Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information

Visible light-induced manganese-catalyzed aminocarbonylation of alkyl iodides under atmosphere pressure at room temperature

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Contents

1. General experimental

Reagents and solvents: Unless otherwise noted, the chemicals were commercially available from Sigma-Aldrich, TCI or Alfa Aesar and were used without further purification. Dioxane bought from Alfa Aesar, HPLC grade, 99% min, packaged under argon in resealable ChemSeal bottles. The reaction does not require the glovebox.

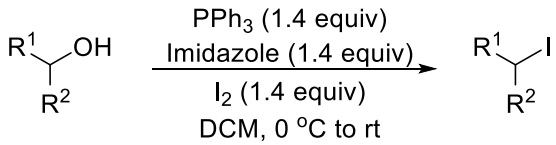
Purification: The products were isolated from the reaction mixture by column chromatography on silica gel 60, 0.063-0.2 mm, 70-230 mesh (Merck). Gradient flash chromatography was conducted eluting with PE/EA, PE refers to pentane and EA refers to ethyl acetate, they were listed as volume/volume ratios.

Irradiation: The light source was placed ca. 23 cm from the reaction vial on top of a manufactured autoclave (Figure S1). In every reaction the strong light source Portable Lumatec SUPERLITE S 04[1] was used with different set filters: blue (λmax = 460 nm).

Data collection: GC-yields were calculated using hexadecane as internal standard. GC analysis was performed on an Agilent HP-7890A instrument with FID detector and HP-5 capillary column (polydimethylsiloxane with 5% phenyl groups, 30 m, 0.32 mm i.d., 0.25 μm film thickness) using argon as carrier gas. High resolution mass spectra (HRMS) were recorded on Agilent 6210. NMR spectra were recorded on Bruker Avance 300 and Bruker ARX 400 spectrometers. Chemical shifts (ppm) are given relative to solvent: references for $CDCl_3$ were 7.26 ppm (^{1}H NMR) and 77.00 ppm (^{13}C NMR). All measurements were carried out at room temperature unless otherwise stated.

2. General procedure for the synthesis of alkyl halides.

The PPh_3 (1.4 equiv.) and imidazole (1.4 equiv.) was dissolved in dry CH_2Cl_2 (3 mL/ 1.0 mmol). Iodine was added over 5 minutes at 0 ℃ and alcohol (1.0 equiv.) was added dropwise over 5 minutes. Then the reaction was stirred for additional 5-12 hours at room temperature. A solution of Na_2SO_3 in water was added and stirred for 10 minutes, The aqueous layer was separated and extracted with DCM. The combine organic layers were washed with brine, dried over Na_2SO_4, filtered, and concentrated under reduced pressure. The resulting iodide was purified by flash chromatography.[2]



3. General Procedure for the carbonylation.

3.1 General procedure: the carbonylation of alkyl iodides.

A 4 mL snap vial was charged with $Mn_2(CO)_{10}$ (0.01 mmol, 5 mol %), K_3PO_4 (0.3 mmol, 1.5 equiv.) and closed with a rubber-based septum. The vial was evacuated and backfilled with argon. Degassed dioxane (0.5 mL), aniline (0.2 mmol, 1 equiv.) and alkyl iodides (0.3 mmol, 1.5 equiv.) were added via syringe. The vial was then connected to atmosphere with a cannula and transferred into a 300 mL photoautoclav, manufactured by Parr instrument company®, under argon counterflow. As depicted, the vials were placed in a revolver-like manner and without an alloy plate, 9 vials did exactly fit (Figure S1). The closed autoclave was flushed three times with nitrogen (~ 5 bar), three times with CO (~ 5 bar), and 1 bar of carbon monoxide (measured by pressure meter) was charged. The autoclave was then placed into an aluminum block on a magnetic stirrer. The reaction mixture was stirred (500

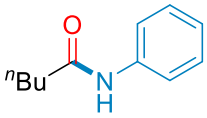
rpm) at 25 °C while being irradiated with light at the chosen wavelength (460 nm – blue) from a portable Lumatec SUPERLITE S 04[1] for the specified time without additional cooling. After irradiation, the light was turned off and cooling to room temperature. The crude product was purified by silica gel chromatography (pentane/EA) to afford the corresponding product.

3.2 Reaction Setup

Up to 9 reactions can be carried out simultaneously. Irradiation is partly indirect by reflection of the autoclave bottom:

Figure S1. Typical setup for the irradiation of nine samples. The top of the autoclave has a window made of quartz glass that allows radiation to reach the center. The arrangement of the reaction bottles inside the autoclave results in an even distribution of light, which is irradiated by the SUPERLITE S04 for consistent reflection.

4. Characterization and procedure of the products



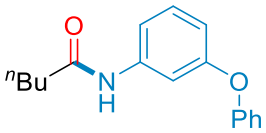
3

***N*-phenylpentanamide (3) [3]:**

White solid (29.5 mg, 83% yield), purified by column chromatography (SiO_2, Pentane/EA= 15:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.51 (d, *J* = 7.6 Hz, 2H), 7.31 (t, *J* = 7.8 Hz, 2H), 7.09 (t, *J* = 7.4 Hz, 1H), 2.36 (t, *J* = 7.6 Hz, 2H), 1.71 (p, *J* = 7.5 Hz, 2H), 1.40 (h, *J* = 7.6 Hz, 2H), 0.94 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.62, 137.95, 128.91, 124.15, 119.86, 37.45, 27.69, 22.34, 13.76.



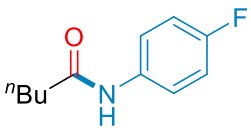
4

***N*-(3-phenoxyphenyl) pentanamide (4) [3]:**

White solid (53.5 mg, 99% yield), purified by column chromatography (SiO_2, Pentane/EA= 10:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.48 (s, 1H), 7.27 – 7.19 (m, 2H), 7.18 – 7.11 (m, 3H), 7.04 – 6.97 (m, 1H), 6.95 – 6.88 (m, 2H), 6.67 – 6.61 (m, 1H), 2.28 – 2.17 (m, 2H), 1.66 – 1.50 (m, 2H), 1.36 – 1.21 (m, 2H), 0.83 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.69, 157.76, 156.83, 139.35, 129.91, 129.70, 123.39, 119.04, 114.51, 114.28, 110.32, 37.39, 27.58, 22.30, 13.74.



5

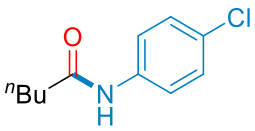
***N*-(4-fluorophenyl) pentanamide (5) [3]:**

White solid (31.6 mg, 83% yield), purified by column chromatography (SiO_2, Pentane/EA= 20:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.65 (s, 1H), 7.52 – 7.39 (m, 2H), 7.03 – 6.91 (m, 2H), 2.35 (d, *J* = 7.4 Hz, 2H), 1.76 – 1.60 (m, 2H), 1.47 – 1.31 (m, 2H), 0.92 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.76, 159.28 (d, *J* = 243.2 Hz), 133.93, 121.81 (d, *J* = 7.8 Hz), 115.48 (d, *J* = 22.5 Hz), 37.22, 27.68, 22.33, 13.74.

^{19}F NMR (282 MHz, $CDCl_3$) δ -118.04 – -118.28 (m).



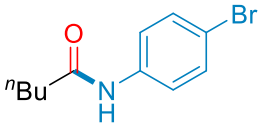
6

***N*-(4-chlorophenyl) pentanamide (6)** [3]**:**

White solid (39.0 mg, 92% yield), purified by column chromatography (SiO_2, Pentane/EA= 10:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.73 (s, 1H), 7.46 (d, *J* = 8.8 Hz, 2H), 7.23 (d, *J* = 8.8 Hz, 2H), 2.34 (t, 2H), 1.76 – 1.60 (m, 2H), 1.44 – 1.30 (m, 2H), 0.92 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.86, 136.51, 129.11, 128.86, 121.21, 37.32, 27.61, 22.32, 13.73.



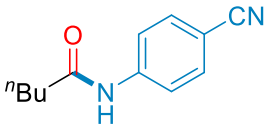
7

***N*-(4-bromophenyl) pentanamide (7)** [3]**:**

White solid (37.0 mg, 72% yield), purified by column chromatography (SiO_2, Pentane/EA= 10:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.64 (s, 1H), 7.39 – 7.26 (m, 4H), 2.26 (t, 2H), 1.69 – 1.52 (m, 2H), 1.38 – 1.22 (m, 2H), 0.85 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.84, 137.01, 131.81, 121.52, 116.70, 37.34, 27.59, 22.31, 13.73.



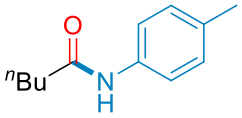
8

***N*-(4-cyanophenyl) pentanamide (8)** [3]**:**

White solid (35.7 mg, 88% yield), purified by column chromatography (SiO_2, Pentane/EA= 5:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.92 (s, 1H), 7.72 – 7.66 (m, 2H), 7.60 – 7.55 (m, 2H), 2.45 – 2.34 (m, 2H), 1.78 – 1.62 (m, 2H), 1.45 – 1.31 (m, 2H), 0.92 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 172.15, 142.29, 133.17, 119.46, 118.93, 106.55, 37.42, 27.40, 22.26, 13.70.



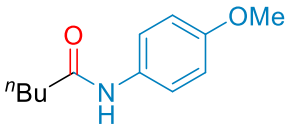
9

***N*-(*p*-tolyl) pentanamide (9) [4]:**

White solid (27.2 mg, 71% yield), purified by column chromatography (SiO_2, Pentane/EA= 10:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.54 (s, 1H), 7.39 (d, *J* = 8.1 Hz, 2H), 7.09 (d, *J* = 8.0 Hz, 2H), 2.34 (t, 2H), 2.30 (s, 3H), 1.69 (p, *J* = 7.4 Hz, 2H), 1.38 (h, *J* = 7.3 Hz, 2H), 0.93 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.63, 135.34, 133.77, 129.36, 120.02, 37.33, 27.74, 22.34, 20.80, 13.77.



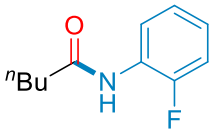
10

***N*-(4-methoxyphenyl) pentanamide (10) [3]:**

White solid (24.0 mg, 57% yield), purified by column chromatography (SiO_2, Pentane/EA= 20:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.56 (s, 1H), 7.45 – 7.34 (m, 2H), 6.88 – 6.76 (m, 2H), 3.77 (s, 3H), 2.38 – 2.27 (m, 2H), 1.76 – 1.60 (m, 2H), 1.47 – 1.30 (m, 2H), 0.92 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.60, 156.31, 131.05, 121.87, 114.02, 55.42, 37.18, 27.78, 22.35, 13.76.



11

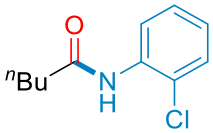
***N*-(2-fluorophenyl) pentanamide (11) [3]:**

Colorless oil (22.8 mg, 60% yield), purified by column chromatography (SiO_2, Pentane/EA= 20:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.32 (t, *J* = 8.0 Hz, 1H), 7.36 (s, 1H), 7.15 – 6.98 (m, 3H), 2.41 (t, *J* = 7.6 Hz, 2H), 1.79 – 1.66 (m, 2H), 1.48 – 1.34 (m, 2H), 0.95 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.43, 152.25 (d, *J* = 242.7 Hz), 126.39 (d, *J* = 10.1 Hz), 124.53 (d, *J* = 3.6 Hz), 124.07 (d, *J* = 7.7 Hz), 121.71, 114.66 (d, *J* = 19.2 Hz), 37.51, 27.54, 22.30, 13.75.

^{19}F NMR (282 MHz, $CDCl_3$) δ -131.60.



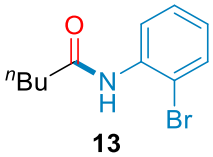
12

***N*-(2-chlorophenyl)pentanamide (12) [5]:**

White solid (29.1 mg, 71% yield), purified by column chromatography (SiO_2, Pentane/EA= 10:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.31 (d, *J* = 8.3 Hz, 1H), 7.56 (s, 1H), 7.28 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.22 – 7.15 (m, 1H), 6.95 (td, *J* = 7.7, 1.6 Hz, 1H), 2.36 (t, 2H), 1.74 – 1.58 (m, 2H), 1.43 – 1.28 (m, 2H), 0.89 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.34, 134.62, 128.89, 127.69, 124.43, 122.50, 121.58, 37.68, 27.55, 22.28, 13.75.



13

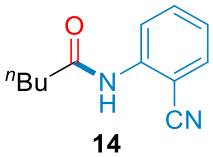
***N*-(2-bromophenyl) pentanamide (13):**

White solid (44.6 mg, 86% yield), purified by column chromatography (SiO_2, Pentane/EA= 20:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.35 (d, *J* = 8.3 Hz, 1H), 7.62 (s, 1H), 7.52 (dd, *J* = 8.0, 1.5 Hz, 1H), 7.34 – 7.26 (m, 1H), 6.96 (td, *J* = 7.9, 1.6 Hz, 1H), 2.43 (t, 2H), 1.79 – 1.68 (m, 2H), 1.43 (dq, *J* = 14.5, 7.3 Hz, 2H), 0.96 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.33, 135.71, 132.13, 128.35, 124.99, 121.88, 37.71, 27.57, 22.28, 13.76.

HRMS (EI) calcd for $C_{11}H_{14}BrNO^+$ $[M]^+$: 255.02533, found: 255.02571.



14

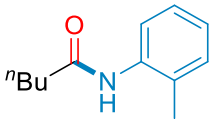
***N*-(2-cyanophenyl) pentanamide (14):**

White solid (23.3 mg, 52% yield), purified by column chromatography (SiO_2, Pentane/EA= 10:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.41 (dd, *J* = 9.0, 1.1 Hz, 1H), 7.65 (s, 1H), 7.61 – 7.54 (m, 2H), 7.16 (td, *J* = 7.6, 1.0 Hz, 1H), 2.49 – 2.42 (m, 2H), 1.79 – 1.67 (m, 2H), 1.50 – 1.38 (m, 2H), 0.96 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.71, 140.54, 134.18, 132.15, 123.96, 121.22, 116.40, 101.70, 37.50, 27.38, 22.25, 13.73.

HRMS (ESI) calcd for $C_{12}H_{15}N_2O$ for $[M+H]^+$: 203.1179, found: 203.1182.



15

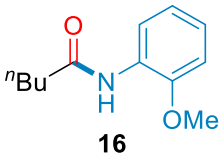
***N*-(*o*-tolyl) pentanamide (15):**

White solid (31.6 mg, 77% yield), purified by column chromatography (SiO_2, Pentane/EA= 10:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.72 (d, *J* = 7.9 Hz, 1H), 7.25 – 7.12 (m, 3H), 7.06 (t, *J* = 7.4 Hz, 1H), 2.37 (t, *J* = 7.6 Hz, 2H), 2.23 (s, 3H), 1.79 – 1.62 (m, 2H), 1.48 – 1.33 (m, 2H), 0.94 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.59, 135.59, 130.37, 129.37, 126.60, 125.17, 123.47, 37.16, 27.86, 22.33, 17.73, 13.75.

HRMS (ESI) calcd for $C_{12}H_{18}NO$ $[M+H]^+$: 192.1383, found: 192.1387.



16

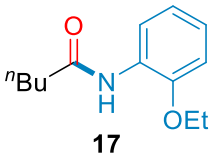
***N*-(2-methoxyphenyl) pentanamide (16):**

Colorless oil (35.5 mg, 89% yield), purified by column chromatography (SiO_2, Pentane/EA= 20:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.39 (dd, *J* = 7.9, 1.8 Hz, 1H), 7.77 (s, 1H), 7.02 (td, *J* = 7.7, 1.8 Hz, 1H), 6.98 – 6.91 (m, 1H), 6.86 (dd, *J* = 7.9, 1.6 Hz, 1H), 3.87 (s, 3H), 2.44 – 2.33 (m, 2H), 1.80 – 1.63 (m, 2H), 1.41 (dq, *J* = 14.5, 7.3 Hz, 2H), 0.95 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.24, 147.61, 127.71, 123.38, 121.02, 119.66, 109.77, 55.59, 37.75, 27.66, 22.31, 13.76.

HRMS (ESI) calcd for $C_{12}H_{18}NO_2$ $[M+H]^+$: 208.1332, found: 208.1336.



17

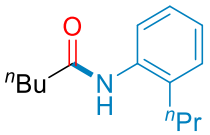
***N*-(2-ethoxyphenyl) pentanamide (17):**

White solid (37.4 mg, 88% yield), purified by column chromatography (SiO_2, Pentane/EA= 20:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.38 (dd, *J* = 7.8, 1.8 Hz, 1H), 7.81 (s, 1H), 7.00 (td, *J* = 7.7, 1.9 Hz, 1H), 6.93 (td, *J* = 7.7, 1.7 Hz, 1H), 6.85 (dd, *J* = 7.8, 1.7 Hz, 1H), 4.10 (q, *J* = 7.0 Hz, 2H), 2.40 (t, 2H), 1.80 – 1.64 (m, 2H), 1.49 – 1.35 (m, 5H), 0.95 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.15, 146.93, 127.77, 123.37, 120.89, 119.69, 110.75, 64.07, 37.73, 27.65, 22.26, 14.80, 13.76.

HRMS (ESI) calcd for $C_{13}H_{20}NO_2$ $[M+H]^+$: 222.1489, found: 222.1490.



18

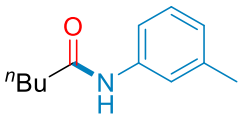
***N*-(2-propylphenyl) pentanamide (18):**

White solid (31.0 mg, 71% yield), purified by column chromatography (SiO_2, Pentane/EA= 10:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.74 (d, *J* = 7.8 Hz, 1H), 7.22 – 7.06 (m, 4H), 2.59 – 2.48 (m, 2H), 2.38 (t, *J* = 7.6 Hz, 2H), 1.78 – 1.67 (m, 2H), 1.60 (dt, *J* = 14.8, 7.5 Hz, 2H), 1.51 – 1.33 (m, 2H), 1.01 – 0.90 (m, 6H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.63, 135.03, 133.70, 129.49, 126.64, 125.32, 124.09, 37.28, 33.44, 27.87, 22.96, 22.34, 13.98, 13.75.

HRMS (EI) calcd for $C_{14}H_{21}NO^+$ $[M]^+$: 219.16177, found: 219.16273.



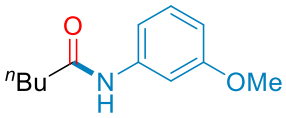
19

***N*-(*m*-tolyl) pentanamide (19) [3]:**

Colorless oil (32.3 mg, 83% yield), purified by column chromatography (SiO_2, Pentane/EA= 10:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.60 (s, 1H), 7.39 (s, 1H), 7.29 (d, *J* = 8.1 Hz, 1H), 7.17 (t, *J* = 7.7 Hz, 1H), 6.90 (d, *J* = 7.4 Hz, 1H), 2.35 (t, *J* = 7.6 Hz, 2H), 2.31 (s, 3H), 1.70 (p, *J* = 7.5 Hz, 2H), 1.39 (h, *J* = 7.3 Hz, 2H), 0.93 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.90, 138.75, 137.81, 128.65, 124.96, 120.64, 117.04, 37.34, 27.74, 22.31, 21.37, 13.75.



20

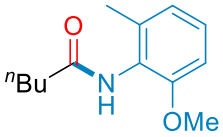
***N*-(3-methoxyphenyl) pentanamide (20):**

Colorless oil (32.8 mg, 80% yield), purified by column chromatography (SiO_2, Pentane/EA= 10:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.60 (s, 1H), 7.32 (t, *J* = 2.3 Hz, 1H), 7.17 (t, *J* = 8.1 Hz, 1H), 6.97 (d, *J* = 8.0 Hz, 1H), 6.64 (dd, *J* = 8.2, 2.6 Hz, 1H), 3.76 (s, 3H), 2.34 (t, 2H), 1.77 – 1.61 (m, 2H), 1.37 (dq, *J* = 14.6, 7.3 Hz, 2H), 0.92 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.73, 160.05, 139.25, 129.52, 111.89, 109.98, 105.47, 55.19, 37.46, 27.62, 22.31, 13.74.

HRMS (ESI) calcd for $C_{12}H_{18}NO_2$ $[M+H]^+$: 208.1332, found: 208.1334.



21

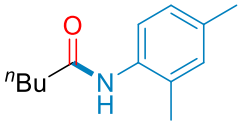
N-(2-methoxy-6-methylphenyl) pentanamide (21):

Colorless oil (27.8 mg, 64% yield), purified by column chromatography (SiO_2, Pentane/EA= 10:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.11 (t, *J* = 8.0 Hz, 1H), 6.95 (s, 1H), 6.82 (d, *J* = 7.7 Hz, 1H), 6.72 (d, *J* = 8.2 Hz, 1H), 3.79 (s, 3H), 2.41 (t, *J* = 7.5 Hz, 2H), 2.22 (s, 3H), 1.81 – 1.65 (m, 2H), 1.53 – 1.35 (m, 2H), 0.96 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.98, 153.67, 136.69, 127.00, 124.32, 122.67, 108.18, 55.58, 36.65, 28.05, 22.33, 18.51, 13.77.

HRMS (ESI) calcd for $C_{13}H_{20}NO_2$ $[M+H]^+$: 222.1489, found: 222.1488.



22

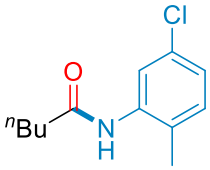
N-(2,4-dimethylphenyl) pentanamide (22):

Colorless oil (24.4 mg, 62% yield), purified by column chromatography (SiO_2, Pentane/EA= 10:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.53 (s, 1H), 6.98 (d, *J* = 7.6 Hz, 2H), 6.80 (d, *J* = 7.7 Hz, 1H), 2.31 (t, *J* = 7.6 Hz, 2H), 2.23 (s, 3H), 2.12 (s, 3H), 1.72 – 1.56 (m, 2H), 1.42 – 1.28 (m, 2H), 0.88 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.50, 136.42, 135.37, 130.17, 125.91, 123.85, 37.27, 27.89, 22.35, 21.03, 17.31, 13.78.

HRMS (ESI) calcd for $C_{13}H_{20}NO$ $[M+H]^+$: 206.1540, found: 206.1544.



23

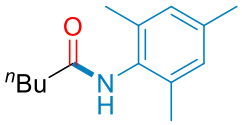
***N*-(5-chloro-2-methylphenyl) pentanamide (23):**

White solid (41.0 mg, 97% yield), purified by column chromatography (SiO_2, Pentane/EA= 10:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.88 (s, 1H), 7.06 (p, *J* = 7.7 Hz, 3H), 2.38 (t, *J* = 7.5 Hz, 2H), 2.19 (s, 3H), 1.70 (p, *J* = 7.1 Hz, 2H), 1.41 (h, *J* = 7.2 Hz, 2H), 0.94 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.49, 136.61, 131.96, 131.20, 126.86, 124.85, 122.84, 37.27, 27.72, 22.33, 17.31, 13.76.

HRMS (ESI) calcd for $C_{12}H_{17}ClNO$ $[M+H]^+$: 226.0993, found: 226.0994.



24

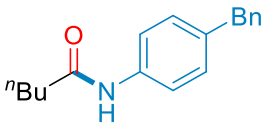
***N*-mesitylpentanamide (24):**

White solid (26.4 mg, 61% yield with 90% purity), purified by column chromatography (SiO_2, Pentane/EA= 10:1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.04 (s, 1H), 6.84 (s, 2H), 2.36 (t, 2H), 2.24 (s, 3H), 2.13 (s, 6H), 1.76 – 1.64 (m, 2H), 1.47 – 1.34 (m, 2H), 0.94 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 172.04, 136.74, 135.09, 131.22, 128.74, 36.32, 28.17, 22.47, 20.85, 18.23, 13.76.

HRMS (ESI) calcd for $C_{14}H_{22}NO$ $[M+H]^+$: 220.1696, found: 220.1701.



25

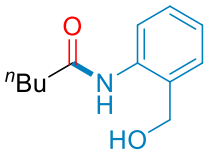
***N*-(4-benzylphenyl) pentanamide (25):**

White solid (25.7 mg, 48% yield), purified by column chromatography (SiO_2, Pentane/EA= 10:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.35 (d, *J* = 8.5 Hz, 2H), 7.28 (s, 1H), 7.22 – 7.16 (m, 2H), 7.14 – 7.01 (m, 5H), 3.85 (s, 2H), 2.30 – 2.19 (m, 2H), 1.61 (p, *J* = 7.7 Hz, 2H), 1.30 (h, *J* = 7.3 Hz, 2H), 0.85 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.46, 141.07, 136.97, 136.02, 129.36, 128.81, 128.41, 126.02, 120.03, 41.28, 37.42, 27.70, 22.33, 13.77.

HRMS (EI) calcd for $C_{18}H_{21}NO^+$ $[M]^+$: 267.16177, found: 267.16133.



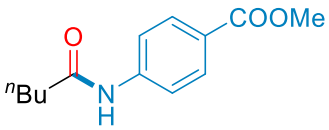
26

***N*-(2-(hydroxymethyl) phenyl) pentanamide (26)[6]:**

White solid (31.5 mg, 77% yield), purified by column chromatography (SiO_2, Pentane/EA= 3:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.69 (s, 1H), 7.95 (d, *J* = 8.1 Hz, 1H), 7.31 – 7.24 (m, 1H), 7.14 (dd, *J* = 7.6, 1.7 Hz, 1H), 7.11 – 6.99 (m, 1H), 4.62 (s, 2H), 2.33 (t, 2H), 1.72 – 1.60 (m, 2H), 1.45 – 1.31 (m, 2H), 0.93 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 172.29, 137.26, 130.04, 128.85, 128.79, 124.29, 122.58, 64.12, 37.53, 27.69, 22.27, 13.73.



27

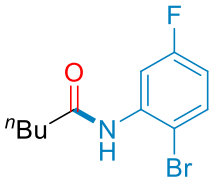
methyl 4-pentanamidobenzoate (27):

White solid (40.5 mg, 86% yield), purified by column chromatography (SiO_2, Pentane/EA= 10:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.00 – 7.94 (m, 2H), 7.81 (s, 1H), 7.64 – 7.59 (m, 2H), 3.88 (s, 3H), 2.43 – 2.32 (m, 2H), 1.78 – 1.61 (m, 2H), 1.45 – 1.31 (m, 2H), 0.92 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.92, 166.68, 142.29, 130.73, 125.31, 118.79, 51.98, 37.49, 27.50, 22.30, 13.72.

HRMS (ESI) calcd for $C_{13}H_{18}NO_3$ $[M+H]^+$: 236.1281, found: 236.1287.



28

***N*-(2-bromo-5-fluorophenyl) pentanamide (28):**

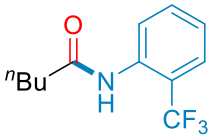
White solid (25.0 mg, 45% yield), purified by column chromatography (SiO_2, Pentane/EA= 20:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.26 (dd, *J* = 11.1, 3.0 Hz, 1H), 7.66 (s, 1H), 7.46 (dd, *J* = 8.8, 5.8 Hz, 1H), 6.71 (ddd, *J* = 8.9, 7.5, 3.0 Hz, 1H), 2.44 (t, 2H), 1.80 – 1.67 (m, 2H), 1.50 – 1.36 (m, 2H), 0.96 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.43, 162.14 (d, *J* = 245.7 Hz), 136.81 (d, *J* = 12.1 Hz), 132.60 (d, *J* = 9.3 Hz), 111.82 (d, *J* = 23.2 Hz), 109.06 (d, *J* = 29.1 Hz), 106.76, 37.75, 27.43, 22.25, 13.74.

^{19}F NMR (282 MHz, $CDCl_3$) δ -111.15 (dt, *J* = 12.2, 6.8 Hz).

HRMS (ESI) calcd for $C_{11}H_{14}BrFNO$ $[M+H]^+$: 274.0237, found: 274.0241.



29

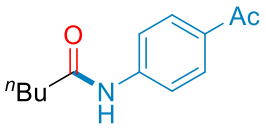
***N*-(2-(trifluoromethyl) phenyl) pentanamide (29) [7]:**

White solid (17.1 mg, 35% yield), purified by column chromatography (SiO_2, Pentane/EA= 20:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.19 (d, *J* = 8.2 Hz, 1H), 7.60 (d, *J* = 7.9 Hz, 1H), 7.54 (t, *J* = 7.9 Hz, 1H), 7.43 (s, 1H), 7.21 (t, *J* = 7.7 Hz, 1H), 2.40 (t, *J* = 7.5 Hz, 2H), 1.77 – 1.69 (m, 2H), 1.49 – 1.35 (m, 2H), 0.95 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.50, 135.29 (d, *J* = 1.8 Hz), 132.82, 125.97 (q, *J* = 5.0 Hz), 124.41 (d, *J* = 14.2 Hz), 122.26, 37.55, 27.47, 22.20, 13.70.

^{19}F NMR (282 MHz, $CDCl_3$) δ -60.64.



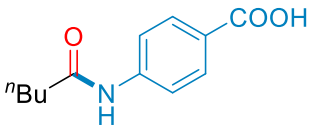
30

***N*-(4-acetylphenyl) pentanamide (30) [3]:**

White solid (37.8 mg, 86% yield), purified by column chromatography (SiO_2, Pentane/EA= 5:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.24 (s, 1H), 7.89 (d, *J* = 7.1 Hz, 2H), 7.66 (d, *J* = 8.2 Hz, 2H), 2.56 (s, 3H), 2.45 – 2.34 (m, 2H), 1.75 – 1.62 (m, 2H), 1.46 – 1.27 (m, 2H), 0.90 (t, *J* = 7.0 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 197.30, 172.23, 142.69, 132.45, 129.64, 118.94, 37.48, 27.50, 26.41, 22.29, 13.72.



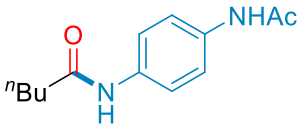
31

4-pentanamidobenzoic acid (31) [8]:

White solid (43.0 mg, 98% yield), purified by column chromatography (SiO_2, Pentane/EA= 10:1).

^{1}H NMR (300 MHz, DMSO) δ 10.17 (s, 1H), 7.88 (d, *J* = 8.6 Hz, 2H), 7.71 (d, *J* = 8.1 Hz, 2H), 2.34 (t, *J* = 7.4 Hz, 2H), 1.66 – 1.50 (m, 2H), 1.42 – 1.20 (m, 2H), 0.90 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, DMSO) δ 172.31, 143.84, 130.82, 118.70, 36.68, 27.58, 22.27, 14.19.



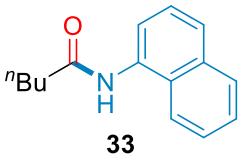
32

***N*-(4-acetamidophenyl) pentanamide (32) [3]:**

White solid (21.3 mg, 77% yield), purified by column chromatography (SiO_2, Pentane/EA= 1:1).

^{1}H NMR (300 MHz, DMSO) δ 9.83 (s, 1H), 9.77 (s, 1H), 7.47 (d, *J* = 1.3 Hz, 4H), 2.27 (t, *J* = 7.4 Hz, 2H), 2.01 (s, 3H), 1.64 – 1.48 (m, 2H), 1.40 – 1.20 (m, 2H), 0.89 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, DMSO) δ 170.93, 167.91, 134.64, 134.58, 119.41, 119.33, 36.04, 27.32, 23.87, 21.85, 13.75.



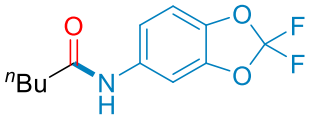
33

***N*-(naphthalen-1-yl) pentanamide (33) [3]:**

White solid (23.6 mg, 53% yield), purified by column chromatography (SiO_2, Pentane/EA= 10:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.84 (dd, *J* = 8.4, 4.6 Hz, 3H), 7.67 (d, *J* = 8.3 Hz, 1H), 7.51 – 7.46 (m, 2H), 7.42 (t, *J* = 7.9 Hz, 1H), 2.47 (t, *J* = 7.6 Hz, 2H), 1.84 – 1.68 (m, 2H), 1.52 – 1.36 (m, 2H), 0.96 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 172.18, 134.06, 132.26, 128.64, 127.32, 126.16, 125.88, 125.78, 125.62, 121.26, 120.74, 37.23, 27.90, 22.41, 13.79.



34

N-(2,2-difluorobenzo[*d*] [1,3] dioxol-5-yl)pentanamide (34):

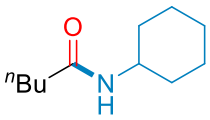
White solid (28.9 mg, 57% yield), purified by column chromatography (SiO_2, Pentane/EA= 10:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.58 (s, 1H), 7.41 (s, 1H), 6.96 (s, 2H), 2.34 (d, *J* = 7.5 Hz, 2H), 1.75 – 1.67 (m, 2H), 1.39 (h, *J* = 7.4 Hz, 2H), 0.94 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.61, 143.83, 140.07, 133.99, 131.73, 114.59, 109.17, 103.25, 37.37, 27.59, 22.34, 13.76.

^{19}F NMR (282 MHz, $CDCl_3$) δ -50.03.

HRMS (ESI) calcd for $C_{12}H_{14}F_2NO_3$ $[M+H]^+$: 258.0937, found: 258.0940.



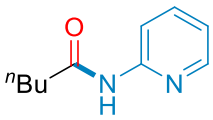
35

***N*-cyclohexylpentanamide (35) [9]:**

White solid (24.4 mg, 69% yield), purified by column chromatography (SiO_2, Pentane/EA= 5:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 3.84 – 3.66 (m, 1H), 2.14 (t, 2H), 1.96 – 1.81 (m, 2H), 1.73 – 1.52 (m, 5H), 1.42 – 1.25 (m, 4H), 1.21 – 1.02 (m, 3H), 0.90 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 172.29, 48.07, 36.69, 33.17, 27.97, 25.49, 24.84, 22.33, 13.76.

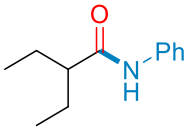


36

***N*-(pyridin-2-yl) pentanamide (36) [3]:**

White sol^{1}H NMR (300 MHz, $CDCl_3$) δ 8.26 (d, *J* = 8.5 Hz, 2H), 7.73 (t, *J* = 7.9 Hz, 1H), 7.05 (dd, *J* = 7.4, 4.2 Hz, 1H), 2.46 – 2.35 (m, 2H), 1.78 – 1.62 (m, 2H), 1.47 – 1.32 (m, 2H), 0.92 (t, *J* = 7.3 Hz, 3H).id (15.4 mg, 44% yield), purified by column chromatography (SiO_2, Pentane/EA= 5:1).

^{13}C NMR (75 MHz, $CDCl_3$) δ 151.42, 146.77, 139.00, 119.57, 114.38, 37.47, 27.35, 22.27, 13.74.



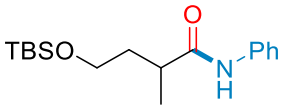
37

2-ethyl-*N*-phenylbutanamide (37) [10]:

White solid (35.9 mg, 95% yield), purified by column chromatography (SiO_2, Pentane/EA= 10:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.49 (d, *J* = 8.0 Hz, 2H), 7.21 (t, *J* = 7.6 Hz, 2H), 7.01 (t, *J* = 7.3 Hz, 1H), 2.07 – 1.92 (m, 1H), 1.68 – 1.54 (m, 2H), 1.54 – 1.38 (m, 2H), 0.87 (t, *J* = 7.3 Hz, 6H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 174.63, 138.03, 128.92, 124.20, 120.16, 52.19, 25.89, 12.10.



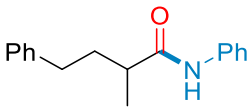
38

4-((*tert*-butyldimethylsilyl) oxy)-2-methyl-*N*-phenylbutanamide (38) [3]:

White solid (57.0 mg, 93% yield), purified by column chromatography (SiO_2, Pentane/EA= 10:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.75 (s, 1H), 7.52 (d, *J* = 7.9 Hz, 2H), 7.36 – 7.23 (m, 2H), 7.14 – 7.02 (m, 1H), 3.79 – 3.61 (m, 2H), 2.74 – 2.56 (m, 1H), 1.98 – 1.81 (m, 1H), 1.78 – 1.61 (m, 1H), 1.23 (d, *J* = 6.9 Hz, 3H), 0.92 (s, 9H), 0.07 (d, *J* = 2.5 Hz, 6H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 174.62, 138.08, 128.85, 123.92, 119.68, 60.69, 38.20, 37.01, 25.90, 18.23, 17.51, -5.36.



39

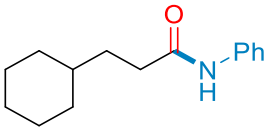
2-methyl-*N*,4-diphenylbutanamide (39):

White solid (48.5 mg, 97% yield), purified by column chromatography (SiO_2, Pentane/EA= 10:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.56 (d, *J* = 7.9 Hz, 2H), 7.48 (s, 1H), 7.36 – 7.26 (m, 4H), 7.25 – 7.16 (m, 3H), 7.15 – 7.08 (m, 1H), 2.80 – 2.56 (m, 2H), 2.42 – 2.27 (m, 1H), 2.20 – 2.02 (m, 1H), 1.86 – 1.72 (m, 1H), 1.26 (d, *J* = 6.8 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 174.70, 141.49, 137.90, 128.90, 128.38, 128.34, 125.93, 124.18, 119.90, 41.56, 35.59, 33.41, 18.01.

HRMS (EI) calcd for $C_{17}H_{19}NO^+$ $[M]^+$: 253.14612, found: 253.14594.



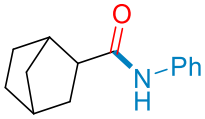
40

3-cyclohexyl-*N*-phenylpropanamide (40) [11]:

White solid (48.5 mg, 97% yield), purified by column chromatography (SiO_2, Pentane/EA= 10:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.75 (s, 1H), 7.52 (d, *J* = 7.4 Hz, 2H), 7.35 – 7.21 (m, 2H), 7.08 (t, *J* = 7.4 Hz, 1H), 2.42 – 2.30 (m, 2H), 1.78 – 1.54 (m, 7H), 1.37 – 1.03 (m, 4H), 1.02 – 0.80 (m, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 172.12, 138.01, 128.84, 124.09, 119.94, 37.26, 35.14, 33.00, 26.45, 26.16.



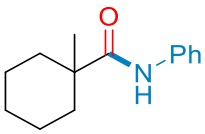
41

***N*-phenylbicyclo [2.2.1] heptane-2-carboxamide (41) [3]:**

White solid (40.1 mg, 95% yield), purified by column chromatography (SiO_2, Pentane/EA= 10:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.52 (d, *J* = 7.9 Hz, 2H), 7.29 (t, 2H), 7.07 (t, *J* = 7.3 Hz, 1H), 2.50 (s, 1H), 2.41 – 2.22 (m, 2H), 2.05 – 1.91 (m, 1H), 1.65 (d, *J* = 10.0 Hz, 1H), 1.59 – 1.42 (m, 3H), 1.29 – 1.13 (m, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 174.13, 138.27, 128.85, 123.86, 119.70, 48.96, 41.67, 36.42, 35.90, 34.18, 29.73, 28.60.



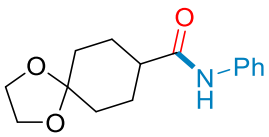
42

1-methyl-*N*-phenylcyclohexane-1-carboxamide (42) [3]:

White solid (34.9 mg, 82% yield), purified by column chromatography (SiO_2, Pentane/EA= 10:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.59 – 7.47 (m, 2H), 7.31 (t, *J* = 7.9 Hz, 2H), 7.15 – 7.03 (m, 1H), 2.08 – 1.95 (m, 2H), 1.70 – 1.32 (m, 8H), 1.25 (s, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 175.89, 138.08, 128.84, 124.03, 120.01, 43.52, 35.66, 26.34, 25.68, 22.84.



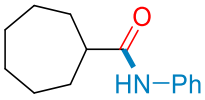
43

***N*-phenyl-1,4-dioxaspiro [4.5]decane-8-carboxamide (43) [3]:**

White solid (55.4 mg, quant. yield), purified by column chromatography (SiO_2, Pentane/EA= 3:1).

^{1}H NMR (300 MHz, DMSO) δ 9.85 (s, 1H), 7.65 – 7.54 (m, 2H), 7.38 – 7.20 (m, 2H), 7.07 – 6.95 (m, 1H), 3.87 (q, *J* = 0.9 Hz, 4H), 2.36 (tt, *J* = 11.1, 3.6 Hz, 1H), 1.87 – 1.65 (m, 6H), 1.48 (td, *J* = 13.8, 5.4 Hz, 2H).

^{13}C NMR (75 MHz, DMSO) δ 174.20, 139.86, 129.08, 123.40, 119.53, 107.86, 64.17, 64.11, 43.77, 34.07, 27.12.



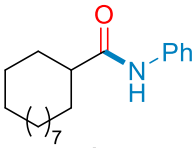
44

***N*-phenylcycloheptanecarboxamide (44) [12]:**

White solid (42.7 mg, 97% yield), purified by column chromatography (SiO_2, Pentane/EA= 20:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.53 (d, *J* = 7.2 Hz, 2H), 7.33 – 7.25 (m, 2H), 7.11 – 7.03 (m, 1H), 2.45 – 2.34 (m, 1H), 2.02 – 1.92 (m, 2H), 1.85 – 1.72 (m, 4H), 1.64 – 1.39 (m, 6H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 175.77, 138.17, 128.83, 123.95, 119.83, 48.26, 31.56, 28.08, 26.49.



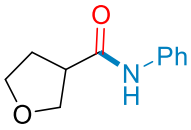
45

***N*-phenylcyclododecanecarboxamide (45) [3]:**

White solid (49.1 mg, 87% yield), purified by column chromatography (SiO_2, Pentane/EA= 20:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.54 (d, *J* = 7.3 Hz, 2H), 7.37 (s, 1H), 7.29 (t, *J* = 7.9 Hz, 2H), 7.14 – 7.02 (m, 1H), 2.50 – 2.35 (m, 1H), 1.78 – 1.63 (m, 4H), 1.54 – 1.20 (m, 18H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 174.93, 138.08, 128.87, 124.02, 119.82, 43.23, 27.34, 23.64, 23.56, 23.34, 22.54.



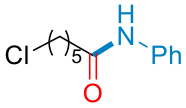
46

***N*-phenyltetrahydrofuran-3-carboxamide (46)** [3]**:**

White solid (23.7 mg, 64% yield), purified by column chromatography (SiO_2, Pentane/EA= 10:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.73 (s, 1H), 7.51 (d, *J* = 7.1 Hz, 2H), 7.30 (t, *J* = 7.8 Hz, 2H), 7.10 (t, *J* = 7.4 Hz, 1H), 4.11 – 3.90 (m, 3H), 3.88 – 3.77 (m, 1H), 3.12 – 2.97 (m, 1H), 2.36 – 2.15 (m, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 172.05, 137.72, 128.96, 124.40, 119.95, 70.94, 68.16, 46.47, 30.56.



47

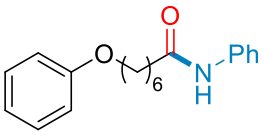
6-chloro-*N*-phenylhexanamide (47):

White solid (32.7 mg, 75% yield), purified by column chromatography (SiO_2, Pentane/EA= 10:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.57 (s, 1H), 7.51 (d, *J* = 7.9 Hz, 2H), 7.30 (t, *J* = 7.9 Hz, 2H), 7.15 – 7.04 (m, 1H), 3.52 (t, *J* = 6.6 Hz, 2H), 2.36 (t, *J* = 7.4 Hz, 2H), 1.87 – 1.66 (m, 4H), 1.58 – 1.39 (m, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.20, 137.83, 128.92, 124.25, 119.92, 44.78, 37.31, 32.22, 26.41, 24.74.

HRMS (EI) calcd for $C_{12}H_{16}ClNO^+$ $[M]^+$: 225.09149, found: 225.09149.



48

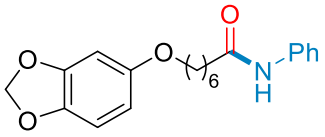
7-phenoxy-*N*-phenylheptanamide (48):

White solid (35.8 mg, 56% yield), purified by column chromatography (SiO_2, Pentane/EA= 5:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.51 (d, *J* = 7.4 Hz, 2H), 7.33 – 7.24 (m, 4H), 7.10 (t, *J* = 7.4 Hz, 1H), 6.97 – 6.87 (m, 3H), 3.95 (t, *J* = 6.4 Hz, 2H), 2.37 (t, *J* = 7.5 Hz, 2H), 1.84 – 1.70 (m, 4H), 1.59 – 1.39 (m, 4H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.30, 159.00, 137.87, 129.39, 128.96, 124.19, 120.49, 119.78, 114.44, 67.60, 37.61, 29.07, 28.92, 25.82, 25.47.

HRMS (EI) calcd for $C_{19}H_{23}NO_2^+$ $[M]^+$: 297.17233, found: 297.17235.



49

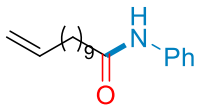
7-(benzo[*d*] [1,3] dioxol-5-yloxy)-*N*-phenylheptanamide (49):

White solid (40.2 mg, 61% yield), purified by column chromatography (SiO_2, Pentane/EA= 5:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.52 (d, *J* = 8.1 Hz, 2H), 7.30 (t, *J* = 7.9 Hz, 2H), 7.09 (t, *J* = 7.4 Hz, 1H), 6.69 (d, *J* = 8.5 Hz, 1H), 6.48 (d, *J* = 2.5 Hz, 1H), 6.30 (dd, *J* = 8.5, 2.5 Hz, 1H), 5.89 (s, 2H), 3.86 (t, *J* = 6.4 Hz, 2H), 2.35 (t, *J* = 7.5 Hz, 2H), 1.80 – 1.66 (m, 4H), 1.53 – 1.35 (m, 4H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.42, 154.52, 148.14, 141.41, 137.92, 128.89, 124.14, 119.82, 107.87, 105.61, 101.00, 97.98, 68.67, 37.51, 29.05, 28.89, 25.75, 25.45.

HRMS (EI) calcd for $C_{20}H_{23}NO_4^+$ $[M]^+$: 341.16216, found: 341.16202.



50

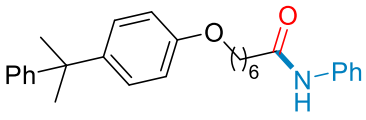
***N*-phenyldodec-11-enamide (50):**

White solid (20.2 mg, 39% yield), purified by column chromatography (SiO_2, Pentane/EA= 10:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.51 (d, *J* = 7.9 Hz, 2H), 7.31 (t, *J* = 7.9 Hz, 2H), 7.13 – 7.05 (m, 1H), 5.91 – 5.71 (m, 1H), 5.06 – 4.86 (m, 2H), 2.35 (t, *J* = 7.6 Hz, 2H), 2.11 – 1.96 (m, 2H), 1.76 – 1.66 (m, 2H), 1.40 – 1.26 (m, 12H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.46, 139.19, 137.92, 128.95, 124.15, 119.76, 114.10, 37.81, 33.77, 29.39, 29.33, 29.23, 29.07, 28.88, 25.61.

HRMS (EI) calcd for $C_{18}H_{27}NO^+$ $[M]^+$: 273.20872, found: 273.20889.



51

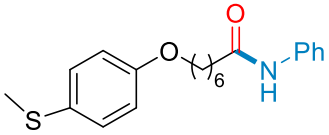
7-(2,2-diphenylpropoxy)-*N*-phenylheptanamide (51):

White solid (61.3 mg, 75% yield), purified by column chromatography (SiO_2, Pentane/EA= 20:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.68 (s, 1H), 7.56 (d, *J* = 7.9 Hz, 2H), 7.38 – 7.25 (m, 6H), 7.24 – 7.09 (m, 4H), 6.88 – 6.78 (m, 2H), 3.95 (t, *J* = 6.3 Hz, 2H), 2.38 (t, *J* = 7.5 Hz, 2H), 1.87 – 1.72 (m, 4H), 1.70 (s, 6H), 1.58 – 1.42 (m, 4H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.55, 156.81, 150.86, 142.64, 137.91, 128.85, 127.86, 127.67, 126.64, 125.43, 124.11, 119.86, 113.74, 67.58, 42.19, 37.47, 30.81, 29.07, 28.88, 25.79, 25.47.

HRMS (EI) calcd for $C_{28}H_{33}NO_2^+$ $[M]^+$: 415.25058, found: 415.25052.



52

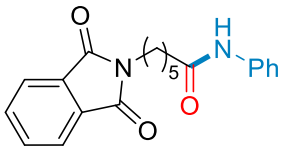
7-(4-(methylthio) phenoxy)-*N*-phenylheptanamide (52):

White solid (20.4 mg, 30% yield), purified by column chromatography (SiO_2, Pentane/EA= 10:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.51 (d, *J* = 8.0 Hz, 2H), 7.35 – 7.32 (m, 1H), 7.29 (d, *J* = 8.1 Hz, 2H), 7.27 – 7.22 (m, 2H), 7.13 – 7.06 (m, 1H), 6.85 – 6.79 (m, 2H), 3.92 (t, *J* = 6.4 Hz, 2H), 2.44 (s, 3H), 2.36 (t, *J* = 7.5 Hz, 2H), 1.83 – 1.69 (m, 4H), 1.55 – 1.37 (m, 4H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.50, 157.69, 137.85, 130.21, 129.02, 128.56, 124.34, 119.89, 115.22, 67.93, 37.56, 29.06, 28.95, 25.83, 25.54, 18.13.

HRMS (EI) calcd for $C_{20}H_{25}NO_2S^+$ $[M]^+$: 343.16005, found: 343.16026.



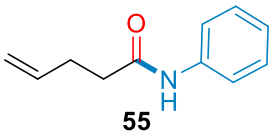
53

7-(1,3-dioxoisoindolin-2-yl)-*N*-phenylheptanamide (53) [3]:

White solid (19.9 mg, 29% yield), purified by column chromatography (SiO_2, Pentane/EA= 5:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.83 – 7.79 (m, 2H), 7.71 – 7.68 (m, 2H), 7.51 (d, *J* = 7.7 Hz, 2H), 7.44 (s, 1H), 7.30 (d, *J* = 7.7 Hz, 2H), 7.07 (t, *J* = 7.4 Hz, 1H), 3.69 (t, *J* = 7.2 Hz, 2H), 2.35 (t, *J* = 7.5 Hz, 2H), 1.86 – 1.64 (m, 4H), 1.50 – 1.36 (m, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.07, 168.45, 137.93, 133.89, 132.04, 128.90, 124.12, 123.16, 119.80, 37.60, 37.37, 28.15, 26.23, 24.89.



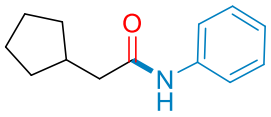
55

***N*-phenylpent-4-enamide (55)[13]:**

White solid (8.6 mg, 25% yield), purified by column chromatography (SiO_2, Pentane/EA= 20:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.50 (d, *J* = 7.2 Hz, 2H), 7.36 – 7.28 (m, 2H), 7.10 (t, *J* = 7.4 Hz, 1H), 5.98 – 5.79 (m, 1H), 5.20 – 5.01 (m, 2H), 2.55 – 2.39 (m, 4H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 170.51, 136.83, 128.99, 124.27, 119.81, 115.96, 77.20, 36.81, 29.68, 29.42.



58

2-cyclopentyl-*N*-phenylacetamide (58)[14]:

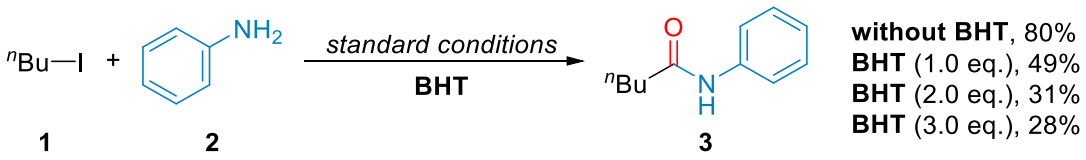
White solid (10.4 mg, 26% yield), purified by column chromatography (SiO_2, Pentane/EA= 20:1).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.51 (d, *J* = 8.0 Hz, 2H), 7.31 (t, *J* = 7.8 Hz, 2H), 7.16 – 7.04 (m, 1H), 2.36 (d, *J* = 1.9 Hz, 2H), 1.97 – 1.80 (m, 2H), 1.73 (s, 1H), 1.70 – 1.61 (m, 2H), 1.60 – 1.52 (m, 2H), 1.27 – 1.13 (m, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.11, 137.92, 128.96, 124.16, 119.81, 44.00, 37.17, 32.54, 24.93.

5. Mechanism studies

5.1 Radical inhibition experiments

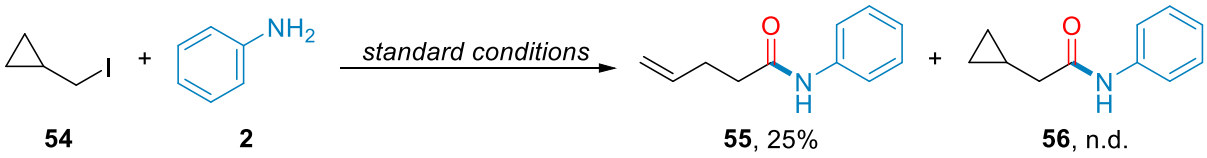


Scheme S1. Radical inhibition experiments

A 4 mL snap vial equipped with a magnetic stir bar was charged with $Mn_2(CO)_{10}$ (0.01 mmol, 5 mol %), K_3PO_4 (0.3 mmol, 1.5 equiv.) and closed with a rubber-based septum. Different amounts of 2,6-di-tert-butyl-4-methylphenol (BHT) (1 – 3 equiv.) were added sequentially to the corresponding vials. The vial was evacuated and backfilled with argon. Degassed dioxane (0.5 mL), aniline (0.2 mmol, 1 equiv.) and alkyl iodides (0.3 mmol, 1.5 equiv.) were added via syringe. The vial was then connected to atmosphere with a cannula and transferred into a 300 mL photoautoclav, manufactured by Parr instrument company®, under argon counterflow. As depicted, the vials were placed in a revolver-like manner and without an alloy plate, 9 vials did exactly fit. The closed autoclave was flushed three times with nitrogen (~ 5 bar), three times with CO (~ 5 bar), and 1 bar of carbon monoxide (measured by pressure meter) was charged. The autoclave was then placed into an aluminum block on a magnetic stirrer. The reaction mixture was stirred (500 rpm) at 25 °C while being irradiated with light at the chosen wavelength (460 nm – blue) from a portable Lumatec SUPERLITE S 04[1] for the specified time without additional cooling. After irradiation, the light was turned off and cooling to room temperature. And a proper amount of solvent was taken for GC analysis. The result is shown above.

As BHT was added to the reaction system, the reaction was gradually inhibited.

