Electronic Supplementary Material (ESI) for ChemComm.

Supplementary Information

Direct, efficient NHC-catalysed aldehyde oxidative amidation: in situ formed benzils as unconventional acylating agents

Vikas Kumar[a] and Stephen J. Connon*,[a]

[a] School of Chemistry Trinity Biomedical Sciences Institute, Trinity College Dublin 152-160 Pearse Street, Dublin 2, Ireland

*Corresponding authors: connons@tcd.ie; Ph: +35318961306

Table of Contents

1 General Methods

Unless otherwise noted, all commercially available compounds were used as provided without further purification. Melting points were determined using a standard melting point apparatus and are uncorrected. Proton NMR spectra were recorded on an Agilent 400 MHz spectrometer using the solvent peak as internal reference ($CDCl_3$: δ H 7.26; δ C 77.0). Multiplicities are indicated, s (singlet), d (doublet), t (triplet), q (quartet), quint (quintet), sept (septet), m (multiplet); coupling constants (J) are in Hertz (Hz). Carbon NMR were recorded on the previously mentioned instrument (100.61 Hz) with total proton decoupling. All melting points are uncorrected. Mass spectra (MS ESI) were recorded using a Waters Micromass LCT-time of flight mass spectrometer (TOF), interfaced to a Waters 2690 (HPLC). The Electron Impact mass spectra were recorded on the same machine in EI mode. TLC analysis was performed on precoated silica gel 60F 254 slides, and visualized by $KMnO_4$ staining. Flash chromatography was carried out using silica gel, particle size 0.2-0.063mm and using the indicated mobile phase as correlated with TLC analysis. Anhydrous tetrahydrofuran was obtained by using Pure Solv MD- 4EN Solvent Purification System. All reactions were carried out using oven-dried 25 mL RB flask. Liquid aldehydes were distilled under vacuum prior to use. Solid aldehydes were washed acid-free with 10% aq. K_2CO_3-solution prior to use. Amines were distilled over CaH_2 prior to use. DBU was distilled over CaH_2 and stored over activated molecular sieves under argon.

2 General Procedures

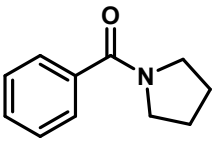
General Procedure (Amidation of Aldehydes by Triazolium Precatalyst using Amine, Table 1)

A 25 mL oven dried 2 neck RB flask, with a magnetic stirring bar, was cooled down under high vacuum and after cooling it was purged with argon using Schlenk line. This flask was then charged with triazolium precatalyst **17** (36 mg, 0.15 mmol), phenazine (**18**) (184 mg, 1.0 mmol) and 1,2,4-triazole **48** (15 mg, 0.20 mmol). This mixture was evacuated under high vacuum and was then purged with argon gas with the help of Schlenk line. This sequence was repeated for 3 times. Dry THF (2.5 mL) was then added to this mixture followed by addition of DBU (170 mL, 1.1 mmol, 110 mol%). After 5 min, aldehyde was added followed by immediate addition of amine. The reaction mixture was stirred for the indicated time at room temperature unless stated otherwise. The solvent was then removed in vacuo and the resulting residue subjected to flash chromatography to yield the amide product.

3 Experimental Data for Amide Products

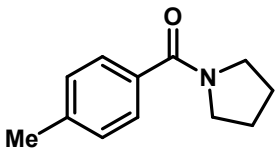
Phenyl(pyrrolidin-1-yl)methanone (29)

Prepared according to general procedure. Purified via flash chromatography (EtOAc:n-hexane, 4:1); 155 mg (88%) as a pale semisolid. ^1H NMR ($CDCl_3$, 400 MHz) δ = 1.82-1.89 (m, 2H), 1.91-1.98 (m, 2H), 3.41 (t, *J* 6.7, 2H), 3.63 (t, *J* 6.7, 2H), 7.36-7.39 (m, 3H), 7.49-7.51 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ = 24.6, 26.5, 46.3, 49.7, 127.2, 128.3, 129.8, 137.4, 169.8; HRMS (ES): calcd. for $[M^+ + H]$ $C_{11}H_{14}NO$ requires 176.1075, found 176.1072.

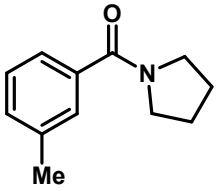


Pyrrolidin-1-yl(p-tolyl)methanone (56)

Prepared according to general procedure. Purified via flash chromatography (n-hexane:EtOAc, 4:1); 177 mg (93%) as a pale yellow solid. M.p. 76-78°C. ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 1.89 (br s, 4H), 2.37 (s, 3H), 3.44 (br s, 2H), 3.63 (br s, 2H), 7.19 (d, *J* 7.8, 2H), 7.42 (d, *J* 7.8, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ = 21.50, 21.51, 24.6, 26.5, 46.3, 49.8, 127.33, 127.34, 128.92, 128.93, 134.5, 139.96, 139.99, 169.9; HRMS (ESI): calcd. for $[M^+ + H]$ $C_{12}H_{16}NO$ requires 190.1232, found 190.1236.



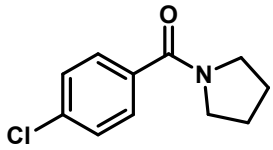
Pyrrolidin-1-yl(m-tolyl)methanone (57)



Prepared according to general procedure. Purified via flash chromatography (n-hexane:EtOAc, 4:1); 164 mg (86%) as a colourless oil. ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 1.86 (quin, *J* 6.4, 2H), 1.95 (quin, *J* 6.9, 2H), 2.37 (s, 3H), 3.41 (t, *J* 6.6, 2H), 3.64 (t, *J* 6.8, 2H), 7.20-7.21 (m, 1H), 7.24-7.29 (m, 2H), 7.33 (brs, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ = 21.3, 24.4, 26.3, 46.0, 49.5, 127.6, 127.9, 130.3, 137.2, 137.9, 169.8; HRMS (ESI): calcd. for $[M^+ + Na]$ $C_{12}H_{15}NNaO$ requires 212.1046, found 212.1062.

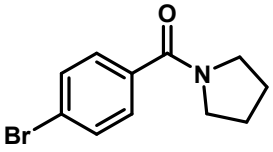
(4-chlorophenyl)(pyrrolidin-1-yl)methanone (58)

Prepared according to general procedure. Purified via flash chromatography (EtOAc:n-hexane:, 4:1); 203 mg (97%) as an off white solid. M.p. 68-70°C. ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 1.88 (app. quin, 2H), 1.96 (app. quin, 2H), 3.41 (t, *J* 6.6, 2H), 3.64 (t, *J* 6.6, 2H), 7.37 (d, *J* 8.5, 2H), 7.47 (d, *J* 8.5, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ = 24.6, 26.6, 46.4, 49.7, 128.6, 128.8, 135.7, 136.0, 168.7; HRMS (ESI): calcd. for $[M^+ + Na]$ $C_{11}H_{12}ClNNaO$ requires 232.0500, found 232.0509.



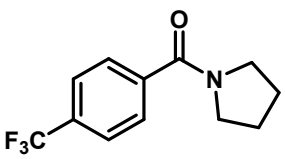
(4-Bromophenyl)(pyrrolidin-1-yl)methanone (59)

Prepared according to general procedure. Purified via flash chromatography (EtOAc:n-hexane:, 3:1); 246 mg (97%) as a white solid. M.p. 76-78°C. ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 1.89-1.96 (m, 4H), 3.41 (brs, 2H), 3.63 (brs, 2H), 7.40 (d, *J* 8.4, 2H), 7.53 (d, *J* 8.4, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ = 24.5, 26.5, 46.4, 49.7, 124.2, 129.0, 131.6, 136.1, 168.7; HRMS (ESI): calcd. for $[M^+ + Na]$ $C_{11}H_{12}BrNNaO$ requires 275.9994, found 275.9996.



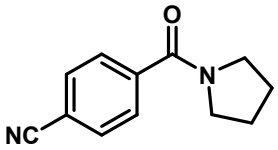
Pyrrolidin-1-yl(4-(trifluoromethyl)phenyl)methanone (60)

Prepared according to general procedure. Purified via flash chromatography (EtOAc:n-hexane:, 3:1); 234 mg (96%) as a pale yellow solid. M.p. 79-80°C. ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 1.86-1.93 (m, 2H), 1.95-2.01 (m, 2H), 3.38 (t, *J* 6.7, 2H), 3.66 (t, *J* 6.7, 2H), 7.62 (d, *J* 8.3, 2H), 7.67 (d, *J* 8.3, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ = 24.4, 26.4, 46.3, 49.5, 123.8 (q, *J* 272.6), 125.4 (q, *J* 3.8), 127.5, 131.6 (q, *J* 32.8), 140.8 (q, *J* 1.4), 168.2; HRMS (ESI): calcd. for $[M^+ + Na]$ $C_{12}H_{12}F_3NNaO$ requires 266.0763, found 266.0769.



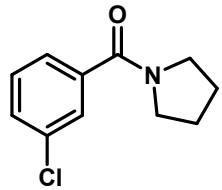
4-(Pyrrolidine-1-carbonyl)benzonitrile (61)

Prepared according to general procedure. Purified via flash chromatography (EtOAc:n-hexane:, 3:1); 172 mg (86%) as a yellow solid. M.p. 78-79°C. ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 1.88-1.94 (m, 2H), 1.95-2.02 (m, 2H), 3.37 (t, *J* 6.7, 2H), 3.66 (t, *J* 6.7, 2H), 7.61 (d, *J* 8.4, 2H), 7.71 (d, *J* 8.3, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ = 24.5, 26.5, 46.5, 49.6, 113.7, 118.3, 127.9, 132.4, 141.5, 167.8; HRMS (ESI): calcd. for [M^+ + Na] $C_{12}H_{12}N_2NaO$ requires 223.0842, found 223.0835.



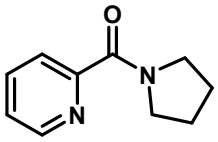
(3-Chlorophenyl)(pyrrolidin-1-yl)methanone (62)

Prepared according to general procedure. Purified via flash chromatography (EtOAc:n-hexane:, 3:1); 180 mg (93%) as a colourless oil. ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 1.84-1.91 (m, 2H), 1.92-1.99 (m, 2H), 3.40 (t, *J* 6.8, 2H), 3.62 (t, *J* 6.8, 2H), 7.30-7.39 (m, 3H), 7.49 (t, *J* 1.5, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ = 24.5, 26.5, 46.4, 49.7, 125.3, 127.4, 129.8, 130.0, 134.4, 139.0, 168.2; HRMS (ESI): calcd. for [M^+ + H] $C_{11}H_{13}ClNO$ requires 210.0680, found 210.0685.



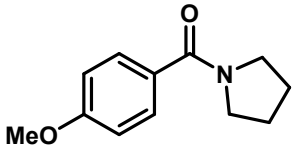
Pyridin-2-yl(pyrrolidin-1-yl)methanone (63)

Prepared according to general procedure. Purified via flash chromatography (EtOAc:n-hexane:, 9:1) 143 mg (81%) as a pale yellow solid. C ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 1.86-1.96 (m, 4H), 3.67 (t, *J* 6.6, 2H), 3.73 (t, *J* 6.6, 2H), 7.32 (ddd, *J* 1.6, 4.9, 6.5, 1H), 7.77 (m, 1H), 7.82 (m, 1H), 8.57 (d, *J* 4.9, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ = 23.9, 26.5, 46.7, 48.9, 123.6, 124.5, 136.7, 147.9, 154.5, 166.4; HRMS (ESI): calcd. for [M^+ + H] $C_{10}H_{13}N_2O$ requires 177.1022, found 177.1019.



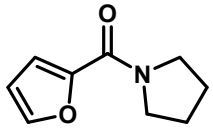
(4-Methoxyphenyl)(pyrrolidin-1-yl)methanone (64)

Prepared according to general procedure. Purified via flash chromatography (n-hexane:EtOAc, 4:1); 173 mg (84%) as a pale yellow solid. M.p. 74-76°C. ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 1.73-1.90 (br d, 4H), 3.46-3.64 (m, 4H), 3.83 (s, 3H), 6.87-6.91 (m, 2H), 7.49-7.53 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ = 24.5, 26.6, 46.4, 49.9, 55.4, 113.5, 129.2, 160.9, 169.5; HRMS (ESI): calcd. for [M^+ + H] $C_{12}H_{16}NO_2$ requires 206.1181, found 206.1177.



Furan-2-yl(pyrrolidin-1-yl)methanone (65)

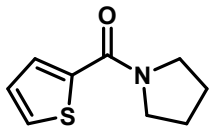
Prepared according to general procedure. Purified via flash chromatography (EtOAc:n-hexane:, 3:1); 159 mg (96%) as a yellow oil. ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 1.89 (quin, *J* 6.7, 2H), 1.99 (quin, *J* 6.7, 2H), 3.65 (t, *J* 6.9, 2H), 3.82 (t, *J* 6.7, 2H), 6.47 (q, *J* 1.7, 1H), 7.05 (dd, *J* 0.7, 3.5, 1H), 7.49 (dd, *J* 0.8, 1.7, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ = 23.7, 26.6, 47.0,



47.8, 111.3, 115.6, 144.0, 148.8, 158.1; HRMS (ESI): calcd. for [M^+ + Na] $C_9H_{11}NNaO$ requires 188.0681, found 188.0675.

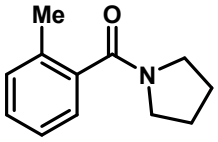
Pyrrolidin-1-yl(thiophen-2-yl)methanone (67)

Prepared according to general procedure. Purified via flash chromatography (EtOAc:n-hexane:, 3:1); 177 mg (98%) as a yellow semisolid. ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 1.91-2.01 (m, 4H), 3.66 (t, *J* 6.0, 2H), 3.76 (t, *J* 6.0, 2H), 7.06 (dd, *J* 3.9, 5.0, 1H), 7.46 (dd, *J* 0.8, 5.0, 1H), 7.51 (dd, *J* 0.7, 3.9, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ = 24.2, 26.8, 47.4, 49.0, 127.1, 129.58, 129.62, 139.7, 161.9; HRMS (ESI): calcd. for [M^+ + H] $C_9H_{12}NOS$ requires 182.0640, found 182.0637.



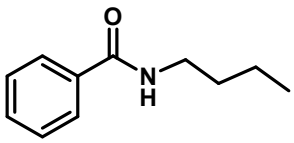
Pyrrolidin-1-yl(o-tolyl)methanone (67)

Prepared according to general procedure. Purified via flash chromatography (EtOAc:n-hexane:, 3:1); 133 mg (70%) as a colourless oil. ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 1.84-1.89 (m, 2H), 1.93-1.97 (m, 2H), 2.31 (s, 3H), 3.13 (t, *J* 6.6, 2H), 3.66 (t, *J* 6.6, 2H), 7.18-7.21 (m, 3H), 7.23-7.27 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ = 19.1, 24.8, 26.2, 45.4, 48.5, 125.7, 126.0, 128.9, 130.5, 133.9, 137.9, 170.1; HRMS (ESI): calcd. for [M^+ + Na] $C_{12}H_{15}NNaO$ requires 212.1046, found 212.1070.



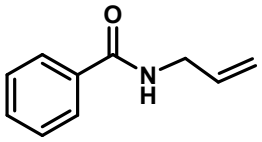
N-Butylbenzamide (70)

Prepared according to general procedure. Purified via flash chromatography (n-hexane:EtOAc, 4:1); 137 mg (77%) as a light yellow oil. ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 0.94 (td, *J* 1.2, 7.4, 3H), 1.35-1.45 (m, 2H), 1.59 (quin, J 7.2, 2H), 3.41-3.47 (m, 2H), 6.14 (br s, 1H), 7.38-7.42 (m, 2H), 7.45-7.49 (m, 1H), 7.27-7.75 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ = 13.9, 20.3, 31.9, 39.9, 126.9, 128.7, 131.4, 135.0, 167.6; HRMS (ES): calcd. for [M^+ + H] $C_{11}H_{16}NO$ requires 178.1232, found 178.1237.



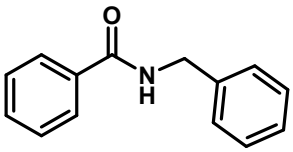
N-allylbenzamide (71)

Prepared according to general procedure. Purified via flash chromatography (n-hexane:EtOAc, 4:1); 129 mg (80%) as a light yellow semisolid. ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 4.08-4.11 (m, 2H), 5.19 (dd, *J* 1.2, 10.2, 1H), 5.27 (dd, *J* 1.4, 17.1, 1H), 5.89-5.99 (m, 1H), 6.23 (br s, 1H), 7.41-7.45 (m, 2H), 7.48-7.52 (m, 1H), 7.77-7.79 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ = 42.5, 116.6, 127.0, 127.1, 128.61, 128.62, 131.5, 134.3, 134.6, 167.5; HRMS (ES): calcd. for [M^+ + H] $C_{10}H_{12}NO$ requires 162.0919, found 162.0914.



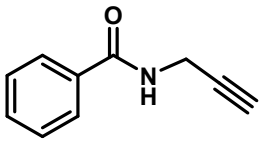
N-Benzylbenzamide (72)

Prepared according to general procedure. Purified via flash chromatography (EtOAc:n-hexane:, 3:7); 165 mg (78%) as an off whit solid. M.p. 94-96°C. ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 4.66 (d, *J* 5.6, 2H), 6.40 (br s, 1H), 7.28.737 (m, 5H), 7.41-7.45 (m, 2H), 7.49-7.52 (m, 1H), 7.78-7.80 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ = 44.3, 127.1, 127.8, 128.1, 128.73, 128.75, 128.9, 131.7, 134.5, 138.3, 167.5; ; HRMS (ESI): calcd. for $[M^+ + H]$ $C_{14}H_{14}NO$ requires 212.1075, found 212.1080.



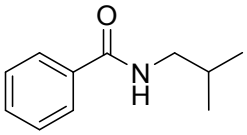
N-(Prop-2-yn-1-yl)benzamide (73)

Prepared according to general procedure. Purified via flash chromatography (n-hexane:EtOAc, 4:1); 116 mg (73%) as a white solid. M.p. 105-107°C. ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 2.28 (t, *J* 2.6, 1H), 4.26 (q, *J* 2.6), 6.36 (brs, 1H), 7.42-7.45 (m, 2H), 7.50-7.53 (m, 1H), 7.77-7.79 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ = 29.9, 72.0, 79.6, 127.1, 128.76, 128.77, 131.9, 133.9, 167.2; HRMS (ES): calcd. for $[M^+ + Na]$ $C_{10}H_9NNaO$ requires 182.0576, found 182.0563.



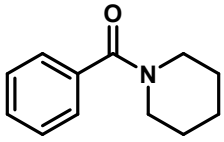
N-Isobutylbenzamide (74)

Prepared according to general procedure. Purified via flash chromatography (n-hexane:EtOAc, 4:1); 126 mg (71%) as a light yellow solid. M.p. 49-51°C. ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 0.95 (dd, *J* 1.3, 6.7, 6H), 1.83-1.93 (m, 1H), 3.23-3.26 (m, 2H), 6.48 (brs, 1H), 7.36-7.40 (m, 2H), 7.44-7.47 (m, 1H), 7.74-7.77 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ = 20.2, 28.6, 47.4, 126.9, 128.5, 131.3, 134.9, 167.8; HRMS (ES): calcd. for $[M^+ + H]$ $C_{11}H_{16}NO$ requires 178.1226, found 178.1227.



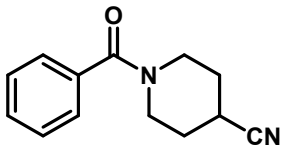
Phenyl(piperidin-1-yl)methanone (75)

Prepared according to general procedure. Purified via flash chromatography (EtOAc:n-hexane, 3:2); 152 mg (80%) as a pale yellow solid. ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 1.51 (brs, 2H), 1.67 (brs, 4H), 3.33 (brs, 2H), 3.70 (brs, 2H), 7.38 (brs, 5H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ = 24.7, 25.7, 26.7, 43.2, 48.8, 126.9, 128.5, 129.4, 136.6, 170.4; HRMS (ESI): calcd. for $[M^+ + H]$ $C_{12}H_{16}NO$ requires 190.1232, found 190.1233.



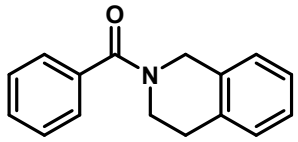
1-Benzoylpiperidine-4-carbonitrile (76)

Prepared according to general procedure. Purified via flash chromatography (n-hexane:EtOAc, 3:1); 157 mg (73%) as a white solid. M.p. 91-92°C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 1.90 (brs, 4H), 2.90-2.95 (m, 1H), 3.45-3.90 (m, 4H), 7.37-7.44 (m, 5H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ = 26.4, 28.7, 40.1, 45.6, 120.7, 126.8, 128.6, 129.9, 135.4, 170.4; HRMS (ES): calcd. for $[M^+ + H]$ $C_{13}H_{15}N_2O$ requires 215.1178, found 215.1186.



