Supporting Information

Rhodium-Catalyzed Carbonylative Coupling of Alkyl Halides with Phenols under Low CO Pressure

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General Remarks

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.

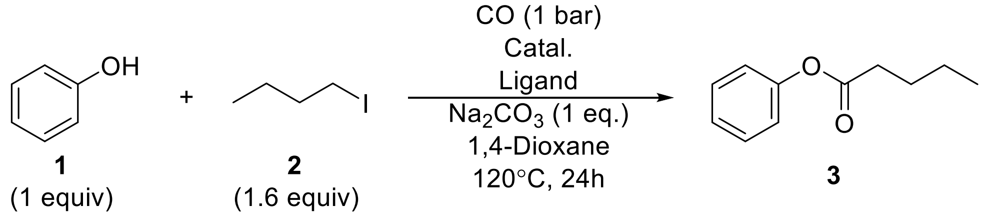
Data collection: GC-yields were calculated using hexadecane as internal standard. GC analysis was performed on an Agilent HP-7890A instrument with FID detector and HP-5 capillary column (polydimethylsiloxane with 5% phenyl groups, 30 m, 0.32 mm i.d., 0.25 μm film thickness) using argon as carrier gas. 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 (^1H NMR) and 77.00 ppm (^{13}C NMR). All measurements were carried out at room temperature unless otherwise stated.

General Procedure for Optimization

Room temperature under air, Na_2CO_3 (0.2 mmol, 1 equiv), catalyst (0.01 mmol, 5 mol%), ligand (0.03 mmol, 15 mol%), phenol (0.2 mmol, 1 equiv) and additive were transferred into an 8-mL vial with a 1.0 cm stirring bar. Then adding dioxane (0.5 mL) with syringe and 1-iodobutane (0.32 mmol, 1.6 equiv) with micro syringe. The vial was then capped (with a needle) and placed in an aluminum rack within a Parr pressure reactor. Next, the reactor was closed and a CO gas cylinder with a pressure regulator was connected. The reactor was flushed with CO gas (5 bar; fill and released) three times and 1 bar of carbon monoxide (measured by pressure meter) was charged. Then

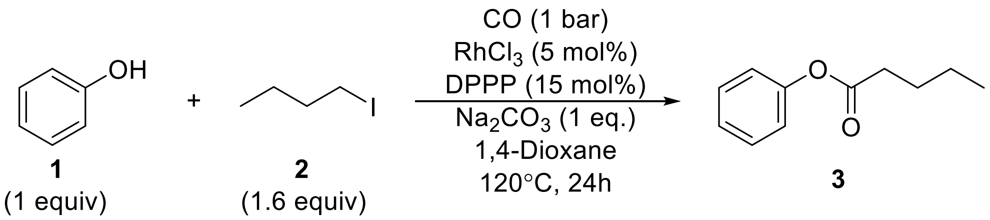
5 bar N_2 was pressurized to prevent solvent evaporation. The reaction was stirred at 120 ℃ with a stir rate at 550 rpm for 24 h. After cooling to room temperature, the CO gas was released and 10 μL of hexadecane as internal standard was added to the vial. The reaction mixture was then stirred for 5 min and a proper amount of solution was taken for GC analysis.

Table S1. Catalyst and ligand investigation



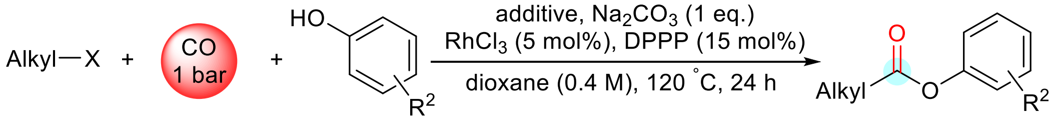
Entry[a]	Catal.(mol%)	Ligand (mol%)	Yield (%)[b]
1	$Rh(CO)_2acac$ (5)	DPPP (15)	72
2	$[Rh(cod)OH]_2$ (2.5)	DPPP (15)	45
3	$[Rh(OAc)_2]_2$ (2.5)	DPPP (15)	49
4	$RhCl(PPh_3)_3$ (5)	DPPP (15)	57
5	$NiBr_2$•dme (5)	DPPP (15)	0
6	$PdCl_2$ (5)	DPPP (15)	Trace
7	$RhCl_3$ (5)	DPPE (15)	41
8	$RhCl_3$ (5)	DPPB (15)	41
9	$RhCl_3$ (5)	DPPF (15)	39
10	$RhCl_3$ (5)	Xantphos (15)	4
11	$RhCl_3$ (5)	PPh_3 (30)	29
12	$RhCl_3$ (5)	PCy_3 (30)	17

Table S2. Additive investigation



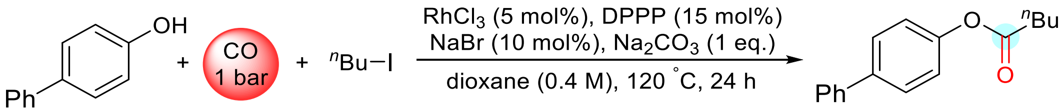
Entry	Additive (10 mol%)	Yield (%)
1	NaCl	95
2	NaI	98
3	NaOAc	61
4	TBAB	89
5	KBr	90

General Procedure for Carbonylative Coupling



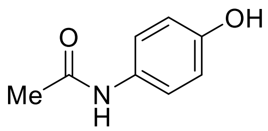
Room temperature under air, Na_2CO_3 (53 mg, 0.5 mmol, 1 equiv), $RhCl_3$ (5.2 mg, 0.025 mmol, 5 mol%), DPPP (30.9 mg, 0.075 mmol, 15 mol%), phenols (if solid) (0.5 mmol, 1 equiv), alkyl halide (if solid) (0.8 mmol, 1.6 equiv) and NaBr (if X = I) (5.1 mg, 0.05 mmol, 10 mol%) or NaI (if X = Br) (60 mg, 0.4 mmol, 80 mol%) were transferred into an 8-mL vial with a 1.0 cm stirring bar. Then adding dioxane (1.25 mL) with syringe and alkyl halide (if liquid) (0.8 mmol, 1.6 equiv) and phenol (if liquid) (0.5 mmol, 1 equiv) with micro syringe. The vial was then capped (with a needle) and placed in an aluminum rack within a Parr pressure reactor. Next, the reactor was closed and a CO gas cylinder with a pressure regulator was connected to the reactor. The reactor was flushed with CO gas three times (5 bar; fill and released) and 1 bar of carbon monoxide (measured by pressure meter) was charged. Then 5 bar of N_2 was pressurized to prevent solvent evaporation. The reaction was stirred at 120 °C with a stir rate at 550 rpm for 24 h. After cooling to room temperature, the CO gas was released carefully, and the crude product was purified by silica gel chromatography (PE/EA) to afford the corresponding ester products.

Procedure for Gram Synthesis



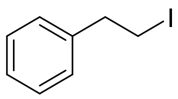
Room temperature under air, Na_2CO_3 (530 mg, 5 mmol, 1 equiv), $RhCl_3$ (52.3 mg, 0.25 mmol, 5 mol%), DPPP (309.3 mg, 0.75 mmol, 15 mol%), 4-phenylphenol (0.85 g, 5 mmol, 1 equiv) and NaBr (51.5 mg, 0.5 mmol, 10 mol%) were transferred into an 25-mL round-bottomed flask with a 3.0 cm stirring bar. Then adding dioxane (12.5 mL) with syringe and 1-iodobutane (8 mmol, 910 µL, 1.6 equiv) with micro syringe. The flask placed in a Parr pressure reactor. Next, the reactor was closed and a CO gas cylinder with a pressure regulator was connected to the reactor. The reactor was flushed with CO gas three times (5 bar; fill and released) and 1 bar of carbon monoxide (measured by pressure meter) was charged. Then 5 bar of N_2 was pressurized to prevent solvent evaporation. The reaction was stirred at 120 °C with a stir rate at 550 rpm for 24 h. After cooling to room temperature, the CO gas was released carefully, and the crude product was purified by silica gel chromatography (PE/EA) to afford the corresponding ester as a white solid (1.27 g, 99% yield).

Preparation of Substrates



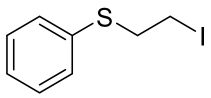
***N*-(4-Hydroxyphenyl)acetamide**[1]

To a solution of 4-aminophenol (218.3 mg, 2 mmol) in 3 mL of absolute ethanol was added acetic anhydride (190 µL, 2 mmol). The solution was stirred for 15 min at room temperature, then evaporated to dryness. The resulting solid was purified by silica gel column chromatography (DCM/MeOH = 95:5) and afforded the desired product as a white powder (299.1 mg, 99% yield). **^{1}H NMR** (400 MHz, DMSO-d$_6$) δ 9.67 (s, 1H), 9.18 (s, 1H), 7.35 (d, J = 8.9 Hz, 2H), 6.69 (d, J = 8.9 Hz, 2H). **^{13}C NMR** (101 MHz, DMSO-d$_6$) δ 167.8, 153.3, 131.2, 121.1, 115.2, 23.9.



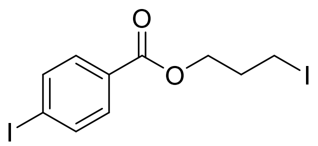
(2-Iodoethyl)benzene[2]

To a 1 M solution of the corresponding (2-bromoethyl)benzene (238 µL, 2 mmol) in acetone was added NaI (899.3 mg, 6 mmol). The mixture was stirred at 60 °C for 12 h. After cooling to RT, CH_2Cl_2 was added until the complete precipitation of salts. Then, the mixture was washed with a 0.1 M aqueous $Na_2S_2O_3$ solution. The organic phase was collected, dried over $MgSO_4$ and filtered. The mixture was concentrated, then evaporated to dryness. The resulting solid was purified by silica gel column chromatography (PE) afforded the desired product as a light yellow oil (417.6 mg, 90% yield). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.39 - 7.19 (m, 5H), 3.44 - 3.33 (m, 2H), 3.27 - 3.15 (m, 2H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 140.6, 128.6, 128.3, 126.8, 40.3, 5.5.



(2-Iodoethyl)(phenyl)sulfane[3]

To a solution of 2-(phenylthio)ethan-1-ol (270 µL, 2 mmol) in THF (3.5 ml) was added imidazole (193.4 mg, 2.4 mmol) and triphenylphosphine (629.5 mg, 2.4 mmol) at 0 °C. Then, iodine (609.1, 2.4 mmol) was slowly added to the reaction and the mixture was stirred for 16 h at 25 °C. Next, the mixture was washed with 0.1 M aqueous $Na_2S_2O_3$ solution. The aqueous layer was extracted with EA and the combined organic extracts were dried over $MgSO_4$ and concentrated under vacuum. The compound was purified by column chromatography (PE/EA = 9:1) and afforded the desired product as a light yellow oil (295.8 mg, 96% yield). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.37 - 7.14 (m, 5H), 3.42 - 3.08 (m, 4H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 134.0, 130.6, 129.2, 127.1, 37.0, 2.5.

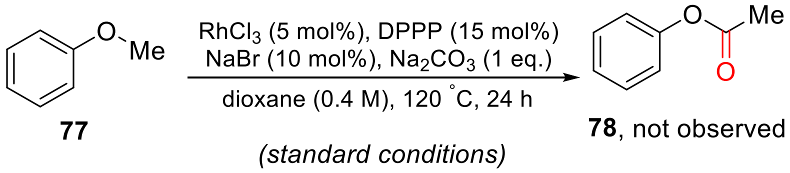


3-Iodopropyl 4-iodobenzoate[4]

To an oven-dried round bottom flask charged with a mixture of 3-iodopropan-1-ol (0.93 g, 5.0 mmol), Et_3N (0.84 mL, 6.0 mmol) in DCM (10 mL) was added 4-Methoxybenzoyl chloride (0.85 g, 5.0 mmol) at 0°C. The reaction mixture was warmed to room temperature and overnight. The reaction mixture was then partitioned with H_2O (2×10 mL) and brine (15 mL). The organic phase was collected, dried over $MgSO_4$ and filtered. The mixture was concentrated, then evaporated to dryness. The resulting solid was purified by silica gel column chromatography (PE/EA = 10/1) afforded the desired product as a light yellow oil (1.87 g, 90% yield). **^{1}H NMR** (300 MHz, $CDCl_3$) δ 7.74 (d, J = 8.8 Hz, 2H), 7.66 (d, J = 8.7 Hz, 2H), 4.33 (t, J = 6.1 Hz, 2H), 3.22 (t, J = 6.8 Hz, 2H), 2.28 - 2.14 (m, 2H). **^{13}C NMR** (75 MHz, $CDCl_3$) δ 165.9, 137.8, 131.0, 129.4, 100.9, 64.8, 32.4, 1.2.

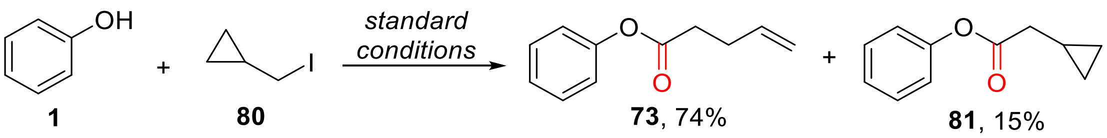
Mechanistic Studies

Carbonylation of anisole



Room temperature under air, Na_2CO_3 (53 mg, 0.5 mmol, 1 equiv), $RhCl_3$ (5.2 mg, 0.025 mmol, 5 mol%), DPPP (30.9 mg, 0.075 mmol, 15 mol%) and NaBr (5.1 mg, 0.05 mmol, 10 mol%) were transferred into an 8-mL vial with a 1.0 cm stirring bar. Then adding dioxane (1.25 mL) with syringe and anisole **77** (54 µL, 0.5 mmol, 1 equiv) with micro syringe. The vial was then capped (with a needle) and placed in an aluminum rack within a Parr pressure reactor. Next, the reactor was closed and a CO gas cylinder with a pressure regulator was connected to the reactor. The reactor was flushed with CO gas three times and 1 bar of carbon monoxide was charged. Then 5 bar of N_2 was pressurized to prevent solvent evaporation. The reaction was stirred at 120 °C with a stirring rate at 550 rpm for 24 h. After cooling to room temperature, the CO gas was released carefully. Then a proper amount of solution was taken for GC-MS analysis, no reaction was observed.

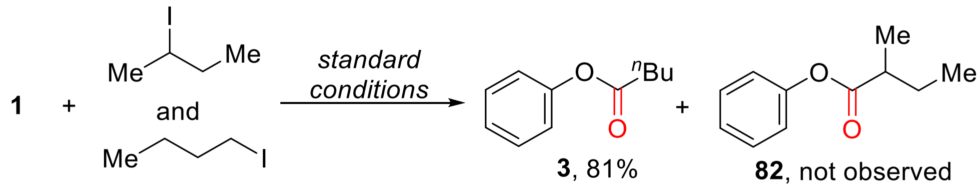
Radical clock experiment



Room temperature under air, Na_2CO_3 (53 mg, 0.5 mmol, 1 equiv), $RhCl_3$ (5.2 mg, 0.025 mmol, 5 mol%), DPPP (30.9 mg, 0.075 mmol, 15 mol%), phenol **1** (47.1 mg, 0.5

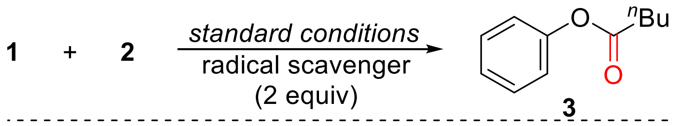
mmol, 1 equiv) and NaBr (5.1 mg, 0.05 mmol, 10 mol%) were transferred into an 8-mL vial with a 1.0 cm stir bar. Then adding dioxane (1.25 mL) with syringe and (iodomethyl)cyclopropane **80** (75 µL, 0.8 mmol, 1.6 equiv) with micro syringe. The vial was then capped (with a needle) and placed in an aluminum rack within a Parr pressure reactor. Next, the reactor was closed and a CO gas cylinder with a pressure regulator was connected to the reactor. The reactor was flushed with CO gas three times and 1 bar of carbon monoxide was charged. Then 5 bar of N_2 was pressurized to prevent solvent evaporation. The reaction was stirred at 120 °C with a stir rate at 550 rpm for 24 h. After cooling to room temperature, the CO gas was released. The crude product was purified by silica gel chromatography (PE/EA) to afford the products. Then the products analyzed by NMR, the result is shown above.

Competition reaction



Room temperature under air, Na_2CO_3 (53 mg, 0.5 mmol, 1 equiv), $RhCl_3$ (5.2 mg, 0.025 mmol, 5 mol%), DPPP (30.9 mg, 0.075 mmol, 15 mol%), phenol **1** (47.1 mg, 0.5 mmol, 1 equiv) and NaBr (5.1 mg, 0.05 mmol, 10 mol%) were transferred into an 8-mL vial with a 1.0 cm stir bar. Then adding dioxane (1.25 mL) with syringe, 1-iodobutane (91 µL, 0.8 mmol, 1.6 equiv) and 2-iodobutane (92 µL, 0.8 mmol, 1.6 equiv) with micro syringe. The vial was then capped (with a needle) and placed in an aluminum rack within a Parr pressure reactor. Next, the reactor was closed and a CO gas cylinder with a pressure regulator was connected to the reactor. The reactor was flushed with CO gas three times and 1 bar of carbon monoxide was charged. Then 5 bar of N_2 was pressurized to prevent solvent evaporation. The reaction was stirred at 120 °C with a stir rate at 550 rpm for 24 h. After cooling to room temperature, the CO gas was released. The crude product was purified by silica gel chromatography (PE/EA) to afford the products. Then the products analyzed by NMR, the result is shown above.

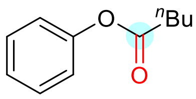
Radical inhibition experiment



TEMPO	trace
2,6-di-*tert*-butylphenol	99%
α-phenyl styrene	99%

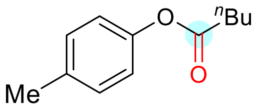
Room temperature under air, Na_2CO_3 (53 mg, 0.5 mmol, 1 equiv), $RhCl_3$ (5.2 mg, 0.025 mmol, 5 mol%), DPPP (30.9 mg, 0.075 mmol, 15 mol%), phenol **1** (47.1 mg, 0.5 mmol, 1 equiv), NaBr (5.1 mg, 0.05 mmol, 10 mol%) and the radical scavenger (1 mmol, 2 equiv) were transferred into an 8-mL vial with a 1.0 cm stirring bar. Then adding dioxane (1.25 mL) with syringe and 1-iodobutane **2** (91 μL, 0.8 mmol, 1.6 equiv) with micro syringe. The vial was then capped (with a needle) and placed in an aluminum rack within a Parr pressure reactor. Next, the reactor was closed and a CO gas cylinder with a pressure regulator was connected to the reactor. The reactor was flushed with CO gas three times and 1 bar of carbon monoxide was charged. Then 5 bar of N_2 was pressurized to prevent solvent evaporation. The reaction was stirred at 120 °C with a stir rate at 550 rpm for 24 h. After cooling to room temperature, the CO gas was released. Then a proper amount of solution was taken for GC and GC-MS analysis. The result is shown above.

Characterization of The Products

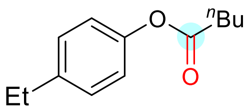


Phenyl pentanoate (3). Prepared according to general procedure using phenol (47.1 mg, 0.5 mmol) and 1-iodobutane (91 μL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title c ompound as a light yellow oil (86.4 mg, 96% yield).

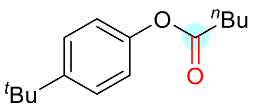
^{1}**H NMR** (400 MHz, $CDCl_3$) δ 7.47 - 7.37 (m, 2H), 7.26 (t, *J* = 7.4 Hz, 1H), 7.13 (d, *J* = 7.4 Hz, 2H), 2.66 - 2.56 (t, 2H), 1.86 - 1.73 (m, 2H), 1.50 (m, *J* = 14.7, 7.4 Hz, 2H), 1.03 (t, *J* = 7.4 Hz, 3H).
^{13}**C NMR** (101 MHz, $CDCl_3$) δ 172.1, 150.7, 129.3, 125.6, 121.5, 34.0, 26.9, 22.1, 13.6.
HRMS (ESI-TOF): calcd. for $[C_{11}H_{14}O_2+Na]^+$ 201.0891, found 201.0893.



***p*-Tolyl pentanoate (4)**. Prepared according to general procedure using *p*-cresol (54.1 mg, 0.5 mmol) and 1-iodobutane (91 μL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title c ompound as a light yellow oil (85.2 mg, 97% yield).
^{1}**H NMR** (300 MHz, $CDCl_3$) δ 7.06 (d, *J* = 8.0 Hz, 2H), 6.86 (d, *J* = 8.3 H z, 2H), 2.50 - 2.33 (t, 2H), 2.24 (s, 3H), 1.73 - 1.56 (m, 2H), 1.35 (m, *J* = 14.5, 7.3 Hz, 2H), 0.88 (t, *J* = 7.3 Hz, 3H).
^{13}**C NMR** (75 MHz, $CDCl_3$) δ 172.4, 148.5, 135.2, 129.8, 121.1, 34.0, 27.0, 2 2.2, 20.7, 13.6.
HRMS (EI): calcd. for $[C_{12}H_{16}O_2]^+$ 192.1145, found 195.1150.

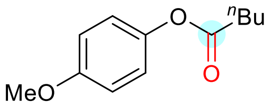


4-Ethylphenyl pentanoate (5). Prepared according to general procedure using 4 -ethylphenol (61.1 mg, 0.5 mmol) and 1-iodobutane (91 μL, 0.8 mmol). The cr ude product was purified by silica gel chromatography (PE/EA = 20:1) to affo rd the title compound as a light yellow oil (102 mg, 99% yield).
^{1}**H NMR** (400 MHz, $CDCl_3$) δ 7.25 (d, *J* = 8.8 Hz, 2H), 7.05 (d, *J* = 8.5 H z, 2H), 2.70 (q, *J* = 7.6 Hz, 2H), 2.64 - 2.56 (m, 2H), 1.86 - 1.73 (m, 2H), 1.51 (m, *J* = 14.7, 7.4 Hz, 2H), 1.29 (t, *J* = 7.6 Hz, 3H), 1.03 (t, *J* = 7.4 H z, 3H).
^{13}**C NMR** (101 MHz, $CDCl_3$) δ 172.3, 148.6, 141.4, 128.6, 121.2, 121.2, 34.0, 28.2, 26.9, 22.1, 15.4, 13.6.
HRMS (EI): calcd. for $[C_{13}H_{18}O_2]^+$ 206.1301, found 206.1304.

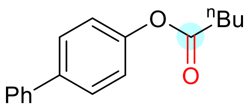


4-(*tert*-Butyl)phenyl pentanoate (6). Prepared according to general procedure u sing 4-*tert*-butylphenol (75.1 mg, 0.5 mmol) and 1-iodobutane (91 μL, 0.8 mm ol). The crude product was purified by silica gel chromatography (PE/EA = 20: 1) to afford the title compound as a colorless oil (108.9 mg, 93% yield).

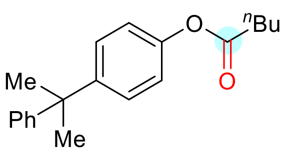
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.44 (d, *J* = 9.0 Hz, 2H), 7.06 (d, *J* = 8.9 Hz, 2H), 2.60 (t, 2H), 1.96 - 1.69 (m, 2H), 1.60 - 1.43 (m, 2H), 1.38 (s, 9H), 1.03 (t, *J* = 7.3 Hz, 3H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 172.3, 148.3, 148.3, 126.1, 120.8, 34.3, 34.0, 31.3, 27.0, 22.2, 13.6.
HRMS (EI): calcd. for $[C_{15}H_{22}O_2]^+$ 234.1614, found 234.1612.



4-Methoxyphenyl pentanoate (7). Prepared according to general procedure using 4-methoxyphenol (62.1 mg, 0.5 mmol) and 1-iodobutane (91 μL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title compound as a colorless oil (103 mg, 99% yield).
^{1}H NMR (400 MHz, $CDCl_3$) δ 6.89 (d, *J* = 9.1 Hz, 2H), 6.77 (d, *J* = 9.1 Hz, 2H), 3.67 (s, 3H), 2.47 - 2.38 (t, 2H), 1.69 - 1.57 (m, 2H), 1.34 (m, *J* = 14.7, 7.4 Hz, 2H), 0.87 (t, *J* = 7.4 Hz, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 172.5, 157.0, 144.1, 122.2, 114.2, 55.3, 33.9, 26.9, 22.1, 13.6.
HRMS (EI): calcd. for $[C_{12}H_{16}O_3]^+$ 208.1094, found 208.1096.

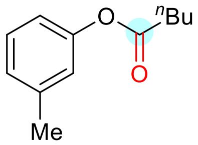


[1,1'-Biphenyl]-4-yl pentanoate (8). Prepared according to general procedure using 4-phenylphenol (85.1 mg, 0.5 mmol) and 1-iodobutane (91 μL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title compound as a light yellow oil (125.7 mg, 99% yield).
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.49 - 7.40 (m, 4H), 7.30 (t, *J* = 7.3 Hz, 2H), 7.21 (t, 1H), 7.03 (d, 2H), 2.45 (t, 2H), 1.70 - 1.58 (m, 2H), 1.42 - 1.27 (m, 2H), 0.87 (t, *J* = 7.3 Hz, 3H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 172.2, 150.1, 140.3, 138.7, 128.7, 128.0, 127.2, 127.0, 121.7, 34.0, 26.9, 22.2, 13.7.
HRMS (EI): calcd. for $[C_{12}H_{16}O_2]^+$ 254.1301, found 254.1313.

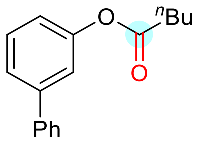


4-(2-Phenylpropan-2-yl)phenyl pentanoate (9). Prepared according to general procedure using 4-(2-phenylpropan-2-yl)phenol (106.2 mg, 0.5 mmol) and 1-iodobutane (91 μL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title compound as a light yellow oil (146.6 mg, 99% yield).

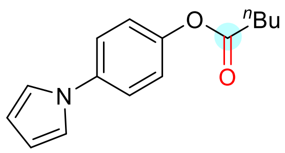
^{1}**H NMR** (400 MHz, $CDCl_3$) δ 7.17 - 7.07 (m, 6H), 7.04 (t, J = 6.6 Hz, 1H), 6.86 (d, J = 8.7 Hz, 2H), 2.42 (t, J = 7.5 Hz, 2H), 1.61 (dd, J = 15.0, 7.7 Hz, 2H), 1.56 (s, 6H), 1.38 - 1.26 (m, 2H), 0.85 (t, J = 7.4 Hz, 3H).
^{13}**C NMR** (101 MHz, $CDCl_3$) δ 172.2, 150.2, 148.5, 147.9, 127.9, 127.9, 127.7, 126.6, 126.6, 126.6, 125.6, 120.7, 42.6, 34.0, 30.7, 26.9, 22.1, 13.6.
HRMS (EI): calcd. for $[C_{20}H_{24}O_2]^+$ 296.1771, found 296.1782.



***m*-Tolyl pentanoate (10)**. Prepared according to general procedure using *m*-cresol (52.3 mg, 0.5 mmol) and 1-iodobutane (91 μL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title compound as a light yellow oil (95.1 mg, 99% yield).
^{1}**H NMR** (300 MHz, $CDCl_3$) δ 7.15 (t, J = 7.6 Hz, 1H), 6.93 (d, J = 7.7 Hz, 1H), 6.80 (s, 2H), 2.54 - 2.36 (m, 2H), 2.26 (s, 3H), 1.77 - 1.57 (m, 2H), 1.36 (m, J = 14.5, 7.3 Hz, 2H), 0.88 (t, J = 7.3 Hz, 3H).
^{13}**C NMR** (101 MHz, $CDCl_3$) δ 172.2, 150.2, 148.5, 147.9, 127.9, 127.9, 127.7, 126.6, 126.6, 126.6, 125.6, 120.7, 42.6, 34.0, 30.7, 26.9, 22.1, 13.6.
HRMS (EI): calcd. for $[C_{12}H_{16}O_2]^+$ 192.1145, found 192.1140.



[1,1'-Biphenyl]-3-yl pentanoate (11). Prepared according to general procedure using 3-(pentyloxy)-1,1'-biphenyl hydrate (85.1 mg, 0.5 mmol) and 1-iodobutane (91 μL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title compound as a light yellow oil (125.8 mg, 99% yield).
^{1}**H NMR** (300 MHz, $CDCl_3$) δ 7.69 - 7.62 (m, 2H), 7.57 - 7.49 (m, 3H), 7.49 - 7.21 (m, 3H), 7.20 - 7.11 (m, 1H), 2.73 - 2.60 (m, 2H), 1.85 (dt, J = 15.2, 7.5 Hz, 2H), 1.55 (m, J = 14.4, 7.3 Hz, 2H), 1.13 - 1.01 (m, 3H).
^{13}**C NMR** (75 MHz, $CDCl_3$) δ 172.1, 151.1, 142.7, 140.1, 129.5, 128.7, 127.5, 127.1, 124.4, 121.8, 120.2, 34.0, 26.9, 22.2, 13.7.
HRMS (EI): calcd. for $[C_{17}H_{18}O_2]^+$ 254.1301, found 254.1300.

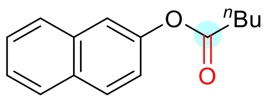


4-(1*H*-Pyrrol-1-yl)phenyl pentanoate (12). Prepared according to general procedure using 4-(1*H*-pyrrol-1-yl)phenol (79.6 mg, 0.5 mmol) and 1-iodobutane (91 μL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title compound as a light brown oil (120.3 mg, 99% yield).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.44 (d, *J* = 8.7 Hz, 2H), 7.20 (d, *J* = 8.7 Hz, 2H), 7.11 (t, 2H), 6.42 (t, 2H), 2.64 (t, 2H), 1.94 - 1.74 (m, 2H), 1.69 - 1.41 (m, 2H), 1.07 (t, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 172.1, 148.2, 138.3, 122.5, 121.3, 119.3, 110.4, 33.9, 26.8, 22.1, 13.6.

HRMS (ESI-TOF): calcd. for $[C_{15}H_{17}NO_2+H]^+$ 244.1337, found 244.1338.

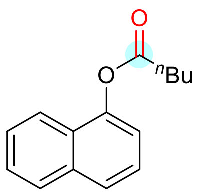


Naphthalen-2-yl pentanoate (13). Prepared according to general procedure using naphthalen-2-ol (72.8 mg, 0.5 mmol) and 1-iodobutane (91 μL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title compound as a light yellow oil (108.5 mg, 95% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 - 7.84 (m, 3H), 7.66 (s, 1H), 7.60 - 7.46 (m, 2H), 7.32 (d, *J* = 8.8 Hz, 1H), 2.69 (t, *J* = 7.5 Hz, 2H), 1.96 - 1.81 (m, 2H), 1.56 (m, *J* = 14.9, 7.4 Hz, 2H), 1.08 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 172.3, 148.3, 133.7, 131.3, 129.2, 127.6, 127.5, 126.4, 125.5, 121.1, 118.4, 34.0, 26.9, 22.2, 13.7.

HRMS (EI): calcd. for $[C_{15}H_{16}O_2]^+$ 228.1145, found 228.1152.

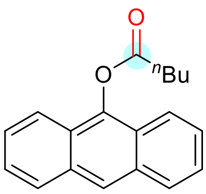


Naphthalen-1-yl pentanoate (14). Prepared according to general procedure using naphthalen-1-ol (72.8 mg, 0.5 mmol) and 1-iodobutane (91 μL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title compound as a colorless oil (112.9 mg, 95% yield).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.78 - 7.67 (m, 2H), 7.58 (d, *J* = 8.2 Hz, 1H), 7.41 - 7.26 (m, 3H), 7.12 (d, *J* = 7.5 Hz, 1H), 2.71 - 2.45 (t, 2H), 1.81 - 1.59 (m, 2H), 1.37 (m, *J* = 14.6, 7.3 Hz, 2H), 0.88 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 172.1, 146.6, 134.5, 127.9, 126.8, 126.3, 125.8, 125.3, 121.0, 118.0, 34.0, 27.0, 22.3, 13.7.

HRMS (EI): calcd. for $[C_{15}H_{16}O_2]^+$ 228.1145, found 228.1144.

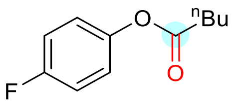


Anthracen-9-yl pentanoate (15). Prepared according to general procedure using anthracen-9-ol (97.1 mg, 0.5 mmol) and 1-iodobutane (91 µL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title compound as a light yellow solid (136.2 mg, 97% yield).

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.40 (s, 1H), 8.11 - 7.94 (m, 4H), 7.60 - 7.47 (m, 4H), 3.04 - 2.88 (t, 2H), 2.11 - 1.94 (m, 2H), 1.75 - 1.56 (m, 2H), 1.13 (t, J = 7.4 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 172.3, 142.1, 131.8, 128.4, 126.1, 125.5, 124.6, 123.9, 121.3, 34.0, 27.3, 22.5, 13.8.

HRMS (EI): calcd. for $[C_{19}H_{18}O_2]^+$ 278.1301, found 278.1301.



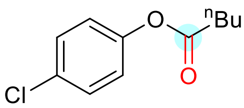
4-Fluorophenyl pentanoate (16). Prepared according to general procedure using 4-fluorophenol (56.1 mg, 0.5 mmol) and 1-iodobutane (91 µL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title compound as a light yellow oil (97.1 mg, 99% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 6.95 (d, J = 1.2 Hz, 2H), 6.94 (s, 2H), 2.48 - 2.41 (t, 2H), 1.69 - 1.59 (m, 2H), 1.41 - 1.28 (m, 2H), 0.87 (t, J = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 172.2, 160.1 (d, J = 244.0 Hz), 146.5 (d, J = 2.9 Hz), 122.9 (d, J = 8.5 Hz), 115.9 (d, J = 23.5 Hz), 33.9, 26.9, 22.1, 1 3.6.

^{19}F NMR (282 MHz, $CDCl_3$) δ -117.3.

HRMS (EI): calcd. for $[C_{11}H_{13}O_2F_1]^+$ 196.0894, found 196.0893.

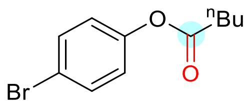


4-Cholrophenyl pentanoate (17). Prepared according to general procedure using 4-chlorophenol (64.3 mg, 0.5 mmol) and 1-iodobutane (91 µL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title compound as a light yellow oil (105 mg, 99% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 (d, J = 8.8 Hz, 2H), 7.05 (d, J = 8.9 H z, 2H), 2.58 (t, 2H), 1.84 - 1.68 (m, 2H), 1.56 - 1.34 (m, 2H), 1.00 (t, J = 7. 4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 171.9, 149.1, 130.9, 129.3, 122.9, 33.9, 26.8, 22.1, 13.6.

HRMS (EI): calcd. for $[C_{11}H_{13}O_2Cl_1]^+$ 212.0599, found 212.0597.

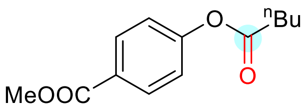


4-Bromophenyl pentanoate (18). Prepared according to general procedure using 4-bromophenol (86.5 mg, 0.5 mmol) and 1-iodobutane (91 μL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title compound as a light yellow oil (126.7 mg, 99% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 (d, J = 8.9 Hz, 2H), 6.88 (d, J = 8.9 Hz, 2H), 2.45 (t, 2H), 1.70 - 1.57 (m, 2H), 1.41 - 1.27 (m, 2H), 0.87 (t, J = 7.3 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 171.8, 149.7, 132.3, 123.3, 118.6, 33.9, 26.8, 22.1, 13.6.

HRMS (EI): calcd. for $[C_{11}H_{13}O_2Br_1]^+$ 256.0093, found 256.0094.

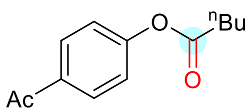


Methyl 4-(pentanoyloxy)benzoate (19). Prepared according to general procedure using methyl 4-hydroxybenzoate (76.1 mg, 0.5 mmol) and 1-iodobutane (91 μL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 10:1) to afford the title compound as a colorless oil (116.9 mg, 99% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.07 (d, J = 8.7 Hz, 2H), 7.16 (d, J = 8.7 Hz, 2H), 3.90 (s, 3H), 2.57 (t, J = 7.5 Hz, 2H), 1.80 - 1.68 (m, 2H), 1.52 - 1.37 (m, 2H), 0.97 (t, J = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 171.5, 166.1, 154.3, 131.0, 127.4, 121.5, 52.0, 34.0, 26.8, 22.1, 13.6.

HRMS (EI): calcd. for $[C_{13}H_{16}O_4]^+$ 236.1043, found 236.1050.

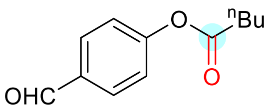


4-Acetylphenyl pentanoate (20). Prepared according to general procedure using 1-(4-hydroxyphenyl)ethan-1-one (68.1 mg, 0.5 mmol) and 1-iodobutane (91 μL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 10:1) to afford the title compound as a colorless oil (102.3 mg, 93% yield).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.89 (d, J = 9.0 Hz, 2H), 7.09 (d, J = 8.9 Hz, 2H), 2.49 (m, 5H), 1.72 - 1.59 (m, 2H), 1.44 - 1.29 (m, 2H), 0.88 (t, J = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 196.7, 171.5, 154.3, 134.5, 129.8, 121.6, 33.9, 26.7, 26.4, 22.1, 13.6.

HRMS (EI): calcd. for $[C_{13}H_{16}O_3]^+$ 220.1094, found 220.1089.

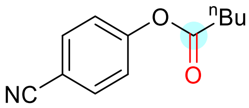


4-Formylphenyl pentanoate (21). Prepared according to general procedure using 4-hydroxybenzaldehyde (61.1 mg, 0.5 mmol) and 1-iodobutane (91 µL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title compound as a light yellow oil (100.1 mg, 97% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 9.96 (s, 1H), 7.89 (d, J = 8.3 Hz, 2H), 7.25 (d, J = 8.9 Hz, 2H), 2.57 (t, J = 7.5 Hz, 2H), 1.80 - 1.66 (m, 2H), 1.51 - 1.36 (m, 2H), 0.96 (t, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 190.7, 171.4, 155.3, 133.7, 131.0, 122.2, 33.9, 26.7, 22.0, 13.5.

HRMS (EI): calcd. for $[C_{12}H_{14}O_3]^+$ 206.0938, found 206.0941.

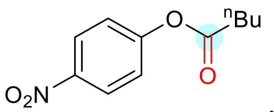


4-Cyanophenyl pentanoate (22). Prepared according to general procedure using 4-hydroxybenzonitrile (59.6 mg, 0.5 mmol) and 1-iodobutane (91 µL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 5:1) to afford the title compound as a light yellow oil (98.9 mg, 97% yield).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.67 (d, J = 8.9 Hz, 2H), 7.22 (d, J = 8.9 Hz, 2H), 2.59 (t, 2H), 1.82 - 1.65 (m, 2H), 1.54 - 1.35 (m, 2H), 0.97 (t, J = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.2, 153.9, 133.4, 122.6, 118.1, 109.4, 33.8, 26.6, 22.0, 13.5.

HRMS (EI): calcd. for $[C_{12}H_{13}O_3N_1]^+$ 203.0941, found 203.0944.

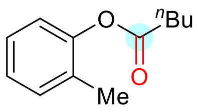


4-Nitrophenyl pentanoate (23). Prepared according to general procedure using 4-nitrophenol (69.6 mg, 0.5 mmol) and 1-iodobutane (91 µL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 5:1) to afford the title compound as a light yellow oil (83.7 mg, 75% yield).

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.19 (d, J = 9.3 Hz, 2H), 7.20 (d, J = 9.3 Hz, 2H), 2.53 (t, 2H), 1.86 - 1.57 (m, 2H), 1.52 - 1.22 (m, 2H), 0.90 (t, J = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.3, 155.5, 145.2, 125.2, 122.4, 34.0, 26.8, 22.2, 13.7.

HRMS (EI): calcd. for $[C_{11}H_{13}O_4N_1]^+$ 223.0839, found 223.0843.

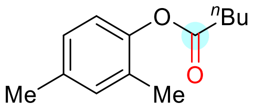


***o*-Tolyl pentanoate (24).** Prepared according to general procedure using *o*-cresol (52 µL, 0.5 mmol) and 1-iodobutane (91 µL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title compound as a colorless oil (95,1 mg, 99% yield).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.32 - 7.12 (m, 3H), 7.05 (d, *J* = 7.7 Hz, 1 H), 2.64 (t, 2H), 2.24 (s, 3H), 1.91 - 1.70 (m, 2H), 1.61 - 1.40 (m, 2H), 1.04 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.9, 149.3, 131.0, 130.0, 126.8, 125.8, 121.8, 33.9, 27.1, 22.2, 16.1, 13.6.

HRMS (EI): calcd. for $[C_{12}H_{16}O_2]^+$ 192,1145, found 192,1149.

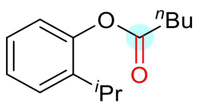


2,4-Dimethylphenyl pentanoate (25). Prepared according to general procedure using 2,4-dimethylphenol (60 µL, 0.5 mmol) and 1-iodobutane (91 µL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title compound as a yellow oil (102 mg, 99% yield).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.10 (s, 1H), 7.07 (d, *J* = 8.1 Hz, 1H), 6.95 (d, *J* = 8.1 Hz, 1H), 2.65 (t, *J* = 7.4 Hz, 2H), 2.37 (s, 3H), 2.21 (s, 3H), 1.9 4 - 1.75 (m, 2H), 1.64 - 1.46 (m, 2H), 1.06 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 172.0, 147.0, 135.3, 131.6, 129.5, 127.3, 121.4, 33.8, 27.0, 22.2, 20.6, 16.0, 13.6.

HRMS (EI): calcd. For $[C_{13}H_{18}O_2]^+$ 206.1301, found 206.1306.

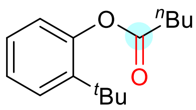


2-Isopropylphenyl pentanoate (26). Prepared according to general procedure using 2-isopropylphenol (67 µL, 0.5 mmol) and 1-iodobutane (91 µL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title compound as a light yellow oil (109 mg, 99% yield).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.26 - 7.16 (m, 1H), 7.14 - 7.04 (m, 2H), 6.9 3 - 6.83 (m, 1H), 2.94 (hept, *J* = 6.9 Hz, 1H), 2.54 - 2.43 (t, 2H), 1.77 - 1. 57 (m, 2H), 1.45 - 1.27 (m, 2H), 1.13 (s, 3H), 1.11 (s, 3H), 0.88 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 172.2, 148.1, 140.0, 126.5, 126.1, 122.2, 34.0, 27.2, 27.0, 22.8, 22.2, 13.6.

HRMS (EI): calcd. For $[C_{14}H_{20}O_2]^+$ 220,1458, found 220,1455.

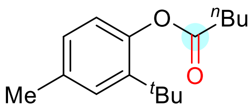


2-(*tert*-Butyl)phenyl pentanoate (27). Prepared according to general procedure using 2-(*tert*-butyl)phenol (77 µL, 0.5 mmol) and 1-iodobutane (91 µL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title compound as a colorless oil (101.3 mg, 87% yield).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.29 (d, *J* = 7.6 Hz, 1H), 7.15 - 7.01 (m, 2 H), 6.89 (d, *J* = 7.7 Hz, 1H), 2.50 (t, *J* = 7.5 Hz, 2H), 1.68 (m, *J* = 15.1, 7. 6 Hz, 2H), 1.37 (m, *J* = 14.5, 7.3 Hz, 2H), 1.26 (s, 9H), 0.89 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 172.2, 149.2, 140.9, 127.1, 126.7, 125.5, 123.9, 34.6, 34.4, 30.1, 26.7, 22.3, 13.7.

HRMS (EI): calcd. For $[C_{15}H_{22}O_2]^+$ 234,1614, found 234,1614.

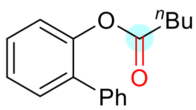


2-(*tert*-Butyl)-4-methylphenyl pentanoate (28). Prepared according to general procedure using 2-(*tert*-butyl)-4-methylphenol (82.1 mg, 0.5 mmol) and 1-iodobutane (91 µL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title compound as a colorless oil (99.3 mg, 80% yield).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.22 (s, 1H), 7.06 (d, *J* = 8.1 Hz, 1H), 6.90 (d, *J* = 8.1 Hz, 1H), 2.63 (t, 2H), 2.37 (s, 3H), 1.93 - 1.72 (m, 2H), 1.60 - 1.45 (m, 2H), 1.39 (s, 9H), 1.03 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 172.5, 146.9, 140.5, 134.9, 127.8, 127.3, 123.7, 34.7, 34.3, 30.2, 29.5, 26.8, 22.3, 21.1, 13.7.

HRMS (EI): calcd. For $[C_{16}H_{24}O_2]^+$ 248.1771, found 248.1772.

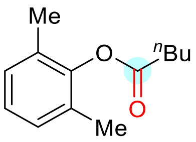


[1,1'-Biphenyl]-2-yl pentanoate (29). Prepared according to general procedure using [1,1'-biphenyl]-2-ol (85.1 mg, 0.5 mmol) and 1-iodobutane (91 µL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title compound as a white solid (125.8 mg, 99% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 - 7.12 (m, 8H), 7.00 (d, *J* = 8.2 Hz, 1 H), 2.22 (t, *J* = 7.5 Hz, 2H), 1.48 - 1.29 (m, 2H), 1.15 - 1.04 (m, 2H), 0.73 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 172.0, 147.7, 137.5, 134.9, 130.7, 128.9, 128. 3, 128.1, 127.3, 126.1, 122.7, 33.8, 26.6, 21.9, 13.6.

HRMS (EI): calcd. For $[C_{17}H_{18}O_2]^+$ 254.1301, found 254.1305.

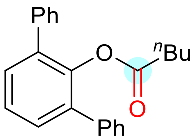


2,6-Dimethylphenyl pentanoate (30). Prepared according to general procedure using 2,6-dimethylphenol (61 mg, 0.5 mmol) and 1-iodobutane (91 µL, 0.8 mmol). The crude product was purified by silica glel chromatography (PE/EA = 20:1) to afford the title compound as a light yellow oil (102 mg, 99% yield).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.19 - 7.09 (m, 3H), 2.68 (t, 2H), 2.23 (s, 6 H), 1.96 - 1.78 (m, 2H), 1.64 - 1.46 (m, 2H), 1.07 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.3, 148.1, 130.0, 128.4, 125.6, 33.7, 27.1, 2 2.3, 16.2, 13.6.

HRMS (ESI-TOF): calcd. For $[C_{13}H_{18}O_2+H]^+$ 229.1204, found 229.1207.

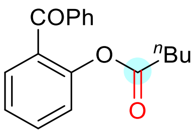


[1,1':3',1''-Terphenyl]-2'-yl pentanoate (31). Prepared according to general procedure using [1,1':3',1''-terphenyl]-2'-ol (123.2 mg, 0.5 mmol) and 1-iodobutane (91 µL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the products as a light yellow oil (162.5 mg, 91% yield and *10% raw materials cannot be separated*).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.68 (d, *J* = 6.9 Hz, 1H), 7.63 - 7.38 (m, 13 H), 2.17 (t, *J* = 7.4 Hz, 2H), 1.46 - 1.26 (m, 2H), 1.20 - 0.97 (m, 2H), 0.85 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.4, 149.2, 145.1, 137.8, 137.5, 135.9, 129.9, 129.9, 129.2, 129.0, 128.7, 128.1, 127.5, 127.3, 126.2, 120.6, 33.5, 26.3, 21.7, 13.5.

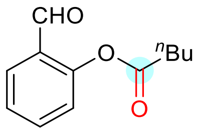
HRMS (EI): calcd. For $[C_{23}H_{22}O_2]^+$ 330.1614, found 330.1613.



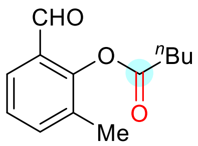
2-Benzoylphenyl pentanoate (32). Prepared according to general procedure using (2-hydroxyphenyl)(phenyl)methanone (99.1 mg, 0.5 mmol) and 1-iodobutane (91 µL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title compound as a light yellow oil (133.2 mg, 94% yield).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.79 (d, *J* = 7.0 Hz, 2H), 7.62 - 7.49 (m, 3 H), 7.45 (t, *J* = 7.5 Hz, 2H), 7.38 - 7.27 (m, 1H), 7.21 (d, *J* = 8.6 Hz, 1H), 2.19 (t, 2H), 1.53 - 1.40 (m, 2H), 1.34 - 1.19 (m, 2H), 0.85 (t, *J* = 7.3 Hz, 3H).

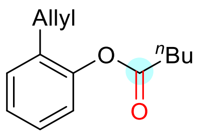
^{13}C NMR (75 MHz, $CDCl_3$) δ 171.3, 148.1, 130.0, 128.4, 125.6, 33.7, 27.1, 2 2.3, 16.2, 13.6.
HRMS (EI): calcd. For $[C_{18}H_{18}O_3]^+$ 282.1251, found 282.1249.



2-Formylphenyl pentanoate (33). Prepared according to general procedure using 2-hydroxybenzaldehyde (53.3 μL, 0.5 mmol) and 1-iodobutane (91 μL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title compound as a colorless oil (78.7 mg, 76% yield).
^{1}H NMR (400 MHz, $CDCl_3$) δ 10.06 (s, 1H), 7.82 (d, *J* = 7.7, 1.7 Hz, 1H), 7.56 (t, *J* = 8.2, 7.4, 1.8 Hz, 1H), 7.37 - 7.28 (t, 1H), 7.11 (d, *J* = 8.1 Hz, 1 H), 2.65 - 2.55 (t, 2H), 1.71 (m, *J* = 15.1, 7.4 Hz, 2H), 1.40 (m, *J* = 14.7, 7. 4 Hz, 2H), 0.92 (t, *J* = 7.4 Hz, 3H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 188.6, 172.0, 151.8, 135.3, 130.8, 128.1, 126.3, 123.5, 33.9, 26.8, 22.3, 13.7.
HRMS (EI): calcd. For $[C_{12}H_{14}O_3]^+$ 206.0938, found 206.0940.



2-Formyl-6-methylphenyl pentanoate (34). Prepared according to general procedure using 2-hydroxy-3-methylbenzaldehyde (61 μL, 0.5 mmol) and 1-iodobutane (91 μL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title compound as a colorless oil (82.6 mg, 75% yield).
^{1}H NMR (300 MHz, $CDCl_3$) δ 10.06 (s, 1H), 7.72 (d, *J* = 7.6 Hz, 1H), 7.51 (d, *J* = 7.5 Hz, 1H), 7.32 (t, *J* = 7.6 Hz, 1H), 2.77 - 2.66 (m, 2H), 2.24 (s, 3H), 1.89 - 1.76 (m, 2H), 1.58 - 1.43 (m, 2H), 1.01 (t, *J* = 7.3 Hz, 3H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 189.0, 171.7, 150.1, 136.9, 132.0, 129.2, 128.2, 126.1, 33.6, 26.8, 22.3, 15.8, 13.7.
HRMS (EI): calcd. For $[C_{13}H_{16}O_3]^+$ 220.1094, found 220.1090.

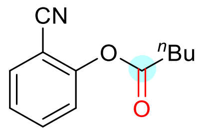


2-Allylphenyl pentanoate (35). Prepared according to general procedure using 2-allylphenol (26 μL, 0.2 mmol) and 1-iodobutane (36 μL, 0.32 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title compound as a colorless oil (43.2 mg, 99% yield).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.20 - 7.02 (m, 3H), 6.97 - 6.86 (m, 1H), 6.01 - 5.62 (m, 1H), 5.04 - 4.80 (m, 2H), 3.21 (d, J = 6.6 Hz, 2H), 2.49 (t, 2H), 1.73 - 1.60 (m, 2H), 1.37 (m, J = 14.5, 7.3 Hz, 2H), 0.89 (t, J = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 189.0, 171.7, 150.1, 136.9, 132.0, 129.2, 128.2, 126.1, 33.6, 26.8, 22.3, 15.8, 13.7.

HRMS (ESI-TOF): calcd. For $[C_{14}H_{18}O_2+H]^+$ 219.1385, found 219.1390.

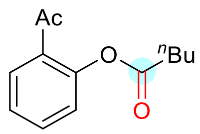


2-Cyanophenyl pentanoate (36). Prepared according to general procedure using 2-hydroxybenzonitrile (59.6 mg, 0.5 mmol) and 1-iodobutane (91 μL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 5:1) to afford the title compound as a colorless oil (78.2 mg, 77% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 - 7.48 (m, 2H), 7.25 (t, J = 7.7 Hz, 1H), 7.20 (d, J = 8.3 Hz, 1H), 2.82 - 2.34 (t, 2H), 1.71 (m, J = 15.2, 7.5 Hz, 2H), 1.46 - 1.20 (m, 2H), 0.90 (t, J = 7.3 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 171.2, 152.4, 133.9, 133.2, 126.1, 123.2, 115.1, 107.1, 33.8, 26.7, 22.1, 13.6.

HRMS (EI): calcd. For $[C_{12}H_{13}O_2N_1]^+$ 203.0941, found 203.0938

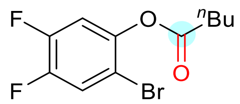


2-Acetylphenyl pentanoate (37). Prepared according to general procedure using 1-(2-hydroxyphenyl)ethan-1-one (69 μL, 0.5 mmol) and 1-iodobutane (91 μL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 10:1) to afford the title compound as a light yellow oil (77.9 mg, 71% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 (d, J = 6.2 Hz, 1H), 7.53 (t, 1H), 7.31 (t, 1H), 7.11 (d, J = 8.1 Hz, 1H), 2.68 - 2.59 (m, 2H), 2.56 (s, 3H), 1.82 - 1.72 (m, 2H), 1.53 - 1.41 (m, 2H), 0.99 (t, J = 7.3 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 197.6, 172.1, 149.0, 133.2, 130.9, 130.1, 125.8, 125.8, 123.7, 34.0, 29.4, 26.5, 22.2, 13.7.

HRMS (EI): calcd. For $[C_{13}H_{16}O_3]^+$ 220.1094, found 220.1093.



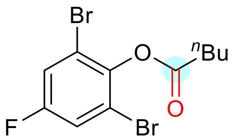
2-Bromo-4,5-difluorophenyl pentanoate (38). Prepared according to general procedure using 2-bromo-4,5-difluorophenol (57 μL, 0.5 mmol) and 1-iodobutane (91 μL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title compound as a light yellow oil (140 mg, 96% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 (dd, *J* = 9.4, 8.0 Hz, 1H), 6.94 (dd, *J* = 10.2, 7.2 Hz, 1H), 2.57 - 2.41 (m, 2H), 1.76 - 1.61 (m, 2H), 1.43 - 1.32 (m, 2H), 0.89 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 170.9, 150.6, 150.5, 149.31 (dd, *J* = 251.5, 1 3.7 Hz), 148.10 (dd, *J* = 251.3, 13.4 Hz), 146.9, 146.8, 144.25 (dd, *J* = 8.4, 3.7 Hz), 121.19 (d, *J* = 21.1 Hz), 113.13 (d, *J* = 20.5 Hz), 110.28 (dd, *J* = 7. 5, 4.2 Hz), 33.7, 26.8, 22.2, 13.7.

^{19}F NMR (282 MHz, $CDCl_3$) δ -134.97, -135.05, -137.87, -137.94.

HRMS (EI): calcd. For $[C_{11}H_{11}O_2Br_1F_2]^+$ 291.9905, found 291.9912.



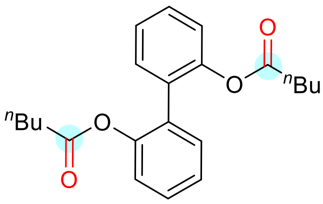
2,6-Dibromo-4-fluorophenyl pentanoate (39). Prepared according to general procedure using 2,6-dibromo-4-fluorophenol (135 mg, 0.5 mmol) and 1-iodobutane (91 μL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title compound as a light yellow oil (117.9 mg, 67% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24 (d, *J* = 7.5 Hz, 2H), 2.72 - 2.47 (t, 2H), 1.78 - 1.62 (m, 2H), 1.52 - 1.30 (m, 2H), 0.90 (t, *J* = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 170.0, 159.40 (d, *J* = 252.9 Hz), 124.43 (d, *J* = 8.9 Hz), 119.68 (d, *J* = 25.6 Hz), 117.79 (d, *J* = 10.7 Hz), 33.6, 26.8, 22. 3, 13.7.

^{19}F NMR (282 MHz, $CDCl_3$) δ -112.98.

HRMS (EI): calcd. For $[C_{11}H_{11}O_2Br_2F_1]^+$ 351.9104, found 351.9101.

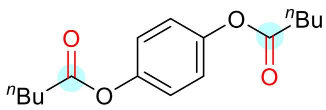


[1,1'-Biphenyl]-2,2'-diyl dipentanoate (40). Prepared according to general procedure using [1,1'-biphenyl]-2,2'-diol (46.6 mg, 0.25 mmol) and 1-iodobutane (91 μL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title compound as a colorless oil (79.2 mg, 90% yield).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.4 (m, *J* = 8.0, 6.4, 2.7 Hz, 2H), 7.4 - 7.2 (m, 4H), 7.2 (d, *J* = 7.9 Hz, 2H), 2.5 - 2.1 (t, 4H), 1.6 - 1.4 (m, 4H), 1.2 (m, *J* = 14.8, 14.3, 7.4 Hz, 4H), 1.0 - 0.8 (t, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.9, 148.2, 131.1, 130.6, 128.8, 125.7, 122.4, 33.7, 26.7, 21.9, 13.6.

HRMS (EI): calcd. For $[C_{22}H_{26}O_4]^+$ 354.1826, found 354.1835.

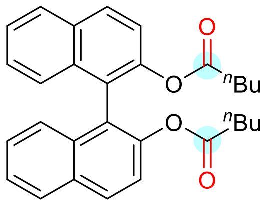


1,4-Phenylene dipentanoate (41). Prepared according to general procedure using hydroquinone (27.5 mg, 0.25 mmol) and 1-iodobutane (91 μL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title compound as a colorless solid (67.6 mg, 97% yield).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.00 (s, 4H), 2.54 - 2.36 (m, 4H), 1.77 - 1.55 (m, 4H), 1.36 (m, J = 14.5, 7.3 Hz, 4H), 0.89 (t, J = 7.3 Hz, 6H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 172.1, 148.1, 122.4, 34.1, 27.0, 22.2, 13.7.

HRMS (EI): calcd. For $[C_{16}H_{22}O_4]^+$ 278.1513, found 278.1510.

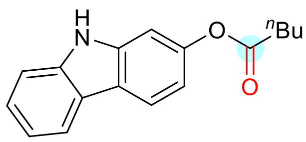


[1,1'-Binaphthalene]-2,2'-diyl dipentanoate (42). Prepared according to general procedure using [1,1'-binaphthalene]-2,2'-diol (71.6 mg, 0.25 mmol) and 1-iodobutane (91 μL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title compound as a light yellow oil (112.4 mg, 99% yield).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.88 (d, J = 8.7 Hz, 2H), 7.81 (d, J = 8.2 H z, 2H), 7.39 - 7.28 (m, 4H), 7.24 - 7.10 (m, 4H), 1.99 (td, J = 7.4, 1.5 Hz, 4H), 1.07 - 0.94 (m, 4H), 0.87 - 0.73 (m, 4H), 0.53 (t, J = 7.2 Hz, 6H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.9, 146.9, 133.4, 131.6, 129.4, 128.0, 126.7, 126.2, 125.7, 123.7, 122.0, 33.8, 26.6, 21.8, 13.6.

HRMS (EI): calcd. For $[C_{30}H_{30}O_4]^+$ 454.2139, found 454.2141.

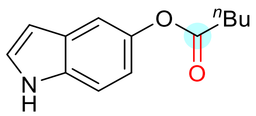


9*H*-Carbazol-2-yl pentanoate (43). Prepared according to general procedure using 9*H*-carbazol-2-ol (91.6 mg, 0.5 mmol) and 1-iodobutane (91 μL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 10:1) to afford the title compound as a light yellow solid (109.9 mg, 82% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.15 (s, 1H), 7.92 (d, J = 7.8 Hz, 1H), 7.88 (d, J = 8.4 Hz, 1H), 7.42 - 7.33 (m, 1H), 7.28 - 7.17 (m, 2H), 7.00 (d, J = 1.7 Hz, 1H), 6.93 (dd, J = 8.4, 2.1 Hz, 1H), 2.77 - 2.63 (m, 2H), 1.95 - 1.8 3 (m, 2H), 1.65 - 1.50 (m, 2H), 1.10 (t, J = 7.4 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 173.4, 149.0, 140.1, 139.9, 125.5, 122.7, 121. 1, 120.7, 120.0, 119.4, 113.0, 110.8, 103.9, 34.3, 27.2, 22.4, 13.9.

HRMS (ESI-TOF): calcd. For $[C_{17}H_{17}NO_2+H]^+$ 290.1156, found 290.1158.

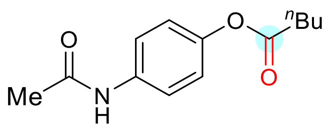


1*H*-Indol-5-yl pentanoate (44). Prepared according to general procedure using 1*H*-indol-5-ol (66.6 mg, 0.5 mmol) and 1-iodobutane (91 µL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 10:1) to afford the title compound as a colorless solid (107.5 mg, 99% yield).

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.38 (s, 1H), 7.40 (dd, *J* = 2.3, 0.7 Hz, 1H), 7.16 (d, *J* = 8.7 Hz, 1H), 7.03 (t, *J* = 2.8 Hz, 1H), 6.92 (dd, *J* = 8.7, 2.3 H z, 1H), 6.62 - 6.40 (m, 1H), 2.69 (t, *J* = 7.5 Hz, 2H), 1.88 (p, *J* = 7.7 Hz, 2 H), 1.67 - 1.47 (m, 2H), 1.08 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 173.7, 144.0, 133.5, 127.9, 125.7, 115.6, 112.2, 111.5, 102.1, 34.1, 27.0, 22.2, 13.6.

HRMS (EI): calcd. For $[C_{13}H_{15}NO_2]^+$ 217.1097, found 217.1097.

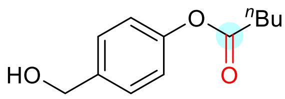


4-Acetamidophenyl pentanoate (45). Prepared according to general procedure using *N*-(4-hydroxyphenyl)acetamide (76 mg, 0.5 mmol) and 1-iodobutane (91 µL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 5:1) to afford the title compound as a brown solid (116.3 mg, 99% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.45 (s, 1H), 7.42 (d, *J* = 8.9 Hz, 2H), 6.92 (d, *J* = 8.9 Hz, 2H), 2.53 (t, *J* = 7.5 Hz, 2H), 2.04 (s, 3H), 1.77 - 1.65 (m, 2H), 1.42 (m, *J* = 14.7, 7.4 Hz, 2H), 0.94 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 172.8, 169.0, 146.5, 135.8, 121.6, 120.9, 33.9, 26.8, 24.0, 22.1, 13.6.

