Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information

Electrochemical ring-opening carboxylation of cyclic carbonate with carbon dioxide

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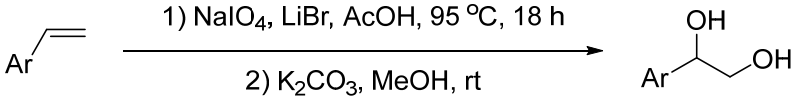
1. General information

Unless otherwise stated, all manipulations were performed using standard Schlenk techniques under a dry argon or carbon dioxide atmosphere. DMF and DCM were distilled with CaH_2. THF and toluene were distilled from sodium/benzophenone. All the solvents were stored over 4A molecular sieves before used. NMR spectra were recorded on 400 M, 600 M (**^{1}H NMR**, 400 MHz, 600 MHz; **^{13}C NMR**, 101 MHz, 151 MHz; **^{19}F NMR**, 376 MHz) spectrometer in $CDCl_3$, DMSO-d_6 or CD_3OD at ambient temperature and chemical shifts are expressed in parts per million (δ, ppm). Proton chemical shifts are referenced to 7.26 ppm ($CDCl_3$), 2.50 ppm (DMSO-d_6) or 3.30 ppm (CD_3OD) and carbon chemical shifts are referenced to 77.0 ppm ($CDCl_3$) or 39.5 ppm (DMSO-d_6) or 49.0 ppm (CD_3OD). High resolution mass spectra (HRMS) were recorded on a Q-TOF mass spectrometry equipped with Z-spray ionization source. Infrared spectra (IR) were measured using a Nicolet NEXUS FT-IR spectrophotometer. Carbon dioxide (99.999%), and other commercially available reagents were purchased from TCI, Sigma-Aldrich, Alfa Aesar, Acros, Adamas, Energy Chemical, or Innochem and used directly without further purification.

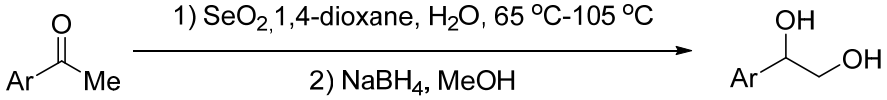
2. General procedure for the preparation of substrates

2.1 General method A for preparation of diol intermediate [1]



A mixture of olefin (7.5 mmol), $NaIO_4$ (30 mol %), and LiBr (20 mol %) was taken in a 100 mL round bottomed flask, and glacial acetic acid (10 mL) was added. The reaction mixture was heated at 95 °C (using an oil bath) for 18 h. The light-yellow colored reaction mixture turned purple after completion of the reaction. The reaction mixture was cooled and then extracted with EtOAc (30 mL × 3), and the combined organic phase was washed with saturated sodium thiosulfate solution, water, and aqueous $NaHCO_3$. The organic layer was dried over anhydrous Na_2SO_4 and concentrated under reduced pressure to give crude product, which was subjected to basic hydrolysis without purification. The hydrolysis was carried out by stirring the reaction mixture with K_2CO_3 (1.5 equiv.) in methanol (20 mL) at 25 °C for 24 h. After completion of the reaction, methanol was removed under reduced pressure, and the reaction mixture was extracted with EtOAc (30 mL × 3). The combined organic phase was washed with water and brine. The organic layer was then dried over anhydrous Na_2SO_4 and concentrated under reduced pressure to give crude diol, which was purified by column chromatography packed with silica gel (petroleum ether/ethyl acetate 7/3) to afford pure diol.

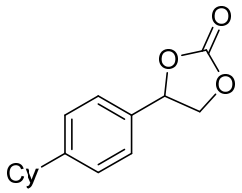
2.2 General method B for preparation of diol intermediate [2]



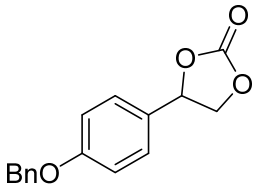
A 250 mL round bottom flask equipped with a magnetic stirring bar was charged with SeO_2 (2.8 g, 25 mmol), 1,4-dioxane (27 mL) and water (3 mL), and stirred at 65 °C until all SeO_2 was dissolved. Then the selected ketone (10 mmol) was added. The reaction mixture was then heated and stirred at 105 °C overnight. After reaction was completed, all the solvent was removed under vacuum, MeOH (30 mL) was then added to the reaction residue. Subsequently, $NaBH_4$ (0.9 g, 25 mmol) was added to the mixture slowly at 0 °C. The reaction mixture was stirred at room temperature for additional 3 h. The solvent was then removed under vacuo and exacted with EtOAc. The organic phase was collected and dried over Na_2SO_4. Then, the filtrate concentrated under reduced pressure to give the residue which was purified via chromatography over silica gel (petroleum ether/ethyl acetate 2/1) to yield the desired glycols.

2.3 General method for preparation of cyclic carbonate substrates [4]

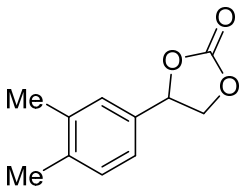
To a solution of diphenyl carbonate (1.0 equiv.) and a vicinal diol (1.1 equiv.) in 2-Me-THF (5 mL) was added TBD (2.0 mol %). The resultant mixture was stirred in an oil bath at the indicated temperature. After completion of the reaction as determined by TLC, drop of acetic acid was added to the reaction mixture. The obtained mixture was directly purified by flash column chromatography on silica gel to afford the desired cyclic carbonate (PE: ethyl acetate was gradually changed from 9:1 to 4:1). When TLC showed that a cyclic carbonate is essentially copular with phenol, the following procedure was applied just prior to flash column chromatography to remove phenol. Upon completion of the reaction, the reaction mixture was diluted with ethyl acetate. The mixture was washed successively with 35% NaOH solution (10 mL × 2), water (10 mL × 2), and brine (10 mL), dried over anhydrous $MgSO_4$, filtered, and concentrated using a rotary evaporator to afford the crude mixture.



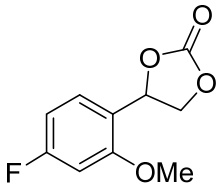
4-(4-Cyclohexylphenyl)-1,3-dioxolan-2-one (1d). White solid, 163 mg, 44% yield, m.p.: 104 – 106 ºC. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.30 (s, 4H), 5.66 (t, J = 8.0 Hz, 1H), 4.79 (t, J = 8.4 Hz, 1H), 4.37 (t, J = 8.3 Hz, 1H), 2.55 (tt, J = 10.7, 3.0 Hz, 1H), 1.91 – 1.84 (m, 4H), 1.78 (d, J = 12.2 Hz, 1H), 1.43 (td, J = 9.6, 3.7 Hz, 4H), 1.33 – 1.23 (m, 1H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 155.0, 150.1, 133.1, 127.8, 126.2, 78.2, 71.2, 44.4, 34.4, 26.9, 26.1. **HRMS** (ESI-TOF) *m/z*: $[M+Na]^+$ Calcd for $C_{15}H_{18}NaO_3$ 269.1153; Found 269.1152. **IR** (neat cm^{-1}) ν 2954, 1732, 2923, 1732, 1462, 805.



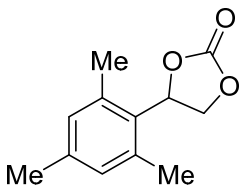
4-(4-(Benzyloxy) phenyl)-1,3-dioxolan-2-one (1f). White solid, 276 mg, 68% yield, m.p.: 124 – 126 ºC. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.44 – 7.29 (m, 7H), 7.03 (d, J = 8.4 Hz, 2H), 5.62 (t, J = 8.1 Hz, 1H), 5.09 (s, 2H), 4.75 (t, J = 8.4 Hz, 1H), 4.35 (t, J = 8.4 Hz, 1H). **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 159.8, 154.8, 136.3, 128.6, 128.1, 127.7, 127.6, 127.4, 115.5, 78.0, 71.0, 70.1. **HRMS** (ESI, m/z): $[M+Na]^+$ calcd for $C_{16}H_{14}NaO_4$ 293.0789; Found 293.0784. **IR** (neat cm^{-1}) ν 2958, 2922, 1735, 1511, 832.



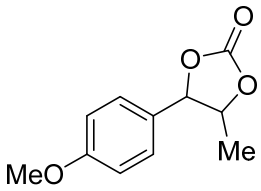
4-(3,4-Dimethylphenyl)-1,3-dioxolan-2-one (1h). White solid, 230 mg, 68% yield, m.p.: 52 – 54 ºC. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.19 (d, J = 7.7 Hz, 1H), 7.13 (s, 1H), 7.09 (d, J = 7.7 Hz, 1H), 5.61 (t, J = 8.0 Hz, 1H), 4.76 (t, J = 8.4 Hz, 1H), 4.34 (t, J = 8.2 Hz, 1H), 2.29 (d, J = 2.3 Hz, 6H). **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 154.9, 138.5, 137.7, 133.0, 130.3, 127.1, 123.4, 78.1, 71.1, 19.7, 19.5. **HRMS** (ESI-TOF) *m*/*z*: $[M+Na]^+$ Calcd for $C_{11}H_{12}NaO_3$ 215.0684; Found 215.0682. **IR** (neat cm^{-1}) *v* 2958, 2923, 1802, 1379, 822.



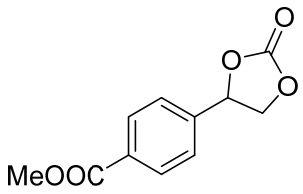
4-(4-Fluoro-2-methoxyphenyl)-1,3-dioxolan-2-one (1i). Colorless oil, 247 mg, 58% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.32 (ddd, J = 8.5, 6.4, 0.8 Hz, 1H), 6.82 – 6.59 (m, 2H), 5.82 – 5.74 (t, J = 8 Hz, 1H), 4.81 (t, J = 8.5 Hz, 1H), 4.26 (dd, J = 8.4, 7.2 Hz, 1H), 3.85 (s, 3H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 165.4, 162.9, 157.6 (d, J = 10.2 Hz), 154.9, 120.5 (d, J = 3.0 Hz), 107.2 (d, J = 21.5 Hz), 99.4 (d, J = 26.4 Hz), 74.6, 70.3, 55.8, 30.8. **^{19}F NMR** (376 MHz, Chloroform-*d*) δ -109.09. **HRMS** (ESI-TOF) *m*/*z*: $[M+Na]^+$ Calcd for $C_{10}H_9FNaO_4$ 235.0382; Found 235.0379. **IR** (neat cm^{-1}) *v* 2960, 1809, 1732, 1283, 830,770.



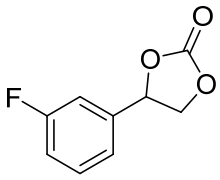
4-Mesityl-1,3-dioxolan-2-one (1j). White solid, 258 mg, 85% yield, m.p.: 105 – 107 ºC**. ^{1}H NMR** (400 MHz, Chloroform-*d*) δ 6.89 (s, 2H), 6.12 (t, J = 9.3 Hz, 1H), 4.72 (t, J = 8.9 Hz, 1H), 4.36 (dd, J = 9.5, 8.6 Hz, 1H), 2.36 (s, 6H), 2.28 (s, 3H). **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 155.1, 139.3, 136.7, 130.7, 127.4, 75.3, 68.8, 20.8, 20.1. **HRMS** (ESI-TOF) *m*/*z*: $[M+Na]^+$ Calcd for $C_{12}H_{14}NaO_3$ 229.0840; Found 229.0836. **IR** (neat cm^{-1}) *v* 2955, 2923, 1788, 1611, 1378, 853, 770.



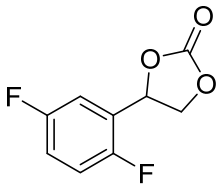
4-(4-Methoxyphenyl)-5-methyl-1,3-dioxolan-2-one (1m). *Trans*, colorless oil, 124 mg, 59% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.39 – 7.15 (m, 2H), 7.04 – 6.74 (m, 2H), 5.07 (d, J = 8.1 Hz, 1H), 4.60 (t, J = 6.5 Hz, 1H), 3.82 (d, J = 1.7 Hz, 3H), 1.52 (dd, J = 6.3, 1.8 Hz, 3H). **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 160.6, 154.3, 127.8, 126.6, 114.5, 84.9, 80.6, 55.3, 18.0. **HRMS** (ESI-TOF) *m*/*z*: $[M+Na]^+$ Calcd for $C_{11}H_{12}NaO_4$ 231.0633; Found 231.0625. **IR** (neat cm^{-1}) *v* 2923, 1798, 1613, 1251, 832



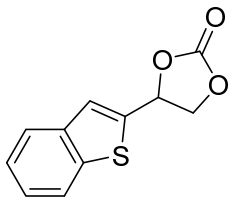
Methyl 4-(2-oxo-1,3-dioxolan-4-yl) benzoate (1q). White solid, 357 mg, 62%, m.p.: 106 – 108 ºC. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 8.11 (d, J = 8.0 Hz, 2H), 7.44 (d, J = 8.0 Hz, 2H), 5.74 (t, J = 8.0 Hz, 1H), 4.85 (t, J = 8.5 Hz, 1H), 4.32 (t, J = 8.2 Hz, 1H), 3.94 (s, 3H). **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 166.2, 154.4, 140.5, 131.4, 130.5, 125.5, 77.1, 70.9, 52.4. **HRMS** (ESI-TOF) *m/z*: $[M+Na]^+$ Calcd for $C_{11}H_{10}NaO_5$ 245.0425; Found 245.0422. **IR** (neat cm^{-1}) *v* 2953, 2923, 1723, 1377, 1163,812.



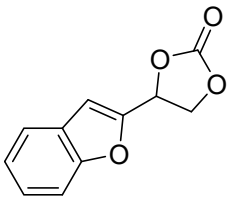
4-(3-Fluorophenyl)-1,3-dioxolan-2-one (1r). White solid, 284 mg, 78% yield, m.p.: 68 – 70 ºC. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.43 (td, J = 8.0, 5.7 Hz, 1H), 7.17 – 7.06 (m, 3H), 5.67 (t, J = 7.9 Hz, 1H), 4.82 (t, J = 8.4 Hz, 1H), 4.32 (t, J = 8.2 Hz, 1H). **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 163.0 (d, J = 248.4 Hz), 154.4, 138.3 (d, J = 7.1 Hz), 131.0 (d, J = 7.9 Hz), 122.5 – 119.2 (m), 116.6 (d, J = 21.5 Hz), 112.8 (d, J = 22.9 Hz), 70.9. **HRMS** (ESI, m/z): $[M+Na]^+$ calcd for $C_9H_7FNaO_3$ 205.0271; Found 205.0271. **IR** (neat cm^{-1}) *v* 2954, 2923, 1738, 1070, 783.



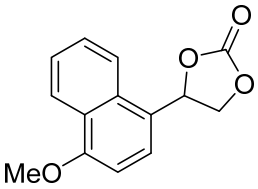
4-(2,5-Difluorophenyl)-1,3-dioxolan-2-one (1s). Colorless oil, 298 mg, 75% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.21 – 7.17 (m, 1H), 7.14 – 7.09 (m, 2H), 5.87 (t, J = 7.9 Hz, 1H), 4.89 (td, J = 8.6, 1.1 Hz, 1H), 4.34 (ddd, J = 8.5, 7.4, 0.9 Hz, 1H). **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 158.7 (d, J = 245.5 Hz), 155.5 (dd, J = 243.0, 3.0 Hz), 154.1, 125.2 (dd, J = 15.3, 7.6 Hz), 117.3 (ddd, J = 39.2, 23.9, 8.7 Hz), 113.7 (d, J = 4.2 Hz), 113.5 (d, J = 4.2 Hz), 72.4 (d, J = 2.9 Hz), 70.0 (d, J = 3.2 Hz). **^{19}F NMR** (565 MHz, Chloroform-*d*) δ -116.57 (d, J = 17.9 Hz), -124.09 (d, J = 17.4 Hz). **HRMS** (ESI, m/z): $[M+Na]^+$ calcd for $C_9H_6F_2NaO_3$ 223.0182; Found 223.0178. **IR** (neat cm^{-1}) *v* 2953, 2922, 1745, 1377, 680.



