Supporting Information

New Electrotriggers: *p*-Methoxycarbonylbenzyl (pMCB) as an Electroremovable Protecting Group for Carboxylic Acids, Phosphoric Acids and Alcohols

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I. General Information

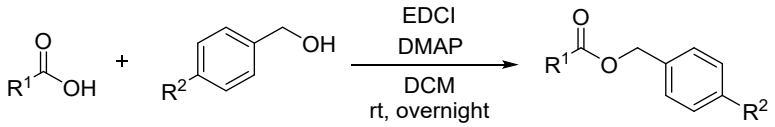
All reactions were performed under air atmosphere, using round bottom flasks. All substrates were obtained from the commercial sources or synthesized following literature procedures. All reagents were commercial and were used without further purification. Phosphate buffer saline (PBS): 8.0 mM of Na_2HPO_4, 137 mM of NaCl and 2.0 mM of NaH_2PO_4 in H_2O. The electrochemical reaction device follows our previous work.[1] The instrument for electrolysis is Single Output DC Power Supply (KRP-305DM) (made in China). Chromatography was carried on flash silica gel (300-400 mesh). All reactions were monitored by TLC, which was performed on percolated aluminum sheets of silica gel 60 (F254). Melting points were uncorrected. The ^1H and ^{13}C NMR data were obtained on a 300 MHz NMR spectrometer with TMS as the internal standard and $CDCl_3$ or DMSO-d_6 as solvent. Multiplicities are indicated as it follows: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; dd, doubled doublet; br, broad. Coupling constants (*J* values) where noted are quoted in Hertz. High-resolution mass spectra (HRMS) were obtained with a time-of-flight (TOF) mass spectrometer (ESI).

Electrode materials/dimensions:

The graphite electrodes are furnished from commercial graphite rod (5 mm × 300 mm), purchased from Zhongnuotansu (Tianjin). The dimensions of the graphite electrode is 5 mm × 50 mm (the submerged height of the electrode is approximately 5 mm).

II. Substrates Synthesis and Characterization

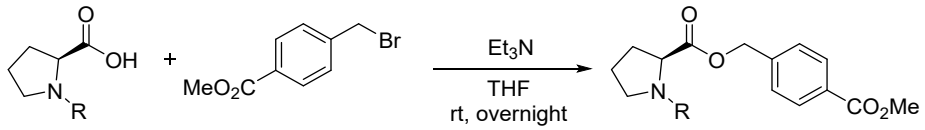
Method A: Synthesis of compounds **1a**–**f**, **42**–**55**, **65**, **66**, **68** and **69**.



1a-1f, **42-55**, **65**, **66**, **68** and **69**

At room temperature, corresponding carboxylic acid (2.4 mmol, 1.2 eq.) was dissolved in DCM (20 mL) in a 50 mL round bottom flask, then corresponding benzyl alcohol (2.0 mmol, 1.0 eq.) and DMAP (0.29 g, 2.4 mmol, 1.2 eq.) was added. Then, EDCI (0.58 g, 3.0 mmol, 1.5 eq.) was added at 0 °C. The resulting solution was stirred overnight at room temperature. The solution was diluted with DCM (30 mL) and quenched with 1*N* HCl aqueous (30 mL). The aqueous layer was extracted with DCM (3 × 30 mL). The combined DCM layers were washed with brine (3 × 20 mL), dried over Na_2SO_4, and concentrated *in vacuo*. Purification by flash column chromatography using PE/EtOAc as eluent afforded the desired product **1a**–**f**, **42**–**55**, **65**, **66**, **68** and **69**.

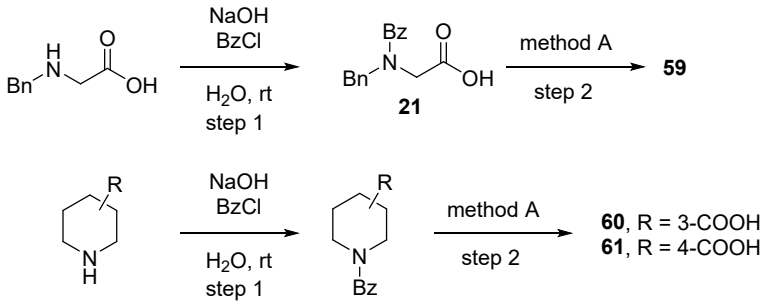
Method B: Synthesis of compounds **56**–**58**.



56, R = Bz
57, R = Boc
58, R = Cbz

To a dried 10 mL round bottom flask were added corresponding amino acid (1.2 mmol, 1.2 eq.), THF (1.0 mL) and methyl 4-(bromomethyl)benzoate (0.23 g, 1.0 mmol, 1.0 eq.) sequentially. Then, Et_3N (0.15 g, 1.5 mmol, 1.5 eq.) was added dropwise. The resulting solution was stirred overnight at room temperature. The solution was diluted with DCM (5 mL) and quenched with 1*N* HCl aqueous (10 mL). The aqueous layer was extracted with DCM (3 × 10 mL). The combined DCM layers were washed with brine (3 × 10 mL), dried over Na_2SO_4, and concentrated *in vacuo*. Purification by flash column chromatography using PE/EtOAc as eluent afforded the desired product **56**–**58**.

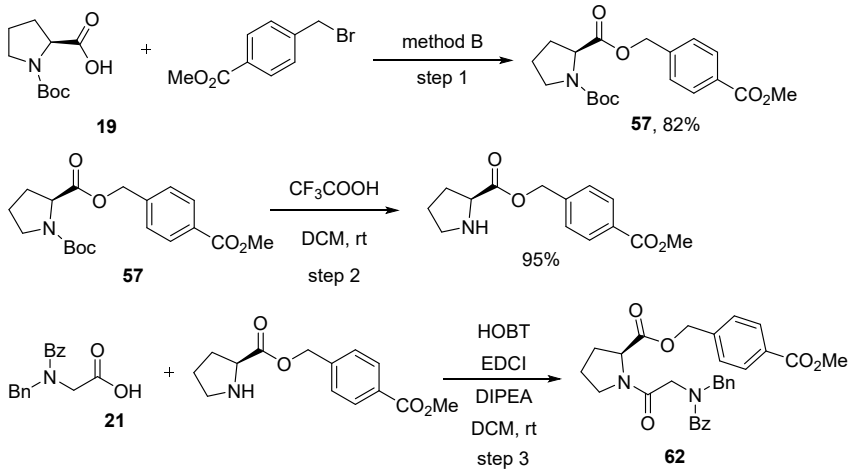
Method C: Synthesis of compounds **59**–**61**.



Step 1: To a dried 100 mL round bottom flask were added corresponding amino acid (20 mmol, 1.0 eq.), H_2O (40 mL) and NaOH (1.6 g, 40 mmol, 2.0 eq.) sequentially. Then, benzoyl chloride (2.8 mL, 24 mmol, 1.2 eq.) was added dropwise at 0 °C. The resulting solution was stirred overnight at room temperature. The solution was diluted with DCM (50 mL) and quenched with 1*N* HCl aqueous (50 mL). The aqueous layer was extracted with DCM (3 × 50 mL). The combined DCM layers were washed with brine (3 × 50 mL), dried over Na_2SO_4 and concentrated *in vacuo* without purification.

Step 2: Prepared according to method A. Purification by flash column chromatography using PE/EtOAc as eluent afforded the desired product **59**–**61**.

Method D: Synthesis of compounds **62** and **63**.



Step 1: Prepared according to method B. Purification by flash column chromatography using PE/EtOAc as eluent afforded the desired product.

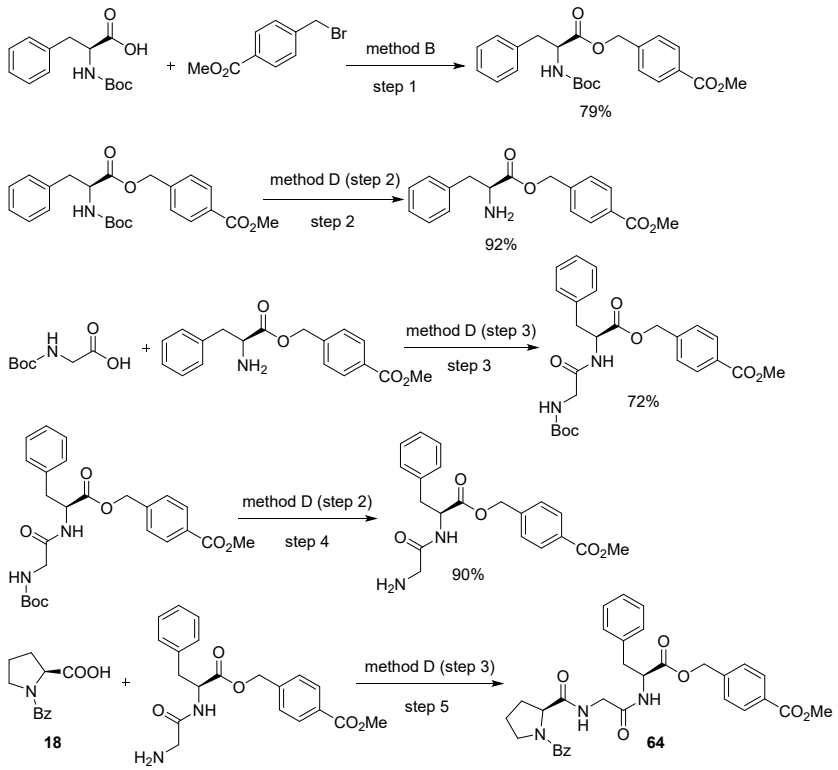
Step 2: To a dried 10 mL round bottom flask were added compound **57** (1.0 mmol, 1.0 eq.), DCM (2.0 mL). Then, trifluoroacetic acid (2.0 mL) was added dropwise at 0 °C. The resulting solution was stirred for 2.0 h at room temperature. The solution was diluted with DCM (10 mL) and quenched with saturated aqueous $NaHCO_3$ (10 mL).

The aqueous layer was extracted with DCM (3 × 10 mL). The combined DCM layers were washed with brine (3 × 10 mL), dried over Na_2SO_4, and concentrated *in vacuo*. The resulting solution was filtered and concentrated without purification.

Step 3: To a dried 25 mL round bottom flask were added corresponding amine (1.0 mmol, 1.0 eq.), DCM (10 mL), compound **21** (1.2 mmol, 1.2 eq.), HOBT (0.16 g, 1.2 mmol, 1.2 eq.), EDCI (0.23 g, 1.2 mmol, 1.2 eq.) and DIPEA (0.26 g, 2.0 mmol, 2.0 eq.) sequentially. The resulting solution was stirred overnight at room temperature. The solution was diluted with DCM (10 mL) and quenched with 1*N* HCl aqueous (10 mL).The aqueous layer was extracted with DCM (3 × 10 mL). The combined DCM layers were washed with brine (3 × 10 mL), dried over Na_2SO_4, and concentrated *in vacuo*. Purification by flash column chromatography using PE/EtOAc as eluent afforded the desired product **62**.

Compound **63** was prepared according to compound **62**, using (*tert*-butoxycarbonyl)-*L*-phenylalanine instead of compound **19**.

Method E: Synthesis of compound **64**.



Step 1: Prepared according to method B. Purification by flash column chromatography using PE/EtOAc (v/v = 50/1) as eluent.

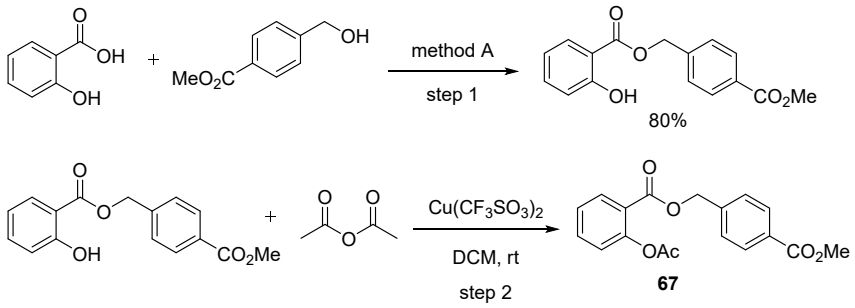
Step 2: Prepared according to method D (step 2) without purification.

Step 3: Prepared according to method D (step 3). Purification by flash column chromatography using PE/EtOAc (v/v = 10/1) as eluent afforded the desired product.

Step 4: Prepared according to method D (step 2) without purification.

Step 5: Prepared according to method D (step 3). Purification by flash column chromatography using PE/EtOAc (v/v = 1/1) as eluent afforded the desired product **64**.

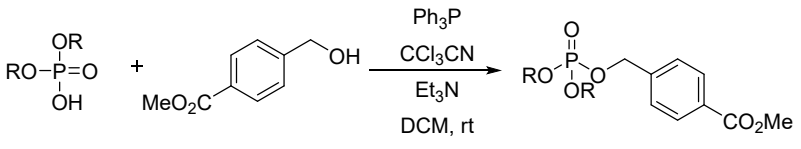
Method F: Synthesis of compound **67**.



Step 1: Prepared according to method A. Purification by flash column chromatography using PE/EtOAc (v/v = 30/1) as eluent.

Step 2: To a dried 10 mL round bottom flask were added 4-(methoxycarbonyl)benzyl 2-hydroxybenzoate (0.29 g, 1.0 mmol, 1.0 eq.), DCM (3.0 mL), and $Cu(CF_3SO_3)_2$ (0.009 g, 0.025 mmol, 0.025 eq.) sequentially. Then, acetic anhydride (0.21 g, 2.0 mmol, 2.0 eq.) was added dropwise. The resulting solution was stirred overnight at room temperature. The solution was diluted with DCM (10 mL) and quenched with saturated aqueous $NaHCO_3$ (10 mL). The aqueous layer was extracted with DCM (3 × 10 mL). The combined DCM layers were washed with brine (3 × 10 mL), dried over Na_2SO_4 and concentrated *in vacuo*. Purification by flash column chromatography using PE/EtOAc (v/v = 50/1) as eluent afforded the desired product **67**.

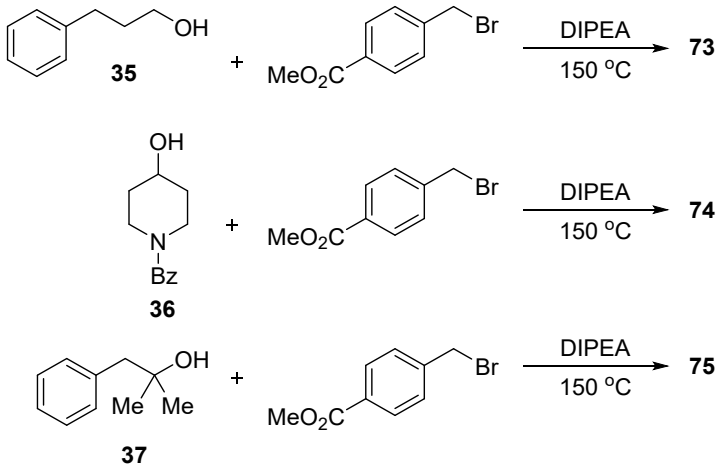
Method G: Synthesis of compounds **70**−**72**.



70, R = binaphthol
71, R = phenol
72, R = benzyl alcohol

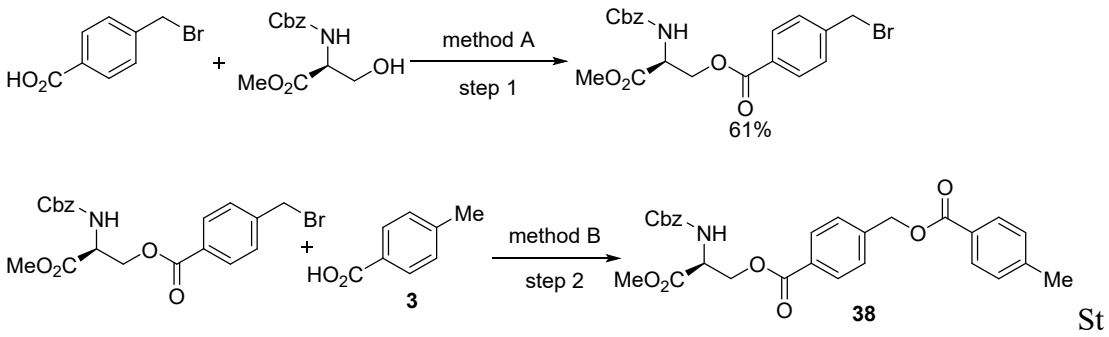
To a dried 25 mL round bottom flask were added corresponding phosphate (1.2 mmol, 1.2 eq.), CCl_3CN (0.43 g, 3.0 mmol, 3.0 eq.), DCM (6 mL) sequentially. Then triphenylphosphine (0.53 g, 2.0 mmol, 2.0 eq.) was added and after the solution was stirred for 30 minutes methyl 4-(hydroxymethyl)benzoate (0.17 g, 1.0 mmol, 1.0 eq.) and Et_3N (0.31 g, 3.0 mmol, 3.0 eq.) were added. The resulting solution was stirred overnight at room temperature. The solution was diluted with DCM (10 mL) and quenched with 1*N* HCl aqueous (10 mL). The aqueous layer was extracted with DCM (3 × 10 mL). The combined DCM layers were washed with brine (3 × 10 mL), dried over Na_2SO_4 and concentrated *in vacuo*. Purification by flash column chromatography using PE/EtOAc as eluent afforded the desired product **70**−**72**.

Method H: Synthesis of compounds **73**−**75**.



To a dried 25 mL round bottom flask were added corresponding alcohol (7.5 mmol, 1.5 eq.), methyl 4-(bromomethyl)benzoate (1.14 g, 5.0 mmol, 1.0 eq.), DIPEA (1.29 g, 10 mmol, 2.0 eq.) sequentially. The resulting mixture was stirred overnight at 150 °C, diluted with DCM (10 mL) and quenched with 1*N* HCl aqueous (10 mL). The aqueous layer was extracted with DCM (3 × 10 mL). The combined DCM layers were washed with brine (3 × 10 mL), dried over Na_2SO_4 and concentrated *in vacuo*. Purification by flash column chromatography using PE/EtOAc as eluent afforded the desired product **73**−**75**.

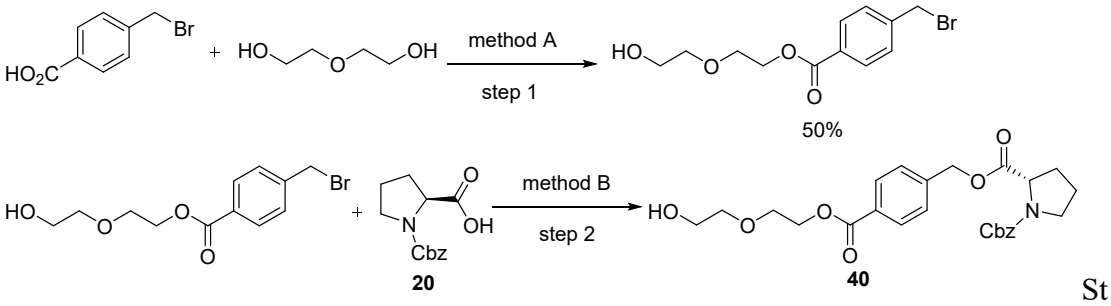
Method I: Synthesis of compound **38**.



Step 1: Prepared according to method A. Purification by flash column chromatography using PE/EtOAc (v/v = 30/1) as eluent.

Step 2: Prepared according to method B. Purification by flash column chromatography using PE/EtOAc (v/v = 10/1) as eluent afforded the desired product **38**.

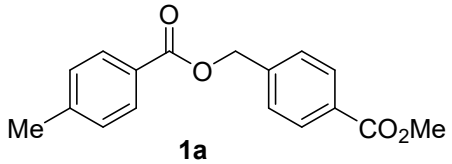
Method J: Synthesis of compound **40**.



Step 1: Prepared according to method A. Purification by flash column chromatography using PE/EtOAc (v/v = 20/1) as eluent.

Step 2: Prepared according to method B. Purification by flash column chromatography using PE/EtOAc (v/v = 1/1) as eluent afforded the desired product **40**.

4-(Methoxycarbonyl)benzyl 4-methylbenzoate (1a)



White solid (483 mg, 85% yield); m.p. 58−59 °C.

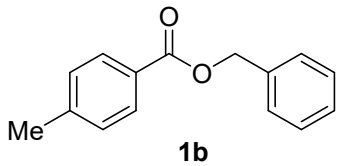
^{1}H NMR (300 MHz, $CDCl_3$): δ 8.06 (d, *J* = 8.5 Hz, 2H), 7.98 (d, *J* = 8.2 Hz, 2H), 7.50 (d, *J* = 8.1 Hz, 2H), 7.26 (dd, *J* = 4.9, 3.6 Hz, 2H), 5.40 (s, 2H), 3.92 (s, 3H), 2.42 (s, 3H).

^{13}C NMR (75 MHz, $CDCl_3$): δ 166.7, 166.3, 143.9, 141.2, 129.8, 129.7, 129.1, 127.5, 127.0, 65.6, 52.1, 21.6.

HRMS (ESI): Calcd. for $C_{17}H_{16}NaO_4^+$ $[M + Na]^+$: 307.0941, found: 307.0945.

Benzyl 4-methylbenzoate (1b)

The ^{1}H spectra data matched with values reported in the literature.[2]



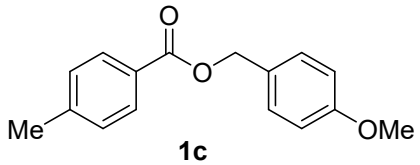
1b

Colorless oil (371 mg, 82% yield).

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.11 – 7.82 (m, 2H), 7.53 – 7.32 (m, 5H), 7.32 – 7.13 (m, 2H), 5.36 (s, 2H), 2.41 (s, 3H).

4-Methoxybenzyl 4-methylbenzoate (1c)

The ^{1}H spectra data matched with values reported in the literature.[3]

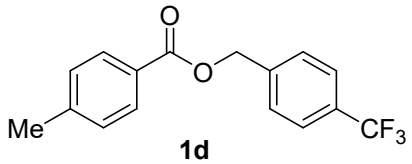


1c

Colorless oil (440 mg, 86% yield).

^{1}H NMR (300 MHz, $CDCl_3$): δ 7.95 (d, J = 8.2 Hz, 2H), 7.39 (d, J = 8.8 Hz, 2H), 7.22 (dd, J = 8.6, 0.6 Hz, 2H), 6.91 (d, J = 8.8 Hz, 2H), 5.28 (s, 2H), 3.82 (s, 3H), 2.40 (s, 3H).

4-(Trifluoromethyl)benzyl 4-methylbenzoate (1d)



1d

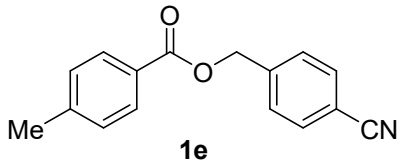
White solid (447 mg, 76% yield); m.p. 89−90 °C.

^{1}H NMR (300 MHz, $CDCl_3$): δ 7.97 (d, J = 8.2 Hz, 2H), 7.77 – 7.64 (m, 2H), 7.63 – 7.47 (m, 2H), 7.34 – 7.15 (m, 2H), 5.40 (s, 2H), 2.42 (s, 3H).

^{13}C NMR (75 MHz, $CDCl_3$): δ 166.2, 143.9, 140.1, 129.6, 129.1, 127.9, 126.9, 125.4 (d, *J* = 3.6 Hz), 122.1, 65.4, 21.5.

HRMS (ESI): Calcd. for $C_{16}H_{14}F_3O_2^+$ $[M + H]^+$: 295.0940, found: 295.0942.

4-Cyanobenzyl 4-methylbenzoate (1e)



1e

Colorless oil (392 mg, 78% yield).

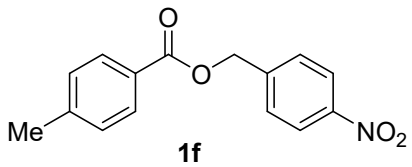
^{1}H NMR (300 MHz, $CDCl_3$): δ 7.98 (d, *J* = 8.2 Hz, 2H), 7.69 (d, *J* = 8.3 Hz, 2H), 7.55 (d, *J* = 8.5 Hz, 2H), 7.27 (t, *J* = 4.0 Hz, 2H), 5.41 (s, 2H), 2.43 (s, 3H).

^{13}C NMR (75 MHz, $CDCl_3$): δ 166.0, 144.1, 141.4, 132.3, 129.6, 129.1, 128.1, 126.6, 118.5, 111.8, 65.1, 21.6.

HRMS (ESI): Calcd. for $C_{16}H_{14}NO_2^+$ $[M + H]^+$: 252.1019, found: 252.1018.

4-Nitrobenzyl 4-methylbenzoate (1f)

The ^{1}H spectra data matched with values reported in the literature.[4]

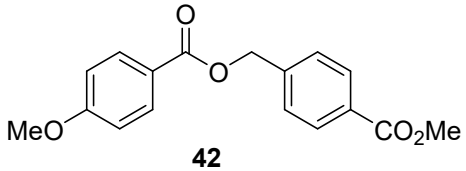


1f

White solid (407 mg, 75 % yield); m.p. 102−103 °C.

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.35 – 8.11 (m, 2H), 8.11 – 7.88 (m, 2H), 7.60 (d, *J* = 8.9 Hz, 2H), 7.26 (dd, *J* = 5.1, 3.4 Hz, 2H), 5.44 (s, 2H), 2.42 (s, 3H).

4-(Methoxycarbonyl)benzyl 4-methoxybenzoate (42)



42

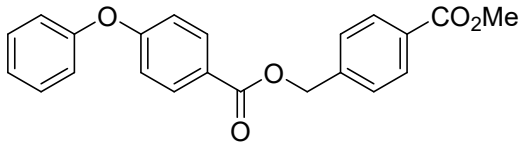
White solid (504 mg, 84% yield); m.p. 68−69 °C.

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.10 – 8.01 (m, 4H), 7.54 – 7.47 (m, 2H), 6.94 (d, *J* = 8.7 Hz, 2H), 5.40 (s, 2H), 3.93 (s, 3H), 3.87 (s, 3H).

^{13}C NMR (75 MHz, $CDCl_3$): δ 166.7, 165.9, 163.5, 141.3, 131.7, 129.8, 127.5, 122.1, 113.6, 65.5, 55.4, 52.1.

HRMS (ESI): Calcd. for $C_{17}H_{17}O_5^+$ $[M + H]^+$: 301.1071, found: 301.1075.

4-(Methoxycarbonyl)benzyl 4-phenoxybenzoate (43)



43

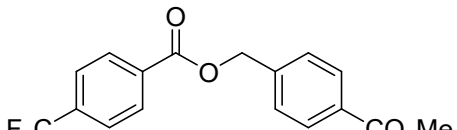
White solid (543 mg, 75% yield); m.p. 49−50 °C.

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.03 (dd, *J* = 8.7, 1.5 Hz, 4H), 7.47 (dd, *J* = 8.1, 0.5 Hz, 2H), 7.42 – 7.30 (m, 2H), 7.21 – 7.12 (m, 1H), 7.07 – 7.01 (m, 2H), 7.00 – 6.94 (m, 2H), 5.37 (s, 2H), 3.90 (s, 3H).

^{13}C NMR (75 MHz, $CDCl_3$): δ 166.6, 165.7, 162.1, 155.5, 141.2, 131.8, 130.0, 129.8, 127.5, 124.5, 124.0, 120.1, 117.3, 65.7, 52.1.

HRMS (ESI): Calcd. for $C_{22}H_{19}O_5^+$ $[M + H]^+$: 363.1227, found: 363.1229.

4-(Methoxycarbonyl)benzyl 4-(trifluoromethyl)benzoate (44)



44

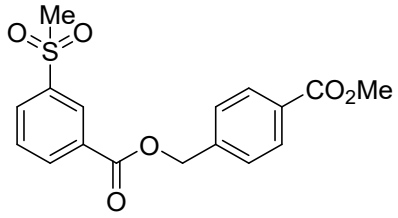
White solid (473 mg, 70% yield); m.p. 80−81 °C.

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.19 (dd, *J* = 8.8, 0.7 Hz, 2H), 8.09 – 8.03 (m, 2H), 7.72 (dd, *J* = 8.8, 0.6 Hz, 2H), 7.51 (dd, *J* = 8.1, 0.5 Hz, 2H), 5.44 (s, 2H), 3.93 (s, 3H).

^{13}C NMR (75 MHz, $CDCl_3$): δ 166.6, 164.9, 140.4, 132.9, 130.1, 130.0, 129.9, 127.7, 125.6, 125.5, 125.4, 125.3, 66.4, 52.2.

HRMS (ESI): Calcd. for $C_{17}H_{14}F_3O_4^+$ $[M + H]^+$: 339.0839, found: 339.0848.

4-(Methoxycarbonyl)benzyl 3-(methylsulfonyl)benzoate (45)



45

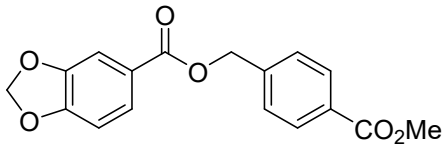
White solid (487 mg, 70% yield); m.p. 116−117 °C.

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.63 (t, *J* = 1.8 Hz, 1H), 8.35 (dt, *J* = 7.8, 1.3 Hz, 1H), 8.19 – 8.11 (m, 1H), 8.11 – 8.00 (m, 2H), 7.69 (t, *J* = 7.8 Hz, 1H), 7.51 (d, *J* = 8.1 Hz, 2H), 5.45 (s, 2H), 3.93 (s, 3H), 3.08 (s, 3H).

^{13}C NMR (75 MHz, $CDCl_3$): δ 166.5, 164.4, 141.3, 140.2, 134.7, 131.6, 131.3, 130.2, 129.9, 129.8, 128.6, 128.0, 66.6, 52.2, 44.4.

HRMS (ESI): Calcd. for $C_{17}H_{17}O_6S^+$ $[M + H]^+$: 349.0740, found: 349.0750.

4-(Methoxycarbonyl)benzyl benzo[*d*][1,3]dioxole-5-carboxylate (46)



46

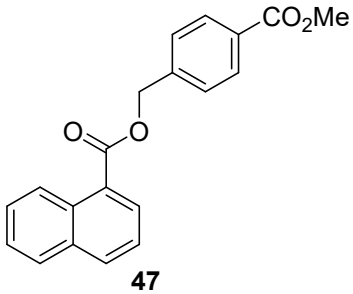
White solid (515 mg, 82% yield); m.p. 80−81 °C.

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.05 (d, *J* = 8.5 Hz, 2H), 7.70 (dd, *J* = 8.2, 1.7 Hz, 1H), 7.49 (dd, *J* = 8.6, 1.1 Hz, 3H), 6.89 – 6.82 (m, 1H), 6.04 (s, 2H), 5.38 (s, 2H), 3.92 (s, 3H).

^{13}C NMR (75 MHz, $CDCl_3$): δ 166.7, 165.5, 151.8, 147.7, 141.1, 129.8, 127.5, 125.5, 123.7, 109.5, 108.0, 101.8, 65.7, 52.1.

HRMS (ESI): Calcd. for $C_{17}H_{15}O_6^+$ $[M + H]^+$: 315.0863, found: 315.0871.

4-(Methoxycarbonyl)benzyl 1-naphthoate (47)



47

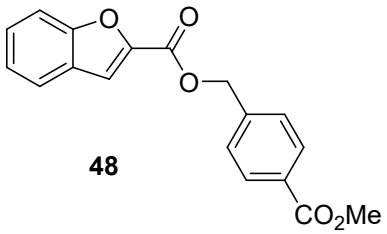
White solid (512 mg, 80% yield); m.p. 81−82 °C.

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.95 (d, *J* = 8.6 Hz, 1H), 8.27 (dd, *J* = 7.3, 1.2 Hz, 1H), 8.12 – 8.03 (m, 3H), 7.93 – 7.88 (m, 1H), 7.66 – 7.47 (m, 5H), 5.52 (s, 2H), 3.94 (s, 3H).

^{13}C NMR (75 MHz, $CDCl_3$): δ 167.0, 166.7, 141.1, 133.8, 133.7, 131.4, 130.4, 129.9, 128.6, 127.9, 127.7, 126.5, 126.3, 125.7, 124.4, 65.9, 52.1.

HRMS (ESI): Calcd. for $C_{20}H_{17}O_4^+$ $[M + H]^+$: 321.1121, found: 321.1114.

4-(Methoxycarbonyl)benzyl benzofuran-2-carboxylate (48)



48

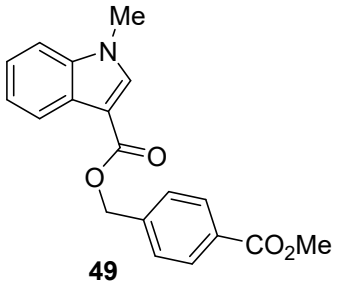
White solid (496 mg, 80% yield); m.p. 122−123 °C.

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.10 – 8.05 (m, 2H), 7.69 (d, *J* = 7.2 Hz, 1H), 7.63 – 7.58 (m, 2H), 7.54 (d, *J* = 8.4 Hz, 2H), 7.50 – 7.43 (m, 1H), 7.35 – 7.28 (m, 1H), 5.47 (s, 2H), 3.93 (s, 3H).

^{13}C NMR (75 MHz, $CDCl_3$): δ 170.6, 166.6, 159.2, 155.8, 145.0, 140.3, 130.1, 129.9, 127.8, 126.8, 123.8, 122.8, 114.5, 112.4, 66.1, 52.2.

HRMS (ESI): Calcd. for $C_{18}H_{15}O_5^+$ $[M + H]^+$: 311.0914, found: 311.0915.

4-(Methoxycarbonyl)benzyl 1-methyl-1*H*-indole-3-carboxylate (49)



49

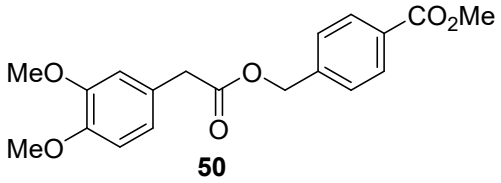
White solid (329 mg, 51% yield); m.p. 101−102 °C.

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.18 – 8.15 (m, 1H), 8.08 – 8.03 (m, 2H), 7.84 (s, 1H), 7.57 – 7.51 (m, 2H), 7.28 – 7.35 (m, 3H), 5.44 (s, 2H), 3.93 (s, 3H), 3.85 (s, 3H).

^{13}C NMR (75 MHz, $CDCl_3$): δ 166.8, 164.5, 142.0, 137.2, 135.5, 129.8, 129.6, 127.5, 126.5, 122.9, 122.0, 121.6, 109.8, 106.4, 64.6, 52.1, 33.4.

HRMS (ESI): Calcd. for $C_{19}H_{18}NO_4^+$ $[M + H]^+$: 324.1230, found: 324.1241.

Methyl 4-((2-(3,4-dimethoxyphenyl)acetoxy)methyl)benzoate (50)



50

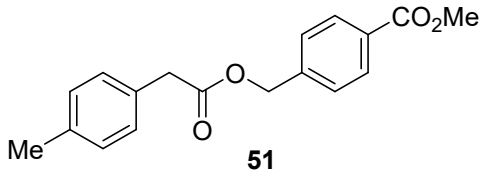
Colorless liquid (482 mg, 70% yield).

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.01 (d, *J* = 8.3 Hz, 2H), 7.36 (d, *J* = 8.3 Hz, 2H), 6.82 (t, *J* = 2.0 Hz, 3H), 5.18 (s, 2H), 3.92 (s, 3H), 3.87 (s, 3H), 3.84 (s, 3H), 3.63 (s, 2H).

^{13}C NMR (75 MHz, $CDCl_3$): δ 171.3, 166.6, 148.9, 148.2, 140.8, 129.8, 129.7, 127.5, 126.1, 121.4, 112.3, 111.2, 65.7, 55.8, 55.8, 52.1, 40.8.

HRMS (ESI): Calcd. for $C_{19}H_{21}O_6^+$ $[M + H]^+$: 345.1333, found: 345.1318.

Methyl 4-((2-(*p*-tolyl)acetoxy)methyl)benzoate (51)



51

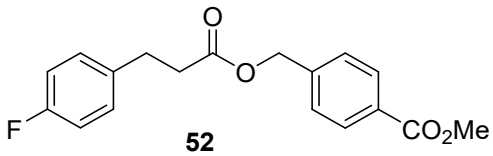
White solid (513 mg, 86% yield); m.p. 46−47 °C.

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.05 – 7.94 (m, 2H), 7.35 (dd, *J* = 8.1, 0.5 Hz, 2H), 7.15 (d, *J* = 5.6 Hz, 4H), 5.17 (s, 2H), 3.92 (s, 3H), 3.65 (s, 2H), 2.34 (s, 3H).

^{13}C NMR (75 MHz, $CDCl_3$): δ 171.4, 166.7, 140.9, 136.8, 130.5, 129.8, 129.7, 129.3, 129.1, 127.5, 65.6, 52.1, 40.8, 21.0.

HRMS (ESI): Calcd. for $C_{18}H_{19}O_4^+$ $[M + H]^+$: 299.1278, found: 299.1265.

Methyl 4-(((3-(4-fluorophenyl)propanoyl)oxy)methyl)benzoate (52)



52

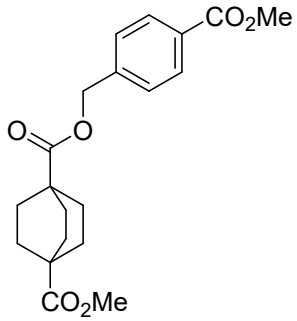
Colorless liquid (556 mg, 88% yield).

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.05 – 7.96 (m, 2H), 7.34 (d, *J* = 8.5 Hz, 2H), 7.19 – 7.07 (m, 2H), 7.02 – 6.88 (m, 2H), 5.14 (s, 2H), 3.92 (s, 3H), 2.94 (t, *J* = 7.6 Hz, 2H), 2.68 (t, *J* = 7.5 Hz, 2H).

^{13}C NMR (75 MHz, $CDCl_3$): δ 172.3, 166.6, 163.1, 159.9, 140.8, 135.8, 129.9, 129.8, 129.7, 129.6, 127.6, 115.4, 115.1, 65.5, 52.1, 35.8, 30.1.

HRMS (ESI): Calcd. for $C_{18}H_{18}FO_4^+$ $[M + H]^+$: 317.1184, found: 317.1177.

1-(4-(Methoxycarbonyl)benzyl) 4-methyl bicyclo[2.2.2]octane-1,4-dicarboxylate (53)



53

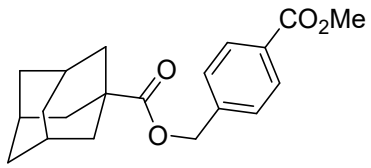
White solid (576 mg, 80% yield); m.p. 104−105°C.

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.03 (d, *J* = 8.1 Hz, 2H), 7.38 (d, *J* = 8.1 Hz, 2H), 5.15 (s, 2H), 3.93 (s, 3H), 3.66 (s, 3H), 1.84 (brs, 12H).

^{13}C NMR (75 MHz, $CDCl_3$): δ 177.6, 176.8, 166.6, 141.2, 129.7, 127.2, 65.2, 52.1, 51.7, 38.6, 38.5, 27.6, 27.6.

HRMS (ESI): Calcd. for $C_{20}H_{25}O_6^+$ $[M + H]^+$: 361.1646, found: 361.1630.

4-(Methoxycarbonyl)benzyl (1*r*,3*R*,5*S*)-adamantane-1-carboxylate (54)



54

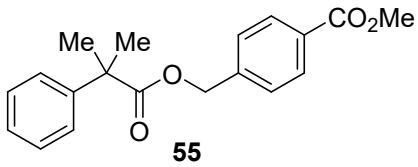
Colorless liquid (558 mg, 85% yield).

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.03 (d, *J* = 8.2 Hz, 2H), 7.47 – 7.33 (m, 2H), 5.15 (s, 2H), 3.91 (s, 3H), 2.03 (s, 3H), 1.93 (brs, 6H), 1.72 (brs, 6H).

^{13}C NMR (75 MHz, $CDCl_3$): δ 177.2, 166.7, 141.6, 129.7, 129.6, 127.2, 64.9, 52.1, 40.7, 38.8, 36.4, 27.9.

HRMS (ESI): Calcd. for $C_{20}H_{25}O_4^+$ $[M + H]^+$: 329.1747, found: 329.1740.

Methyl 4-(((2-methyl-2-phenylpropanoyl)oxy)methyl)benzoate (55)



55

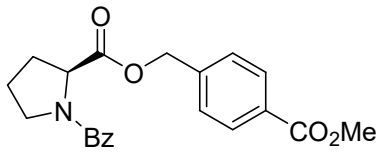
Colorless liquid (487 mg, 78% yield).

^{1}H NMR (300 MHz, $CDCl_3$): δ 7.95 (d, *J* = 8.4 Hz, 2H), 7.33 (dd, *J* = 4.0, 0.9 Hz, 4H), 7.21 (d, *J* = 8.6 Hz, 3H), 5.15 (s, 2H), 3.91 (s, 3H), 1.62 (s, 6H).

^{13}C NMR (75 MHz, $CDCl_3$): δ 176.3, 166.7, 144.2, 141.2, 129.6, 129.5, 128.4, 127.1, 126.8, 125.6, 65.5, 52.1, 46.5, 26.3.

HRMS (ESI): Calcd. for $C_{19}H_{20}NaO_4^+$ $[M + Na]^+$: 335.1254, found: 335.1239.

4-(Methoxycarbonyl)benzyl benzoyl-*L*-prolinate (56)



56

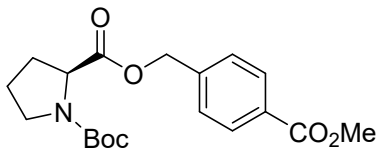
White solid (294 mg, 80% yield); m.p. 83−84 °C.

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.03 (d, *J* = 8.3 Hz, 2H), 7.58 – 7.28 (m, 7H), 5.33 – 4.92 (m, 2H), 4.80 – 4.36 (m, 1H), 3.92 (s, 3H), 3.87 – 3.50 (m, 2H), 2.31 – 2.36 (m, 1H), 2.13 – 1.85 (m, 3H).

^{13}C NMR (75 MHz, $CDCl_3$): δ 173.2, 173.1, 171.8, 166.5, 166.2, 140.8, 140.7, 140.2, 130.0, 129.8, 129.7, 128.2, 127.6, 127.5, 127.2, 127.1, 65.9, 59.2, 51.9, 49.7, 48.9, 46.6, 33.8, 29.3, 25.3, 24.8.

HRMS (ESI): Calcd. for $C_{21}H_{22}NO_5^+$ $[M + H]^+$: 368.1492, found: 368.1487.

1-(*tert*-Butyl) 2-(4-(methoxycarbonyl)benzyl) (*S*)-pyrrolidine-1,2-dicarboxylate (57)



57

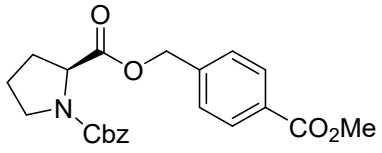
Colorless liquid (298 mg, 82% yield).

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.02 (dd, *J* = 8.3, 4.9 Hz, 2H), 7.41 (d, *J* = 8.1 Hz, 2H), 5.34 – 5.08 (m, 2H), 4.29 – 4.36 (m, 1H), 3.92 (s , 3H), 3.62 – 3.29 (m, 2H), 2.31 – 2.11 (m, 1H), 1.99 – 1.79 (m, 3H), 1.46 (s, 4H), 1.34 (s, 5H).

^{13}C NMR (75 MHz, $CDCl_3$): δ 172.8, 172.6, 166.5, 140.8, 140.5, 129.9, 129.7, 129.6, 127.7, 127.4, 79.8, 79.7, 65.7, 59.0, 58.7, 52.1, 46.5, 46.2, 30.8, 29.8, 28.3, 28.2, 24.3, 23.5.

HRMS (ESI): Calcd. for $C_{19}H_{26}NO_6^+$ $[M + H]^+$: 364.1755, found: 364.1743.

1-Benzyl 2-(4-(methoxycarbonyl)benzyl) (*S*)-pyrrolidine-1,2-dicarboxylate (58)



58

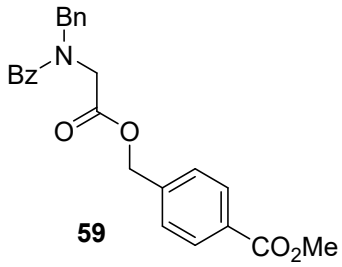
Colorless liquid (345 mg, 87% yield).

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.00 (dd, *J* = 17.9, 8.2 Hz, 2H), 7.43 – 7.23 (m, 7H), 5.29 – 4.97 (m, 4H), 4.49 – 4.37 (m, 1H), 3.93 (s, 3H), 3.69 – 3.44 (m, 2H), 2.20 – 2.31 (m, 1H), 2.10 – 1.82 (m, 3H).

^{13}C NMR (75 MHz, $CDCl_3$): δ 172.4, 172.3, 166.6, 166.5, 154.8, 154.1, 140.7, 140.4, 136.5, 136.4, 129.9, 129.7, 128.4, 128.3, 127.9, 127.8, 127.7, 127.5, 127.4, 66.9, 66.8, 65.9, 65.8, 59.2, 58.8, 52.1, 46.9, 46.4, 30.8, 29.8, 24.3, 23.5.

HRMS (ESI): Calcd. for $C_{22}H_{24}NO_6^+$ $[M + H]^+$: 398.1598, found: 398.1585.

Methyl 4-(((*N*-benzoyl-*N*-benzylglycyl)oxy)methyl)benzoate (59)



59

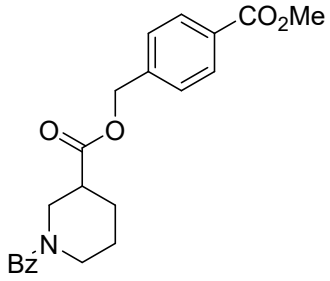
Colorless liquid (650 mg, 78% yield).

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.04 (d, *J* = 8.2 Hz, 2H), 7.51 (d, *J* = 7.3 Hz, 1.5H), 7.45 – 7.28 (m, 9H), 7.19 (d, *J* = 6.9 Hz, 1.5H), 5.25 (s, 1.5H), 5.15 (s, 0.5H), 4.82 (s, 0.5H), 4.63 (s, 1.5H), 4.22 (s, 1.5H), 3.92 (s, 3.5H).

^{13}C NMR (75 MHz, $CDCl_3$): δ 172.5, 168.7, 166.5, 140.4, 140.0, 136.1, 135.8, 135.1, 130.0, 129.8, 128.9, 128.7, 128.5, 127.8, 127.6, 126.9, 126.8, 126.5, 66.1, 53.8, 52.1, 49.8, 49.1, 46.4.

HRMS (ESI): Calcd. for $C_{25}H_{24}NO_5^+$ $[M + H]^+$: 418.1649, found: 418.1646.

4-(Methoxycarbonyl)benzyl 1-benzoylpiperidine-3-carboxylate (60)



60

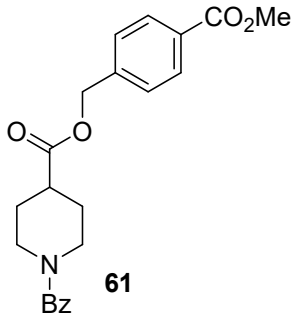
Colorless liquid (609 mg, 80% yield).

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.02 (d, *J* = 8.1 Hz, 2H), 7.47 – 7.30 (m, 7H), 5.17 (s, 2H), 4.39 – 4.65 (m, 0.5H), 3.92 (s, 3H), 3.67 (s, 0.5H), 3.02 – 3.11 (m, 2H), 2.68 (s, 1H), 2.13 – 2.16 (m, 1H), 1.79 – 1.83 (m, 3H), 1.57 (s, 1H).

^{13}C NMR (75 MHz, $CDCl_3$): δ 172.3, 170.5, 166.5, 140.6, 135.7, 129.9, 129.7, 129.6, 128.4, 127.5, 126.8, 65.5, 60.3, 52.1, 41.6, 41.4, 41.1, 27.3, 24.7, 24.1.

HRMS (ESI): Calcd. for $C_{22}H_{24}NO_5^+$ $[M + H]^+$: 382.1649, found: 382.1634.

4-(Methoxycarbonyl)benzyl 1-benzoylpiperidine-4-carboxylate (61)



61

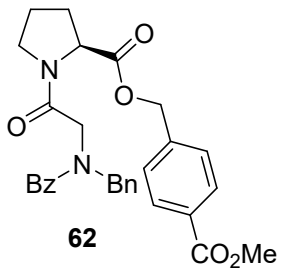
White solid (615 mg, 82% yield); m.p. 84−85 °C.

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.08 – 7.99 (m, 2H), 7.43 – 7.33 (m, 7H), 5.19 (s, 2H), 4.52 (s, 1H), 3.92 (s, 3H), 3.72 (s, 1H), 3.04 (s, 2H), 2.61 – 2.66 (m, 1H), 1.73 (s, 4H).

^{13}C NMR (75 MHz, $CDCl_3$): δ 173.6, 170.3, 166.5, 140.7, 135.8, 129.9, 129.8, 129.6, 128.4, 127.5, 126.7, 65.5, 52.1, 46.8, 40.9, 28.1.

HRMS (ESI): Calcd. for $C_{22}H_{24}NO_5^+$ $[M + H]^+$: 382.1649, found: 382.1634.

4-(Methoxycarbonyl)benzyl *N*-benzoyl-*N*-benzylglycyl-*L*-prolinate (62)



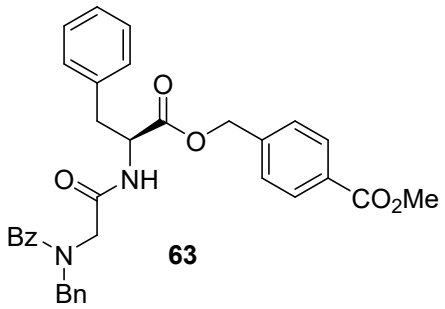
Colorless liquid (388 mg, 75% yield).

^{1}H NMR (300 MHz, $CDCl_3$): δ 7.98 – 8.06 (m, 2H), 7.53 (d, *J* = 7.7 Hz, 1.5H), 7.48 – 7.30 (m, 9H), 7.19 (d, *J* = 6.8 Hz, 1.5H), 5.33 – 4.35 (m, 6H), 3.86 – 3.91 (m, 4H), 3.02 – 3.57 (m, 2H), 2.21 (d, *J* = 3.7 Hz, 1H), 2.10 – 1.75 (m, 3H).

^{13}C NMR (75 MHz, $CDCl_3$): δ 173.3, 173.2, 172.2, 171.4, 171.1, 170.9, 140.3, 136.1, 135.1, 129.5, 129.5, 129.4, 128.5, 128.3, 128.1, 127.4, 127.3, 127.2, 126.8, 126.6, 65.7, 65.6, 58.6, 53.2, 51.7, 49.2, 48.5, 45.9, 45.6, 45.5, 28.5, 28.4, 24.4, 24.3.

HRMS (ESI): Calcd. for $C_{30}H_{31}N_2O_6^+$ $[M + H]^+$: 515.2177, found: 515.2151.

Methyl 4-(((*N*-benzoyl-*N*-benzylglycyl-*L*-phenylalanyl)oxy)methyl)benzoate (63)



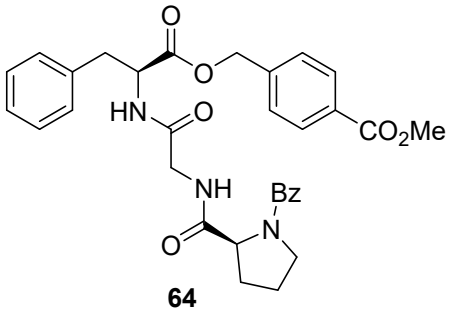
White solid (451 mg, 80% yield); m.p. 82−83 °C.

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.02 (d, *J* = 8.0 Hz, 2H), 7.48 – 7.28 (m, 11H), 7.28 – 6.82 (m, 7H), 5.28 – 5.09 (m, 2H), 4.93 (s, 1H), 4.49 (s, 2H), 4.17 – 3.98 (m, 1.5H), 3.93 (s, 3H), 3.68 – 3.76 (m, 0.5H), 3.16 – 3.23 (m, 2H).

^{13}C NMR (75 MHz, $CDCl_3$): δ 172.8, 170.9, 168.3, 166.5, 139.9, 135.5, 134.8, 130.1, 129.8, 129.2, 128.8, 128.6, 128.5, 127.9, 127.8, 127.1, 127.05, 127.0, 126.9, 126.8, 66.4, 53.7, 53.1, 52.1, 48.7, 37.7.