HRMS (ESI-TOF): calcd. For $[C_{13}H_{17}NO_3+H]^+$ 236.1286, found 236.1284.

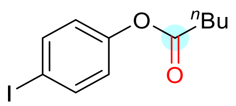


4-(Hydroxymethyl)phenyl pentanoate (46). Prepared according to general procedure using 4-(hydroxymethyl)phenol (62.1 mg, 0.5 mmol) and 1-iodobutane (91 µL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 10:1) to afford the title compound as a light brown oil (82 mg, 80% yield).

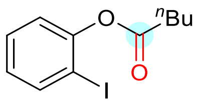
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.33 (d, *J* = 8.8 Hz, 2H), 7.05 (d, *J* = 8.7 H z, 2H), 4.60 (s, 2H), 2.57 (m, 3H), 1.84 - 1.65 (m, 2H), 1.56 - 1.34 (m, 2H), 0.99 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 172.4, 149.9, 138.4, 127.9, 121.5, 64.3, 34.0, 2 6.9, 22.1, 13.6.

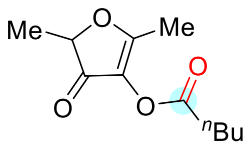
HRMS (EI): calcd. For $[C_{12}H_{16}O_3]^+$ 208.1094, found 208.1010.



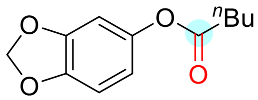
4-Iodophenyl pentanoate (47). Prepared according to general procedure using 4-iodophenol (110 mg, 0.5 mmol) and 1-iodobutane (91 μL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title compound as a light yellow oil (93.3 mg, 61% yield).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 (d, J = 8.9 Hz, 2H), 6.77 (d, J = 8.9 Hz, 2H), 2.50 - 2.39 (m, 2H), 1.71 - 1.57 (m, 2H), 1.42 - 1.28 (m, 2H), 0.88 (t, J = 7.4 Hz, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 171.8, 150.5, 138.3, 123.7, 89.6, 34.0, 26.8, 22.2, 13.7.
HRMS (EI): calcd. For $[C_{11}H_{13}O_2I]^+$ 303.9955, found 303.9958.



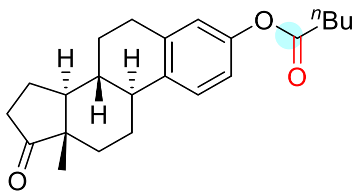
2-Iodophenyl pentanoate (48). Prepared according to general procedure using 2-iodophenol (110 mg, 0.5 mmol) and 1-iodobutane (91 μL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title compound as a light yellow oil (145.6 mg, 96% yield).
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 (d, J = 6.4 Hz, 1H), 7.25 (t, 1H), 6.99 (d, J = 6.6 Hz, 1H), 6.86 (t, 1H), 2.54 (t, 2H), 1.76 - 1.64 (m, 2H), 1.46 - 1.32 (m, 2H), 0.89 (t, J = 7.4 Hz, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 171.4, 151.2, 139.4, 129.4, 127.5, 123.1, 90.6, 34.2, 26.9, 22.4, 13.8.
HRMS (EI): calcd. For $[C_{11}H_{13}O_2I]^+$ 303.9955, found 303.9960.



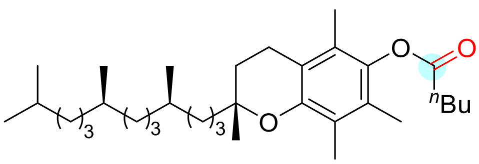
2,5-Dimethyl-4-oxo-4,5-dihydrofuran-3-yl pentanoate (49). Prepared according to general procedure using *Furaneol* (64.1 mg, 0.5 mmol) and 1-iodobutane (91 μL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title compound as a light yellow oil (80.1 mg, 76% yield).
^{1}H NMR (300 MHz, $CDCl_3$) δ 4.49 (q, J = 7.3 Hz, 1H), 2.46 (t, 2H), 2.08 (s, 3H), 1.69 - 1.56 (m, 2H), 1.42 (d, J = 7.2 Hz, 3H), 1.39 - 1.27 (m, 2H), 0.87 (t, J = 7.3 Hz, 3H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 195.7, 179.8, 170.4, 128.9, 81.2, 33.1, 26.7, 22.0, 16.2, 13.9, 13.5.
HRMS (EI): calcd. For $[C_{11}H_{16}O_4]^+$ 212.1043, found 212.1043.



Benzo[*d*][1,3]dioxol-5-yl pentanoate (50). Prepared according to general procedure using *Sesamol* (69.1 mg, 0.5 mmol) and 1-iodobutane (91 μL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title compound as a yellow oil (109.9 mg, 99% yield).
^{1}H NMR (300 MHz, $CDCl_3$) δ 6.67 (d, *J* = 8.4 Hz, 1H), 6.50 (d, *J* = 2.0 Hz, 1H), 6.42 (dd, *J* = 8.4, 2.3 Hz, 1H), 5.86 (m, 2H), 2.49 - 2.35 (t, 2H), 1.71 - 1.54 (m, 2H), 1.34 (m, *J* = 14.5, 7.4 Hz, 2H), 0.94 - 0.76 (t, 3H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 172.4, 147.9, 145.1, 145.0, 113.8, 107.8, 103.6, 101.6, 33.9, 26.9, 22.1, 13.6.
HRMS (EI): calcd. For $[C_{12}H_{14}O_4]^+$ 222.0887, found 222.0882.



(8*R*,9*S*,13*S*,14*S*)-13-Methyl-17-oxo-7,8,9,11,12,13,14,15,16,17-decahydro-6H-cyclopenta[a]phenanthren-3-yl pentanoate (51). Prepared according to general procedure using *Estrone* (135.2 mg, 0.5 mmol) and 1-iodobutane (91 μL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title compound as a white solid (169.4 mg, 96% yield).
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.28 (d, *J* = 8.4 Hz, 1H), 6.84 (d, *J* = 8.4 Hz, 1H), 6.81 (s, 1H), 2.91 (dd, *J* = 8.0, 3.4 Hz, 2H), 2.60 - 2.34 (m, 4H), 2.35 - 1.90 (m, 5H), 1.80 - 1.36 (m, 10H), 0.97 (t, *J* = 7.3 Hz, 3H), 0.91 (s, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ 220.5, 172.4, 148.5, 137.8, 137.1, 126.2, 121.4, 118.6, 50.2, 47.8, 44.0, 37.8, 35.7, 33.9, 31.4, 29.2, 26.9, 26.2, 25.6, 22.1, 21.4, 13.7, 13.6.
HRMS (EI): calcd. For $[C_{23}H_{30}O_3]^+$ 354.2190, found 354.2182.

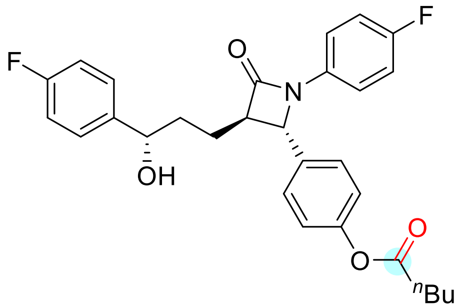


(*R*)-2,5,7,8-Tetramethyl-2-((4*R*,8*R*)-4,8,12-trimethyltridecyl)chroman-6-yl pentanoate (52). Prepared according to general procedure using *Vitamin E* (215.4 mg, 0.5 mmol) and 1-iodobutane (91 μL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title compound as a yellow oil (235.8 mg, 92% yield).

^{1}H NMR (300 MHz, $CDCl_3$) δ 2.64 (dd, J = 8.1, 7.2 Hz, 4H), 2.14 (s, 3H), 2.06 (s, 3H), 2.02 (s, 3H), 1.93 - 1.73 (m, 4H), 1.67 - 1.10 (m, 27H), 1.03 (t, J = 7.3 Hz, 3H), 0.96 - 0.84 (m, 12H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 172.22, 149.26, 140.47, 126.61, 124.81, 122.90, 117.22, 74.90, 39.33, 37.48, 37.42, 37.35, 37.25, 33.80, 32.72, 32.64, 31.03, 27.93, 27.18, 24.78, 24.76, 24.40, 22.67, 22.58, 22.41, 20.98, 20.55, 19.70, 19.63, 19.54, 13.69, 12.88, 12.03, 11.76.

HRMS (EI): calcd. For $[C_{34}H_{58}O_3]^+$ 514.4381, found 514.4379.



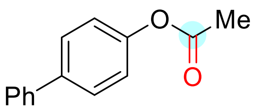
4-((2*S*,3*R*)-1-(4-Fluorophenyl)-3-((*S*)-3-(4-fluorophenyl)-3-hydroxypropyl)-4-oxoazetidin-2-yl)phenyl pentanoate (53). Prepared according to general procedure using *Ezetimibe* (204.7 mg, 0.5 mmol) and 1-iodobutane (91 µL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 10:1) to afford the title compound as a brown solid (149 mg, 61% yield).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.39 - 7.17 (m, 6H), 7.11 (d, J = 8.6 Hz, 2H), 6.96 (dt, J = 20.9, 8.6 Hz, 4H), 4.90 - 4.52 (m, 2H), 3.13 - 2.94 (m, 1H), 2.57 (t, J = 7.5 Hz, 2H), 2.40 - 2.13 (m, 1H), 2.06 - 1.84 (m, 4H), 1.75 (p, J = 7.4 Hz, 2H), 1.46 (dp, J = 14.6, 7.3 Hz, 2H), 0.98 (t, J = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 172.02, 167.32, 163.49, 160.44, 160.24, 157.21, 150.66, 140.04, 140.01, 134.69, 133.50, 133.47, 127.27, 127.17, 126.70, 122.29, 118.27, 118.17, 115.83, 115.53, 115.16, 114.88, 72.65, 60.59, 60.11, 36.37, 33.83, 26.72, 24.77, 22.00, 13.51.

^{19}F NMR (282 MHz, $CDCl_3$) δ -114.99, -117.62.

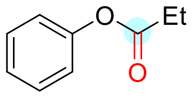
HRMS (ESI-TOF): calcd. For $[C_{29}H_{29}NO_4F_2+H]^+$ 494.2143, found 494.2148.



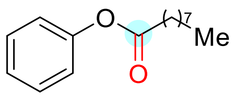
[1,1'-Biphenyl]-4-yl acetate (54). Prepared according to general procedure using [1,1'-biphenyl]-4-ol (85.1 mg, 0.5 mmol) and iodomethane (50 µL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title compound as a white solid (103.9 mg, 97% yield).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.48 - 7.41 (m, 4H), 7.30 (t, J = 7.3 Hz, 2H), 7.21 (t, 1H), 7.03 (d, J = 8.9 Hz, 2H), 2.17 (s, 3H).

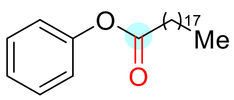
^{13}**C NMR** (75 MHz, $CDCl_3$) δ 169.4, 150.0, 140.2, 138.8, 128.7, 128.0, 127.2, 127.0, 121.7, 21.0.
HRMS (EI): calcd. For $[C_{14}H_{12}O_2]^+$ 212.0832, found 212.0834.



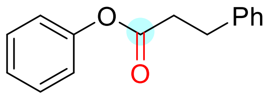
Phenyl propionate (55). Prepared according to general procedure using phenol (47.1 mg, 0.5 mmol) and iodoethane (64 μL, 0.8 mmol). The crude product w as purified by silica gel chromatography (PE/EA = 20:1) to afford the title co mpound as a light yellow oil (72.9 mg, 99% yield).
^{1}**H NMR** (300 MHz, $CDCl_3$) δ 7.42 (t, 2H), 7.26 (t, 1H), 7.13 (d, J = 7.4 H z, 2H), 2.63 (q, J = 7.5 Hz, 2H), 1.31 (t, J = 7.6 Hz, 3H).
^{13}**C NMR** (75 MHz, $CDCl_3$) δ 172.8, 150.7, 129.3, 125.6, 121.5, 77.4, 77.0, 7 6.6, 27.7, 9.0.
HRMS (EI): calcd. For $[C_9H_{10}O_2]^+$ 150.0675, found 150.0676.



Phenyl nonanoate (56). Prepared according to general procedure using phenol (47.1 mg, 0.5 mmol) and 1-iodooctane (144 μL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title compound as a light yellow oil (115.9 mg, 99% yield).
^{1}**H NMR** (300 MHz, $CDCl_3$) δ 7.48 - 7.36 (m, 2H), 7.32 - 7.20 (m, 1H), 7.1 4 (d, J = 7.4 Hz, 2H), 2.66 - 2.53 (t, 2H), 1.81 (m, J = 7.2 Hz, 2H), 1.60 - 1.29 (m, 11H), 1.06 - 0.90 (t, 3H).
^{13}**C NMR** (75 MHz, $CDCl_3$) δ 172.1, 150.7, 129.2, 125.5, 121.5, 77.4, 77.0, 7 6.6, 34.3, 31.7, 29.1, 29.0, 29.0, 24.8, 22.5, 14.0.
HRMS (EI): calcd. For $[C_{15}H_{22}O_2]^+$ 234.1614, found 234.1625.



Phenyl nonadecanoate (57). Prepared according to general procedure using phe nol (47.1 mg, 0.5 mmol) and 1-iodooctadecane (304 mg, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford t he title compound as a white solid (185.3 mg, 99% yield).
^{1}**H NMR** (300 MHz, $CDCl_3$) δ 7.42 (t, 2H), 7.26 (t, 1H), 7.13 (d, J = 7.4 H z, 2H), 2.60 (t, J = 7.5 Hz, 2H), 1.92 - 1.70 (m, 2H), 1.34 (m, 30H), 1.02 - 0.90 (t, 3H).
^{13}**C NMR** (75 MHz, $CDCl_3$) δ 172.1, 150.7, 129.2, 125.5, 121.5, 34.3, 31.9, 2 9.7, 29.6, 29.6, 29.4, 29.3, 29.2, 29.1, 24.9, 22.7, 14.1.
HRMS (EI): calcd. For $[C_{25}H_{42}O_2]^+$ 374.3179, found 374.3174.

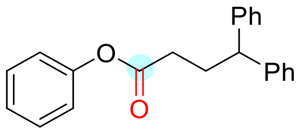


Phenyl 3-phenylpropanoate (58). Prepared according to general procedure using phenol (18.8 mg, 0.2 mmol) and (2-iodoethyl)benzene (74.3 mg, 0.32 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title compound as a colorless oil (43.5 mg, 96% yield).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.47 - 7.23 (m, 9H), 7.13 - 7.03 (m, 2H), 3.15 (t, *J* = 7.6 Hz, 2H), 2.95 (t, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.3, 150.6, 140.1, 129.3, 128.5, 128.3, 126.4, 125.7, 121.5, 35.9, 30.9.

HRMS (EI): calcd. For $[C_{15}H_{14}O_2]^+$ 226.0988, found 226.0990.

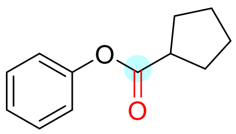


Phenyl 3-phenylpropanoate (59). Prepared according to general procedure using phenol (18.8 mg, 0.2 mmol) and (3-iodopropane-1,1-diyl)dibenzene (103 mg, 0.32 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title compound as a colorless oil (49.6 mg, 78% yield).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.52 - 7.21 (m, 13H), 7.12 (d, *J* = 7.4 Hz, 2H), 4.18 - 4.05 (m, 1H), 2.68 - 2.50 (m, 4H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.8, 150.6, 143.9, 129.3, 128.6, 127.8, 126.4, 125.7, 121.5, 50.4, 32.8, 30.5.

HRMS (EI): calcd. For $[C_{22}H_{20}O_2]^+$ 316.1458, found 316.1460.

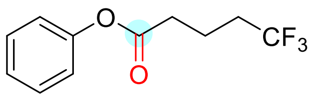


Phenyl cyclopentanecarboxylate (60). Prepared according to general procedure using phenol (47.1 mg, 0.5 mmol) and iodocyclopentane (93 µL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title compound as a light yellow oil (43,7 mg, 46% yield).

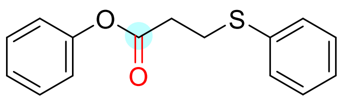
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.36 - 7.24 (m, 2H), 7.20 - 7.08 (m, 1H), 7.00 (d, *J* = 7.4 Hz, 2H), 3.00 - 2.83 (m, 1H), 2.01 - 1.47 (m, 9H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 175.2, 150.9, 129.3, 125.6, 121.5, 43.9, 30.1, 25.9.

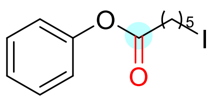
HRMS (EI): calcd. For $[C_{12}H_{14}O_2]^+$ 190,0988, found 192,0992.



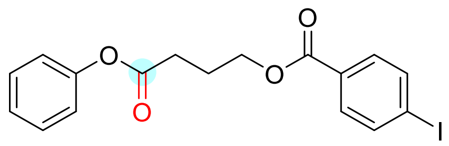
Phenyl 5,5,5-trifluoropentanoate (61). Prepared according to general procedure using phenol (47.1 mg, 0.5 mmol) and 1,1,1-trifluoro-4-iodobutane (103 μL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title compound as a colorless oil (114.9 mg, 99% yield).
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.3 - 7.2 (t, 2H), 7.2 - 7.1 (t, 1H), 7.0 (d, J = 7.4 Hz, 2H), 2.5 (t, J = 7.3 Hz, 2H), 2.2 - 2.0 (m, 2H), 2.0 - 1.8 (m, 2H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 170.9, 150.5, 129.4, 125.8, 121.4, 33.3, 32.9, 32.7, 32.5, 32.1, 17.3 (q, J = 3.3 Hz).
^{19}F NMR (282 MHz, $CDCl_3$) δ -66.26.
HRMS (EI): calcd. For $[C_{11}H_{11}O_2F_3]^+$ 232.0706, found 232.0702.



Phenyl 3-(phenylthio)propanoate (62). Prepared according to general procedure using phenol (18.8 mg, 0.2 mmol) and (2-iodoethyl)(phenyl)sulfane (84.5 mg, 0.32 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title compound as a light yellow oil (127.7 mg, 99% yield).
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.58 - 7.31 (m, 6H), 7.35 - 7.22 (m, 2H), 7.20 - 7.08 (m, 2H), 3.33 (t, J = 7.4 Hz, 2H), 2.92 (t, J = 7.4 Hz, 2H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 170.2, 150.5, 134.9, 130.4, 129.4, 129.1, 126.7, 125.8, 121.4, 34.5, 29.2.
HRMS (ESI-TOF): calcd. For $[C_{15}H_{14}O_2S+H]^+$ 259.0793, found 259.0790.



Phenyl 6-iodohexanoate (63). Prepared according to general procedure using phenol (47.1 mg, 0.5 mmol) and 1,5-diiodopentane (119 μL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title compound as a colorless oil (61 mg, 38% yield).
^{1}H NMR (300 MHz, $CDCl_3$) δ 7.36 - 7.22 (m, 2H), 7.20 - 7.08 (m, 1H), 7.00 (d, J = 7.4 Hz, 2H), 3.13 (t, J = 6.9 Hz, 2H), 2.50 (t, J = 7.4 Hz, 2H), 1.88 - 1.62 (m, 4H), 1.54 - 1.35 (m, 2H).
^{13}C NMR (75 MHz, $CDCl_3$) δ 171.9, 150.7, 129.4, 125.8, 121.6, 34.1, 33.1, 29.9, 23.9, 6.6.
HRMS (ESI-TOF): calcd. For $[C_{12}H_{15}O_2I+H]^+$ 319.0195, found 319.0201.

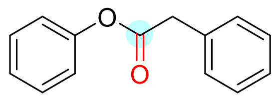


4-Oxo-4-phenoxybutyl 4-iodobenzoate (64). Prepared according to general proc edure using phenol (18.8 mg, 0.2 mmol) and 3-iodopropyl 4-iodobenzoate (133. 1 mg, 0.32 mmol). The crude product was purified by silica gel chromatograp hy (PE/EA = 20:1) to afford the title compound as a yellow oil (61 mg, 38% yield).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.87 - 7.69 (m, 4H), 7.43 - 7.30 (m, 2H), 7.2 9 - 7.17 (m, 1H), 7.06 (d, J = 7.4 Hz, 2H), 4.44 (t, J = 6.3 Hz, 2H), 2.74 (t, J = 7.3 Hz, 2H), 2.33 - 2.16 (m, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.3, 166.0, 150.6, 137.8, 131.1, 129.5, 129.4, 125.9, 121.5, 100.9, 64.1, 31.1, 24.1.

HRMS (ESI-TOF): calcd. For $[C_{17}H_{15}O_4I+H]^+$ 411.0093, found 411.0098.

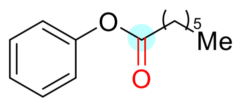


Phenyl 2-phenylacetate (65). Prepared according to general procedure using ph enol (47.1 mg, 0.5 mmol) and (bromomethyl)benzene (95 µL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to aff ord the title compound as a light yellow oil (105 mg, 99% yield).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.56 - 7.40 (m, 1H), 7.31 (t, J = 7.4 Hz, 0H), 7.19 (d, J = 8.6 Hz, 0H), 3.96 (s, 1H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 150.6, 133.4, 129.2, 129.2, 128.6, 127.2, 125.7, 121.3, 41.2.

HRMS (EI): calcd. For $[C_{14}H_{12}O_2]^+$ 212.0832, found 212.0830.

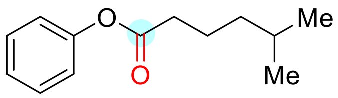


Phenyl heptanoate (66). Prepared according to general procedure using phenol (47.1 mg, 0.5 mmol) and 1-bromohexane (112 µL, 0.8 mmol). The crude prod uct was purified by silica gel chromatography (PE/EA = 20:1) to afford the tit le compound as a colorless oil (102 mg, 99% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 (t, 2H), 7.26 (t, J = 7.4 Hz, 1H), 7.13 (d, J = 7.4 Hz, 2H), 2.60 (t, 2H), 1.96 - 1.65 (m, 2H), 1.58 - 1.21 (m, 6H), 0.98 (t, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 172.1, 150.7, 129.2, 125.5, 121.5, 34.3, 31.4, 28.7, 24.8, 22.4, 13.9.

HRMS (EI): calcd. For $[C_{13}H_{18}O_2]^+$ 206.1301, found 206.1301.

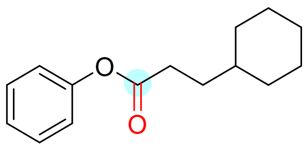


Phenyl 5-methylhexanoate (67). Prepared according to general procedure using phenol (47.1 mg, 0.5 mmol) and 1-bromo-4-methylpentane (117 µL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title compound as a colorless oil (91.1 mg, 88% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44 - 7.34 (t, 2H), 7.26 (t, *J* = 7.4 Hz, 1H), 7.12 (d, *J* = 7.5 Hz, 2H), 2.58 (t, *J* = 7.5 Hz, 2H), 1.88 - 1.74 (m, 2H), 1.6 5 (m, *J* = 13.3, 6.7 Hz, 1H), 1.40 - 1.25 (m, 2H), 0.98 (s, 3H), 0.96 (s, 3H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 172.2, 150.7, 129.3, 125.6, 121.5, 38.3, 34.5, 27.7, 22.8, 22.4.

HRMS (EI): calcd. For $[C_{13}H_{18}O_2]^+$ 206.1301, found 206.1305.

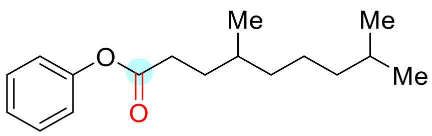


Phenyl 3-cyclohexylpropanoate (68). Prepared according to general procedure using phenol (47.1 mg, 0.5 mmol) and (2-bromoethyl)cyclohexane (125 µL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title compound as a colorless oil (92.9 mg, 80% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28 (t, 2H), 7.12 (t, *J* = 7.4 Hz, 1H), 6.99 (d, *J* = 7.5 Hz, 2H), 2.47 (t, 2H), 1.73 - 1.52 (m, 7H), 1.28 - 1.02 (m, 4H), 0.95 - 0.78 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 172.5, 150.7, 129.3, 125.6, 121.5, 37.2, 32.9, 32.3, 32.0, 26.5, 26.2.

HRMS (EI): calcd. For $[C_{15}H_{20}O_2]^+$ 232.1458, found 232.1461.

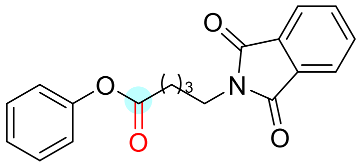


Phenyl 4,8-dimethylnonanoate (69). Prepared according to general procedure u sing phenol (47.1 mg, 0.5 mmol) and 1-bromo-3,7-dimethyloctane (166 µL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title compound as a colorless oil (98.8 mg, 75% yield).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.33 - 7.22 (m, 2H), 7.18 - 7.06 (m, 1H), 6.9 9 (d, *J* = 7.4 Hz, 2H), 2.57 - 2.40 (m, 2H), 1.85 - 1.65 (m, 1H), 1.57 - 1.39 (m, 3H), 1.30 - 1.16 (m, 4H), 1.14 - 1.01 (m, 4H), 0.85 (d, *J* = 6.4 Hz, 3 H), 0.81 (s, 3H), 0.78 (s, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 172.4, 150.7, 129.3, 125.6, 121.5, 39.2, 36.9, 3 2.4, 32.2, 31.8, 27.9, 24.6, 22.6, 22.6, 19.3.

HRMS (ESI-TOF): calcd. For $[C_{17}H_{26}O_2+H]^+$ 263.2011, found 263.2018.

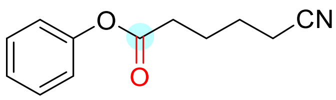


Phenyl 5-(1,3-dioxoisoindolin-2-yl)pentanoate (70). Prepared according to gene ral procedure using phenol (47.1 mg, 0.5 mmol) and 2-(4-bromobutyl)isoindolin e-1,3-dione (225.7 mg, 0.8 mmol). The crude product was purified by silica ge l chromatography (PE/EA = 20:1) to afford the title compound as a colorless s olid (128.9 mg, 80% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 (dd, J = 5.5, 3.0 Hz, 2H), 7.68 (dd, J = 5.4, 3.1 Hz, 2H), 7.35 (t, J = 8.0 Hz, 2H), 7.20 (t, J = 7.4 Hz, 1H), 7.07 (d, J = 7.5 Hz, 2H), 3.74 (t, J = 6.7 Hz, 2H), 2.62 (t, J = 7.0 Hz, 2H), 1.87 - 1.74 (m, 4H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 171.4, 168.1, 150.5, 133.7, 131.8, 129.2, 125. 5, 123.0, 121.4, 37.2, 33.5, 27.7, 21.9.

HRMS (ESI-TOF): calcd. For $[C_{19}H_{17}NO_4+H]^+$ 346.1055, found 346.1053.

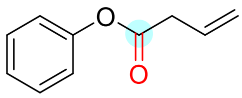


Phenyl 5-cyanopentanoate (71). Prepared according to general procedure using phenol (47.1 mg, 0.5 mmol) and 5-bromopentanenitrile (93 μL, 0.8 mmol). Th e crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title compound as a yellow oil (85.1 mg, 84% yield).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.40 (t, 2H), 7.25 (t, 1H), 7.10 (d, J = 7.4 H z, 2H), 2.62 (t, J = 7.2 Hz, 2H), 2.39 (t, 2H), 1.96 - 1.85 (m, 2H), 1.84 - 1. 72 (m, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.1, 150.4, 129.3, 125.7, 121.3, 119.2, 33.1, 24.6, 23.6, 16.8.

HRMS (ESI-TOF): calcd. For $[C_{12}H_{13}NO_2+H]^+$ 226.0843, found 226.0842.

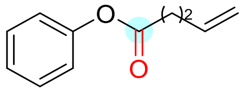


Phenyl but-3-enoate (72). Prepared according to general procedure using pheno l (47.1 mg, 0.5 mmol) and 3-bromoprop-1-ene (69 μL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford th e title compound as a light yellow oil (56.9 mg, 70% yield).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.37 - 7.25 (t, 2H), 7.22 - 7.09 (t, 2H), 7.02 (d, J = 7.5 Hz, 2H), 6.06 - 5.83 (m, 1H), 5.30 - 5.11 (m, 2H), 3.27 (dt, J = 6.9, 1.5 Hz, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 169.9, 150.6, 129.6, 129.4, 125.8, 121.5, 119.2, 39.1.

HRMS (EI): calcd. For $[C_{10}H_{10}O_2]^+$ 16.0675, found 162.0675.

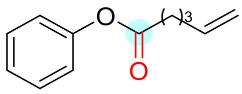


Phenyl pent-4-enoate (73). Prepared according to general procedure using phen ol (47.1 mg, 0.5 mmol) and 4-bromobut-1-ene (81 μL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford th e title compound as a light yellow oil (80.1 mg, 91% yield).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.42 (t, 2H), 7.26 (t, 1H), 7.13 (d, J = 7.4 H z, 2H), 6.07 - 5.82 (m, 1H), 5.34 - 5.00 (m, 2H), 2.71 (t, 2H), 2.62 - 2.49 (m, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 169.9, 150.6, 129.6, 129.4, 125.8, 121.5, 119.2, 39.1.

HRMS (EI): calcd. For $[C_{11}H_{12}O_2]^+$ 176.0832, found 176.0831.

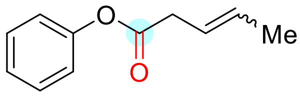


Phenyl hex-5-enoate (74). Prepared according to general procedure using phen ol (47.1 mg, 0.5 mmol) and 5-bromopent-1-ene (95 μL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford th e title compound as a light yellow oil (82.7 mg, 87% yield).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 - 7.37 (m, 1H), 7.26 (t, J = 7.5 Hz, 1H), 7.13 (d, J = 7.5 Hz, 1H), 5.88 (td, J = 16.9, 6.7 Hz, 1H), 5.18 - 5.05 (m, 1 H), 2.62 (t, J = 7.5 Hz, 2H), 2.29 - 2.18 (m, 2H), 1.91 (p, J = 7.6 Hz, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 172.0, 150.7, 137.5, 129.4, 125.7, 121.5, 115. 6, 33.6, 33.0, 24.0.

HRMS (EI): calcd. For $[C_{12}H_{14}O_2]^+$ 190.0988, found 190.0983.

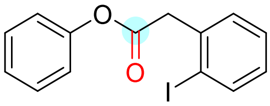


Phenyl pent-3-enoate (75). Prepared according to general procedure using phenol (47.1 mg, 0.5 mmol) and *trans*-1-bromo-2-butene (81.5 μL, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title compound as a light yellow oil (61.1 mg, 69% yield, Z : E = 1 : 2.3).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.33 - 7.25 (m, 3H), 7.18 - 7.08 (m, 1.6H), 7. 04 - 6.97 (m, 3H), 5.86 - 5.40 (m, 2.8H), 3.26 (dt, J = 6.4, 1.0 Hz, 0.87H), 3.18 (dd, J = 5.4, 1.3 Hz, 2H), 1.77 - 1.58 (m, 5.1H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 170.5, 170.4, 150.7, 130.1, 129.3, 129.3, 128.2, 125.7, 122.1, 121.5, 121.5, 121.1, 114.6, 38.1, 32.8, 17.9, 13.0.

HRMS (ESI-TOF): calcd. For $[C_{11}H_{12}O_2+H]^+$ 177.0915, found 177.0916.

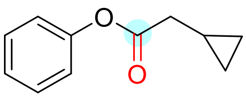


Phenyl 2-(2-iodophenyl)acetate (76). Prepared according to general procedure using phenol (47.1 mg, 0.5 mmol) and 1-(bromomethyl)-2-iodobenzene (238 mg, 0.8 mmol). The crude product was purified by silica gel chromatography (PE/EA = 20:1) to afford the title compound as a light yellow oil (61.1 mg, 75% yield).

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.93 (d, J = 7.9, 1.1 Hz, 1H), 7.48 - 7.37 (m, 4H), 7.36 - 7.18 (m, 1H), 7.19 (dd, J = 8.6, 1.2 Hz, 2H), 7.04 (td, J = 7.9, 6.9, 2.2 Hz, 1H), 4.09 (s, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 168.8, 150.6, 139.5, 137.2, 130.7, 129.3, 129.0, 128.5, 125.8, 121.4, 100.9, 46.3.

HRMS (EI): calcd. For $[C_{14}H_{11}O_2I_1]^+$ 337.9798, found 337.9791.



Phenyl 2-cyclopropylacetate (81). Prepared according to *Mechanistic Studies*. The yield of **73/81** = 5/1.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.35 - 7.21 (m, 2.7H), 7.19 - 7.07 (m, 1.4H), 7.06 - 6.94 (m, 2.7H), 6.01 - 5.55 (m, 1H), 5.20 - 4.60 (dd, 2.1H), 2.64 - 2.52 (t, 2.1H), 2.49 - 2.32 (m, 2.5H), 0.65 - 0.40 (m, 0.39H), 0.18 (m, J = 6.0 Hz, 0.39H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.4, 150.6, 136.3, 129.3, 125.7, 121.5, 115.8, 39.4, 33.6, 28.8, 6.9, 4.4.

HRMS (EI): calcd. For $[C_{11}H_{12}O_2]^+$ 176.0832, found 176.0831.

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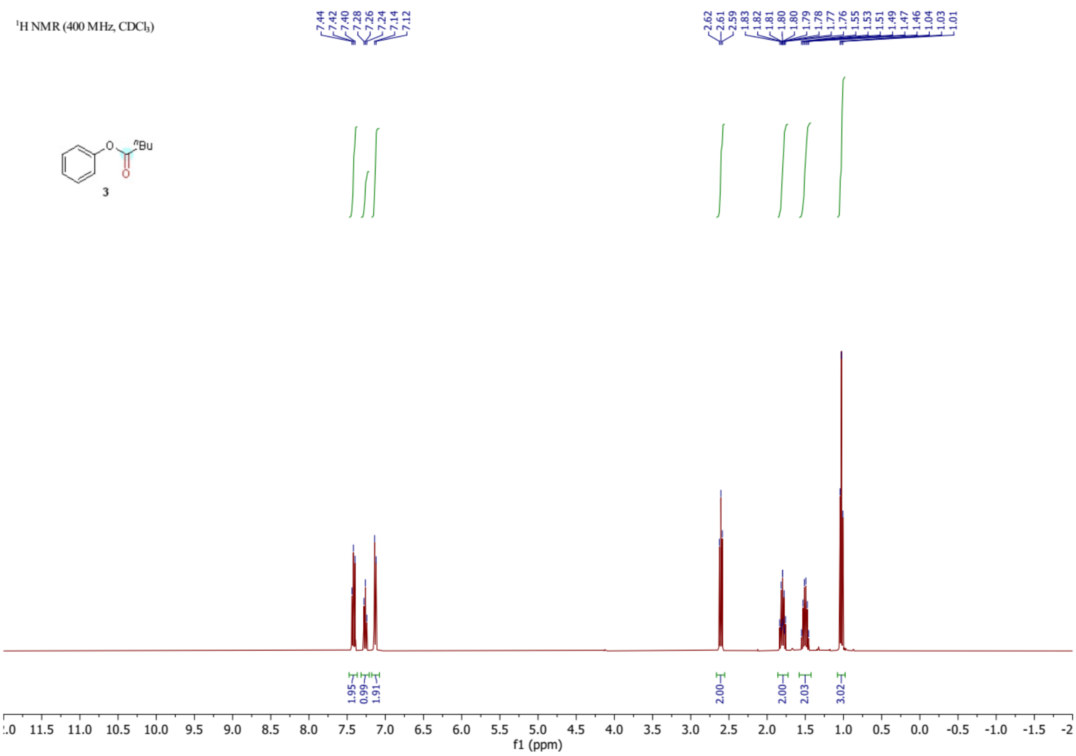
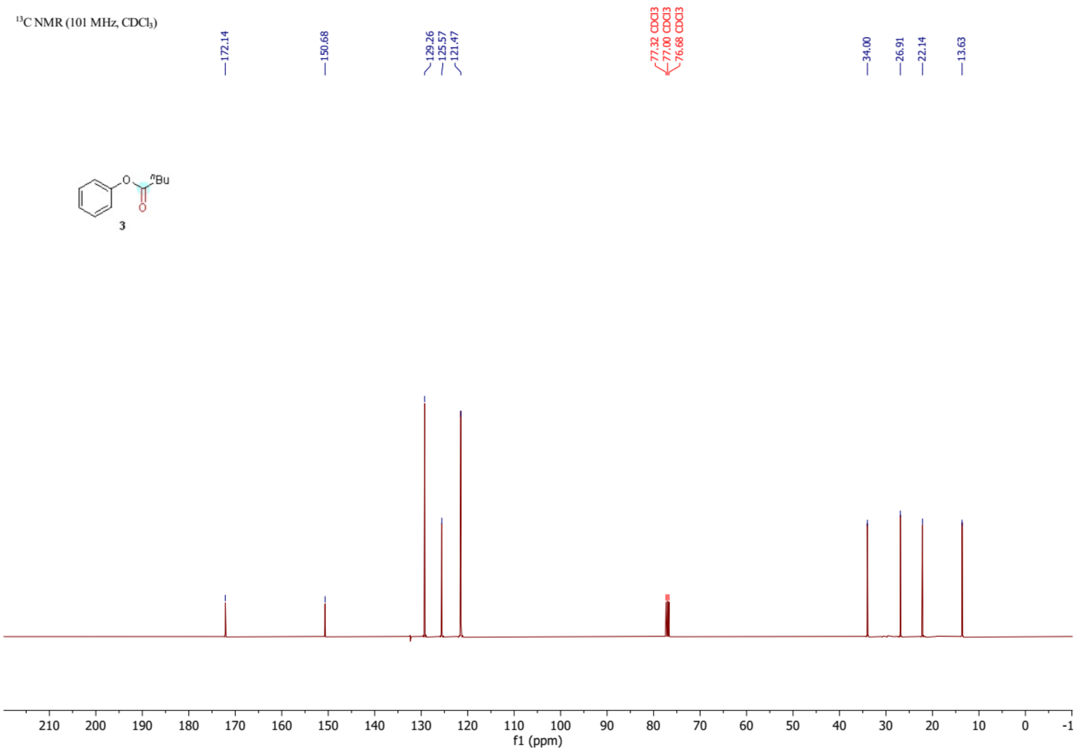
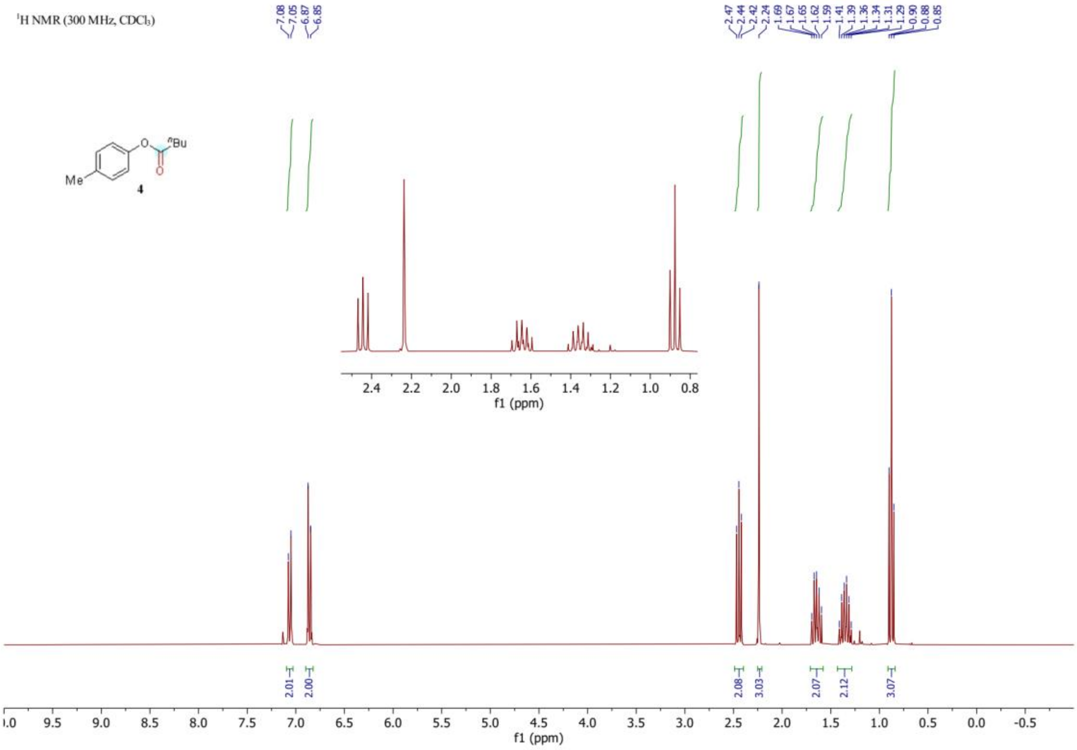
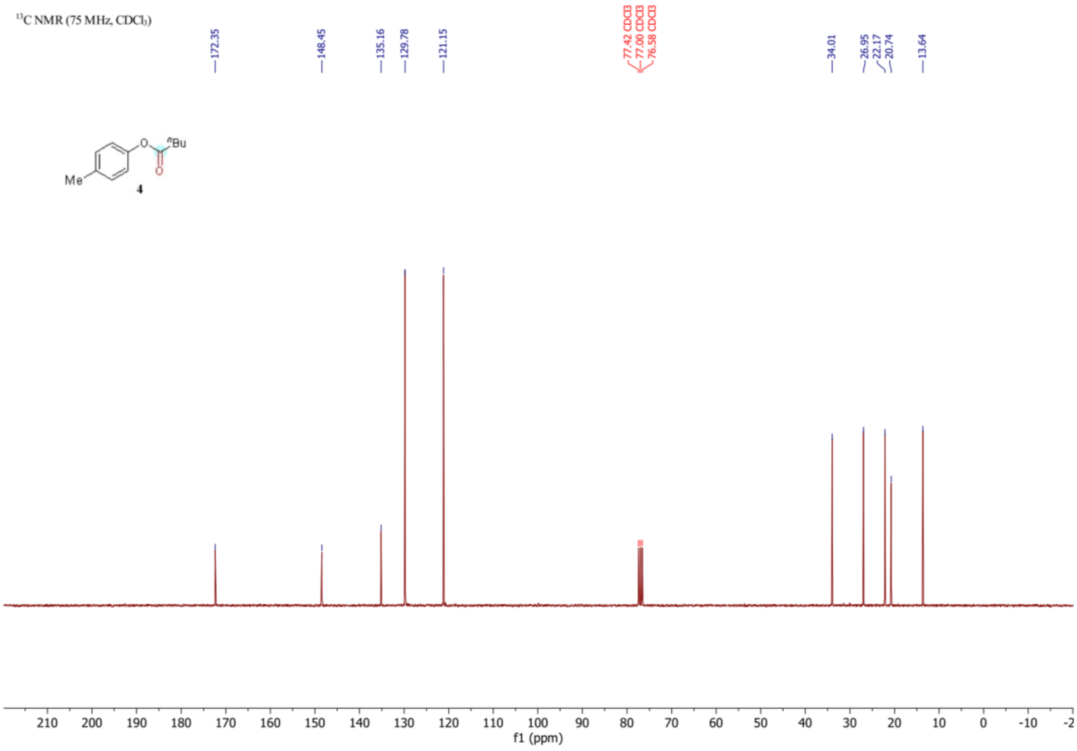
NMR Spectra





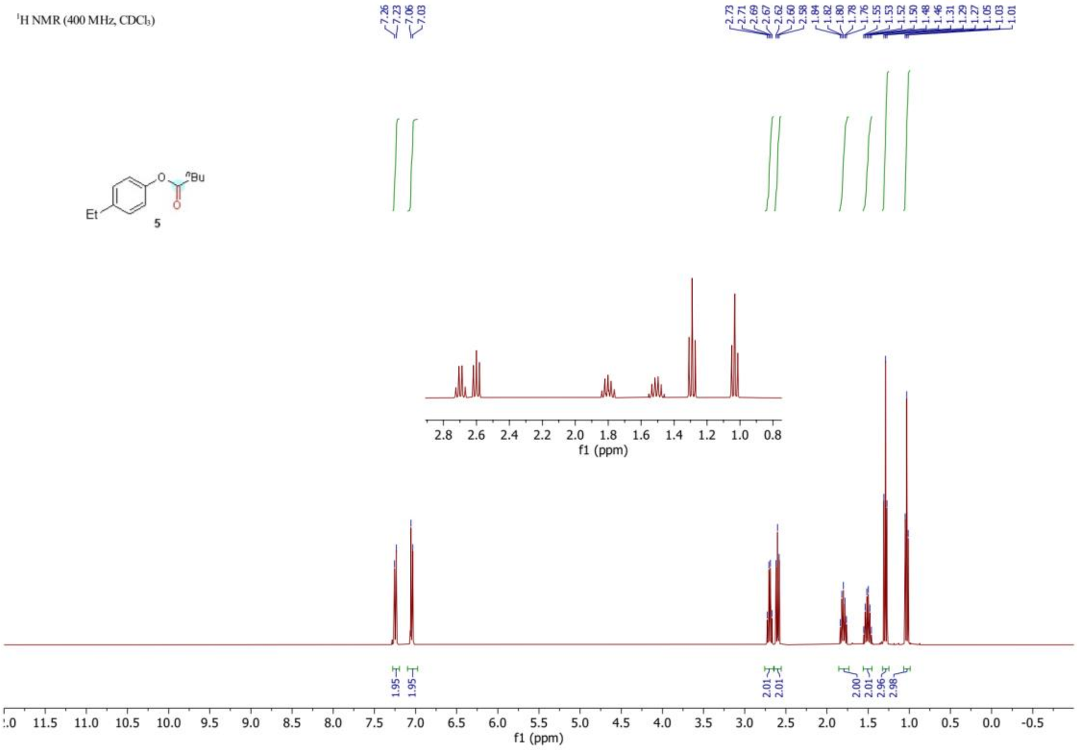


1H NMR (300 MHz, CDCl3)
7.08
7.05
6.87
6.85
2.47
2.44
2.42
2.24
1.69
1.67
1.65
1.62
1.59
1.41
1.39
1.36
1.34
1.31
1.29
0.90
0.88
0.85
nBu
Me
4
2.01
2.00
2.08
3.03
2.07
2.12
3.07
f1 (ppm)



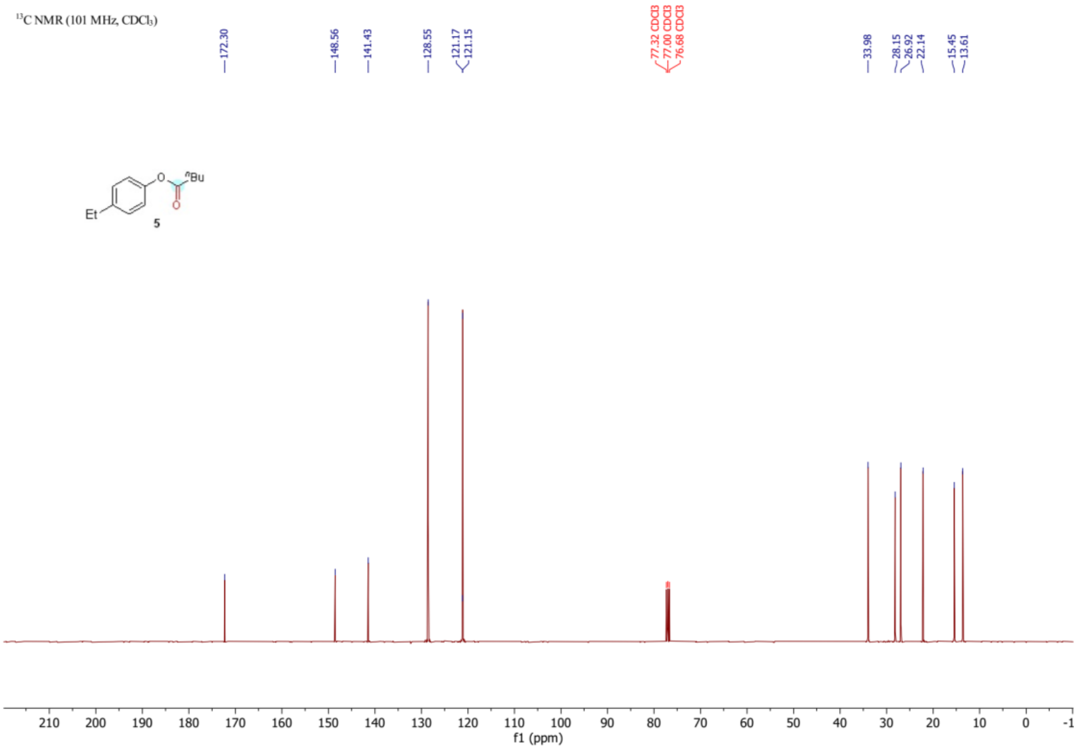
13C NMR (75 MHz, CDCl3)
172.35
148.45
135.16
129.78
121.15
77.42 CDCl3
77.00 CDCl3
76.58 CDCl3
34.01
26.95
22.17
20.74
13.64
nBu
Me
4
f1 (ppm)

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

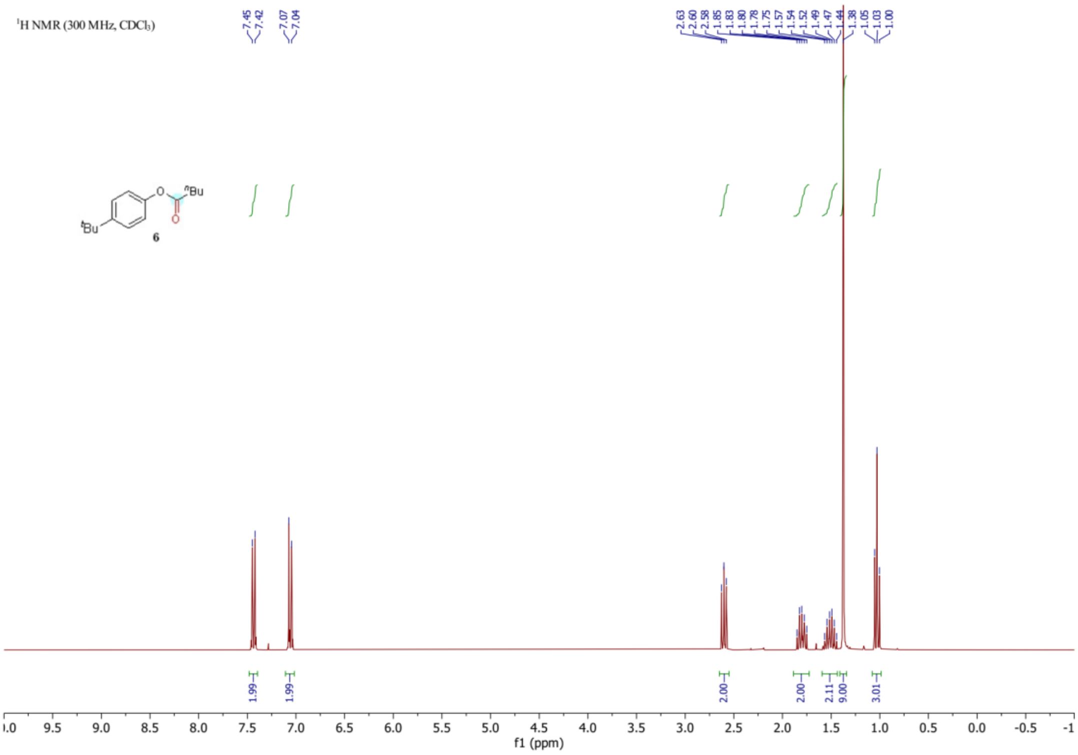


5

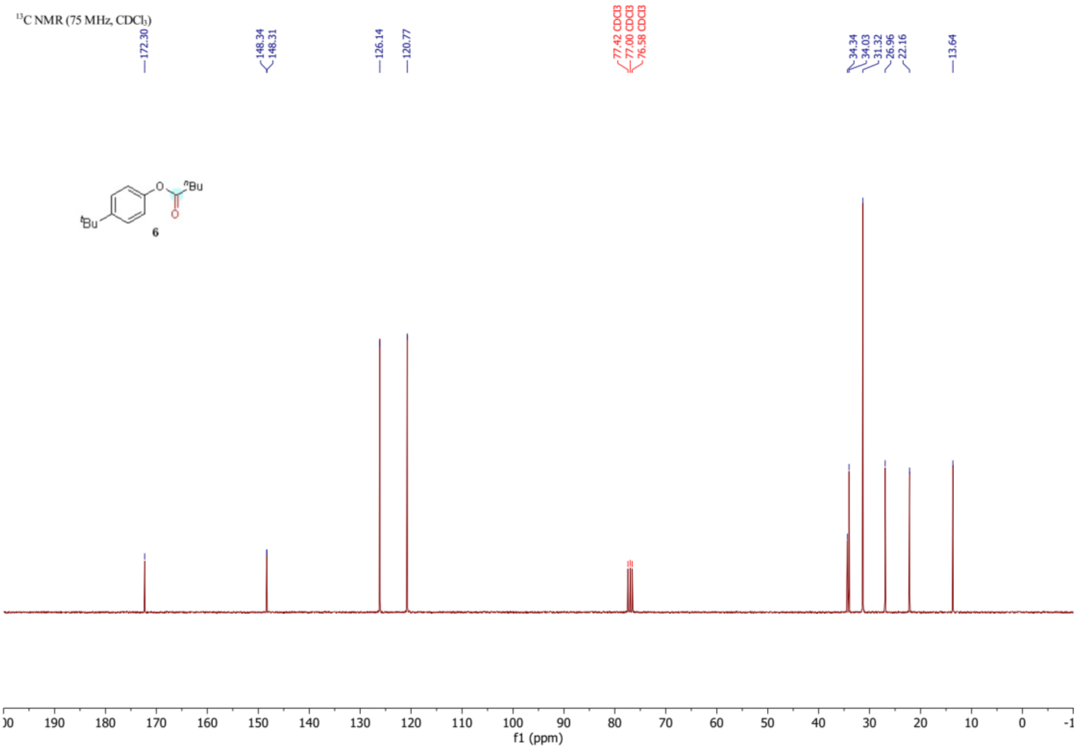
^{13}C NMR (101 MHz, $CDCl_3$)



5

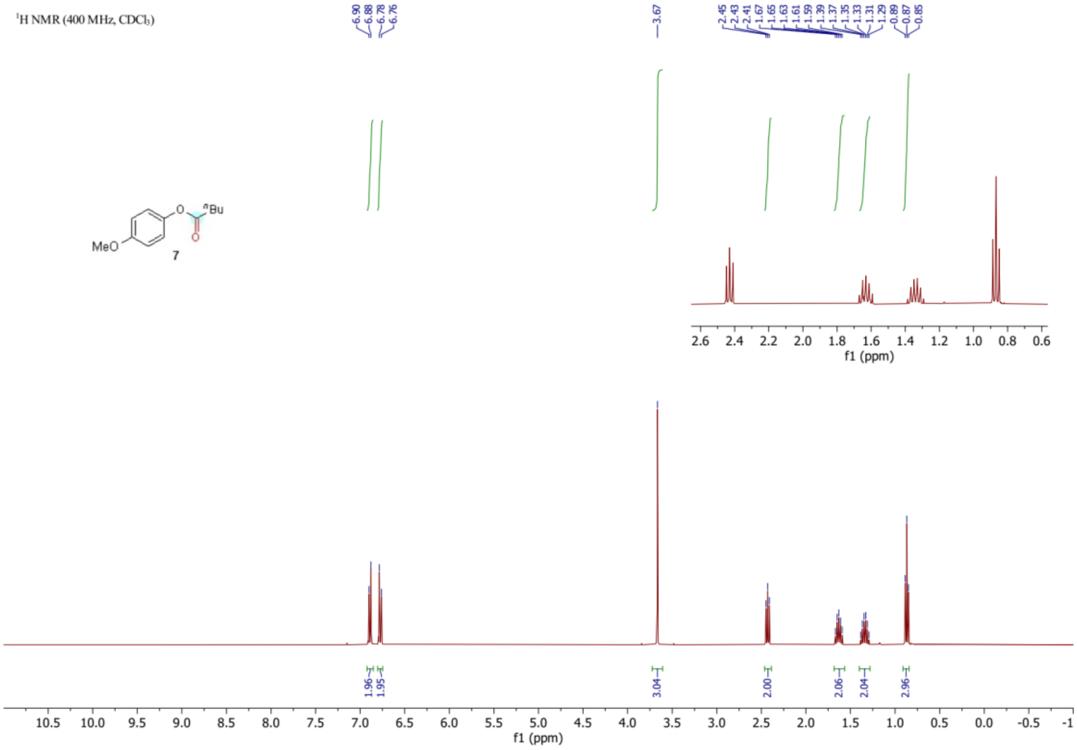


^{1}H NMR (300 MHz, CDCl$_3$)
7.45
7.42
7.07
7.04
2.63
2.60
2.58
1.85
1.83
1.80
1.78
1.75
1.57
1.54
1.52
1.49
1.47
1.44
1.38
1.05
1.03
1.00
^{t}Bu
^{n}Bu
6
1.99
1.99
2.00
2.00
2.11
9.00
3.01
f1 (ppm)

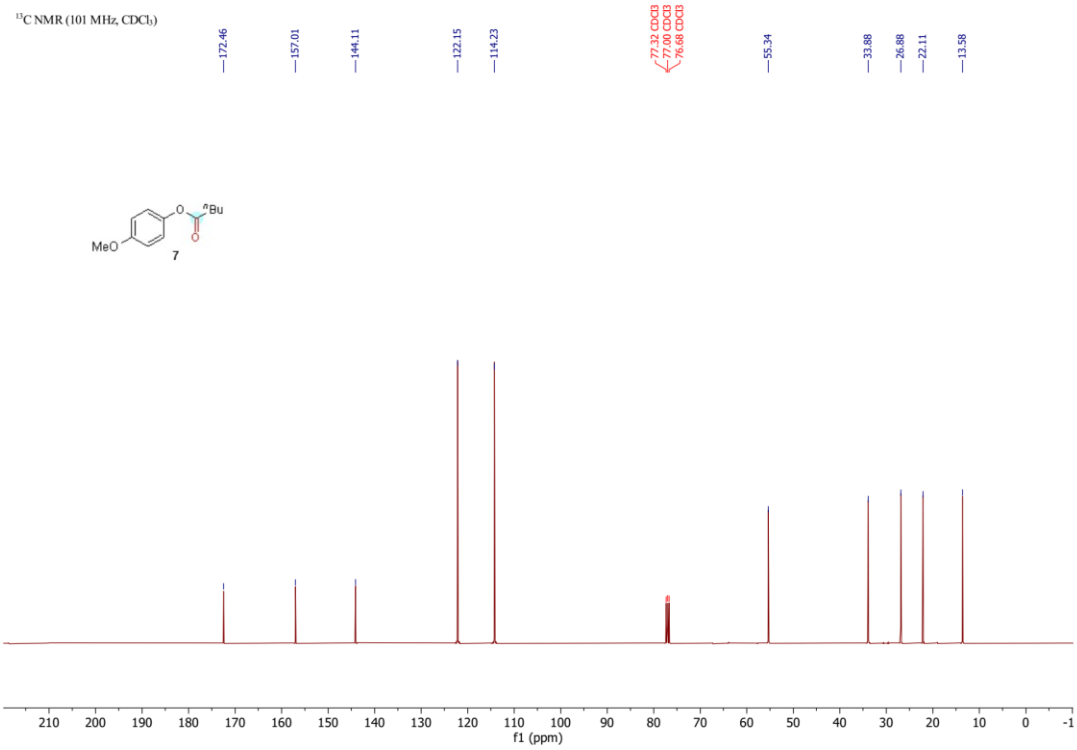


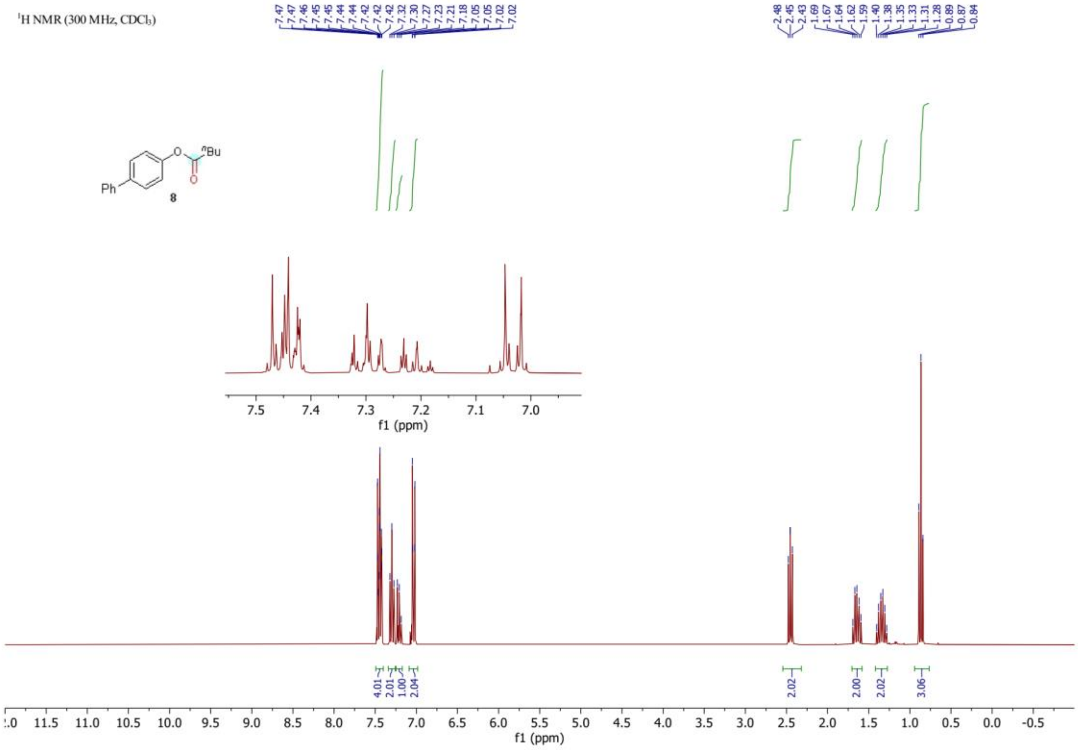
^{13}C NMR (75 MHz, CDCl$_3$)
172.30
148.34
148.31
126.14
120.77
77.42 CDCl3
77.00 CDCl3
76.58 CDCl3
34.34
34.03
31.32
26.96
22.16
13.64
^{t}Bu
^{n}Bu
6
f1 (ppm)

