Electronic Supplementary Material (ESI) for RSC Advances

Metal-Free Pinnick-type Oxidative Amidation of Aldehydes

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Supporting Information

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1. General Experimental

^{1}H and ^{13}C NMR spectra were recorded on a Bruker ACF300 (300MHz) or Bruker DPX300 (300MHz) spectrometer. Chemical shifts are reported in parts per million (ppm). The residual solvent peak was used as an internal reference. Low resolution mass spectra were obtained on a Finnigan/MAT LCQ spectrometer in ESI mode. High resolution mass spectra were obtained on a Finnigan/MAT 95XL-T spectrometer. Enantiomeric excess values were determined by chiral HPLC analysis on Dionex Ultimate 3000 HPLC units, including a Ultimate 3000 Pump, Ultimate 3000 variable Detectors. Melting points were determined on a BÜCHI B-540 melting point apparatus. Flash chromatography separations were performed on Merck 60 (0.040 - 0.063mm) mesh silica gel. Toluene was distilled from sodium/benzophenone and stored under N_2 atmosphere. MeCN was dried by Molecular Sieve. Other reagents and solvents were commercial grade and were used as supplied without further purification, unless otherwise stated. All experiments were monitored by analytical thin layer chromatography (TLC). Instrumentations: Proton nuclear magnetic resonance (^{1}H NMR) and carbon NMR (^{13}C NMR) spectra were recorded in $CDCl_3$ unless otherwise stated.

2. General Procedure

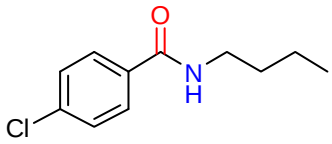
Aldehyde (0.10 mmol, 1 equiv), amine (0.15 mmol, 1.5 equiv) and 2,3-dimethylbut-2-ene (0.50 mmol, 5 equiv) were added into toluene (0.5 mL) and allowed to stir for 5 minutes. $NaClO_2$ (0.3125 mmol, 3.125 equiv) and NaH_2PO_4 (0.35 mmol, 3.5 equiv) were then added to the reaction mixture. The reaction mixture was allowed to stir at 40 ^{o}C for 15 – 26 hours and monitored by TLC. After the reaction was completed, the reaction mixture was diluted with anhydrous diethyl ether (5 mL), followed by adding saturated K_2CO_3 (5 mL). The aqueous layer was extracted with anhydrous diethyl ether (3 × 10 mL). The combined organic layers were dried over anhydrous sodium sulphate and the solvent was removed *in vacuo*. The residue was purified by flash chromatography (gradient elution with hexane/ethyl acetate 20/1 to 2/1).

3. Procedure for Gram-Scale Synthesis of Amide 22

Ethyl glyoxylate (ca. 50% soln. in toluene) (1.98 mL, 10.0 mmol, 1 equiv) and n-butylamine (1.48 mL, 15.0 mmol, 1.5 equiv) and 2,3-dimethylbut-2-ene (5.94 mL, 50.0 mmol, 5 equiv) were added into toluene (50 mL) in a 100 mL round bottom flask and allowed to stir for 5 minutes. $NaClO_2$ (2.826 g, 31.25 mmol, 3.125 equiv) and NaH_2PO_4 (4.83 g, 35.0 mmol, 3.5 equiv) were then added to the reaction mixture. The reaction mixture was allowed to stir at 40 ^{o}C for 18 hours. After the reaction was completed, the reaction mixture was diluted with anhydrous diethyl ether (50 mL), followed by adding saturated K_2CO_3 (50 mL). The aqueous layer was extracted with anhydrous diethyl ether (3 × 50 mL). The combined organic layers were dried over anhydrous sodium sulphate and the solvent was removed *in vacuo*. The residue was purified by flash chromatography (gradient elution with hexane/ethyl acetate 15/1 to 2/1), providing amide **22** (1.52 g, 88%) as a pale yellow oil.

4. Characterization of Products

Amide **1**: N-butyl-4-chlorobenzamide

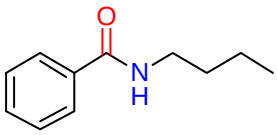


Following the above general procedure with 4-chlorobenzaldehyde (14.1 mg, 0.10 mmol, 1 equiv) and n-butylamine (14.8 µL, 0.15 mmol, 1.5 equiv), the crude reaction mixture was purified by flash chromatography to provide amide **1** (20.3 mg, 96%) as a white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.69 (d, *J* = 8.5 Hz, 2H), 7.37 (d, *J* = 8.4 Hz, 2H), 6.24 (s, 1H), 3.43 (dd, *J* = 13.3, 6.6 Hz, 2H), 1.63 – 1.53 (m, 2H), 1.40 – 1.35 (m, 2H), 0.94 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 166.47, 137.47, 133.16, 128.72, 128.27, 39.88, 31.64, 20.11, 13.73.

LRMS (ESI) m/z 212.1 ($M + H^+$)
HRMS (ESI) m/z 212.0841 ([$M + H^+$]), calc. for [$C_{11}H_{14}ClNO + H^+$] 212.0837.

Amide **2**: N-butylbenzamide

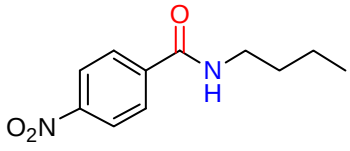


Following the above general procedure with benzaldehyde (10.2 µL, 0.10 mmol, 1 equiv) and n-butylamine (14.8 µL, 0.15 mmol, 1.5 equiv), the crude reaction mixture was purified by flash chromatography to provide amide **2** (14.2 mg, 80%) as a pale yellow oil.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.75 (dd, *J* = 8.2, 1.4 Hz, 2H), 7.51 – 7.37 (m, 3H), 6.24 (s, 1H), 3.44 (dd, *J* = 12.9, 7.0 Hz, 2H), 1.64 – 1.54 (m, 2H), 1.46 – 1.34 (m, 2H), 0.95 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 167.55, 134.82, 131.26, 128.49, 126.80, 39.78, 31.70, 20.12, 13.74.

LRMS (ESI) m/z 178.1 ($M + H^+$)
HRMS (ESI) m/z 178.1227 ([$M + H^+$]), calc. for [$C_{11}H_{15}NO + H^+$] 178.1226.

Amide **3**: N-butyl-4-nitrobenzamide

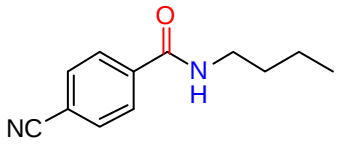


Following the above general procedure with 4-nitrobenzaldehyde (15.1 mg, 0.10 mmol, 1 equiv) and n-butylamine (14.8 µL, 0.15 mmol, 1.5 equiv), the crude reaction mixture was purified by flash chromatography to provide amide **3** (20.0 mg, 90%) as a white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.25 (d, *J* = 8.6 Hz, 2H), 7.92 (d, *J* = 8.7 Hz, 2H), 6.41 (s, 1H), 3.46 (dd, *J* = 13.0, 7.1 Hz, 2H), 1.66 – 1.56 (m, 2H), 1.47 – 1.34 (m, 2H), 0.95 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 165.49, 149.44, 140.41, 128.04, 123.73, 40.13, 31.52, 20.09, 13.69.

LRMS (ESI) m/z 244.9 ($M + Na^+$)
HRMS (ESI) m/z 245.0905 ([$M + Na^+$]), calc. for [$C_{11}H_{14}N_2O_3 + Na^+$] 245.0897.

Amide **4**: N-butyl-4-cyanobenzamide

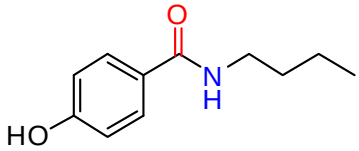


Following the above general procedure with 4-cyanobenzaldehyde (13.1 mg, 0.10 mmol, 1 equiv) and n-butylamine (14.8 µL, 0.15 mmol, 1.5 equiv), the crude reaction mixture was purified by flash chromatography to provide amide **4** (19.4 mg, 96%) as a white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.86 (d, *J* = 8.6 Hz, 2H), 7.70 (d, *J* = 8.5 Hz, 2H), 6.39 (s, 1H), 3.44 (dd, *J* = 12.9, 7.1 Hz, 2H), 1.64 – 1.54 (m, 2H), 1.45 – 1.33 (m, 2H), 0.94 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 165.71, 138.72, 132.34, 127.58, 117.99, 114.82, 40.03, 31.51, 20.07, 13.68.

LRMS (ESI) m/z 203.1 ($M + H^+$)
HRMS (ESI) m/z 203.1181 ([$M + H^+$]), calc. for [$C_{12}H_{14}N_2O + H^+$] 203.1179.

Amide **5**: N-butyl-4-hydroxybenzamide

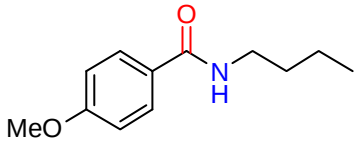


Following the above general procedure with 4-hydroxybenzaldehyde (12.2 mg, 0.10 mmol, 1 equiv) and n-butylamine (14.8 µL, 0.15 mmol, 1.5 equiv) in Toluene:EA (4:1) mixture as the solvent, the crude reaction mixture was purified by flash chromatography to provide amide **5** (15.6 mg, 81%) as a colourless oil.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.61 (d, *J* = 8.7 Hz, 2H), 6.86 (d, *J* = 8.7 Hz, 2H), 6.19 (s, 1H), 3.44 (dd, *J* = 12.8, 7.0 Hz, 2H), 1.64 – 1.54 (m, 2H), 1.46 – 1.34 (m, 2H), 0.94 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 168.33, 160.06, 128.75, 115.59, 39.97, 31.61, 20.10, 13.73.

LRMS (ESI) m/z 194.1 ($M + H^+$)
HRMS (ESI) m/z 194.1179 ([$M + H^+$]), calc. for [$C_{11}H_{15}NO_2 + H^+$] 194.1176.

Amide **6**: N-butyl-4-methoxybenzamide

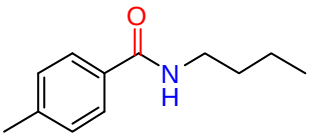


Following the above general procedure with 4-methoxybenzaldehyde (12.2 µL, 0.10 mmol, 1 equiv) and n-butylamine (14.8 µL, 0.15 mmol, 1.5 equiv), the crude reaction mixture was purified by flash chromatography to provide amide **6** (15.5 mg, 75%) as a light brown crystalline solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.72 (d, *J* = 8.9 Hz, 2H), 6.90 (d, *J* = 8.9 Hz, 2H), 6.19 (s, 1H), 3.83 (s, 3H), 3.42 (dd, *J* = 12.2, 7.0 Hz, 2H), 1.63 – 1.53 (m, 2H), 1.45 – 1.33 (m, 2H), 0.94 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 167.07, 162.01, 128.59, 127.05, 113.66, 55.34, 39.74, 31.77, 20.13, 13.75.

LRMS (ESI) m/z 208.1 ($M + H^+$)
HRMS (ESI) m/z 208.1338 ([$M + H^+$]), calc. for [$C_{12}H_{17}NO_2 + H^+$] 208.1332.

Amide **7**: N-butyl-4-methylbenzamide

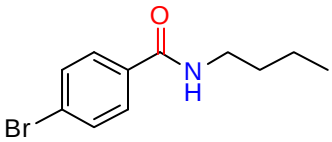


Following the above general procedure with 4-methylbenzaldehyde (11.8 μL, 0.10 mmol, 1 equiv) and n-butylamine (14.8 μL, 0.15 mmol, 1.5 equiv), the crude reaction mixture was purified by flash chromatography to provide amide **7** (16.4 mg, 86%) as a pale brown oil.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.65 (d, *J* = 8.1 Hz, 2H), 7.21 (d, *J* = 8.3 Hz, 2H), 6.19 (s, 1H), 3.43 (dd, *J* = 13.1, 6.7 Hz, 2H), 2.38 (s, 3H), 1.58 – 1.53 (m, 2H), 1.46 – 1.34 (m, 2H), 0.94 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 167.49, 141.63, 131.92, 129.13, 126.79, 39.72, 31.72, 21.37, 20.12, 13.75.

LRMS (ESI) m/z 192.1 ($M + H^+$)
HRMS (ESI) m/z 192.1387 ([$M + H^+$]), calc. for [$C_{12}H_{17}NO + H^+$] 192.1383.

Amide **8**: 4-bromo-N-butylbenzamide

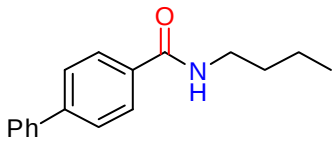


Following the above general procedure with 4-bromobenzaldehyde (18.5 mg, 0.10 mmol, 1 equiv) and n-butylamine (14.8 μL, 0.15 mmol,1.5 equiv), the crude reaction mixture was purified by flash chromatography to provide amide **8** (18.2 mg, 71%) as an off-white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.62 (d, *J* = 8.6 Hz, 2H), 7.54 (d, *J* = 8.6 Hz, 2H), 6.21 (s, 1H), 3.43 (dd, *J* = 12.8, 7.1 Hz, 2H), 1.63 – 1.54 (m, 2H), 1.46 – 1.33 (m, 2H), 0.95 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 166.55, 133.64, 131.72, 128.46, 125.91, 39.89, 31.65, 20.12, 13.73.

LRMS (ESI) m/z 256.1 ($M + H^+$)
HRMS (ESI) m/z 256.0336 ([$M + H^+$]), calc. for [$C_{11}H_{14}BrNO + H^+$] 256.0332.

Amide **9**: N-butyl-[1,1'-biphenyl]-4-carboxamide

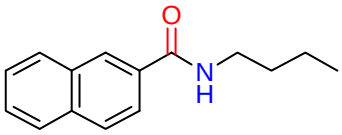


Following the above general procedure with 4-phenylbenzaldehyde (18.2 mg, 0.10 mmol, 1 equiv) and n-butylamine (14.8 μL, 0.15 mmol, 1.5 equiv), the crude reaction mixture was purified by flash chromatography to provide amide **9** (18.2 mg, 72%) as a white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.84 (d, *J* = 8.3 Hz, 2H), 7.70 – 7.54 (m, 4H), 7.52 – 7.32 (m, 3H), 6.28 (s, 1H), 3.48 (dd, *J* = 12.7, 7.0 Hz, 2H), 1.67 – 1.55 (m, 2H), 1.49 – 1.37 (m, 2H), 0.97 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 167.25, 144.10, 140.02, 133.45, 128.87, 127.92, 127.35, 127.16, 39.83, 31.74, 20.15, 13.76.

LRMS (ESI) m/z 254.1 ($M + H^+$)
HRMS (ESI) m/z 254.1544 ([$M + H^+$]), calc. for [$C_{17}H_{19}NO + H^+$] 254.1539.

Amide **10**: N-butyl-2-naphthamide

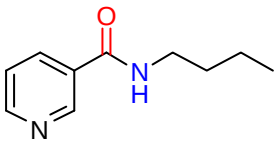


Following the above general procedure with 2-naphthaldehyde (15.6 mg, 0.10 mmol, 1 equiv) and n-butylamine (14.8 µL, 0.15 mmol, 1.5 equiv), the crude reaction mixture was purified by flash chromatography to provide amide **10** (18.6 mg, 82%) as an off-white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.27 (s, 1H), 7.98 – 7.75 (m, 4H), 7.63 – 7.44 (m, 2H), 6.43 (s, 1H), 3.50 (dd, *J* = 12.7, 7.0 Hz, 2H), 1.69 – 1.59 (m, 2H), 1.50 – 1.37 (m, 2H), 0.97 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 167.60, 134.62, 132.60, 132.02, 128.83, 128.35, 127.68, 127.49, 127.20, 126.65, 123.56, 39.93, 31.75, 20.16, 13.76.

LRMS (ESI) m/z 228.1 ($M + H^+$)
HRMS (ESI) m/z 228.1392 ([$M + H^+$]), calc. for [$C_{15}H_{17}NO + H^+$] 228.1383.

Amide **11**: N-butylnicotinamide

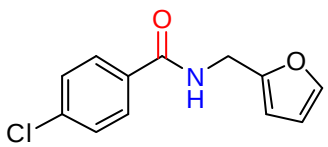


Following the above general procedure with 3-pyridinecarboxaldehyde (9.40 µL, 0.10 mmol, 1 equiv) and n-butylamine (14.8 µL, 0.15 mmol, 1.5 equiv), the crude reaction mixture was purified by flash chromatography to provide amide **11** (14.6 mg, 82%) as a pale yellow oil.

^{1}H NMR (300 MHz, $CDCl_3$) δ 9.05 (s, 1H), 8.68 (d, *J* = 4.9 Hz, 1H), 8.18 (dd, *J* = 7.9, 1.7 Hz, 1H), 7.52 – 7.31 (m, 1H), 6.77 (s, 1H), 3.45 (dd, *J* = 13.5, 6.5 Hz, 2H), 1.65 – 1.55 (m, 2H), 1.46 – 1.33 (m, 2H), 0.94 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 165.22, 151.11, 147.17, 136.01, 130.88, 123.75, 39.95, 31.54, 20.10, 13.69.

LRMS (ESI) m/z 179.2 ($M + H^+$)
HRMS (ESI) m/z 179.1178 ([$M + H^+$]), calc. for [$C_{10}H_{14}N_2O + H^+$] 179.1179.

Amide **12**: 4-chloro-N-(furan-2-ylmethyl)benzamide

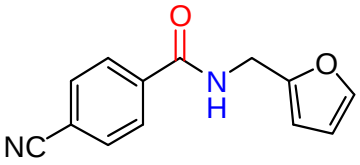


Following the above general procedure with 4-chlorobenzaldehyde (14.1 mg, 0.10 mmol, 1 equiv) and furfurylamine (13.3 µL, 0.15 mmol, 1.5 equiv), the crude reaction mixture was purified by flash chromatography to provide amide **12** (18.9 mg, 80%) as a pale yellow solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.72 (d, *J* = 8.4 Hz, 2H), 7.38 (m, 3H), 6.56 (s, 1H), 6.33 (dd, *J* = 3.1, 1.9 Hz, 1H), 6.28 (d, *J* = 3.2 Hz, 1H), 4.61 (d, *J* = 5.4 Hz, 2H).**^{13}C NMR (75 MHz, $CDCl_3$)** δ 166.17, 150.90, 142.35, 137.85, 132.48, 128.79, 128.44, 110.52, 107.81, 37.03.

LRMS (ESI) m/z 235.9 ($M + H^+$)
HRMS (ESI) m/z 236.0476 ([$M + H^+$]), calc. for [$C_{12}H_{10}ClNO_2 + H^+$] 236.0473.

Amide **13**: 4-cyano-N-(furan-2-ylmethyl)benzamide

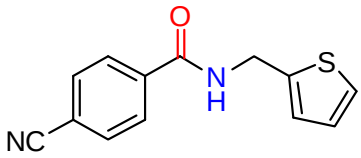


Following the above general procedure with 4-cyanobenzaldehyde (13.1 mg, 0.10 mmol, 1 equiv), furfurylamine (13.3 µL, 0.15 mmol, 1.5 equiv), $NaClO_2$ (45.2 mg, 0.50 mmol, 5 equiv) and NaH_2PO_4 (55.2 mg, 0.40 mmol, 4 equiv), the crude reaction mixture was purified by flash chromatography to provide amide **13** (19.9 mg, 88%) as a pale yellow solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.88 (d, *J* = 8.3 Hz, 2H), 7.71 (d, *J* = 8.0 Hz, 2H), 7.41 – 7.34 (m, 1H), 6.68 (s, 1H), 6.36 – 6.32 (m, 1H), 6.30 (d, *J* = 3.2 Hz, 1H), 4.63 (d, *J* = 5.5 Hz, 2H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 165.42, 150.46, 142.48, 138.02, 132.40, 127.75, 117.92, 115.16, 110.57, 108.07, 37.13.

LRMS (ESI) m/z 225.2 ($M - H^+$)
HRMS (ESI) m/z 225.0658 ([$M - H^+$]), calc. for [$C_{13}H_{10}N_2O_2 - H^+$] 256.0670.

Amide **14**: 4-cyano-N-(thiophen-2-ylmethyl)benzamide

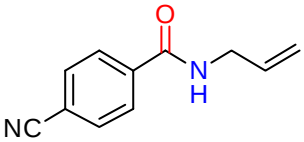


Following the above general procedure with 4-cyanobenzaldehyde (13.1 mg, 0.10 mmol, 1 equiv), 2-thiophene methylamine (15.4 µL, 0.15 mmol, 1.5 equiv), $NaClO_2$ (45.2 mg, 0.50 mmol, 5 equiv) and NaH_2PO_4 (55.2 mg, 0.40 mmol, 4 equiv), the crude reaction mixture was purified by flash chromatography to provide amide **14** (18.9 mg, 78%) as an off-white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.87 (d, *J* = 8.3 Hz, 2H), 7.71 (d, *J* = 8.1 Hz, 2H), 7.34 – 7.17 (m, 1H), 7.04 (d, *J* = 3.4 Hz, 1H), 6.97 (dd, *J* = 5.0, 3.5 Hz, 1H), 6.64 (s, 1H), 4.81 (d, *J* = 5.6 Hz, 2H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 165.34, 139.91, 138.00, 132.44, 127.72, 127.06, 126.58, 125.64, 117.91, 115.22, 38.97.

LRMS (ESI) m/z 241.2 ($M - H^+$)
HRMS (ESI) m/z 241.0430 ([$M - H^+$]), calc. for [$C_{13}H_{10}N_2OS - H^+$] 241.0441.

Amide **15**: N-allyl-4-cyanobenzamide

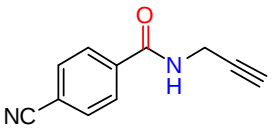


Following the above general procedure with 4-cyanobenzaldehyde (13.1 mg, 0.10 mmol, 1 equiv) and allylamine (11.2 µL, 0.15 mmol, 1.5 equiv), the crude reaction mixture was purified by flash chromatography to provide amide **15** (15.8 mg, 85%) as a white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.88 (d, *J* = 8.5 Hz, 2H), 7.72 (d, *J* = 8.1 Hz, 2H), 6.45 (s, 1H), 5.93 – 5.85 (m, 1H), 5.28 – 5.18 (m, 2H), 4.08 (t, *J* = 5.7 Hz, 2H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 165.52, 138.32, 133.46, 132.41, 127.65, 117.94, 117.21, 115.05, 42.62.

LRMS (ESI) m/z 185.2 ($M - H^+$)
HRMS (ESI) m/z 185.0718 ([$M - H^+$]), calc. for [$C_{11}H_{10}N_2O - H^+$] 185.0720.

Amide **16**: 4-cyano-N-(prop-2-yn-1-yl)benzamide

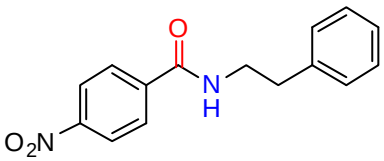


Following the above general procedure with 4-cyanobenzaldehyde (13.1 mg, 0.10 mmol, 1 equiv), prop-2-yn-1-amine (10.3 µL, 0.15 mmol, 1.5 equiv), $NaClO_2$ (45.2 mg, 0.50 mmol, 5 equiv) and NaH_2PO_4 (55.2 mg, 0.40 mmol, 4 equiv), the crude reaction mixture was purified by flash chromatography to provide amide **16** (16.5 mg, 90%) as a pale yellow solid.

^{1}H NMR (300 MHz, CD_3OD) δ 7.90 (d, *J* = 8.5 Hz, 2H), 7.77 (d, *J* = 8.5 Hz, 2H), 4.10 (d, *J* = 2.5 Hz, 2H), 2.57 (t, *J* = 2.5 Hz, 1H). **^{13}C NMR (75 MHz, CD_3OD)** δ 167.91, 139.31, 133.55, 129.26, 119.00, 116.24, 80.38, 72.33, 30.08.

LRMS (ESI) m/z 183.1 ($M - H^+$)
HRMS (ESI) m/z 183.0558 ([$M - H^+$]), calc. for [$C_{11}H_8N_2O - H^+$] 183.0564.

Amide **17**: 4-nitro-N-phenethylbenzamide

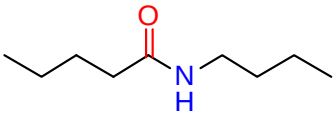


Following the above general procedure with 4-nitrobenzaldehyde (15.1 mg, 0.10 mmol, 1 equiv), phenethylamine (18.9 µL, 0.15 mmol, 1.5 equiv), the crude reaction mixture was purified by flash chromatography to provide amide **17** (22.7 mg, 84%) as an off-white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.22 (d, *J* = 8.7 Hz, 2H), 7.82 (d, *J* = 8.8 Hz, 2H), 7.31 (d, *J* = 7.4 Hz, 2H), 7.25 – 7.19 (m, 3H), 6.37 (s, 1H), 3.72 (dd, *J* = 12.9, 6.8 Hz, 2H), 2.94 (t, *J* = 6.9 Hz, 2H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 165.45, 149.48, 140.16, 138.45, 128.77, 128.71, 127.99, 126.76, 123.75, 41.35, 35.42.

LRMS (ESI) m/z 269.3 ($M - H^+$)
HRMS (ESI) m/z 269.0921 ([$M - H^+$]), calc. for [$C_{15}H_{14}N_2O_3 - H^+$] 269.0932.

Amide **18**: N-butylpentanamide

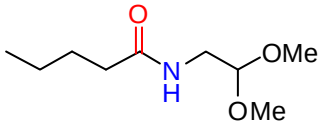


Following the above general procedure with pentanal (10.6 µL, 0.10 mmol, 1 equiv) and n-butylamine (14.8 µL, 0.15 mmol, 1.5 equiv), the crude reaction mixture was purified by flash chromatography to provide amide **18** (10.5 mg, 67%) as a pale yellow oil.

^{1}H NMR (300 MHz, $CDCl_3$) δ 5.80 (s, 1H), 3.22 (dd, *J* = 12.8, 6.8 Hz, 2H), 2.18 – 2.13 (m, 2H), 1.64 – 1.54 (m, 2H), 1.51 – 1.41 (m, 2H), 1.38 – 1.26 (m, 4H), 0.92 – 0.87 (m, 6H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 173.33, 39.22, 36.44, 31.63, 27.90, 22.34, 20.00, 13.72, 13.67.

LRMS (ESI) m/z 158.1 ($M + H^+$),
HRMS (ESI) m/z 158.1544 ([$M + H^+$]), calc. for [$C_9H_{19}NO + H^+$] 158.1539.

Amide **19**: N-(2,2-dimethoxyethyl)pentanamide

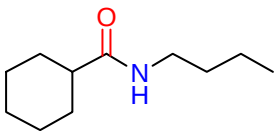


Following the above general procedure with pentanal (10.6 μL, 0.10 mmol, 1 equiv) and 2,2-dimethoxyethylamine (16.3 μL, 0.15 mmol, 1.5 equiv), the crude reaction mixture was purified by flash chromatography to provide amide **19** (17.6 mg, 93%) as a pale brown solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 5.76 (s, 1H), 4.35 (t, *J* = 5.3 Hz, 1H), 3.45 – 3.44 (m, 1H), 3.39 (s, 1H), 3.37 (s, 6H), 2.21 – 2.14 (m, 2H), 1.61 – 1.54 (m, 2H), 1.36 -1.26 (m, 2H), 0.89 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 173.42, 102.65, 54.37, 40.83, 36.33, 27.70, 22.29, 13.70.

LRMS (ESI) m/z 212.0 ($M + Na^+$),
HRMS (ESI) m/z 212.1266 ([$M + Na^+$]), calc. for [$C_9H_{19}NO_3 + Na^+$] 212.1257.

Amide **20**: N-butylcyclohexanecarboxamide

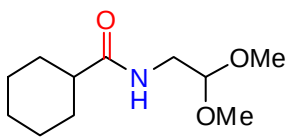


Following the above general procedure with cyclohexanecarboxaldehyde (12.0 μL, 0.10 mmol, 1 equiv) and n-butylamine (14.8 μL, 0.15 mmol, 1.5 equiv), the crude reaction mixture was purified by flash chromatography to provide amide **20** (13.5 mg, 74%) as an off-white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 5.68 (s, 1H), 3.23 (dd, *J* = 12.5, 6.7 Hz, 2H), 2.12 – 2.03 (m, 1H), 1.88 – 1.74 (m, 4H), 1.68 – 1.63 (m, 1H), 1.51 – 1.18 (m, 9H), 0.91 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 176.27, 45.51, 39.10, 31.66, 29.68, 25.71, 20.01, 13.72.

LRMS (ESI) m/z 184.1 ($M + H^+$),
HRMS (ESI) m/z 184.1704 ([$M + H^+$]), calc. for [$C_{11}H_{21}NO + H^+$] 184.1696.

Amide **21**: N-(2,2-dimethoxyethyl)cyclohexanecarboxamide

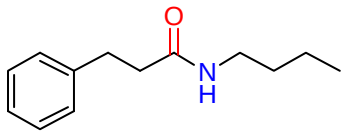


Following the above general procedure with cyclohexanecarboxaldehyde (12.0 μL, 0.10 mmol, 1 equiv) and 2,2-dimethoxyethylamine (16.3 μL, 0.15 mmol, 1.5 equiv), the crude reaction mixture was purified by flash chromatography to provide amide **21** (19.4 mg, 90%) as a white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 5.70 (s, 1H), 4.34 (t, *J* = 5.3 Hz, 1H), 3.50 – 3.42 (m, 1H), 3.37 (s, 6H), 2.11 – 2.02 (m, 1H), 1.87 – 1.71 (m, 4H), 1.70 – 1.60 (m, 1H), 1.46 – 1.35 (m, 2H), 1.32 – 1.14 (m, 4H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 176.28, 102.71, 54.40, 45.38, 40.72, 29.57, 25.65, 25.63.

LRMS (ESI) m/z 216.0 ($M + H^+$),
HRMS (ESI) m/z 216.1590 ([$M + H^+$]), calc. for [$C_{11}H_{21}NO_3 + H^+$] 216.1594.

Amide **22**: N-butyl-3-phenylpropanamide

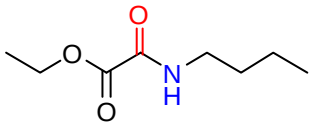


Following the above general procedure with 3-phenylpropanal (13.3 µL, 0.10 mmol, 1 equiv) and n-butylamine (14.8 µL, 0.15 mmol, 1.5 equiv), the crude reaction mixture was purified by flash chromatography to provide amide **22** (12.9 mg, 63%) as a pale yellow oil.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.27 (dd, *J* = 9.1, 5.3 Hz, 2H), 7.23 – 7.16 (m, 3H), 5.77 (s, 1H), 3.20 (dd, *J* = 11.5, 6.6 Hz, 2H), 2.97 (t, *J* = 7.6 Hz, 2H), 2.50 (t, *J* = 7.7 Hz, 2H), 1.49 – 1.34 (m, 2H), 1.31 – 1.18 (m, 2H), 0.88 (t, *J* = 7.2 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 172.43, 140.67, 128.50, 128.34, 126.25, 39.39, 38.32, 31.85, 31.46, 19.93, 13.66.

LRMS (ESI) m/z 206.1 ($M + H^+$),
HRMS (ESI) m/z 206.1547 ([$M + H^+$]), calc. for [$C_{13}H_{19}NO + H^+$] 206.1539.

Amide **23**: Ethyl 2-(butylamino)-2-oxoacetate

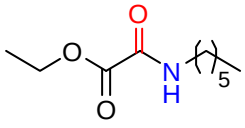


Following the above general procedure with ethyl glyoxylate (ca. 50% soln. in toluene) (19.8 µL, 0.10 mmol, 1 equiv) and n-butylamine (14.8 µL, 0.15 mmol, 1.5 equiv), the crude reaction mixture was purified by flash chromatography to provide amide **23** (16.1 mg, 93%) as a pale yellow oil.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.26 (s, 1H), 4.21 (q, *J* = 7.1 Hz, 2H), 3.22 (dd, *J* = 13.3, 7.1 Hz, 2H), 1.49 – 1.39 (m, 2H), 1.28 – 1.23 (m, 5H), 0.81 (t, *J* = 7.3 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 160.56, 156.39, 62.72, 39.32, 30.82, 19.66, 13.65, 13.32.

LRMS (ESI) m/z 172.1 ($M - H^+$),
HRMS (ESI) m/z 172.0974 ([$M - H^+$]), calc. for [$C_8H_{15}NO_3 - H^+$] 172.0979.

Amide **24**: Ethyl 2-(hexylamino)-2-oxoacetate

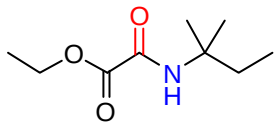


Following the above general procedure with ethyl glyoxylate (ca. 50% soln. in toluene) (19.8 µL, 0.10 mmol, 1 equiv) and n-hexylamine (19.8 µL, 0.15 mmol, 1.5 equiv), the crude reaction mixture was purified by flash chromatography to provide amide **24** (18.9 mg, 94%) as a pale yellow oil.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.13 (s, 1H), 4.32 (q, *J* = 7.1 Hz, 2H), 3.31 (dd, *J* = 6.9, 3.5 Hz, 2H), 1.57 – 1.50 (m, 2H), 1.37 – 1.22 (m, 9H), 0.86 (t, *J* = 6.5 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 160.84, 156.48, 63.09, 39.88, 31.31, 29.03, 26.41, 24.78, 22.43, 13.91.

LRMS (ESI) m/z 202.1 ($M + H^+$)
HRMS (ESI) m/z 212.1440 ([$M + H^+$]), calc. for [$C_{10}H_{19}NO_3 + H^+$] 202.1438.

Amide **25**: Ethyl 2-oxo-2-(tert-pentylamino)acetate

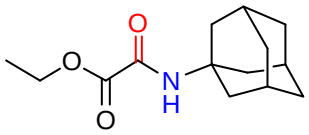


Following the above general procedure with ethyl glyoxylate (ca. 50% soln. in toluene) (19.8 µL, 0.10 mmol, 1 equiv) and tert-pentylamine (17.5 µL, 0.15 mmol, 1.5 equiv), the crude reaction mixture was purified by flash chromatography to provide amide **25** (12.7 mg, 68%) as a pale yellow oil.

^{1}H NMR (300 MHz, $CDCl_3$) δ 6.88 (s, 1H), 4.31 (q, *J* = 7.1 Hz, 2H), 1.75 (q, *J* = 7.5 Hz, 2H), 1.40 – 1.34 (m, 9H), 0.86 (t, *J* = 7.5 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 161.40, 155.44, 63.08, 54.71, 32.54, 25.74, 13.92, 8.24.

LRMS (ESI) m/z 187.9 ($M + H^+$)
HRMS (ESI) m/z 188.1274 ([$M + H^+$]), calc. for [$C_9H_{17}NO_3 + H^+$] 188.1281.

Amide **26**: Ethyl 2-((3s,5s,7s)-adamantan-1-ylamino)-2-oxoacetate

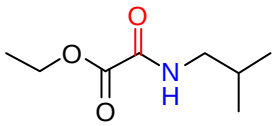


Following the above general procedure with ethyl glyoxylate (ca. 50% soln. in toluene) (19.8 µL, 0.10 mmol, 1 equiv) and 1-adamantylamine (22.7 mg, 0.15 mmol, 1.5 equiv), the crude reaction mixture was purified by flash chromatography to provide amide **26** (17.8 mg, 71%) as a colourless oil.

^{1}H NMR (300 MHz, $CDCl_3$) δ 6.83 (s, 1H), 4.29 (q, *J* = 7.1 Hz, 2H), 2.09 – 2.01 (m, 9H), 1.69 – 1.67 (m, 6H), 1.36 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 161.45, 155.10, 63.00, 52.45, 40.88, 36.10, 29.24, 13.89.

LRMS (ESI) m/z 252.1 ($M + H^+$)
HRMS (ESI) m/z 252.1604 ([$M + H^+$]), calc. for [$C_{14}H_{21}NO_3 + H^+$] 252.1594.

Amide **27**: Ethyl 2-(isobutylamino)-2-oxoacetate

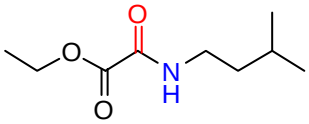


Following the above general procedure with ethyl glyoxylate (ca. 50% soln. in toluene) (19.8 µL, 0.10 mmol, 1 equiv) and isobutylamine (14.8 µL, 0.15 mmol, 1.5 equiv), the crude reaction mixture was purified by flash chromatography to provide amide **27** (14.2 mg, 82%) as a colourless oil.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.16 (s, 1H), 4.34 (q, *J* = 7.1 Hz, 2H), 3.16 (t, *J* = 6.6 Hz, 2H), 1.88 – 1.79 (m, 1H), 1.38 (t, *J* = 7.1 Hz, 3H), 0.93 (d, *J* = 6.7 Hz, 6H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 160.91, 156.59, 63.15, 47.12, 28.28, 19.97, 13.94.

LRMS (ESI) m/z 196.1 ($M + Na^+$)
HRMS (ESI) m/z 196.0942 ([$M + Na^+$]), calc. for [$C_8H_{15}NO_3 + Na^+$] 196.0944.

Amide **28**: Ethyl 2-(isopentylamino)-2-oxoacetate

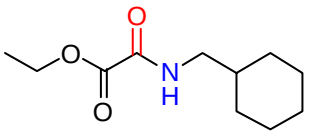


Following the above general procedure with ethyl glyoxylate (ca. 50% soln. in toluene) (19.8 µL, 0.10 mmol, 1 equiv) and isopentylamine (17.4 µL, 0.15 mmol, 1.5 equiv), the crude reaction mixture was purified by flash chromatography to provide amide **28** (15.6 mg, 85%) as a pale yellow oil.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.08 (s, 1H), 4.33 (q, *J* = 7.1 Hz, 2H), 3.34 (dd, *J* = 14.1, 6.7 Hz, 2H), 1.67 – 1.58 (m, 1H), 1.48 – 1.41 (m, 2H), 1.37 (t, *J* = 7.1 Hz, 3H), 0.91 (d, *J* = 6.6 Hz, 6H).**^{13}C NMR (75 MHz, $CDCl_3$)** δ 160.83, 156.46, 63.11, 38.16, 37.88, 25.69, 22.30, 13.93.

LRMS (ESI) m/z 188.0 ($M + H^+$)
HRMS (ESI) m/z 188.1279 ([$M + H^+$]), calc. for [$C_9H_{17}NO_3 + H^+$] 188.1281.

Amide **29**: Ethyl 2-((cyclohexylmethyl)amino)-2-oxoacetate

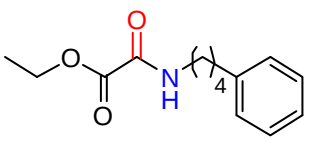


Following the above general procedure with ethyl glyoxylate (ca. 50% soln. in toluene) (19.8 µL, 0.10 mmol, 1 equiv) and cyclohexylmethylamine (19.5 µL, 0.15 mmol, 1.5 equiv), the crude reaction mixture was purified by flash chromatography to provide amide **29** (16.6 mg, 78%) as a colourless oil.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.16 (s, 1H), 4.33 (q, *J* = 7.3 Hz, 2H), 3.17 (t, *J* = 6.6 Hz, 2H), 1.74 – 1.63 (m, 5H), 1.57 – 1.47 (m, 1H), 1.37 (t, *J* = 7.1 Hz, 3H), 1.26 – 1.16 (m, 3H), 1.00 – 0.89 (m, 2H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 160.91, 156.57, 63.13, 45.99, 37.59, 30.65, 26.19, 25.64, 13.94.

LRMS (ESI) m/z 212.0 ($M - H^+$)
HRMS (ESI) m/z 212.1293 ([$M - H^+$]), calc. for [$C_{11}H_{19}NO_3 - H^+$] 212.1292.

Amide **30**: Ethyl 2-oxo-2-((4-phenylbutyl)amino)acetate

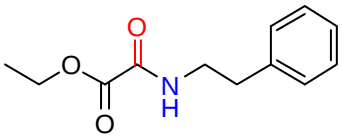


Following the above general procedure with ethyl glyoxylate (ca. 50% soln. in toluene) (19.8 µL, 0.10 mmol, 1 equiv) and 4-phenylbutylamine (23.7 µL, 0.15 mmol, 1.5 equiv), the crude reaction mixture was purified by flash chromatography to provide amide **30** (23.2 mg, 93%) as a pale yellow oil.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.27 (dd, *J* = 9.8, 4.5 Hz, 2H), 7.22 – 7.14 (m, 3H), 4.33 (q, *J* = 7.1 Hz, 2H), 3.35 (q, *J* = 6.7 Hz, 2H), 2.64 (t, *J* = 7.2 Hz, 2H), 1.66 – 1.54 (m, 4H), 1.37 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 160.76, 156.51, 141.74, 128.32, 125.84, 63.11, 39.67, 35.30, 28.62, 28.44, 13.92.