HRMS (ESI): Calcd. for $C_{34}H_{33}N_2O_6^+$ $[M + H]^+$: 565.2333, found: 565.2306.

Methyl 4-(((benzoyl-*L*-prolylglycyl-*L*-phenylalanyl)oxy)methyl)benzoate (64)



64

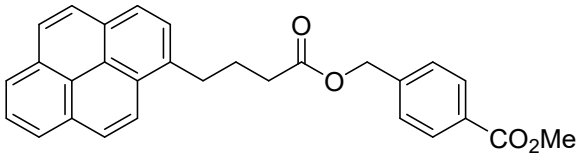
White solid (336 mg, 65% yield); m.p. 65−66 °C.

^{1}H NMR (300 MHz, $CDCl_3$): δ 7.97 (d, *J* = 8.1 Hz, 2H), 7.50 (s, 2H), 7.29 – 7.40 (m, 4H), 7.24 – 6.96 (m, 8H), 5.19 – 4.99 (m, 2H), 4.94 – 4.76 (m, 1H), 4.57 (s, 1H), 4.06 – 4.11 (m, 1H), 3.92 (s, 3H), 3.87 – 3.44 (m, 3H), 3.03 – 3.08 (m, 2H), 2.37 – 2.09 (m, 2H), 1.80 (s, 2H).

^{13}C NMR (75 MHz, $CDCl_3$): δ 172.1, 171.9, 171.5, 171.1, 169.1, 169.0, 166.6, 166.5, 140.3, 140.2, 136.2, 136.1, 135.7, 135.6, 130.4, 129.7, 129.2, 129.1, 128.4, 128.3, 128.2, 128.1, 127.6, 127.3, 127.2, 126.8, 126.7, 66.0, 60.9, 60.8, 53.8, 53.7, 52.1, 50.5, 43.0, 37.5, 37.4, 28.6, 28.5, 25.6, 25.5.

HRMS (ESI): Calcd. for $C_{32}H_{34}N_3O_7^+$ $[M + H]^+$: 572.2391, found: 572.2369.

Methyl 4-(((4-(pyren-1-yl)butanoyl)oxy)methyl)benzoate (65)



65

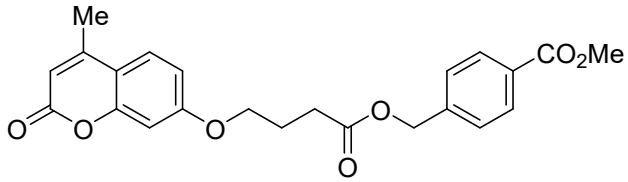
Yellow solid (567 mg, 65% yield); m.p. 91−92 °C.

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.27 (d, *J* = 9.3 Hz, 1H), 8.20 – 7.98 (m, 9H), 7.85 (d, *J* = 7.8 Hz, 1H), 7.40 (d, *J* = 8.2 Hz, 2H), 5.17 (s, 2H), 3.92 (s, 3H), 3.49 – 3.31 (m, 2H), 2.54 (t, *J* = 7.3 Hz, 2H), 2.31 – 2.10 (m, 2H).

^{13}C NMR (75 MHz, $CDCl_3$): δ 173.0, 166.6, 140.9, 135.4, 131.3, 130.8, 129.9, 129.8, 129.7, 128.7, 127.6, 127.4, 127.4, 127.3, 126.7, 125.8, 125.0, 124.9, 124.8, 123.2, 65.4, 52.1, 33.7, 32.6, 26.6.

HRMS (ESI): Calcd. for $C_{29}H_{24}NaO_4^+$ $[M + Na]^+$: 459.1567, found: 459.1553.

Methyl 4-(((4-((4-methyl-2-oxo-2*H*-chromen-7-yl)oxy)butanoyl)oxy)methyl)benzoate (66)



66

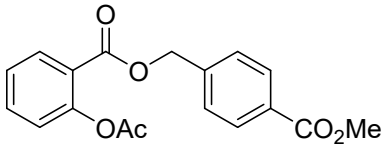
White solid (558 mg, 68% yield); m.p. 75−76 °C.

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.00 – 7.84 (m, 2H), 7.36 (dd, *J* = 21.2, 8.6 Hz, 3H), 6.72 (dt, *J* = 7.4, 2.4 Hz, 2H), 6.05 (d, *J* = 1.2 Hz, 1H), 5.11 (s, 2H), 3.99 (t, *J* = 6.1 Hz, 2H), 3.84 (s, 3H), 2.54 (t, *J* = 7.2 Hz, 2H), 2.31 (d, *J* = 1.2 Hz, 3H), 2.16 – 1.98 (m, 2H).

^{13}C NMR (75 MHz, $CDCl_3$): δ 172.5, 166.6, 161.6, 161.1, 155.1, 152.4, 140.8, 129.9, 129.8, 127.6, 125.5, 113.6, 112.3, 111.9, 101.4, 67.1, 65.5, 52.1, 30.5, 24.3, 18.6.

HRMS (ESI): Calcd. for $C_{23}H_{23}O_7^+$ $[M + H]^+$: 411.1438, found: 411.1423.

4-(Methoxycarbonyl)benzyl 2-acetoxybenzoate (67)



67

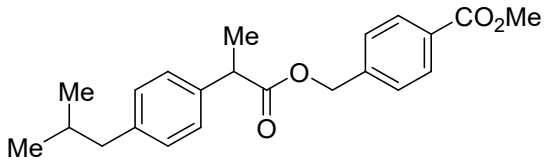
Colorless liquid (246 mg, 75% yield).

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.15 – 7.99 (m, 3H), 7.59 (ddd, *J* = 8.1, 7.5, 1.7 Hz, 1H), 7.49 (d, *J* = 8.5 Hz, 2H), 7.37 – 7.30 (m, 1H), 7.11 (dd, *J* = 8.1, 1.0 Hz, 1H), 5.36 (s, 2H), 3.93 (s, 3H), 2.18 (s, 3H).

^{13}C NMR (75 MHz, $CDCl_3$): δ 169.5, 166.5, 164.1, 150.6, 140.5, 134.1, 131.8, 130.0, 129.8, 127.8, 126.0, 123.8, 122.8, 66.1, 52.1, 20.7.

HRMS (ESI): Calcd. for $C_{18}H_{17}O_6^+$ $[M + H]^+$: 329.1020, found: 329.1008.

Methyl 4-(((2-(4-isobutylphenyl)propanoyl)oxy)methyl)benzoate (68)



68

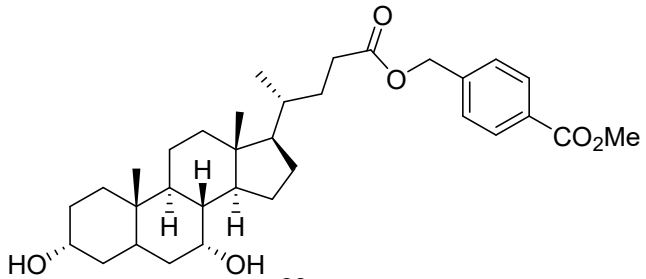
Colorless liquid (609 mg, 86% yield).

^{1}H NMR (300 MHz, $CDCl_3$): δ 7.99 – 7.90 (m, 2H), 7.26 – 7.18 (m, 4H), 7.09 (d, *J* = 8.2 Hz, 2H), 5.15 (d, *J* = 3.1 Hz, 2H), 3.91 (s, 3H), 3.77 (q, *J* = 7.2 Hz, 1H), 2.46 (d, *J* = 7.2 Hz, 2H), 1.85 (dt, *J* = 13.7, 6.9 Hz, 1H), 1.52 (d, *J* = 7.2 Hz, 3H), 0.90 (d, *J* = 6.6 Hz, 6H).

^{13}C NMR (75 MHz, $CDCl_3$): δ 174.2, 166.6, 141.1, 140.6, 137.3, 129.6, 129.6, 129.3, 127.1, 65.4, 52.0, 45.0, 44.9, 30.1, 22.3, 18.2.

HRMS (ESI): Calcd. for $C_{22}H_{27}O_4^+$ [M + H]$^+$: 355.1904, found: 355.1897.

Methyl 4-((((4*R*)-4-((3*R*,7*R*,8*R*,9*S*,10*S*,13*R*,14*S*,17*R*)-3,7-dihydroxy-10,13-dimethylhexadecahydro-1*H*-cyclopenta[*a*]phenanthren-17-yl)pentanoyl)oxy)methyl)benzoate (69)



69

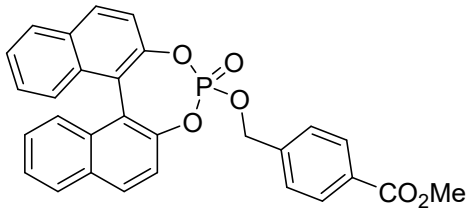
White solid (950 mg, 88% yield); m.p. 71−73 °C.

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.03 (d, *J* = 8.5 Hz, 2H), 7.41 (d, *J* = 8.5 Hz, 2H), 5.15 (s, 2H), 3.91 (s, 3H), 3.84 (s, 1H), 3.46 (s, 1H), 2.50 – 2.12 (m, 3H), 2.06 – 1.75 (m, 6H), 1.72 – 1.59 (m, 5H), 1.53 – 1.21 (m, 10H), 1.21 – 0.96 (m, 4H), 0.93 – 0.88 (m, 6H), 0.63 (s, 3H).

^{13}C NMR (75 MHz, $CDCl_3$): δ 173.8, 166.7, 141.1, 129.7, 127.6, 71.9, 68.4, 65.2, 55.6, 52.1, 50.4, 42.6, 41.4, 39.8, 39.5, 39.3, 35.3, 35.2, 34.9, 34.5, 32.7, 31.1, 30.8, 30.6, 28.1, 23.6, 22.7, 20.5, 18.2, 11.7.

HRMS (ESI): Calcd. for $C_{33}H_{49}O_6^+$ [M + H]$^+$: 541.3524, found: 541.3532.

Methyl 4-(((4-oxidodinaphtho[2,1-*d*:1',2'-*f*][1,3,2]dioxaphosphepin-4-yl)oxy) methyl)benzoate (70)



70

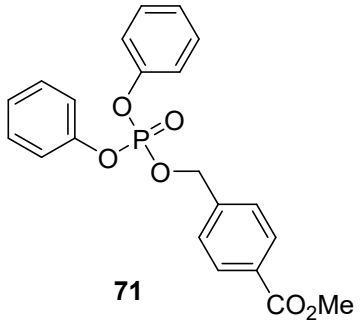
Yellow solid (352 mg, 71% yield); m.p. 77−78 °C.

^{1}H NMR (300 MHz, $CDCl_3$): δ 7.97 – 8.02 (m, 5H), 7.72 – 7.42 (m, 6H), 7.42 – 7.29 (m, 4H), 7.20 – 7.22 (m, 1H), 5.29 – 5.46 (m, 2H), 3.94 (s, 3H).

^{13}C NMR (75 MHz, $CDCl_3$): δ 166.5, 147.2 (d, *J* = 11.1 Hz), 146.1 (d, *J* = 8.4 Hz), 139.9 (d, *J* = 5.9 Hz), 132.2 (d, *J* = 5.5 Hz), 131.9 (d, *J* = 8.3 Hz), 131.5 (d, *J* = 7.3 Hz), 131.1, 130.5, 129.9, 128.4 (d, *J* = 6.0 Hz), 127.6, 127.1, 126.9 (d, *J* = 10.0 Hz), 125.8, 121.2 (d, *J* = 13.6 Hz), 120.3 (d, *J* = 38.6 Hz), 69.9 (dt, *J* = 5.3, 2.7 Hz), 52.2 (d, *J* = 2.2 Hz).

HRMS (ESI): Calcd. for $C_{29}H_{22}O_6P^+$ $[M + H]^+$: 497.1149, found: 497.1128.

Methyl 4-(((diphenoxyphosphoryl)oxy)methyl)benzoate (71)



71

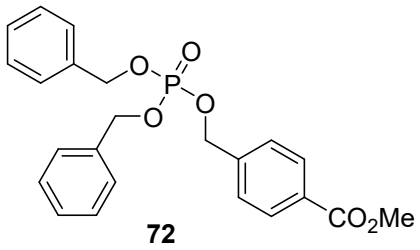
White solid (260 mg, 66% yield); m.p. 69−70 °C.

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.00 – 7.96 (m, 2H), 7.40 – 7.26 (m, 6H), 7.23 – 7.13 (m, 6H), 5.28 (d, *J* = 8.5 Hz, 2H), 3.91 (s, 3H).

^{13}C NMR (75 MHz, $CDCl_3$): δ 166.5, 150.4 (d, *J* = 6.9 Hz), 140.0 (d, *J* = 6.9 Hz), 130.3, 129.8 (d, *J* = 5.0 Hz), 129.6, 127.4, 125.4, 120.0 (d, *J* = 4.7 Hz), 69.6 (d, *J* = 5.5 Hz), 52.2 (d, *J* = 1.6 Hz).

HRMS (ESI): Calcd. for $C_{21}H_{20}O_6P^+$ [M + H] $^+$: 399.0992, found: 399.1009.

Methyl 4-(((bis(benzyloxy)phosphoryl)oxy)methyl)benzoate (72)



72

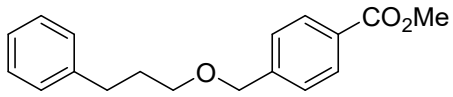
Colorless liquid (302 mg, 71% yield).

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.01 – 7.95 (m, 2H), 7.30 – 7.33 (m, 12H), 5.03 (dd, *J* = 8.2, 3.8 Hz, 6H), 3.92 (s, 3H).

^{13}C NMR (75 MHz, $CDCl_3$): δ 166.6, 140.7 (d, *J* = 7.1 Hz), 138.1, 135.6 (d, *J* = 6.8), 130.0, 129.7, 128.5, 127.9, 127.2, 69.4 (d, *J* = 5.5 Hz), 68.3 (d, *J* = 5.3 Hz), 52.1.

HRMS (ESI): Calcd. for $C_{23}H_{24}O_6P^+$ [M + H] $^+$: 427.1305, found: 427.1285.

Methyl 4-((3-phenylpropoxy)methyl)benzoate (73)



73

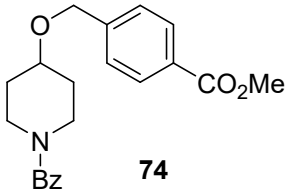
Colorless liquid (1.1 g, 78% yield).

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.02 (d, *J* = 8.5 Hz, 2H), 7.41 (dd, *J* = 8.0, 0.6 Hz, 2H), 7.33 – 7.24 (m, 2H), 7.24 – 7.14 (m, 3H), 4.56 (s, 2H), 3.92 (s, 3H), 3.51 (t, *J* = 6.4 Hz, 2H), 2.80 – 2.65 (m, 2H), 2.08 – 1.87 (m, 2H).

^{13}C NMR (75 MHz, $CDCl_3$): δ 166.9, 143.9, 141.7, 129.6, 129.2, 128.4, 128.3, 127.1, 125.7, 72.2, 69.8, 52.0, 32.3, 31.3.

HRMS (ESI): Calcd. for $C_{18}H_{21}O_3^+$ [M + H] $^+$: 285.1485, found: 285.1476.

Methyl 4-(((1-benzoylpiperidin-4-yl)oxy)methyl)benzoate (74)



74

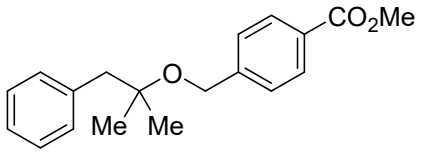
Yellow solid (635 mg, 36% yield); m.p. 65−66 °C.

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.08 – 7.99 (m, 2H), 7.39 – 7.42 (m, 7H), 4.62 (s, 2H), 4.06 (s, 1H), 3.91 (s, 3H), 3.79 – 3.14 (m, 4H), 1.59 – 1.83 (m, 4H).

^{13}C NMR (75 MHz, $CDCl_3$): δ 170.3, 166.8, 143.7, 136.0, 129.6, 129.5, 129.3, 128.4, 126.9, 126.7, 73.8, 69.4, 51.9, 44.6, 39.2, 31.5, 30.5.

HRMS (ESI): Calcd. for $C_{21}H_{24}NO_4^+$ $[M + H]^+$: 354.1700, found: 354.1683.

Methyl 4-(((2-methyl-1-phenylpropan-2-yl)oxy)methyl)benzoate (75)



75

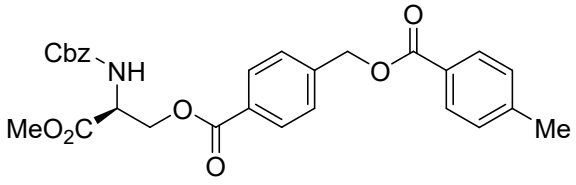
Colorless liquid (462 mg, 31% yield).

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.00 (d, *J* = 8.2 Hz, 2H), 7.40 (d, *J* = 8.1 Hz, 2H), 7.31 – 7.16 (m, 5H), 4.58 (s, 2H), 3.92 (s, 3H), 2.89 (s, 2H), 1.25 (s, 6H).

^{13}C NMR (75 MHz, $CDCl_3$): δ 161.9, 140.1, 132.9, 125.4, 124.4, 123.6, 122.6, 121.5, 120.9, 70.8, 58.1, 46.8, 42.2, 20.0.

HRMS (ESI): Calcd. for $C_{19}H_{23}O_3^+$ $[M + H]^+$: 299.1642, found: 299.1636.

(*S*)-4-((2-(((Benzyloxy)carbonyl)amino)-3-methoxy-3-oxopropoxy)carbonyl)benzyl 4-methylbenzoate (38)



38

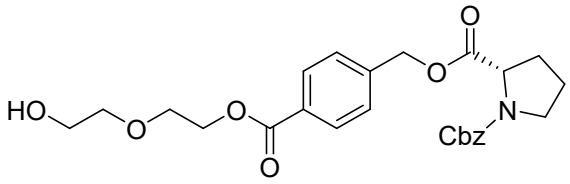
White solid (394 mg, 78% yield); m.p. 56−57 °C.

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.03 – 7.93 (m, 4H), 7.49 (d, *J* = 8.1 Hz, 2H), 7.39 – 7.31 (m, 5H), 7.27 – 7.20 (m, 2H), 5.63 – 5.64 (m, 1H), 5.40 (s, 2H), 5.13 (s, 2H), 4.76 – 4.77 (m, 1H), 4.71 – 4.57 (m, 2H), 3.79 (s, 3H), 2.42 (s, 3H).

^{13}C NMR (75 MHz, $CDCl_3$): δ 169.8, 166.2, 165.5, 155.6, 143.9, 141.8, 135.9, 129.9, 129.6, 129.1, 128.9, 128.5, 128.2, 128.1, 127.5, 126.9, 67.2, 65.5, 64.6, 53.4, 52.8, 21.6.

HRMS (ESI): Calcd. for $C_{28}H_{28}NO_8^+$ $[M + H]^+$: 506.1809, found: 506.1819.

1-Benzyl 2-(4-((2-(2-hydroxyethoxy)ethoxy)carbonyl)benzyl) (*S*)-pyrrolidine-1,2-dicarboxylate (40)



40

Colorless liquid (268 mg, 57% yield).

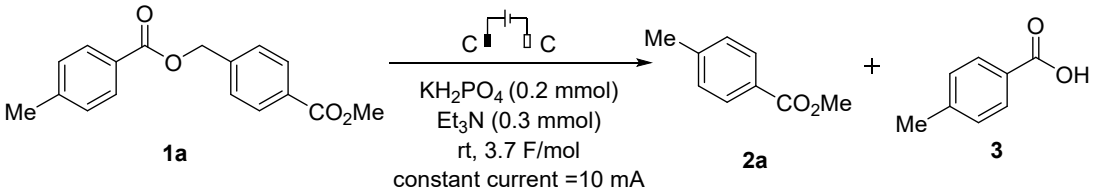
^{1}H NMR (300 MHz, $CDCl_3$): δ 8.01 (dd, *J* = 17.2, 8.3 Hz, 2H), 7.45 – 7.23 (m, 7H), 5.30 – 4.94 (m, 4H), 4.52 – 4.34 (m, 3H), 3.85 (dd, *J* = 5.5, 3.9 Hz, 2H), 3.75 (d, *J* = 4.6 Hz, 2H), 3.70 – 3.47 (m, 4H), 2.31 – 2.16 (m, 1H), 2.10 – 1.85 (m, 4H).

^{13}C NMR (75 MHz, $CDCl_3$): δ 172.4, 172.3, 166.1, 166.0, 154.2, 140.9, 140.6, 136.4, 129.8, 129.7, 129.6, 128.4, 128.3, 127.9, 127.8, 127.7, 127.5, 127.4, 72.3, 69.1, 67.0, 66.9, 65.9, 65.8, 64.0, 61.7, 59.2, 58.8, 46.9, 46.4, 30.9, 29.8, 24.3, 23.5.

HRMS (ESI): Calcd. for $C_{25}H_{30}NO_8^+$ $[M + H]^+$: 472.1966, found: 472.1945.

III. Investigation of Reaction Conditions

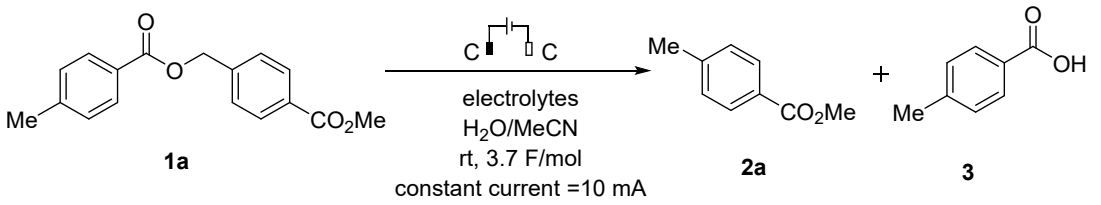
Table S1. Investigation of solvents[a]:



entry	solvents (mL)	**2a** yield (%)[b]	**3** yield (%)[b]
1	H_2O/DCM = (0.1/3)	0	0
2	H_2O/MeCN = (0.1/3)	0	0
3	H_2O/DMSO = (0.1/3)	0	0
4	H_2O/MeCN = (0.75/2.25)	10	15
5	H_2O/DMSO = (0.75/2.25)	0	0

[a]Reaction conditions: graphite rod anode (d = 5 mm), graphite rod cathode (d = 5 mm), **1a** (0.2 mmol), Et_3N (0.3 mmol), solvents, undivided cell, constant current = 10 mA, rt, 4.0 h (3.7 F/mol).[b]Isolated yield.

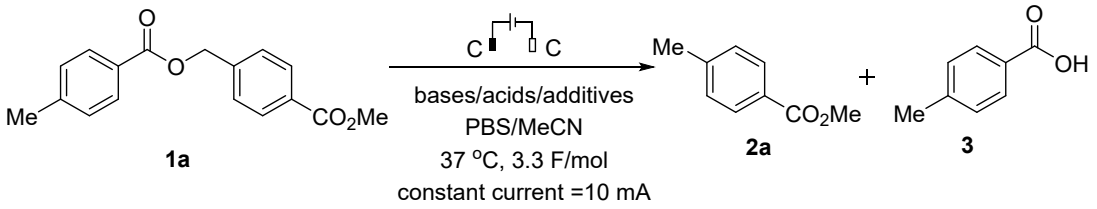
Table S2. Investigation of electrolytes[a]:



entry	variation of electrolytes	**2a** yield (%)[b]	**3** yield (%)[b]
1	KH_2PO_4 (0.3 mmol)	trace	trace
2	K_3PO_4 (0.3 mmol)	trace	trace
3	KOAc (0.3 mmol)	trace	trace
4	PBS (1.5 mL)[c]	14	51
5	PBS (1.5 mL)[d]	trace	trace

[a]Reaction conditions: graphite rod anode (d = 5 mm), graphite rod cathode (d = 5 mm), **1a** (0.2 mmol), H_2O/MeCN (v/v = 1/3, 3.0 mL), electrolytes, undivided cell, constant current = 10 mA, rt, 4.0 h (3.7 F/mol). [b]Isolated yield. [c] PBS/MeCN (v/v = 1/1, 3.0 mL) instead of H_2O/MeCN (v/v = 1/3, 3.0 mL). [d] PBS/DMSO (v/v = 1/1, 3.0 mL) instead of H_2O/MeCN (v/v = 1/3, 3.0 mL). PBS: 8.0 mM of Na_2HPO_4, 137 mM of NaCl and 2.0 mM of NaH_2PO_4 in H_2O.

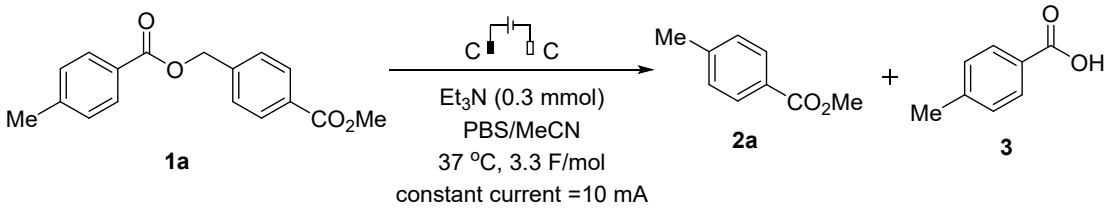
Table S3. Investigation of bases/acids/additives[a]:



entry	variation of bases/acids/additives	**2a** yield (%)[b]	**3** yield (%)[b]
1	Et_3N	97	96
2	pivalic acid	trace	trace
3	acetic acid	trace	trace
4	ascorbic acid	0	0
5	HFIP	trace	trace

[a]Reaction conditions: graphite rod anode (d = 5 mm), graphite rod cathode (d = 5 mm), **1a** (0.2 mmol), PBS/MeCN (v/v = 1/1, 3.0 mL), bases/acids/additives (0.3 mmol), undivided cell, constant current = 10 mA, 37 °C, 3.5 h (3.3 F/mol). [b]Isolated yield. PBS: 8.0 mM of Na_2HPO_4, 137 mM of NaCl and 2.0 mM of NaH_2PO_4 in H_2O.

Table S4. Investigation of other conditions[a]:

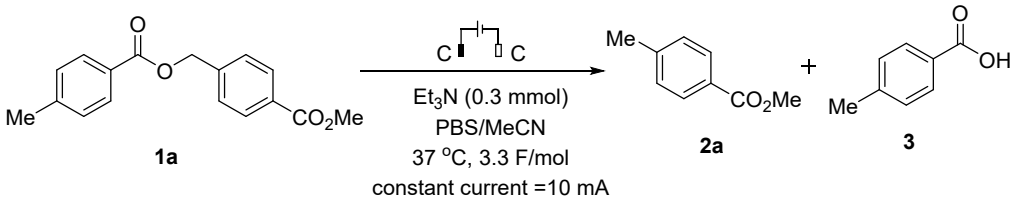


entry	variation from standard conditions	**2a** yield (%)[b]	**3** yield (%)[b]
1	none	97	96
2	no Et_3N	14	51
3	EtOH instead of MeCN	no	no
4	DMSO instead of MeCN	trace	trace
5	I = 5mA instead of I = 10mA	trace	trace
6	I = 7.5mA instead of I = 10mA	80	77
7	no electric current	0	0

[a]Standard conditions: graphite rod anode (d = 5 mm), graphite rod cathode (d = 5 mm), **1a** (0.2 mmol), Et_3N (0.3 mmol), PBS/MeCN (v/v = 1/1, 3.0 mL), undivided cell, constant current = 10 mA, 37 °C, 3.5 h (3.3 F/mol). [b]Isolated yield. PBS: 8.0 mM of Na_2HPO_4, 137 mM of NaCl and 2.0 mM of NaH_2PO_4 in H_2O.

IV. Experimental Procedures and Compound Characterization

General procedure for electrochemical deprotection of pMCB substrates (Procedure A):

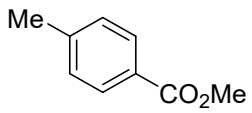


A 10 mL distillation flask equipped with a magnetic stir bar was charged with compound **1a** (0.2 mmol, 1.0 eq.), Et_3N (0.3 mmol) and PBS/MeCN (v/v = 1/1, 3.0 mL). The flask equipped with graphite rod anode (d = 5 mm) and graphite rod cathode (d = 5 mm). The resulting solution was stirred and electrolyzed at a constant current of 10 mA (Single Output DC Power Supply: KRP-305DM) for 3.5 h (3.3 F/mol) at 37 °C. The solution was diluted with DCM (5 mL), and extracted with DCM (3 × 20 mL). The combined organic layers were washed with brine (3 × 20 mL), dried over Na_2SO_4, and concentrated *in vacuo*. Purification by flash column chromatography using PE/EtOAc (v/v = 10/1) as eluent afforded the desired product **2a** and **3**.

Electrochemical setup for 0.2 mmol scale

Methyl 4-methylbenzoate (2a)

The ^1H spectra data matched with values reported in the literature.[5]



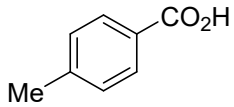
2a

Colorless liquid (29.1 mg, 97% yield).

^{1}H NMR (300 MHz, $CDCl_3$): δ 7.93 (d, *J* = 8.3 Hz, 2H), 7.24 (dd, *J* = 10.0, 2.1 Hz, 2H), 3.90 (s, 3H), 2.40 (s, 3H).

4-Methylbenzoic acid (3)

The ^{1}H spectra data matched with values reported in the literature.[6]



3

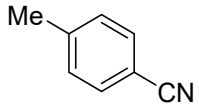
White solid (26.1 mg, 96% yield); m.p. 178−179 °C.

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.00 (d, *J* = 8.3 Hz, 2H), 7.32 – 7.23 (m, 2H), 2.43 (s, 3H).

4-Methylbenzonitrile (2e)

Following Procedure A, using 4.0 h (3.7 F/mol) instead of 3.5 h (3.3 F/mol).

The ^{1}H spectra data matched with values reported in the literature.[7]



2e

Colorless liquid (17.8 mg, 76% yield).

^{1}H NMR (300 MHz, $CDCl_3$): δ 7.55 (d, *J* = 8.3 Hz, 2H), 7.33 – 7.06 (m, 2H), 2.43 (s, 3H).

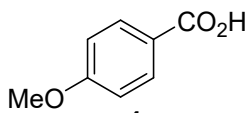
87% Yield of compound **3** (23.7 mg) was obtained.

4-Methoxybenzoic acid (4)

Following Procedure A, using 3.0 h (2.8 F/mol) instead of 3.5 h (3.3 F/mol).

90% Yield of compound **2a** (27.0 mg) was obtained.

The ^{1}H spectra data matched with values reported in the literature.[6]



4

White solid (26.1 mg, 86% yield); m.p. 184−185 °C.

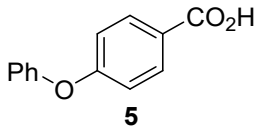
^{1}H NMR (300 MHz, $CDCl_3$): δ 8.13 – 7.99 (m, 2H), 7.01 – 6.88 (m, 2H), 3.88 (s, 3H).

4-Phenoxybenzoic acid (5)

Following Procedure A, using 3.0 h (2.8 F/mol) instead of 3.5 h (3.3 F/mol).

77% Yield of compound **2a** (23.1 mg) was obtained.

The ^{1}H spectra data matched with values reported in the literature.[8]



5

White solid (33.4 mg, 78% yield); m.p. 156−157 °C.

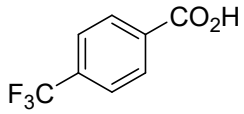
^{1}H NMR (300 MHz, $CDCl_3$): δ 8.12 – 8.05 (m, 2H), 7.41 (dt, *J* = 10.7, 2.1 Hz, 2H), 7.22 (ddd, *J* = 8.4, 4.1, 3.1 Hz, 1H), 7.09 (dt, *J* = 9.0, 1.9 Hz, 2H), 7.05 – 6.97 (m, 2H).

4-(Trifluoromethyl)benzoic acid (6)

Following Procedure A, using 3.0 h (2.8 F/mol) instead of 3.5 h (3.3 F/mol).

80% Yield of compound **2a** (24.0 mg) was obtained.

The ^{1}H spectra data matched with values reported in the literature.[9]



6

White solid (32.3 mg, 85% yield); m.p. 158−160 °C.

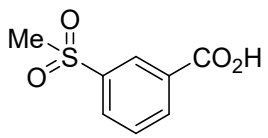
^{1}H NMR (300 MHz, DMSO-d_6): δ 13.48 (s, 1H), 8.13 (d, *J* = 8.0 Hz, 2H), 7.87 (d, *J* = 8.2 Hz, 2H).

3-(Methylsulfonyl)benzoic acid (7)

Following Procedure A, using 2.0 h (1.9 F/mol) instead of 3.5 h (3.3 F/mol).

70% Yield of compound **2a** (21.0 mg) was obtained.

The ^{1}H spectra data matched with values reported in the literature.[10]



7

White solid (29.2 mg, 73% yield); m.p. 227−229 °C.

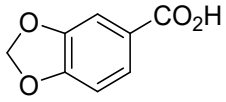
^{1}H NMR (300 MHz, DMSO-d_6): δ 13.55 (s, 1H), 8.40 (t, *J* = 1.6 Hz, 1H), 8.30 – 8.11 (m, 2H), 7.80 (q, *J* = 7.9 Hz, 1H), 3.28 (s, 3H).

Benzo[*d*][1,3]dioxole-5-carboxylic acid (8)

Following Procedure A, using 3.0 h (2.8 F/mol) instead of 3.5 h (3.3 F/mol).

80% Yield of compound **2a** (24.0 mg) was obtained.

The ^{1}H spectra data matched with values reported in the literature.[11]



8

White solid (28.2 mg, 85% yield); m.p. 223−225 °C.

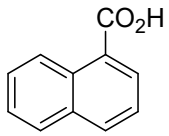
^{1}H NMR (300 MHz, DMSO-d_6): δ 12.75 (s, 1H), 7.54 (dd, J = 8.2, 1.7 Hz, 1H), 7.36 (d, J = 1.6 Hz, 1H), 6.99 (d, J = 8.1 Hz, 1H), 6.12 (s, 2H).

1-Naphthoic acid (9)

Following Procedure A, using 2.0 h (1.9 F/mol) instead of 3.5 h (3.3 F/mol).

80% Yield of compound **2a** (24.0 mg) was obtained.

The ^{1}H spectra data matched with values reported in the literature.[12]



9

White solid (27.5 mg, 80% yield); m.p. 159−160 °C.

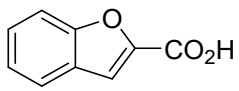
^{1}H NMR (300 MHz, $CDCl_3$): δ 9.11 (d, J = 8.7 Hz, 1H), 8.43 (dd, J = 7.3, 1.3 Hz, 1H), 8.11 (d, J = 8.2 Hz, 1H), 7.93 (d, J = 8.2 Hz, 1H), 7.70 – 7.63 (m, 1H), 7.63 – 7.51 (m, 2H).

Benzofuran-2-carboxylic acid (10)

Following Procedure A, using 0.1 mmol substrate instead of 0.2 mmol and using 2.0 h (3.7 F/mol) instead of 3.5 h (3.3 F/mol).

80% Yield of compound **2a** (12.0 mg) was obtained.

The ^{1}H spectra data matched with values reported in the literature.[13]



10

White solid (12.1 mg, 75% yield); m.p. 193−194 °C.

^{1}H NMR (300 MHz, DMSO-d_6): δ 7.73 – 7.68 (m, 1H), 7.61 (dd, J = 8.3, 0.8 Hz, 1H),

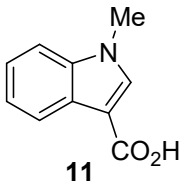
7.39 (ddd, *J* = 8.4, 7.2, 1.4 Hz, 1H), 7.33 (d, *J* = 0.9 Hz, 1H), 7.30 – 7.23 (m, 1H).

1-Methyl-1*H*-indole-3-carboxylic acid (11)

Following Procedure A, using 2.0 h (1.9 F/mol) instead of 3.5 h (3.3 F/mol).

80% Yield of compound **2a** (24.0 mg) was obtained.

The ^{1}H spectra data matched with values reported in the literature.[14]



11

White solid (22.1 mg, 63% yield); m.p. 180−182 °C.

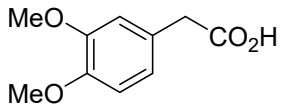
^{1}H NMR (300 MHz, $CDCl_3$): δ 8.29 – 8.17 (m, 1H), 7.88 (s, 1H), 7.43 – 7.29 (m, 3H), 3.86 (s, 3H).

2-(3,4-Dimethoxyphenyl)acetic acid (12)

Following Procedure A, using 3.0 h (2.8 F/mol) instead of 3.5 h (3.3 F/mol).

67% Yield of compound **2a** (20.1 mg) was obtained.

The ^{1}H spectra data matched with values reported in the literature.[15]



12

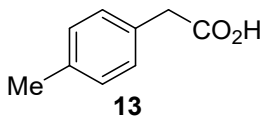
White solid (20.4 mg, 52% yield); m.p. 95−96 °C.

^{1}H NMR (300 MHz, $CDCl_3$): δ 6.82 (t, *J* = 2.7 Hz, 3H), 3.87 (s, 3H), 3.86 (s, 3H), 3.59 (s, 2H).

2-(*p*-Tolyl)acetic acid (13)

Following Procedure A, 95% Yield of compound **2a** (28.5 mg) was obtained.

The ^{1}H spectra data matched with values reported in the literature.[16]



13

White solid (27.0 mg, 90% yield); m.p. 87−89 °C.

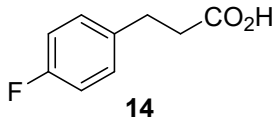
^{1}H NMR (300 MHz, $CDCl_3$): δ 7.22 – 7.06 (m, 4H), 3.61 (s, 2H), 2.34 (s, 3H).

3-(4-Fluorophenyl)propanoic acid (14)

Following Procedure A, using 2.5 h (2.3 F/mol) instead of 3.5 h (3.3 F/mol).

94% Yield of compound **2a** (28.2 mg) was obtained.

The ^{1}H spectra data matched with values reported in the literature.[17]



14

White solid (33.4 mg, 100% yield); m.p. 83−84 °C.

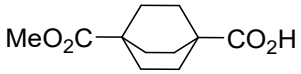
^{1}H NMR (300 MHz, $CDCl_3$): δ 7.21 – 7.07 (m, 2H), 7.03 – 6.89 (m, 2H), 2.93 (t, *J* = 7.6 Hz, 2H), 2.65 (t, *J* = 7.7 Hz, 2H).

4-(Methoxycarbonyl)bicyclo[2.2.2]octane-1-carboxylic acid (15)

Following Procedure A, using 0.1 mmol substrate instead of 0.2 mmol and using 2.0 h (3.7 F/mol) instead of 3.5 h (3.3 F/mol).

94% Yield of compound **2a** (14.1 mg) was obtained.

The ^{1}H spectra data matched with values reported in the literature.[18]



15

White solid (20.1 mg, 95% yield); m.p. 171−173 °C.

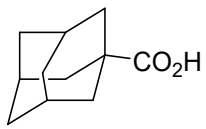
^{1}H NMR (300 MHz, $CDCl_3$): δ 3.65 (s, 3H), 1.82 (brs, 12H).

(1*R*,3*R*,5*S*)-Adamantane-1-carboxylic acid (16)

Following Procedure A, using 2.0 h (1.9 F/mol) instead of 3.5 h (3.3 F/mol).

84% Yield of compound **2a** (25.2 mg) was obtained.

The ^{1}H spectra data matched with values reported in the literature.[19]



16

White solid (31.0 mg, 86% yield); m.p. 171−172 °C.

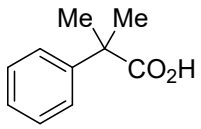
^{1}H NMR (300 MHz, $CDCl_3$): δ 2.02 (s, 3H), 1.91 (d, *J* = 2.9 Hz, 6H), 1.70 (t, *J* = 7.4 Hz, 6H).

2-Methyl-2-phenylpropanoic acid (17)

Following Procedure A, using 3.0 h (2.8 F/mol) instead of 3.5 h (3.3 F/mol).

80% Yield of compound **2a** (24.0 mg) was obtained.

The ^{1}H spectra data matched with values reported in the literature.[20]



17

White solid (24.9 mg, 76% yield); m.p. 78−79 °C.

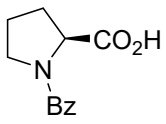
^{1}H NMR (300 MHz, $CDCl_3$): δ 7.53 – 7.24 (m, 5H), 1.60 (s, 6H).

Benzoyl-*L*-proline (18)

Following Procedure A, using 15 mA instead of 10 mA and using 1.5 h (5.6 F/mol) instead of 3.5 h (3.3 F/mol).

80% Yield of compound **2a** (24.0 mg) was obtained.

The ^{1}H spectra data matched with values reported in the literature.[21]



18

White solid (32.1 mg, 73% yield); m.p. 154−156°C.

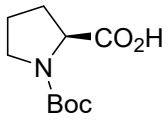
^{1}H NMR (300 MHz, $CDCl_3$): δ 8.69 (s, 1H), 7.55 (dd, *J* = 7.7, 1.7 Hz, 2H), 7.49 – 7.33 (m, 3H), 4.74 (dd, *J* = 8.0, 5.2 Hz, 1H), 3.57 (t, *J* = 6.7 Hz, 2H), 2.28 (ddt, *J* = 28.3, 13.5, 6.9 Hz, 2H), 2.10 – 1.81 (m, 2H).

(*tert*-Butoxycarbonyl)-*L*-proline (19)

Following Procedure A, using 0.1 mmol substrate instead of 0.2 mmol and using 1.0 h (1.9 F/mol)instead of 3.5 h (3.3 F/mol).

87% Yield of compound **2a** (13.1 mg) was obtained.

The ^{1}H spectra data matched with values reported in the literature.[22]



19

White solid (19.6 mg, 91% yield); m.p. 131−132°C.

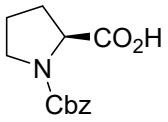
^{1}H NMR (300 MHz, $CDCl_3$): δ 4.41 – 4.13 (m, 1H), 3.36 – 3.44 (m, 2H), 2.31 (s, 1H), 1.90 – 2.06 (m, 3H), 1.48 (s, 5H), 1.42 (s, 4H).

((Benzyloxy)carbonyl)-*L*-prolinee (20)

Following Procedure A, using 0.1 mmol substrate instead of 0.2 mmol and using 1.5 h (2.8 F/mol) instead of 3.5 h (3.3 F/mol).

93% Yield of compound **2a** (14.0 mg) was obtained.

The ^{1}H spectra data matched with values reported in the literature.[23]



20

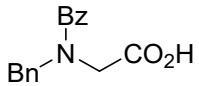
Colorless liquid (22.9 mg, 92% yield).

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.35 (s, 1H), 7.41 – 7.27 (m, 5H), 5.22 – 5.06 (m, 2H), 4.48 – 4.33 (m, 1H), 3.70 – 3.35 (m, 2H), 1.91 – 2.24 (m, 4H).

***N*-Benzoyl-*N*-benzylglycine (21)**

Following Procedure A, using 0.1 mmol substrate instead of 0.2 mmol and using 1.0 h (1.9 F/mol) instead of 3.5 h (3.3 F/mol).

87% Yield of compound **2a** (13.0 mg) was obtained.



21

White solid (22.1 mg, 82% yield); m.p. 77−78 °C.

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.62 (s, 1H), 7.57 – 7.49 (m, 1.5H), 7.49 – 7.28 (m, 7H), 7.21 (d, J = 6.9 Hz, 1.5H), 4.82 (s, 0.5H), 4.63 (s, 1.5H), 4.19 (s, 1.5H), 3.86 (s, 0.5H).

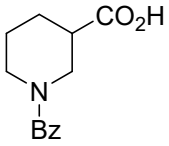
^{13}C NMR (75 MHz, $CDCl_3$): δ 173.3, 173.2, 173.1, 173.1, 135.6, 134.6, 130.3, 123.0, 129.0, 128.8, 128.5, 128.0, 127.8, 127.0, 126.9, 126.6, 126.5, 53.9, 49.4, 48.9, 46.3.

HRMS (ESI): Calcd. for $C_{16}H_{16}NO_3^+$ $[M + H]^+$: 270.1125, found: 270.1119.

1-Benzoylpiperidine-3-carboxylic acid (22)

Following Procedure A, using 0.1 mmol substrate instead of 0.2 mmol and using 2.0 h (3.7 F/mol) instead of 3.5 h (3.3 F/mol).

80% Yield of compound **2a** (12.0 mg) was obtained.



22

White solid (16.5 mg, 71% yield); m.p. 188−189 °C.

^{1}H NMR (300 MHz, DMSO-d_6): δ 12.42 (s, 1H), 7.47 – 7.32 (m, 5H), 4.27 (d, J = 87.3 Hz, 1H), 3.79 – 2.83 (m, 3H), 2.56 – 2.37 (m, 1H), 1.98 (dd, J = 8.3, 4.0 Hz, 1H), 1.79 – 1.31 (m, 3H).

^{13}C NMR (75 MHz, DMSO-d_6): δ 174.2, 169.1, 136.3, 129.3, 128.3, 126.6, 48.7, 47.3, 43.4, 41.6, 40.7, 26.8, 24.4, 23.7.

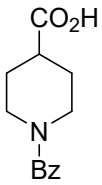
HRMS (ESI): Calcd. for $C_{13}H_{16}NO_3^+$ $[M + H]^+$: 234.1125, found: 234.1125.

1-Benzoylpiperidine-4-carboxylic acid (23)

Following Procedure A, using 0.1 mmol substrate instead of 0.2 mmol and using 3.0 h (5.6 F/mol) instead of 3.5 h (3.3 F/mol).

80% Yield of compound **2a** (12.0 mg) was obtained.

The ^{1}H spectra data matched with values reported in the literature.[24]



23

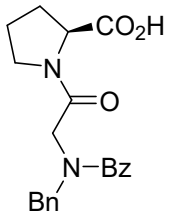
White solid (11.9 mg, 51% yield); m.p. 136−138°C.

^{1}H NMR (300 MHz, $CDCl_3$): δ 7.49 – 7.33 (m, 5H), 4.52 (s, 1H), 3.76 (s, 1H), 3.09 (t, J = 11.1 Hz, 2H), 2.63 (tt, J = 10.6, 4.1 Hz, 1H), 1.75 – 1.95 (m, 4H).

***N*-Benzoyl-*N*-benzylglycyl-*L*-proline (24)**

Following Procedure A, using 0.1 mmol substrate instead of 0.2 mmol and using 1.0 h (1.9 F/mol)instead of 3.5 h (3.3 F/mol).

93% Yield of compound **2a** (14.0 mg) was obtained.



24

White solid (31.8 mg, 87% yield); m.p. 66−68 °C.

^{1}H NMR (300 MHz, $CDCl_3$): δ 7.48 – 7.55 (m, 1.5H), 7.30 – 7.40 (m, 7H), 7.18 – 7.22 (m, 1.5H), 4.96 – 4.36 (m, 4H), 3.85 – 3.93 (m, 1H), 3.64 – 2.77 (m, 2H), 2.46 – 1.65 (m, 4H).

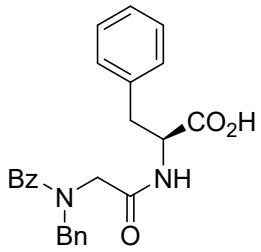
^{13}C NMR (75 MHz, $CDCl_3$): δ 174.0, 173.9, 172.8, 172.3, 168.2, 167.8, 136.6, 136.1, 135.6, 135.0, 129.9, 129.8, 128.8, 128.7, 128.4, 128.3, 127.7, 127.6, 127.1, 126.9, 126.8, 59.7, 59.5, 54.2, 53.8, 49.5, 49.0, 46.6, 46.2, 28.3, 28.1, 24.7, 22.2.

HRMS (ESI): Calcd. for $C_{21}H_{23}N_2O_4^+$ $[M + H]^+$: 367.1652, found: 367.1644.

***N*-Benzoyl-*N*-benzylglycyl-*L*-phenylalanine (25)**

Following Procedure A, using 0.05 mmol substrate instead of 0.2 mmol and using 1.0 h (3.7 F/mol) instead of 3.5 h (3.3 F/mol).

67% Yield of compound **2a** (5.0 mg) was obtained.



25

White solid (13.7 mg, 66% yield); m.p. 189−190 °C.

^{1}H NMR (300 MHz, DMSO-d_6): δ 7.96 – 7.75 (m, 1H), 7.51 – 6.95 (m, 16H), 4.30 – 4.57(m, 3H), 4.07 – 3.42 (m, 2H), 3.16 (d, J = 9.9 Hz, 1H), 2.99 – 2.70 (m, 1H).

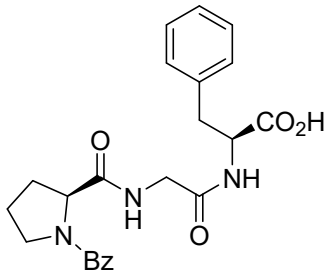
^{13}C NMR (75 MHz, DMSO-d_6): δ 174.7, 171.2, 166.8, 138.7, 136.9, 136.4, 135.9, 129.4, 129.3, 129.2, 128.5, 128.3, 127.8, 127.7, 127.4, 127.2, 126.9, 126.7, 126.4, 125.9, 54.7, 50.6, 48.0, 37.2.

HRMS (ESI): Calcd. for $C_{25}H_{25}N_2O_4^+$ $[M + H]^+$: 417.1809, found: 417.1808.

Benzoyl-*L*-prolylglycyl-*L*-phenylalanine (26)

Following Procedure A, using 0.05 mmol substrate instead of 0.2 mmol and using 2.0 h (7.5 F/mol) instead of 3.5 h (3.3 F/mol).

67% Yield of compound **2a** (5.0 mg) was obtained.



26

White solid (14.8 mg, 70% yield); m.p. 184−185 °C.

^{1}H NMR (300 MHz, DMSO-d_6): δ 8.29 – 8.32 (m, 1H), 7.99 – 8.05 (m, 1H), 7.67 – 7.07 (m, 11H), 4.12 – 4.42 (m, 2H), 3.94 – 3.45 (m, 4H), 2.86 – 3.05 (m, 2H), 2.14 – 2.17 (m, 1H), 2.00 – 1.68 (m, 3H).

^{13}C NMR (75 MHz, DMSO-d_6): δ 173.5, 171.9, 168.9, 168.6, 138.2, 136.5, 130.1, 129.3, 129.2, 128.2, 127.4, 127.3, 126.6, 126.2, 60.4, 54.3, 49.9, 42.0, 37.0, 29.5, 24.9.

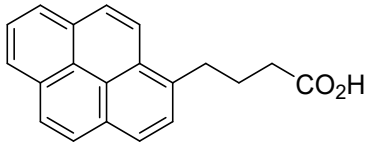
HRMS (ESI): Calcd. for $C_{23}H_{26}N_3O_5^+$ $[M + H]^+$: 424.1867, found: 424.1849.

4-(Pyren-1-yl)butanoic acid (27)

Following Procedure A, using 0.05 mmol substrate instead of 0.2 mmol, PBS/MeCN (v/v = 1/2, 6.0 mL) instead of PBS/MeCN (v/v = 1/1, 3.0 mL) and using 1.5 h (5.6 F/mol) instead of 3.5 h (3.3 F/mol).

80% Yield of compound **2a** (6.0 mg) was obtained.

The ^{1}H spectra data matched with values reported in the literature.[25]



27

White solid (12.0 mg, 83% yield); m.p. 184−185°C.

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.29 (d, J = 9.2 Hz, 1H), 8.13 (dd, J = 14.2, 8.2 Hz, 4H), 8.02 – 7.94 (m, 3H), 7.86 (d, J = 7.8 Hz, 1H), 3.48 – 3.35 (m, 2H), 2.51 (t, J = 7.2

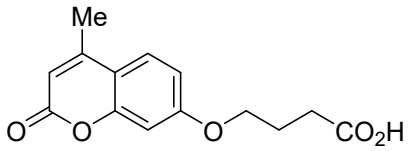
Hz, 2H), 2.22 (dd, *J* = 15.0, 7.3 Hz, 2H).

4-((4-Methyl-2-oxo-2*H*-chromen-7-yl)oxy)butanoic acid (28)

Following Procedure A, using 0.05 mmol substrate instead of 0.2 mmol, 4.0 mA instead of 10 mA and using 2.0 h (3.0 F/mol) instead of 3.5 h (3.3 F/mol).

