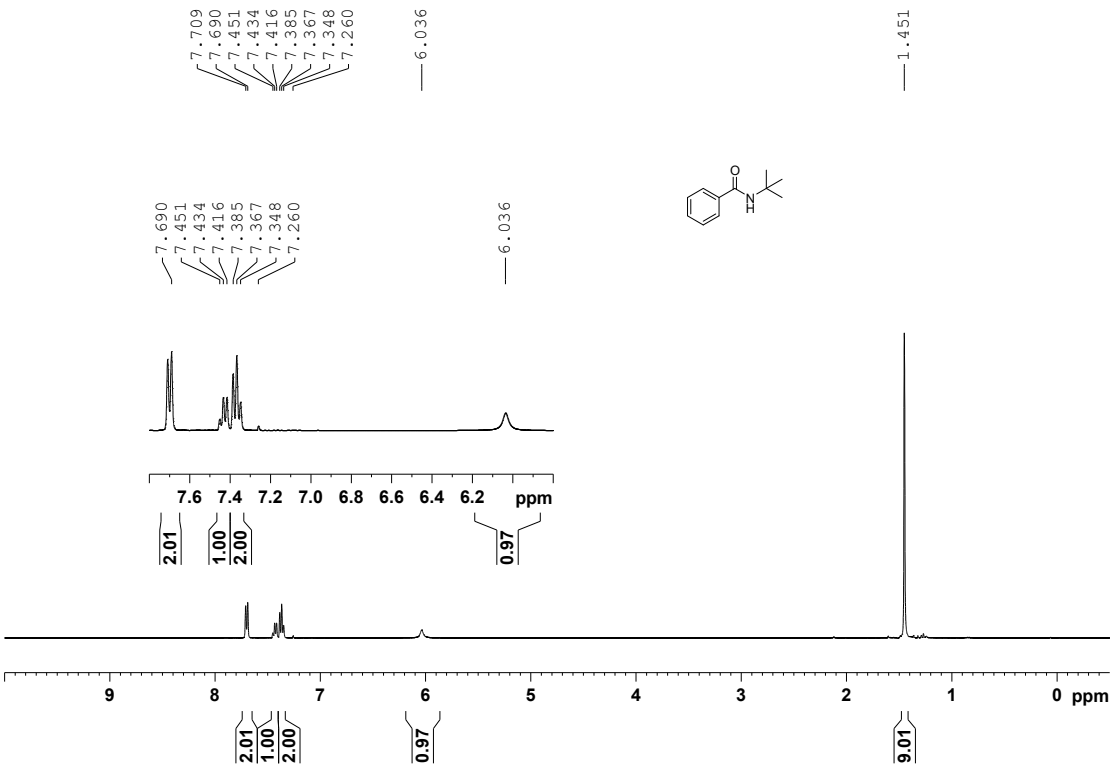
3b 1H NMR (400 MHz, CDCl3)



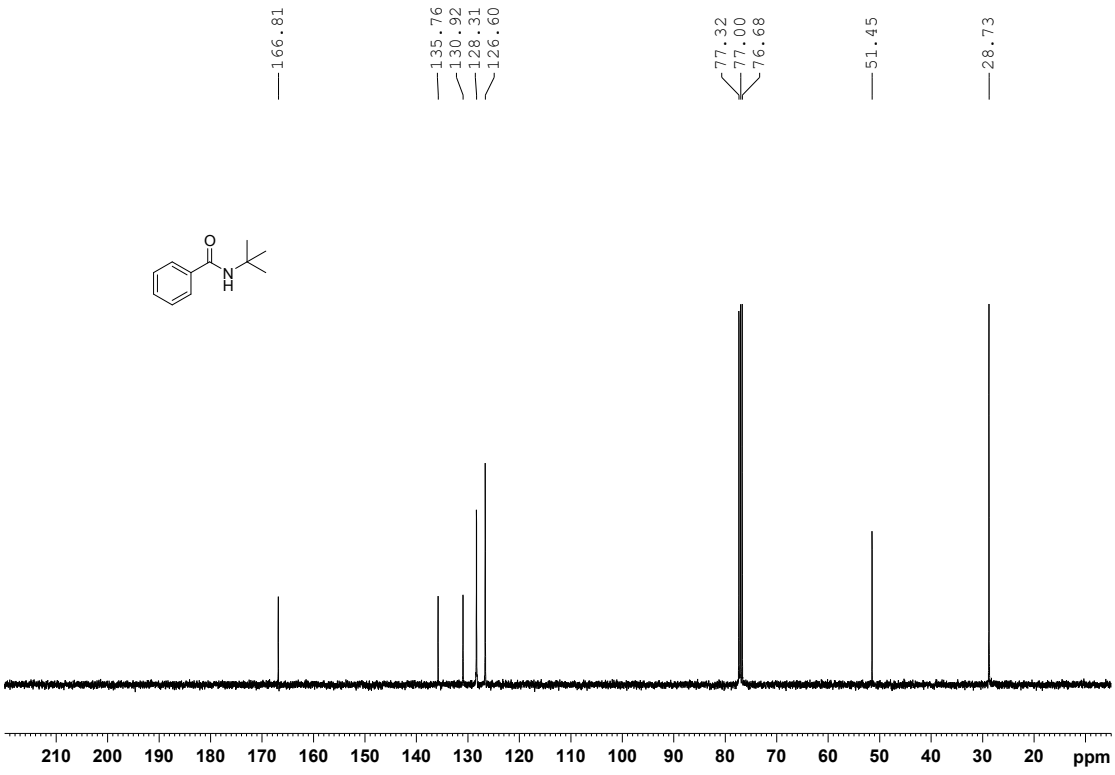
3b ^{13}C NMR (101 MHz, $CDCl_3$)



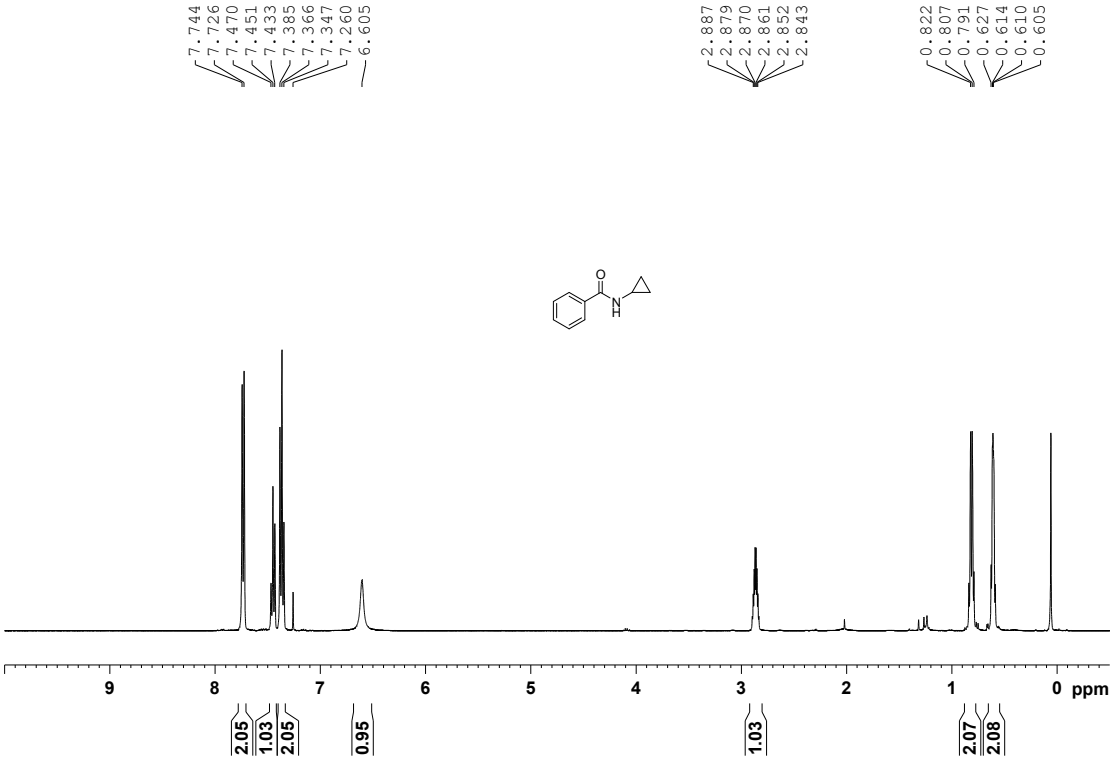
3c ^{1}H NMR (400 MHz, CDCl3)



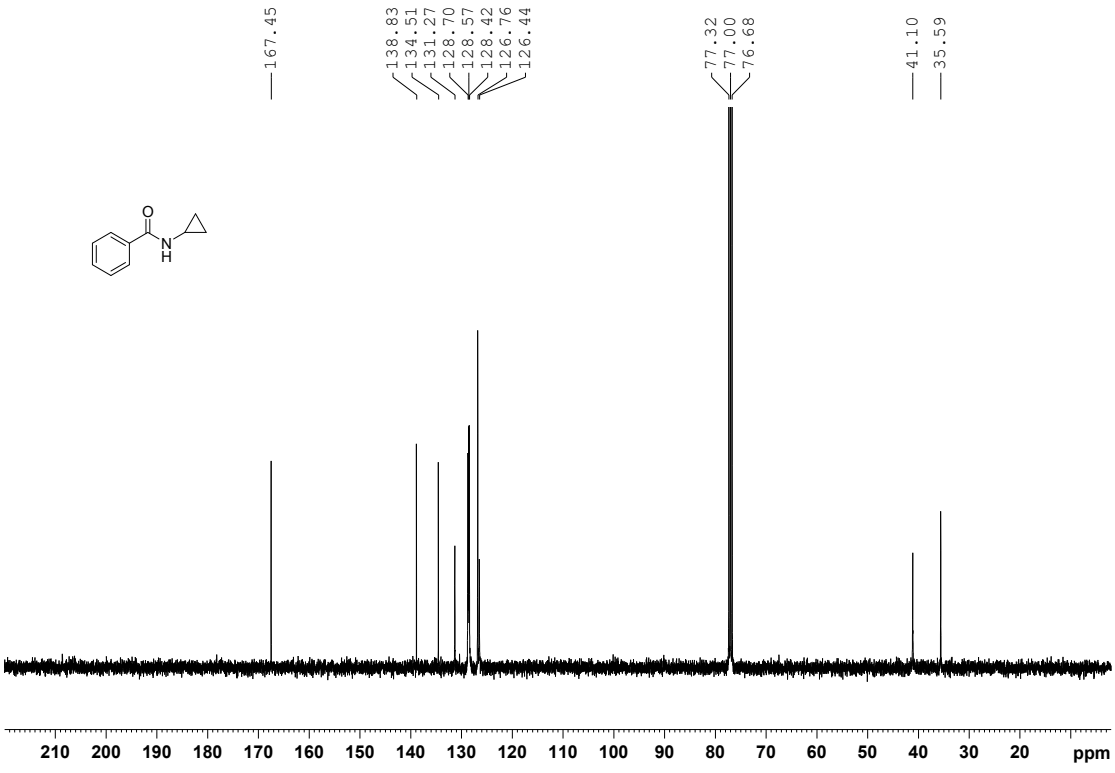
3c ^{13}C NMR (101 MHz, $CDCl_3$)



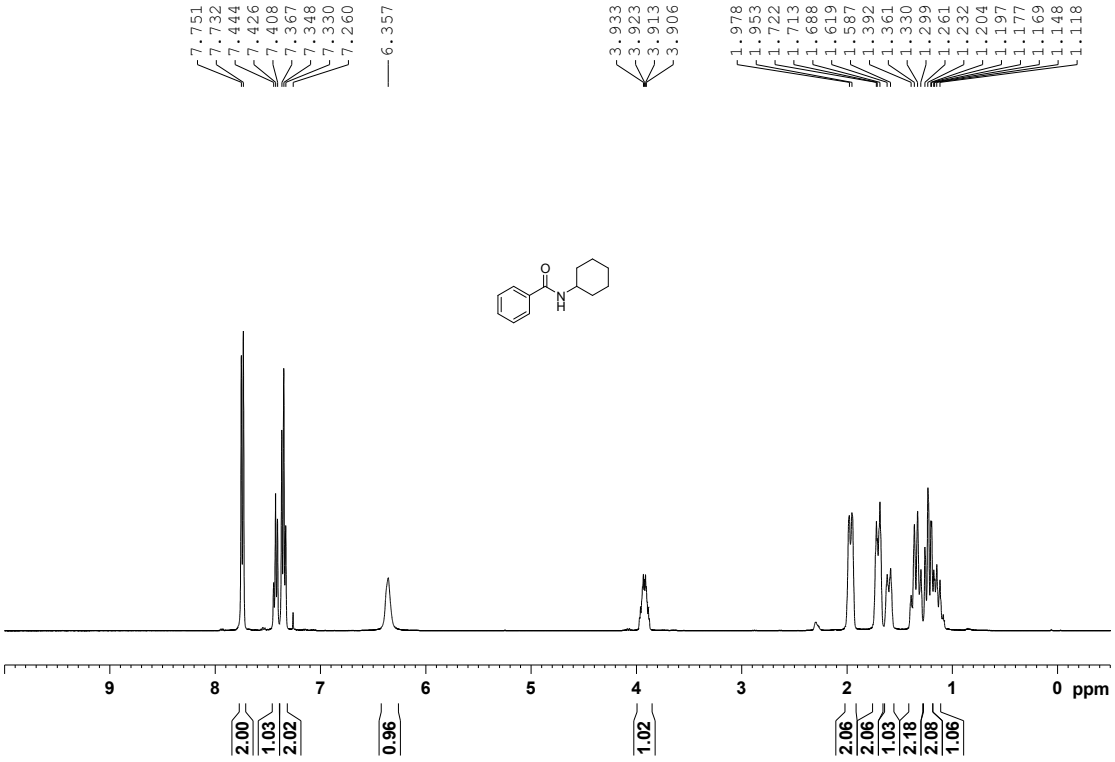
3d ^{1}H NMR (400 MHz, $CDCl_3$)



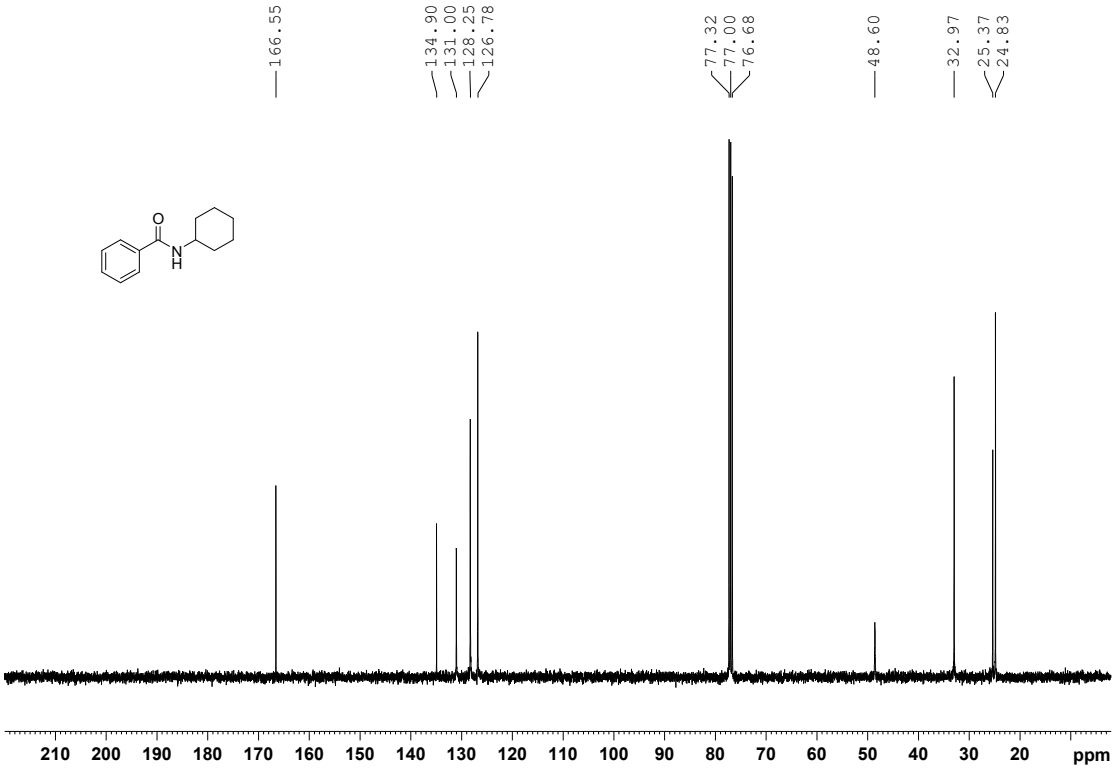
3d ^{13}C NMR (101 MHz, $CDCl_3$)



