

Supporting Information for

**Selective *N*-Formylation / *N*-Methylation of Amines and
N-Formylation of Amides and Carbamates with Carbon
Dioxide and Hydrosilanes: Promotion of Basic Counter
Anion of the Zinc Catalyst**

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Experimental section

Materials

CO₂ was purchased from SHOWA DENKO GAS PRODUCTS CO. LTD. All of the other chemicals were purchased from Sigma-Aldrich, Tokyo Chemical Industry (TCI), or FUJIFILM Wako Pure Chemical Corporation in the best grade, stored under N₂, and used without further purification.

Caution: High pressure CO₂ gas cylinders should be handled with care and located in an open area with fresh air, although we did not encounter any accident.

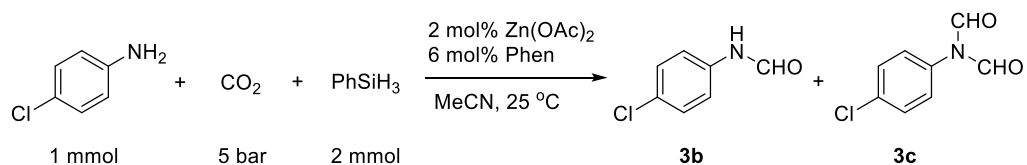
Instruments

The catalytic reactions were carried out in a 10 mL stainless-steel autoclave with a gas-pressure monitor (max. 25 MPa). All of the oxygen-free operations were done in a glovebox. If required, reaction mixtures were heated in Sibata Chemi-300 Synthesizer. ¹H, ¹³C {¹H} and ²⁹Si {¹H} NMR spectra were recorded with a 400 MHz Bruker NMR Spectrometer at room temperature. Products were isolated with a Yamazen AI-580 Single Channel Automated Flash Chromatography System by using *n*-hexane and ethyl acetate as eluents. Molecular weights were determined with a Shimadzu GCMS-QP2010 Plus Gas Chromatograph Mass Spectrometer (GC–MS) with electron ionization mode.

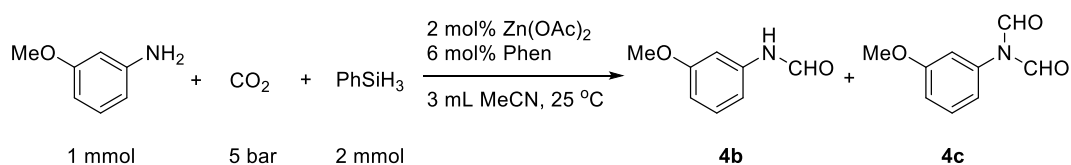
General procedure for the synthesis of 1a

To a 10 mL steel autoclave, Zn(OAc)₂ (3.7 mg, 0.02 mmol), phen (10.8 mg, 0.06 mmol), MeCN (3 mL), *N*-methylaniline (10.7 mg, 1 mmol), and PhSiH₃ (108 mg, 1 mmol) were added in a glove box. The autoclave was sealed tightly and filled with CO₂ to 5 bar at room temperature. After 4 h reaction in a 25°C, CO₂ was released slowly. The solvent was removed in vacuo and re-dissolved in 30 mL dichloromethane washed with water (30 mL, 3 times). Isolated product was purified with automated flash chromatography with *n*-hexane and ethyl acetate as the eluents. A colorless liquid (124 mg, 92%) was obtained after dried in vacuo. The product was characterized with ¹H NMR, ¹³C {¹H} NMR, and GC–MS, consistent with authentic chemical. Other products were synthesized in the same way, and consistent with either authentic materials or literatures.

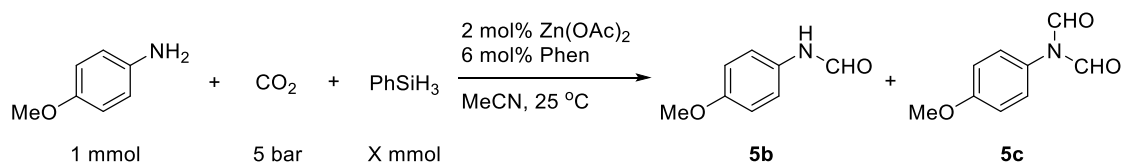
Reaction condition screening of the synthesis of mono-*N*-formylation products.



1 h	30%	N.D.
2 h	48%	4%
3 h	68%	7%
4 h	48%	9%



1 h	58%	N.D.
2 h	52%	8%
3 h	45%	9%
4 h	44%	10%

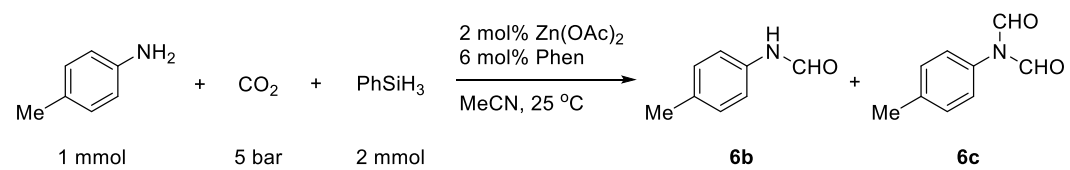


X = 1.5

1 h	42%	N.D.
2 h	54%	trace
3 h	57%	5%
4 h	65%	30%

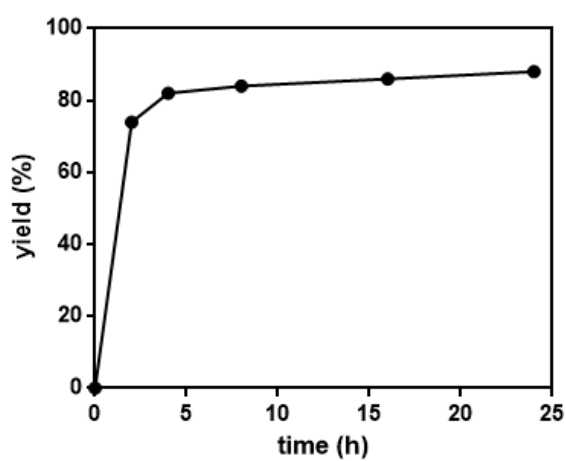
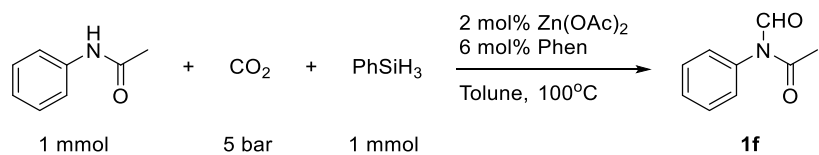
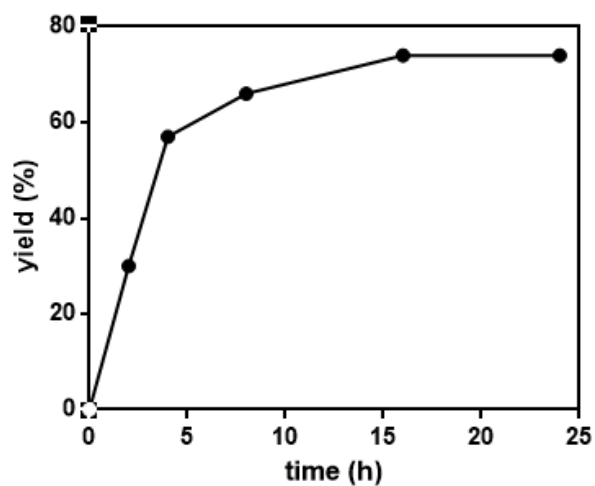
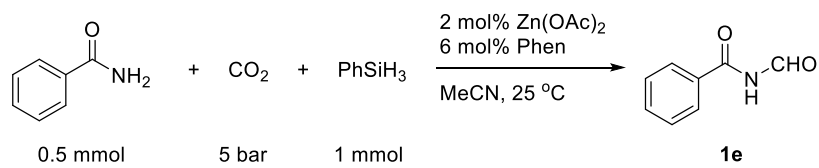
X = 2

1 h	54%	4%
2 h	66%	8%
3 h	42%	24%
4 h	40%	39%

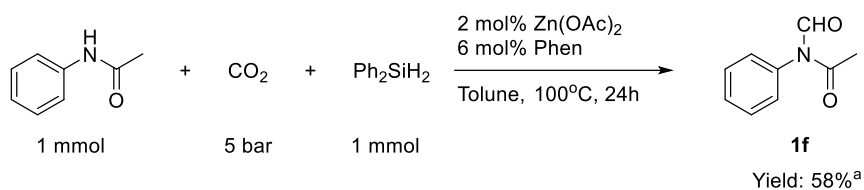


1 h	52%	3%
2 h	64%	15%
3 h	58%	16%
4 h	24%	49%

Time dependent reactions for the *N*-formylation of amides.

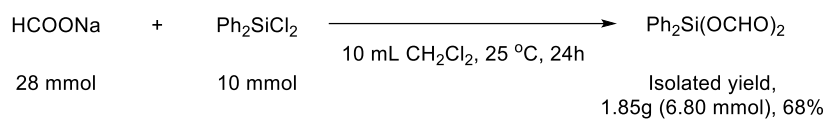


***N*-Formylation of acetanilide with CO₂ and Ph₂SiH₂**



^a The yield was determined by ¹H NMR in CDCl₃ using mesitylene as the internal standard.

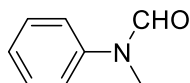
Synthesis of Ph₂Si(OCHO)₂^{S1}



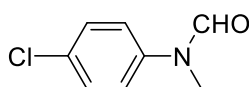
NMR / MS data and NMR spectra

N-Formylation of secondary amines

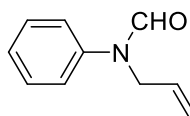
(Products **1a-13a** contain conformational isomers.)^{S2-S5}



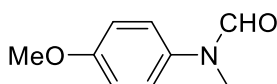
Product **1a**. ^1H NMR (CDCl_3 , 400 MHz) δ 8.47 (s, 1H), 7.41 (t, $J = 7.5$ Hz, 2H), 7.26 (t, $J = 7.5$ Hz, 1H), 7.16 (d, $J = 8.0$ Hz, 2H), 3.31 (s, 3H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 162.4, 142.2, 129.6, 126.4, 122.4, 32.1. GC-MS (m/z): 135 [M^+] for $\text{C}_8\text{H}_9\text{NO}$ (yield, 124 mg (0.92 mmol), 92%).



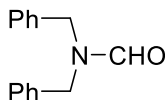
Product **2a**. ^1H NMR (CDCl_3 , 400 MHz): δ 8.41, 8.31 (s, total 1H), 7.40-7.29 (m, 2H), 7.12-7.07 (m, 2H), 3.30, 3.26 (s, total 3H). ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 162.0, 140.8, 132.0, 129.8, 123.5, 32.0. GC-MS (m/z): 169 [M^+] for $\text{C}_8\text{H}_8\text{ClNO}$ (yield, 125 mg (0.74 mmol), 74%).



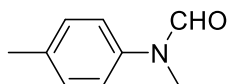
Product **3a**. ^1H NMR (CDCl_3 , 400 MHz) δ 8.51, 8.40 (s, total 1H), 7.44-7.37 (m, 2H), 7.33-7.28 (m, 1H), 7.23-7.20 (m, 2H), 5.92-5.82 (m, 1H), 5.25-5.18 (m, 2H), 4.44, 4.31 (d, $J = 5.8$ Hz, total 2H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 162.0, 141.2, 132.5, 129.6, 126.7, 123.5, 117.6, 47.9; GC-MS (m/z): 161 [M^+] for $\text{C}_{10}\text{H}_{11}\text{NO}$ (yield, 128 mg (0.80 mmol), 80%).



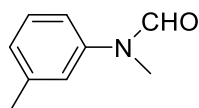
Product **4a**. ^1H NMR (CDCl_3 , 400 MHz) δ 8.25 (s, 1H), 7.01 (d, $J = 9.1$ Hz, 2H), 6.84 (d, $J = 9.1$ Hz, 2H), 3.72 (s, 3H), 3.17 (s, 3H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 162.4, 158.2, 135.2, 124.5, 114.7, 55.5, 32.6. GC-MS (m/z): 165 [M^+] for $\text{C}_9\text{H}_{11}\text{NO}_2$ (yield, 140 mg (0.85 mmol), 85%).



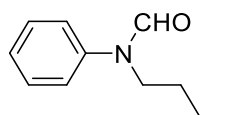
Product **5a**. ^1H NMR (CDCl_3 , 400 MHz) δ 8.42 (s, 1H), 7.40-7.18 (m, 10H), 4.42 (s, 2H), 4.26 (s, 2H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 162.9, 136.0, 135.6, 128.9, 128.7, 128.5, 128.2, 127.7, 127.6, 50.3, 44.7. GC-MS (m/z): 225 [M^+] for $\text{C}_{15}\text{H}_{15}\text{NO}$ (yield, 164 mg (0.73 mmol), 73%).



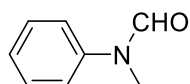
Product **6a**. ^1H NMR (CDCl_3 , 400 MHz) δ 8.41 (s, 1H), 7.20 (d, J = 8.2 Hz, 2H), 7.05 (d, J = 8.4 Hz, 2H), 3.28 (s, 3H), 2.36 (s, 3H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 162.4, 139.7, 136.4, 130.2, 122.6, 32.3, 20.9. GC-MS (m/z): 149 [M^+] for $\text{C}_9\text{H}_{11}\text{NO}$ (yield, 143 mg (0.96 mmol), 96%).



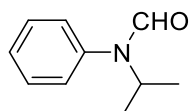
Product **7a**. ^1H NMR (CDCl_3 , 400 MHz) δ 8.43 (s, 1H), 7.30-7.22 (m, 1H), 7.09-7.03 (m, 1H), 6.97-6.92 (m, 2H), 3.28 (s, 3H), 2.36 (s, 3H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 162.3, 142.2, 139.7, 129.4, 127.2, 123.1, 119.4, 32.0, 21.4. GC-MS (m/z): 149 [M^+] for $\text{C}_9\text{H}_{11}\text{NO}$ (yield, 141 mg (0.95 mmol) 95%).



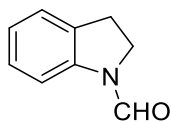
Product **8a**. ^1H NMR (CDCl_3 , 400 MHz) δ 8.37 (s, 1H), 7.44-7.39 (m, 2H), 7.32-7.27 (m, 1H), 7.19-7.15 (m, 2H), 3.82 (t, J = 7.6 Hz, 2H), 1.57-1.48 (m, 2H), 1.37-1.26 (m, 2H), 0.92-0.86 (m, 3H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 162.4, 141.1, 129.6, 126.8, 124.2, 44.8, 29.7, 20.0, 13.7. GC-MS (m/z): 177 [M^+] for $\text{C}_{11}\text{H}_{15}\text{NO}$ (yield, 141 mg (0.80 mmol), 80%).



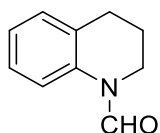
Product **9a**. ^1H NMR (CDCl_3 , 400 MHz) δ 8.35 (s, 1H), 7.43-7.38 (m, 2H), 7.32-7.26 (m, 1H), 7.18-7.15 (m, 2H), 3.86 (q, J = 7.2 Hz, 2H), 1.6 (t, J = 7.2 Hz, 3H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 162.1, 140.8, 129.7, 126.9, 124.3, 40.1, 13.1. GC-MS (m/z): 149 [M^+] for $\text{C}_9\text{H}_{11}\text{NO}$ (yield, 140 mg (0.94 mmol), 94%,).



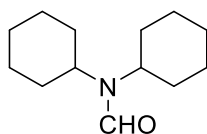
Product **10a**. ^1H NMR (CDCl_3 , 400 MHz) δ 8.37, 8.12 (s, and s, total 1H), 7.41-7.29 (m, 3H), 7.13-7.08 (m, 2H), 4.75, 4.01 (hept, J = 6.8 Hz, and hept, J = 6.8 Hz, total 1H), 1.23, 1.15 (d, J = 6.8 Hz, and d, J = 6.8 Hz, total 6H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 162.5, 138.4, 129.2, 128.9, 128.1, 45.8, 20.9. GC-MS (m/z): 163 [M^+] for $\text{C}_{10}\text{H}_{13}\text{NO}$ (yield, 107 mg (0.66 mmol), 66%).



Product **11a**. ^1H NMR (CDCl_3 , 400 MHz) δ 8.88 (s, 1H), 7.24-7.09 (m, 3H), 7.05-6.99 (m, 1H), 4.10-3.97 (m, 2H), 3.17-3.05 (m, 2H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 157.5, 141.0, 131.9, 127.5, 126.0, 124.2, 109.4, 44.6, 27.1. GC-MS (m/z): 147 [M^+] for $\text{C}_9\text{H}_9\text{NO}$ (yield, 107 mg (0.73 mmol), 73%).



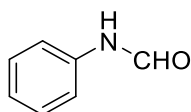
Product **12a**. ^1H NMR (CDCl_3 , 400 MHz) δ 8.73 (s, 1H), 7.20-6.99 (m, 4H), 3.75, 3.60 (t, J = 6.2 Hz, and t, J = 6.2 Hz, total 2H), 2.76, 2.84 (t, J = 6.4 Hz, and t, J = 6.4 Hz, total 2H), 2.01-1.93, (m, 2H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 161.1, 137.2, 129.6, 128.9, 127.1, 124.6, 117.0, 40.3, 27.1, 22.3. GC-MS (m/z): 161 [M^+] for $\text{C}_{10}\text{H}_{11}\text{NO}$ (yield, 140 mg (0.87 mmol), 87%).



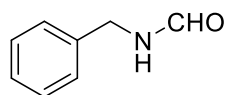
Product **13a**. ^1H NMR (CDCl_3 , 400 MHz): δ 8.21 (s, 1H), 3.99-3.89 (m, 1H), 3.11-3.00 (m, 1H), 1.91-1.05 (m, 20H). ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 161.7, 55.1, 52.6, 34.6, 30.4, 26.3, 25.9, 25.4, 25.3. GC-MS (m/z): 209 [M^+] for $\text{C}_{13}\text{H}_{23}\text{NO}$ (yield, 196 mg (0.94 mmol), 94%).

Mono-*N*-formylation of primary amines

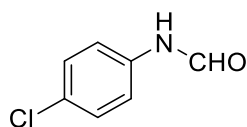
(Products **1b-6b** contains conformational isomers.)^{S3,S6,S7}



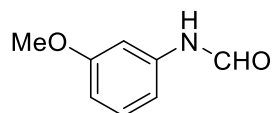
Product **1b**. ^1H NMR (CDCl_3 , 400 MHz) δ 9.35, 8.74 (br and br, total 1H), 8.71, 8.31 (d, J = 11.6, and d, J = 1.8, total 1H), 7.61-7.56 (m, 1H), 7.36-7.55 (m, 2H), 7.20-7.08 (m, 2H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 163.4, 160.0, 137.2, 137.0, 129.7, 129.1, 125.3, 124.8, 120.3, 118.8. GC-MS (m/z): 121 [M^+] for $\text{C}_7\text{H}_7\text{NO}$ (yield, 89 mg (0.74 mmol), 74%).



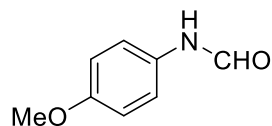
Product **2b**. ^1H NMR (CDCl_3 , 400 MHz) δ 8.23, 8.15 (s, and d, $J = 11.8$, total 1H), 7.40-7.22 (m, 5H), 6.07 (br, 1H), 4.46, 4.39 (d, $J = 6.45$, and d, $J = 6.45$, total 2H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 161.0, 137.6, 128.8, 127.8, 127.7, 42.2. GC-MS (m/z): 135 [M^+] for $\text{C}_8\text{H}_9\text{NO}$ (yield, 131 mg (0.97 mmol), 97%).



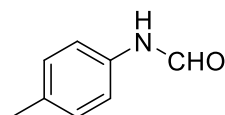
Product **3b**. ^1H NMR (CDCl_3 , 400 MHz) δ 8.64, 8.37 (d, $J = 11.44$ Hz, and s, total 1H), 7.49 (d, $J = 8.92$, 1H), 7.35-7.27 (m, 2H), 7.02 (d, $J = 8.77$, 1H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 162.5, 159.1, 135.4, 135.3, 130.8, 129.9, 129.9, 129.2, 121.2, 120.1. GC-MS (m/z): 155 [M^+] for $\text{C}_7\text{H}_6\text{ClNO}$ (yield, 105 mg (0.68 mmol), 68%).



Product **4b**. ^1H NMR (CDCl_3 , 400 MHz) δ 8.63, 8.26 (s, and d, $J = 1.73$ Hz, total 1H), 8.61, 7.81 (br, and br, total 1H), 7.26-7.09 (m, 1.5H), 6.97-6.93 (m, 0.5H), 6.66-6.53 (m, 2H), 3.71 (d, $J = 2.30$, 3H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 162.3, 160.7, 160.1, 159.4, 138.2, 138.0, 130.6, 129.8, 112.2, 110.9, 110.5, 110.4, 106.0, 104.9, 55.4, 55.3. GC-MS (m/z): 151 [M^+] for $\text{C}_8\text{H}_9\text{NO}_2$ (yield, 87 mg (0.58 mmol), 58%).

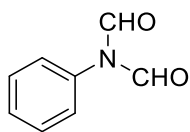


Product **5b**. ^1H NMR (CDCl_3 , 400 MHz) δ 8.51, 8.31 (d, $J = 11.54$ Hz, and d, $J = 1.87$ Hz, total 1H), 7.44 (d, $J = 9.13$, 1H), 7.03 (d, $J = 8.91$, 1H), 6.90-6.83 (m, 2H), 3.79 (d, $J = 6.00$, 3H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 163.2, 159.0, 157.6, 156.7, 130.0, 129.6, 121.8, 121.6, 114.9, 114.2, 55.6, 55.5. GC-MS (m/z): 151 [M^+] for $\text{C}_8\text{H}_9\text{NO}_2$ (yield, 98 mg (0.65 mmol), 65%).

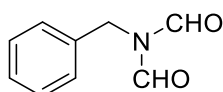


Product **6b**. ^1H NMR (CDCl_3 , 400 MHz) δ 8.62, 8.35 (d, $J = 11.35$ Hz, and br, total 1H), 7.42 (d, $J = 8.72$, 1H), 7.15 (t, $J = 8.54$, 2H), 6.97 (d, $J = 8.01$, 1H), 2.32 (d, $J = 6.47$, 3H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 162.9, 159.3, 135.2, 134.6, 134.2, 134.1, 130.2, 129.6, 120.2, 119.2, 20.9, 20.8. GC-MS (m/z): 135 [M^+] for $\text{C}_8\text{H}_9\text{NO}$ (yield, 86 mg (0.64 mmol), 64%).

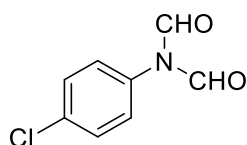
Double-*N*-formylation of primary amines



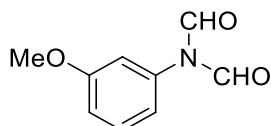
Product **1c**. ^1H NMR (CDCl_3 , 400 MHz) δ 8.98 (br, 2H), 7.52-7.38 (m, 3H), 7.22-7.13 (m, 2H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 163.3, 129.6, 129.3, 127.3. GC-MS (m/z): 149 [M^+] for $\text{C}_8\text{H}_7\text{NO}_2$ (yield, 67 mg (0.45 mmol), 90%).



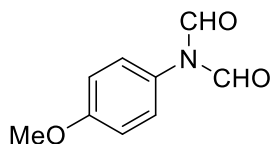
Product **2c**. ^1H NMR (CDCl_3 , 400 MHz) δ 8.79 (s, 2H), 7.39-7.23 (m, 5H), 4.76 (s, 2H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 164.2, 135.6, 128.8, 128.7, 128.0, 42.1. GC-MS (m/z): 163 [M^+] for $\text{C}_9\text{H}_9\text{NO}_2$ (yield, 69 mg (0.42 mmol), 85%).



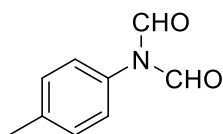
Product **3c**. ^1H NMR (CDCl_3 , 400 MHz) δ 9.02 (br, 2H), 7.44 (d, $J = 8.1$, 2H), 7.14 (d, $J = 7.9$, 2H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 162.7, 135.3, 129.8, 128.7. GC-MS (m/z): 183 [M^+] for $\text{C}_8\text{H}_6\text{ClNO}_2$ (yield, 67 mg (0.37 mmol), 73%).



Product **4c**. ^1H NMR (CDCl_3 , 400 MHz) δ 9.09 (br, 2H), 7.40 (d, $J = 8.23$, 1H), 7.01-6.97 (m, 1H), 6.82-6.79 (d, $J = 7.92$, 1H), 7.76 (br, 1H), 3.82 (s, 3H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 162.8, 160.5, 130.4, 119.4, 115.0, 113.2, 55.5. GC-MS (m/z): 179 [M^+] for $\text{C}_9\text{H}_9\text{NO}_3$ (yield, 74 mg (0.41 mmol), 83%).

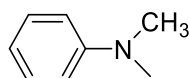


Product **5c**. ^1H NMR (CDCl_3 , 400 MHz) δ 9.06 (br, 2H), 7.13 (d, $J = 8.78$, 2H), 6.99 (d, $J = 8.94$, 2H), 3.83 (s, 3H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 163.2, 160.1, 128.5, 114.9, 55.6. GC-MS (m/z): 179 [M^+] for $\text{C}_9\text{H}_9\text{NO}_3$ (yield, 79 mg (0.45 mmol), 89%).

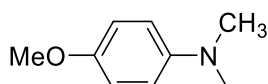


Product **6c**. ^1H NMR (CDCl_3 , 400 MHz) δ 9.01 (br, 2H), 7.28 (d, J = 8.72, 1H), 7.08 (d, J = 8.25, 2H), 2.38 (s, 3H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 163.3, 139.5, 130.3, 127.1, 21.2. GC-MS (m/z): 163 [M^+] for $\text{C}_9\text{H}_9\text{NO}_2$ (yield, 75 mg (0.46 mmol), 92%).

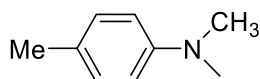
N-Methylation of secondary amines



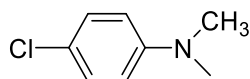
Product **1d**. ^1H NMR (CDCl_3 , 400 MHz) δ 7.30 (t, J = 7.6 Hz, 2H), 6.80 (m, 3H), 7.16 (d, J = 8.0 Hz, 2H), 2.99 (s, 6H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 150.6, 129.4, 116.8, 112.8, 40.7. GC-MS (m/z): 121 [M^+] for $\text{C}_8\text{H}_{11}\text{N}$ (yield, 110 mg (0.92 mmol), 92%).



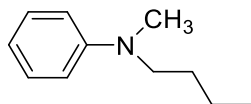
Product **2d**. ^1H NMR (CDCl_3 , 400 MHz) δ 6.87-6.82 (m, 2H), 6.78-6.73 (m, 2H), 3.76 (s, 3H), 2.87 (s, 6H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 152.0, 145.8, 114.9, 114.7, 55.8, 41.8. GC-MS (m/z): 151 [M^+] for $\text{C}_9\text{H}_{13}\text{NO}$ (yield, 139 mg (0.92 mmol), 92%).



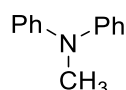
Product **3d**. ^1H NMR (CDCl_3 , 400 MHz) δ 7.06 (d, J = 8.5 Hz, 2H), 6.71 (d, J = 8.5 Hz, 2H), 2.91 (s, 6H), 2.27 (s, 3H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 148.9, 129.7, 126.3, 113.4, 41.2, 20.3. GC-MS (m/z): 135 [M^+] for $\text{C}_9\text{H}_{13}\text{N}$ (yield, 119 mg (0.88 mmol), 88%).



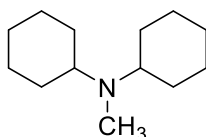
Product **4d**. ^1H NMR (CDCl_3 , 400 MHz) δ 7.17 (d, J = 9.2 Hz, 2H), 6.64 (d, J = 9.2 Hz, 2H), 2.92 (s, 6H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 149.2, 128.8, 121.4, 113.7, 40.7. GC-MS (m/z): 155 [M^+] for $\text{C}_8\text{H}_{10}\text{ClN}$ (yield, 97 mg (0.63 mmol), 63%).



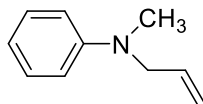
Product **5d**. ^1H NMR (CDCl_3 , 400 MHz) δ 7.25 (m, 2H), 6.74-6.67 (m, 3H), 3.33 (t, J = 7.5 Hz, 2H), 2.95 (s, 3H), 1.62-1.55 (m, 2H), 1.42-1.33 (m, 2H), 0.98 (t, J = 7.3 Hz, 3H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 149.4, 129.1, 115.7, 112.1, 52.5, 38.3, 28.9, 20.4, 14.0. GC-MS (m/z): 163 [M^+] for $\text{C}_{11}\text{H}_{17}\text{N}$ (yield, 161 mg (0.99 mmol), 99%).



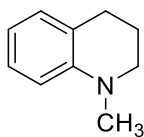
Product **6d**. ^1H NMR (CDCl_3 , 400 MHz) δ 7.46-6.90 (m, 10H), 3.38 (s, 3H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 149.0, 129.3, 121.3, 120.5, 40.3. GC-MS (m/z): 183 [M^+] for $\text{C}_{13}\text{H}_{13}\text{N}$ (yield, 141 mg (0.77 mmol), 77%).



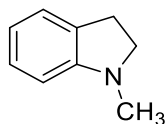
Product **7d**. ^1H NMR (CDCl_3 , 400 MHz) δ 2.53-2.41 (m, 2H), 2.21 (s, 3H), 1.81-1.71 (m, 8H), 1.62-1.55 (m, 2H), 1.27-1.00 (m, 10H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 59.4, 32.9, 30.5, 26.3, 26.2. GC-MS (m/z): 195 [M^+] for $\text{C}_{13}\text{H}_{25}\text{N}$ (yield, 156 mg (0.80 mmol), 80%).



Product **8d**. ^1H NMR (CDCl_3 , 400 MHz) δ 7.32-7.25 (m, 2H), 6.82-6.73 (m, 3H), 5.97-5.85 (m, 1H), 5.27-5.17 (m, 2H), 3.99-3.94 (t, 2H), 2.99 (s, 3H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 149.5, 133.7, 129.1, 116.5, 116.2, 112.5, 55.3, 38.0. GC-MS (m/z): 147 [M^+] for $\text{C}_{10}\text{H}_{13}\text{N}$ (yield, 123 mg (0.84 mmol), 84%).

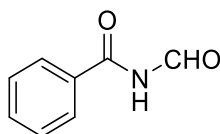


Product **9d**. ^1H NMR (CDCl_3 , 400 MHz) δ 7.12 (t, J =7.8, 1H), 6.99 (d, J =7.5, 1H), 6.68-6.62 (m, 2H), 3.28 (t, J =5.7, 2H), 2.93 (s, 3H), 2.81 (t, J =6.5, 2H), 2.07-1.99 (m, 2H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 146.8, 128.9, 127.1, 122.9, 116.3, 111.0, 51.3, 39.2, 27.9, 22.5. GC-MS (m/z): 147 [M^+] for $\text{C}_{10}\text{H}_{13}\text{N}$ (yield, 116 mg (0.79 mmol), 79%).

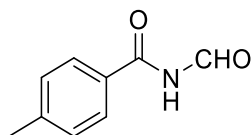


Product **10d**. ^1H NMR (CDCl_3 , 400 MHz) δ 7.12-7.07 (m, 2H), 6.69 (t, J = 7.3, 1H), 6.51 (d, J = 8.0, 1H), 3.30 (t, J = 8.2, 2H), 2.95 (t, J = 8.2, 2H), 2.77 (m, 3H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 153.4, 130.3, 127.3, 124.3, 117.8, 107.2, 56.2, 36.3, 28.8. GC-MS (m/z): 133 [M^+] for $\text{C}_9\text{H}_{11}\text{N}$ (yield, 86 mg (0.65 mmol), 65%).

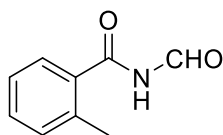
N-Formylation of primary amides



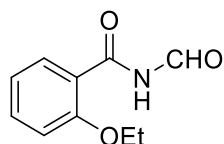
Product **1e**. ^1H NMR (CDCl_3 , 400 MHz) δ 9.86 (s, 1H), δ 9.41 (d, J = 5.3 Hz, 1H), 7.98 (d, J = 3.7 Hz, 2H), 7.70-7.63 (m, 2H), 7.57-7.51 (m, 2H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 166.8, 165.1, 134.0, 131.1, 129.1, 128.2. GC-MS (m/z): 149 [M^+] for $\text{C}_8\text{H}_7\text{NO}_2$ (yield, 55 mg (0.37 mmol), 74%).



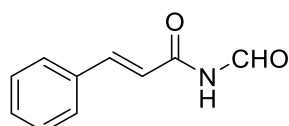
Product **2e**. ^1H NMR (CDCl_3 , 400 MHz) δ 9.89 (d, J = 4.7 Hz, 1H), 9.38 (d, J = 4.7 Hz, 1H), 7.89-7.85 (m, 2H), 7.35-7.31 (m, 2H), 2.44 (s, 3H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 166.6, 164.8, 145.0, 129.8, 128.3, 128.2, 21.7. GC-MS (m/z): 163 [M^+] for $\text{C}_9\text{H}_9\text{NO}_2$ (yield, 61 mg (0.37 mmol), 75%).



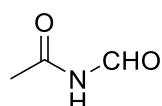
Product **3e**. ^1H NMR (CDCl_3 , 400 MHz) δ 9.24 (d, J = 10.1 Hz, 1H), 8.57 (br, 1H), 7.51-7.43 (m, 2H), 7.34-7.28 (m, 2H), 2.53 (s, 3H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 168.6, 163.3, 138.4, 132.2, 132.0, 132.0, 127.3, 126.2, 20.3. GC-MS (m/z): 163 [M^+] for $\text{C}_9\text{H}_9\text{NO}_2$ (yield, 60 mg (0.37 mmol), 74%).



Product **4e**. ^1H NMR (CDCl_3 , 400 MHz) δ 10.34 (br, 1H), 9.38 (d, J = 9.9 Hz, 1H), 8.19 (dd, J = 8.0 and 1.9), 7.57-7.52 (m, 1H), 7.13-7.07 (m, 1H), 7.00 (d, J = 8.7 Hz, 1H), 4.25 (q, J = 7.0 Hz, 2H), 1.56 (t, J = 7.0 Hz, 3H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 165.3, 163.1, 157.9, 135.4, 132.7, 121.7, 118.9, 112.7, 65.3, 14.6. GC-MS (m/z): 193 [M^+] for $\text{C}_{10}\text{H}_{11}\text{NO}_3$ (yield, 68 mg (0.35 mmol), 71%).

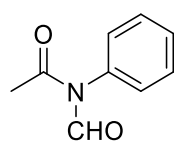


Product **5e**. ^1H NMR (CDCl_3 , 400 MHz) δ 9.29 (d, J = 4.8 Hz, 1H), 9.13 (s, 1H), 7.88 (d, J = 7.8 Hz, 1H), 7.61-7.56 (m, 2H), 7.48-7.40 (m, 3H), 6.51 (d, J = 7.8 Hz, 1H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 156.5, 164.5, 146.6, 133.7, 131.2, 129.1, 128.6, 118.0. GC-MS (m/z): 175 [M^+] for $\text{C}_{10}\text{H}_9\text{NO}_2$ (yield, 48 mg (0.27 mmol), 55%).

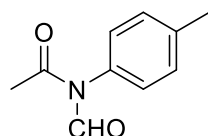


Product **6e**. ^1H NMR (CDCl_3 , 400 MHz) δ 9.08 (d, J = 9.2 Hz, 1H), 8.90 (br, 1H), 2.20 (s, 3H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 170.5, 162.3, 23.7. GC-MS (m/z): 87 [M^+] for $\text{C}_3\text{H}_5\text{NO}_2$ (yield, 37 mg (0.43 mmol), 86%).

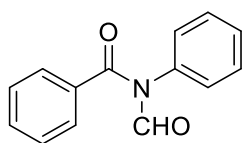
N-Formylation of secondary amides



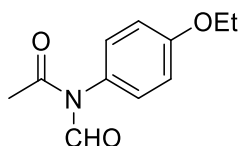
Product **1f**. ^1H NMR (CDCl_3 , 400 MHz) δ 9.57 (s, 1H), 7.52-7.43 (m, 3H), 7.18-7.14 (m, 2H), 2.06 (s, 2H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 172.9, 162.5, 135.6, 130.0, 129.5, 128.4, 24.4. GC-MS (m/z): 163 [M^+] for $\text{C}_9\text{H}_9\text{NO}_2$ (yield, 142 mg (0.87 mmol), 87%).



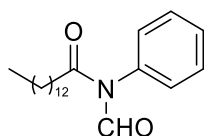
Product **2f**. ^1H NMR (CDCl_3 , 400 MHz) δ 9.57 (s, 1H), 7.34-7.30 (m, 2H), 7.08-7.04 (m, 2H), 2.43 (s, 3H), 2.09 (s, 3H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 173.1, 162.6, 139.6, 132.9, 130.6, 128.1, 24.4, 21.2. GC-MS (m/z): 177 [M^+] for $\text{C}_{10}\text{H}_{11}\text{NO}_2$ (yield, 161 mg (0.91 mmol), 91%).



Product **3f**. ^1H NMR (CDCl_3 , 400 MHz) δ 9.46 (s, 1H), 7.58-7.13 (m, 10H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 171.8, 163.4, 135.6, 133.2, 132.3, 129.4, 128.5, 128.1. GC-MS (m/z): 225 [M^+] for $\text{C}_{14}\text{H}_{11}\text{NO}_2$ (yield, 196 mg (0.87 mmol), 87%).

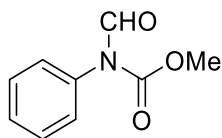


Product **4f**. ^1H NMR (CDCl_3 , 400 MHz) δ 9.56 (s, 1H), 7.07-7.02 (m, 2H), 6.99-6.94 (m, 2H), 4.05 (q, J = 7.0 Hz, 2H), 2.06 (s, 3H), 1.43 (t, J = 7.0 Hz, 3H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 173.4, 162.6, 159.5, 129.4, 127.8, 115.6, 63.8, 24.3, 14.7. GC-MS (m/z): 207 [M^+] for $\text{C}_{11}\text{H}_{13}\text{NO}_3$ (yield, 167 mg (0.81 mmol), 81%).

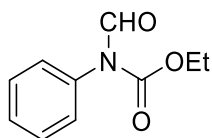


Product **5f**. ^1H NMR (CDCl_3 , 400 MHz) δ 9.60 (s, 1H), 7.53-7.45 (m, 3H), 7.17-7.13 (m, 2H), 2.22 (t, J = 7.3 Hz, 2H), 1.65-1.56 (m, 2H), 1.29-1.19 (m, 20H), 0.88 (t, J = 6.8 Hz, 3H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 175.7, 162.6, 135.3, 130.0, 129.4, 128.6, 36.1, 31.9, 29.7, 29.6, 29.6, 29.5, 29.4, 29.4, 29.3, 28.9, 24.1, 22.7, 14.1. GC-MS (m/z): 331 [M^+] for $\text{C}_{21}\text{H}_{33}\text{NO}_2$ (yield, 327 mg (0.99 mmol), 99%).

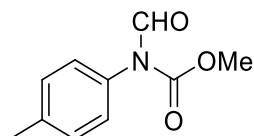
***N*-Formylation of carbamates**



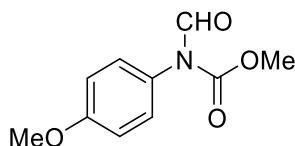
Product **1g**. ^1H NMR (CDCl_3 , 400 MHz) δ 9.47 (s, 1H), 7.48-7.39 (m, 3H), 7.17-7.13 (m, 2H), 3.86 (s, 3H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 162.7, 154.5, 134.0, 129.3, 128.9, 128.2, 54.2. GC-MS (m/z): 179 [M^+] for $\text{C}_9\text{H}_9\text{NO}_3$ (yield, 130 mg (0.73 mmol), 73%).



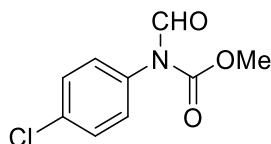
Product **2g**. ^1H NMR (CDCl_3 , 400 MHz) δ 9.47 (s, 1H), 7.49-7.39 (m, 3H), 7.17-7.13 (m, 2H), 4.33 (q, J = 7.0 Hz, 2H), 1.29 (t, J = 7.0 Hz, 3H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 162.8, 153.9, 134.1, 129.2, 128.8, 128.2, 63.7, 14.2. GC-MS (m/z): 193 [M^+] for $\text{C}_{10}\text{H}_{11}\text{NO}_3$ (yield, 121 mg (0.63 mmol), 63%).



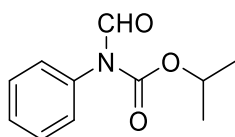
Product **3g**. ^1H NMR (CDCl_3 , 400 MHz) δ 9.46 (s, 1H), 7.26 (d, J = 8.0, 2H), 7.03 (d, J = 8.0, 2H), 3.86 (s, 3H), 2.39 (s, 3H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 162.9, 154.7, 139.0, 131.3, 130.1, 129.6, 127.9, 54.2, 21.2. GC-MS (m/z): 193 [M^+] for $\text{C}_{10}\text{H}_{11}\text{NO}_3$ (yield, 152 mg (0.79 mmol), 79%).



Product **4g**. ^1H NMR (CDCl_3 , 400 MHz) δ 9.45 (s, 1H), 7.08-7.04 (m, 2H), 6.97-6.93 (m, 2H), 3.85 (s, 3H), 3.82 (s, 3H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 163.0, 159.7, 154.8, 129.2, 126.5, 114.6, 114.3, 55.5, 54.2. GC-MS (m/z): 209 [M^+] for $\text{C}_{10}\text{H}_{11}\text{NO}_4$ (yield, 175 mg (0.84 mmol), 84%).

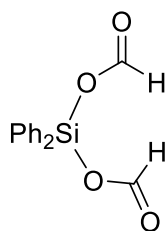


Product **5g**. ^1H NMR (CDCl_3 , 400 MHz) δ 9.45 (s, 1H), 7.45-7.41 (m, 2H), 7.11-7.07 (m, 2H), 3.87 (s, 3H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 162.5, 154.1, 134.9, 132.4, 129.6, 129.6, 54.3. GC-MS (m/z): 213 [M^+] for $\text{C}_9\text{H}_8\text{ClNO}_3$ (yield, 157 mg (0.74 mmol), 74%).



Product **6g**. ^1H NMR (CDCl_3 , 400 MHz) δ 9.46 (s, 1H), 7.48-7.37 (m, 3H), 7.16-7.12 (m, 2H), 5.13 (sept, J = 6.3 Hz, 1H), 1.28 (d, J = 6.3 Hz, 6H); ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 100 MHz) δ 162.9, 153.4, 134.2, 129.1, 128.6, 128.2, 72.1, 21.7. GC-MS (m/z): 207 [M^+] for $\text{C}_{11}\text{H}_{13}\text{NO}_3$ (yield, 159 mg (0.77 mmol), 77%).