68% Yield of compound **2a** (5.1 mg) was obtained.

The ^{1}H spectra data matched with values reported in the literature.[26]



28

White solid (8.4 mg, 64% yield); m.p. 171−172 °C.

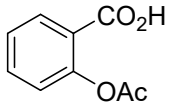
^{1}H NMR (300 MHz, DMSO-d_6): δ 12.20 (s, 1H), 7.85 – 7.52 (m, 1H), 6.96 (dd, *J* = 8.0, 2.0 Hz, 2H), 6.21 (d, *J* = 1.2 Hz, 1H), 4.10 (t, *J* = 6.5 Hz, 2H), 2.46 – 2.32 (m, 5H), 1.96 (p, *J* = 6.8 Hz, 2H).

2-Acetoxybenzoic acid (29)

Following Procedure A, using 4.0 h (3.7 F/mol) instead of 3.5 h (3.3 F/mol).

80% Yield of compound **2a** (24.0 mg) was obtained.

The ^{1}H spectra data matched with values reported in the literature.[27]



29

White solid (29.1 mg, 81% yield); m.p. 132−134 °C.

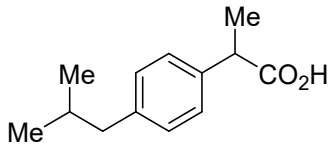
^{1}H NMR (300 MHz, $CDCl_3$): δ 8.13 (dd, *J* = 7.9, 1.8 Hz, 1H), 7.63 (ddd, *J* = 8.1, 7.5, 1.7 Hz, 1H), 7.36 (td, *J* = 7.6, 1.2 Hz, 1H), 7.14 (dd, *J* = 8.1, 1.1 Hz, 1H), 2.35 (s, 3H).

2-(4-Isobutylphenyl)propanoic acid (30)

Following Procedure A, using 2.0 h (1.9 F/mol) instead of 3.5 h (3.3 F/mol).

67% Yield of compound **2a** (20.1 mg) was obtained.

The ^{1}H spectra data matched with values reported in the literature.[28]



30

White solid (26.4 mg, 64% yield); m.p. 53−54 °C.

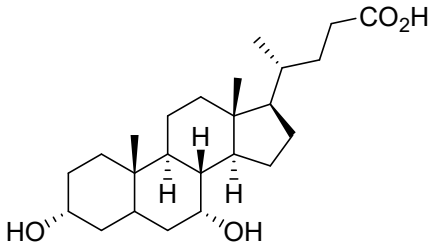
^{1}H NMR (300 MHz, $CDCl_3$): δ 7.23 (dd, *J* = 13.1, 5.9 Hz, 2H), 7.10 (t, *J* = 8.8 Hz, 2H), 3.70 (q, *J* = 7.1 Hz, 1H), 2.44 (d, *J* = 7.1 Hz, 2H), 1.84 (dt, *J* = 13.5, 6.8 Hz, 1H), 1.48 (d, *J* = 7.1 Hz, 3H), 0.90 (s, 3H), 0.88 (s, 3H).

(4*R*)-4-((3*R*,7*R*,8*R*,9*S*,10*S*,13*R*,14*S*,17*R*)-3,7-Dihydroxy-10,13-dimethylhexadecahydro-1*H*-cyclopenta[*a*]phenanthren-17-yl)pentanoic acid (31)

Following Procedure A, using 0.1 mmol substrate instead of 0.2 mmol, PBS/MeCN (v/v = 1/3, 3.0 mL) instead of PBS/MeCN (v/v = 1/1, 3.0 mL) and using 3.0 h (5.6 F/mol) instead of 3.5 h (3.3 F/mol).

60% Yield of compound **2a** (9.0 mg) was obtained.

The ^{1}H spectra data matched with values reported in the literature.[29]



31

White solid (16.1 mg, 41% yield); m.p. 200−201 °C.

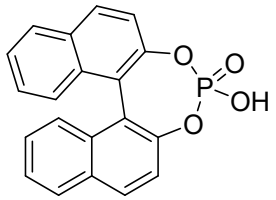
^{1}H NMR (300 MHz, DMSO-d_6): δ 11.94 (s, 1H), 4.31 (d, *J* = 4.7 Hz, 1H), 4.11 (d, *J* = 3.3 Hz, 1H), 3.62 (s, 1H), 3.26 (d, *J* = 41.6 Hz, 3H), 2.18 (ddd, *J* = 30.8, 15.5, 9.4 Hz, 3H), 1.96 – 1.59 (m, 7H), 1.50 – 1.31 (m, 7H), 1.29 – 1.03 (m, 7H), 0.88 (d, *J* = 6.4 Hz, 3H), 0.83 (s, 3H), 0.60 (s, 3H).

4-Hydroxydinaphtho[2,1-*d*:1',2'-*f*][1,3,2]dioxaphosphepine 4-oxide (32)

Following Procedure A, using 0.1 mmol substrate instead of 0.2 mmol and using 1.5 h (2.8 F/mol) instead of 3.5 h (3.3 F/mol).

80% Yield of compound **2a** (12.0 mg) was obtained.

The ^{1}H spectra data matched with values reported in the literature.[30]



32

White solid (27.1 mg, 78% yield); m.p. 238−240 °C.

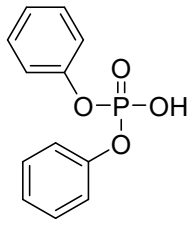
^{1}H NMR (300 MHz, DMSO-d_6): δ 8.18 (d, J = 8.9 Hz, 2H), 8.09 (d, J = 8.0 Hz, 2H), 7.61 – 7.49 (m, 4H), 7.37 (ddd, J = 8.2, 6.8, 1.3 Hz, 2H), 7.23 (d, J = 8.3 Hz, 2H).

Diphenyl hydrogen phosphate (33)

Following Procedure A, using 0.1 mmol substrate instead of 0.2 mmol and using 1.0 h (1.9 F/mol) instead of 3.5 h (3.3 F/mol).

94% Yield of compound **2a** (14.1 mg) was obtained.

The ^{1}H spectra data matched with values reported in the literature.[31]



33

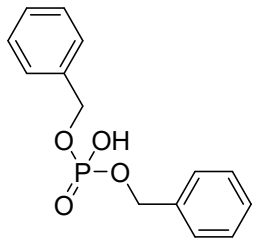
White solid (23.0 mg, 92% yield); m.p. 67−68 °C.

^{1}H NMR (300 MHz, $CDCl_3$): δ 8.86 (s, 1H), 7.33 – 7.25 (m, 4H), 7.16 (t, J = 7.6 Hz, 6H).

Dibenzyl hydrogen phosphate (34)

Following Procedure A, using 0.1 mmol substrate instead of 0.2 mmol and using 1.5 h (2.8 F/mol) instead of 3.5 h (3.3 F/mol).

73% Yield of compound **2a** (11.0 mg) was obtained.



34

White solid (18.9 mg, 68% yield); m.p. 76−78 °C.

^{1}H NMR (300 MHz, DMSO-d_6): δ 7.23 (brs, 10H), 4.88 (d, J = 5.6 Hz, 4H).

^{13}C NMR (75 MHz, DMSO-d_6): δ 136.8, 136.7, 128.3, 128.0, 127.5, 67.4 (q, J = 4.4 Hz).

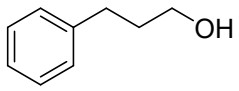
HRMS (ESI): Calcd. for $C_{14}H_{16}O_4P^+$ $[M + H]^+$: 279.0781, found: 279.0777.

3-Phenylpropan-1-ol (35)

Following Procedure A, using 4.0 h (3.7 F/mol) instead of 3.5 h (3.3 F/mol).

50% Yield of compound **2a** (15.0 mg) was obtained.

The ^{1}H spectra data matched with values reported in the literature.[32]



35

Colorless liquid (16.3 mg, 60% yield).

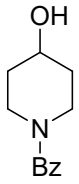
^{1}H NMR (300 MHz, $CDCl_3$): δ 7.32 – 7.24 (m, 2H), 7.23 – 7.13 (m, 3H), 3.68 (t, J = 6.4 Hz, 2H), 2.75 – 2.66 (m, 2H), 1.96 – 1.82 (m, 2H).

(4-Hydroxypiperidin-1-yl)(phenyl)methanone (36)

Following Procedure A, using 4.0 h (3.7 F/mol) instead of 3.5 h (3.3 F/mol).

80% Yield of compound **2a** (24.0 mg) was obtained.

The ^{1}H spectra data matched with values reported in the literature.[33]



36

White solid (32.5 mg, 81% yield); m.p. 89−90°C.

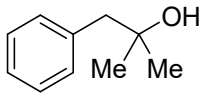
^{1}H NMR (300 MHz, $CDCl_3$): δ 7.45 – 7.33 (m, 5H), 4.18 (s, 1H), 3.95 (tt, J = 8.0, 3.8 Hz, 1H), 3.66 (s, 1H), 3.27 (d, J = 45.7 Hz, 2H), 1.95 (s, 3H), 1.52 (s, 2H).

2-Methyl-1-phenylpropan-2-ol (37)

Following Procedure A, using 6.0 h (5.6 F/mol) instead of 3.5 h (3.3 F/mol).

50% Yield of compound **2a** (15.0 mg) was obtained.

The ^{1}H spectra data matched with values reported in the literature.[34]

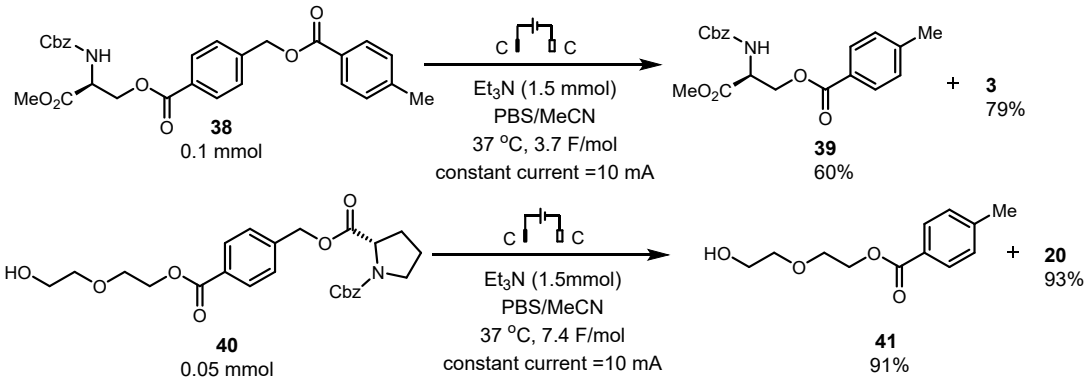


37

Colorless liquid (16.2 mg, 54% yield).

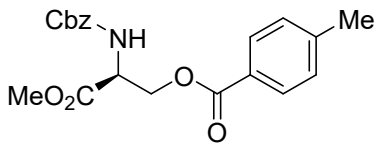
^{1}H NMR (300 MHz, $CDCl_3$): δ 7.38 – 7.14 (m, 5H), 2.77 (s, 2H), 1.23 (s, 6H).

Procedure for electrodeprotection of serine or diethylene glycol derived esters. (Procedure B):



A 10 mL distillation flask equipped with a magnetic stir bar was charged with compound **38** (0.1 mmol) or **40** (0.05 mmol), Et_3N (0.3 mmol) and PBS/MeCN (v/v = 1/1, 3.0 mL). The flask equipped with graphite rod anode (d = 5 mm) and graphite rod cathode (d = 5 mm). The resulting solution was stirred and electrolyzed at a constant current of 10 mA (Single Output DC Power Supply: KRP-305DM) for 2.0 h at 37 °C. The solution was diluted with DCM (5 mL), and extracted with DCM (3 × 20 mL). The combined organic layers were washed with brine (3 × 20 mL), dried over Na_2SO_4 and concentrated *in vacuo*. Purification by flash column chromatography using PE/EtOAc (v/v = 10/1) as eluent afforded the desired product **39** and **41**.

(*S*)-2-(((Benzyloxy)carbonyl)amino)-3-methoxy-3-oxopropyl 4-methylbenzoate (39)



39

White solid (22.3 mg, 60% yield); m.p. 53−54 °C.

^{1}H NMR (300 MHz, $CDCl_3$): δ 7.86 (d, *J* = 8.3 Hz, 2H), 7.39 – 7.28 (m, 5H), 7.28 – 7.17 (m, 2H), 5.66 (d, *J* = 7.7 Hz, 1H), 5.13 (s, 2H), 4.88 – 4.48 (m, 3H), 3.79 (s, 3H), 2.41 (s, 3H).

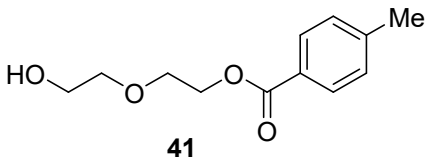
^{13}C NMR (75 MHz, $CDCl_3$): δ 167.0, 166.0, 155.7, 144.1, 135.9, 129.7, 129.1, 128.5, 128.2, 128.1, 126.5, 67.2, 64.5, 53.4, 52.8, 21.6.

HRMS (ESI): Calcd. for $C_{20}H_{22}NO_6^+$ $[M + H]^+$: 372.1442, found: 372.1435.

79% Yield of compound **3** (10.7 mg) was obtained.

2-(2-Hydroxyethoxy)ethyl 4-methylbenzoate (41)

The ^{1}H spectra data matched with values reported in the literature.[35]



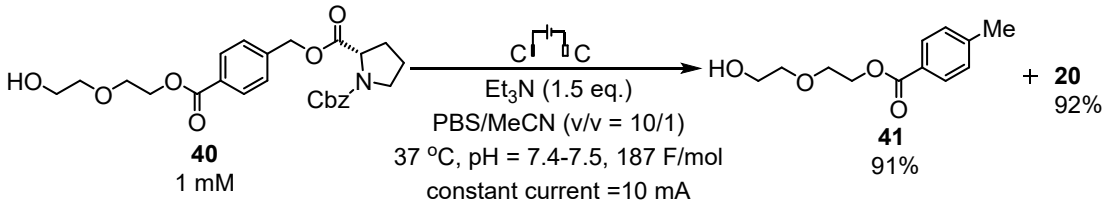
41

Colorless liquid (10.2 mg, 91% yield).

^{1}H NMR (300 MHz, $CDCl_3$): δ 7.99 – 7.89 (m, 2H), 7.28 – 7.19 (m, 2H), 4.50 – 4.45 (m, 2H), 3.87 – 3.80 (m, 2H), 3.75 (dd, *J* = 5.2, 3.4 Hz, 2H), 3.69 – 3.61 (m, 2H), 2.41 (s, 3H).

93% Yield of compound **20** (11.6 mg) was obtained.

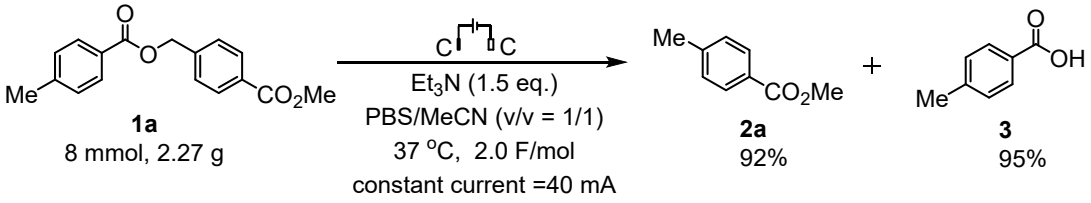
Procedure for reaction at 1.0 mM concentration. (Procedure C):



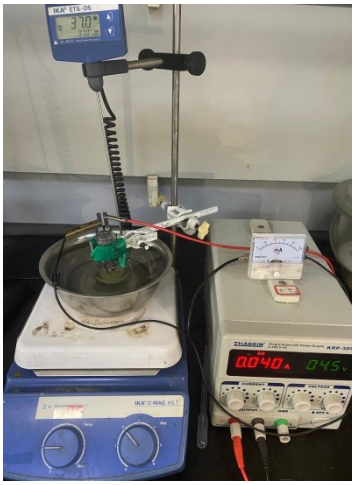
A 10 mL distillation flask equipped with a magnetic stir bar was charged with compound **40** (0.003 mmol, 1.0 eq.), Et_3N (0.0045 mmol, 1.5 eq.) and PBS/MeCN (v/v = 10/1, 3.0 mL). The flask equipped with graphite rod anode (d = 5 mm) and graphite rod cathode (d = 5 mm). The resulting solution was stirred and electrolyzed at a constant current of 10 mA (Single Output DC Power Supply: KRP-305DM) for 1.0 h (187 F/mol) at 37 °C. The solution was diluted with DCM (5 mL), and extracted with DCM (3 × 20 mL). The combined organic layers were washed with brine (3 × 20 mL), dried over Na_2SO_4, and concentrated *in vacuo* afforded the desired product **41** (91%) and **20**

(92%) (analysis by ^{1}H NMR using 1,3,5-trimethoxybenzene as the internal standard).

Procedure for gram-scale experiment. (Procedure D):



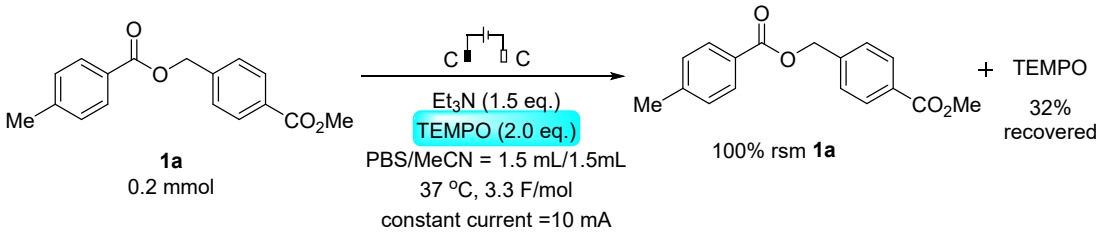
A 50 mL distillation flask equipped with a magnetic stir bar was charged with compound **1a** (8 mmol, 1.0 eq.), Et_3N (12 mmol, 1.5 eq.) and PBS/MeCN (v/v = 1/1, 30 mL). The flask equipped with graphite rod anode (d = 5 mm) and graphite rod cathode (d = 5 mm). The resulting solution was stirred and electrolyzed at a constant current of 40 mA (Single Output DC Power Supply: KRP-305DM) for 21 h (2.0 F/mol) at 37 °C. The solution was diluted with DCM (50 mL), and extracted with DCM (3 × 50 mL). The combined organic layers were washed with brine (3 × 50 mL), dried over Na_2SO_4, and concentrated *in vacuo*. Purification by flash column chromatography using PE/EtOAc (v/v = 10/1) as eluent afforded the desired product **2a** (1.103g, 92%) and **3** (1.031g, 95%).



Electrochemical setup for 8 mmol scale

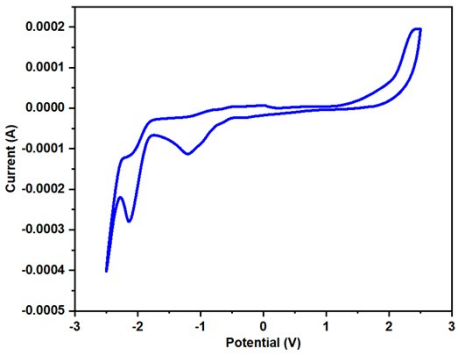
V Mechanism Research

Scheme S1: Radical-trapping Experiment.



Scheme S2: **Cyclic Voltammetry Experiment:**

Cyclic voltammetry of compound **1a** at a scan rate of 100 $mV \cdot s^{-1}$. The electrochemical experiments were performed in a three-compartment cell fitted with a glassy carbon as the working electrode, a saturated calomel electrode (SCE) as the reference electrode, and a platinum wire as the counter electrode. Electrolyte: 0.1 M *n*-Bu_4NPF_6 in MeCN; Concentration of a sample: 0.01 M. The reduction potentials of the starting material **1a** is -1.22 V and -2.16 V.



Cyclic voltammograms of compound **1a**

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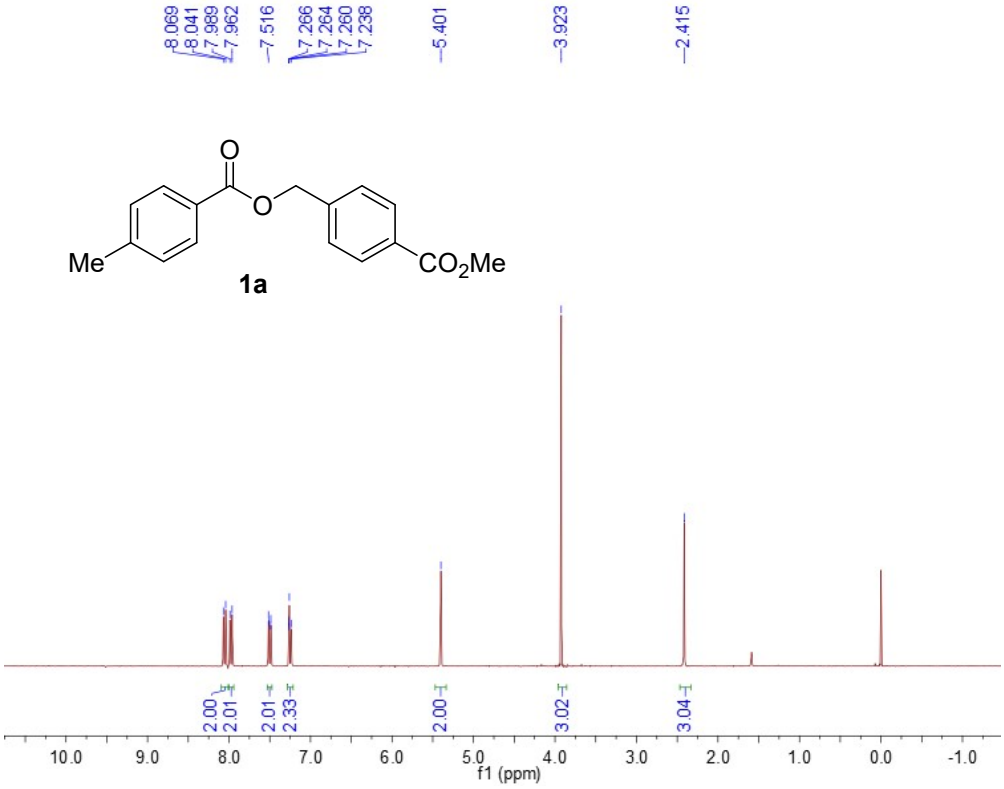
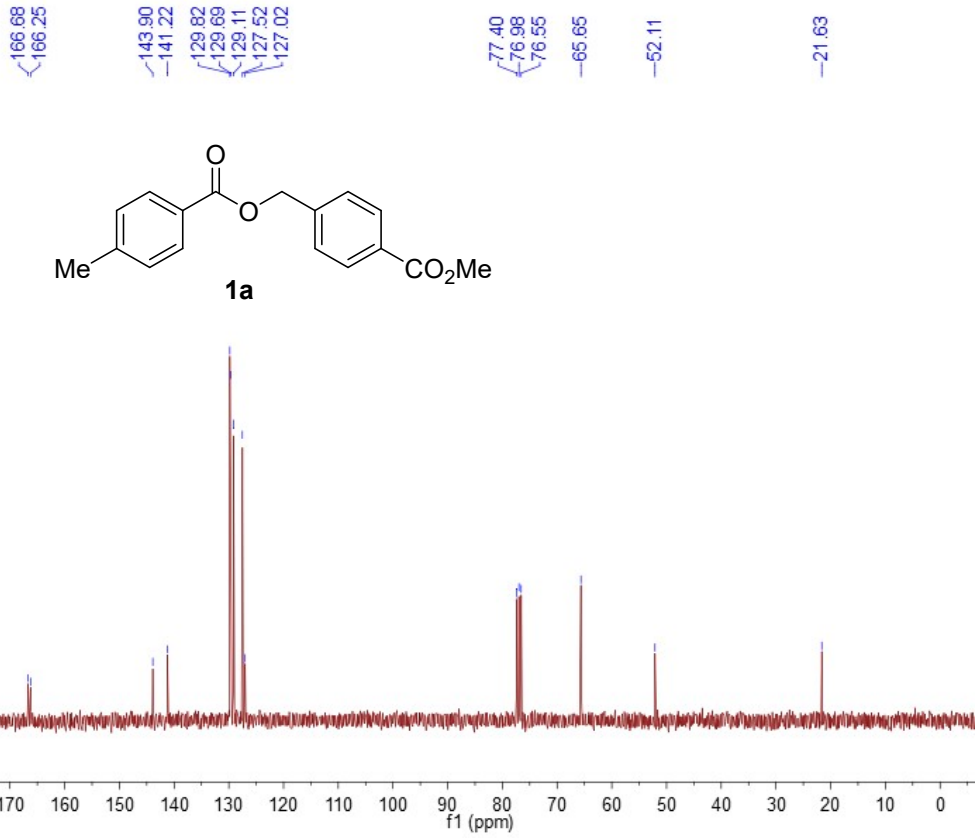
VII. Spectra

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



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



1a

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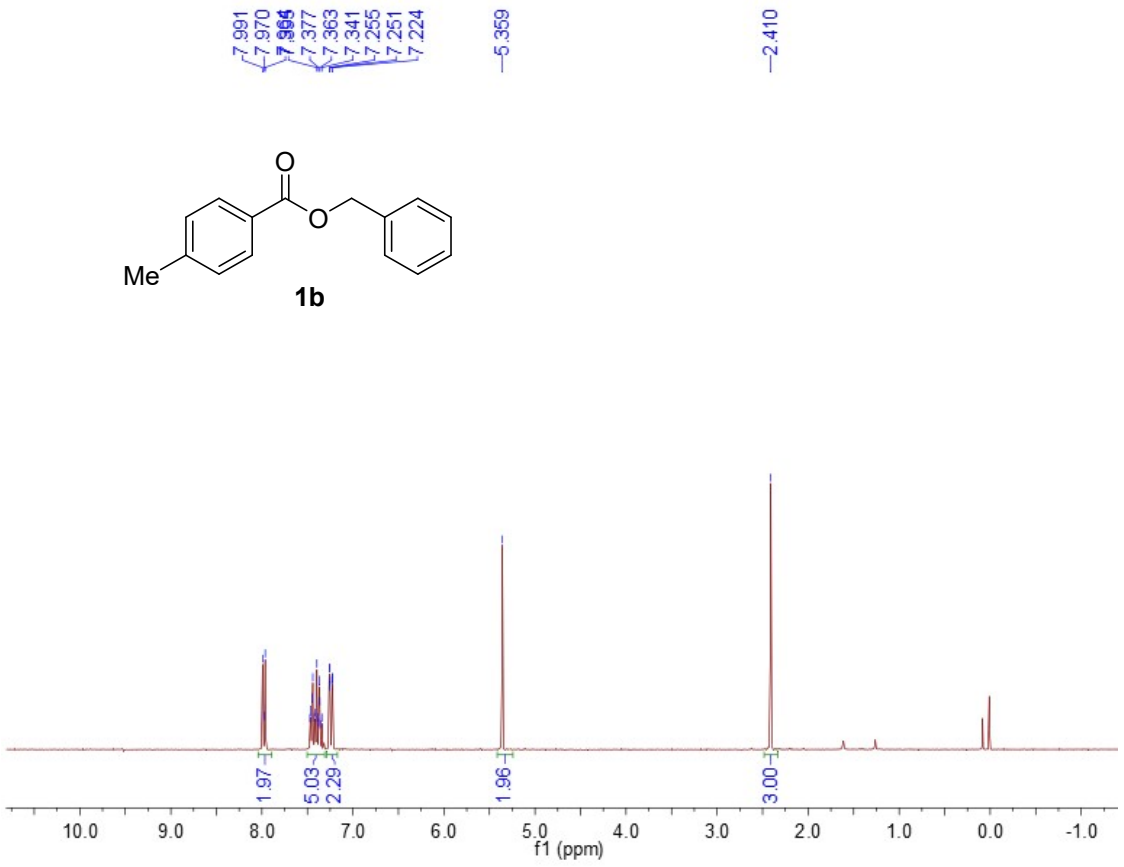

1b

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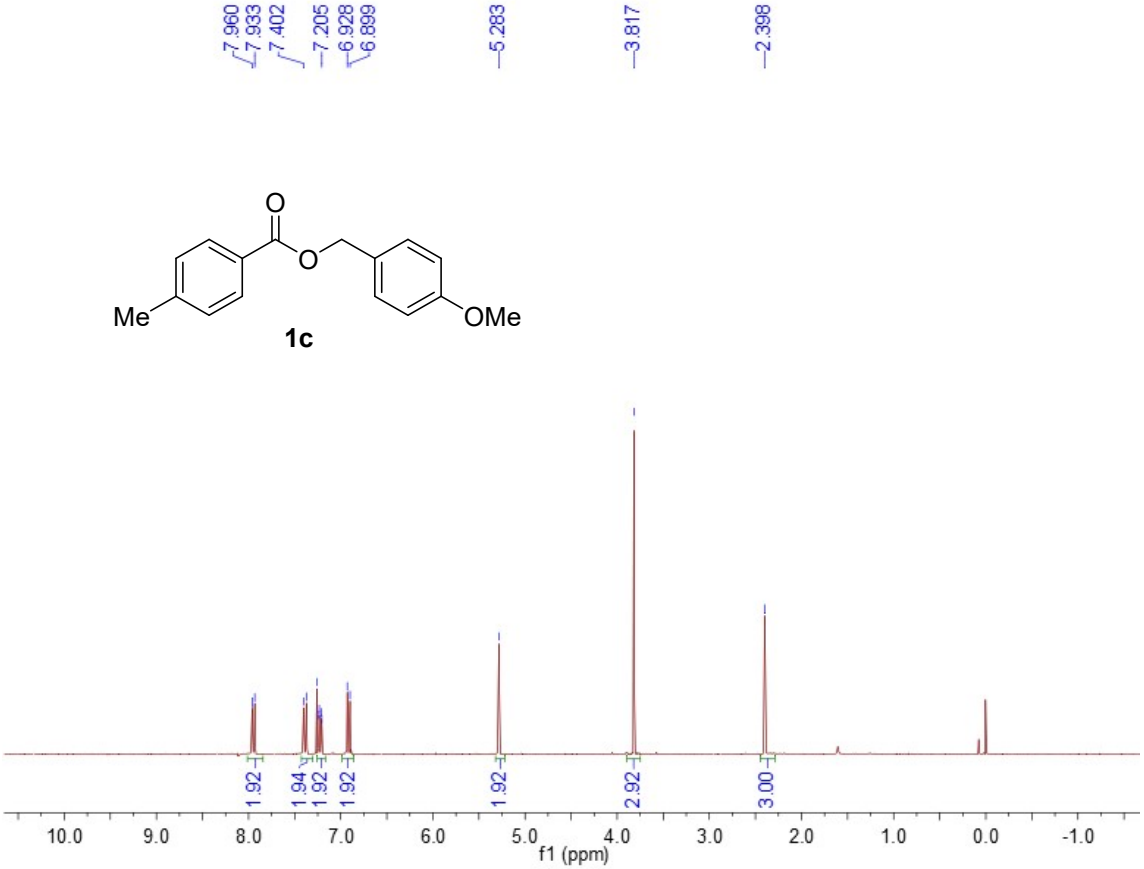

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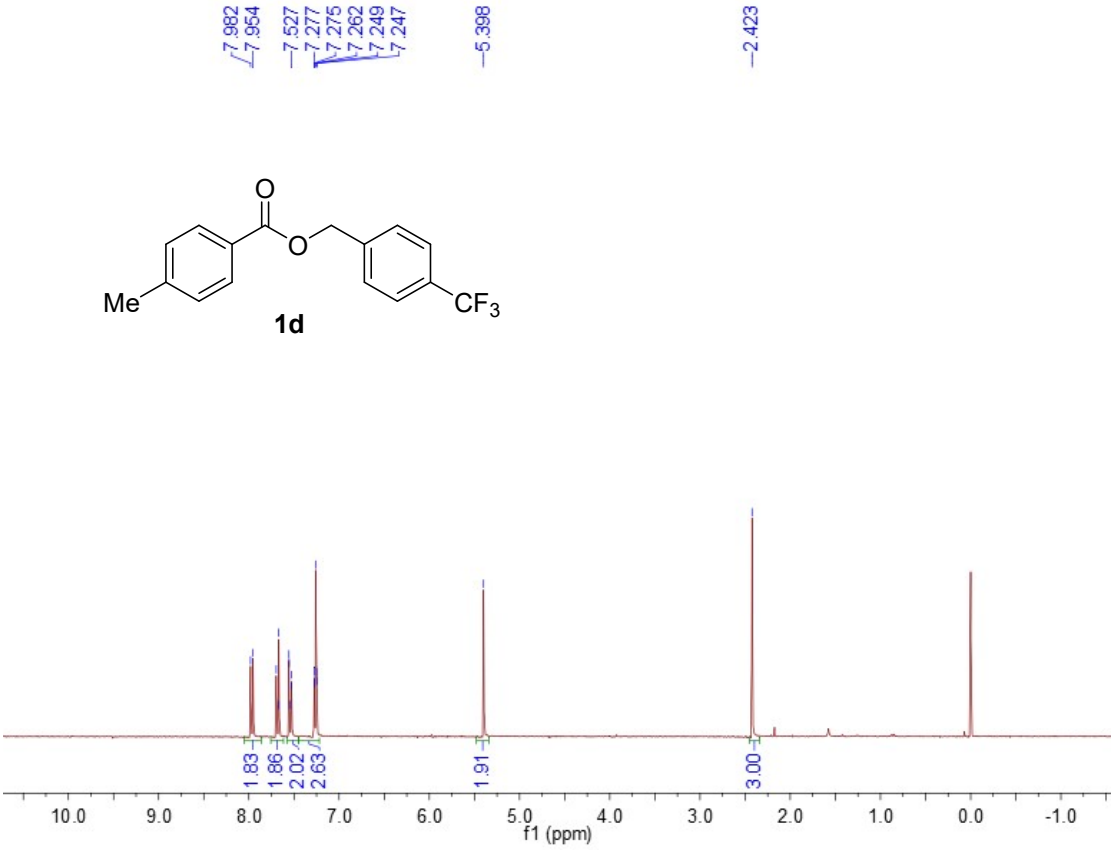

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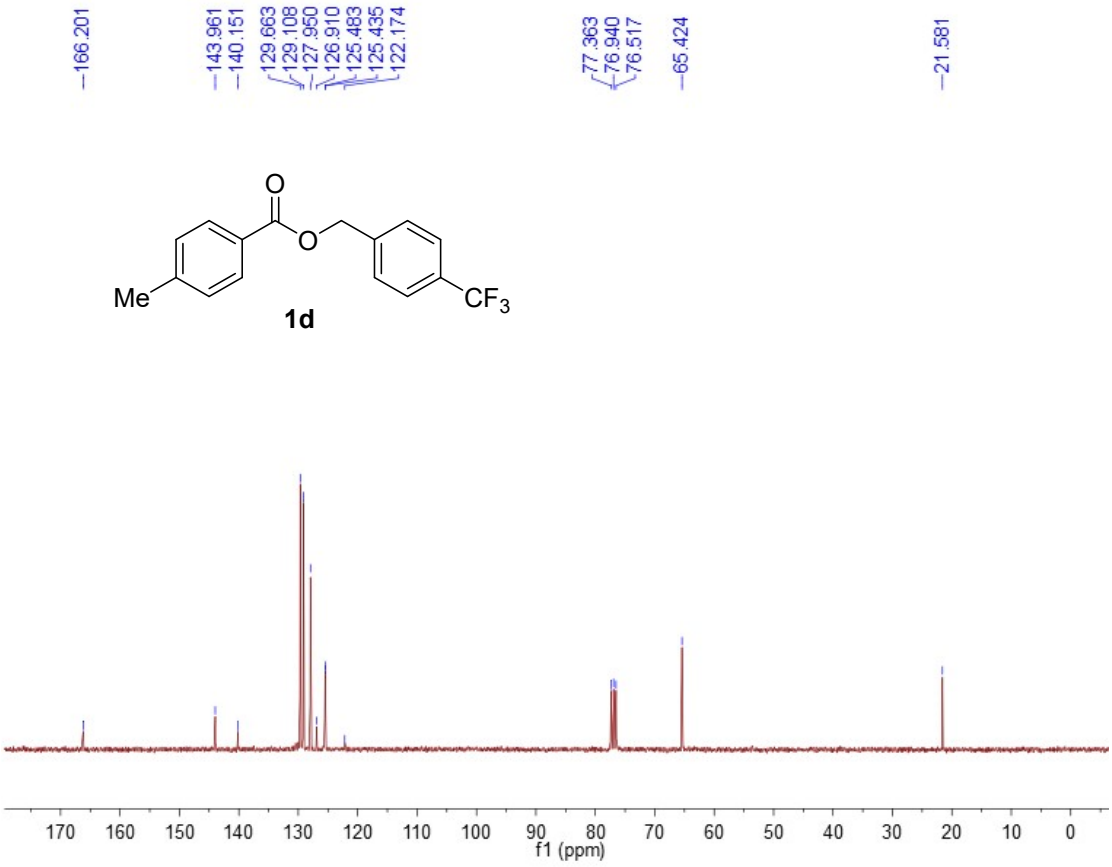

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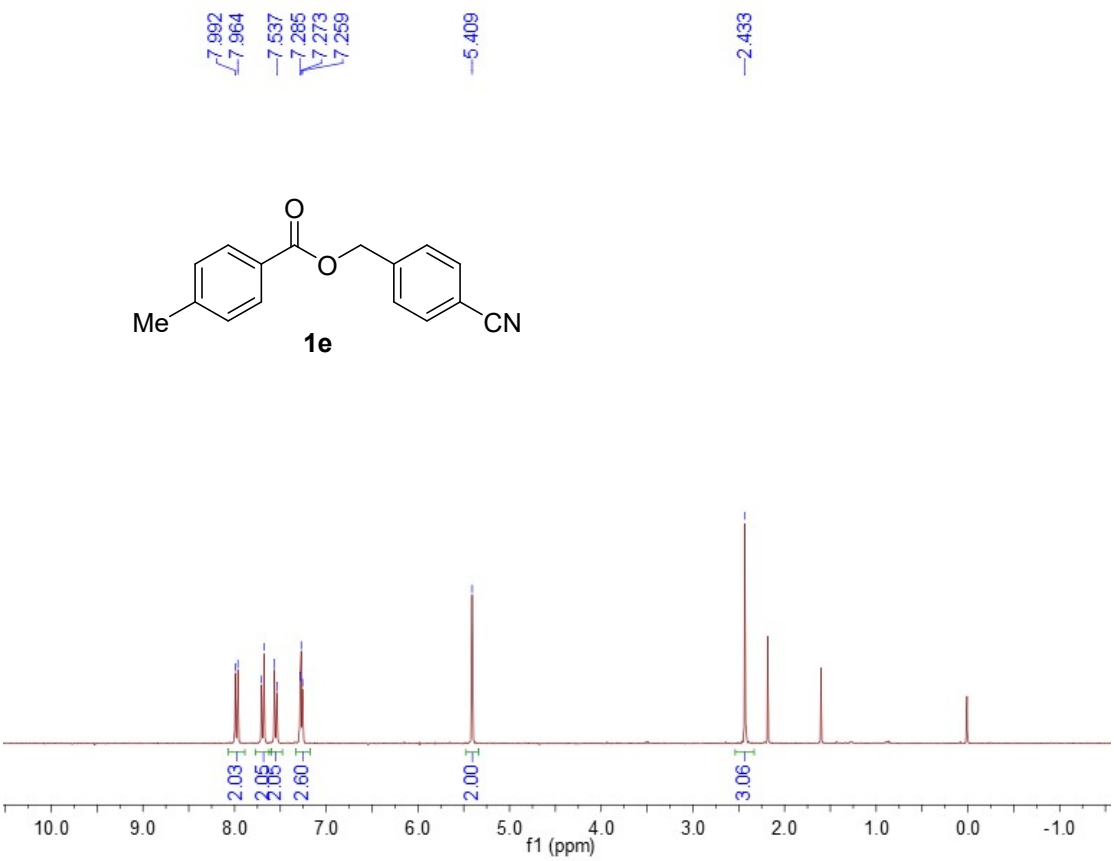

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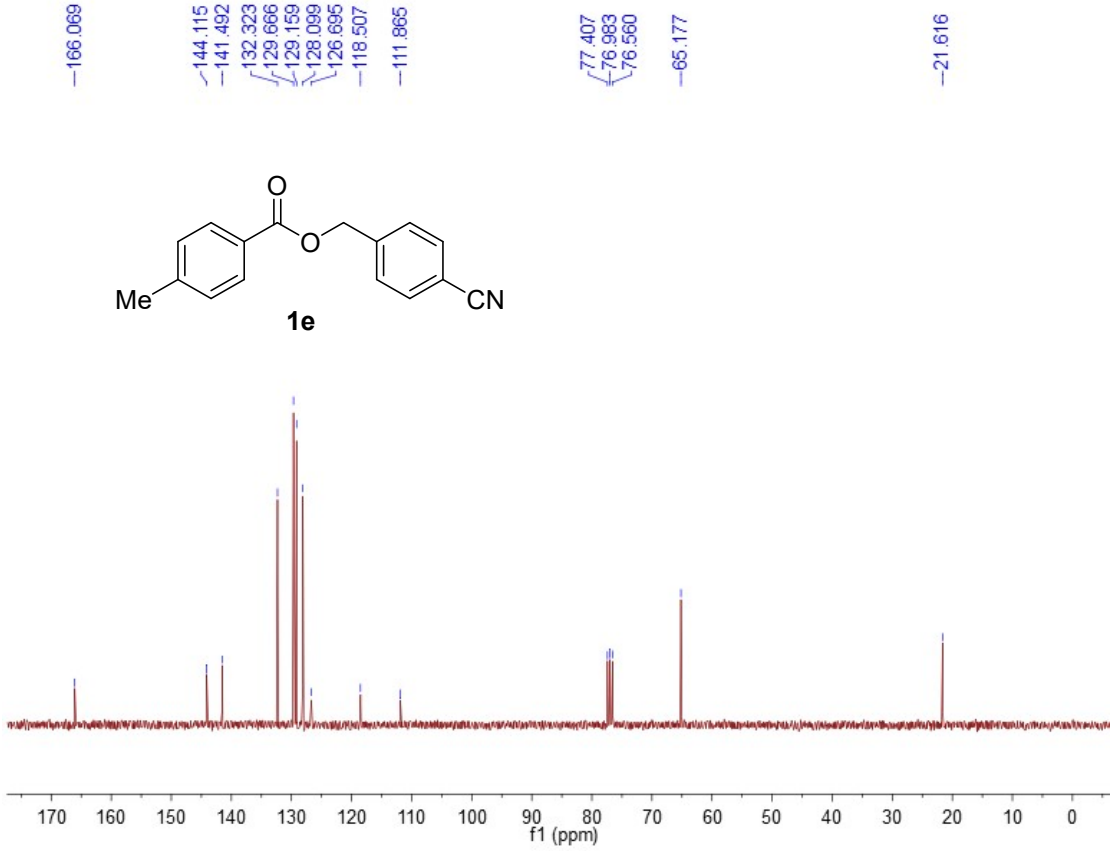

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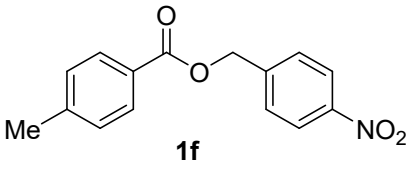

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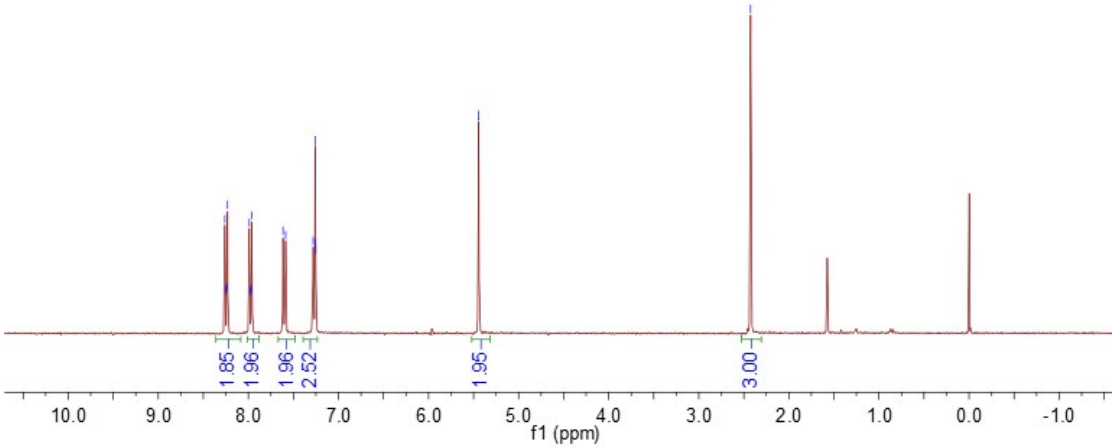

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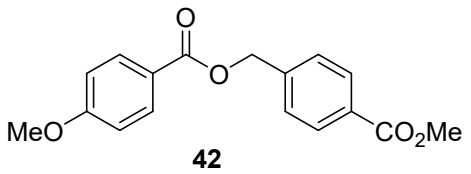

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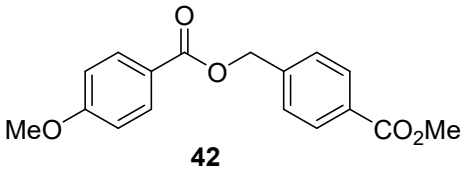

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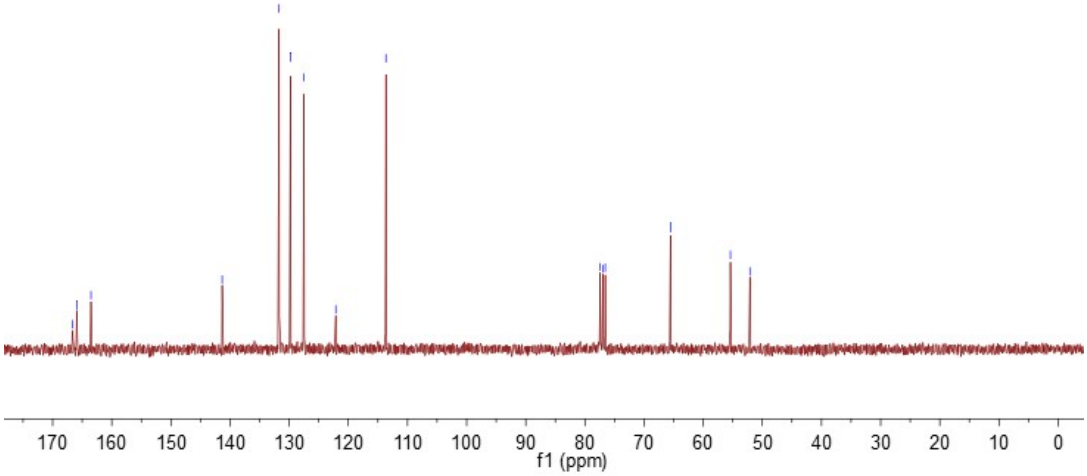

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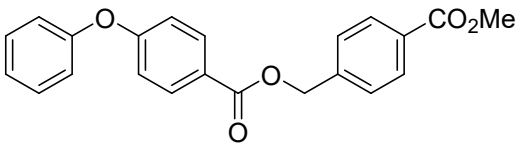

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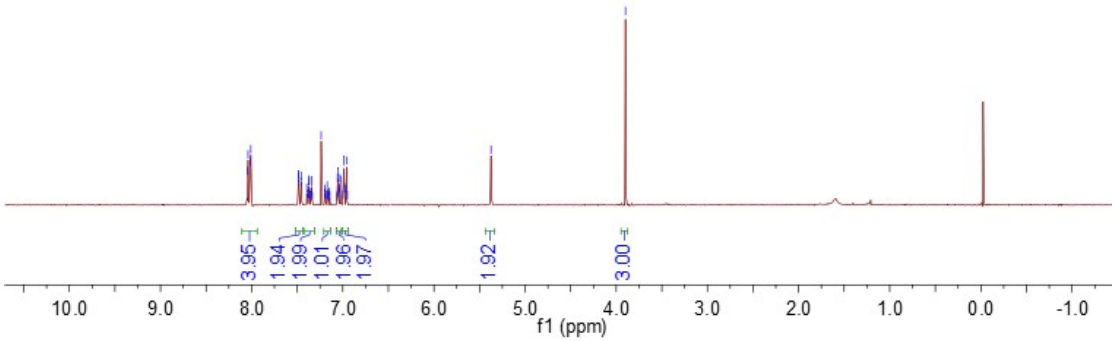

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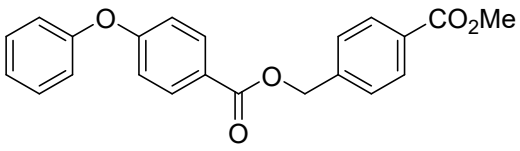

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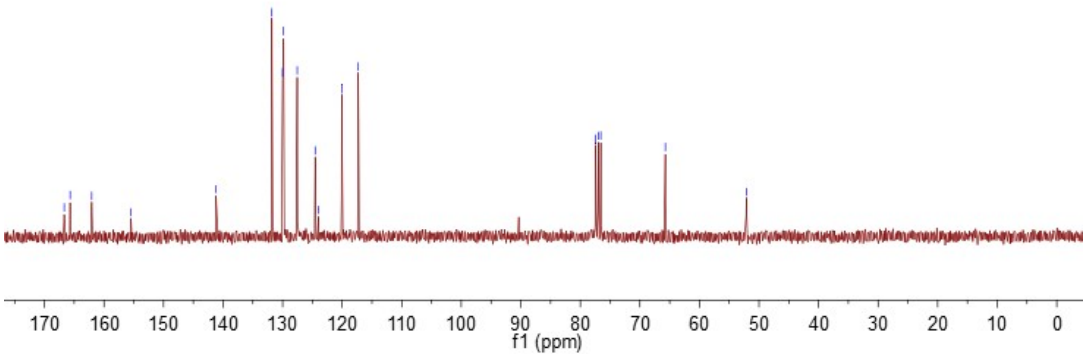

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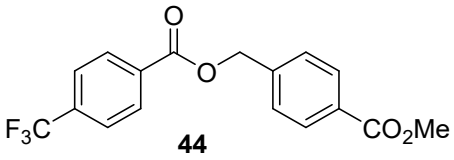

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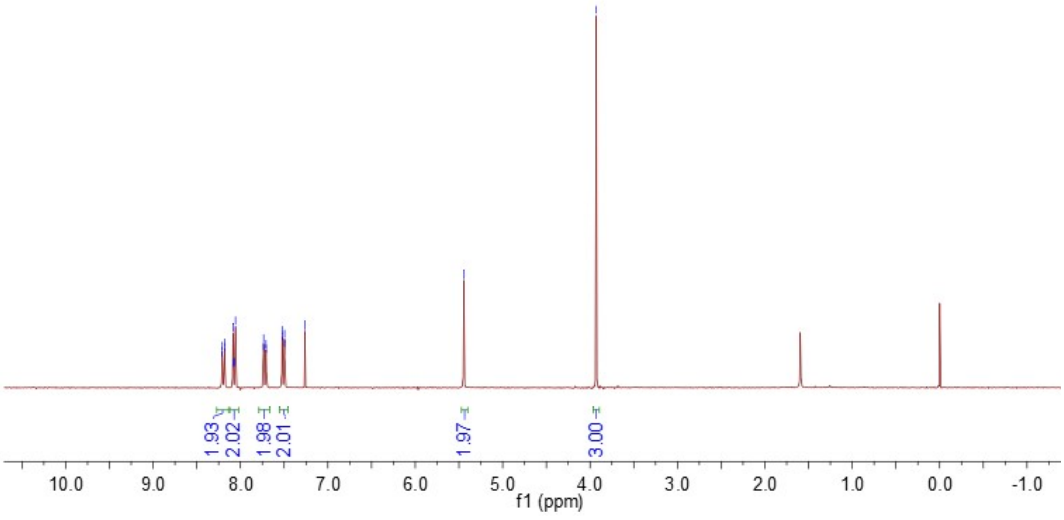

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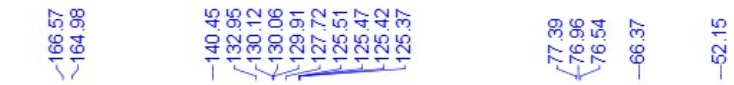

