Table of Contents

General Methods

^{1}H-NMR spectra were recorded on Varian 400 (400 MHz) spectrometer. Chemical shifts are reported in ppm from TMS with the solvent resonance as the internal standard (deuterochloroform: 7.24 ppm). Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, dd= doublet-doublet, t = triplet, td = triple doublet, dt = double triplet, q = quartet, sext = sextet, sept = septet, p = pseudo, b = broad, m = multiplet), coupling constants (Hz). ^{13}C-NMR spectra were recorded on a Varian 400 (100 MHz) spectrometer with complete proton decoupling. Chemical shifts are reported in ppm from TMS with the solvent as the internal standard (deuterochloroform: 77.0 ppm).

Chromatographic purification was done with 240-400 mesh silica gel. Other anhydrous solvents were supplied by Sigma Aldrich in Sureseal® bottles and used without any further purification. Commercially available chemicals were purchased from Sigma Aldrich, Stream and TCI and used without any further purification.

All the starting materials were synthesized through Morita–Baylis–Hillman reaction starting from the respective benzaldehydes, following an unmodified literature procedure. Spectral data match with the ones reported.[17-21]

X-Ray Photoelectron spectroscopy (XPS): High-resolution XPS by using a Phoibos 100 hemispherical energy analyzer (Specs GmbH, Berlin, Germany), using Mg Kα radiation (ħω = 1253.6 eV; X-Ray power = 125W) in constant analyzer energy (CAE) mode, with analyzer pass energies of 10 eV. Base pressure in the analysis chamber during analysis was 2.2×10^{-8} mbar. Spectra were fitted by using CasaXPS (www.casaxps.com) after Tougaard background subtraction and all spectra were calibrated to the C1s binding energy (285.0 eV). XPS samples were prepared as tablet from the dry powder of each material and fixing it on the sample holder by conductive carbon tape. The parameters used for the fitting of C1s and N1s signal are reported in more details in our previous work.[22]

^{1}H NMR of MBH Alcohols Starting Materials 1

1a ^{1}H-NMR (400 MHz, $CDCl_3$) δ = 7.43 – 7.27 (m, 5H), 6.32 (s, 1H), 5.84 (s, 1H), 5.55 (s, 1H), 3.77 (s, 3H), 3.10 (br s, 1H). Full characterization data can be found on reference [17].

1b ^{1}H-NMR (400 MHz, $CDCl_3$) δ = 7.39 – 7.35 (m, 2H), 7.32 – 7.28 (m, 2H), 6.37 (s, 1H), 5.88 (d, J = 1.0 Hz, 1H), 5.55 (s, 1H), 3.73 (s, 3H), 2.88 (s, 1H), 1.39 (s, 9H). Full characterization data can be found on reference [17].

1c ^{1}H-NMR (400 MHz, $CDCl_3$) δ = 7.28 – 7.24 (m, 2H), 7.17 – 7.14 (m, 2H), 6.33 (s, 1H), 5.89 (s, 1H), 5.53 (s, 1H), 3.71 (s, 3H), 2.78 (s, 1H), 2.34 (s, 3H). Full characterization data can be found on reference [17].

1d ^{1}H-NMR (400 MHz, $CDCl_3$) δ = 7.46 – 7.39 (m, 1H), 7.27 – 7.15 (m, 3H), 6.32 (s, 1H), 5.81 (s, 1H), 5.60 (t, J = 1.2 Hz, 1H), 3.76 (s, 3H), 2.34 (s, 3H). The -OH signal was not detected. Full characterization data can be found on reference [17].

1e ^{1}H-NMR (400 MHz, $CDCl_3$) δ = 7.24 – 7.08 (m, 4H), 6.34 (t, J = 0.9 Hz, 1H), 5.85 (t, J = 1.2 Hz, 1H), 5.54 (s, 1H), 3.74 (s, 3H), 2.28 (s, 3H). The -OH signal was not detected. Full characterization data can be found on reference [17].

1f ^{1}H-NMR (400 MHz, $CDCl_3$) δ = 7.27 – 7.24 (m, 1H), 6.96 – 6.-92 (m, 2H), 6.82 (dd, *J* = 8.3, 3.5 Hz, 1H), 6.31 (s, 1H), 5.82 (s, 1H), 5.55 (s, 1H), 3.80 (s, 3H), 3.73 (s 3H), 3.52 (s, 1H). Full characterization data can be found on reference [18].

1g ^{1}H-NMR (400 MHz, $CDCl_3$) δ = 7.54 (d, J = 7.6 Hz, 1H), 7.37 – 7.24 (m, 3H) 6.33 (s, 1H), 6.00 (d, J = 4.8 Hz, 1H), 5.61 (s, 1H), 3.78 (s, 3H), 3.55 (s, 1H). Full characterization data can be found on reference [19].

1h ^{1}H-NMR (400 MHz, $CDCl_3$) δ = 7.39 – 7.31 (m, 2H), 7.09 – 6.99 (m, 2H), 6.32 (s, 1H), 5.80 (t, J = 0.9 Hz, 1H), 5.55 (d, J = 3.6 Hz, 1H), 3.71 (s, 3H), 3.03 (s, 1H). Full characterization data can be found on reference [17].

1i ^{1}H-NMR (400 MHz, $CDCl_3$) δ = 7.31 (*pseudo*s, 4H), 6.35 (s, 1H), 5.82 (s, 1H), 5.54 (s, 1H), 3.73 (s, 3H), 2.88 (s, 1H). Full characterization data can be found on reference [17].

1j ^{1}H-NMR (400 MHz, $CDCl_3$) δ = 7.49 – 7.45 (m, 2H), 7.28 – 7.24 (m, 2H), 6.34 (s, 1H), 5.82 (t, J = 0.9 Hz, 1H), 5.49 (d, J = 5.3 Hz, 1H), 3.73 (s, 3H), 3.18 (s, 1H). Full characterization data can be found on reference [17].

1k ^{1}H-NMR (400 MHz, $CDCl_3$) δ = 7.55 – 7.51 (m, 1H), 7.42 (dd, J = 7.8, 2.0, 1H), 7.30 (dd, J = 7.7, 1.8, 1H), 7.22 (t, J = 7.7 Hz, 1H), 6.34 (s, 1H), 5.82 (s, 1H), 5.52 (s, 1H), 3.76 (s, 3H), 3.19 (s, 1H). Full characterization data can be found on reference [20].

1l ^{1}H-NMR (400 MHz, $CDCl_3$) δ = 7.63 – 7.57 (m, 4H), 7.48 – 7.40 (m, 4H), 7.36 – 7.33 (m, 1H), 6.38 (s, 1H), 5.89 (s, 1H), 5.61 (s, 1H), 3.73 (s, 3H), 3.11 (s, 1H). Full characterization data can be found on reference [17].

1m ^{1}H-NMR (400 MHz, $CDCl_3$) δ = 7.88 – 7.79 (m, 4H), 7.51 – 7.45 (m, 3H), 6.37 (t, J = 0.9 Hz, 1H), 5.91 (t, J = 1.2 Hz, 1H), 5.73 (s, 1H), 3.70 (s, 3H), 3.15 (s, 1H). Full characterization data can be found on reference [21].

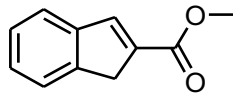
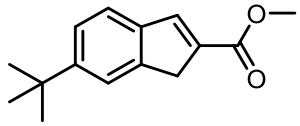
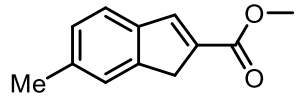
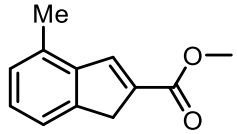
1n ^{1}H-NMR (400 MHz, $CDCl_3$) δ = 8.04 – 7.99 (m, 1H), 7.91 – 7.85 (m, 1H), 7.82 (dt, J = 8.2, 1.0 Hz, 1H), 7.66 (dt, J = 7.2, 1.0 Hz, 1H), 7.55 – 7.47 (m, 3H), 6.40 – 6.36 (m, 1H), 6.33 (t, J = 0.9 Hz, 1H), 5.59 (t, J = 1.2 Hz, 1H), 3.77 (s, 3H), 3.11 (s, 1H). Full characterization data can be found on reference [21].

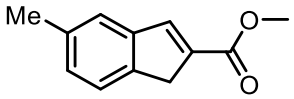
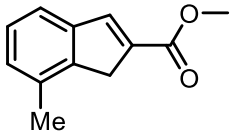
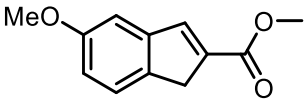
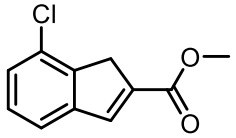
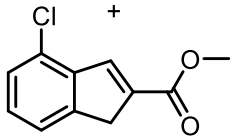
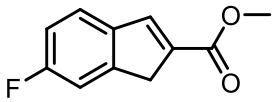
spectra were recorded on Varian 400 (400 MHz) spectrometer. Chemical shifts are reported in ppm from TMS with the solvent resonance as the internal standard (deuterochloroform: 7.24 ppm). Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, dd= doublet-doublet, t = triplet, td = triple doublet, dt = double triplet, q = quartet, sext = sextet, sept = septet, p = pseudo, b = broad, m = multiplet), coupling constants (Hz). ^{13}C-NMR spectra were recorded on a Varian 400 (100 MHz) spectrometer with complete

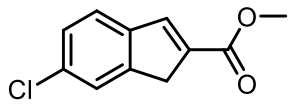
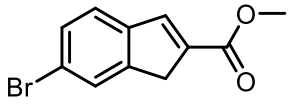
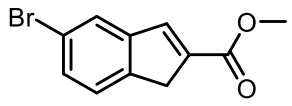
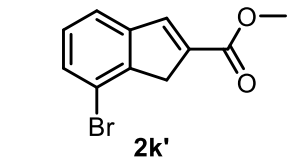
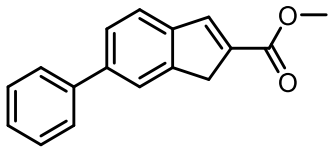
proton decoupling. Chemical shifts are reported in ppm from TMS with the solvent as the internal standard (deuterochloroform: 77.0 ppm).

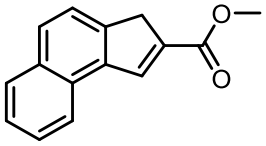
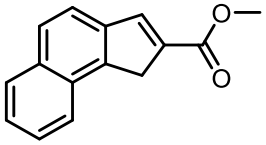
General procedure for GO catalysis.

In a 4 mL screw capped vial, **1** (0.1 mmol) was introduced, 27 mg of graphene oxide (GO) and 1mL of 1,2-dichloroethane were added. The vial was then sealed and placed in an oil bath at 80 °C where it was vigorously stirred for 16 h. After cooling to room temperature, the reaction mixture was filtered through a Celite pad to remove GO and washed with EtOAc (3 x 5 mL). The solvents were then removed under reduced pressure (rotary evaporator) and the residue was purified by flash column chromatography (FC) on silica gel (*n*Hex/EtOAc = 20/1) to afford pure products **2**. In the following characterization table, the appropriate amount of GO, temperature and reaction time is specified case by case when different from the standard procedure.

	**2a**. Reaction time: 16 h, GO: 27 mg (150 wt%), temperature 80 °C. White solid. FC eluent: *n*Hex/EtOAc: 20:1. Yield = 69%, (0.069 mmol, 12.0 mg); **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.73 (d, *J* = 0.9 Hz, 1H), 7.54 -7.50 (m, 2H), 7.38 – 7.30 (m, 2H), 3.85 (s, 3H), 3.69 (d, *J* = 2.0 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 165.4, 144.75, 142.67, 141.19, 137.01, 127.54, 126.83, 124.24, 123.36, 51.59, 38.34; **Anal. Calc.** for ($C_{11}H_{10}O_2$: 174.07): C, 75.84; H, 5.79; found: C, 75.60; H, 5.91.
	**2b**. Reaction time: 16 h, GO: 27 mg (150 wt%), temperature 80 °C. White solid. FC eluent: *n*Hex/EtOAc: 20:1. Yield = 66%, (0.066 mmol, 15.2 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.70 (q, *J* = 1.7 Hz, 1H), 7.58 – 7.54 (m, 1H), 7.46 (d, *J* = 8.0 Hz, 1H), 7.38 (dd, *J* = 8.0, 1.7 Hz, 1H), 3.84 (s, 3H), 3.67 (d, *J* = 2.0 Hz, 2H), 1.36 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 165.4, 151.2, 144.9, 141.1, 140.1, 136.3, 124.1, 122.8, 121.3, 51.5, 38.3, 35.7, 34.9 (3C), 31.4; **Anal. Calc.** for ($C_{15}H_{18}O_2$: 230.13): C, 78.23; H, 7.88; found: C, 78.45; H, 8.01.
	**2c** Reaction time: 72 h, GO: 40.5 mg (250 wt%), temperature 80 °C. White solid. FC eluent: *n*Hex/EtOAc: 20:1. Yield = 47%, (0.047 mmol, 8.9 mg). **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.70 (s, 1H), 7.40 (d, *J* = 7.8 Hz, 1H), 7.32 (s, 1H), 7.15 (d, *J* = 7.7 Hz, 1H), 3.84 (s, 3H), 3.64 (d, *J* = 2.1 Hz, 2H), 2.42 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 165.6, 145.2, 141.2, 140.1, 137.8, 135.9, 127.8, 125.1, 123.0, 51.5, 38.1, 21.7; **Anal. Calc.** for ($C_{12}H_{12}O_2$: 188.08): C, 76.57; H, 6.43; found: C, 76.39; H, 6.60.
	**2d.** Reaction time: 72 h, GO: 40.5 mg (250 wt%), temperature 80 °C. White solid. FC eluent: *n*Hex/EtOAc: 20:1. Yield = 56%, (0.056 mmol, 10.5 mg). **^{1}H NMR (**400 MHz, $CDCl_3$) δ =7.90 (t, *J* = 0.9 Hz, 1H), 7.37 (d, *J* = 7.5 Hz, 1H), 7.29 (t, *J* = 7.6 Hz, 1H), 7.16 (d, *J* = 7.5 Hz, 1H), 3.89 (s, 3H), 3.72 (d, *J* = 1.4 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 165.7, 145.0, 142.0, 139.8, 136.4, 133.1, 127.9, 127.9, 121.8, 51.7, 38.7, 18.6;

	Anal. Calc. for ($C_{12}H_{12}O_2$: 188.08): C, 76.57; H, 6.43; found: C, 76.69; H, 6.29.
 **2e**  **2e'**	**2e** Reaction time: 16 h, GO: 27 mg (150 wt%), temperature 80 °C. White solid. FC eluent: *n*Hex/EtOAc: 20:1. Yield = 66%, (0.066 mmol, 15.2 mg); **2e**:**2e'** = 2.4:1. This compound was isolated along with 7% of **1e''** (unseparable mixture by chromatography). The reported yield is corrected for this contamination. **^{1}H NMR** (600 MHz, $CDCl_3$) δ = 7.74 (t, *J* = 2.0 Hz, 1H **2e'**), 7.68 (td, *J* = 2.0, 0.8 Hz, 1H **2e**), 7.43 – 7.31 (m, 2H **2e** + 1H **2e'**), 7.26 (t, *J* = 7.6 Hz, 1H **2e'**), 7.18 – 7.14 (m, 1H **2e** + 1H **2e'**), 3.86 (s, 3H **2e'**), 3.84 (s, 3H **2e**), 3.66 – 3.63 (m, 2H **2e**), 3.59 (d, *J* = 2.0 Hz, 2H **2e'**), 2.41 (s, 3H **2e**), 2.39 (s, 3H **2e'**); **^{13}C NMR** (150 MHz, $CDCl_3$) δ = 165.5 (**2e**+**2e'** overlapped), 143.6 (**2e'**), 142.9 (**2e**), 142.3 (**2e'**), 141.9 (**2e**), 141.6 (**2e'**), 141.3 (**2e**), 137.2 (**2e**), 136.5 (**2e'**), 130.5 (**2e**), 130.3 (**2e'**), 128.7 (**2e'**), 128.6 (**2e**), 123.9 (**2e'**), 123.9 (**2e**), 51.6 (**2e'**), 51.6 (**2e**), 38.0 (**2e**), 37.4 (**2e'**), 21.3 (**2e**), 18.6 (**2e'**); **Anal. Calc.** for ($C_{12}H_{12}O_2$: 188.08): C, 76.57; H, 6.43; found: C, 76.71; H, 6.55.
	**2f** Reaction time: 72 h, GO: 40.5 mg (250 wt%), temperature 80 °C. White solid. FC eluent: *n*Hex/EtOAc: 20:1. Yield = 51%, (0.051 mmol, 10.4 mg); **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.67 (t, *J* = 0.8 Hz, 1H), 7.39 (dt, *J* = 8.2, 0.8 Hz, 1H), 7.05 (d, *J* = 2.4 Hz, 1H), 6.91 (dd, *J* = 8.3, 2.4 Hz, 1H), 3.84 (s, 6H), 3.62 (d, *J* = 2.1, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 165.5, 159.3, 144.1, 141.3, 138.4, 137.2, 124.9, 114.5, 108.2, 55.7, 51.8, 37.8. **Anal. Calc.** for ($C_{12}H_{12}O_3$: 204.08): C, 70.58; H, 5.92; found: C, 70.37; H, 5.99.
 + 	**2g** Reaction time: 16 h, GO: 40.5 mg (250 wt%), temperature 80 °C. White solid. FC eluent: *n*Hex/EtOAc: 20:1. Yield = 52%, (0.052 mmol, 10.9 mg). **^{1}H NMR** (600 MHz, $CDCl_3$) δ = 7.79 (s, 1H), 7.65 (s, 1H), 7.37 (d, *J* = 6.9 Hz, 1H), 7.34 (d, *J* = 7.3 Hz, 1H), 7.28 – 7.19 (m, 4H), 3.81 (s, 6H), 3.70 (s, 2H), 3.67 (s, 2H); all peaks of the two isomers are given, without assignment; **^{13}C NMR** (150 MHz, $CDCl_3$) δ = 165.2 (2C overlapped), 146.4, 144.3, 142.6, 141.3, 140.7, 138.6, 137.9, 137.9, 130.5, 129.6, 128.9, 128.8, 127.8, 127.3, 122.7, 122.0, 51.9 (2C overlapped), 39.4, 38.3; all peaks of the two isomers are given, without assignment; **Anal. Calc.** for ($C_{11}H_9ClO_2$: 208.03): C, 63.32; H, 4.05; found: C, 63.44; H, 3.89.
	**2h** Reaction time: 48 h, GO: 40.5 mg (250 wt%), temperature 80 °C. White solid. FC eluent: *n*Hex/EtOAc: 20:1. Yield = 80%, (0.080 mmol, 15.4 mg); **^{1}H NMR** (600 MHz, $CDCl_3$) δ = 7.68 (s, 1H), 7.45 (dd, *J* = 8.4, 5.1 Hz, 1H), 7.22 (dd, *J* = 8.7, 2.3 Hz, 1H), 7.04 (td, *J* = 8.9, 2.5 Hz, 1H), 3.84 (s, 3H), 3.68 (s, 2H); **^{13}C NMR** (150 MHz, $CDCl_3$) δ = 165.3, 163.2 (d, *J* = 247.0 Hz), 147.2 (d, *J* = 9.0 Hz), 140.5, 138.9 (d, *J* = 2.4 Hz), 124.4 (d, *J* = 9.1 Hz), 114.4 (d, *J* = 23.3 Hz), 112.1 (d, *J* = 23.4 Hz), 51.8, 38.7 (d, *J* = 2.7 Hz);