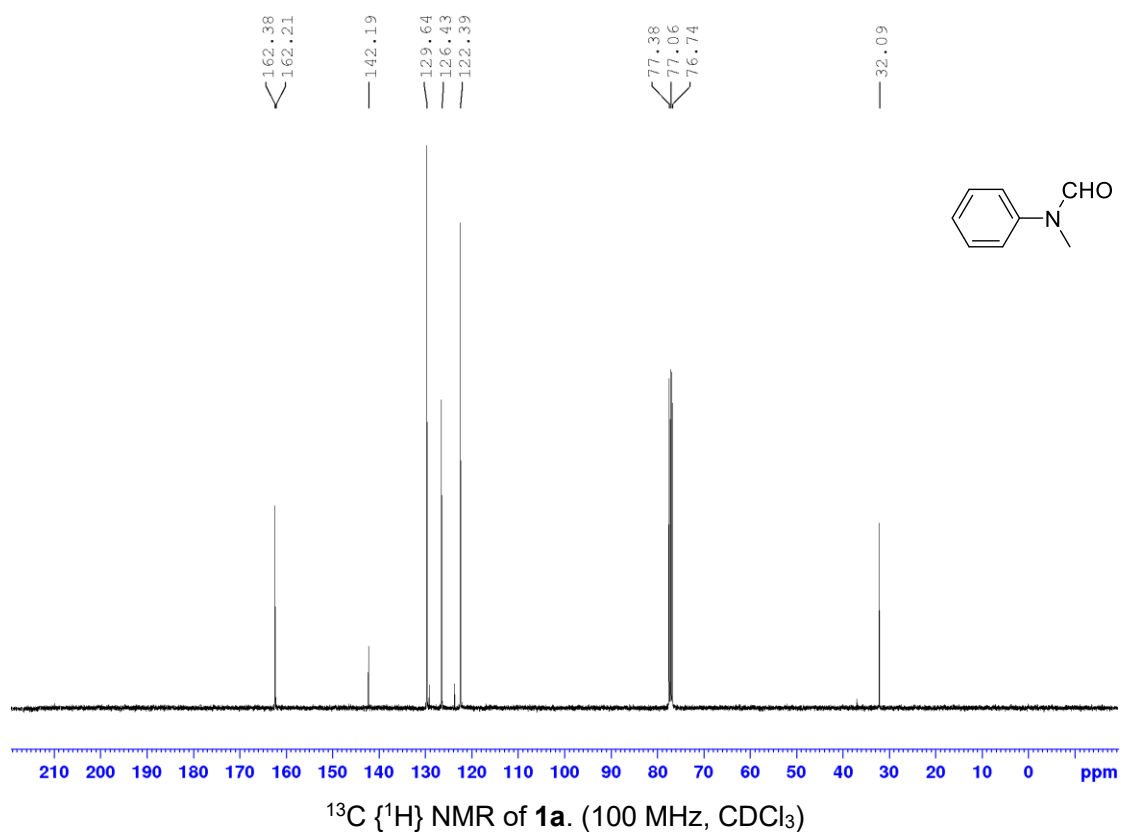
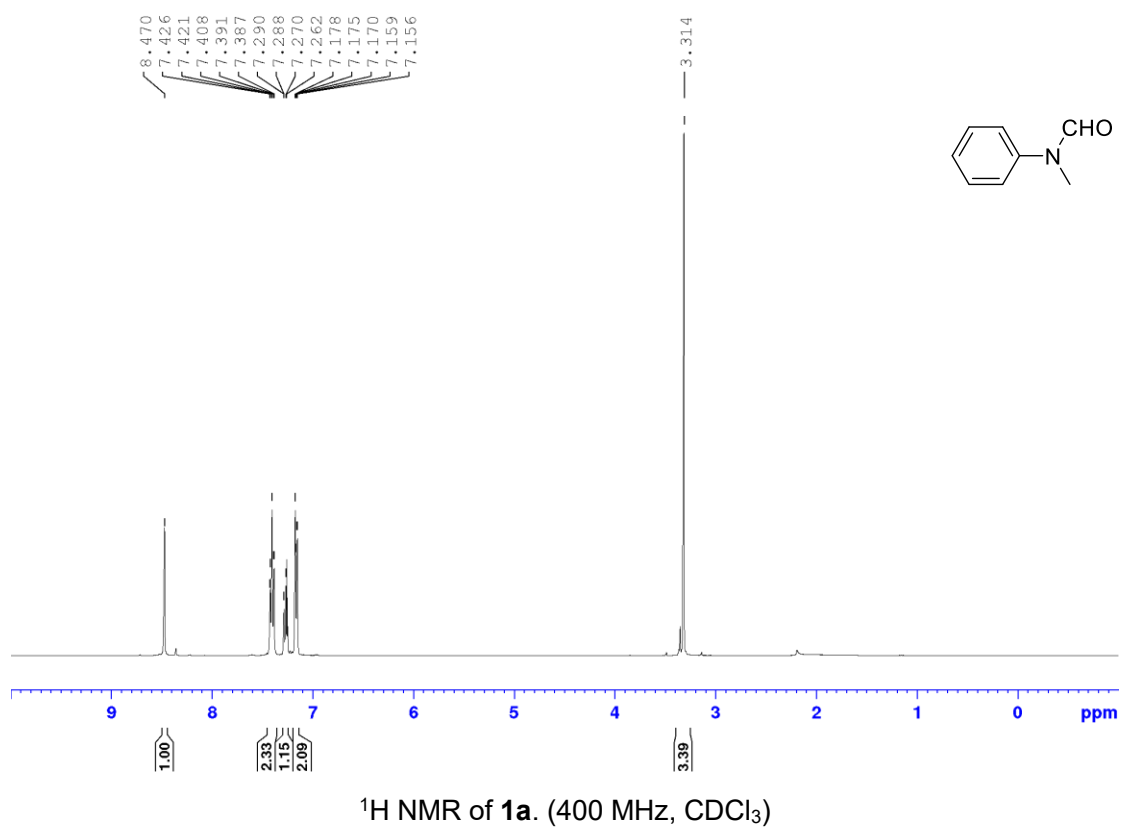
Data of Ph₂Si(OCHO)₂^{S1}

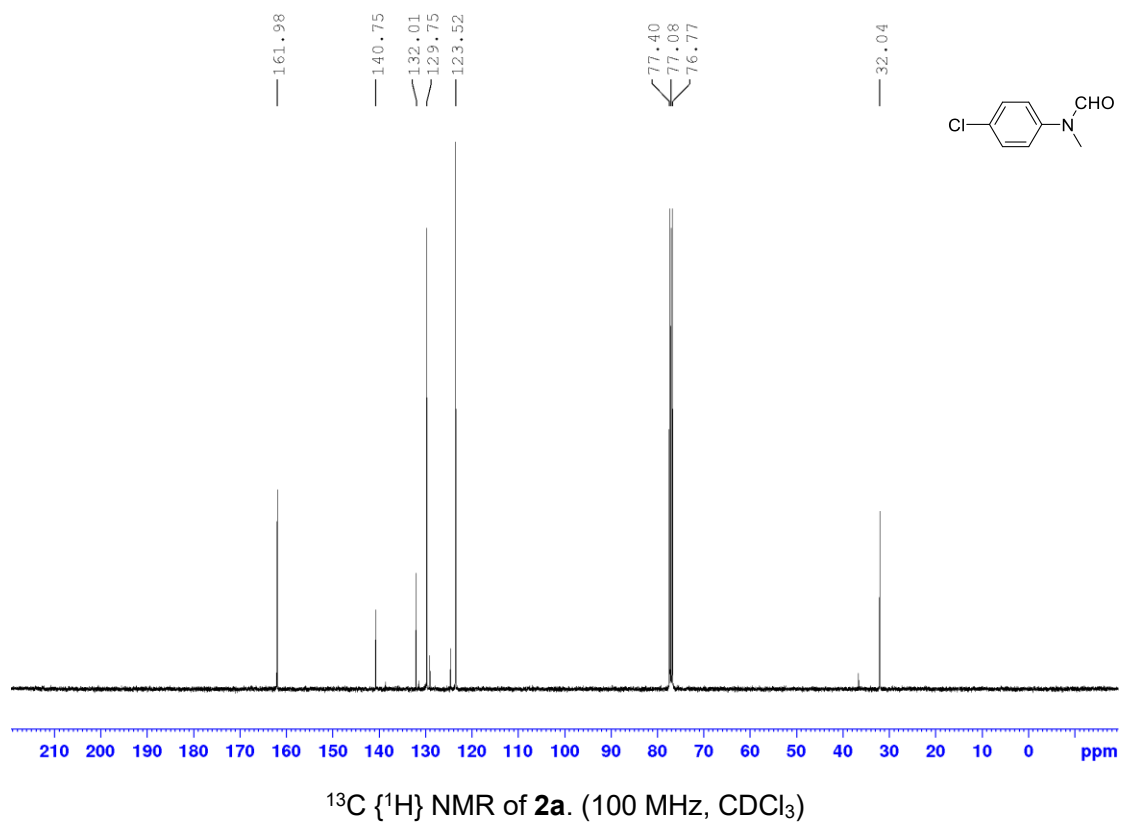
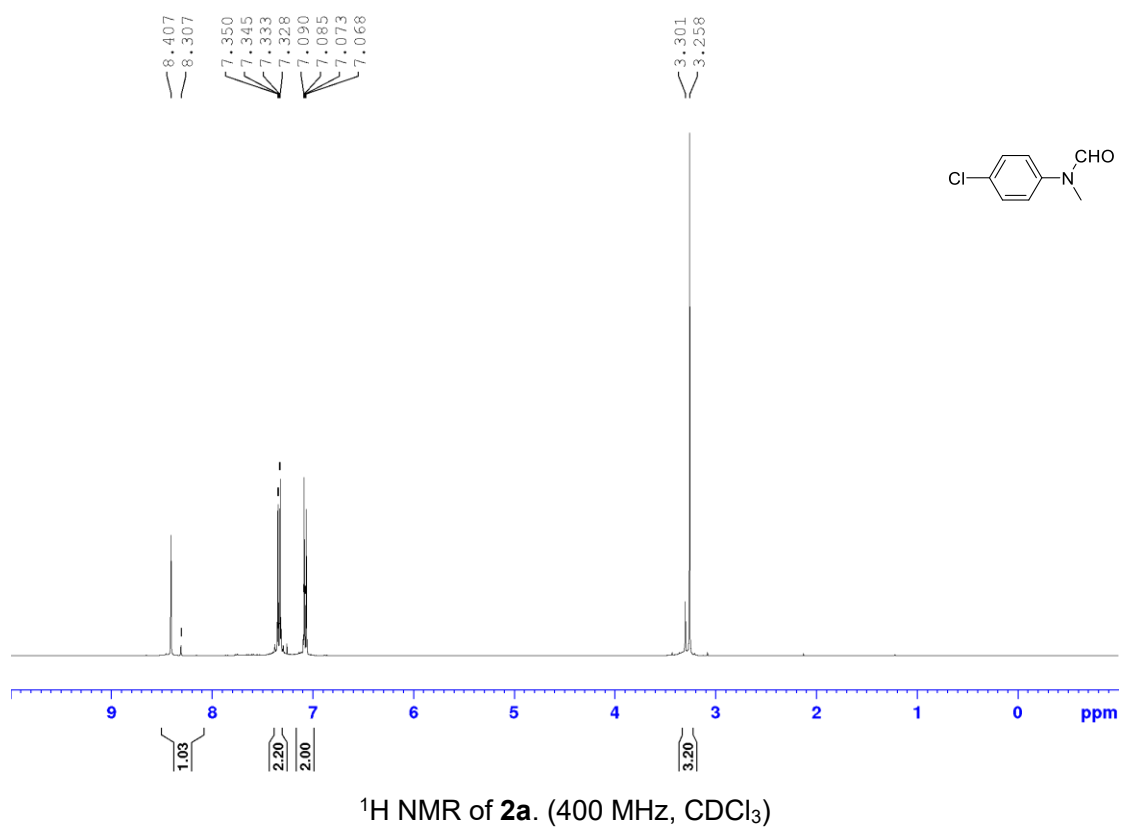


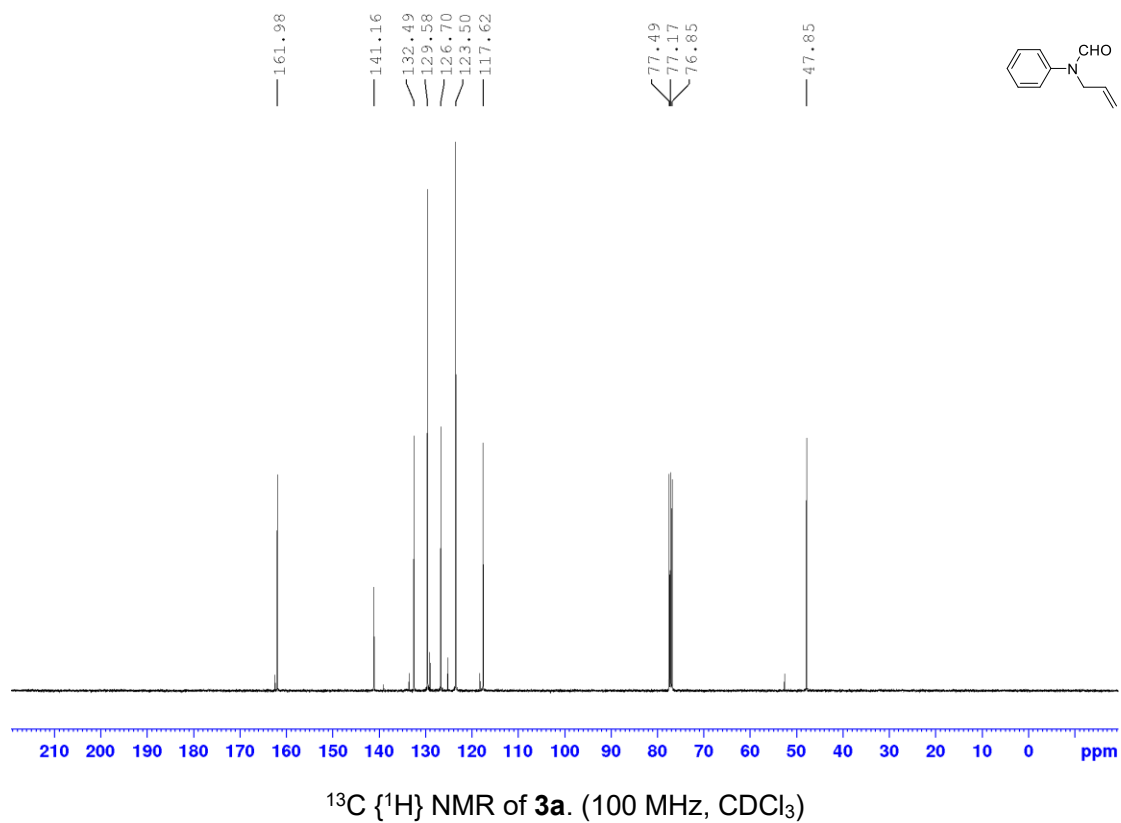
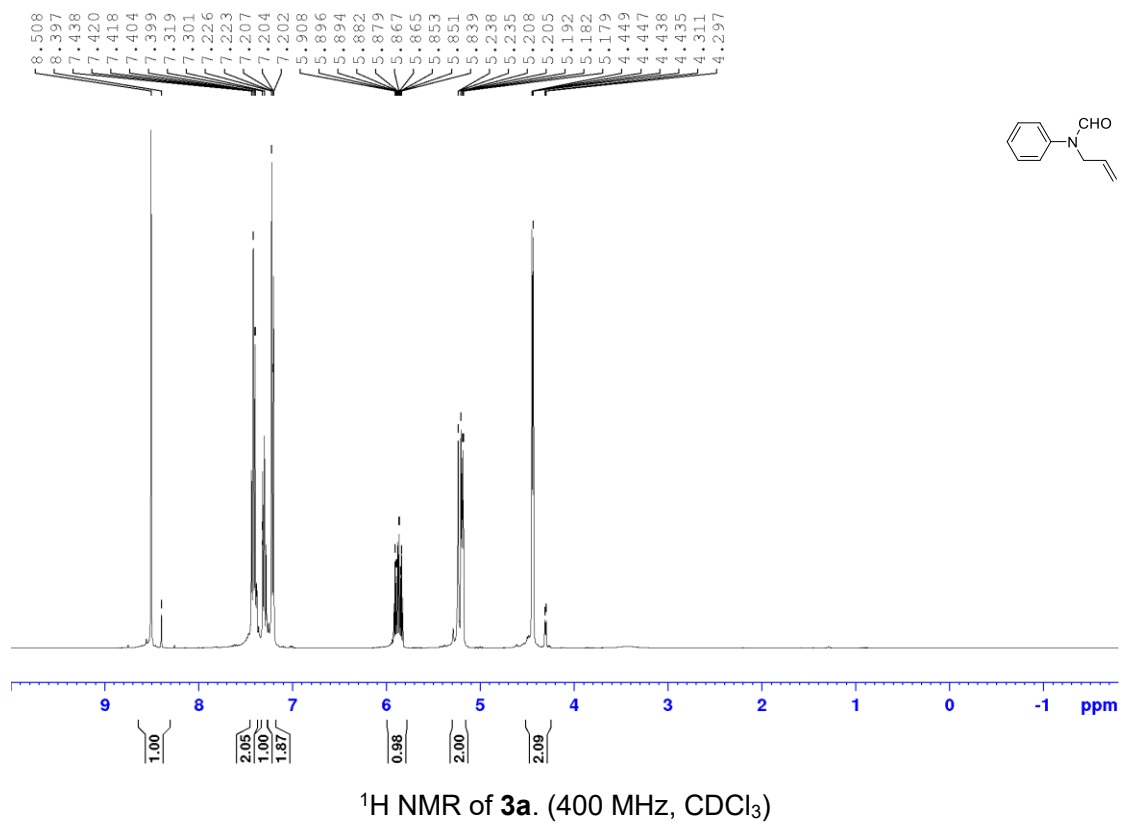
¹H NMR (CD₃CN, 400 MHz) δ 8.28 (s, 1H), 7.80 (d, *J* = 8.0 Hz, 4H), 7.61 (t, *J* = 8.0 Hz, 2H), 7.52 (t, *J* = 8.0 Hz, 4H); ¹³C {¹H} NMR (CD₃CN, 100 MHz) δ 160.5, 135.8, 133.0, 129.4, 129.2; ²⁹Si {¹H} NMR (CD₃CN, 79.5 MHz): δ -27.7. GC-MS (*m/z*): 272 [M⁺] for C₁₄H₁₂O₄Si.

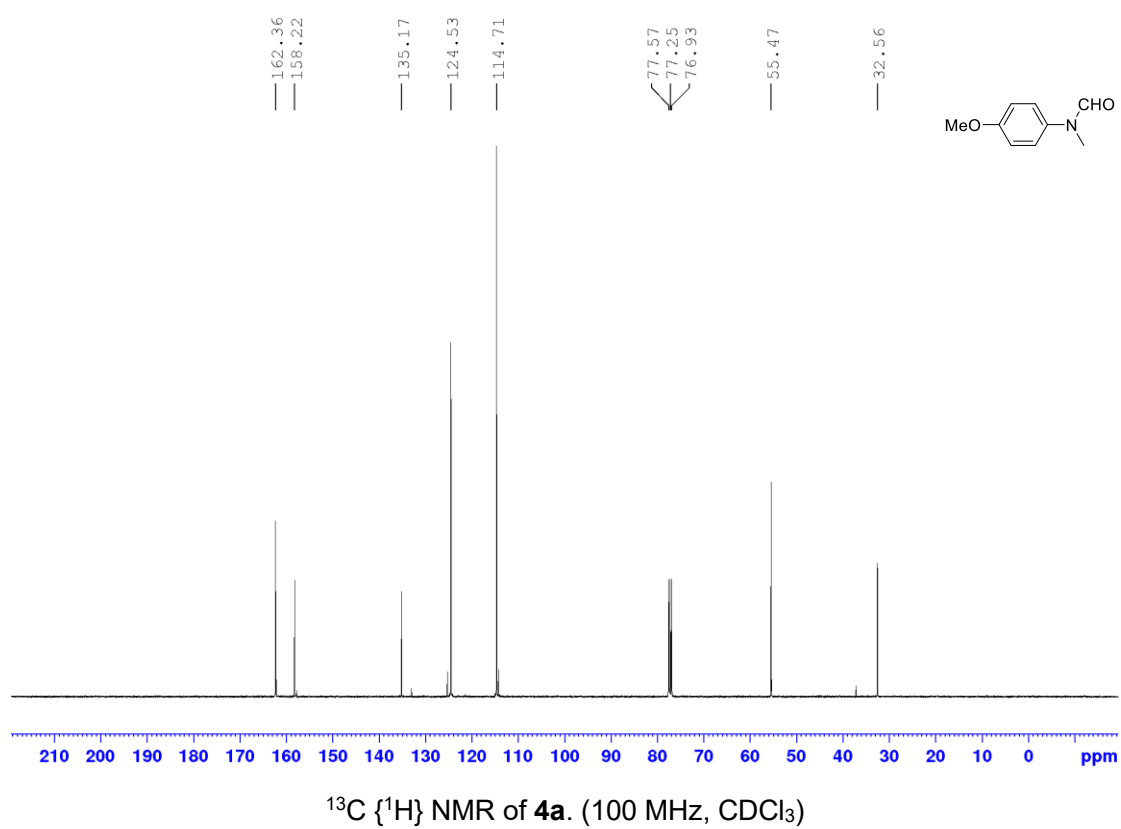
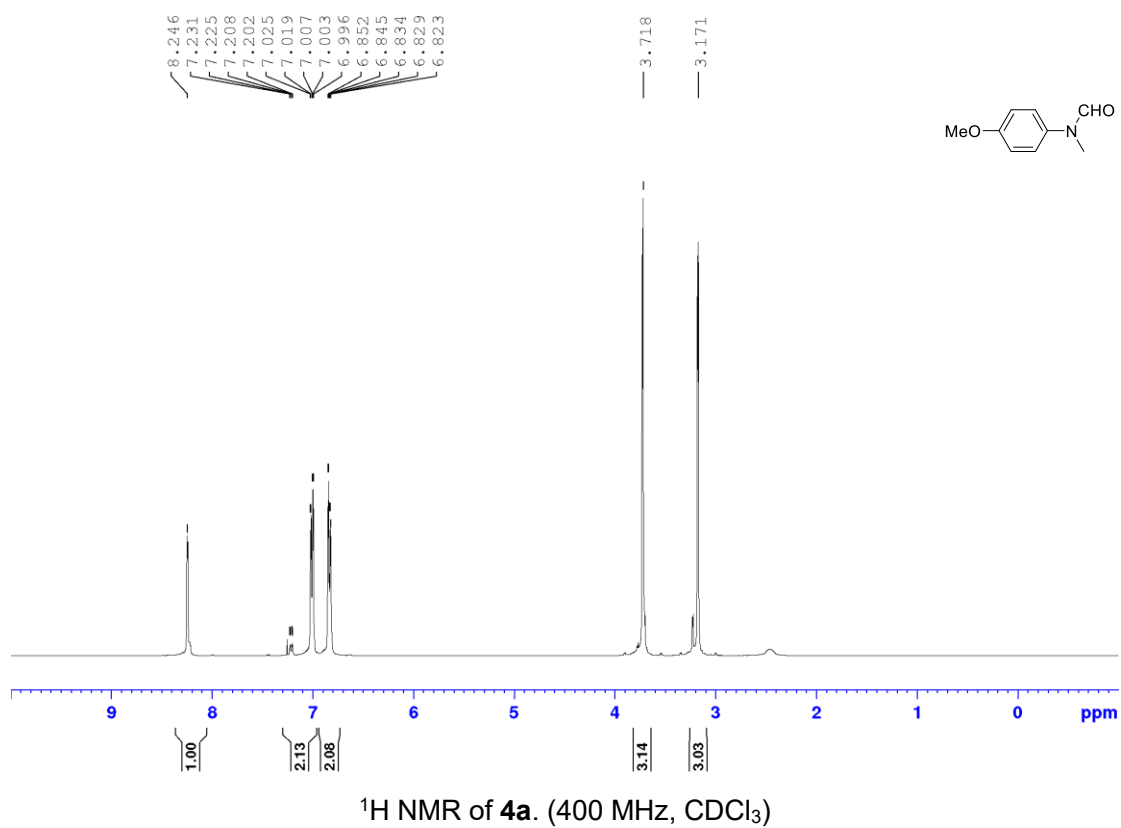
References

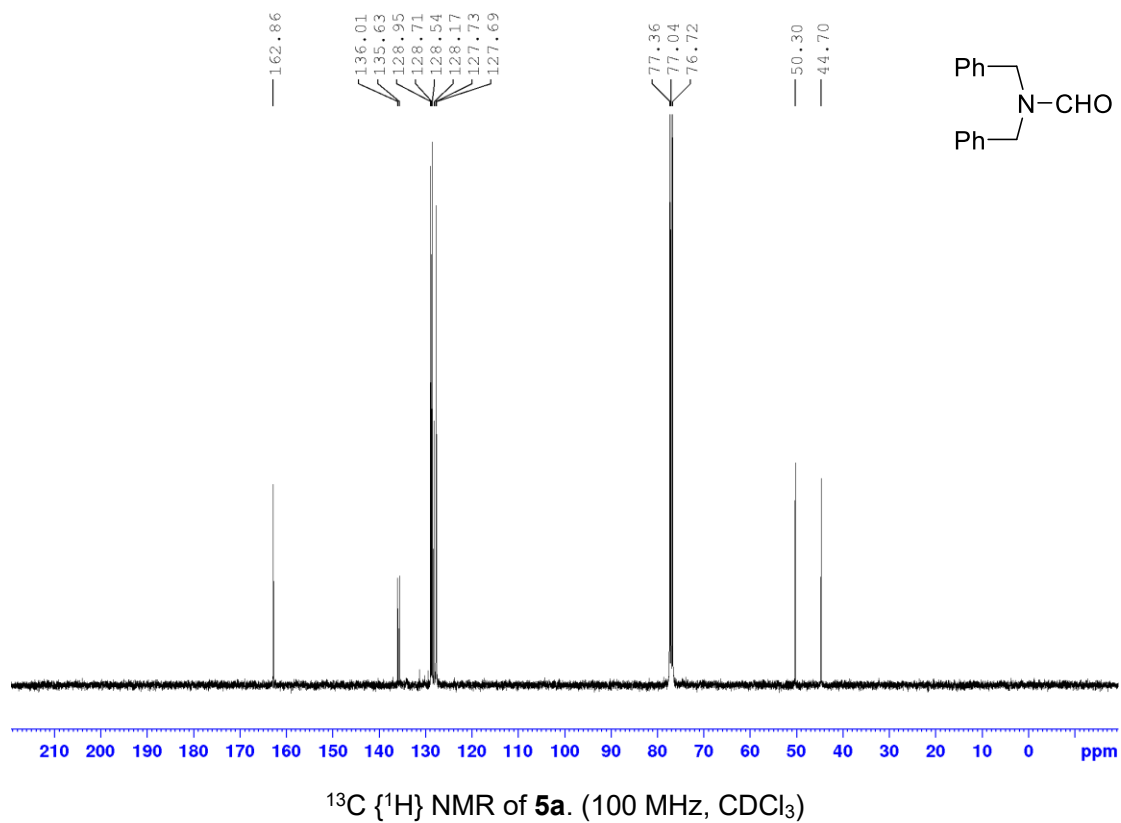
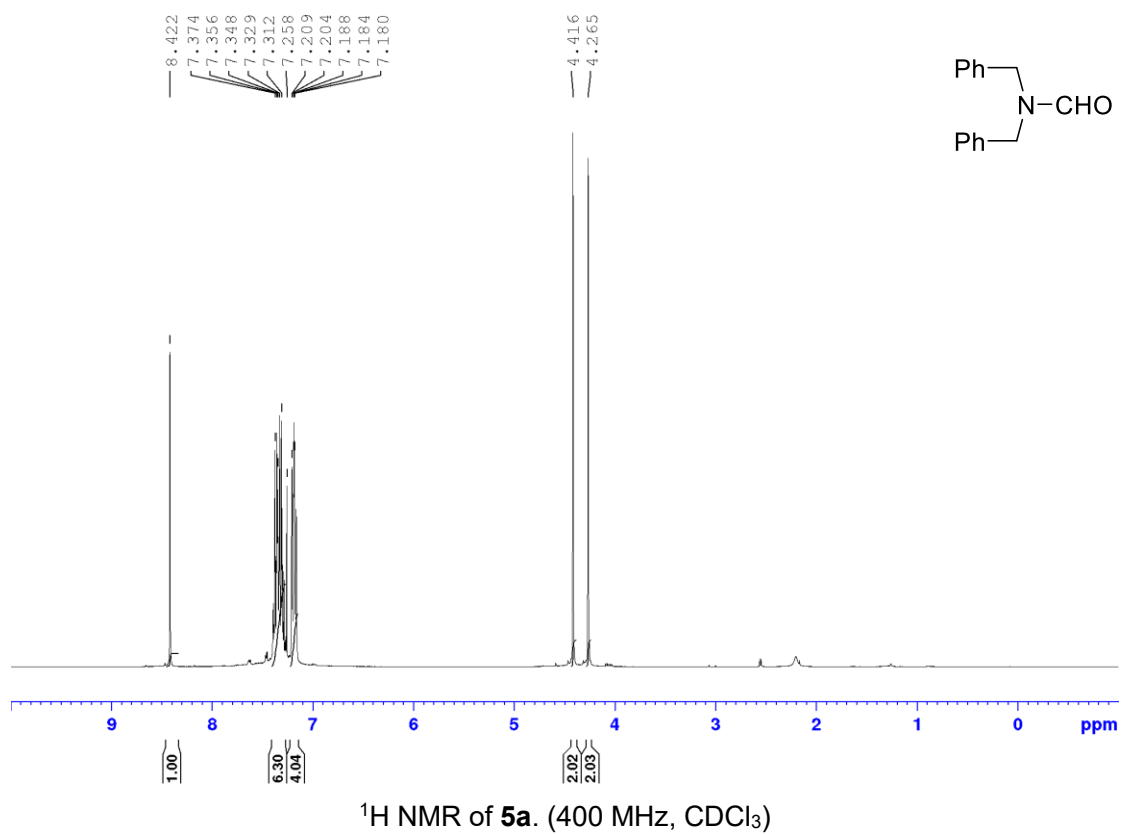
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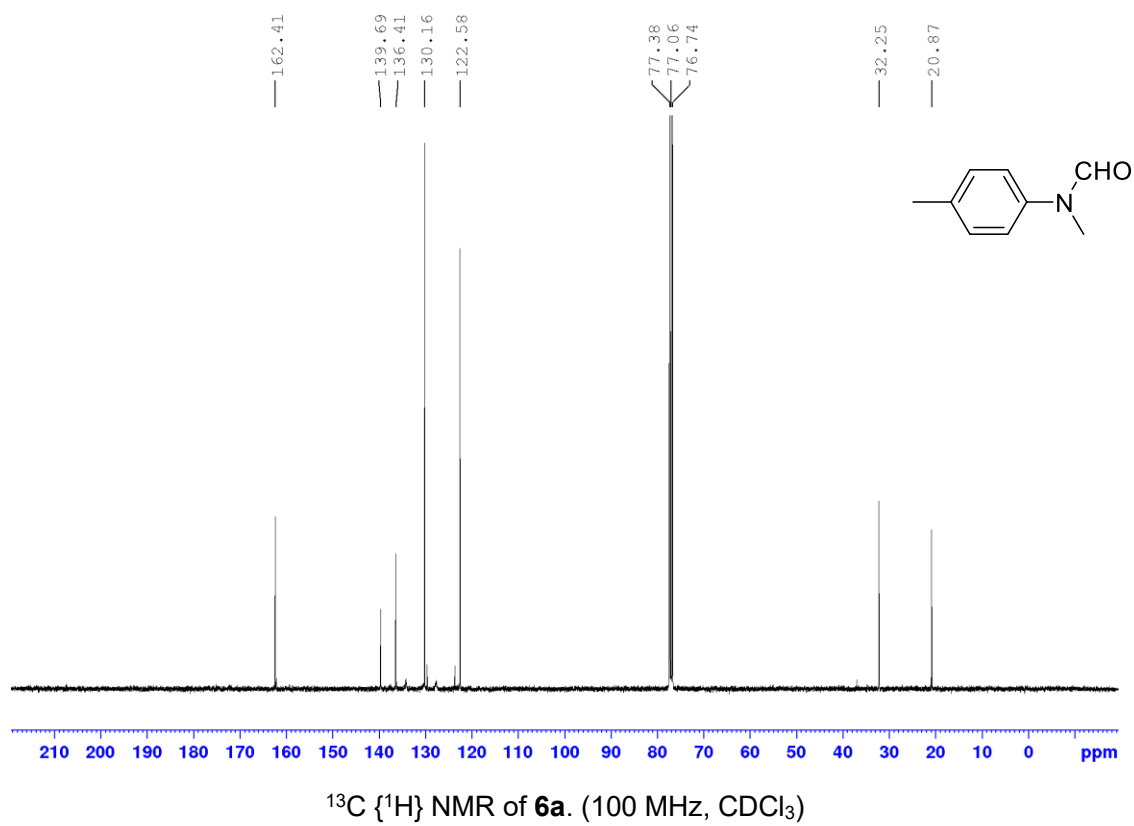
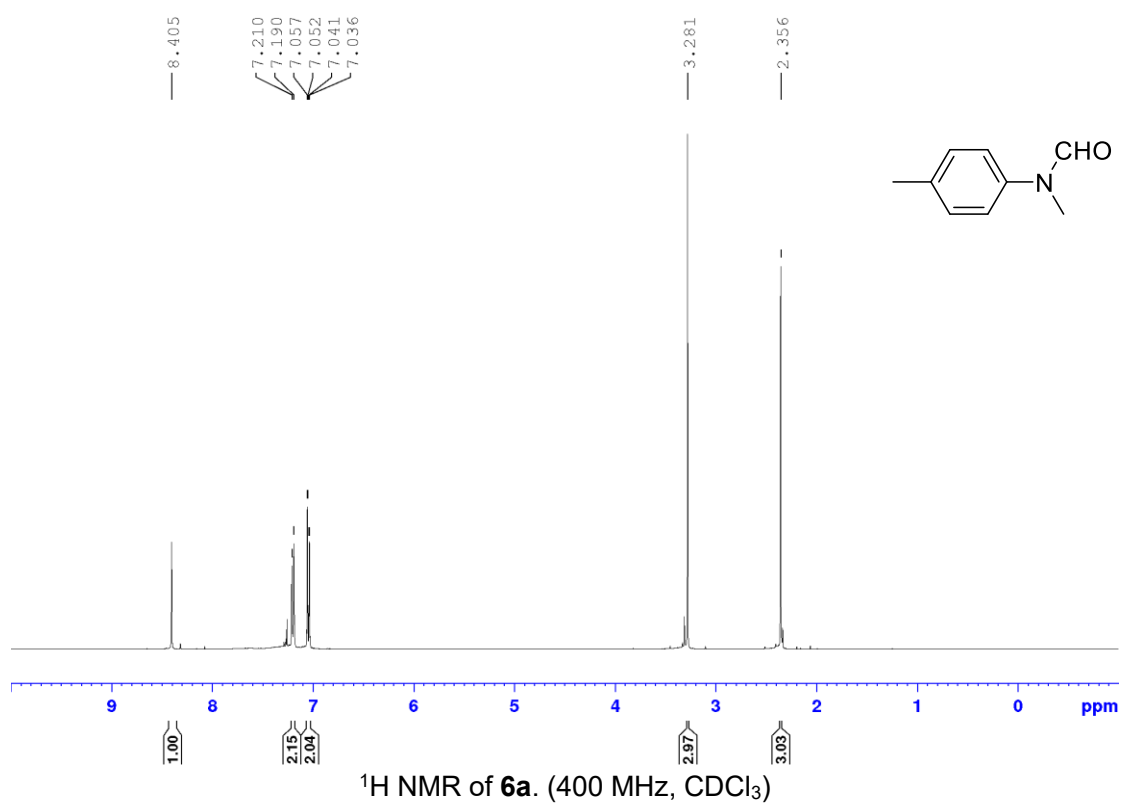


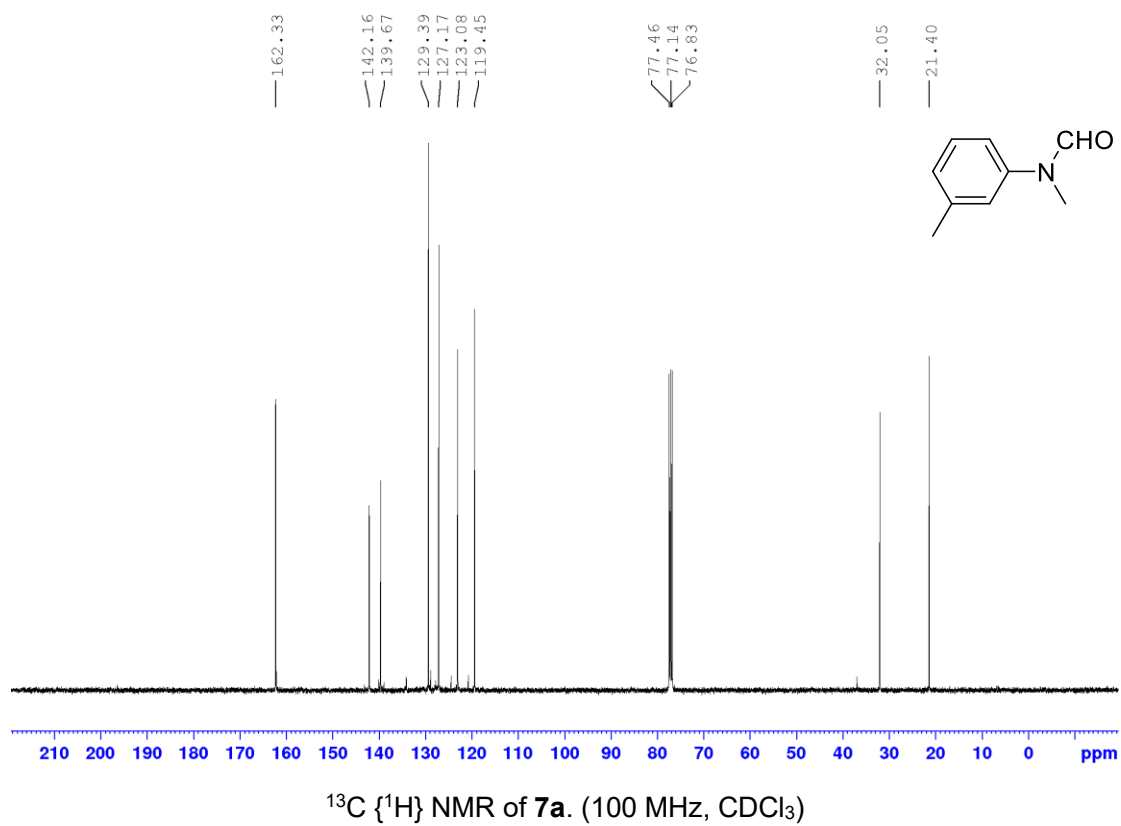
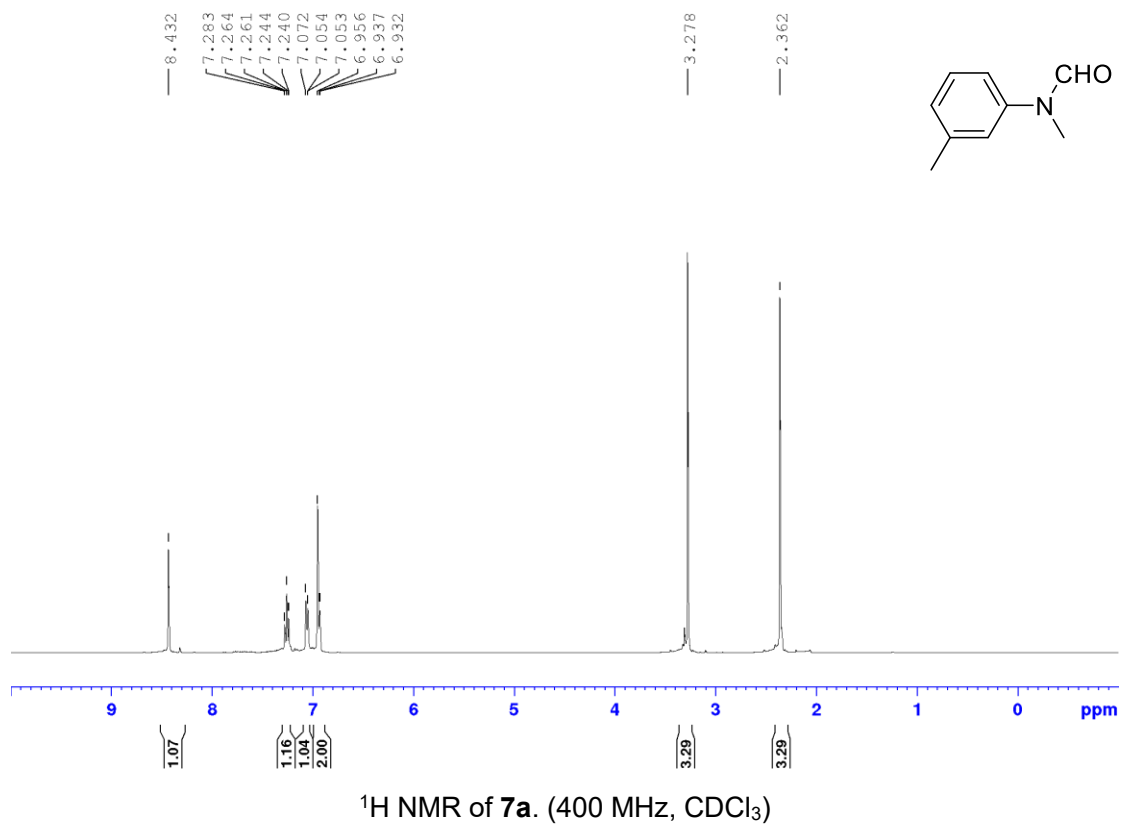


