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Supporting Information

for

Transition-metal-catalyst-free electroreductive alkene hydroarylation with aryl halides under visible-light irradiation

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Experimental procedures, characterization data, and copies of NMR spectra of the products

Table of Contents

1. General information

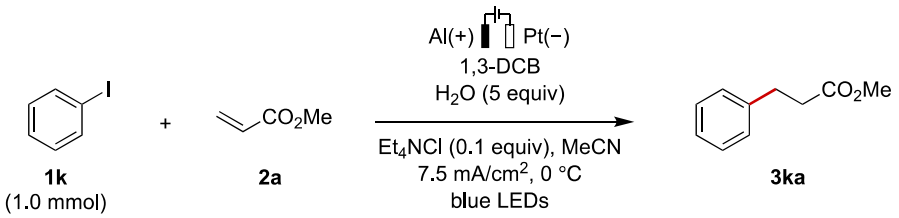
Unless otherwise noted, all reactions were performed under air atmosphere. Electrochemical reactions were carried out using a DC power supply (GP 050-2, *Takasago Ltd.*) with a Coulomb/Amperehour meter (HF-201A, *Hokuto Denko Corporation*). A *Kessil* A160WE Tuna Blue LED lamp (40W) was used for light irradiation. The products were isolated by flash column chromatography (CHROMATOREX 60B, *Fuji silysia*). Melting points (mp) were measured with a *YANAKO* MP-J3 Micro Melting Point Apparatus and reported without correction. Infrared (IR) spectra were recorded on a *SHIMADZU* IRAffinity-1 spectrometer and expressed as frequency of absorption (cm^{-1}). ^{1}H, $^{13}C\{^{1}H\}$, and ^{19}F NMR spectra were recorded on a *JEOL* JNM-ECZ400R (400 MHz for ^{1}H NMR, 100 MHz for $^{13}C\{^{1}H\}$ NMR, and 376 MHz for ^{19}F NMR). Chemical shift values are expressed in parts per million (ppm) relative to internal TMS (δ 0.00 ppm for ^{1}H NMR) and $CDCl_3$ (δ 77.0 ppm for $^{13}C\{^{1}H\}$ NMR). Abbreviations are as follows: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; br, broad. High-resolution mass spectra (HRMS) were recorded on a *JEOL* JMS-T100TD spectrometer using the electrospray ionization (ESI) technique.

Commercially available chemicals were purchased from Sigma-Aldrich, Tokyo Chemical Industry Co., Ltd., Nacalai Tesque, Inc., and FUJIFILM Wako Pure Chemical Corporation and used as received unless otherwise noted. Anhydrous acetonitrile was purchased from FUJIFILM Wako Pure Chemical Corporation (*super dehydrated* grade) and used as received. *Super dehydrated* grade of acetonitrile was dried over 4Å molecular sieves prior to use for a deuterium-labeling experiment.

2. Experimental procedures and characterization data

2.1. Optimization of reaction conditions for aryl iodide 1k

Table S1. Optimization of the reaction conditions

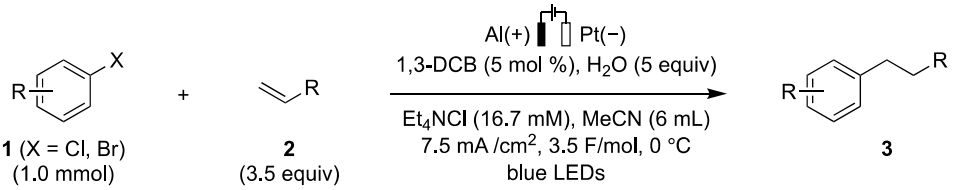


entry	1,3-DCB (mol %)	**2a** (equiv)	MeCN (mL)	charge (F/mol)	yield (%)[a]
1	5	3.5	6	3.5	25
2	50	3.5	6	3.5	61 (8)[b]
3	100	3.5	6	3.5	63
4	50	5	6	3.5	61 (16)[b]
5	50	5	6	4.5	75
6	50	5	3	4.5	75
7[c]	50	5	3	4.5	42

[a]Isolated yield. [b]NMR yield of unreacted **1k** in parentheses.
[c]No LEDs.

2.2. General procedure for the electroreductive hydroarylation of alkenes

General procedure A (for aryl chlorides and bromides)



A cylinder-type undivided cell with a stir bar was charged with Et_4NCl (16.6 mg, 0.1 mmol), 1,3-dicyanobenzene (1,3-DCB, 6.4 mg, 0.05 mmol), alkene **2** (3.5 mmol), and aryl halide **1** (1.0 mmol). Anhydrous MeCN (6.0 mL) and H_2O (90 μL) were successively added. The reaction vessel was equipped with a Pt plate cathode (immersed surface area: 1 × 2 cm^2) and an Al plate anode (immersed surface area: 1 × 2 cm^2) under air, then 3.5 F/mol of electricity was passed through at a constant current condition (7.5 mA/cm^2) under visible-light irradiation (approximate distance from the cathode: 2.5 cm) at 0 °C in an ice bath (Figure S1). Most of solvent was removed under reduced pressure, and the residue was diluted with AcOEt. The organic layer was washed with H_2O, dried over Na_2SO_4, filtered, and concentrated under reduced pressure. The residue was purified by flash silica gel column chromatography to afford the desired product **3**.

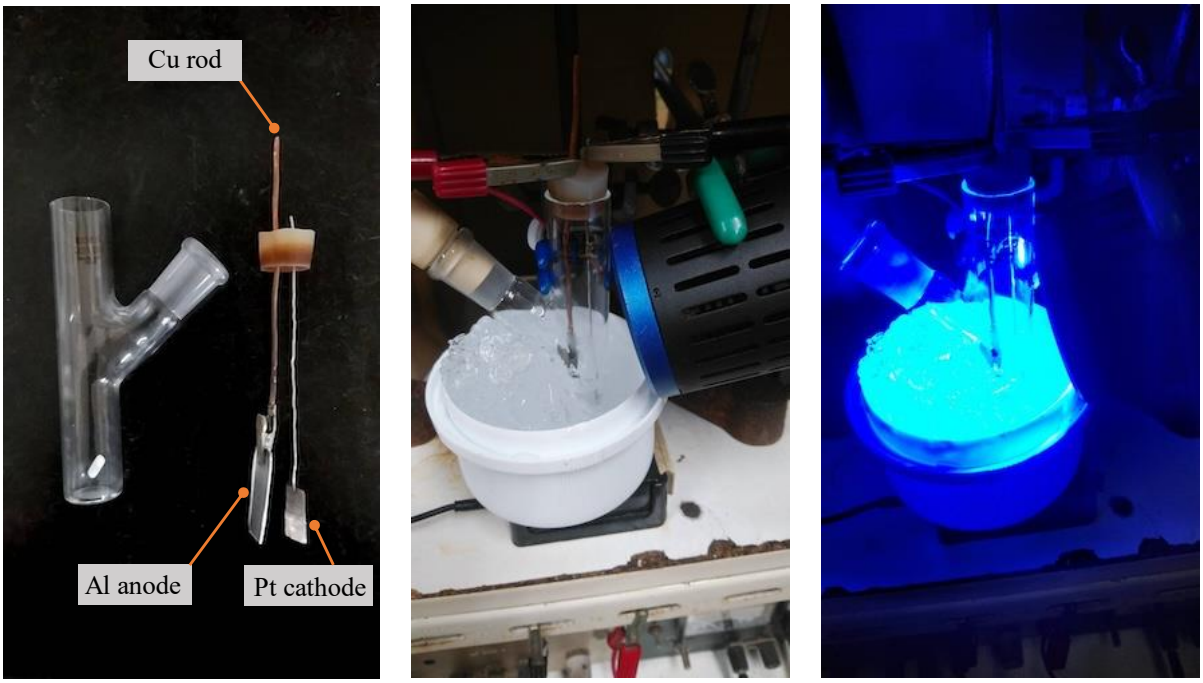
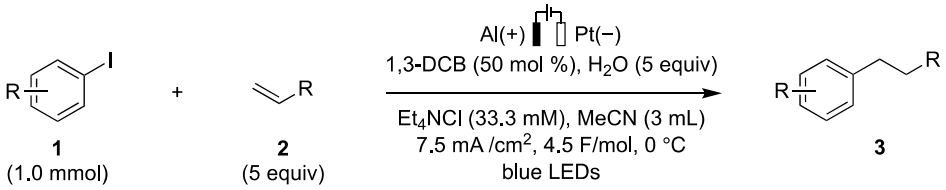


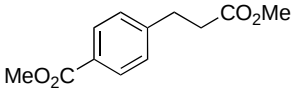
Figure S1. A reaction vessel and a silicone stopper equipped with Al and Pt electrodes (left) and a typical reaction setup for the electroreductive hydroarylation under visible-right irradiation (middle and right).

General procedure B (for aryl iodides)



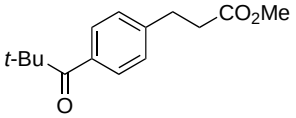
A cylinder-type undivided cell with a stir bar was charged with Et_4NCl (16.6 mg, 0.1 mmol), 1,3-dicyanobenzene (1,3-DCB, 64.1 mg, 0.5 mmol), alkene **2** (5.0 mmol), and aryl iodide **1** (1.0 mmol). Anhydrous MeCN (3.0 mL) and H_2O (90 μL) were successively added. The reaction vessel was equipped with a Pt plate cathode (immersed surface area: 1 × 2 cm^2) and an Al plate anode (immersed surface area: 1 × 2 cm^2) under air, then 4.5 F/mol of electricity was passed through at a constant current condition (7.5 mA/cm^2) under visible-light irradiation (approximate distance from the cathode: 2.5 cm) at 0 °C in an ice bath. Most of solvent was removed under reduced pressure, and the residue was diluted with AcOEt. The organic layer was washed with H_2O, dried over Na_2SO_4, filtered, and concentrated under reduced pressure. The residue was purified by flash silica gel column chromatography to afford the desired product **3**.

Methyl 4-(3-methoxy-3-oxopropyl)benzoate (3aa)



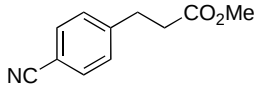
The title compound was obtained from methyl 4-chlorobenzoate (171 mg, 1.0 mmol) and methyl acrylate (301 mg, 3.5 mmol). Silica gel column chromatography (hexane/AcOEt = 8:1) gave **3aa** (183.3 mg, 0.825 mmol, 82%) as a colorless oil. ^1H NMR (400 MHz, $CDCl_3$): δ 7.98-7.95 (m, 2H), 7.28-7.26 (m, 2H), 3.90 (s, 3H), 3.67 (s, 3H), 3.01 (t, J = 7.7 Hz, 2H), 2.66 (t, J = 7.8 Hz, 2H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 172.8, 166.8, 145.8, 129.7, 128.2, 128.1, 51.8, 51.5, 35.0, 30.7.
The ^1H and $^{13}C\{^1H\}$ NMR spectra are in accordance with those reported in the literature.[1]

Methyl 3-(4-pivaloylphenyl)propanoate (3ba)



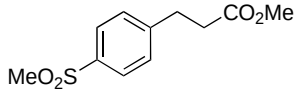
The title compound was obtained from 1-(4-chlorophenyl)-2,2-dimethyl-1-propanone (197 mg, 1.0 mmol) and methyl acrylate (301 mg, 3.5 mmol) with 4.5 F/mol of electricity. Silica gel column chromatography (hexane/AcOEt = 20:1) gave **3ba** (202.7 mg, 0.816 mmol, 82%) as a colorless oil. ^1H NMR (400 MHz, $CDCl_3$): δ 7.69-7.66 (m, 2H), 7.23 (d, J = 8.2 Hz, 2H), 3.68 (s, 3H), 2.99 (t, J = 7.8 Hz, 2H), 2.65 (t, J = 7.8 Hz, 2H), 1.35 (s, 9H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 208.1, 172.8, 143.7, 136.1, 128.3, 127.8, 51.5, 43.9, 35.0, 30.5, 27.9; IR (ATR): 2953, 1736, 1670, 1607, 1275, 1171, 959 cm^{-1}; HRMS (ESI) *m/z*: $[M+Na]^+$ calcd for $C_{15}H_{20}NaO_3$ 271.1310, found 271.1316.

Methyl 3-(4-cyanophenyl)propanoate (3ca)



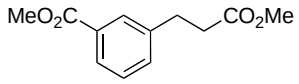
The title compound was obtained from 4-chlorobenzonitrile (138 mg, 1.0 mmol) and methyl acrylate (301 mg, 3.5 mmol) with 4.5 F/mol of electricity. Silica gel column chromatography (hexane/AcOEt = 4:1) gave **3ca** (114.6 mg, 0.606 mmol, 61%) as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.60-7.58 (m, 2H), 7.33-7.31 (m, 2H), 3.67 (s, 3H), 3.01 (t, J = 7.6 Hz, 2H), 2.66 (t, J = 7.6 Hz, 2H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 172.6, 146.0, 132.3, 129.1, 118.8, 110.2, 51.7, 34.7, 30.8.
The ^{1}H and ^{13}C{^{1}H} NMR spectra are in accordance with those reported in the literature.[2]

Methyl 3-(4-(methylsulfonyl)phenyl)propanoate (3da)



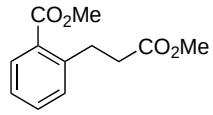
The title compound was obtained from 4-bromophenyl methyl sulfone (235 mg, 1.0 mmol) and methyl acrylate (301 mg, 3.5 mmol). Silica gel column chromatography (hexane/AcOEt = 2:1) gave **3da** (170.8 mg, 0.705 mmol, 71%) as a white solid. mp 67–68 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.89-7.85 (m, 2H), 7.42-7.40 (m, 2H), 3.68 (s, 3H), 3.051 (t, J = 7.7 Hz, 2H), 3.046 (s, 3H), 2.68 (t, J = 7.7 Hz, 2H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 172.4, 146.8, 138.3, 129.1, 127.3, 51.5, 44.2, 34.6, 30.3; IR (ATR): 2911, 1730, 1597, 1290, 1141, 1087 cm^{-1}; HRMS (ESI) *m/z*: $[M+Na]^+$ calcd for $C_{11}H_{14}NaO_4S$ 265.0511, found 265.0513.
The ^{1}H and ^{13}C{^{1}H} NMR spectra are in accordance with those reported in the literature.[3]

Methyl 3-(3-methoxy-3-oxopropyl)benzoate (3ea)



The title compound was obtained from methyl 3-bromobenzoate (215 mg, 1.0 mmol) and methyl acrylate (301 mg, 3.5 mmol). Silica gel column chromatography (hexane/AcOEt = 20:1) gave **3ea** (182.3 mg, 0.820 mmol, 82%) as a yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.90-7.87 (m, 2H), 7.42-7.34 (m, 2H), 3.91 (s, 3H), 3.67 (s, 3H), 3.00 (t, J = 7.8 Hz, 2H), 2.66 (t, J = 7.8 Hz, 2H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 172.9, 166.9, 140.7, 132.9, 130.2, 129.2, 128.4, 127.5, 52.0, 51.5, 35.3, 30.5; IR (ATR): 2953, 1719, 1435, 1281, 1198 cm^{-1}; HRMS (ESI) *m/z*: $[M+Na]^+$ calcd for $C_{12}H_{14}NaO_4$ 245.0790, found 245.0778.

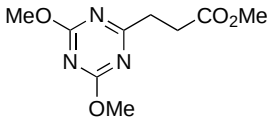
Methyl 2-(3-methoxy-3-oxopropyl)benzoate (3fa)



The title compound was obtained from methyl 2-chlorobenzoate (171 mg, 1.0 mmol) and methyl

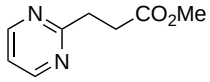
acrylate (431 mg, 5.0 mmol) with 4.5 F/mol of electricity using MeCN (3.0 mL). Silica gel column chromatography (hexane/AcOEt/CH_2Cl_2 = 12:2:1) gave **3fa** (156.9 mg, 0.706 mmol, 71%) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.93-7.91 (m, 1H), 7.46-7.42 (m, 1H), 7.30-7.26 (m, 2H), 3.90 (s, 3H), 3.67 (s, 3H), 3.28 (t, J = 7.8 Hz, 2H), 2.68 (t, J = 7.8 Hz, 2H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 173.2, 167.4, 142.3, 132.1, 131.0, 130.7, 129.1, 126.3, 51.8, 51.3, 35.4, 29.7.
The ^{1}H and ^{13}C{^{1}H} NMR spectra are in accordance with those reported in the literature.[4]

Methyl 3-(4,6-dimethoxy-1,3,5-triazin-2-yl)propanoate (3ga)



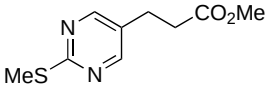
The title compound was obtained from 2-chloro-4,6-dimethoxy-1,3,5-triazine (176 mg, 1.0 mmol) and methyl acrylate (301 mg, 3.5 mmol) with 5 F/mol of electricity. Silica gel column chromatography (hexane/AcOEt = 3:1) gave **3ga** (135.6mg, 0.597 mmol, 60%) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 4.02 (s, 6H), 3.69 (s, 3H), 3.10 (t, J = 7.1 Hz, 2H), 2.85 (t, J = 7.0 Hz, 2H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 181.2, 172.9, 172.1, 54.9, 51.5, 32.8, 30.0; IR (ATR): 2959, 1728, 1543, 1340, 1277, 1171, 1105 cm^{-1}; HRMS (ESI) *m/z*: $[M+Na]^+$ calcd for $C_9H_{13}N_3NaO_4$ 250.0804, found 250.0797.

Methyl 3-(pyrimidin-2-yl)propanoate (3ha)



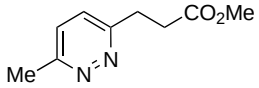
The title compound was obtained from 2-chloropyrimidine (115 mg, 1.0 mmol) and methyl acrylate (301 mg, 3.5 mmol). Silica gel column chromatography (hexane/AcOEt = 4:1) gave **3ha** (94.6 mg, 0.569 mmol, 57%) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.66 (d, J = 5.0 Hz, 2H), 7.14 (t, J = 4.9 Hz, 1H), 3.69 (s, 3H), 3.32 (t, J = 7.2 Hz, 2H), 2.90 (t, J = 7.2 Hz, 2H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 173.3, 169.2, 156.8, 118.6, 51.5, 33.6, 31.3; IR (ATR): 2953, 1730, 1562, 1427, 1368, 1167 cm^{-1}; HRMS (ESI) *m/z*: $[M+Na]^+$ calcd for $C_8H_{10}N_2NaO_2$ 189.0640, found 189.0643.

Methyl 3-(2-(methylthio)pyrimidin-5-yl)propanoate (3ia)



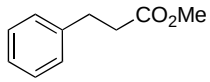
The title compound was obtained from 5-chloro-2-(methylthio)pyrimidine (161 mg, 1.0 mmol) and methyl acrylate (301 mg, 3.5 mmol) with 4.5 F/mol of electricity. Silica gel column chromatography (hexane/AcOEt = 4:1) gave **3ia** (140.0 mg, 0.660 mmol, 66%) as a yellow solid. mp 37–38 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.41 (s, 2H), 3.68 (s, 3H), 2.87 (t, J = 7.3 Hz, 2H), 2.63 (t, J = 7.3 Hz, 2H), 2.56 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 172.1, 170.3, 156.9, 128.0, 51.6, 34.5, 24.7, 13.8; IR (ATR): 2918, 1728, 1581, 1537, 1389, 1206, 1167 cm^{-1}; HRMS (ESI) *m/z*: $[M+Na]^+$ calcd for $C_9H_{12}N_2NaO_2S$ 235,0517, found 235.0515.

Methyl 3-(6-methylpyridazin-3-yl)propanoate (3ja)



The title compound was obtained from 3-chloro-6-methylpyridazine (129 mg, 1.0 mmol) and methyl acrylate (301 mg, 3.5 mmol). Silica gel column chromatography (AcOEt 100%) gave **3ja** (110.6 mg, 0.614 mmol, 61%) as a colorless viscous oil which solidified in a freezer. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.28 (d, J = 8.7 Hz, 1H), 7.23 (d, J = 8.5 Hz, 1H), 3.67 (s, 3H), 3.23 (t, J = 7.2 Hz, 2H), 2.93 (t, J = 7.2 Hz, 2H), 2.68 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 173.1, 159.1, 158.0, 126.8, 126.6, 51.5, 32.4, 30.4, 21.8; IR (ATR): 1953, 1728, 1443, 1361, 1206, 1161 cm^{-1}; HRMS (ESI) *m/z*: $[M+Na]^+$ calcd for $C_9H_{12}N_2NaO_2$ 203.0797, found 203.0791.

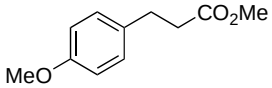
Methyl 3-phenylpropanoate (3ka)



The title compound was obtained from iodobenzene (204 mg, 1.0 mmol) and methyl acrylate (431 mg, 5.0 mmol). Silica gel column chromatography (hexane/AcOEt = 20:1) gave **3ka** (123.4mg, 0.752 mmol, 75%) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.31-7.27 (m, 2H), 7.22-7.19 (m, 3H), 3.67 (s, 3H), 2.96 (t, J = 7.9 Hz, 2H), 2.66-2.62 (m, 2H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 173.2, 140.4, 128.4, 128.2, 126.2, 51.5, 35.6, 30.8.

The ^{1}H and ^{13}C{^{1}H} NMR spectra are in accordance with those reported in the literature.[2]

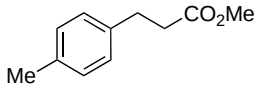
Methyl 3-(4-methoxyphenyl)propanoate (3la)



The title compound was obtained from 4-iodoanisole (234 mg, 1.0 mmol) and methyl acrylate (431 mg, 5.0 mmol). Silica gel column chromatography (hexane/AcOEt = 20:1) gave **3la** (126.8 mg, 0.653 mmol, 65%) as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.14-7.10 (m, 2H), 6.85-6.82 (m, 2H), 3.79 (s, 3H), 3.67 (s, 3H), 2.89 (t, J = 7.9 Hz, 2H), 2.60 (t, J = 7.8 Hz, 2H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 173.3, 157.9, 132.4, 129.1, 113.7, 55.0, 51.4, 35.9, 29.9.

The ^{1}H and ^{13}C{^{1}H} NMR spectra are in accordance with those reported in the literature.[2]

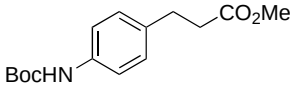
Methyl 3-(p-tolyl)propanoate (3ma)



The title compound was obtained from 4-iodotoluene (218 mg, 1.0 mmol) and methyl acrylate (431 mg, 5.0 mmol). Silica gel column chromatography (hexane/AcOEt = 20:1) gave **3ma** (134.0 mg, 0.752 mmol, 75%) as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.12-7.07 (m, 4H), 3.67 (s, 3H), 2.91 (t, J = 7.8 Hz, 2H), 2.61 (t, J = 7.8 Hz, 2H), 2.32 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 173.4,

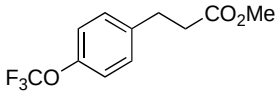
137.4, 135.7, 129.1, 128.1, 51.6, 35.8, 30.5, 21.0.
The ^{1}H and $^{13}C\{^{1}H\}$ NMR spectra are in accordance with those reported in the literature.[2]

Methyl 3-(4-(tert-butoxycarbonylamino)phenyl)propanoate (3na)



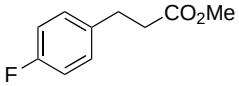
The title compound was obtained from *N*-Boc-4-iodoaniline (319 mg, 1.0 mmol) and methyl acrylate (431 mg, 5.0 mmol). Silica gel column chromatography (hexane/AcOEt = 8:1) gave **3na** (170.4 mg, 0.610 mmol, 61%) as a white solid. mp 65–66 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.28-7.26 (m, 2H), 7.12 (d, J = 8.5 Hz, 2H), 6.41 (br s, 1H), 3.66 (s, 3H), 2.90 (t, J = 7.8 Hz, 2H), 2.59 (t, J = 7.8 Hz, 2H), 1.51 (s, 9H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$): δ 173.3, 152.8, 136.5, 135.1, 128.7, 118.7, 80.4, 51.6, 35.8, 30.2, 28.3; IR (ATR): 3343, 2980, 1715, 1699, 1525, 1234, 1158 cm^{-1}; HRMS (ESI) *m/z*: $[M+Na]^+$ calcd for $C_{15}H_{21}NNaO_4$ 302.1368, found 302.1362.
The ^{1}H NMR spectrum is in accordance with those reported in the literature.[5]

Methyl 3-(4-(trifluoromethoxy)phenyl)propanoate (3oa)



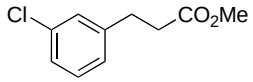
The title compound was obtained from 1-iodo-4-(trifluoromethoxy)benzene (288 mg, 1.0 mmol) and methyl acrylate (431 mg, 5.0 mmol). Silica gel column chromatography (hexane/AcOEt = 20:1) gave **3oa** (161.1 mg, 0.649 mmol, 65%) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.24-7.20 (m, 2H), 7.15-7.12 (m, 2H), 3.67 (s, 3H), 2.96 (t, J = 7.8 Hz, 2H), 2.63 (t, J = 7.7 Hz, 2H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$): δ 173.0, 147.7 (q, J = 1.9 Hz), 139.2, 129.6, 121.0, 120.4 (q, J = 256.6 Hz), 51.6, 35.4, 30.1; ^{19}F NMR (376 MHz, $CDCl_3$): δ −57.8.
The ^{1}H and $^{13}C\{^{1}H\}$ NMR spectra are in accordance with those reported in the literature.[6]

Methyl 3-(4-fluorophenyl)propanoate (3pa)



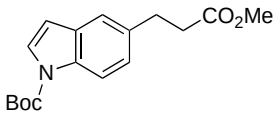
The title compound was obtained from 4-fluoroiodobenzene (222 mg, 1.0 mmol) and methyl acrylate (431 mg, 5.0 mmol). Silica gel column chromatography (hexane/AcOEt = 20:1) gave **3pa** (93.1 mg, 0.511 mmol, 51%) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.18-7.13 (m, 2H), 7.00-6.94 (m, 2H), 3.67 (s, 3H), 2.92 (t, J = 7.7 Hz, 2H), 2.61 (t, J = 7.7 Hz, 2H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$): δ 173.1, 161.4 (d, J = 243.7 Hz), 136.0 (d, J = 3.9 Hz), 129.6 (d, J = 7.7 Hz), 115.1 (d, J = 21.2 Hz), 51.5, 35.7, 30.0; ^{19}F NMR (376 MHz, $CDCl_3$): δ −116.9.
The ^{1}H and $^{13}C\{^{1}H\}$ NMR spectra are in accordance with those reported in the literature.[2]

Methyl 3-(3-chlorophenyl)propanoate (3qa)



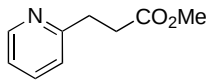
The title compound was obtained from 3-chloroiodobenzene (239 mg, 1.0 mmol) and methyl acrylate (431 mg, 5.0 mmol). Silica gel column chromatography (hexane/AcOEt = 20:1) gave **3qa** (100.0 mg, 0.503 mmol, 50%) as a yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.24-7.17 (m, 3H), 7.10-7.07 (m, 1H), 3.68 (s, 3H), 2.93 (t, J = 7.8 Hz, 2H), 2.63 (t, J = 7.8 Hz, 2H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 173.0, 142.5, 134.2, 129.7, 128.4, 126.5, 51.7, 35.3, 30.5.
The ^{1}H and ^{13}C{^{1}H} NMR spectra are in accordance with those reported in the literature.[7]

tert-Butyl 5-(3-methoxy-3-oxopropyl)-1H-indole-1-carboxylate (3ra)



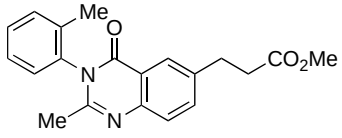
The title compound was obtained from *N*-Boc-5-iodoindole (343 mg, 1.0 mmol) and methyl acrylate (431 mg, 5.0 mmol). Silica gel column chromatography (hexane/AcOEt = 20:1) gave **3ra** (134.0 mg, 0.442 mmol, 44%) as a colorless viscous oil which solidified in a freezer. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.04 (d, J = 8.2 Hz, 1H), 7.57 (d, J = 3.4 Hz, 1H), 7.38 (d, J = 1.1 Hz, 1H), 7.15 (dd, J = 8.6, 1.7 Hz, 1H), 6.51 (d, J = 3.7 Hz, 1H), 3.66 (s, 3H), 3.04 (t, J = 7.8 Hz, 2H), 2.67 (t, J = 7.9 Hz, 2H), 1.66 (s, 9H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 173.4, 149.7, 134.8, 133.8, 130.8, 126.1, 124.6, 120.2, 115.0, 107.0, 83.5, 51.5, 36.2, 30.8, 28.1; IR (ATR): 2974, 1728, 1468, 1350, 1157, 1128 cm^{-1}; HRMS (ESI) *m/z*: $[M+Na]^+$ calcd for $C_{17}H_{21}NNaO_4$ 326.1368, found 326.1367.

Methyl 3-(pyridine-2-yl)propanoate (3sa)



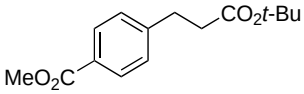
The title compound was obtained from 2-iodopyridine (205 mg, 1.0 mmol) and methyl acrylate (431 mg, 5.0 mmol). Silica gel column chromatography (hexane/AcOEt = 4:1) gave **3sa** (127.9 mg, 0.774 mmol, 77%) as a yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.53 (ddd, J = 4.9, 1.7, 0.9 Hz, 1H), 7.60 (td, J = 7.7, 1.8 Hz, 1H), 7.19 (d, J = 7.8 Hz, 1H), 7.12 (ddd, J = 7.5, 4.9, 0.9 Hz, 1H), 3.67 (s, 3H), 3.12 (t, J = 7.5 Hz, 2H), 2.82 (t, J = 7.5 Hz, 2H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 173.4, 159.8, 149.2, 136.3, 122.9, 121.3, 51.5, 33.1, 32.7.
The ^{1}H and ^{13}C{^{1}H} NMR spectra are in accordance with those reported in the literature.[8]

Methyl 3-(2-methyl-4-oxo-3-(o-tolyl)-3,4-dihydroquinazolin-6-yl)propanoate (3ta)



The title compound was obtained from 6-iodo-2-methyl-3-(*o*-tolyl)quinazolin-4(3*H*)-one (376 mg, 1.0 mmol) and methyl acrylate (431 mg, 5.0 mmol). Silica gel column chromatography (hexane/AcOEt = 2:1, then benzene/AcOEt = 4/1) gave **3ta** (180.0 mg, 0.535 mmol, 54%) as a yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.10 (d, J = 0.3 Hz, 1H), 7.66-7.61 (m, 2H), 7.44-7.35 (m, 3H), 7.15 (d, J = 7.3 Hz, 1H), 3.68 (s, 3H), 3.10 (t, J = 7.7 Hz, 2H), 2.71 (t, J = 7.7 Hz, 2H), 2.17 (s, 3H), 2.13 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 172.9, 161.5, 153.8, 146.2, 139.2, 136.8, 135.3, 135.2, 131.5, 129.5, 127.8, 127.6, 126.9, 125.9, 120.6, 51.7, 35.3, 30.5, 23.8, 17.3; IR (ATR): 2951, 1732, 1676, 1597, 1487, 1269, 1200, 1165 cm^{-1}; HRMS (ESI) *m/z*: [M+Na]$^+$ calcd for $C_{20}H_{20}N_2NaO_3$ 359.1372, found 359.1379.

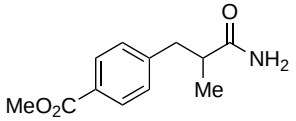
Methyl 4-(3-(tert-butoxy)-3-oxopropyl)benzoate (3ab)



The title compound was obtained from methyl 4-chlorobenzoate (171 mg, 1.0 mmol) and *tert*-butyl acrylate (449 mg, 3.5 mmol). Silica gel column chromatography (hexane/AcOEt = 8:1) gave **3ab** (206.7 mg, 0.782 mmol, 78%) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.97-7.94 (m, 2H), 7.27 (d, J = 8.2 Hz, 5H), 3.90 (s, 3H), 2.96 (t, J = 7.7 Hz, 2H), 2.56 (t, J = 7.7 Hz, 2H), 1.41 (s, 9H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 171.8, 167.0, 146.2, 129.7, 128.3, 128.1, 80.5, 51.9, 36.4, 31.0, 28.0; IR (ATR): 2978, 1719, 1611, 1435, 1366, 1275, 1144, 1103 cm^{-1}; HRMS (ESI) *m/z*: [M+Na]$^+$ calcd for $C_{15}H_{20}NaO_4$ 287.1259, found 287.1252.

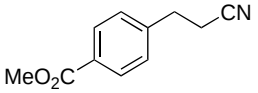
The ^{1}H NMR spectrum is in accordance with those reported in the literature.[9]

Methyl 4-(3-amino-2-methyl-3-oxopropyl)benzoate (3ac)



The title compound was obtained from methyl 4-chlorobenzoate (171 mg, 1.0 mmol) and methacrylamide (298 mg, 3.5 mmol) with 5 F/mol of electricity. Silica gel column chromatography (AcOEt 100%) gave **3ac** (175.0 mg, 0.791 mmol, 79%) as a white solid. mp 146–147 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.96 (d, J = 8.2 Hz, 2H), 7.27 (d, J = 8.2 Hz, 2H), 5.25 (br s, 2H), 3.90 (s, 3H), 3.06 (dd, J = 13.5, 8.0 Hz, 1H), 2.74 (dd, J = 13.5, 6.6 Hz, 1H), 2.53 (dqd, J = 8.0, 6.9, 6.6 Hz, 1H), 1.21 (d, J = 6.9 Hz, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 177.7, 167.0, 145.1, 129.7, 129.0, 128.3, 52.0, 42.6, 40.0, 17.7; IR (ATR): 3410, 3194, 2974, 1682, 1661, 1287, 1179, 1115 cm^{-1}; HRMS (ESI) *m/z*: [M+Na]$^+$ calcd for $C_{12}H_{15}NNaO_3$ 244.0950, found 244.0945.

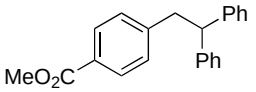
Methyl 4-(2-cyanoethyl)benzoate (3ad)



The title compound was obtained from methyl 4-chlorobenzoate (171 mg, 1.0 mmol) and acrylonitrile

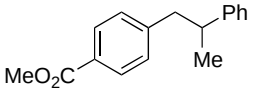
(186 mg, 3.5 mmol) with 4.5 F/mol of electricity. Silica gel column chromatography (hexane/AcOEt = 4:1) gave **3ad** (154.6 mg, 0.817 mmol, 82%) as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.03-8.01 (m, 2H), 7.32 (d, J = 8.2 Hz, 2H), 3.92 (s, 3H), 3.02 (t, J = 7.3 Hz, 2H), 2.66 (t, J = 7.3 Hz, 2H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 166.5, 143.0, 129.9, 128.9, 128.2, 118.6, 51.9, 31.2, 18.7.
The ^{1}H and ^{13}C{^{1}H} NMR spectra are in accordance with those reported in the literature.[10]

Methyl 4-(2,2-diphenylethyl)benzoate (3ae)



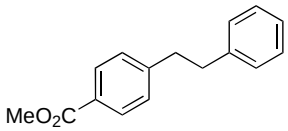
The title compound was obtained from methyl 4-chlorobenzoate (171 mg, 1.0 mmol) and 1,1-diphenylethylene (631 mg, 3.5 mmol). Silica gel column chromatography (hexane/AcOEt = 60:1) gave **3ae** (250.8 mg, 0.793 mmol, 79%) as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85-7.82 (m, 2H), 7.27-7.23 (m, 4H), 7.20-7.14 (m, 6H), 7.06 (d, J = 8.5 Hz, 2H), 4.23 (t, J = 7.9 Hz, 1H), 3.87 (s, 3H), 3.41 (d, J = 7.8 Hz, 2H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 166.9, 145.6, 143.8, 129.3, 129.0, 128.3, 127.8, 127.7, 126.2, 52.7, 51.8, 41.9.
The ^{1}H and ^{13}C{^{1}H} NMR spectra are in accordance with those reported in the literature.[11]

Methyl 4-(2-phenylpropyl)benzoate (3af)



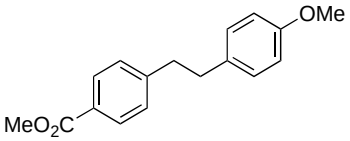
The title compound was obtained from methyl 4-chlorobenzoate (171 mg, 1.0 mmol) and α-methylstyrene (414 mg, 3.5 mmol). Silica gel column chromatography (hexane/AcOEt = 80:1) gave **3af** (200.9 mg, 0.790 mmol, 79%) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.89 (d, J = 8.0 Hz, 2H), 7.29-7.25 (m, 2H), 7.20-7.11 (m, 5H), 3.89 (s, 3H), 3.05-2.94 (m, 2H), 2.85 (dd, J = 12.8, 7.5 Hz, 1H), 1.26 (d, J = 6.6 Hz, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 166.9, 146.1, 146.0, 129.3, 129.0, 128.2, 127.7, 126.8, 126.0, 51.7, 44.8, 41.5, 21.1; IR (ATR): 3028, 2855, 1717, 1609, 1433, 1275, 1179, 1105 cm^{-1}; HRMS (ESI) *m/z*: $[M+Na]^+$ calcd for $C_{17}H_{18}NaO_2$ 277.1205, found 277.1207.

Methyl 4-(2-phenylethyl)benzoate (3ag)



The title compound was obtained from methyl 4-chlorobenzoate (171 mg, 1.0 mmol) and styrene (521 mg, 5.0 mmol) using MeCN (3.0 mL). Silica gel column chromatography (hexane/AcOEt = 80:1) gave **3ag** (183.6 mg, 0.764 mmol, 76%) as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.96-7.93 (m, 2H), 7.29-7.14 (m, 7H), 3.90 (s, 3H), 3.01-2.91 (m, 4H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 167.0, 147.0, 141.0, 129.6, 128.4, 128.3, 128.3, 127.8, 126.0, 51.8, 37.7, 37.3.
The ^{1}H and ^{13}C{^{1}H} NMR spectra are in accordance with those reported in the literature.[12]

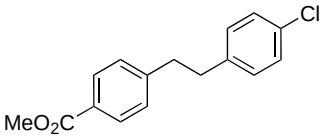
Methyl 4-(2-(4-methoxyphenyl)ethyl)benzoate (3ah)



The title compound was obtained from methyl 4-chlorobenzoate (171 mg, 1.0 mmol) and 4-methoxystyrene (470 mg, 3.5 mmol) using MeCN (3.0 mL). Silica gel column chromatography (hexane/AcOEt = 20:1) gave **3ah** (165.2 mg, 0.611 mmol, 61%) as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.95-7.92 (m, 2H), 7.21 (d, J = 8.2 Hz, 2H), 7.07-7.04 (m, 2H), 6.83-6.79 (m, 2H), 3.90 (s, 3H), 3.79 (s, 3H), 2.96-2.85 (m, 4H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 167.1, 157.8, 147.2, 133.1, 129.6, 129.3, 128.5, 127.8, 113.7, 55.1, 51.9, 38.1, 36.5.

The ^{1}H and ^{13}C{^{1}H} NMR spectra are in accordance with those reported in the literature.[12]

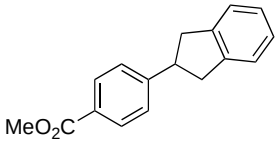
Methyl 4-(2-(4-chlorophenyl)ethyl)benzoate (3ai)



The title compound was obtained from methyl 4-chlorobenzoate (171 mg, 1.0 mmol) and 4-chlorostyrene (485 mg, 3.5 mmol) using MeCN (3.0 mL). Silica gel column chromatography (hexane/AcOEt = 80:1) gave **3ai** (234.7 mg, 0.854 mmol, 85%) as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.96-7.93 (m, 2H), 7.24-7.18 (m, 4H), 7.07-7.04 (m, 2H), 3.91 (s, 3H), 2.97-2.88 (m, 4H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 167.0, 146.6, 139.4, 131.7, 129.8, 129.6, 128.5, 128.4, 128.0, 52.0, 37.6, 36.7.