	^{19}F NMR (564 MHz, $CDCl_3$) δ = -117.98 (m, 1F); **Anal. Calc.** for ($C_{11}H_9FO_2$: 192.06): C, 68.75; H, 4.72; found: C, 68.88; H, 4.61.
	**2i** Reaction time: 48 h, GO: 27 mg (150 wt%), temperature 80 °C. White solid. FC eluent: nHex/EtOAc: 20:1. Yield = 61%, (0.061 mmol, 12.7 mg); **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.66 (s, 1H), 7.48 (s, 1H), 7.42 (d, *J* = 8.1 Hz, 1H), 7.31 (dd, *J* = 8.1, 2.0 Hz, 1H), 3.84 (s, 3H), 3.67 (d, *J* = 2.0 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 161.1, 142.3, 137.2, 136.2, 133.4, 129.8, 123.3, 120.7, 120.1, 47.8, 34.4; **Anal. Calc.** for ($C_{11}H_9ClO_2$: 208.03): C, 63.32; H, 4.05; found: C, 63.47; H, 4.18.
	**2j** Reaction time: 16 h, GO: 40.5 mg (250 wt%), temperature 80 °C. White solid. FC eluent: nHex/EtOAc: 20:1. Yield = 54%, (0.054 mmol, 13.7 mg); **^{1}H NMR** (600 MHz, $CDCl_3$) δ = 7.66 – 7.64 (m, 2H), 7.47 (dd, *J* = 8.1, 1.7 Hz, 1H), 7.37 (d, *J* = 8.1 Hz, 1H), 3.84 (s, 3H), 3.67 (d, *J* = 1.9 Hz, 2H); **^{13}C NMR** (151 MHz, , $CDCl_3$) δ = 165.0, 146.5, 141.5, 140.2, 137.4, 130.1, 127.6, 124.4, 122.0, 51.7, 38.3; **Anal. Calc.** for ($C_{11}H_9BrO_2$: 251.98): C, 52.20; H, 3.58; found: C, 52.38; H, 3.79.
 **2k**  **2k'**	**2k** Reaction time: 48 h, GO: 27 mg (150 wt%), temperature 80 °C. White solid. FC eluent: *n*Hex/EtOAc: 20:1. Yield = 56%, (0.056 mmol, 14.2 mg). **2k**:**2k'** = 3.0:1 ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.73 (t, *J* = 2.0 Hz, 1H **2k'**), 7.65 (d, *J* = 1.8 Hz, 1H **2k**), 7.64 (t, *J* = 2.1 Hz, 1H **2k**), 7.49 – 7.43 (m, 2H **2k'** + 1H **2k**), 7.37 (d, *J* = 8.0 Hz, 1H **2k**), 7.22 (t, *J* = 7.7 Hz, 1H **2k'**), 3.86 (s, 3H **2k'**), 3.85 (s, 3H **2k**), 3.68 (d, *J* = 1.9 Hz, 2H **2k'**), 3.64 (d, *J* = 2.0 Hz, 2H **2k**); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 165.0, 144.7, 143.3, 139.8, 130.3, 126.3, 125.6, 122.4, 120.8, 51.8, 38.1; only the peaks relative to major isomer **2k** are given; **Anal. Calc.** for ($C_{11}H_9BrO_2$: 251.98): C, 52.20; H, 3.58; found: C, 52.40; H, 3.44.
	**2l** Reaction time: 72 h, GO: 40.5 mg (250 wt%), temperature 80 °C. White solid. FC eluent: *n*Hex/EtOAc: 25:1. Yield = 45%, (0.45 mmol, 11.3 mg); **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.78 – 7.71 (m, 2H), 7.64 – 7.60 (m, 2H), 7.58 (*pseudo*s, 2H), 7.50 – 7.42 (m, 2H), 7.39 – 7.33 (m, 1H), 3.86 (s, 3H), 3.76 (s, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ = ^{13}C NMR (101 MHz, cdcl$_3$) δ 165.4, 145.5, 141.9, 141.1, 140.9, 140.8, 137.2, 128.8, 127.4, 127.3, 126.1, 123.6, 123.1, 51.6, 38.5. **Anal. Calc.** for ($C_{17}H_{14}O_2$: 250.10): C, 81.58; H, 5.64; found: C, 81.45; H, 5.69.

	**2m** Reaction time: 72 h, GO: 40.5 mg (250 wt%), temperature 80 °C. White solid. FC eluent: *n*Hex/EtOAc: 25:1. Yield = 60%, (0.060 mmol, 13.3 mg); **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.35 (d, *J* = 0.8 Hz, 1H), 8.16 (dd, *J* = 8.2, 1.1 Hz, 1H), 7.95 – 7.90 (m, 1H), 7.85 (d, *J* = 8.3 Hz, 1H), 7.68 – 7.48 (m, 3H), 3.90 (s, 3H), 3.86 (d, *J* = 1.9 Hz, 2H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 165.4, 136.9, 136.5, 132.9, 132.6, 129.9, 128.9, 128.3, 126.7, 126.6, 126.1, 125.6, 123.9, 51.7, 39.7; **Anal. Calc.** for ($C_{15}H_{12}O_2$: 224.08): C, 80.34; H, 5.39; found: C, 80.22; H, 5.55.
	**2n** Reaction time: 72 h, GO:40.5 mg (250 wt%), temperature 60 °C. White solid. FC eluent: nHex/EtOAc: 25:1. Yield = 51%, (0.051 mmol, 11.4 mg); **^{1}H NMR** (600 MHz, $CDCl_3$) δ = 8.03 – 7.98 (m, 1H), 7.91 – 7.87 (m, 1H), 7.85 (t, *J* = 1.9 Hz, 1H), 7.81 (d, *J* = 8.4 Hz, 1H), 7.63 (d, *J* = 8.4 Hz, 1H), 7.53 – 7.49 (m, 2H), 4.05 – 4.00 (m, 2H), 3.89 (s, 3H); **^{13}C NMR** (150 MHz, $CDCl_3$) δ = 165.4, 142.8, 142.1, 140.3, 136.7, 133.0, 130.1, 129.0, 128.1, 126.9, 126.2, 124.1, 121.2, 51.8, 37.7; **Anal. Calc.** for ($C_{15}H_{12}O_2$: 224.08): C, 80.34; H, 5.39; found: C, 80.28; H, 5.21.

Hot Filtration experiment

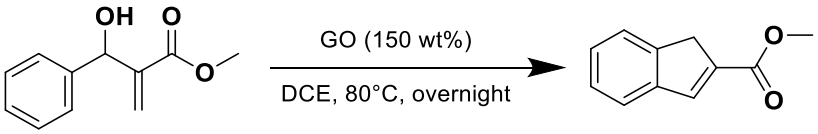
In a vial, 0.1 mmol of **1a** (18 mg) was introduced with 27 mg of graphene oxide and 1 mL of 1,2-dichloroethane. The vial was then sealed and placed in a pre-heated oil bath at 80 °C, and vigorously stirred for 4 h. After cooling to room temperature, the reaction mixture was quickly filtered through a Celite pad to remove GO, washed with a minimum amount of EtOAc (0.5 mL) and then placed again in an oil bath at 80 °C for 12 h.

Reaction profile monitoring

The reaction profile of the model transformation (**1a** $\rightarrow$ **2a**) was monitored over 210 minutes. Several equal reactions were analyzed by ^{1}H-NMR spectroscopy using 1,3,5-trimethylbenzene (0.1 mmol, 13.9 µL) as an internal standard to determine the yield of **1a'**, **2a** and the remaining **1a**.

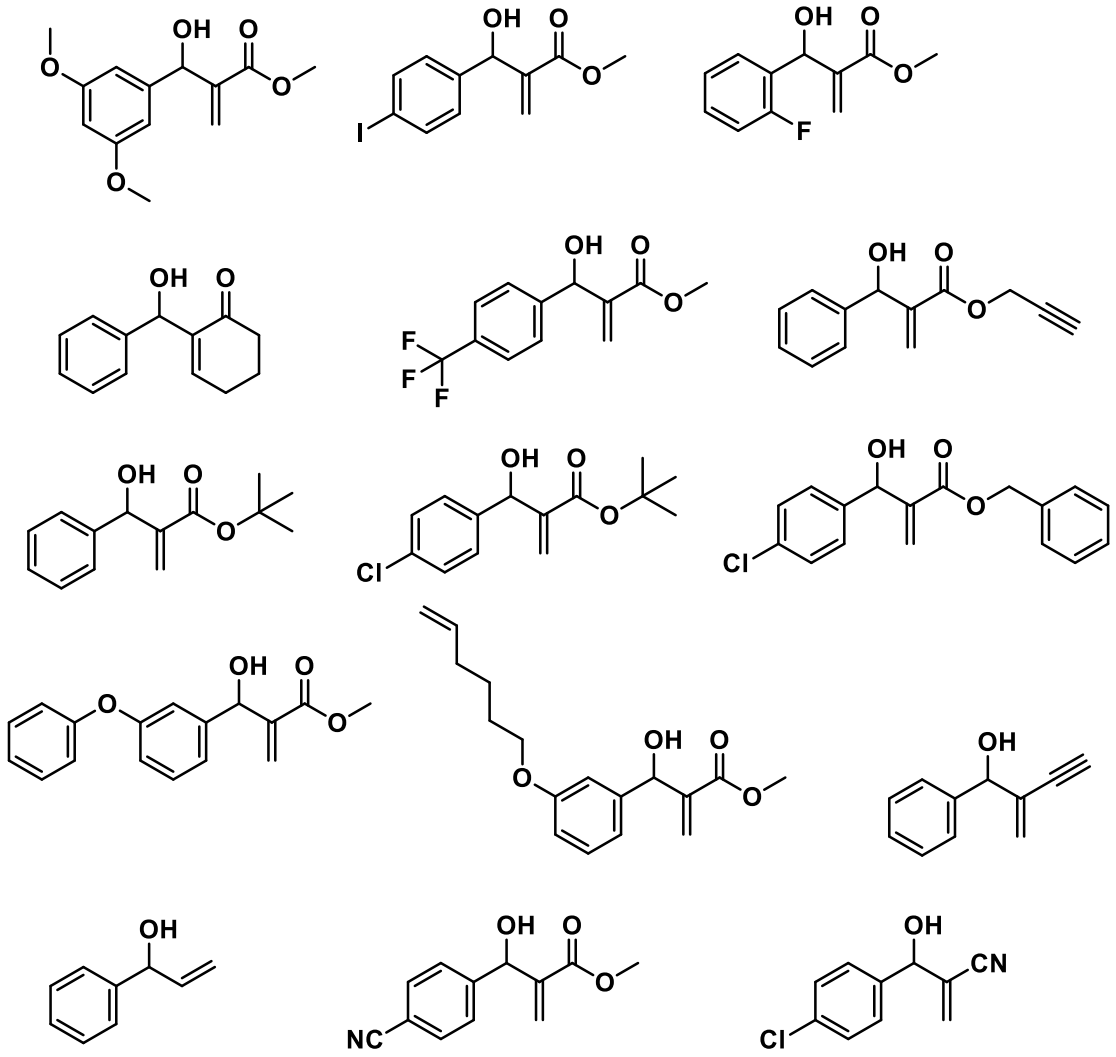
Reaction time (min)	Yield of **2a** (%)	Yield of **1a'** (%)	Unreacted **1a** (%)
0	0	0	100
30	4	23	37
60	7	34	22
90	20	28	7
120	25	26	5
150	38	11	2
210	49	9	2

Additional Optimization Screening



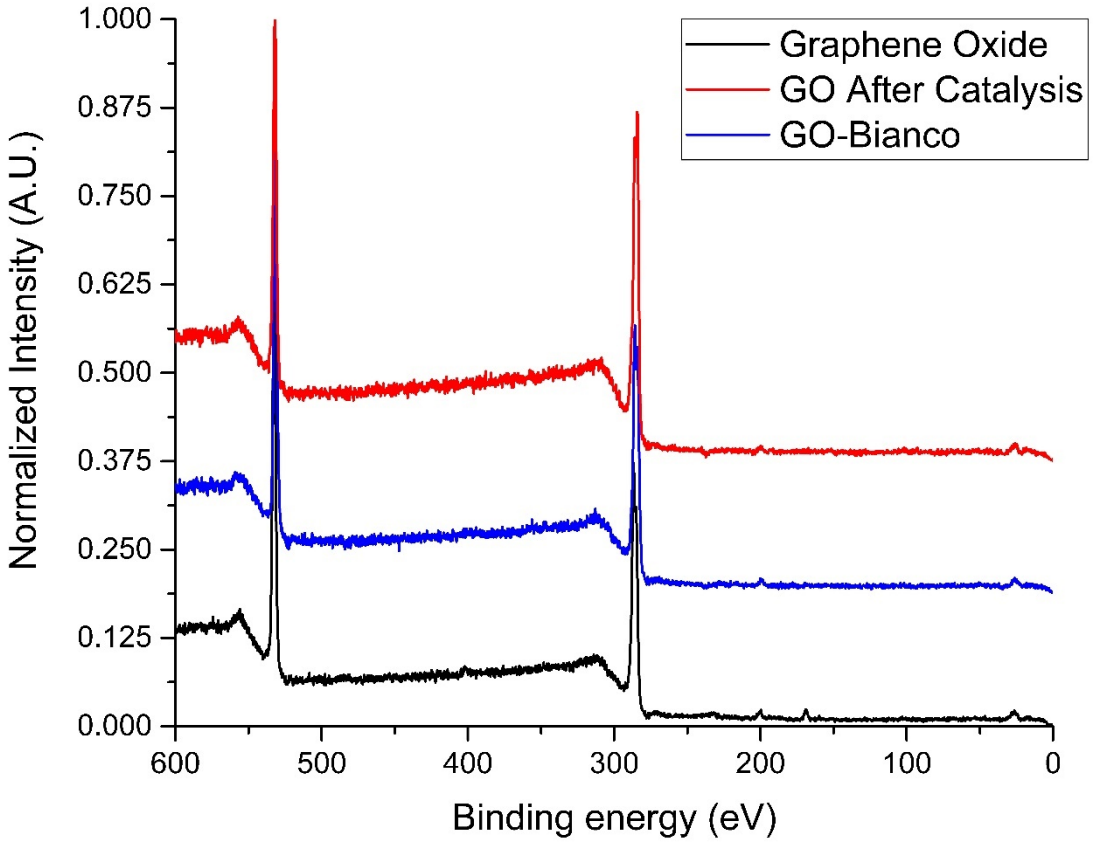
Run	Variotion from optimal: promoter (wt%)	Variations from optimal: conditions	Yield (%) **2a**[b]
1	GO (100)	-	40%
2	-	100°C	36%
3	-	rt	NR
4	rGO (150)	-	NR
5	-	H_2O (3 equiv)	Traces (**1a'** formation)
6	pTsOH (100%mol)	-	NR
7	rGO (150), pTsOH (100%mol)	-	NR
8	-	CPME as solvent	NR
9	-	2-MeTHF as solvent	Traces (**1a'** formation)

Unsuccessful substrates



XPS Analysis

The pristine GO presents C 1s (285.0 eV), O 1s (532.6 eV), N 1s (401.5 eV), Cl 2p (200.2 eV) and S 2p (168.6 eV) signals.