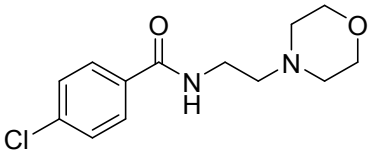
(3,4-Dihydroisoquinolin-2(1H)-yl)(phenyl)methanone (77)

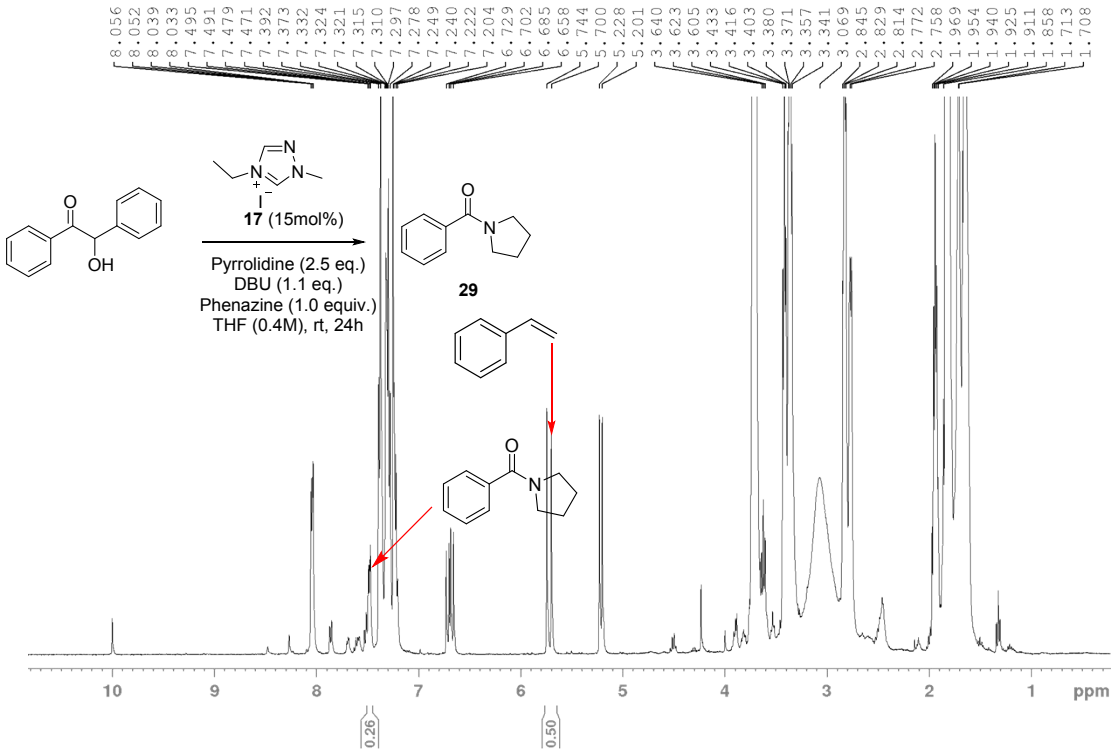
Prepared according to general procedure. Purified via flash chromatography (EtOAc:n-hexane, 2:3); 197 mg (83%) as a light yellow solid. M.p. 127-129°C. ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 2.87-297 (app. d, 2H), 3.64 (br s, 1.23 H), 3.99 (br s, 0.82H), 4.59 (br s, 0.82H), 4.90 (br s, 1.22H), 7.18-7.21 (m, 4H), 7.42-7.47 (m, 5H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ = 28.3, 29.6, 40.5, 44.9, 45.3, 49.8, 125.9, 126.6, 126.9, 128.5, 128.7, 129.0, 129.8, 132.9, 133.9, 133.9, 134.7, 134.8, 136.1, 170.5, 170.9; HRMS (ESI): calcd. for $[M^+ + H]$ $C_{16}H_{16}NO$ requires 238.1232, found 238.1230.



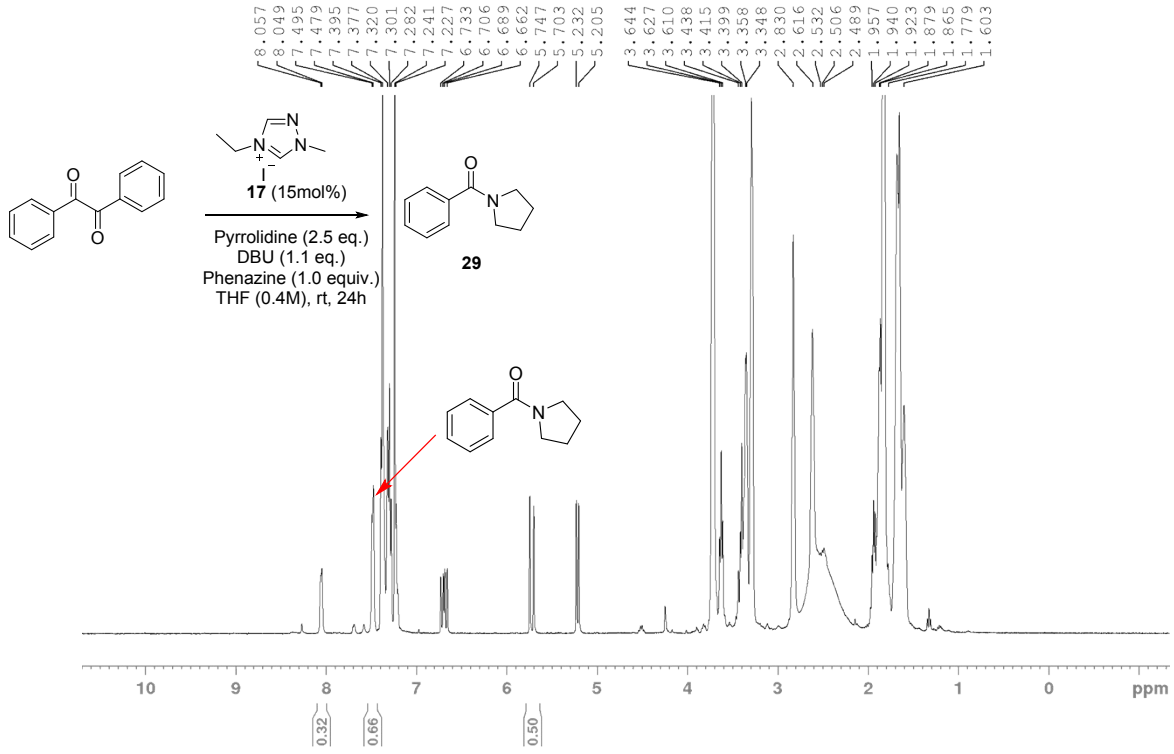
4-Chloro-N-(2-morpholinoethyl)benzamide (Moclobemide)

Prepared according to general procedure. Purified via flash chromatography (DCM:MeOH, 19:1); 247 mg (92%) as an off white solid. M.p. 130-132°C. ^{1}H NMR ($CDCl_3$, 400 MHz) δ = 2.52 (brs, 4H), 2.61 (t, *J* 5.6, 2H), 3.55 (q, *J* 5.5, 2H), 3.72-3.74 (m, 4H), 6.77 (brs, 1H), 7.41 (d, *J* 8.4, 2H), 7.72 (d, *J* 8.3, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ = 36.1, 53.4, 57.0, 67.0, 128.5, 128.9, 133.1, 137.8, 166.4; HRMS (ES): calcd. for $[M^+ + H]$ $C_{13}H_{18}ClN_2O_2$ requires 269.1051, found 269.1059.

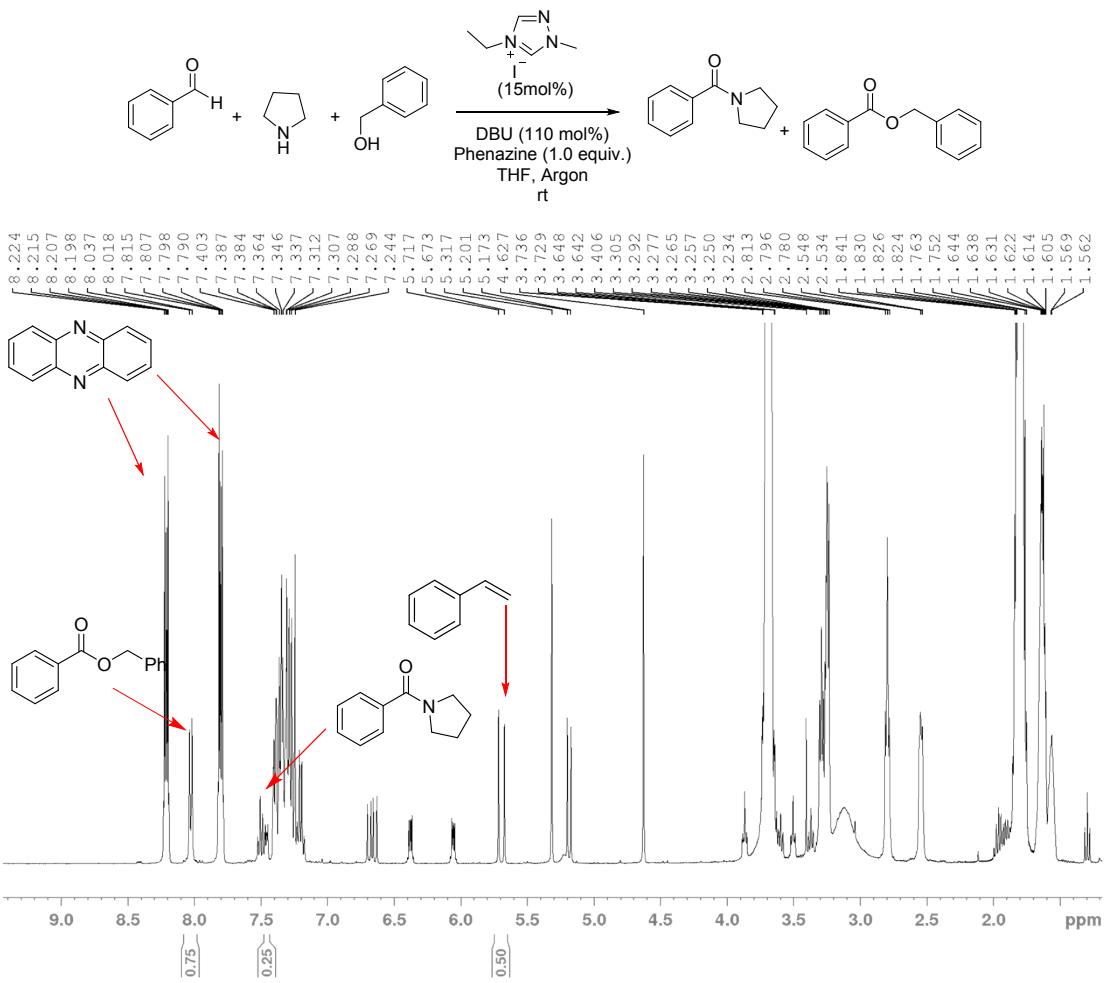




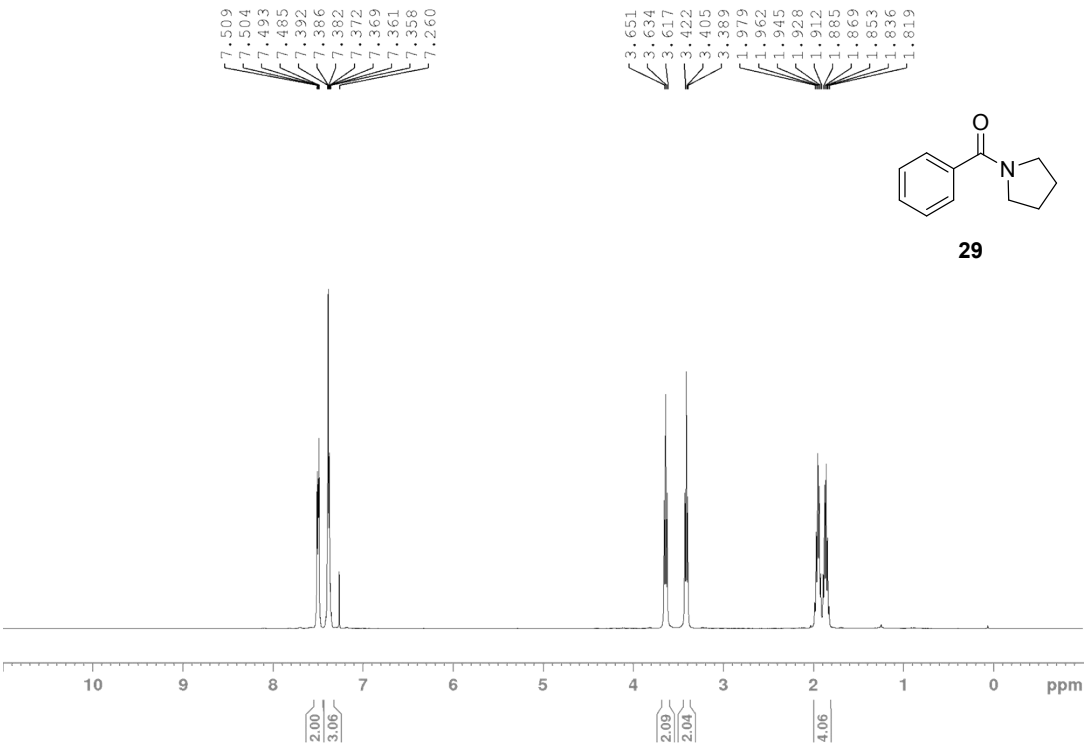
Oxidative amidation of benzoin: ^{1}H NMR spectrum of the crude ($CDCl_3$, 400 MHz)



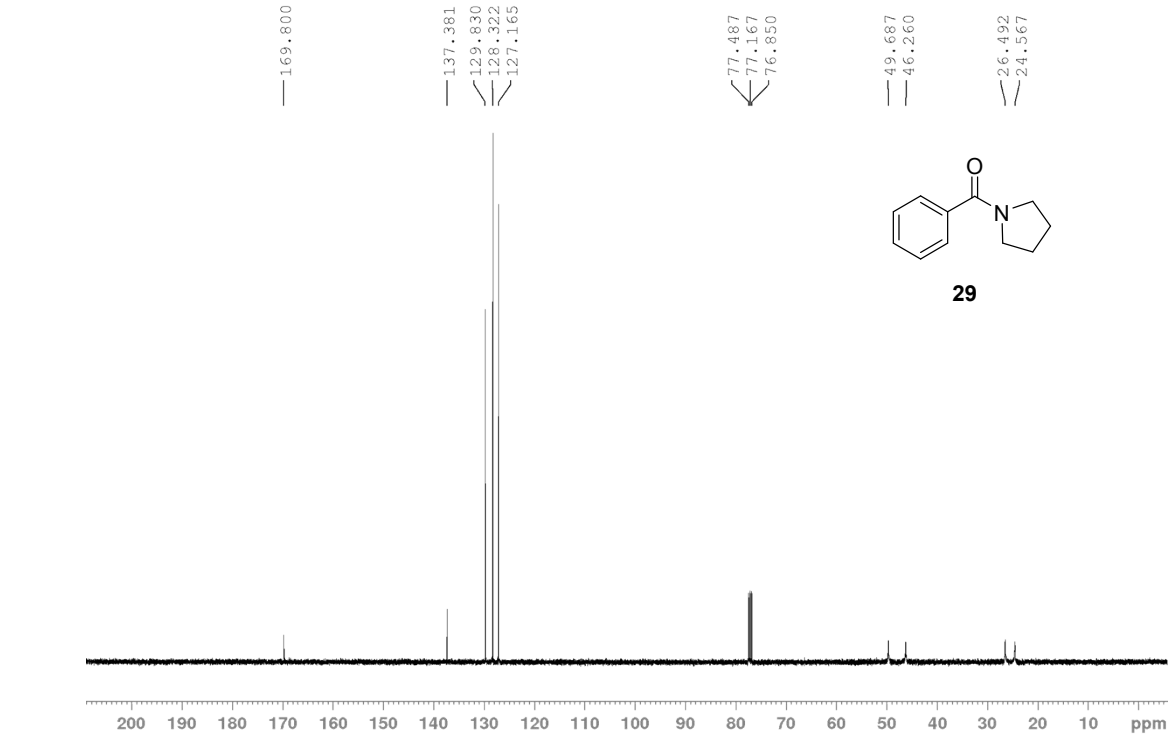
Oxidative amidation of benzil: ^{1}H NMR spectrum of the crude ($CDCl_3$, 400 MHz)



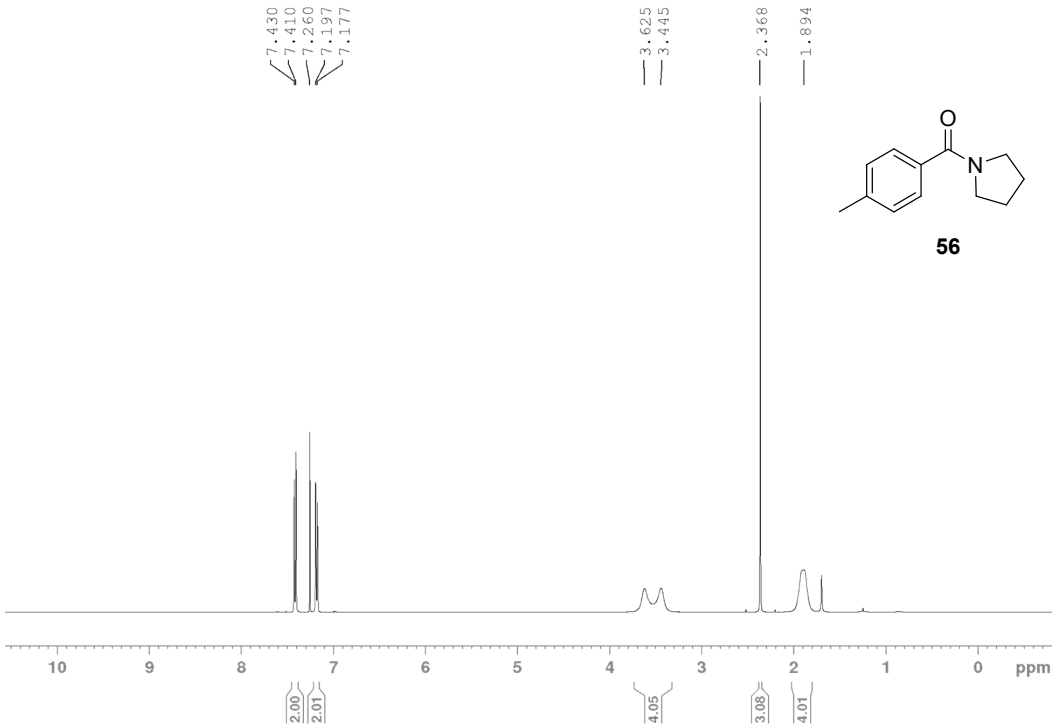
Competition between benzyl alcohol and pyrrolidie in oxidative coupling: ^{1}H NMR spectrum of the crude ($CDCl_3$, 400 MHz)



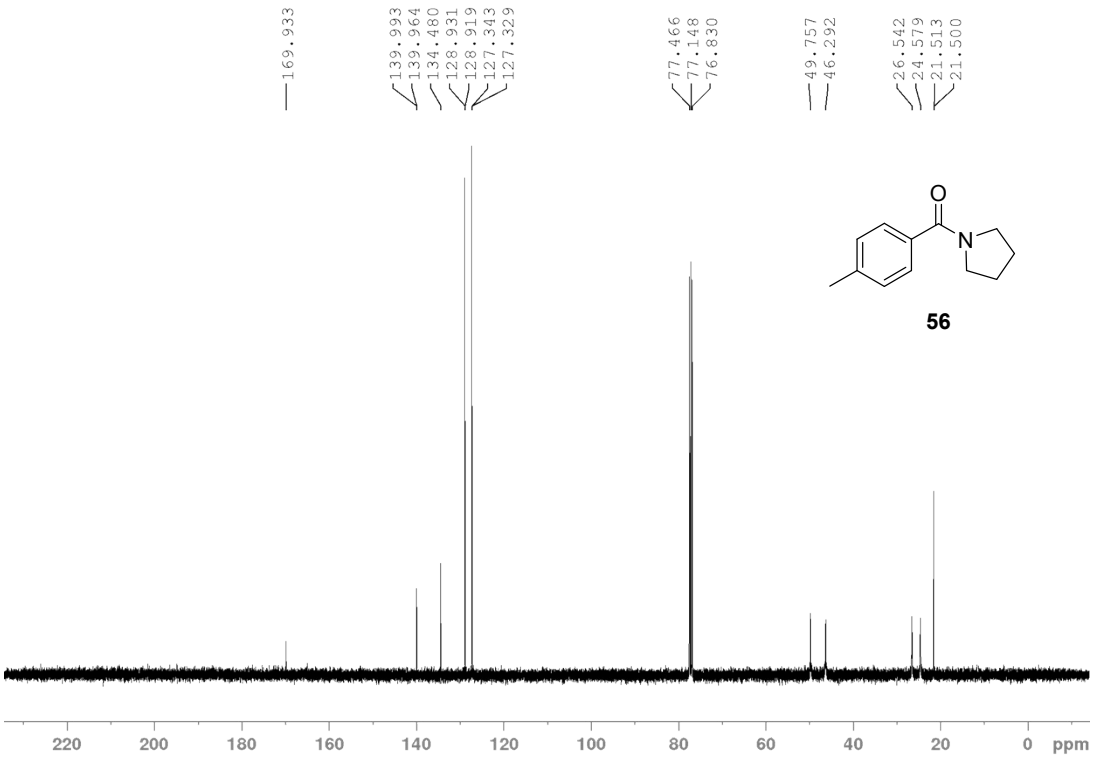
^{1}H NMR ($CDCl_3$, 400 MHz) Spectrum of Compound 29