The ^{1}H and ^{13}C{^{1}H} NMR spectra are in accordance with those reported in the literature.[13]

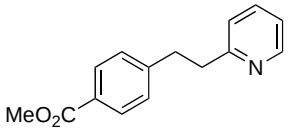
Methyl 4-(2,3-dihydro-1H-inden-2-yl)benzoate (3aj)



The title compound was obtained from methyl 4-chlorobenzoate (171 mg, 1.0 mmol) and 1*H*-indene (581 mg, 5.0 mmol) using MeCN (3.0 mL). Silica gel column chromatography (hexane/AcOEt = 80:1) gave **3aj** (140.2 mg, 0.556 mmol, 56%) as a yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.99-7.96 (m, 2H), 7.38-7.35 (m, 2H), 7.26-7.23 (m, 2H), 7.22-7.17 (m, 2H), 3.91 (s, 3H), 3.79-3.70 (m, 1H), 3.38 (dd, J = 15.3, 8.2 Hz, 2H), 3.09 (dd, J = 15.4, 8.6 Hz, 2H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 166.9, 150.8, 142.4, 129.7, 128.0, 126.9, 126.5, 124.2, 51.8, 45.2, 40.5.

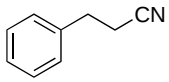
The ^{1}H and ^{13}C{^{1}H} NMR spectra are in accordance with those reported in the literature.[14]

Methyl 4-(2-(pyridin-2-yl)ethyl)benzoate (3ak)



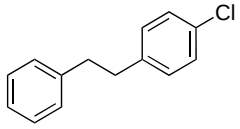
The title compound was obtained from methyl 4-chlorobenzoate (171 mg, 1.0 mmol) and 2-vinylpyridine (526 mg, 5.0 mmol) using MeCN (3.0 mL). Silica gel column chromatography (hexane/AcOEt = 5:1) gave **3ak** (199.3 mg, 0.826 mmol, 83%) as a white solid. mp 70–71 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.56 (ddd, J = 4.9, 1.8, 0.9 Hz, 1H), 7.95-7.92 (m, 2H), 7.56 (td, J = 7.7, 1.8 Hz, 1H), 7.26-7.24 (m, 2H), 7.12 (ddd, J = 7.6, 4.9, 1.1 Hz, 1H), 7.04 (dt, J = 7.8, 1.0 Hz, 1H), 3.90 (s, 3H), 3.16-3.07 (m, 4H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 167.0, 160.5, 149.3, 147.0, 136.3, 129.6, 128.4, 127.8, 122.9, 121.2, 51.9, 39.6, 35.8.; IR (ATR): 2943, 1707, 1589, 1439, 1281, 1107 cm^{-1}; HRMS (ESI) *m/z*: $[M+H]^+$ calcd for $C_{15}H_{16}NO_2$ 242.1181, found 242.1178.
The ^{1}H and ^{13}C{^{1}H} NMR spectra are in accordance with those reported in the literature.[15]

3-Phenylpropanenitrile (3kd)



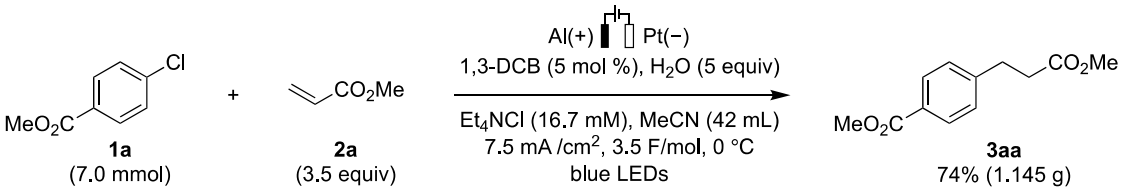
The title compound was obtained from iodobenzene (204 mg, 1.0 mmol) and acrylonitrile (265 mg, 5.0 mmol). Silica gel column chromatography (hexane/AcOEt = 8:1) gave **3kd** (111.8 mg, 0.852 mmol, 85%) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.37-7.33 (m, 2H), 7.30-7.23 (m, 3H), 2.97 (t, J = 7.4 Hz, 2H), 2.63 (t, J = 7.4 Hz, 2H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 137.9, 128.7, 128.1, 127.0, 119.0, 31.3, 19.1.
The ^{1}H and ^{13}C{^{1}H} NMR spectra are in accordance with those reported in the literature.[16]

1-Chloro-4-(2-phenylethyl)benzene (3ki)



The title compound was obtained from iodobenzene (204 mg, 1.0 mmol) and 4-chlorostyrene (693 mg, 5.0 mmol). Silica gel column chromatography (hexane 100%) gave **3ki** (158.6 mg, 0.732 mmol, 73%) as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.30-7.14 (m, 7H), 7.10-7.06 (m, 2H), 2.89 (s, 4H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 141.2, 140.1, 131.6, 129.8, 128.42, 128.35, 128.3, 126.0, 37.7, 37.2.
The ^{1}H and ^{13}C{^{1}H} NMR spectra are in accordance with those reported in the literature.[17]

2.3. Gram-scale experiment



A cylinder-type undivided cell with a stir bar was charged with Et_4NCl (116 mg, 0.7 mmol), 1,3-dicyanobenzene (1,3-DCB, 44.8 mg, 0.35 mmol), **2a** (2.11 g, 24.5 mmol), and **1a** (1.19 g, 7.0 mmol). Anhydrous MeCN (42 mL) and H_2O (0.63 mL) were successively added. The reaction vessel was equipped with seven Pt plate cathodes (immersed surface area: 1 × 2 cm^2 each) and seven Al plate anodes (immersed surface area: 1 × 2 cm^2 each) under air (Figure S2), then 3.5 F/mol of electricity was passed through at a constant current condition (7.5 mA/cm^2) under visible-light irradiation (approximate distance from the cathodes: 2.5 cm) at 0 °C in an ice bath (Figure S3). Most of solvent was removed under reduced pressure, and the residue was diluted with AcOEt. The organic layer was washed with H_2O, dried over Na_2SO_4, filtered, and concentrated under reduced pressure. The residue was purified by flash silica gel column chromatography (hexane/AcOEt = 8/1, then benzene 100%) to afford **3aa** (1.145 g, 5.15 mmol, 74 % yield).

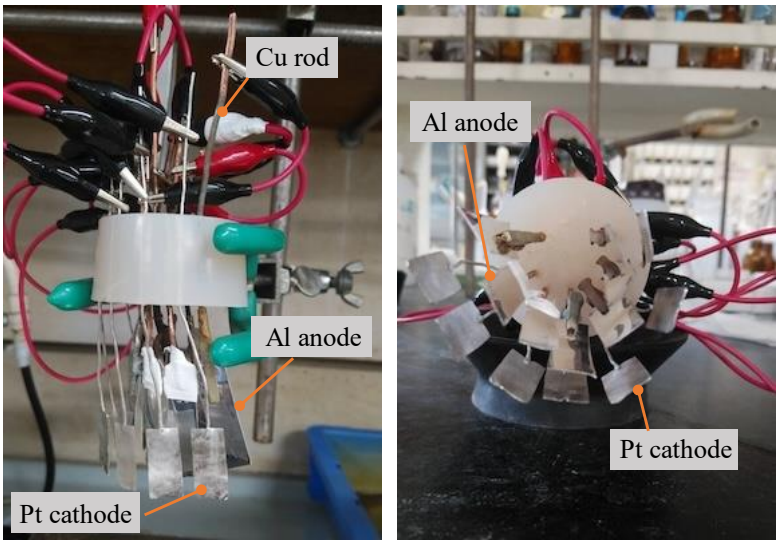


Figure S2. Side view (left) and bottom view (right) of a silicone stopper equipped with Al and Pt electrodes used for the gram-scale reaction.

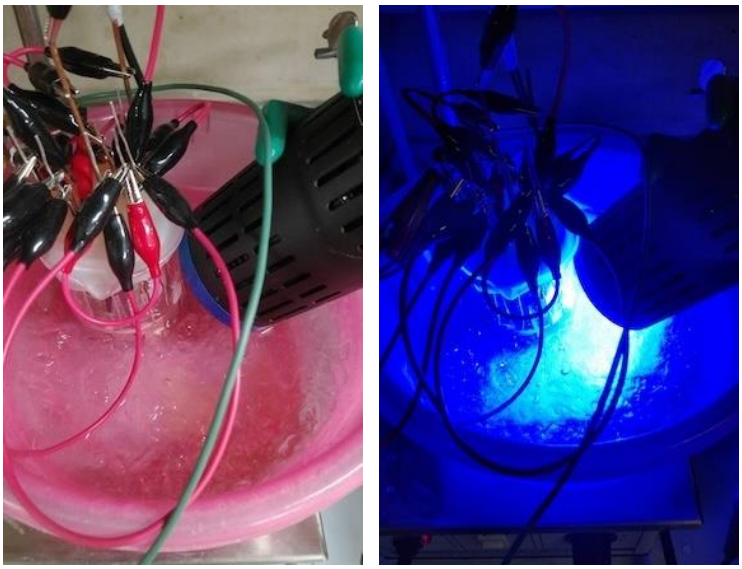
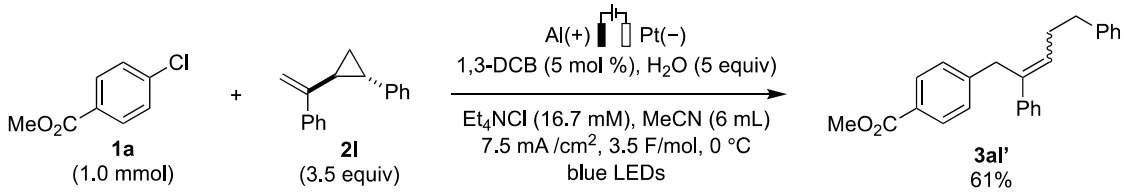


Figure S3. A reaction setup for the gram-scale reaction.

2.4. Radical-clock experiment

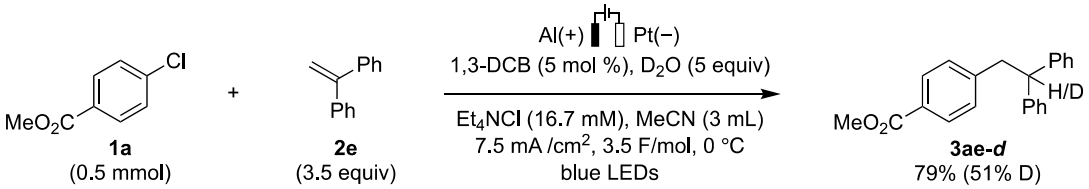


A cylinder-type undivided cell with a stir bar was charged with Et_4NCl (16.6 mg, 0.1 mmol), 1,3-dicyanobenzene (1,3-DCB, 6.4 mg, 0.05 mmol), **2l** (771 mg, 3.5 mmol), and **1a** (170 mg, 1.0 mmol). Anhydrous MeCN (6.0 mL) and H_2O (90 μL) were successively added. The reaction vessel was equipped with a Pt plate cathode (immersed surface area: 1 × 2 cm^2) and an Al plate anode (immersed surface area: 1 × 2 cm^2) under air, then 3.5 F/mol of electricity was passed through at a constant current condition (7.5 mA/cm^2) under visible-light irradiation (approximate distance from the cathode: 2.5 cm) at 0 °C in an ice bath. Most of solvent was removed under reduced pressure, and the residue was diluted with AcOEt. The organic layer was washed with H_2O, dried over Na_2SO_4, filtered, and concentrated under reduced pressure. The residue was purified by flash silica gel column chromatography (hexane/AcOEt = 40:1) gave **3al'** (216.6 mg, 0.608 mmol, 61%).

Methyl 4-(2,5-diphenylpent-2-en-1-yl)benzoate (3al')

Colorless oil; ^1H NMR (400 MHz, $CDCl_3$): δ 7.86 (d, J = 8.0 Hz, 2H), 7.31-7.12 (m, 12H), 6.02 (t, J = 7.3 Hz, 1H), 3.87 (s, 3H), 3.84 (s, 2H), 2.78 (t, J = 7.5 Hz, 2H), 2.56 (td, J = 7.5, 7.3 Hz, 2H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 166.9, 145.2, 142.3, 141.4, 137.3, 130.2, 129.6, 128.4, 128.3, 128.12, 128.06, 127.7, 126.7, 126.1, 125.9, 51.8, 35.69, 35.67, 30.9; IR (ATR): 3024, 2947, 1717, 1609, 1495, 1275, 1177, 1105 cm^{-1}; HRMS (ESI) *m/z*: $[M+Na]^+$ calcd for $C_{25}H_{24}NaO_2$ 379.1674, found 379.1672.

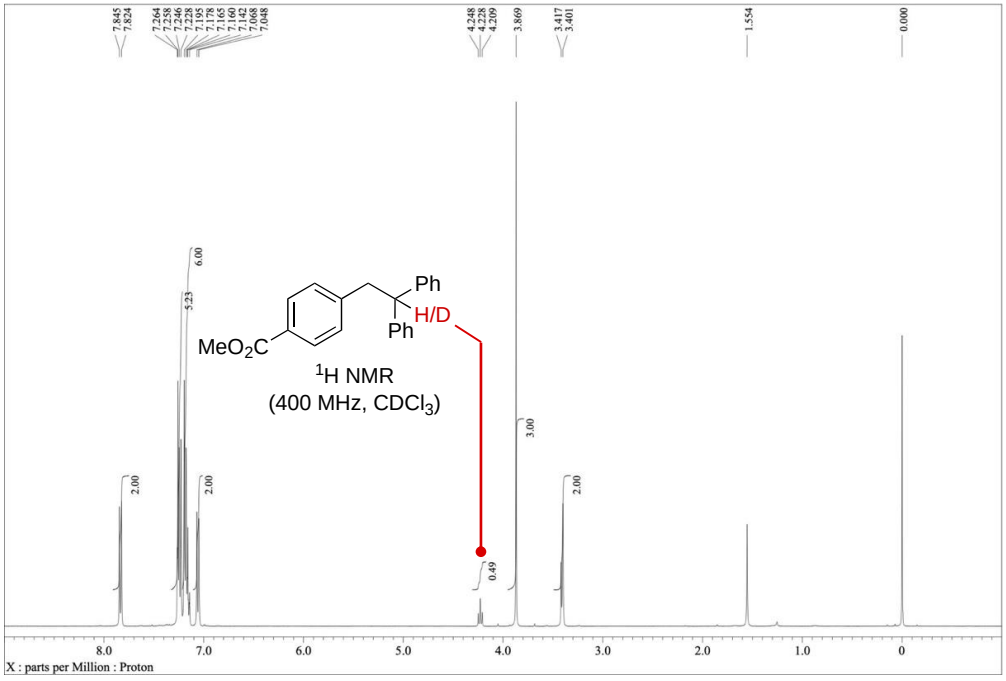
2.5. Deuterium-labeling experiment



A cylinder-type undivided cell with a stir bar was charged with Et_4NCl (8.3 mg, 0.05 mmol), 1,3-dicyanobenzene (1,3-DCB, 3.2 mg, 0.025 mmol), **2e** (315 mg, 1.75 mmol), and **1a** (85 mg, 0.5 mmol). Anhydrous MeCN (3.0 mL) and D_2O (45 μL) were successively added. The reaction vessel was equipped with a Pt plate cathode (immersed surface area: 1 × 2 cm^2) and an Al plate anode (immersed surface area: 1 × 2 cm^2) under air, then 3.5 F/mol of electricity was passed through at a constant current condition (7.5 mA/cm^2) under visible-light irradiation (approximate distance from the cathode: 2.5 cm) at 0 °C in an ice bath. Most of solvent was removed under reduced pressure, and the residue was diluted with AcOEt. The organic layer was washed with H_2O, dried over Na_2SO_4, filtered, and concentrated under reduced pressure. The residue was purified by flash silica gel column chromatography to afford the desired product **3ae-*d*** (124.9 mg, 0.394 mmol, 79% yield, 51% D).

Compound 3ae-d (51% D)

^1H NMR (400 MHz, $CDCl_3$): δ 7.83 (d, J = 8.2 Hz, 2H), 7.26-7.23 (m, 5H), 7.19-7.14 (m, 6H), 7.06 (d, J = 8.0 Hz, 2H), 4.23 (t, J = 7.9 Hz, 0.49H), 3.87 (s, 3H), 3.41 (d, J = 6.6 Hz, 2H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 167.0, 145.7, 143.9, 143.8, 129.4, 129.1, 128.4, 127.88, 127.87, 127.8, 126.3, 77.3, 77.0, 76.7, 52.8, 52.3 (t, J = 19.8 Hz), 51.9, 42.04, 41.96; IR (ATR): 3024, 2926, 1713, 1609, 1491, 1279, 1179, 1103 cm^{-1}; HRMS (ESI) *m/z*: $[M+Na]^+$ calcd for $C_{22}H_{19}{}^2HNaO_2$ 340.1424, found 340.1439.



3. References

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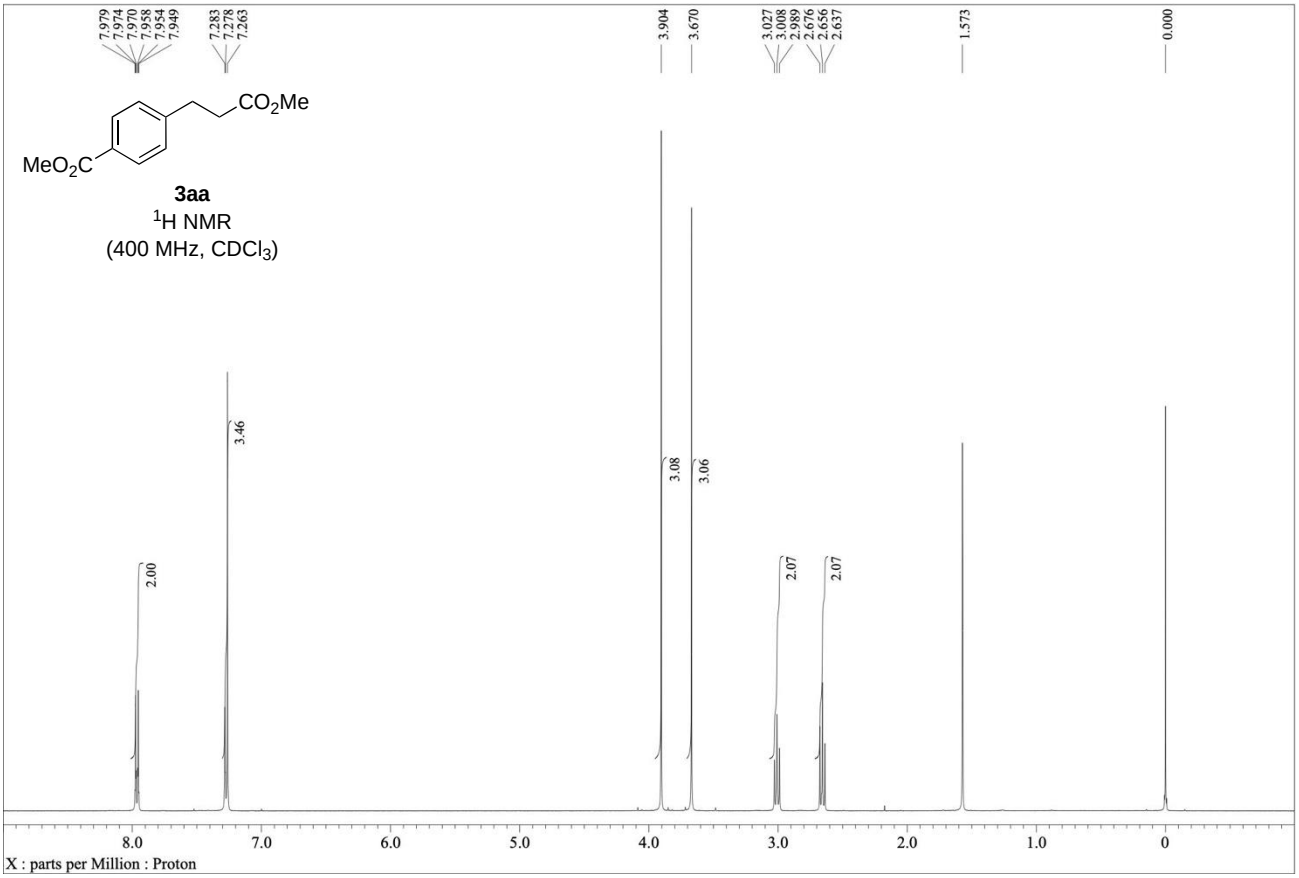
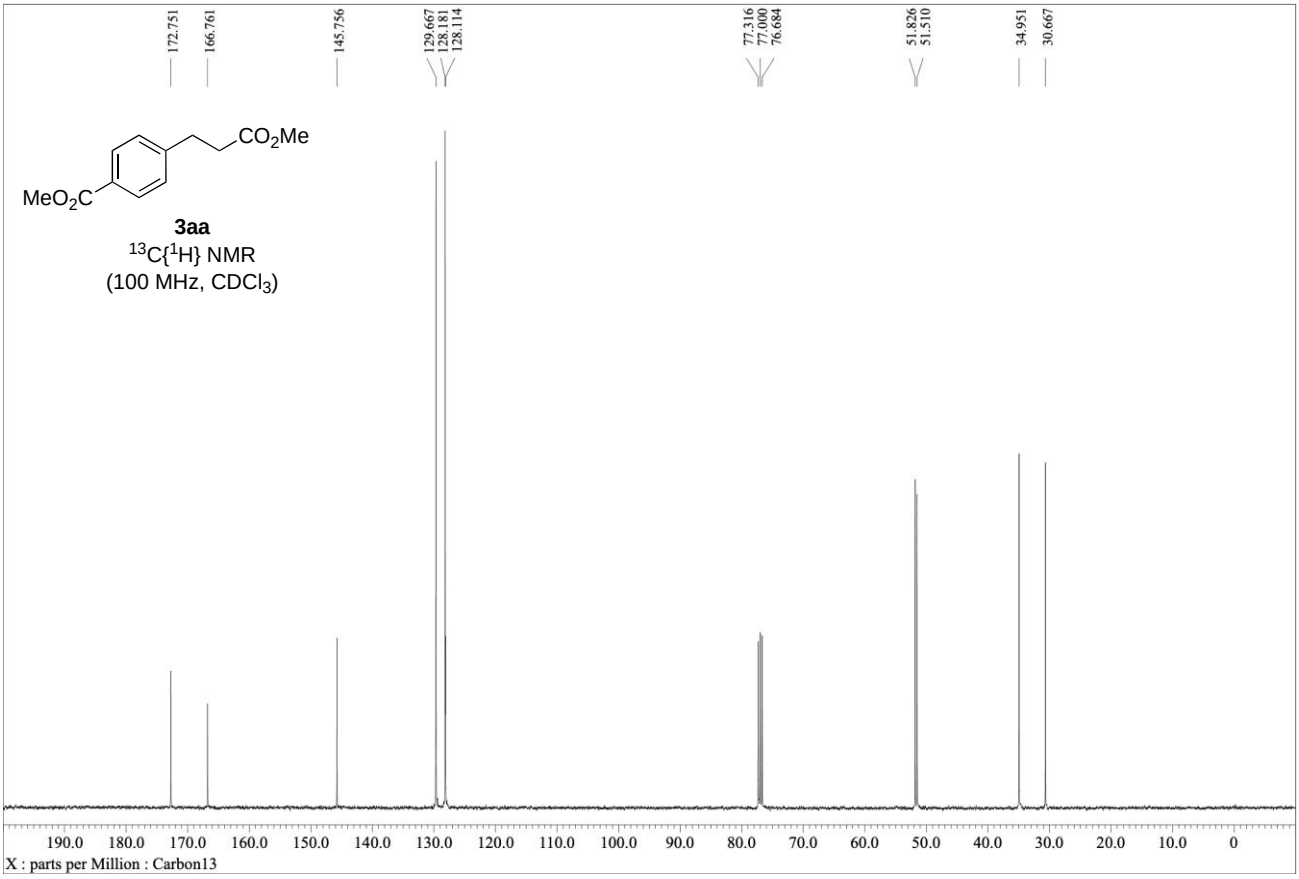
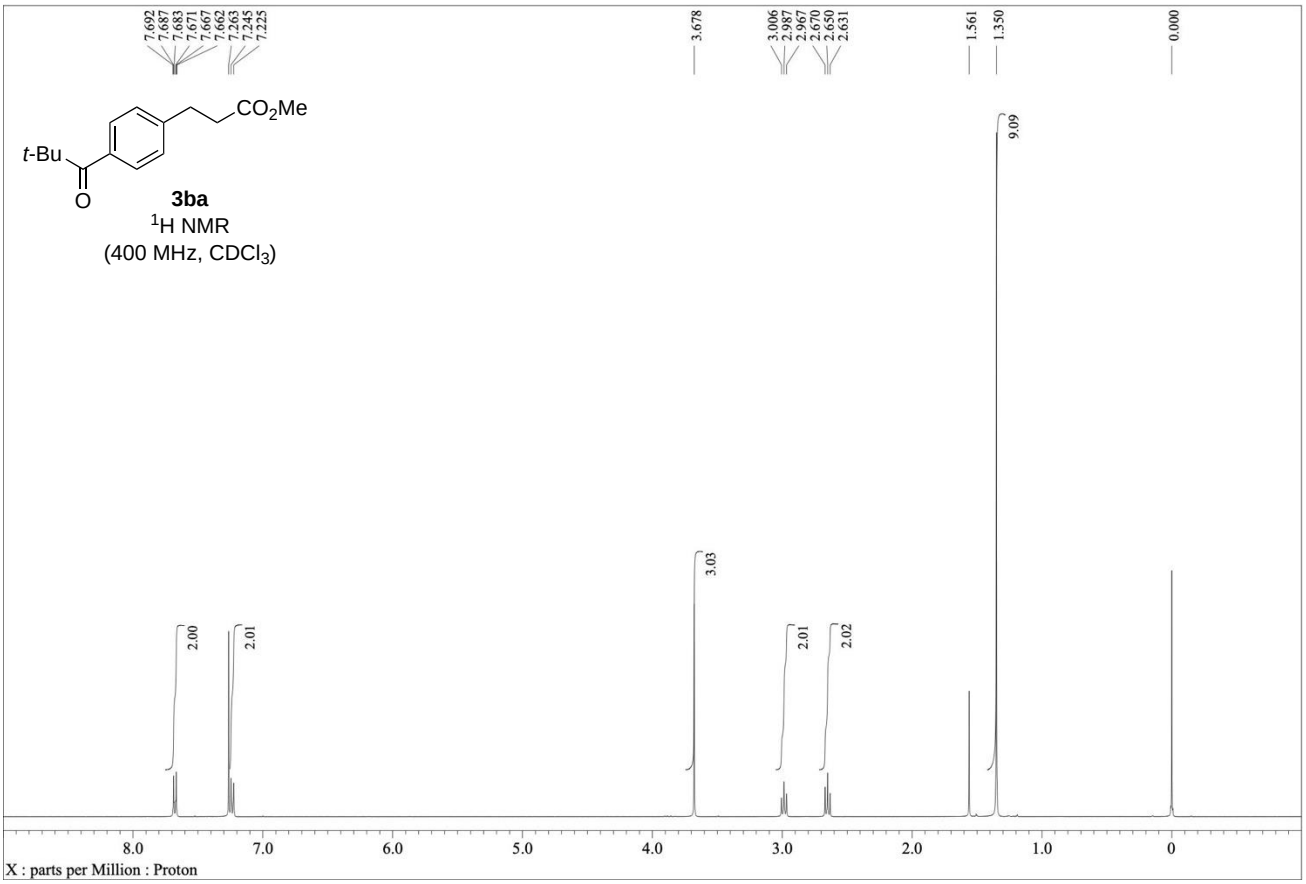
4. ^{1}H, $^{13}C\{^{1}H\}$, and ^{19}F NMR spectra



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7.662
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7.245
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3.006
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2.967
2.670
2.650
2.631
1.561
1.350
0.000
9.09
3.03
2.00
2.01
2.01
2.02
CO2Me
t-Bu
O
3ba
1H NMR
(400 MHz, CDCl3)
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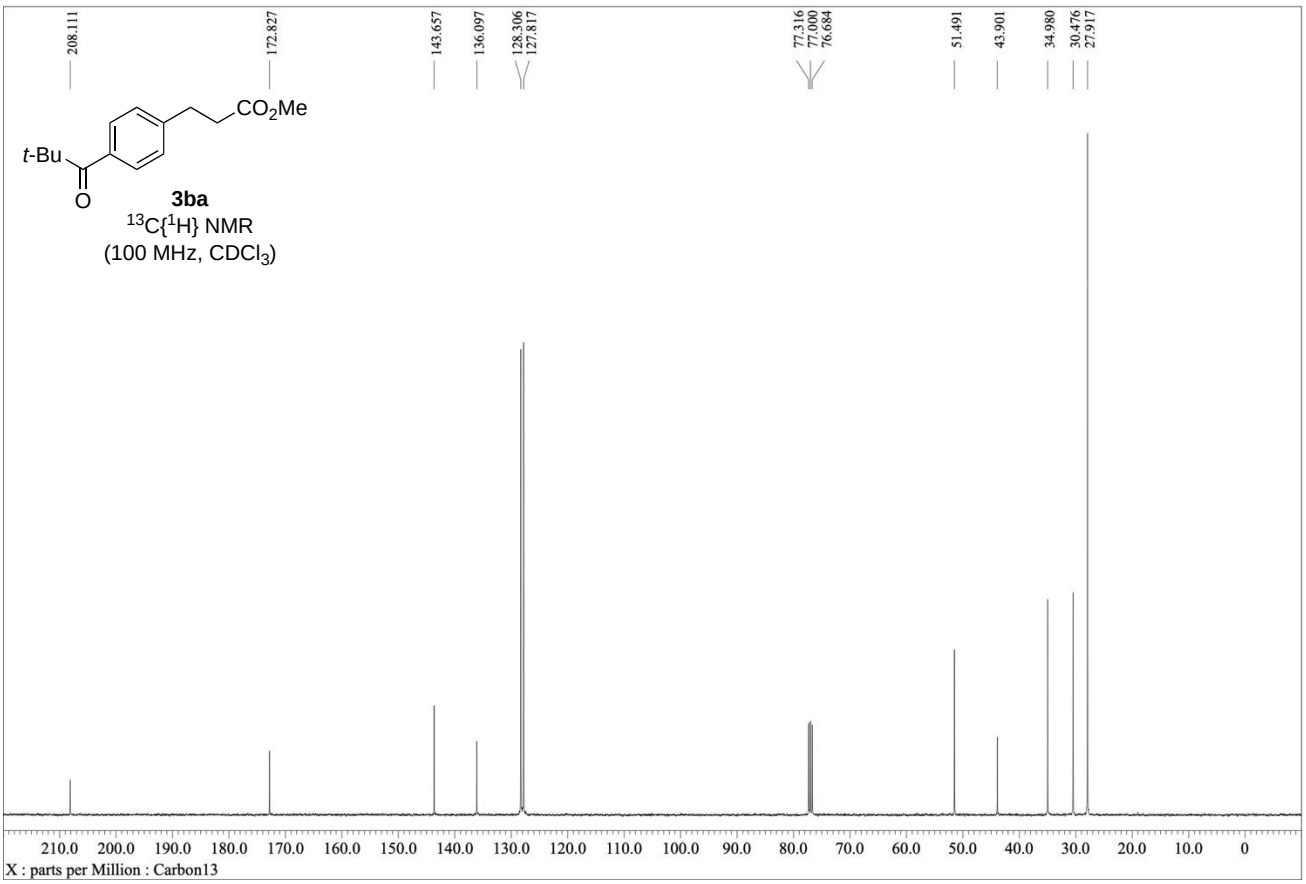
6.0
5.0
4.0
3.0
2.0
1.0
0
X : parts per Million : Proton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27.917
CO2Me
t-Bu
O
3ba
13C{1H} NMR
(100 MHz, CDCl3)
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200.0
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180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
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20.0
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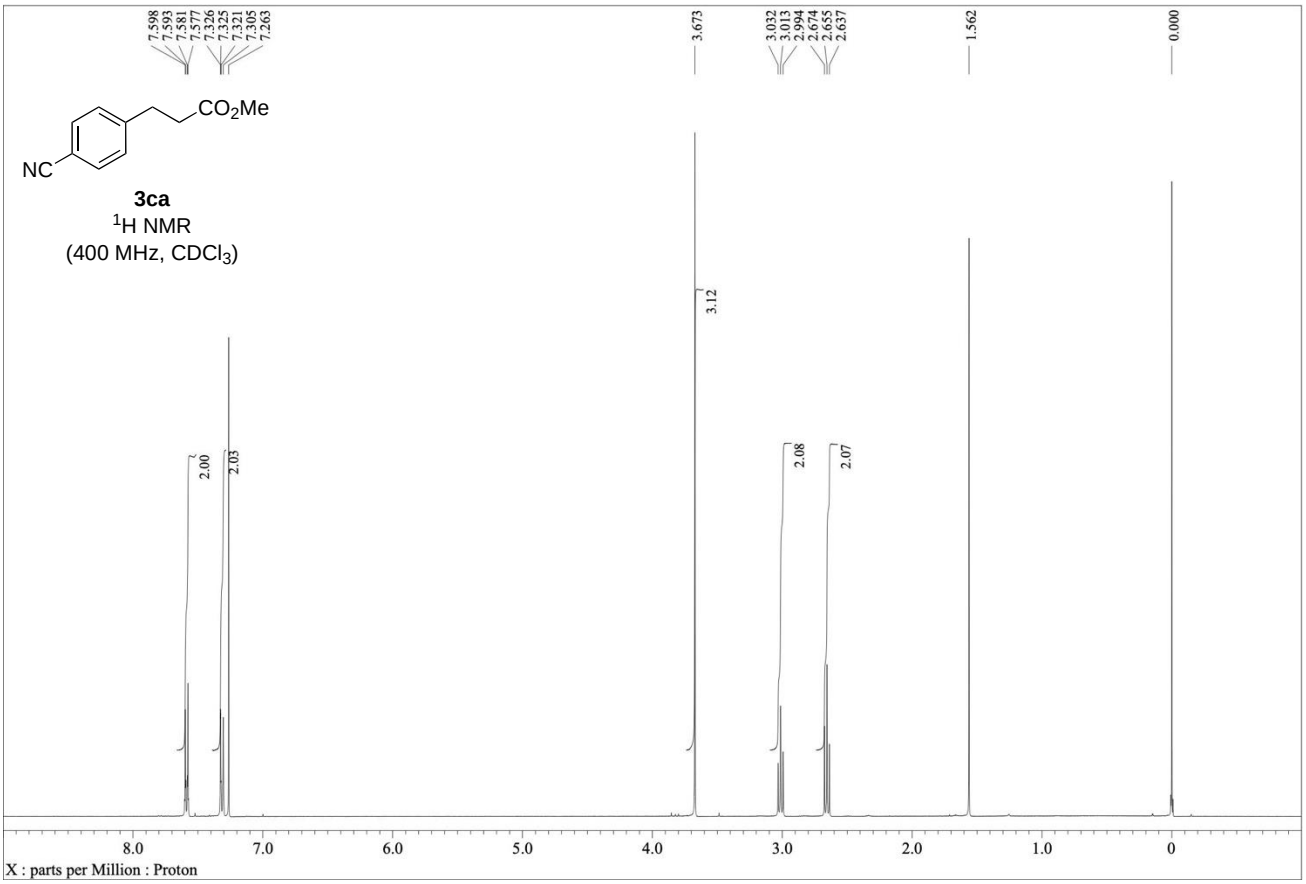
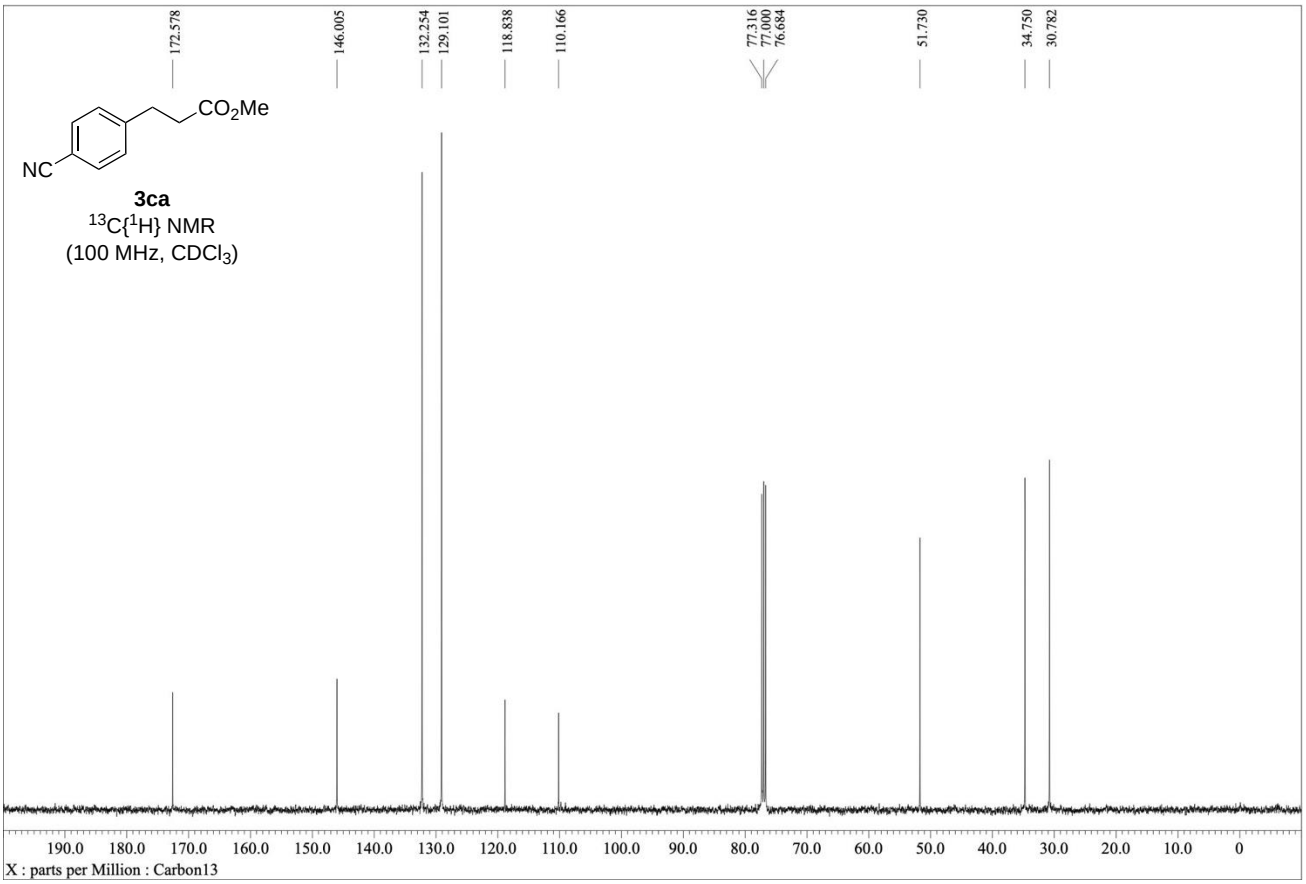
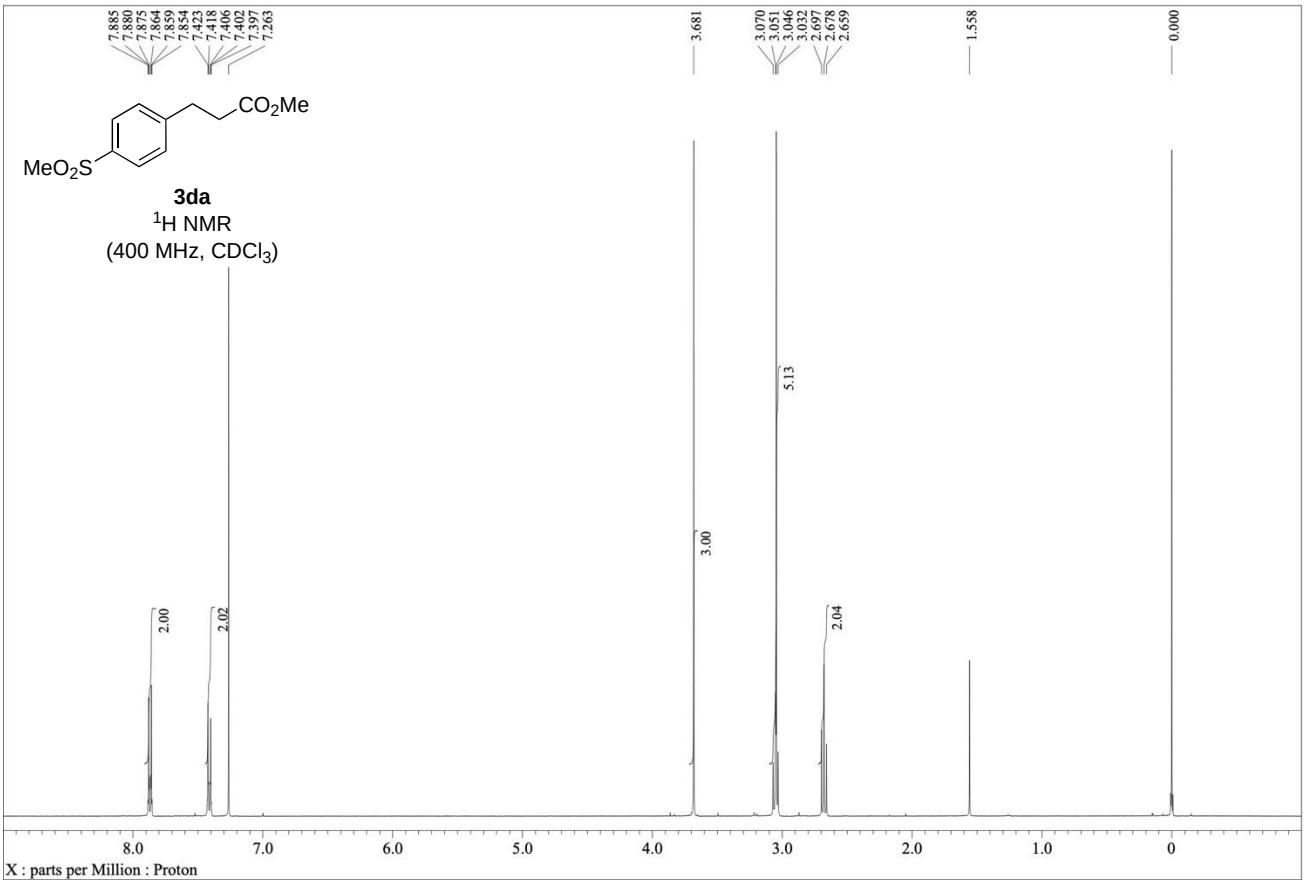