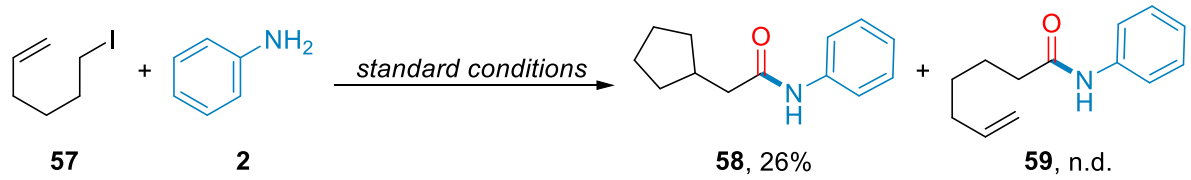
5.2 Radical clock experiments



Scheme S2. Radical clock reaction of alkyl iodides

A 4 mL snap vial equipped with a magnetic stir bar was charged with $Mn_2(CO)_{10}$ (0.01 mmol, 5 mol %), K_3PO_4 (0.3 mmol, 1.5 equiv.) and closed with a rubber-based septum. The vial was evacuated and backfilled with argon. Degassed dioxane (0.5 mL), aniline (0.2 mmol, 1 equiv.) and (iodomethyl)cyclopropane (0.3 mmol, 1.5 equiv.) were added via syringe. The vial was then connected to atmosphere with a cannula and transferred into a 300 mL photoautoclav, manufactured by Parr instrument company®, under argon counterflow. As depicted, the vials were placed in a revolver-like manner and without an alloy plate, 9 vials did exactly fit. The closed autoclave was flushed three times with nitrogen (~ 5 bar), three times with CO (~ 5 bar), and 1 bar of carbon monoxide (measured by pressure meter) was charged. The autoclave was then placed into an aluminum block on a magnetic stirrer. The reaction mixture was stirred (500 rpm) at 25 °C while being irradiated with light at the chosen wavelength (460 nm – blue) from a portable Lumatec SUPERLITE S 04 for the specified time without additional cooling. After irradiation, the light was turned off and cooling to room temperature. Then, a proper amount of solvent was taken

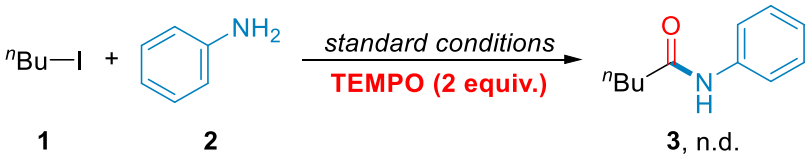
for GC-MS analysis. The result is shown above. And the crude residue was purified by silica gel chromatography (pentane/EA = 10:1, Rf = 0.4) to give the product 76 as a colorless oil (8.6 mg, 25%).



Scheme S3. Radical cyclization reaction of alkyl iodides

A 4 mL snap vial equipped with a magnetic stir bar was charged with $Mn_2(CO)_{10}$ (0.01 mmol, 5 mol %), K_3PO_4 (0.3 mmol, 1.5 equiv.) and closed with a rubber-based septum. The vial was evacuated and backfilled with argon. Degassed dioxane (0.5 mL), aniline (0.2 mmol, 1 equiv.) and 6-iodohex-1-ene (0.3 mmol, 1.5 equiv.) were added via syringe. The vial was then connected to atmosphere with a cannula and transferred into a 300 mL photoautoclav, manufactured by Parr instrument company®, under argon counterflow. As depicted, the vials were placed in a revolver-like manner and without an alloy plate, 9 vials did exactly fit. The closed autoclave was flushed three times with nitrogen (~ 5 bar), three times with CO (~ 5 bar), and 1 bar of carbon monoxide (measured by pressure meter) was charged. The autoclave was then placed into an aluminum block on a magnetic stirrer. The reaction mixture was stirred (500 rpm) at 25 °C while being irradiated with light at the chosen wavelength (460 nm – blue) from a portable Lumatec SUPERLITE S 04 for the specified time without additional cooling. After irradiation, the light was turned off and cooling to room temperature. Then, a proper amount of solvent was taken for GC-MS analysis. The result is shown above. And the crude residue was purified by silica gel chromatography (pentane/EA = 20:1, Rf = 0.2) to give the product 58 as a white solid (10.4 mg, 26%).

5.3 Radical capture experiment



Scheme S4. Radical capture experiment

A 4 mL snap vial equipped with a magnetic stir bar was charged $Mn_2(CO)_{10}$ (0.01 mmol, 5 mol %), K_3PO_4 (0.3 mmol, 1.5 equiv.), 2,2,6,6-tetramethylpiperidine-1-oxyl (TEMPO) (2 equiv.) and closed with a rubber-based septum. The vial was evacuated and backfilled with argon. Degassed dioxane (0.5 mL) and substrate alkyl iodides (0.3 mmol, 1.5 equiv) were added via syringe. The vial was then connected to atmosphere with a cannula and transferred into a 300 mL photoautoclav, manufactured by Parr instrument company®, under argon counterflow. As depicted, the vials were placed in a revolver-like manner and without an alloy plate, 9 vials did exactly fit. The closed autoclave was flushed three times with nitrogen (~ 5 bar), three times with CO (~ 5 bar), and 1 bar of carbon monoxide (measured by pressure meter) was charged. The autoclave was then placed into an aluminum block on a magnetic stirrer. The reaction mixture was stirred (500 rpm) at 25 °C while being irradiated with light at the chosen wavelength (460 nm – blue) from a portable Lumatec SUPERLITE S 04 for the specified time without additional cooling. After irradiation, the light was turned off and cooling to room temperature. And a proper amount of solvent was taken for GC analysis. The result is shown above.

6. Reference

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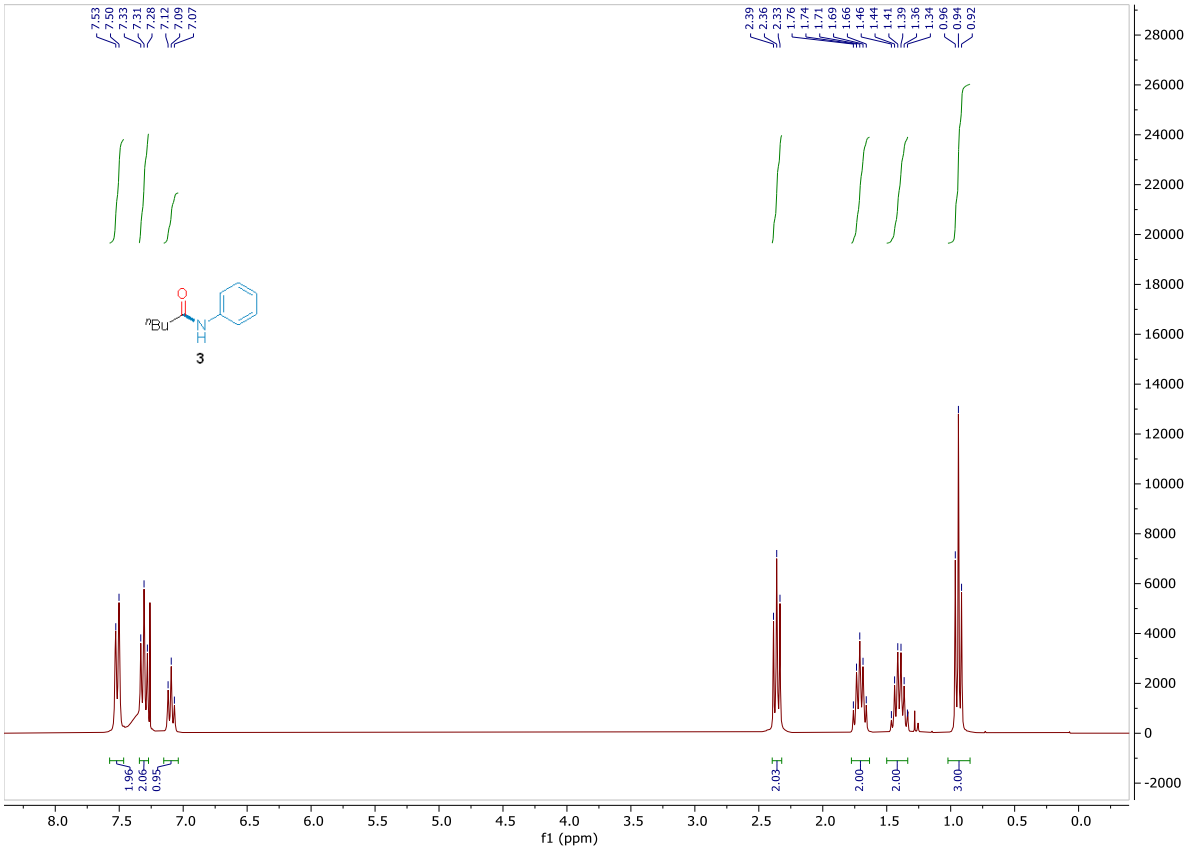
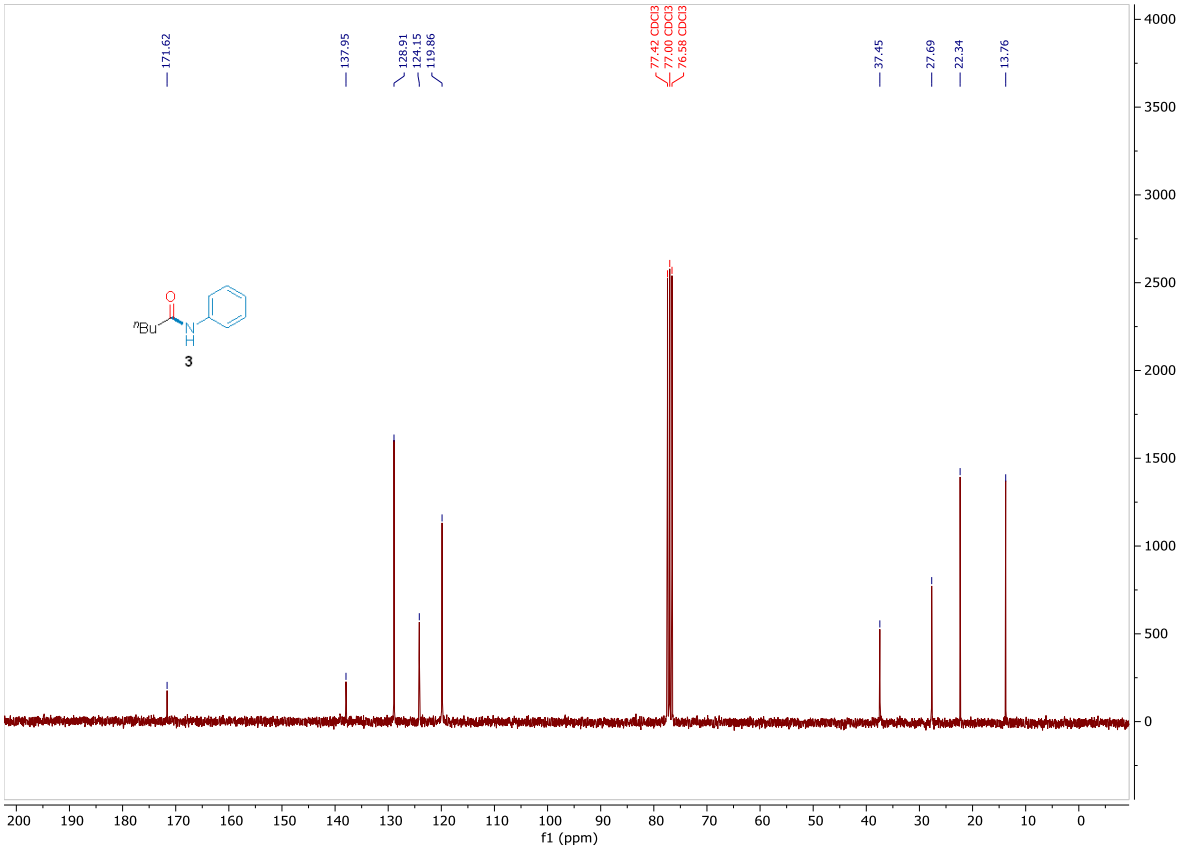
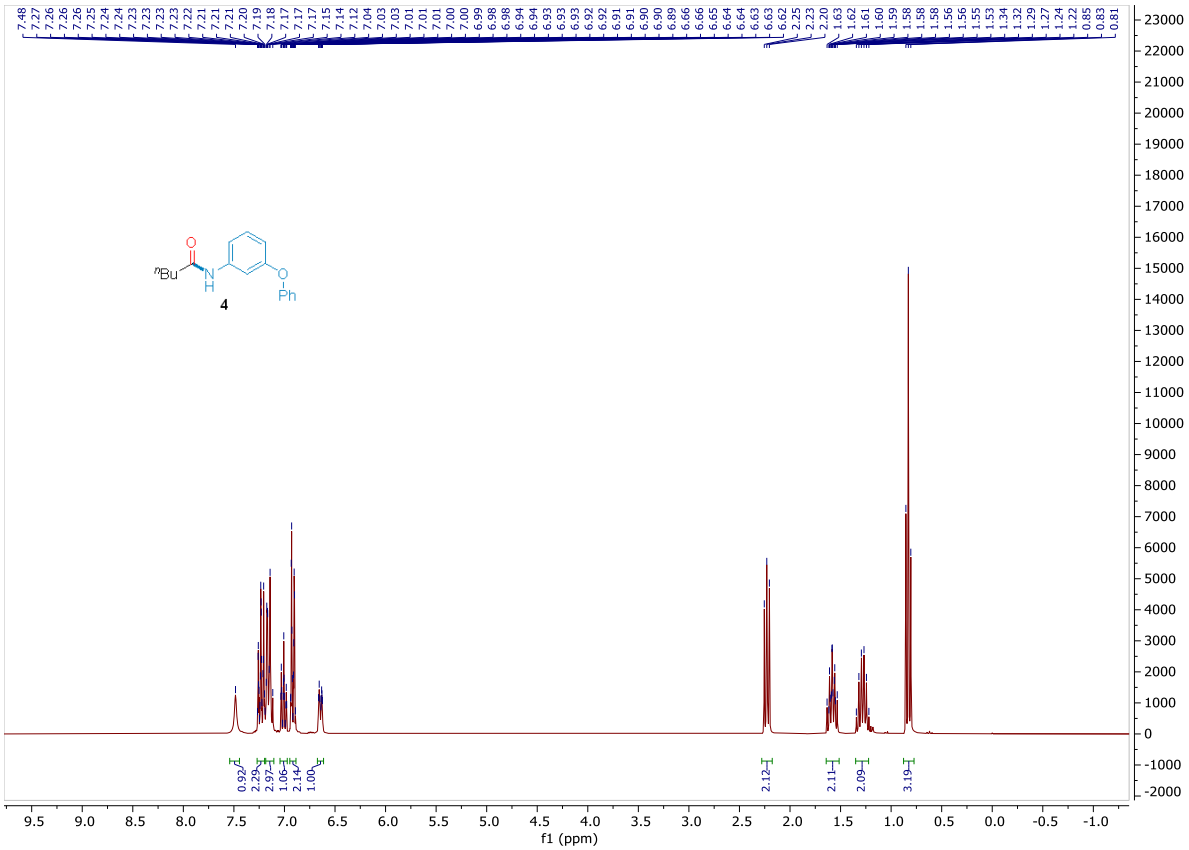
7. NMR spectra of products



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O
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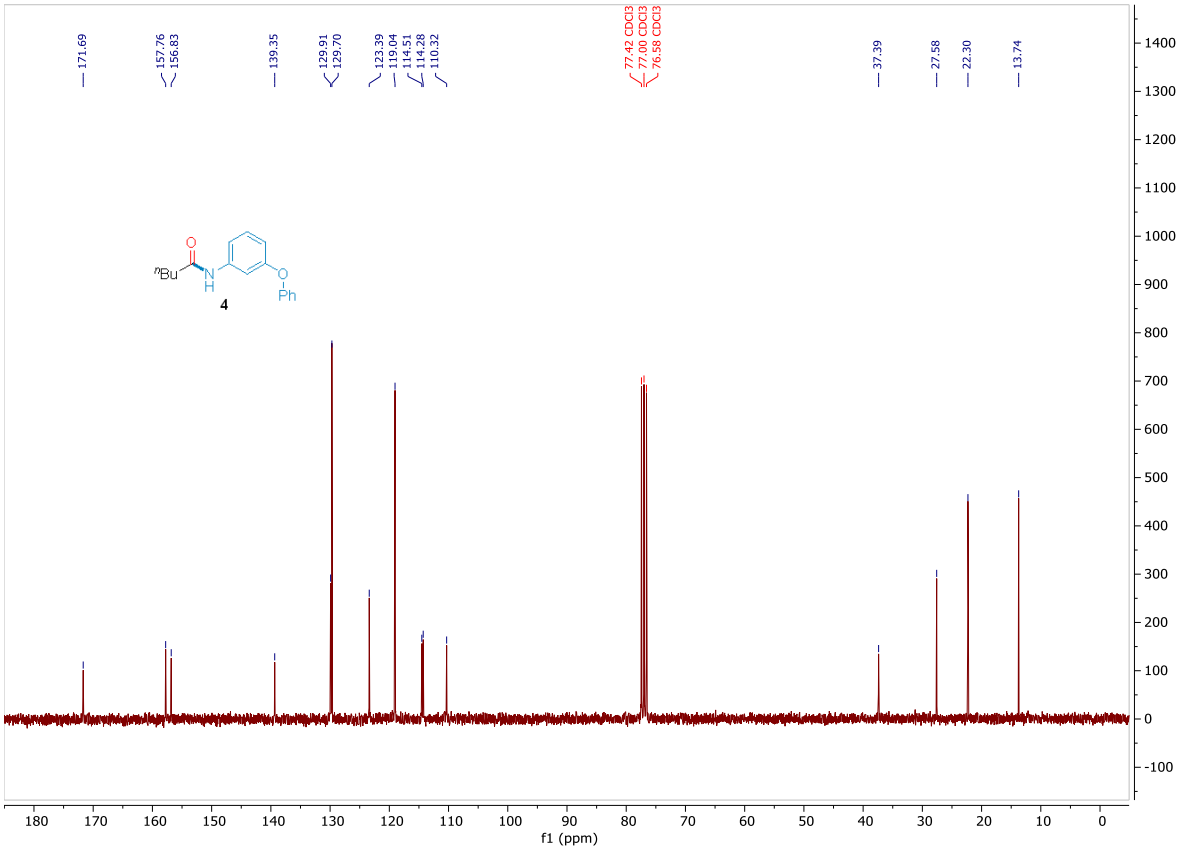
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Ph
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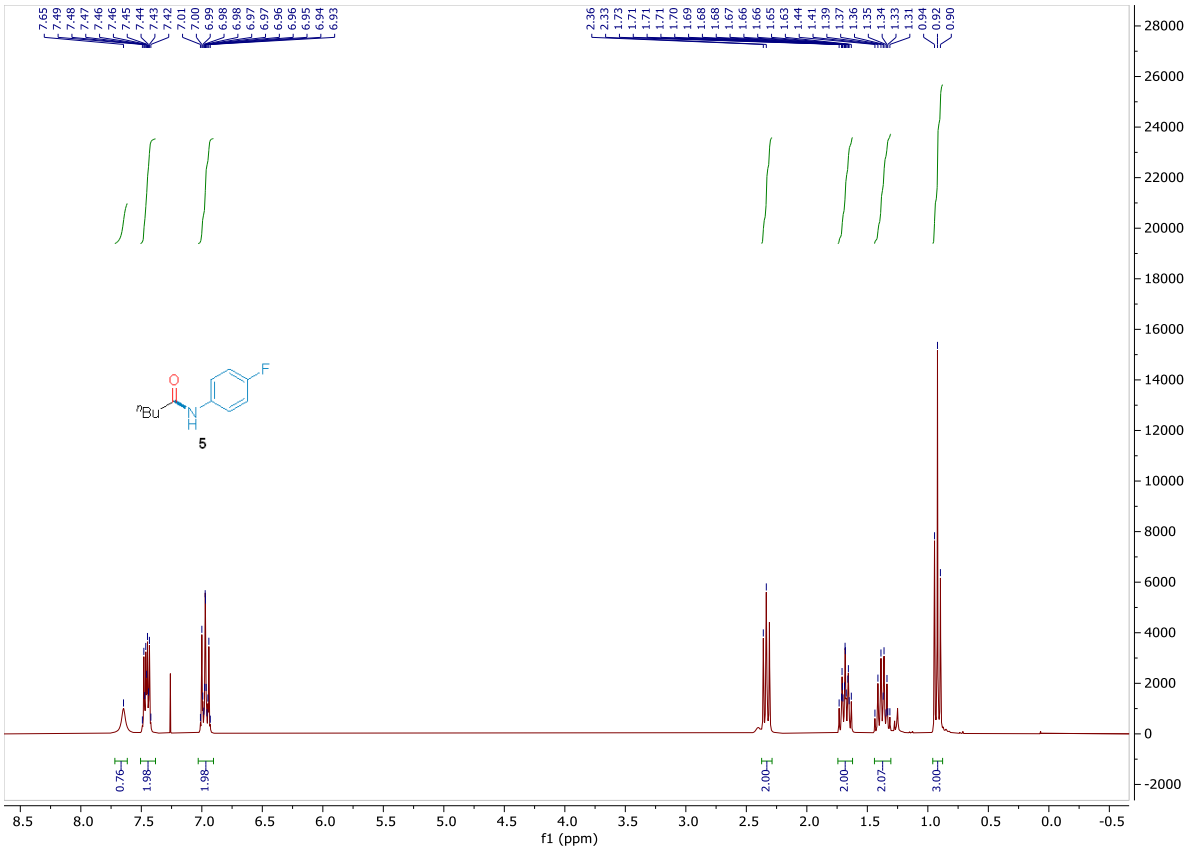
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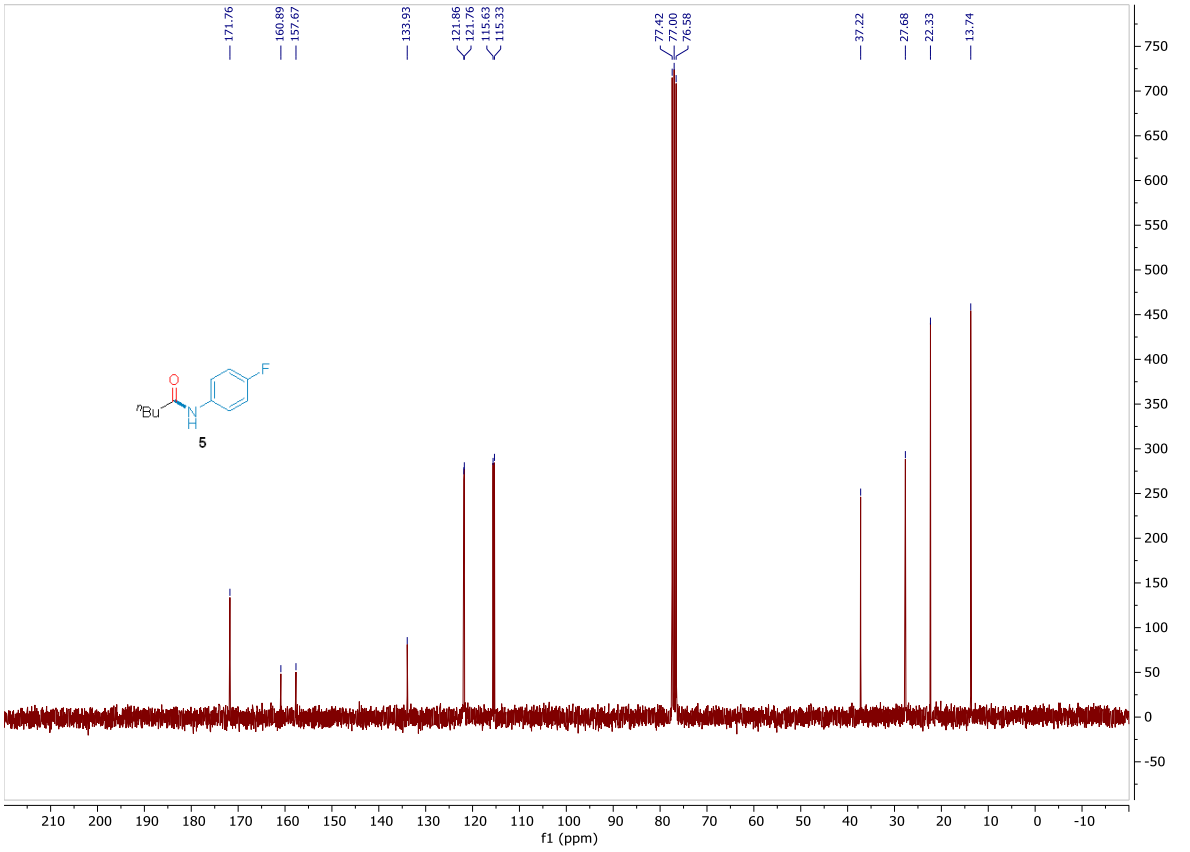
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F
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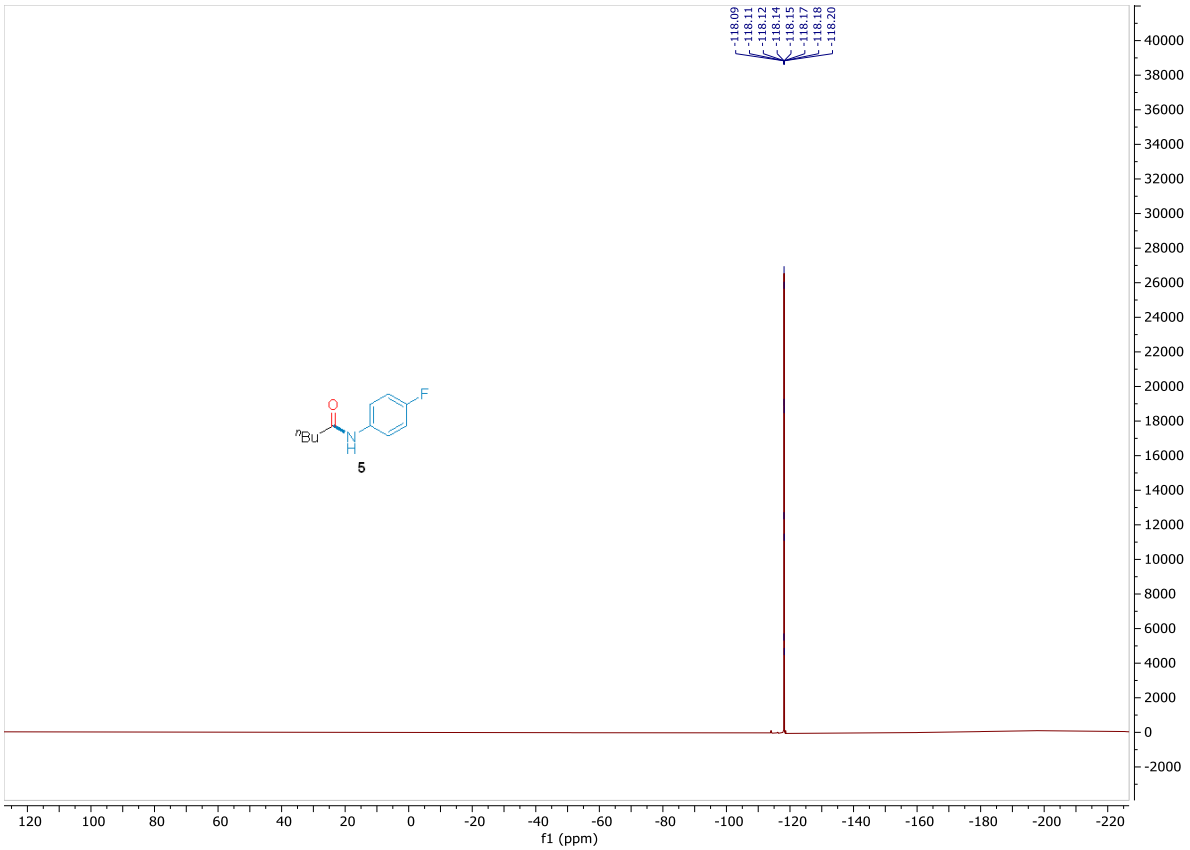
f1 (ppm)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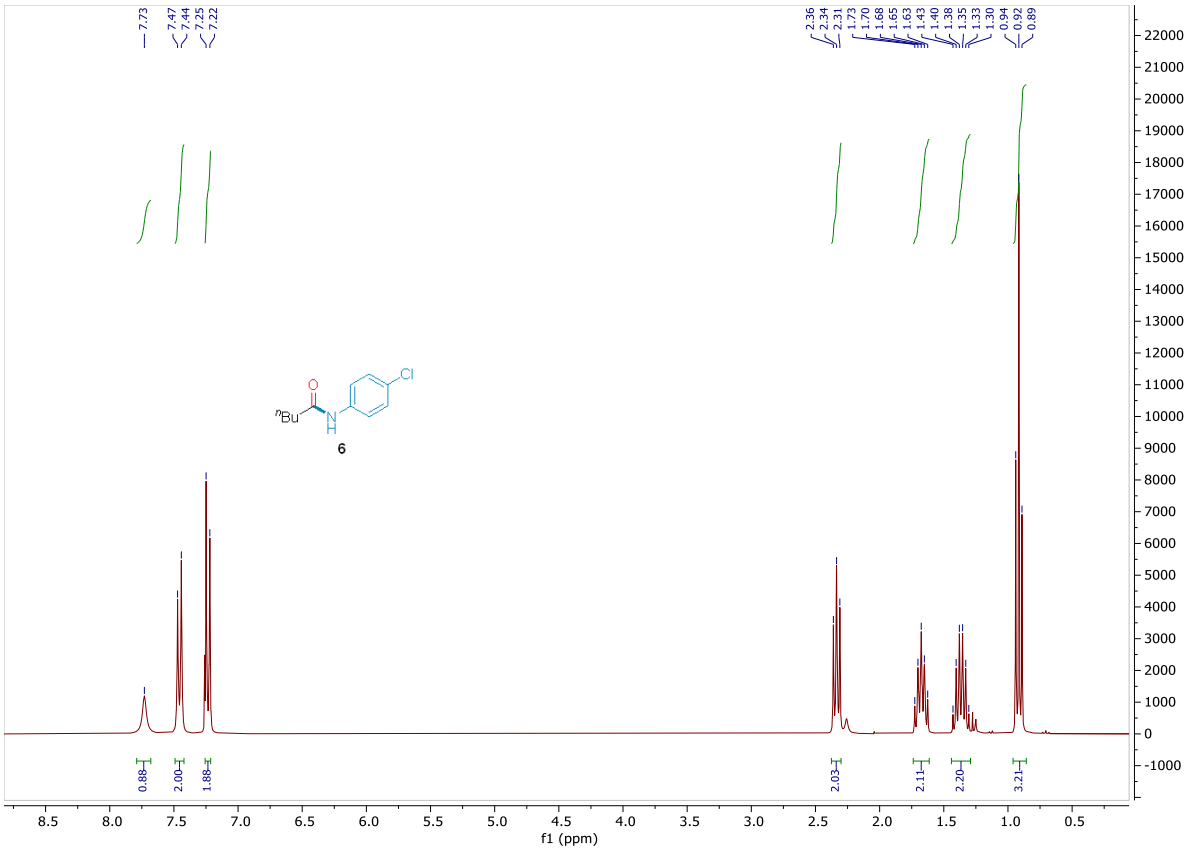
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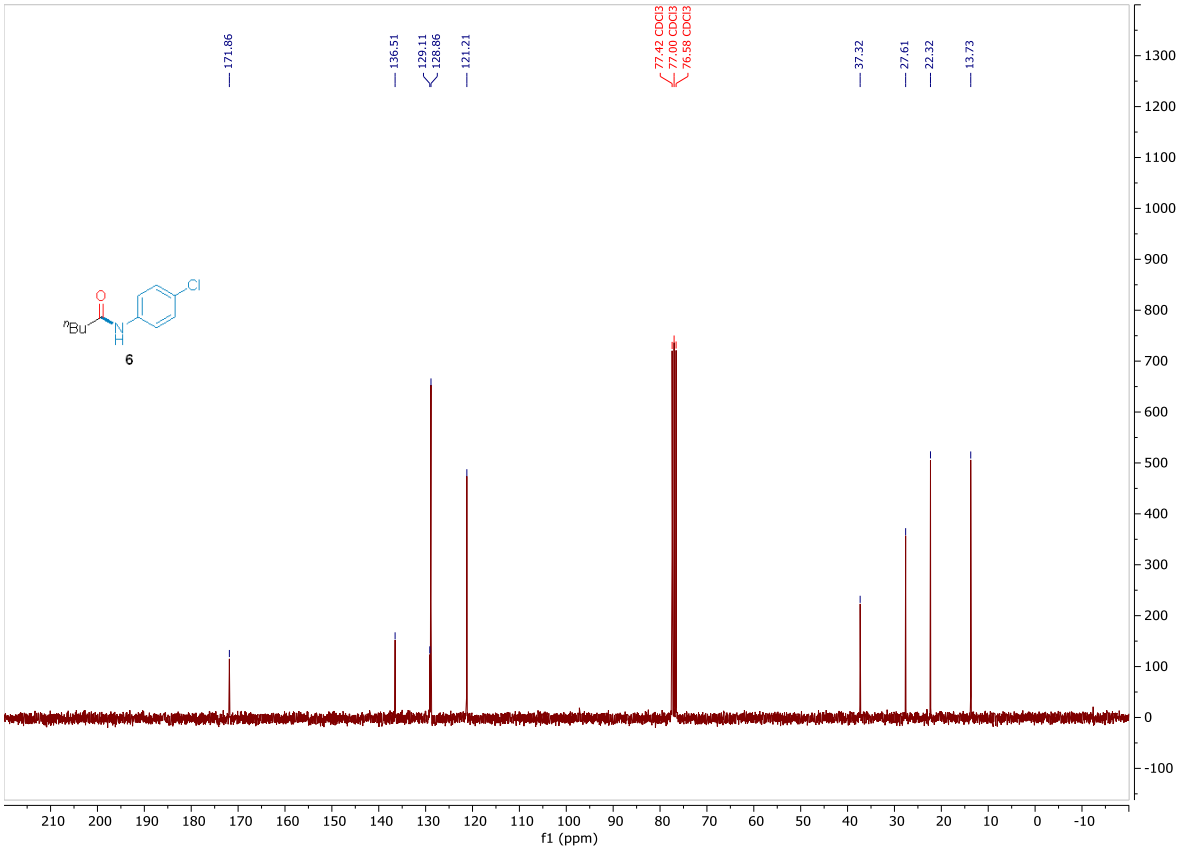
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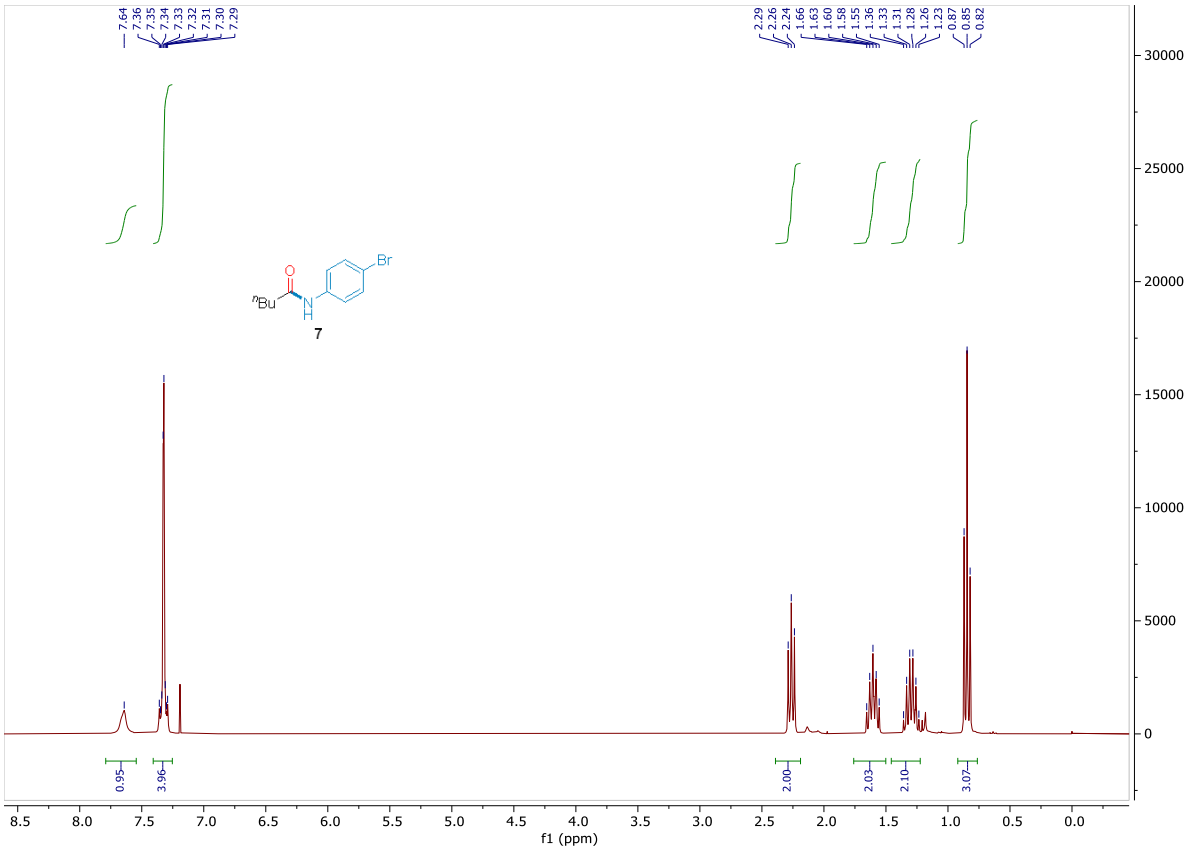
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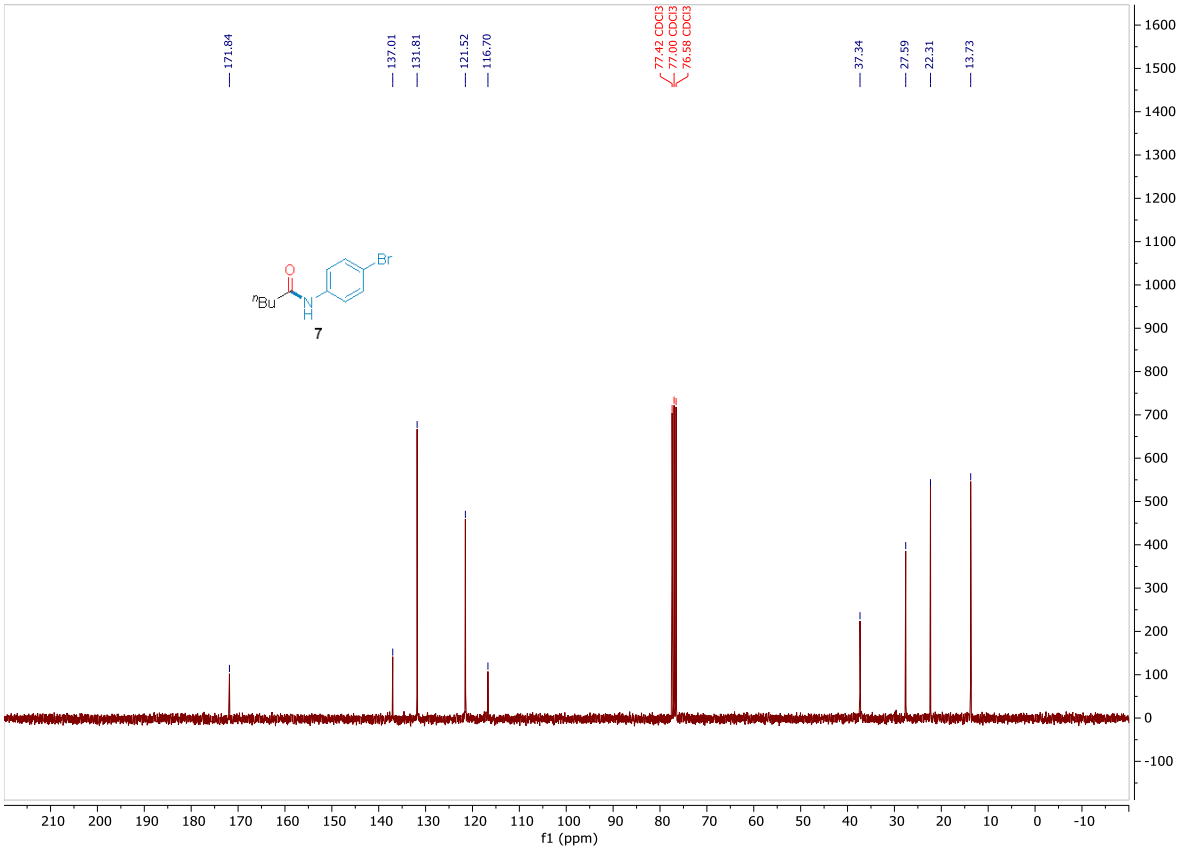
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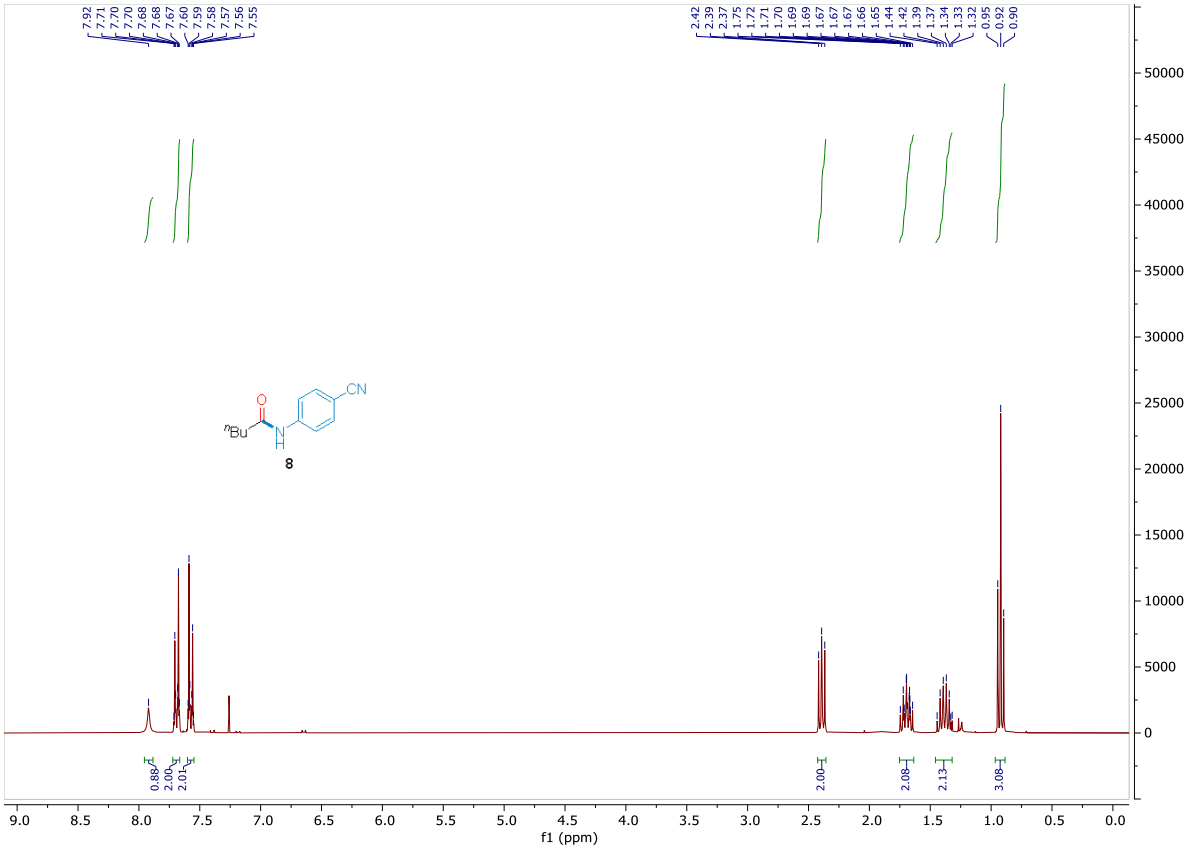
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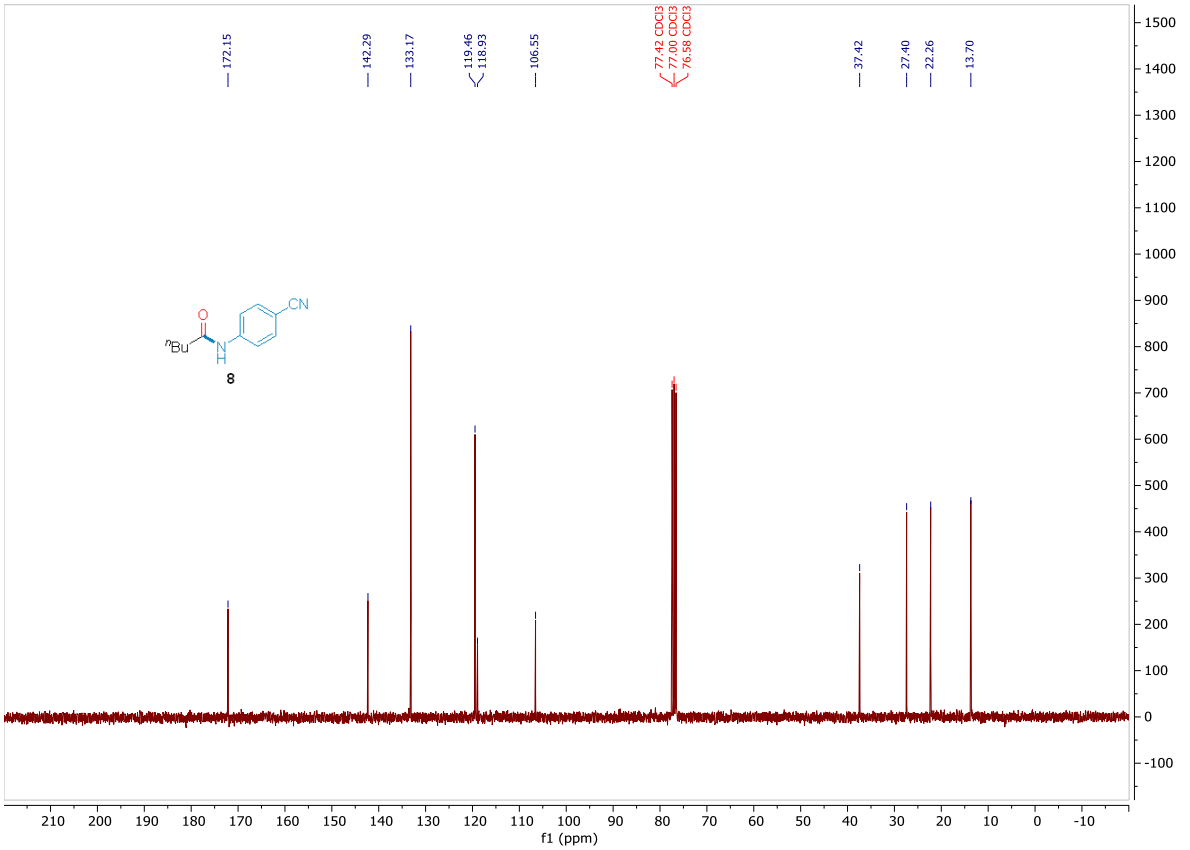
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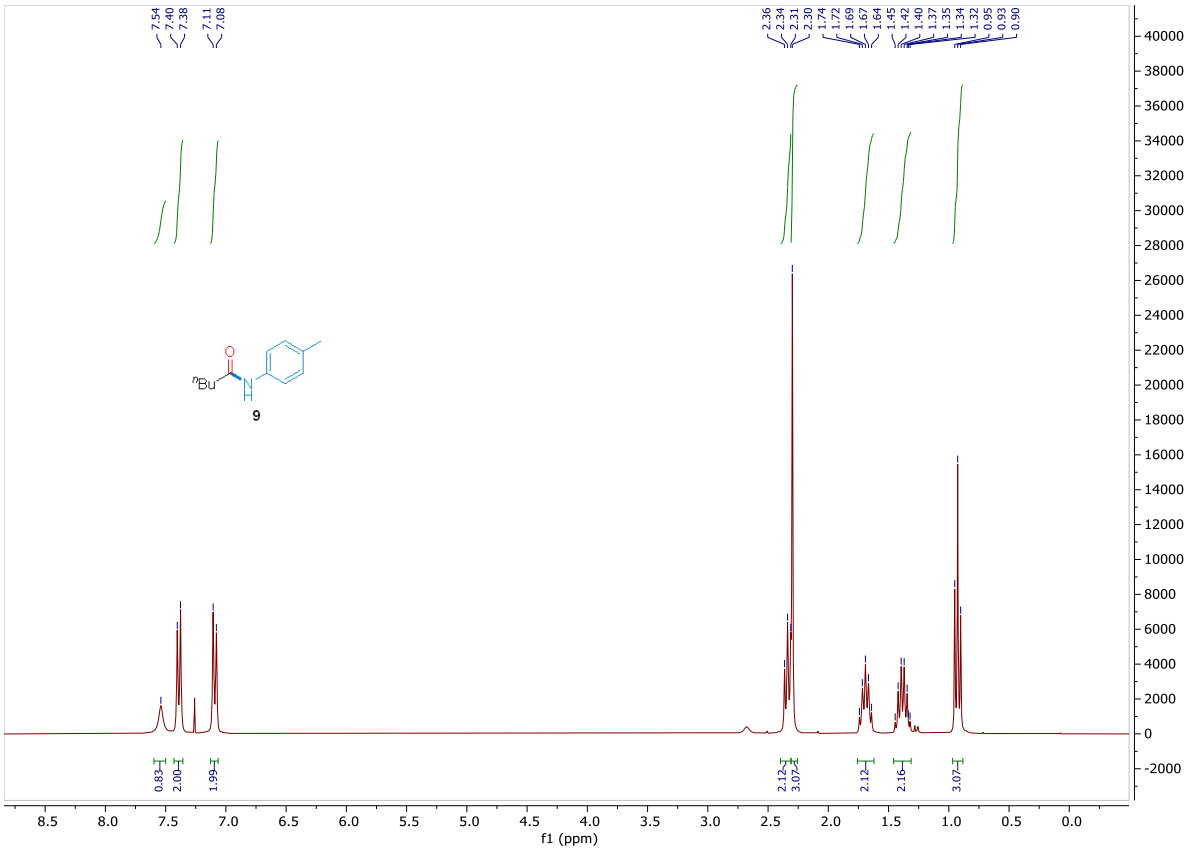
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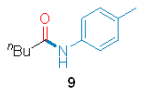
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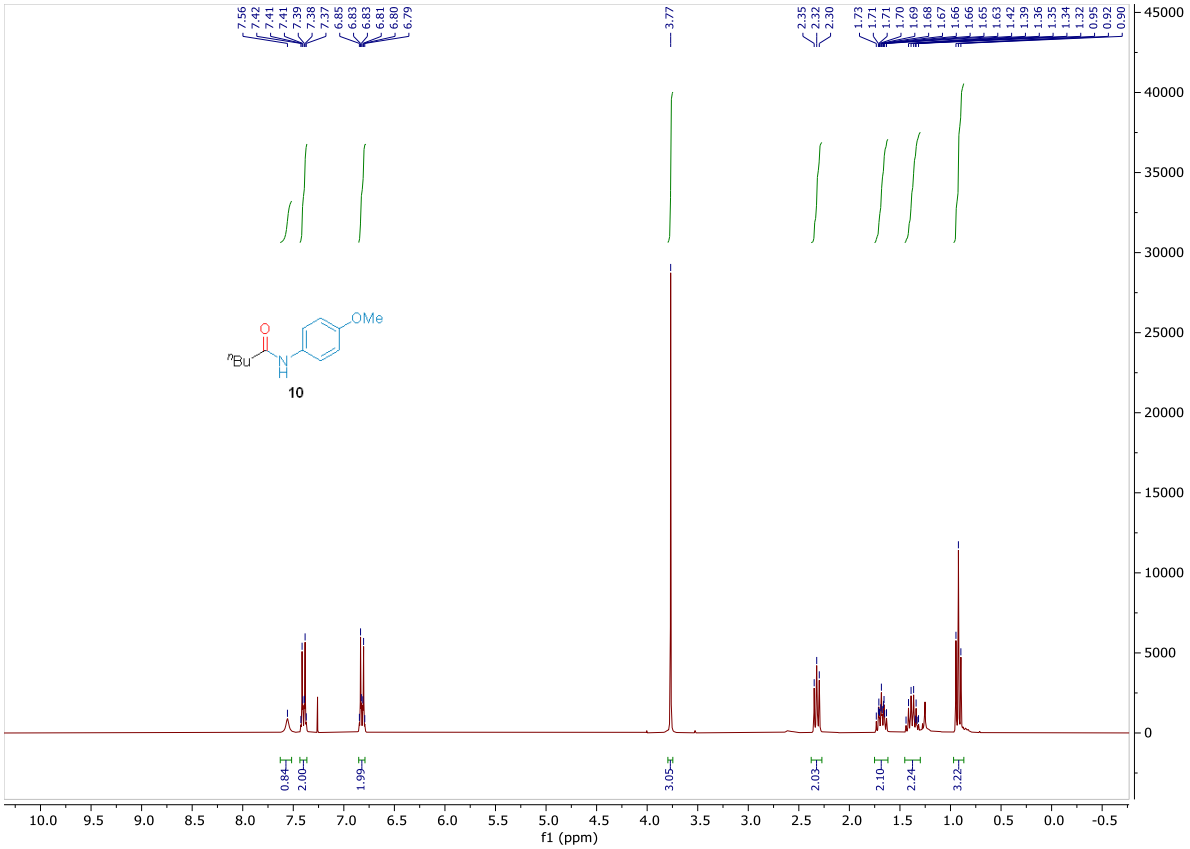
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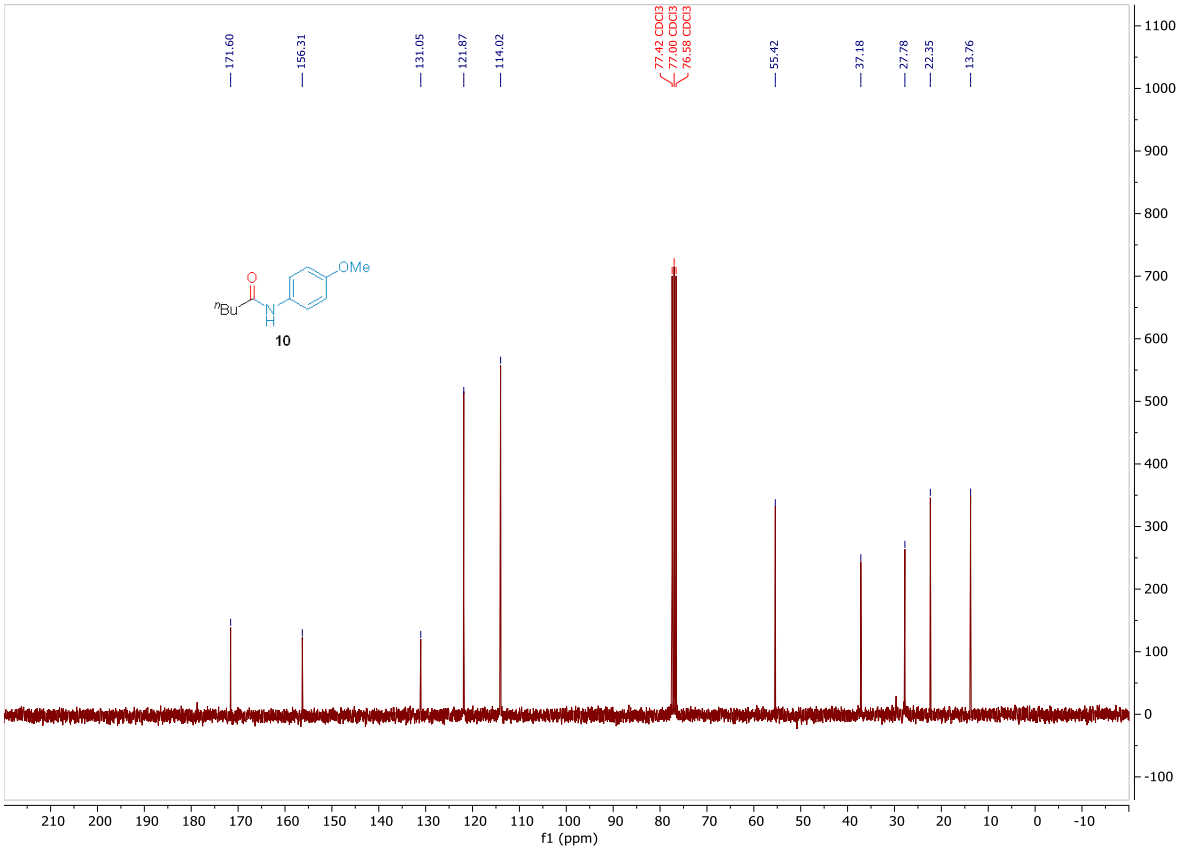
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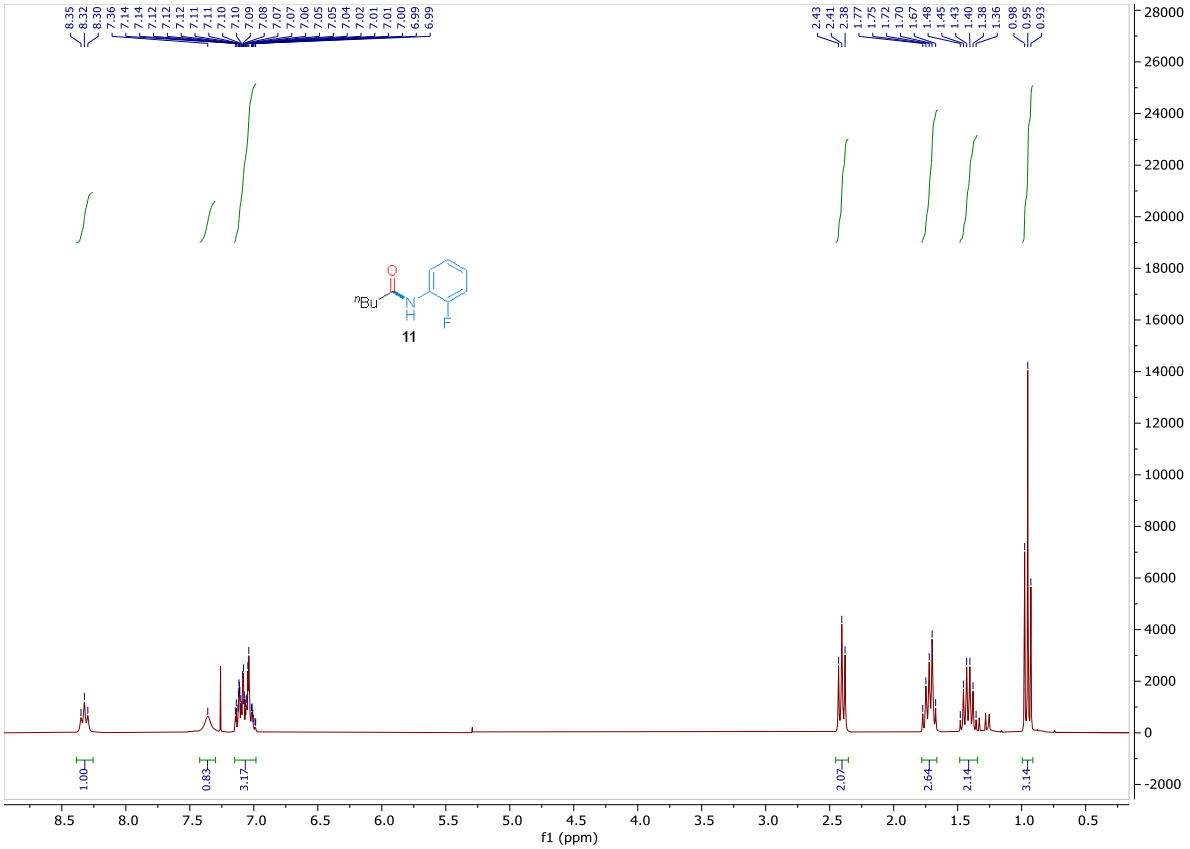
f1 (ppm)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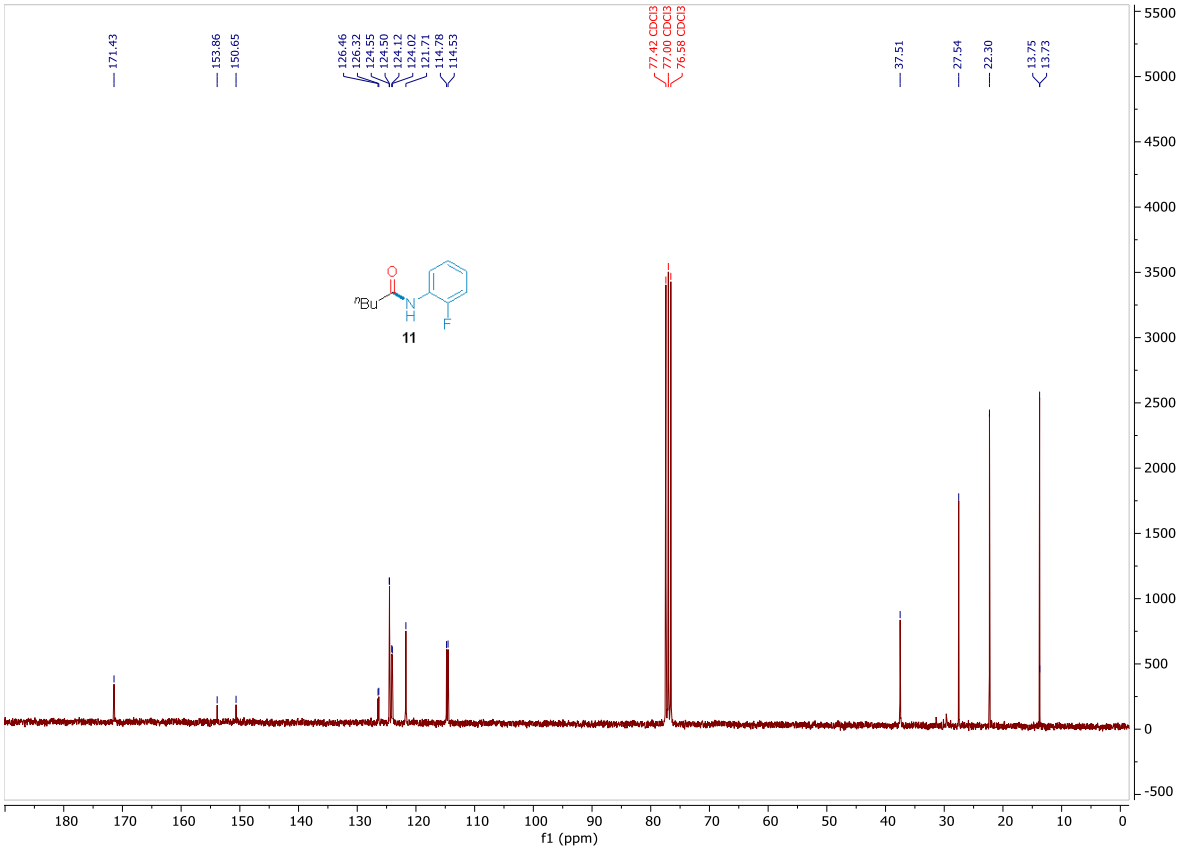
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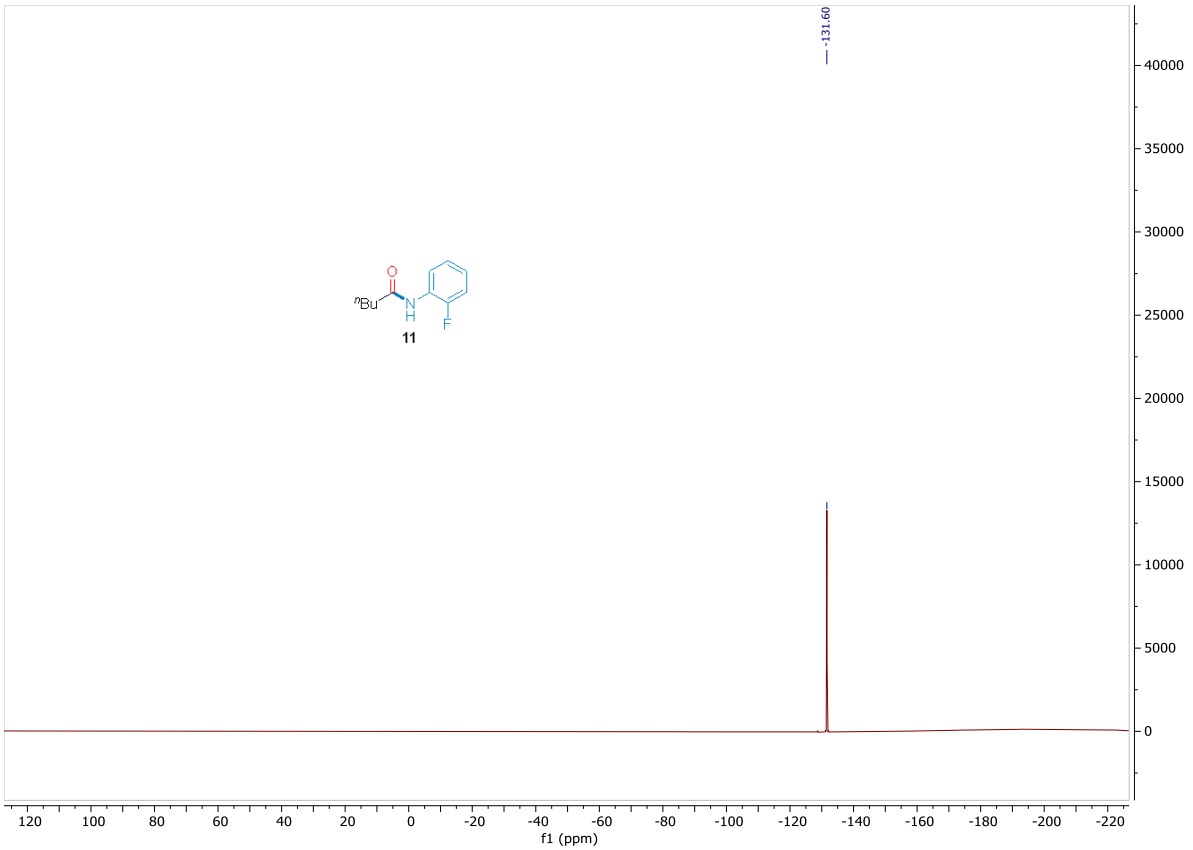
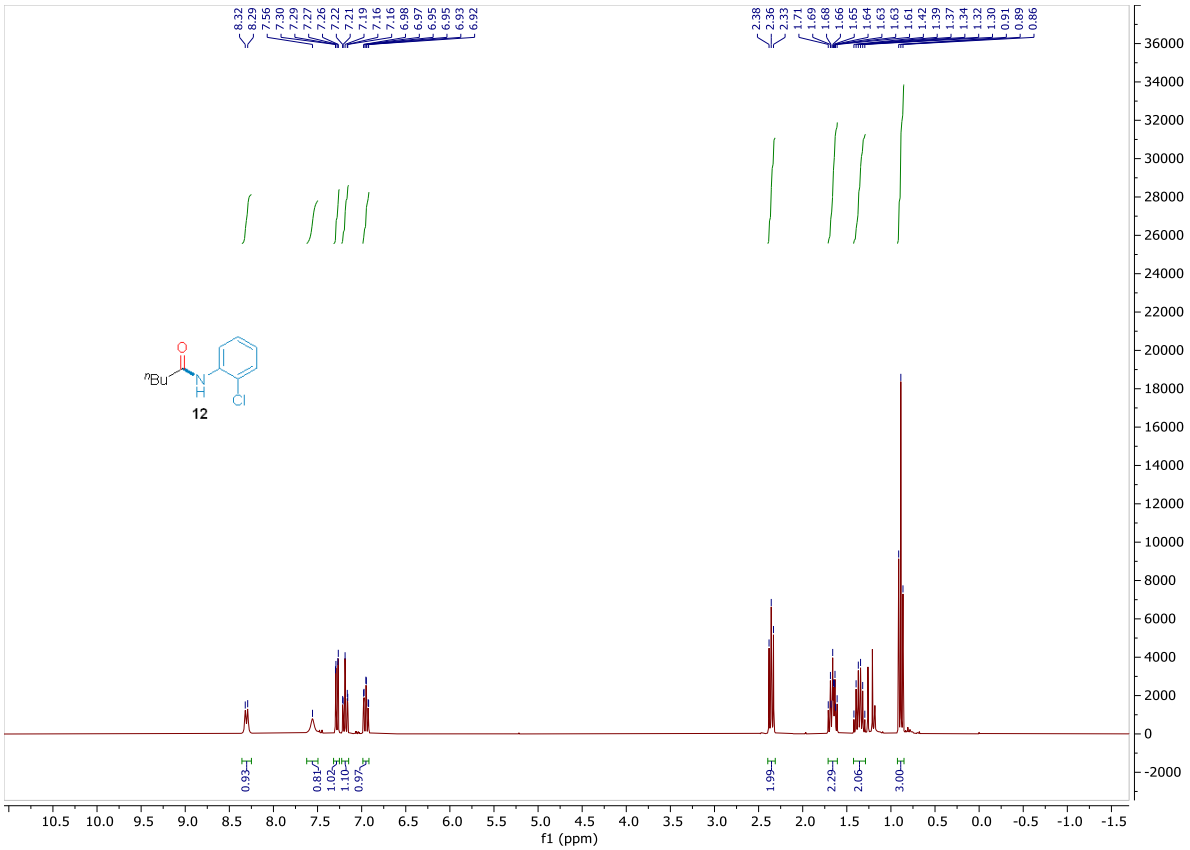
F
11
f1 (ppm)


