Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Electronic supplementary information

Carboxylic Acid-Assisted Sterically Demanding Reductive Cross-Coupling between Cycloalkenyl and Alkyl Bromides

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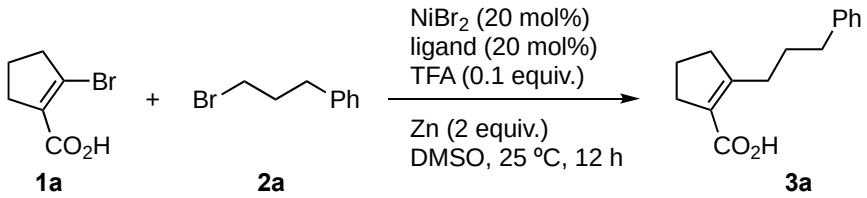
1. General Information

All reactions that require anhydrous conditions were performed in flame-dried glassware under Ar atmosphere and all the commercial reagents were used without purification unless otherwise noted. Solvent purification was conducted according to Purification of Laboratory Chemicals (Peerrin, D. D.; Armarego, W. L. and Perrins, D. R., Pergamon Press: Oxford, 1980). The products were purified by flash column chromatography on silica gel (200-300 meshes) from the Anhui Liangchen Silicon Material Company (China). Reactions were monitored by thin layer chromatography (TLC) supplied by Yantai Chemicals (China). Visualization was accomplished with UV light, exposure to iodine, stained with ethanolic solution of phosphomolybdic acid or basic solution of $KMnO_4$. ^1H NMR and ^{13}C NMR spectra were recorded on Varian INOVA-400/54 and Agilent DD2-600/54 instruments and calibrated by using residual undeuterated chloroform (δ, ^1H NMR = 7.26, ^{13}C NMR = 77.16). The following abbreviations were used to explain the multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, br = broad, td = triple doublet, dt = double triplet, m = multiplet, and coupling constants (J) are reported in Hertz (Hz). Infrared (IR) spectra were recorded on a Perkin Elmer Spectrum Two FT-IR spectrometer. The specific optical rotation was obtained from *Rudolph Research Analytical Autopol VI* automatic polarimeter. High-resolution mass spectra (HRMS) were recorded on Bruker Apex IV FTMS or Thermo Scientific LTQ Orbitrap XL ESI mass spectrometers. LC-MS analysis was performed on HP Agilent 6420 Triple Quad LC/MS.

2. Optimization of the Reaction Conditions

Table S1. Effect of Ligands



Entry	Ligand	Yield (%)[b]
1	No	NR
2	**L1**	73
3	**L2**	50
4	**L3**	42
5	**L4**	32
6	**L5**	30
7	**L6**	24
8	**L7**	17
9	**L8**	10
10	**L9**	5
11	**L10**	35
12	**L11**	30
13	**L12**	42
14	**L13**	9

[a]Alkenyl bromide **1a** (0.53 mmol, 1.0 equiv.) and alkyl bromide **2a** (2.0 equiv.) were used in all reactions unless otherwise stated. [b]Yields were determined according to the isolated material.

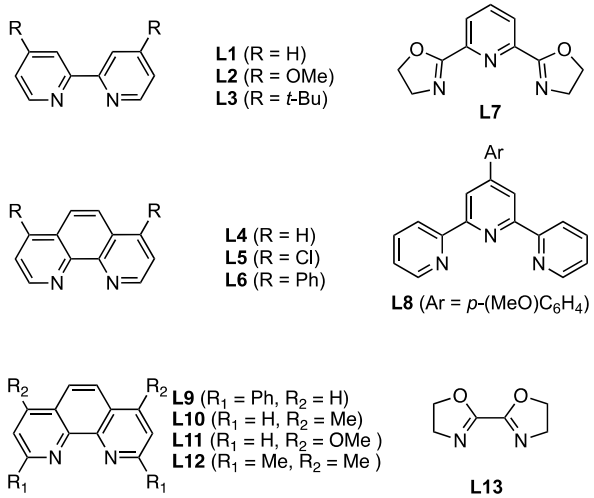
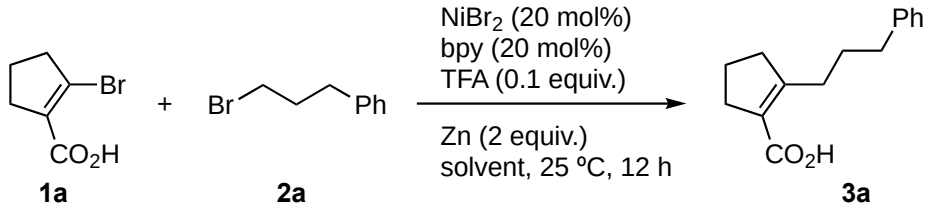


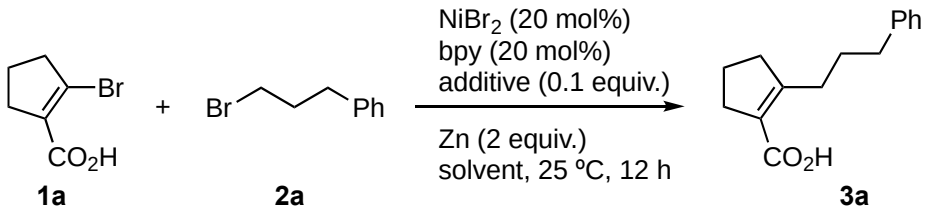
Table S2. Effect of Solvents



Entry	Solvent	Yield (%)[b]
1	DMSO	73
2	pyridine	25
3	DMPU	8
4	NMP	NR
5	DMF	15
6	CH_3CN	NR
7	THF	NR
8	$CHCl_3$	NR
9	PhMe	NR
10	PhH	NR
11	DCM	NR
12	dioxane	NR
13	TBME	NR
14	PhOMe	NR
15	DME	NR

[a]Alkenyl bromide **1a** (0.53 mmol, 1.0 equiv.) and alkyl bromide **2a** (2.0 equiv.) were used in all reactions unless otherwise stated. [b]Yields were determined according to the isolated material.

Table S3. Effect of Additives

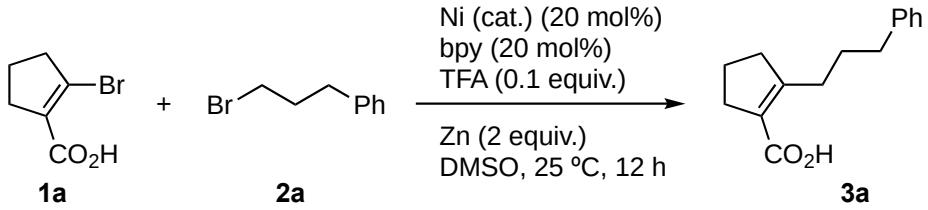


Entry	Additive	Yield (%)[b]
1	NO	Trace
2	TFA	73
3	NaI	68

4	TBAI	43
5	TMSCl	36
6	LiBr	38
7	pyridine	42

[a]Alkenyl bromide **1a** (0.53 mmol, 1.0 equiv.) and alkyl bromide **2a** (2.0 equiv.) were used in all reactions unless otherwise stated. [b]Yields were determined according to the isolated material.

Table S4. Effect of Ni (cat.)

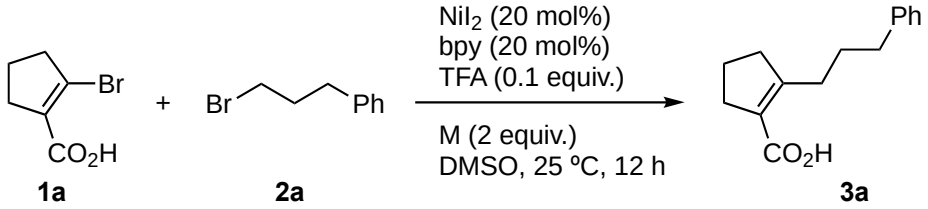


1a **2a** **3a**

Entry	Ni (cat.)	Yield (%)[b]
1	$NiCl_2$	64
2	$NiBr_2$	73
3	NiI_2	92
4	$NiCl_2·6H_2O$	50
5	$NiCl_2(PPh_3)_2$	49
6	$Ni(acac)_2$	26
7	$Ni(CO)_2(PPh_3)_2$	73
8	$Ni(OTf)_2$	29
9	$Ni(COD)_2$	39
10	$NiBr_2·3H_2O$	22
11	$Ni(DPPE)Cl_2$	37
12	$NiCl_2(PCy_3)_2$	72
13	$(DPPP)NiCl_2$	66
14	$(dme)NICl_2$	48
15	$NiI_2·6H_2O$	55
16	$Ni(OAc)_2·4H_2O$	54
17	$NiCl(PPh_3)_2(dppf)$	81
18	$NiBr_2(PPh)_3$	52
19	$(dme)NiBr_2$	85
20	$Ni(ClO_4)_2·6H_2O$	60

[a]Alkenyl bromide **1a** (0.53 mmol, 1.0 equiv.) and alkyl bromide **2a** (2.0 equiv.) were used in all reactions unless otherwise stated. [b]Yields were determined according to the isolated material.

Table S5. Effect of Reducing Agents

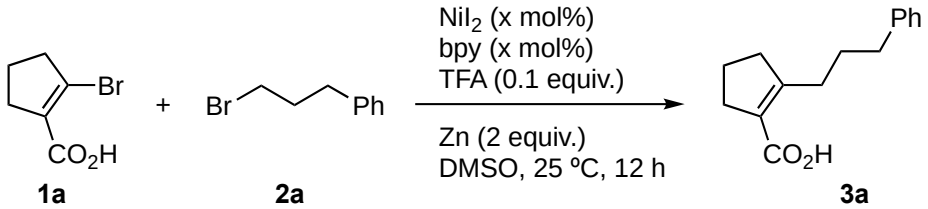


1a **2a** **3a**

Entry	M	Yield (%)[b]
1	Zn	92
2	Mn	<5
3	Fe	NR

[a]Alkenyl bromide **1a** (0.53 mmol, 1.0 equiv.) and alkyl bromide **2a** (2.0 equiv.) were used in all reactions unless otherwise stated. [b]Yields were determined according to the isolated material.

Table S6. Effect of Different Loading of Ni (cat.) and Ligands

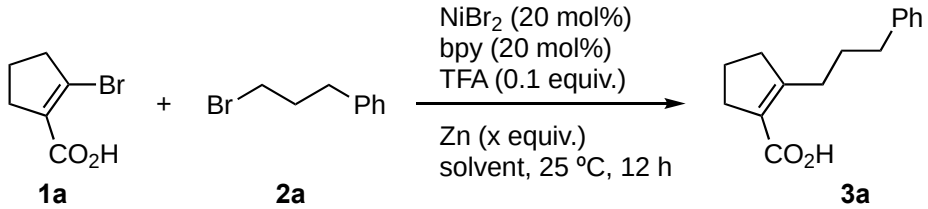


1a **2a** **3a**

Entry	x	Yield (%)[b]
1	25	89
2	20	92
3	15	88
4	10	75[c]
5	7.5	69[c]
6	5	52[c]

[a]Alkenyl bromide **1a** (0.53mmol, 1.0 equiv.) and alkyl bromide **2a** (2.0 equiv.) were used in all reactions unless otherwise stated. [b]Yields were determined according to the isolated material. [c]The reaction time was 24 h.

Table S7. Effect of Different Equivalent of Reducing Agents

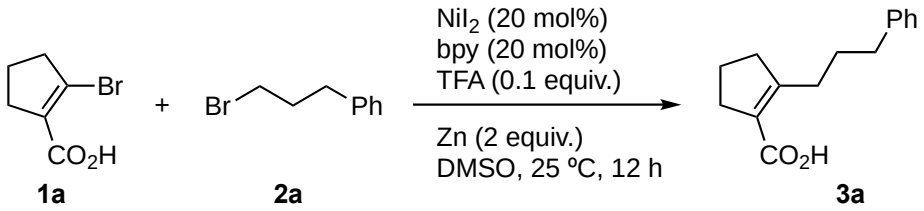


1a **2a** **3a**

Entry	equivalent	Yield (%)[b]
1	2.5	89
2	2.0	92
3	1.75	74 [c]
4	1.5	67 [c]
5	1.25	63 [c]

[a]Alkenyl bromide **1a** (0.53 mmol, 1.0 equiv.) and alkyl bromide **2a** (2.0 equiv.) were used in all reactions unless otherwise stated. [b]Yields were determined according to the isolated material. [c]The reaction time was 24 h.

Table S8. Effect of Different Equivalent of Alkyl Bromide

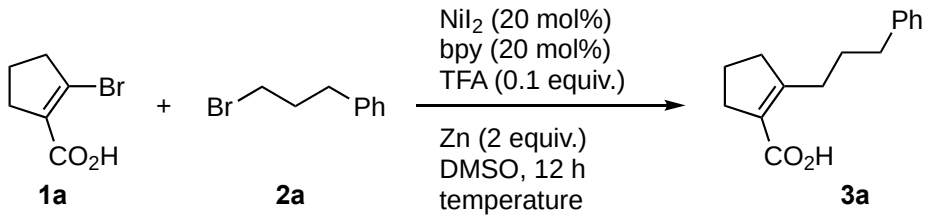


1a **2a** **3a**

Entry	Equivalent of **2a**	Yield (%)[b]
1	2.5	89
2	2.25	90
3	2.0	92
4	1.75	84[c]
5	1.5	78[c]
6	1.25	70[c]

[a]Alkenyl bromide **1a** (0.53 mmol, 1.0 equiv.) and alkyl bromide **2a** (x equiv.) were used in all reactions unless otherwise stated. [b]Yields were determined according to the isolated material. [c]The reaction time was 24 h.

Table S9. Effect of Temperature



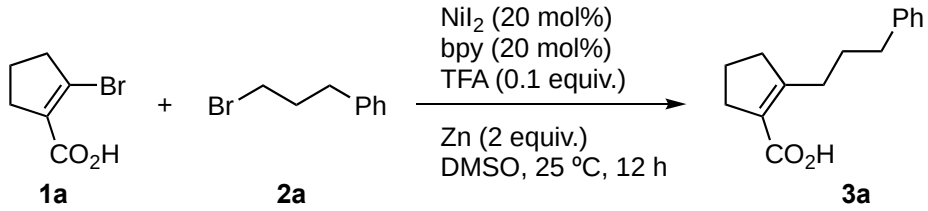
1a **2a** **3a**

Entry	Temperature	Yield (%)[b]
1	0 ℃	NR
2	RT	92

3	50 ℃	85
4	75 ℃	75

[a]Alkenyl bromide **1a** (0.530 mmol, 1.0 equiv.) and alkyl bromide **2a** (2.0 equiv.) were used in all reactions unless otherwise stated. [b]Yields were determined according to the isolated material.

Table S10. Effect of Concentration of 1a

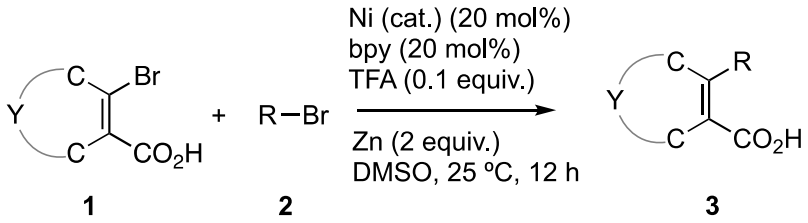


1a **2a** **3a**

Entry	Conc.(mmol/mL) of **1a**	Yield (%)[b]
1	0.075	88
2	0.1	**92**
3	0.25	62[c]
4	0.5	56[c]
5	0.75	54[c]
6	1.0	51[c]

[a]Alkenyl bromide **1a** (0.530 mmol, 1.0 equiv.) and alkyl bromide **2a** (2.0 equiv.) were used in all reactions unless otherwise stated. [b]Yields were determined according to the isolated material. [c]The reaction time was 24 h.

3. General Procedure for the Ni-Catalyzed Cross-Electrophile Coupling

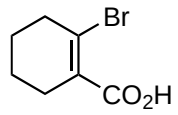


1 **2** **3**

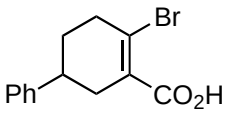
Under argon atmosphere, a solution of acid (100 mg, 1.0 equiv), NiI_2 (0.2 equiv), bpy (0.2 equiv), Zn (2.0 equiv), alkyl bromide compound (2.0 equiv) and TFA (0.1 equiv) in degassed dry DMSO (6 mL) was stirred at room temperature. The mixture was stirred at room temperature for 12 h, after the reaction was completed as indicated by TLC and LC-MS, it was carefully quenched with 0.5-1.0 N HCl aqueous solution (6 mL), and extracted with methyl *tert*-butyl ether (3 × 20 mL). The combined organic phase was dried over

anhydrous $MgSO_4$ and concentrated under vacuum. The crude residue was purified by silica gel flash column chromatography (petroleum ether/ethyl acetate 30/1 to 5/1, v/v, containing 0.5% acetic acid) to afford the corresponding coupling product.

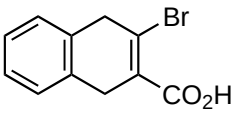
4. Synthesis and Characterization of Substrate



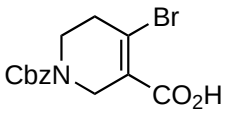
1b



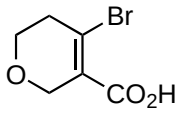
1c



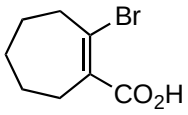
1d



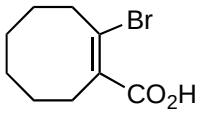
1e



1f

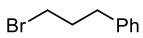


1g



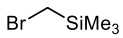
1h

Compounds **1b**–**1h** were prepared according to literature methods.[1 – 3]



2a

2b



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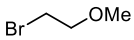

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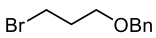

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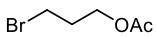

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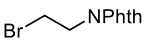

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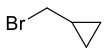

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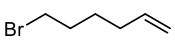

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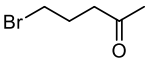

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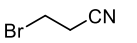

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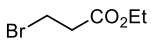

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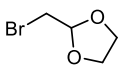

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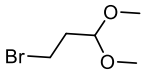

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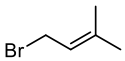

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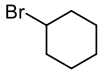
2p

2q



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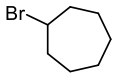

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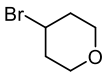

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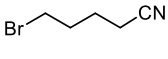

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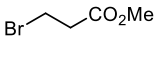

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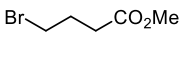
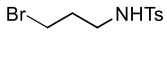
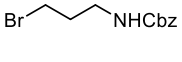


2w

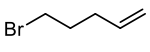


2x

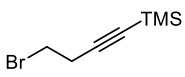


2y

2z



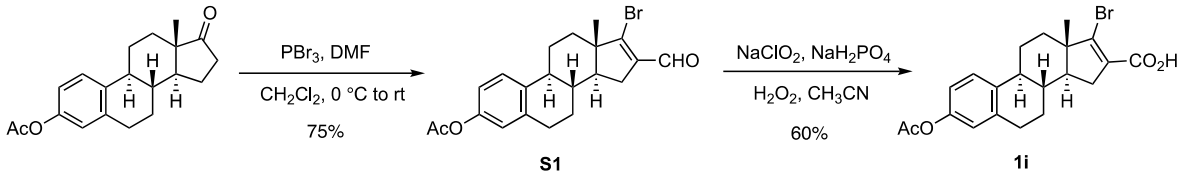
2aa



2ab

Compounds **2a**–**2ab** are commercially available.

Synthesis of Substrate 1i

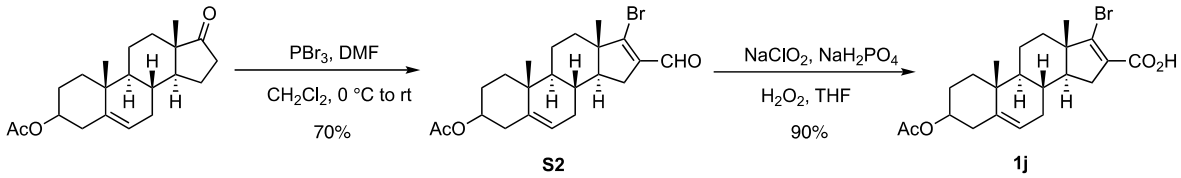


To a mixture of DMF (2.5 mL) and CH_2Cl_2 (20 mL) was slowly added PBr_3 (16.0 mmol, 2.0 equiv, 1.5 mL) at 0 ℃ under Ar. The pale yellow slurry was stirred at room temperature for 2 h. A solution of Estrone (8.0 mmol, 1.0 equiv, 2.5 g) in CH_2Cl_2 (5 mL) was added dropwise to the above solution at room temperature. The mixture was stirred at this temperature for 2 h, then poured into NaOH aqueous solution (20 mL), and extracted with ethyl acetate (3 × 20 mL). The extract was dried over $MgSO_4$. The organic phase was removed under reduced pressure, and the crude product was purified by column chromatography (eluent: petroleum ether/EtOAc = 10/1, v/v) to afford **S1** (2.4 g, 75%).

A solution of $NaClO_2$ (8.4 mmol, 1.4 equiv, 0.9 g) in water (10 mL) was added dropwise in 2 h to a stirred mixture of **S1** (6.0 mmol, 1.0 equiv, 2.4 g) in CH_3CN (10.0 mL), NaH_2PO_4 (1.6 mmol, 0.27 equiv, 0.2 g) in water (5 mL) and 30% aqueous H_2O_2 (0.7 mL), keeping the temperature at 0 ℃ with water cooling. After completion of the reaction (monitored by TLC), the mixture was poured into saturated Na_2CO_3 aqueous solution, and washed with ethyl acetate. The aqueous phase was poured into HCl solution, and extracted with ethyl acetate. The extract was dried over $MgSO_4$. The organic phase was removed under reduced pressure and the crude product was purified by column chromatography (eluent: petroleum ether/EtOAc = 3/1, v/v) to afford **1i** (1.5 g, 60%). Amorphous powder; R_f = 0.55 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27 (d, J = 8.4 Hz, 1H), 6.92 – 6.79 (m, 2H), 2.92 (dt, J = 8.8, 4.4 Hz, 2H), 2.68 (dd, J = 14.8, 6.4 Hz, 1H), 2.44 (m, 1H), 2.31 (d, J = 14.8 Hz, 5H), 2.03 – 1.92 (m, 2H), 1.78 (m, 1H), 1.72 – 1.52 (m, 3H), 1.46 (m, 1H), 0.94 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.0, 169.8, 149.8, 148.7, 138.1, 137.5, 130.5, 126.2, 121.7, 118.8, 53.3, 52.3, 44.3, 37.2, 34.6, 32.7, 29.4, 27.0, 26.0, 21.2, 15.2; IR (neat): ν_{max} = 2927, 2870, 1766, 1691, 1664, 1588, 1495, 1370,

1250, 1203, 1180, 934. 720. 414 cm^{-1}; HRMS (ESI): m/z calculated for $C_{21}H_{23}BrO_4$, $[M - H]^-$ 417.0707, found, 417.0700, $[\alpha]^{25}_D = +41.06$ (c 0.50, $CHCl_3$).

Synthesis of Substrate 1j

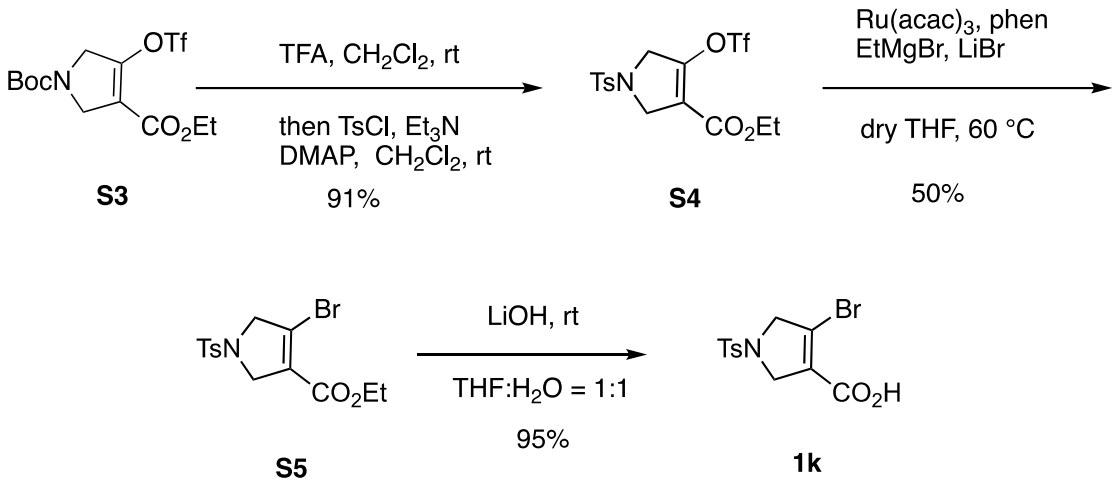


To a mixture of DMF (2.5 mL) and CH_2Cl_2 (20 mL) was slowly added PBr_3 (16.0 mmol, 2.0 equiv, 1.5 mL) at 0 ℃ under Ar. The pale yellow slurry was stirred at room temperature for 2 h. A solution of dehydroepiandrosterone (8.0mmol, 1.0 equiv, 2.6 g) in CH_2Cl_2 (5 mL) was added dropwise to the above solution at room temperature. The mixture was stirred at this temperature for 2 h, then poured into NaOH aqueous solution (20 mL), and extracted with ethyl acetate (3 × 20 mL). The extract was dried over $MgSO_4$. The organic phase was removed under reduced pressure, and the crude product was purified by column chromatography (eluent: petroleum ether/EtOAc = 100/1, v/v) to afford **S2** (2.4 g, 70%).

A solution of $NaClO_2$ (8.0 mmol, 1.4 equiv, 0.9 g) in water (10 mL) was added dropwise in 2 h to a stirred mixture of **S2** (5.7 mmol, 1.0 equiv, 2.4 g) in THF (20 mL), NaH_2PO_4 (1.5 mmol, 0.27 equiv, 0.2 g) in water (5 mL) and 30% aqueous H_2O_2 (0.6 mL), keeping the temperature at 0 ℃ with water cooling. After completion of the reaction (monitored by TLC), the mixture was poured into saturated Na_2CO_3 aqueous solution, and washed with ethyl acetate. The aqueous phase was poured into HCl solution, and extracted with ethyl acetate. The extract was dried over $MgSO_4$. The organic phase was removed under reduced pressure to afford **1j** (2.2 g, 90%). Amorphous powder; R_f = 0.54 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid); ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.08 (s, 1H), 5.37 (d, J = 5.0 Hz, 1H), 4.58 (tt, J = 10.6, 4.9 Hz, 1H), 2.54 (dd, J = 14.9, 6.3 Hz, 1H), 2.32 (q, J = 11.0, 9.4 Hz, 2H), 2.20 (dd, J = 14.8, 11.6 Hz, 1H), 2.01 (s, 4H), 1.90 – 1.77 (m, 3H), 1.75 – 1.60 (m, 3H), 1.53 (q, J = 14.0, 11.4 Hz, 3H), 1.36 (td, J = 12.6, 4.4 Hz, 1H), 1.18 – 0.99 (m, 5H), 0.90 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.8, 169.2, 149.5, 140.2, 130.5, 122.0, 73.9, 54.0, 52.0, 50.3, 38.2, 36.9, 34.5, 33.0, 31.0, 30.8, 27.8, 21.6, 20.6, 19.4, 15.1; IR (neat): ν_{max}

= 2924, 2854, 1763, 1667, 1607, 1495, 1454, 1370, 1257, 1207, 1090, 1015, 942, 900, 800, 751, 699, 667, 564, 493, 439 cm^{-1}; HRMS (ESI): m/z calculated for $C_{22}H_{29}BrO_4$, $[M - H]^-$ 435.1176, found, 435.1159; $[\alpha]^{25}_D$ = + 34.14 (c 0.74, $CHCl_3$).

Synthesis of Substrate 1k

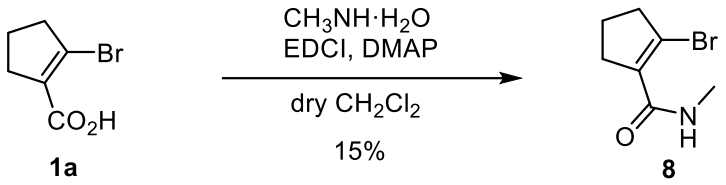


S3 (0.510 mmol, 1.0 equiv, 0.2 g) in CH_2Cl_2 (5 mL) was stirred with TFA (13.0 mmol, 25.5 equiv, 1.0 mL) at room temperature for 3 h. The solvent was removed under reduced pressure. Then the crude residue was dissolved in CH_2Cl_2 (5 mL), to which were sequentially added Et_3N (1.5 mmol, 3.0 equiv, 0.2 mL), DMAP (0.26 mmol, 0.5 equiv, 31.0 mg) and TsCl (1.0 mmol, 2.0 equiv, 196 mg). The solution was stirred at room temperature for 2 h. The reaction was extracted with water (15 mL). The organic layer was dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The crude residue was purified by silica gel flash column chromatography (petroleum ether/ethyl acetate 12/1 to 8/1, v/v) to afford **S4** (207 mg, 91%). White solid; R_f = 0. 32 (petroleum ether/ethyl acetate 6/1, v/v); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.71 (d, J = 8.0 Hz, 2H), 7.36 (d, J = 8.0 Hz, 2H), 4.34 (s, 4H), 4.24 (q, J = 7.2 Hz, 2H), 2.44 (s, 3H), 1.28 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.5, 145.9, 144.8, 133.1, 130.3, 127.6, 119.5, 118.3 (q, J = 319 Hz), 62.1, 52.5, 51.9, 21.7, 14.0; IR (neat): ν_{max} = 2986, 2863, 1732, 1691, 1598, 1427, 1345, 1275, 1215, 1200, 1152, 1136, 1100, 1042, 853, 817, 756, 709, 682, 620, 596, 584, 529, 502, 470, 420, 410 cm^{-1}; HRMS (ESI): m/z calculated for $C_{15}H_{16}F_3NO_7S_2$, $[M + Na]^+$ 466.0212, found, 466.0214.

To a THF solution (0.9 mL) of $Ru(acac)_3$ (0.038 mmol, 0.06 equiv, 10.8 mg), o-phenanthroline(0.038 mmol, 0.06 equiv, 10.8 mg)and LiBr (0.54 mmol, 1.2 equiv, 47 mg) placed in an oven-dried Schlenk tube was added ethylmagnesium bromide (0.11 mmol, 1.0 M THF solution, 0.1 mL,). After stirring for 10 min at room temperature, **S4** (0.45 mmol, 1.0 equiv, 200 mg) was added at 20 °C and stirring was continued for 72 h.[4] The reaction was concentrated under reduced pressure. Purification by passing through a pad of silica gel using Dichloromethane/Acetonitrile (200/1, v/v) afforded **S5** (85 mg, 50%). Amorphous powder; R_f = 0.55 (petroleum ether/ethyl acetate 6/1 ~10/1, v/v); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.73 (d, J = 8.2 Hz, 2H), 7.35 (d, J = 8.0 Hz, 2H), 4.40 – 4.25 (m, 4H), 4.21 (q, J = 7.2 Hz, 2H), 2.44 (s, 3H), 1.29 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 161.1, 144.4, 133.4, 130.2, 127.7, 124.7, 61.5, 61.3, 55.1, 21.7, 14.2; IR (neat): ν_{max} = 2987, 2926, 2856, 1725, 1709, 1641, 1597, 1452, 1494, 1452, 1346, 1305, 1288, 1263, 1162, 1097, 1084, 1016, 948, 861, 835, 815, 761, 708, 668, 617, 610, 563, 547, 510, 465, 410 cm^{-1}; HRMS (ESI): m/z calculated for $C_{14}H_{16}BrNO_4S$, $[M + Na]^+$ 395.9876, found, 395.9880.

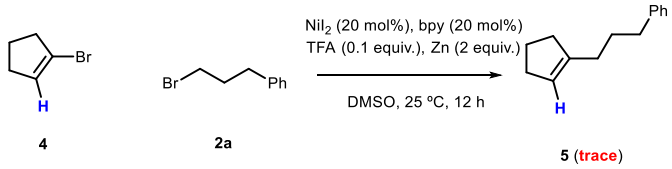
A round bottom flask was charged with **S5** (0.27 mmol, 1.0 equiv, 100 mg), LiOH (2.7 mmol, 10.0 equiv, 64 mg), and THF/H_2O (1/1, v/v, 0.1 M). The reaction was stirred at room temperature 12h before it was acidified with 3 N HCl. The aqueous phase was extracted with ethyl acetate (3 × 10 mL). The combined organic phase was dried over anhydrous $MgSO_4$, filtered, and evaporated under reduced pressure. The residue was purified by column chromatography on silica gel (dichloromethane/methanol = 10/1 ~ 5/1, v/v) to give the corresponding unsaturated acid **1k** (175 mg, 95%). Amorphous powder; R_f = 0.61 (dichloromethane/methanol = 10/1, v/v); ^{1}H NMR (400 MHz, DMSO-d_6) δ 7.77 (d, J = 8.2 Hz, 2H), 7.45 (d, J = 8.0 Hz, 2H), 4.34 (t, J = 4.3 Hz, 2H), 4.16 (t, J = 4.3 Hz, 2H), 2.40 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) δ 162.1, 144.1, 132.6, 130.2, 127.5, 60.7, 55.0, 21.0; IR (neat): ν_{max} = 2986, 2863, 1732, 1691, 1598, 1427, 1345, 1275, 1215, 1200, 1152, 1136, 1100, 1042, 853, 817, 756, 709, 682, 620, 596, 584, 529, 502, 470, 420, 410 cm^{-1}; HRMS (ESI): m/z calculated for $C_{12}H_{12}BrNO_4S$, $[M – H]^-$ 343.9598, found, 343.9590.

Synthesis of Substrate 8

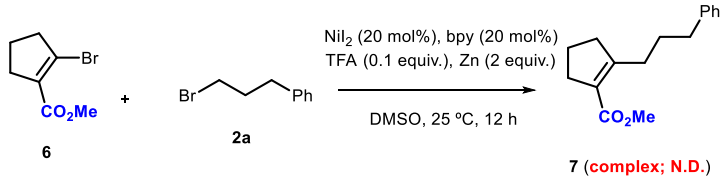


1a (1.05 mmol, 1.0 equiv, 0.2 g) in CH_2Cl_2 (10 mL) was stirred with $CH_3NH{\cdot}H_2O$ (13.0 mmol, 25.5 equiv, 1.0 mL), DMAP (1.05 mmol, 1.0 equiv, 122 mg) and EDCI (1.58 mmol, 1.5 equiv, 301.9 mg) at room temperature for 12 h. The reaction was extracted with water (15 mL). The organic layer was dried over $MgSO_4$, filtered, and concentrated under reduced pressure. The crude residue was purified by silica gel flash column chromatography (petroleum ether/ethyl acetate 12/1 to 4/1, v/v) to afford **8** (29 mg, 15%). Amorphous powder; R_f = 0.25 (petroleum ether/ethyl acetate = 5/1, v/v); ^{1}H NMR (600 MHz, $CDCl_3$) δ 6.42 (s, 1H), 2.91 (dd, J = 5.2, 1.8 Hz, 3H), 2.87 – 2.76 (m, 2H), 2.70 (t, J = 6.2 Hz, 2H), 1.95 (p, J = 6.8 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.8, 136.1, 122.9, 42.9, 33.9, 26.3, 21.4; IR (neat): ν_{max} = 3310, 2936, 2851, 1643, 1532, 1441, 1408, 1303, 1164, 1092, 913, 679, 644, 464 cm^{-1}; HRMS (ESI): m/z calculated for $C_7H_{10}BrNO$, $[M + Na]^+$ 202.9946, found, 202.9941.

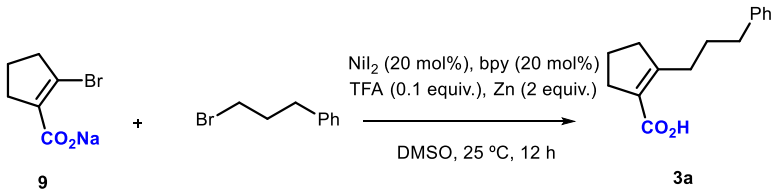
5. Control Experiments



Under argon atmosphere, a solution of acid **4** (100 mg, 1.0 equiv), NiI_2 (0.2 equiv), bpy (0.2 equiv), Zn (2.0 equiv), **2a** (2.0 equiv) and TFA (0.1 equiv) in degassed dry DMSO (6 mL) was stirred at room temperature for 12 h. After the reaction was completed as indicated by TLC and LC-MS, it was carefully quenched with 0.5–1.0 N HCl aqueous solution (6 mL), and extracted with methyl *tert*-butyl ether (3 × 20 mL). The combined organic phase was dried over anhydrous $MgSO_4$ and concentrated under vacuum. The crude residue was purified by silica gel flash column chromatography (petroleum ether/ethyl acetate 30/1 to 5/1, v/v, containing 0.5% acetic acid) to afford the corresponding coupling product.

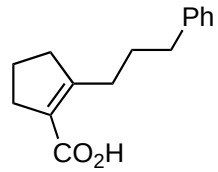


Under argon atmosphere, a solution of acid **6** (100 mg, 1.0 equiv), NiI_2 (0.2 equiv), bpy (0.2 equiv), Zn (2.0 equiv), **2a** (2.0 equiv) and TFA (0.1 equiv) in degassed dry DMSO (6 mL) was stirred at room temperature for 12 h. After the reaction was completed as indicated by TLC and LC-MS, it was carefully quenched with 0.5–1.0 N HCl aqueous solution (6 mL), and extracted with methyl *tert*-butyl ether (3 × 20 mL). The combined organic phase was dried over anhydrous $MgSO_4$ and concentrated under vacuum. The crude residue was purified by silica gel flash column chromatography (petroleum ether/ethyl acetate 30/1 to 5/1, v/v, containing 0.5% acetic acid) to afford the corresponding coupling product.



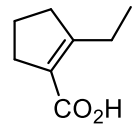
Under argon atmosphere, a solution of acid **9** (110.5 mg, 1.0 equiv), NiI_2 (0.2 equiv), bpy (0.2 equiv), Zn (2.0 equiv), **2a** (2.0 equiv) and TFA (0.1 equiv) in degassed dry DMSO (6 mL) was stirred at room temperature for 12 h. After the reaction was completed as indicated by TLC and LC-MS, it was carefully quenched with 0.5–1.0 N HCl aqueous solution (6 mL), and extracted with methyl *tert*-butyl ether (3 × 20 mL). The combined organic phase was dried over anhydrous $MgSO_4$ and concentrated under vacuum. The crude residue was purified by silica gel flash column chromatography (petroleum ether/ethyl acetate 30/1 to 5/1, v/v, containing 0.5% acetic acid) to afford the coupling product **3a** (101mg, 84%).

6. Characterization Data



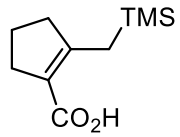
3a

The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3a** (110 mg, 92% yield) as an amorphous powder. R_f = 0.74 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24 (t, J = 7.2 Hz, 2H), 7.20 – 7.11 (m, 3H), 2.64 (m, 6H), 2.49 (t, J = 7.6 Hz, 2H), 1.78 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.1, 163.1, 142.3, 128.5, 128.4, 127.2, 125.9, 38.7, 36.1, 33.5, 30.1, 29.9, 21.5; IR (neat): ν_{max} = 3026, 2921, 1604, 1495, 1460, 1081, 1030, 726, 693, 542, 520, 463, 436, 407 cm^{-1}; HRMS (ESI): m/z calculated for $C_{15}H_{17}O_2$, $[M - H]^-$ 229.1234, found, 229.1225.



3c

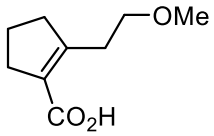
The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3c** (110 mg, 92% yield) as an amorphous powder. R_f = 0.71 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid);^{1}H NMR (400 MHz, $CDCl_3$) δ 2.63 (m, 4H), 2.54 (t, J = 7.6 Hz, 2H), 1.83 (m, 2H), 1.05 (t, J = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.6, 165.0, 126.1, 38.2, 33.6, 23.5, 21.5, 12.6; IR (neat): ν_{max} = 2961, 2918, 2862, 2623, 2573, 1678, 1660, 1626, 1454, 1431, 1412, 1372, 1350, 1299, 1249, 1136, 1121, 1059, 1014, 939, 750, 775, 556, 508, 431, 413 cm^{-1}; HRMS (ESI): m/z calculated for $C_8H_{12}O_2$, $[M - H]^-$ 139.0765, found, 139.0759.



3d

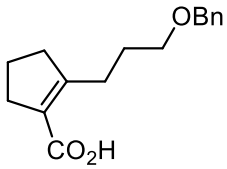
The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3d** (80 mg, 75% yield) as an amorphous powder. R_f = 0.63 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid); ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.62 (t, J = 7.2 Hz, 2H), 2.46 (t, J = 7.6 Hz, 2H), 2.31 (s, 2H), 1.80 (m, 2H), 0.05 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 72.2, 163.6, 122.9, 41.7, 33.1, 24.2, 21.9, -0.6; IR (neat): ν_{max} = 2953, 2592, 1652, 1606, 1431, 1413, 1338, 1413, 1301, 1276, 1276, 1248, 1206, 1151, 1124, 1028, 970, 884, 837, 779, 739, 694, 612, 546, 492, 427, 409 cm^{-1}; HRMS (ESI):

m/z calculated for $C_{10}H_{18}O_2Si$, $[M - H]^-$ 197.1003, found, 197.1006.



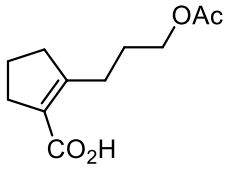
3e

The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3e** (53 mg, 59% yield) as an amorphous powder. R_f = 0.6 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid); ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.53 (t, J = 6.4 Hz, 2H), 3.35 (s, 3H), 2.87 (t, J = 6.4 Hz, 2H), 2.65 (t, J = 7.6 Hz,2H), 2.57 (t, J = 7.2 Hz, 2H), 1.84 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.8, 158.5, 129.1, 70.9, 58.7, 39.1, 33.7, 30.6, 21.5; IR (neat): ν_{max} = 3061, 2928, 2870, 1674, 1632, 1430, 1265, 1195, 1110, 1066, 949, 817, 733, 706, 563, 411 cm^{-1}; HRMS (ESI): m/z calculated for $C_9H_{14}O_3$, $[M - H]^-$ 169.0870, found, 169.0867.



3f

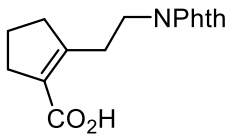
The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3f** (89 mg, 65% yield) as an amorphous powder. R_f = 0.50 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 – 7.16 (m, 5H), 4.42 (s, 2H), 3.41 (t, J = 6.8 Hz, 2H), 2.60 (s, 2H), 2.56 (t, J = 7.2 Hz, 2H), 2.46 (t, J = 7.6Hz, 2H), 1.72 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.7, 171.6, 162.7, 162.7, 138.6, 128.5, 127.8, 127.6, 127.3, 73.0, 70.3, 38.8, 33.6, 28.2, 27.1, 21.5; IR (neat): ν_{max} = 2923, 2855, 1670, 1628, 1495, 1453, 1429, 1412, 1363, 1299, 1271, 1203, 1099, 1027, 941, 734, 696, 609, 565, 459, 425, 407 cm^{-1}; HRMS (ESI): m/z calculated for $C_{16}H_{20}O_3$, $[M - H]^-$ 259.1340, found, 259.1332.



3g

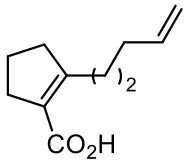
The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3g** (85 mg, 76% yield) as an amorphous powder. R_f = 0.57 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid); ^{1}H NMR (600 MHz, $CDCl_3$) δ 4.06 (t, J = 6.8 Hz, 2H), 2.66 (m, 4H), 2.53 (t, J = 7.6 Hz, 2H), 2.04 (s, 3H), 1.82 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.5, 171.3, 161.7, 127.8, 64.3, 38.7, 33.5, 27.0, 26.7, 21.5, 21.1; IR (neat): ν_{max} = 2955, 1738, 1716, 1672, 1631, 1431, 1389, 1366, 1236, 1117, 1042, 967, 944, 748, 633, 606, 566, 408 cm^{-1}; HRMS (ESI): m/z calculated

for $C_{11}H_{16}O_2$, $[M - H]^-$ 211.0976, found, 211.0970.



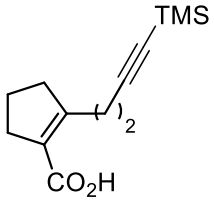
3h

The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3h** (115 mg, 70% yield) as an amorphous powder. R_f = 0.47 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 (m, 2H), 7.69 (m, 2H), 3.87 (t, J = 6.8 Hz, 2H), 2.95 (t, J = 6.8 Hz, 2H), 2.67 – 2.52 (m, 4H), 1.84 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.0, 168.3, 158.0, 132.2, 129.6, 129.6, 123.3, 38.8, 36.1, 33.6, 29.3, 21.7; IR (neat): ν_{max} = 1770, 1705, 1668, 1441, 1406, 1371, 1278, 1100, 1030, 715, 531, 421; HRMS (ESI): m/z calculated for $C_{16}H_{14}NO_4$, $[M - H]^-$ 284.0928, found, 284.0920.



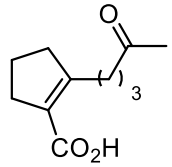
3i

The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3i** (66 mg, 70% yield) as an amorphous powder. R_f = 0.62 (petroleum ether/ethyl acetate 2:1, v/v, containing 0.5% acetic acid); ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.82 (m, 1H), 5.09 – 4.90 (m, 2H), 2.64 (m, 4H), 2.53 (t, J = 7.6 Hz, 2H), 2.12 – 2.03 (m, 2H), 1.83 (m, 2H), 1.63 – 1.50 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.3, 163.0, 138.5, 126.8, 114.7, 38.6, 33.8, 33.5, 29.8, 27.3, 21.4; IR (neat): ν_{max} = 2925, 2856, 1674, 1631, 1430, 1413, 1343, 1270, 1115, 992, 910, 748, 565, 420, 407 cm^{-1}; HRMS (ESI): m/z calculated for $C_{11}H_{16}O_2$, $[M - H]^-$ 179.1078, found, 179.1070.



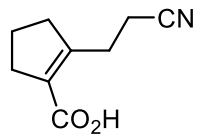
3j

The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3j** (79 mg, 63% yield) as an amorphous powder. R_f = 0.75 (petroleum ether/ethyl acetate 2:1, v/v, containing 0.5% acetic acid); ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.81 (t, J = 7.2 Hz, 2H), 2.72 – 2.58 (m, 4H), 2.42 (t, J = 7.2 Hz, 2H), 1.85 (m, 2H), 0.13 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.4, 161.3, 128.1, 106.7, 85.3, 39.3, 33.5, 29.2, 21.5, 18.8, 0.2; IR (neat): ν_{max} = 2957, 2611, 2177, 1675, 1629, 1432, 1410, 1346, 1298, 1274, 1248, 1116, 1044, 942, 838, 760, 699, 638, 562, 513, 440, 406 cm^{-1} ; HRMS (ESI): m/z calculated for $C_{13}H_{20}O_2Si$, $[M - H]^-$ 235.1160, found, 235.1152.



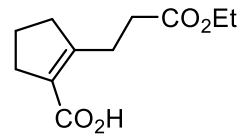
3k

The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3k** (59 mg, 57% yield) as an amorphous powder. R_f = 0.47 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid); ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.62 (q, J = 8.4 Hz, 4H), 2.53 (t, J = 7.6 Hz, 2H), 2.45 (t, J = 7.6 Hz, 2H), 2.13 (s, 3H), 1.79 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 209.0, 171.5, 162.4, 127.6, 43.3, 38.6, 33.5, 30.1, 29.4, 22.0, 21.5; IR (neat): ν_{max} = 2948, 2156, 1708, 1671, 1628, 1410, 1356, 1272, 1189, 1115, 1062, 940, 776, 745, 565, 430, 406; HRMS (ESI): m/z calculated for $C_{11}H_{16}O_2$, $[M - H]^-$ 195.1027, found, 195.1019.



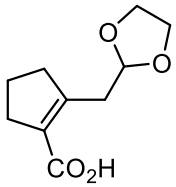
3l

The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3l** (80 mg, 91% yield) as an amorphous powder. R_f = 0.6 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid); ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.94 (t, J = 7.2 Hz, 2H), 2.67 (m, 4H), 2.56 (t, J = 7.2 Hz, 2H), 1.91 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.8, 158.0, 130.0, 119.3, 38.9, 33.3, 25.8, 21.3, 15.9; IR (neat): ν_{max} = 2961, 2918, 2869, 2611, 2245, 1804, 1669, 1632, 1436, 1413, 1354, 1342, 1296, 1272, 1223, 1137, 1119, 1042, 944, 774, 760, 705, 555, 469, 430, 407 cm^{-1}; HRMS (ESI): m/z calculated for $C_{11}H_{16}O_2$, $[M - H]^-$ 179.1078, found, 179.1071.



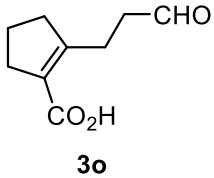
3m

The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3m** (90 mg, 80% yield) as an amorphous powder. R_f = 0.58 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid); ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.13 (q, J = 7.2 Hz, 1H), 2.90 (t, J = 8.0 Hz, 1H), 2.64 (t, J = 7.6 Hz, 1H), 2.51 (M, 2H), 1.84 (p, J = 7.6 Hz, 1H), 1.25 (t, J = 7.2 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.1, 170.7, 160.1, 128.2, 60.6, 38.4, 33.4, 32.5, 25.4, 21.4, 14.2; IR (neat): ν_{max} = 2959, 1733, 1676, 1633, 1431, 1373, 1348, 1274, 1179, 1049, 1030, 943, 860, 784, 747, 544, 418 cm^{-1}; HRMS (ESI): m/z calculated for $C_{11}H_{16}O_4$, $[M - H]^-$ 211.0976, found, 211.0970.



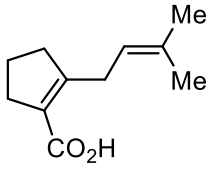
3n

The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3n** (58 mg, 56% yield) as an amorphous powder. R_f = 0.57 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid); ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.07 (t, J = 5.2 Hz, 1H), 4.06 – 3.93 (m, 2H), 3.91 – 3.81 (m, 2H), 2.97 (d, J = 4.8 Hz, 2H), 2.65 (m, 6H), 1.86 (p, J = 7.6 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.6, 155.7, 129.9, 103.3, 65.0, 39.7, 35.2, 33.6, 21.6; IR (neat): ν_{max} = 2887, 1674, 1634, 1427, 1277, 1126, 1040, 942, 827, 748, 566, 408; HRMS (ESI): m/z calculated for $C_{10}H_{14}O_4$, $[M - H]^-$ 197.0819, found, 197.0815.



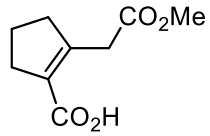
3o

The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3o** (110 mg, 70% yield) as an amorphous powder. R_f = 0.56 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid); ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.78 (s, 1H), 2.91 (t, J = 7.2 Hz, 2H), 2.61 (M, 4H), 2.53 (t, J = 7.2 Hz, 2H), 1.84 (M, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.8, 171.4, 160.7, 128.3, 42.1, 38.8, 33.4, 23.0, 21.4; IR (neat): ν_{max} = 2948, 2932, 1677, 1633, 1434, 1388, 1300, 1193, 1125,1059, 961, 444 cm^{-1}; HRMS (ESI): m/z calculated for $C_9H_{12}O_3$, $[M - H]^-$ 167.0714, found, 167.0706.



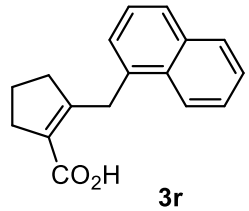
3p

The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3p** (66 mg, 760% yield) as an amorphous powder. R_f = 0.63 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid); ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.14 (t, J = 7.6 Hz, 1H), 3.33 (d, J = 7.2 Hz, 2H), 2.64 (t, J = 7.2 Hz, 2H), 2.51 (t, J = 7.6 Hz, 2H), 1.81 (m, 2H), 1.68 (d, J = 15.6 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.7, 162.0, 133.5, 126.0, 120.2, 38.4, 33.4, 29.7, 29.4, 25.7, 21.3, 17.9; IR (neat): ν_{max} = 2926, 2858, 2609, 1674, 1629, 1430, 1414, 1345, 1268, 1203, 1116, 992, 909, 745, 636, 554, 433, 408 cm^{-1} ; HRMS (ESI): m/z calculated for $C_{11}H_{16}O_2$, $[M - H]^-$ 179.1078, found, 179.1071.



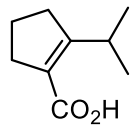
3q

The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3q** (67 mg, 70% yield) as an amorphous powder. R_f = 0.64 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid); ^{1}H NMR (600 MHz, $CDCl_3$) δ 4.71 (s, 2H), 3.82 – 3.76 (m, 3H), 2.87 – 2.81 (m, 2H), 2.74 – 2.68 (m, 2H), 2.00 (p, J = 7.8 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 168.3, 163.2, 135.0, 131.0, 60.6, 52.4, 43.4, 33.0, 21.8; IR (neat): ν_{max} = 2955, 2852, 1763, 1728, 1620, 1436, 1380, 1331, 1259, 1219, 1134, 1086, 1033, 975, 892, 775, 702, 641, 570, 455, 404 cm^{-1}; HRMS (ESI): m/z calculated for $C_{11}H_{16}O_2$, $[M - H]^-$ 183.0663, found, 183.0667.



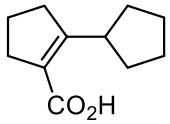
3r

The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3r** (26 mg, 20% yield) as an amorphous powder. R_f = 0.37 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid);^{1}H NMR (400 MHz, $CDCl_3$) δ 8.13 – 8.03 (m, 1H), 7.85 (dd, J = 7.2, 2.2 Hz, 1H), 7.75 (d, J = 8.1 Hz, 1H), 7.55 – 7.31 (m, 4H), 4.49 (s, 2H), 2.78 – 2.66 (m, 2H), 2.28 (t, J = 8.0 Hz, 2H), 1.74 (p, J = 7.6 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.4, 161.4, 135.3, 133.9, 132.5, 128.8, 127.7, 127.3, 127.0, 126.2, 125.8, 125.6, 124.1, 38.4, 34.1, 33.7, 21.3, 0.1; IR (neat): ν_{max} = 2924, 1672, 1629, 1597, 1510, 1428, 1344, 1276, 1221, 1199, 1018, 942, 776, 688, 556, 528, 413 cm^{-1}; HRMS (ESI): m/z calculated for $C_{11}H_{16}O_2$, $[M - H]^-$ 251.1078, found, 251.1074.



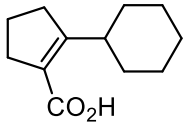
3s

The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3s** (66 mg, 82% yield) as an amorphous powder. R_f = 0.64 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid); ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.72 (m, 1H), 2.62 (m, 2H), 2.51 (m, 2H), 1.80 (p, J = 7.6 Hz, 2H), 1.03 (d, J = 7.0 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.4, 161.3, 128.1, 106.7, 85.3, 39.3, 33.5, 29.2, 21.5, 18.8, 0.2; HRMS (ESI): m/z calculated for $C_9H_{14}O_2$, $[M - H]^-$ 153.0921, found, 153.0915.



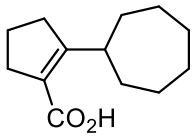
3t

The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3t** (65 mg, 69% yield) as an amorphous powder. R_f = 0.71 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid); ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.78 (m, 1H), 2.63 (t, J = 7.6 Hz, 2H), 2.52 (t, J = 7.6 Hz, 2H), 1.81 (m, 4H), 1.72 – 1.56 (m, 4H), 1.46 – 1.31 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.2, 166.6, 126.5, 39.8, 34.4, 33.6, 26.0, 21.7; IR (neat): ν_{max} = 2949, 2865, 1668, 1622, 1409, 1347, 1300, 1265, 1206, 1137, 953, 731, 568, 500, 416 cm^{-1}; HRMS (ESI): m/z calculated for $C_{11}H_{16}O_2$, $[M - H]^-$ 179.1078, found, 179.1071.



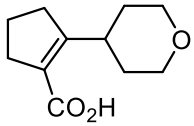
3u

The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3u** (51 mg, 50% yield) as an amorphous powder. R_f = 0.72 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid); ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.49 (s, 1H), 3.38 (m, 1H), 2.70 – 2.57 (m, 2H), 2.55 – 2.46 (m, 2H), 1.86 – 1.58 (m, 7H), 1.43 – 1.13 (m, 5H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.2, 168.1, 125.4, 38.7, 34.7, 33.5, 31.3, 26.3, 26.3, 21.6; IR (neat): ν_{max} = 2919, 2852, 1669, 1623, 1439, 1409, 1346, 1302, 1279, 937, 756, 566, 460, 406 cm^{-1}; HRMS (ESI): m/z calculated for $C_{12}H_{18}O_2$, $[M - H]^-$ 193.1234, found, 193.1226



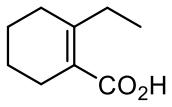
3v

The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3v** (60 mg, 55% yield) as an amorphous powder. R_f = 0.6 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid); ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.56 (m, 1H), 2.60 (t, J = 7.6 Hz, 2H), 2.52 (t, J = 7.6 Hz, 2H), 1.78 (m, 2H), 1.73 – 1.58 (m, 6H), 1.51 (m, 4H), 1.41 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.7, 168.8, 124.0, 39.8, 34.3, 33.2, 33.1, 28.2, 27.3, 21.7; IR (neat): ν_{max} = 2916, 2852, 1668, 1620, 1440, 1408, 1344, 1301, 1280, 1199, 1135 ,933, 825, 755, 736, 564, 425 cm^{-1}; HRMS (ESI): m/z calculated for $C_{13}H_{20}O_2$, $[M - H]^-$ 207.1391, found, 207.1385.