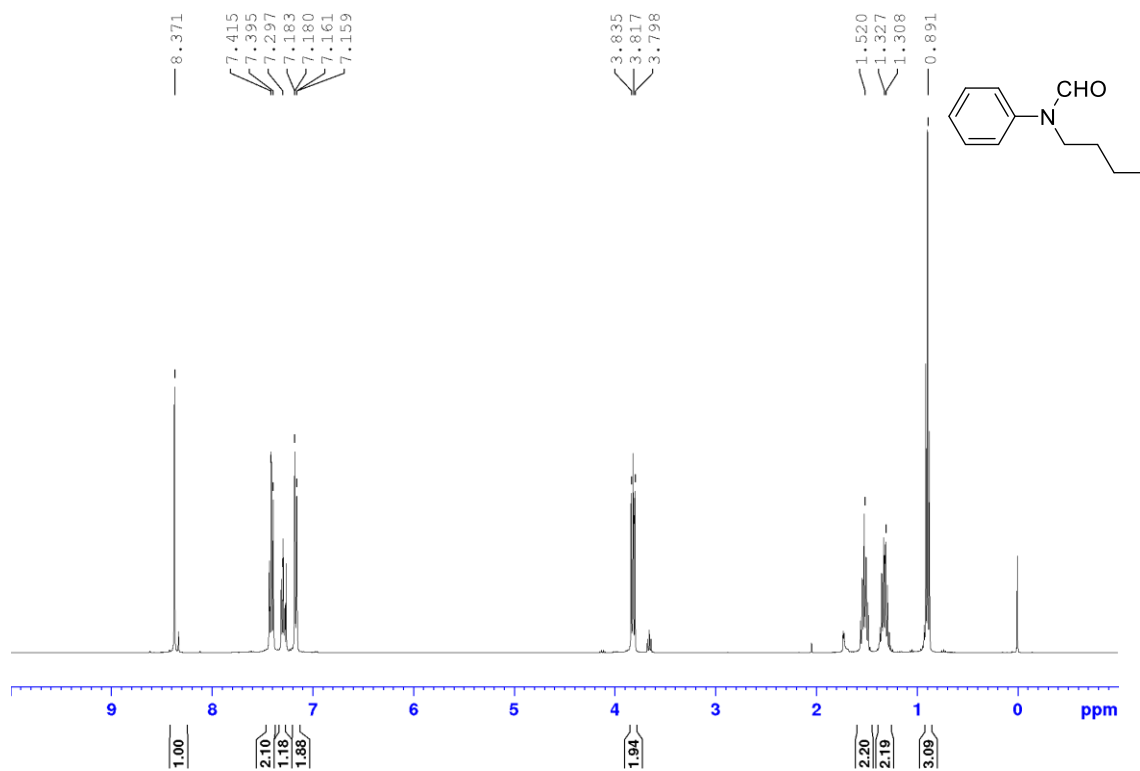




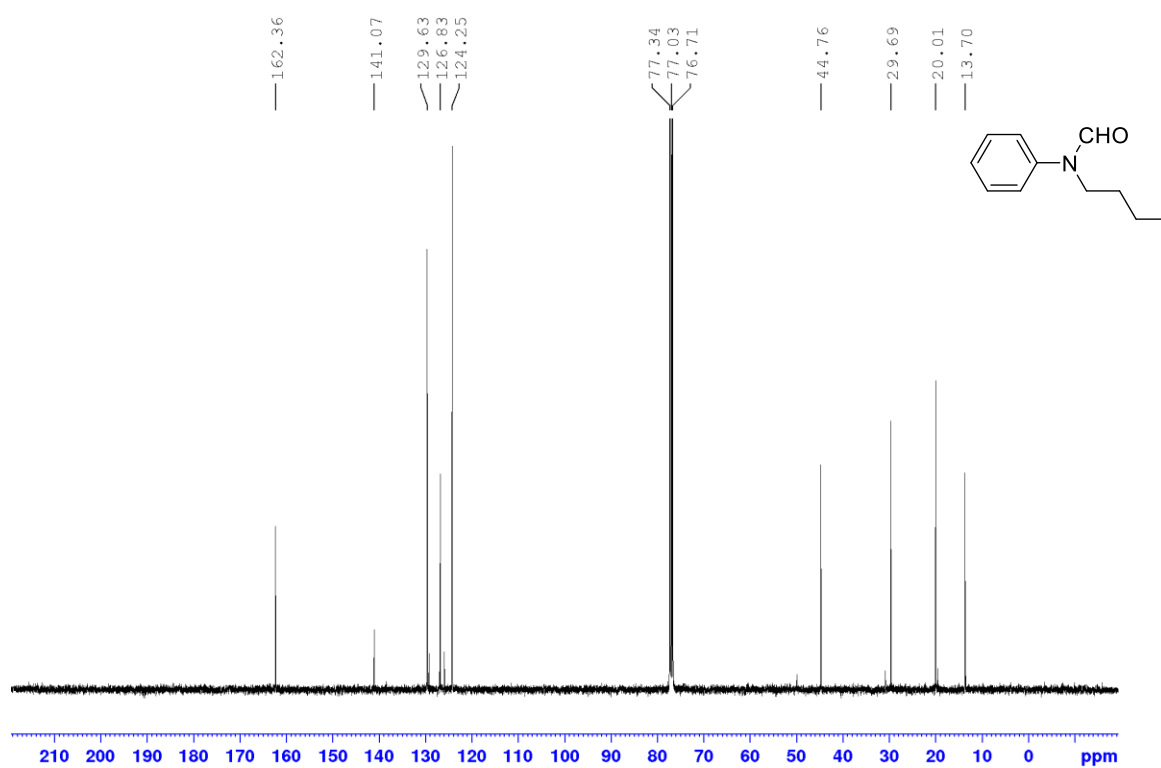




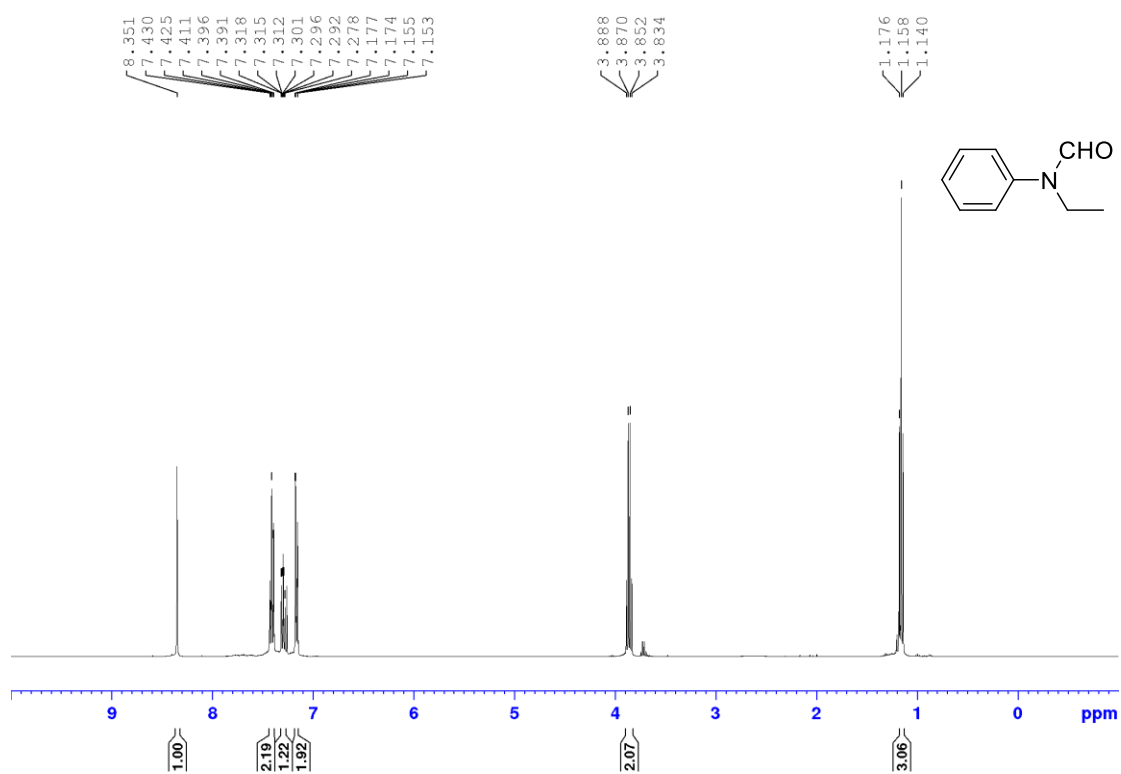




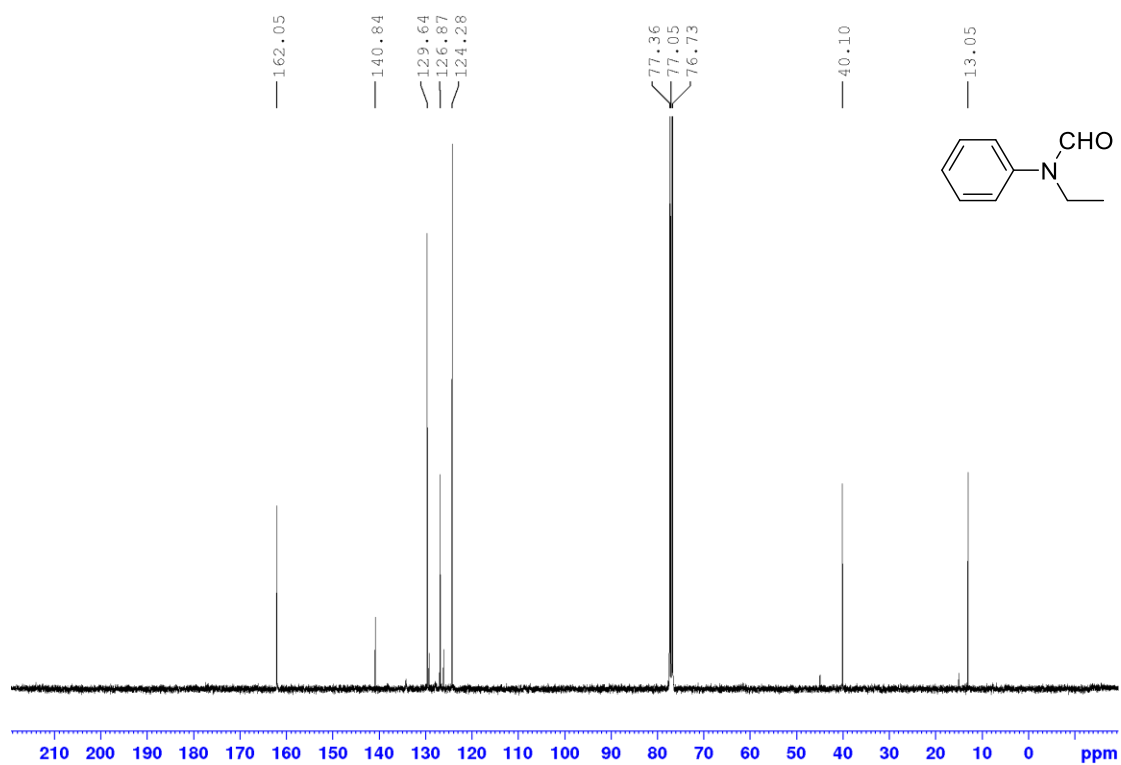
^1H NMR of **8a**. (400 MHz, CDCl_3)



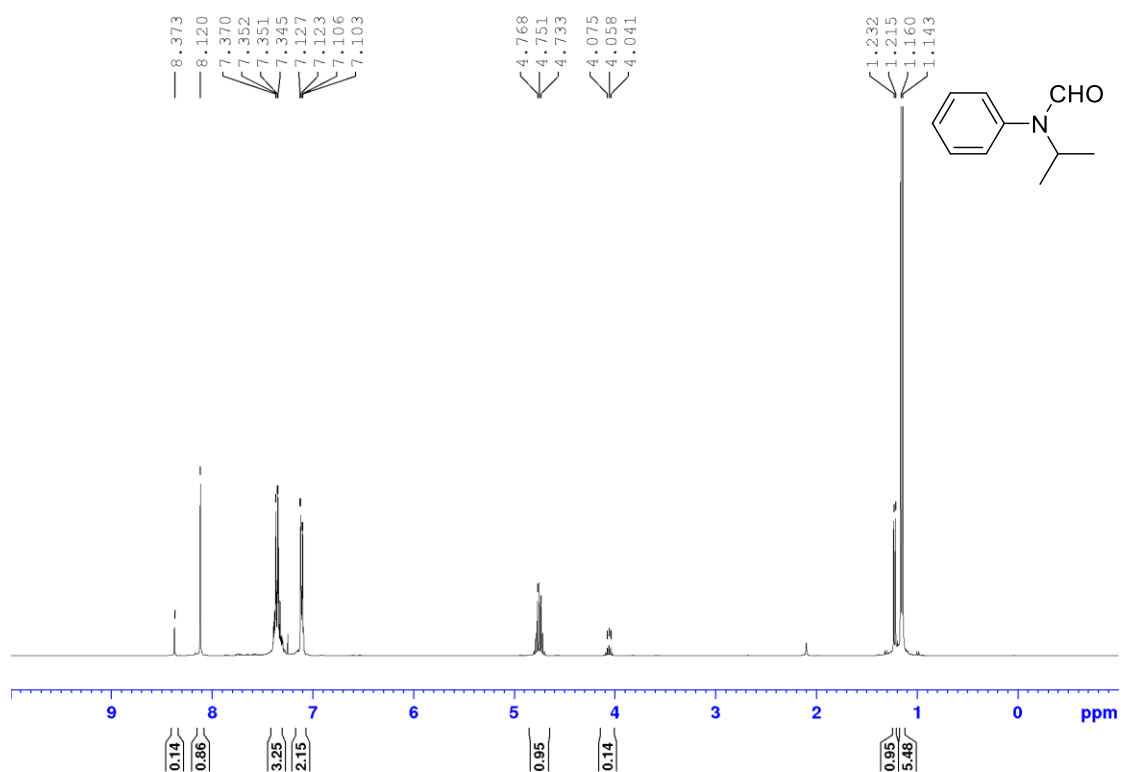
^{13}C $\{^1\text{H}\}$ NMR of **8a**. (100 MHz, CDCl_3)



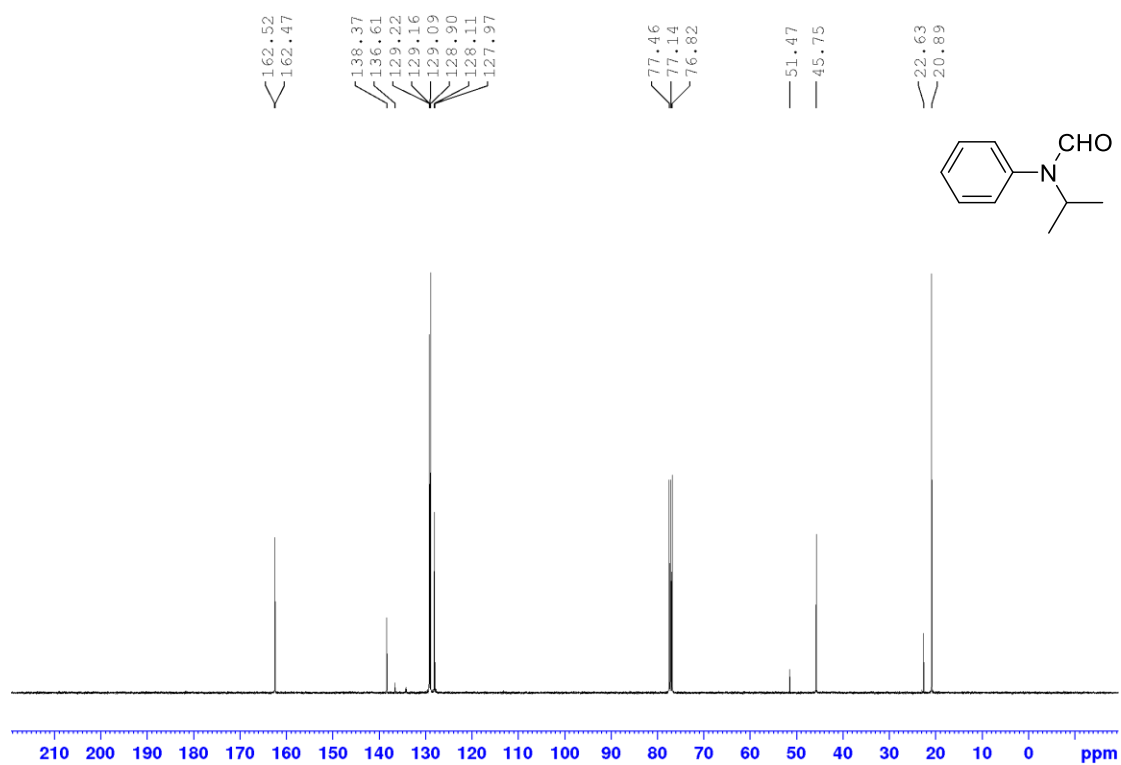
¹H NMR of **9a**. (400 MHz, CDCl₃)



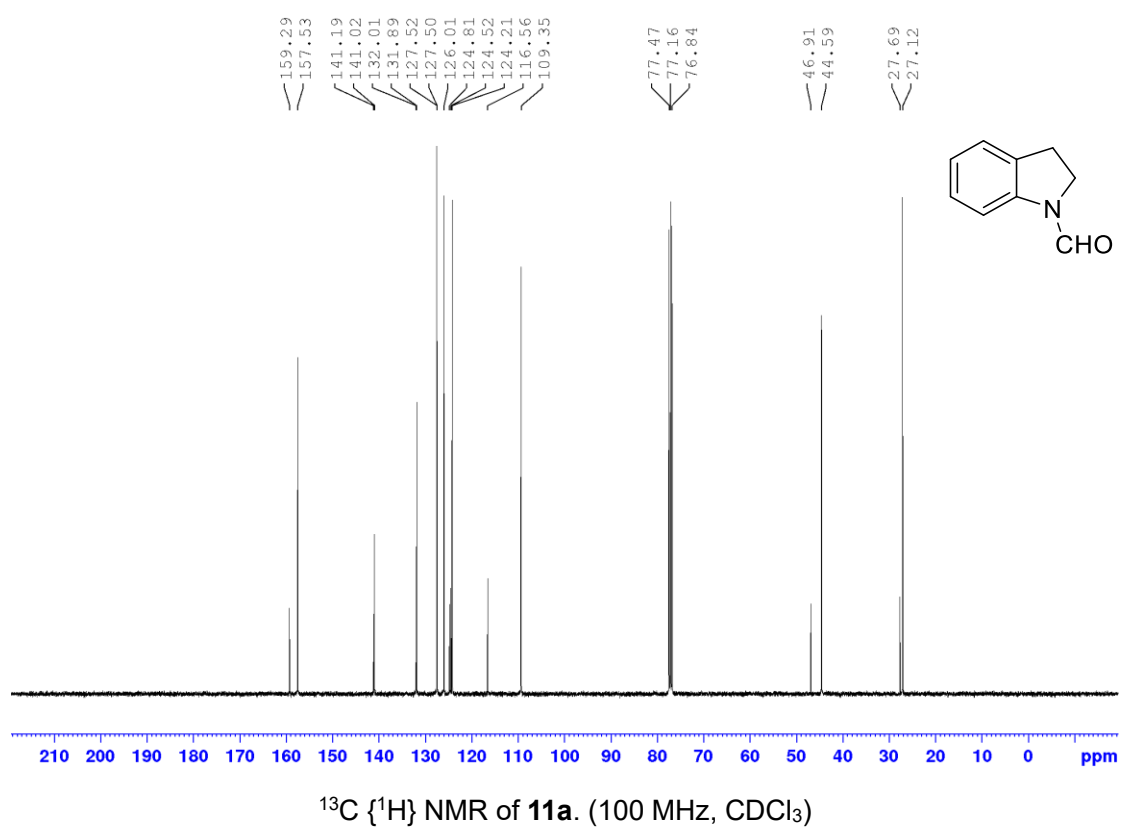
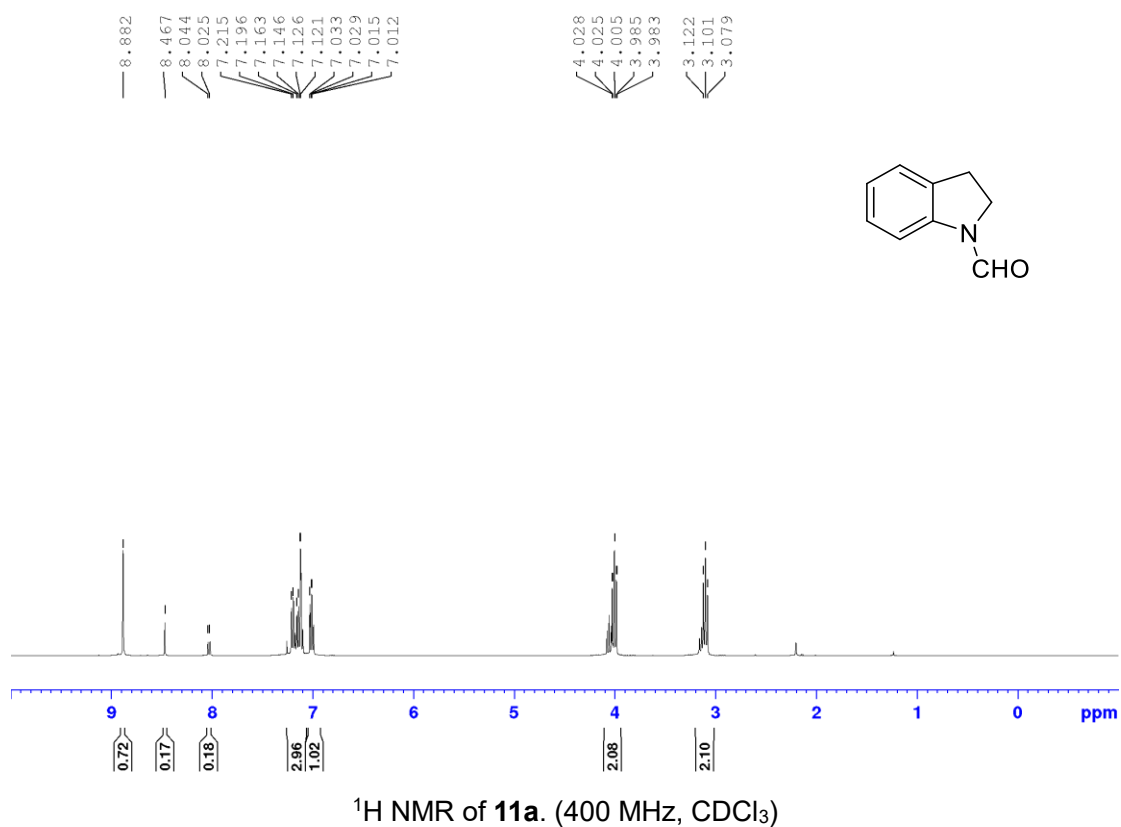
¹³C {¹H} NMR of **9a**. (100 MHz, CDCl₃)

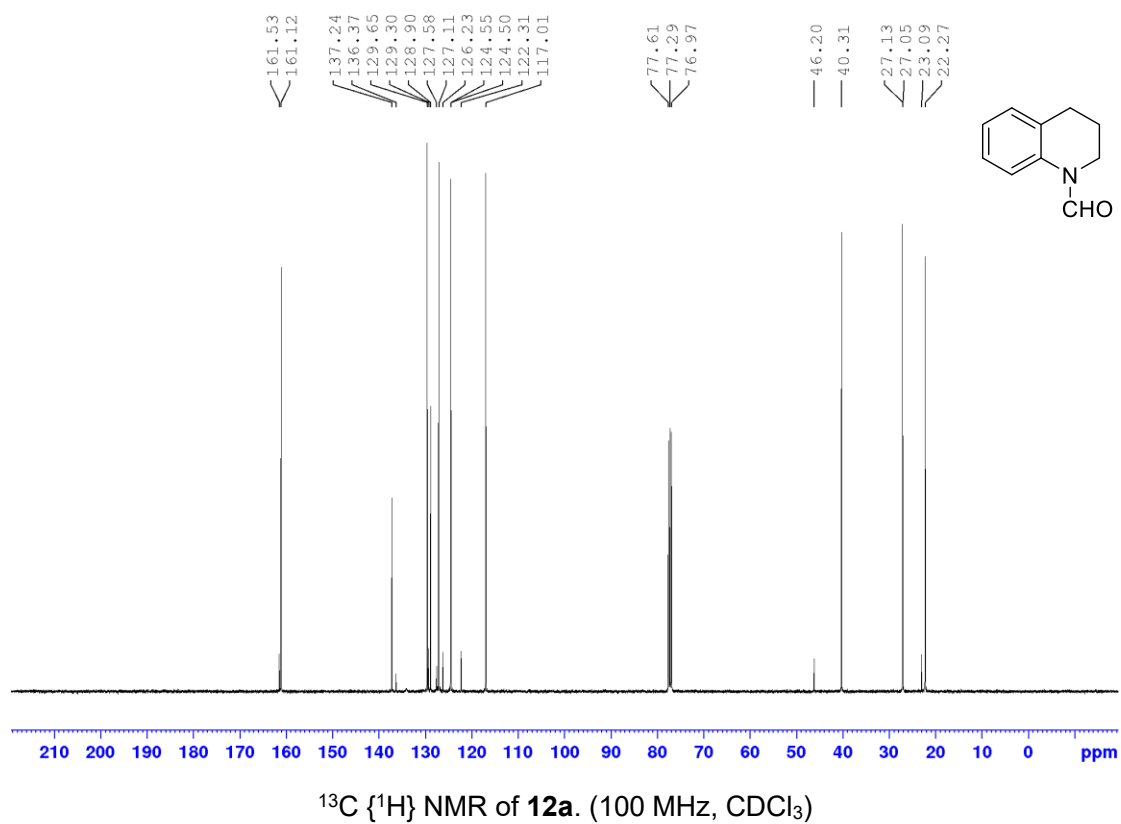
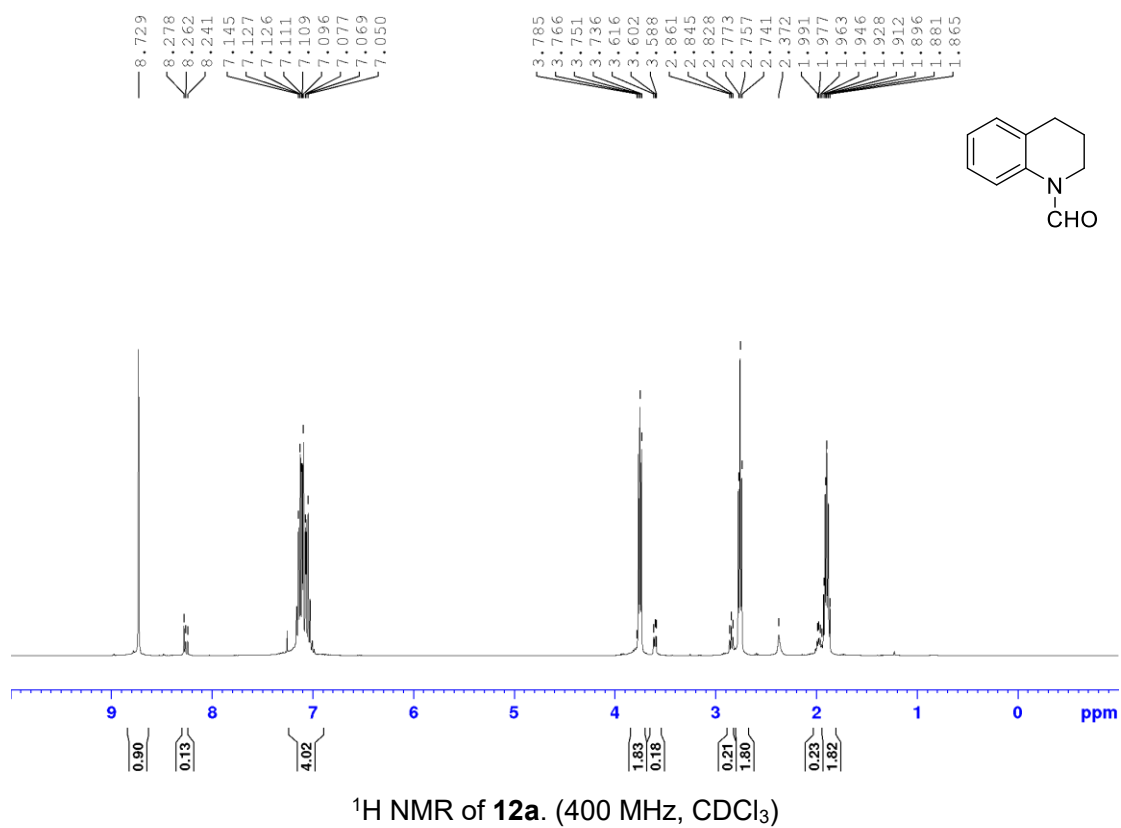


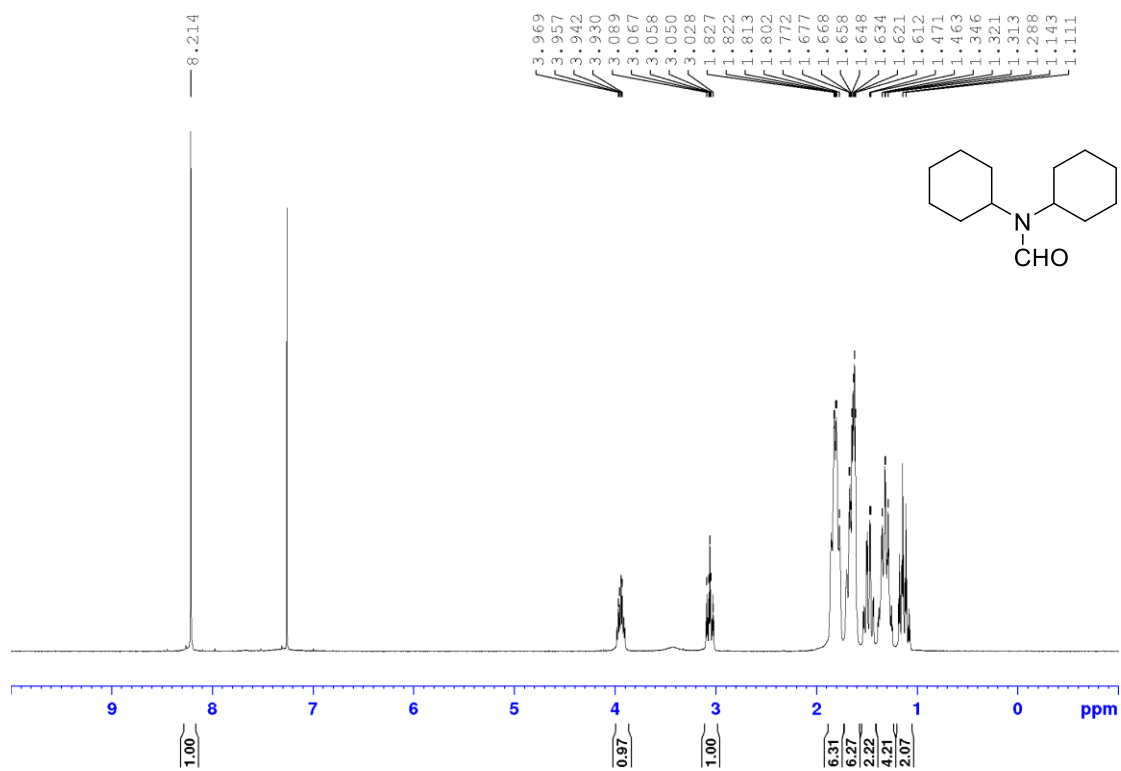
¹H NMR of **10a**. (400 MHz, CDCl₃)



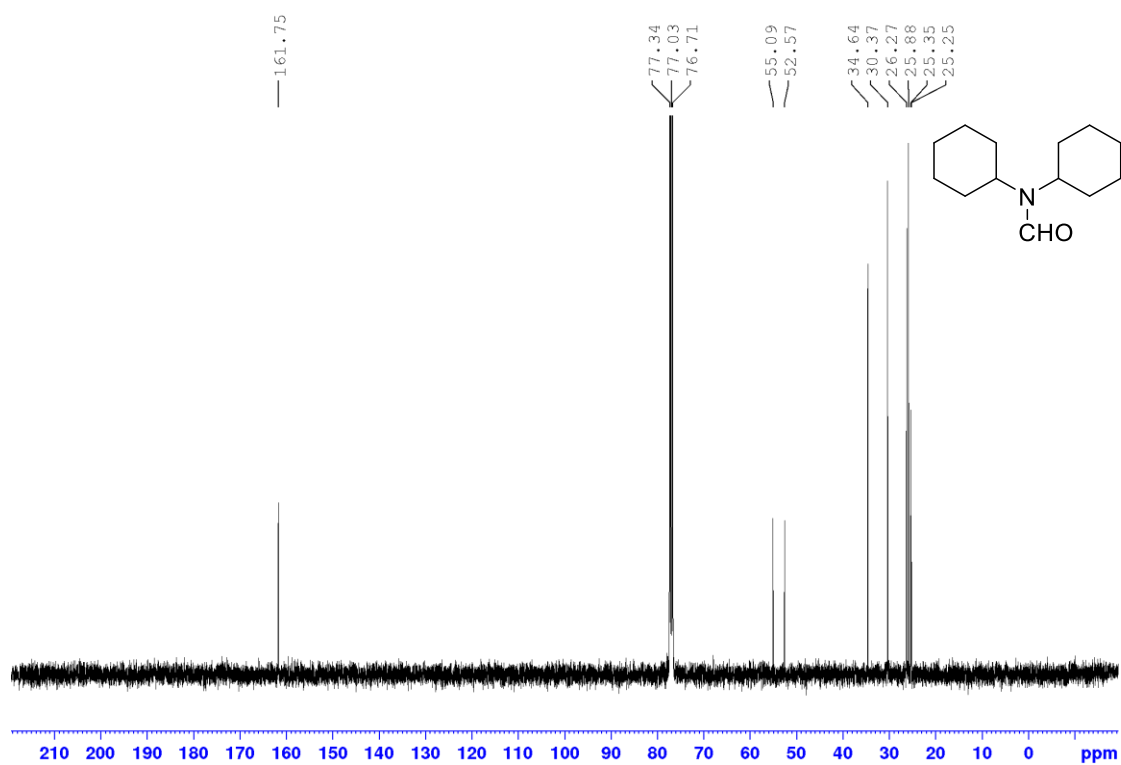
¹³C {¹H} NMR of **10a**. (100 MHz, CDCl₃)



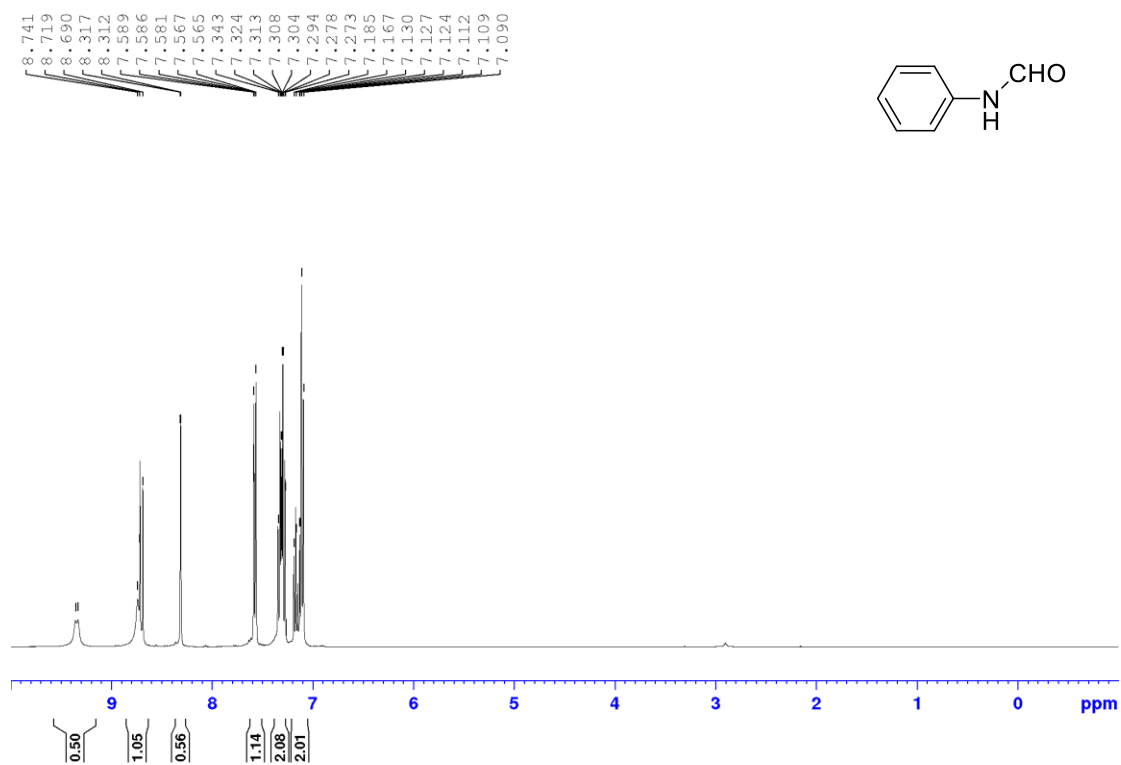




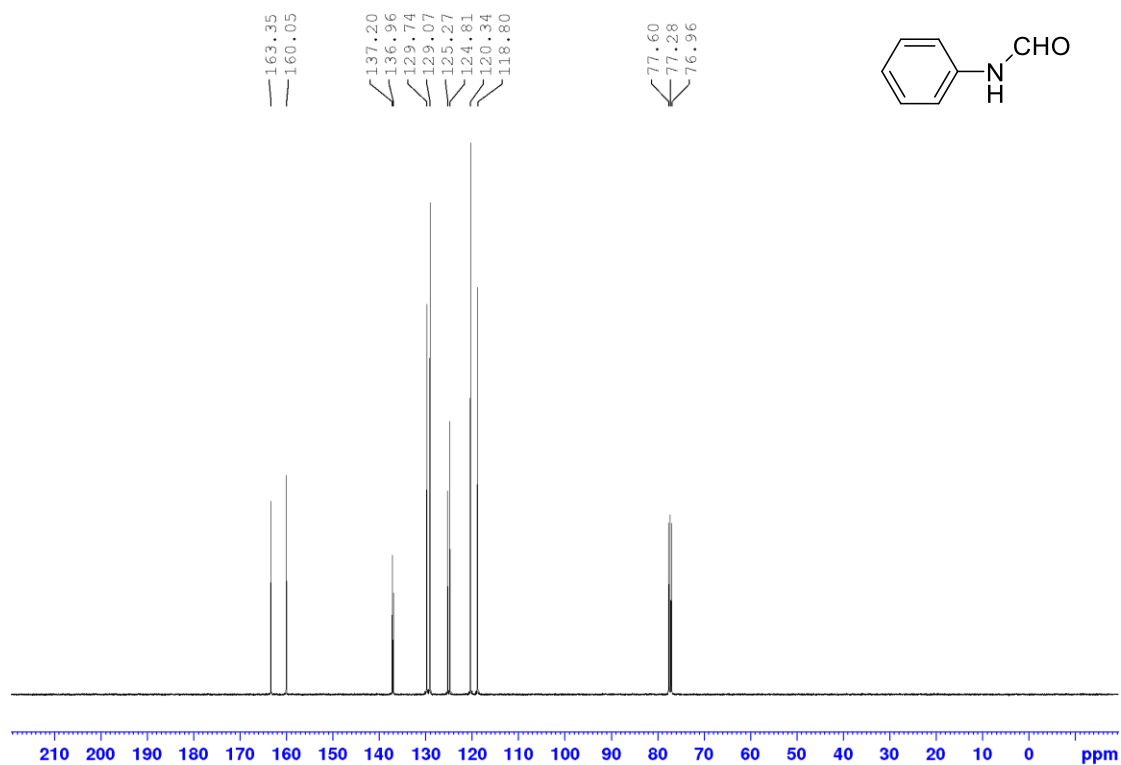
¹H NMR of **13a**. (400 MHz, CDCl₃)



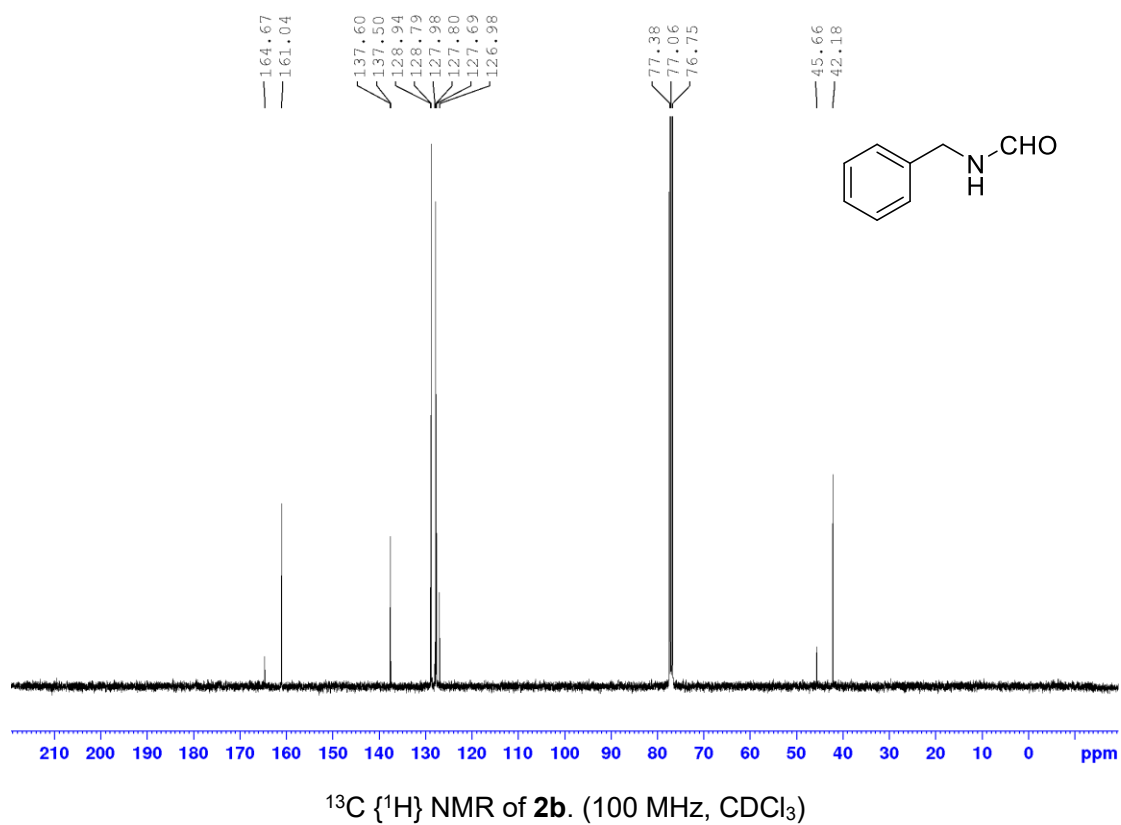
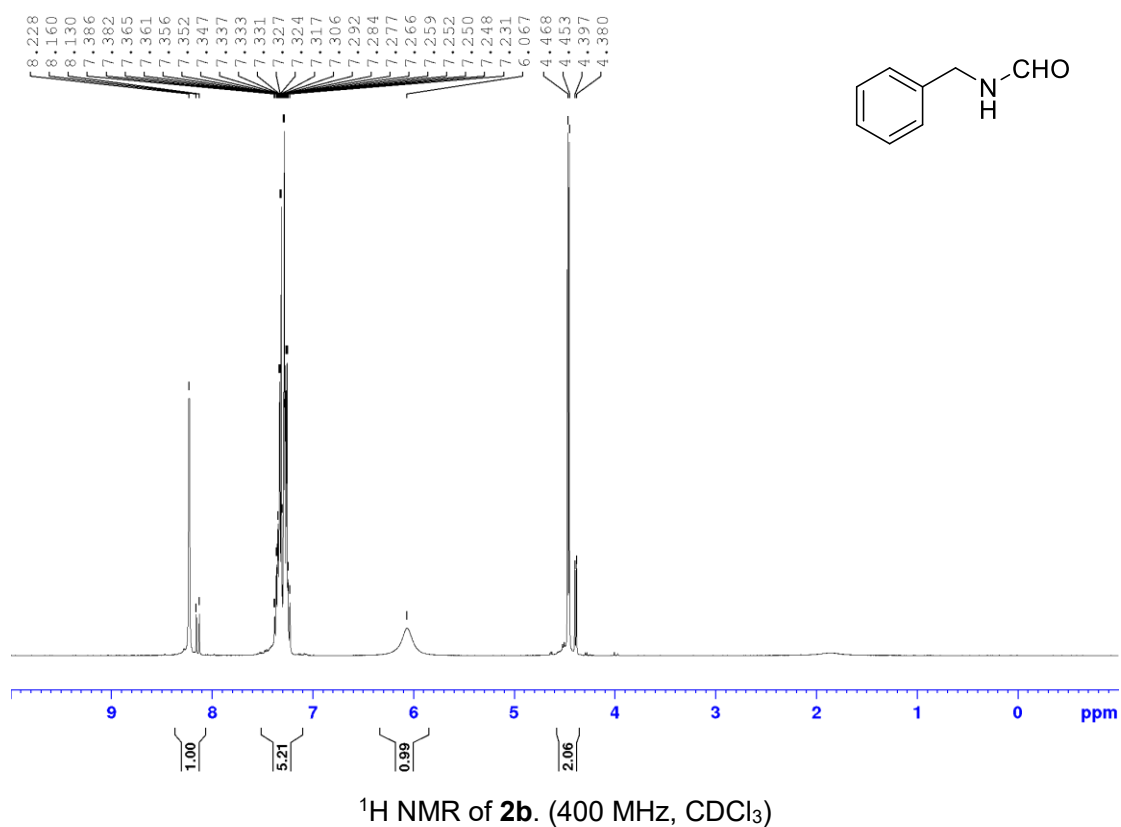
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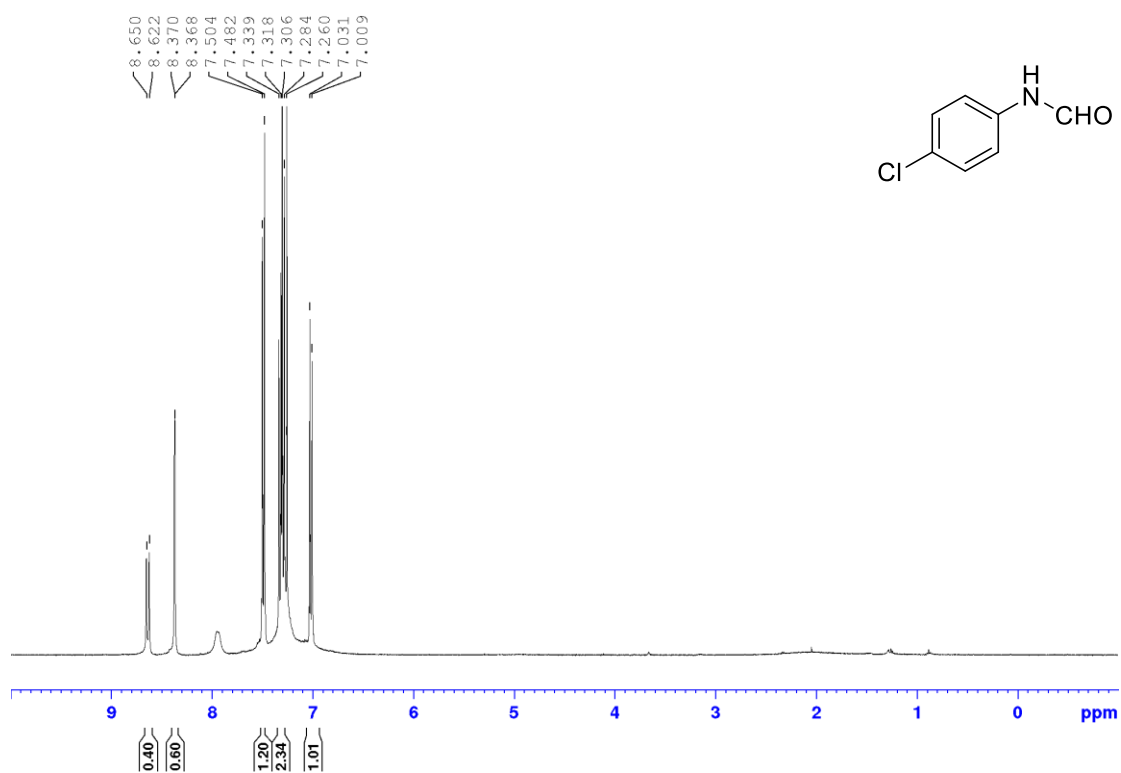