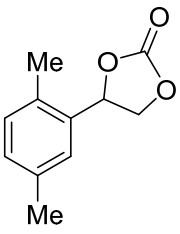
4-(Benzo[b]thiophen-2-yl)-1,3-dioxolan-2-one (1v). White solid, 313mg, 71% yield, m.p.: 143 – 145 ºC. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.87 – 7.84 (m, 2H), 7.43 – 7.40 (m, 3H), 5.99 (ddd, J = 8.1, 7.4, 0.8 Hz, 1H), 4.84 (dd, J = 8.8, 8.0 Hz, 1H), 4.57 (dd, J = 8.8, 7.4 Hz, 1H)**.** **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 154.0, 139.9, 138.7, 138.1, 125.6, 125.0, 124.3, 122.7, 74.5, 70.5. **HRMS** (ESI-TOF) *m/z*: $[M+Na]^+$ Calcd for $C_{11}H_8NaO_3S$ 243.0091; Found 243.0090. **IR** (neat cm^{-1}) *v* 2955, 1784, 1458, 1069, 748.



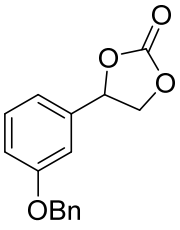
4-(Benzofuran-2-yl)-1,3-dioxolan-2-one (1w). White solid, 354 mg, 87% yield, m.p.: 129 – 131 ºC. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.61 (ddd, J = 7.8, 1.4, 0.7 Hz, 1H), 7.52 (dq, J = 8.3, 0.9 Hz, 1H), 7.38 (ddd, J = 8.4, 7.3, 1.4 Hz, 1H), 7.29 (td, J = 7.6, 1.0 Hz, 1H), 6.94 (s, 1H), 5.81 (t, J = 7.7 Hz, 1H), 4.78 (d, J = 1.3 Hz, 1H), 4.76 (d, J = 0.7 Hz, 1H). **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 155.4, 154.1, 149.4, 127.0, 125.9, 123.5, 121.8, 111.7, 108.1, 71.3, 67.4. **HRMS** (ESI, m/z): $[M+Na]^+$ Calcd for $C_{11}H_8NaO_4$ 227.0320; Found 227.0321. **IR** (neat cm^{-1}) v 2955, 2923, 2360, 1804, 1732, 752.



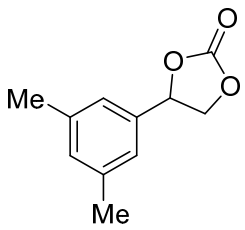
4-(4-Methoxynaphthalen-1-yl)-1,3-dioxolan-2-one (1x). White solid, 255 mg, 52%, m.p.: 90 – 92 ºC. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 8.39 – 8.36 (m, 1H), 7.64 – 7.55 (m, 4H), 6.84 (d, J = 8.1 Hz, 1H), 6.33 (t, J = 7.4 Hz, 1H), 4.99 (t, J = 8.3 Hz, 1H), 4.42 (dd, J = 8.5, 7.5 Hz, 1H), 4.03 (s, 3H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 156.5, 154.9, 130.4, 127.6, 125.9, 125.6, 123.4, 123.1, 121.3, 102.9, 75.8, 70.6, 55.6, 30.8. **HRMS** (ESI-TOF) m/z: $[M+Na]^+$ Calcd for $C_{14}H_{12}NaO_4$ 267.0633; Found 267.0625. **IR** (neat cm^{-1}) v 2955, 2932, 1804, 1734, 764.



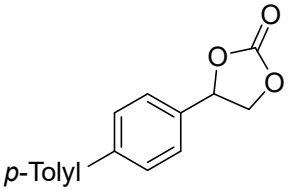
4-(2,5-Dimethylphenyl)-1,3-dioxolan-2-one (1ab). Colorless oil, 273 mg, 71% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.23 (s, 1H), 7.10 (s, 2H), 5.87 (t, J = 8.0 Hz, 1H), 4.81 (t, J = 8.3 Hz, 1H), 4.26 (t, J = 8.1 Hz, 1H), 2.35 (s, 3H), 2.26 (s, 3H). **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 154.9, 136.5, 133.8, 131.2, 130.9, 129.8, 125.2, 75.5, 70.4, 21.0, 18.4. **HRMS** (ESI-TOF) m/z: $[M+Na]^+$ Calcd for $C_{11}H_{12}NaO_3$ 215.0684; Found 215.0681. **IR** (neat cm^{-1}) v 2954, 1804, 1731, 1461, 1378, 880.



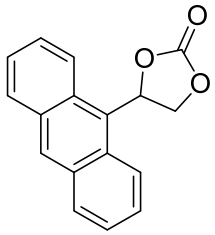
4-(3-(Benzyloxy) phenyl)-1,3-dioxolan-2-one (1ad). White solid, 214 mg, 40% yield, m.p.: 76 – 78 ºC. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.45 – 7.33 (m, 6H), 7.02 – 6.92 (m, 3H), 5.64 (t, J = 8.0 Hz, 1H), 5.08 (s, 2H), 4.78 (t, J = 8.4 Hz, 1H), 4.31 (dd, J = 8.6, 7.8 Hz, 1H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 159.3, 154.7, 137.4, 136.3, 130.4, 128.6, 128.1, 127.4, 118.0, 115.9, 112.2, 77.6, 71.0, 70.1. **HRMS** (ESI-TOF) m/z: $[M+Na]^+$ Calcd for $C_{16}H_{14}NaO_4$ 293.0789; Found 293.0781.**IR** (neat cm^{-1}) v 2954, 1801, 1733, 1601, 1291,876.



4-(3,5-Dimethylphenyl)-1,3-dioxolan-2-one (1ae). White solid, 264 mg, 69% yield, m.p.: 58 – 60 ºC. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.04 (s, 1H), 6.96 (s, 2H), 5.59 (t, J = 8.0 Hz, 1H), 4.76 (t, J = 8.4 Hz, 1H), 4.33 (dd, J = 8.6, 7.8 Hz, 1H), 2.34 (s, 6H). **^{13}C NMR** (151 MHz, Chloroform-*d*) δ 155.0, 139.0, 135.7, 131.3, 123.5, 78.1, 71.2, 21.2. **HRMS** (ESI-TOF) *m*/*z*: $[M+Na]^+$ Calcd for $C_{11}H_{12}NaO_3$ 215.0684; Found 215.0679. **IR** (neat cm^{-1}) *v* 2958, 2922, 1745, 1377, 770.

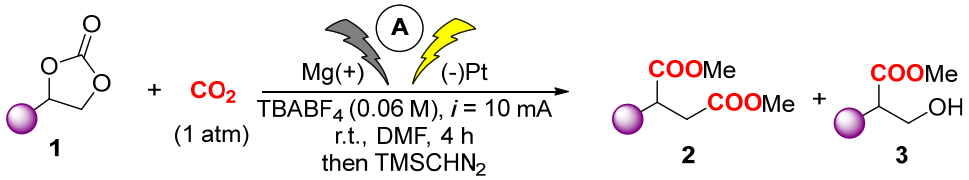


4-(4'-Methyl-[1,1'-biphenyl]-4-yl)-1,3-dioxolan-2-one (1ag). White solid, 137 mg, 54% yield, m.p.: 166 – 168 ºC. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.66 – 7.64 (m, 2H), 7.50 – 7.41 (m, 4H), 7.27 (d, J = 8.5 Hz, 2H), 5.72 (t, J = 8.0 Hz, 1H), 4.82 (t, J = 8.4 Hz, 1H), 4.39 (dd, J = 8.6, 7.9 Hz, 1H), 2.41 (s, 3H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 154.77, 142.72, 137.79, 137.10, 134.22, 129.64, 127.69, 126.94, 126.37, 77.89, 71.10, 21.10. **HRMS** (ESI-TOF) *m*/*z*: $[M+Na]^+$ Calcd for $C_{16}H_{14}NaO_3$ 277.0840; Found 277.0833. **IR** (neat cm^{-1}) *v* 2953, 2922, 2360, 1790,1167, 808.



4-(Anthracen-9-yl)-1,3-dioxolan-2-one (1ai). Yellow solid, 212 mg, 52% yield, m.p.: 144 – 146 ºC. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 8.55 (s, 1H), 8.16 (d, J = 9.0 Hz, 2H), 8.06 (d, J = 7.9 Hz, 2H), 7.61 – 7.50 (m, 4H), 7.20 (t, J = 9.6 Hz, 1H), 4.95 (t, J = 8.9 Hz, 1H), 4.84 (dd, J = 10.2, 8.7 Hz, 1H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 155.1, 131.3, 131.1, 130.2, 129.9, 127.5, 125.1, 122.7, 122.2, 74.9, 69.7. **HRMS** (ESI, m/z): $[M+Na]^+$ calcd for $C_{17}H_{12}NaO_3$ 287.0684; Found 287.0683. **IR** (neat cm^{-1}) *v* 2925, 1805, 1168, 1377, 730.

3. General procedure for the electrocarboxylation reactions



A 50 mL oven-dried electrochemical cell containing a stir bar was evacuated and back-filled under CO_2 flow for at least 3 times. **1** (0.3 mmol), $TBABF_4$ (0.3 mmol) and DMF (5 mL) were added to the cell and the cell was installed with a Pt cathode and Mg anode, and the cell was closed under CO_2 atmosphere. Then, the reaction mixture was electrolyzed under a constant current of 10 mA for 4 h. The reaction mixture was carefully quenched with HCl (1M, 10 mL) and extracted three times with ethyl acetate (3 × 20 mL). The combined organic phase was dried by anhydrous $MgSO_4$ and evaporated in vacuum. The residue was methylated at 0 °C with $TMSCHN_2$ (2 M in hexane, 0.6 mL) in 2 mL of ether/methanol (1:1 v/v) solvent. Finally, the corresponding methylation product was purified by column chromatography (10:1-3:1 v/v petroleum ether/ethyl acetate).

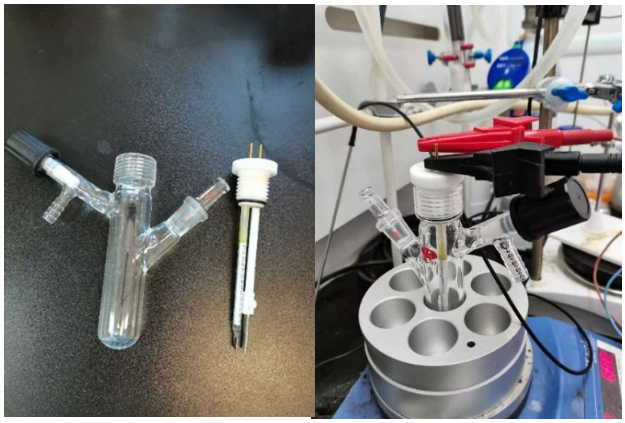


Figure S1. Photos for the electrocaboxylation. Pt (10 mm × 10 mm × 0.3 mm), Mg (20 mm × 10 mm × 0.5 mm). Electrochemical cell: 50 mL.

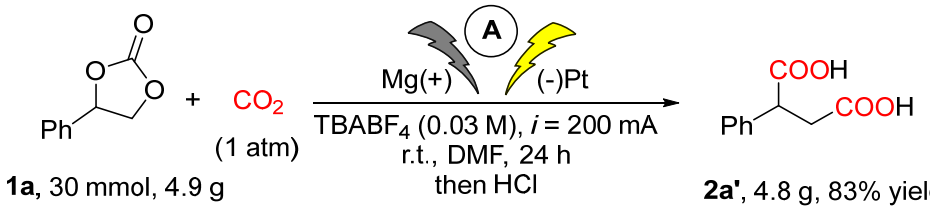
4. Optimization of electrocarboxylation of cyclic carbonate

Table S1. Optimization of electrocarboxylation of cyclic carbonate. [a]

Entry	Anode	Cathode	Electrolyte	T	Solvent	Current	t	Yield [b]
1	Pt	Mg	Bu_4NBF_4	rt	DMF	10 mA	4 h	89(78[c])
2	Pt	Zn	Bu_4NBF_4	rt	DMF	10 mA	4 h	64
3	Pt	Ni	Bu_4NBF_4	rt	DMF	10 mA	4 h	76
4	Ni	Mg	Bu_4NBF_4	rt	DMF	10 mA	4 h	37
5	Cu	Mg	Bu_4NBF_4	rt	DMF	10 mA	4 h	50
6	C	Mg	Bu_4NBF_4	rt	DMF	10 mA	4 h	61
7	Pt	Mg	Bu_4NPF_6	rt	DMF	10 mA	4 h	60
8	Pt	Mg	Bu_4NI	rt	DMF	10 mA	4 h	70
9	Pt	Mg	Bu_4NClO_4	rt	DMF	10 mA	4 h	67
10	Pt	Mg	Et_4NI	rt	DMF	10 mA	4 h	71
11	Pt	Mg	Bu_4NBF_4	rt	DMSO	10 mA	4 h	87
12	Pt	Mg	Bu_4NBF_4	rt	THF	10 mA	4 h	52
13	Pt	Mg	Bu_4NBF_4	rt	CH_3CN	10 mA	4 h	49
14	Pt	Mg	Bu_4NBF_4	rt	NMP	10 mA	4 h	51
15	Pt	Mg	Bu_4NBF_4	rt	DMF	5 mA	4 h	37
16	Pt	Mg	Bu_4NBF_4	rt	DMF	7.5 mA	4 h	58
17	Pt	Mg	Bu_4NBF_4	rt	DMF	12.5 mA	4 h	87
18	Pt	Mg	Bu_4NBF_4	rt	DMF	15 mA	4 h	63
19	Pt	Mg	Bu_4NBF_4	rt	DMF	20 mA	2 h	72
20	Pt	Mg	Bu_4NBF_4	rt	DMF	5 mA	8 h	69
21	Pt	Mg	Bu_4NBF_4	0 ℃	DMF	10 mA	4 h	62
22	Pt	Mg	Bu_4NBF_4	40 ℃	DMF	10 mA	4 h	76

[a] Reaction condition: **1** (0.3 mmol), $TBABF_4$ (0.3 mmol), CO_2 (1 atm), DMF (5 mL), constant current i = 10 mA, undivided cell, rt. [b] Determined by NMR spectroscopy using mesitylene as the internal standard. [c] Isolated yield

5. General procedure for the gram-scale reaction



1a, 30 mmol, 4.9 g

2a', 4.8 g, 83% yield

A 500 mL oven-dried electrochemical cell containing a stir bar was evacuated and back-filled under CO_2 flow for at least 3 times. **1a** (30 mmol), $TBABF_4$ (15 mmol) and DMF (400 mL) were added to the cell and the cell was installed with a Pt cathode and Mg anode and the cell was closed under CO_2 atmosphere. Then, the reaction mixture was electrolyzed under a constant current of 200 mA for 24 h at room temperature. Then the reaction mixture was carefully quenched with HCl (1M, 300 mL) at 0 °C and extracted three times with ethyl acetate (3 × 300 mL). The combined organic layers were washed with brine (2 × 300 mL) and dried over Na_2SO_4. The solvents were removed under reduced pressure and the crude residue was purified by flash column chromatography (petroleum ether/ethyl acetate/AcOH 1/1/0.005) to give desired product.