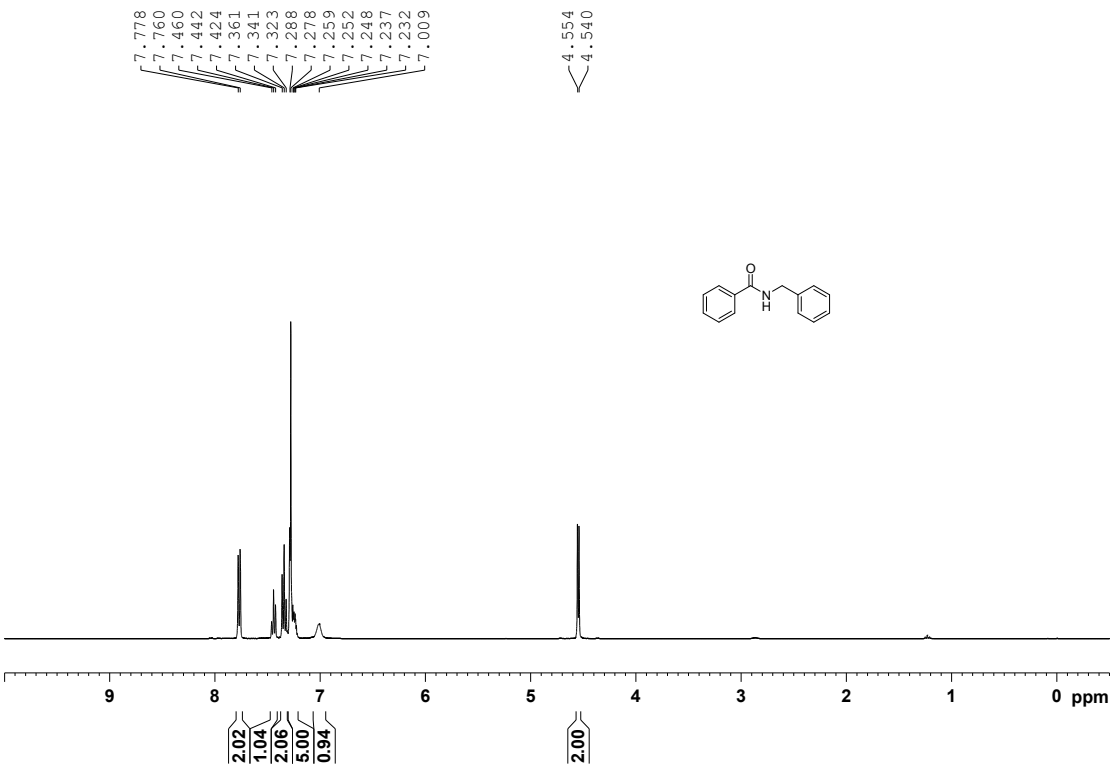
3e ^{1}H NMR (400 MHz, $CDCl_3$)



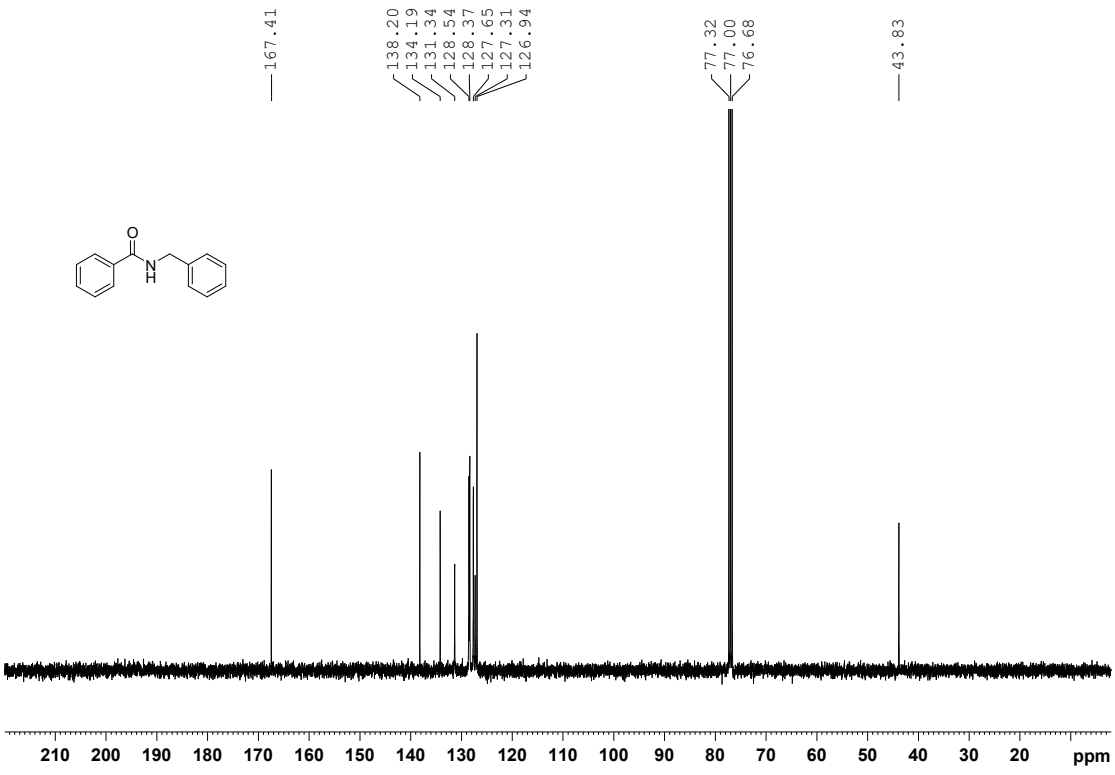
3e ^{13}C NMR (101 MHz, $CDCl_3$)



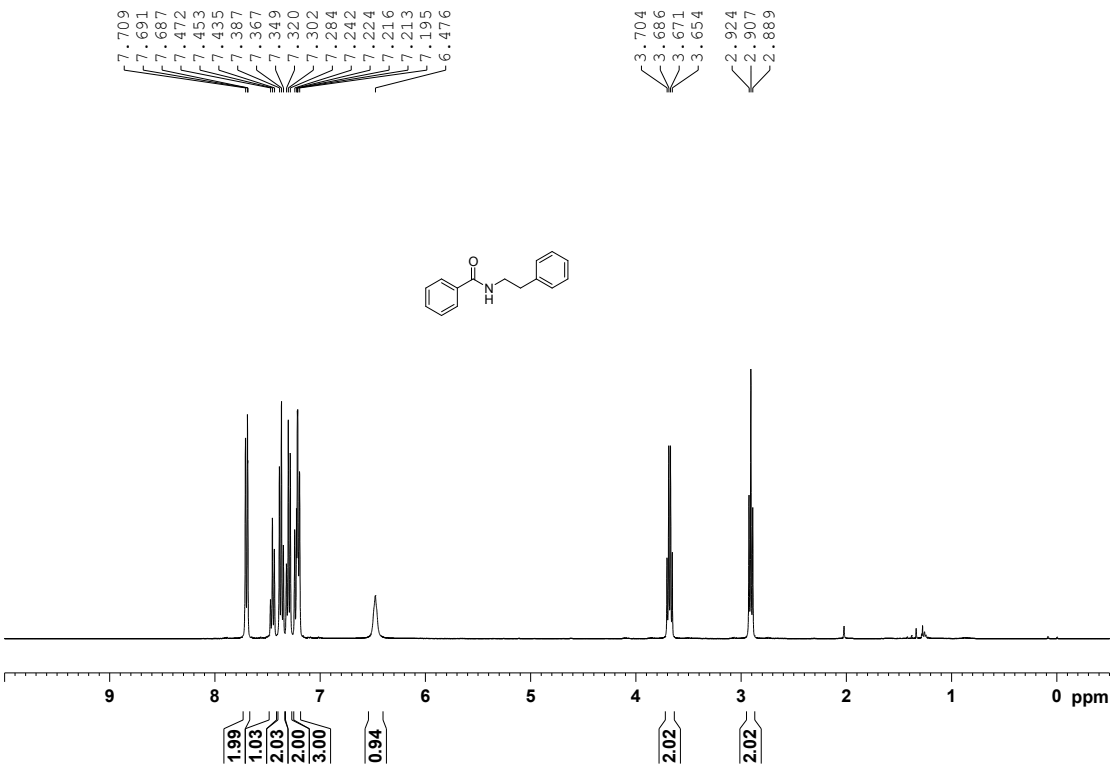
3f ^{1}H NMR (400 MHz, $CDCl_3$)



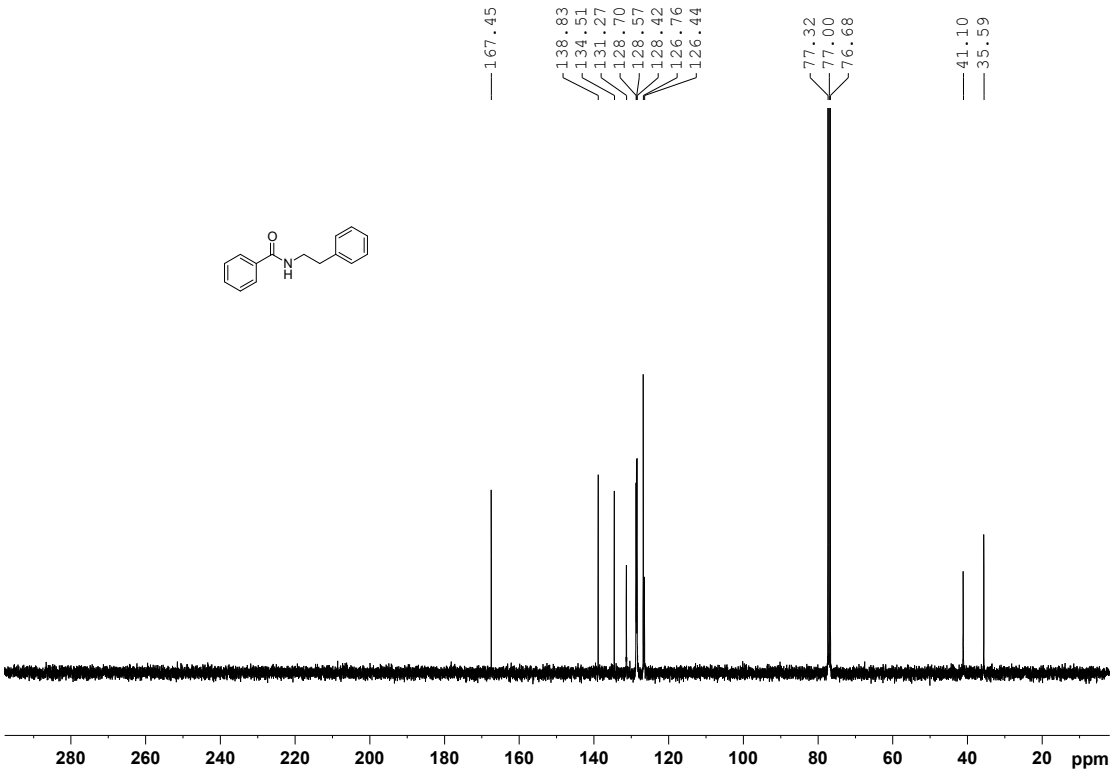
3f ^{13}C NMR (101 MHz, $CDCl_3$)



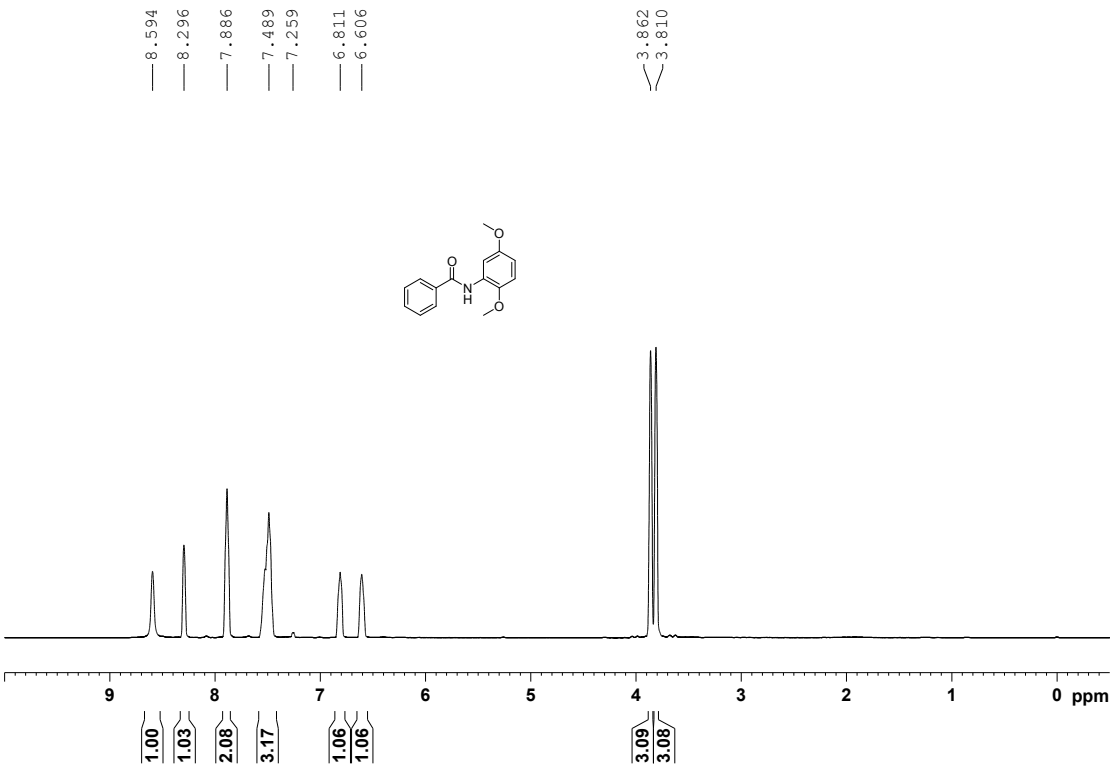
3g ^{1}H NMR (400 MHz, CDCl3)



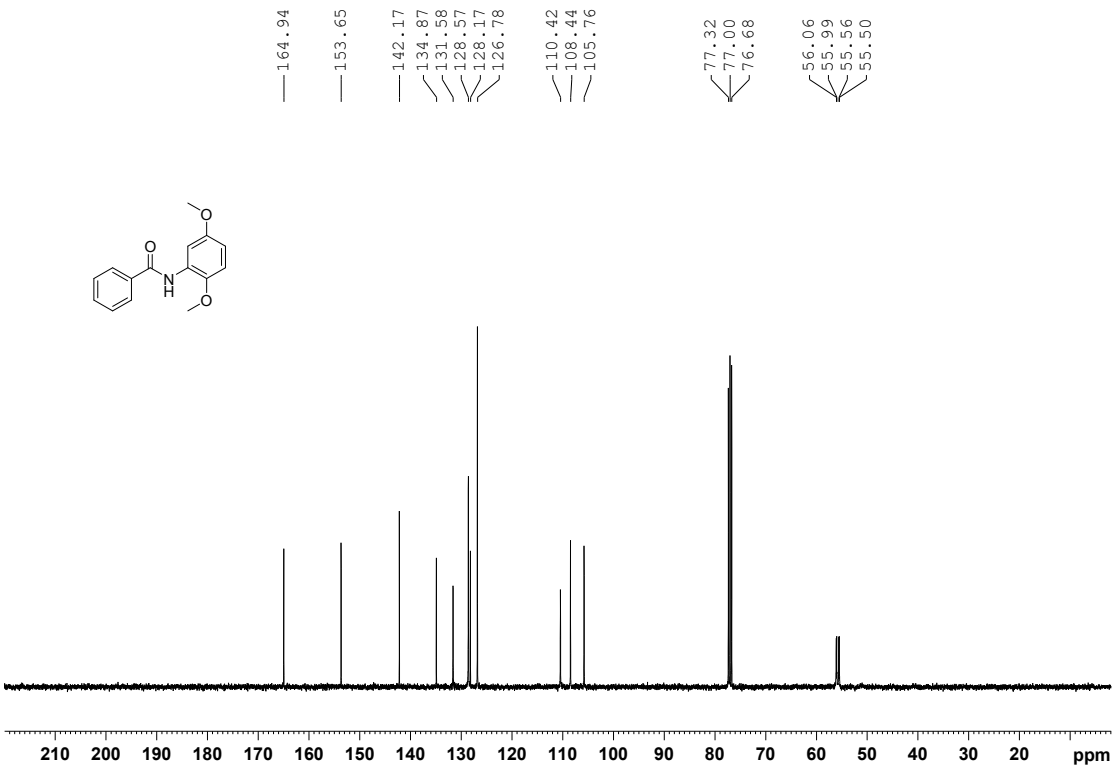
3g ^{13}C NMR (101 MHz, $CDCl_3$)