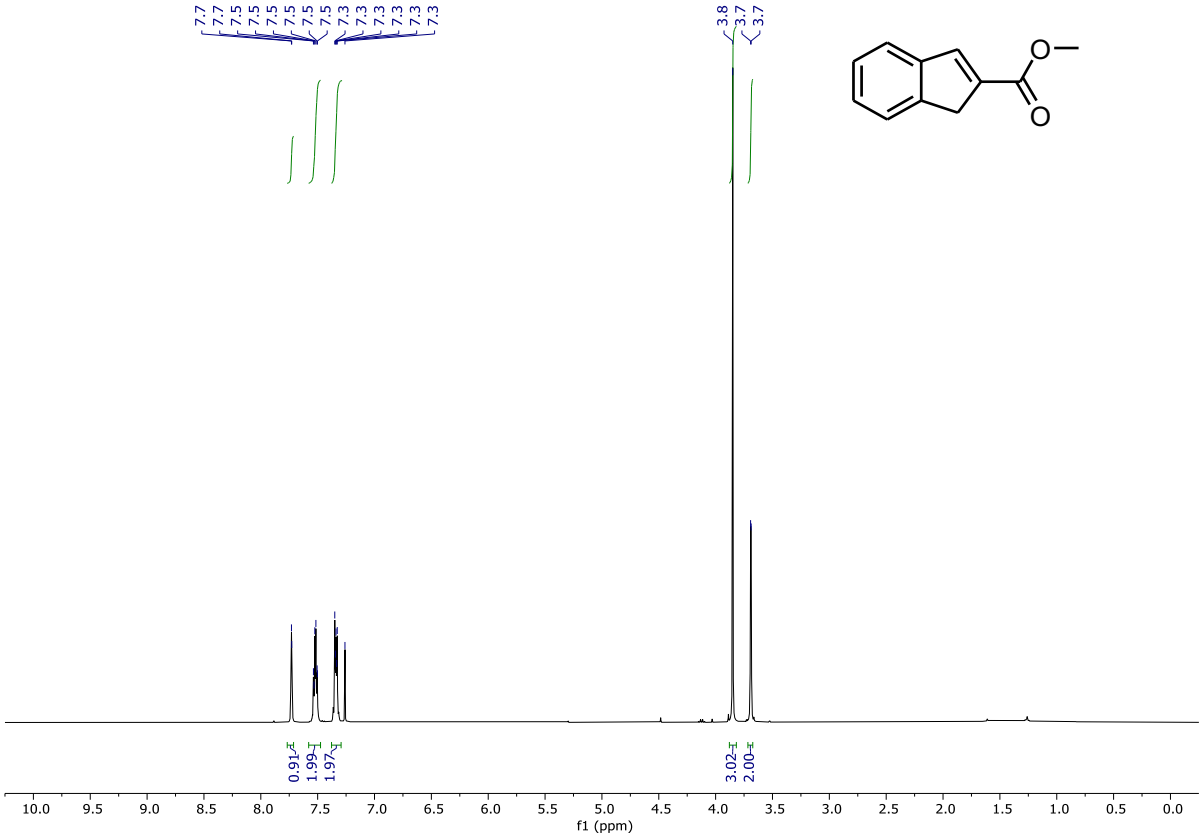


Sample	C 1s 285.0	O 1s 531.5 eV	S 2p 167.8 eV	N 1s 400.8 eV	Cl 2p 198.8	O/C
Graphene Oxide	72.0	25.8	1.0	0.8	0.5	0.36
GO Control	73.1	25.8	0.5	\\	0.6	0.35
GO after Catalysis	77.8	21.9	\\	\\	0.3	0.28

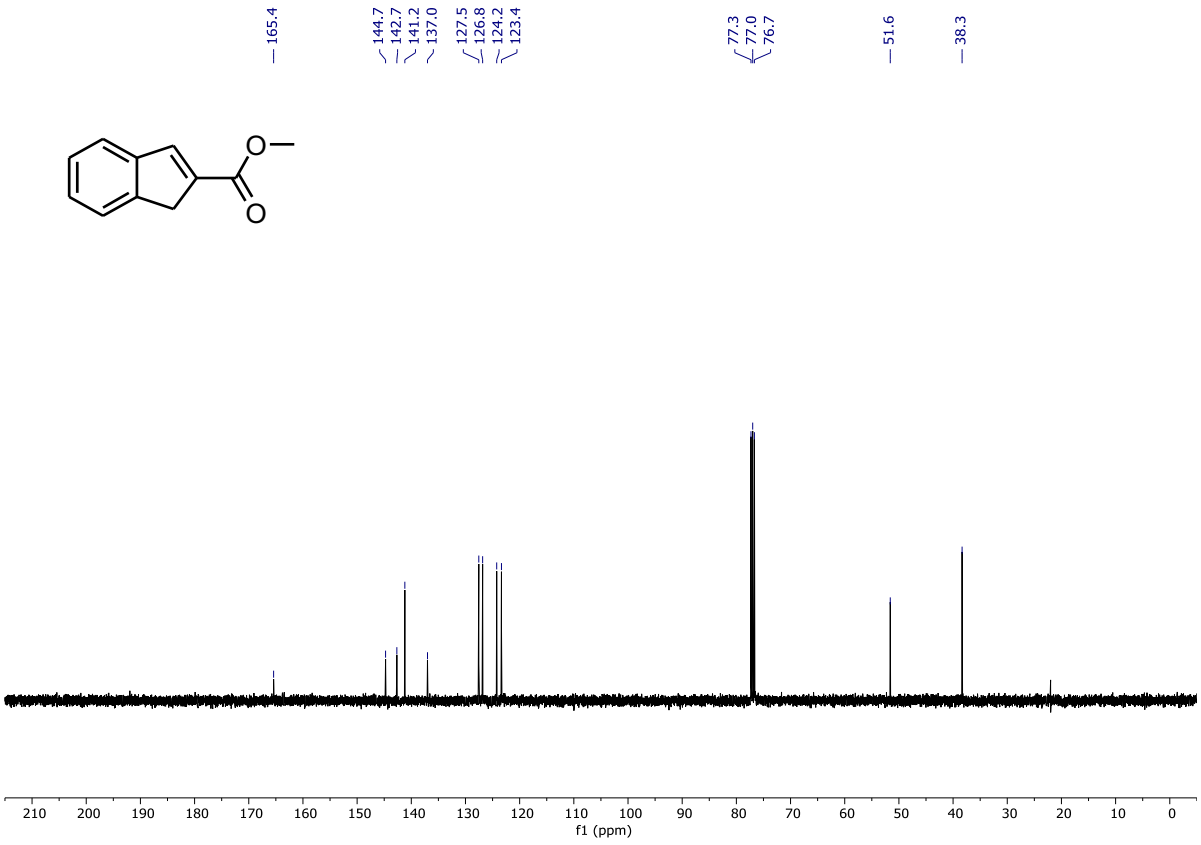
Sample	C sp^2	C sp^3	C=C*	C-OH	C-O-C	C=O	O-C=O
Graphene Oxide	31.6	7.5	4.9	14.8	33	5	5.2
GO Control	27.8	11.5	12.3	16.3	24.2	5.3	2.7
GO after Catalysis	38.4	7.5	7.0	20.4	16.5	5.2	5.2

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

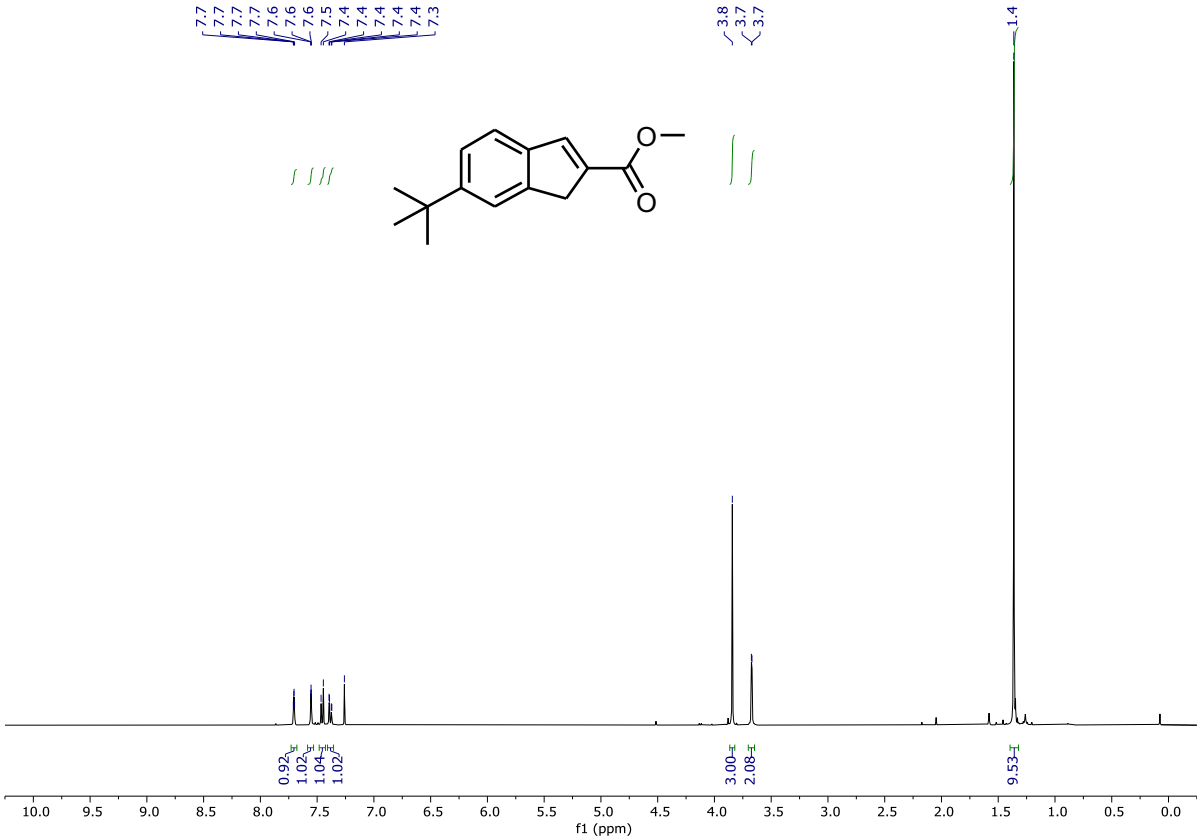
2a ^{1}H NMR (400 MHz, $CDCl_3$)



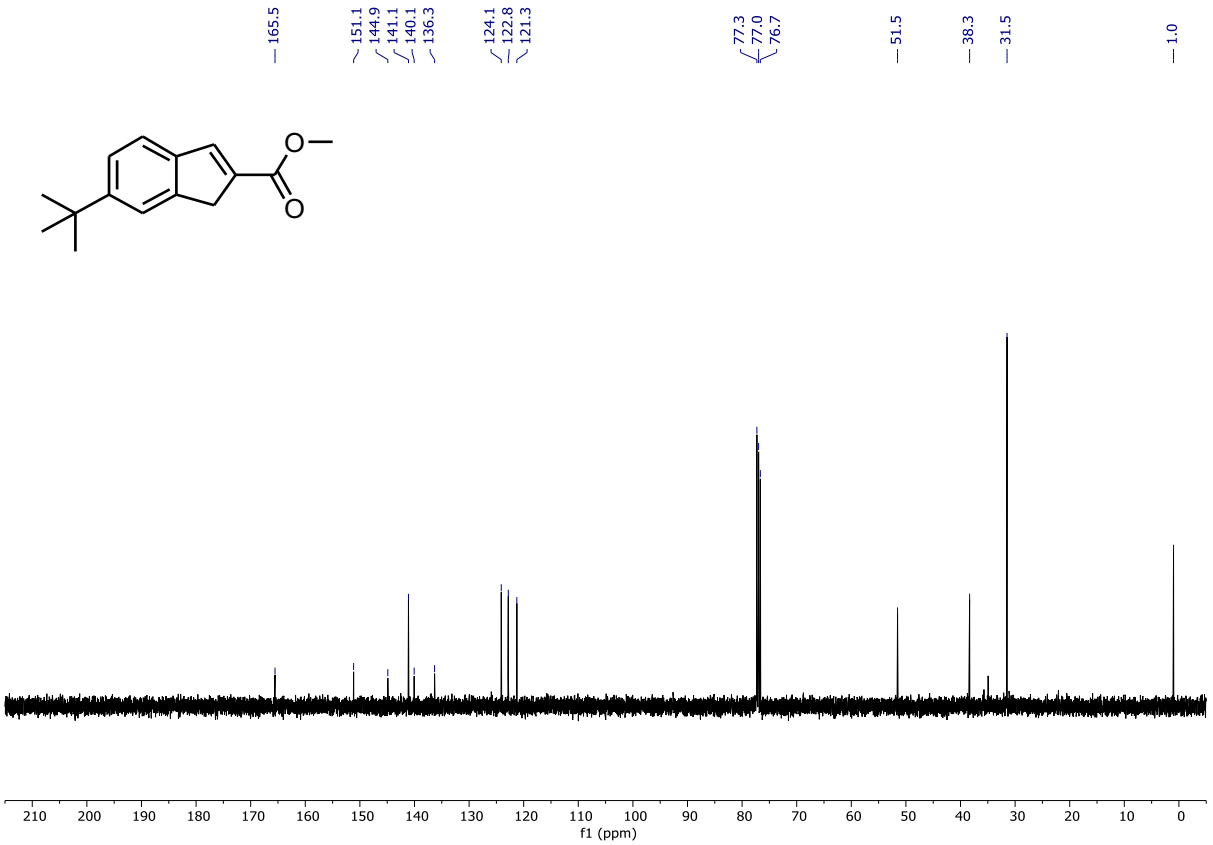
2a ^{13}C NMR (101 MHz, $CDCl_3$)



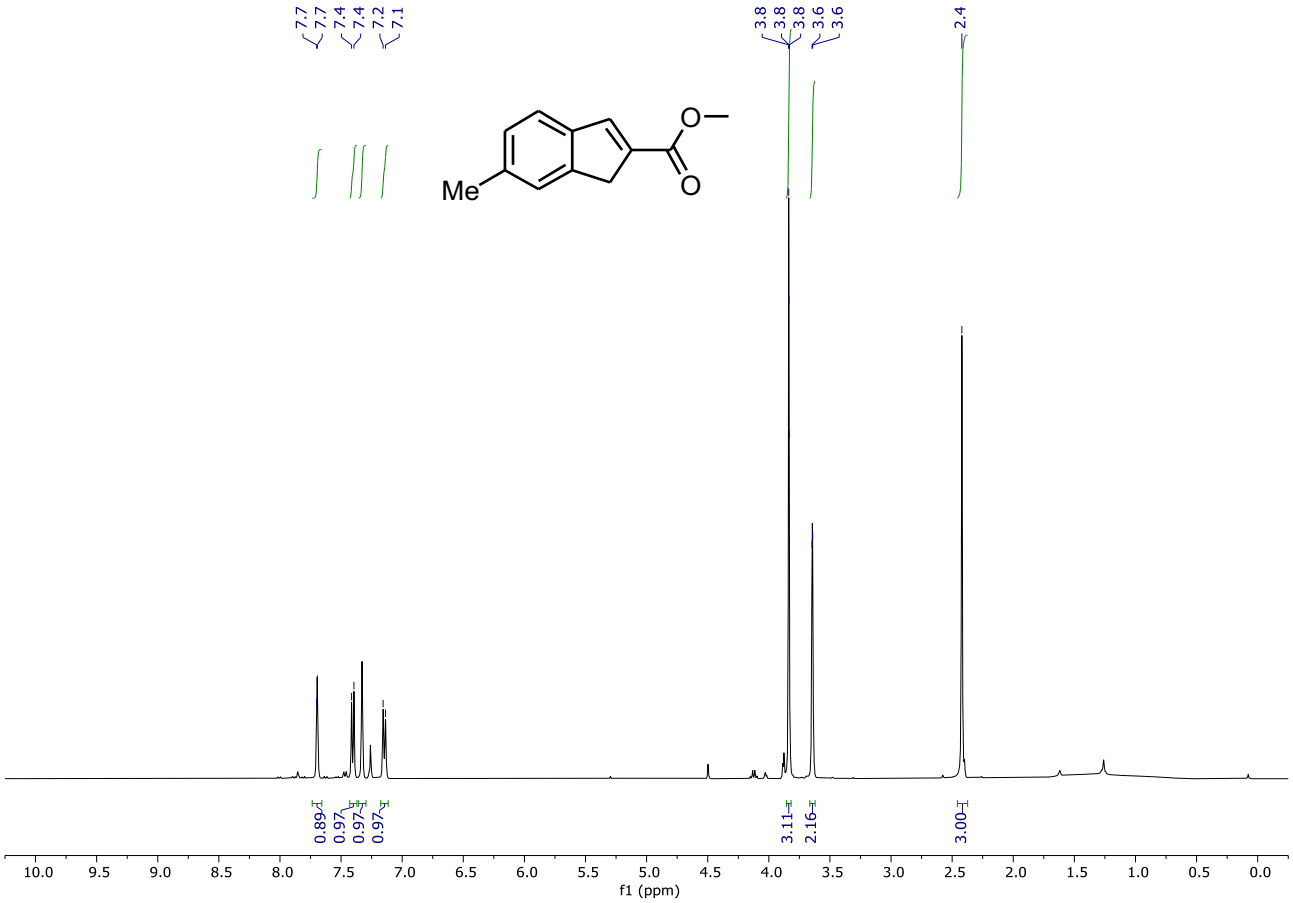
2b ^{1}H NMR (400 MHz, $CDCl_3$)