¹H NMR of **1b**. (400 MHz, CDCl₃)

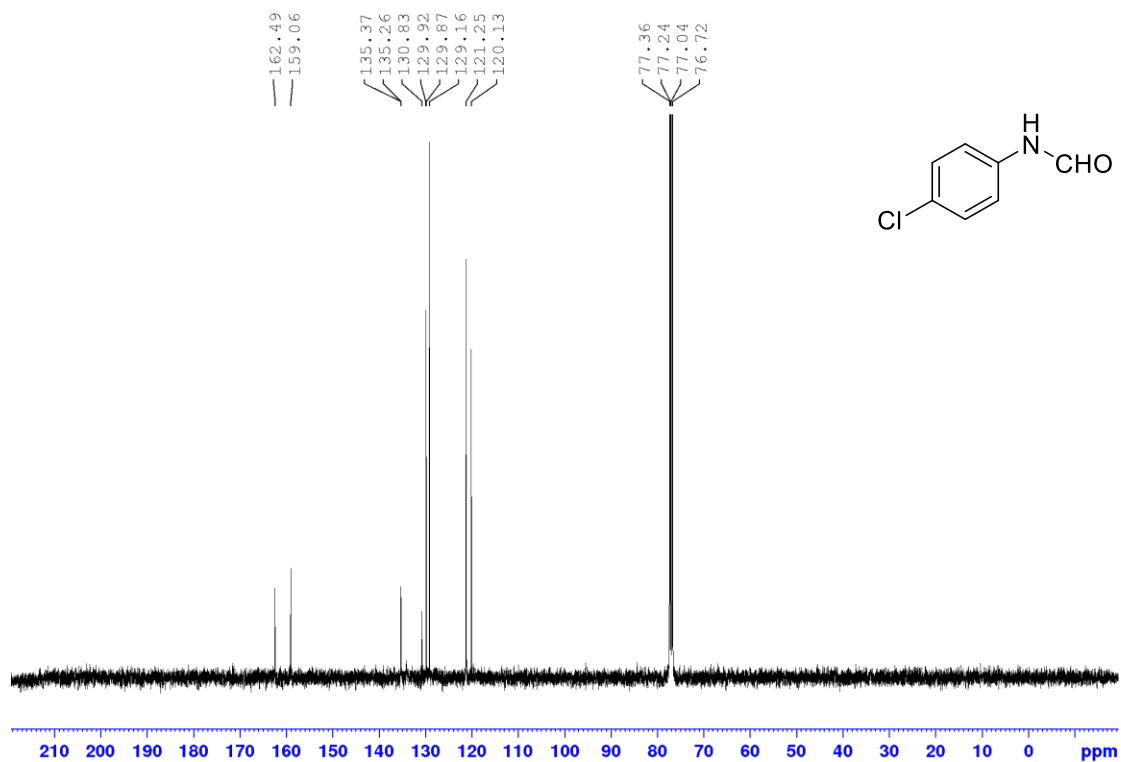


¹³C {¹H} NMR of **1b**. (100 MHz, CDCl₃)

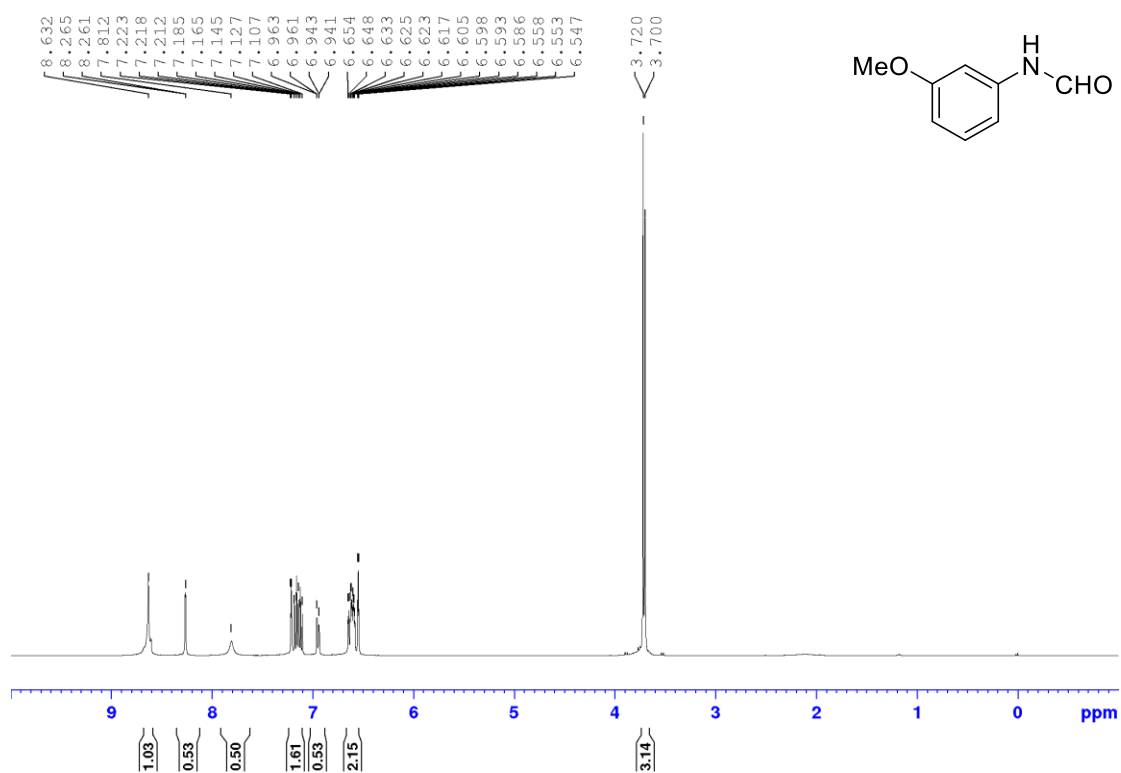




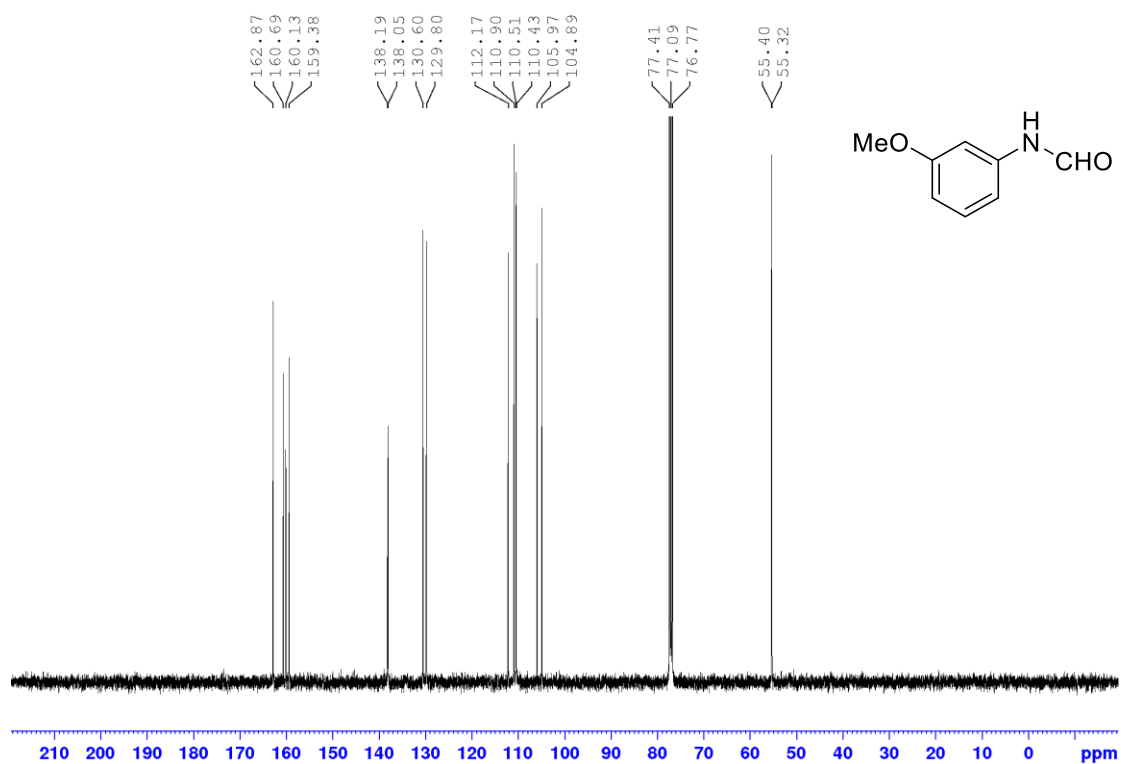
¹H NMR of **3b**. (400 MHz, CDCl₃)



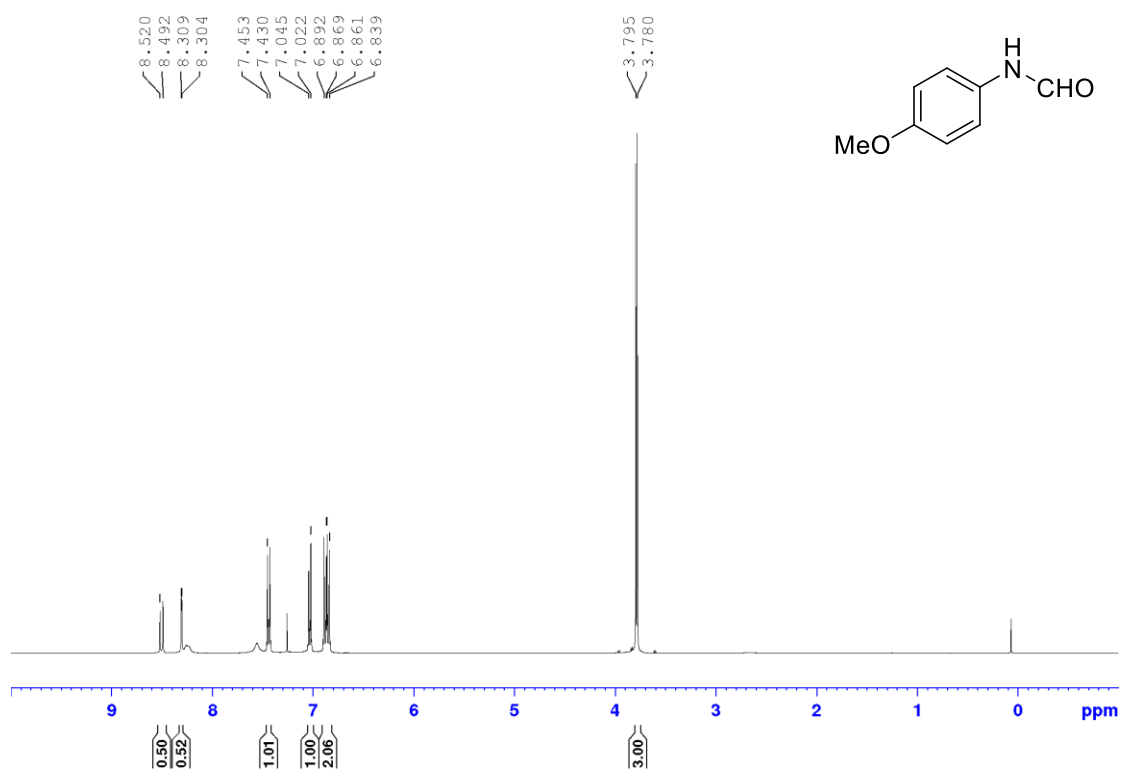
¹³C {¹H} NMR of **3b**. (100 MHz, CDCl₃)



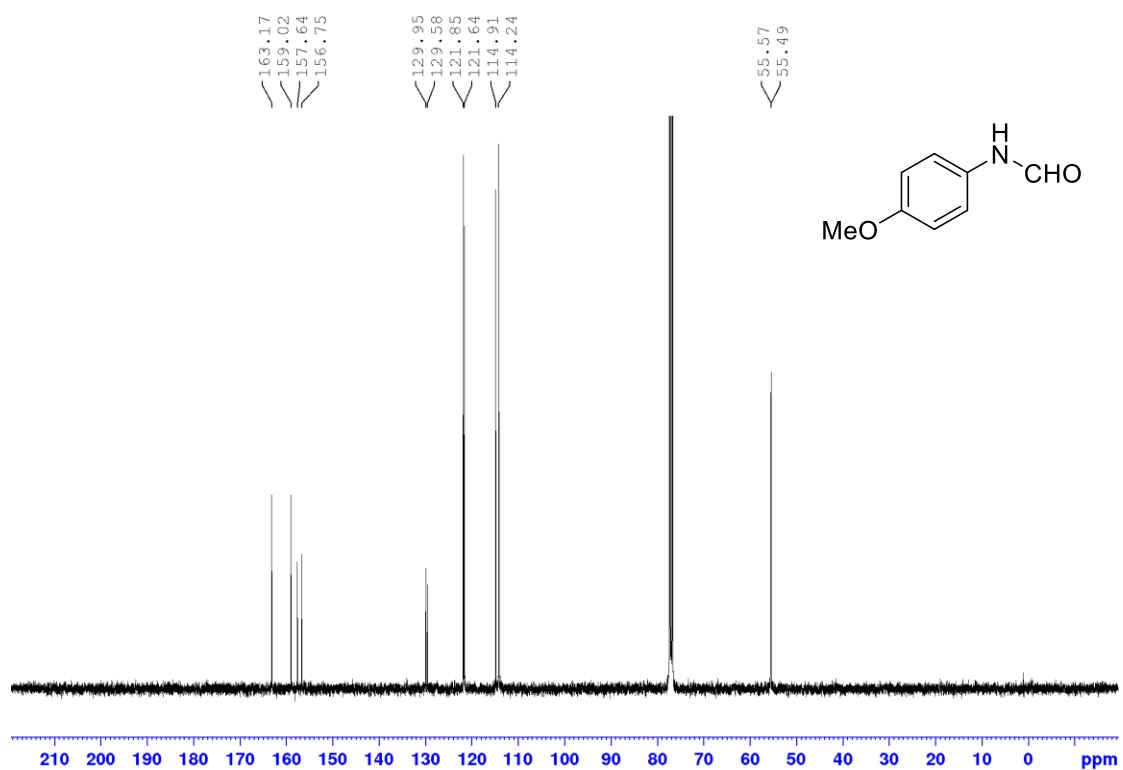
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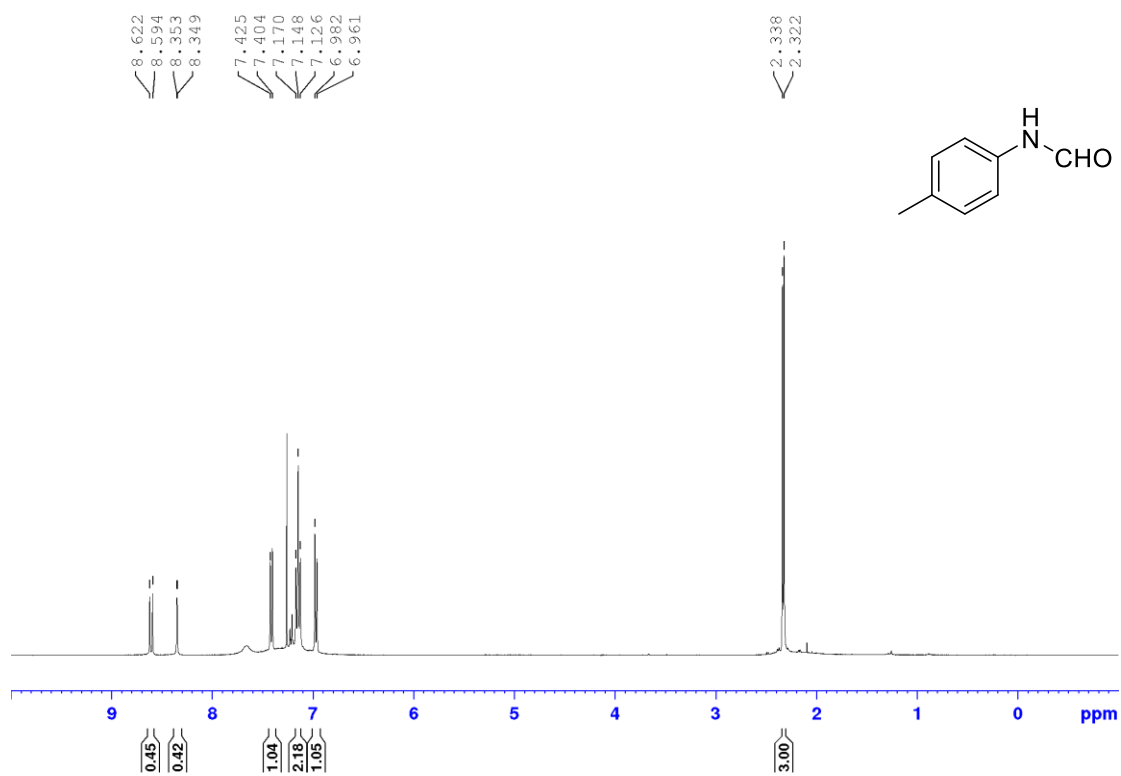
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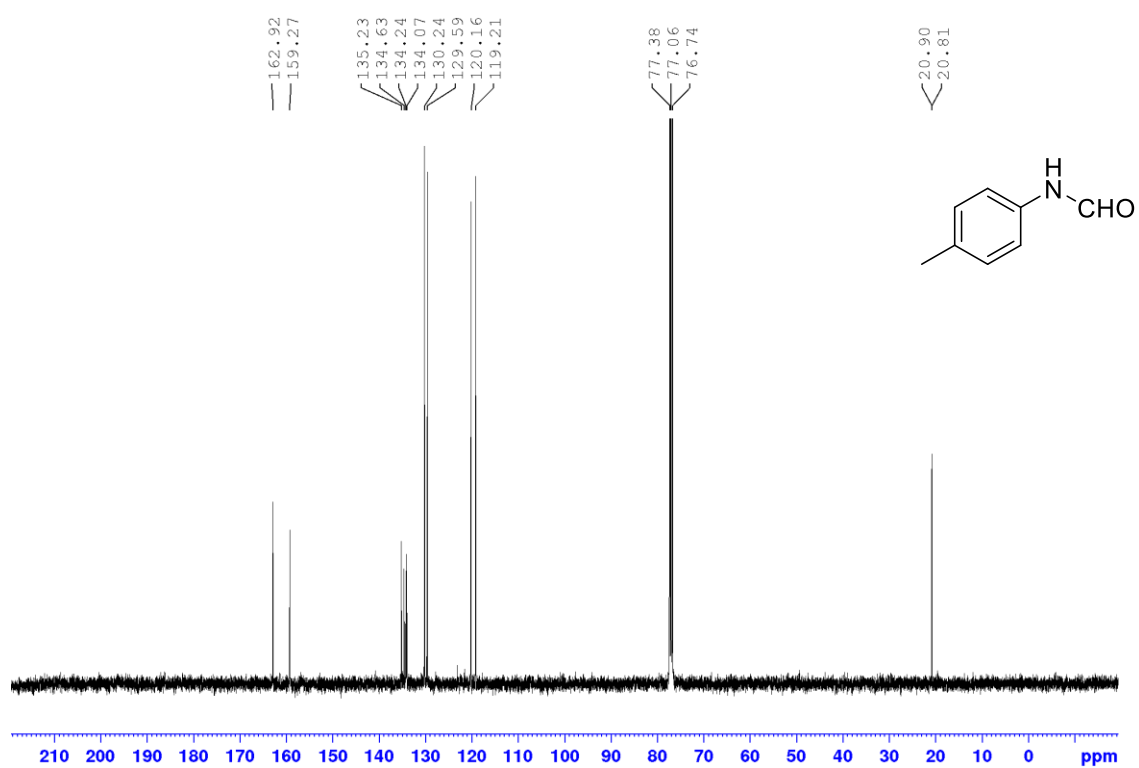
¹H NMR of **5b**. (400 MHz, CDCl₃)



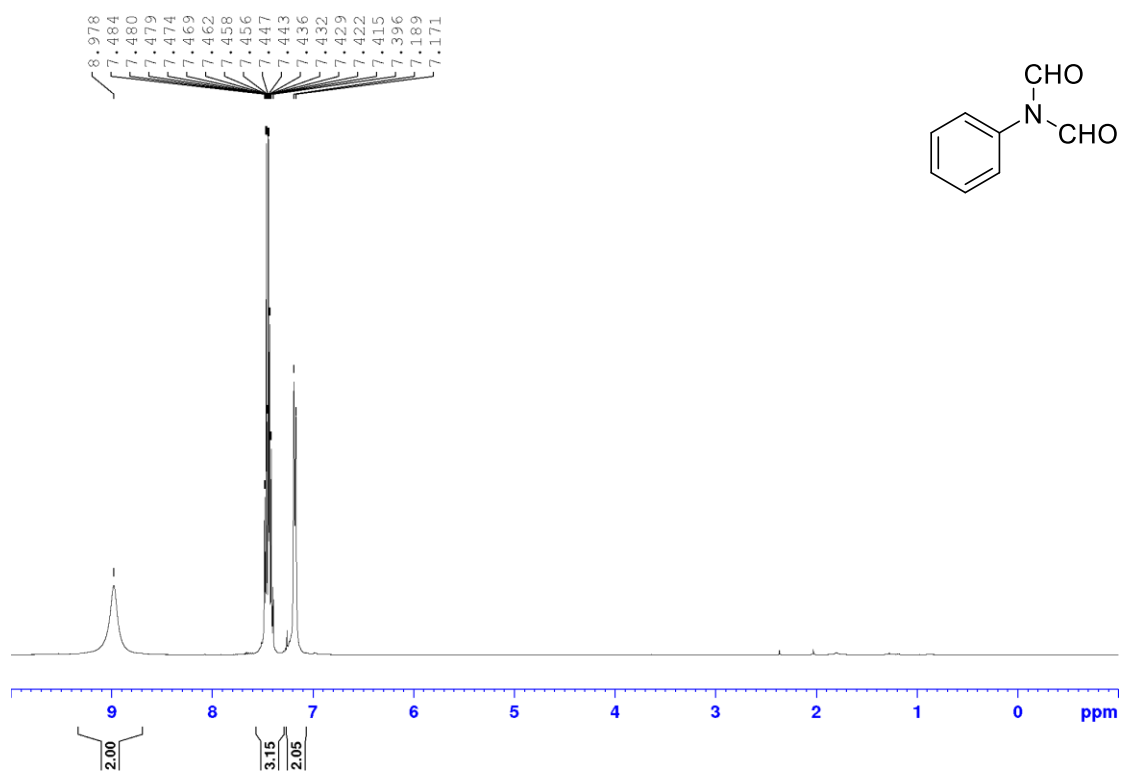
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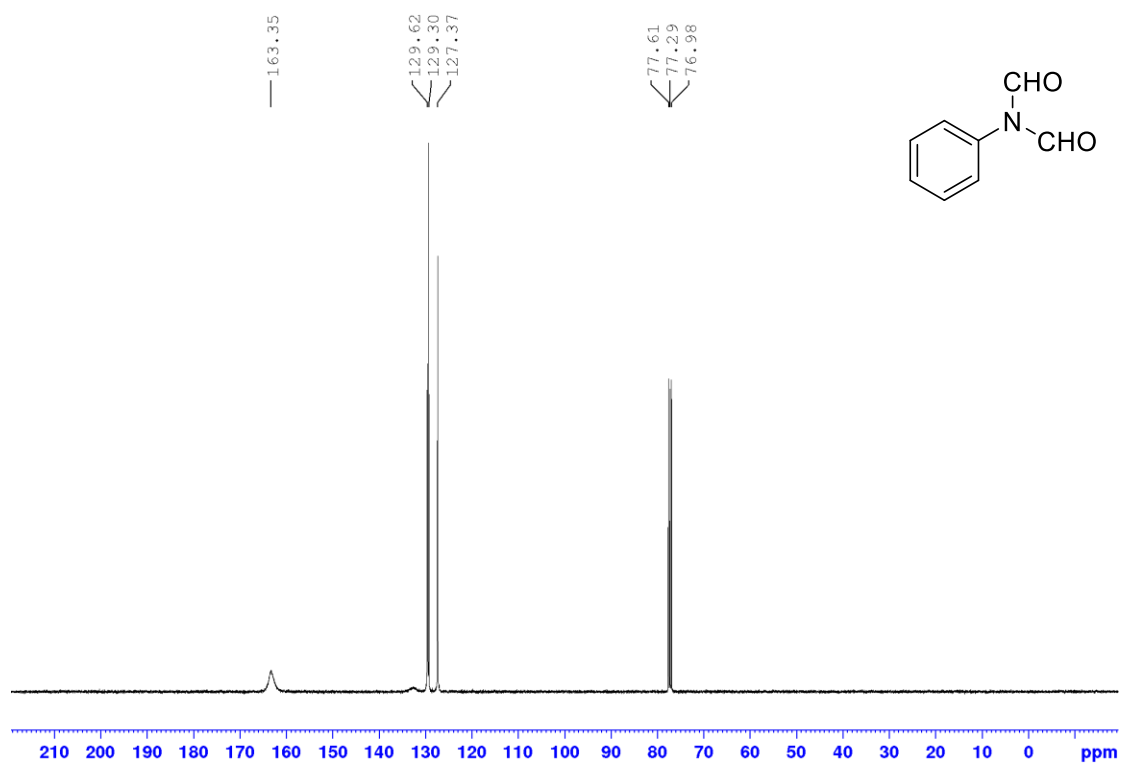
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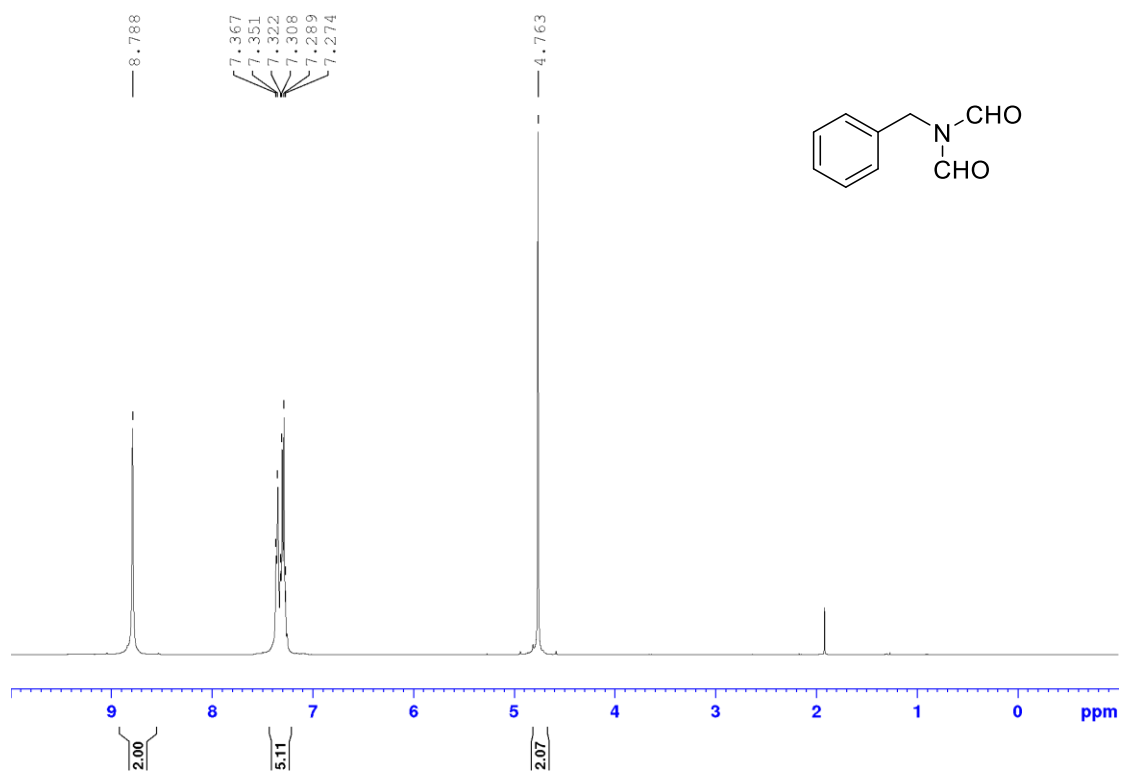
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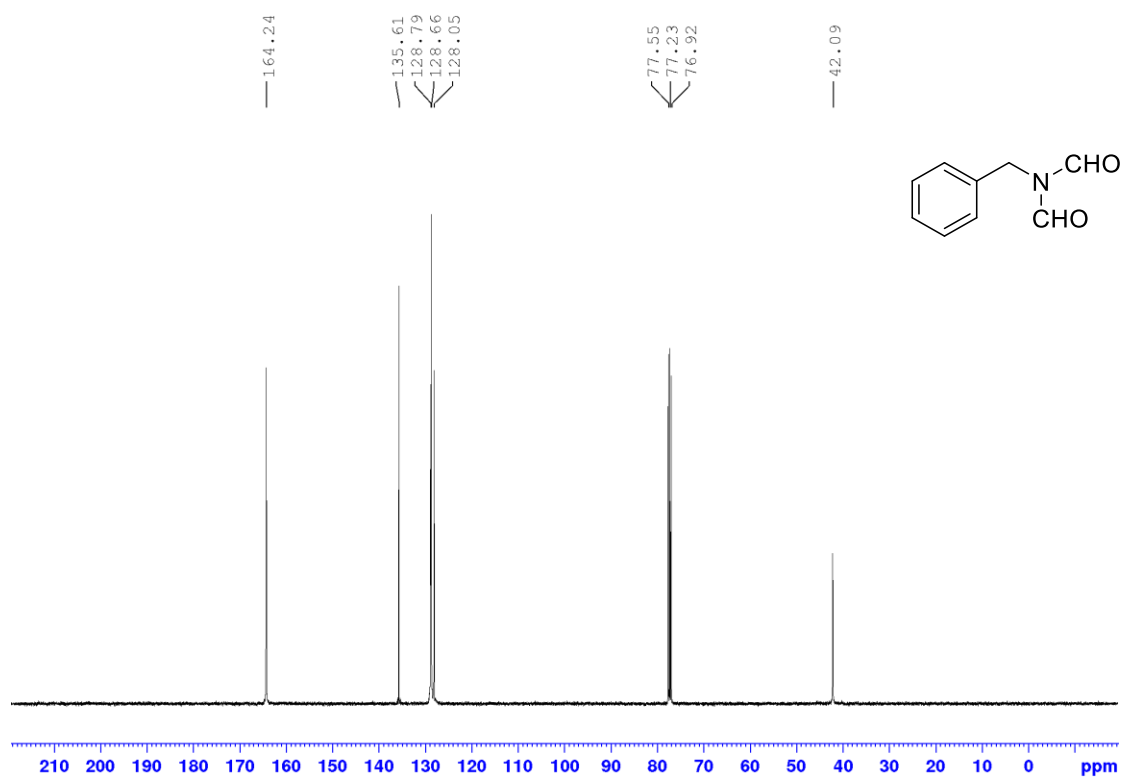
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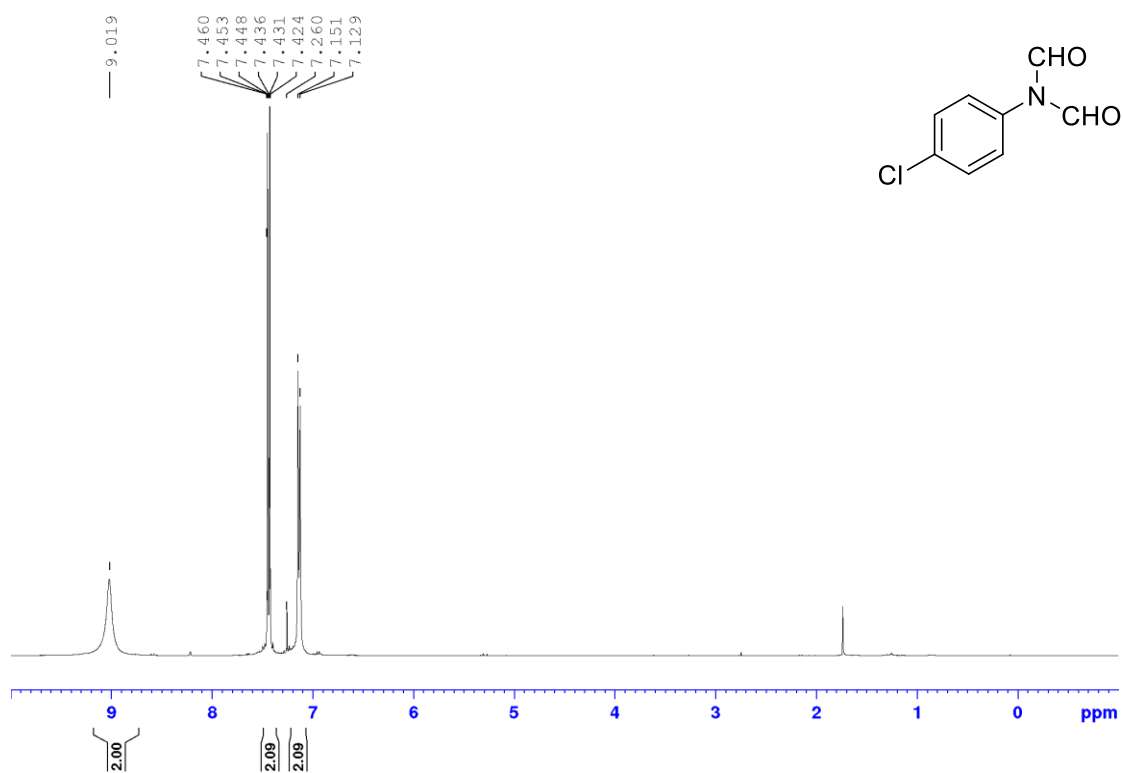
¹³C {¹H} NMR of **1c**. (100 MHz, CDCl₃)



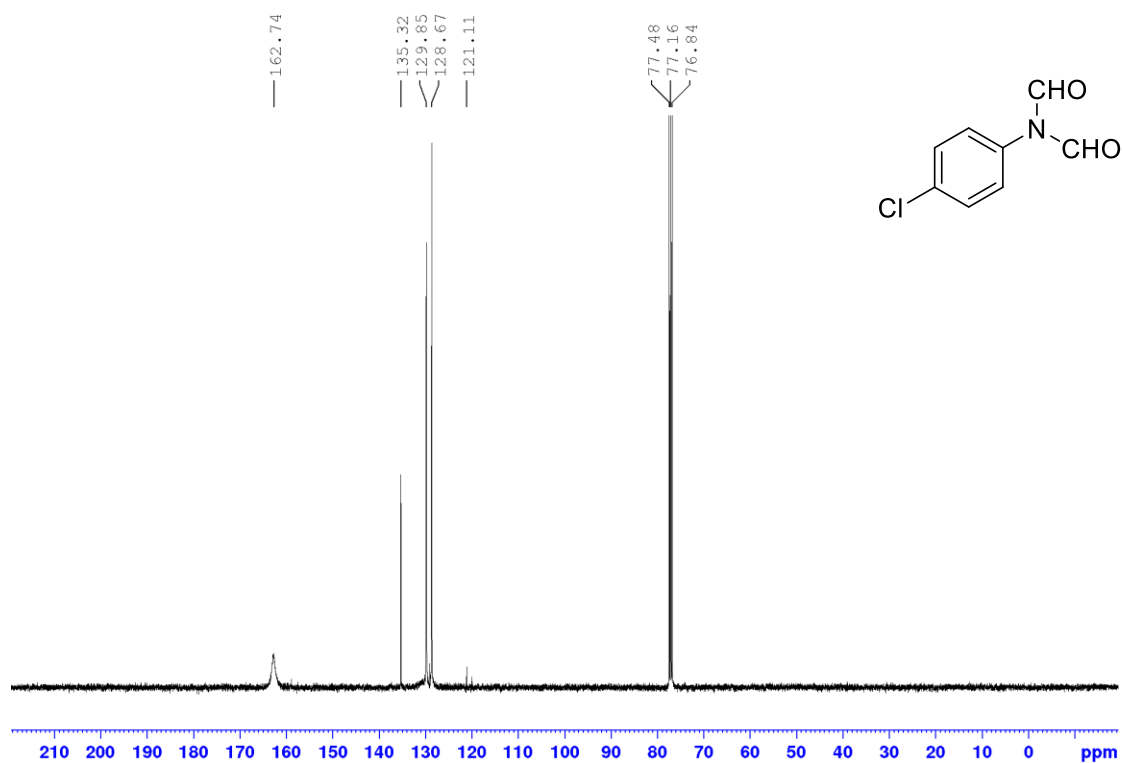
¹H NMR of **2c**. (400 MHz, CDCl₃)



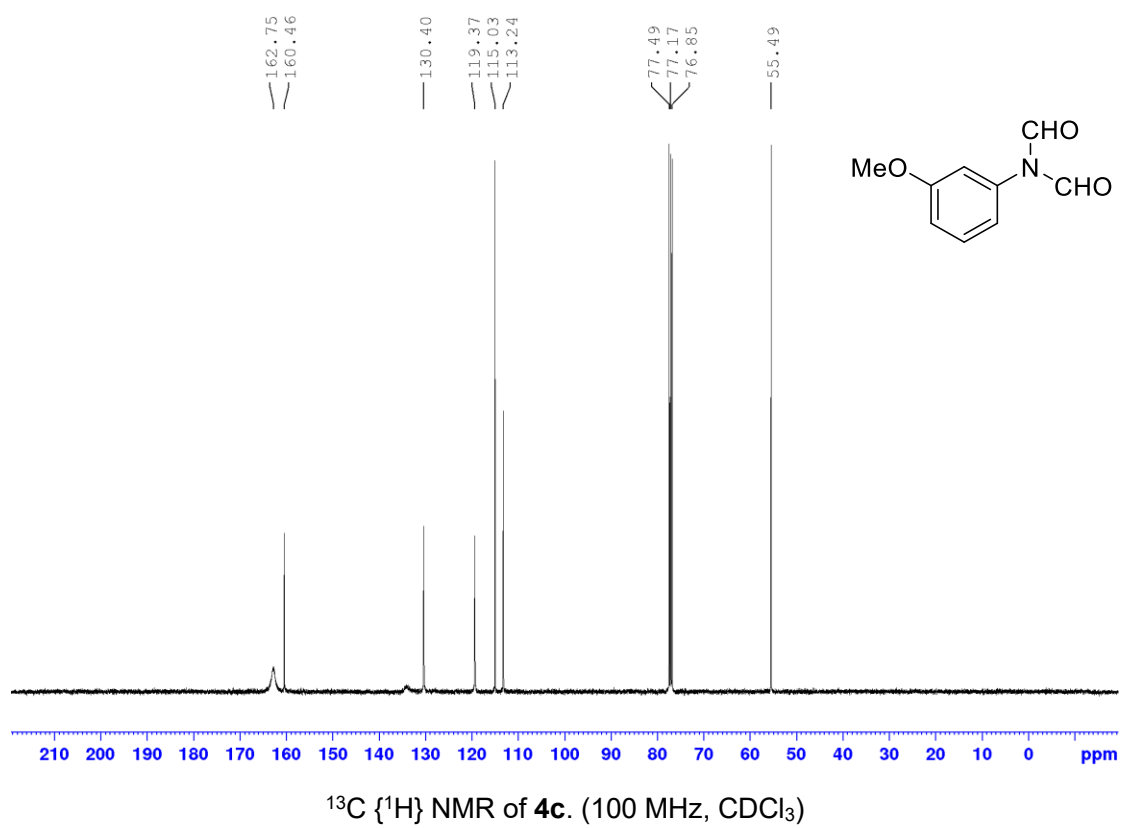
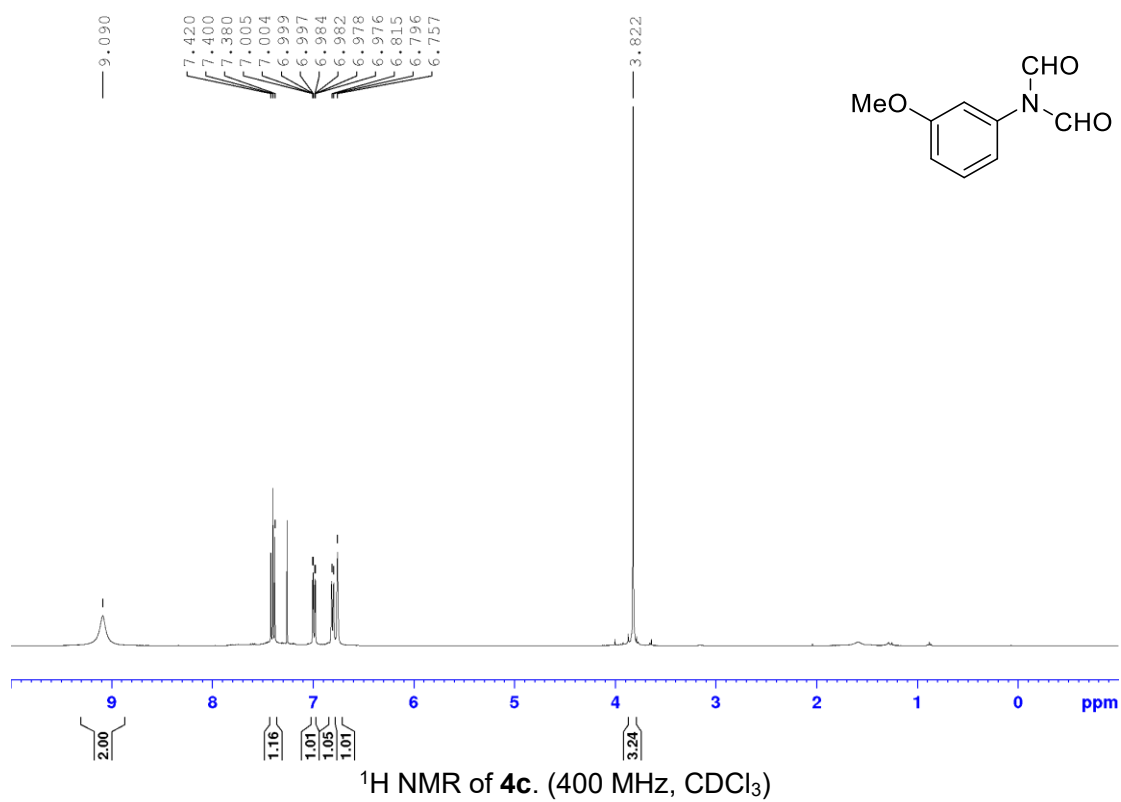
¹³C {¹H} NMR of **2c**. (100 MHz, CDCl₃)