-131.60
40000
35000
30000
25000
20000
15000
10000
5000
0
O
nBu
N
H
F
11
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
f1 (ppm)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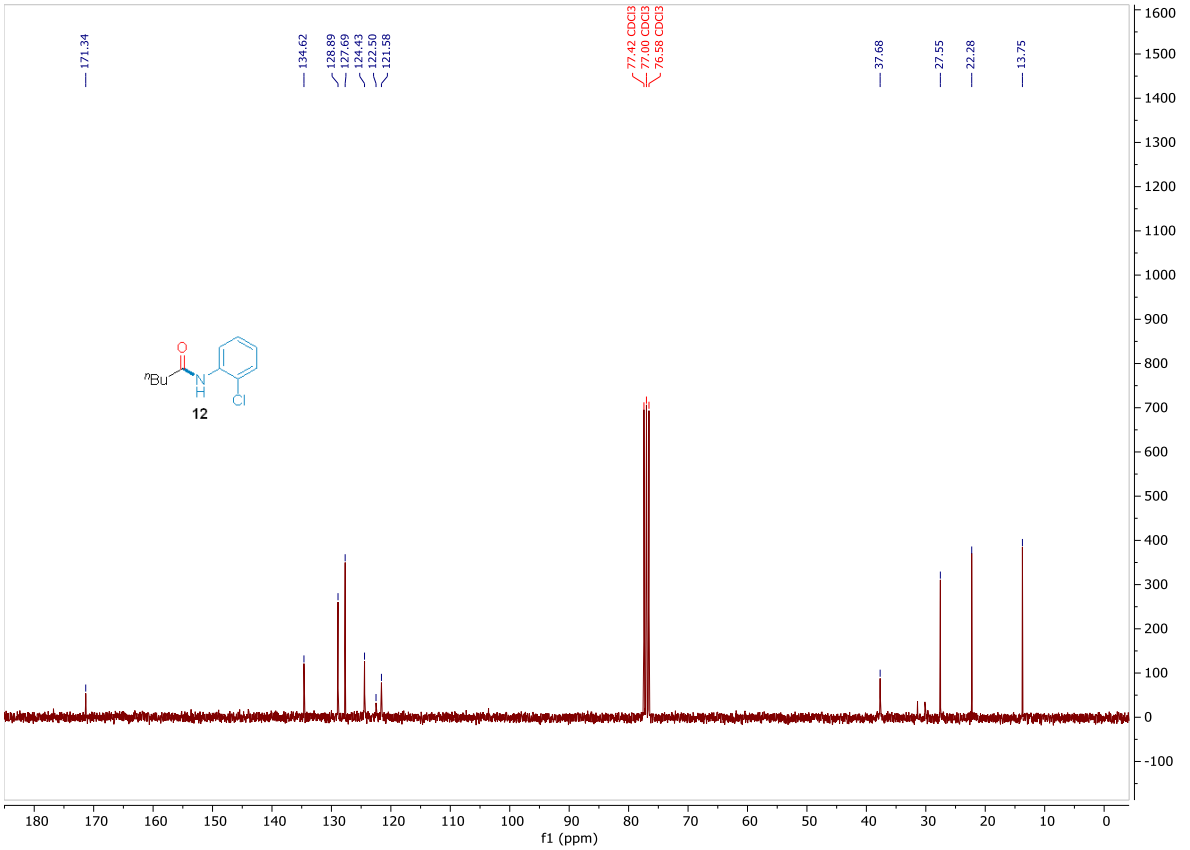
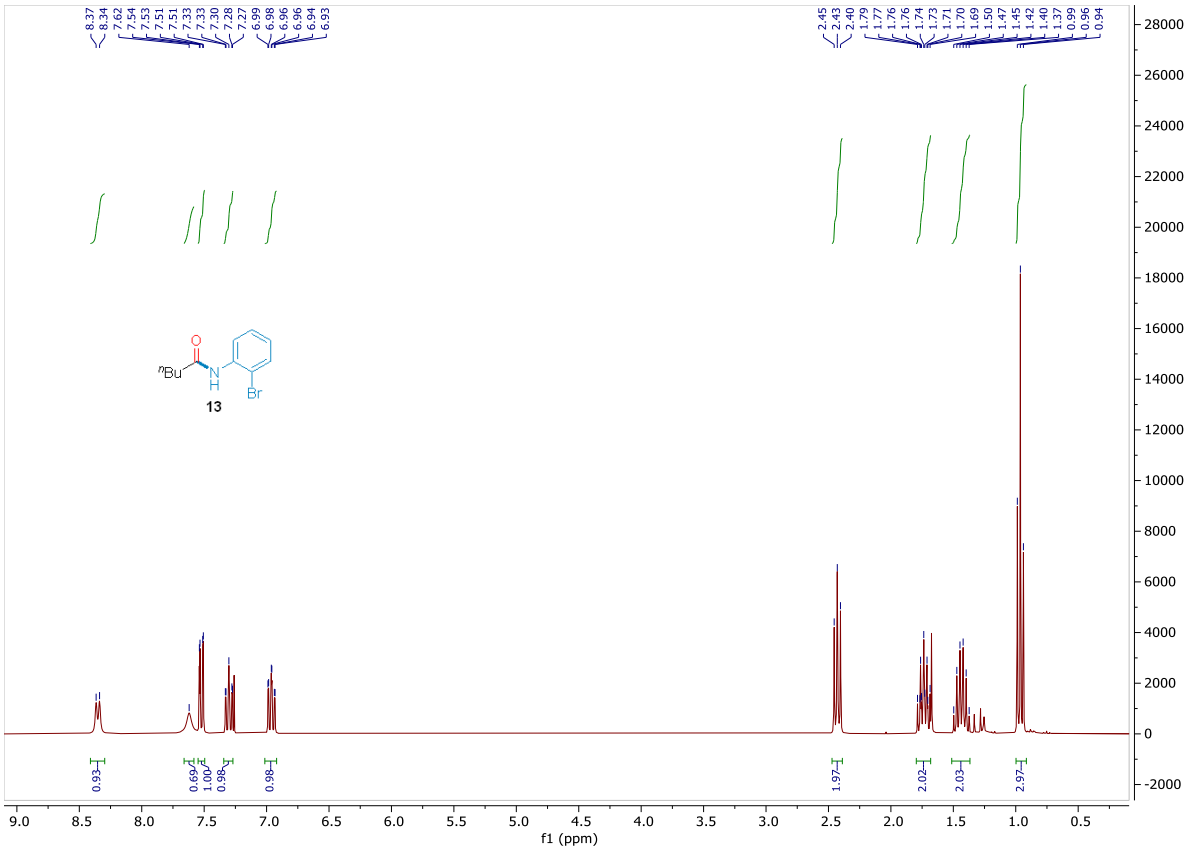
7.30
7.29
7.27
7.26
7.22
7.21
7.19
7.16
7.16
6.98
6.97
6.95
6.95
6.93
6.92
2.38
2.36
2.33
1.71
1.69
1.68
1.66
1.65
1.64
1.63
1.63
1.61
1.42
1.39
1.37
1.34
1.32
1.30
0.91
0.89
0.86
nBu
N
H
Cl
12
0.93
0.81
1.02
1.10
0.97
1.99
2.29
2.06
3.00
f1 (ppm)


171.34
134.62
128.89
127.69
124.43
122.50
121.58
77.42 CDCl3
77.00 CDCl3
76.58 CDCl3
37.68
27.55
22.28
13.75
nBu
N
H
Cl
12
f1 (ppm)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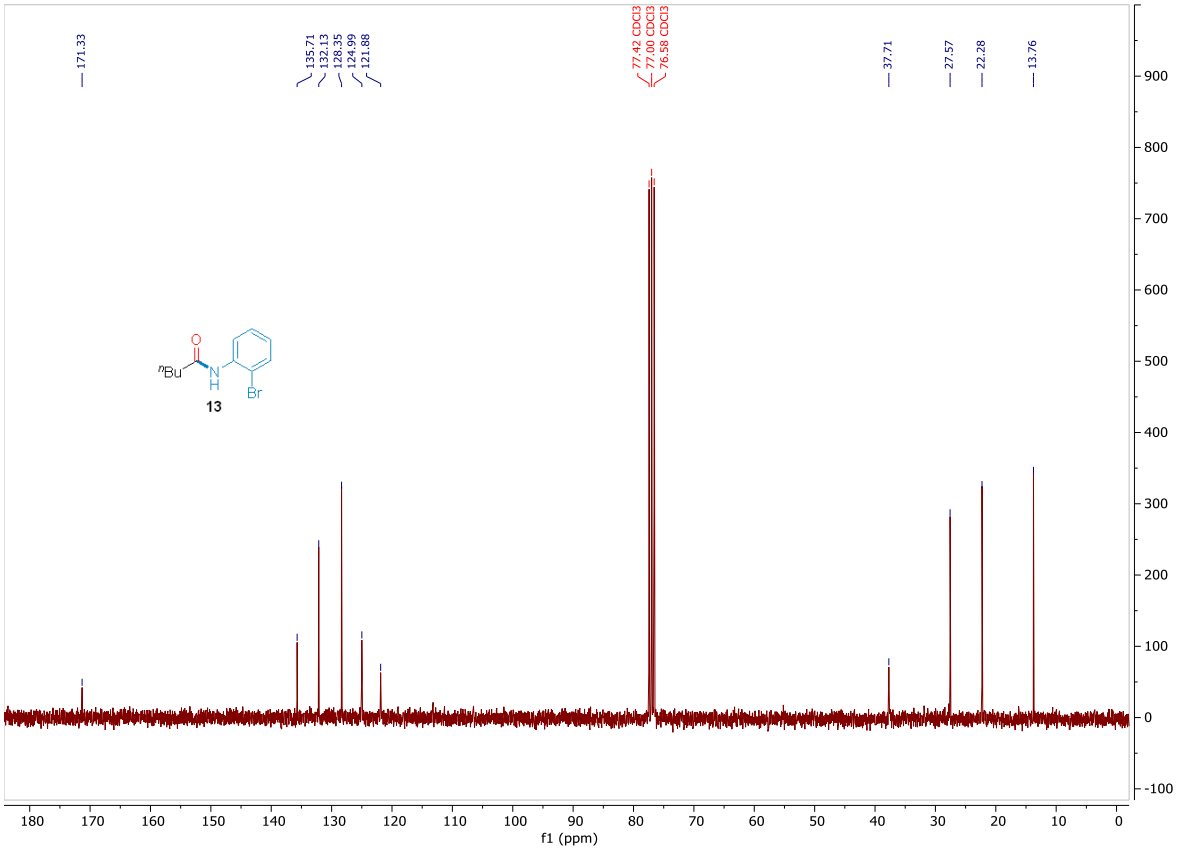
8.34
7.62
7.54
7.53
7.51
7.51
7.33
7.30
7.28
7.27
6.99
6.98
6.96
6.96
6.94
6.93
2.45
2.43
2.40
1.77
1.76
1.76
1.74
1.73
1.71
1.70
1.69
1.50
1.47
1.45
1.42
1.40
1.37
0.99
0.96
0.94
nBu
N
H
O
Br
13
0.93
0.69
1.00
0.98
0.98
1.97
2.02
2.03
2.97
f1 (ppm)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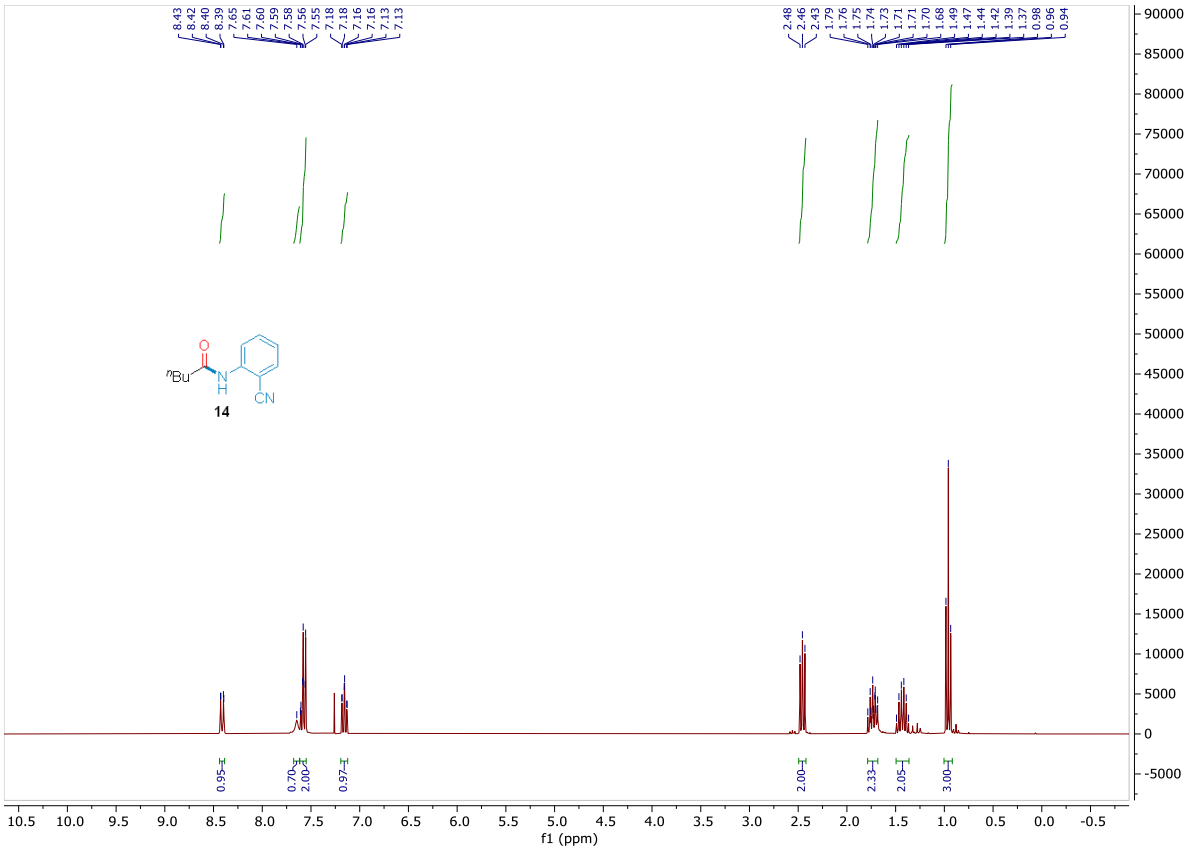
128.35
124.99
121.88
77.42 CDCl3
77.00 CDCl3
76.58 CDCl3
37.71
27.57
22.28
13.76
nBu
N
H
O
Br
13
f1 (ppm)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8.42
8.40
8.39
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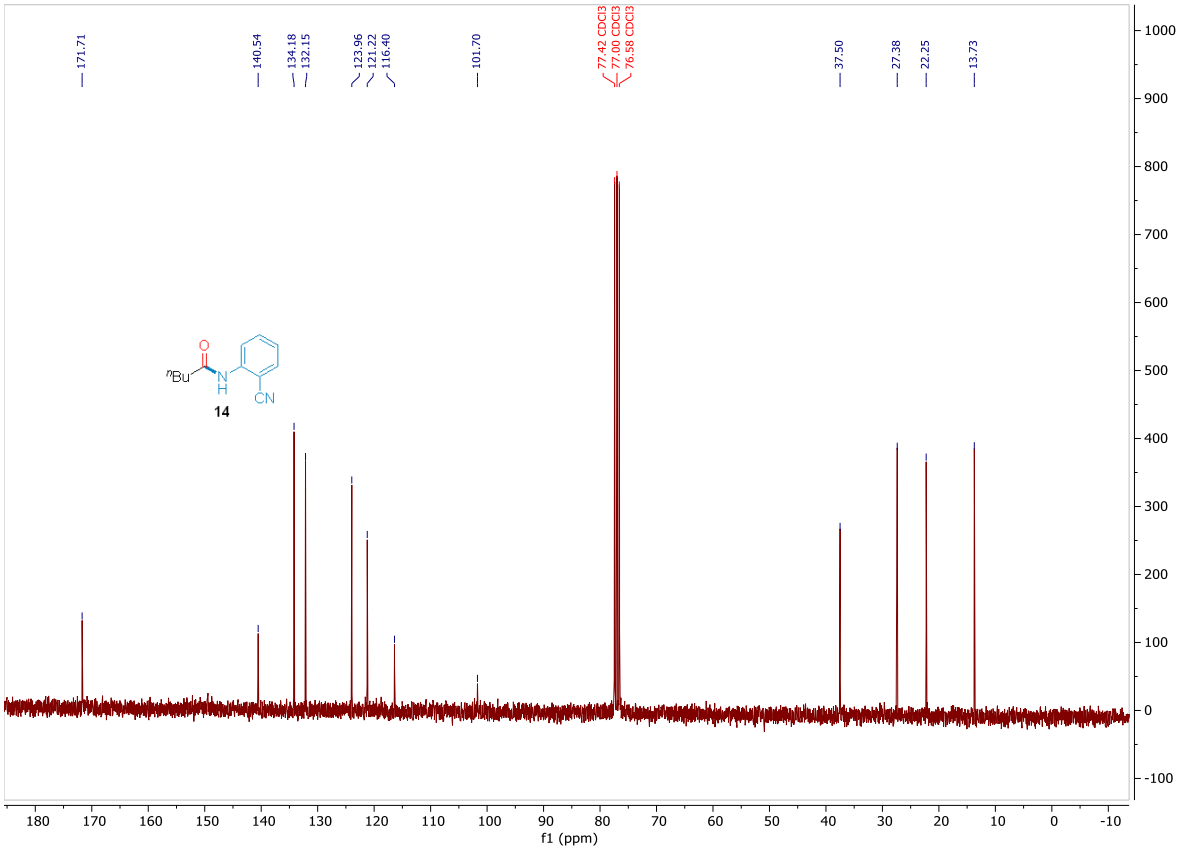
7.61
7.60
7.59
7.58
7.56
7.55
7.18
7.18
7.16
7.16
7.13
7.13
2.48
2.46
2.43
1.79
1.76
1.75
1.74
1.73
1.71
1.71
1.70
1.68
1.49
1.47
1.44
1.42
1.39
1.37
0.98
0.96
0.94
nBu
O
N
H
CN
14
0.95
0.70
2.00
0.97
2.00
2.33
2.05
3.00
f1 (ppm)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140.54
134.18
132.15
123.96
121.22
116.40
101.70
77.42 CDCl3
77.00 CDCl3
76.58 CDCl3
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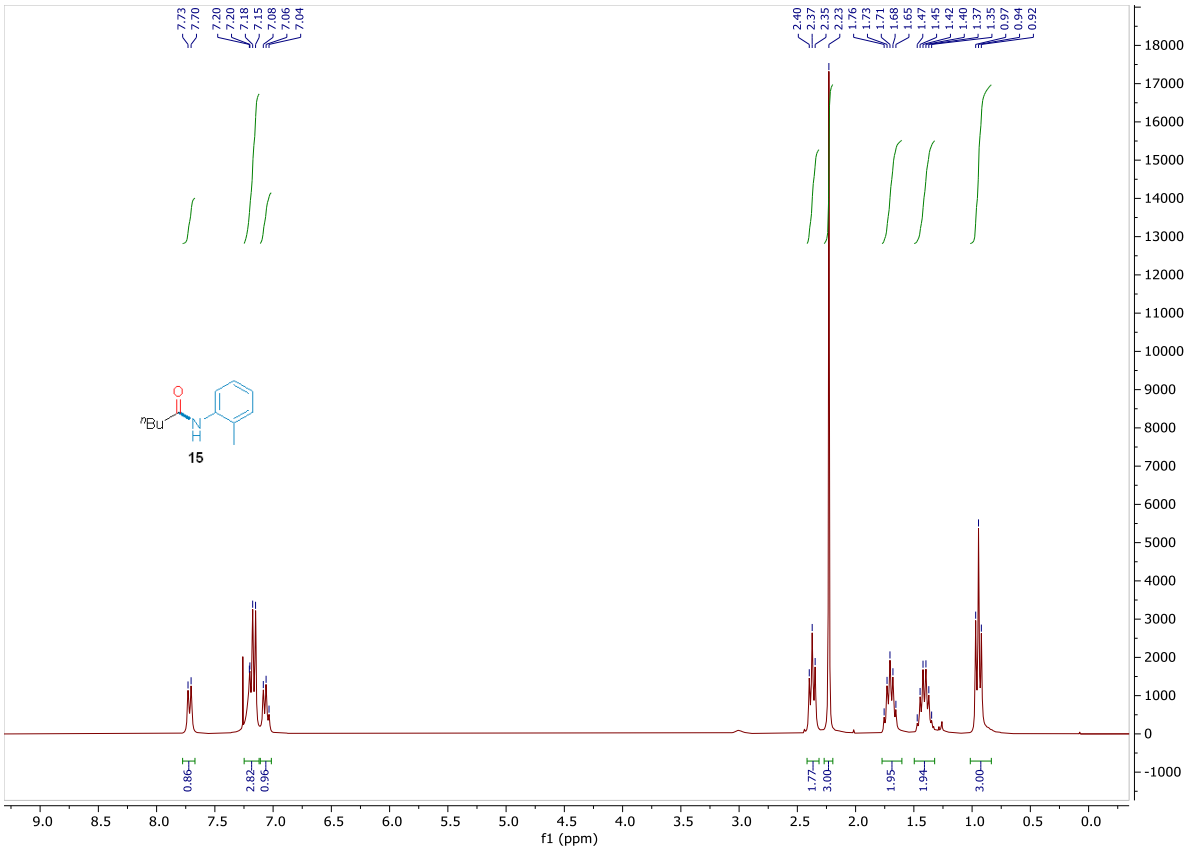
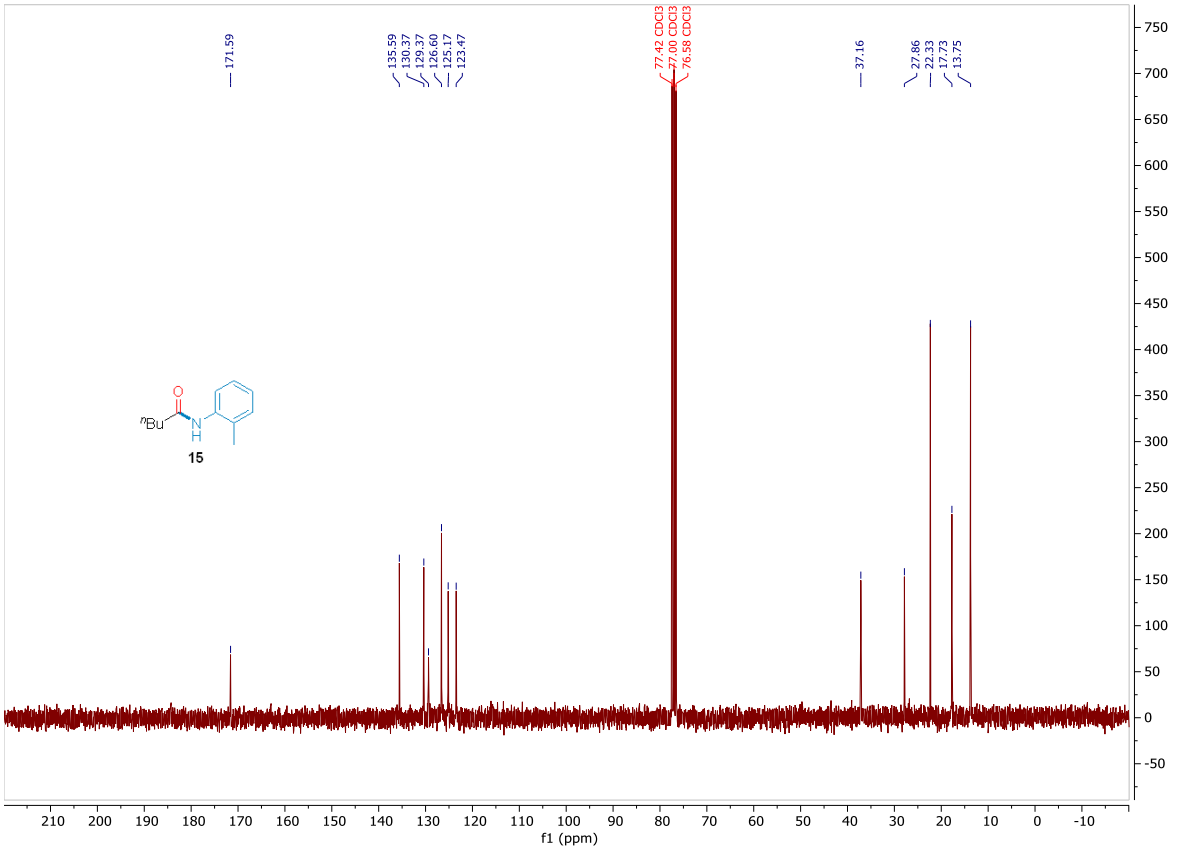
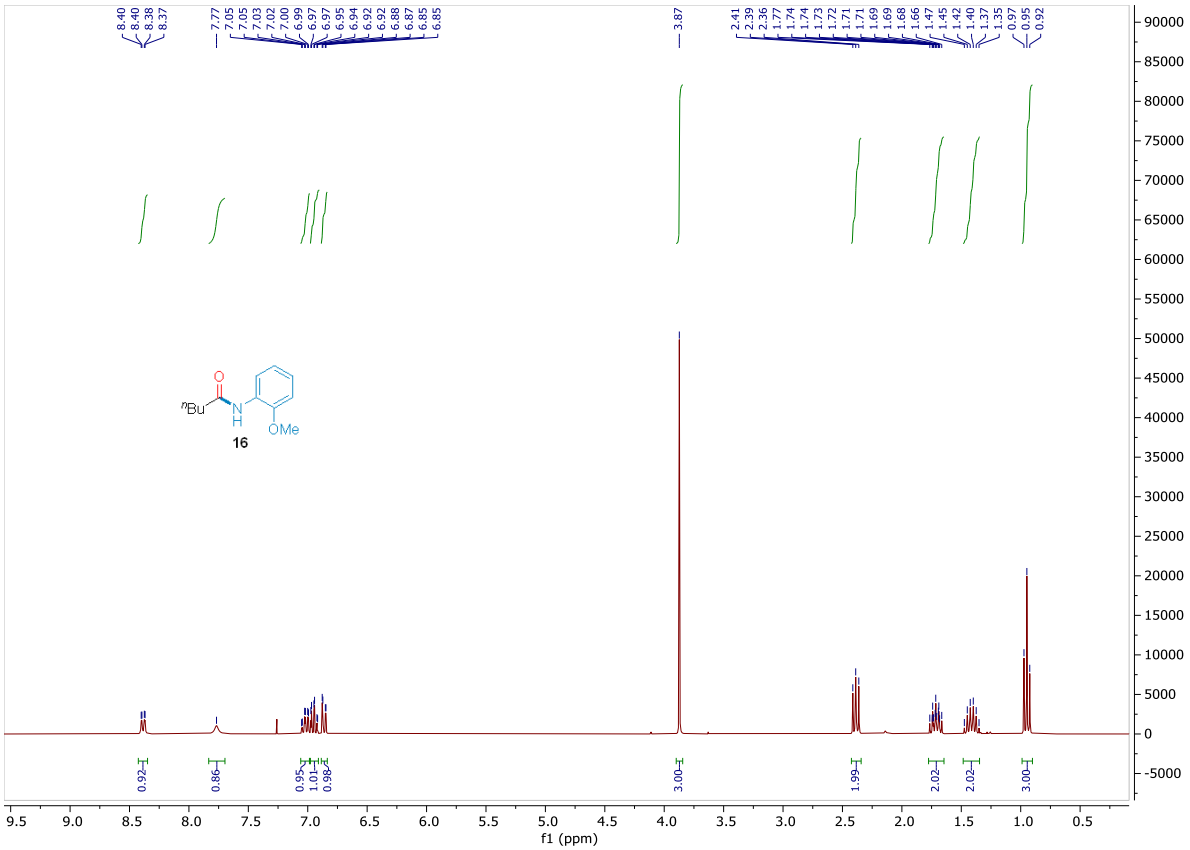
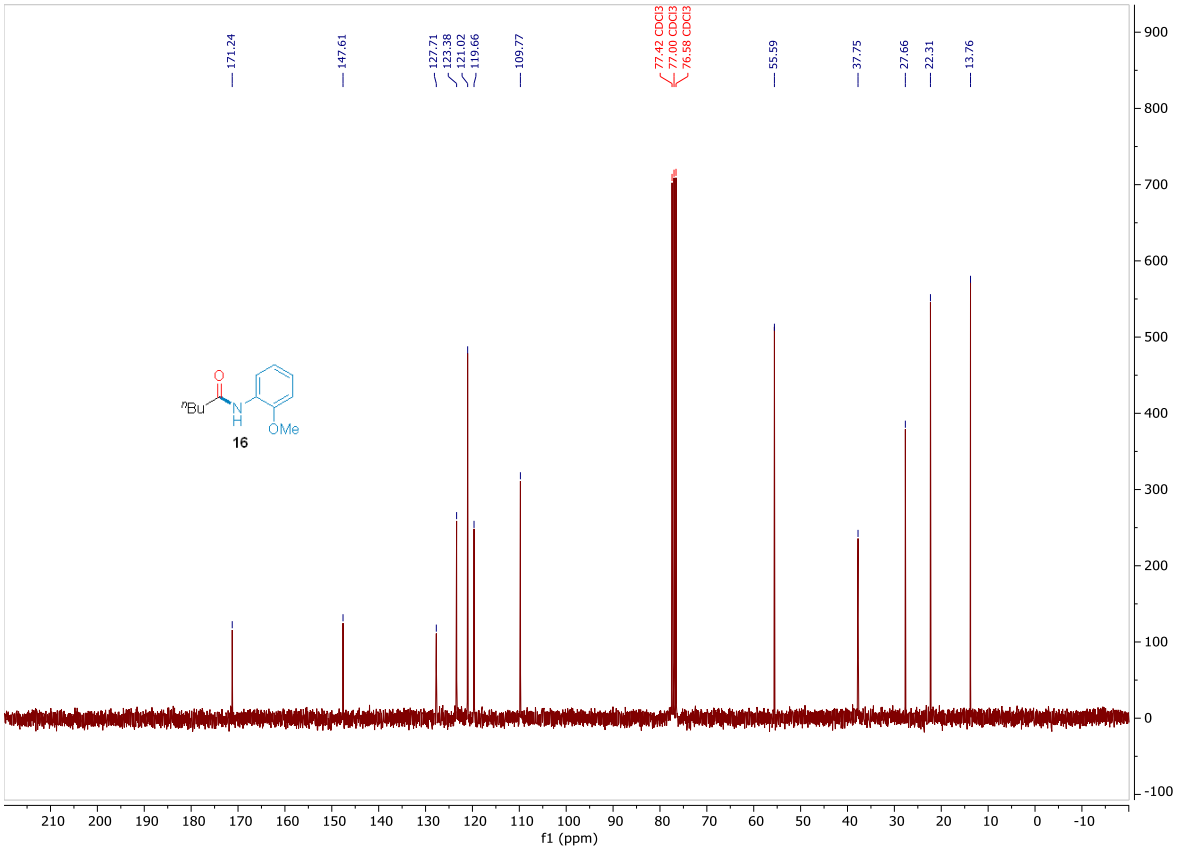
nBu
O
N
H
CN
14
f1 (ppm)


7.73
7.70
7.20
7.20
7.18
7.15
7.08
7.06
7.04
2.40
2.37
2.35
2.23
1.76
1.73
1.71
1.68
1.65
1.47
1.45
1.42
1.40
1.37
1.35
0.97
0.94
0.92
0.86
2.82
0.96
1.77
3.00
1.95
1.94
3.00
f1 (ppm)
15


171.59
135.59
130.37
129.37
126.60
125.17
123.47
77.42 CDCl3
77.00 CDCl3
76.58 CDCl3
37.16
27.86
22.33
17.73
13.75
f1 (ppm)
15


8.40
8.40
8.38
8.37
7.77
7.05
7.05
7.03
7.02
7.00
6.99
6.97
6.97
6.95
6.94
6.92
6.92
6.88
6.87
6.85
6.85
3.87
2.41
2.39
2.36
1.77
1.74
1.74
1.73
1.72
1.71
1.71
1.69
1.69
1.68
1.66
1.47
1.45
1.42
1.40
1.37
1.35
0.97
0.95
0.92
nBu
N
H
O
OMe
16
0.92
0.86
0.95
1.01
0.98
3.00
1.99
2.02
2.02
3.00
f1 (ppm)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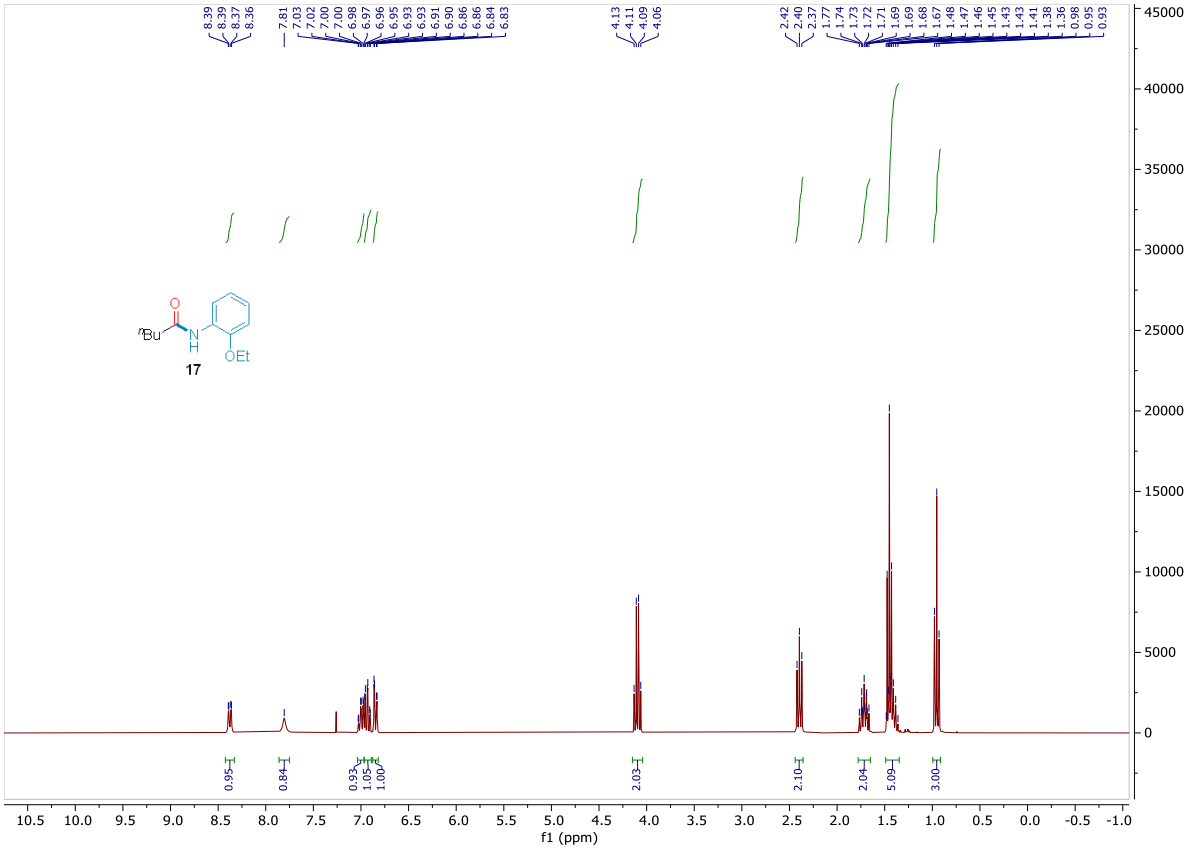
147.61
127.71
123.38
121.02
119.66
109.77
77.42 CDCl3
77.00 CDCl3
76.58 CDCl3
55.59
37.75
27.66
22.31
13.76
nBu
N
H
O
OMe
16
f1 (ppm)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8.39
8.37
8.36
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7.03
7.02
7.00
7.00
6.98
6.97
6.96
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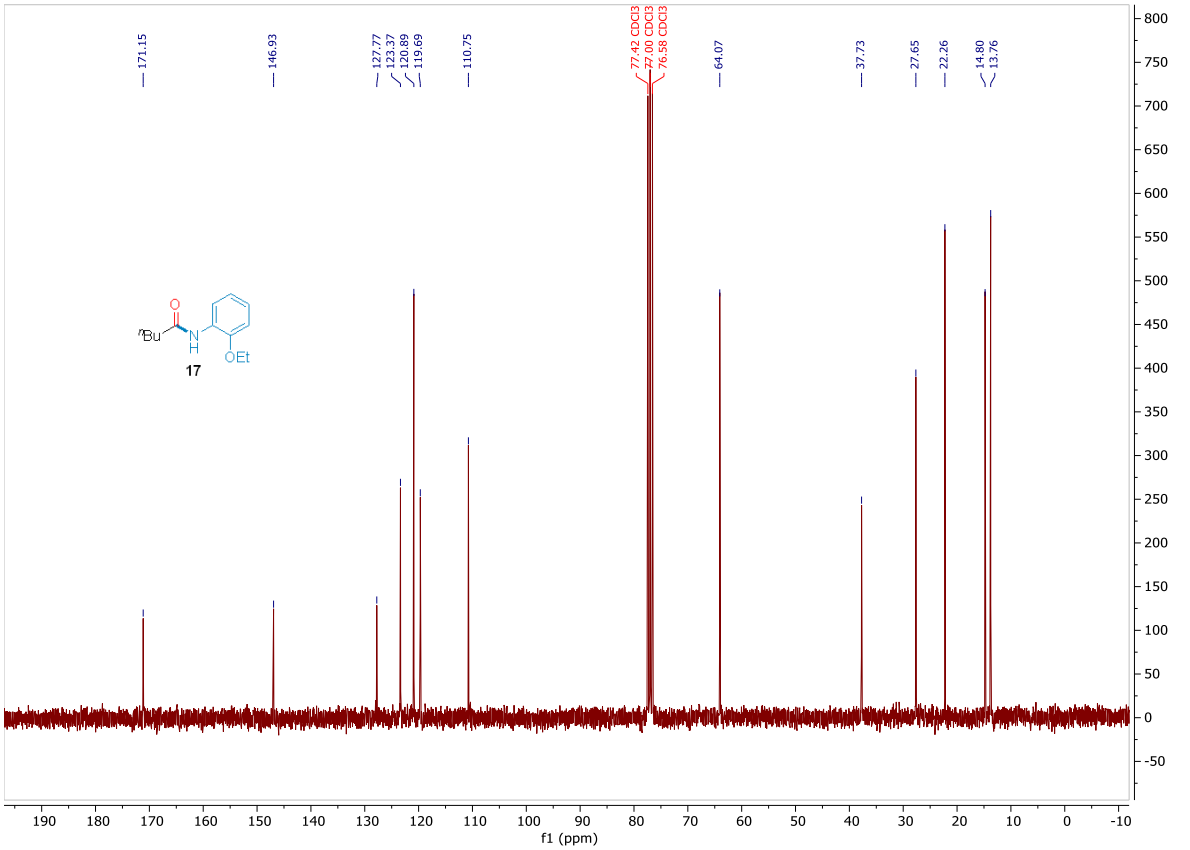
6.93
6.93
6.91
6.90
6.86
6.86
6.84
6.83
4.13
4.11
4.09
4.06
2.42
2.40
2.37
1.77
1.74
1.73
1.72
1.71
1.69
1.69
1.68
1.67
1.48
1.47
1.46
1.45
1.43
1.43
1.41
1.38
1.36
1.08
0.98
0.95
0.93
0.95
0.84
0.93
1.05
1.00
2.03
2.10
2.04
5.09
3.00
nBu
O
N
H
OEt
17
f1 (ppm)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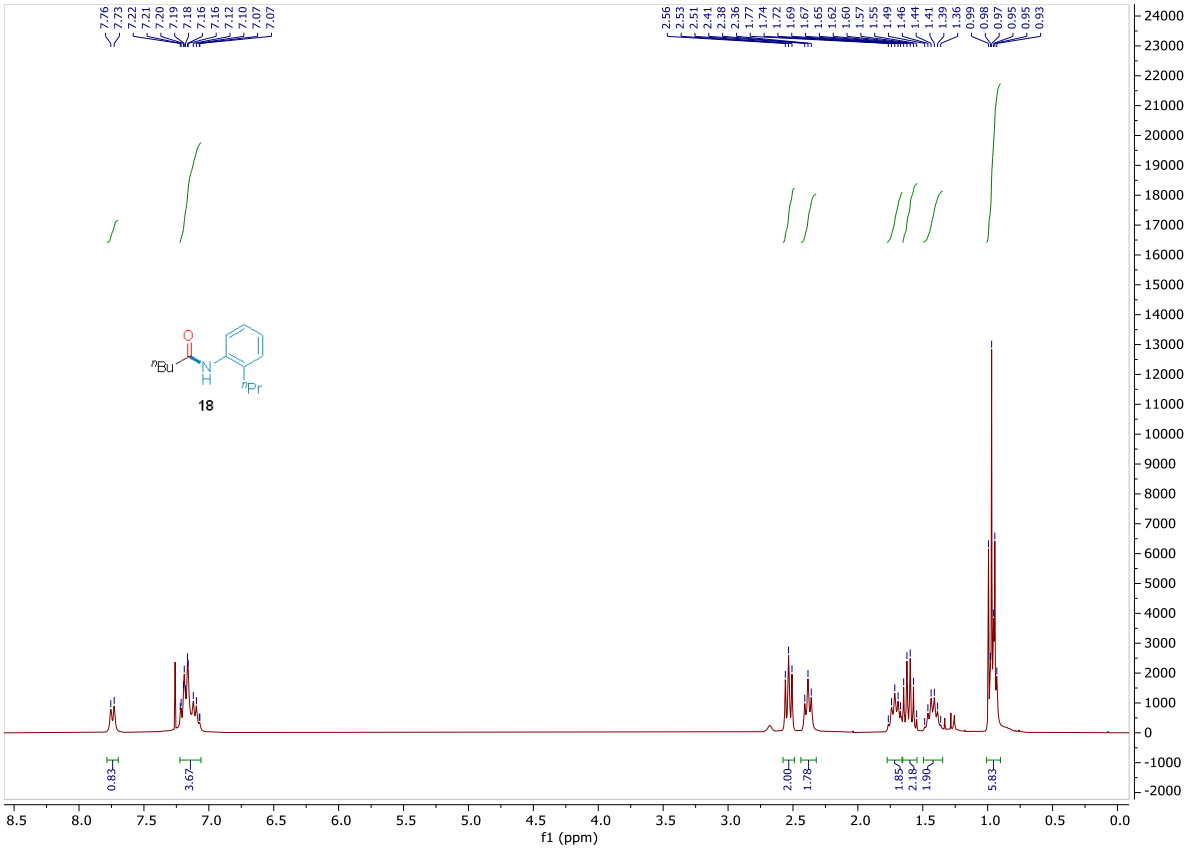
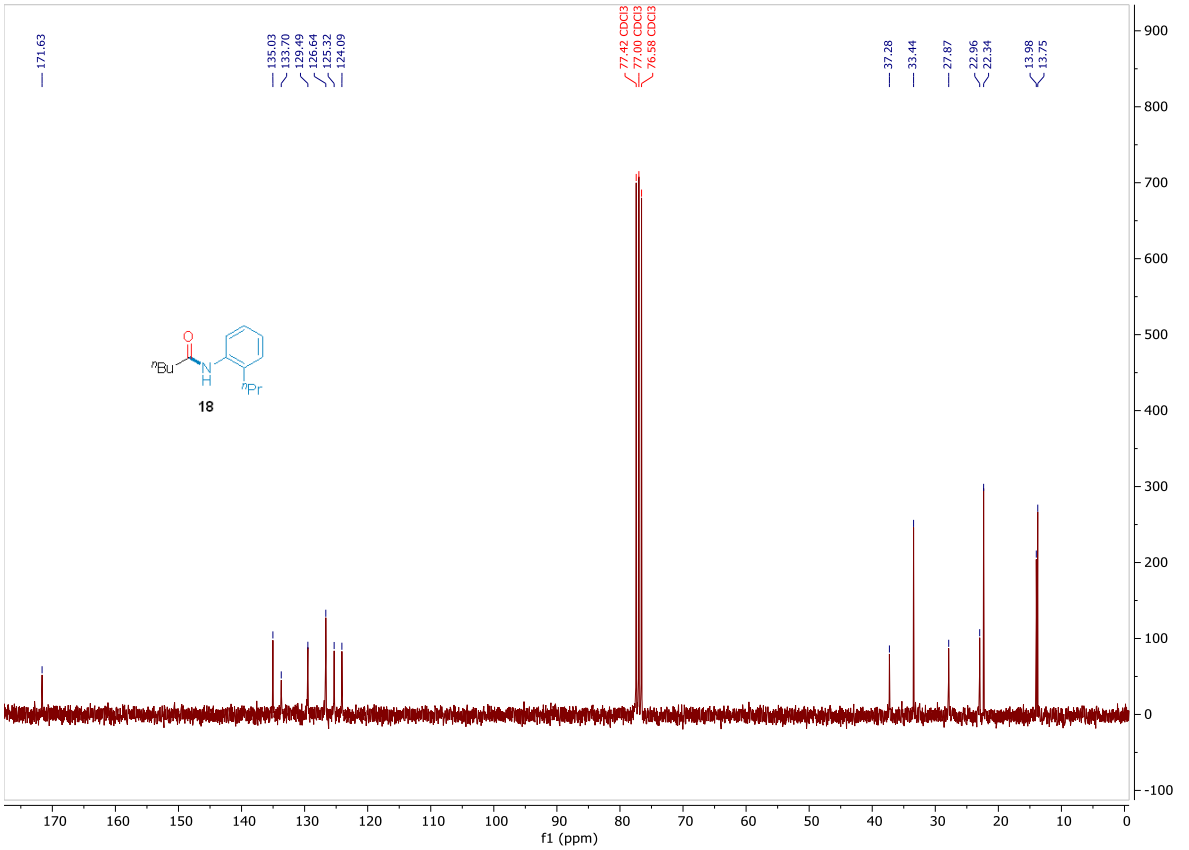
146.93
127.77
123.37
120.89
119.69
110.75
77.42 CDCl3
77.00 CDCl3
76.58 CDCl3
64.07
37.73
27.65
22.26
14.80
13.76
nBu
O
N
H
OEt
17
f1 (ppm)