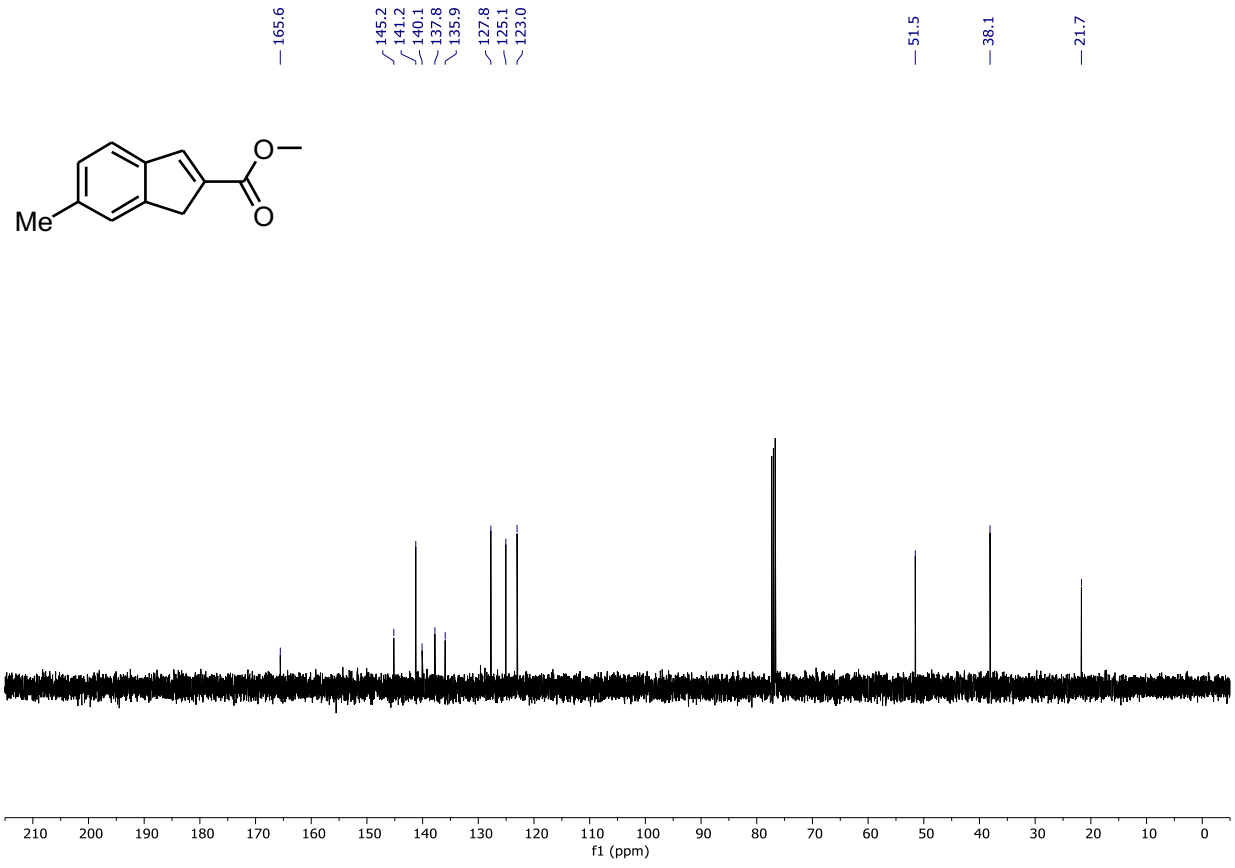
2b ^{13}C NMR (101 MHz, $CDCl_3$)



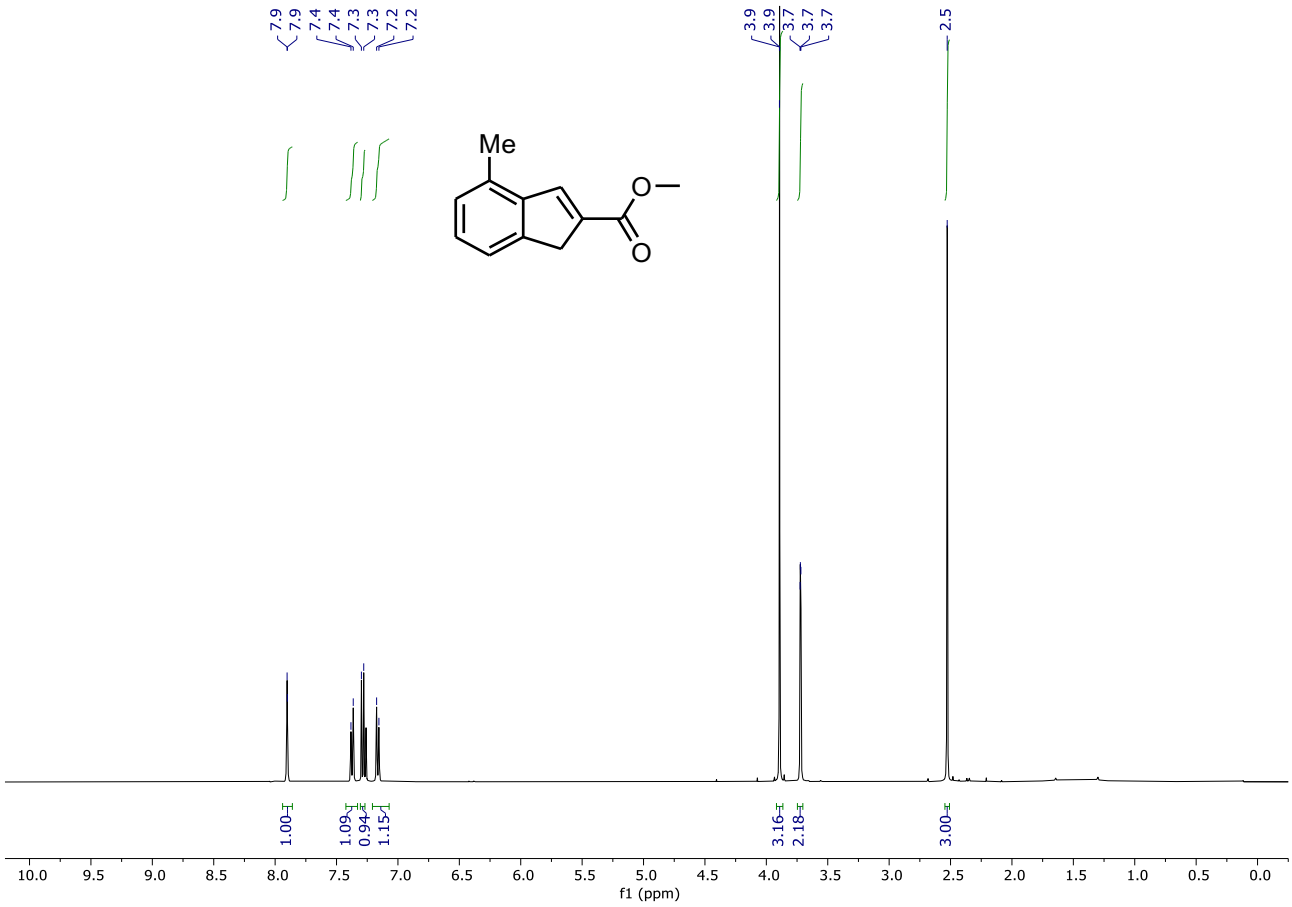
2c ^{1}H NMR (400 MHz, $CDCl_3$)



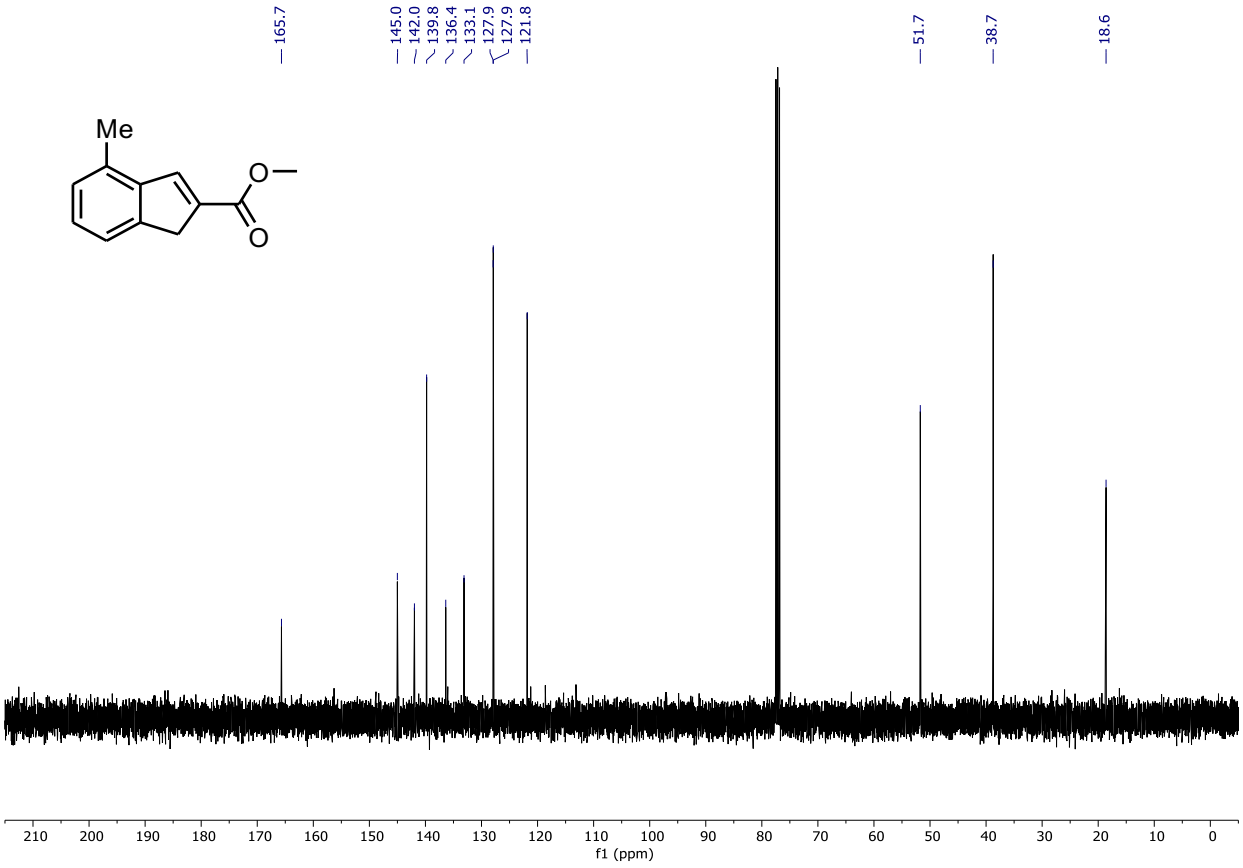
2c ^{13}C NMR (101 MHz, $CDCl_3$)



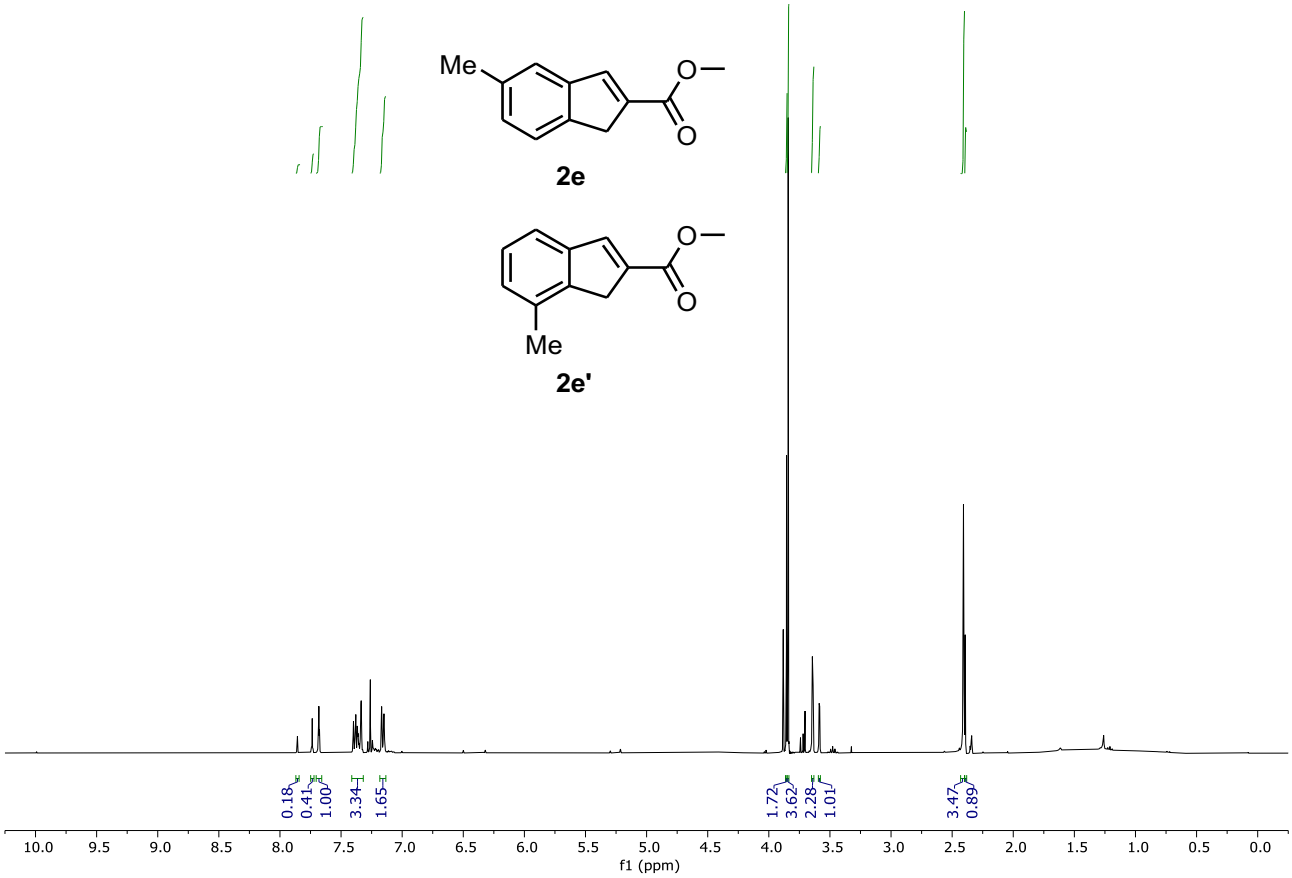
2d ^{1}H NMR (400 MHz, $CDCl_3$)



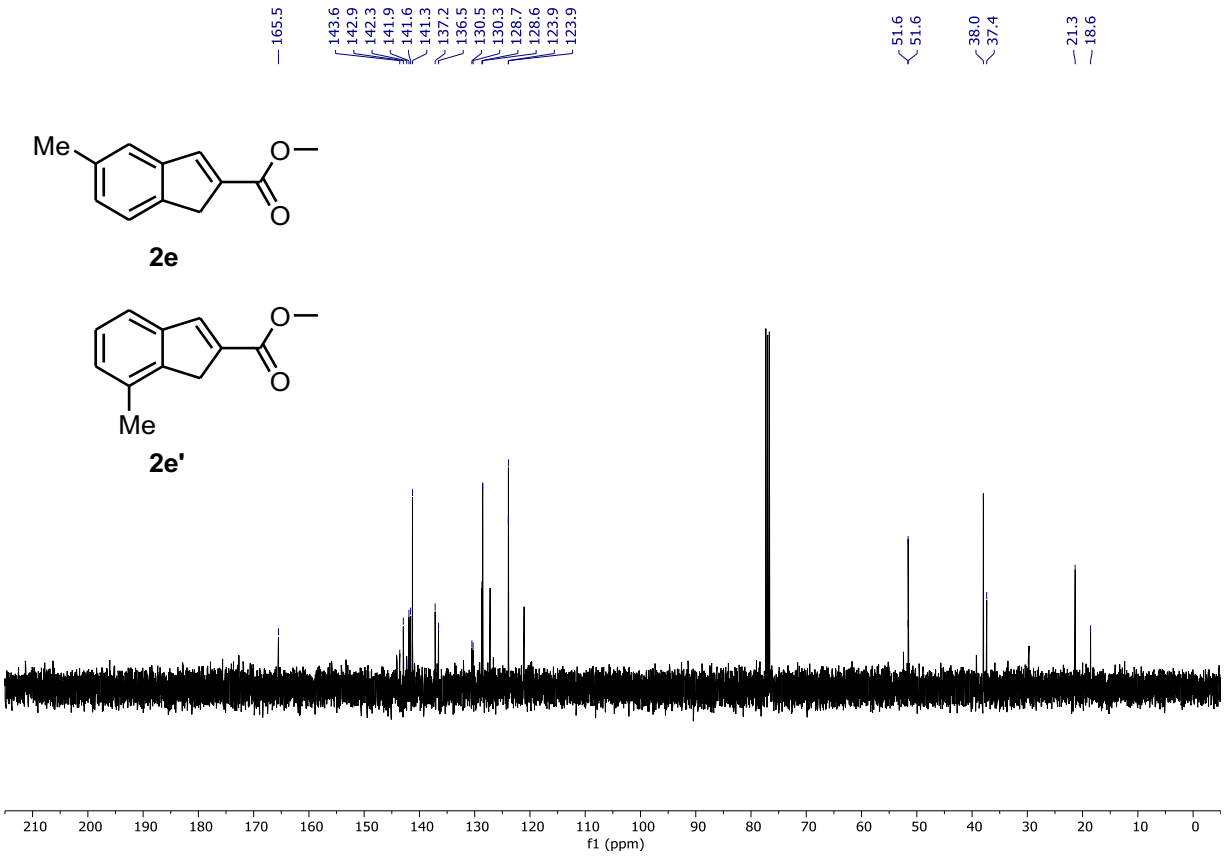
2d ^{13}C NMR (101 MHz, $CDCl_3$)