3ca
^{1}H NMR
(400 MHz, $CDCl_3$)



3ca
$^{13}C\{^{1}H\}$ NMR
(100 MHz, $CDCl_3$)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3.032
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MeO2S
3da
1H NMR
(400 MHz, CDCl3)
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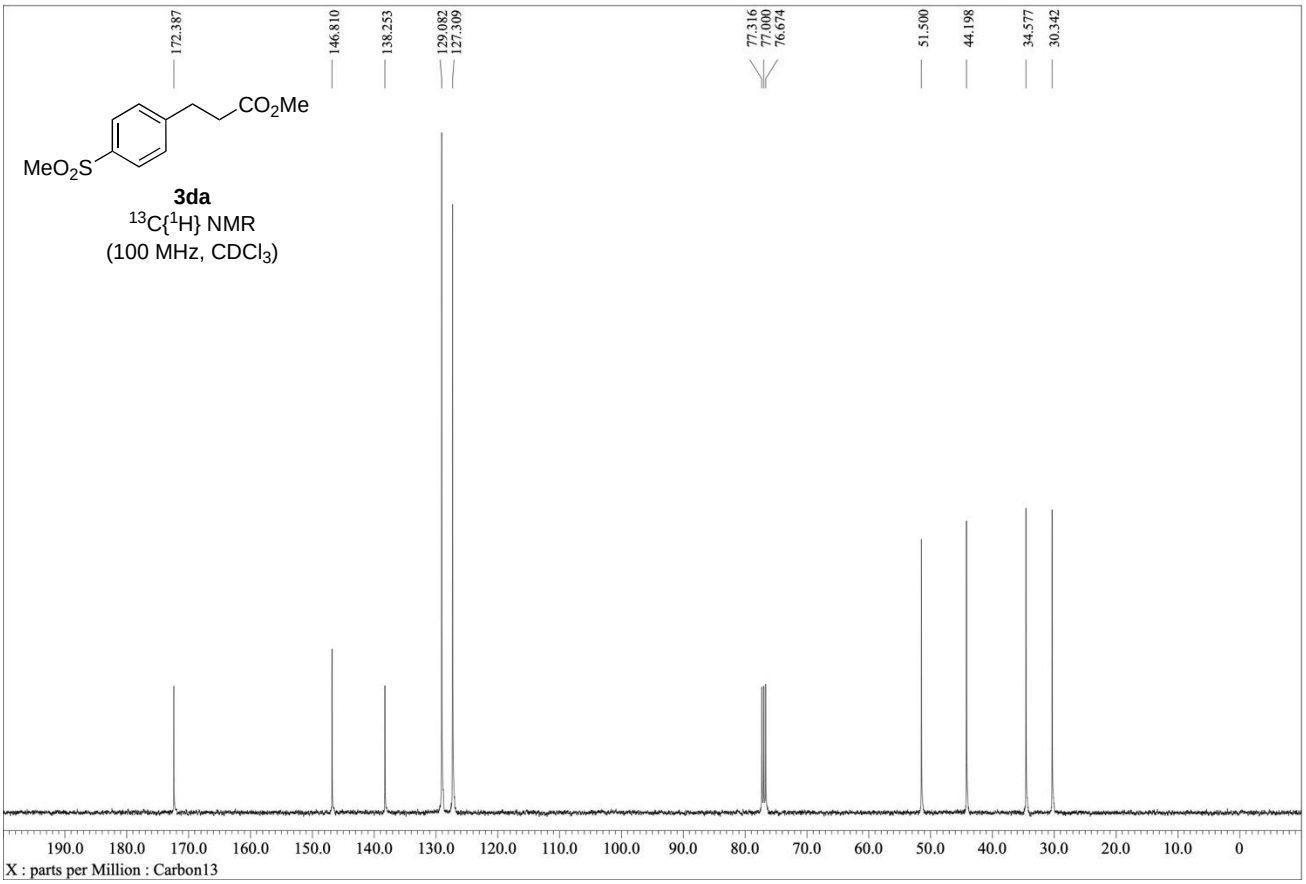
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3da
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(100 MHz, CDCl3)
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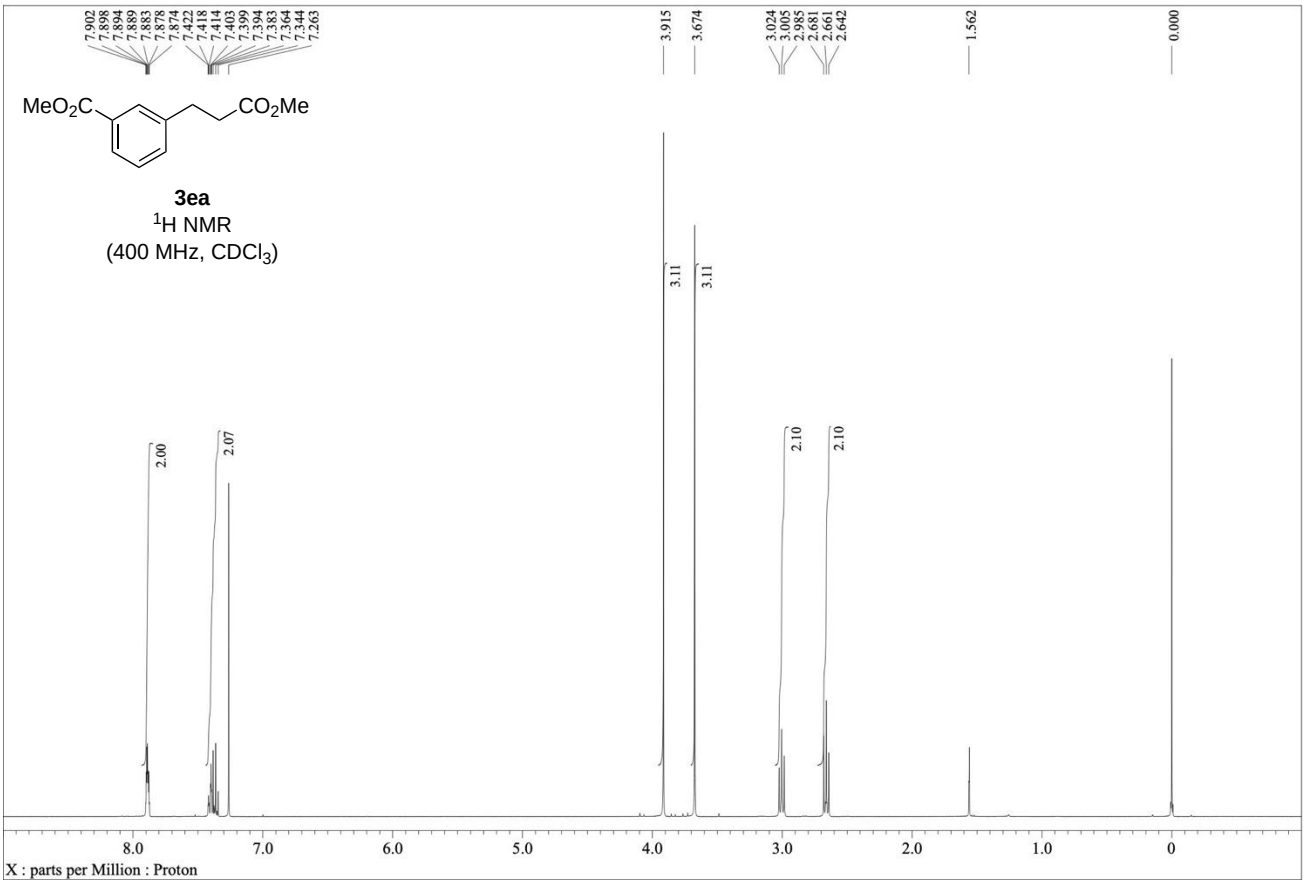
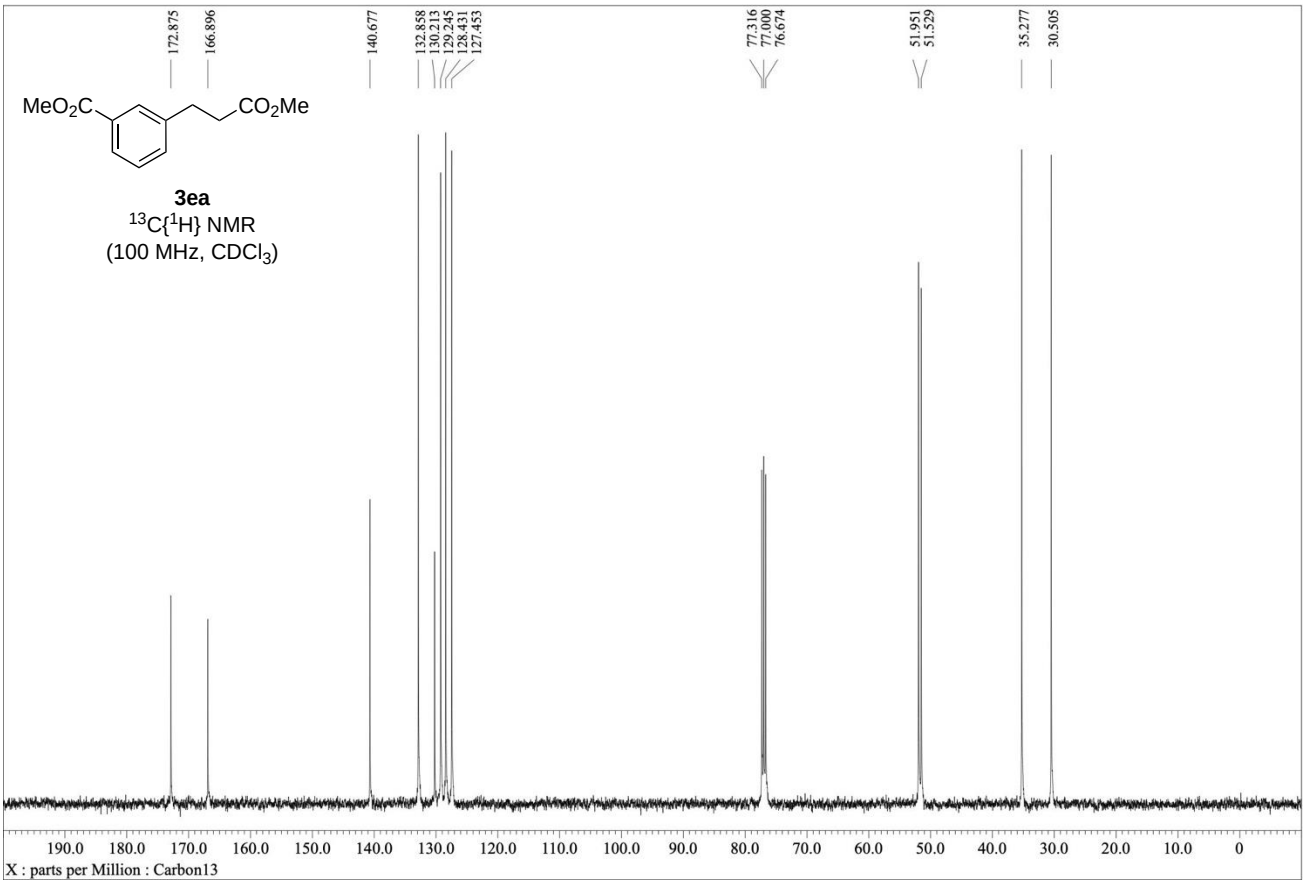
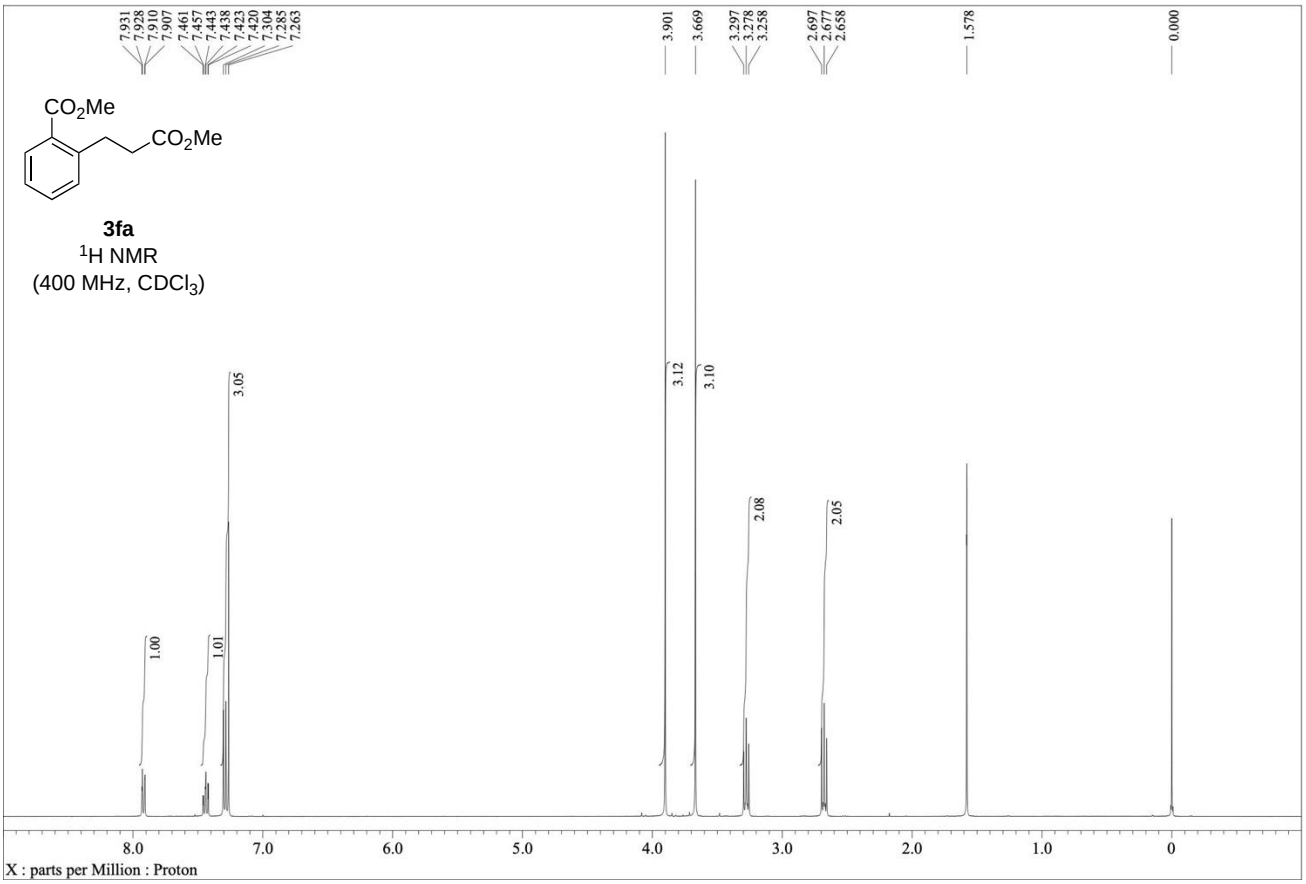


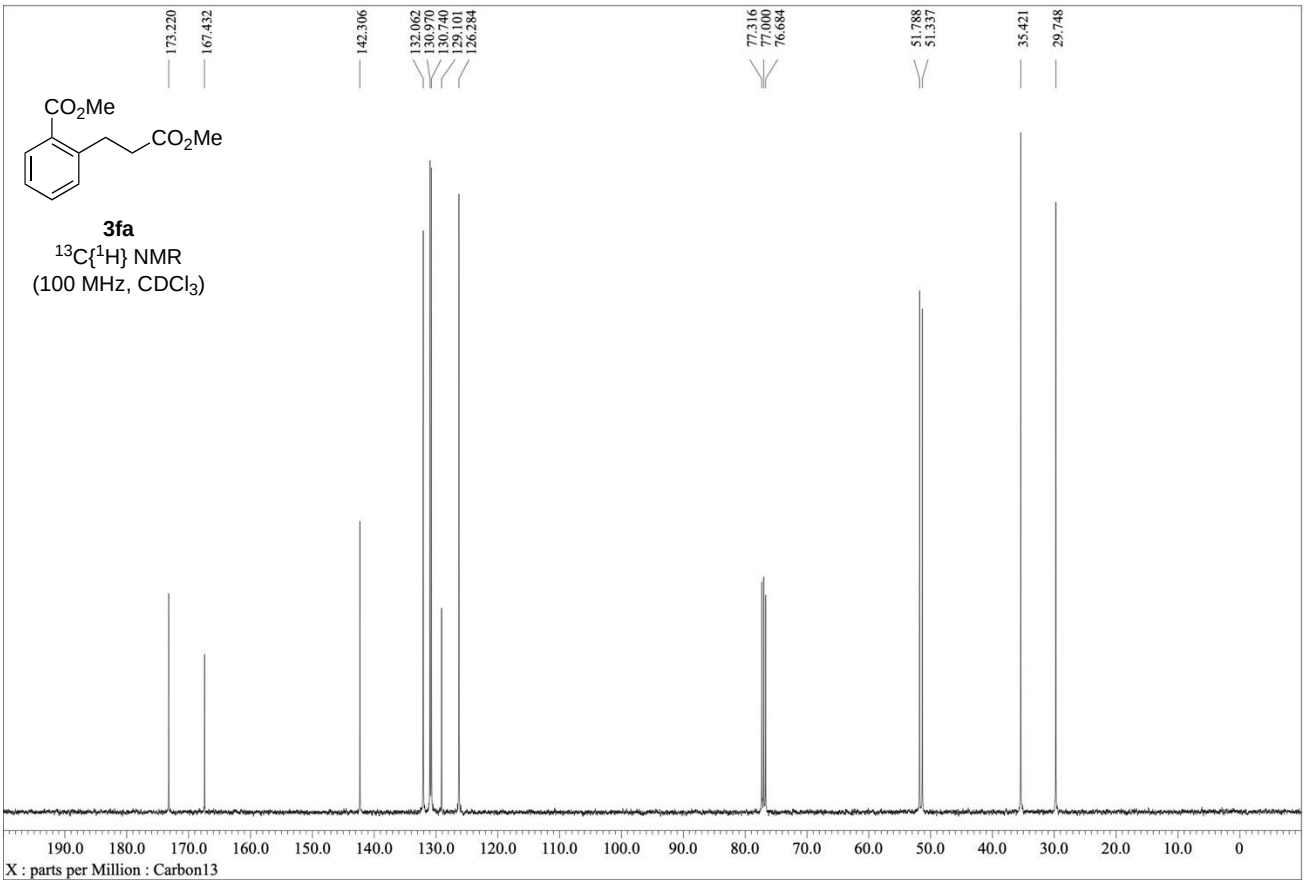
3ea
^{1}H NMR
(400 MHz, $CDCl_3$)



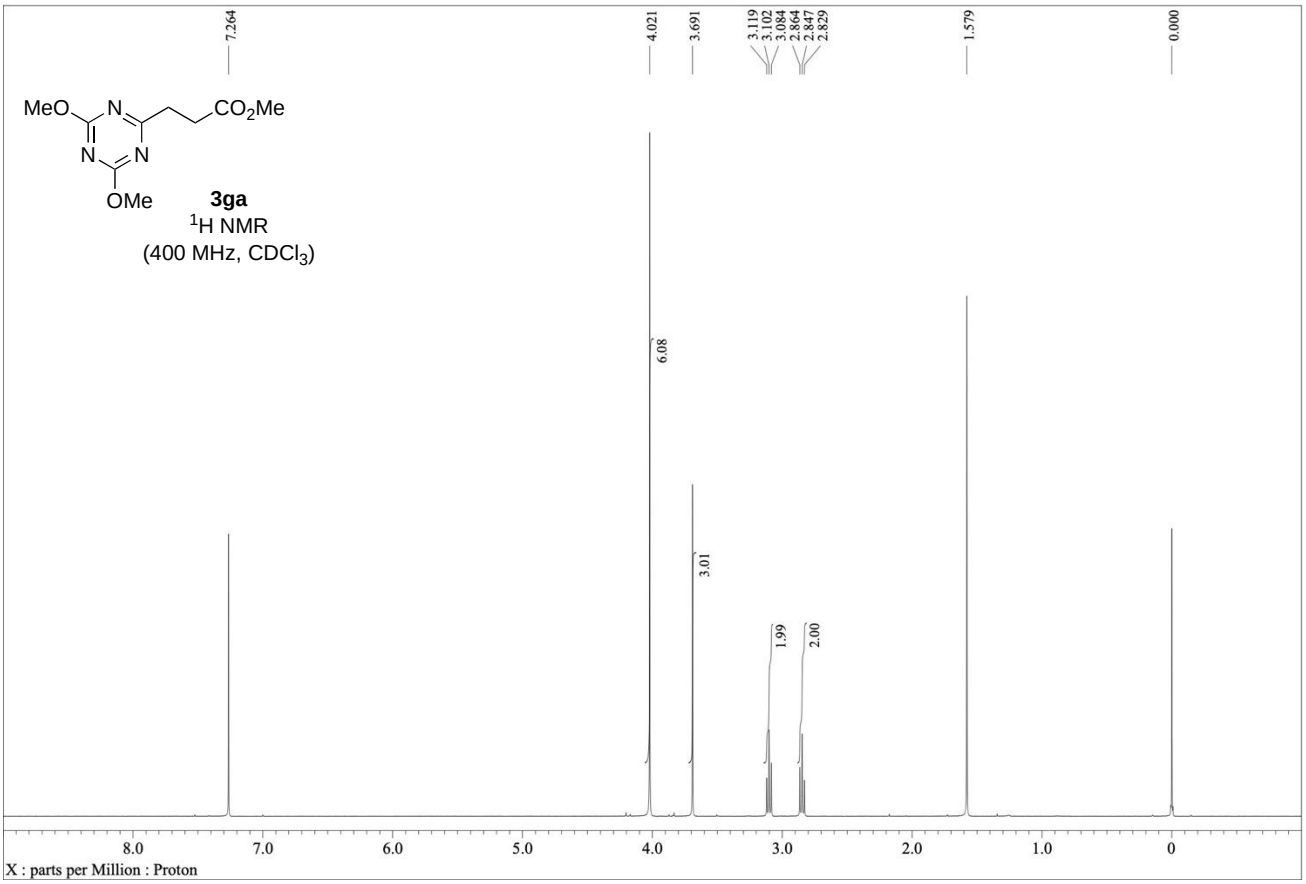
3ea
^{13}C{^{1}H} NMR
(100 MHz, $CDCl_3$)



3fa
^{1}H NMR
(400 MHz, $CDCl_3$)



3fa
^{13}C{^{1}H} NMR
(100 MHz, $CDCl_3$)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CO2Me
N
N
OMe
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1H NMR
(400 MHz, CDCl3)
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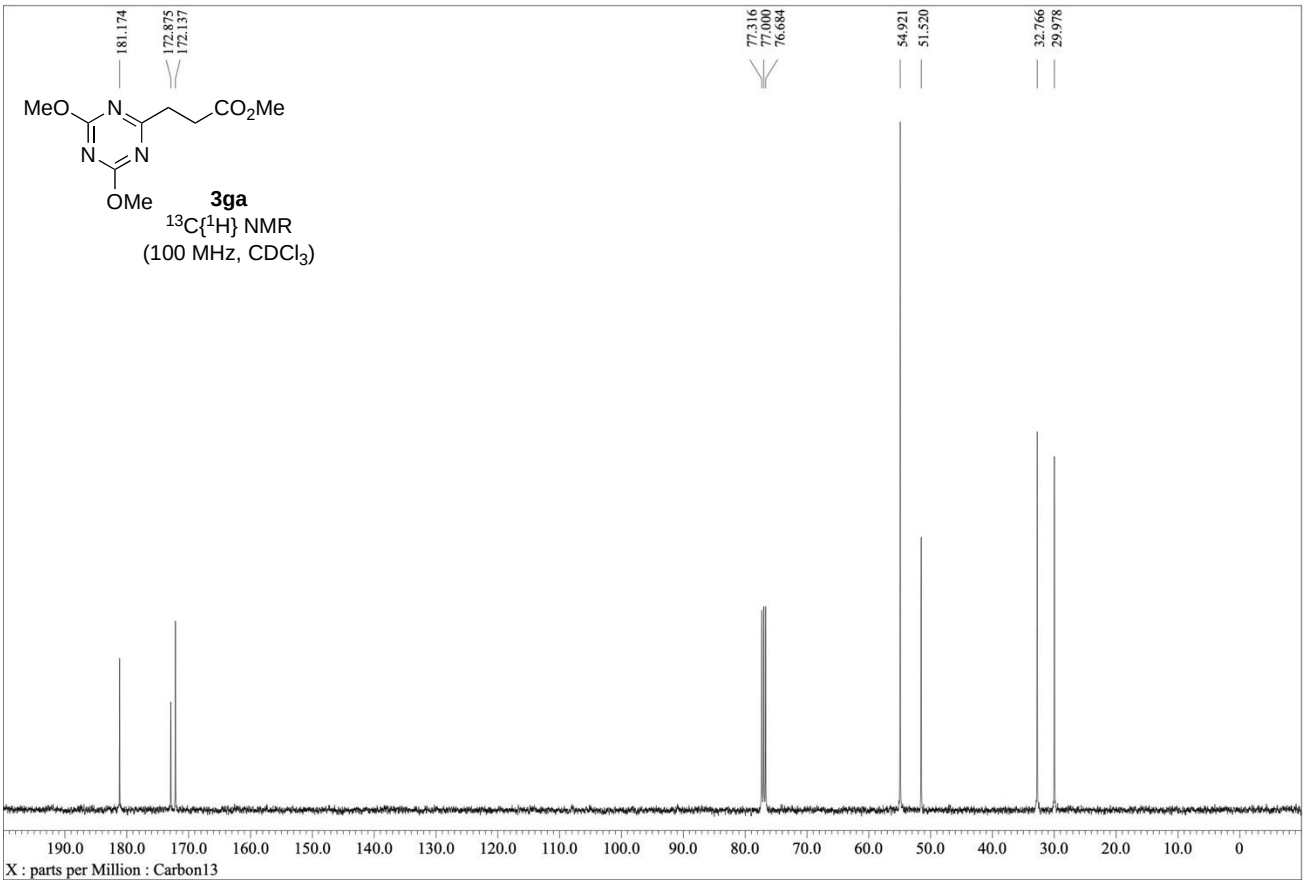
X : parts per Million : Proton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CO2Me
N
N
OMe
3ga
13C{1H} NMR
(100 MHz, CDCl3)
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180.0
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160.0
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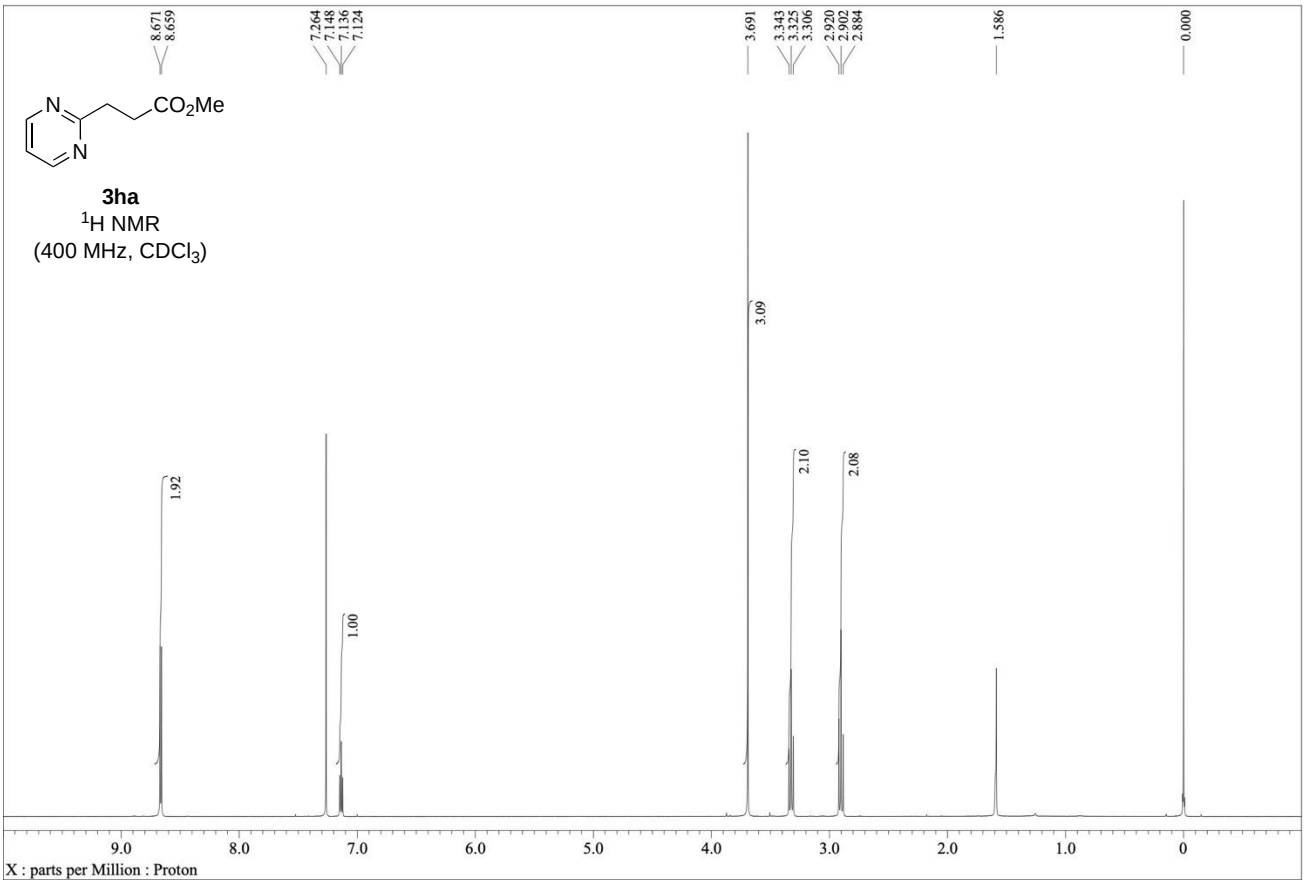
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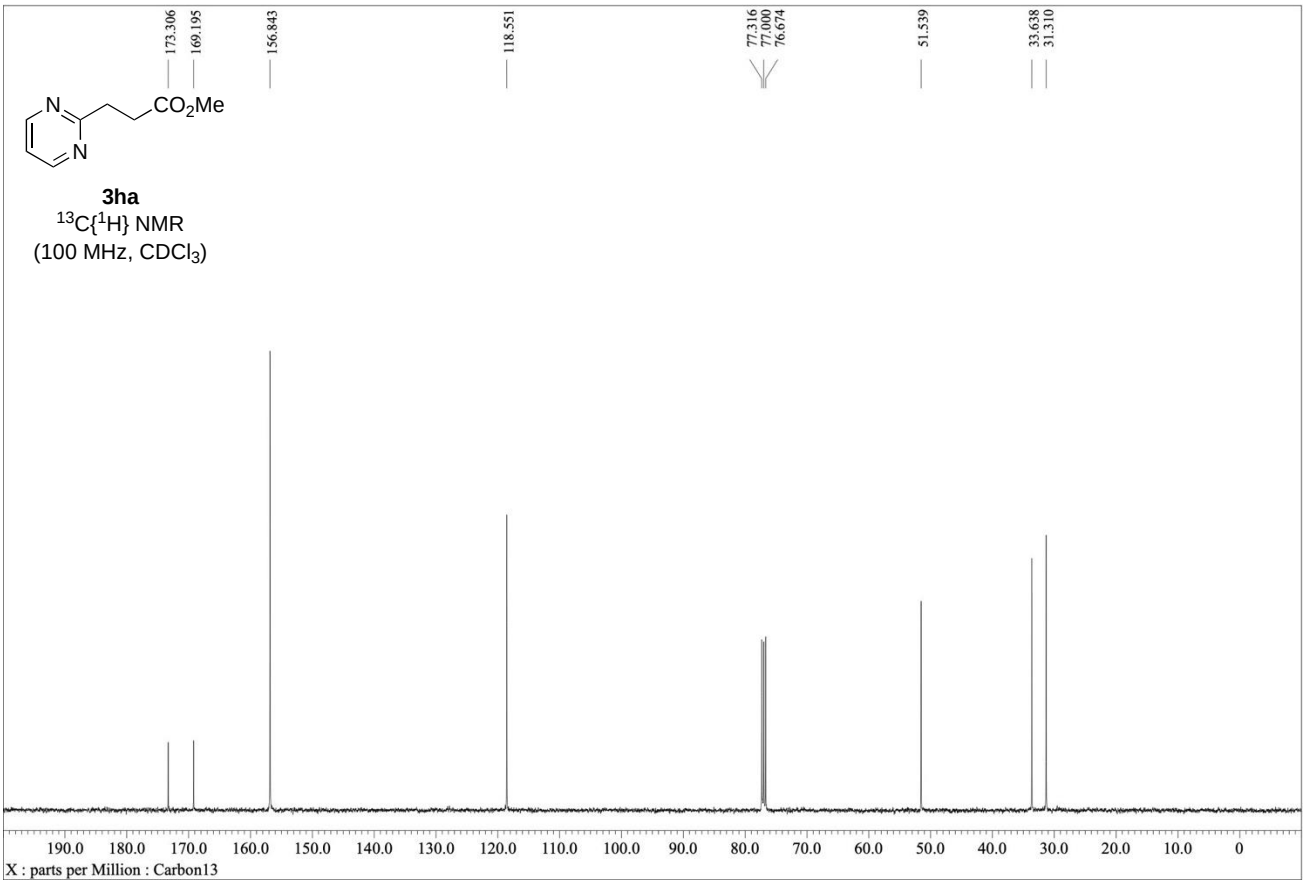
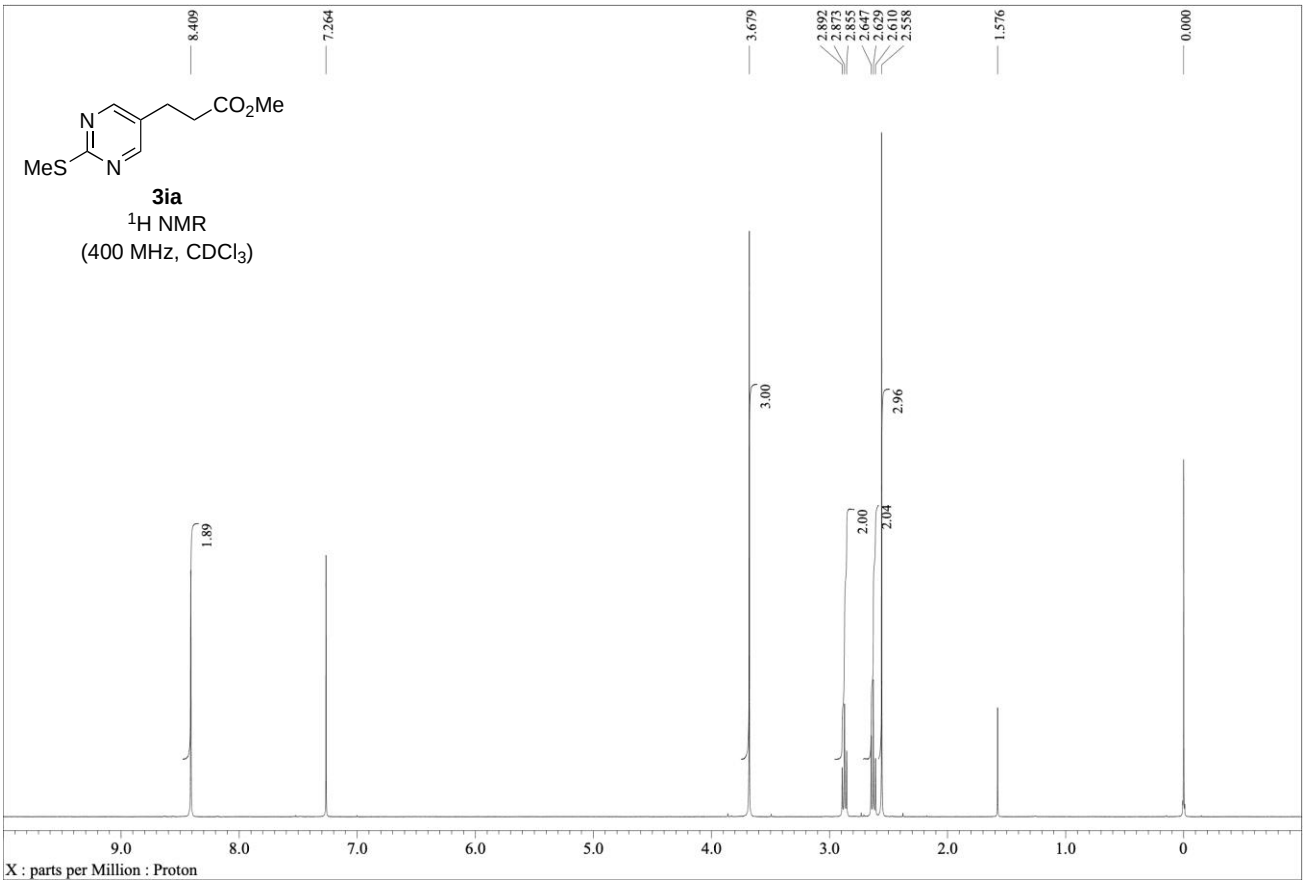
2.884
1.586
0.000
N
N
CO2Me
3ha
1H NMR
(400 MHz, CDCl3)
3.09
2.10
2.08
1.92
1.00
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
X : parts per Million : Proton