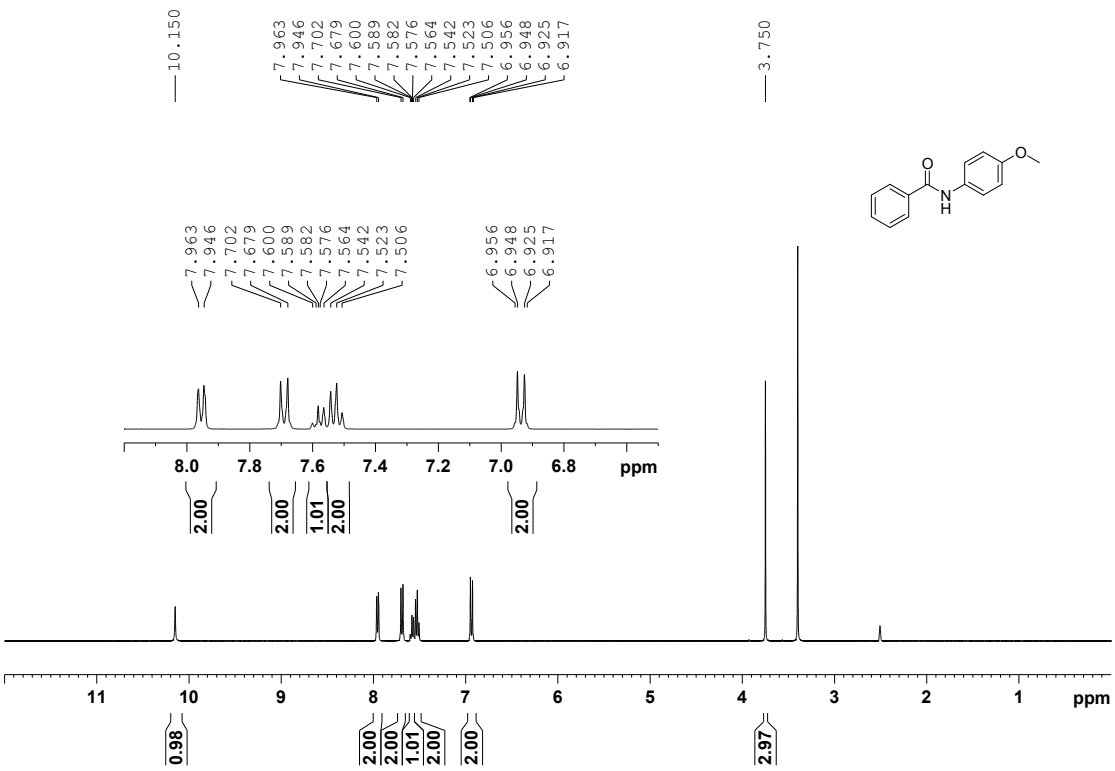
3h ^{1}H NMR (400 MHz, $CDCl_3$)



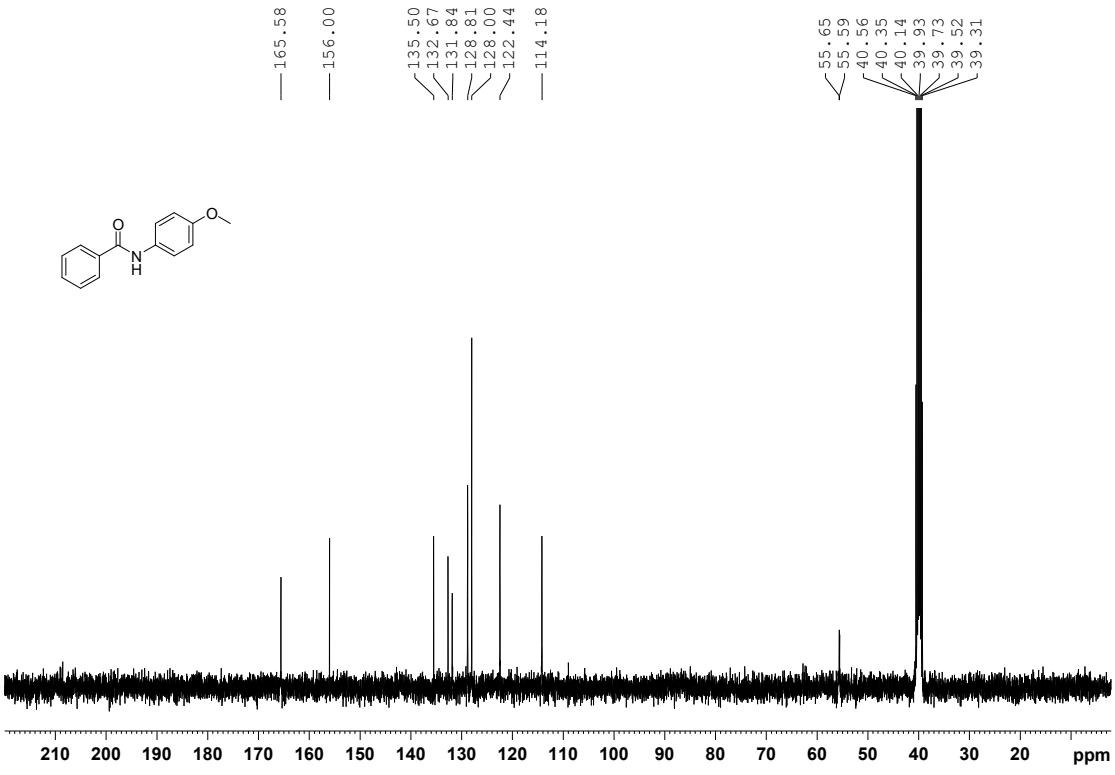
3h ^{13}C NMR (101 MHz, $CDCl_3$)



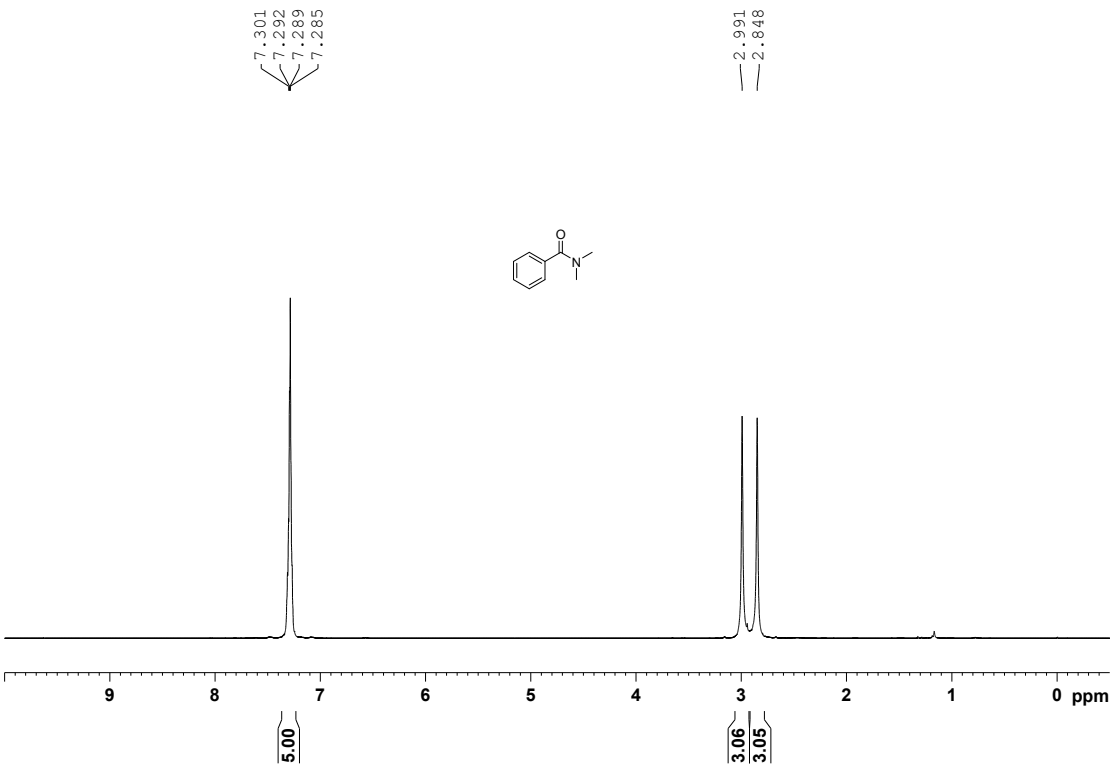
3i ^{1}H NMR (400 MHz, DMSO)



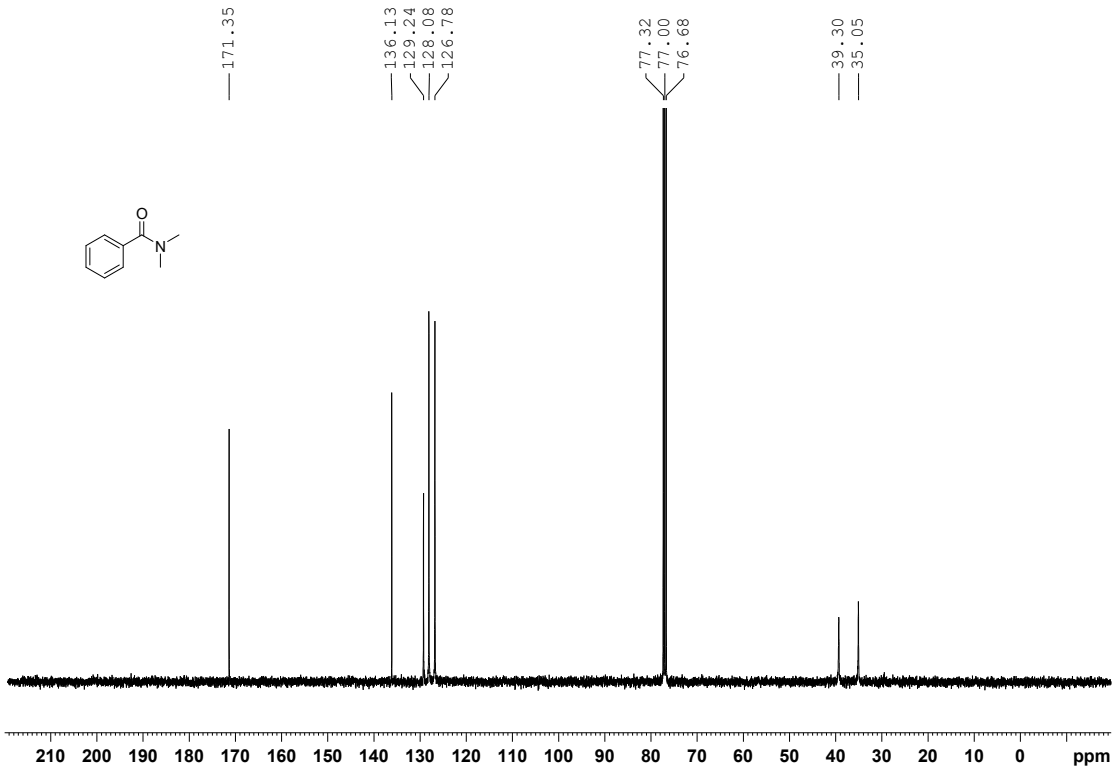
3i ^{13}C NMR (101 MHz, DMSO)



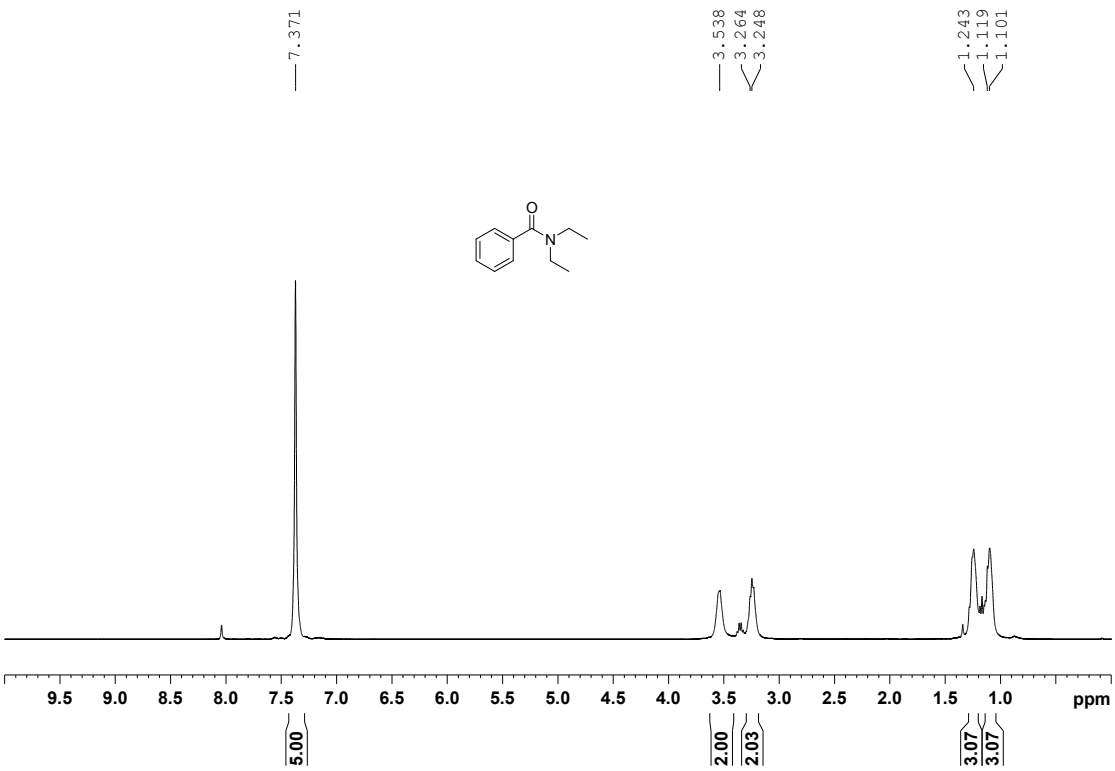
3*J* ^{1}H NMR (400 MHz, $CDCl_3$)