LRMS (ESI) m/z 250.1 ($M + H^+$)
HRMS (ESI) m/z 250.1444 ([$M + H^+$]), calc. for [$C_{14}H_{19}NO_3 + H^+$] 250.1438.

Amide **31**: Ethyl 2-oxo-2-(phenethylamino)acetate

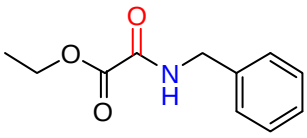


Following the above general procedure with ethyl glyoxylate (ca. 50% soln. in toluene) (19.8 µL, 0.10 mmol, 1 equiv) and phenethylamine (18.9 µL, 0.15 mmol, 1.5 equiv), the crude reaction mixture was purified by flash chromatography to provide amide **31** (20.1 mg, 91%) as a pale yellow oil.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.32 (dd, *J* = 7.4, 4.5 Hz, 2H), 7.25 – 7.18 (m, 3H), 4.32 (q, *J* = 7.1 Hz, 2H), 3.60 (dd, *J* = 13.4, 7.0 Hz, 2H), 2.87 (t, *J* = 7.1 Hz, 2H), 1.36 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 160.61, 156.50, 138.05, 128.72, 128.63, 126.72, 63.13, 40.95, 35.18, 13.91.

LRMS (ESI) m/z 222.1 ($M + H^+$)
HRMS (ESI) m/z 222.1127 ([$M + H^+$]), calc. for [$C_{12}H_{15}NO_3 + H^+$] 222.1125.

Amide **32**: Ethyl 2-(benzylamino)-2-oxoacetate

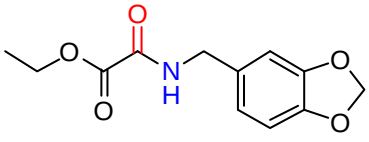


Following the above general procedure with ethyl glyoxylate (ca. 50% soln. in toluene) (19.8 µL, 0.10 mmol, 1 equiv) and benzylamine (16.4 µL, 0.15 mmol, 1.5 equiv), the crude reaction mixture was purified by flash chromatography to provide amide **32** (19.0 mg, 92%) as a pale yellow oil.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.44 (s, 1H), 7.37 – 7.28 (m, 5H), 4.51 (d, *J* = 6.1 Hz, 2H), 4.33 (q, *J* = 7.1 Hz, 2H), 1.37 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 160.57, 156.44, 136.68, 128.78, 127.92, 127.88, 63.21, 43.87, 13.90.

LRMS (ESI) m/z 207.9 ($M + H^+$)
HRMS (ESI) m/z 208.0968 ([$M + H^+$]), calc. for [$C_{11}H_{13}NO_3 + H^+$] 208.0968.

Amide **33**: Ethyl 2-((benzo[d][1,3]dioxol-5-ylmethyl)amino)-2-oxoacetate

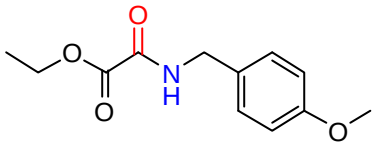


Following the above general procedure with ethyl glyoxylate (ca. 50% soln. in toluene) (19.8 µL, 0.10 mmol, 1 equiv) and piperonylamine (18.7 µL, 0.15 mmol, 1.5 equiv), the crude reaction mixture was purified by flash chromatography to provide amide **33** (17.8 mg, 71%) as a yellow solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.35 (s, 1H), 6.78 – 6.75 (m, 3H), 5.94 (s, 2H), 4.41 (d, *J* = 6.1 Hz, 2H), 4.34 (q, *J* = 7.1 Hz, 2H), 1.37 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 160.60, 156.36, 148.02, 147.34, 130.49, 121.44, 108.55, 108.39, 101.16, 63.26, 43.76, 13.94.

LRMS (ESI) m/z 251.9 ($M + H^+$)
HRMS (ESI) m/z 252.0872 ([$M + H^+$]), calc. for [$C_{12}H_{13}NO_5 + H^+$] 252.0866.

Amide **34**: Ethyl 2-((4-methoxybenzyl)amino)-2-oxoacetate

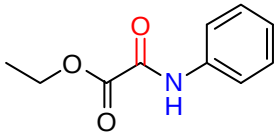


Following the above general procedure with ethyl glyoxylate (ca. 50% soln. in toluene) (19.8 µL, 0.10 mmol, 1 equiv) and 4-methoxybenzylamine (19.6 µL, 0.15 mmol, 1.5 equiv), the crude reaction mixture was purified by flash chromatography to provide amide **34** (21.3 mg, 90%) as a pale yellow solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.35 (s, 1H), 7.22 (d, *J* = 8.7 Hz, 2H), 6.86 (d, *J* = 8.7 Hz, 2H), 4.44 (d, *J* = 6.0 Hz, 2H), 4.33 (q, *J* = 7.1 Hz, 2H), 3.79 (s, 3H), 1.37 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 160.63, 156.33, 129.37, 128.72, 114.17, 113.69, 63.19, 55.26, 43.39, 13.92.

LRMS (ESI) m/z 236.1 ($M - H^+$)
HRMS (ESI) m/z 236.0928 ([$M - H^+$]), calc. for [$C_{12}H_{15}NO_4 - H^+$] 236.0928.

Amide **35**: Ethyl 2-oxo-2-(phenylamino)acetate

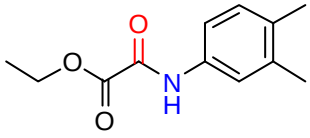


Following the above general procedure with ethyl glyoxylate (ca. 50% soln. in toluene) (19.8 µL, 0.10 mmol, 1 equiv) and aniline (13.7 µL, 0.15 mmol, 1.5 equiv), the crude reaction mixture was purified by flash chromatography to provide amide **35** (16.0 mg, 83%) as a brown oil.

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.89 (s, 1H), 7.64 (d, *J* = 7.6 Hz, 2H), 7.40 – 7.35 (m, 2H), 7.21 – 7.16 (m, 1H), 4.42 (q, *J* = 7.2 Hz, 2H), 1.43 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 160.99, 153.85, 136.30, 129.20, 125.49, 119.79, 63.71, 13.96.

LRMS (ESI) m/z 192.1 ($M - H^+$)
HRMS (ESI) m/z 192.0664 ([$M - H^+$]), calc. for [$C_{10}H_{11}NO_3 - H^+$] 192.0666.

Amide **36**: Ethyl 2-((3,4-dimethylphenyl)amino)-2-oxoacetate

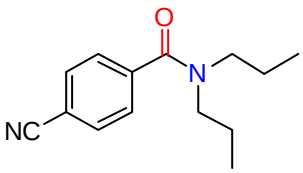


Following the above general procedure with ethyl glyoxylate (ca. 50% soln. in toluene) (19.8 µL, 0.10 mmol, 1 equiv) and 3,4-dimethylaniline (18.2 mg, 0.15 mmol, 1.5 equiv), the crude reaction mixture was purified by flash chromatography to provide amide **36** (20.3 mg, 92%) as a dark yellow oil.

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.80 (s, 1H), 7.39 – 7.37 (m, 2H), 7.13 – 7.10 (m, 1H), 4.40 (q, *J* = 7.1 Hz, 2H), 2.26 (s, 3H), 2.23 (s, 3H), 1.42 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 161.11, 153.68, 137.49, 134.02, 133.98, 130.14, 120.99, 117.27, 63.57, 19.84, 19.23, 13.95.

LRMS (ESI) m/z 222.1 ($M + H^+$)
HRMS (ESI) m/z 222.1133 ([$M + H^+$]), calc. for [$C_{12}H_{15}NO_3 + H^+$] 222.1125.

Amide **37**: 4-cyano-N,N-dipropylbenzamide

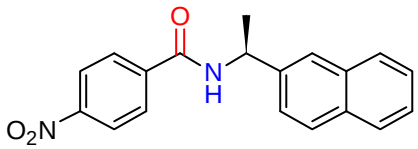


Following the above general procedure with 4-cyanobenzaldehyde (13.1 mg, 0.10 mmol, 1 equiv) and N,N-dipropylamine (20.5 μL, 0.15 mmol, 1.5 equiv), the crude reaction mixture was purified by flash chromatography to provide amide **37** (12.7 mg, 55%) as a yellow oil.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.69 (d, *J* = 7.6 Hz, 2H), 7.45 (d, *J* = 7.7 Hz, 2H), 3.45 (t, *J* = 7.3 Hz, 2H), 3.09 (t, *J* = 7.3 Hz, 2H), 1.93 (s, 1H), 1.68 (dd, *J* = 14.5, 7.3 Hz, 2H), 1.51 (dd, *J* = 14.2, 7.1 Hz, 2H), 0.97 (t, *J* = 7.1 Hz, 3H), 0.74 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 169.67, 141.65, 132.34, 127.19, 118.17, 112.91, 50.59, 46.38, 21.87, 20.61, 11.35, 10.97.

LRMS (ESI) m/z 231.1 ($M + H^+$)
HRMS (ESI) m/z 231.1499 ([$M + H^+$]), calc. for [$C_{14}H_{18}N_2O + H^+$] 231.1492.

Amide **38**: (S)-N-(1-(naphthalen-2-yl)ethyl)-4-nitrobenzamide



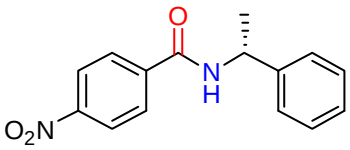
Following the above general procedure with 4-nitrobenzaldehyde (15.1 mg, 0.10 mmol, 1 equiv) and (S)-(-)-1-(2-naphthyl)ethylamine (25.7 mg, 0.15 mmol, 1.5 equiv), the crude reaction mixture was purified by flash chromatography to provide amide **38** (19.8 mg, 62%) as a pale yellow solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.19 (d, *J* = 8.8 Hz, 2H), 7.91 – 7.80 (m, 6H), 7.47 (dd, *J* = 6.1, 3.2 Hz, 3H), 6.69 (s, 1H), 5.50 – 5.41 (m, 1H), 1.69 (d, *J* = 6.9 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 164.61, 149.52, 140.00, 139.75, 133.30, 132.83, 128.74, 128.13, 127.85, 127.64, 126.43, 126.15, 124.78, 124.56, 123.71, 49.80, 21.37.

LRMS (ESI) m/z 343.1 ($M + Na^+$)
HRMS (ESI) m/z 343.1060 ([$M + Na^+$]), calc. for [$C_{19}H_{16}N_2O_3 + Na^+$] 343.1053.

HPLC analysis: Chiralcel OD-H (Hex/IPA = 70/30, 1.0 mL/min, 254 nm, 23°C), 14.63 min, 65.99 min (major), 98% *ee*.

Amide **39**: (R)-4-nitro-N-(1-phenylethyl)benzamide



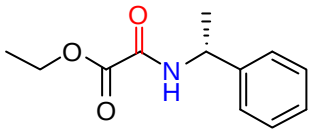
Following the above general procedure with 4-nitrobenzaldehyde (15.1 mg, 0.10 mmol, 1 equiv) and (R)-(+)-alpha-methylbenzylamine (19.3 µL, 0.15 mmol, 1.5 equiv), the crude reaction mixture was purified by flash chromatography to provide amide **39** (19.7 mg, 73%) as an off-white solid.

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.23 (d, *J* = 8.4 Hz, 2H), 7.91 (d, *J* = 8.7 Hz, 2H), 7.38 – 7.27 (m, 5H), 6.61 (s, 1H), 5.36 – 5.26 (m, 1H), 1.62 (d, *J* = 6.9 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 164.57, 149.51, 142.45, 140.10, 128.84, 128.14, 127.73, 126.21, 123.72, 49.73, 21.50.

LRMS (ESI) m/z 292.9 ($M + Na^+$)
HRMS (ESI) m/z 293.0911 ([$M + Na^+$]), calc. for [$C_{15}H_{14}N_2O_3 + Na^+$] 293.0897.

HPLC analysis: Chiralcel OD-H (Hex/IPA = 80/20, 1.0 mL/min, 254 nm, 23°C), 17.66 min, 20.94 min (major), 98% *ee*.

Amide **40**: (R)-ethyl 2-oxo-2-((1-phenylethyl)amino)acetate



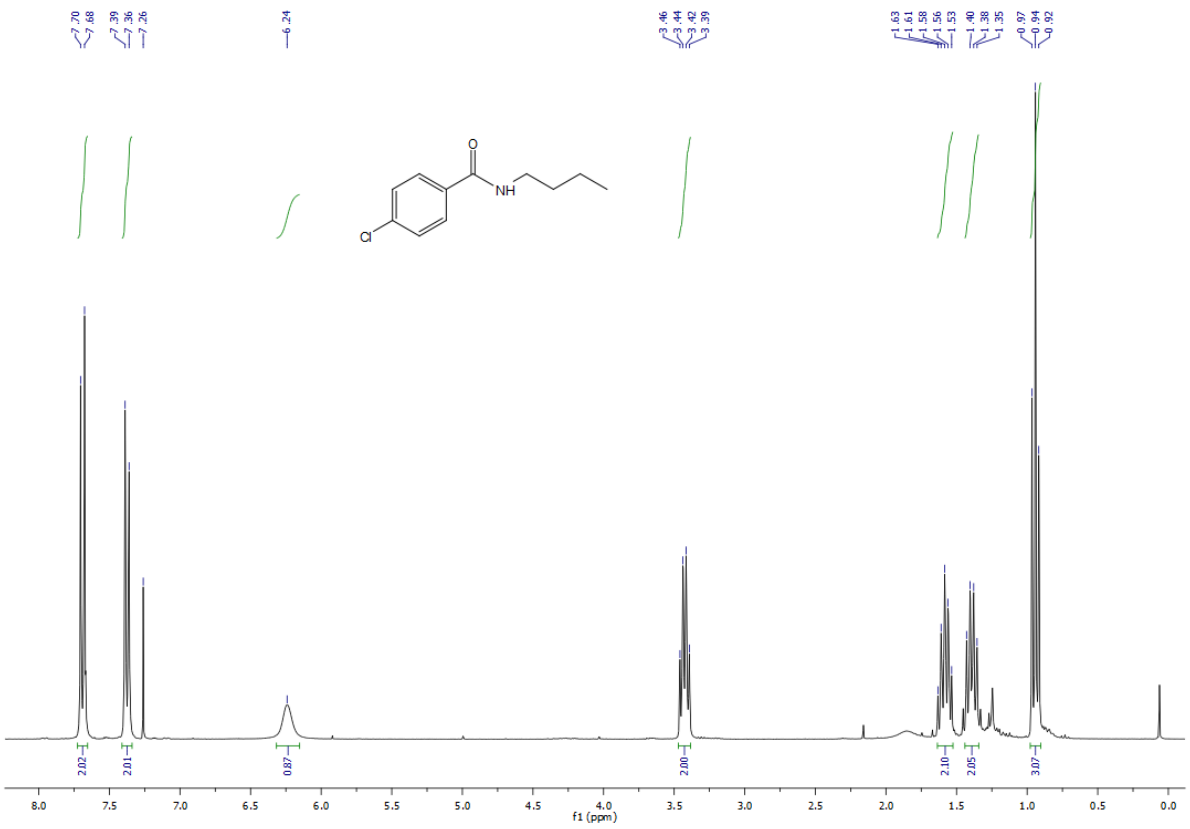
Following the above general procedure with ethyl glyoxylate (ca. 50% soln. in toluene) (19.8 µL, 0.10 mmol, 1 equiv) and (R)-(+)-alpha-methylbenzylamine (19.3 µL, 0.15 mmol, 1.5 equiv), the crude reaction mixture was purified by flash chromatography to provide amide **40** (18.8 mg, 85%) as a colourless oil.

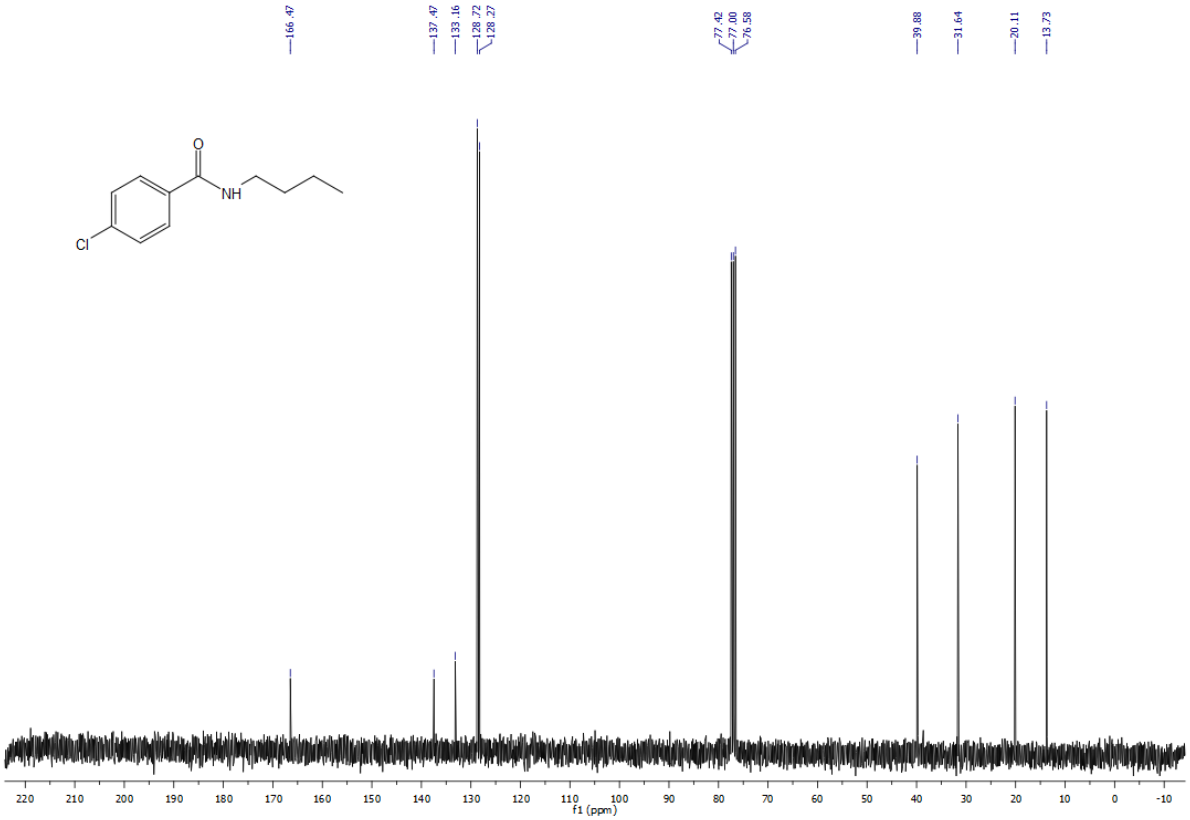
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.38 – 7.27 (m, 5H), 5.19 – 5.09 (m, 1H), 4.33 (q, *J* = 7.1 Hz, 2H), 1.56 (d, *J* = 6.9 Hz, 3H), 1.37 (t, *J* = 7.1 Hz, 3H). **^{13}C NMR (75 MHz, $CDCl_3$)** δ 160.78, 155.57, 141.67, 128.78, 127.78, 126.23, 63.22, 49.40, 21.22, 13.92.

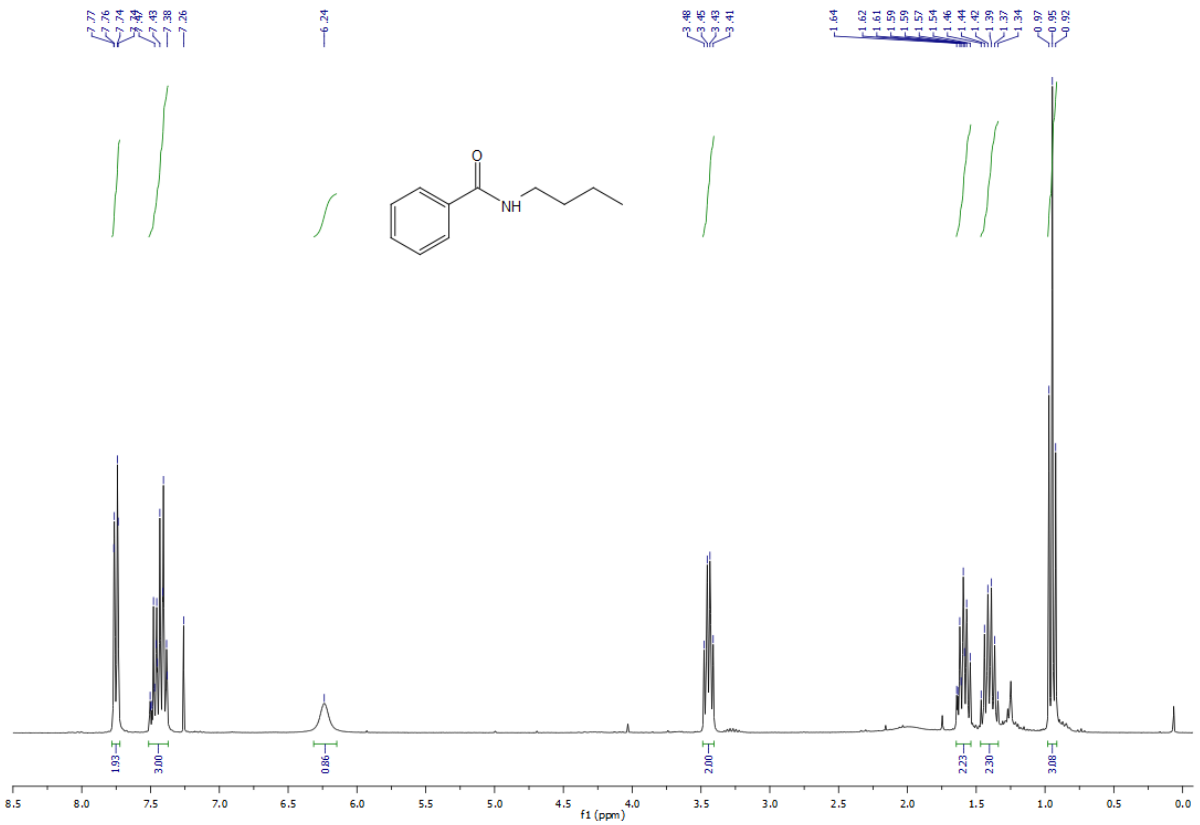
LRMS (ESI) m/z 220.0 ($M - H^+$)
HRMS (ESI) m/z 220.0981 ([$M - H^+$]), calc. for [$C_{12}H_{15}NO_3 - H^+$] 220.0979.

HPLC analysis: Chiralcel OD-H (Hex/IPA = 80/20, 1.0 mL/min, 210 nm, 23°C), 6.30 min (major), 7.67 min, 97% *ee*.