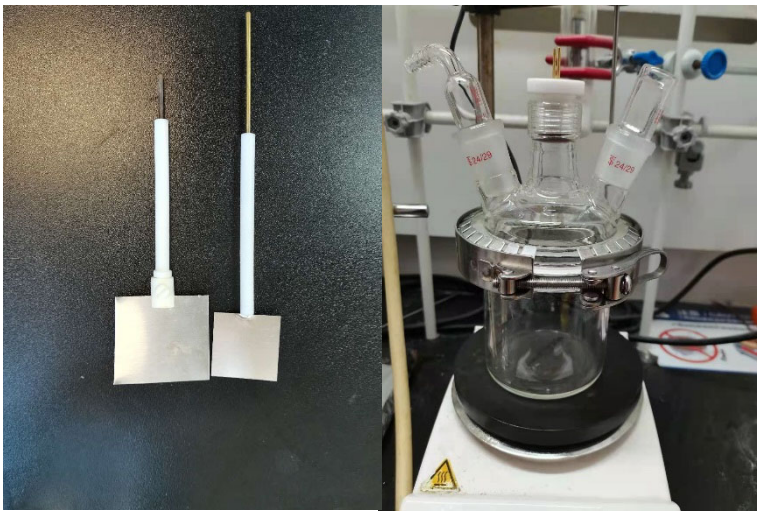
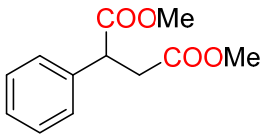
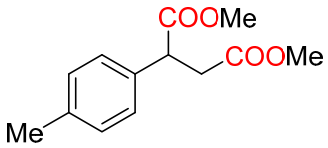


Figure S2. Photos for the gram-scale reaction. Pt 30 mm × 30 mm × 0.1 mm), Mg (50 mm × 60 mm × 0.5 mm). Electrochemical cell: 400 mL.

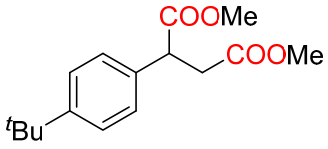
6. Characterization of products



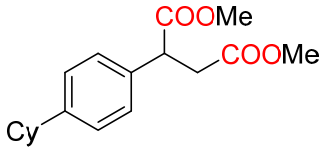
Dimethyl 2-phenylsuccinate (2a). White solid, 52 mg, 78% yield, m.p.: 60 – 62 °C. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.32 – 7.26 (m, 5H), 4.09 (dd, J = 10.1, 5.2 Hz, 1H), 3.67 (d, J = 2.0 Hz, 6H), 3.21 (dd, J = 17.0, 10.1 Hz, 1H), 2.67 (dd, J = 17.0, 5.2 Hz, 1H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 173.4, 171.9, 137.6, 128.8, 127.7, 127.6, 52.3, 51.8, 47.0, 37.6. **HRMS** (ESI-TOF) *m/z*: $[M+H]^+$ Calcd for $C_{12}H_{15}O_4$ 223.0970; Found 223.0963. **IR** (neat cm^{-1}) ν 3031, 2953, 1735, 1436, 699



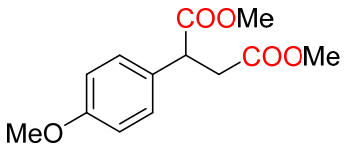
Dimethyl 2-(*p*-tolyl) succinate (2b). White solid, 42 mg, 57% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.18 – 7.12 (m, 4H), 4.06 (dd, J = 10.1, 5.3 Hz, 1H), 3.67 (s, 6H), 3.19 (dd, J = 17.0, 10.1 Hz, 1H), 2.65 (dd, J = 16.9, 5.3 Hz, 1H), 2.32 (s, 3H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 173.5, 172.0, 137.3, 134.6, 129.5, 127.5, 52.2, 51.7, 46.6, 37.6, 20.9. **HRMS** (ESI-TOF) *m/z*: $[M+Na]^+$ Calcd for $C_{13}H_{16}NaO_4$ 259.0946; Found 259.0947. **IR** (neat cm^{-1}) ν 2953, 2341, 1733, 1371, 817



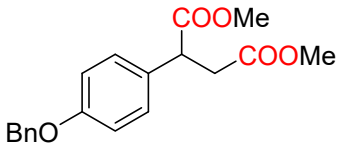
Dimethyl 2-(4-(tert-butyl) phenyl) succinate (2c). White solid, 60 mg, 72% yield, m.p.: 94 – 96 °C. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.26 (d, J = 8.4 Hz, 2H), 7.12 (d, J = 8.3 Hz, 2H), 3.99 (dd, J = 10.4, 4.9 Hz, 1H), 3.59 (s, 6H), 3.12 (dd, J = 17.0, 10.4 Hz, 1H), 2.59 (d, J = 5.0 Hz, 1H), 1.22 (s, 9H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 173.5, 172.0, 150.4, 134.5, 127.2, 125.7, 52.1, 51.7, 46.5, 37.5, 34.4, 31.2. **HRMS** (ESI-TOF) *m/z*: $[M+Na]^+$ Calcd for $C_{16}H_{22}NaO_4$ 301.1415; Found 301.1414. **IR** (neat cm^{-1})., 2953, 1738, 1513, 1436, 849



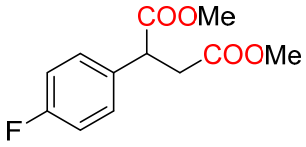
Dimethyl 2-(4-cyclohexylphenyl) succinate (2d). White solid, 56 mg, 62%. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.20 – 7.14 (m, 4H), 4.06 (dd, J = 10.4, 4.9 Hz, 1H), 3.67 (s, 6H), 3.20 (dd, J = 17.0, 10.4 Hz, 1H), 2.64 (dd, J = 17.0, 4.9 Hz, 1H), 2.49 – 2.44 (m, 1H), 1.86 – 1.80 (m, 4H), 1.74 (ddt, J = 12.0, 2.9, 1.4 Hz, 1H), 1.44 – 1.32 (m, 4H), 1.26 – 1.22 (m, 1H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 173.5, 172.0, 147.4, 134.8, 127.4, 127.2, 52.2, 51.7, 46.6, 44.0, 37.6, 34.3, 26.7, 26.0. **HRMS** (ESI-TOF) *m/z*: $[M+Na]^+$ Calcd for $C_{18}H_{24}NaO_4$ 327.1572; Found 327.1568. **IR** (neat cm^{-1}) ν 2953, 2924, 1738, 1342, 824, 724.



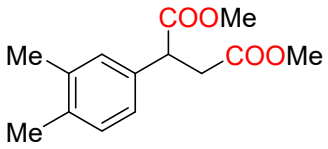
Dimethyl 2-(4-methoxyphenyl) succinate (2e). White solid, 40 mg, 53 % yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.19 (d, J = 8.7 Hz, 2H), 6.85 (d, J = 8.7 Hz, 2H), 4.03 (dd, J = 9.9, 5.4 Hz, 1H), 3.78 (s, 3H), 3.66 (d, J = 1.7 Hz, 6H), 3.26 – 3.10 (m, 1H), 2.64 (dd, J = 16.9, 5.5 Hz, 1H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 173.6, 171.9, 159.0, 129.6, 128.7, 114.2, 55.2, 52.2, 51.8, 46.2, 37.6. **HRMS** (ESI-TOF) *m/z*: $[M+Na]^+$ Calcd for $C_{13}H_{16}NaO_5$ 275.0895; Found 275.0900. **IR** (neat cm^{-1}) ν 2953, 2923, 1731, 1256, 830.



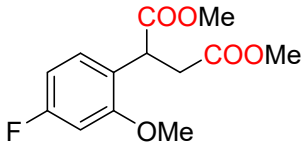
Dimethyl 2-(4-(benzyloxy) phenyl) succinate (2f). White solid, 40 mg, 41% yield, m.p.: 158 – 160 °C. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.43 – 7.31 (m, 5H), 7.20 (d, J = 8.5 Hz, 2H), 6.93 (d, J = 8.5 Hz, 2H), 5.04 (s, 2H), 4.04 (dd, J = 10.0, 5.4 Hz, 1H), 3.67 (d, J = 1.9 Hz, 6H), 3.18 (dd, J = 16.9, 10.0 Hz, 1H), 2.65 (dd, J = 16.9, 5.4 Hz, 1H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 173.6, 171.9, 158.2, 136.8, 129.9, 128.7, 128.5, 127.9, 127.4, 115.1, 70.0, 52.2, 51.8, 46.2, 37.6. **HRMS** (ESI-TOF) *m/z*: $[M+H]^+$ Calcd for $C_{19}H_{21}O_5$ 329.1389; Found 329.1380. **IR** (neat cm^{-1}) ν 2955, 2923, 1737, 1246, 848.



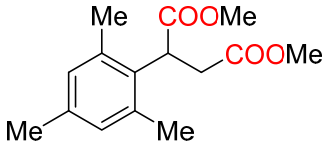
Dimethyl 2-(4-fluorophenyl) succinate (2g). White solid, 38 mg, 53% yield, m.p.: 64 – 66 °C. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.20 – 7.16 (m, 2H), 6.96 – 6.92 (m, 2H), 4.00 (dd, J = 9.7, 5.6 Hz, 1H), 3.60 (d, J = 4.6 Hz, 6H), 3.10 (dd, J = 16.9, 9.7 Hz, 1H), 2.59 (dd, J = 16.9, 5.6 Hz, 1H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 173.2, 171.7, 162.2 (d, J = 246.5 Hz), 133.3 (d, J = 3.5 Hz), 129.3 (d, J = 8.1 Hz), 115.7 (d, J = 21.5 Hz), 52.3, 51.8, 46.2, 37.5. **^{19}F NMR** (376 MHz, Chloroform-*d*) δ -114.61. **HRMS** (ESI-TOF) *m/z*: $[M+Na]^+$ Calcd for $C_{12}H_{13}FNaO_4$ 263.0695; Found 263.0686. **IR** (neat cm^{-1}) ν 2955, 2923, 1735, 1377, 822.



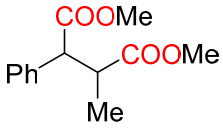
Dimethyl 2-(3,4-dimethylphenyl) succinate (2h). White solid, 50 mg, 67% yield, m.p.: 46 – 48 °C. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.10 – 6.99 (m, 3H), 4.03 (dd, J = 10.2, 5.2 Hz, 1H), 3.68 (d, J = 1.1 Hz, 6H), 3.20 (dd, J = 17.0, 10.2 Hz, 1H), 2.64 (dd, J = 17.0, 5.1 Hz, 1H), 2.24 (d, J = 5.5 Hz, 6H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 173.6, 172.0, 137.0, 135.9, 135.0, 130.0, 128.8, 124.9, 52.2, 51.7, 46.5, 37.6, 19.7, 19.3. **HRMS** (ESI-TOF) *m/z*: $[M+Na]^+$ Calcd for $C_{14}H_{18}NaO_4$ 273.1102; Found 273.1097. **IR** (neat cm^{-1}) ν 2952, 1738, 1384, 1202, 848,737.



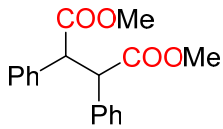
Dimethyl 2-(4-fluoro-2-methoxyphenyl) succinate (2i). White solid, 30 mg, 37% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.13 – 7.09 (m, 1H), 6.63 – 6.59 (m, 2H), 4.36 (dd, J = 9.3, 5.5 Hz, 1H), 3.80 (s, 3H), 3.66 (d, J = 0.7 Hz, 6H), 3.11 (dd, J = 16.8, 9.3 Hz, 1H), 2.57 (dd, J = 16.8, 5.6 Hz, 1H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 173.5, 172.2, 163.0 (d, J = 245.8 Hz), 157.7 (d, J = 10.0 Hz), 129.7 (d, J = 9.9 Hz), 122.3 (d, J = 3.1 Hz), 107.0 (d, J = 21.3 Hz), 99.3 (d, J = 25.7 Hz), 55.6, 52.2, 51.7, 41.2, 36.3. **^{19}F NMR** (565 MHz, Chloroform-*d*) δ -116.60. **HRMS** (ESI-TOF) *m/z*: $[M+Na]^+$ Calcd for $C_{13}H_{15}FNaO_5$ 293.0801; Found 293.0797. **IR** (neat cm^{-1}) *v* 2954, 2924, 1736, 1230, 837,759.



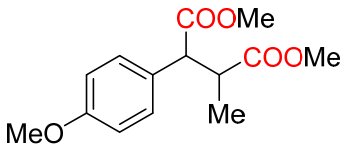
Dimethyl 2-mesitylsuccinate (2j). White solid, 40 mg, 51% yield, m.p.: 57 – 59 °C. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 6.85 (s, 2H), 4.64 (dd, J = 9.5, 4.5 Hz, 1H), 3.69 (d, J = 14.1 Hz, 6H), 3.31 (dd, J = 16.7, 9.5 Hz, 1H), 2.42 (dd, J = 16.7, 4.5 Hz, 1H), 2.25 (s, 9H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 174.3, 172.5, 136.7, 136.3, 132.6, 129.8, 52.3, 51.8, 41.6, 35.1, 20.7. **HRMS** (ESI-TOF) *m/z*: $[M+Na]^+$ Calcd for $C_{15}H_{20}NaO_4$ 287.1259; Found 287.1257. **IR** (neat cm^{-1}) *v* 2952, 2923, 1736, 1378, 1198, 825,731.



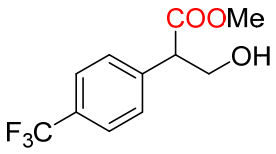
Dimethyl 2-methyl-3-phenylsuccinate (2k). White solid, 33 mg, 47% yield, *trans*/*cis* = 3:1. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.32 – 7.23 (m, 5H), 3.81 (d, J = 10.8 Hz, 1H), 3.67 (s, 3H), 3.42 (s, 3H), 3.25 (dq, J = 10.9, 6.9 Hz, 1H), 1.29 (d, J = 6.8 Hz, 3H). **^{13}C NMR** (101 MHz, Chloroform-*d*) mixture of isomers: δ 176.1, 174.5, 173.6, 172.6, 136.6, 136.2, 128.8, 128.5, 128.3, 128.2, 127.7, 127.6, 54.6, 54.0, 52.1, 51.9, 51.5, 43.6, 42.2, 16.3, 15.3. **HRMS** (ESI-TOF) *m/z*: $[M+Na]^+$ Calcd for $C_{13}H_{16}NaO_4$ 259.0946; Found 259.0940. **IR** (neat cm^{-1}) *v* 2953, 2924, 1732, 1377, 699.



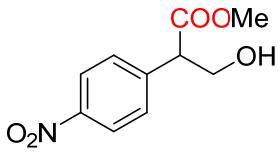
Dimethyl 2,3-diphenylsuccinate (2l). White solid, 67 mg, 75% yield, *trans*/*cis*=3:1. *trans*-isomer: **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.21 – 7.12 (m, 6H), 7.08 – 7.00 (m, 4H), 4.27 (s, 2H), 3.72 (s, 6H). *Trans* -**^{13}C NMR** (101 MHz, Chloroform-*d*) δ 173.6, 135.5, 128.4, 128.3, 127.4, 54.6, 52.4. *cis*-isomer: **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.50 – 7.48 (m, 4H), 7.37 – 7.27 (m, 6H), 4.40 (s, 2H), 3.41 (s, 6H). *Cis*-**^{13}C NMR** (101 MHz, Chloroform-*d*) δ 171.8, 136.3, 128.7, 128.3, 127.9, 54.9, 51.9. **HRMS** (ESI-TOF) *m/z*: $[M+Na]^+$ Calcd for $C_{18}H_{18}NaO_4$ 321.1102; Found 321.1096. **IR** (neat cm^{-1}). 2954, 1740, 1436, 1260, 763.