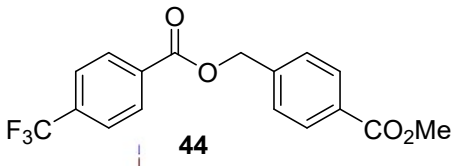

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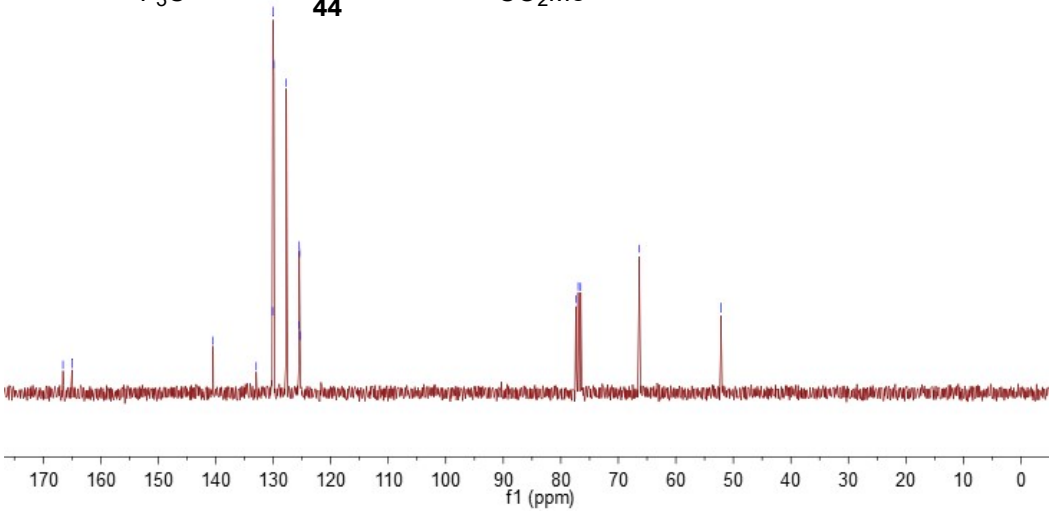

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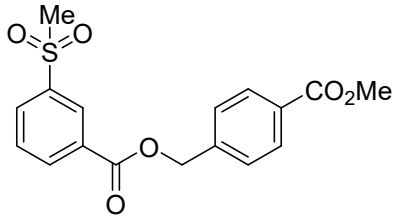

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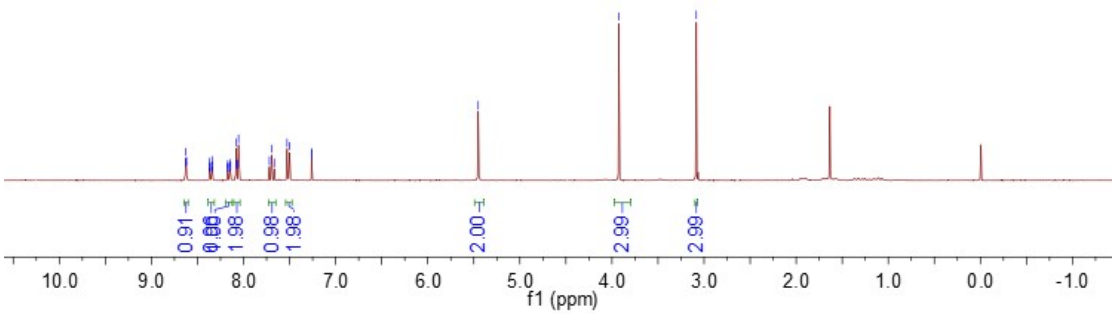

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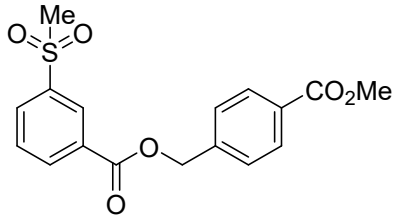

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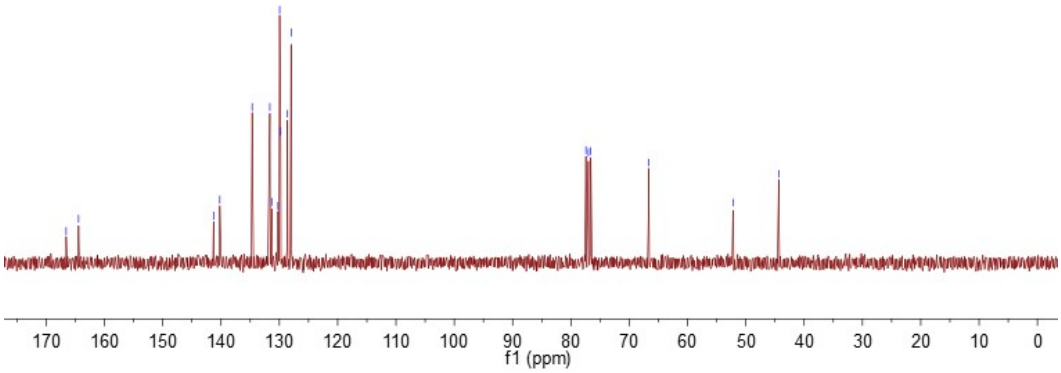

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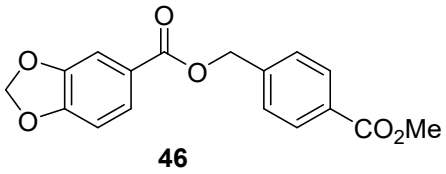

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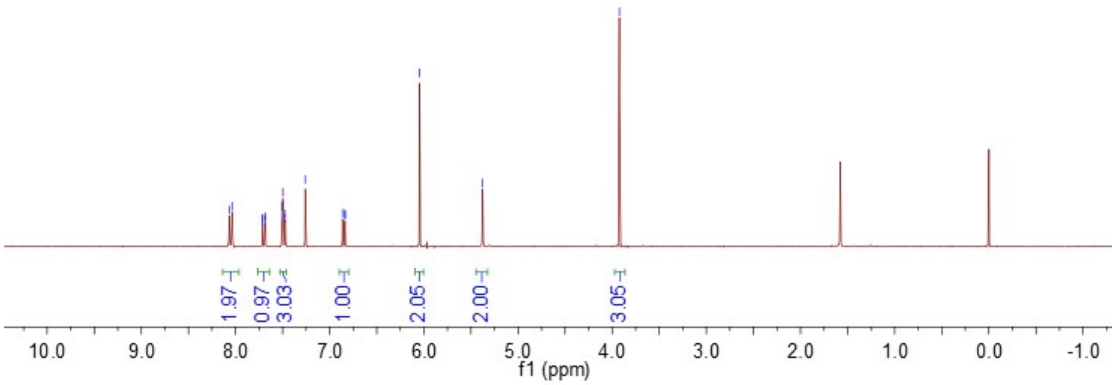

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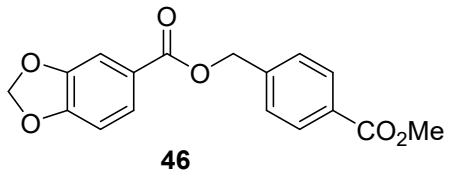

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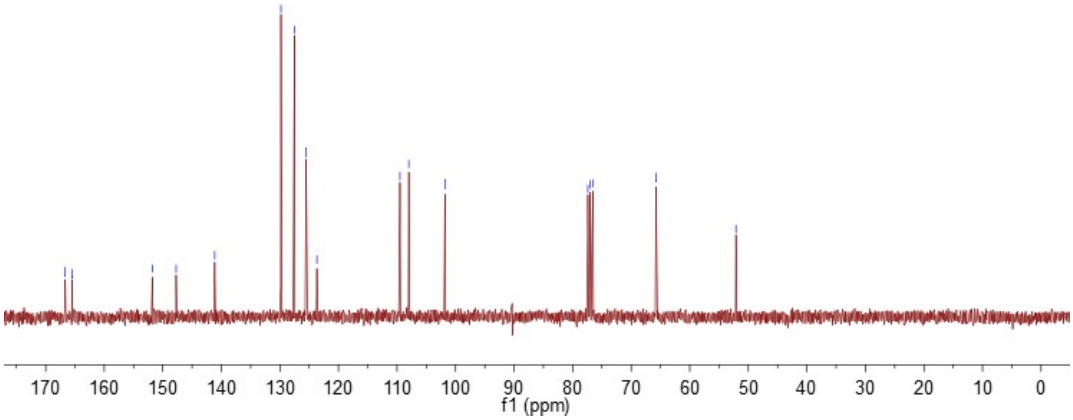

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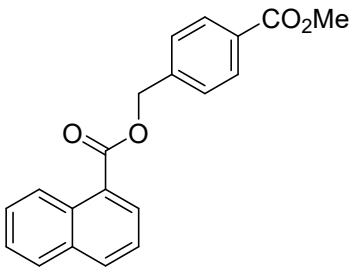

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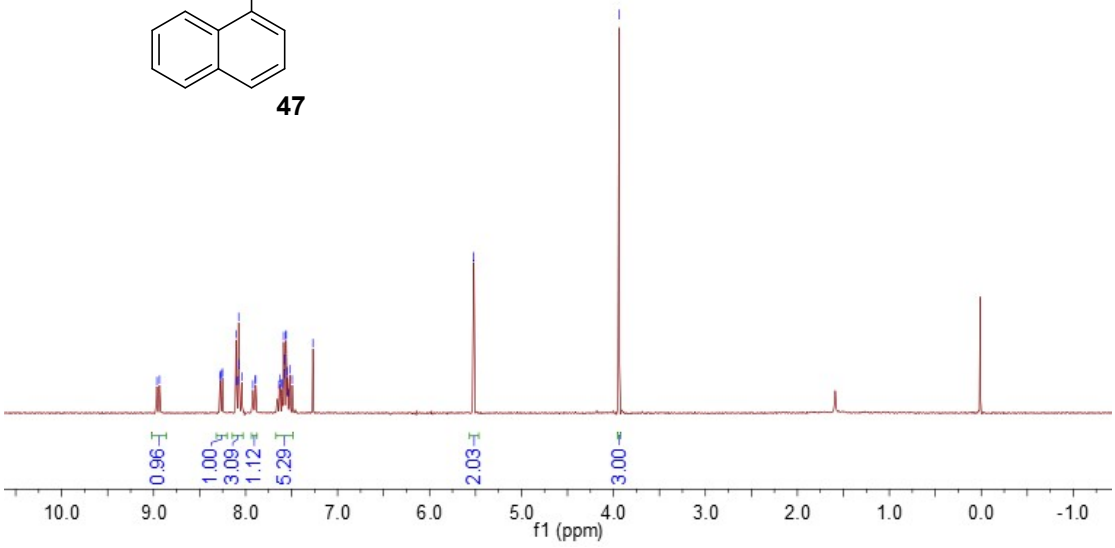

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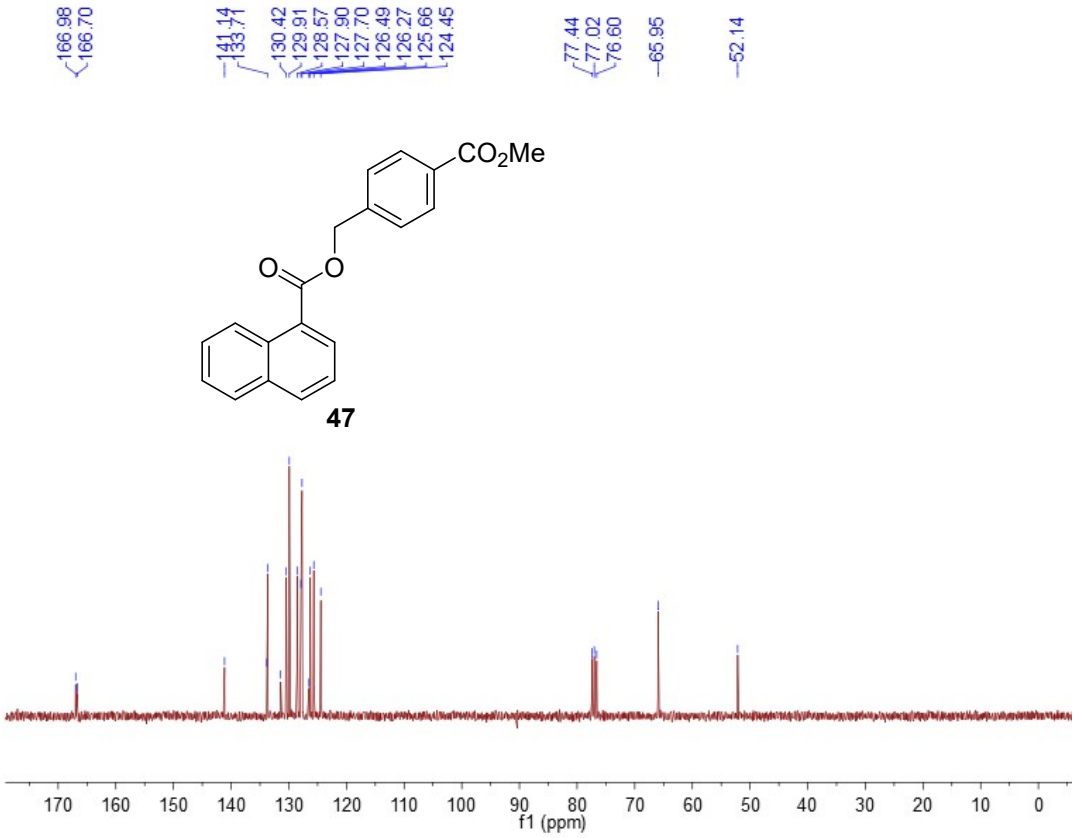

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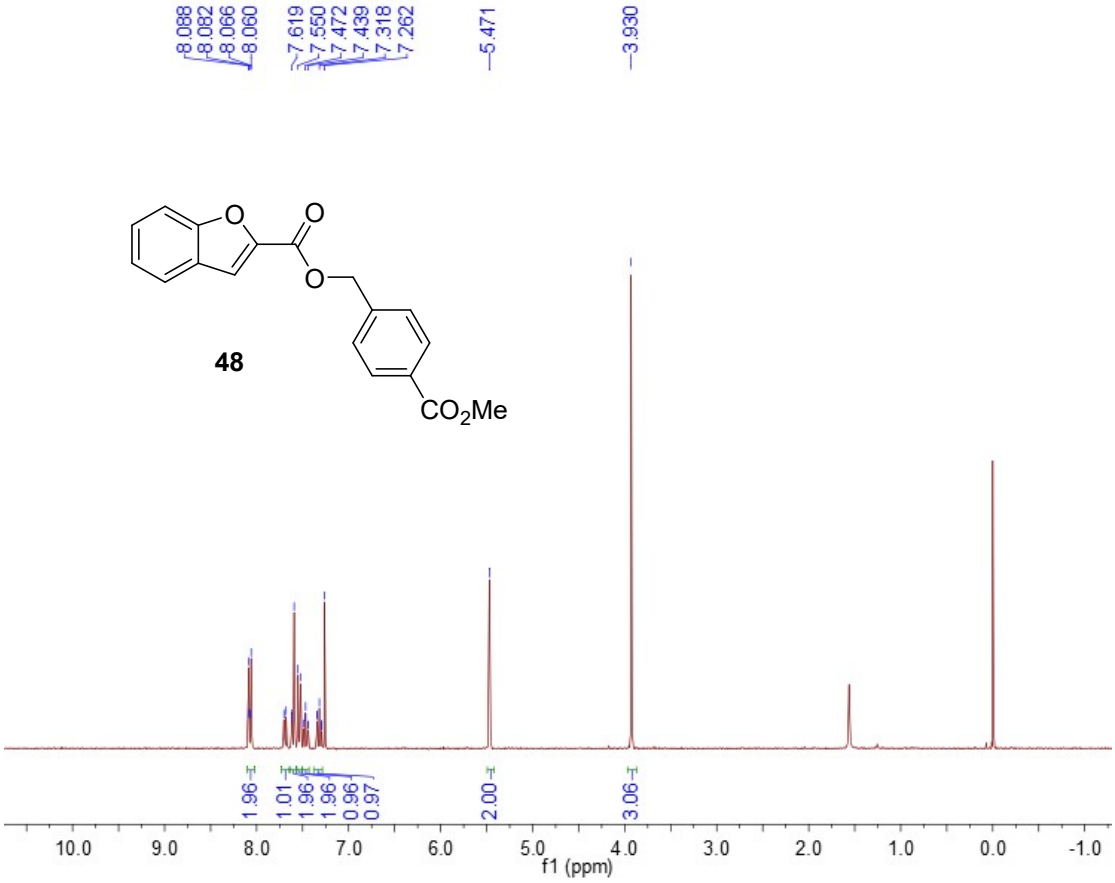

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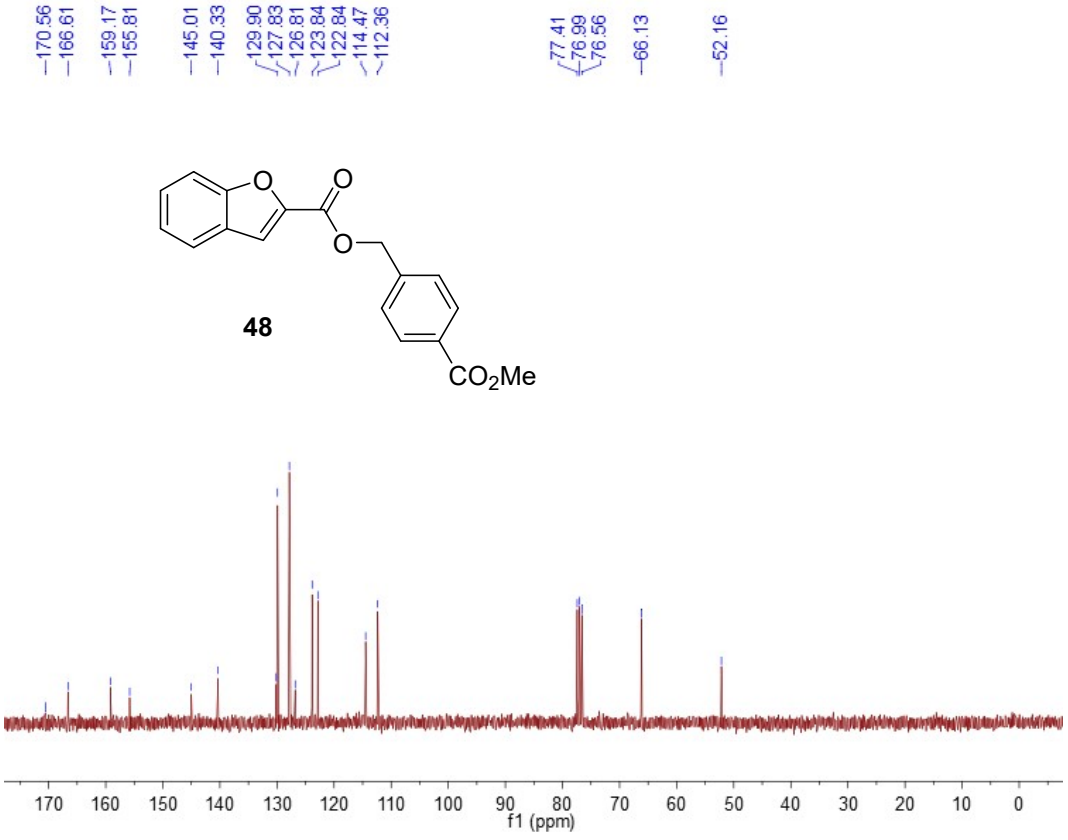

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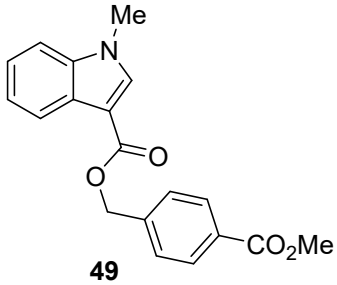

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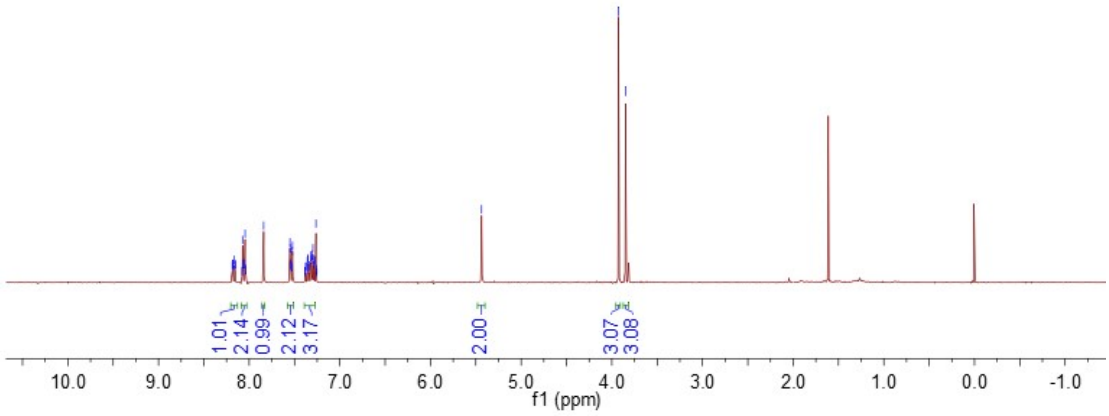

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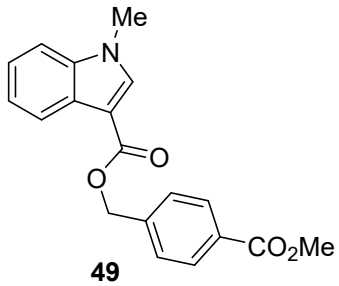

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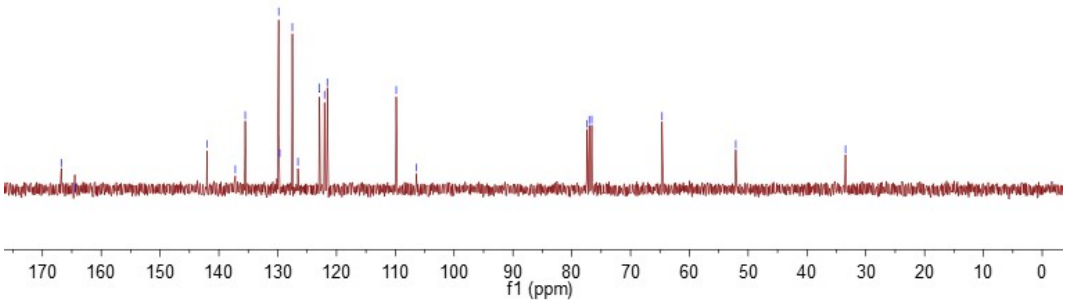

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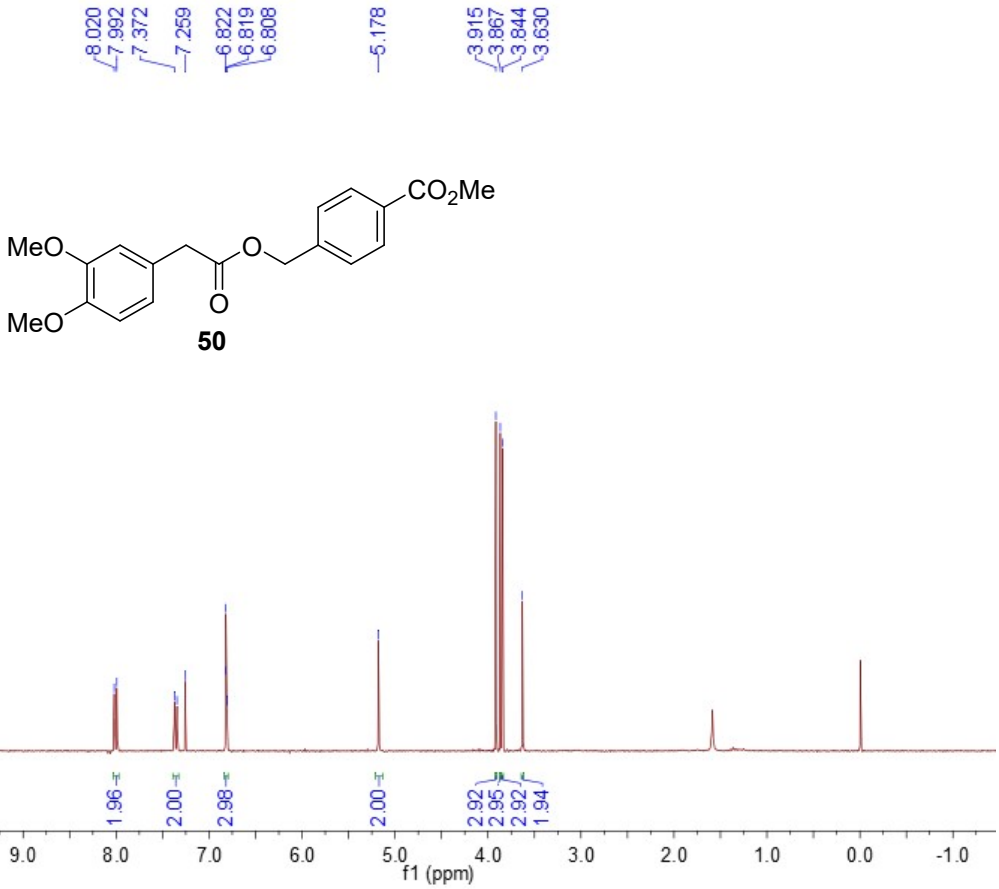
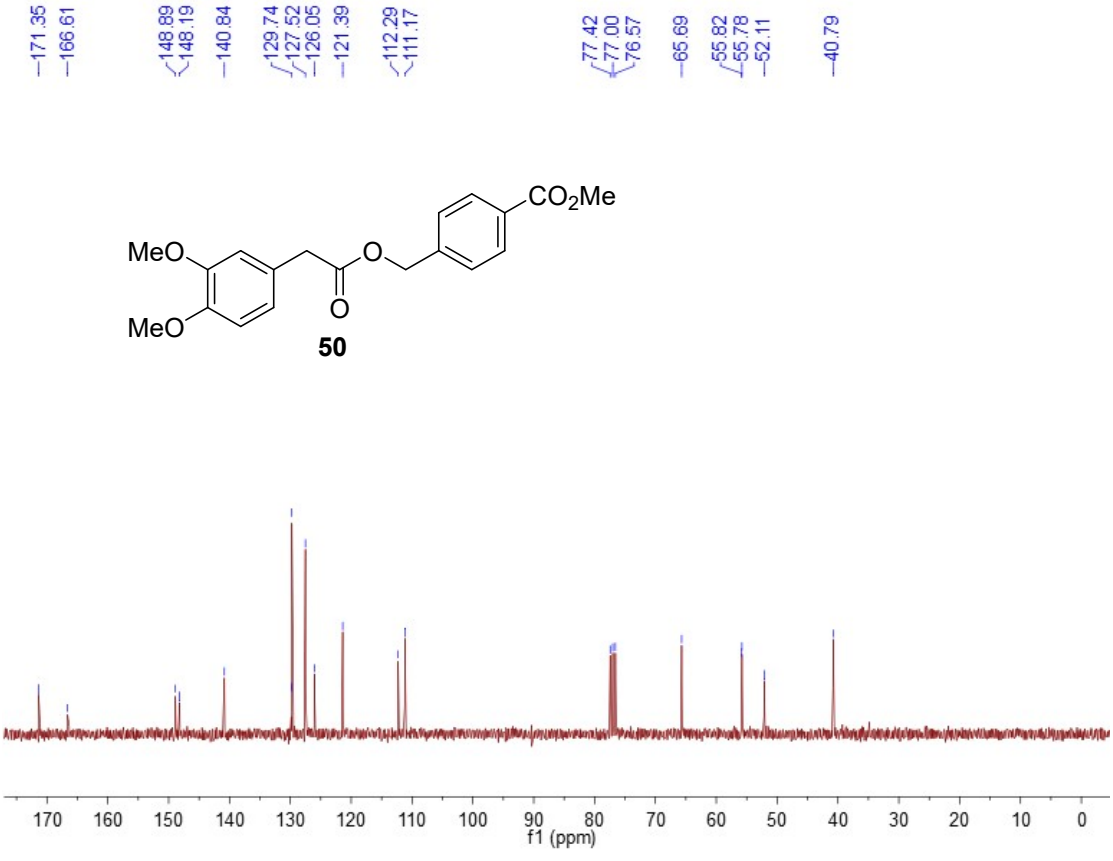


50

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



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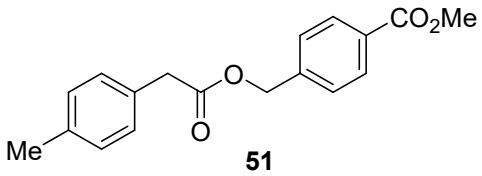
^{1}H NMR (300 MHz, $CDCl_3$):



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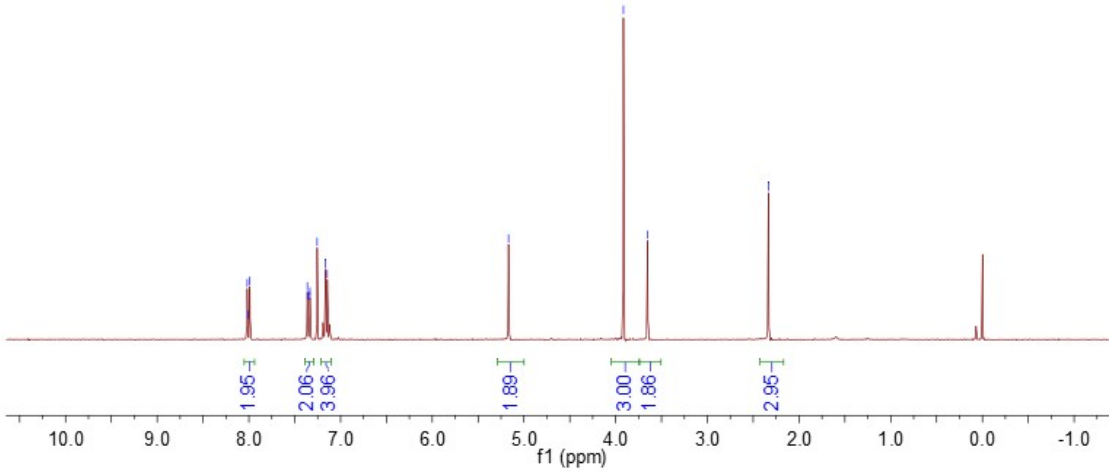
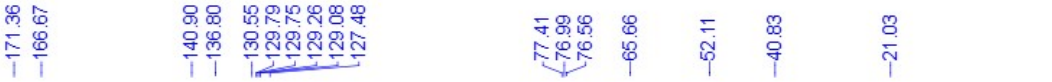
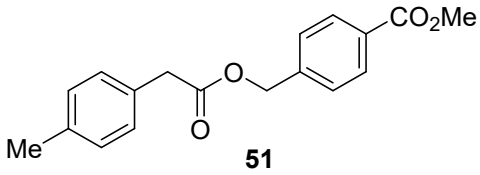


^{13}C NMR (75 MHz, $CDCl_3$):





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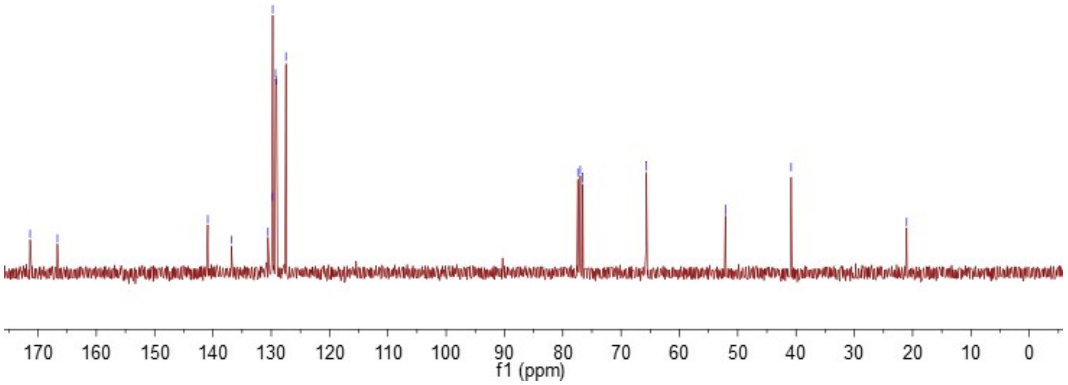

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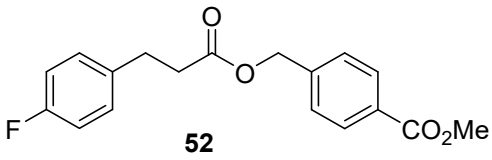

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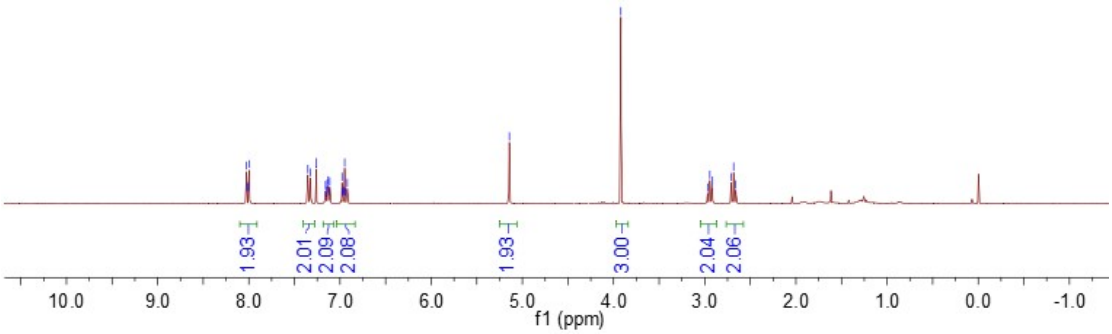
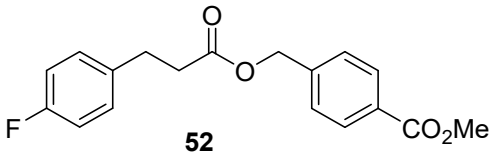


^{13}C NMR (75 MHz, $CDCl_3$):



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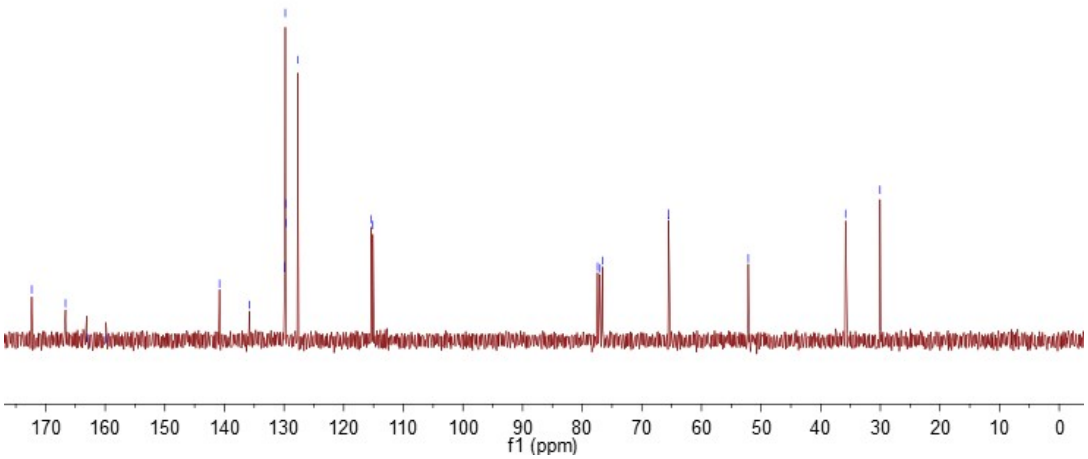

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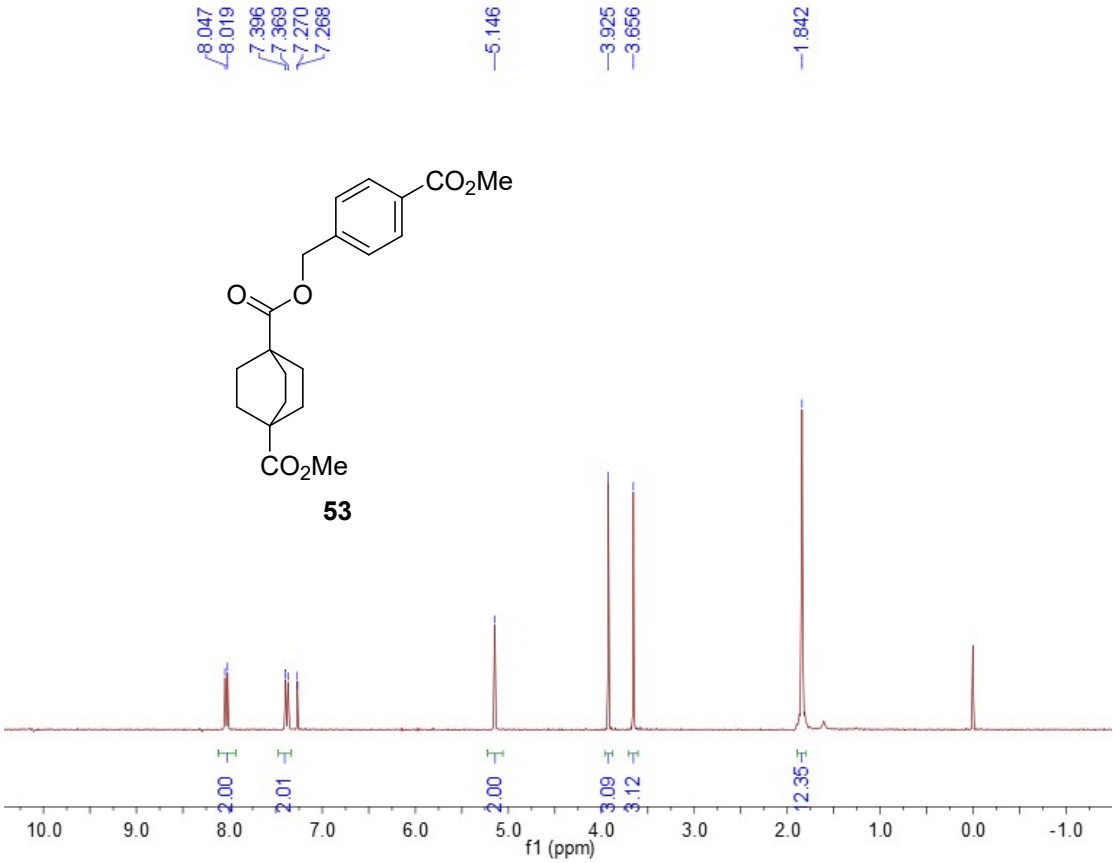

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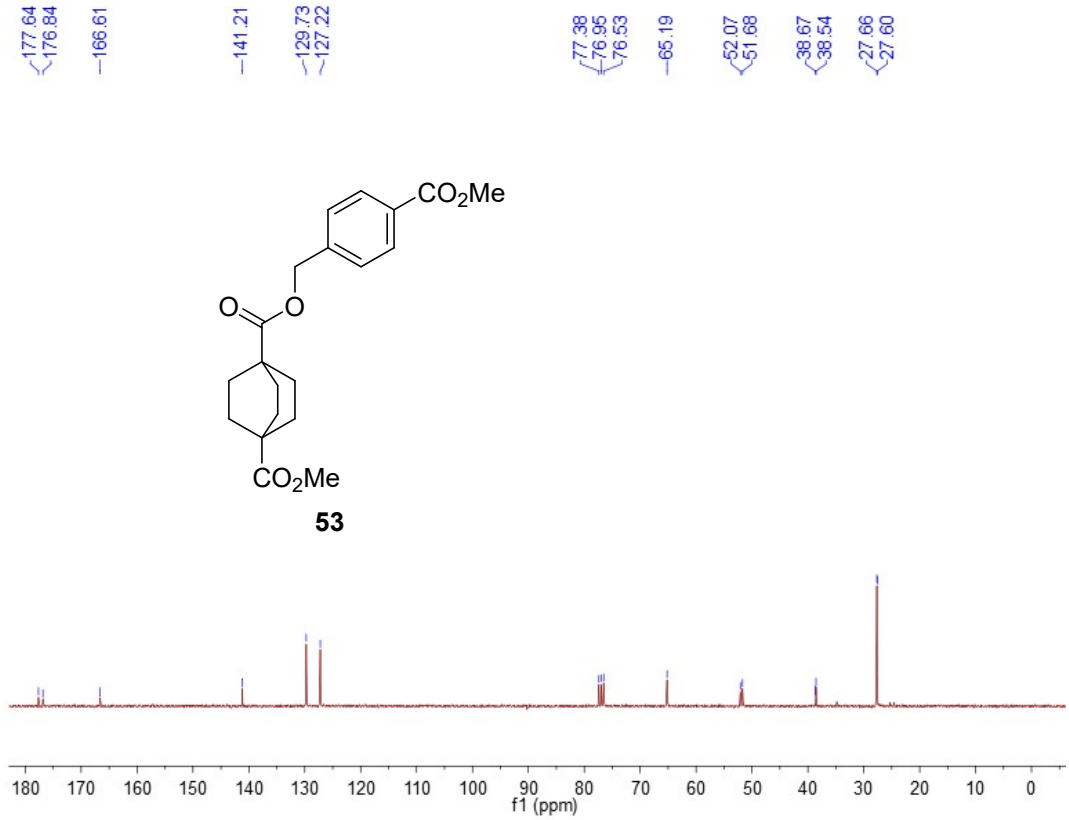

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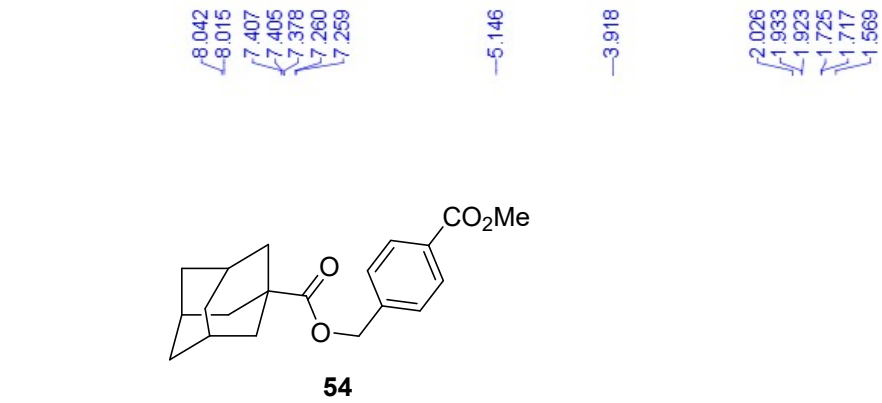
^{1}H NMR (300 MHz, $CDCl_3$):



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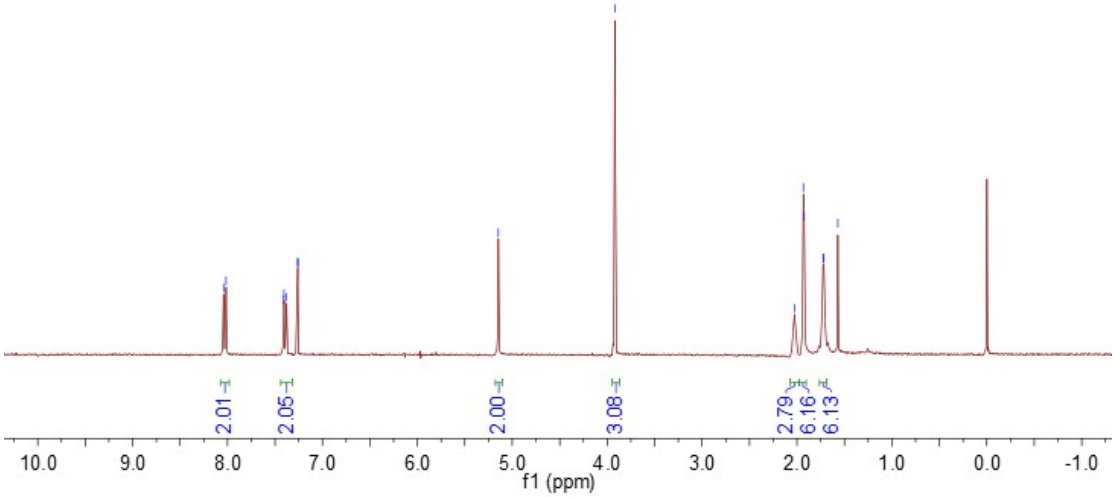

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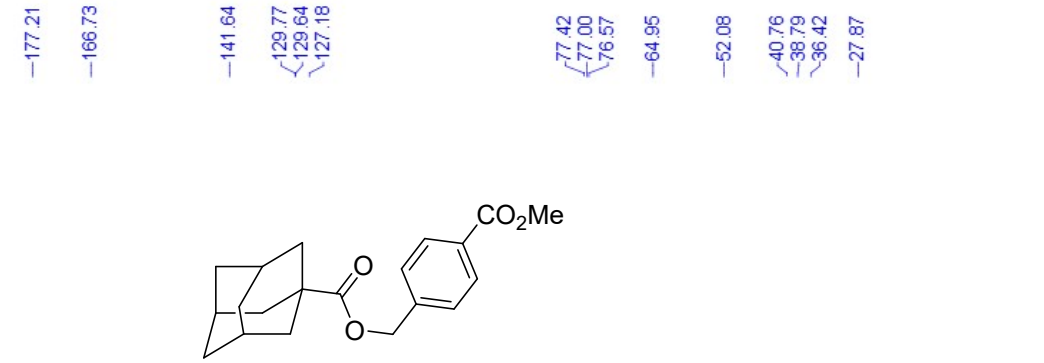

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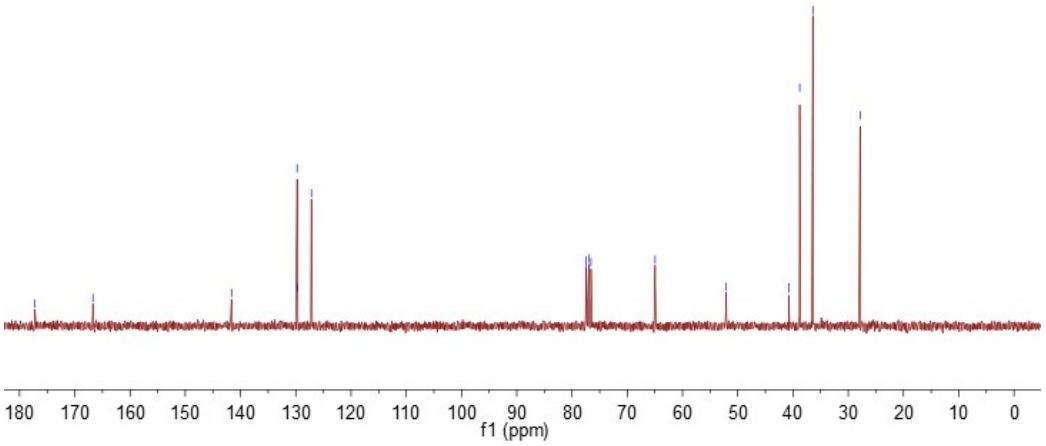

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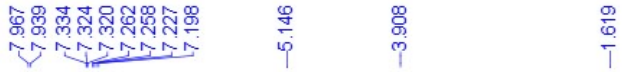

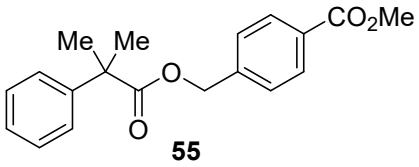

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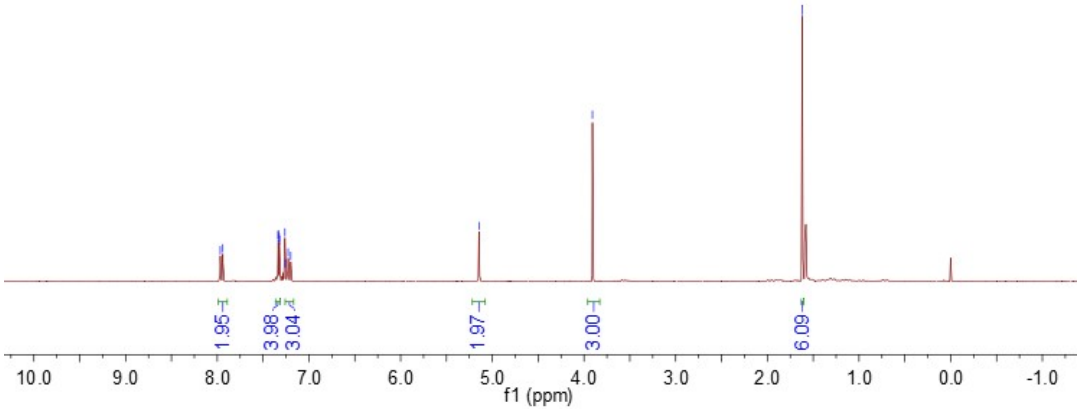

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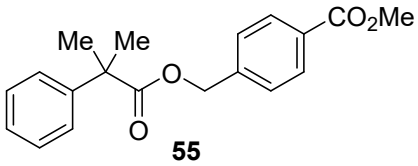

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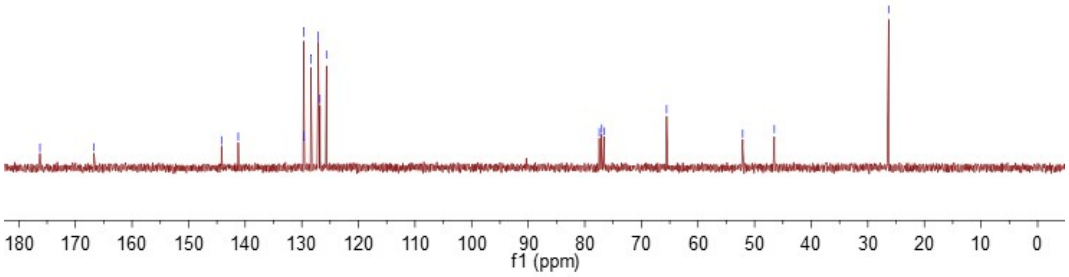

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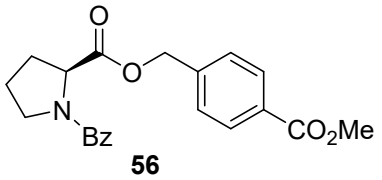

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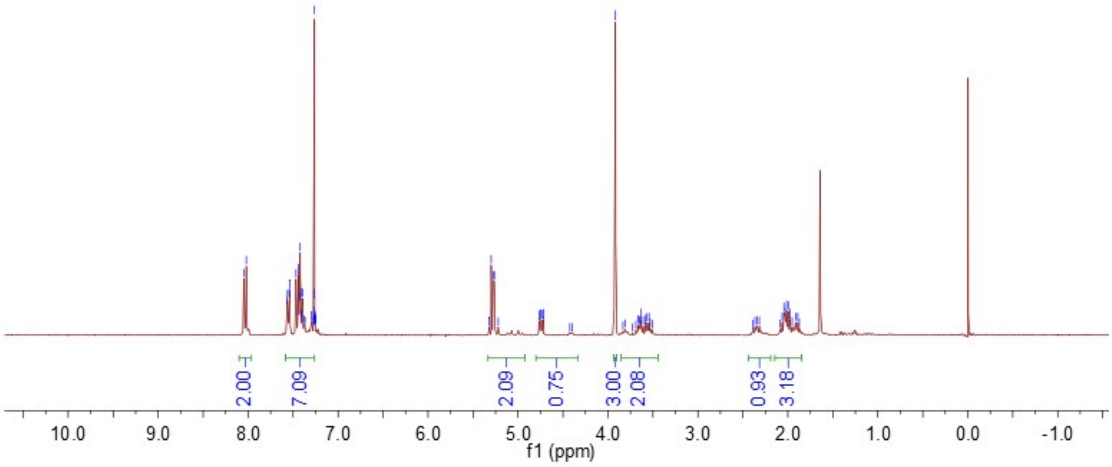

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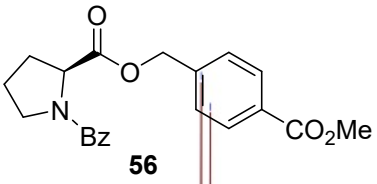