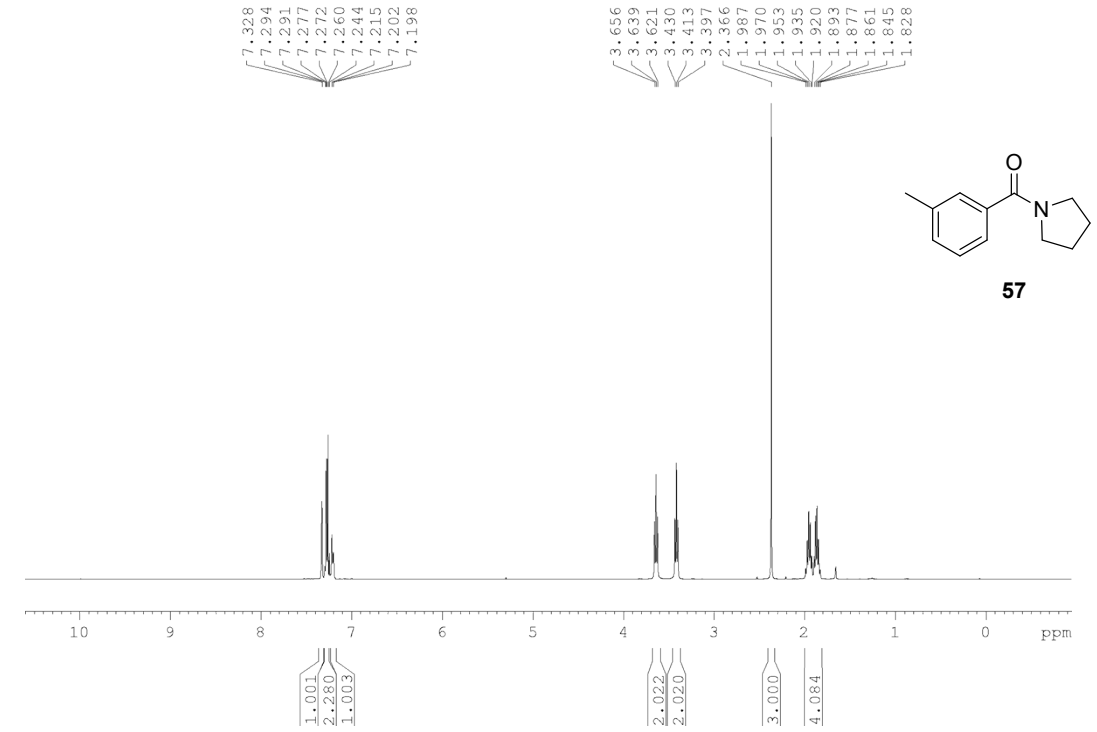
^{13}C NMR ($CDCl_3$, 100 MHz) Spectrum of Compound 29



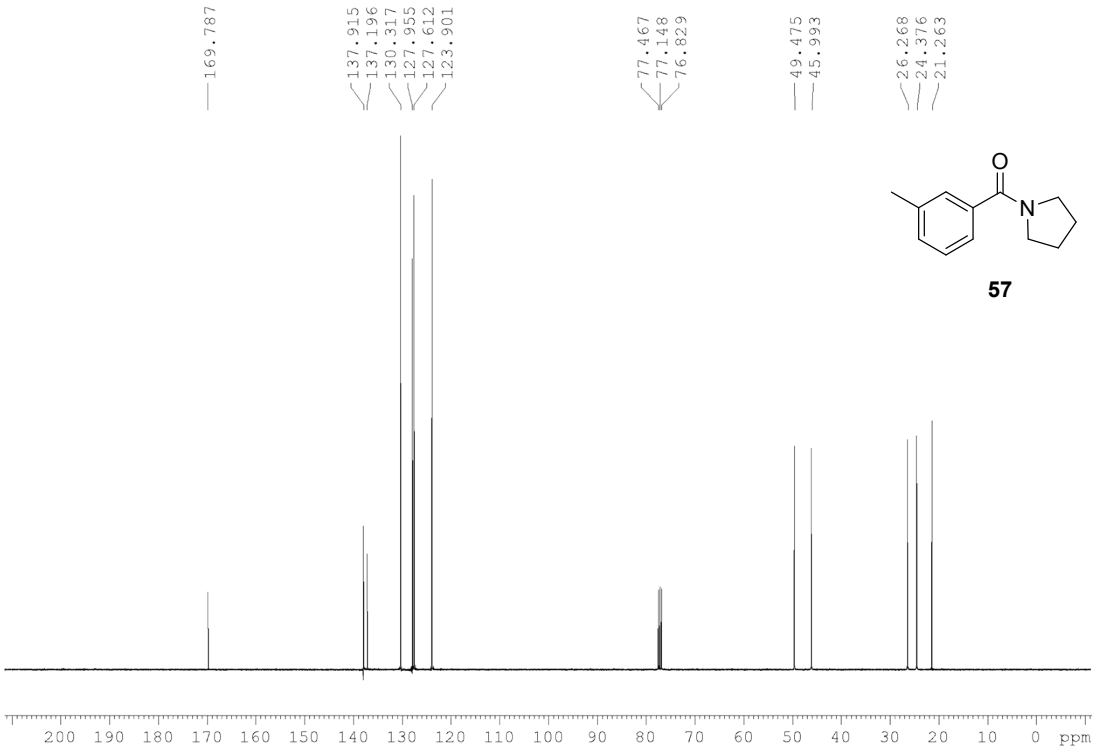
^{1}H NMR ($CDCl_3$, 400 MHz) Spectrum of Compound 56



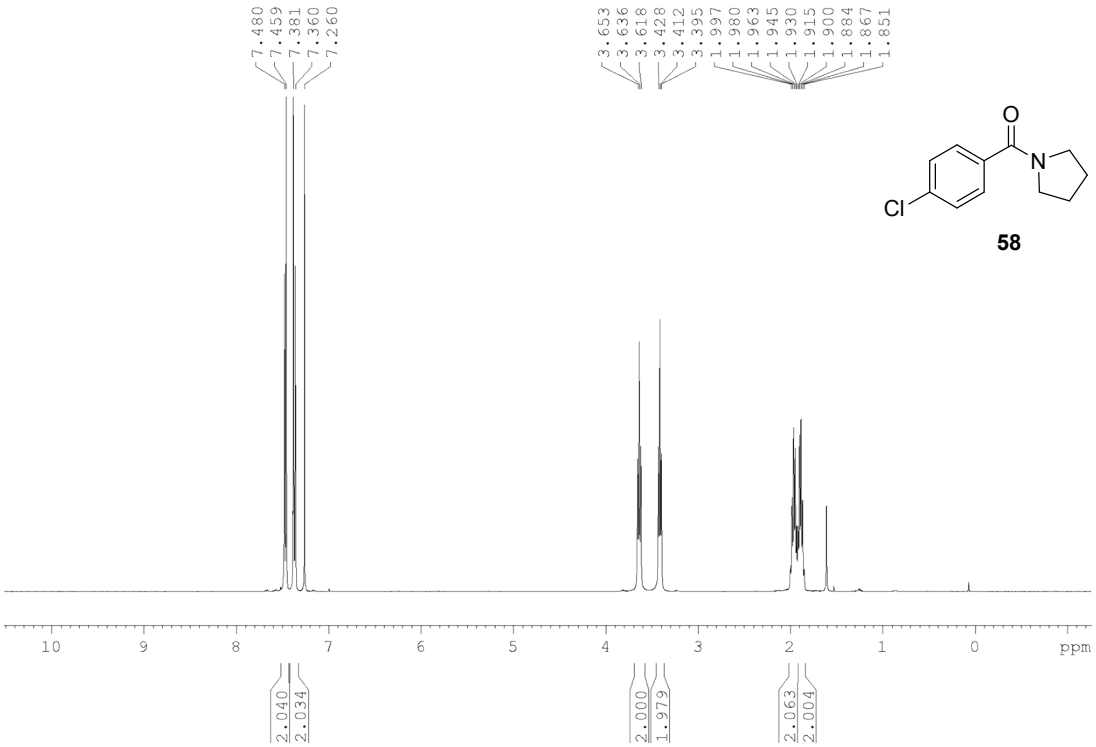
^{13}C NMR ($CDCl_3$, 100 MHz) Spectrum of Compound 56



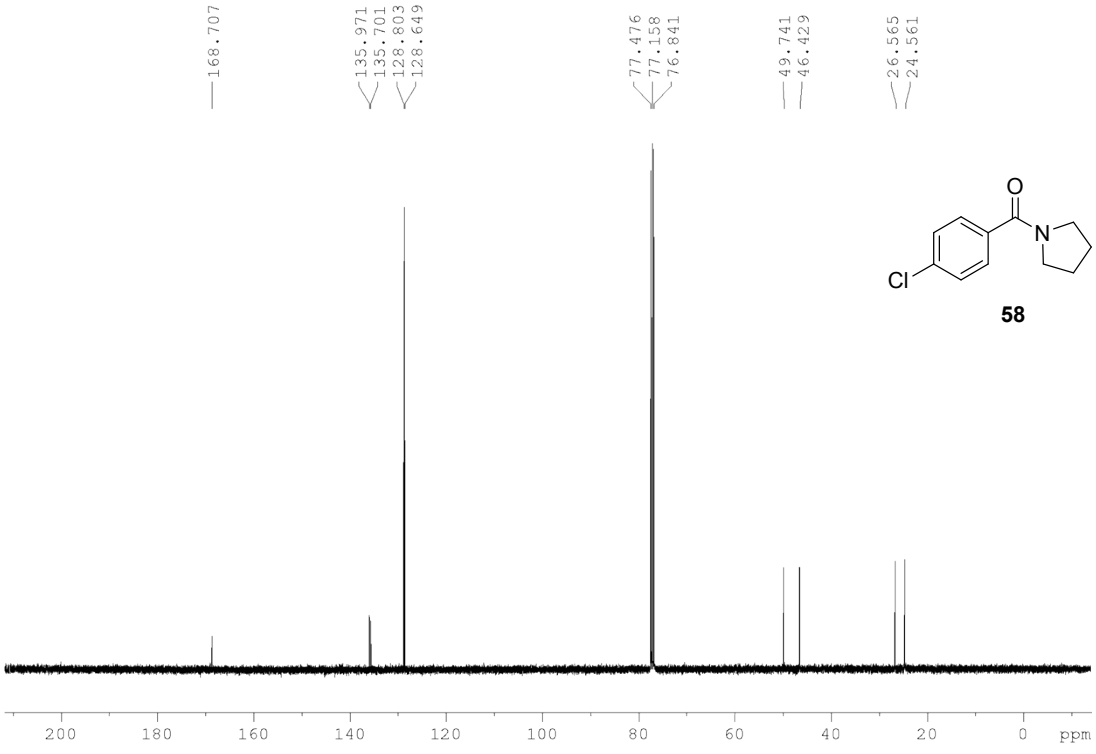
^{1}H NMR ($CDCl_3$, 400 MHz) Spectrum of Compound 57



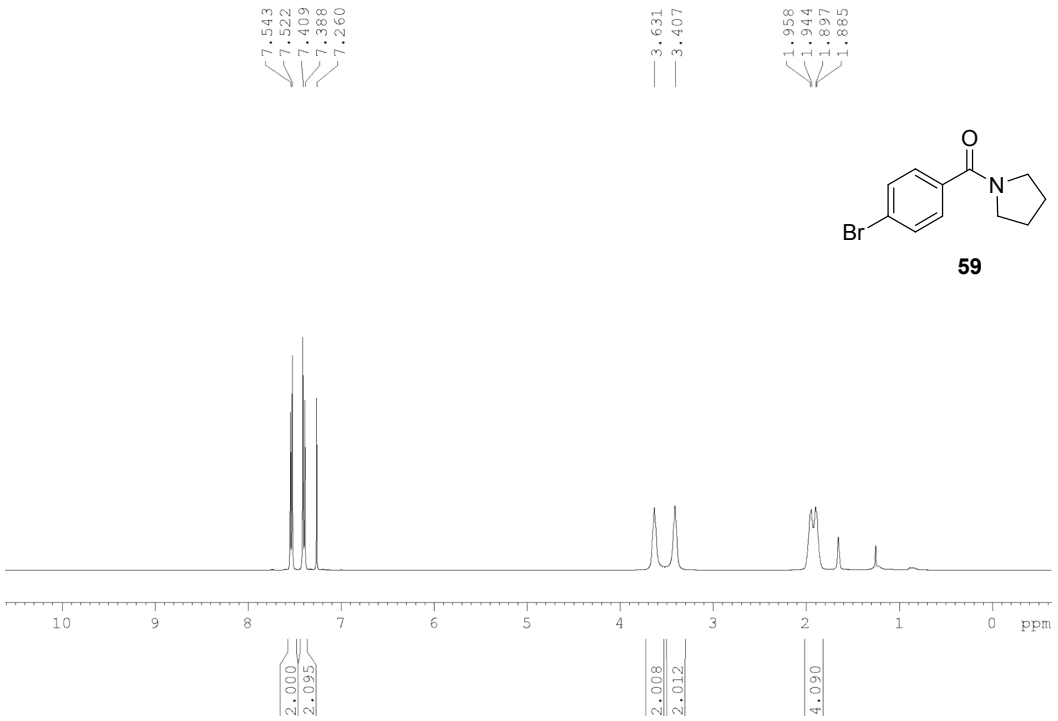
^{13}C NMR ($CDCl_3$, 100 MHz) Spectrum of Compound 57



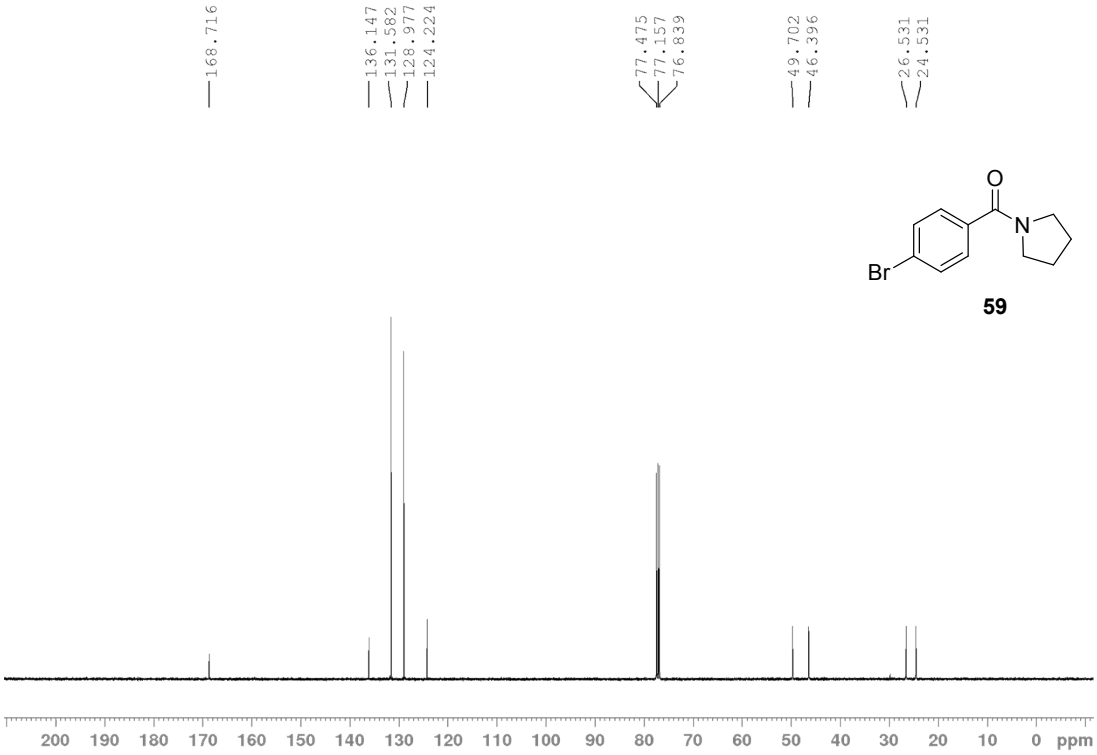
^{1}H NMR ($CDCl_3$, 400 MHz) Spectrum of Compound 58



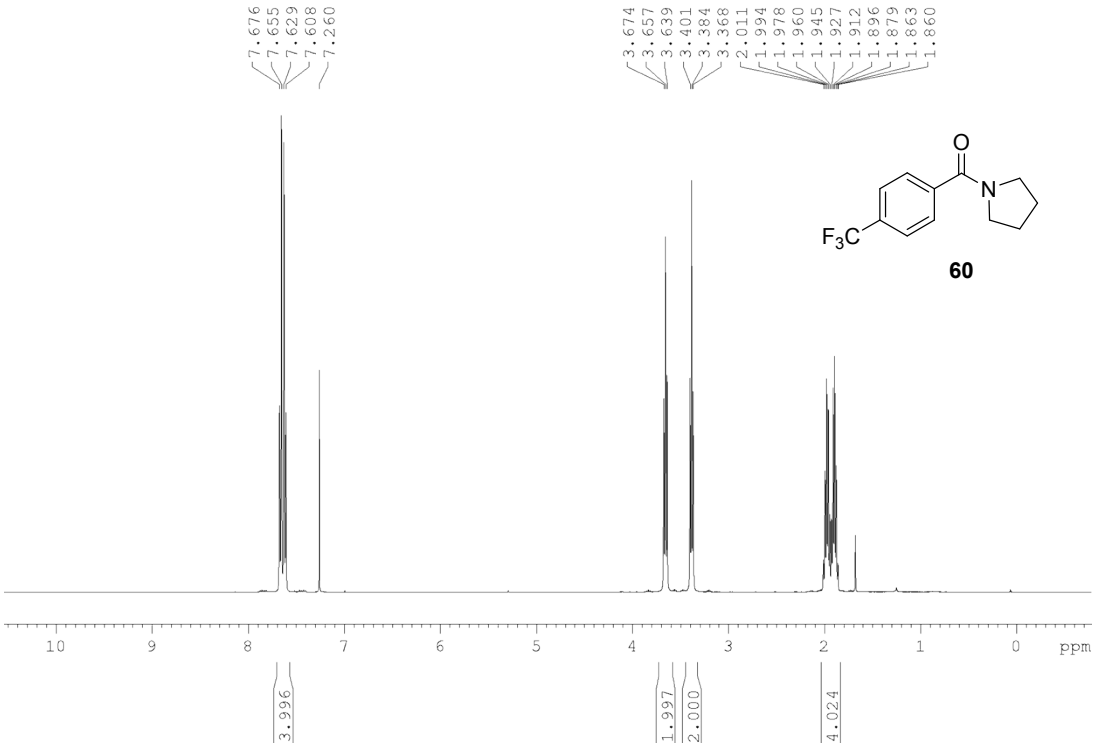
^{13}C NMR ($CDCl_3$, 100 MHz) Spectrum of Compound 58