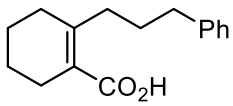
3w

The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3w** (67 mg, 75% yield) as an amorphous powder. R_f = 0.57 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid); ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.99 (dd, J = 11.2, 4.0 Hz, 2H), 3.65 (m, 1H), 3.54 – 3.44 (m, 2H), 2.69 – 2.59 (m, 2H), 2.53 (t, J = 7.6 Hz, 2H), 1.82 (m, 2H), 1.65 (m, 2H), 1.57 – 1.47 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.4, 165.4, 126.6, 68.0, 35.8, 34.8, 33.6, 30.7, 21.5; IR (neat): ν_{max} = 2956, 2920, 2851, 1661, 1620, 1437, 1410 ,1387, 1351, 1305, 1257, 1118, 1080, 1009, 984, 877, 822, 725, 570, 549, 462, 421 cm^{-1}; HRMS (ESI): m/z calculated for $C_{11}H_{16}O_3$, $[M - H]^-$ 195.1027, found, 195.1021.



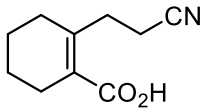
3x

The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3x** (64 mg, 85% yield) as an amorphous powder. R_f = 0.65 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid); ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.45 (q, J = 7.6 Hz, 2H), 2.30 (m, 2H), 2.18 (m, 2H), 1.61 (p, J = 3.2 Hz, 4H), 1.06 (t, J = 7.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 174.6, 155.7, 123.0, 31.6, 28.9, 26.4, 22.5, 22.4, 13.3; IR (neat): ν_{max} = 3051, 2934, 2857, 2627, 1676, 1619, 1455, 1404, 1285, 1263, 1247, 1180, 1088, 1044, 936, 875,855, 828, 788,739, 636, 531 cm^{-1}; HRMS (ESI): m/z calculated for $C_9H_{14}O_2$ $[M - H]^-$ 153.0921, found, 153.0914.



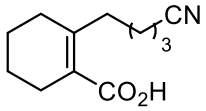
3y

The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3y** (107 mg, 90% yield) as an amorphous powder. R_f = 0.74 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 – 7.10 (m, 5H), 2.65 (t, J = 7.6 Hz, 2H), 2.50 (t, J = 7.6 Hz, 2H), 2.32 (m, 2H), 2.15 (m, 2H), 1.79 (m, 2H), 1.59 (s, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 174.4, 153.8, 142.5, 128.5, 128.4, 125.8, 36.2, 35.6, 32.2, 30.5, 26.5, 22.5, 22.4; HRMS (ESI): m/z calculated for $C_{16}H_{20}O_2$, $[M - H]^-$ 243.1391, found, 243.1384.



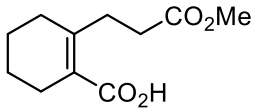
3z

The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3z** (75 mg, 86% yield) as an amorphous powder. R_f = 0.38 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid); ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.76 (t, J = 7.6 Hz, 2H), 2.57 (t, J = 7.2 Hz, 2H), 2.33 (p, J = 4.0 Hz, 2H), 2.28 (m, 2H), 1.64 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.3, 150.6, 126.5, 119.6, 32.9, 31.4, 26.2, 22.0, 21.9, 16.3; IR (neat): ν_{max} = 2939, 2869, 2614, 2243, 2154, 2012, 1666, 1624, 1451, 1404, 1343, 1285, 1264, 1188, 1160, 1138, 1079, 1049, 946, 901, 882, 823, 763, 737, 704, 666, 551, 506, 454, 433 cm^{-1}; HRMS (ESI): m/z calculated for $C_{10}H_{13}NO_2$, $[M - H]^-$ 178.0874, found, 178.0863.



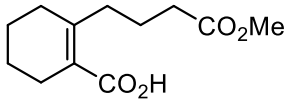
3aa

The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3aa** (85 mg, 84% yield) as an amorphous powder. R_f = 0.41 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid); ^{1}H NMR (600 MHz, $CDCl_3$) δ 2.49 – 2.44 (m, 2H), 2.37 (t, J = 7.2 Hz, 2H), 2.31 (s, 2H), 2.18 (s, 2H), 1.73 – 1.67 (m, 2H), 1.63 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.8, 153.0, 124.3, 119.9, 77.5, 77.2, 76.8, 34.7, 32.2, 27.6, 26.5, 25.5, 22.4, 22.3, 17.1; IR (neat): ν_{max} = 2931, 2861, 2628, 2247, 1675, 1622, 1450, 1421, 1282, 1256, 1179, 1135, 1111, 1079, 938, 771, 741, 659, 538, 407 cm^{-1}; HRMS (ESI): m/z calculated for $C_{12}H_{17}NO_2$, $[M - H]^-$ 206.1187, found, 206.1200.



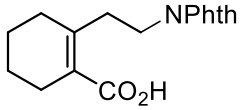
3ab

The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3ab** (80 mg, 77% yield) as an amorphous powder. R_f = 0.66 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid); ^{1}H NMR (600 MHz, $CDCl_3$) δ 3.68 (s, 3H), 2.71 (t, J = 5.2 Hz, 2H), 2.51 (t, J = 5.2 Hz, 1H), 2.31 (m, 2H), 2.19 – 2.13 (m, 2H), 1.61 (s, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 174.0, 173.1, 150.3, 125.4, 51.7, 32.7, 31.6, 30.8, 26.4, 22.1; IR (neat): ν_{max} = 2933, 2861, 2632, 1737, 1679, 1626, 1436, 1368, 1260, 1194, 1172, 1074, 1047, 970, 948, 876, 785, 732, 709, 641, 465 cm^{-1}; HRMS (ESI): m/z calculated for $C_{11}H_{16}O_4$, $[M - H]^-$ 211.0976, found, 211.0969.



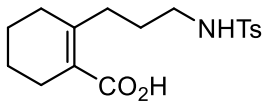
3ac

The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3ac** (84 mg, 72% yield) as an amorphous powder. R_f = 0.6 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid); ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.67 (s, 3H), 2.47 (t, J = 8.0 Hz, 2H), 2.39 – 2.27 (m, 4H), 2.22 – 2.13 (m, 2H), 1.80 (p, J = 7.6 Hz, 2H), 1.60 (p, J = 3.2 Hz, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 174.2, 173.7, 152.8, 124.3, 51.6, 34.9, 34.0, 32.0, 26.5, 23.9, 22.4, 22.3; IR (neat): ν_{max} = 2933, 2860, 2630, 1737, 1680, 1625, 1637, 1369, 1282, 1258, 1196, 1175, 1142, 1078, 1025, 941, 742, 547 cm^{-1}; HRMS (ESI): m/z calculated for $C_{12}H_{18}O_4$, $[M - H]^-$ 225.1132, found, 225.1124.



3ad

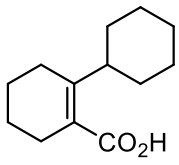
The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3ad** (104 mg, 71% yield) as an amorphous powder. R_f = 0.23 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 (m, 2H), 7.68 (m, 2H), 3.86 (t, J = 6.8 Hz, 2H), 2.78 (t, J = 6.8 Hz, 2H), 2.29 – 2.18 (m, 4H), 1.62 (s, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.5, 168.3, 148.1, 133.7, 132.2, 126.5, 123.1, 36.4, 34.3, 31.9, 26.5, 22.1, 22.0; IR (neat): ν_{max} = 2933, 1771, 1709, 1467, 1437, 1395, 1360, 1282, 1264, 1188, 1099, 1039, 1006, 940, 873, 794, 718, 530, 407 cm^{-1}; HRMS (ESI): m/z calculated for $C_{17}H_{17}NO_4$, $[M - H]^-$ 298.1085, found, 225.1124.



3ae

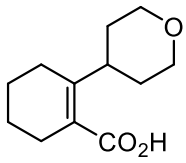
The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3ae** (98 mg, 60% yield) as an amorphous powder. R_f = 0.6 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 – 7.55 (m, 2H), 7.27 (m, 3H), 5.56 (s, 1H), 3.11 – 2.72 (m, 2H), 2.42 (m, 5H), 2.27 (s, 2H), 2.08 (s, 2H), 1.67 (m, 2H), 1.56 (s, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 174.2, 152.7, 143.2, 137.2, 129.7, 127.2, 124.6, 42.5, 32.3, 31.8, 27.8, 26.4, 22.3, 22.1, 21.6; IR (neat): ν_{max} = 3271, 2929, 2859, 1674, 1623, 1598, 1495, 1419, 1322, 1304,

1286, 1262, 1212, 1182, 1153, 1092, 1019, 943, 908, 813, 730, 706, 661, 569, 549 cm^{-1}; HRMS (ESI): m/z calculated for $C_{17}H_{23}NO_4S$, $[M - H]^-$ 336.1275, found, 336.1264.



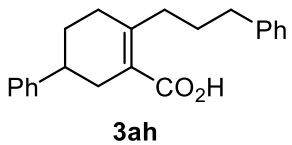
3af

The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3af** (48mg, 47% yield) as an amorphous powder. ^1H NMR (400 MHz, $CDCl_3$) δ 3.12 (t, J = 9.3 Hz, 1H), 2.31 (s, 2H), 2.12 (s, 2H), 1.79 – 1.53 (m, 8H), 1.41 – 0.91 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 174.6, 155.4, 122.9, 42.5, 31.1, 27.0, 26.6, 26.4, 26.1, 22.4, 22.4; IR (neat): ν_{max} = 2923, 2852, 2620, 1679, 1611, 1449, 1404, 1281, 1259, 1242, 1136, 1043, 988, 939, 744, 689, 561, 513, 478, 407 cm^{-1}; HRMS (ESI): m/z calculated for $C_{13}H_{20}O_2$, $[M - H]^-$ 207.1391, found, 207.1384.



3ag

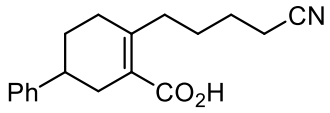
The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3ag** (65 mg, 63% yield) as an amorphous powder. R_f = 0.54 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid); ^1H NMR (400 MHz, $CDCl_3$) δ 3.99 (dd, J = 11.2, 4.4 Hz, 2H), 3.59 – 3.33 (m, 3H), 2.32 (m, 2H), 2.18 – 2.08 (m, 2H), 1.71 (m, 2H), 1.60 (4, 2H), 1.47 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 174.4, 153.3, 124.2, 68.1, 39.5, 30.4, 26.8, 26.2, 22.1; IR (neat): ν_{max} = 2946, 2927, 2856, 1680, 1627, 1442, 1419, 1390, 1362, 1284, 1259, 1239, 1205, 1141, 1116, 1041, 1005, 981, 964, 905, 847, 826, 814, 752, 703, 611, 561, 510 ,462, 485 cm^{-1}; HRMS (ESI): m/z calculated for $C_{12}H_{18}O_3$, $[M - H]^-$ 209.1183, found, 209.1175.



3ah

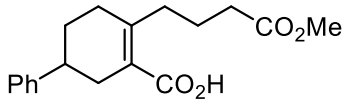
The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3ah** (99 mg, 87% yield) as an amorphous powder. R_f = 0.65 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid); ^1H NMR (400 MHz, $CDCl_3$) δ 7.24 (m, 10H), 2.82 – 2.56 (m, 5H), 2.49 (m, 1H), 2.35 (m, 3H), 2.01 – 1.65 (m, 4H); ^{13}C NMR 100 MHz, $CDCl_3$) δ 173.9, 153.7, 146.1, 142.4, 128.6, 128.5, 128.4, 127.0, 126.4, 125.9, 123.5, 39.8, 36.2, 35.2, 34.4, 32.8, 30.5, 29.3; IR (neat): ν_{max} =

3024, 2924, 2858, 2607, 1681, 1661, 1620, 1495, 1454, 1421, 1400, 1283, 1260, 1156, 1078, 1030, 944, 908, 751, 699, 586, 525, 489, 457, 412, 404 cm^{-1}; HRMS (ESI): m/z calculated for $C_{22}H_{24}O_2$, $[M - H]^-$ 319.1704, found, 319.1695.



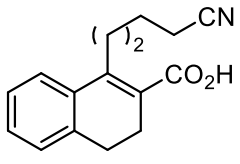
3ai

The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3ai** (82 mg, 80% yield) as an amorphous powder. R_f = 0.58 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 (m, 2H), 7.26 – 7.17 (m, 3H), 2.76 (m, 2H), 2.59 (m, 1H), 2.54 – 2.46 (m, 1H), 2.46 – 2.31 (m, 5H), 1.98 (m, 1H), 1.71 (m, 5H); ^{13}C NMR 100 MHz, $CDCl_3$) δ 173.6, 152.9, 145.8, 128.5, 126.8, 126.3, 123.9, 119.8, 39.7, 34.3, 34.3, 32.8, 29.1, 27.6, 25.4, 16.9; IR (neat): ν_{max} = 3028, 2926, 2863, 2606, 2246, 1678, 1623, 1494, 1455, 1422, 1281, 1260, 1197, 1073, 1030, 940, 916, 761, 701, 604, 520, 413 cm^{-1}; HRMS (ESI): m/z calculated for $C_{18}H_{21}NO_2$, $[M - H]^-$ 282.1500, found, 282.1491.



3aj

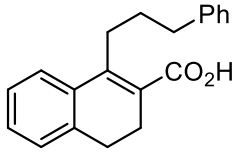
The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3aj** (90 mg, 84% yield) as an amorphous powder. R_f = 0.55 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 (t, J = 7.6 Hz, 2H), 7.25 – 7.18 (m, 3H), 3.68 (s, 3H), 2.85 – 2.69 (m, 2H), 2.60 (m, 1H), 2.53 – 2.46 (m, 1H), 2.36 (q, J = 10.8, 9.2 Hz, 5H), 2.01 – 1.69 (m, 4H); ^{13}C NMR 100 MHz, $CDCl_3$) δ 174.1, 152.7, 146.0, 128.5, 126.9, 126.4, 124.1, 51.6, 39.8, 34.5, 34.4, 33.9, 32.6, 29.2, 23.9; IR (neat): ν_{max} = 3024, 2924, 2858, 2607, 1681, 1661, 1620, 1495, 1454, 1421, 1400, 1283, 1260, 1156, 1078, 1030, 944, 908, 751, 699, 586, 525, 489, 457, 412, 404 cm^{-1}; HRMS (ESI): m/z calculated for $C_{18}H_{22}O_4$, $[M - H]^-$ 301.1445, found, 301.1436.



3ak

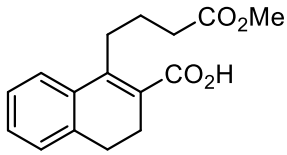
The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3ak** (64 mg, 64% yield) as an amorphous powder. R_f = 0.48 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5%

acetic acid); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 – 7.44 (m, 1H), 7.33 – 7.25 (m, 2H), 7.21 (m, 1H), 3.08 (t, J = 7.6 Hz, 2H), 2.76 (m, 2H), 2.61 (m, 2H), 2.40 (t, J = 6.8 Hz, 2H), 1.84 – 1.67 (m, 4H); ^{13}C NMR 100 MHz, $CDCl_3$) δ 174.0, 148.0, 138.4, 134.0, 129.2, 127.9, 126.8, 125.7, 125.0, 119.8, 28.7, 28.2, 28.1, 25.4, 24.9, 16.9; IR (neat): ν_{max} = 2937, 2882, 2630, 2246, 1671, 1601, 1563, 1451, 1425, 1265, 1214, 1168, 1133, 1030, 942, 846, 767, 736, 562, 493, 431 cm^{-1}; HRMS (ESI): m/z calculated for $C_{16}H_{17}NO_2$, $[M - H]^-$ 254.1187, found, 254.1178.



3al

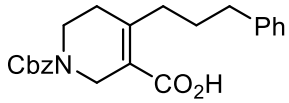
The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3al** (74 mg, 71% yield) as an amorphous powder. R_f = 0.71 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 – 7.18 (m, 9H), 3.15 – 3.06 (m, 2H), 2.83 – 2.74 (m, 4H), 2.64 (m, 2H), 1.94 (m, 2H); ^{13}C NMR 100 MHz, $CDCl_3$) δ 174.7, 148.9, 142.2, 138.4, 134.5, 128.9, 128.6, 128.3, 127.7, 126.7, 125.8, 125.3, 125.2, 36.1, 31.3, 29.0, 28.4, 24.9; IR (neat): ν_{max} = 3061, 3024, 2934, 2606, 1668, 1600, 1562, 1495, 1451, 1400, 1338, 1302, 1279, 1263, 1213, 1161, 1134, 1080, 1065, 1029, 906, 843, 767, 729, 697, 648, 621, 600, 561, 543, 483, 427 cm^{-1}; HRMS (ESI): m/z calculated for $C_{20}H_{20}O_2$, $[M - H]^-$ 291.1391, found, 291.1384.



3am

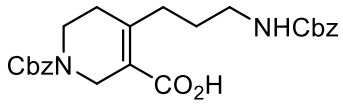
The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3am** (66 mg, 61% yield) as an amorphous powder. R_f = 0.6 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57 (dd, J = 6.8, 2.0 Hz, 1H), 7.32 – 7.24 (m, 2H), 7.22 – 7.17 (m, 1H), 3.68 (s, 3H), 3.08 (t, J = 8.0 Hz, 2H), 2.76 (dd, J = 9.2, 6.4 Hz, 2H), 2.60 (dd, J = 9.2, 6.4 Hz, 2H), 2.44 (t, J = 7.2 Hz, 2H), 1.90 (p, J = 7.6 Hz, 2H); ^{13}C NMR 100 MHz, $CDCl_3$) δ 174.2, 147.9, 138.4, 134.2, 129.1, 127.8, 126.9, 125.4, 51.7, 33.9, 28.4, 28.4, 25.0, 24.8; IR (neat): ν_{max} = 2950, 2605, 1734, 1670, 1601, 1564, 1450, 1436, 1407, 1364, 1337, 1301, 1260, 1197, 1157, 1134, 1048, 1030, 942, 861, 767, 735, 593, 547,

562, 428 cm^{-1}; HRMS (ESI): m/z calculated for $C_{16}H_{18}O_4$, $[M - H]^-$ 273.1132, found, 273.1125.



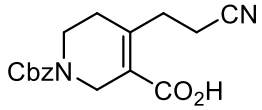
3an

The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3an** (91 mg, 82% yield) as an amorphous powder. R_f = 0.6 (petroleum ether/ethyl acetate 1/1, v/v, containing 0.5% acetic acid e); ^1H NMR (400 MHz, $CDCl_3$) δ 7.41 – 7.13 (m, 10H), 5.17 (s, 2H), 4.22 (s, 2H), 3.52 (t, J = 5.2 Hz, 2H), 2.63 (m, 4H), 2.29 (s, 2H), 1.80 (p, J = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 177.3, 171.1, 155.4/155.2, 154.7/154.0, 141.9, 136.6, 128.6, 128.4, 128.4, 128.4, 128.1, 128.1, 125.9, 121.4/120.8, 67.4, 62.2/l60.5, 43.5, 39.8, 36.0, 34.9, 31.5, 20.9; IR (neat): ν_{max} = 2930, 2915, 2607, 1685, 1629, 1428, 1116, 1085, 937, 774, 748, 691, 523, 435, 409 cm^{-1} ; HRMS (ESI): m/z calculated for $C_{23}H_{25}NO_4$, $[M - H]^-$ 378.1711, found, 378.1704.



3ao

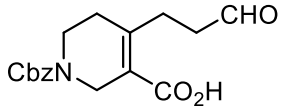
The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3ao** (83 mg, 61% yield) as an amorphous powder. R_f = 0.58 (petroleum ether/ethyl acetate 1/1, v/v, containing 0.5% acetic acid); ^1H NMR (400 MHz, $CDCl_3$) δ 7.34 (m, 10H), 6.02 (s, 0H), 5.39 – 5.22 (m, 1H), 5.15 (s, 2H), 5.08 (s, 1H), 4.19 (s, 2H), 3.51 (m Hz, 2H), 3.16 (m, 2H), 2.54 (m, 2H), 2.24 (m, 2H), 1.67 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.7, 156.7, 136.8, 136.7, 129.0, 128.7, 128.7, 128.6, 128.5, 128.2, 128.2, 128.2, 127.9, 121.2, 67.5/66.7, 44.0, 40.8/39.7, 32.4/31.4, 28.3; IR (neat): ν_{max} = 3342, 3033, 2935, 1688, 1529, 1498, 1416, 1337, 1289, 1239, 1212, 1121, 1081, 1027, 981, 909, 730, 695, 647, 605, 458, 407 cm^{-1}; HRMS (ESI): m/z calculated for $C_{25}H_{28}N_2O_6$, $[M - H]^-$ 451.1875, found, 451.1866.



3ap

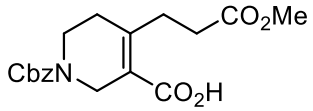
The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3ap** (83 mg, 90% yield) as an amorphous powder. R_f = 0.55 (petroleum ether/ethyl acetate 1/1, v/v, containing 0.5% acetic acid); ^1H NMR (400 MHz, $CDCl_3$) δ 7.51 – 7.30 (m, 5H), 5.17 (s, 2H), 4.25 (s, 2H), 3.59 (m, 2H),

2.87 (m, 2H), 2.59 (t, J = 0.8 Hz, 2H), 2.44 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 169.6, 155.1, 150.6/149.9, 136.6, 128.7, 128.3, 128.2, 124.6/123.9, 119.3, 67.6, 43.7/43.5, 40.1/39.6, 32.7/32.3, 30.9, 16.2; IR (neat): ν_{max} = 2932, 2248, 1694, 1498, 1430, 1363, 1340, 1291, 1244, 1213, 1160, 1121, 1052, 1017, 962, 733, 700, 607, 462, 408 cm^{-1}; HRMS (ESI): m/z calculated for $C_{17}H_{18}N_2O_4$, $[M - H]^-$ 313.1194, found, 313.1186.



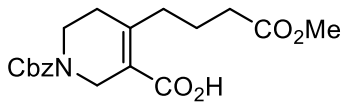
3aq

The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3aq** (61 mg, 66% yield) as an amorphous powder. R_f = 0.33 (petroleum ether/ethyl acetate 1/1, v/v, containing 0.5% acetic acid); ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.79 (s, 1H), 7.48 – 7.29 (m, 5H), 5.16 (s, 2H), 4.20 (s, 2H), 3.55 (t, J = 6.0 Hz, 2H), 2.83 (t, J = 7.6 Hz, 2H), 2.71 – 2.61 (m, 2H), 2.32 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.4, 170.4, 155.4, 152.7, 136.6, 128.7, 128.3, 128.2, 122.9/122.2, 67.5, 43.6/42.4, 40.1/39.7, 31.8, 27.9, 20.9; IR (neat): ν_{max} = 2924, 1699, 1497, 1434, 1363, 1338, 1291, 1243, 1213, 1122, 1027, 981, 764, 698 cm^{-1}; HRMS (ESI): m/z calculated for $C_{17}H_{19}NO_5$, $[M - H]^-$ 316.1190, found, 316.1183.



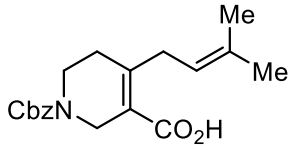
3ar

The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3ar** (110 mg, 90% yield) as an amorphous powder. R_f = 0.58 (petroleum ether/ethyl acetate 1/1, v/v, containing 0.5% acetic acid); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 – 7.29 (m, 5H), 5.16 (s, 2H), 4.21 (s, 2H), 3.67 (s, 3H), 3.55 (q, J = 6.0 Hz, 2H), 2.83 (t, J = 8.0 Hz, 2H), 2.50 (t, J = 8.0 Hz, 2H), 2.32 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.5, 170.3/170.0, 155.4/155.1, 151.9/151.2, 136.6, 128.6, 128.2, 128.1, 122.8/122.3, 67.4, 51.9, 43.5, 40.0/39.7, 32.4, 31.6/31.4, 30.4; IR (neat): ν_{max} = 2951, 1694, 1497, 1433, 1362, 1339, 1288, 1239, 1195, 1116, 1054, 1027, 965, 912, 878, 764, 732, 697, 655, 606, 569, 460, 405 cm^{-1}; HRMS (ESI): m/z calculated for $C_{18}H_{21}NO_6$, $[M - H]^-$ 346.1296, found, 346.1290.



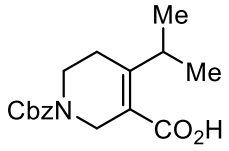
3as

The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3as** (110 mg, 81% yield) as an amorphous powder. R_f = 0.57 (petroleum ether/ethyl acetate 1/1, v/v, containing 0.5% acetic acid); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 – 7.29 (m, 5H), 5.16 (s, 2H), 4.21 (s, 2H), 3.66 (s, 3H), 3.54 (m, 2H), 2.59 (m, 2H), 2.35 (m, 4H), 1.80 (p, J = 7.6 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.8, 170.6/169.9, 155.3/153.8, 153.7, 136.6, 136.5, 128.5, 128.1, 128.1, 128.1, 128.0, 121.3, 67.4/ 67.3, 51.6, 43.6, 42.4, 40.1/39.8, 34.2, 33.7, 31.5/31.3, 26.9, 25.5, 23.3; IR (neat): ν_{max} =2928, 1679, 1422, 1264, 909, 730, 708, 651, 419, 407 cm^{-1}; HRMS (ESI): m/z calculated for $C_{19}H_{23}NO_6$, $[M - H]^-$ 360.1453, found, 360.1455.



3at

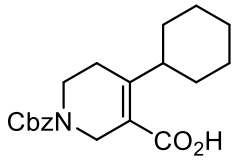
The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3at** (59 mg, 61% yield) as an amorphous powder. R_f = 0.67 (petroleum ether/ethyl acetate 1/1, v/v, containing 0.5% acetic acid); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 – 7.27 (m, 5H), 5.16 (s, 2H), 5.09 (t, J = 7.6 Hz, 1H), 4.21 (s, 2H), 3.52 (t, J = 6.0 Hz, 2H), 3.30 (d, J = 7.2 Hz, 2H), 2.29 (s, 1H), 1.69 (d, J = 16.8 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.1, 155.0/153.8, 136.8, 134.2, 128.8, 128.6, 128.2, 128.1, 120.5, 67.4, 43.6, 40.0, 34.0, 31.0, 25.9, 18.2; IR (neat): ν_{max} = 2925, 1697, 1497, 1431, 1362, 1337, 1291, 1237, 1209, 1116, 1046, 1018, 990, 916, 875, 764, 735, 697, 658, 607, 566, 528, 458, 407 cm^{-1}; HRMS (ESI): m/z calculated for $C_{19}H_{23}NO_4$, $[M - H]^-$ 328.1554, found, 328.1557.



3au

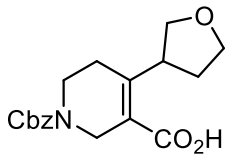
The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3au** (75 mg, 80% yield) as an amorphous powder. R_f = 0.6 (petroleum ether/ethyl acetate 1/1, v/v, containing 0.5% acetic acid); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 – 7.30 (m, 5H), 5.17 (s, 2H), 4.21 (s, 2H), 3.81 (m, 1H), 3.52 (t, J = 5.6 Hz, 2H), 2.28 (s, 2H), 1.02 (d, J = 6.8 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 171.4, 158.7, 155.3, 136.7, 128.5, 128.1, 128.0, 119.6, 119.6, 67.2, 43.8/ 43.5, 40.2/39.8,

30.4/29.7, 24.7, 20.4; IR (neat): ν_{max} = 2961, 2871, 1677, 1497, 1429, 1361, 1341, 1292, 1240, 1198, 1116, 1028, 978, 911, 890, 877, 764, 739, 716, 697, 645, 610, 542, 462 cm^{-1}; HRMS (ESI): m/z calculated for $C_{17}H_{21}NO_4$, $[M - H]^-$ 302.1398, found, 302.1389.



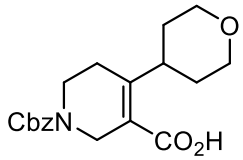
3av

The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3av** (48 mg, 48% yield) as an amorphous powder. R_f = 0.62 (petroleum ether/ethyl acetate 1/1, v/v, containing 0.5% acetic acid); ^1H NMR (400 MHz, $CDCl_3$) δ 7.41 – 7.29 (m, 5H), 5.16 (s, 2H), 4.21 (t, J = 2.4 Hz, 2H), 3.50 (t, J = 5.6 Hz, 2H), 3.47 – 3.35 (m, 1H), 2.29 (s, 2H), 1.73 (dd, J = 23.6, 12.9 Hz, 3H), 1.59 (d, J = 11.6 Hz, 2H), 1.43 – 1.18 (m, 5H); ^{13}C NMR 100 MHz, $CDCl_3$) δ 170.8, 155.3, 136.7, 128.7, 128.5, 128.1, 128.0, 67.2, 43.6, 41.5/ 41.5, 40.3, 39.9/39.9, 30.5, 29.7, 26.2/26.1, 22.7, 14.1; IR (neat): ν_{max} = 2924, 2851, 1681, 1497, 1432, 1362, 1339, 1292, 1239, 1208, 1136, 1113, 1028, 999, 980, 908, 889, 847, 802, 764, 729, 696, 647, 606, 543, 460 cm^{-1}; HRMS (ESI): m/z calculated for $C_{20}H_{25}NO_4$, $[M - H]^-$ 342.1711, found, 342.1708.



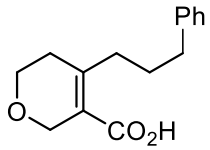
3aw

The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3aw** (50 mg, 53% yield) as an amorphous powder. R_f = 0.59 (petroleum ether/ethyl acetate 1/1, v/v, containing 0.5% acetic acid); ^1H NMR (400 MHz, $CDCl_3$) δ 7.35 (m, 5H), 5.17 (s, 2H), 4.28 (d, J = 16.4 Hz, 2H), 4.17 (d, J = 18.0 Hz, 1H), 4.03 – 3.93 (m, 1H), 3.89 (m, 1H), 3.75 (m, 1H), 3.67 – 3.56 (m, 2H), 3.45 (mz, 1H), 2.33 (s, 2H), 2.18 – 2.10 (m, 1H), 1.87 – 1.74 (m, 1H); ^{13}C NMR 100 MHz, $CDCl_3$) δ 169.8, 155.3, 150.8, 136.5, 128.5, 128.5, 128.1, 128.1, 127.7, 123.4, 70.6, 68.2, 67.3, 43.9, 40.4/ 39.7, 29.7/29.6, 27.2/26.7, 25.6; IR (neat): ν_{max} = 2930, 2915, 2607, 1685, 1629, 1428, 1116, 1085, 937, 774, 748, 691, 523, 435, 409 cm^{-1}; HRMS (ESI): m/z calculated for $C_{18}H_{21}NO_5$, $[M - H]^-$ 330.1347, found, 330.1339.



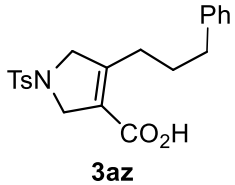
3ax

The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3ax** (110 mg, 69% yield) as an amorphous powder. R_f = 0.6 (petroleum ether/ethyl acetate 1/1, v/v, containing 0.5% acetic acid); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 (m, 5H), 5.17 (s, 2H), 4.23 (s, 2H), 4.01 (dd, J = 11.2 Hz, 3.6 Hz, 2H), 3.71 (t, J = 12.0 Hz, 1H), 3.58 – 3.38 (m, 4H), 2.30 (s, 2H), 1.68 (m, 2H), 1.48 (d, J = 12.0 Hz, 2H); ^{13}C NMR 100 MHz, $CDCl_3$) δ 170.5, 155.7/155.2, 136.6, 128.5, 128.1, 128.1, 121.6, 67.8, 67.3, 43.8, 40.2/39.8, 38.8, 30.0, 26.5; IR (neat): ν_{max} = 2953, 2849, 1698, 1497, 1432, 1387, 1362, 1341, 1293, 1239, 1211, 1126, 1080, 1014, 983, 913, 878, 820, 764, 733, 698, 662, 606, 460, 413 cm^{-1}; HRMS (ESI): m/z calculated for $C_{19}H_{23}NO_5$, $[M - H]^-$ 344.1503, found, 344.1500.



3ay

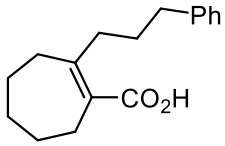
The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3ay** (55 mg, 66% yield) as an amorphous powder. R_f = 0.75(petroleum ether/ethyl acetate 1/1, v/v, containing 0.5% acetic acid); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 – 7.23 (m, 2H), 7.19 (d, J = 7.6 Hz, 3H), 4.32 (s, 2H), 3.74 (t, J = 5.6 Hz, 2H), 2.65 (m, 4H), 2.29 (s, 2H), 1.82 (p, J = 8.0 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 170.8, 154.3, 142.1, 128.5, 128.5, 126.0, 122.6, 65.6, 63.9, 36.1, 34.8, 31.4, 30.0; IR (neat): ν_{max} = 2925, 2854, 2249, 1683, 1636, 1496, 1453, 1417, 1268, 1131, 1086, 1023, 975, 906, 866, 727, 699, 648, 581, 544, 492, 408 cm^{-1}; HRMS (ESI): m/z calculated for $C_{15}H_{18}O_3$, $[M - H]^-$ 245.1183, found, 245.1175.



3az

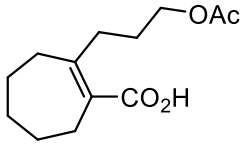
The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3az** (78 mg, 70% yield) as an amorphous powder. R_f = 0.62 (Dichloromethane/Methanol = 10/1, v/v, containing 0.5% acetic acid); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 (d, J = 8.4 Hz, 2H), 7.32 (d, J = 8.0 Hz, 2H), 7.26 (d, J = 7.6 Hz, 2H), 7.15 (m, 3H), 4.29 (t, J = 4.4 Hz, 2H), 4.23 (t, J = 4.4 Hz, 2H), 2.57 (t, J = 8.0 Hz, 4H), 2.41 (s, 3H), 1.77 – 1.65 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.9, 155.7, 144.0, 141.2, 133.5, 130.0, 128.3, 127.5, 126.1, 122.4, 58.6, 55.0, 35.7, 29.3,

27.5, 21.6; IR (neat): ν_{max} = 3026, 2925, 2858, 1687, 1653, 1598, 1495, 1453, 1345, 1289, 1163, 1100, 1076, 1017, 910, 815, 748, 700, 666, 601, 586, 547, 493, 408 cm^{-1}; HRMS (ESI): m/z calculated for $C_{21}H_{23}NO_4S$, $[M - H]^-$ 384.1275, found, 384.1269.



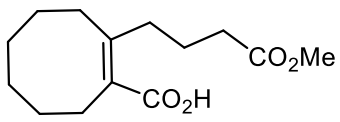
3ba

The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3ba** (110 mg, 86% yield) as an amorphous powder. R_f = 0.6 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid); ^1H NMR (400 MHz, $CDCl_3$) δ 7.26 (t, J = 3.6 Hz, 2H), 7.18 (m, 3H), 2.66 (t, J = 7.8 Hz, 2H), 2.55 – 2.46 (m, 4H), 2.35 – 2.27 (m, 2H), 1.86 – 1.72 (m, 4H), 1.55 – 1.47 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 174.9, 158.6, 142.5, 130.1, 128.6, 128.4, 125.8, 37.1, 36.3, 36.2, 32.5, 30.0, 29.7, 26.6, 25.8; IR (neat): ν_{max} = 2923, 2852, 2620, 1679, 1611, 1449, 1404, 1281, 1259, 1242, 1136, 1043, 988, 939, 744, 689, 561, 513, 478, 407 cm^{-1}; HRMS (ESI): m/z calculated for $C_{17}H_{22}O_2$, $[M - H]^-$ 257.1547, found, 257.1540.



3bb

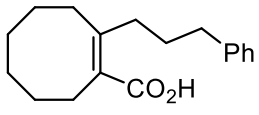
The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3bb** (83 mg, 80% yield) as an amorphous powder. R_f = 0.68 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid); ^1H NMR (400 MHz, $CDCl_3$) δ 4.07 (t, J = 6.8 Hz, 2H), 2.49 (t, J = 7.6 Hz, 4H), 2.39 – 2.28 (m, 2H), 2.04 (s, 3H), 1.87 – 1.71 (m, 4H), 1.58 – 1.46 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 175.3, 171.4, 157.5, 130.8, 64.4, 36.2, 33.6, 32.4, 29.9, 27.0, 26.4, 25.7, 21.1; IR (neat): ν_{max} = 2923, 2852, 2620, 1679, 1611, 1449, 1404, 1281, 1259, 1242, 1136, 1043, 988, 939, 744, 689, 561, 513, 478, 407 cm^{-1}; HRMS (ESI): m/z calculated for $C_{13}H_{20}O_4$, $[M - H]^-$ 239.1289, found, 239.1282.



3bc

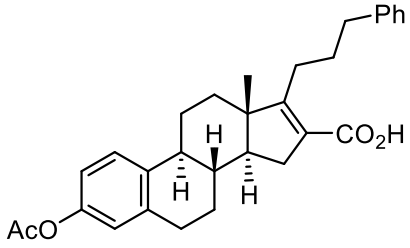
The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3bc** (110 mg, 83% yield) as an amorphous powder. R_f = 0.6 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid); ^1H NMR (400 MHz, $CDCl_3$) δ 3.67 (s, 3H), 2.54 – 2.41 (m, 4H), 2.34 (m, 4H), 1.82

(p, J = 7.6 Hz, 2H), 1.71 – 1.59 (m, 4H), 1.46 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 174.3, 174.1, 155.6, 127.4, 51.6, 34.2, 34.1, 33.4, 29.9, 29.2, 28.4, 26.8, 26.5, 23.9; IR (neat): ν_{max} = 2922, 2852, 1738, 1677, 1617, 1437, 1406, 1371, 1295, 1248, 1197, 1176, 1144, 1102, 945, 892, 765, 418, 407 cm^{-1}; HRMS (ESI): m/z calculated for $C_{14}H_{22}O_4$, $[M - H]^-$ 253.1445, found, 253.1436.



3bd

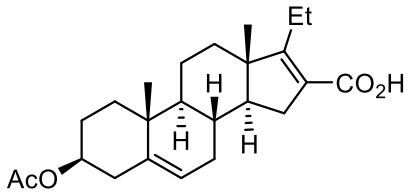
The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3bd** (97 mg, 83% yield) as an amorphous powder. R_f = 0.77 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 – 7.12 (m, 5H), 2.78 – 2.60 (m, 2H), 2.47 (dd, J = 15.2, 10.0 Hz, 4H), 2.40 – 2.25 (m, 2H), 1.83 (p, J = 8.0 Hz, 2H), 1.64 (d, J = 23.2 Hz, 4H), 1.46 (s, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 174.4, 156.7, 142.5, 128.6, 128.4, 126.7, 125.8, 36.4, 35.0, 33.8, 30.4, 30.0, 29.2, 28.4, 26.9, 26.5; IR (neat): ν_{max} =2928, 1679, 1422, 1264, 909, 730, 708, 651, 419, 407 cm^{-1}; HRMS (ESI): m/z calculated for $C_{18}H_{24}O_2$, $[M - H]^-$ 271.1704, found, 271.1695.



3be

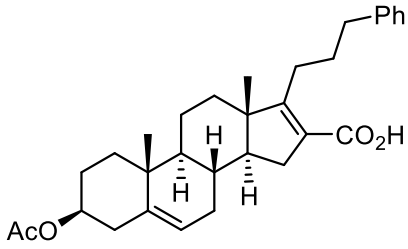
The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3be** (70 mg, 64% yield) as an amorphous powder. R_f = 0.68 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44 – 7.38 (m, 3H), 7.33 (m, 3H), 7.01 – 6.90 (m, 2H), 3.07 – 2.91 (m, 3H), 2.84 (m, 2H), 2.71 (d, 1H), 2.53 – 2.47 (m, 1H), 2.41 (s, 6H), 2.13 – 1.89 (m, 4H), 1.72 (m, 4H), 1.58 (m, 1H), 0.97 (s, 3H); ^{13}C NMR 100 MHz, $CDCl_3$) δ 172.8, 171.8, 169.9, 148.5, 142.2, 138.3, 137.9, 128.5, 128.3, 126.7, 126.0, 125.8, 121.6, 118.6, 53.8, 50.5, 44.2, 37.0, 36.5, 34.0, 32.0, 30.9, 29.4, 27.4, 27.2, 26.1, 21.1, 15.7; IR (neat): ν_{max} = 3025, 2928, 2853, 1761, 1667, 1605, 1493, 1452, 1418, 1368, 1297, 1267, 1246, 1203, 1186, 1149, 1089, 1013, 976, 940, 919, 899, 822, 797, 780, 733, 698, 650, 613, 595, 564, 534, 513, 494, 448, 406 cm^{-}

[1]; HRMS (ESI): m/z calculated for $C_{30}H_{34}O_4$, $[M - H]^-$ 457.2384, found, 457.2376; $[\alpha]^{25}_D$ = + 41.06 (c 0.50, $CHCl_3$).



3bf

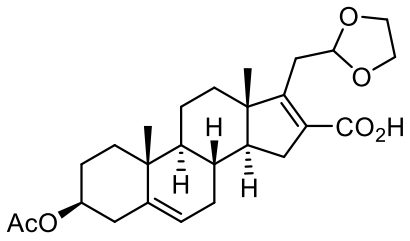
The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3bf** (49 mg, 53% yield) as an amorphous powder. R_f = 0.62 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid); ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.40 (d, J = 5.2 Hz, 1H), 4.61 (m, 1H), 2.79 – 2.66 (m, 1H), 2.47 (dd, J = 14.8, 6.8 Hz, 1H), 2.34 (d, J = 6.0 Hz, 2H), 2.25 (m, 1H), 2.15 (dd, J = 14.8, 11.6 Hz, 1H), 2.03 (s, 3H), 1.92 – 1.78 (m, 3H), 1.74 – 1.52 (m, 5H), 1.41 (m, 3H), 1.16 (dd, J = 14.6, 4.4 Hz, 1H), 1.12 – 1.03 (m, 7H), 0.85 (s, 3H); ^{13}C NMR 100 MHz, $CDCl_3$) δ 174.5, 171.7, 170.6, 139.9, 126.0, 122.4, 73.8, 54.6, 50.3, 50.2, 38.1, 36.9, 36.8, 33.9, 32.2, 31.4, 30.5, 27.7, 21.4, 20.6, 20.4, 19.2, 15.5, 13.7; IR (neat): ν_{max} = 2936, 1734, 1672, 1611, 1373, 1245, 1032, 406; HRMS (ESI): m/z calculated for $C_{24}H_3O_4$, $[M - H]^-$ 385.2384, found, 385.2376; $[\alpha]^{25}_D$ = –78.62 (c 0.46, $CHCl_3$).



3bg

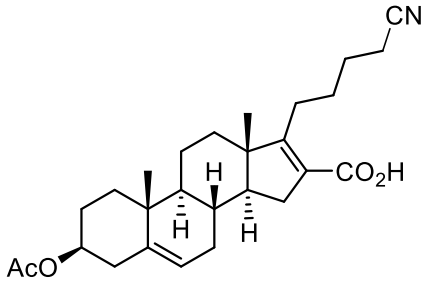
The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3bg** (69 mg, 64% yield) as an amorphous powder. R_f = 0.76 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 – 7.23 (m, 2H), 7.23 – 7.12 (m, 3H), 5.40 (d, J = 4.8 Hz, 1H), 4.61 (m, 1H), 2.73 (m, 3H), 2.48 (dd, J = 14.8, 6.8 Hz, 1H), 2.37 – 2.30 (m, 2H), 2.26 (m, 1H), 2.16 (dd, J = 14.8, 11.6 Hz, 1H), 2.03 (s, 3H), 1.86 (m, 3H), 1.80 – 1.70 (m, 2H), 1.68 – 1.59 (m, 3H), 1.55 (m, 1H), 1.39 (m, 2H), 1.26 (s, 2H), 1.20 – 1.08 (m, 1H), 1.05 (s, 4H), 0.84 (s, 3H); ^{13}C NMR 100 MHz, $CDCl_3$) δ 172.8, 171.5, 170.7, 142.3, 140.0, 128.6, 128.4, 126.8, 125.9, 122.5, 74.0, 54.7, 50.4, 50.3, 38.2, 37.0, 36.9, 36.6, 34.1, 32.4, 31.5, 30.9, 30.6, 27.9, 27.3, 21.6, 20.7, 19.4, 15.7; IR (neat): ν_{max} = 2934, 2852, 1732, 1671, 1609, 1496, 1453, 1436, 1373, 1244, 1091, 1032, 955, 907,

815, 933, 699, 948, 912, 514 cm^{-1}; HRMS (ESI): m/z calculated for $C_{31}H_{40}O_4$, $[M - H]^-$ 475.2854, found, 475.2846; $[\alpha]^{25}_D = -27.5$ (c 0.76, $CHCl_3$).



3bh

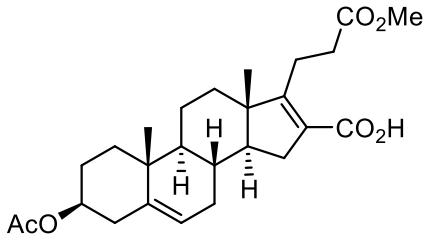
The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3bh** (51 mg, 51% yield) as an amorphous powder. R_f = 0.42 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid); ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.40 (d, J = 4.8 Hz, 1H), 4.66 – 4.54 (m, 1H), 2.56 – 2.44 (m, 2H), 2.38 – 2.25 (m, 2H), 2.18 – 2.08 (m, 1H), 2.03 (s, 4H), 1.92 – 1.84 (m, 2H), 1.83 (s, 1H), 1.76 – 1.47 (m, 7H), 1.37 – 1.23 (m, 3H), 1.21 – 0.98 (m, 6H), 0.83 (s, 3H), 0.07 (s, 9H); ^{13}C NMR 100 MHz, $CDCl_3$) δ 174.4, 172.7, 170.7, 139.9, 122.6, 122.1, 74.0, 54.3, 50.4, 50.3, 38.2, 37.0, 36.9, 34.2, 31.9, 31.5, 30.7, 27.9, 21.6, 20.8, 20.2, 19.4, 15.5, 0.4; IR (neat): ν_{max} = 2941, 1732, 1674, 1611, 1373, 1245, 1125, 1033, 832, 733, 412 cm^{-1}; HRMS (ESI): m/z calculated for $C_{26}H_{36}O_6$, $[M - H]^-$ 443.2439, found, 443.2430; $[\alpha]^{25}_D = -39.96$ (c 0.89, $CHCl_3$).



3bi

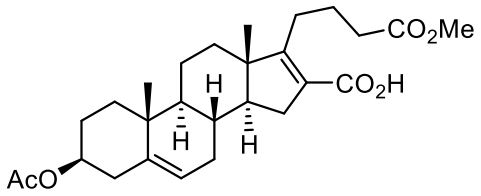
The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3bi** (54 mg, 53% yield) as an amorphous powder. R_f = 0.58 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid); ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.38 (d, J = 5.2 Hz, 1H), 4.59 (m, 1H), 2.74 (dt, J = 10.9, 7.5 Hz, 1H), 2.46 (dd, J = 14.8, 6.8 Hz, 1H), 2.41 – 2.24 (m, 5H), 2.24 – 2.15 (m, 1H), 2.15 – 2.09 (m, 1H), 2.08 (s, 2H), 2.02 (s, 4H), 1.90 – 1.76 (m, 3H), 1.76 – 1.68 (m, 3H), 1.68 – 1.53 (m, 7H), 1.53 – 1.41 (m, 2H), 1.35 (m, 2H), 1.29 – 1.04 (m, 3H), 1.04 (s, 3H), 0.84 (s, 3H); ^{13}C NMR 100 MHz, $CDCl_3$) δ 177.5, 171.9, 170.7, 139.9, 127.5, 122.3, 119.7, 73.9, 54.6, 50.2, 38.1, 36.9, 36.8, 34.1, 32.3, 31.4, 30.5, 28.1, 27.8, 26.3, 25.6, 21.5, 20.6, 19.3, 16.9, 15.6; IR (neat): ν_{max} = 2940, 2853, 2250, 1729, 1669, 1609, 1434, 1373, 1304, 1244, 1135, 1091, 1031, 971, 954, 909, 834, 815, 728,

687, 647, 613, 545, 516, 455 cm^{-1}; HRMS (ESI): m/z calculated for $C_{27}H_{37}NO_4$, $[M - H]^-$ 438.2650, found, 438.2650; $[\alpha]^{25}_D = -73.77$ (c 1.18, $CHCl_3$).



3bj

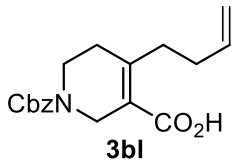
The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3bj** (69 mg, 68% yield) as an amorphous powder. R_f = 0.56 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid); ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.39 (d, J = 5.0 Hz, 1H), 4.66 – 4.55 (m, 1H), 3.67 (s, 3H), 2.75 (m, 1H), 2.47 (dd, J = 14.8, 6.8 Hz, 1H), 2.41 – 2.29 (m, 4H), 2.25 (dd, J = 12.4, 7.6 Hz, 1H), 2.14 (dd, J = 14.8, 11.6 Hz, 1H), 2.03 (s, 4H), 1.91 – 1.75 (m, 5H), 1.66 (t, J = 10.0 Hz, 3H), 1.61 – 1.52 (m, 2H), 1.52 – 1.43 (m, 1H), 1.43 – 1.31 (m, 2H), 1.13 (m, 1H), 1.05 (s, 3H), 0.85 (s, 3H); ^{13}C NMR 100 MHz, $CDCl_3$) δ 171.7, 171.7, 170.6, 139.9, 127.4, 122.3, 122.3, 73.8, 54.5, 51.5, 50.2, 38.1, 36.9, 36.8, 34.1, 33.9, 32.3, 31.4, 30.4, 27.7, 26.5, 24.2, 21.4, 20.5, 19.2, 15.5; IR (neat): ν_{max} = 2941, 2854, 1731, 1694, 1614, 1418, 1373, 1242, 1178, 1094, 1031, 971, 937, 855, 834, 816, 714, 668, 612, 544, 514, 466, 412 cm^{-1}; HRMS (ESI): m/z calculated for $C_{26}H_{36}O_6$, $[M - H]-$ 443.2439, found, 443.2432; $[\alpha]^{25}_D = -65.62$ (c 0.57, $CHCl_3$).



3bk

The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3bk** (64 mg, 63% yield) as an amorphous powder. R_f = 0.56 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid); ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.39 (d, J = 5.0 Hz, 1H), 4.70 – 4.49 (m, 1H), 3.66 (s, 4H), 2.74 (dt, J = 11.2, 8.0 Hz, 1H), 2.46 (dd, J = 14.8, 6.8 Hz, 1H), 2.40 – 2.22 (m, 6H), 2.19 – 2.08 (m, 1H), 2.03 (s, 3H), 1.82 (m, 4H), 1.71 – 1.54 (m, 6H), 1.43 (dd, J = 12.0, 4.4 Hz, 1H), 1.40 – 1.30 (m, 2H), 1.14 (m, 1H), 1.05 (s, 3H), 0.85 (s, 3H); ^{13}C NMR 100 MHz, $CDCl_3$) δ 174.1, 171.8, 170.7, 140.0, 127.5, 122.4, 74.0, 54.6, 51.6, 50.4, 50.3, 38.2, 37.0, 36.9, 34.3, 34.1, 32.4, 31.5, 30.6, 27.8, 26.7, 24.4, 21.6, 20.7, 19.4, 15.6; IR (neat): ν_{max} = 2946, 2852,

2255, 1731, 1669, 1610, 1435, 1372, 1302, 1241, 1199, 1169, 1091, 1031, 972, 955, 914, 882, 834, 815, 731, 687, 647, 612, 544, 517, 454 cm^{-1}; HRMS (ESI): m/z calculated for $C_{26}H_{36}O_6$, $[M - H]^-$ 457.2596, found, 457.2587; $[\alpha]^{25}_D = -72.32$ (c 0.46, $CHCl_3$).



3bl

The reaction was conducted according to general experimental procedure noted above, which proceeded at room temperature for 12 hours to give compound **3bl** (79 mg, 85% yield) as an amorphous powder. R_f = 0.67 (petroleum ether/ethyl acetate 2/1, v/v, containing 0.5% acetic acid); ^1H NMR (400 MHz, $CDCl_3$) δ 7.34 (m, 5H), 5.83 (m, 1H), 5.17 (s, 2H), 5.10 – 4.91 (m, 2H), 4.22 (s, 2H), 3.53 (m, 2H), 2.66 (t, *J* = 8.0 Hz, 2H), 2.40 – 2.15 (m, 4H); ^{13}C NMR (150 MHz, $CDCl_3$) δ 170.6, 155.4/154.5, 137.9, 136.8, 128.7, 128.2, 128.1, 121.6, 115.3, 67.4, 43.8/43.6, 40.2/39.9, 32.6, 32.0/31.8, 18.1; IR (neat): ν_{max} = 3037, 2917, 2865, 1699, 1640, 1498, 1434, 1363, 1338, 1292, 1243, 1292, 1212, 1126, 995, 913, 1736, 698, 606, 407 cm^{-1}; HRMS (ESI): m/z calculated for $C_{18}H_{21}NO_4$, $[M - H]^-$ 314.1389, found, 314.1385.

7. References

[1] Karaki, F.; Ohgane, K.; Fukuda, H.; Nakamura, M.; Dodo, K.; Hashimoto, Y. Structure-activity relationship study of non-steroidal NPC1L1 ligands identified through cell-based assay using pharmacological chaperone effect as a readout. *Bioorg. Med. Chem.* **2014**, *22*, 3587– 3609.

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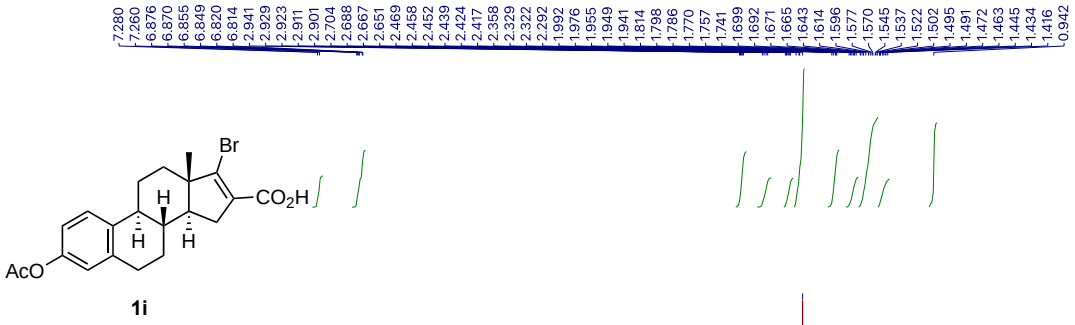
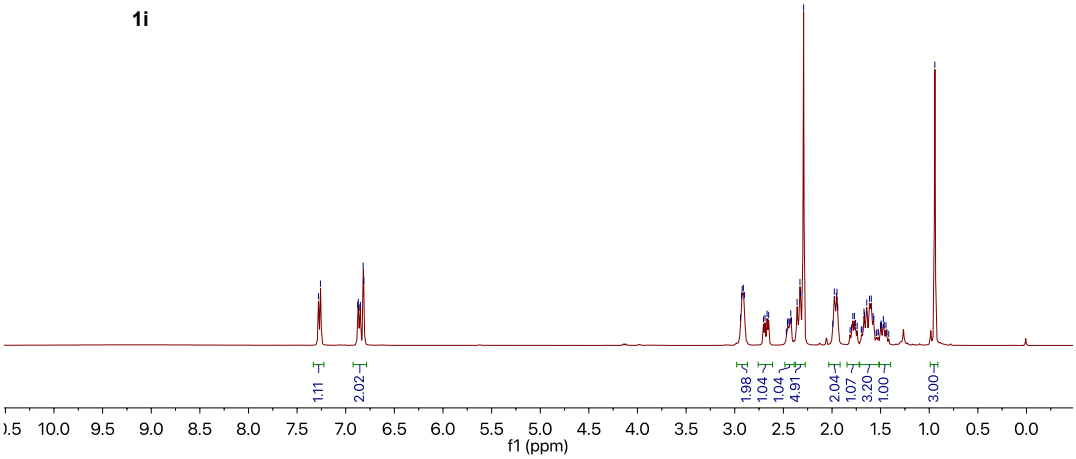
[3] Ren, H.; Li, Z.; Knochel, P. Chemoselective C(sp3)–H Bond Activation for the Preparation of Condensed N-Heterocycles. *Chem.─Asian J.* **2007**, *2*, 416 – 433.

[4] Shirakawa, E.; Imazaki, Y.; Hayashi, T. Ruthenium-catalyzed transformation of alkenyl triflates to alkenyl halides. *Chem. Commun.* **2009**, 5088 – 5090.