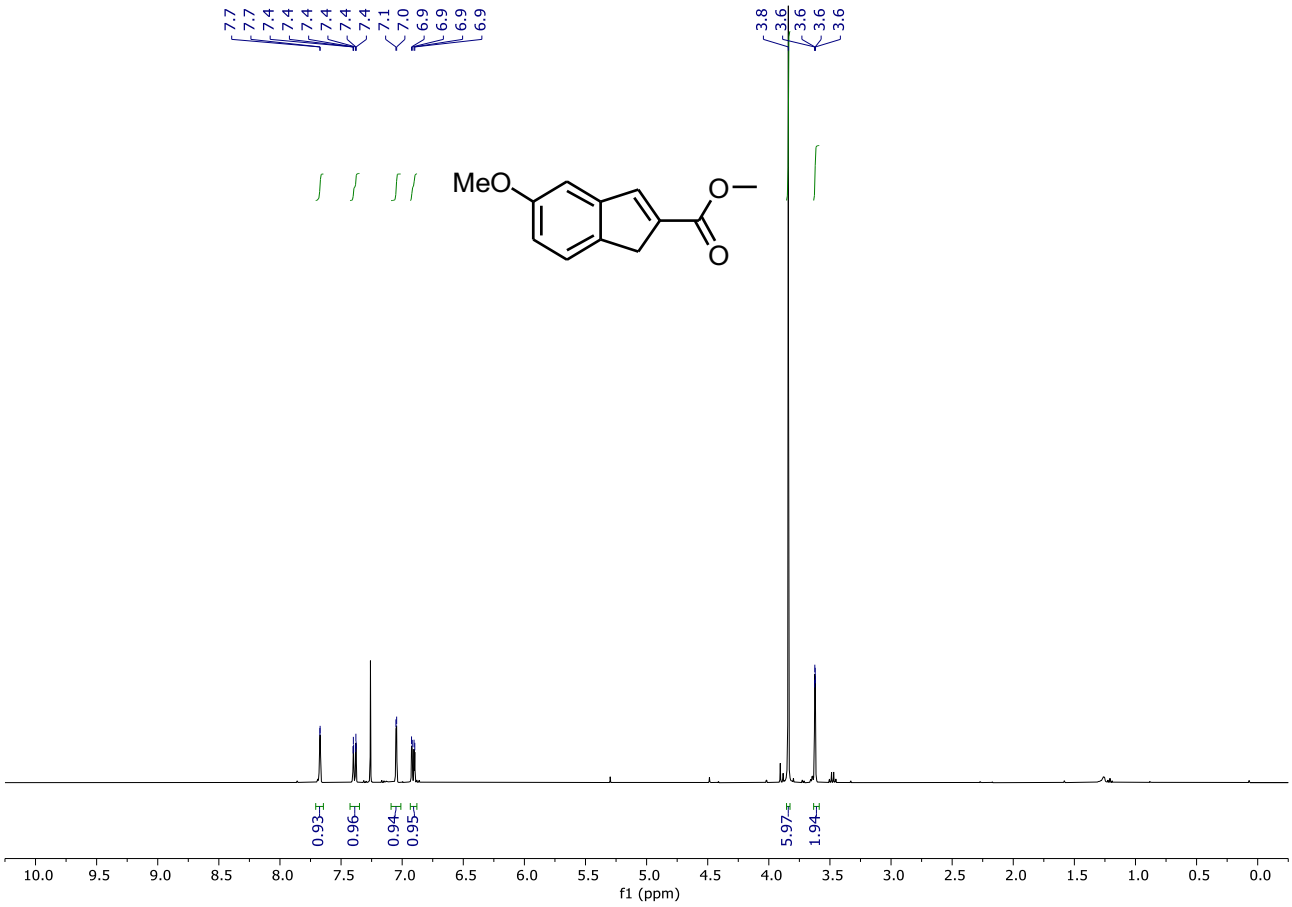
2e ^{1}H NMR (600 MHz, $CDCl_3$)



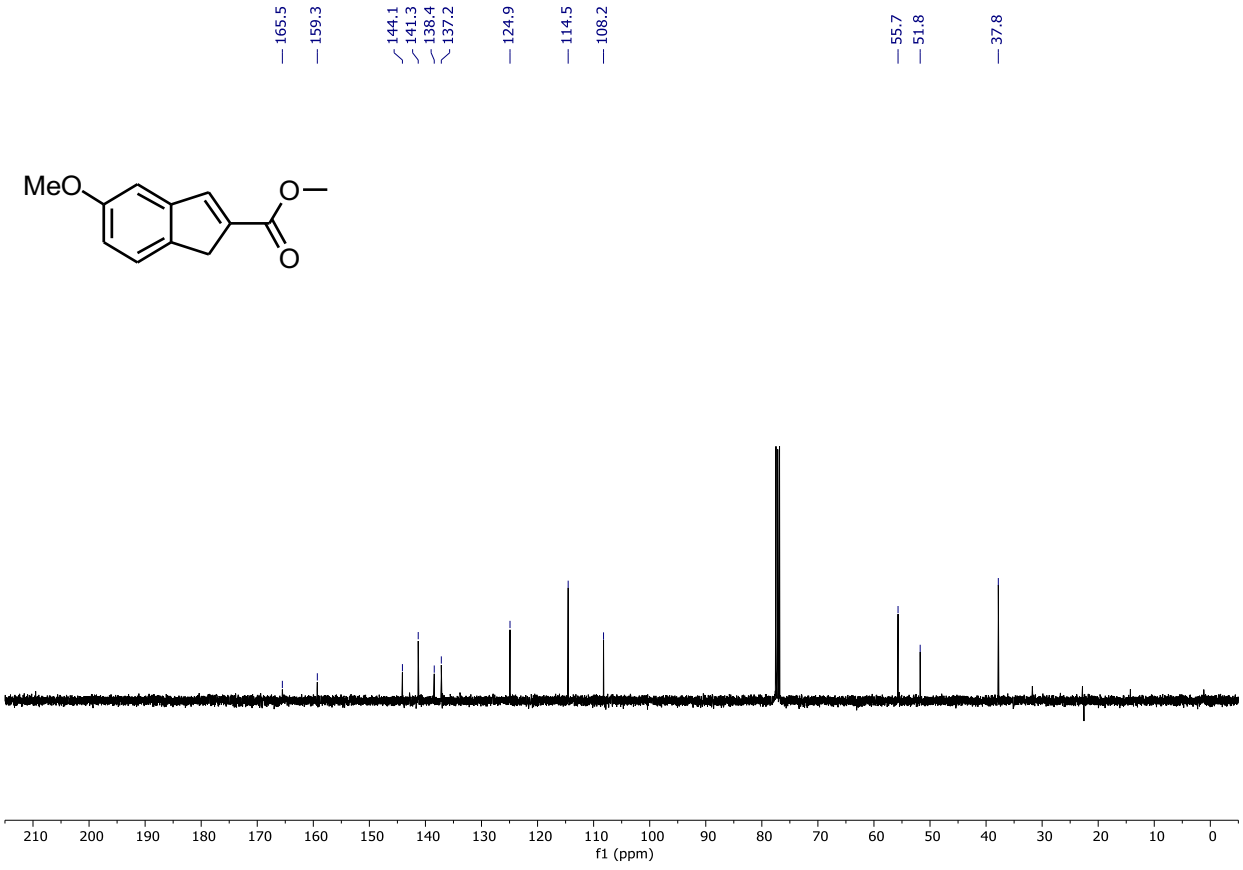
2e ^{13}C NMR (151 MHz, $CDCl_3$)



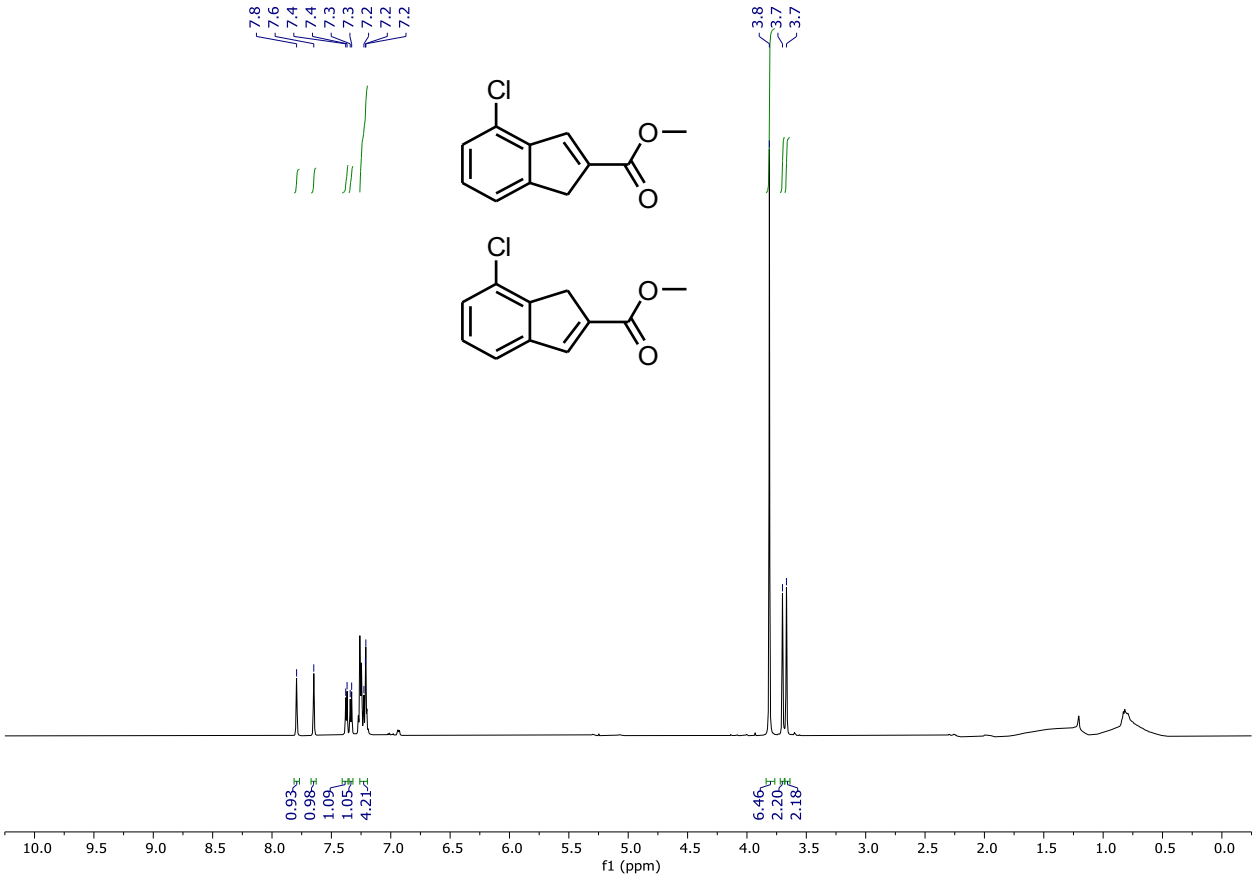
2f ^{1}H NMR (400 MHz, $CDCl_3$)



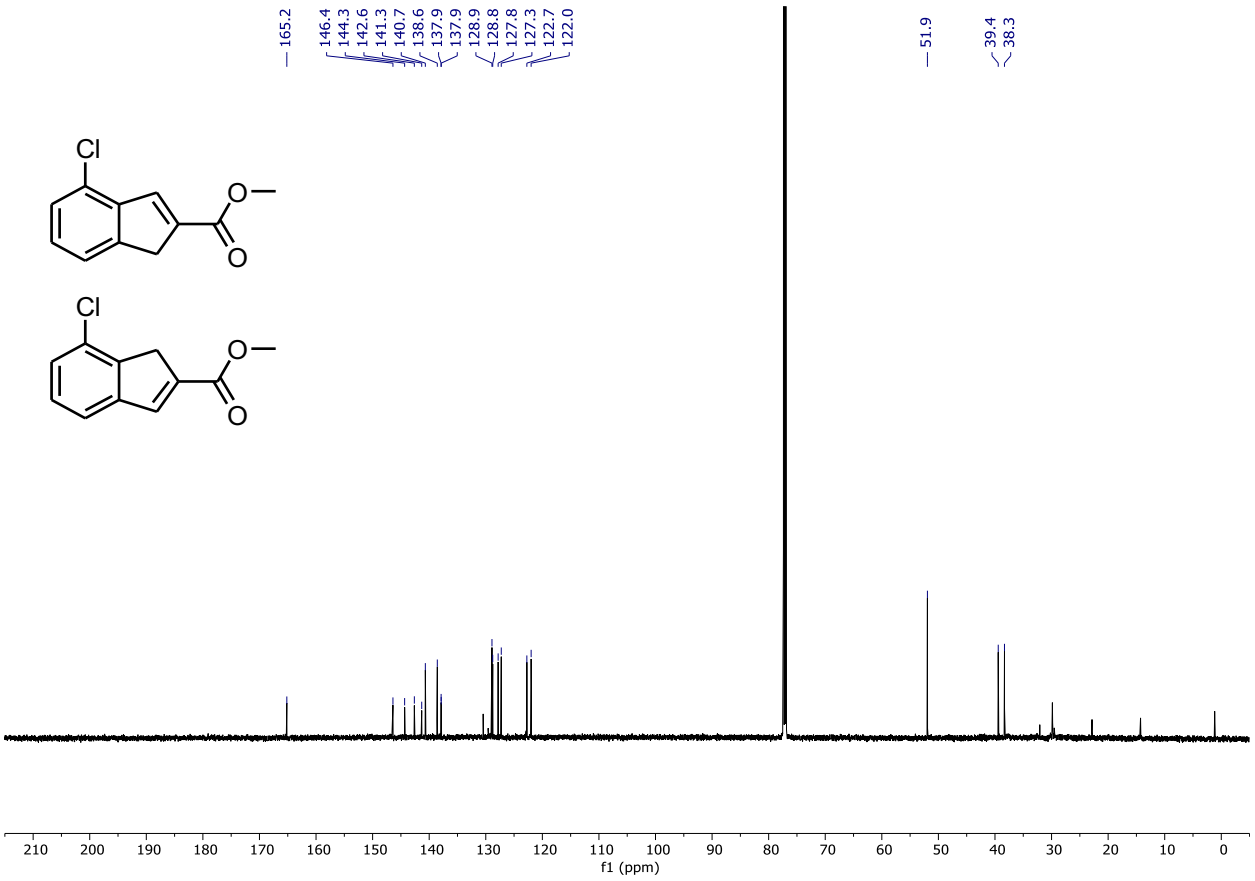
2f ^{13}C NMR (101 MHz, $CDCl_3$)



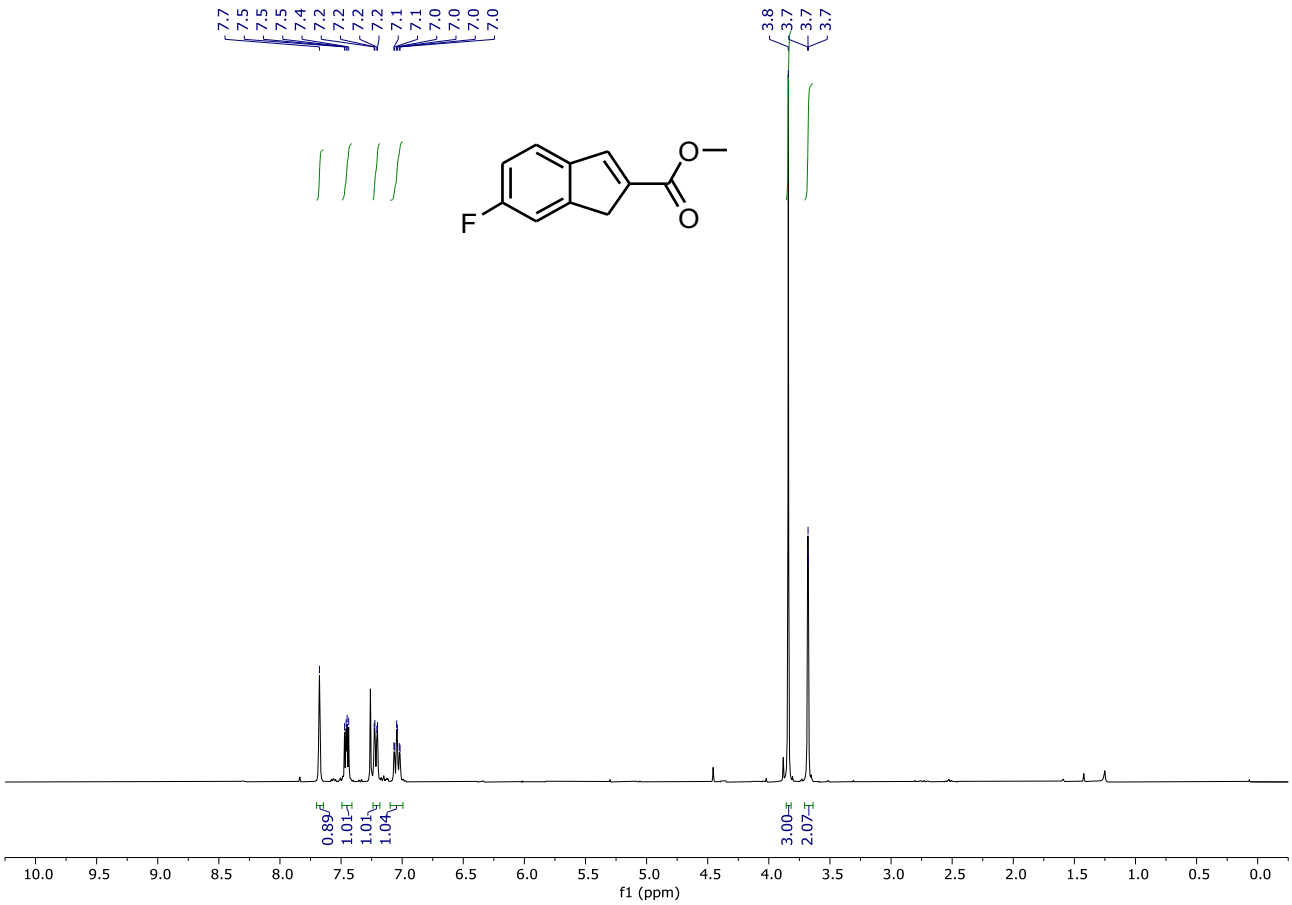
2g ^{1}H NMR (600 MHz, $CDCl_3$)