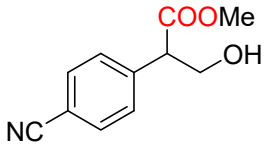
Dimethyl 2-(4-methoxyphenyl)-3-methylsuccinate (2m). White solid, 17 mg, 22% yield, *trans*/*cis* = 5:1. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.23 (d, J = 8.7 Hz, 2H), 6.82 (d, J = 8.7 Hz, 2H), 3.77 (s, 3H), 3.73 (d, J = 6.7 Hz, 1H), 3.67 (s, 3H), 3.44 (s, 3H), 3.21 (dq, J = 11.0, 6.9 Hz, 1H), 1.27 (d, J = 6.8 Hz, 3H). **^{13}C NMR** (101 MHz, Chloroform-*d*) mixture of isomers: δ 174.6, 172.9, 129.4, 129.3, 128.7, 114.2, 113.9, 55.1, 53.9, 52.0, 51.5, 43.8, 42.3, 30.8, 16.3. **HRMS** (ESI-TOF) *m*/*z*: $[M+H]^+$ Calcd for $C_{14}H_{19}O_5$ 267.1232; Found 267.1229. **IR** (neat cm^{-1}) ν 2952, 2923, 1731, 1251, 829.



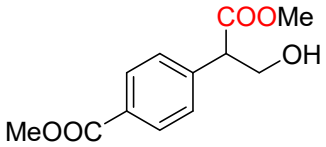
Methyl 3-hydroxy-2-(4-(trifluoromethyl) phenyl) propanoate (3n). White solid, 41 mg, 55% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.60 (d, J = 8.7 Hz, 2H), 7.40 (d, J = 8.0 Hz, 2H), 4.13 (t, J = 9.6 Hz, 1H), 3.91 (dd, J = 8.0, 5.1 Hz, 1H), 3.88 – 3.84 (m, 1H), 3.72 (s, 3H), 2.51 (s, 1H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 172.9, 139.5, 130.0 (d, J = 32.6 Hz), 128.6, 125.7 (q, J = 3.8 Hz), 125.2, 122.5, 64.2, 53.5, 52.4. **^{19}F NMR** (565 MHz, Chloroform-*d*) δ -62.67. **HRMS** (ESI-TOF) *m*/*z*: $[M-H]^-$ Calcd for $C_{11}H_{10}F_3O_3$ 247.0582; Found 247.0589. **IR** (neat cm^{-1}) ν 3357, 2957, 1735, 1327, 841.



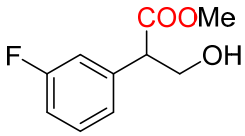
Methyl 3-hydroxy-2-(4-nitrophenyl) propanoate (3o). White solid, 25 mg, 37% yield, m.p.: 110 – 112 °C. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 8.21 (d, J = 8.7 Hz, 2H), 7.47 (d, J = 8.4 Hz, 2H), 4.15 – 4.11 (m, 1H), 3.98 – 3.91 (m, 2H), 3.74 (s, 3H), 2.36 (t, J = 6.5 Hz, 1H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 172.3, 147.5, 142.9, 129.2, 123.9, 64.0, 53.4, 52.5. **HRMS** (ESI-TOF) *m*/*z*: $[M-H]^-$ Calcd for $C_{10}H_{10}NO_5$ 224.0559; Found 224.0564. **IR** (neat cm^{-1}) ν 3416, 2954, 2924, 1727, 1515, 1345, 851.



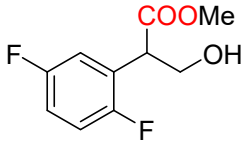
Methyl 2-(4-cyanophenyl)-3-hydroxypropanoate (3p). White solid, 27 mg, 49% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.63 (d, J = 7.8 Hz, 2H), 7.40 (d, J = 7.9 Hz, 2H), 4.09 (d, J = 9.9 Hz, 1H), 3.88 (q, J = 10.9, 8.0 Hz, 2H), 3.72 (s, 3H), 2.49 (s, 1H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 172.4, 140.9, 132.5, 129.1, 118.4, 111.6, 63.9, 53.6, 52.5. **HRMS** (ESI-TOF) *m*/*z*: $[M-H]^-$ Calcd for $C_{11}H_{10}NO_3$ 204.0660; Found 204.0670. **IR** (neat cm^{-1}) ν 3436, 2954, 2924, 2230, 1733, 837.



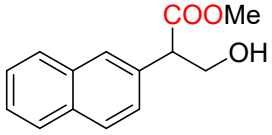
Methyl 4-(3-hydroxy-1-methoxy-1-oxopropan-2-yl) benzoate (3q). White solid, 51 mg, 72% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.99 (d, J = 8.0 Hz, 2H), 7.33 (d, J = 7.9 Hz, 2H), 4.12 (dd, J = 10.8, 8.3 Hz, 1H), 3.91 – 3.88 (m, 4H), 3.83 (dd, J = 10.8, 5.2 Hz, 1H), 3.70 (s, 3H), 2.60 (s, 1H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 172.8, 166.6, 140.7, 130.0, 129.5, 128.2, 64.2, 53.8, 52.2, 52.0. **HRMS** (ESI-TOF) *m/z*: $[M+Na]^+$ Calcd for $C_{12}H_{14}NaO_5$ 261.0738; Found 261.0737. **IR** (neat cm^{-1}) ν 3446, 2954, 1724, 1166, 824.



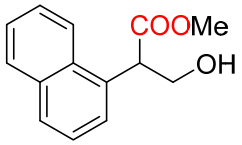
Methyl 2-(3-fluorophenyl)-3-hydroxypropanoate (3r). White solid, 45 mg, 76% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.30 (tdd, J = 7.2, 6.0, 1.3 Hz, 1H), 7.06 – 6.96 (m, 3H), 4.14 – 4.08 (m, 1H), 3.86 – 3.80 (m, 2H), 3.72 (s, 3H), 2.44 (s, 1H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 173.0, 162.8 (d, J = 246.7 Hz), 137.8 (d, J = 7.4 Hz), 130.3 (d, J = 8.5 Hz), 123.9 (d, J = 2.9 Hz), 115.2 (d, J = 22.1 Hz), 114.7 (d, J = 21.2 Hz), 64.3, 53.4, 52.3. **^{19}F NMR** (376 MHz, Chloroform-*d*) δ -112.28. **HRMS** (ESI, m/z): $[M+Na]^+$ calcd for $C_{10}H_{11}FNaO_3$ 221.0589; Found 221.0583. **IR** (neat cm^{-1}) ν 3418, 2954, 2931, 1737, 1249, 691.



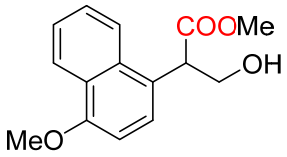
Methyl 2-(2,5-difluorophenyl)-3-hydroxypropanoate (3s). White solid, 37 mg, 57% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.03 – 6.93 (m, 3H), 4.12 – 4.05 (m, 2H), 3.86 – 3.83 (m, 1H), 3.73 (s, 3H), 2.67 (s, 1H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 172.7, 157.5 (dd, J = 451.8, 2.3 Hz),) 157.5 (dd, J = 33.7, 2.5 Hz), 124.7 (d, J = 7.9 Hz), 124.5 (d, J = 8.1 Hz), 116.88 – 115.6 (m), 63.2, 52.5, 46.6. **^{19}F NMR** (376 MHz, Chloroform-*d*) δ -118.09 (d, J = 17.9 Hz), -123.71 (d, J = 16.7 Hz). **HRMS** (ESI-TOF) *m/z*: $[M+Na]^+$ Calcd for $C_{10}H_{10}F_2NaO_3$ 239.0495; Found 239.0493. **IR** (neat cm^{-1}) ν 3435, 2955, 2925, 1736, 1377, 875, 816.



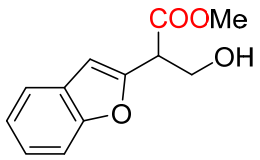
Methyl 3-hydroxy-2-(naphthalen-2-yl) propanoate (3t). White solid, 38 mg, 53% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.84 – 7.81 (m, 3H), 7.74 (d, J = 1.7 Hz, 1H), 7.52 – 7.46 (m, 2H), 7.39 (dd, J = 8.5, 1.9 Hz, 1H), 4.25 (dd, J = 11.1, 8.7 Hz, 1H), 4.03 (dd, J = 8.6, 5.3 Hz, 1H), 3.91 (dd, J = 11.1, 5.3 Hz, 1H), 3.73 (s, 3H), 2.44 (s, 1H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 173.5, 133.3, 132.9, 132.7, 128.6, 127.7, 127.6, 127.1, 126.3, 126.1, 125.8, 64.5, 53.9, 52.2. **HRMS** (ESI-TOF) *m/z*: $[M+Na]^+$ Calcd for $C_{14}H_{14}NaO_3$ 253.0840; Found 253.0839. **IR** (neat cm^{-1}) ν 3445, 2951, 1733, 1599, 749.



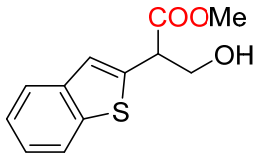
Methyl 3-hydroxy-2-(naphthalen-1-yl) propanoate (3u). White solid, 45 mg, 65% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 8.11 (d, J = 8.4 Hz, 1H), 7.85 (dd, J = 30.0, 7.8 Hz, 2H), 7.59 – 7.50 (m, 2H), 7.44 (t, J = 7.6 Hz, 1H), 7.38 (d, J = 7.1 Hz, 1H), 4.69 (dd, J = 9.1, 4.6 Hz, 1H), 4.32 (t, J = 10.3 Hz, 1H), 3.92 – 3.88 (m, 1H), 3.72 (s, 3H), 2.64 (s, 1H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 174.3, 134.0, 131.7, 131.3, 129.0, 128.3, 126.6, 125.8, 125.4, 125.3, 122.8, 64.1, 52.2, 49.6. **HRMS** (ESI-TOF) *m/z*: $[M+Na]^+$ Calcd for $C_{14}H_{14}NaO_3$ 253.0840; Found 253.0835. **IR** (neat cm^{-1}) ν 3456, 2958, 2924, 1732, 735.



Methyl 3-hydroxy-2-(4-methoxynaphthalen-1-yl) propanoate (3v). White solid, 33 mg, 42%, m.p.: 106 – 108 °C. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 8.34 (d, J = 8.3 Hz, 1H), 8.04 (d, J = 8.5 Hz, 1H), 7.58 – 7.49 (m, 2H), 7.29 (d, J = 8.0 Hz, 1H), 6.77 (d, J = 8.0 Hz, 1H), 4.58 (dd, J = 9.2, 4.7 Hz, 1H), 4.28 (dd, J = 11.3, 9.2 Hz, 1H), 3.99 (s, 3H), 3.87 (dd, J = 11.3, 4.7 Hz, 1H), 3.71 (s, 3H), 2.63 (s, 1H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 174.6, 155.2, 132.2, 127.1, 126.0, 125.5, 125.1, 123.6, 122.8, 122.5, 103.2, 64.1, 55.4, 52.2, 49.1. **HRMS** (ESI-TOF) *m/z*: $[M+Na]^+$ Calcd for $C_{15}H_{16}NaO_4$ 283.0946; Found 283.0943. **IR** (neat cm^{-1}) ν 3440, 2958, 2924, 1732, 1250, 754.

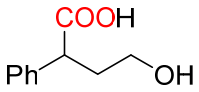


Methyl 2-(benzofuran-2-yl)-3-hydroxypropanoate (3w). White solid, 35 mg, 53% yield, m.p.: 49 – 51 °C. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.53 (d, J = 7.7 Hz, 1H), 7.45 (d, J = 8.1 Hz, 1H), 7.29 – 7.20 (m, 2H), 6.67 (s, 1H), 4.25 (dd, J = 12.1, 6.9 Hz, 1H), 4.10 (td, J = 11.4, 5.1 Hz, 2H), 3.78 (s, 3H), 2.44 (s, 1H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 171.0, 154.7, 151.9, 128.1, 124.2, 122.9, 120.8, 111.1, 104.9, 62.2, 52.6, 47.9. **HRMS** (ESI-TOF) *m/z*: $[M+Na]^+$ Calcd for $C_{12}H_{12}NaO_4$ 243.0633; Found 243.0632 **IR** (neat cm^{-1}) ν 3448, 2954, 2923, 1740, 1250, 751.

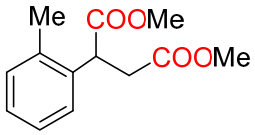


Methyl 2-(benzo[b]thiophen-2-yl)-3-hydroxypropanoate (3x). Red oil, 40 mg, 57% yield, m.p.: 142 – 144 °C. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.79 (ddd, J = 7.4, 1.8, 0.8 Hz, 1H), 7.73 (dd, J = 6.8, 1.8 Hz, 1H), 7.33 (pd, J = 7.1, 1.5 Hz, 2H), 7.23 (s, 1H), 4.20 – 4.19 (m, 2H), 4.03 – 4.00 (m, 1H), 3.77 (s, 3H), 2.49 (s, 1H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 172.1, 139.5, 139.3, 137.8, 124.4, 124.3, 123.3, 123.0, 122.1, 64.3, 52.5, 49.5. **HRMS** (ESI-TOF) *m/z*: $[M+H]^+$ Calcd for $C_{12}H_{12}NaO_3S$ 259.0397; Found 259.0404. **IR** (neat cm^{-1}) ν 3448,

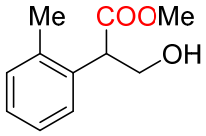
2952, 2925, 1736, 1197, 748.



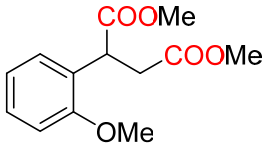
4-Hydroxy-2-phenylbutanoic acid (3y'). White solid, 33 mg, 62% yield. **^{1}H NMR** (400 MHz, DMSO-d_6) δ 7.34 – 7.24 (m, 5H), 3.65 (t, J = 7.5 Hz, 1H), 3.36 – 3.26 (m, 3H), 2.13 (dd, J = 13.6, 6.9 Hz, 1H), 1.75 (dd, J = 13.4, 6.7 Hz, 1H). **^{13}C NMR** (101 MHz, DMSO-d_6) δ 175.2, 140.2, 128.9, 128.3, 127.3, 58.9, 47.7, 36.5. **HRMS** (ESI, m/z): $[M-H]^-$ calcd for target $C_{10}H_{11}O_3$ 179.0714; Found 179.0713. **IR** (neat cm^{-1}) v 3350, 2954, 2921, 1764, 694.



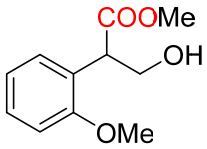
Dimethyl 2-(*o*-tolyl) succinate (2z). Colorless oil, 35 mg, 50% yield. **^{1}H NMR** (400 MHz, Chloroform-d) δ 7.18 – 7.17 (m, 4H), 4.37 (dd, J = 10.0, 5.1 Hz, 1H), 3.67 (d, J = 4.5 Hz, 6H), 3.19 (dd, J = 17.0, 10.0 Hz, 1H), 2.60 (dd, J = 17.0, 5.0 Hz, 1H), 2.42 (s, 3H). **^{13}C NMR** (101 MHz, Chloroform-d) δ 173.8, 172.2, 136.2, 136.1, 130.8, 127.5, 126.7, 126.5, 52.3, 51.9, 42.7, 37.0, 19.7. **HRMS** (ESI-TOF) m/z: $[M+Na]^+$ Calcd for $C_{13}H_{16}NaO_4$ 259.0946; Found 259.0944. **IR** (neat cm^{-1}) v 2954, 2924, 1742, 1376, 735.