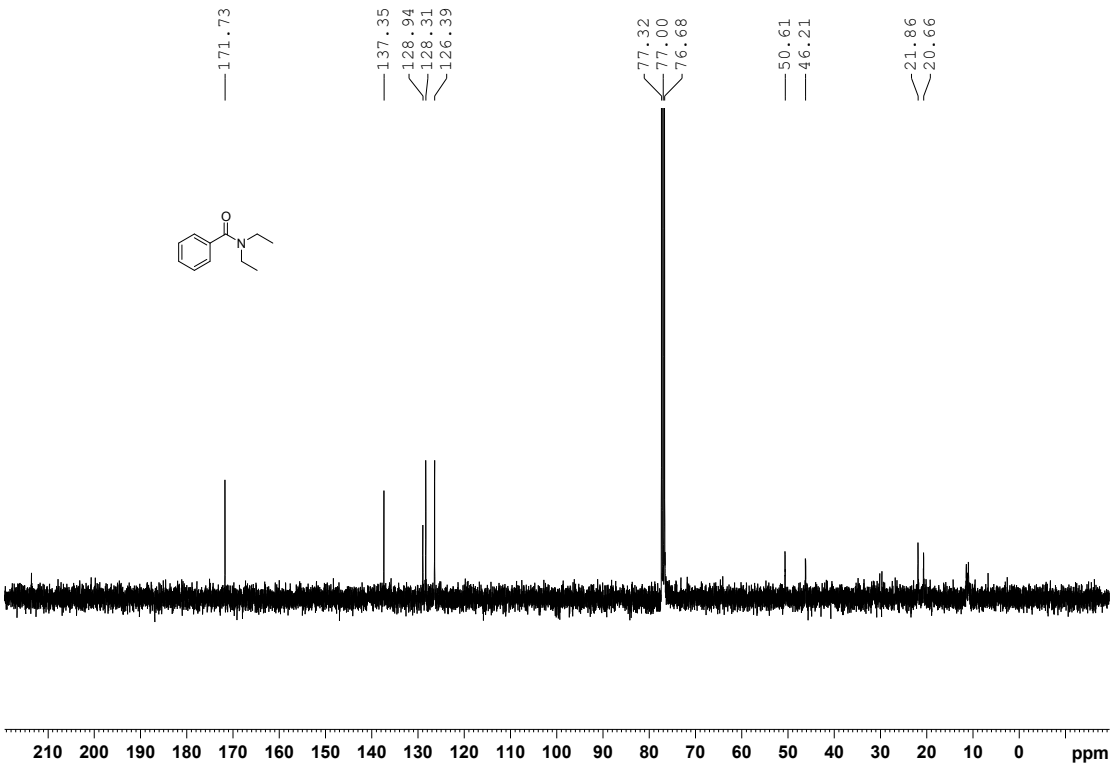
3*J* ^{13}C NMR (101 MHz, $CDCl_3$)



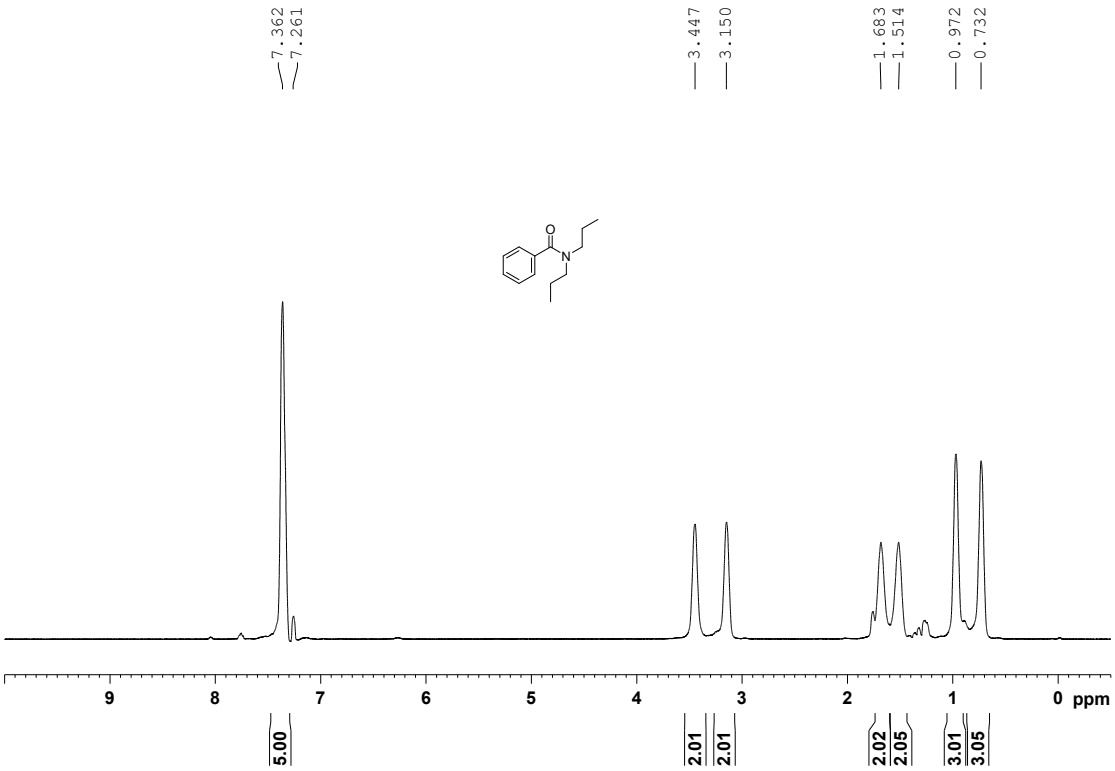
3k ^{1}H NMR (400 MHz, $CDCl_3$)



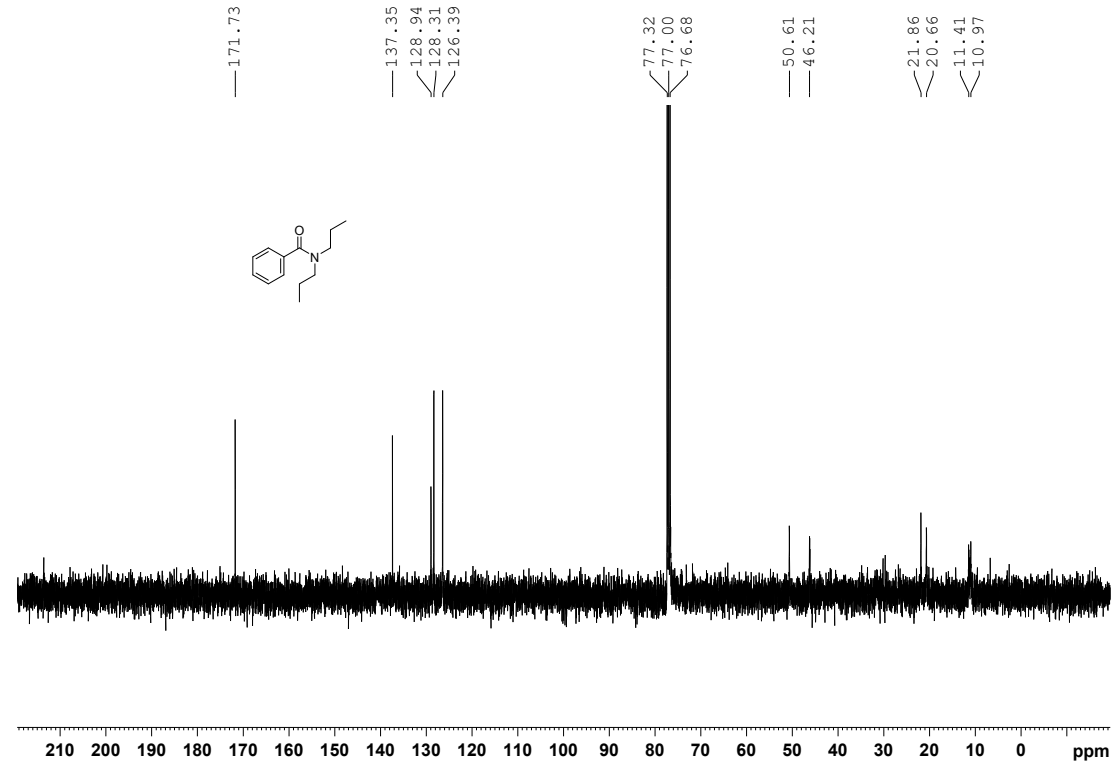
3k ^{13}C NMR (101 MHz, $CDCl_3$)



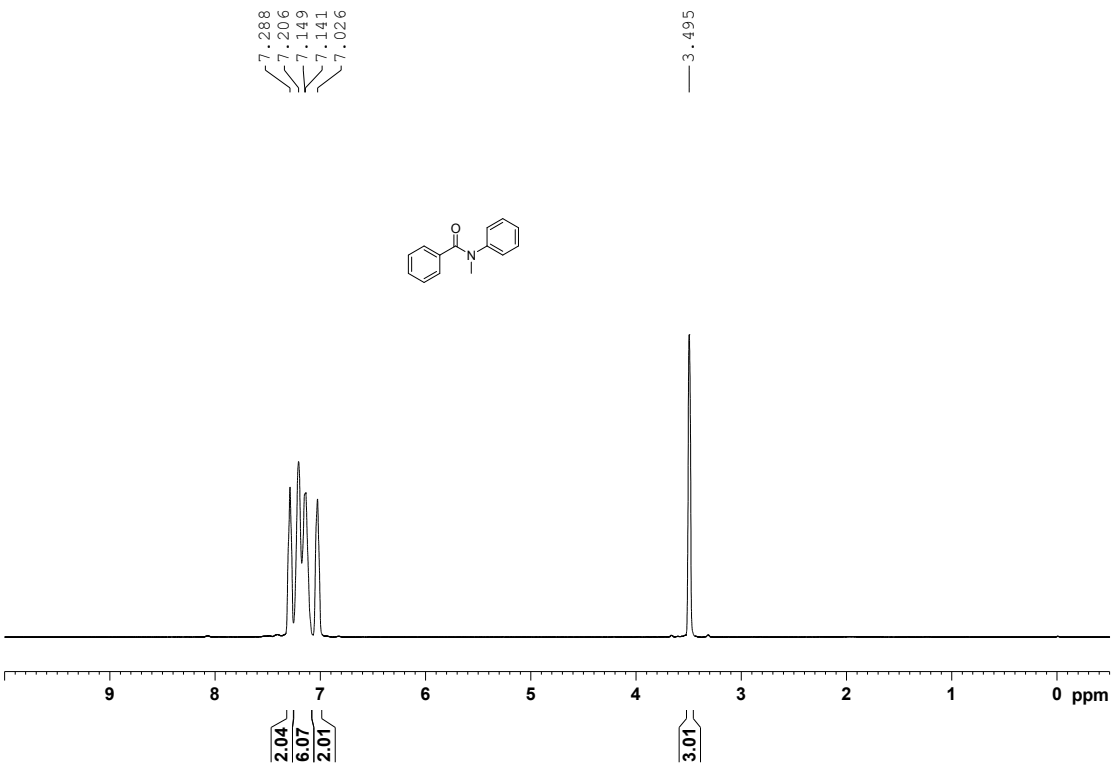
3l ^{1}H NMR (400 MHz, $CDCl_3$)



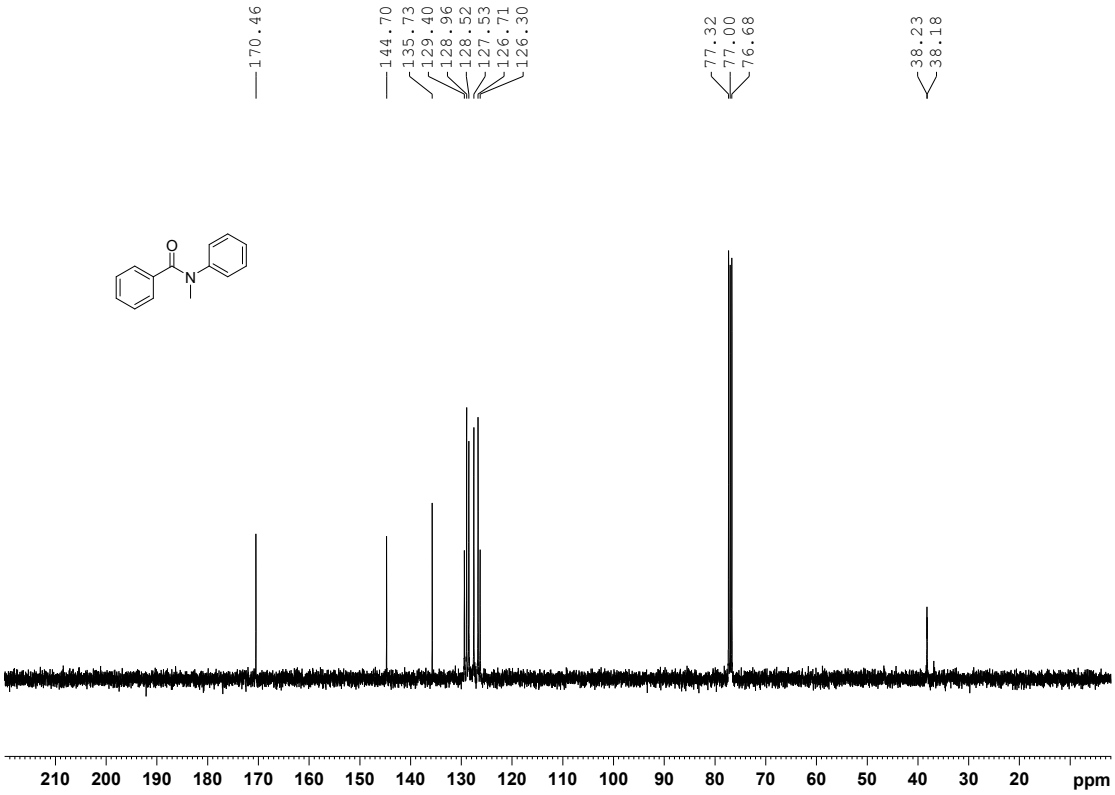
3l ^{13}C NMR (101 MHz, $CDCl_3$)



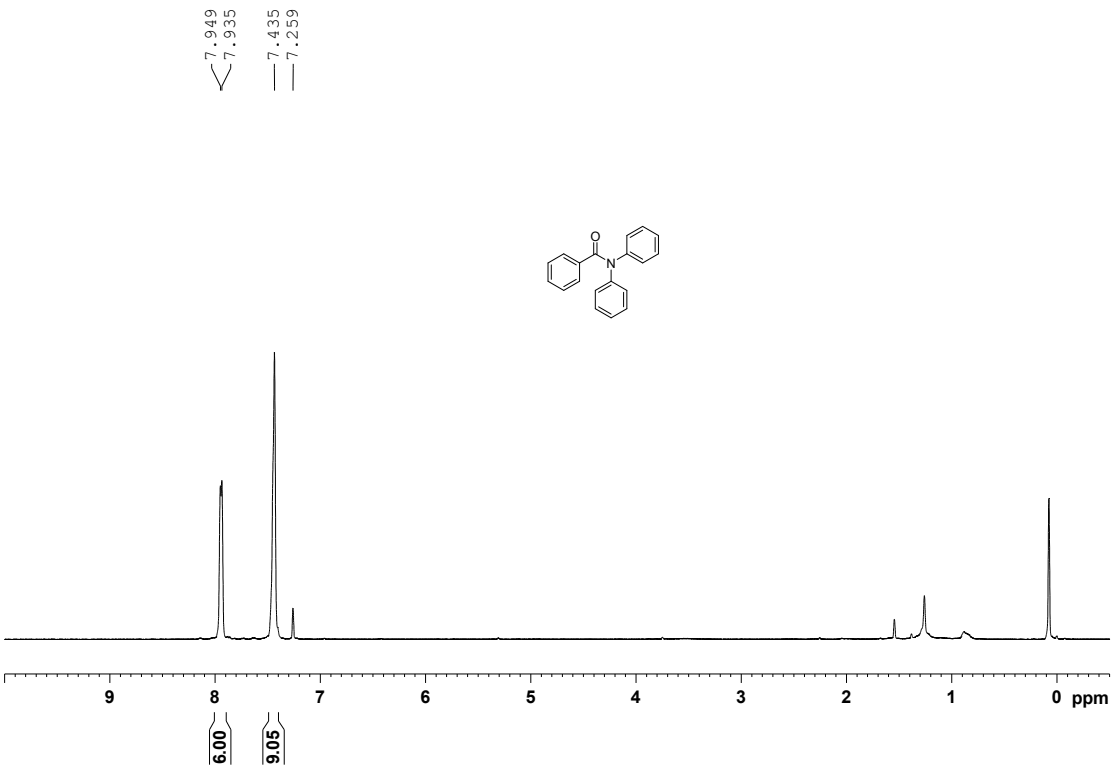
3m ^{1}H NMR (400 MHz, $CDCl_3$)