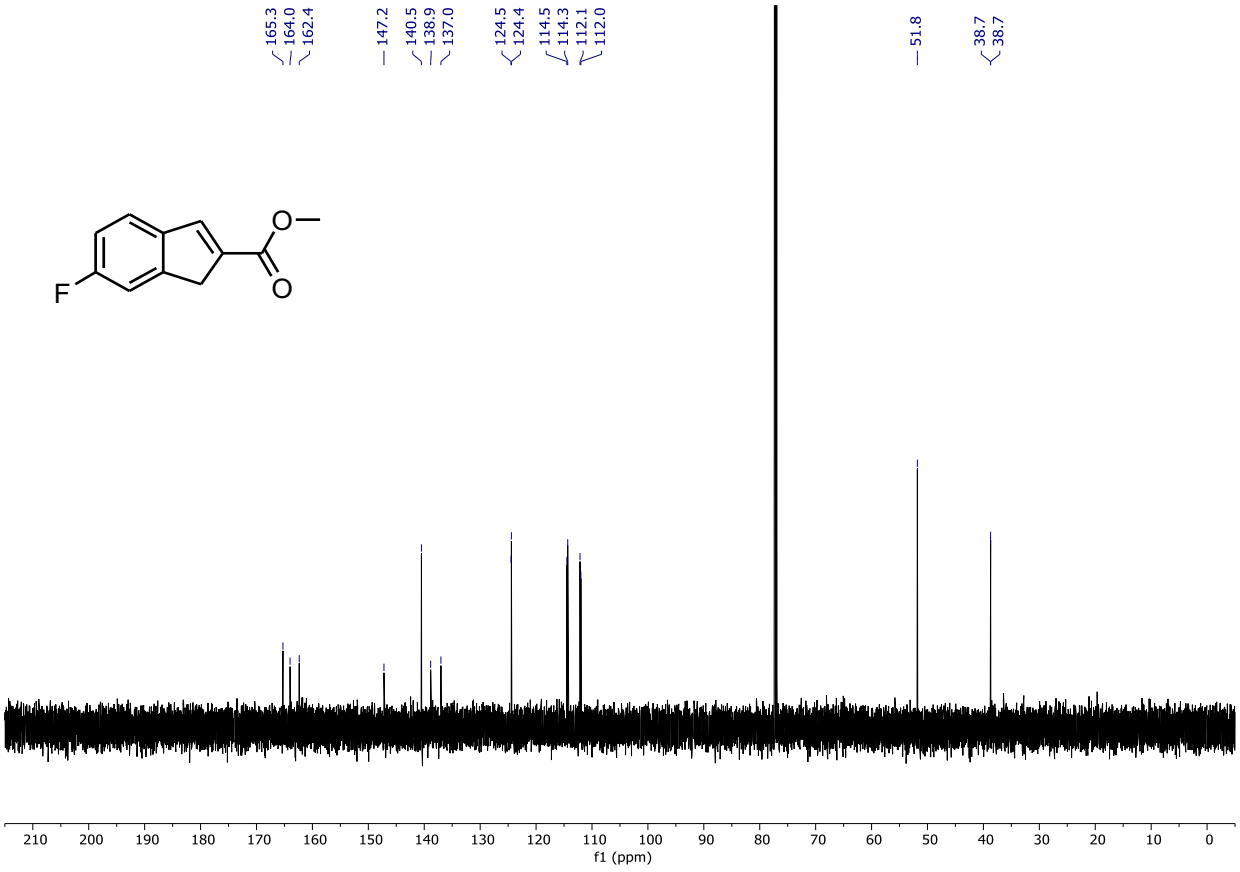
2g ^{13}C NMR (151 MHz, $CDCl_3$)



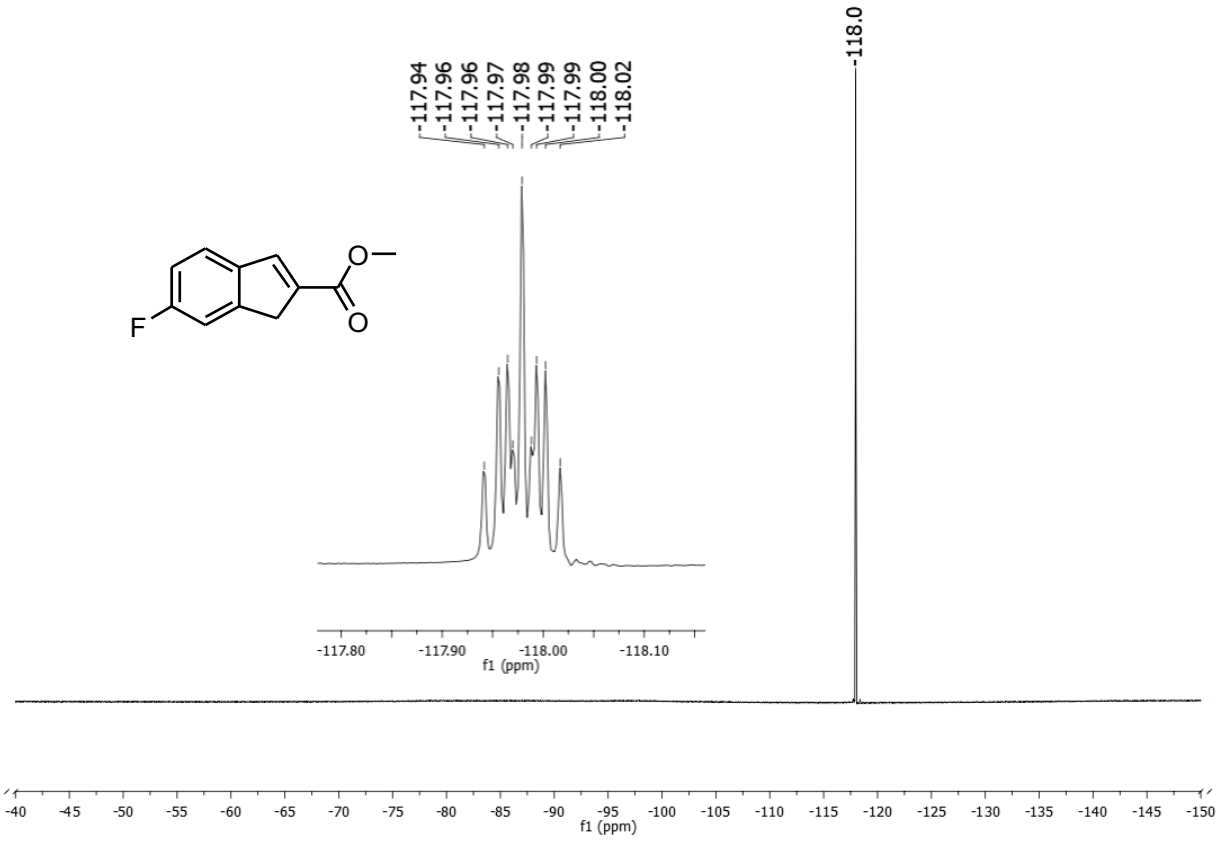
2h ^{1}H NMR (600 MHz, $CDCl_3$)



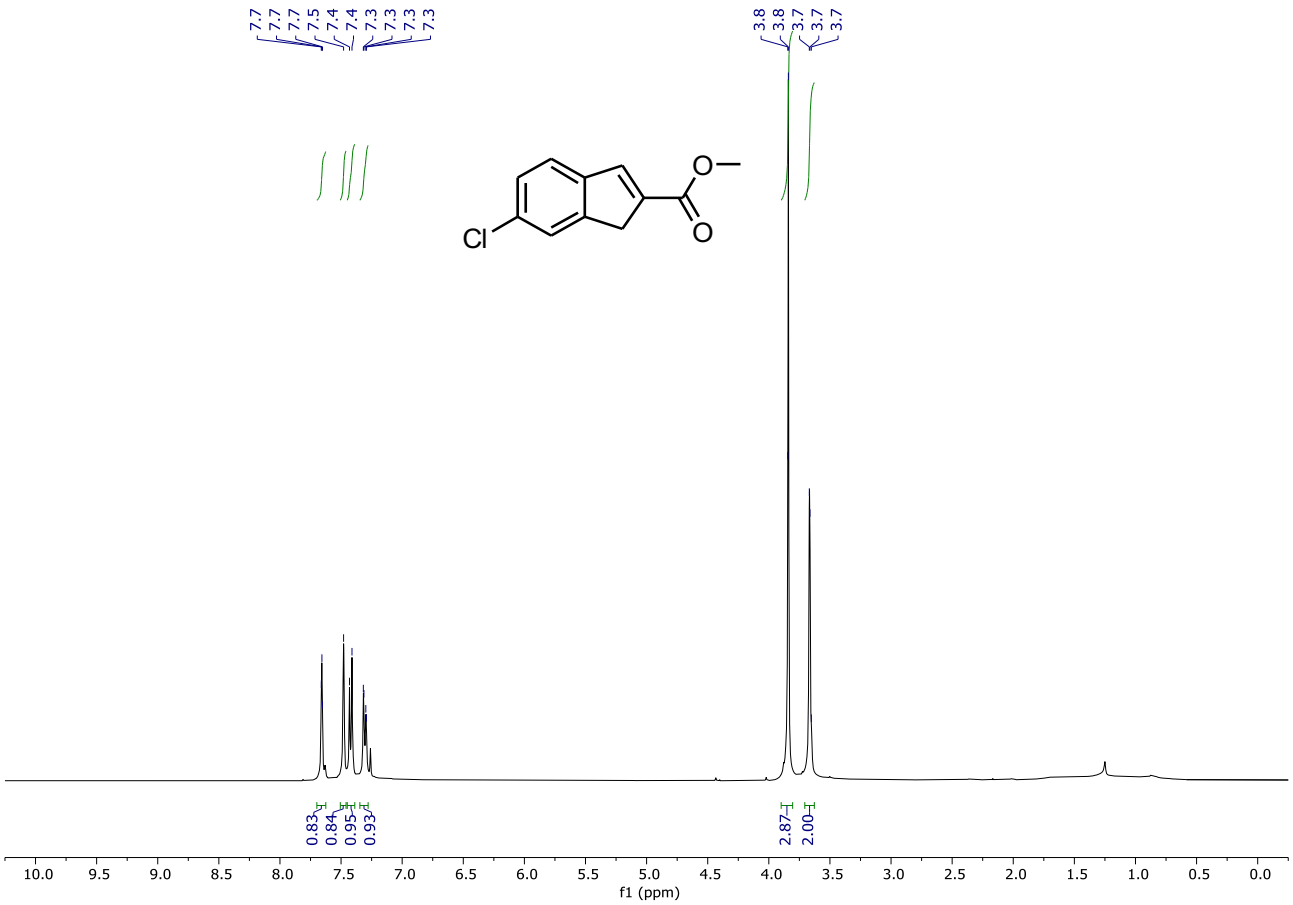
2h ^{13}C NMR (151 MHz, $CDCl_3$)



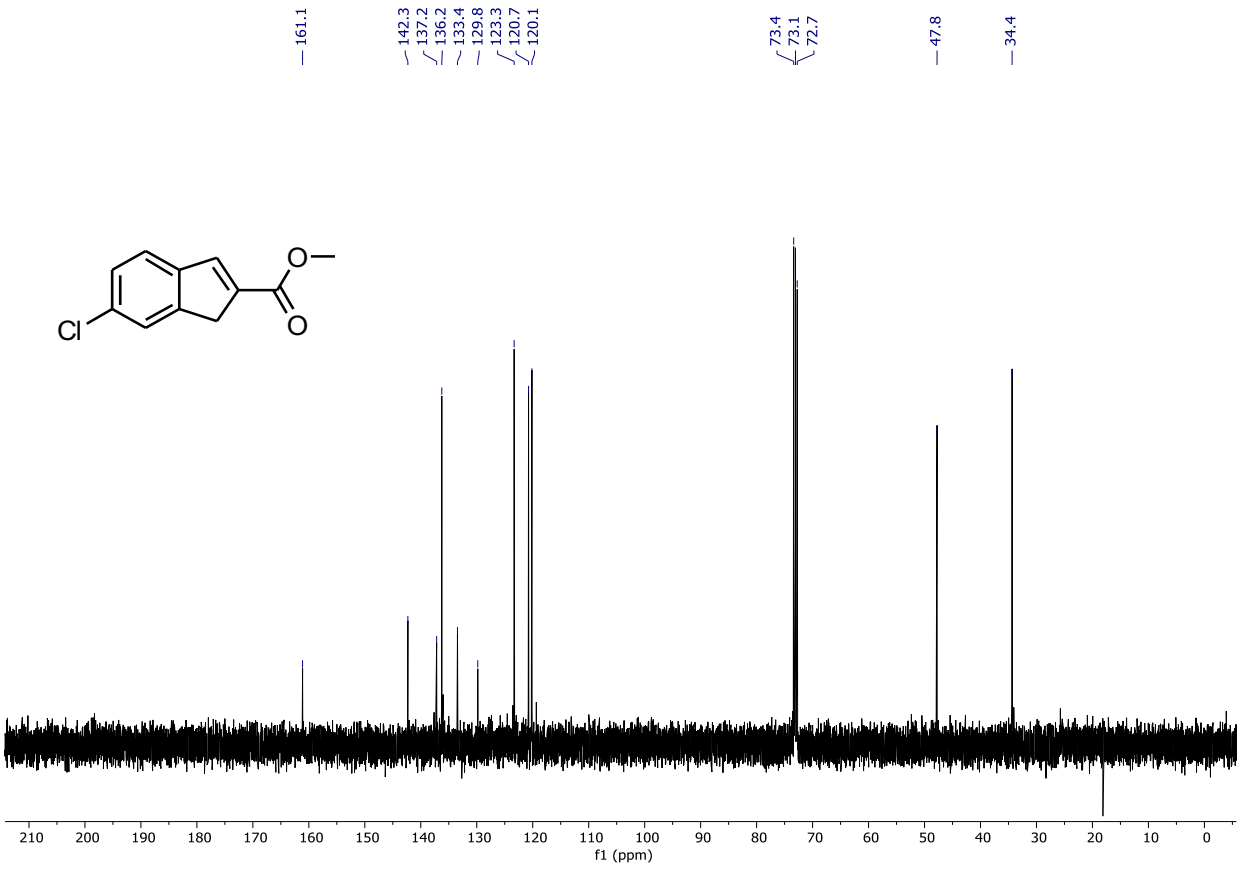
2h ^{19}F NMR (564 MHz, $CDCl_3$)



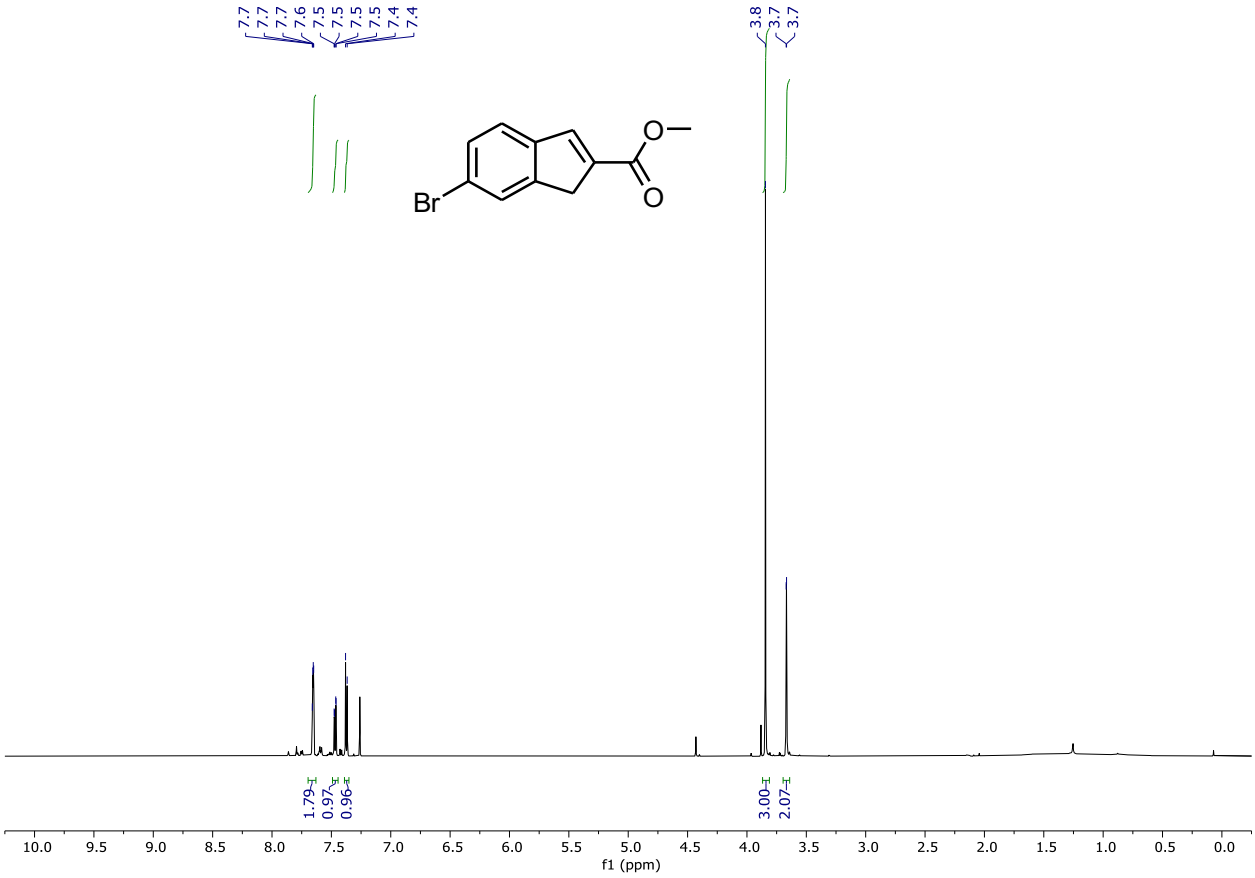
2i ^{1}H NMR (400 MHz, $CDCl_3$)