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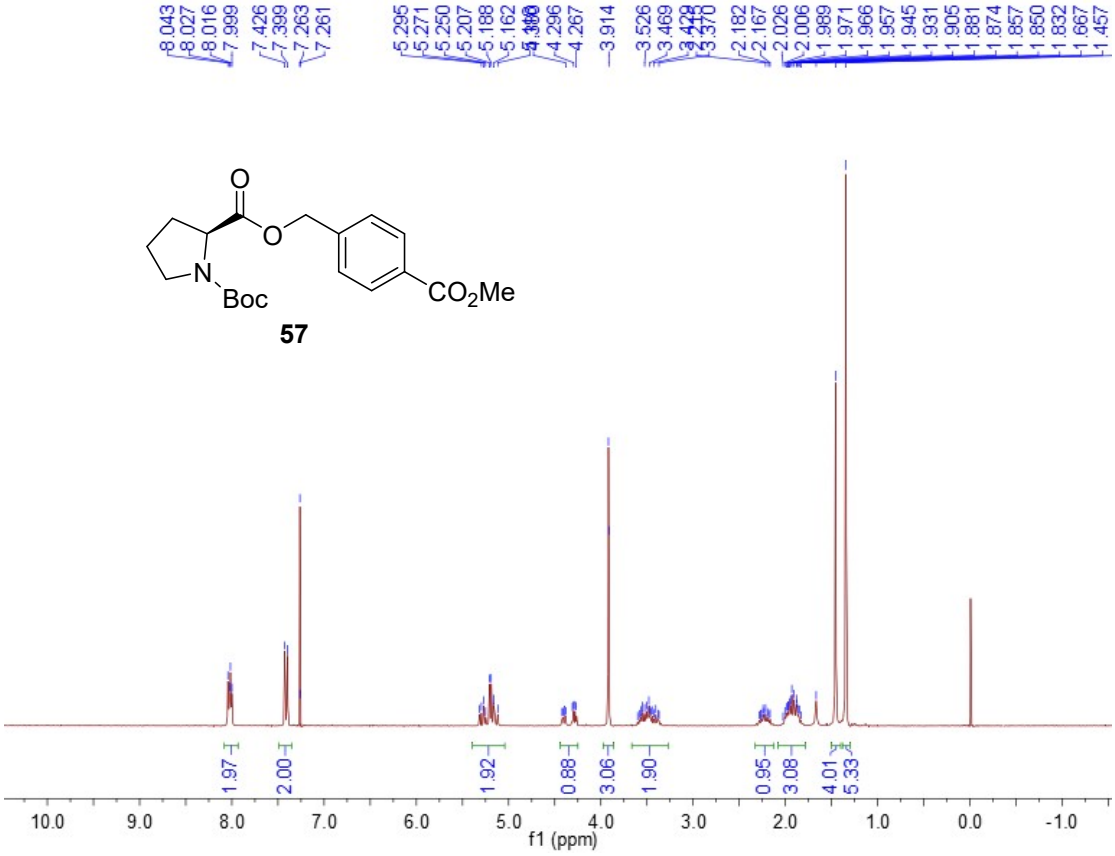

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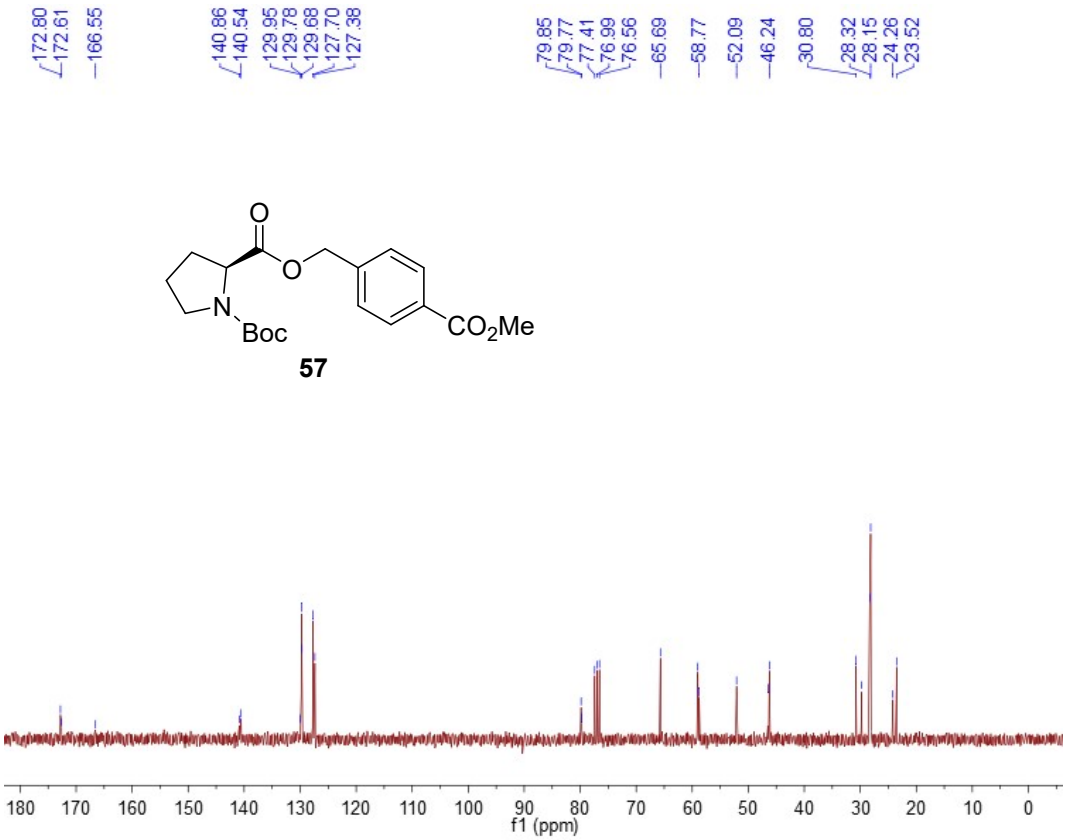

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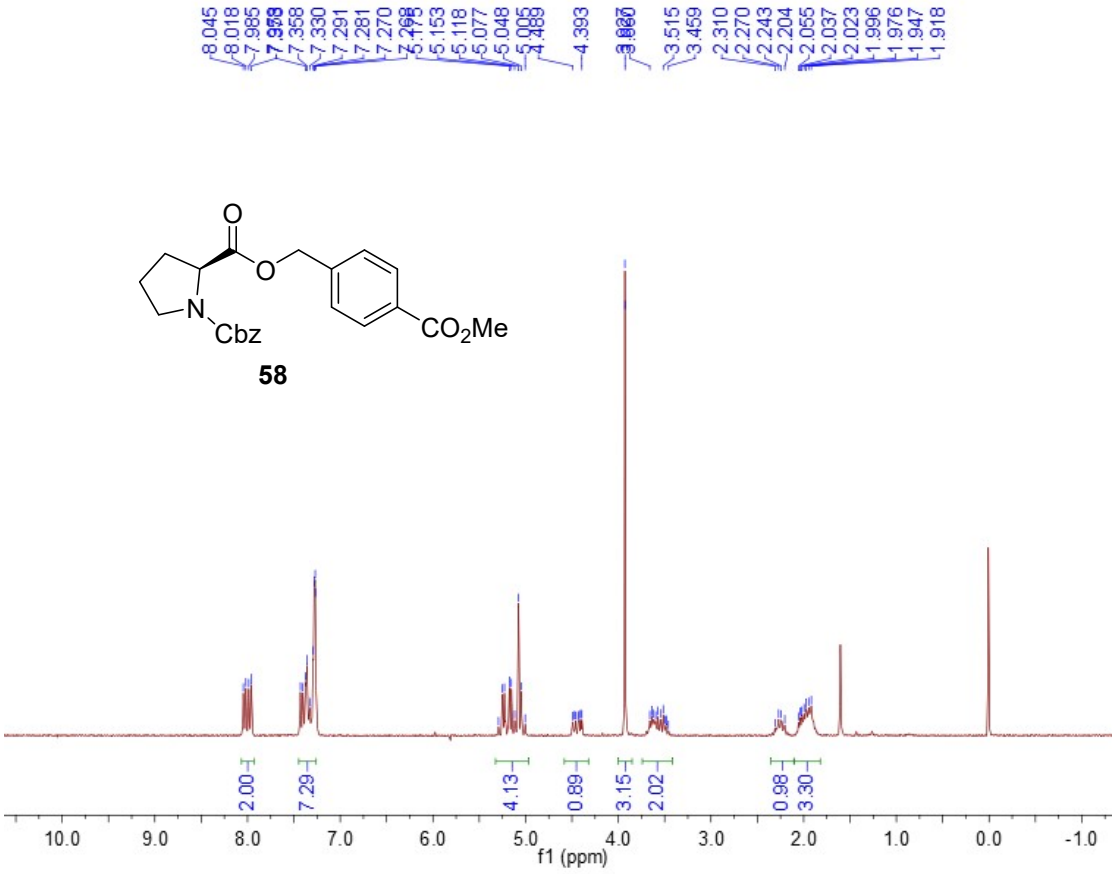

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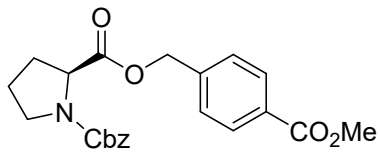

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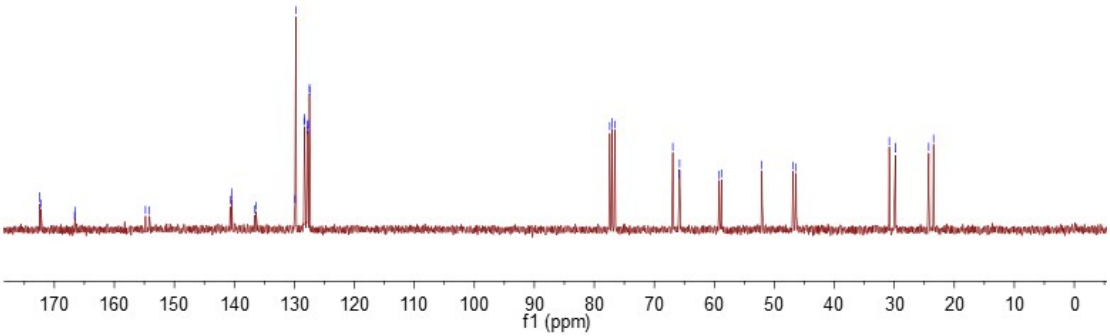

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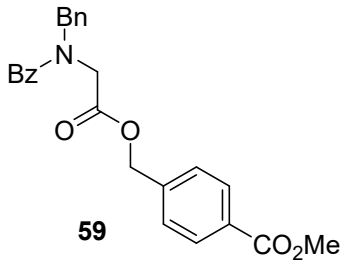

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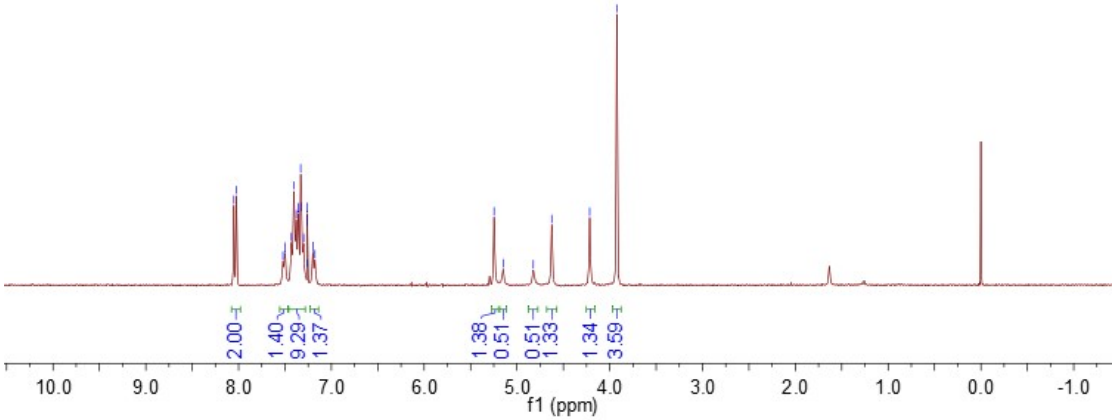

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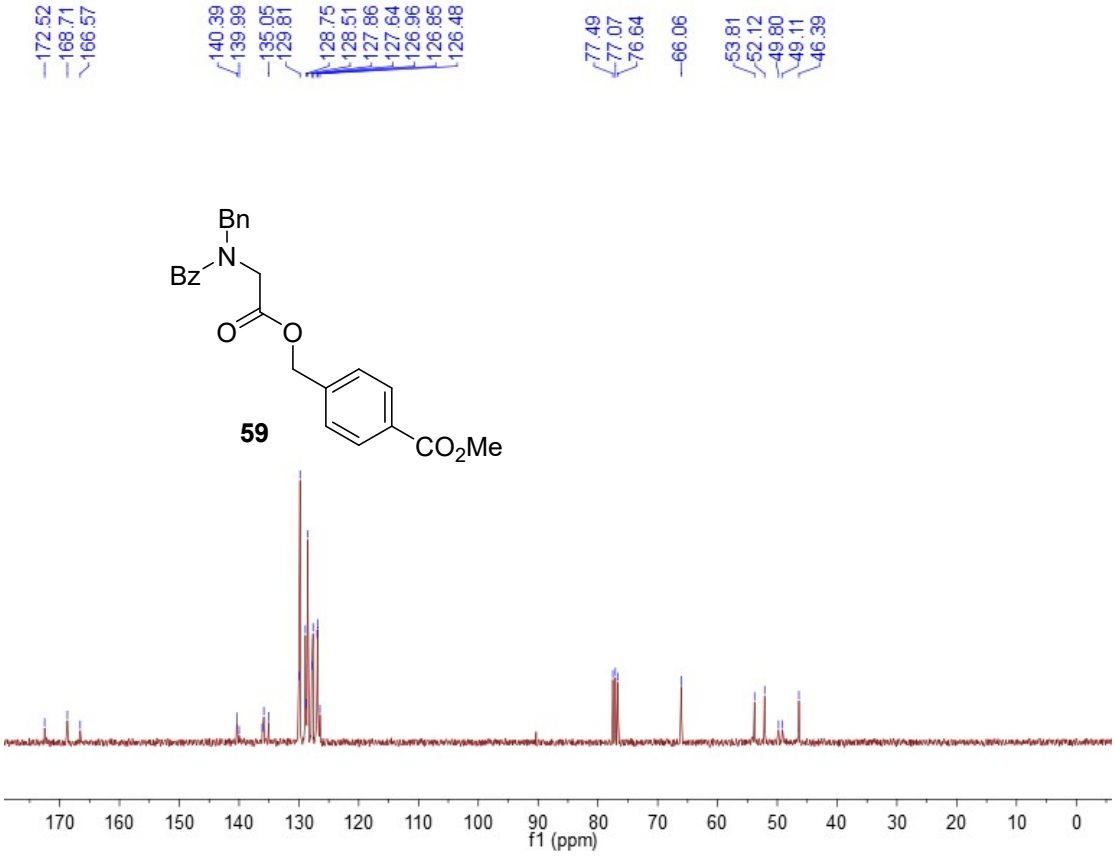

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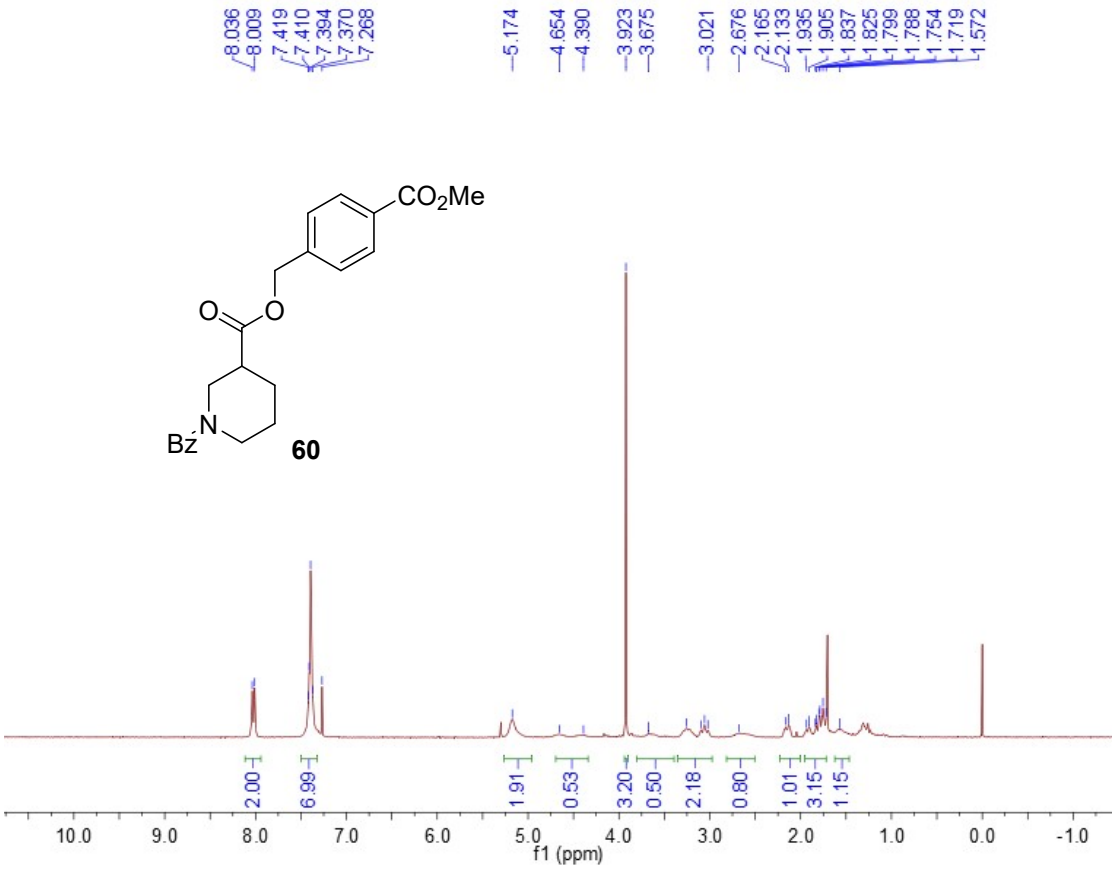

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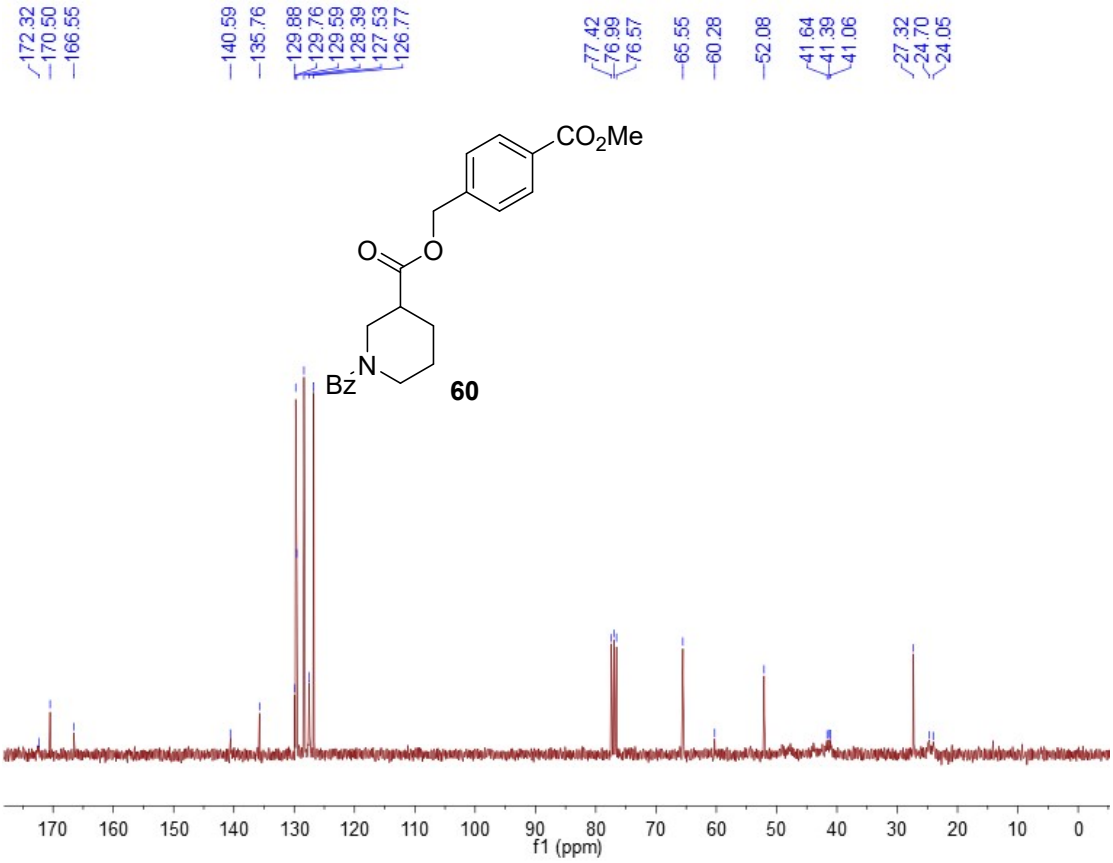

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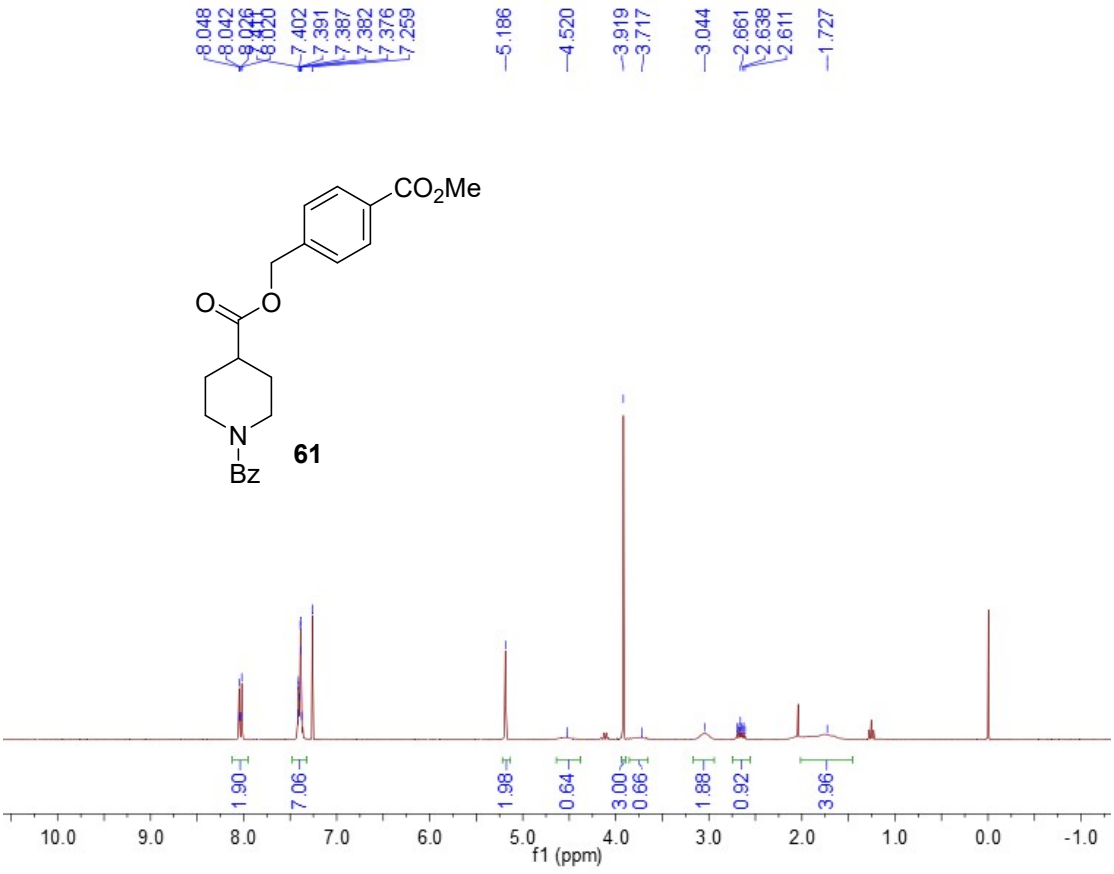
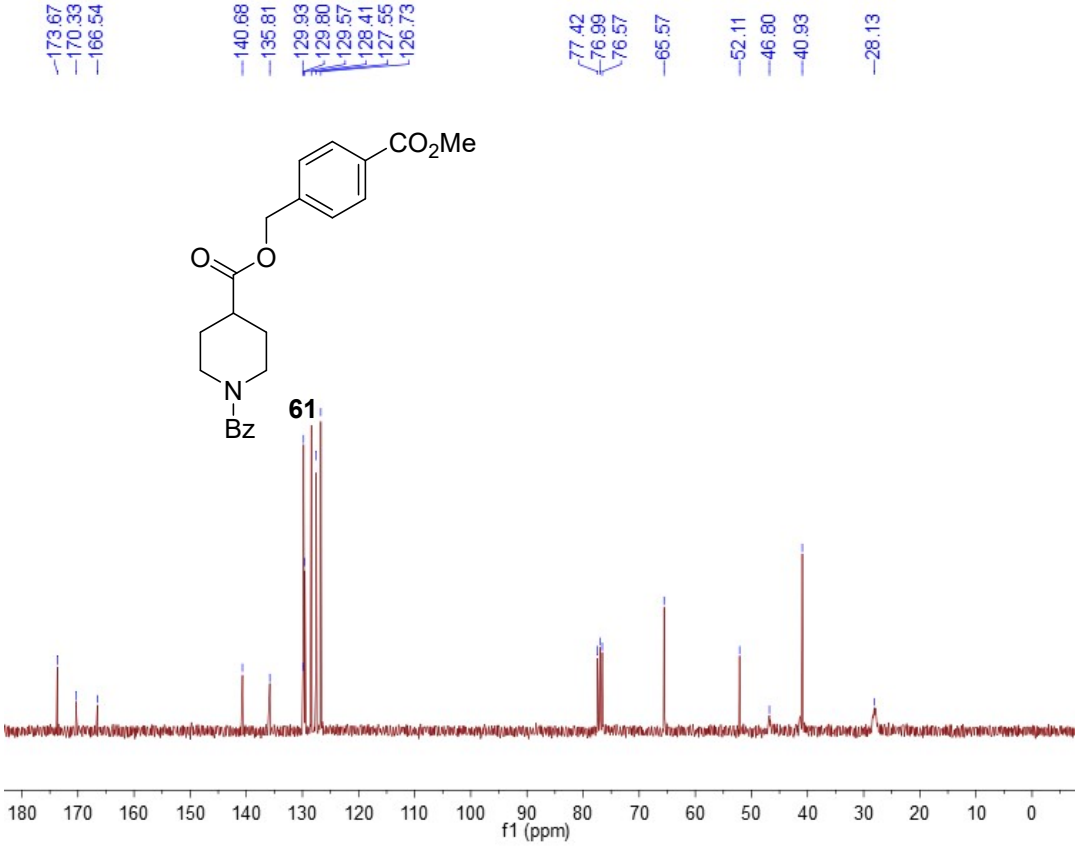


^{13}C NMR (75 MHz, $CDCl_3$):



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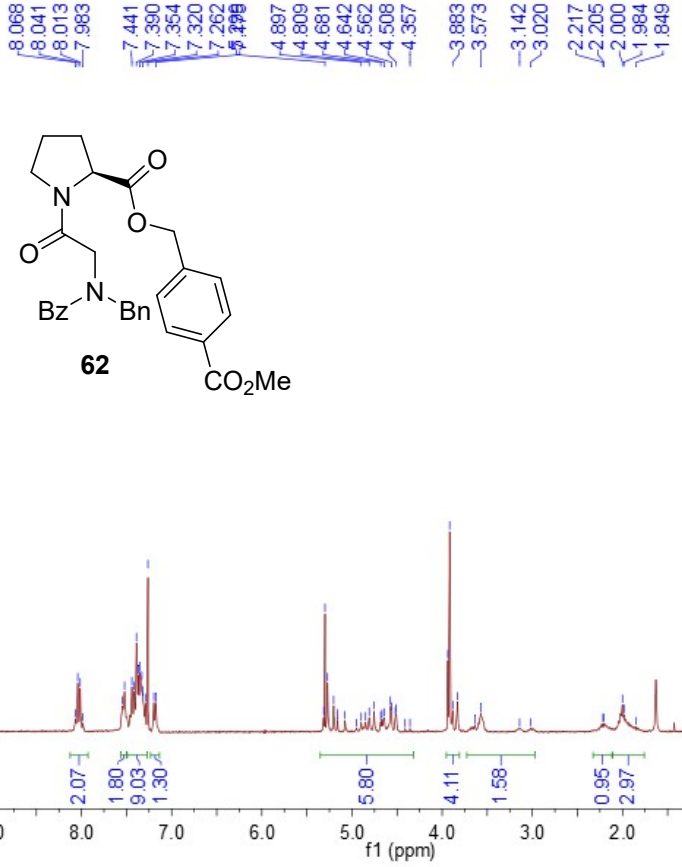
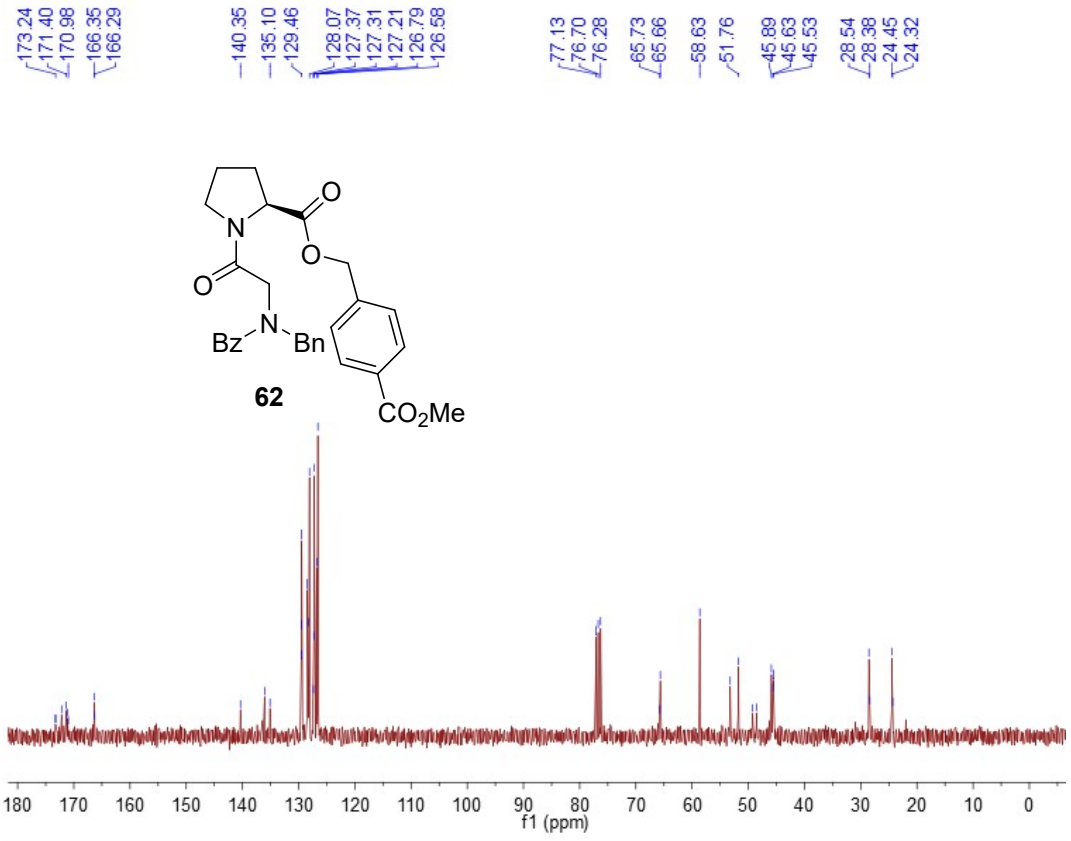


^{13}C NMR (75 MHz, $CDCl_3$):



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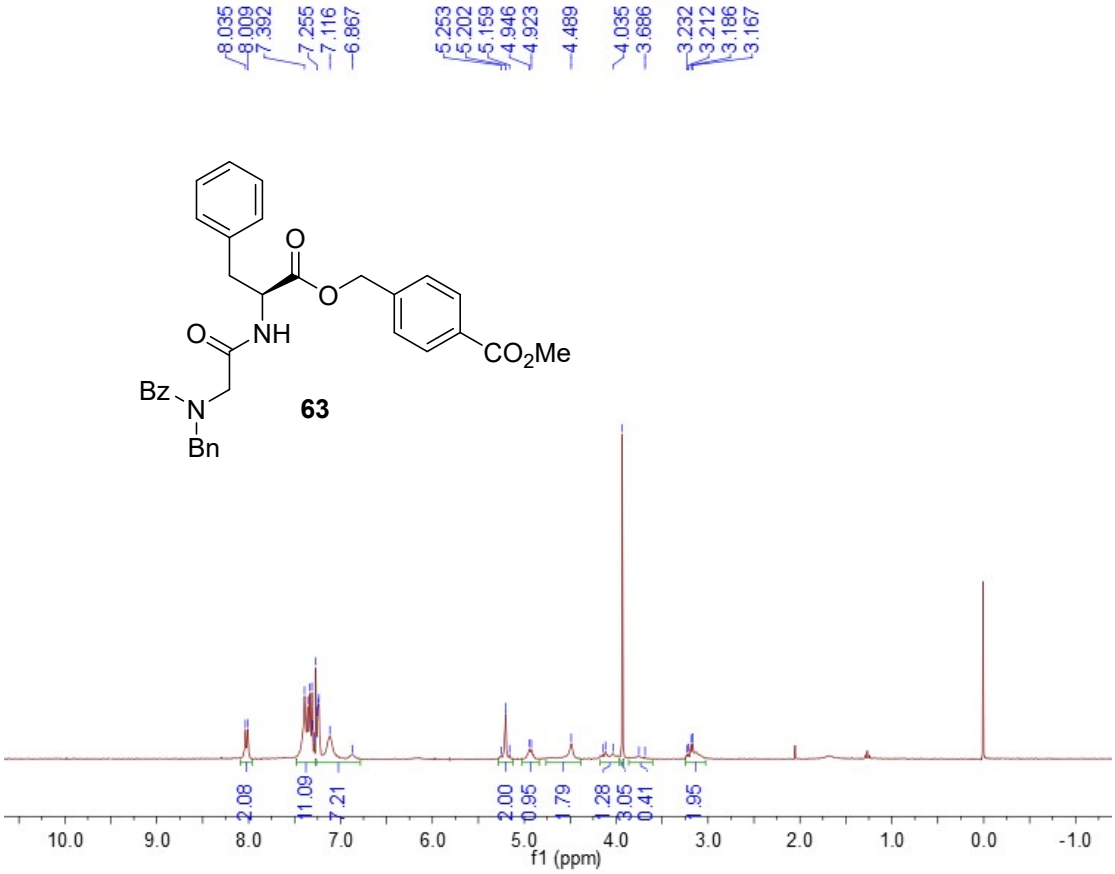

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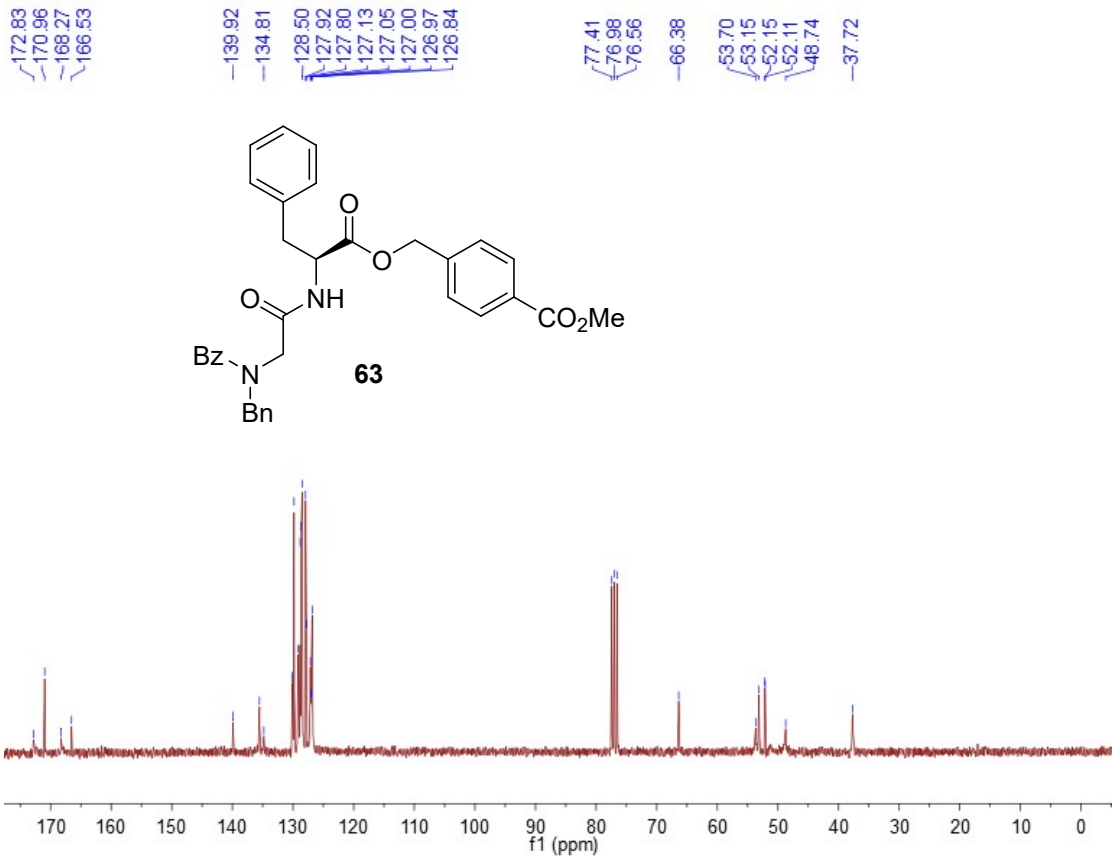

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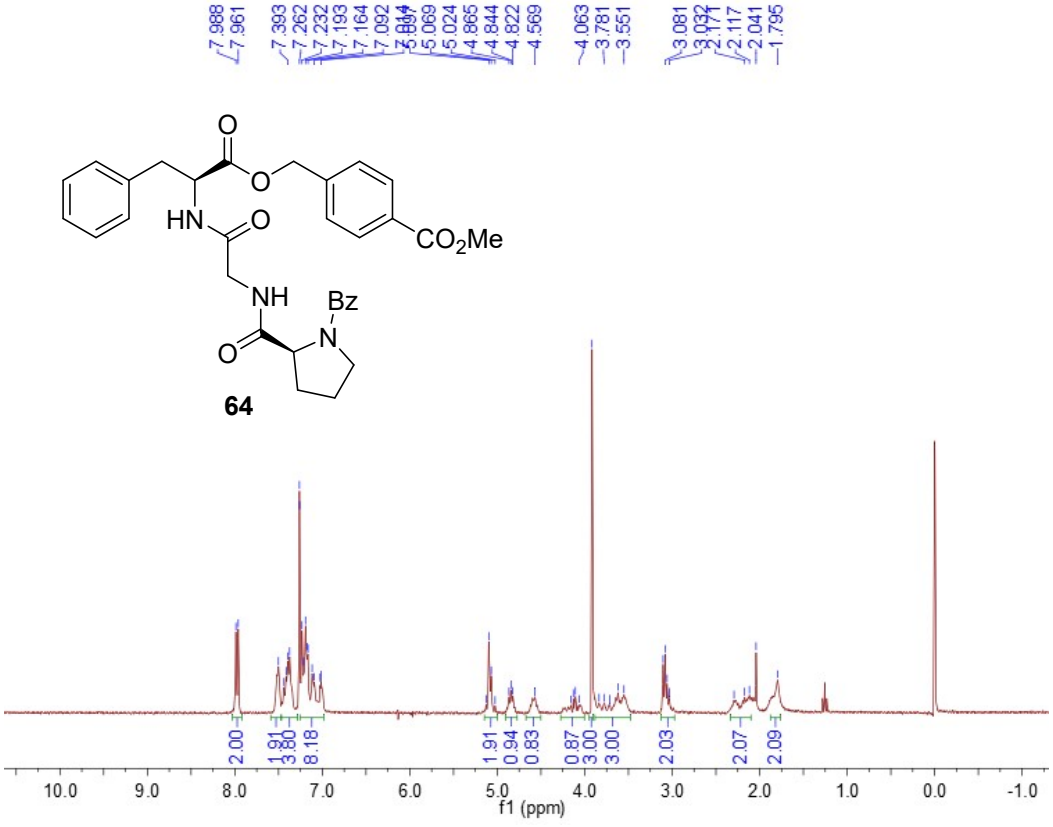

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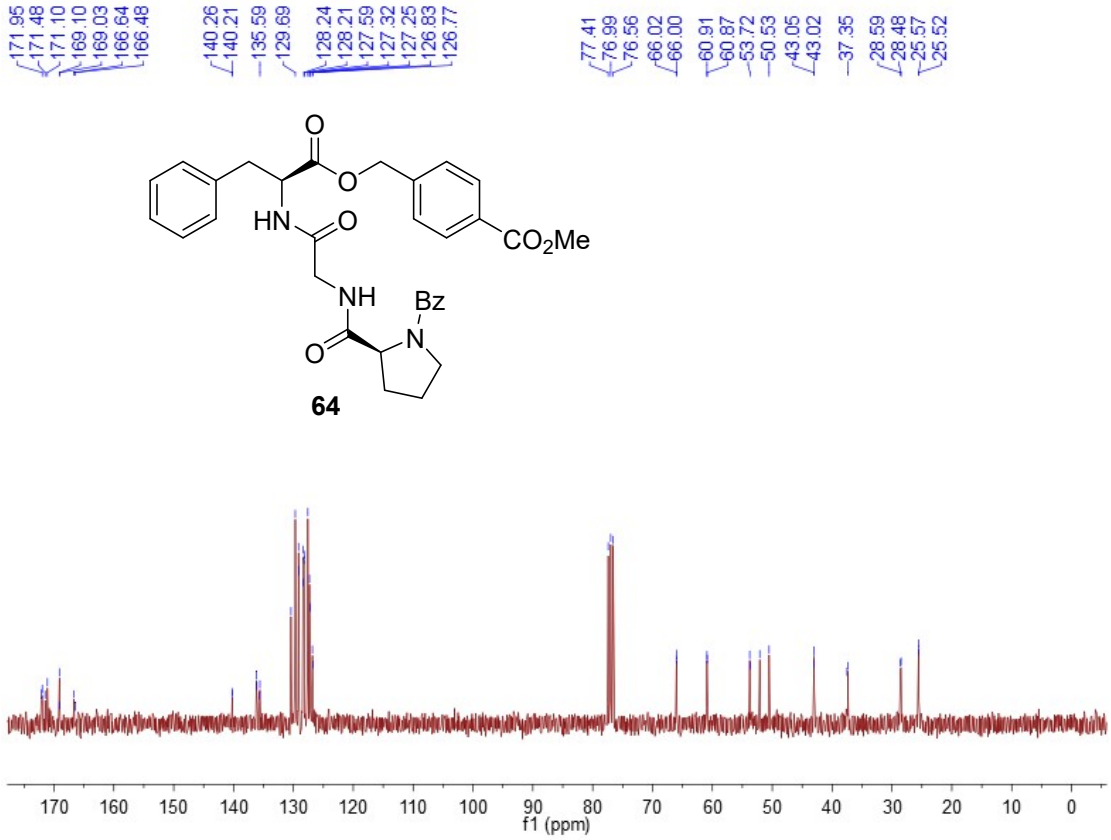

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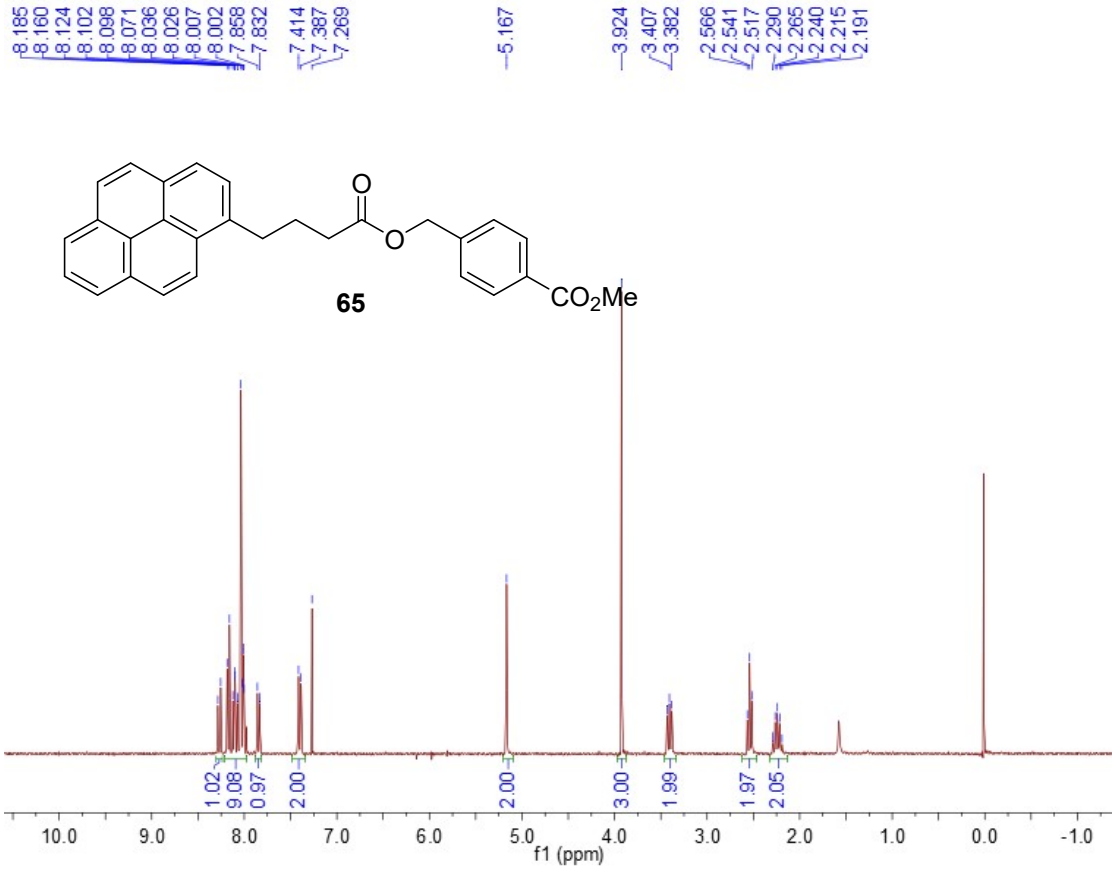
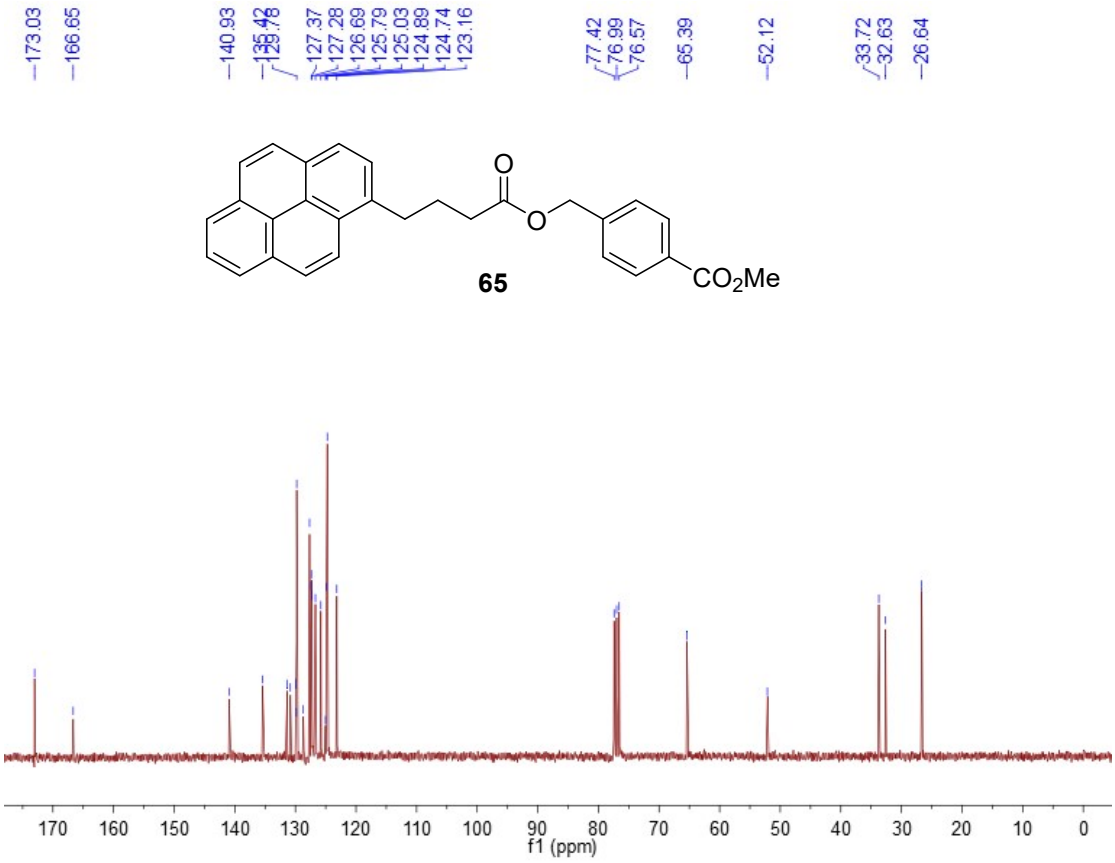


^{13}C NMR (75 MHz, $CDCl_3$):



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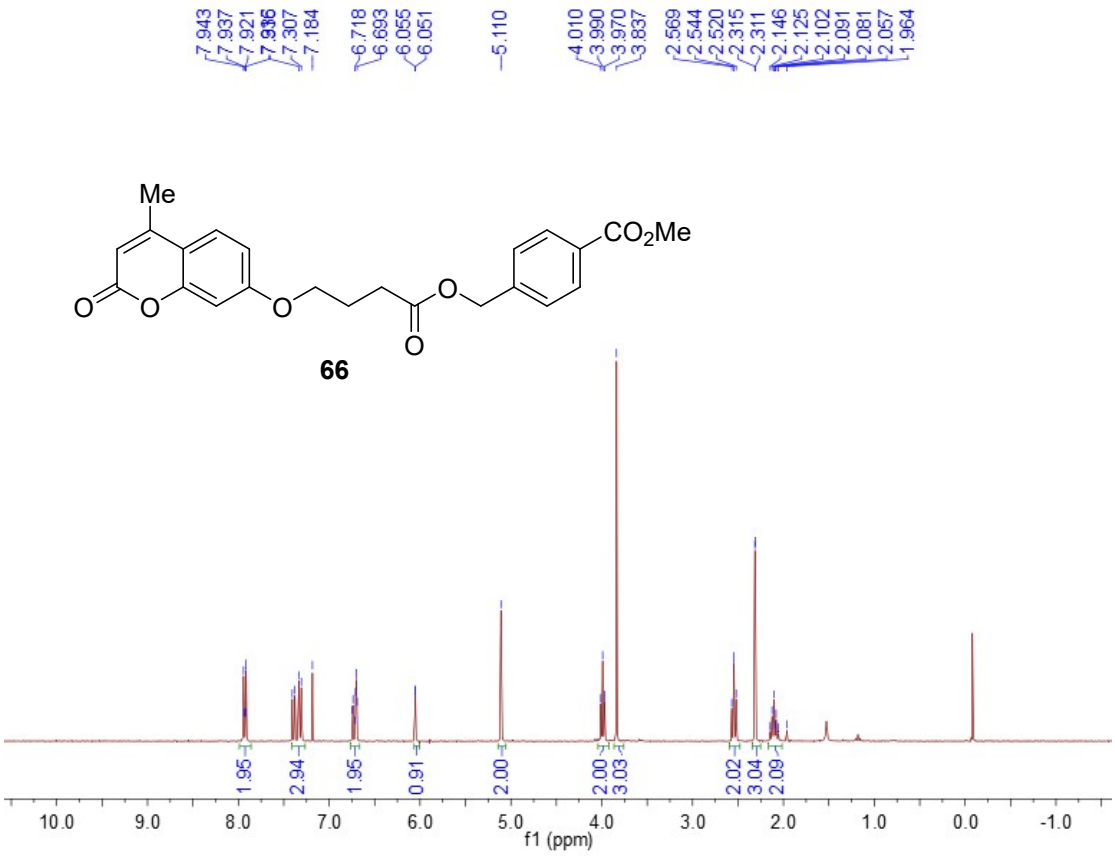