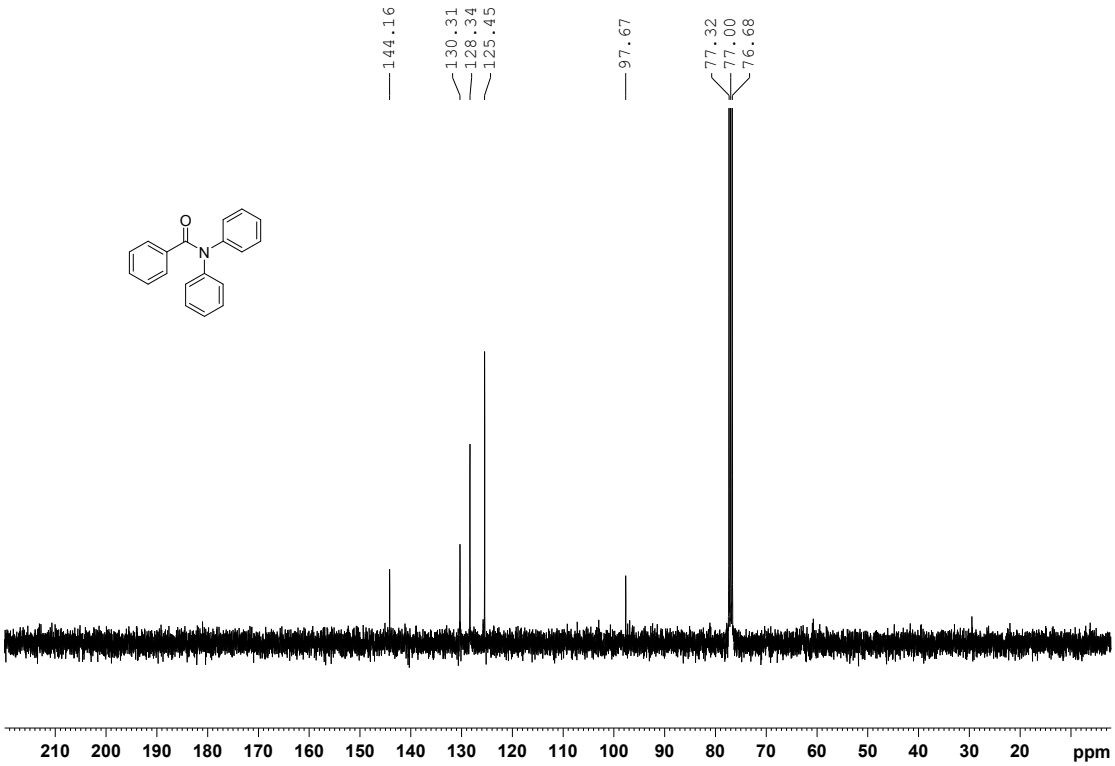
3m ^{13}C NMR (101 MHz, $CDCl_3$)



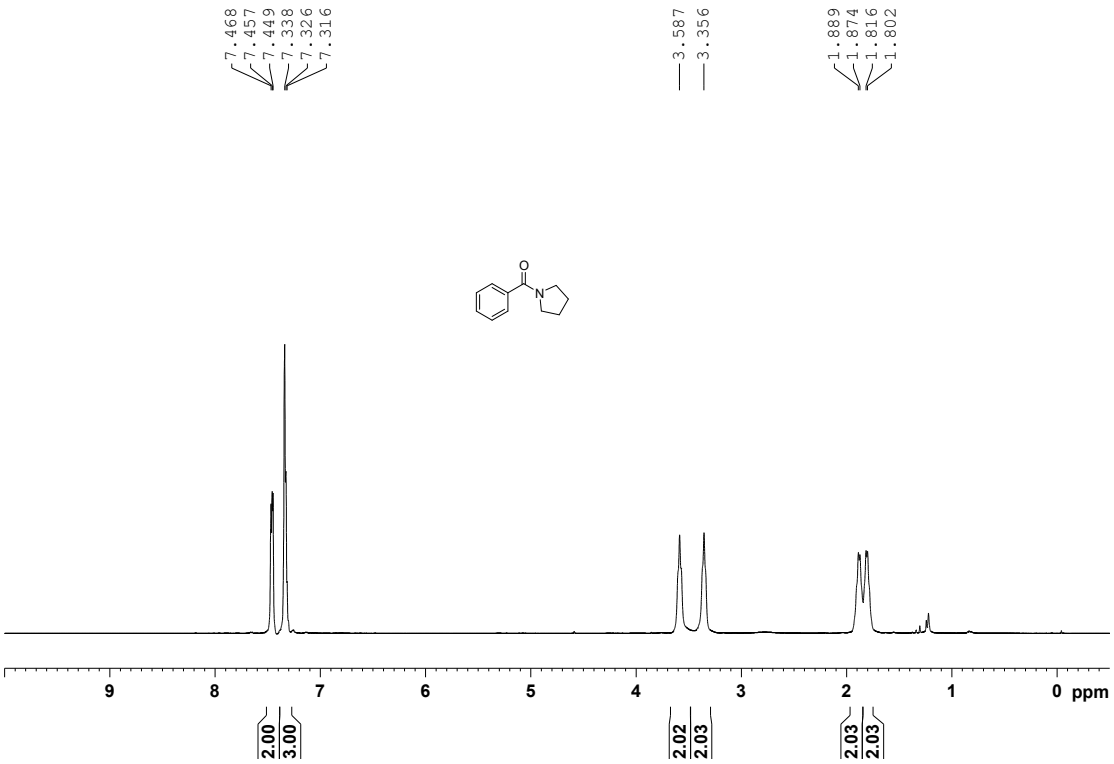
3n ^{1}H NMR (400 MHz, $CDCl_3$)



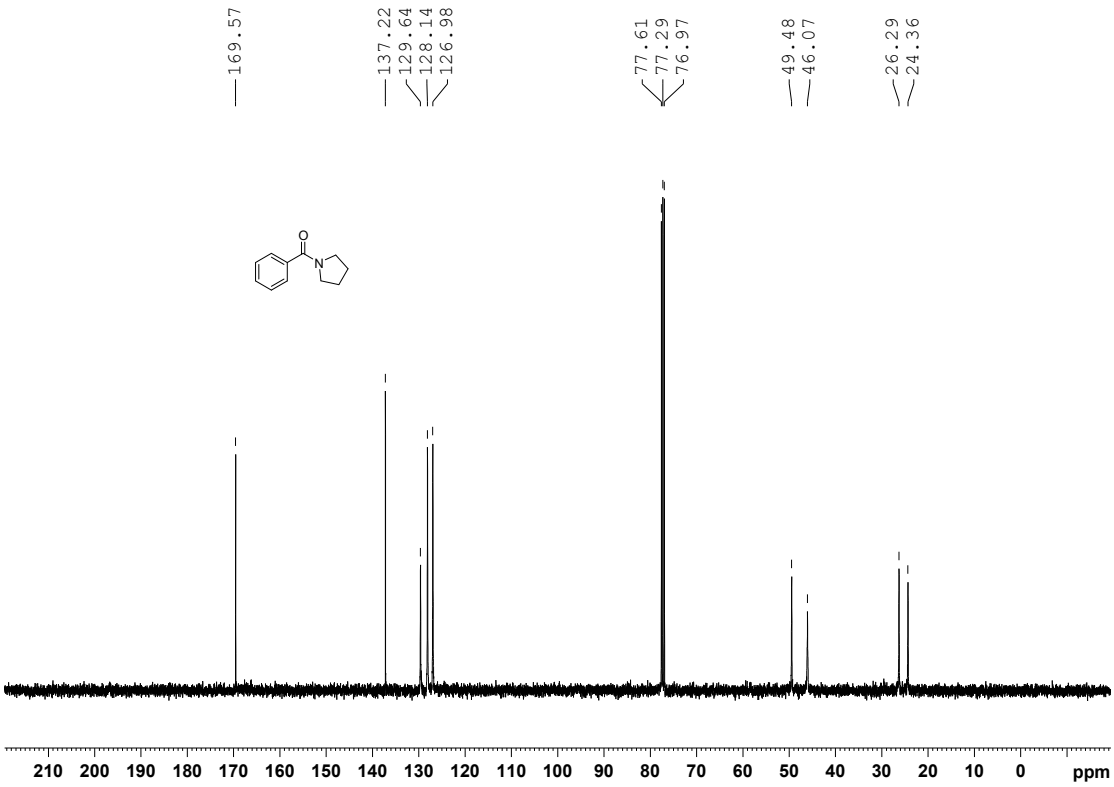
3n ^{13}C NMR (101 MHz, $CDCl_3$)



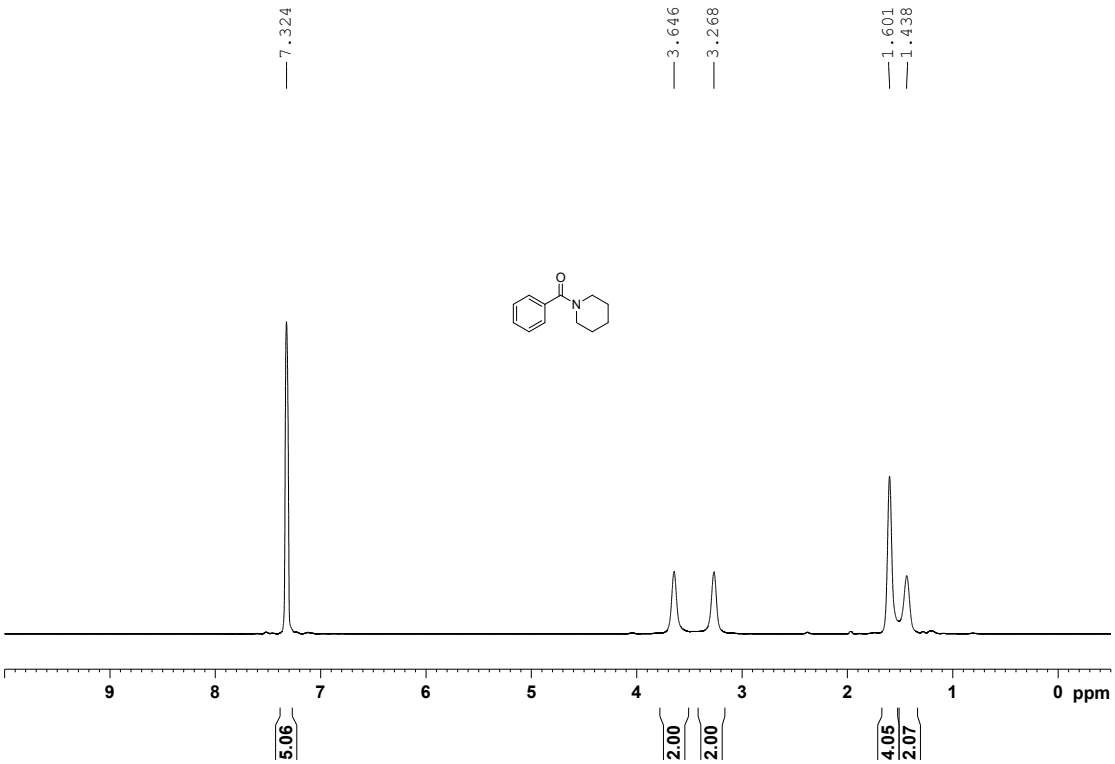
3o ^{1}H NMR (400 MHz, $CDCl_3$)



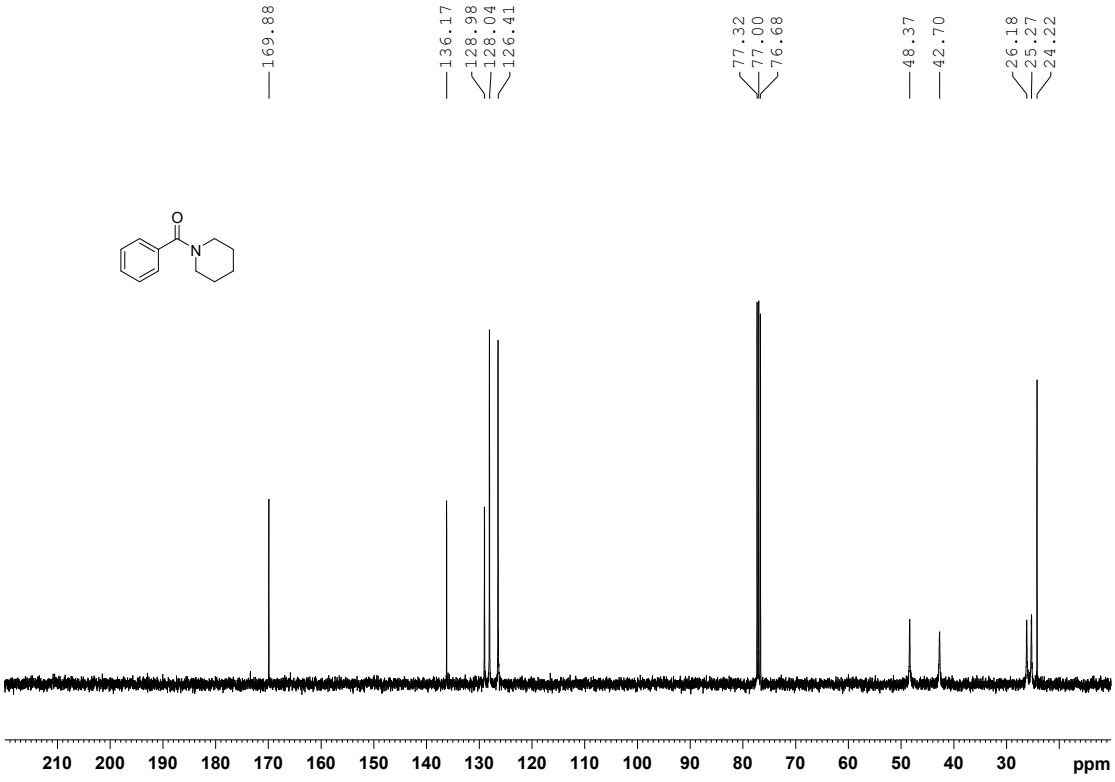
3o ^{13}C NMR (101 MHz, $CDCl_3$)



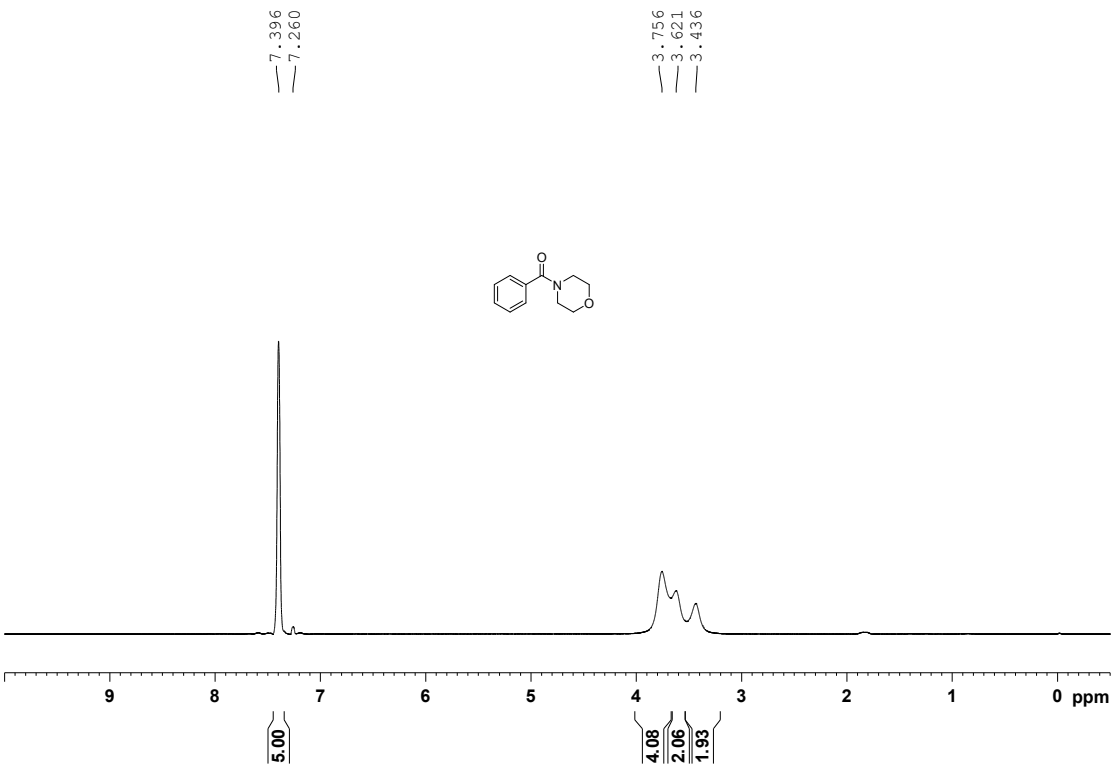
3p ^{1}H NMR (400 MHz, $CDCl_3$)