8. NMR Spectra



1i



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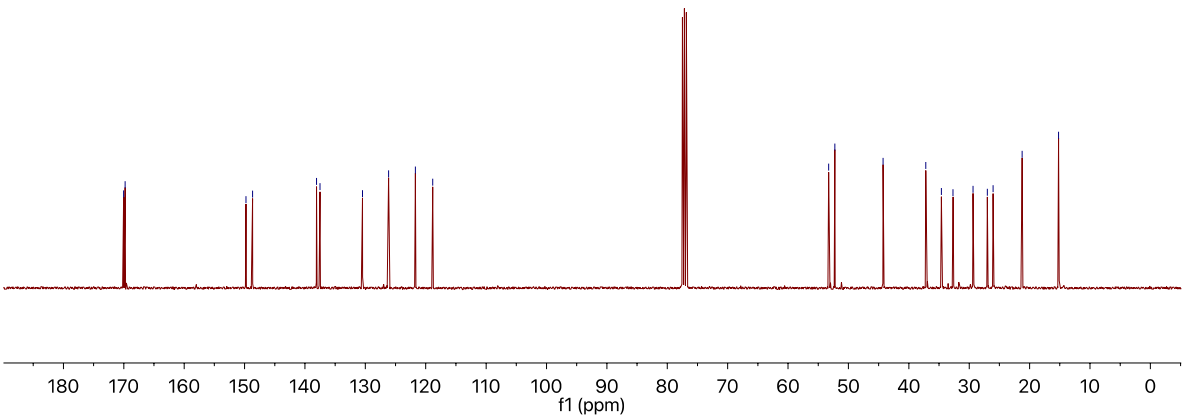

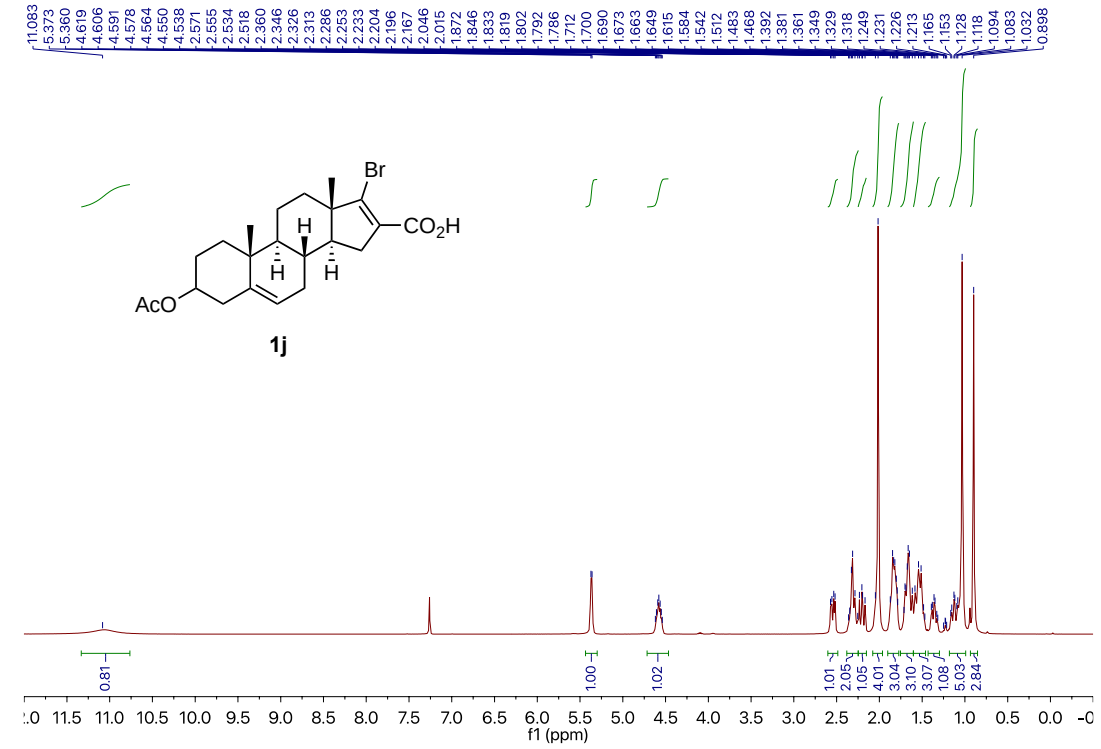
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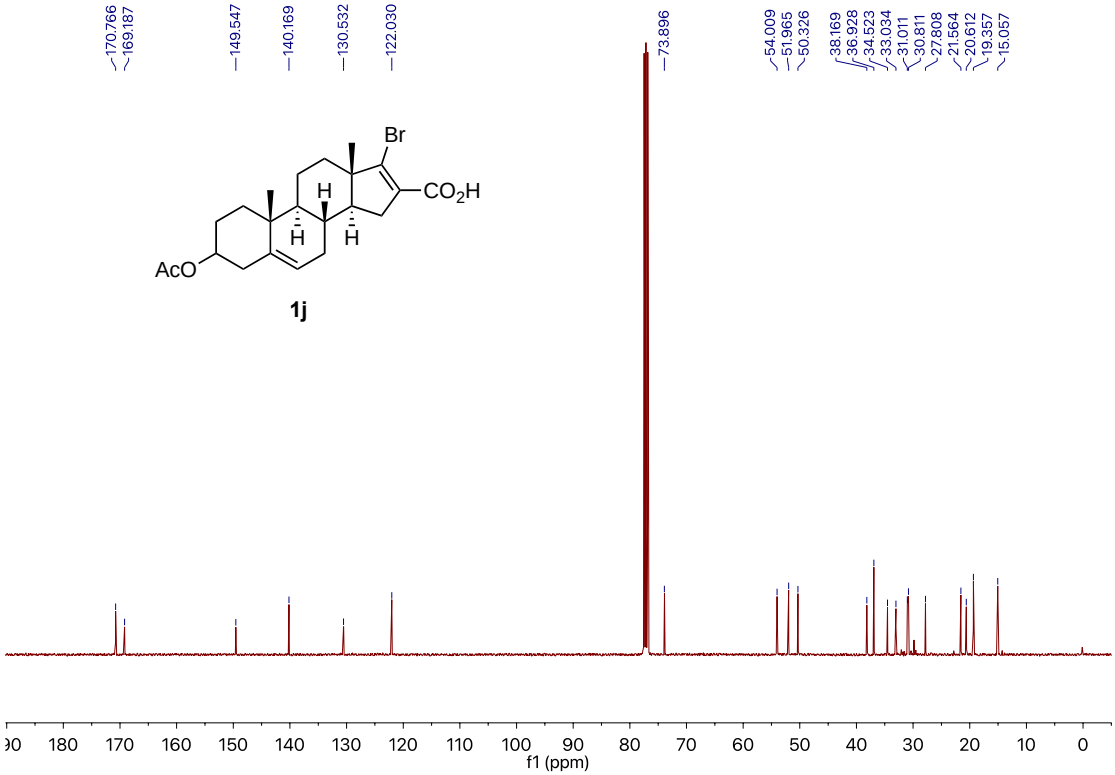
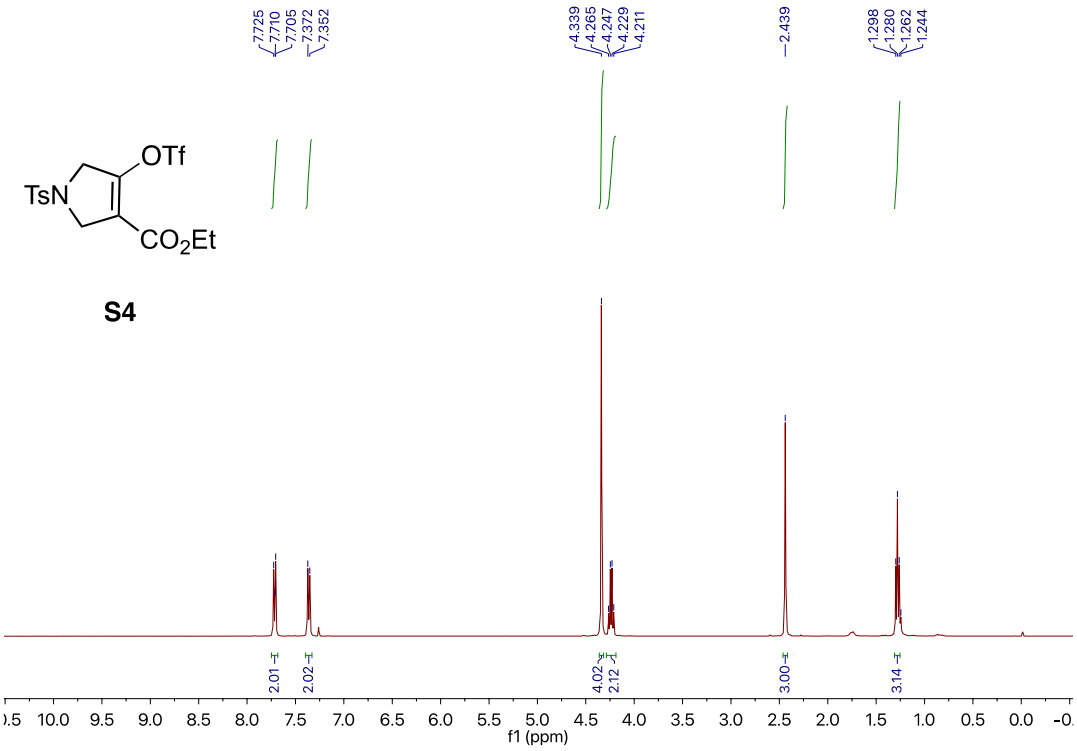
H
H
H
AcO
1j
f1 (ppm)


Br
CO2H
H
H
H
AcO
1j
f1 (ppm)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1.262
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CO2Et
2.01
2.02
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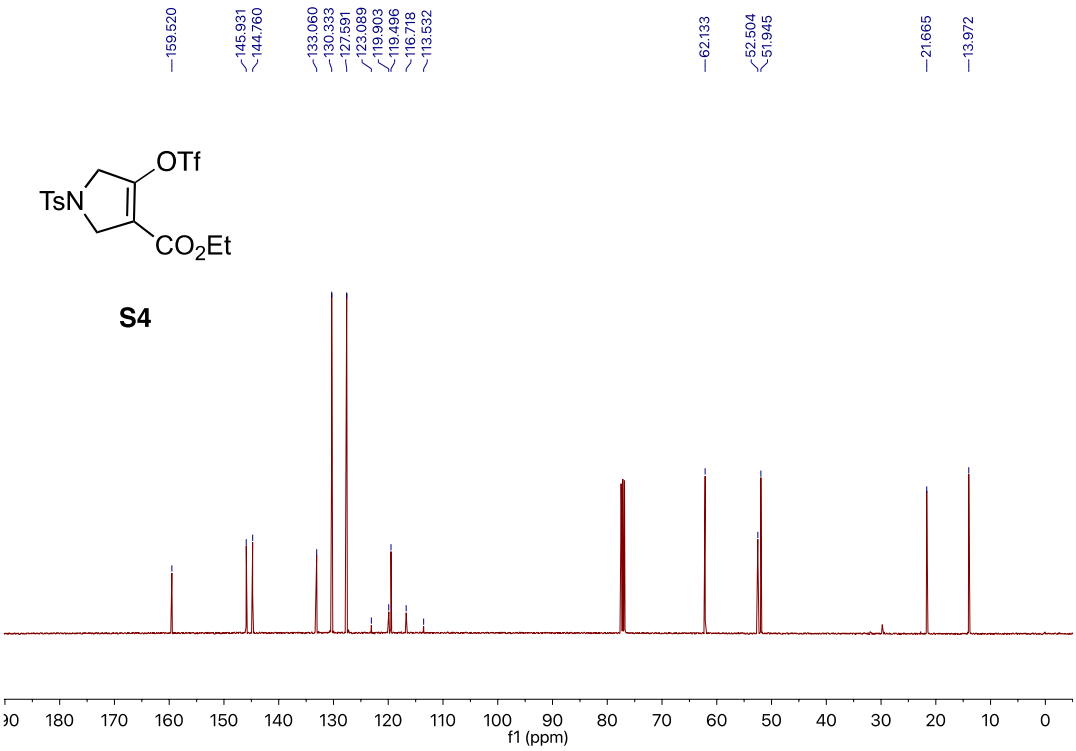
3.00
3.14
f1 (ppm)

S4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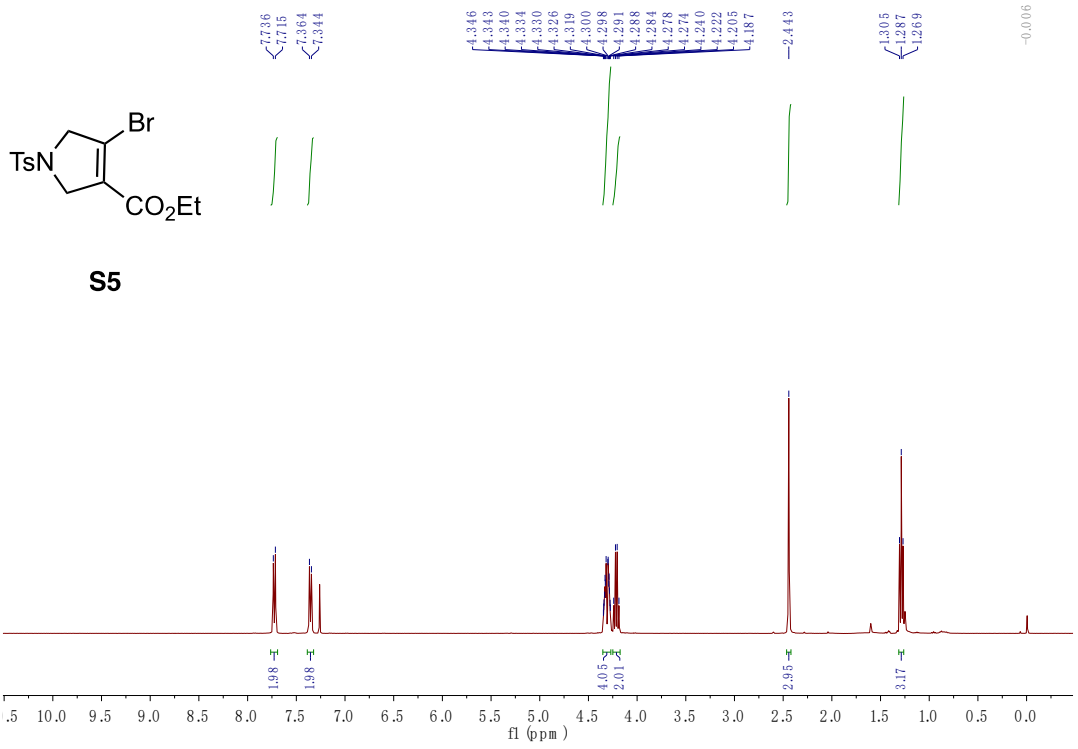
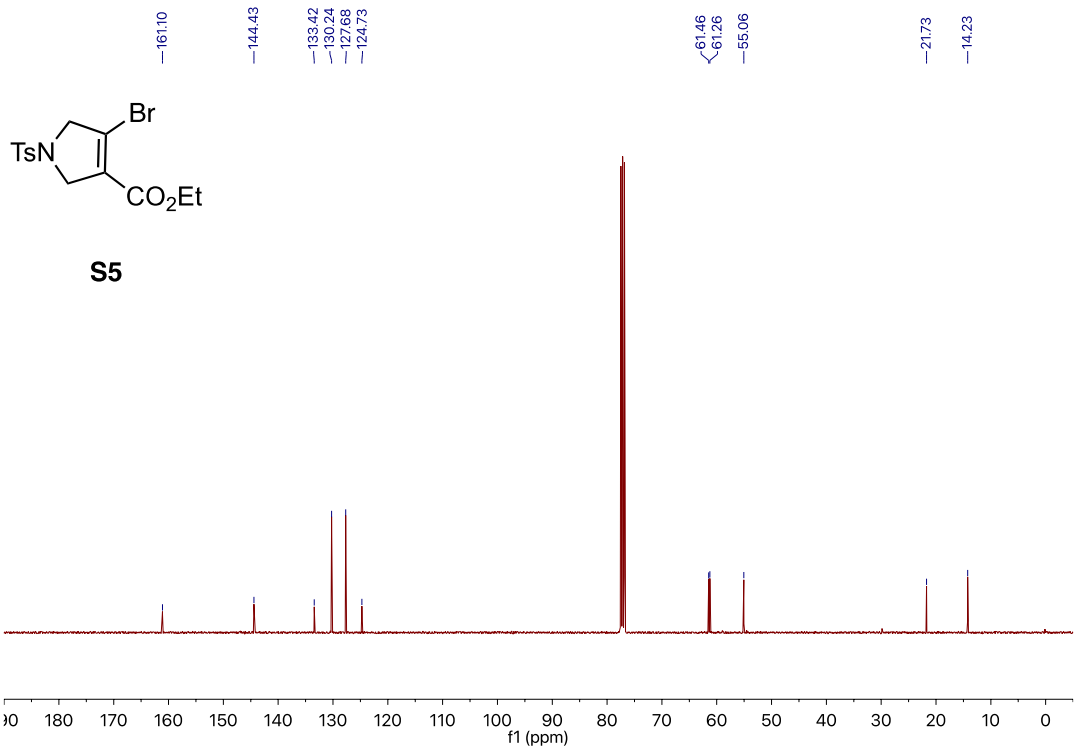
62.133
52.504
51.945
21.665
13.972
OTf
TsN
CO2Et
f1 (ppm)

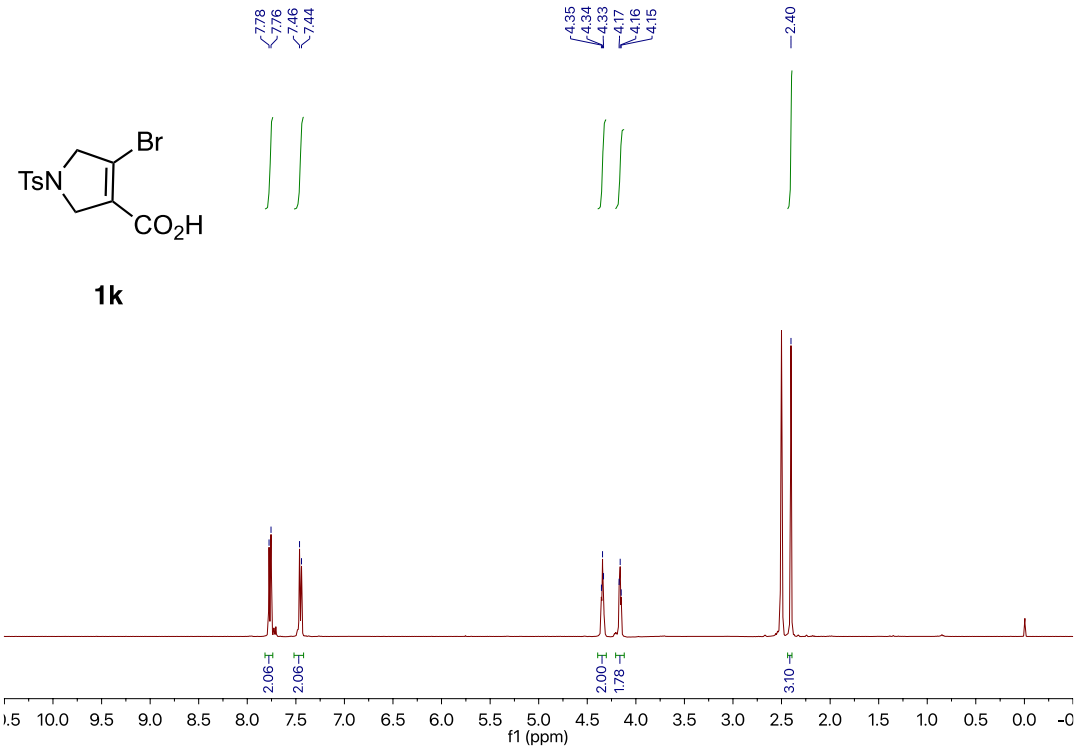
S4


Br
TsN
CO2Et
7.736
7.715
7.364
7.344
4.346
4.343
4.340
4.334
4.330
4.326
4.319
4.300
4.298
4.291
4.288
4.284
4.278
4.274
4.240
4.222
4.205
4.187
2.443
1.305
1.287
1.269
-0.006
1.98
1.98
4.05
2.01
2.95
3.17
f1 (ppm)

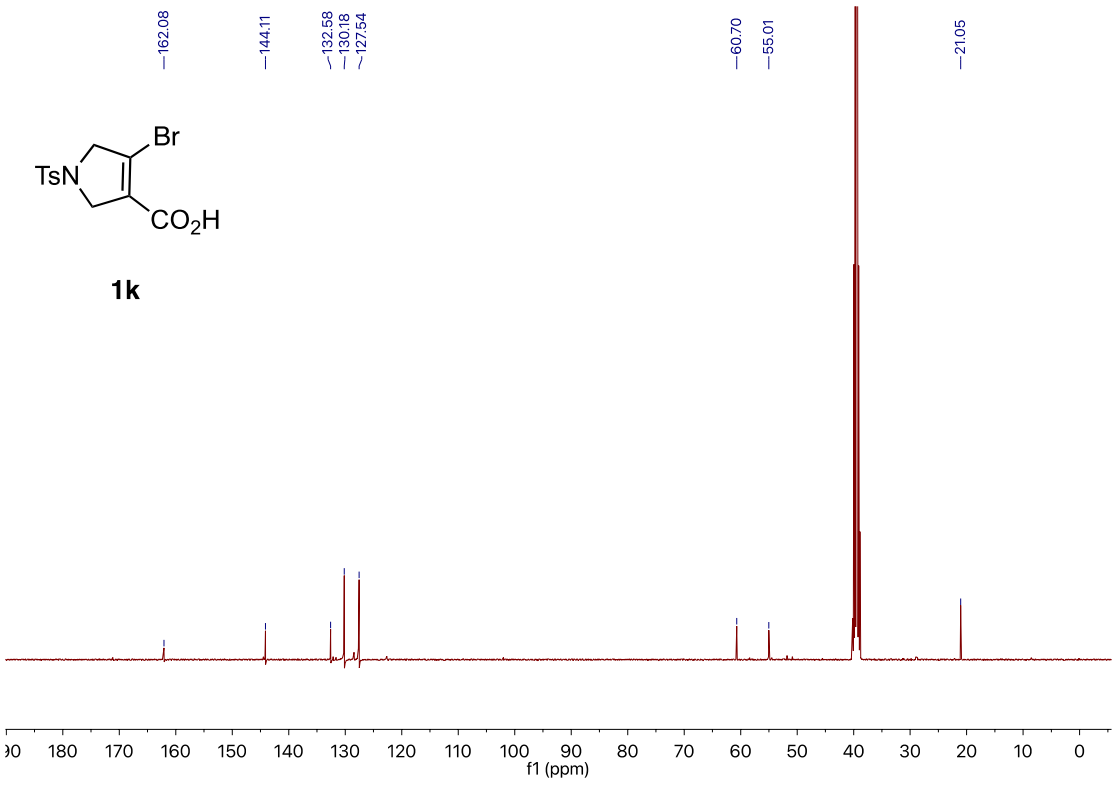
S5


Br
TsN
CO2Et
161.10
144.43
133.42
130.24
127.68
124.73
61.46
61.26
55.06
21.73
14.23
f1 (ppm)

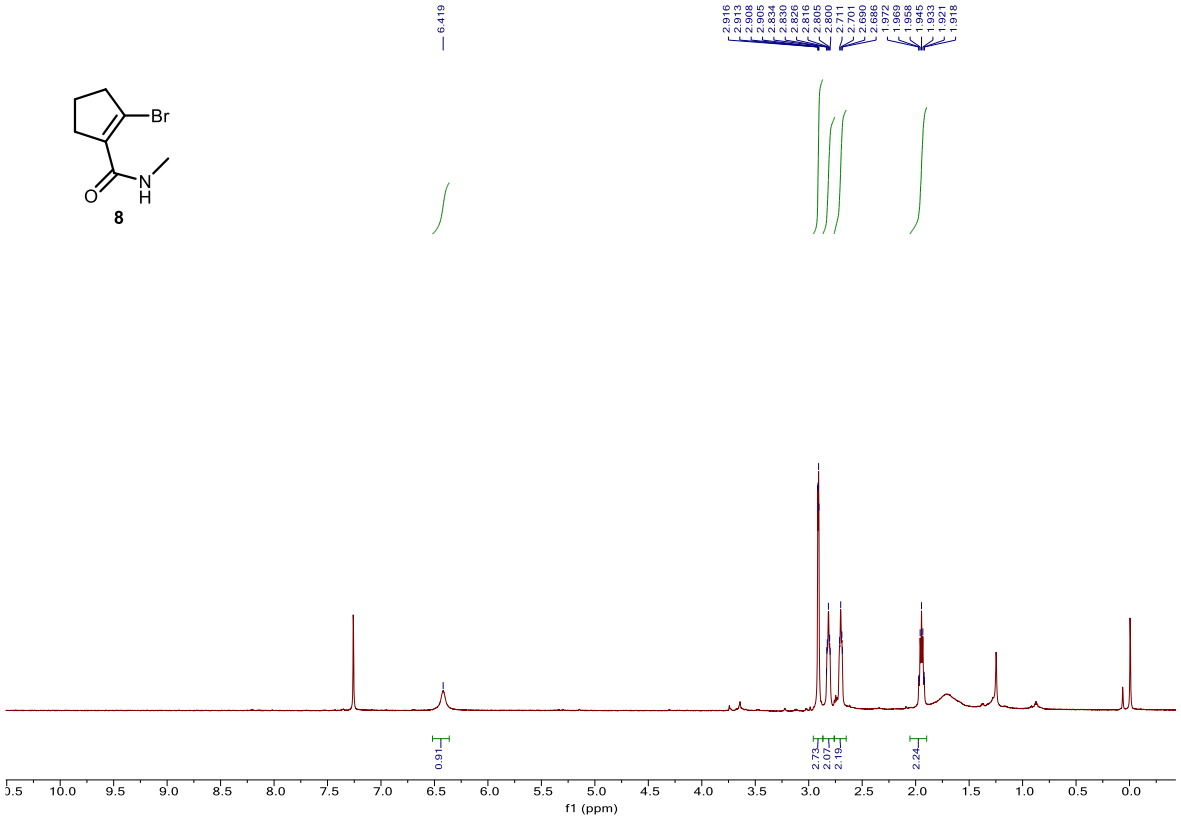
S5

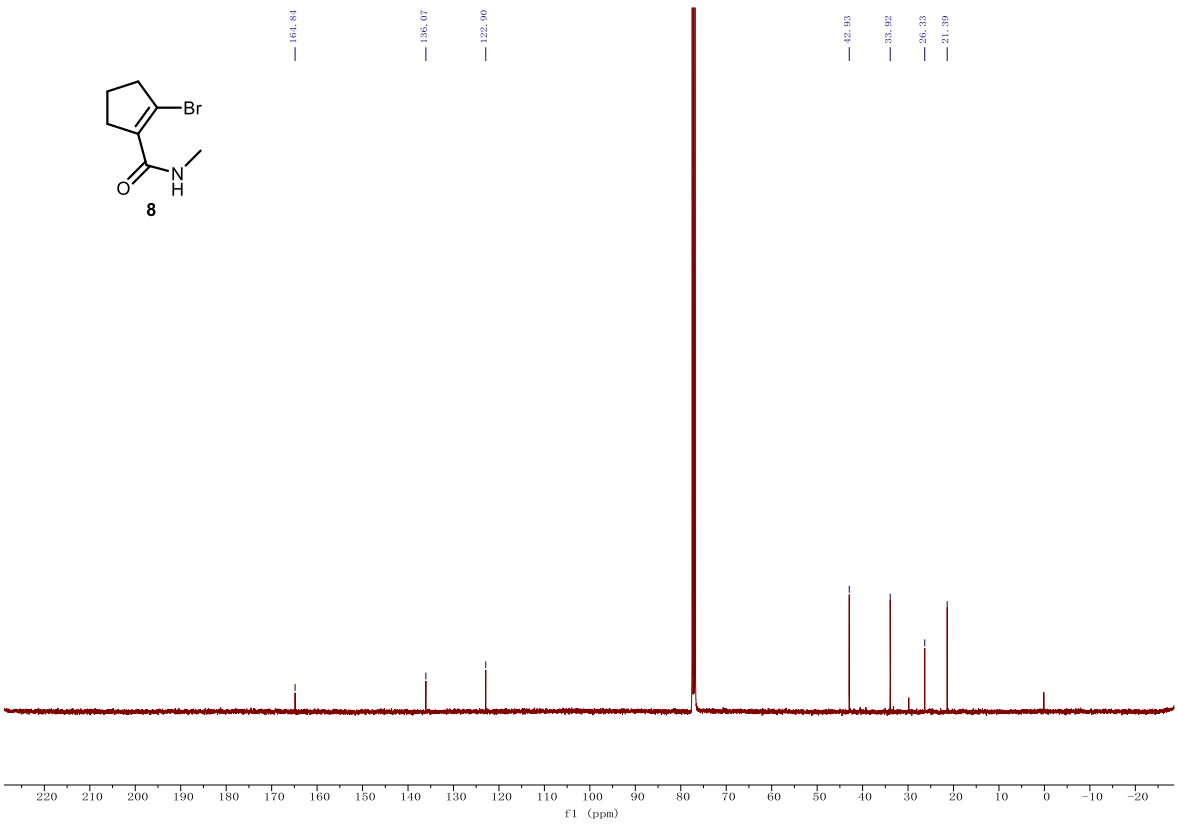

7.78
7.76
7.46
7.44
4.35
4.34
4.33
4.17
4.16
4.15
2.40
Br
TsN
CO2H
2.06
2.06
2.00
1.78
3.10
f1 (ppm)

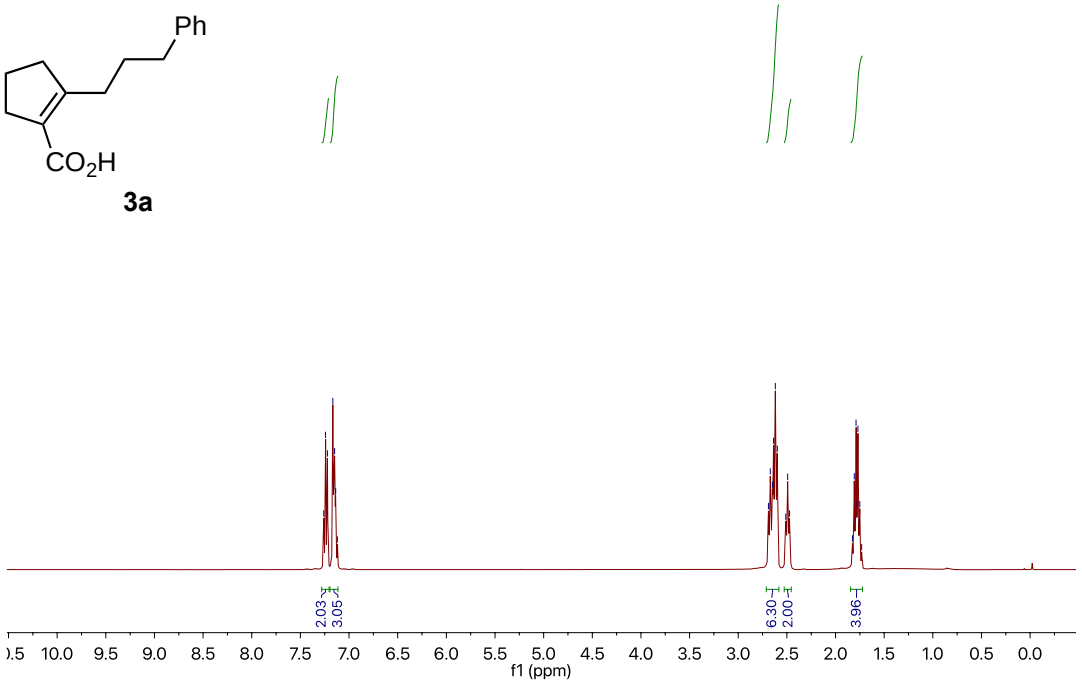
1k

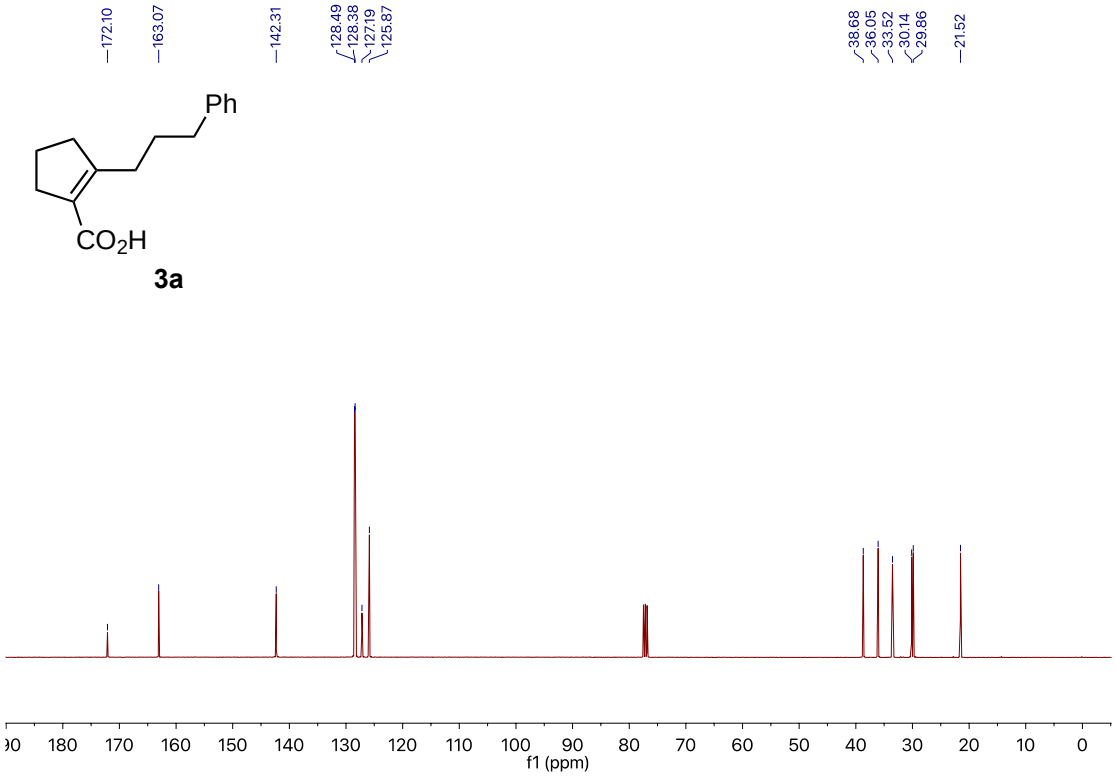

162.08
144.11
132.58
130.18
127.54
60.70
55.01
21.05
Br
TsN
CO2H
f1 (ppm)

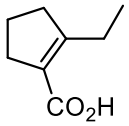
1k


Br
O
N
H
8
6.419
2.916
2.913
2.908
2.905
2.834
2.830
2.826
2.816
2.805
2.800
2.711
2.701
2.690
2.686
1.972
1.969
1.958
1.945
1.933
1.921
1.918
0.91
2.73
2.07
2.19
2.24
f1 (ppm)

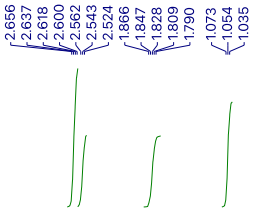

Br
O
N
H
8
164.84
136.07
122.90
42.93
33.92
26.33
21.39
f1 (ppm)

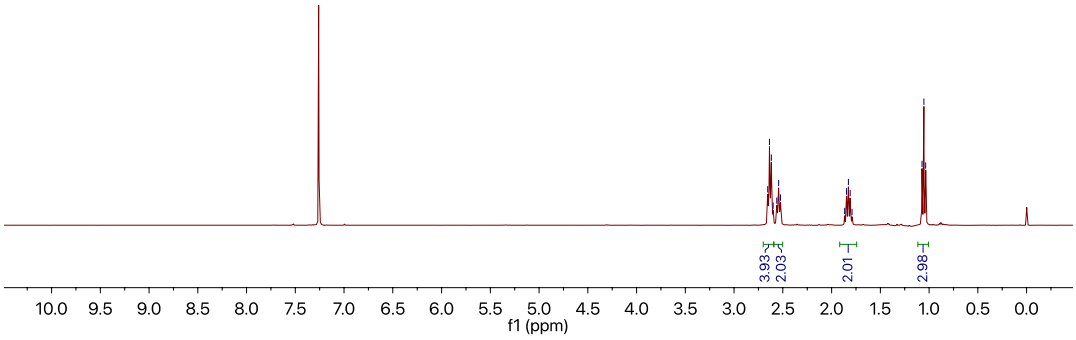

7.260
7.241
7.223
7.167
7.158
7.149
7.138
7.120
2.690
2.670
2.649
2.637
2.618
2.598
2.511
2.492
2.473
1.826
1.808
1.789
1.778
1.770
1.756
1.750
1.731
Ph
CO2H
3a
2.03
3.05
6.30
2.00
3.96
f1 (ppm)


172.10
163.07
142.31
128.49
128.38
127.19
125.87
38.68
36.05
33.52
30.14
29.86
21.52
Ph
CO2H
3a
f1 (ppm)

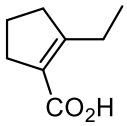

CO2H

3c


2.656
2.637
2.618
2.600
2.562
2.543
2.524
1.866
1.847
1.828
1.809
1.790
1.073
1.054
1.035

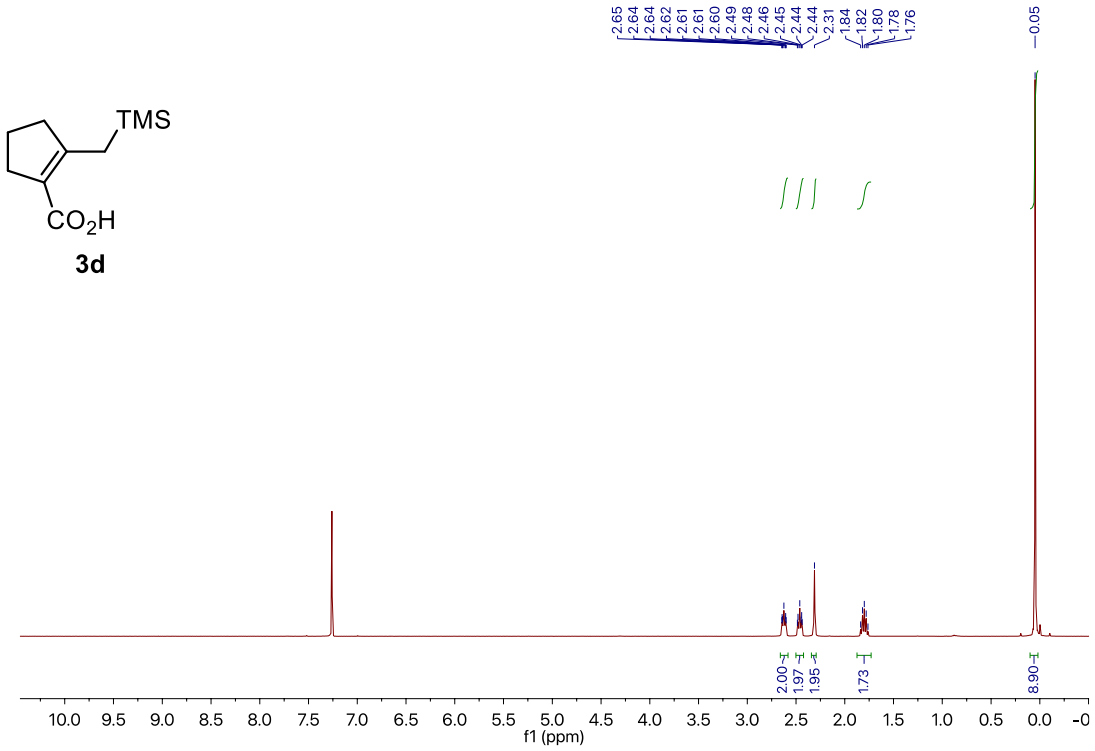

3.93
2.03
2.01
2.98
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

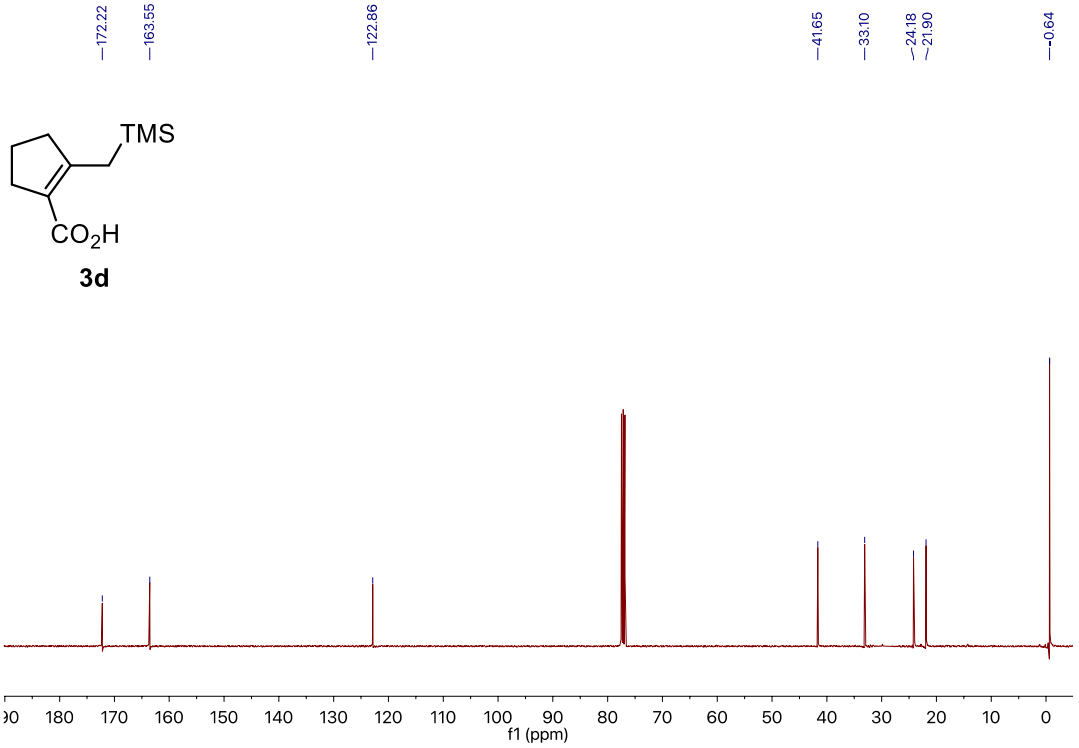

171.60
164.96
126.06
38.25
33.57
23.54
21.48
12.57

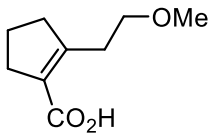

CO2H

3c

180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


TMS
CO2H
3d
2.65
2.64
2.64
2.62
2.61
2.61
2.60
2.49
2.48
2.46
2.45
2.44
2.44
2.31
1.84
1.82
1.80
1.78
1.76
0.05
2.00
1.97
1.95
1.73
8.90
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0
f1 (ppm)

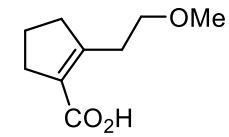

172.22
163.55
122.86
41.65
33.10
24.18
21.90
0.64
TMS
CO2H
3d
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


OMe
CO2H

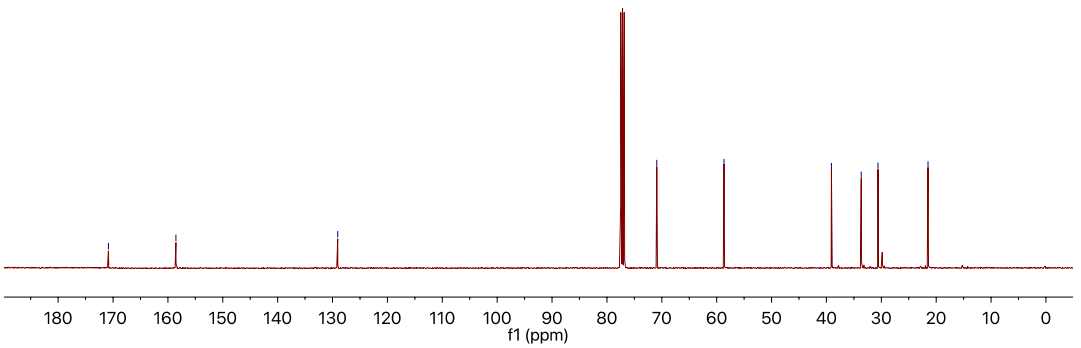
3e

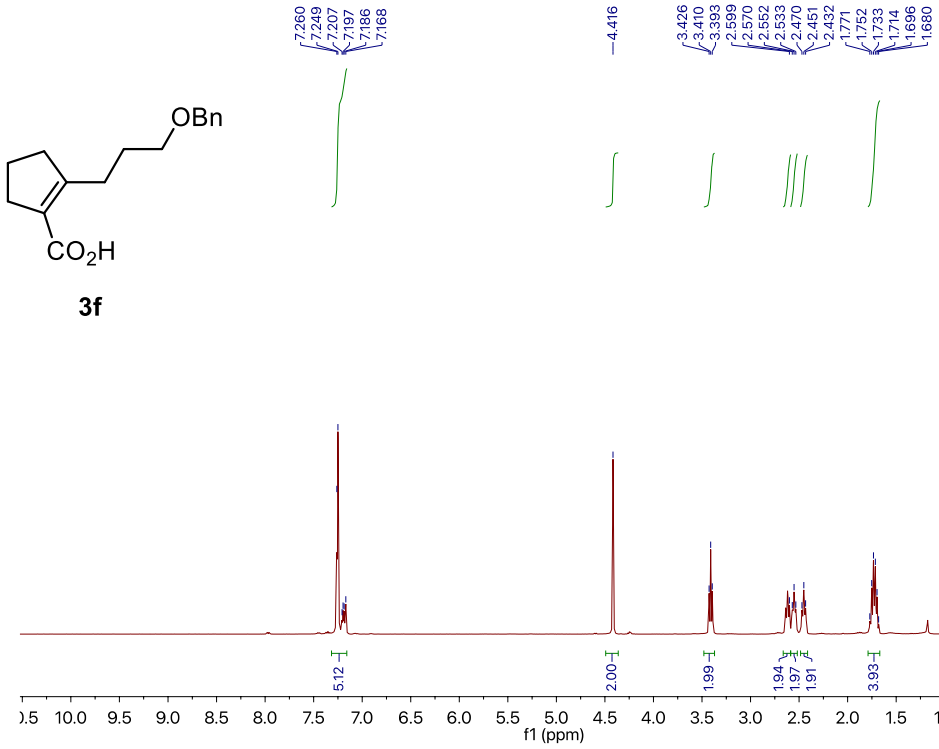
3.546
3.530
3.514
3.346
2.885
2.869
2.853
2.667
2.648
2.630
2.594
2.575
2.556
1.879
1.860
1.841
1.822
1.803
2.07
3.00
2.06
2.01
1.94
1.99
f1 (ppm)


170.84
158.55
129.06
70.92
58.67
39.09
33.66
30.60
21.49

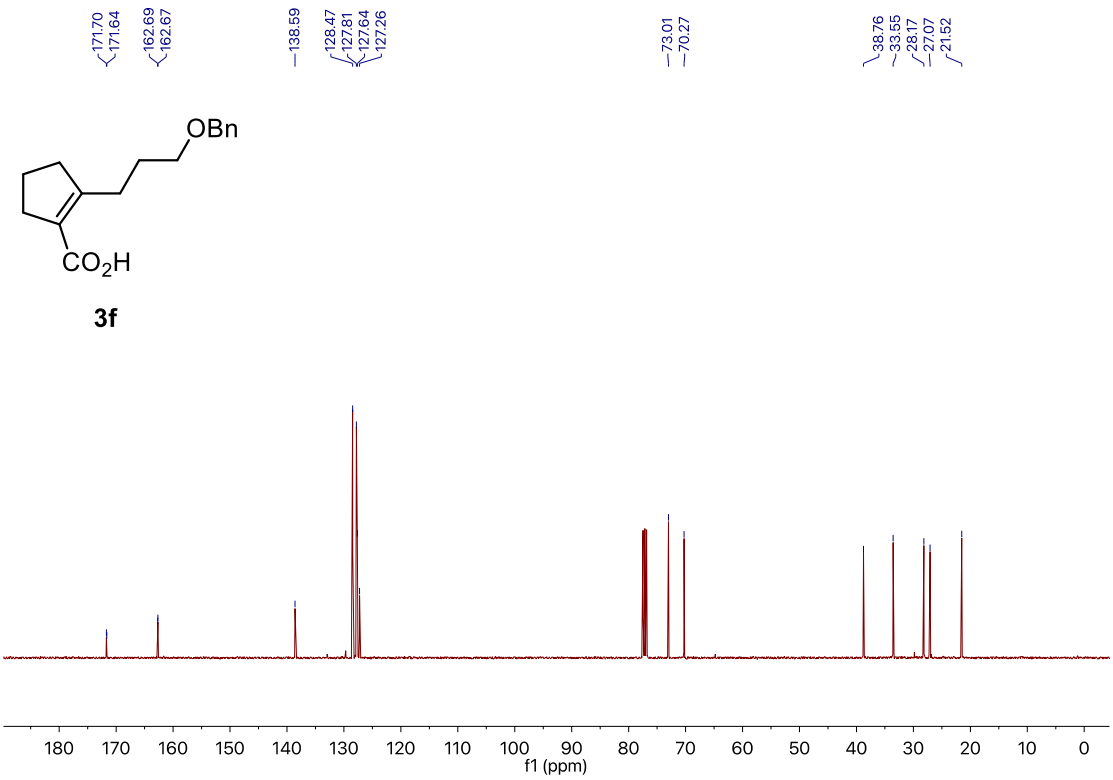

OMe
CO2H

3e

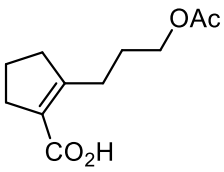

f1 (ppm)


7.260
7.249
7.207
7.197
7.186
7.168
4.416
3.426
3.410
3.393
2.599
2.570
2.552
2.533
2.470
2.451
2.432
1.771
1.752
1.733
1.714
1.696
1.680
OBn
CO2H
5.12
2.00
1.99
1.94
1.97
1.91
3.93
f1 (ppm)

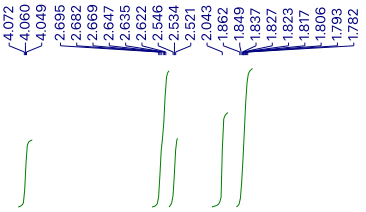
3f

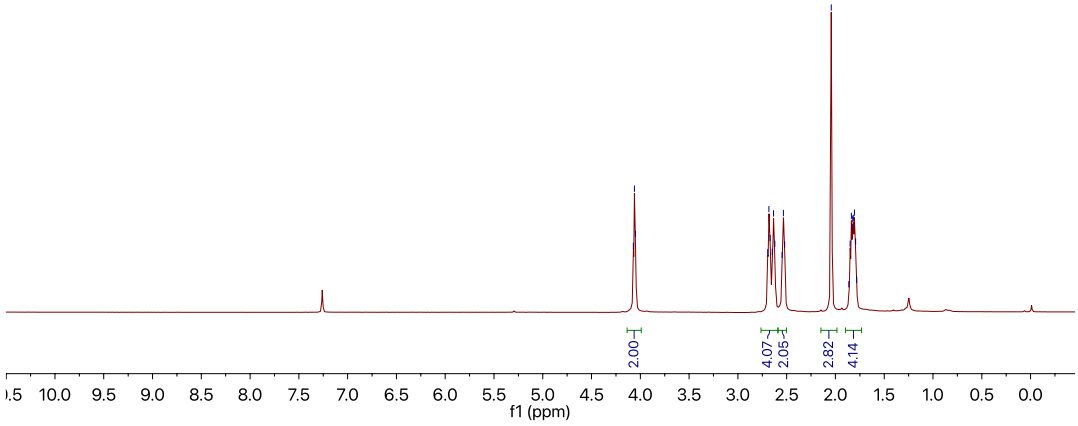

171.70
171.64
162.69
162.67
138.59
128.47
127.81
127.64
127.26
73.01
70.27
38.76
33.55
28.17
27.07
21.52
OBn
CO2H
f1 (ppm)

3f

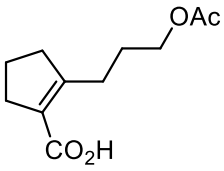

OAc
CO2H

3g

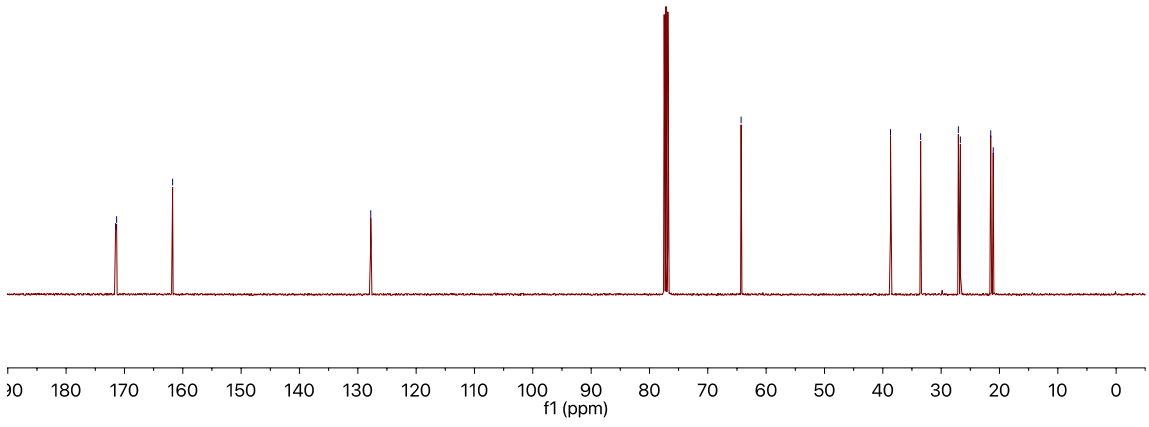

4.072
4.060
4.049
2.695
2.682
2.669
2.647
2.635
2.622
2.546
2.534
2.521
2.043
1.862
1.849
1.837
1.827
1.823
1.817
1.806
1.793
1.782

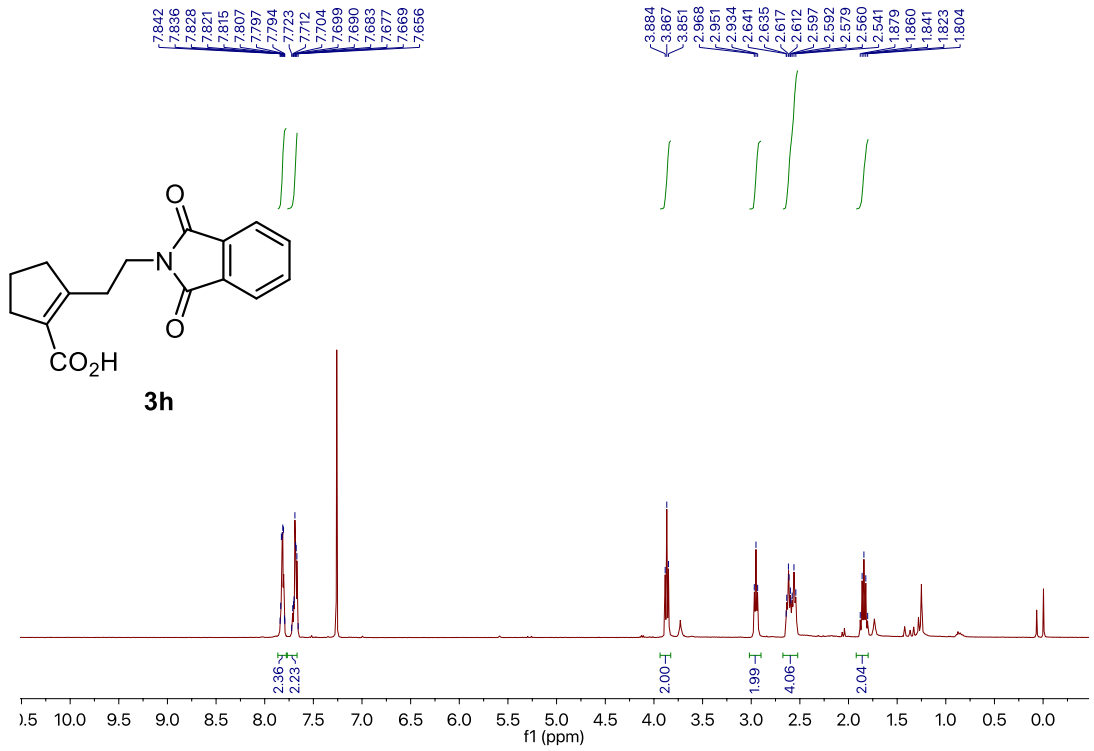

2.00
4.07
2.05
2.82
4.14
f1 (ppm)