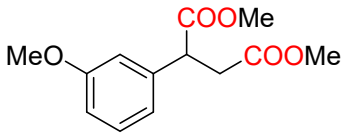
Methyl 3-hydroxy-2-(*o*-tolyl) propanoate (3z). Colorless oil, 20 mg, 34% yield. **^{1}H NMR** (400 MHz, Chloroform-d) δ 7.20 – 7.15 (m, 4H), 4.15 – 4.10 (m, 2H), 3.76 – 3.67 (m, 4H), 2.43 – 2.40 (m, 4H). **^{13}C NMR** (101 MHz, Chloroform-d) δ 174.2, 136.3, 133.9, 130.8, 127.6, 127.0, 126.4, 63.8, 52.1, 49.7, 19.6. **HRMS** (ESI-TOF) m/z: $[M+Na]^+$ Calcd for $C_{11}H_{14}NaO_3$ 217.0840; Found 217.0839. **IR** (neat cm^{-1}) v 3447, 2953, 2925, 1735, 1377, 736.



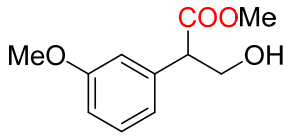
Dimethyl 2-(2-methoxyphenyl) succinate (2aa). Colorless oil, 35 mg, 46% yield. **^{1}H NMR** (400 MHz, Chloroform-d) δ 7.25 (td, J = 7.8, 1.7 Hz, 1H), 7.16 (dd, J = 7.5, 1.7 Hz, 1H), 6.95 – 6.84 (m, 2H), 4.42 (dd, J = 9.6, 5.1 Hz, 1H), 3.82 (s, 3H), 3.67 (s, 3H), 3.14 (dd, J = 16.8, 9.7 Hz, 1H), 2.59 (dd, J = 16.8, 5.1 Hz, 1H). **^{13}C NMR** (101 MHz, Chloroform-d) 173.7, 172.4, 156.6, 128.9, 128.7, 126.6, 120.7, 110.8, 55.4, 52.1, 51.7, 41.7, 36.4. **HRMS** (ESI, m/z): $[M+Na]^+$ calcd for $C_{13}H_{16}NaO_5$ 275.0895; Found 275.0887. **IR** (neat cm^{-1}) v 2954, 2922, 1736, 1039, 760.



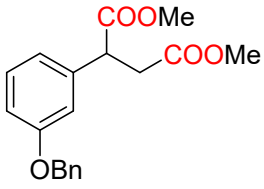
Methyl 3-hydroxy-2-(2-methoxyphenyl) propanoate (**3aa**). Colorless oil, 20 mg, 31% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.28 – 7.24 (m, 1H), 6.86 – 6.80 (m, 3H), 4.16 – 4.08 (m, 1H), 3.85 – 3.79 (m, 5H), 3.71 (s, 3H), 2.29 (s, 1H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 173.4, 159.8, 136.9, 129.8, 120.4, 113.9, 113.0, 64.5, 55.2, 53.8, 52.2. **HRMS** (ESI, m/z): $[M+Na]^+$ calcd for $C_{11}H_{14}NaO_4$ 233.0789; Found 233.0783. **IR** (neat cm^{-1}) *v* 3389, 2952, 2923, 1735, 755.



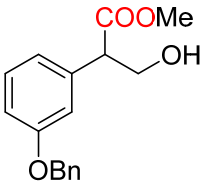
Dimethyl 2-(3-methoxyphenyl) succinate (2ab). Colorless oil, 27 mg, 36% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.18 – 7.17 (m, 4H), 4.37 (dd, *J* = 10.0, 5.1 Hz, 1H), 3.67 (d, *J* = 4.5 Hz, 6H), 3.19 (dd, *J* = 17.0, 10.0 Hz, 1H), 2.60 (dd, *J* = 17.0, 5.0 Hz, 1H), 2.42 (s, 3H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 173.86, 172.23, 136.25, 136.12, 130.85, 127.52, 126.71, 126.57, 52.35, 51.92, 42.77, 37.06, 19.72. **HRMS** (ESI-TOF) *m/z*: $[M+H]^+$ Calcd for $C_{13}H_{17}O_5$ 253.1076; Found 253.1076. **IR** (neat cm^{-1}) *v* 2955, 2924, 1735, 1261, 780.



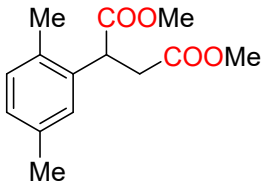
Methyl 3-hydroxy-2-(3-methoxyphenyl) propanoate (3ab). Colorless oil, 15 mg, 24%. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.26 (t, *J* = 2.0 Hz, 1H), 6.86 – 6.80 (m, 3H), 4.14 – 4.12 (m, 1H), 3.89 – 3.79 (m, 5H), 3.71 (s, 3H), 2.29 (s, 1H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 173.4, 159.8, 136.9, 129.8, 120.4, 113 .9, 113.0, 64.5, 55.2, 53.8, 52.2. **HRMS** (ESI-TOF) *m/z*: $[M+Na]^+$ Calcd for $C_{11}H_{14}NaO_4$ 233.0789; Found 233.0779. **IR** (neat cm^{-1}) *v* 3447, 2953, 2925, 1735, 1244, 693.



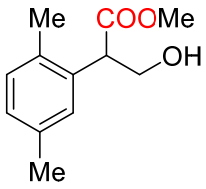
Dimethyl 2-(3-(benzyloxy) phenyl) succinate (2ac). White solid, 28 mg, 29% yield, m.p.: 210 – 212 °C. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.44 – 7.33 (m, 5H), 7.25 – 7.22 (m, 1H), 6.91 – 6.86 (m, 3H), 5.05 (s, 2H), 4.06 (dd, *J* = 10.2, 5.1 Hz, 1H), 3.67 (d, *J* = 1.7 Hz, 6H), 3.19 (dd, *J* = 17.0, 10.3 Hz, 1H), 2.66 (dd, *J* = 17.0, 5.1 Hz, 1H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 173.2, 171.9, 159.0, 139.1, 136.7, 129.9, 128.5, 128.0, 127.5, 120.3, 114.3, 113.9, 69.9, 52.3, 51.8, 47.0, 37.5. **HRMS** (ESI-TOF) *m/z*: $[M+Na]^+$ Calcd for $C_{19}H_{20}NaO_5$ 351.1208; Found 351.1202. **IR** (neat cm^{-1}) *v* 2953, 2923, 1737, 1162, 782.



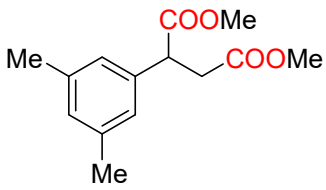
Methyl 2-(3-(benzyloxy) phenyl)-3-hydroxypropanoate (3ac). White solid, 22 mg, 26% yield, m.p.: 263 – 265 ºC. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.44 – 7.33 (m, 5H), 7.26 (d, J = 15.6 Hz, 1H), 6.92 – 6.85 (m, 3H), 5.05 (s, 2H), 4.15 – 4.09 (m, 1H), 3.84 – 3.80 (m, 2H), 3.70 (s, 3H), 2.26 (s, 1H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 173.4, 159.0, 137.0, 136.7, 129.9, 128.5, 128.0, 127.5, 120.7, 114.8, 113.9, 70.0, 64.5, 53.8, 52.2. **HRMS** (ESI-TOF) *m/z*: $[M+Na]^+$ Calcd for $C_{17}H_{18}NaO_4$ 309.1102; Found 309.1098. **IR** (neat cm^{-1}) *v* 3382, 2954, 2923, 1732, 1260, 790.



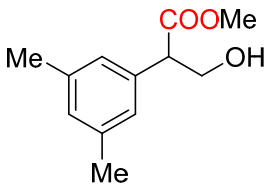
Dimethyl 2-(2,5-dimethylphenyl) succinate (2ad). Colorless oil, 44 mg, 59% yield. **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.07 (d, J = 7.6 Hz, 1H), 6.98 (d, J = 7.6 Hz, 2H), 4.33 (dd, J = 10.2, 4.9 Hz, 1H), 3.68 (d, J = 7.0 Hz, 6H), 3.19 (dd, J = 17.0, 10.1 Hz, 1H), 2.58 (dd, J = 17.1, 4.9 Hz, 1H), 2.37 (s, 3H), 2.28 (s, 3H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 173.8, 172.1, 135.9, 132.8, 130.6, 128.2, 127.3, 52.2, 51.8, 42.6, 37.0, 20.9, 19.1. **HRMS** (ESI-TOF) *m/z*: $[M+Na]^+$ Calcd for $C_{14}H_{18}NaO_4$ 273.1102; Found 273.1097. **IR** (neat cm^{-1}) *v* 2953, 2922, 1737, 1377, 810



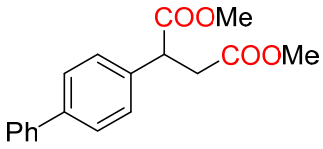
Methyl 2-(2,5-dimethylphenyl)-3-hydroxypropanoat (3ad). White solid, 18 mg, 29% yield, m.p.: 49 – 51 ºC. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.09 (d, J = 7.7 Hz, 1H), 6.99 (d, J = 7.8 Hz, 1H), 6.95 (s, 1H), 4.16 – 4.07 (m, 2H), 3.71 – 3.68 (m, 4H), 2.41 (s, 1H), 2.35 (s, 3H), 2.29 (s, 3H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 174.3, 135.9, 133.7, 133.1, 130.7, 128.4, 127.7, 63.9, 52.1, 49.7, 20.9, 19.1. **HRMS** (ESI-TOF) *m/z*: $[M+Na]^+$ Calcd for $C_{12}H_{16}NaO_3$ 231.0997; Found 231.0994. **IR** (neat cm^{-1}) *v* 3436, 2954, 2924, 1732,1377, 811.



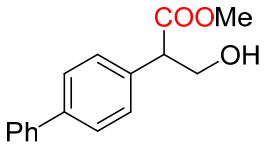
Dimethyl 2-(3,5-dimethylphenyl) succinate (2ae). Colorless oil, 39 mg, 52% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 6.91 (s, 1H), 6.88 (s, 2H), 4.01 (dd, J = 10.4, 5.0 Hz, 1H), 3.68 (s, 6H), 3.19 (dd, J = 17.0, 10.4 Hz, 1H), 2.63 (dd, J = 17.0, 5.0 Hz, 1H), 2.29 (s, 6H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 173.5, 172.0, 138.4, 137.4, 129.3, 125.3, 52.2, 51.7, 46.8, 37.6, 21.1. **HRMS** (ESI-TOF) *m/z*: $[M+Na]^+$ Calcd for $C_{14}H_{18}NaO_4$ 273.1102; Found 273.1098. **IR** (neat cm^{-1}) *v* 2954, 2923, 1740, 1376, 690



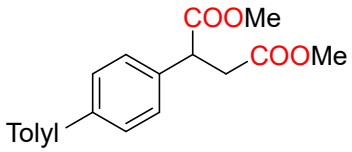
Methyl 2-(3,5-dimethylphenyl)-3-hydroxypropanoate (3ae). Colorless oil, 15 mg, 24% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 6.93 (s, 1H), 6.86 (s, 2H), 4.15 – 4.05 (m, 1H), 3.78 (q, J = 5.4 Hz, 2H), 3.71 (s, 3H), 2.30 (s, 6H), 2.23 (s, 1H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 173.7, 138.5, 135.3, 129.5, 125.8, 64.6, 53.8, 52.1, 21.2. **HRMS** (ESI, m/z): $[M-H]^-$ Calcd for $C_{12}H_{15}O_3$ 207.1021; Found 207.1027. **IR** (neat cm^{-1}) ν 3443, 2954, 2923, 1738, 691



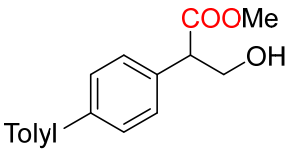
Dimethyl 2-([1,1'-biphenyl]-4-yl) succinate (2af). White solid, 13 mg, 11% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.56 (td, J = 5.4, 1.5 Hz, 4H), 7.43 (t, J = 7.6 Hz, 2H), 7.35 (d, J = 7.8 Hz, 3H), 4.15 (dd, J = 10.0, 5.4 Hz, 1H), 3.70 (d, J = 6.4 Hz, 6H), 3.28 – 3.21 (m, 1H), 2.74 (d, J = 5.3 Hz, 1H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 173.4, 171.9, 140.6, 140.5, 136.6, 128.7, 128.1, 127.6, 127.4, 127.0, 52.4, 51.9, 46.7, 37.5. **HRMS** (ESI-TOF) *m/z*: $[M+Na]^+$ Calcd for $C_{18}H_{18}NaO_4$ 321.1102; Found 321.1097. **IR** (neat cm^{-1}) ν 2953, 1735, 1505, 1170, 811.



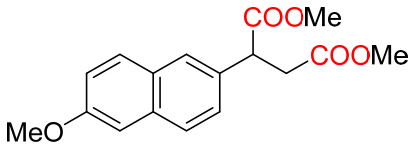
Methyl 2-([1,1'-biphenyl]-4-yl)-3-hydroxypropanoate (3af). White solid, 54 mg, 66% yield, m.p.: 79 – 81 °C. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.58 (d, J = 8.0 Hz, 4H), 7.44 (t, J = 7.6 Hz, 2H), 7.37 – 7.34 (m, 3H), 4.18 (dd, J = 10.3, 7.8 Hz, 1H), 3.93 – 3.86 (m, 2H), 3.74 (s, 3H), 2.51 (s, 1H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 173.5, 140.7, 140.4, 134.5, 128.7, 128.5, 127.5, 127.3, 127.0, 64.5, 53.5, 52.2. **HRMS** (ESI-TOF) *m/z*: $[M+Na]^+$ Calcd for $C_{16}H_{16}NaO_3$ 279.0997; Found 279.0994. **IR** (neat cm^{-1}) ν 3345, 2952, 1732, 1582, 1377, 833.



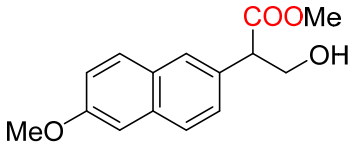
Dimethyl 2-(4'-methyl-[1,1'-biphenyl]-4-yl) succinate (2ag). White solid, 7 mg, 8% yield, m.p.: 115 – 117 °C. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.54 (d, J = 8.0 Hz, 2H), 7.47 (d, J = 7.9 Hz, 2H), 7.33 (d, J = 8.0 Hz, 2H), 7.24 (d, J = 7.8 Hz, 2H), 4.14 (dd, J = 10.0, 5.3 Hz, 1H), 3.70 (d, J = 5.5 Hz, 6H), 3.24 (dd, J = 17.0, 10.0 Hz, 1H), 2.71 (dd, J = 17.0, 5.3 Hz, 1H), 2.39 (s, 3H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 173.4, 171.9, 140.5, 137.6, 137.2, 136.3, 129.5, 128.0, 127.3, 126.8, 52.3, 51.8, 46.7, 37.6, 21.0. **HRMS** (ESI-TOF) *m/z*: $[M+Na]^+$ Calcd for $C_{19}H_{20}NaO_4$ 335.1259; Found 335.1253. **IR** (neat cm^{-1}) ν 3292, 1732, 1461, 1377, 811.