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

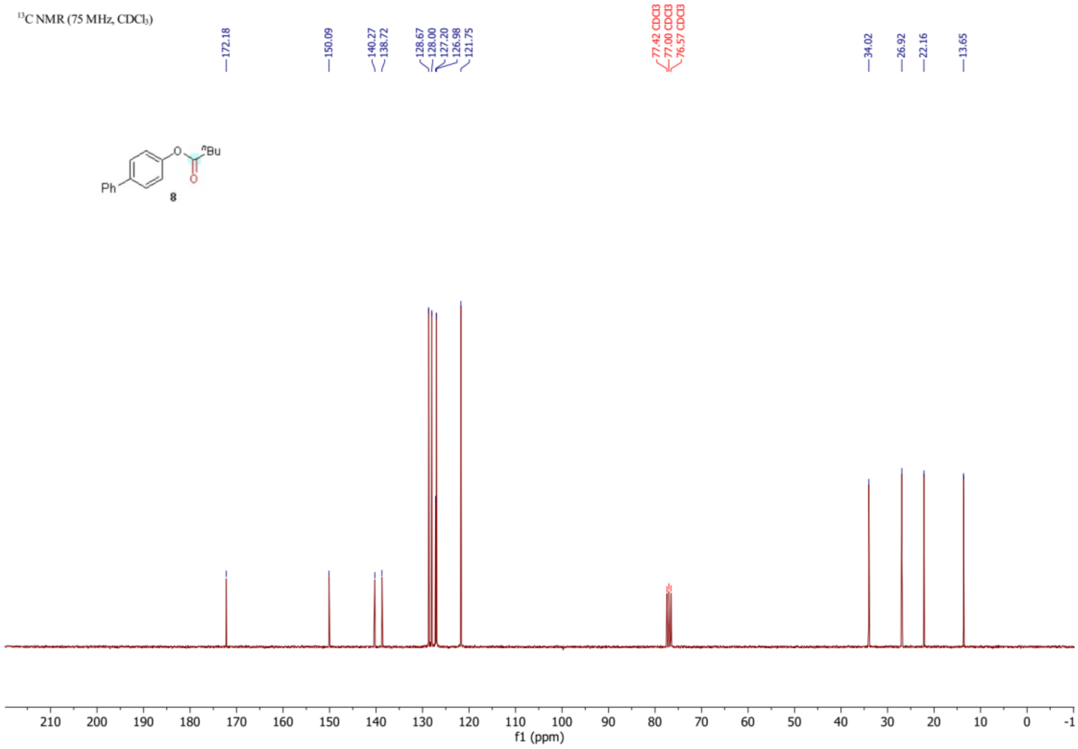


^{13}C NMR (101 MHz, $CDCl_3$)



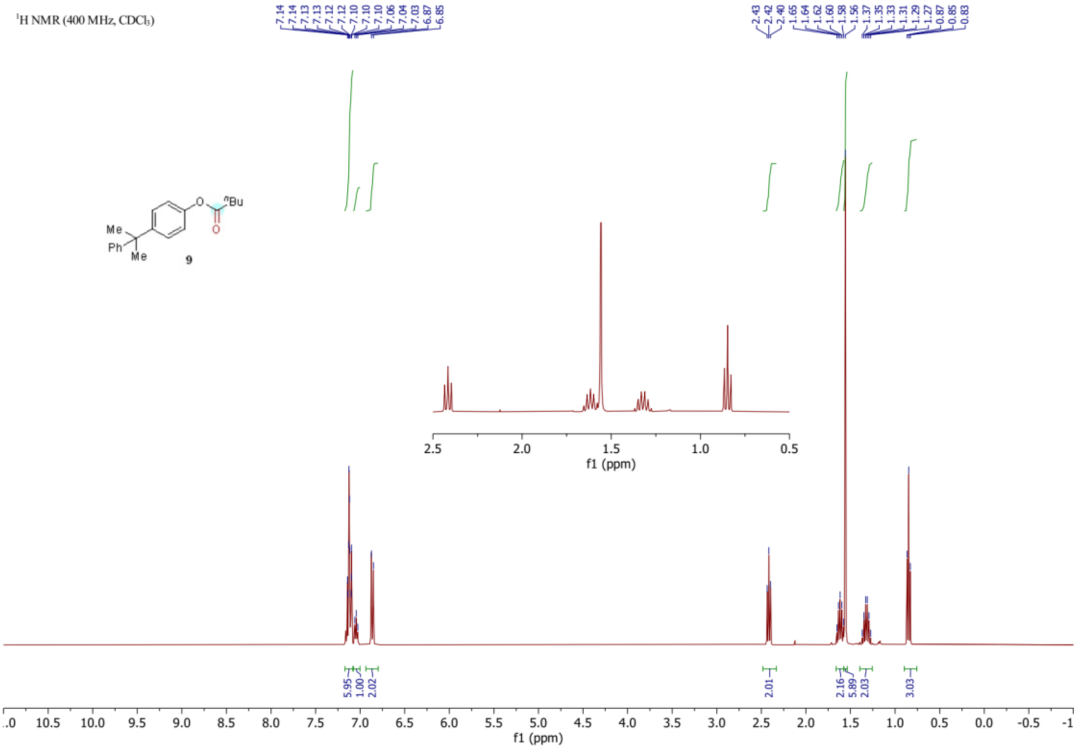


^{1}H NMR (300 MHz, CDCl$_3$)
7.47 7.47 7.46 7.45 7.45 7.44 7.44 7.42 7.42 7.32 7.30 7.27 7.23 7.21 7.18 7.05 7.05 7.02 7.02
2.48 2.45 2.43 1.69 1.67 1.64 1.62 1.59 1.40 1.38 1.35 1.33 1.31 1.28 0.89 0.87 0.84
O
^{n}Bu
O
Ph
8
7.5 7.4 7.3 7.2 7.1 7.0
f1 (ppm)
4.01 2.01 1.00 2.04 2.02 2.00 2.02 3.06
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

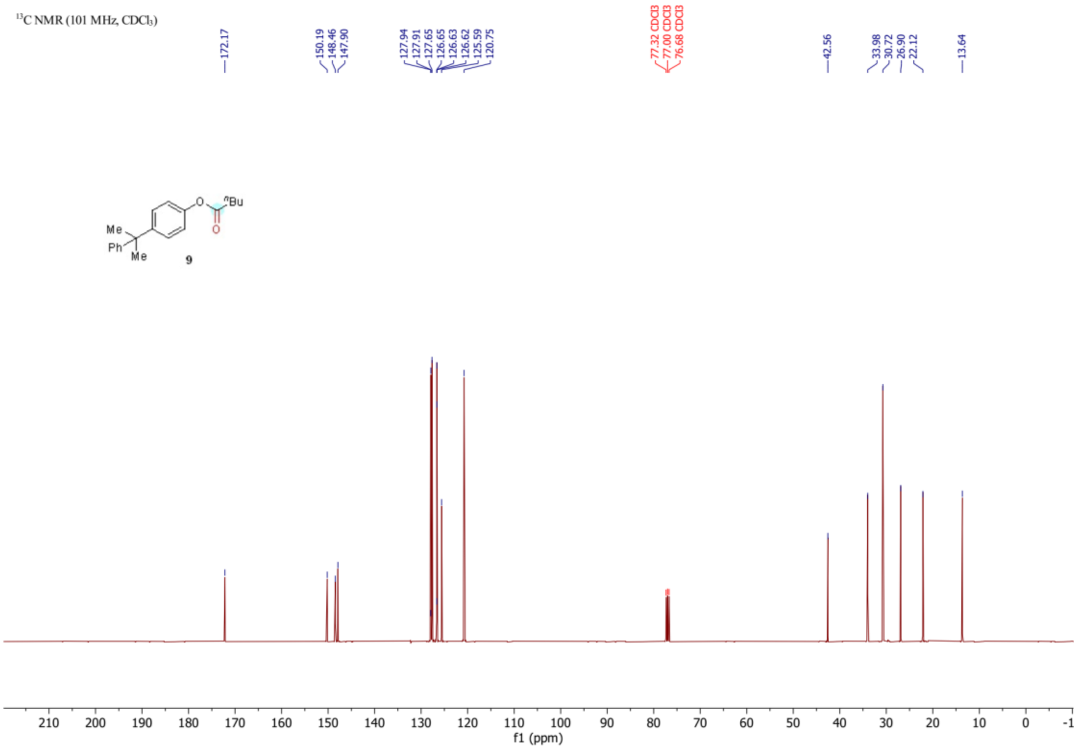


^{13}C NMR (75 MHz, CDCl$_3$)
172.18
150.09
140.27 138.72
128.67 128.00 127.20 126.98 121.75
77.42 CDCl3 77.00 CDCl3 76.57 CDCl3
34.02
26.92
22.16
13.65
O
^{n}Bu
O
Ph
8
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -1
f1 (ppm)

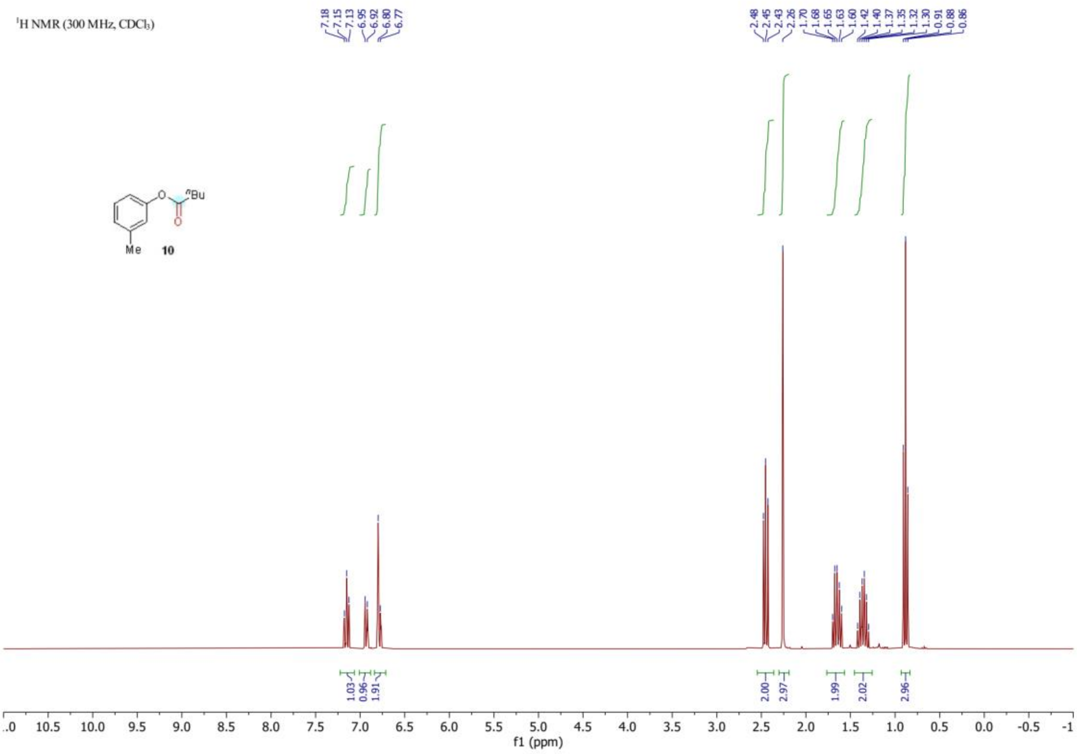
^{1}H NMR (400 MHz, $CDCl_3$)



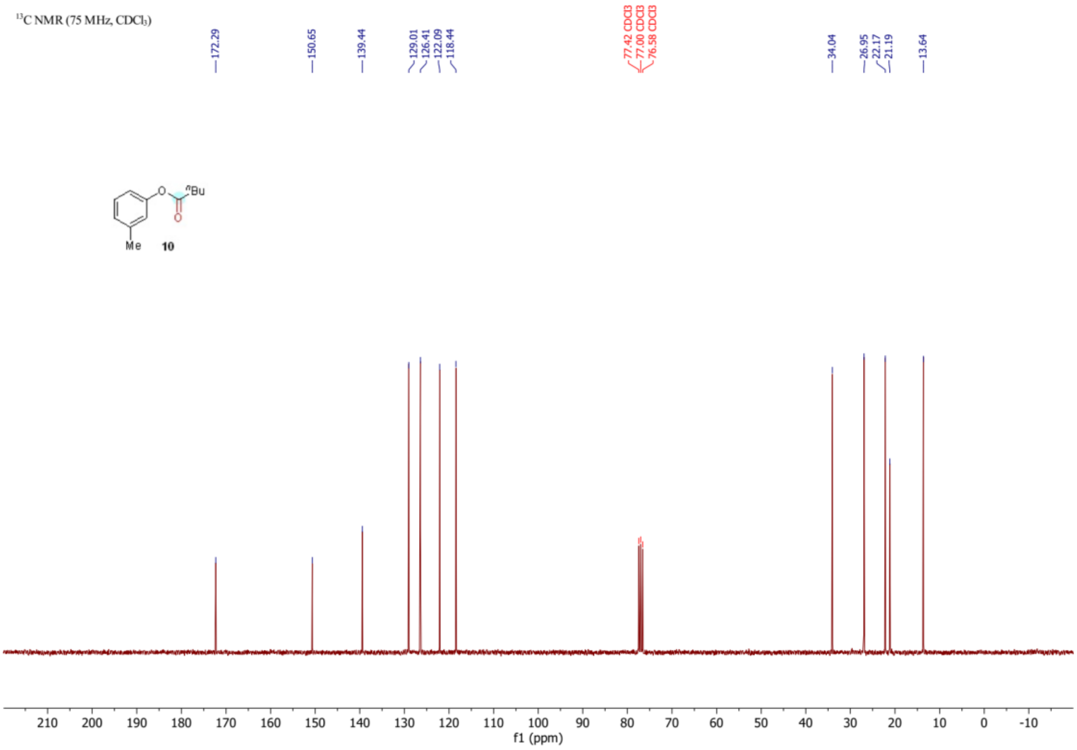
^{13}C NMR (101 MHz, $CDCl_3$)

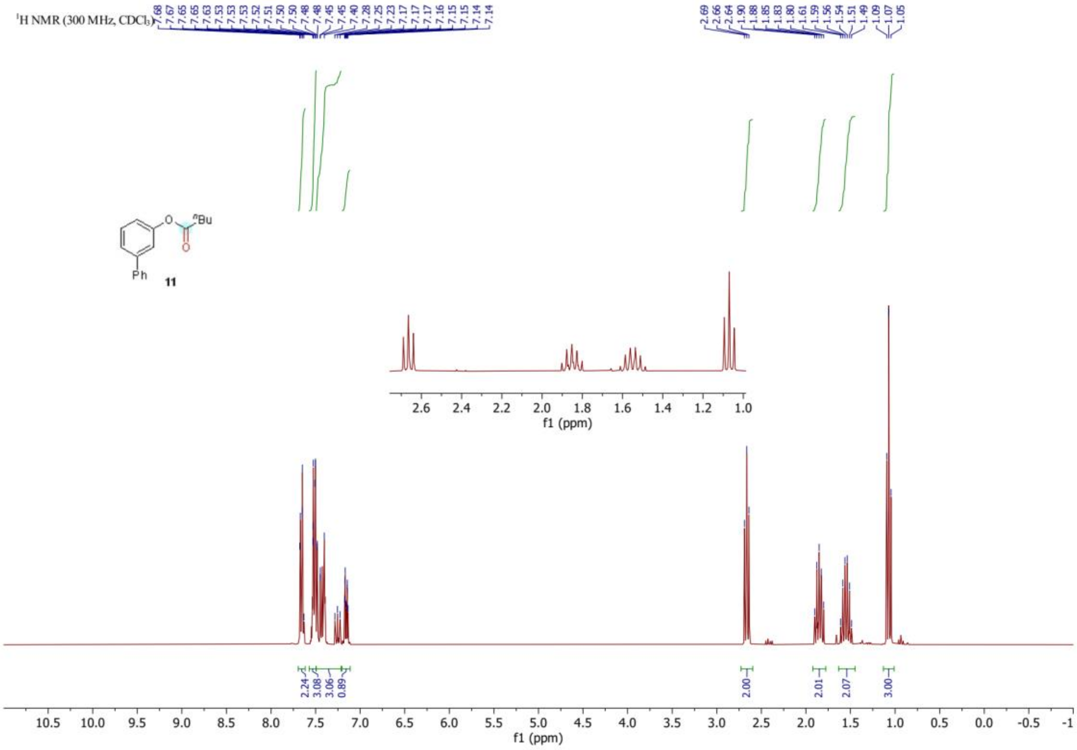


^{1}H NMR (300 MHz, CDCl$_3$)

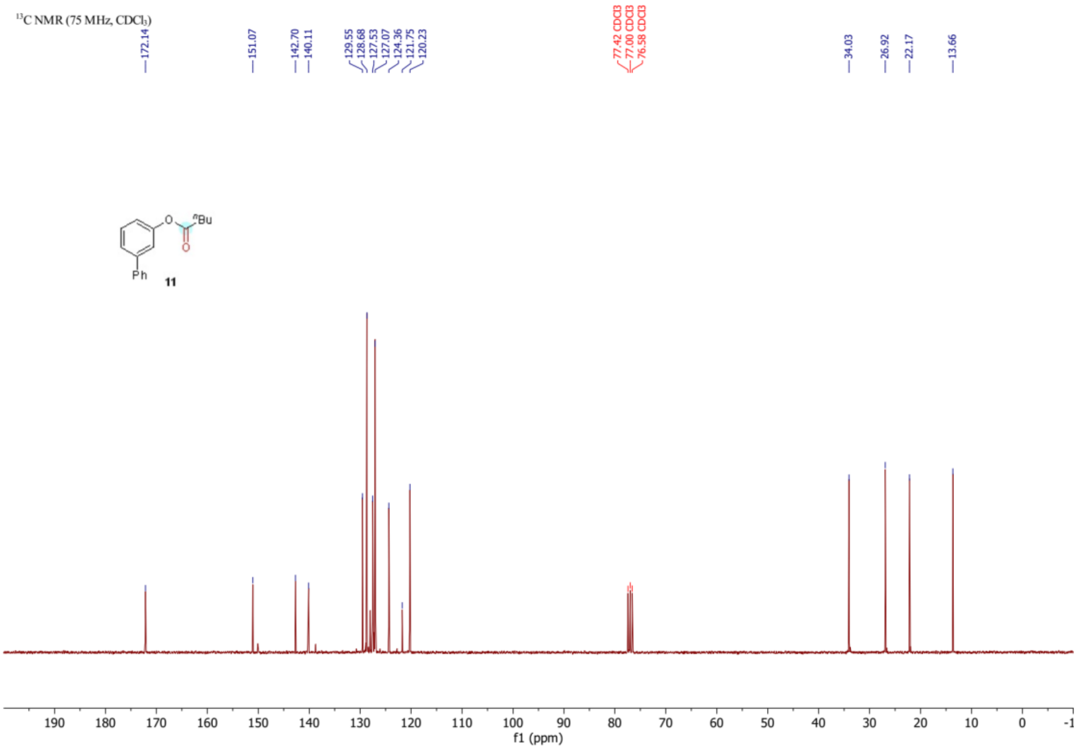


^{13}C NMR (75 MHz, CDCl$_3$)

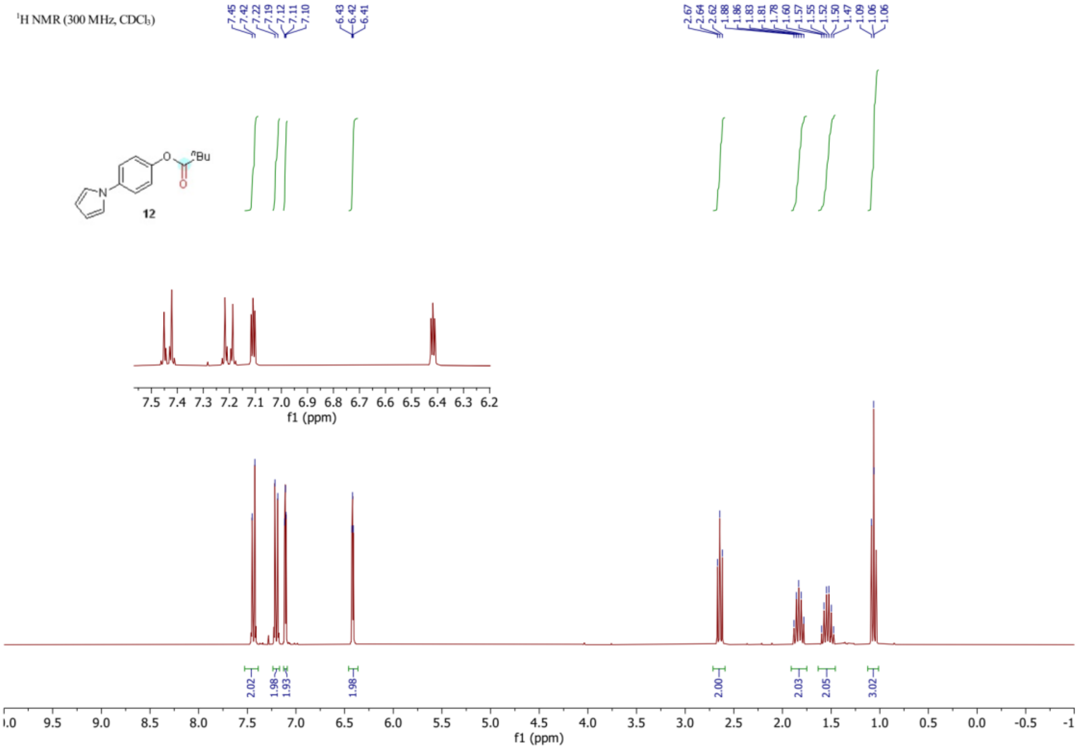




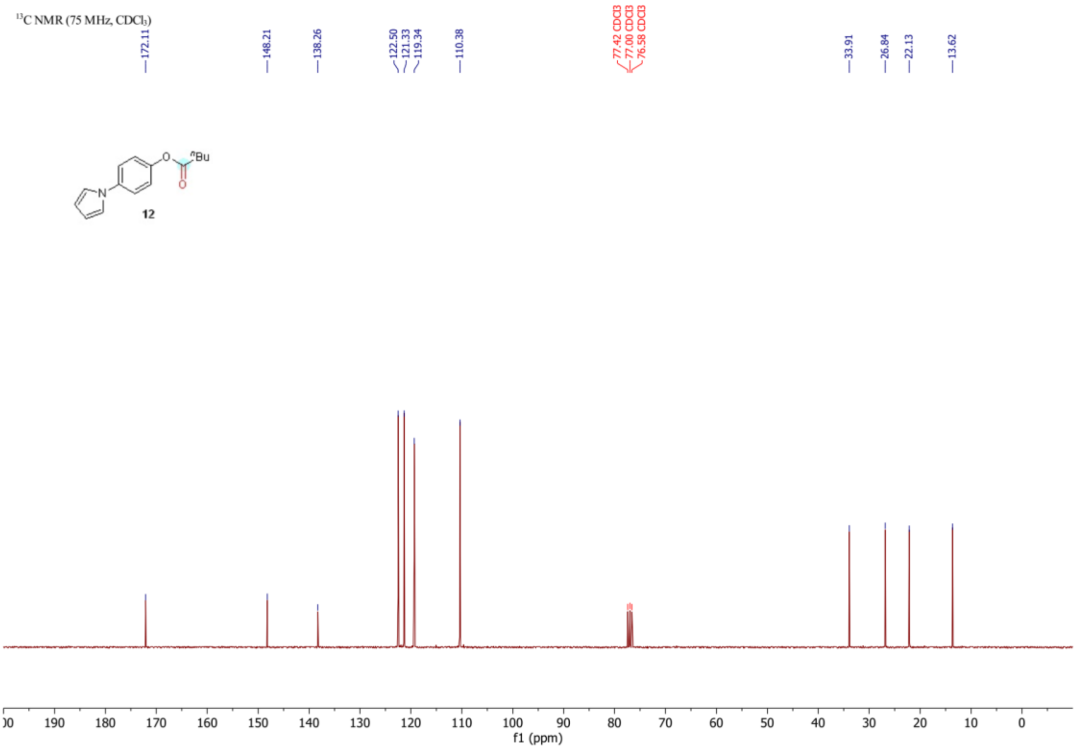
¹H NMR (300 MHz, CDCl₃)
ⁿBu
Ph
11
f1 (ppm)



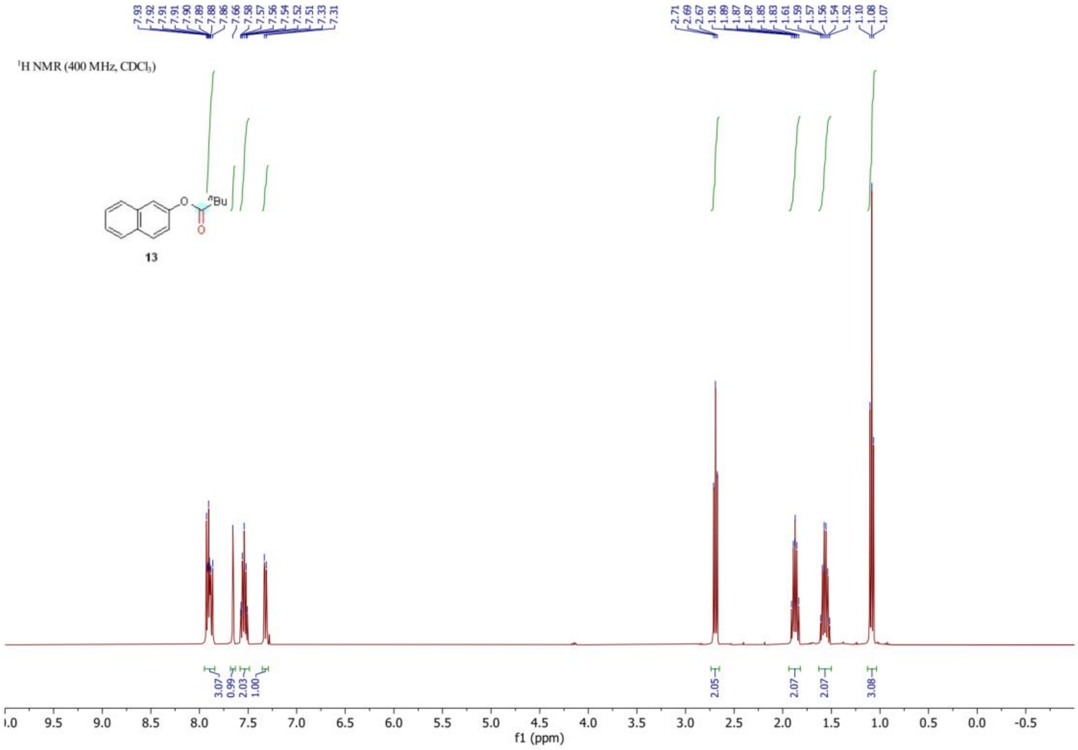
¹³C NMR (75 MHz, CDCl₃)
172.14
151.07
142.70
140.11
129.55
128.68
127.53
127.07
124.36
121.75
120.23
77.42 CDCl3
77.00 CDCl3
76.58 CDCl3
34.03
26.92
22.17
13.66
ⁿBu
Ph
11
f1 (ppm)



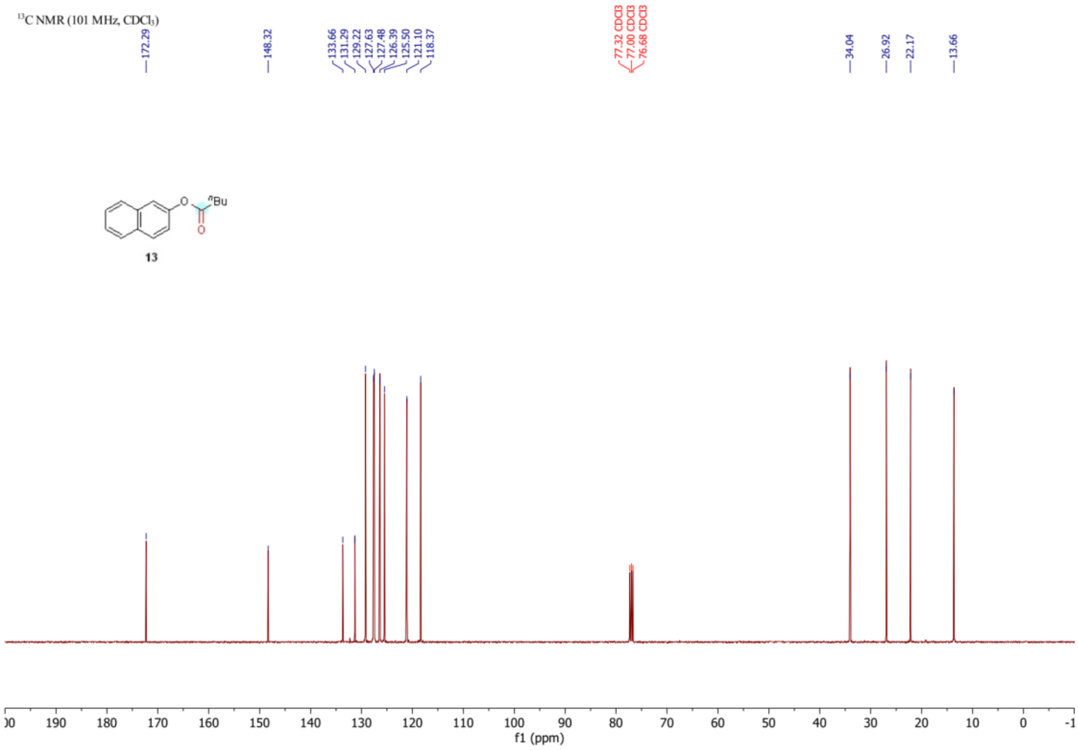
^1H NMR (300 MHz, $CDCl_3$)
12



^{13}C NMR (75 MHz, $CDCl_3$)
12

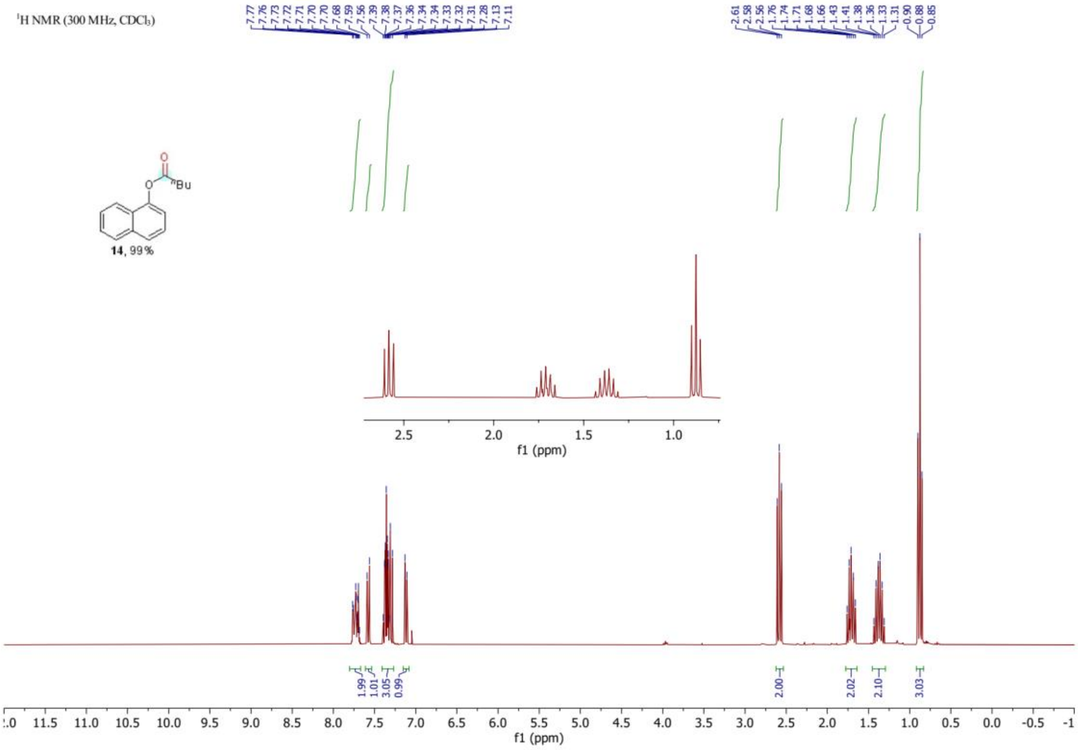


1H NMR (400 MHz, CDCl3)
7.93
7.92
7.91
7.91
7.90
7.89
7.88
7.86
7.66
7.58
7.57
7.56
7.54
7.52
7.51
7.33
7.31
2.71
2.69
2.67
1.91
1.89
1.87
1.87
1.85
1.83
1.61
1.59
1.57
1.56
1.54
1.52
1.10
1.08
1.07
nBu
O
13
3.07
0.99
2.03
1.00
2.05
2.07
2.07
3.08
f1 (ppm)

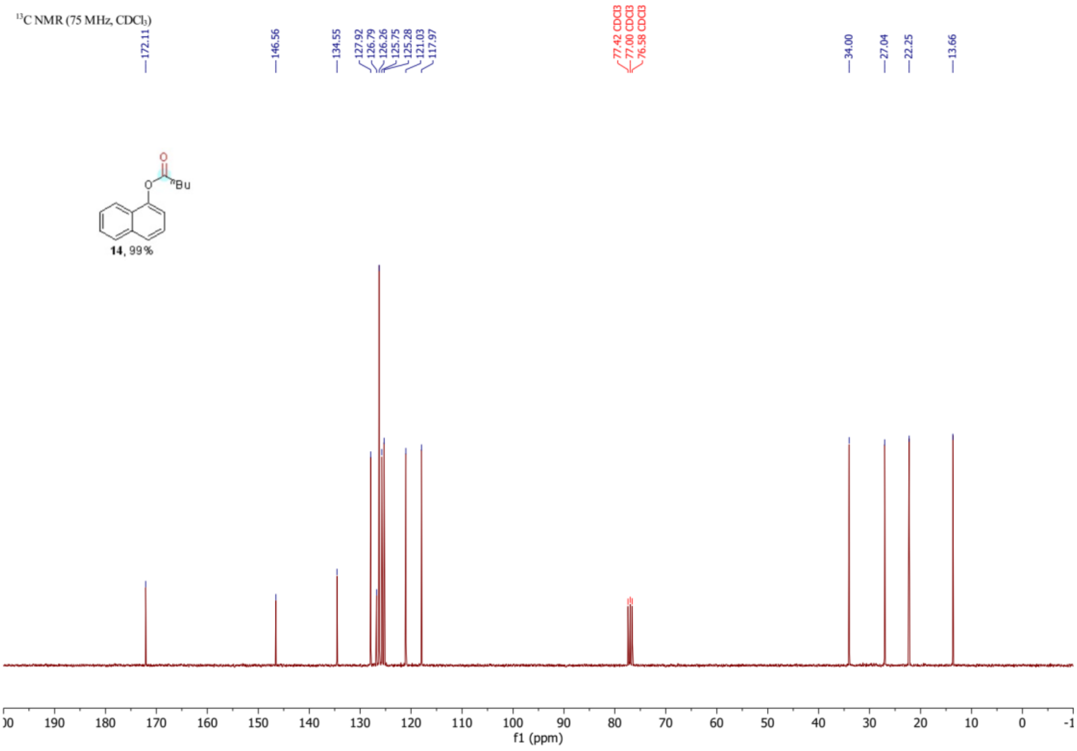


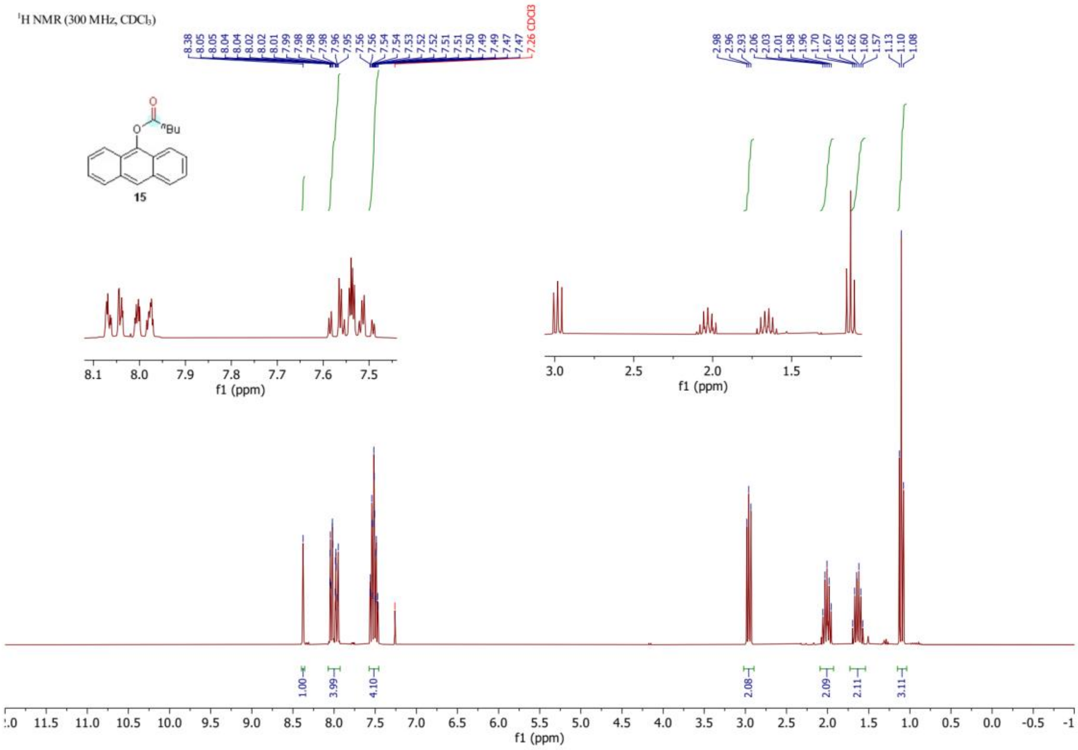
13C NMR (101 MHz, CDCl3)
172.29
148.32
133.66
131.29
129.22
127.63
127.48
126.39
125.50
121.10
118.37
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
34.04
26.92
22.17
13.66
nBu
O
13
f1 (ppm)

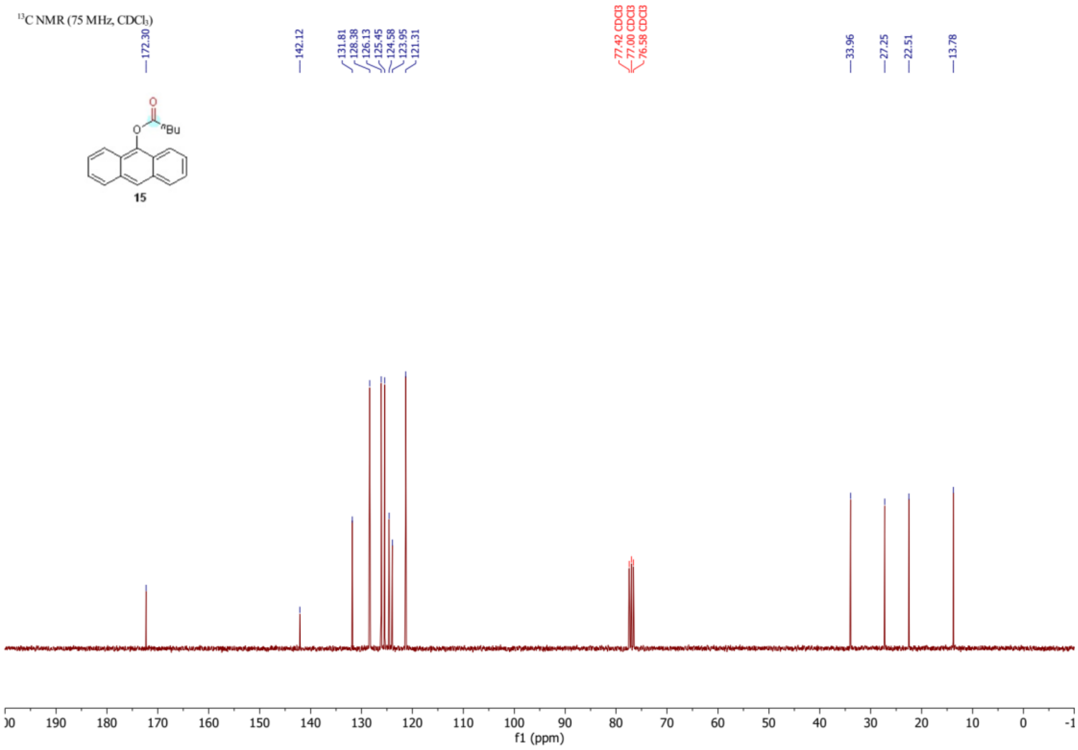
^{1}H NMR (300 MHz, $CDCl_3$)

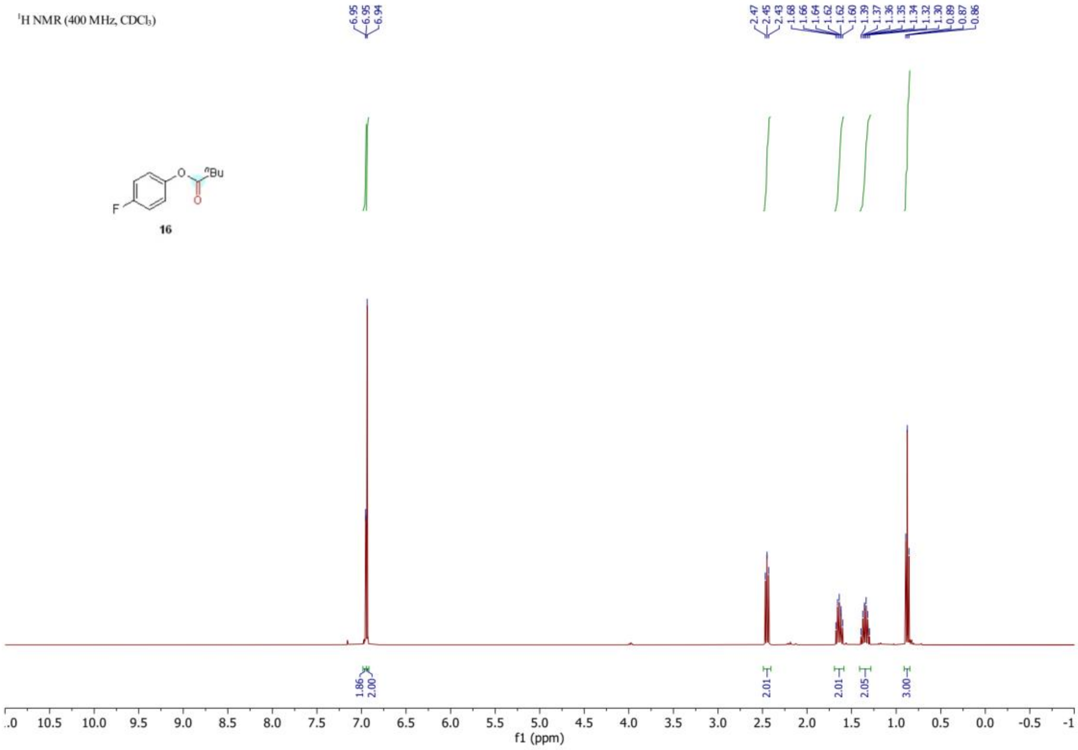


^{13}C NMR (75 MHz, $CDCl_3$)

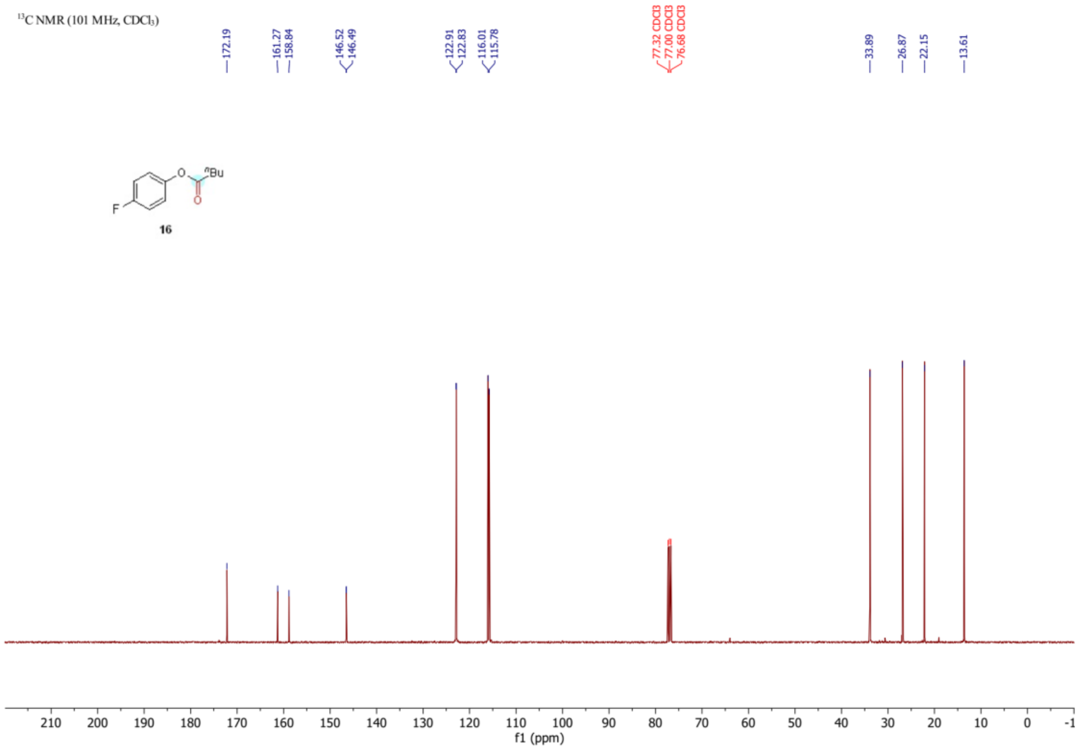



1H NMR (300 MHz, CDCl3)
15
f1 (ppm)


13C NMR (75 MHz, CDCl3)
15
f1 (ppm)

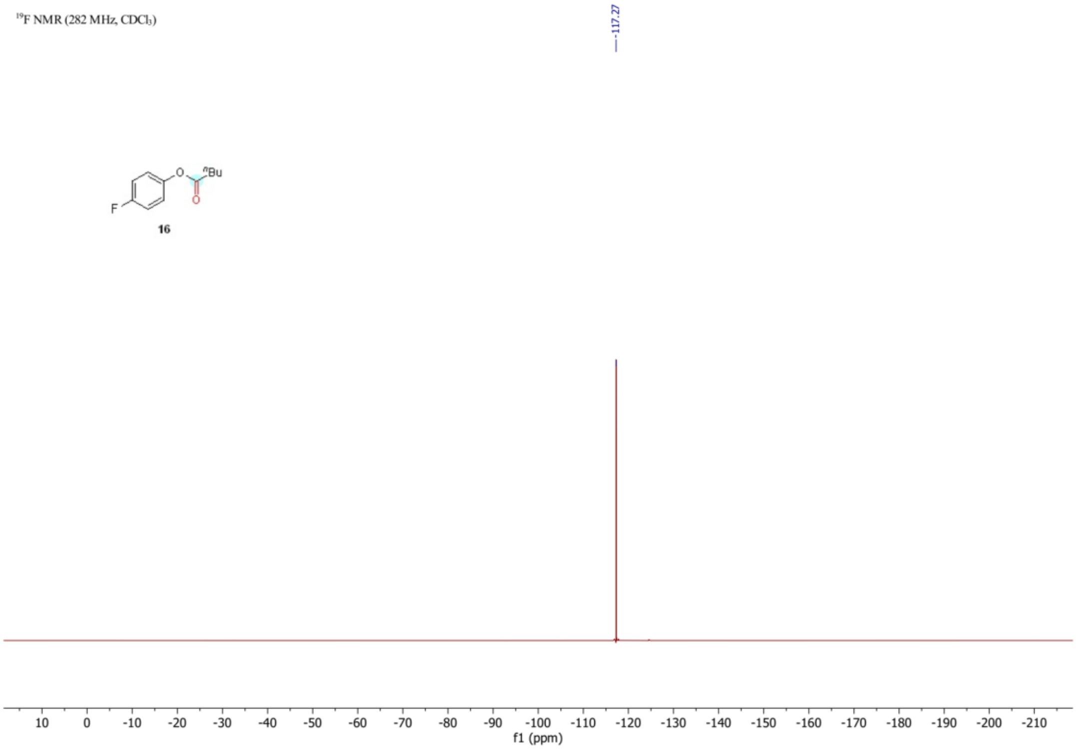


1H NMR (400 MHz, CDCl3)
nBu
F
O
16
1.86
2.00
2.01
2.01
2.05
3.00
f1 (ppm)

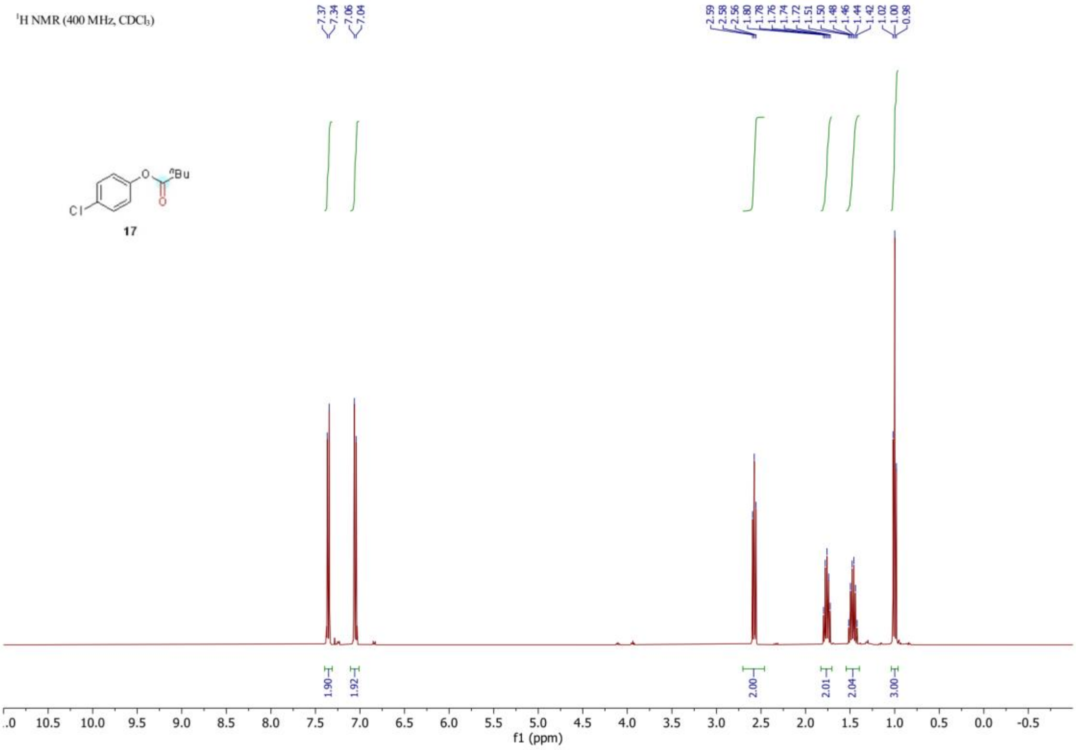


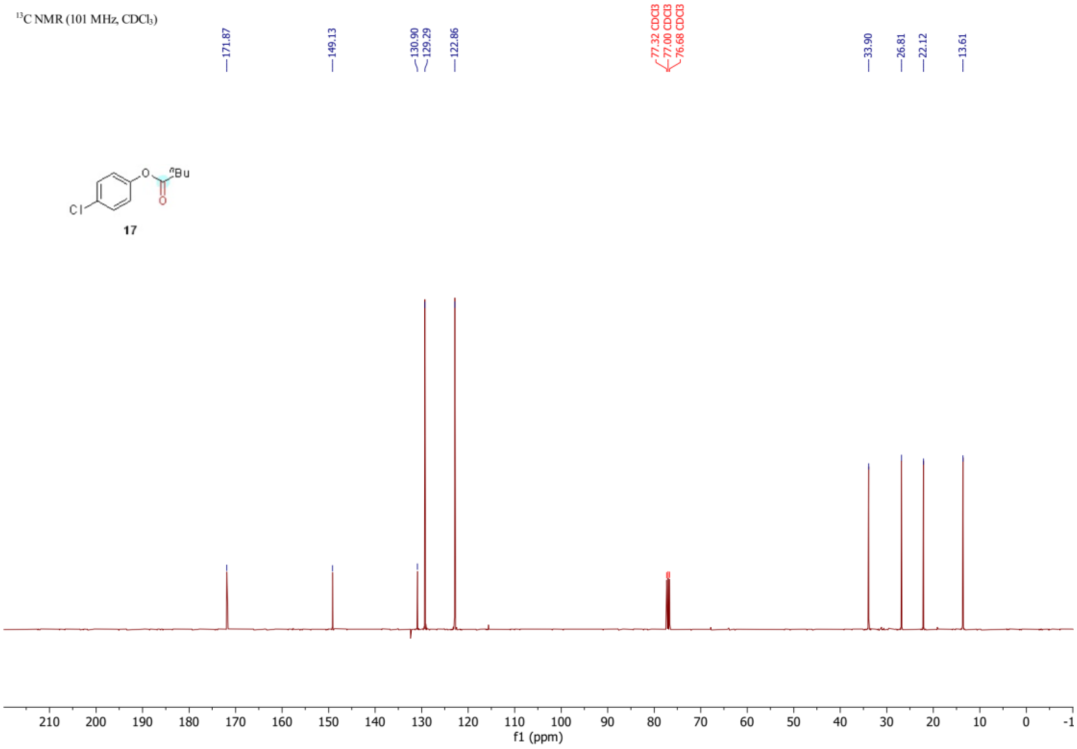
13C NMR (101 MHz, CDCl3)
172.19
161.27
158.84
146.52
146.49
122.91
122.83
116.01
115.78
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
33.89
26.87
22.15
13.61
nBu
F
O
16
f1 (ppm)

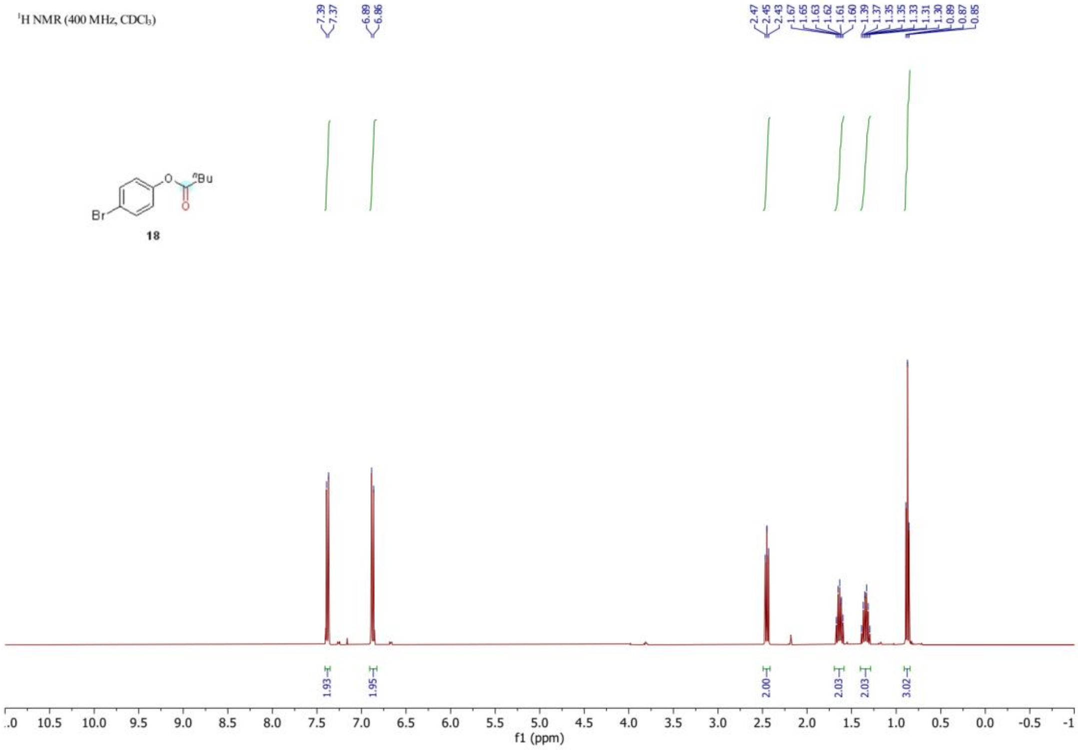
^{19}F NMR (282 MHz, $CDCl_3$)



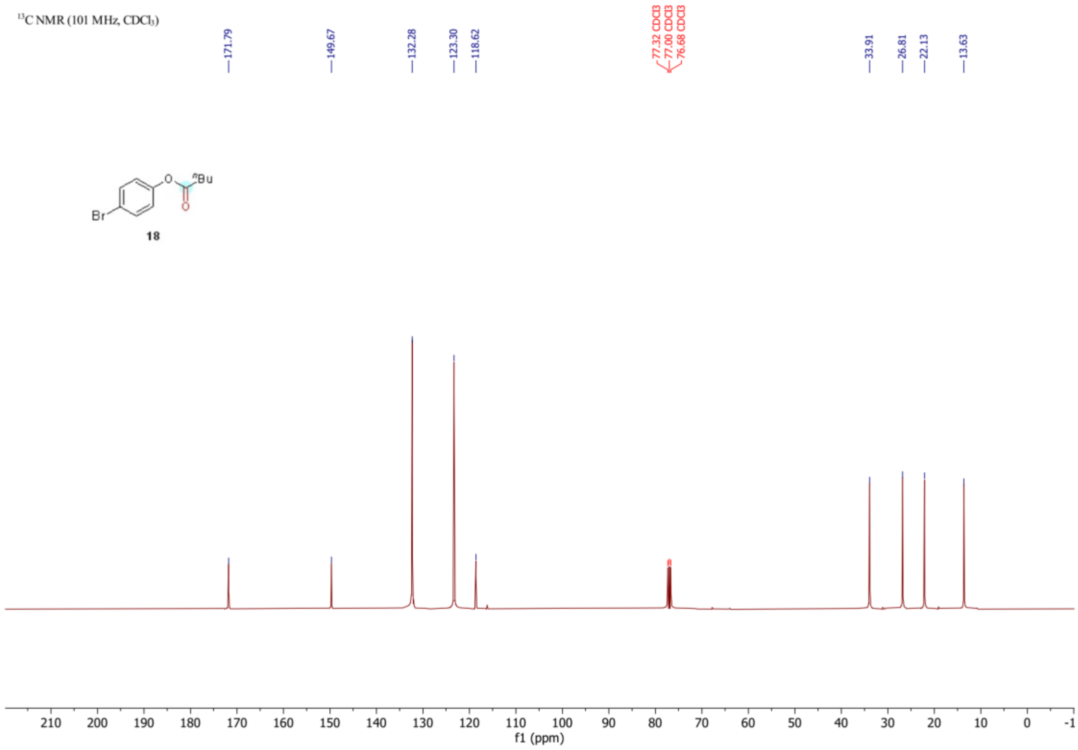
16


1H NMR (400 MHz, CDCl3)
Cl
O
nBu
O
17
f1 (ppm)

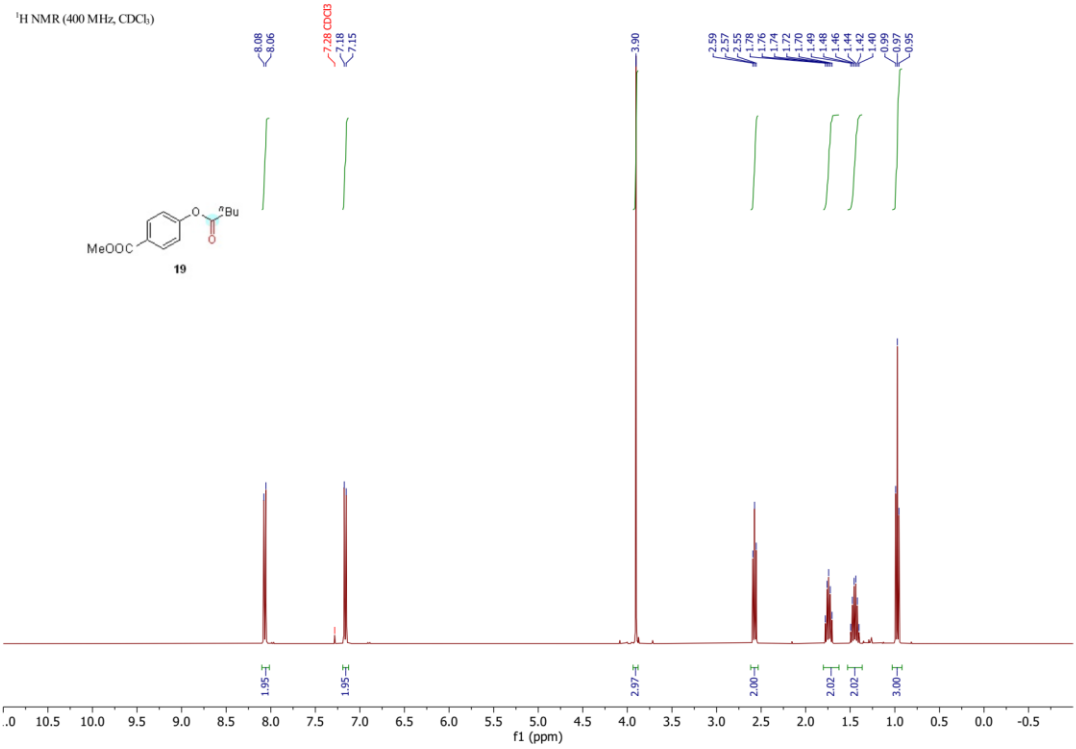

13C NMR (101 MHz, CDCl3)
171.87
149.13
130.90
129.29
122.86
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
33.90
26.81
22.12
13.61
Cl
O
nBu
O
17
f1 (ppm)