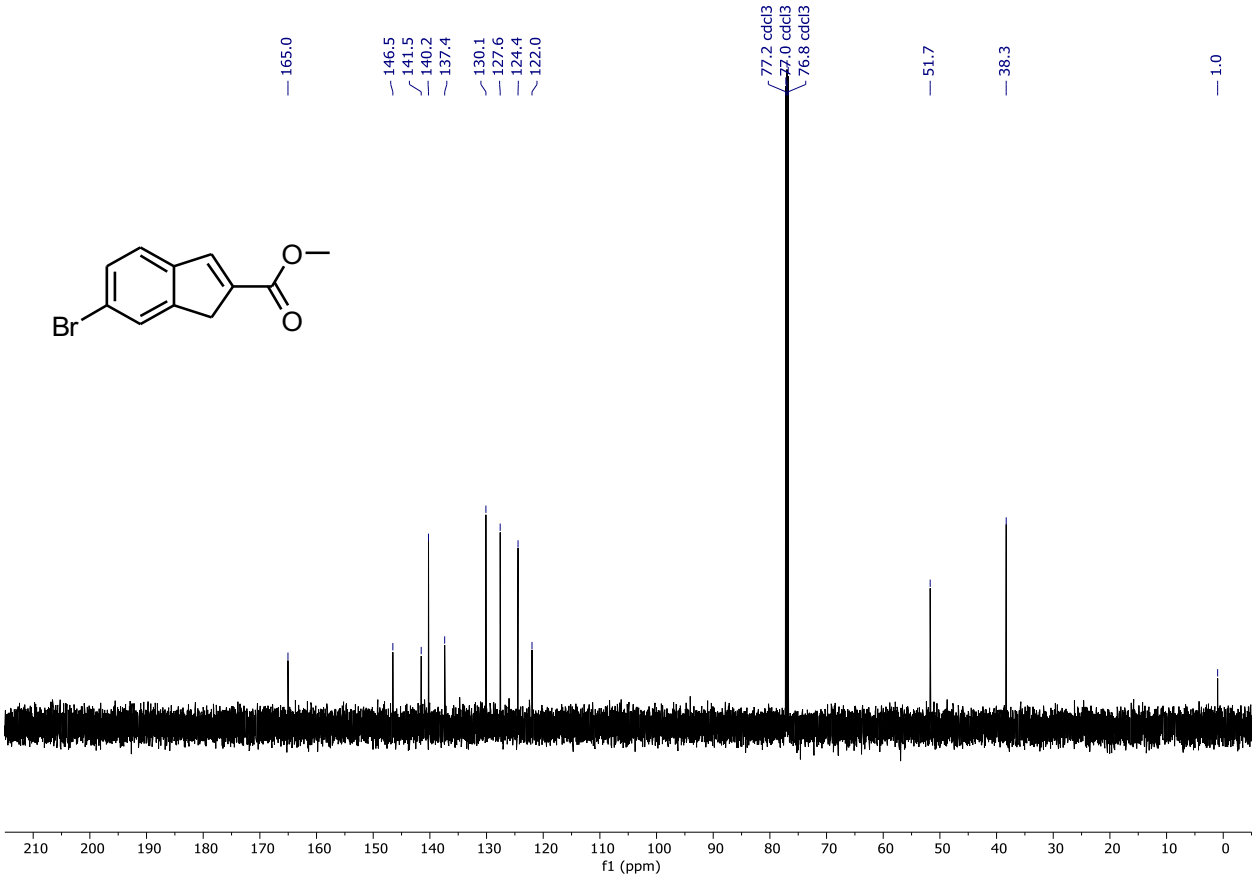
2i ^{13}C NMR (101 MHz, $CDCl_3$)



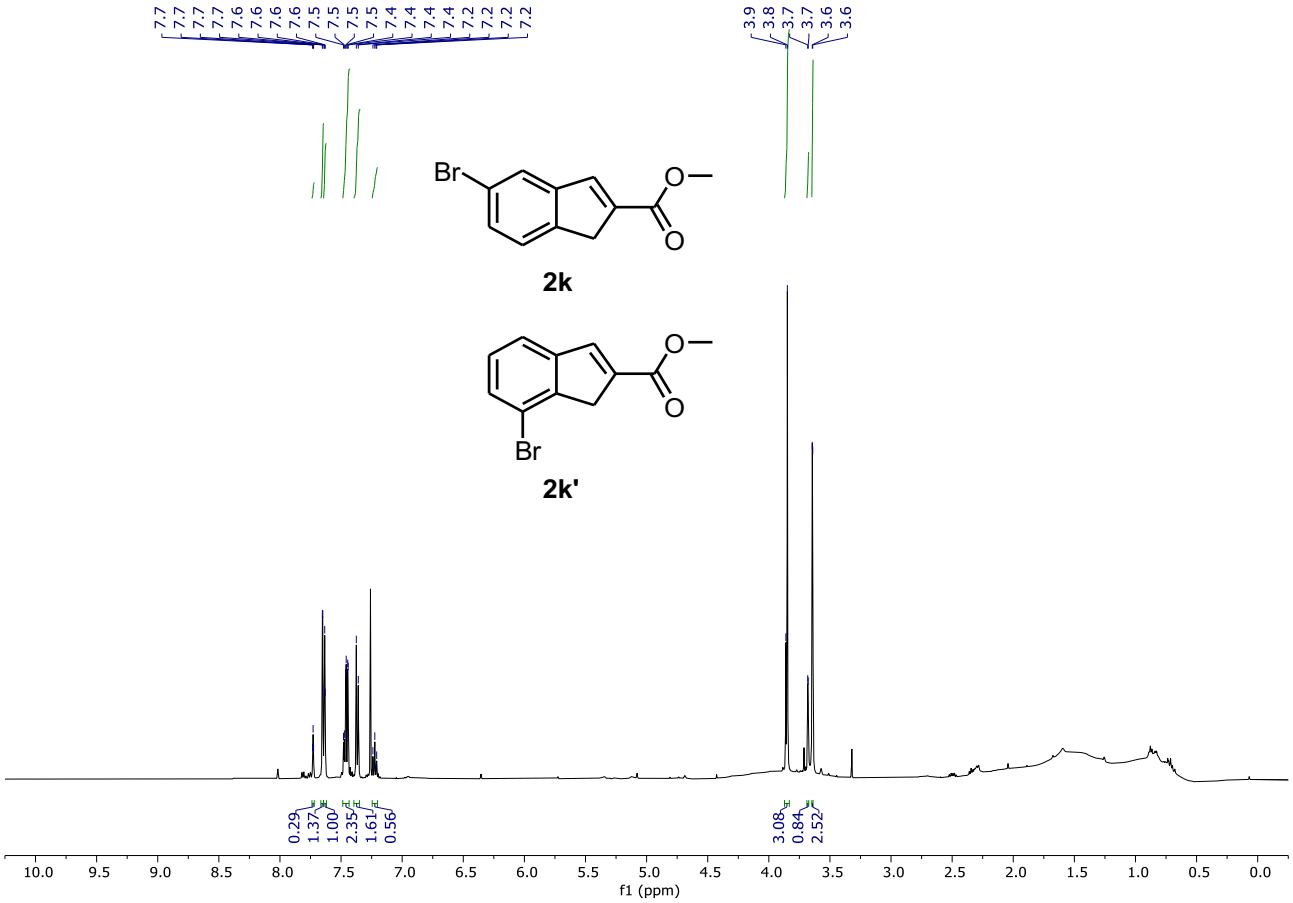
2j ^{1}H NMR (600 MHz, $CDCl_3$)



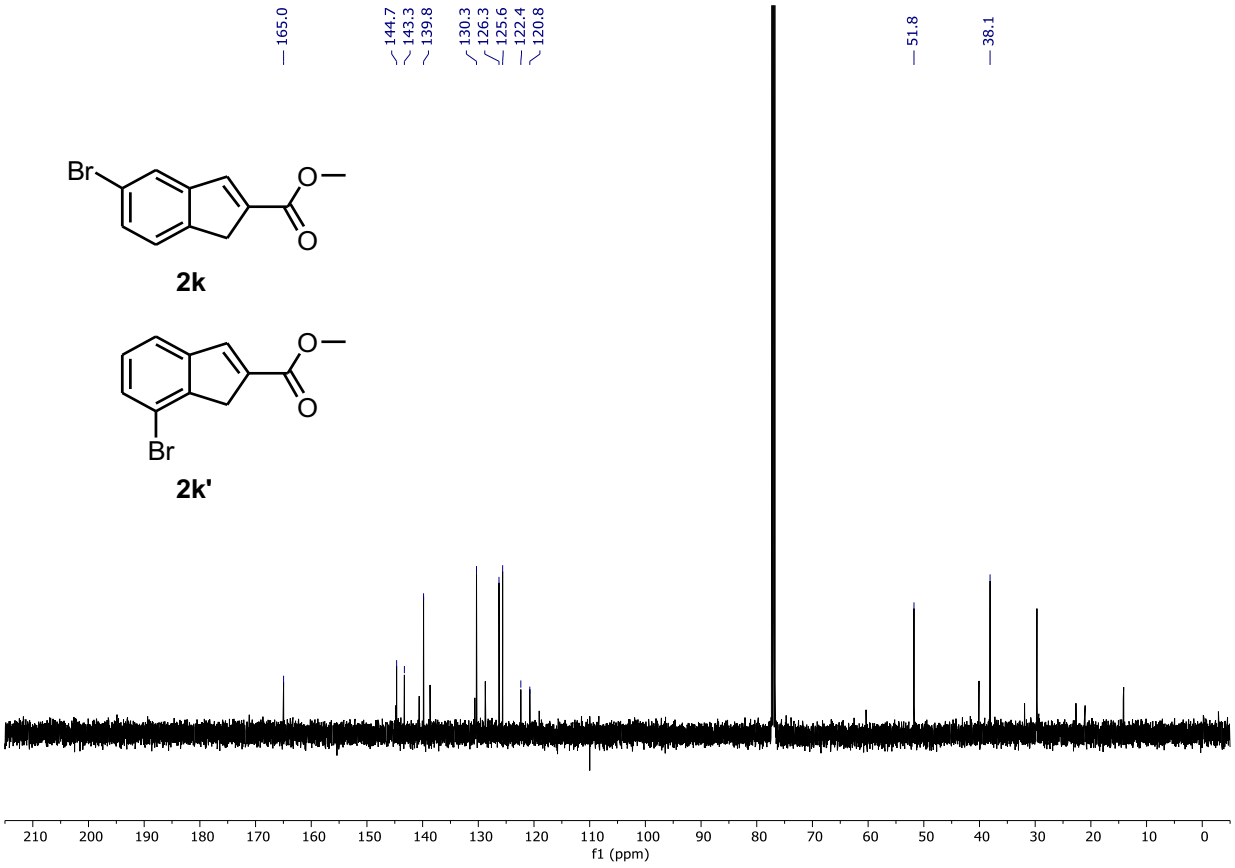
2j ^{13}C NMR (151 MHz, $CDCl_3$)



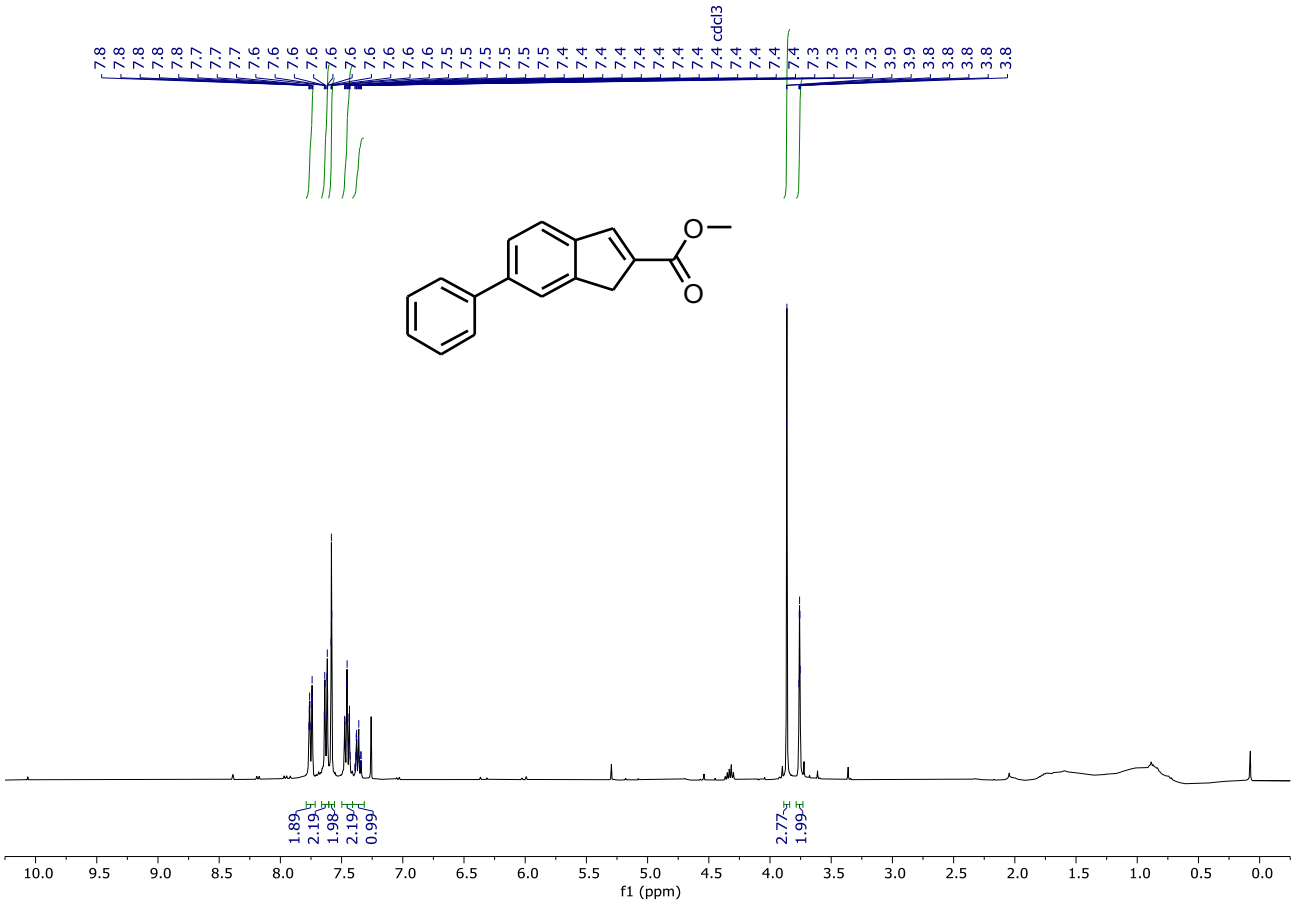
2k and 2k' ^{1}H NMR (400 MHz, $CDCl_3$)