7.76
7.73
7.22
7.21
7.20
7.19
7.18
7.16
7.16
7.12
7.10
7.07
7.07
2.56
2.53
2.51
2.41
2.38
2.36
1.77
1.74
1.72
1.69
1.67
1.65
1.62
1.60
1.57
1.55
1.49
1.46
1.44
1.41
1.39
1.36
0.99
0.98
0.97
0.95
0.95
0.93
nBu
N
H
nPr
18
0.83
3.67
2.00
1.78
1.85
2.18
1.90
5.83
f1 (ppm)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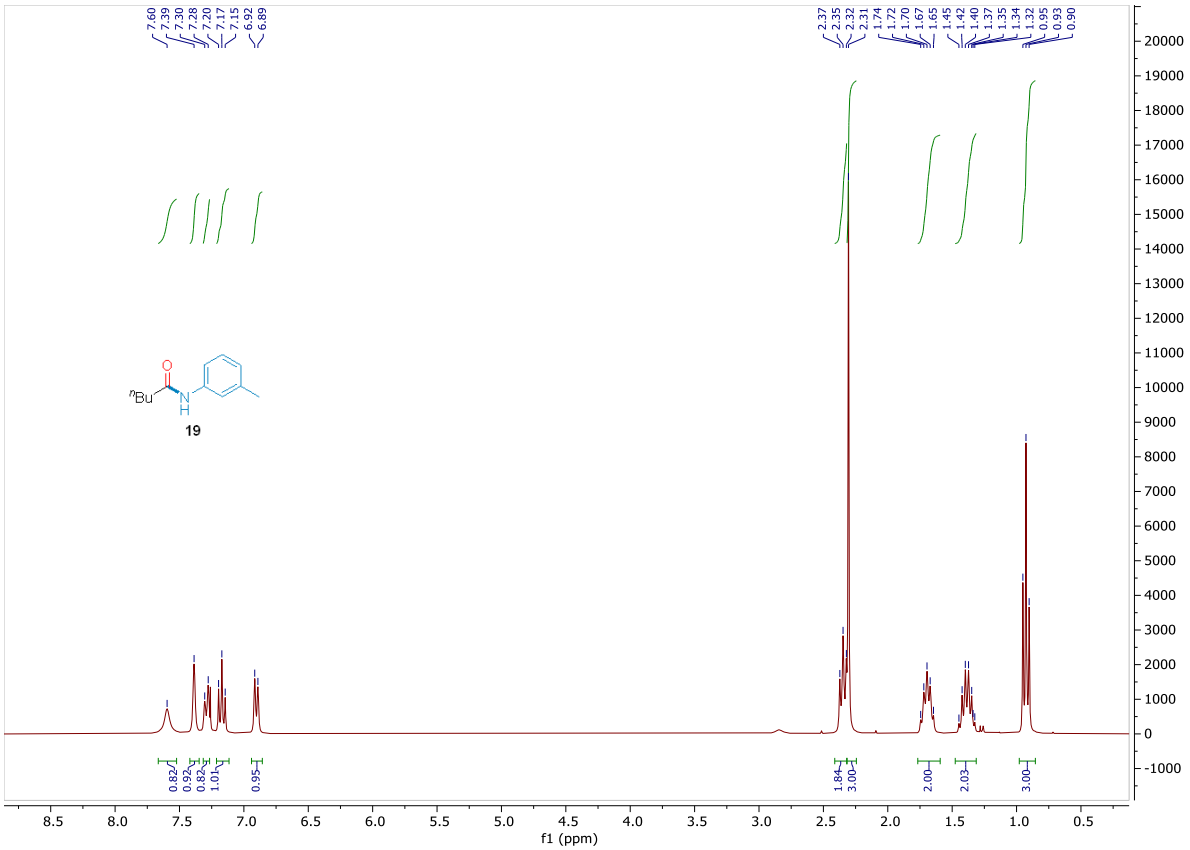
135.03
133.70
129.49
126.64
125.32
124.09
77.42 CDCl3
77.00 CDCl3
76.58 CDCl3
37.28
33.44
27.87
22.96
22.34
13.98
13.75
nBu
N
H
nPr
18
f1 (ppm)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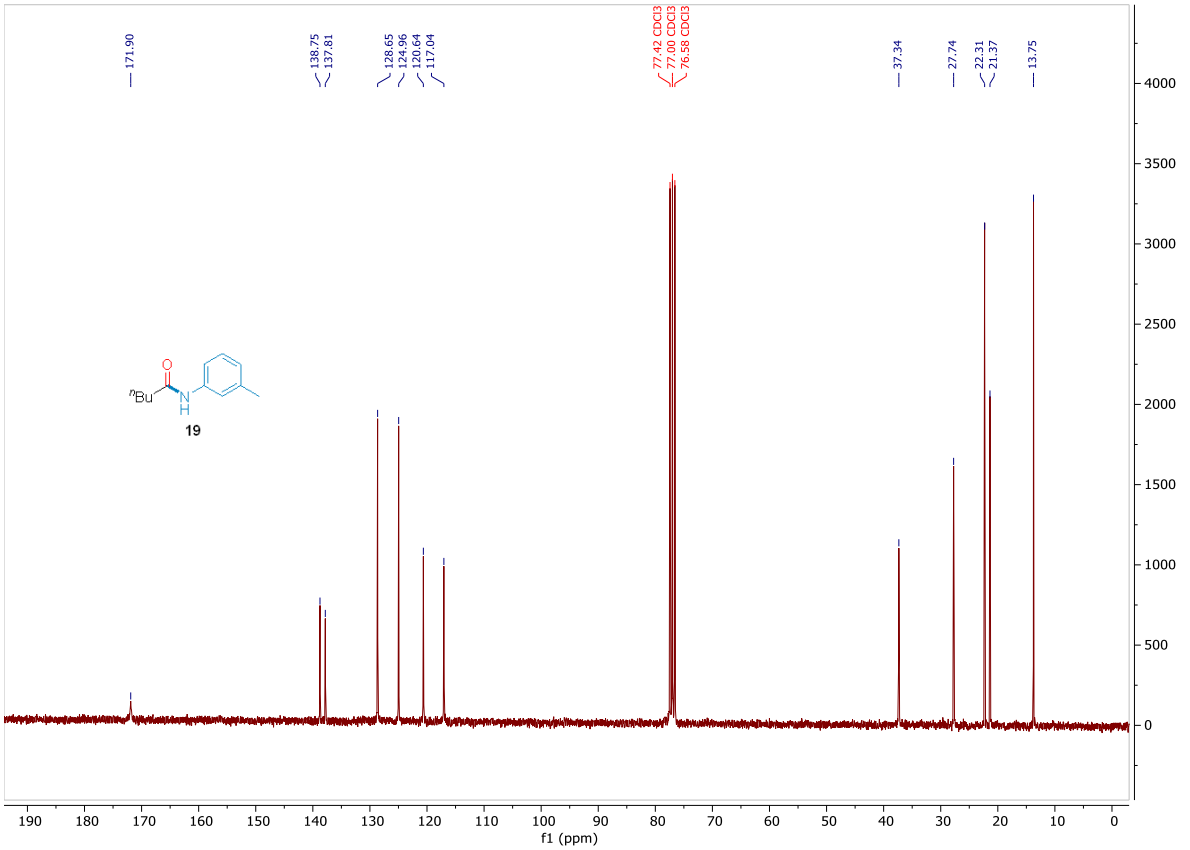
7.39
7.30
7.28
7.20
7.17
7.15
6.92
6.89
2.37
2.35
2.32
2.31
1.74
1.72
1.70
1.67
1.65
1.45
1.42
1.40
1.37
1.35
1.34
1.32
0.95
0.93
0.90
0.82
0.92
0.82
1.01
0.95
1.84
3.00
2.00
2.03
3.00
f1 (ppm)
19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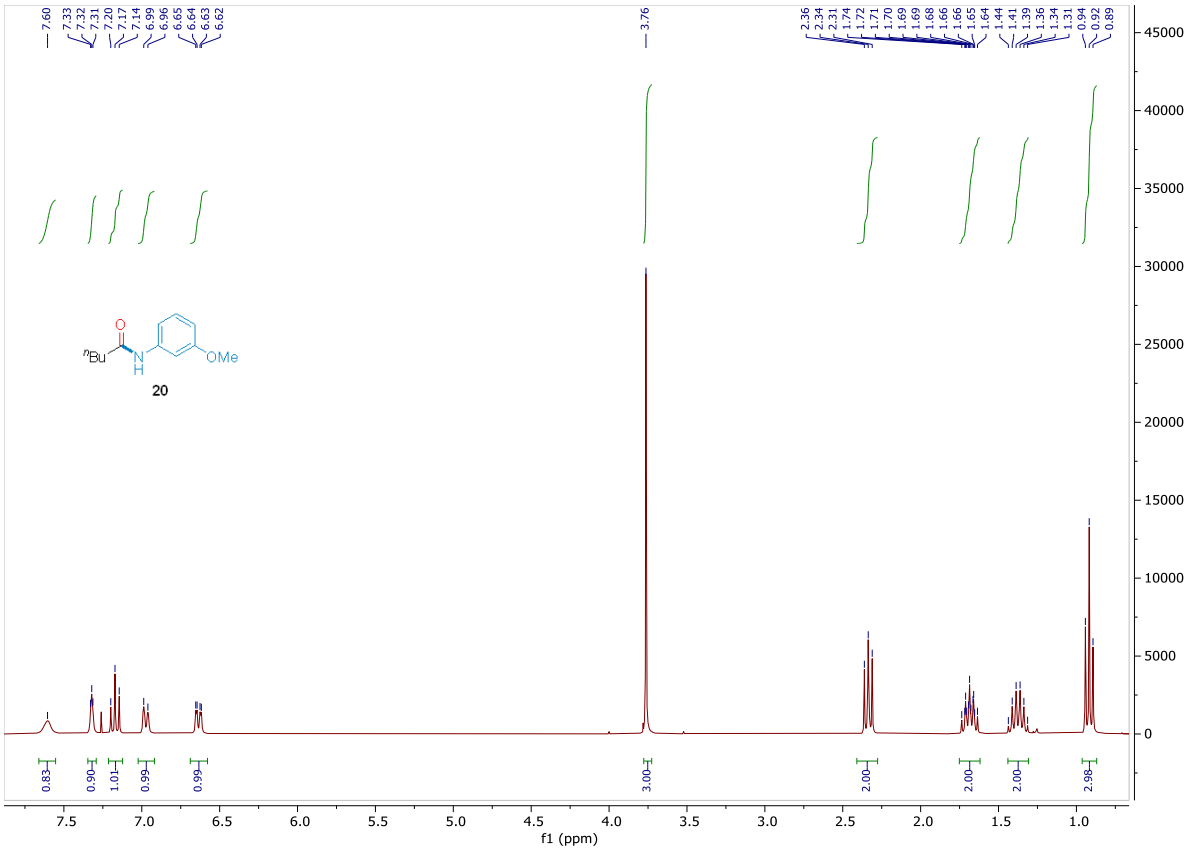
137.81
128.65
124.96
120.64
117.04
77.42 CDCl3
77.00 CDCl3
76.58 CDCl3
37.34
27.74
22.31
21.37
13.75
f1 (ppm)
19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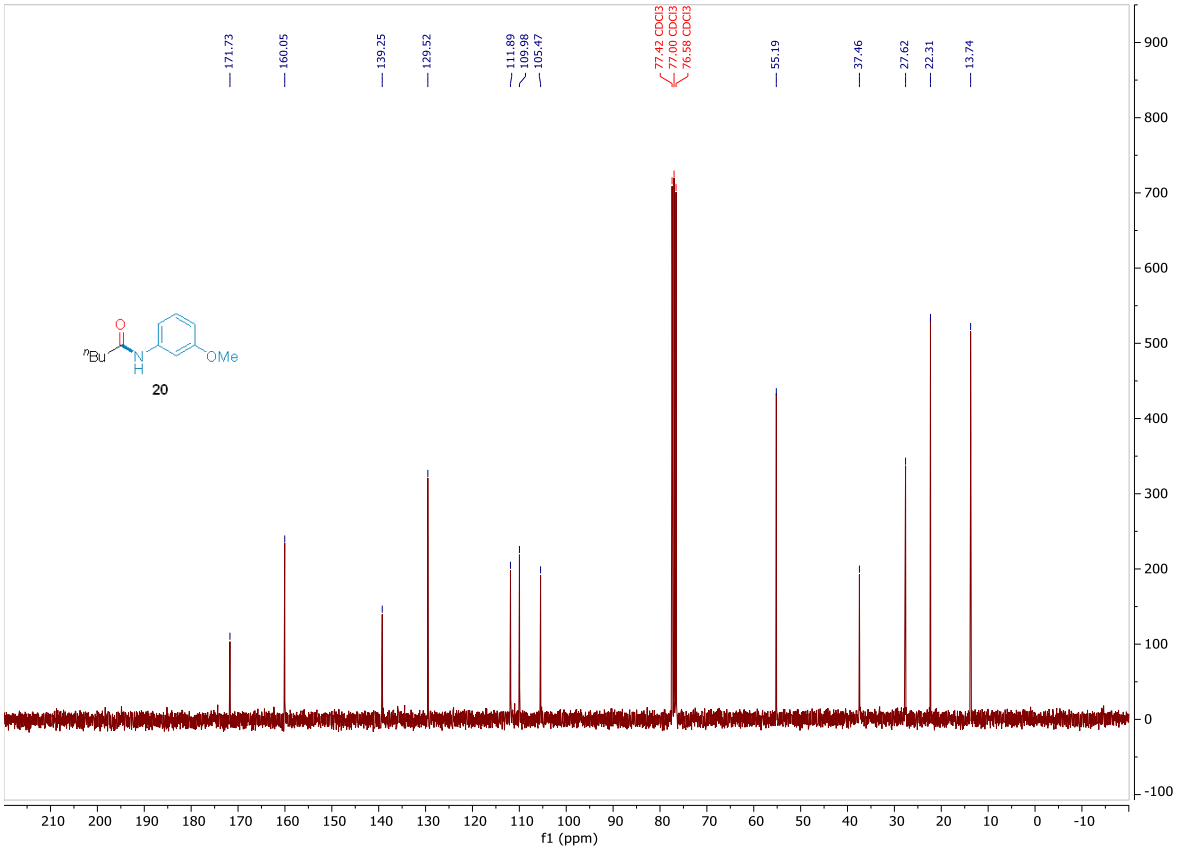
7.33
7.32
7.31
7.20
7.17
7.14
6.99
6.96
6.65
6.64
6.63
6.62
3.76
2.36
2.34
2.31
1.74
1.72
1.71
1.70
1.69
1.69
1.68
1.66
1.66
1.65
1.64
1.44
1.41
1.39
1.36
1.34
1.31
0.94
0.92
0.89
nBu
N
H
OMe
20
0.83
0.90
1.01
0.99
0.99
3.00
2.00
2.00
2.00
2.98
f1 (ppm)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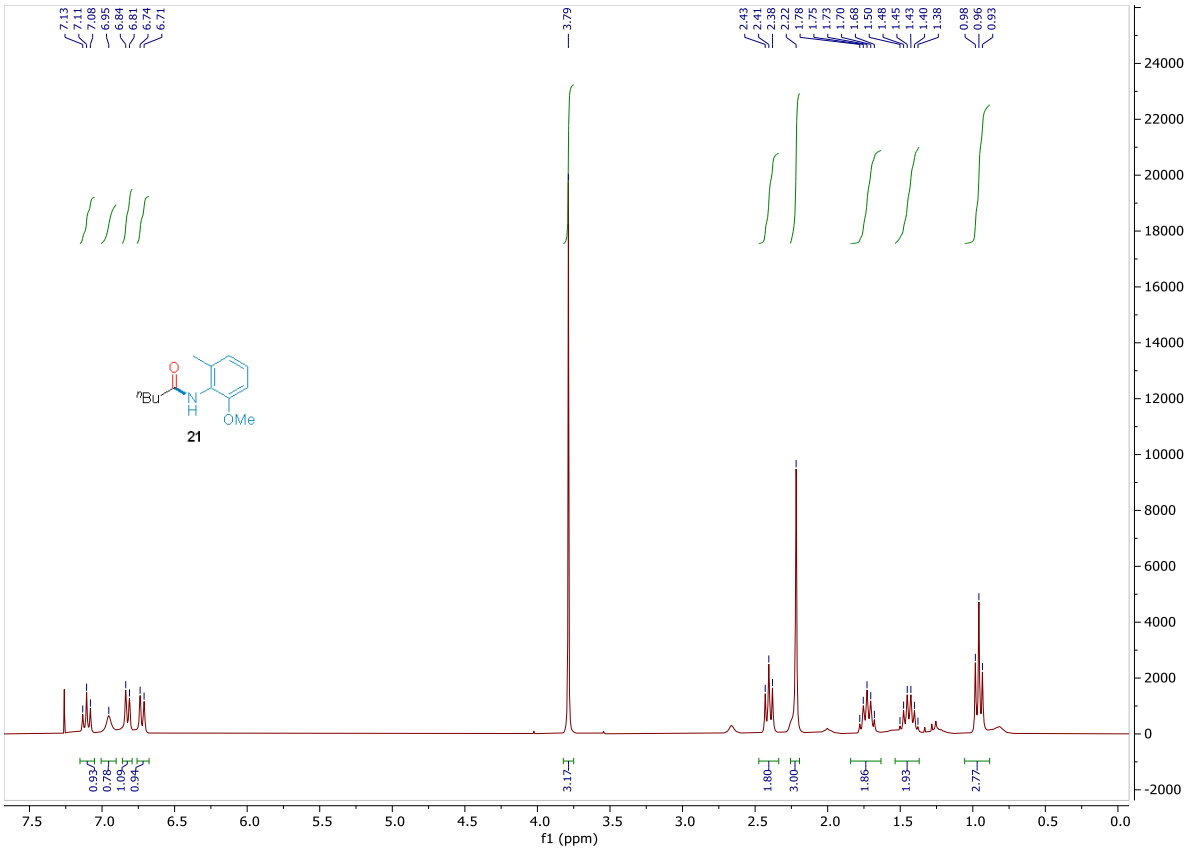
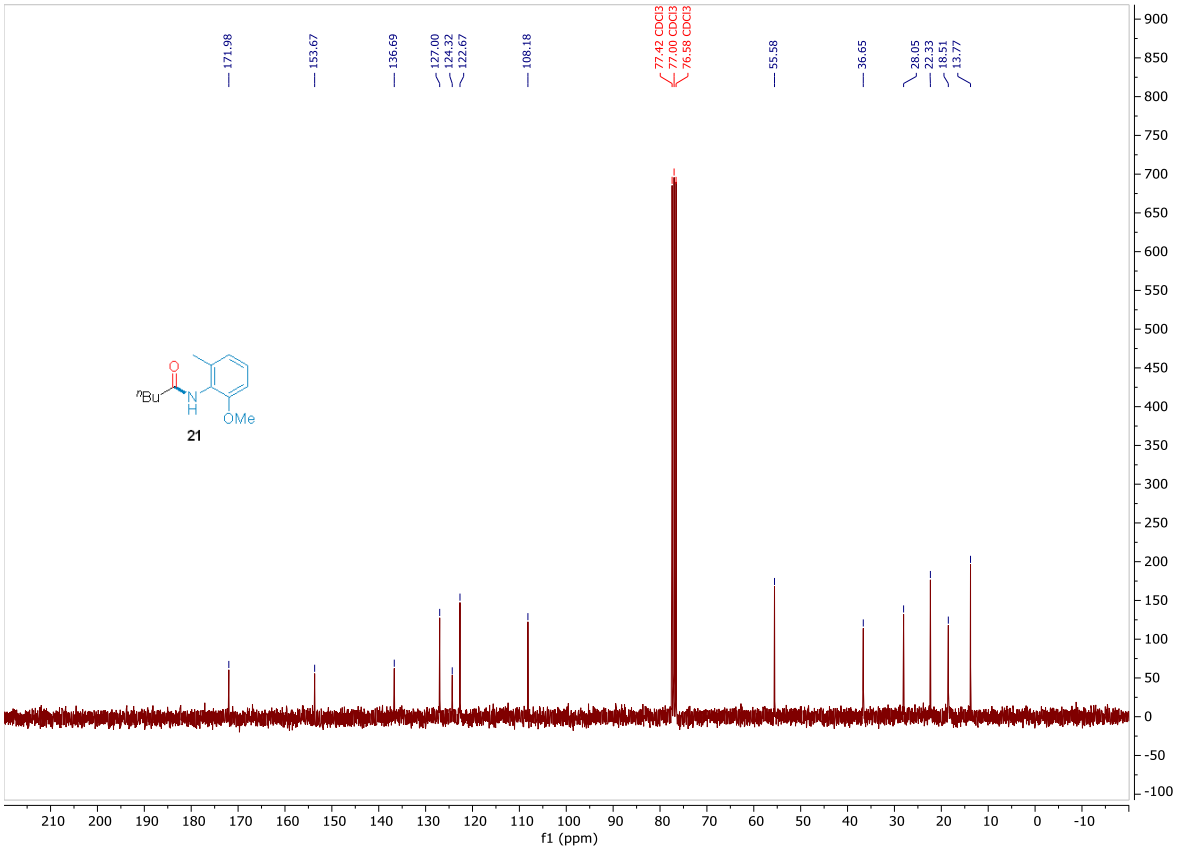
160.05
139.25
129.52
111.89
109.98
105.47
77.42 CDCl3
77.00 CDCl3
76.58 CDCl3
55.19
37.46
27.62
22.31
13.74
nBu
N
H
OMe
20
f1 (ppm)


7.13
7.11
7.08
6.95
6.84
6.81
6.74
6.71
3.79
2.43
2.41
2.38
2.22
1.78
1.75
1.73
1.70
1.68
1.50
1.48
1.45
1.43
1.40
1.38
0.98
0.96
0.93
nBu
N
H
O
OMe
21
0.93
0.78
1.09
0.94
3.17
1.80
3.00
1.86
1.93
2.77
f1 (ppm)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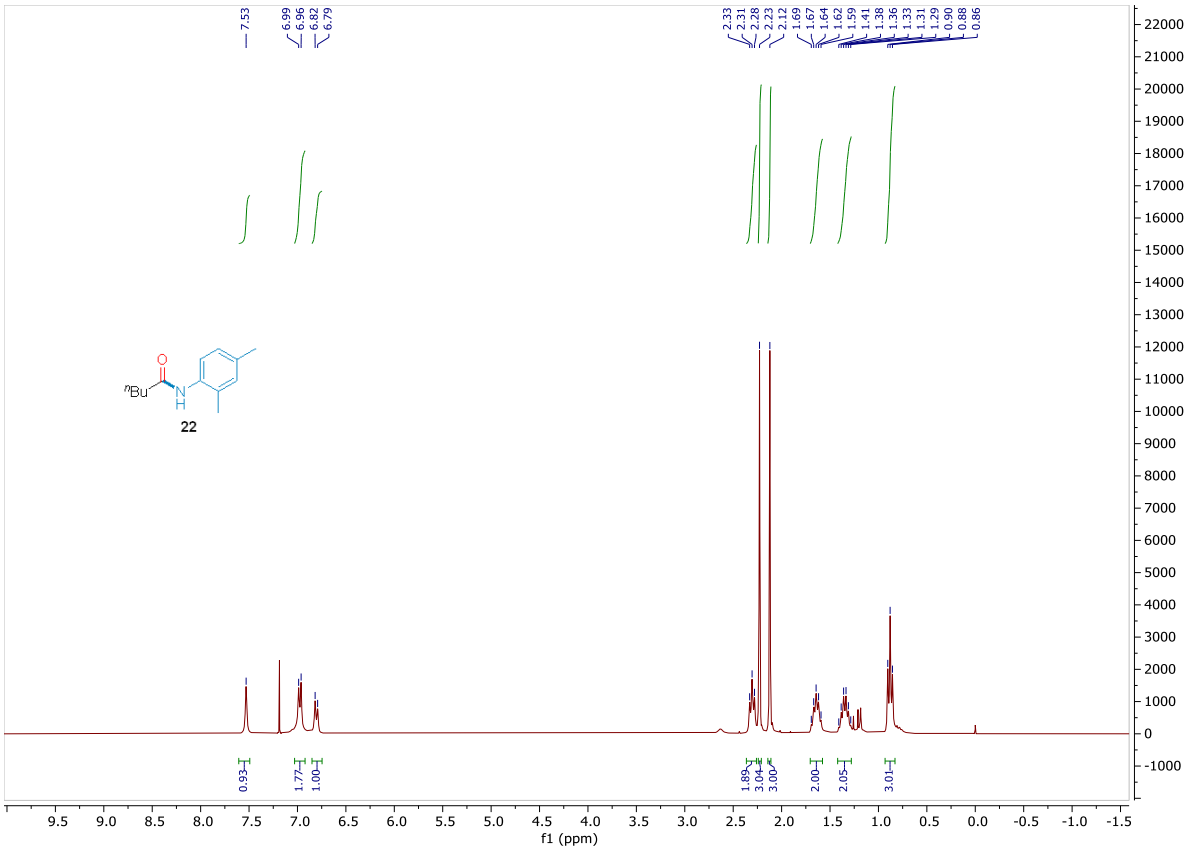
136.69
127.00
124.32
122.67
108.18
77.42 CDCl3
77.00 CDCl3
76.58 CDCl3
55.58
36.65
28.05
22.33
18.51
13.77
nBu
N
H
O
OMe
21
f1 (ppm)


7.53
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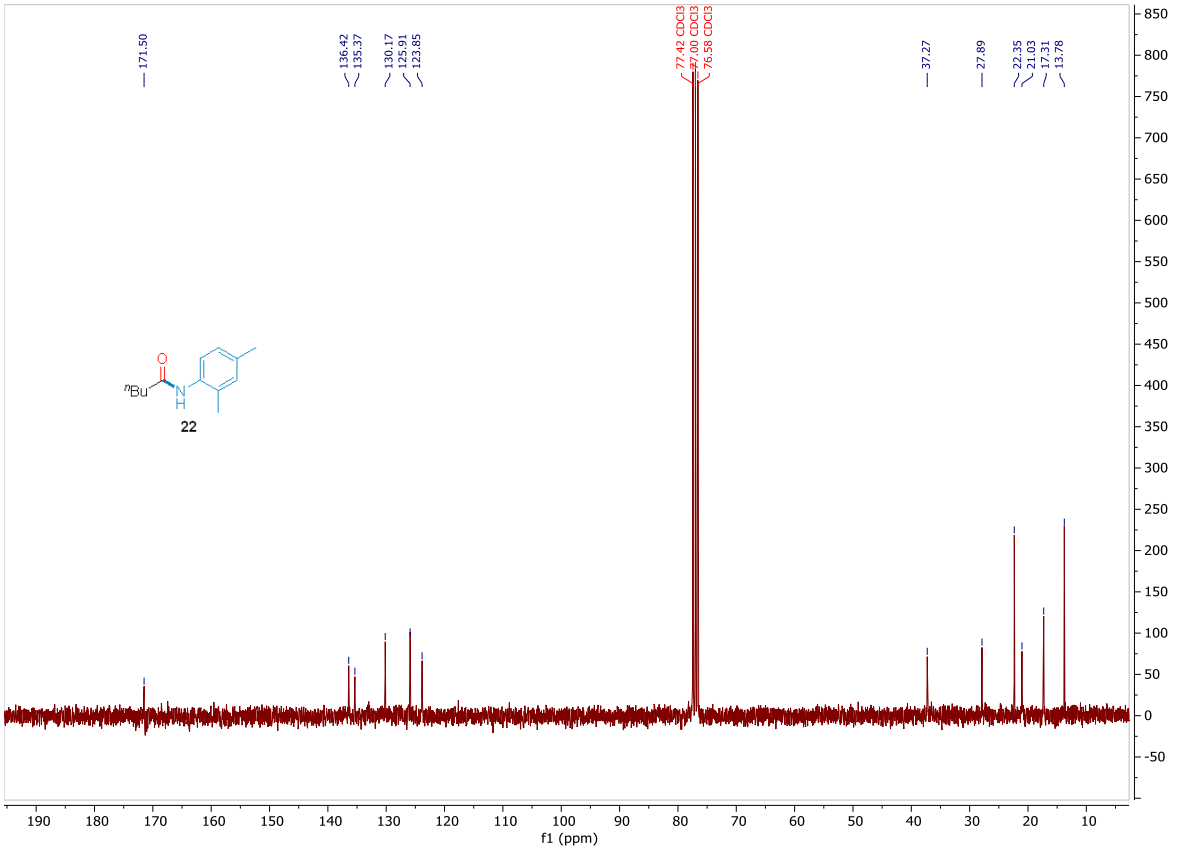
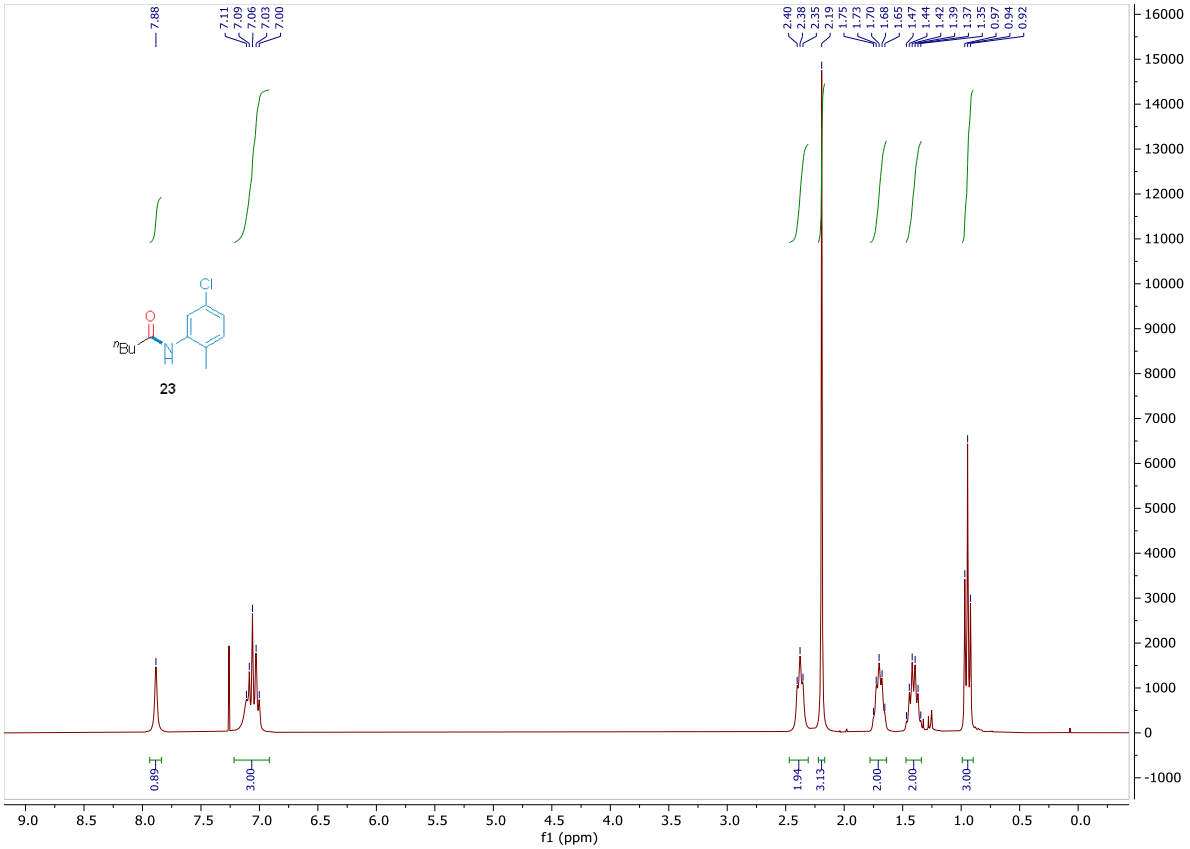
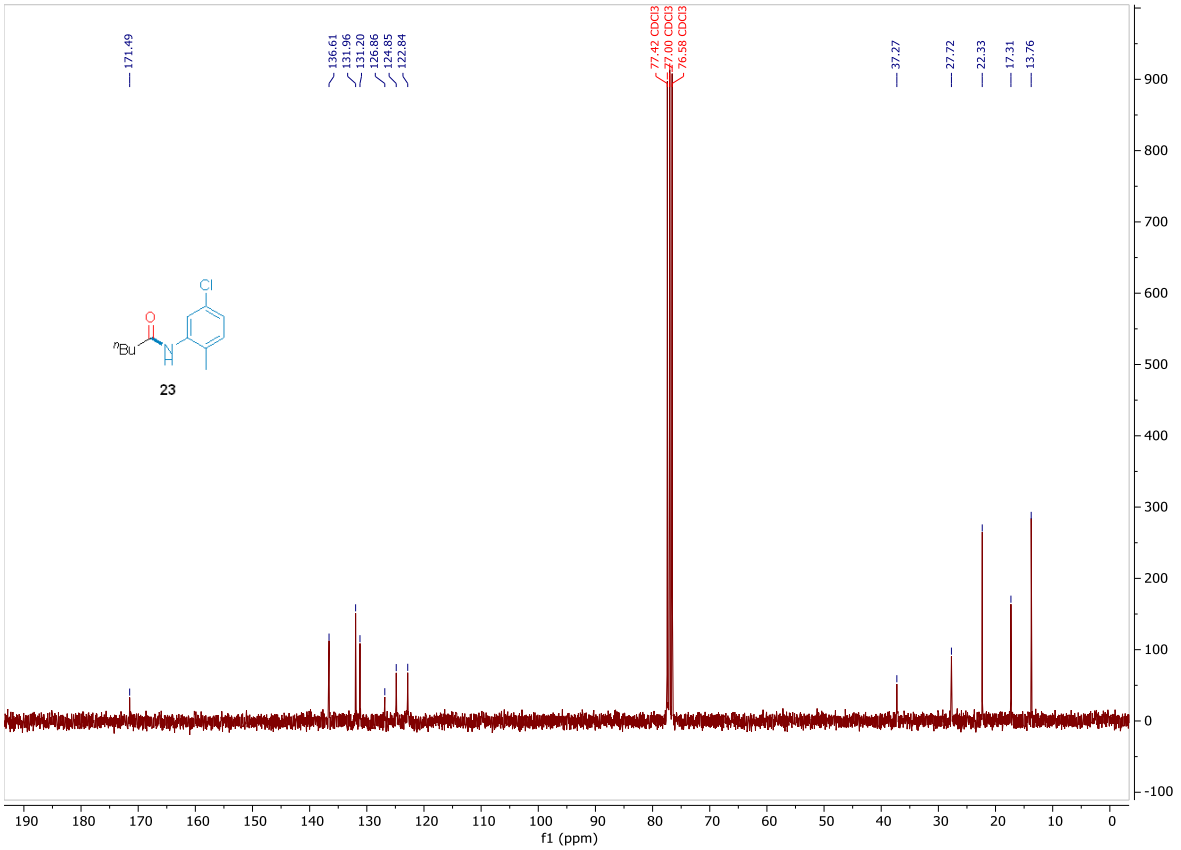
6.82
6.79
2.33
2.31
2.28
2.23
2.12
1.69
1.67
1.64
1.62
1.59
1.41
1.38
1.36
1.33
1.31
1.29
0.90
0.88
0.86
0.93
1.77
1.00
1.89
3.04
3.00
2.00
2.05
3.01
f1 (ppm)

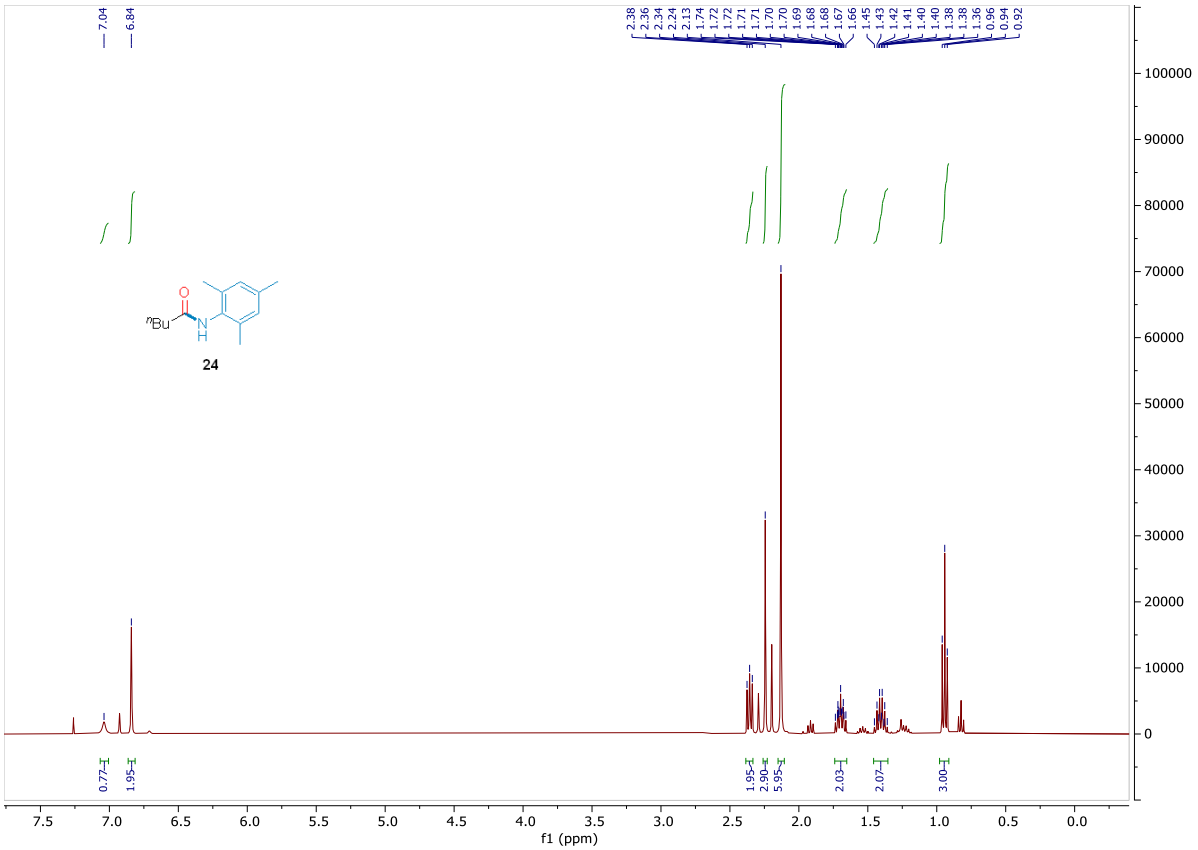
22

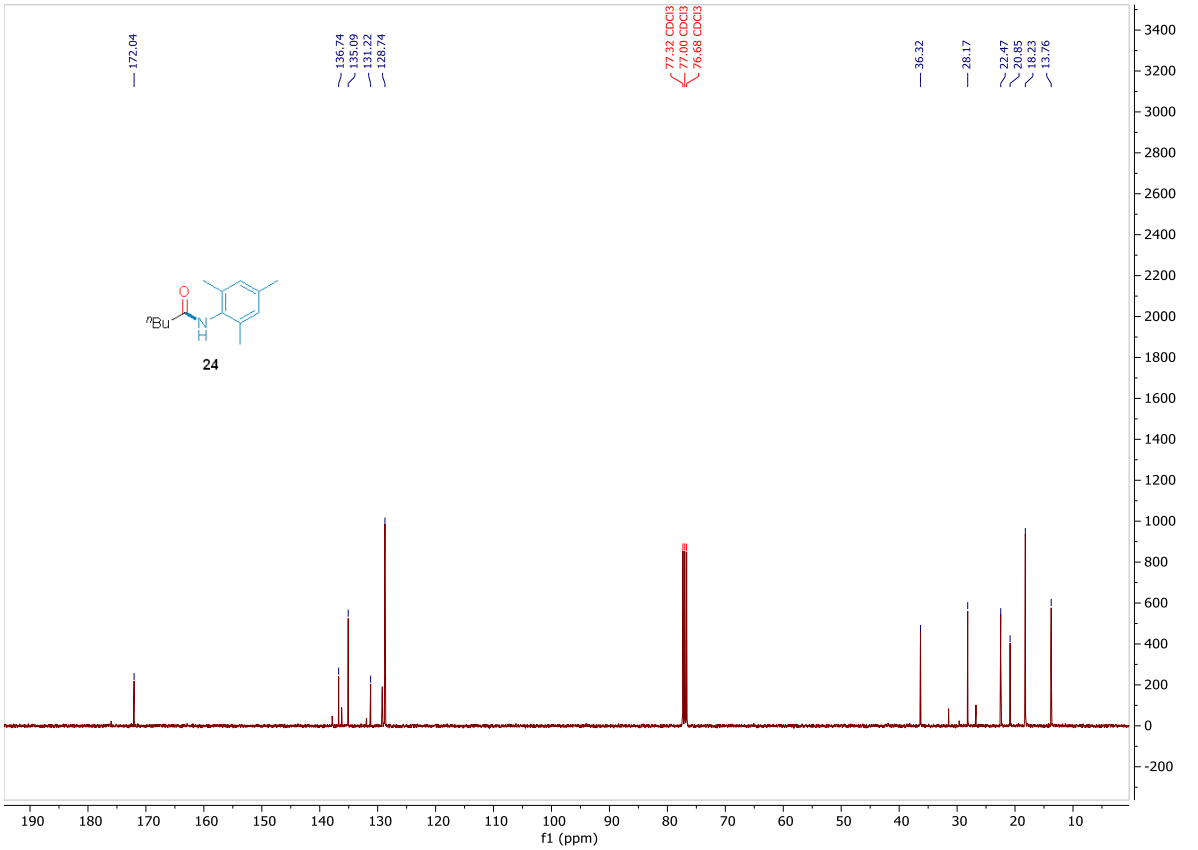

171.50
136.42
135.37
130.17
125.91
123.85
77.42 CDCl3
77.00 CDCl3
76.58 CDCl3
37.27
27.89
22.35
21.03
17.31
13.78
f1 (ppm)

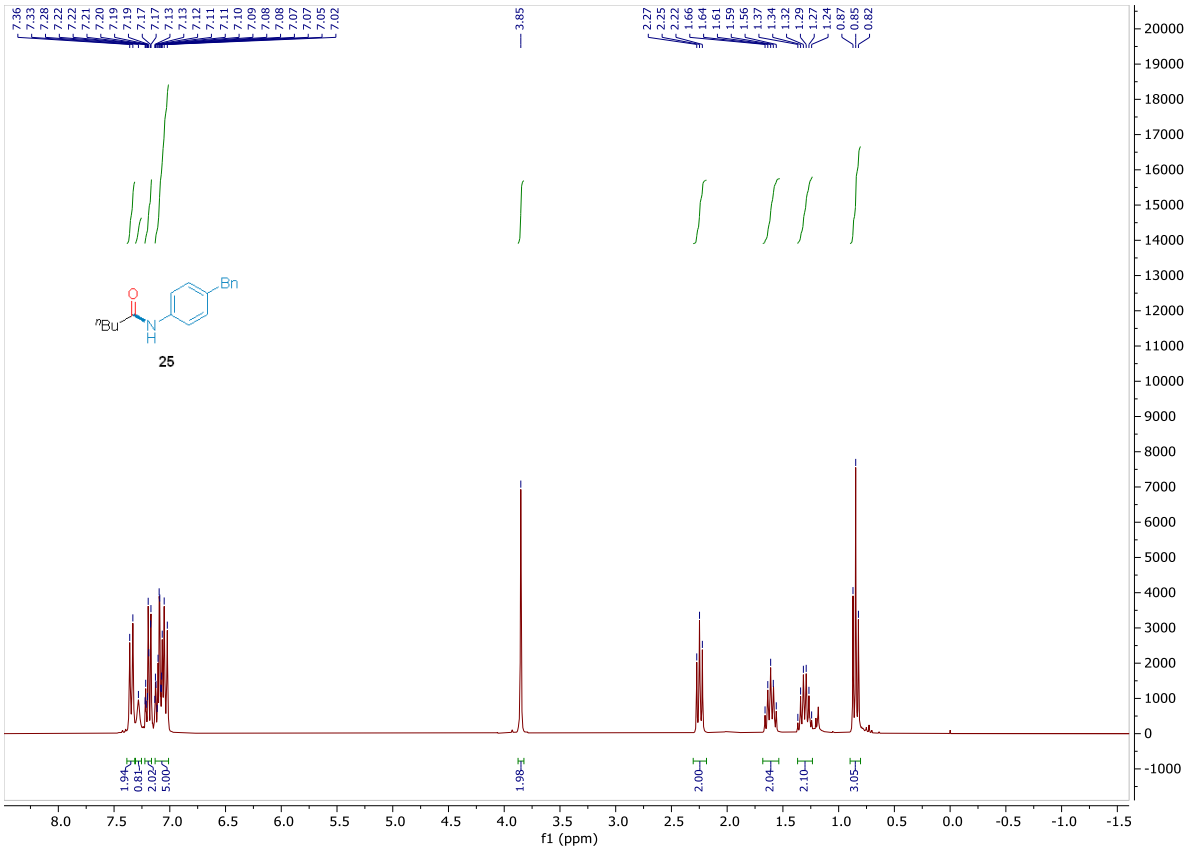
22


7.88
7.11
7.09
7.06
7.03
7.00
2.40
2.38
2.35
2.19
1.75
1.73
1.70
1.68
1.65
1.47
1.44
1.42
1.39
1.37
1.35
0.97
0.94
0.92
Cl
O
N
H
nBu
23
0.89
3.00
1.94
3.13
2.00
2.00
3.00
f1 (ppm)


171.49
136.61
131.96
131.20
126.86
124.85
122.84
77.42 CDCl3
77.00 CDCl3
76.58 CDCl3
37.27
27.72
22.33
17.31
13.76
Cl
O
N
H
nBu
23
f1 (ppm)


7.04
6.84
2.38
2.36
2.34
2.24
2.13
1.74
1.72
1.72
1.71
1.71
1.70
1.70
1.69
1.68
1.68
1.67
1.66
1.45
1.43
1.42
1.41
1.40
1.40
1.38
1.36
0.96
0.94
0.92
nBu
N
H
O
24
0.77
1.95
1.95
2.90
5.95
2.03
2.07
3.00
f1 (ppm)


172.04
136.74
135.09
131.22
128.74
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
36.32
28.17
22.47
20.85
18.23
13.76
nBu
N
H
O
24
f1 (ppm)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7.28
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7.22
7.21
7.20
7.19
7.19
7.17
7.17
7.13
7.13
7.12
7.11
7.11
7.10
7.09
7.08
7.08
7.07
7.07
7.05
7.02
3.85
2.27
2.25
2.22
1.66
1.64
1.61
1.59
1.56
1.37
1.34
1.32
1.29
1.27
1.24
0.87
0.85
0.82
Bn
nBu
O
N
H
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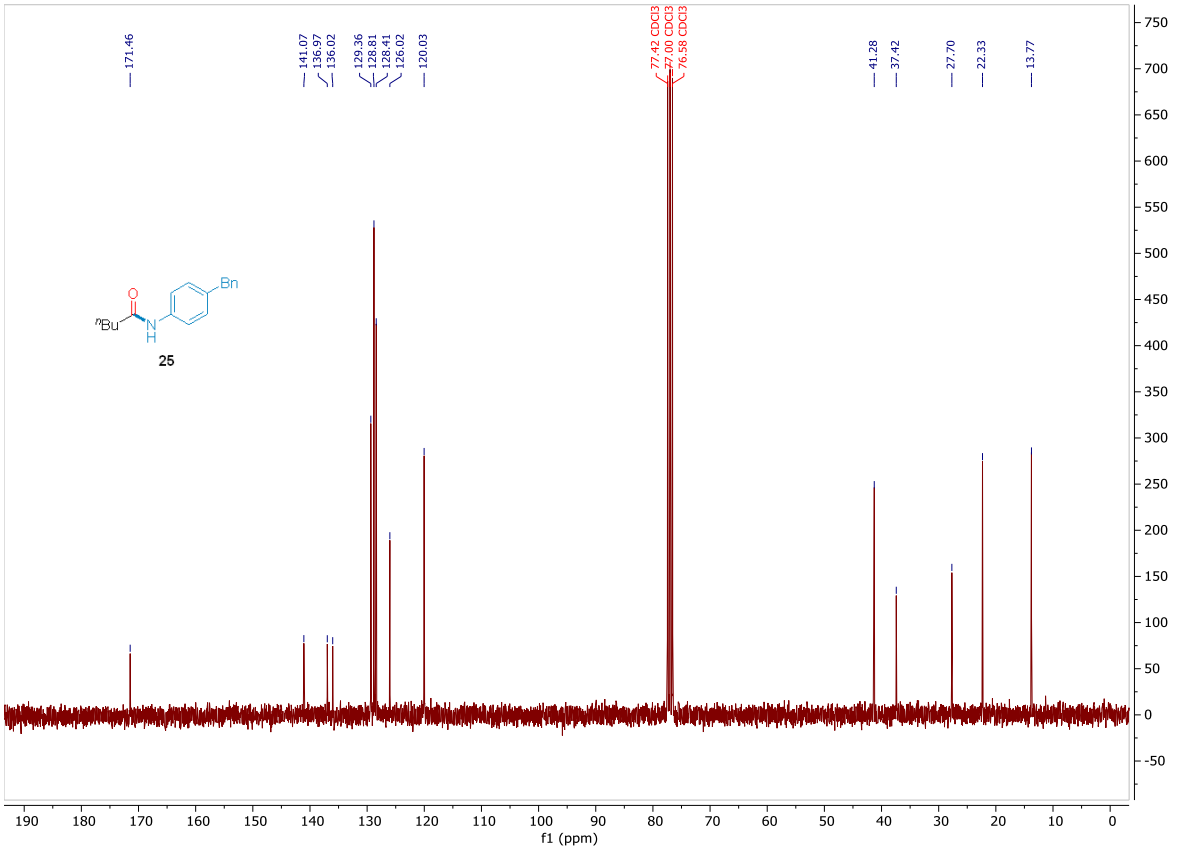
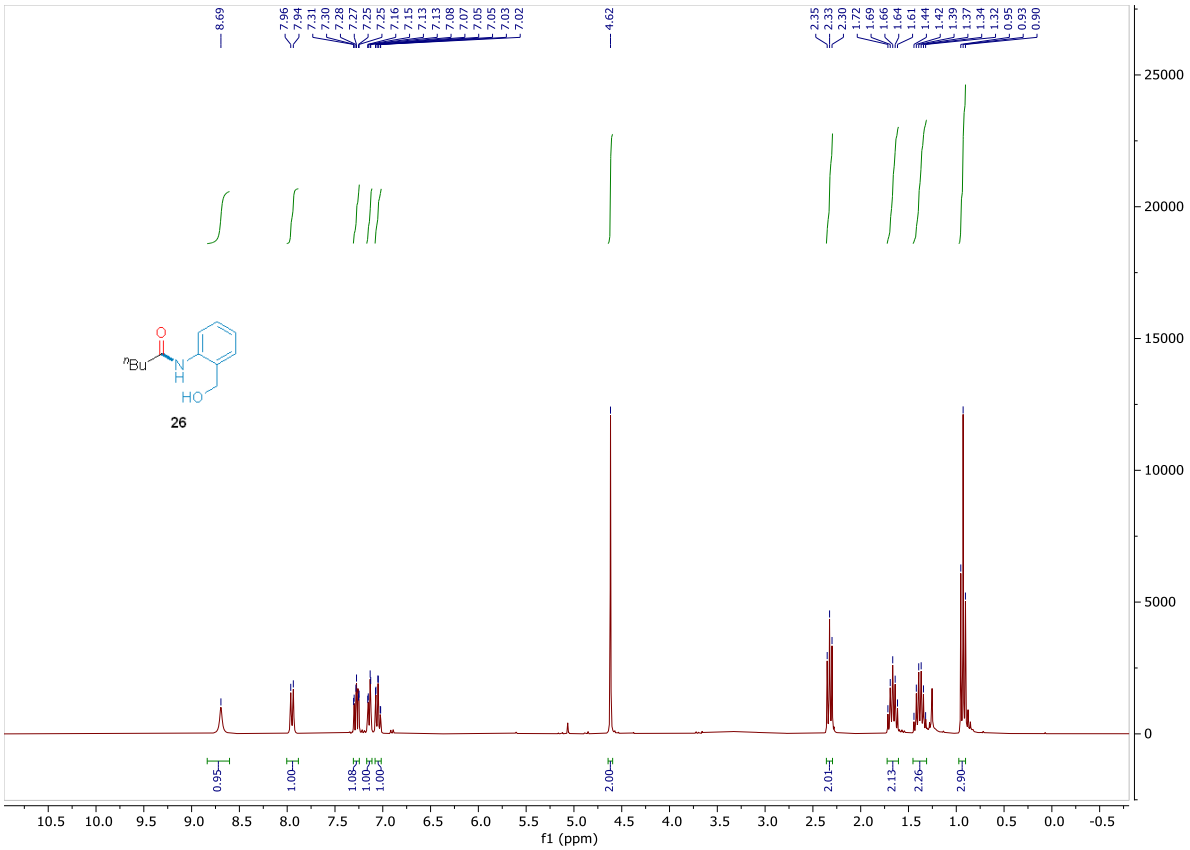
0.81
2.02
5.00
1.98
2.00
2.04
2.10
3.05
f1 (ppm)