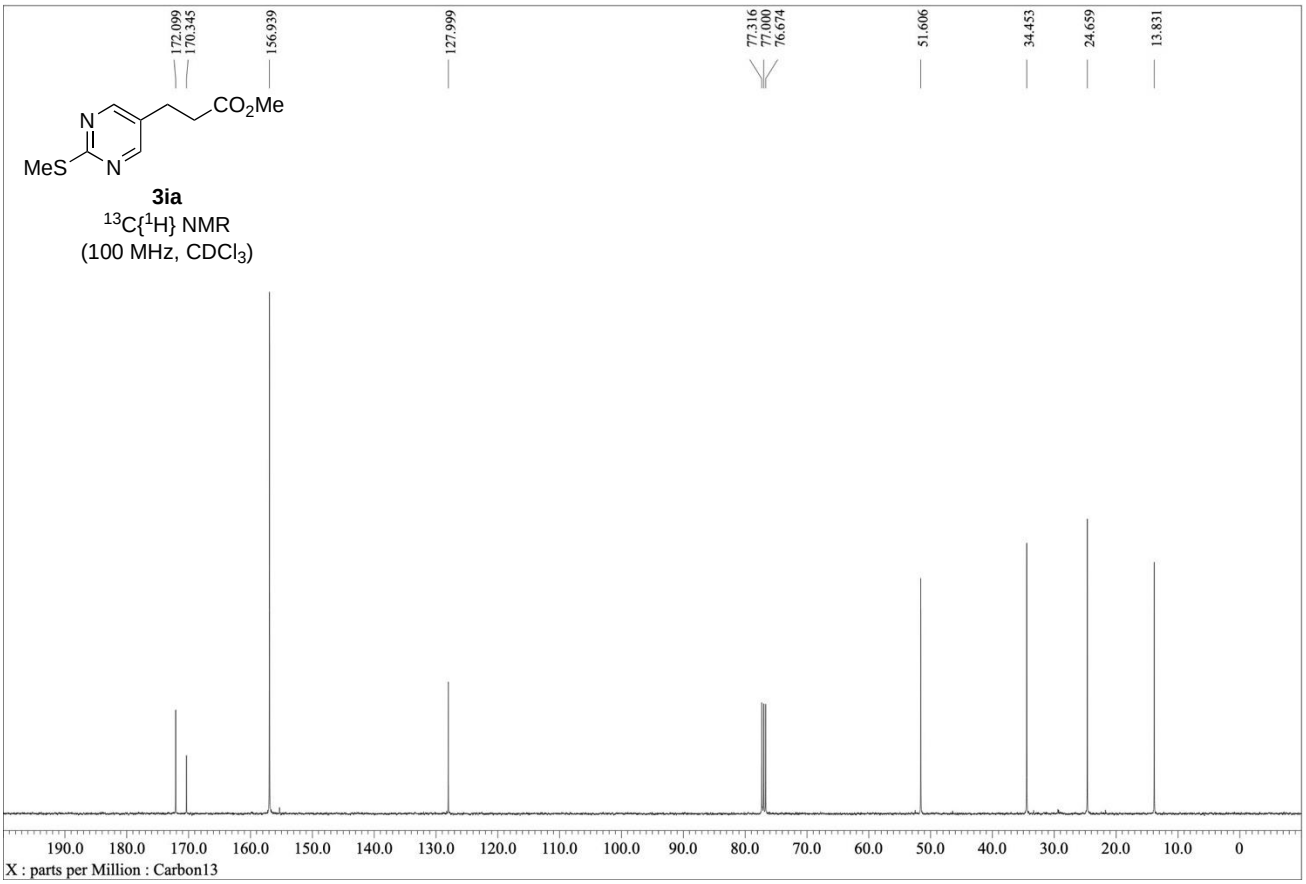

173.306
169.195
156.843
118.551
77.316
77.000
76.674
51.539
33.638
31.310
N
N
CO2Me
3ha
13C{1H} NMR
(100 MHz, CDCl3)
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
X : parts per Million : Carbon13



3ia

^{1}H NMR

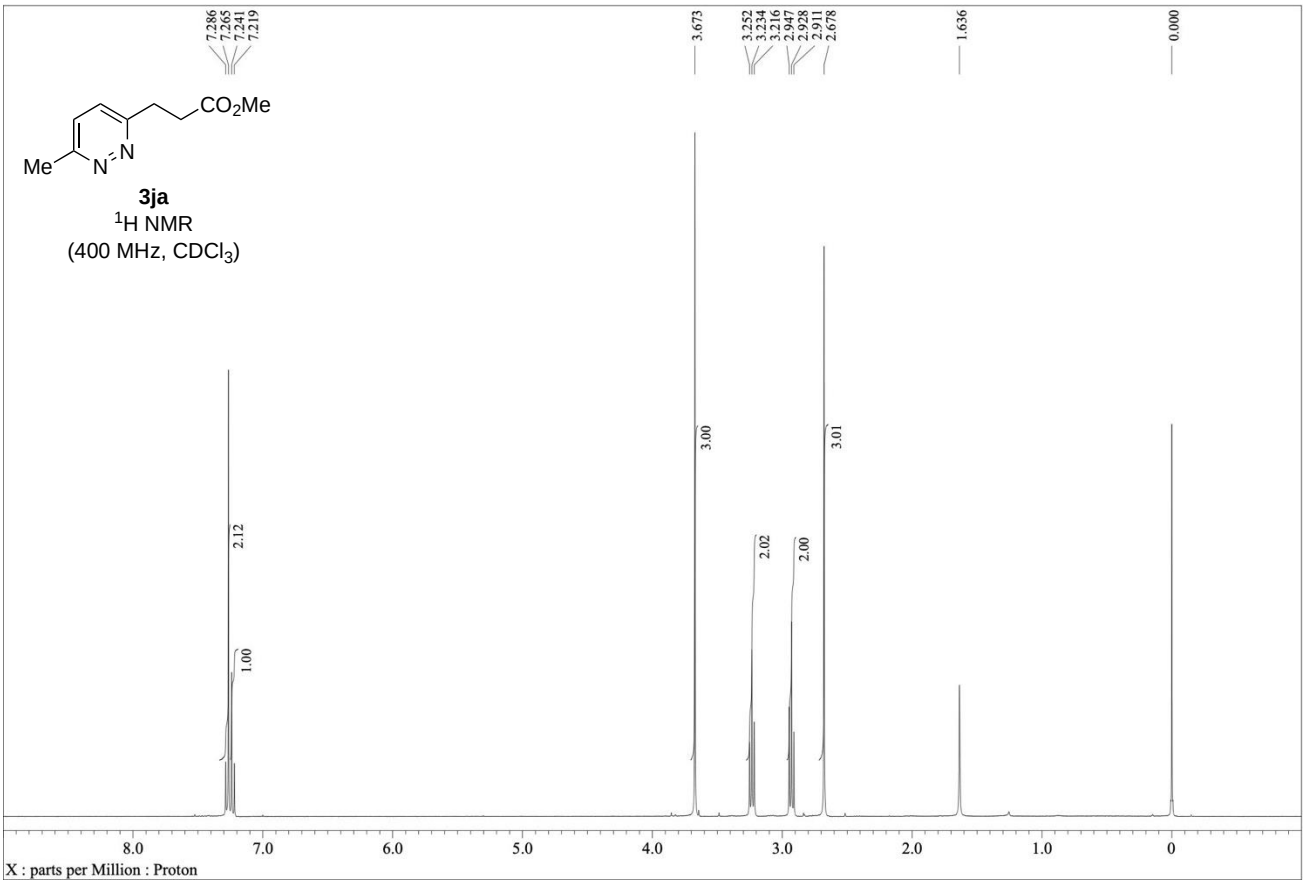
(400 MHz, $CDCl_3$)

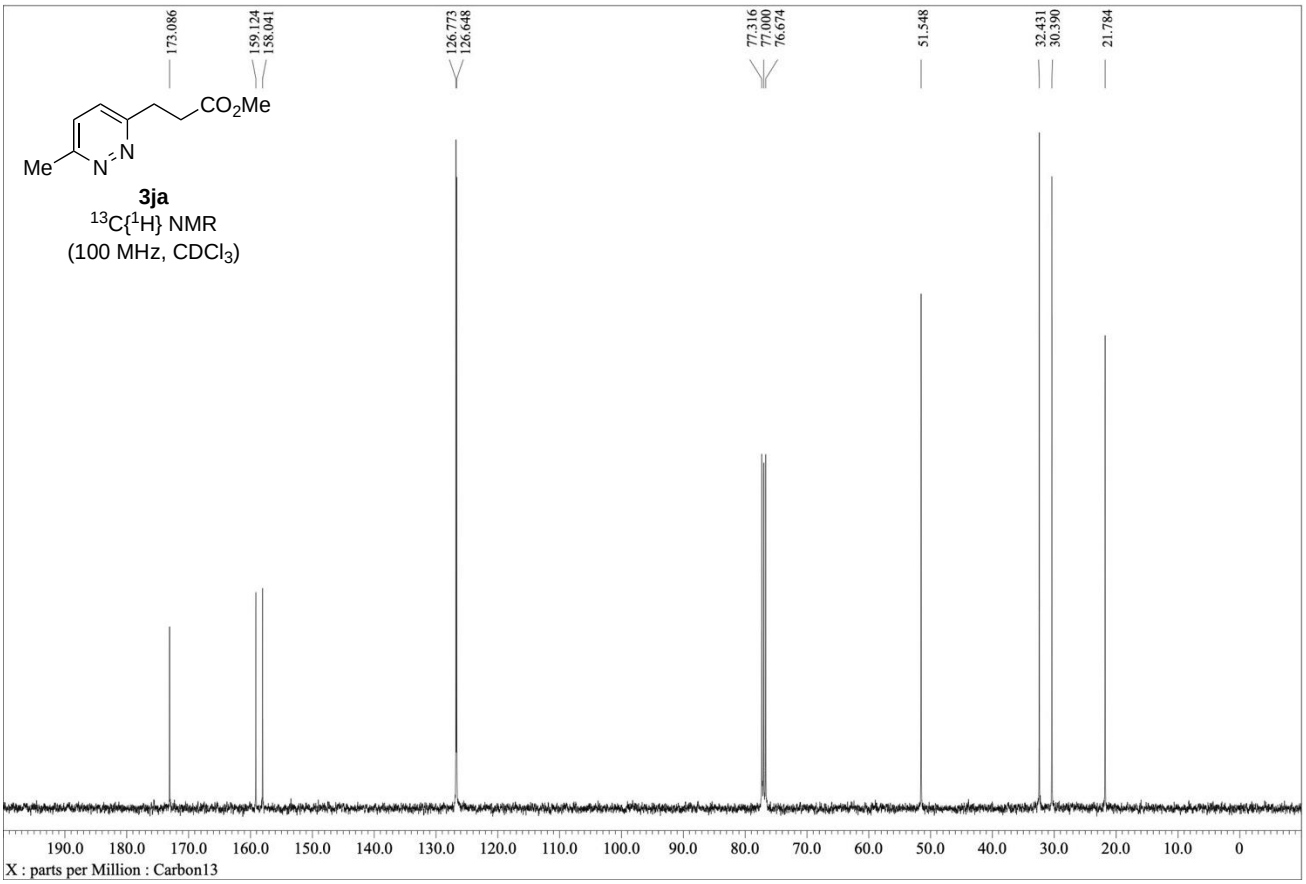


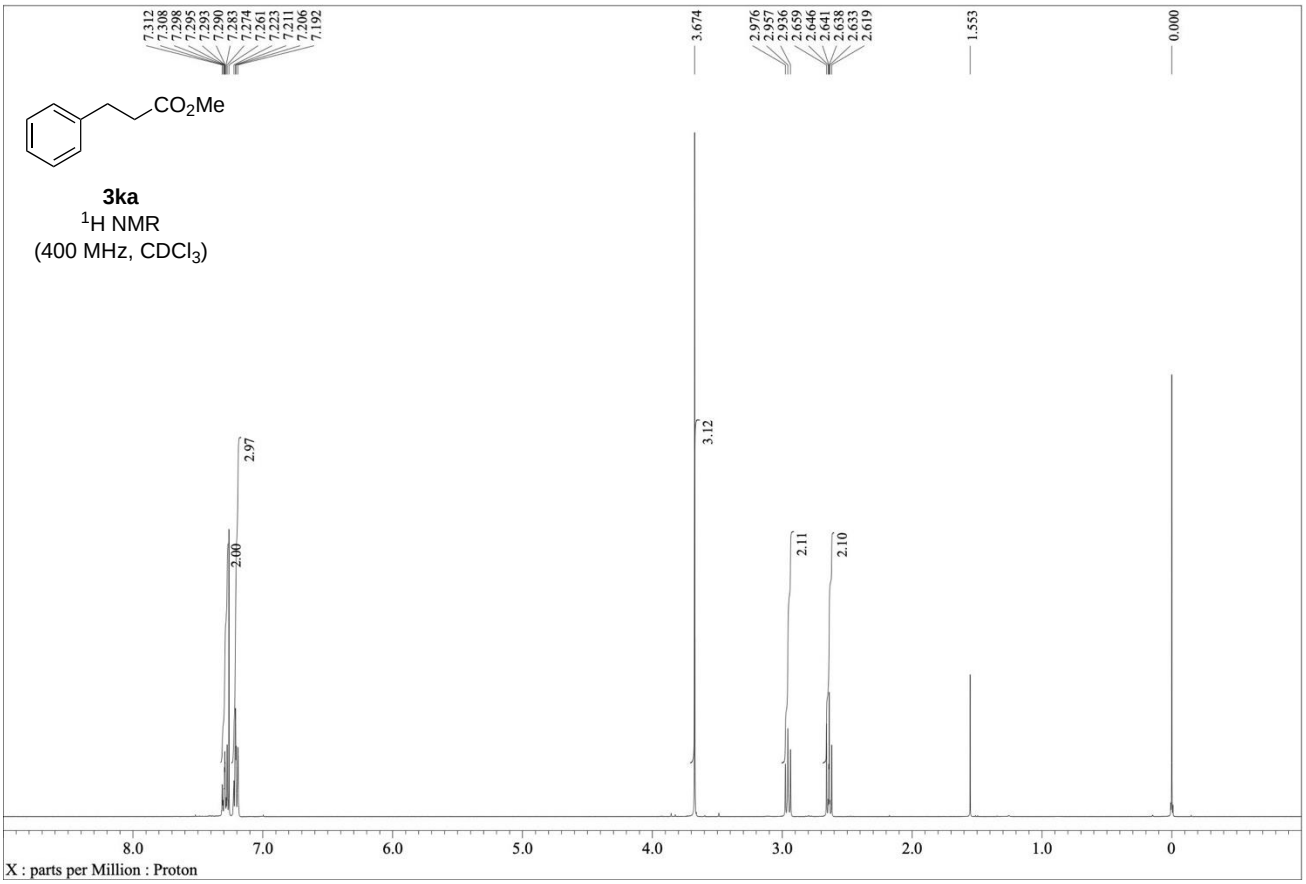
3ia

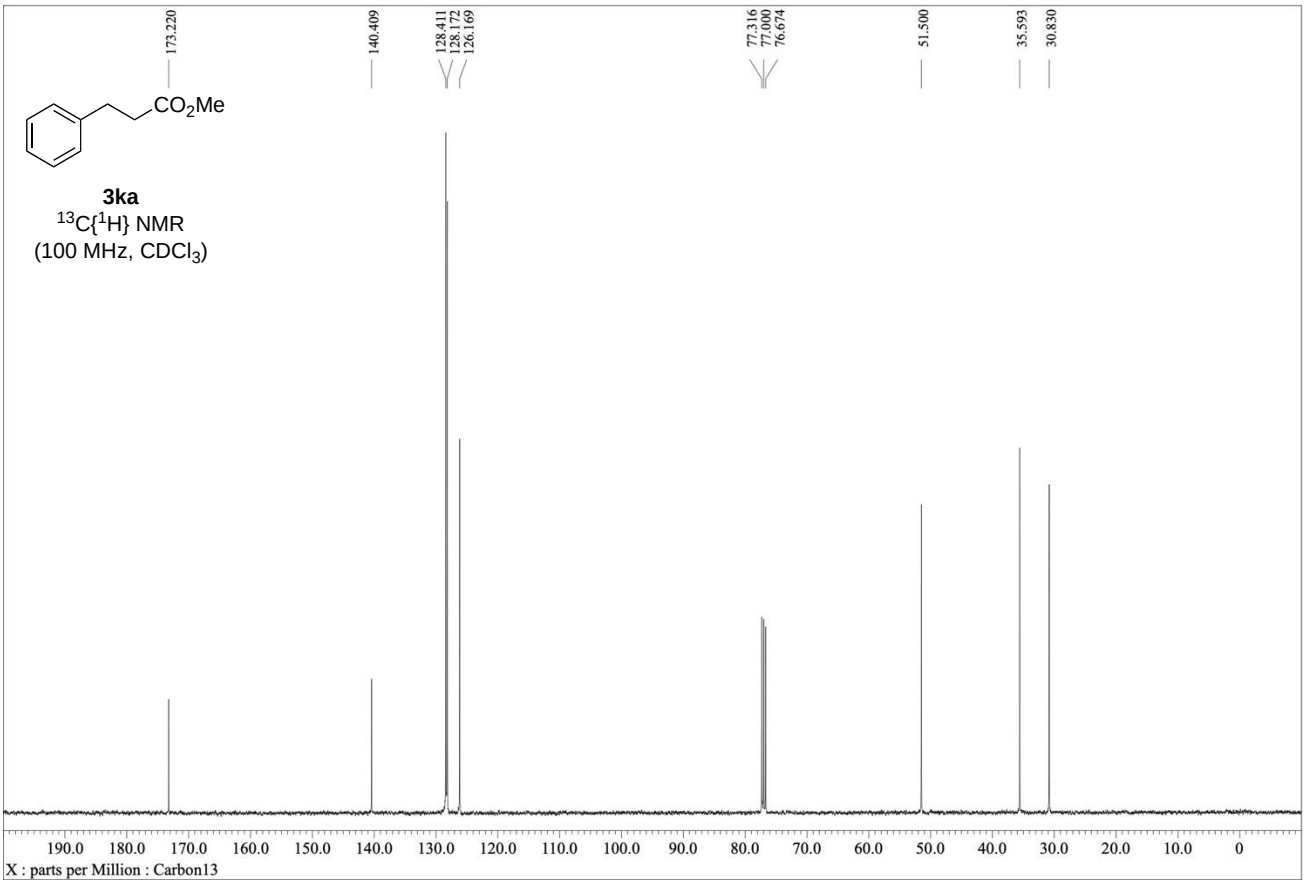
^{13}C{^{1}H} NMR

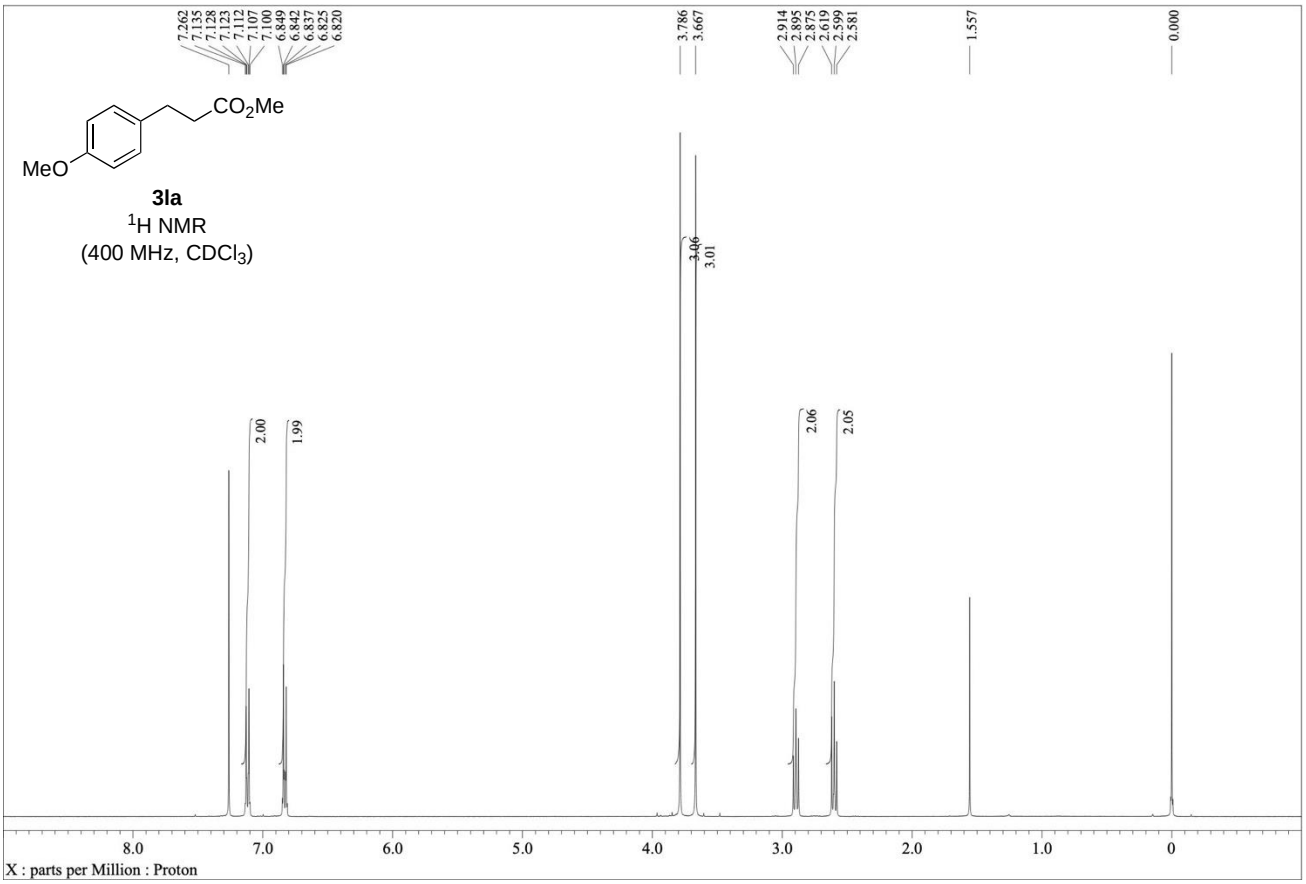
(100 MHz, $CDCl_3$)


7.286
7.265
7.241
7.219
3.673
3.252
3.234
3.216
2.947
2.928
2.911
2.678
1.636
0.000
CO2Me
Me
N
N
3ja
1H NMR
(400 MHz, CDCl3)
2.12
1.00
3.00
2.02
2.00
3.01
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
X : parts per Million : Proton


173.086
159.124
158.041
126.773
126.648
77.316
77.000
76.674
51.548
32.431
30.390
21.784
CO2Me
Me
N
N
3ja
13C{1H} NMR
(100 MHz, CDCl3)
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
X : parts per Million : Carbon13


7.312
7.308
7.298
7.295
7.293
7.290
7.283
7.274
7.261
7.223
7.211
7.206
7.192
3.674
2.976
2.957
2.936
2.659
2.646
2.641
2.638
2.633
2.619
1.553
0.000
CO2Me
3ka
1H NMR
(400 MHz, CDCl3)
2.97
2.00
3.12
2.11
2.10
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
X : parts per Million : Proton

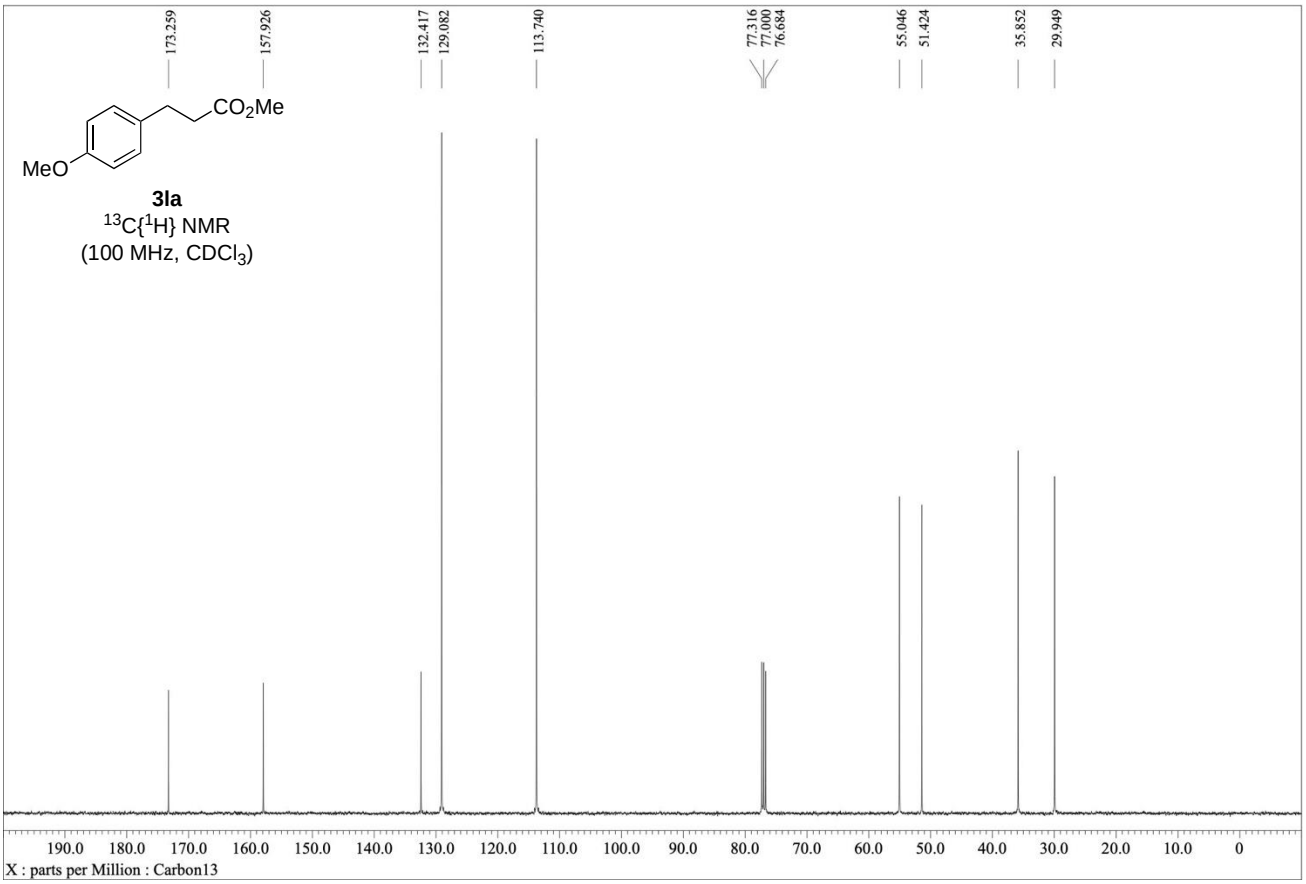

173.220
140.409
128.411
128.172
126.169
77.316
77.000
76.674
51.500
35.593
30.830
CO2Me
3ka
13C{1H} NMR
(100 MHz, CDCl3)
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
X : parts per Million : Carbon13



3la

^{1}H NMR

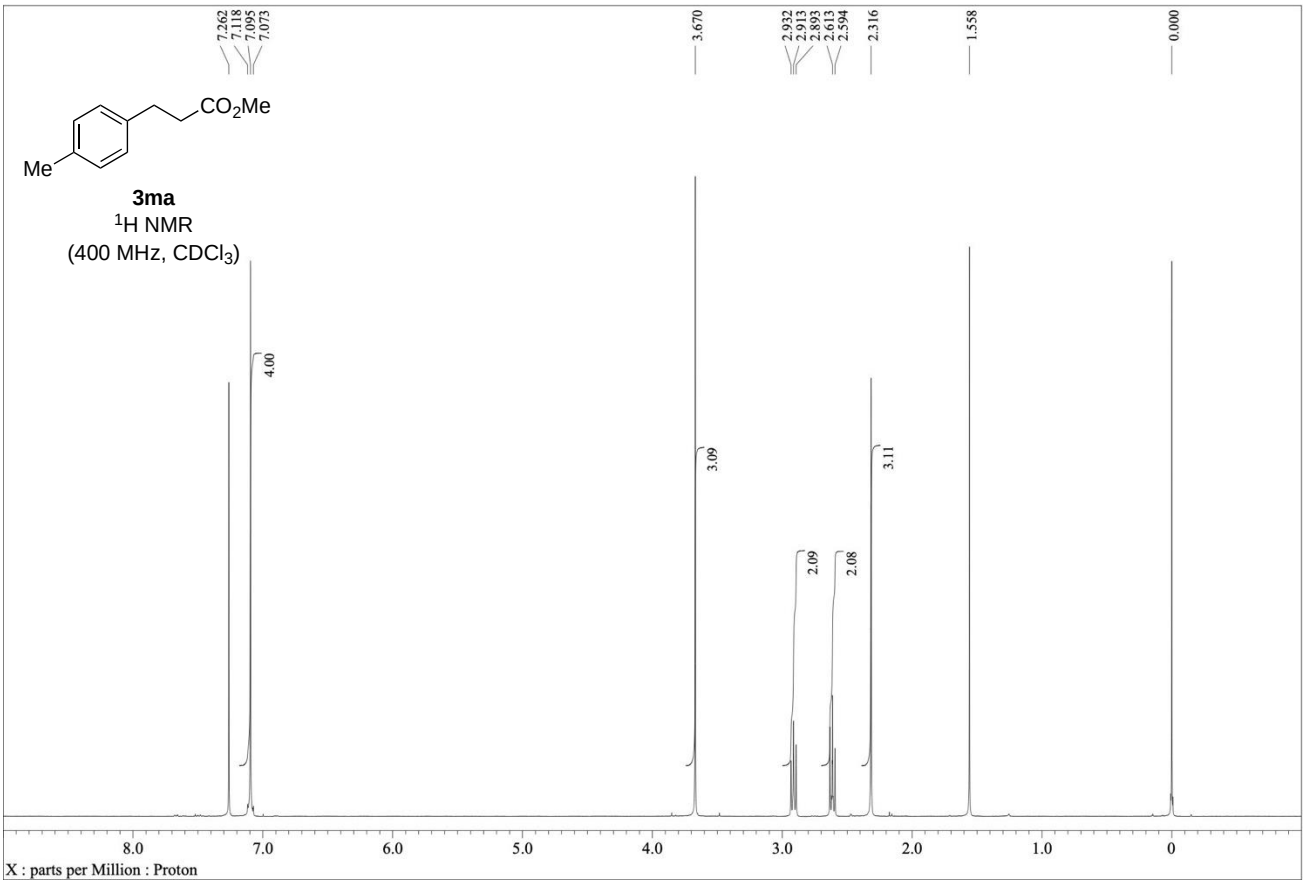
(400 MHz, $CDCl_3$)

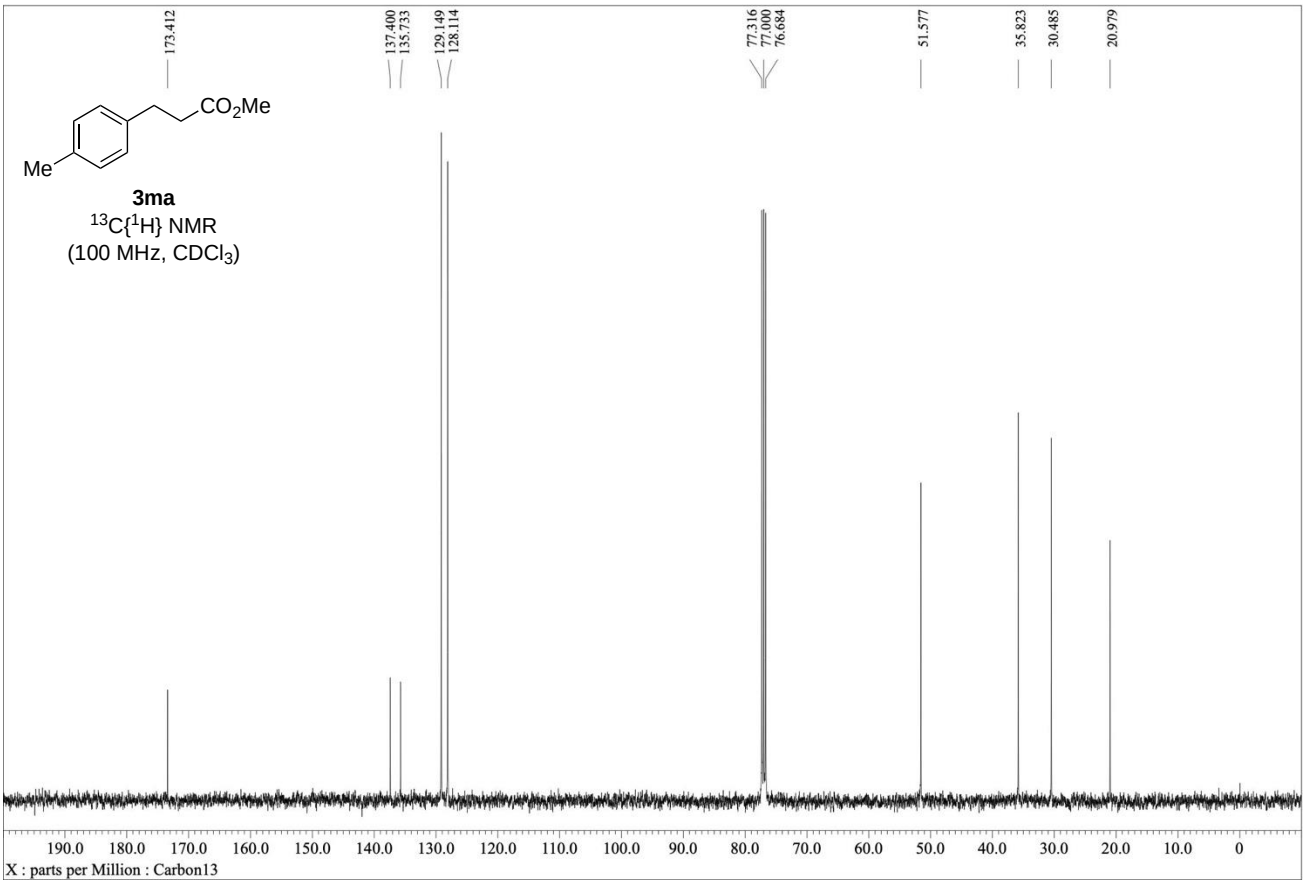


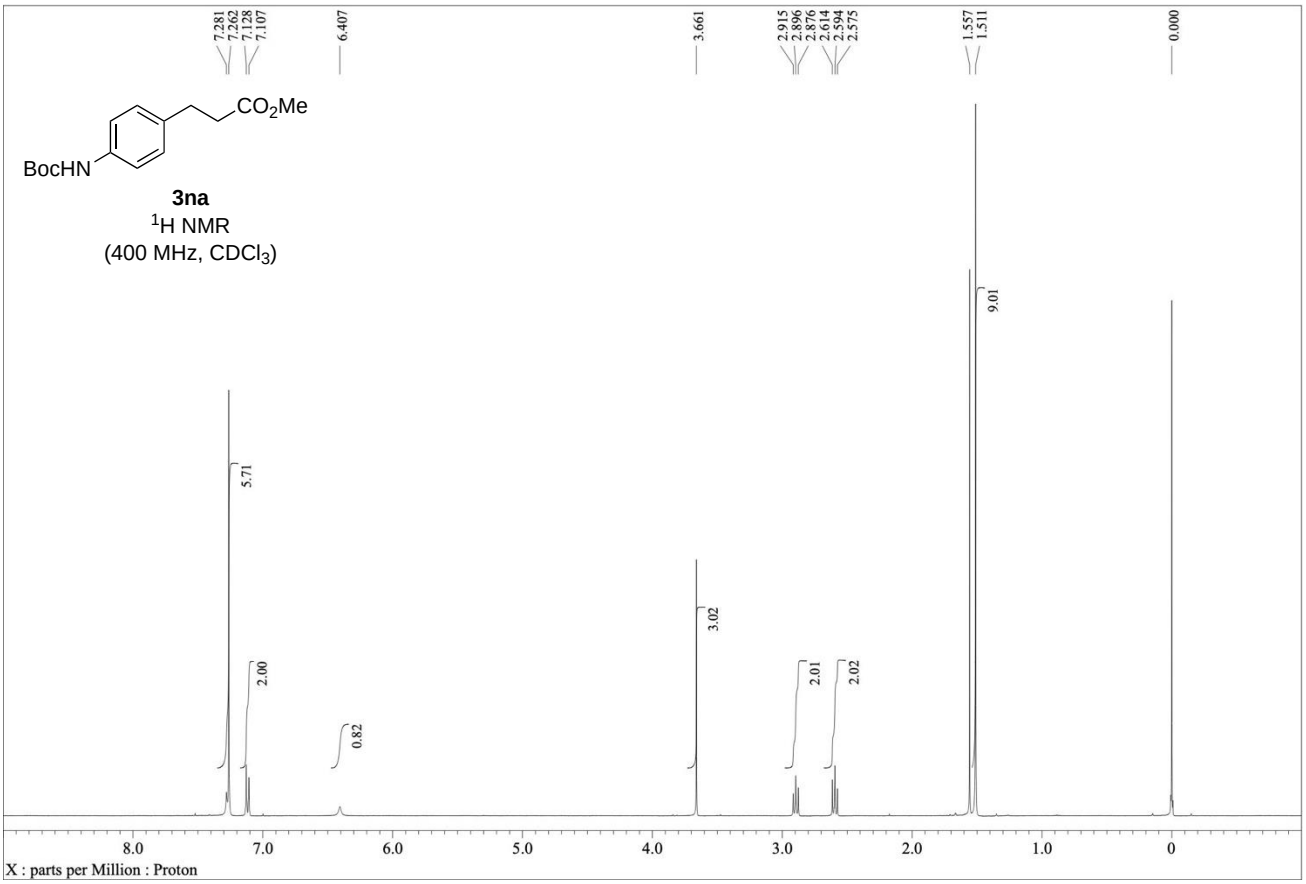
3la

$^{13}C\{^{1}H\}$ NMR

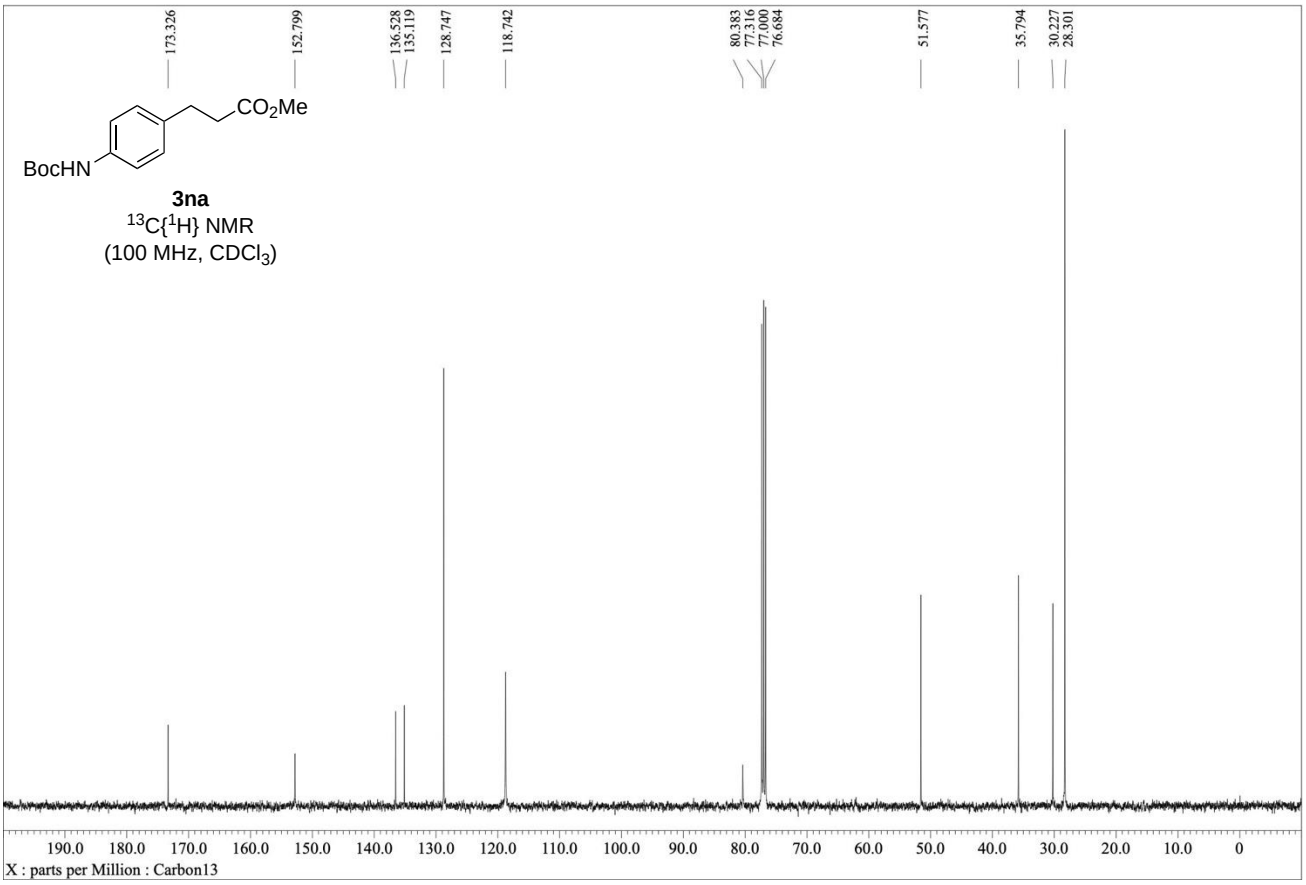
(100 MHz, $CDCl_3$)


7.262
7.118
7.095
7.073
3.670
2.932
2.913
2.893
2.613
2.594
2.316
1.558
0.000
CO2Me
Me
3ma
1H NMR
(400 MHz, CDCl3)
4.00
3.09
2.09
2.08
3.11
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
X : parts per Million : Proton