5. NMR Spectra of Products



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7.26
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3.43
3.41
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1.62
1.61
1.59
1.57
1.54
1.46
1.44
1.42
1.39
1.37
1.34
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0.92
O
NH
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3.00
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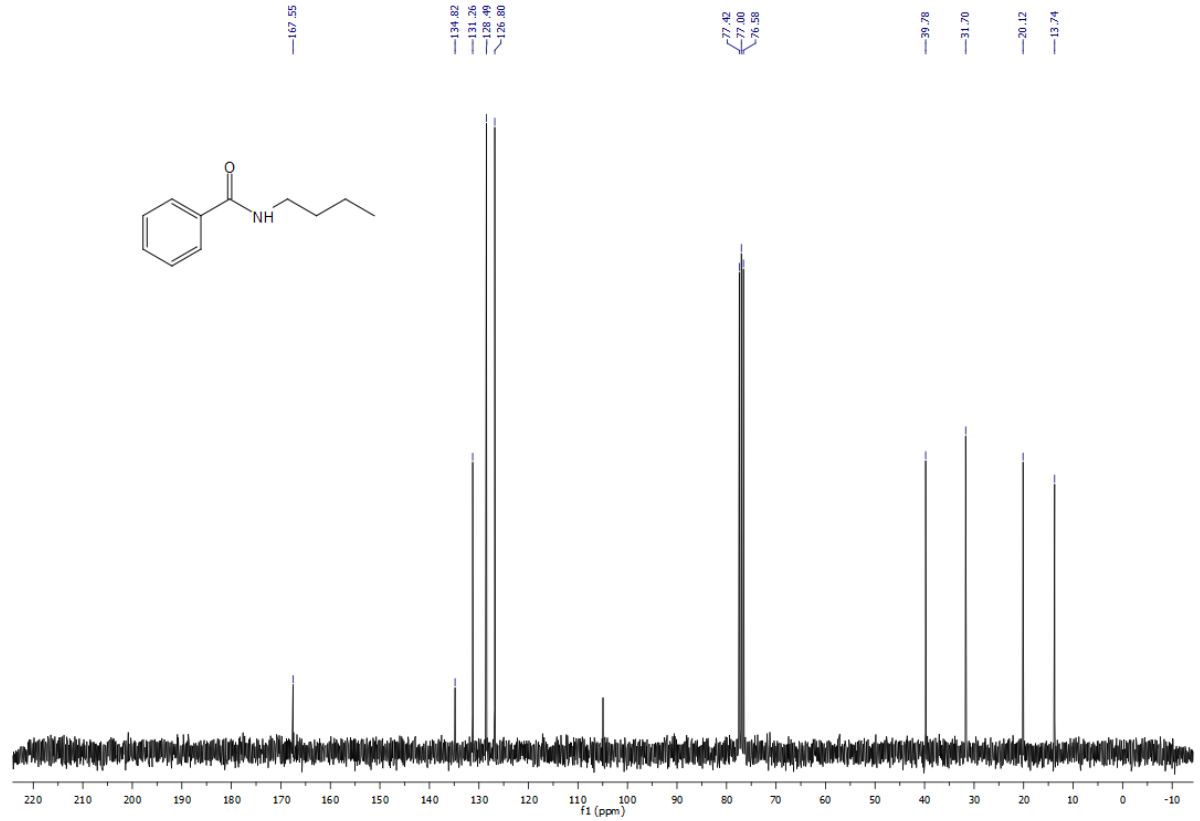
f1 (ppm)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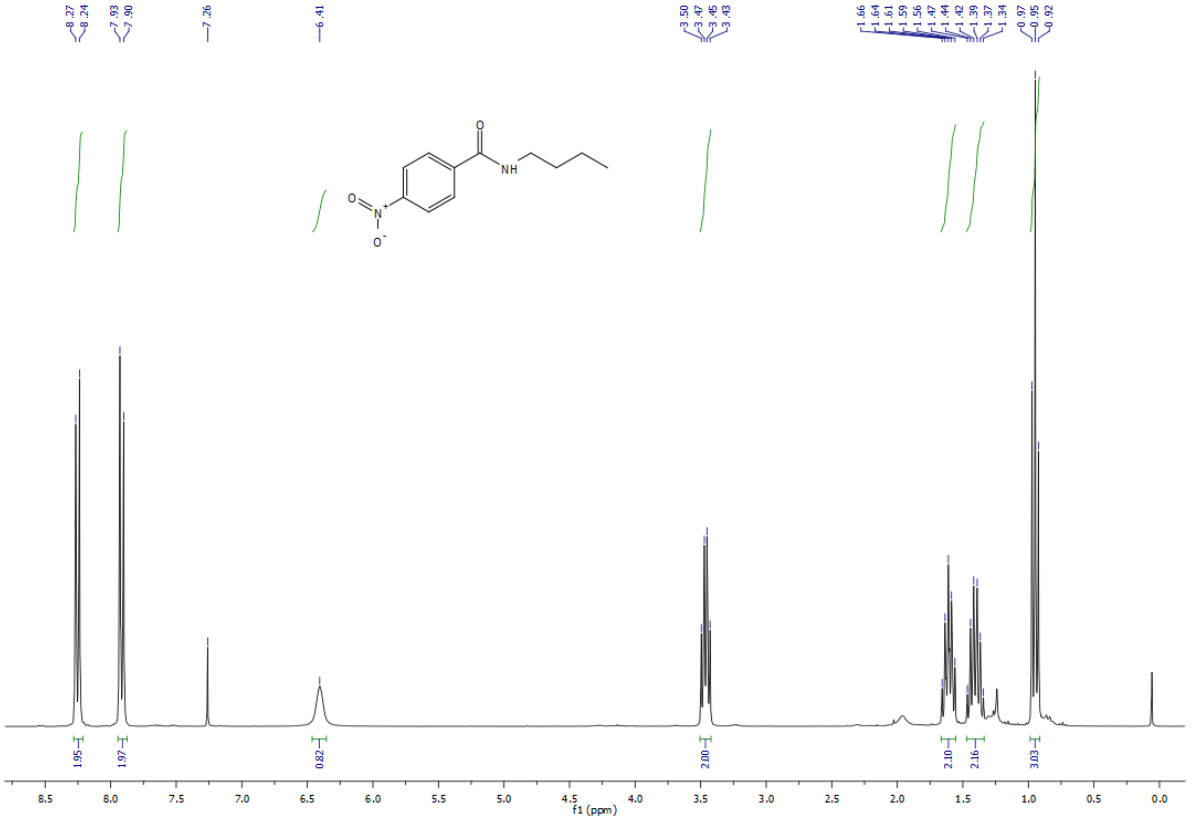
O
NH
f1 (ppm)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1.59
1.56
1.47
1.44
1.42
1.39
1.37
1.34
0.97
0.95
0.92
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O
N+
O-
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1.97
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2.10
2.16
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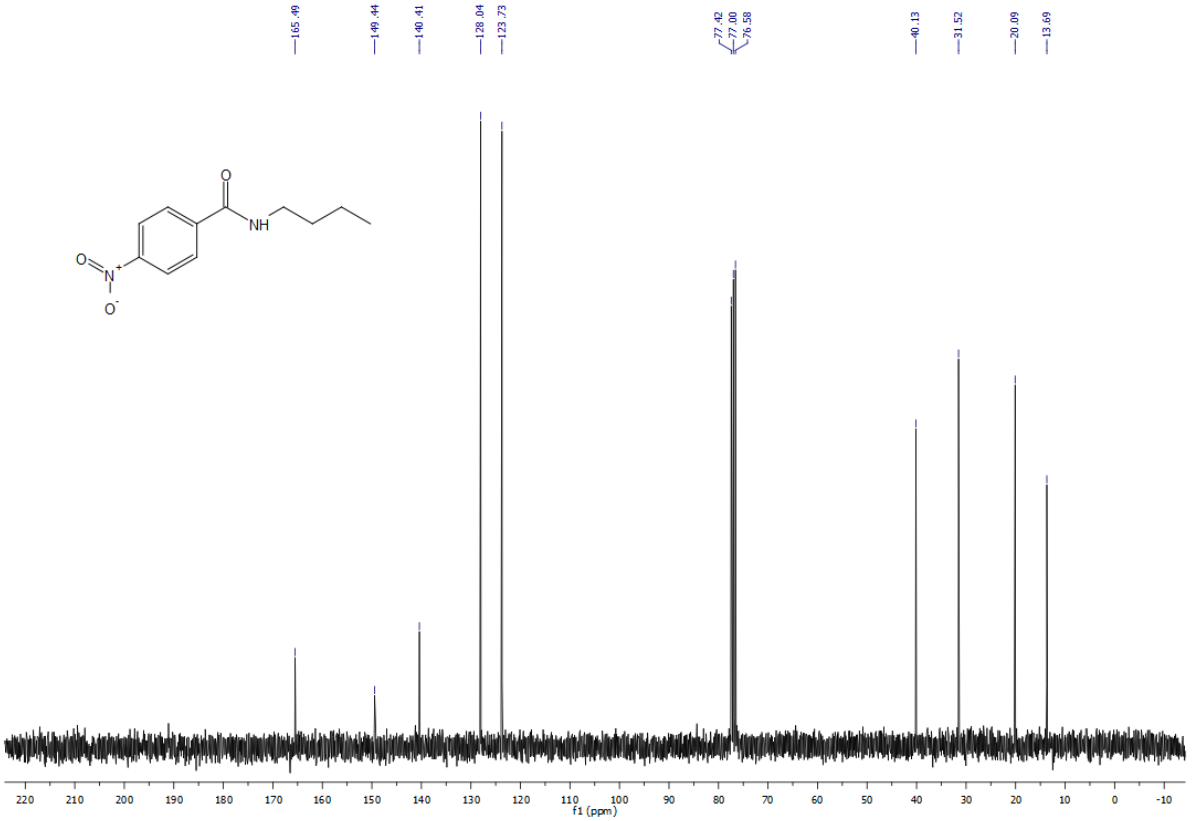
f1 (ppm)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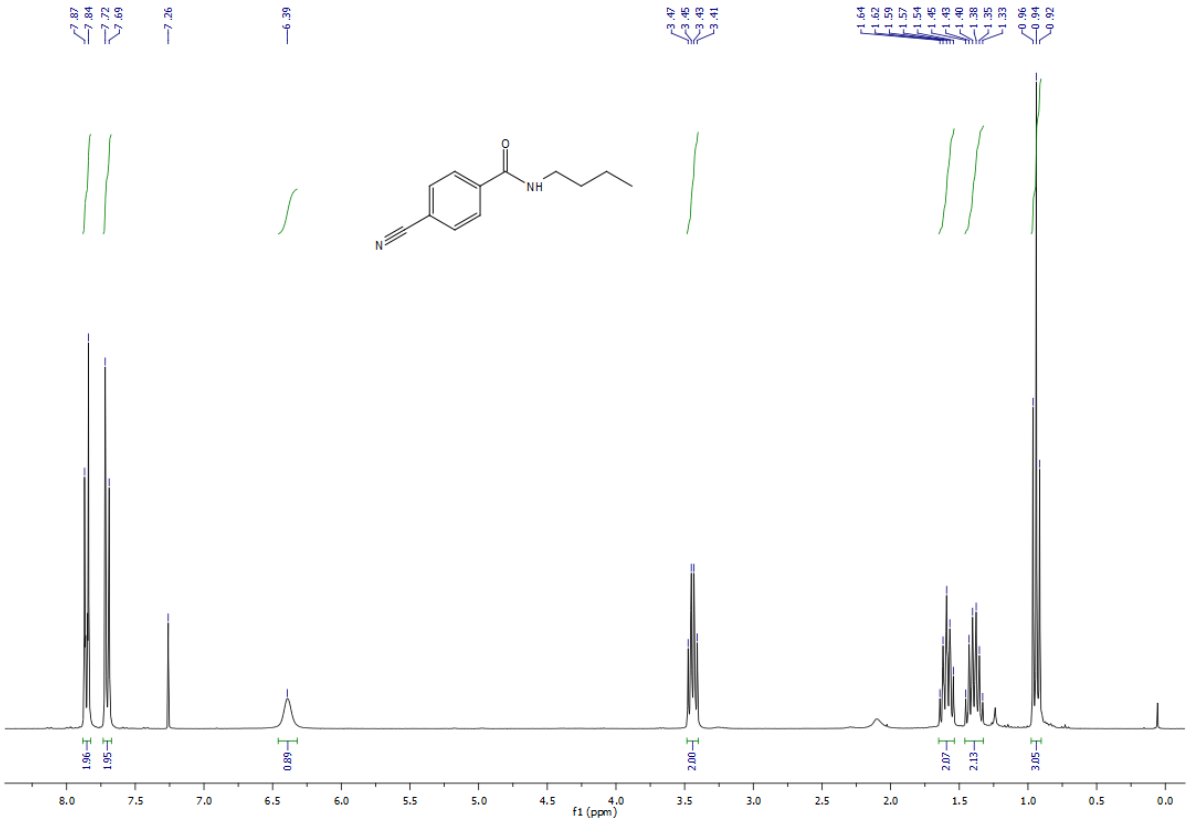
O-
f1 (ppm)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1.57
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1.45
1.43
1.40
1.38
1.35
1.33
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0.94
0.92
O
NH
N
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1.95
0.89
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2.13
3.05
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7.0
6.5
6.0
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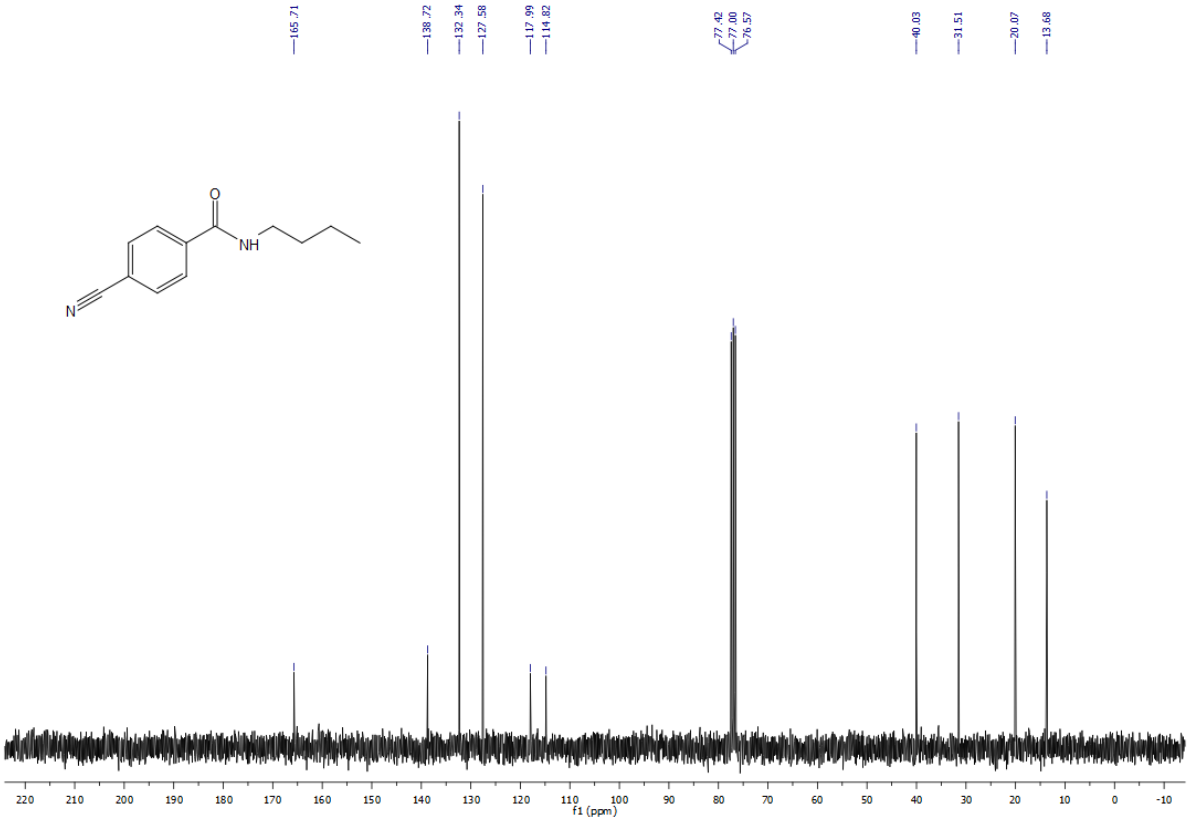
f1 (ppm)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N
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210
200
190
180
170
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40
30
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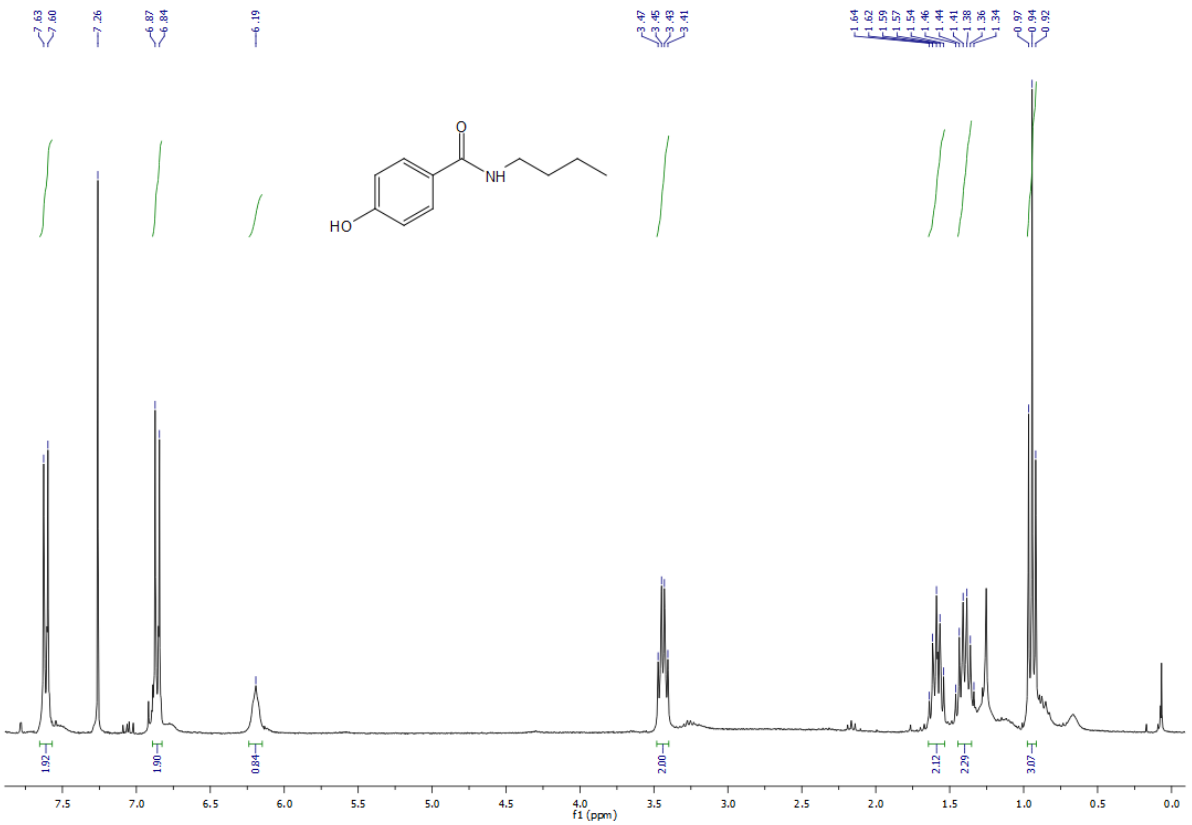
0
-10
f1 (ppm)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6.19
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1.57
1.54
1.46
1.44
1.41
1.38
1.36
1.34
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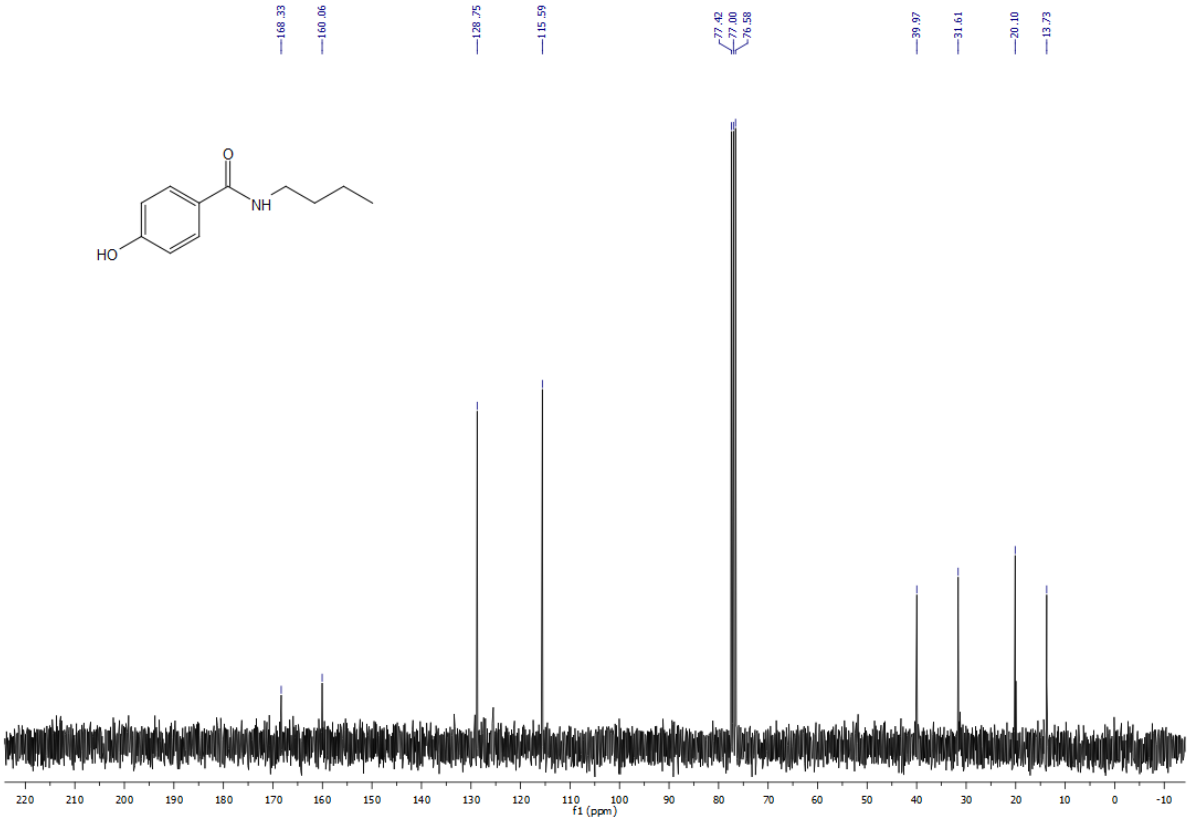
f1 (ppm)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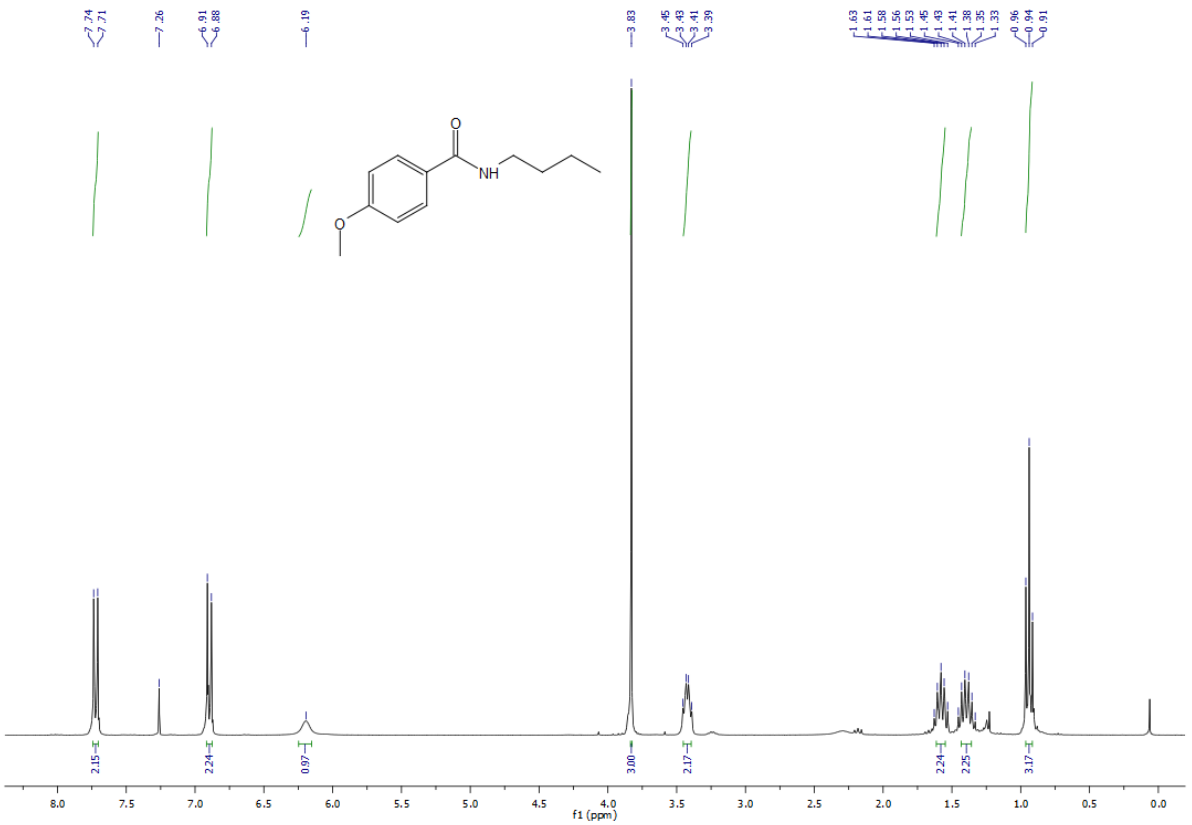
HO
f1 (ppm)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1.58
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1.43
1.41
1.38
1.35
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0.94
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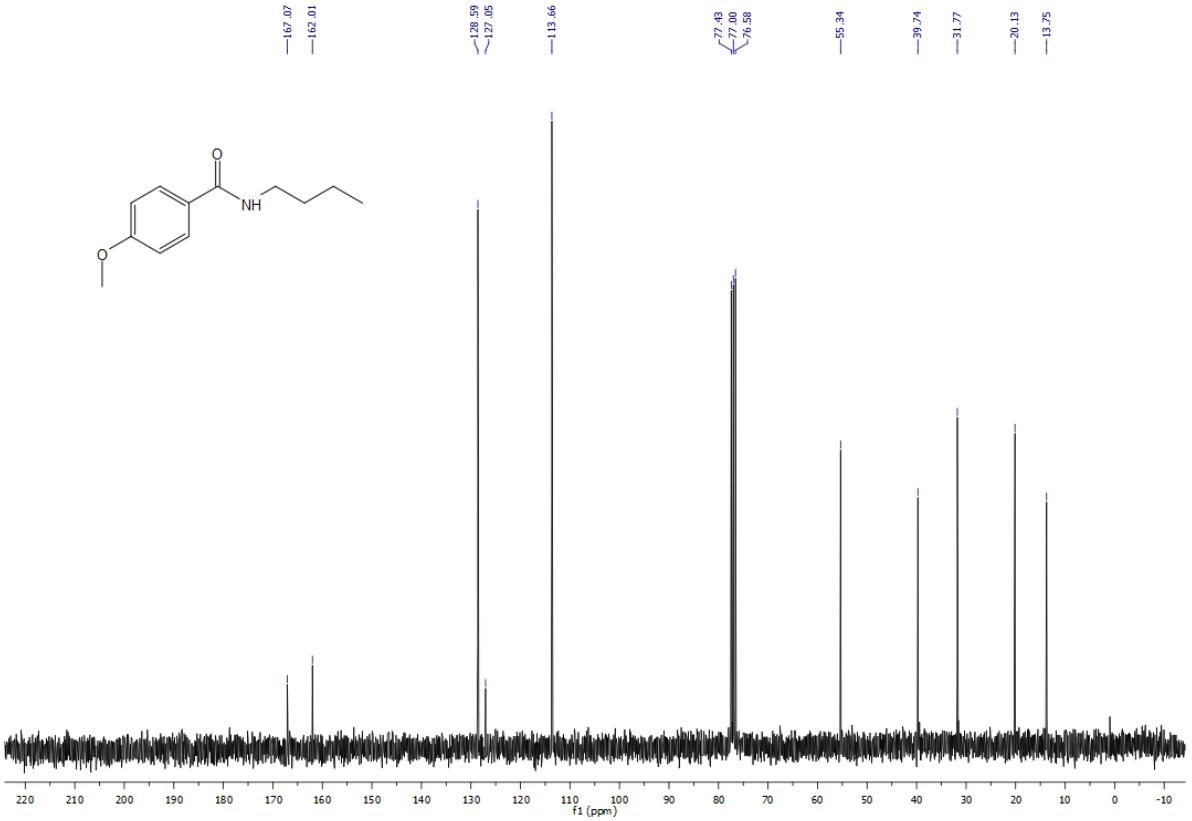
3.17
f1 (ppm)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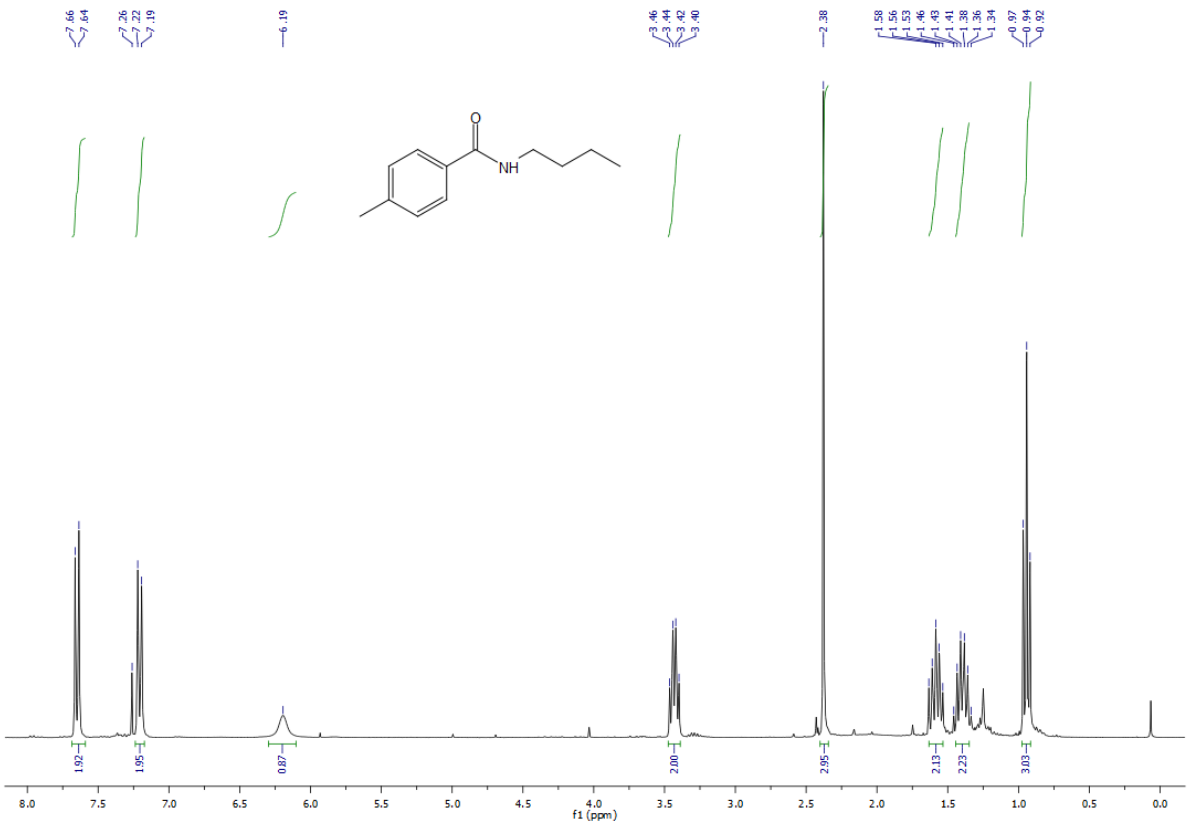
NH
O
f1 (ppm)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1.46
1.43
1.41
1.38
1.36
1.34
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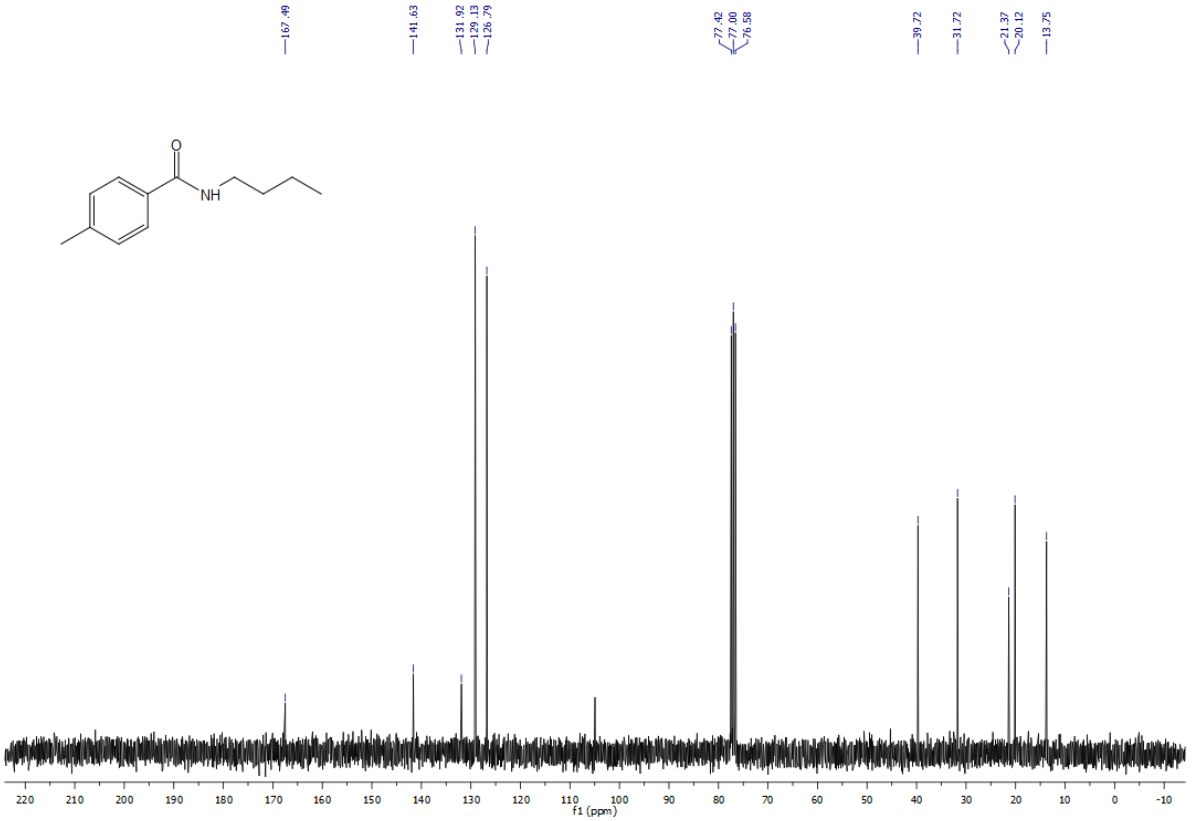
f1 (ppm)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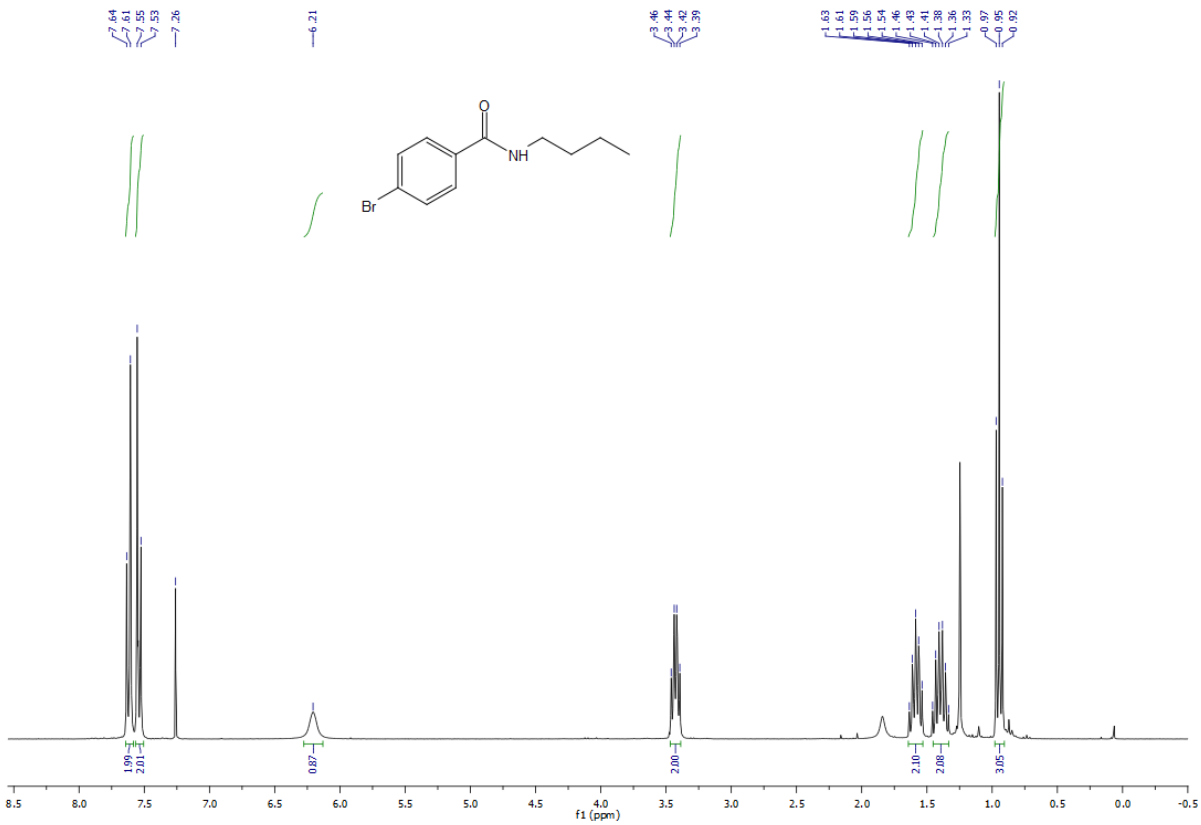
13.75
O
NH
f1 (ppm)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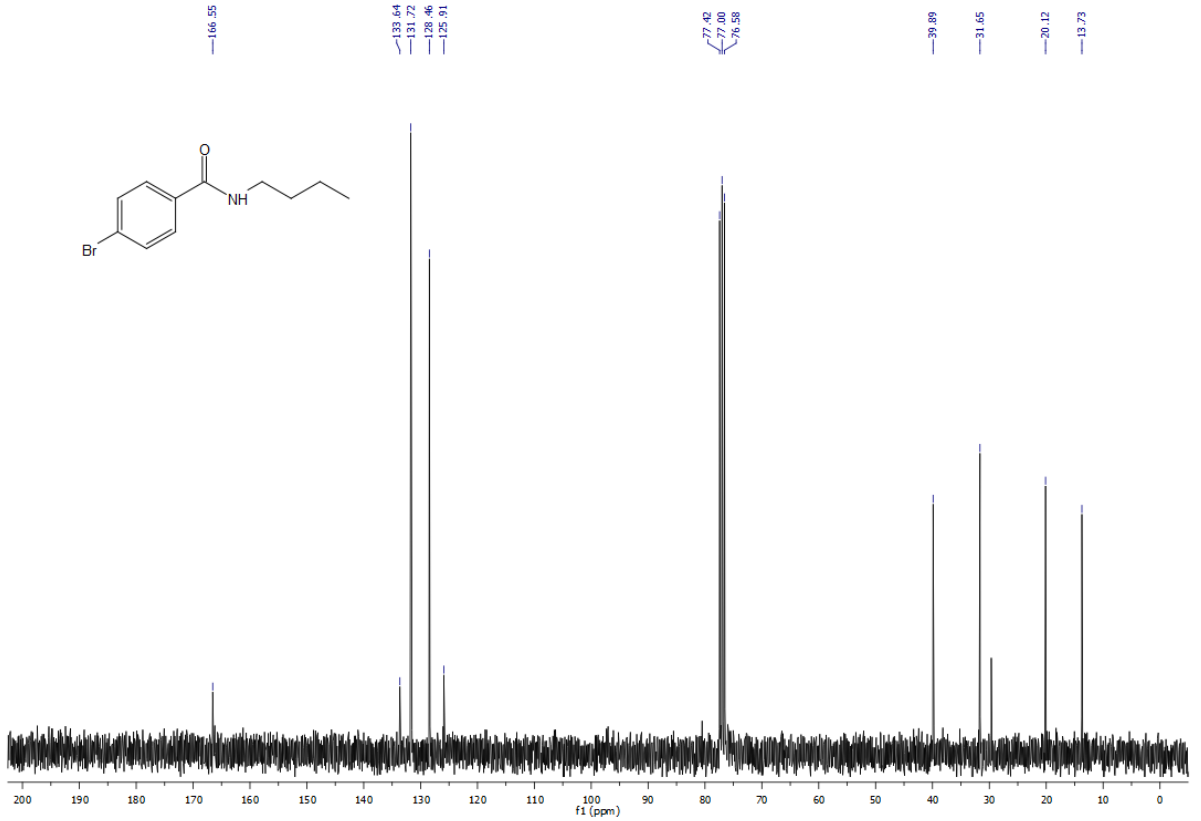
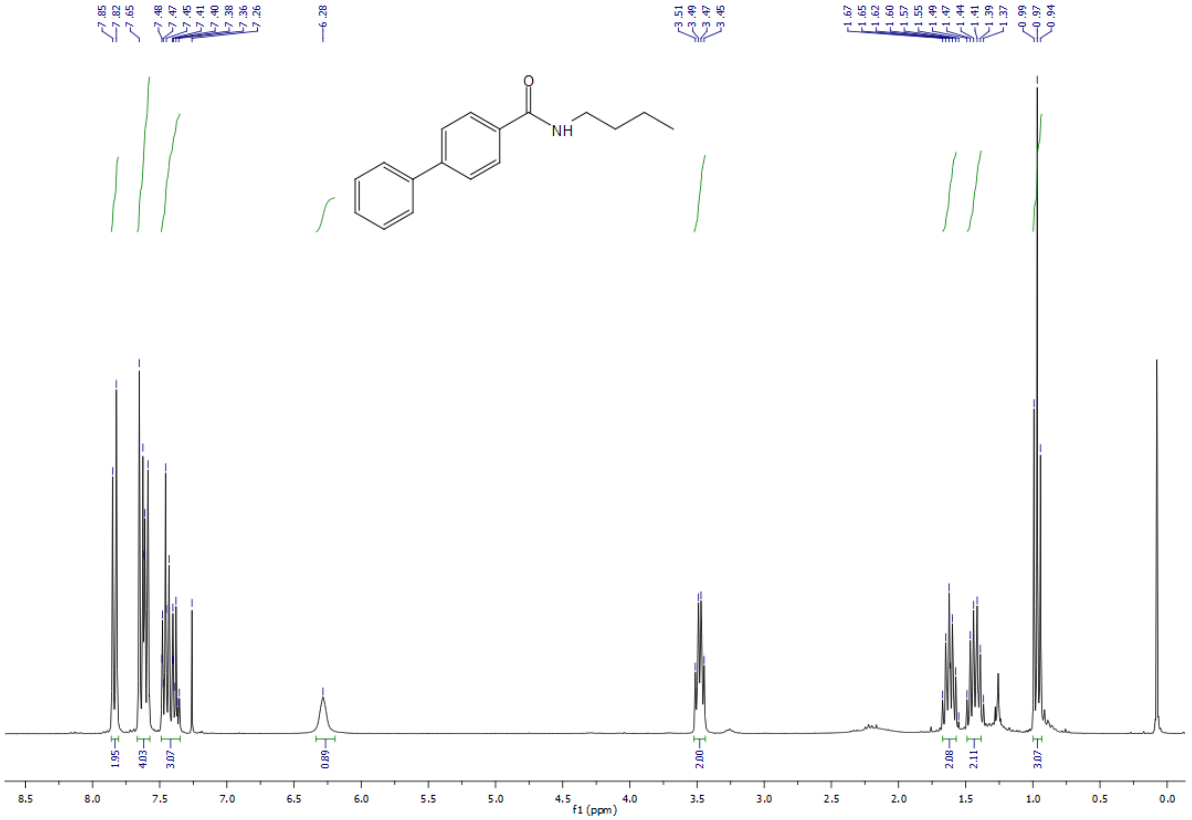
7.61
7.55
7.53
7.26
6.21
3.46
3.44
3.42
3.39
1.63
1.61
1.59
1.56
1.54
1.46
1.43
1.41
1.38
1.36
1.33
0.97
0.95
0.92
O
NH
Br
1.99
2.01
0.87
2.00
2.10
2.08
3.05
f1 (ppm)


166.55
133.64
131.72
128.46
125.91
77.42
77.00
76.58
39.89
31.65
20.12
13.73
O
NH
Br
f1 (ppm)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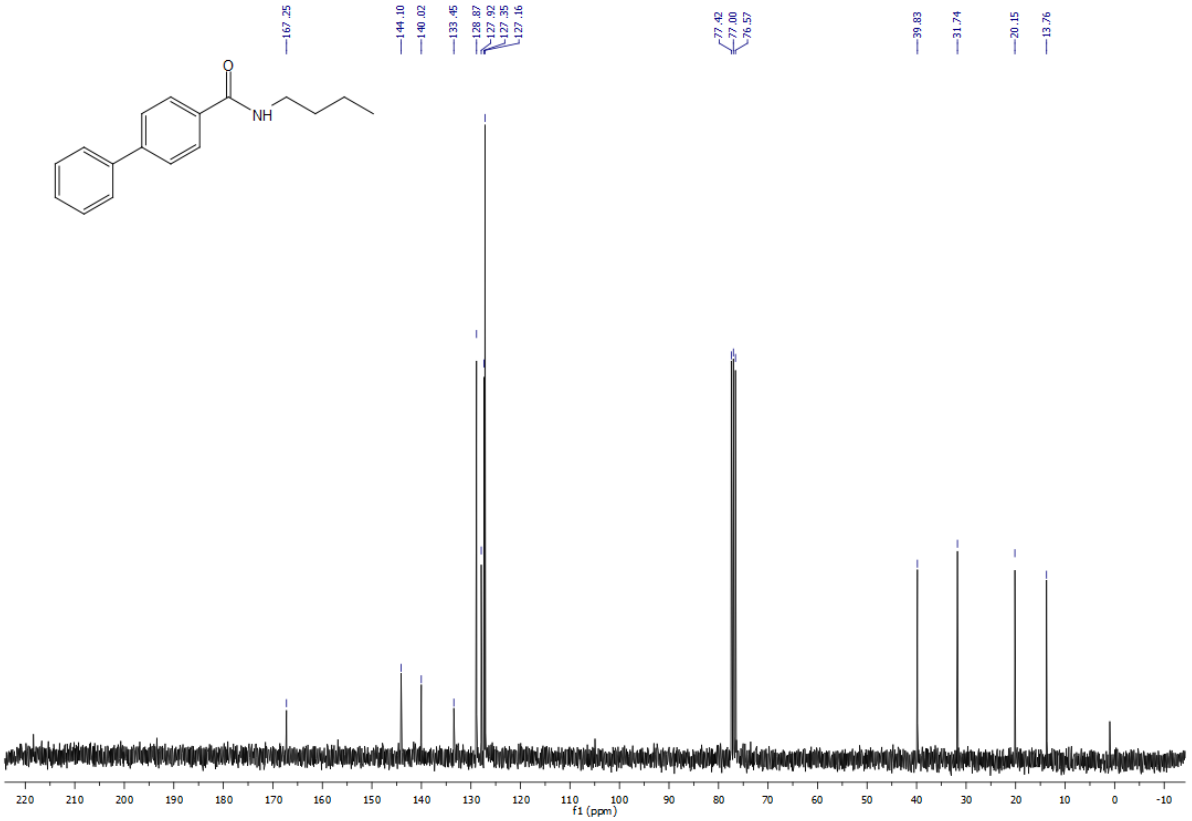
7.82
7.65
7.48
7.47
7.45
7.41
7.40
7.38
7.36
7.26
6.28
3.51
3.49
3.47
3.45
1.67
1.65
1.62
1.60
1.57
1.55
1.49
1.47
1.44
1.41
1.39
1.37
0.99
0.97
0.94
O
NH
1.95
4.03
3.07
0.89
2.00
2.08
2.11
3.07
f1 (ppm)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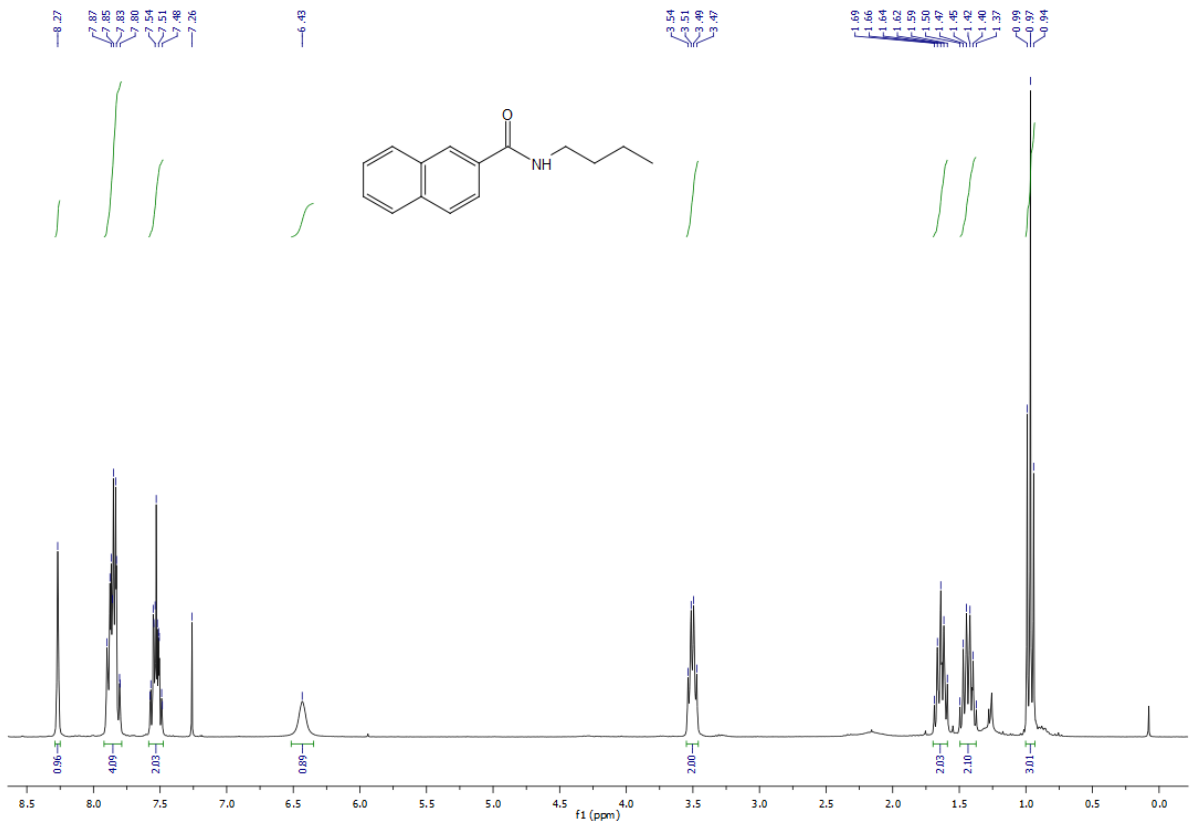
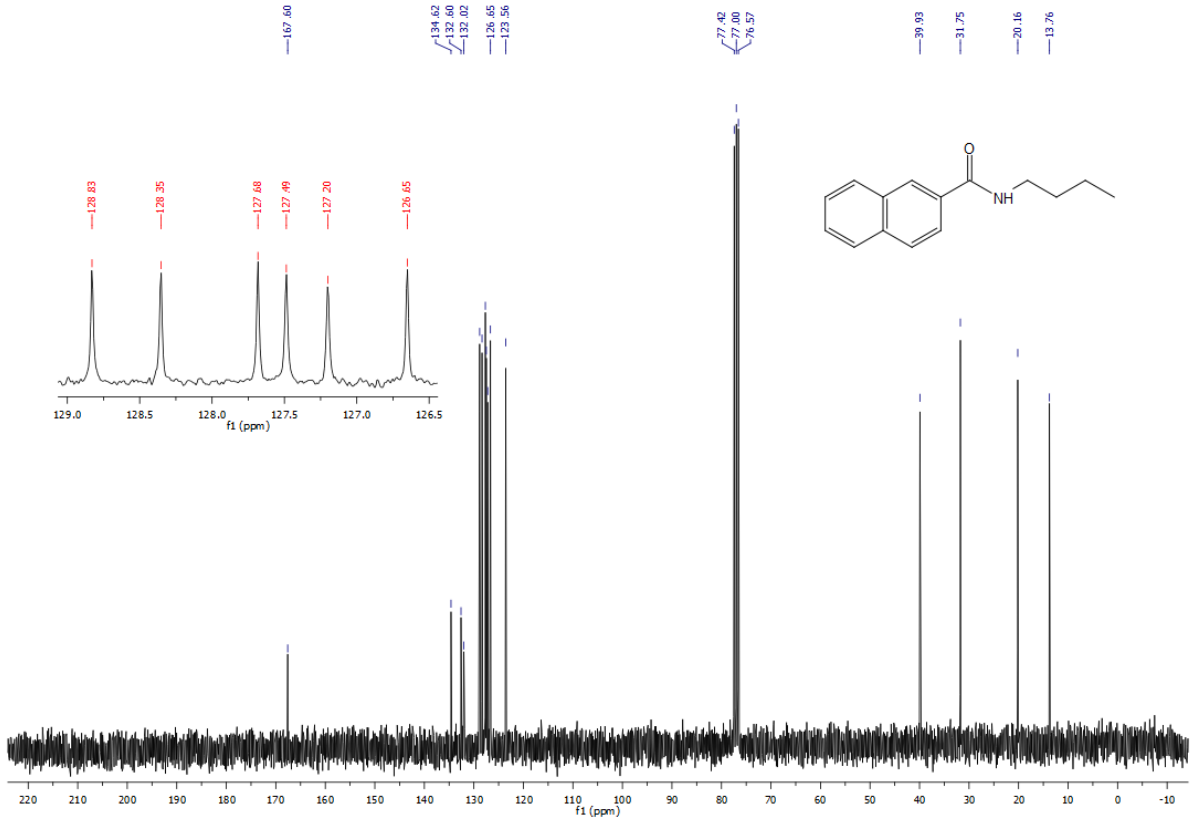
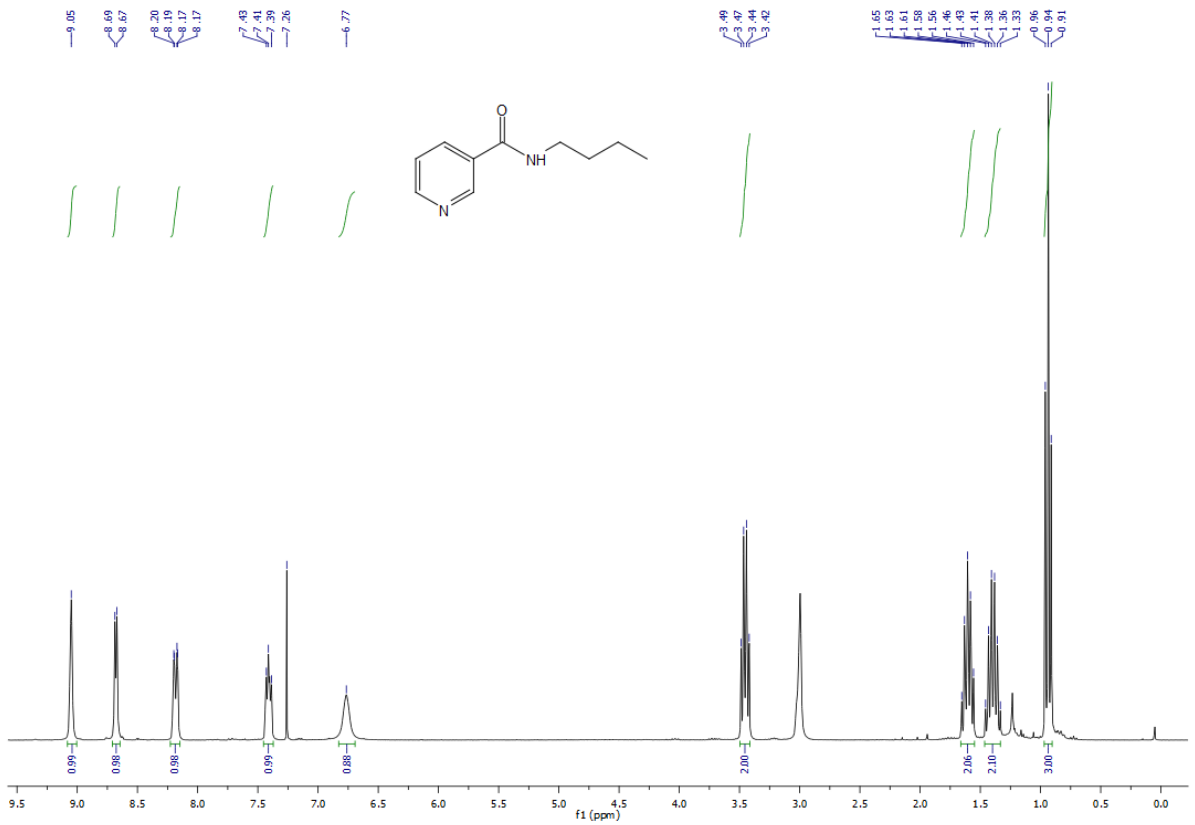
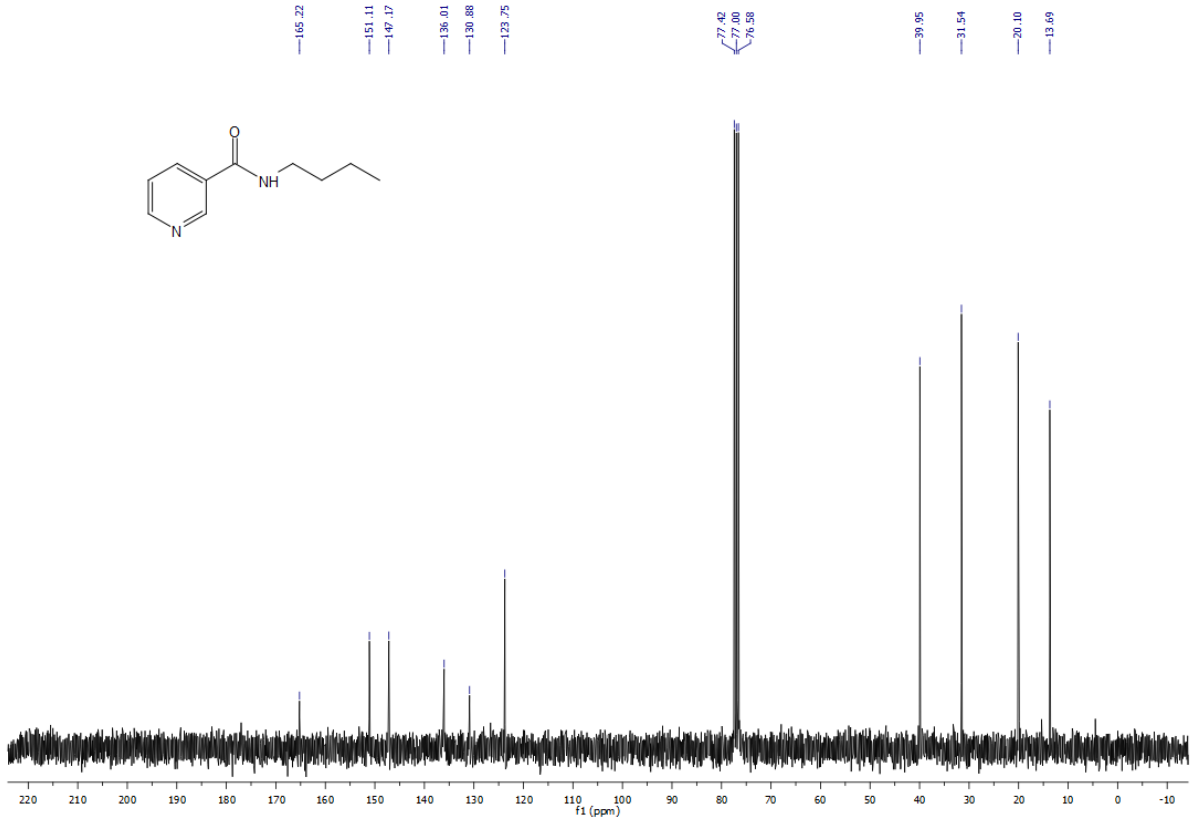
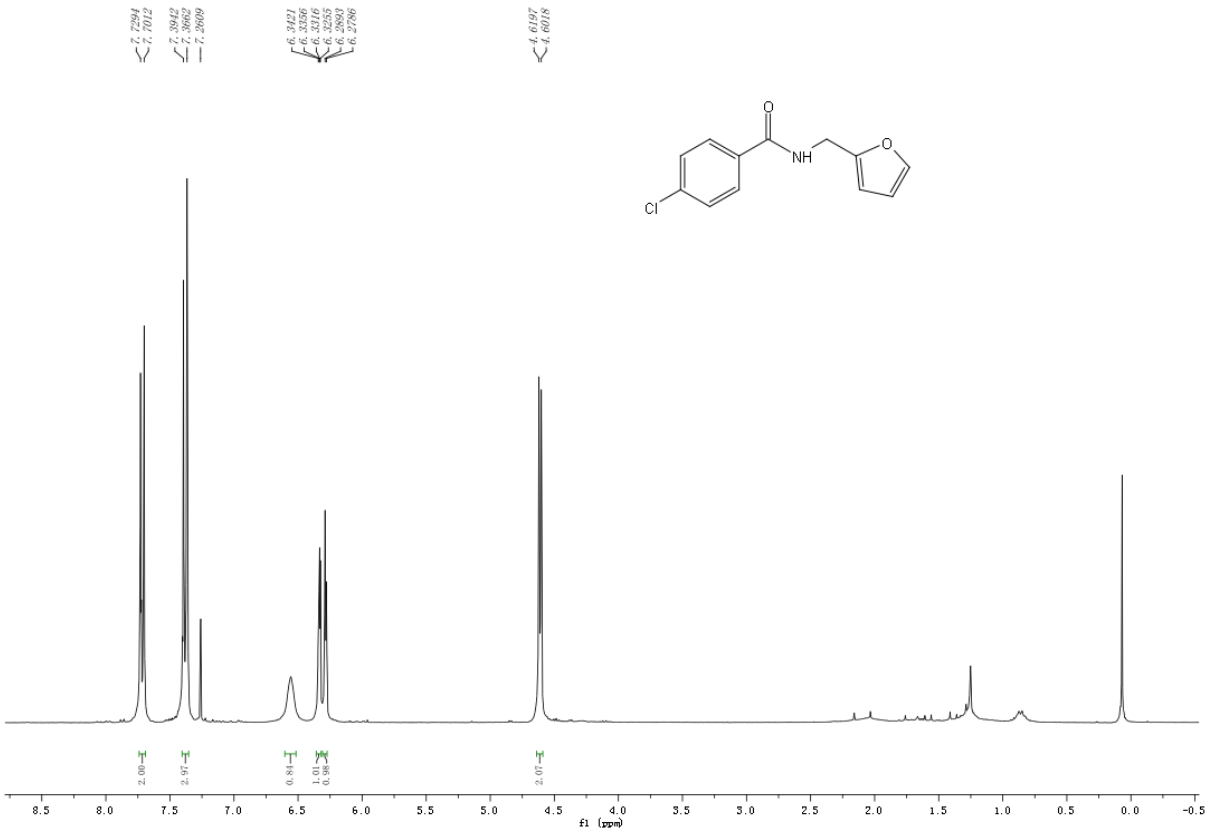
144.10
140.02
133.45
128.87
127.92
127.35
127.16
77.42
77.00
76.57
39.83
31.74
20.15
13.76
O
NH
f1 (ppm)