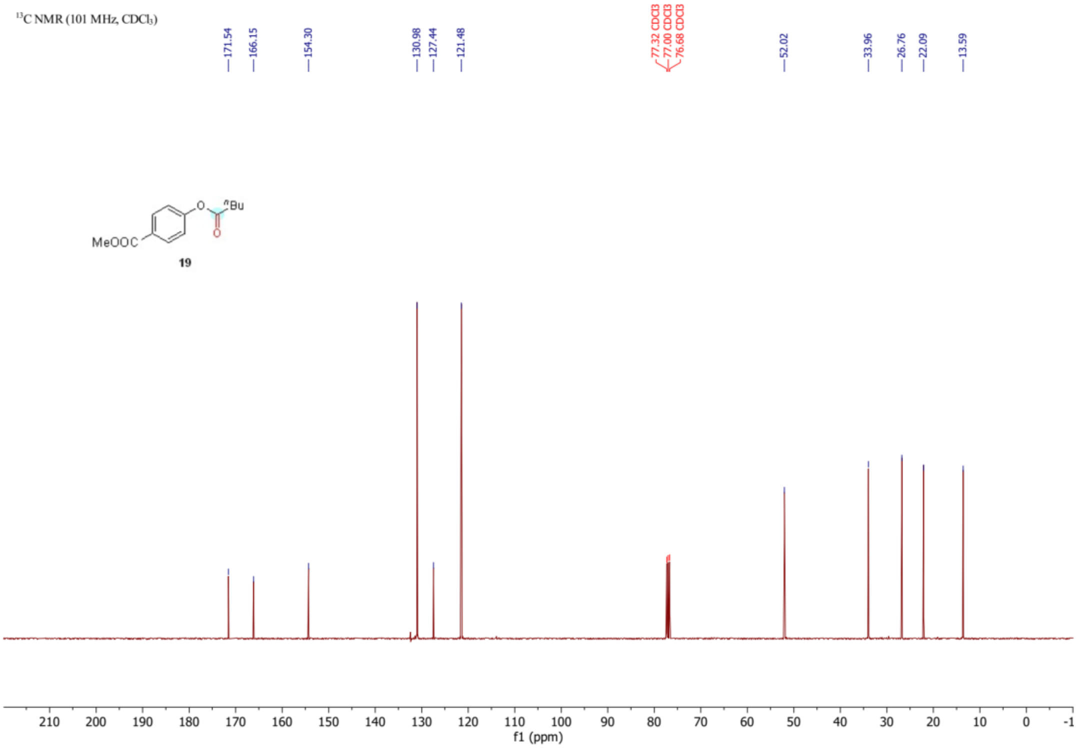
1H NMR (400 MHz, CDCl3)
7.39
7.37
6.89
6.86
2.47
2.45
2.43
1.67
1.65
1.63
1.62
1.61
1.60
1.39
1.37
1.35
1.35
1.33
1.31
1.30
0.89
0.87
0.85
Br
O
O
nBu
18
1.93
1.95
2.00
2.03
2.03
3.02
f1 (ppm)



13C NMR (101 MHz, CDCl3)
171.79
149.67
132.28
123.30
118.62
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
33.91
26.81
22.13
13.63
Br
O
O
nBu
18
f1 (ppm)

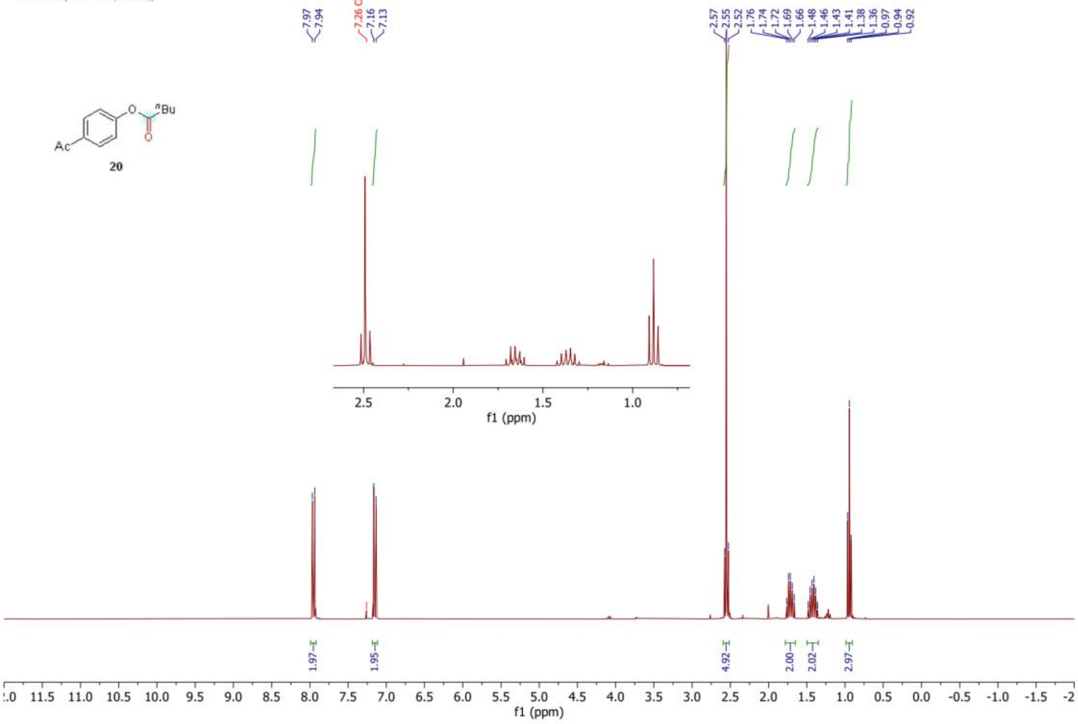


^{1}H NMR (400 MHz, $CDCl_3$)
8.08
8.06
7.28 CDCl3
7.18
7.15
3.90
2.59
2.57
2.55
1.78
1.76
1.74
1.72
1.70
1.49
1.48
1.46
1.44
1.42
1.40
0.99
0.97
0.95
MeOOC
O
nBu
O
19
1.95
1.95
2.97
2.00
2.02
2.02
3.00
f1 (ppm)



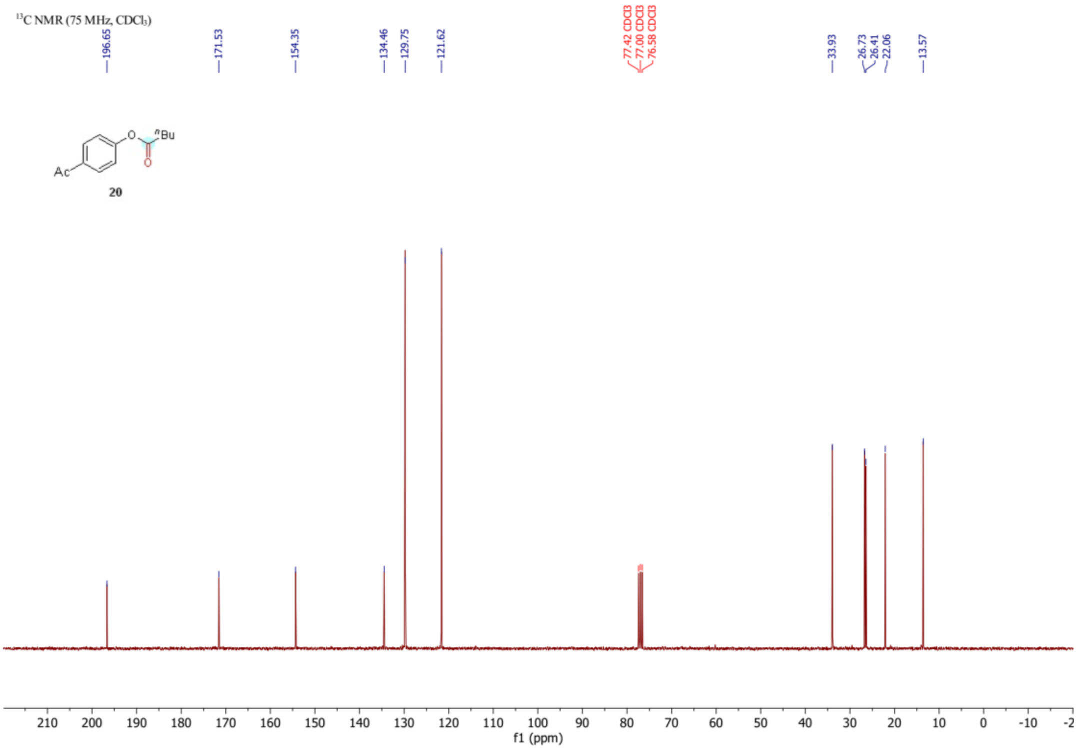
^{13}C NMR (101 MHz, $CDCl_3$)
171.54
166.15
154.30
130.98
127.44
121.48
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
52.02
33.96
26.76
22.09
13.59
MeOOC
O
nBu
O
19
f1 (ppm)

^{1}H NMR (300 MHz, $CDCl_3$)

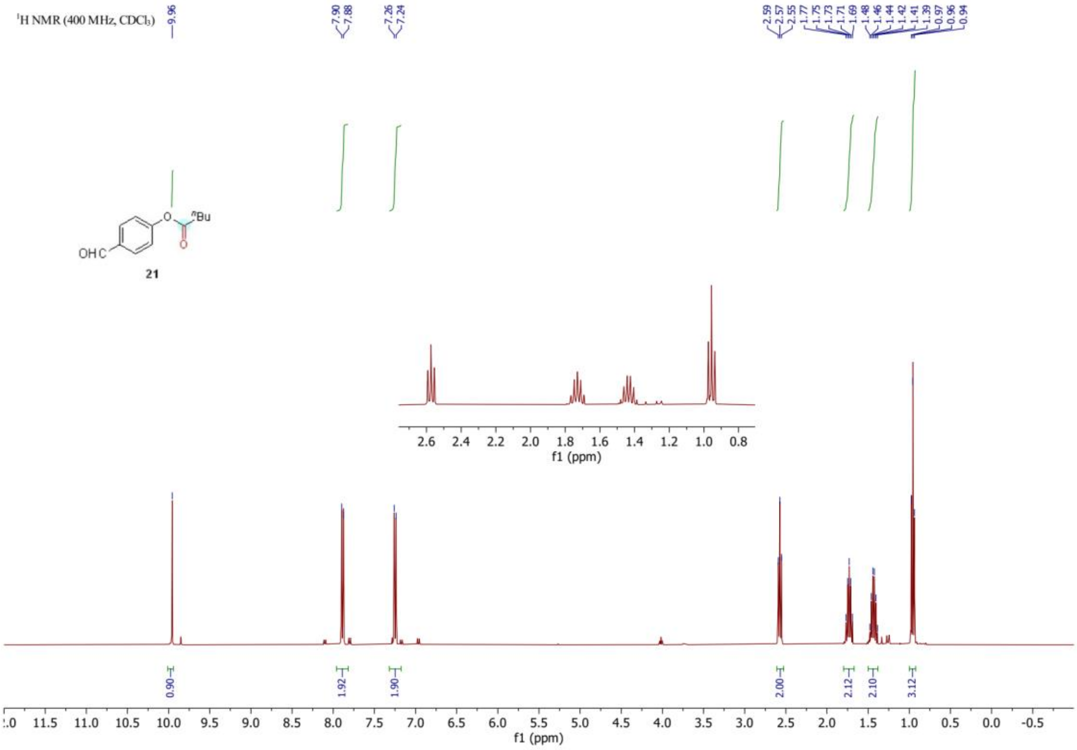


20

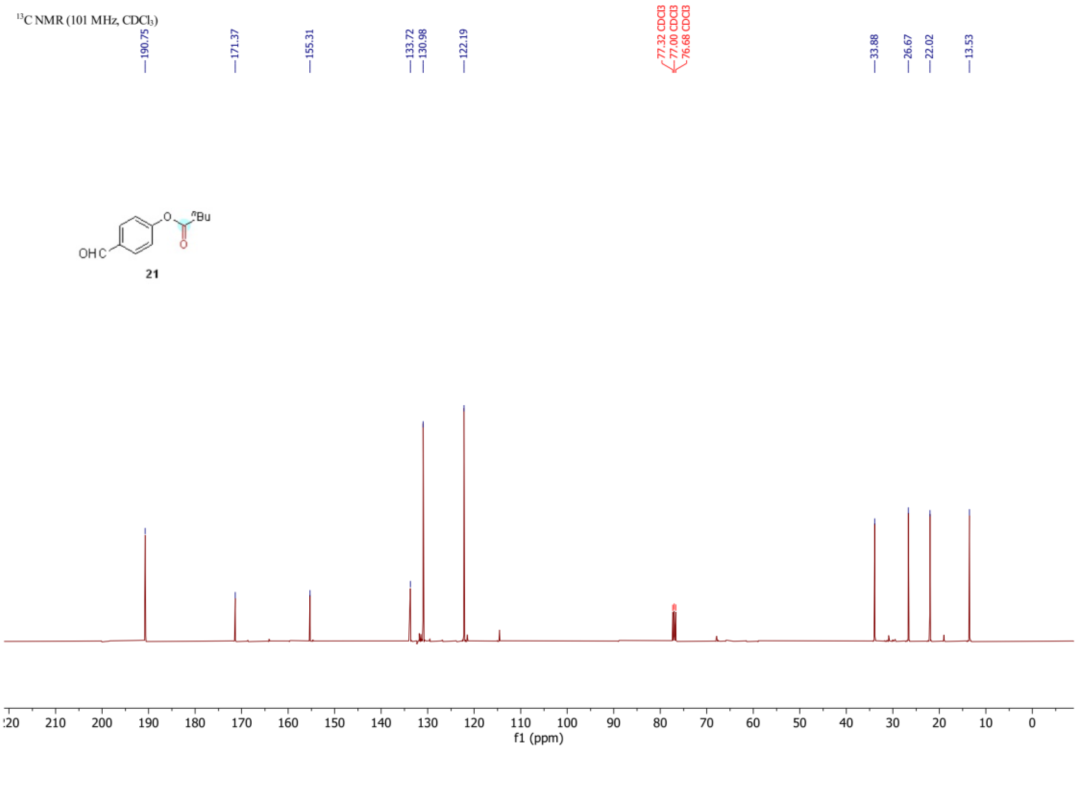
^{13}C NMR (75 MHz, $CDCl_3$)



20

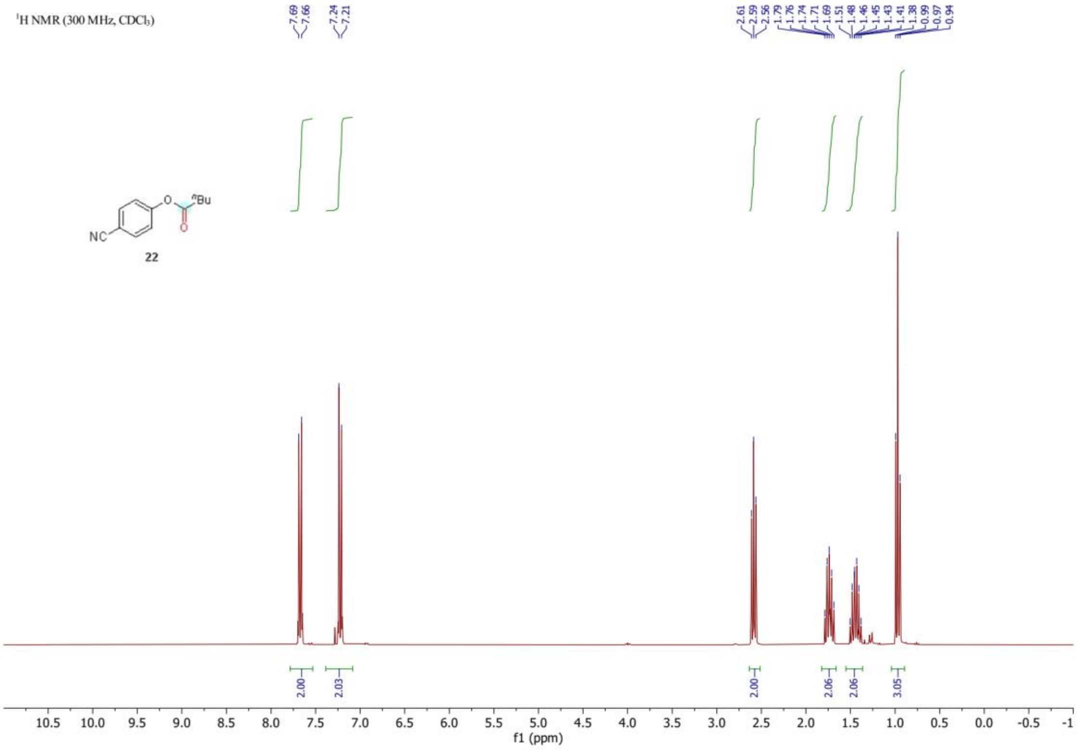


^{1}H NMR (400 MHz, CDCl$_3$)
9.96
7.90
7.88
7.26
7.24
2.59
2.57
2.55
1.77
1.75
1.73
1.71
1.69
1.48
1.46
1.44
1.42
1.41
1.39
0.97
0.96
0.94
OHC
^{n}Bu
21
0.90
1.92
1.90
2.00
2.12
2.10
3.12
f1 (ppm)

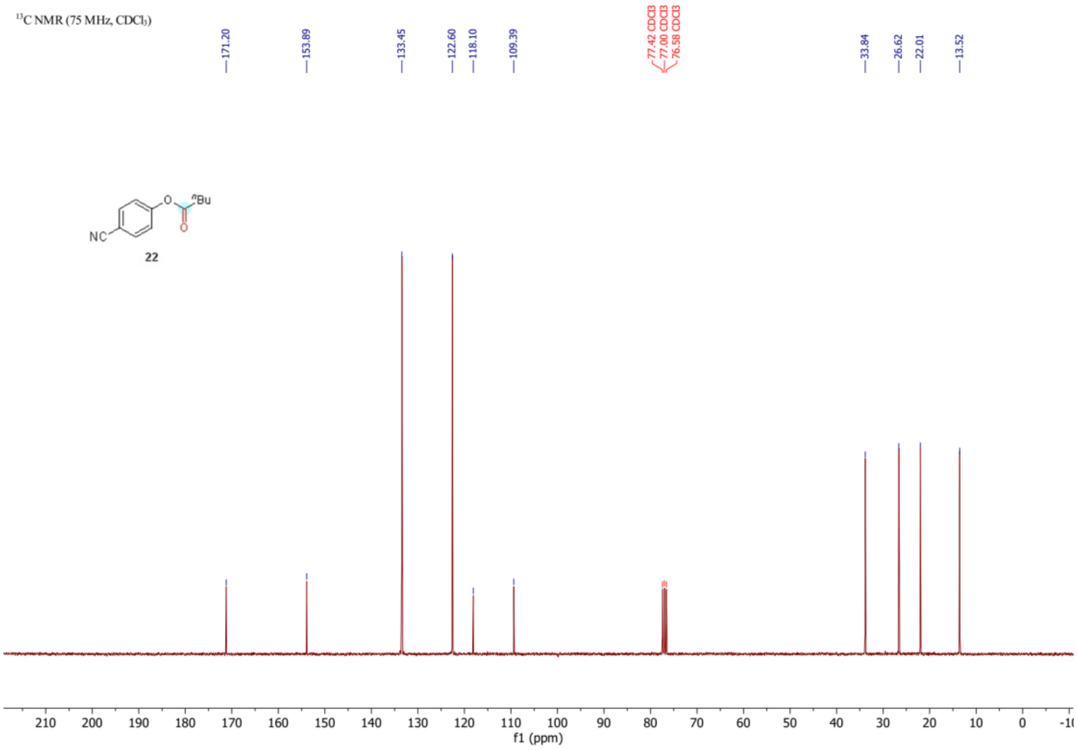


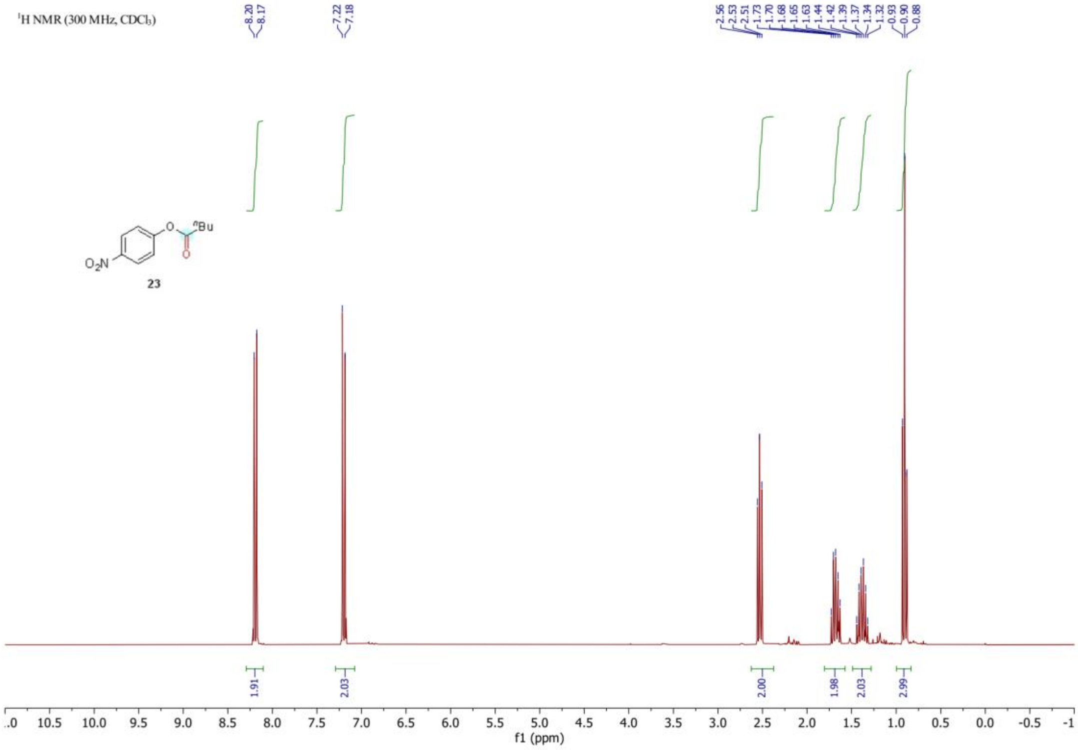
^{13}C NMR (101 MHz, CDCl$_3$)
190.75
171.37
155.31
133.72
130.98
122.19
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
33.88
26.67
22.02
13.53
OHC
^{n}Bu
21
f1 (ppm)

[1]H NMR (300 MHz, $CDCl_3$)

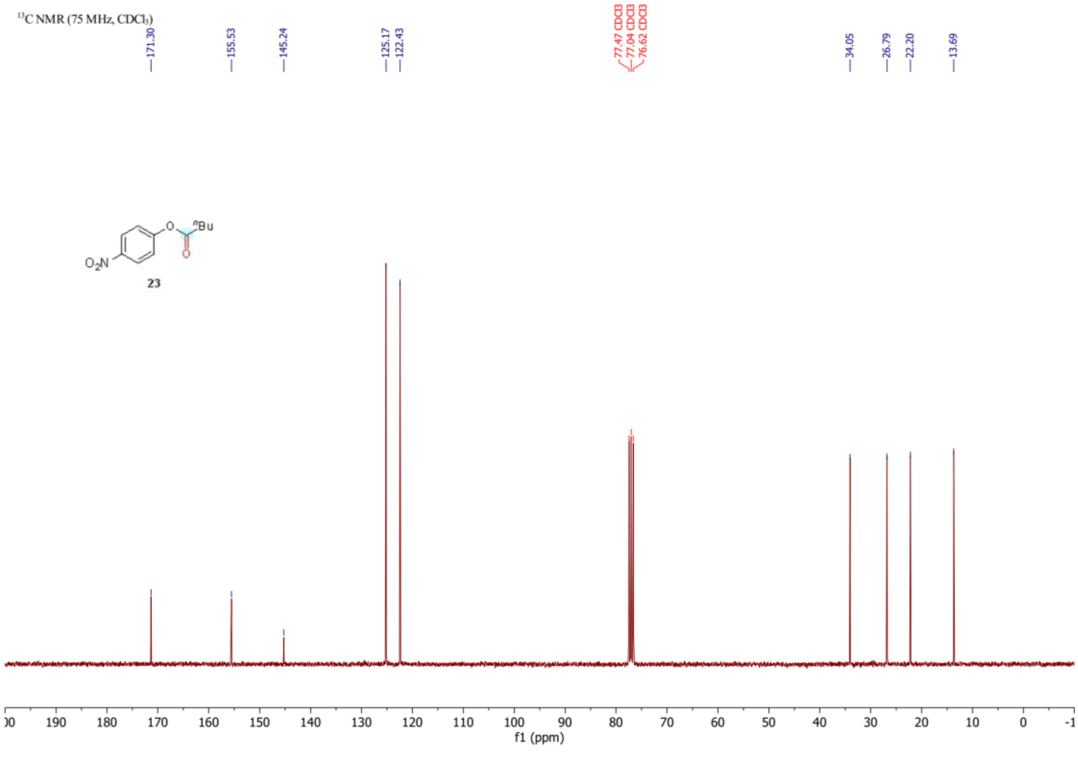


^{13}C NMR (75 MHz, $CDCl_3$)

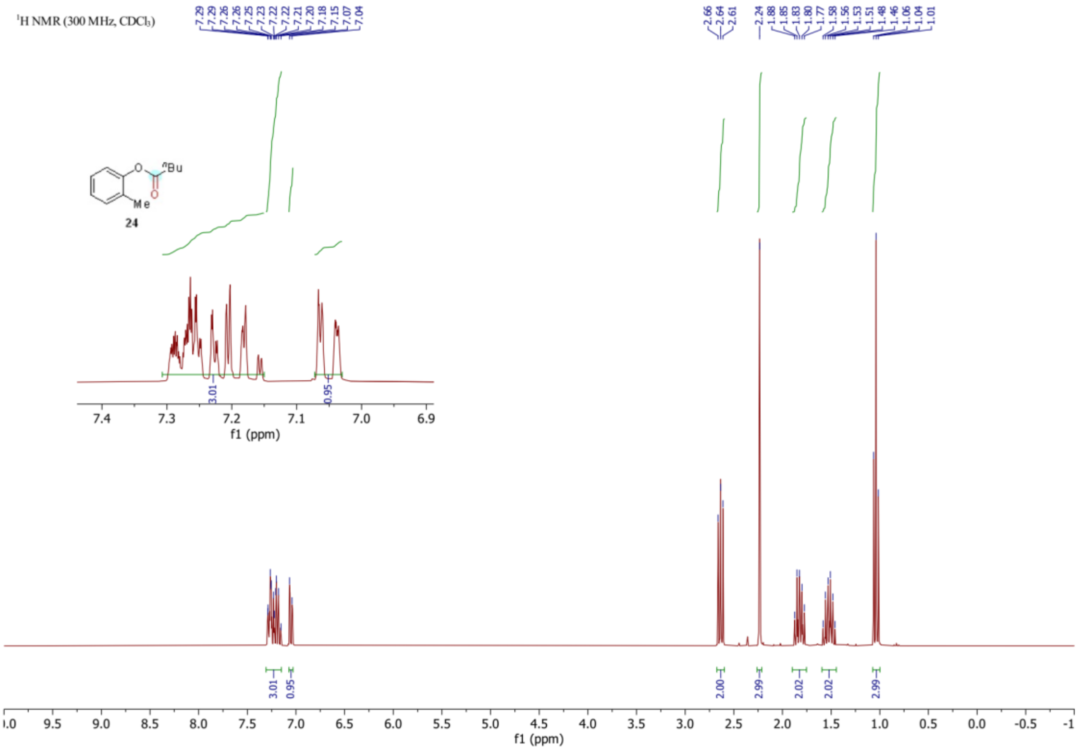


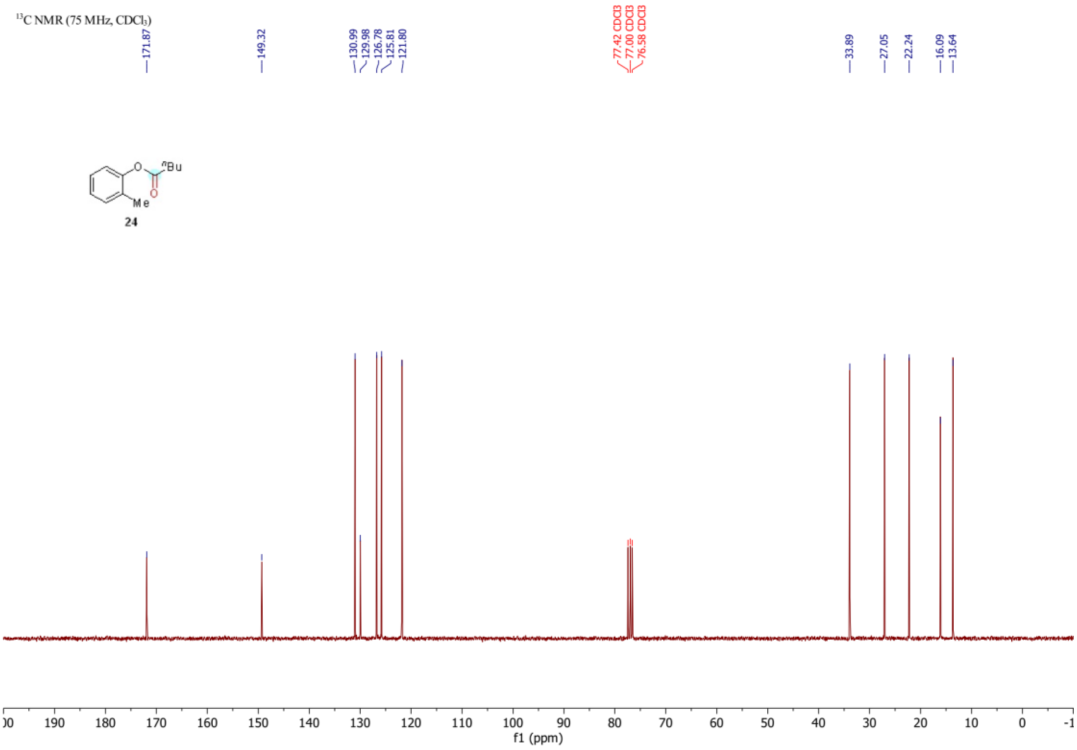


^{1}H NMR (300 MHz, CDCl$_3$)
8.20
8.17
7.22
7.18
2.56
2.53
2.51
1.73
1.70
1.68
1.65
1.63
1.44
1.42
1.39
1.37
1.34
1.32
0.93
0.90
0.88
1.91
2.03
2.00
1.98
2.03
2.99
f1 (ppm)

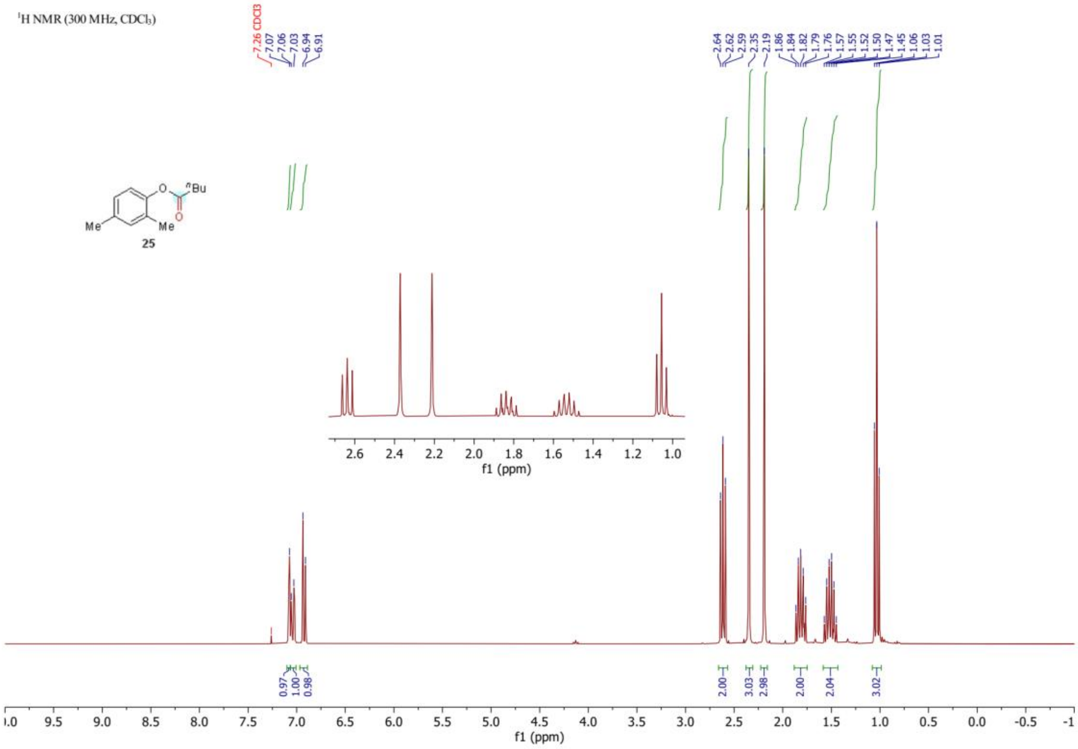


^{13}C NMR (75 MHz, CDCl$_3$)
171.30
155.53
145.24
125.17
122.43
77.47 CDCl3
77.04 CDCl3
76.62 CDCl3
34.05
26.79
22.20
13.69
f1 (ppm)

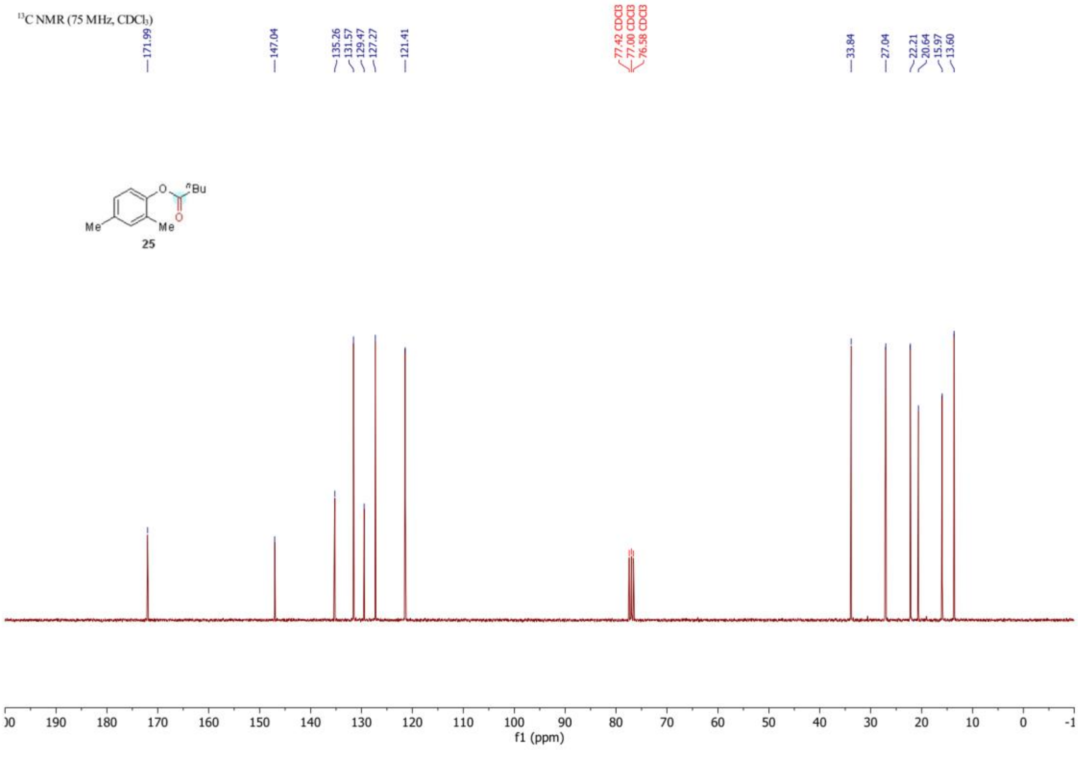

1H NMR (300 MHz, CDCl3)
24
f1 (ppm)


13C NMR (75 MHz, CDCl3)
24
f1 (ppm)

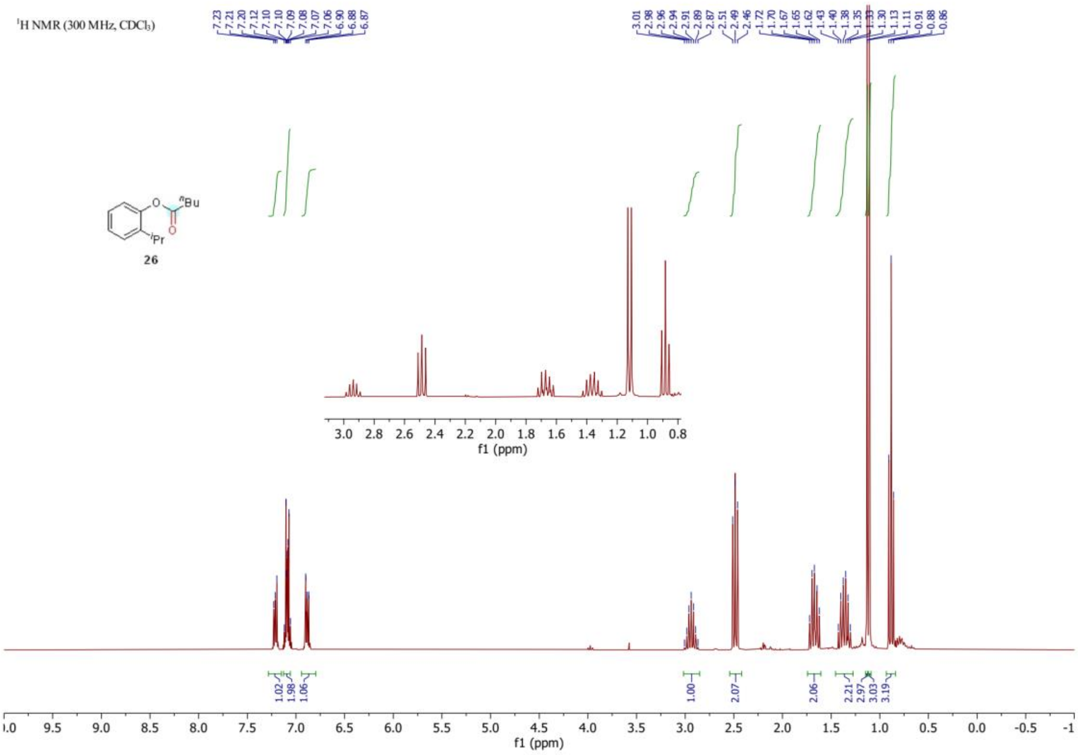
^{1}H NMR (300 MHz, $CDCl_3$)



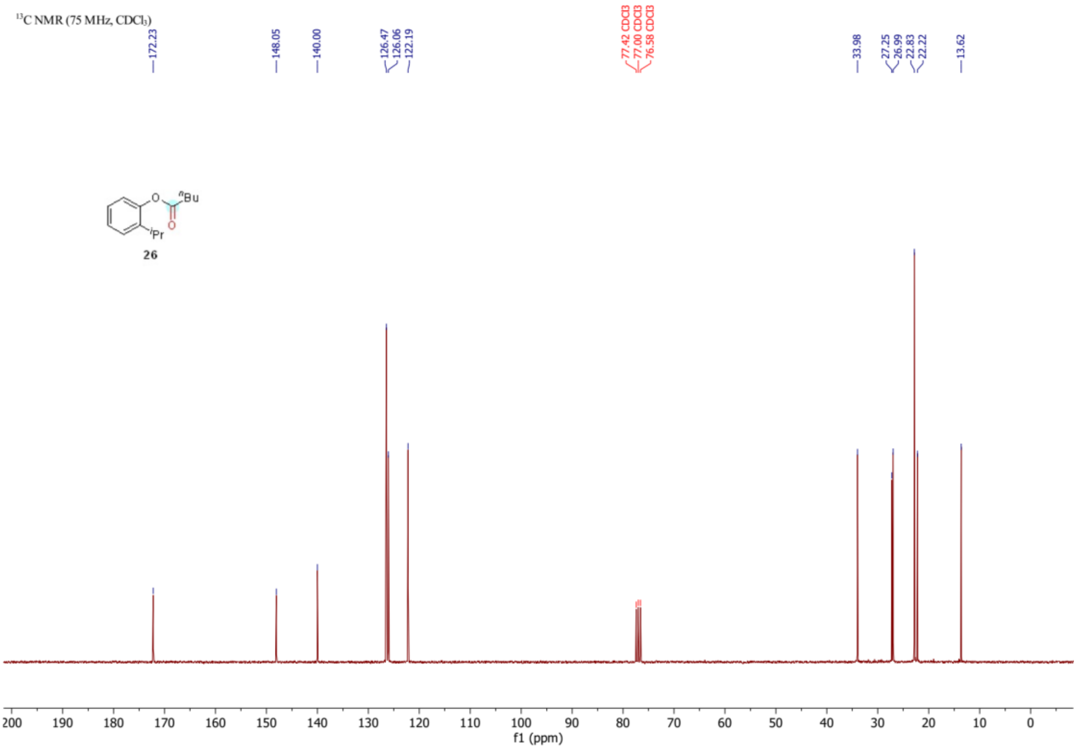
^{13}C NMR (75 MHz, $CDCl_3$)

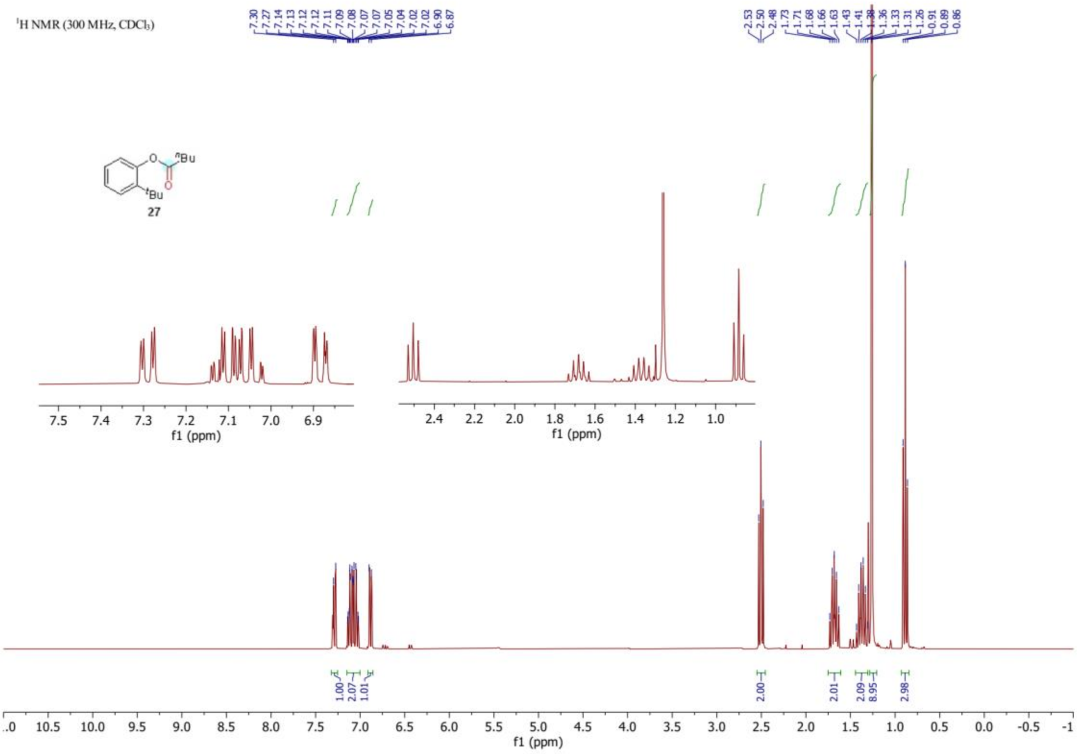


^{1}H NMR (300 MHz, $CDCl_3$)

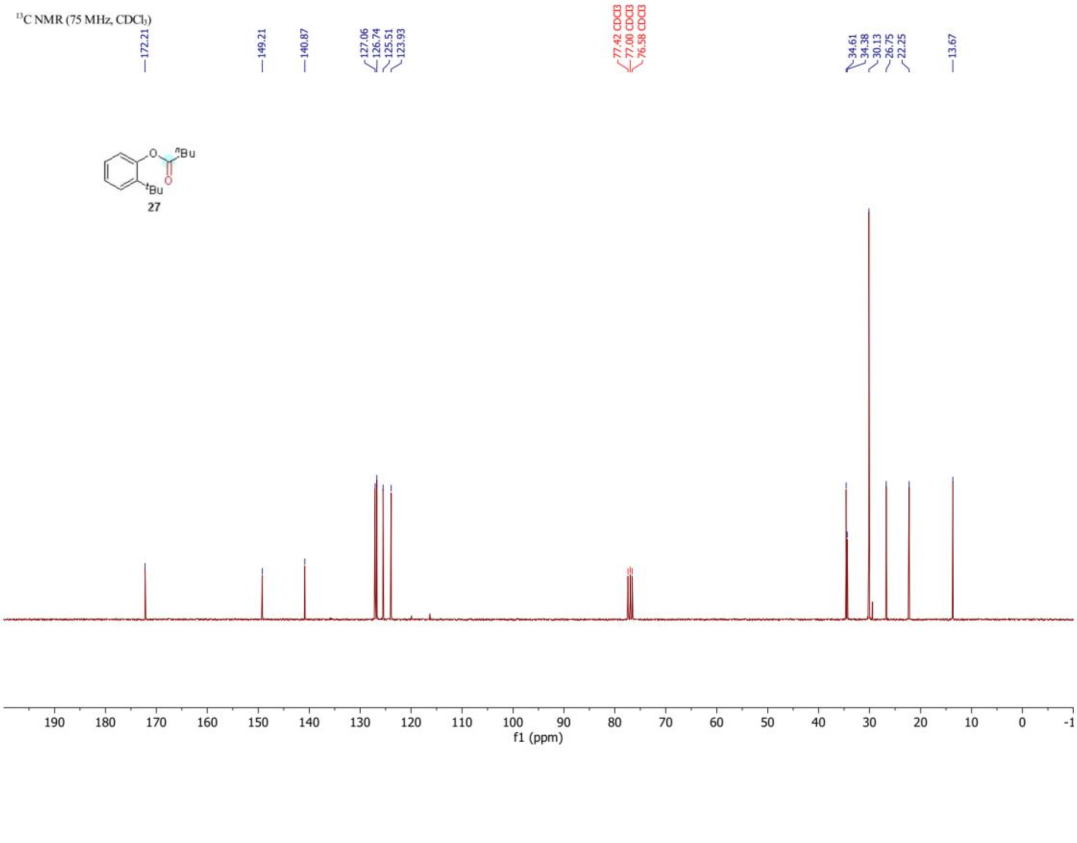


^{13}C NMR (75 MHz, $CDCl_3$)



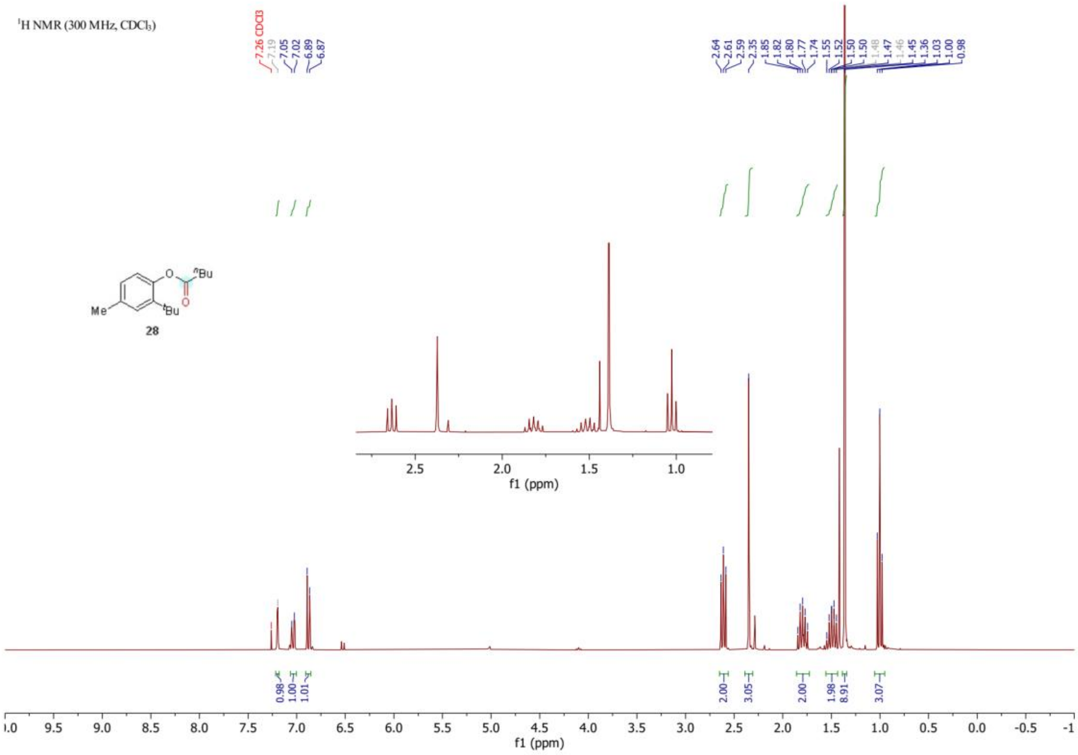


^{1}H NMR (300 MHz, CDCl$_3$)
27
f1 (ppm)

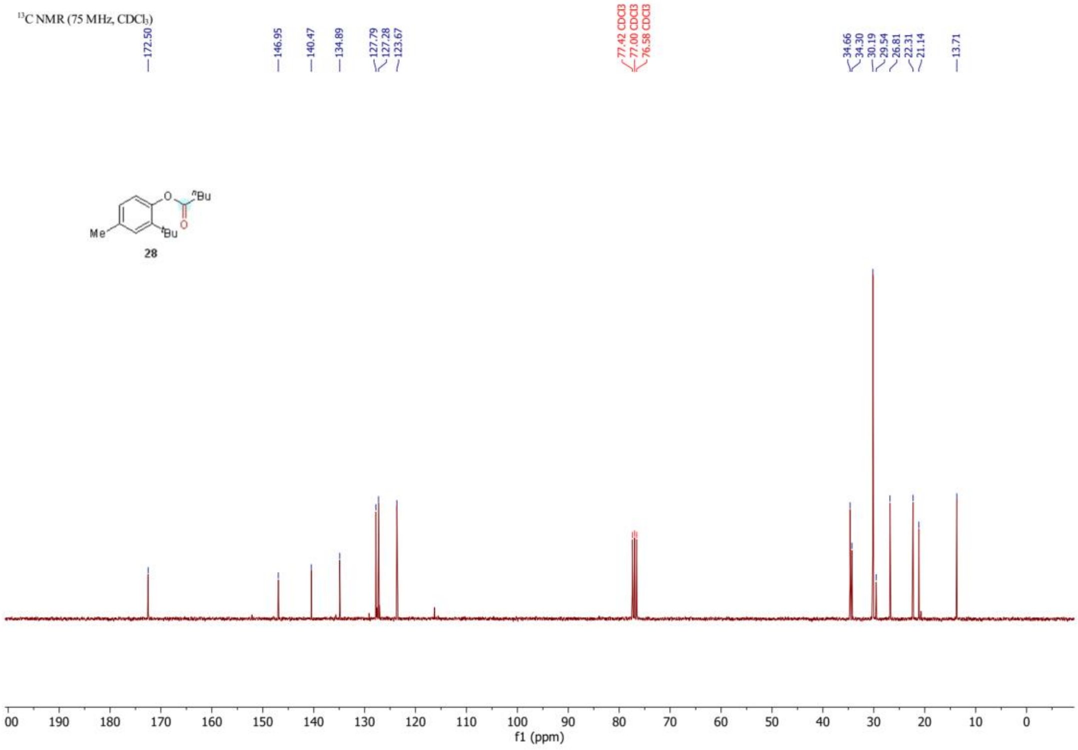


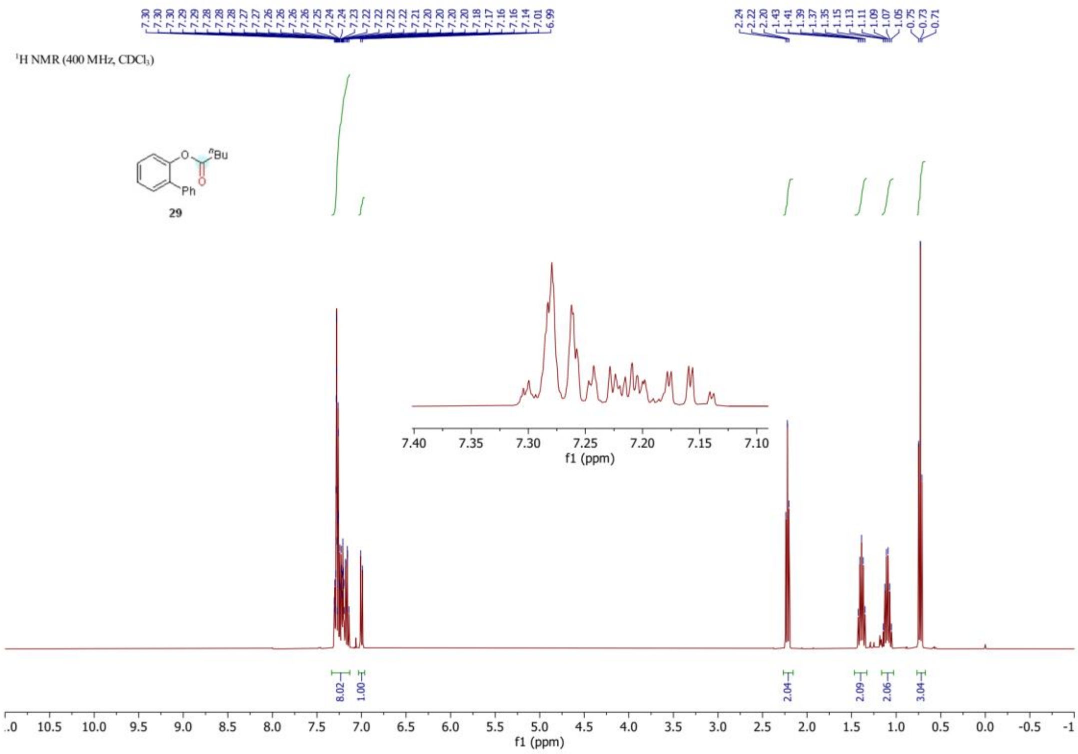
^{13}C NMR (75 MHz, CDCl$_3$)
27
f1 (ppm)

^1H NMR (300 MHz, $CDCl_3$)

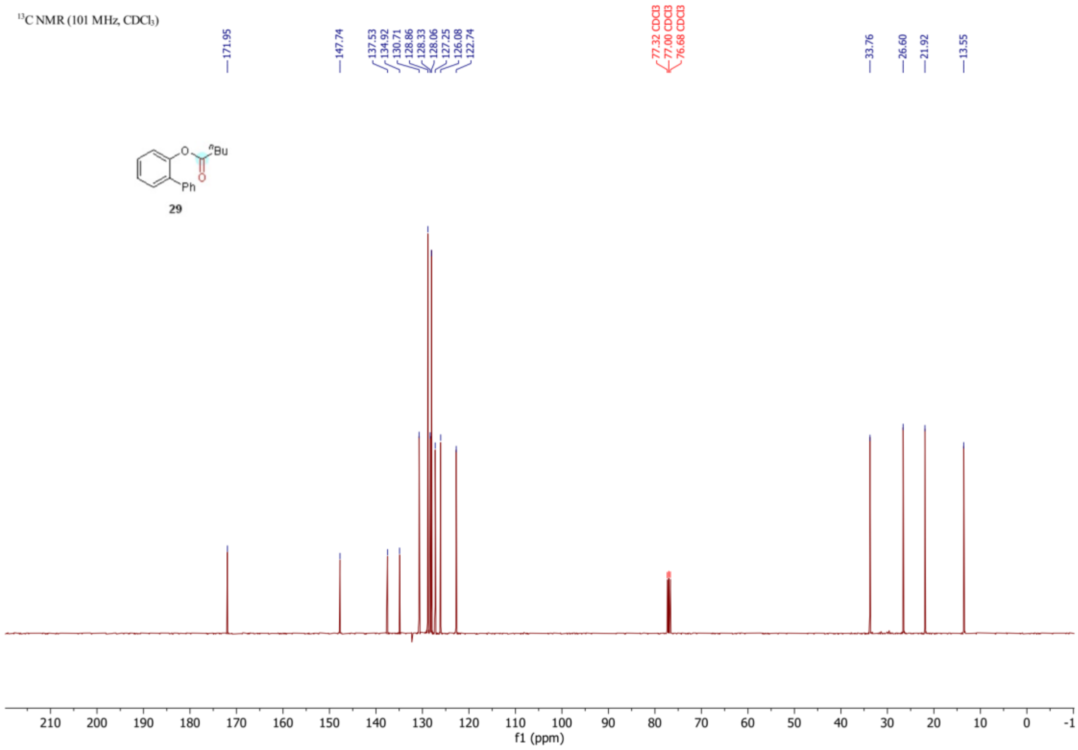


^{13}C NMR (75 MHz, $CDCl_3$)



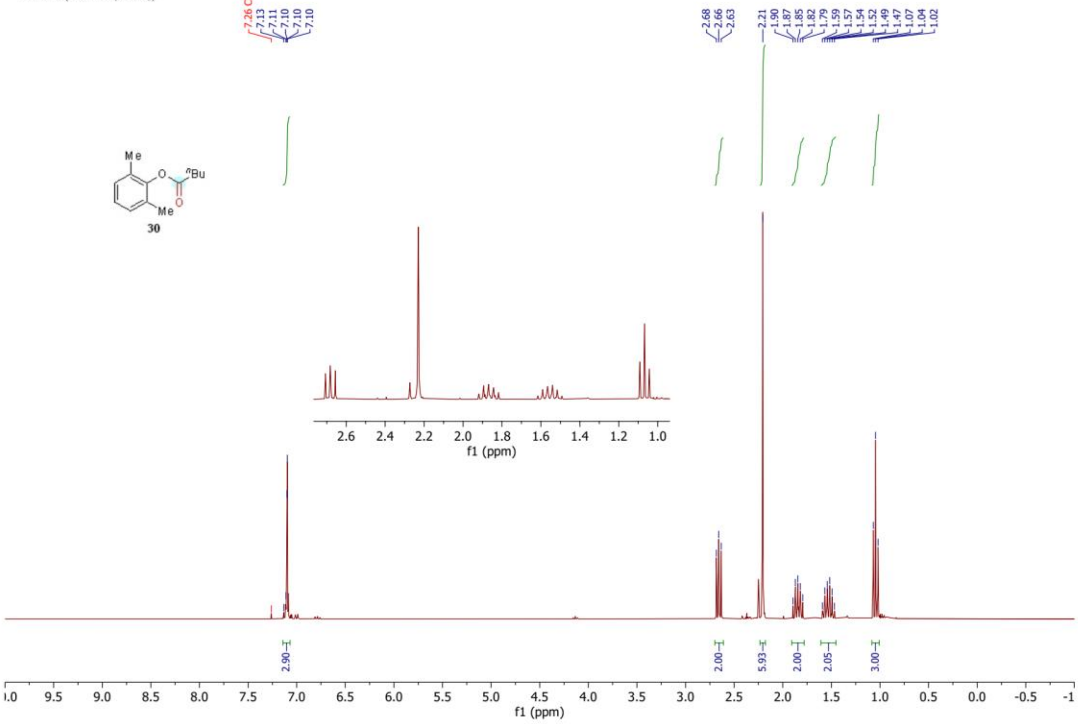


^{1}H NMR (400 MHz, CDCl$_3$)
29
f1 (ppm)

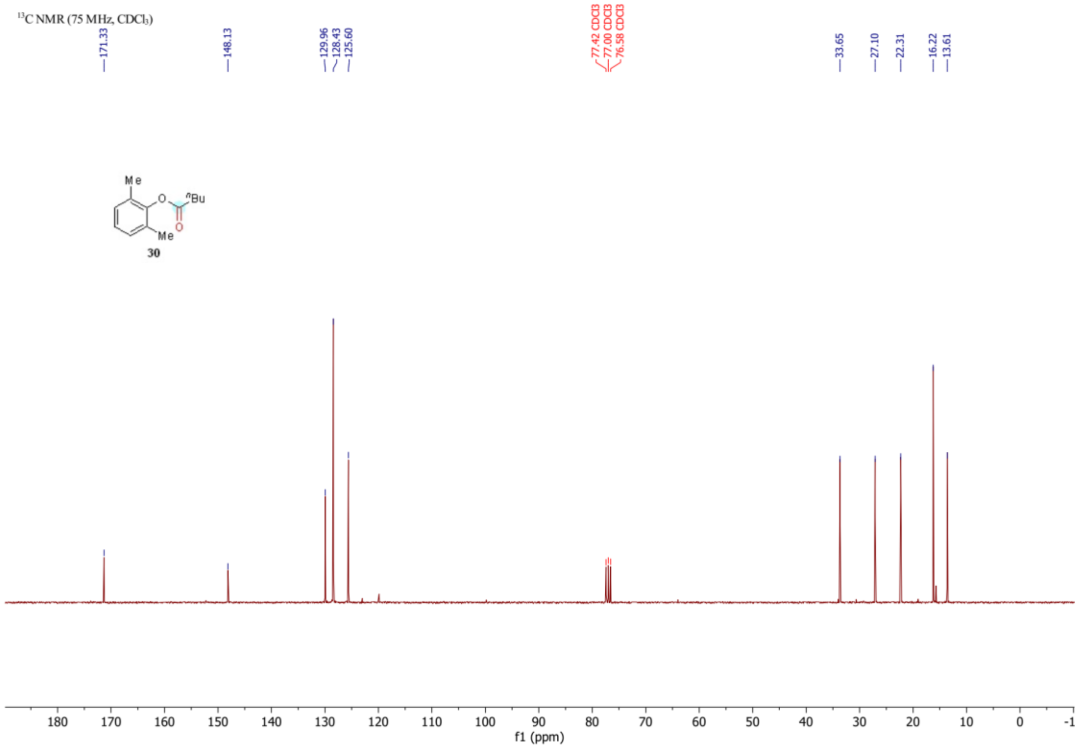


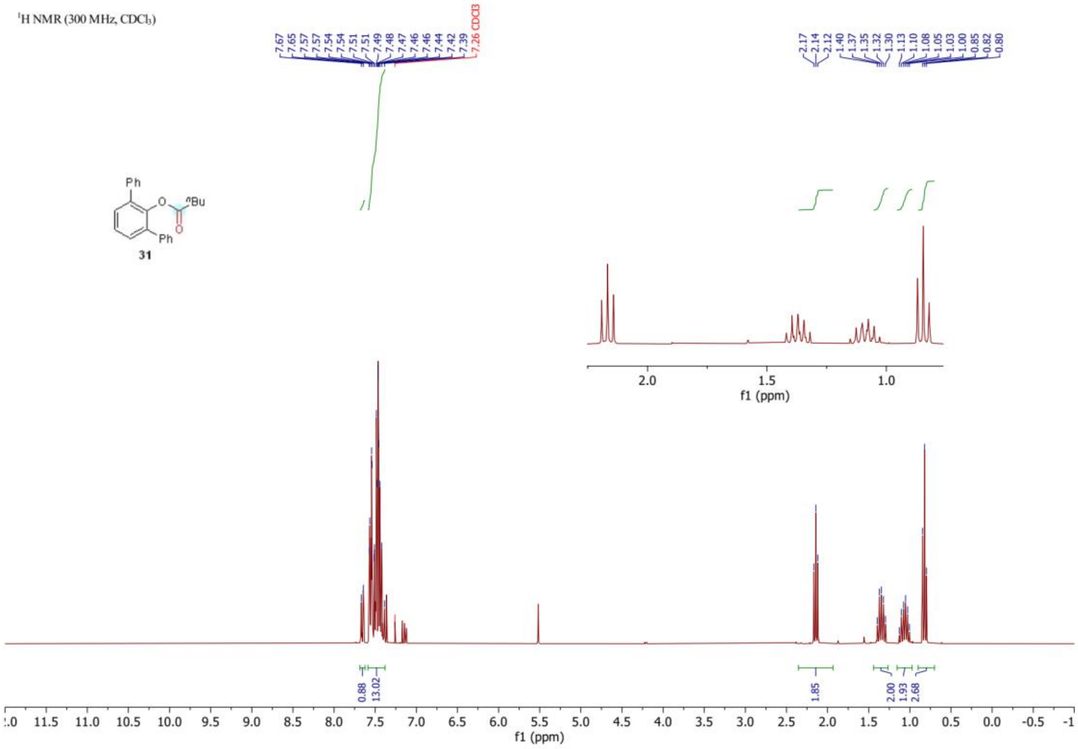
^{13}C NMR (101 MHz, CDCl$_3$)
29
f1 (ppm)