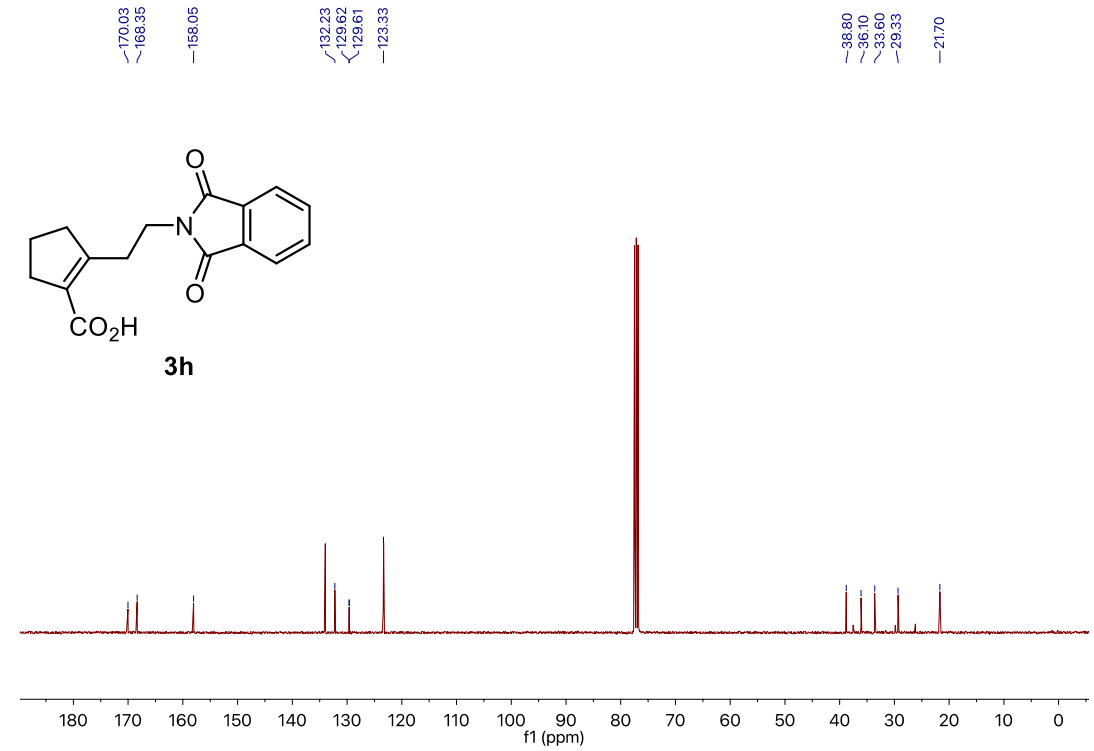

171.51
171.35
161.75
127.77
64.29
38.68
33.51
27.05
26.70
21.50
21.07

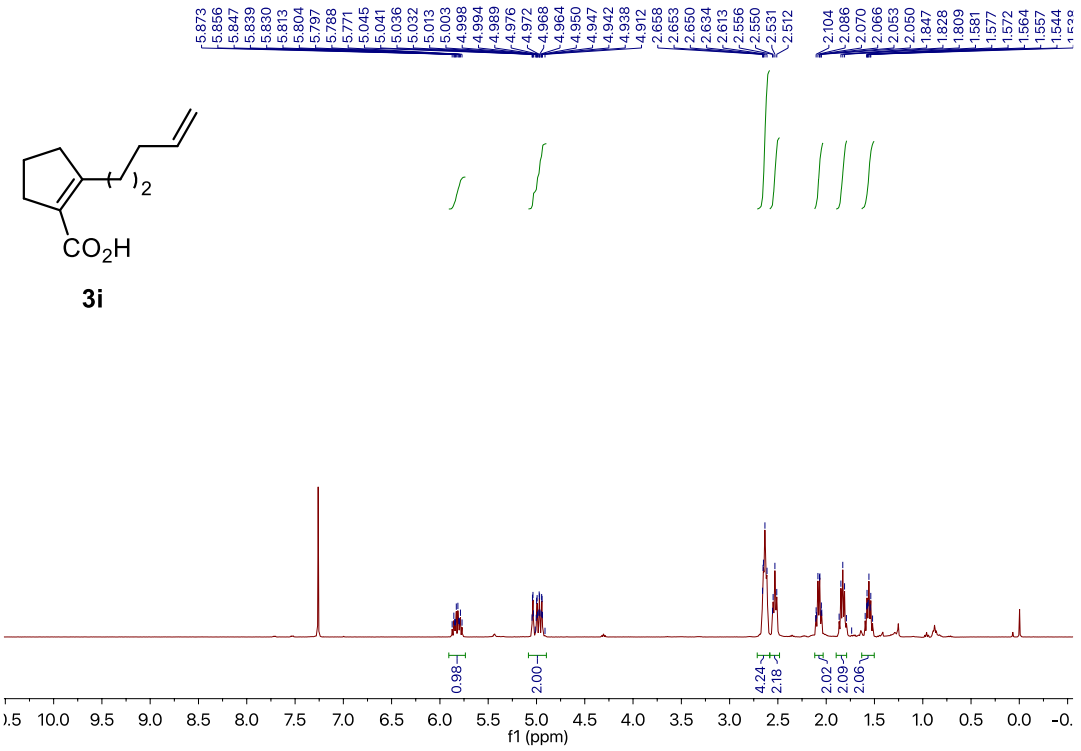

OAc
CO2H

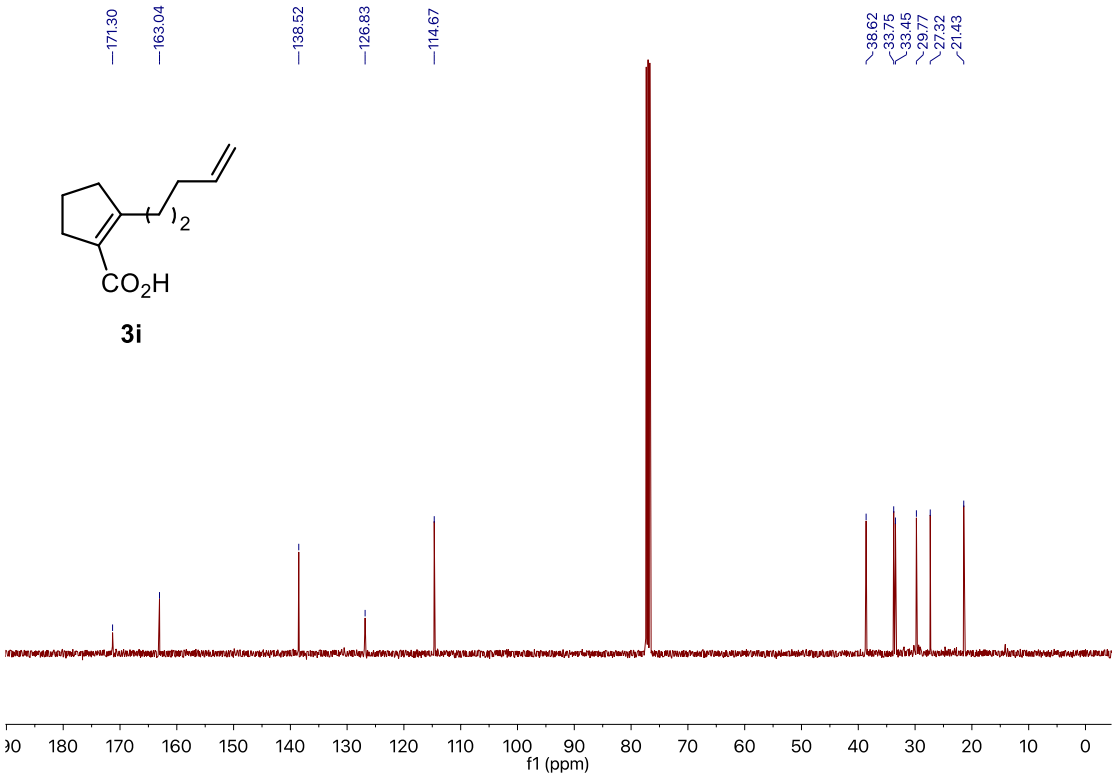
3g

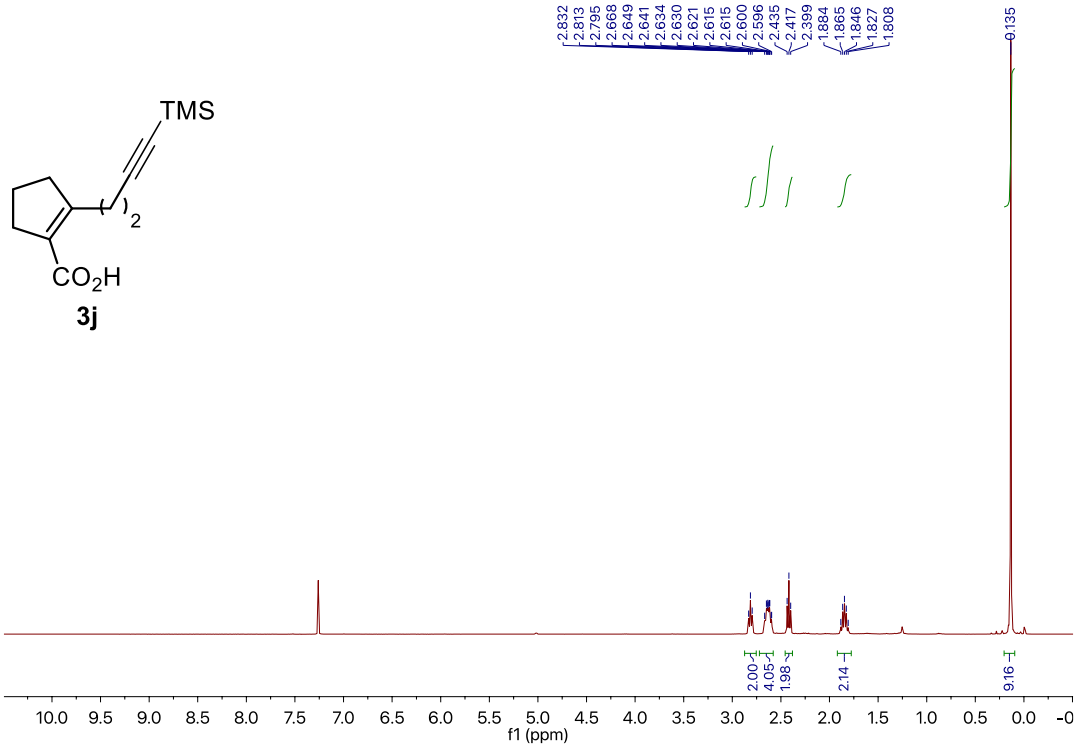

f1 (ppm)

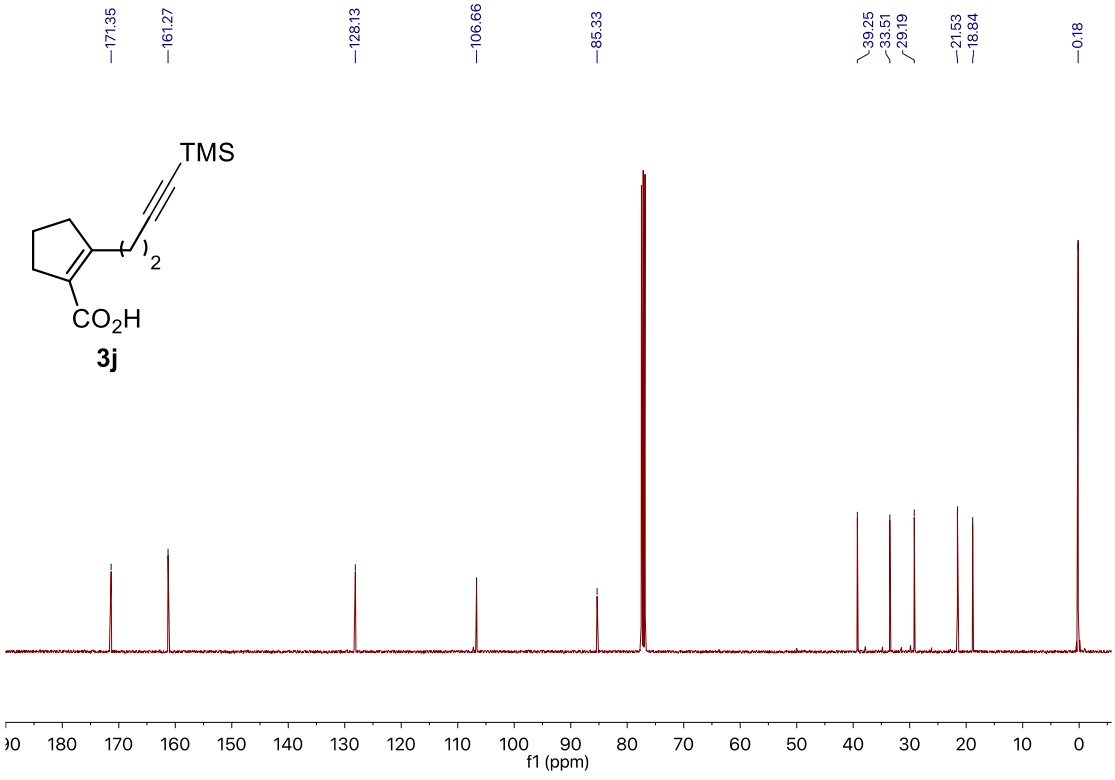

7.842
7.836
7.828
7.821
7.815
7.807
7.797
7.794
7.723
7.712
7.704
7.699
7.690
7.683
7.677
7.669
7.656
3.884
3.867
3.851
2.968
2.951
2.934
2.641
2.635
2.617
2.612
2.597
2.592
2.579
2.560
2.541
1.879
1.860
1.841
1.823
1.804
O
N
O
CO2H
3h
2.36
2.23
2.00
1.99
4.06
2.04
f1 (ppm)

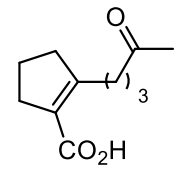

170.03
168.35
158.05
132.23
129.62
129.61
123.33
38.80
36.10
33.60
29.33
21.70
O
N
O
CO2H
3h
f1 (ppm)


5.873
5.856
5.847
5.839
5.830
5.813
5.804
5.797
5.788
5.771
5.045
5.041
5.036
5.032
5.013
5.003
4.998
4.994
4.989
4.976
4.972
4.968
4.964
4.950
4.947
4.942
4.938
4.912
2.658
2.653
2.650
2.634
2.613
2.556
2.550
2.531
2.512
2.104
2.086
2.070
2.066
2.053
2.050
1.847
1.828
1.809
1.581
1.577
1.572
1.564
1.557
1.544
CO2H
3i
0.98
2.00
4.24
2.18
2.02
2.09
2.06
f1 (ppm)

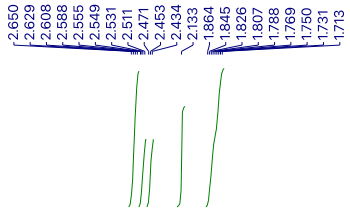

171.30
163.04
138.52
126.83
114.67
38.62
33.75
33.45
29.77
27.32
21.43
CO2H
3i
f1 (ppm)

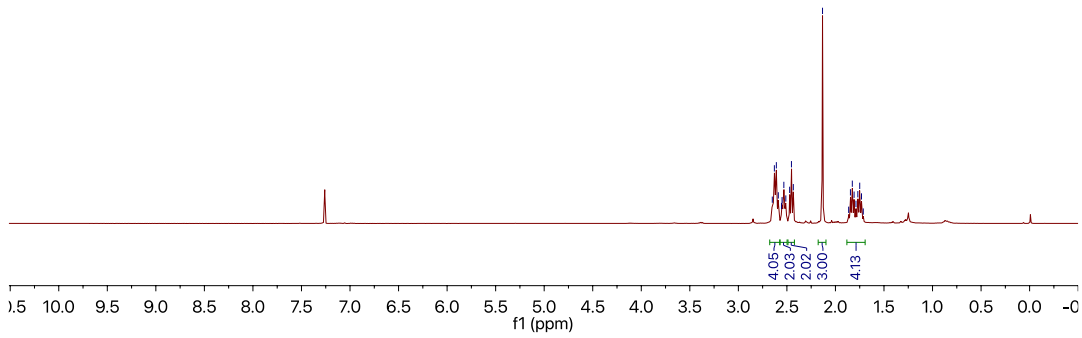

TMS
2
CO2H
3j
2.832
2.813
2.795
2.668
2.649
2.641
2.634
2.630
2.621
2.615
2.615
2.600
2.596
2.435
2.417
2.399
1.884
1.865
1.846
1.827
1.808
0.135
2.00
4.05
1.98
2.14
9.16
f1 (ppm)

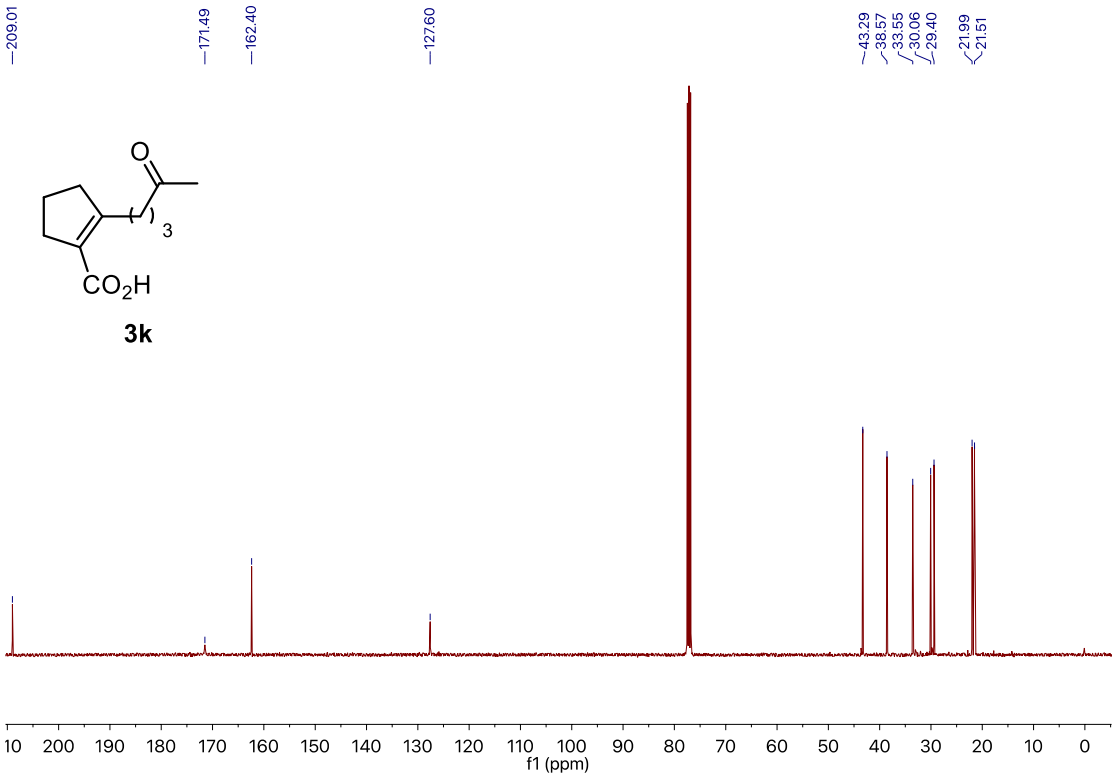

171.35
161.27
128.13
106.66
85.33
39.25
33.51
29.19
21.53
18.84
0.18
TMS
2
CO2H
3j
f1 (ppm)



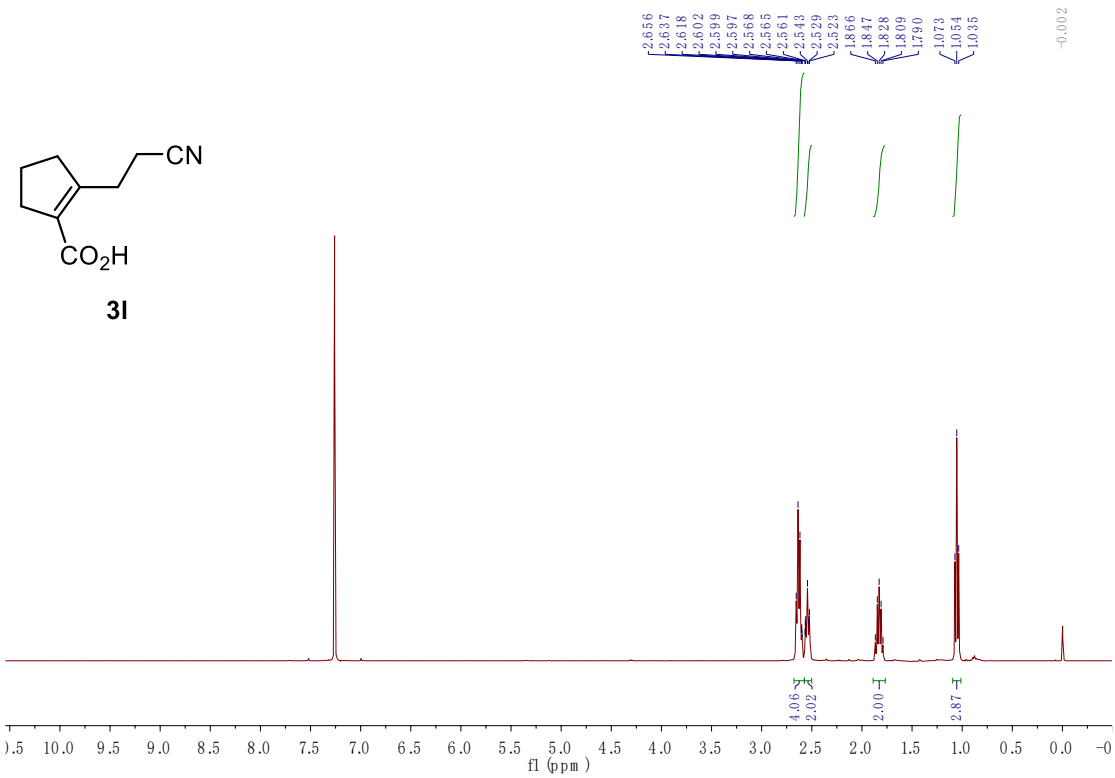
3k

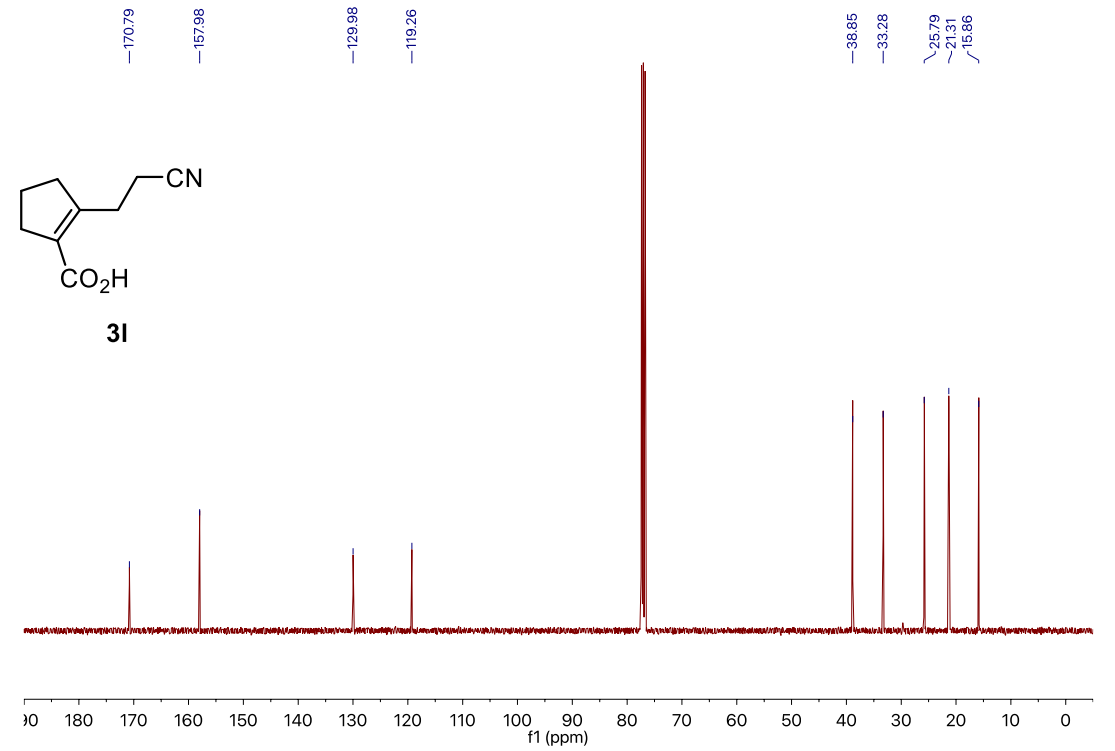

2.650
2.629
2.608
2.588
2.555
2.549
2.531
2.511
2.471
2.453
2.434
2.133
1.864
1.845
1.826
1.807
1.788
1.769
1.750
1.731
1.713

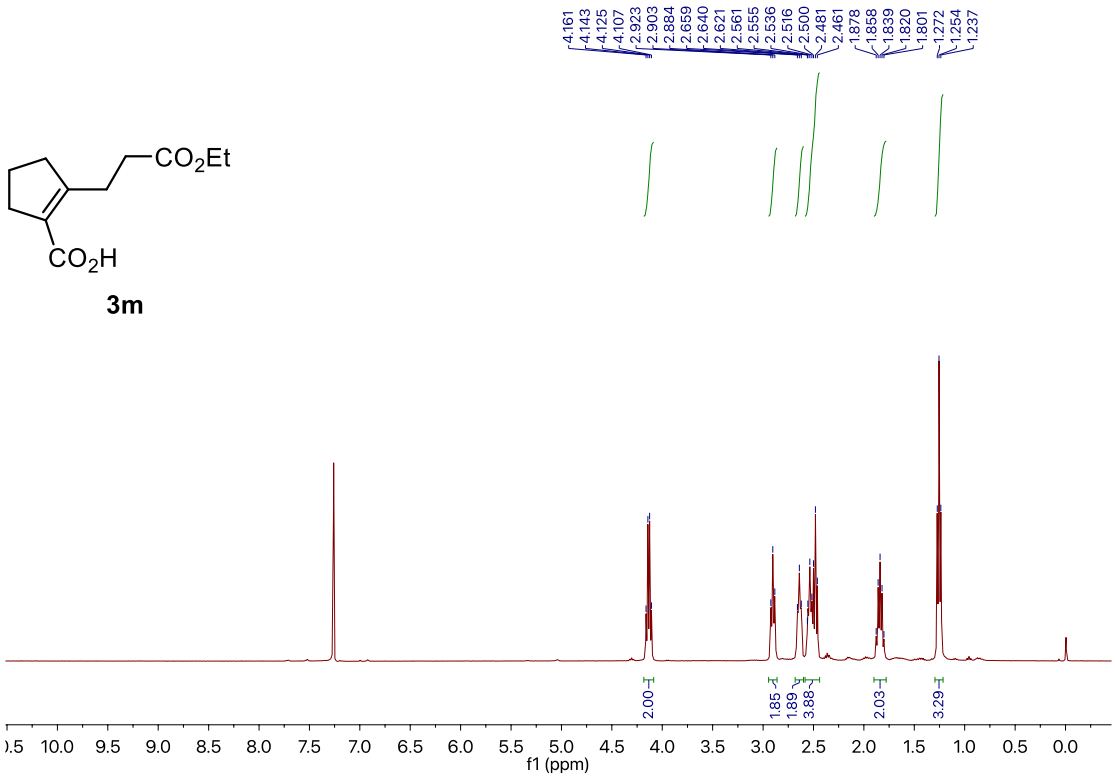

4.05
2.03
2.02
3.00
4.13
f1 (ppm)

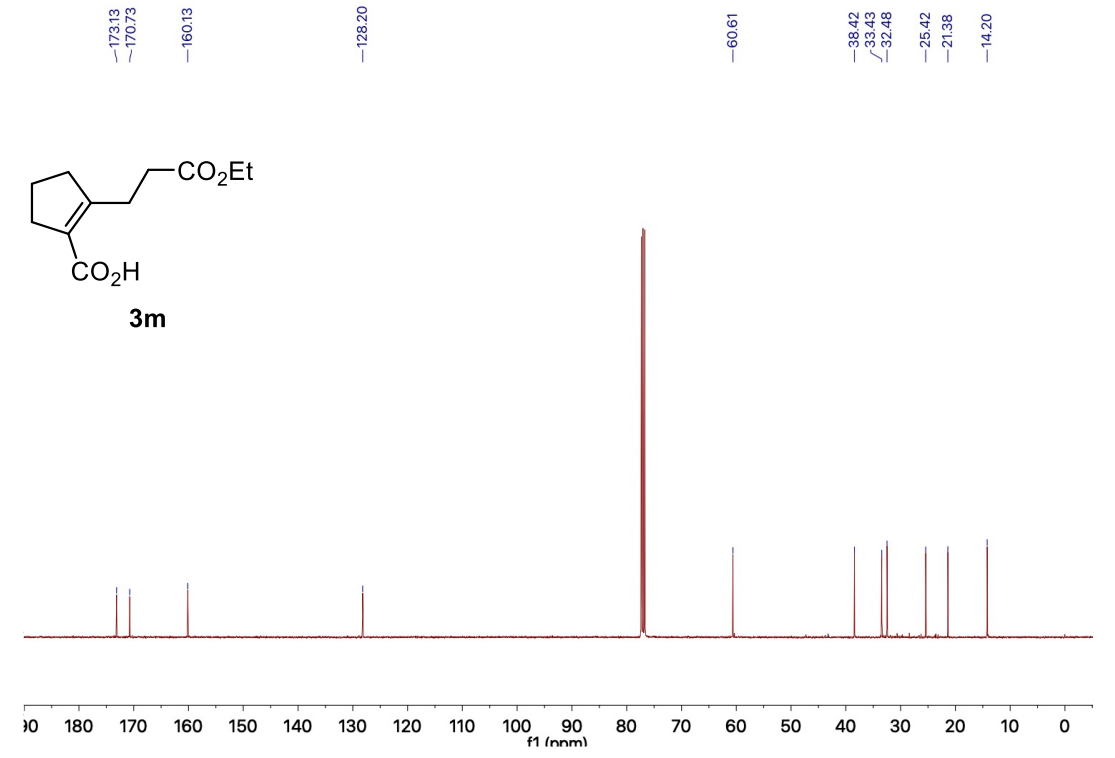

209.01
171.49
162.40
127.60
43.29
38.57
33.55
30.06
29.40
21.99
21.51
f1 (ppm)

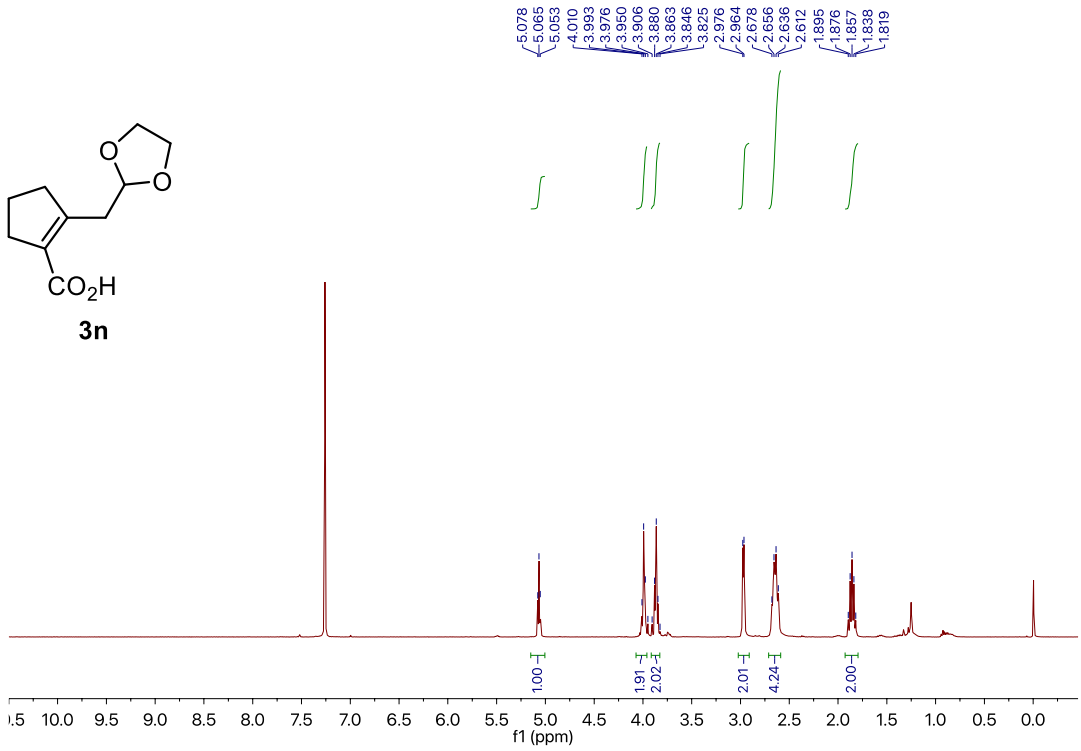
3k

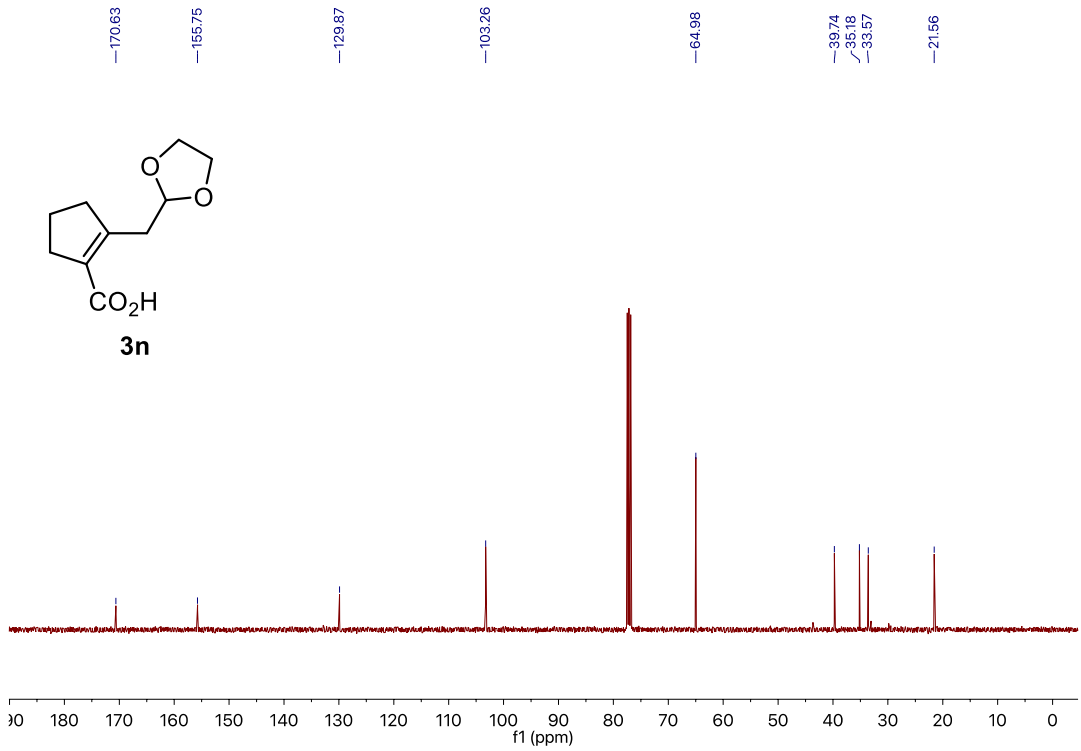

CN
CO2H
3l
2.656
2.637
2.618
2.602
2.599
2.597
2.568
2.565
2.561
2.543
2.529
2.523
1.866
1.847
1.828
1.809
1.790
1.073
1.054
1.035
-0.002
4.06
2.02
2.00
2.87
f1 (ppm)

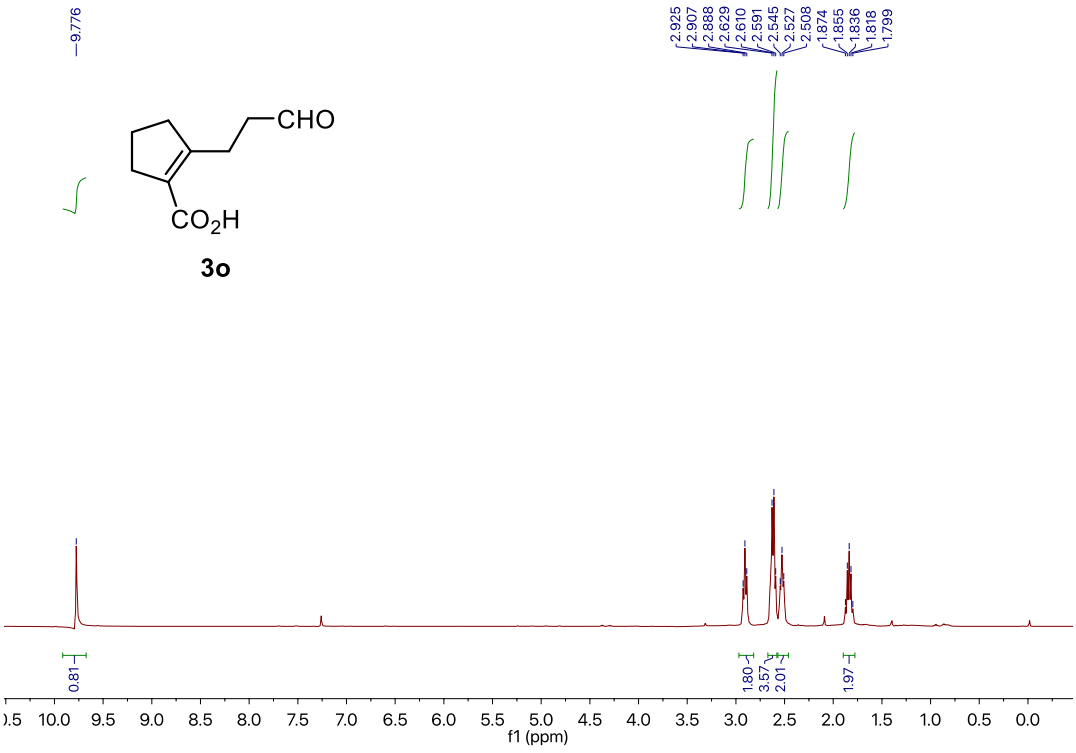

CN
CO2H
3l
170.79
157.98
129.98
119.26
38.85
33.28
25.79
21.31
15.86
f1 (ppm)


4.161
4.143
4.125
4.107
2.923
2.903
2.884
2.659
2.640
2.621
2.561
2.555
2.536
2.516
2.500
2.481
2.461
1.878
1.858
1.839
1.820
1.801
1.272
1.254
1.237
CO2Et
CO2H
3m
2.00
1.85
1.89
3.88
2.03
3.29
f1 (ppm)

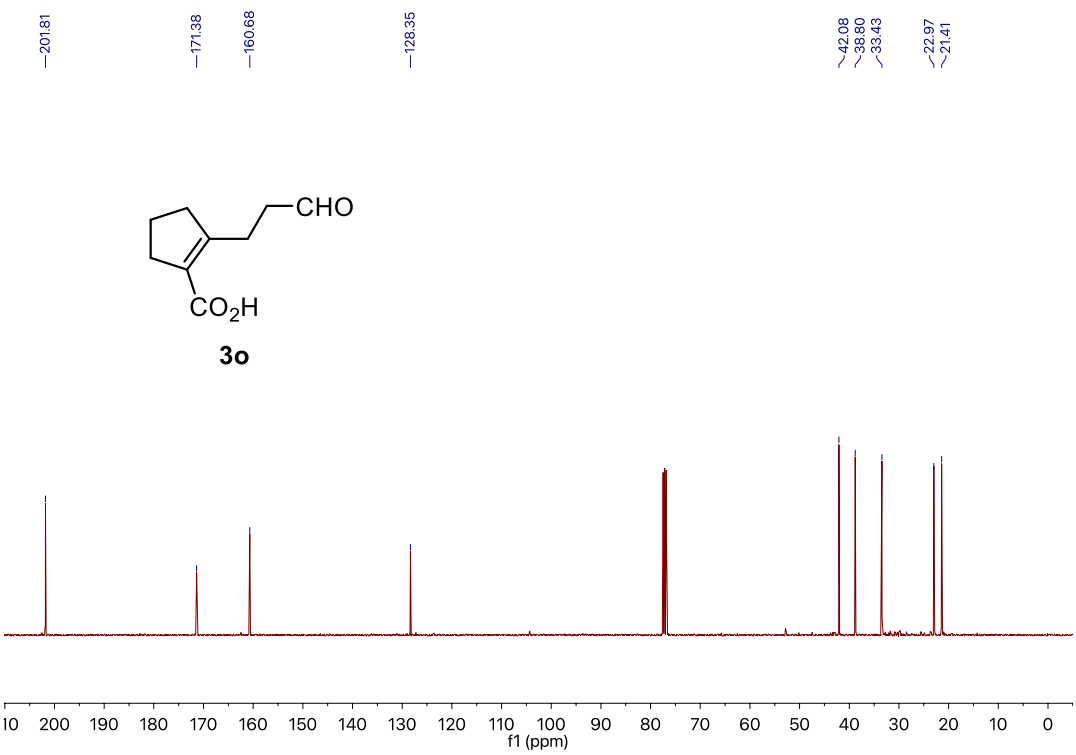

173.13
170.73
160.13
128.20
60.61
38.42
33.43
32.48
25.42
21.38
14.20
CO2Et
CO2H
3m
f1 (ppm)


5.078
5.065
5.053
4.010
3.993
3.976
3.950
3.906
3.880
3.863
3.846
3.825
2.976
2.964
2.678
2.656
2.636
2.612
1.895
1.876
1.857
1.838
1.819
O
O
O
CO2H
3n
1.00
1.91
2.02
2.01
4.24
2.00
f1 (ppm)

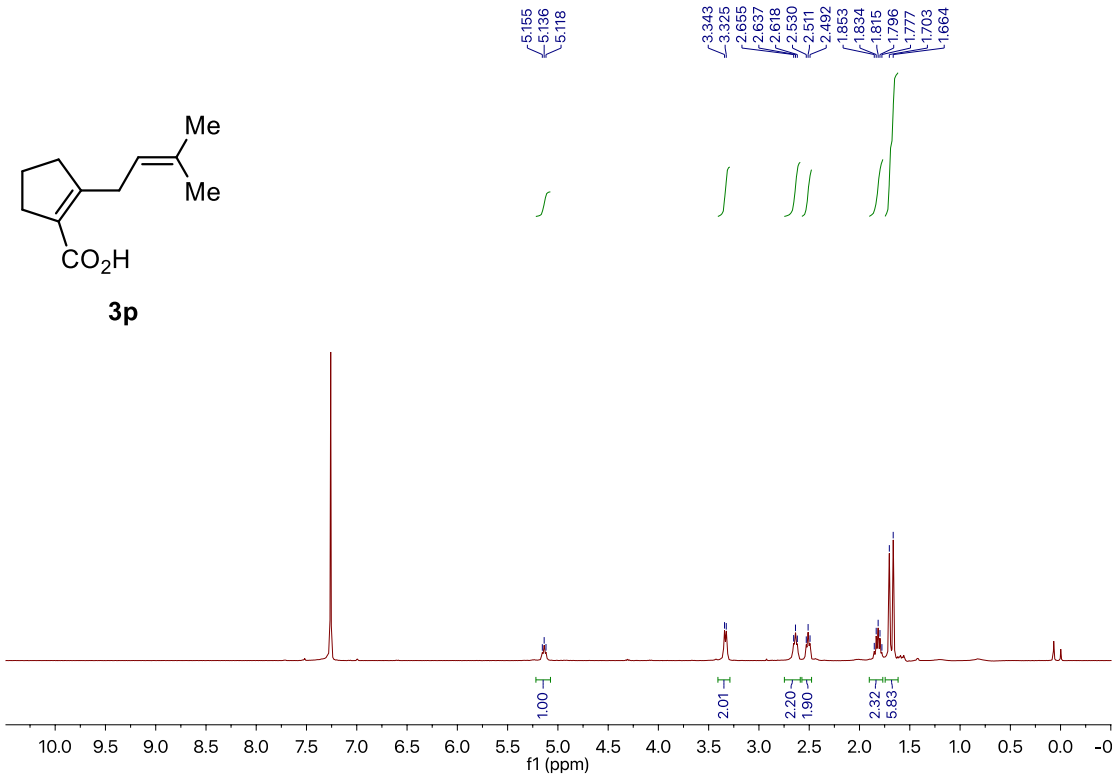

170.63
155.75
129.87
103.26
64.98
39.74
35.18
33.57
21.56
O
O
O
CO2H
3n
f1 (ppm)

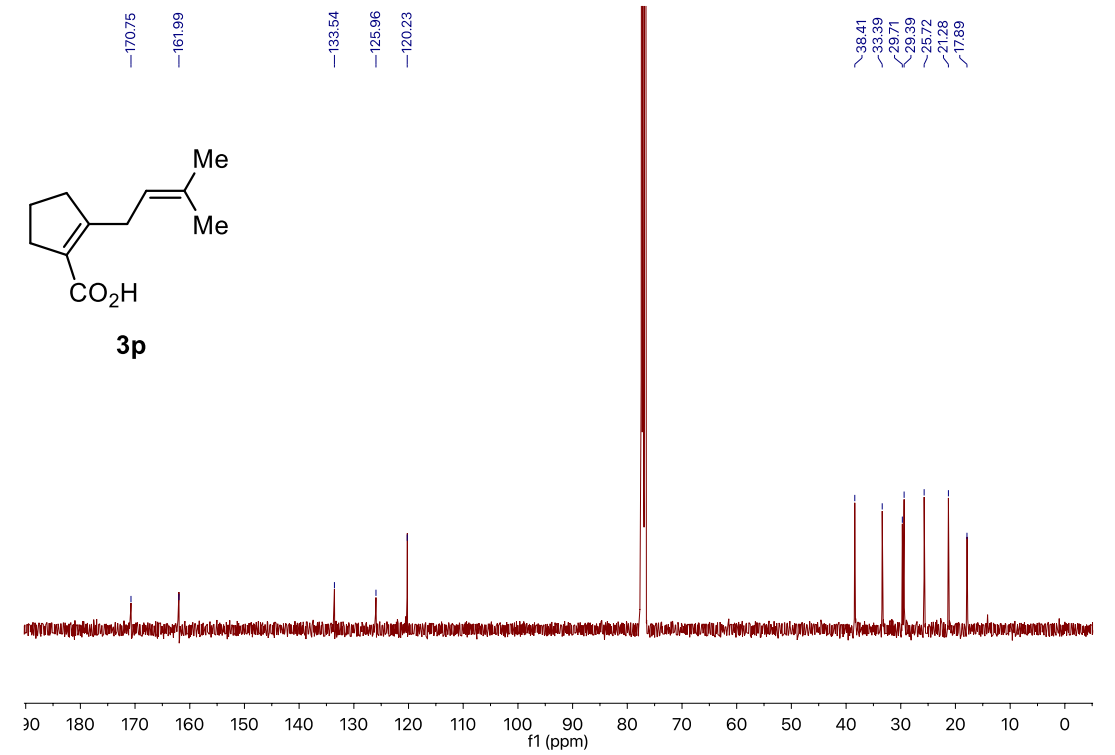

9.776
2.925
2.907
2.888
2.629
2.610
2.591
2.545
2.527
2.508
1.874
1.855
1.836
1.818
1.799
CHO
CO2H
0.81
1.80
3.57
2.01
1.97
f1 (ppm)

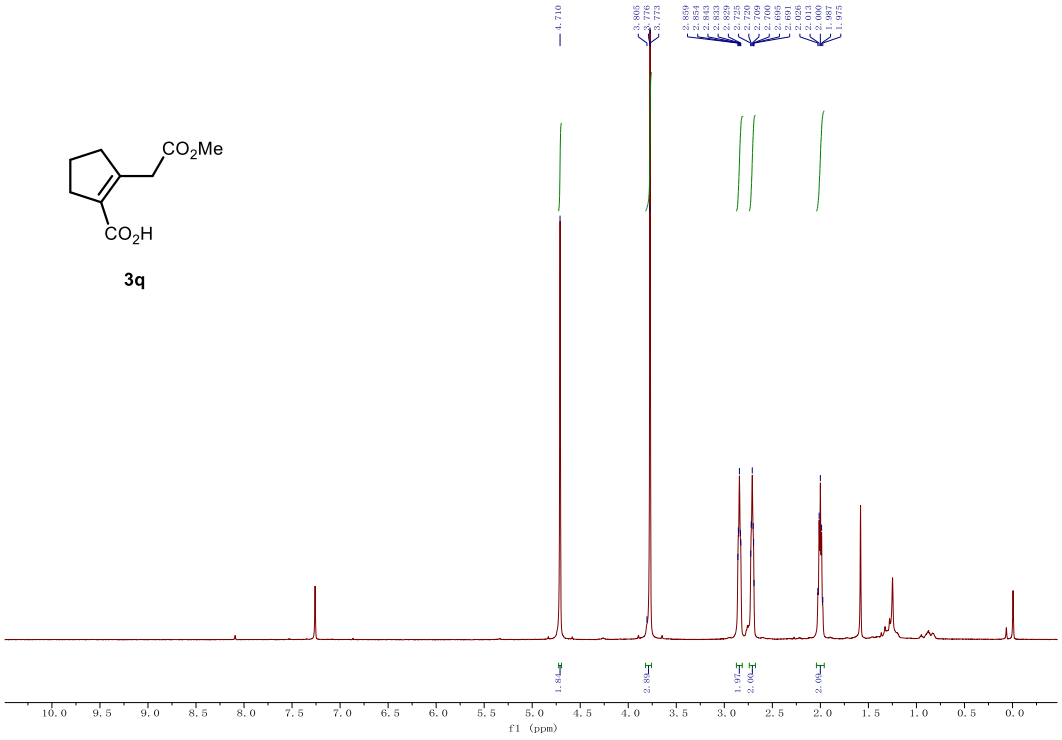
3o

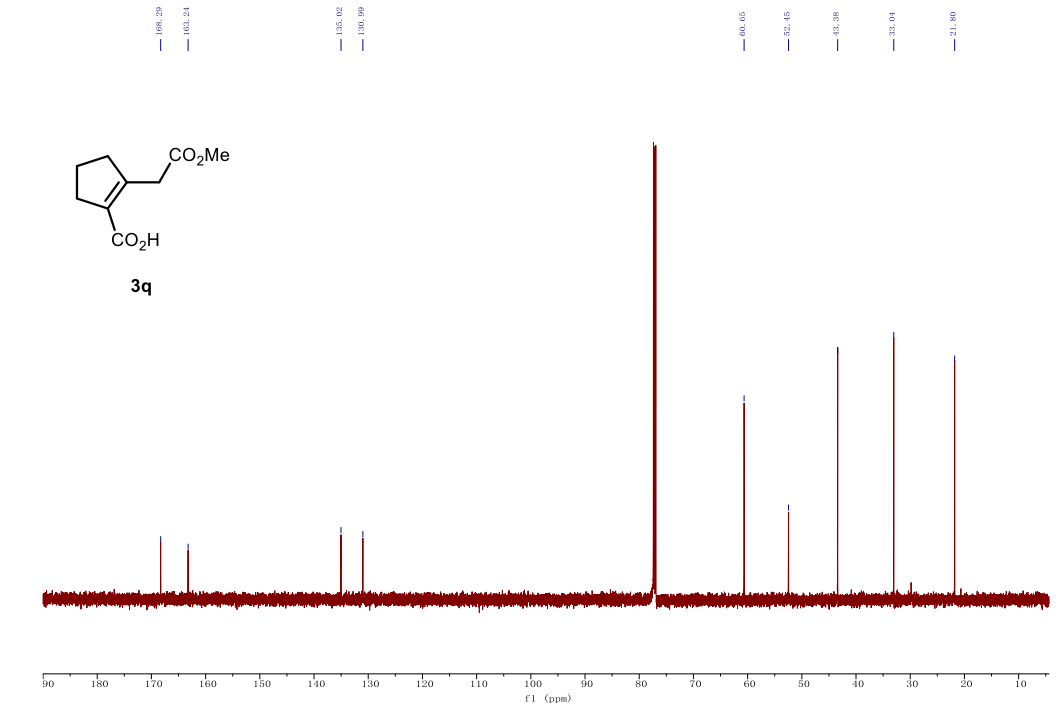

201.81
171.38
160.68
128.35
42.08
38.80
33.43
22.97
21.41
CHO
CO2H
f1 (ppm)

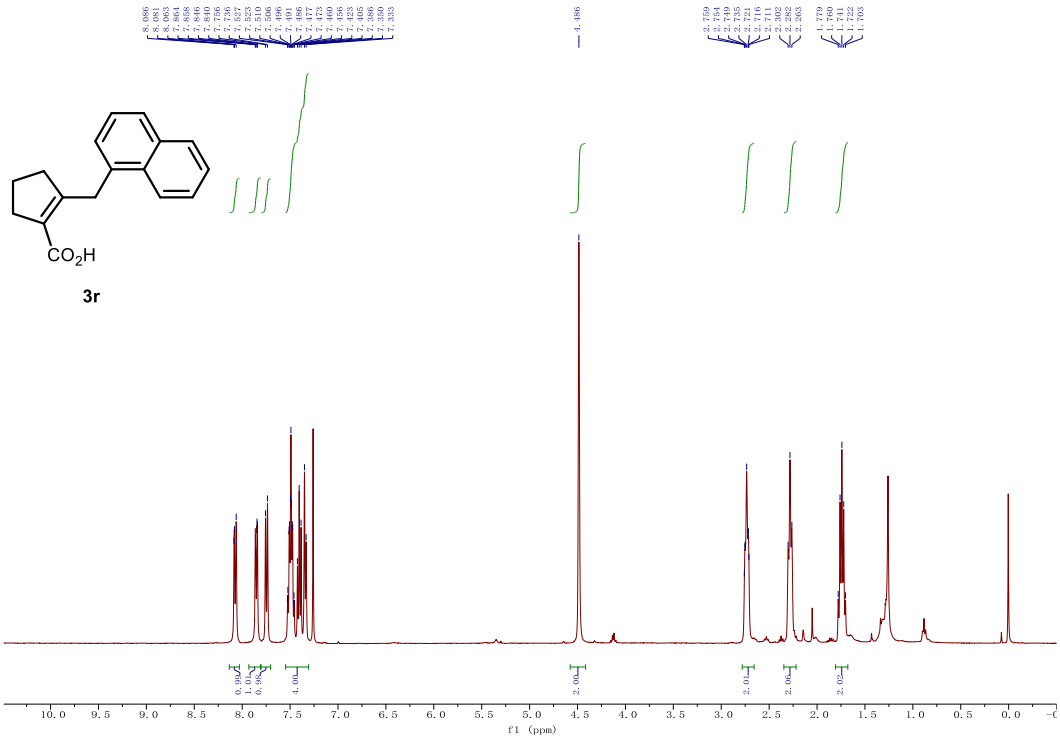
3o

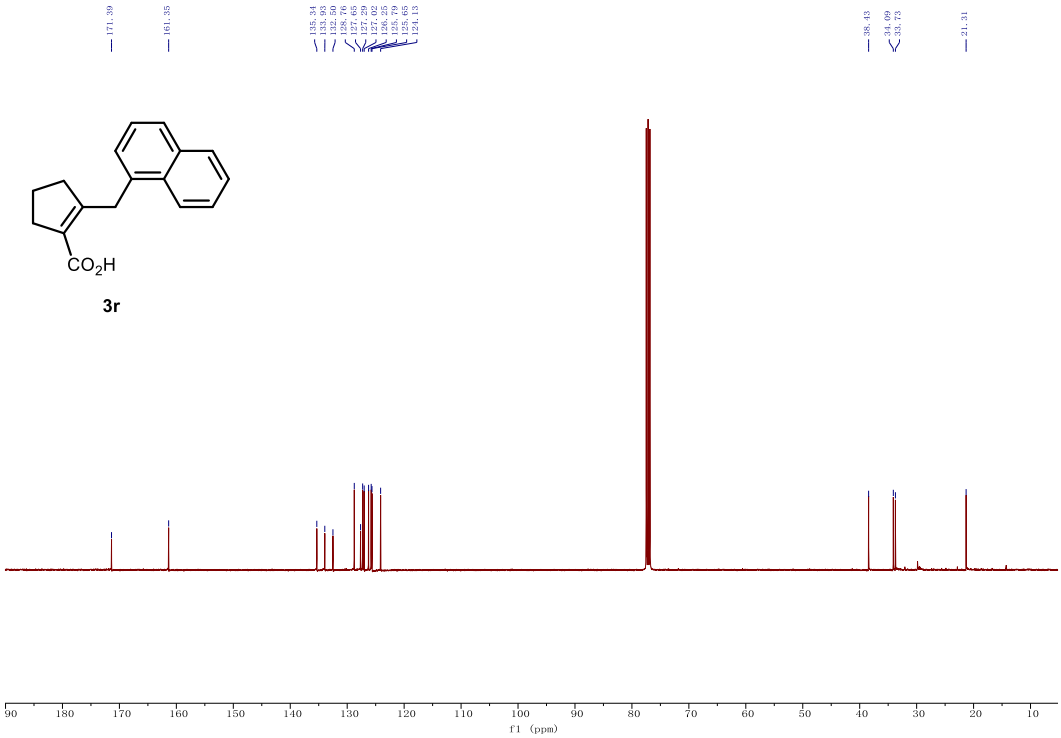

5.155
5.136
5.118
3.343
3.325
2.655
2.637
2.618
2.530
2.511
2.492
1.853
1.834
1.815
1.796
1.777
1.703
1.664
Me
Me
CO2H
3p
1.00
2.01
2.20
1.90
2.32
5.83
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0
f1 (ppm)

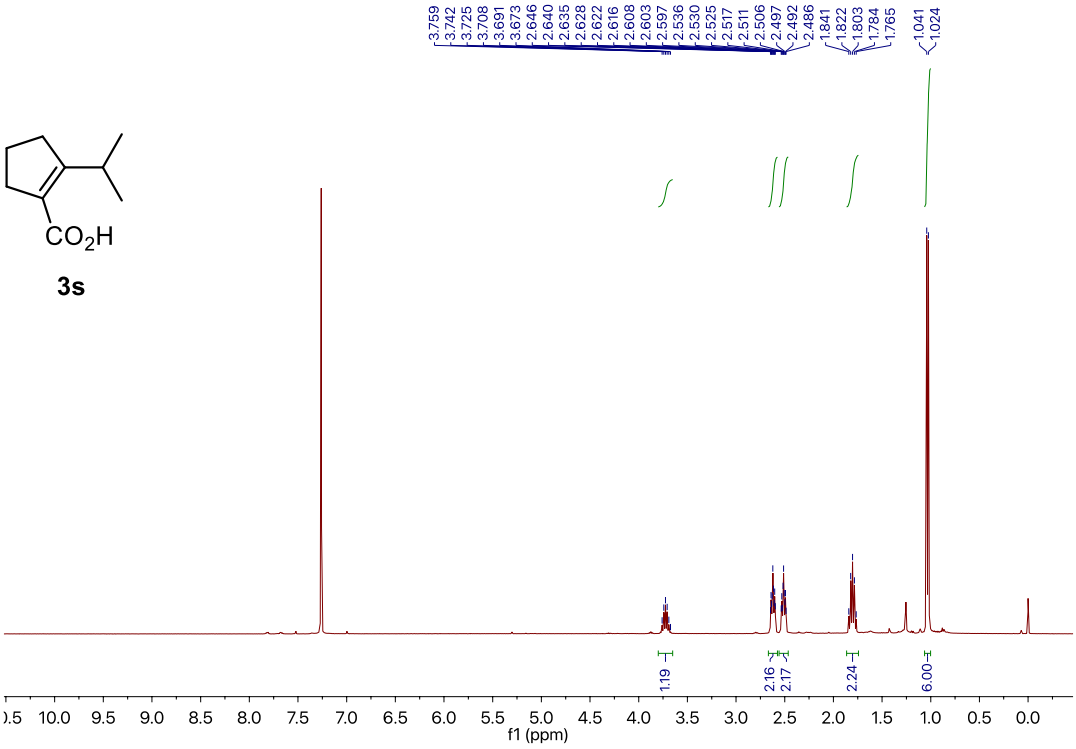

170.75
161.99
133.54
125.96
120.23
38.41
33.39
29.71
29.39
25.72
21.28
17.89
Me
Me
CO2H
3p
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