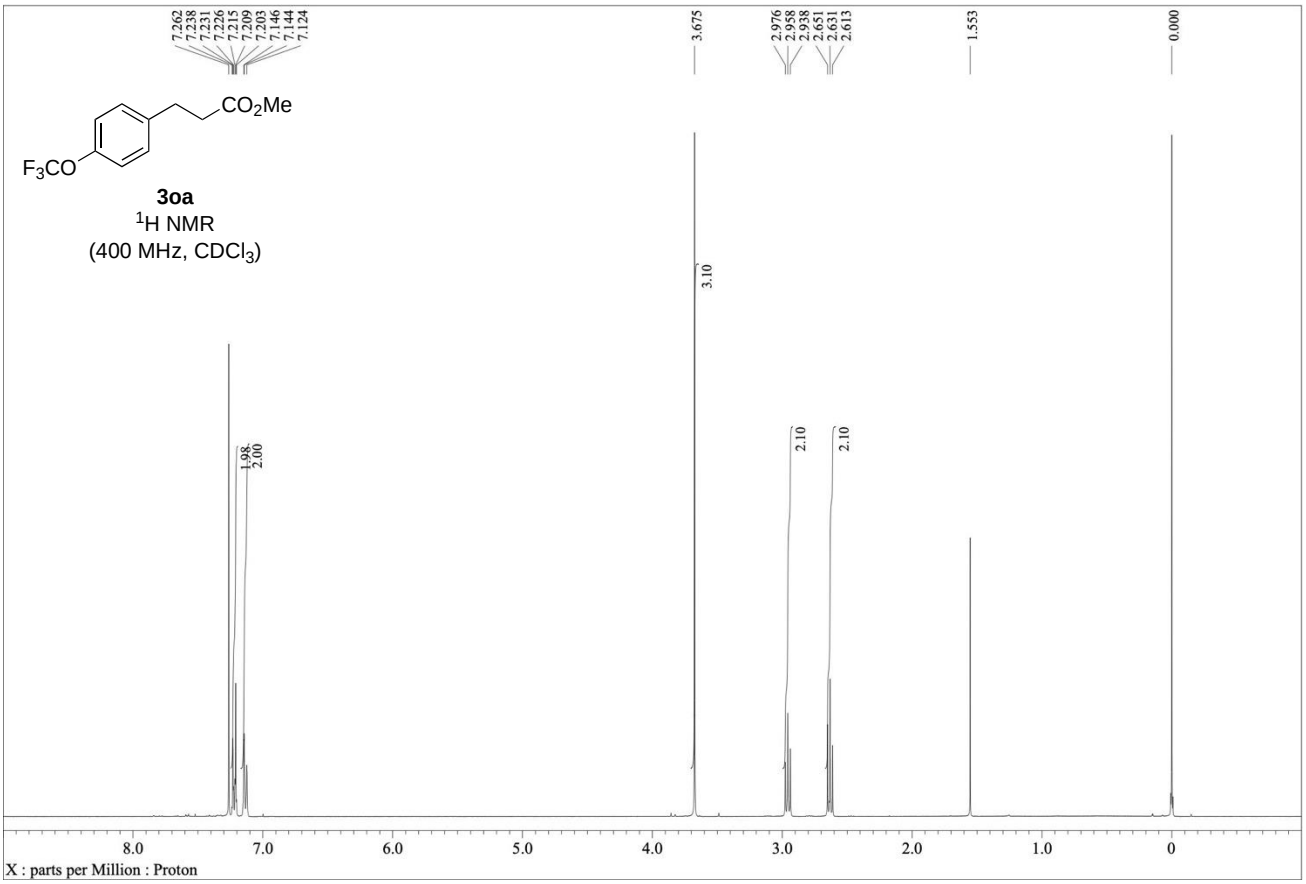

173.412
137.400
135.733
129.149
128.114
77.316
77.000
76.684
51.577
35.823
30.485
20.979
CO2Me
Me
3ma
13C{1H} NMR
(100 MHz, CDCl3)
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
X : parts per Million : Carbon13

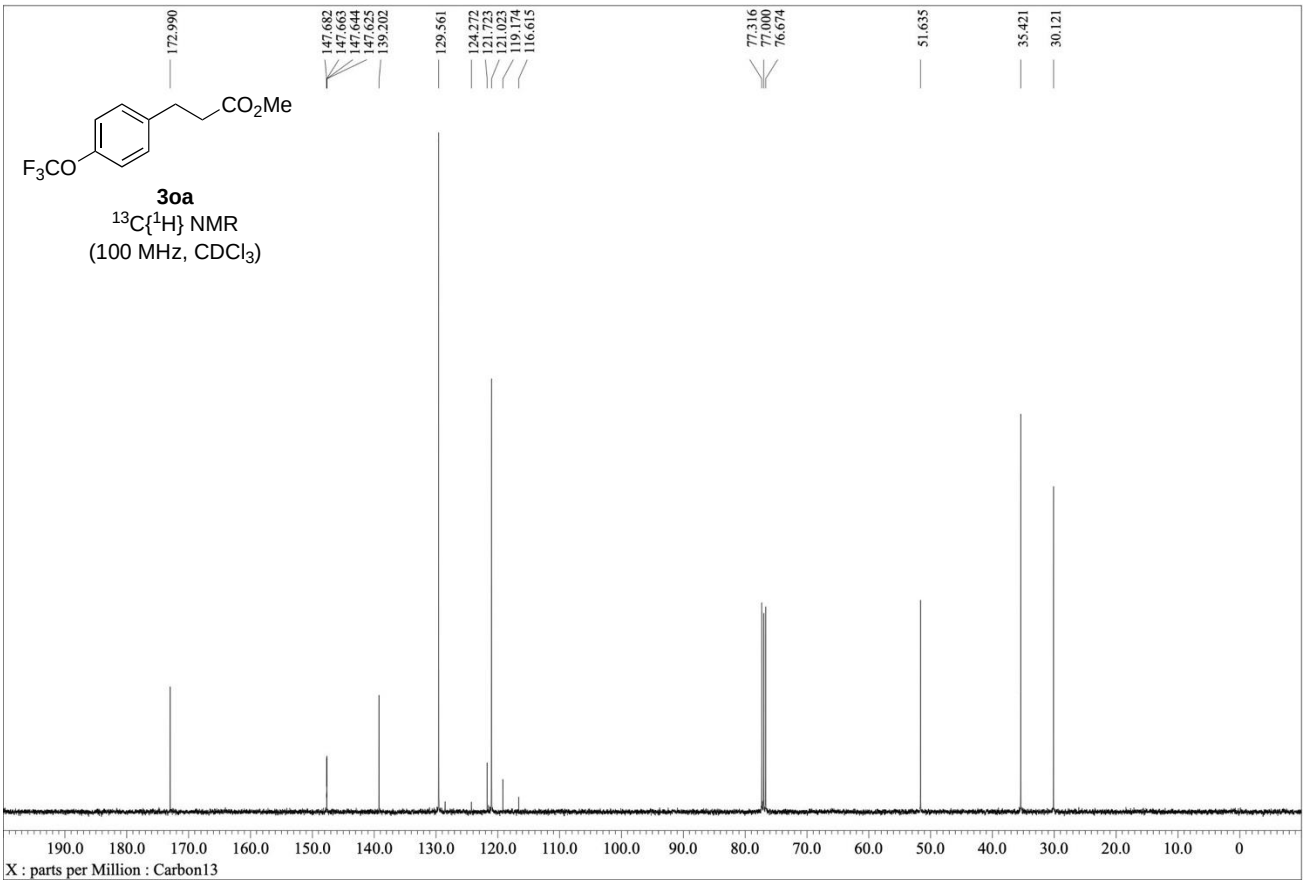


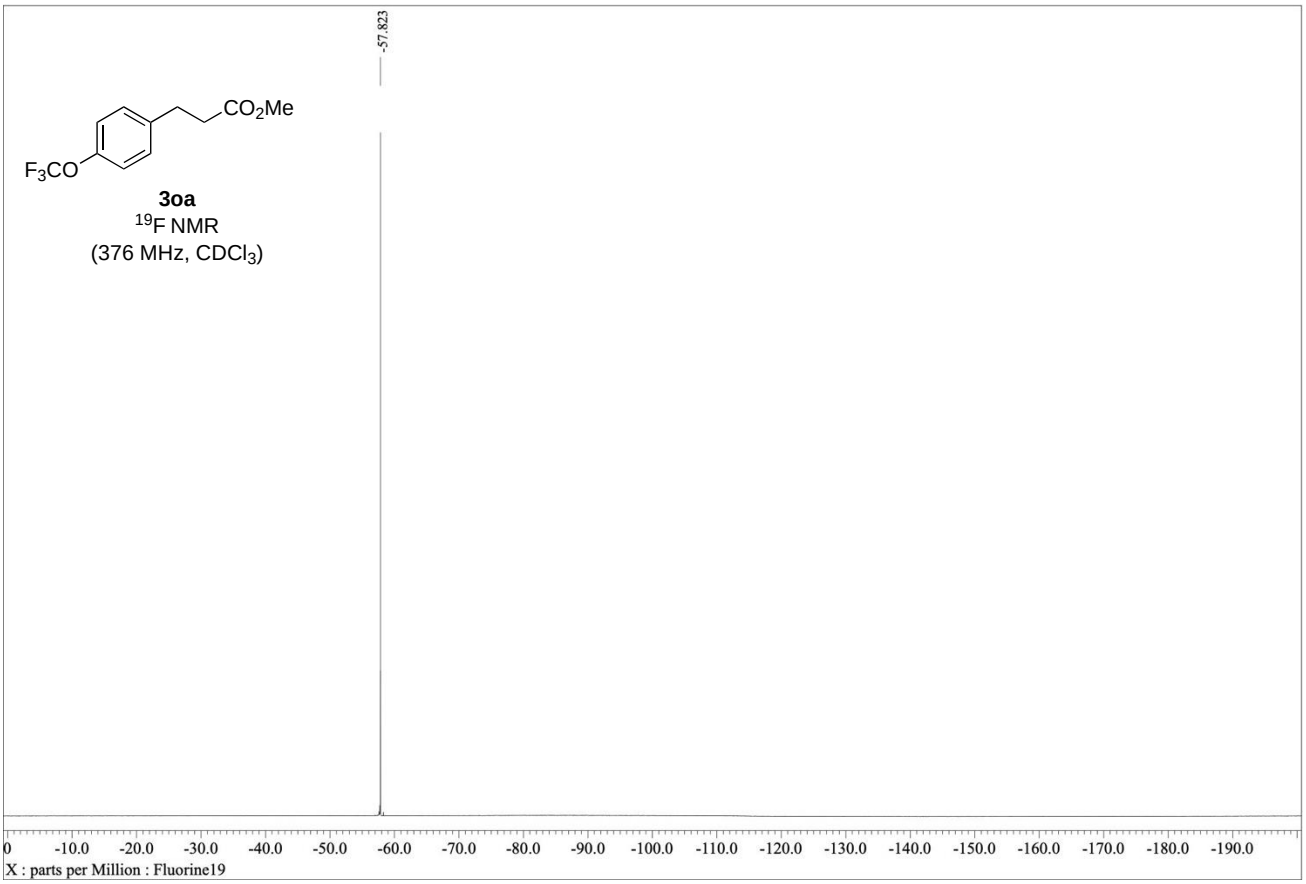
3na
^{1}H NMR
(400 MHz, $CDCl_3$)



3na
$^{13}C\{^{1}H\}$ NMR
(100 MHz, $CDCl_3$)


7.262
7.238
7.231
7.226
7.215
7.209
7.203
7.146
7.144
7.124
3.675
2.976
2.958
2.938
2.651
2.631
2.613
1.553
0.000
CO2Me
F3CO
3oa
1H NMR
(400 MHz, CDCl3)
3.10
1.98
2.00
2.10
2.10
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
X : parts per Million : Proton

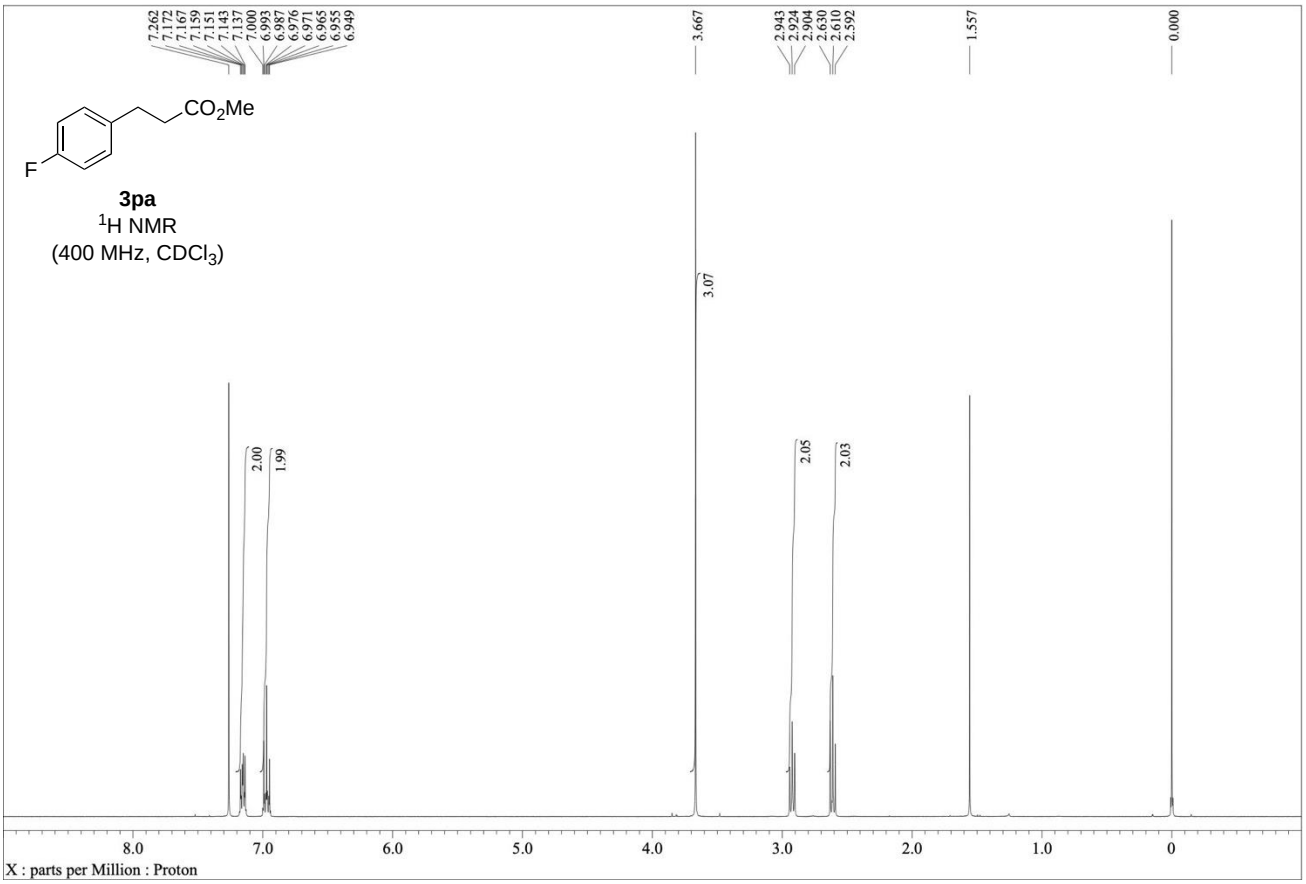

172.990
147.682
147.663
147.644
147.625
139.202
129.561
124.272
121.723
121.023
119.174
116.615
77.316
77.000
76.674
51.635
35.421
30.121
CO2Me
F3CO
3oa
13C{1H} NMR
(100 MHz, CDCl3)
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
X : parts per Million : Carbon13

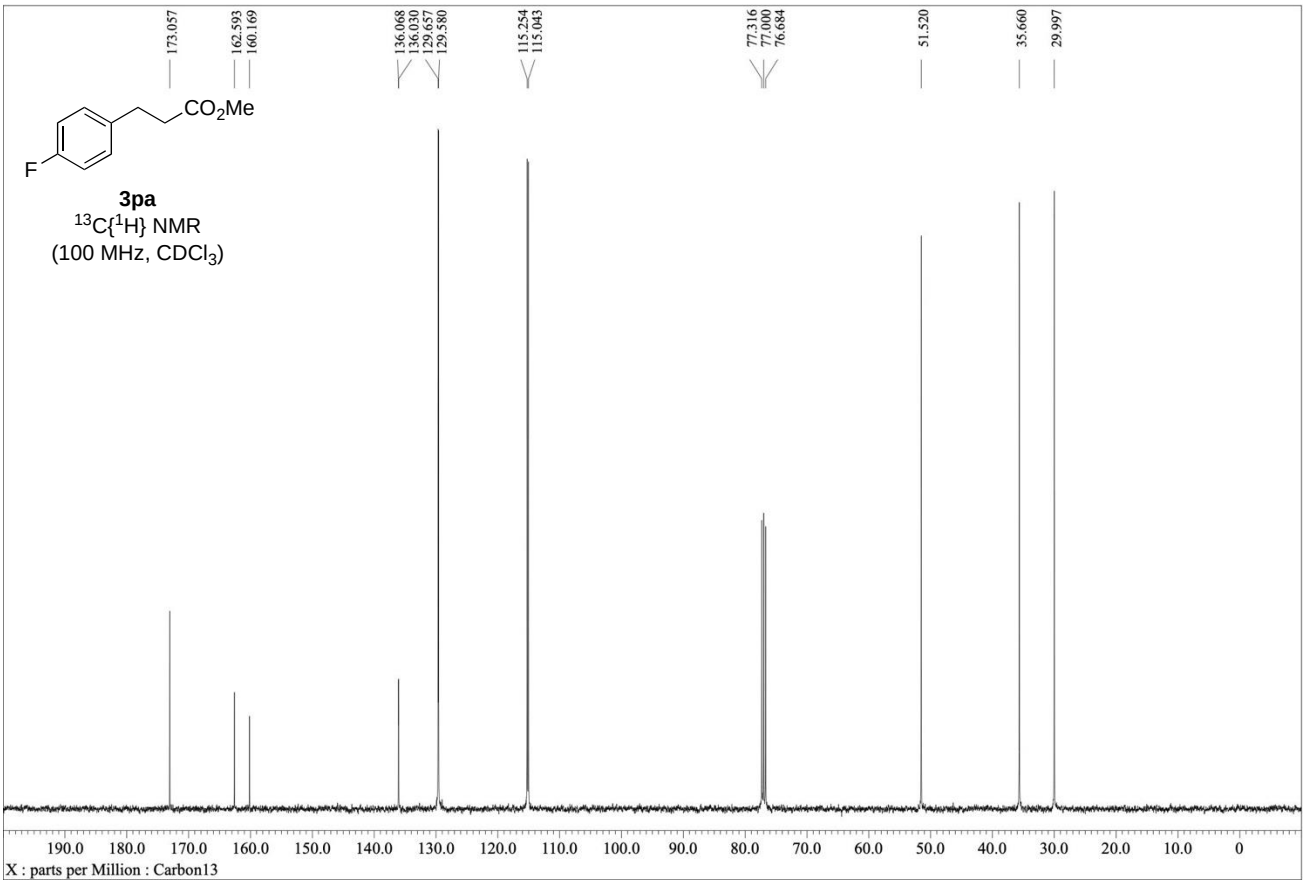


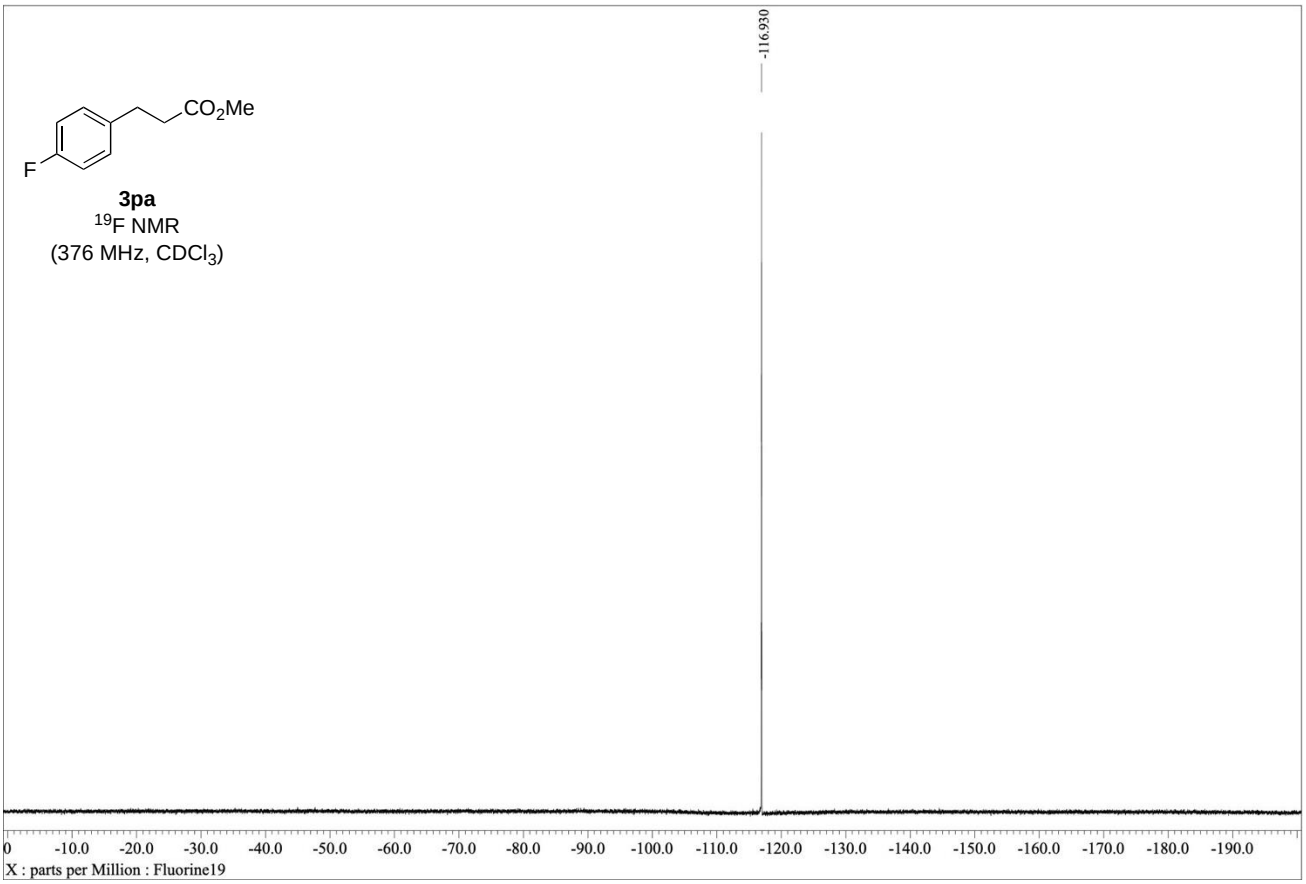
3oa

^{19}F NMR

(376 MHz, $CDCl_3$)


7.262
7.172
7.167
7.159
7.151
7.143
7.137
7.000
6.993
6.987
6.976
6.971
6.965
6.955
6.949
3.667
2.943
2.924
2.904
2.630
2.610
2.592
1.557
0.000
CO2Me
F
3pa
1H NMR
(400 MHz, CDCl3)
3.07
2.00
1.99
2.05
2.03
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
X : parts per Million : Proton

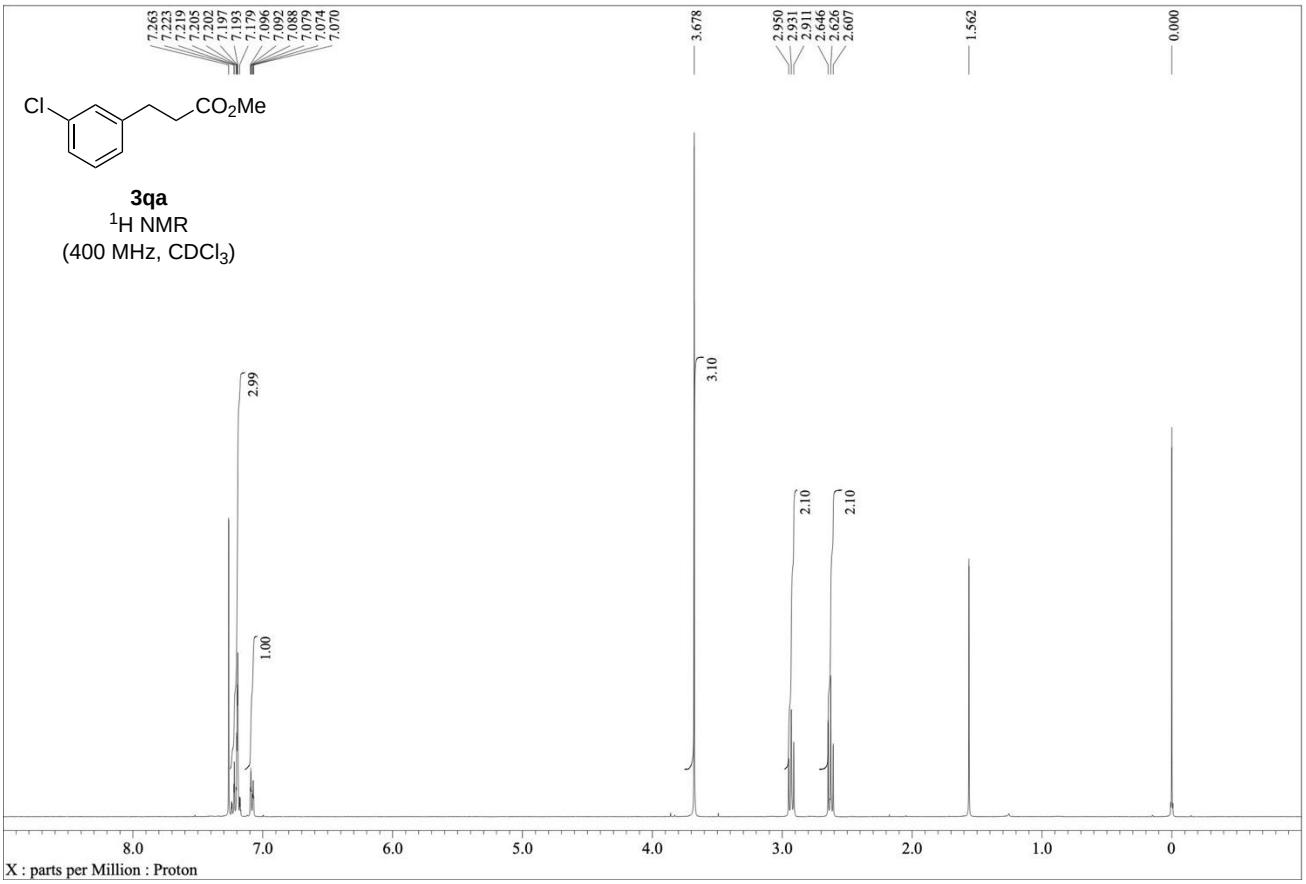

173.057
162.593
160.169
136.068
136.030
129.657
129.580
115.254
115.043
77.316
77.000
76.684
51.520
35.660
29.997
CO2Me
F
3pa
13C{1H} NMR
(100 MHz, CDCl3)
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
X : parts per Million : Carbon13



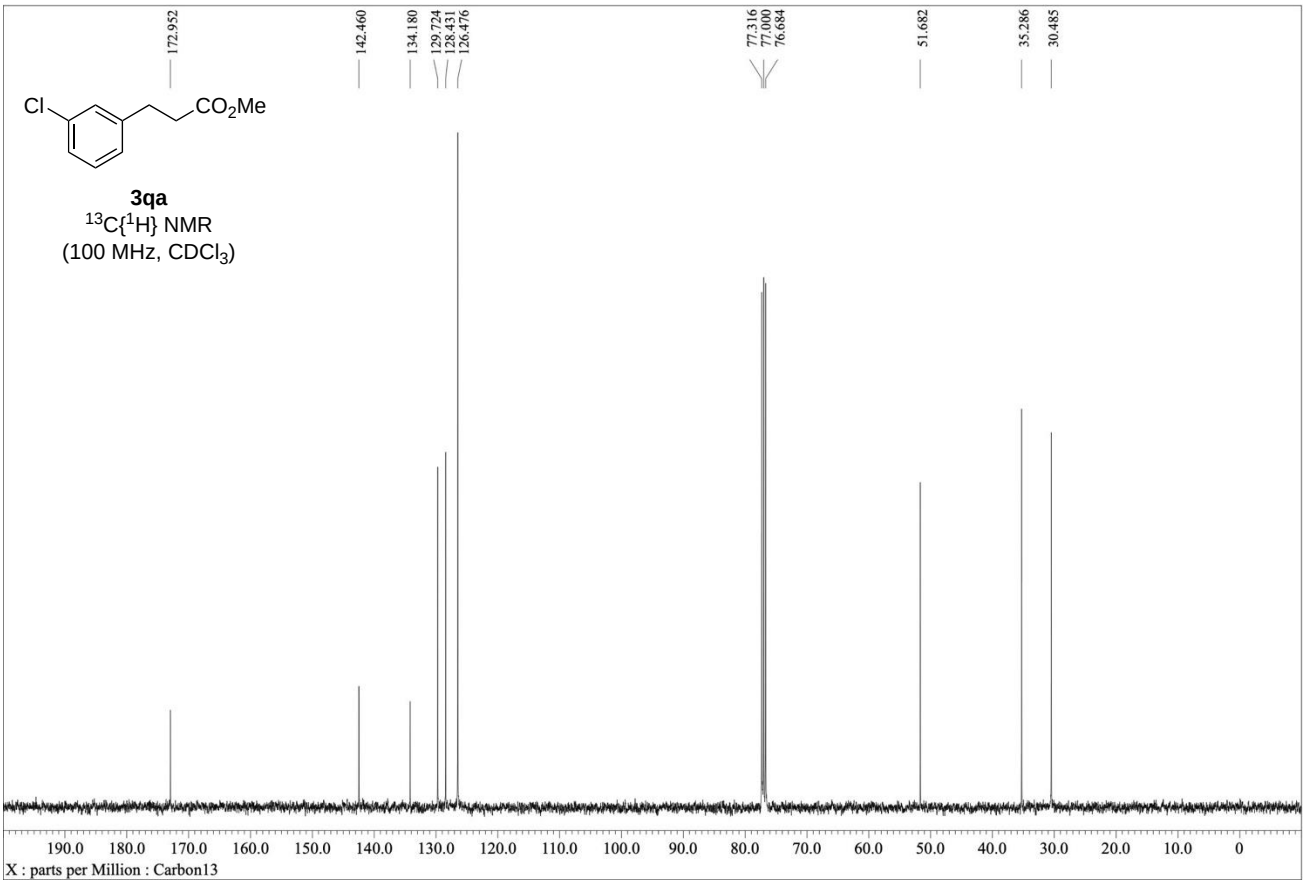
3pa

^{19}F NMR

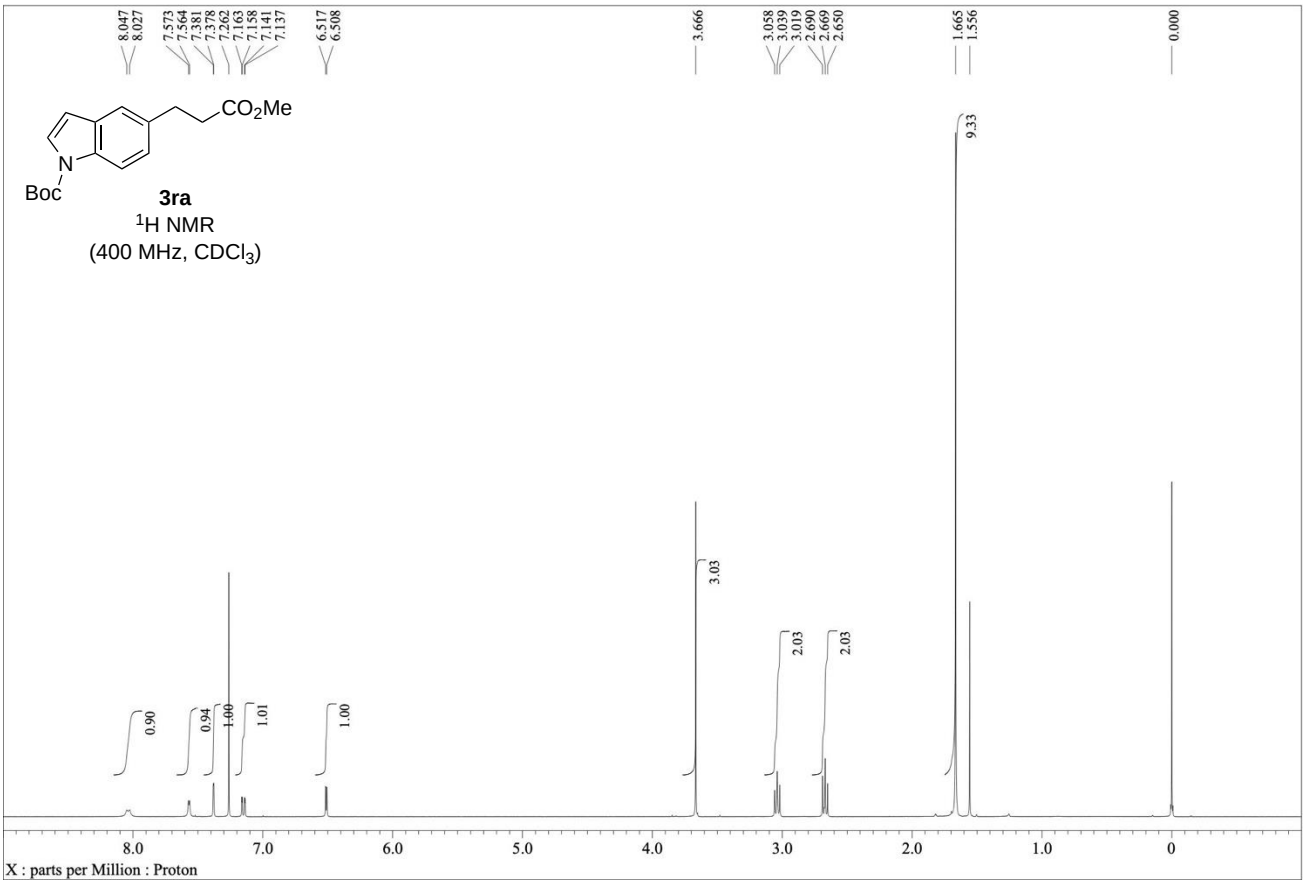
(376 MHz, $CDCl_3$)

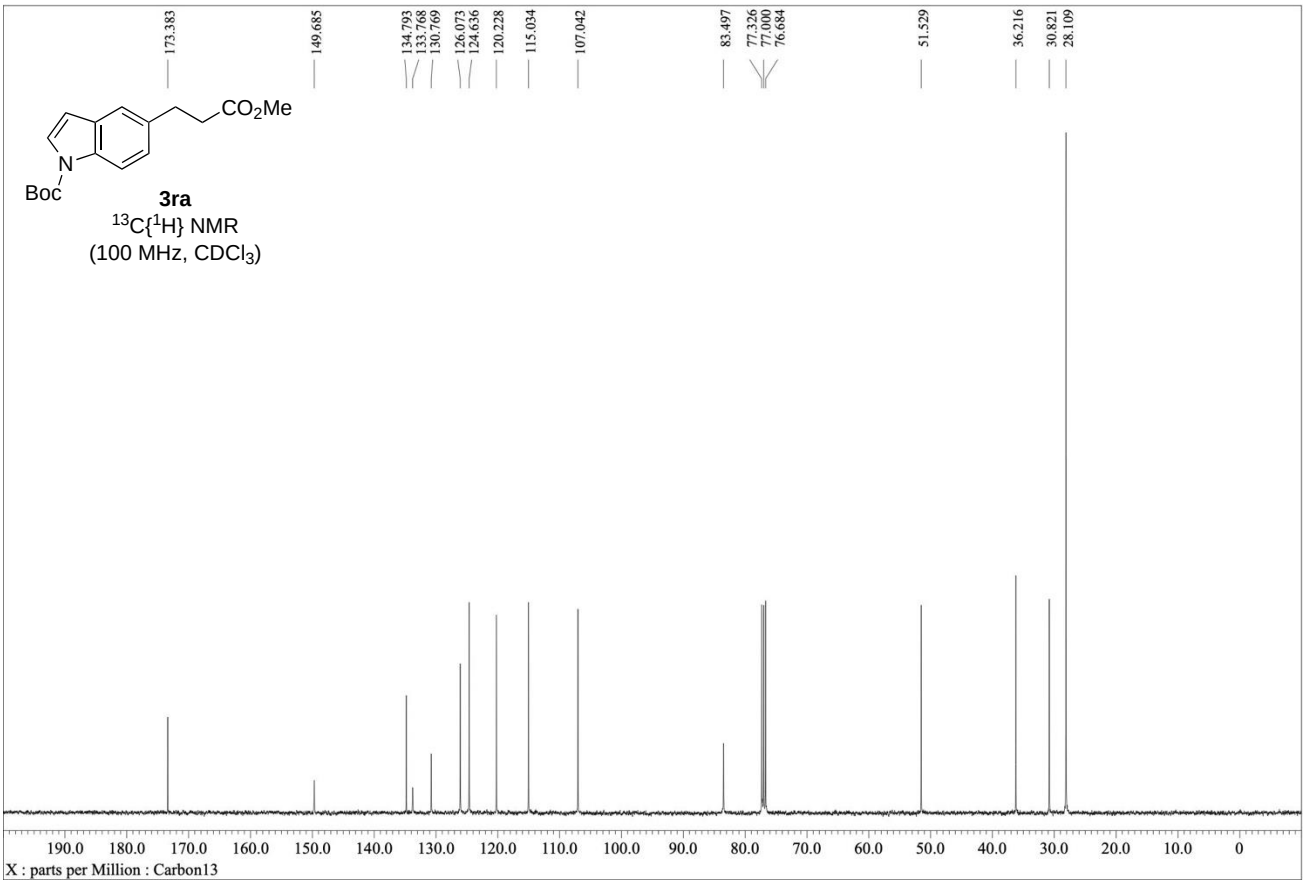


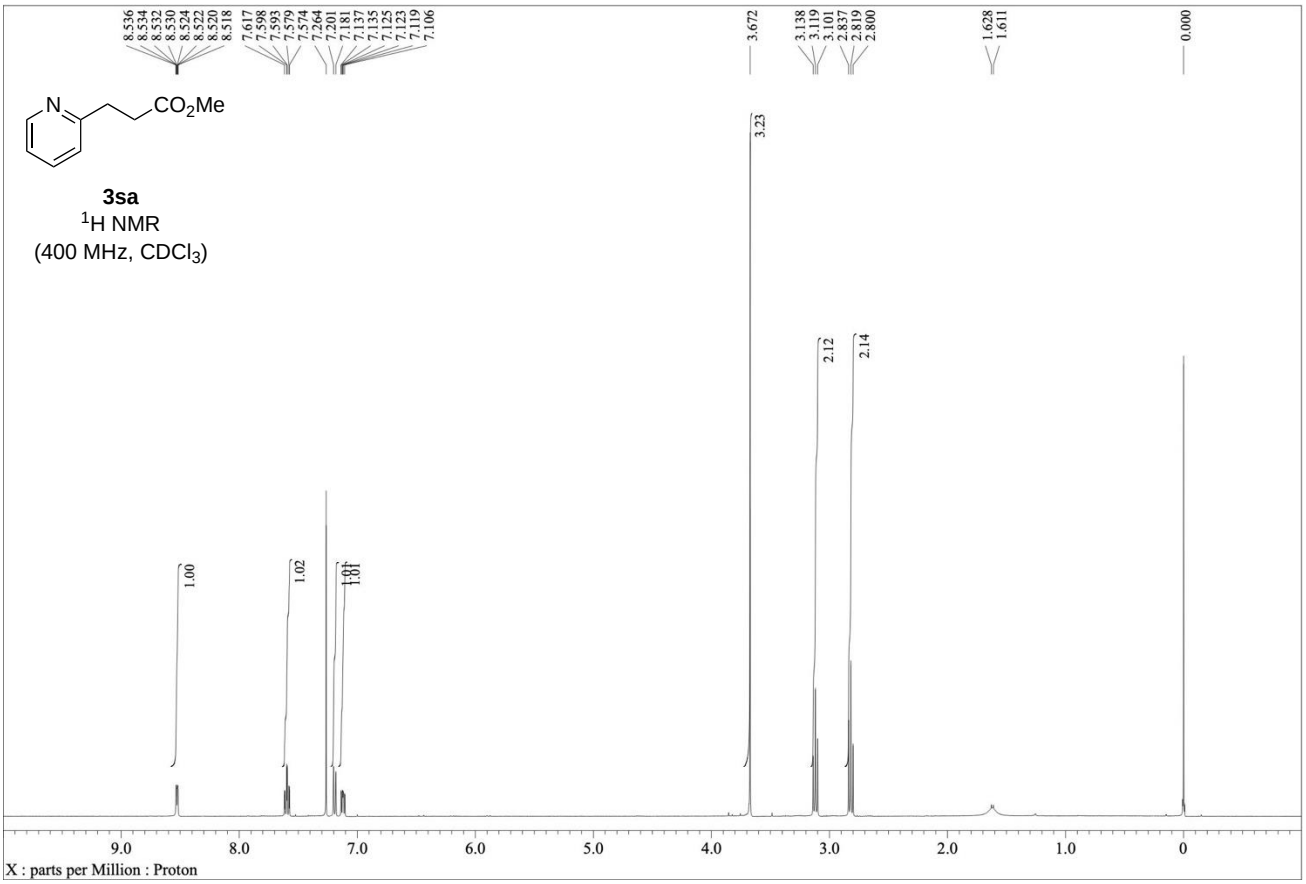
3qa
^{1}H NMR
(400 MHz, $CDCl_3$)