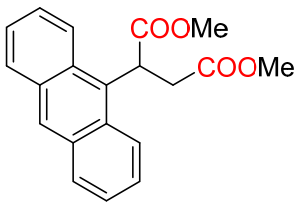
Methyl 3-hydroxy-2-(4'-methyl-[1,1'-biphenyl]-4-yl) propanoate (3ag). White solid, 58 mg, 72% yield, m.p.: 105 – 107 °C. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.58 (d, J = 7.9 Hz, 2H), 7.50 (d, J = 7.8 Hz, 2H), 7.36 (d, J = 7.9 Hz, 2H), 7.28 (d, J = 7.7 Hz, 2H), 4.2 (t, J = 8 Hz, 1H), 3.94 – 3.87 (m, 2H), 3.77 (s, 3H), 2.47 (s, 1H), 2.43 (s, 3H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 173.5, 140.6, 137.5, 137.1, 134.2, 129.4, 128.5, 127.3, 126.8, 64.5, 53.5, 52.2, 21.0. **HRMS** (ESI-TOF) *m/z*: $[M+Na]^+$ Calcd for $C_{17}H_{18}NaO_3$ 293.1153; Found 293.1144. **IR** (neat cm^{-1}) ν 3400, 2955, 2924, 1732, 1381, 850.



Dimethyl 2-(6-methoxynaphthalen-2-yl) succinate (2ah). White solid, 12 mg, 13% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.70 (d, J = 9.0 Hz, 2H), 7.66 (d, J = 1.8 Hz, 1H), 7.36 (dd, J = 8.5, 1.9 Hz, 1H), 7.15 (dd, J = 8.9, 2.6 Hz, 1H), 7.11 (d, J = 2.5 Hz, 1H), 4.22 (dd, J = 9.9, 5.4 Hz, 1H), 3.91 (s, 3H), 3.68 (d, J = 2.7 Hz, 6H), 3.29 (dd, J = 16.9, 9.9 Hz, 1H), 2.75 (dd, J = 16.9, 5.4 Hz, 1H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 173.5, 172.0, 157.8, 133.9, 132.7, 129.3, 128.8, 127.4, 126.5, 126.0, 119.2, 105.5, 55.3, 52.3, 51.8, 47.0, 37.6. **HRMS** (ESI-TOF) *m/z*: $[M+Na]^+$ Calcd for $C_{17}H_{18}NaO_5$ 325.1051; Found 325.1048. **IR** (neat cm^{-1}) ν 2954, 1732, 1382, 1250, 748.

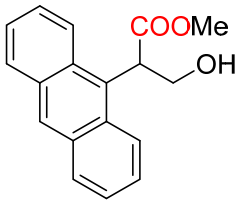


Methyl 3-hydroxy-2-(6-methoxynaphthalen-2-yl) propanoate (3ah). White solid, 30 mg, 38% yield, m.p.: 97 – 99 °C. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.71 (dd, J = 8.7, 4.7 Hz, 2H), 7.66 (s, 1H), 7.35 (dd, J = 8.5, 1.9 Hz, 1H), 7.16 (dd, J = 8.9, 2.6 Hz, 1H), 7.11 (d, J = 2.5 Hz, 1H), 4.22 (dd, J = 11.0, 8.6 Hz, 1H), 3.99 (dd, J = 8.6, 5.3 Hz, 1H), 3.91 – 3.87 (m, 4H), 3.72 (s, 3H), 2.45 (s, 1H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 173.7, 157.8, 133.9, 130.5, 129.2, 128.8, 127.4, 126.9, 126.3, 119.2, 105.5, 64.5, 55.2, 53.8, 52.1. **HRMS** (ESI-TOF) *m/z*: $[M+Na]^+$ Calcd for $C_{15}H_{16}NaO_4$ 283.0946; Found 283.0943. **IR** (neat cm^{-1}) ν 3450, 2953, 1732, 1382, 1120, 748.

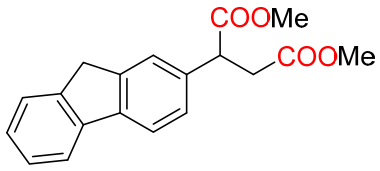


Dimethyl 2-(anthracen-9-yl) succinate (2ai). White solid, 11 mg, 11% yield, m.p.: 101 – 103 °C. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.61 (d, J = 7.6 Hz, 1H), 7.53 (d, J = 7.5 Hz, 1H), 7.44 (d, J = 6.7 Hz, 1H), 7.40 – 7.26 (m, 5H), 6.28 (t, J = 7.6 Hz, 1H), 4.96 (s, 1H), 3.76 (s, 3H), 3.67 (dd, J = 17.2, 6.9 Hz, 1H), 3.59 – 3.52 (m, 4H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 172.1, 171.5, 138.5, 137.4, 134.5, 134.3, 132.0, 128.4, 127.9, 127.8, 127.5, 127.4, 127.1, 127.0, 124.4, 119.9, 52.9, 52.4, 52.0, 35.3. **HRMS** (ESI-TOF)

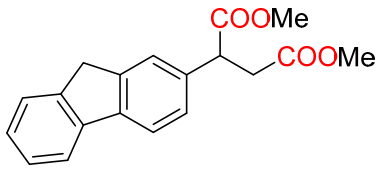
m/z: $[M+Na]^+$ Calcd for $C_{20}H_{18}NaO_4$ 345.1102; Found 345.1107. **IR** (neat cm^{-1}) *v* 2953, 2923, 1737, 1600, 730.



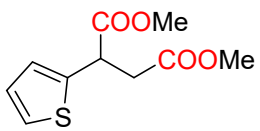
Methyl 2-(anthracen-9-yl)-3-hydroxypropanoate (3ai). White solid, 44 mg, 52% yield, m.p.: 71 – 73 °C. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.60 (d, J = 7.6 Hz, 1H), 7.43 – 7.26 (m, 7H), 6.28 (dd, J = 8.2, 5.2 Hz, 1H), 4.98 (s, 1H), 4.67 (dd, J = 13.4, 8.2 Hz, 1H), 4.56 (dd, J = 13.4, 5.2 Hz, 1H), 3.55 (s, 3H), 2.22 (s, 1H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 171.7, 138.2, 136.4, 134.3, 134.0, 132.0, 128.2, 128.1, 128.0, 127.9, 127.6, 127.5, 127.1, 127.1, 60.6, 52.6, 52.4. **HRMS** (ESI-TOF) *m/z*: $[M+Na]^+$ Calcd for $C_{18}H_{16}NaO_3$ 303.0997; Found 303.0992. **IR** (neat cm^{-1}) *v* 3457, 2955, 2927, 1735, 1599, 776.



Dimethyl 2-(9H-fluoren-2-yl) succinate (2aj). White solid, 12 mg, 13% yield, m.p.: 106 – 108 °C. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.74 (dd, J = 11.1, 7.6 Hz, 2H), 7.53 (d, J = 7.5 Hz, 1H), 7.47 (s, 1H), 7.37 (t, J = 7.3 Hz, 1H), 7.30 (t, J = 6.7 Hz, 2H), 4.17 (dd, J = 10.0, 5.3 Hz, 1H), 3.88 (s, 2H), 3.69 (d, J = 6.3 Hz, 6H), 3.26 (dd, J = 17.0, 10.0 Hz, 1H), 2.73 (dd, J = 17.0, 5.3 Hz, 1H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 173.6, 172.0, 143.9, 143.2, 141.3, 141.1, 136.1, 126.8, 126.7, 126.4, 125.0, 124.3, 120.1, 119.9, 52.3, 51.8, 47.2, 37.8, 36.8. **HRMS** (ESI-TOF) *m/z*: $[M+Na]^+$ Calcd for $C_{19}H_{18}NaO_4$ 333.1102; Found 333.1096. **IR** (neat cm^{-1}) *v* 2955, 2923, 1925, 1195, 870.

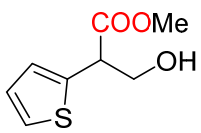


Methyl 2-(9H-fluoren-2-yl)-3-hydroxypropanoate (3aj). White solid, 47 mg, 58% yield, m.p.: 112 – 114 °C. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.78 (t, J = 8.5 Hz, 2H), 7.56 (d, J = 7.5 Hz, 1H), 7.49 (s, 1H), 7.40 (t, J = 7.5 Hz, 1H), 7.32 (dd, J = 15.5, 7.8 Hz, 2H), 4.22 (t, J = 10 Hz 1H), 4.00 – 3.93 (m, 1H), 3.91 – 3.88 (m, 3H), 3.76 (s, 3H), 2.51 (s, 1H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 173.7, 143.8, 143.2, 141.3, 141.0, 133.9, 126.81, 126.80, 126.7, 124.9, 124.7, 120.1, 119.8, 64.6, 53.9, 52.1, 36.7. **HRMS** (ESI-TOF) *m/z*: $[M+Na]^+$ Calcd for $C_{17}H_{16}NaO_3$ 291.0997; Found: 291.0994. **IR** (neat cm^{-1}) *v* 3661, 2953, 1736, 1166, 878.



Dimethyl 2-(thiophen-2-yl) succinate (2ak). Red oil, 8 mg, 12% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.21 (s, 1H), 6.96 – 6.94 (m, 2H), 4.38 (dd, J = 10.0, 5.3 Hz, 1H), 3.72 (s, 3H), 3.69 (s, 3H), 3.23 (dd, J

= 17.0, 10.0 Hz, 1H), 2.79 (dd, *J* = 16.9, 5.3 Hz, 1H). **^{13}C NMR** (101 MHz, Chloroform-*d*) *δ* 172.44, 171.49, 139.48, 126.91, 125.60, 124.94, 52.61, 51.97, 42.22, 38.09. **HRMS** (ESI-TOF) *m*/*z*: $[M+Na]^+$ Calcd for $C_{10}H_{12}NaO_4S$ 251.0354; Found 251.0345. **IR** (neat cm^{-1}) *v* 2953, 2920, 1738, 1728, 703.



Methyl 3-hydroxy-2-(thiophen-2-yl) propanoate (3ak). Red oil, 21 mg, 38% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) *δ* 7.25 – 7.24 (m, 1H), 6.99 – 6.98 (m, 2H), 4.13 (d, *J* = 2.2 Hz, 2H), 3.96 – 3.90 (m, 1H), 3.75 (s, 3H), 2.32 (s, 1H). **^{13}C NMR** (101 MHz, Chloroform-*d*) *δ* 172.58, 136.91, 126.90, 126.22, 125.20, 64.73, 52.50, 48.78. **HRMS** (ESI-TOF) *m*/*z*: $[M+Na]^+$ Calcd for $C_8H_{10}NaO_3S$ 209.0248; Found 209.0241. **IR** (neat cm^{-1}) *v* 3434, 2952, 2923, 1728, 850.

7. X-ray crystallographic data

Single crystals of **2a,** *trans*-**2l, 3v**, **2ah** was obtained by recrystallization from $CDCl_3$ at room temperature. The intensities were collected on a Bruker SMART APEX CCD diffractometer equipped with a graphite monochromated Mo−Kα (λ = 0.71073 Å) radiation source; the data were acquired using the SMART and SAINT programs. The structures were solved by direct methods and refined on F^2 by full matrix least-squares methods using the SHELXTL version 5.1 software.

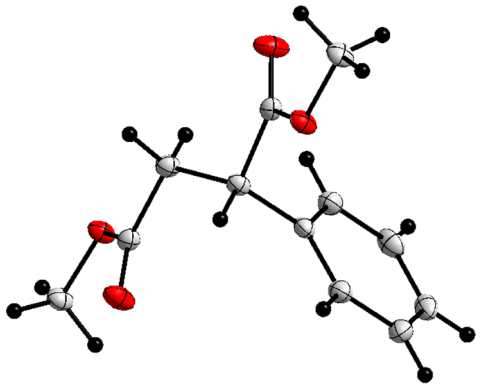


Figure S3. ORTEP plot of **2a**. Thermal ellipsoids are shown at 30% probability level.

Table S2. Crystal parameter of **2a**

Formula	$C_{12}H_{14}O_2$
Formula weight	255.3170
Crystal system	Monoclinic
Wavelength	0.71073
a (Å)	5.8827(3)
b (Å)	24.9167(9)
c (Å)	8.1339(3)
α (º)	90
β (º)	110.149(1)
γ (º)	90
Volume (Å^3)	1119.28(8)
Z	4
T (K)	120K
D calcd (g cm^{-3})	1.319
μ (mm^{-1})	0.099
F (000)	472.0
No. of rflns. collected	1951
No. of indep. rflns. /*R*int	8018/ 0.0176
No. of obsd. rflns. [$I_0 > 2\sigma(I_0)$]	1951
Data / restraints / parameters	1951/ 0 /145
R_1 / $wR2$ [$I_0 > 2\sigma(I_0)$]	0.0409 (1785)
R_1 / $wR2$ (all data)	0.1014 (1951)
GOF (on F^2)	1.043
CCDC No.	2234688

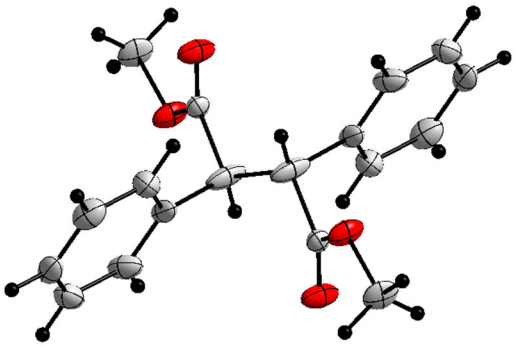


Figure S4. ORTEP plot of *trans*-**2l**. Thermal ellipsoids are shown at 30% probability level.

Table S3. Crystal parameter of *trans*-**2l**

Formula	$C_{18}H_{18}O_4$
Formula weight	298.1205
Crystal system	Monoclinic
Wavelength	0.71073
a (Å)	5.8450(12)
b (Å)	17.181(3)
c (Å)	7.9441(15)
α (º)	90
β (º)	107.273(5)
γ (º)	90
Volume (Å^3)	761.8(3)
Z	2
T (K)	292K
D calcd (g cm^{-3})	1.301
μ (mm^{-1})	0.091
F (000)	316.0
No. of rflns. collected	1343
No. of indep. rflns. /*R*int	5821/0.1244
No. of obsd. rflns. [$I_0 > 2\sigma(I_0)$]	1343
Data / restraints / parameters	1343/ 0 /101
R_1 / $wR2$ [$I_0 > 2\sigma(I_0)$]	0.2097 (795)
R_1 / $wR2$ (all data)	0.2355 (1343)
GOF (on F^2)	1.099
CCDC No.	2234689

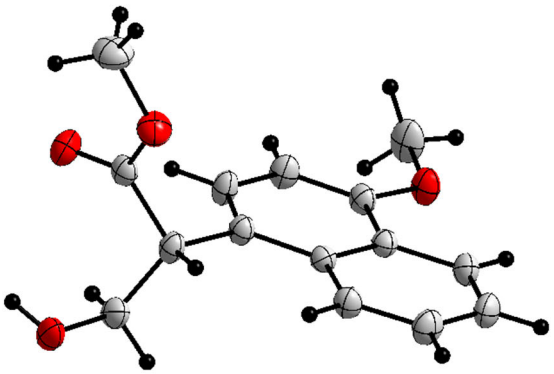


Figure S5. ORTEP plot of **3v**. Thermal ellipsoids are shown at 30% probability level.