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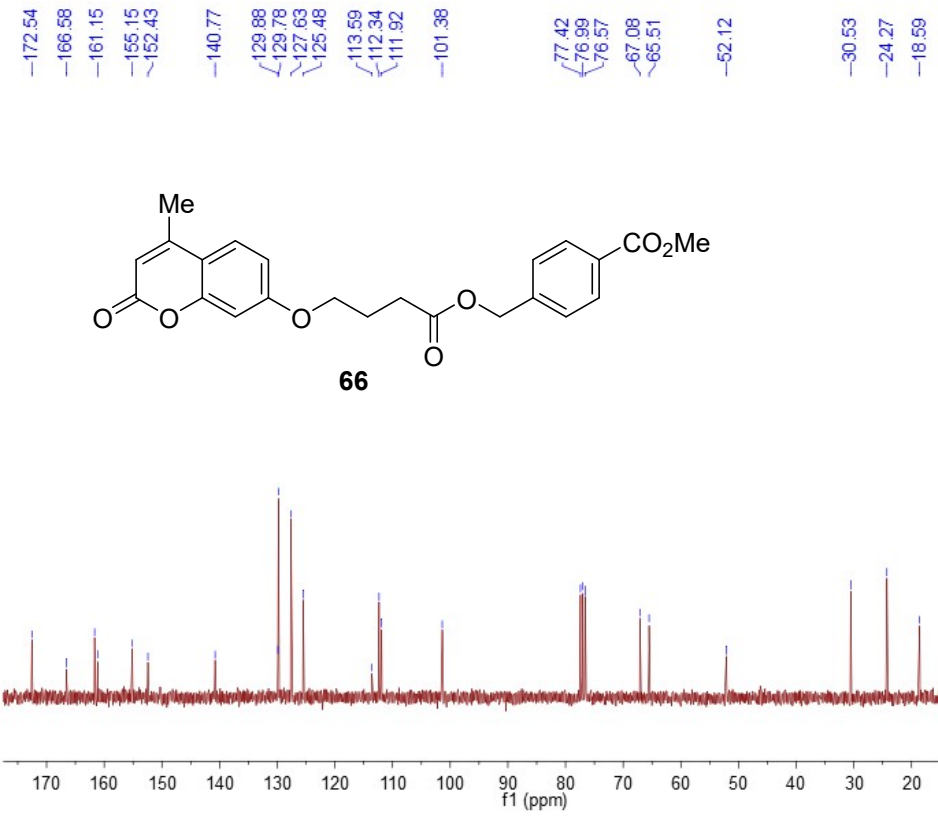

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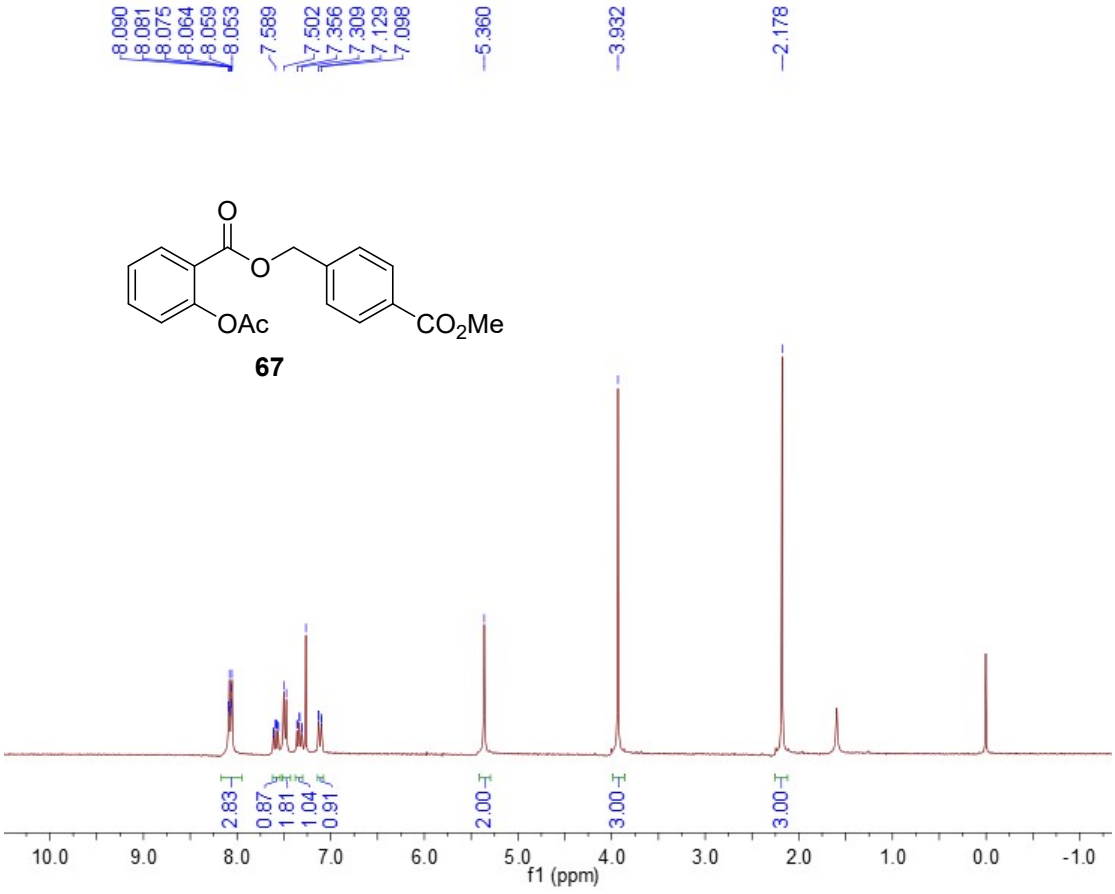

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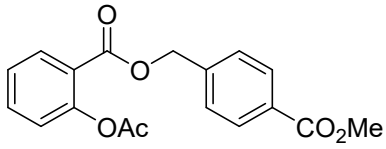

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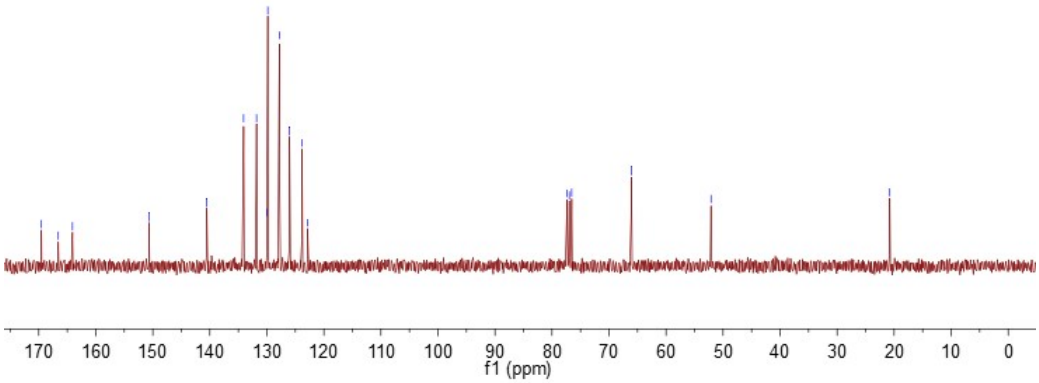

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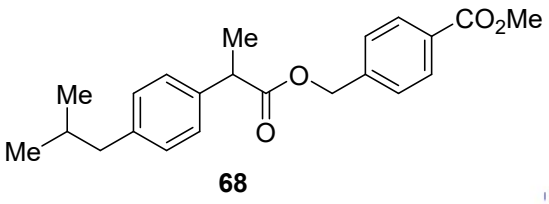

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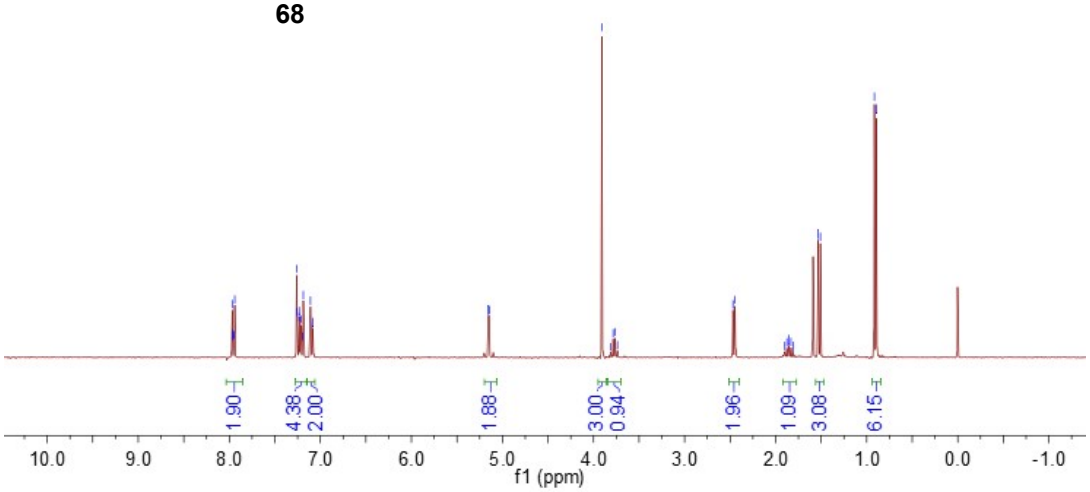

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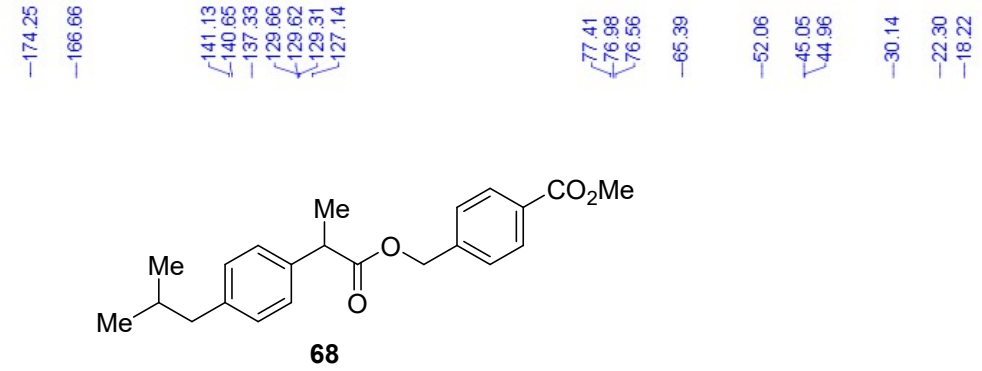

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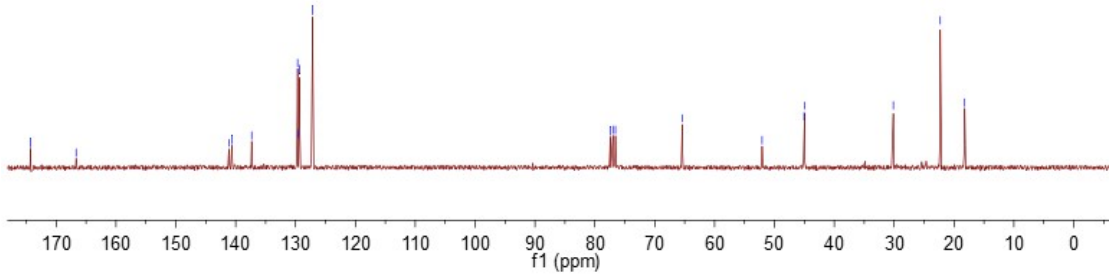

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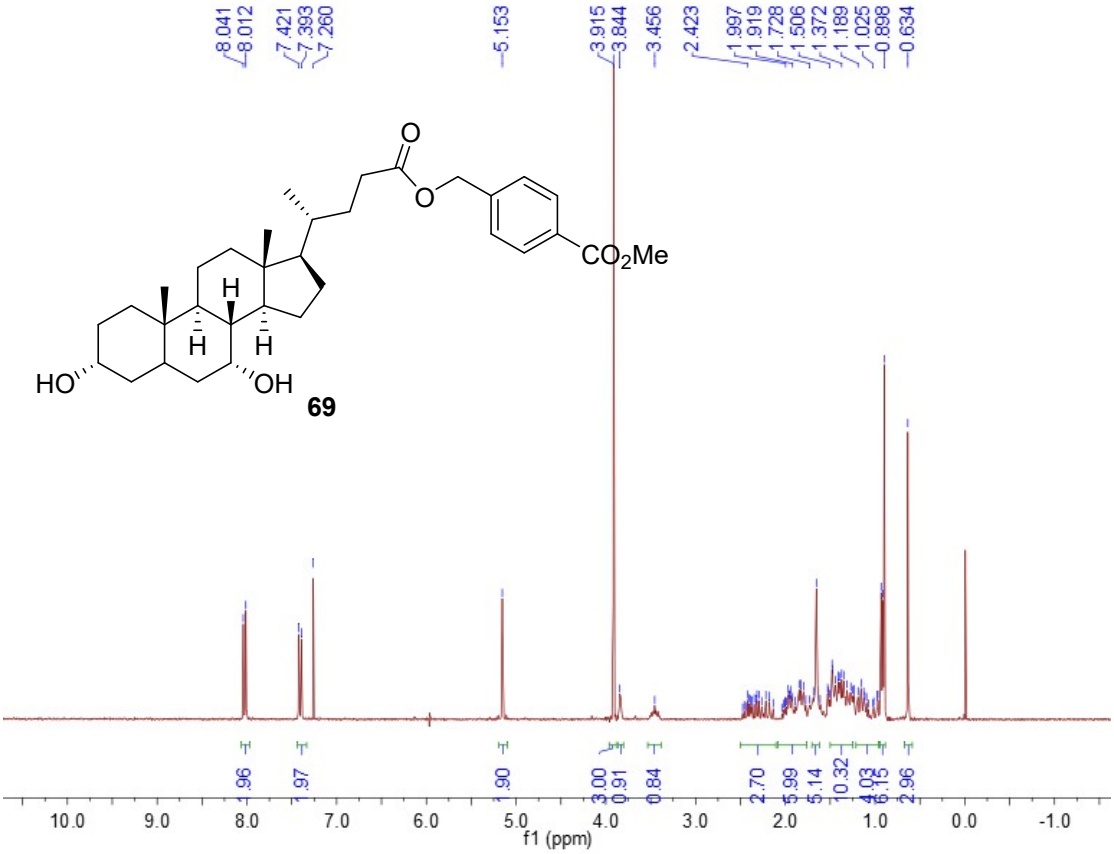

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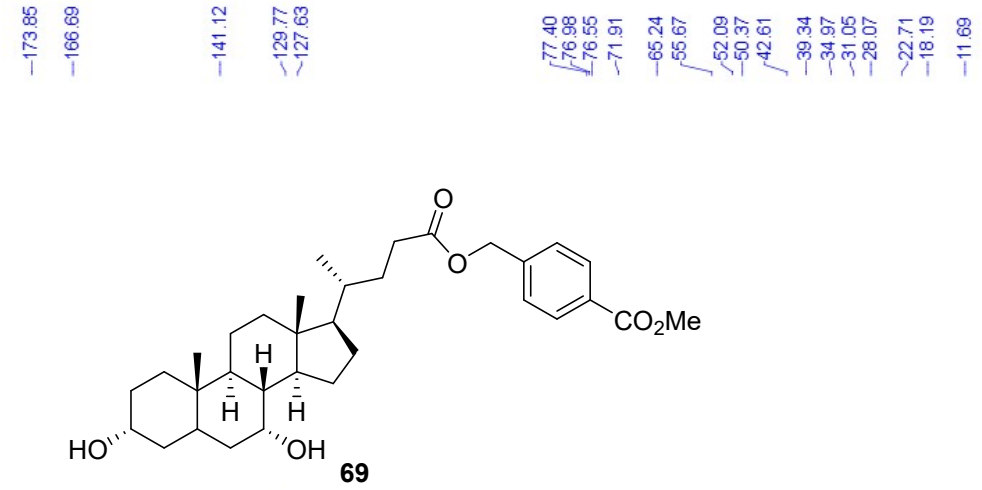

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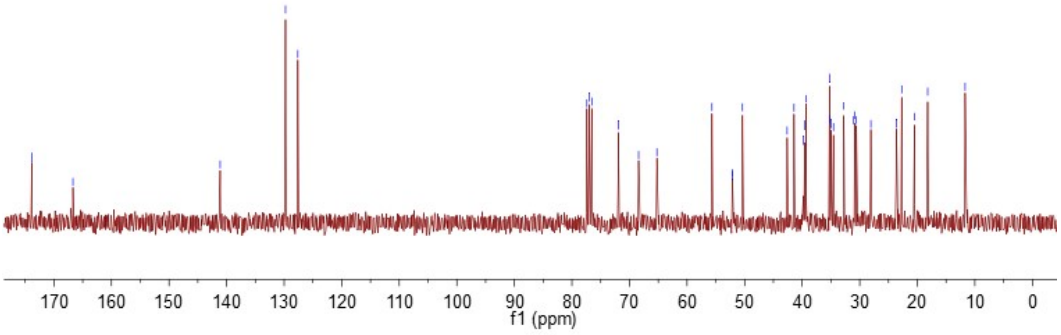
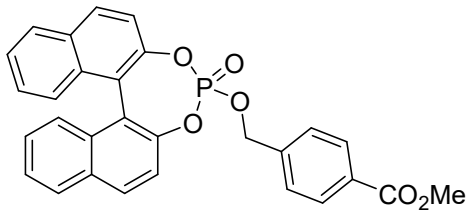


^{1}H NMR (300 MHz, $CDCl_3$):



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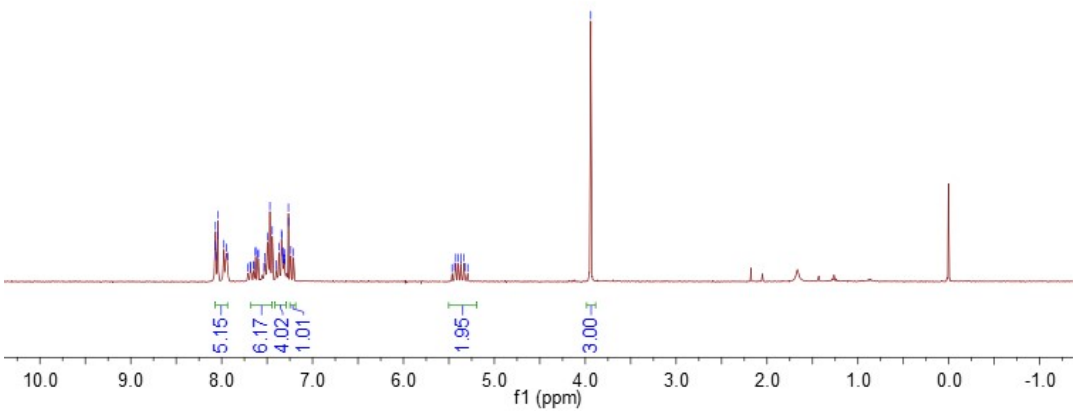

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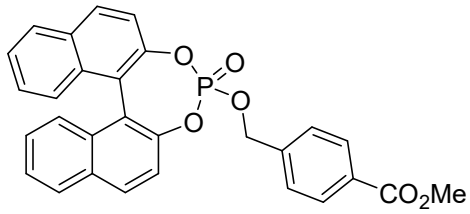

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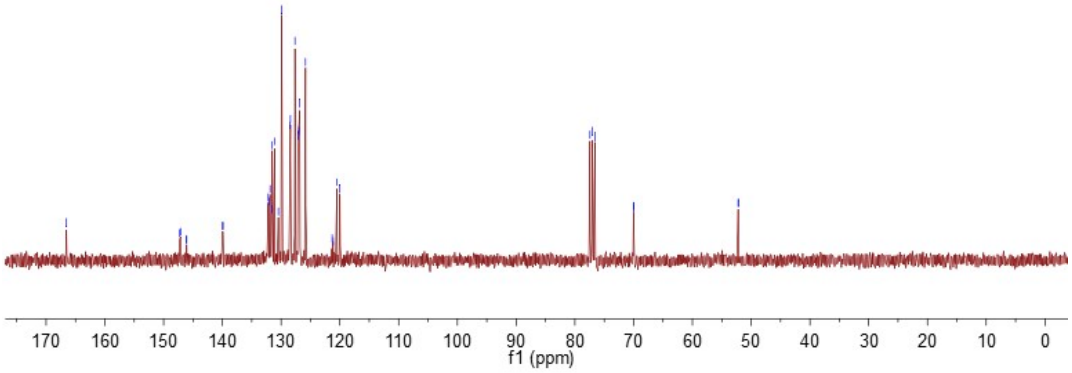
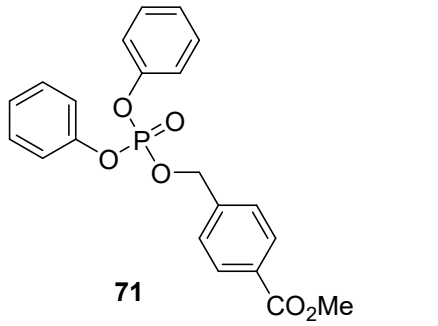


^{1}H NMR (300 MHz, $CDCl_3$):



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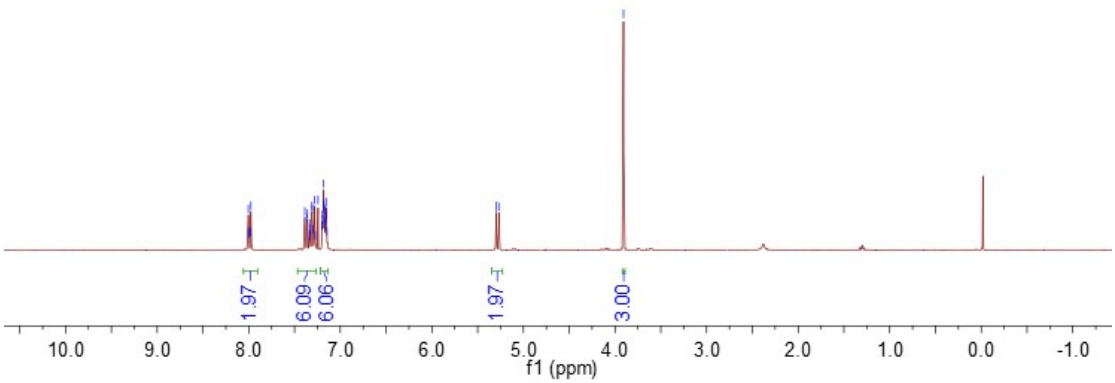

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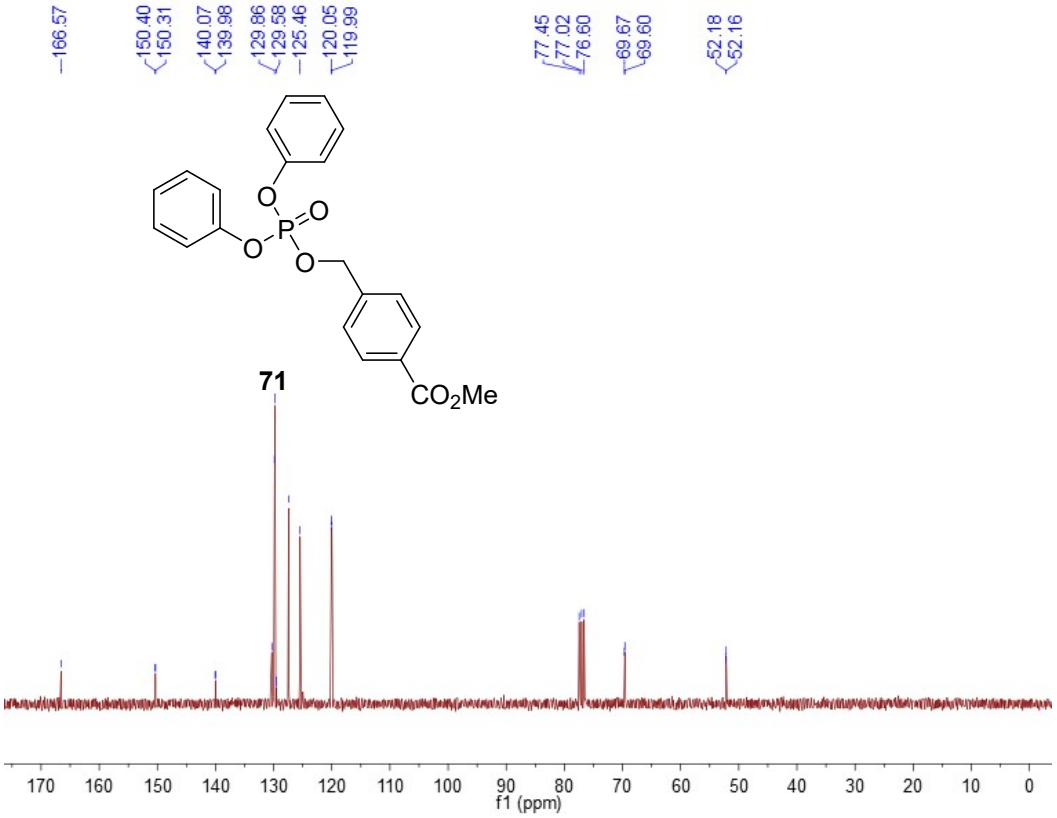

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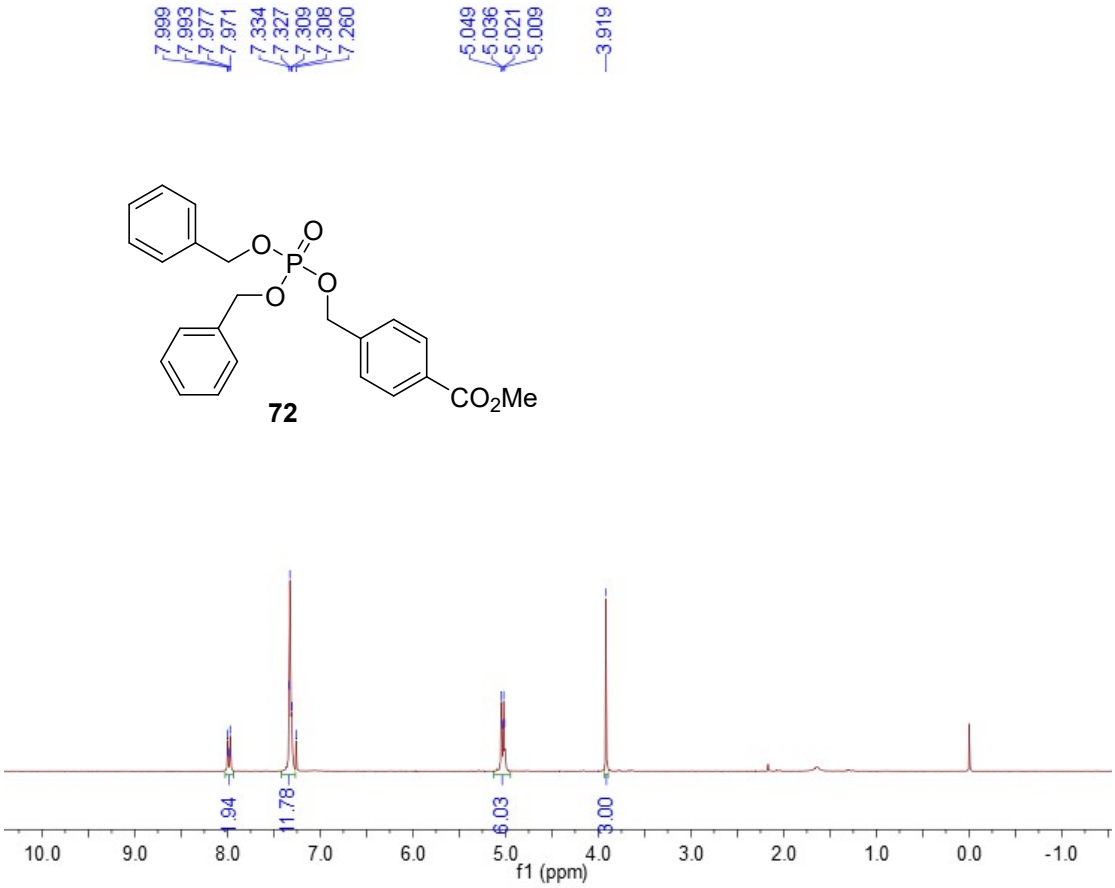

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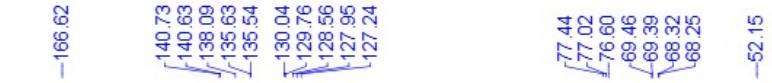

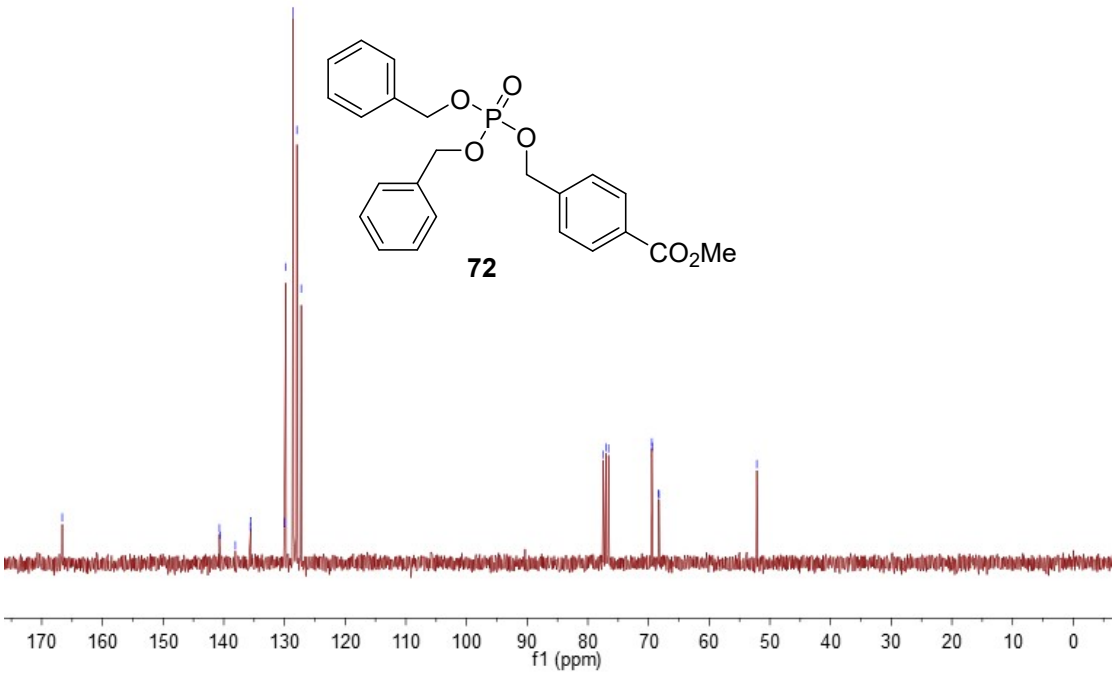

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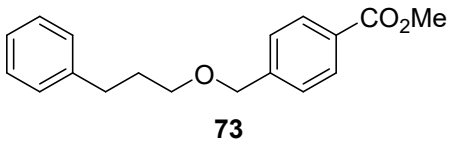

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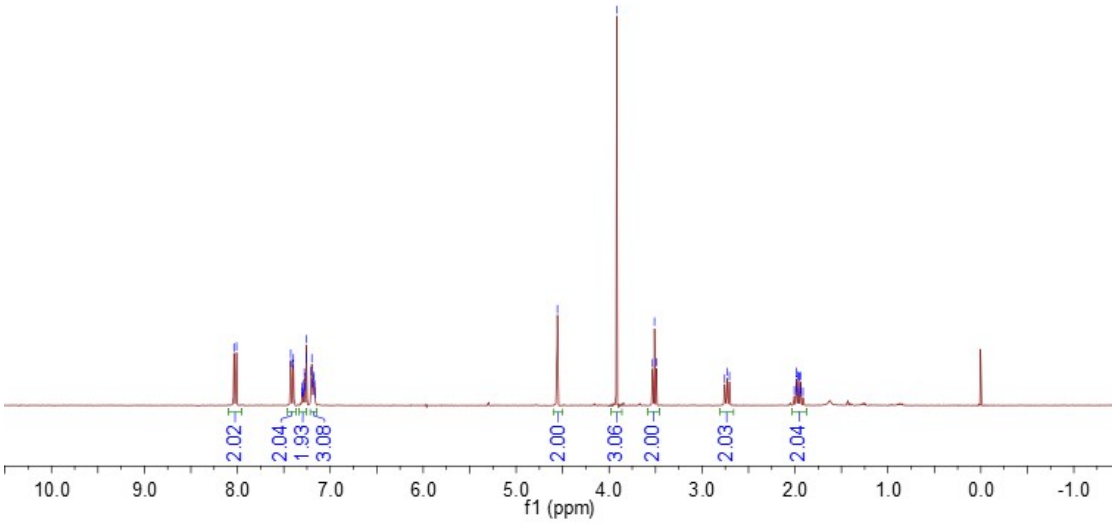

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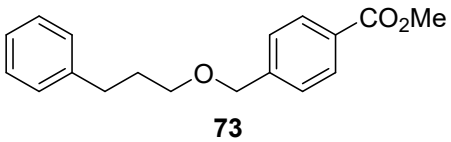

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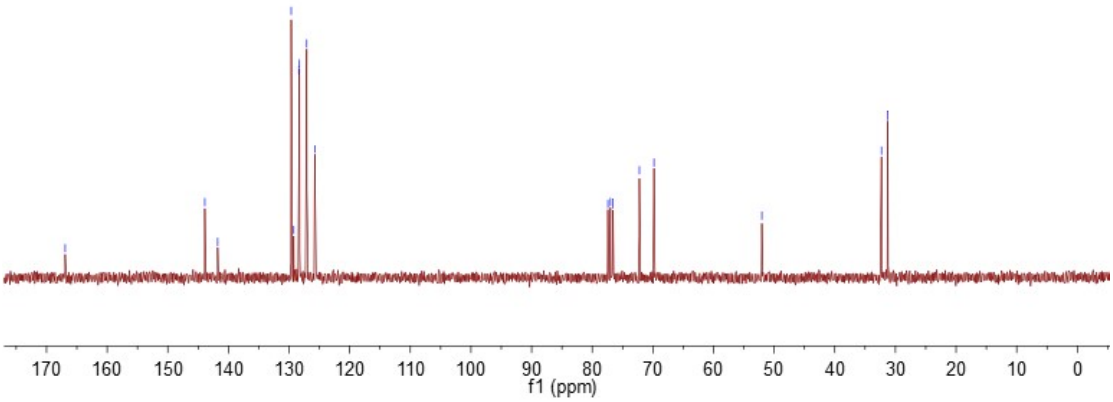

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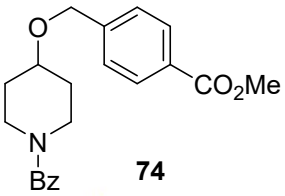

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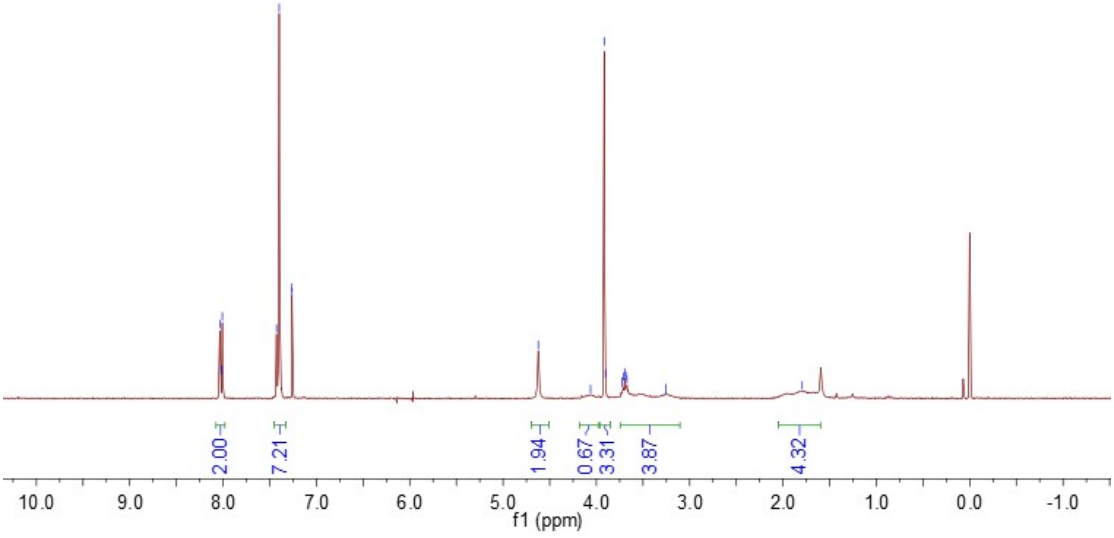

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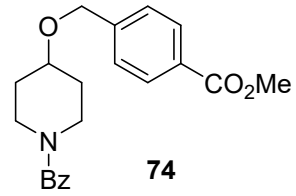

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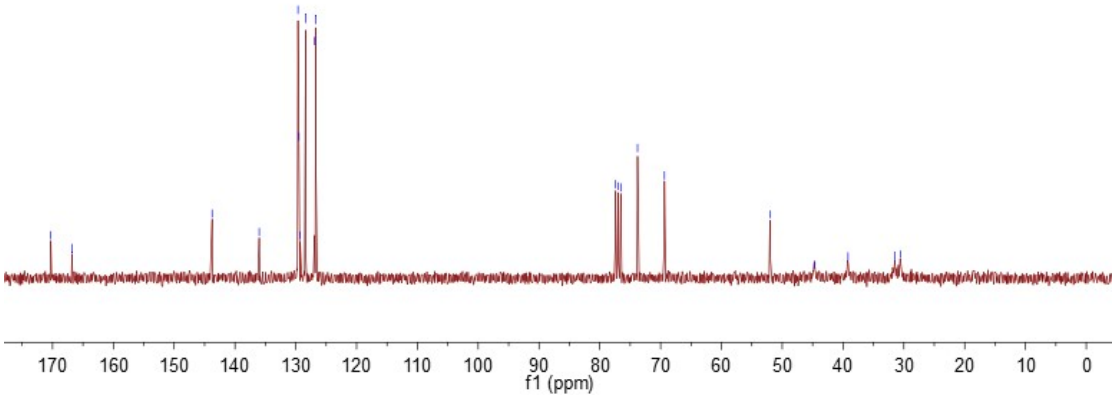

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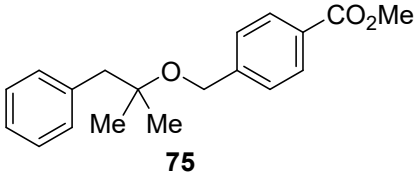

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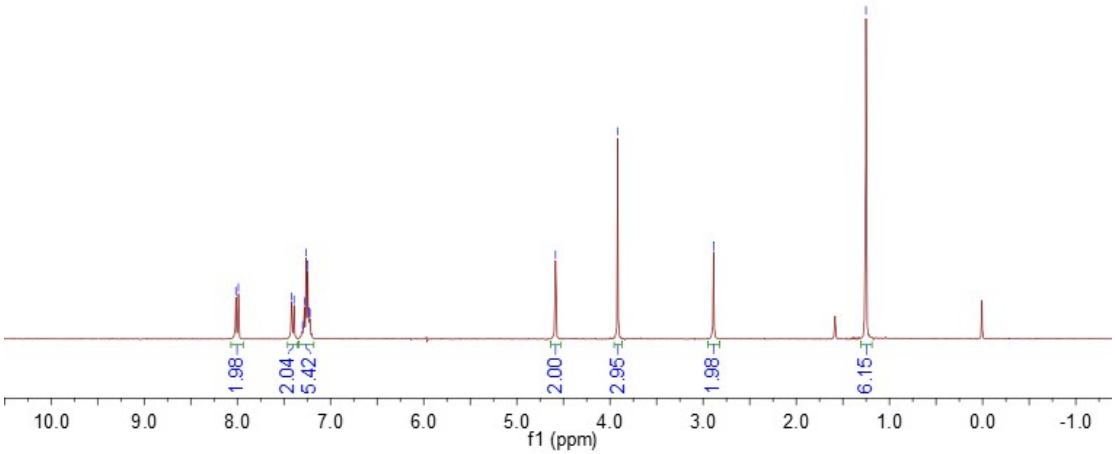

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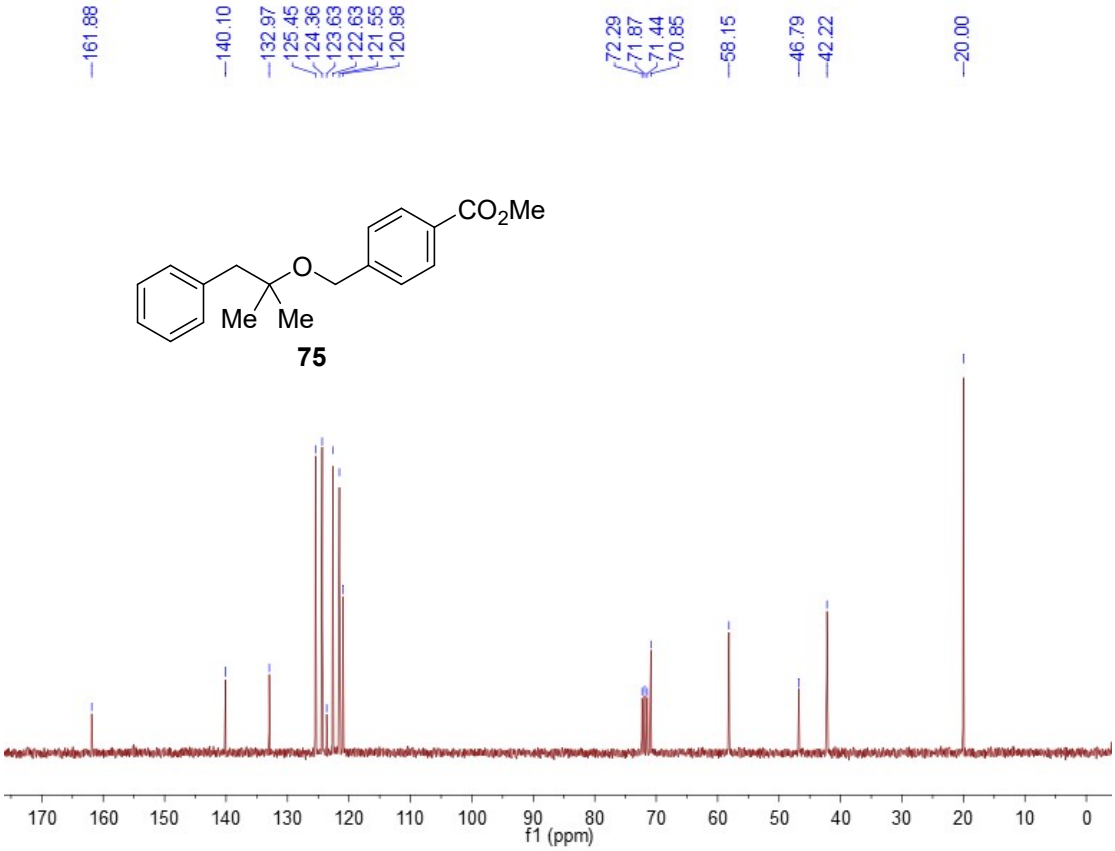

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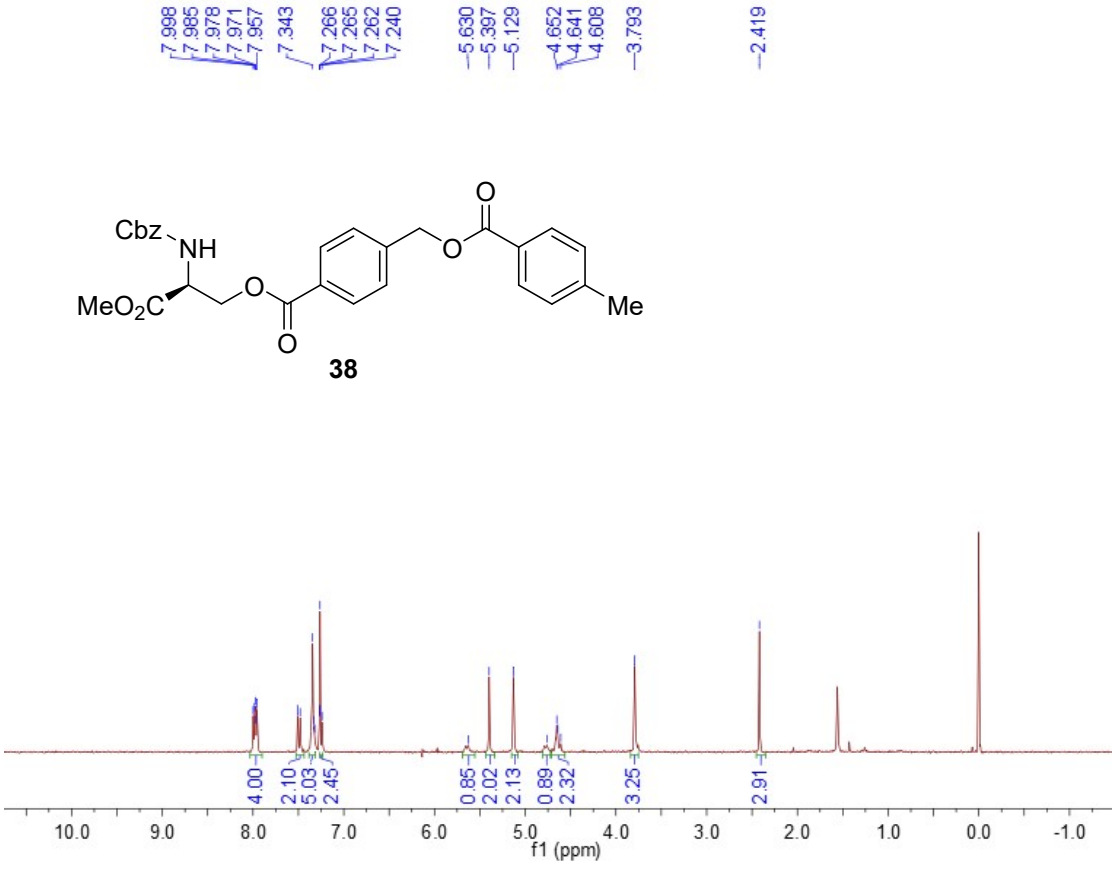

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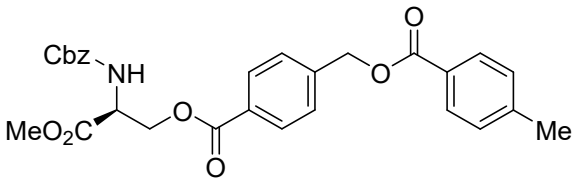

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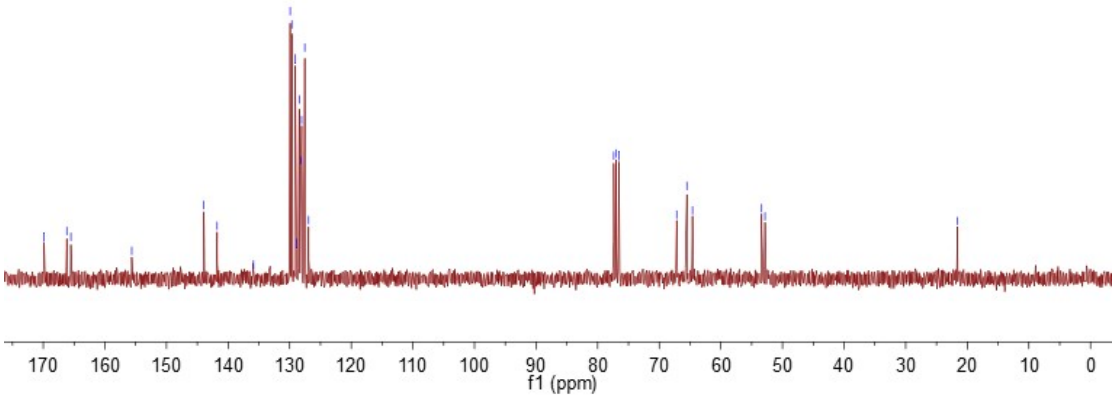

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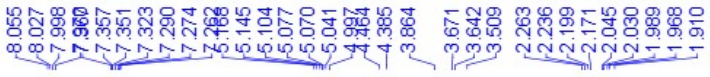

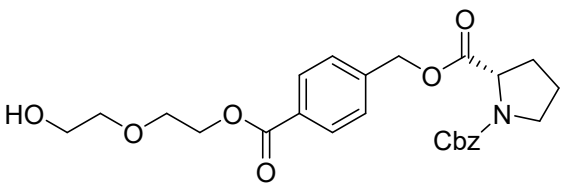

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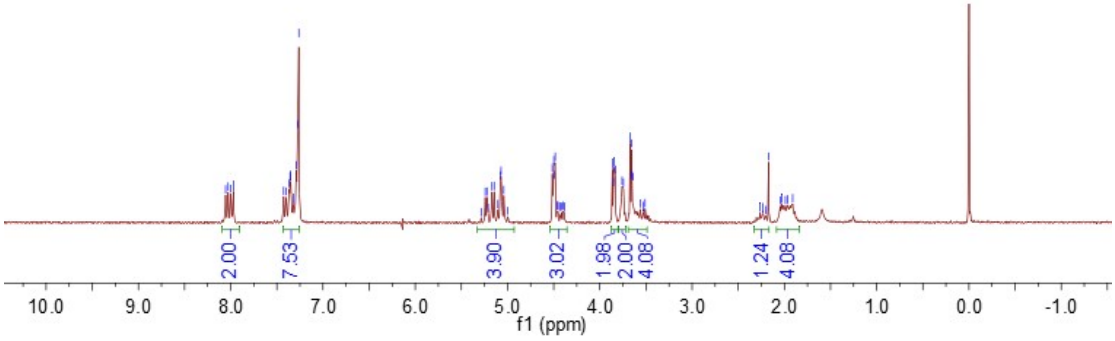

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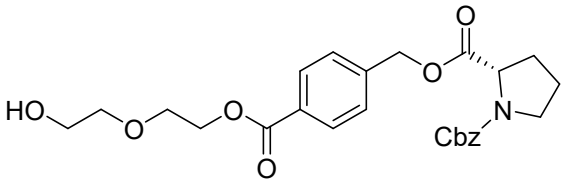

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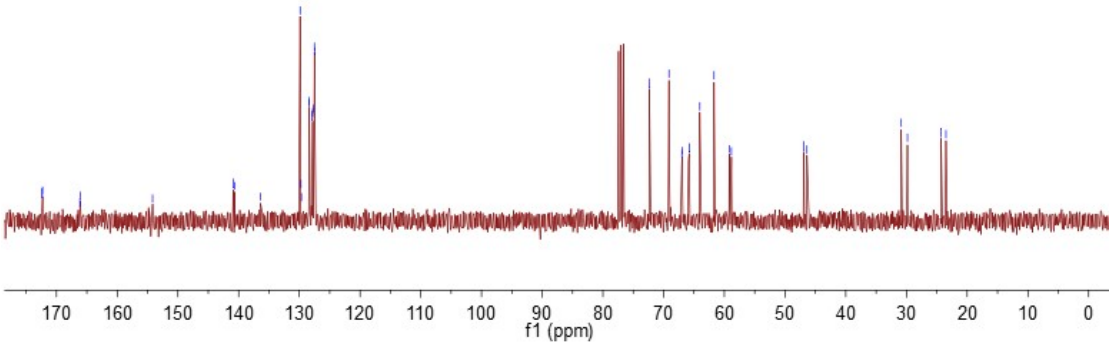

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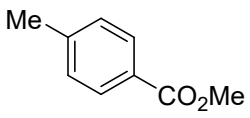