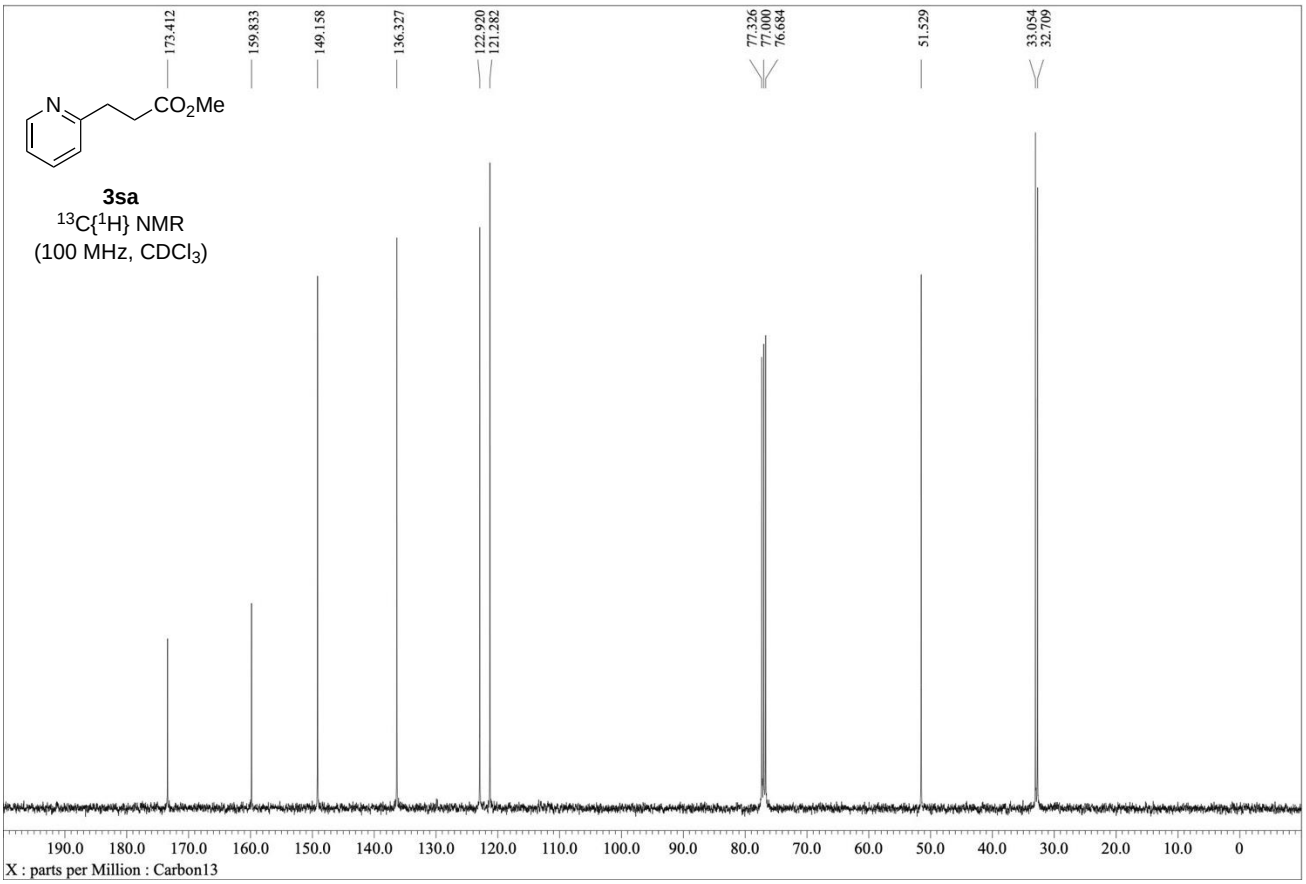
3qa
$^{13}C\{^{1}H\}$ NMR
(100 MHz, $CDCl_3$)


8.047
8.027
7.573
7.564
7.381
7.378
7.262
7.163
7.158
7.141
7.137
6.517
6.508
3.666
3.058
3.039
3.019
2.690
2.669
2.650
1.665
1.556
0.000
CO2Me
N
Boc
3ra
1H NMR
(400 MHz, CDCl3)
9.33
3.03
2.03
2.03
0.90
0.94
1.00
1.01
1.00
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
X : parts per Million : Proton

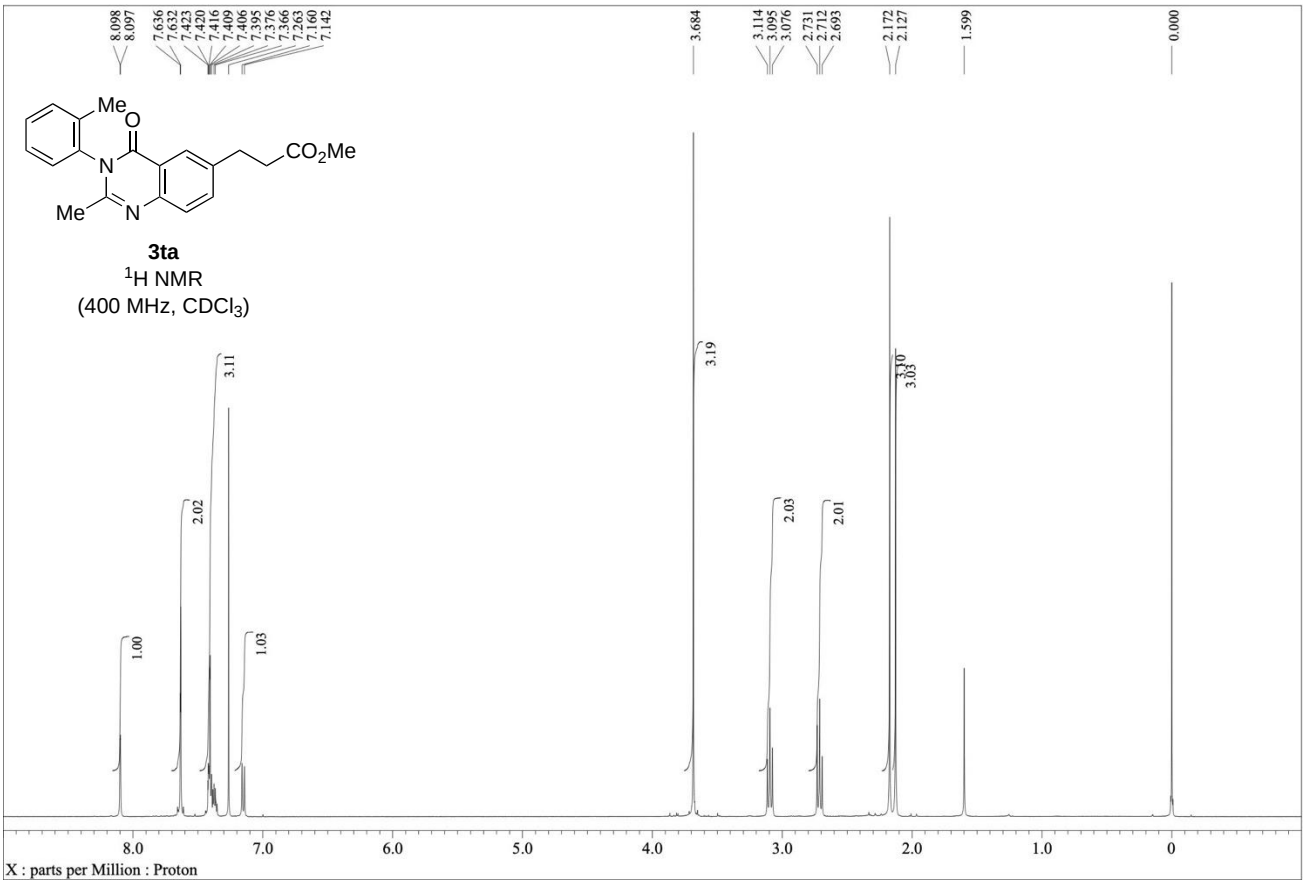

173.383
149.685
134.793
133.768
130.769
126.073
124.636
120.228
115.034
107.042
83.497
77.326
77.000
76.684
51.529
36.216
30.821
28.109
CO2Me
N
Boc
3ra
13C{1H} NMR
(100 MHz, CDCl3)
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
X : parts per Million : Carbon13



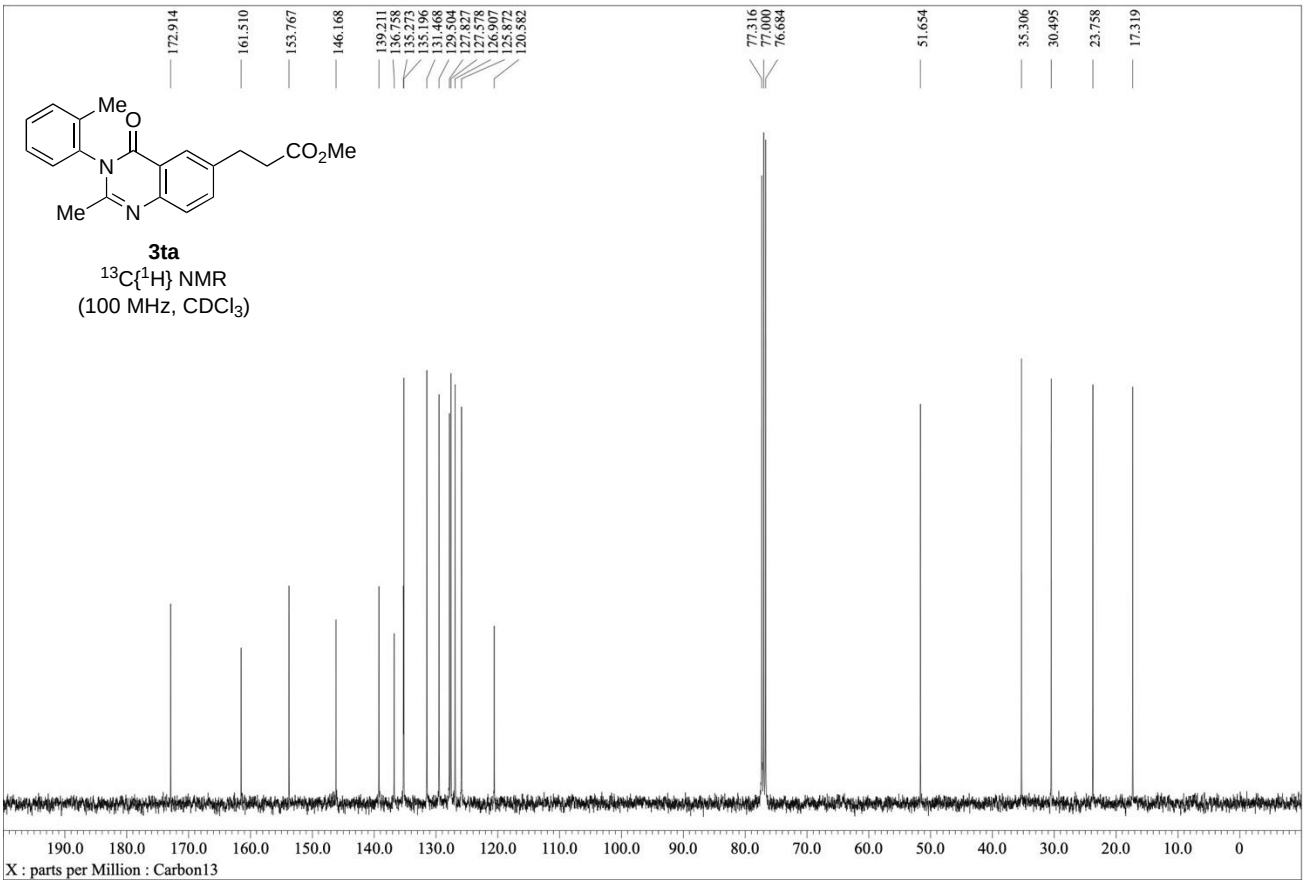
3sa
^{1}H NMR
(400 MHz, $CDCl_3$)



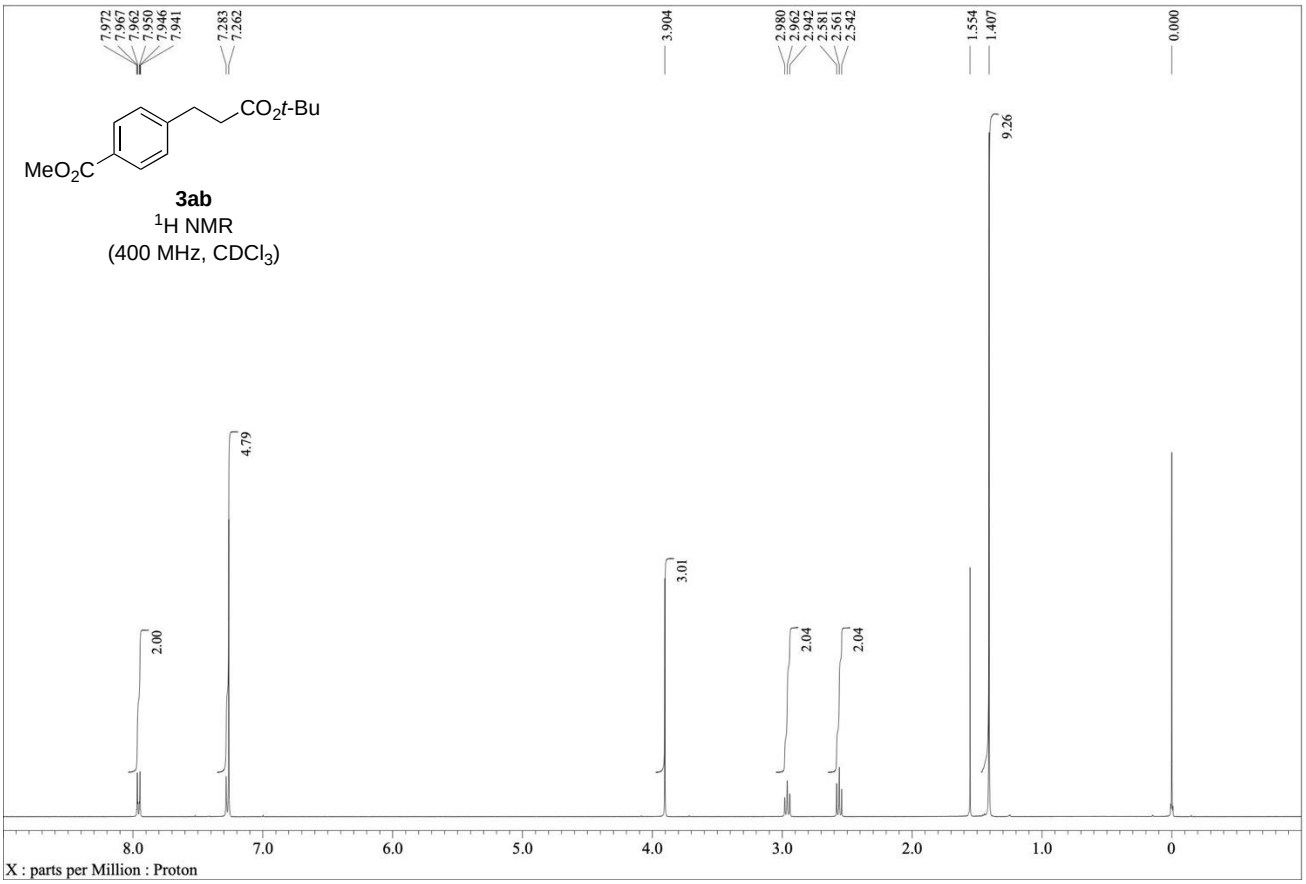
3sa
^{13}C{^{1}H} NMR
(100 MHz, $CDCl_3$)



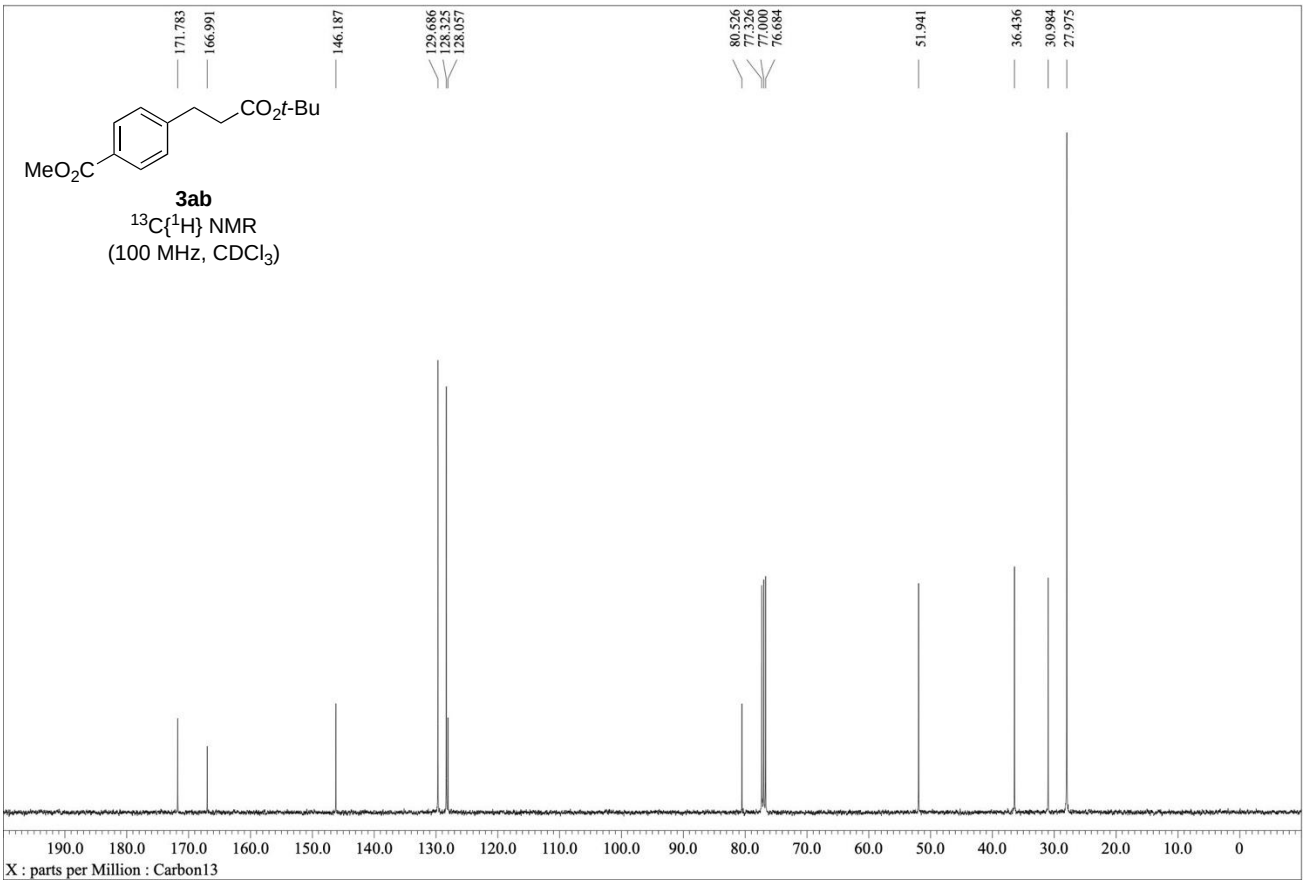
3ta
^{1}H NMR
(400 MHz, $CDCl_3$)



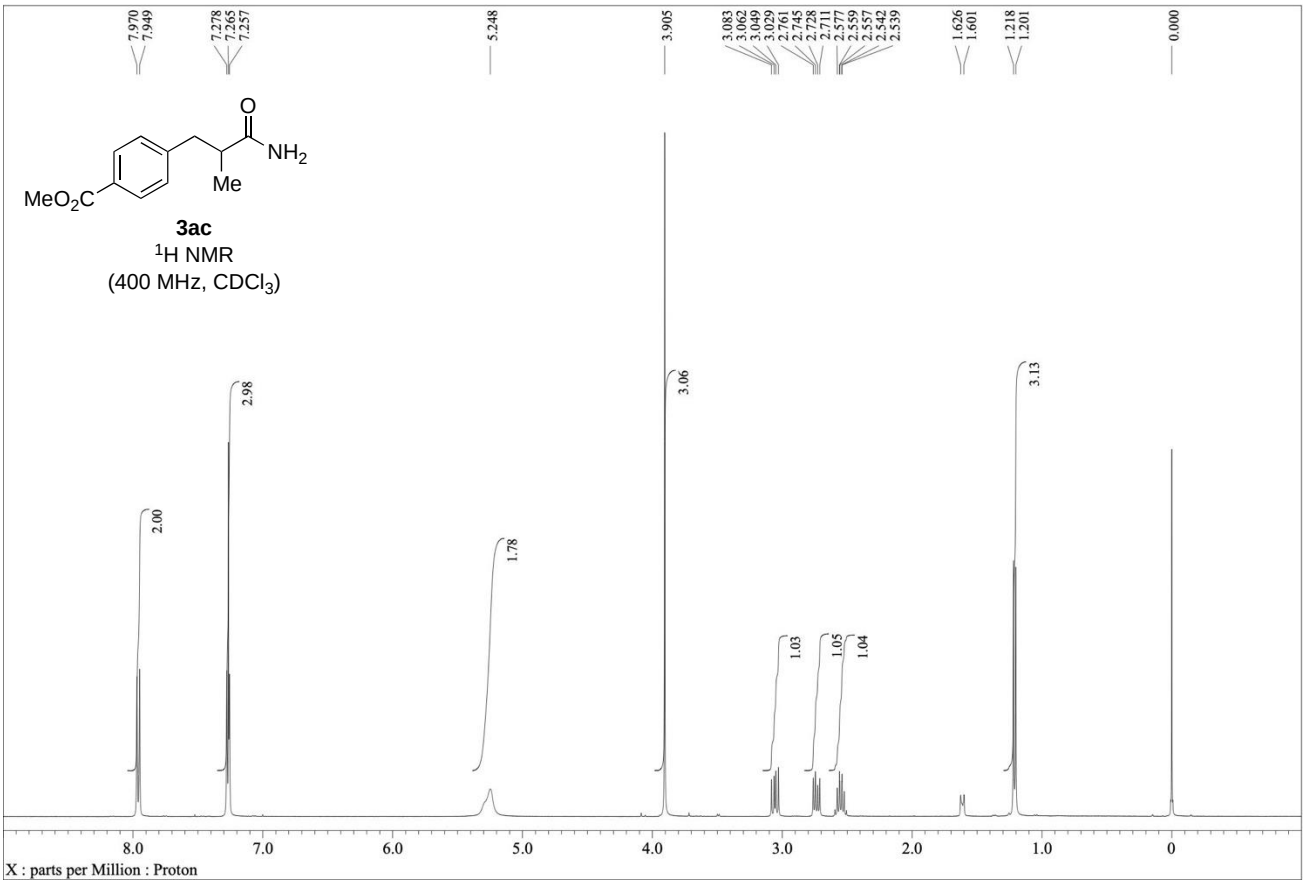
3ta
^{13}C{^{1}H} NMR
(100 MHz, $CDCl_3$)



3ab
^{1}H NMR
(400 MHz, $CDCl_3$)



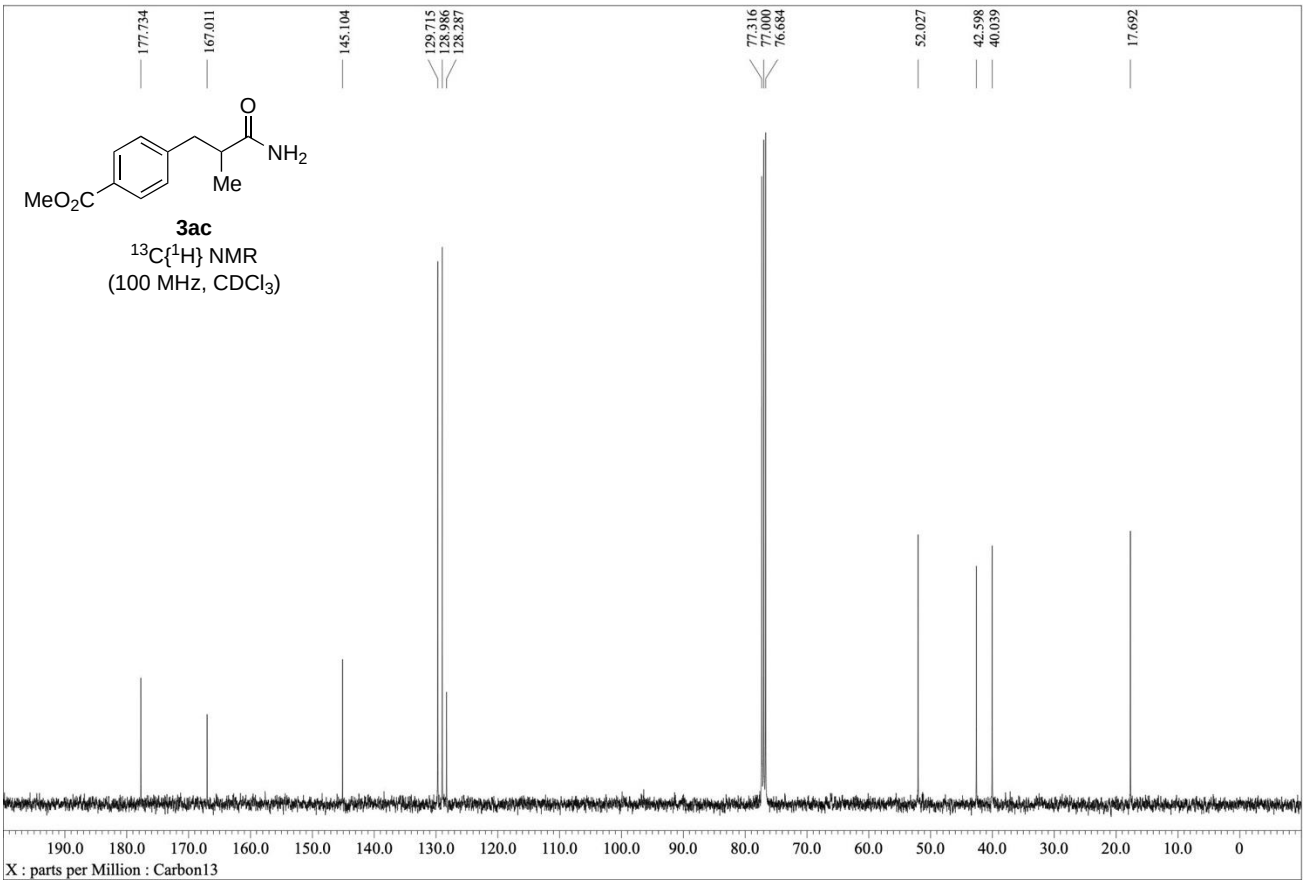
3ab
^{13}C{^{1}H} NMR
(100 MHz, $CDCl_3$)



3ac

^{1}H NMR

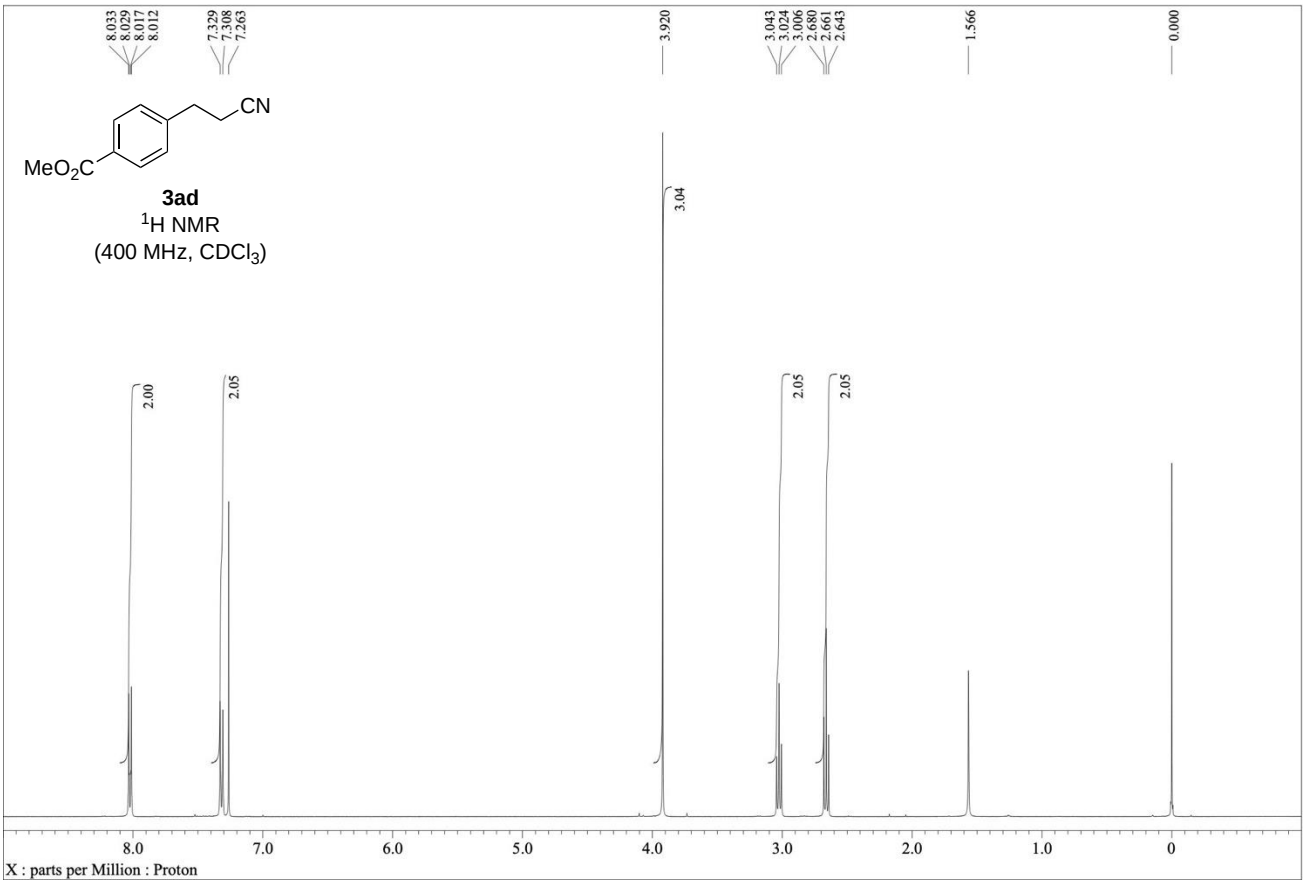
(400 MHz, $CDCl_3$)



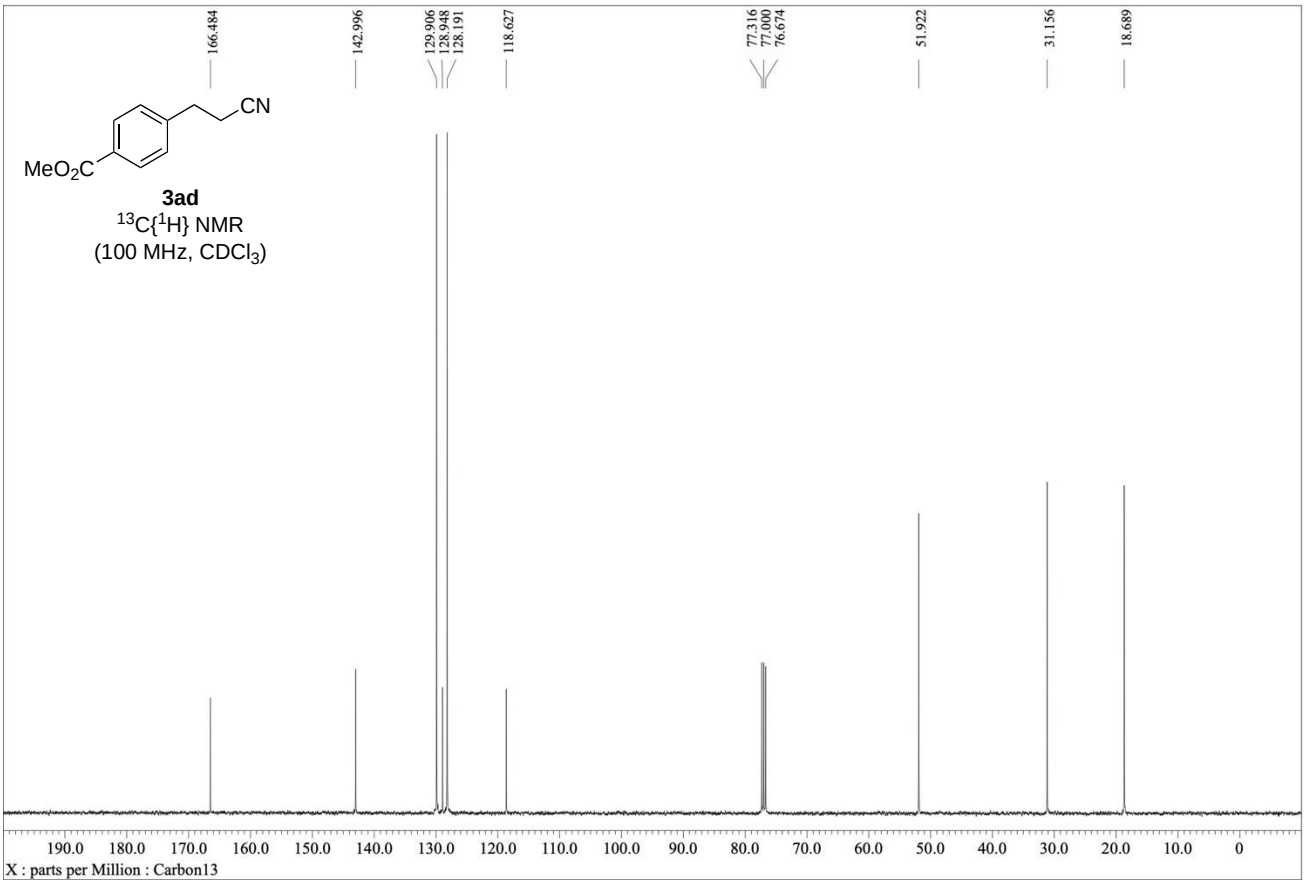
3ac

$^{13}C\{^{1}H\}$ NMR

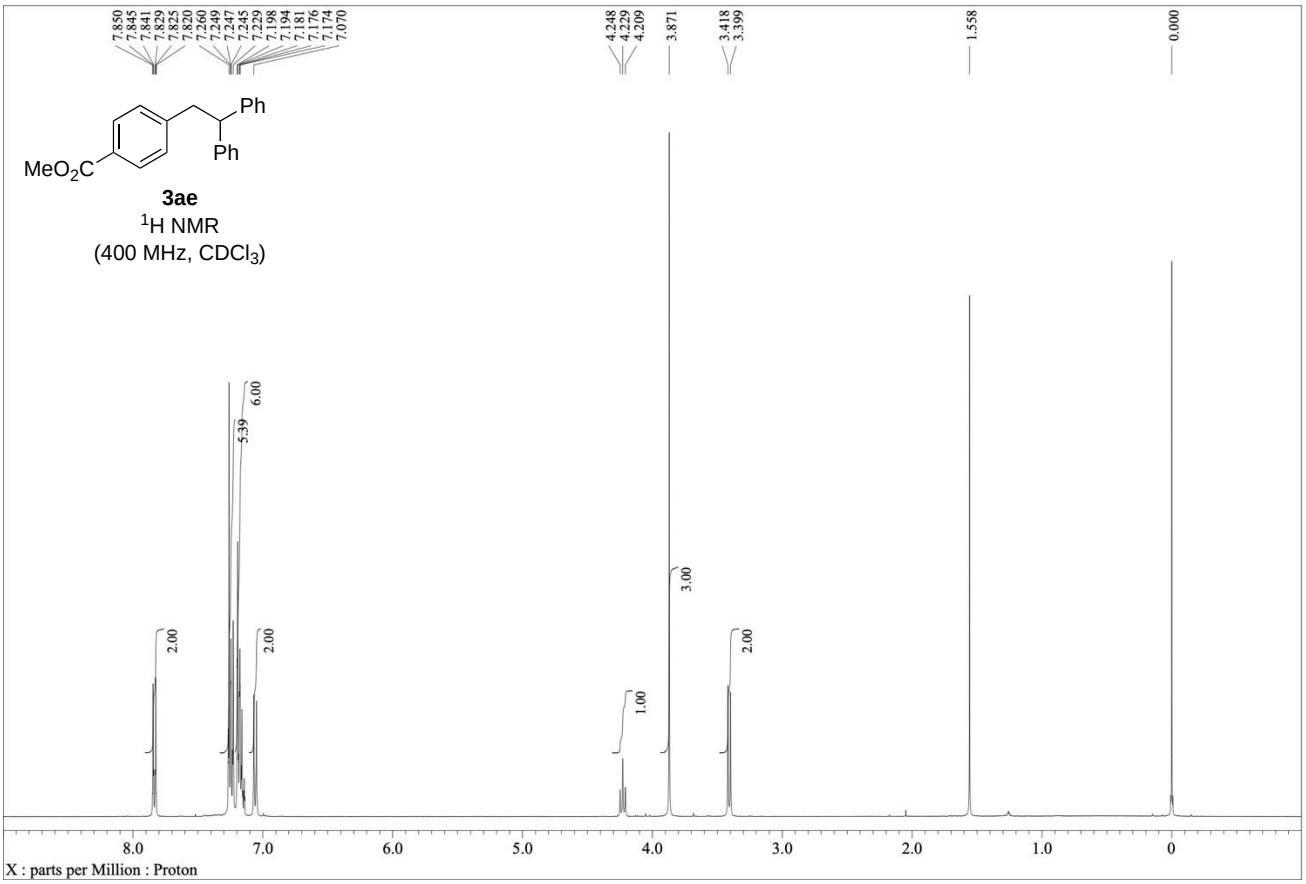
(100 MHz, $CDCl_3$)