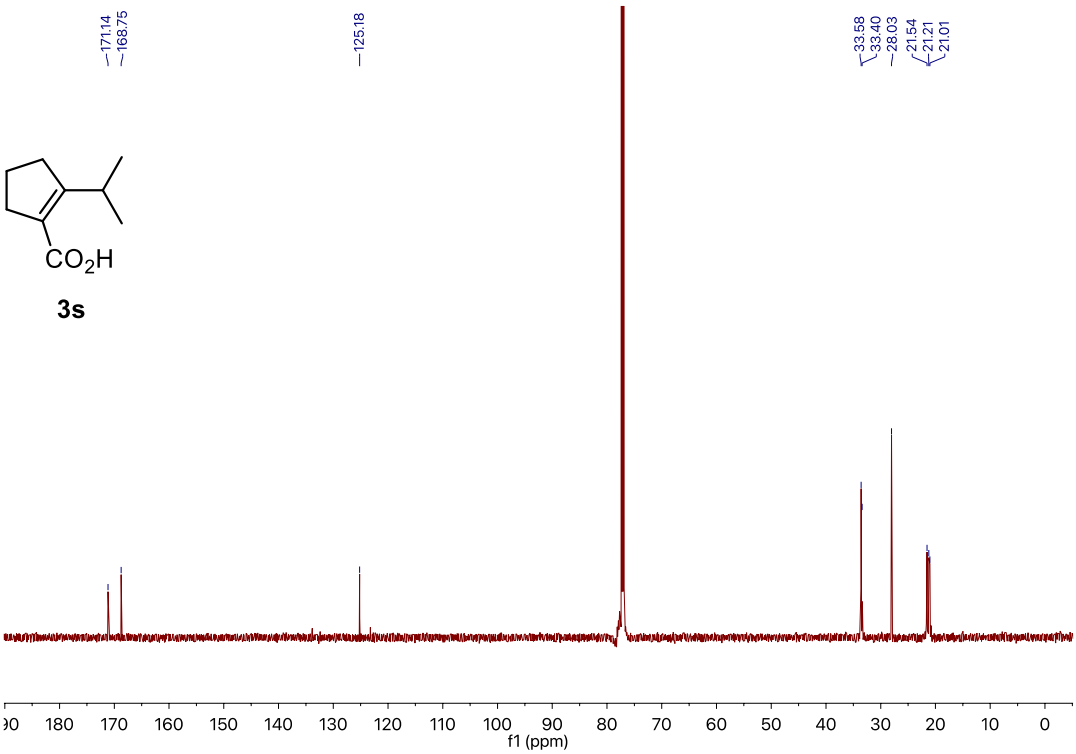

CO2Me
CO2H
3q
4.710
3.805
3.776
3.773
2.859
2.854
2.843
2.833
2.829
2.725
2.720
2.709
2.700
2.695
2.691
2.026
2.013
2.000
1.987
1.975
1.84
2.84
1.97
2.00
2.04
f1 (ppm)

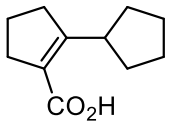

168.29
163.24
135.02
130.99
69.65
52.45
43.38
33.04
21.80
CO2Me
CO2H
3q
f1 (ppm)


8.086
8.081
8.065
7.864
7.858
7.846
7.840
7.756
7.736
7.527
7.523
7.510
7.506
7.496
7.491
7.486
7.477
7.473
7.460
7.456
7.453
7.405
7.386
7.350
7.343
4.486
2.759
2.754
2.749
2.735
2.721
2.716
2.711
2.302
2.282
2.263
1.779
1.760
1.741
1.722
1.703
CO2H
3r
0.99
1.01
0.98
4.00
2.00
2.01
2.06
2.02
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


171.39
161.35
135.34
133.93
132.50
128.76
127.65
127.39
127.02
126.25
125.79
125.65
124.13
38.43
34.09
33.73
21.31
CO2H
3r
90 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

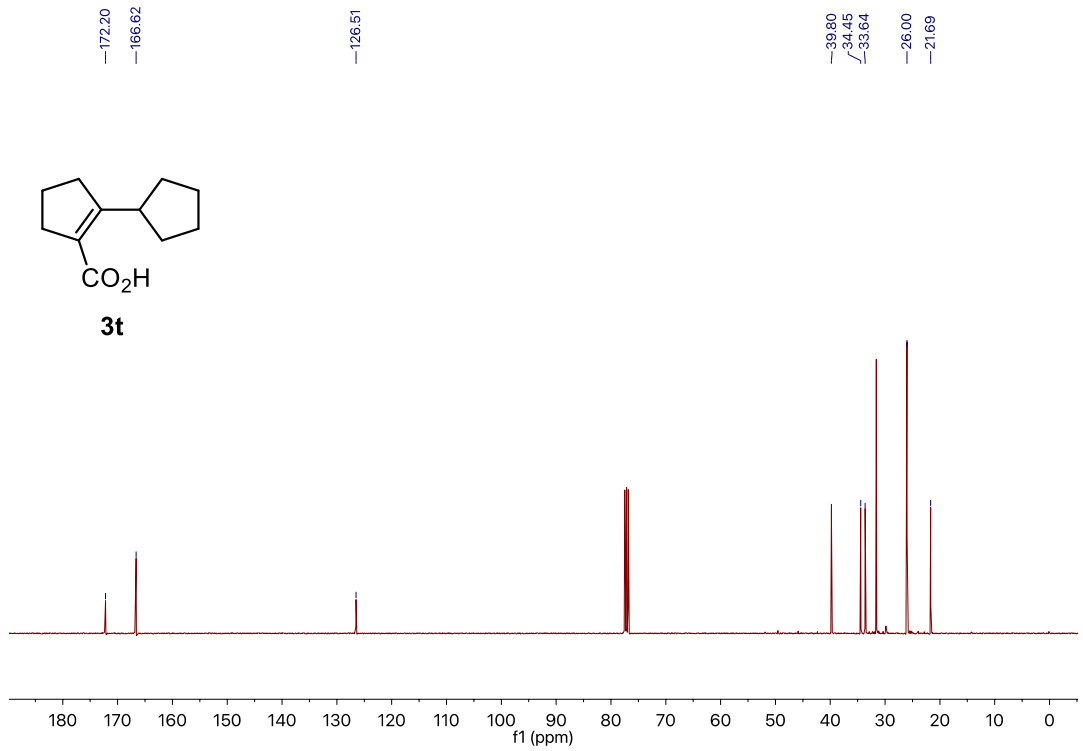

3.759
3.742
3.725
3.708
3.691
3.673
2.646
2.640
2.635
2.628
2.622
2.616
2.608
2.603
2.597
2.536
2.530
2.525
2.517
2.511
2.506
2.497
2.492
2.486
1.841
1.822
1.803
1.784
1.765
1.041
1.024
CO2H
3s
1.19
2.16
2.17
2.24
6.00
f1 (ppm)


171.14
168.75
125.18
33.58
33.40
28.03
21.54
21.21
21.01
CO2H
3s
f1 (ppm)


CO2H

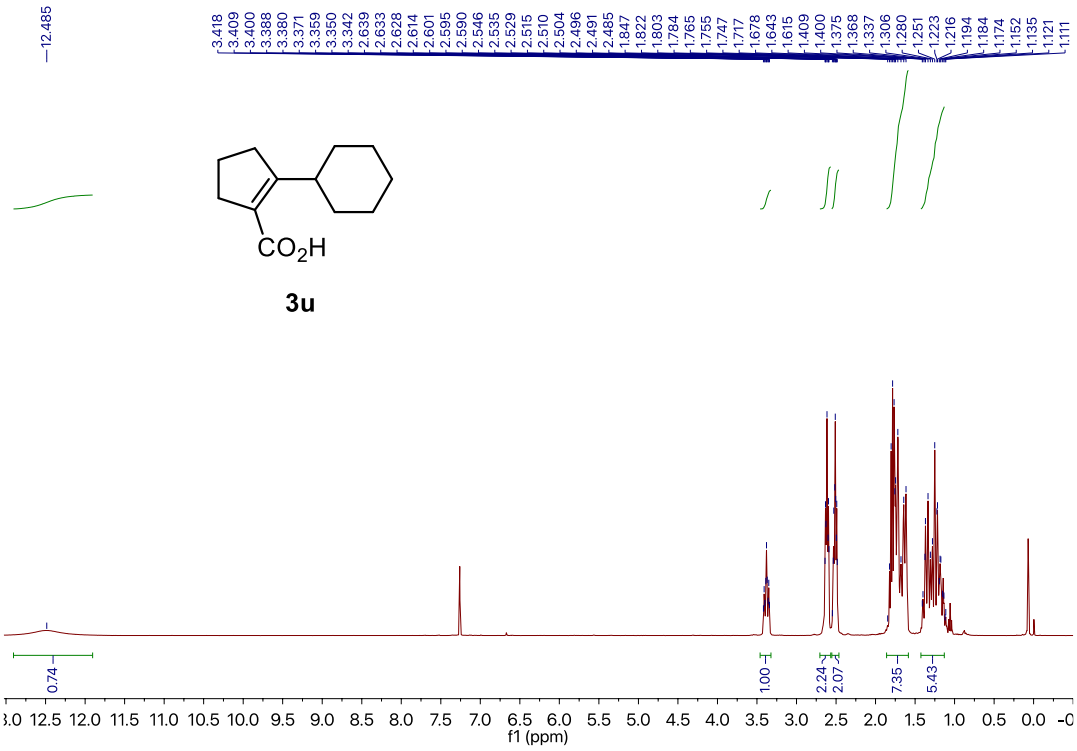
3t

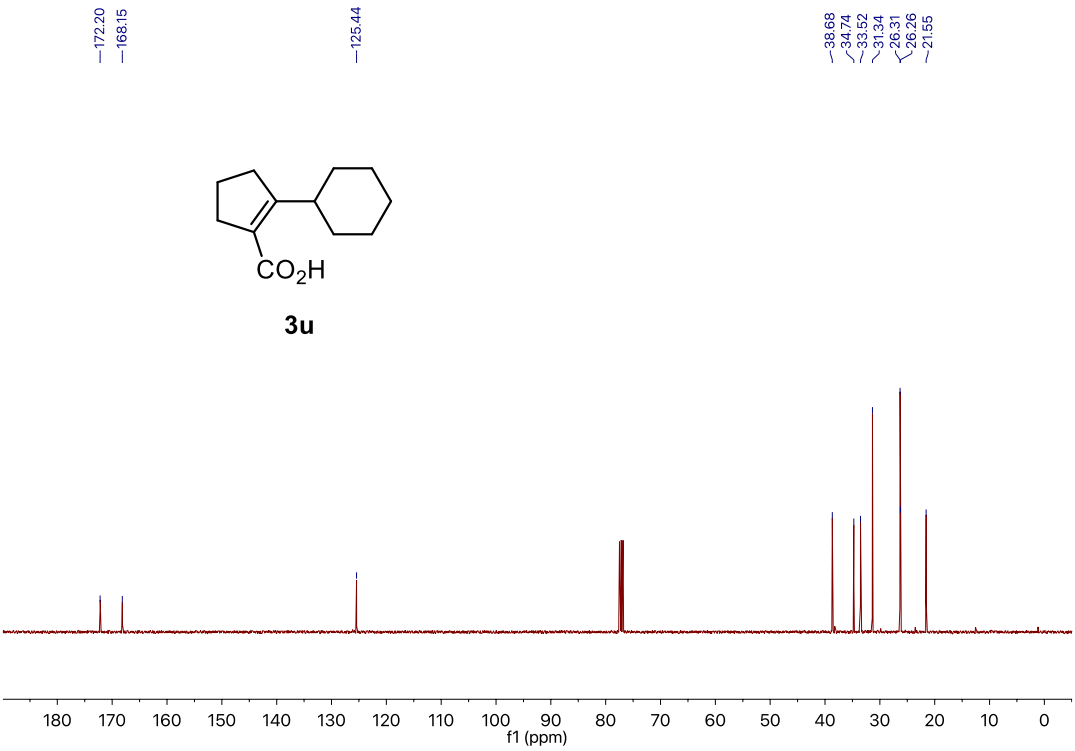
3.824
3.801
3.780
3.759
3.736
2.645
2.626
2.607
2.537
2.518
2.499
1.844
1.825
1.806
1.787
1.685
1.676
1.668
1.662
1.653
1.635
1.623
1.616
1.606
1.391
1.00
2.03
2.06
4.16
4.15
2.13
f1 (ppm)

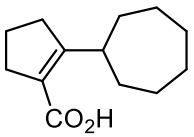

172.20
166.62
126.51
39.80
34.45
33.64
26.00
21.69
f1 (ppm)

CO2H

3t

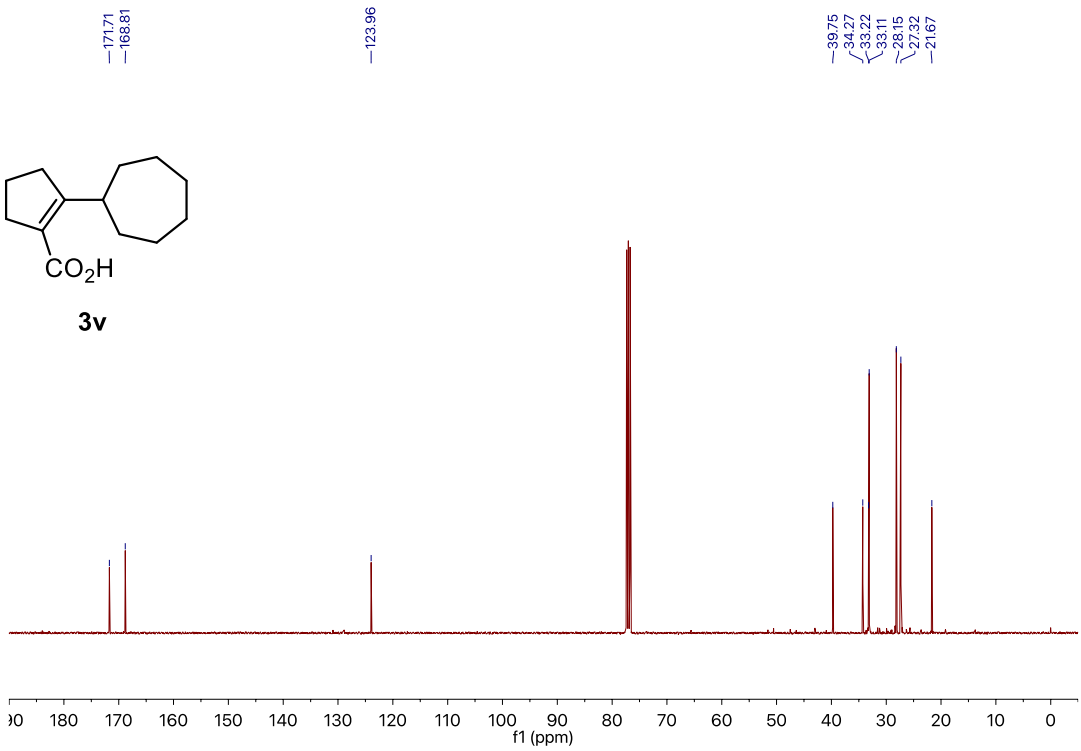

12.485
3.418
3.409
3.400
3.388
3.380
3.371
3.359
3.350
3.342
2.639
2.633
2.628
2.614
2.601
2.595
2.590
2.546
2.535
2.529
2.515
2.510
2.504
2.496
2.491
2.485
1.847
1.822
1.803
1.784
1.765
1.755
1.747
1.717
1.678
1.643
1.615
1.409
1.400
1.375
1.368
1.337
1.306
1.280
1.251
1.223
1.216
1.194
1.184
1.174
1.152
1.135
1.121
1.111
CO2H
3u
0.74
1.00
2.24
2.07
7.35
5.43
f1 (ppm)


172.20
168.15
125.44
38.68
34.74
33.52
31.34
26.31
26.26
21.55
CO2H
3u
f1 (ppm)


CO2H

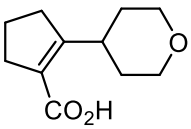
3v

3.594
3.585
3.560
3.534
2.615
2.596
2.577
2.533
2.515
2.496
1.820
1.801
1.782
1.763
1.744
1.692
1.663
1.653
1.645
1.619
1.511
1.496
1.444
1.419
0.98
1.89
1.94
2.11
6.05
4.06
2.23
f1 (ppm)

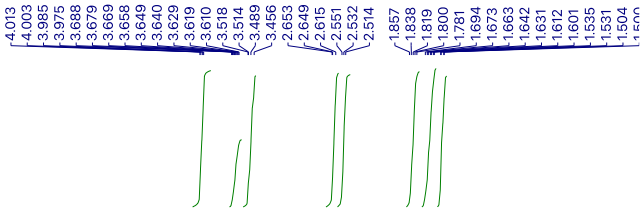

171.71
168.81
123.96
39.75
34.27
33.22
33.11
28.15
27.32
21.67
f1 (ppm)

CO2H

3v



3w


4.013
4.003
3.985
3.975
3.688
3.679
3.669
3.658
3.649
3.640
3.629
3.619
3.610
3.518
3.514
3.489
3.456
2.653
2.649
2.615
2.551
2.532
2.514
1.857
1.838
1.819
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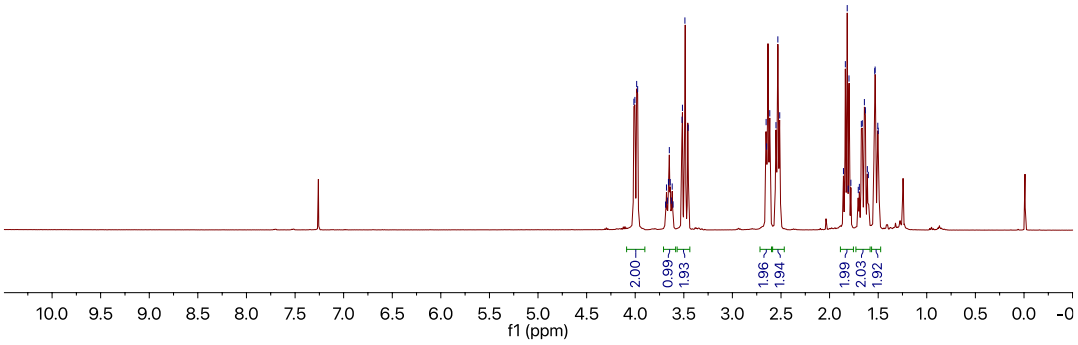
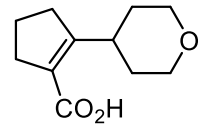
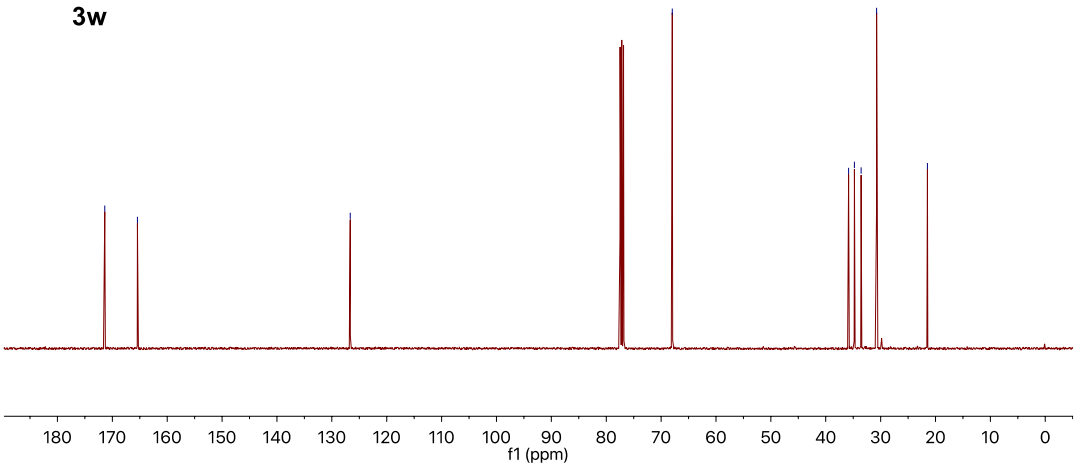
1.612
1.601
1.535
1.531
1.504

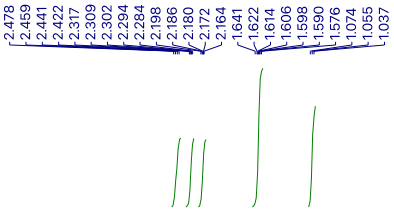

2.00
0.99
1.93
1.96
1.94
1.99
2.03
1.92
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0
f1 (ppm)

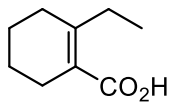

171.38
165.43
126.65
67.96
35.84
34.78
33.55
30.74
21.47



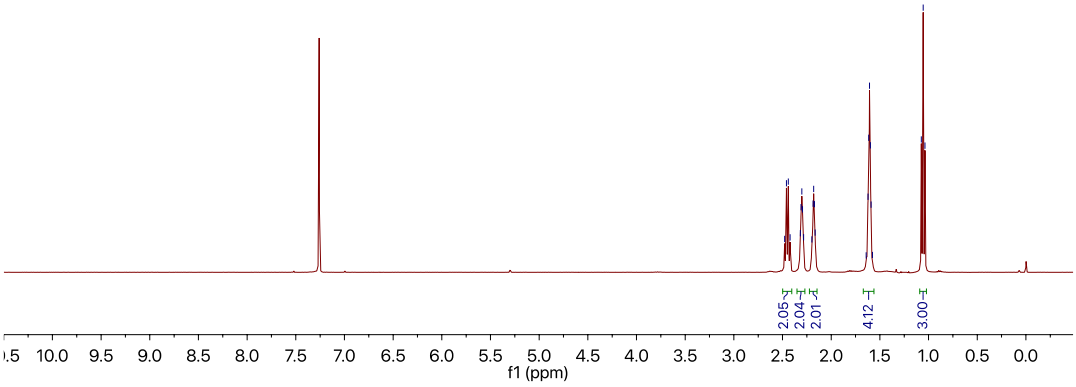
3w


180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

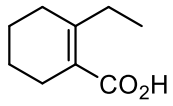

2.478
2.459
2.441
2.422
2.317
2.309
2.302
2.294
2.284
2.198
2.186
2.180
2.172
2.164
1.641
1.622
1.614
1.606
1.598
1.590
1.576
1.074
1.055
1.037


CO2H

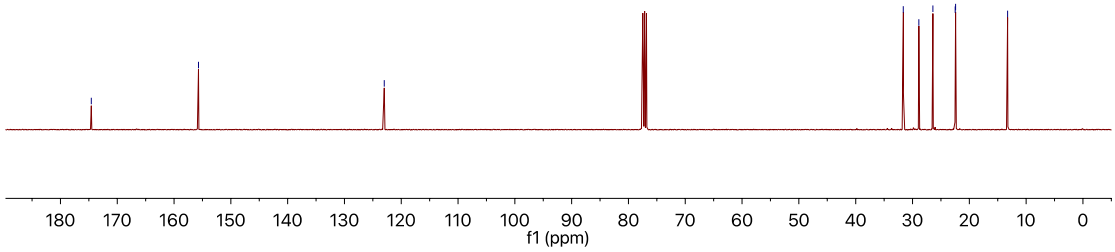
3x

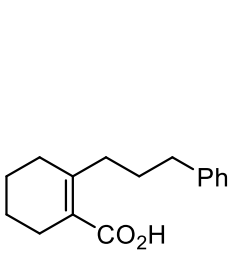

2.05
2.04
2.01
4.12
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


174.56
155.70
122.99
31.61
28.88
26.41
22.49
22.41
13.25


CO2H

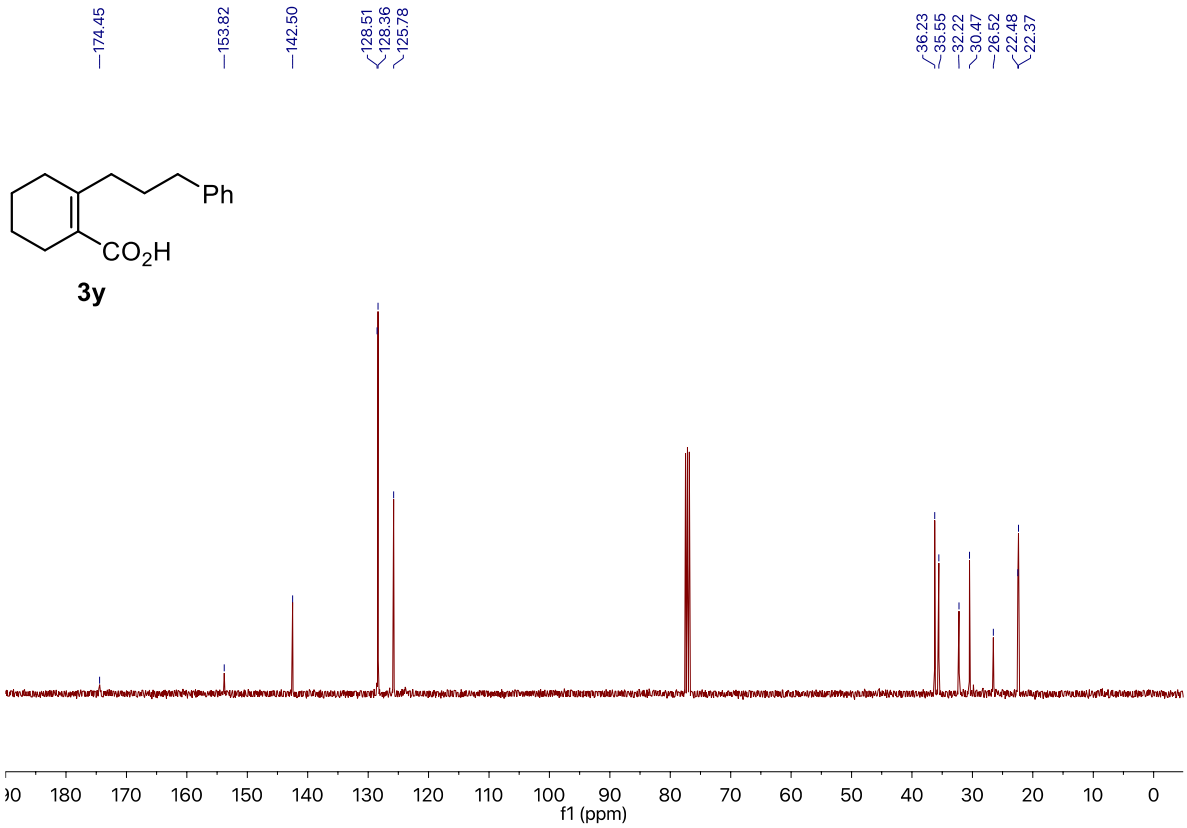
3x


180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


Ph
CO2H

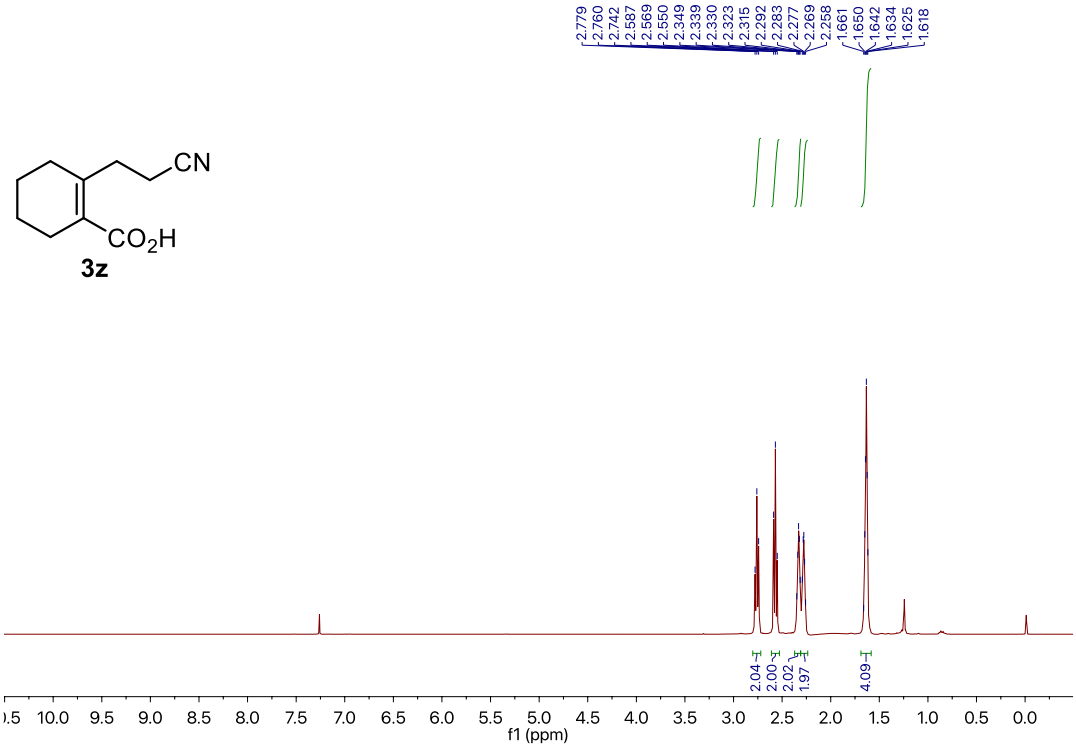
3y

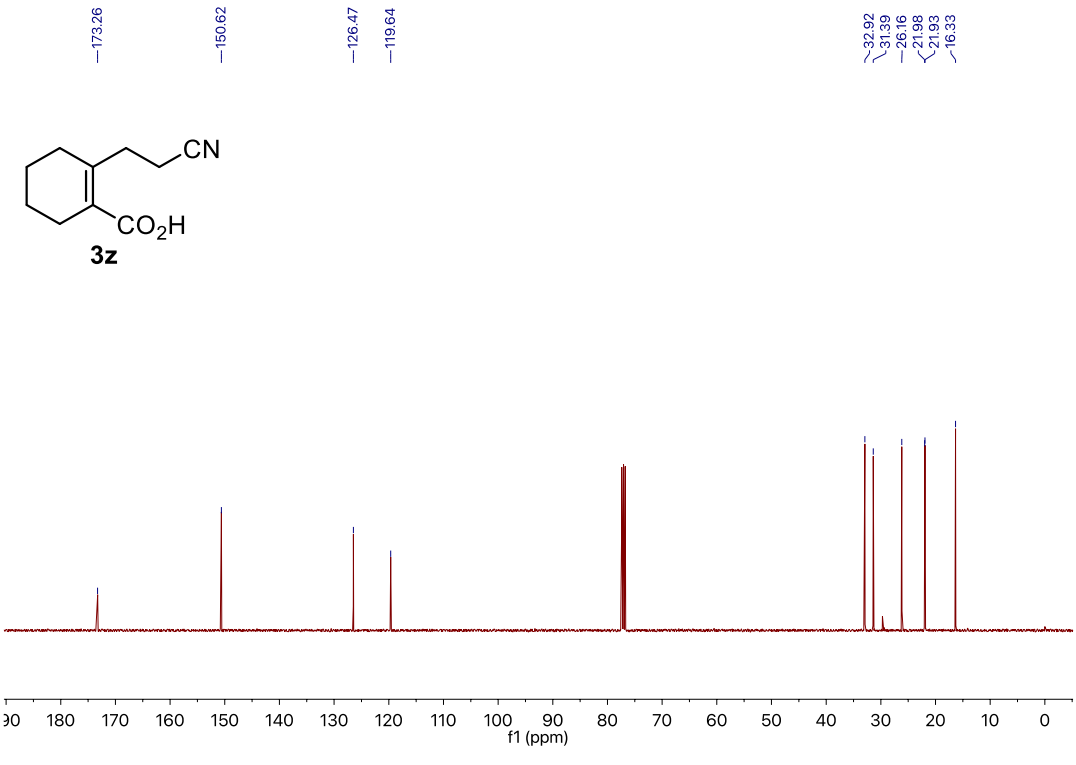
7.276
7.258
7.239
7.193
7.174
7.154
7.136
2.667
2.647
2.628
2.515
2.496
2.476
2.327
2.313
2.298
2.174
2.159
2.147
1.829
1.810
1.790
1.770
1.751
1.591
5.20
2.00
1.94
1.93
1.94
1.97
3.98
f1 (ppm)

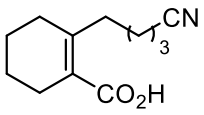

174.45
153.82
142.50
128.51
128.36
125.78
36.23
35.55
32.22
30.47
26.52
22.48
22.37
f1 (ppm)

Ph
CO2H

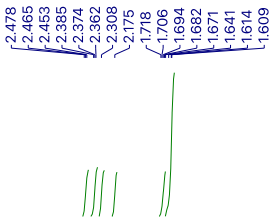
3y

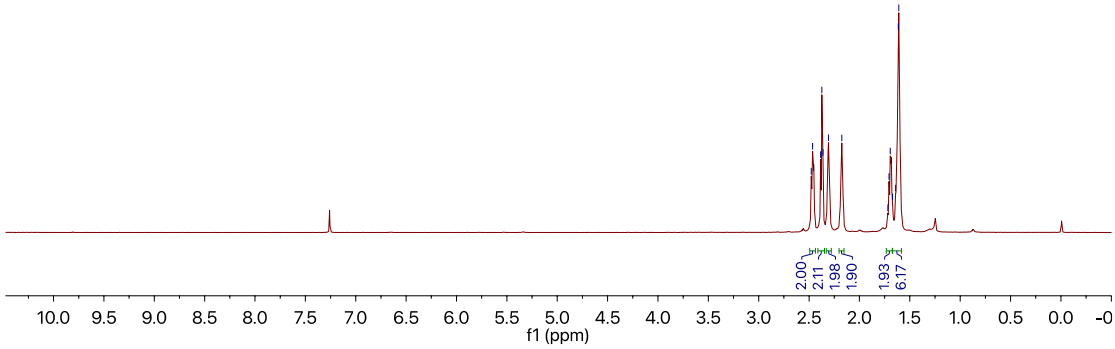

2.779
2.760
2.742
2.587
2.569
2.550
2.349
2.339
2.330
2.323
2.315
2.292
2.283
2.277
2.269
2.258
1.661
1.650
1.642
1.634
1.625
1.618
CN
CO2H
3z
2.04
2.00
2.02
1.97
4.09
f1 (ppm)

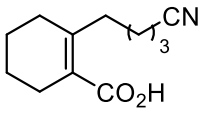

173.26
150.62
126.47
119.64
32.92
31.39
26.16
21.98
21.93
16.33
CN
CO2H
3z
f1 (ppm)


CN
3
CO2H

3aa

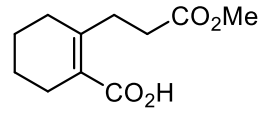

2.478
2.465
2.453
2.385
2.374
2.362
2.308
2.175
1.718
1.706
1.694
1.682
1.671
1.641
1.614
1.609


2.00
2.11
1.98
1.90
1.93
6.17
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0
f1 (ppm)


CN
3
CO2H

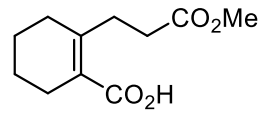
3aa

173.81
153.04
124.26
119.95
34.66
32.20
27.65
26.46
25.46
22.37
22.28
17.05
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


CO2Me
CO2H

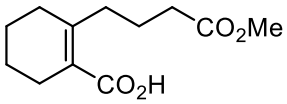
3ab

3.679
2.722
2.709
2.695
2.518
2.505
2.492
2.314
2.307
2.297
2.170
2.163
2.158
2.153
2.148
1.606
2.70
2.11
2.11
2.07
2.10
4.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0
f1 (ppm)


CO2Me
CO2H

3ab

174.04
173.08
150.33
125.38
51.75
32.67
31.64
30.79
26.37
22.13
90 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


CO2Me
CO2H

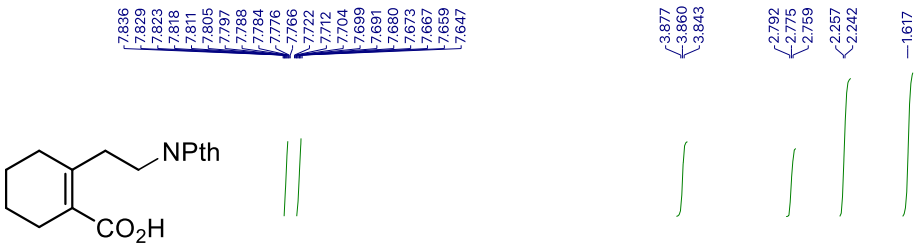
3ac

3.667
2.488
2.468
2.448
2.362
2.343
2.324
2.308
2.300
2.298
2.285
2.188
2.181
2.173
2.170
2.164
1.814
1.800
1.795
1.776
1.621
1.613
1.605
1.596
2.78
2.00
3.97
2.09
1.98
4.17
f1 (ppm)

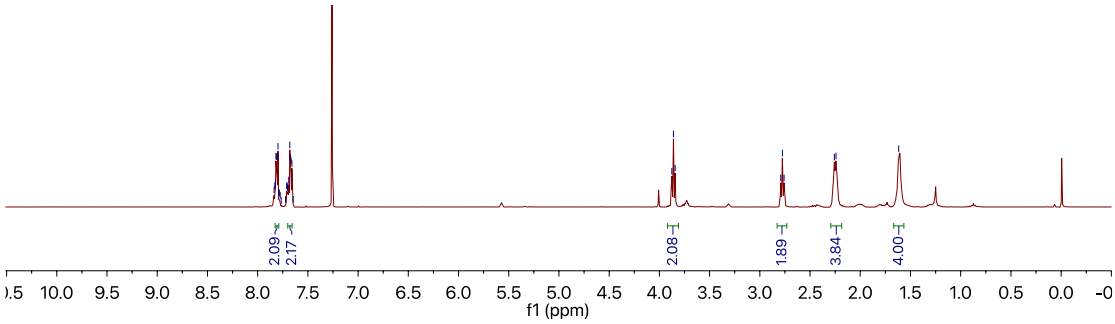

CO2Me
CO2H

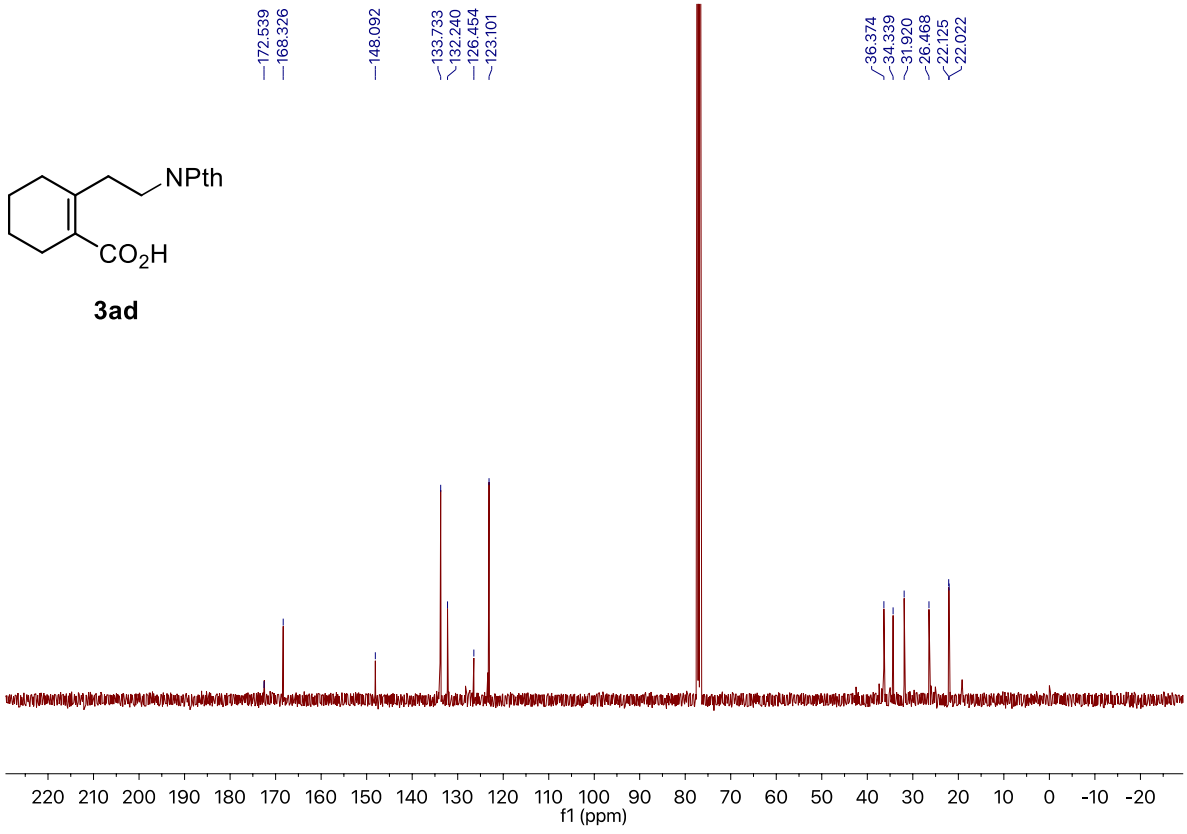
3ac

174.22
173.65
152.82
124.32
51.64
34.86
34.00
32.01
26.51
23.89
22.41
22.30
f1 (ppm)

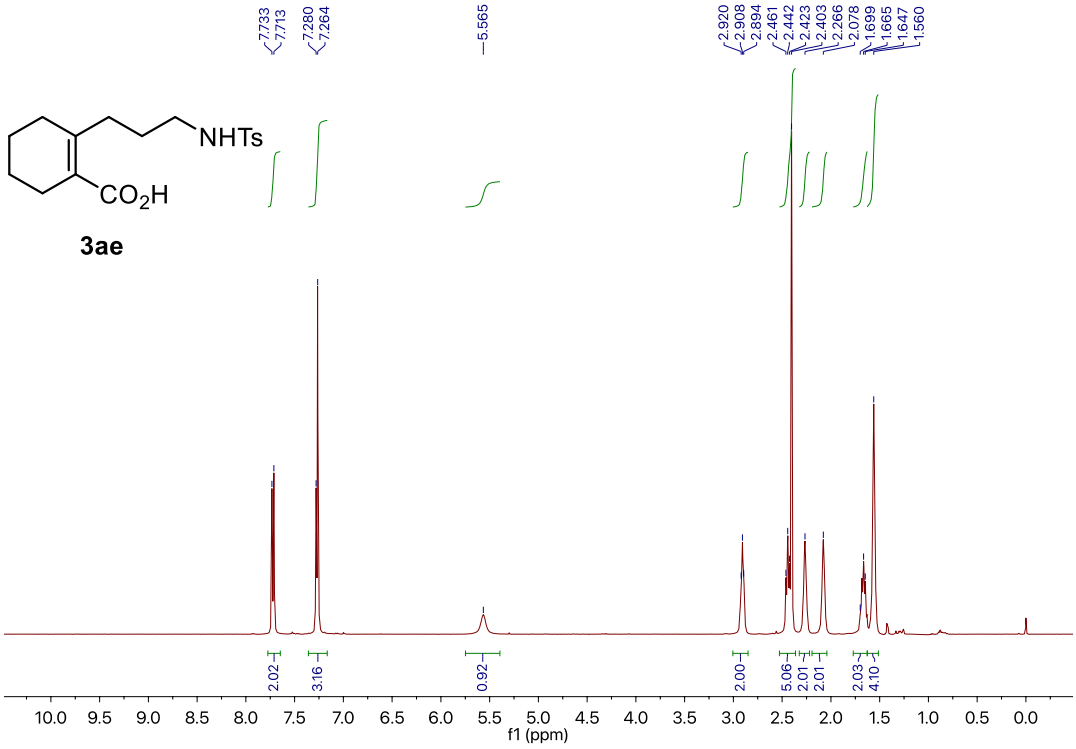

7.836
7.829
7.823
7.818
7.811
7.805
7.797
7.788
7.784
7.776
7.766
7.722
7.712
7.704
7.699
7.691
7.680
7.673
7.667
7.659
7.647
3.877
3.860
3.843
2.792
2.775
2.759
2.257
2.242
1.617
NPth
CO2H

3ad

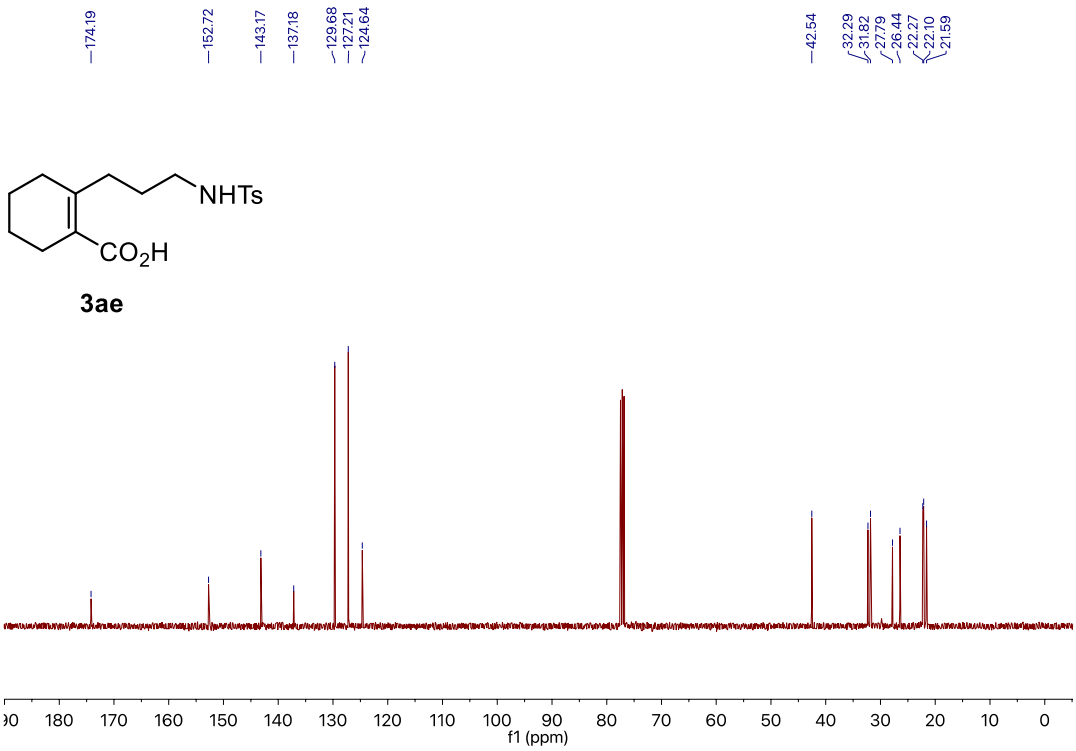

2.09
2.17
2.08
1.89
3.84
4.00
f1 (ppm)


172.539
168.326
148.092
133.733
132.240
126.454
123.101
36.374
34.339
31.920
26.468
22.125
22.022
NPth
CO2H
f1 (ppm)

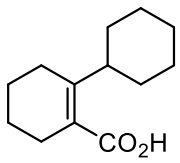
3ad


7.733
7.713
7.280
7.264
5.565
2.920
2.908
2.894
2.461
2.442
2.423
2.403
2.266
2.078
1.699
1.665
1.647
1.560
NHTs
CO2H
2.02
3.16
0.92
2.00
5.06
2.01
2.01
2.03
4.10
f1 (ppm)

3ae

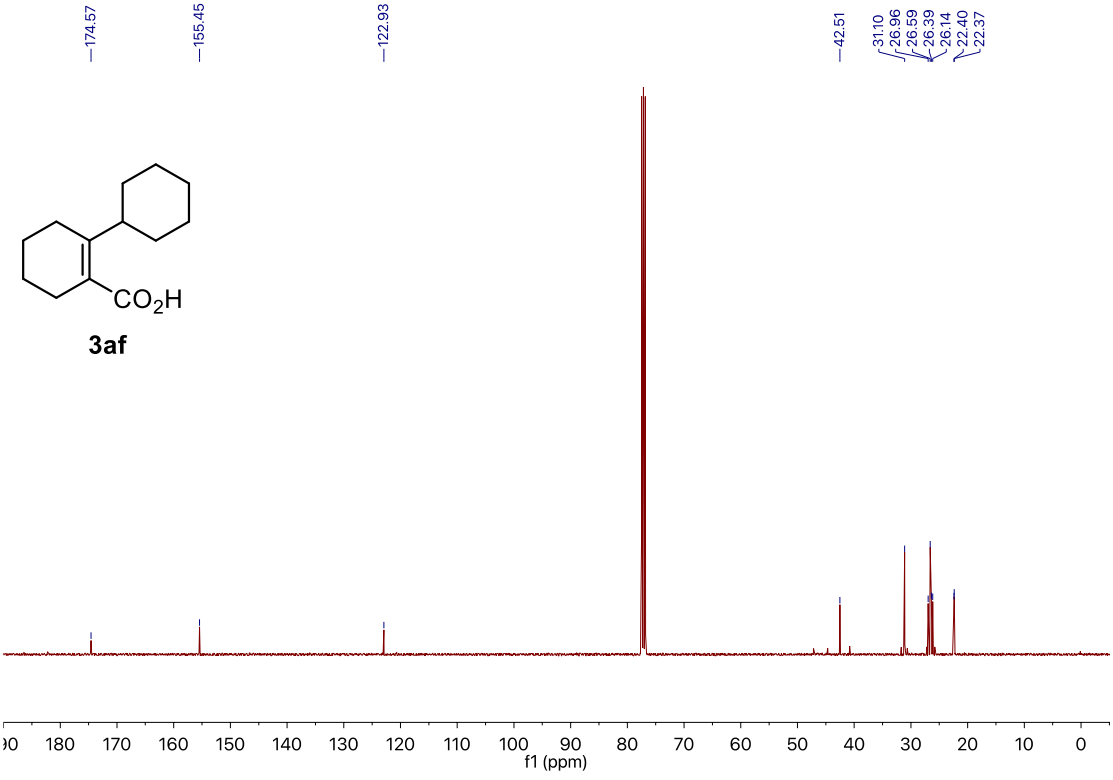

174.19
152.72
143.17
137.18
129.68
127.21
124.64
42.54
32.29
31.82
27.79
26.44
22.27
22.10
21.59
NHTs
CO2H
f1 (ppm)

3ae


CO2H

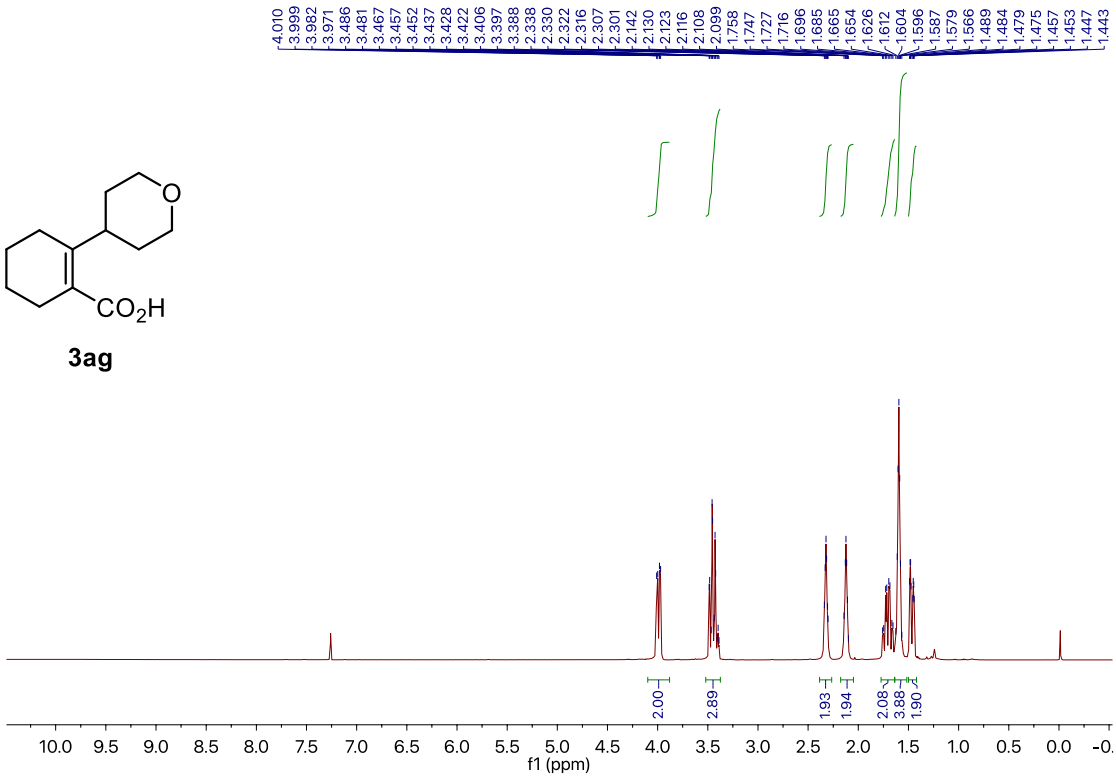
3af

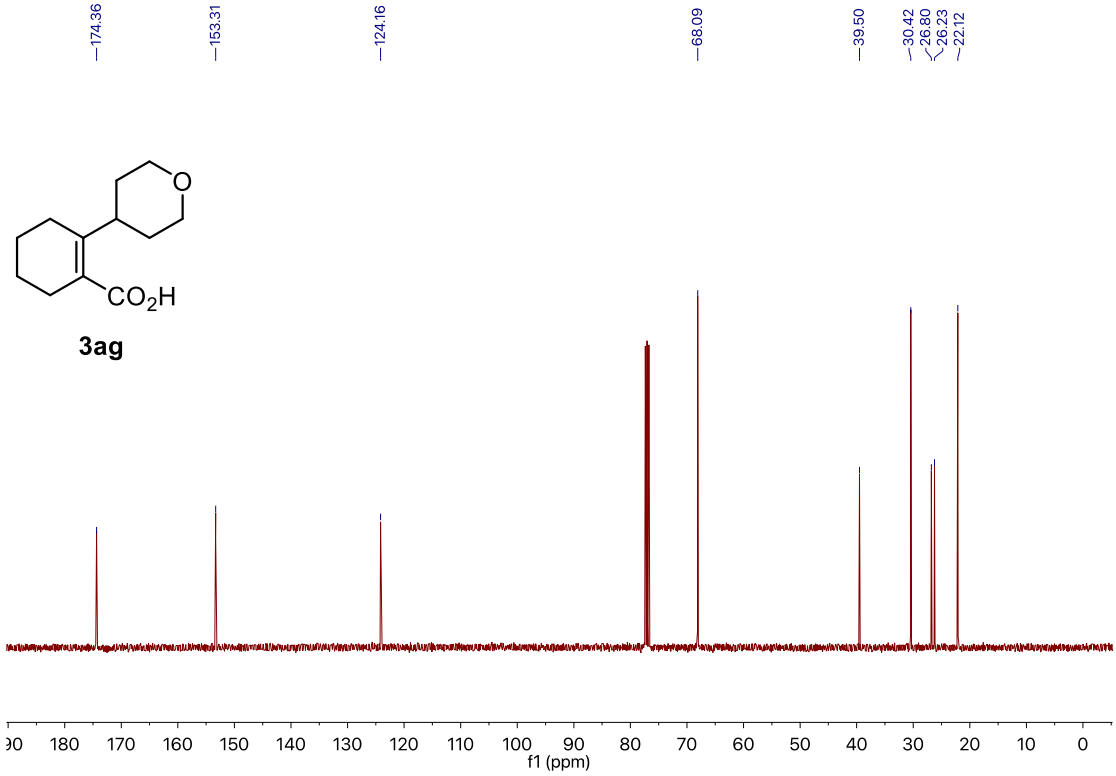
3.145
3.118
3.098
2.309
2.116
1.721
1.688
1.656
1.590
1.341
1.317
1.297
1.273
1.254
1.241
1.176
1.145
1.107
0.93
1.72
1.70
8.27
6.00
f1 (ppm)