171.46
141.07
136.97
136.02
129.36
128.81
128.41
126.02
120.03
77.42 CDCl3
77.00 CDCl3
76.58 CDCl3
41.28
37.42
27.70
22.33
13.77
Bn
nBu
O
N
H
25
f1 (ppm)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7.96
7.94
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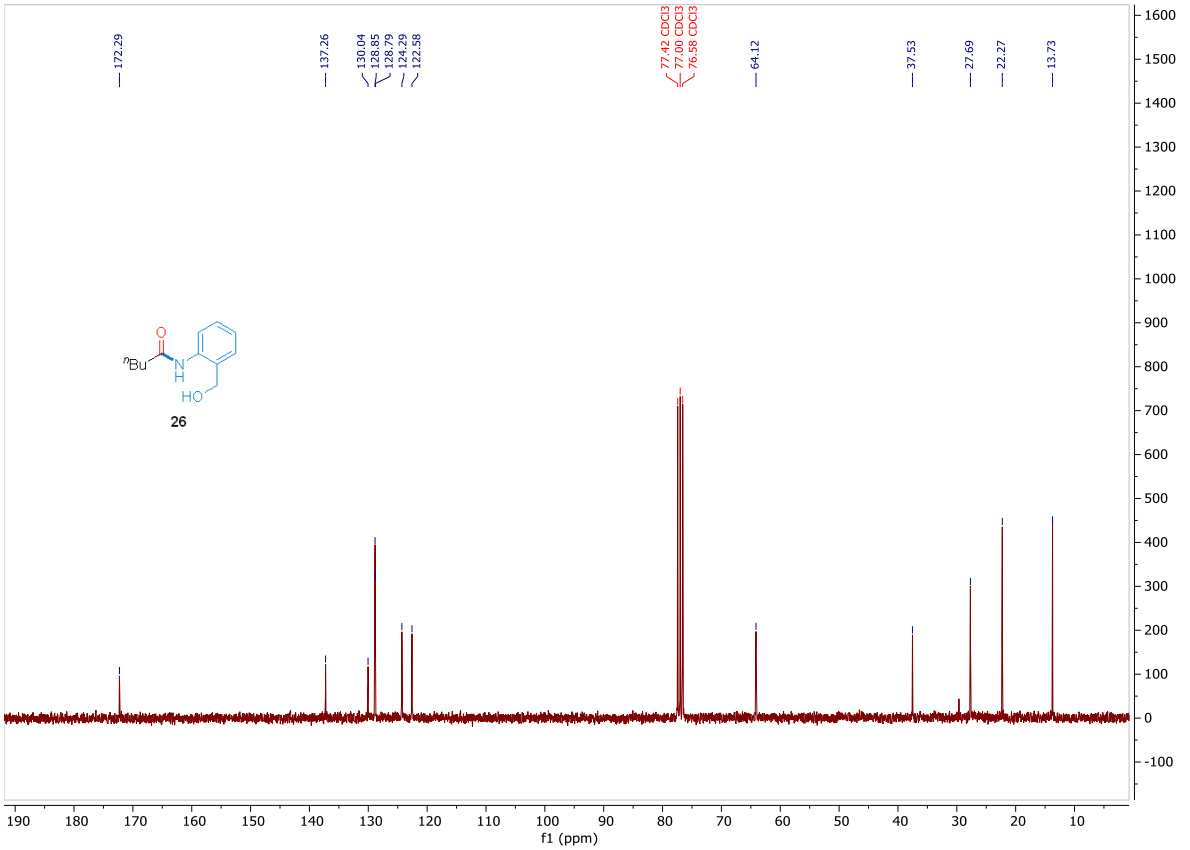
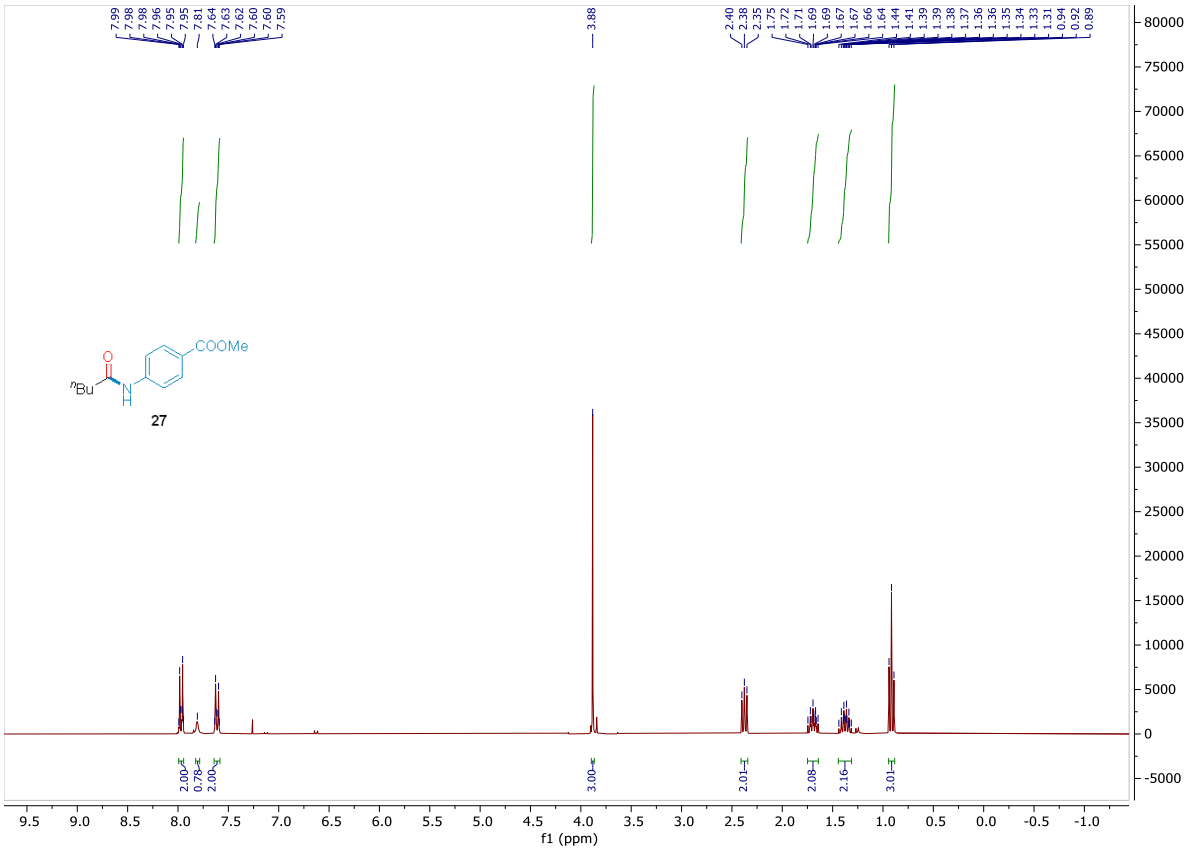
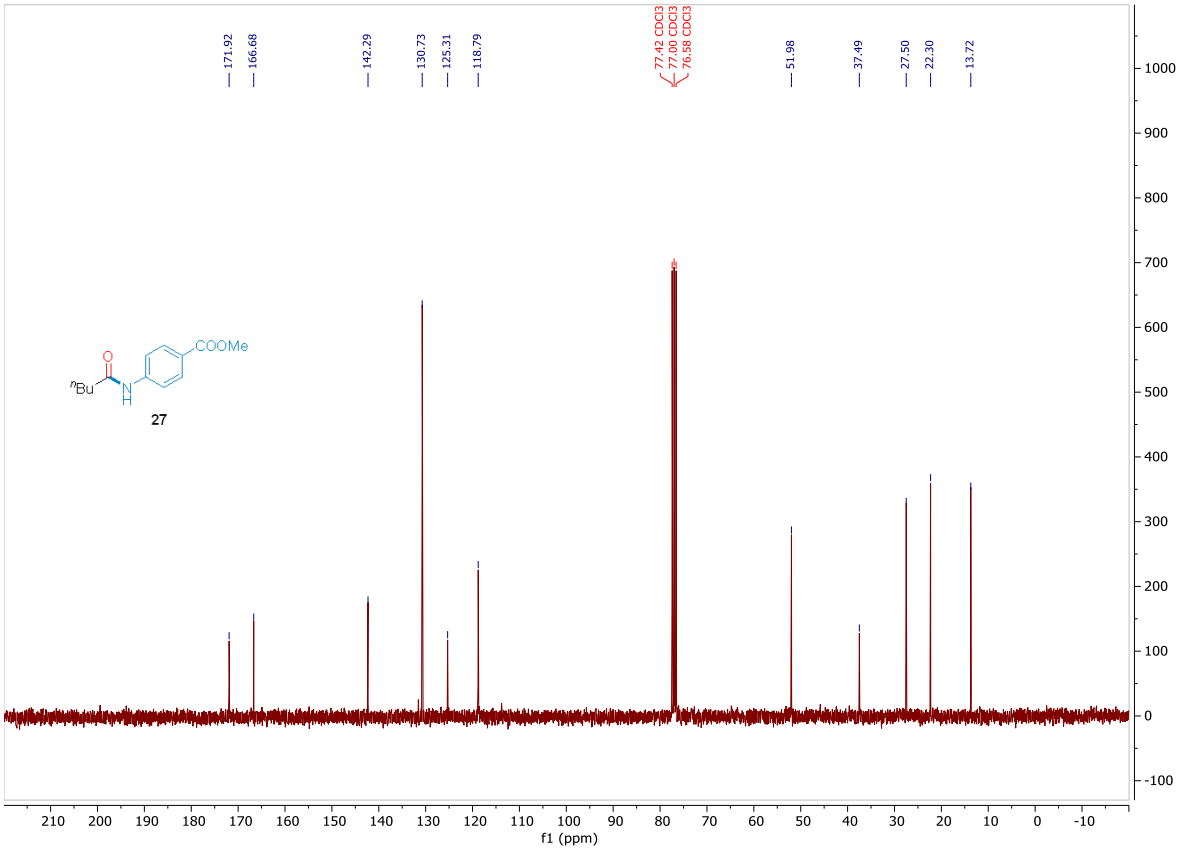
7.28
7.27
7.25
7.25
7.16
7.15
7.13
7.13
7.08
7.07
7.05
7.05
7.03
7.02
4.62
2.35
2.33
2.30
1.72
1.69
1.66
1.64
1.61
1.44
1.42
1.39
1.37
1.34
1.32
0.95
0.93
0.90
nBu
N
H
O
HO
26
0.95
1.00
1.08
1.00
1.00
2.00
2.01
2.13
2.26
2.90
f1 (ppm)


172.29
137.26
130.04
128.85
128.79
124.29
122.29
122.58
77.42 CDCl3
77.00 CDCl3
76.58 CDCl3
64.12
37.53
27.69
22.27
13.73
nBu
N
H
O
HO
26
f1 (ppm)


7.99
7.98
7.98
7.96
7.95
7.95
7.81
7.64
7.63
7.62
7.60
7.60
7.59
3.88
2.40
2.38
2.35
1.75
1.72
1.71
1.69
1.69
1.67
1.67
1.66
1.64
1.44
1.41
1.39
1.39
1.38
1.37
1.36
1.36
1.35
1.34
1.33
1.31
0.94
0.92
0.89
COOMe
nBu
N
H
27
2.00
0.78
2.00
3.00
2.01
2.08
2.16
3.01
f1 (ppm)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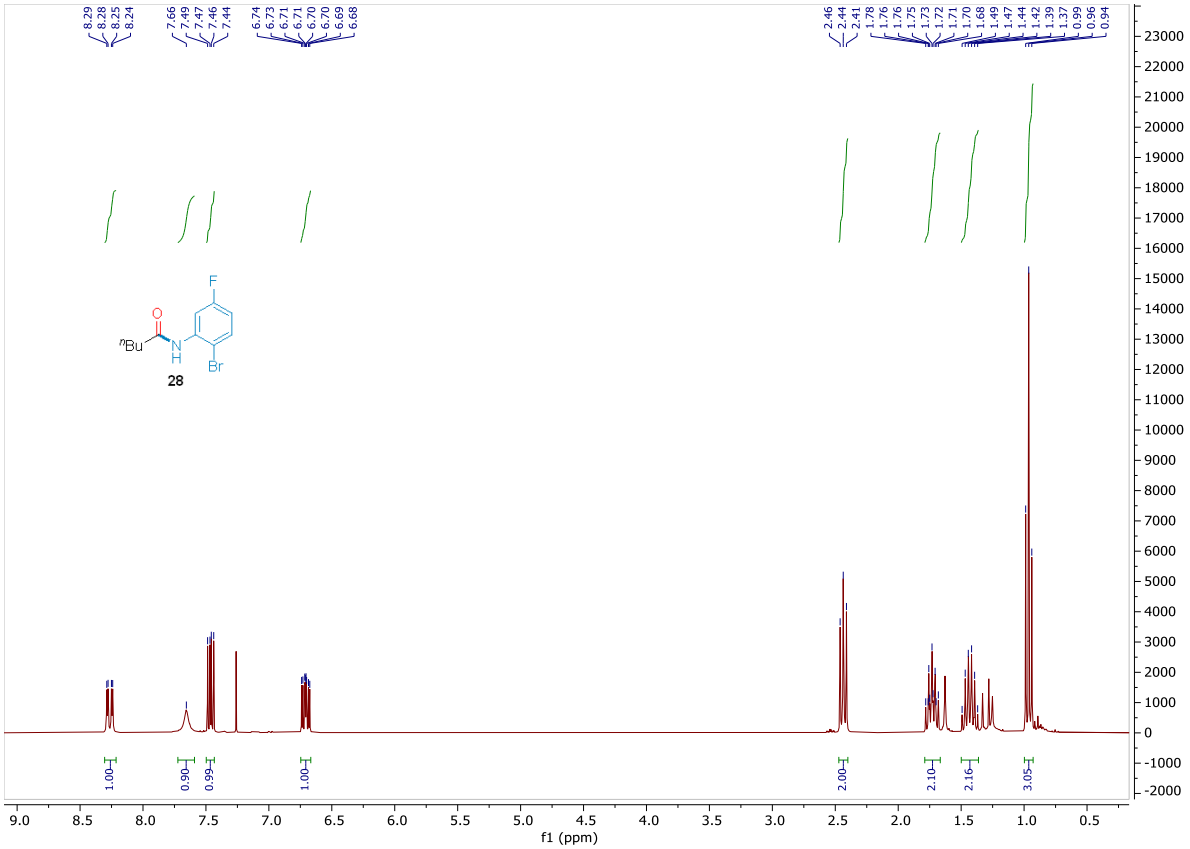
166.68
142.29
130.73
125.31
118.79
77.42 CDCl3
77.00 CDCl3
76.58 CDCl3
51.98
37.49
27.50
22.30
13.72
COOMe
nBu
N
H
27
f1 (ppm)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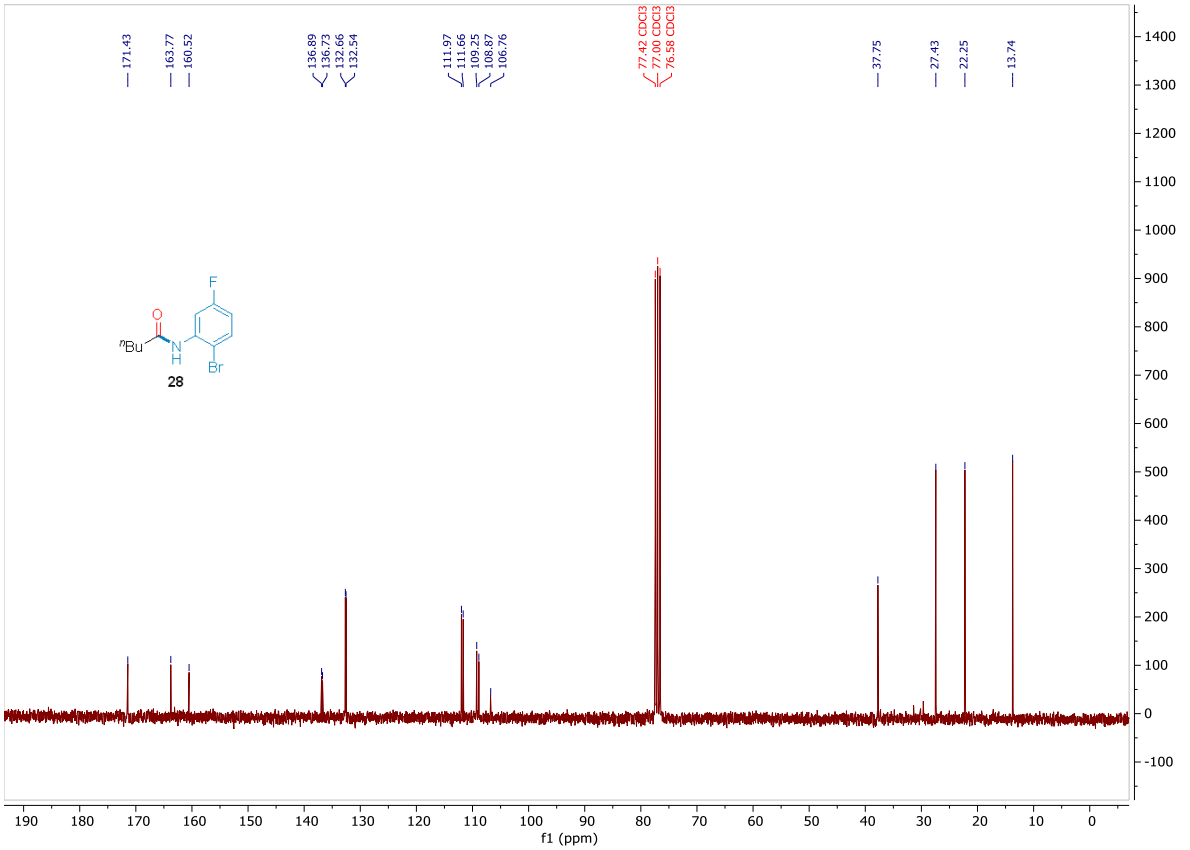
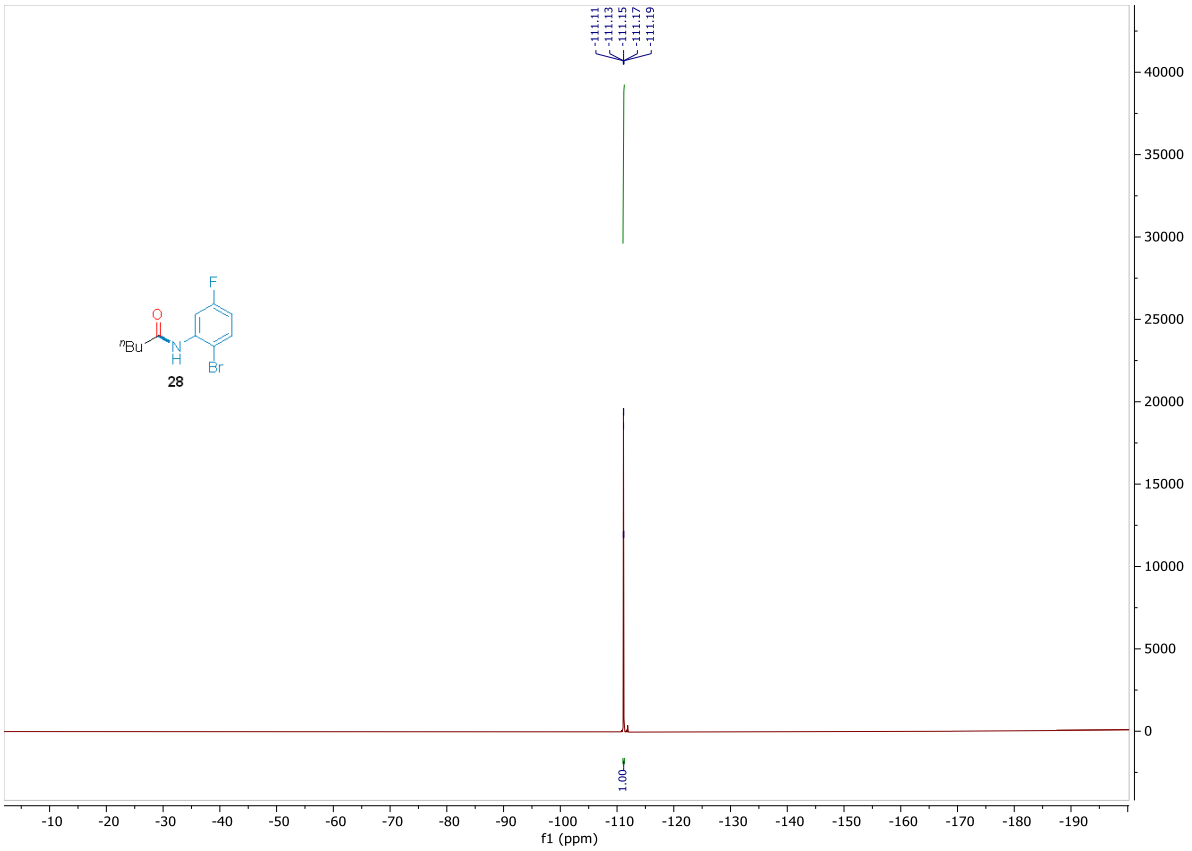
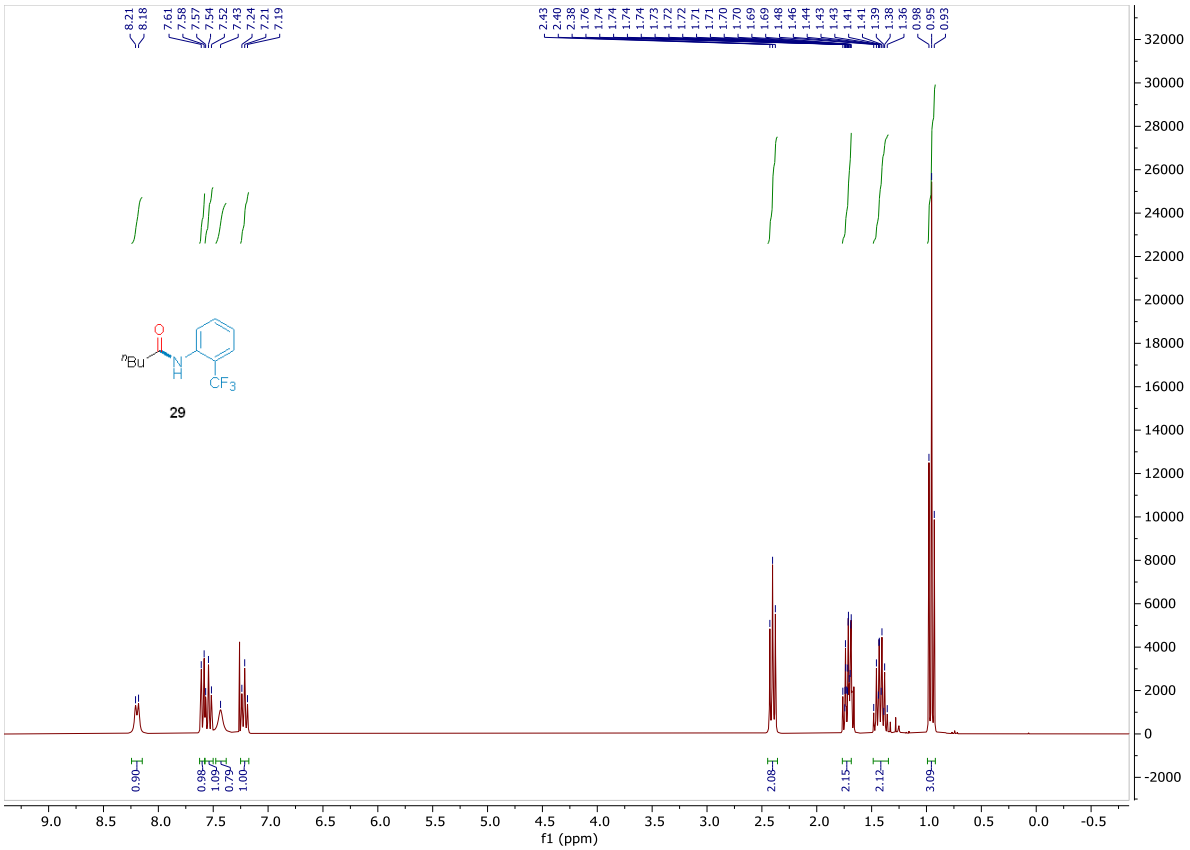
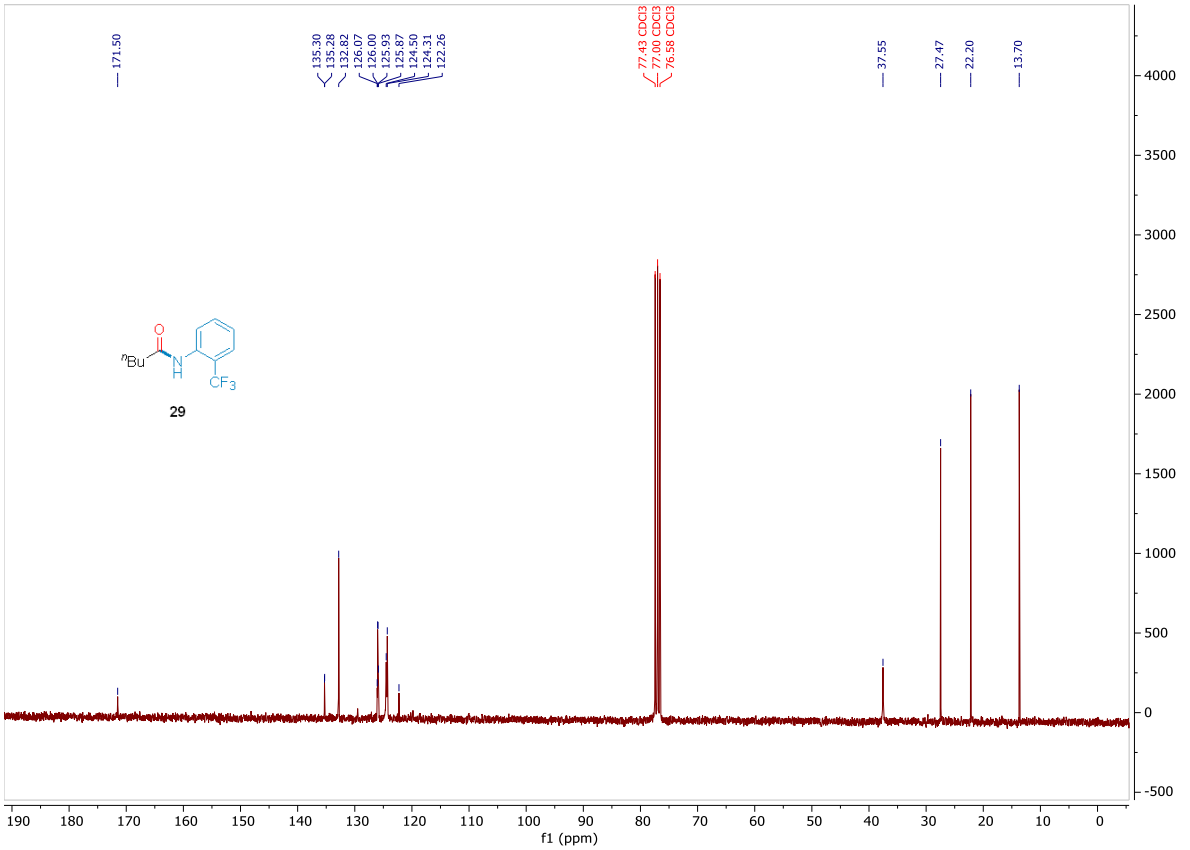
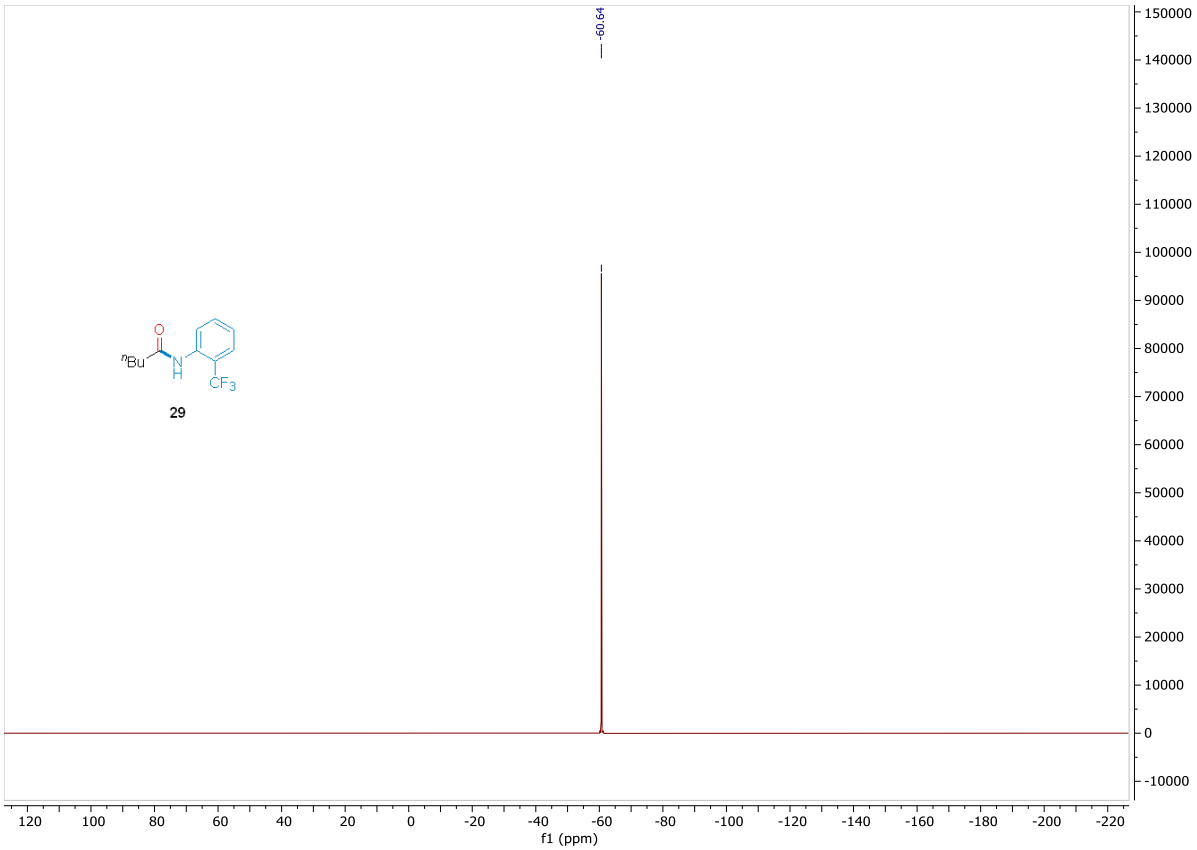
8.28
8.25
8.24
7.66
7.49
7.47
7.46
7.44
6.74
6.73
6.71
6.70
6.69
6.68
2.46
2.44
2.41
1.78
1.76
1.75
1.73
1.72
1.71
1.70
1.68
1.49
1.47
1.44
1.42
1.39
1.37
0.99
0.96
0.94
F
O
nBu
N
H
Br
28
1.00
0.90
0.99
1.00
2.00
2.10
2.16
3.05
f1 (ppm)


171.43
163.77
160.52
136.89
136.73
132.66
132.54
111.97
111.66
109.25
108.87
106.76
77.42 CDCl3
77.00 CDCl3
76.58 CDCl3
37.75
27.43
22.25
13.74
F
O
nBu
N
H
Br
28
f1 (ppm)

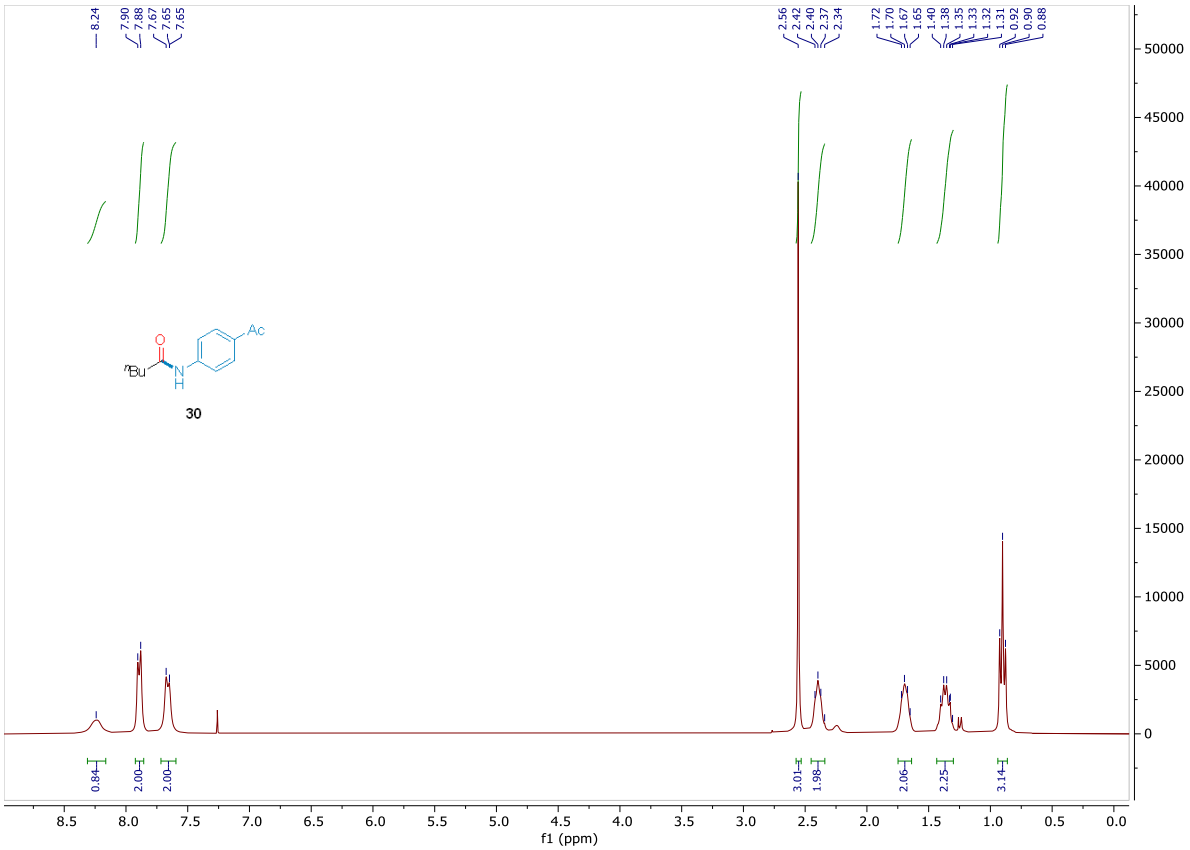

-111.11
-111.13
-111.15
-111.17
-111.19
40000
35000
30000
25000
20000
15000
10000
5000
0
1.00
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
f1 (ppm)
F
O
nBu
N
H
Br
28

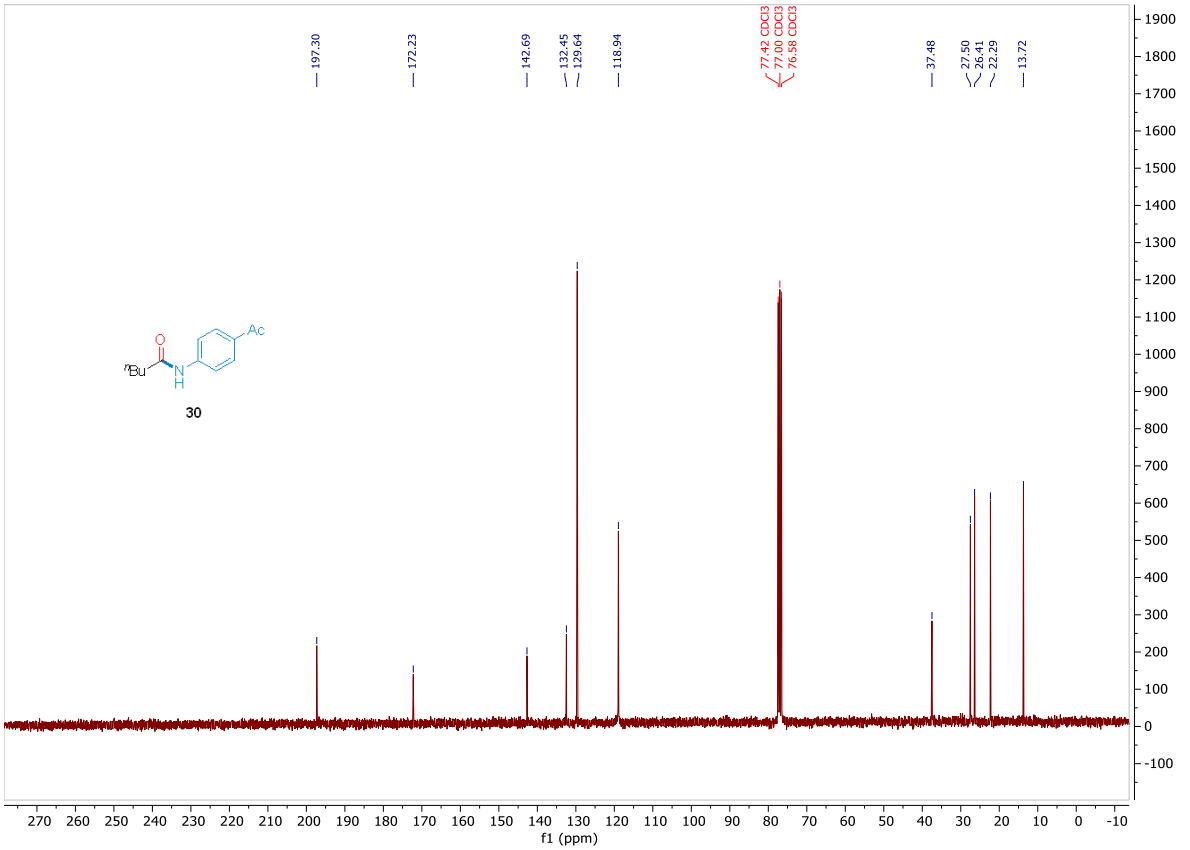

8.21
8.18
7.61
7.58
7.57
7.54
7.52
7.43
7.24
7.21
7.19
2.43
2.40
2.38
1.76
1.74
1.74
1.74
1.73
1.72
1.72
1.71
1.71
1.70
1.70
1.69
1.69
1.48
1.46
1.44
1.43
1.43
1.41
1.41
1.39
1.38
1.36
0.98
0.95
0.93
nBu
CF3
29
0.90
0.98
1.09
0.79
1.00
2.08
2.15
2.12
3.09
f1 (ppm)

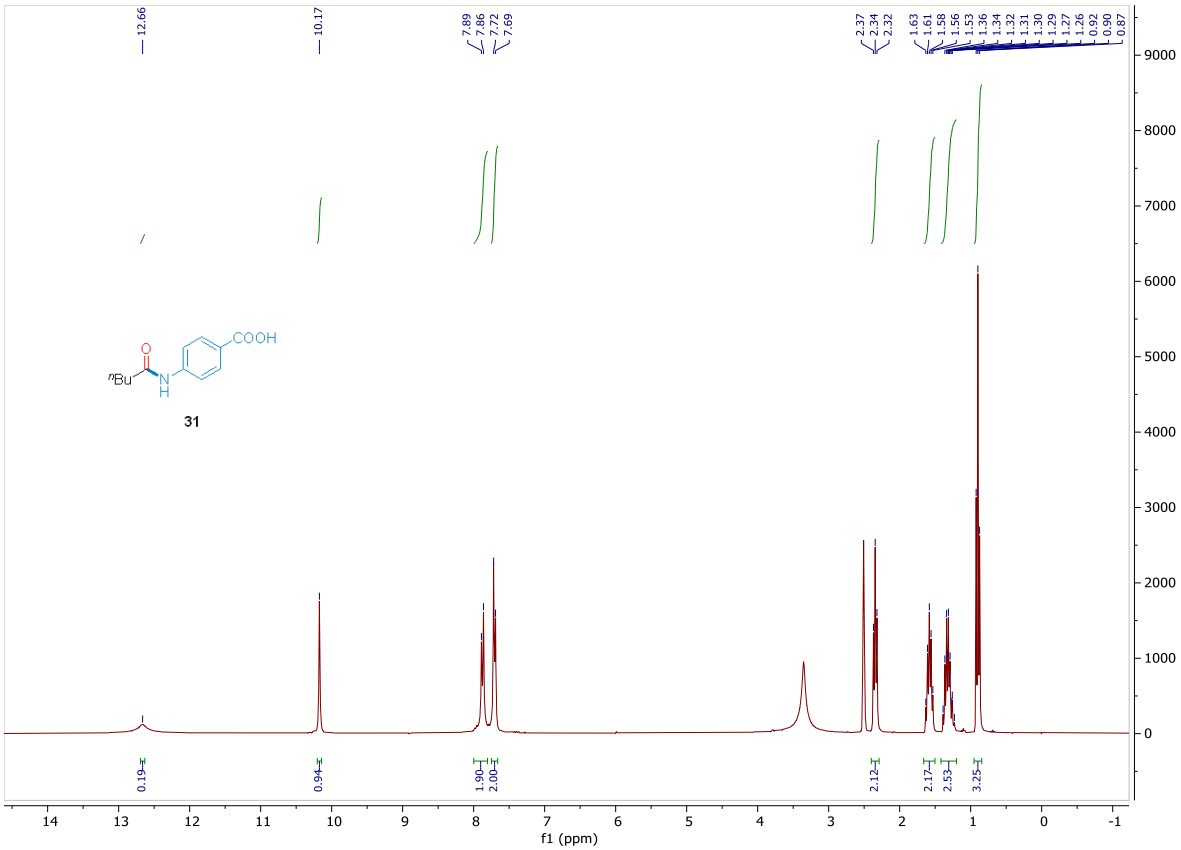

171.50
135.30
135.28
132.82
126.07
126.00
125.93
125.87
124.50
124.31
122.26
77.43 CDCl3
77.00 CDCl3
76.58 CDCl3
37.55
27.47
22.20
13.70
nBu
CF3
29
f1 (ppm)

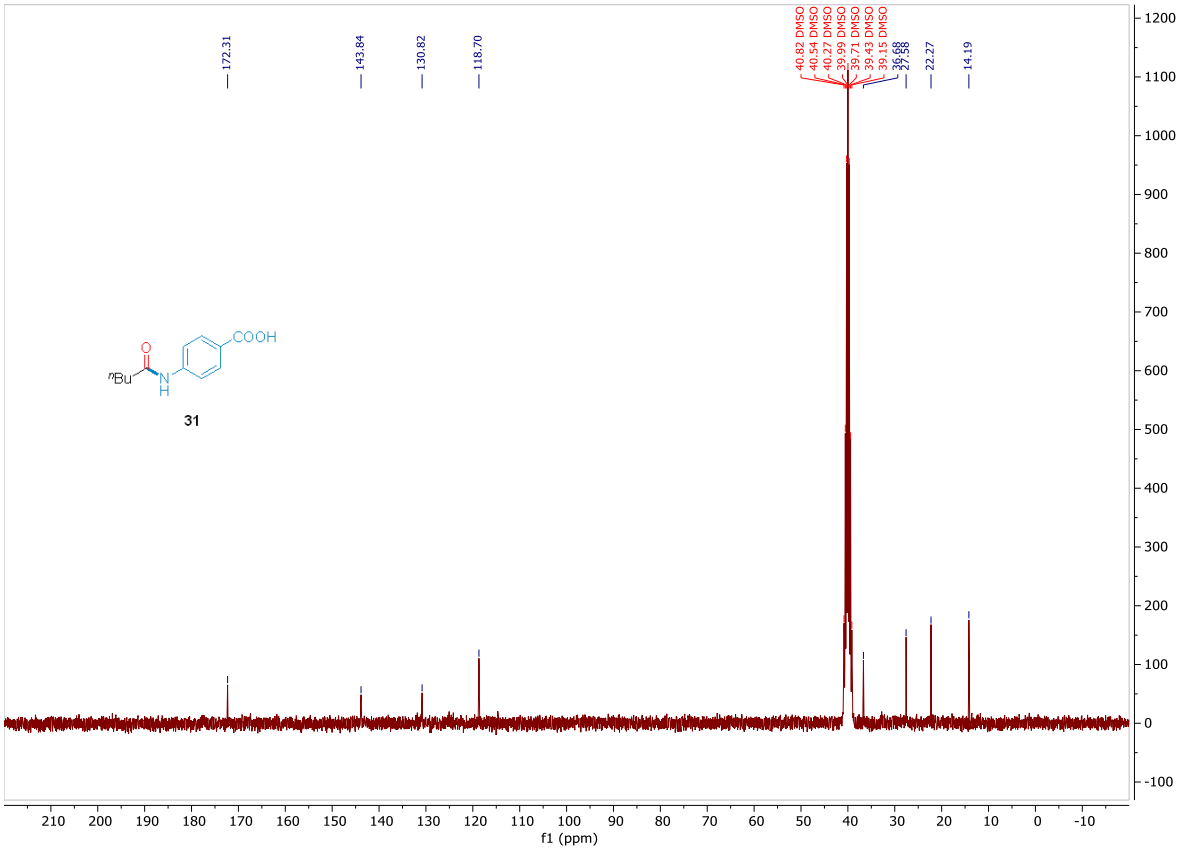

-60.64
150000
140000
130000
120000
110000
100000
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
-10000
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
f1 (ppm)
nBu
O
N
H
CF3

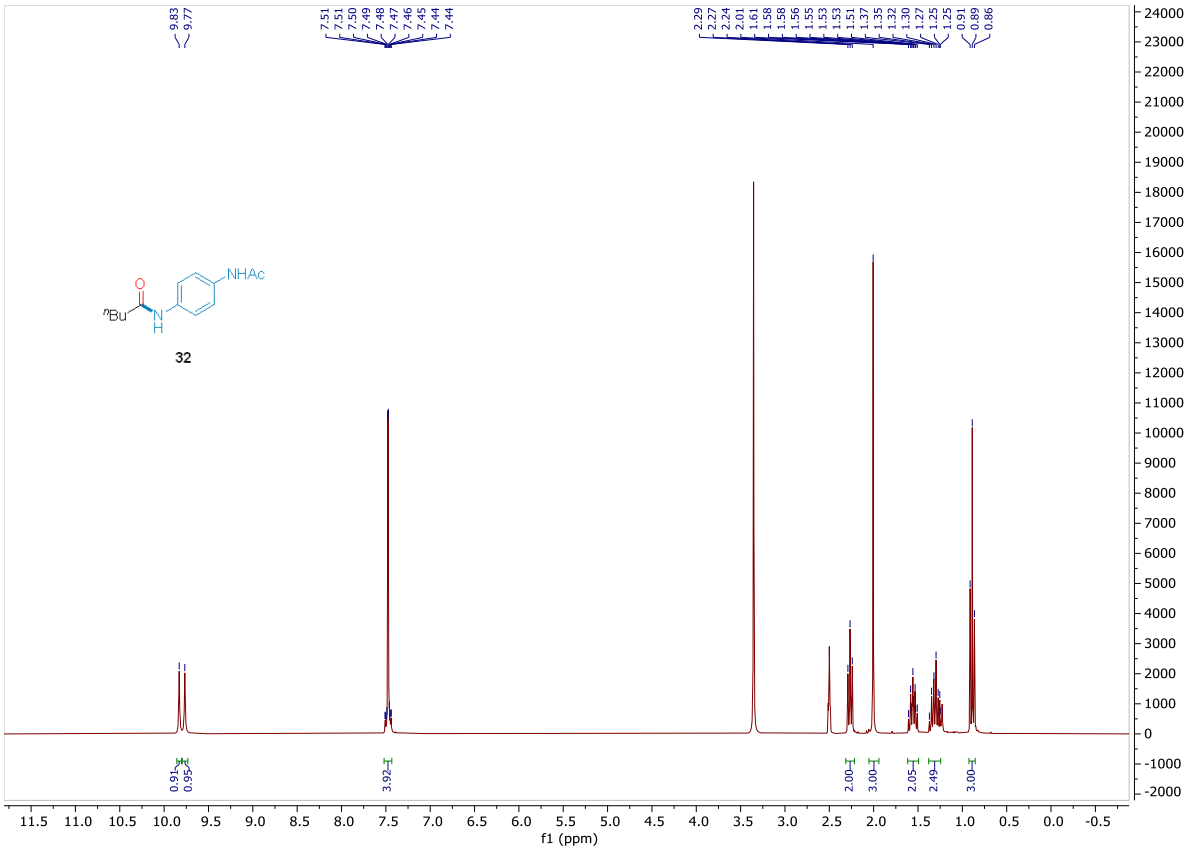
29

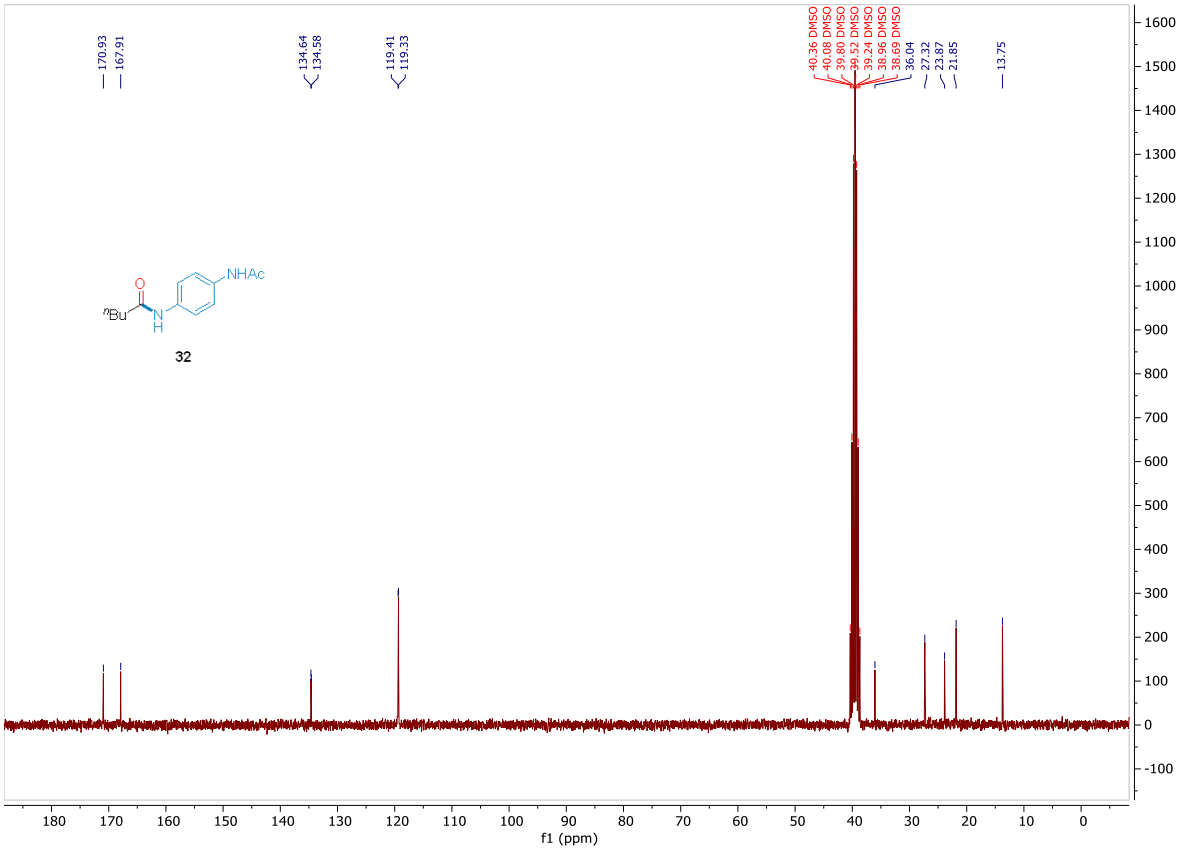

8.24
7.90
7.88
7.67
7.65
7.65
2.56
2.42
2.40
2.37
2.34
1.72
1.70
1.67
1.65
1.40
1.38
1.35
1.33
1.32
1.31
0.92
0.90
0.88
0.84
2.00
2.00
3.01
1.98
2.06
2.25
3.14
Ac
nBu
30
f1 (ppm)