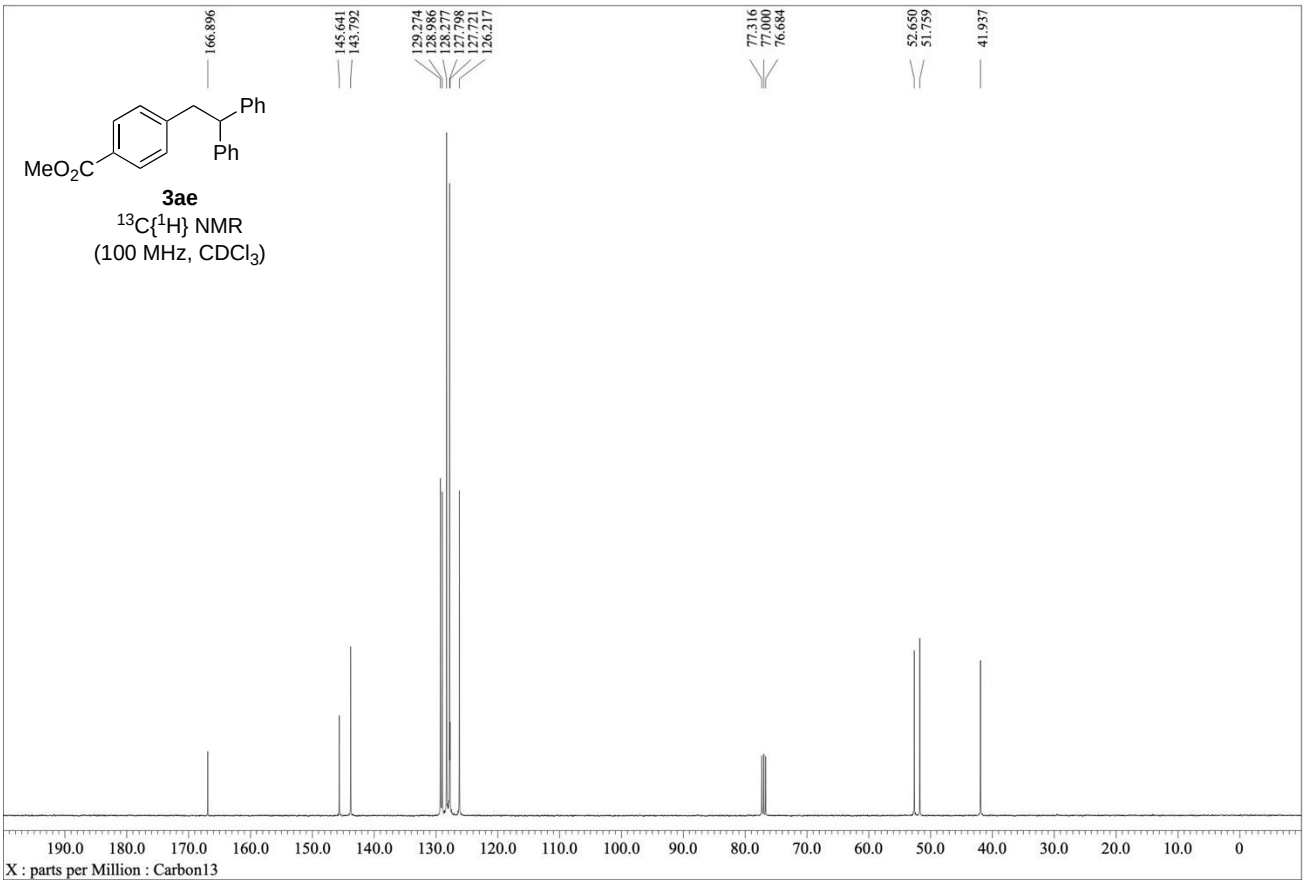


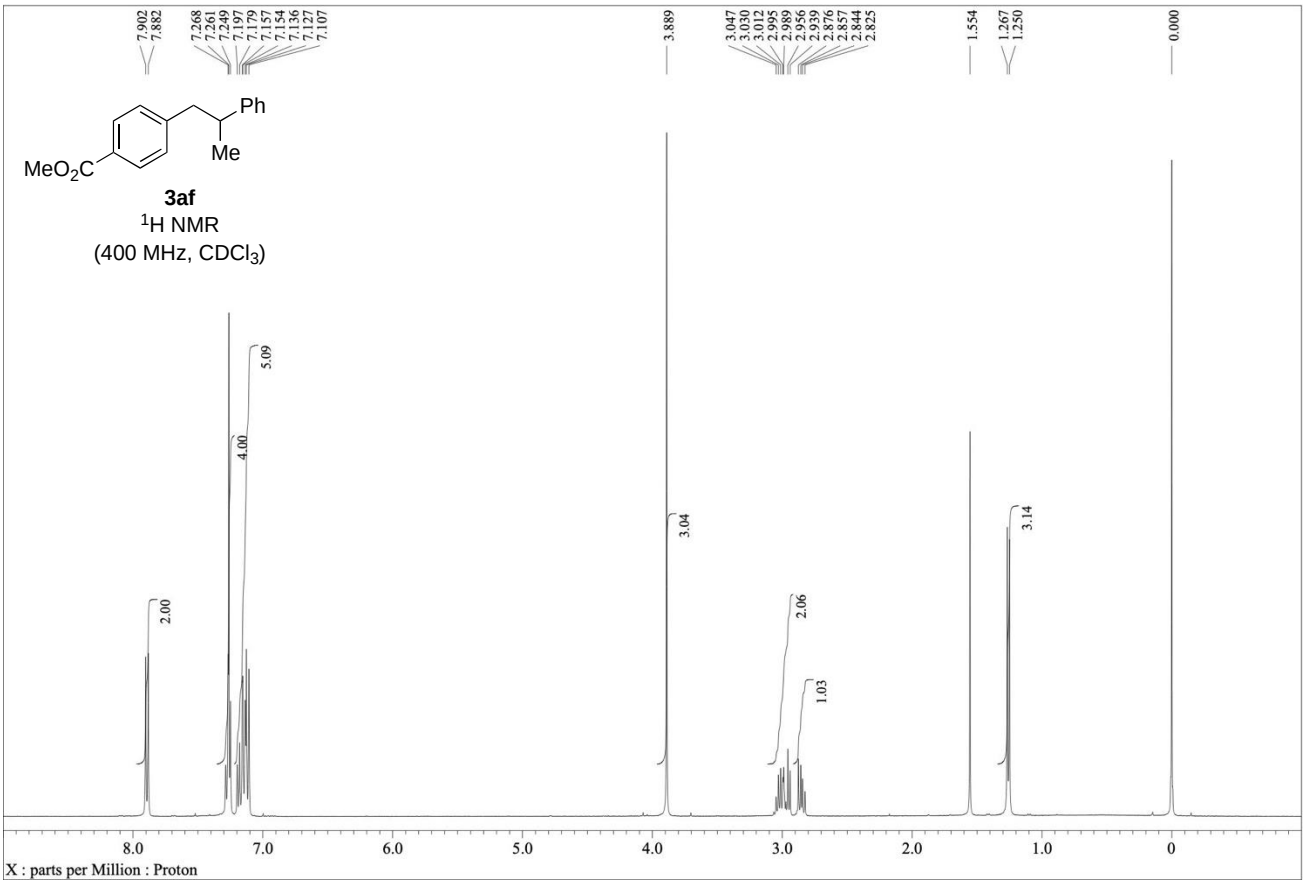
3ad
^{1}H NMR
(400 MHz, $CDCl_3$)

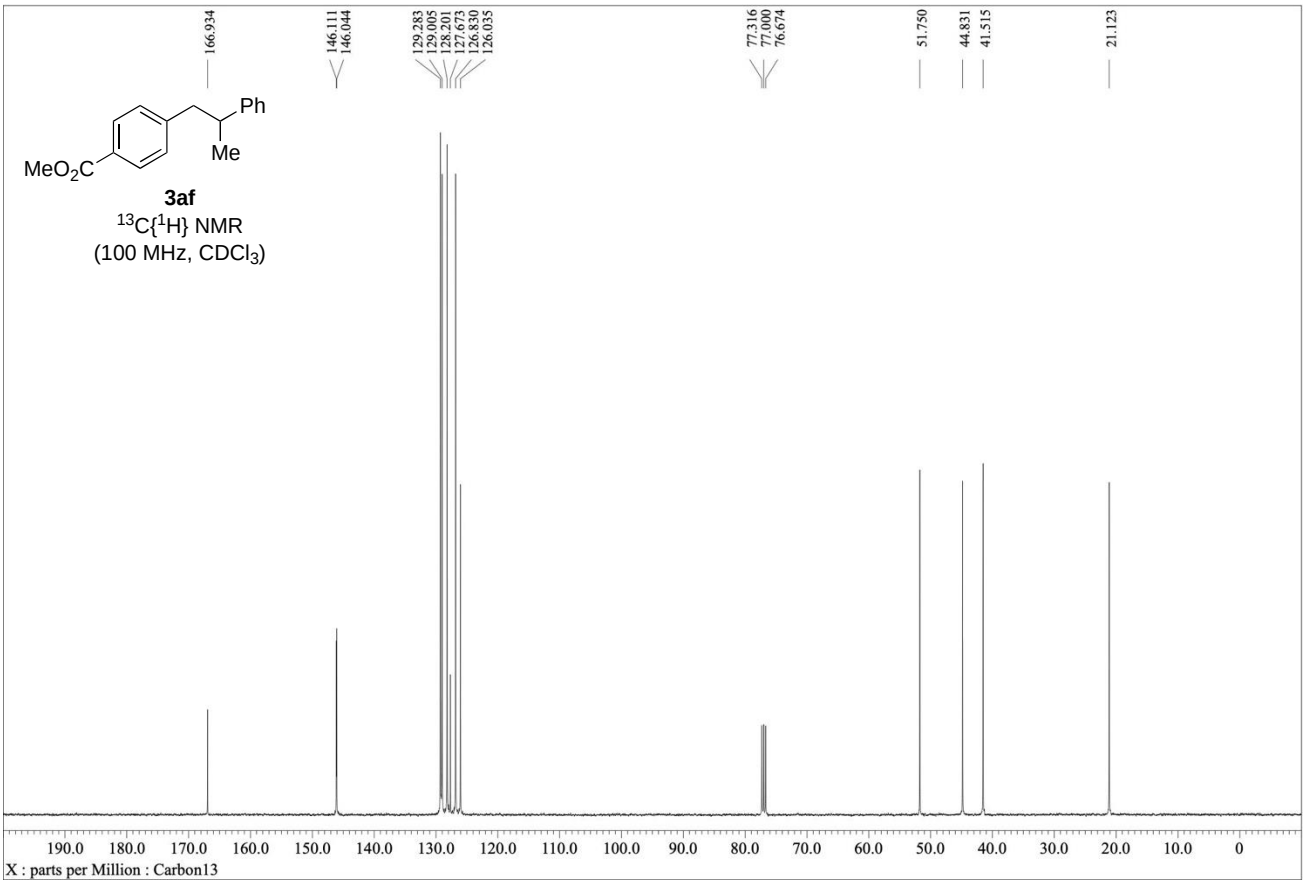


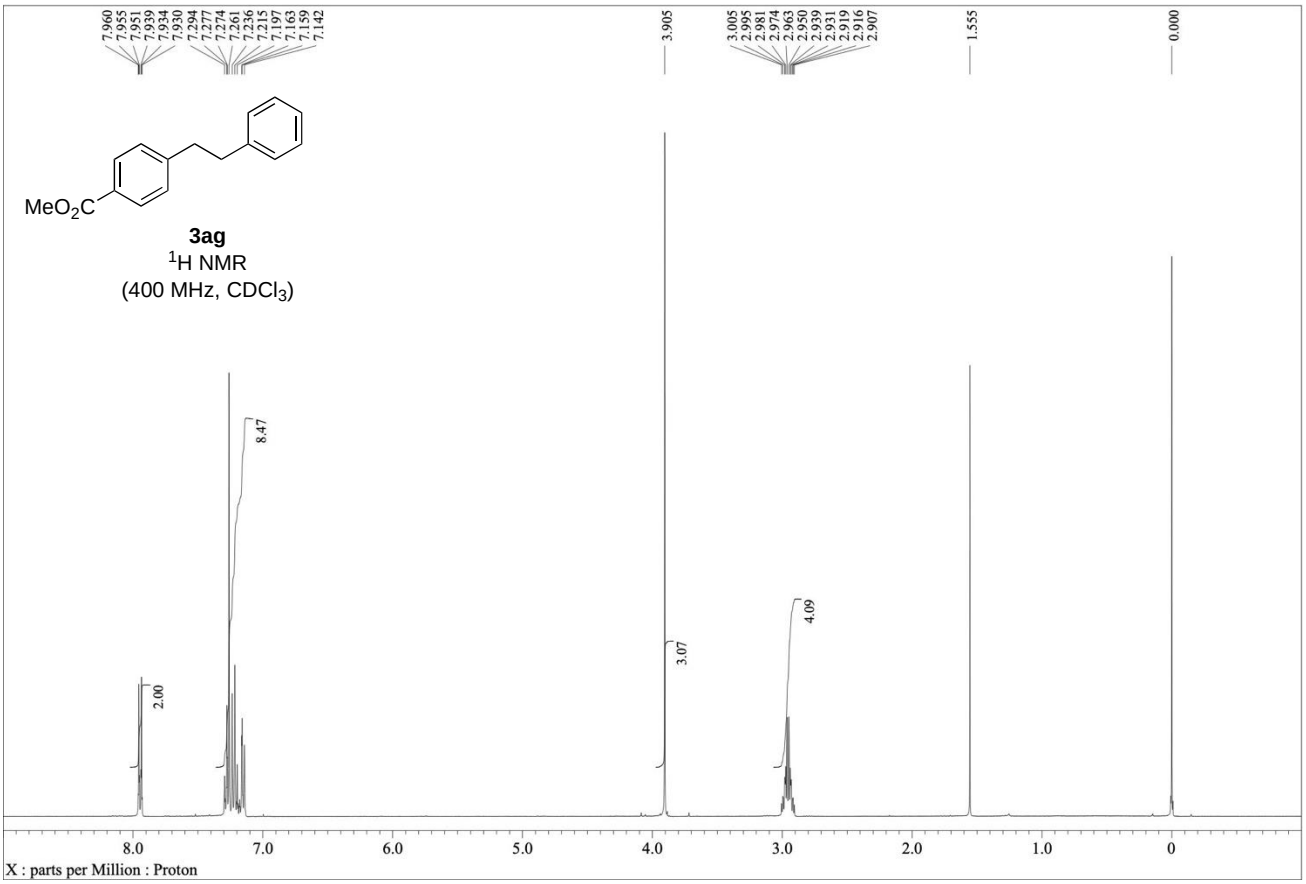
3ad
$^{13}C\{^{1}H\}$ NMR
(100 MHz, $CDCl_3$)

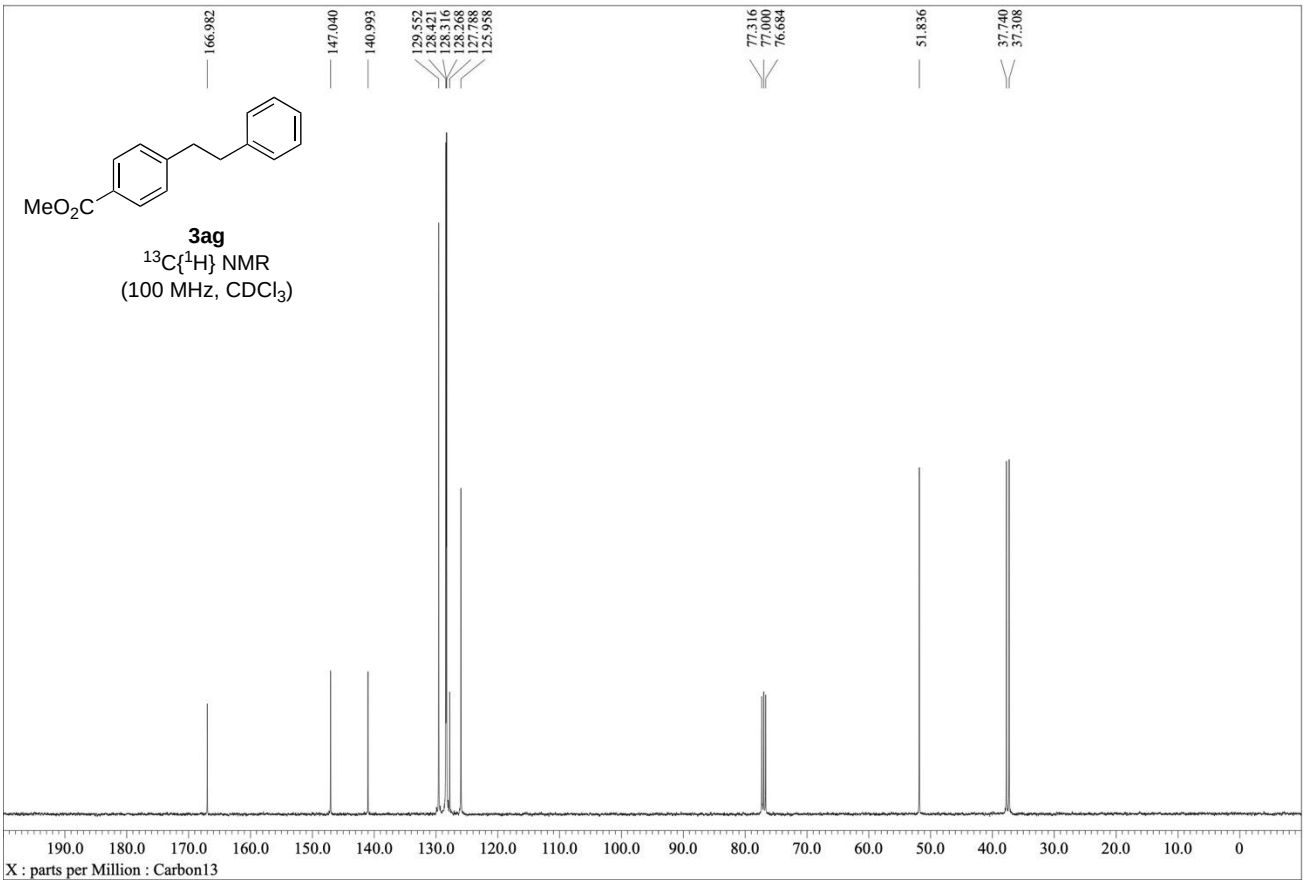

7.850
7.845
7.841
7.829
7.825
7.820
7.260
7.249
7.247
7.245
7.229
7.198
7.194
7.181
7.176
7.174
7.070
4.248
4.229
4.209
3.871
3.418
3.399
1.558
0.000
Ph
Ph
MeO2C
3ae
1H NMR
(400 MHz, CDCl3)
6.00
5.39
2.00
2.00
1.00
3.00
2.00
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
X : parts per Million : Proton

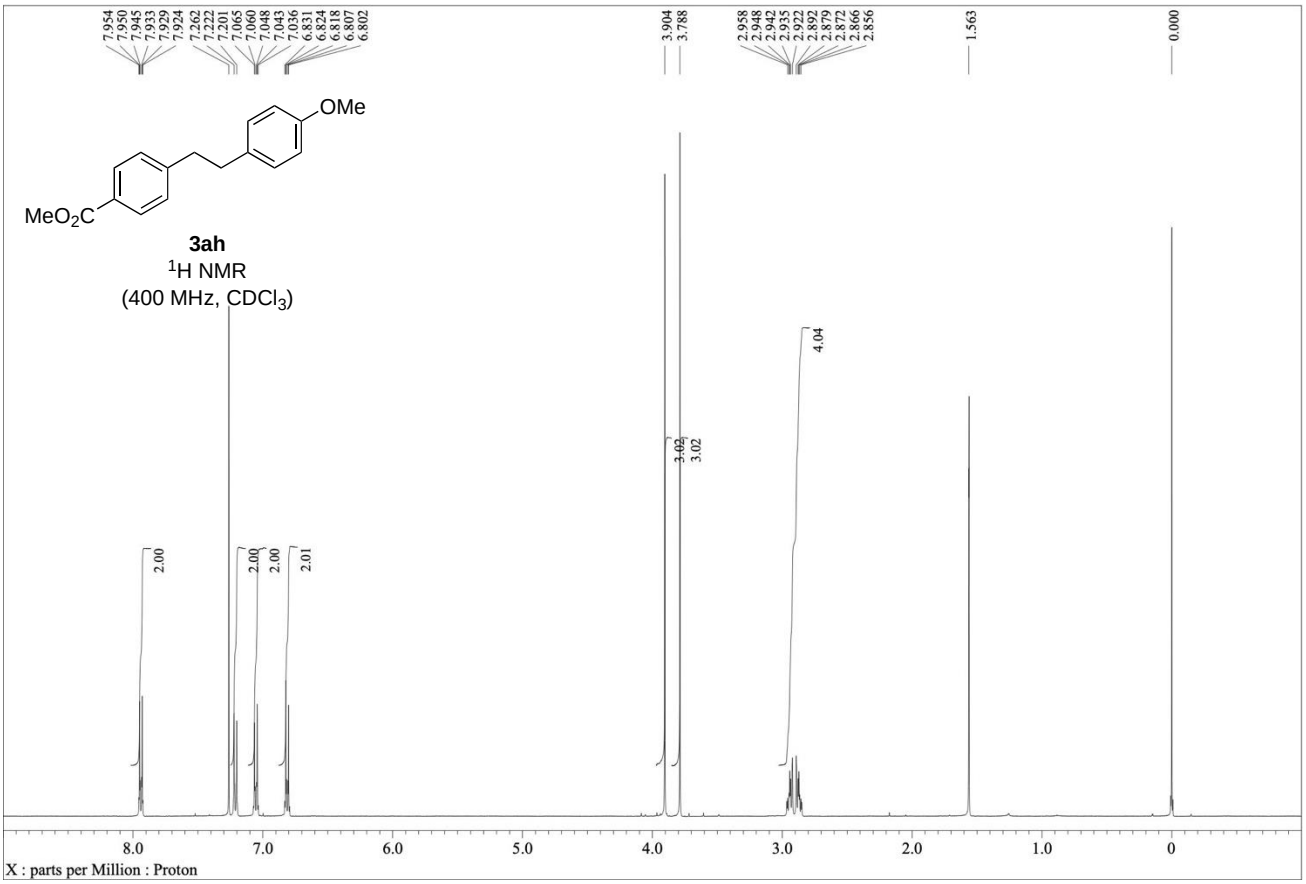

166.896
145.641
143.792
129.274
128.986
128.277
127.798
127.721
126.217
77.316
77.000
76.684
52.650
51.759
41.937
Ph
Ph
MeO2C
3ae
13C{1H} NMR
(100 MHz, CDCl3)
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
X : parts per Million : Carbon13

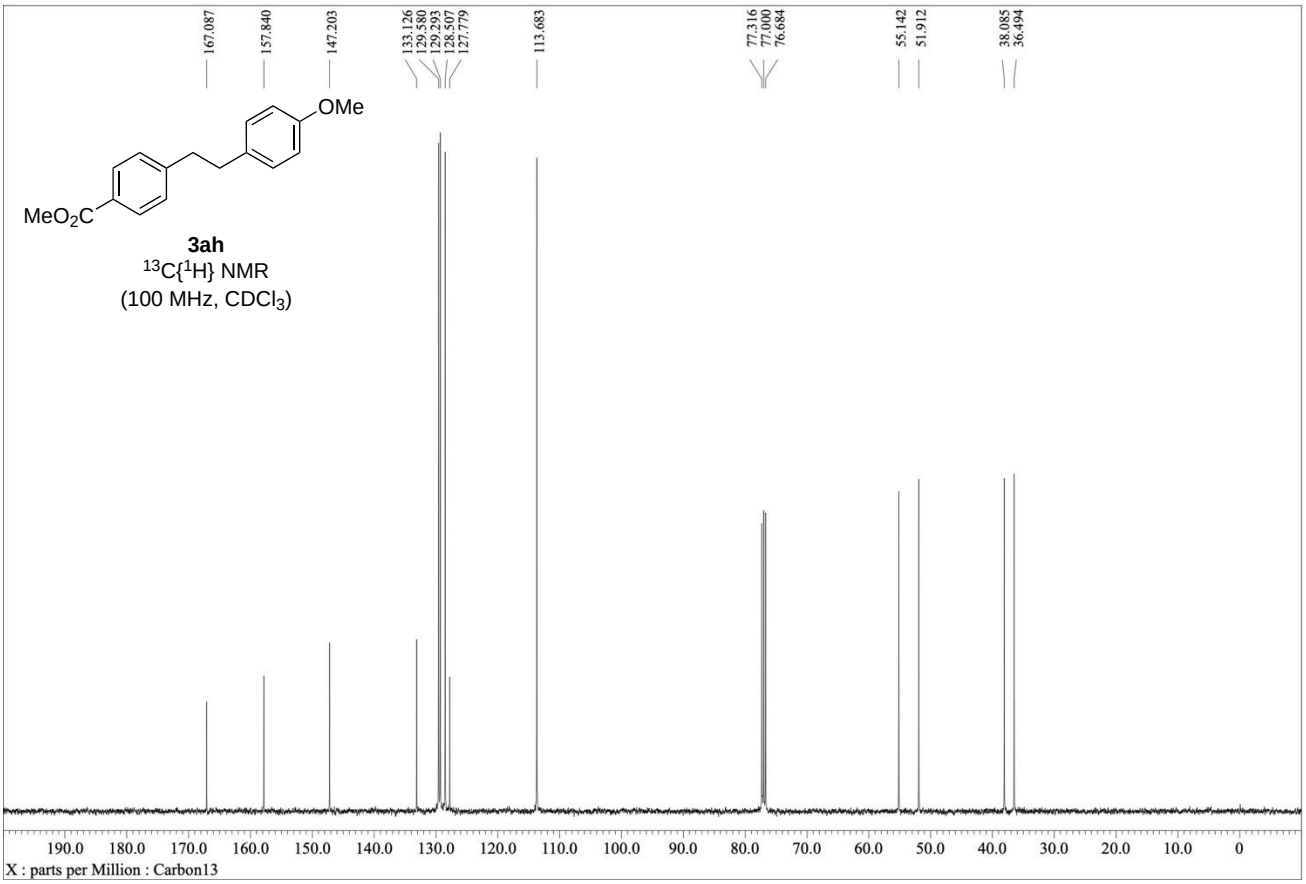

7.902
7.882
7.268
7.261
7.249
7.197
7.179
7.157
7.154
7.136
7.127
7.107
3.889
3.047
3.030
3.012
2.995
2.989
2.956
2.939
2.876
2.857
2.844
2.825
1.554
1.267
1.250
0.000
Ph
Me
MeO2C
3af
1H NMR
(400 MHz, CDCl3)
5.09
4.00
3.04
3.14
2.06
2.00
1.03
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
X : parts per Million : Proton

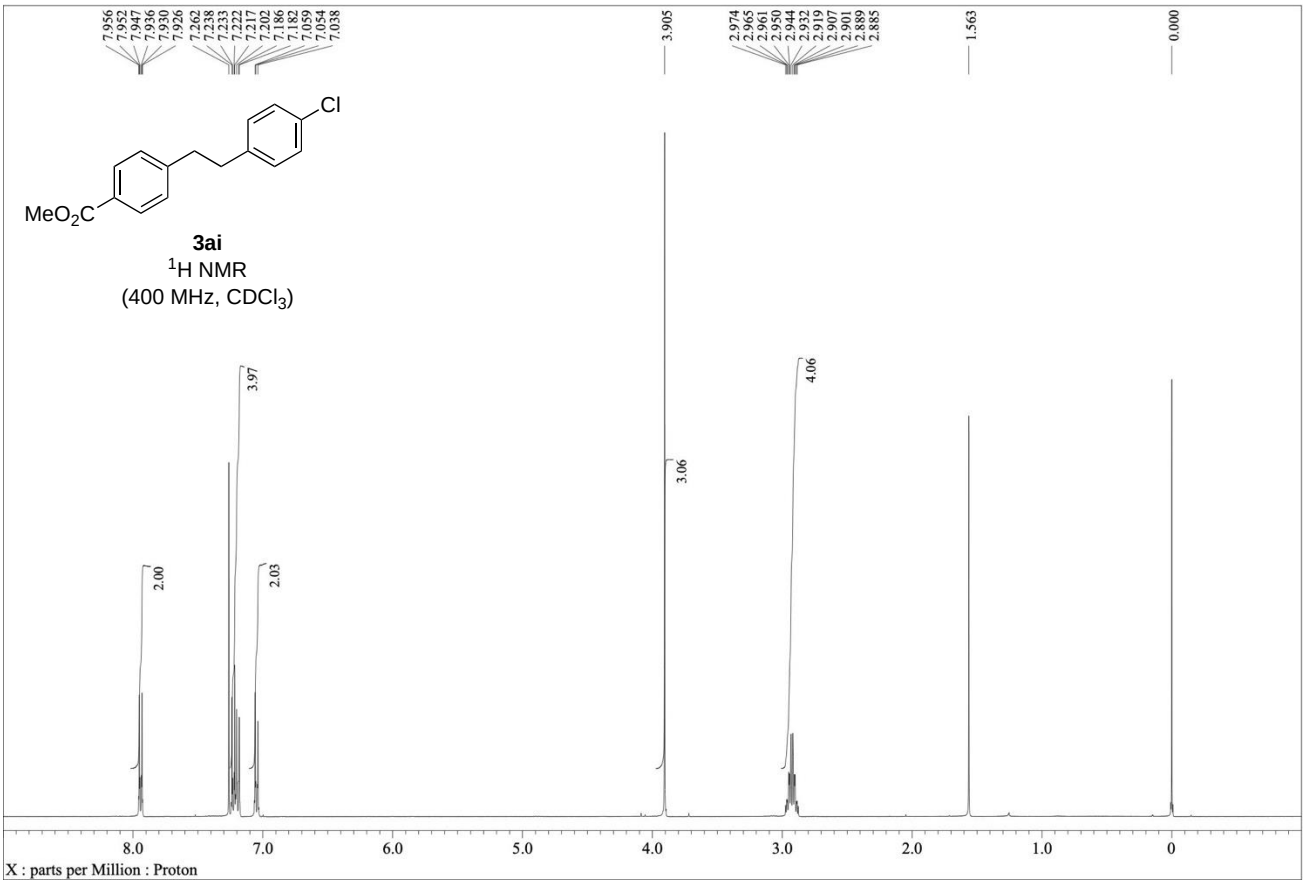

166.934
146.111
146.044
129.283
129.005
128.201
127.673
126.830
126.035
77.316
77.000
76.674
51.750
44.831
41.515
21.123
Ph
Me
MeO2C
3af
13C{1H} NMR
(100 MHz, CDCl3)
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
X : parts per Million : Carbon13

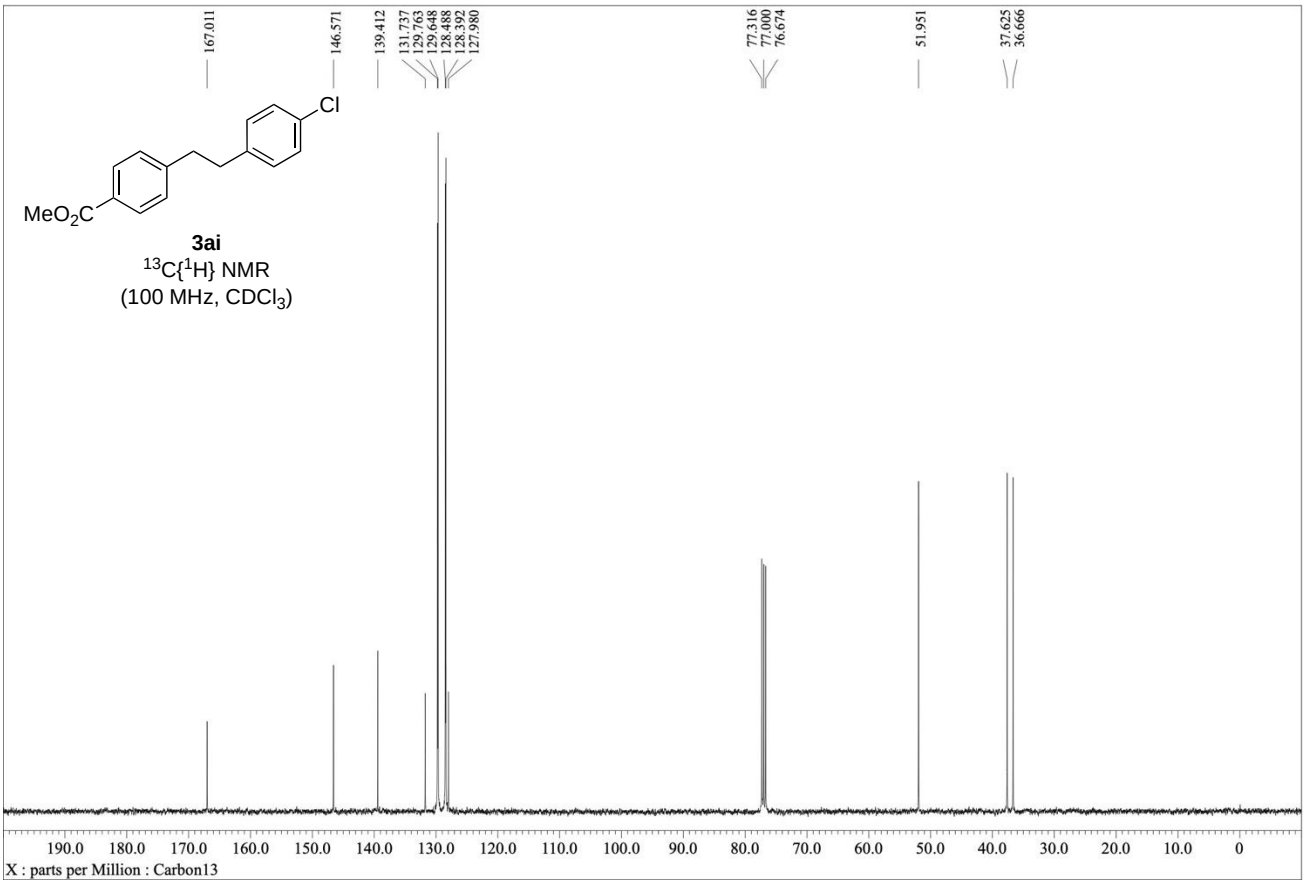

7.960
7.955
7.951
7.939
7.934
7.930
7.294
7.277
7.274
7.261
7.236
7.215
7.197
7.163
7.159
7.142
3.905
3.005
2.995
2.981
2.974
2.963
2.950
2.939
2.931
2.919
2.916
2.907
1.555
0.000
MeO2C
3ag
1H NMR
(400 MHz, CDCl3)
8.47
4.09
3.07
2.00
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
X : parts per Million : Proton

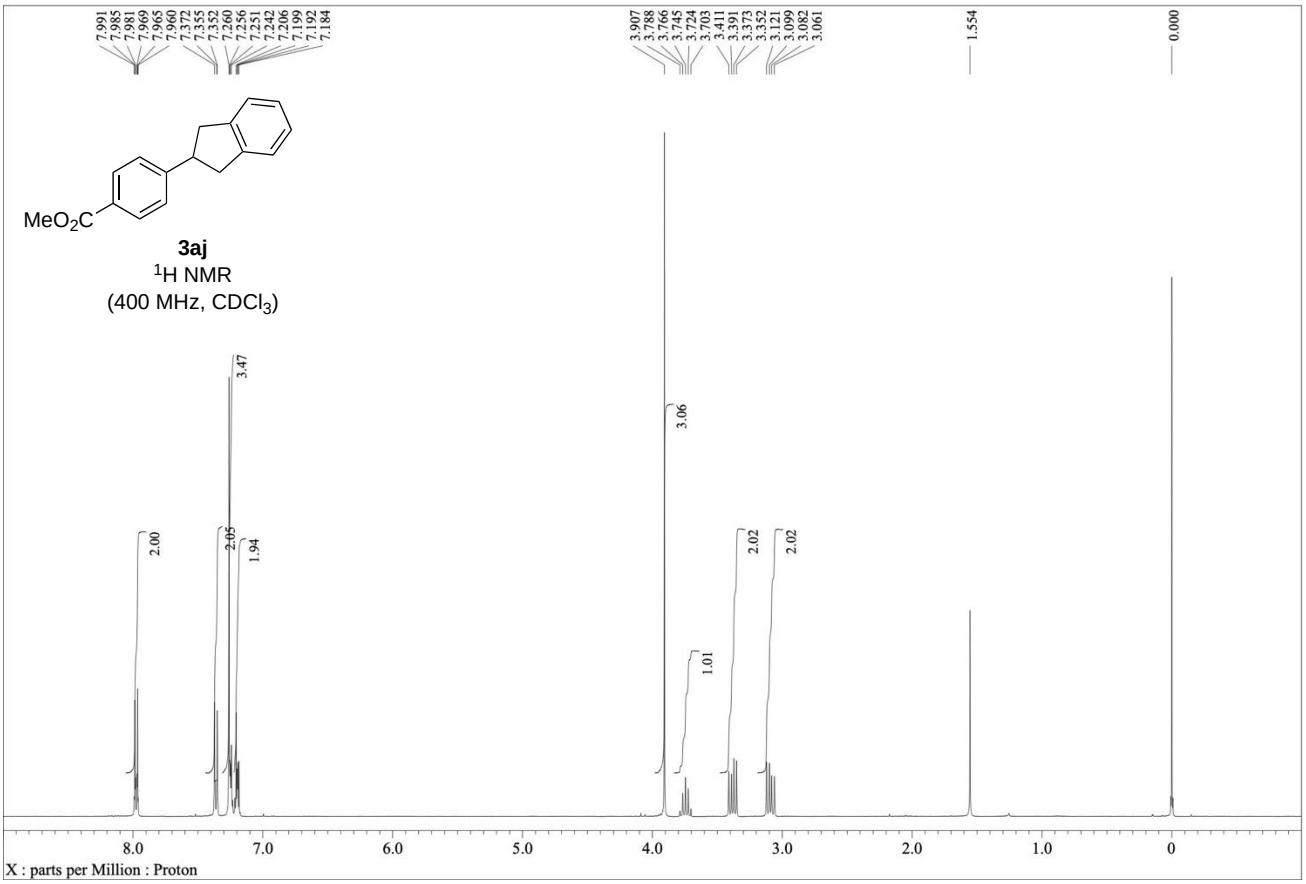

166.982
147.040
140.993
129.552
128.421
128.316
128.268
127.788
125.958
77.316
77.000
76.684
51.836
37.740
37.308
MeO2C
3ag
13C{1H} NMR
(100 MHz, CDCl3)
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
X : parts per Million : Carbon13


7.954
7.950
7.945
7.933
7.929
7.924
7.262
7.222
7.201
7.065
7.060
7.048
7.043
7.036
6.831
6.824
6.818
6.807
6.802
3.904
3.788
2.958
2.948
2.942
2.935
2.922
2.892
2.879
2.872
2.866
2.856
1.563
0.000
OMe
MeO2C
3ah
1H NMR
(400 MHz, CDCl3)
4.04
3.02
3.02
2.00
2.00
2.00
2.01
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
X : parts per Million : Proton

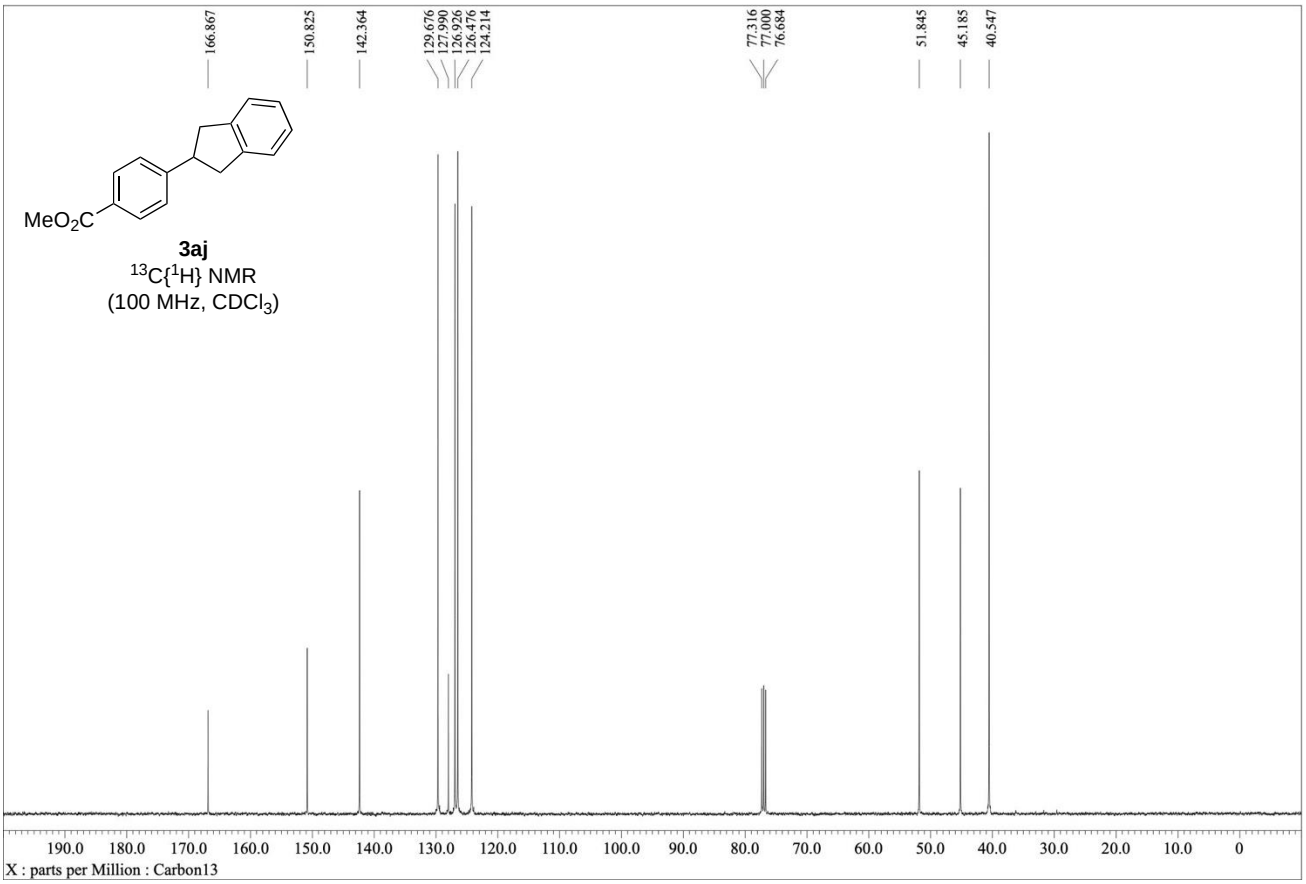

167.087
157.840
147.203
133.126
129.580
129.293
128.507
127.779
113.683
77.316
77.000
76.684
55.142
51.912
38.085
36.494
OMe
MeO2C
3ah
13C{1H} NMR
(100 MHz, CDCl3)
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
X : parts per Million : Carbon13