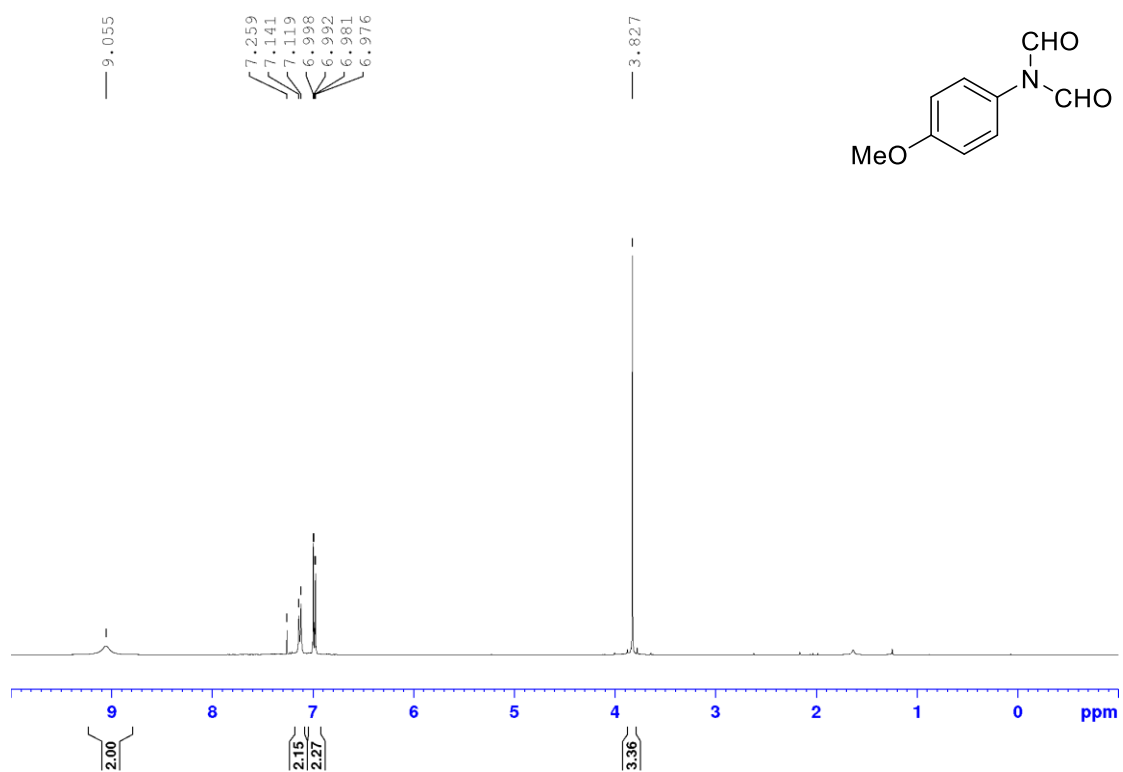


¹H NMR of **3c**. (400 MHz, CDCl₃)

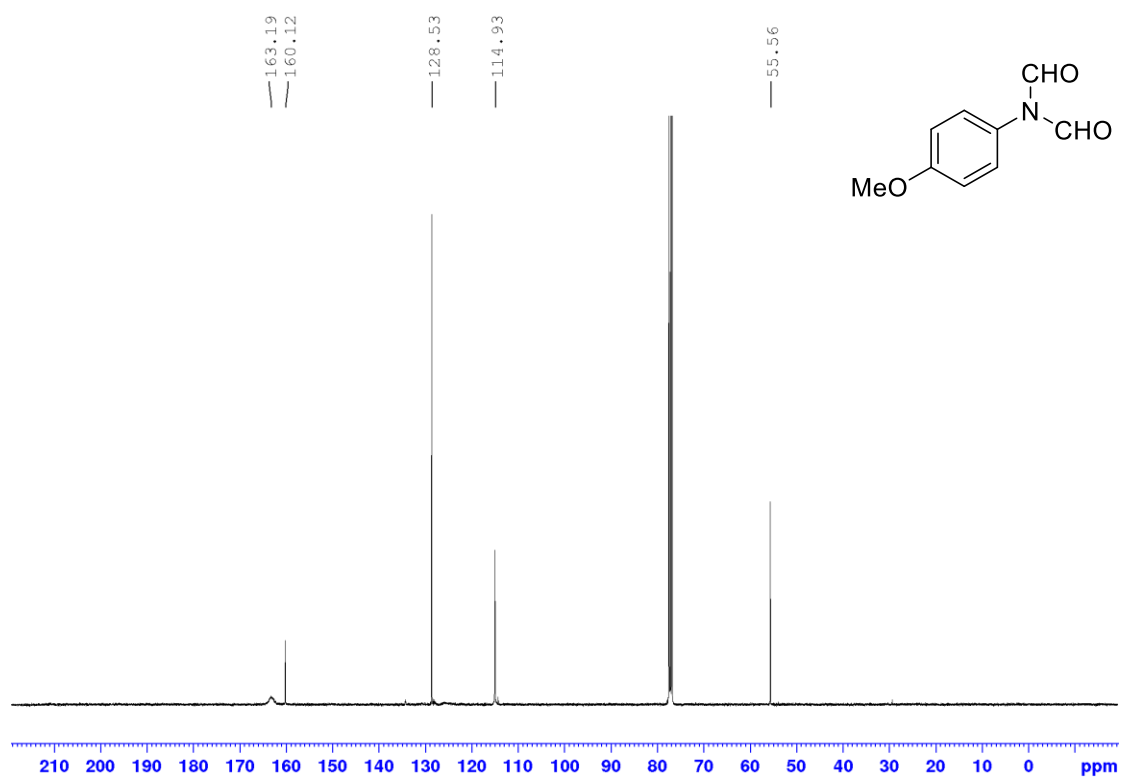


¹³C {¹H} NMR of **3c**. (100 MHz, CDCl₃)

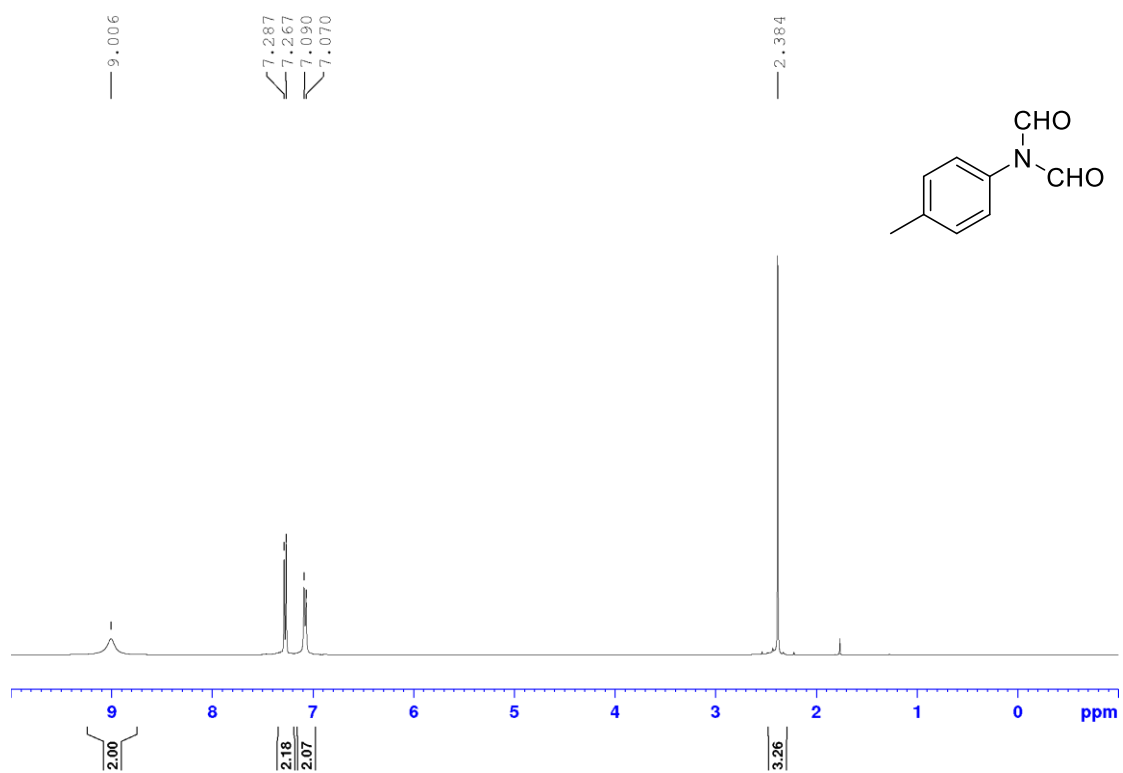




^1H NMR of **5c**. (400 MHz, CDCl_3)



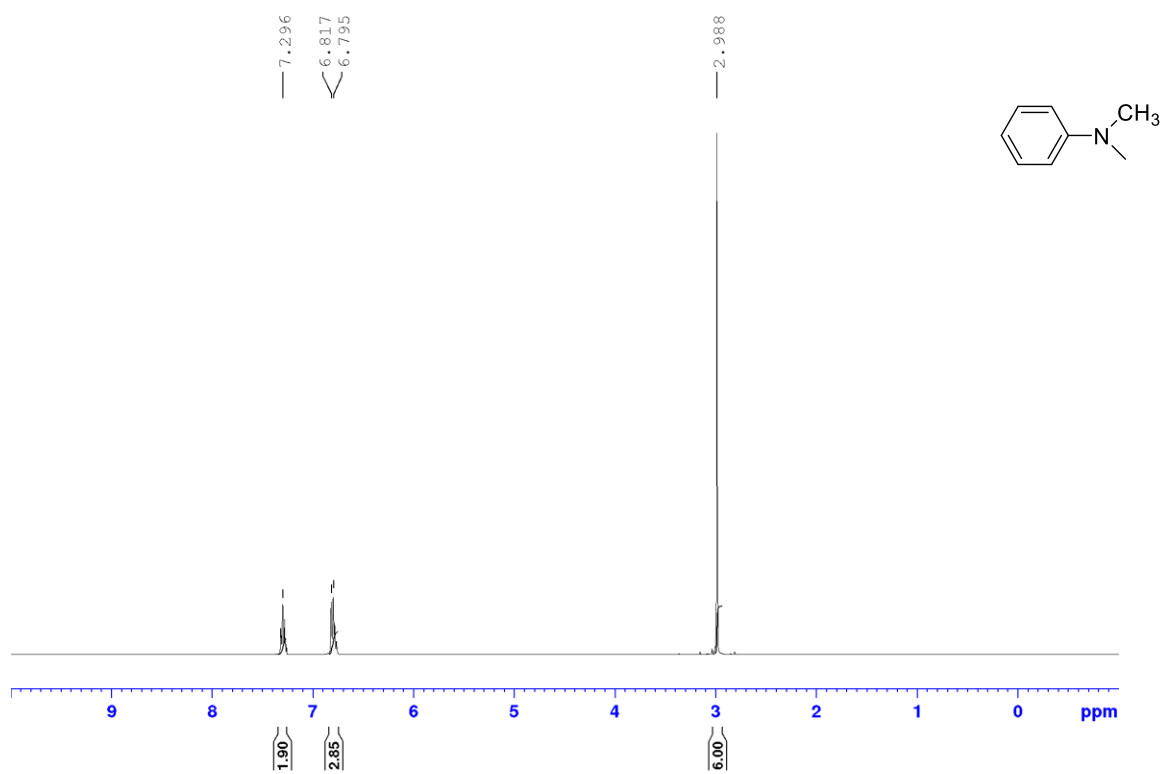
^{13}C $\{^1\text{H}\}$ NMR of **5c**. (100 MHz, CDCl_3)



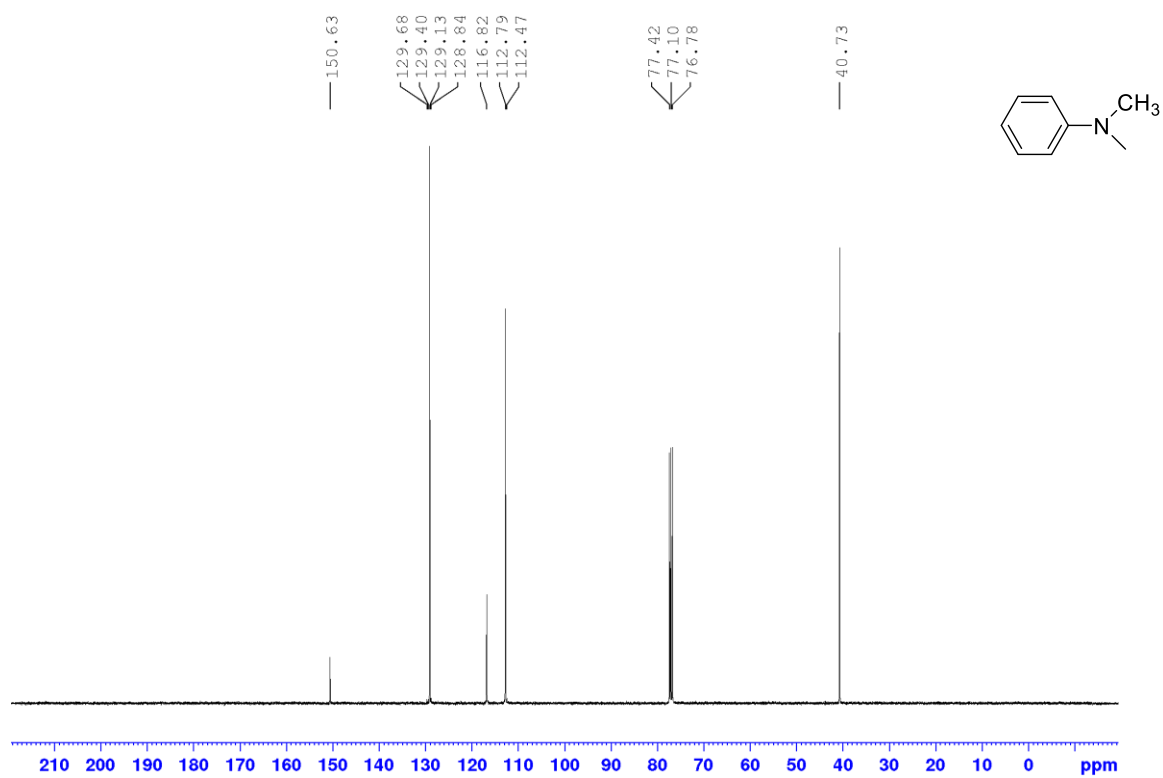
¹H NMR of **6c**. (400 MHz, CDCl₃)



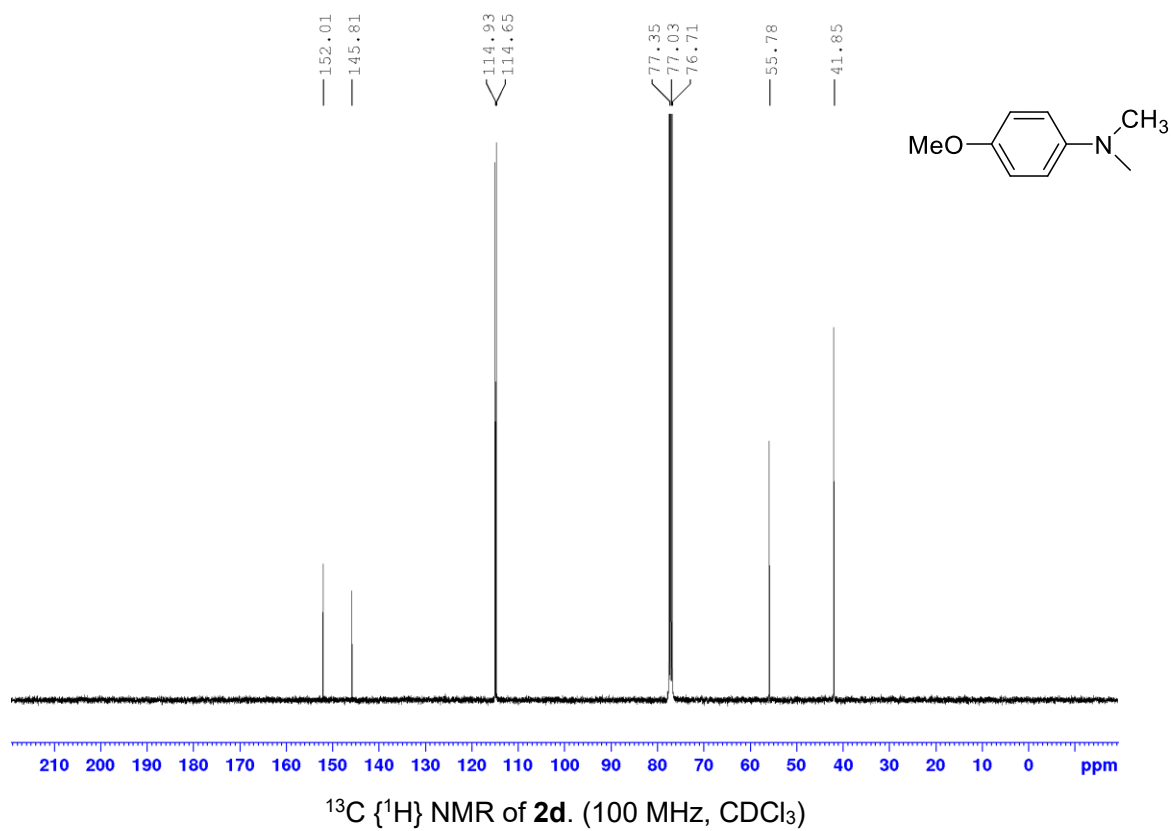
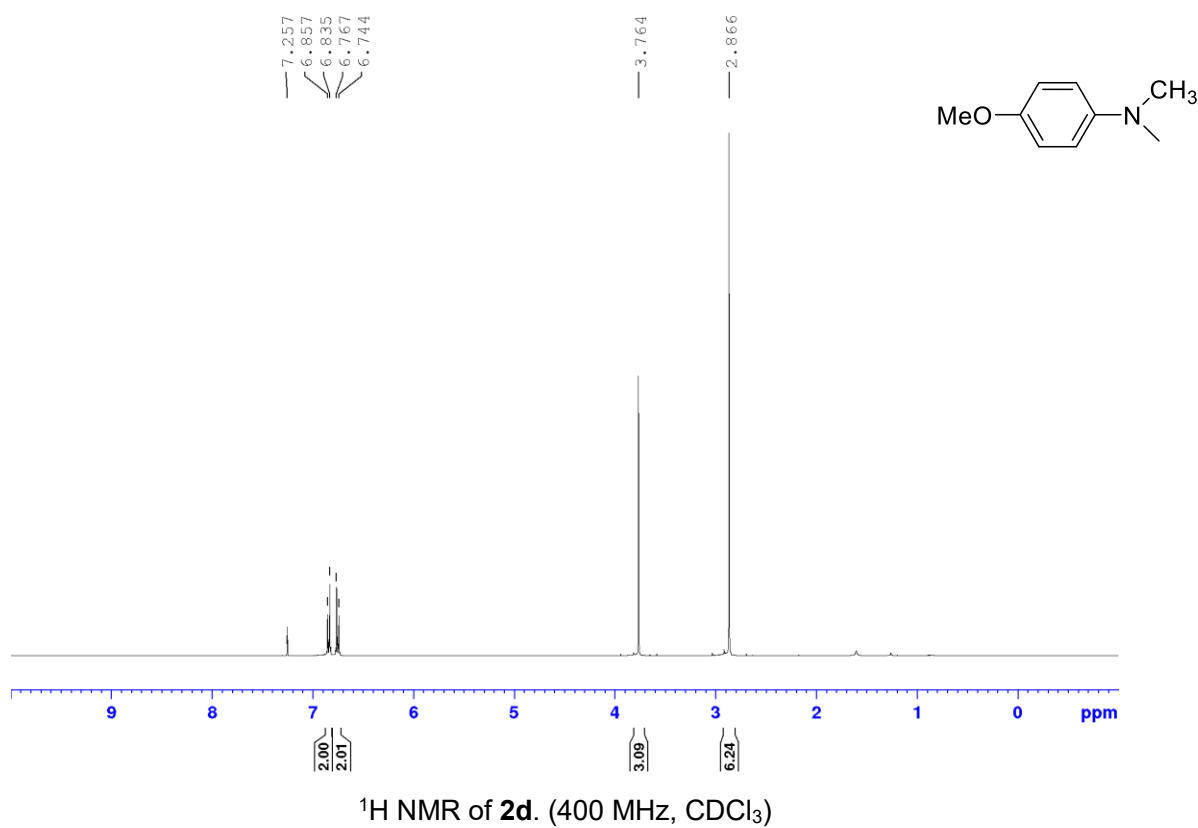
¹³C {¹H} NMR of **6c**. (100 MHz, CDCl₃)

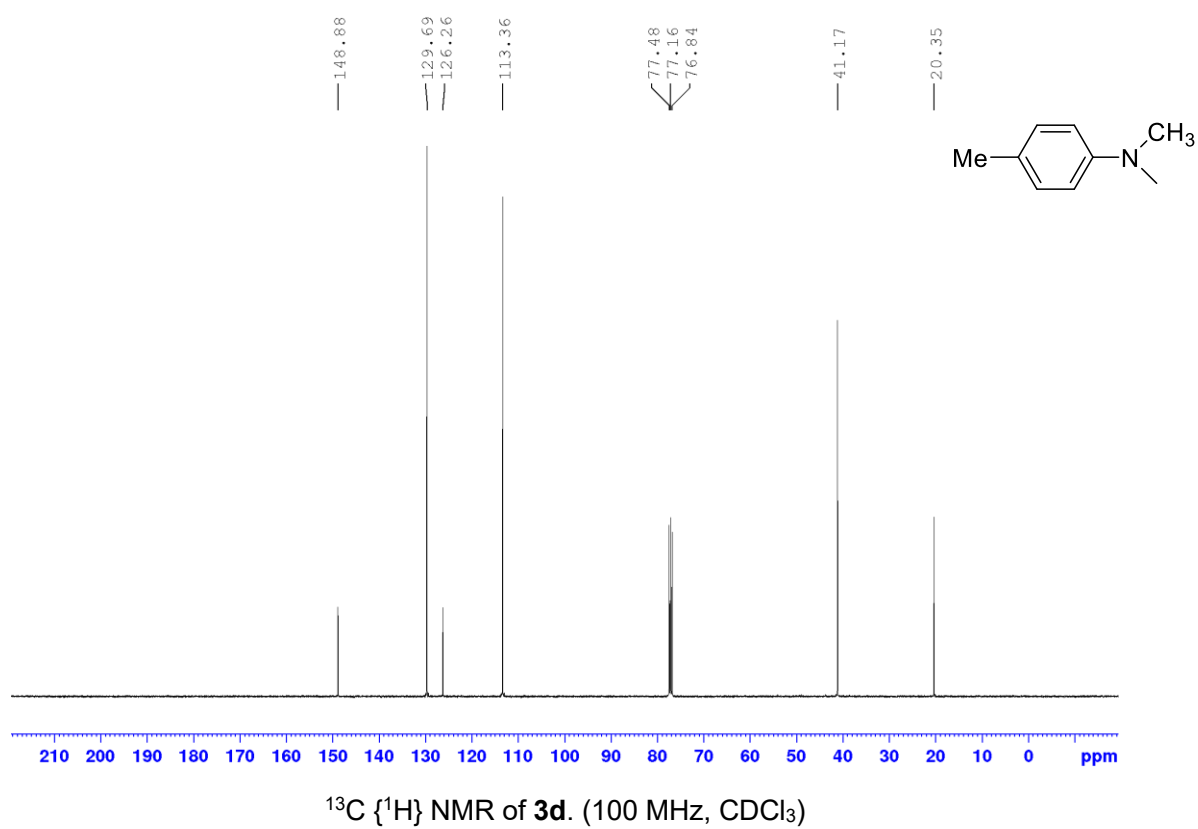
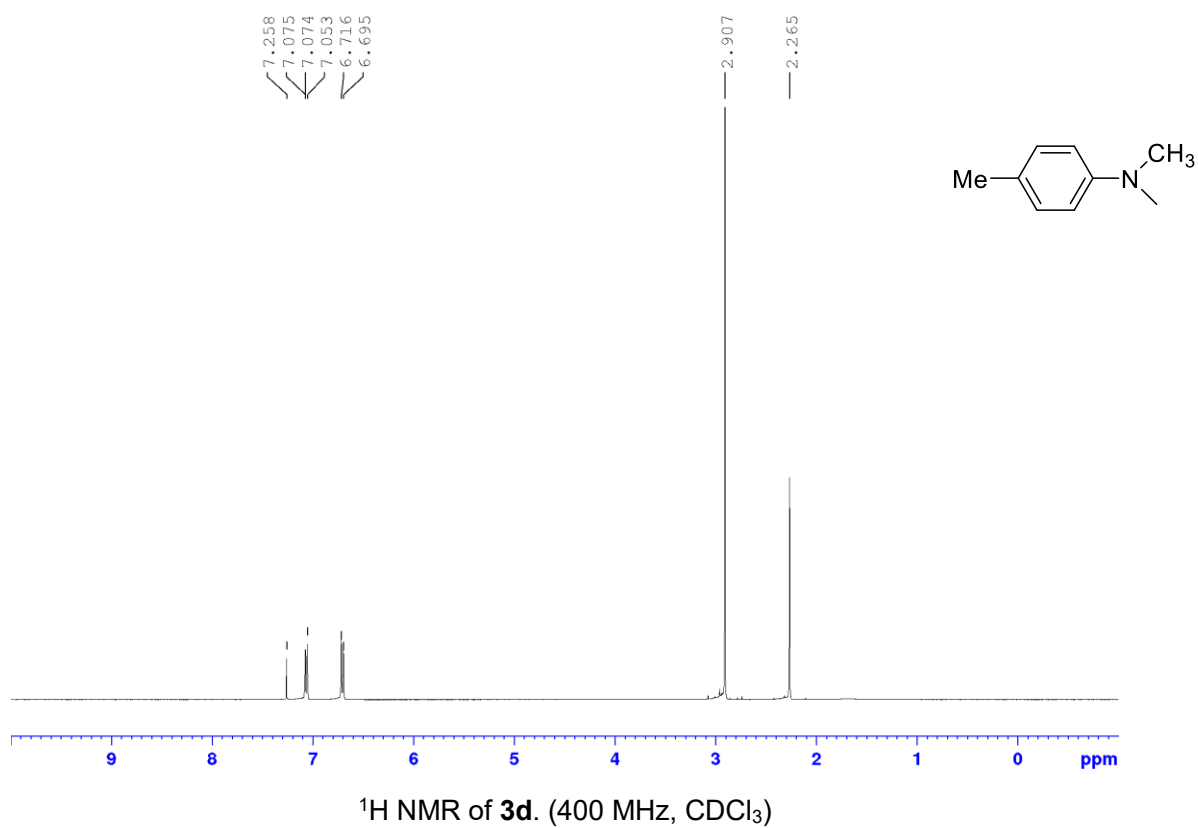


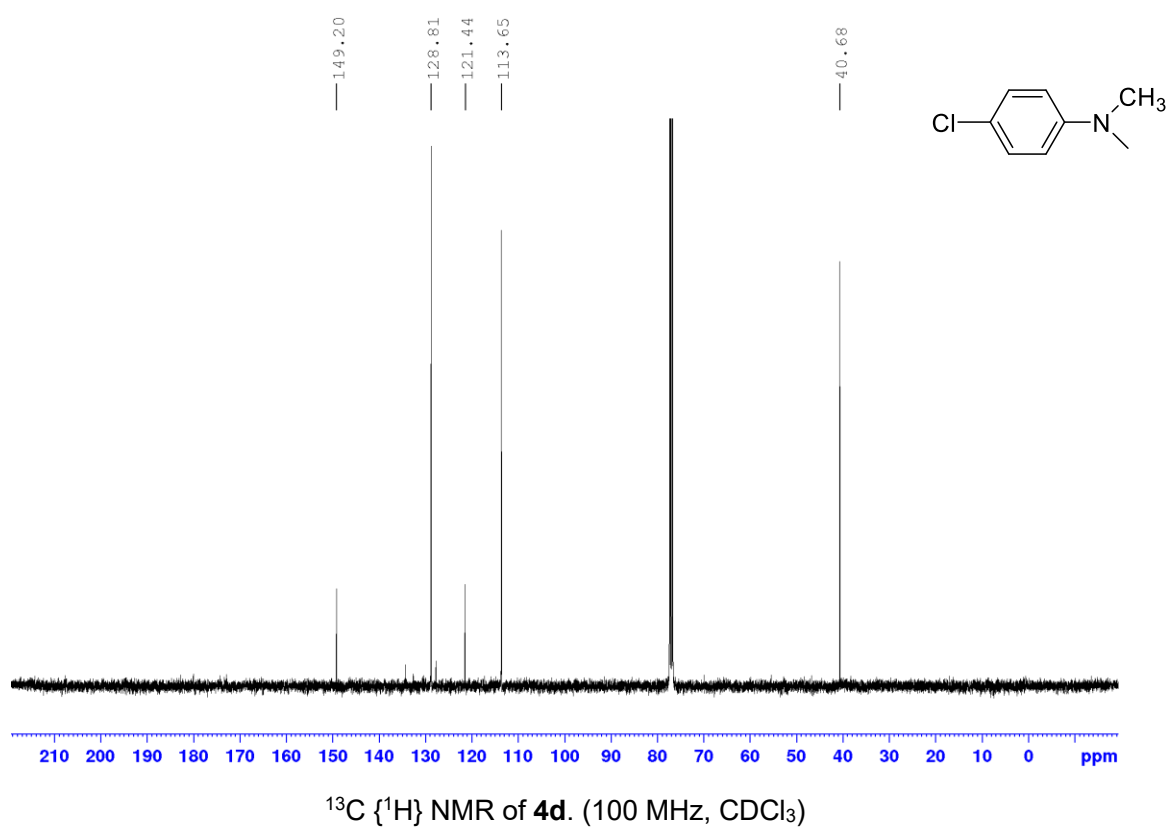
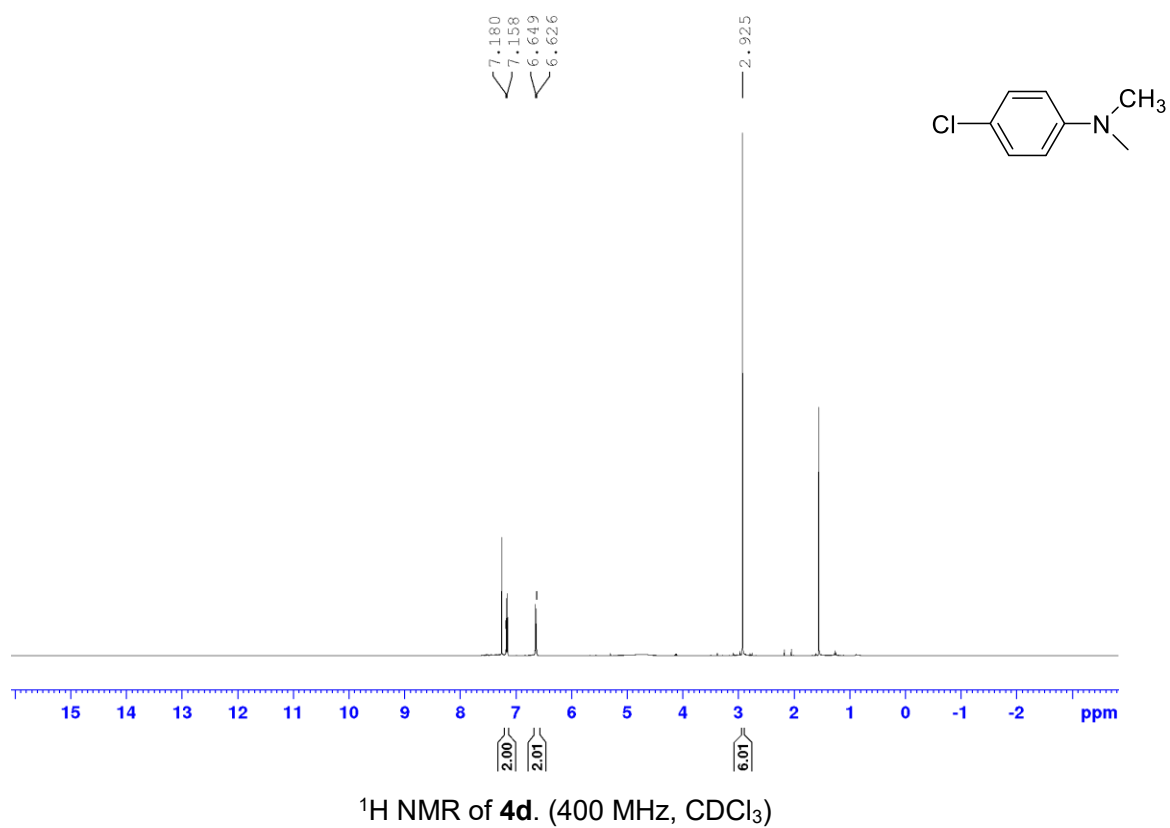
¹H NMR of **1d**. (400 MHz, CDCl₃)

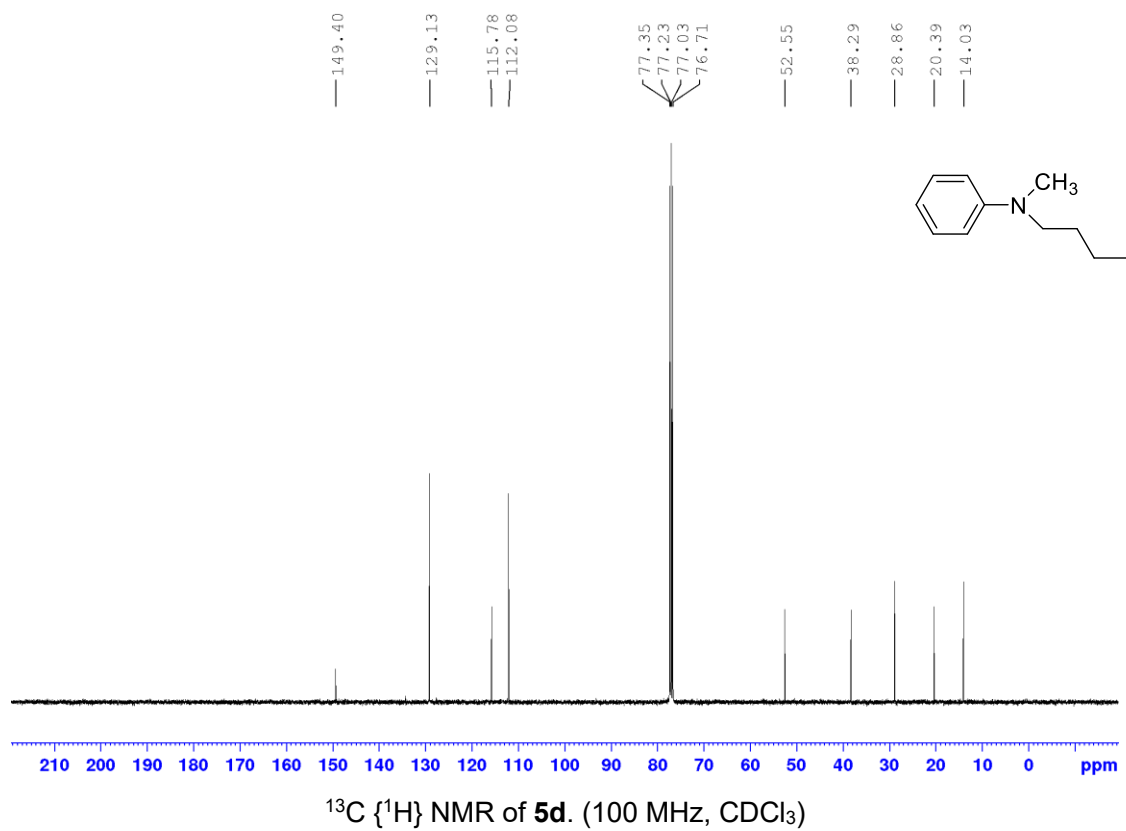
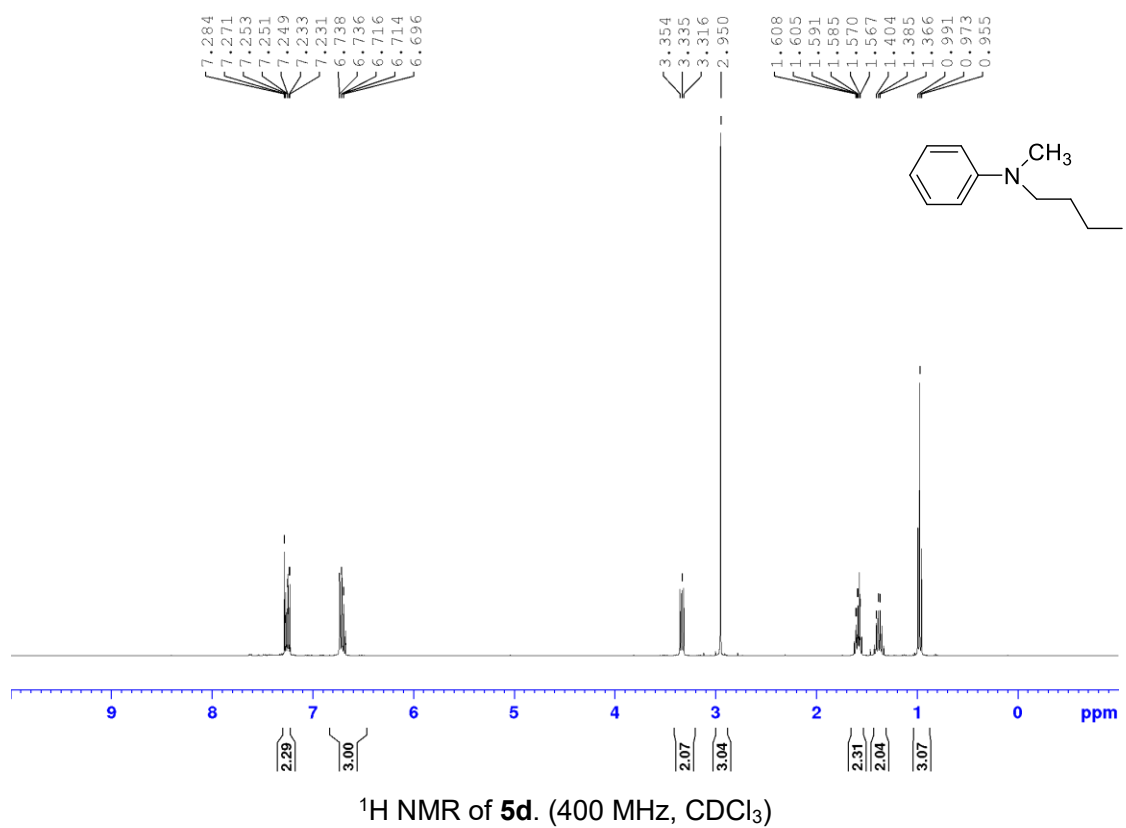


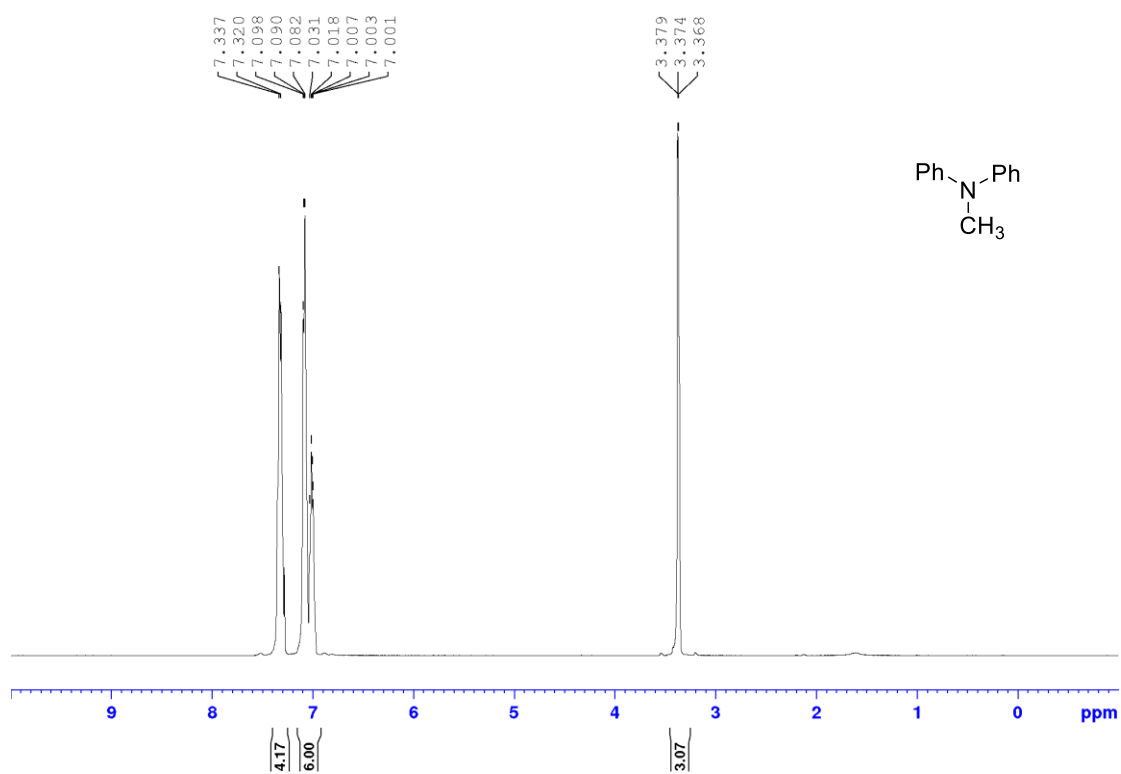
¹³C {¹H} NMR of **1d**. (100 MHz, CDCl₃)