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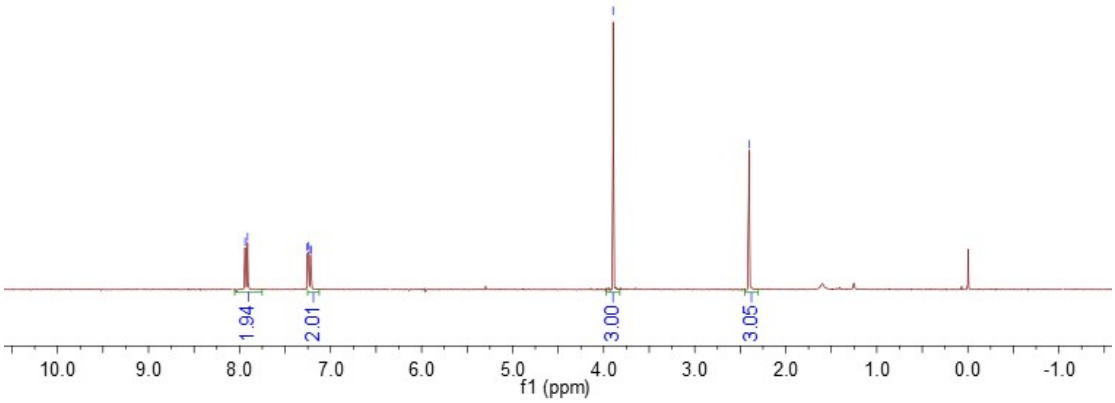

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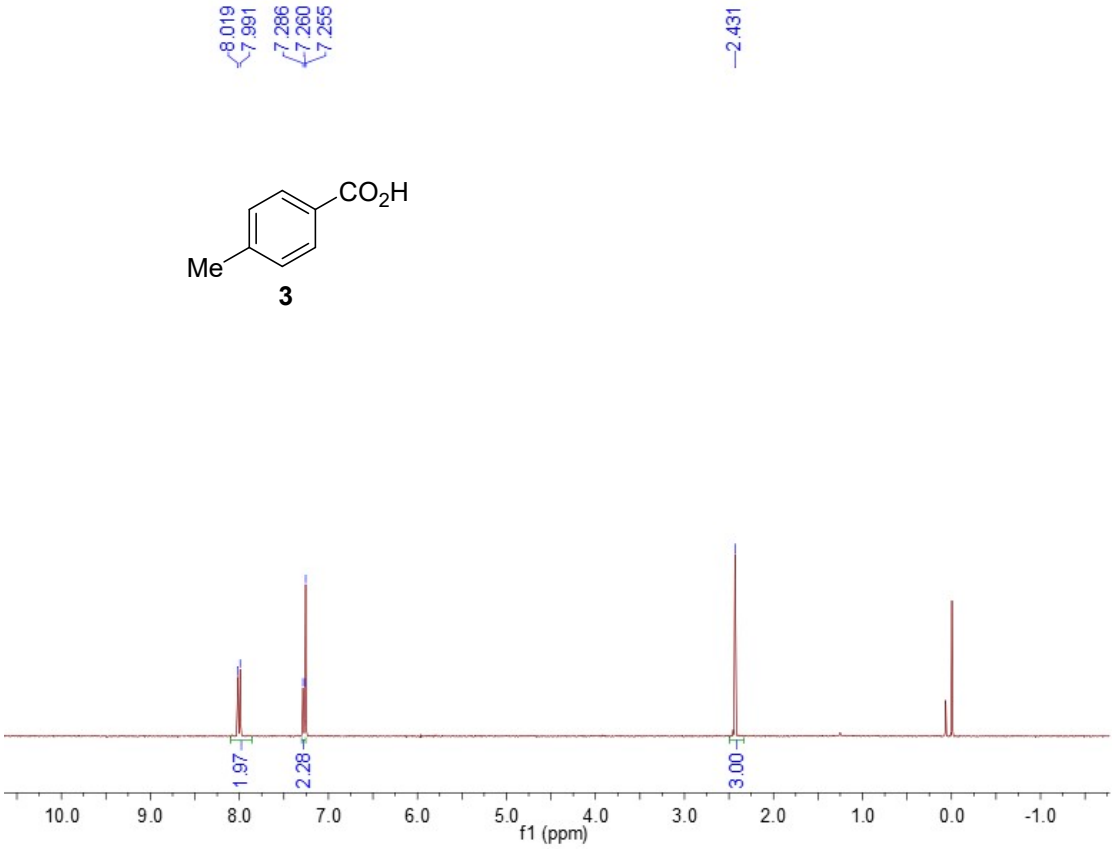

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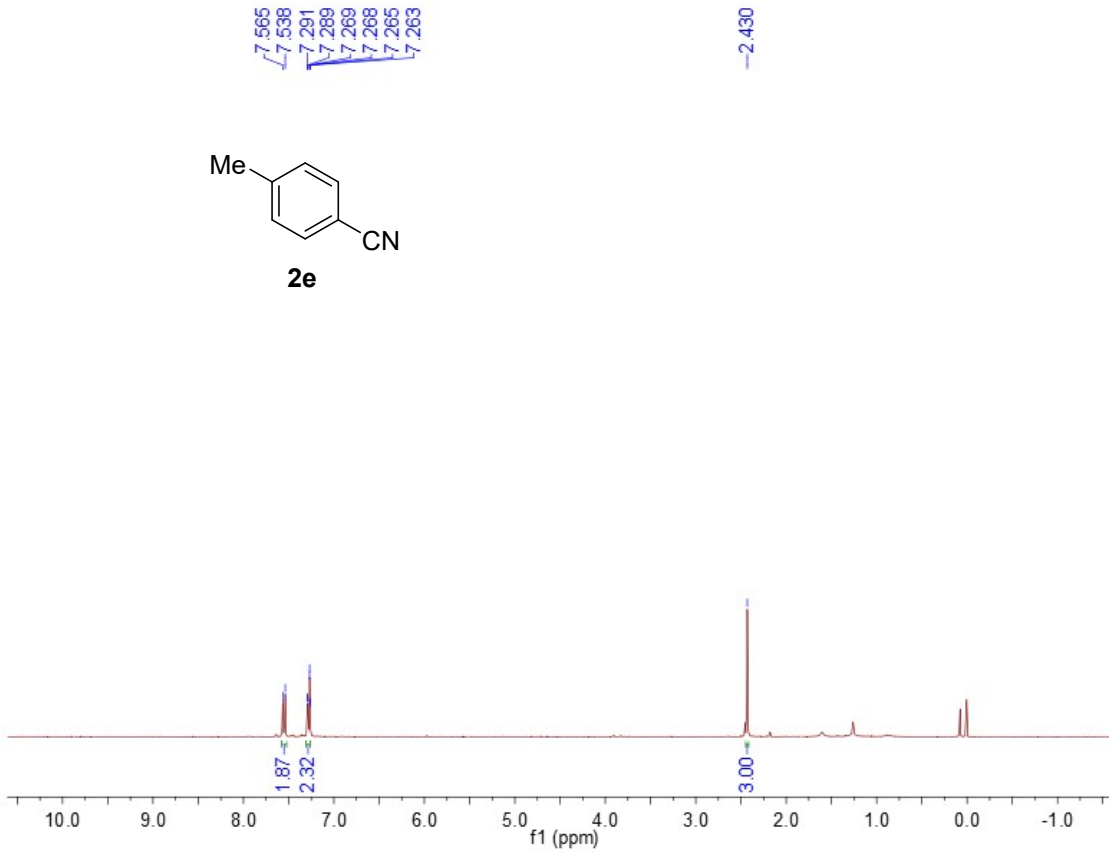
^{1}H NMR (300 MHz, $CDCl_3$):



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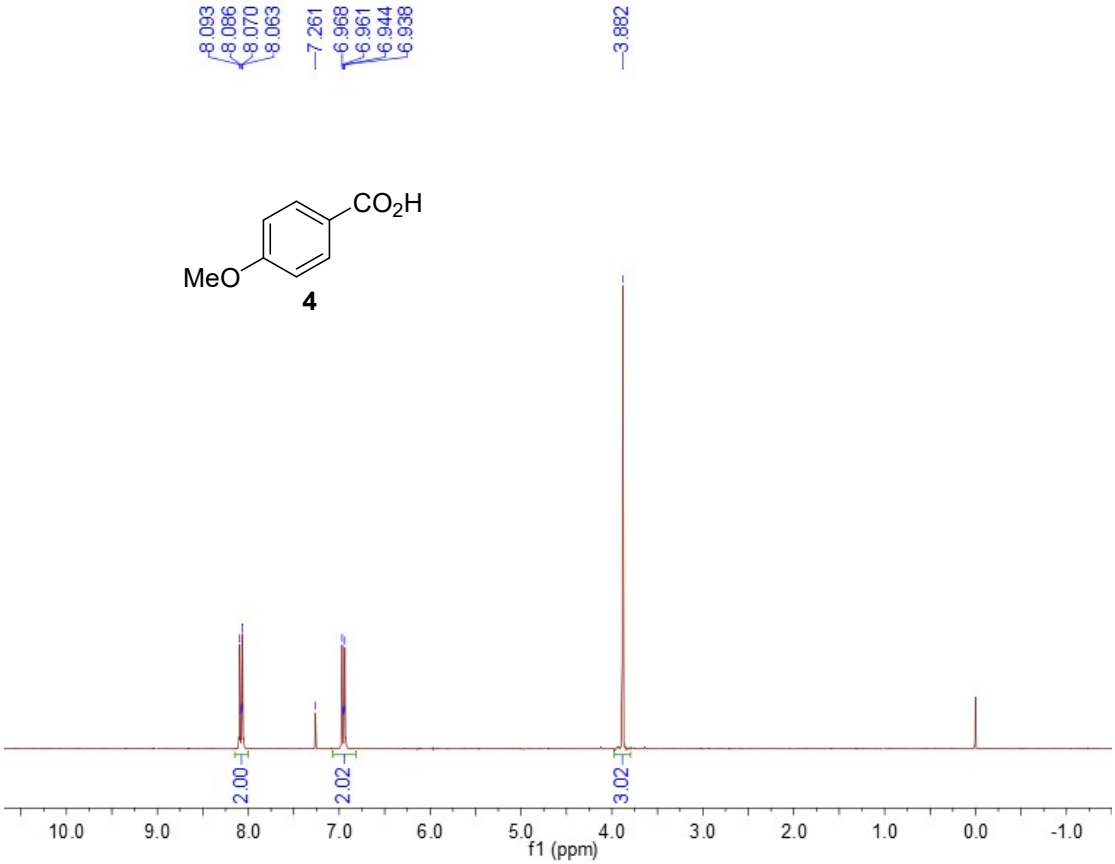

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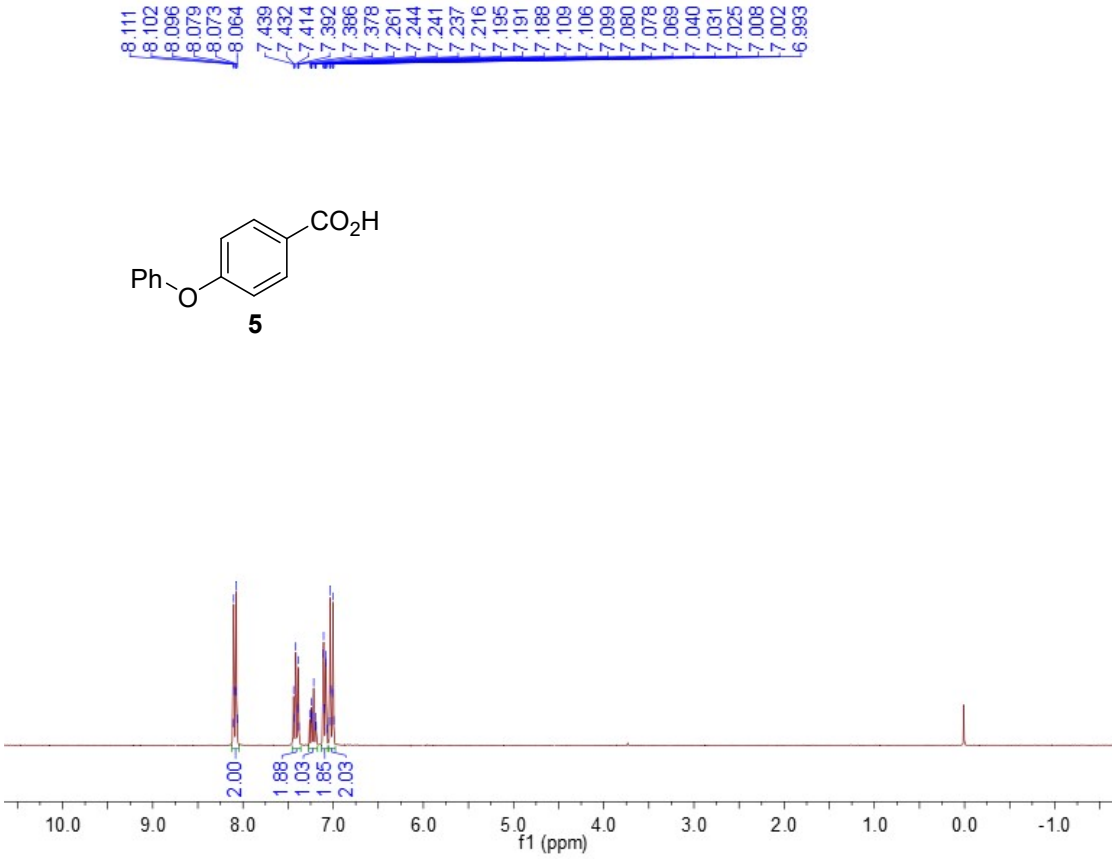
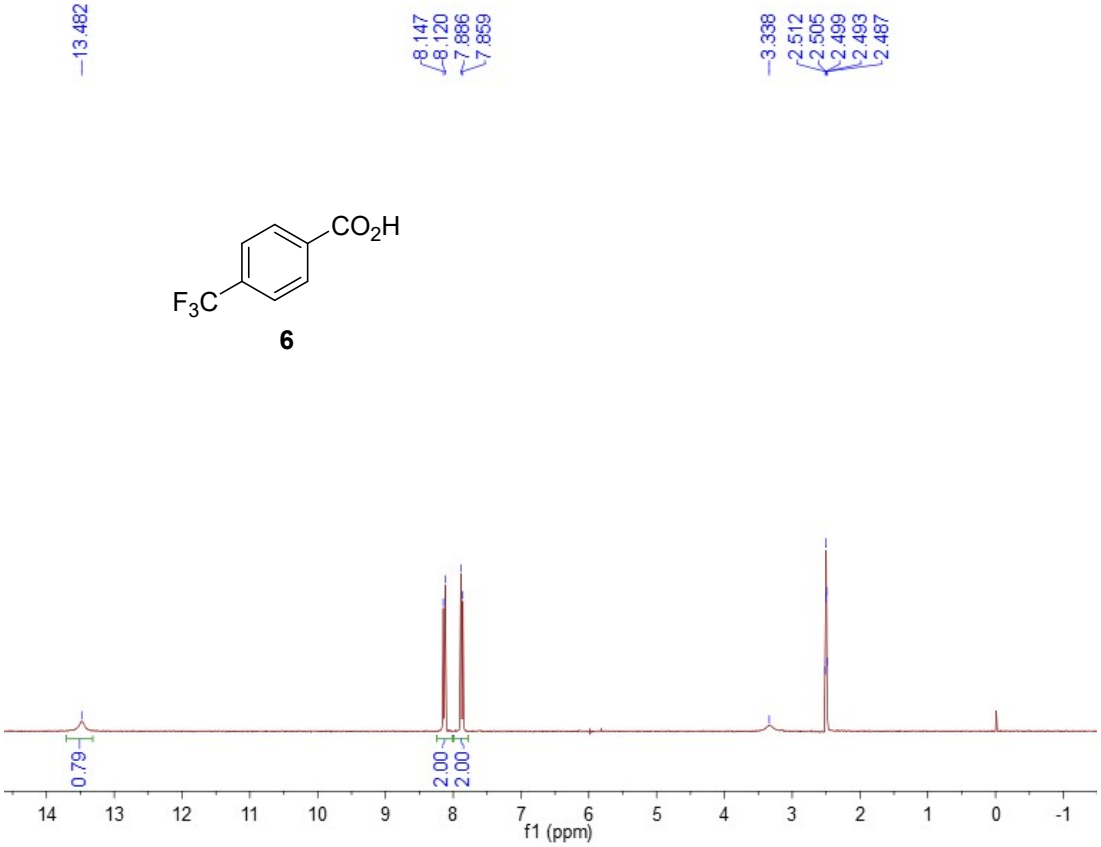


^{1}H NMR (300 MHz, DMSO-d_6):



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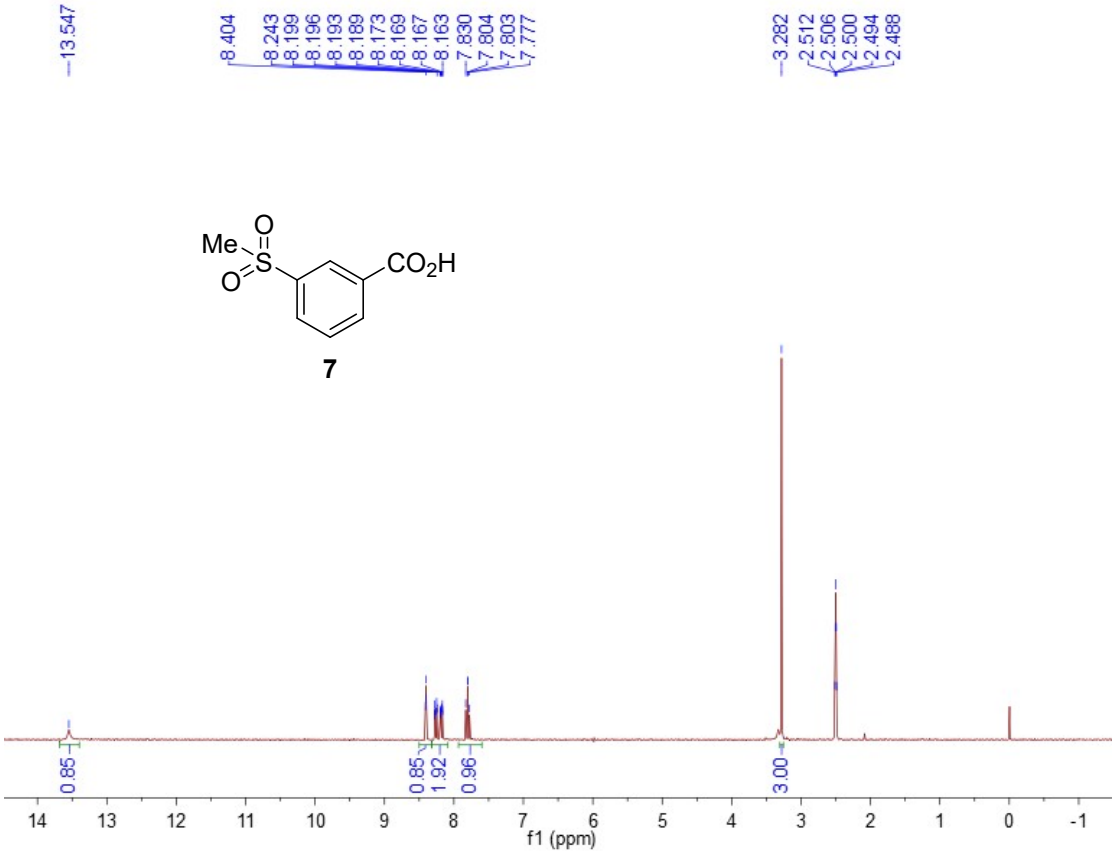

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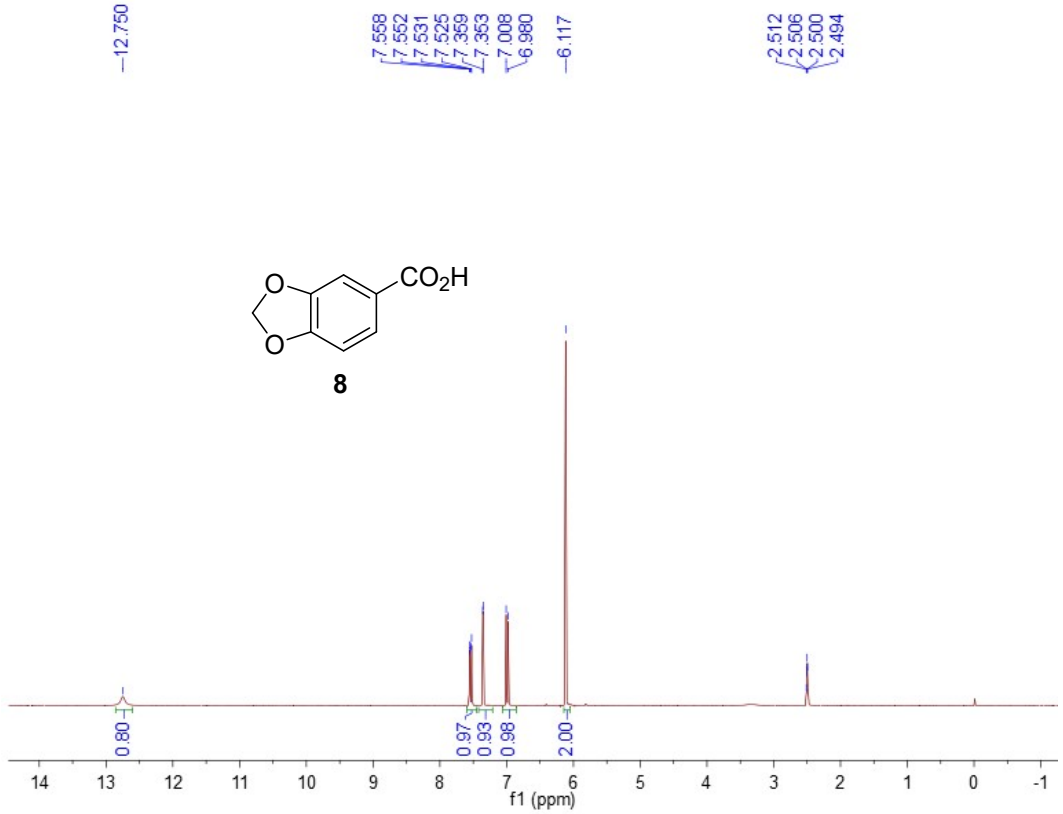

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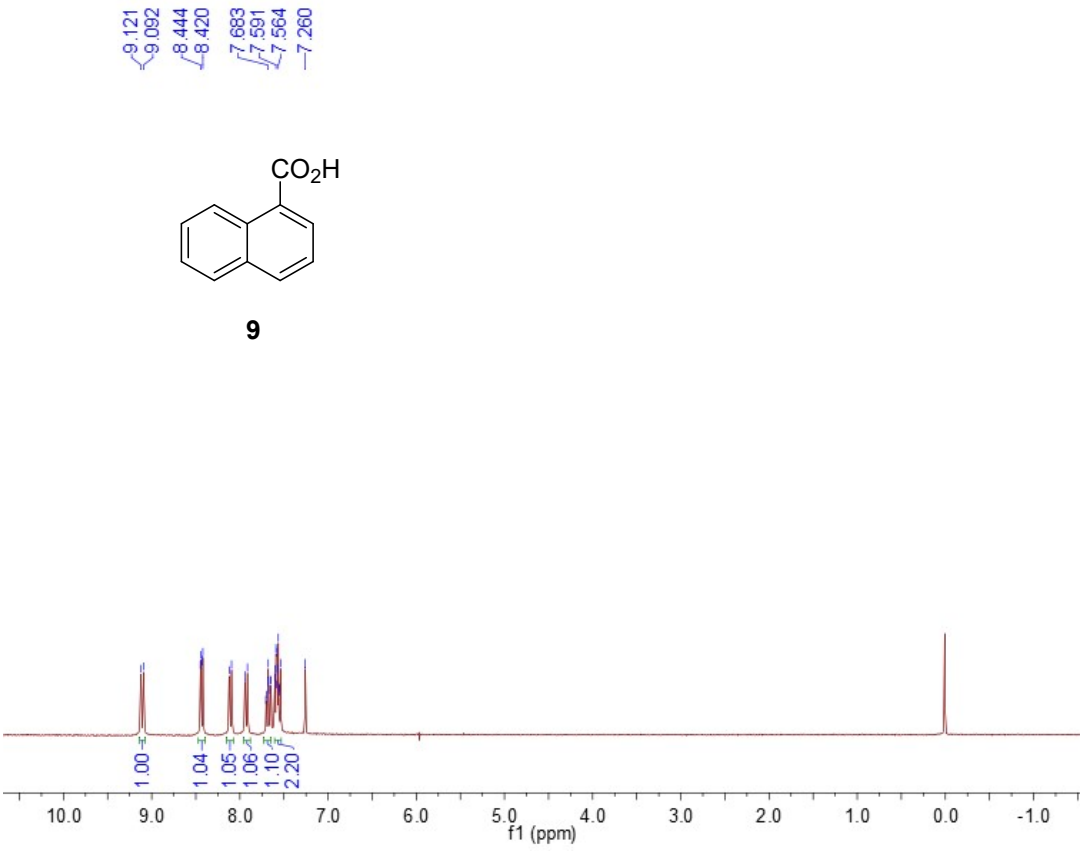

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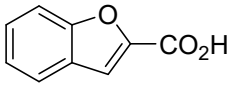

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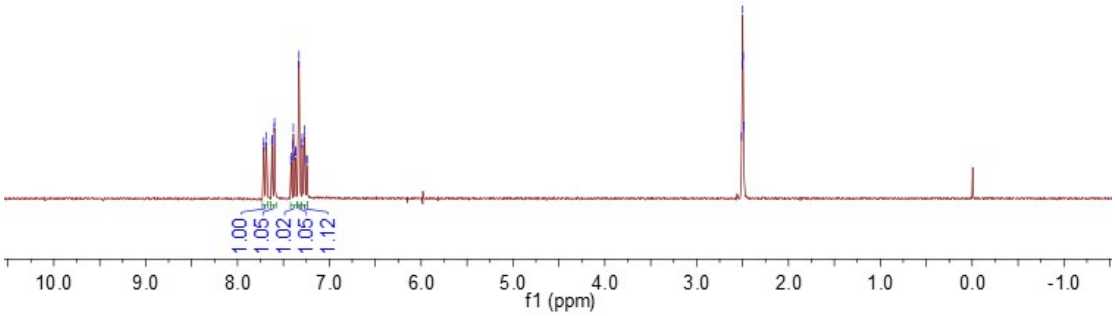

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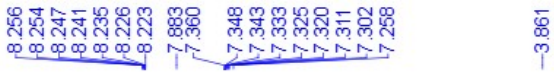

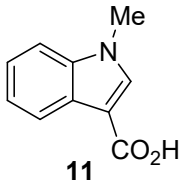

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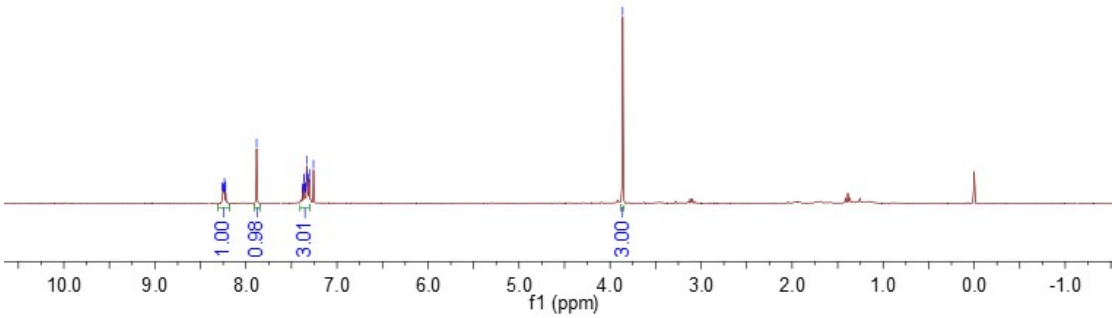

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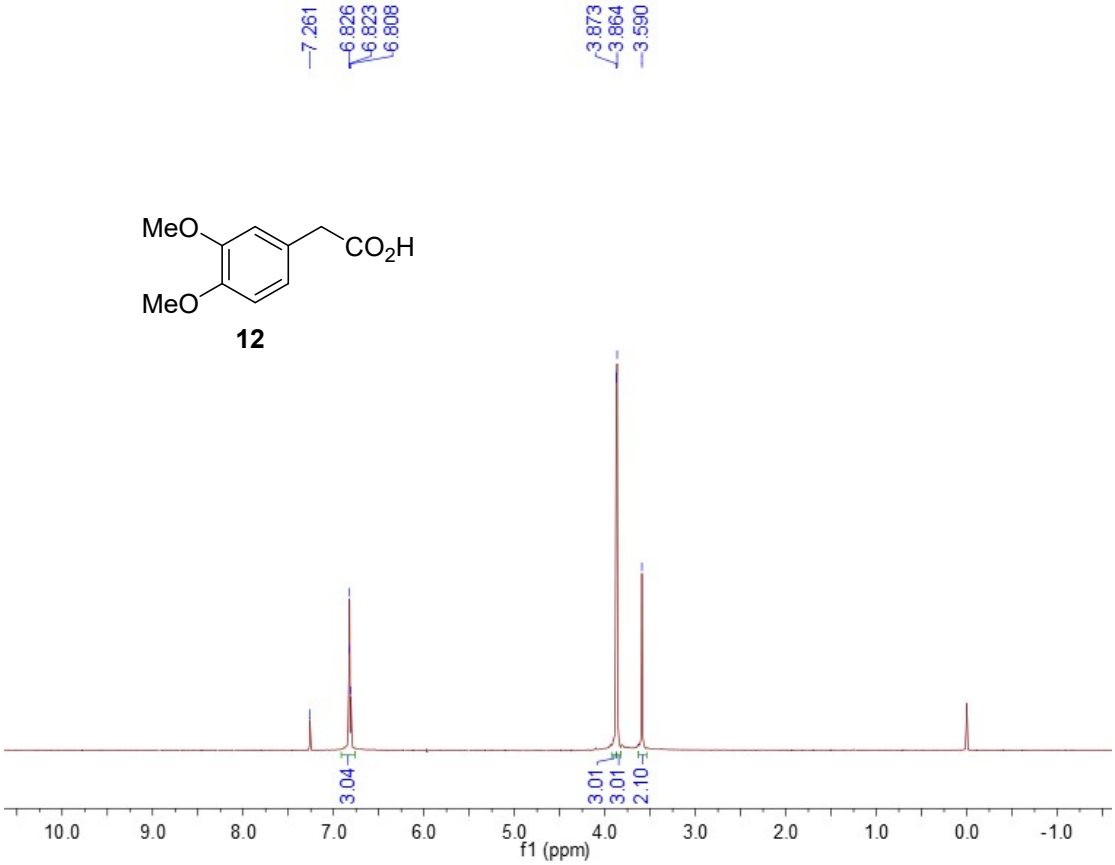

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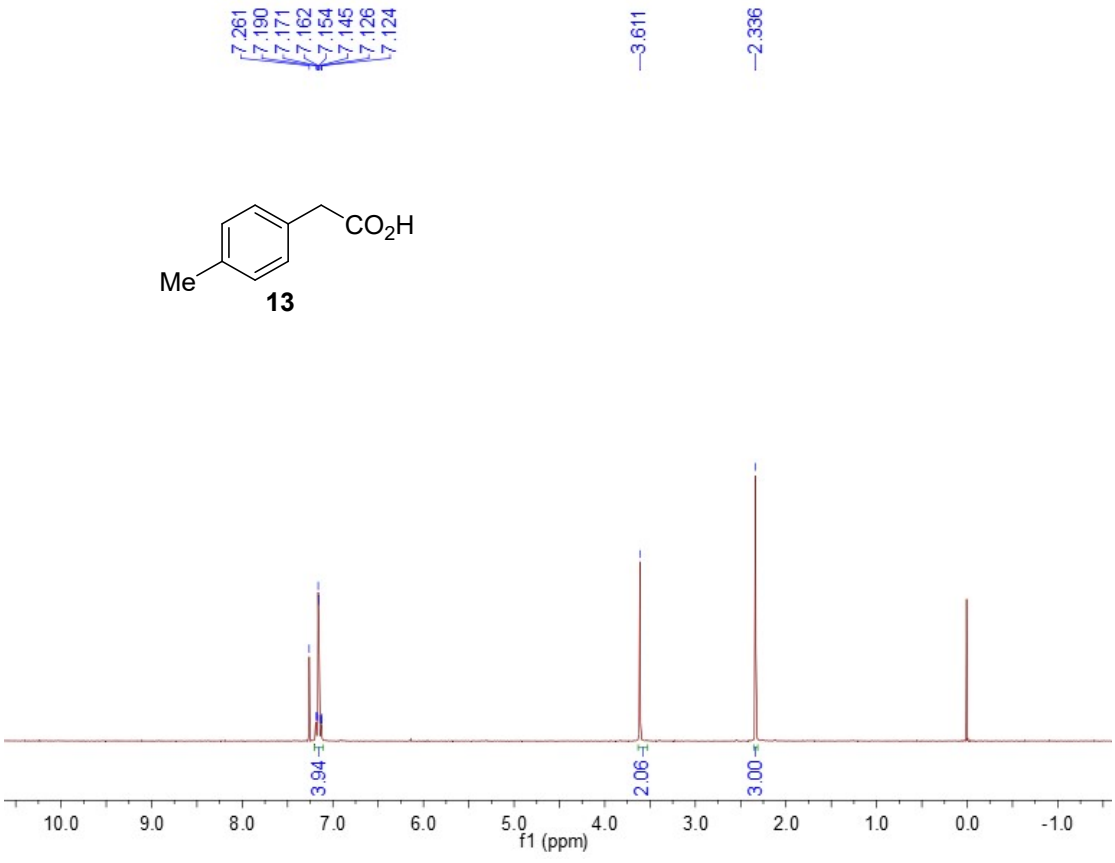

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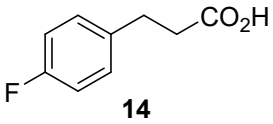

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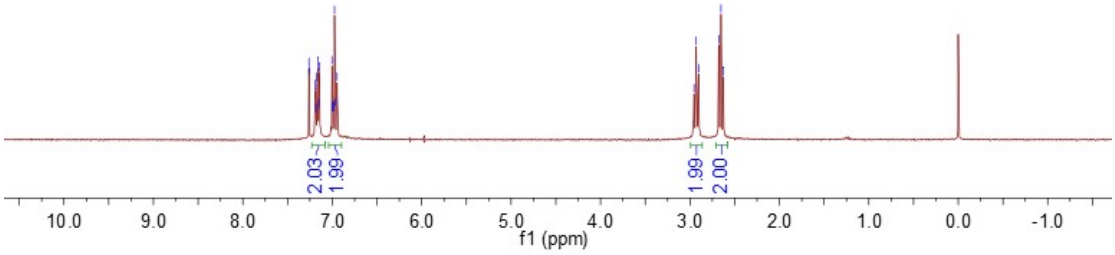

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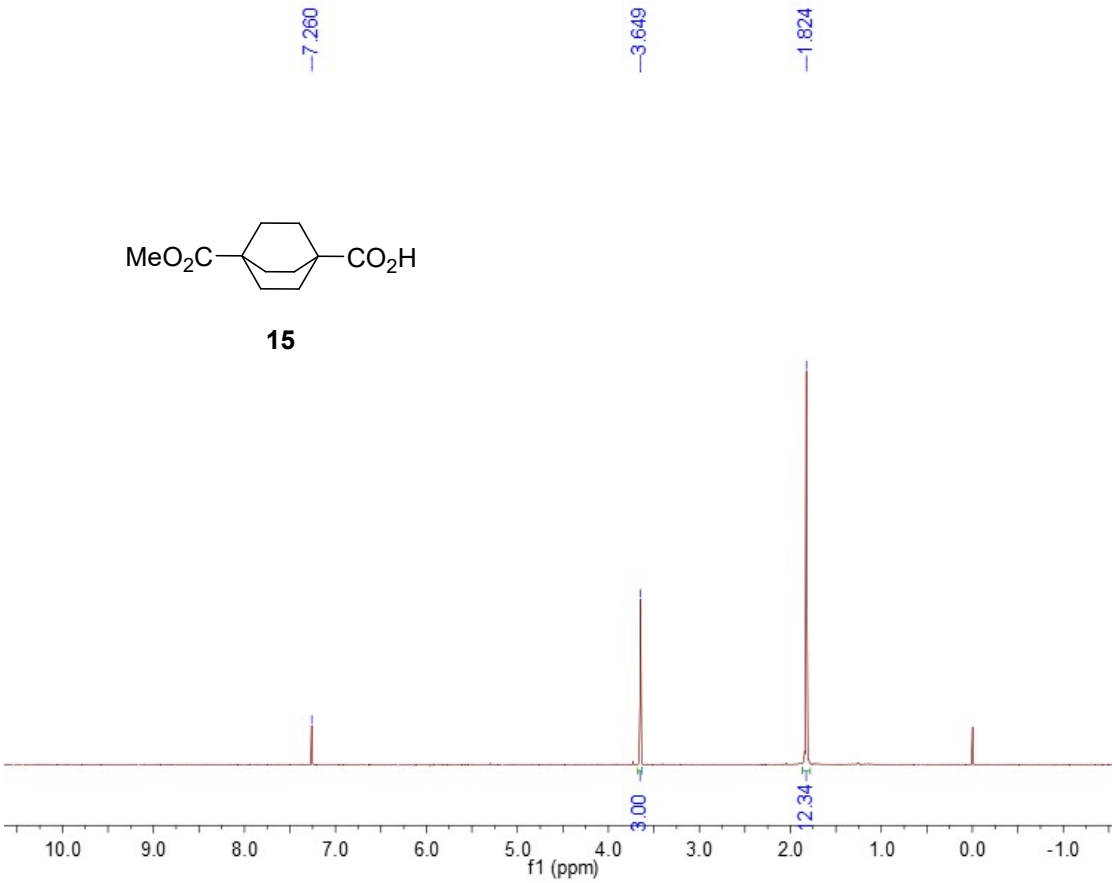

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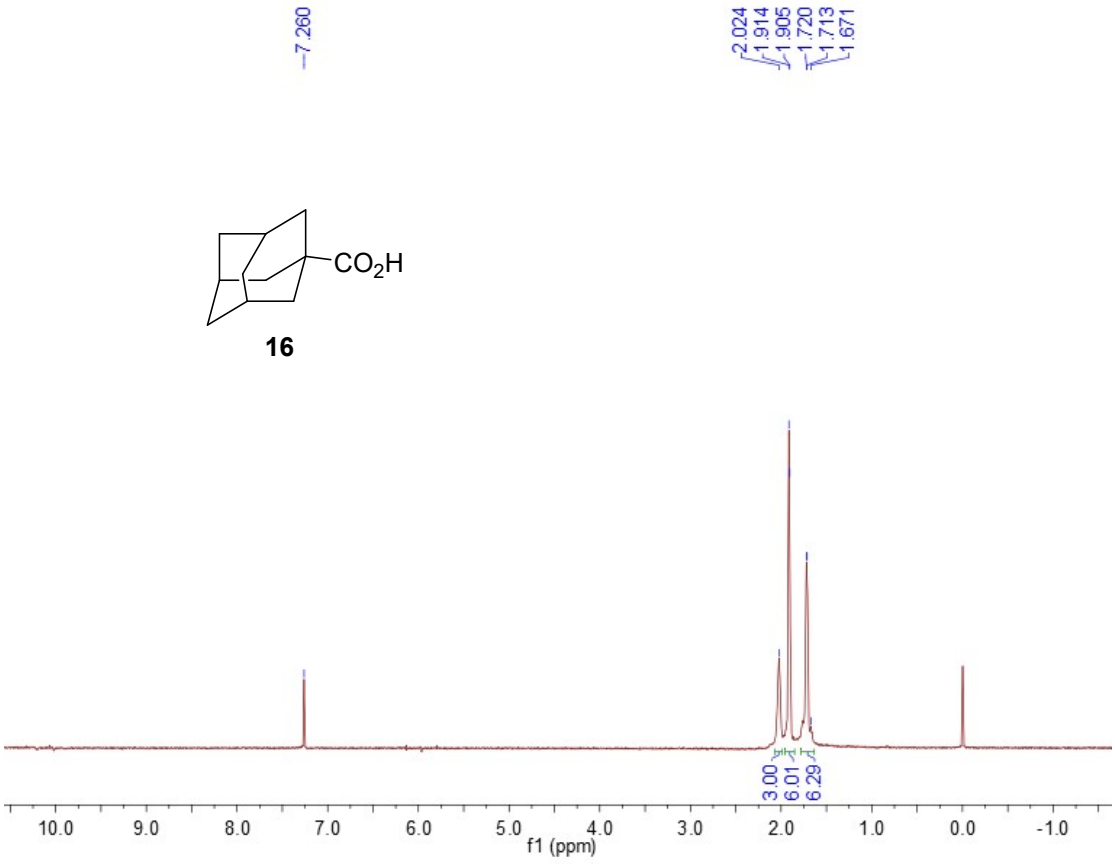

16

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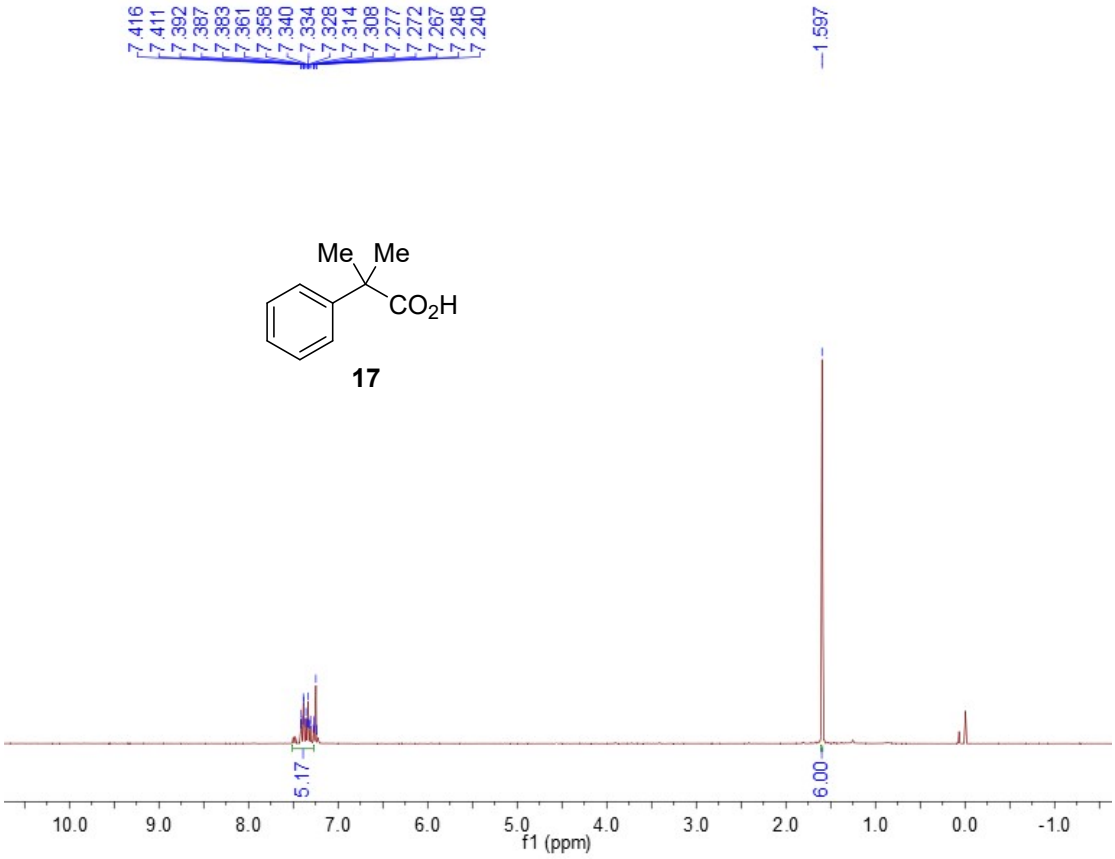

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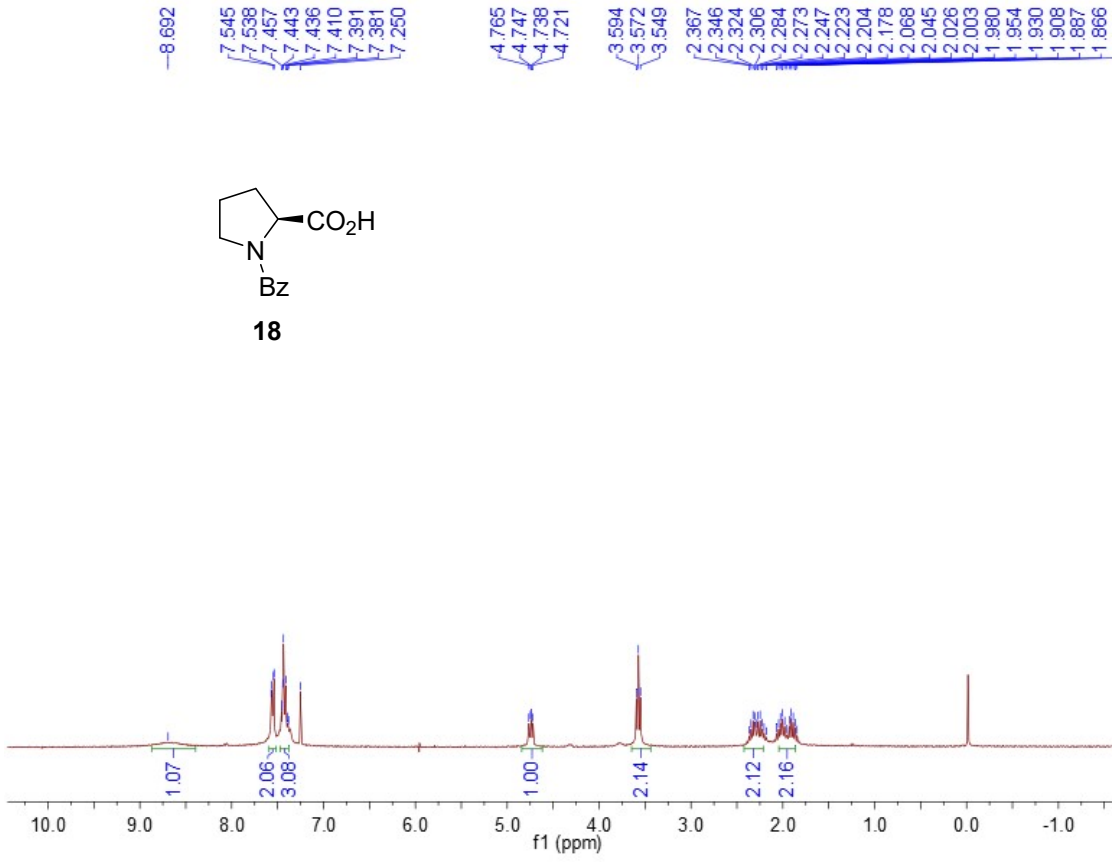

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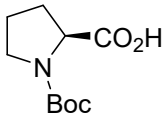

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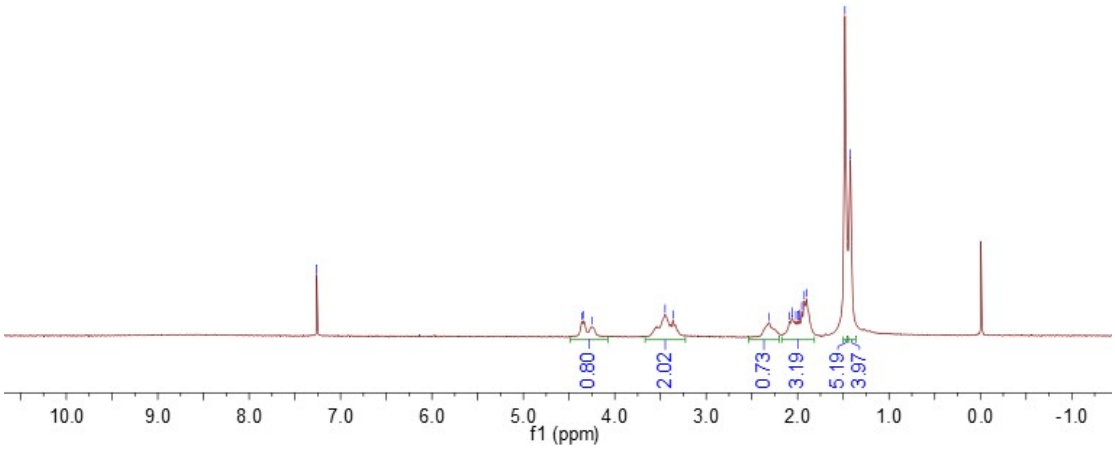

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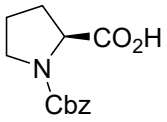

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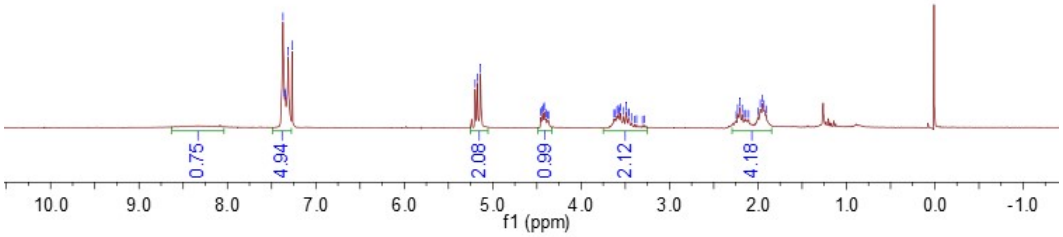
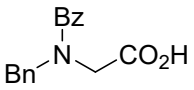


^{1}H NMR (300 MHz, $CDCl_3$):



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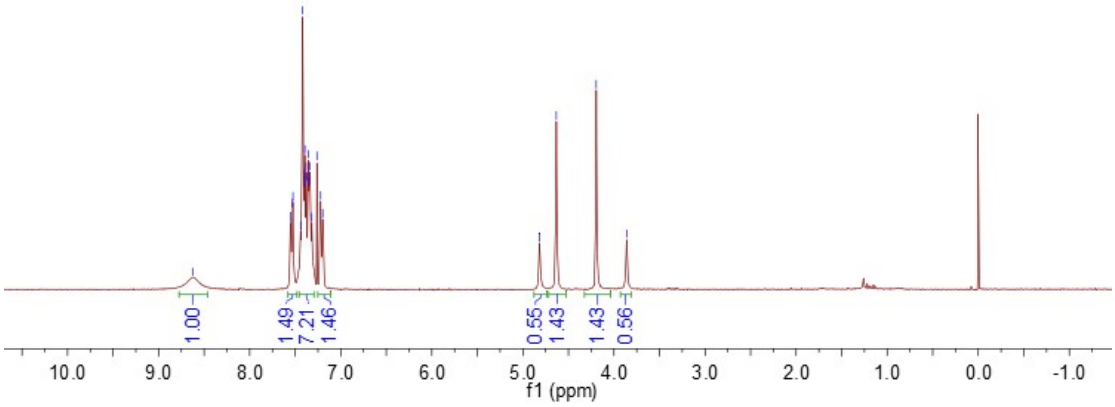
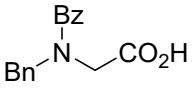


^{13}C NMR (75 MHz, $CDCl_3$):



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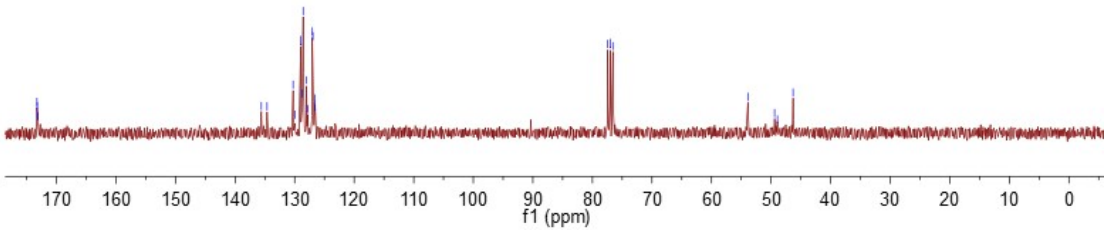
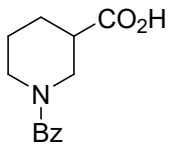


^{1}H NMR (300 MHz, DMSO-d_6):