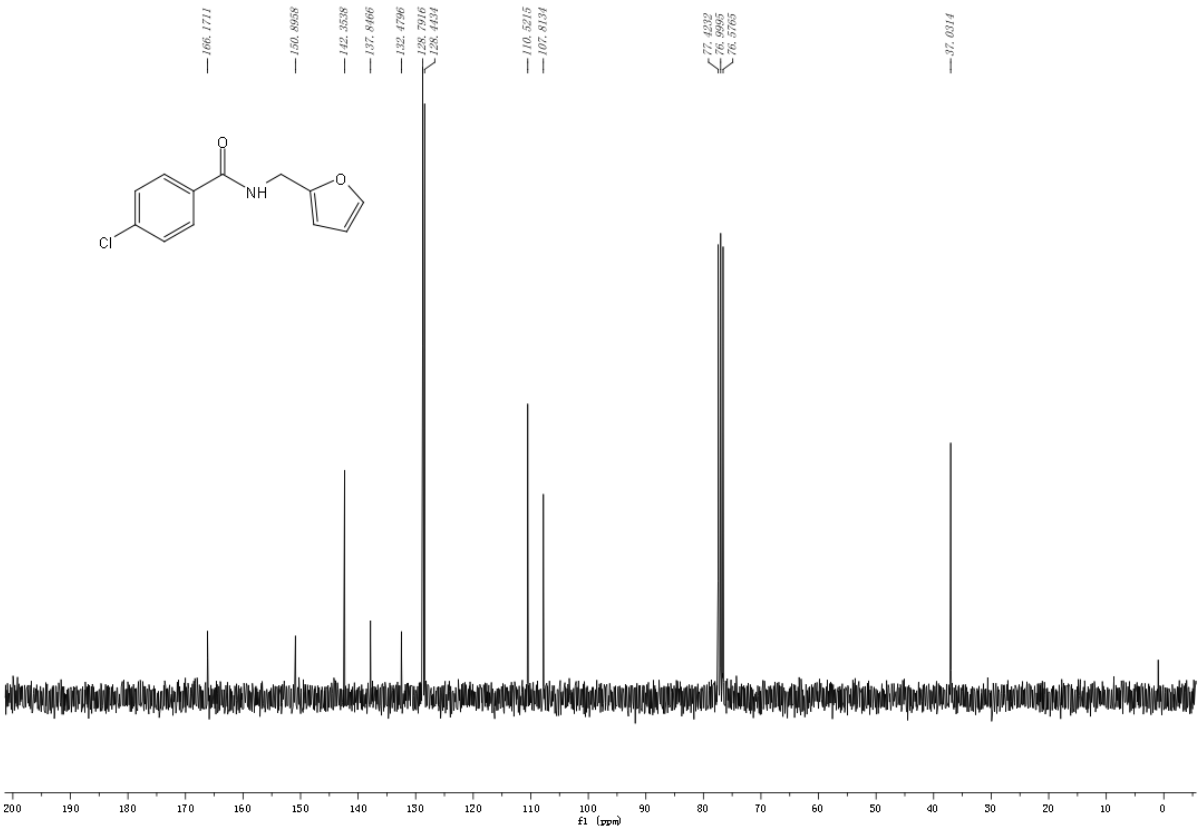

8.27
7.87
7.85
7.83
7.80
7.54
7.51
7.48
7.26
6.43
3.54
3.51
3.49
3.47
1.69
1.66
1.64
1.62
1.59
1.50
1.47
1.45
1.42
1.40
1.37
0.99
0.97
0.94
O
NH
0.96
4.09
2.03
0.89
2.00
2.03
2.10
3.01
f1 (ppm)

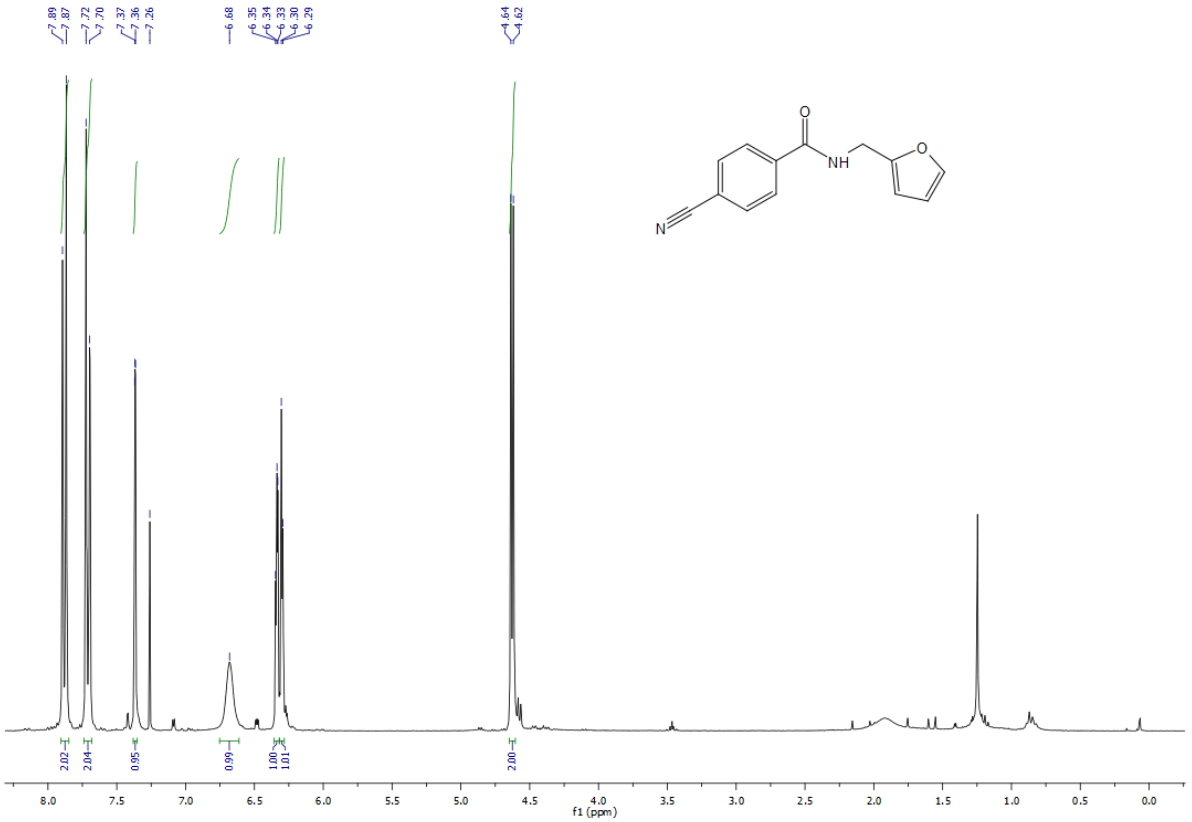

167.60
134.62
132.60
132.02
126.65
123.56
77.42
77.00
76.57
39.93
31.75
20.16
13.76
128.83
128.35
127.68
127.49
127.20
126.65
O
NH
f1 (ppm)
f1 (ppm)

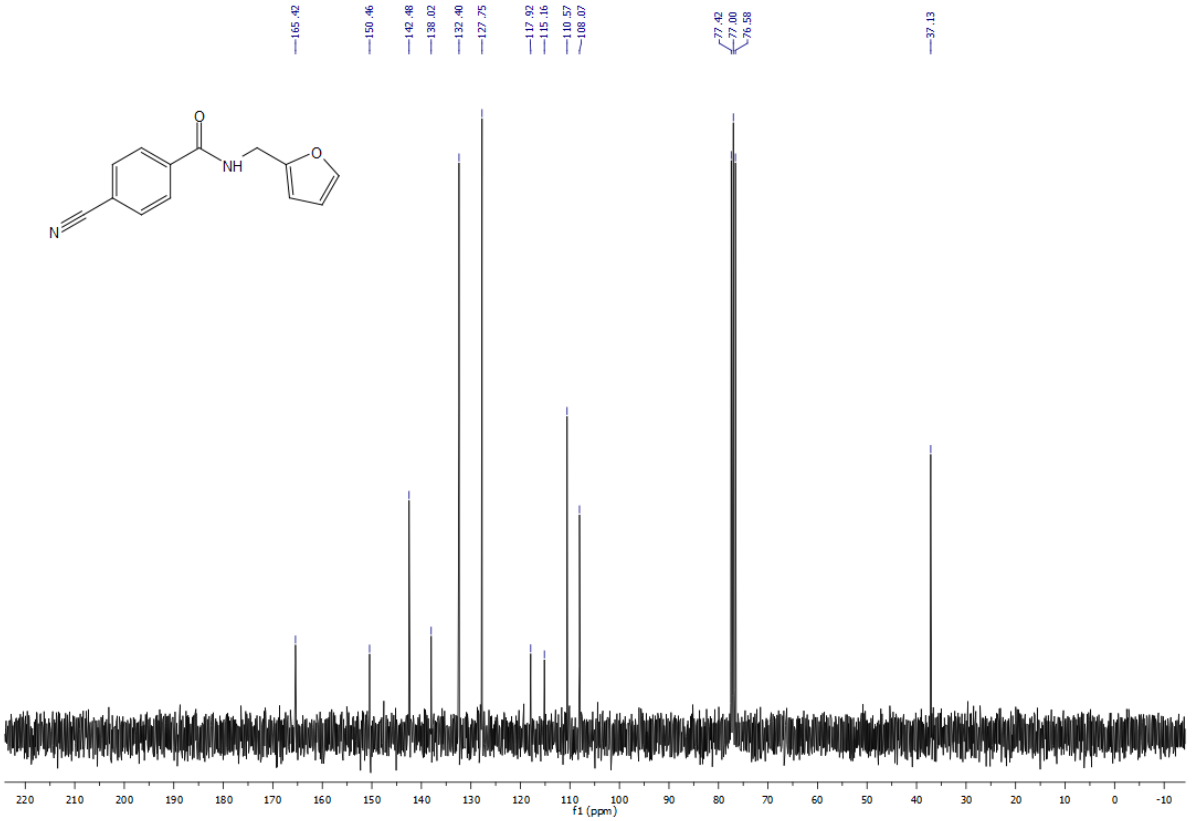


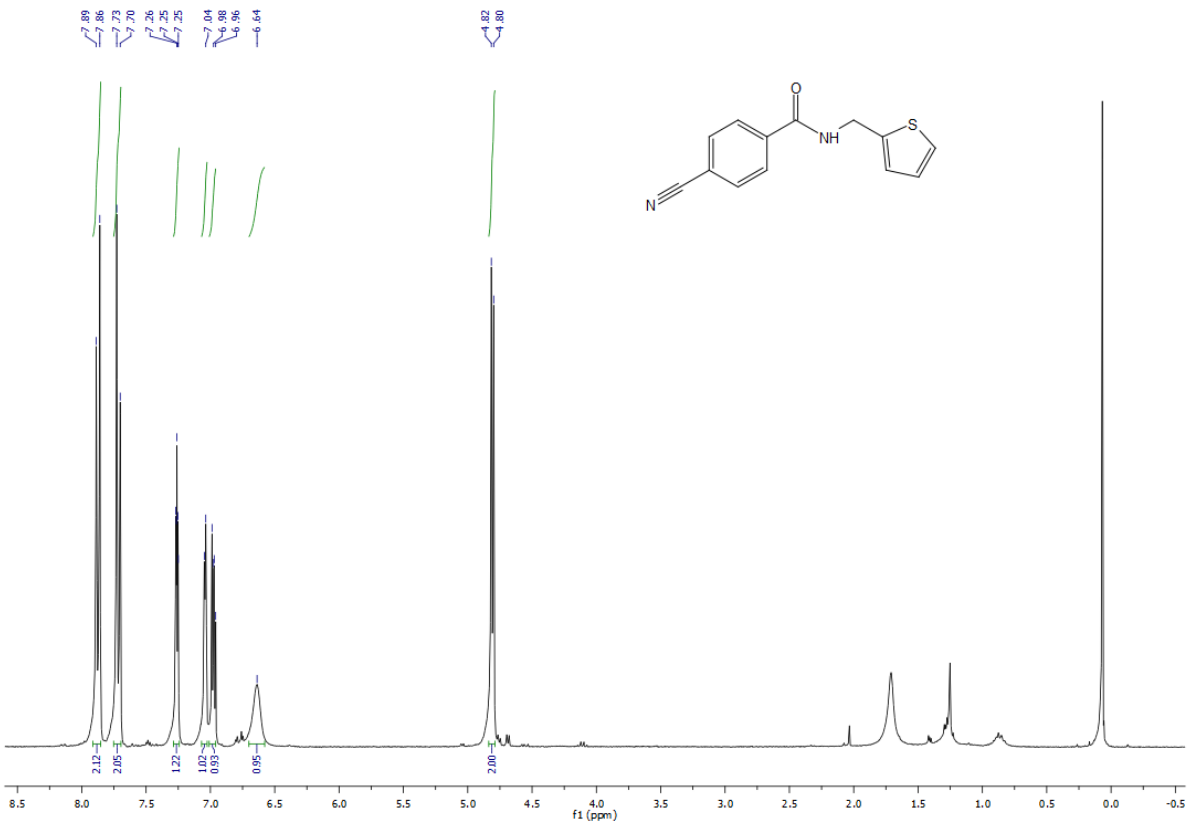


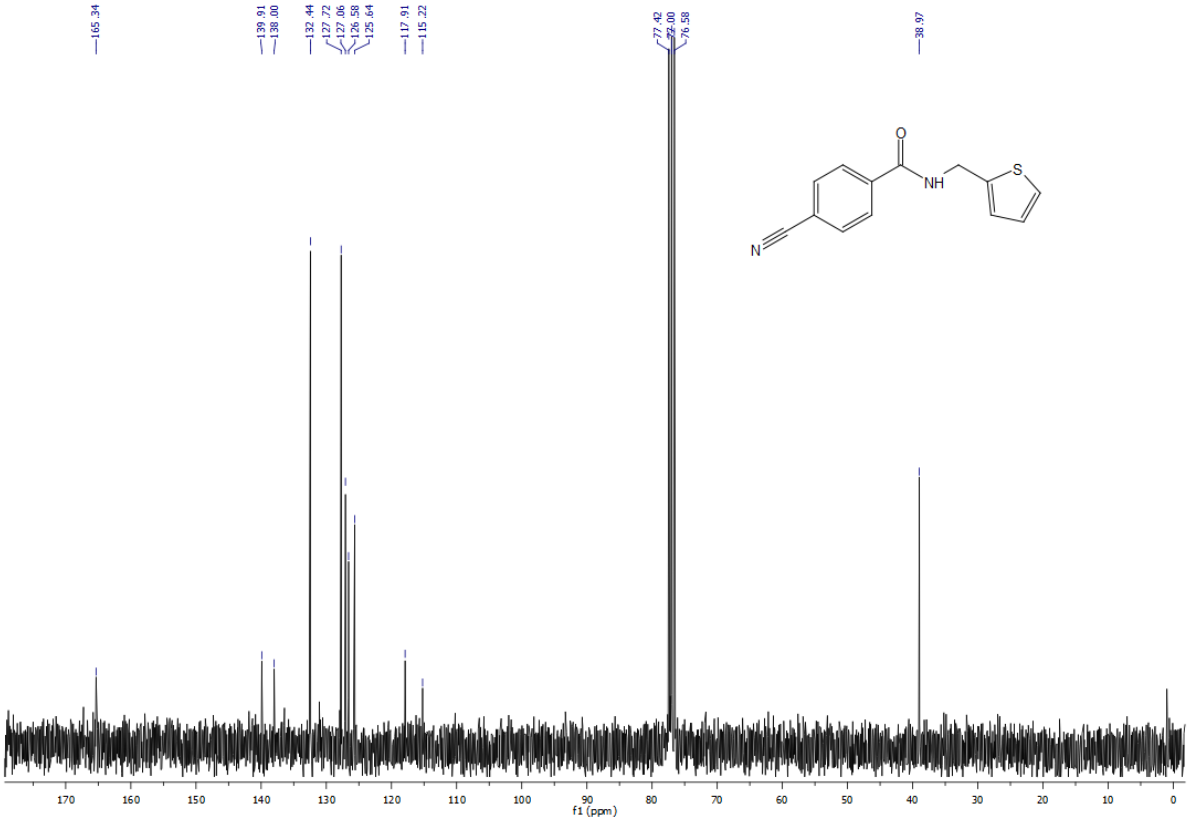

7.7294
7.7012
7.3942
7.3662
7.2609
6.3421
6.3356
6.3316
6.3255
6.2993
6.2786
4.6197
4.6018
O
NH
O
Cl
2.00
2.97
0.84
1.01
0.98
2.07
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

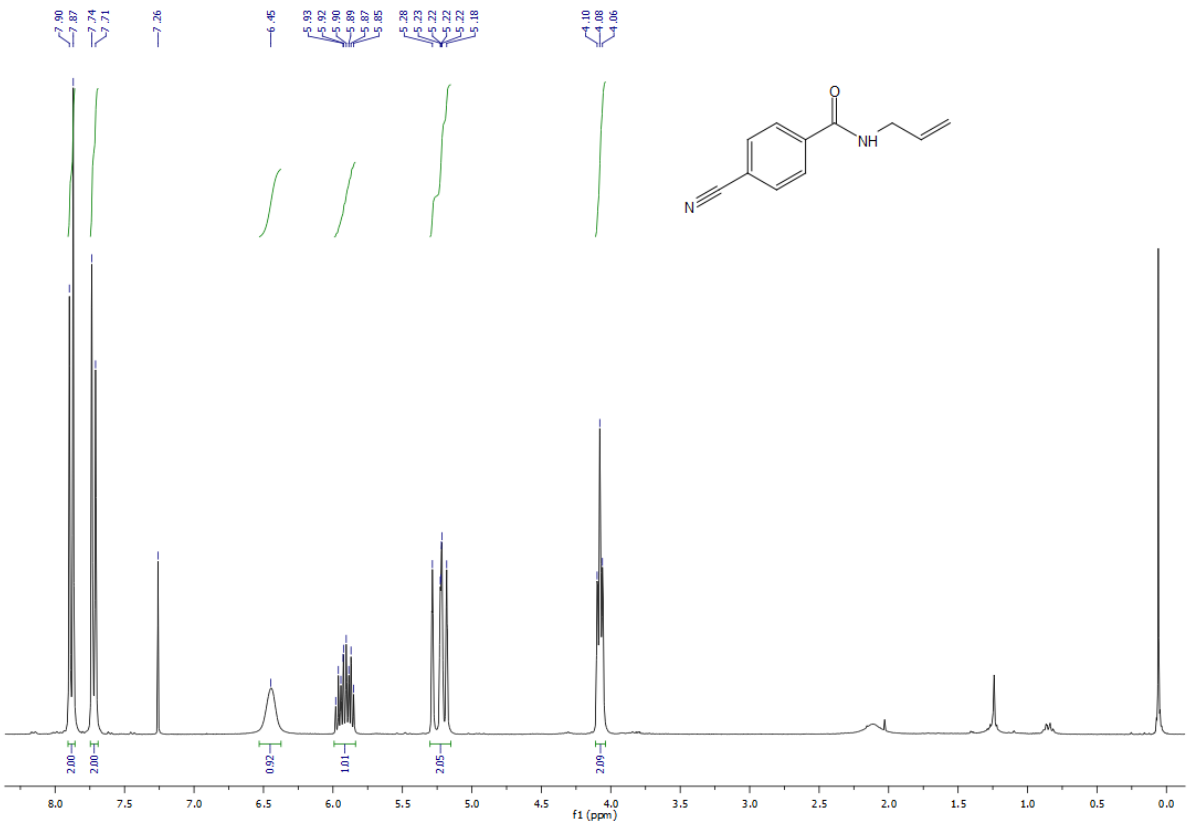

166.1711
150.8958
142.3538
137.8466
132.4796
128.7916
128.4434
110.5215
107.8134
77.4232
76.9995
76.5765
37.0314
O
NH
O
Cl
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

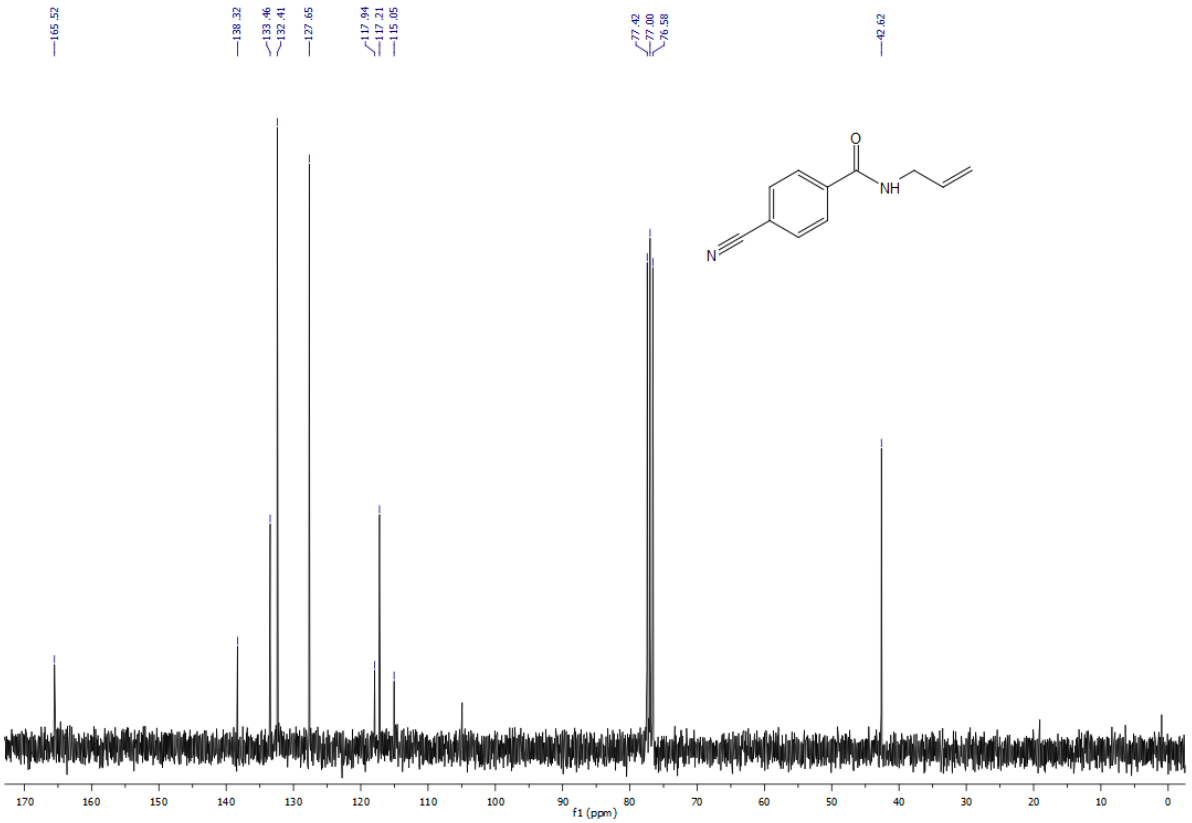




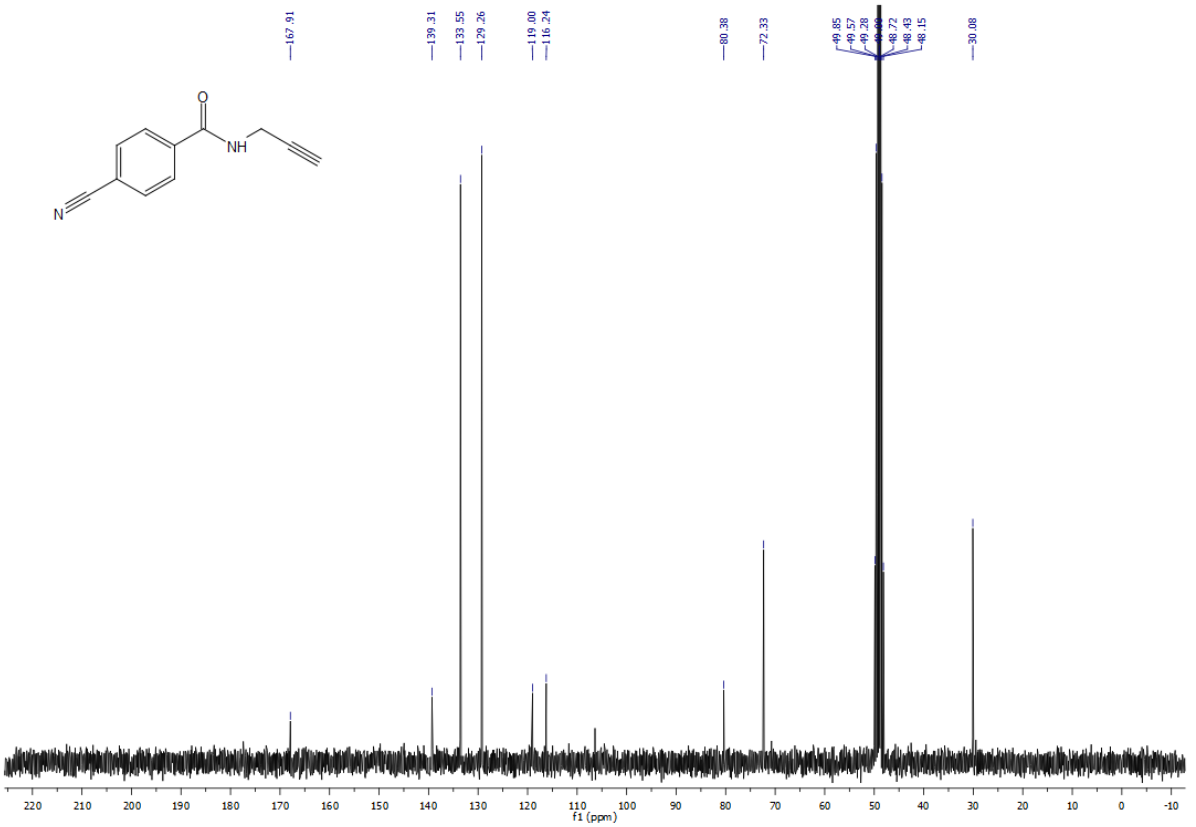

7.89
7.86
7.73
7.70
7.26
7.25
7.25
7.04
6.98
6.96
6.64
4.82
4.80
O
NH
S
N
2.12
2.05
1.22
1.02
0.93
0.95
2.00
f1 (ppm)

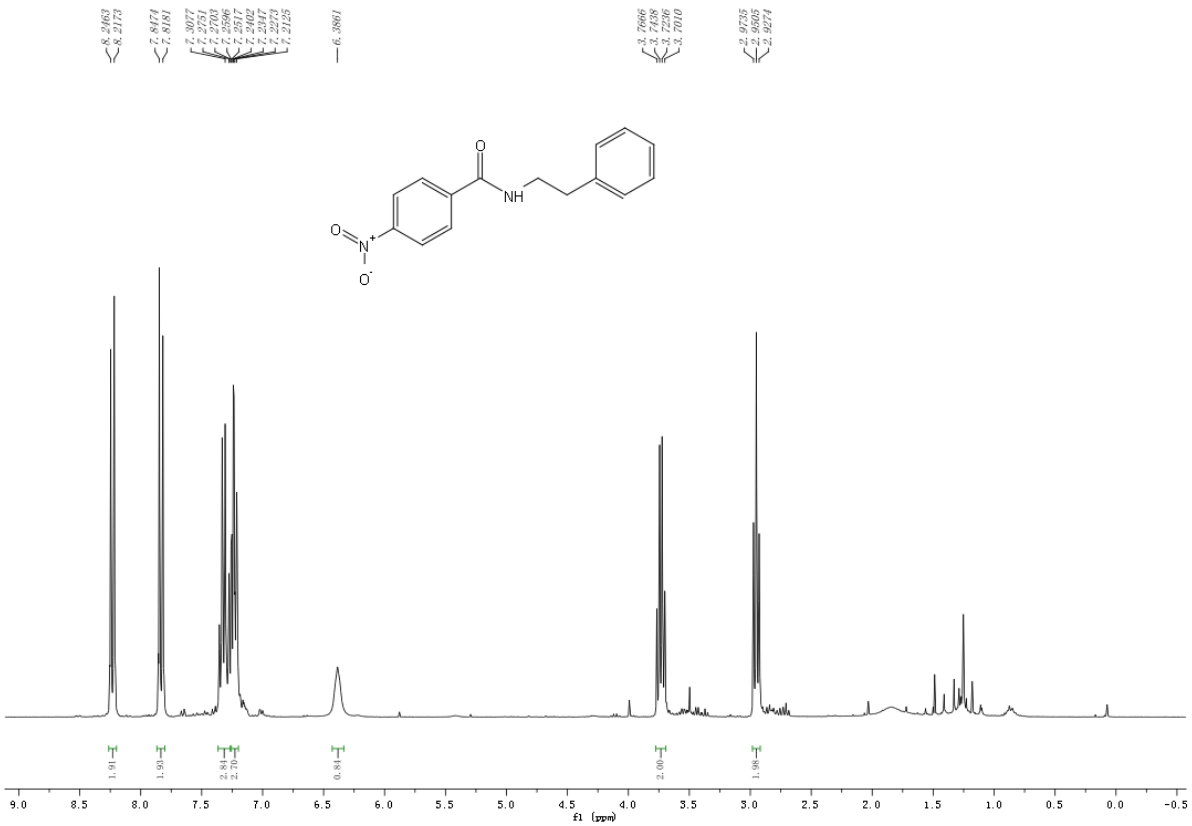

165.34
139.91
138.00
132.44
127.72
127.06
126.58
125.64
117.91
115.22
77.42
77.00
76.58
38.97
O
NH
S
N
f1 (ppm)

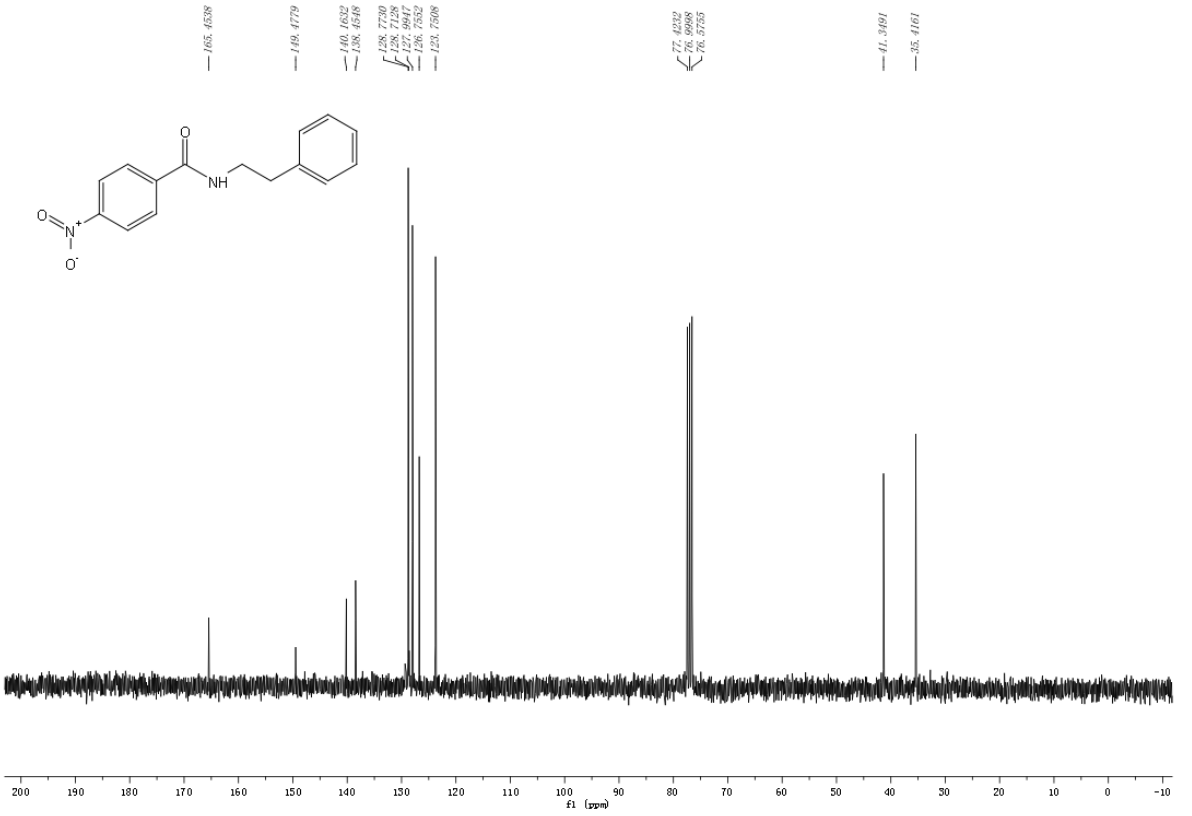

7.90
7.87
7.74
7.71
7.26
6.45
5.93
5.92
5.90
5.89
5.87
5.85
5.28
5.23
5.22
5.22
5.22
5.18
4.10
4.08
4.06
O
NH
N
2.00
2.00
0.92
1.01
2.05
2.09
f1 (ppm)

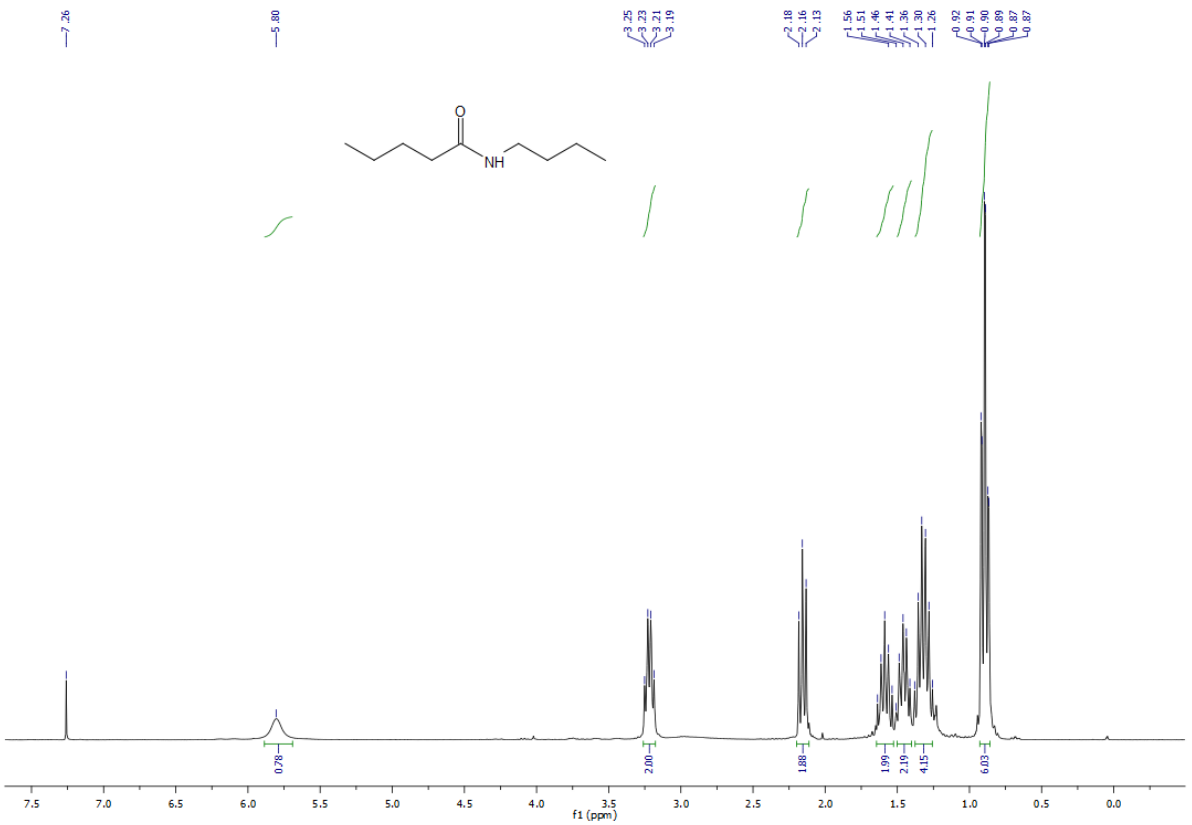

165.52
138.32
133.46
132.41
127.65
117.94
117.21
115.05
77.42
77.00
76.58
42.62
O
NH
N
f1 (ppm)