Table S4. Crystal parameter of **3v**

Formula	$C_{15}H_{16}O_4$
Formula weight	260.2890
Crystal system	Monoclinic
Wavelength	1.54178
a (Å)	10.8280(12)
b (Å)	7.9485(9)
c (Å)	15.2171(18)
α (º)	90
β (º)	95.439(6)
γ (º)	90
Volume (Å^3)	1303.8(3)
Z	4
T (K)	297 K
D calcd (g cm^{-3})	1.326
μ (mm^{-1})	0.790
F (000)	552.0
No. of rflns. collected	2632
No. of indep. rflns. /Rint	10313/ 0.0444
No. of obsd. rflns. [$I_0 > 2\sigma(I_0)$]	2632
Data / restraints / parameters	2632/ 0 /175
R_1 / $wR2$ [$I_0 > 2\sigma(I_0)$]	0.0475(2194)
R_1 / $wR2$ (all data)	0.1431(2632)
GOF (on F^2)	1.068
CCDC No.	2245962

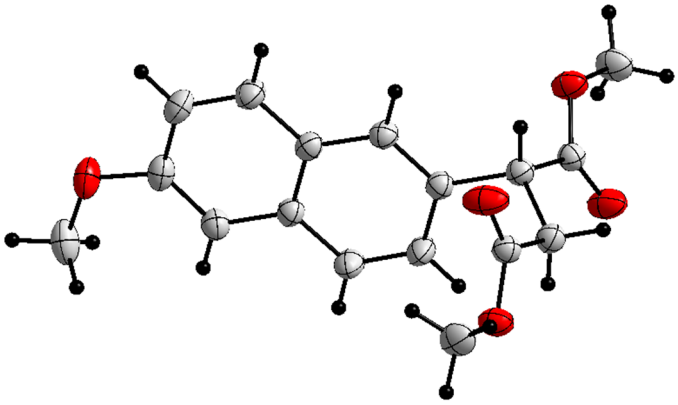


Figure S6. ORTEP plot of **2ah**. Thermal ellipsoids are shown at 30% probability level.

Table S5. Crystal parameter of **2ah**

Formula	$C_{17}H_{18}O_5$
Formula weight	302.1154
Crystal system	Monoclinic
Wavelength	0.71073
a (Å)	5.8137(4)
b (Å)	8.5017(5)
c (Å)	16.6639(10)
α (º)	99.886(3)
β (º)	92.872(3)
γ (º)	108.302(3)
Volume (Å^3)	761.8(3)
Z	2
T (K)	297K
D calcd (g cm^{-3})	1.311
μ (mm^{-1})	0.799
F (000)	320.0
No. of rflns. collected	3034
No. of indep. rflns. /Rint	7734/0.0264
No. of obsd. rflns. [$I_0 > 2\sigma(I_0)$]	3034
Data / restraints / parameters	3034/ 0 /202
R_1 / $wR2$ [$I_0 > 2\sigma(I_0)$]	0.0523(2583)
R_1 / $wR2$ (all data)	0.1730(3034)
GOF (on F^2)	1.092
CCDC No.	2245959

8. Synthetic applications

Diacid product **2a'** can be efficiently converted to succinic anhydride **4a** with a 69% yield.[4] The reduction of **2a'** with $LiAlH_4$ gave product **5a**, which can be further converted into Phensuximide, an antispasmodic succinimide.[5] Additionally, oxidative cyclization of **2a'** produced phenylmaleic anhydride 6a with an outstanding 99% yield.[6] **2a'** can also be efficiently transformed via an intramolecular Friedel–Crafts reaction to produce compound **7a**, which is a crucial intermediate to prepare novel analgesics.[7]

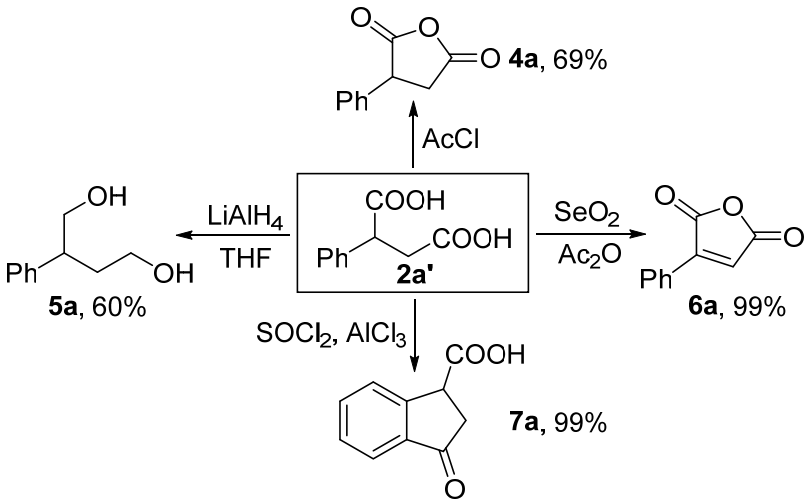
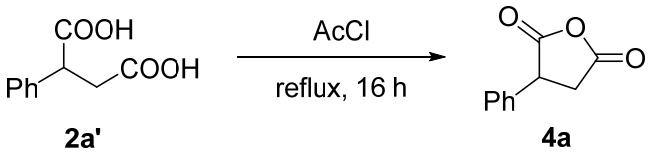
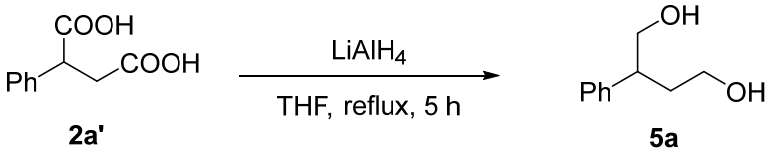


Figure S7. Synthetic applications of diacid products



A 50 mL round-bottomed flask contained a stirring bar was charged with phenyl succinic acid **2a** (500 mg, 2.5 mmol). Freshly distilled acetyl chloride (5 mL) was added, the flask was fitted with a condenser and the reaction mixture was heated at reflux temperature (oil bath) under an argon atmosphere for 16 h. The solvents were removed under reduced pressure and the crude residue was purified by flash column chromatography (petroleum ether/ethyl acetate 7/1/) to give desired product **4a**.

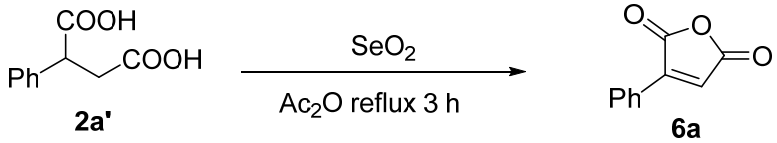
3-Phenyldihydrofuran-2,5-dione (4a).[4] White solid, 310 mg, 69% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.42 – 7.25 (m, 3H), 7.26 (d, J = 7.9 Hz, 2H), 4.34 (dd, J = 10.3, 6.5 Hz, 1H), 3.45 (ddd, J = 18.9, 10.3, 1.2 Hz, 1H), 3.10 (ddd, J = 19.0, 6.6, 1.3 Hz, 1H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 171.6, 169.5, 134.5, 129.4, 128.5, 127.2, 46.4, 36.5.



2a' **5a**

The **2a** (1.35 mmol) was dissolved in THF (5 mL) under argon and the reaction mixture was cooled to 0 °C. $LiAlH_4$ (4 mmol dissolved in 2 mL THF) was added dropwise with stirring. The mixture is heated to reflux for 4 h (oil bath) and cooled to 0 ºC afterwards. The mixture was carefully quenched with water and extracted three times with ethyl acetate (3 × 20 mL). The combined extracts were washed with brine, and dried over anhydrous Na_2SO_4. The solvents were removed under reduced pressure and the crude residue was washed with cold Et_2O to give the pure desired product.

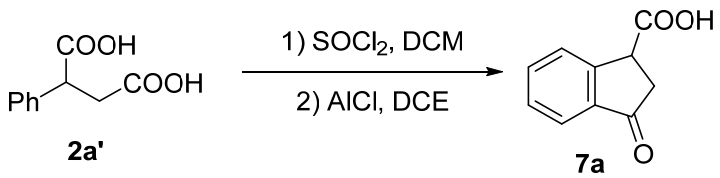
2-Phenylbutane-1,4-diol (5a).[8] White solid, 134 mg, 60% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.28 (t, J = 7.5 Hz, 2H), 7.20 (d, J = 7.1 Hz, 1H), 7.16 (d, J = 7.7 Hz, 2H), 3.71 – 3.43 (m, 6H), 2.88 (p, J = 6.7 Hz, 1H), 1.99 (td, J = 14.0, 6.1 Hz, 1H), 1.81 (ddd, J = 13.9, 8.9, 4.5 Hz, 1H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 142.3, 128.5, 127.7, 126.6, 67.2, 60.6, 45.6, 35.6.



2a' **6a**

To a mechanically stirred slurry of selenium dioxide (400 mg, 3.6 mmol) in freshly distilled acetic anhydride (5 mL) was added phenyl succinic acid (450 mg, 2.4mol), and this mixture was heated at reflux (oil bath) for 3 h, during which time it turned from yellow to black. While still hot, the mixture was filtered through a celite pad to remove precipitated selenium salts. Subsequent solvent evaporation afforded crude product. Recrystallization from EA-PE afforded **6a** as pale-yellow crystals.

Phenylmaleic anhydride (6a).[9] Pale-yellow crystals, 403 mg, 99% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.97 (d, J = 7.6 Hz, 2H), 7.53 (dt, J = 15.1, 7.4 Hz, 3H), 7.00 (s, 1H). **^{13}C NMR** (101 MHz, Chloroform-*d*) δ 164.5, 163.6, 146.8, 132.7, 129.3, 128.9, 126.8, 124.4.



2a' **7a**

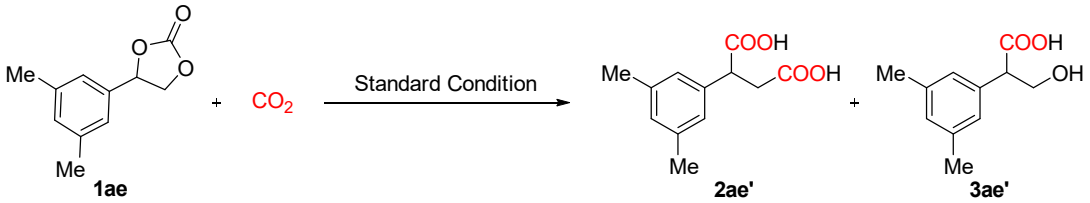
An oven-dried 50 mL three-neck bottle containing a condenser and a Teflon-coated stir bar was charged with 2-phenylsuccinic acid **2a** (0.49 g, 2.5 mmol). The reaction flask was then

sealed with rubber septum, charged with nitrogen gas. After adding 10 mL of dichloromethane (DCM) and thionyl chloride (0.3 mL, 4 mmol) sequentially, the temperature was raised to 75 °C (oil bath). Once the 2-phenylsuccinic acid **2a** was totally dissolved, the solvent was removed in vacuo using a Dean-Stark trap. Charged the reaction vessel with 10 mL of 1,2-dichloroethane (DCE) followed by adding 0.7 g (5 mmol) $AlCl_3$ partitively. Stirred the reaction mixture at room temperature for 13 h and raised the temperature to 60 °C for 4.5 h. The reaction was quenched with 6 M HCl (5 mL) and water (10 mL), extracted with AcOEt, then concentrated in vacuo. The residue was purified by silica gel flash chromatography (PE/AcOEt = 2:1) to give the pure desired product **7a**.

3-Oxo-2,3-dihydro-1H-indene-1-carboxylic acid (7a).[10] White solid, 441 mg, 99%.**^{1}H NMR** (400 MHz, DMSO-d_6) δ 7.73 (d, J = 4.7 Hz, 2H), 7.67 (d, J = 7.6 Hz, 1H), 7.51 (ddd, J = 7.9, 5.5, 2.2 Hz, 1H), 4.32 (t, J = 5.6 Hz, 1H), 2.87 (d, J = 4.9 Hz, 2H). **^{13}C NMR** (101 MHz, DMSO-d_6) δ 173.7, 172.5, 152.5, 136.3, 135.5, 129.0, 127.1, 123.5, 43.9, 21.4.

9. Mechanistic studies

9.1 Detection of products ratio over reaction time.



A 50 mL oven-dried electrochemical cell containing a stir bar was evacuated and back-filled under CO_2 flow for at least 3 times. **1ae** (0.3 mmol), $TBABF_4$ (0.3 mmol) and DMF (5 mL) were added to the cell and the cell was installed with a Pt cathode and Mg anode, and the cell was closed under CO_2 atmosphere. Then, the reaction mixture was electrolyzed under a constant current of 10 mA for 4 h. Take samples every half hour, carefully quenched with HCl (1M, 1 mL) and extracted with ethyl acetate. The solvents were removed under reduced pressure and the crude residue was detected by ^1H NMR.

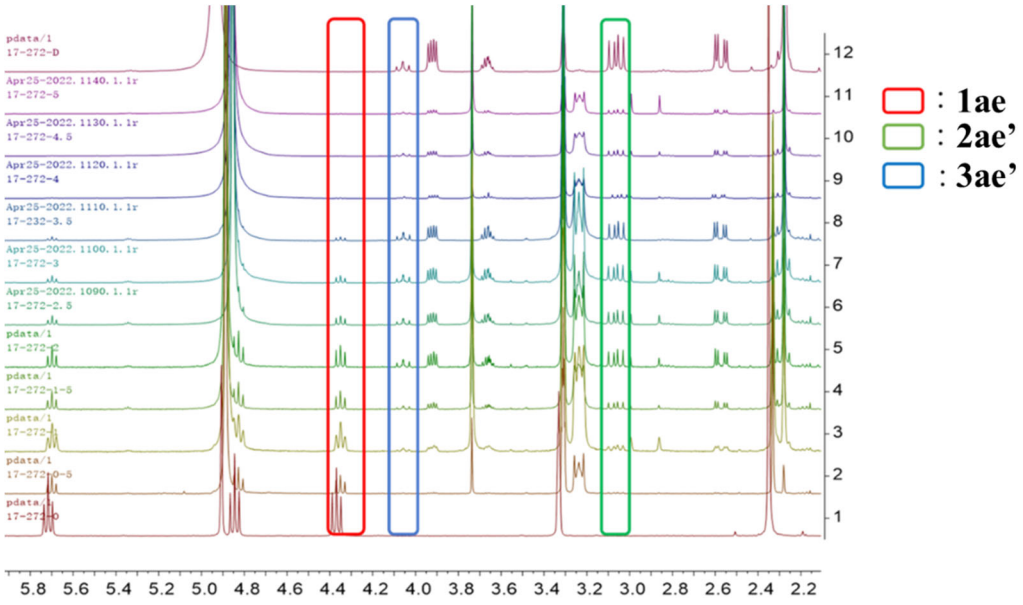
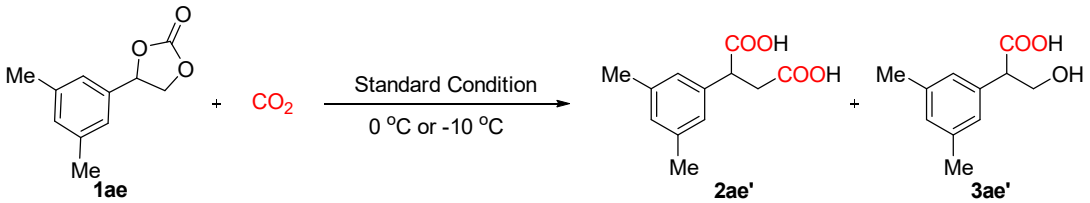


Figure S8. ^1H NMR spectrum of different times for reaction of **1ae**.