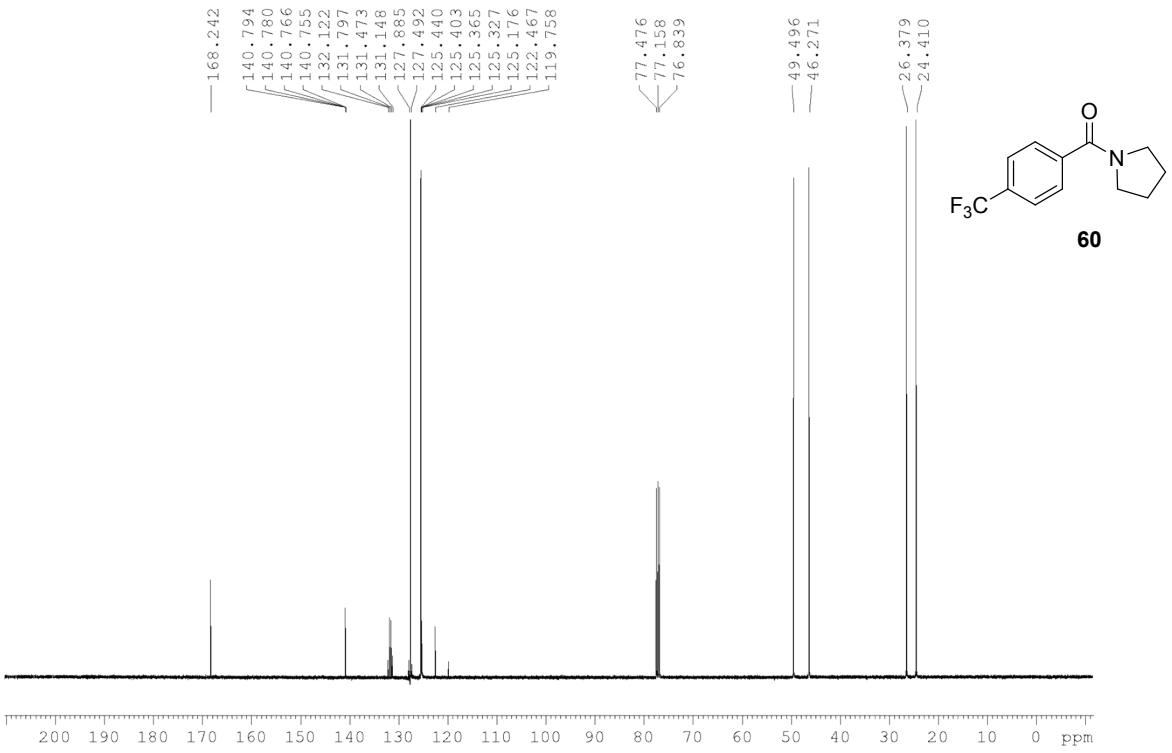
^{1}H NMR ($CDCl_3$, 400 MHz) Spectrum of Compound 59



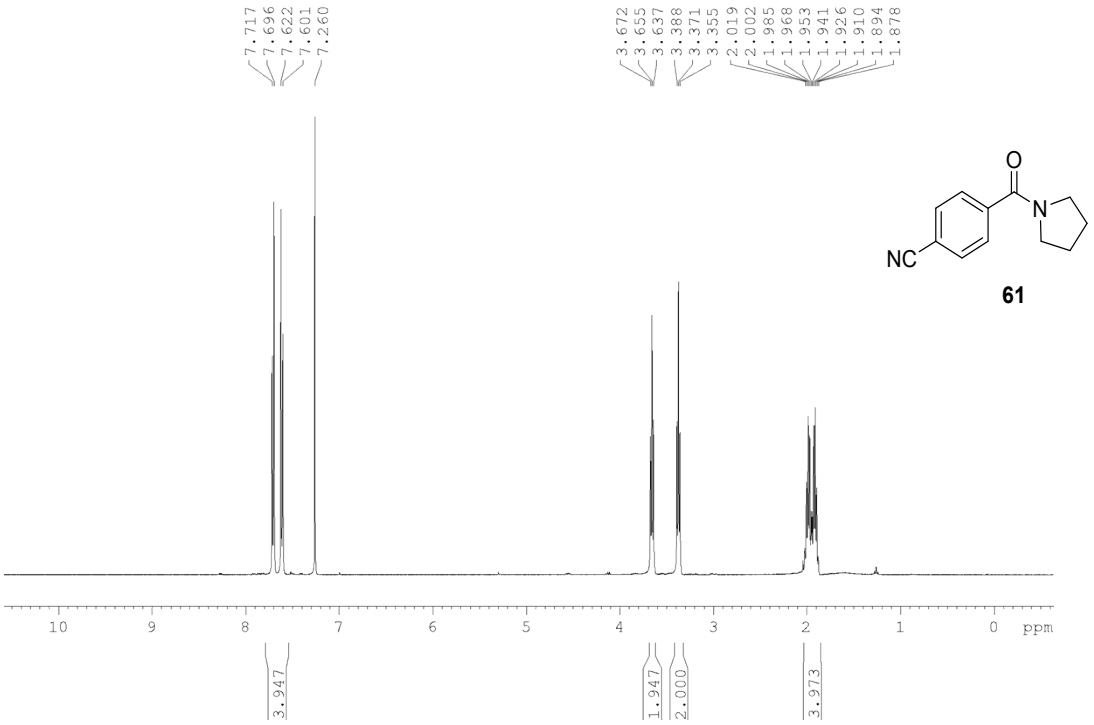
^{13}C NMR ($CDCl_3$, 100 MHz) Spectrum of Compound 59



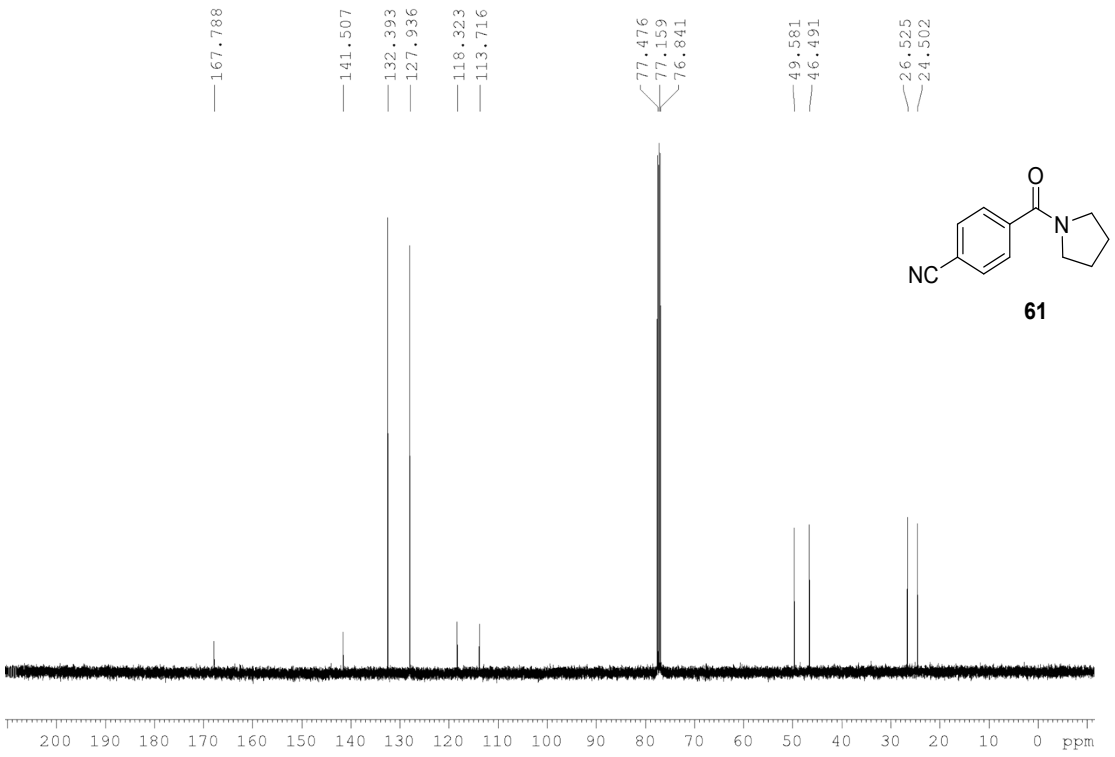
^{1}H NMR ($CDCl_3$, 400 MHz) Spectrum of Compound 60



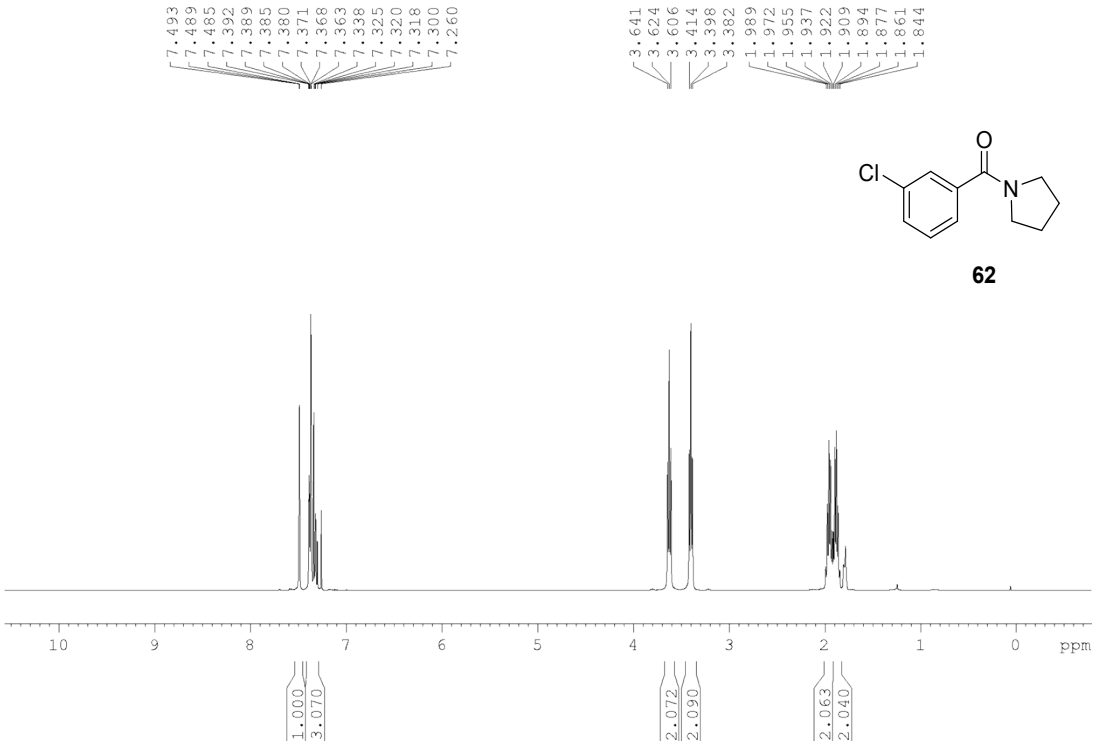
^{13}C NMR ($CDCl_3$, 100 MHz) Spectrum of Compound 60



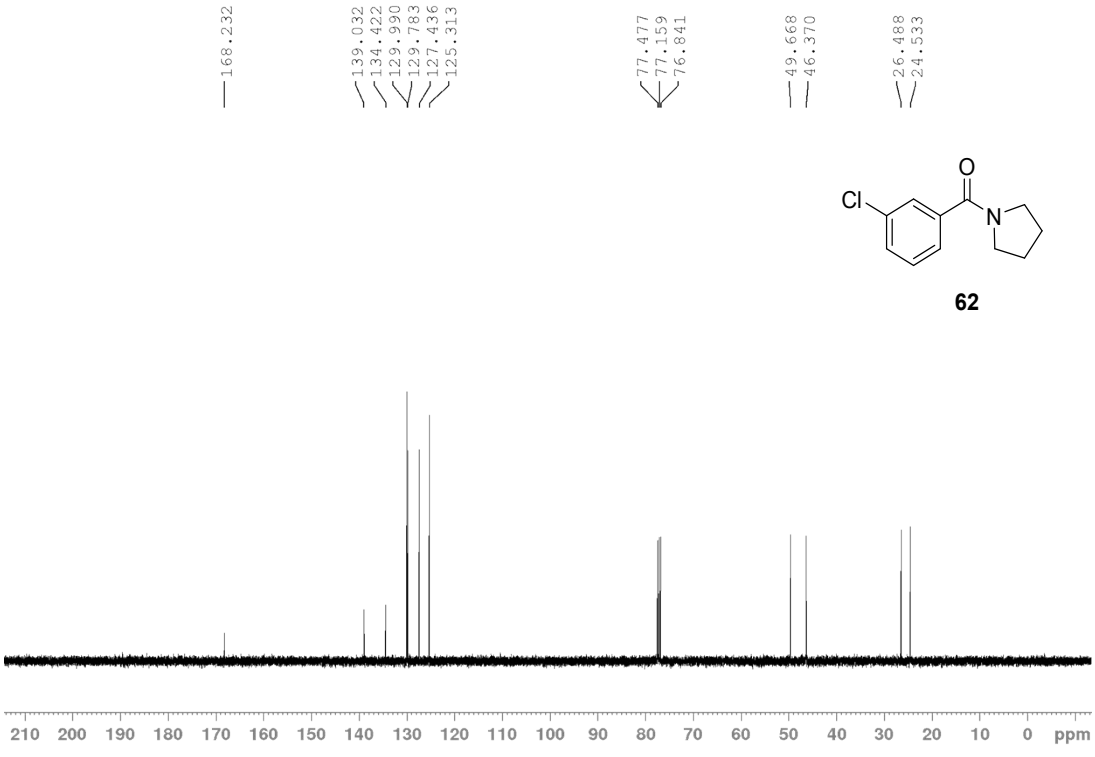
^{1}H NMR ($CDCl_3$, 400 MHz) Spectrum of Compound 61



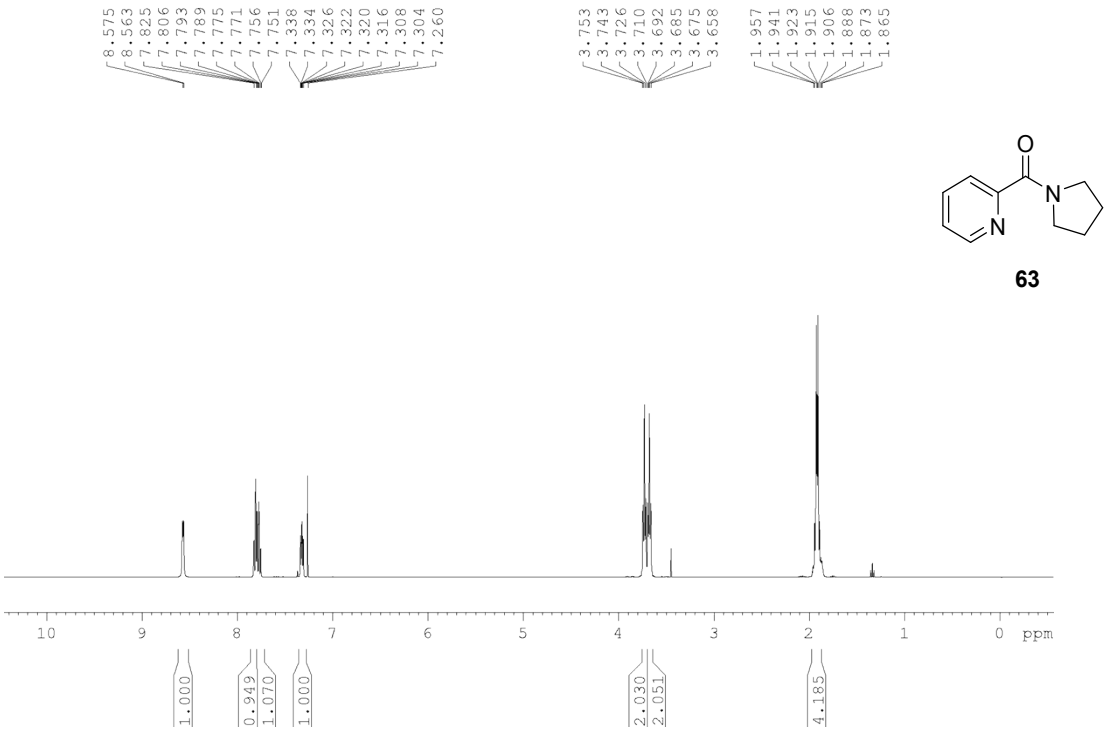
^{13}C NMR ($CDCl_3$, 100 MHz) Spectrum of Compound 61



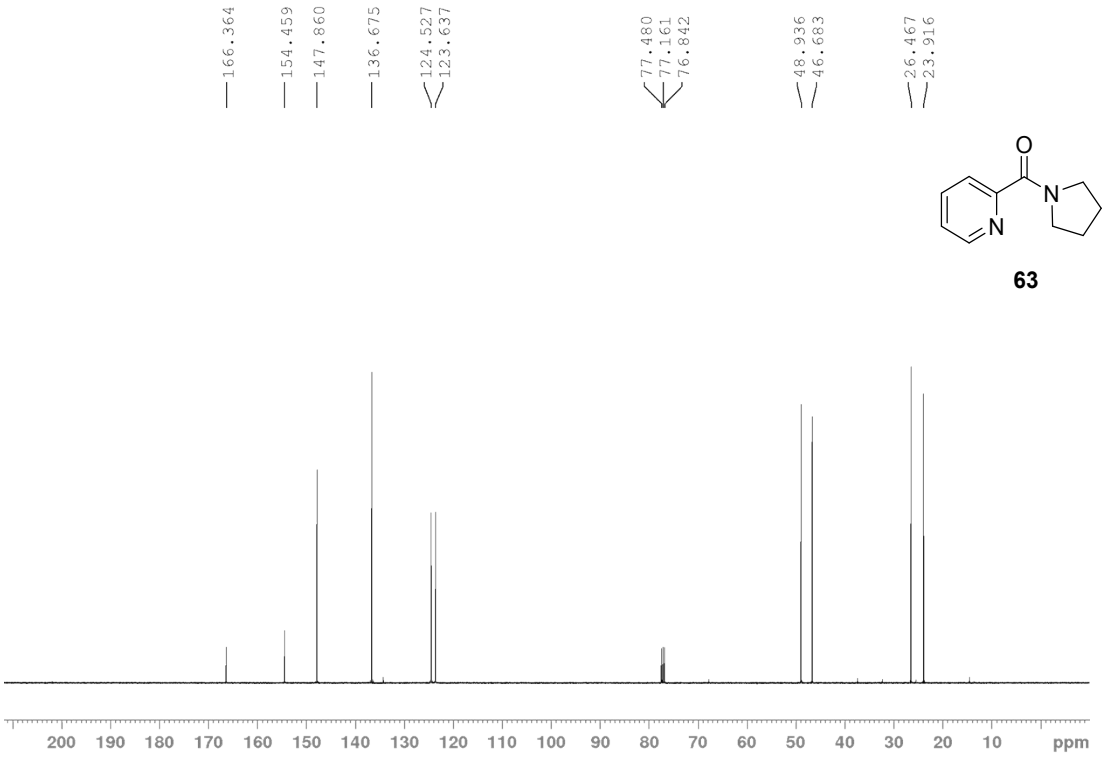
^{1}H NMR ($CDCl_3$, 400 MHz) Spectrum of Compound 62



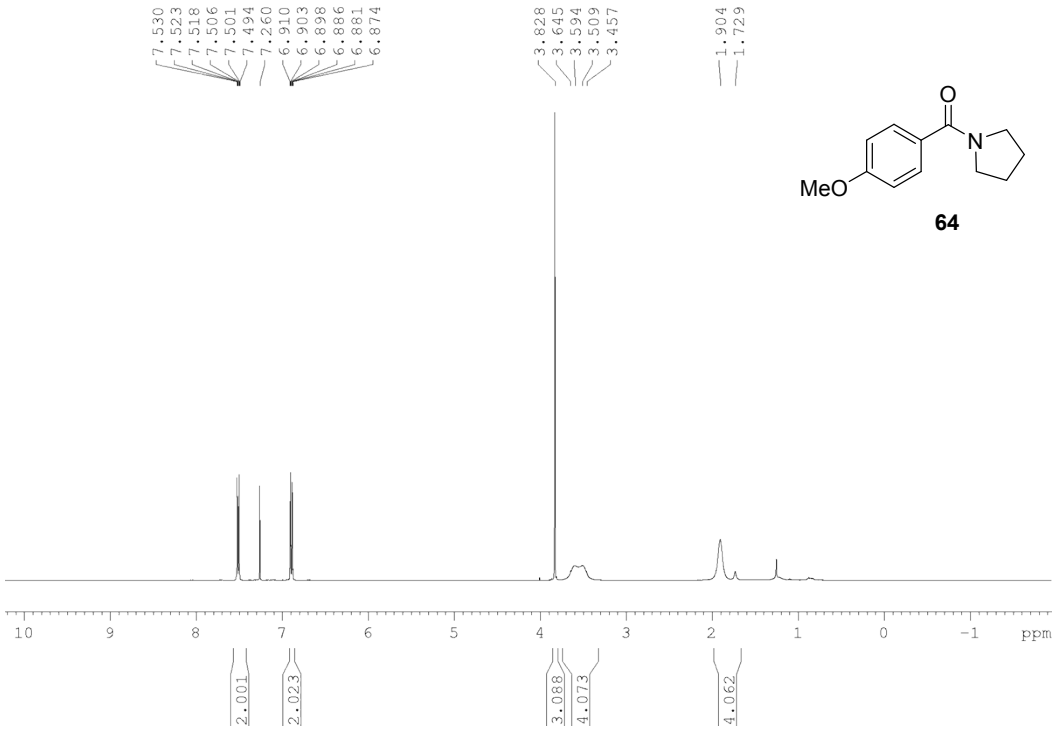
^{13}C NMR ($CDCl_3$, 100 MHz) Spectrum of Compound 62