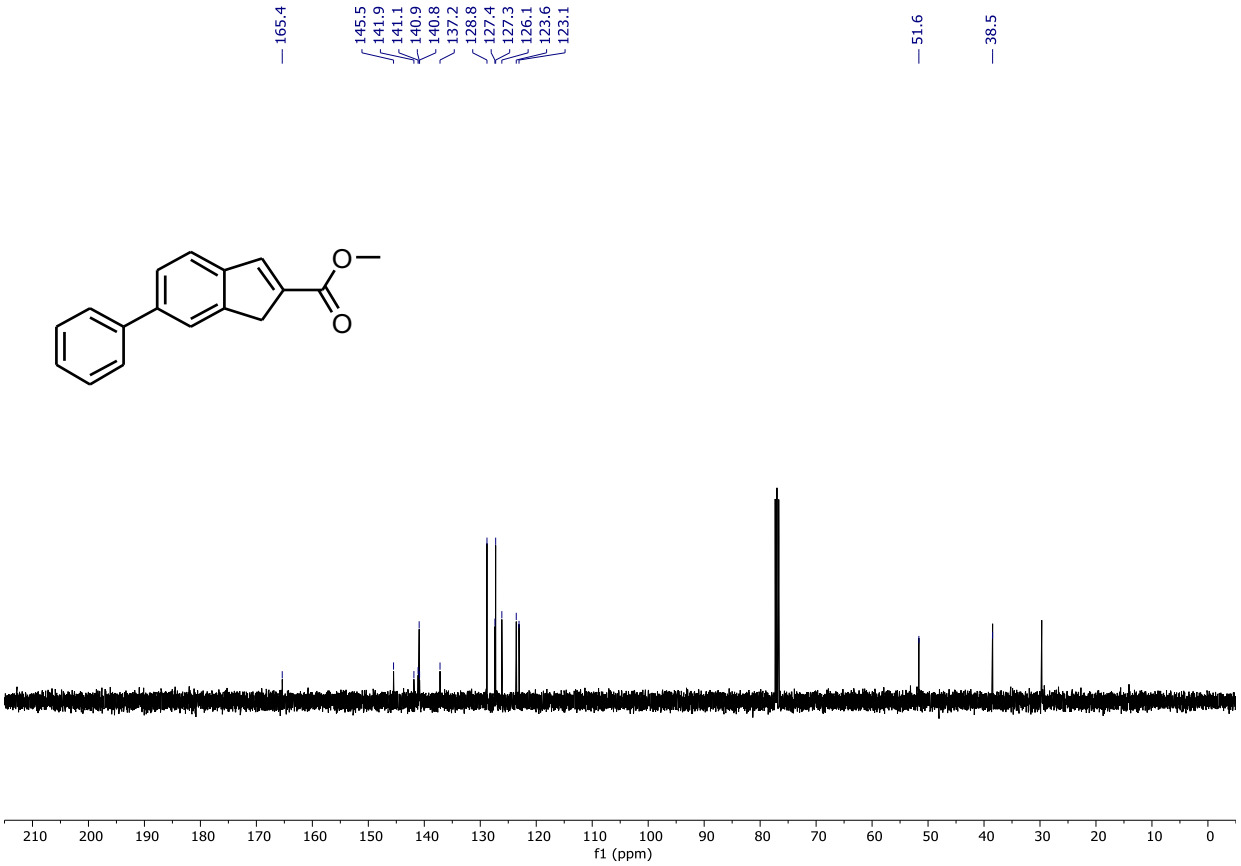
2k and 2k' ^{13}C NMR (100 MHz, $CDCl_3$)



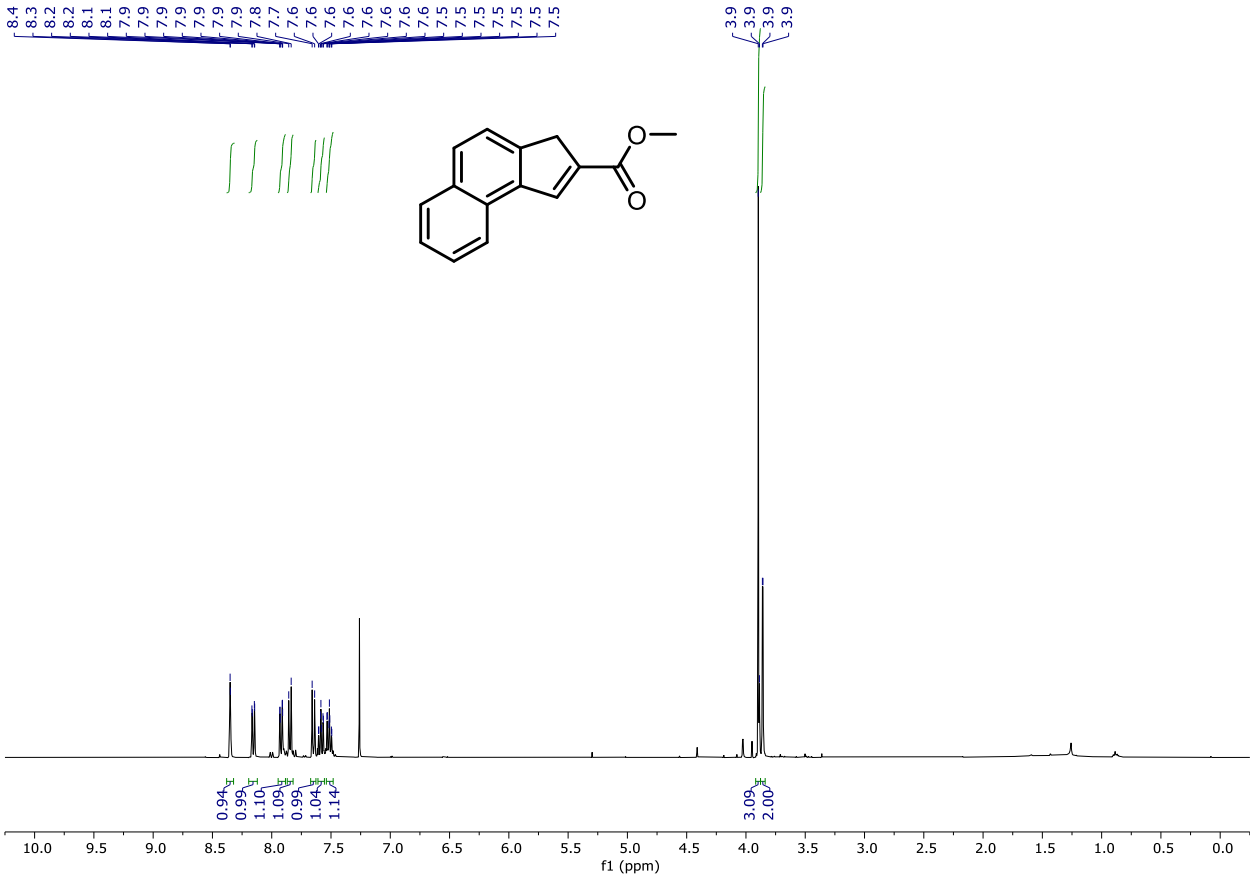
2l ^{1}H NMR (400 MHz, $CDCl_3$)



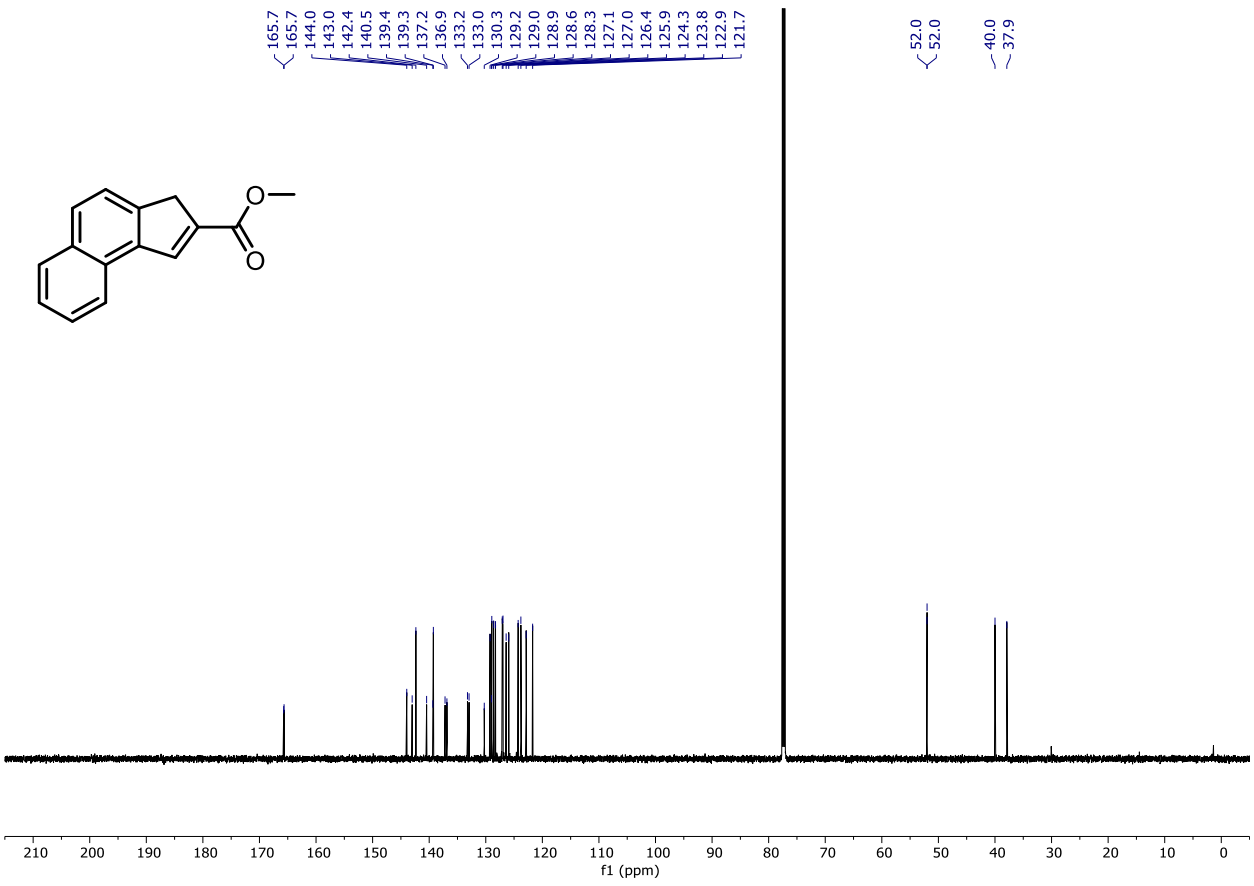
2l ^{13}C NMR (100 MHz, $CDCl_3$)



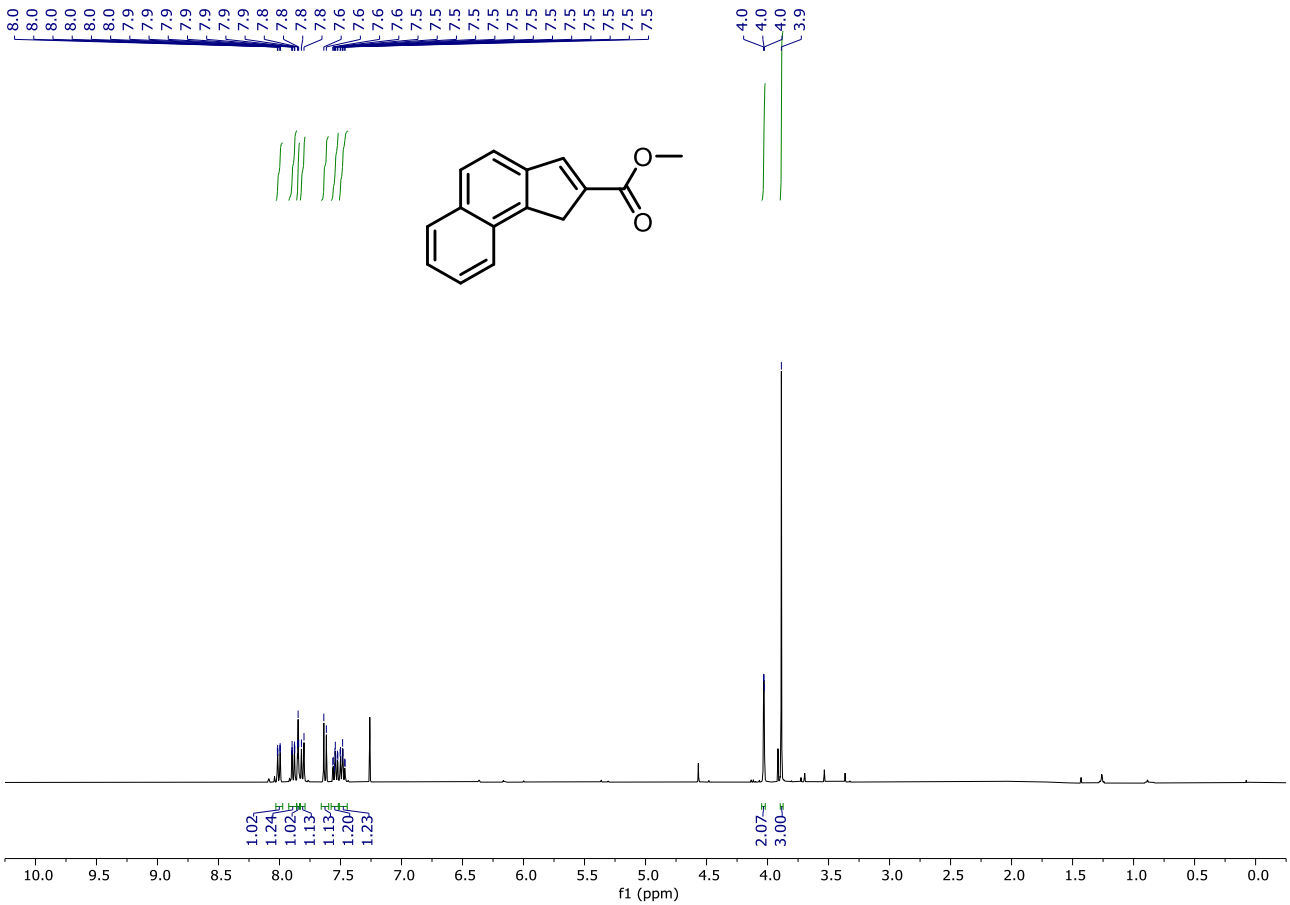
2m ^{1}H NMR (400 MHz, $CDCl_3$)



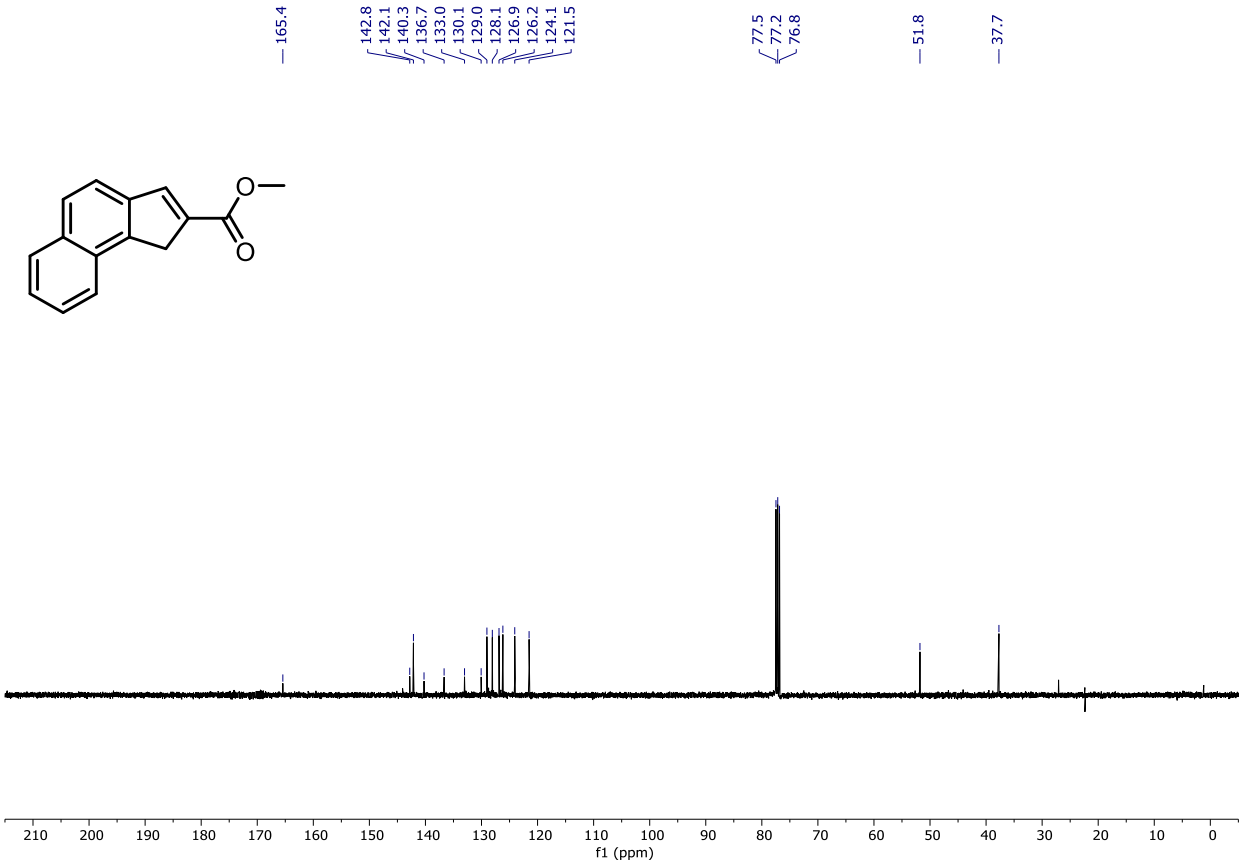
2m ^{13}C NMR (101 MHz, $CDCl_3$)



2n ^{1}H NMR (600 MHz, $CDCl_3$)



2n ^{13}C NMR (151 MHz, $CDCl_3$)



Reference

[17] C. Rasson, A. Stouse, A. Boreux, V. Cirriez, O. Riant, *Chem. Eur. J.* **2018***,* 24*,* 9234-9237.

[18] E. Jacobsen, M. K Chavda, K. M. Zikpi, S. L. Waggoner, D. J. Passini, J. A. Wolfe, R. Larson, C. Beckley, C. G. hamaker, S.R. Hitchcock, *Tetrahedron Lett.* **2017**, *58*, 3073–3077.

[19] M. M. Lorion, D. Gasperini, J. Oble, G. Poli, *Org. Lett.* **2013**, *15*, 3050-3053.

[20] V. Dočekal, M. Šimek, M. Dračinský, J. Vaselý, *Chem. Eur. J.* **2018**, *24*, 13441-13445.

[21] H. J. Hamanna, N. S. Abutalebb, R. Palb, M. N. Seleemb, P. V. Ramachandran, *Bioorg. Chem.* **2020**, 104, 104183.

[22] A. Kovtun, D. Jones, S. Dell'Elce, E. Treossi, A. Liscio, V. Palermo, *Carbon* **2019**, 143, 268-275.