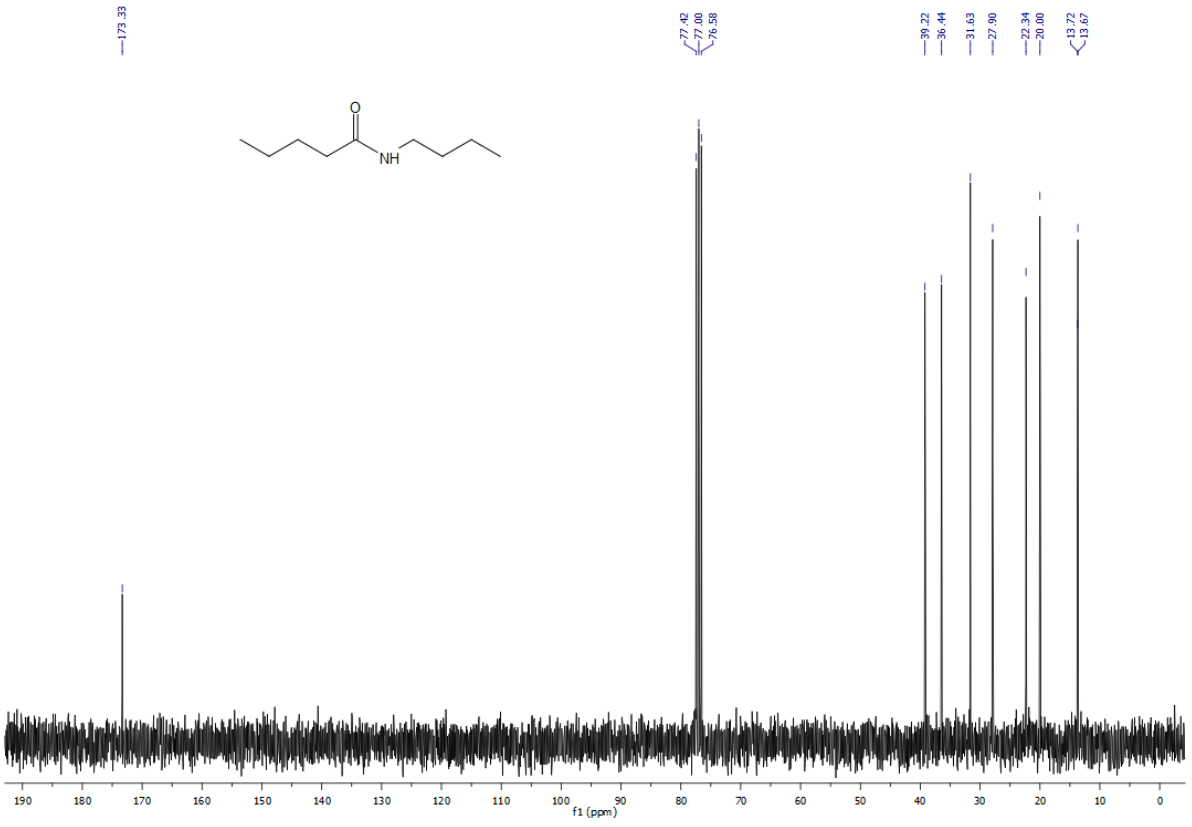

7.92
7.89
7.79
7.76
4.79
4.11
4.10
3.26
3.25
3.25
3.24
3.24
2.58
2.57
2.56
O
NH
N
2.00
2.02
1.99
0.93
f1 (ppm)

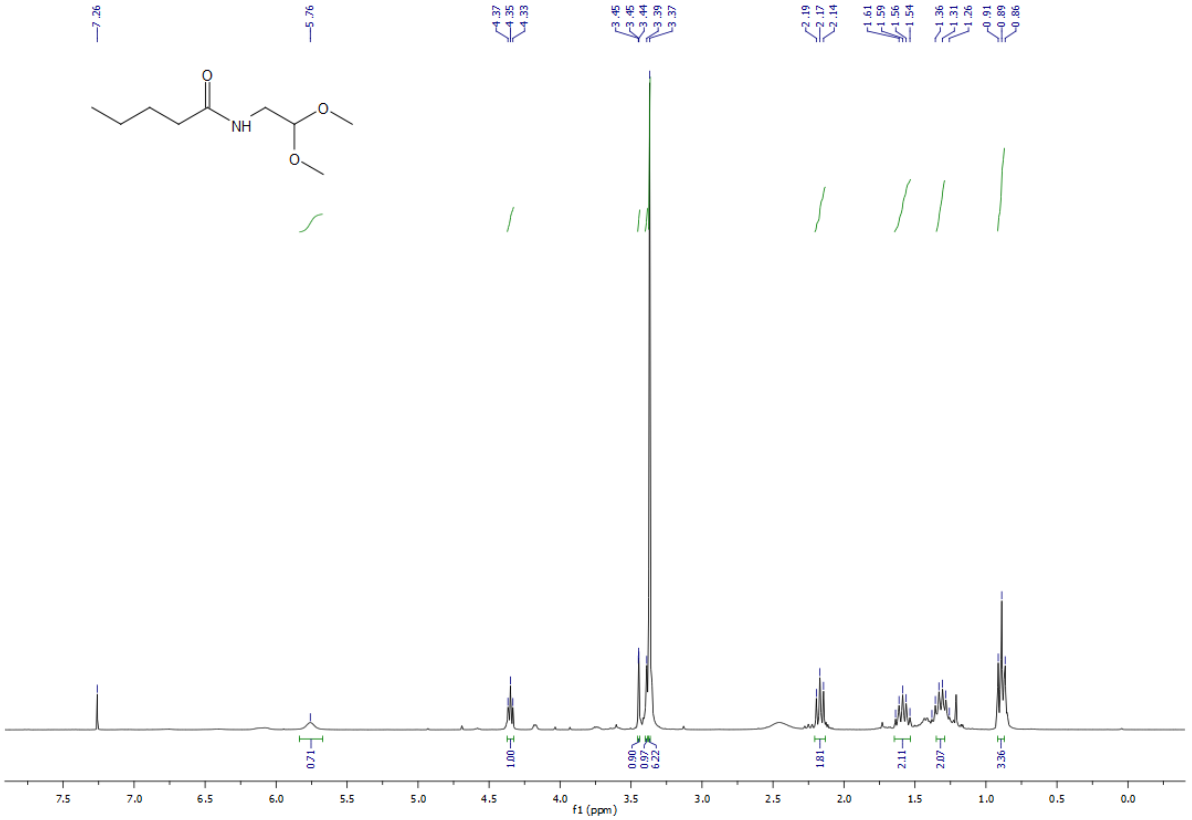

167.91
139.31
133.55
129.26
119.00
116.24
80.38
72.33
49.85
49.57
49.28
48.72
48.43
48.15
30.08
O
NH
N
f1 (ppm)

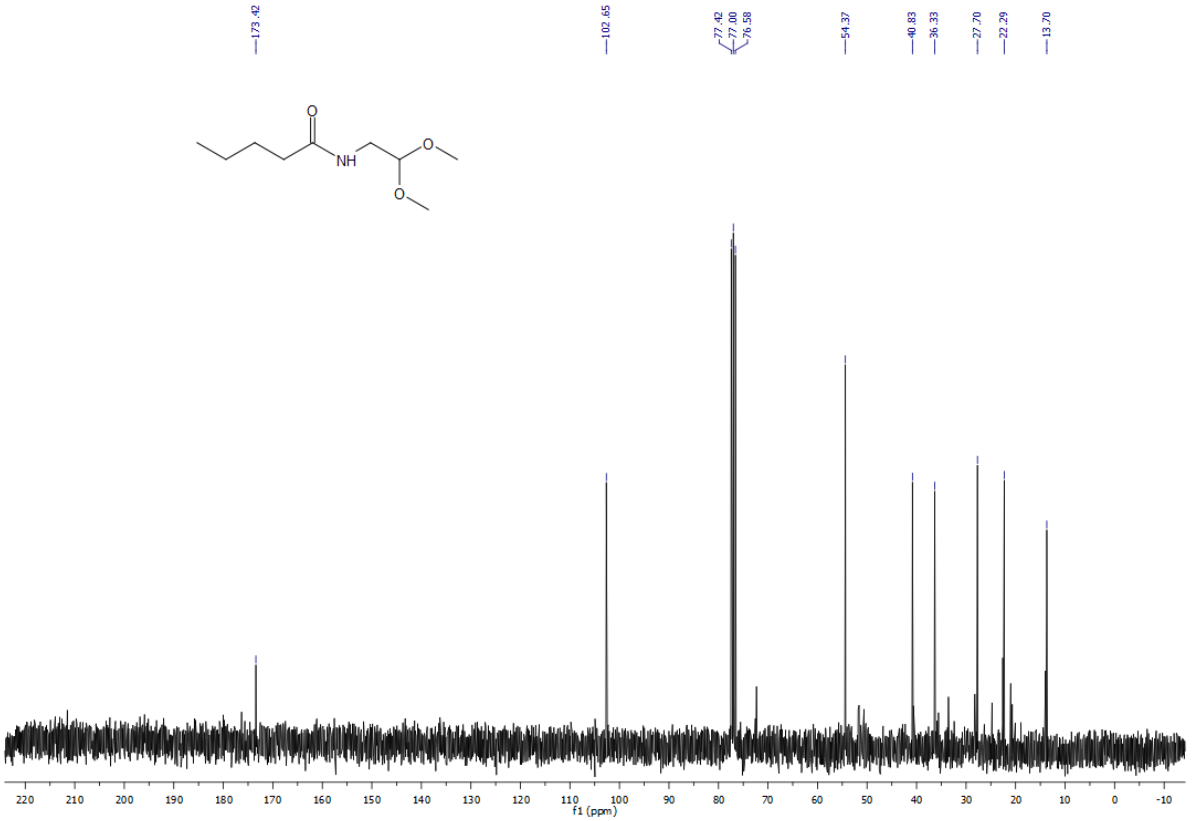

8.2463
8.2173
7.8474
7.8181
7.3077
7.2751
7.2703
7.2596
7.2517
7.2402
7.2341
7.2273
7.2125
6.3861
3.7666
3.7438
3.7236
3.7010
2.9735
2.9385
2.9274
O
NH
O
N+
O-
1.91
1.93
2.84
2.70
0.84
2.00
1.98
f1 (ppm)

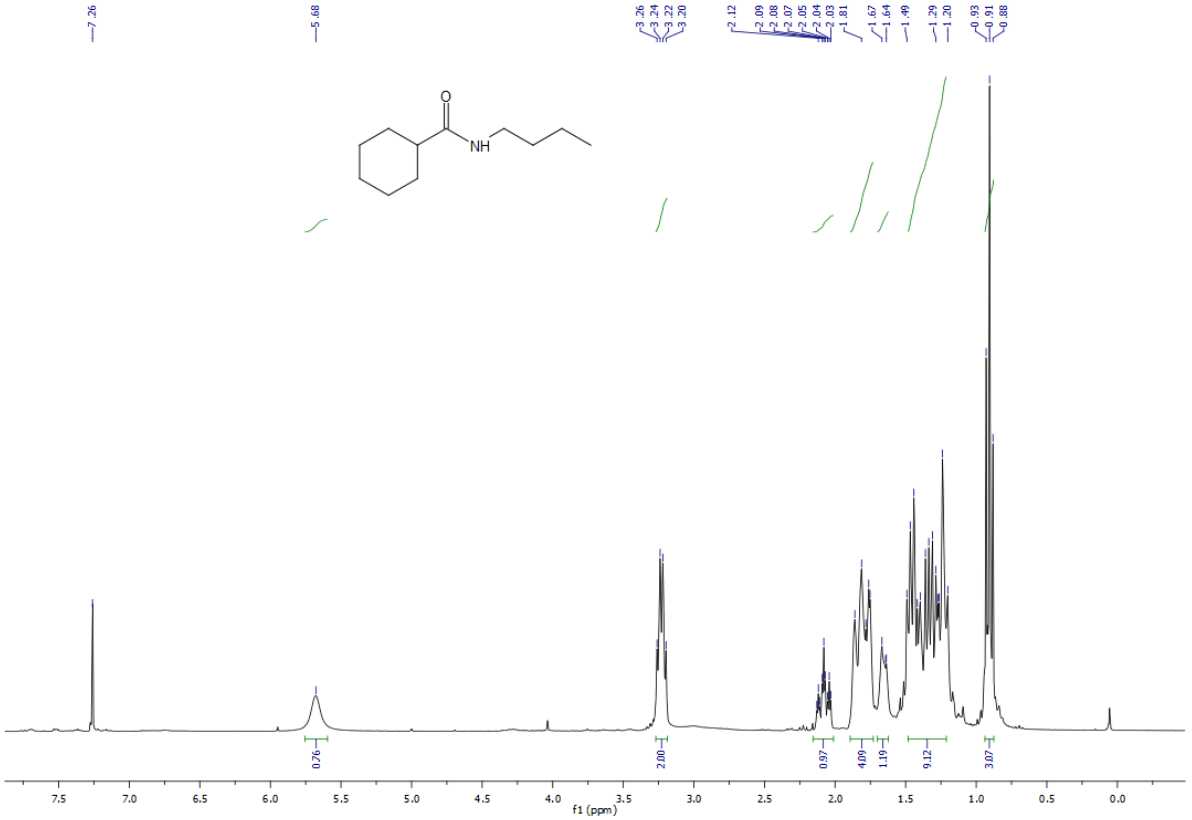

165.4538
149.4179
140.1632
136.4548
128.7730
128.7128
127.9947
126.7552
123.7508
77.4232
76.9998
76.5765
41.3491
35.4161
O
NH
O
N+
O-
f1 (ppm)

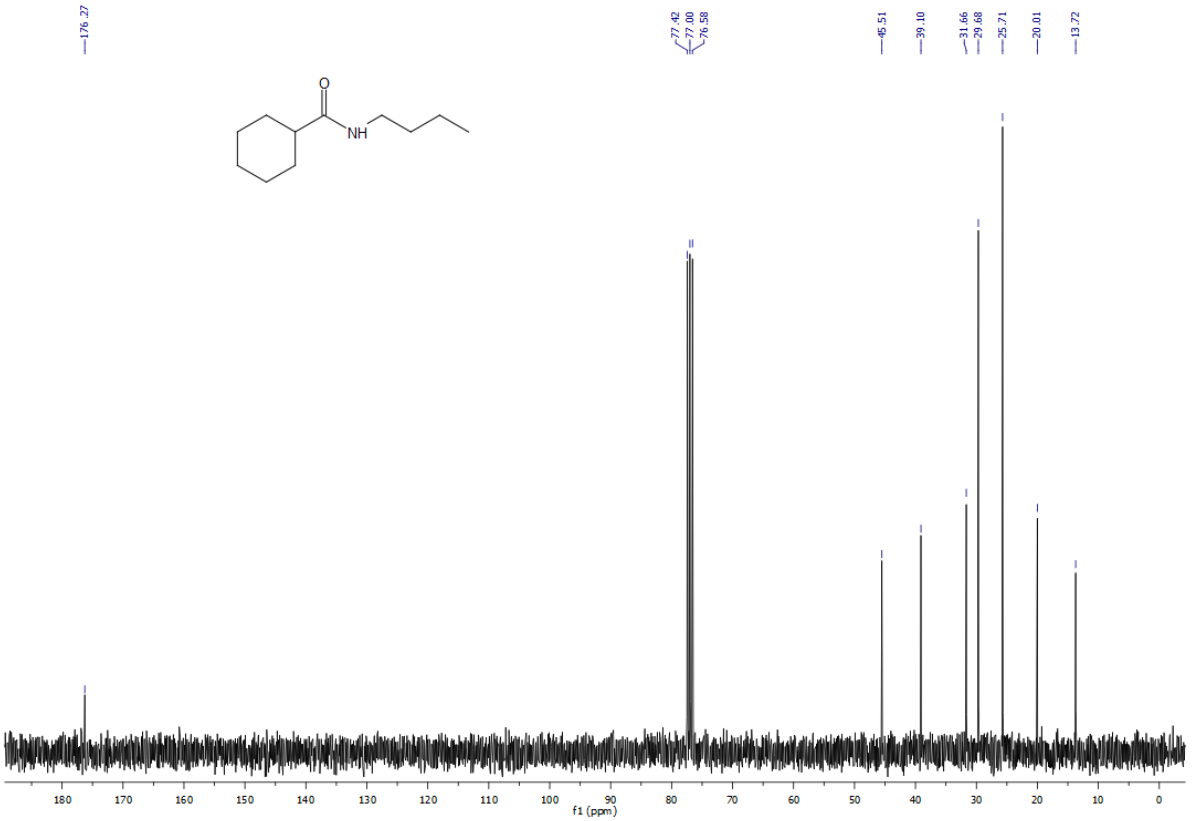


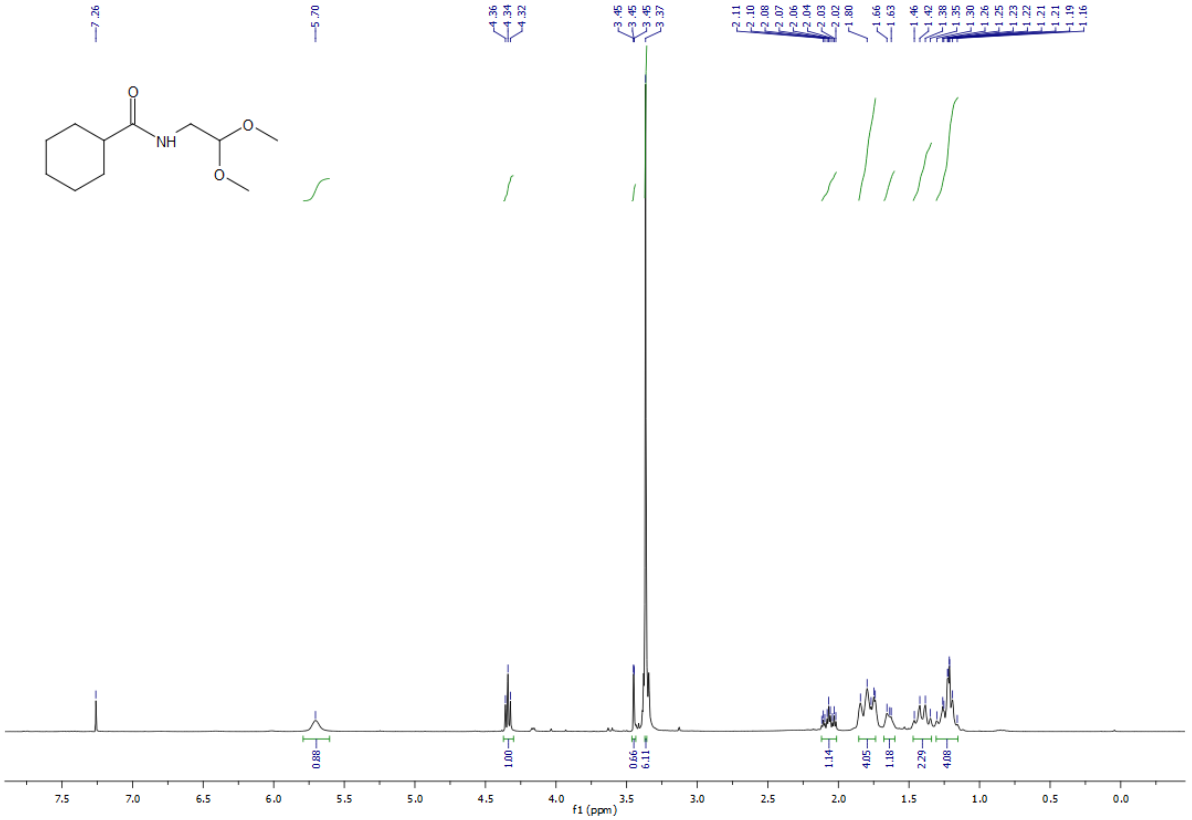







7.26
5.68
3.26
3.24
3.22
3.20
2.12
2.09
2.08
2.07
2.05
2.04
2.03
1.81
1.67
1.64
1.49
1.29
1.20
0.93
0.91
0.88
O
NH
0.76
2.00
0.97
4.09
1.19
9.12
3.07
f1 (ppm)


176.27
77.42
77.00
76.58
45.51
39.10
31.66
29.68
25.71
20.01
13.72
O
NH
f1 (ppm)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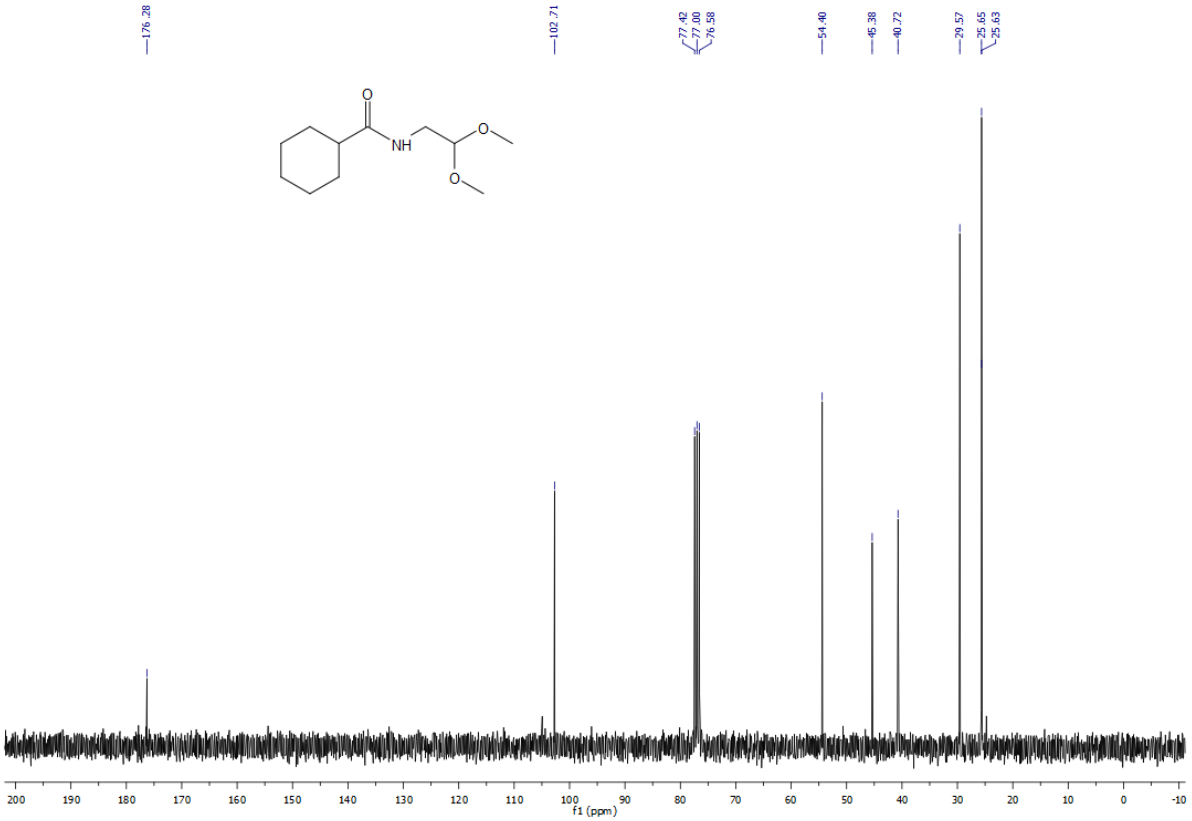
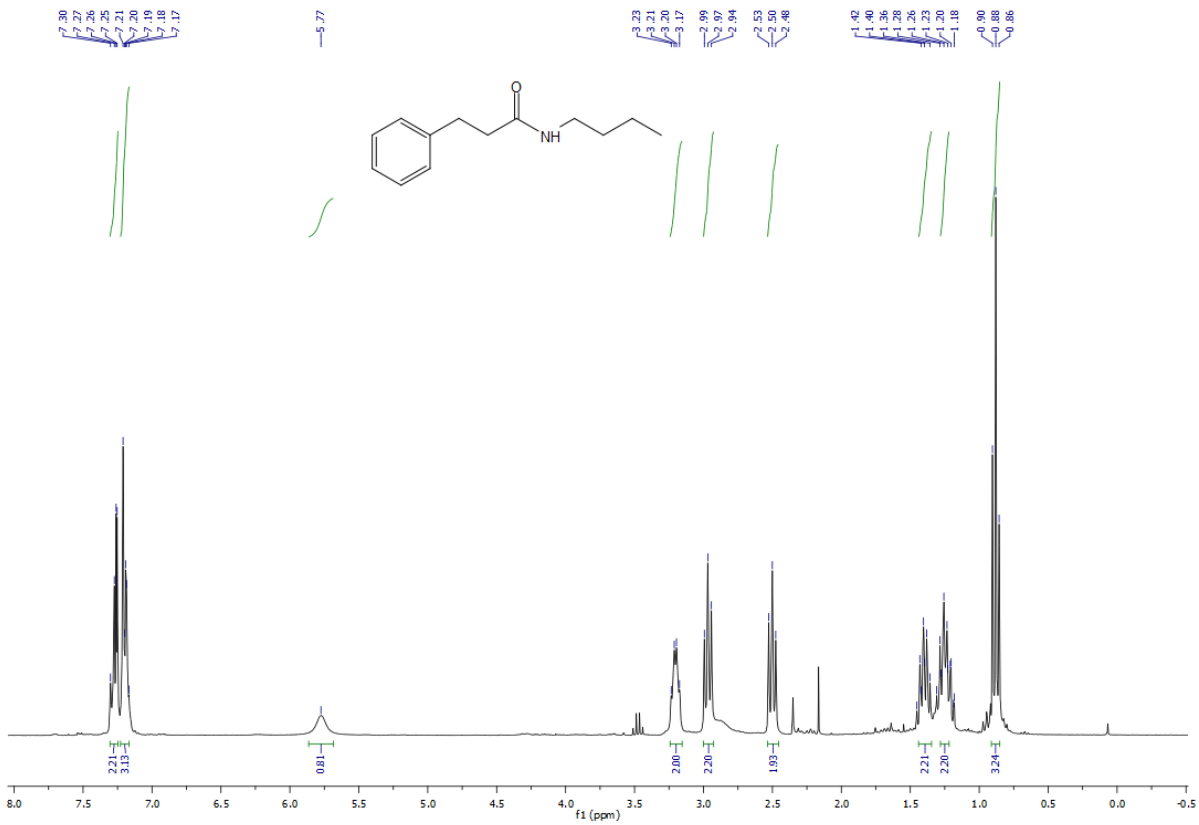
5.70
4.36
4.34
4.32
3.45
3.45
3.45
3.37
2.11
2.10
2.08
2.07
2.06
2.04
2.03
2.02
1.80
1.66
1.63
1.46
1.42
1.38
1.35
1.30
1.26
1.25
1.23
1.22
1.21
1.21
1.19
1.16
O
NH
O
O
0.88
1.00
0.66
6.11
1.14
4.05
1.18
2.29
4.08
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


176.28
102.71
77.42
77.00
76.58
54.40
45.38
40.72
29.57
25.65
25.63
O
NH
O
O
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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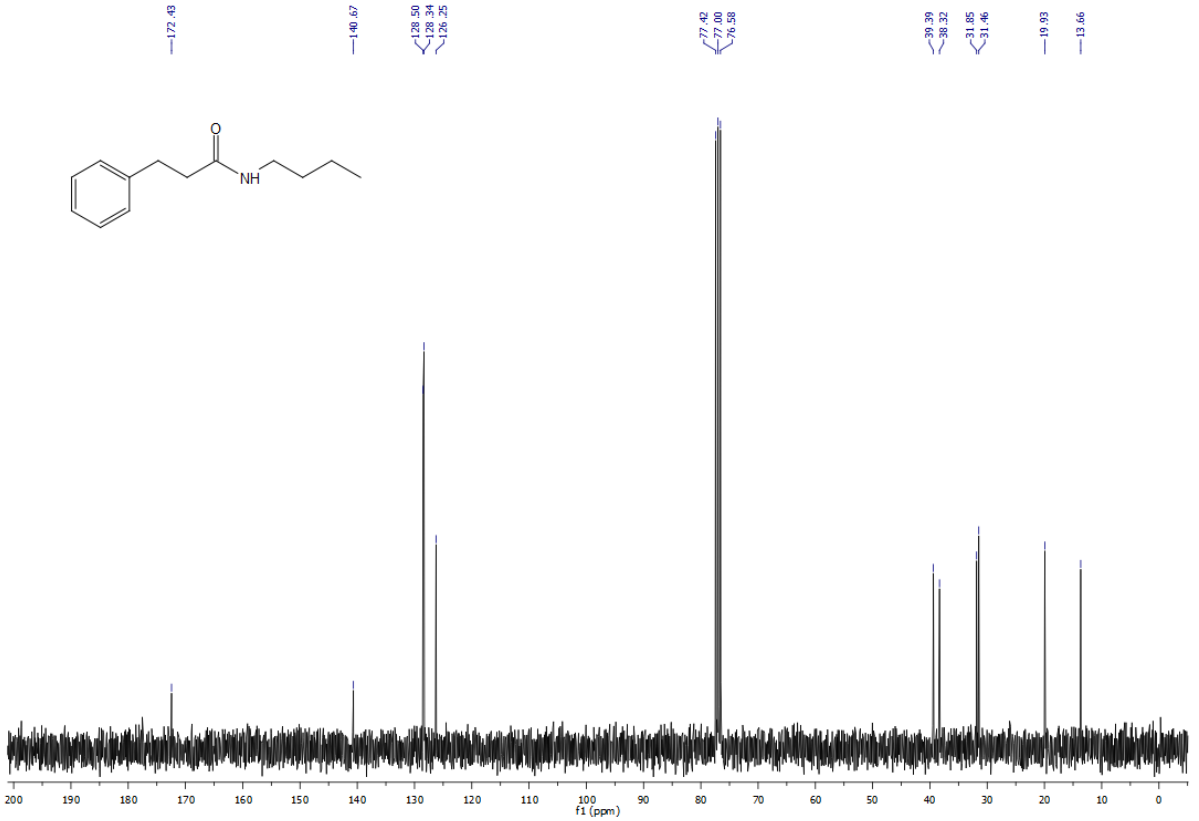
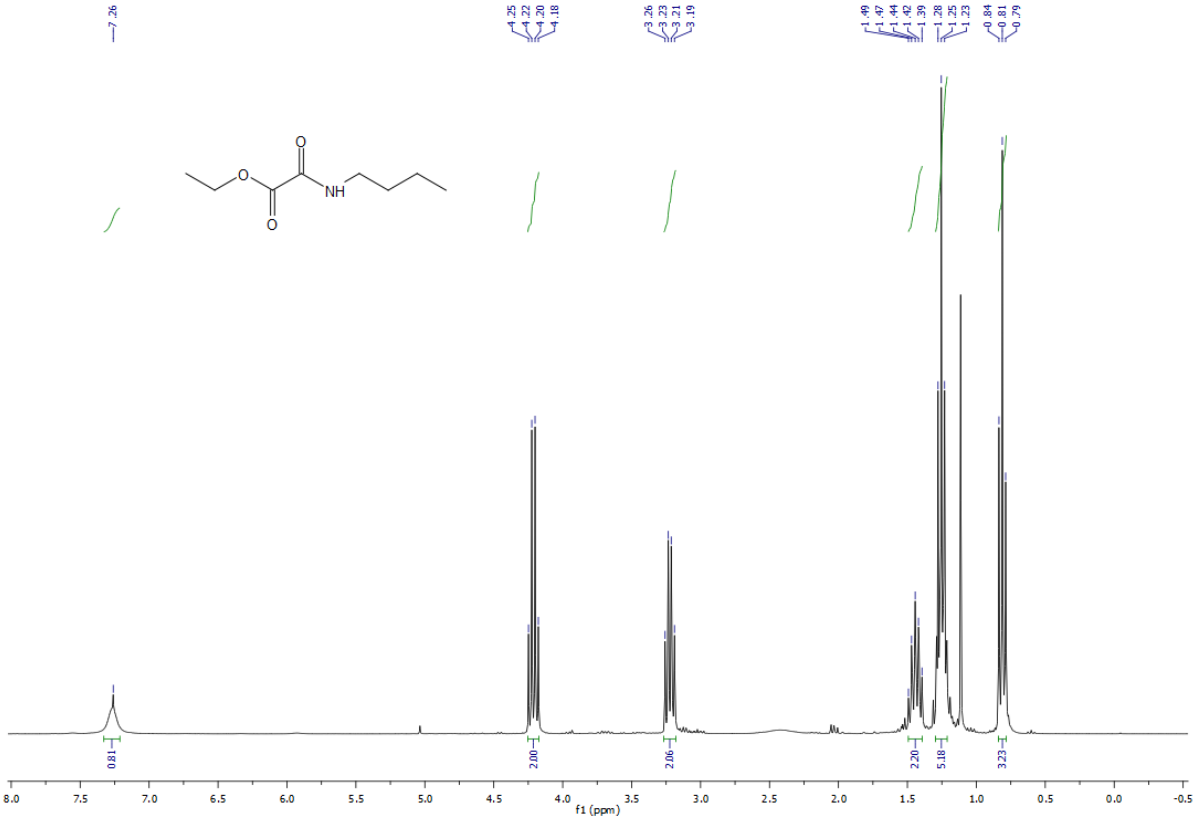
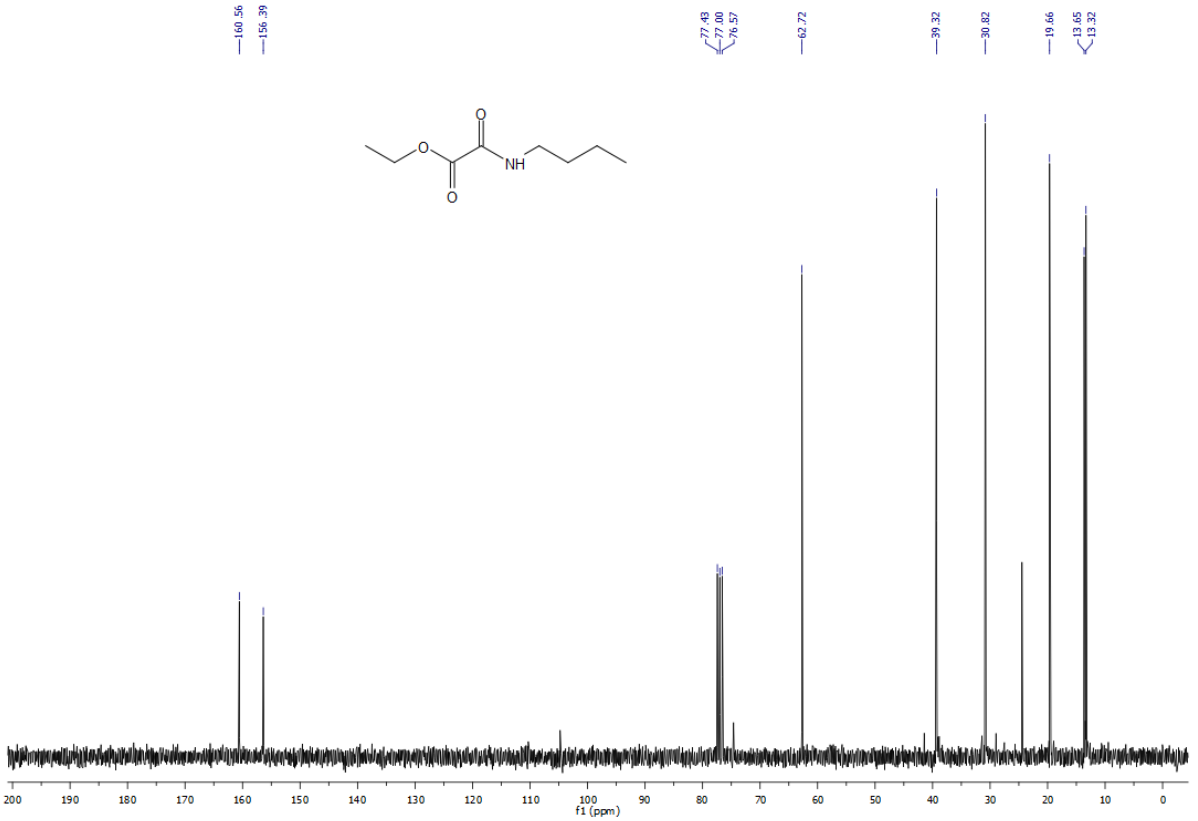
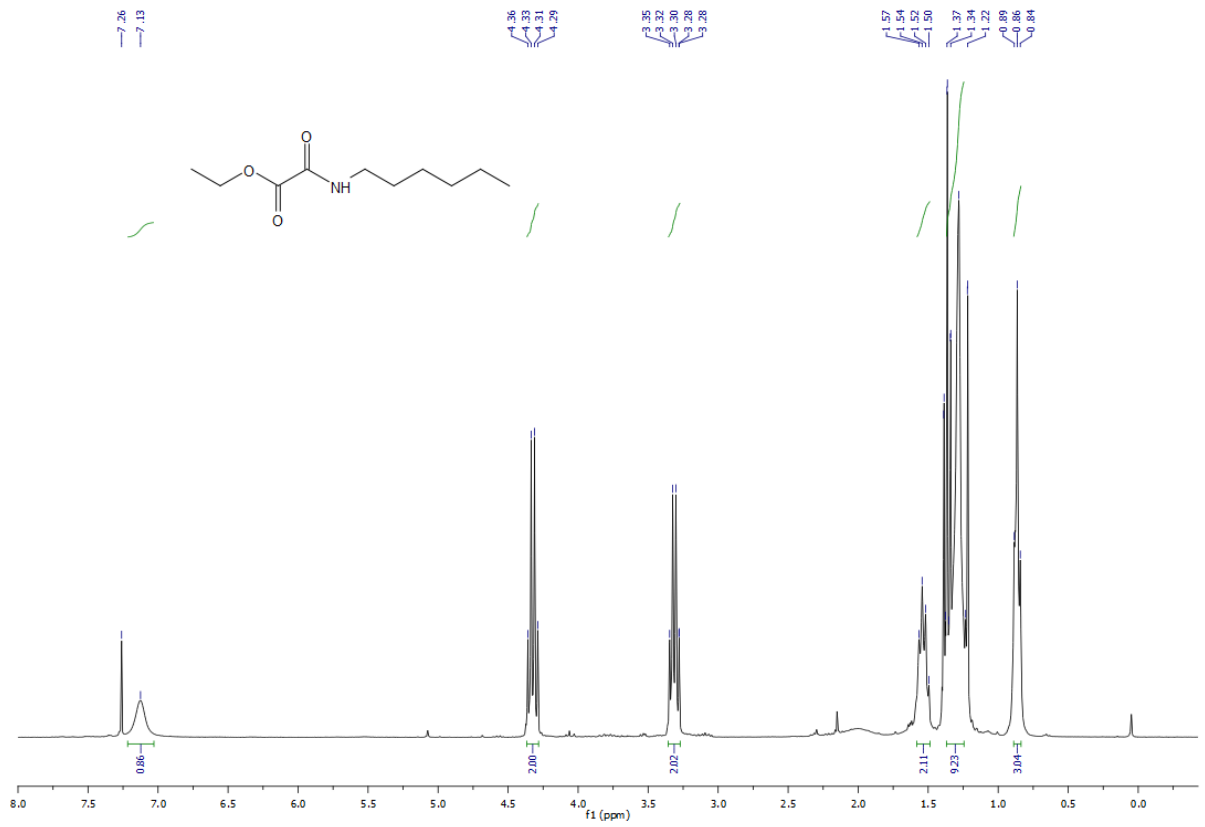
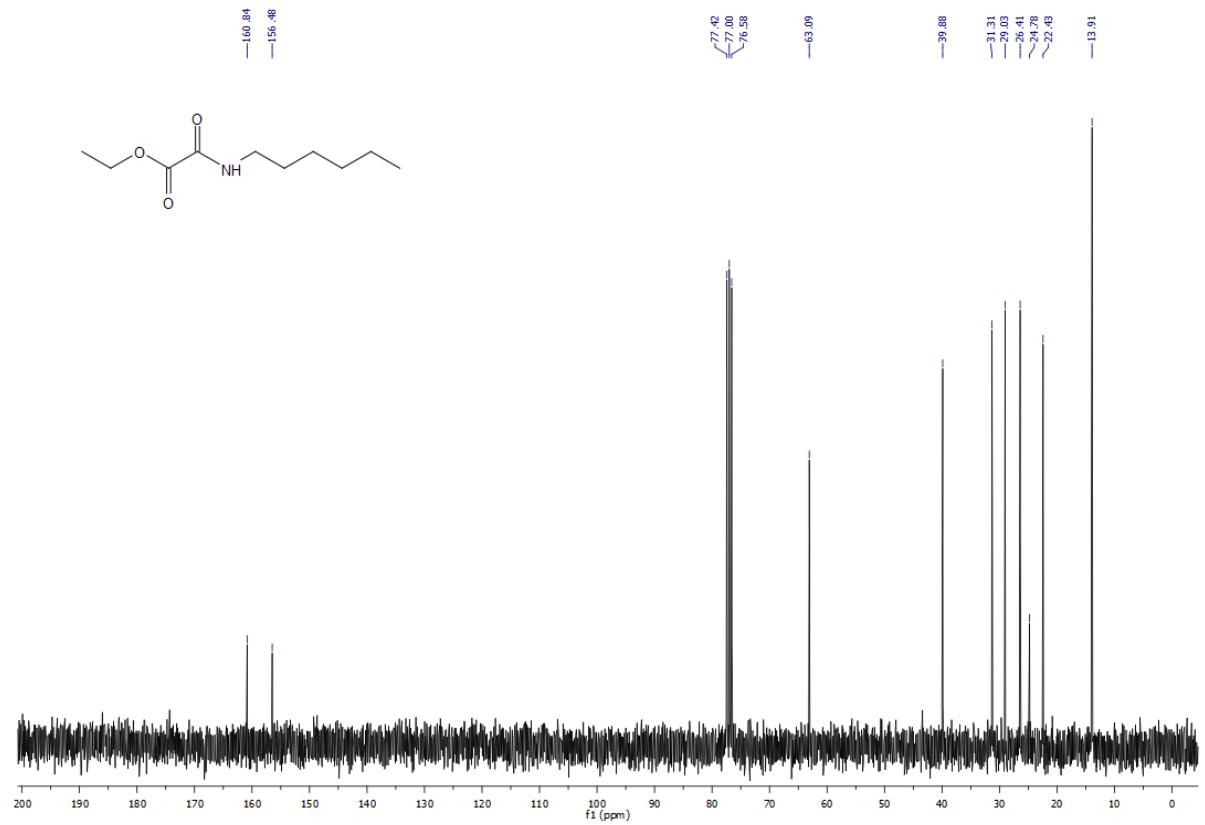
7.27
7.26
7.25
7.21
7.20
7.19
7.18
7.17
5.77
3.23
3.21
3.20
3.17
2.99
2.97
2.94
2.53
2.50
2.48
1.42
1.40
1.36
1.28
1.26
1.23
1.20
1.18
0.90
0.88
0.86
O
NH
2.21
3.13
0.81
2.00
2.20
1.93
2.21
2.20
3.24
f1 (ppm)