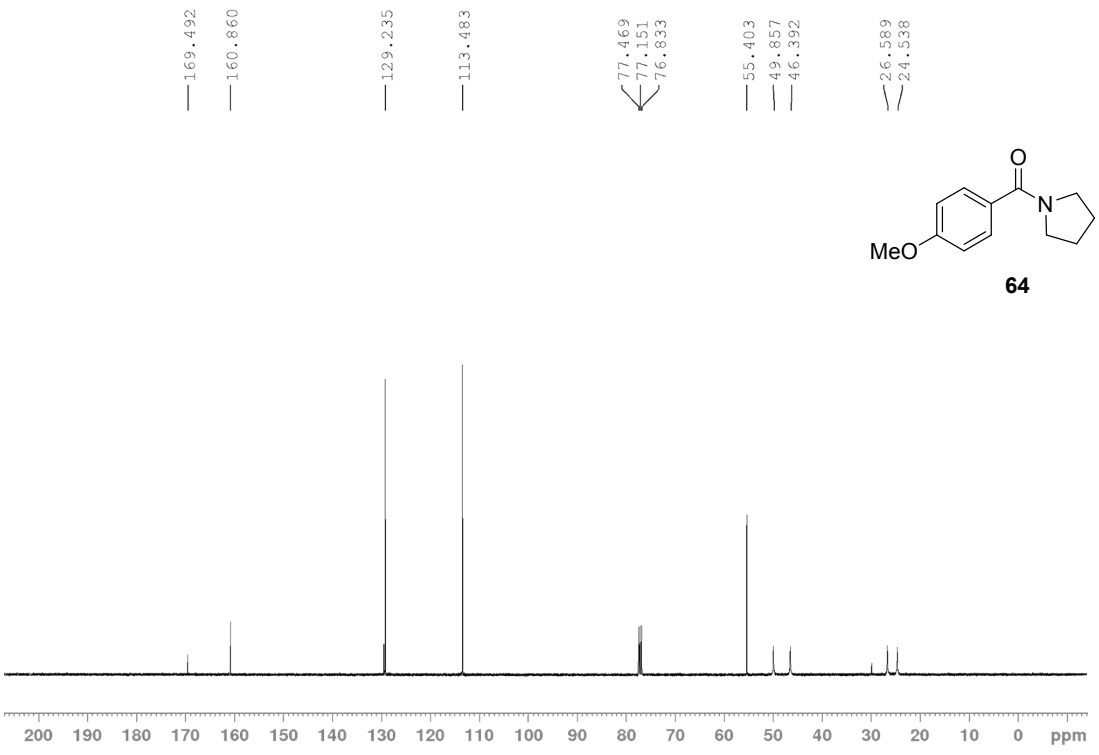
^{1}H NMR ($CDCl_3$, 400 MHz) Spectrum of Compound 63



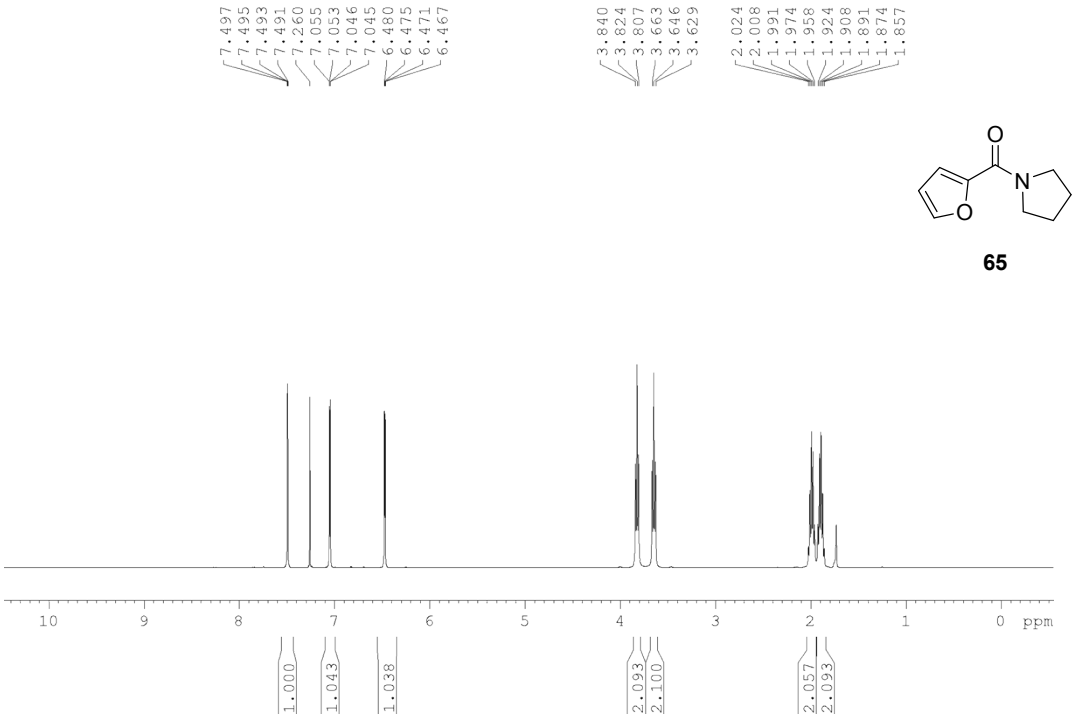
^{13}C NMR ($CDCl_3$, 100 MHz) Spectrum of Compound 63



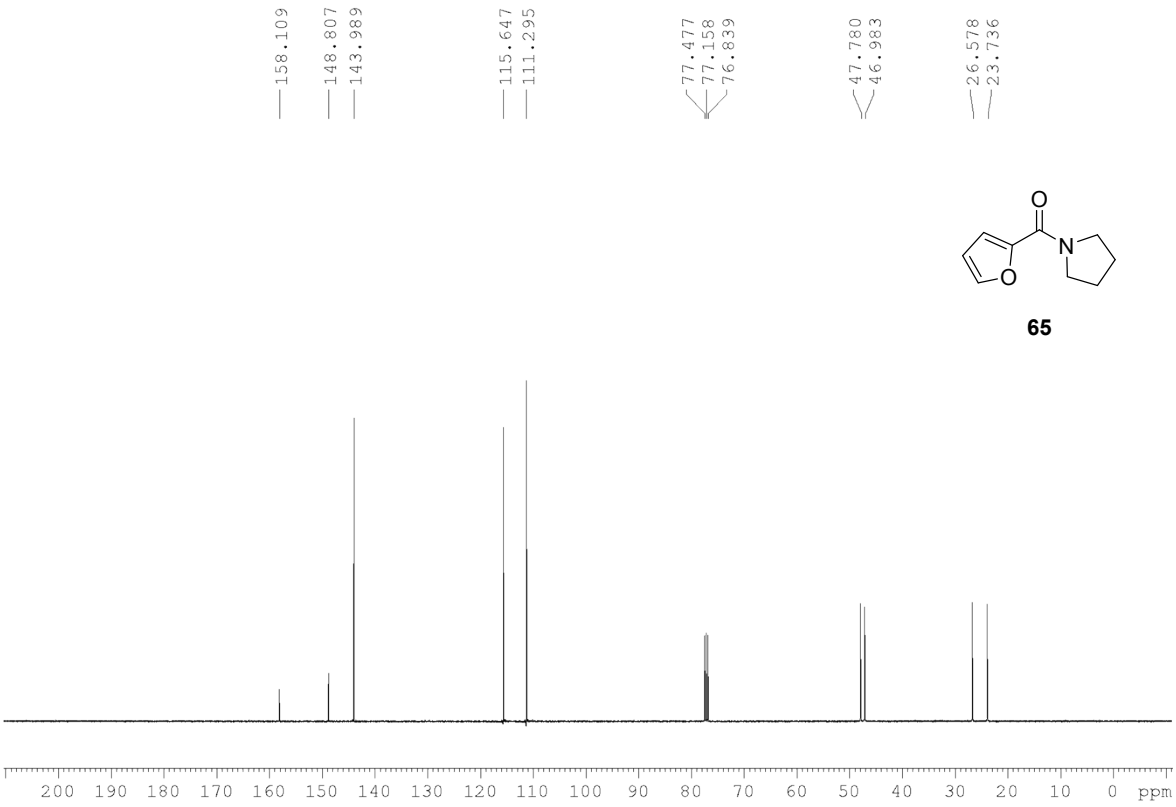
^{1}H NMR ($CDCl_3$, 400 MHz) Spectrum of Compound 64



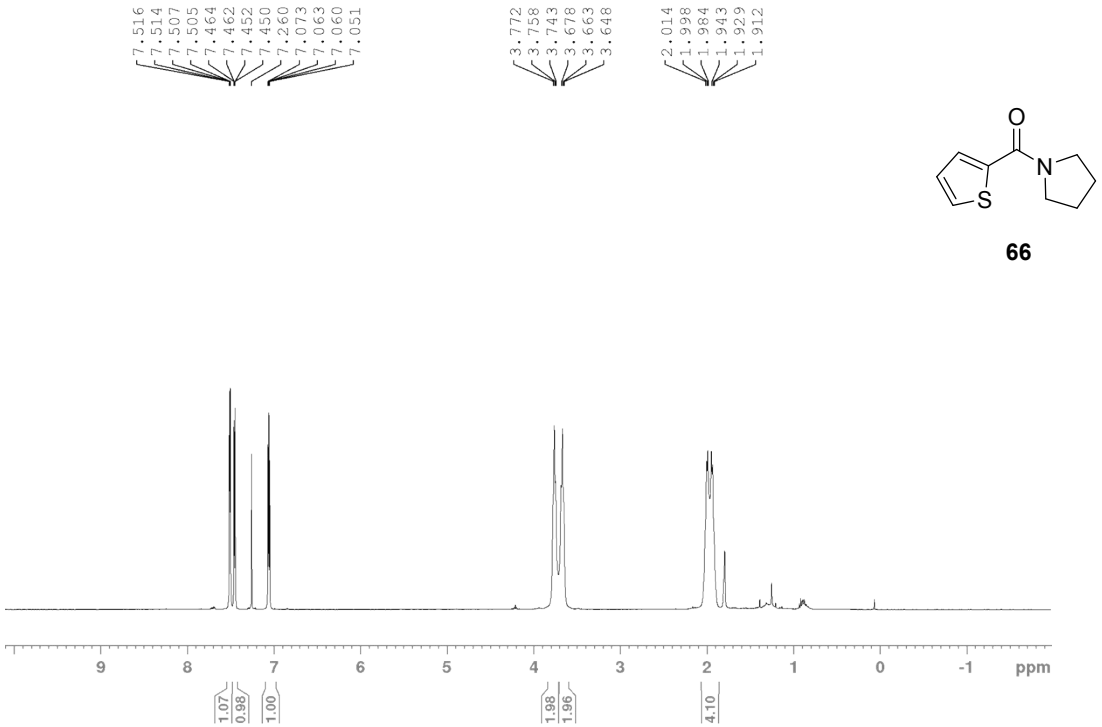
^{13}C NMR ($CDCl_3$, 100 MHz) Spectrum of Compound 64



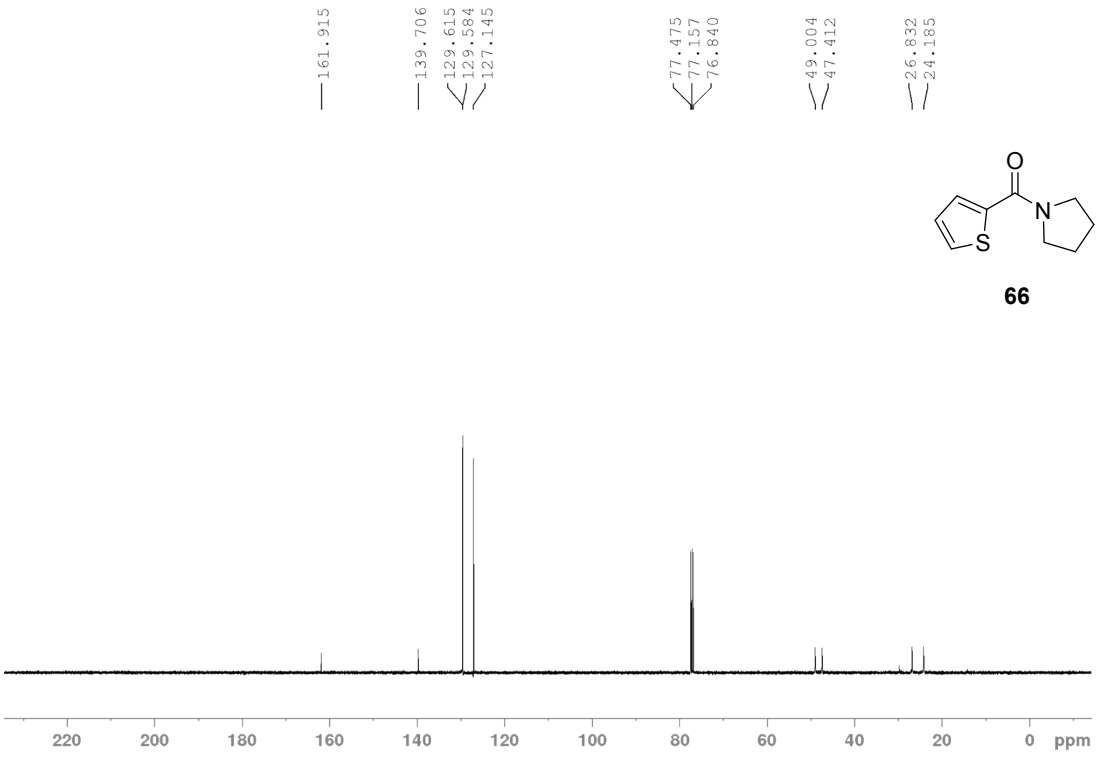
^{1}H NMR ($CDCl_3$, 400 MHz) Spectrum of Compound 65



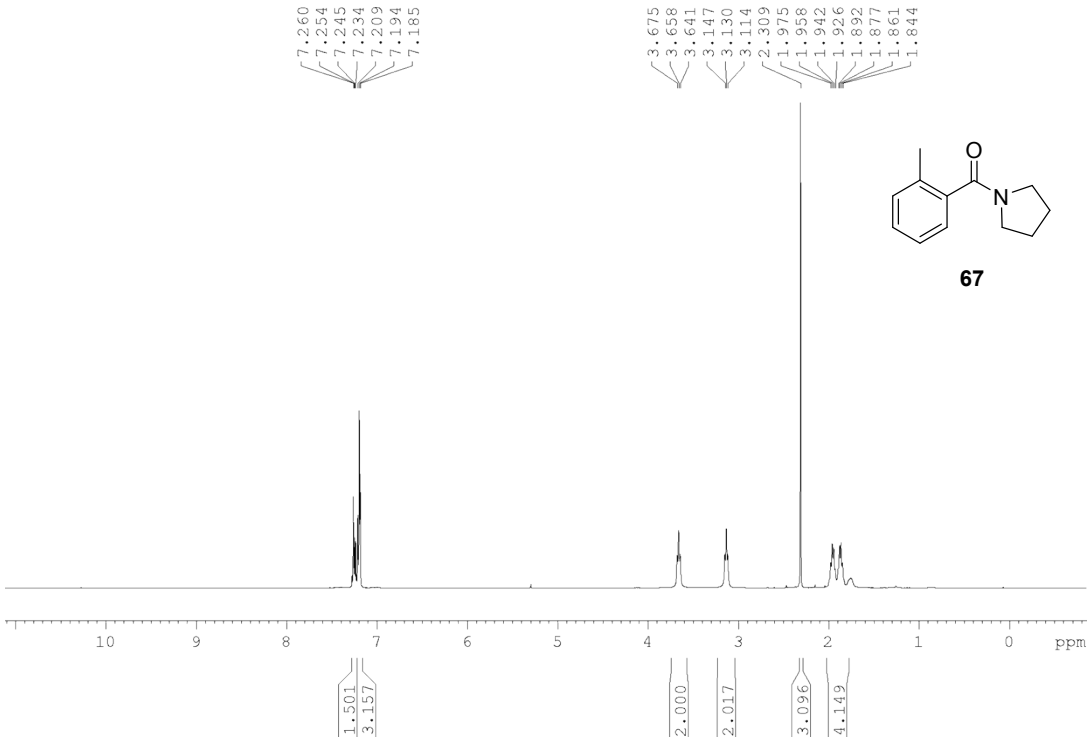
^{13}C NMR ($CDCl_3$, 100 MHz) Spectrum of Compound 65



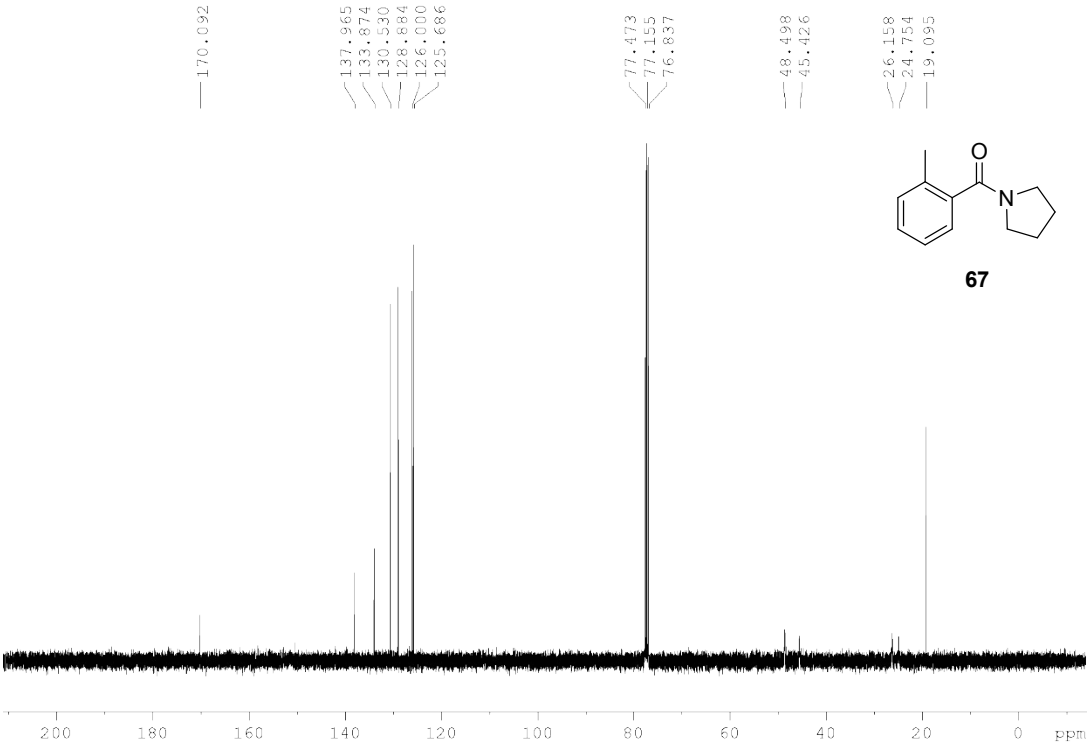
^{1}H NMR ($CDCl_3$, 400 MHz) Spectrum of Compound 66



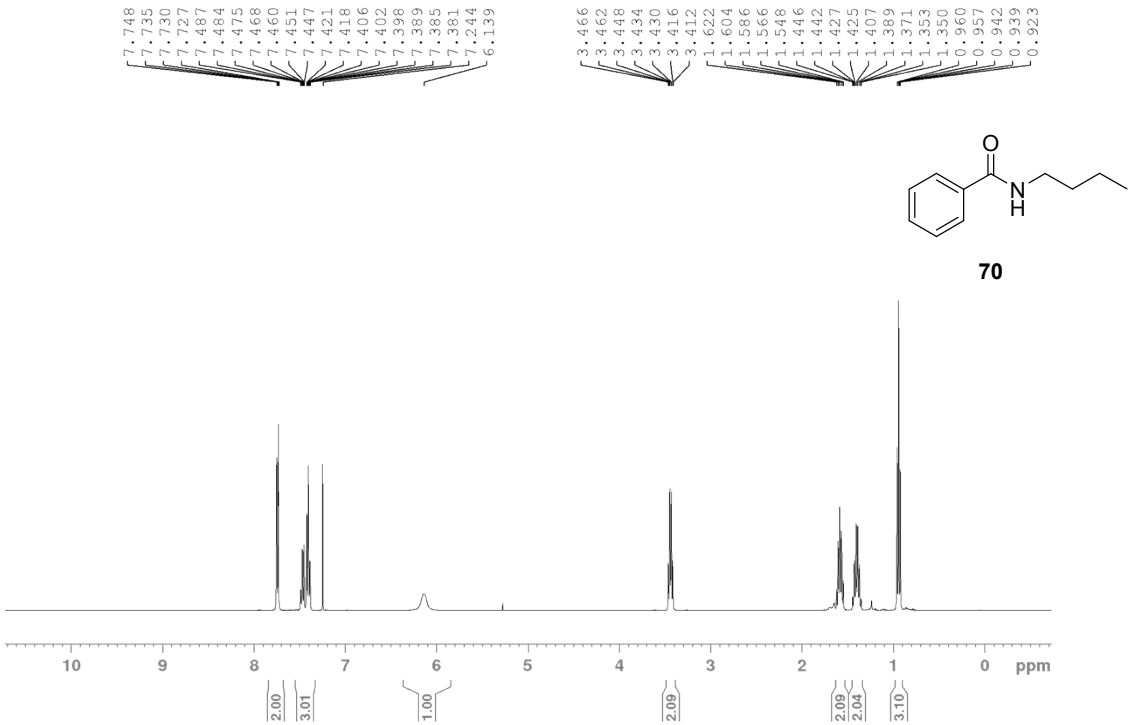
^{13}C NMR ($CDCl_3$, 100 MHz) Spectrum of Compound 66