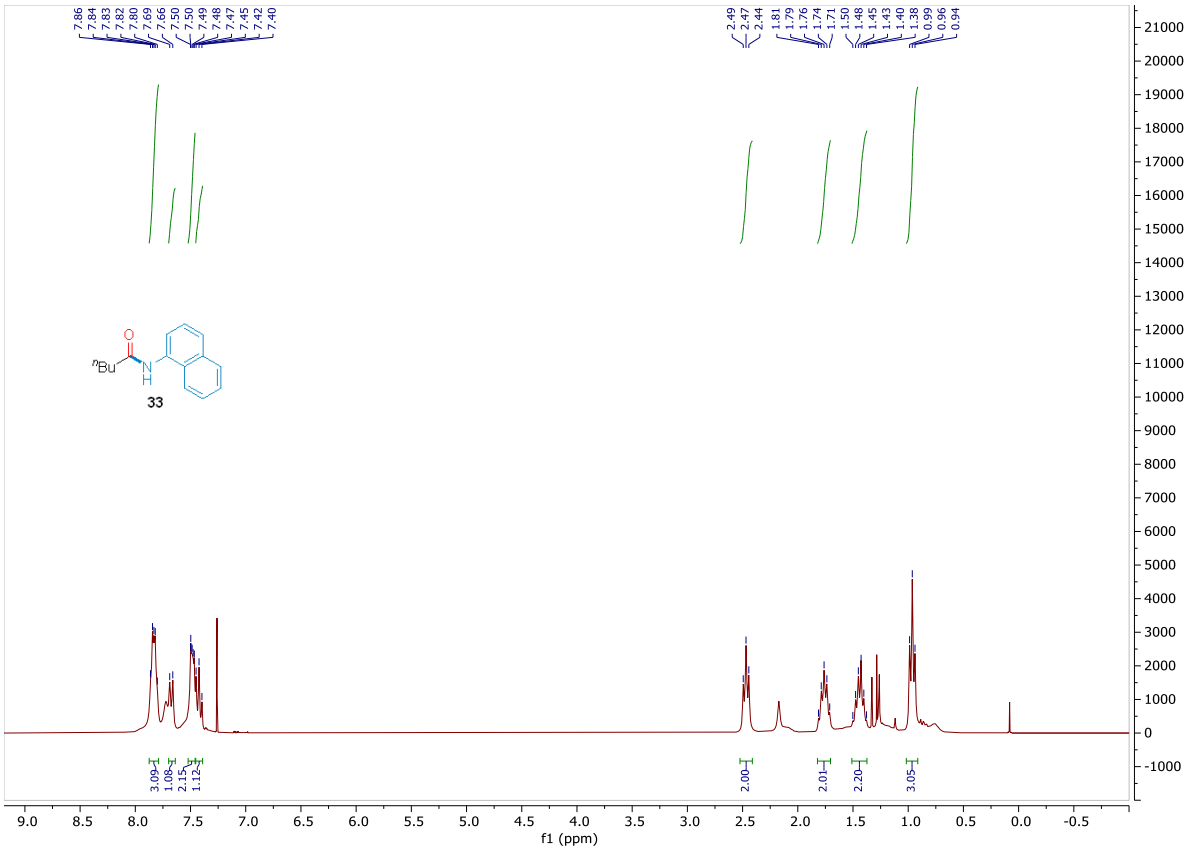

197.30
172.23
142.69
132.45
129.64
118.94
77.42 CDCl3
77.00 CDCl3
76.58 CDCl3
37.48
27.50
26.41
22.29
13.72
Ac
nBu
30
f1 (ppm)

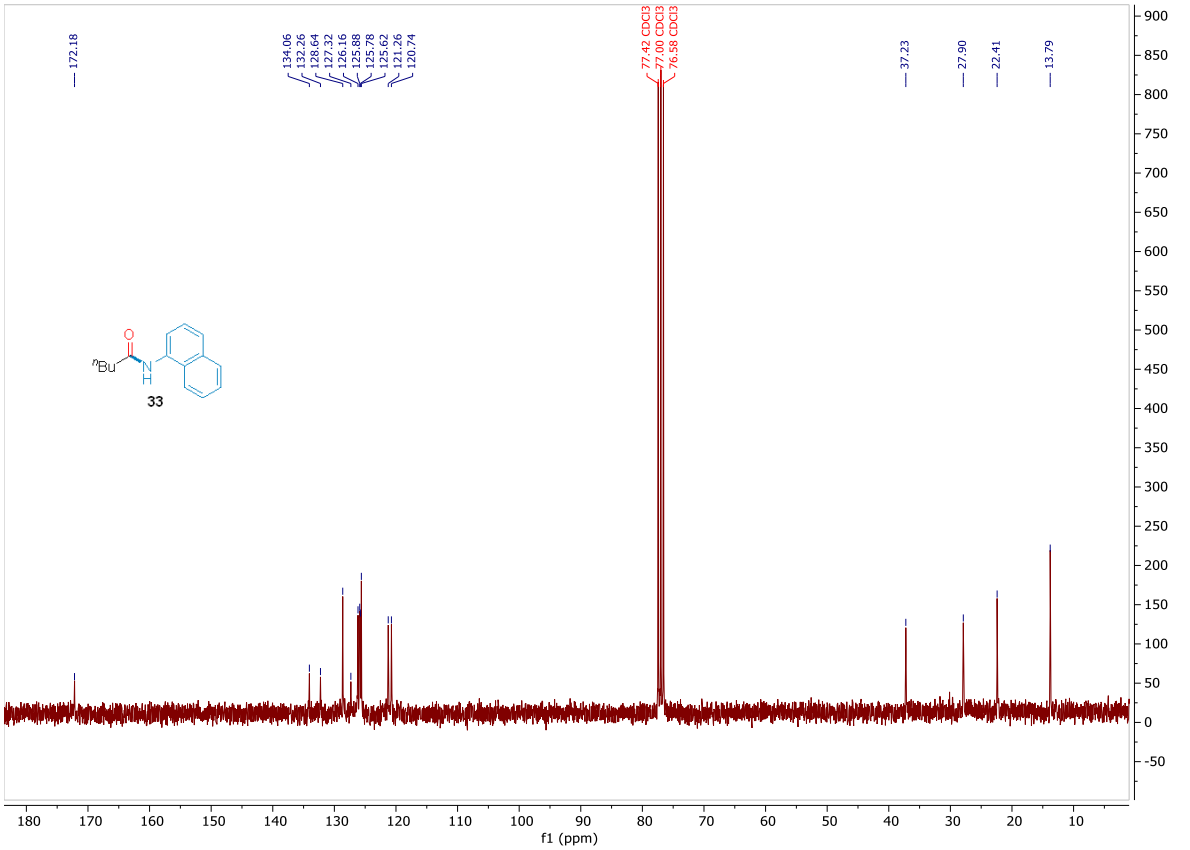

12.66
10.17
7.89
7.86
7.72
7.69
2.37
2.34
2.32
1.63
1.61
1.58
1.56
1.53
1.36
1.34
1.32
1.31
1.30
1.29
1.27
1.26
0.92
0.90
0.87
COOH
nBu
N
H
O
31
0.19
0.94
1.90
2.00
2.12
2.17
2.53
3.25
f1 (ppm)

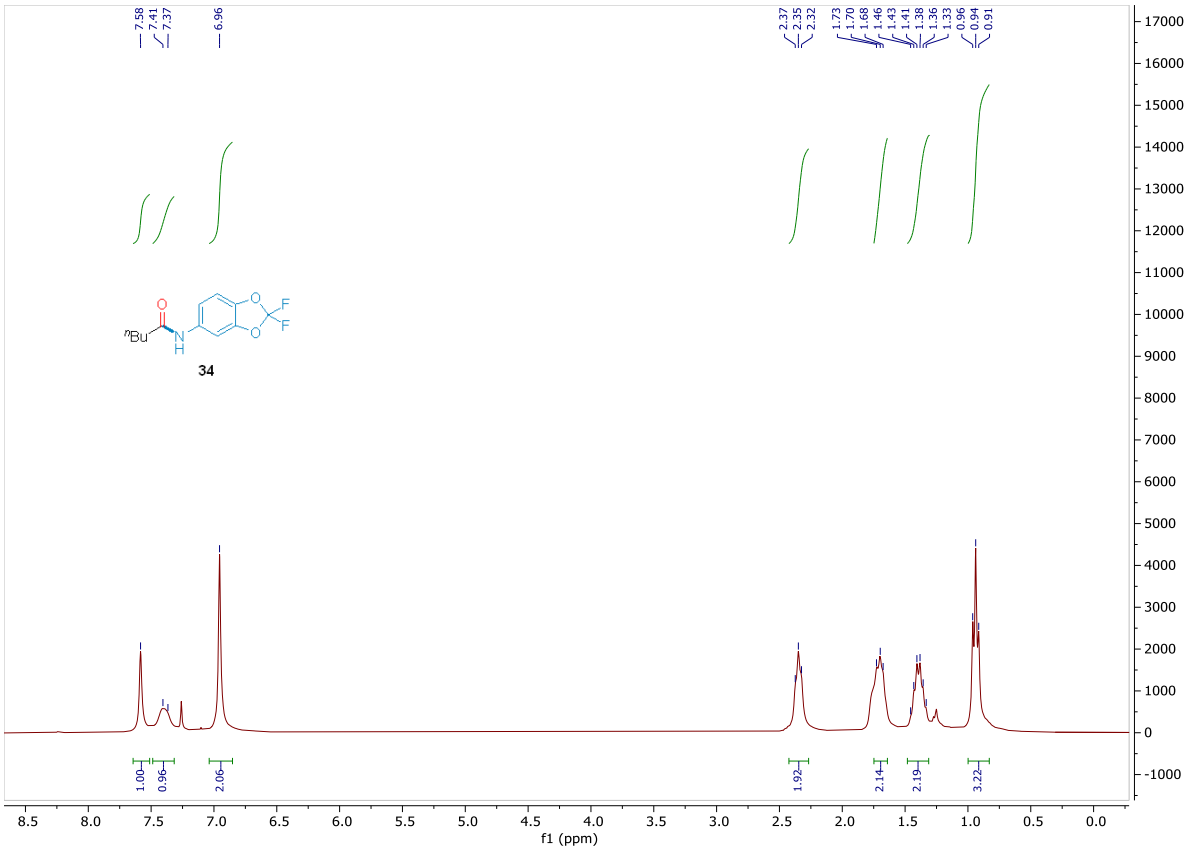

172.31
143.84
130.82
118.70
40.82 DMSO
40.54 DMSO
40.27 DMSO
39.99 DMSO
39.71 DMSO
39.43 DMSO
39.15 DMSO
22.27
14.19
COOH
nBu
N
H
O
31
f1 (ppm)

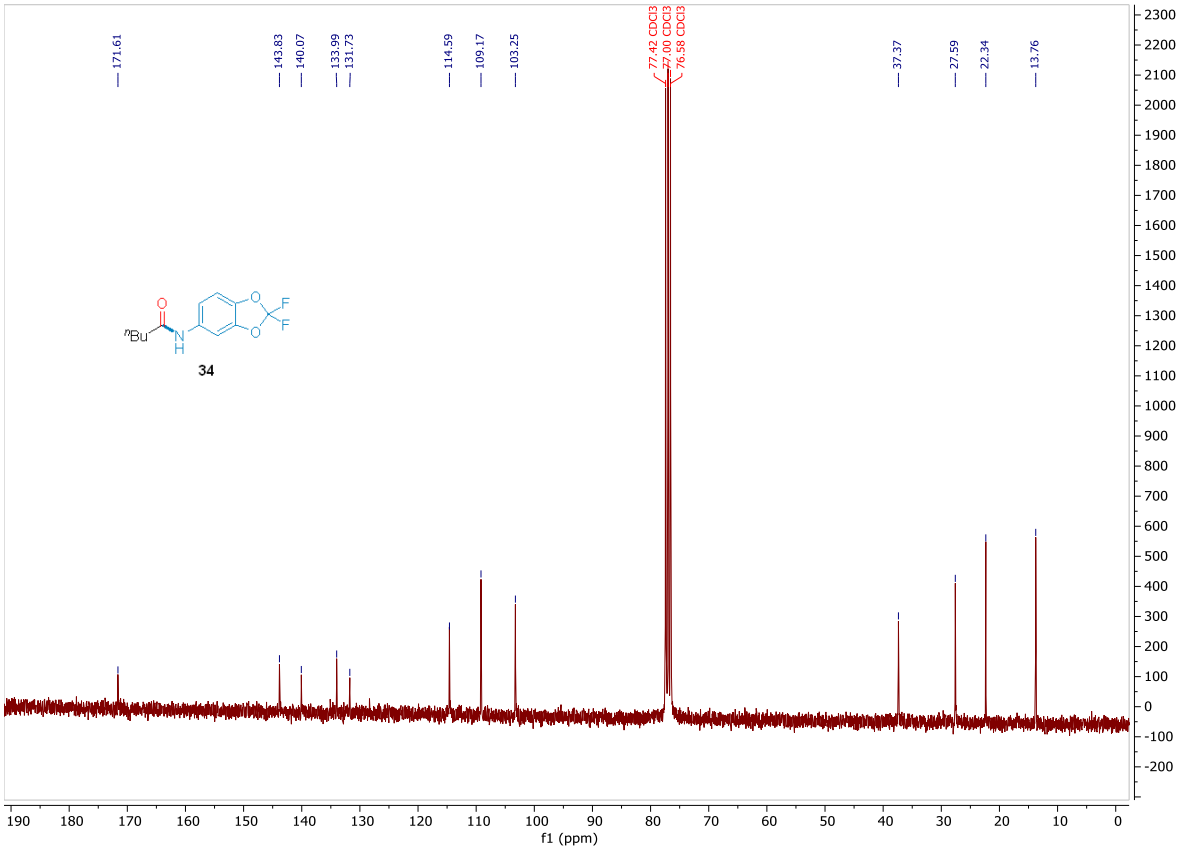

9.83
9.77
7.51
7.51
7.50
7.49
7.48
7.47
7.46
7.45
7.44
7.44
2.29
2.27
2.24
2.01
1.61
1.58
1.58
1.56
1.55
1.53
1.53
1.51
1.37
1.35
1.32
1.30
1.27
1.25
1.25
0.91
0.89
0.86
NHAc
nBu
N
H
O
32
0.91
0.95
3.92
2.00
3.00
2.05
2.49
3.00
f1 (ppm)

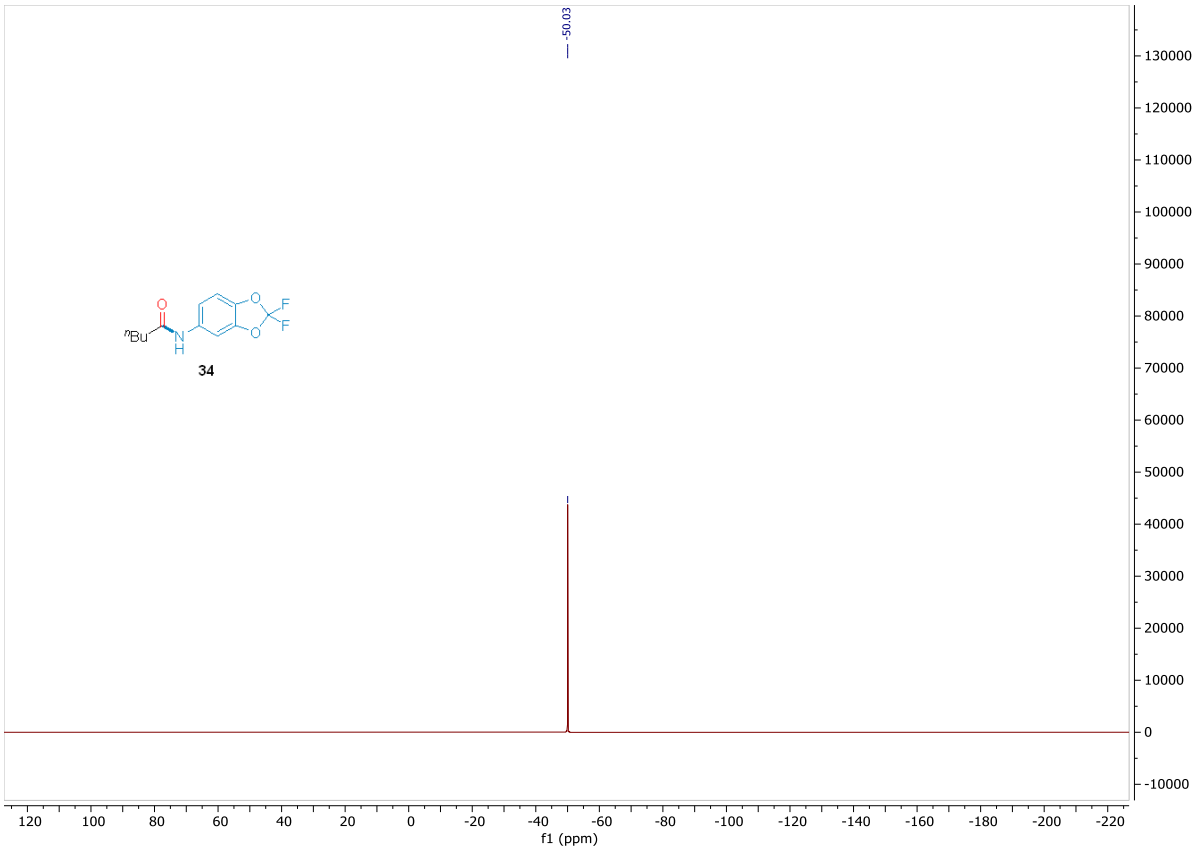

170.93
167.91
134.64
134.58
119.41
119.33
40.36 DMSO
40.08 DMSO
39.80 DMSO
39.52 DMSO
39.24 DMSO
38.96 DMSO
38.69 DMSO
36.04
27.32
23.87
21.85
13.75
NHAc
nBu
N
H
O
32
f1 (ppm)

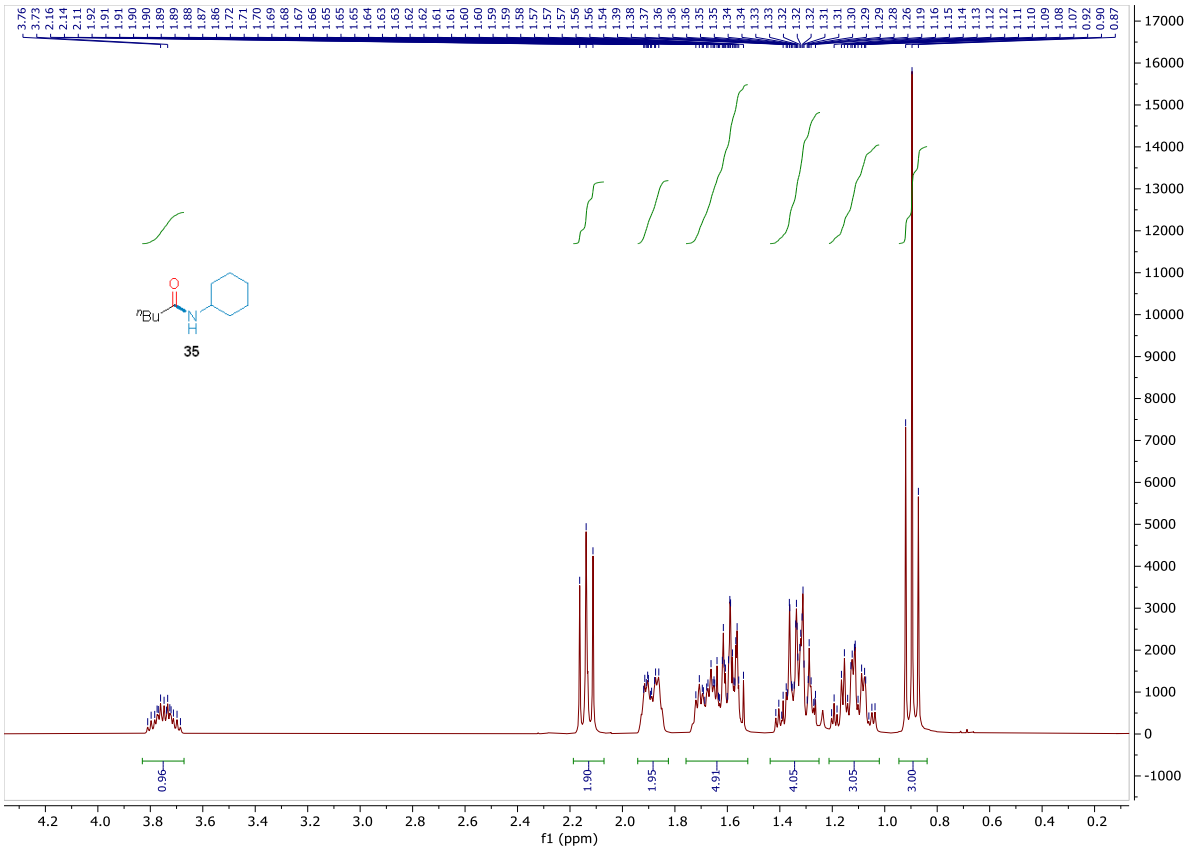

7.86
7.84
7.83
7.82
7.80
7.69
7.66
7.50
7.49
7.48
7.47
7.45
7.42
7.40
2.49
2.47
2.44
1.81
1.79
1.76
1.74
1.71
1.50
1.48
1.45
1.43
1.40
1.38
0.99
0.96
0.94
nBu
33
3.09
1.08
2.15
1.12
2.00
2.01
2.20
3.05
f1 (ppm)

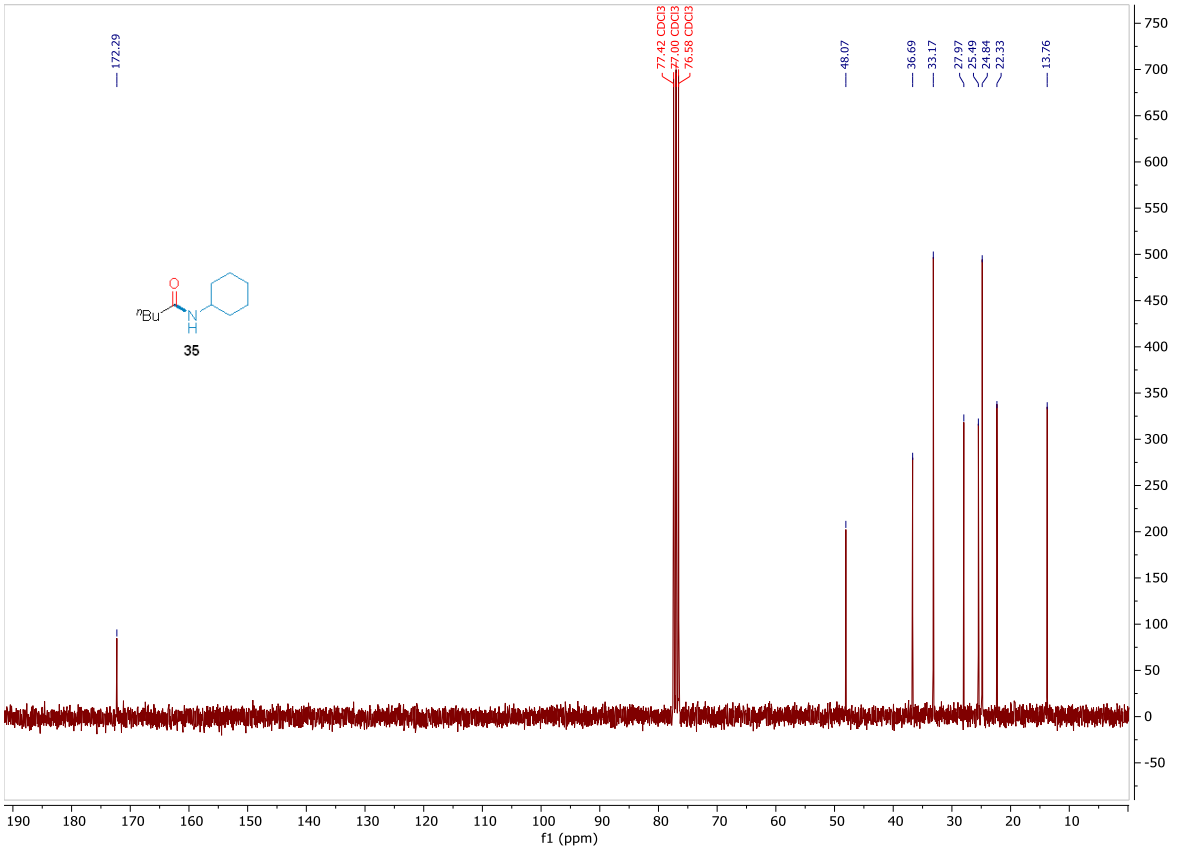

172.18
134.06
132.26
128.64
127.32
126.16
125.88
125.78
125.62
121.26
120.74
77.42 CDCl3
77.00 CDCl3
76.58 CDCl3
37.23
27.90
22.41
13.79
nBu
33
f1 (ppm)

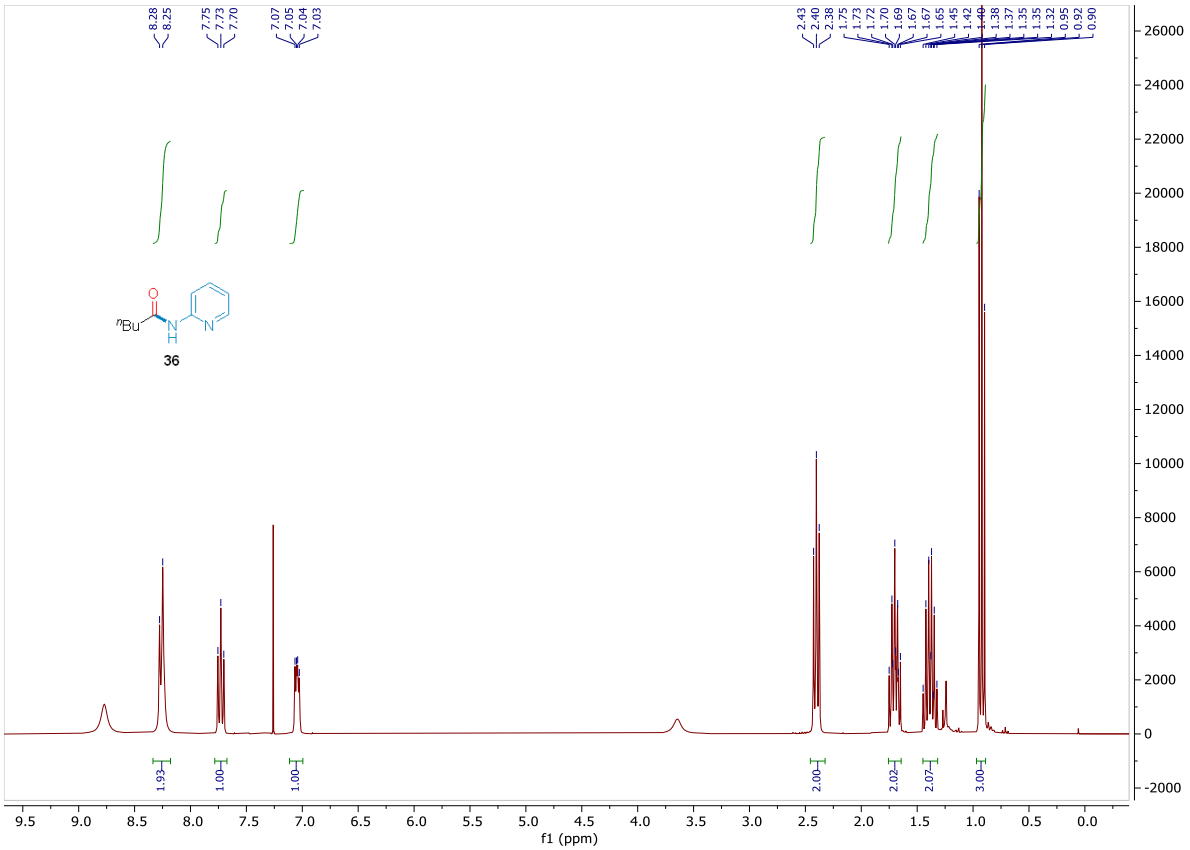

7.58
7.41
7.37
6.96
2.37
2.35
2.32
1.73
1.70
1.68
1.46
1.43
1.41
1.38
1.36
1.33
0.96
0.94
0.91
34
1.00
0.96
2.06
1.92
2.14
2.19
3.22
f1 (ppm)

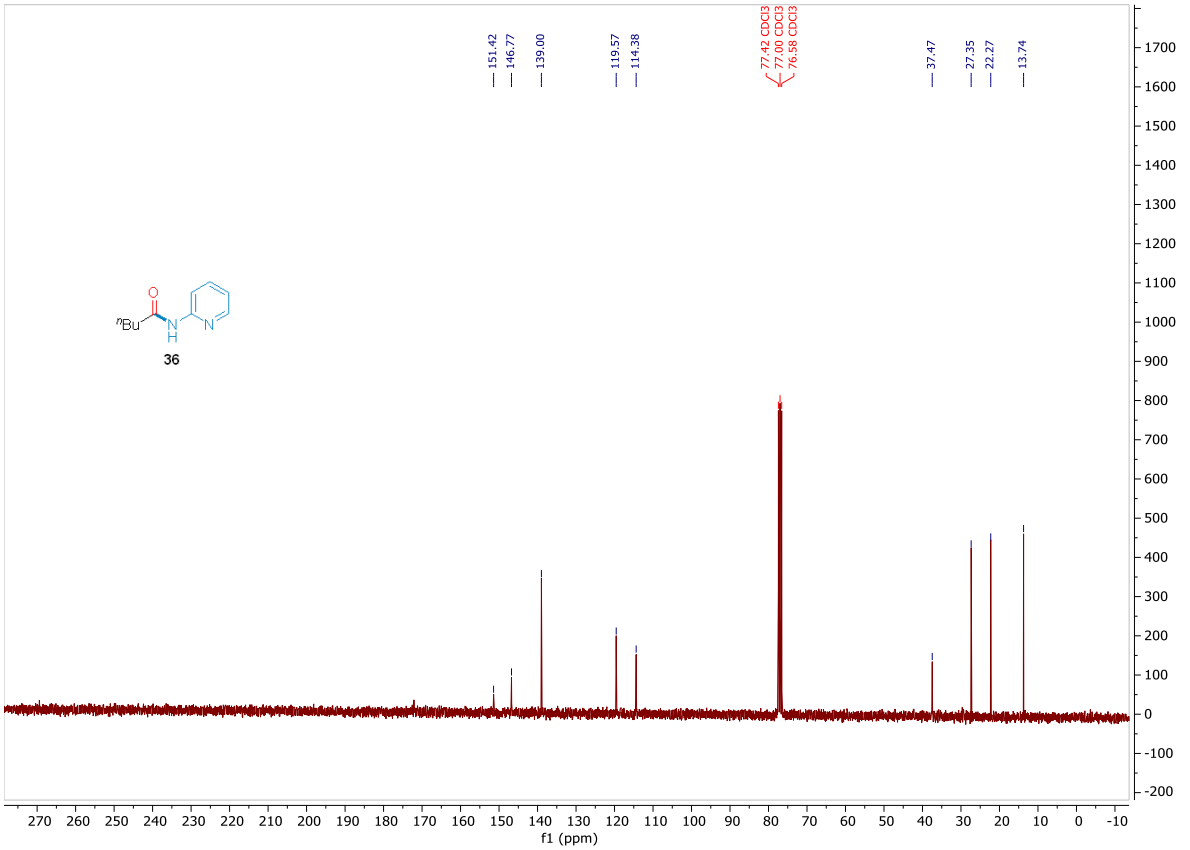

171.61
143.83
140.07
133.99
131.73
114.59
109.17
103.25
77.42 CDCl3
77.00 CDCl3
76.58 CDCl3
37.37
27.59
22.34
13.76
34
f1 (ppm)

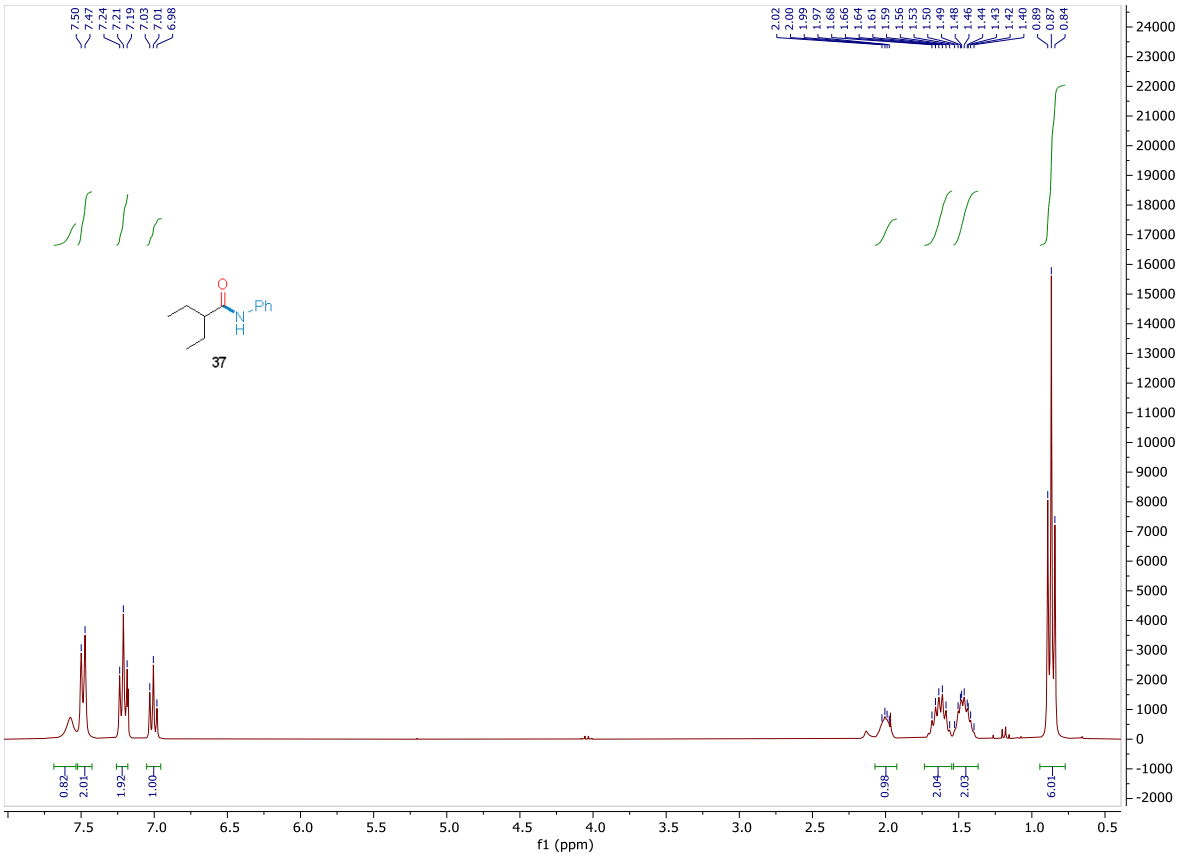

-50.03
O
nBu
N
H
O
O
F
F
34
130000
120000
110000
100000
90000
80000
70000
60000
50000
40000
30000
20000
10000
0
-10000
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-120
-140
-160
-180
-200
-220
f1 (ppm)

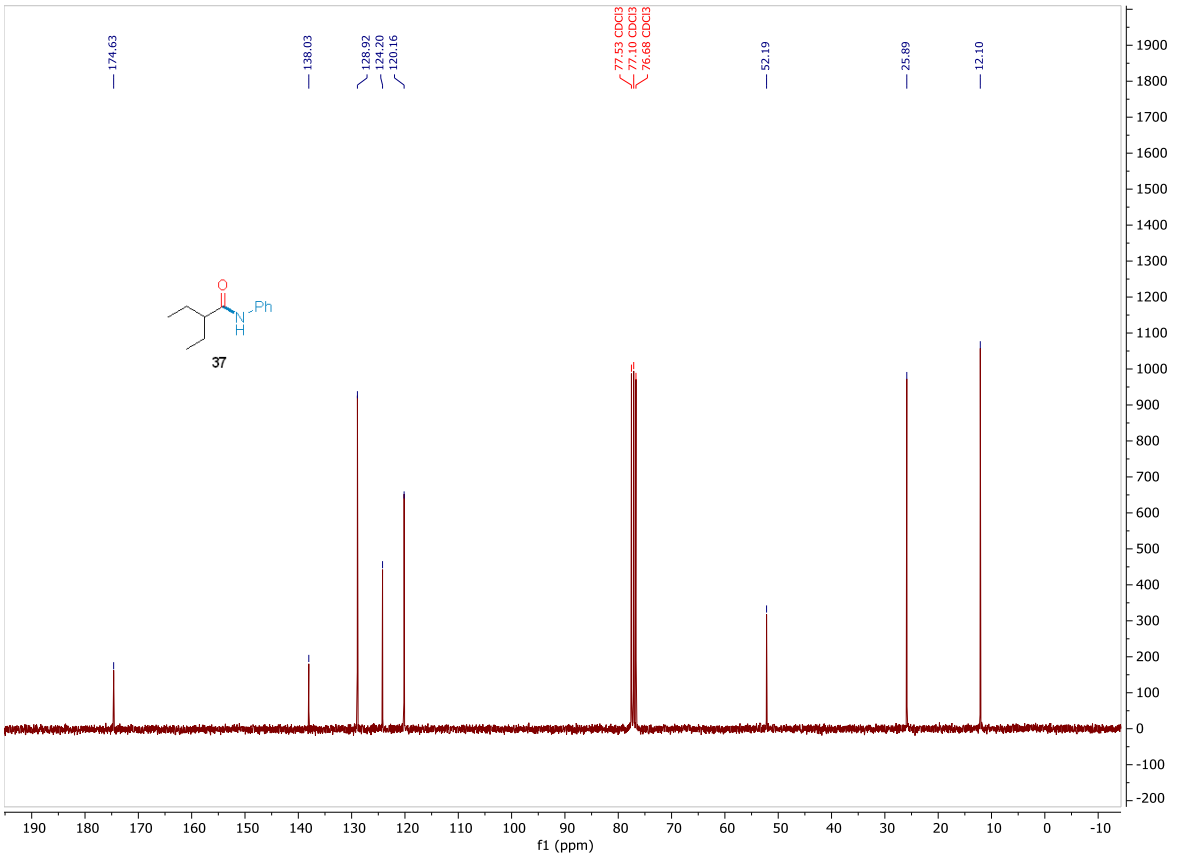

35
f1 (ppm)

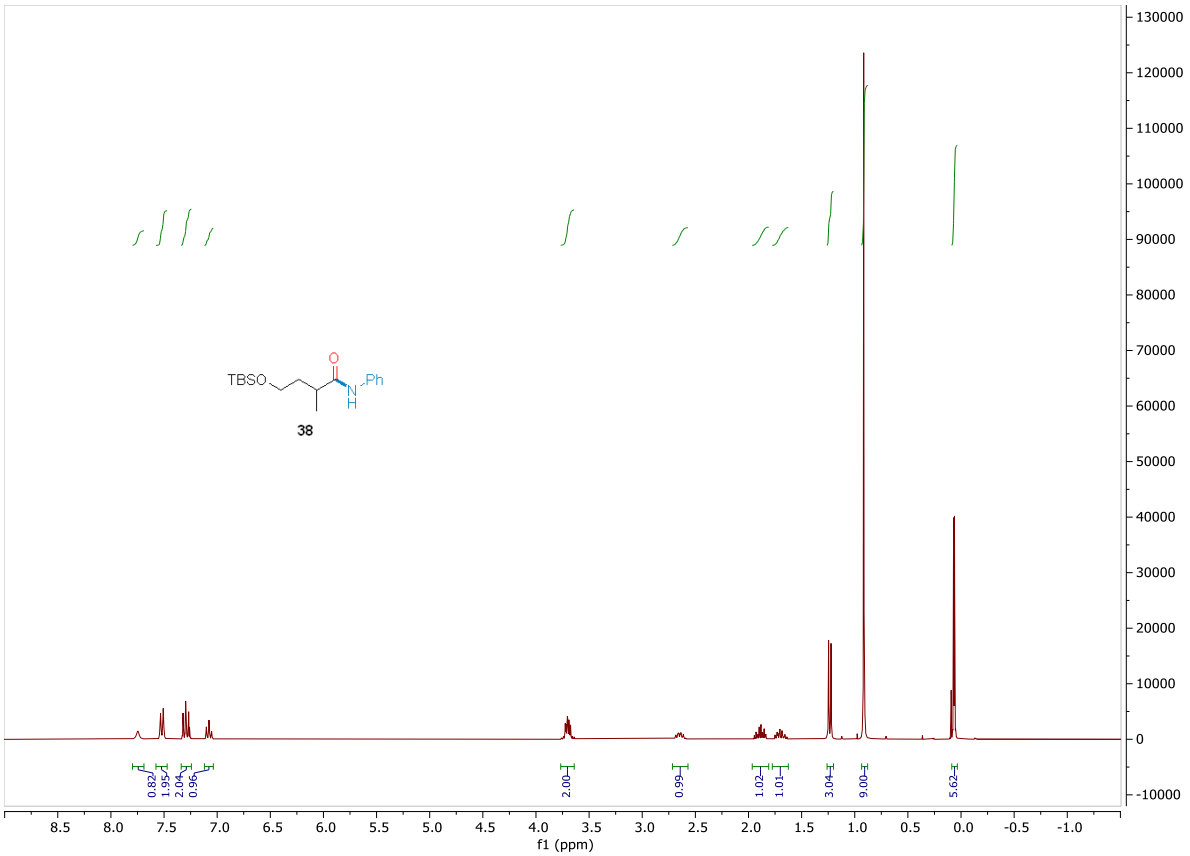

172.29
77.42 CDCl3
77.00 CDCl3
76.58 CDCl3
48.07
36.69
33.17
27.97
25.49
24.84
22.33
13.76
35
f1 (ppm)

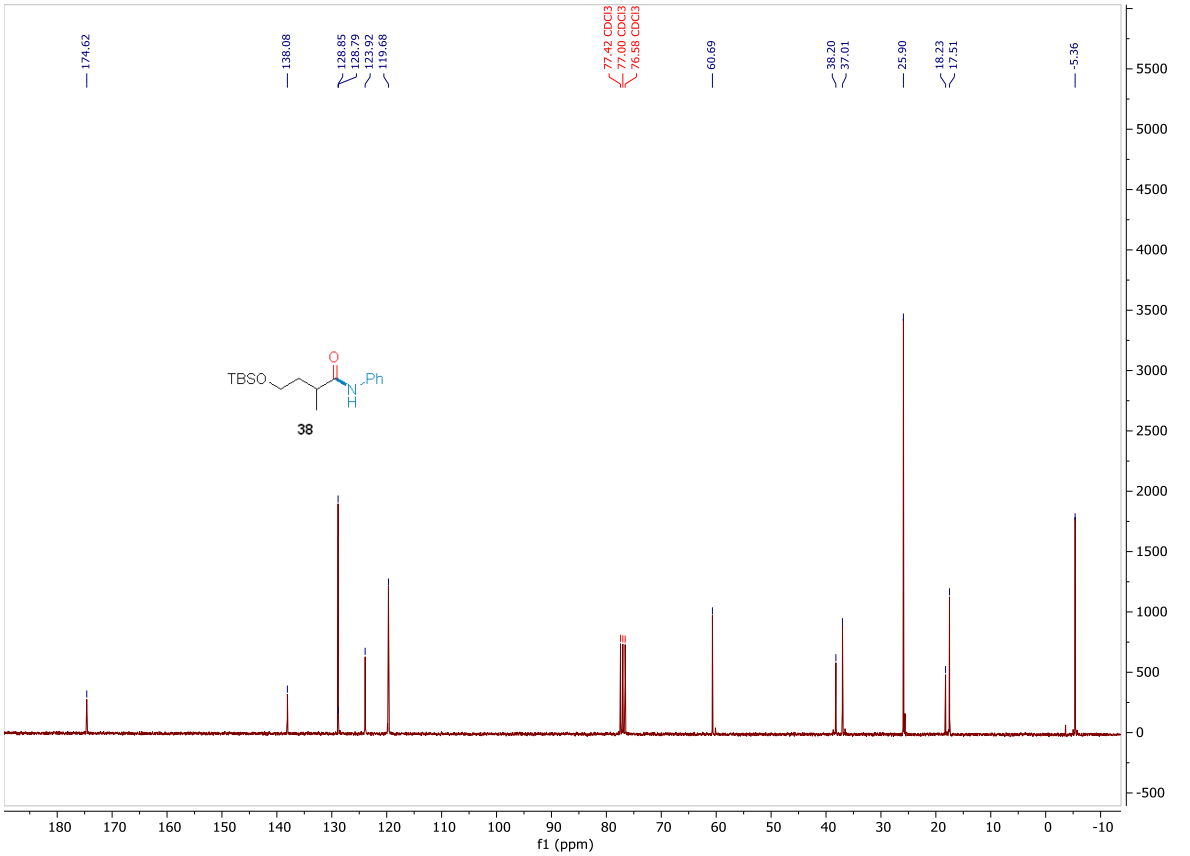

8.28
8.25
7.75
7.73
7.70
7.07
7.05
7.04
7.03
2.43
2.40
2.38
1.75
1.73
1.72
1.70
1.69
1.67
1.67
1.65
1.45
1.42
1.38
1.37
1.35
1.35
1.32
0.95
0.92
0.90
nBu
N
H
O
N
36
1.93
1.00
1.00
2.00
2.02
2.07
3.00
f1 (ppm)

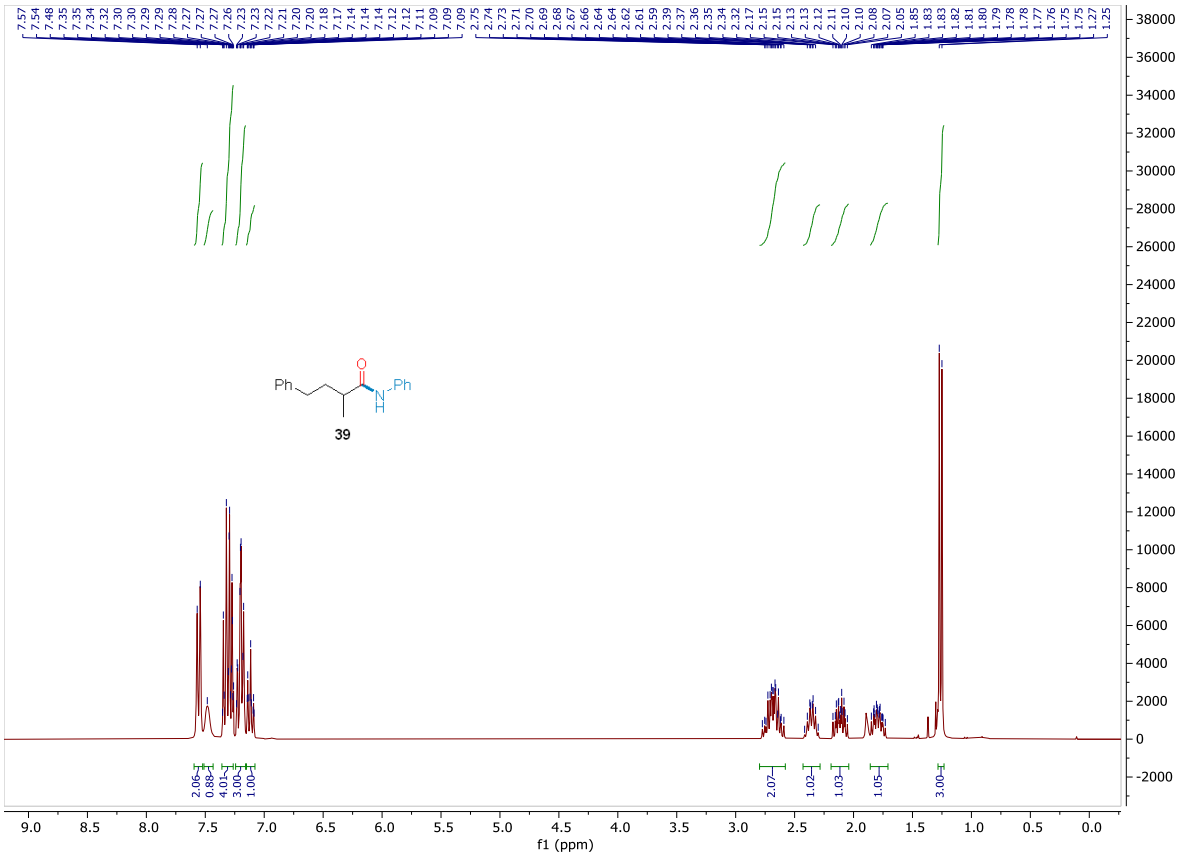

151.42
146.77
139.00
119.57
114.38
77.42 CDCl3
77.00 CDCl3
76.58 CDCl3
37.47
27.35
22.27
13.74
nBu
N
H
O
N
36
f1 (ppm)

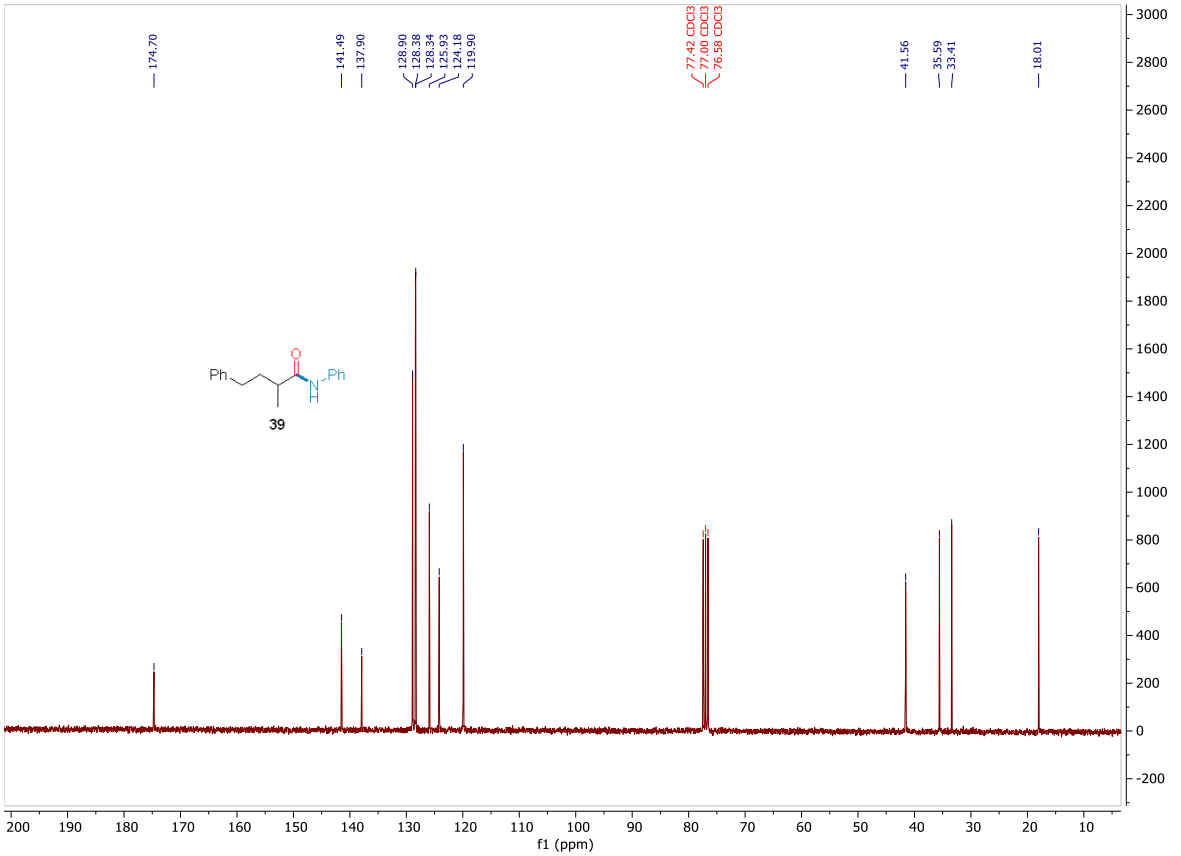

7.50
7.47
7.24
7.21
7.19
7.03
7.01
6.98
2.02
2.00
1.99
1.97
1.68
1.66
1.64
1.61
1.59
1.56
1.53
1.50
1.49
1.48
1.46
1.44
1.43
1.42
1.40
0.89
0.87
0.84
O
N
H
Ph
37
0.82
2.01
1.92
1.00
0.98
2.04
2.03
6.01
f1 (ppm)

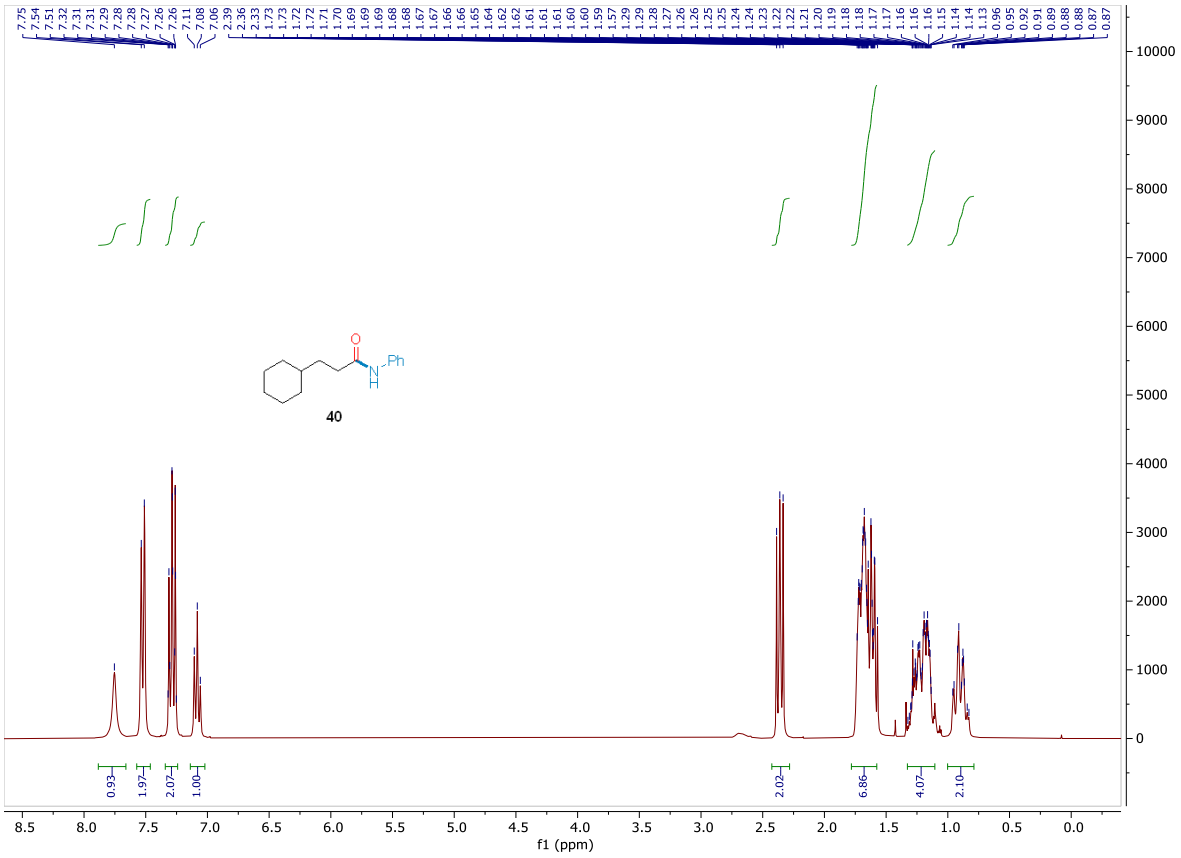

174.63
138.03
128.92
124.20
120.16
77.53 CDCl3
77.10 CDCl3
76.68 CDCl3
52.19
25.89
12.10
O
N
H
Ph
37
f1 (ppm)