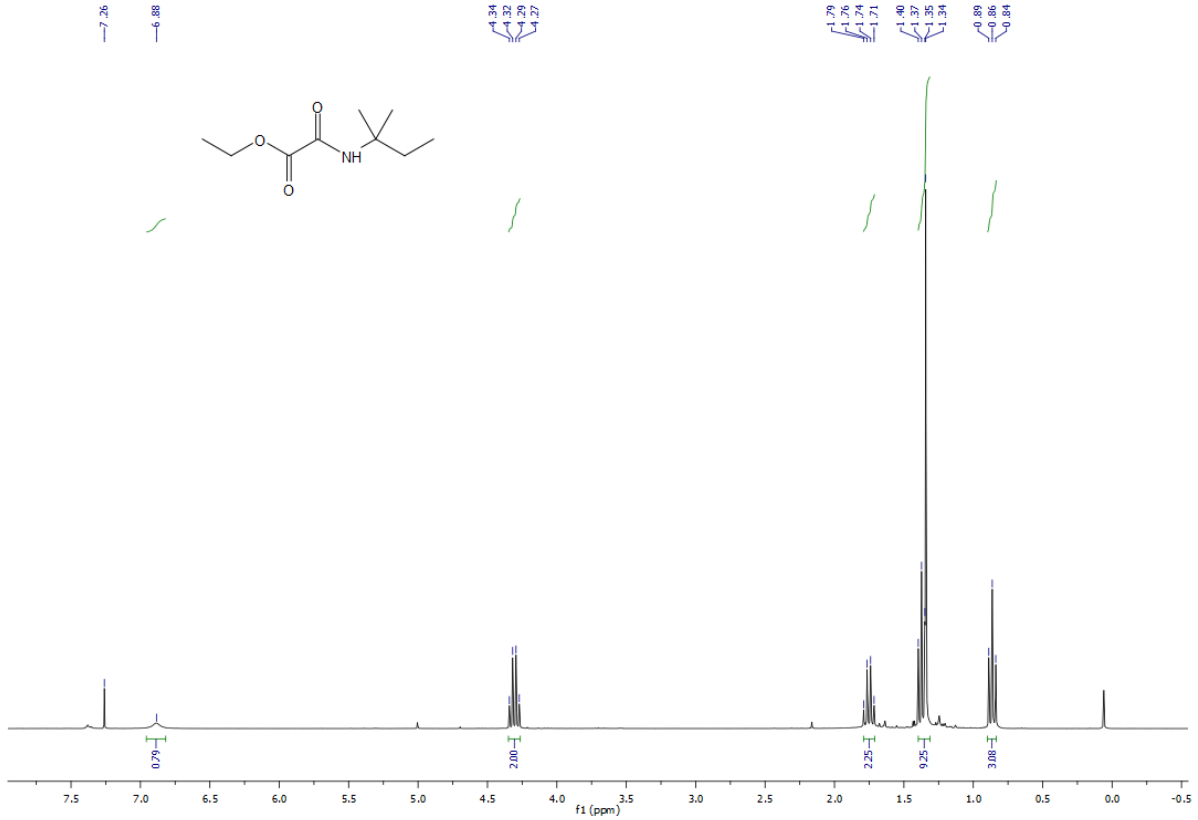

172.43
140.67
128.50
128.34
126.25
77.42
77.00
76.58
39.39
38.32
31.85
31.46
19.93
13.66
O
NH
f1 (ppm)

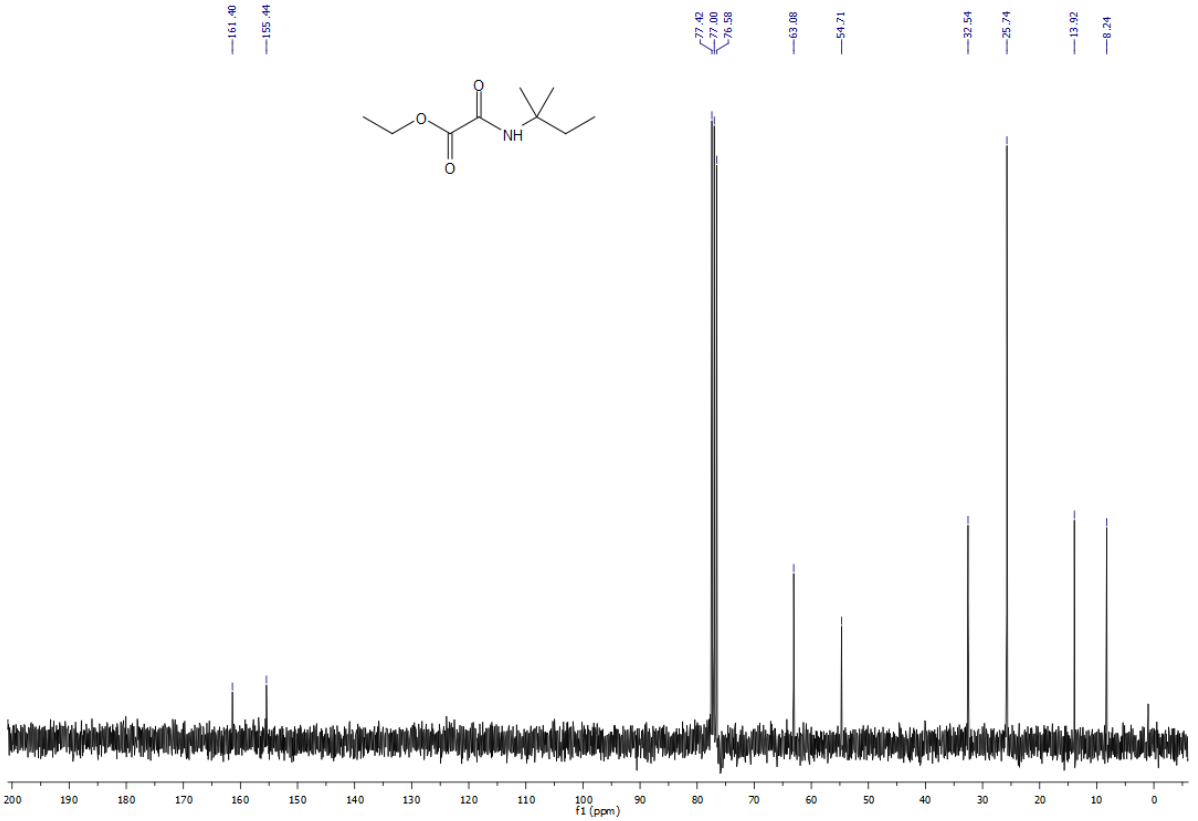

7.26
4.25
4.22
4.20
4.18
3.26
3.23
3.21
3.19
1.49
1.47
1.44
1.42
1.39
1.28
1.25
1.23
0.84
0.81
0.79
O
O
NH
O
0.81
2.00
2.06
2.20
5.18
3.23
f1 (ppm)

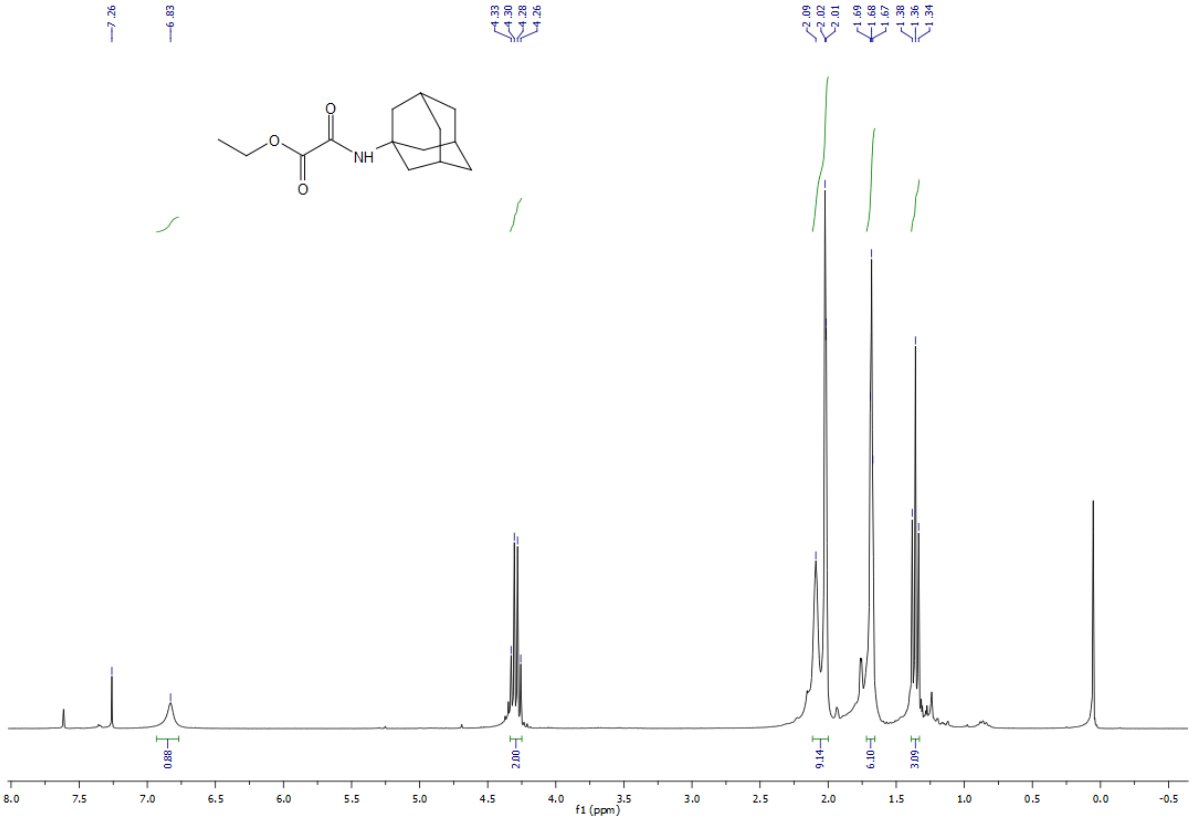

160.56
156.39
77.43
77.00
76.57
62.72
39.32
30.82
19.66
13.65
13.32
O
O
NH
O
f1 (ppm)

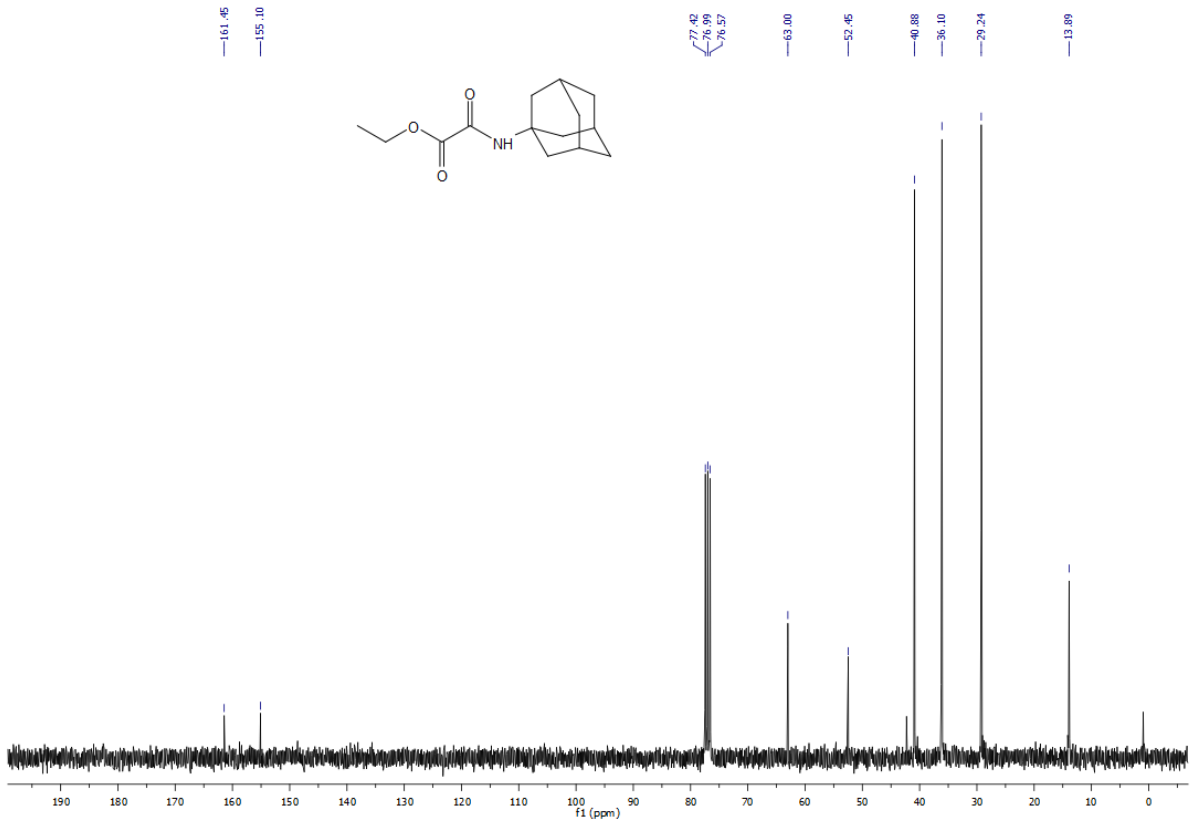

7.26
7.13
4.36
4.33
4.31
4.29
3.35
3.32
3.30
3.28
3.28
1.57
1.54
1.52
1.50
1.37
1.34
1.22
0.89
0.86
0.84
O
O
NH
O
0.86
2.00
2.02
2.11
9.23
3.04
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

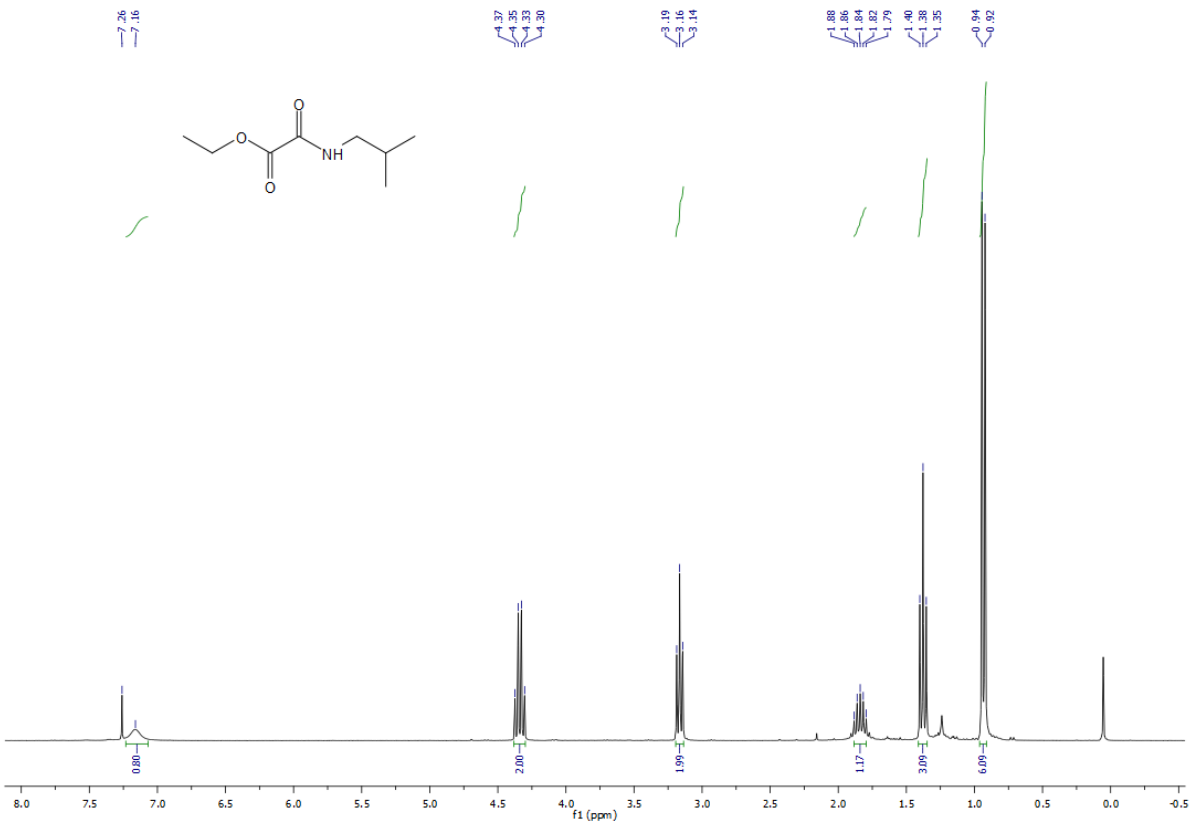

160.84
156.48
77.42
77.00
76.58
63.09
39.88
31.31
29.03
26.41
24.78
22.43
13.91
O
O
NH
O
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

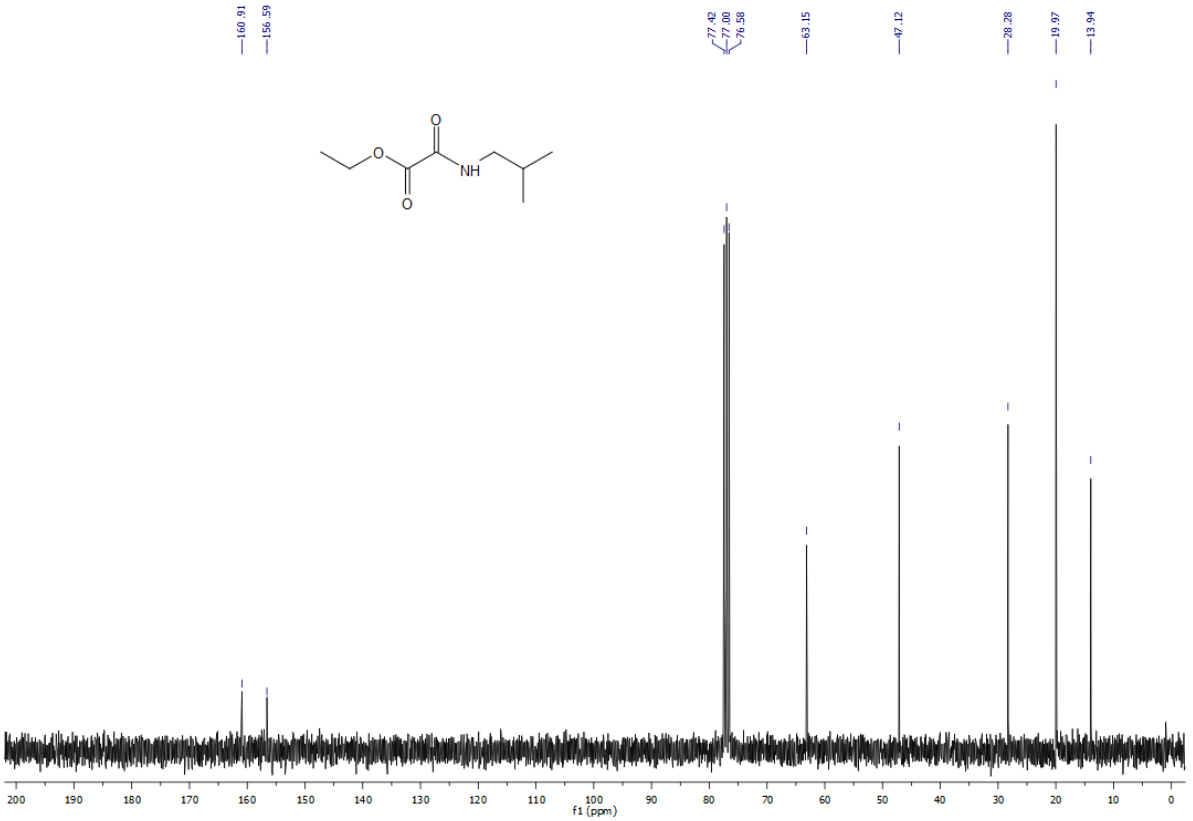

7.26
6.88
4.34
4.32
4.29
4.27
1.79
1.76
1.74
1.71
1.40
1.37
1.35
1.34
0.89
0.86
0.84
O
O
NH
O
0.79
2.00
2.25
9.25
3.08
f1 (ppm)

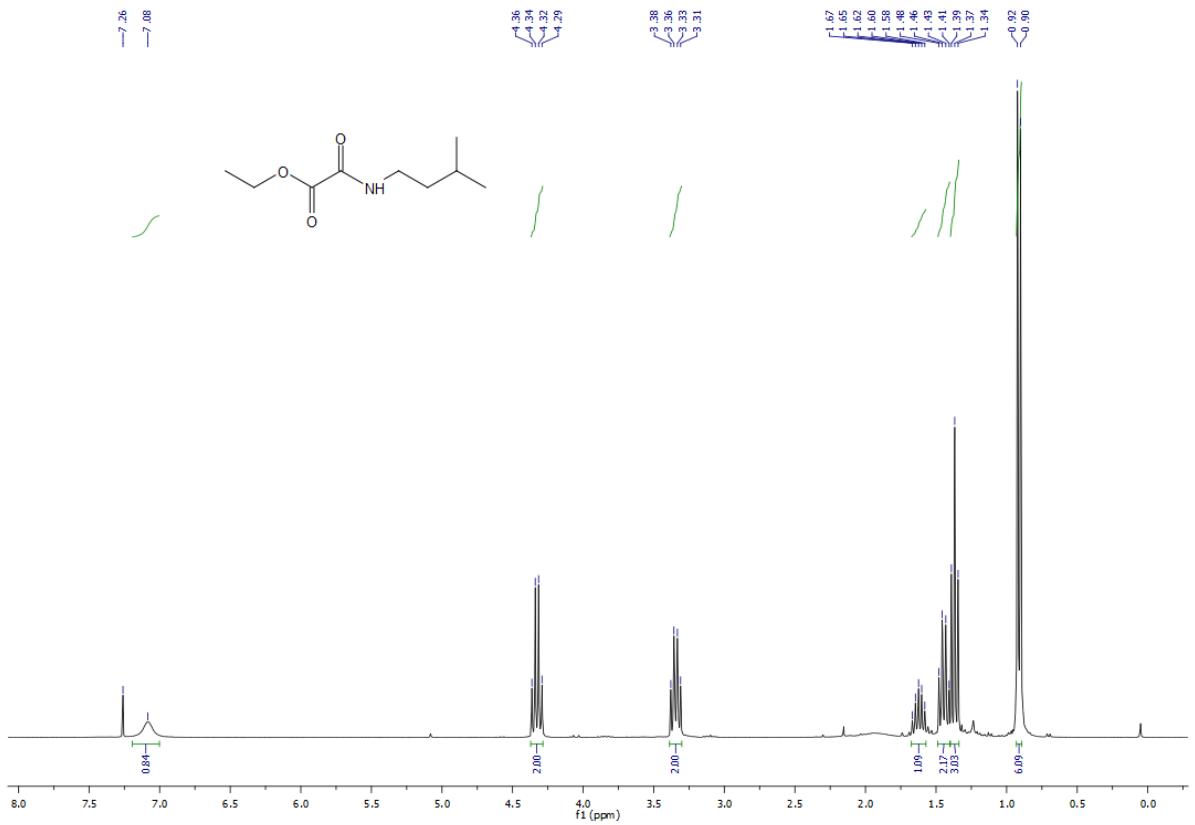

161.40
155.44
77.42
77.00
76.58
63.08
54.71
32.54
25.74
13.92
8.24
O
O
NH
O
f1 (ppm)

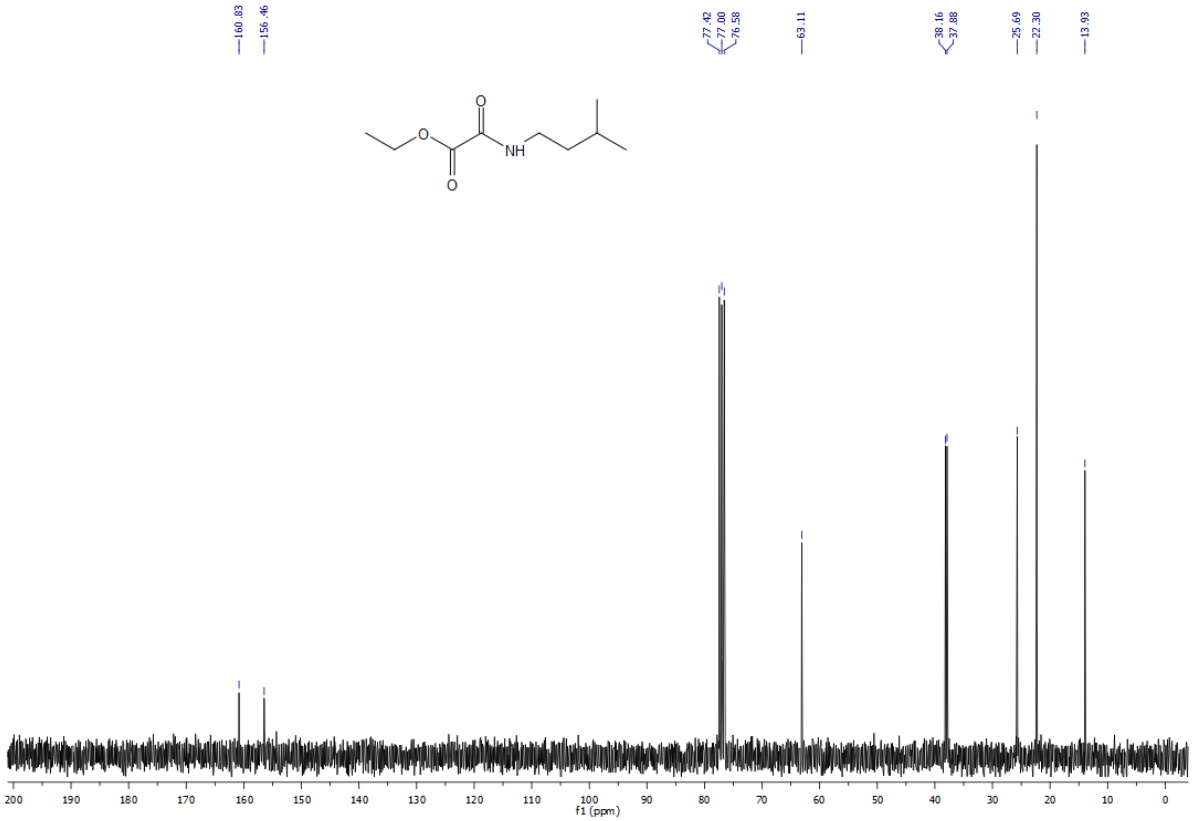


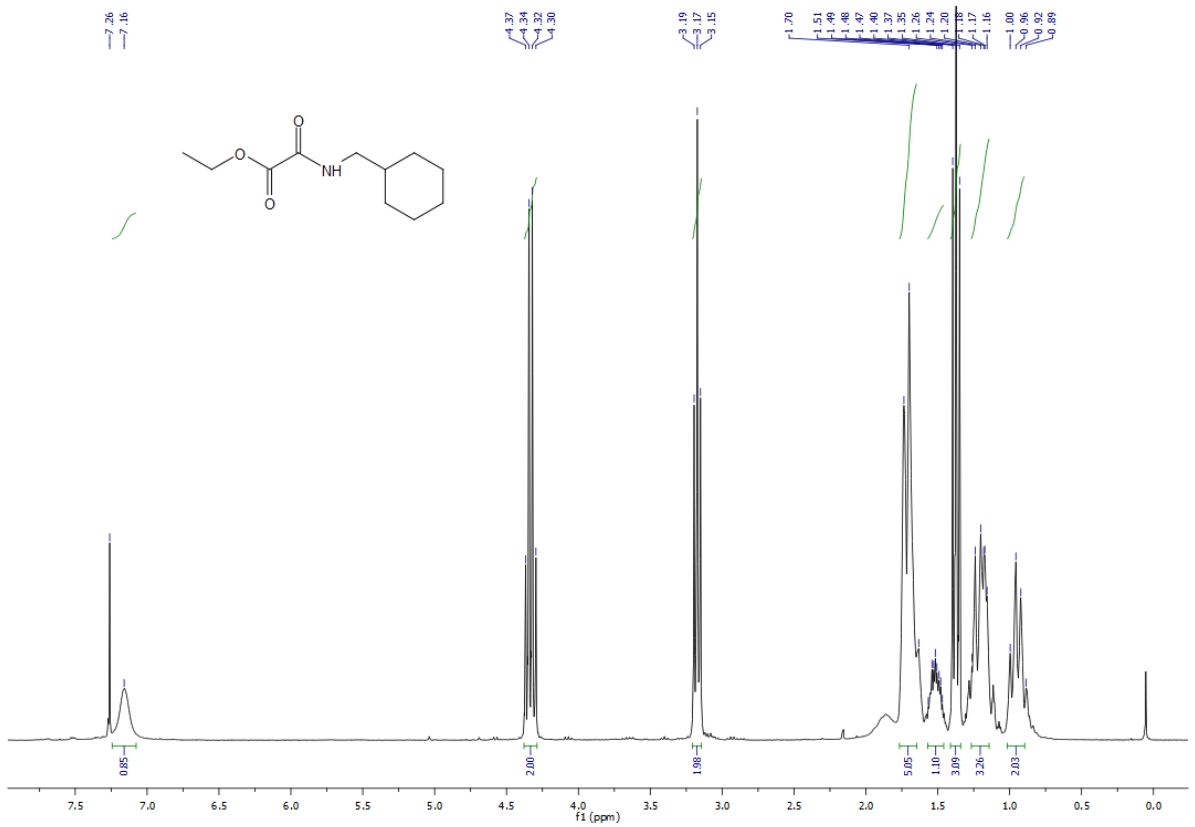


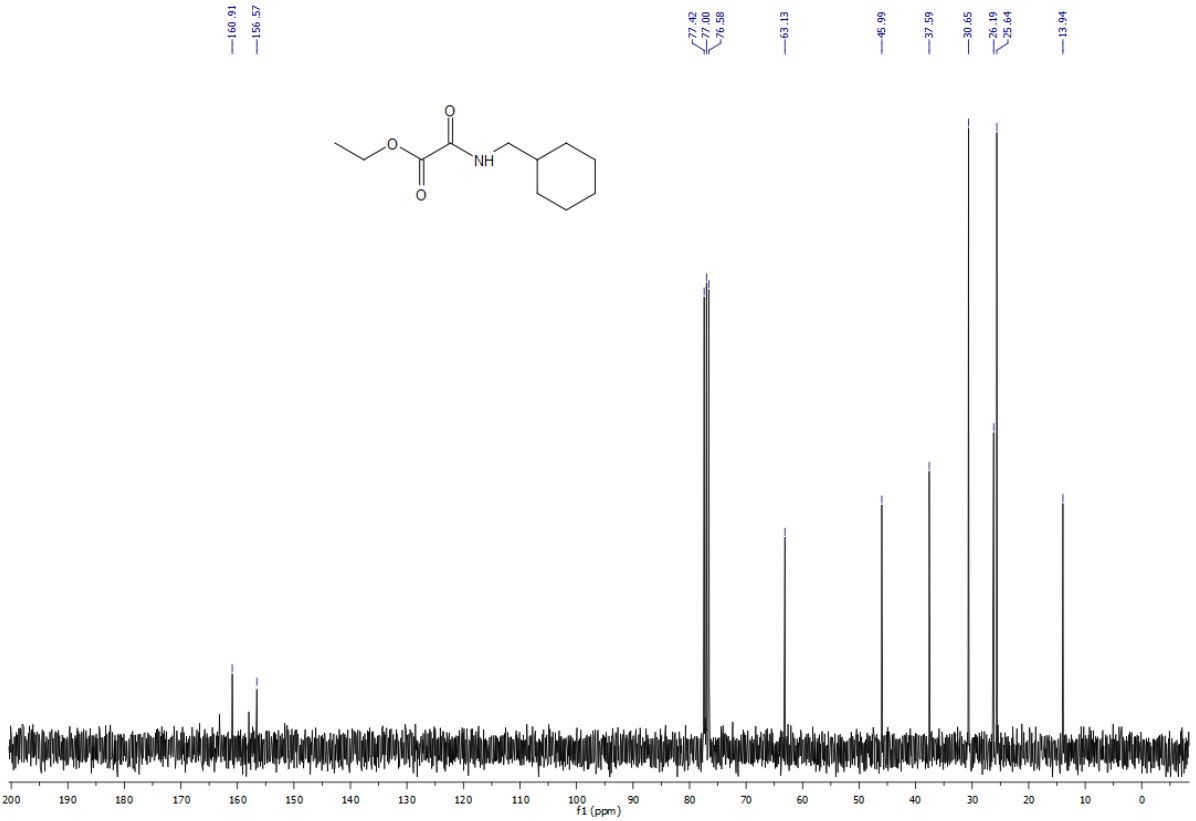

7.26
7.16
4.37
4.35
4.33
4.30
3.19
3.16
3.14
1.88
1.86
1.84
1.82
1.79
1.40
1.38
1.35
0.94
0.92
O
O
NH
O
0.80
2.00
1.99
1.17
3.09
6.09
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

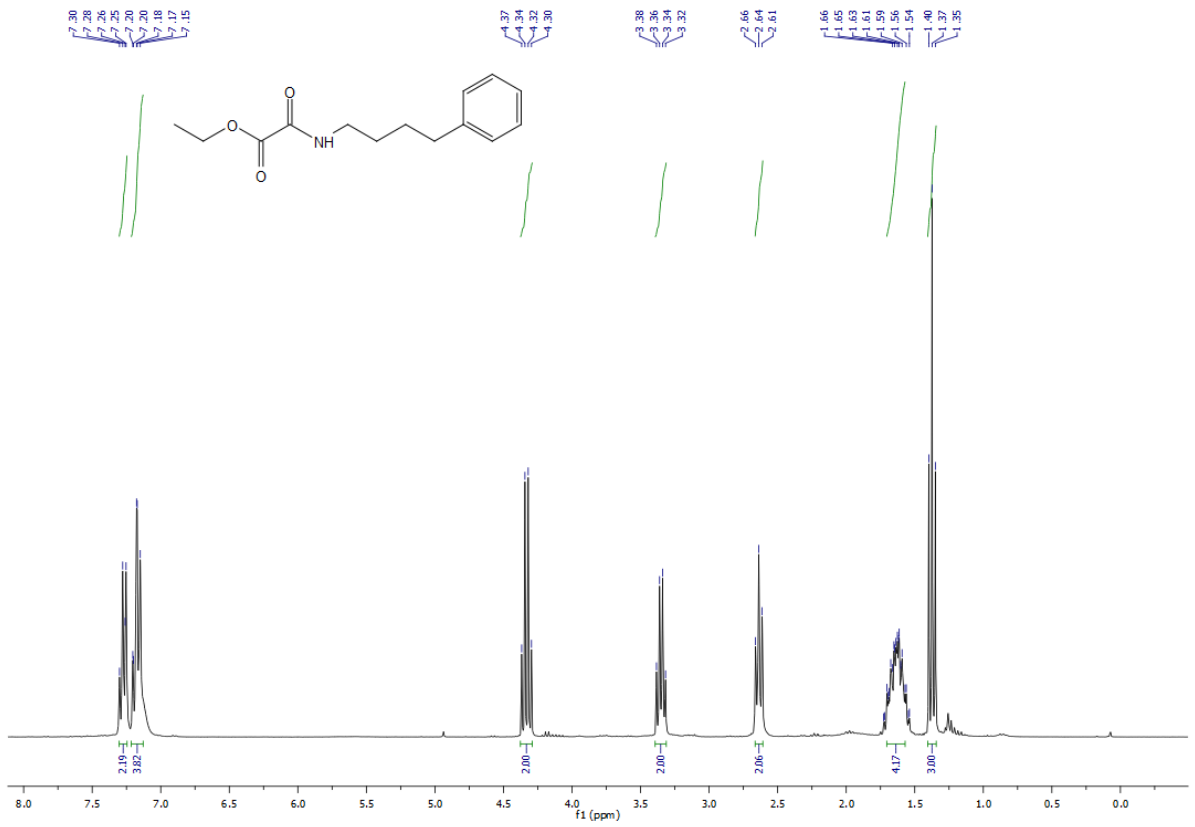

160.91
156.59
77.42
77.00
76.58
63.15
47.12
28.28
19.97
13.94
O
O
NH
O
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

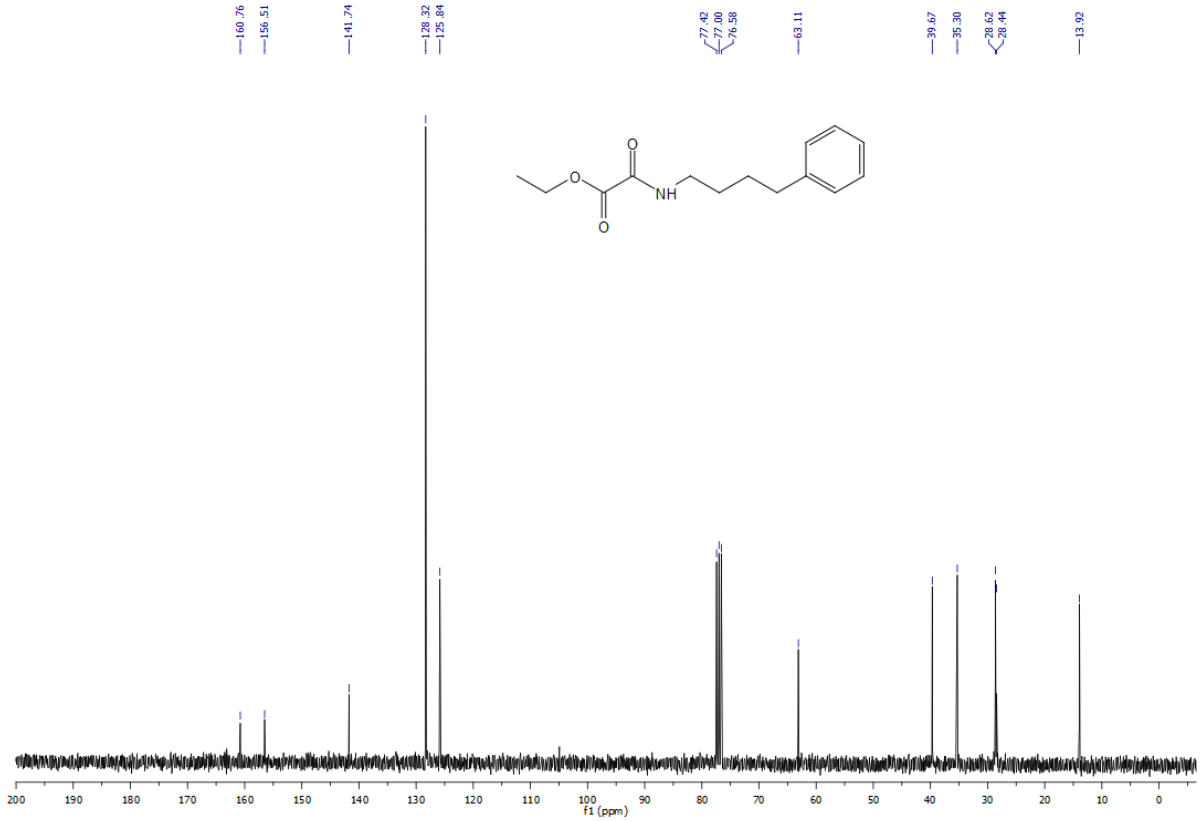

7.26
7.08
4.36
4.34
4.32
4.29
3.38
3.36
3.33
3.31
1.67
1.65
1.62
1.60
1.58
1.48
1.46
1.43
1.41
1.39
1.37
1.34
0.92
0.90
O
O
O
NH
0.84
2.00
2.00
1.09
2.17
3.03
6.09
f1 (ppm)