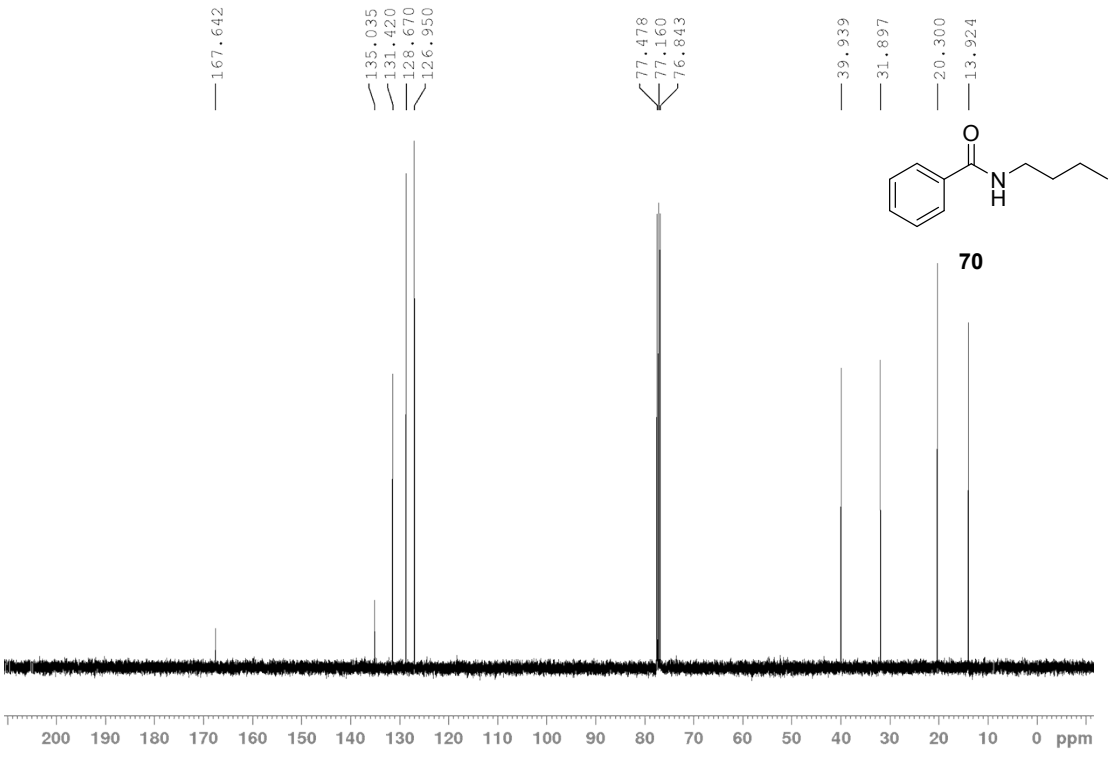
^{1}H NMR ($CDCl_3$, 400 MH) Spectrum of Compound 67



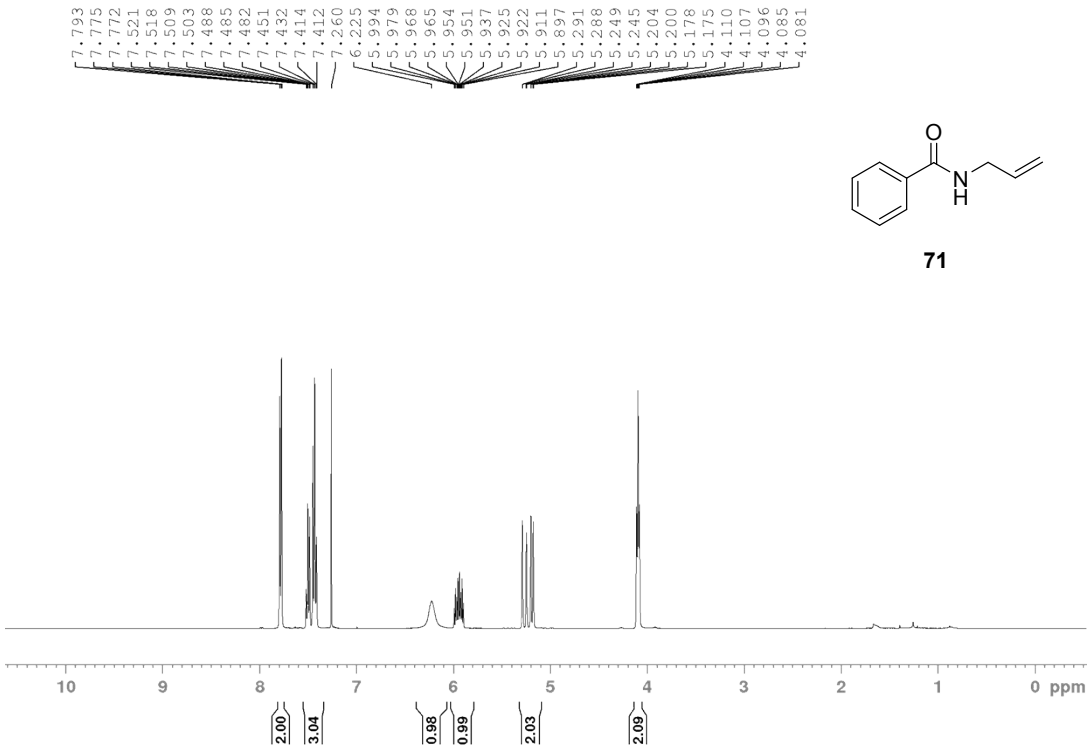
^{13}C NMR ($CDCl_3$, 100 MHz) Spectrum of Compound 67



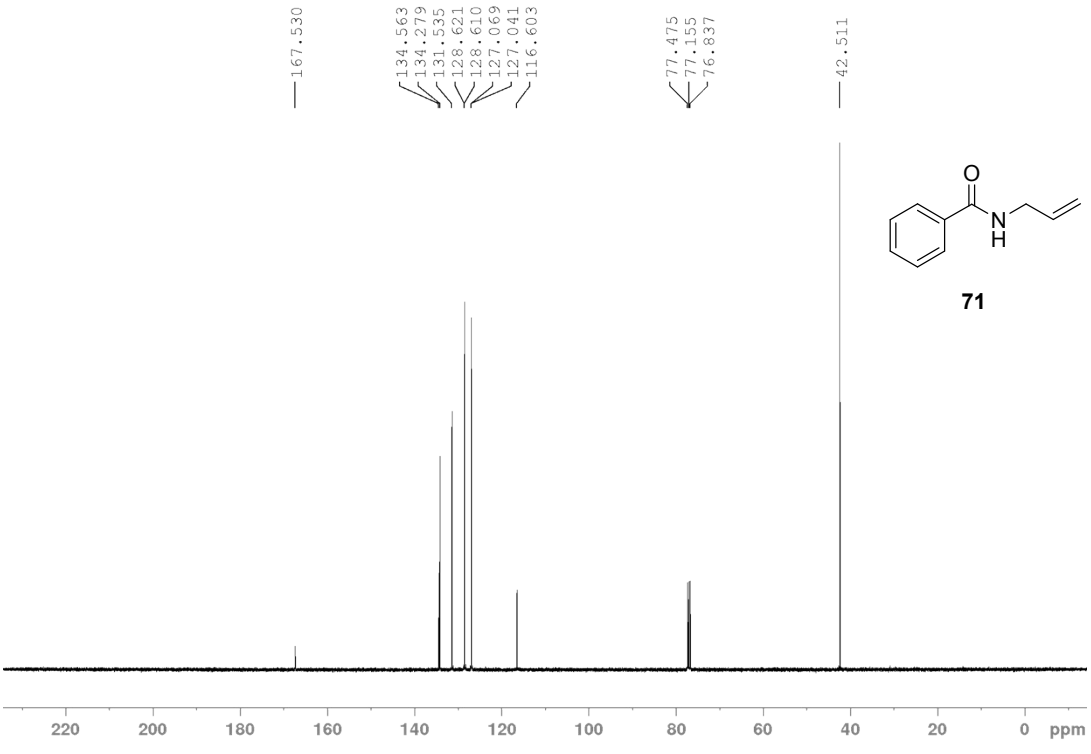
^{1}H NMR ($CDCl_3$, 400 MHz) Spectrum of Compound 70



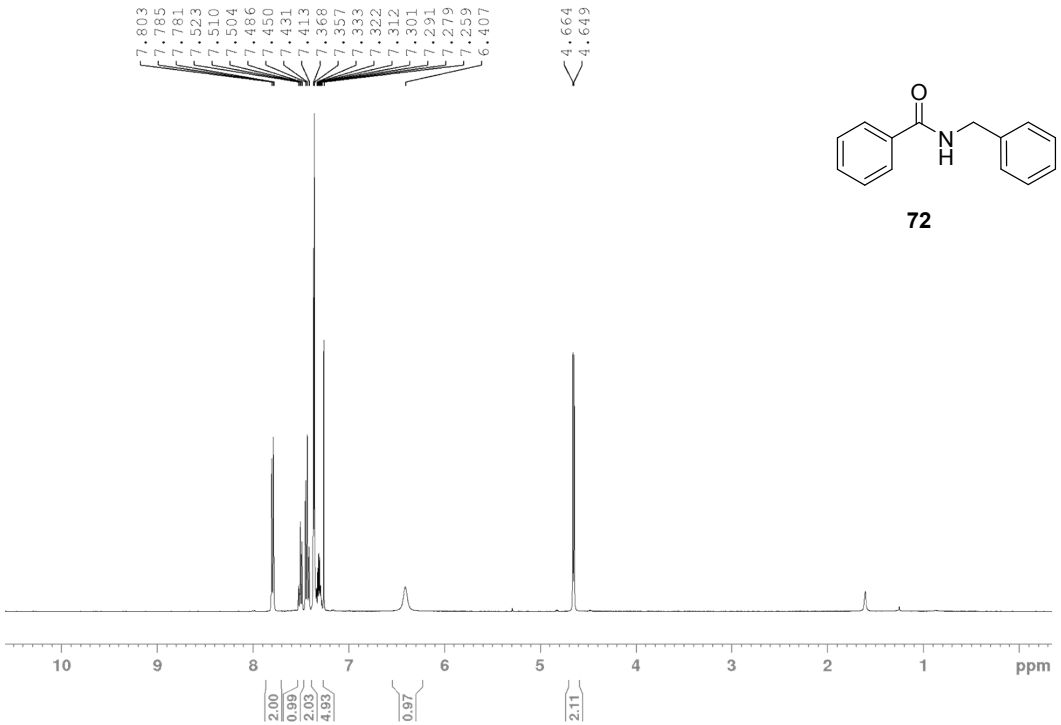
^{13}C NMR ($CDCl_3$, 100 MHz) Spectrum of Compound 70



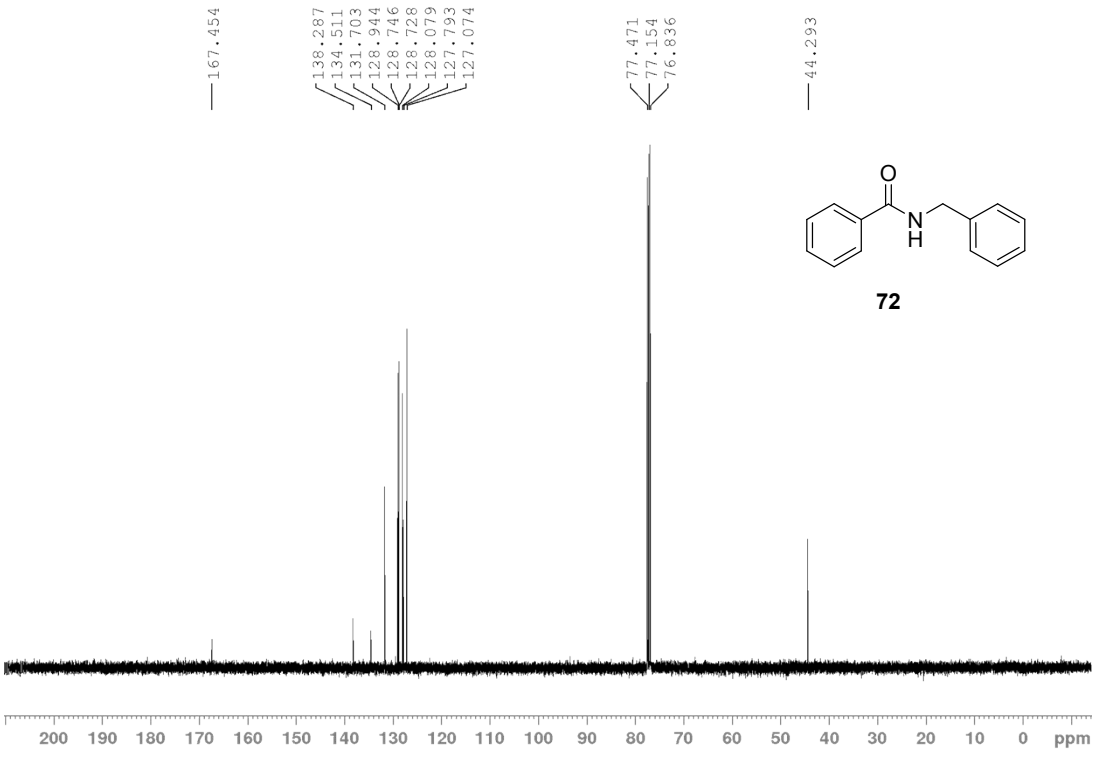
^{1}H NMR ($CDCl_3$, 400 MHz) Spectrum of Compound 71



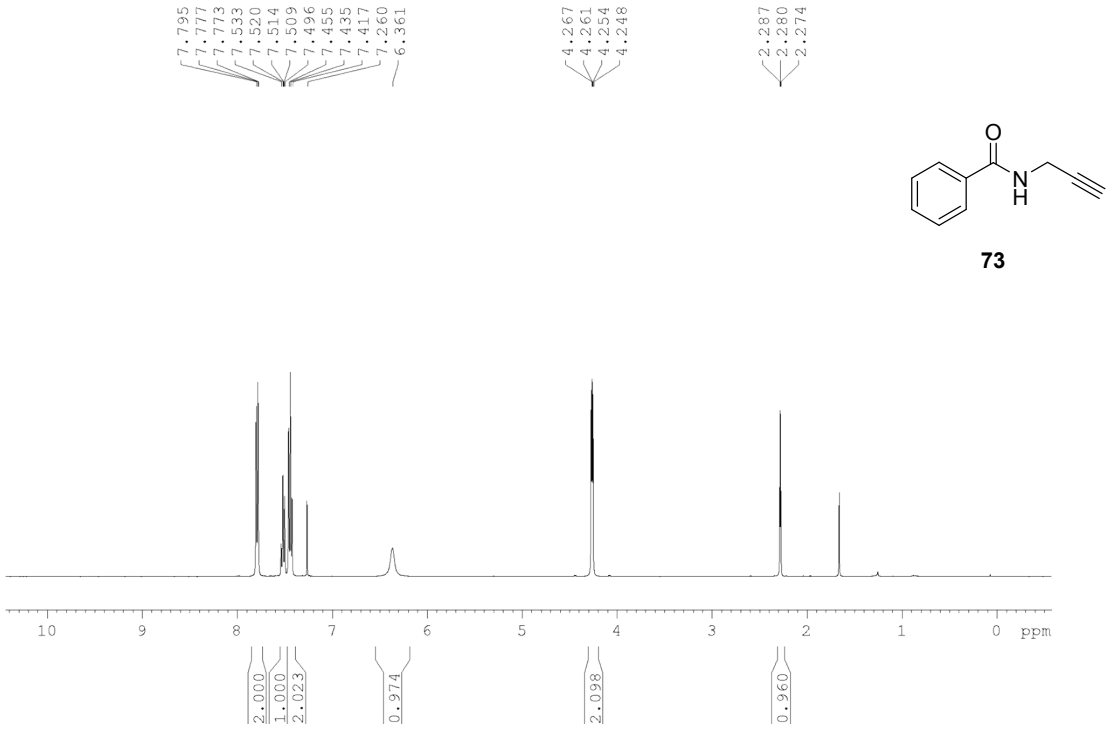
^{13}C NMR ($CDCl_3$, 100 MHz) Spectrum of Compound 71



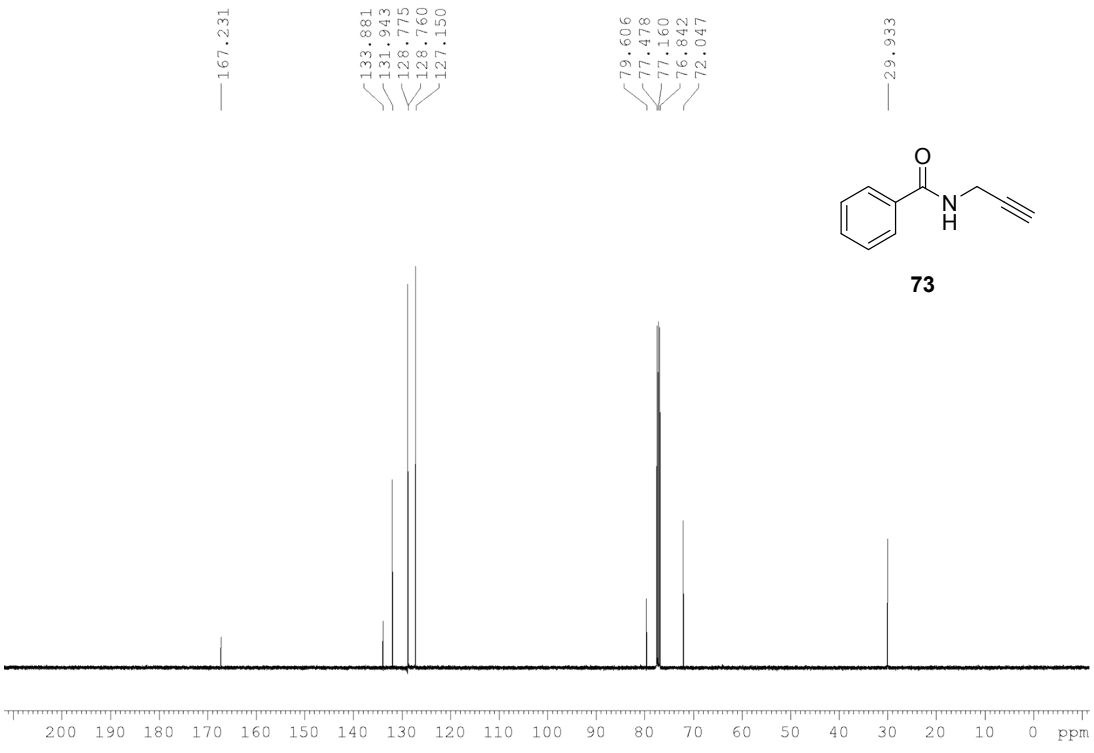
^{1}H NMR ($CDCl_3$, 400 MHz) Spectrum of Compound 72



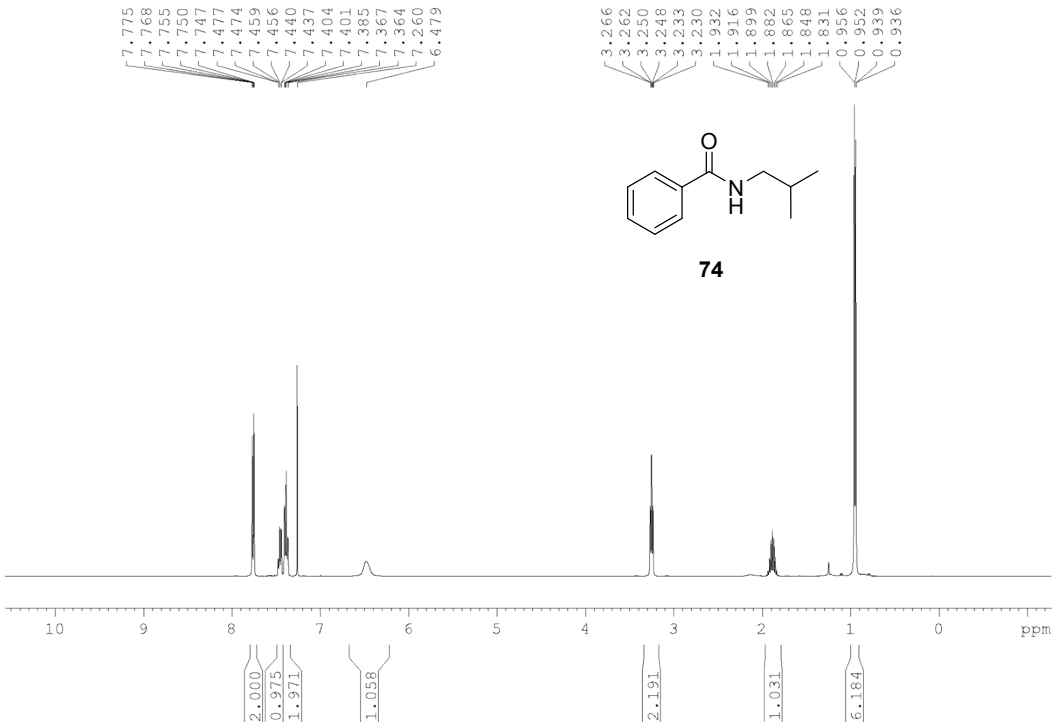
^{13}C NMR ($CDCl_3$, 100 MHz) Spectrum of Compound 72