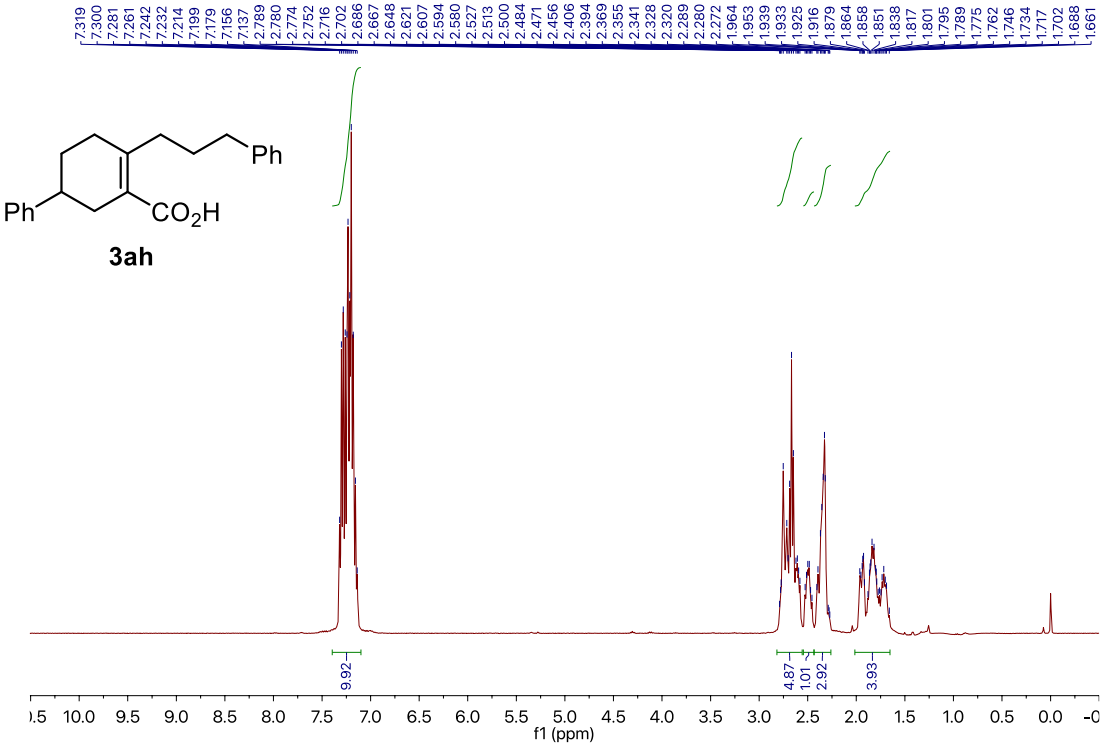

174.57
155.45
122.93
42.51
31.10
26.96
26.59
26.39
26.14
22.40
22.37
f1 (ppm)

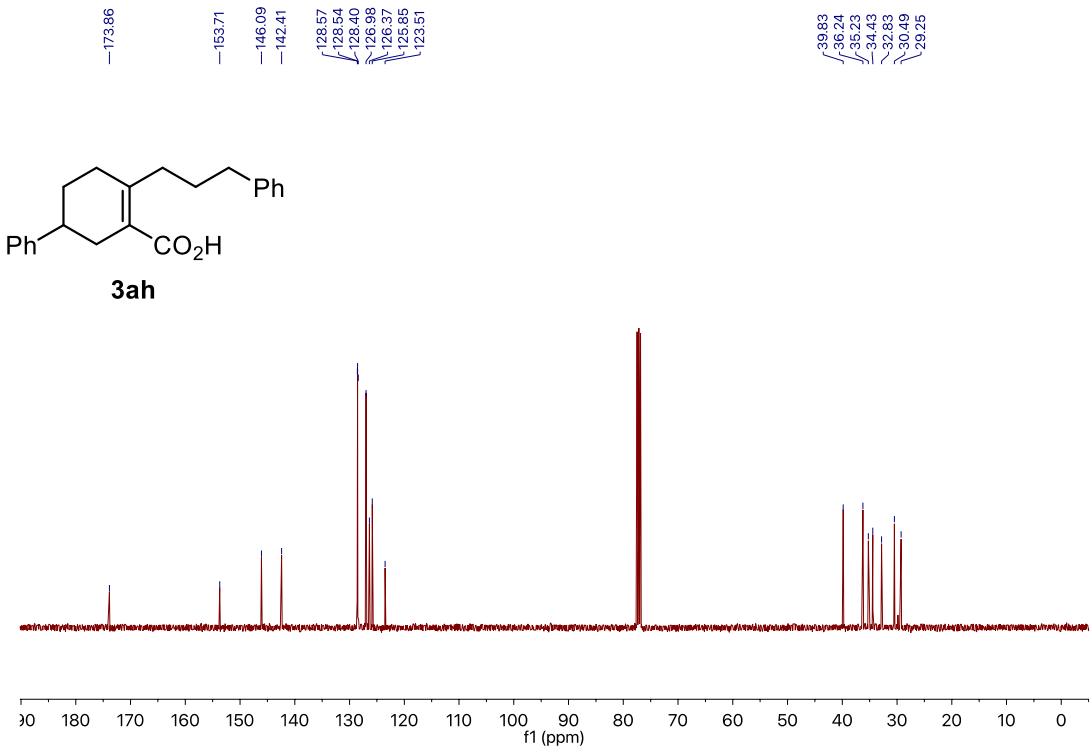
CO2H

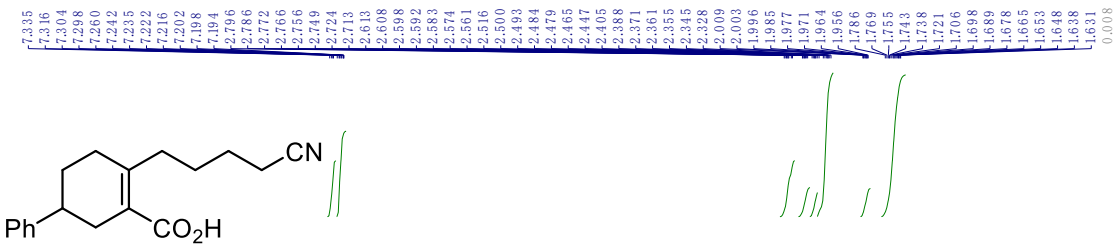
3af


4.010
3.999
3.982
3.971
3.486
3.481
3.467
3.457
3.452
3.437
3.428
3.422
3.406
3.397
3.388
2.338
2.330
2.322
2.316
2.307
2.301
2.142
2.130
2.123
2.116
2.108
2.099
1.758
1.747
1.727
1.716
1.696
1.685
1.665
1.654
1.626
1.612
1.604
1.596
1.587
1.579
1.566
1.489
1.484
1.479
1.475
1.457
1.453
1.447
1.443
O
CO2H
3ag
2.00
2.89
1.93
1.94
2.08
3.88
1.90
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

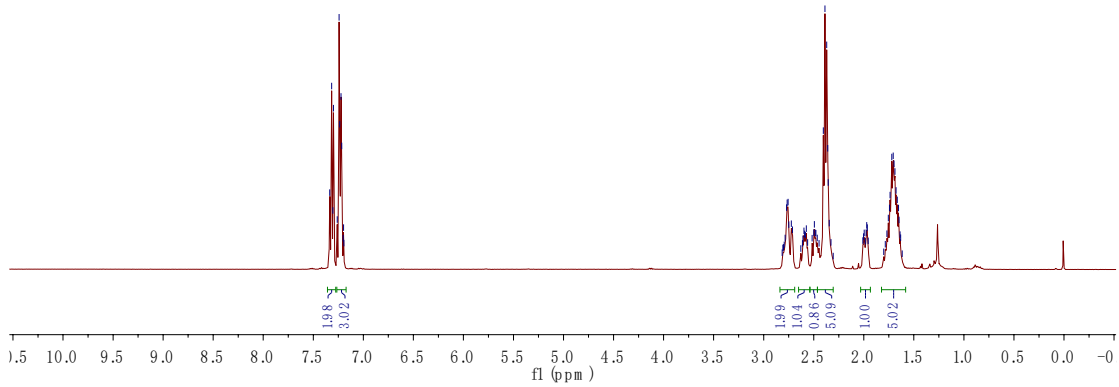

174.36
153.31
124.16
68.09
39.50
30.42
26.80
26.23
22.12
O
CO2H
3ag
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


Ph
Ph
CO2H
3ah
9.92
4.87
1.01
2.92
3.93
f1 (ppm)

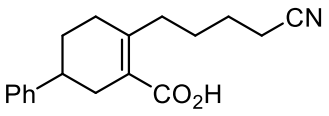

Ph
Ph
CO2H
3ah
f1 (ppm)


CN
Ph
CO2H

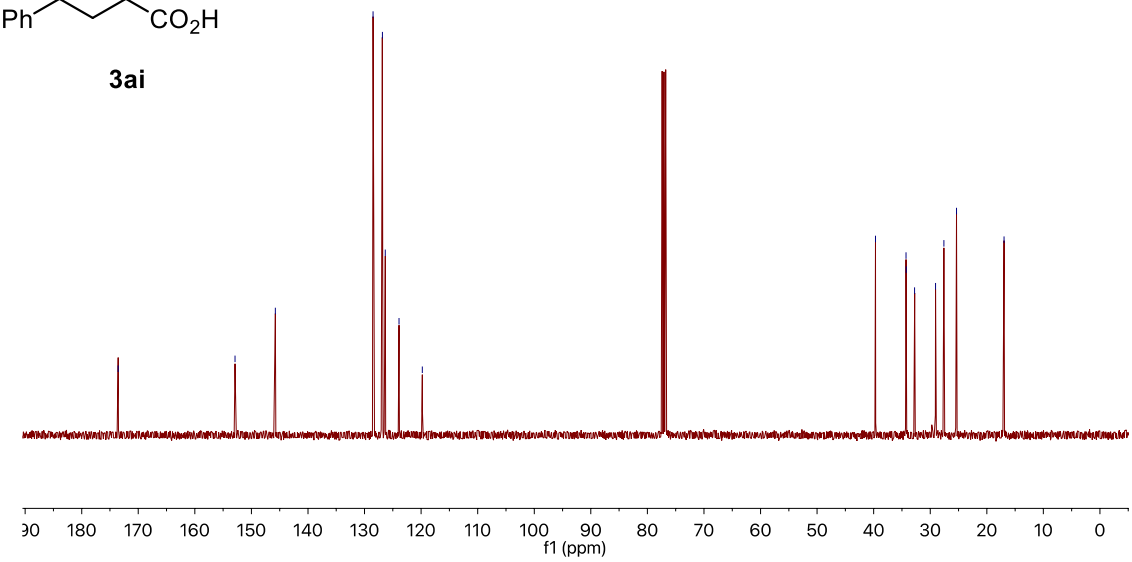
3ai

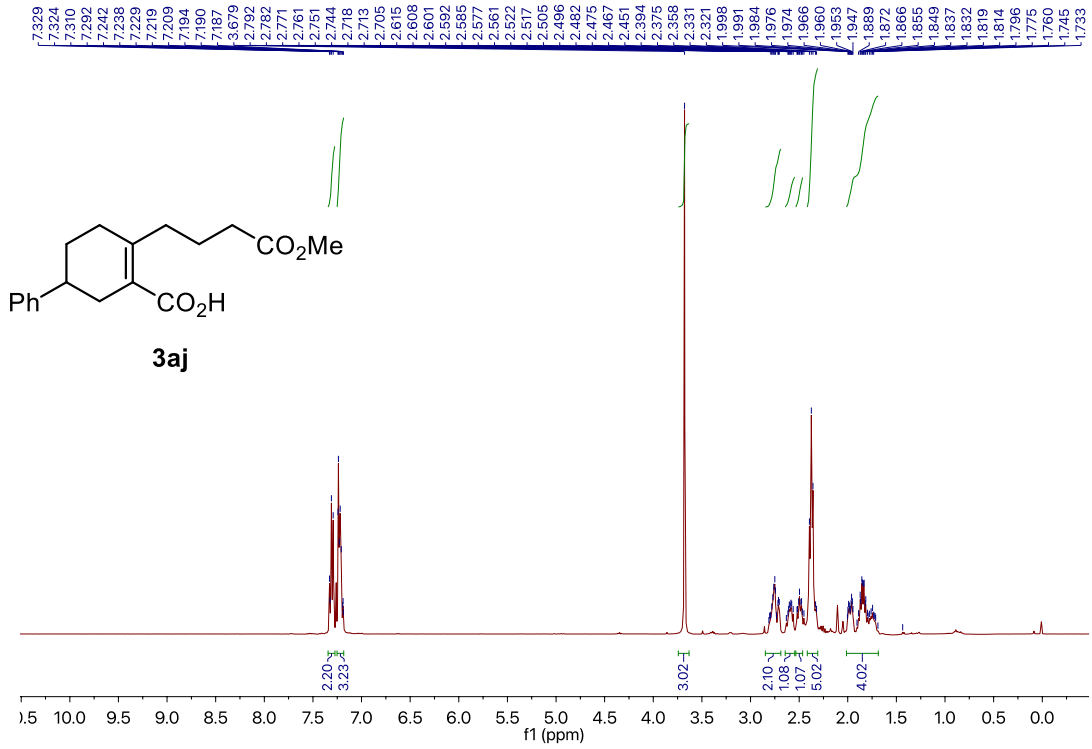

1.98
3.02
1.99
1.04
0.86
5.09
1.00
5.02
f1 (ppm)


173.56
152.89
145.77
128.50
126.85
126.34
123.91
119.79
39.68
34.29
34.25
32.76
29.06
27.58
25.35
16.95


CN
Ph
CO2H

3ai


f1 (ppm)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7.324
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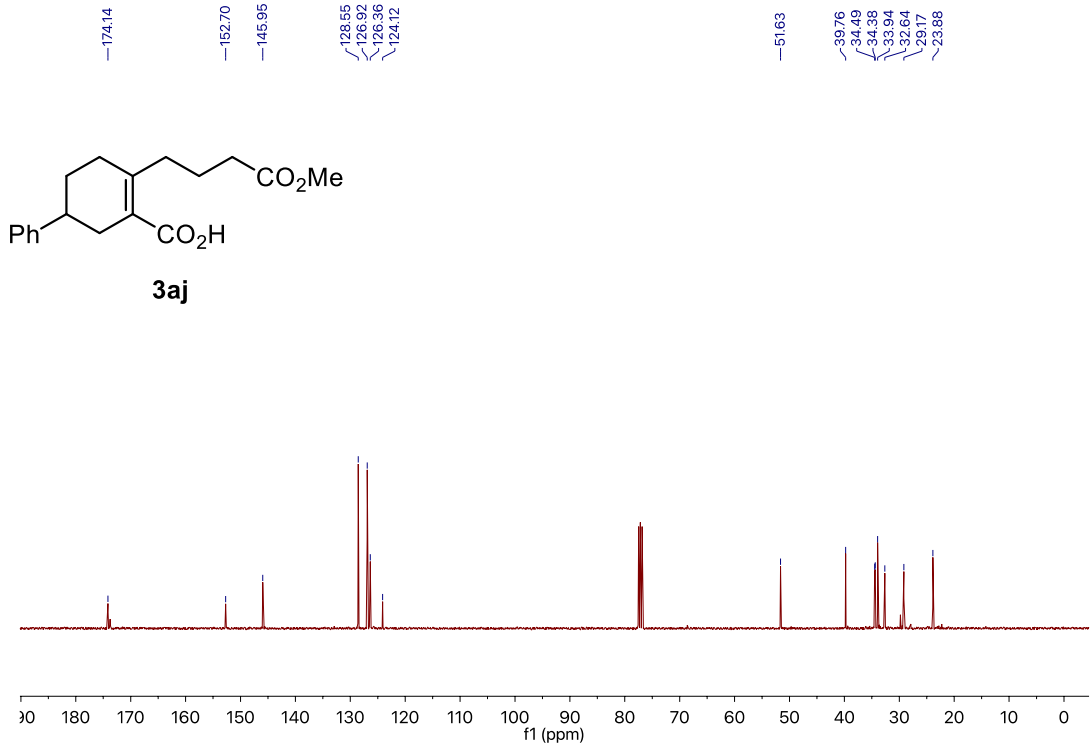
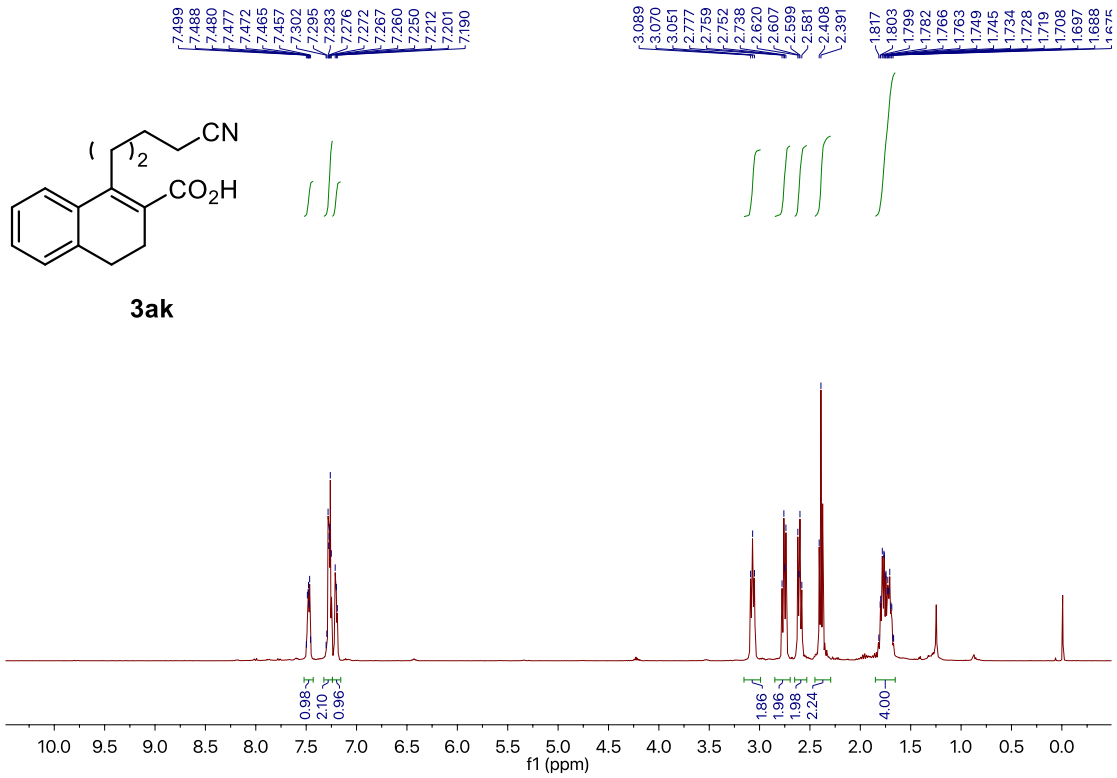
7.292
7.242
7.238
7.229
7.219
7.209
7.194
7.190
7.187
3.679
2.792
2.782
2.771
2.761
2.751
2.744
2.718
2.713
2.705
2.615
2.608
2.601
2.592
2.585
2.577
2.561
2.522
2.517
2.505
2.496
2.482
2.475
2.467
2.451
2.394
2.375
2.358
2.331
2.321
1.998
1.991
1.984
1.976
1.974
1.966
1.960
1.953
1.947
1.889
1.872
1.866
1.855
1.849
1.837
1.832
1.819
1.814
1.796
1.775
1.760
1.745
1.733
CO2Me
Ph
CO2H
3aj
2.20
3.23
3.02
2.10
1.08
1.07
5.02
4.02
f1 (ppm)


174.14
152.70
145.95
128.55
126.92
126.36
124.12
51.63
39.76
34.49
34.38
33.94
32.64
29.17
23.88
CO2Me
Ph
CO2H
3aj
f1 (ppm)


7.499
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7.472
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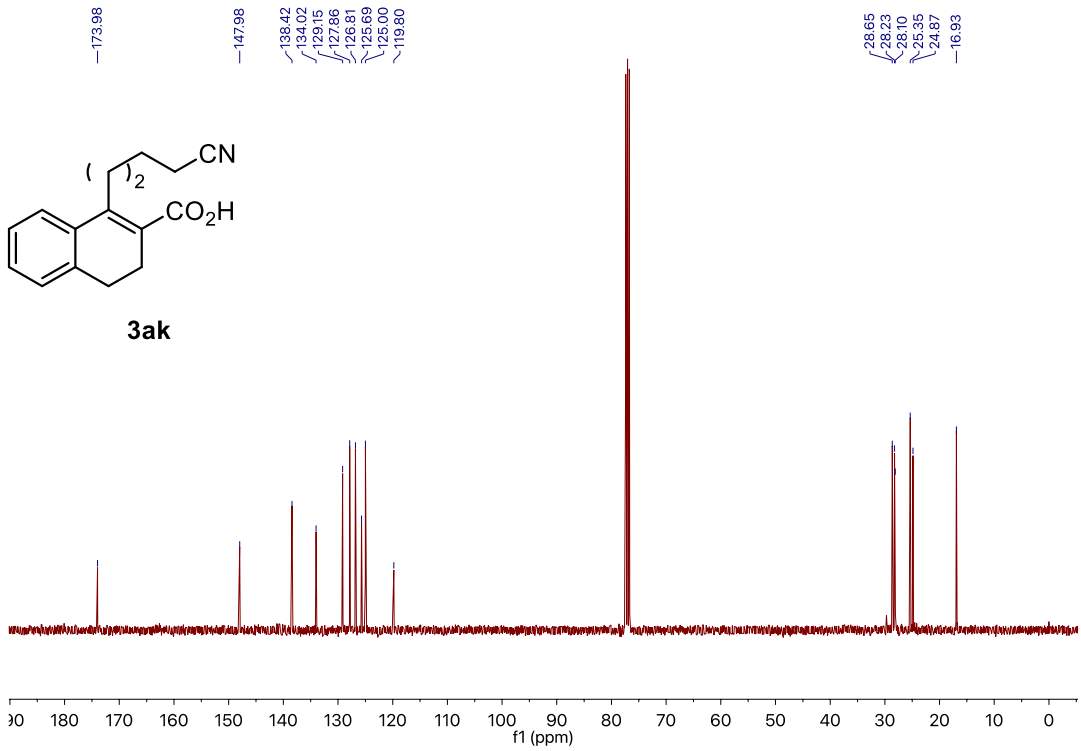
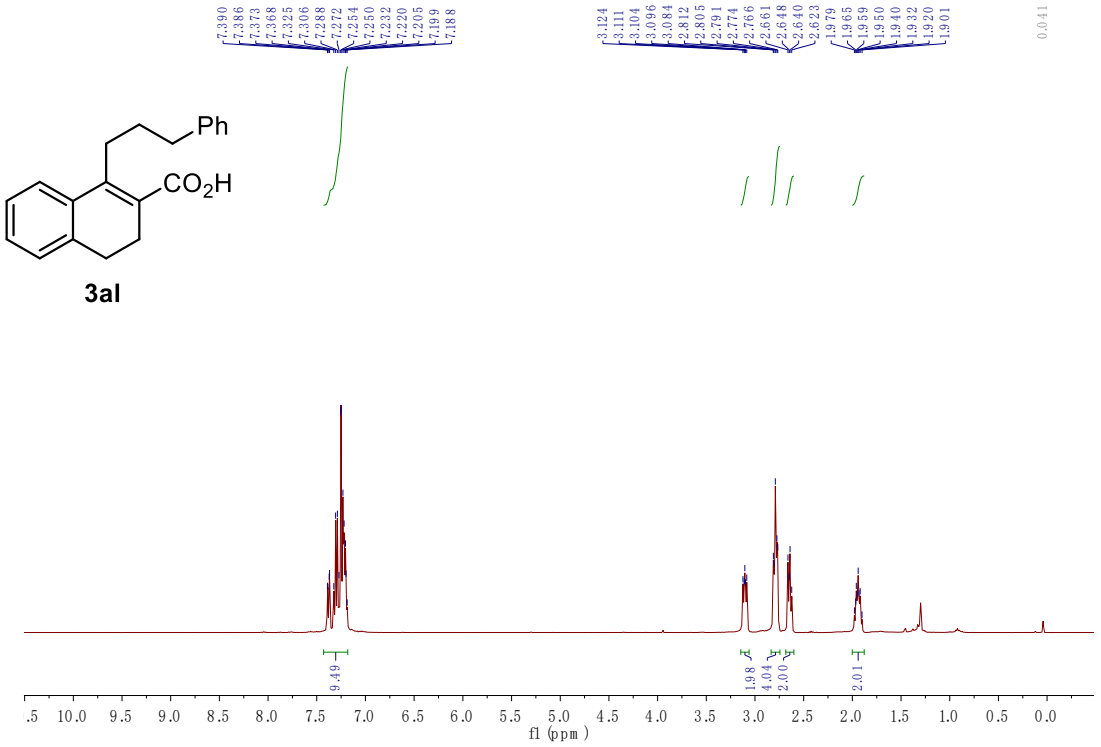
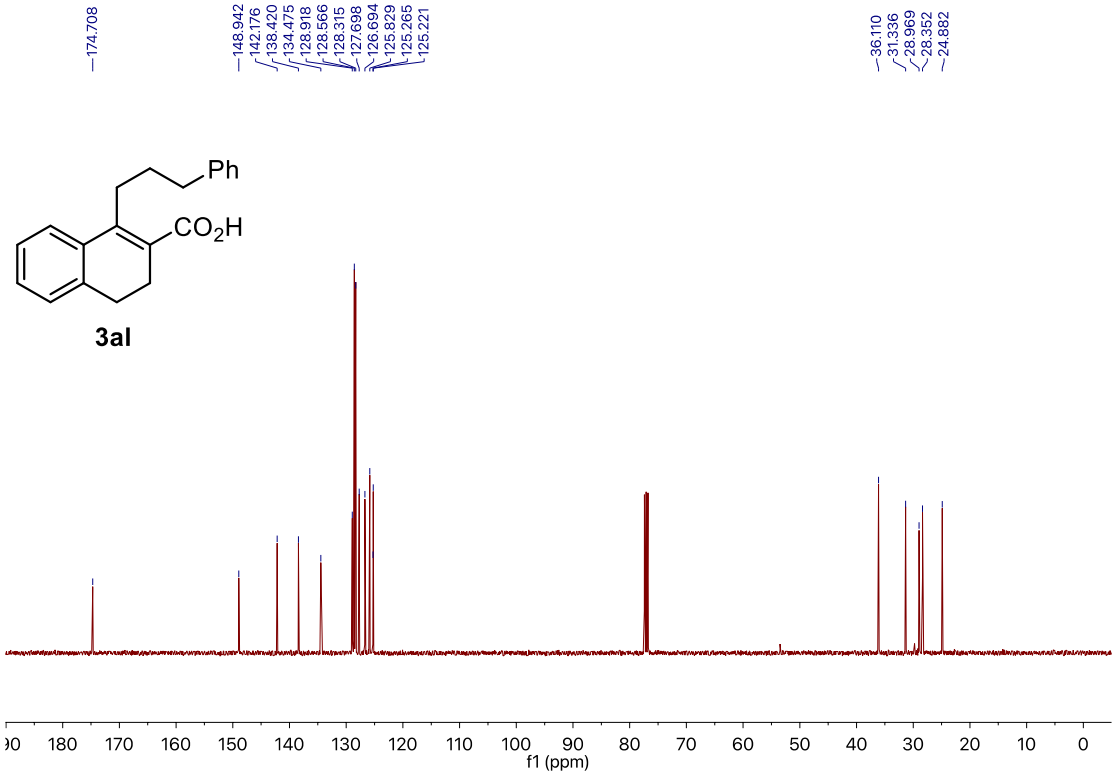
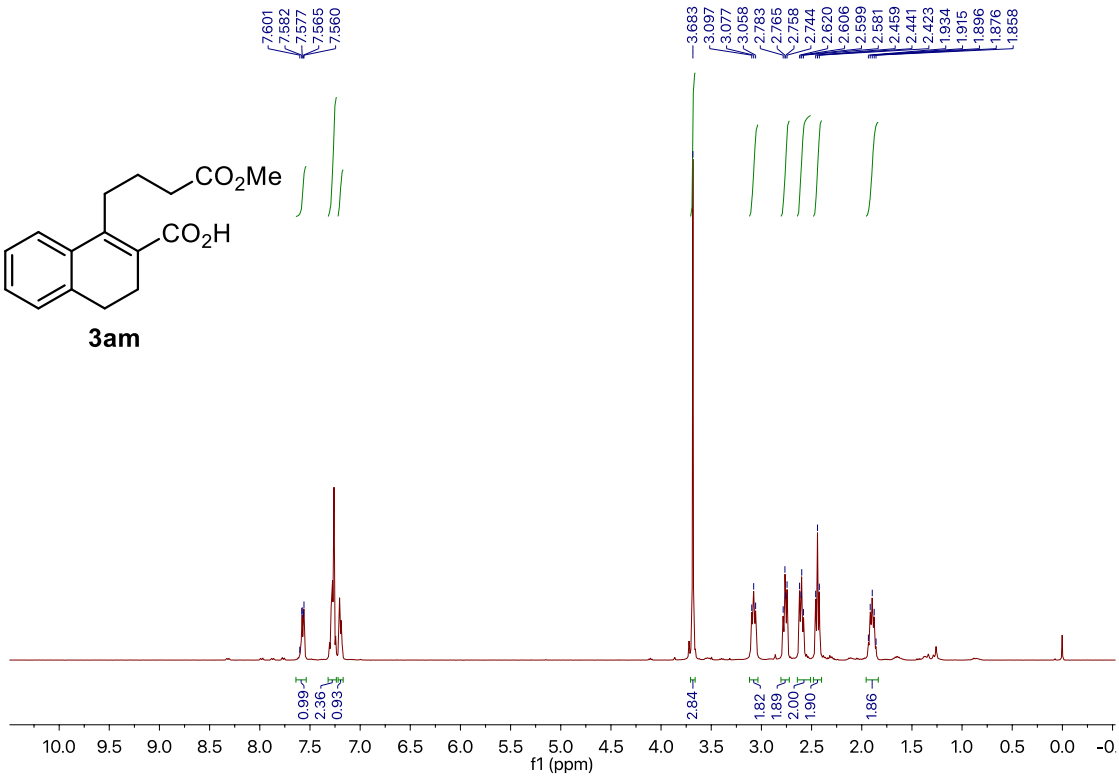
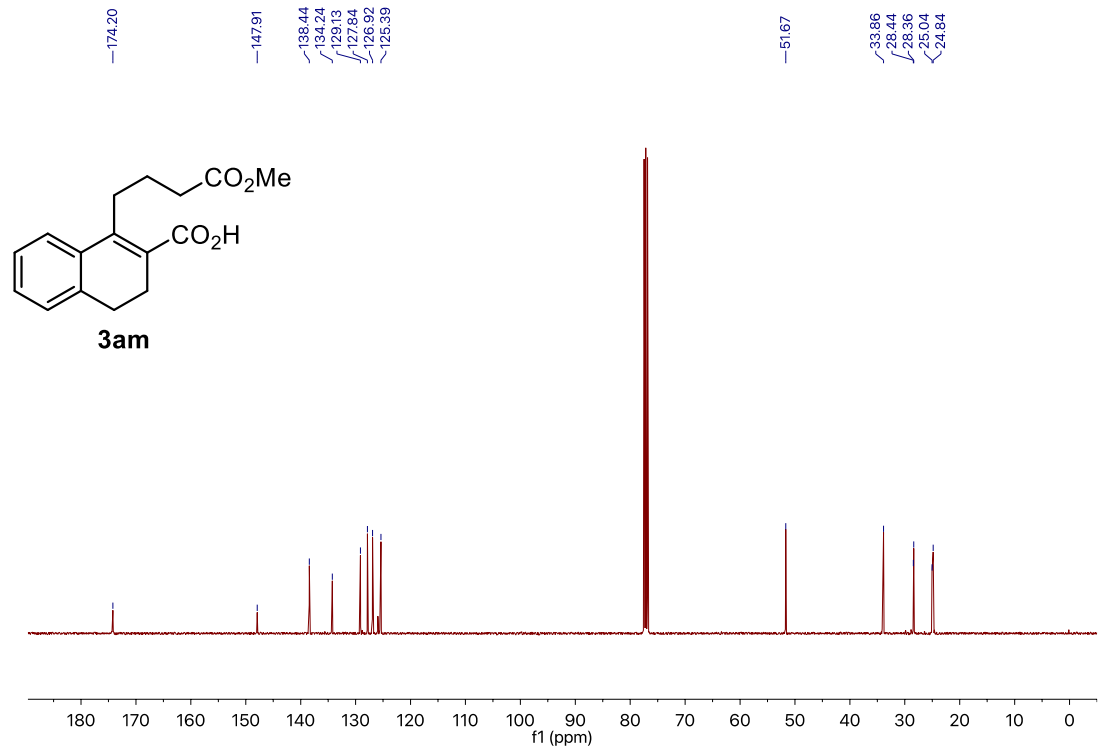
7.457
7.302
7.295
7.283
7.276
7.272
7.267
7.260
7.250
7.212
7.201
7.190
3.089
3.070
3.051
2.777
2.759
2.752
2.738
2.620
2.607
2.599
2.581
2.408
2.391
1.817
1.803
1.799
1.782
1.766
1.763
1.749
1.745
1.734
1.728
1.719
1.708
1.697
1.688
CN
CO2H
3ak
0.98
2.10
0.96
1.86
1.96
1.98
2.24
4.00
f1 (ppm)

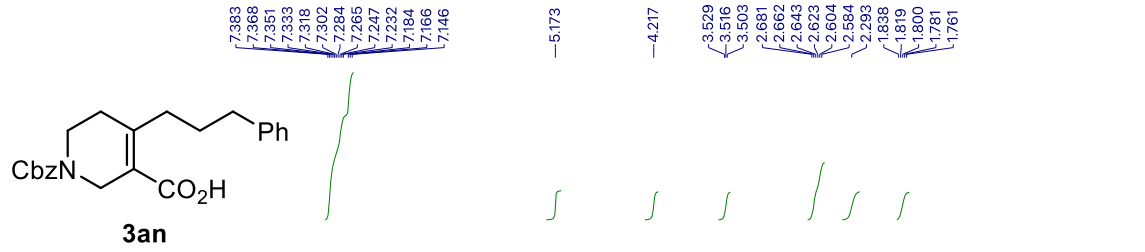

173.98
147.98
138.42
134.02
129.15
127.86
126.81
125.69
125.00
119.80
28.65
28.23
28.10
25.35
24.87
16.93
CN
CO2H
3ak
f1 (ppm)


Ph
CO2H
3al
7.390
7.386
7.373
7.368
7.325
7.306
7.288
7.272
7.254
7.250
7.232
7.220
7.205
7.199
7.188
3.124
3.111
3.104
3.096
3.084
2.812
2.805
2.791
2.774
2.766
2.661
2.648
2.640
2.623
1.979
1.965
1.959
1.950
1.940
1.932
1.920
1.901
0.041
9.49
1.98
4.04
2.00
2.01
f1 (ppm)


Ph
CO2H
3al
174.708
148.942
142.176
138.420
134.475
128.918
128.566
128.315
127.698
126.694
125.829
125.265
125.221
36.110
31.336
28.969
28.352
24.882
f1 (ppm)


7.601
7.582
7.577
7.565
7.560
3.683
3.097
3.077
3.058
2.783
2.765
2.758
2.744
2.620
2.606
2.599
2.581
2.459
2.441
2.423
1.934
1.915
1.896
1.876
1.858
CO2Me
CO2H
3am
0.99
2.36
0.93
2.84
1.82
1.89
2.00
1.90
1.86
f1 (ppm)


174.20
147.91
138.44
134.24
129.13
127.84
126.92
125.39
51.67
33.86
28.44
28.36
25.04
24.84
CO2Me
CO2H
3am
f1 (ppm)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7.232
7.184
7.166
7.146
5.173
4.217
3.529
3.516
3.503
2.681
2.662
2.643
2.623
2.604
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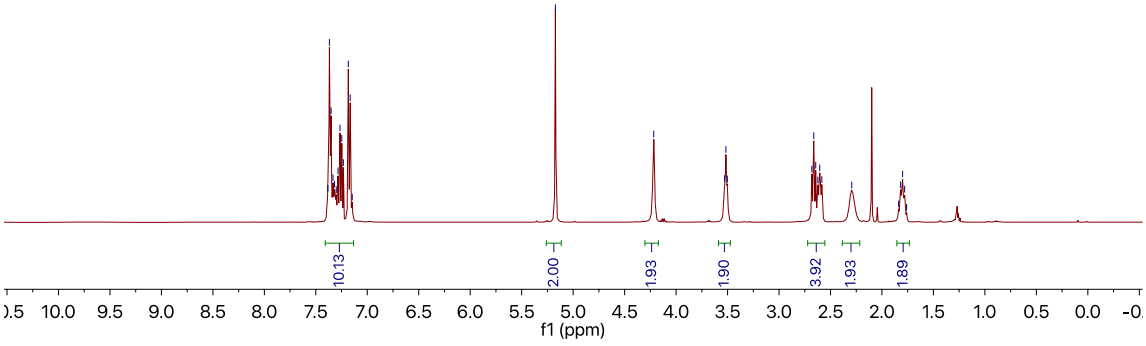
2.293
1.838
1.819
1.800
1.781
1.761
Ph
CbzN
CO2H

3an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2.00
1.93
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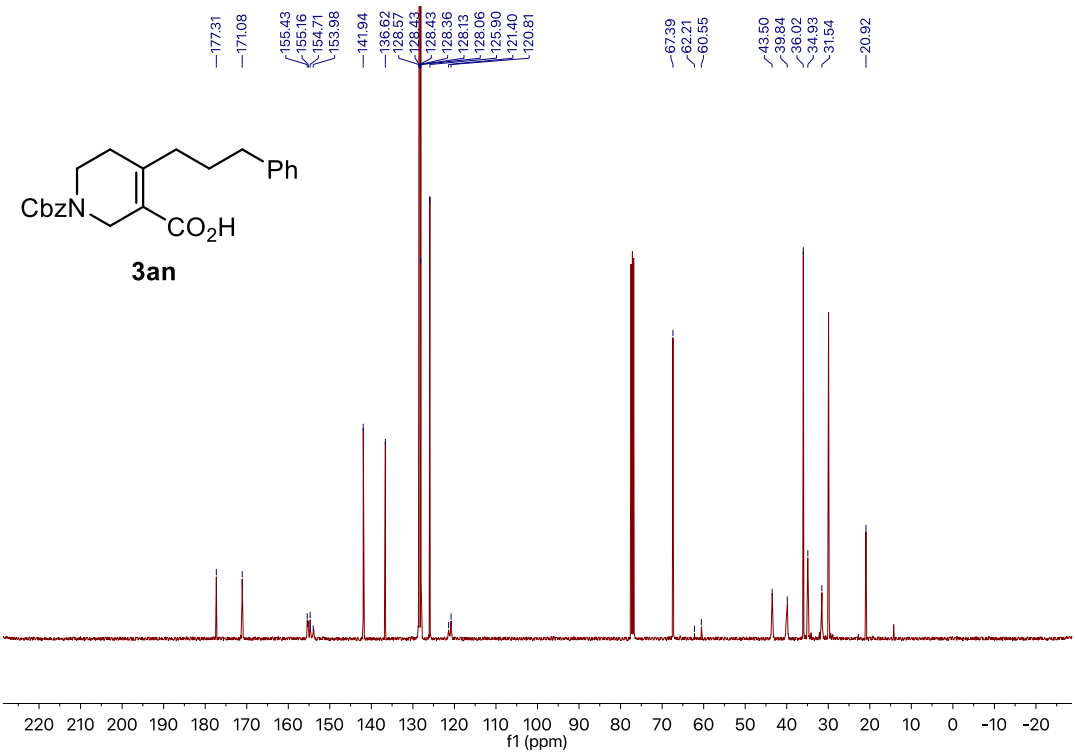
3.92
1.93
1.89
f1 (ppm)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171.08
155.43
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154.71
153.98
141.94
136.62
128.57
128.43
128.43
128.36
128.13
128.06
125.90
121.40
120.81
67.39
62.21
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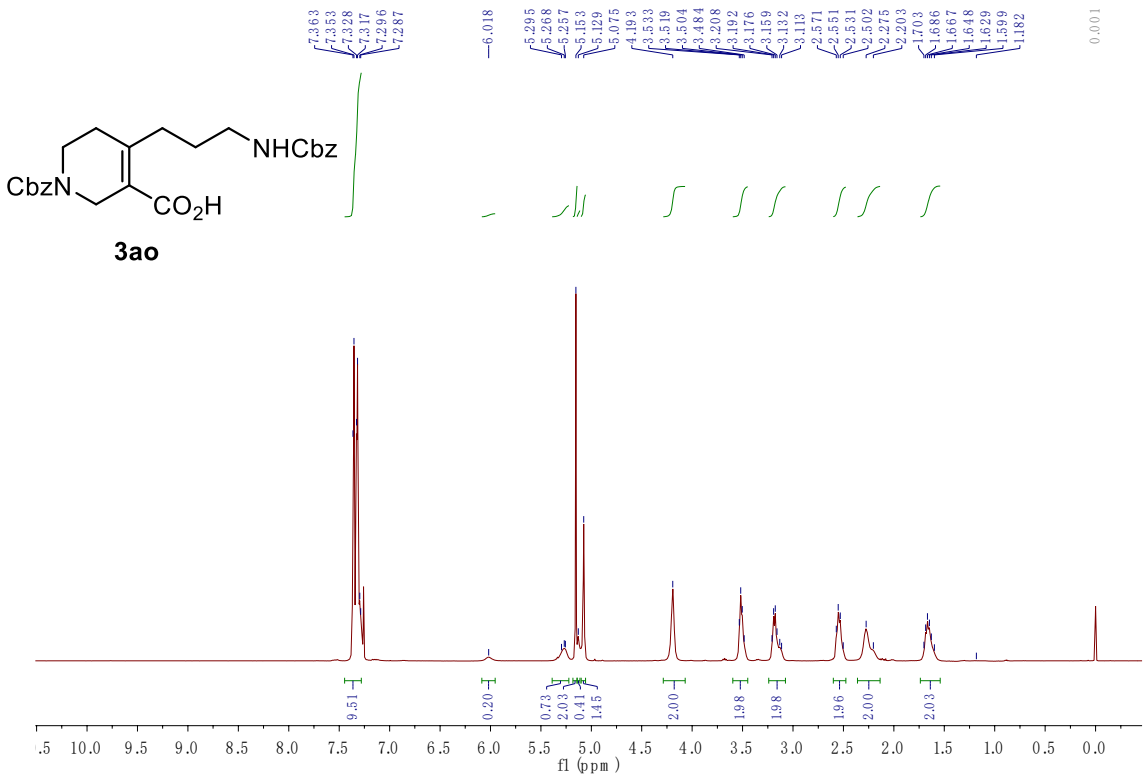
43.50
39.84
36.02
34.93
31.54
20.92
Ph
CbzN
CO2H
f1 (ppm)

3an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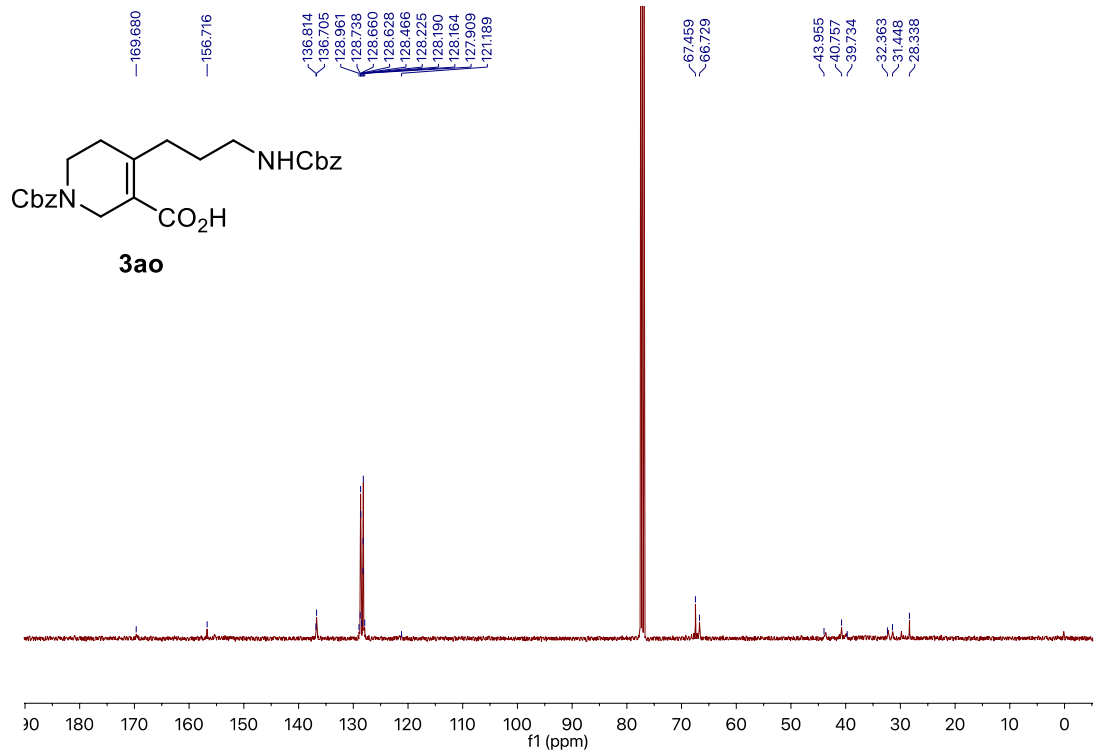
CbzN
CO2H
3ao
f1 (ppm)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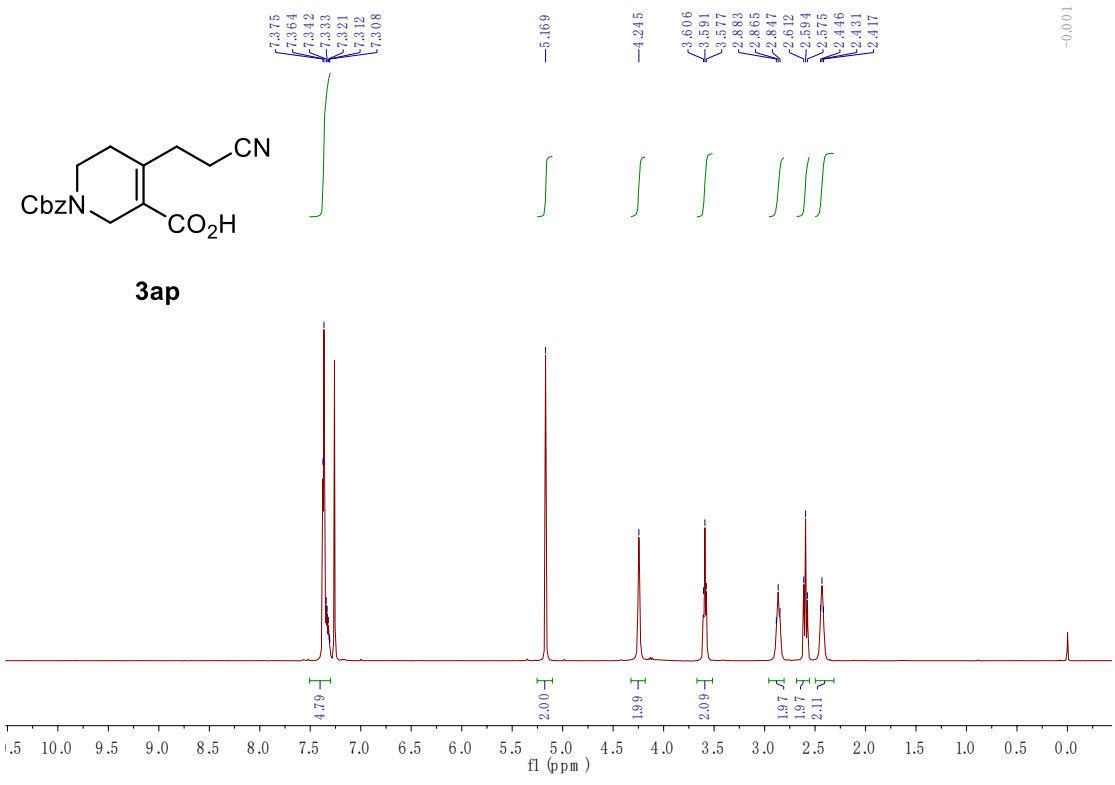
CbzN
CO2H
3ao
f1 (ppm)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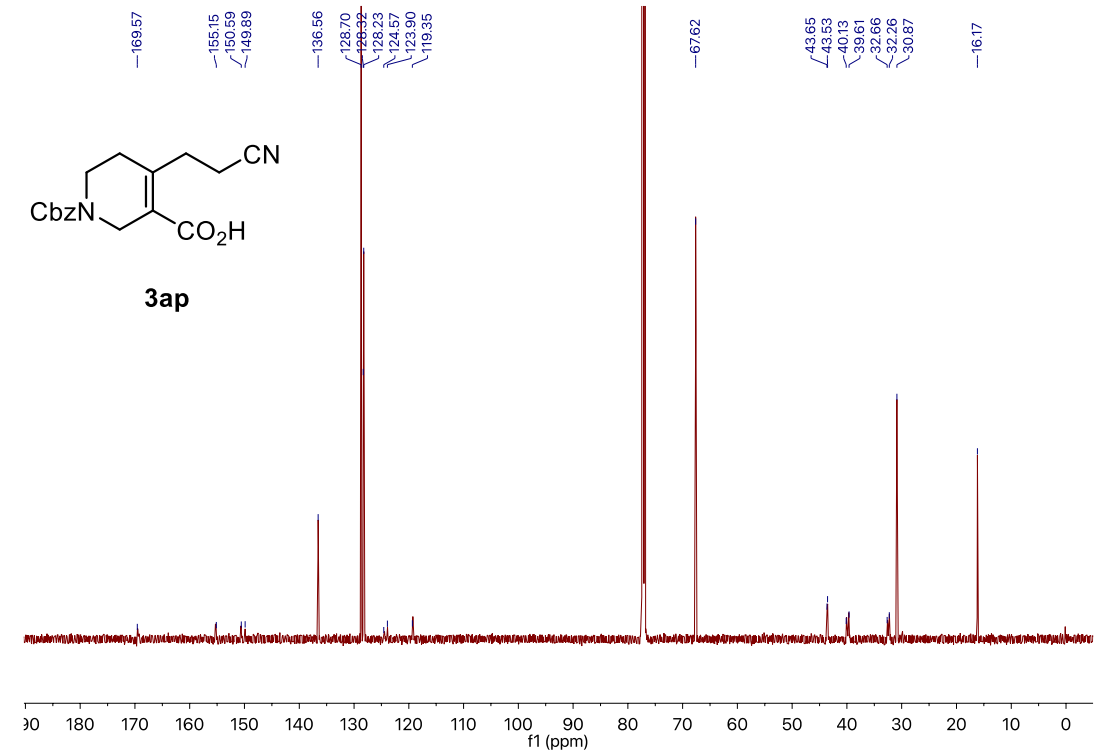
7.333
7.321
7.312
7.308
5.169
4.245
3.606
3.591
3.577
2.883
2.865
2.847
2.612
2.594
2.575
2.446
2.431
2.417
0.001
CN
CbzN
CO2H
4.79
2.00
1.99
2.09
1.97
1.97
2.11
f1 (ppm)

3ap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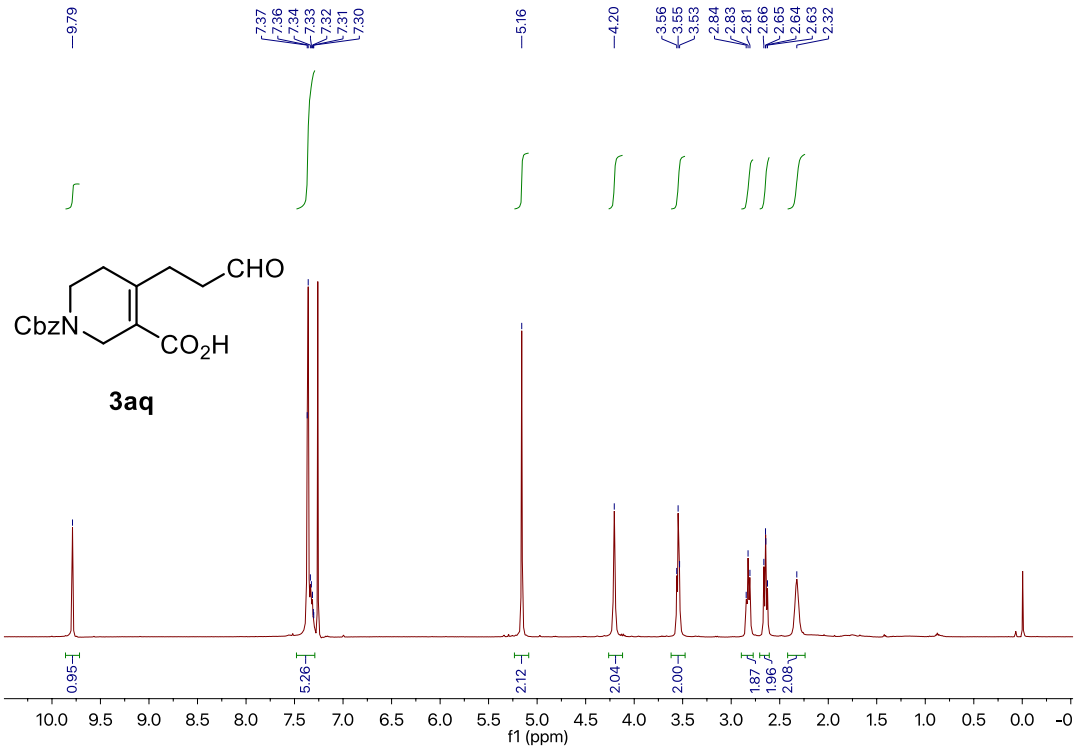
155.15
150.59
149.89
136.56
128.70
128.32
128.23
124.57
123.90
119.35
67.62
43.65
43.53
40.13
39.61
32.66
32.26
30.87
16.17
CN
CbzN
CO2H
f1 (ppm)