^{1}H NMR (300 MHz, $CDCl_3$)

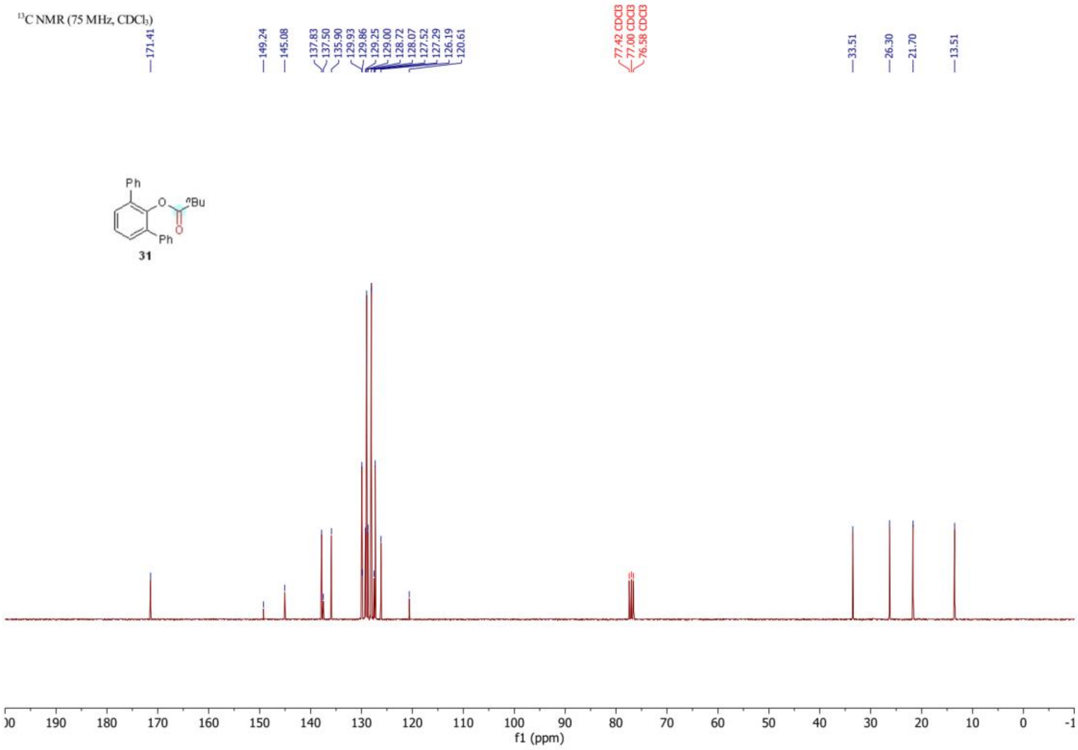


^{13}C NMR (75 MHz, $CDCl_3$)

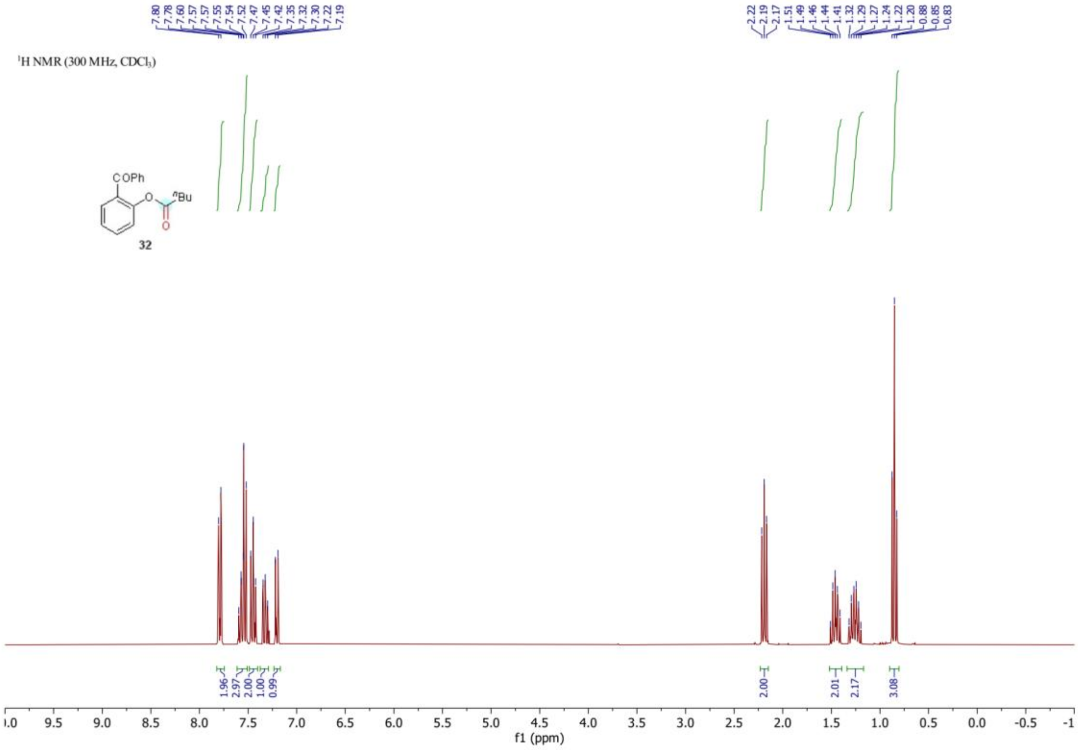




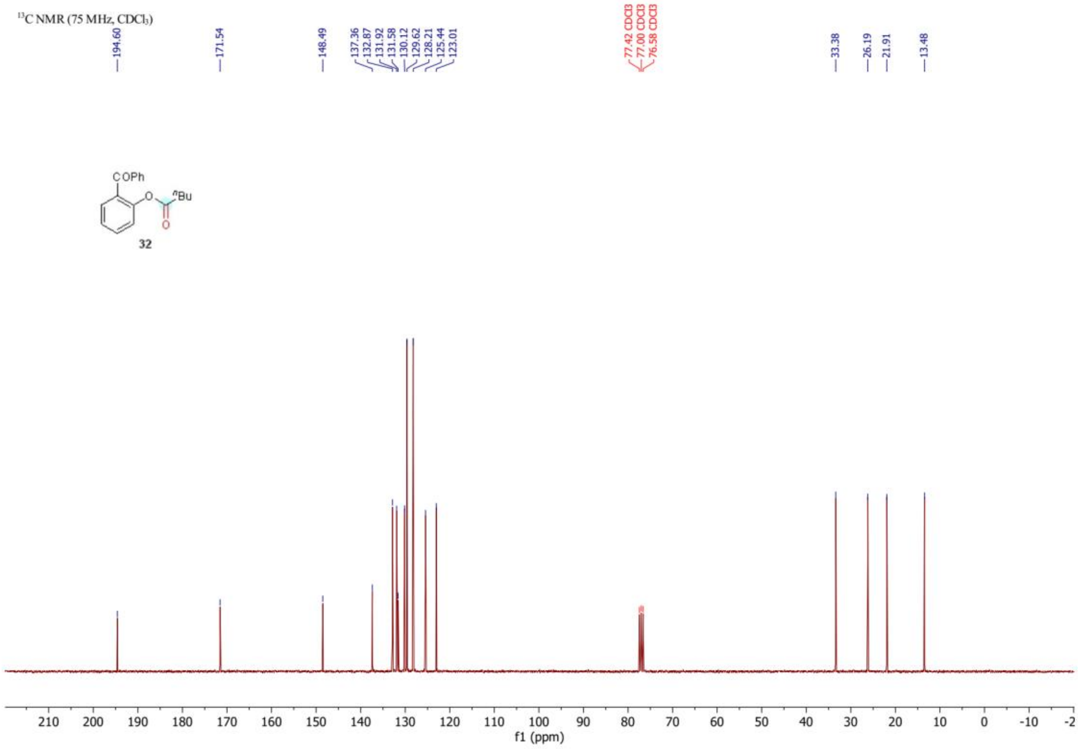
1H NMR (300 MHz, CDCl3)
7.67
7.65
7.57
7.57
7.54
7.54
7.51
7.51
7.49
7.48
7.47
7.46
7.46
7.44
7.42
7.39
7.26 CDCl3
2.17
2.14
2.12
1.40
1.37
1.35
1.32
1.30
1.13
1.10
1.08
1.05
1.03
1.00
0.85
0.82
0.80
Ph
O
nBu
O
Ph
31
0.88
13.02
1.85
2.00
1.93
2.68
f1 (ppm)



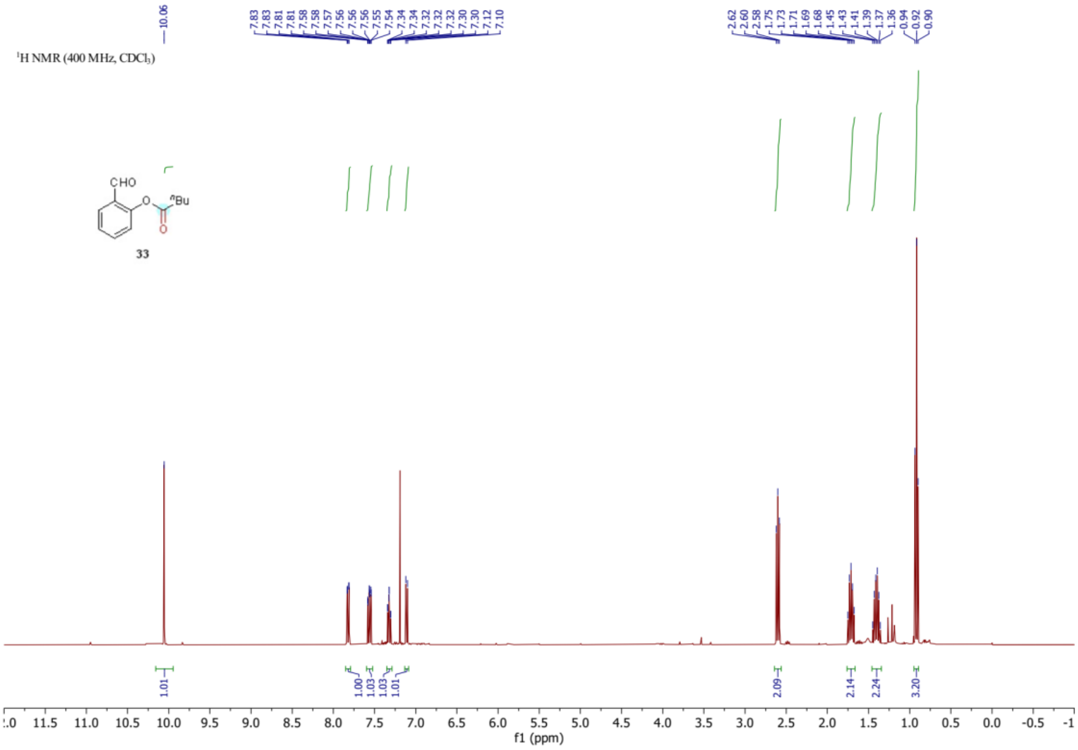
13C NMR (75 MHz, CDCl3)
171.41
149.24
145.08
137.83
137.50
135.90
129.93
129.86
129.25
129.00
128.72
128.07
127.52
127.29
126.19
120.61
77.42 CDCl3
77.00 CDCl3
76.58 CDCl3
33.51
26.30
21.70
13.51
Ph
O
nBu
O
Ph
31
f1 (ppm)



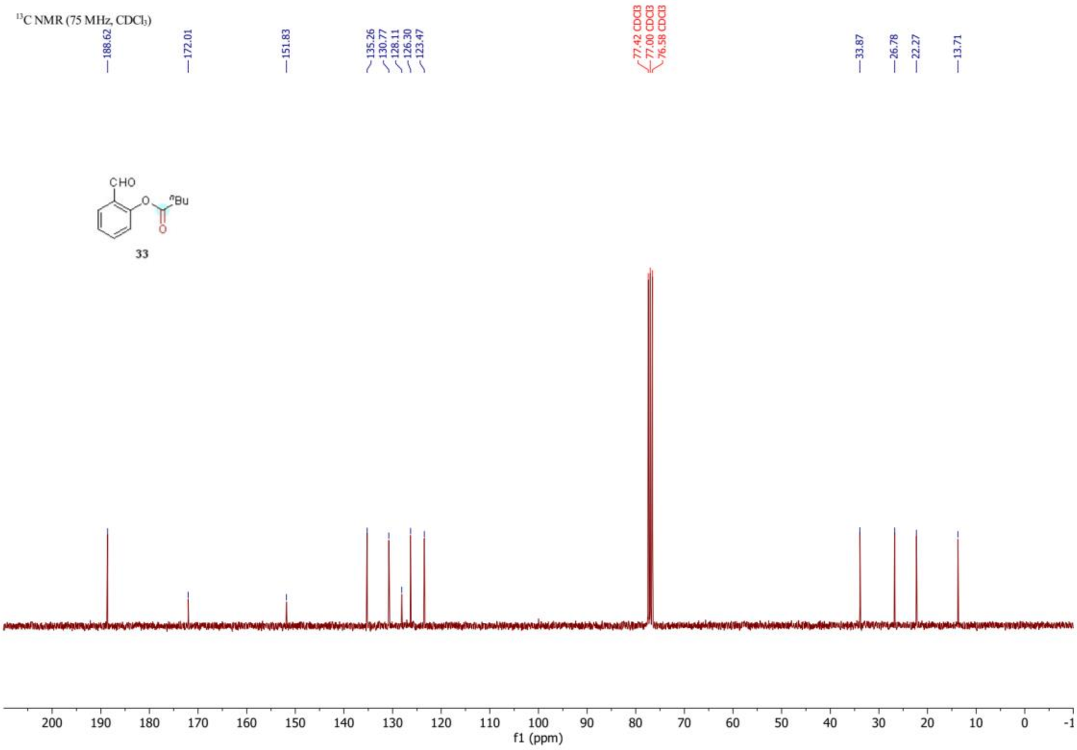
7.80
7.78
7.60
7.57
7.57
7.55
7.54
7.52
7.47
7.45
7.42
7.35
7.32
7.30
7.22
7.19
2.22
2.19
2.17
1.51
1.49
1.46
1.44
1.41
1.32
1.29
1.27
1.24
1.22
1.20
0.88
0.85
0.83
¹H NMR (300 MHz, CDCl₃)
COPh
O
O
nBu
32
1.96
2.97
2.00
1.00
0.99
2.00
2.01
2.17
3.08
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
f1 (ppm)



¹³C NMR (75 MHz, CDCl₃)
194.60
171.54
148.49
137.36
132.87
131.92
131.58
130.12
129.62
128.21
125.44
123.01
77.42 CDCl3
77.00 CDCl3
76.58 CDCl3
33.38
26.19
21.91
13.48
COPh
O
O
nBu
32
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -2
f1 (ppm)

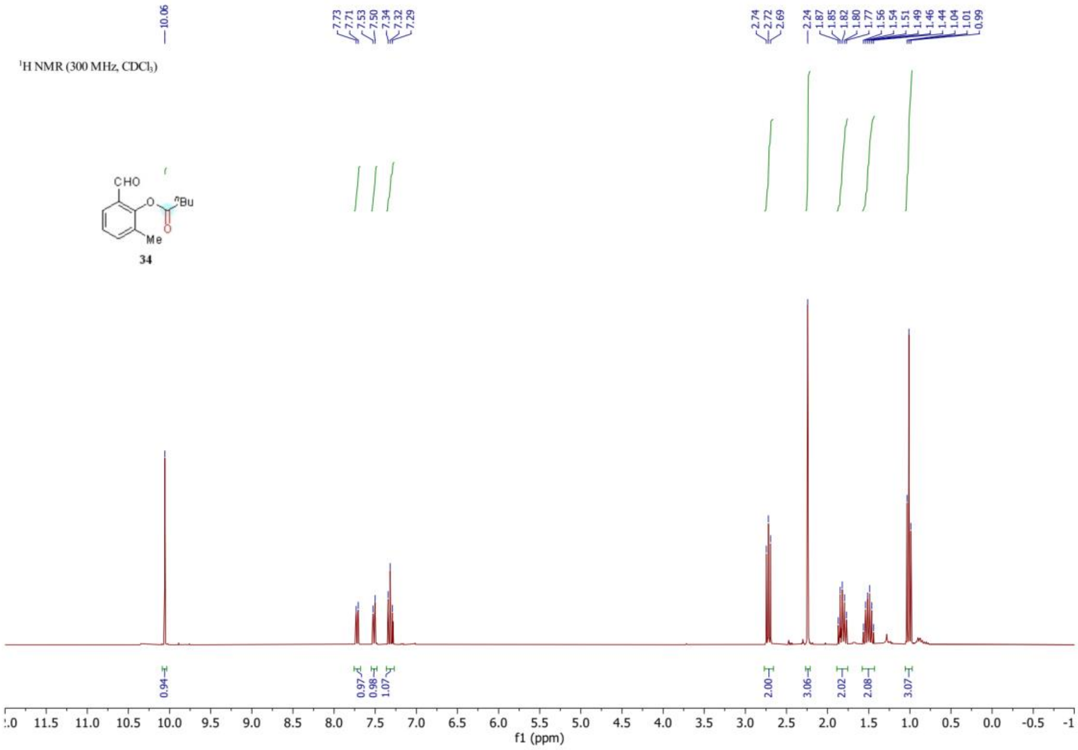


33

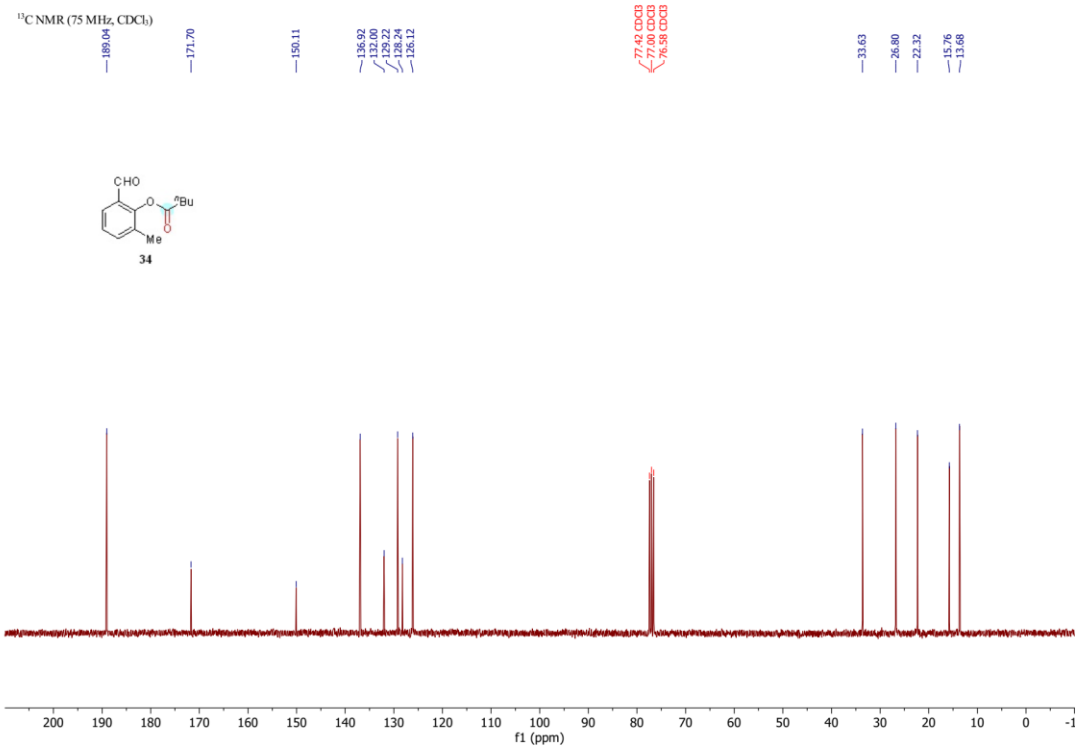


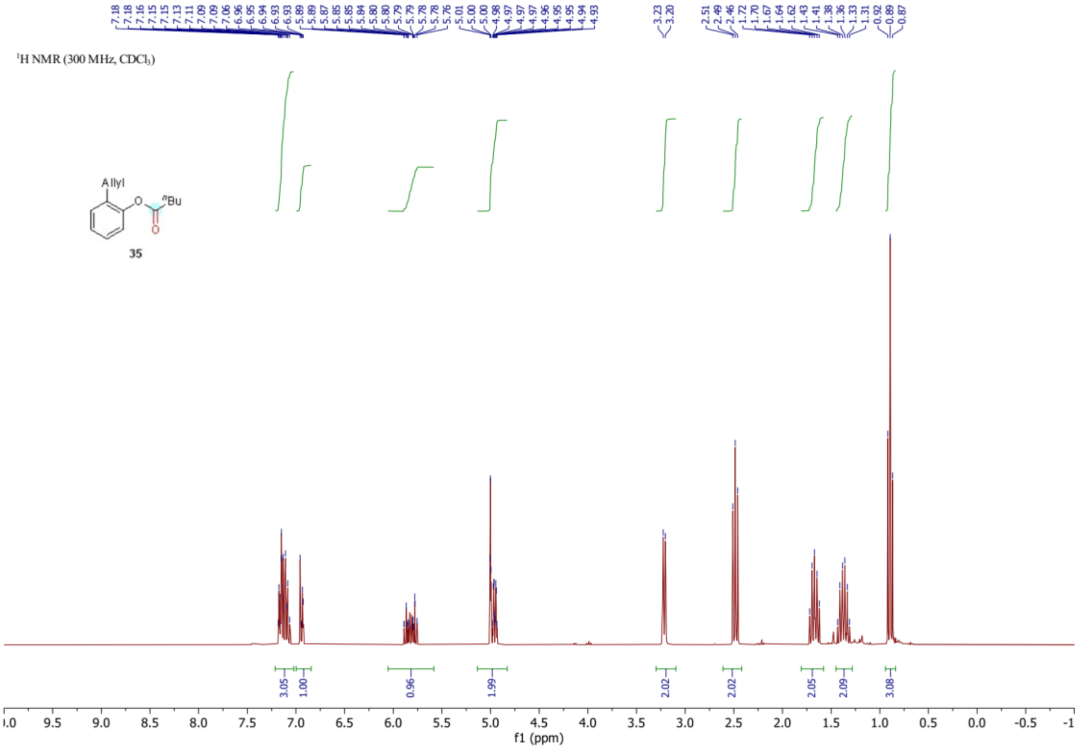
33

^{1}H NMR (300 MHz, $CDCl_3$)

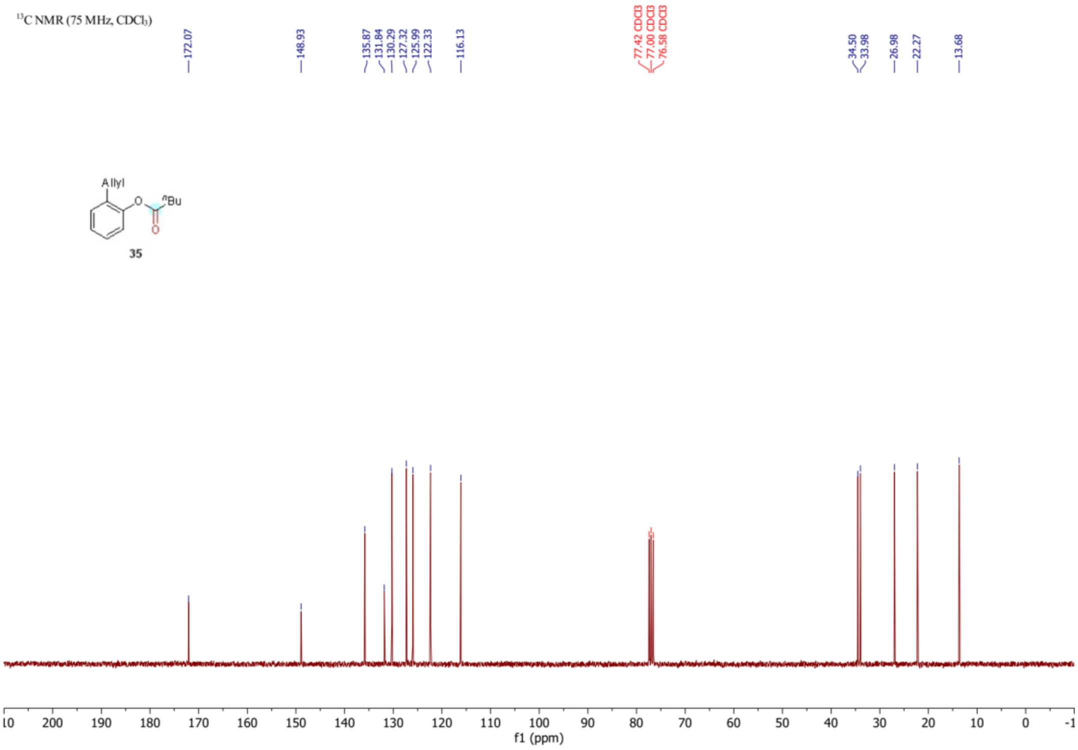


^{13}C NMR (75 MHz, $CDCl_3$)

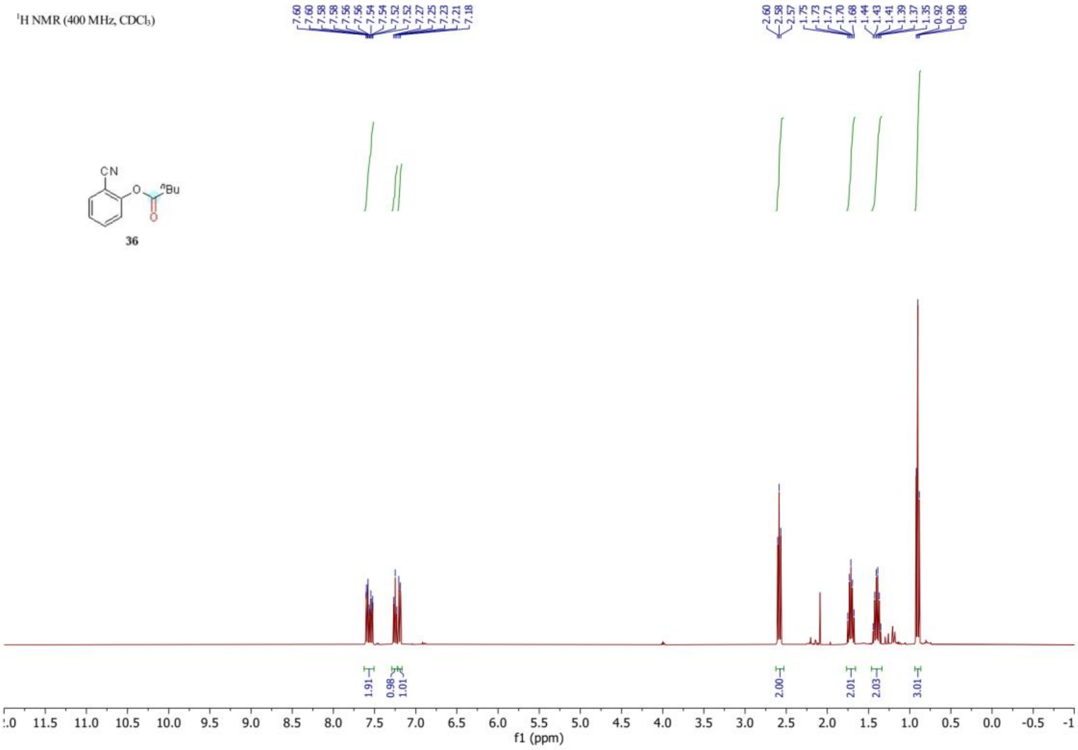




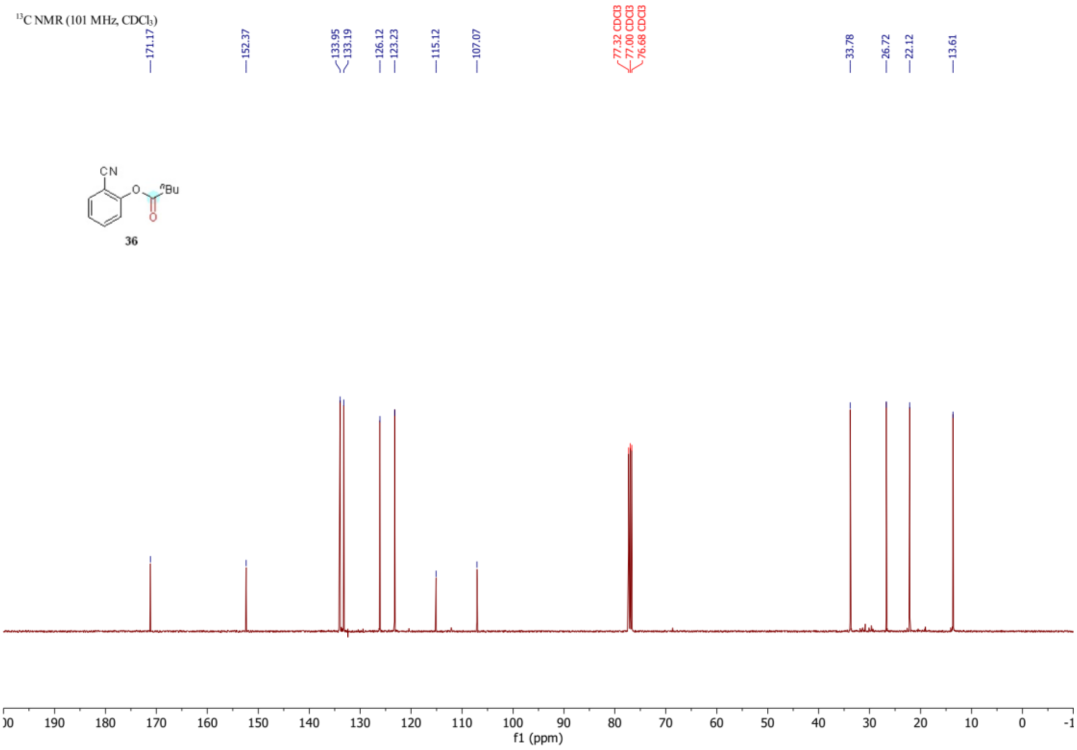
7.18 7.18 7.16 7.15 7.15 7.13 7.11 7.09 7.09 7.06 6.96 6.95 6.94 6.93 6.93 5.89 5.89 5.87 5.85 5.85 5.84 5.80 5.80 5.79 5.79 5.78 5.78 5.76 5.01 5.00 5.00 4.98 4.97 4.97 4.96 4.95 4.95 4.94 4.93 3.23 3.20 2.51 2.49 2.46 1.72 1.70 1.67 1.64 1.62 1.43 1.41 1.38 1.36 1.33 1.31 0.92 0.89 0.87
1H NMR (300 MHz, CDCl3)
Allyl
O
nBu
O
35
3.05 1.00 0.96 1.99 2.02 2.02 2.05 2.09 3.08
f1 (ppm)



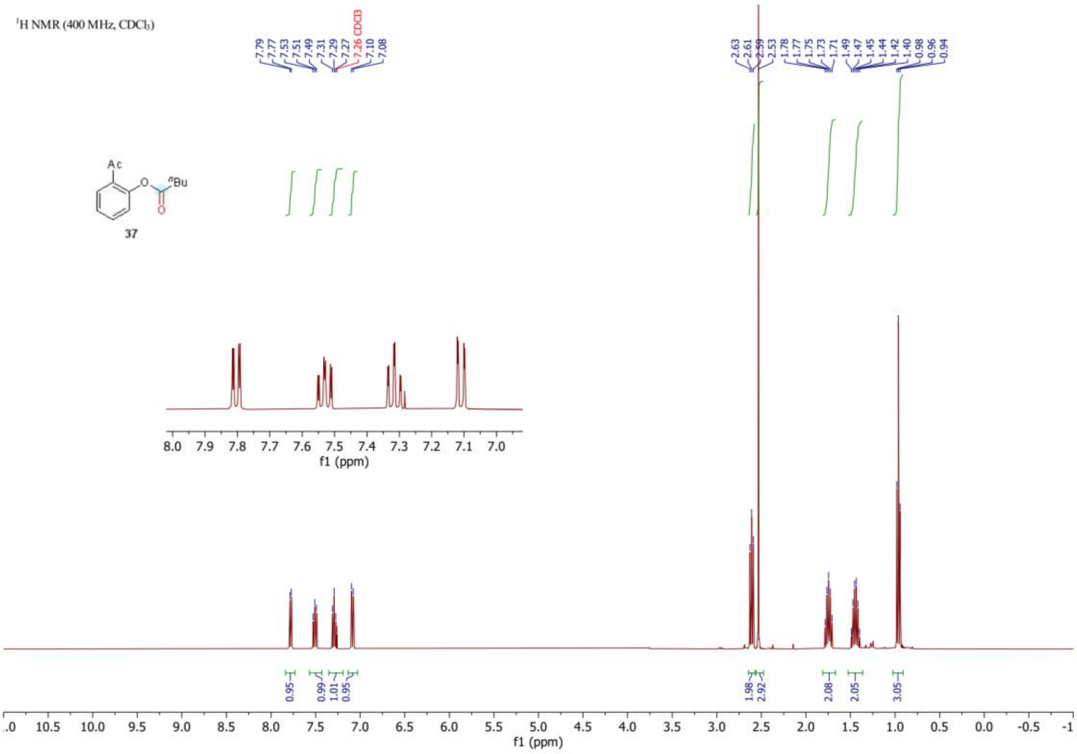
13C NMR (75 MHz, CDCl3)
172.07
148.93
135.87 131.84 130.29 127.32 125.99 122.33
116.13
77.42 CDCl3 77.00 CDCl3 76.58 CDCl3
34.50 33.98
26.98
22.27
13.68
Allyl
O
nBu
O
35
f1 (ppm)



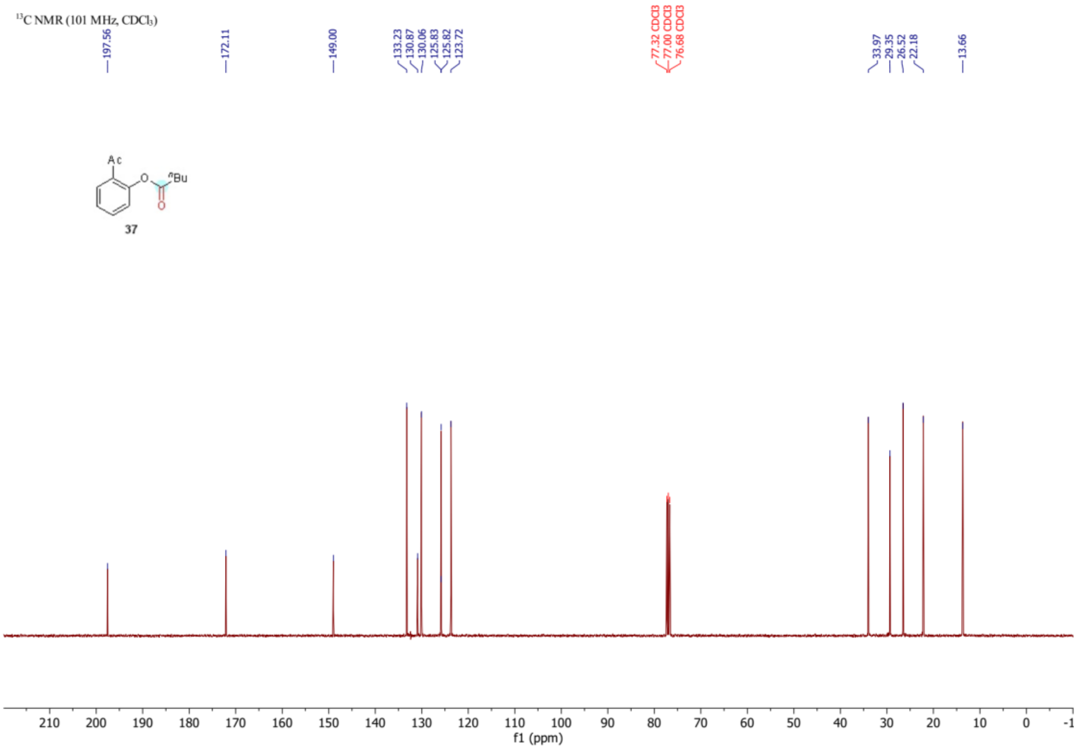
^{1}H NMR (400 MHz, CDCl$_3$)
CN
O
^{n}Bu
O
36
f1 (ppm)



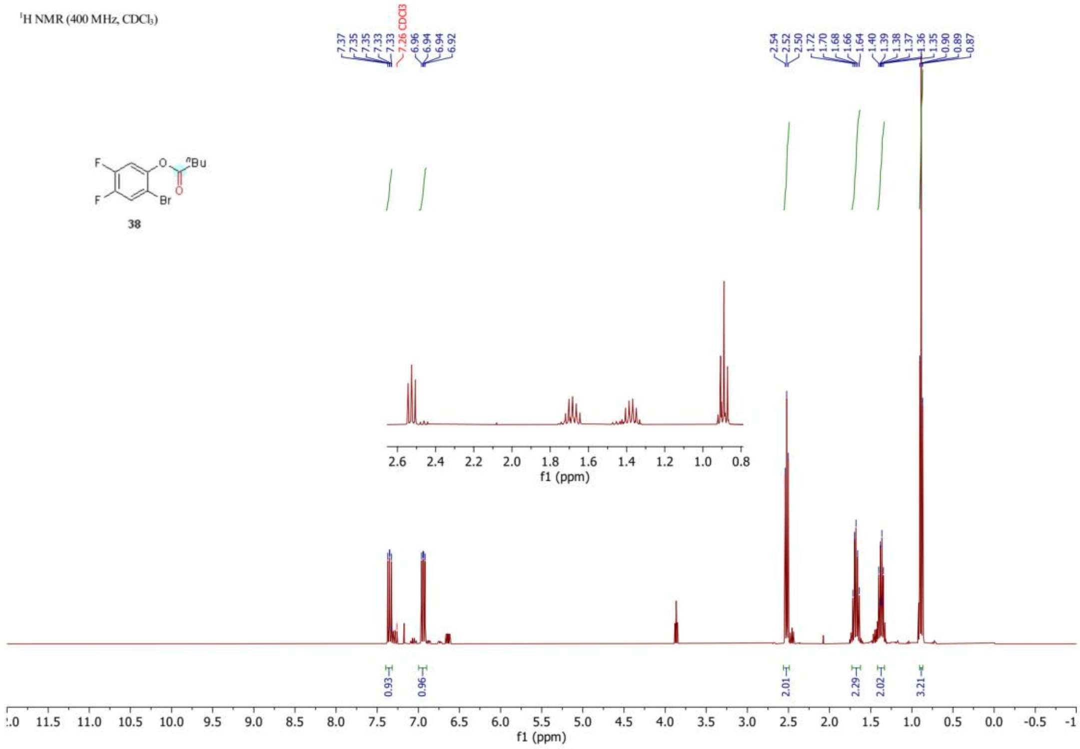
^{13}C NMR (101 MHz, CDCl$_3$)
CN
O
^{n}Bu
O
36
f1 (ppm)



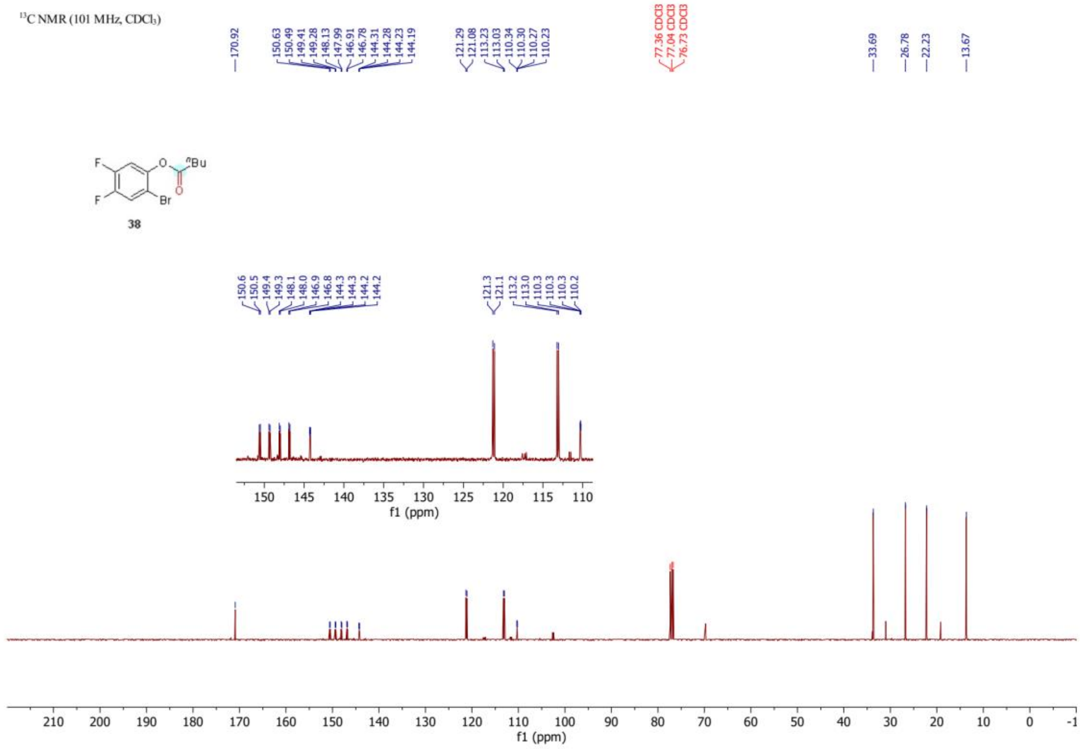
^{1}H NMR (400 MHz, CDCl$_3$)
Ac
O
^{n}Bu
O
37
7.79
7.77
7.53
7.51
7.49
7.31
7.29
7.27
7.26 CDCl3
7.10
7.08
2.63
2.61
2.59
2.53
1.78
1.77
1.75
1.73
1.71
1.49
1.47
1.45
1.44
1.42
1.40
0.98
0.96
0.94
0.95
0.99
1.01
0.95
1.98
2.92
2.08
2.05
3.05
f1 (ppm)



^{13}C NMR (101 MHz, CDCl$_3$)
197.56
172.11
149.00
133.23
130.87
130.06
125.83
125.82
123.72
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
33.97
29.35
26.52
22.18
13.66
Ac
O
^{n}Bu
O
37
f1 (ppm)

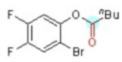


¹H NMR (400 MHz, CDCl₃)
7.37
7.35
7.35
7.33
7.33
7.26 CDCl3
6.96
6.94
6.94
6.92
2.54
2.52
2.50
1.72
1.70
1.68
1.66
1.64
1.40
1.39
1.38
1.37
1.36
1.35
0.90
0.89
0.87
38
0.93
0.96
2.01
2.29
2.02
3.21
f1 (ppm)

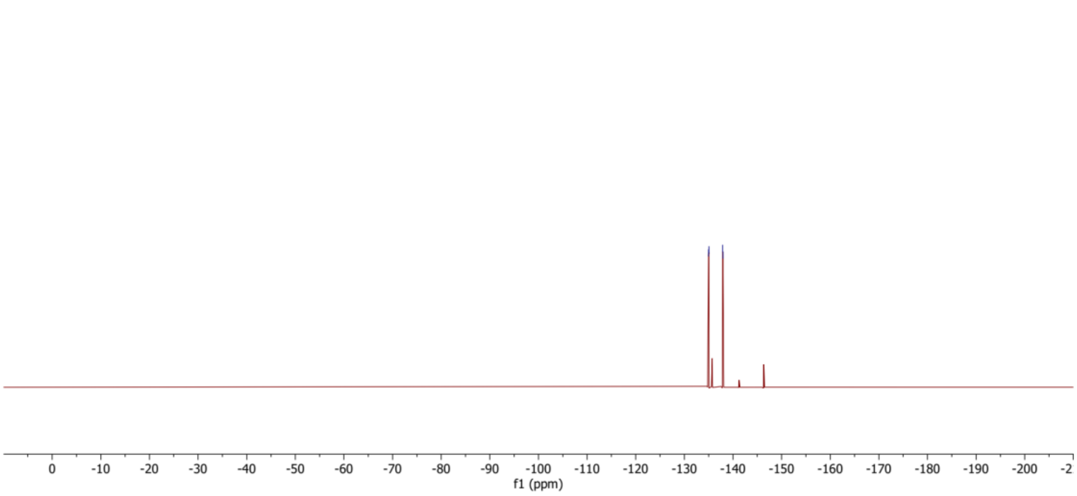


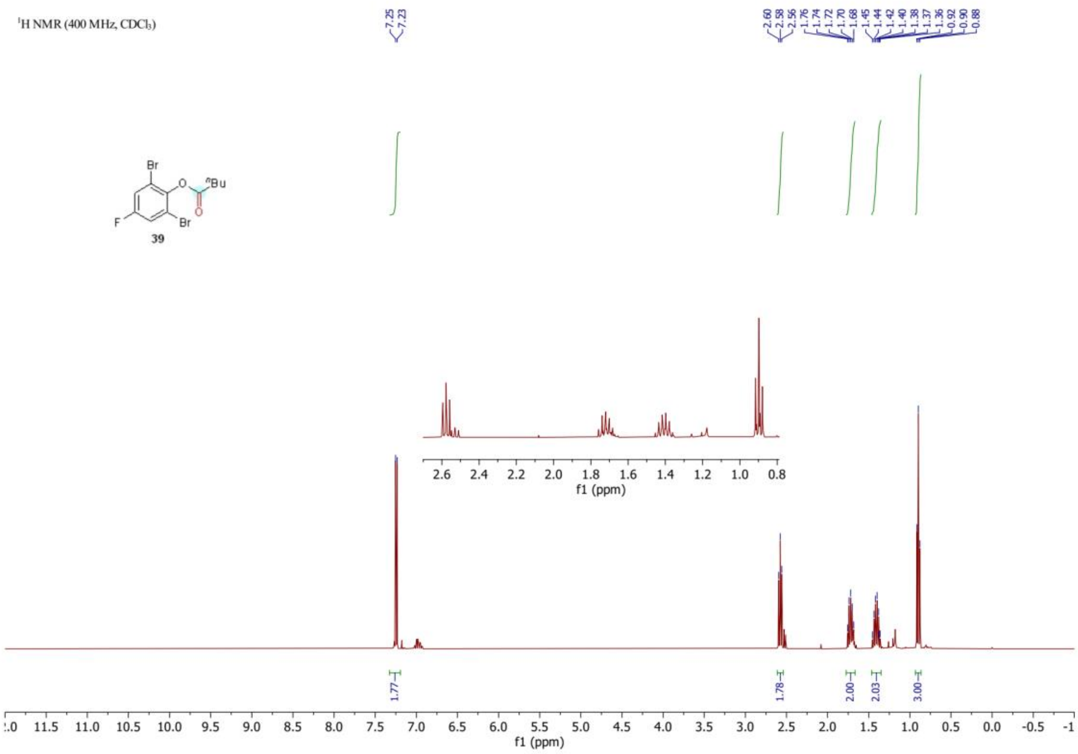
¹³C NMR (101 MHz, CDCl₃)
170.92
150.63
150.49
149.41
149.28
148.13
147.99
146.91
146.78
144.31
144.28
144.23
144.19
121.29
121.08
113.23
113.03
110.34
110.30
110.27
110.23
77.36 CDCl3
77.04 CDCl3
76.73 CDCl3
33.69
26.78
22.23
13.67
38
f1 (ppm)

^{19}F NMR (282 MHz, CDCl$_3$)

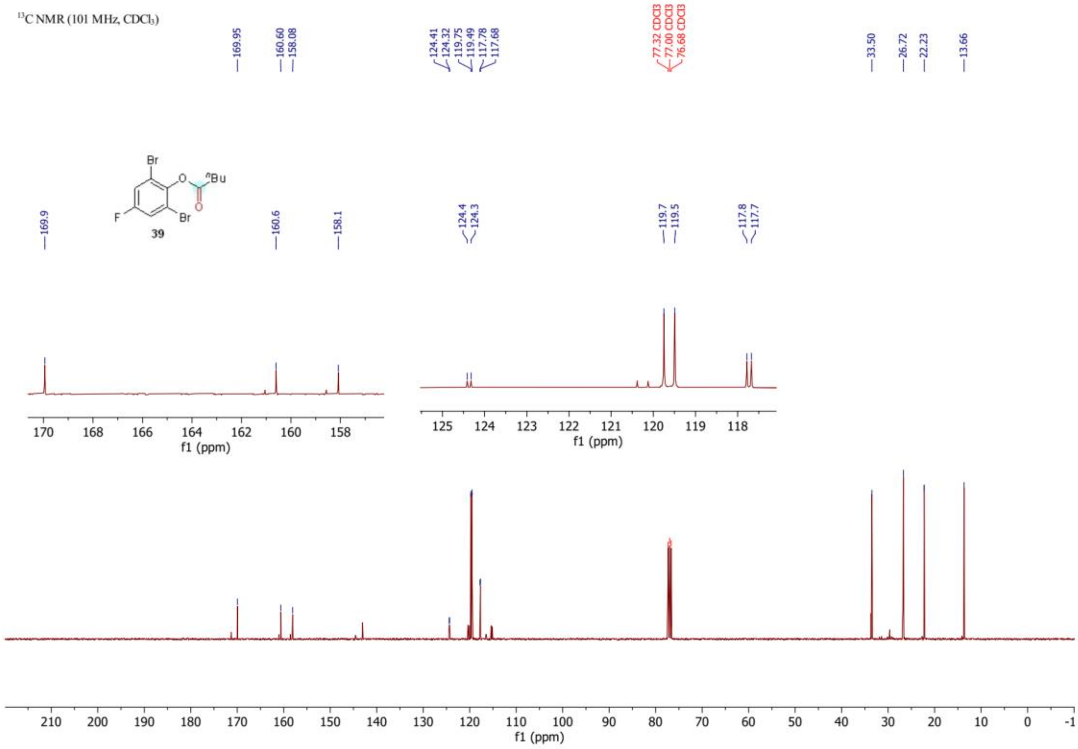


38



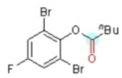


^{1}H NMR (400 MHz, CDCl$_3$)
7.25
7.23
2.60
2.58
2.56
1.76
1.74
1.72
1.70
1.68
1.45
1.44
1.42
1.40
1.38
1.37
1.36
0.92
0.90
0.88
Br
O
^{n}Bu
O
F
Br
39
1.77
1.78
2.00
2.03
3.00
f1 (ppm)

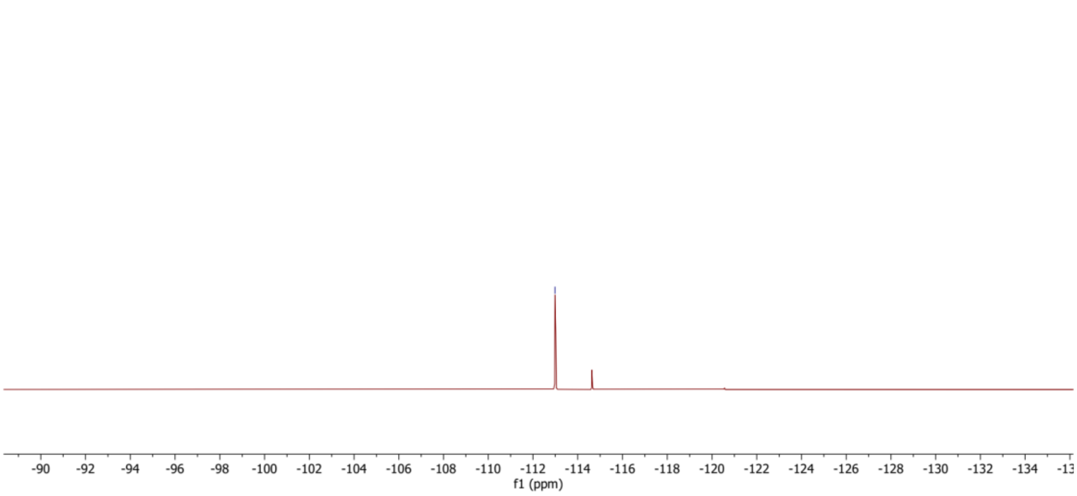


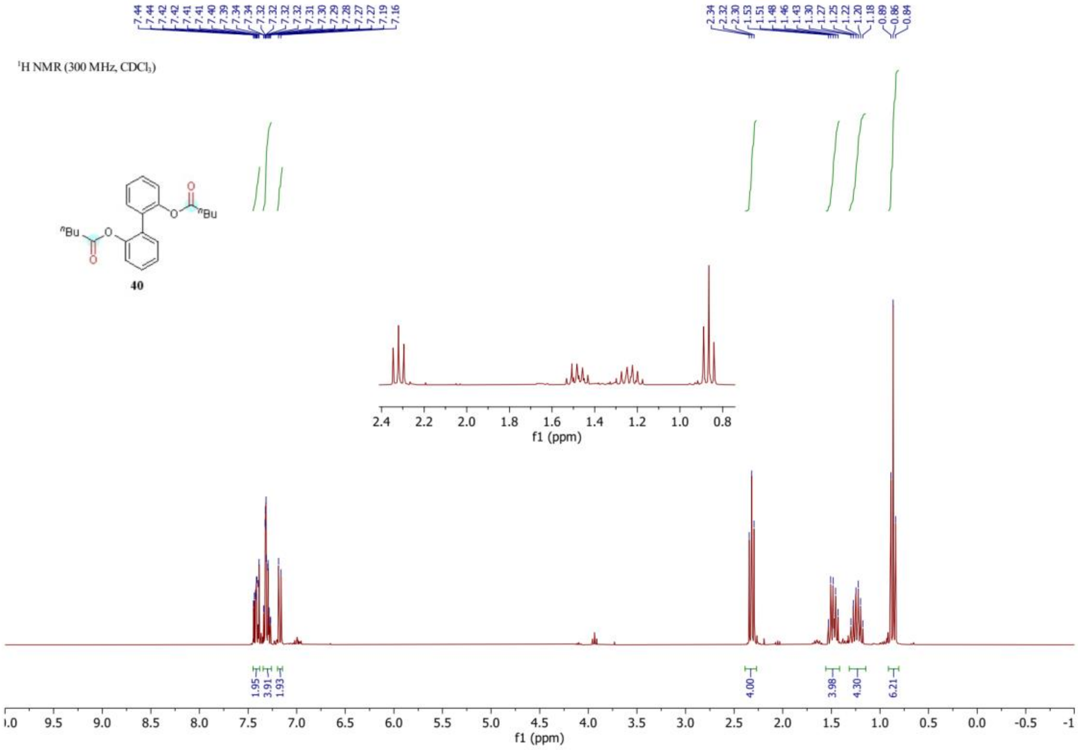
^{13}C NMR (101 MHz, CDCl$_3$)
169.95
160.60
158.08
124.41
124.32
119.75
119.49
117.78
117.68
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
33.50
26.72
22.23
13.66
Br
O
^{n}Bu
O
F
Br
39
169.9
160.6
158.1
124.4
124.3
119.7
119.5
117.8
117.7
f1 (ppm)

^{19}F NMR (282 MHz, $CDCl_3$)

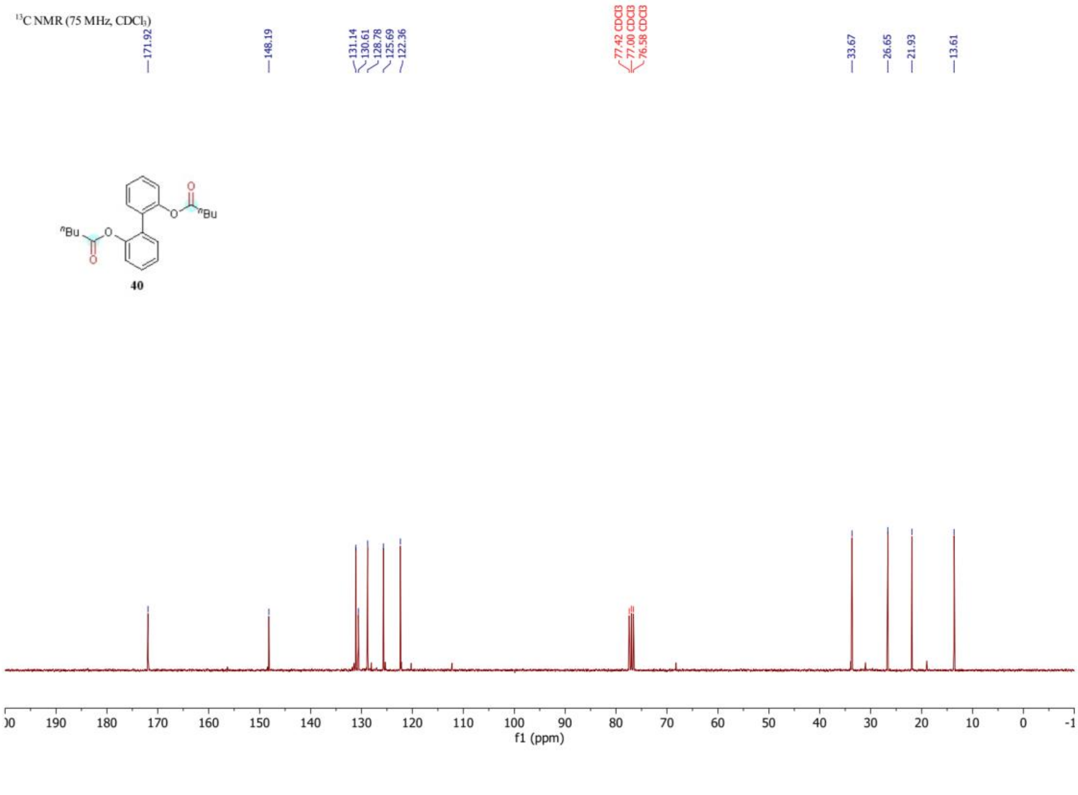


39



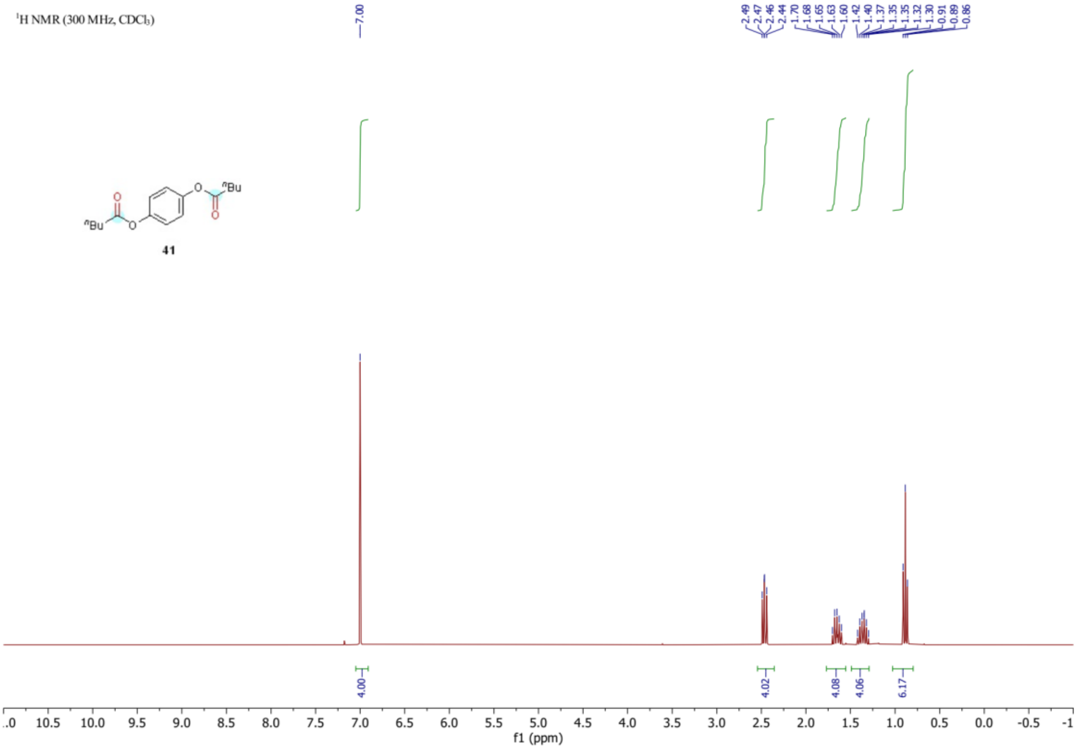


^{1}H NMR (300 MHz, CDCl$_3$)
40

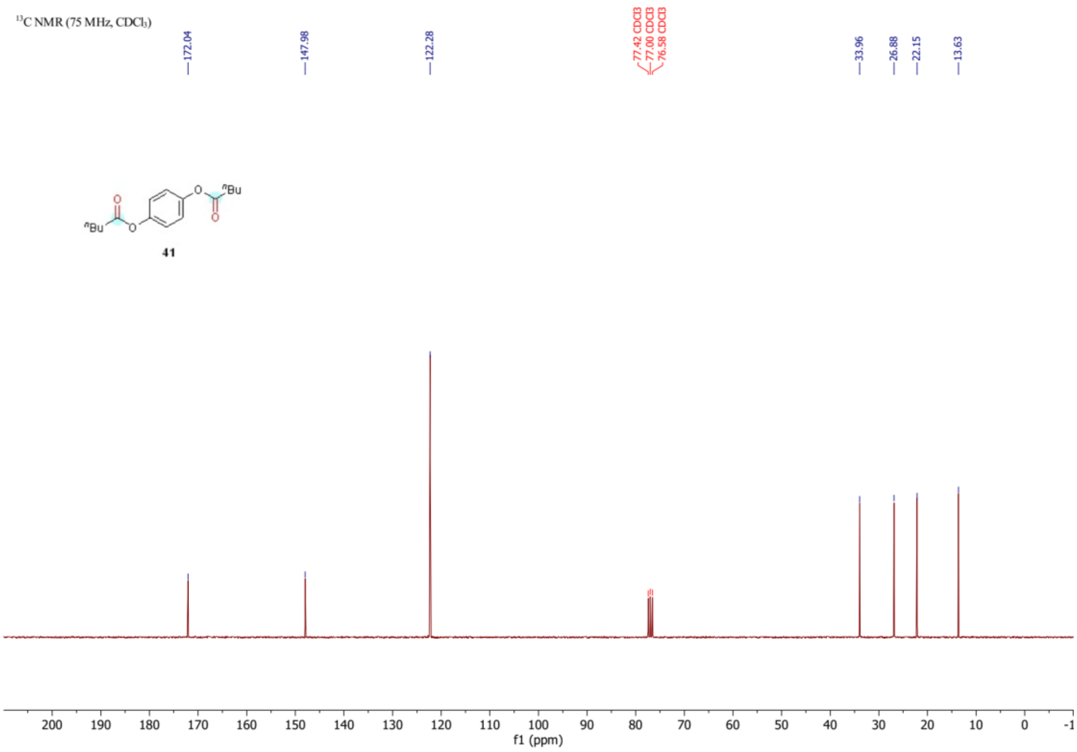


^{13}C NMR (75 MHz, CDCl$_3$)
171.92
148.19
131.14
130.61
128.78
125.69
122.36
77.42 CDCl3
77.00 CDCl3
76.58 CDCl3
33.67
26.65
21.93
13.61
40
f1 (ppm)