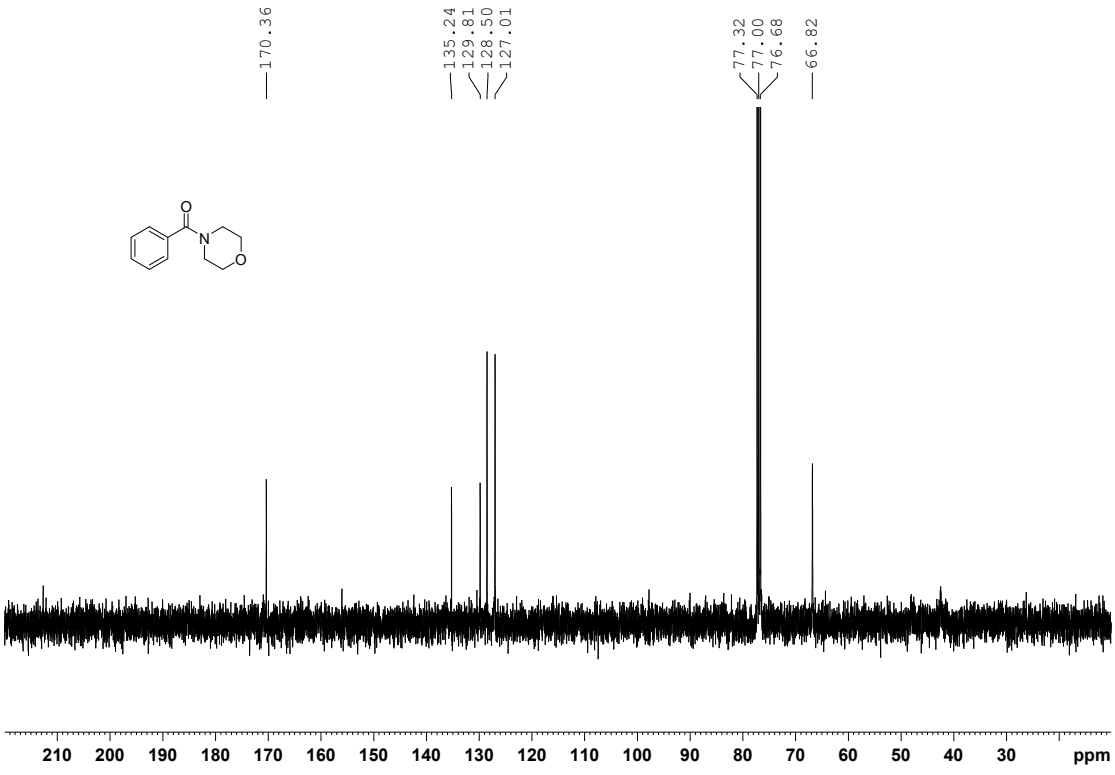
3p 13C NMR (101 MHz, CDCl3)



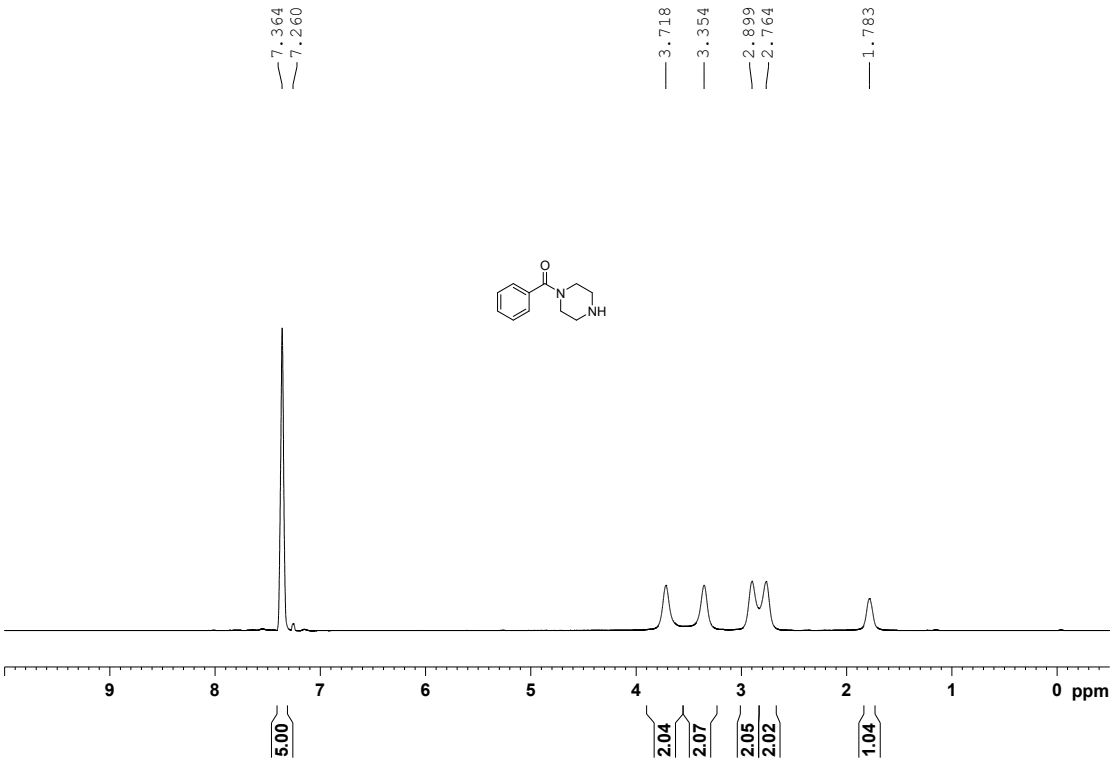
3q ^{1}H NMR (400 MHz, $CDCl_3$)



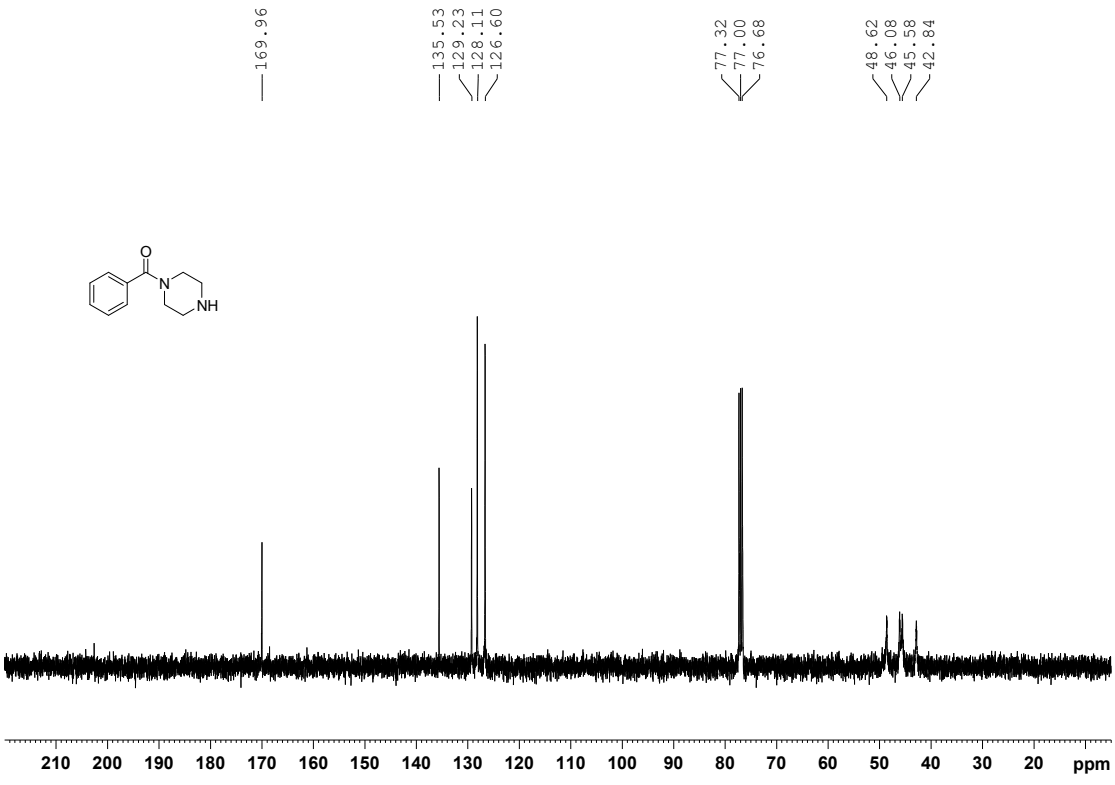
3q ^{13}C NMR (101 MHz, $CDCl_3$)



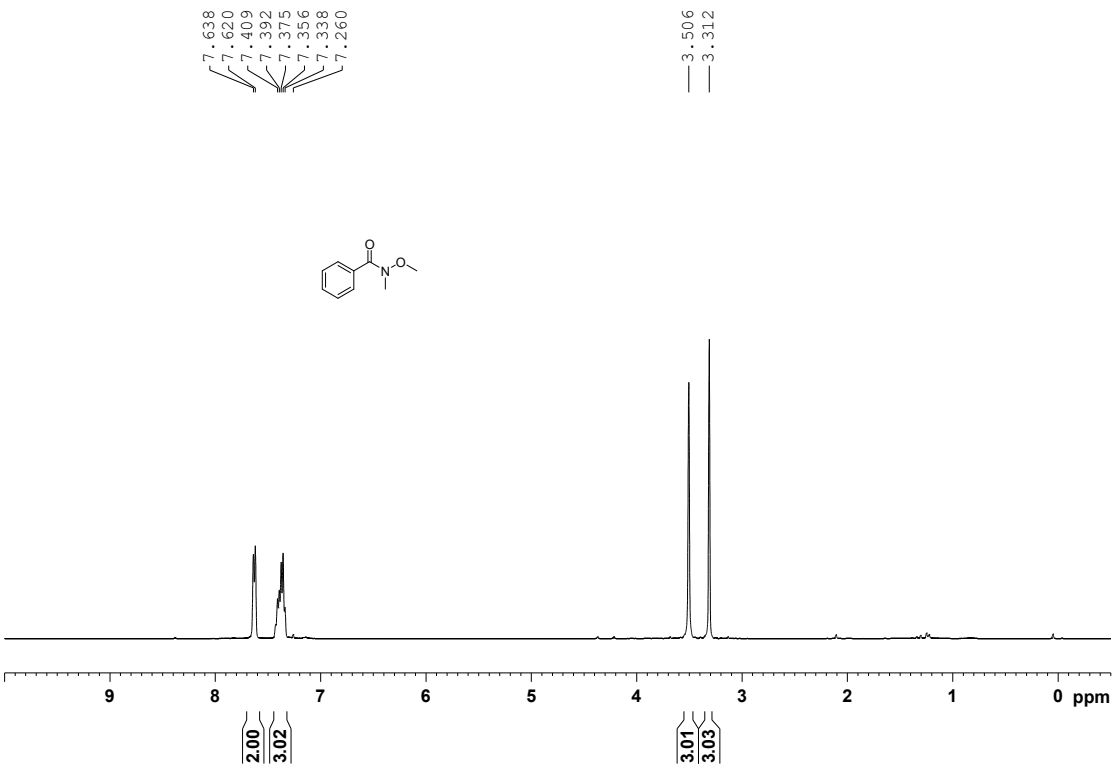
3r ^{1}H NMR (400 MHz, $CDCl_3$)



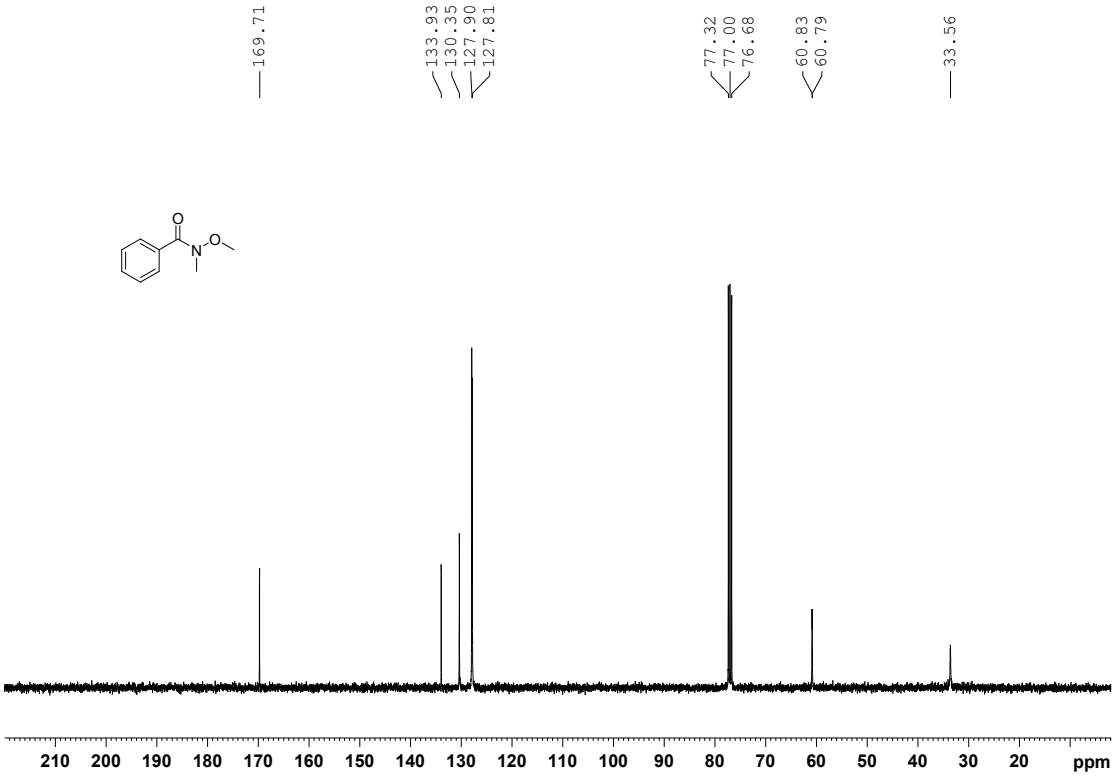
3r ^{13}C NMR (101 MHz, $CDCl_3$)



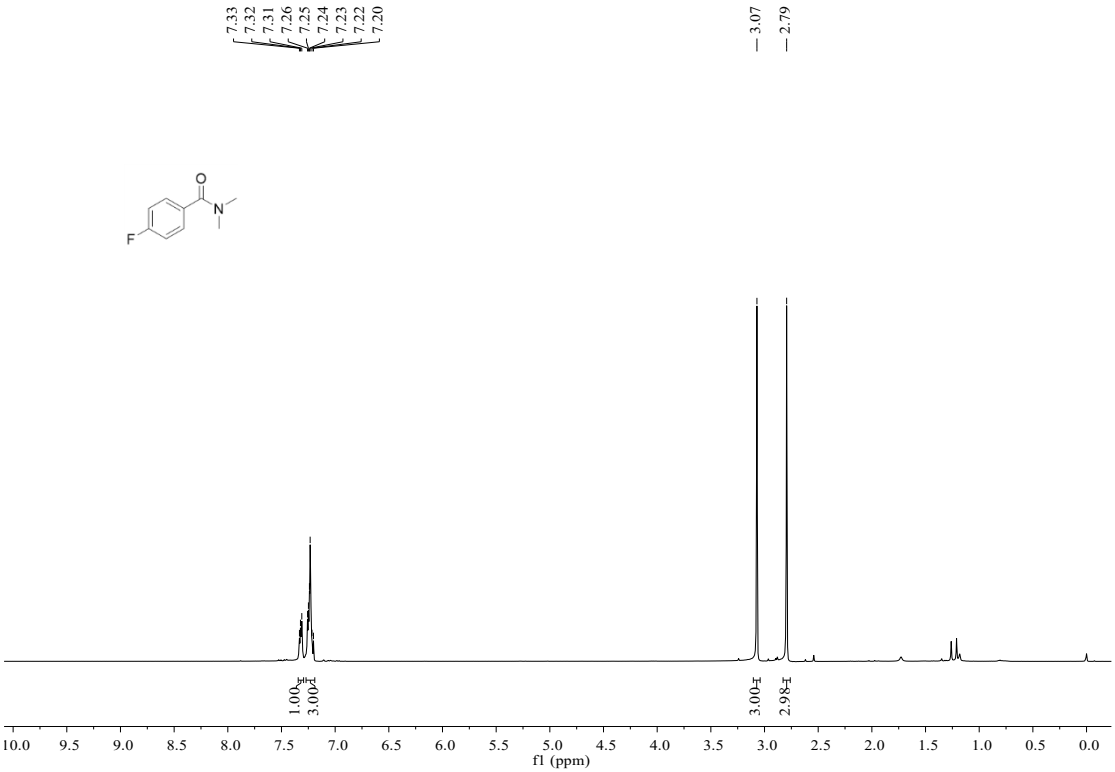
3s ^{1}H NMR (400 MHz, $CDCl_3$)