3ap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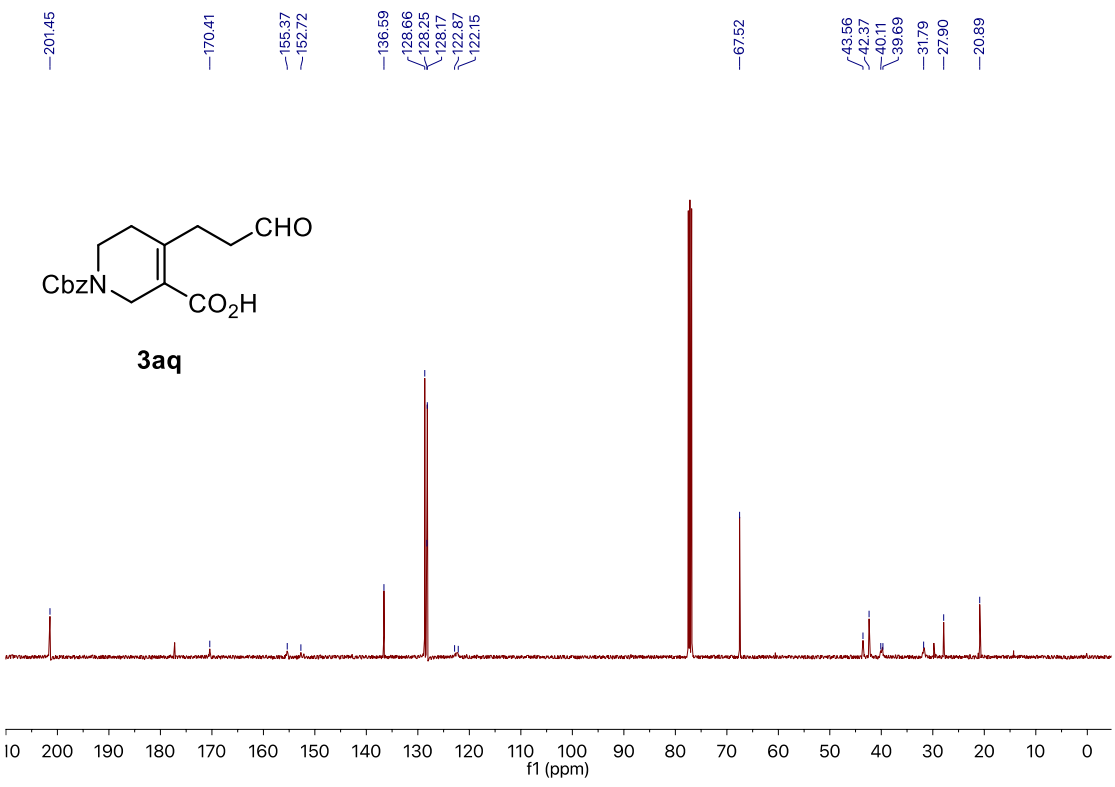
7.37
7.36
7.34
7.33
7.32
7.31
7.30
5.16
4.20
3.56
3.55
3.53
2.84
2.83
2.81
2.66
2.65
2.64
2.63
2.32
CHO
CbzN
CO2H
3aq
0.95
5.26
2.12
2.04
2.00
1.87
1.96
2.08
f1 (ppm)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170.41
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152.72
136.59
128.66
128.25
128.17
122.87
122.15
67.52
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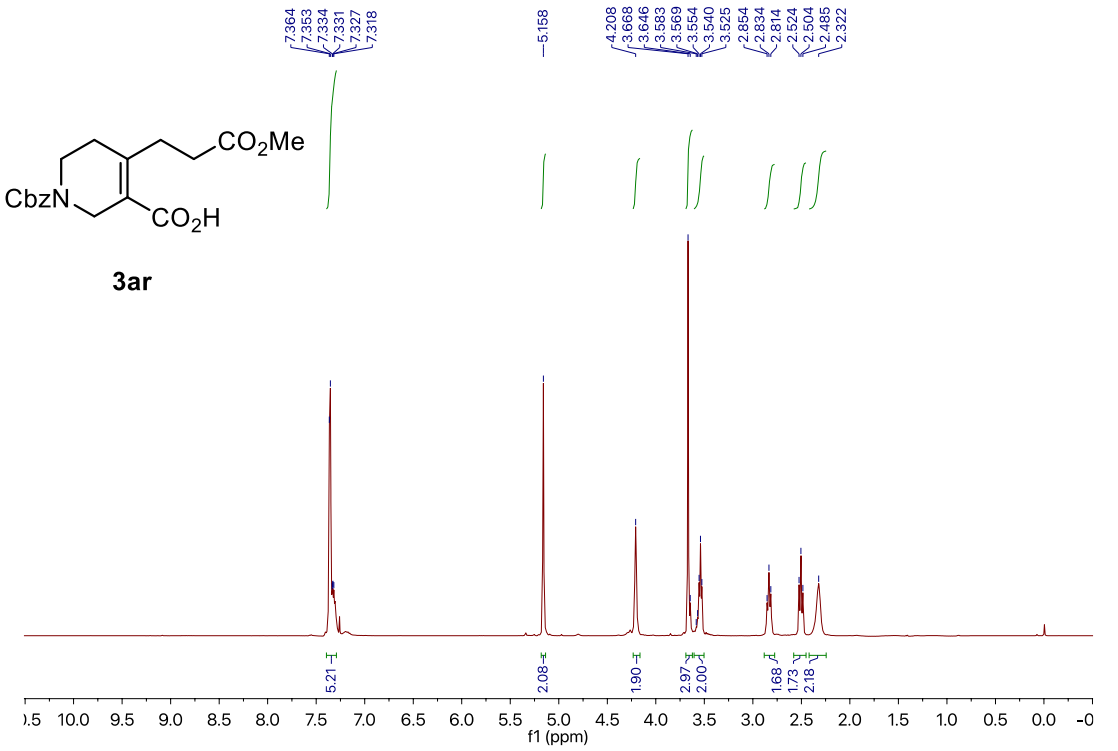
42.37
40.11
39.69
31.79
27.90
20.89
CHO
CbzN
CO2H
3aq
f1 (ppm)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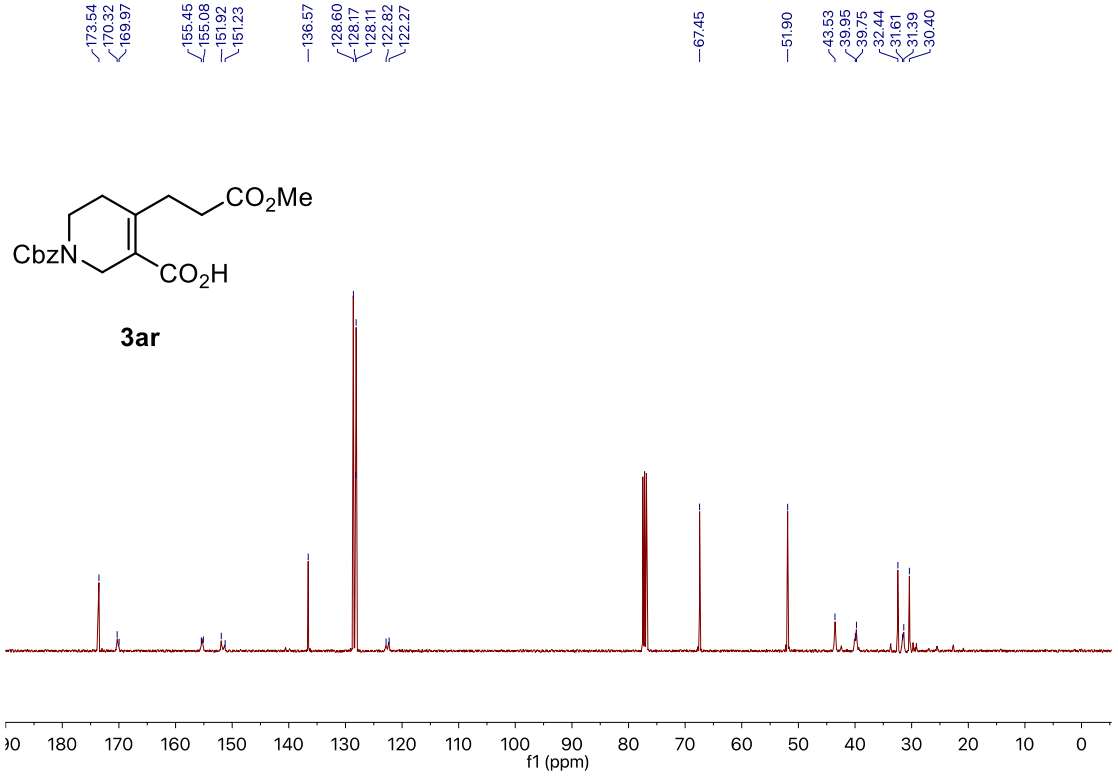
7.353
7.334
7.331
7.327
7.318
5.158
4.208
3.668
3.646
3.583
3.569
3.554
3.540
3.525
2.854
2.834
2.814
2.524
2.504
2.485
2.322
CO2Me
CbzN
CO2H
3ar
5.21
2.08
1.90
2.97
2.00
1.68
1.73
2.18
f1 (ppm)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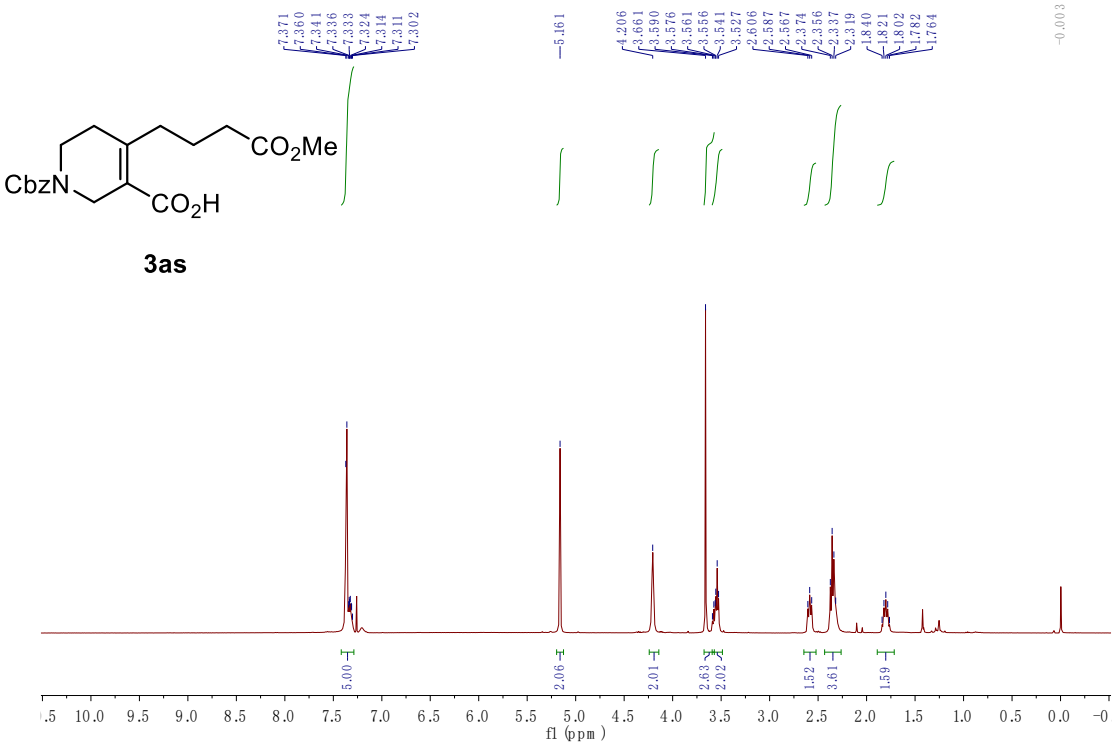
155.08
151.92
151.23
136.57
128.60
128.17
128.11
122.82
122.27
67.45
51.90
43.53
39.95
39.75
32.44
31.61
31.39
30.40
CO2Me
CbzN
CO2H
3ar
f1 (ppm)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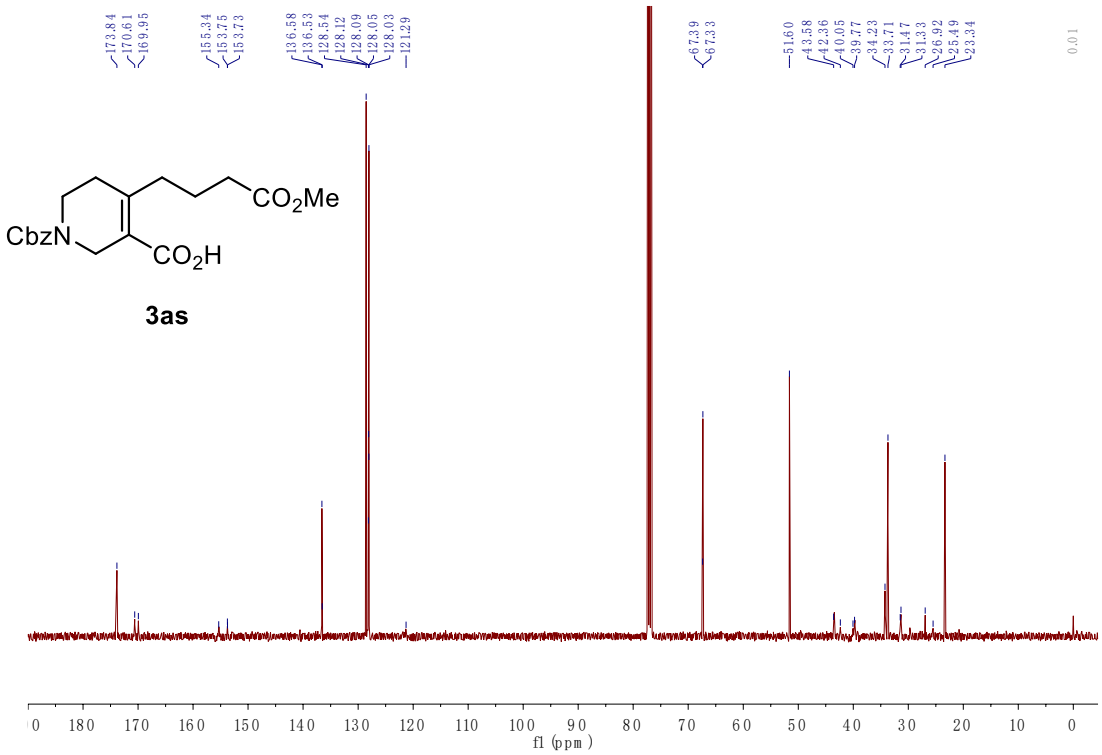
7.360
7.341
7.336
7.333
7.324
7.314
7.311
7.302
5.161
4.206
3.661
3.590
3.576
3.561
3.556
3.541
3.527
2.606
2.587
2.567
2.374
2.356
2.337
2.319
1.840
1.821
1.802
1.782
1.764
-0.003
CbzN
CO2Me
CO2H
5.00
2.06
2.01
2.63
2.02
1.52
3.61
1.59
f1 (ppm)

3as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155.34
153.75
153.73
136.58
136.53
128.54
128.12
128.09
128.05
128.03
121.29
67.39
67.33
51.60
43.58
42.36
40.05
39.77
34.23
33.71
31.47
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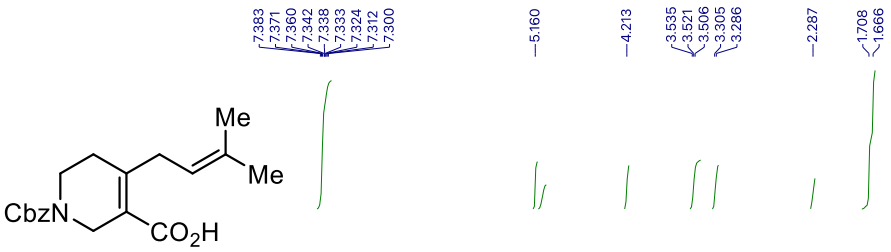
26.92
25.49
23.34
0.01
CbzN
CO2Me
CO2H
f1 (ppm)

3as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7.333
7.324
7.312
7.300
5.160
4.213
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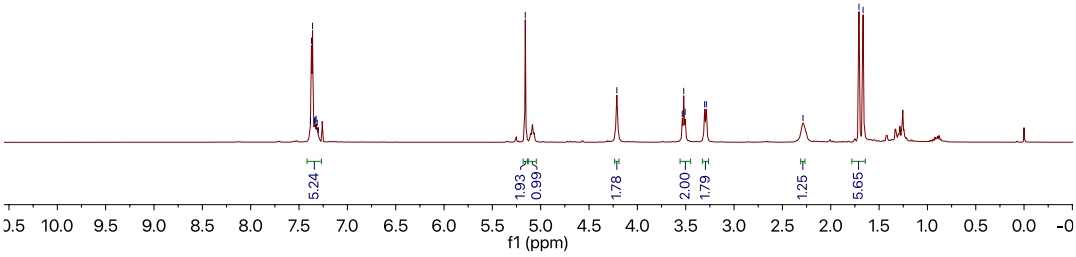
3.521
3.506
3.305
3.286
2.287
1.708
1.666
Me
Me
CbzN
CO2H

3at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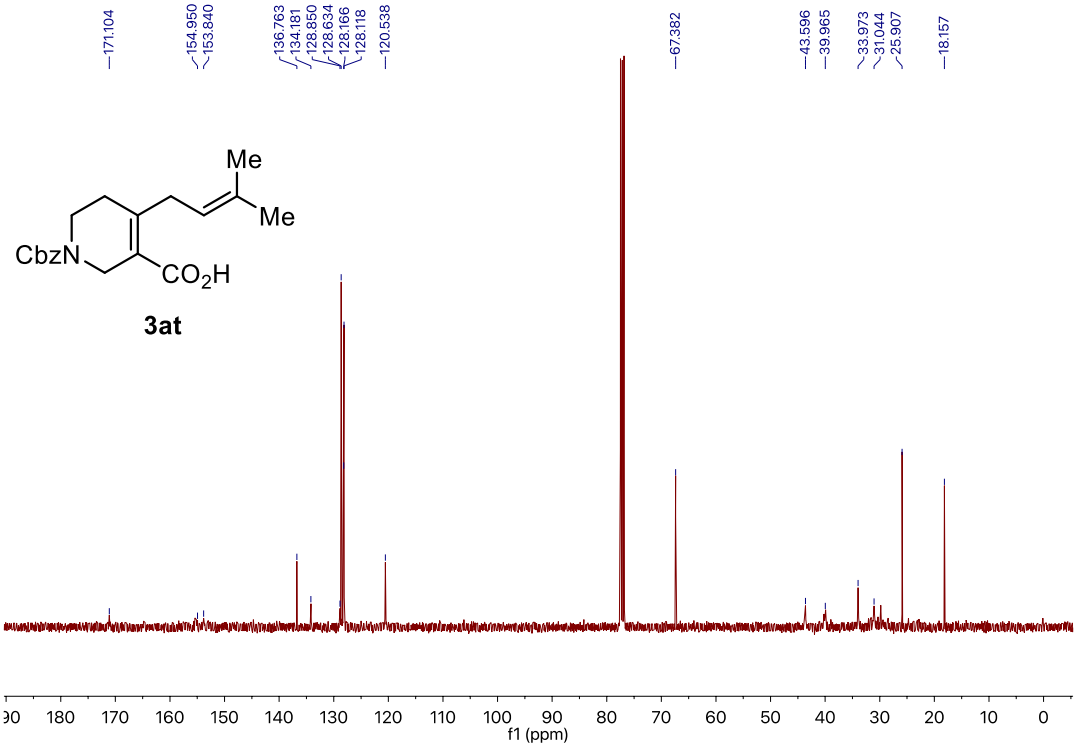
1.93
0.99
1.78
2.00
1.79
1.25
5.65
f1 (ppm)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134.181
128.850
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128.166
128.118
120.538
67.382
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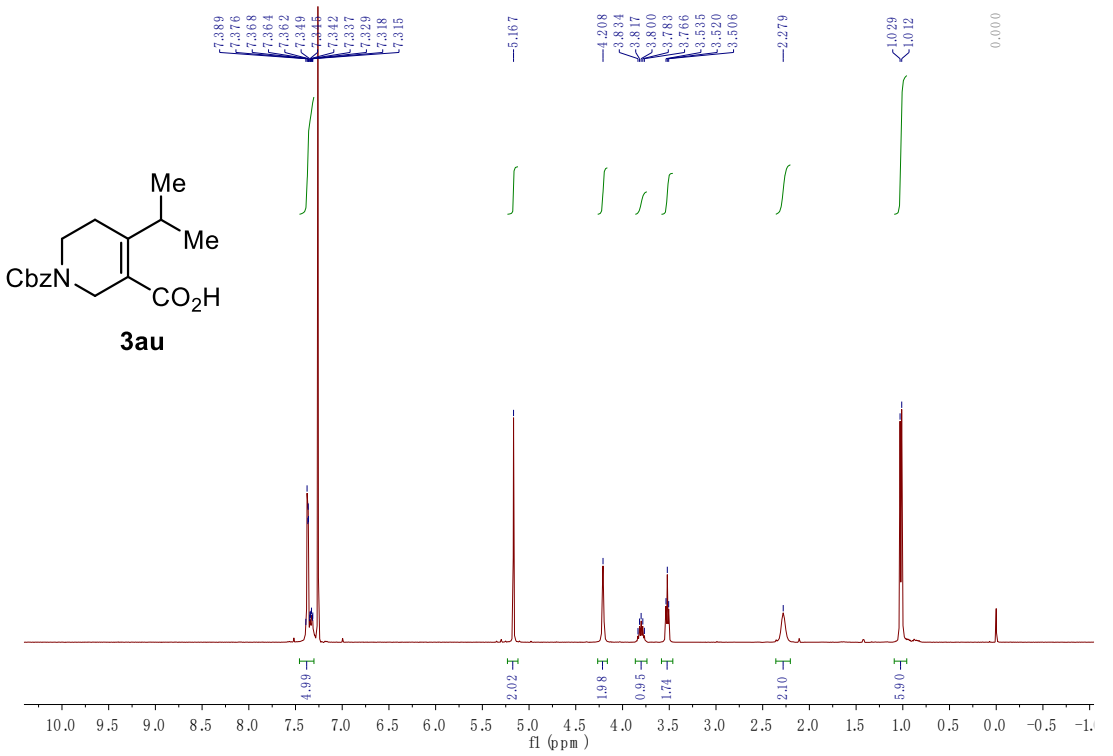
33.973
31.044
25.907
18.157
Me
Me
CbzN
CO2H
f1 (ppm)

3at


Me
Me
CbzN
CO2H
3au
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7.368
7.364
7.362
7.349
7.345
7.342
7.337
7.329
7.318
7.315
5.167
4.208
3.834
3.817
3.800
3.783
3.766
3.535
3.520
3.506
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0.000
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2.02
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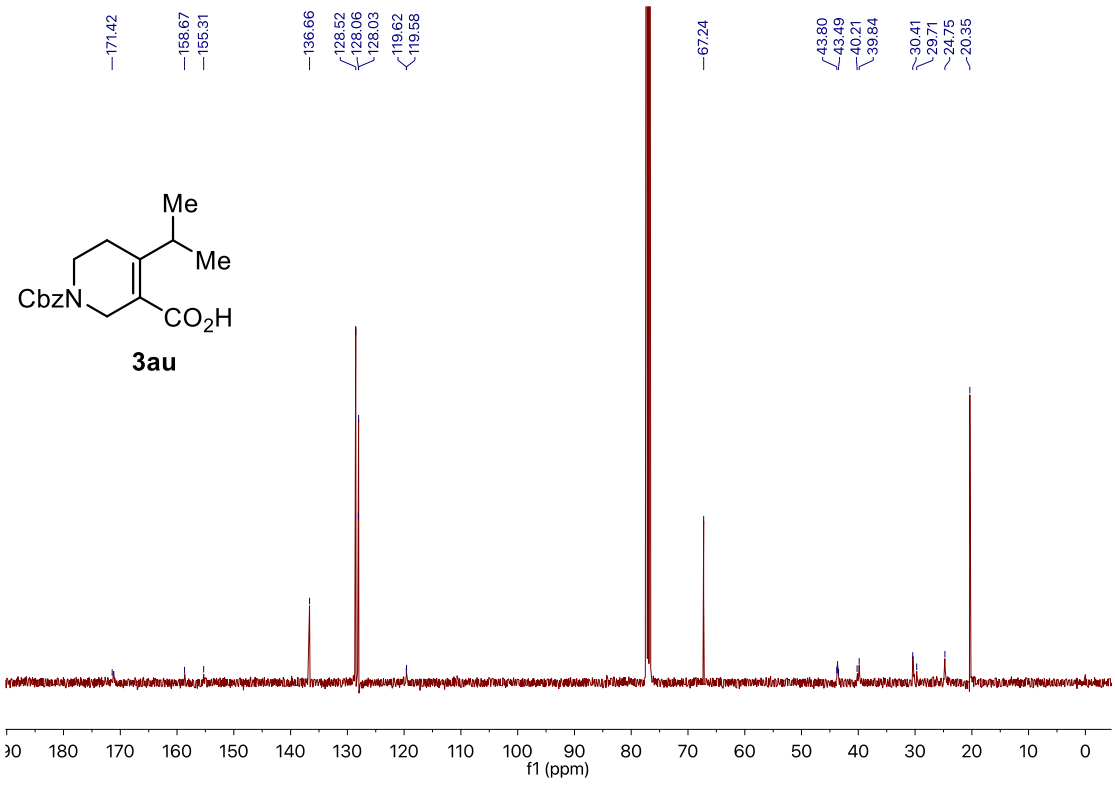
0.95
1.74
2.10
5.90
f1 (ppm)


Me
Me
CbzN
CO2H
3au
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155.31
136.66
128.52
128.06
128.03
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119.58
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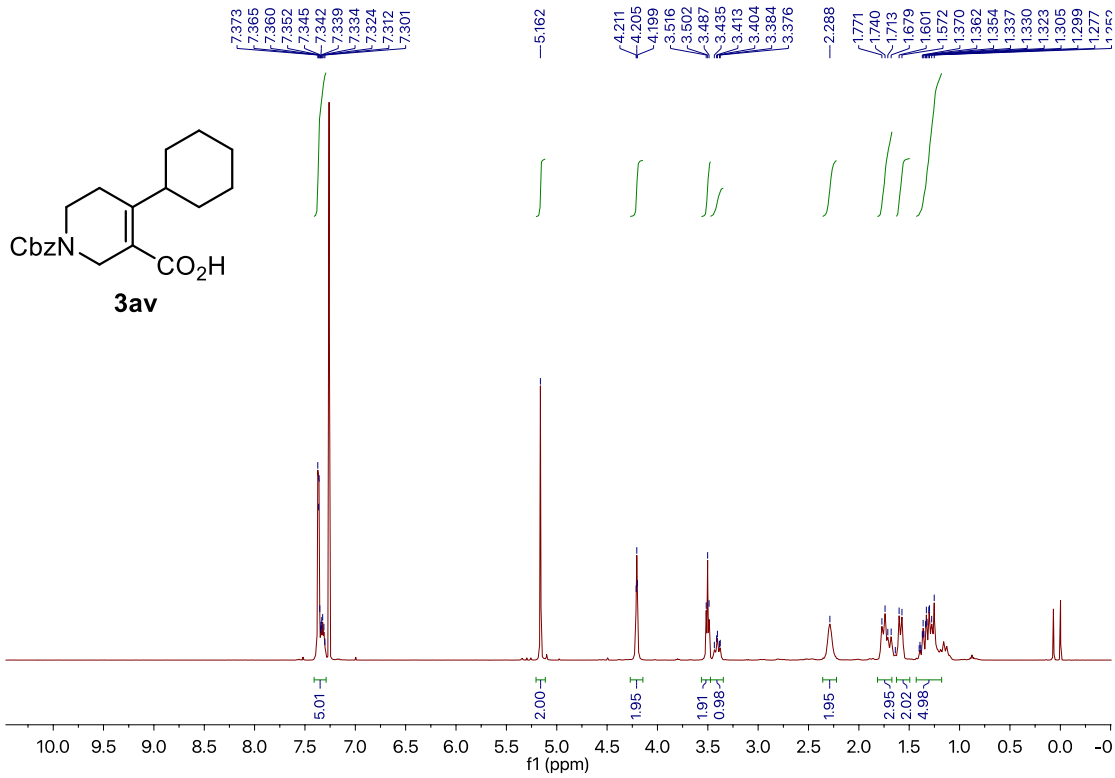
30.41
29.71
24.75
20.35
f1 (ppm)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7.334
7.324
7.312
7.301
5.162
4.211
4.205
4.199
3.516
3.502
3.487
3.435
3.413
3.404
3.384
3.376
2.288
1.771
1.740
1.713
1.679
1.601
1.572
1.370
1.362
1.354
1.337
1.330
1.323
1.305
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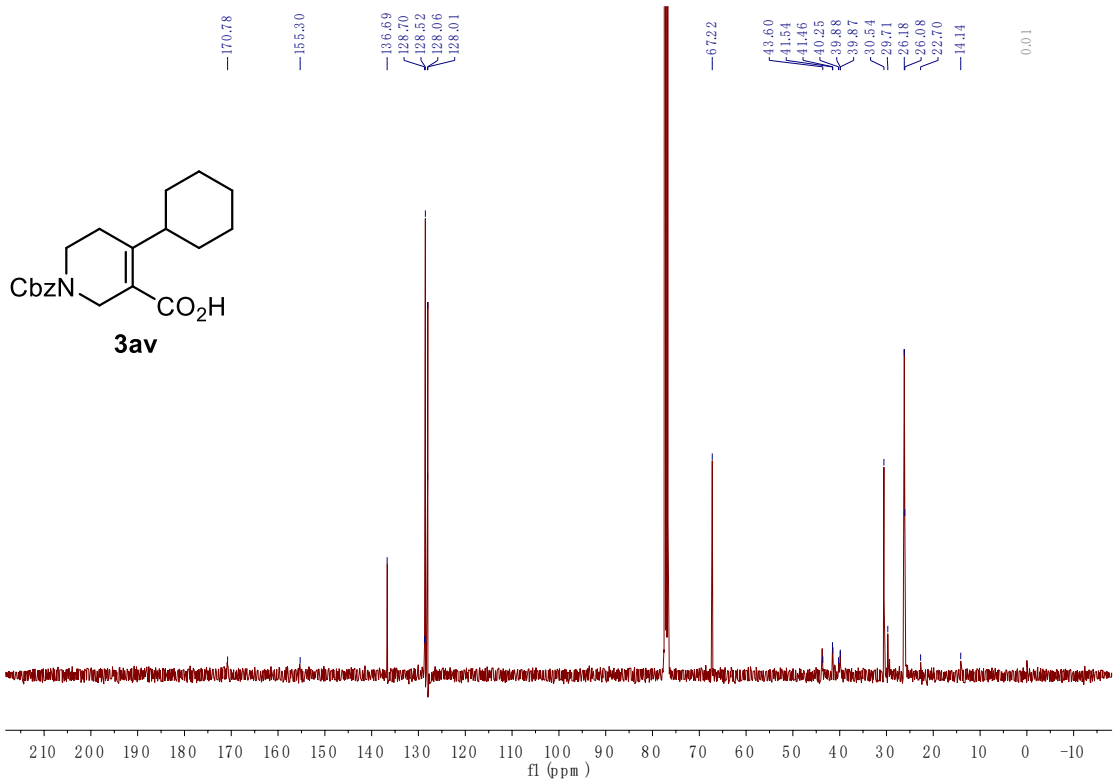
2.00
1.95
1.91
0.98
1.95
2.95
2.02
4.98
f1 (ppm)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128.52
128.06
128.01
67.22
43.60
41.54
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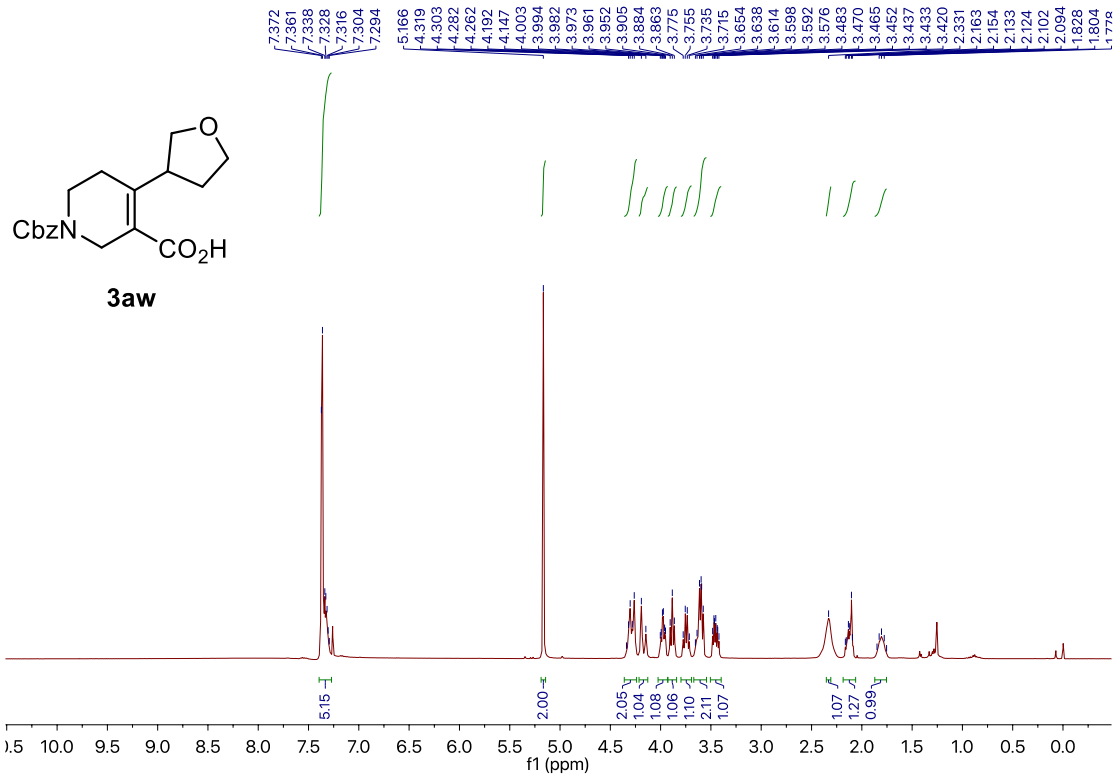
39.87
30.54
29.71
26.18
26.08
22.70
14.14
0.01
f1 (ppm)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4.147
4.003
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3.982
3.973
3.961
3.952
3.905
3.884
3.863
3.775
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3.735
3.715
3.654
3.638
3.614
3.598
3.592
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3.483
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3.452
3.437
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3.420
2.331
2.163
2.154
2.133
2.124
2.102
2.094
1.828
1.804
CbzN
CO2H
3aw
5.15
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2.05
1.04
1.08
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1.10
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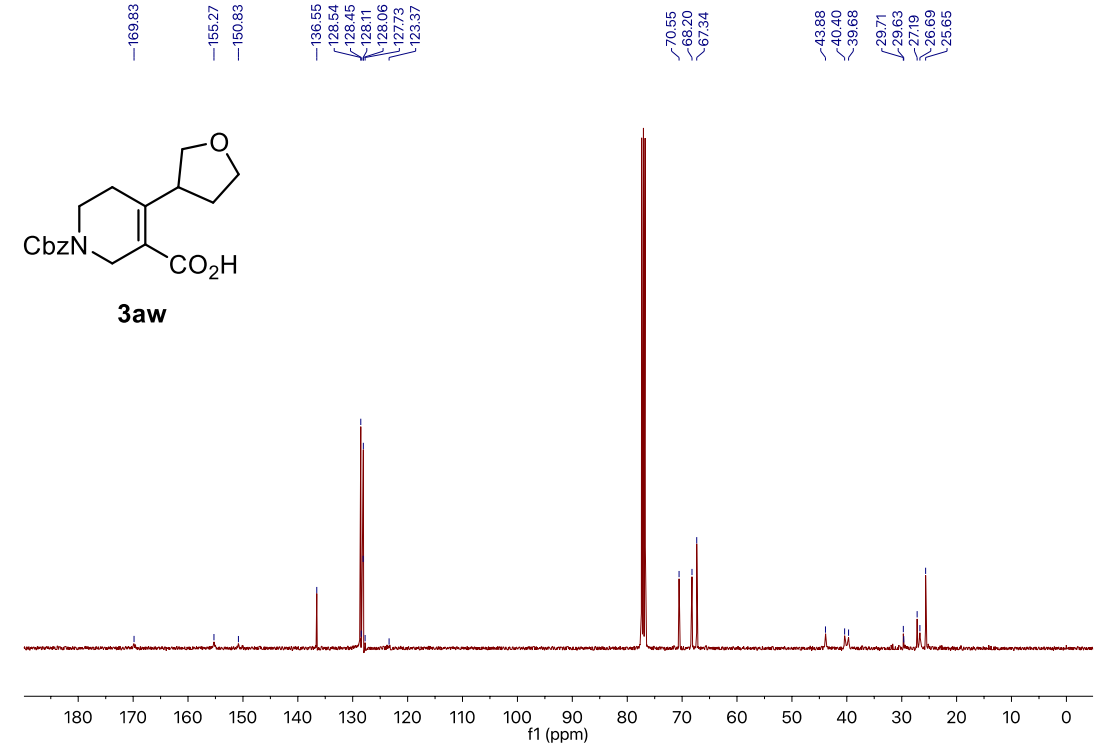
1.07
1.27
0.99
f1 (ppm)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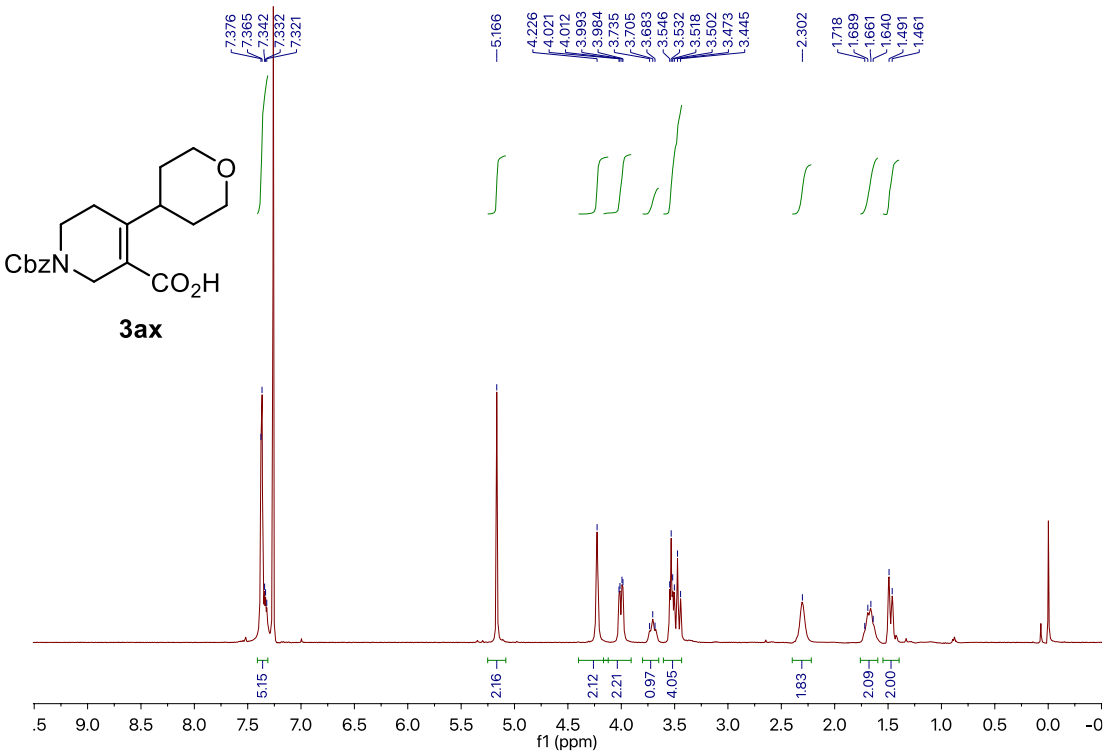
128.45
128.11
128.06
127.73
123.37
70.55
68.20
67.34
43.88
40.40
39.68
29.71
29.63
27.19
26.69
25.65
CbzN
CO2H
3aw
f1 (ppm)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7.321
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4.021
4.012
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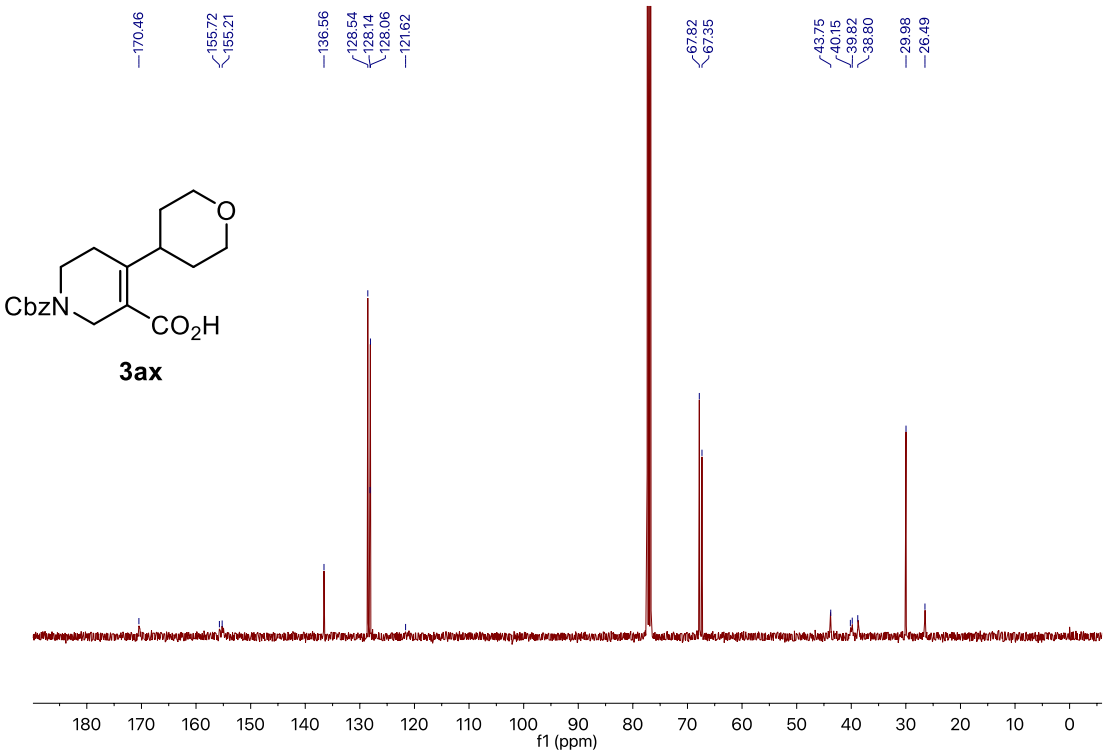
3.705
3.683
3.546
3.532
3.518
3.502
3.473
3.445
2.302
1.718
1.689
1.661
1.640
1.491
1.461
CbzN
O
CO2H
3ax
5.15
2.16
2.12
2.21
0.97
4.05
1.83
2.09
2.00
f1 (ppm)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128.06
121.62
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39.82
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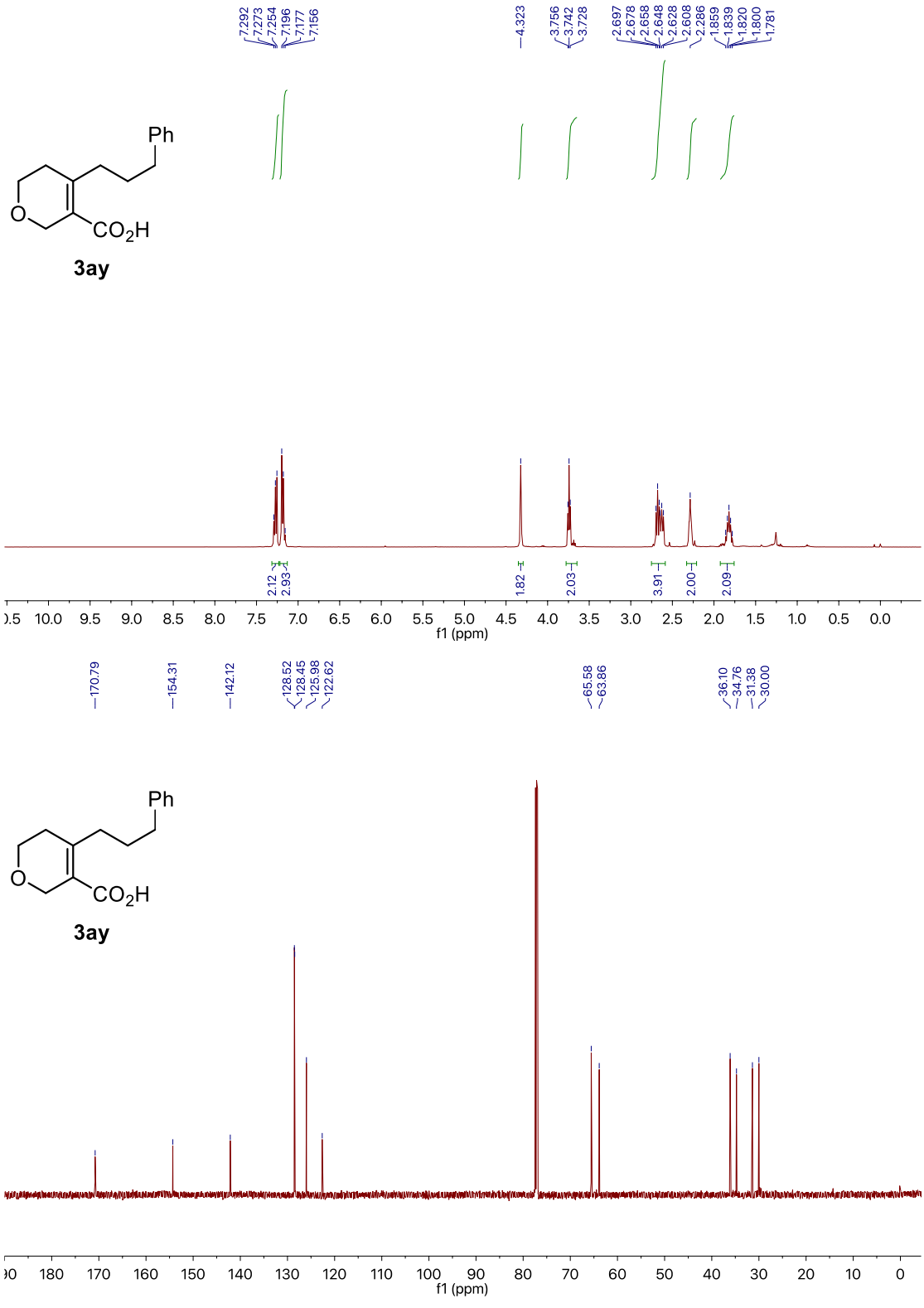
26.49
CbzN
O
CO2H
3ax
f1 (ppm)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2.658
2.648
2.628
2.608
2.286
1.859
1.839
1.820
1.800
1.781
Ph
O
CO2H
3ay
2.12
2.93
1.82
2.03
3.91
2.00
2.09
f1 (ppm)
170.79
154.31
142.12
128.52
128.45
125.98
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63.86
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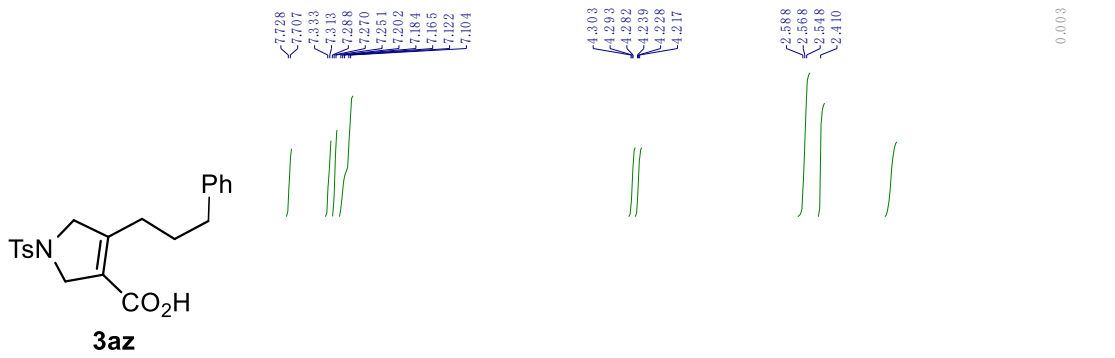
Ph
O
CO2H
3ay
f1 (ppm)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7.104
4.303
4.293
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4.217
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2.568
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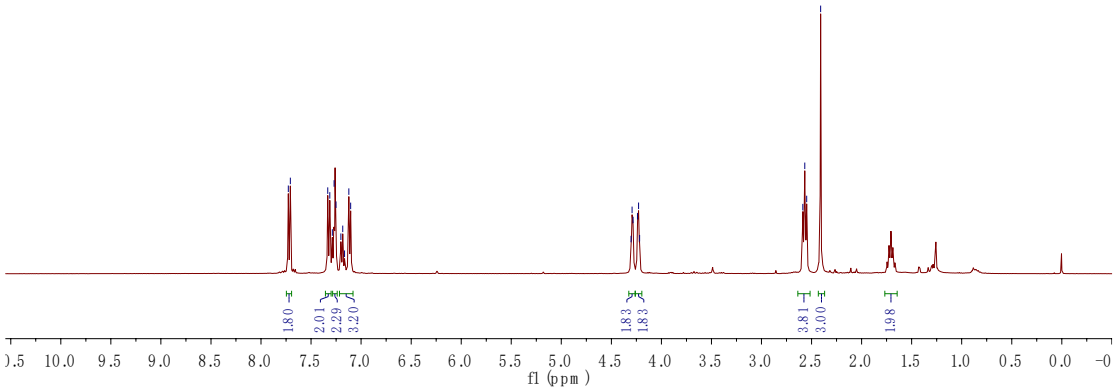
0.003
Ph
TsN
CO2H

3az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1.83
3.81
3.00
1.98
f1 (ppm)


167.91
155.73
143.96
141.22
133.54
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128.31
127.52
126.11
122.40
58.63
54.96
35.65
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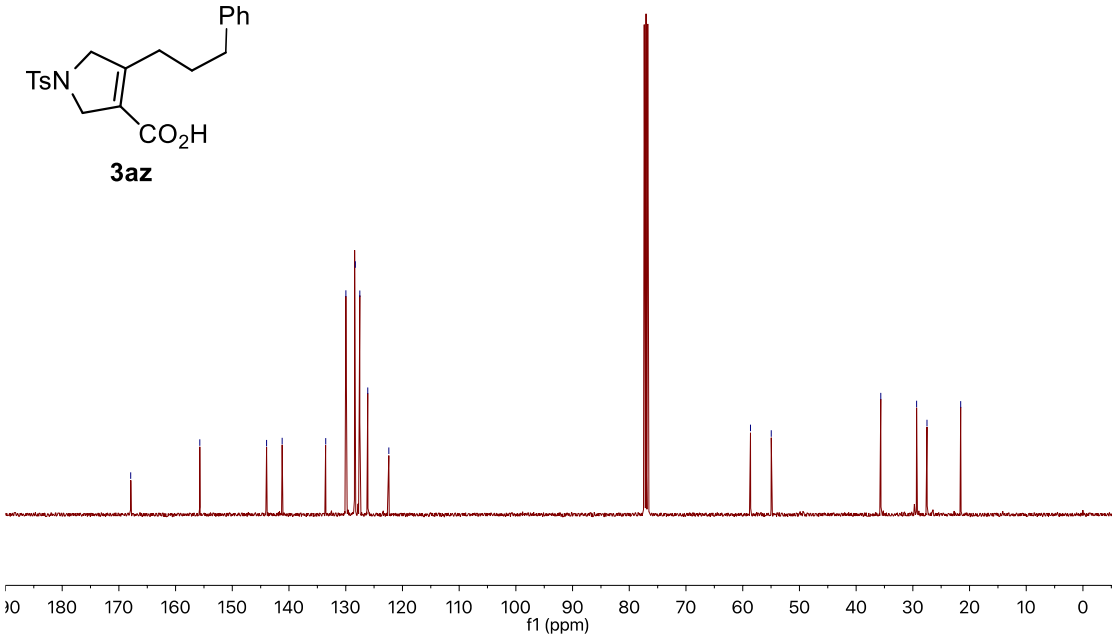
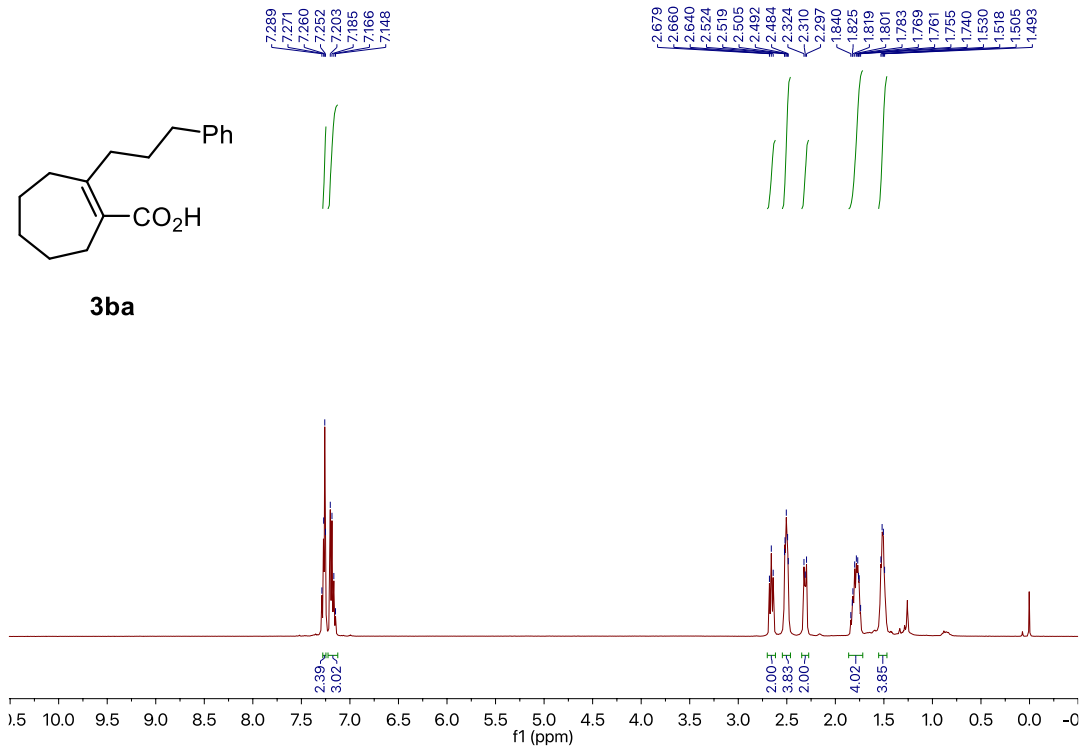
27.51
21.55


Ph
TsN
CO2H
f1 (ppm)

3az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7.271
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2.505
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2.310
2.297
1.840
1.825
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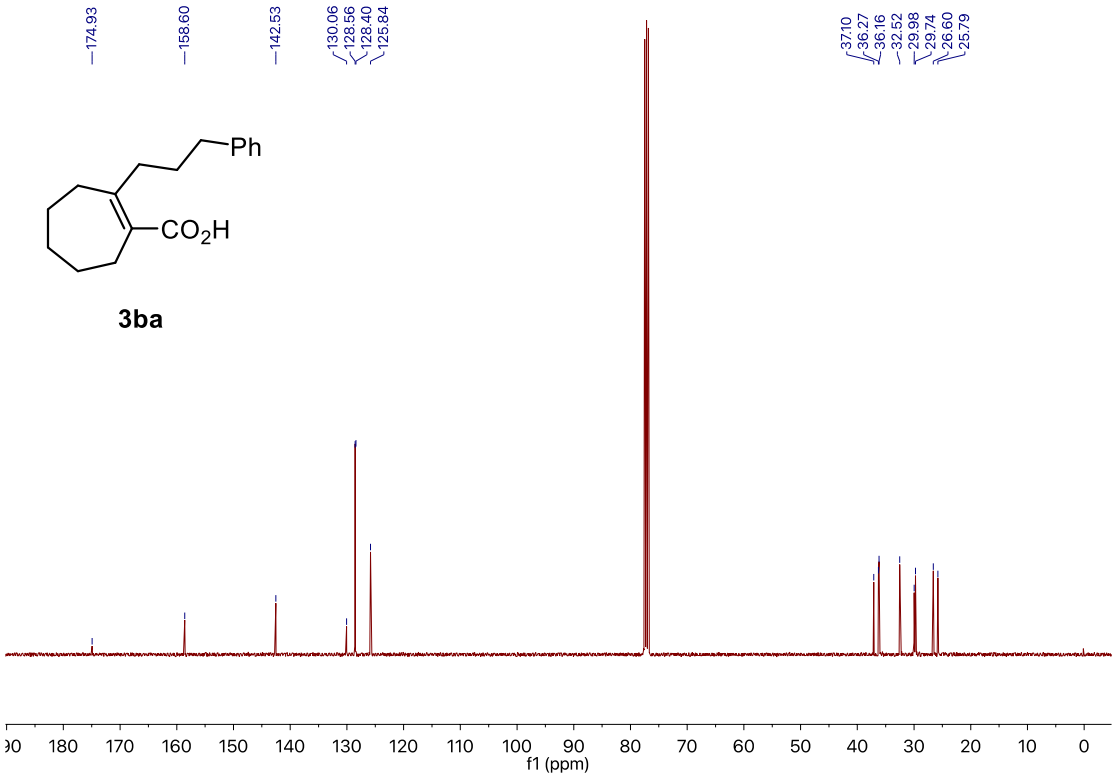
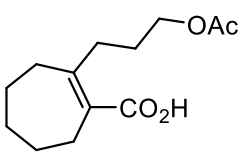
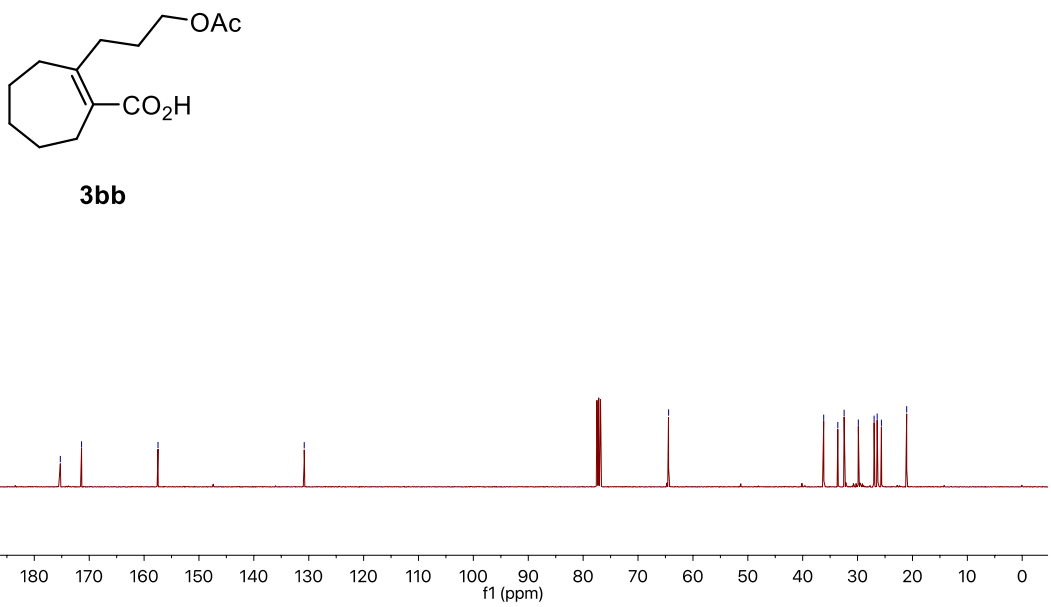
1.801
1.783
1.769
1.761
1.755
1.740
1.530
1.518
1.505
1.493
Ph
CO2H
3ba
2.39
3.02
2.00
3.83
2.00
4.02
3.85
f1 (ppm)


174.93
158.60
142.53
130.06
128.56
128.40
125.84
37.10
36.27
36.16
32.52
29.98
29.74
26.60
25.79
Ph
CO2H
3ba
f1 (ppm)