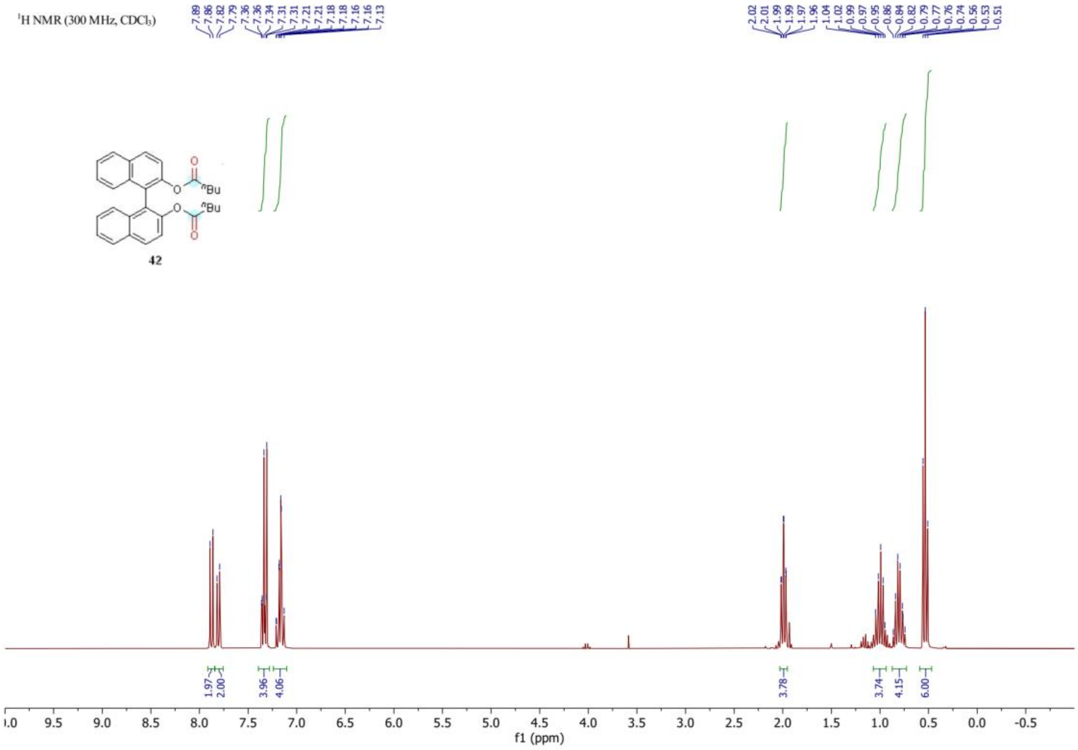
^{1}H NMR (300 MHz, $CDCl_3$)



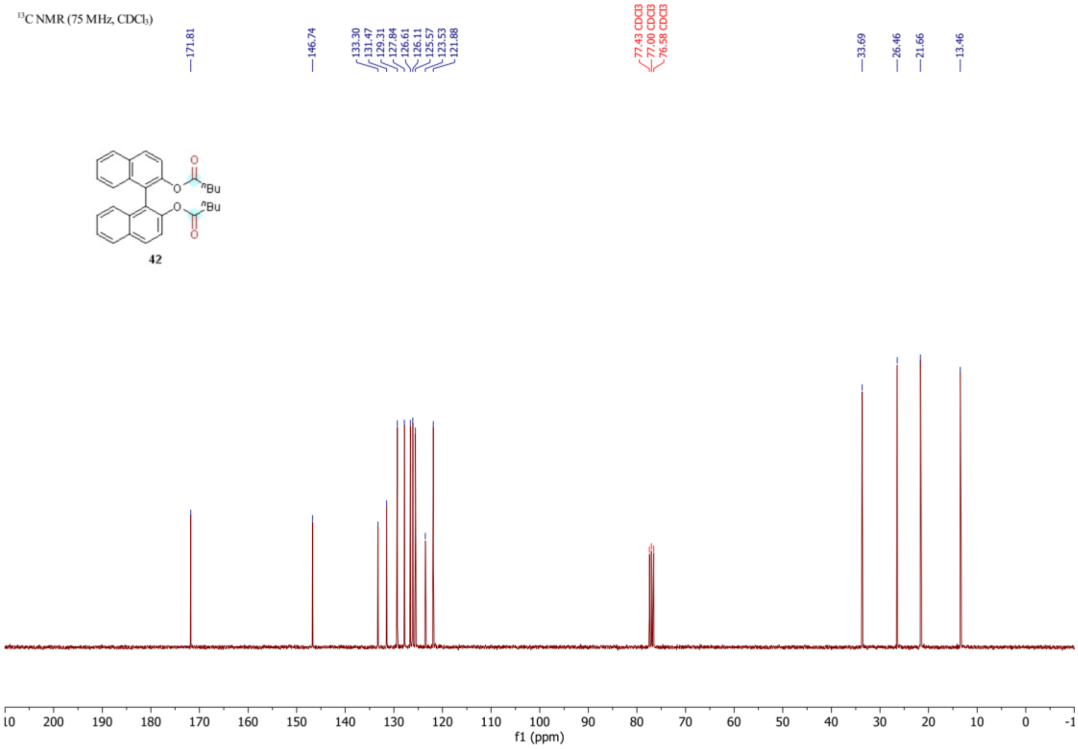
^{13}C NMR (75 MHz, $CDCl_3$)

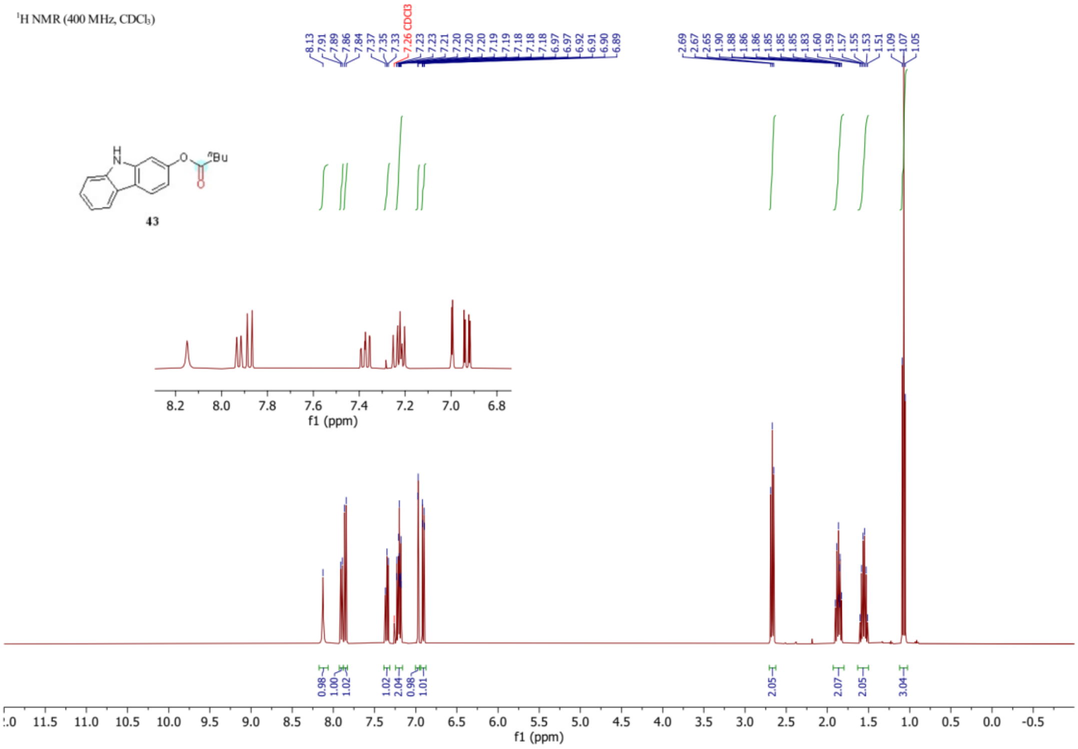


^{1}H NMR (300 MHz, $CDCl_3$)

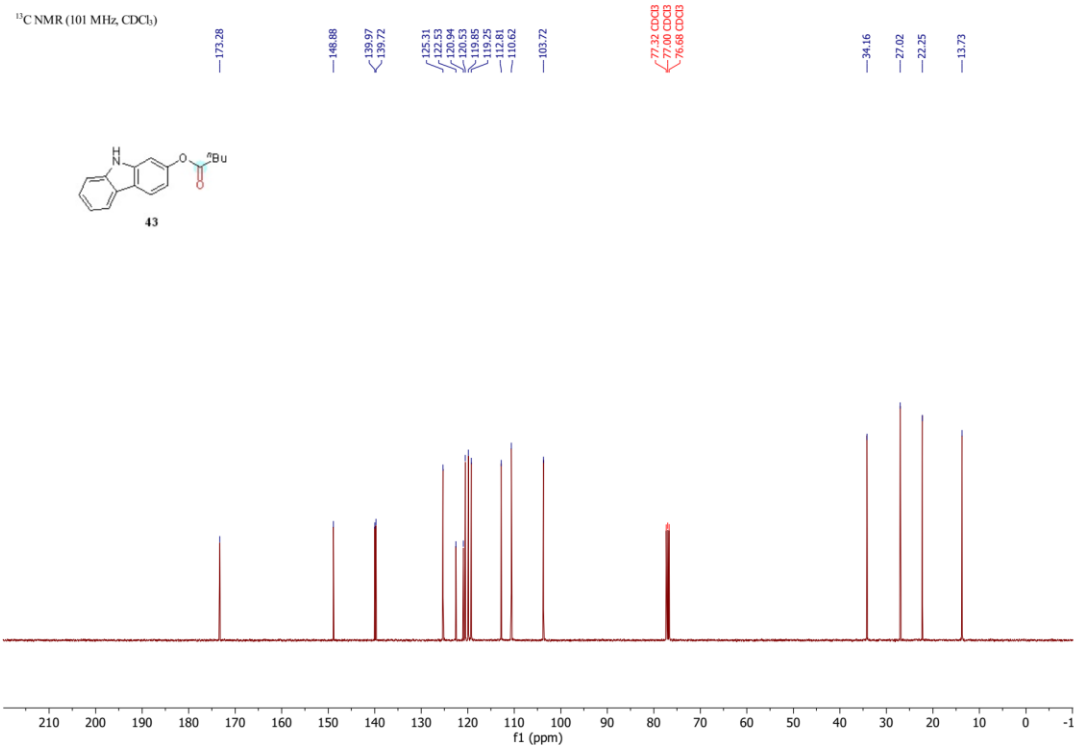


^{13}C NMR (75 MHz, $CDCl_3$)



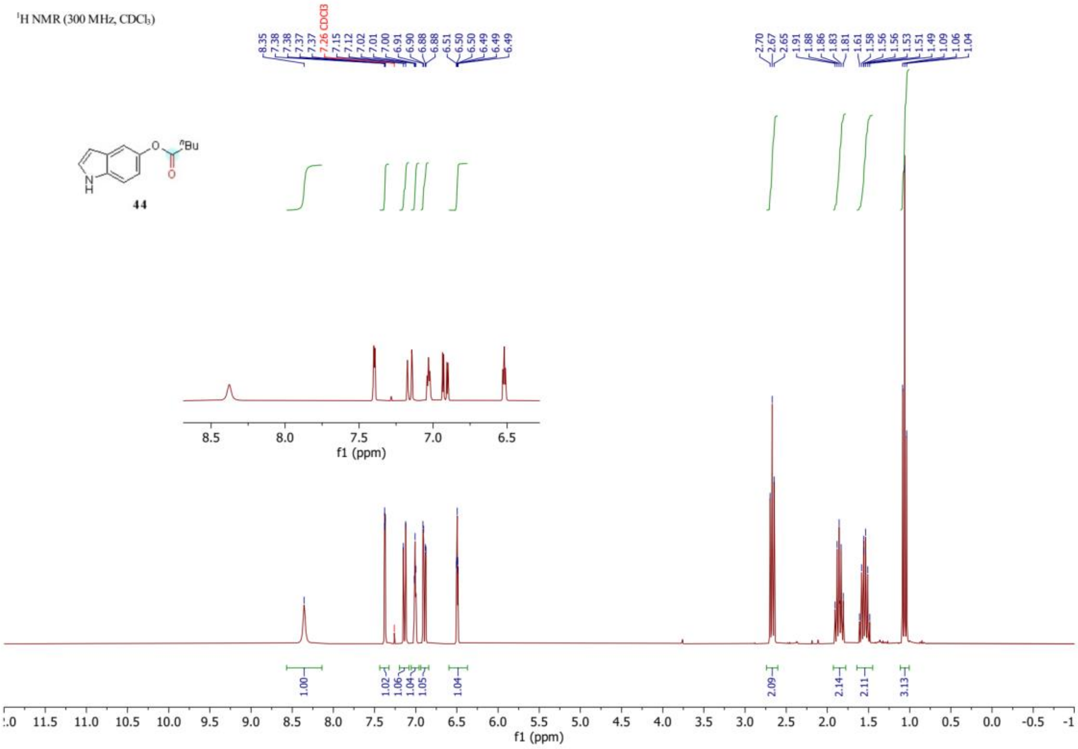


1H NMR (400 MHz, CDCl3)
8.13 7.91 7.89 7.86 7.84 7.37 7.35 7.33 7.26 CDCl3 7.23 7.23 7.21 7.20 7.20 7.19 7.19 7.18 7.18 7.18 6.97 6.97 6.92 6.91 6.90 6.89
2.69 2.67 2.65 1.90 1.88 1.86 1.86 1.85 1.85 1.85 1.83 1.60 1.59 1.57 1.55 1.53 1.51 1.09 1.07 1.05
nBu
43
0.98 1.00 1.02 1.02 2.04 0.98 1.01 2.05 2.07 2.05 3.04
f1 (ppm)

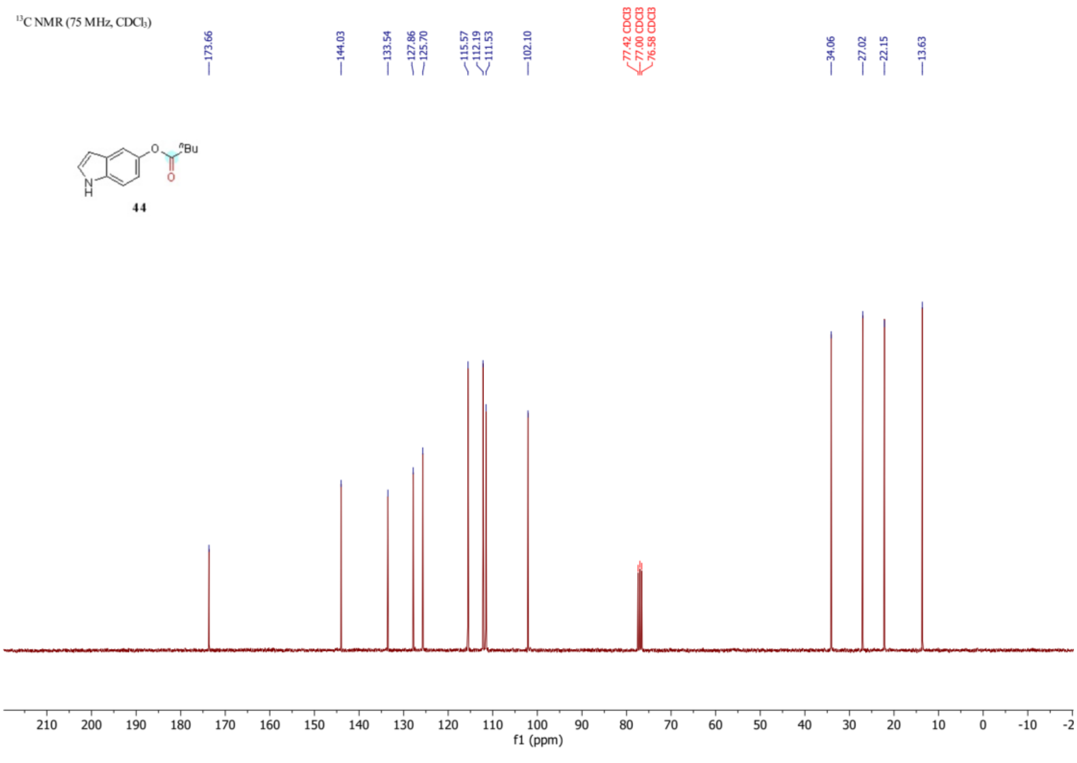


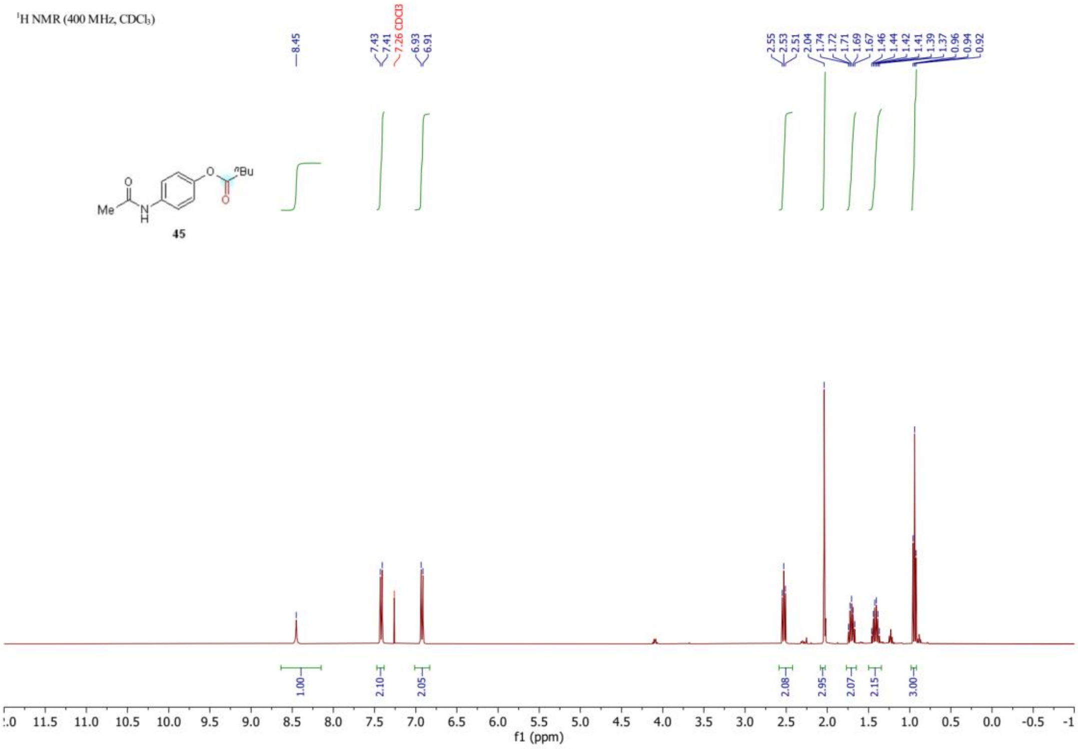
13C NMR (101 MHz, CDCl3)
173.28 148.88 139.97 139.72 125.31 122.53 120.94 120.53 119.85 119.25 112.81 110.62 103.72
77.32 CDCl3 77.00 CDCl3 76.68 CDCl3
34.16 27.02 22.25 13.73
nBu
43
f1 (ppm)

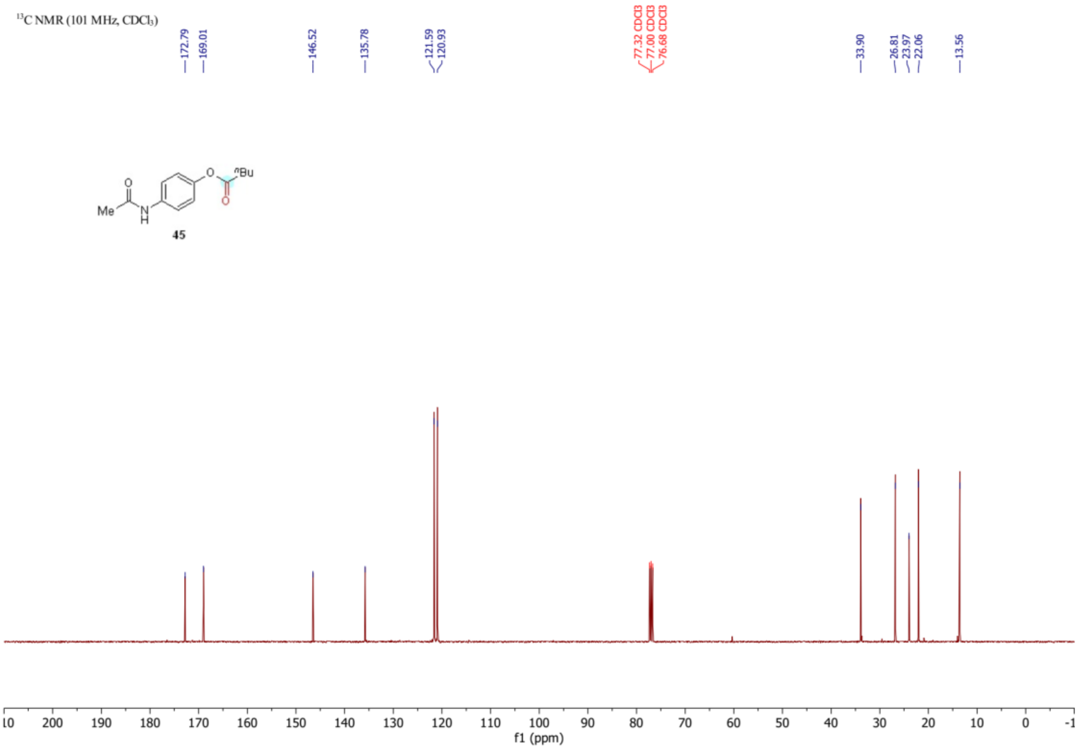
^{1}H NMR (300 MHz, CDCl$_3$)



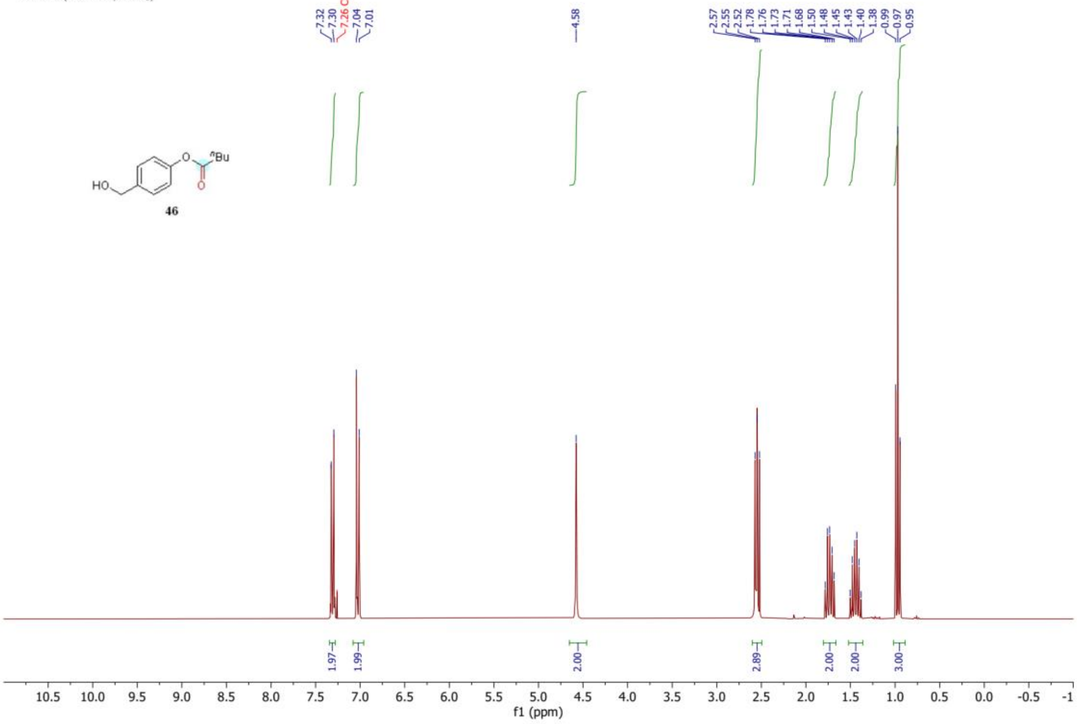
^{13}C NMR (75 MHz, CDCl$_3$)



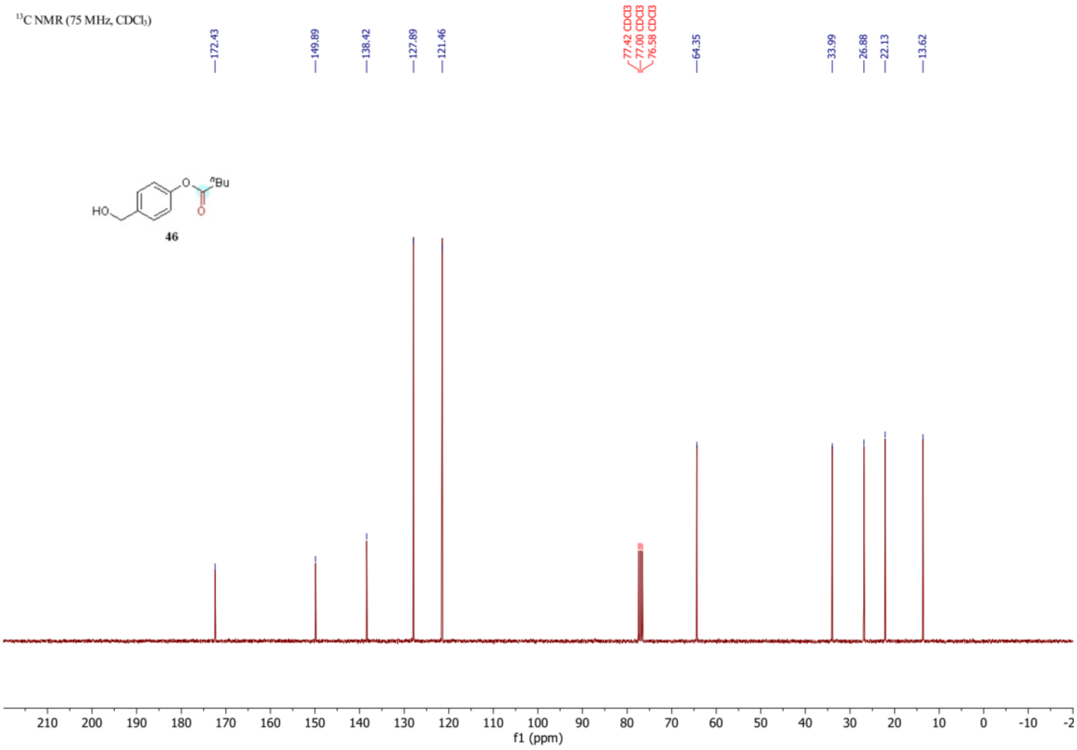

^{1}H NMR (400 MHz, CDCl$_3$)
8.45
7.43
7.41
7.26 CDCl3
6.93
6.91
2.55
2.53
2.51
2.04
1.74
1.72
1.71
1.69
1.67
1.46
1.44
1.42
1.41
1.39
1.37
0.96
0.94
0.92
45
1.00
2.10
2.05
2.08
2.95
2.07
2.15
3.00
f1 (ppm)


^{13}C NMR (101 MHz, CDCl$_3$)
172.79
169.01
146.52
135.78
121.59
120.93
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
33.90
26.81
23.97
22.06
13.56
45
f1 (ppm)

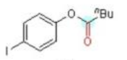
^{1}H NMR (300 MHz, $CDCl_3$)



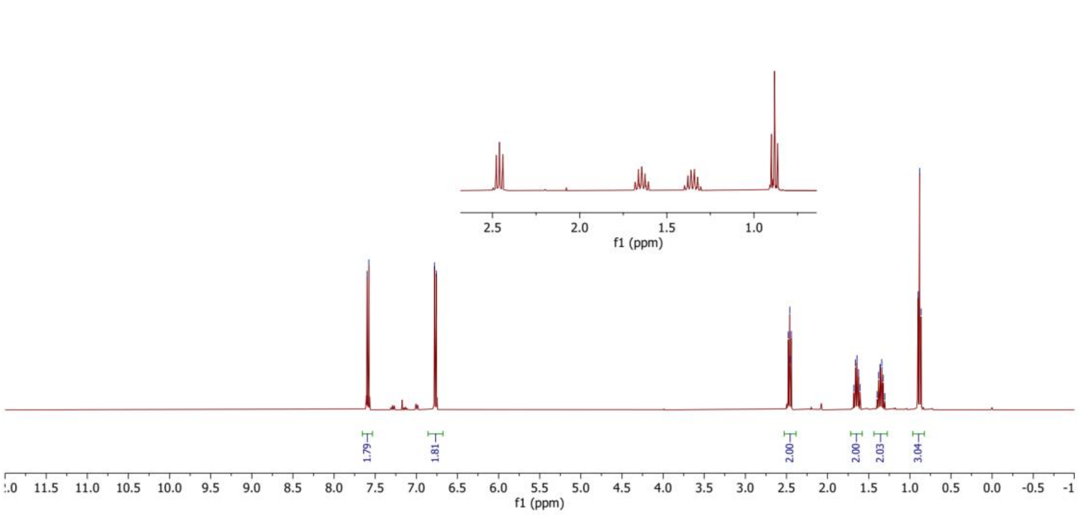
^{13}C NMR (75 MHz, $CDCl_3$)



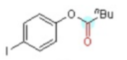
^{1}H NMR (400 MHz, $CDCl_3$)



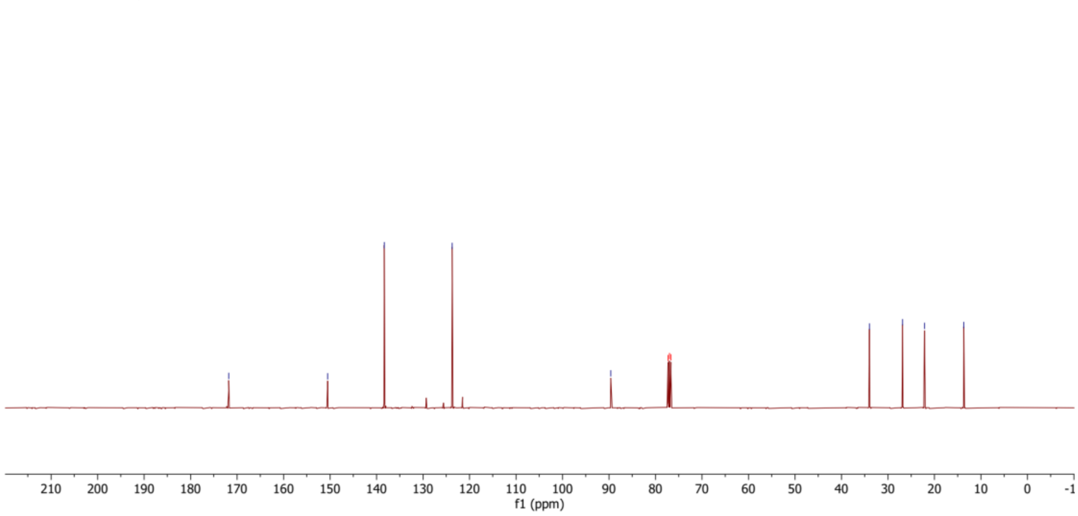
47



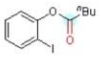
^{13}C NMR (101 MHz, $CDCl_3$)



47



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



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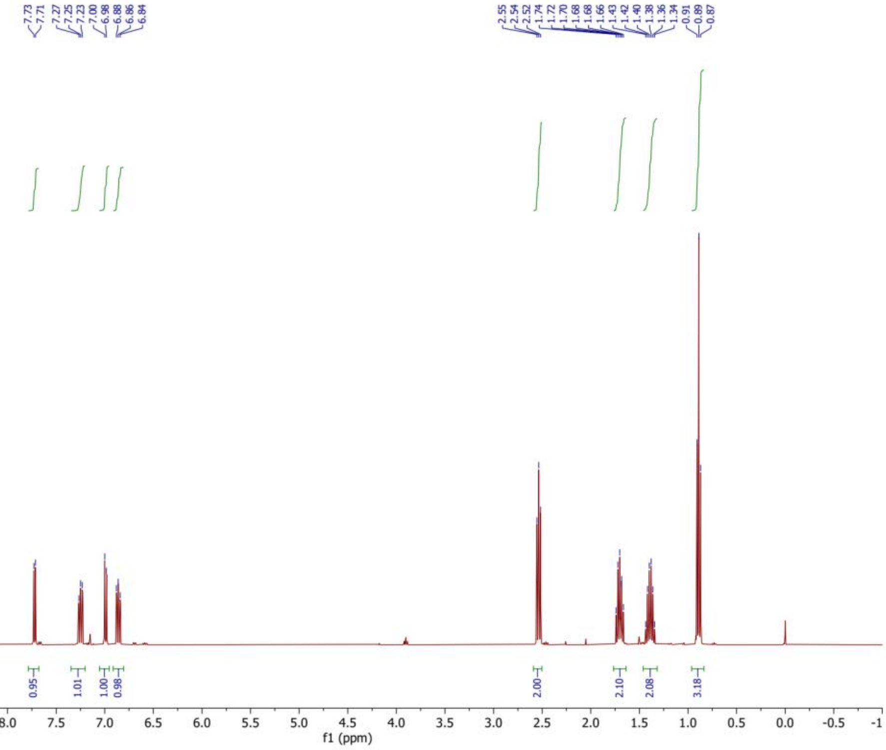
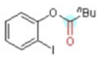
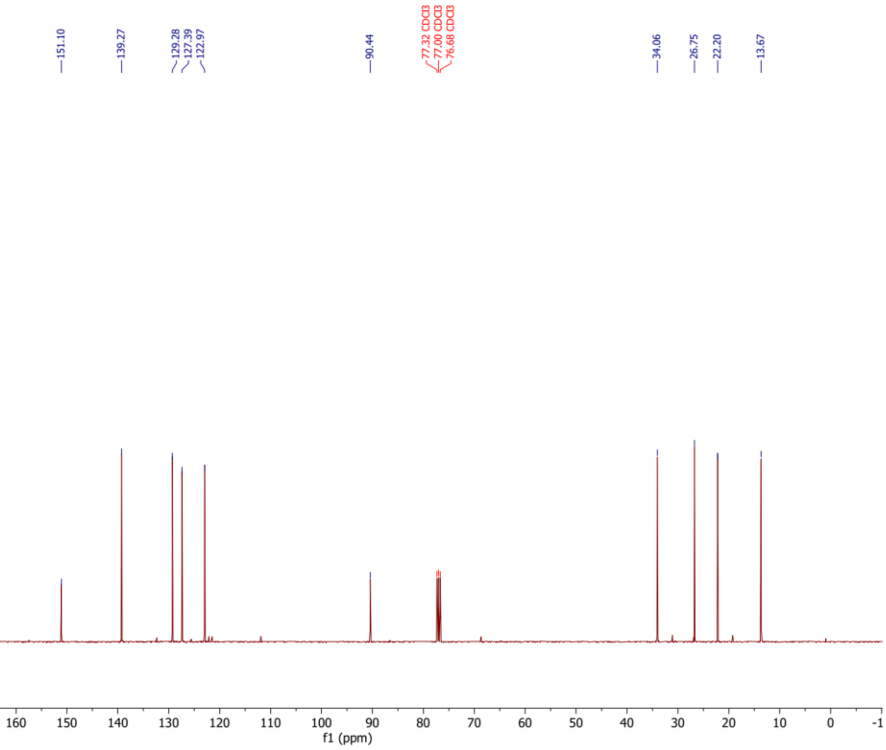
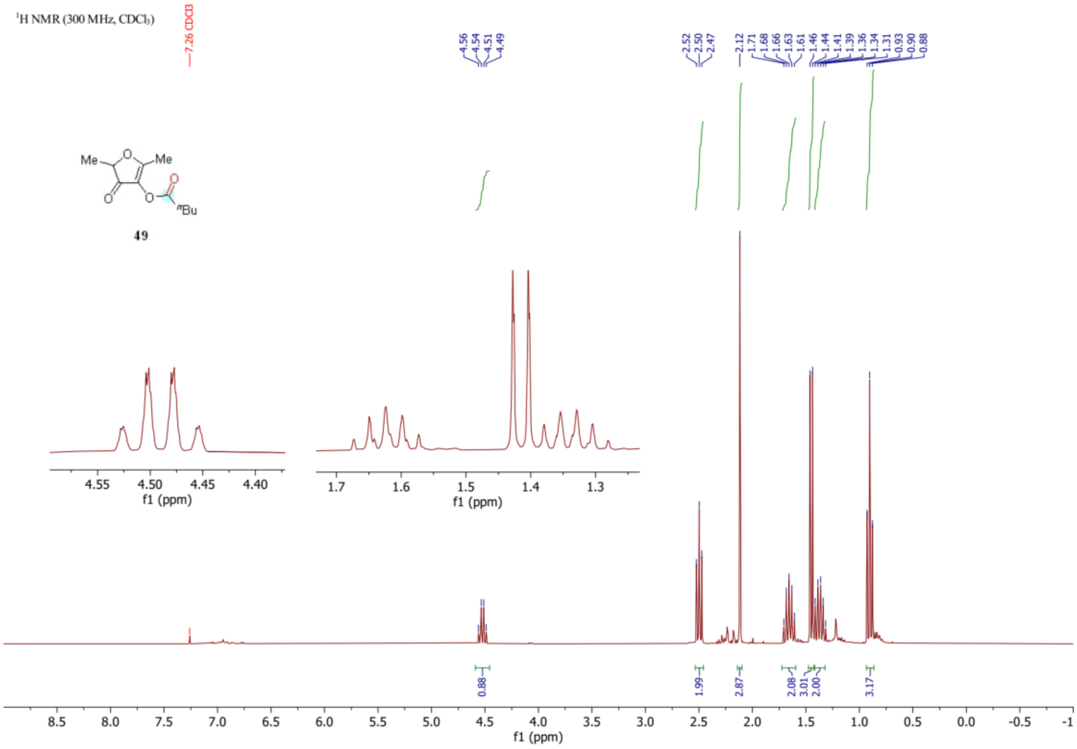


^{13}C NMR (101 MHz, $CDCl_3$)

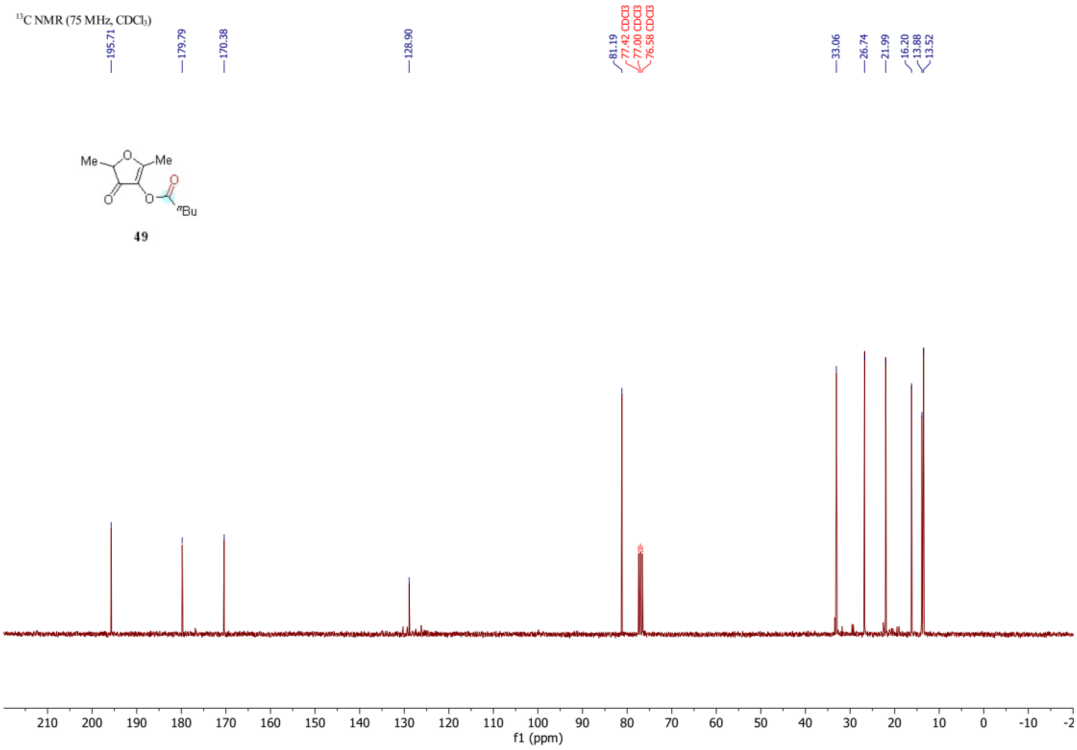


48

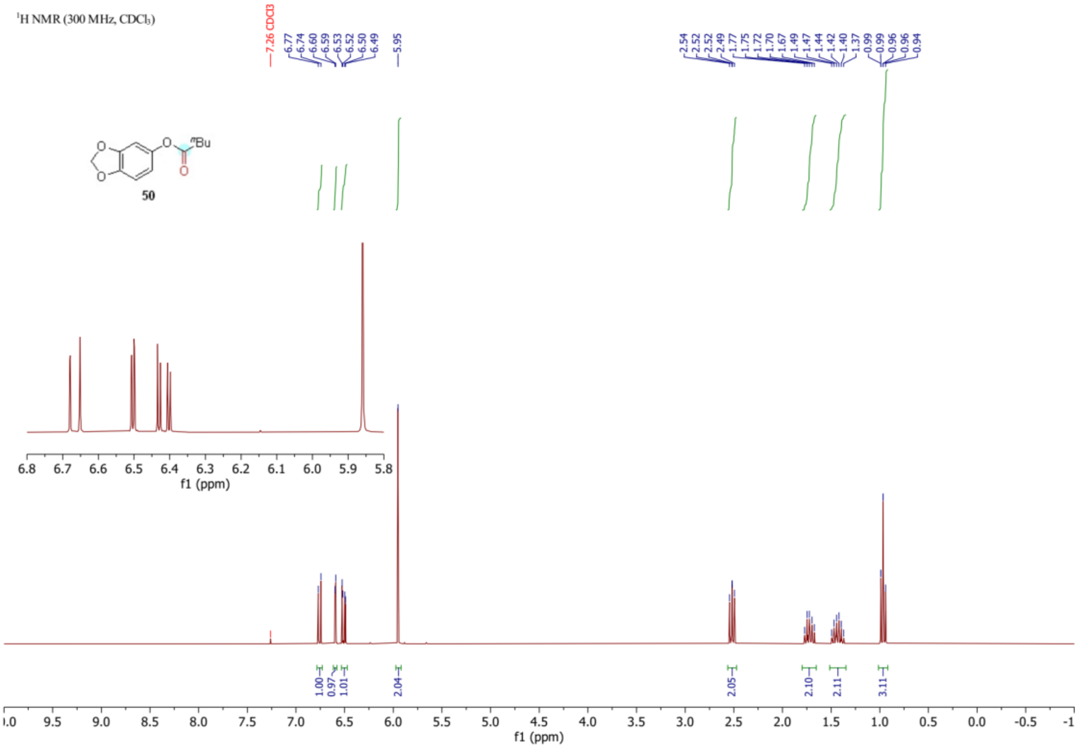


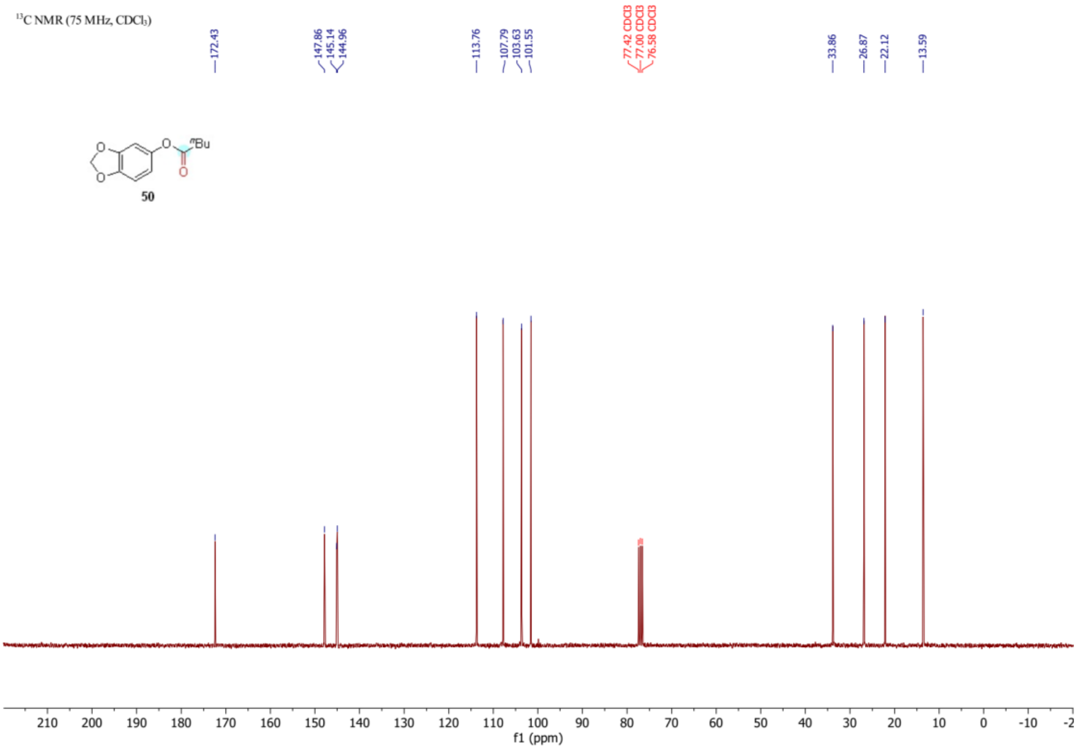


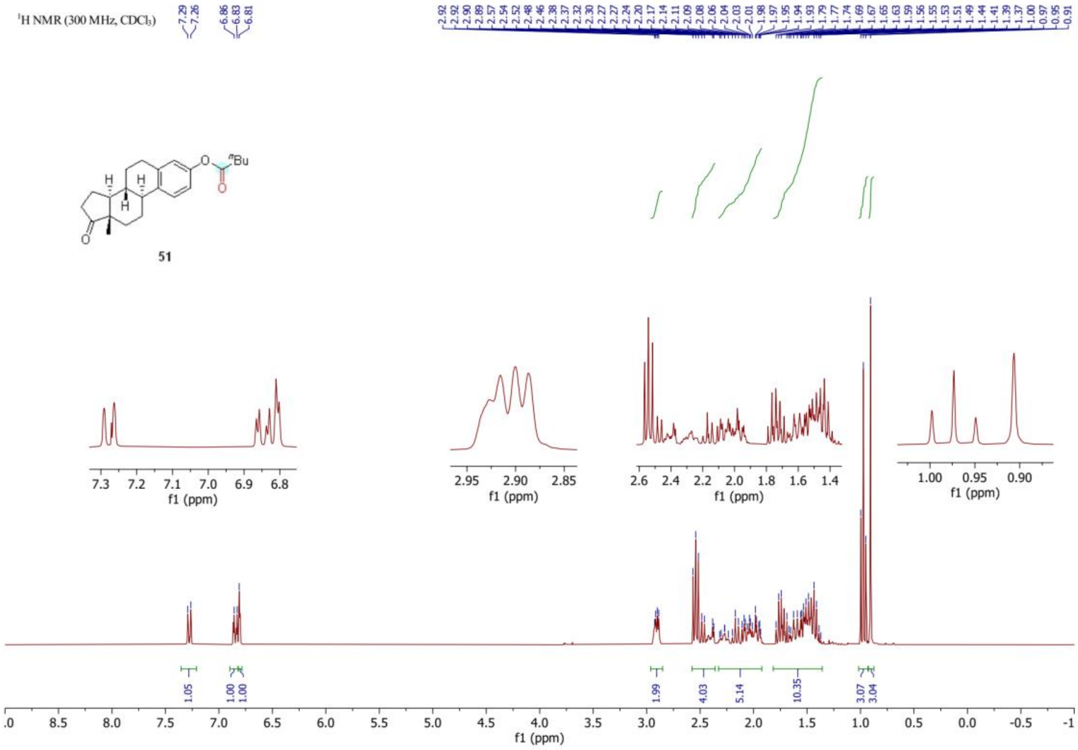
¹H NMR (300 MHz, CDCl₃)
Me
O
Me
O
O
O
ⁿBu
49
f1 (ppm)

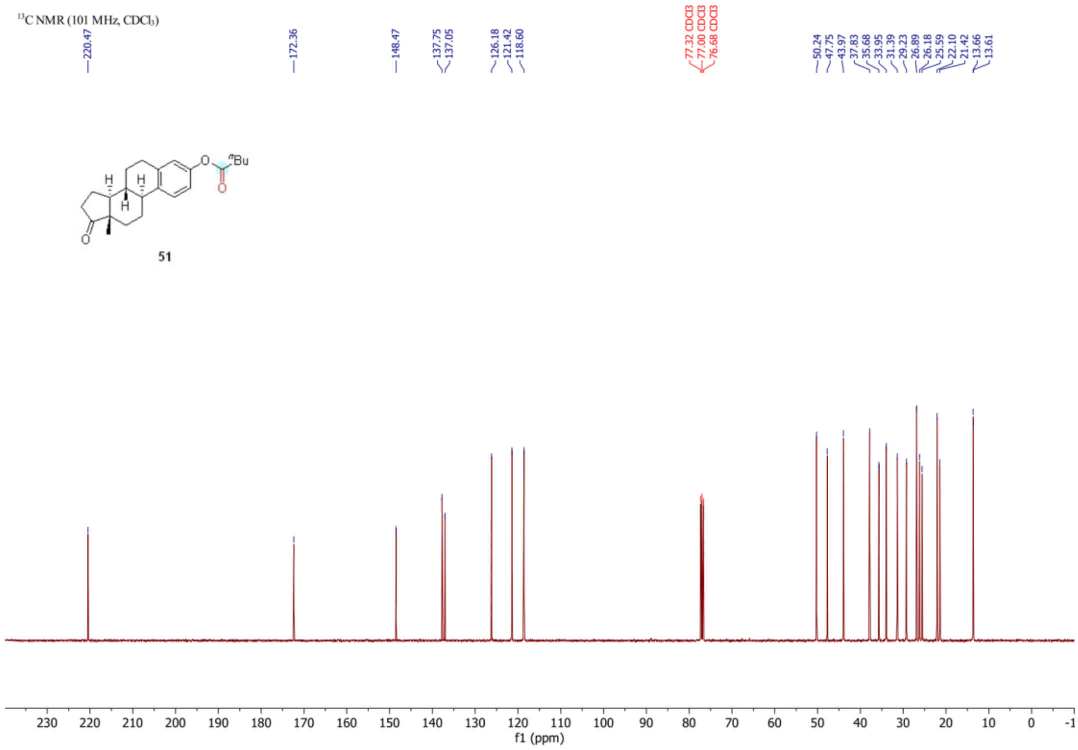


¹³C NMR (75 MHz, CDCl₃)
Me
O
Me
O
O
O
ⁿBu
49
f1 (ppm)

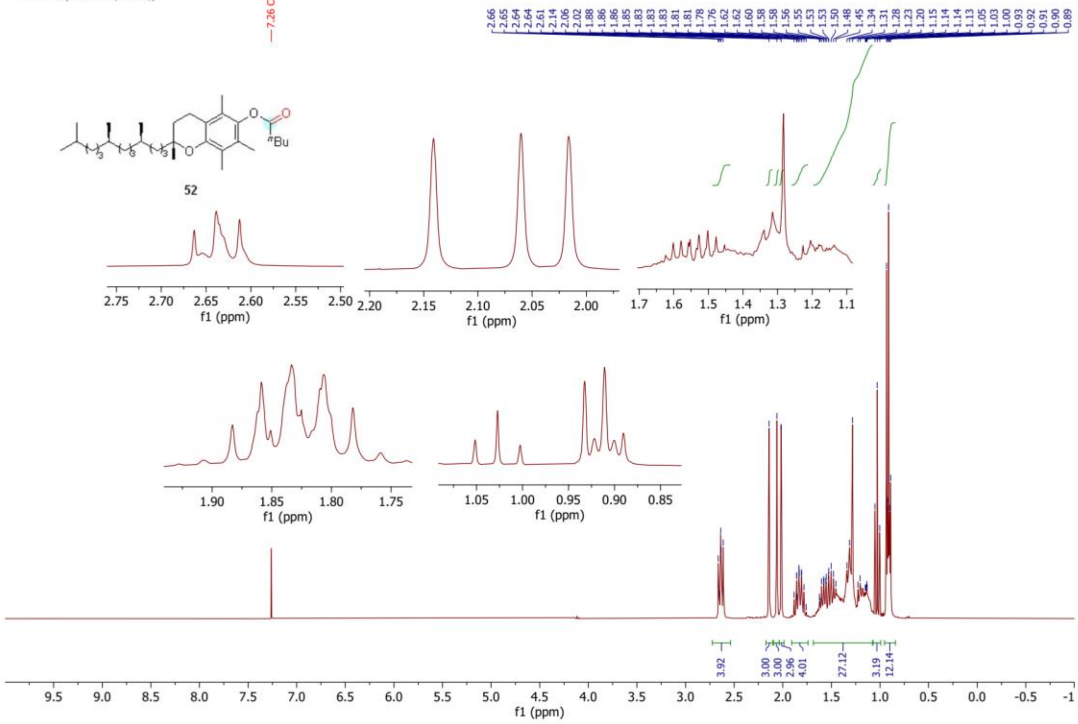

1H NMR (300 MHz, CDCl3)
50
f1 (ppm)


13C NMR (75 MHz, CDCl3)
50
f1 (ppm)

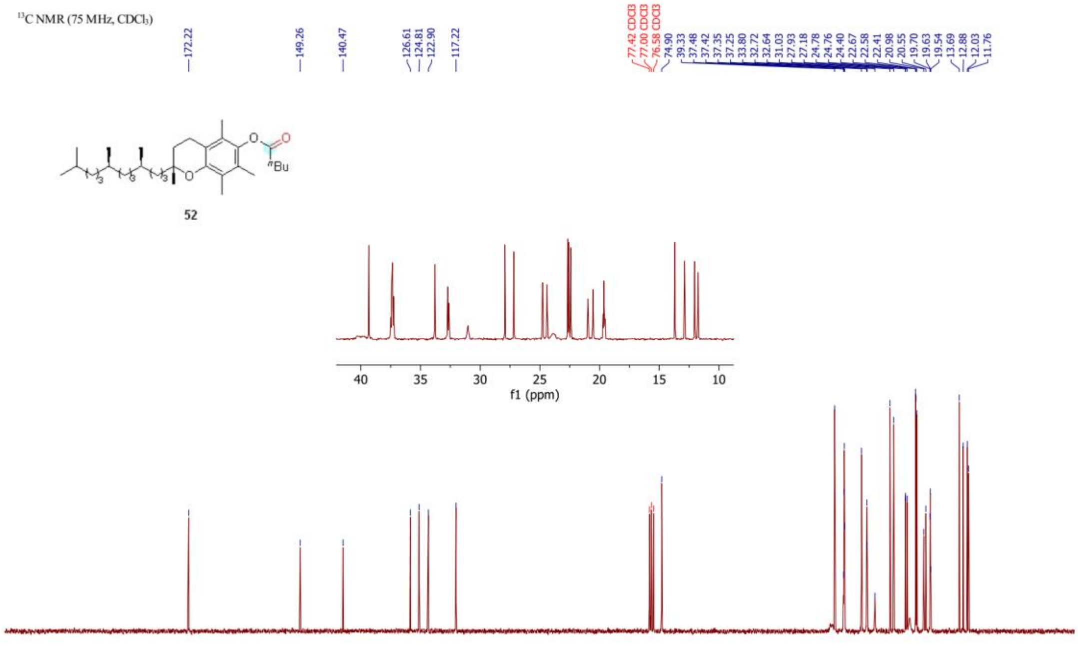

1H NMR (300 MHz, CDCl3)
7.29
7.26
6.86
6.83
6.81
2.92
2.92
2.90
2.89
2.57
2.54
2.52
2.48
2.46
2.38
2.37
2.32
2.30
2.27
2.27
2.24
2.20
2.17
2.14
2.11
2.09
2.08
2.06
2.04
2.03
2.01
1.98
1.97
1.95
1.94
1.93
1.77
1.74
1.69
1.67
1.65
1.63
1.59
1.56
1.55
1.53
1.51
1.49
1.44
1.41
1.39
1.37
1.00
0.97
0.95
0.91
nBu
51
1.05
1.00
1.00
1.99
4.03
5.14
10.35
3.07
3.04
f1 (ppm)

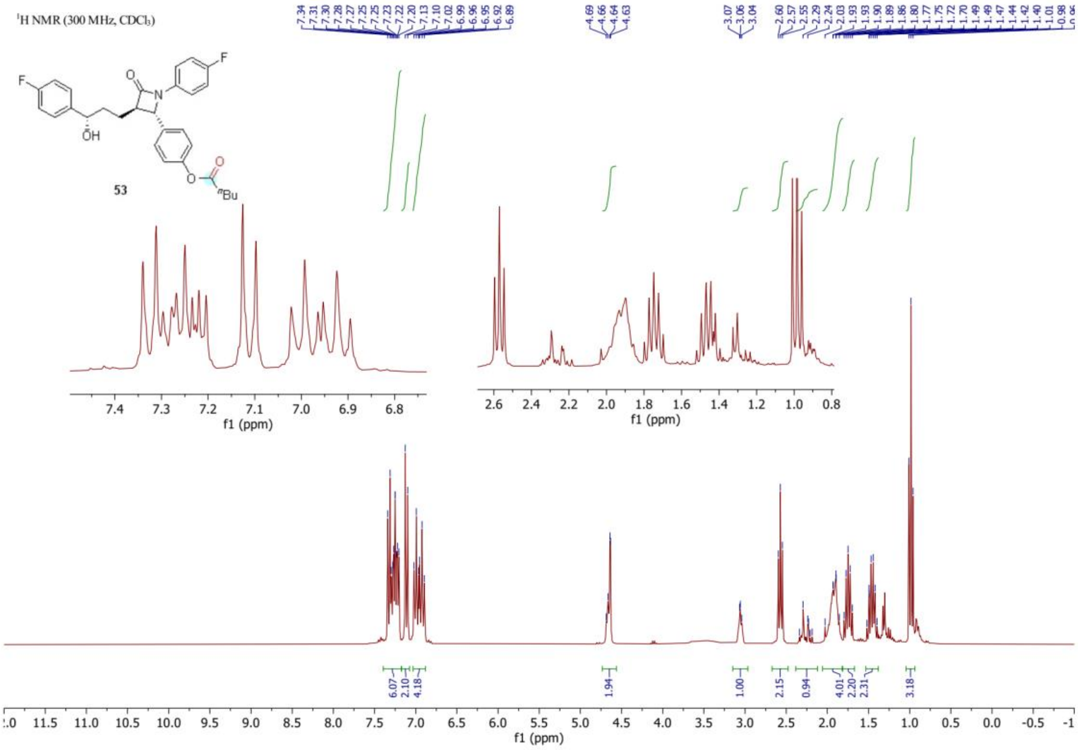

13C NMR (101 MHz, CDCl3)
220.47
172.36
148.47
137.75
137.05
126.18
121.42
118.60
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
50.24
47.75
43.97
37.83
35.68
33.95
31.39
29.23
26.89
26.18
25.59
22.10
21.42
13.66
13.61
nBu
51
f1 (ppm)

^{1}H NMR (300 MHz, $CDCl_3$)