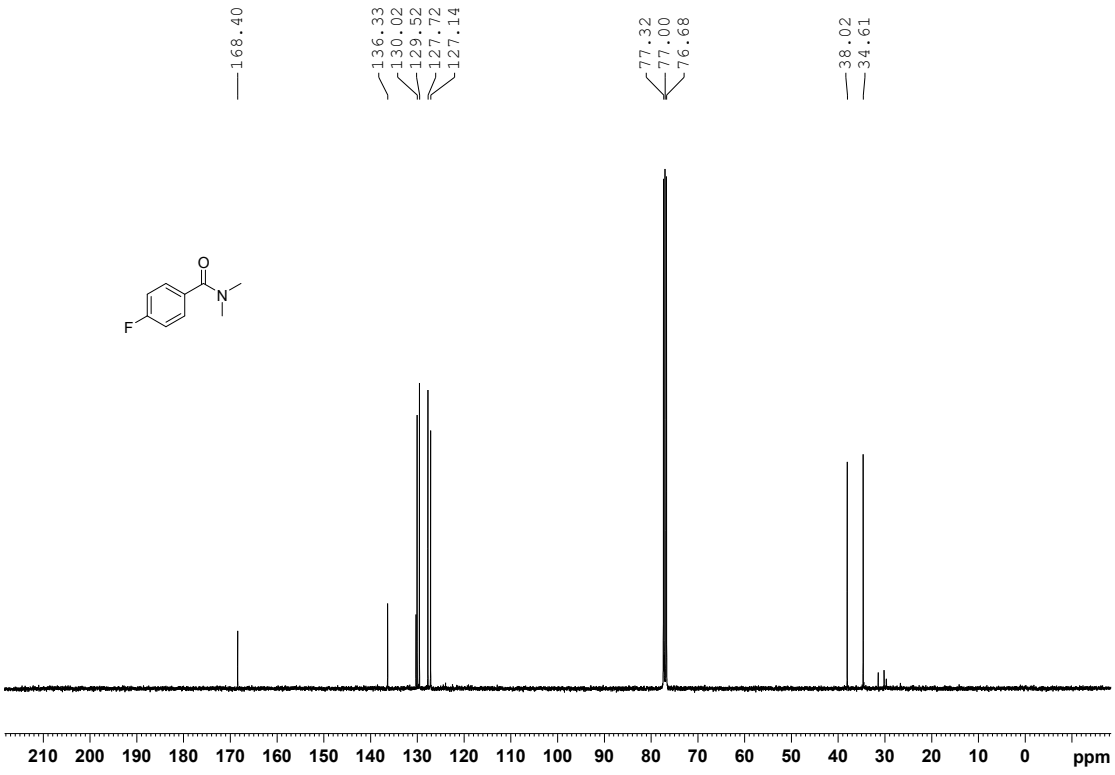
3s ^{13}C NMR (101 MHz, $CDCl_3$)



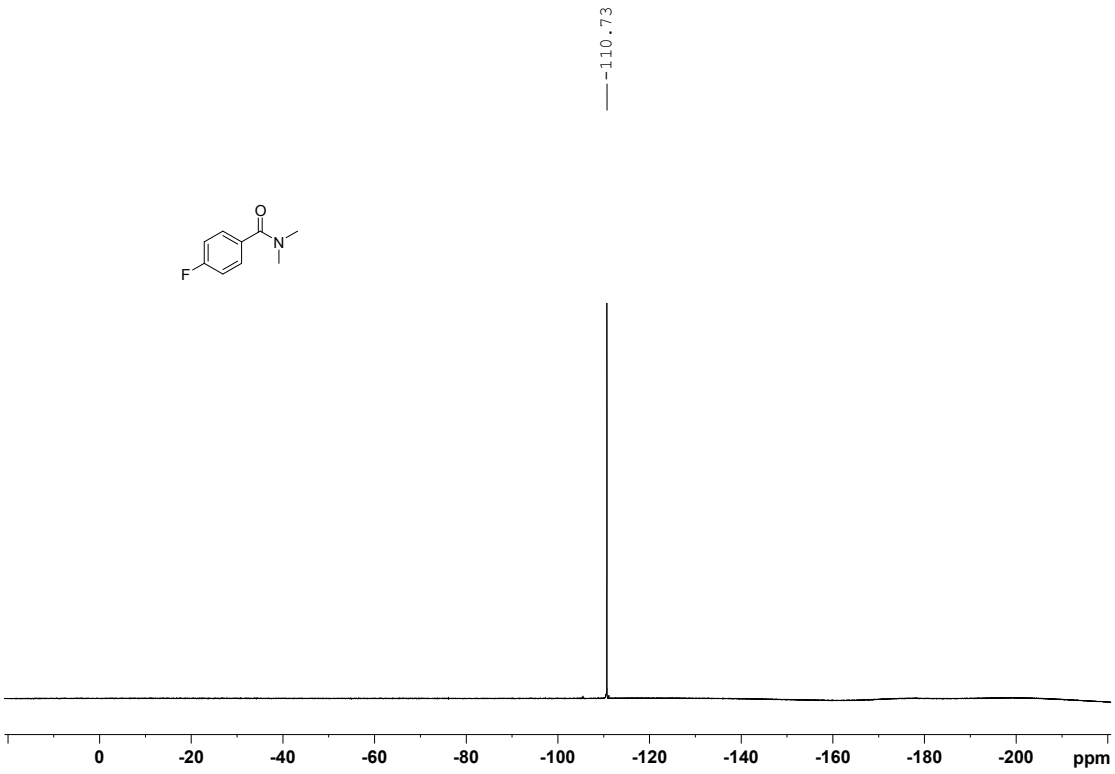
3t ^{1}H NMR (400 MHz, $CDCl_3$)



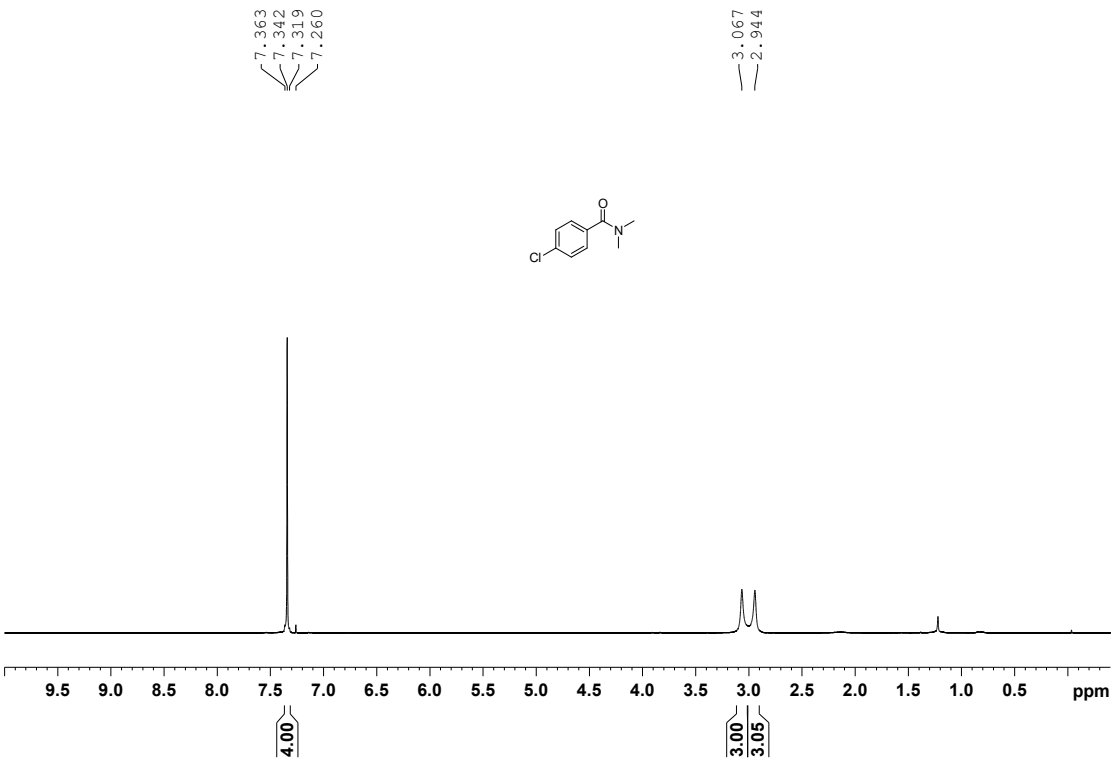
3t ^{13}C NMR (101 MHz, $CDCl_3$)



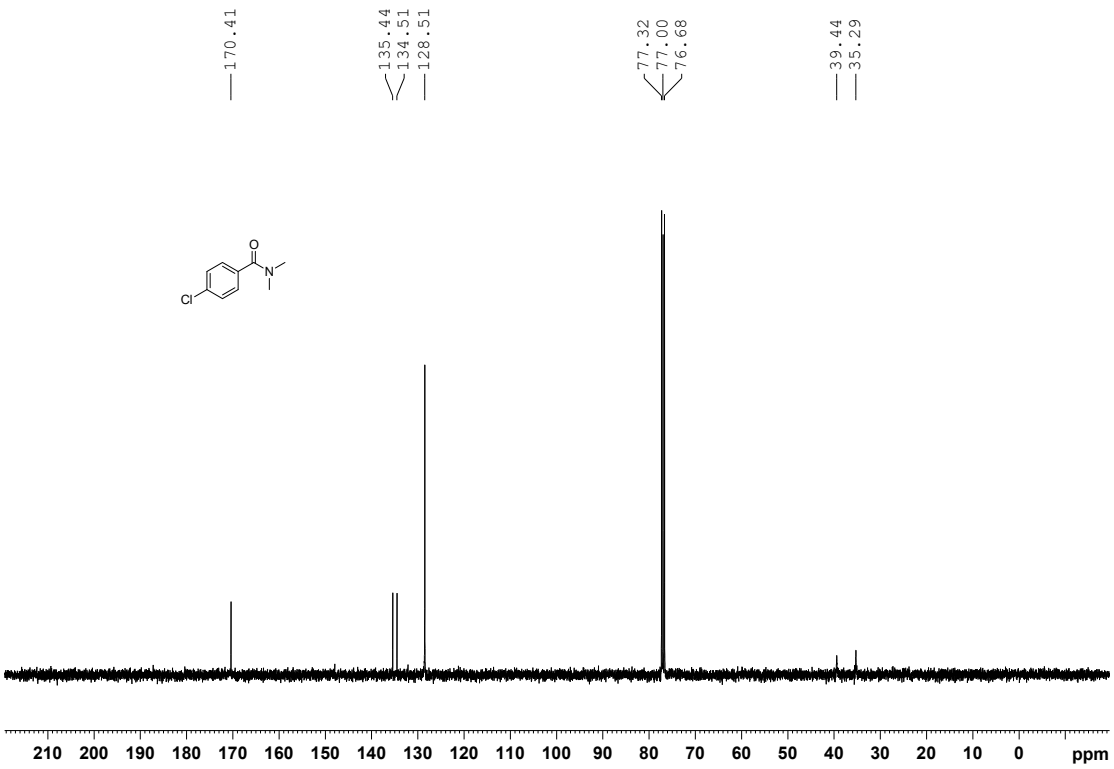
3t ^{19}F NMR (376 MHz, $CDCl_3$)



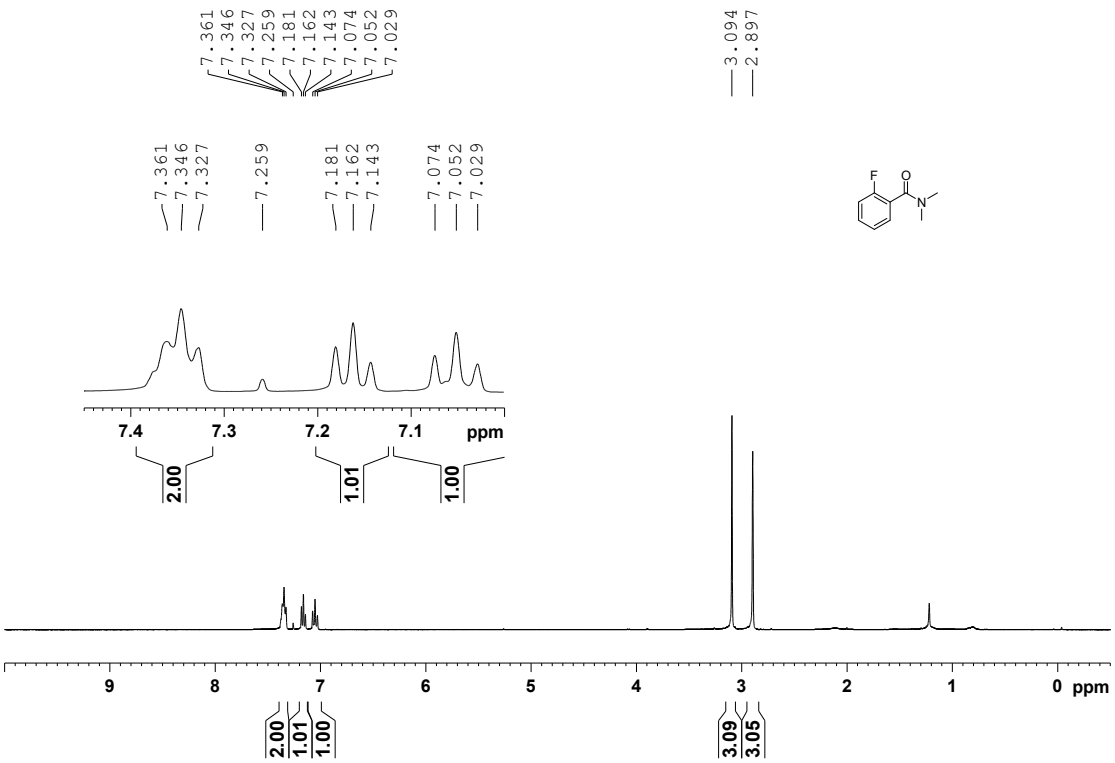
3u ^{1}H NMR (400 MHz, $CDCl_3$)