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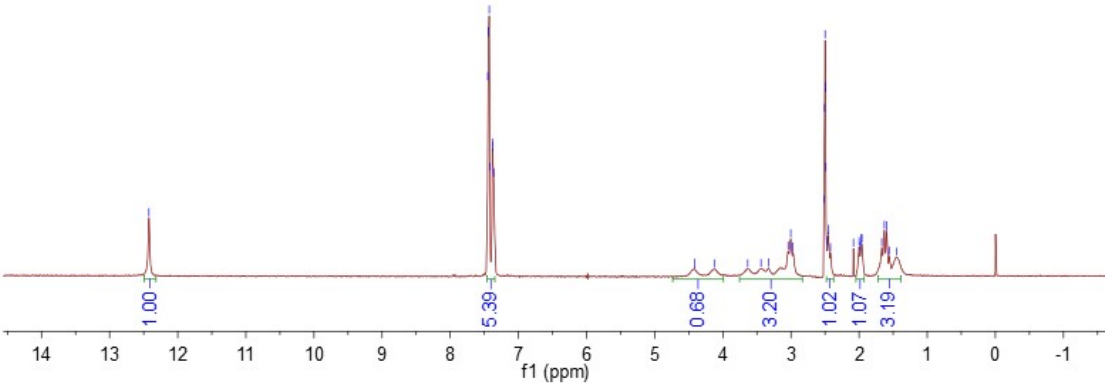
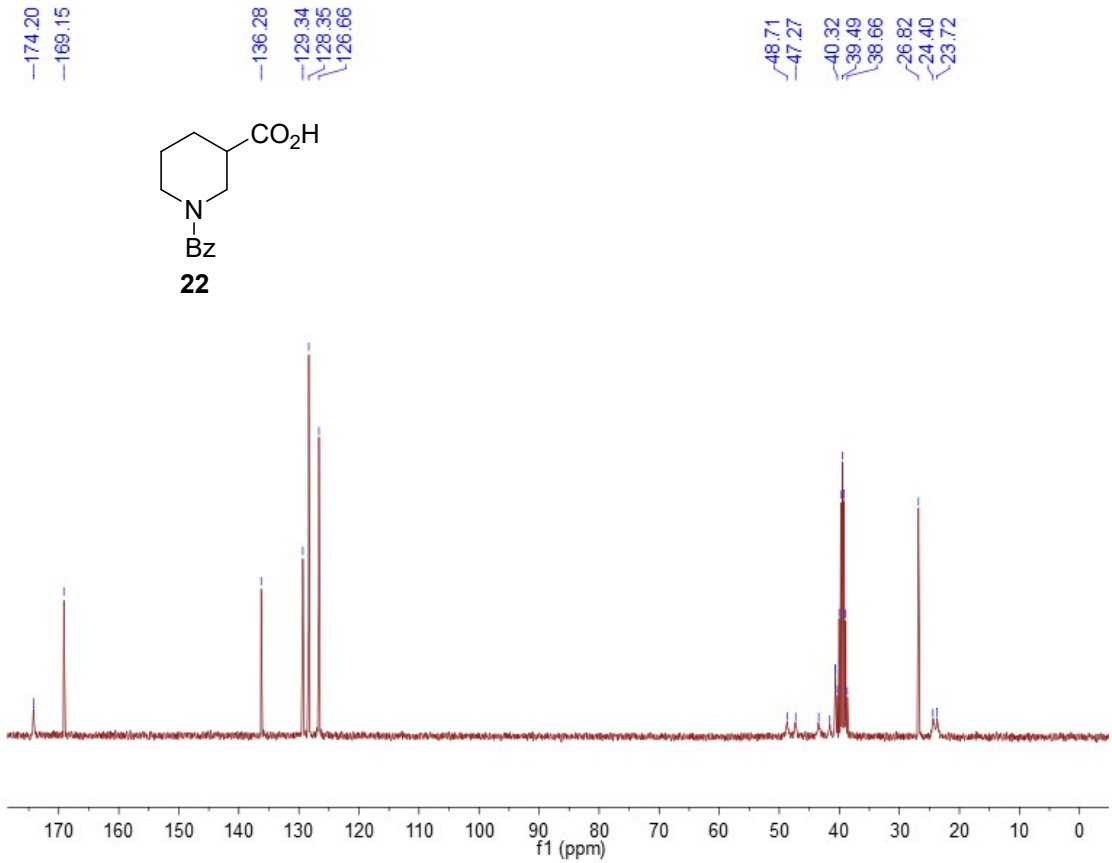


^{13}C NMR (75 MHz, DMSO-d_6):



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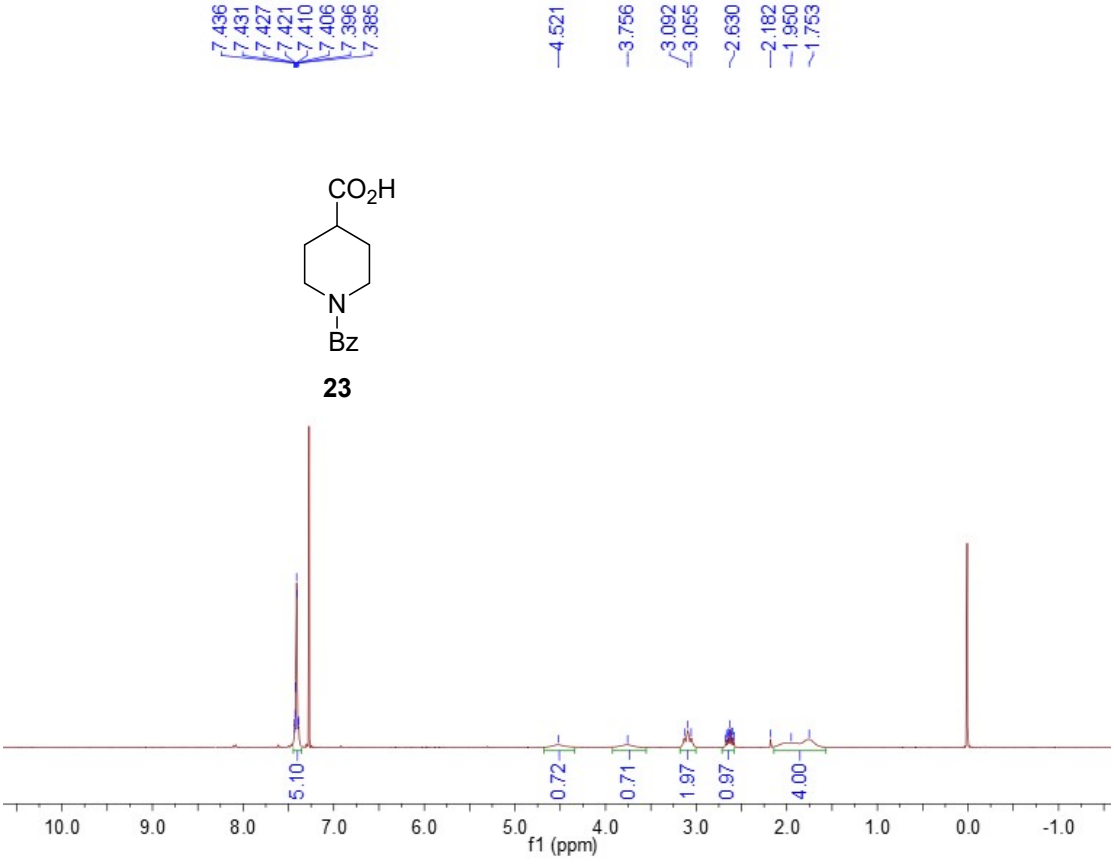
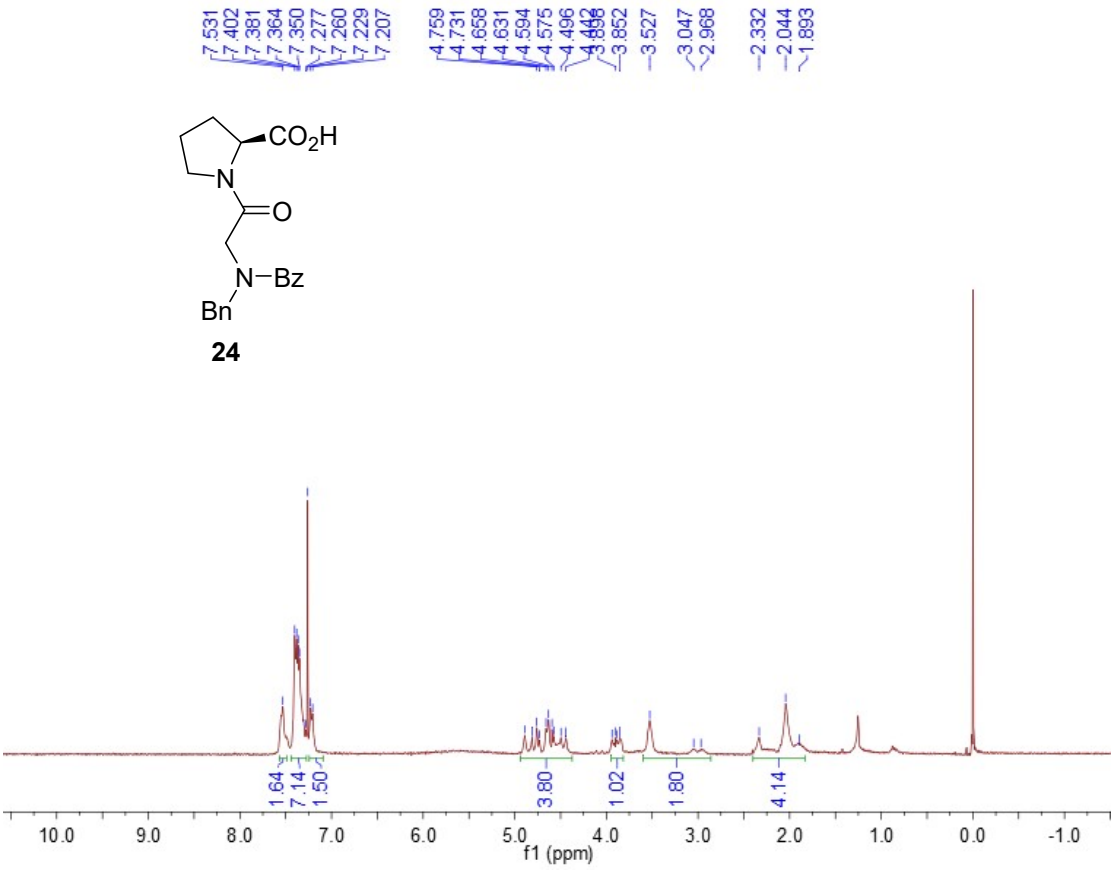
^{1}H NMR (300 MHz, $CDCl_3$):



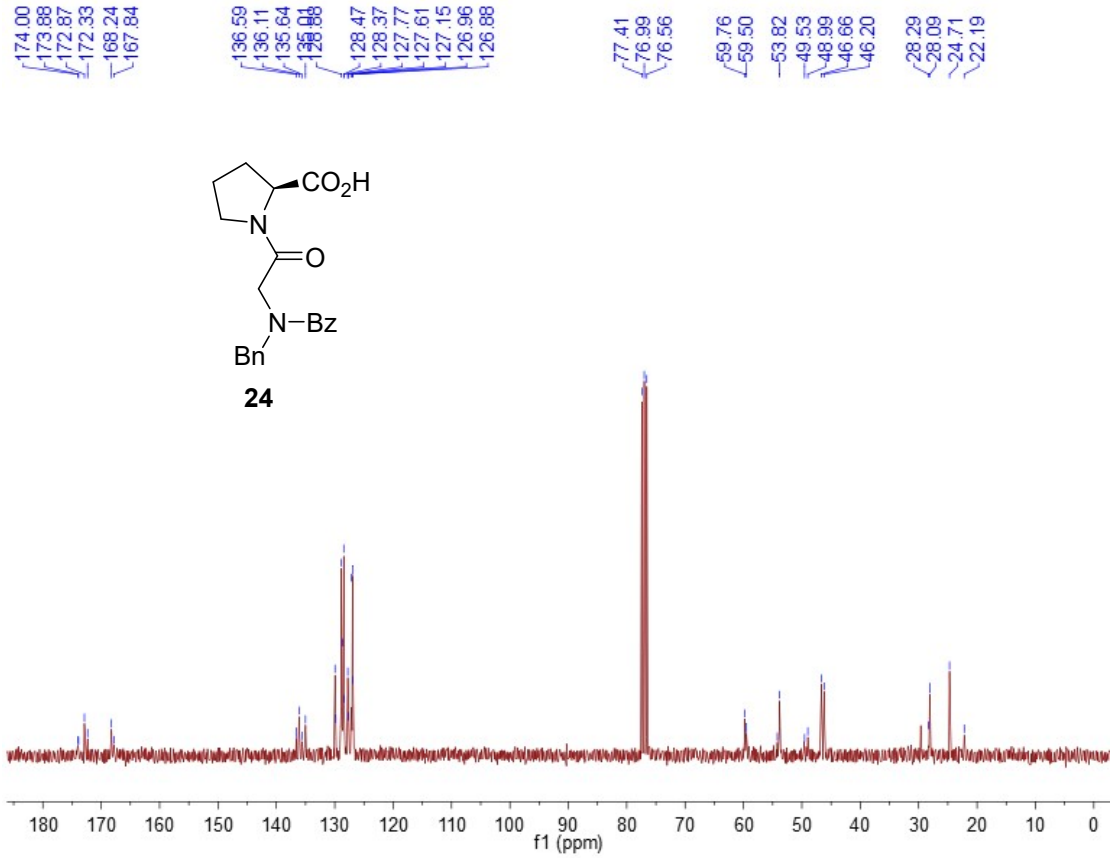
23

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



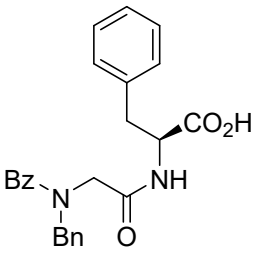
24

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



24

^{1}H NMR (300 MHz, DMSO-d_6):



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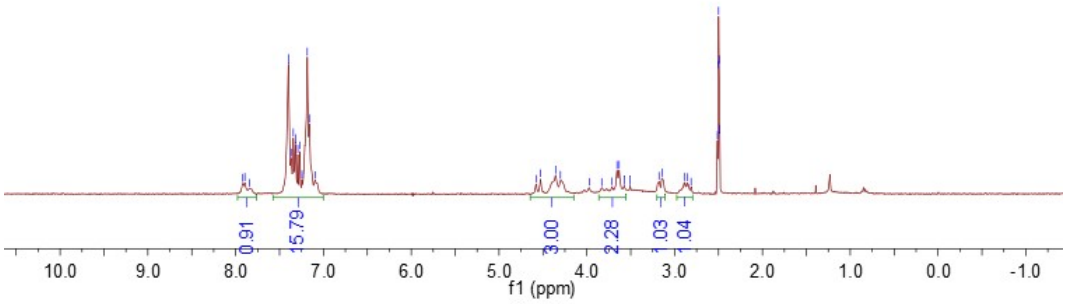
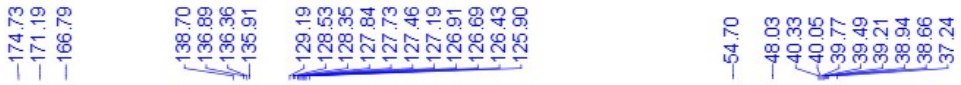
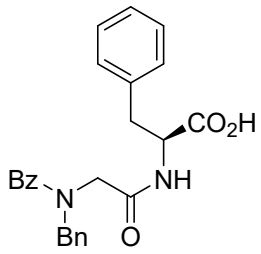


^{13}C NMR (75 MHz, DMSO-d_6):





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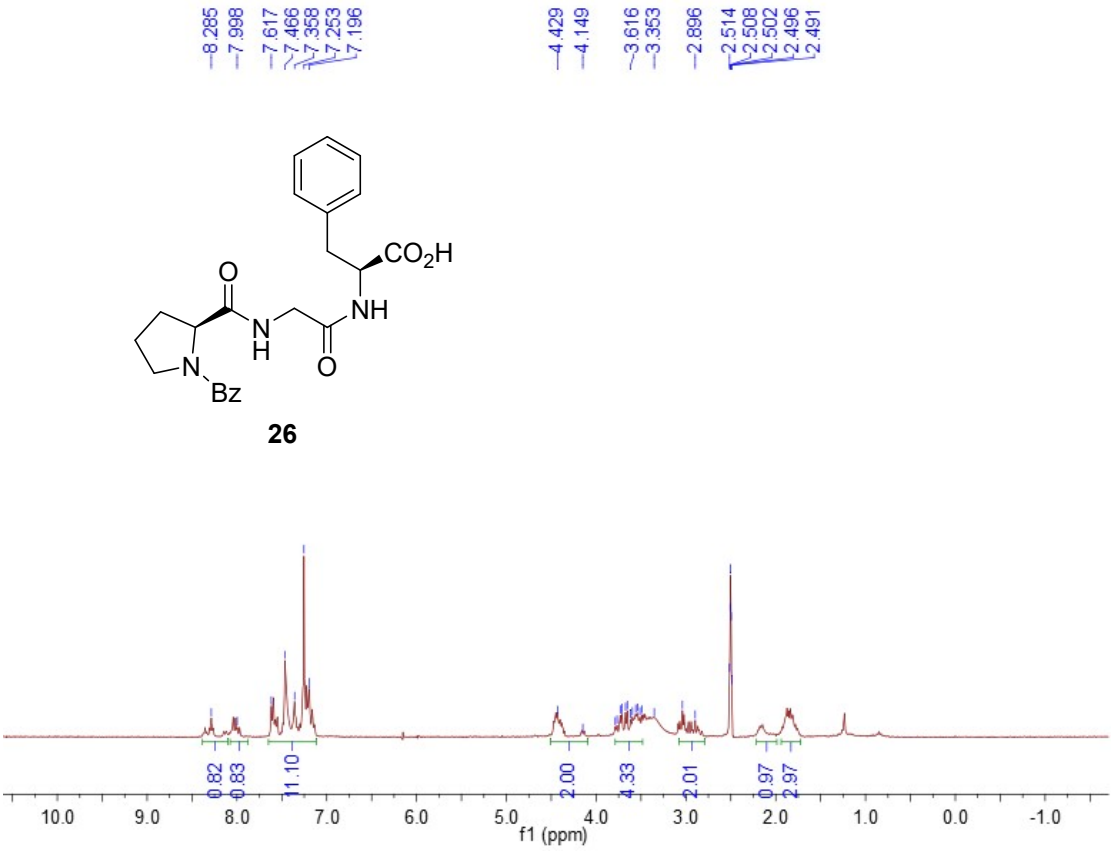
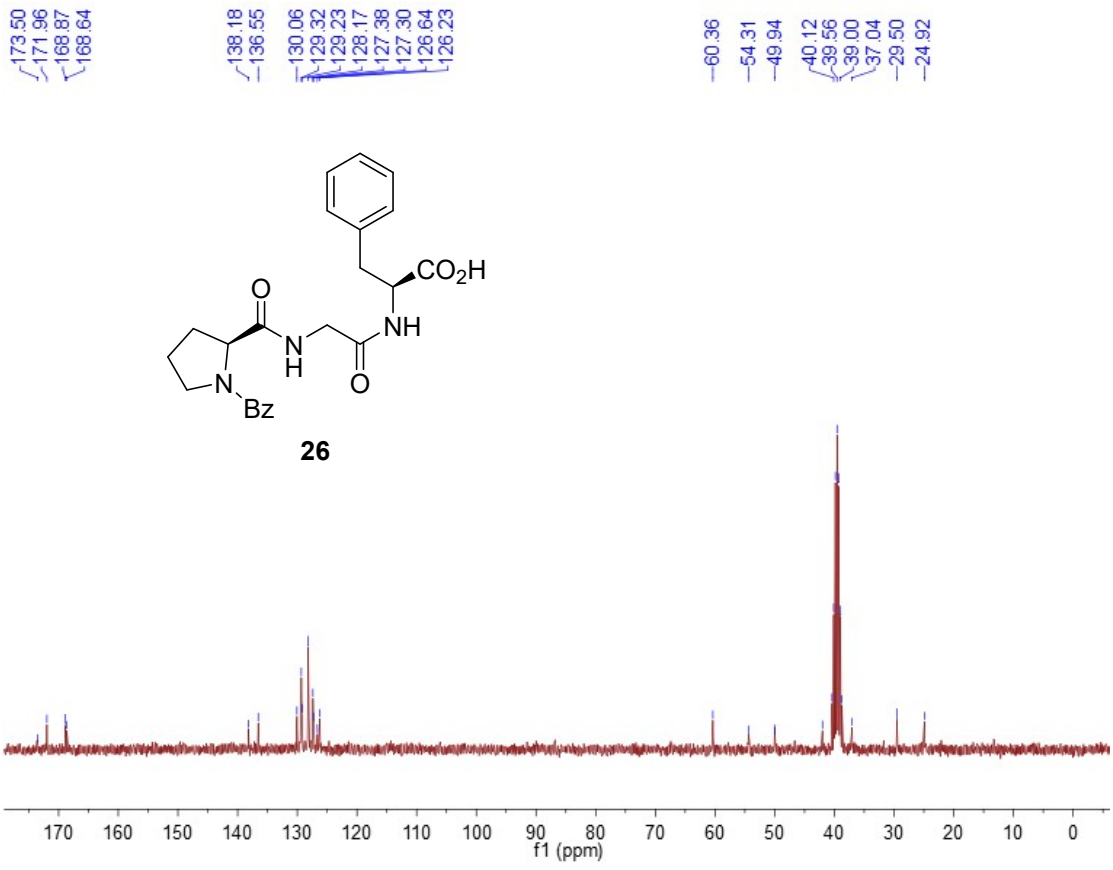
^{1}H NMR (300 MHz, DMSO-d_6):



26

^{13}C NMR (75 MHz, DMSO-d_6):



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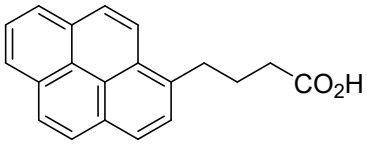
^{1}H NMR (300 MHz, $CDCl_3$):



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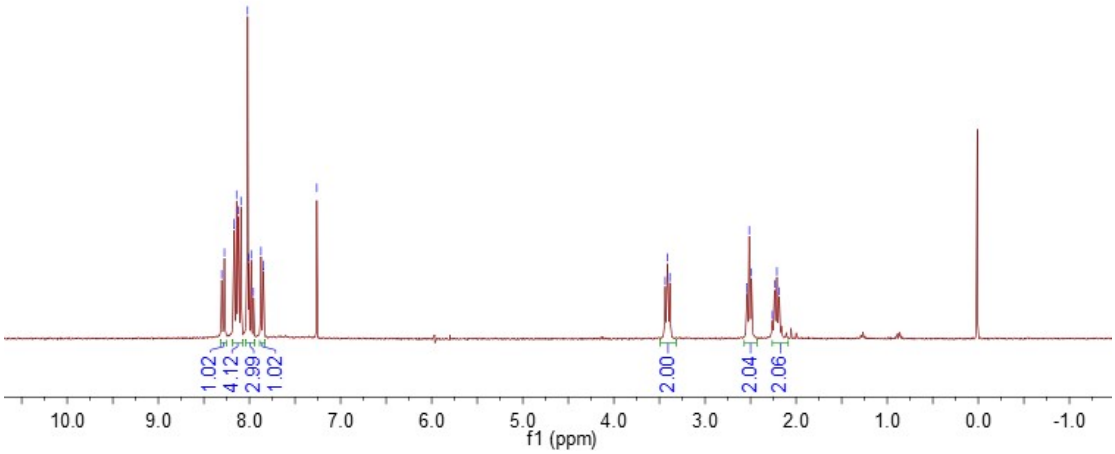
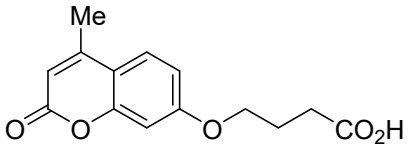


^{1}H NMR (300 MHz, DMSO-d_6):



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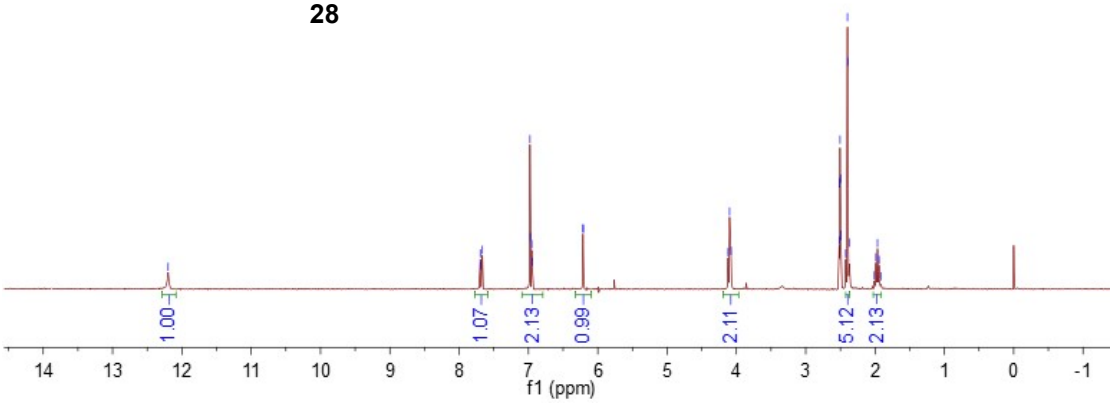

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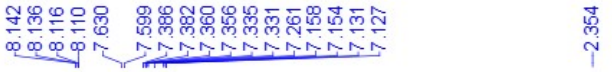

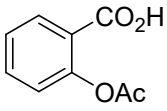

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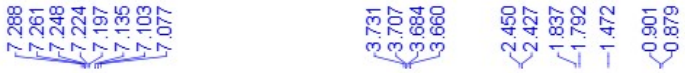
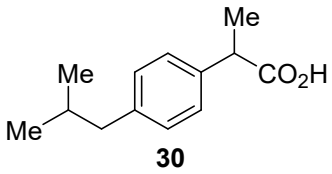
^{1}H NMR (300 MHz, $CDCl_3$):





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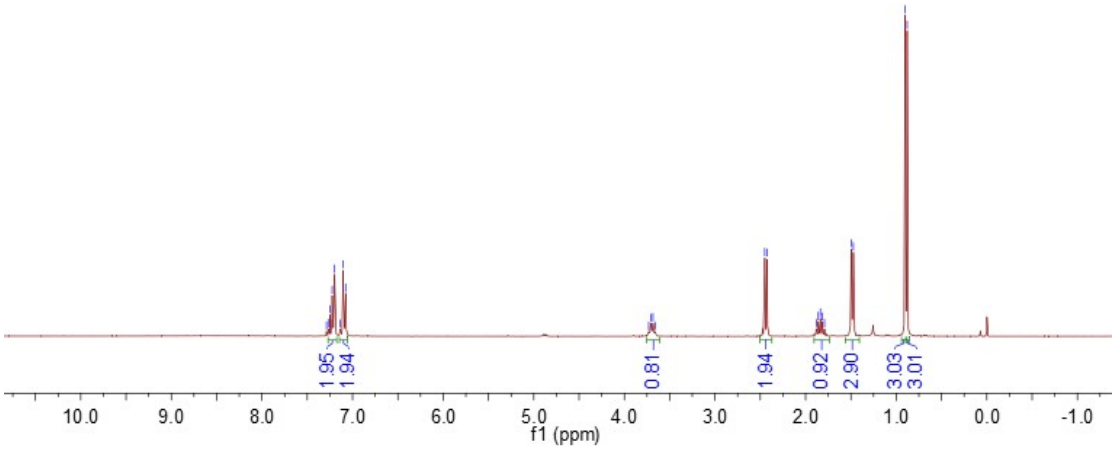
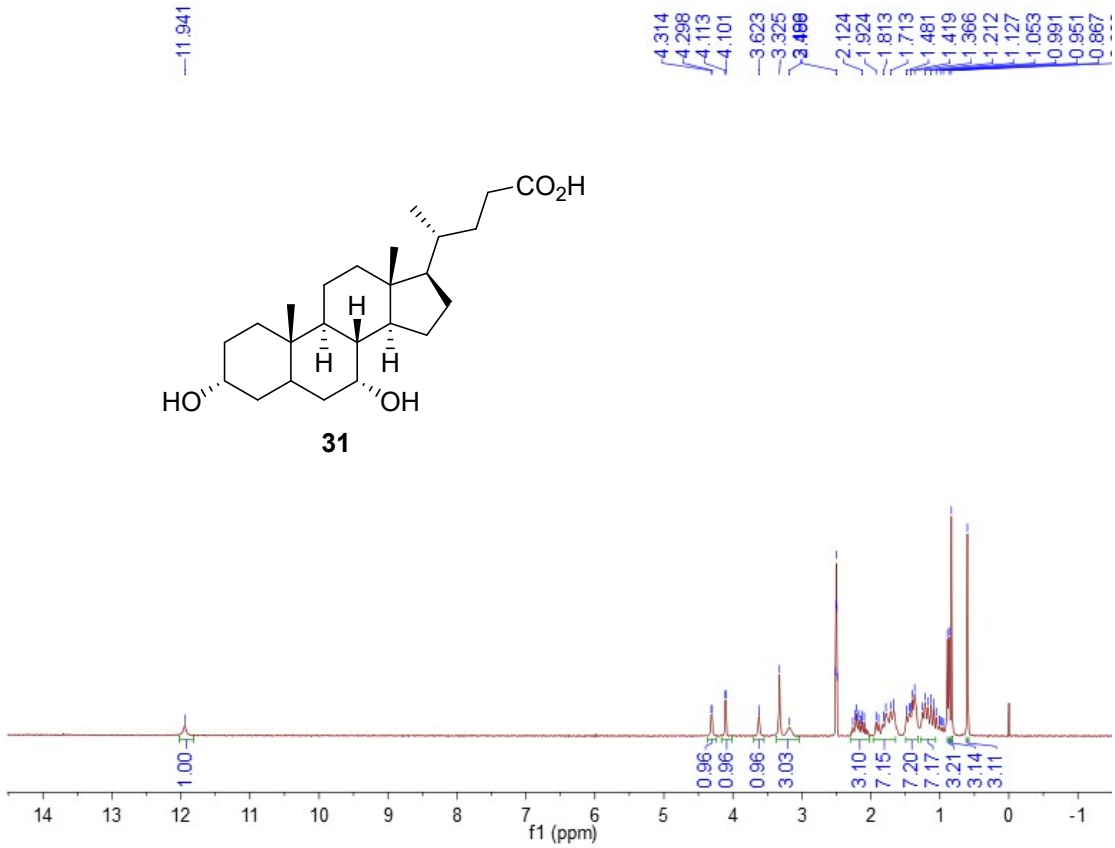
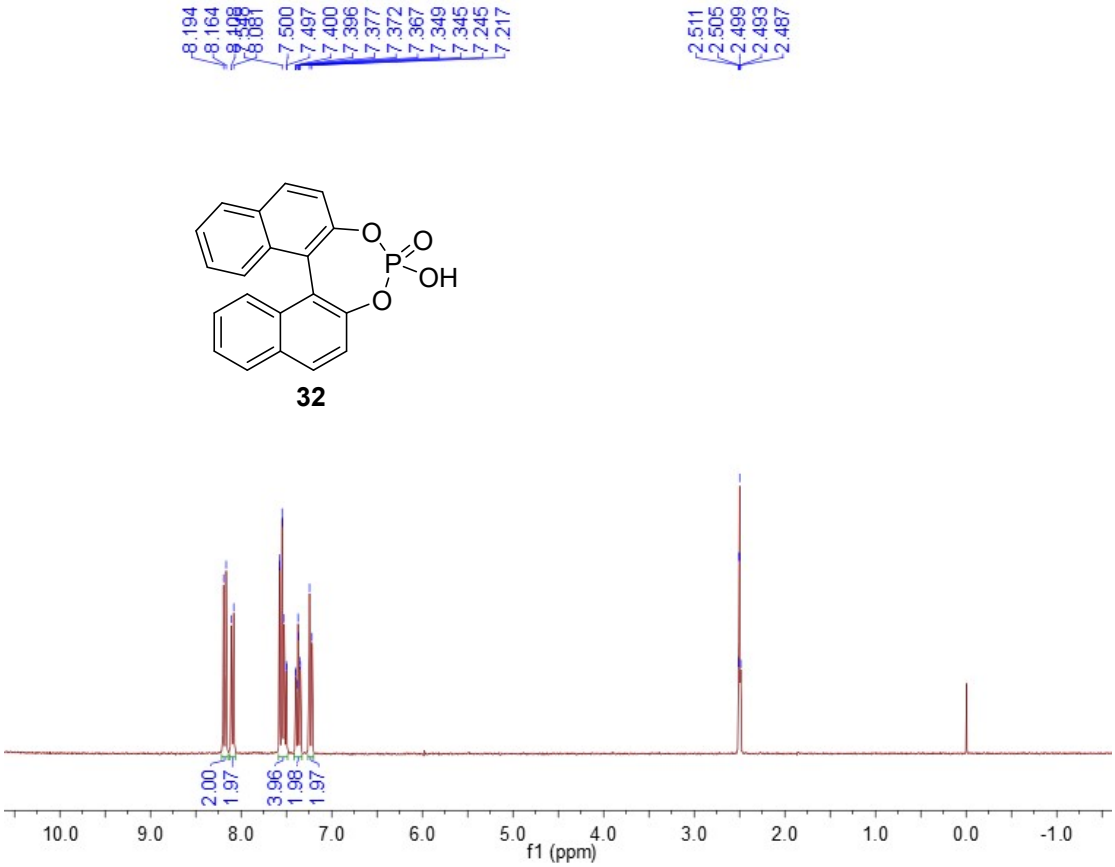


^{1}H NMR (300 MHz, DMSO-d_6):



31

^{1}H NMR (300 MHz, DMSO-d_6):



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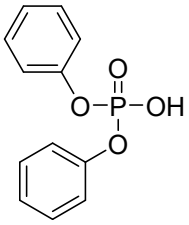
^{1}H NMR (300 MHz, $CDCl_3$):



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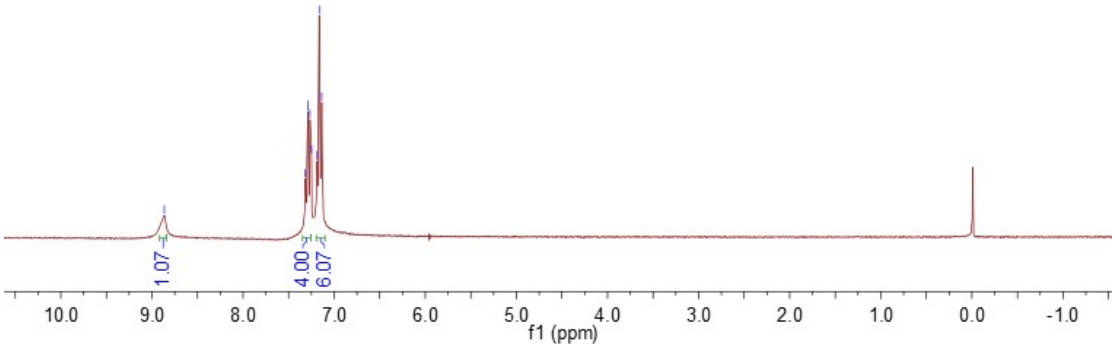
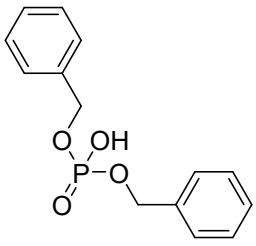


^{1}H NMR (300 MHz, DMSO-d_6):



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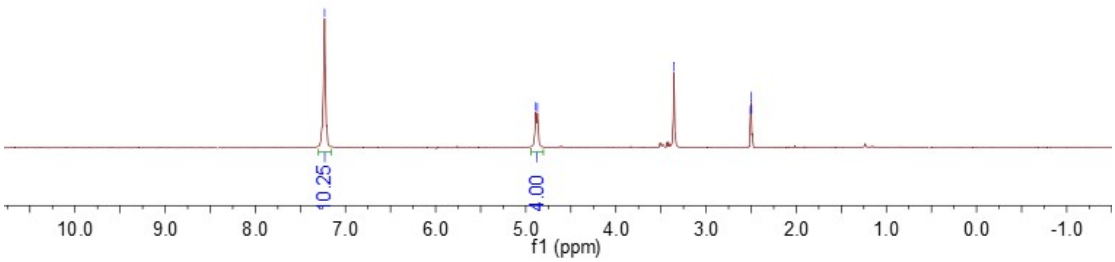
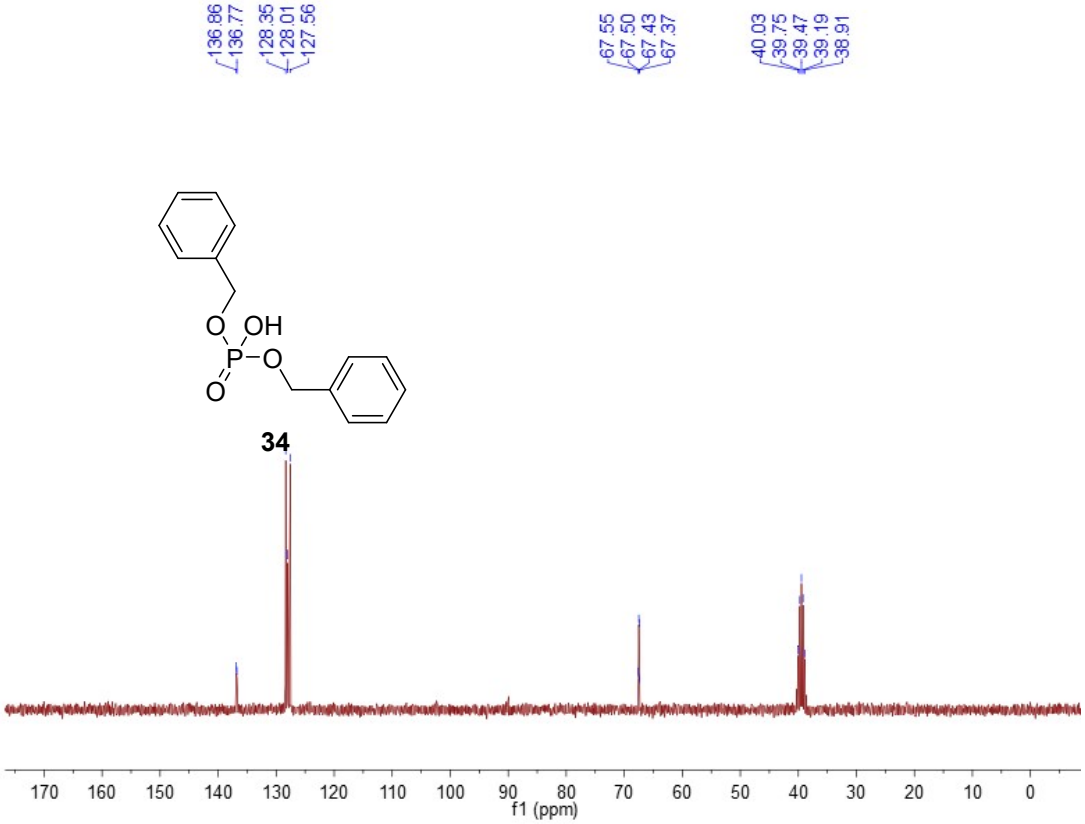
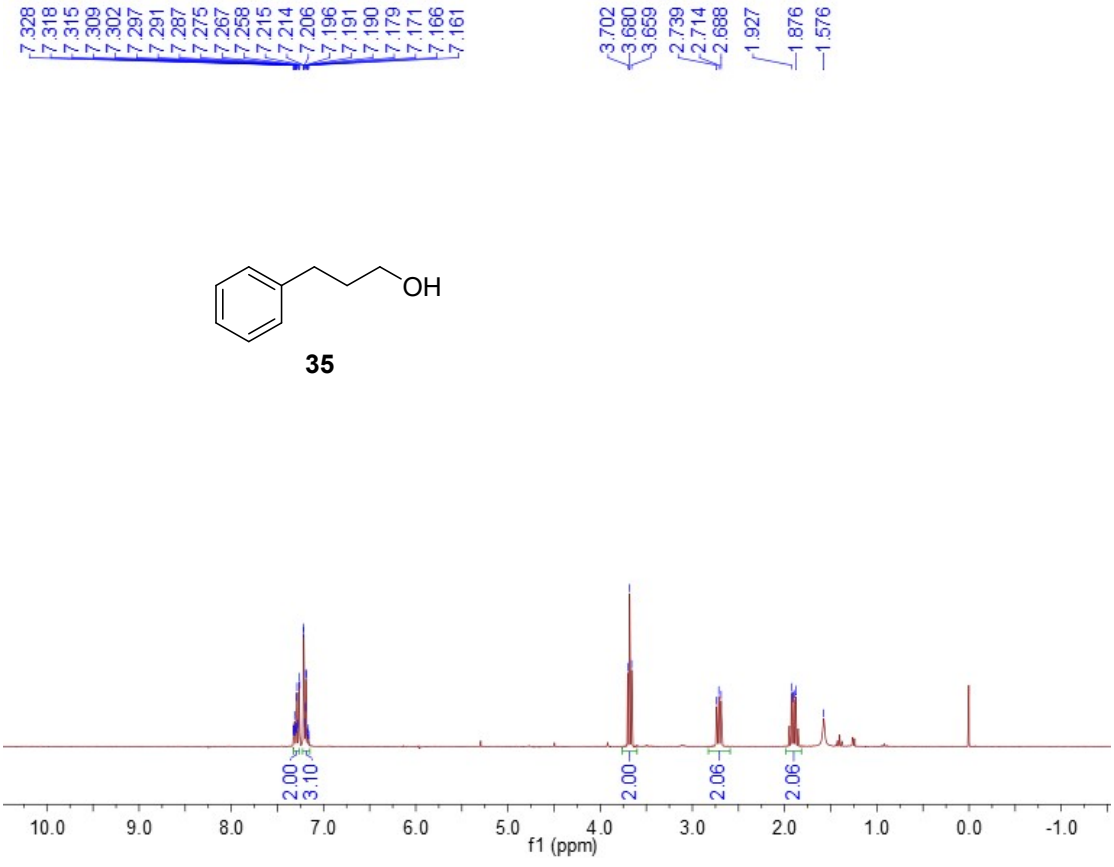


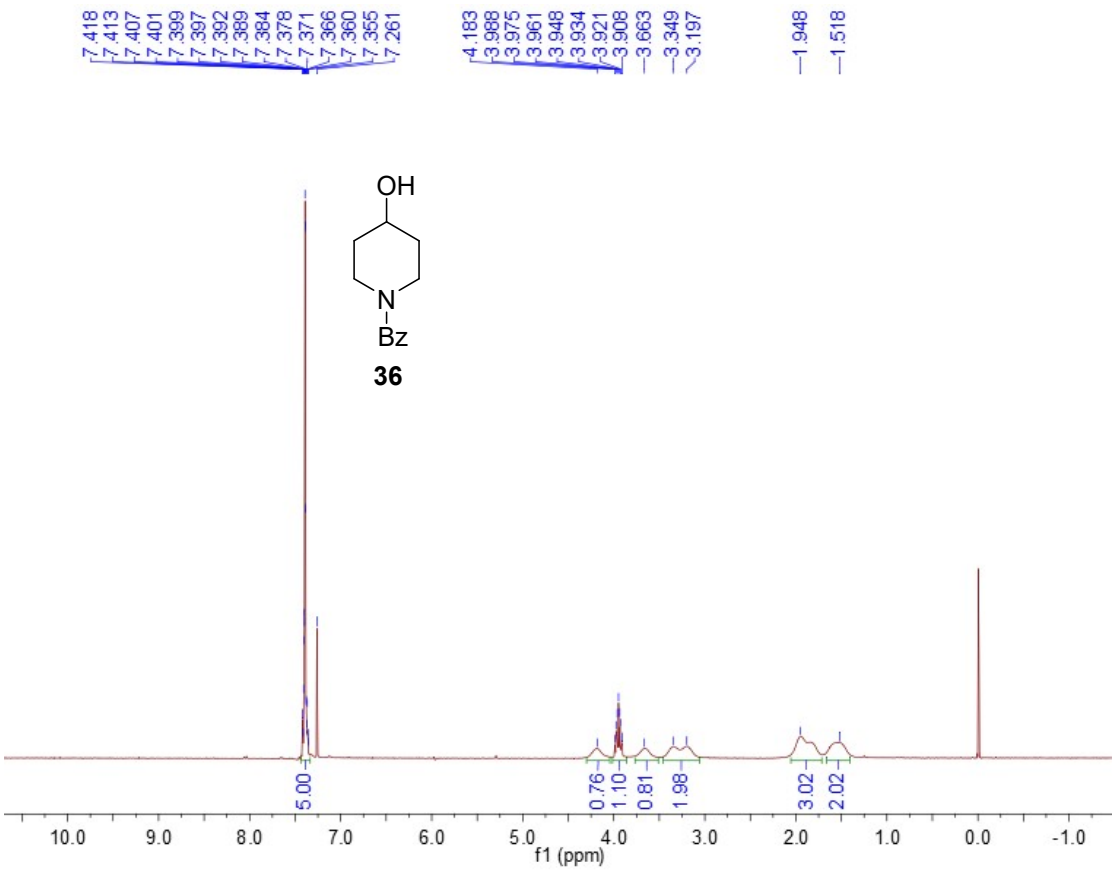
^{13}C NMR (75 MHz, DMSO-d_6):



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

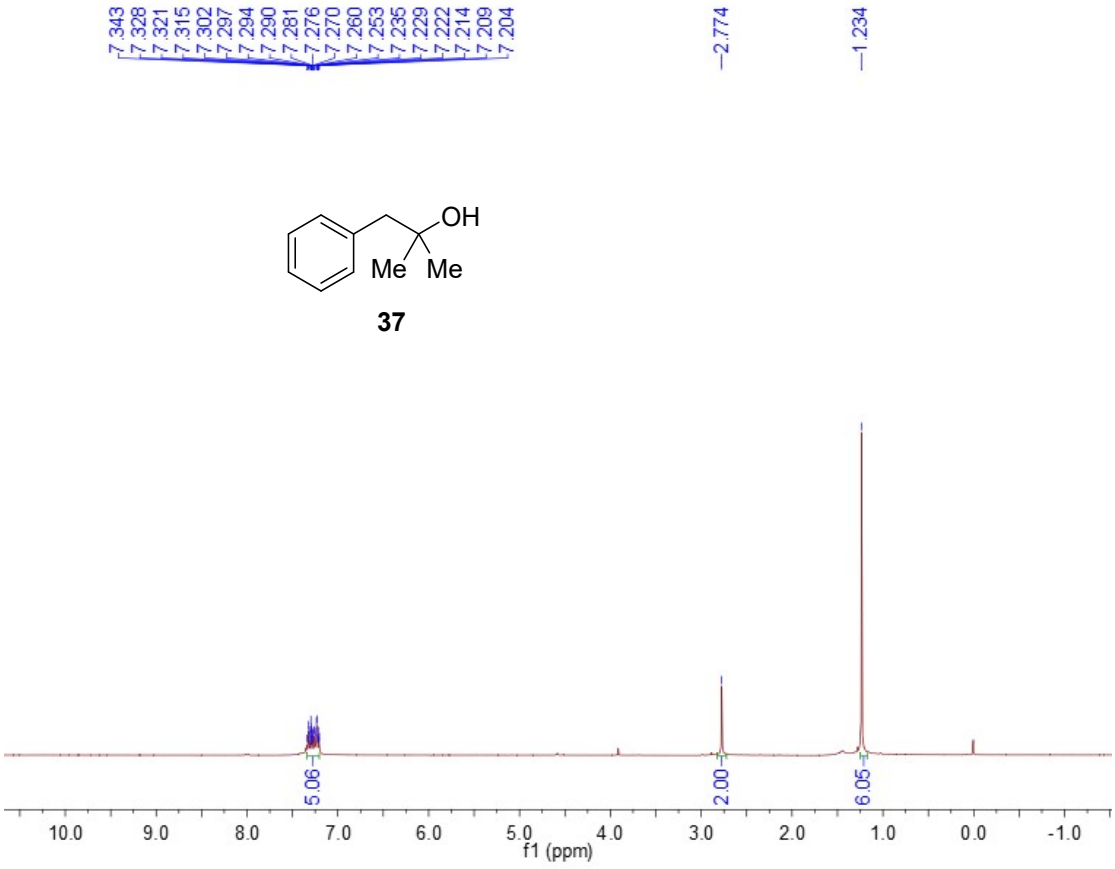


^{1}H NMR (300 MHz, $CDCl_3$):



36

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



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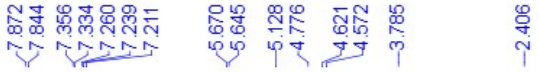
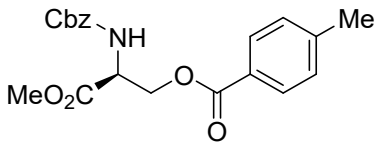
^{1}H NMR (300 MHz, $CDCl_3$):





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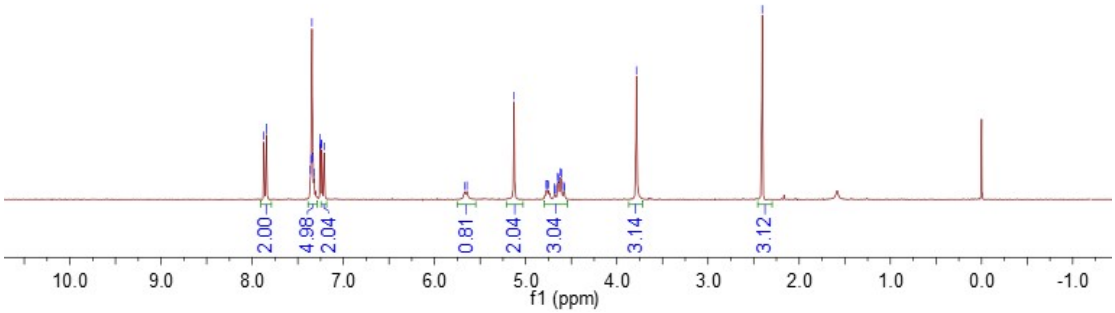
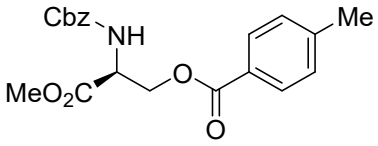


^{13}C NMR (75 MHz, $CDCl_3$):



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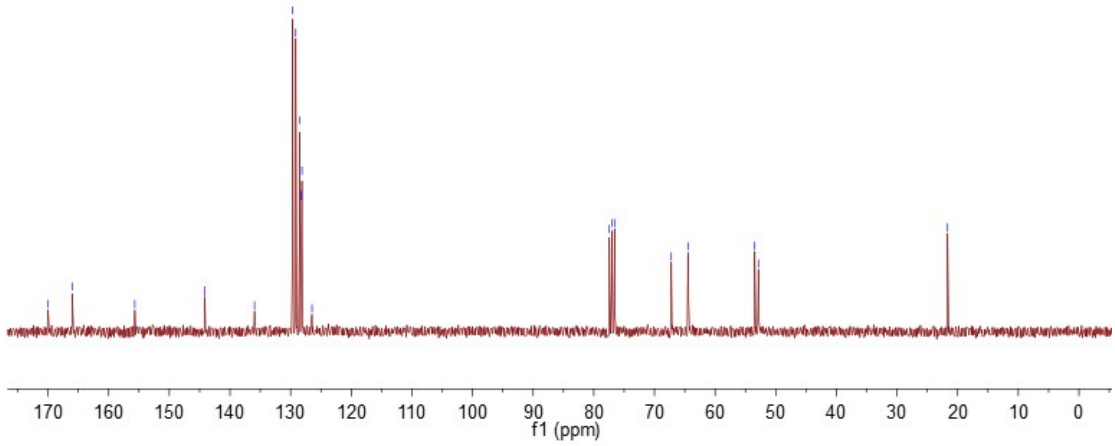
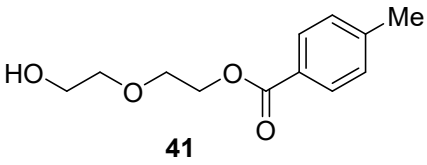


^{1}H NMR (300 MHz, $CDCl_3$):



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