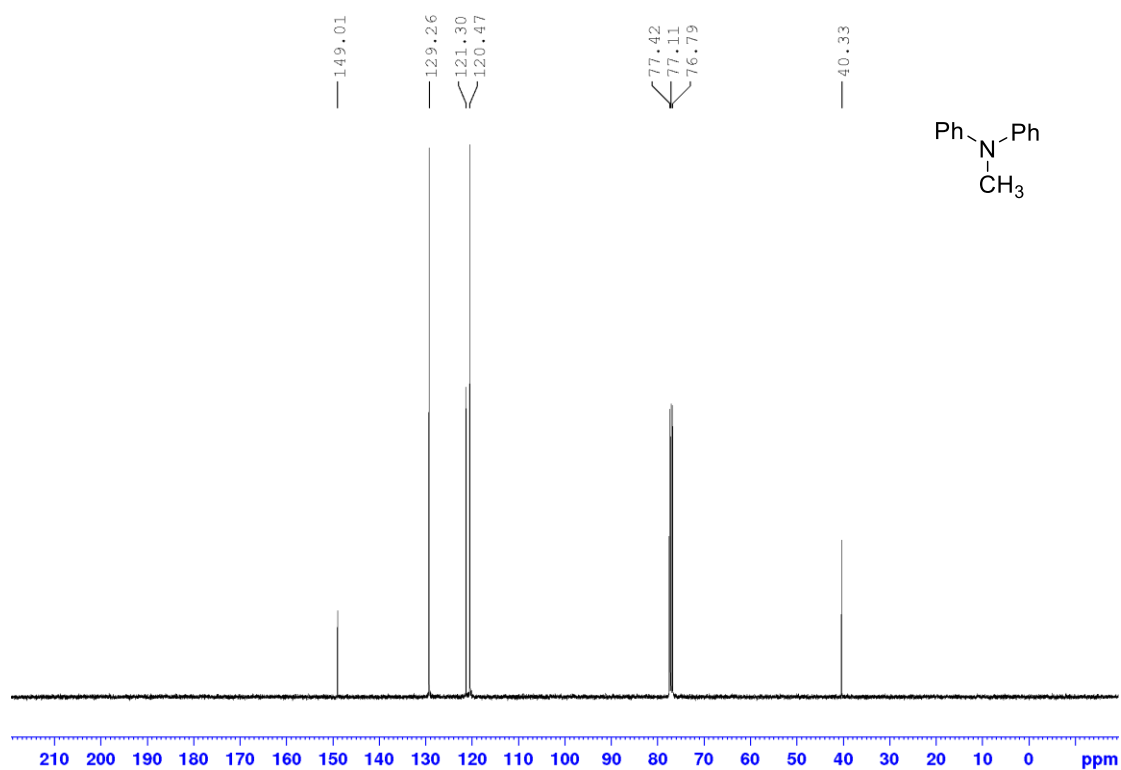




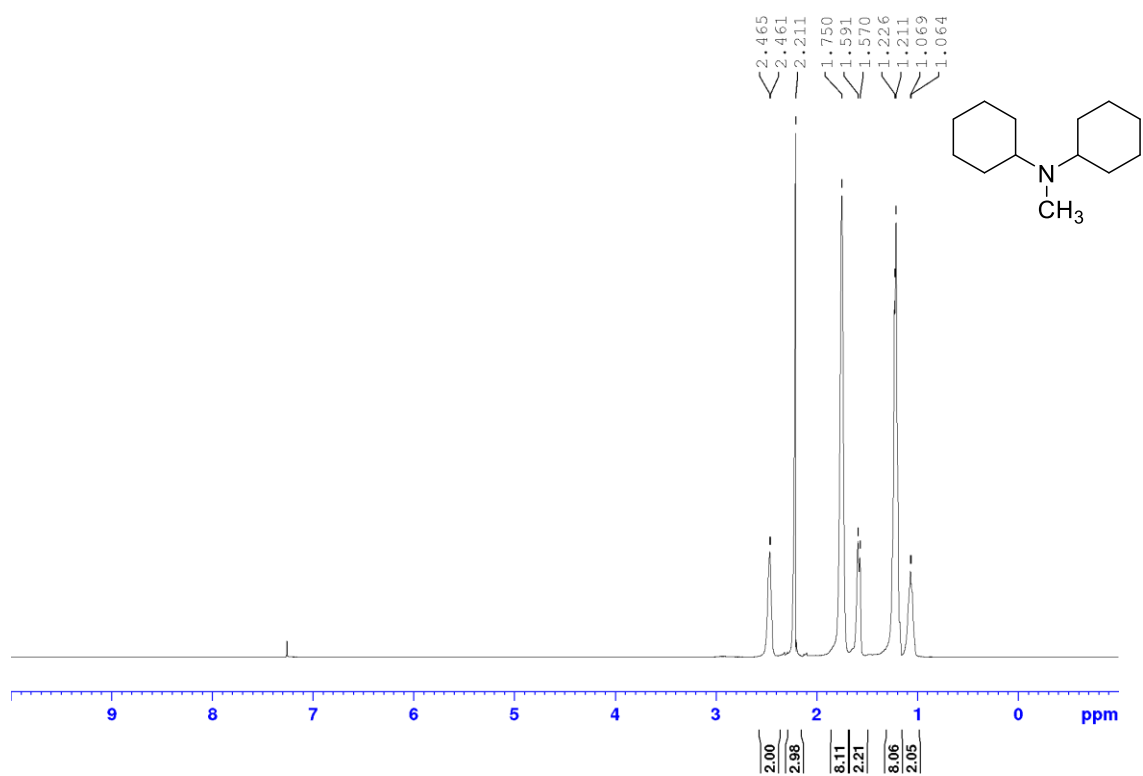




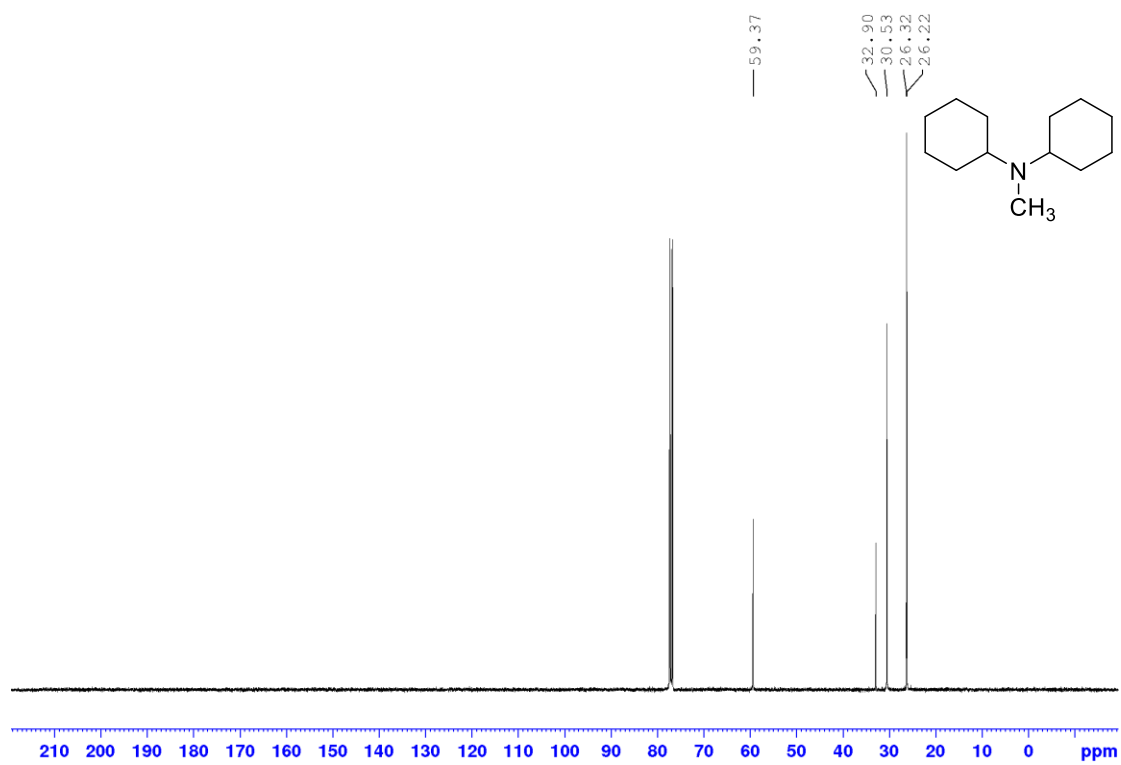
¹H NMR of **6d**. (400 MHz, CDCl₃)



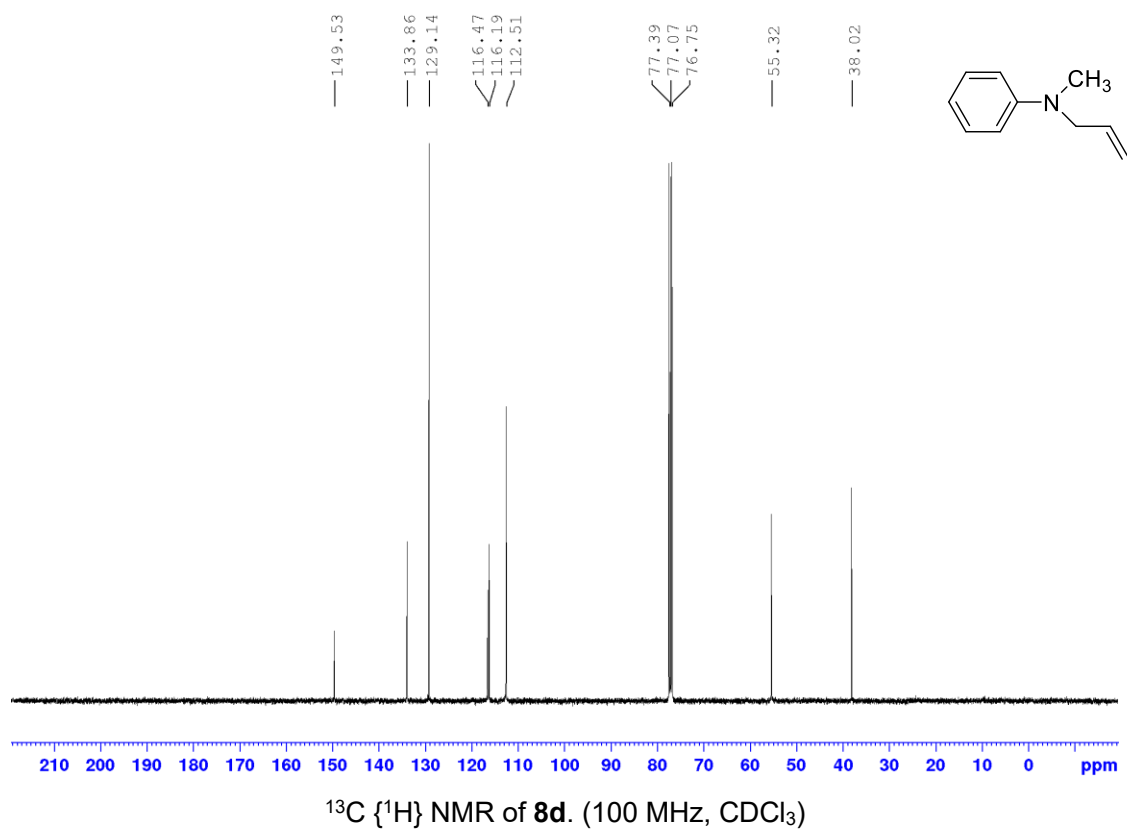
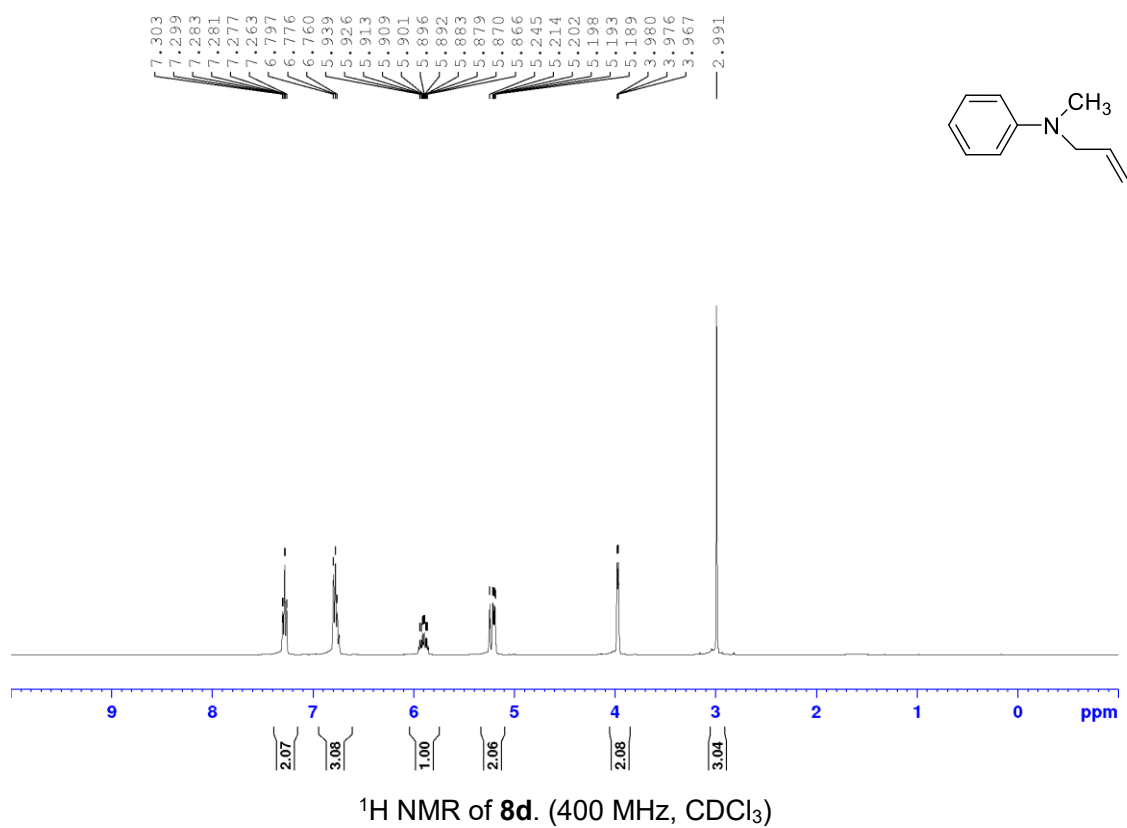
¹³C {¹H} NMR of **6d**. (100 MHz, CDCl₃)

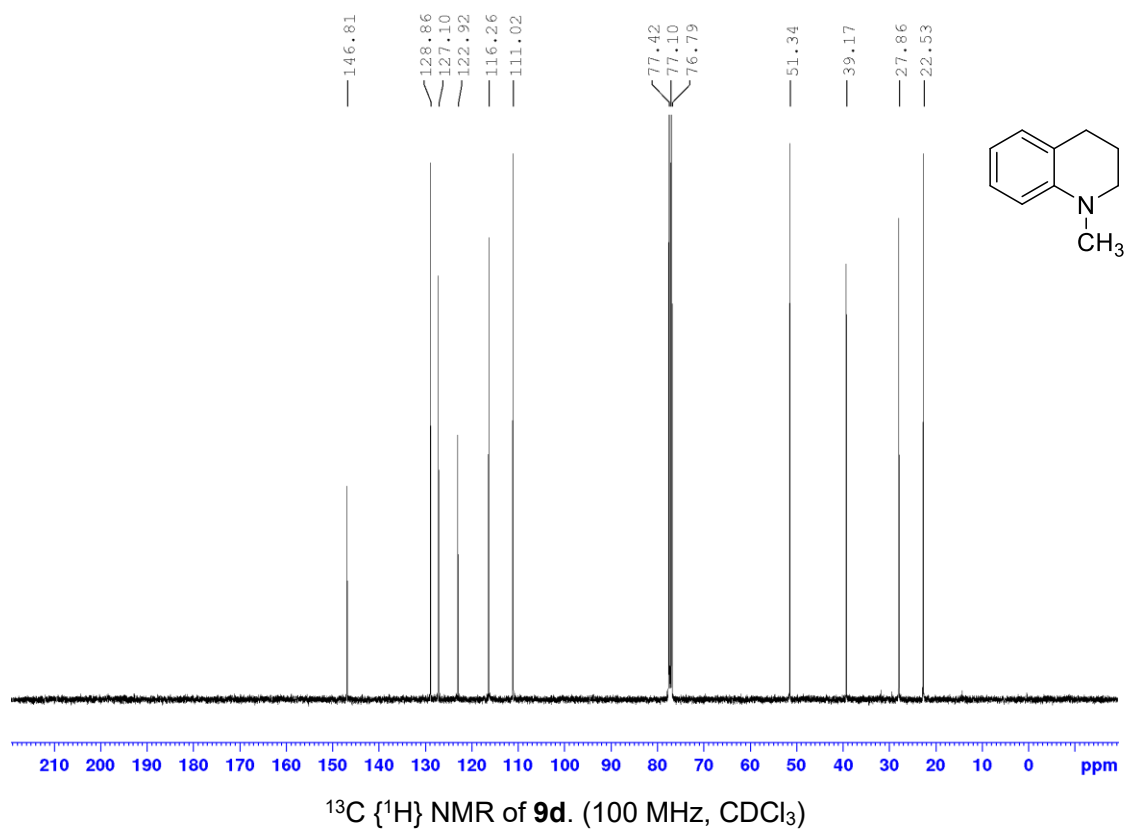
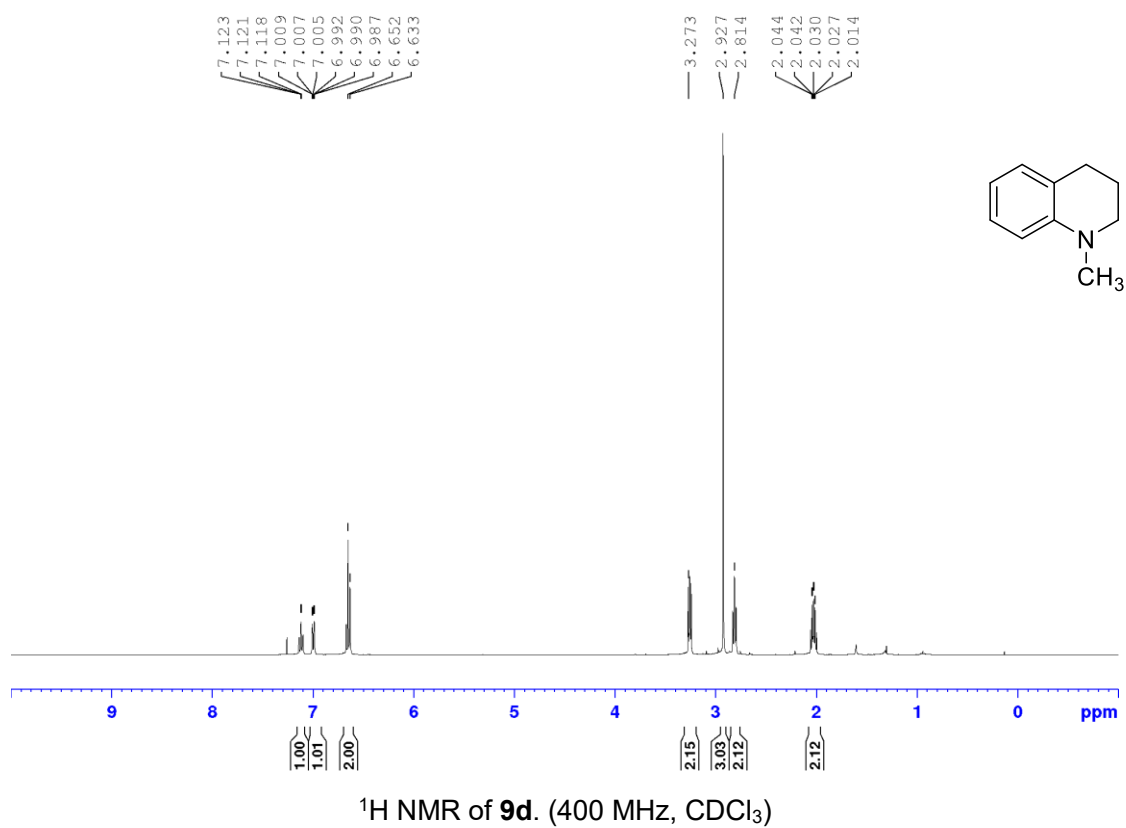


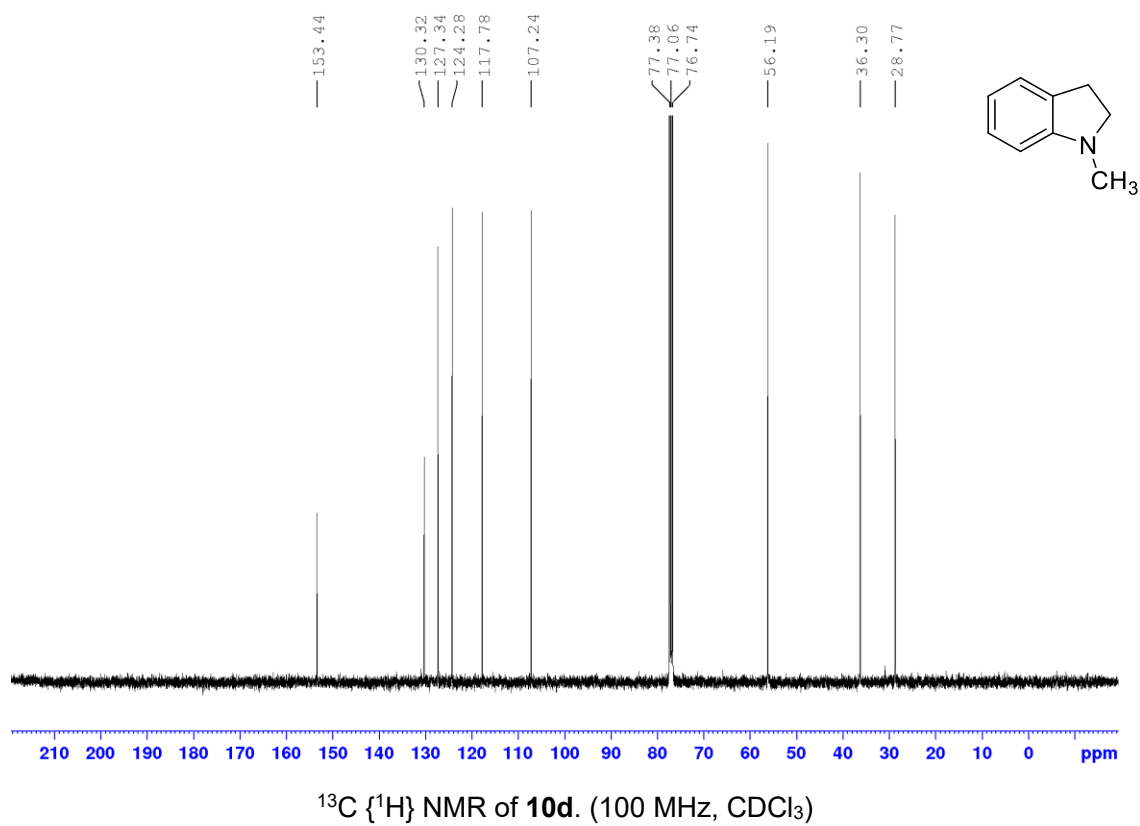
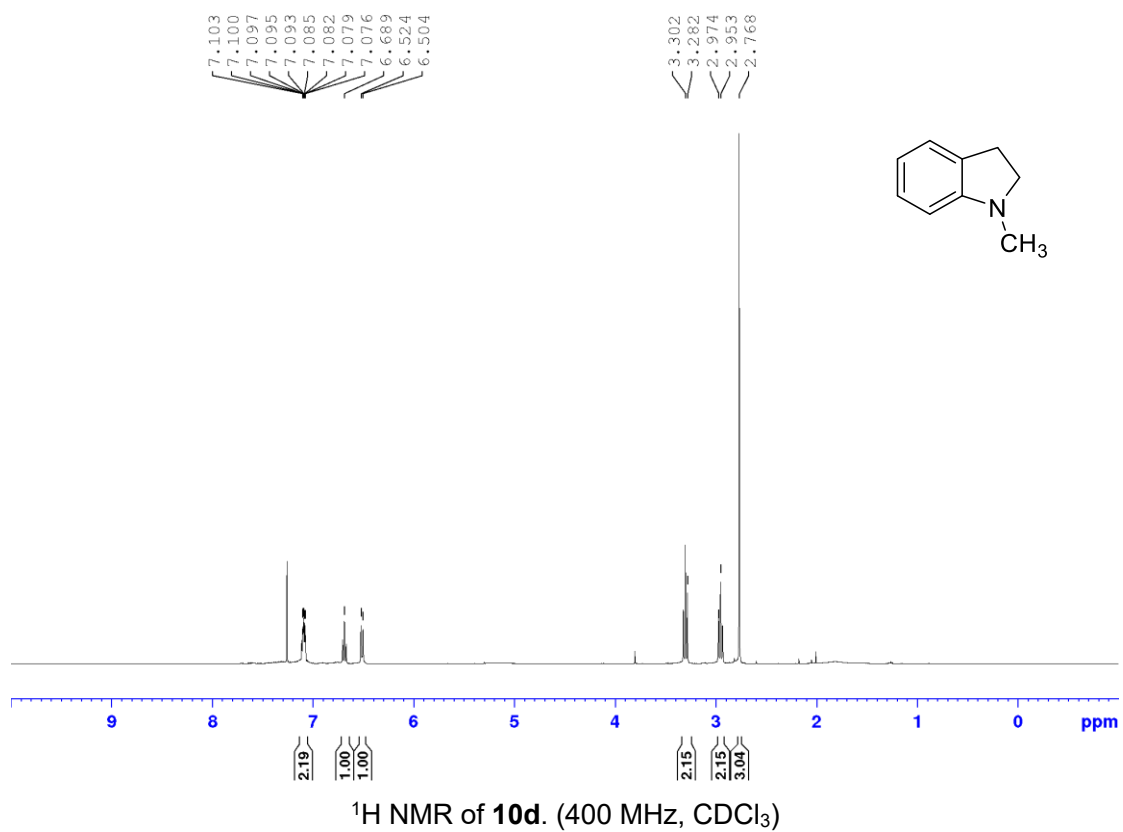
¹H NMR of **7d**. (400 MHz, CDCl₃)

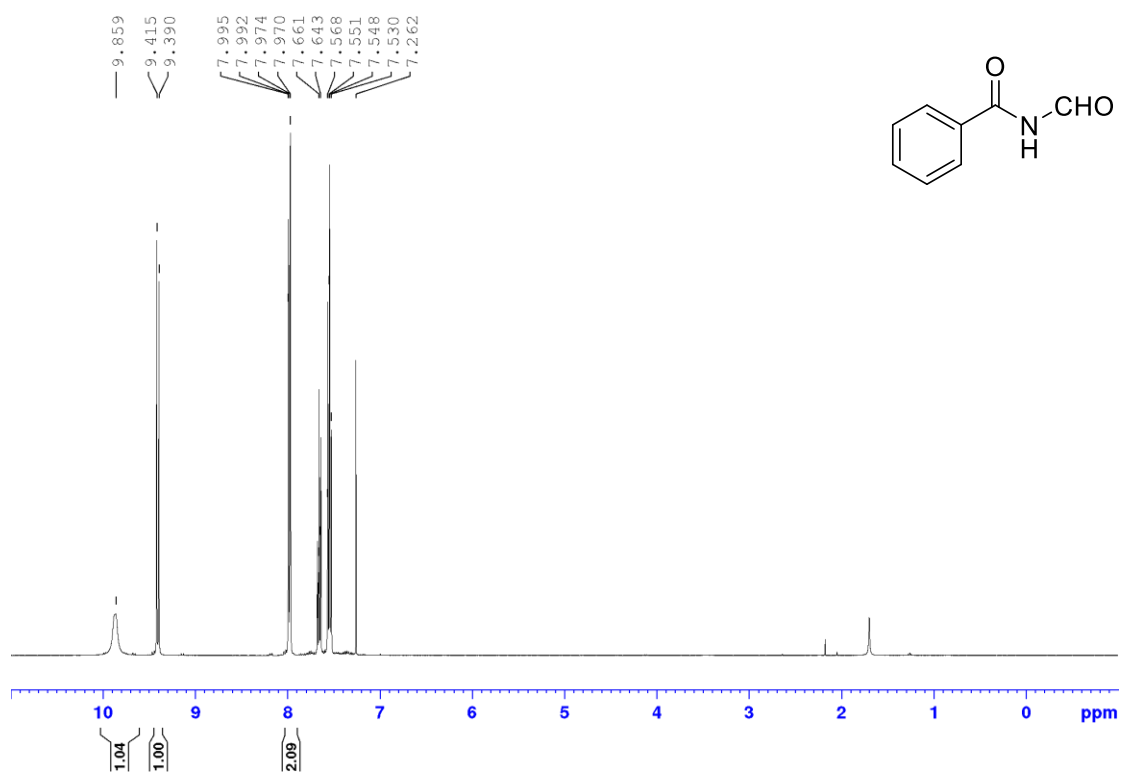


¹³C {¹H} NMR of **7d**. (100 MHz, CDCl₃)

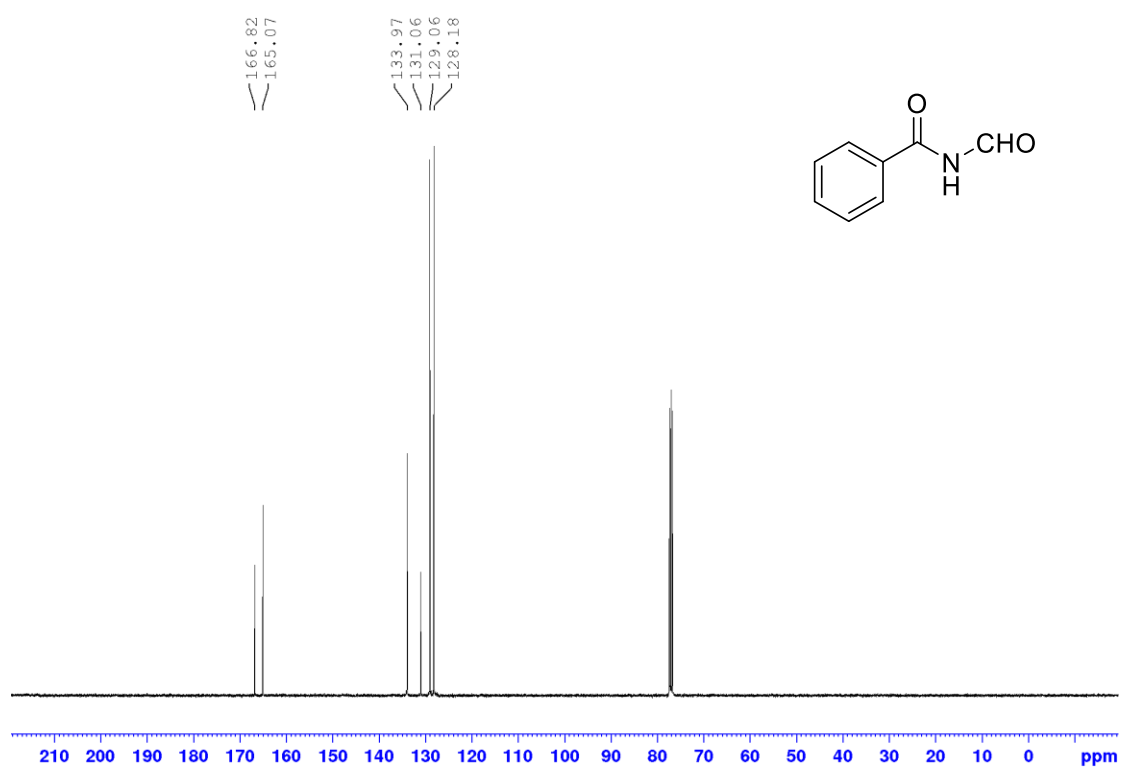




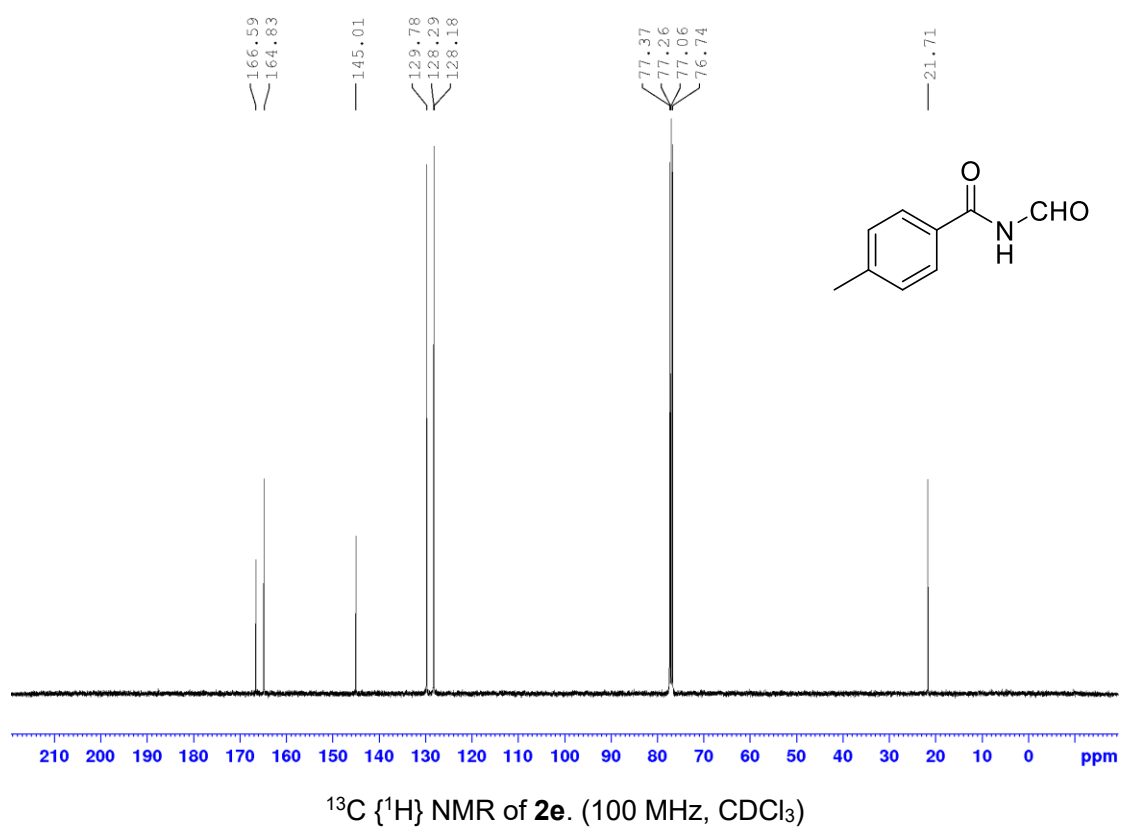
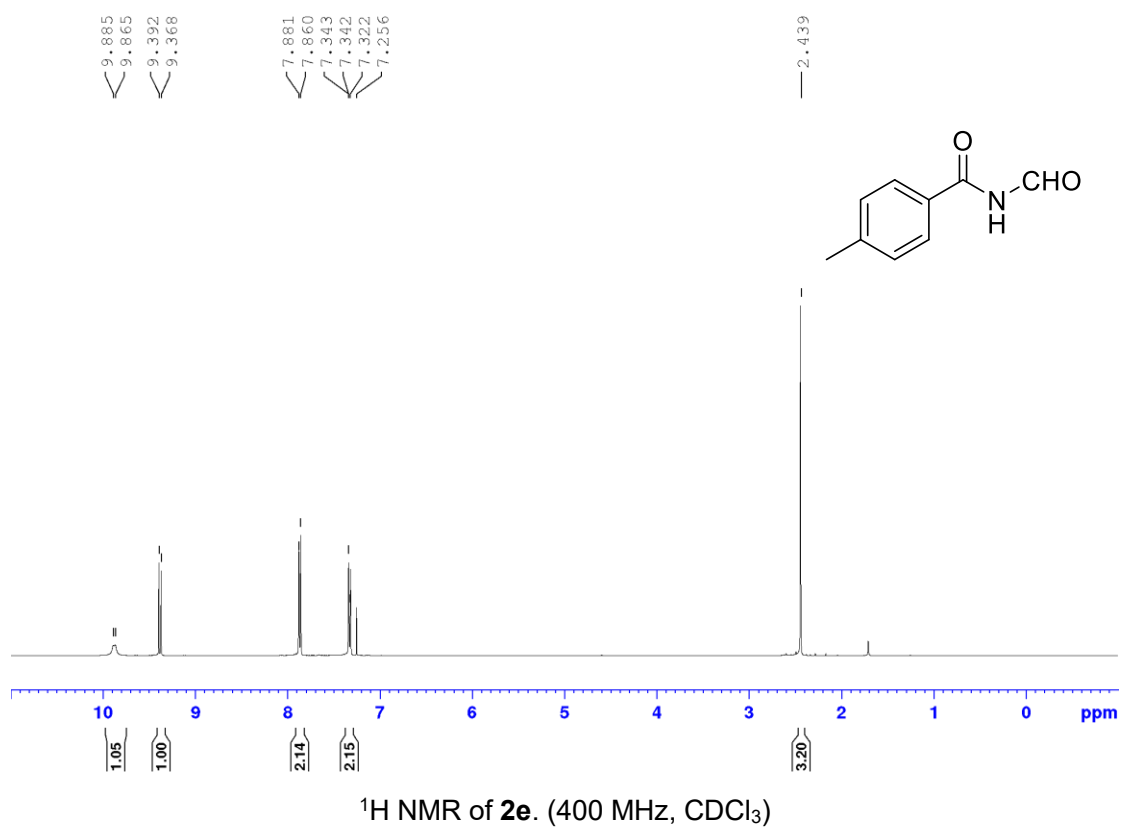


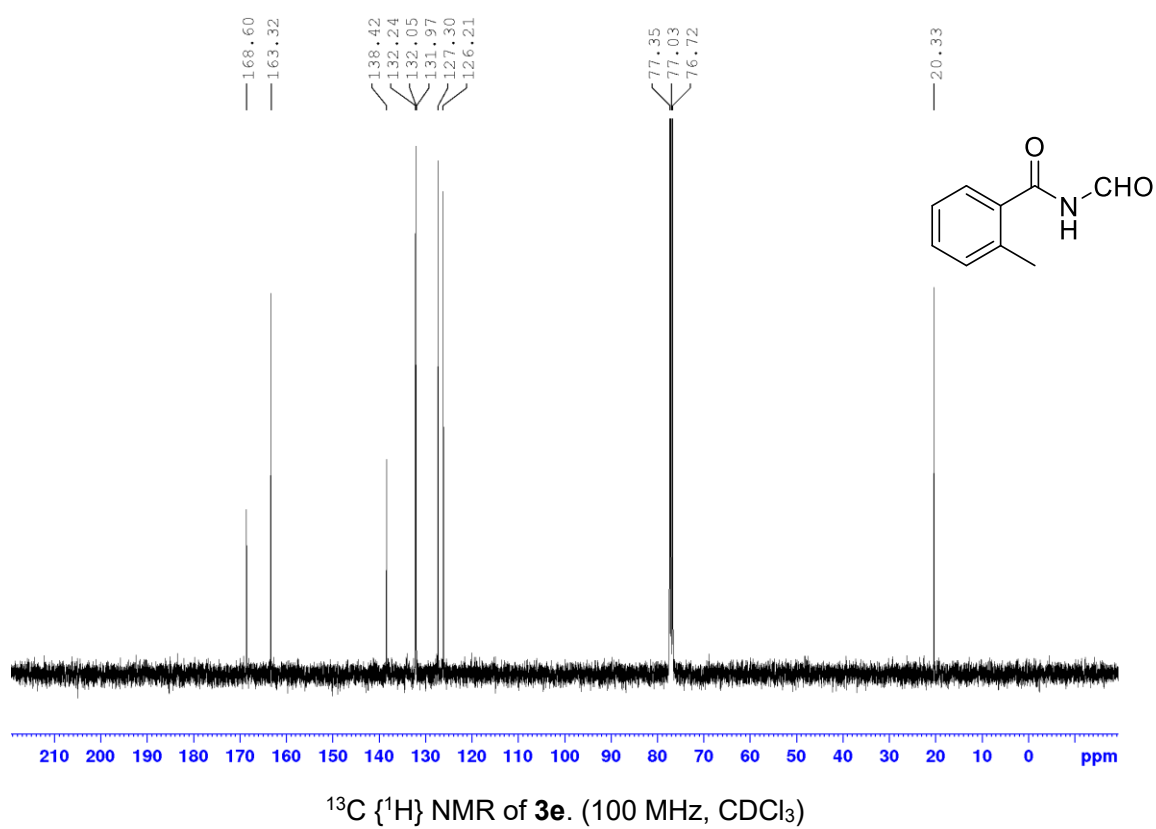
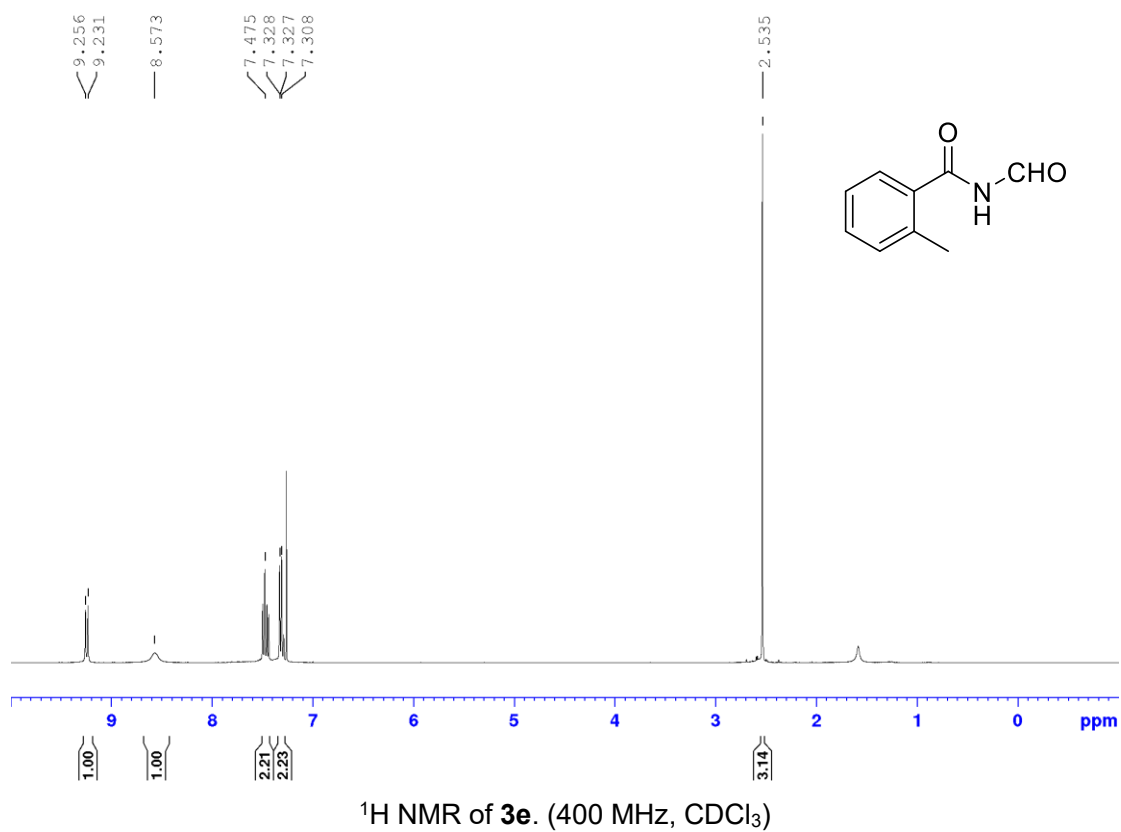