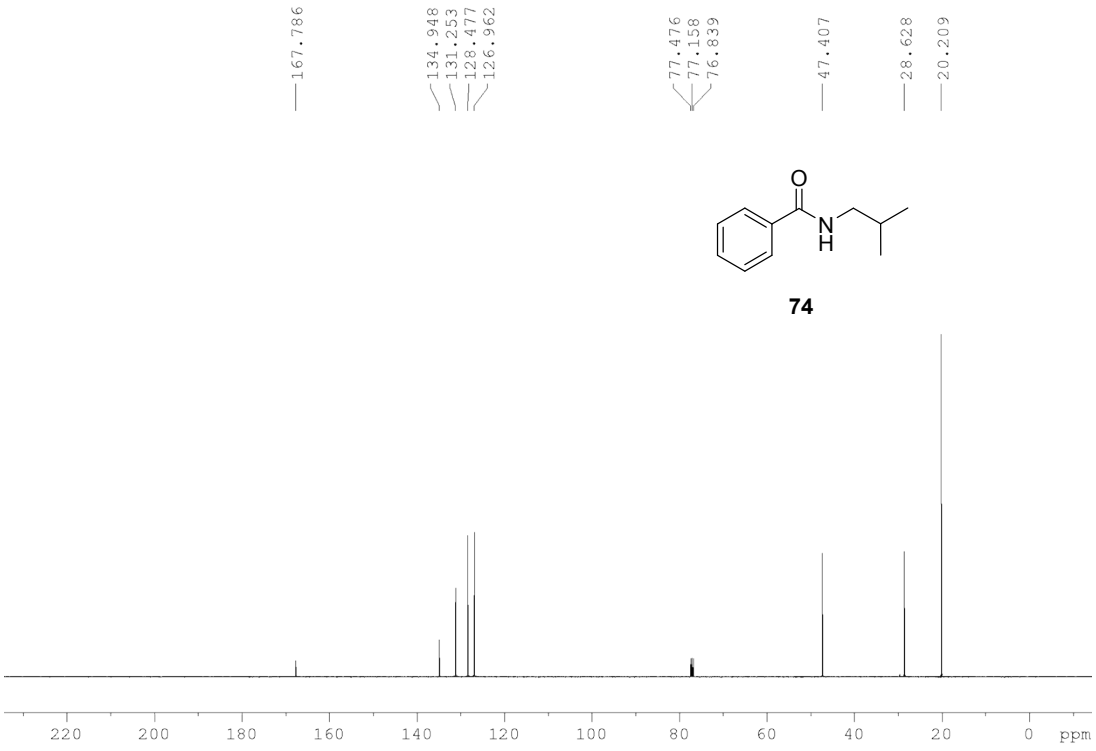
^{1}H NMR ($CDCl_3$, 400 MHz) Spectrum of Compound 73



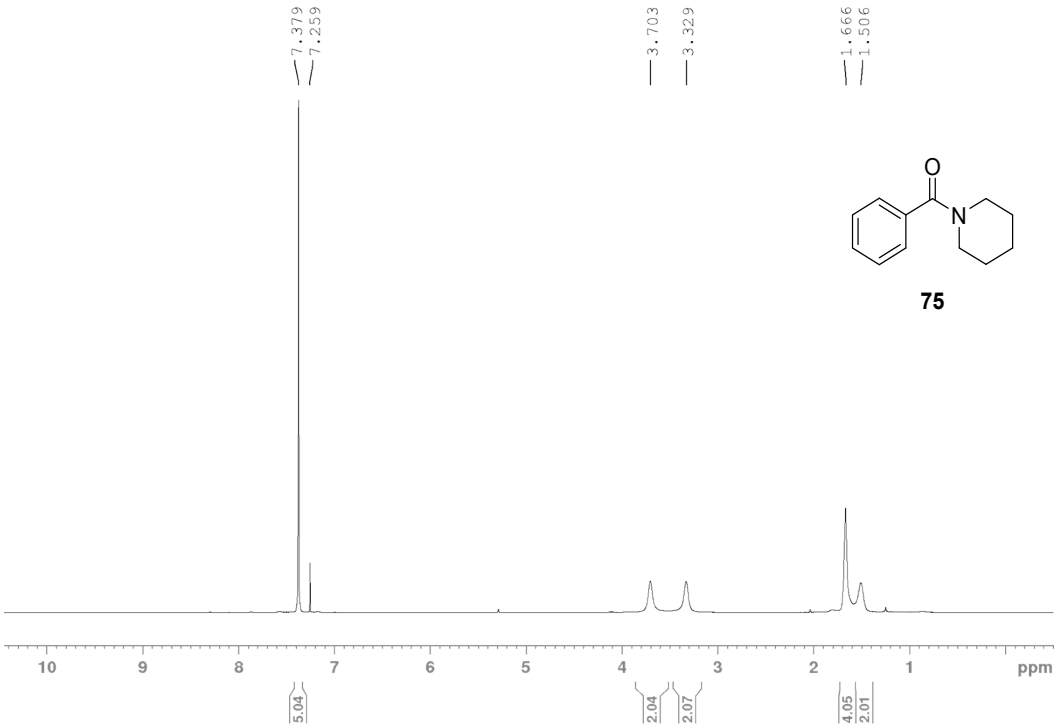
^{13}C NMR ($CDCl_3$, 100 MHz) Spectrum of Compound 73



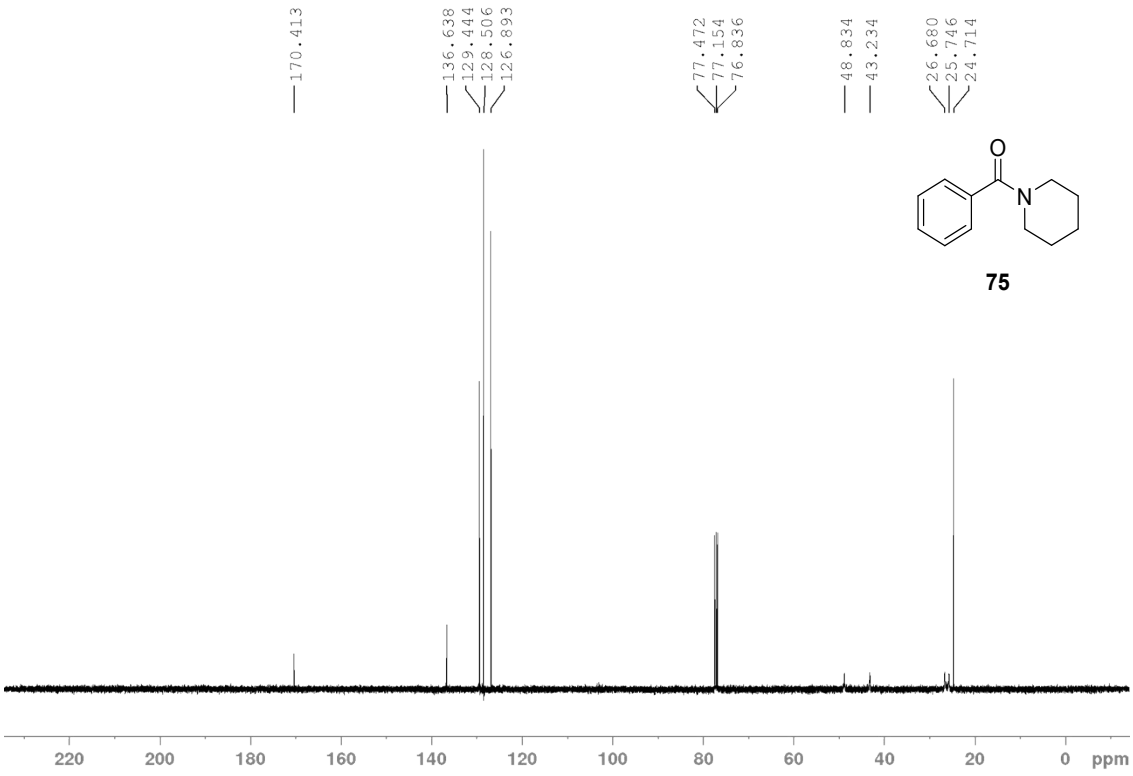
^{1}H NMR ($CDCl_3$, 400 MHz) Spectrum of Compound 74



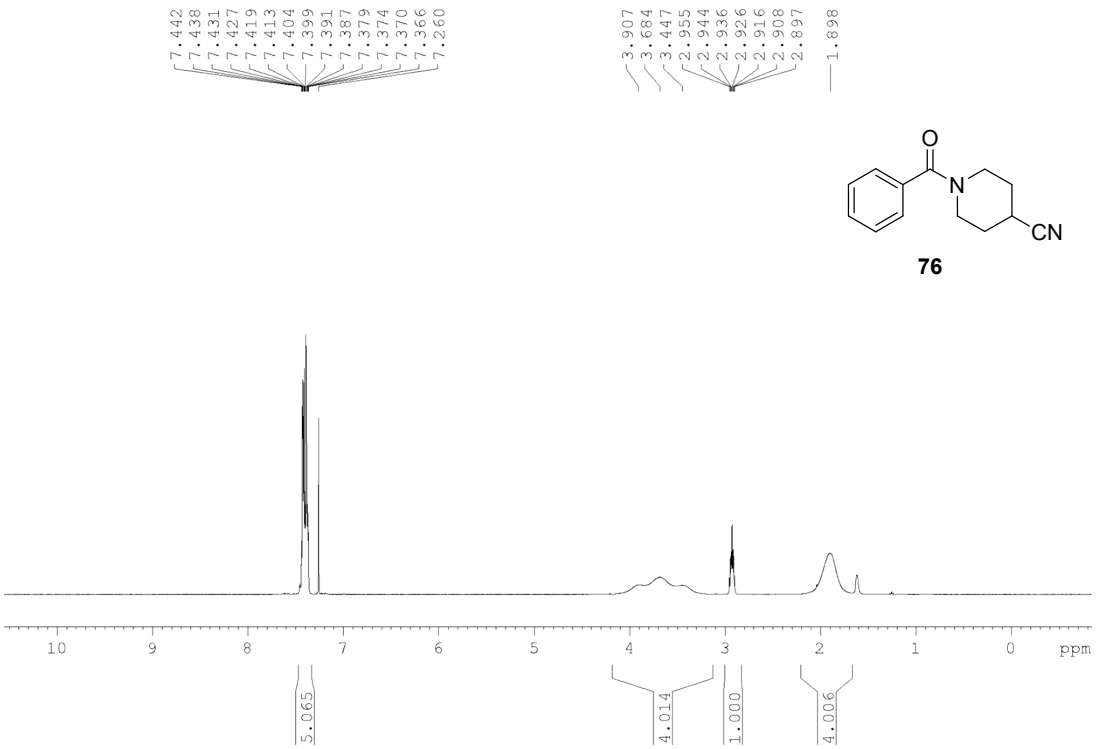
^{13}C NMR ($CDCl_3$, 100 MHz) Spectrum of Compound 74



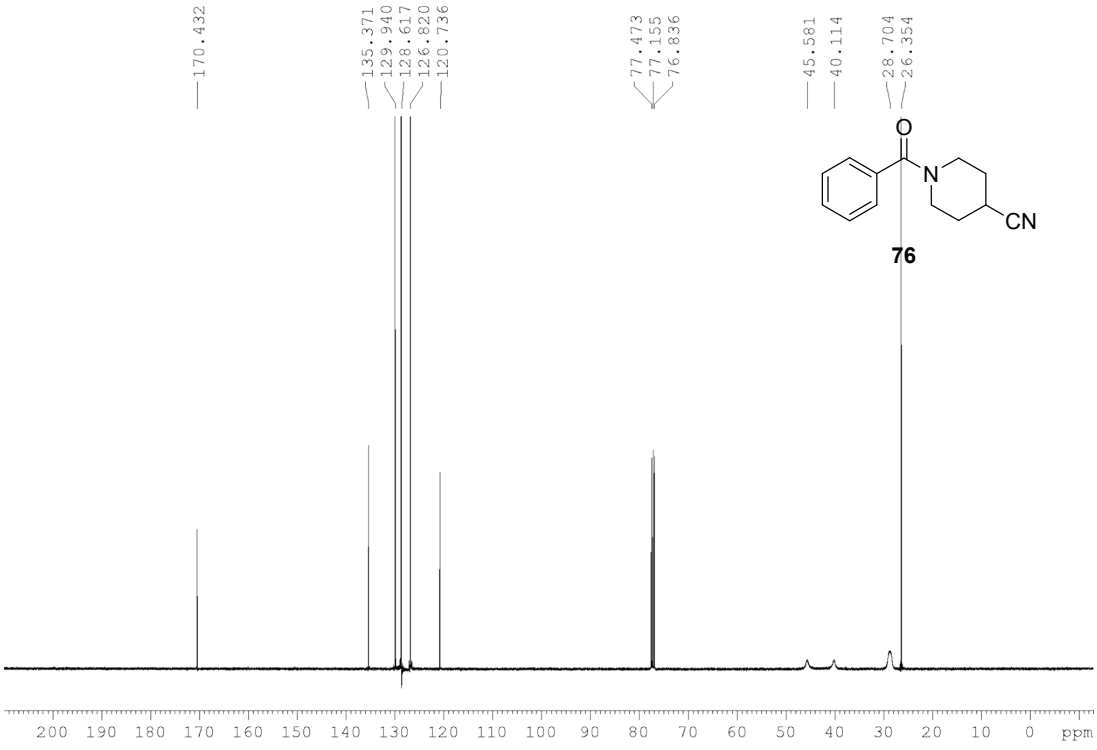
^{1}H NMR ($CDCl_3$, 400 MHz) Spectrum of Compound 75



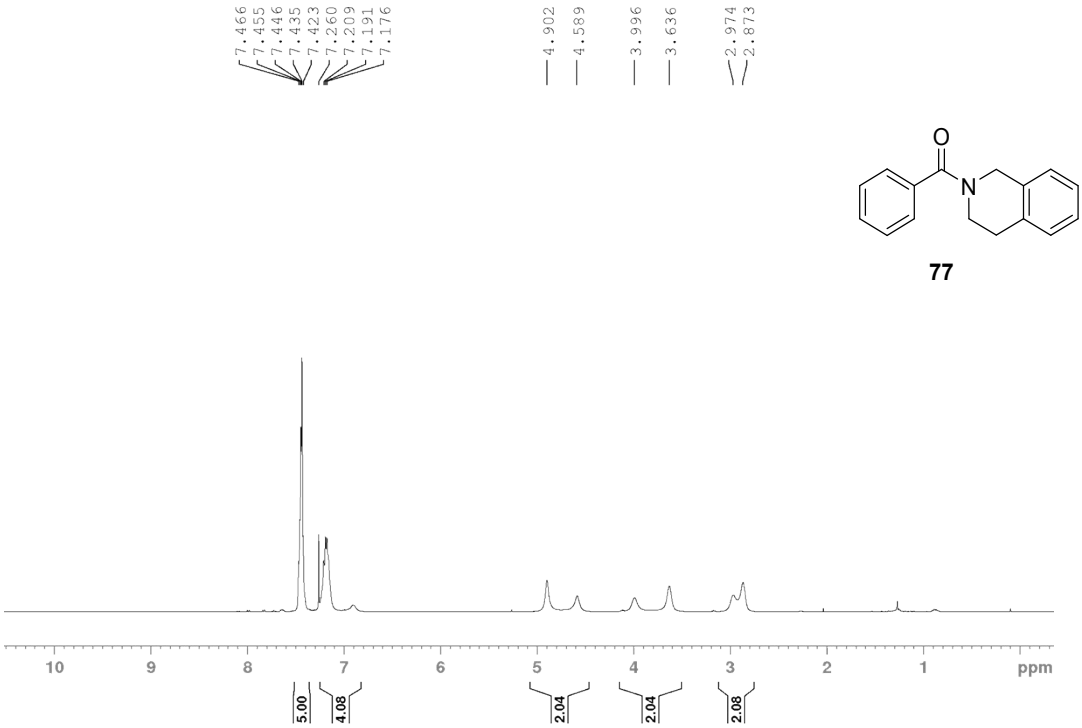
^{13}C NMR ($CDCl_3$, 100 MHz) Spectrum of Compound 75



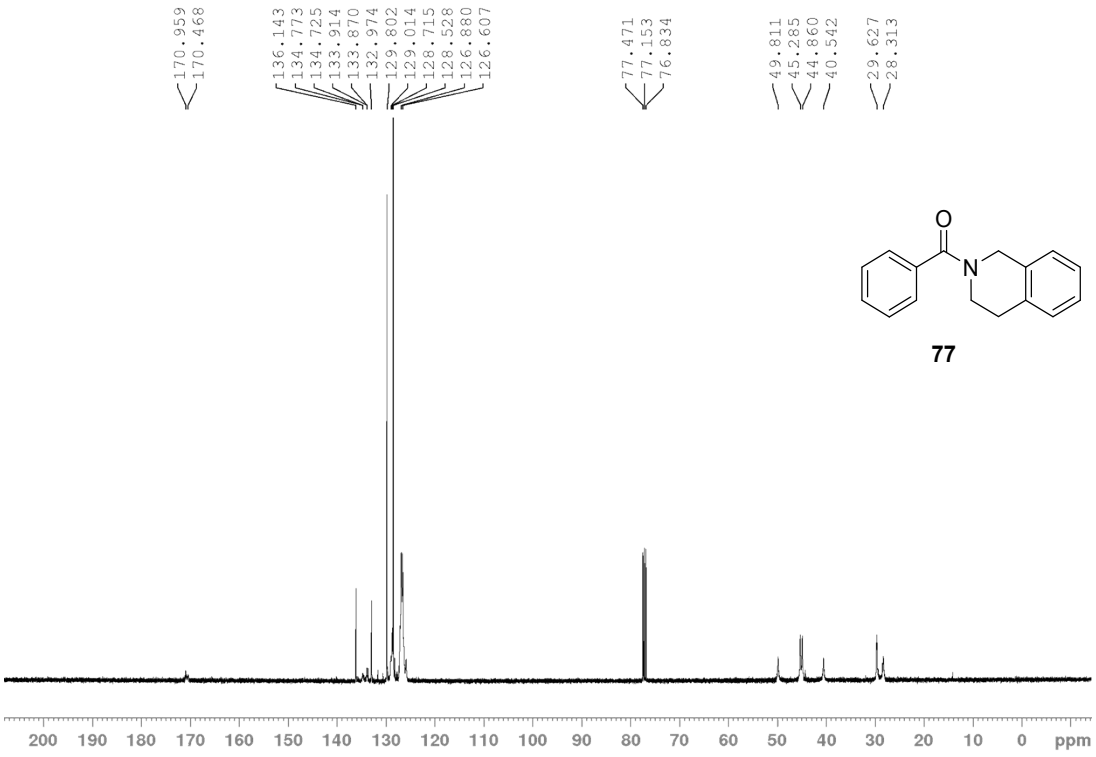
^{1}H NMR ($CDCl_3$, 400 MHz) Spectrum of Compound 76



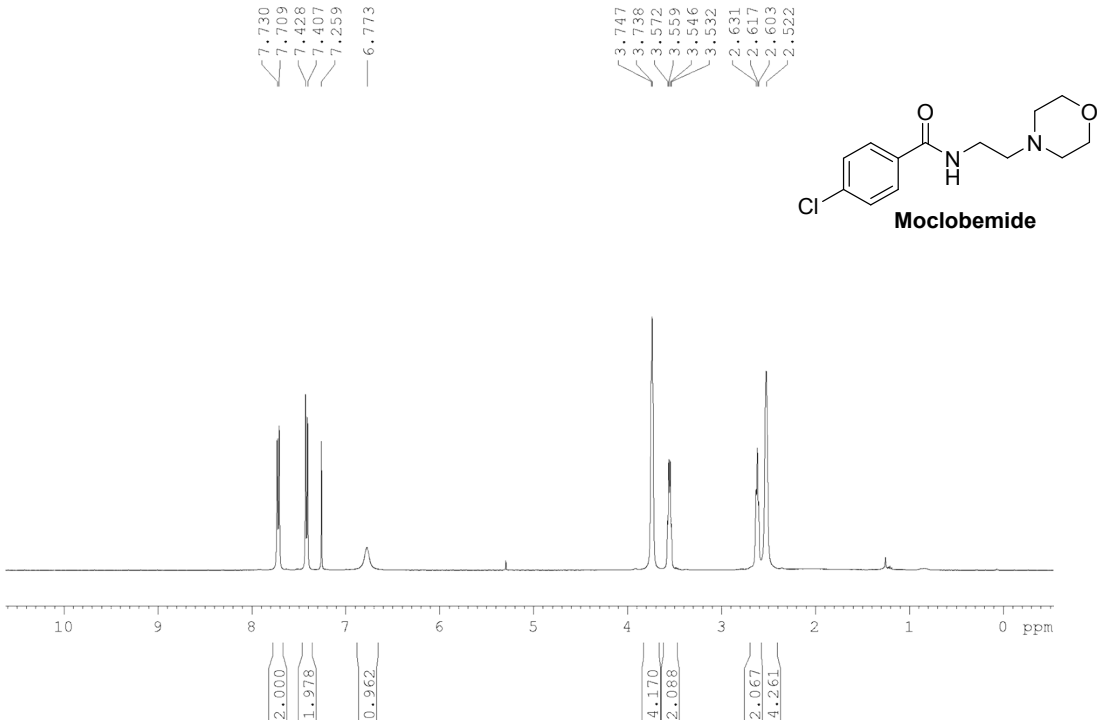
^{13}C NMR ($CDCl_3$, 100 MHz) Spectrum of Compound 76