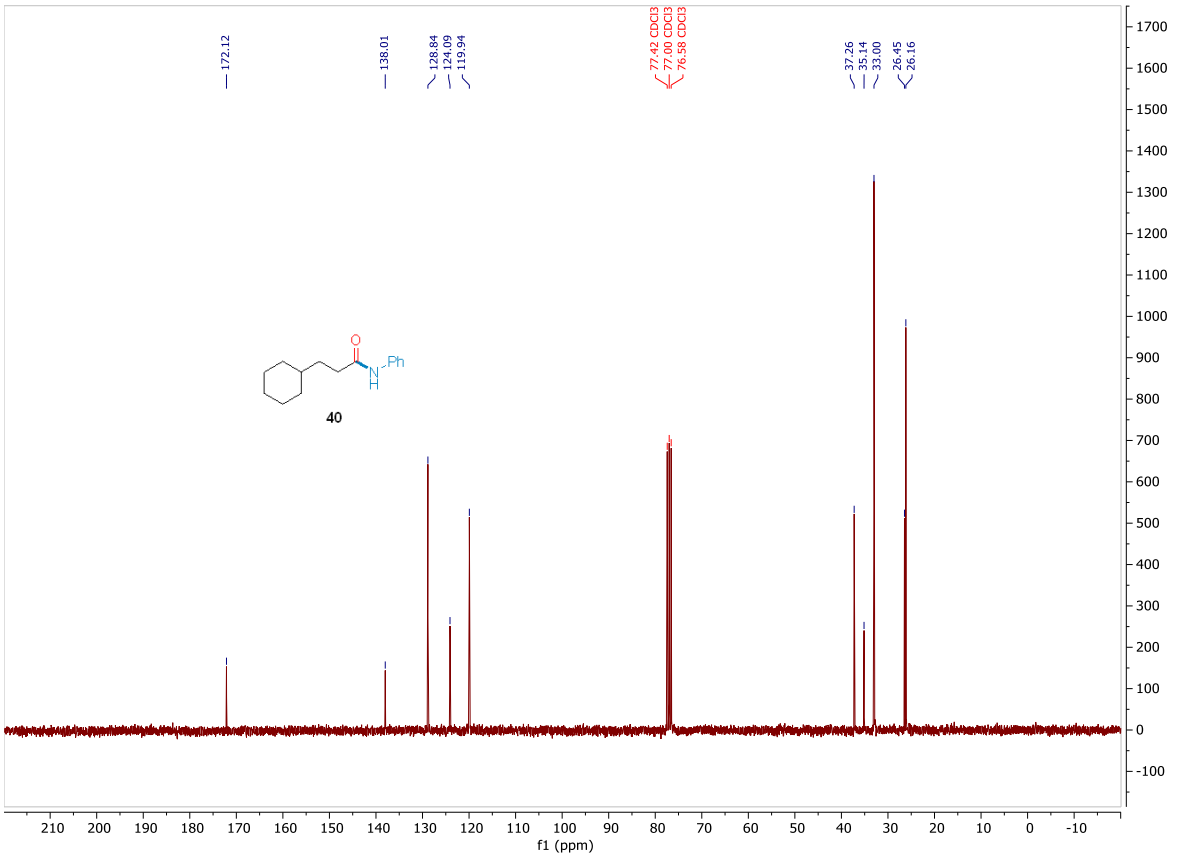

TBSO
O
N
H
Ph
38
0.82
1.95
2.04
0.96
2.00
0.99
1.02
1.01
3.04
9.00
5.62
f1 (ppm)

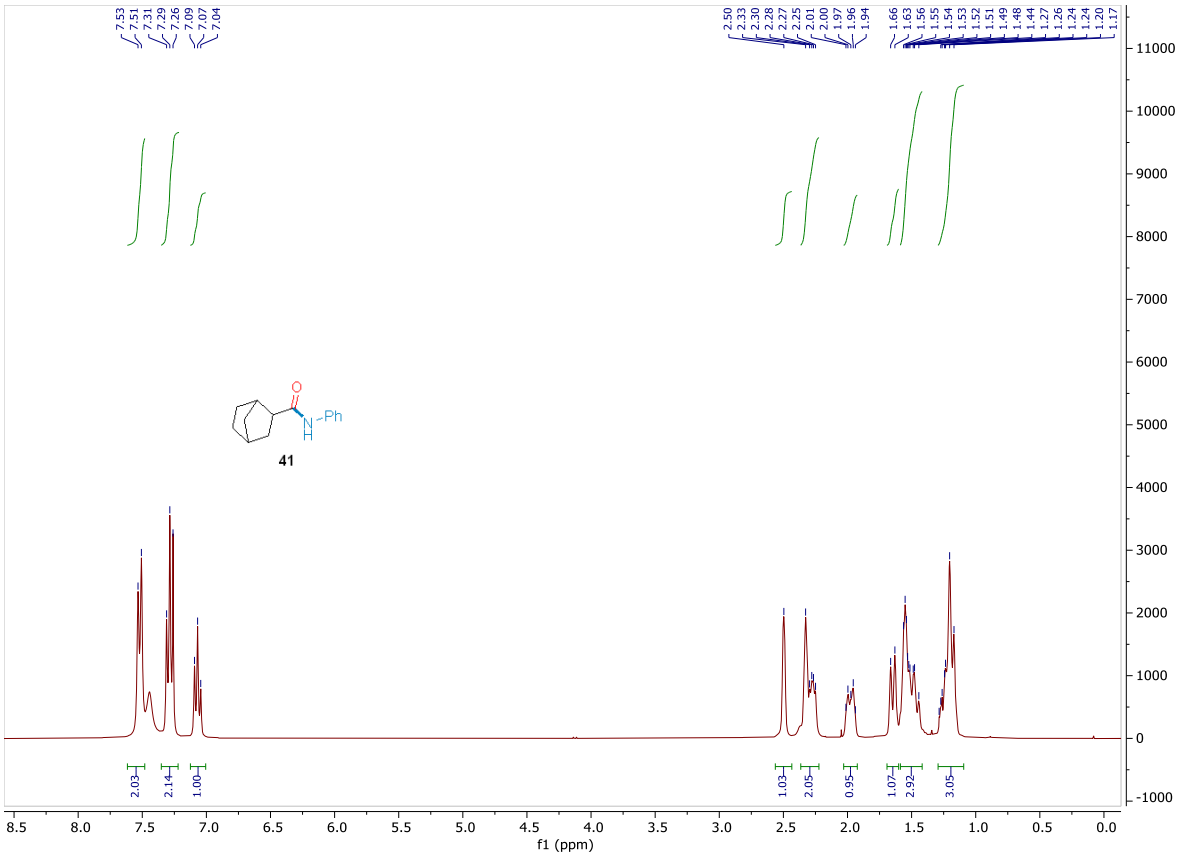

174.62
138.08
128.85
128.79
123.92
119.68
77.42 CDCl3
77.00 CDCl3
76.58 CDCl3
60.69
38.20
37.01
25.90
18.23
17.51
-5.36
TBSO
O
N
H
Ph
38
f1 (ppm)

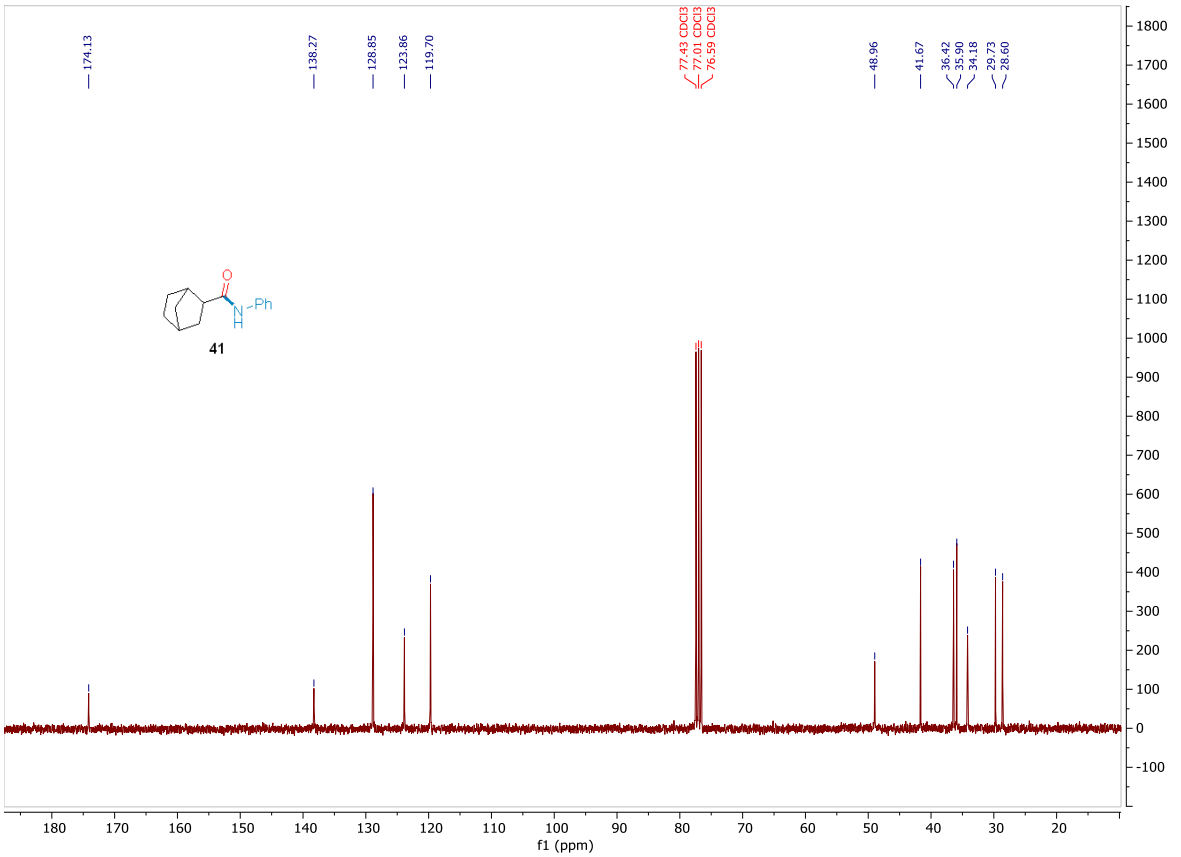

39
f1 (ppm)

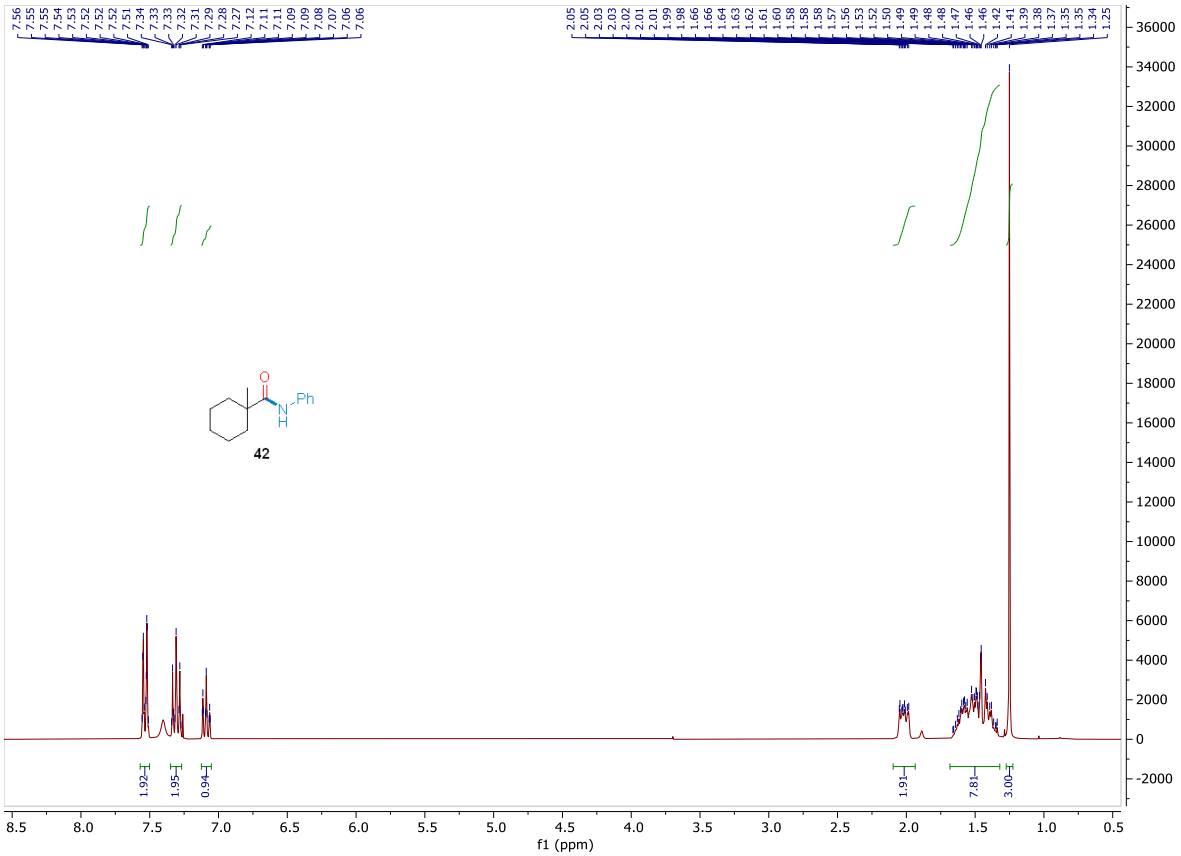

39
f1 (ppm)

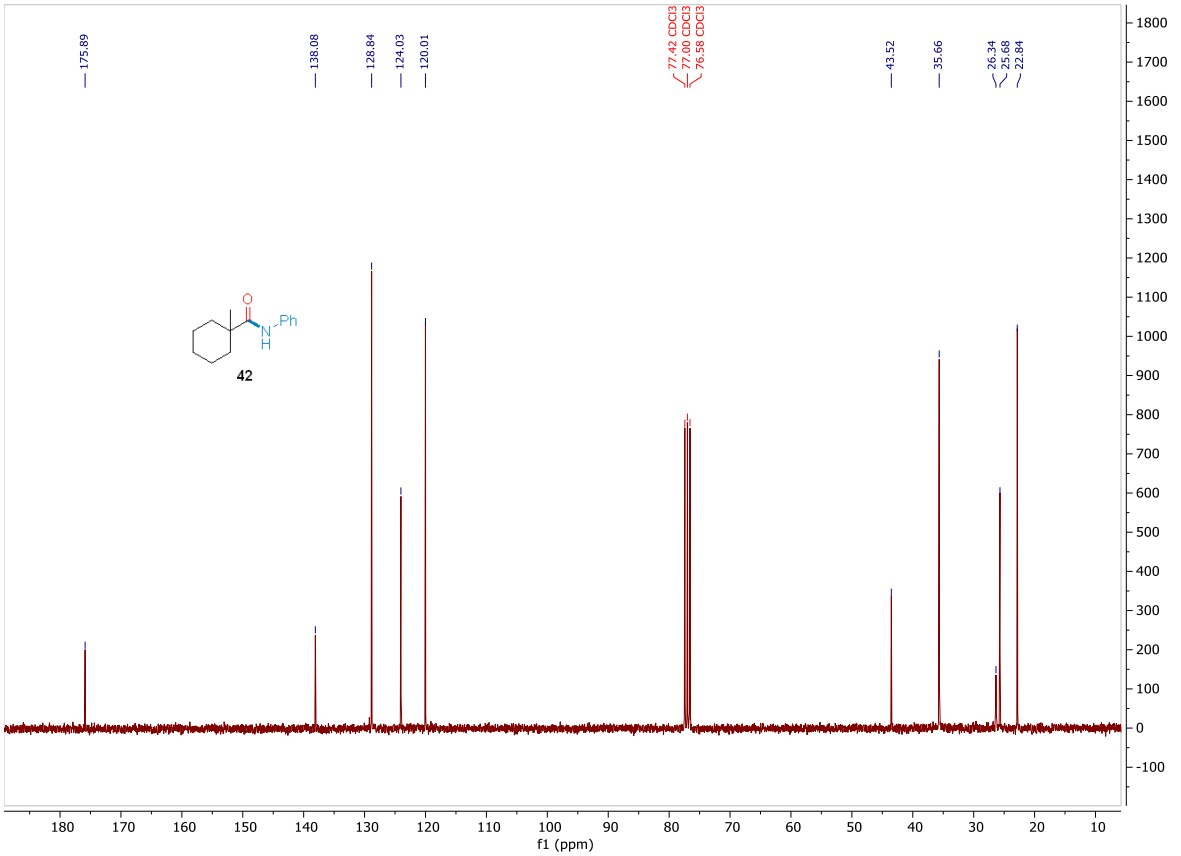

7.75 7.54 7.51 7.32 7.31 7.31 7.29 7.28 7.28 7.27 7.26 7.26 7.11 7.08 7.06 2.39 2.36 2.33 1.73 1.72 1.72 1.71 1.70 1.69 1.69 1.69 1.68 1.68 1.67 1.67 1.66 1.66 1.65 1.64 1.62 1.62 1.61 1.61 1.61 1.60 1.60 1.59 1.57 1.29 1.29 1.28 1.27 1.26 1.26 1.25 1.25 1.24 1.24 1.23 1.22 1.22 1.21 1.20 1.19 1.18 1.18 1.17 1.17 1.16 1.16 1.16 1.15 1.14 1.14 1.13 0.96 0.95 0.92 0.91 0.89 0.88 0.88 0.87 0.87
O
N
H
Ph
40
0.93
1.97
2.07
1.00
2.02
6.86
4.07
2.10
f1 (ppm)

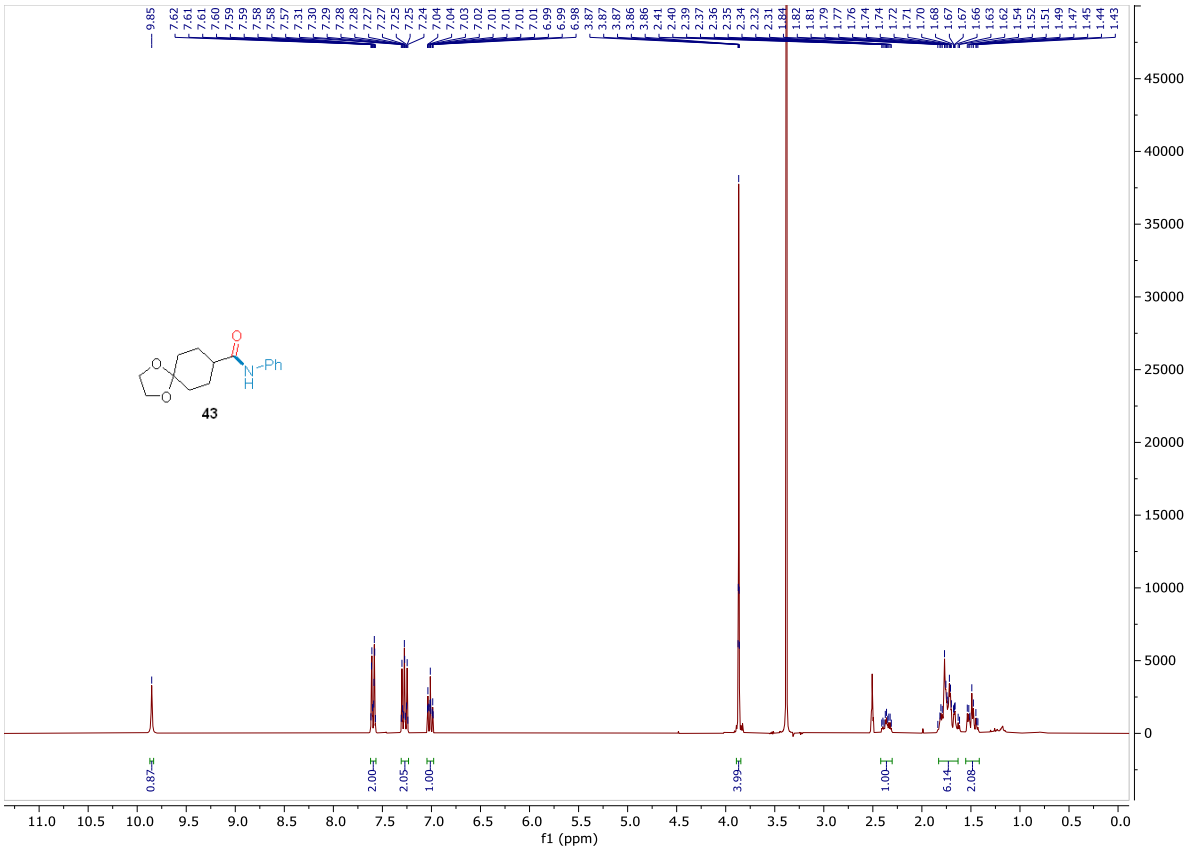

172.12
138.01
128.84
124.09
119.94
77.42 CDCl3
77.00 CDCl3
76.58 CDCl3
37.26
35.14
33.00
26.45
26.16
O
N
H
Ph
40
f1 (ppm)

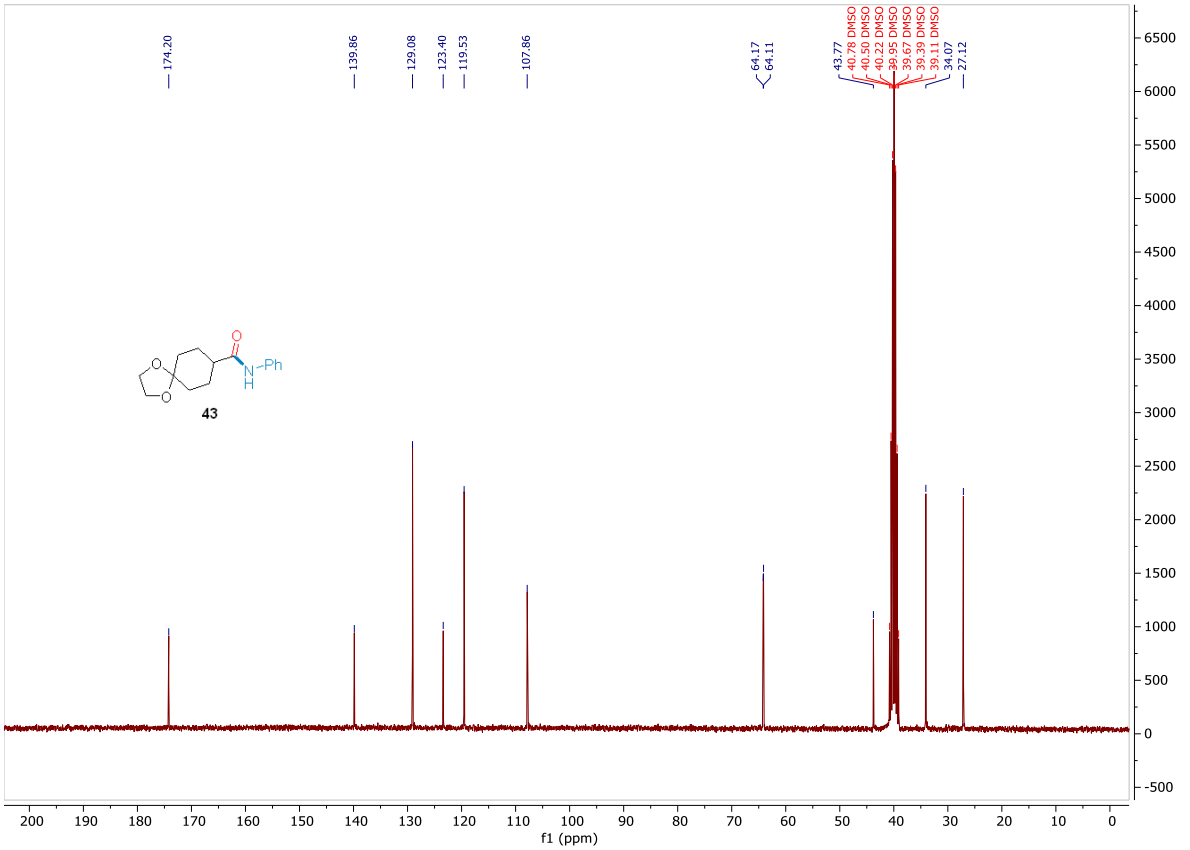

7.53
7.51
7.31
7.29
7.26
7.09
7.07
7.04
2.50
2.33
2.30
2.28
2.27
2.25
2.01
2.00
1.97
1.96
1.94
1.66
1.63
1.56
1.55
1.54
1.53
1.52
1.51
1.49
1.48
1.44
1.27
1.26
1.24
1.24
1.20
1.17
O
N
H
Ph
41
2.03
2.14
1.00
1.03
2.05
0.95
1.07
2.92
3.05
f1 (ppm)

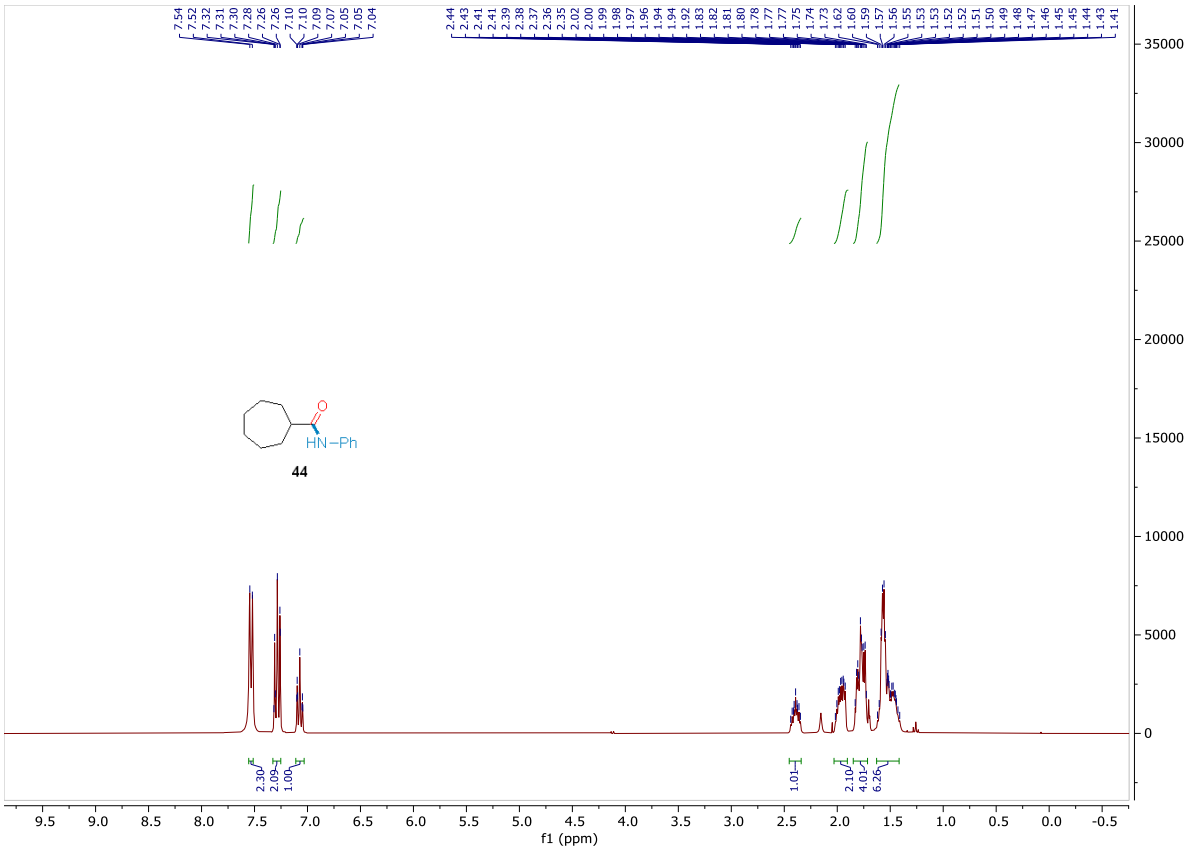

174.13
138.27
128.85
123.86
119.70
77.43 CDCl3
77.01 CDCl3
76.59 CDCl3
48.96
41.67
36.42
35.90
34.18
29.73
28.60
O
N
H
Ph
41
f1 (ppm)

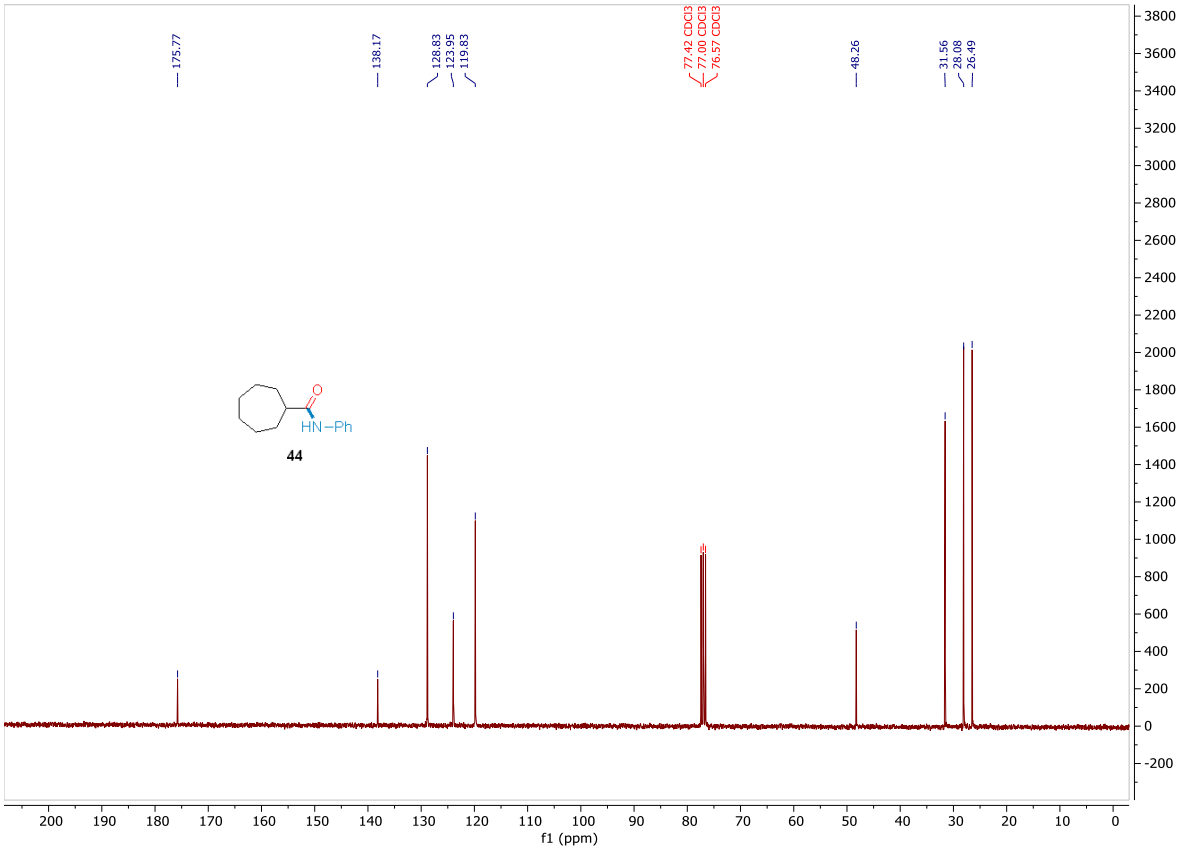

7.56 7.55 7.55 7.54 7.53 7.52 7.52 7.52 7.51 7.34 7.33 7.33 7.32 7.31 7.29 7.28 7.27 7.12 7.11 7.11 7.09 7.09 7.08 7.07 7.06 7.06
2.05 2.05 2.03 2.03 2.02 2.01 2.01 1.99 1.98 1.66 1.66 1.64 1.63 1.62 1.61 1.60 1.58 1.58 1.58 1.57 1.56 1.53 1.52 1.50 1.49 1.49 1.48 1.48 1.47 1.46 1.46 1.42 1.41 1.39 1.38 1.37 1.35 1.35 1.34 1.25
O
N
H
Ph
42
1.92
1.95
0.94
1.91
7.81
3.00
f1 (ppm)


175.89
138.08
128.84
124.03
120.01
77.42 CDCl3
77.00 CDCl3
76.58 CDCl3
43.52
35.66
26.34
25.68
22.84
O
N
H
Ph
42
f1 (ppm)


9.85
7.62
7.61
7.61
7.60
7.59
7.59
7.58
7.58
7.57
7.31
7.30
7.29
7.28
7.28
7.27
7.27
7.25
7.25
7.24
7.04
7.04
7.03
7.02
7.01
7.01
7.01
7.01
6.99
6.99
6.98
3.87
3.87
3.87
3.86
3.86
2.41
2.40
2.39
2.37
2.36
2.35
2.34
2.32
2.31
1.84
1.82
1.81
1.79
1.77
1.76
1.74
1.74
1.72
1.71
1.70
1.68
1.67
1.67
1.66
1.65
1.62
1.54
1.52
1.51
1.49
1.47
1.45
1.44
1.43
O
N
H
Ph
43
0.87
2.00
2.05
1.00
3.99
1.00
6.14
2.08
f1 (ppm)


174.20
139.86
129.08
123.40
119.53
107.86
64.17
64.11
43.77
40.78 DMSO
40.50 DMSO
40.22 DMSO
39.95 DMSO
39.67 DMSO
39.39 DMSO
39.11 DMSO
34.07
27.12
O
N
H
Ph
43
f1 (ppm)


HN–Ph
44
f1 (ppm)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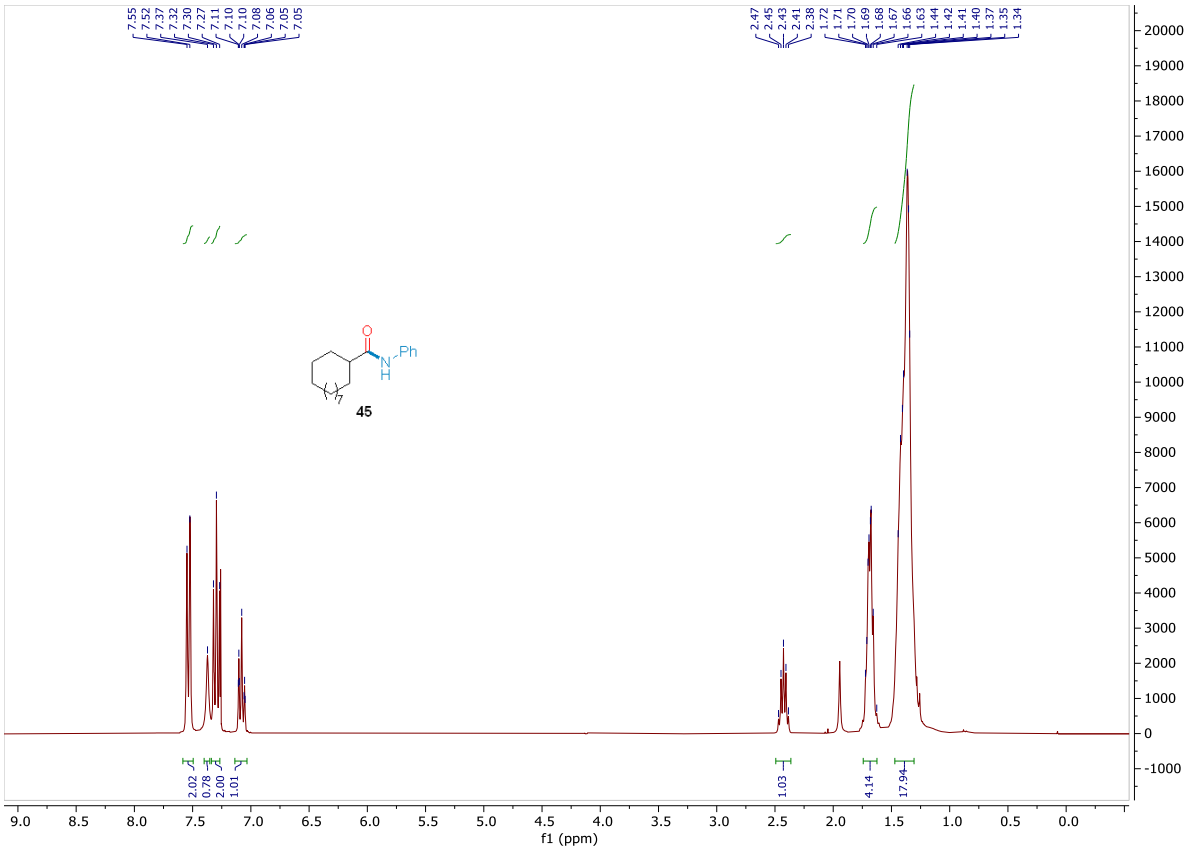
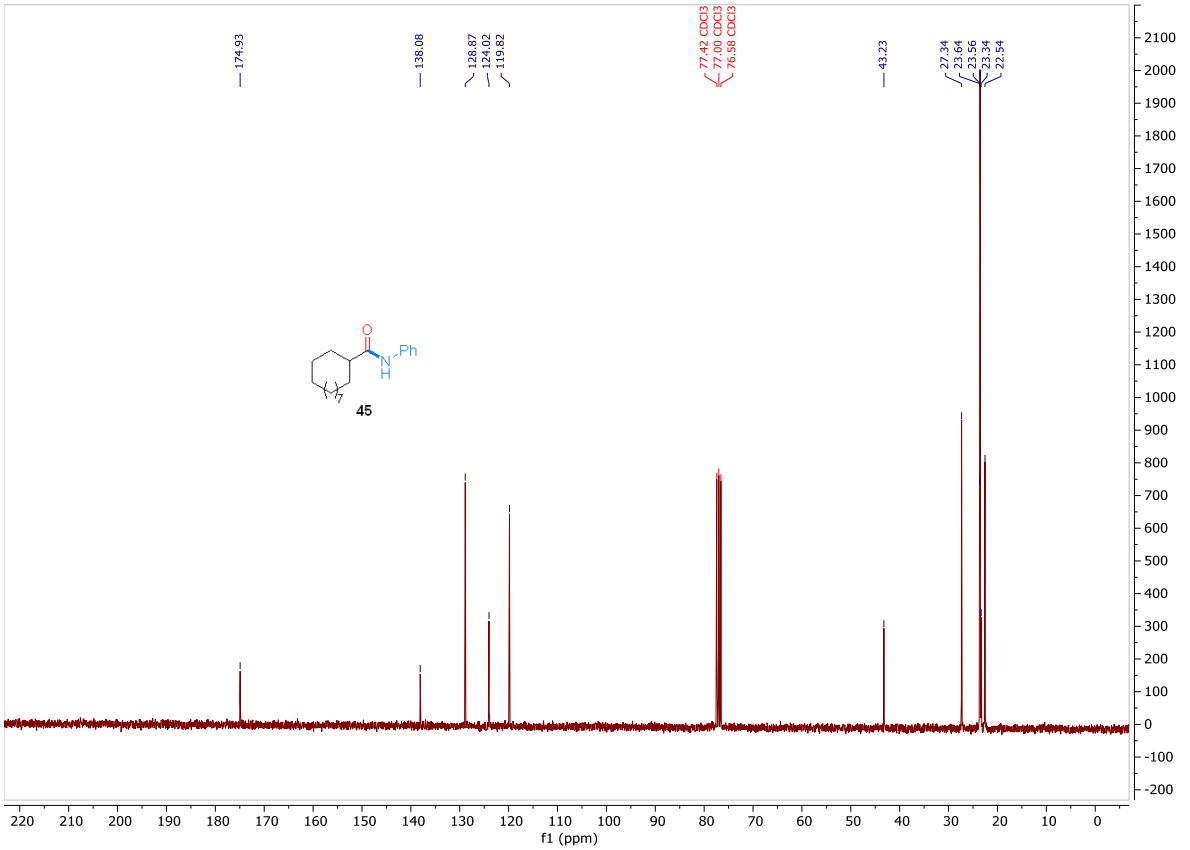
138.17
128.83
123.95
119.83
77.42 CDCl3
77.00 CDCl3
76.57 CDCl3
48.26
31.56
28.08
26.49
HN–Ph
44
f1 (ppm)


7.55
7.52
7.37
7.32
7.30
7.27
7.11
7.10
7.08
7.06
7.05
7.05
2.47
2.45
2.43
2.41
2.38
1.72
1.71
1.70
1.69
1.68
1.67
1.66
1.63
1.44
1.42
1.41
1.40
1.37
1.35
1.34
O
N
H
Ph
45
2.02
0.78
2.00
1.01
1.03
4.14
17.94
f1 (ppm)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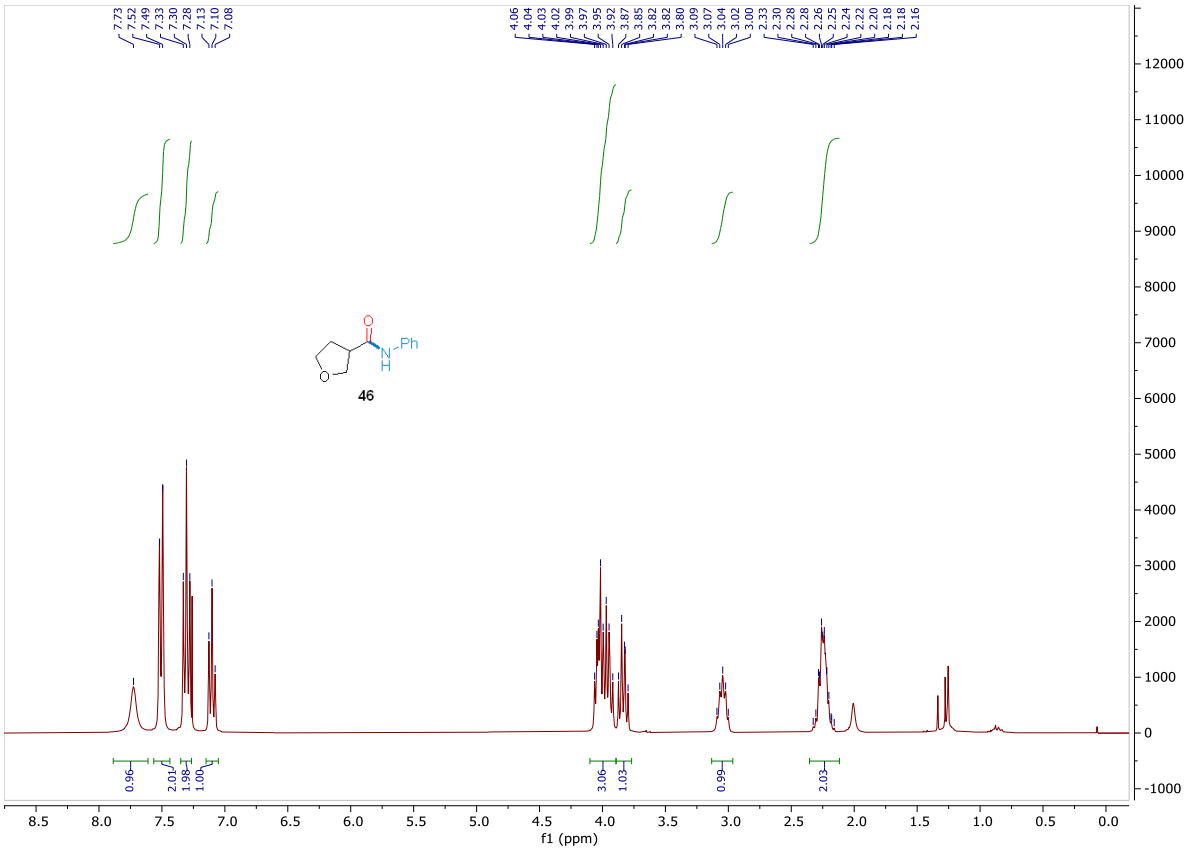
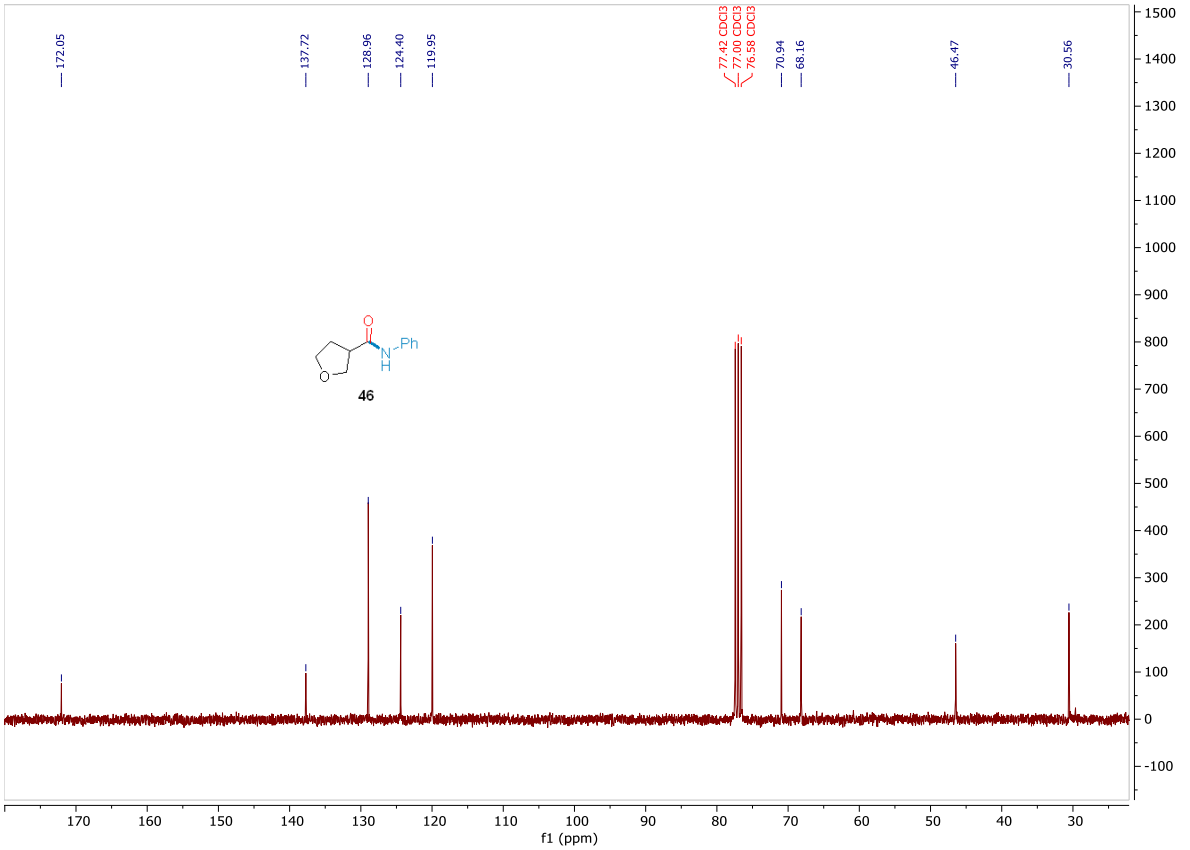
138.08
128.87
124.02
119.82
77.42 CDCl3
77.00 CDCl3
76.58 CDCl3
43.23
27.34
23.64
23.56
23.34
22.54
O
N
H
Ph
45
f1 (ppm)


7.73
7.52
7.49
7.33
7.30
7.28
7.13
7.10
7.08
4.06
4.04
4.03
4.02
3.99
3.97
3.95
3.92
3.87
3.85
3.82
3.82
3.80
3.09
3.07
3.04
3.02
3.00
2.33
2.30
2.28
2.28
2.26
2.25
2.24
2.22
2.20
2.18
2.18
2.16
O
N
H
Ph
46
0.96
2.01
1.98
1.00
3.06
1.03
0.99
2.03
f1 (ppm)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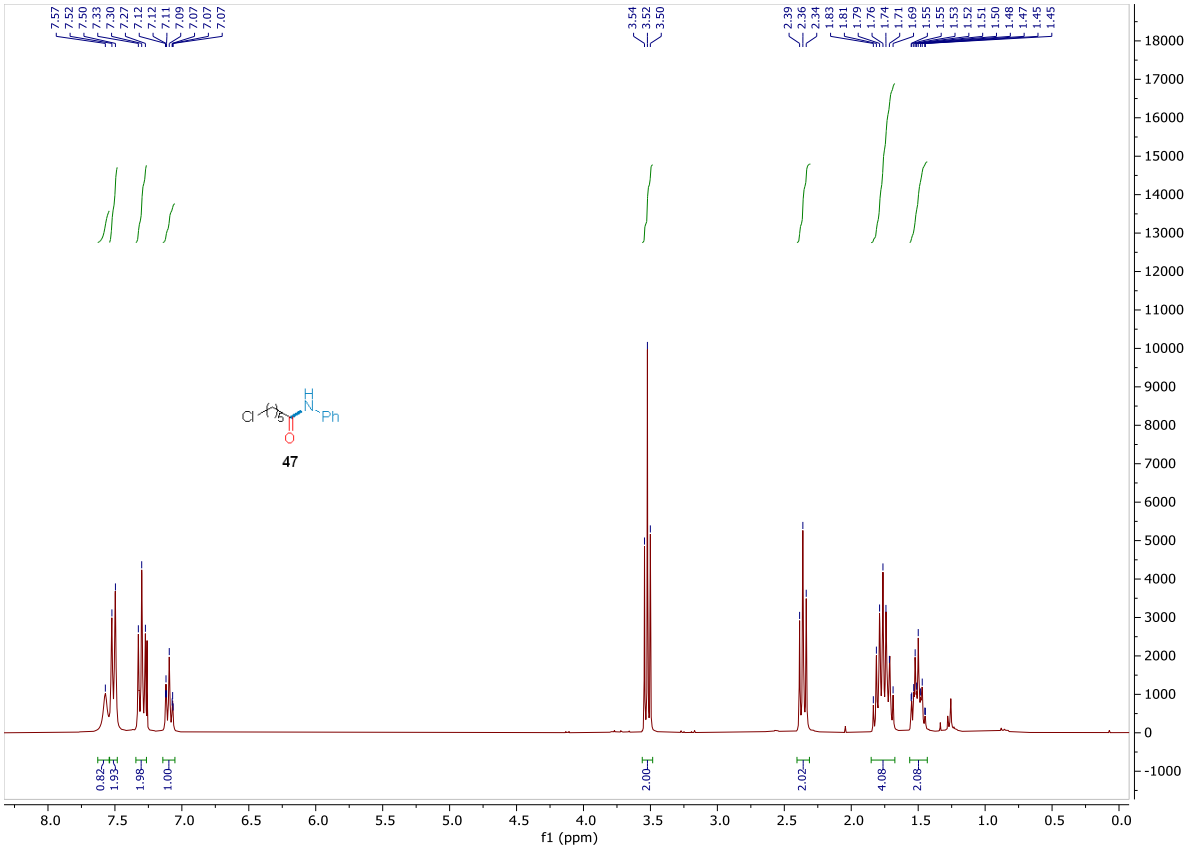
137.72
128.96
124.40
119.95
77.42 CDCl3
77.00 CDCl3
76.58 CDCl3
70.94
68.16
46.47
30.56
O
N
H
Ph
46
f1 (ppm)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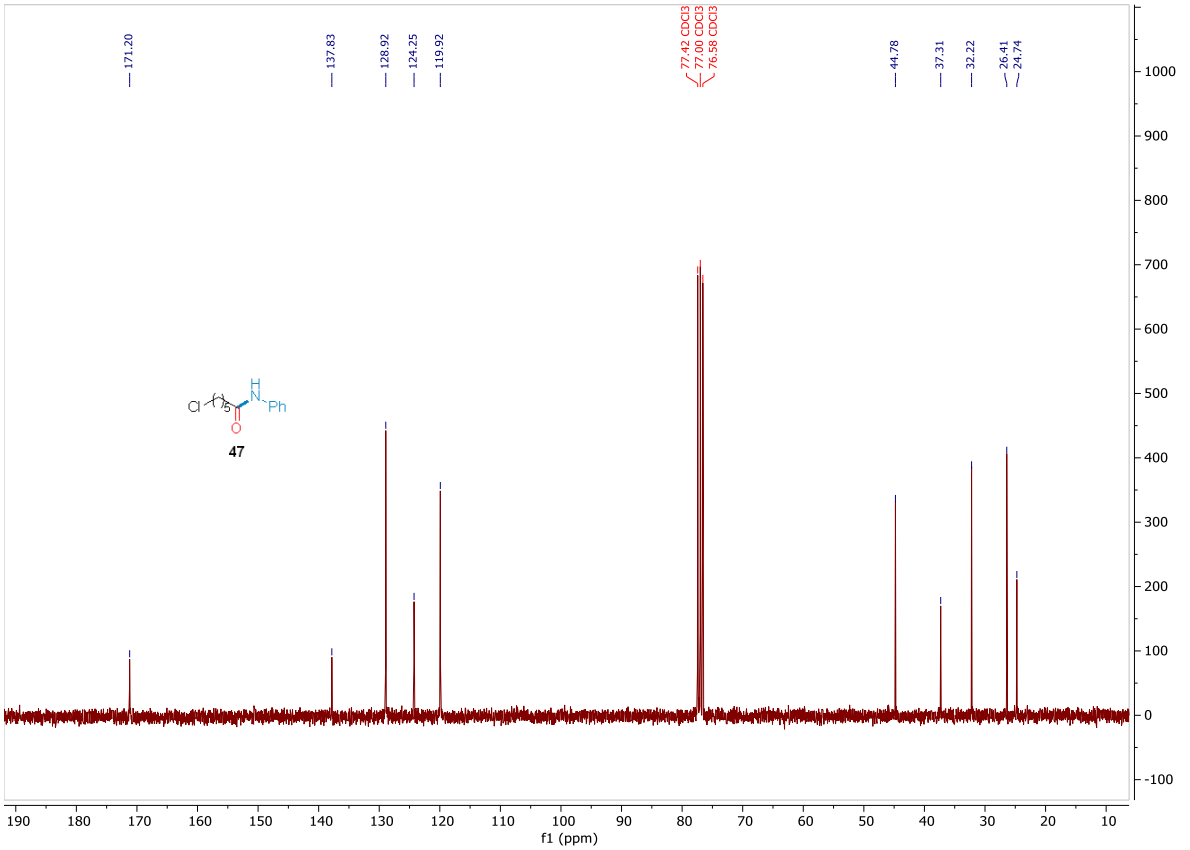
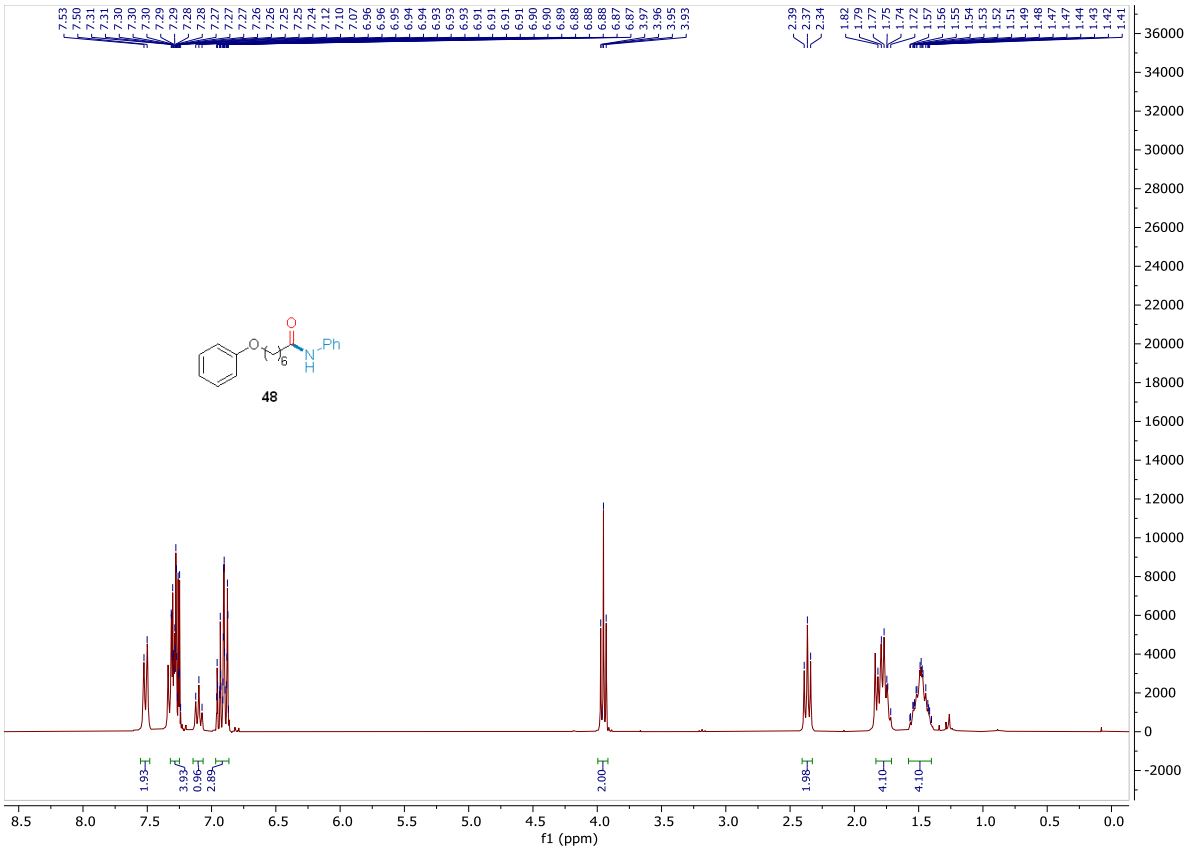
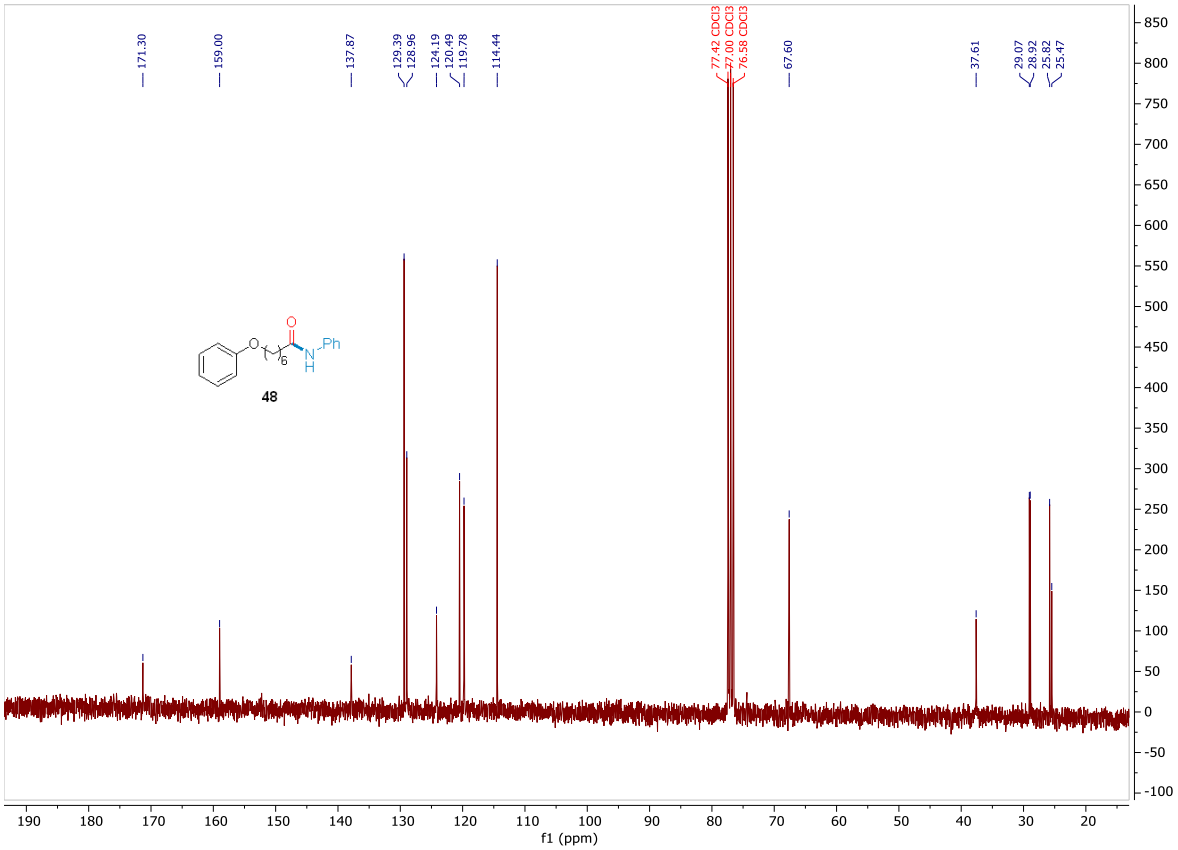
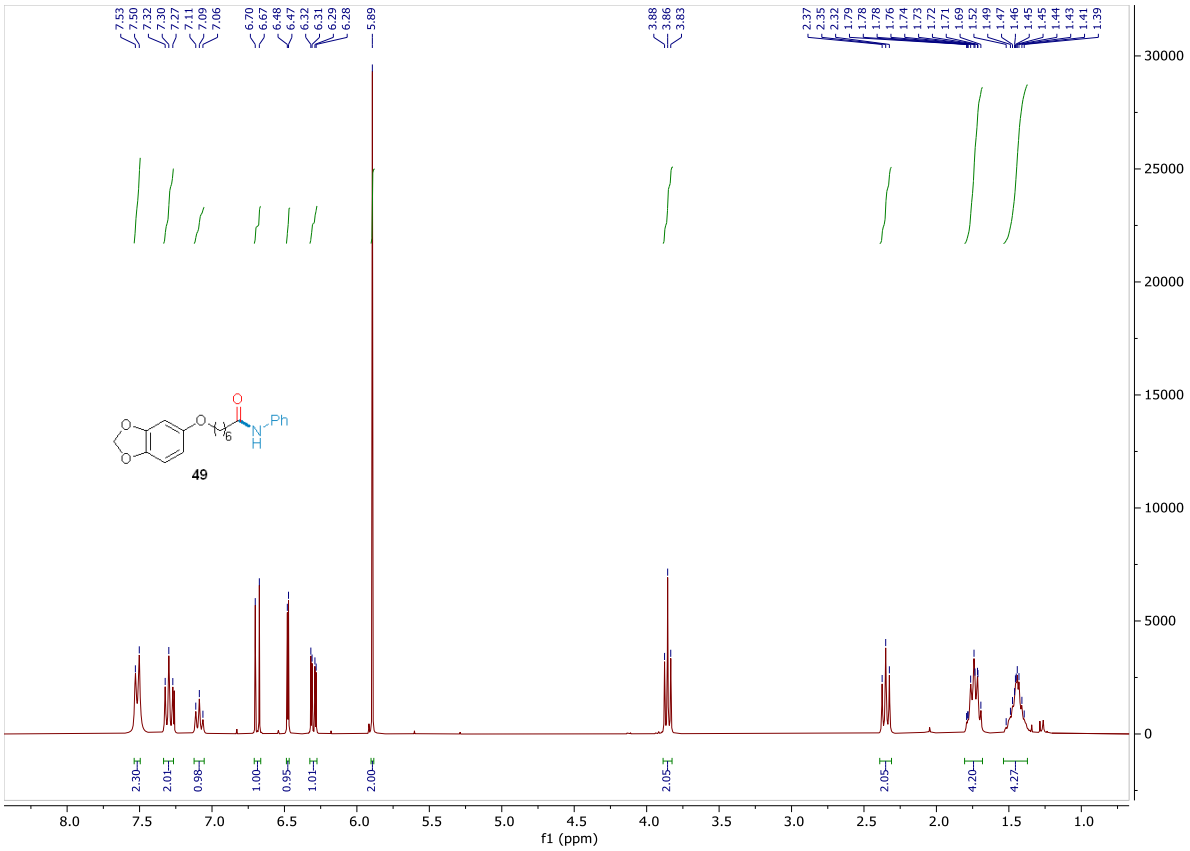
7.52
7.50
7.33
7.30
7.27
7.12
7.11
7.09
7.07
7.07
7.07
3.54
3.52
3.50
2.39
2.36
2.34
1.83
1.81
1.79
1.76
1.74
1.71
1.69
1.55
1.55
1.53
1.52
1.51
1.50
1.48
1.47
1.45
1.45
Cl
Ph
O
47
0.82
1.93
1.98
1.00
2.00
2.02
4.08
2.08
f1 (ppm)