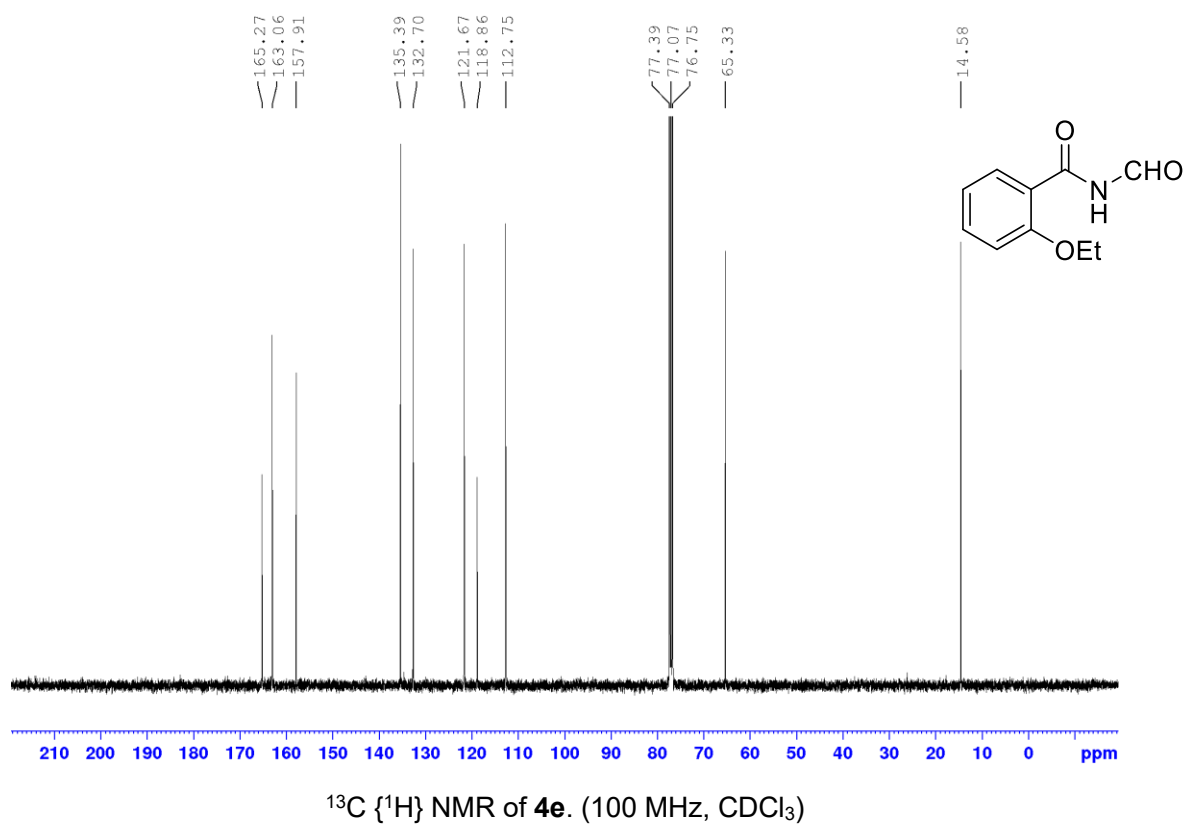
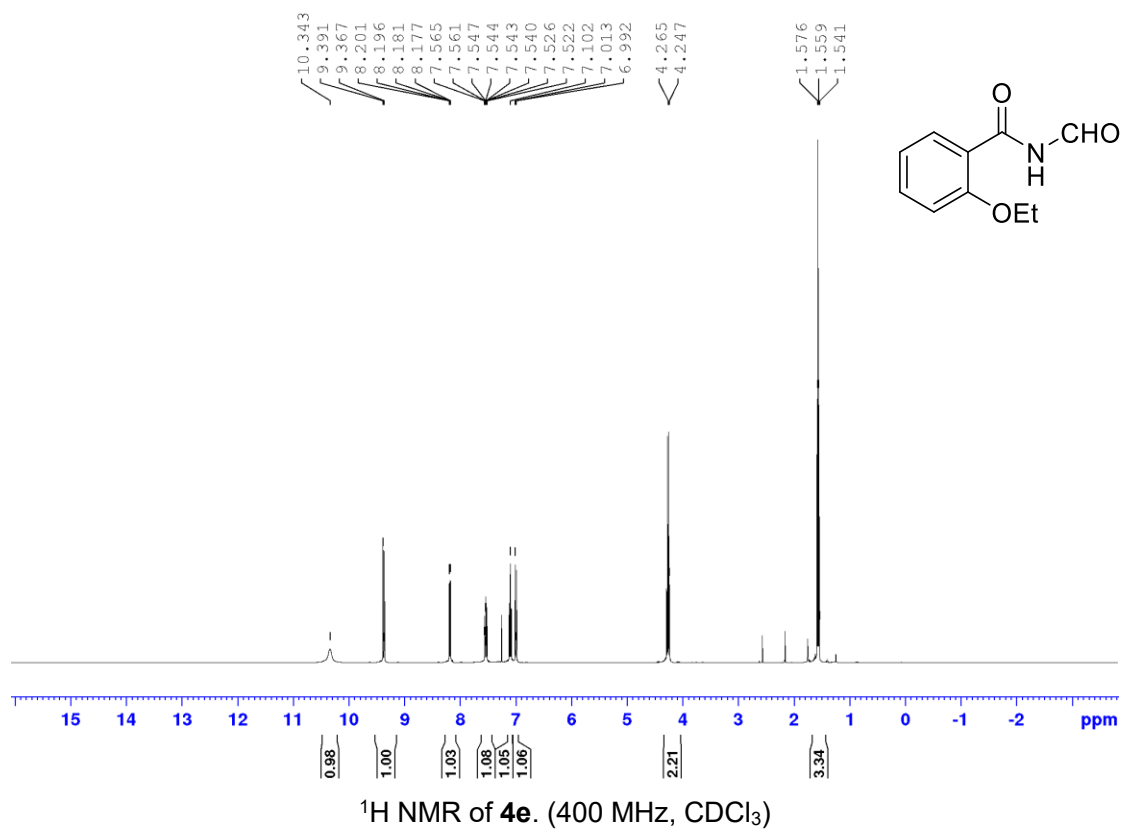
¹H NMR of **1e**. (400 MHz, CDCl₃)

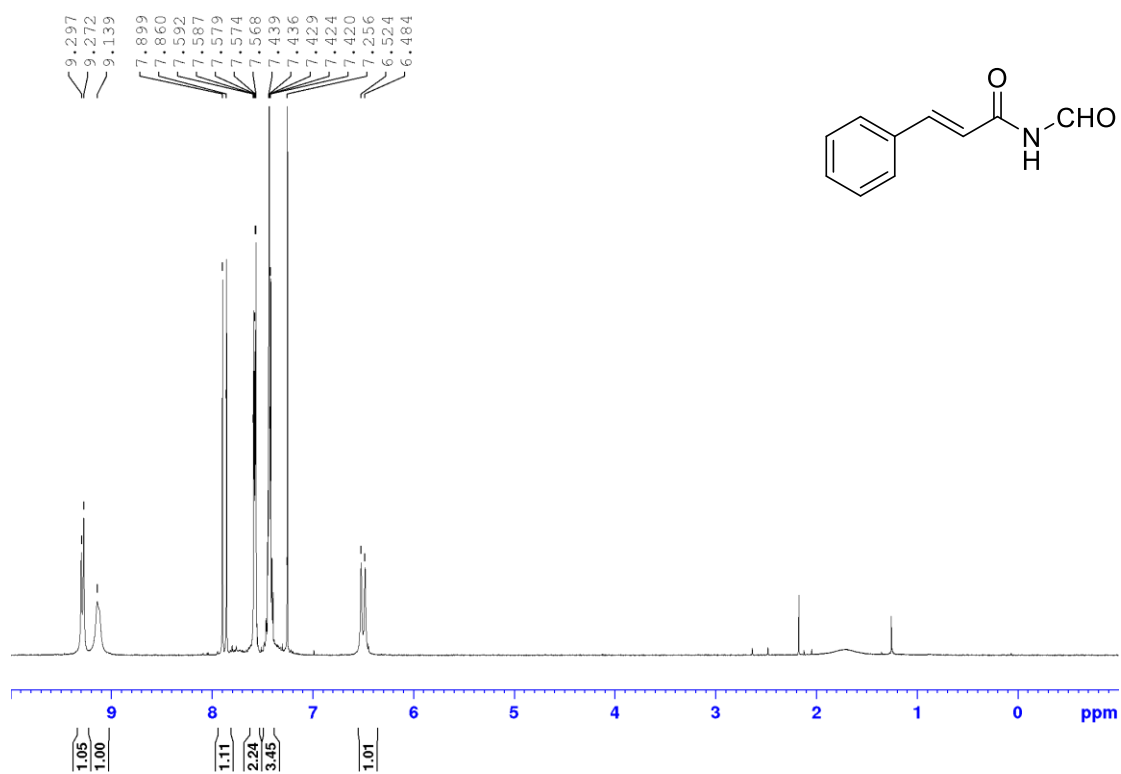


¹³C {¹H} NMR of **1e**. (100 MHz, CDCl₃)

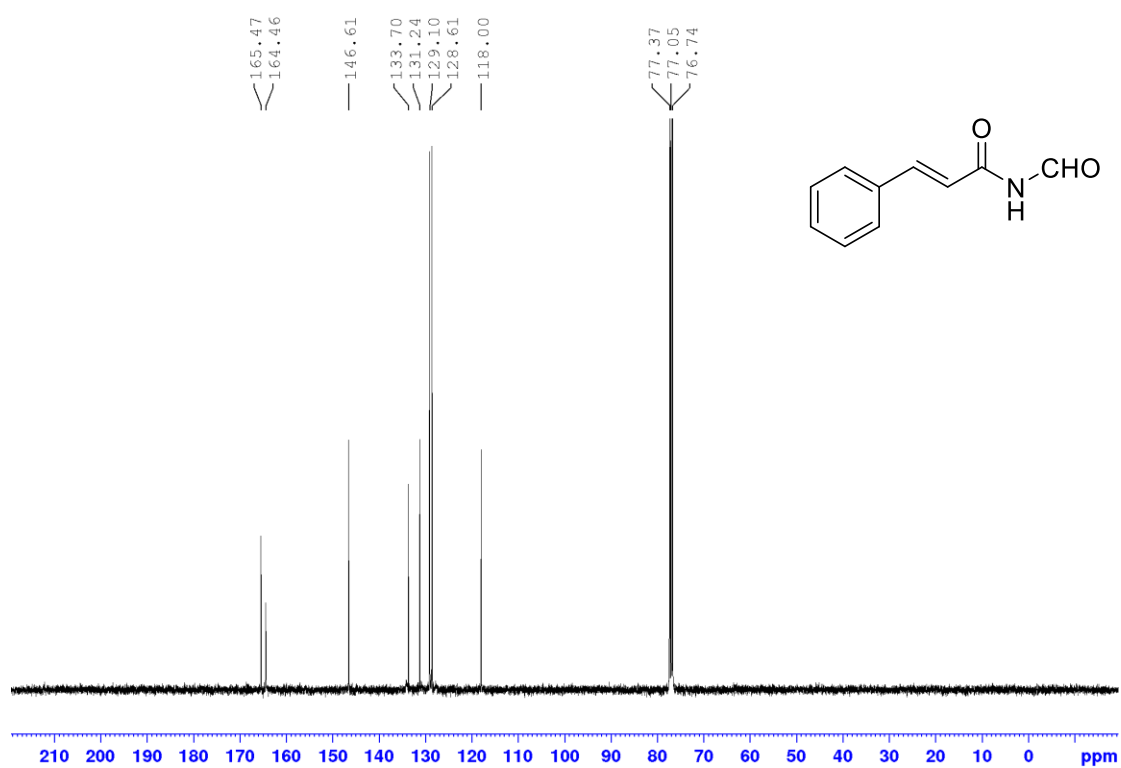




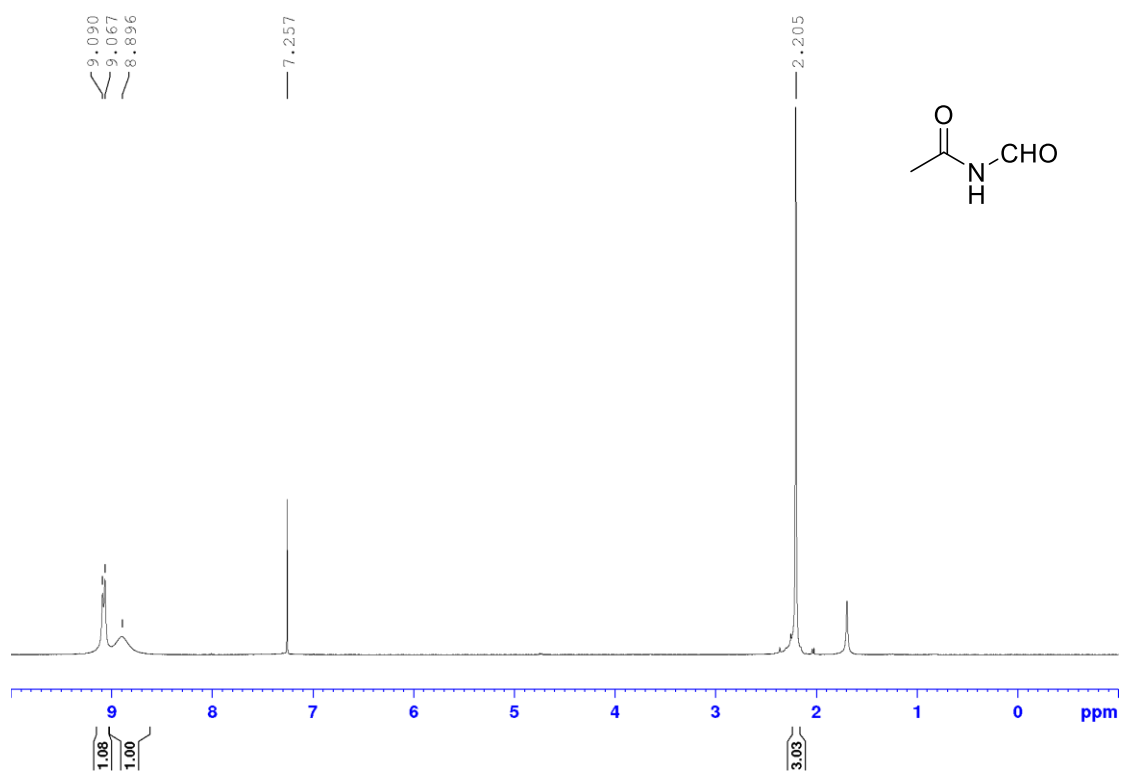




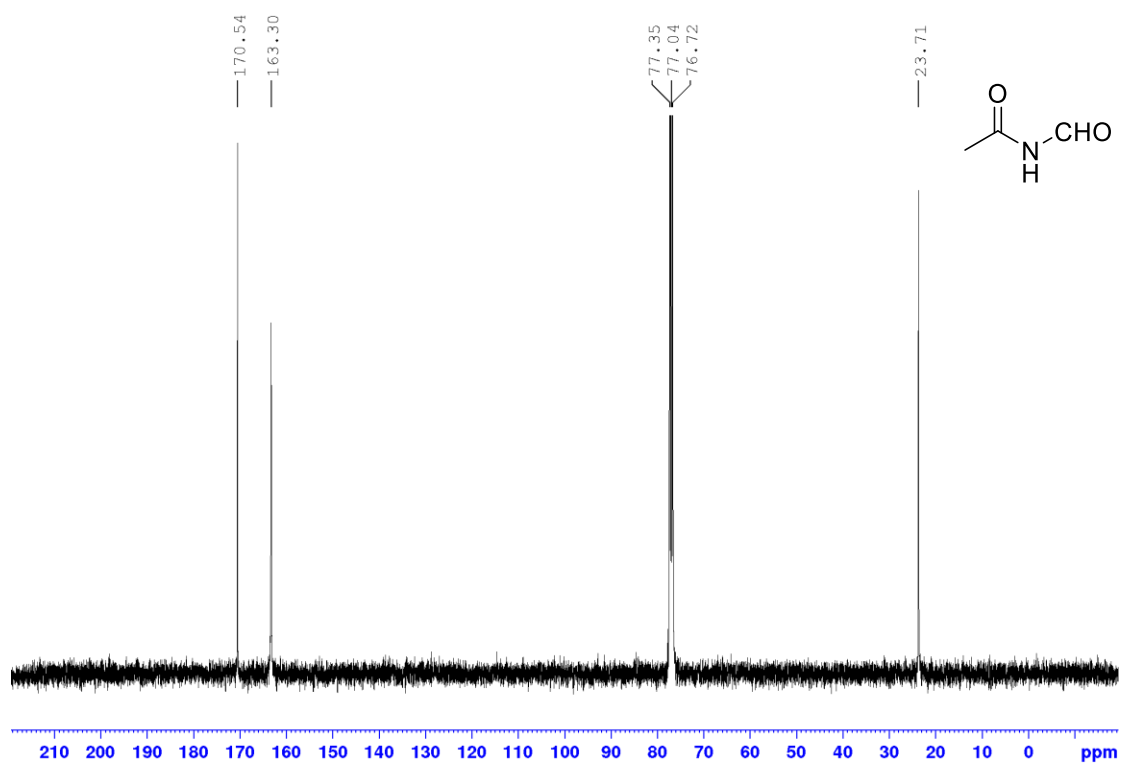
¹H NMR of **5e**. (400 MHz, CDCl₃)



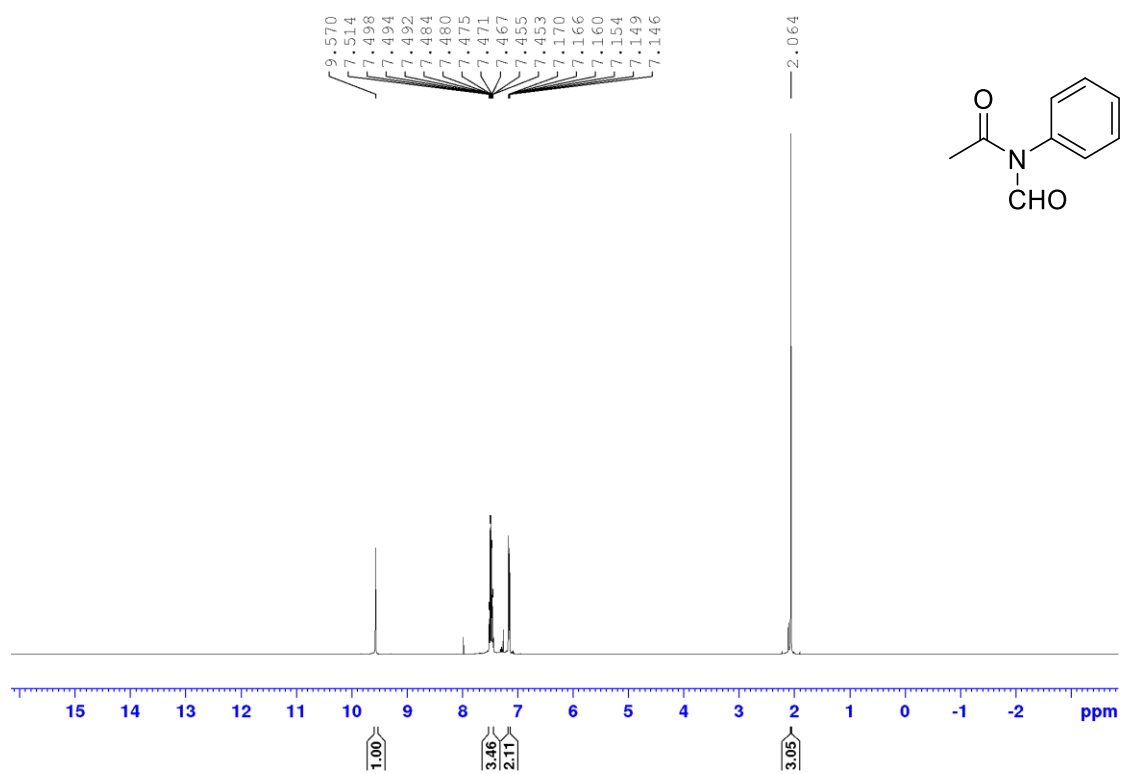
¹³C {¹H} NMR of **5e**. (100 MHz, CDCl₃)



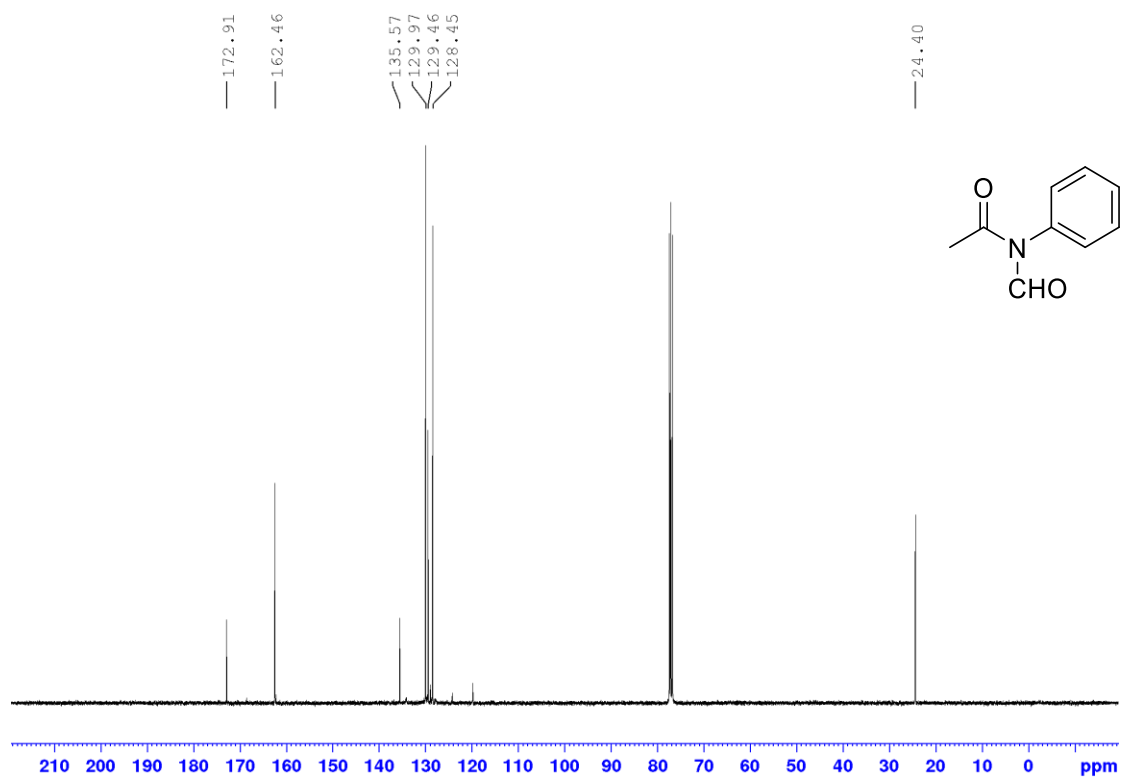
¹H NMR of **6e**. (400 MHz, CDCl₃)



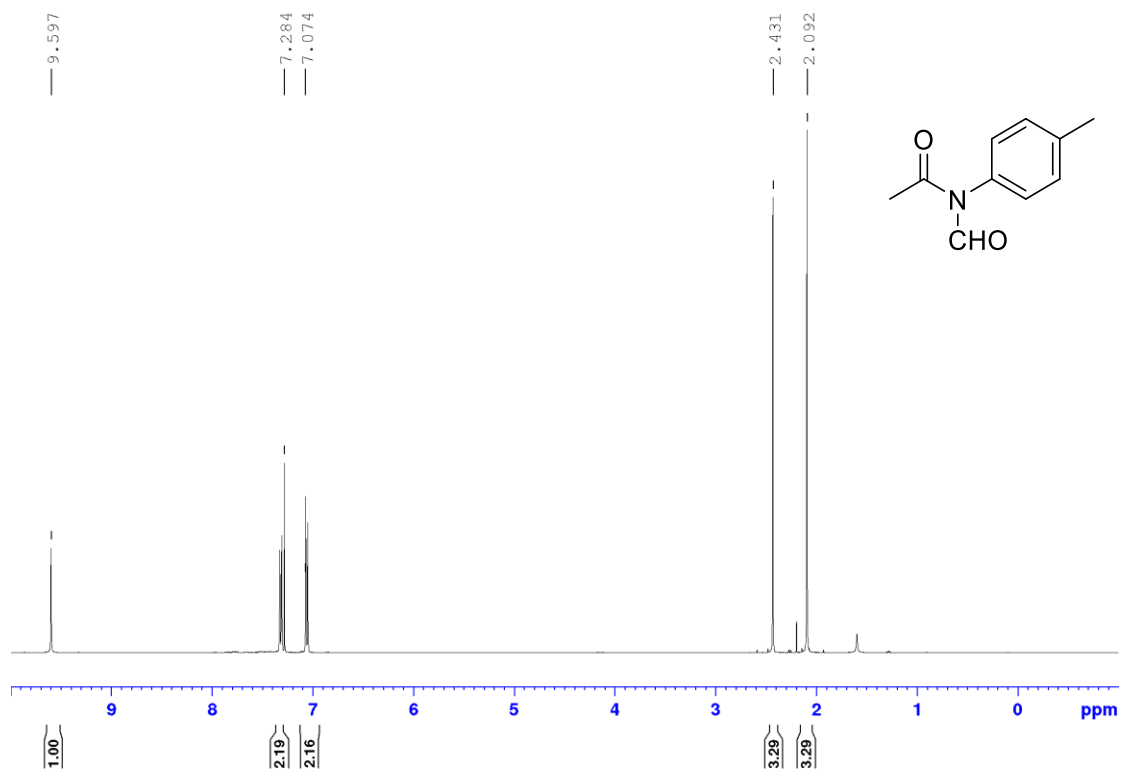
¹³C {¹H} NMR of **6e**. (100 MHz, CDCl₃)



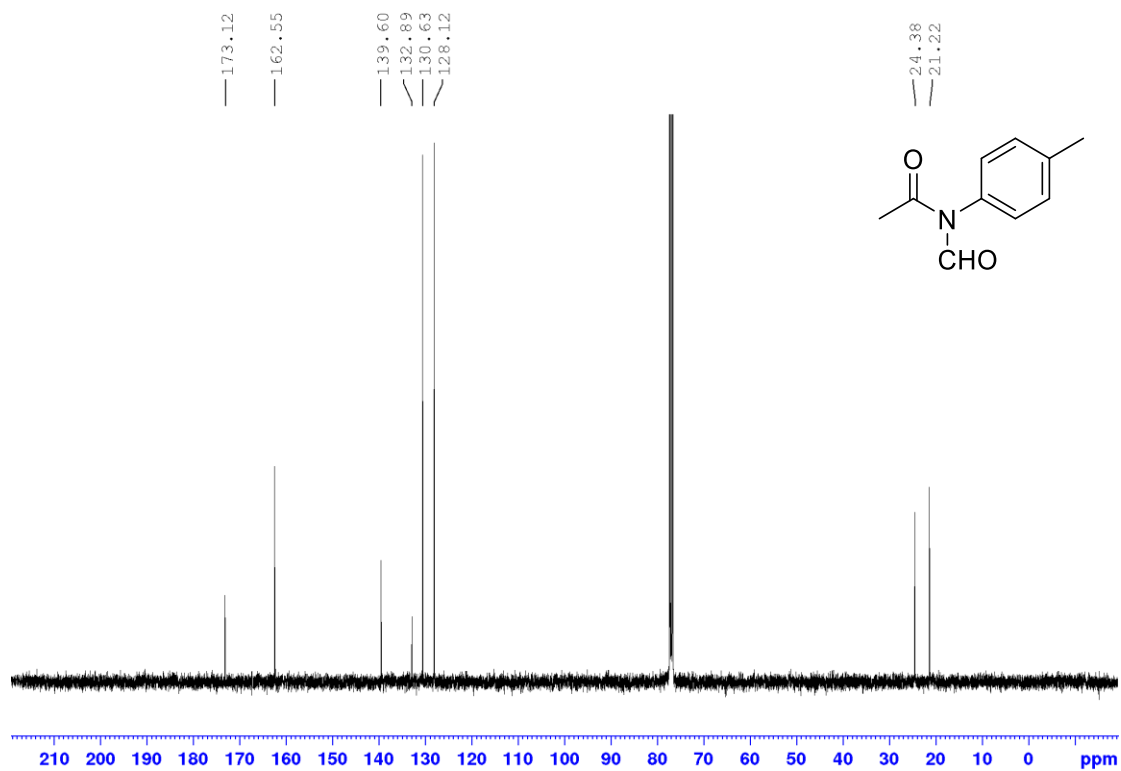
¹H NMR of **1f**. (400 MHz, CDCl₃)



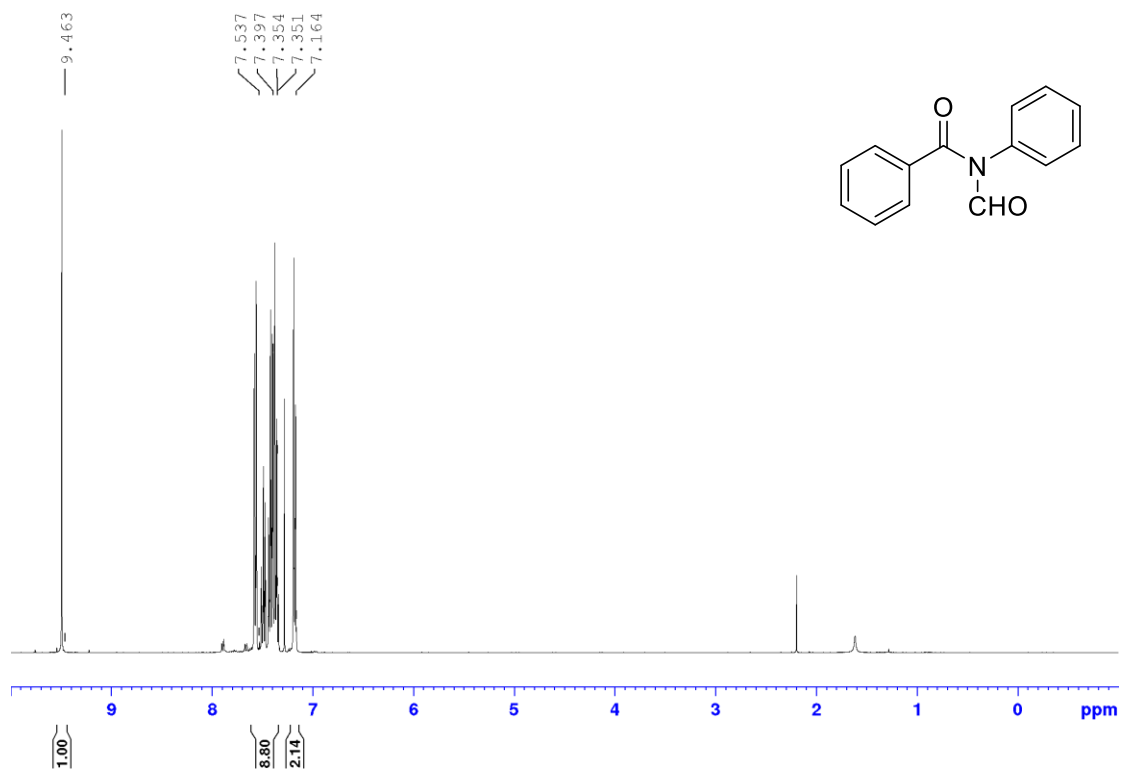
¹³C {¹H} NMR of **1f**. (100 MHz, CDCl₃)



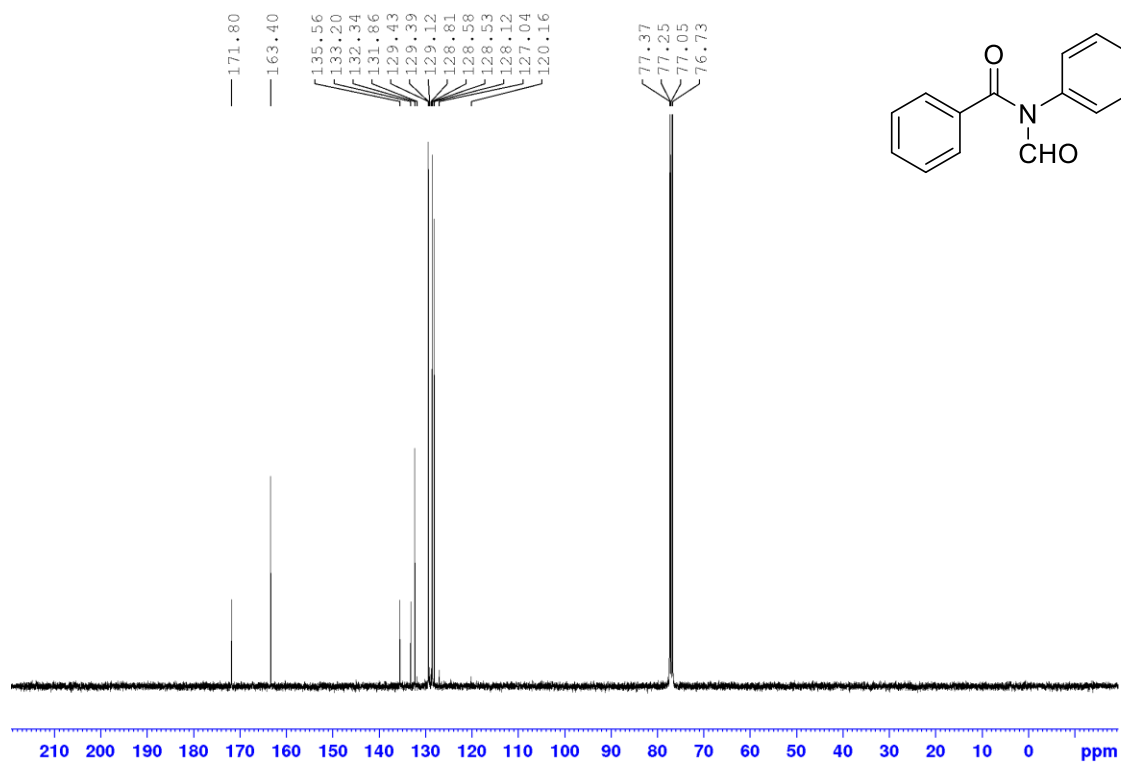
^1H NMR of **2f**. (400 MHz, CDCl_3)



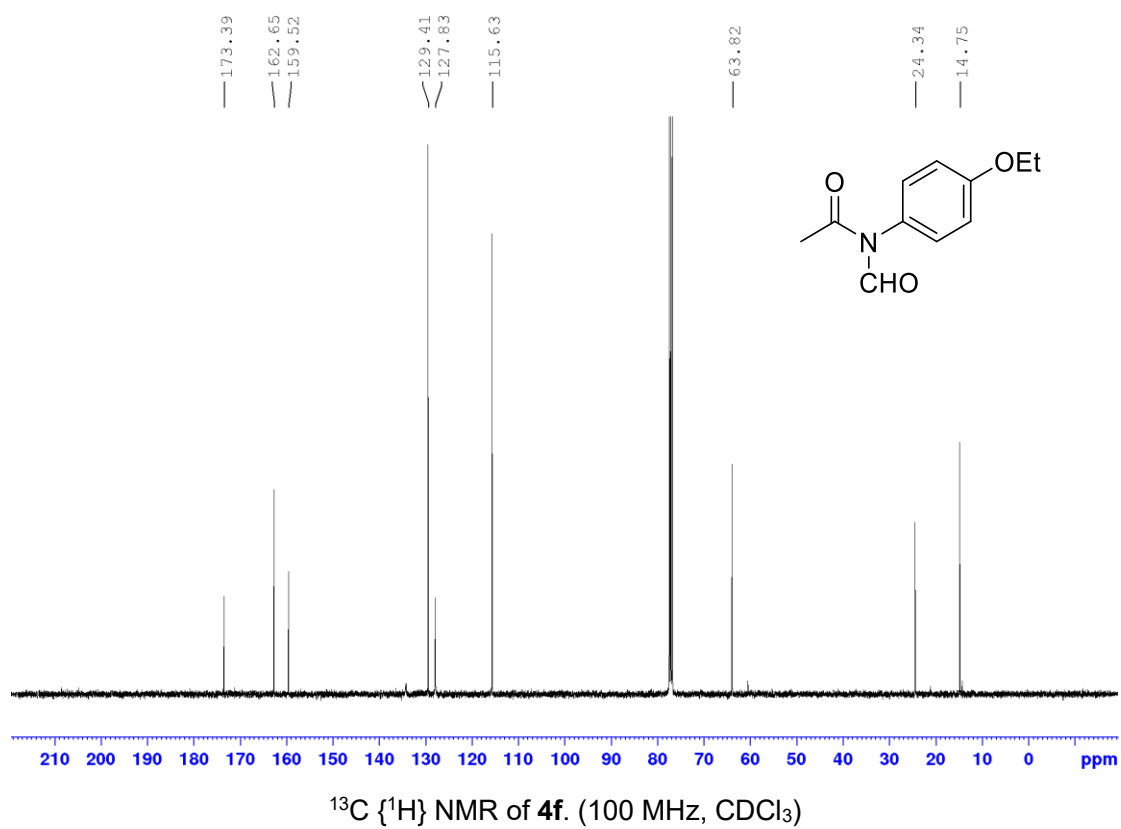
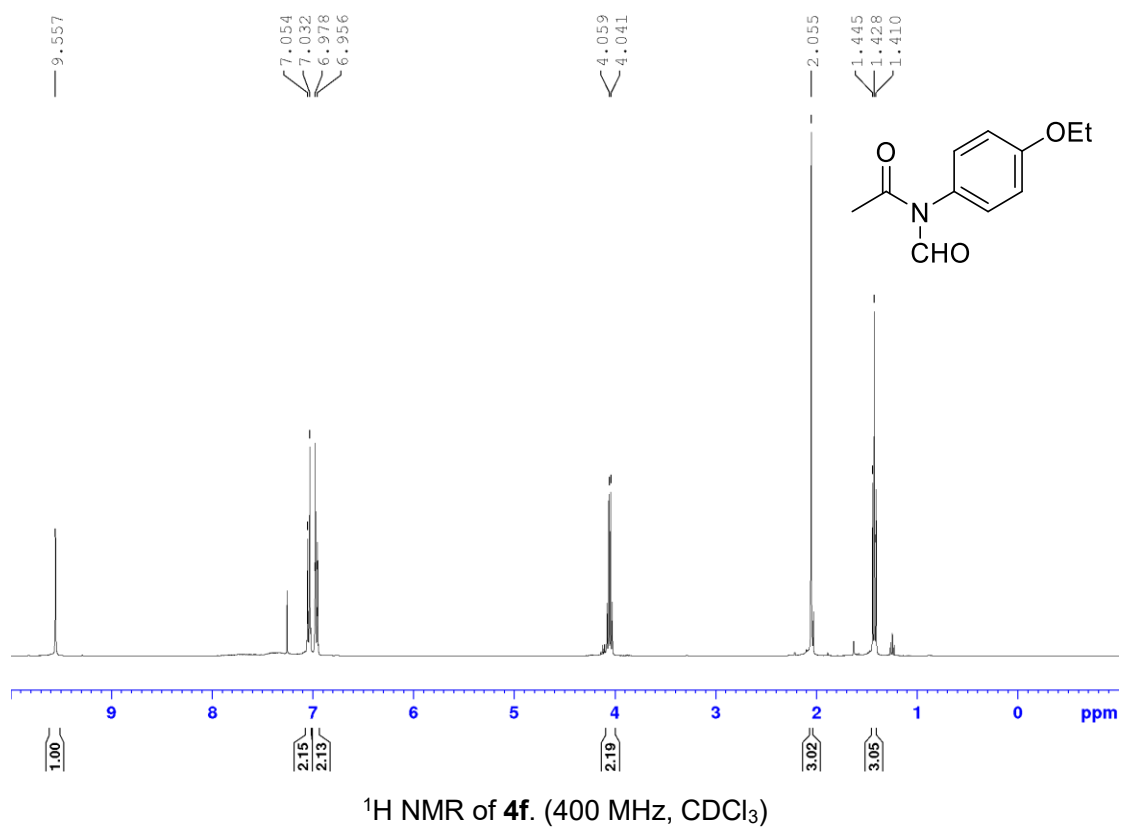
^{13}C $\{^1\text{H}\}$ NMR of **2f**. (100 MHz, CDCl_3)

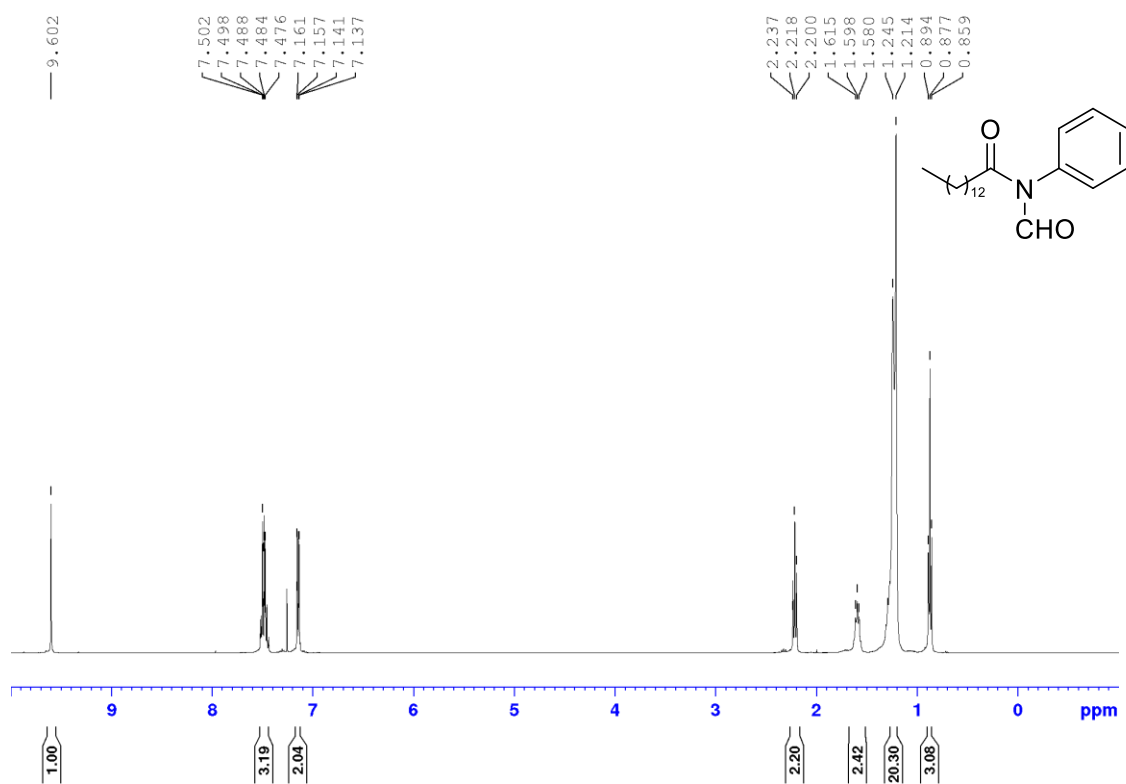


^1H NMR of **3f**. (400 MHz, CDCl_3)