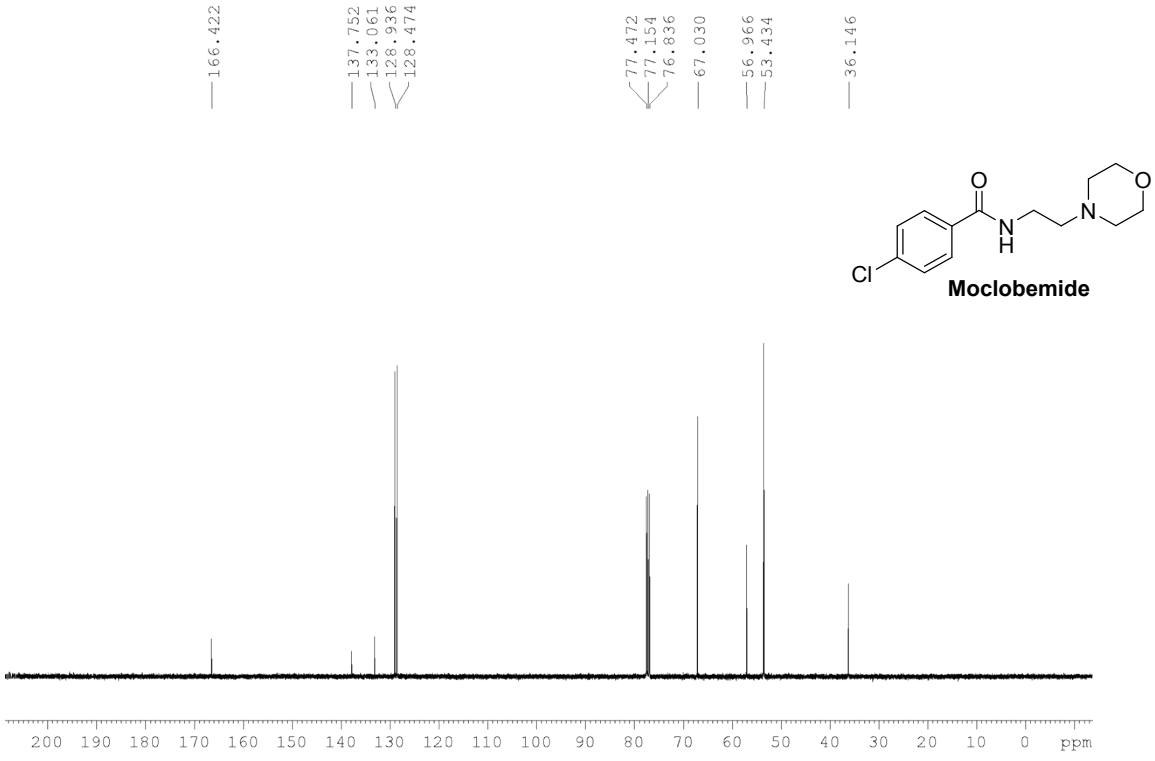
^{1}H NMR ($CDCl_3$, 400 MHz) Spectrum of Compound 77



^{13}C NMR ($CDCl_3$, 100 MHz) Spectrum of Compound 77



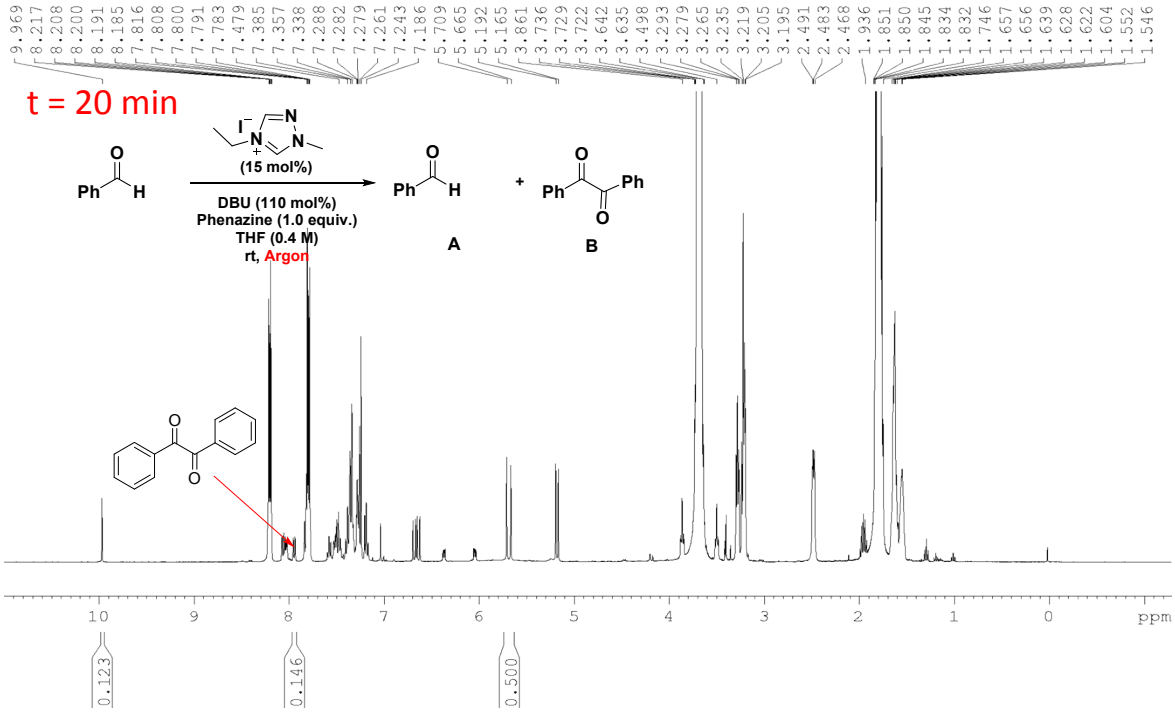
^{1}H NMR ($CDCl_3$, 400 MHz) Spectrum of Moclobemide

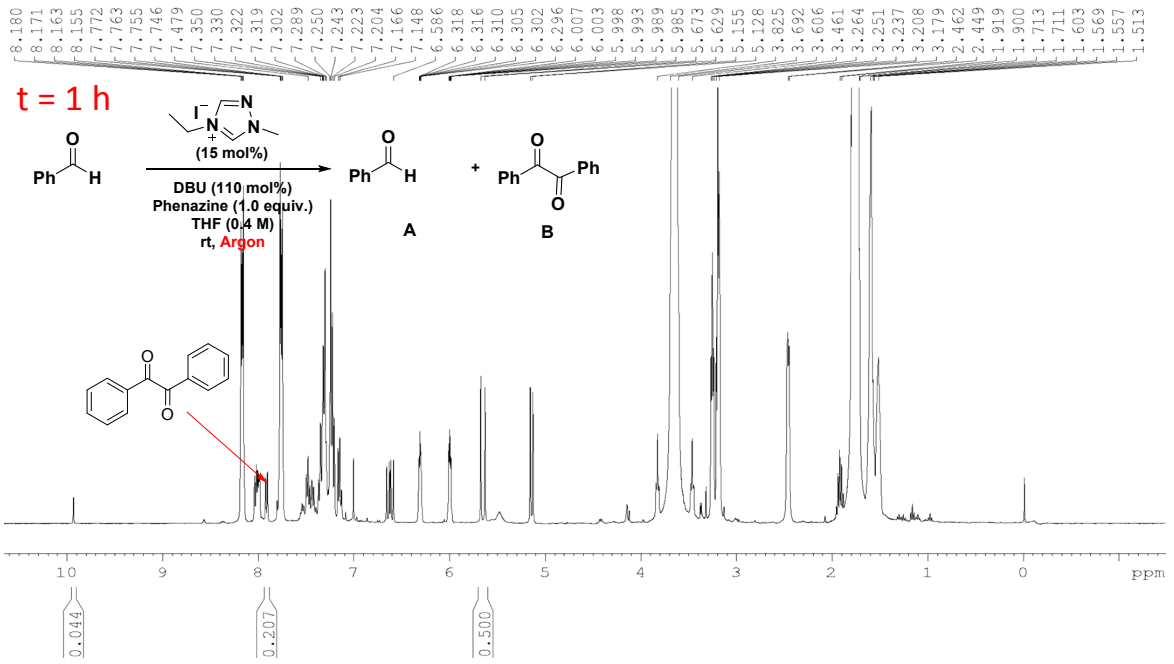


^{13}C NMR ($CDCl_3$, 100 MHz) Spectrum of Moclobemide

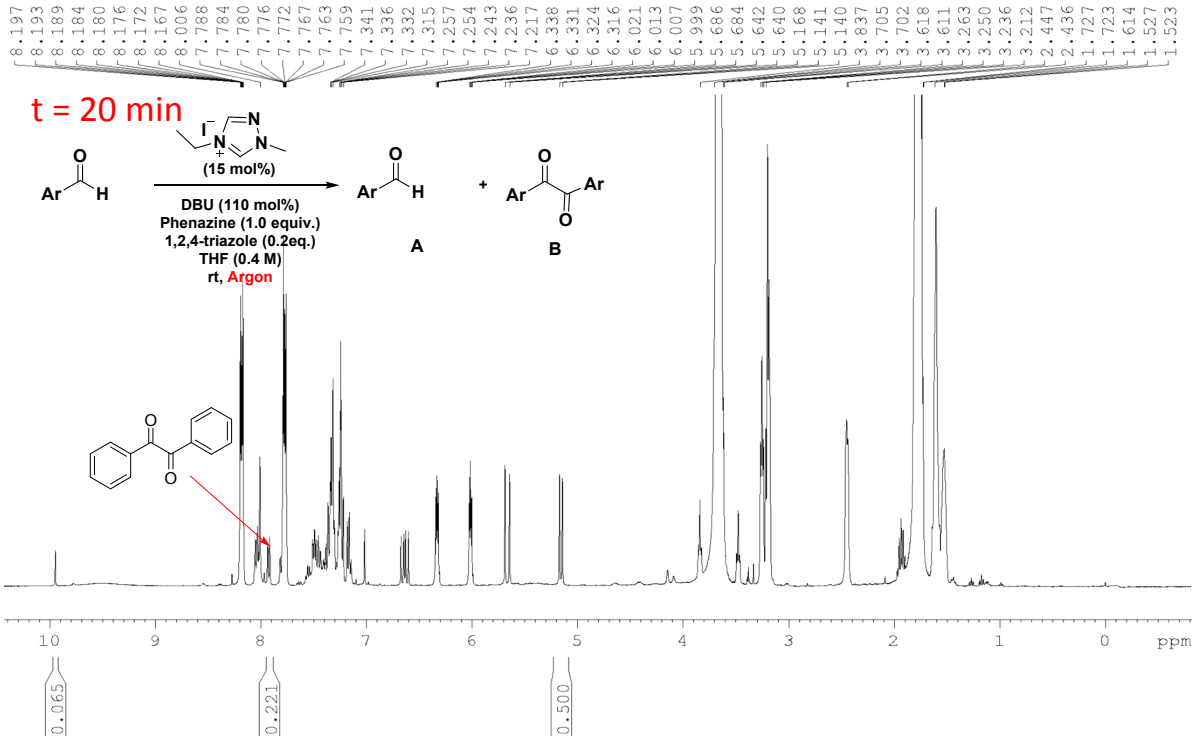
^{1}H NMR spectroscopic evidence for the intermediacy of benzil in the amidation process

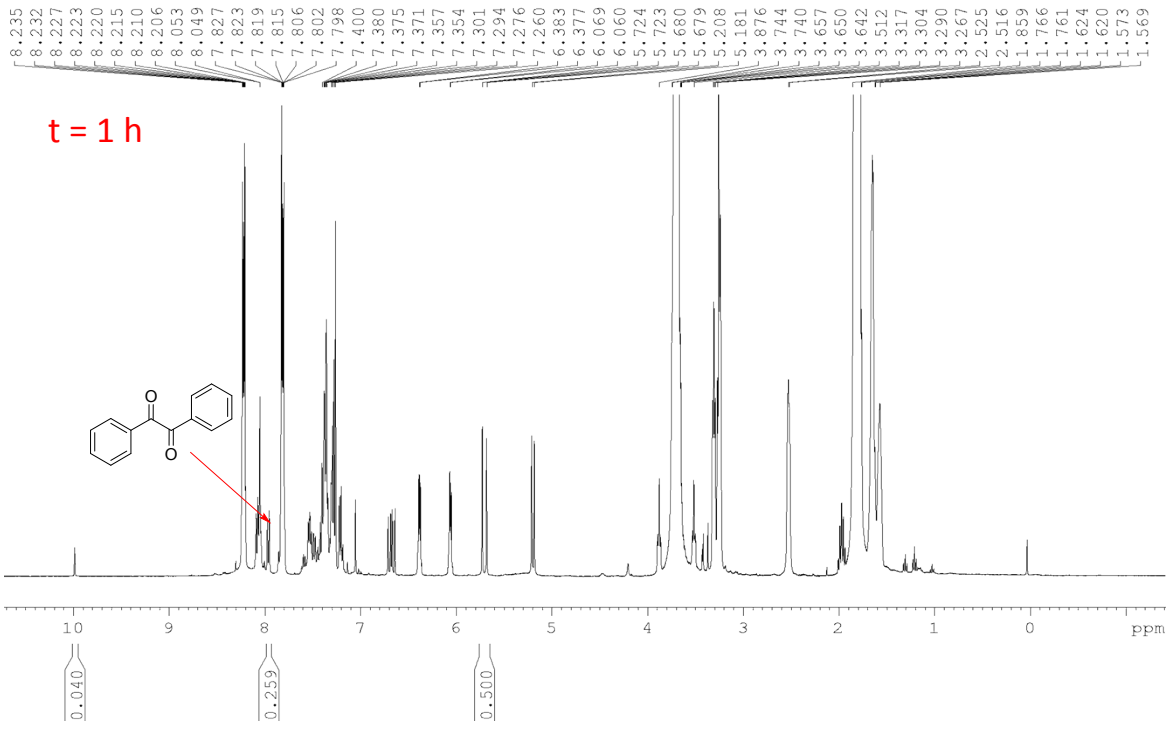
i) In absence of 1,2,4-triazole (**48**)



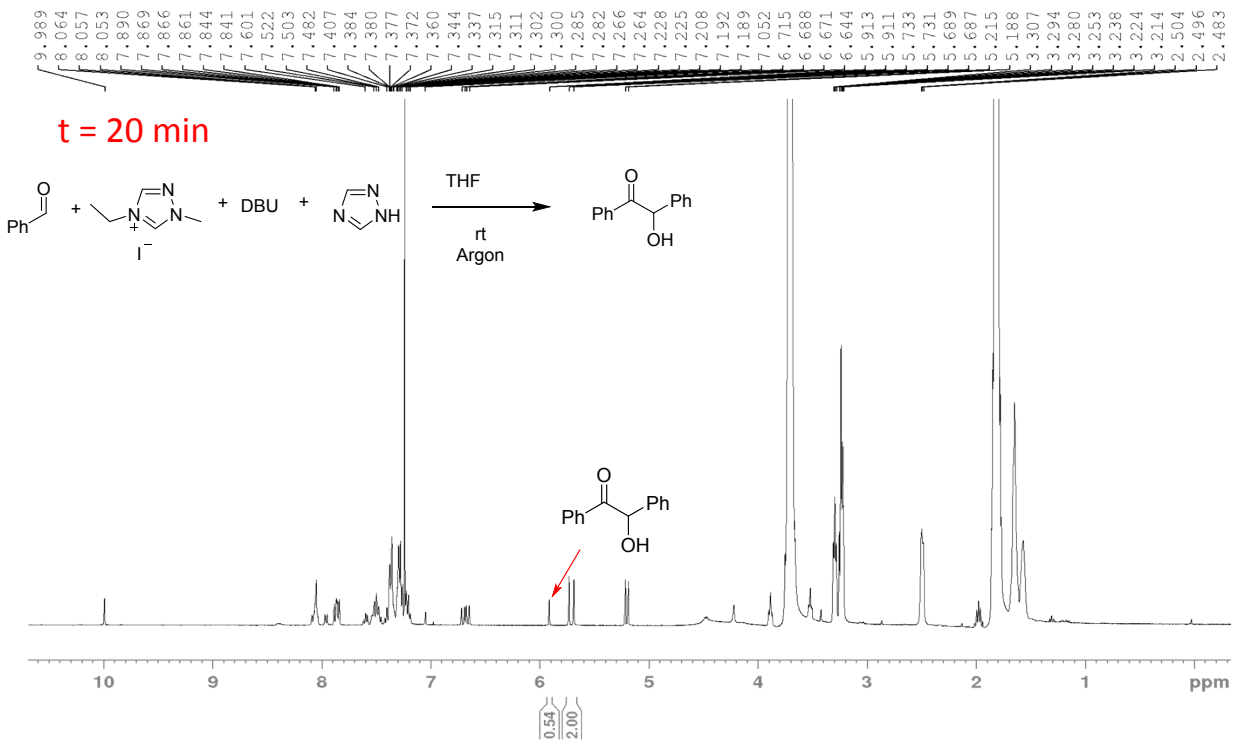


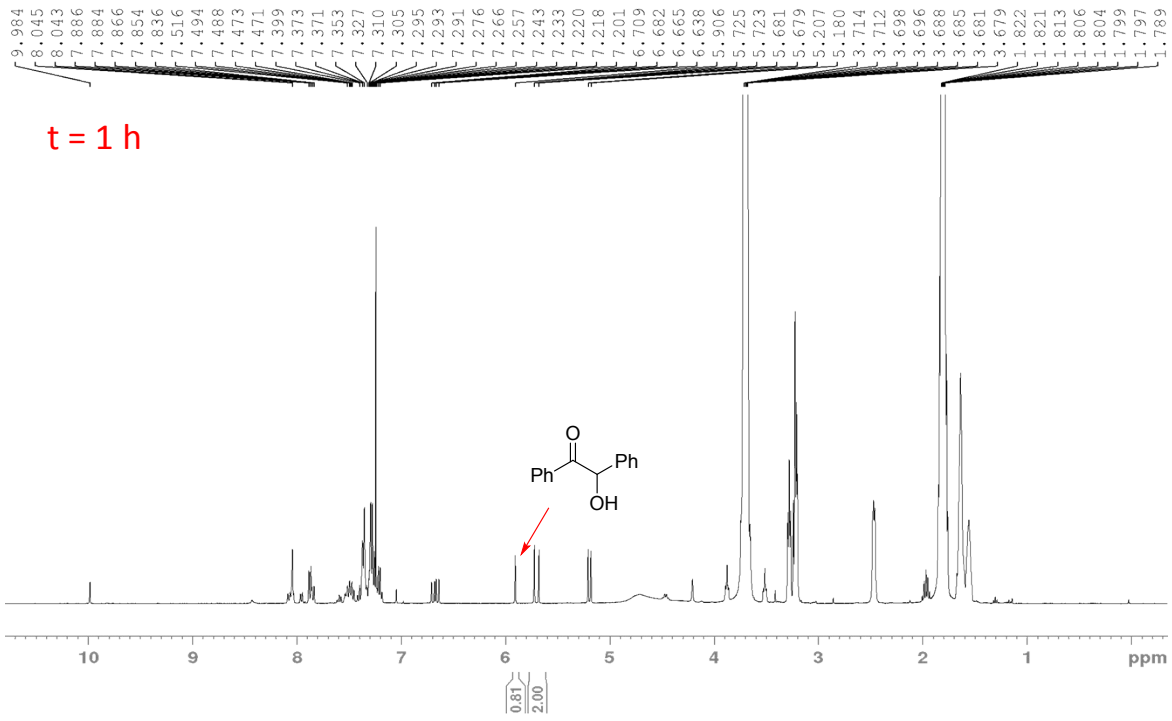
ii) With 1,2,4-triazole (**48**)





iii) Reaction carried out without phenazine under argon





24a is not an efficient acylating agent in the presence of the catalyst