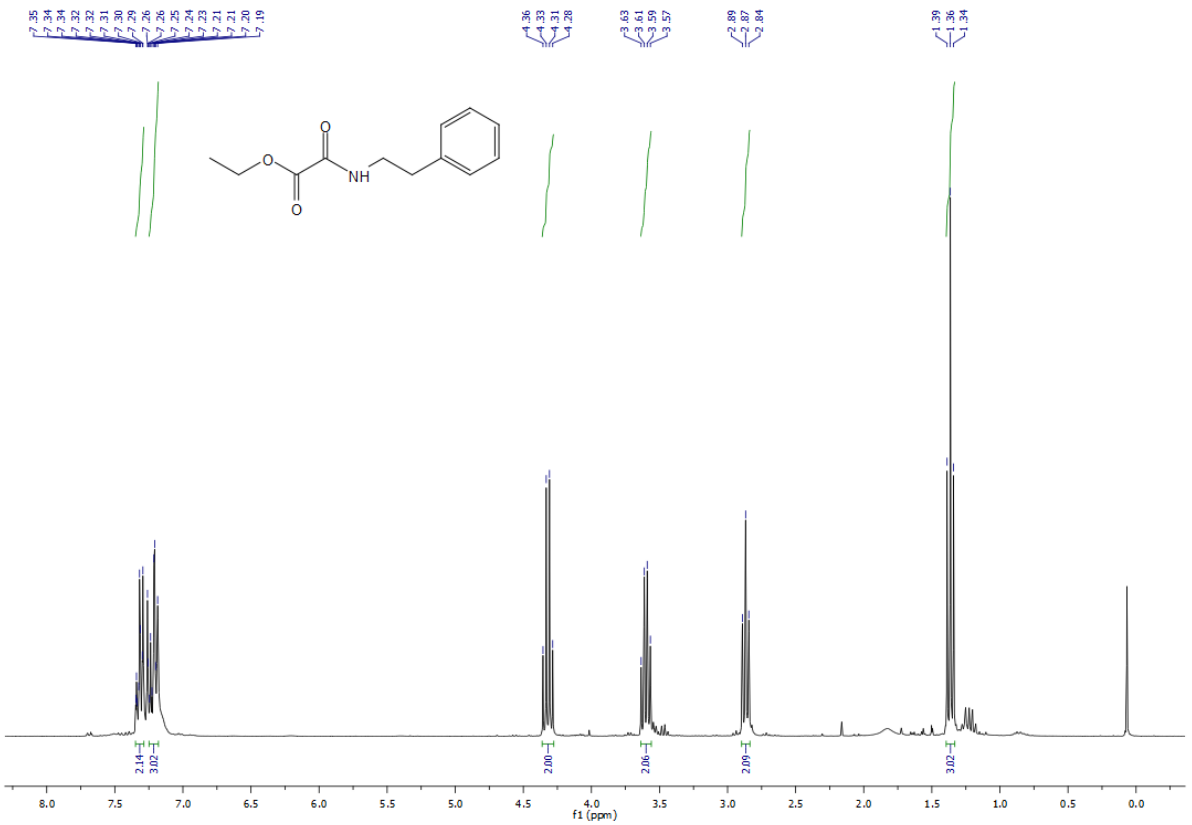

160.83
156.46
77.42
77.00
76.58
63.11
38.16
37.88
25.69
22.30
13.93
O
O
O
NH
f1 (ppm)

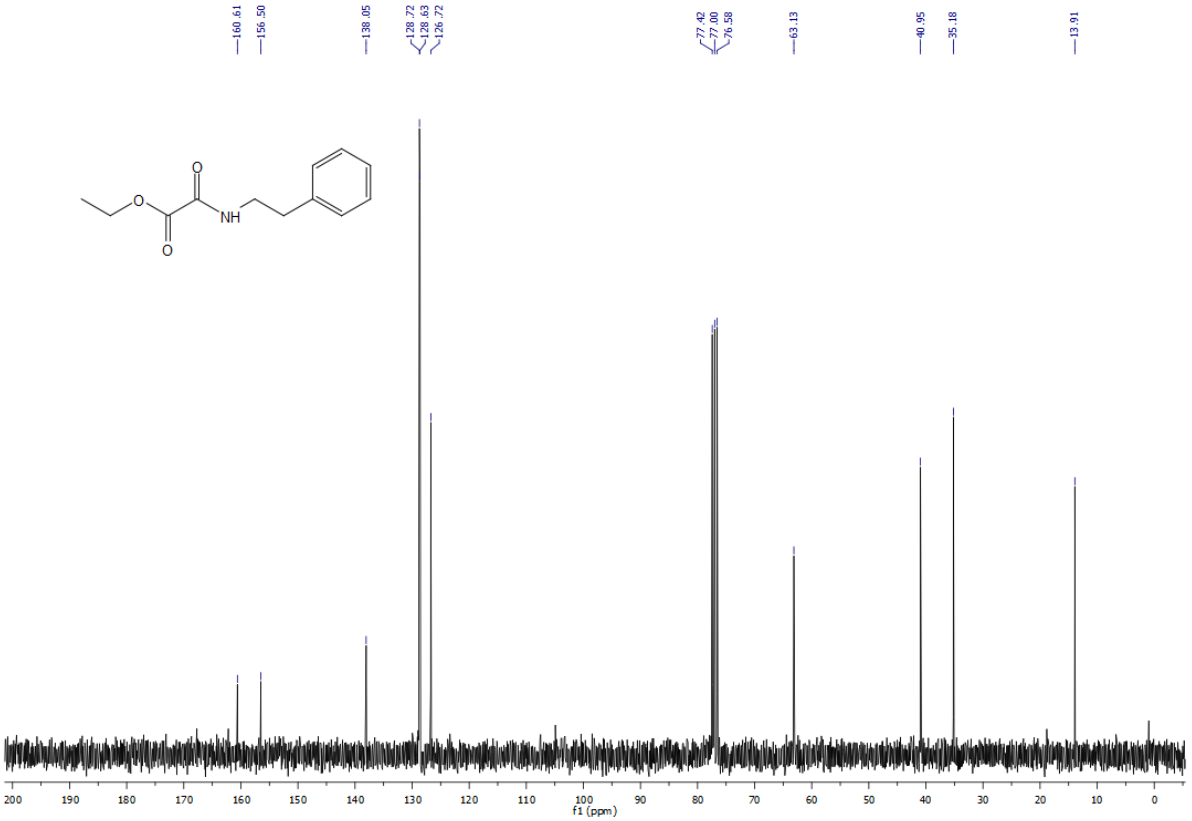

7.26
7.16
4.37
4.34
4.32
4.30
3.19
3.17
3.15
1.70
1.51
1.49
1.48
1.47
1.40
1.37
1.35
1.26
1.24
1.20
1.18
1.17
1.16
1.00
0.96
0.92
0.89
O
O
NH
O
0.85
2.00
1.98
5.05
1.10
3.09
3.26
2.03
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

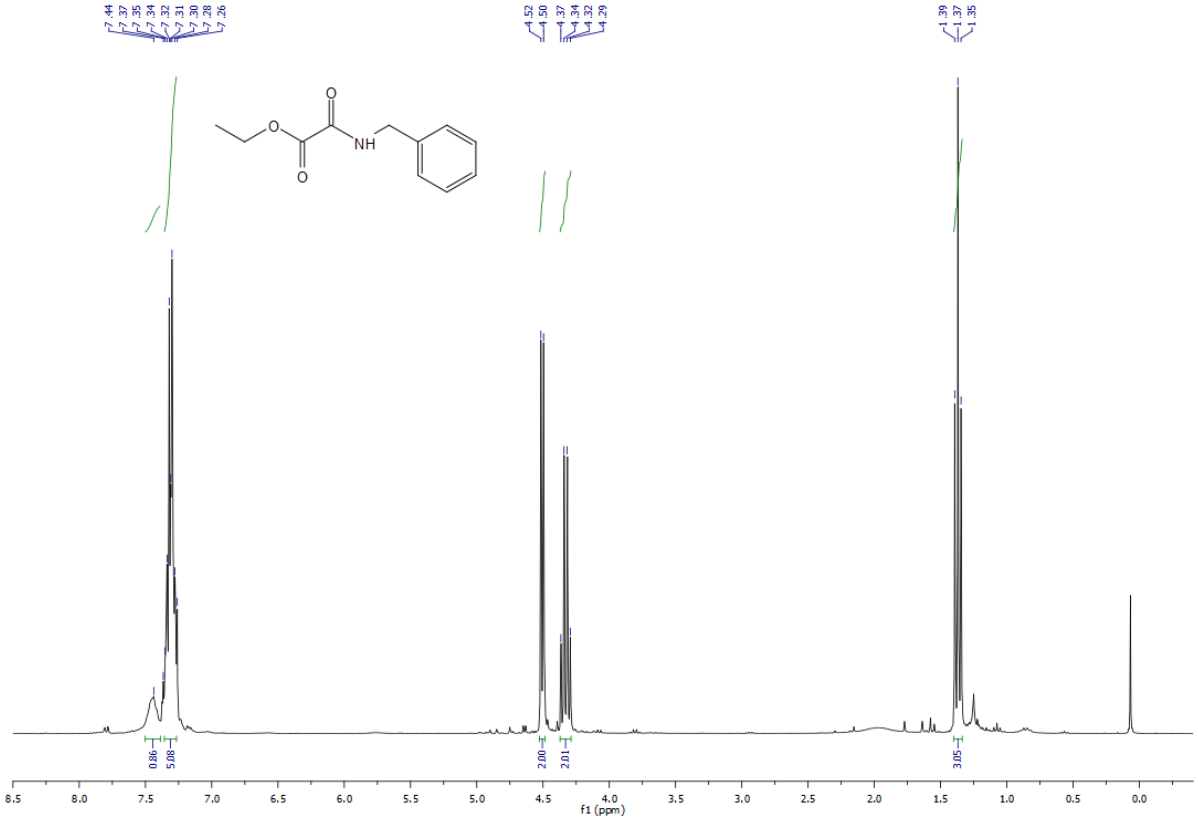

160.91
156.57
77.42
77.00
76.58
63.13
45.99
37.59
30.65
26.19
25.64
13.94
O
O
NH
O
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

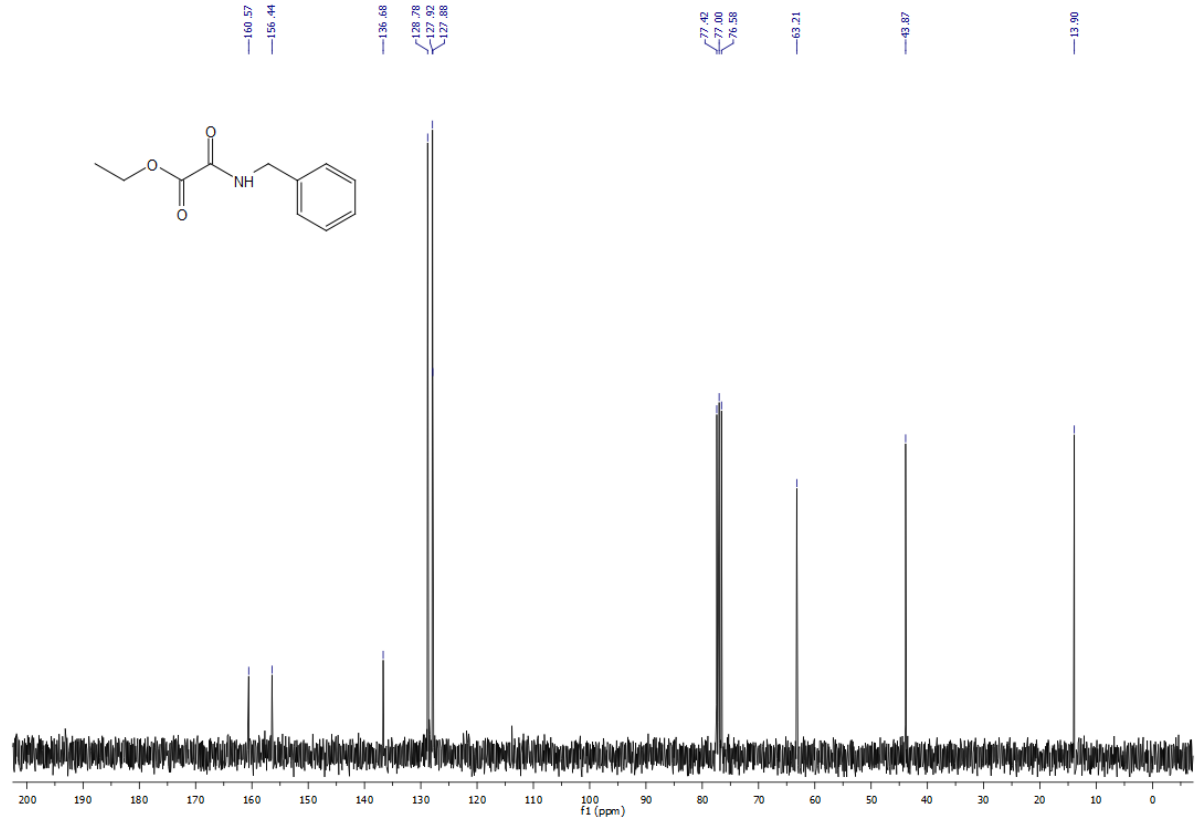

7.30
7.28
7.26
7.25
7.20
7.20
7.18
7.17
7.15
4.37
4.34
4.32
4.30
3.38
3.36
3.34
3.32
2.66
2.64
2.61
1.66
1.65
1.63
1.61
1.59
1.56
1.54
1.40
1.37
1.35
O
O
NH
O
2.19
3.82
2.00
2.00
2.06
4.17
3.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

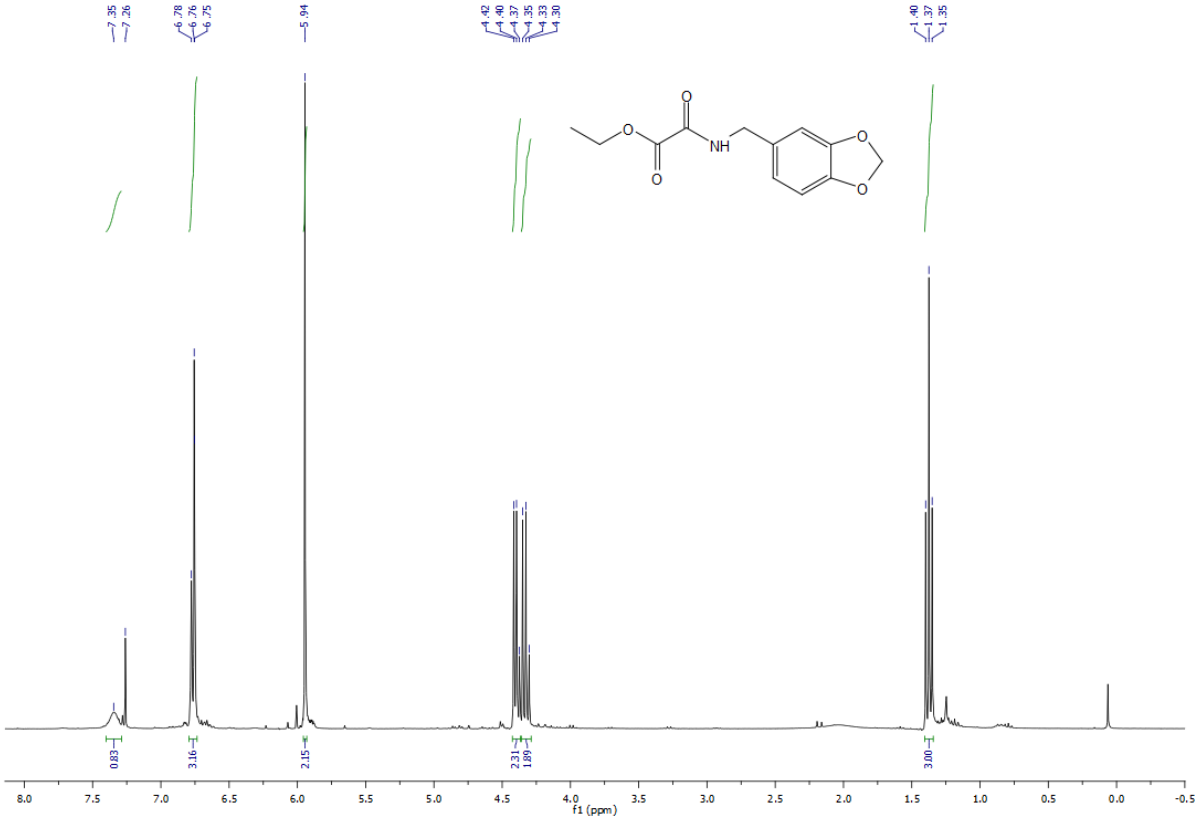

160.76
156.51
141.74
128.32
125.84
77.42
77.00
76.58
63.11
39.67
35.30
28.62
28.44
13.92
O
O
NH
O
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

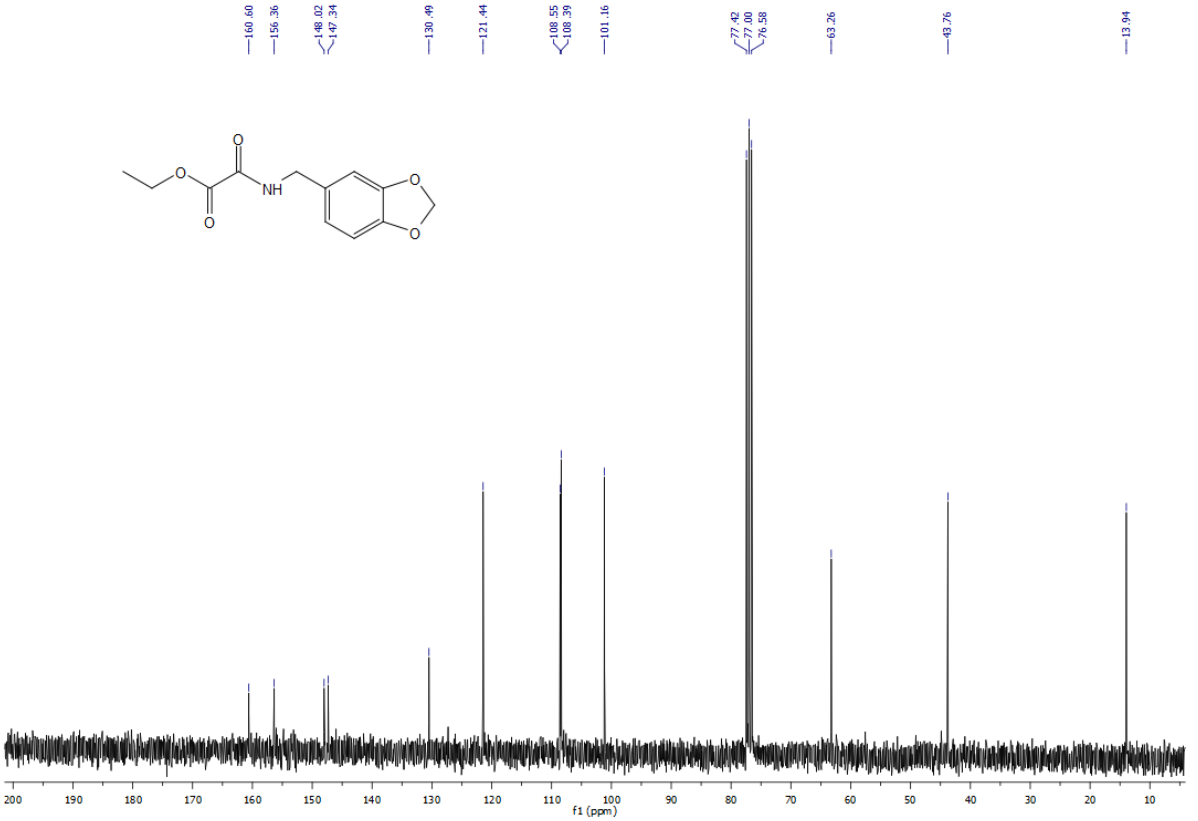

7.35
7.34
7.34
7.32
7.32
7.31
7.30
7.29
7.26
7.26
7.25
7.24
7.23
7.21
7.21
7.20
7.19
4.36
4.33
4.31
4.28
3.63
3.61
3.59
3.57
2.89
2.87
2.84
1.39
1.36
1.34
O
O
NH
O
2.14
3.02
2.00
2.06
2.09
3.02
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

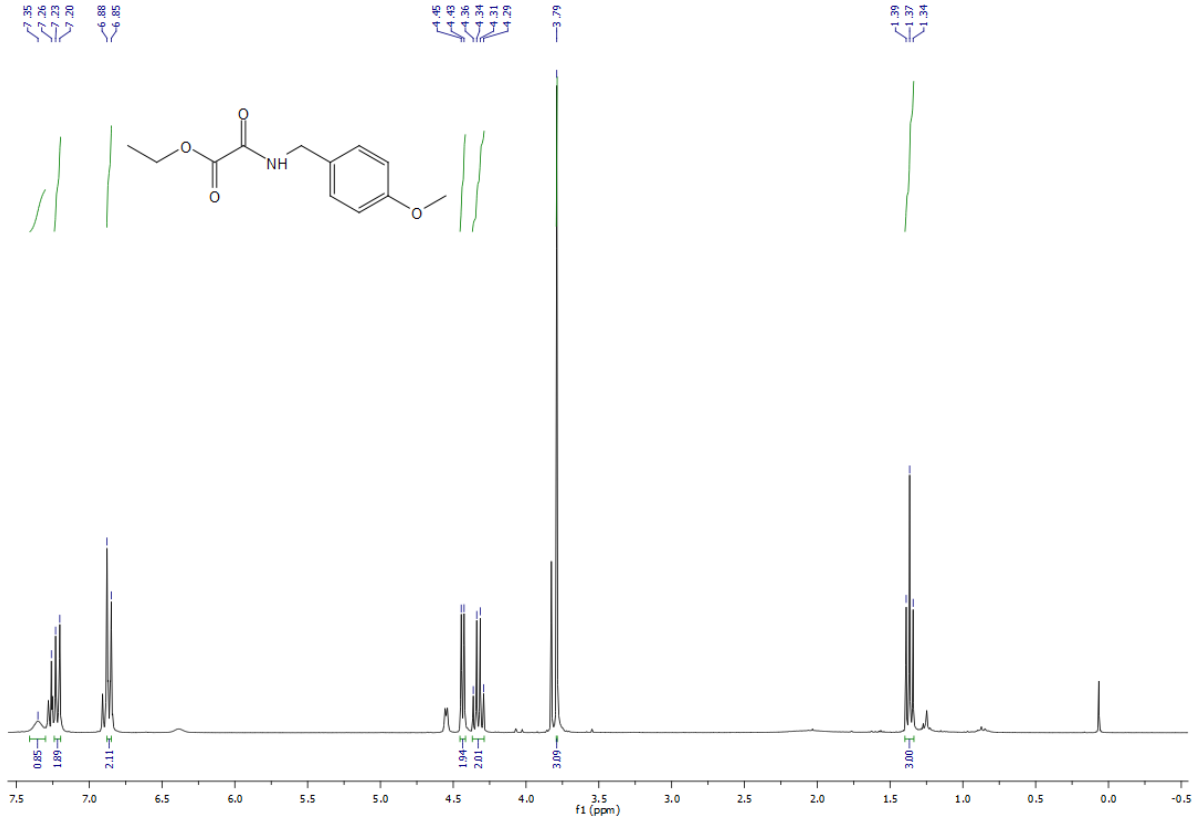

160.61
156.50
138.05
128.72
128.63
126.72
77.42
77.00
76.58
63.13
40.95
35.18
13.91
O
O
NH
O
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

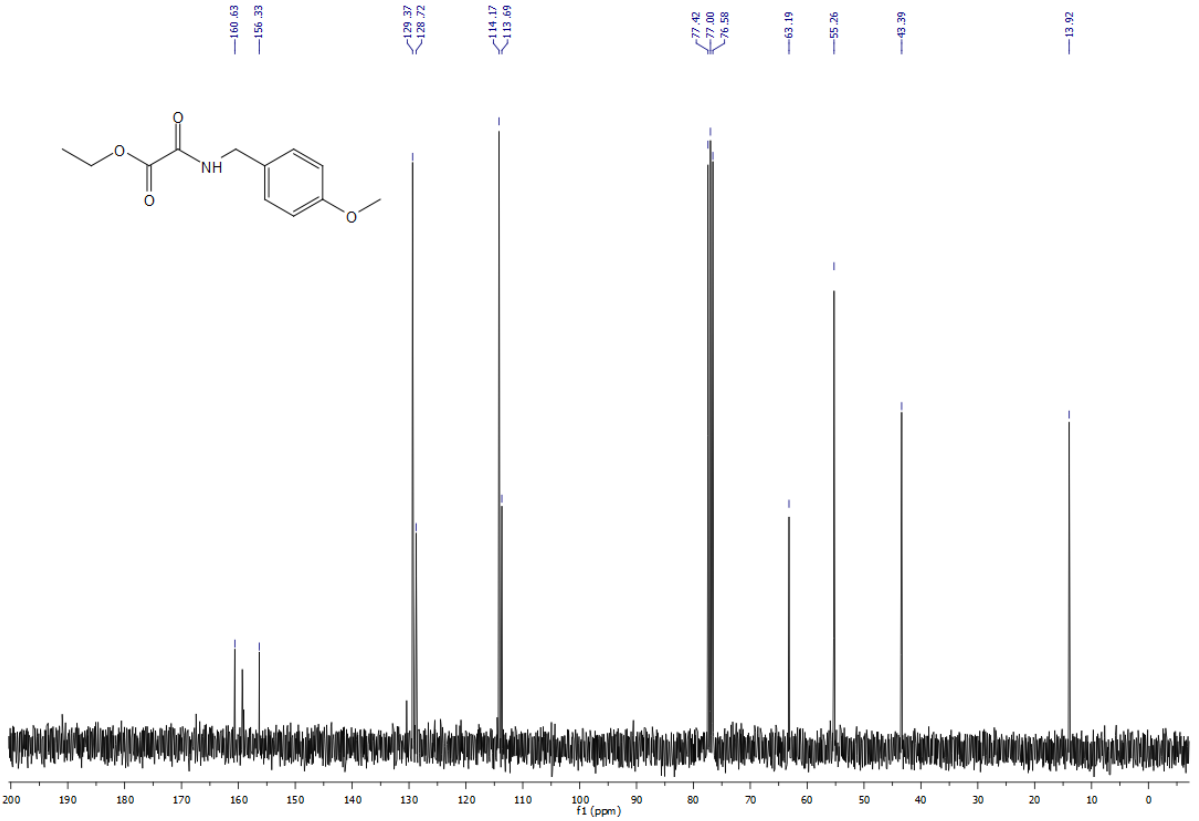

7.44
7.37
7.35
7.34
7.32
7.31
7.30
7.28
7.26
4.52
4.50
4.37
4.34
4.32
4.29
1.39
1.37
1.35
O
O
NH
O
0.86
5.08
2.00
2.01
3.05
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

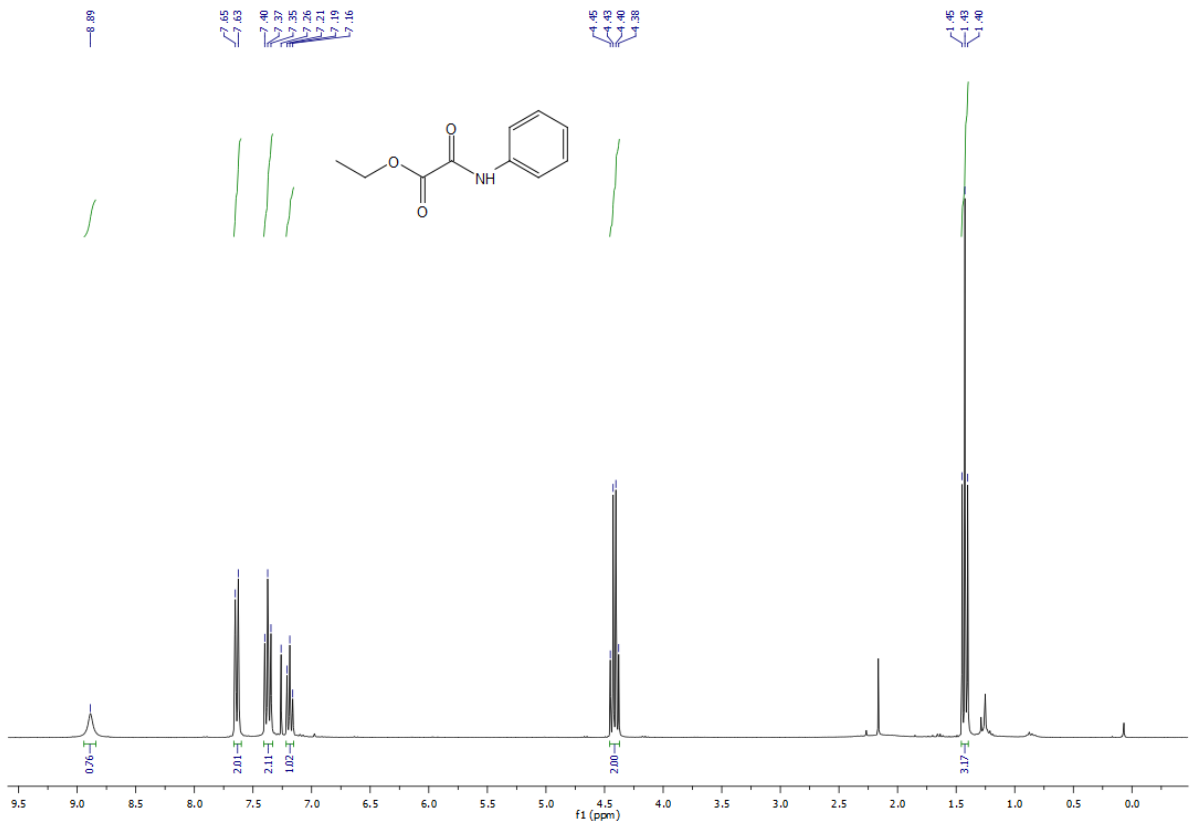

160.57
156.44
136.68
128.78
127.92
127.88
77.42
77.00
76.58
63.21
43.87
13.90
O
O
NH
O
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

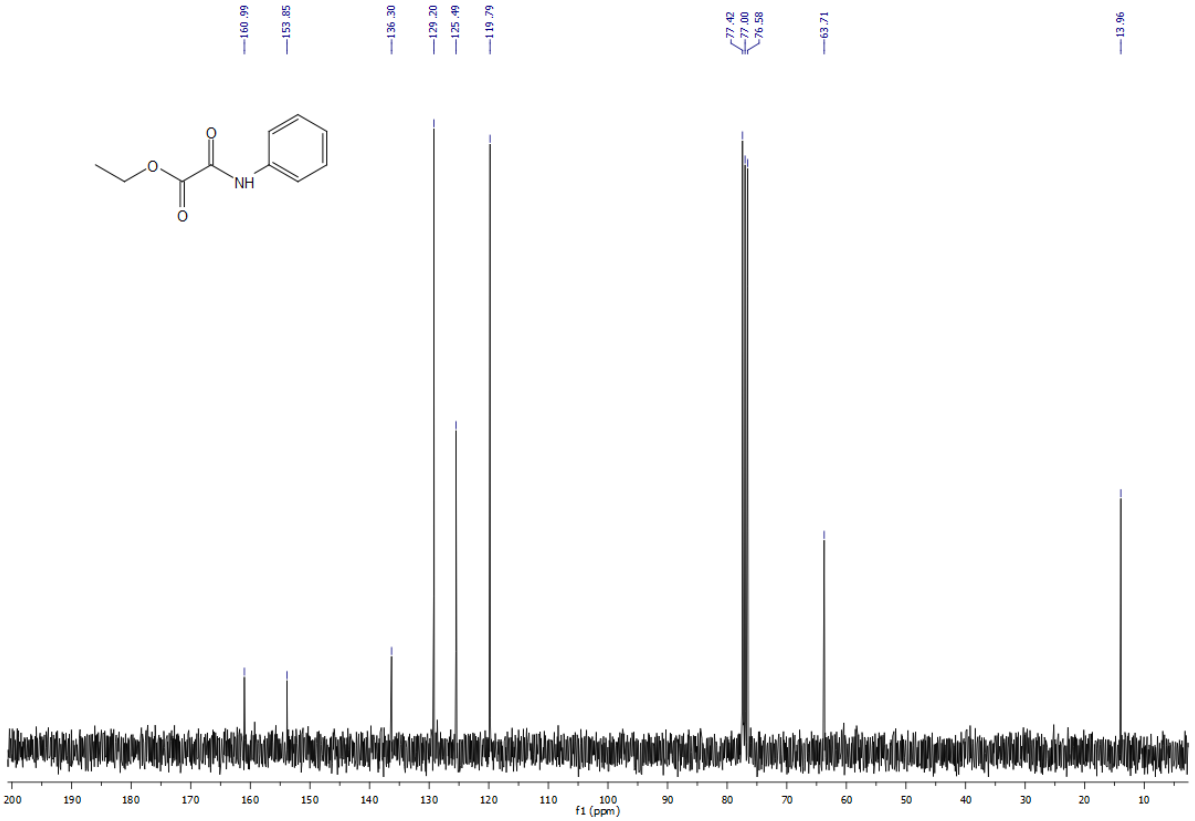

7.35
7.26
6.78
6.76
6.75
5.94
4.42
4.40
4.37
4.35
4.33
4.30
1.40
1.37
1.35
O
O
O
NH
O
O
0.83
3.16
2.15
2.31
1.89
3.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

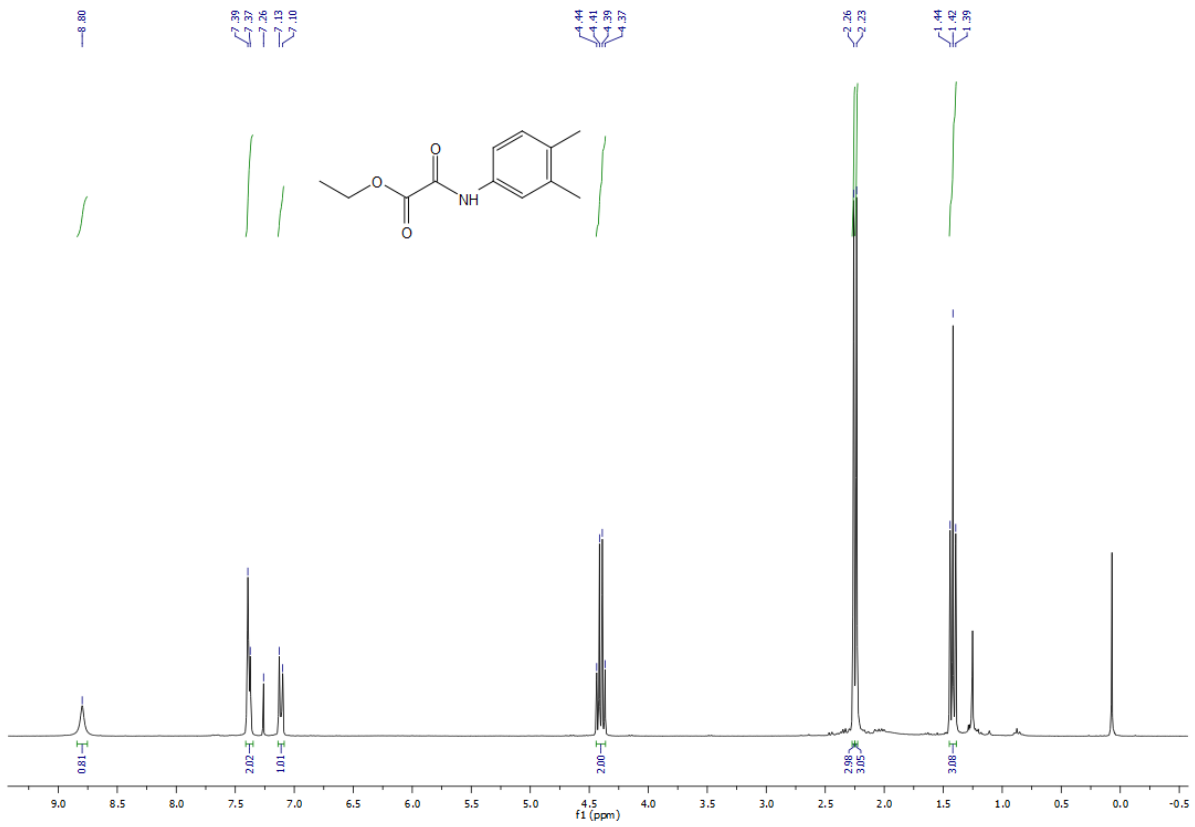

160.60
156.36
148.02
147.34
130.49
121.44
108.55
108.39
101.16
77.42
77.00
76.58
63.26
43.76
13.94
O
O
O
NH
O
O
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

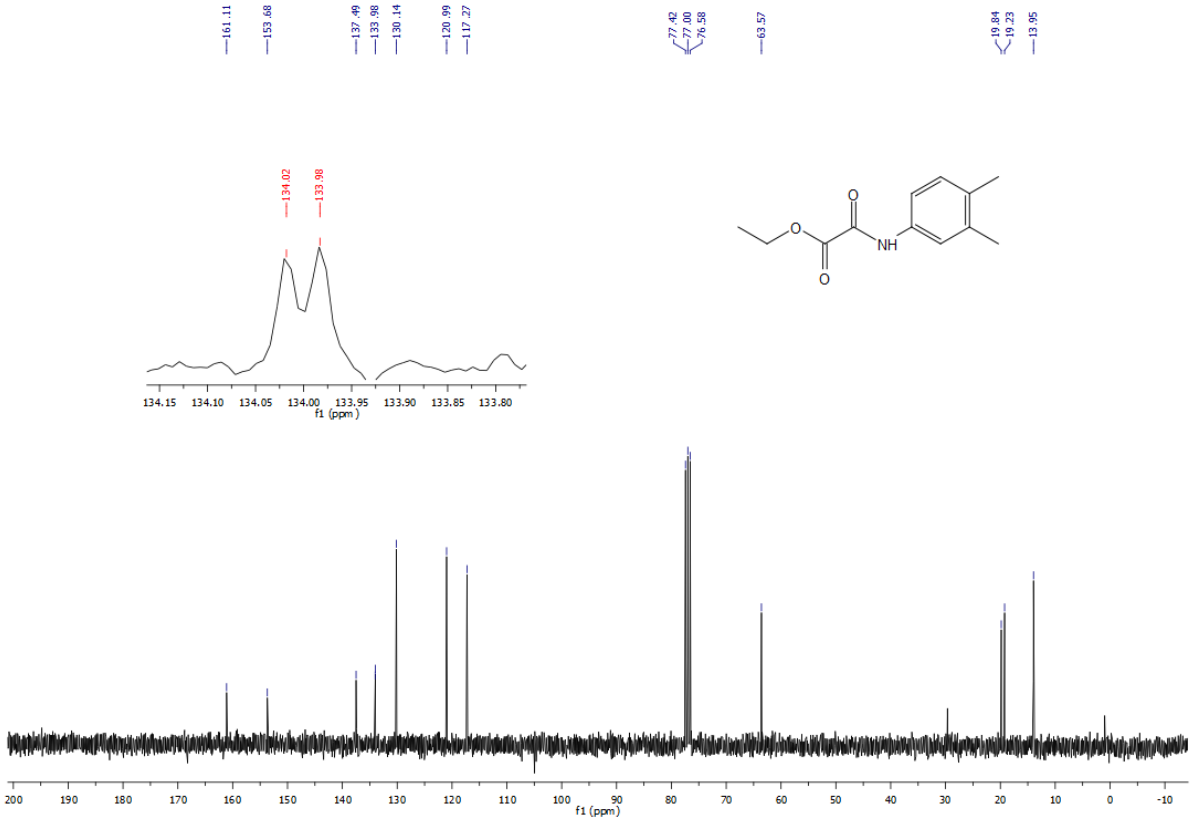

7.35
7.26
7.23
7.20
6.88
6.85
4.45
4.43
4.36
4.34
4.31
4.29
3.79
1.39
1.37
1.34
O
O
NH
O
O
0.85
1.89
2.11
1.94
2.01
3.09
3.00
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