OAc
CO2H

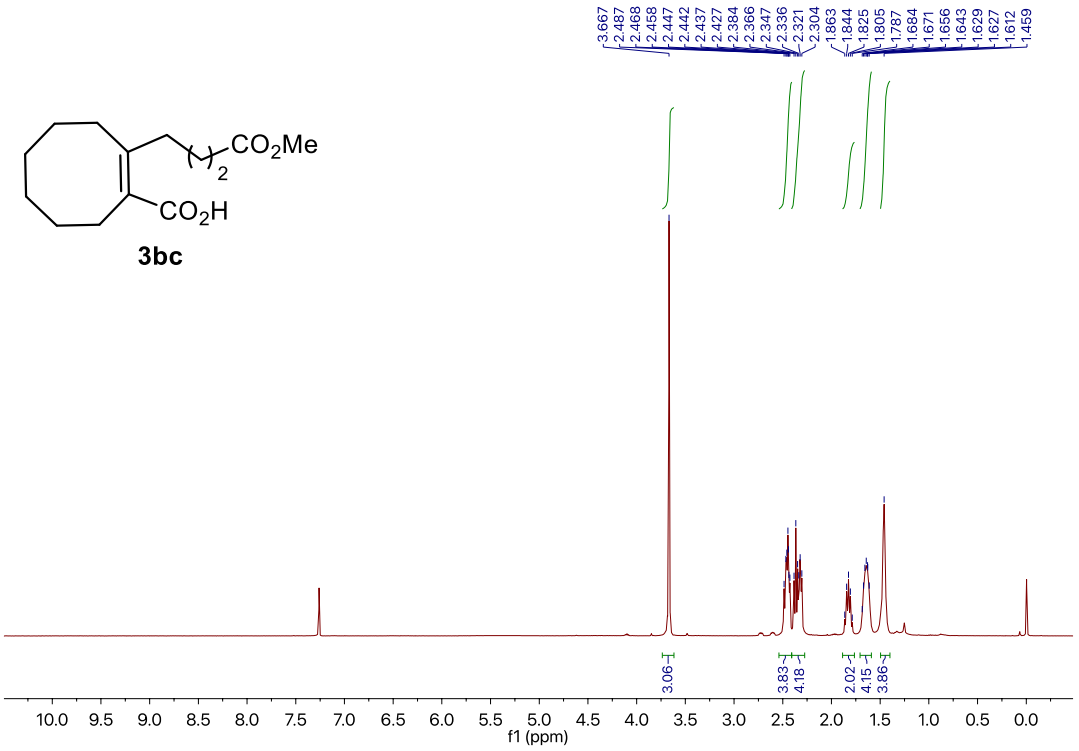
3bb

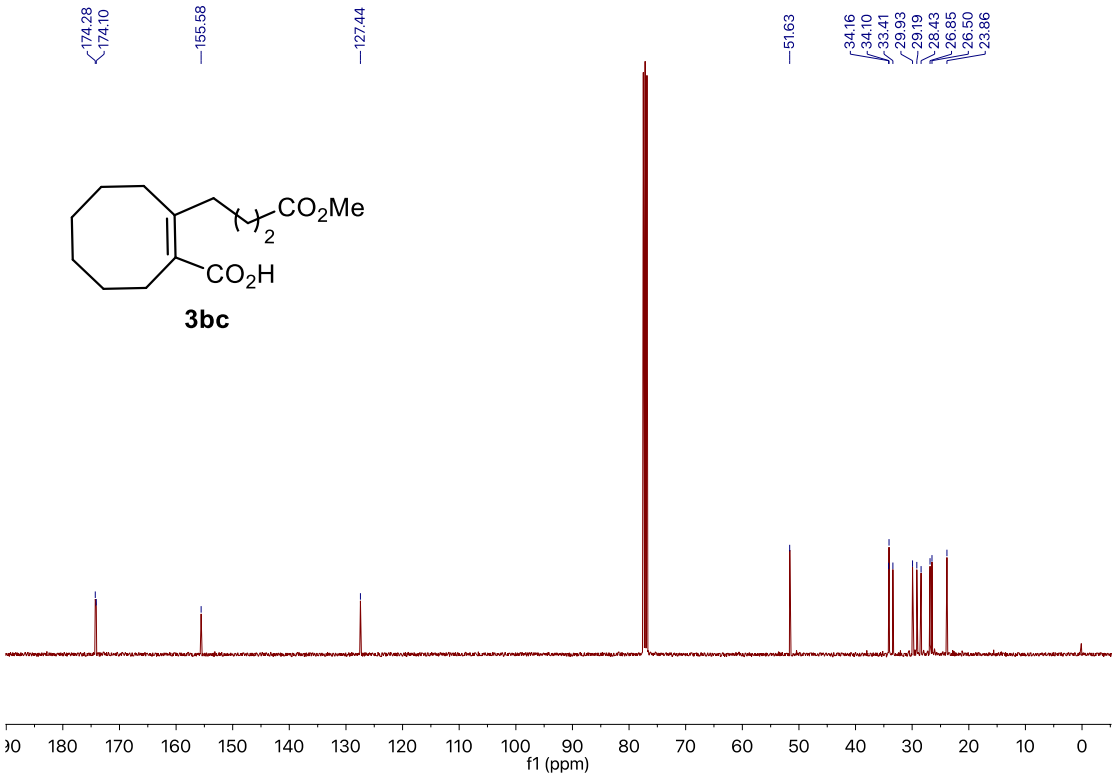
4.089
4.073
4.056
2.514
2.495
2.476
2.467
2.327
2.314
2.300
2.037
1.828
1.811
1.793
1.774
1.763
1.756
1.747
1.733
1.514
1.503
1.493
2.00
3.99
2.05
3.15
4.19
4.09
f1 (ppm)


175.25
171.40
157.46
130.82
64.44
36.17
33.59
32.44
29.87
26.98
26.43
25.66
21.06
f1 (ppm)

OAc
CO2H

3bb


3.667
2.487
2.468
2.458
2.447
2.442
2.437
2.427
2.384
2.366
2.347
2.336
2.321
2.304
1.863
1.844
1.825
1.805
1.787
1.684
1.671
1.656
1.643
1.629
1.627
1.612
1.459
CO2Me
2
CO2H
3bc
3.06
3.83
4.18
2.02
4.15
3.86
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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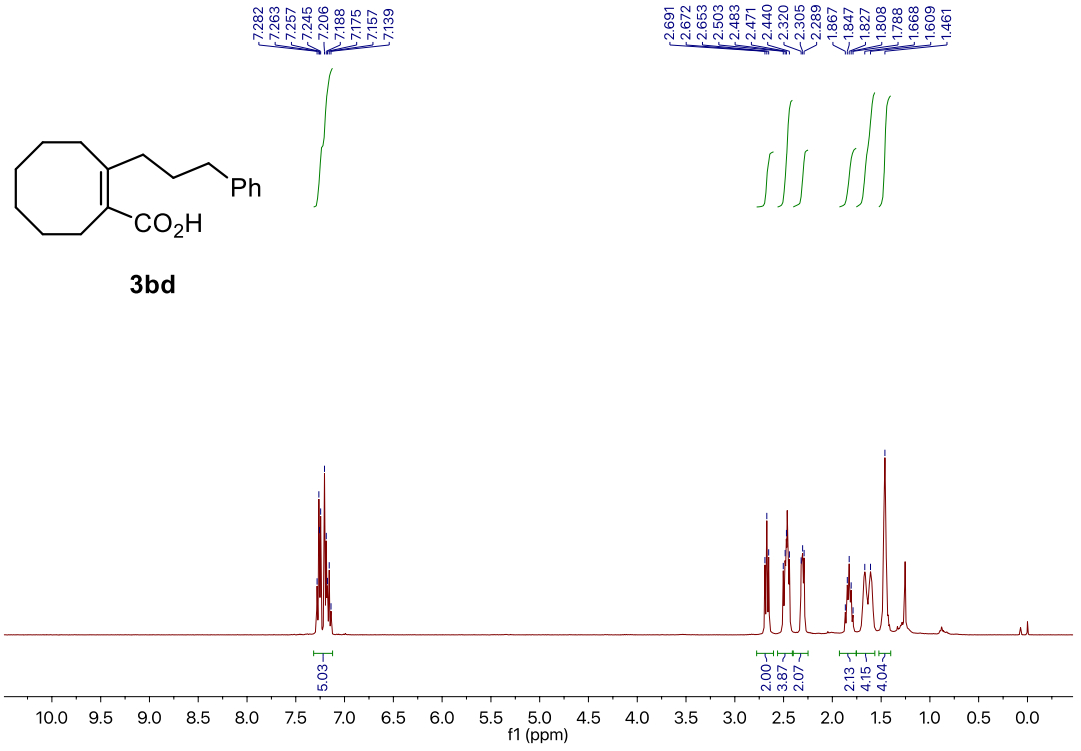
174.10
155.58
127.44
51.63
34.16
34.10
33.41
29.93
29.19
28.43
26.85
26.50
23.86
CO2Me
2
CO2H
3bc
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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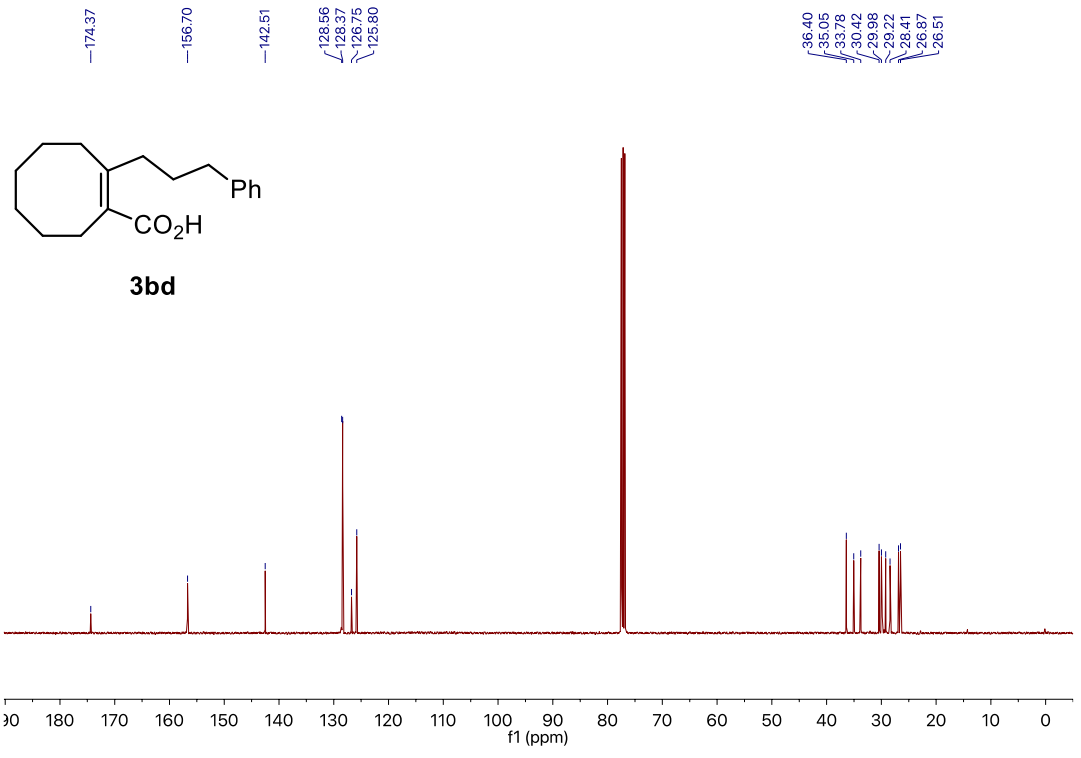
7.263
7.257
7.245
7.206
7.188
7.175
7.157
7.139
2.691
2.672
2.653
2.503
2.483
2.471
2.440
2.320
2.305
2.289
1.867
1.847
1.827
1.808
1.788
1.668
1.609
1.461
Ph
CO2H
3bd
5.03
2.00
3.87
2.07
2.13
4.15
4.04
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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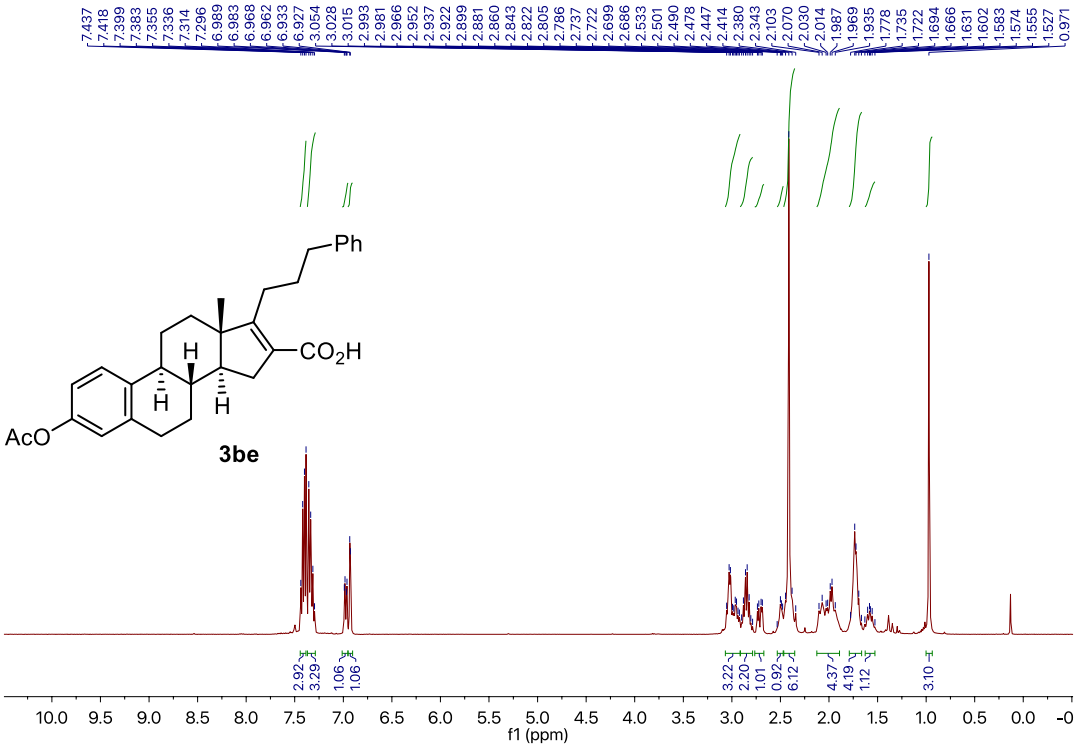
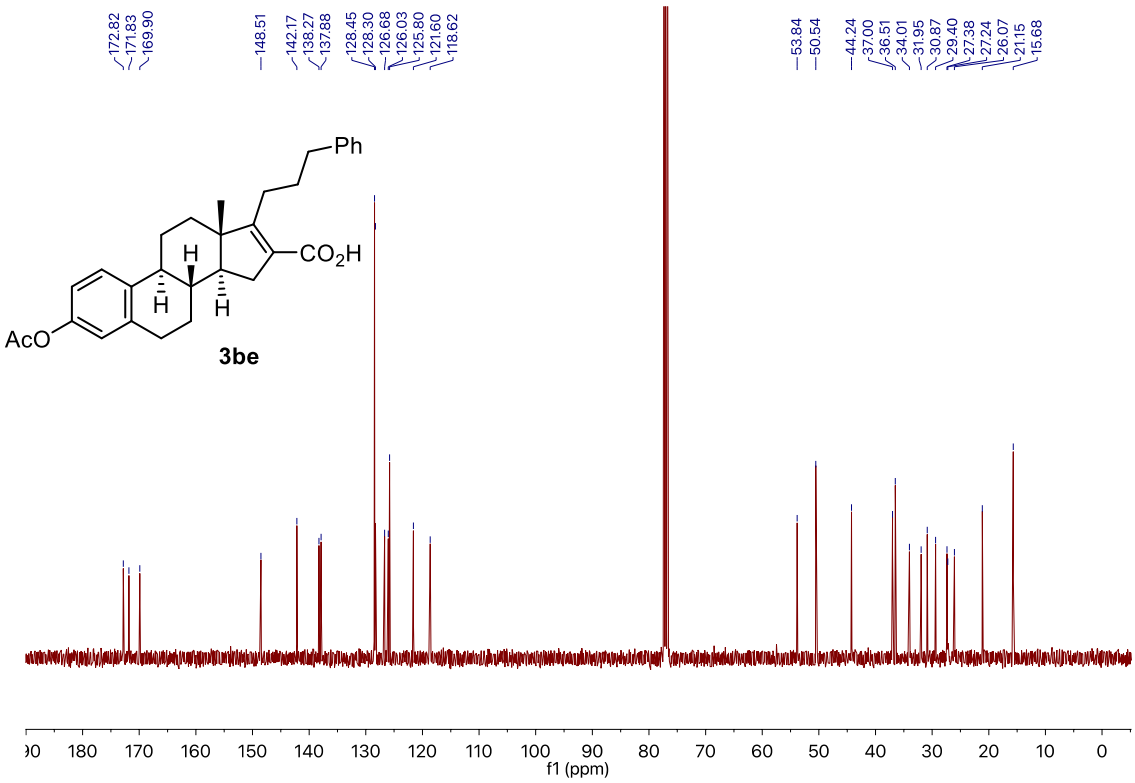
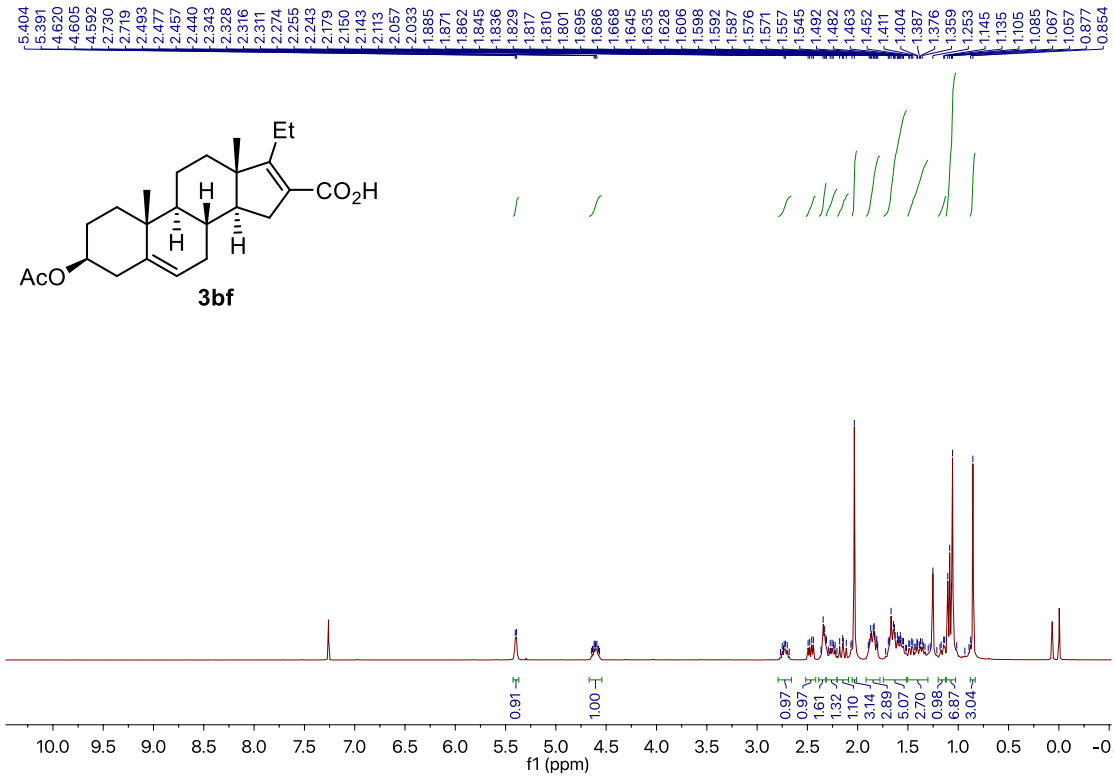
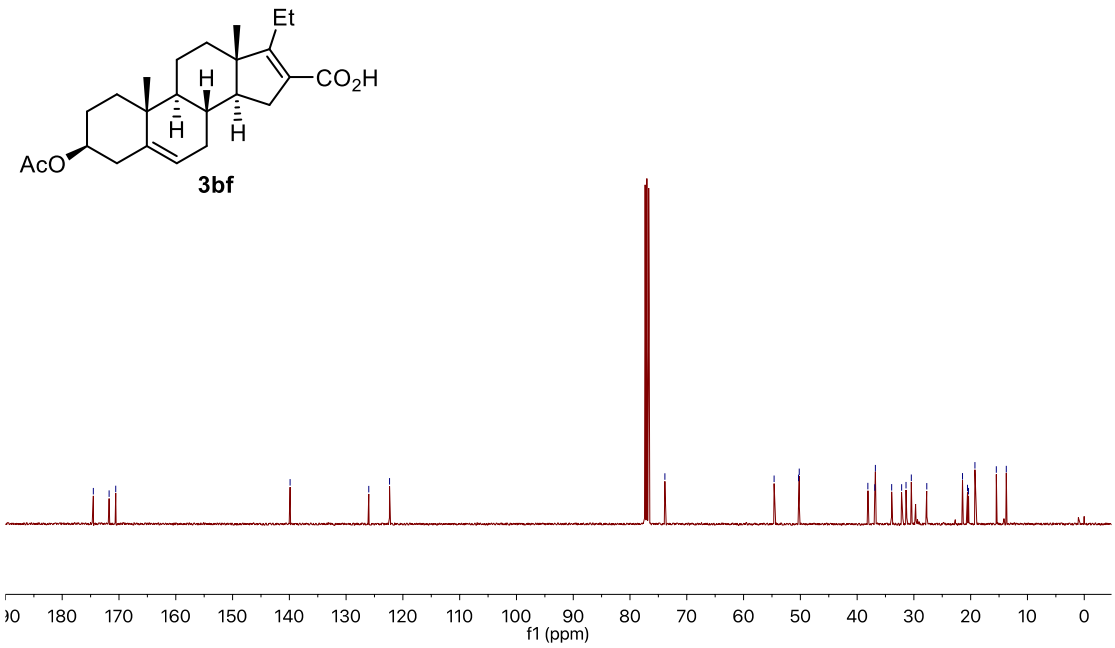
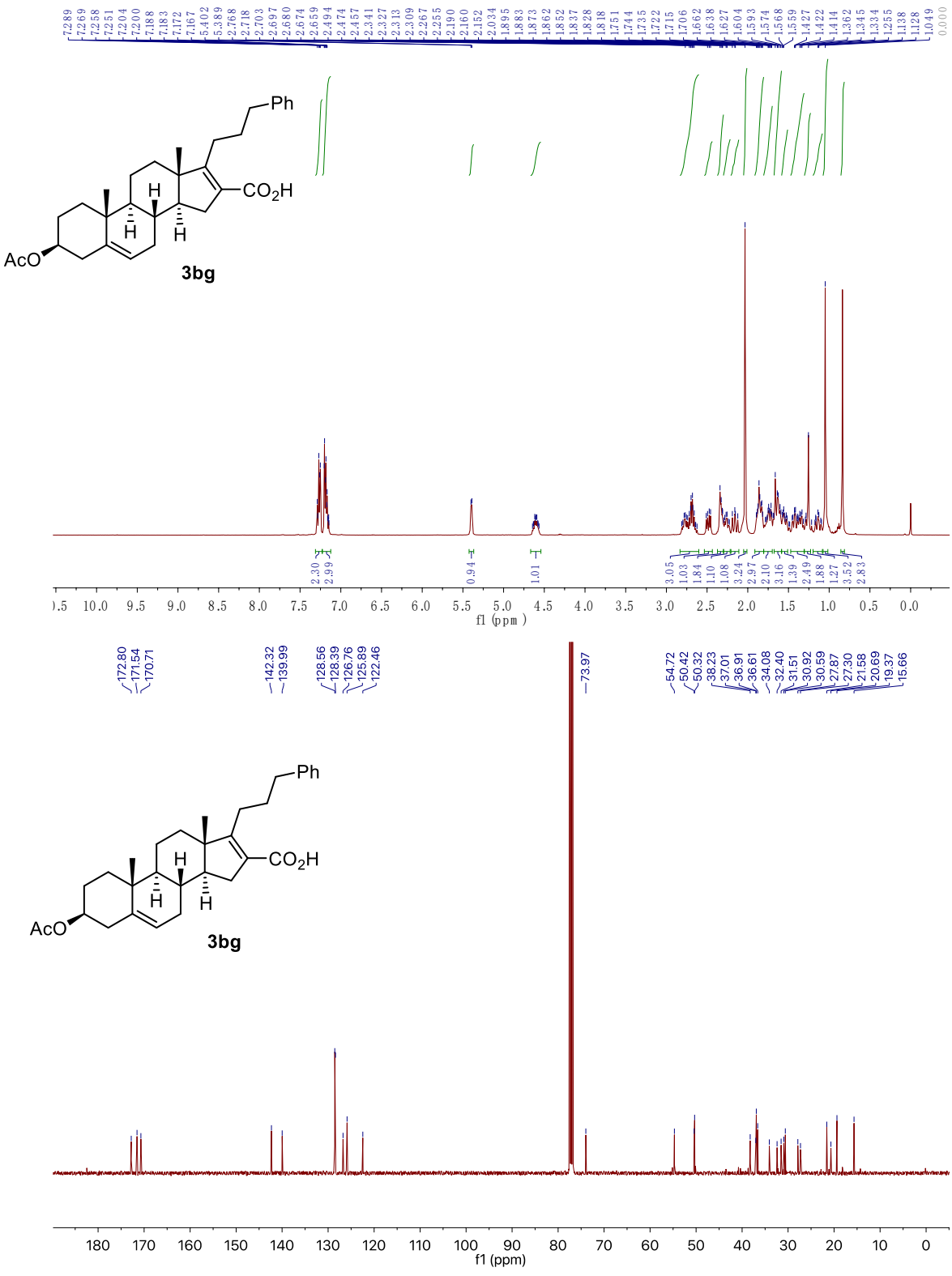
156.70
142.51
128.56
128.37
126.75
125.80
36.40
35.05
33.78
30.42
29.98
29.22
28.41
26.87
26.51
Ph
CO2H
3bd
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

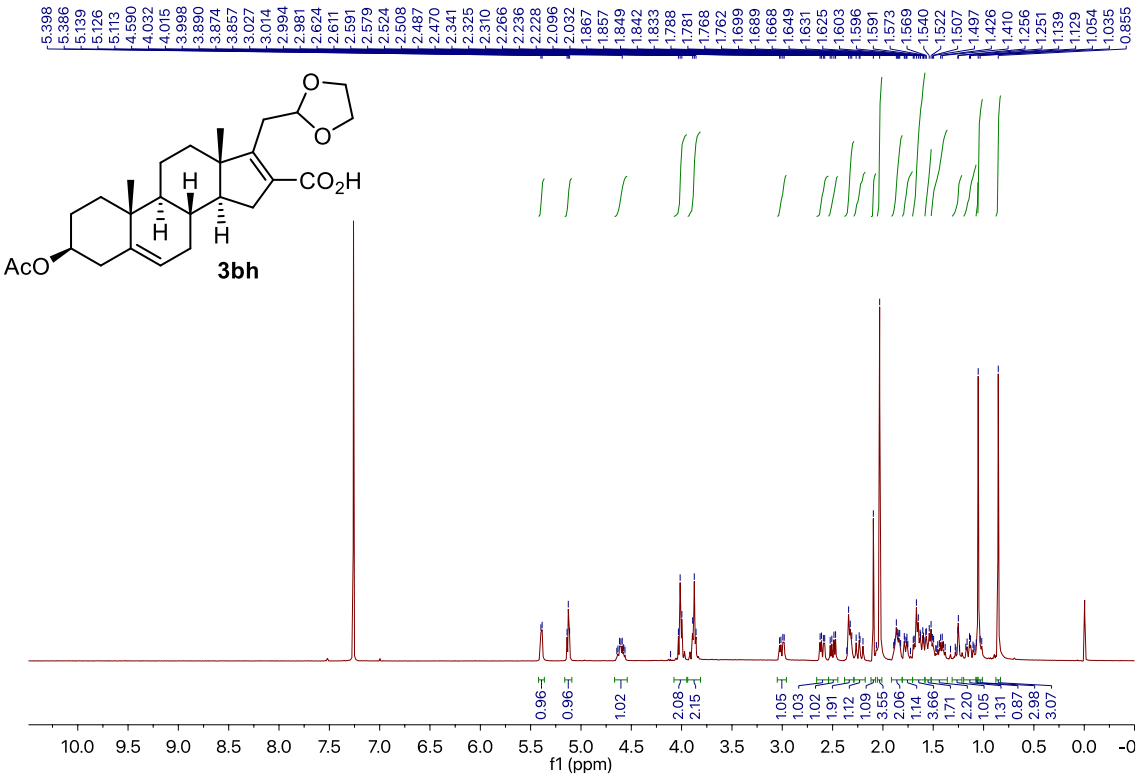

Ph
CO2H
H
H
H
AcO
3be
f1 (ppm)


Ph
CO2H
H
H
H
AcO
3be
f1 (ppm)


Et
CO2H
H
H
H
AcO
3bf
f1 (ppm)


Et
CO2H
H
H
H
AcO
3bf
f1 (ppm)


Ph
CO2H
H
H
H
AcO
3bg
fl (ppm)
f1 (ppm)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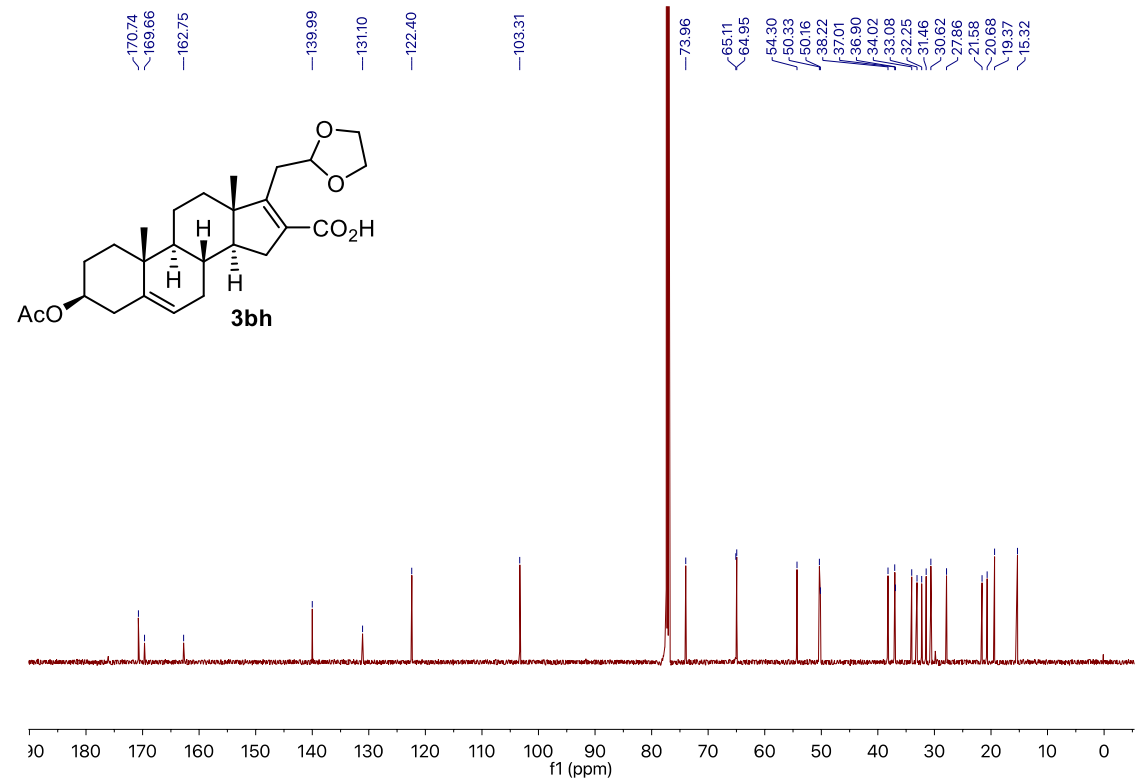
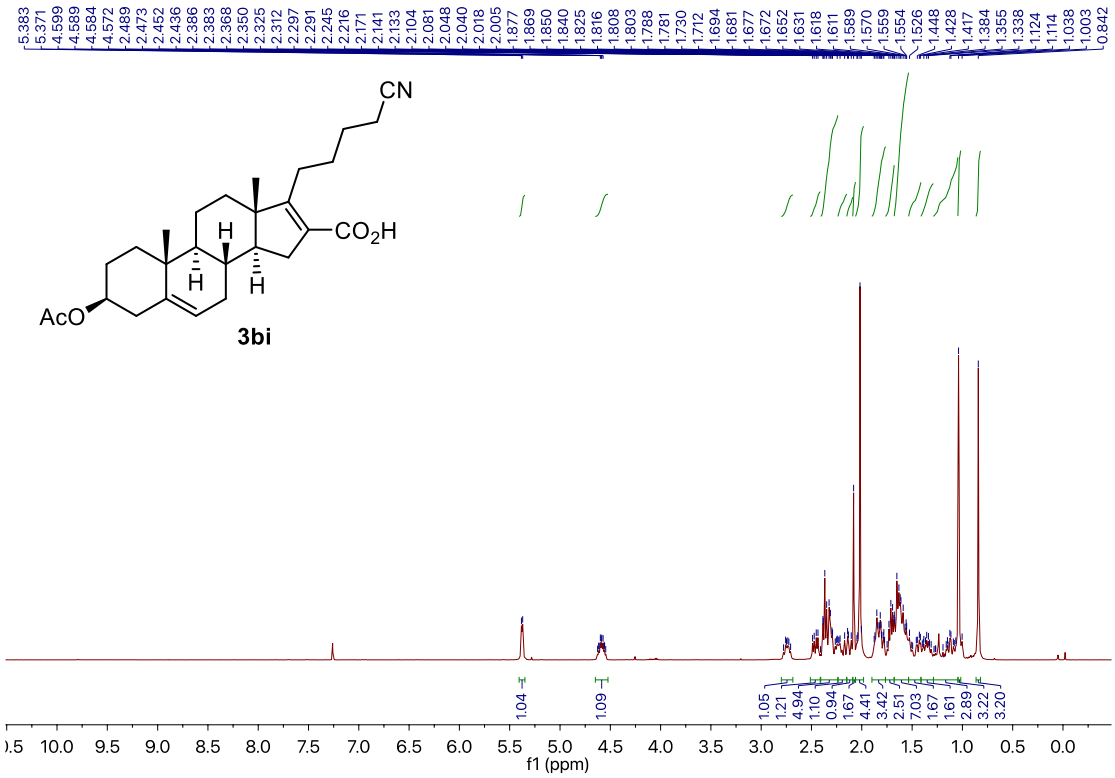
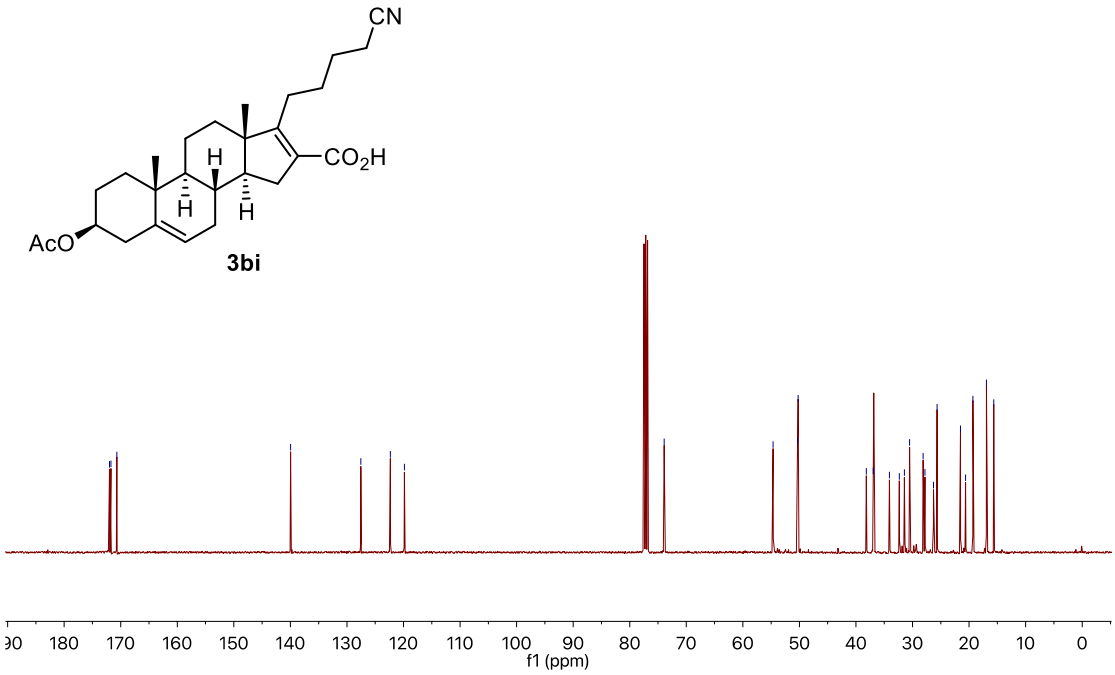
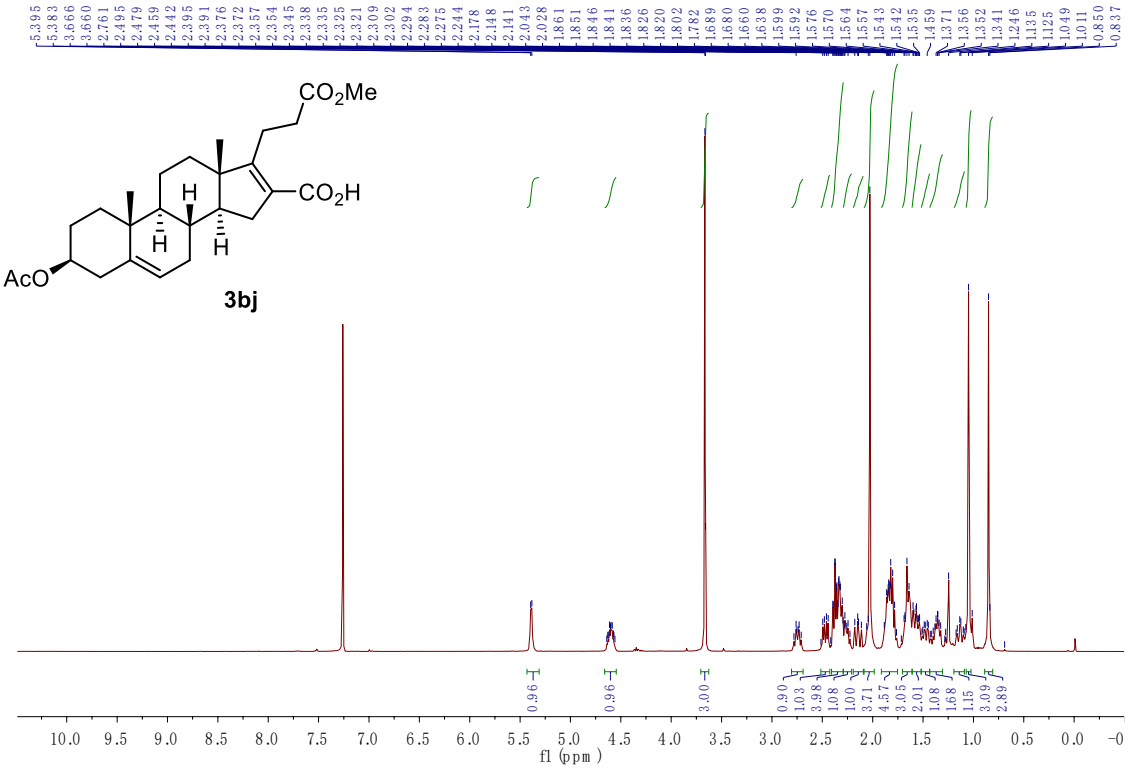
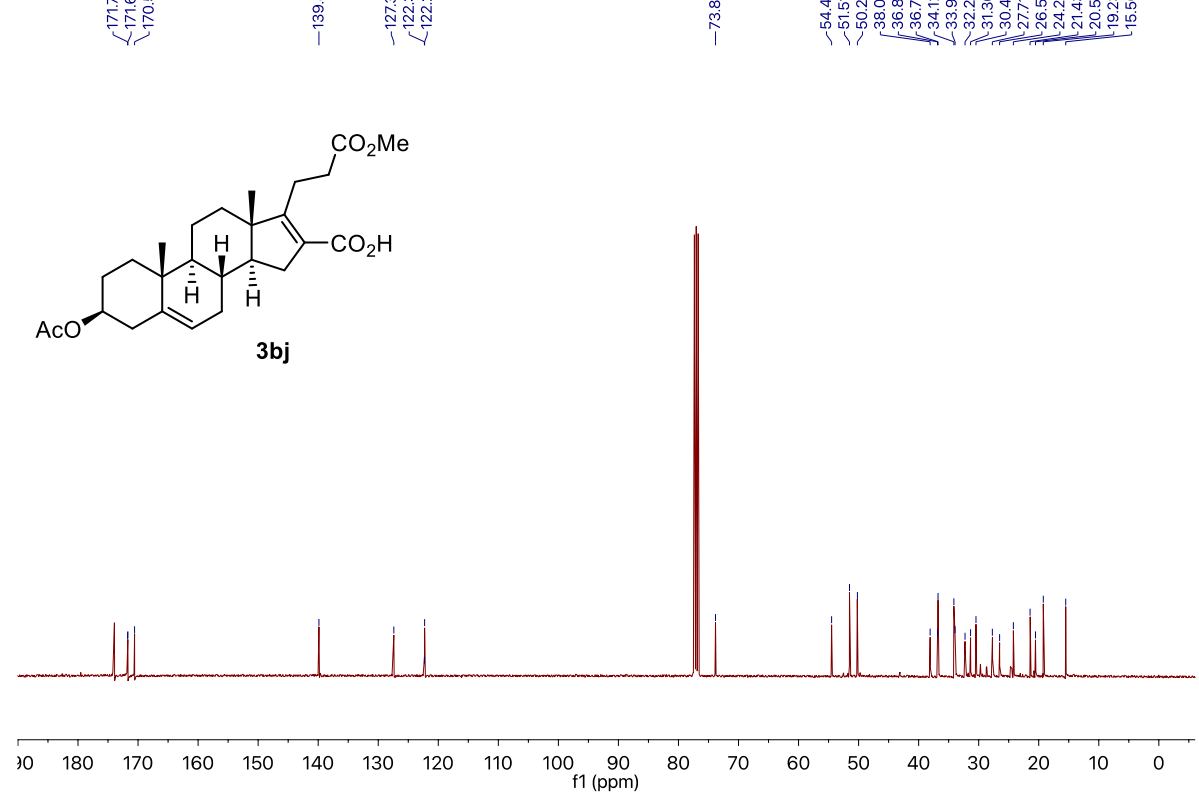
5.126
5.113
4.590
4.032
4.015
3.998
3.890
3.874
3.857
3.027
3.014
2.994
2.981
2.624
2.611
2.591
2.579
2.524
2.508
2.487
2.470
2.341
2.325
2.310
2.266
2.236
2.228
2.096
2.032
1.867
1.857
1.849
1.842
1.833
1.788
1.781
1.768
1.762
1.699
1.689
1.668
1.649
1.631
1.625
1.603
1.596
1.591
1.573
1.569
1.540
1.522
1.507
1.497
1.426
1.410
1.256
1.251
1.139
1.129
1.054
1.035
0.855
O
O
CO2H
H
H
H
AcO
3bh
0.96
0.96
1.02
2.08
2.15
1.05
1.03
1.02
1.91
1.12
1.09
3.55
2.06
1.14
3.66
1.71
2.20
1.05
1.31
0.87
2.98
3.07
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0
f1 (ppm)

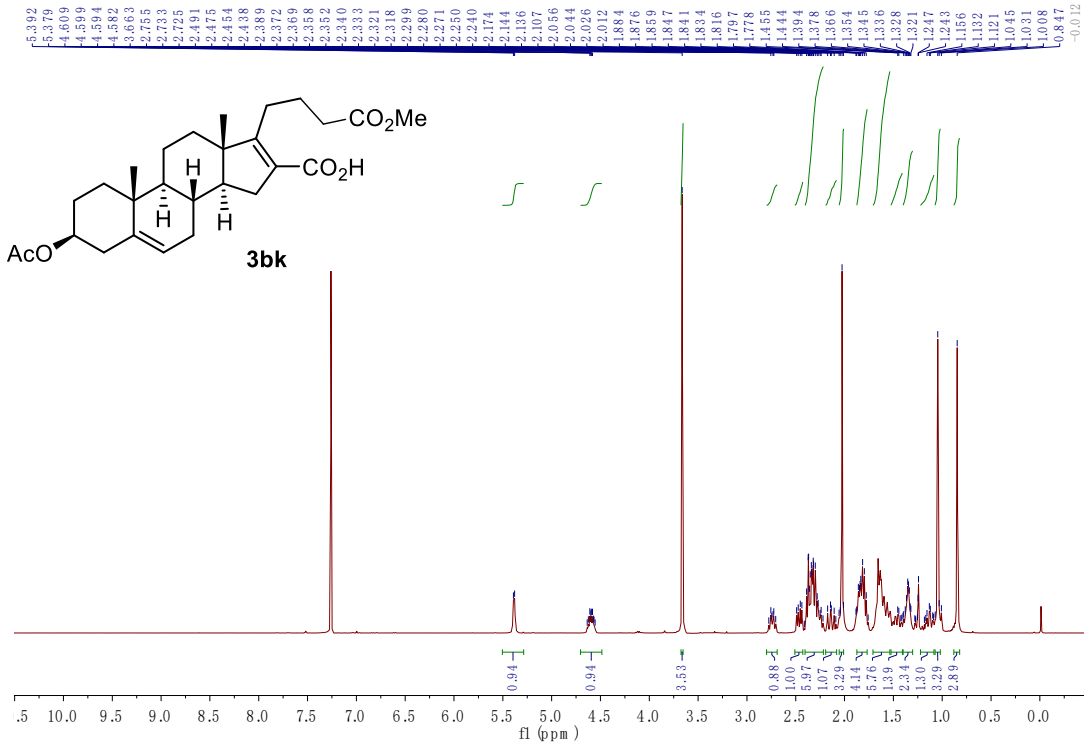

170.74
169.66
162.75
139.99
131.10
122.40
103.31
73.96
65.11
64.95
54.30
50.33
50.16
38.22
37.01
36.90
34.02
33.08
32.25
31.46
30.62
27.86
21.58
20.68
19.37
15.32
O
O
CO2H
H
H
H
AcO
3bh
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

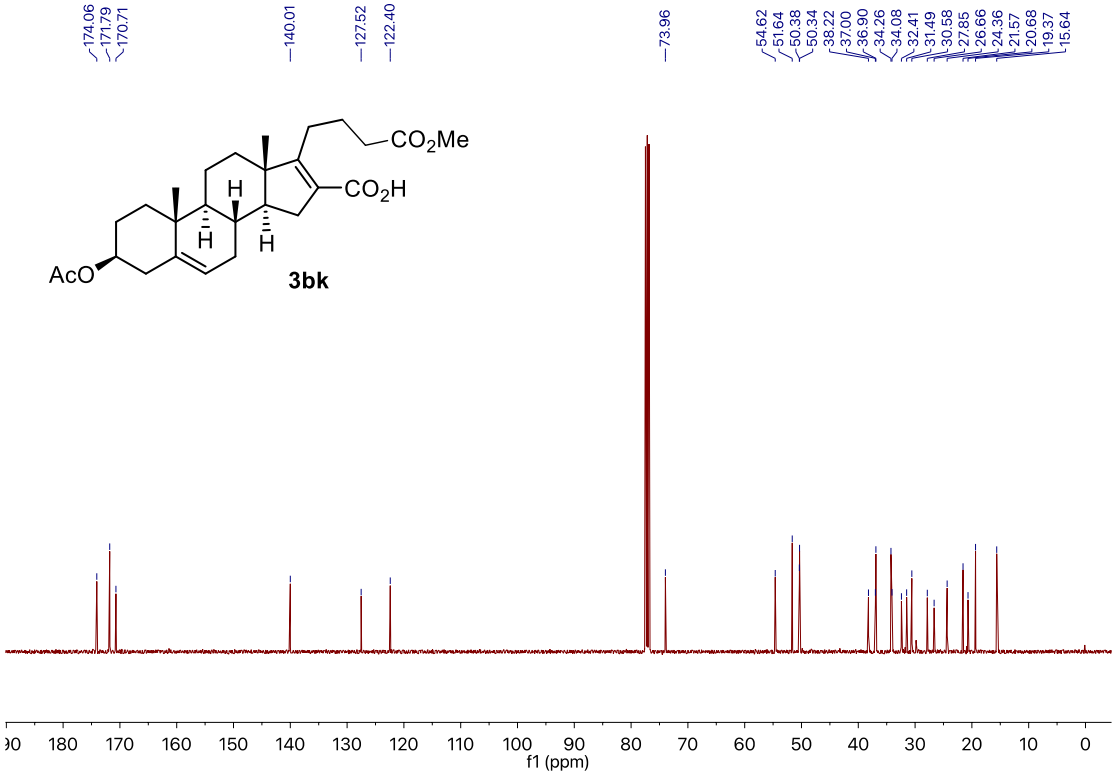

CN
CO2H
H
H
H
AcO
3bi
f1 (ppm)

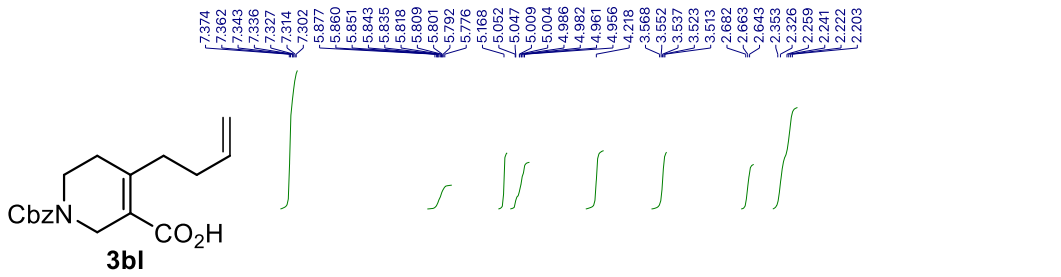

CN
CO2H
H
H
H
AcO
3bi
f1 (ppm)


CO2Me
CO2H
H
H
H
AcO
3bj
fl (ppm)


CO2Me
CO2H
H
H
H
AcO
3bj
f1 (ppm)


CO2Me
CO2H
H
H
H
AcO
3bk
f1 (ppm)


CO2Me
CO2H
H
H
H
AcO
3bk
f1 (ppm)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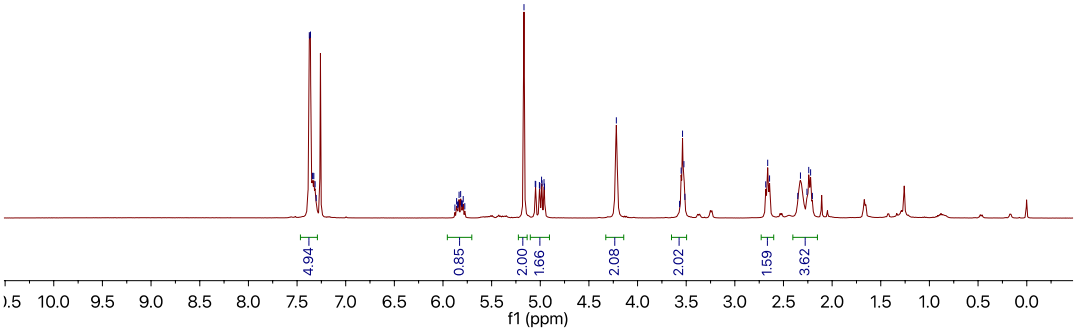
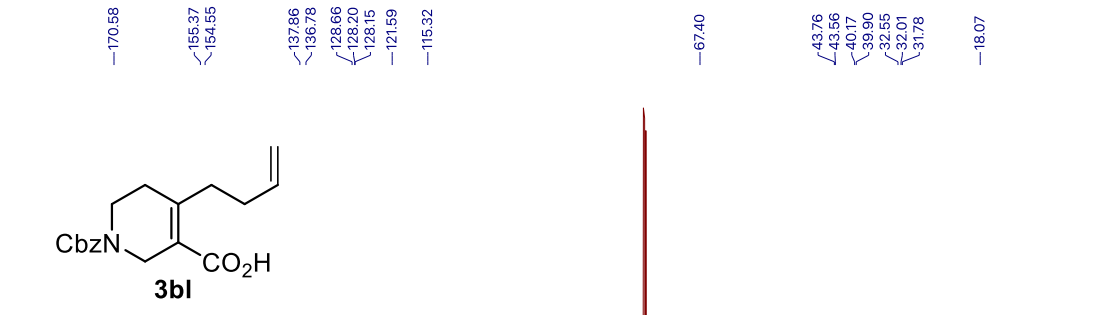
7.343
7.336
7.327
7.314
7.302
5.877
5.860
5.851
5.843
5.835
5.818
5.809
5.801
5.792
5.776
5.168
5.052
5.047
5.009
5.004
4.986
4.982
4.961
4.956
4.218
3.568
3.552
3.537
3.523
3.513
2.682
2.663
2.643
2.353
2.326
2.259
2.241
2.222
2.203

CbzN
CO2H

3bl

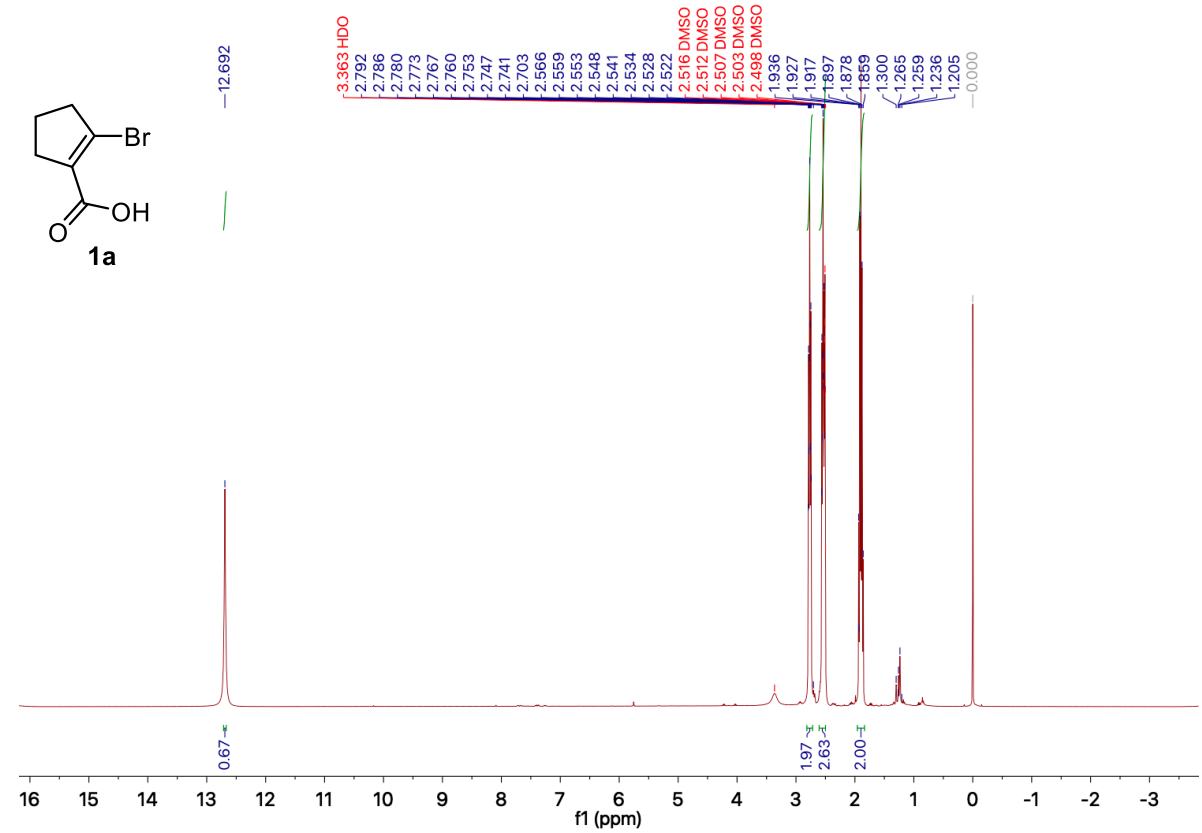

4.94
0.85
2.00
1.66
2.08
2.02
1.59
3.62
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

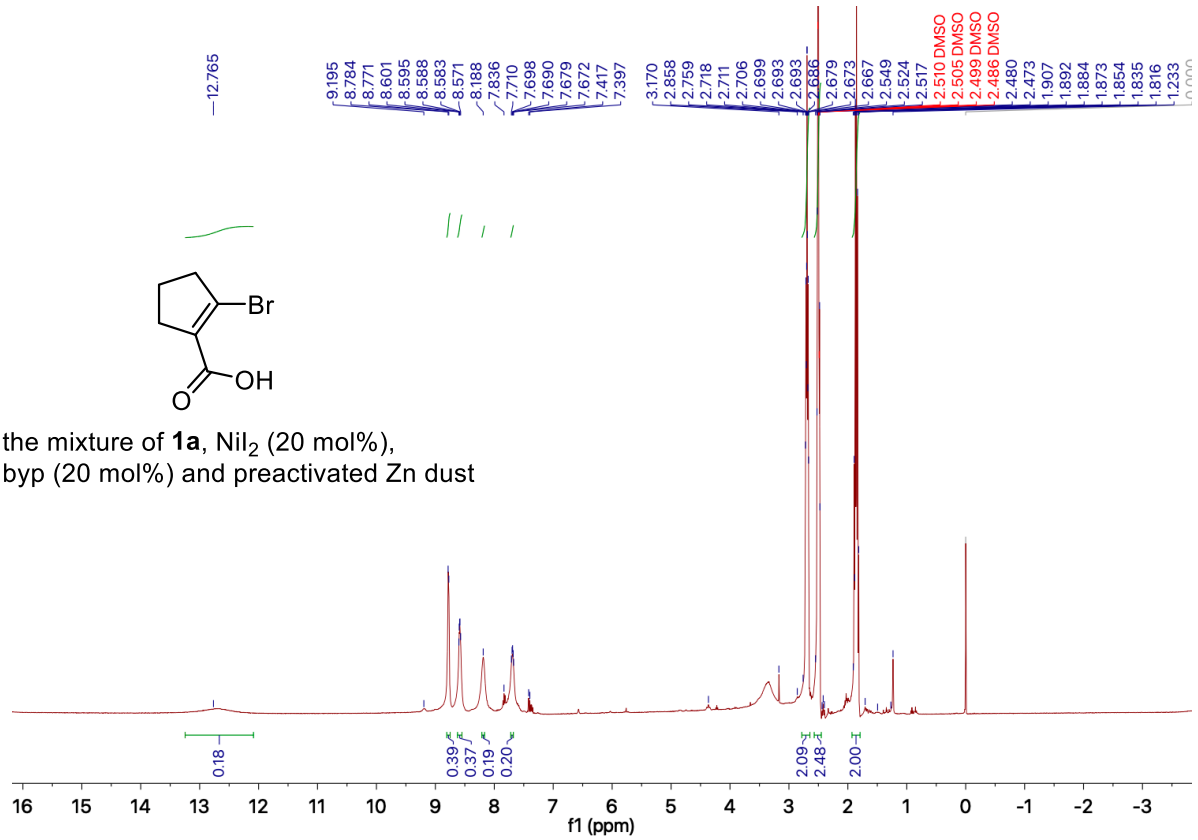

170.58
155.37
154.55
137.86
136.78
128.66
128.20
128.15
121.59
115.32
67.40
43.76
43.56
40.17
39.90
32.55
32.01
31.78
18.07

CbzN
CO2H

3bl

180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)





the mixture of **1a**, NiI_2 (20 mol%),
byp (20 mol%) and preactivated Zn dust