7.956
7.952
7.947
7.936
7.930
7.926
7.262
7.238
7.233
7.222
7.217
7.202
7.186
7.182
7.059
7.054
7.038
3.905
2.974
2.965
2.961
2.950
2.944
2.932
2.919
2.907
2.901
2.889
2.885
1.563
0.000
Cl
MeO2C
3ai
1H NMR
(400 MHz, CDCl3)
3.97
4.06
3.06
2.00
2.03
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
X : parts per Million : Proton

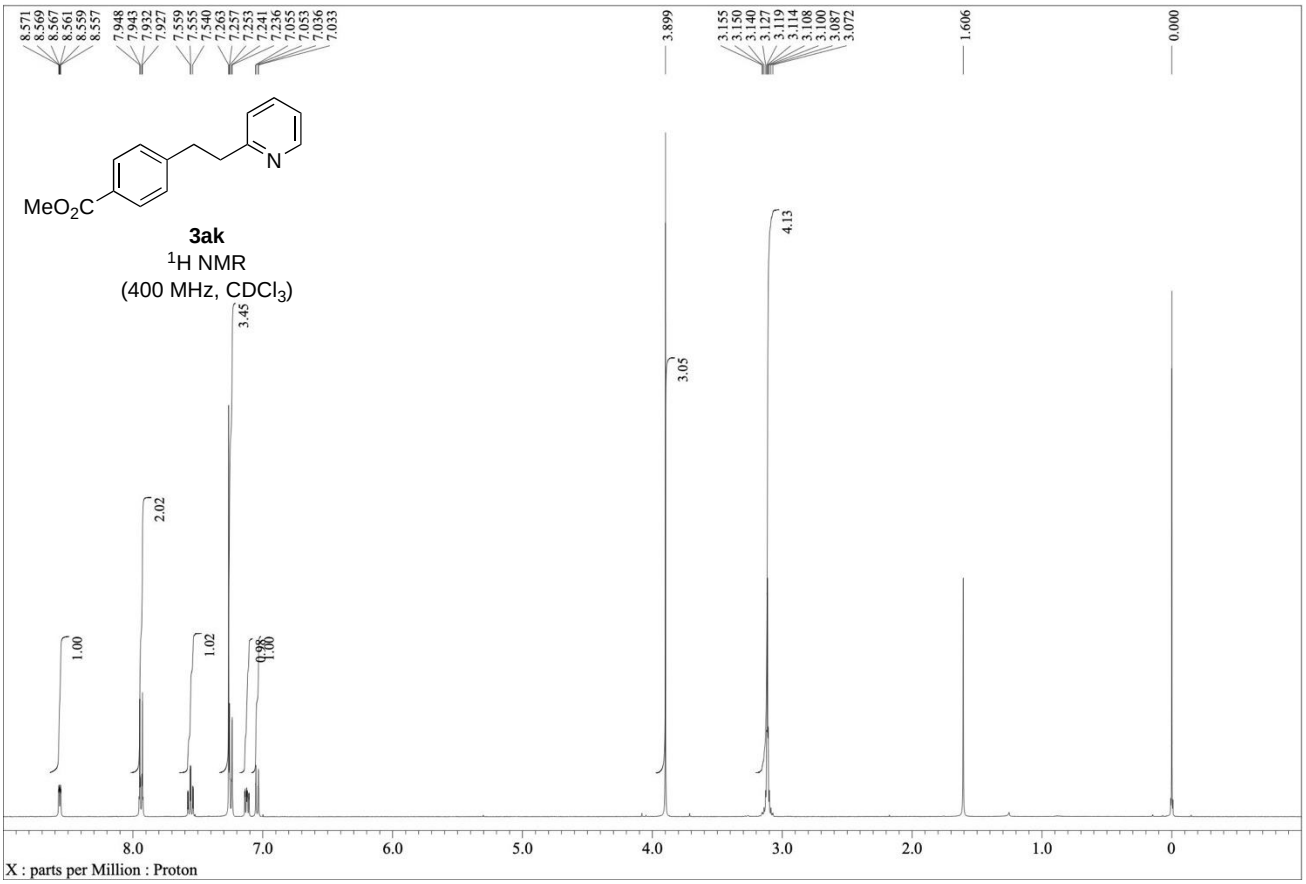

167.011
146.571
139.412
131.737
129.763
129.648
128.488
128.392
127.980
77.316
77.000
76.674
51.951
37.625
36.666
Cl
MeO2C
3ai
13C{1H} NMR
(100 MHz, CDCl3)
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
X : parts per Million : Carbon13

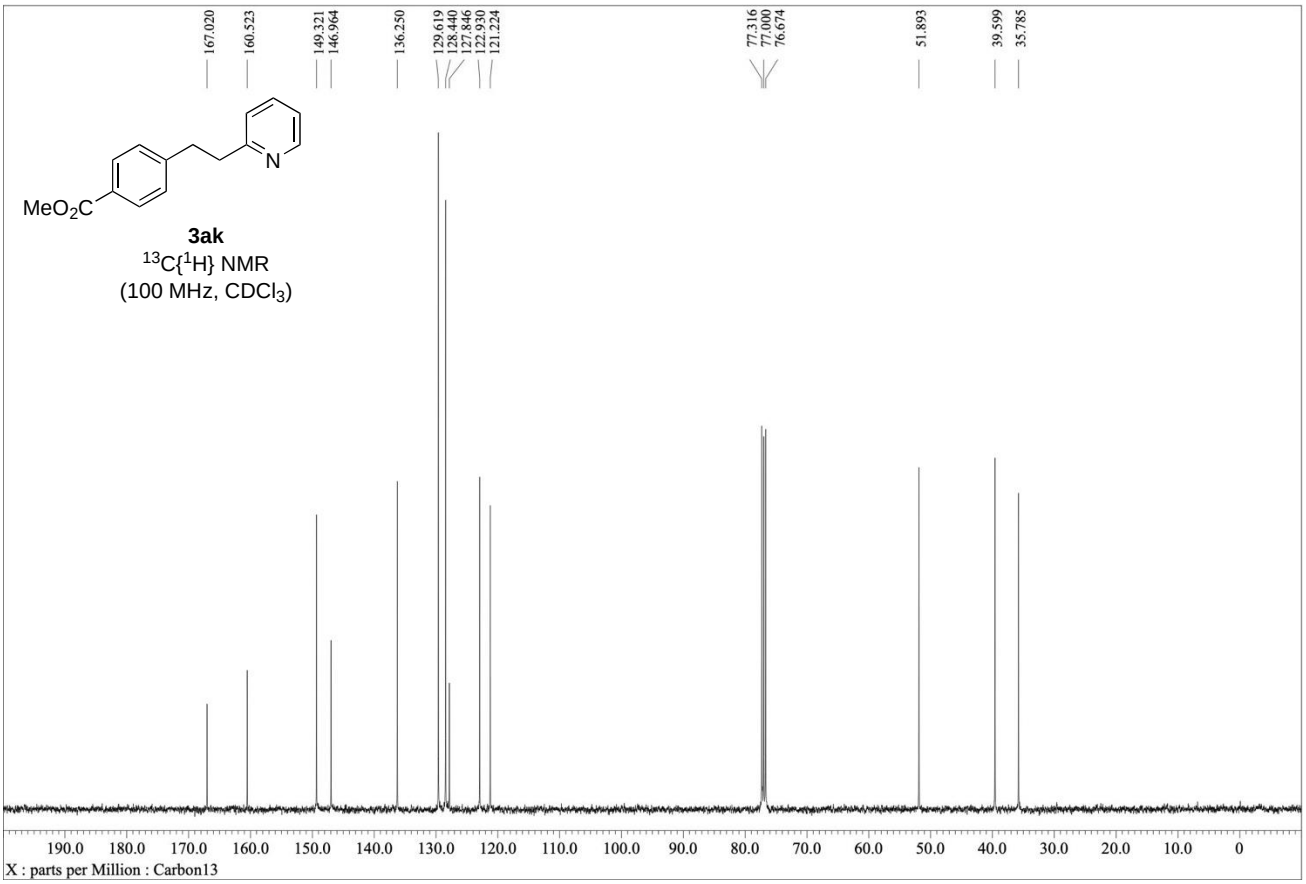


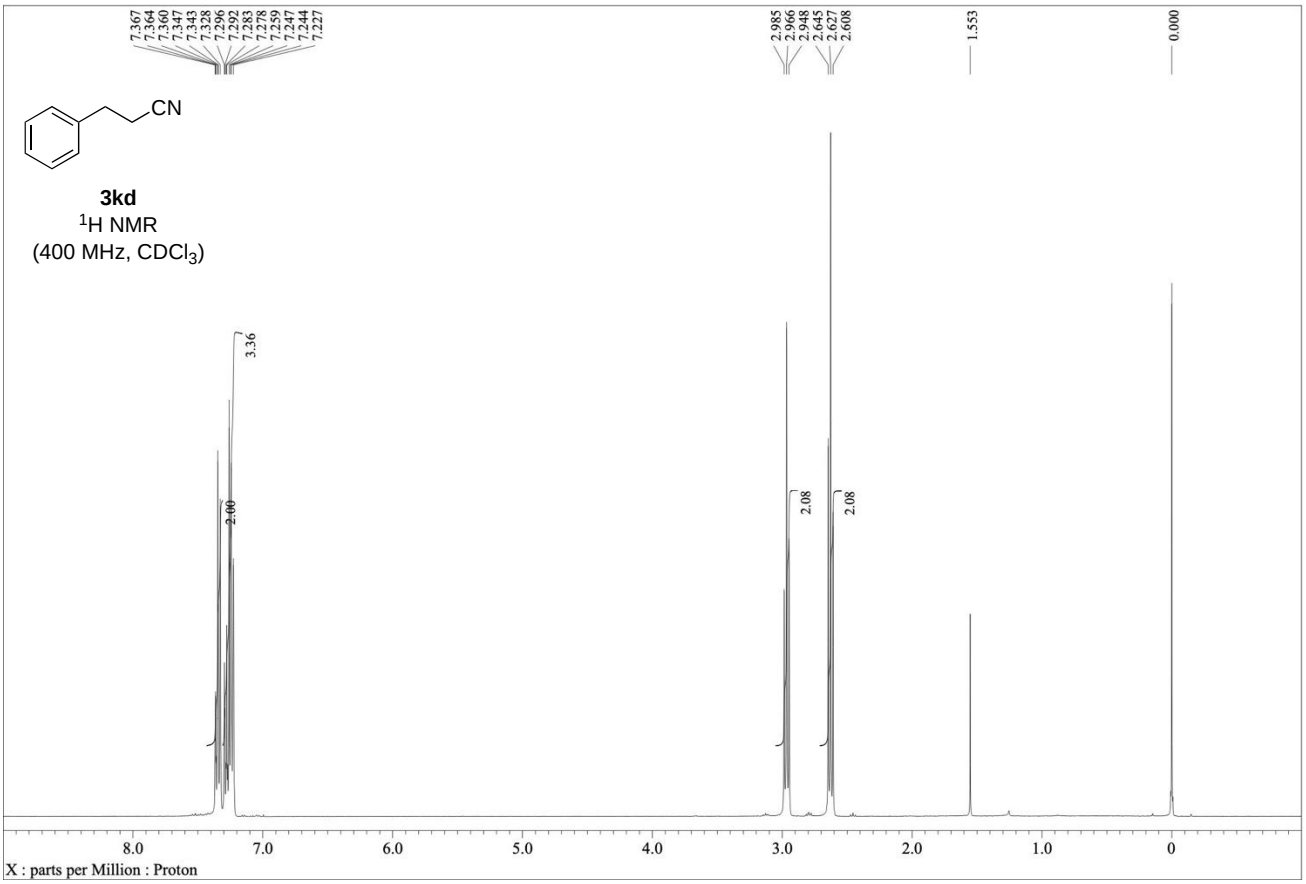
3aj
^{1}H NMR
(400 MHz, $CDCl_3$)

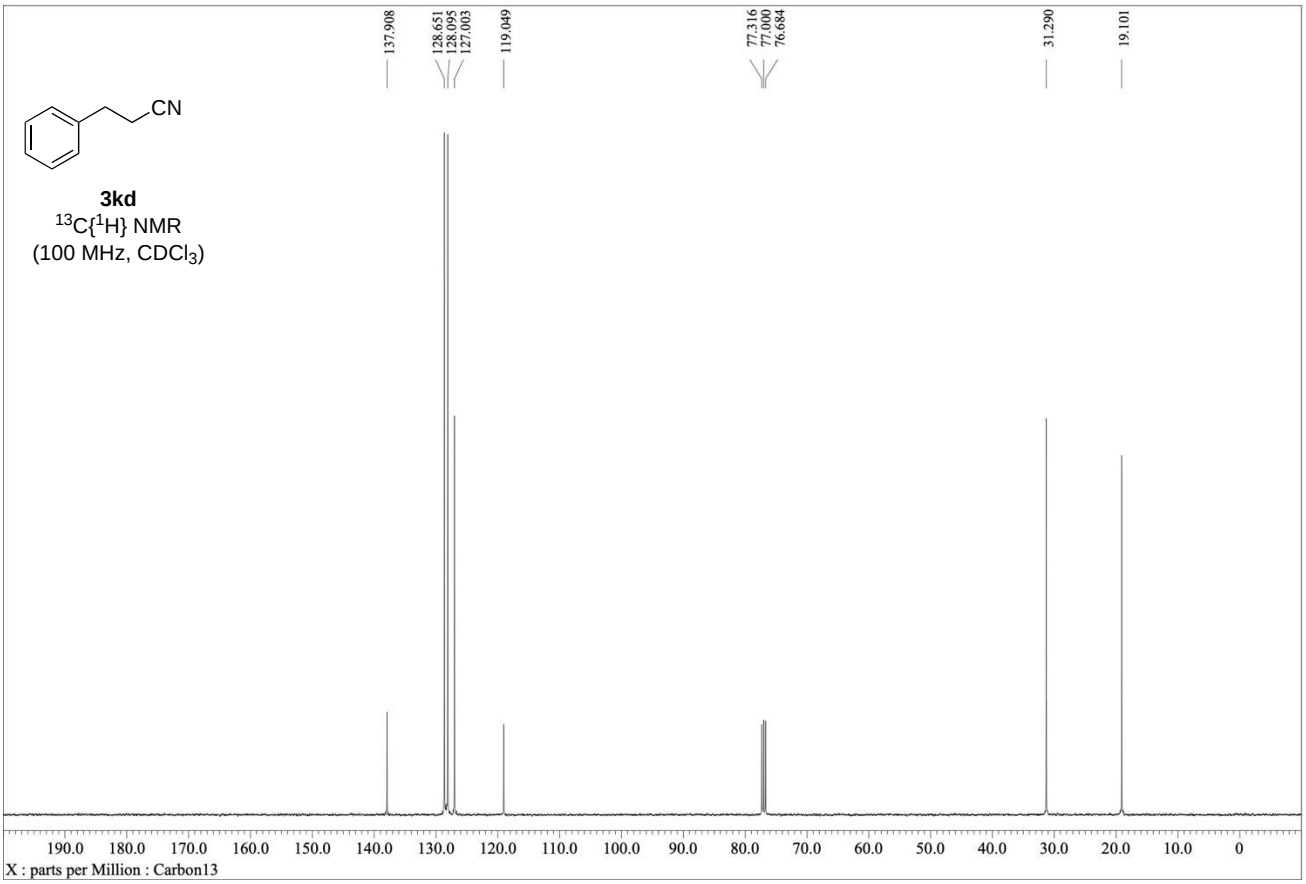


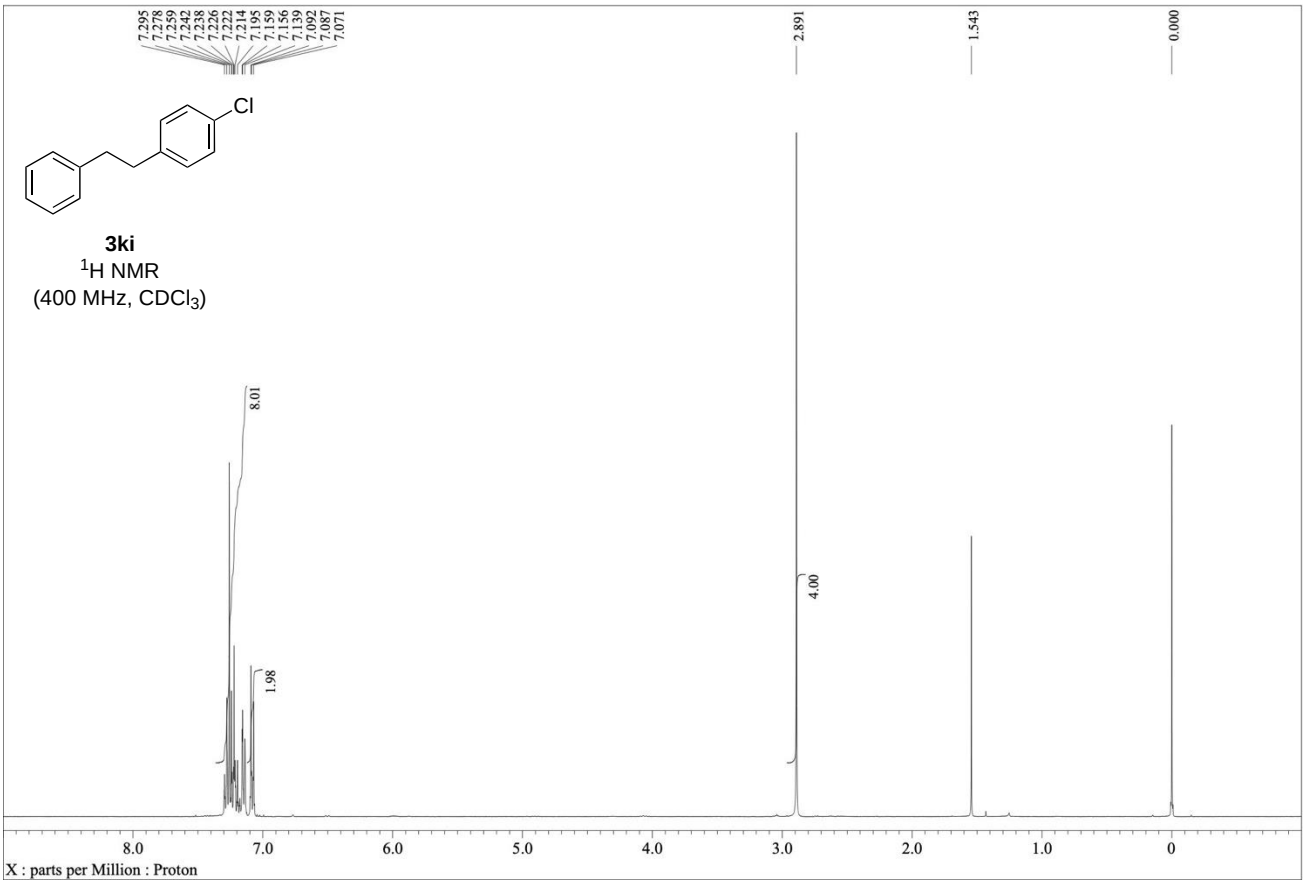
3aj
$^{13}C\{^{1}H\}$ NMR
(100 MHz, $CDCl_3$)

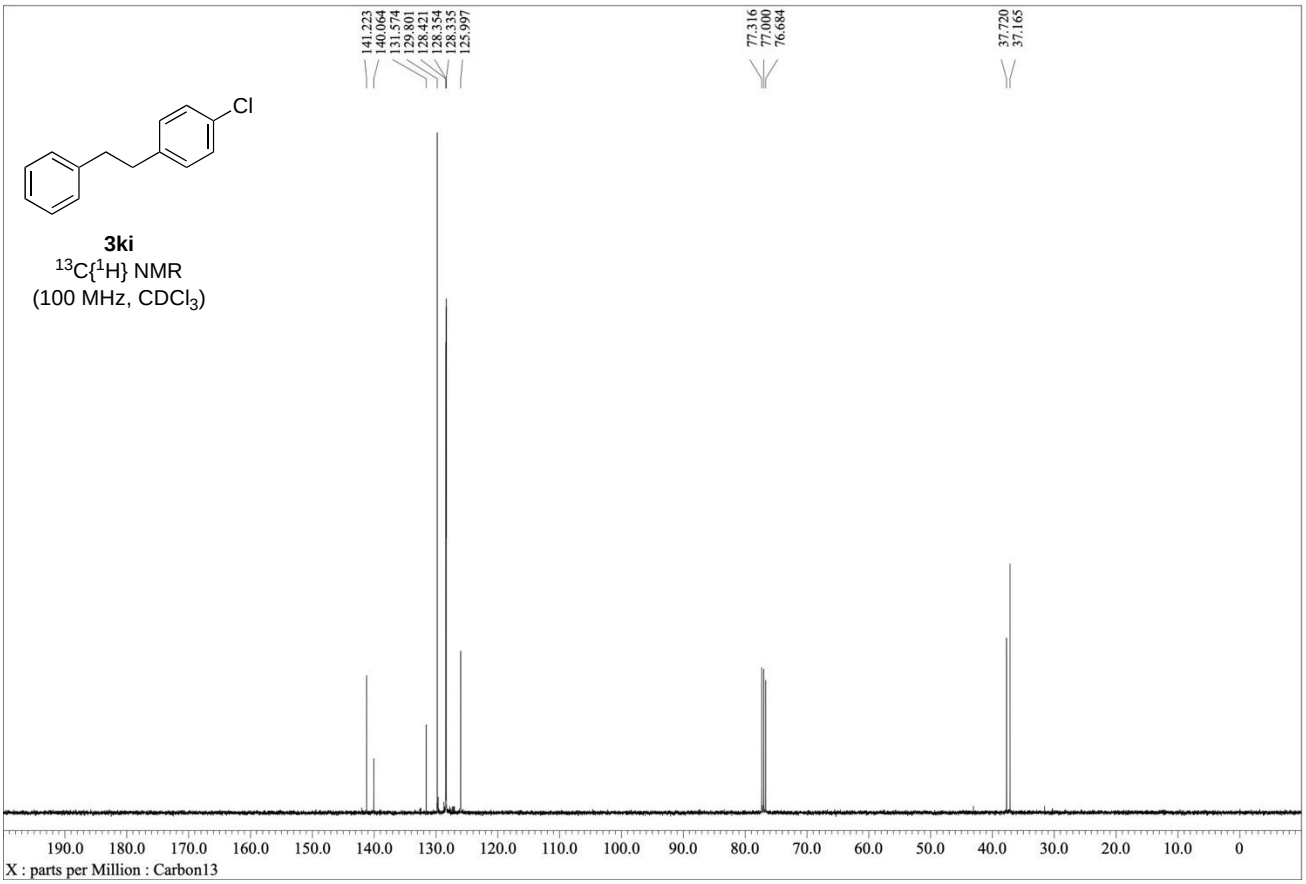

8.571
8.569
8.567
8.561
8.559
8.557
7.948
7.943
7.932
7.927
7.559
7.555
7.540
7.263
7.257
7.253
7.241
7.236
7.055
7.053
7.036
7.033
3.899
3.155
3.150
3.140
3.127
3.119
3.114
3.108
3.100
3.087
3.072
1.606
0.000
N
MeO2C
3ak
1H NMR
(400 MHz, CDCl3)
3.45
4.13
3.05
2.02
1.00
1.02
0.98
1.00
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
X : parts per Million : Proton

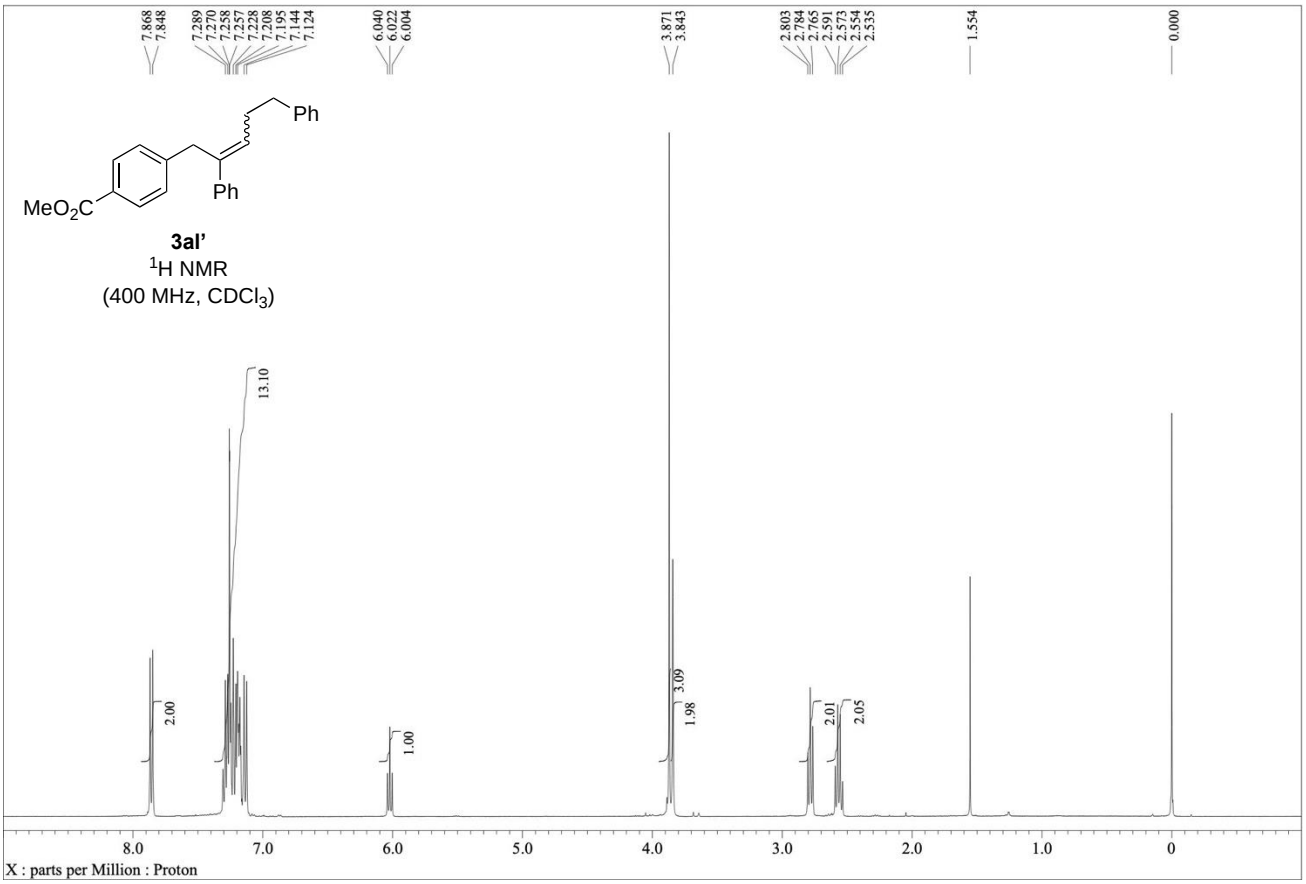

167.020
160.523
149.321
146.964
136.250
129.619
128.440
127.846
122.930
121.224
77.316
77.000
76.674
51.893
39.599
35.785
N
MeO2C
3ak
13C{1H} NMR
(100 MHz, CDCl3)
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
X : parts per Million : Carbon13


7.367
7.364
7.360
7.347
7.343
7.328
7.296
7.292
7.283
7.278
7.259
7.247
7.244
7.227
2.985
2.966
2.948
2.645
2.627
2.608
1.553
0.000
CN
3kd
1H NMR
(400 MHz, CDCl3)
3.36
2.00
2.08
2.08
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
X : parts per Million : Proton

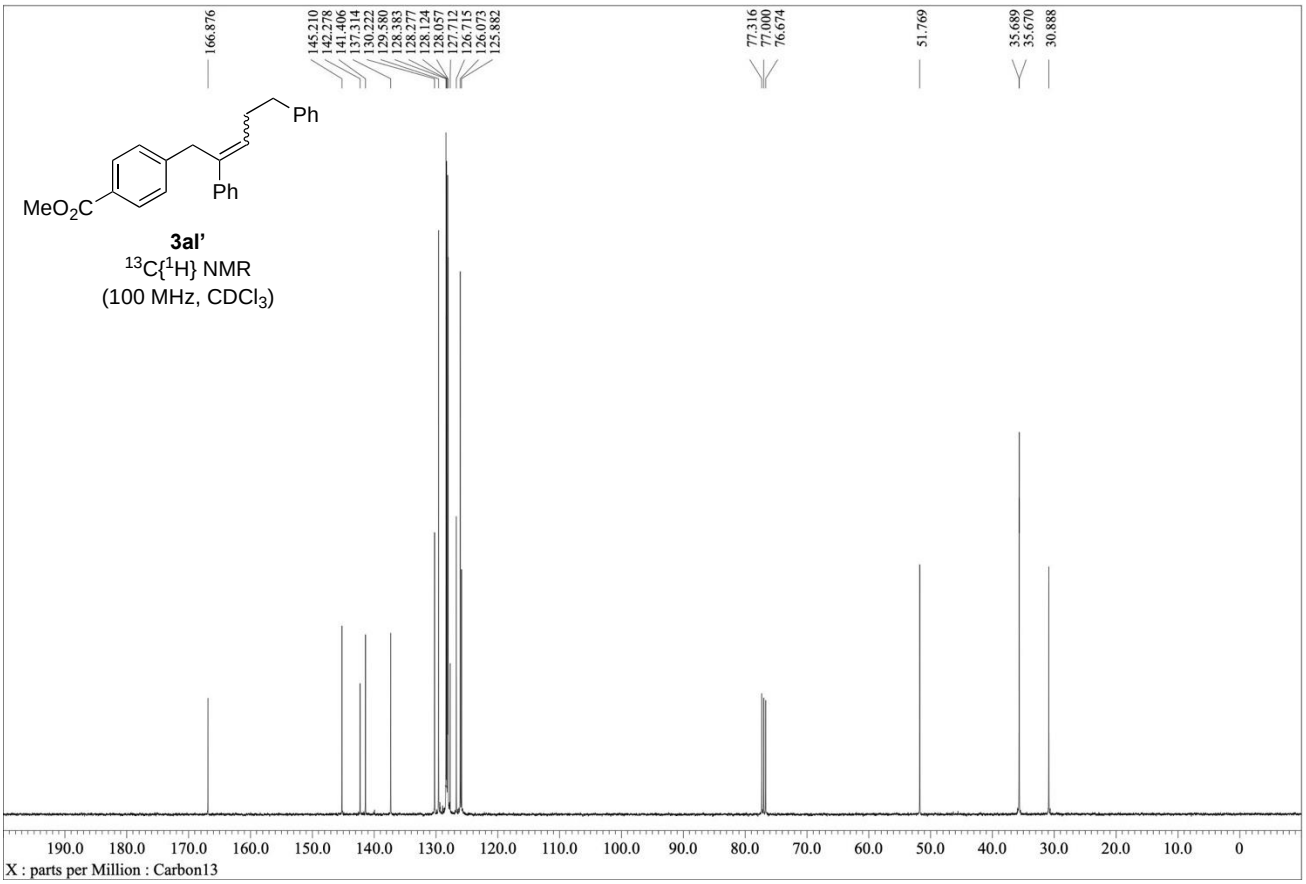

137.908
128.651
128.095
127.003
119.049
77.316
77.000
76.684
31.290
19.101
CN
3kd
13C{1H} NMR
(100 MHz, CDCl3)
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
X : parts per Million : Carbon13


7.295
7.278
7.259
7.242
7.238
7.226
7.222
7.214
7.195
7.159
7.156
7.139
7.092
7.087
7.071
2.891
1.543
0.000
Cl
3ki
1H NMR
(400 MHz, CDCl3)
8.01
4.00
1.98
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
X : parts per Million : Proton

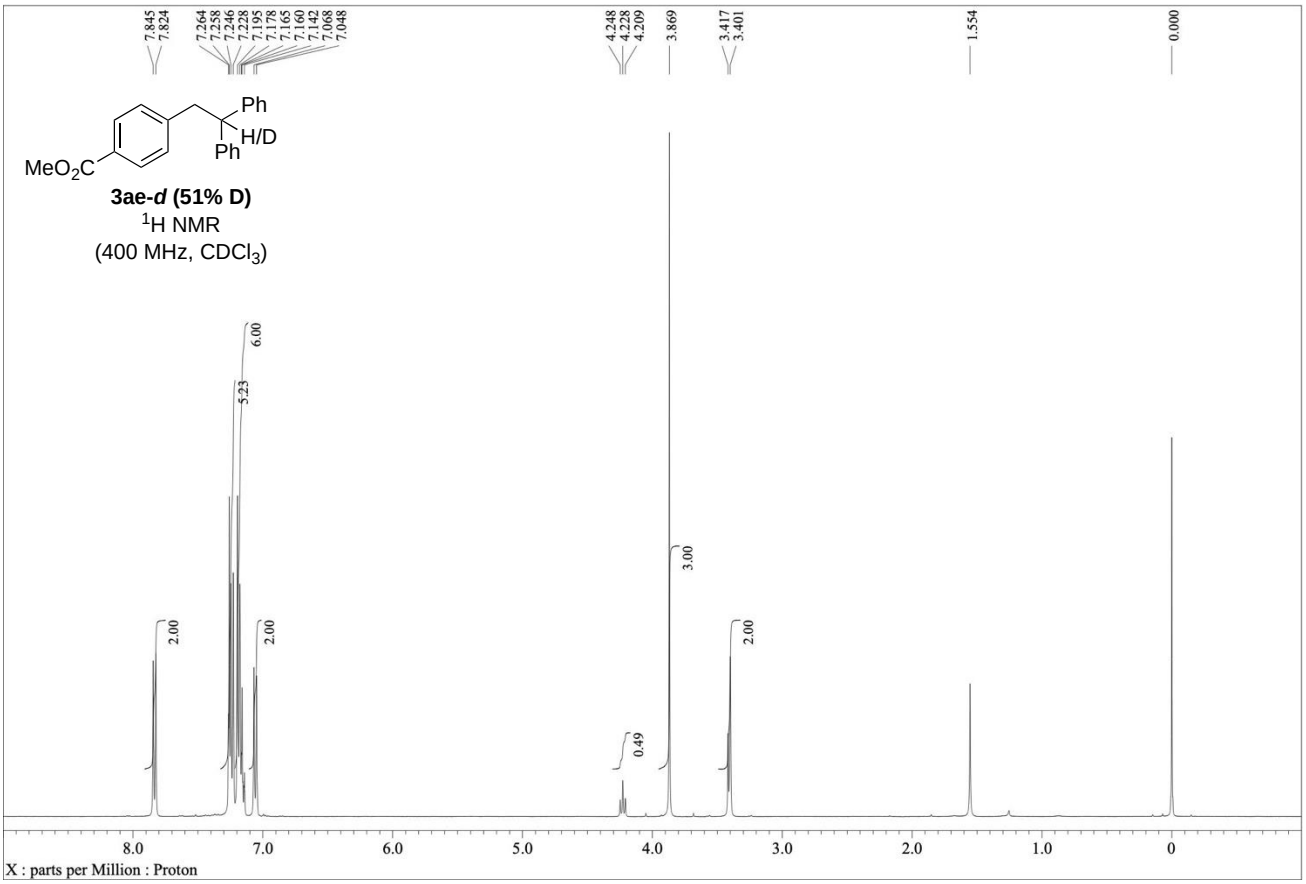

141.223
140.064
131.574
129.801
128.421
128.354
128.335
125.997
77.316
77.000
76.684
37.720
37.165
Cl
3ki
13C{1H} NMR
(100 MHz, CDCl3)
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
X : parts per Million : Carbon13

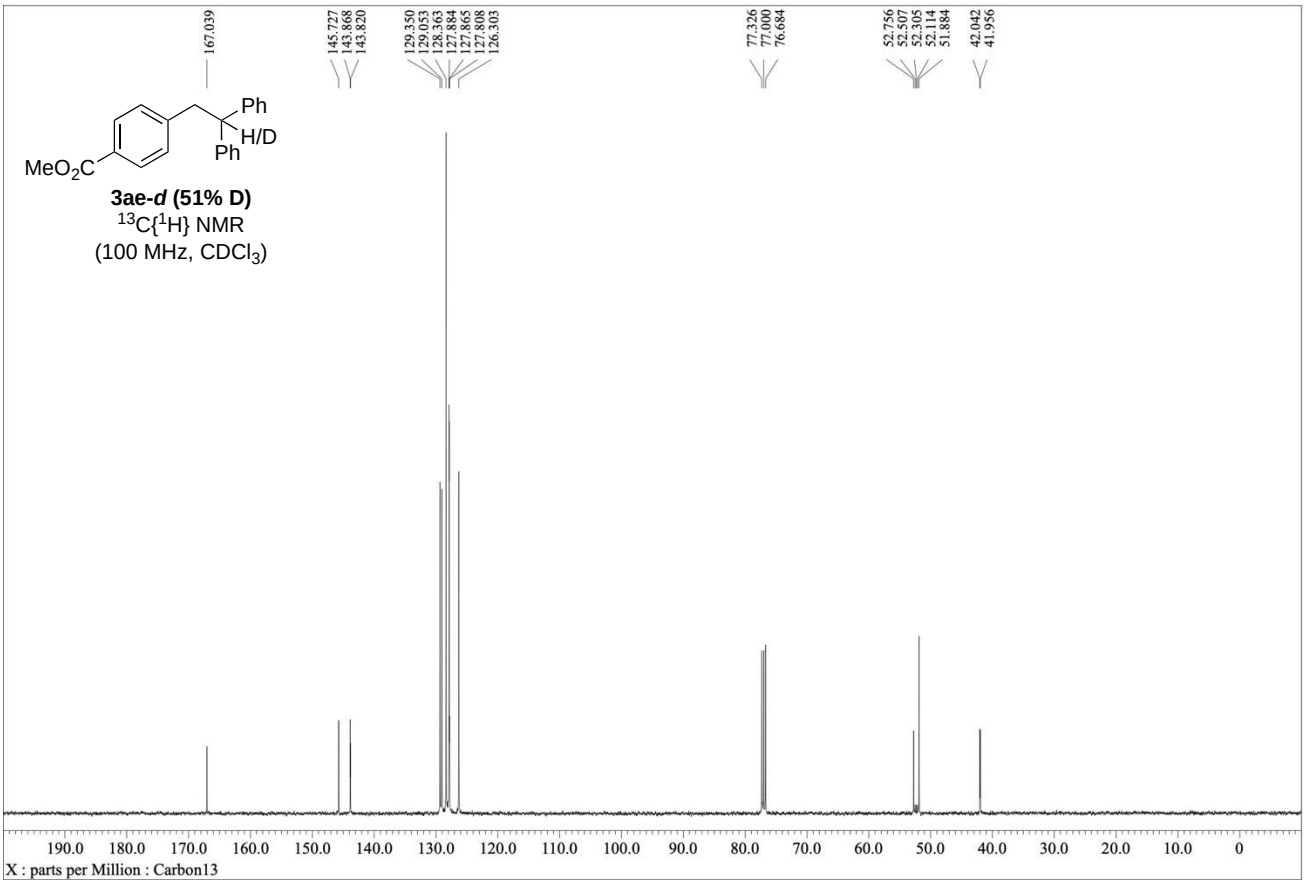


3al'
^{1}H NMR
(400 MHz, $CDCl_3$)



3al'
$^{13}C\{^{1}H\}$ NMR
(100 MHz, $CDCl_3$)


7.845
7.824
7.264
7.258
7.246
7.228
7.195
7.178
7.165
7.160
7.142
7.068
7.048
4.248
4.228
4.209
3.869
3.417
3.401
1.554
0.000
Ph
H/D
Ph
MeO2C
3ae-d (51% D)
1H NMR
(400 MHz, CDCl3)
6.00
5.23
2.00
2.00
0.49
3.00
2.00
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
X : parts per Million : Proton


167.039
145.727
143.868
143.820
129.350
129.053
128.363
127.884
127.865
127.808
126.303
77.326
77.000
76.684
52.756
52.507
52.305
52.114
51.884
42.042
41.956
Ph
H/D
Ph
MeO2C
3ae-d (51% D)
13C{1H} NMR
(100 MHz, CDCl3)
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
X : parts per Million : Carbon13