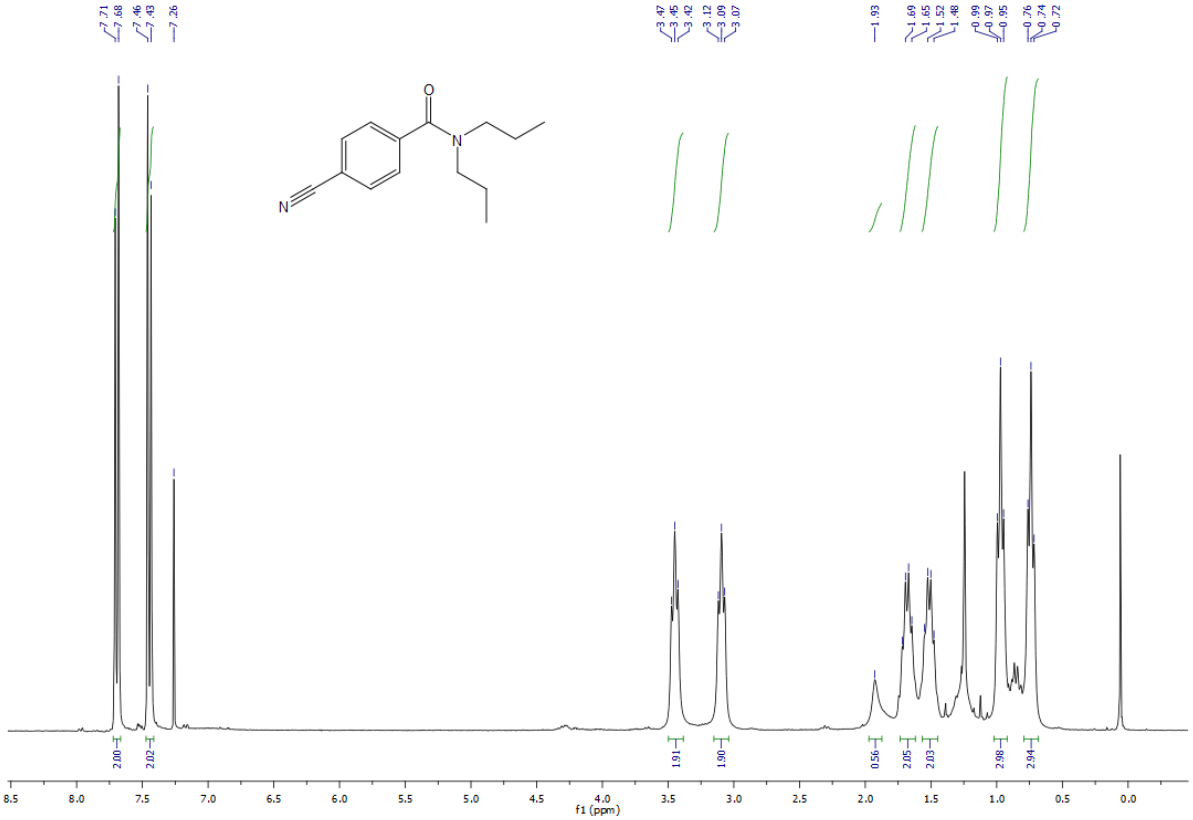

160.63
156.33
129.37
128.72
114.17
113.69
77.42
77.00
76.58
63.19
55.26
43.39
13.92
O
O
NH
O
O
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

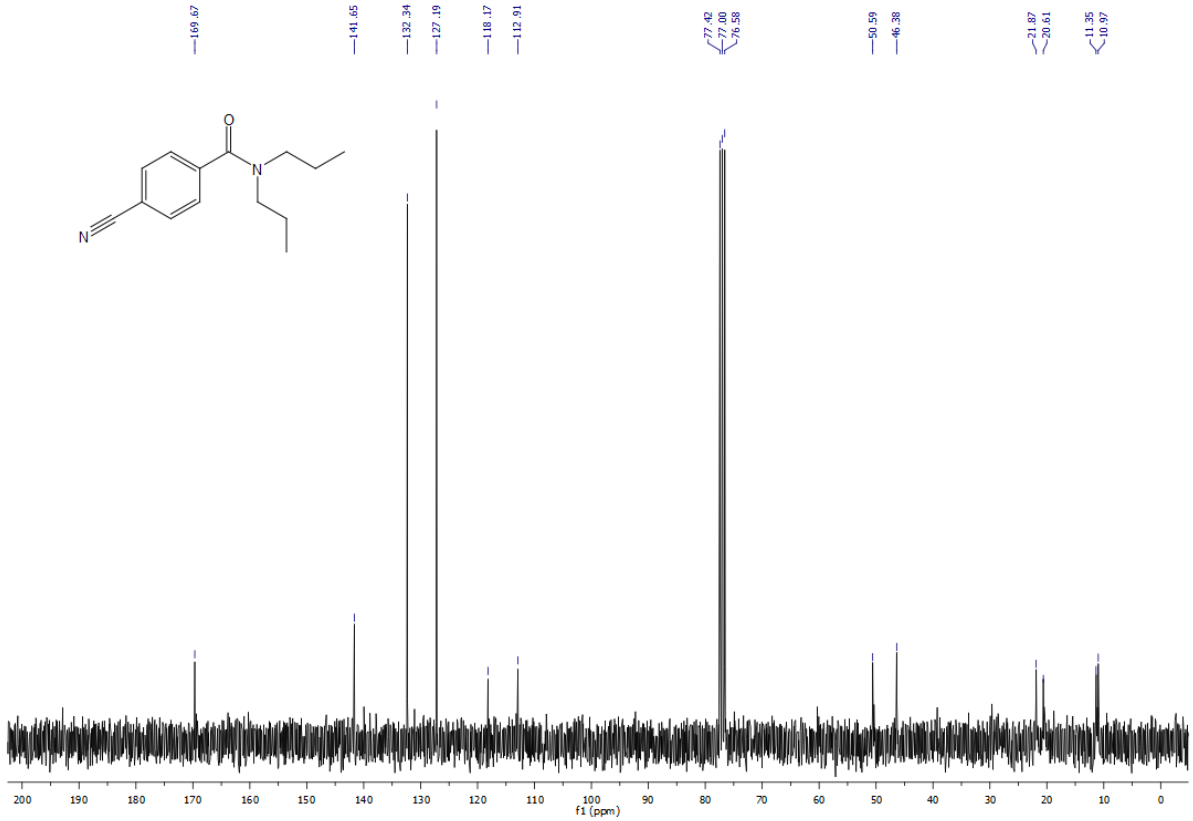


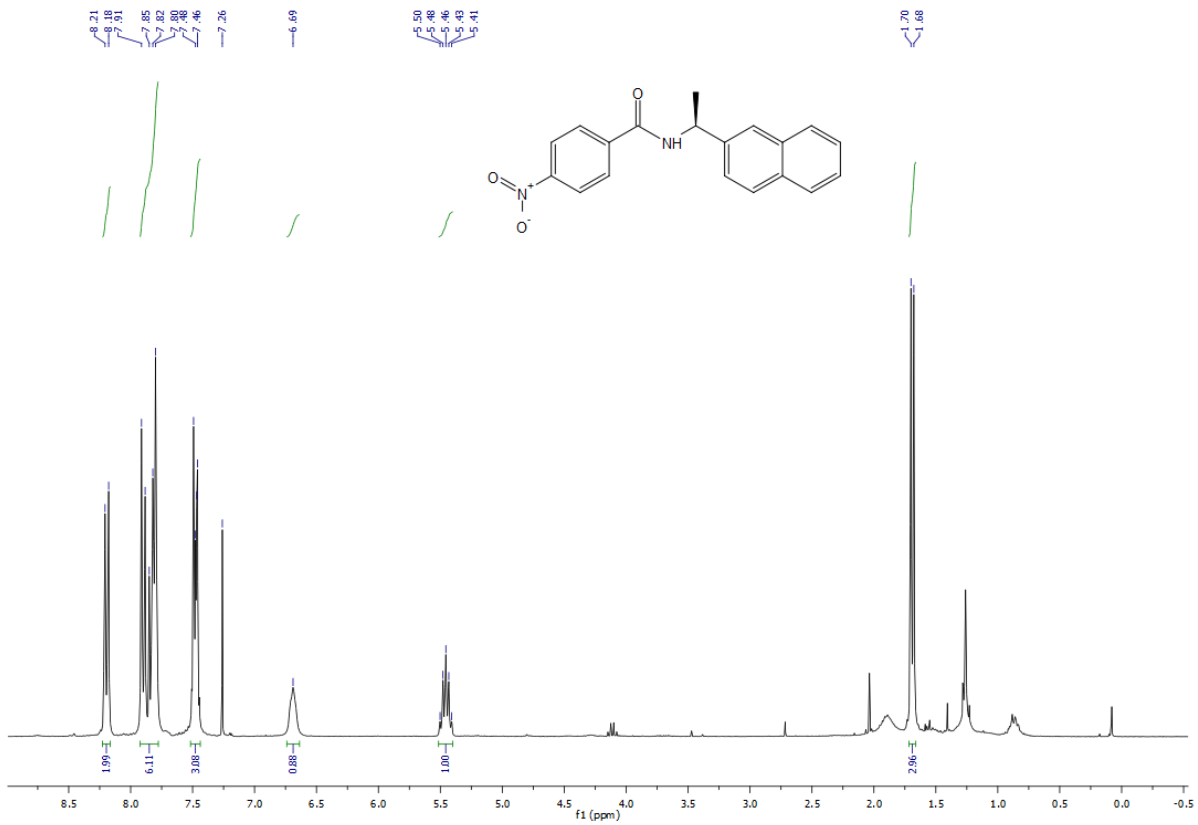


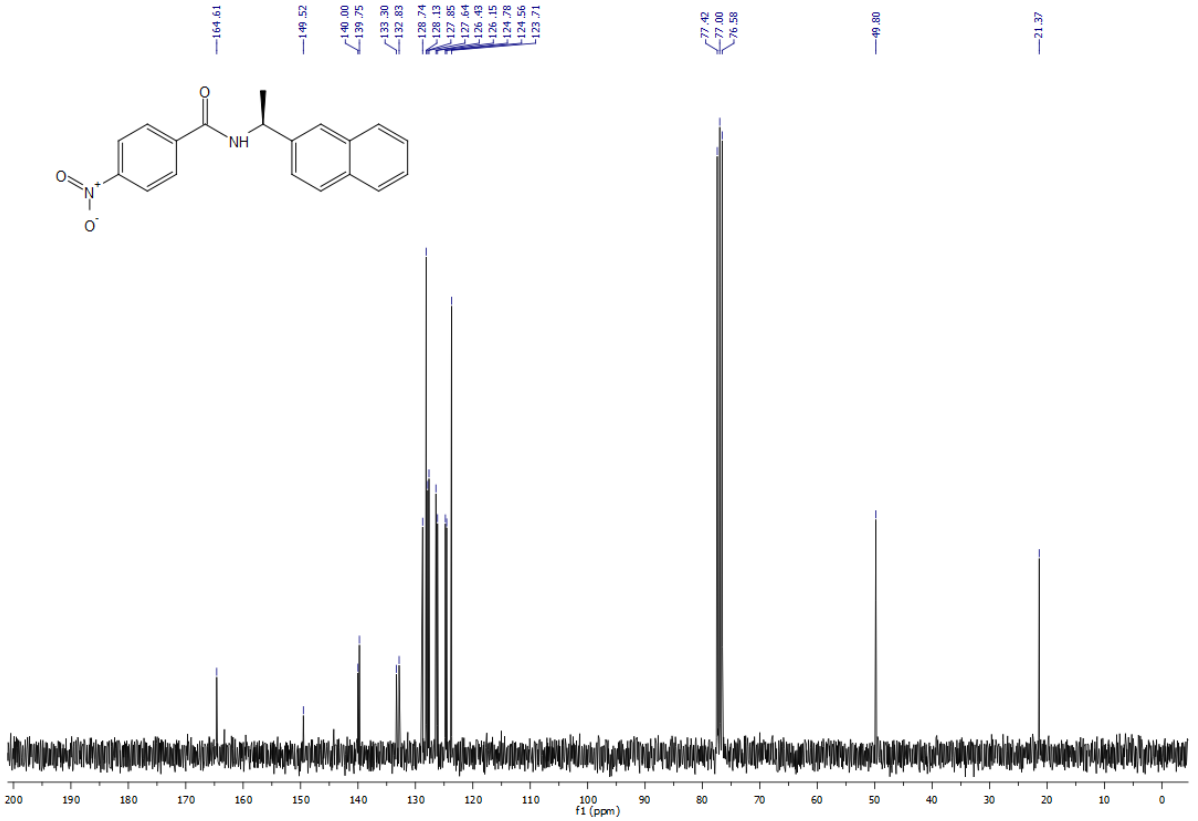


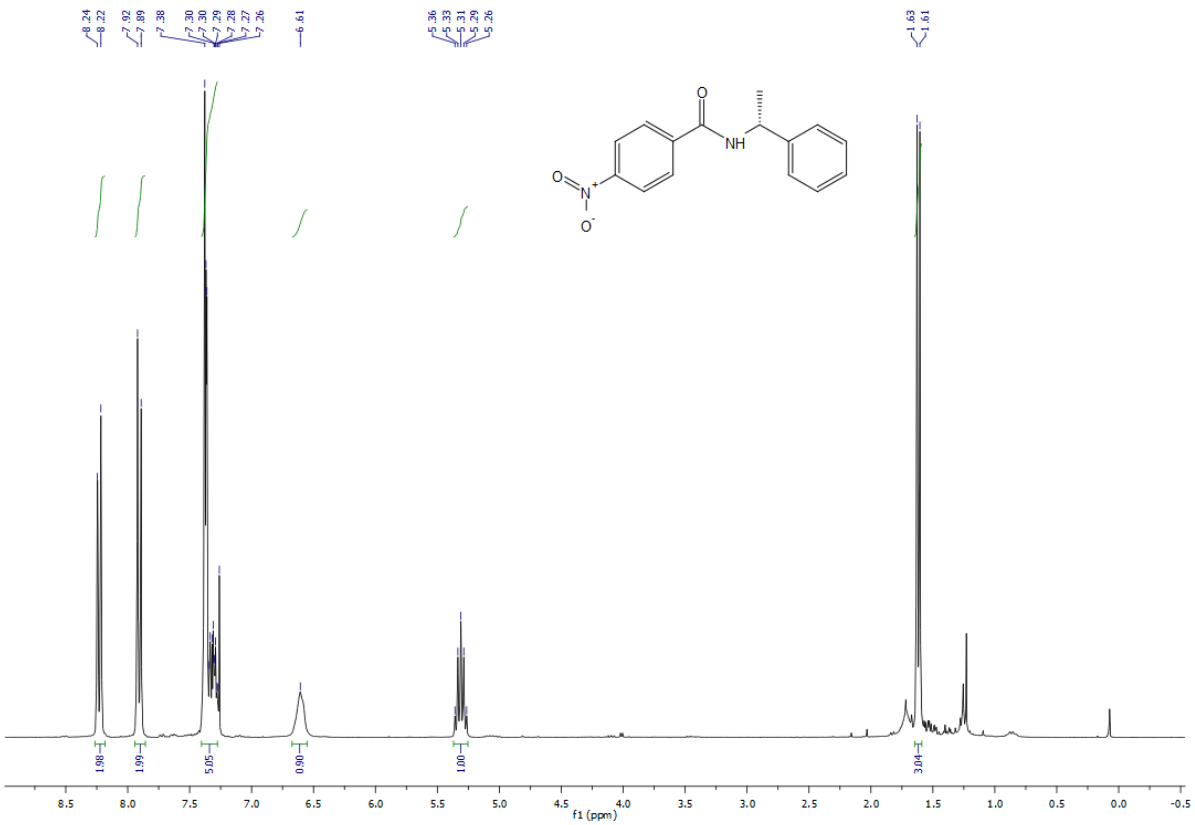


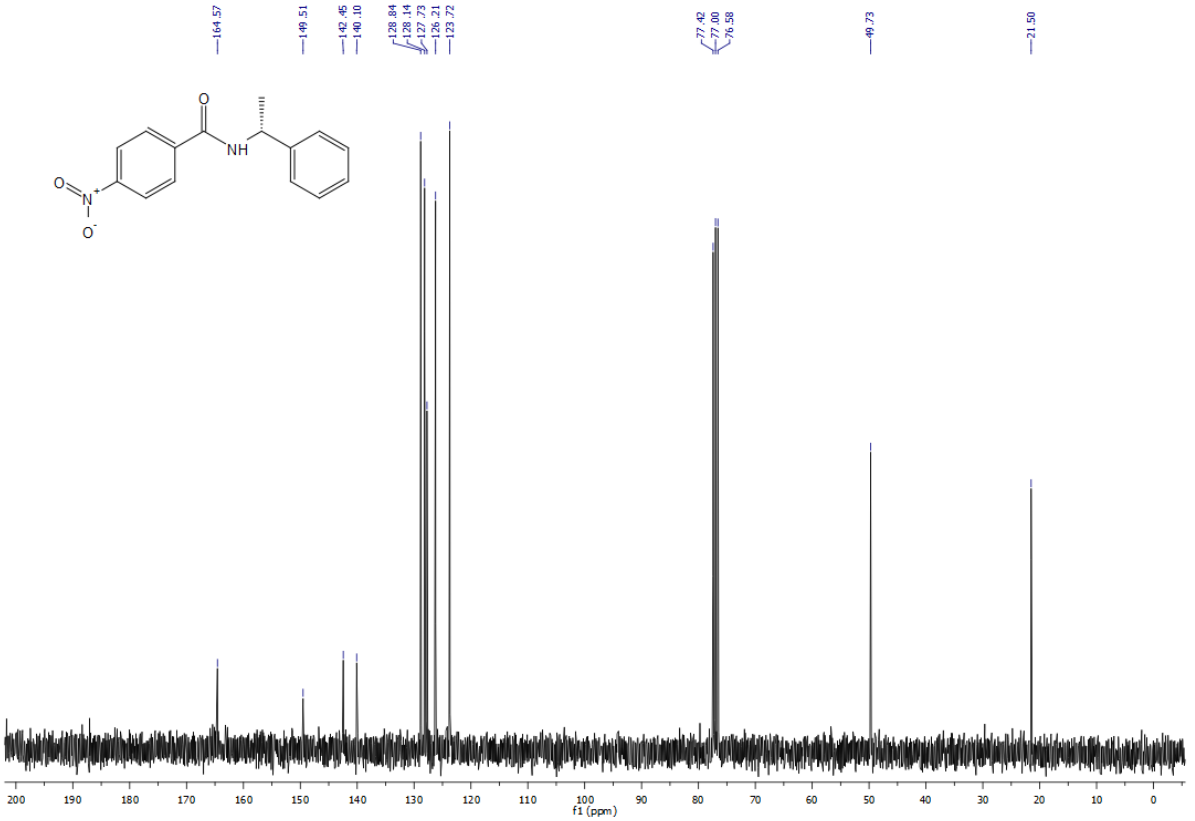

7.71
7.68
7.46
7.43
7.26
3.47
3.45
3.42
3.12
3.09
3.07
1.93
1.69
1.65
1.52
1.48
0.99
0.97
0.95
0.76
0.74
0.72
O
N
N
2.00
2.02
1.91
1.90
0.56
2.05
2.03
2.98
2.94
f1 (ppm)

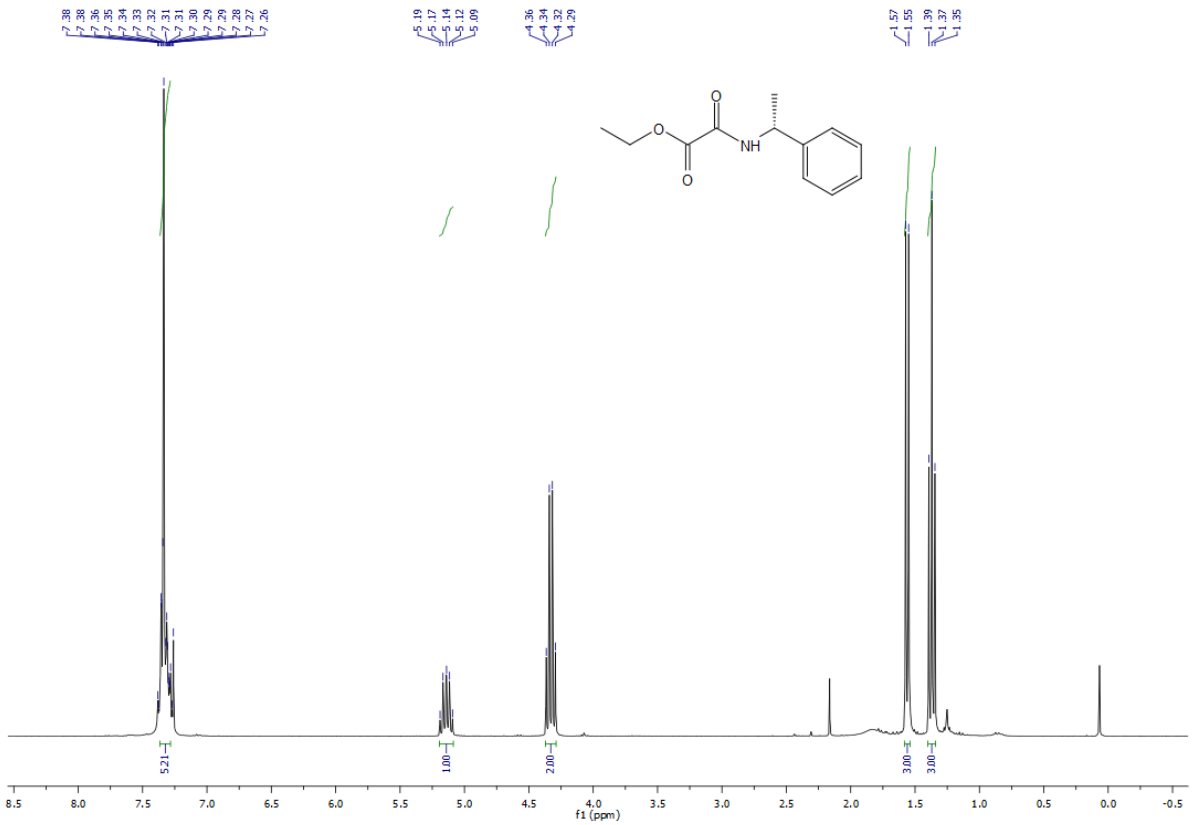

169.67
141.65
132.34
127.19
118.17
112.91
77.42
77.00
76.58
50.59
46.38
21.87
20.61
11.35
10.97
O
N
N
f1 (ppm)

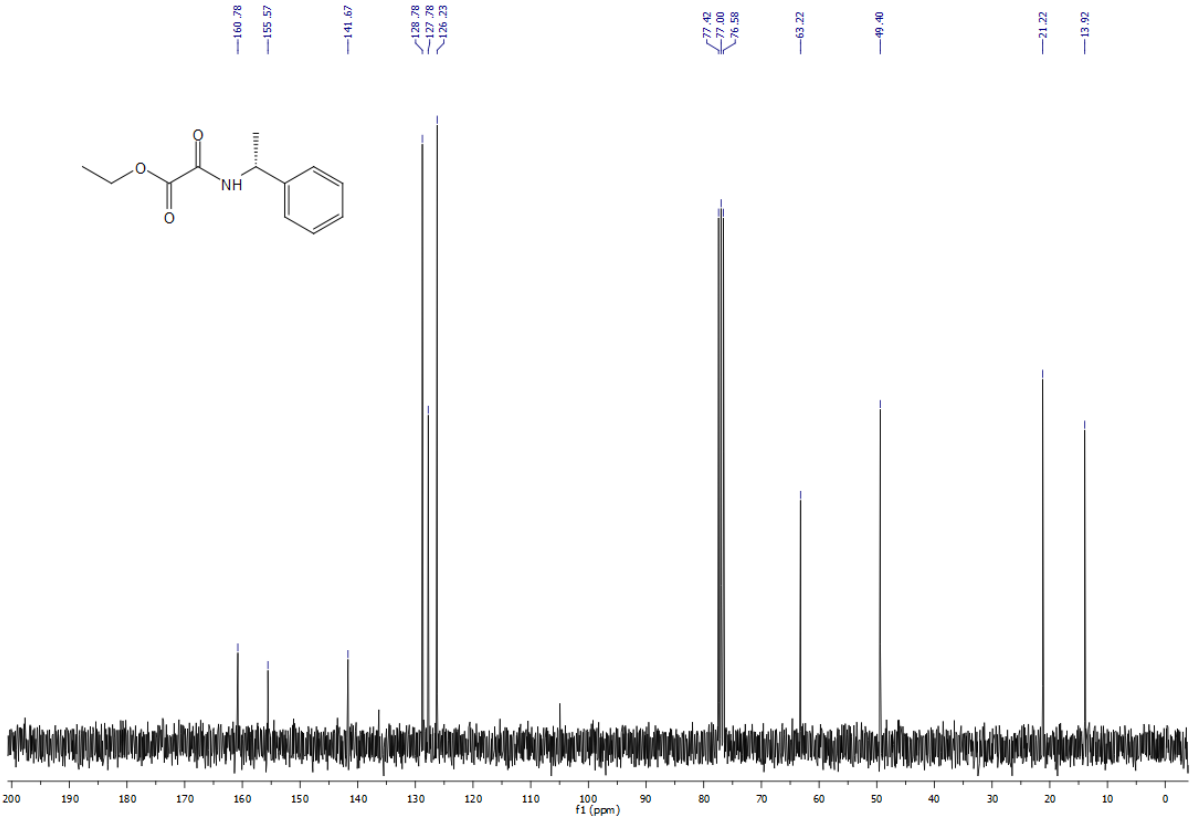





8.24
8.22
7.92
7.89
7.38
7.30
7.30
7.29
7.28
7.27
7.26
6.61
5.36
5.33
5.31
5.29
5.26
1.63
1.61
O
NH
O
N
O
1.98
1.99
5.05
0.90
1.00
3.04
f1 (ppm)

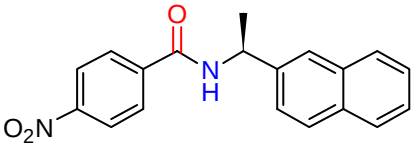

164.57
149.51
142.45
140.10
128.84
128.14
127.73
126.21
123.72
77.42
77.00
76.58
49.73
21.50
O
NH
O
N
O
f1 (ppm)



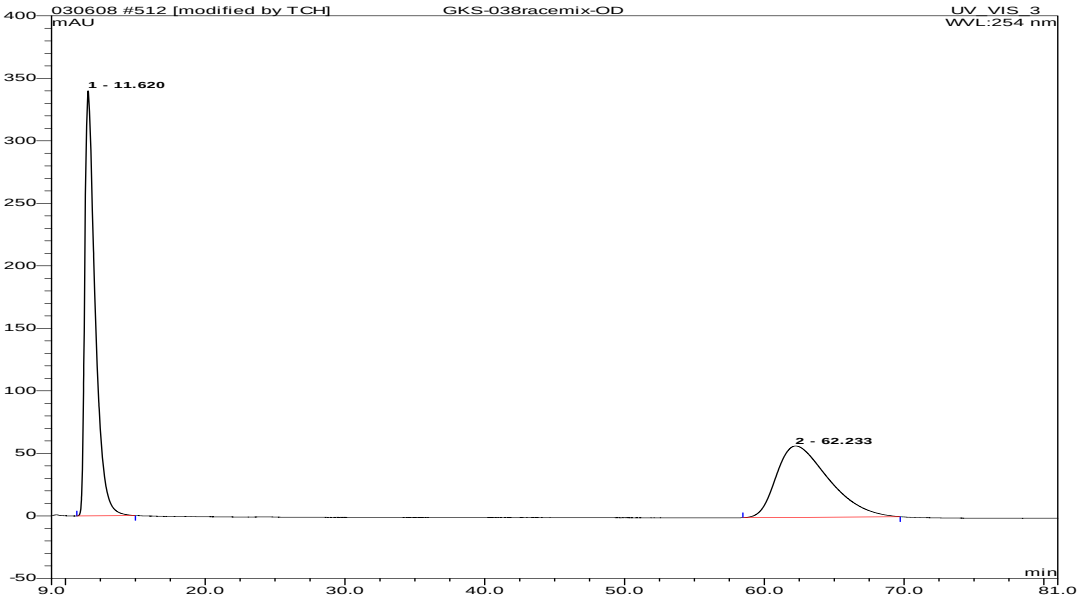


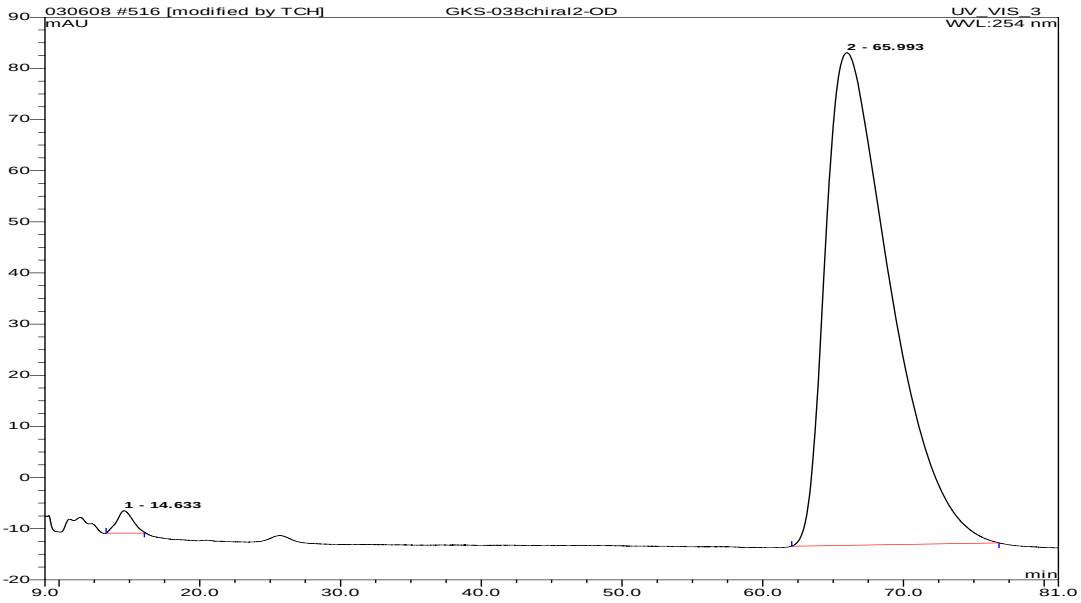
6. Chiral HPLC Chromatograms for Amides 38 - 40

Amide **38**: (S)-N-(1-(naphthalen-2-yl)ethyl)-4-nitrobenzamide



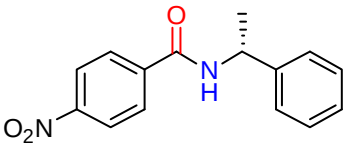
98 % *ee*



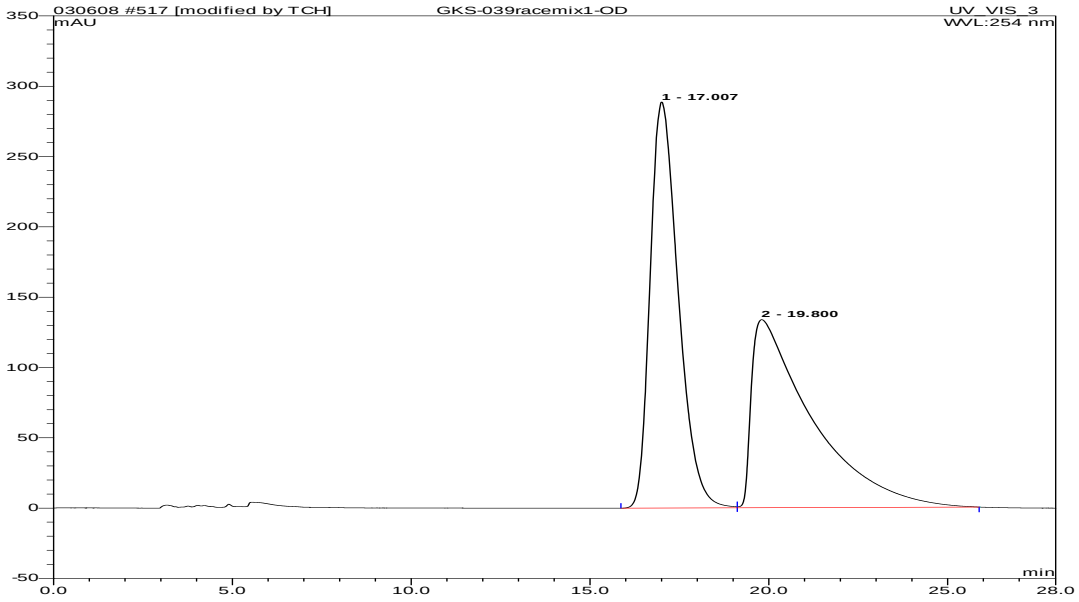


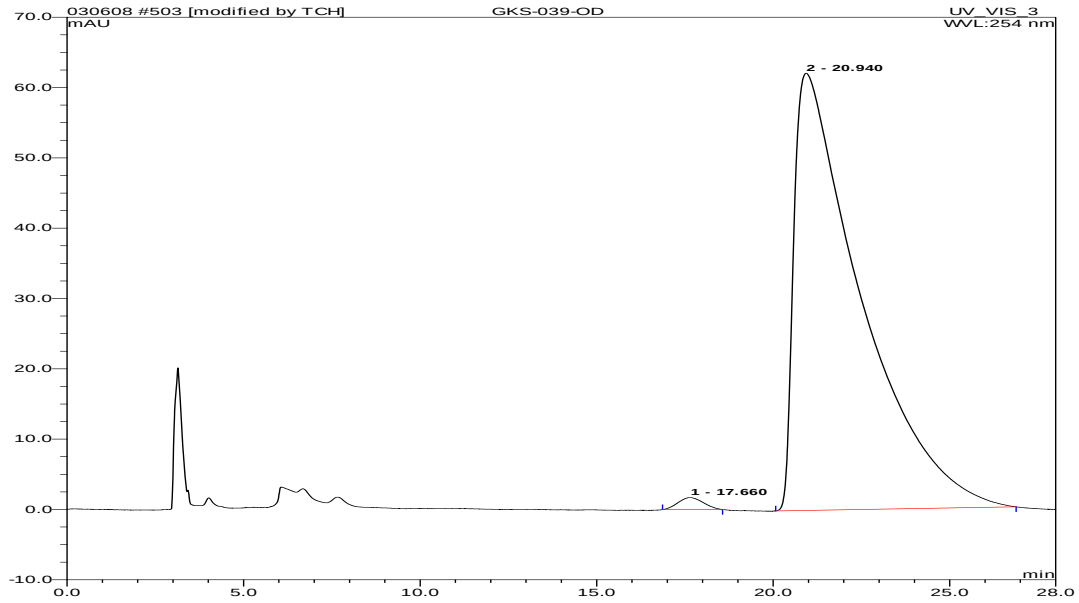
No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %
1	14.63	n.a.	4.386	5.897	1.13
2	65.99	n.a.	96.304	516.844	98.87
Total:			100.690	522.741	100.00

Amide **39**: (R)-4-nitro-N-(1-phenylethyl)benzamide



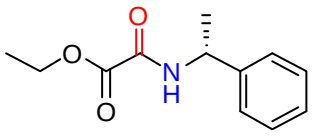
98 % *ee*



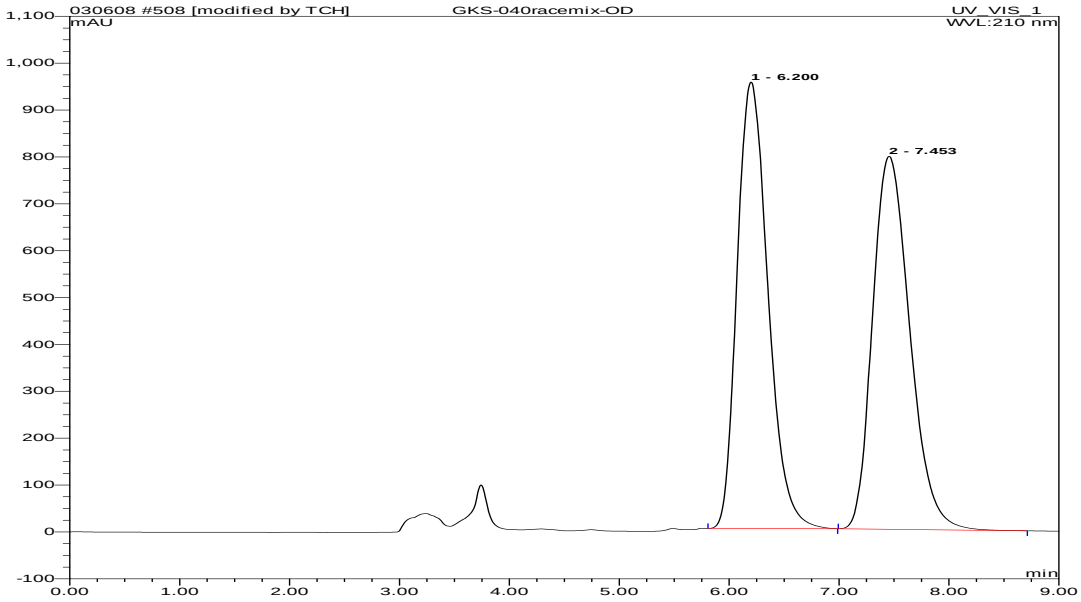


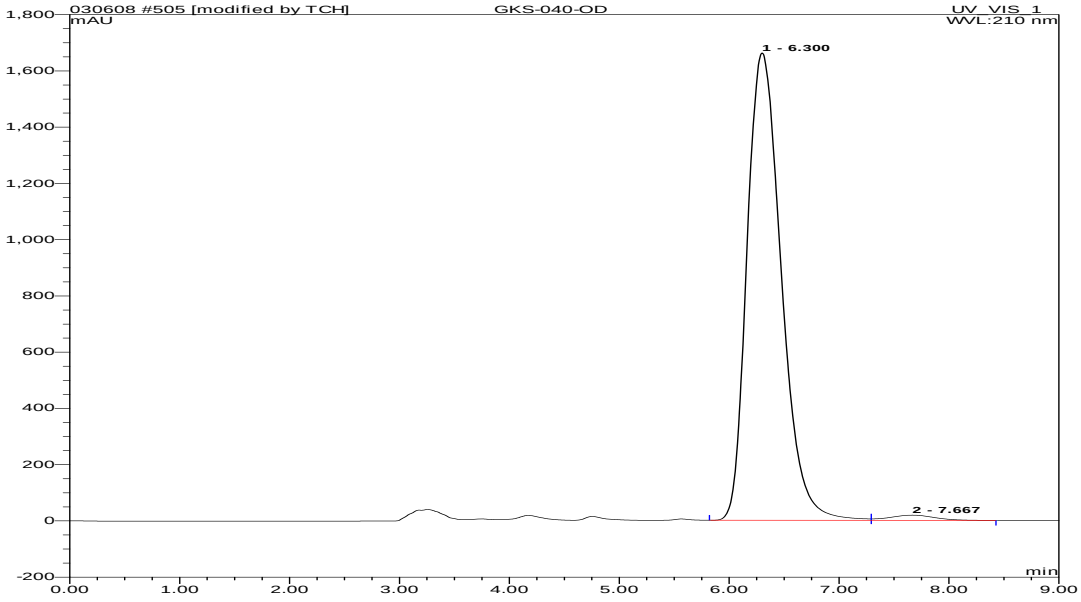
No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %
1	17.66	n.a.	1.704	1.411	1.03
2	20.94	n.a.	62.169	136.083	98.97
Total:			63.873	137.493	100.00

Amide **40**: (R)-ethyl 2-oxo-2-((1-phenylethyl)amino)acetate



97 % *ee*





No.	Ret.Time min	Peak Name	Height mAU	Area mAU*min	Rel.Area %
1	6.30	n.a.	1661.561	600.702	98.59
2	7.67	n.a.	18.418	8.604	1.41
Total:			1679.979	609.306	100.00