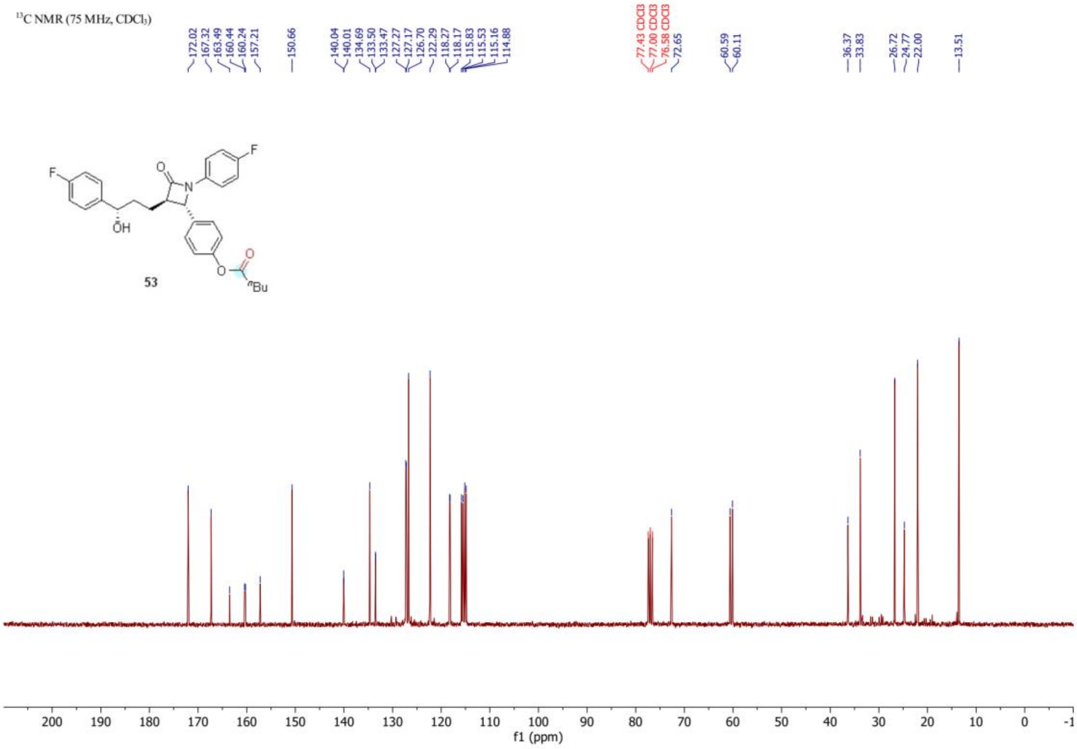


^{13}C NMR (75 MHz, $CDCl_3$)



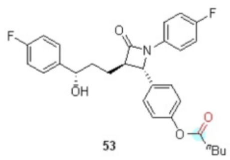


1H NMR (300 MHz, CDCl3)
53
nBu
f1 (ppm)

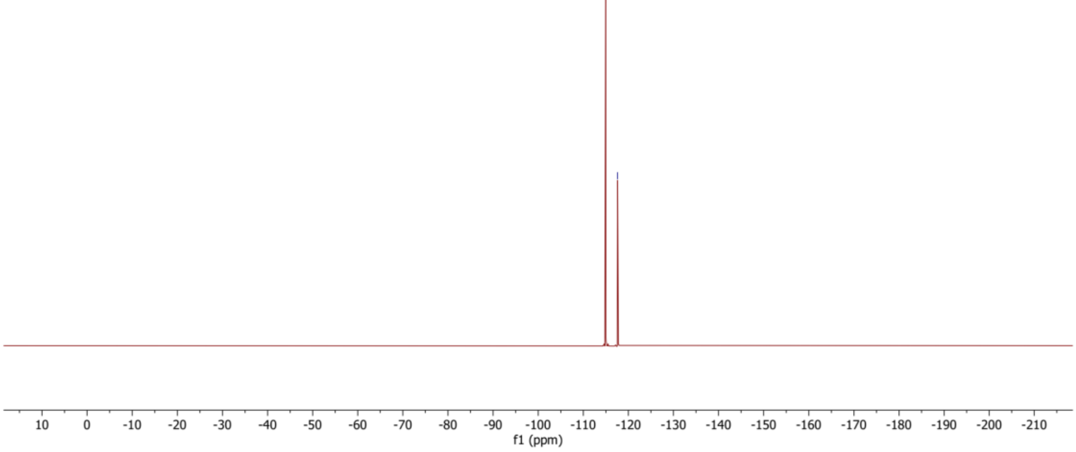


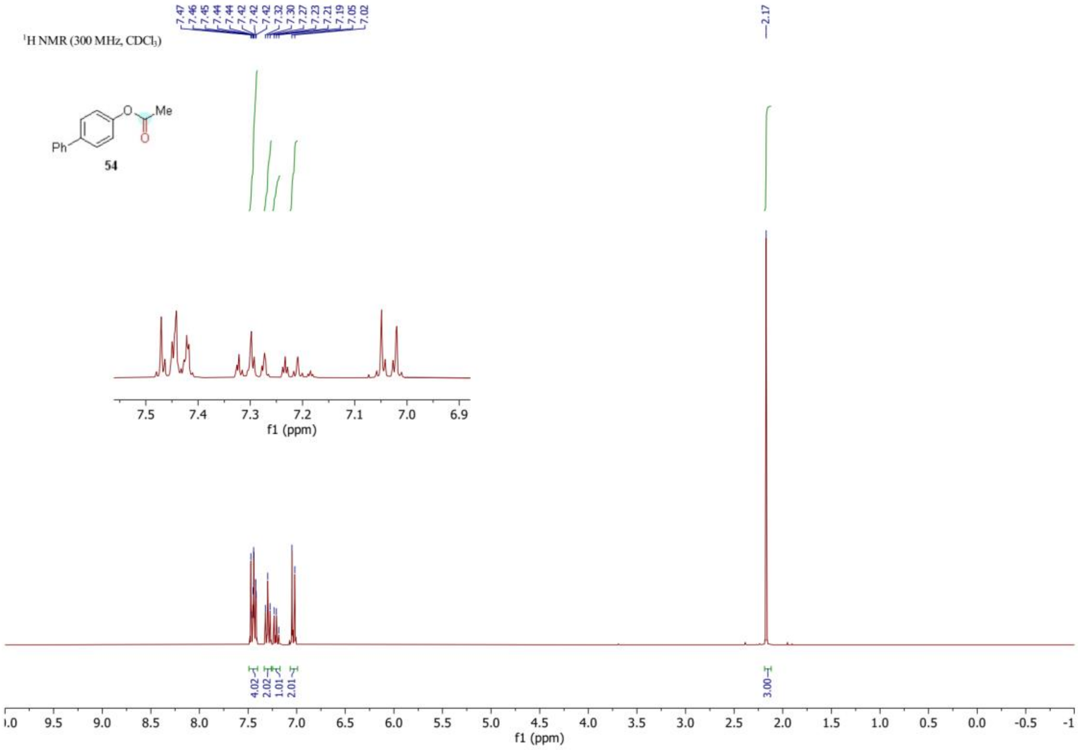
13C NMR (75 MHz, CDCl3)
53
nBu
f1 (ppm)

^{19}F NMR (282 MHz, $CDCl_3$)

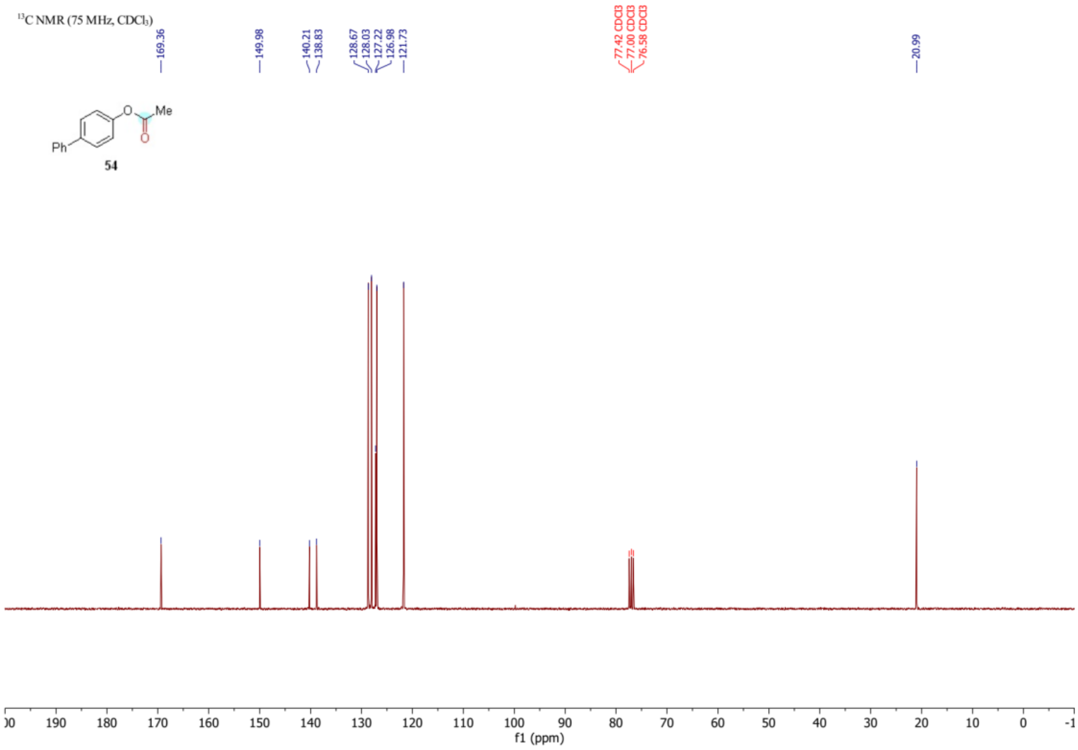


53

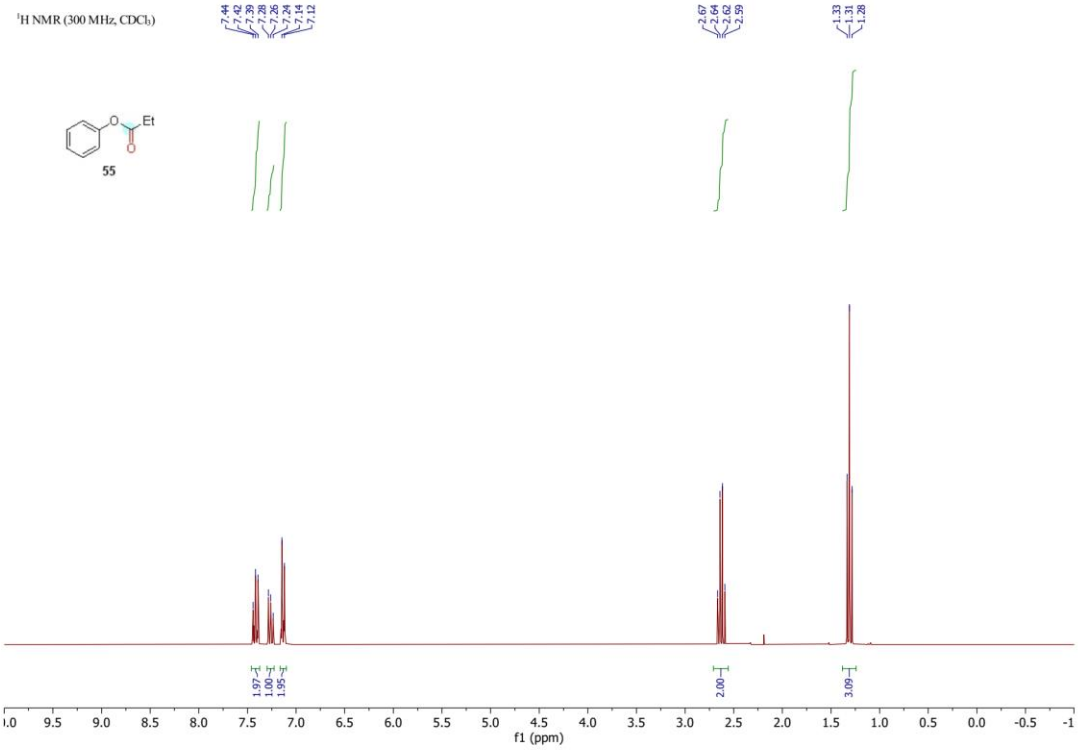




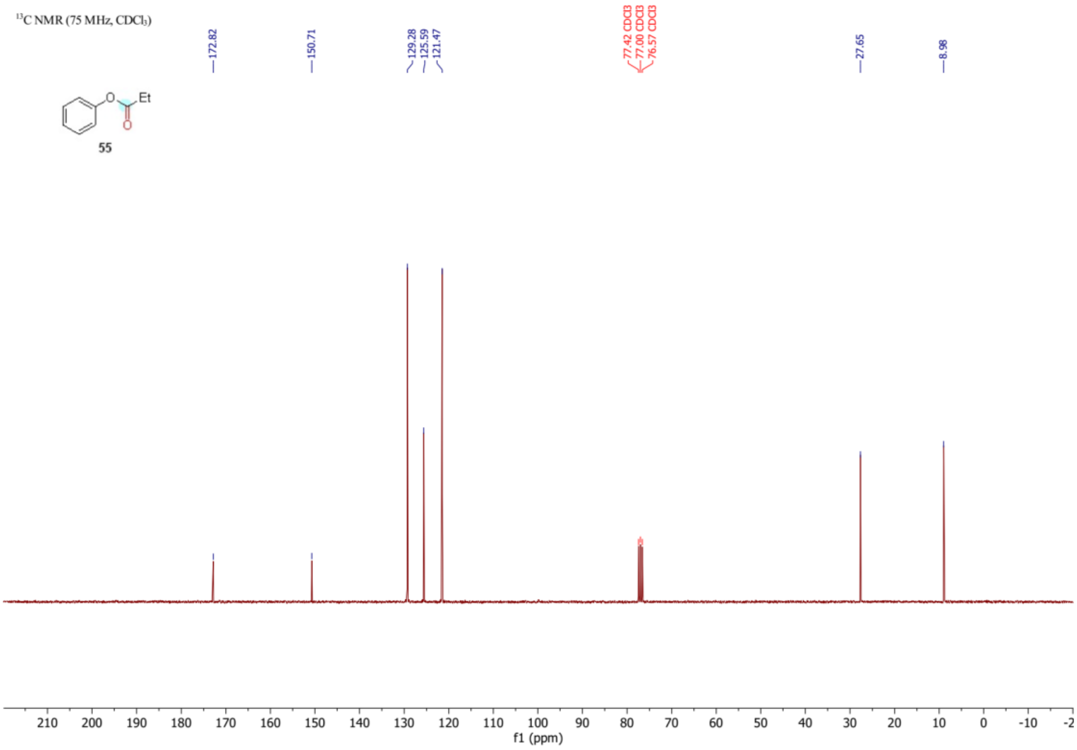
1H NMR (300 MHz, CDCl3)
Me
O
O
Ph
54
7.47
7.46
7.45
7.44
7.44
7.42
7.42
7.42
7.32
7.30
7.27
7.23
7.21
7.19
7.05
7.02
2.17
4.02
2.02
1.01
2.01
3.00
f1 (ppm)



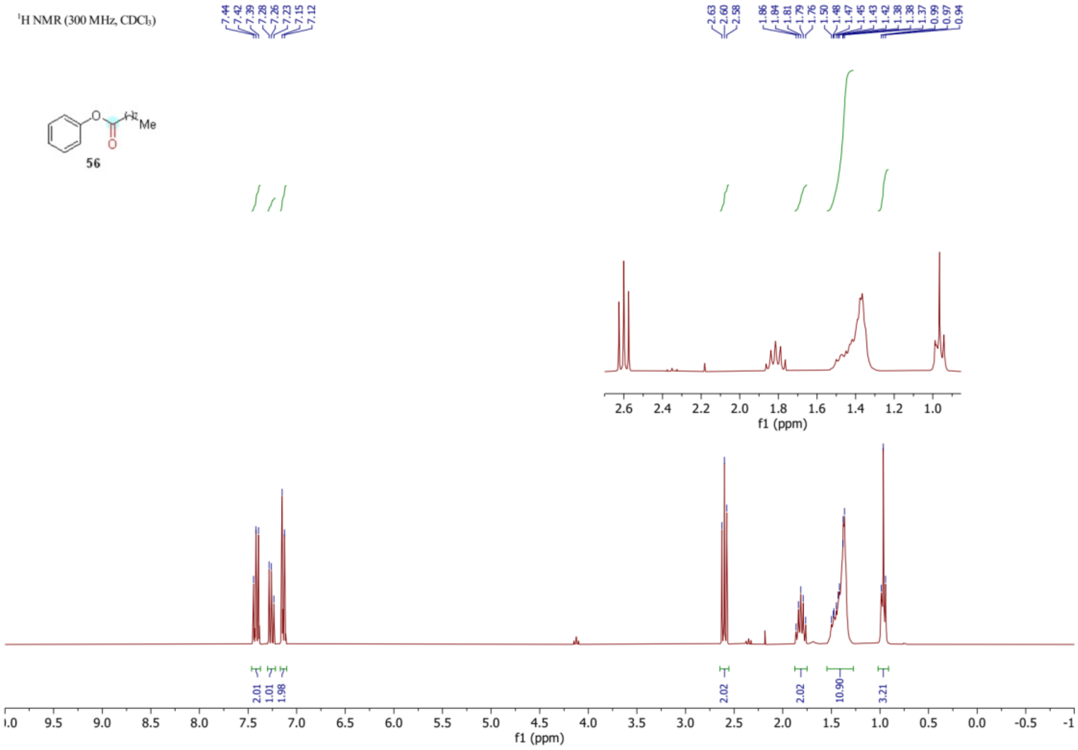
13C NMR (75 MHz, CDCl3)
Me
O
O
Ph
54
169.36
149.98
140.21
138.83
128.67
128.03
127.22
126.98
121.73
77.42 CDCl3
77.00 CDCl3
76.58 CDCl3
20.99
f1 (ppm)



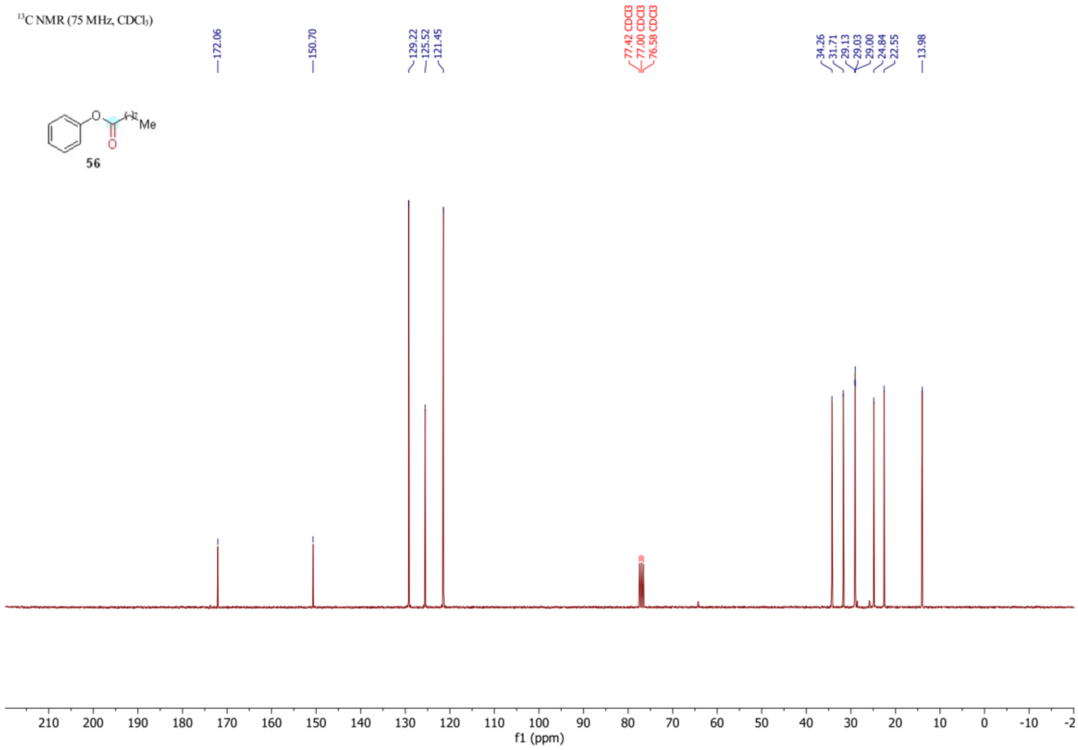
^{1}H NMR (300 MHz, CDCl$_3$)
55



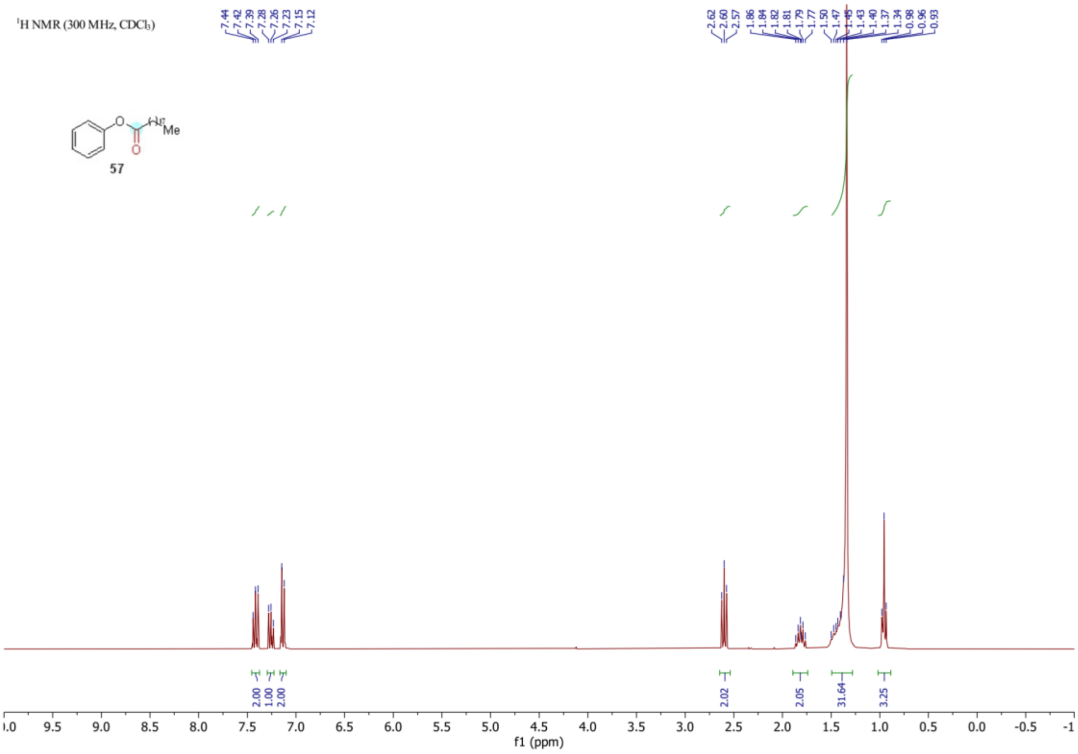
^{13}C NMR (75 MHz, CDCl$_3$)
55

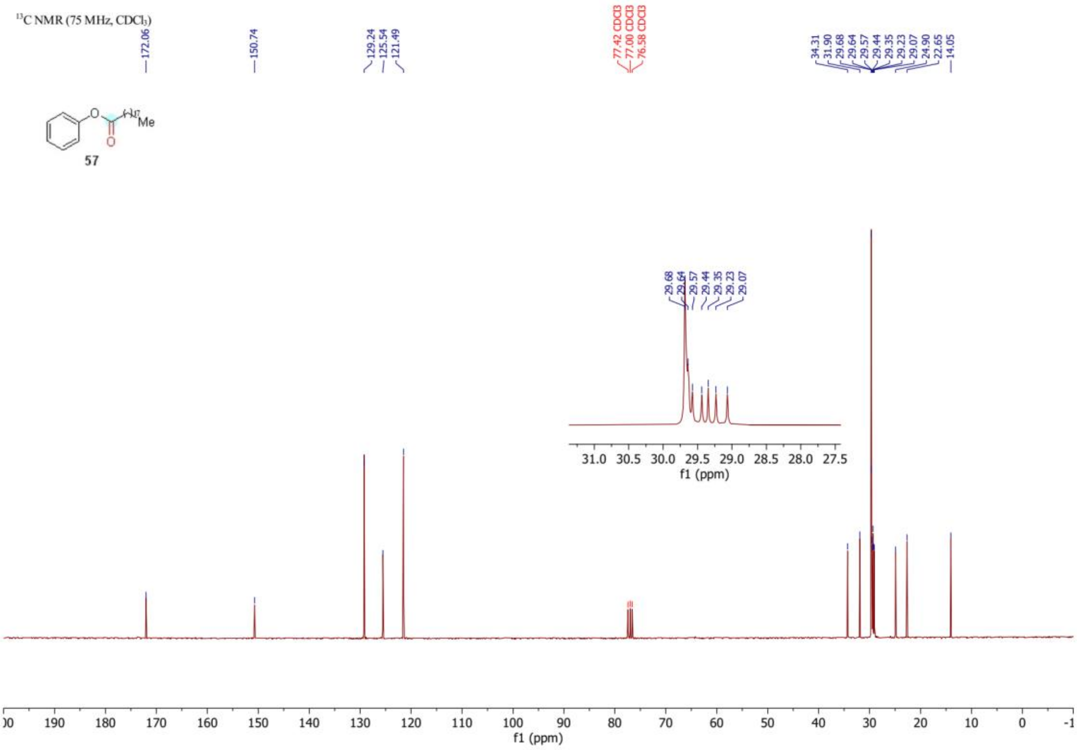


[1]H NMR (300 MHz, CDCl3)
56
f1 (ppm)

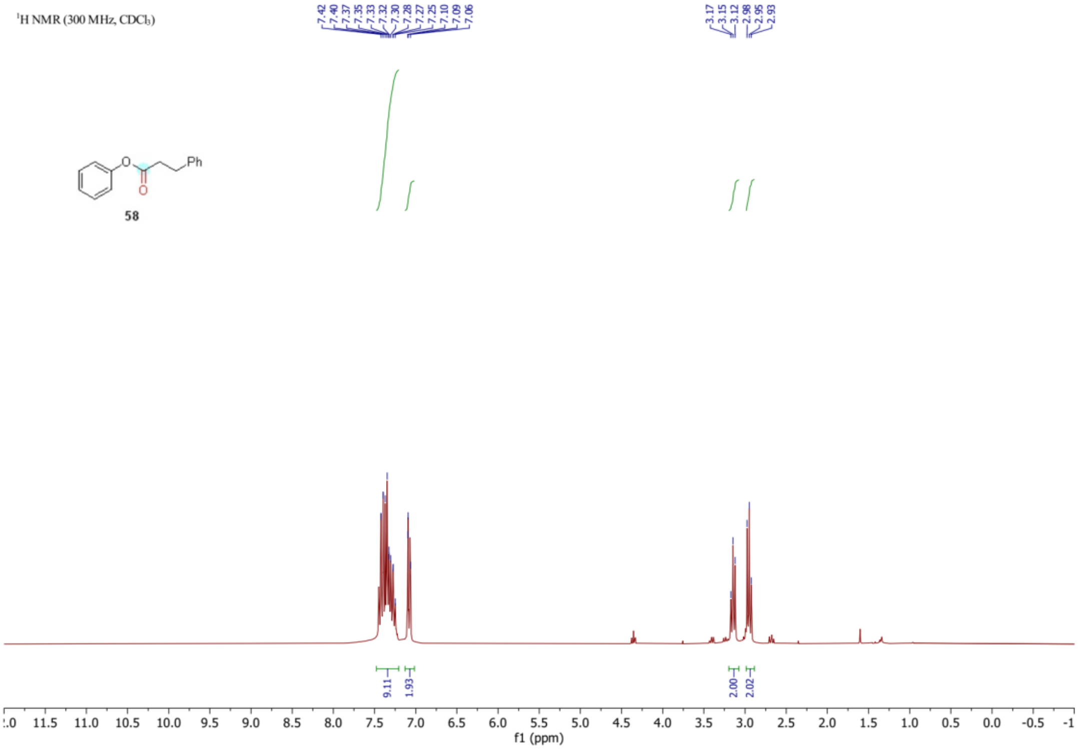


[13]C NMR (75 MHz, CDCl3)
56
f1 (ppm)

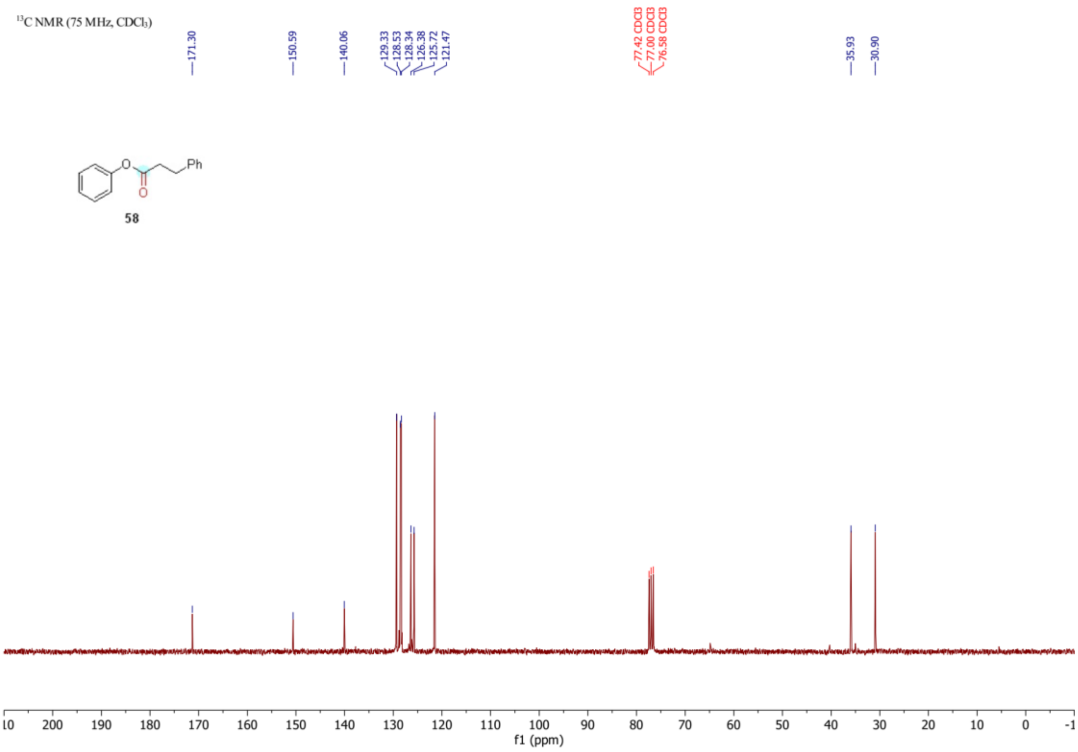

1H NMR (300 MHz, CDCl3)
57
Me
O
f1 (ppm)


13C NMR (75 MHz, CDCl3)
57
Me
O
172.06
150.74
129.24
125.54
121.49
77.42 CDCl3
77.00 CDCl3
76.58 CDCl3
34.31
31.90
29.68
29.64
29.57
29.44
29.35
29.23
29.07
24.90
22.65
14.05
f1 (ppm)

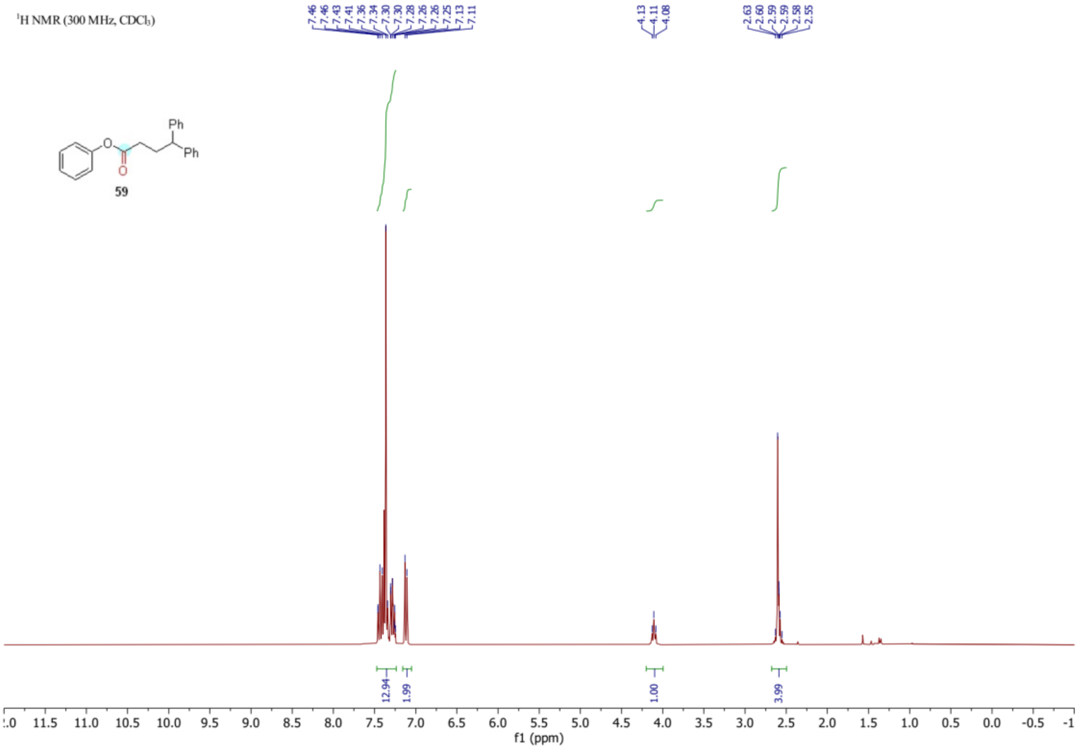
^1H NMR (300 MHz, $CDCl_3$)



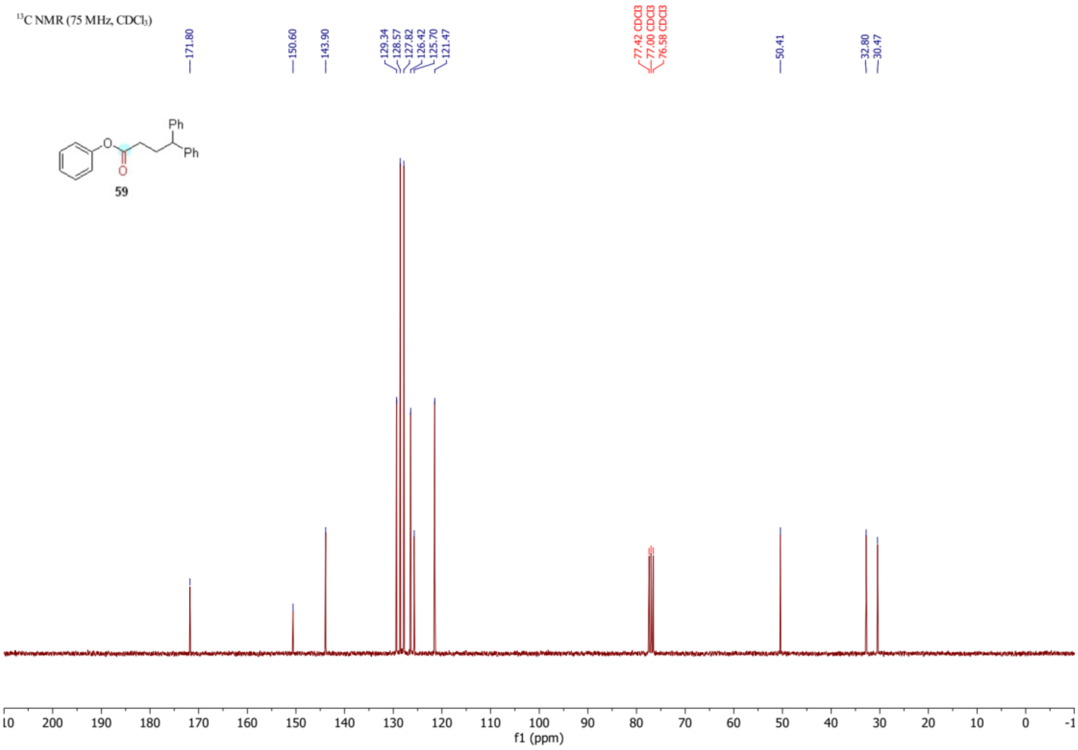
^{13}C NMR (75 MHz, $CDCl_3$)



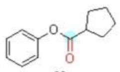
[1]H NMR (300 MHz, CDCl3)



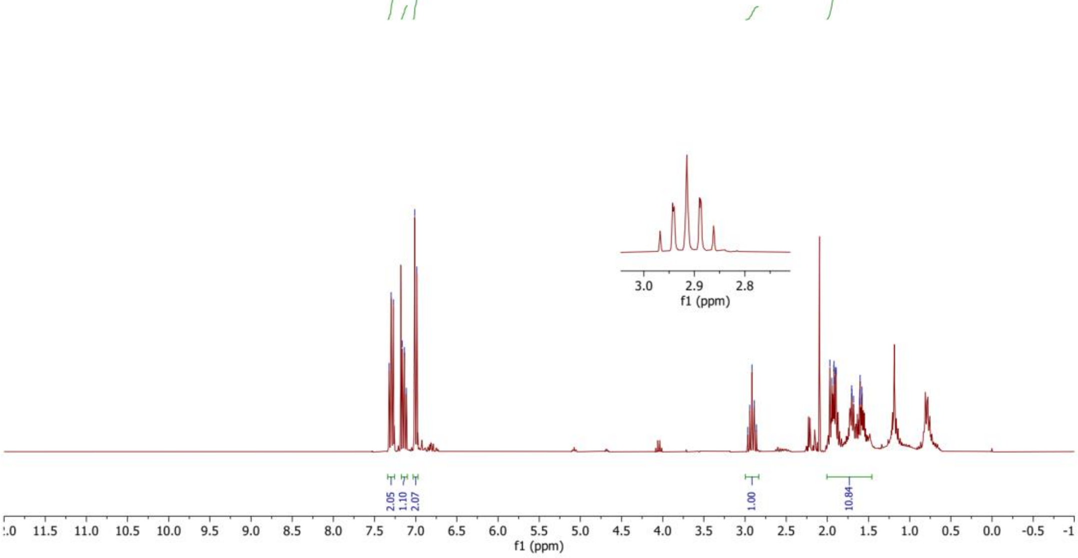
[13]C NMR (75 MHz, CDCl3)



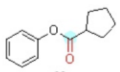
^{1}H NMR (300 MHz, $CDCl_3$)



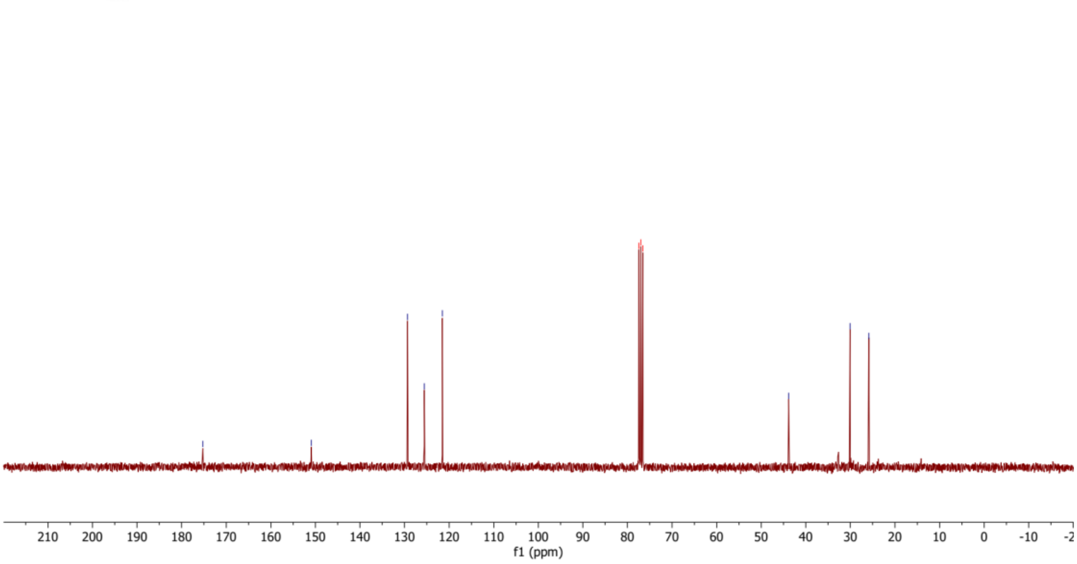
60



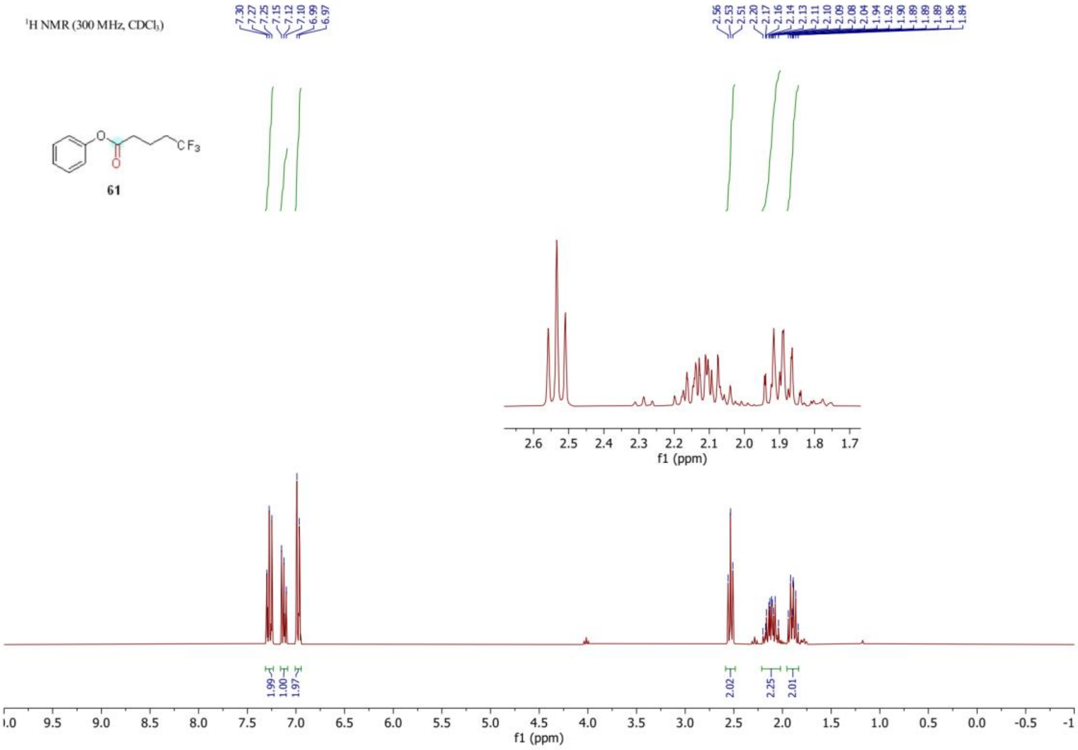
^{13}C NMR (75 MHz, $CDCl_3$)



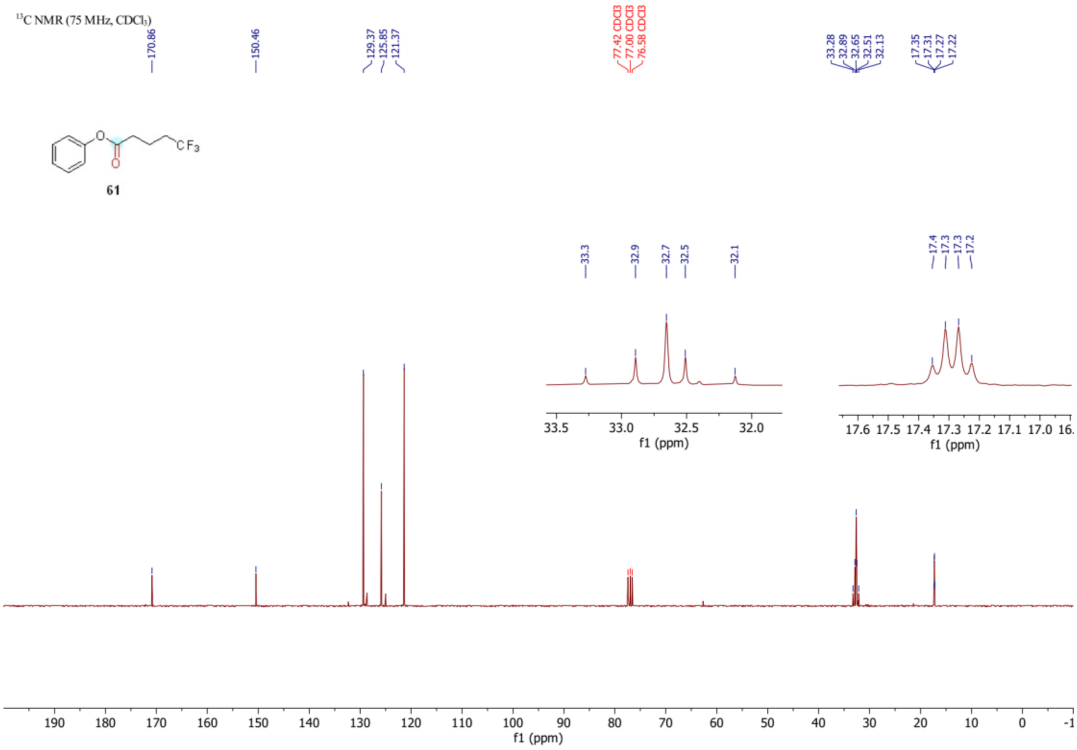
60



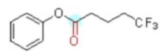
[1]H NMR (300 MHz, $CDCl_3$)



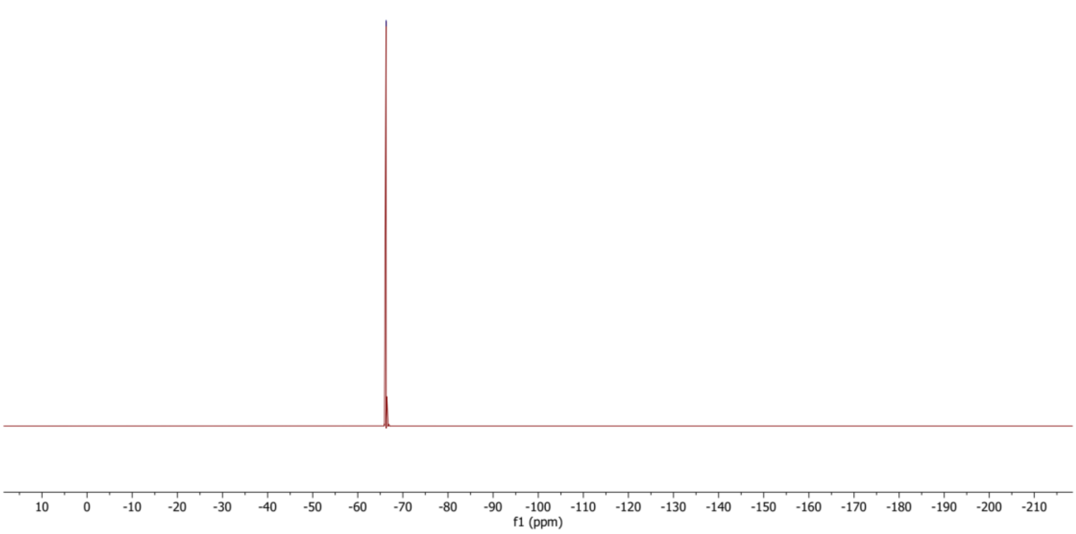
[13]C NMR (75 MHz, $CDCl_3$)



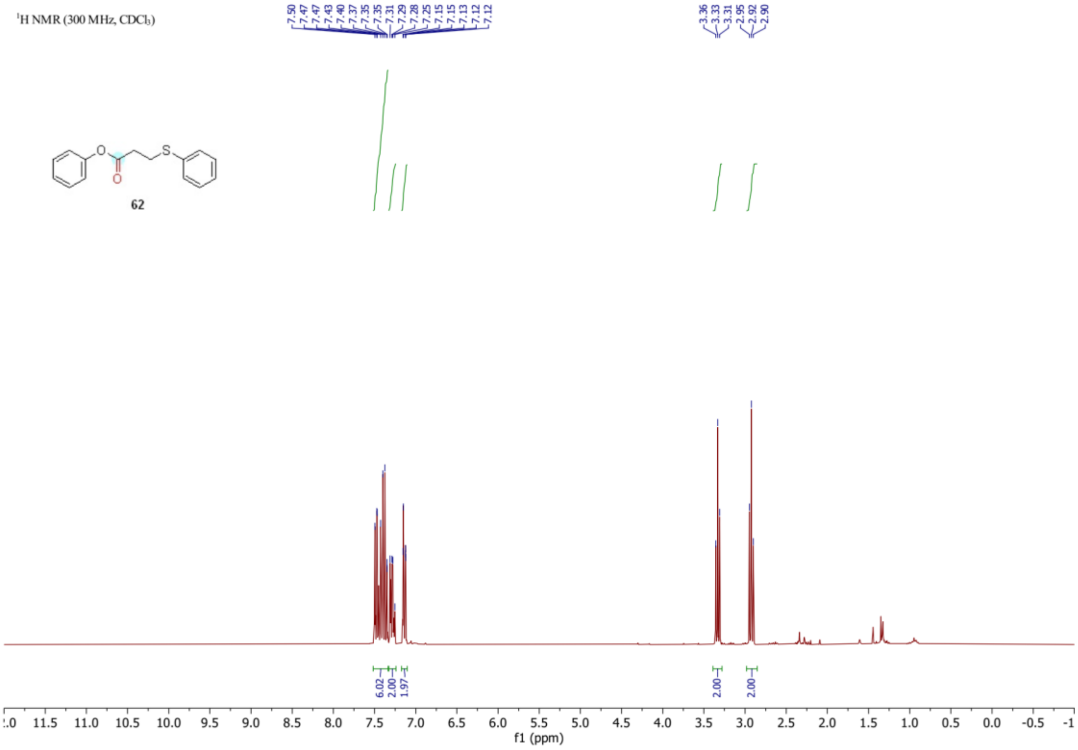
19F NMR (282 MHz, $CDCl_3$) δ -66.26.



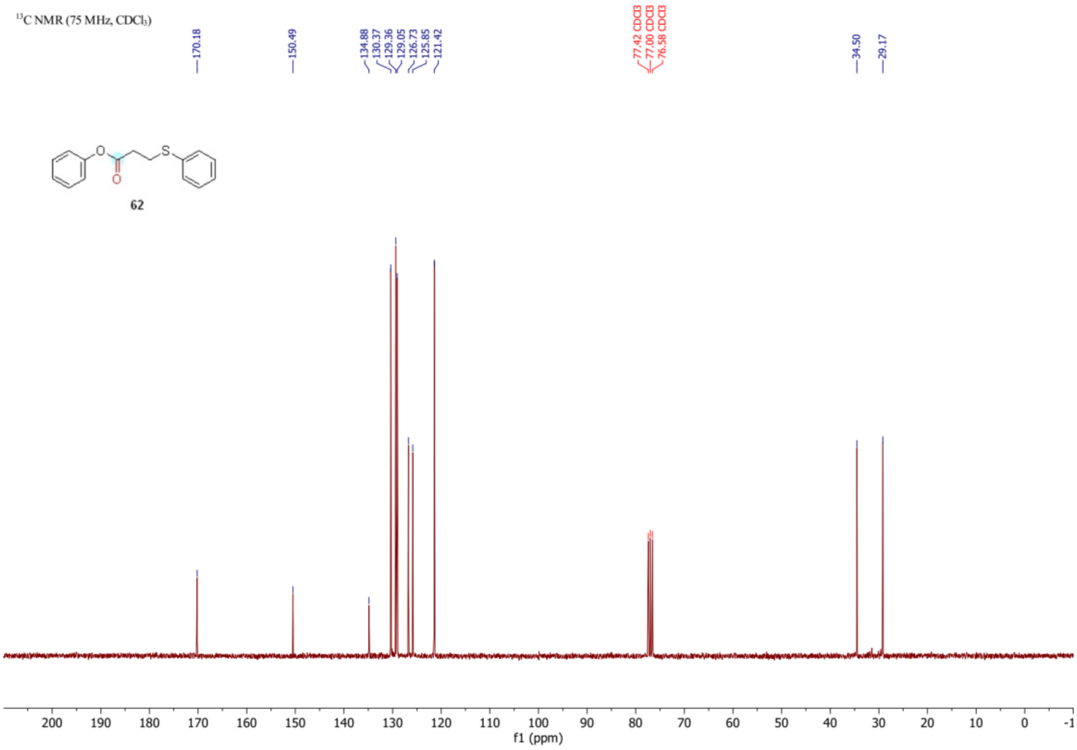
61



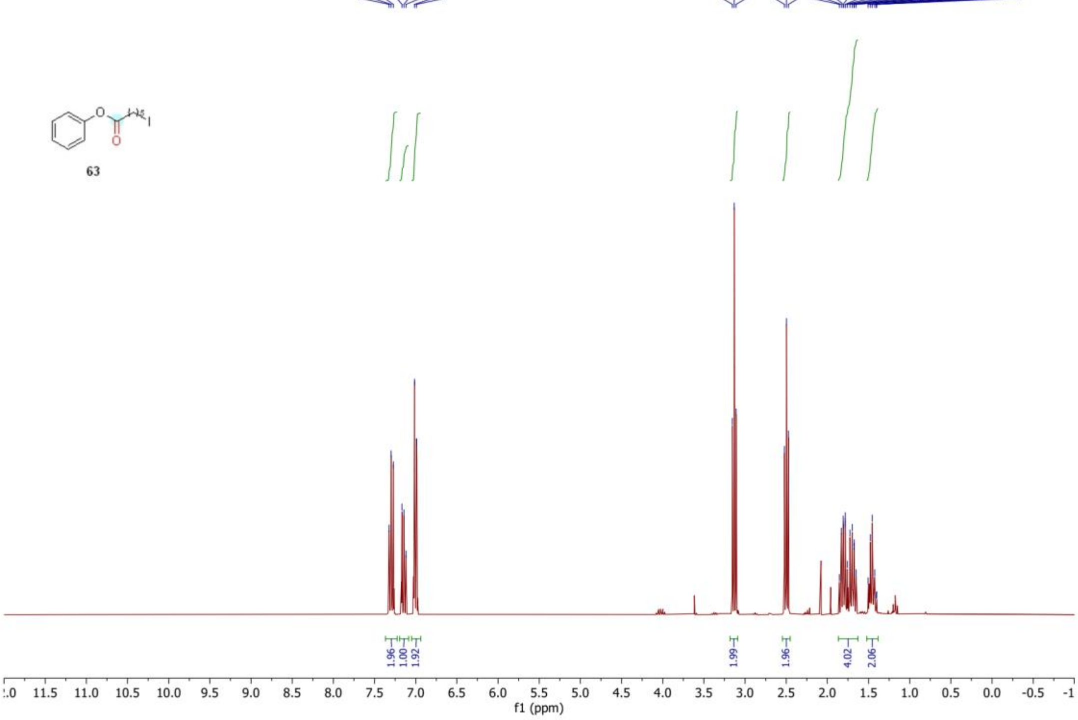
^{1}H NMR (300 MHz, $CDCl_3$)



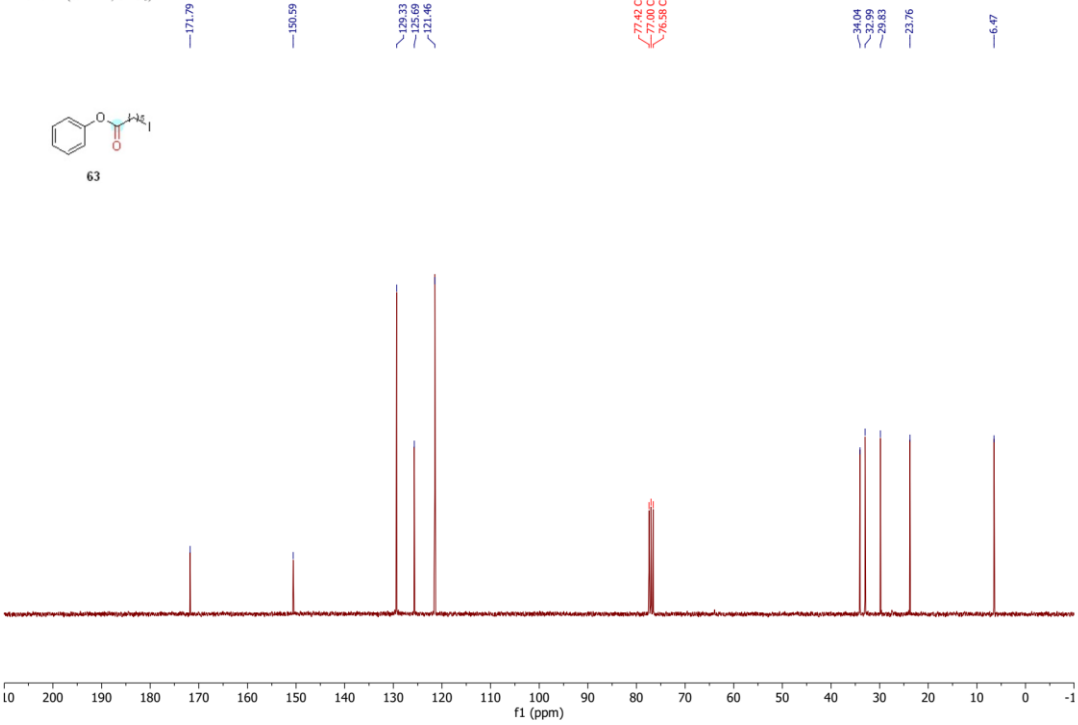
^{13}C NMR (75 MHz, $CDCl_3$)



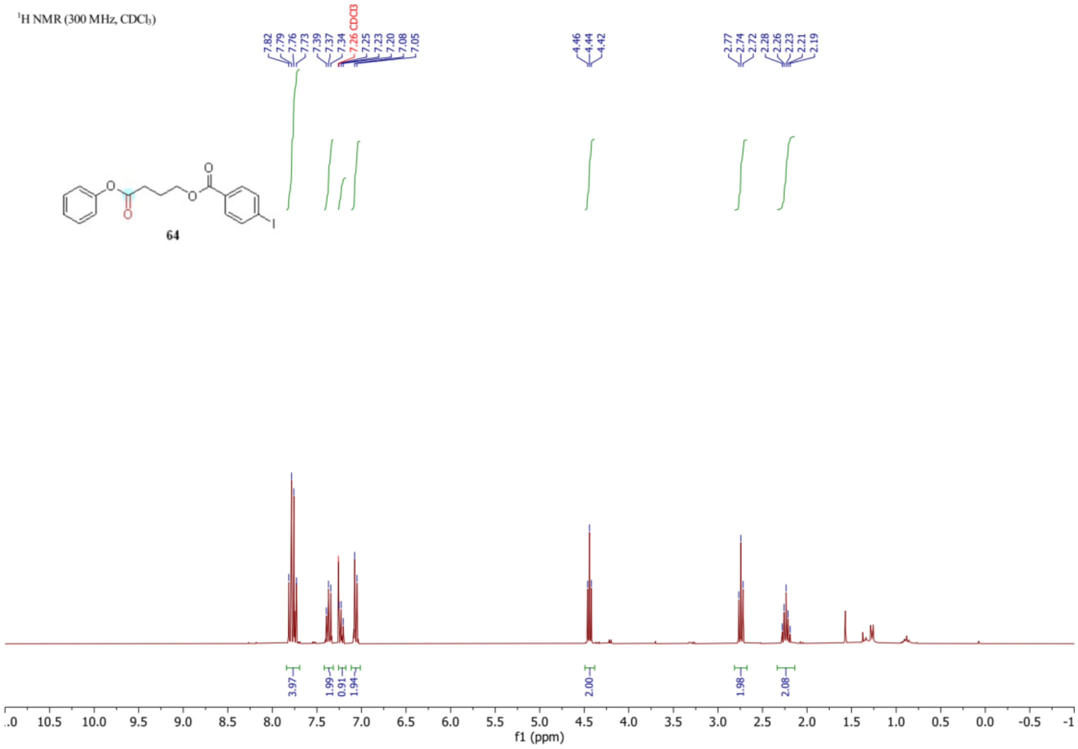
^{1}H NMR (300 MHz, $CDCl_3$)



^{13}C NMR (75 MHz, $CDCl_3$)

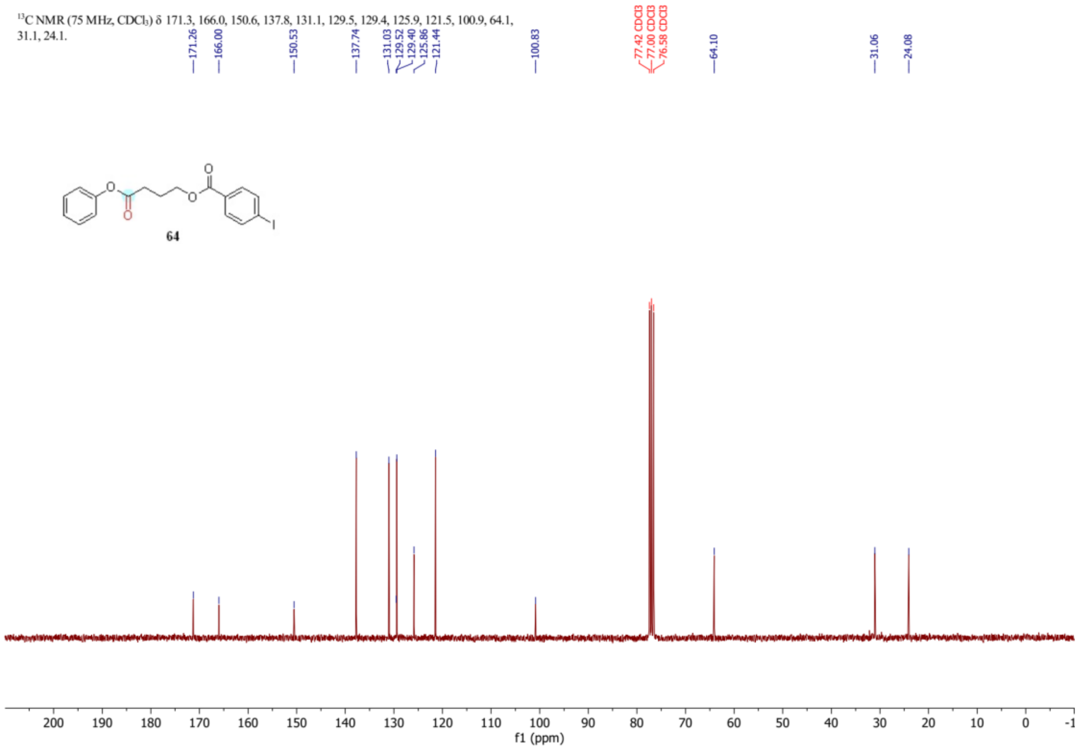


[1]H NMR (300 MHz, $CDCl_3$)



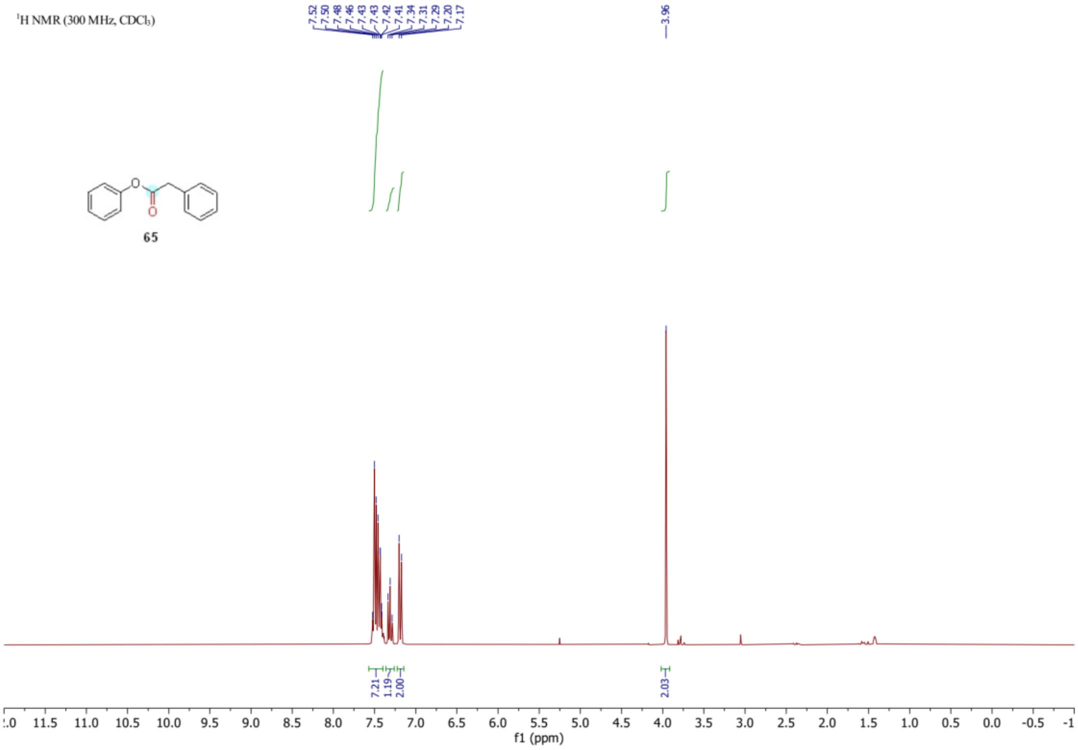
64

[13]C NMR (75 MHz, $CDCl_3$) δ 171.3, 166.0, 150.6, 137.8, 131.1, 129.5, 129.4, 125.9, 121.5, 100.9, 64.1, 31.1, 24.1.