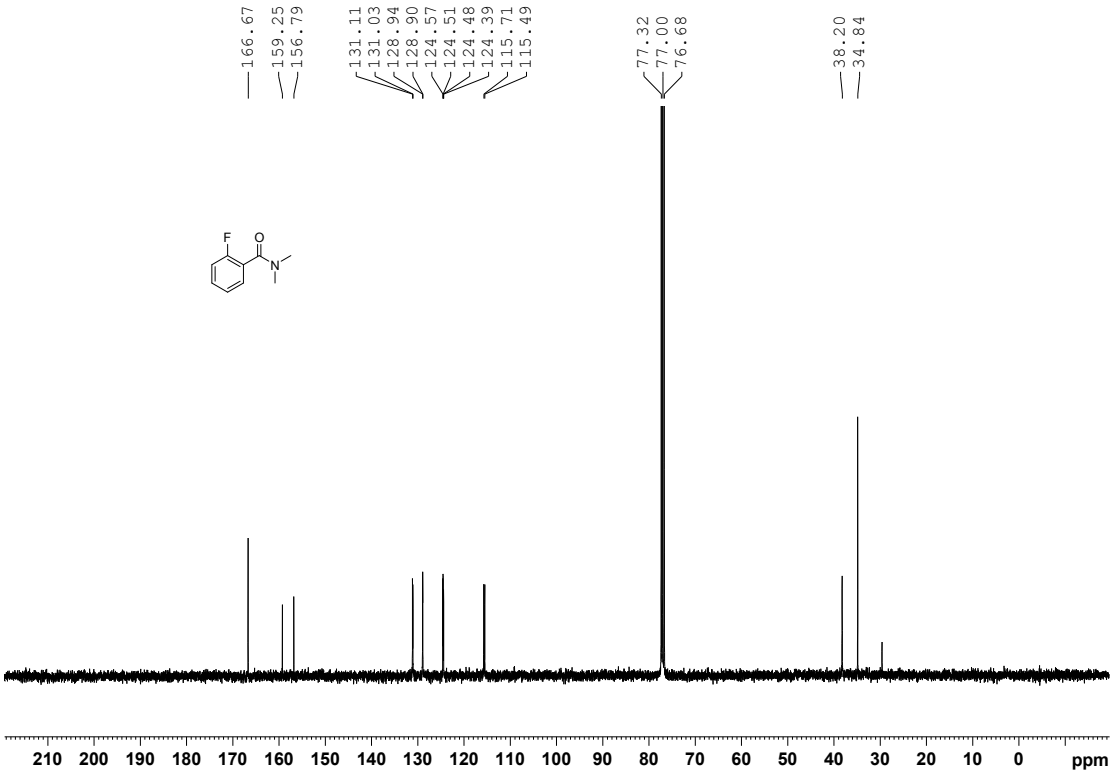
3u ^{13}C NMR (101 MHz, $CDCl_3$)



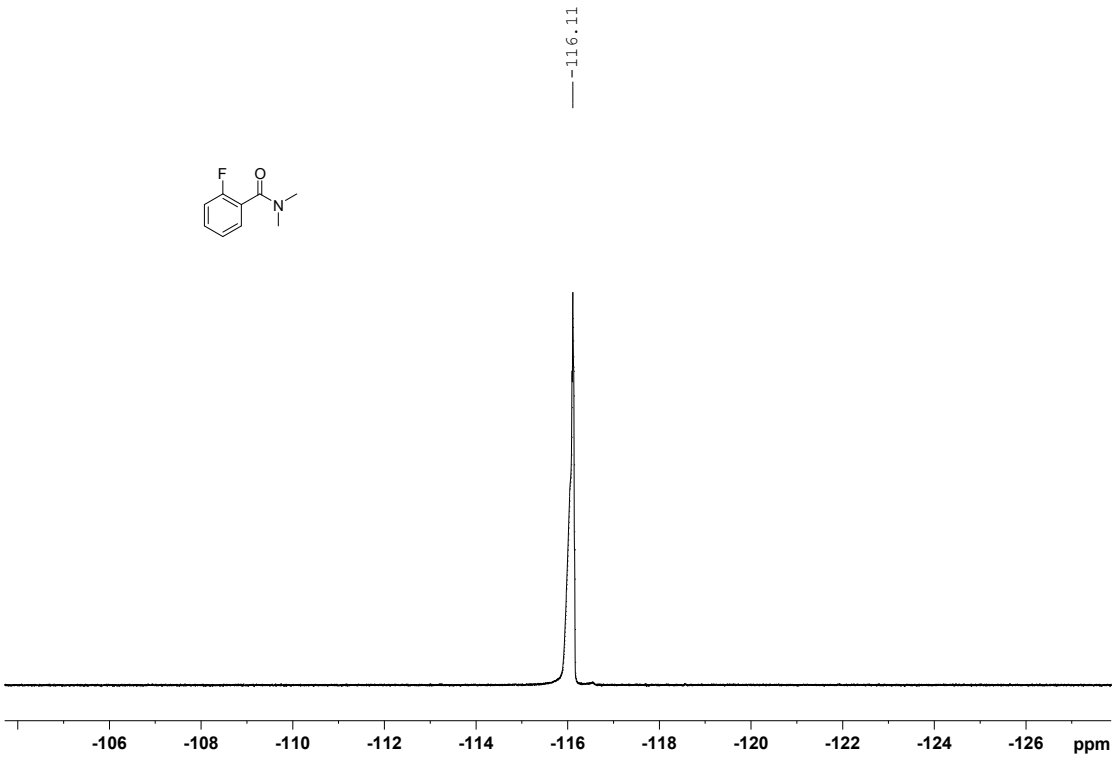
3v ^{1}H NMR (400 MHz, $CDCl_3$)



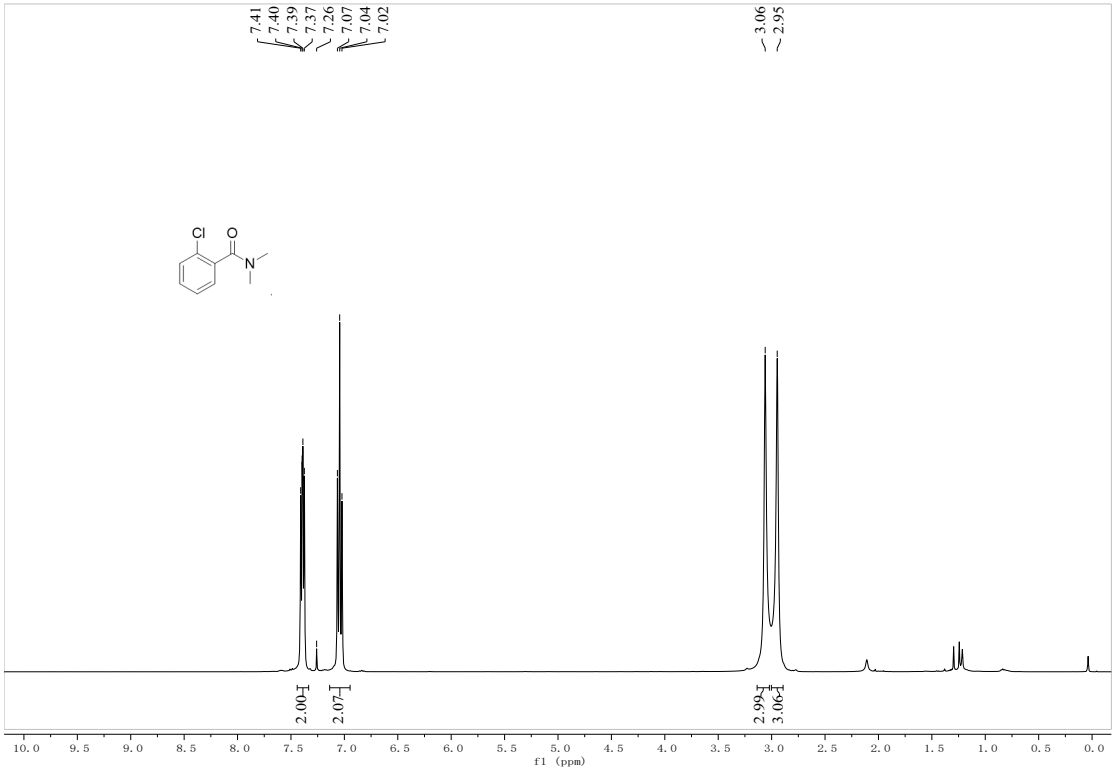
3v ^{13}C NMR (101 MHz, $CDCl_3$)



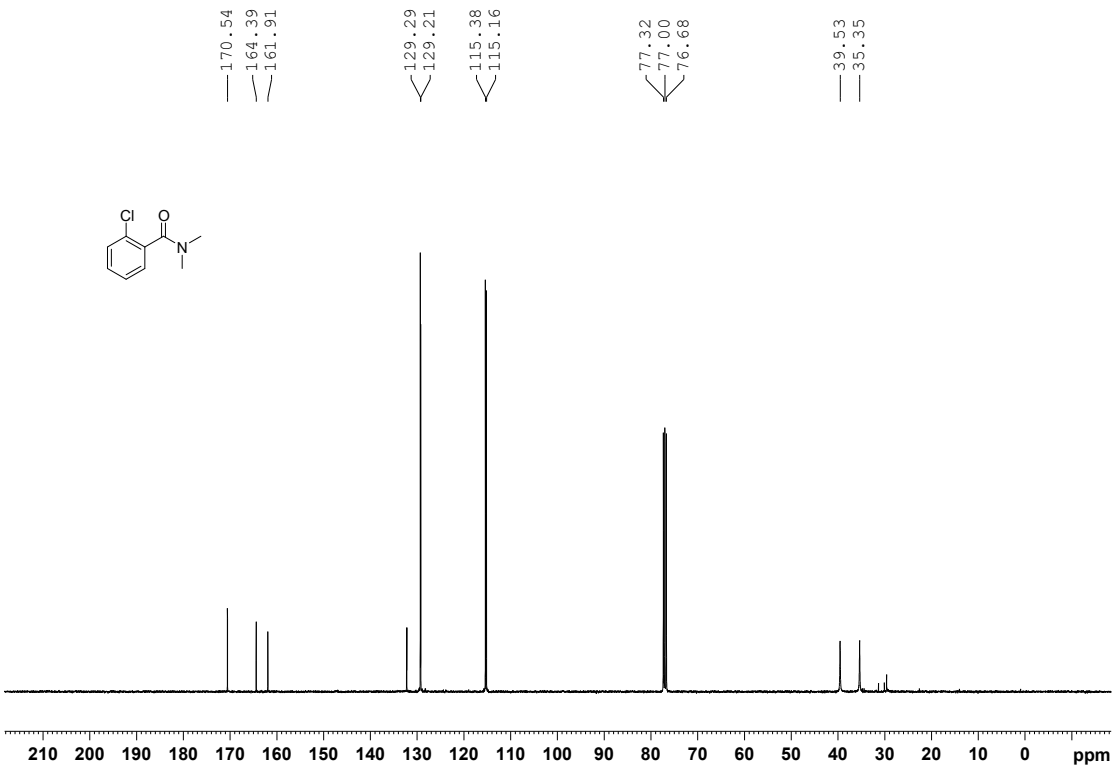
3v ^{19}F NMR (376 MHz, $CDCl_3$)



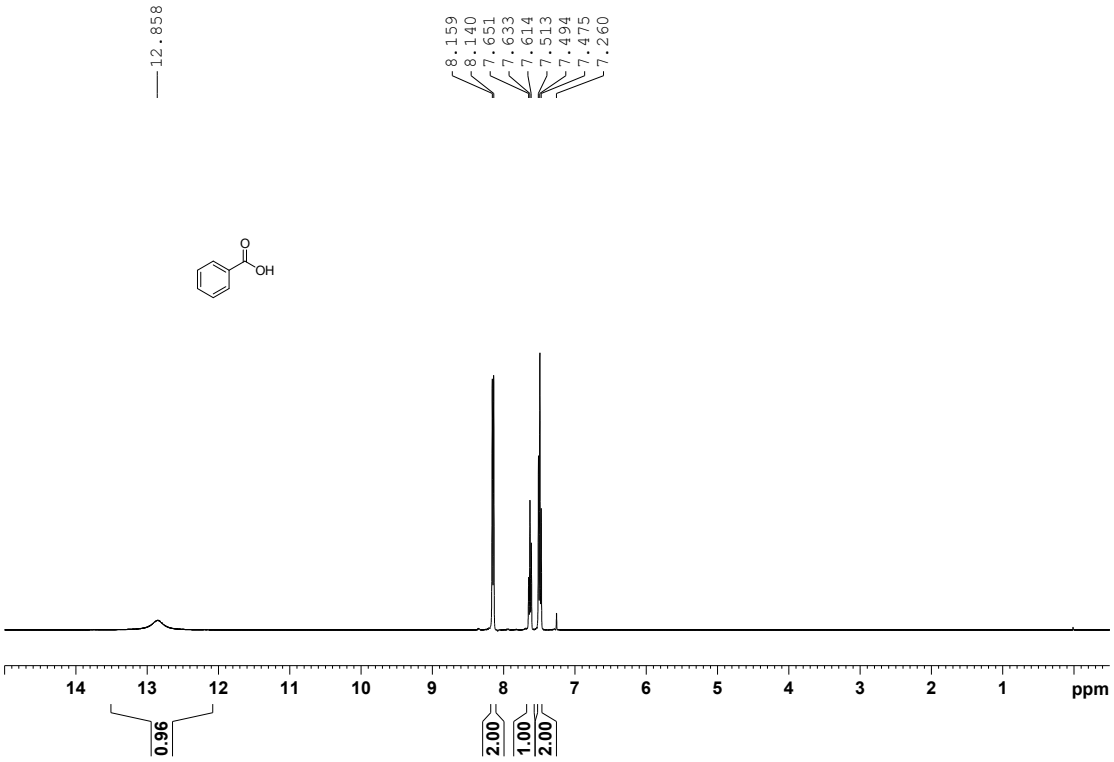
3w ^{1}H NMR (400 MHz, $CDCl_3$)



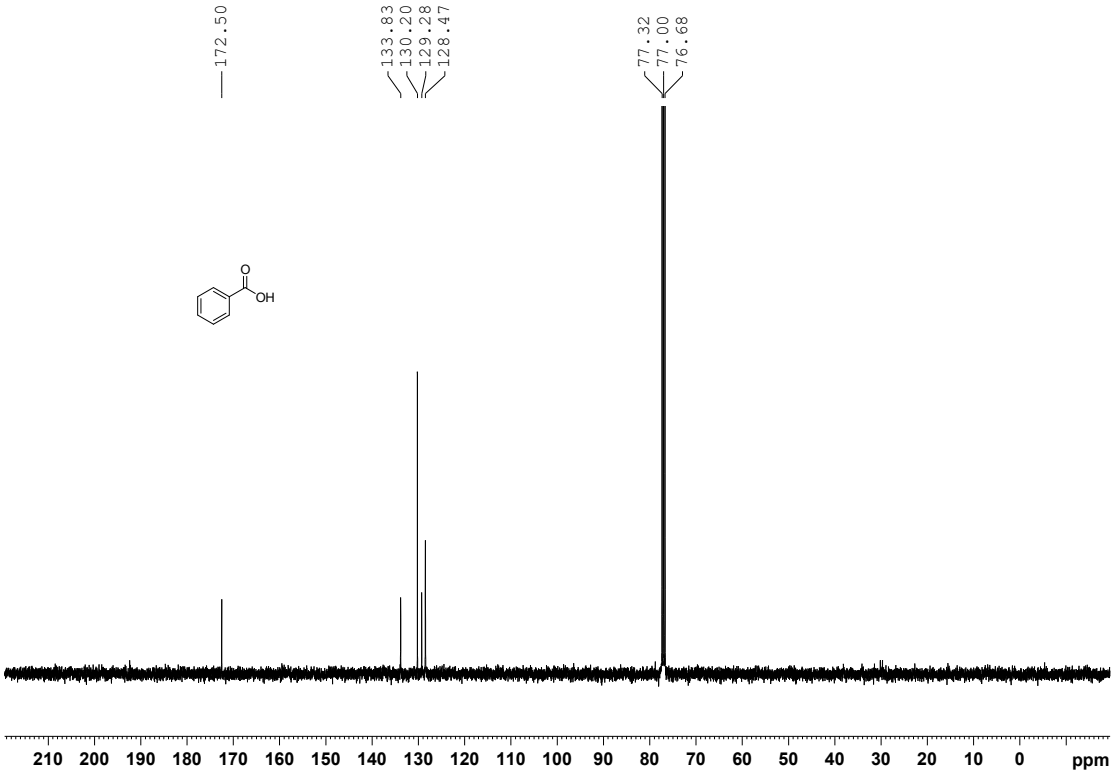
3w 13C NMR (101 MHz, CDCl3)



benzoic acid ^{1}H NMR (400 MHz, $CDCl_3$)



benzoic acid 13C NMR (101 MHz, CDCl3)



Isotope reaction mass spectrometry data