171.20
137.83
128.92
124.25
119.92
77.42 CDCl3
77.00 CDCl3
76.58 CDCl3
44.78
37.31
32.22
26.41
24.74
Cl
Ph
O
47
f1 (ppm)


48
f1 (ppm)


48
f1 (ppm)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7.50
7.32
7.30
7.27
7.11
7.09
7.06
6.70
6.67
6.48
6.47
6.32
6.31
6.29
6.28
5.89
3.88
3.86
3.83
2.37
2.35
2.32
2.19
2.18
2.18
2.16
2.14
2.13
2.12
2.11
2.09
2.08
2.07
2.06
2.05
2.04
2.03
2.02
2.01
2.00
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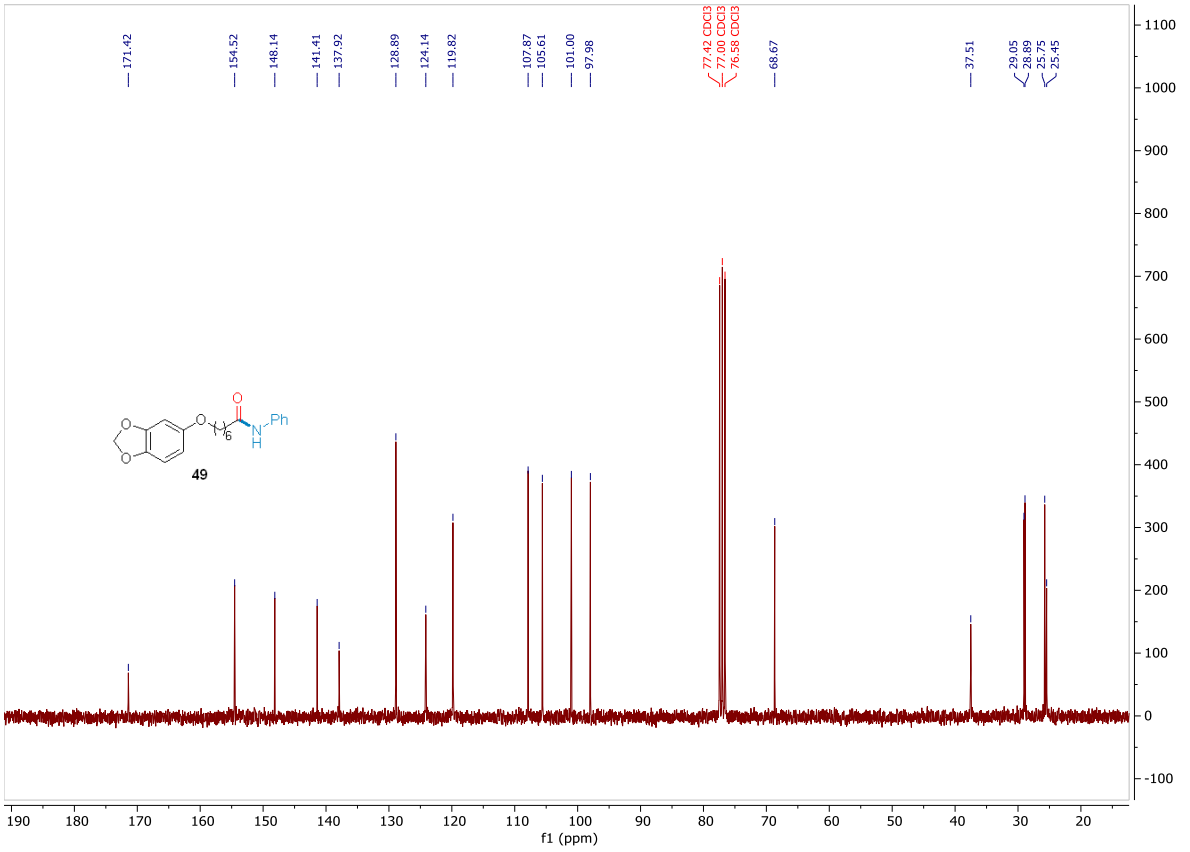
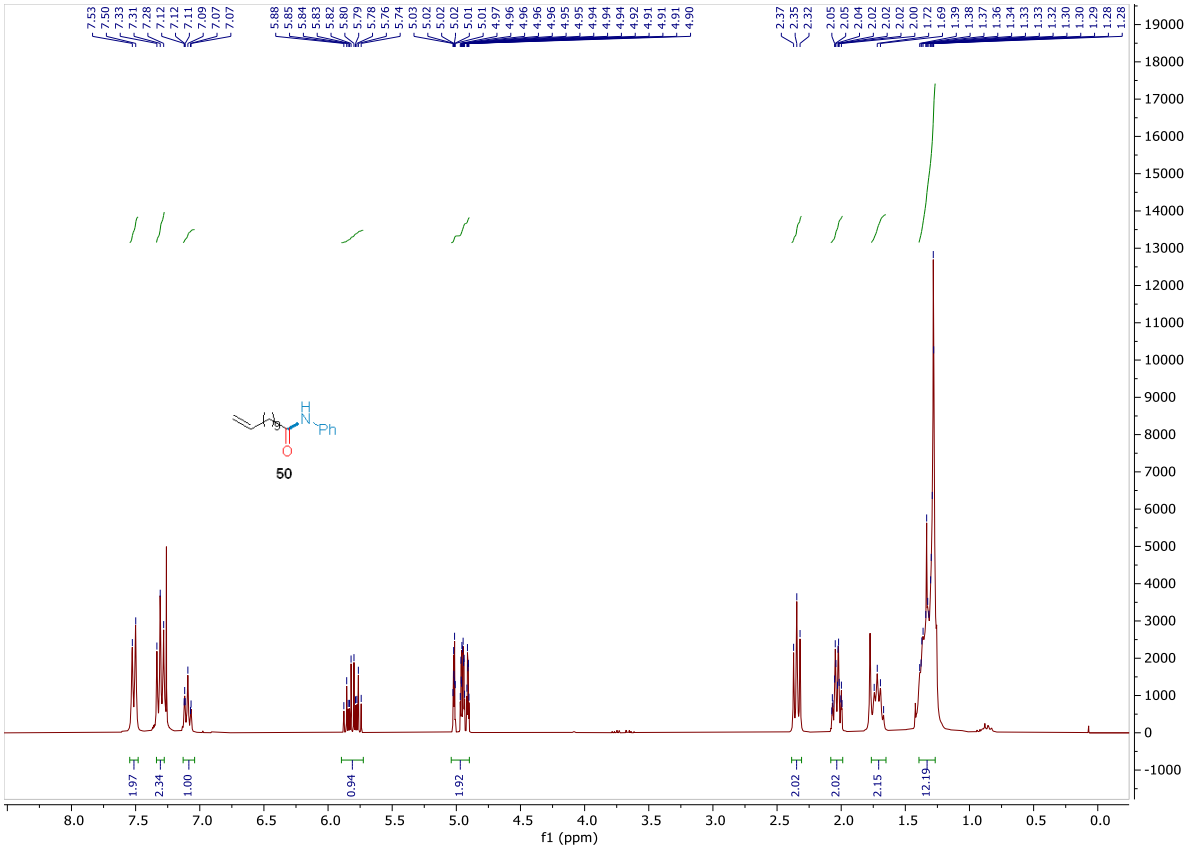
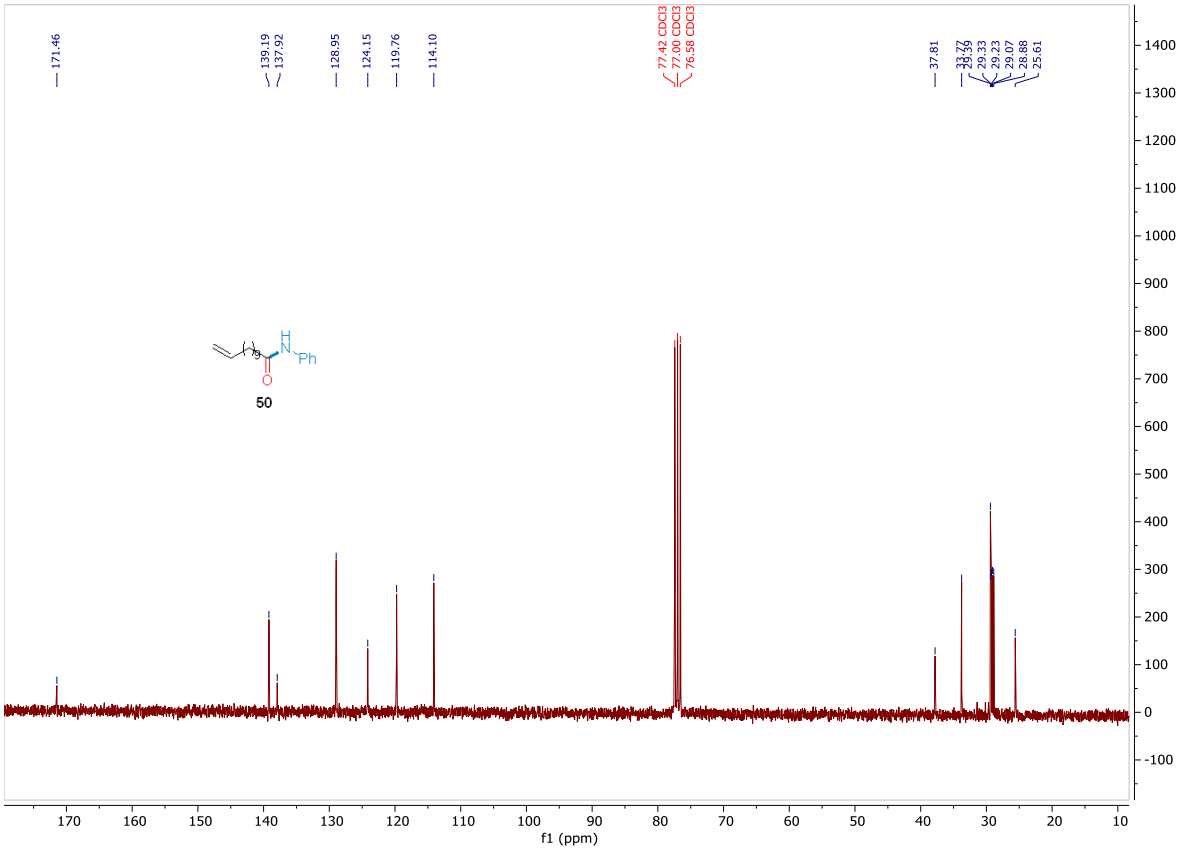
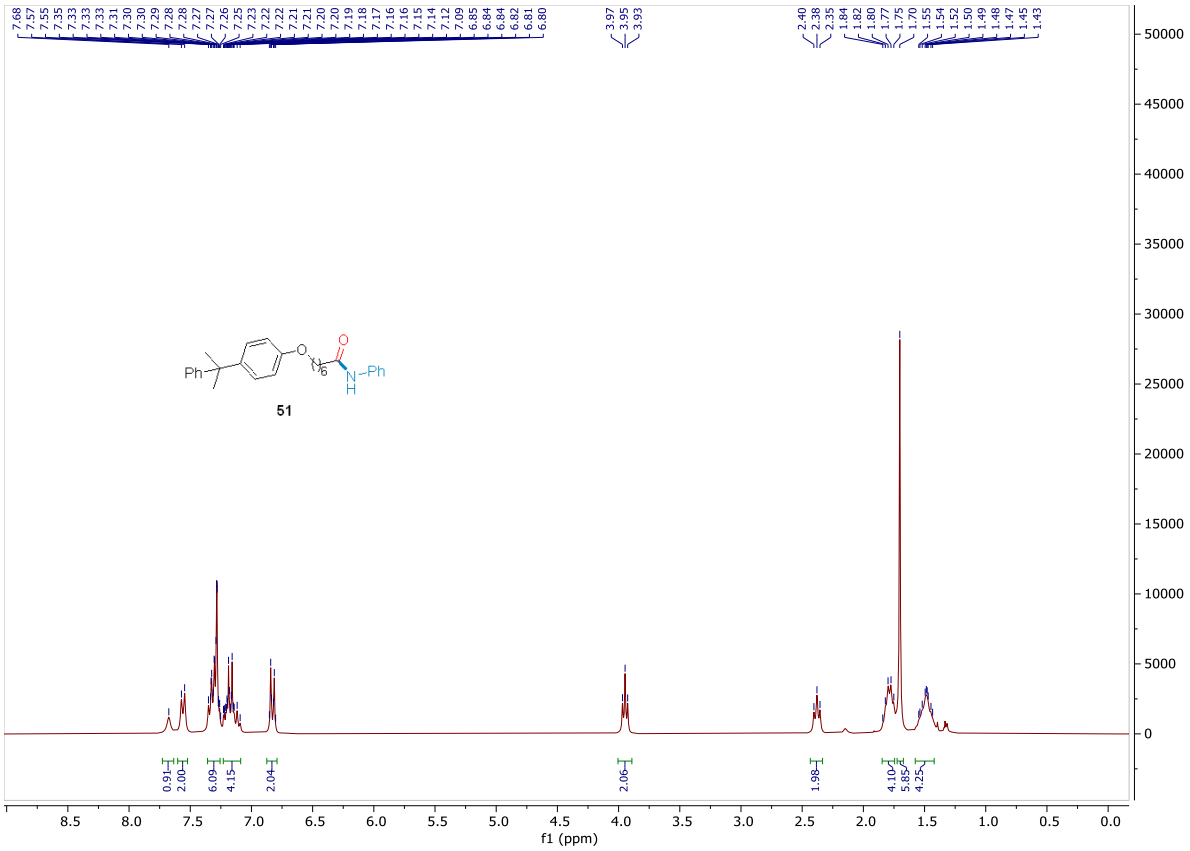
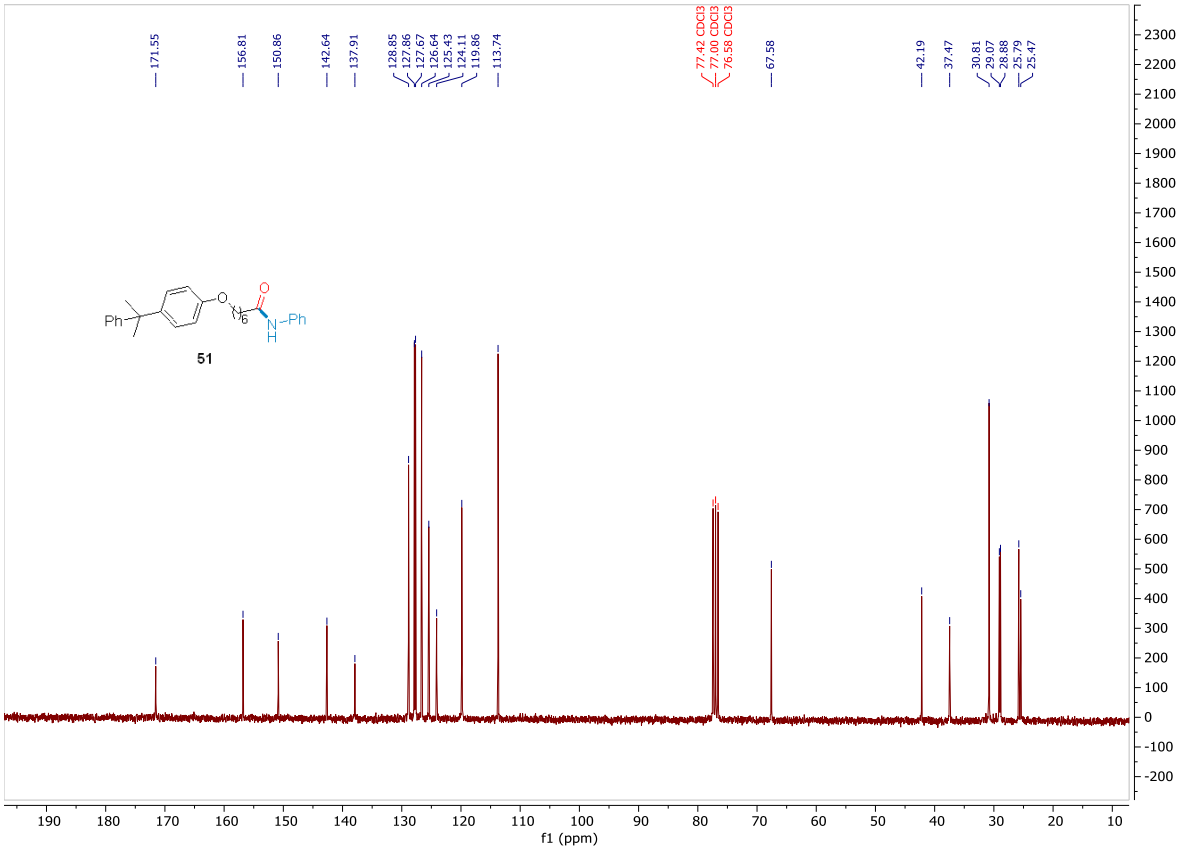
1.98
1.97
1.96
1.95
1.94
1.93
1.92
1.91
1.90
1.89
1.88
1.87
1.86
1.85
1.84
1.83
1.82
1.81
1.80
1.79
1.78
1.78
1.76
1.74
1.73
1.72
1.71
1.69
1.52
1.49
1.47
1.46
1.45
1.45
1.44
1.43
1.41
1.39
O
O
O
6
N
H
Ph
49
2.30
2.01
0.98
1.00
0.95
1.01
2.00
2.05
2.05
4.20
4.27
30000
25000
20000
15000
10000
5000
0
8.0
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
f1 (ppm)


171.42
154.52
148.14
141.41
137.92
128.89
124.14
119.82
107.87
105.61
101.00
97.98
77.42 CDCl3
77.00 CDCl3
76.58 CDCl3
68.67
37.51
29.05
28.89
25.75
25.45
O
O
O
6
N
H
Ph
49
1100
1000
900
800
700
600
500
400
300
200
100
0
-100
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
f1 (ppm)


7.53 7.50 7.33 7.31 7.28 7.12 7.12 7.11 7.09 7.07 7.07
5.88 5.85 5.84 5.83 5.82 5.80 5.79 5.78 5.76 5.74 5.03 5.02 5.02 5.01 5.01 4.97 4.96 4.96 4.96 4.95 4.95 4.94 4.94 4.94 4.92 4.91 4.91 4.91 4.90
2.37 2.35 2.32
2.05 2.05 2.04 2.02 2.02 2.02 2.00 1.72 1.69 1.39 1.38 1.37 1.36 1.34 1.33 1.33 1.32 1.30 1.30 1.29 1.28 1.28
1.97 2.34 1.00 0.94 1.92 2.02 2.02 2.15 12.19
f1 (ppm)
50


171.46
139.19 137.92 128.95 124.15 119.76 114.10
77.42 CDCl3 77.00 CDCl3 76.58 CDCl3
37.81 33.77 29.39 29.33 29.23 29.07 28.88 25.61
f1 (ppm)
50


7.68 7.57 7.55 7.35 7.33 7.33 7.31 7.30 7.30 7.29 7.28 7.28 7.27 7.27 7.26 7.25 7.23 7.22 7.22 7.21 7.21 7.20 7.20 7.19 7.18 7.17 7.16 7.16 7.15 7.14 7.12 7.09 6.85 6.84 6.84 6.82 6.81 6.81 6.80
3.97 3.95 3.93
2.40 2.38 2.35 1.84 1.82 1.80 1.77 1.75 1.70 1.55 1.54 1.52 1.50 1.49 1.48 1.47 1.45 1.43
Ph
O
N
H
Ph
51
0.91 2.00 6.09 4.15 2.04 2.06 1.98 4.10 5.85 4.25
f1 (ppm)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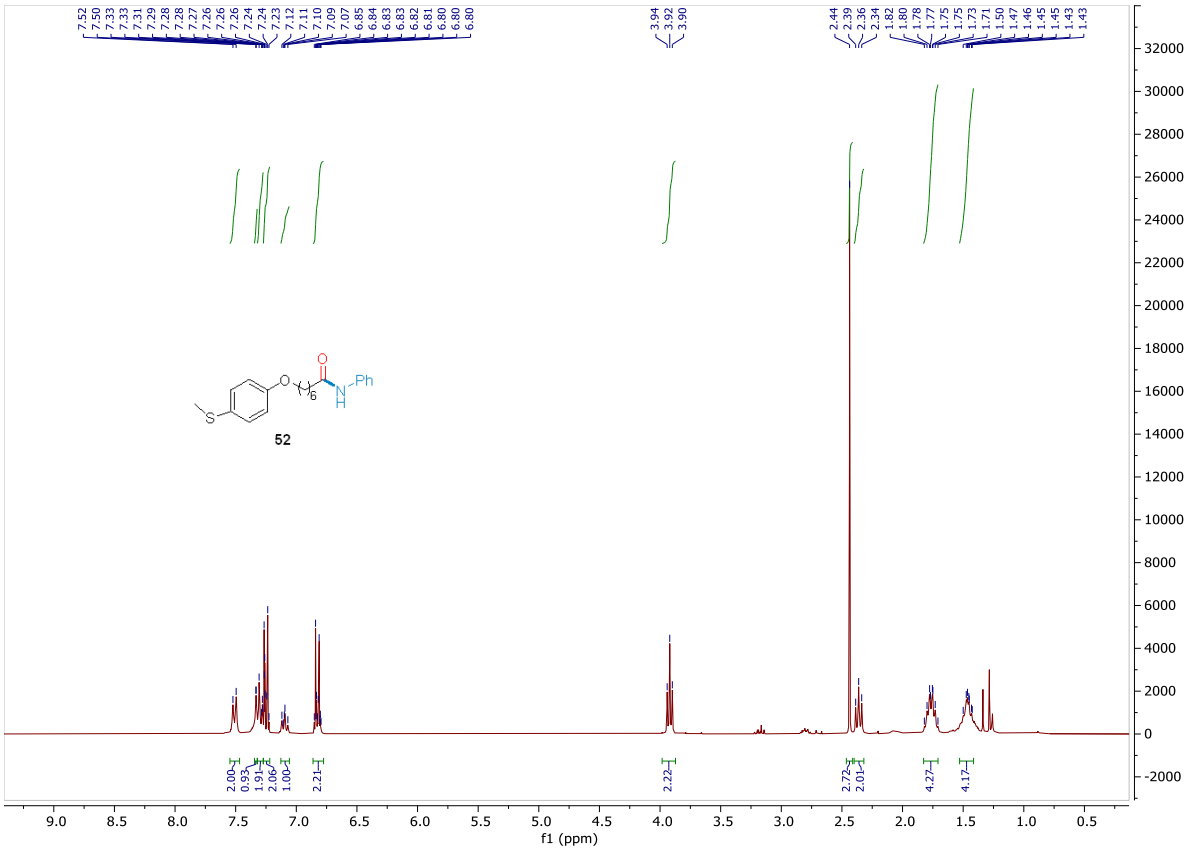
77.42 CDCl3
77.00 CDCl3
76.58 CDCl3
67.58
42.19 37.47 30.81 29.07 28.88 25.79 25.47
Ph
O
N
H
Ph
51
f1 (ppm)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7.26
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7.23
7.12
7.11
7.10
7.09
7.07
6.85
6.84
6.83
6.82
6.81
6.80
3.94
3.92
3.90
2.44
2.39
2.36
2.34
1.82
1.80
1.78
1.77
1.75
1.73
1.71
1.50
1.47
1.46
1.45
1.43
Ph
52
2.00
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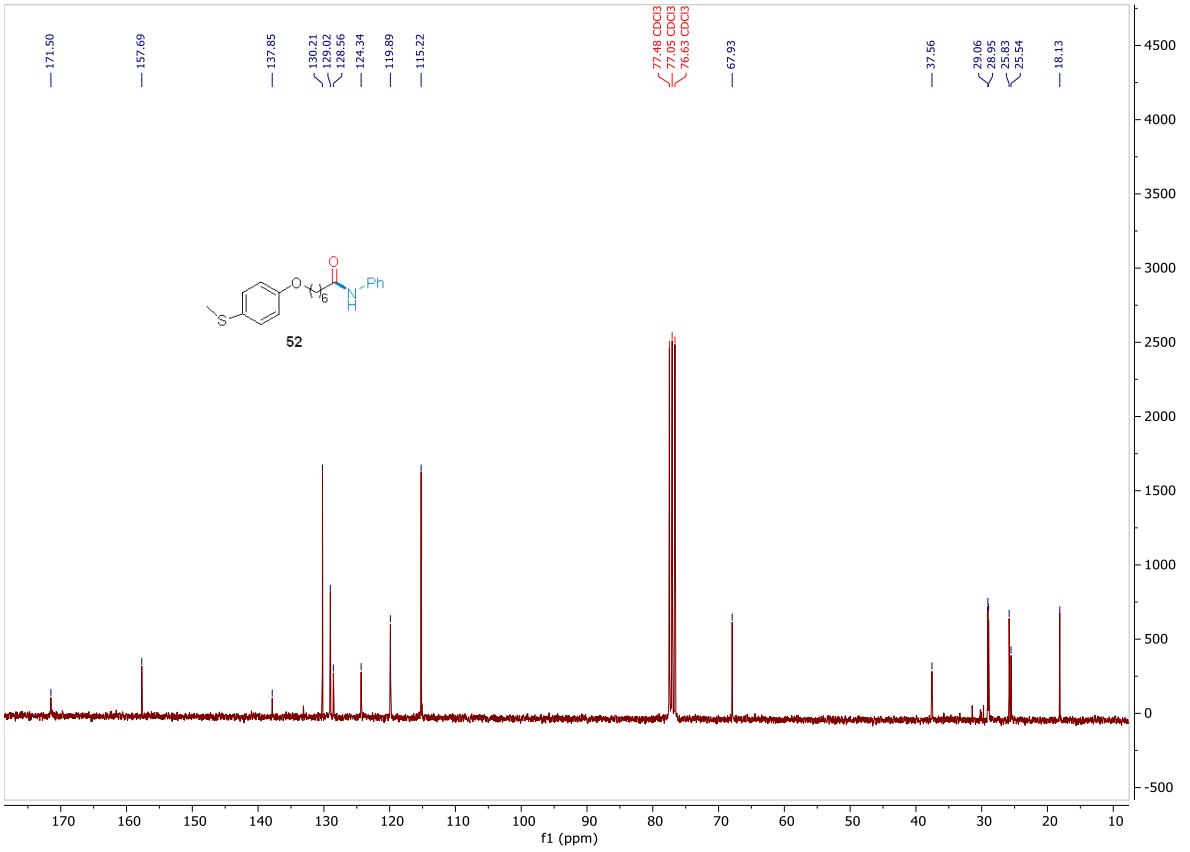
2.06
1.00
2.21
2.22
2.72
2.01
4.27
4.17
f1 (ppm)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128.56
124.34
119.89
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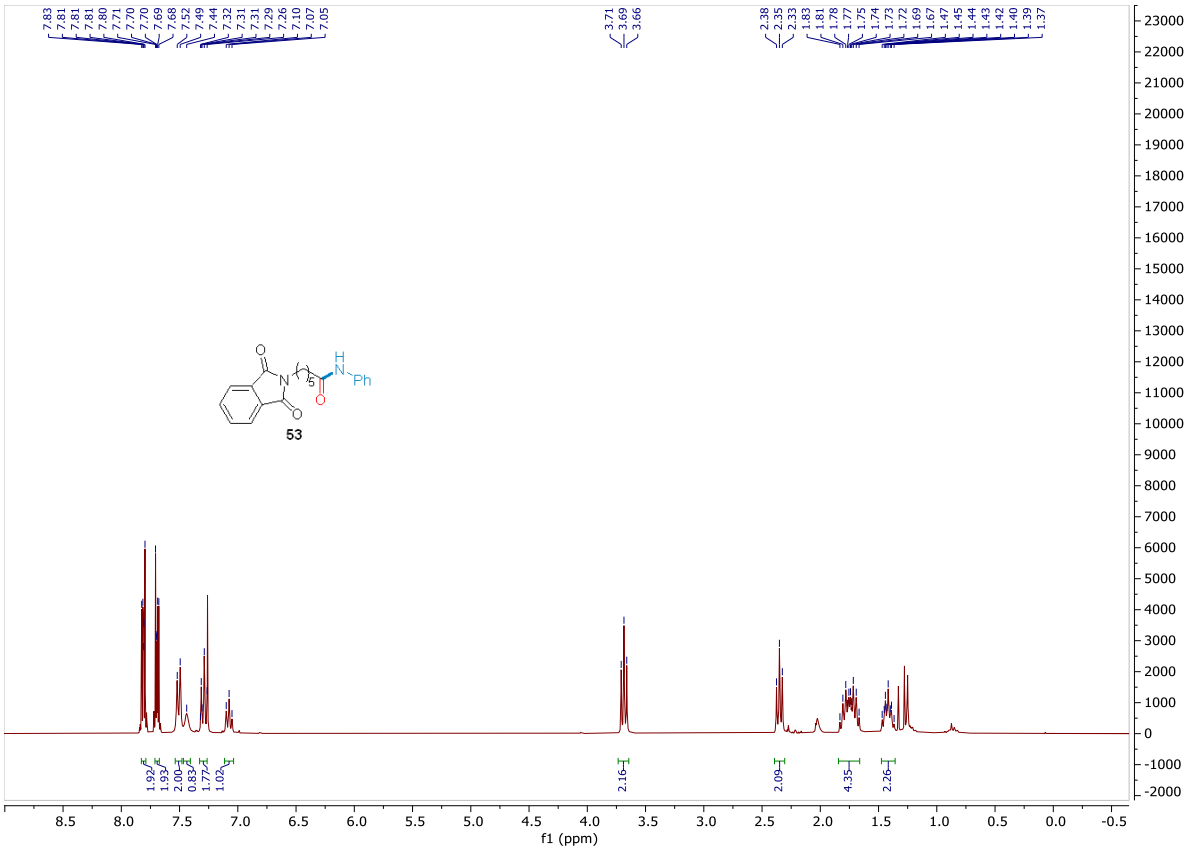
67.93
37.56
29.06
28.95
25.83
25.54
18.13
Ph
52
f1 (ppm)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7.44
7.32
7.31
7.31
7.29
7.26
7.10
7.07
7.05
3.71
3.69
3.66
2.38
2.35
2.33
1.83
1.81
1.78
1.77
1.75
1.74
1.73
1.72
1.69
1.67
1.47
1.45
1.44
1.43
1.42
1.40
1.39
1.37
O
N
5
O
H
N
Ph
O
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1.02
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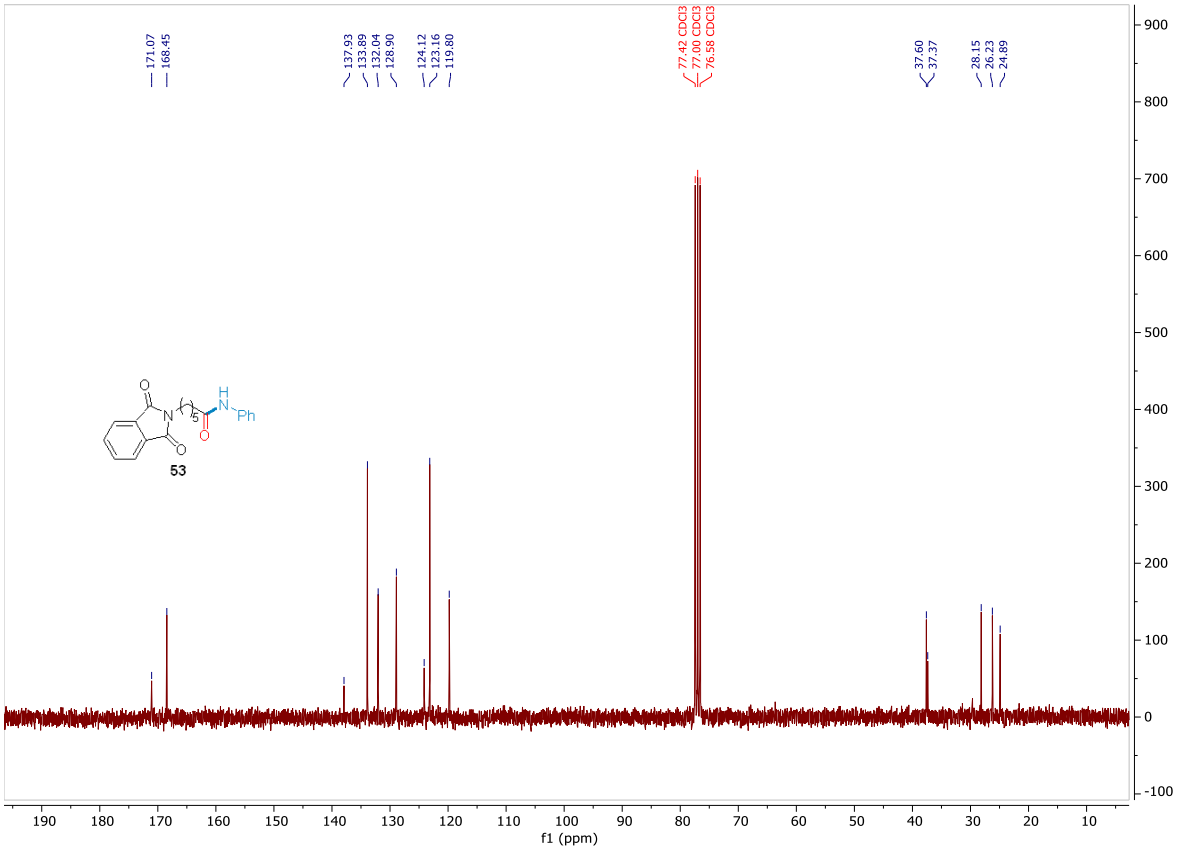
2.09
4.35
2.26
f1 (ppm)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128.90
124.12
123.16
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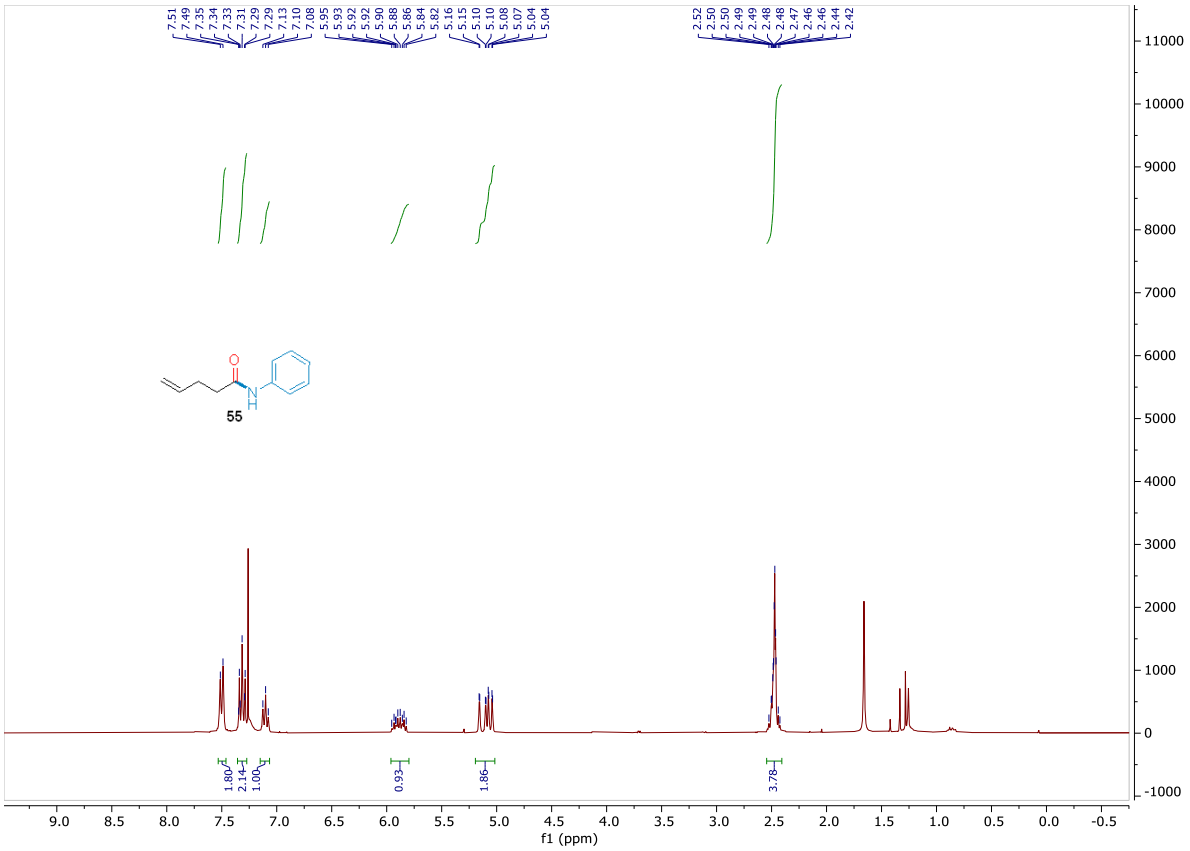
77.00 CDCl3
76.58 CDCl3
37.60
37.37
28.15
26.23
24.89
O
N
5
O
H
N
Ph
O
53
f1 (ppm)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7.08
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5.93
5.92
5.92
5.90
5.88
5.86
5.84
5.82
5.16
5.15
5.10
5.10
5.08
5.07
5.04
5.04
2.52
2.50
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2.49
2.49
2.48
2.48
2.47
2.46
2.46
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H
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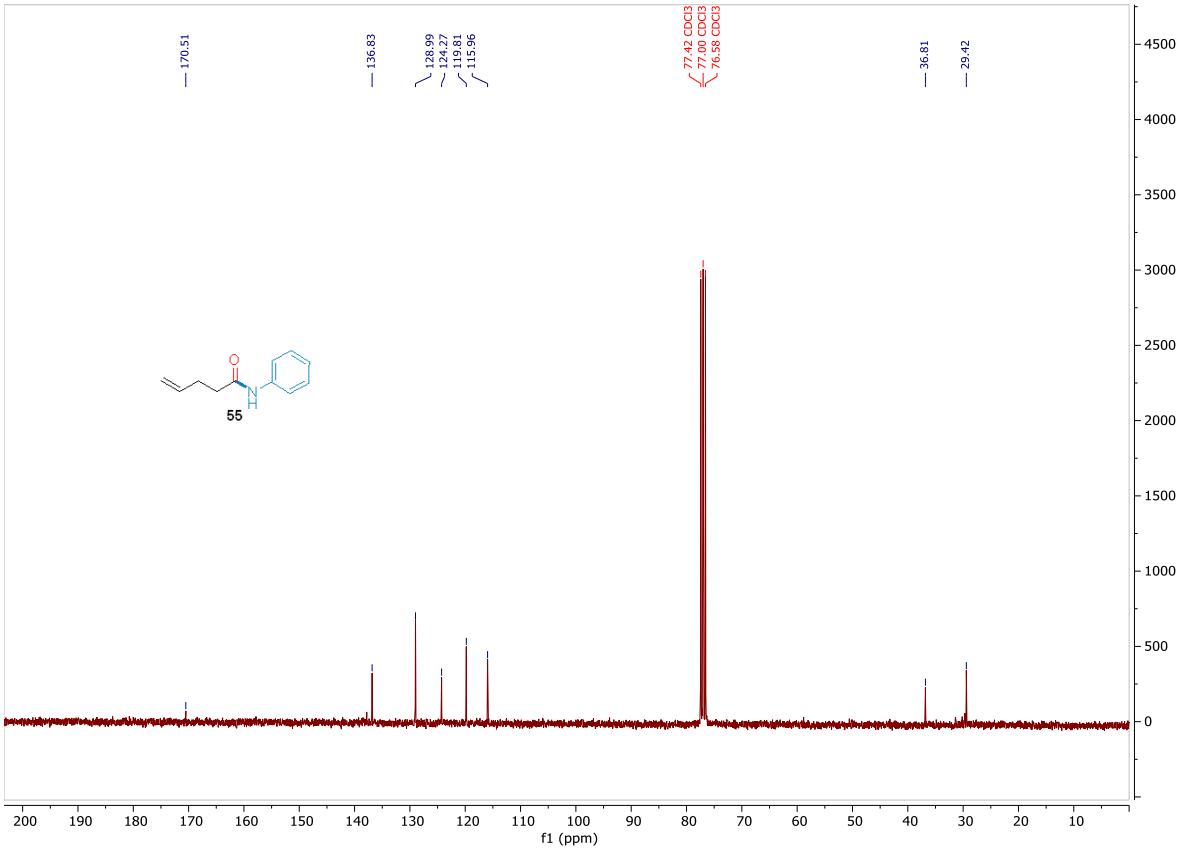
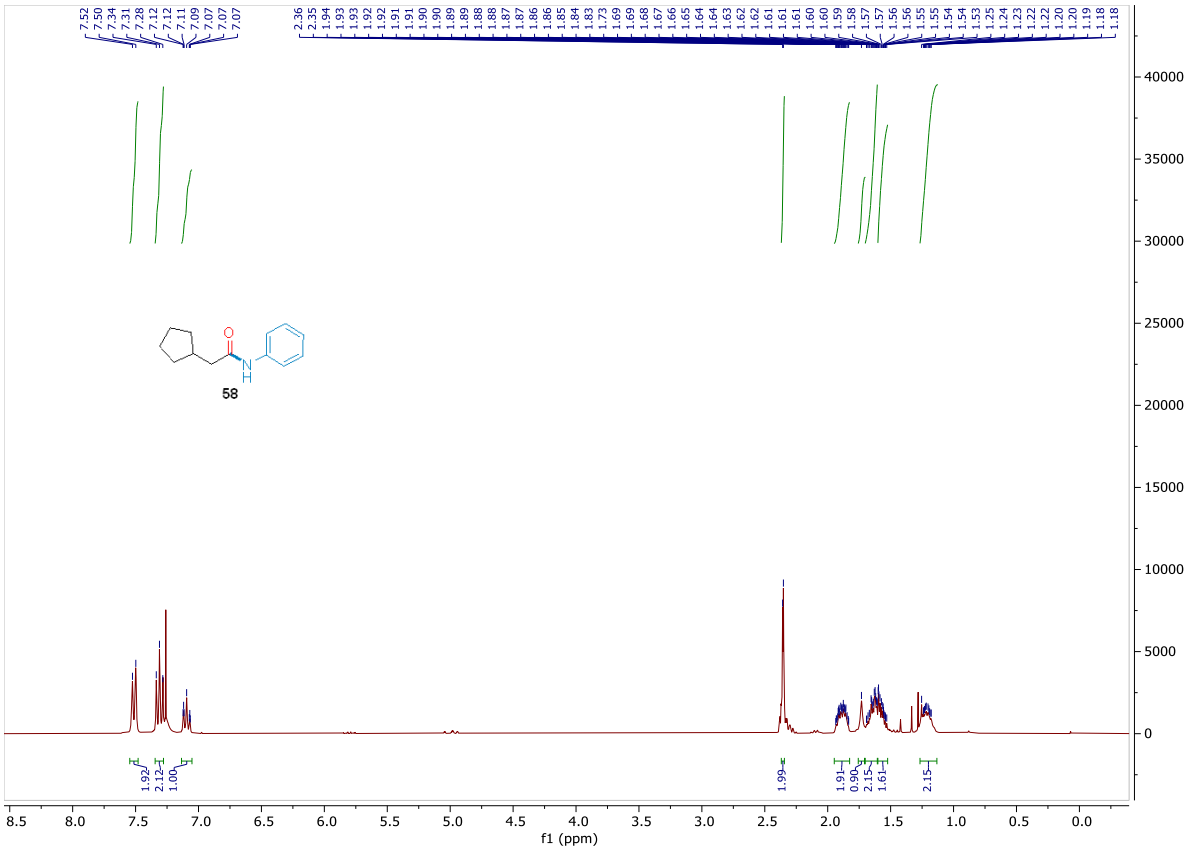
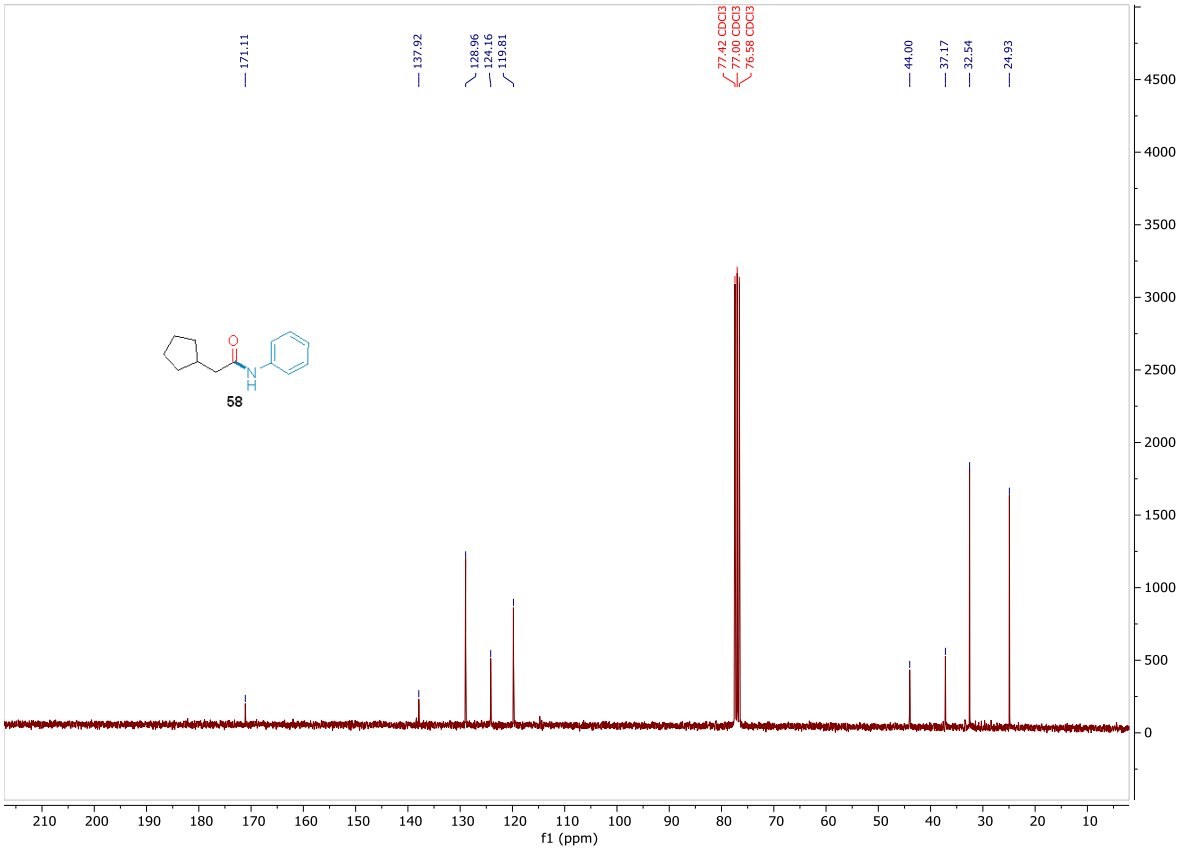
0.93
1.86
3.78
f1 (ppm)


170.51
136.83
128.99
124.27
119.81
115.96
77.42 CDCl3
77.00 CDCl3
76.58 CDCl3
36.81
29.42
O
N
H
55
f1 (ppm)


7.52 7.50 7.34 7.31 7.28 7.12 7.12 7.11 7.09 7.07 7.07 7.07 7.07
2.36 2.35 1.94 1.93 1.93 1.92 1.92 1.91 1.91 1.90 1.90 1.89 1.89 1.88 1.88 1.87 1.87 1.86 1.86 1.85 1.84 1.83 1.73 1.69 1.69 1.68 1.67 1.66 1.65 1.64 1.64 1.63 1.62 1.62 1.61 1.61 1.61 1.60 1.60 1.59 1.58 1.57 1.57 1.56 1.56 1.55 1.55 1.54 1.54 1.53 1.25 1.24 1.23 1.22 1.22 1.20 1.20 1.19 1.18 1.18
58
1.92 2.12 1.00 1.99 1.91 0.90 2.15 1.61 2.15
f1 (ppm)


171.11 137.92 128.96 124.16 119.81 77.42 CDCl3 77.00 CDCl3 76.58 CDCl3 44.00 37.17 32.54 24.93
58
f1 (ppm)