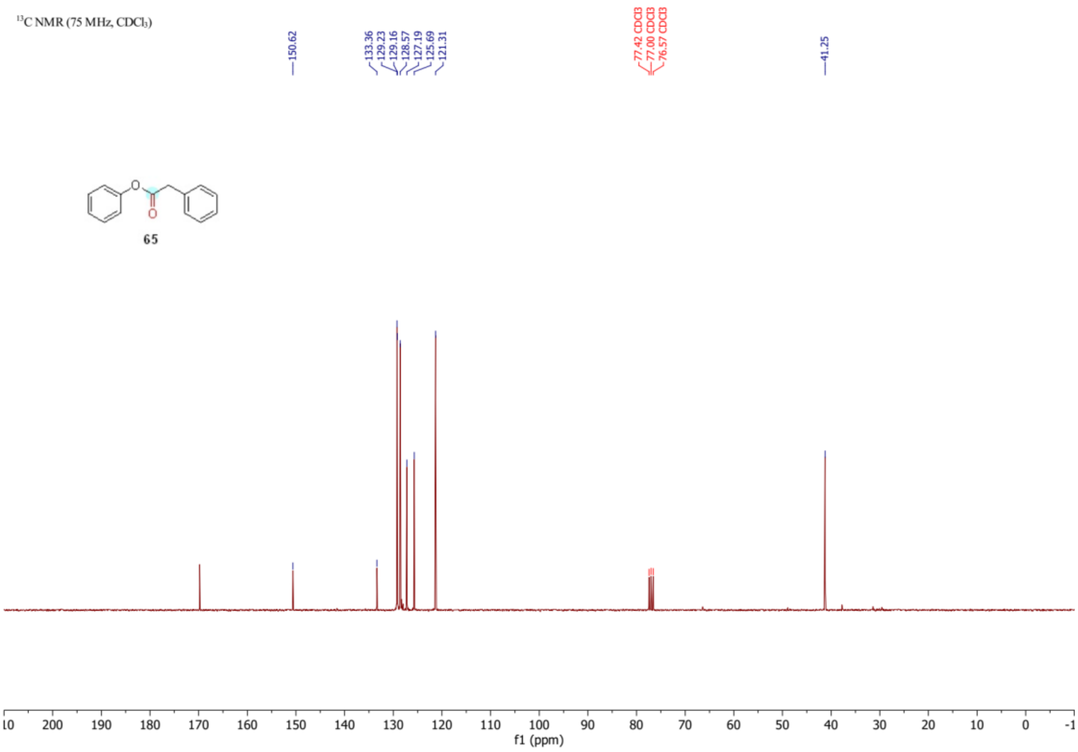
64

^{1}H NMR (300 MHz, $CDCl_3$)



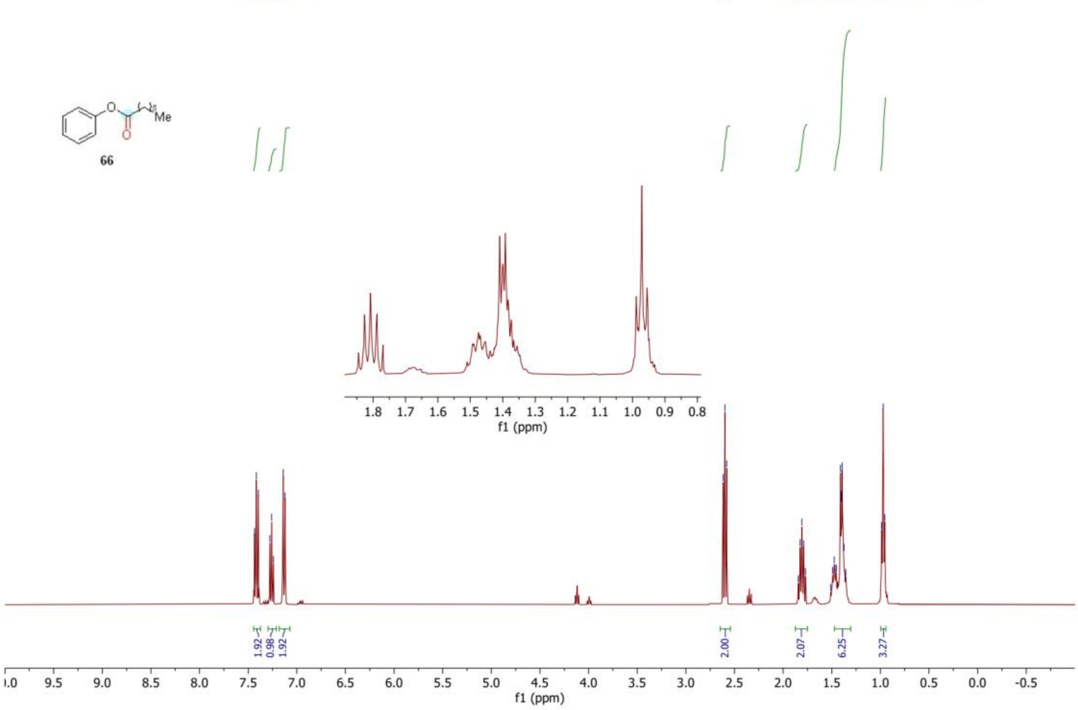
65

^{13}C NMR (75 MHz, $CDCl_3$)

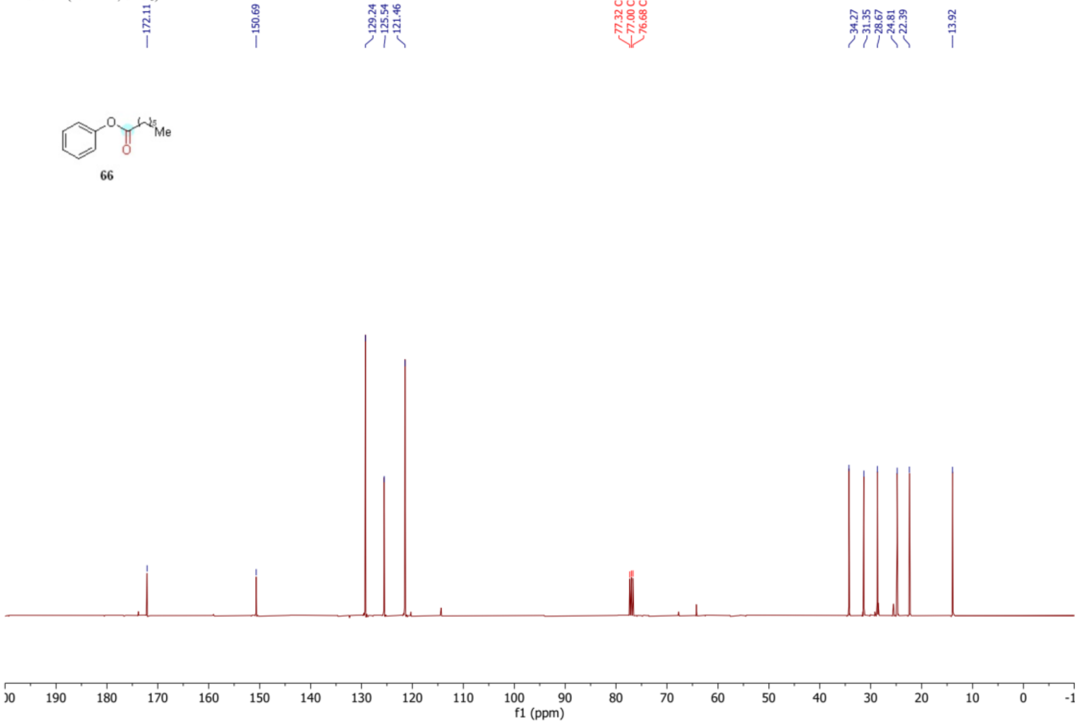


65

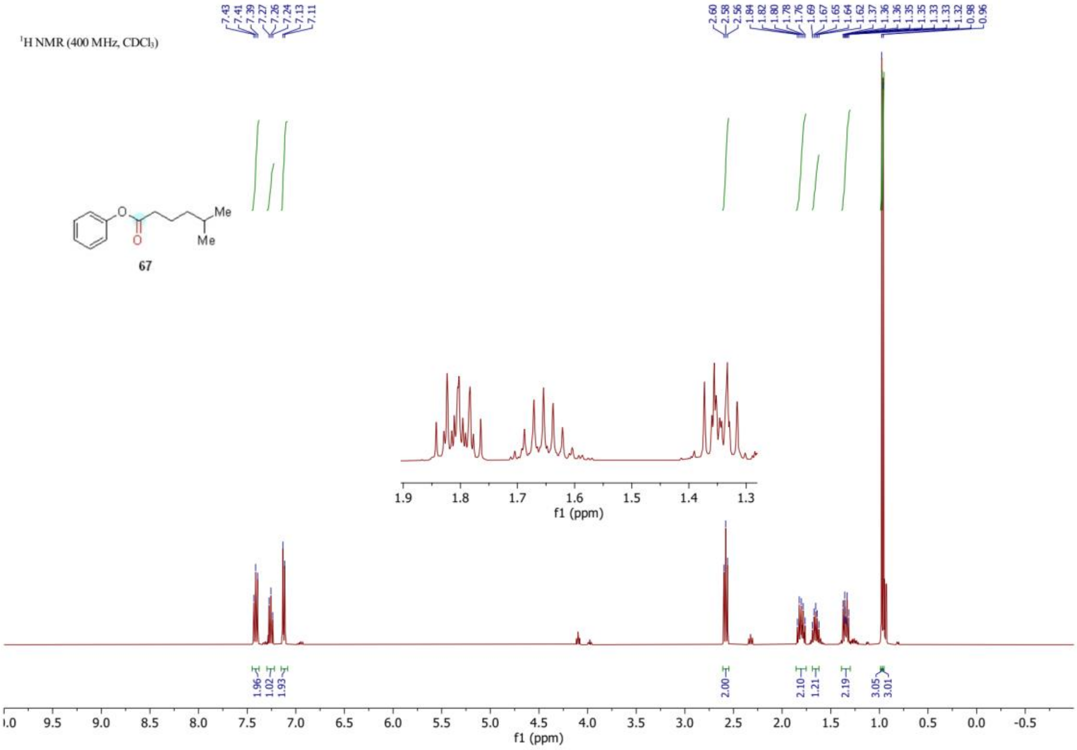
^{1}H NMR (400 MHz, $CDCl_3$)



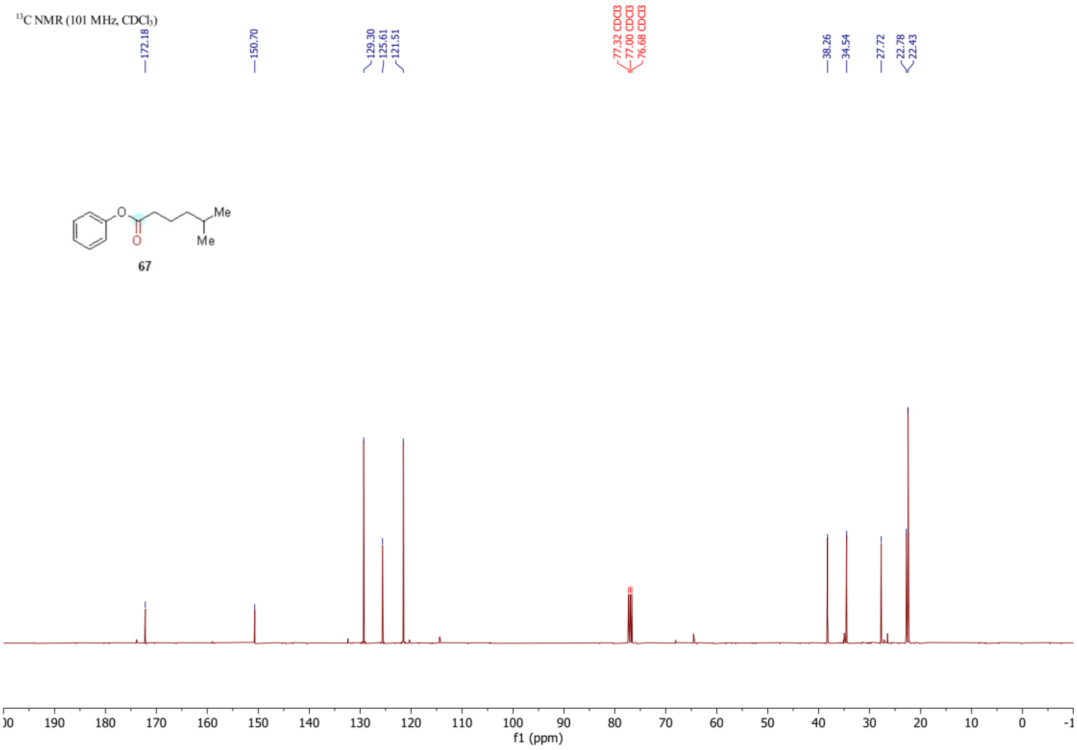
^{13}C NMR (101 MHz, $CDCl_3$)



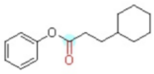
^1H NMR (400 MHz, $CDCl_3$)



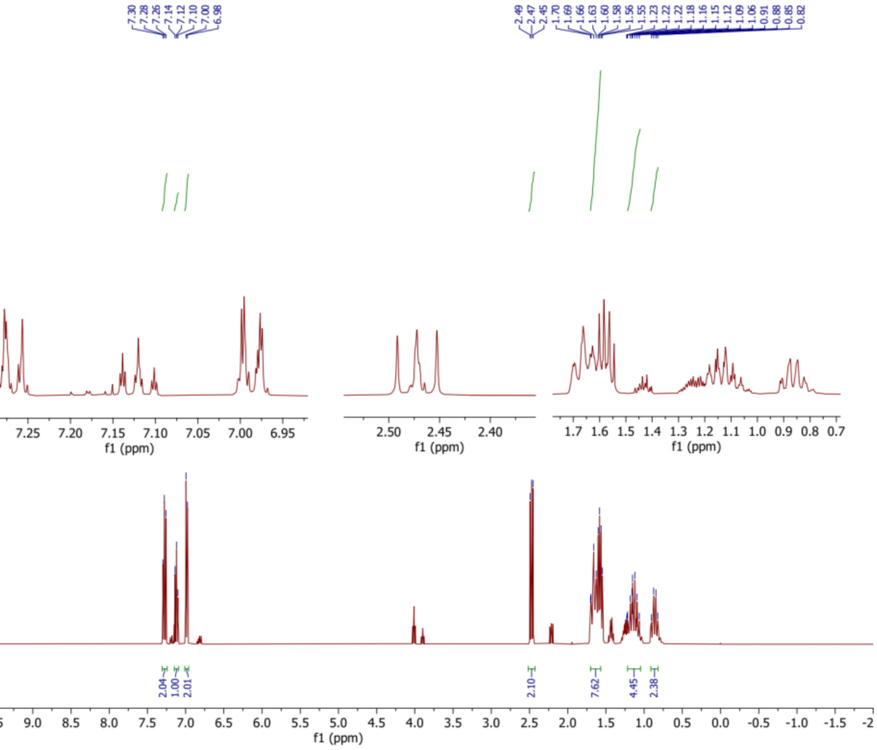
^{13}C NMR (101 MHz, $CDCl_3$)



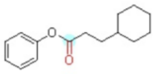
^1H NMR (400 MHz, $CDCl_3$)



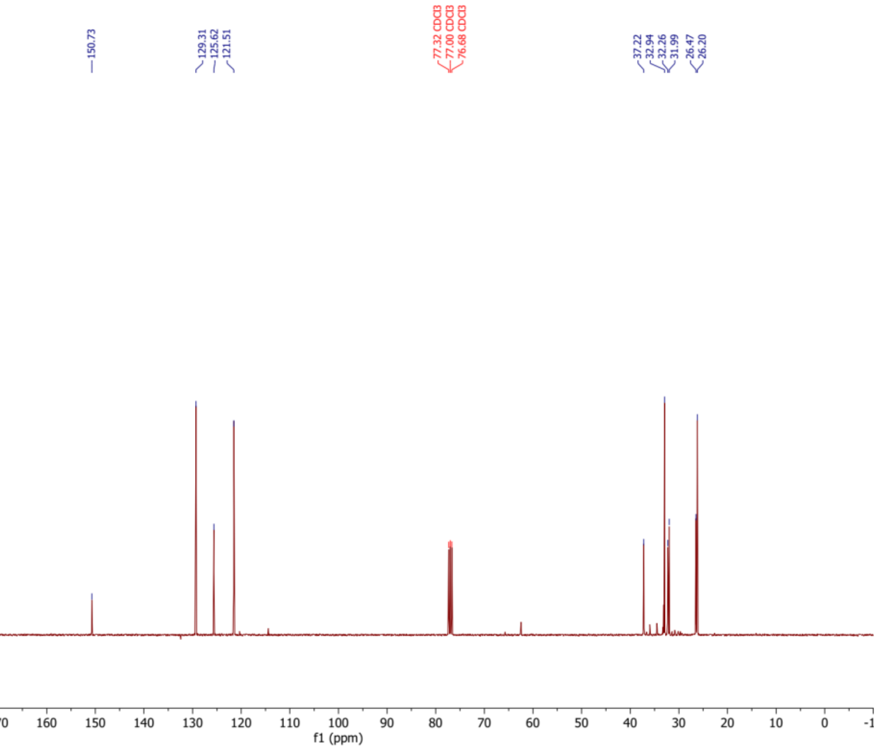
68

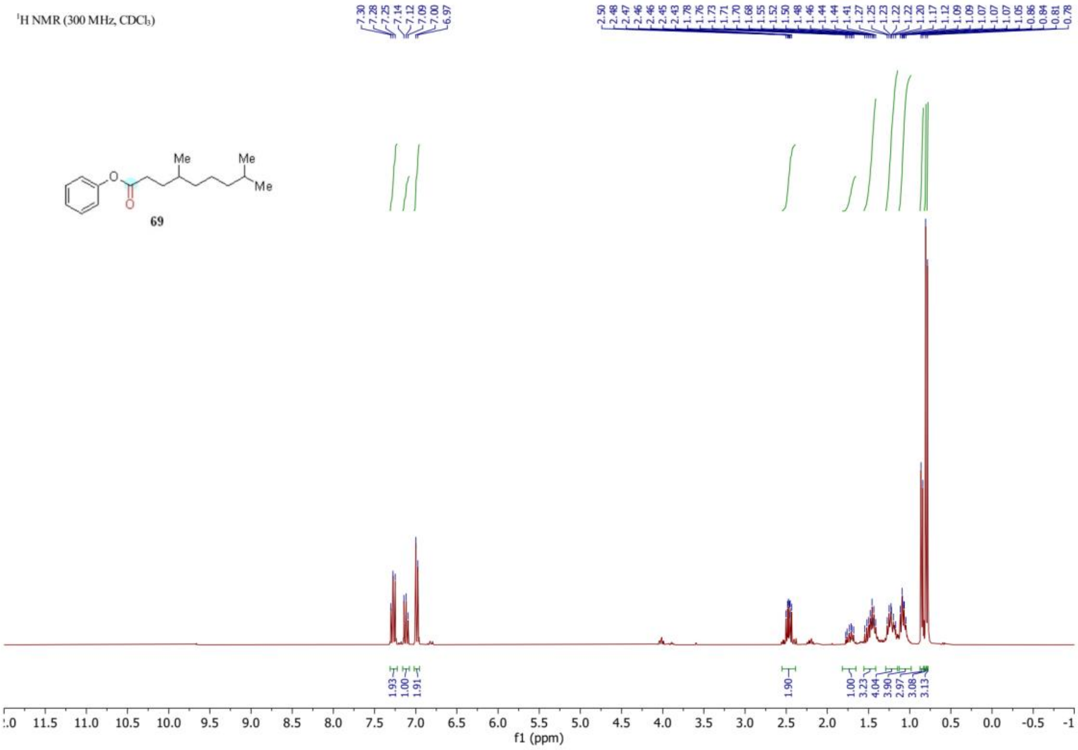


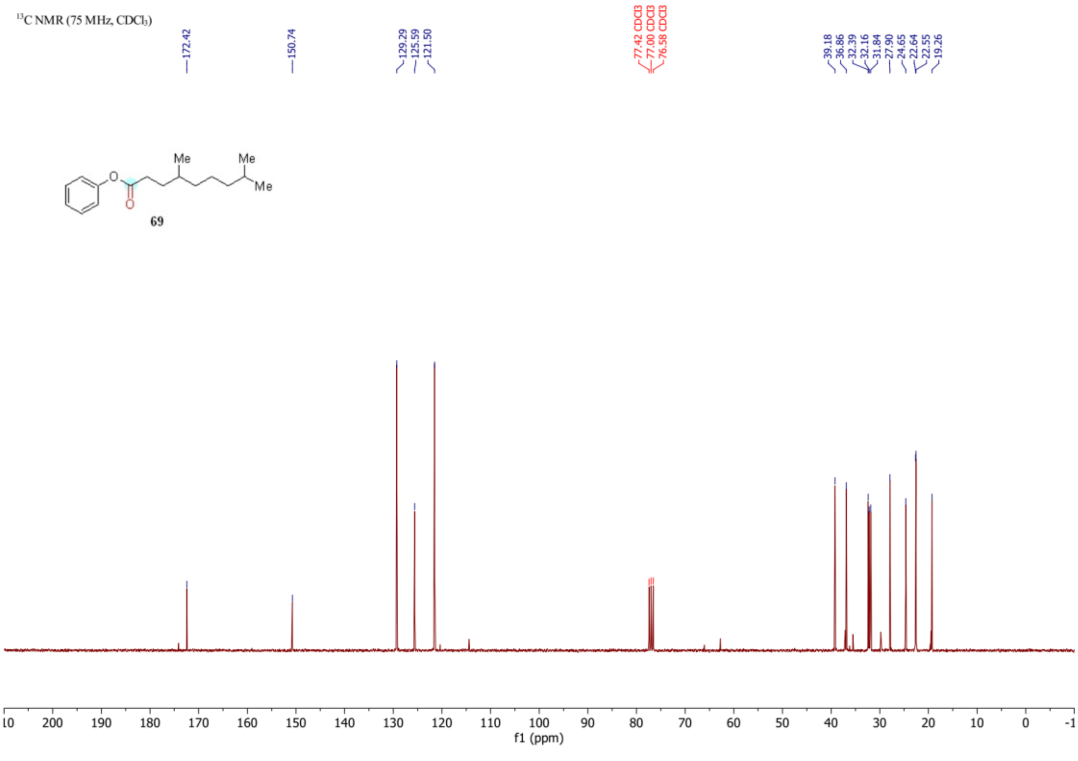
^{13}C NMR (101 MHz, $CDCl_3$)



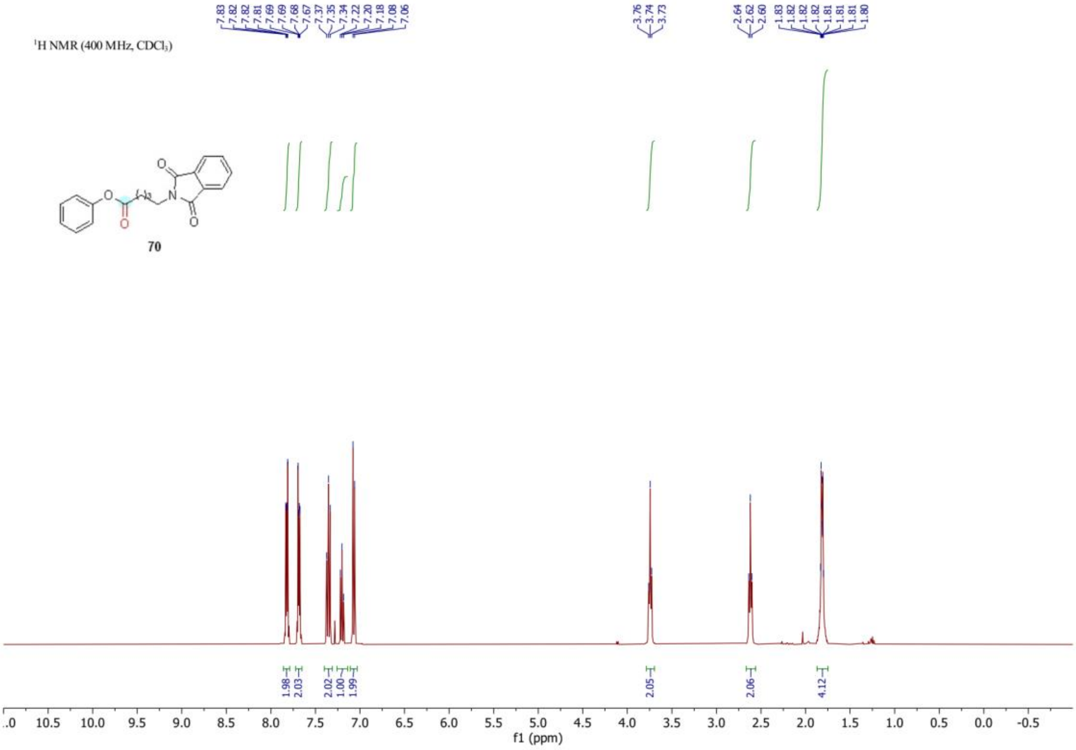
68




1H NMR (300 MHz, CDCl3)
69
f1 (ppm)

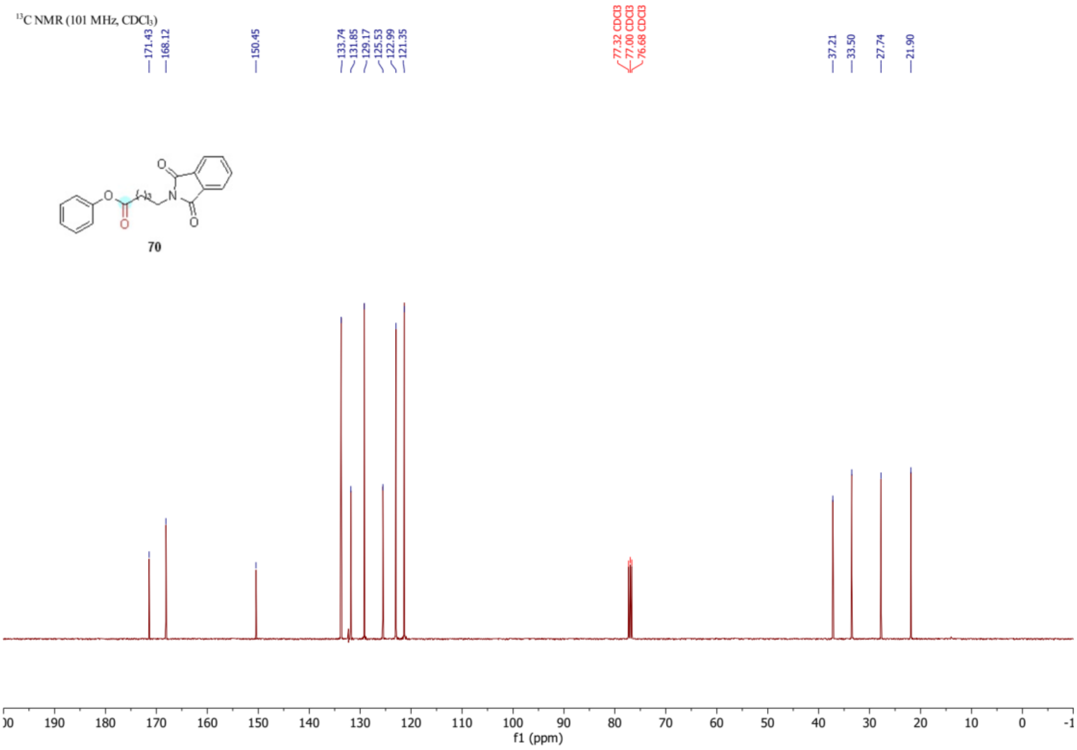

13C NMR (75 MHz, CDCl3)
69
f1 (ppm)

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



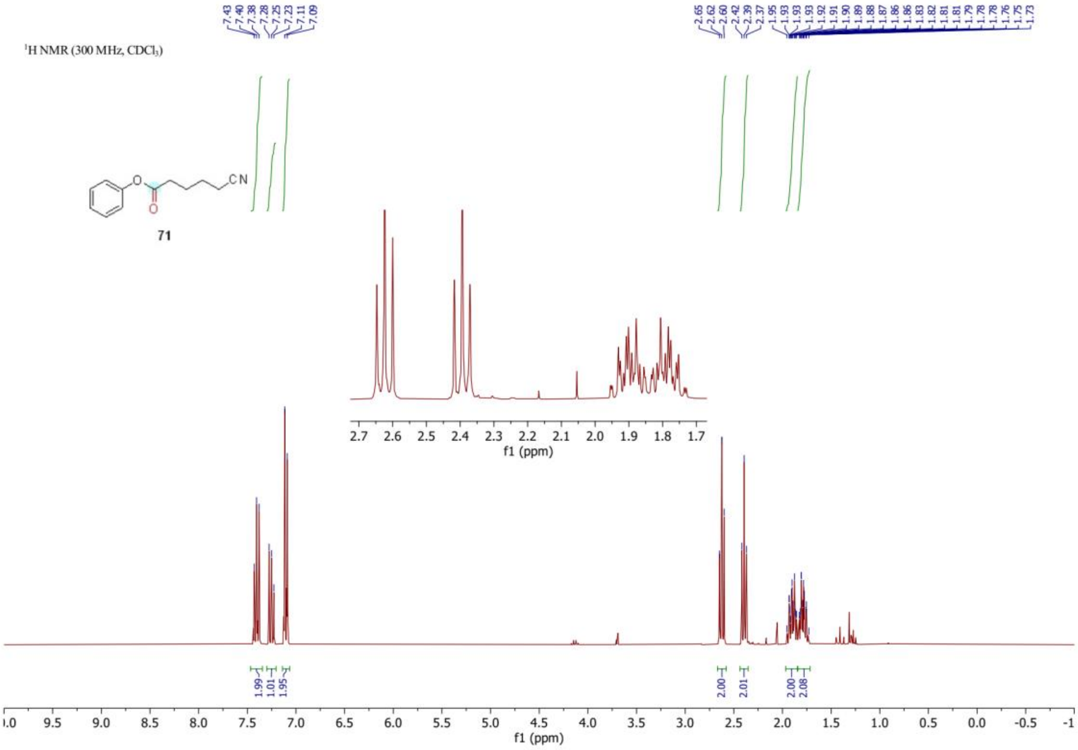
70

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



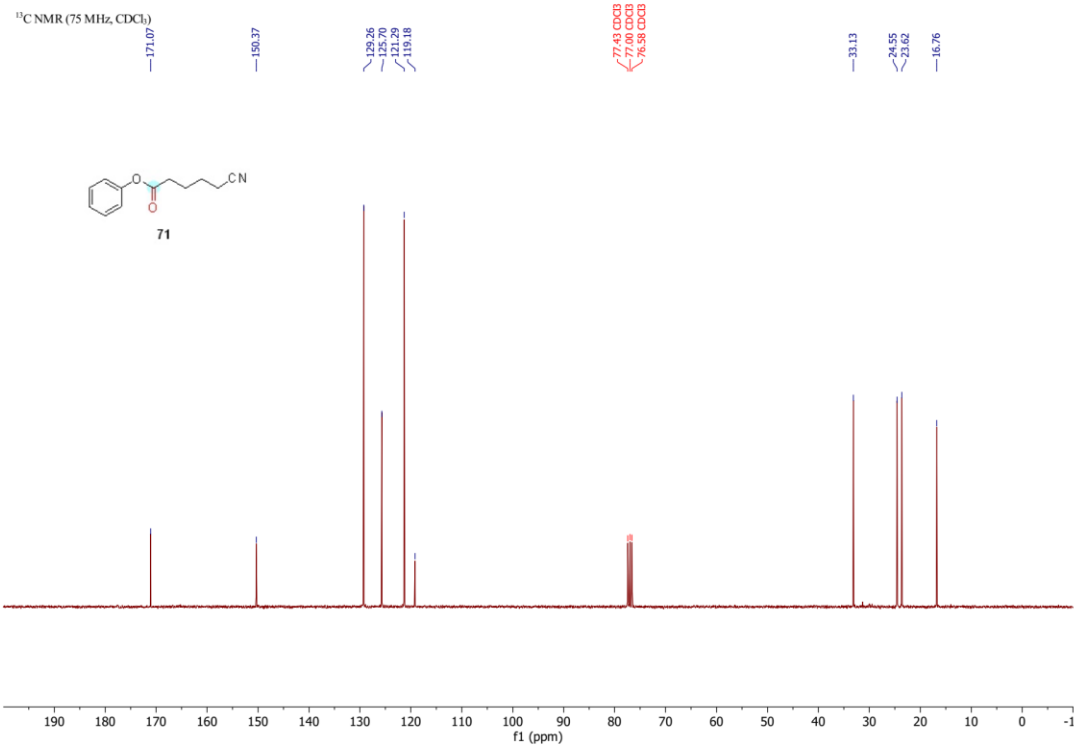
70

^{1}H NMR (300 MHz, CDCl$_3$)



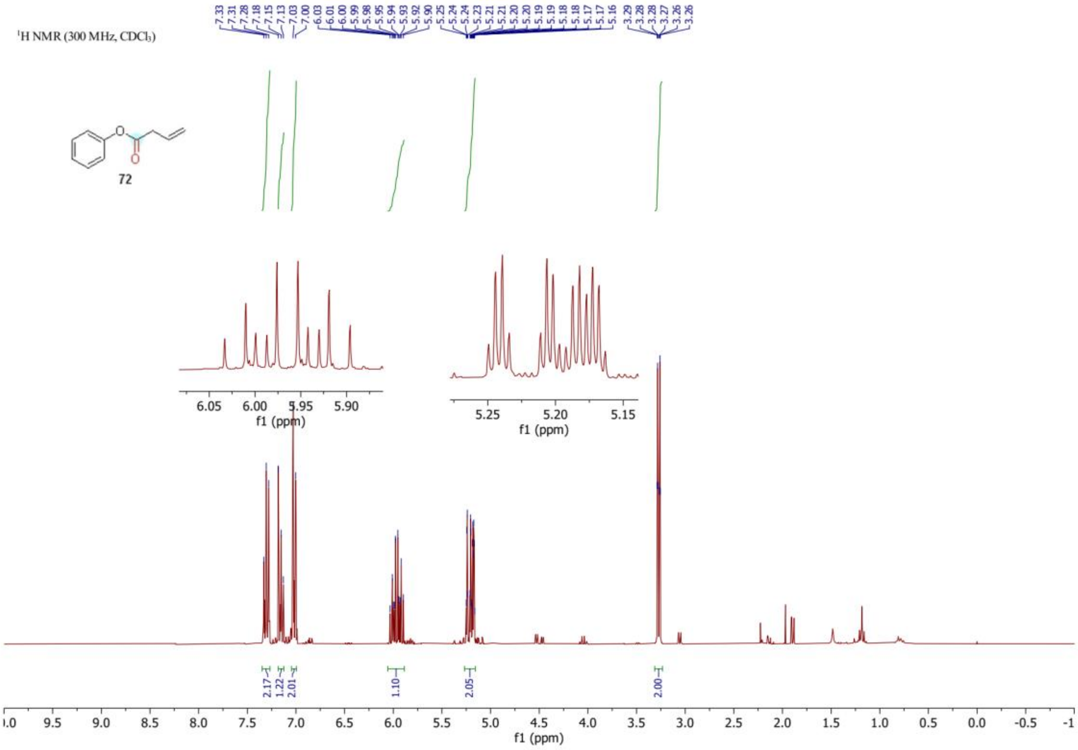
71

^{13}C NMR (75 MHz, CDCl$_3$)



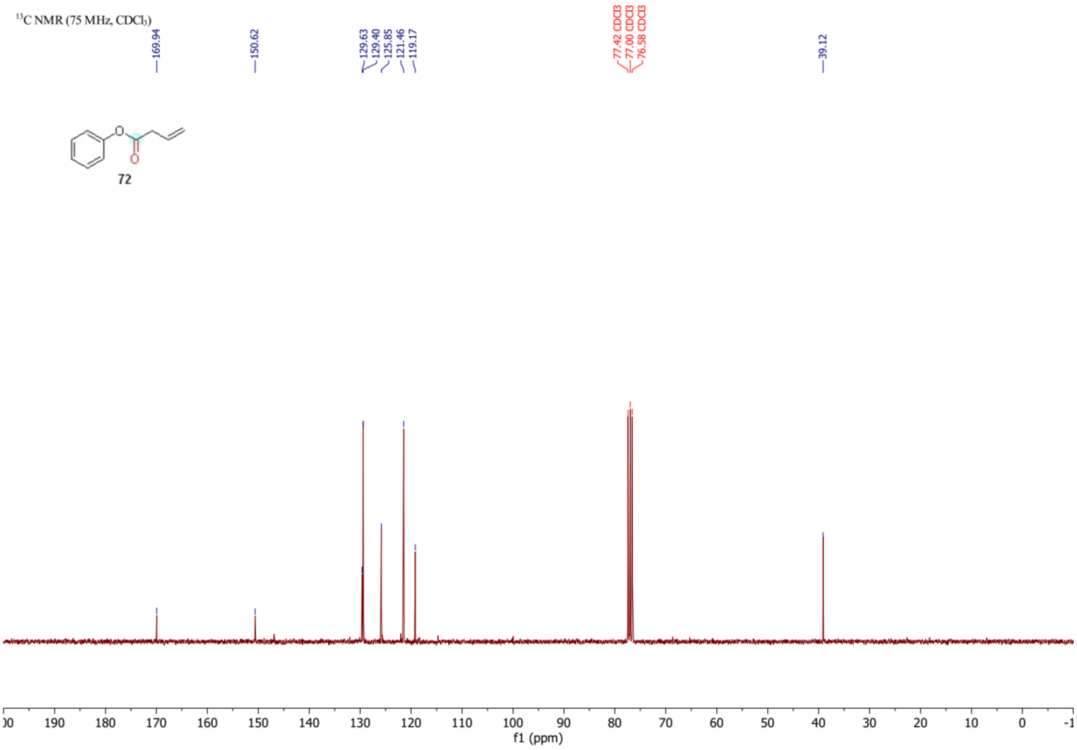
71

¹H NMR (300 MHz, CDCl₃)



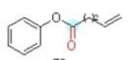
72

¹³C NMR (75 MHz, CDCl₃)

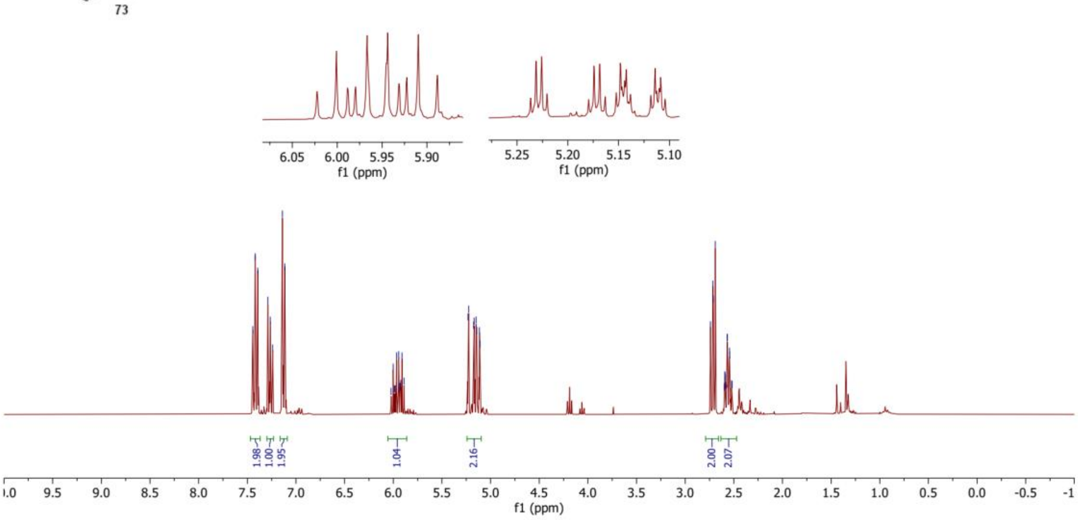


72

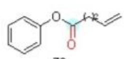
¹H NMR (300 MHz, $CDCl_3$)



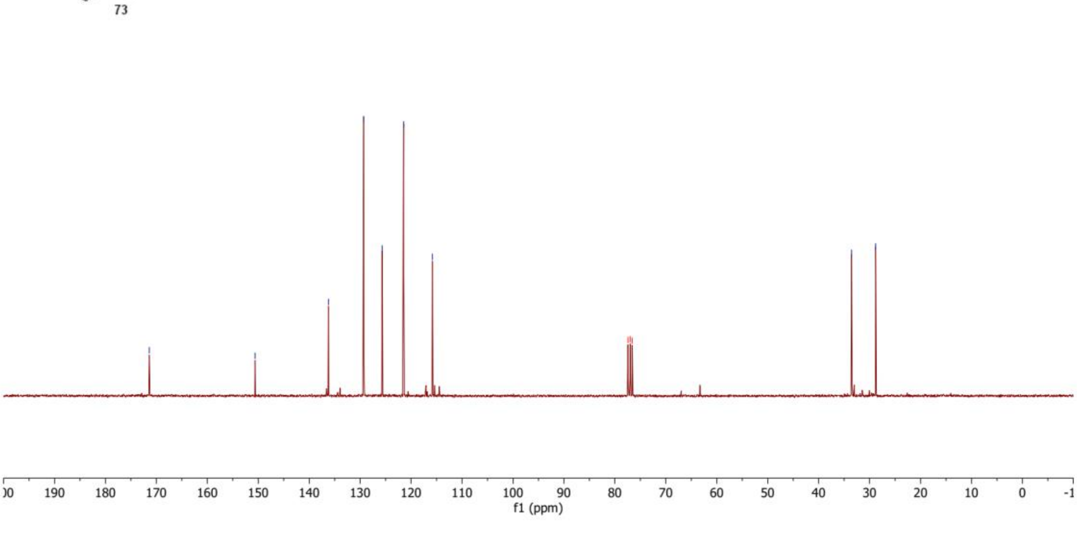
73



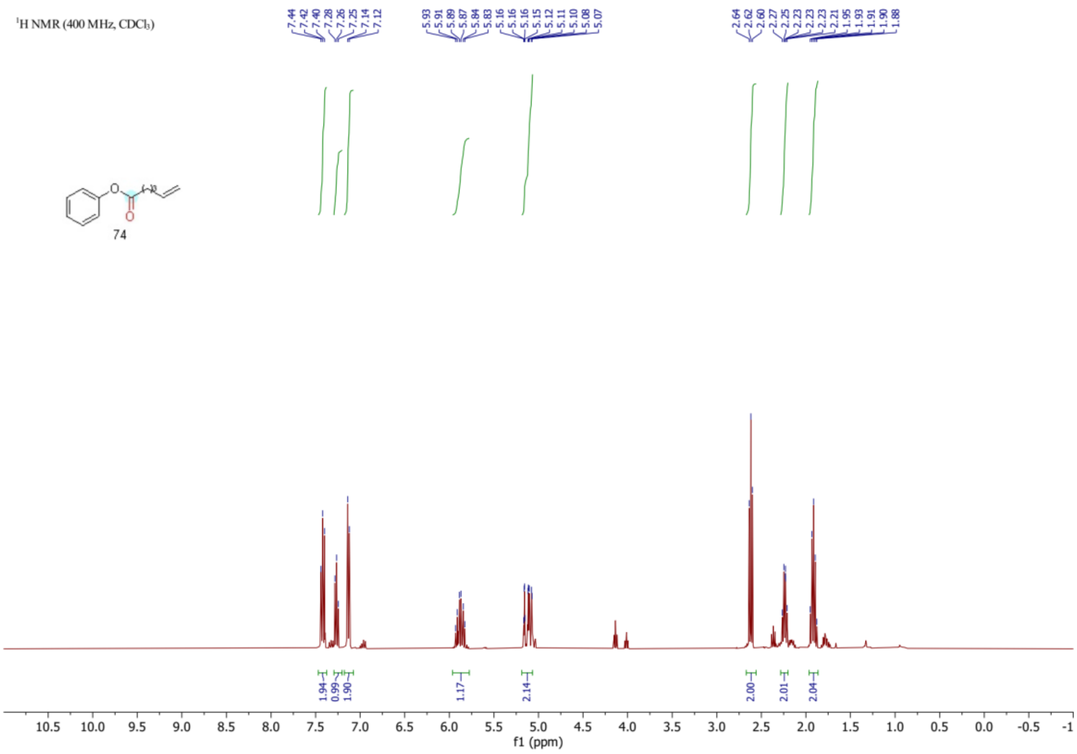
¹³C NMR (75 MHz, $CDCl_3$)



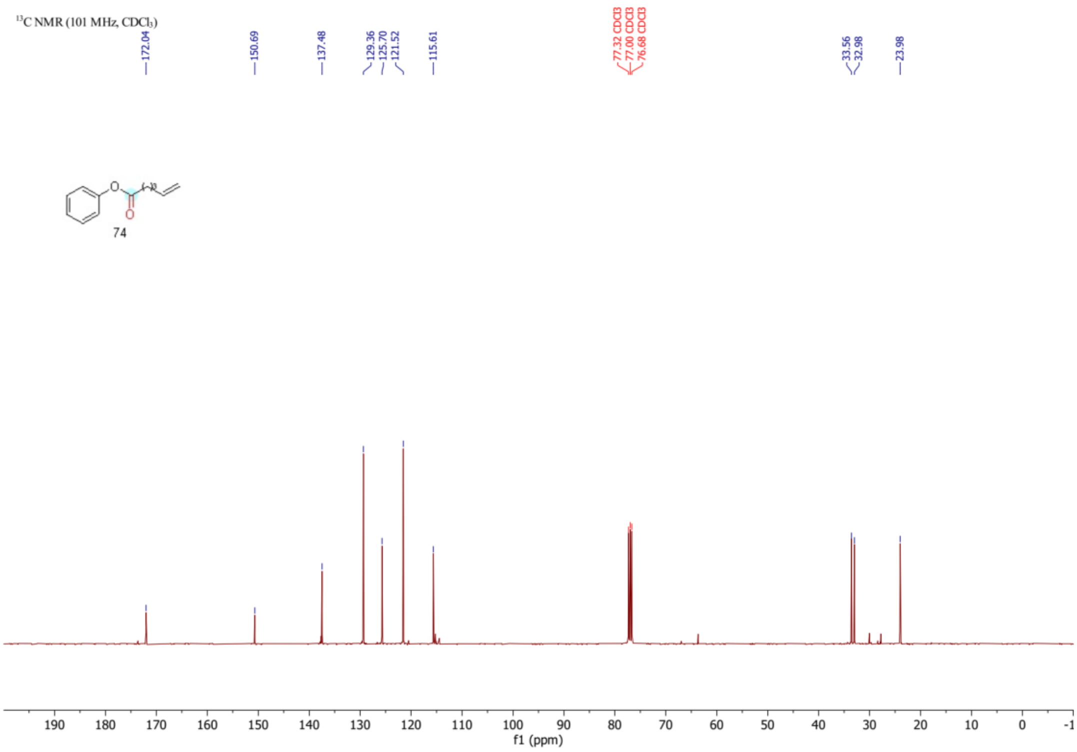
73



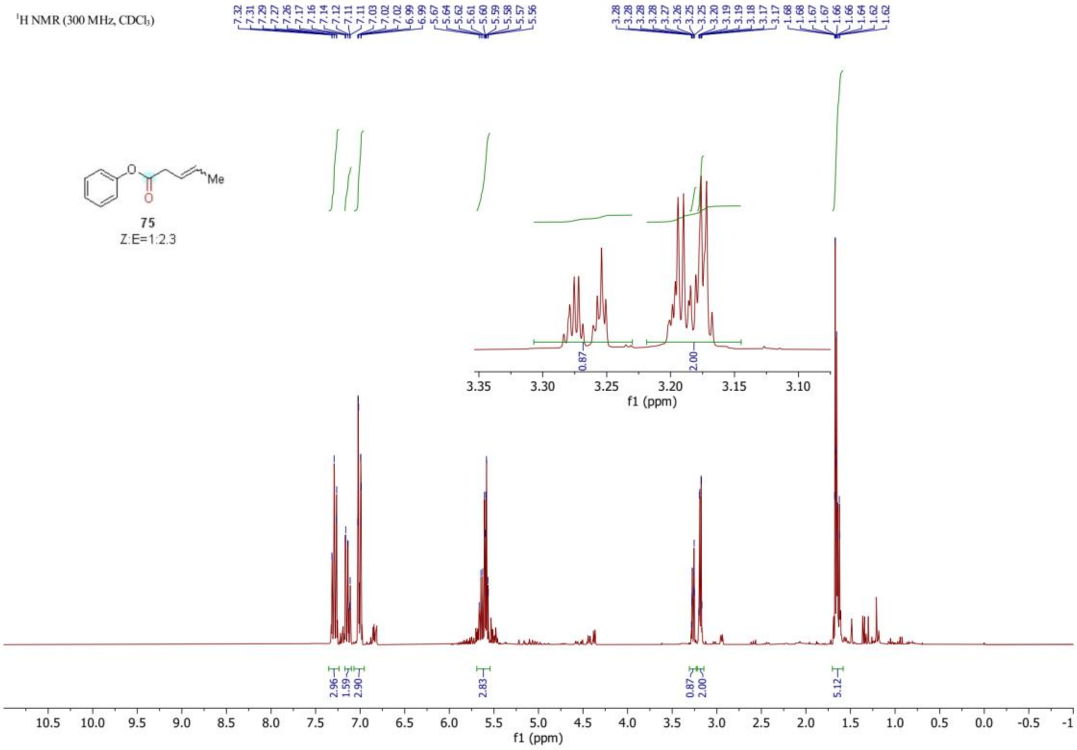
[1]H NMR (400 MHz, $CDCl_3$)



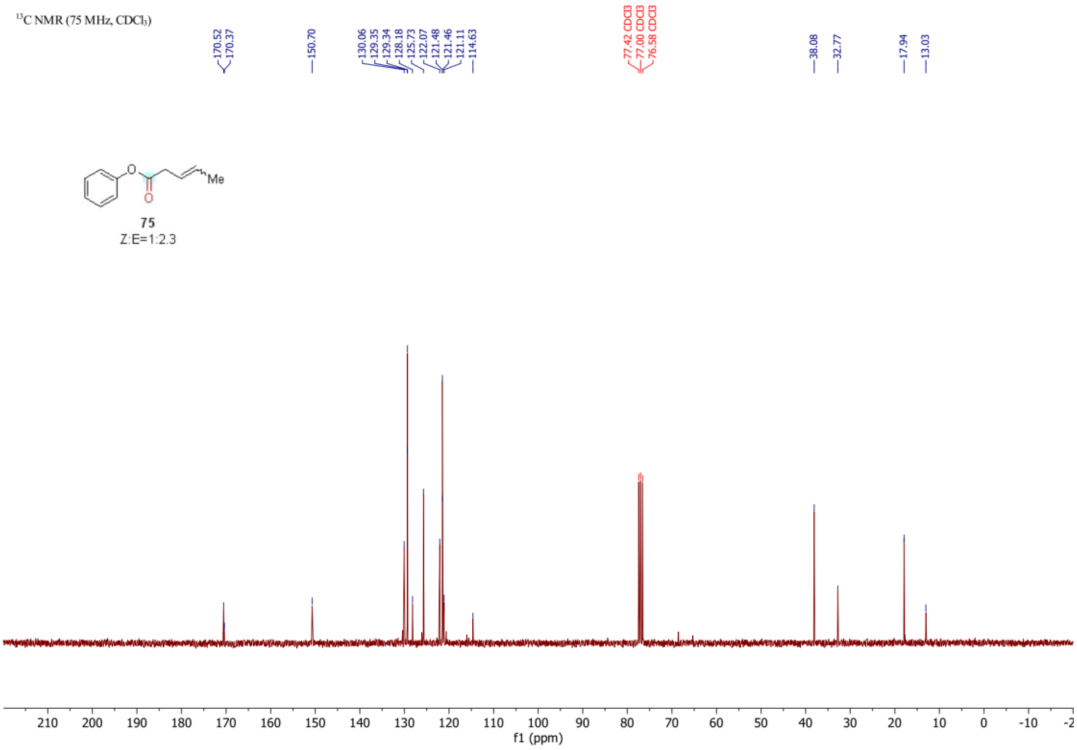
[13]C NMR (101 MHz, $CDCl_3$)

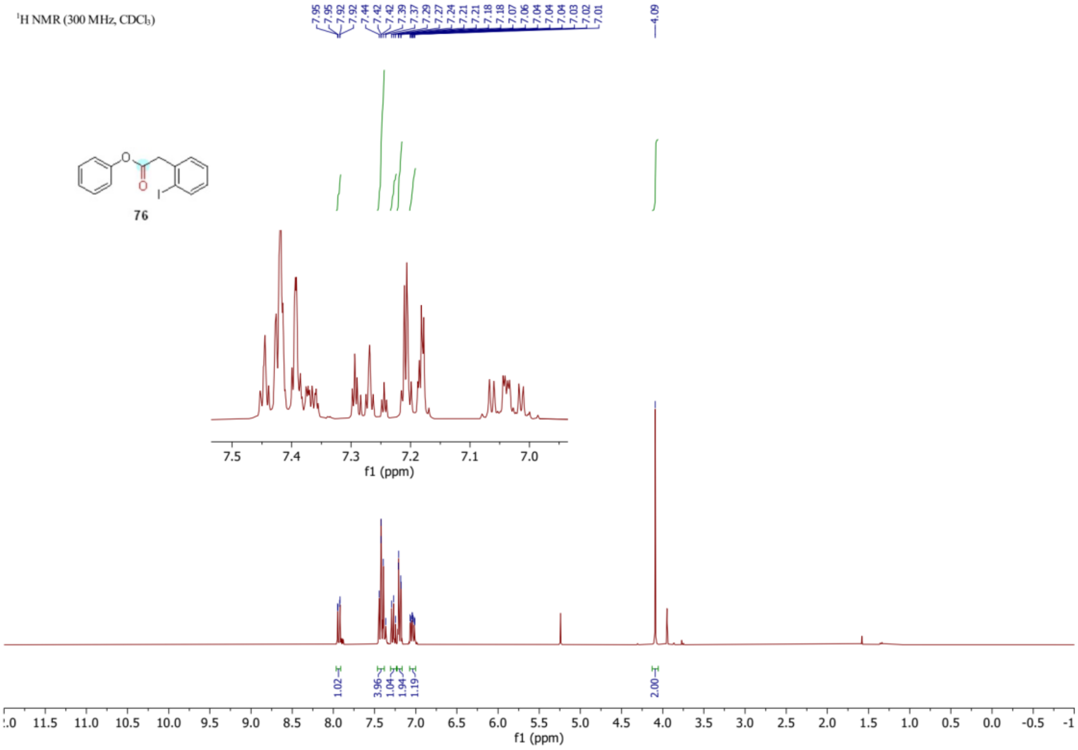


^{1}H NMR (300 MHz, $CDCl_3$)

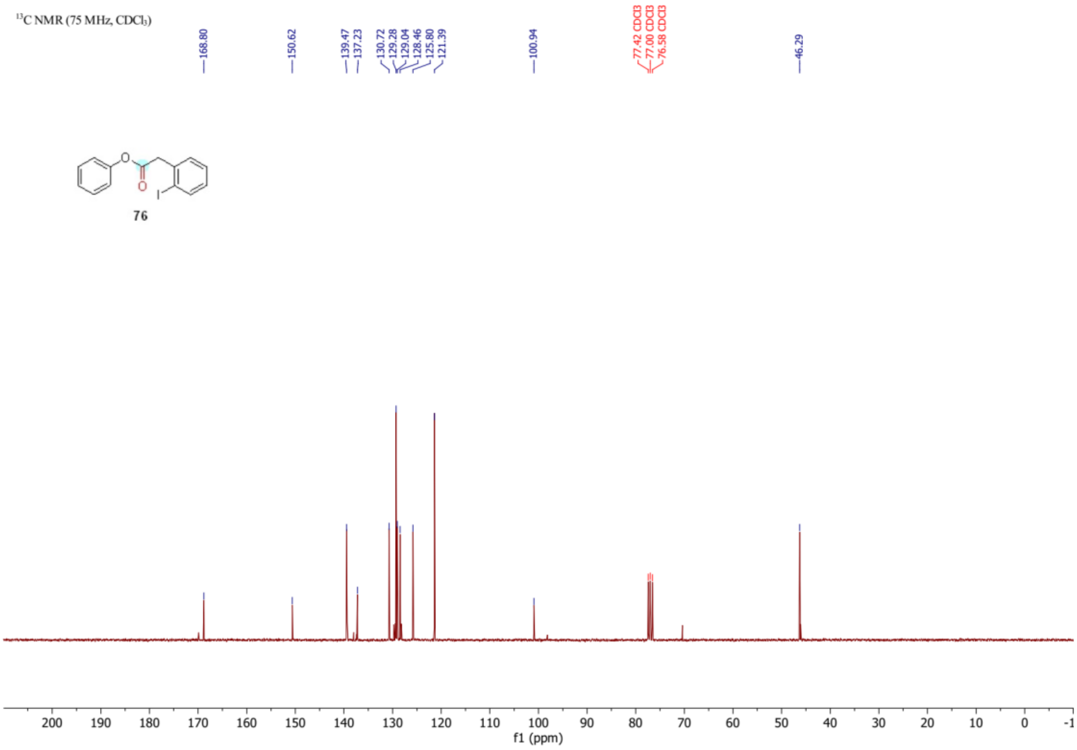


^{13}C NMR (75 MHz, $CDCl_3$)



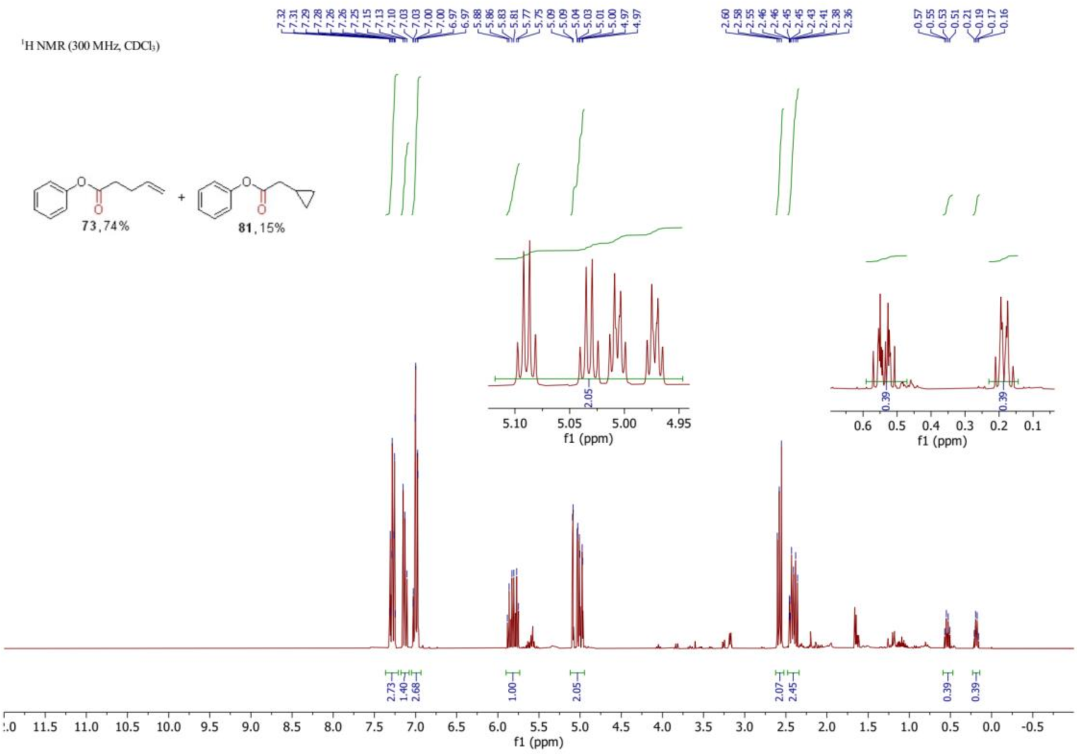


^{1}H NMR (300 MHz, $CDCl_3$)
76
f1 (ppm)



^{13}C NMR (75 MHz, $CDCl_3$)
168.80
150.62
139.47
137.23
130.72
129.28
129.04
128.46
125.80
121.39
100.94
77.42 CDCl3
77.00 CDCl3
76.58 CDCl3
46.29
76
f1 (ppm)

^{1}H NMR (300 MHz, $CDCl_3$)



^{13}C NMR (75 MHz, $CDCl_3$)