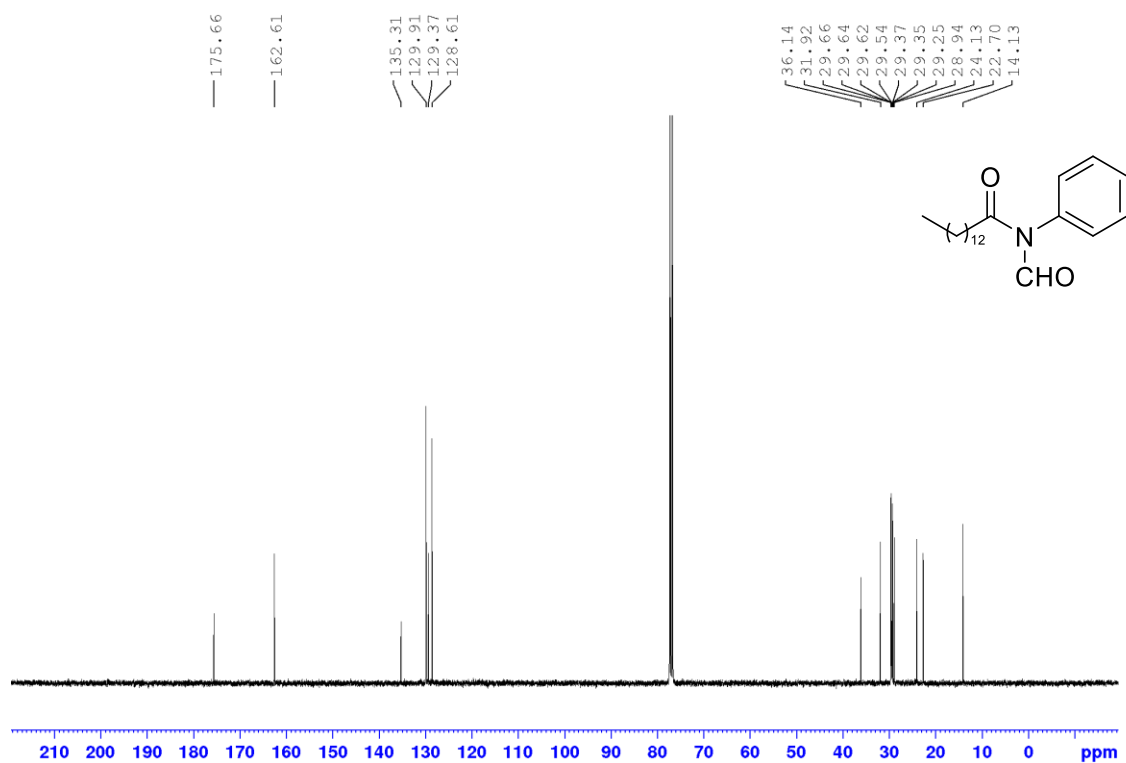


^{13}C $\{^1\text{H}\}$ NMR of **3f**. (100 MHz, CDCl_3)

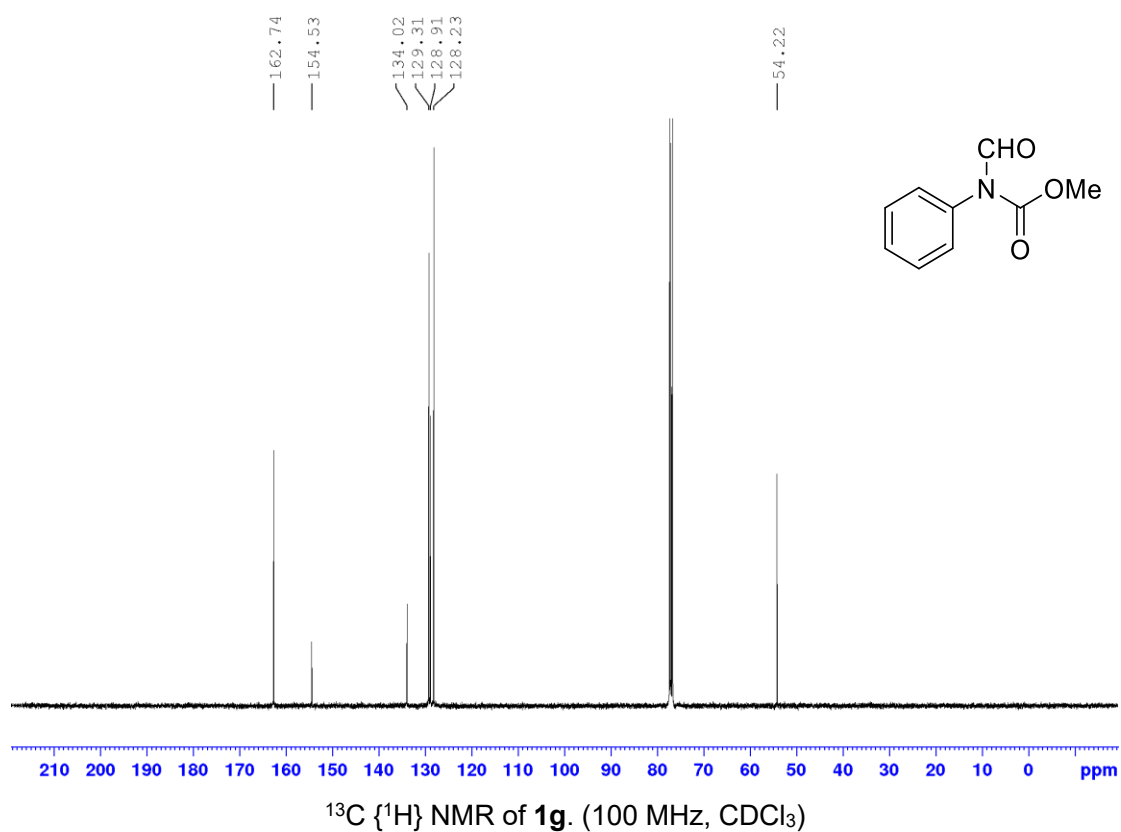
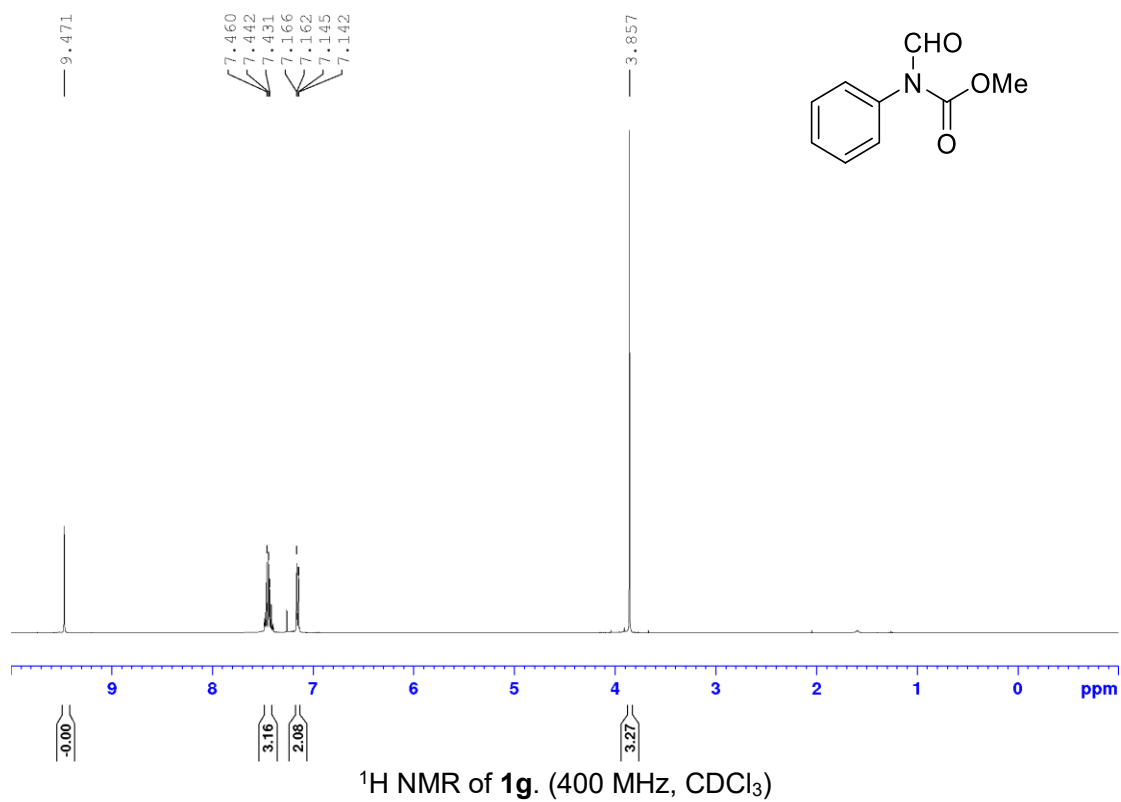


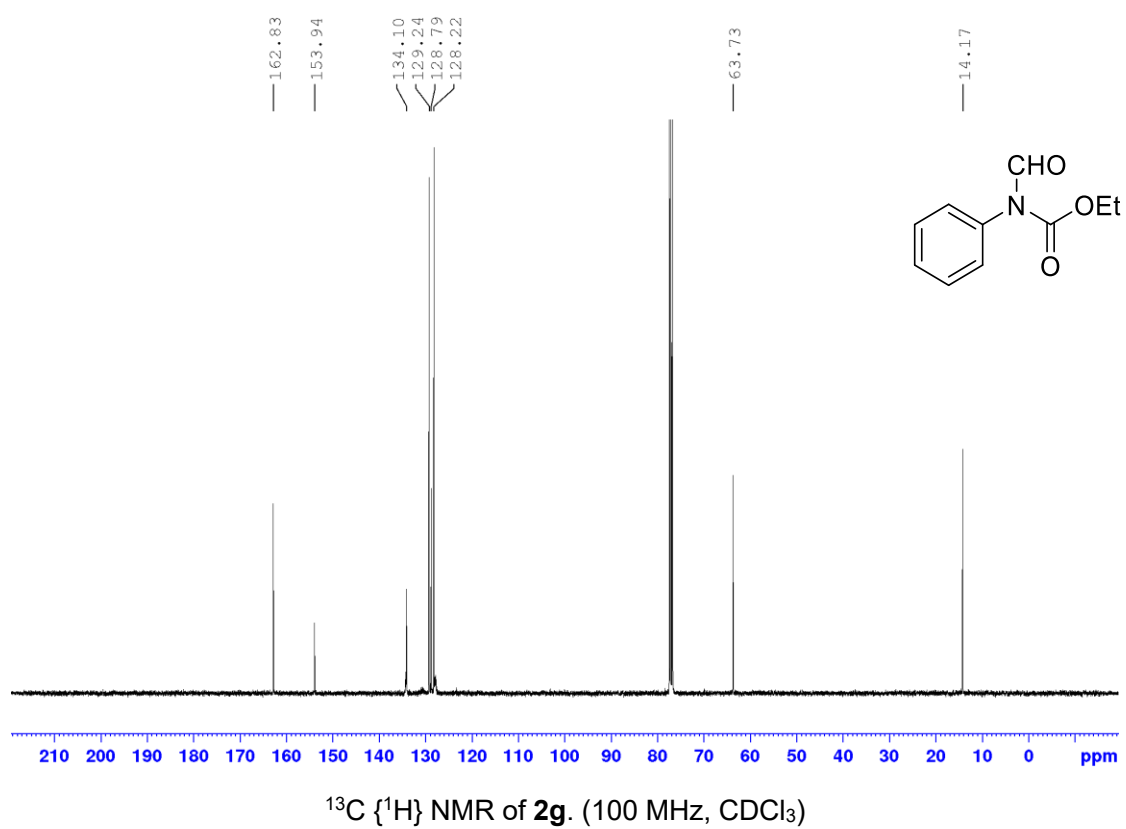
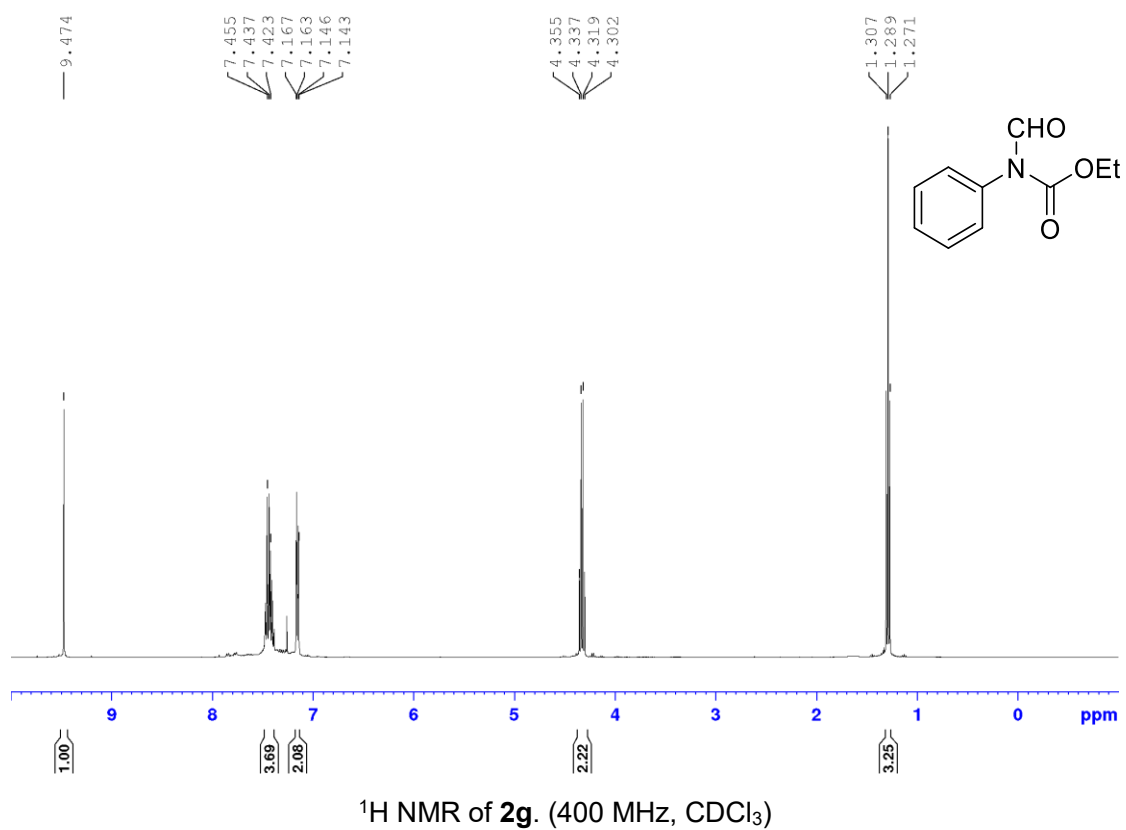


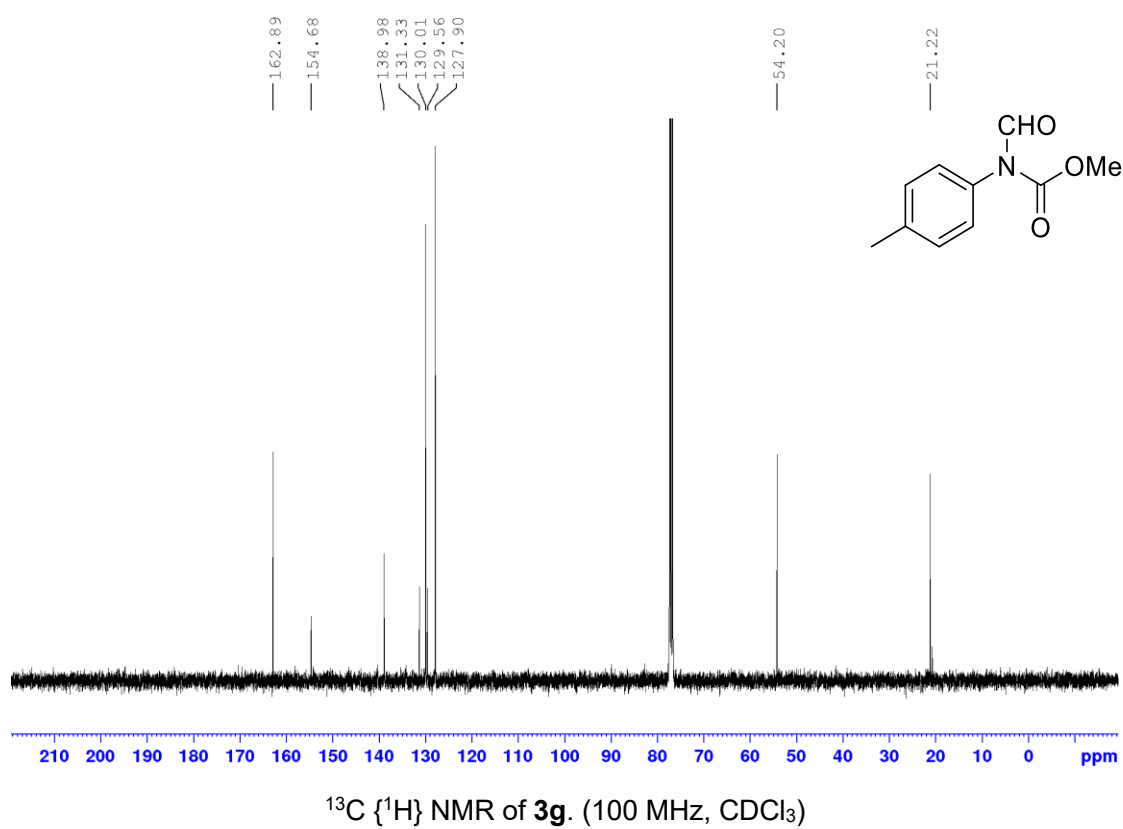
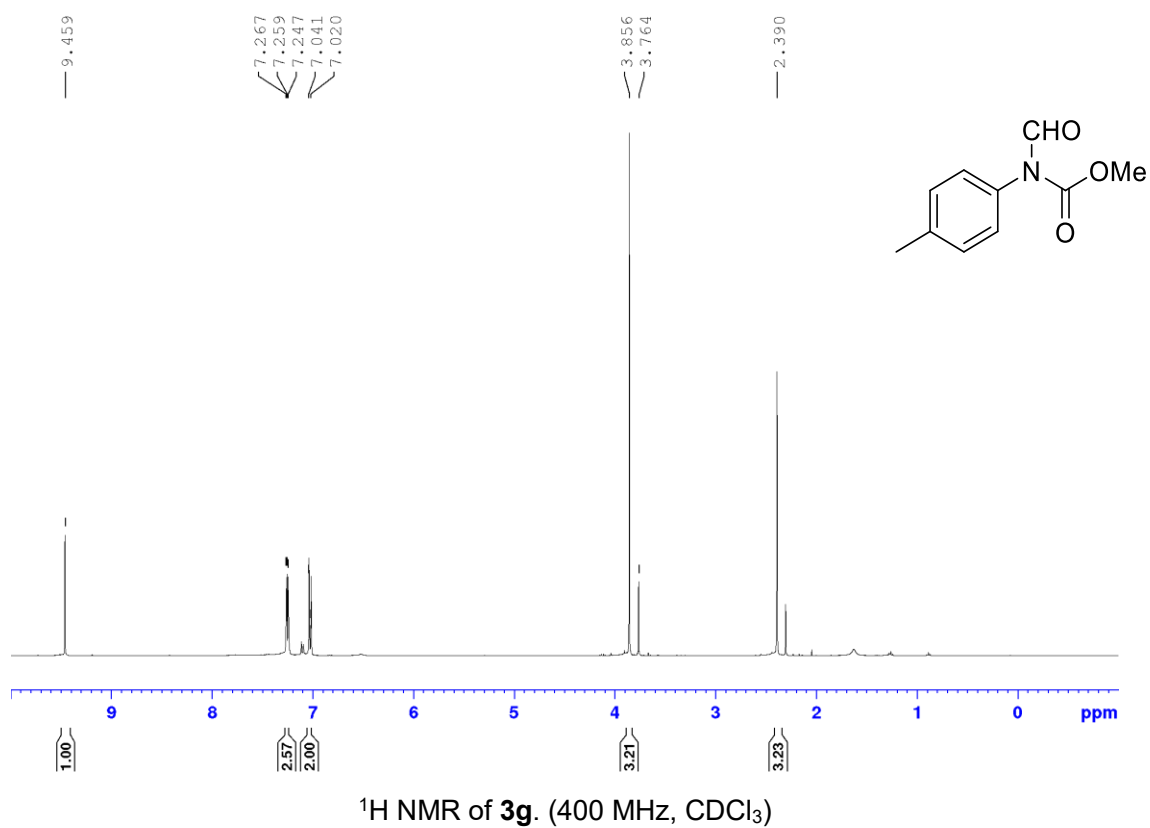
¹H NMR of **5f**. (400 MHz, CDCl₃)

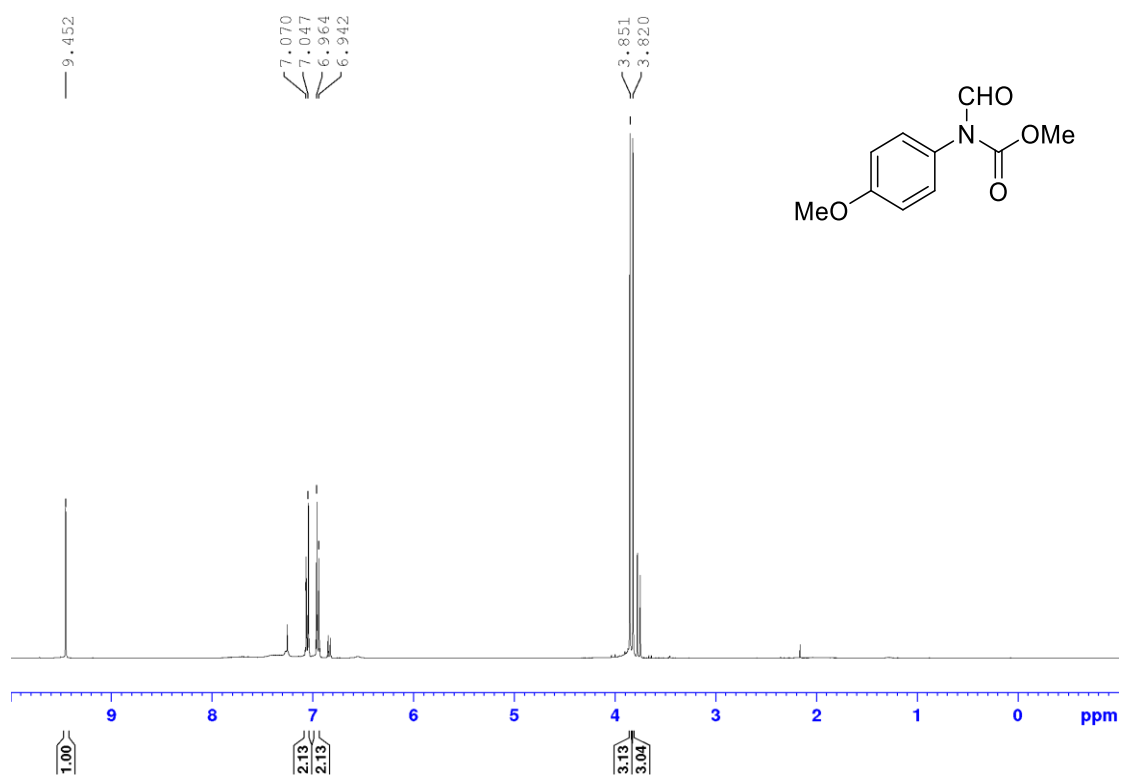


¹³C {¹H} NMR of **5f**. (100 MHz, CDCl₃)

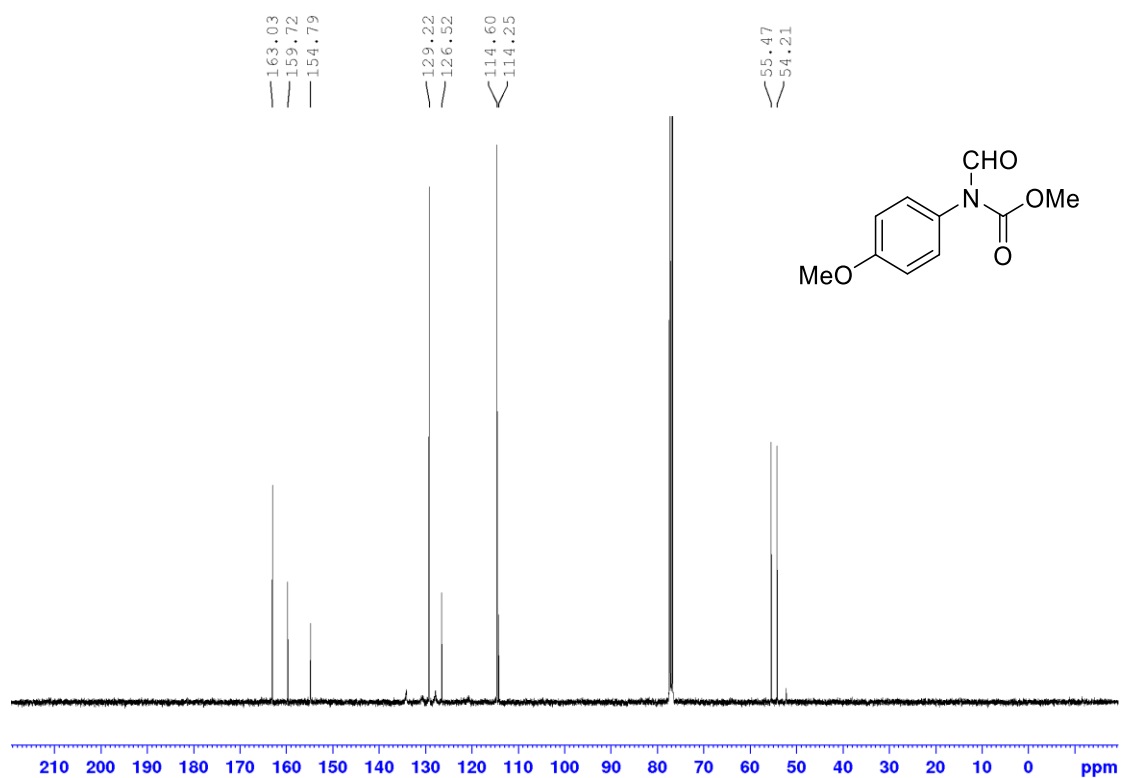




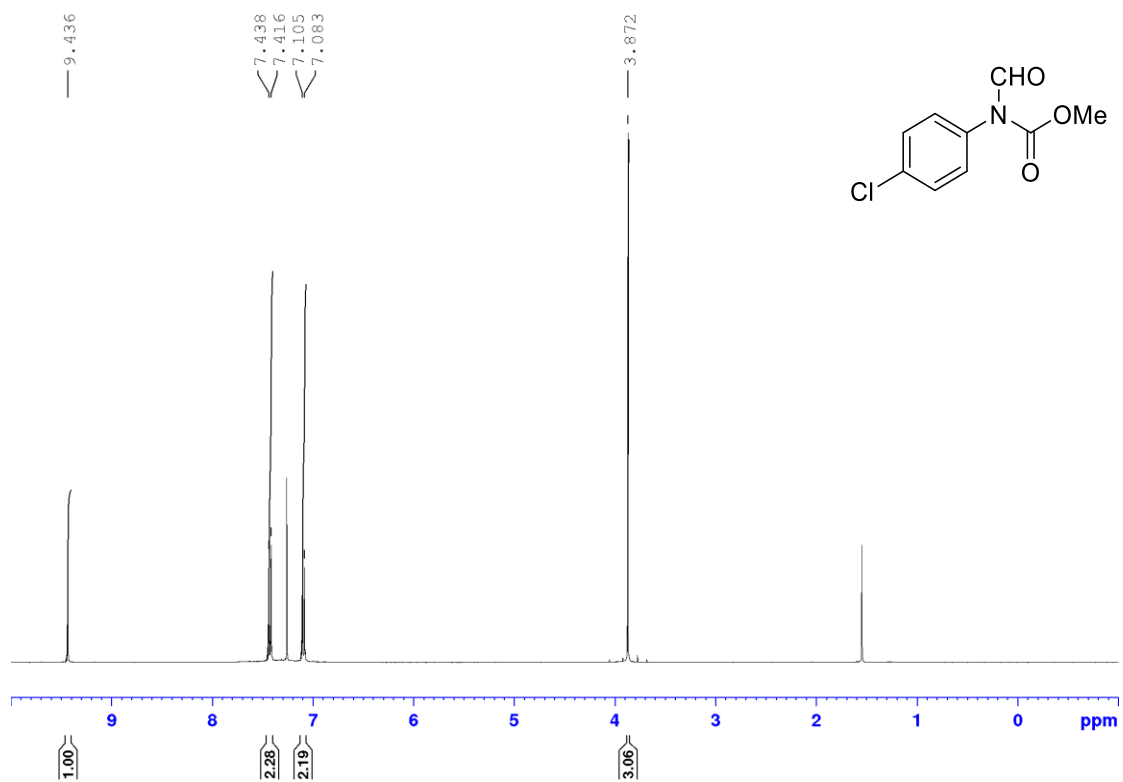




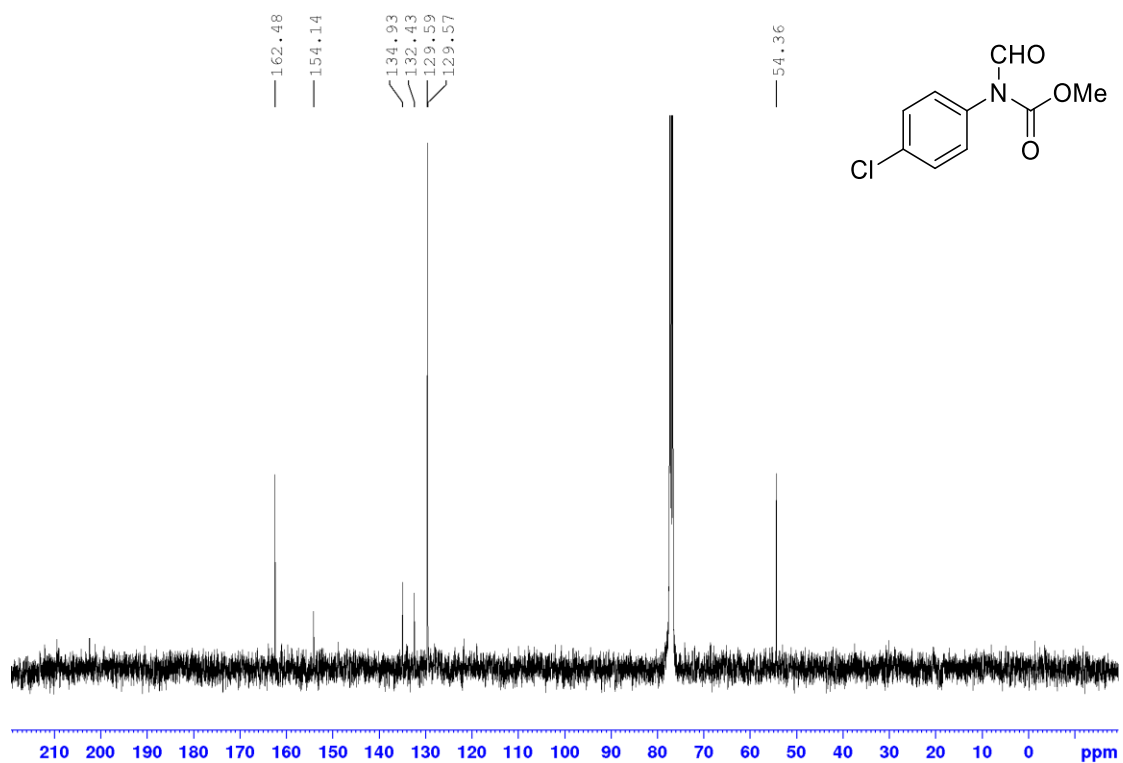
¹H NMR of **4g**. (400 MHz, CDCl₃)



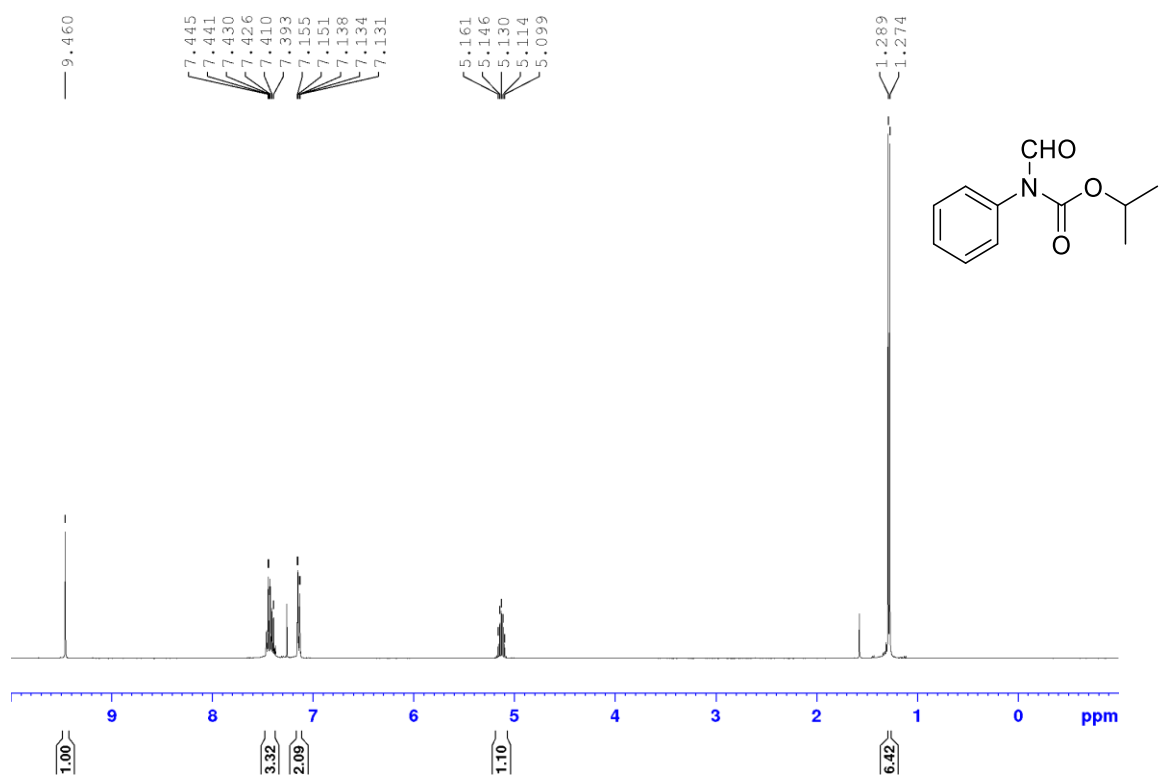
¹³C {¹H} NMR of **4g**. (100 MHz, CDCl₃)



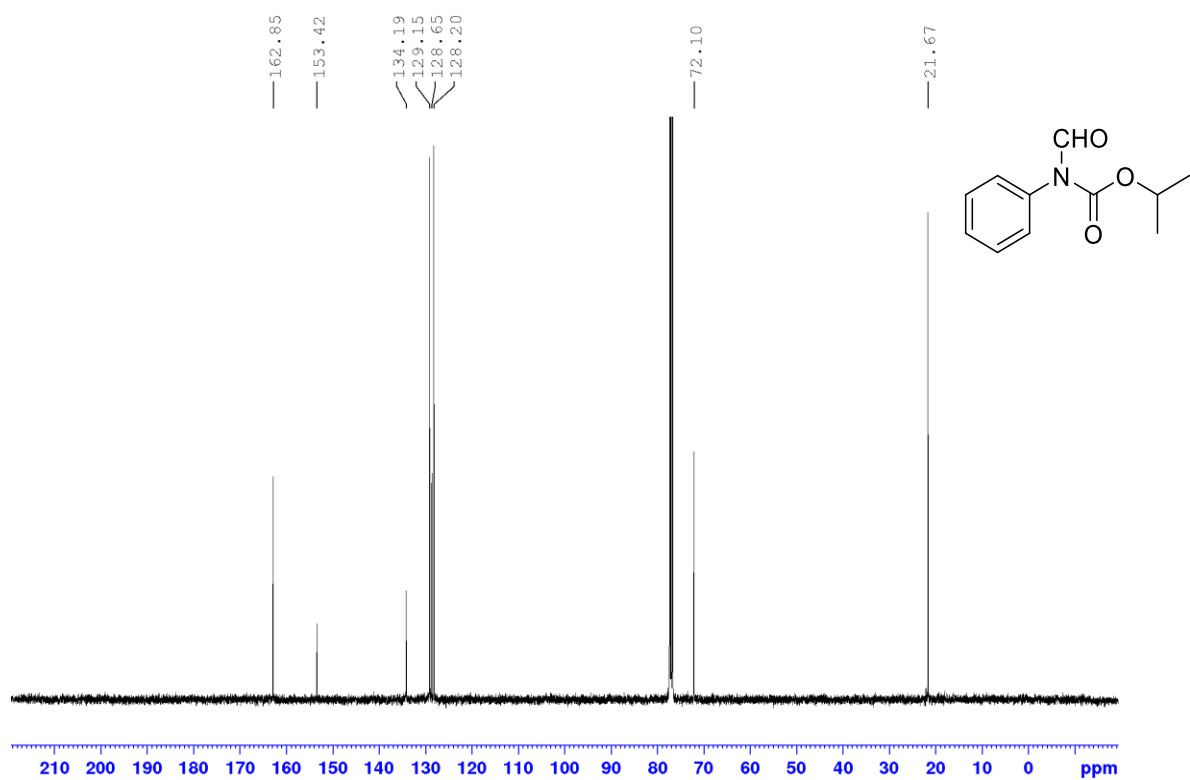
^1H NMR of **5g**. (400 MHz, CDCl_3)



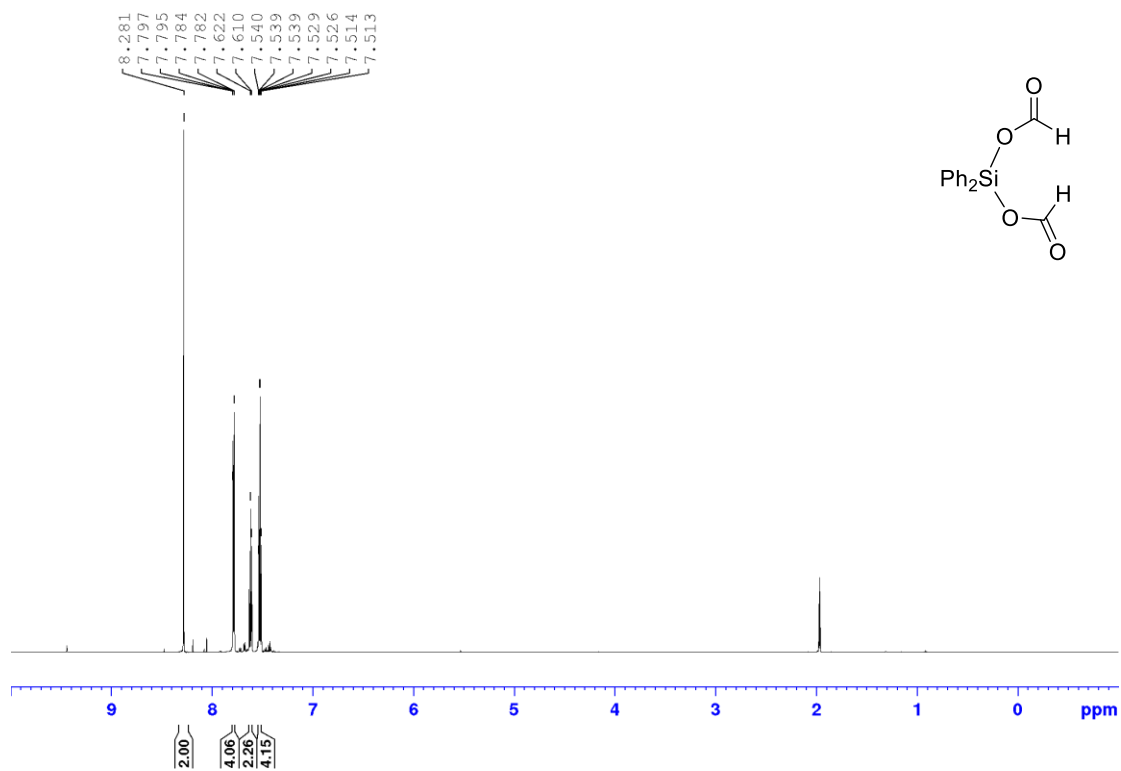
^{13}C $\{^1\text{H}\}$ NMR of **5g**. (100 MHz, CDCl_3)



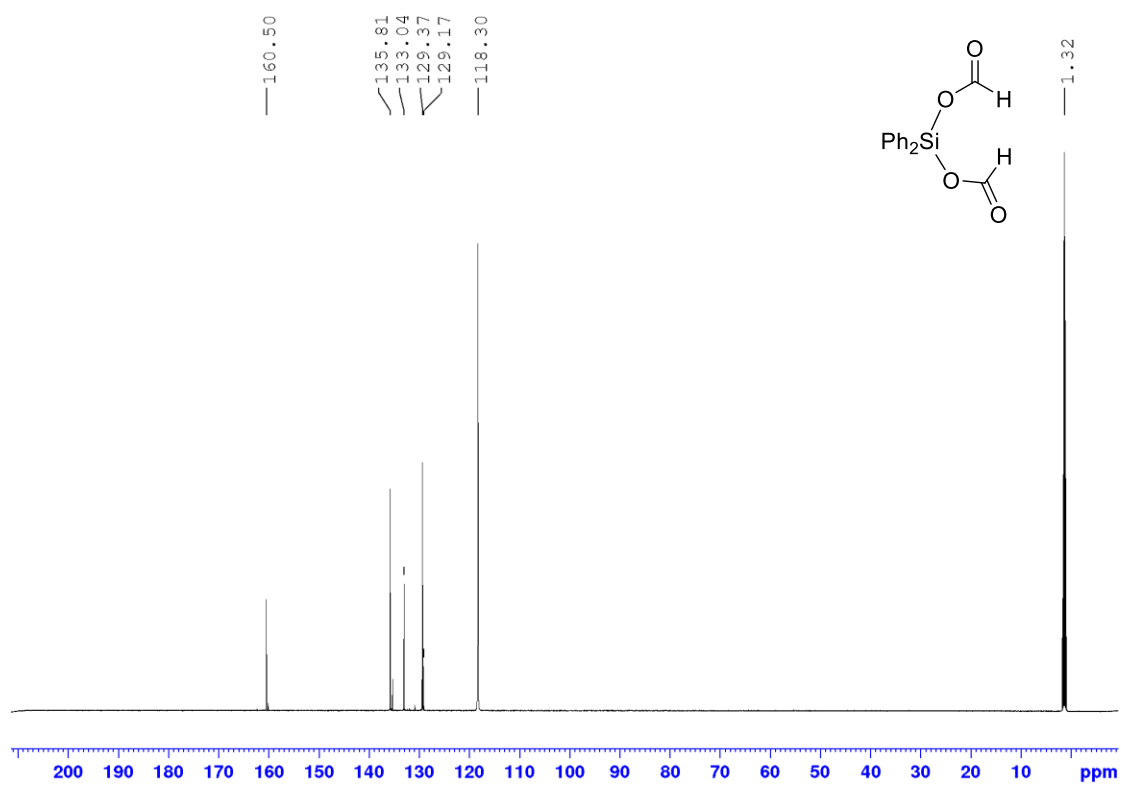
¹H NMR of **6g**. (400 MHz, CDCl₃)



¹³C {¹H} NMR of **6g**. (100 MHz, CDCl₃)



¹H NMR of $\text{Ph}_2\text{Si}(\text{OCHO})_2$. (400 MHz, CD_3CN)



¹³C {¹H} NMR of $\text{Ph}_2\text{Si}(\text{OCHO})_2$. (100 MHz, CD_3CN)