A 50 mL oven-dried electrochemical cell containing a stir bar was evacuated and back-filled under CO_2 flow for at least 3 times. **1ae** (0.3 mmol), $TBABF_4$ (0.3 mmol) and DMF (5

mL) were added to the cell and the cell was installed with a Pt cathode and Mg anode, and the cell was closed under CO_2 atmosphere. Then, the reaction mixture was electrolyzed under a constant current of 10 mA for 4 h under different temperature. After the reaction finished, the reaction was carefully quenched with HCl (1M, 1 mL) and extracted with ethyl acetate. The solvents were removed under reduced pressure and the crude residue was detected by ^{1}H NMR.

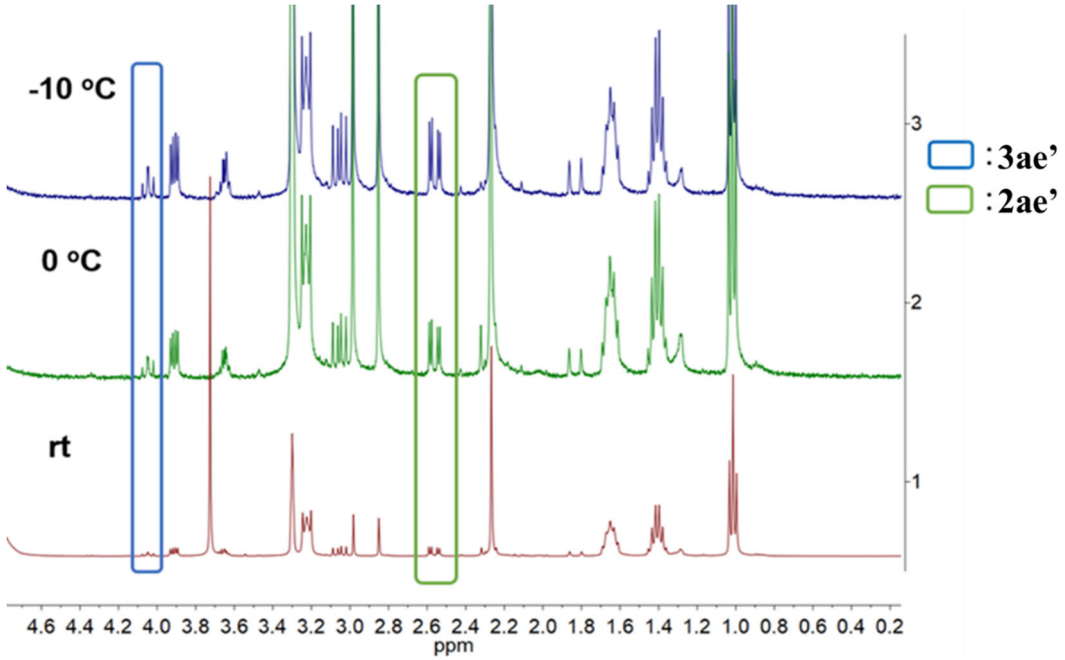


Figure S9. ^{1}H NMR spectrum of different temperature for reaction of **1ae**.

9.2 Cyclic voltammetry studies

Cyclic voltammograms were recorded with a CHI760E potentiostat at room temperature (20 ºC) in DMF. $TBABF_4$ was used as the supporting electrolyte, and a Glass Carbon electrode (d = 3 mm) was used as the working electrode. The auxiliary electrode was a Pt sheet (10 mm × 10mm × 0.3 mm). All potentials are referenced against the Ag/AgCl redox couple. The scan rate is 100 mV s^{-1}. The solution was purged with Ar flow for 5 minutes before the measurement. Polishing of Glass Carbon electrode can be accomplished by putting a few drops of water/1.0-micron alumina slurry onto a polishing pad. The electrode is polished using a figure 8 motion to avoid grooving the electrode. Once the electrode has been polished, it should be rinsed with water, then sonicated in acetone, ethanol or distilled water for few mins to remove any residual particulate matter if present.

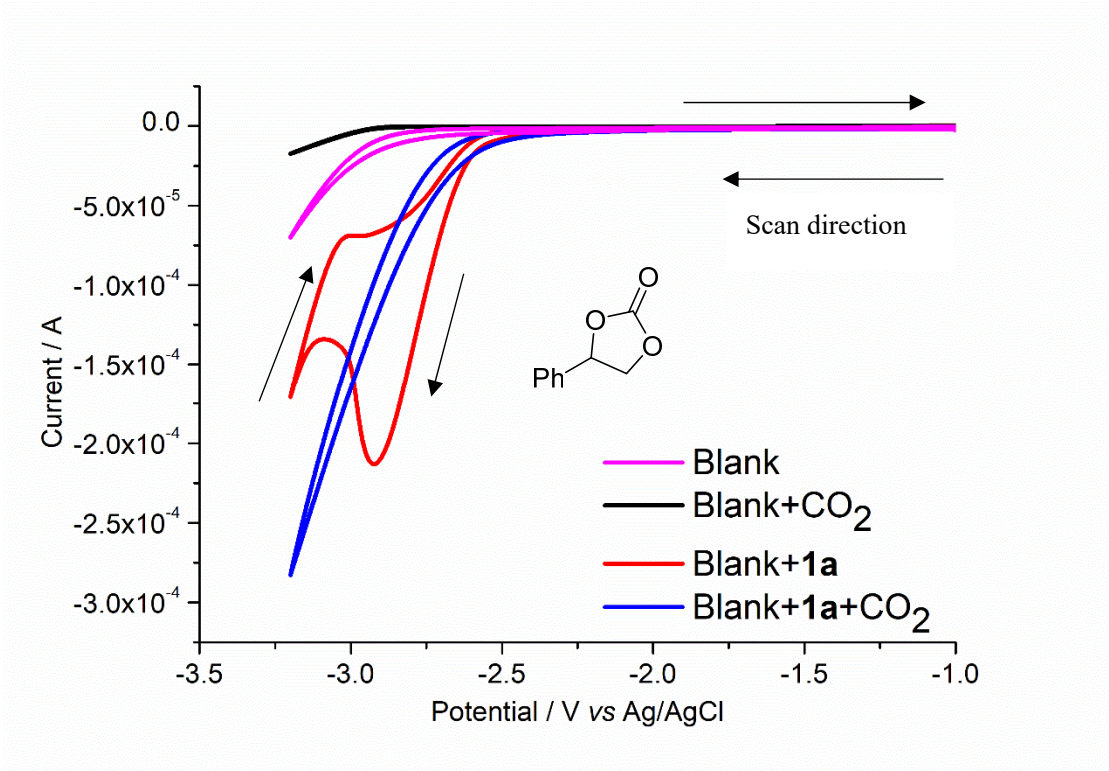


Figure S10. Cyclic voltammograms recorded on a Glass Carbon electrode (d = 3 mm). $TBABF_4$ (0.3 mmol), DMF (5 mL), **1a** (0.3 mmol). (maintaining IUPAC convention)

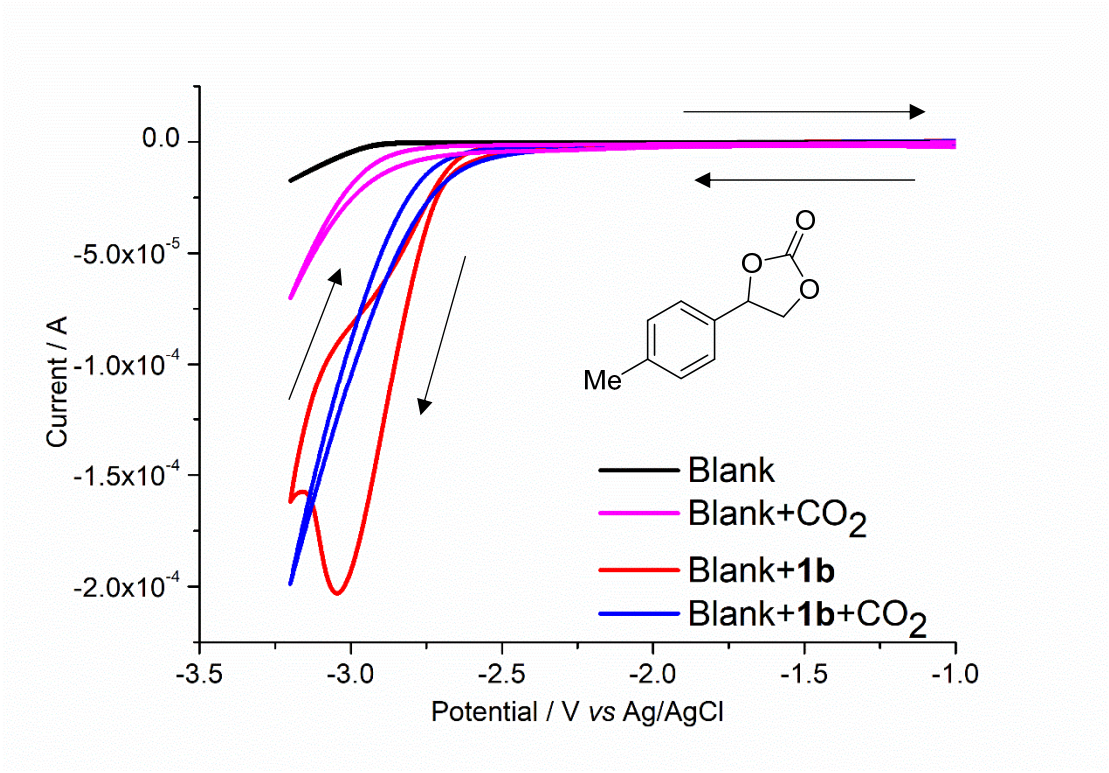


Figure S11. Cyclic voltammograms recorded on a Glass Carbon electrode (d = 3 mm). $TBABF_4$ (0.3 mmol), DMF (5 mL), **1b** (0.3 mmol). (maintaining IUPAC convention)

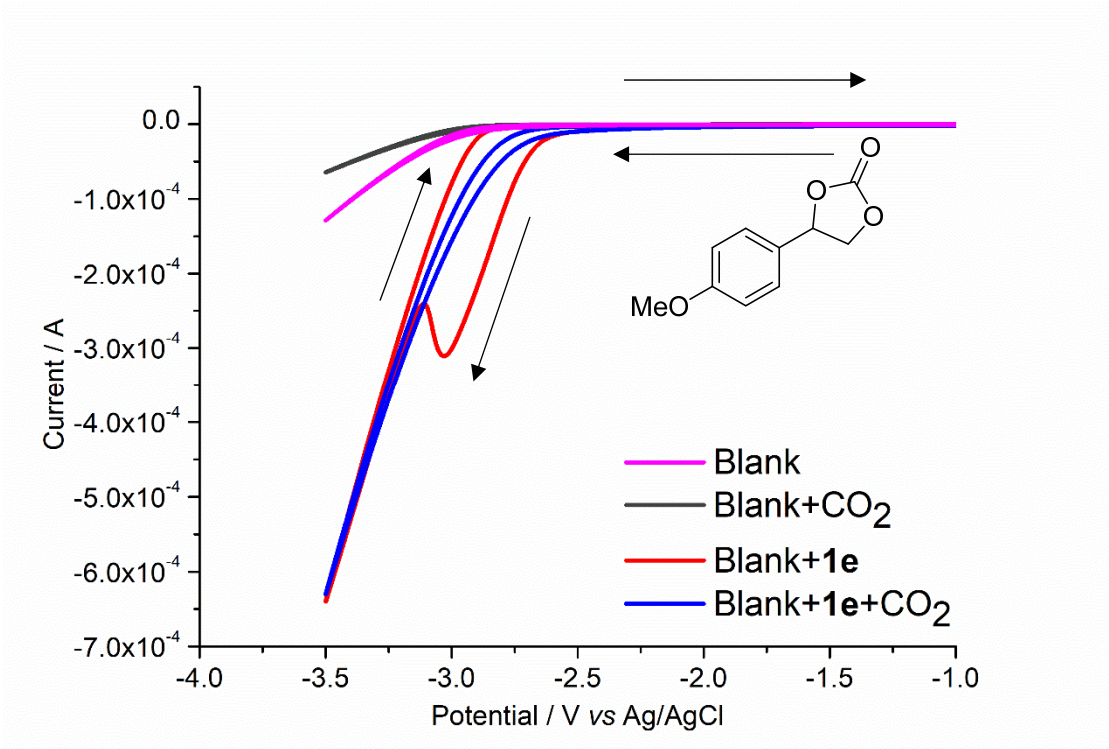


Figure S12. Cyclic voltammograms recorded on a Glass Carbon electrode (d = 3 mm). $TBABF_4$ (0.3 mmol), DMF (5 mL), **1e** (0.3 mmol). (maintaining IUPAC convention)

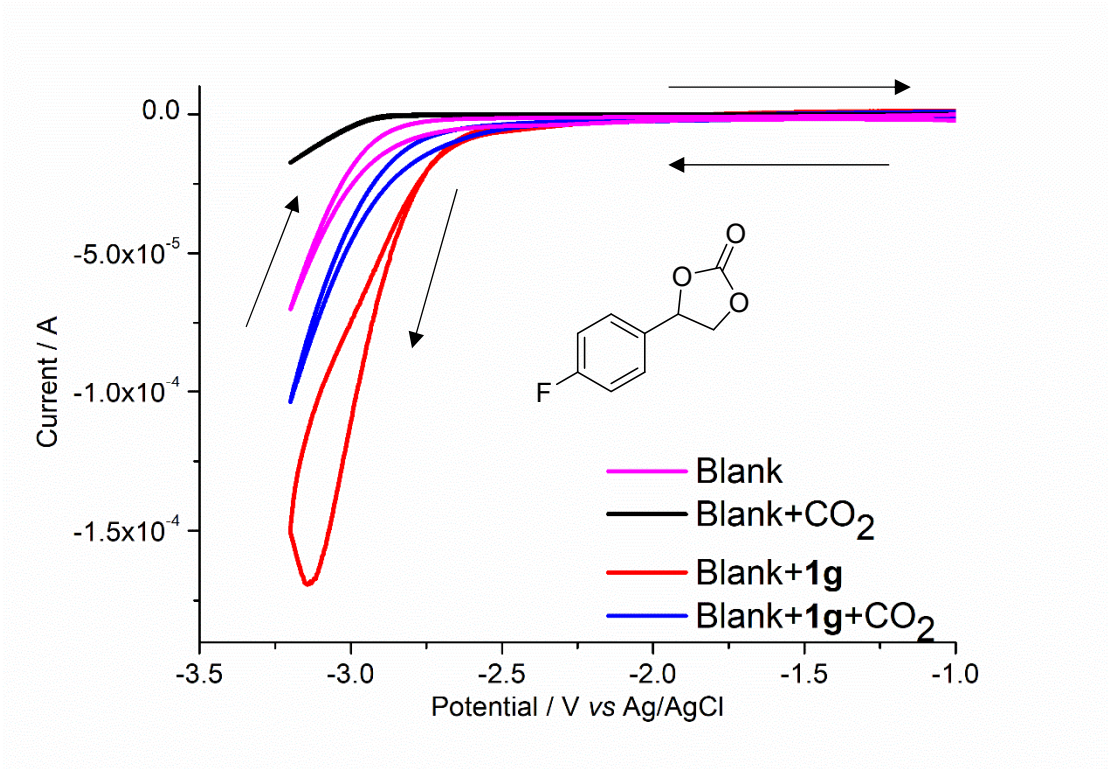


Figure S13. Cyclic voltammograms recorded on a Glass Carbon electrode (d = 3 mm). $TBABF_4$ (0.3 mmol), DMF (5 mL), **1g** (0.3 mmol). (maintaining IUPAC convention)

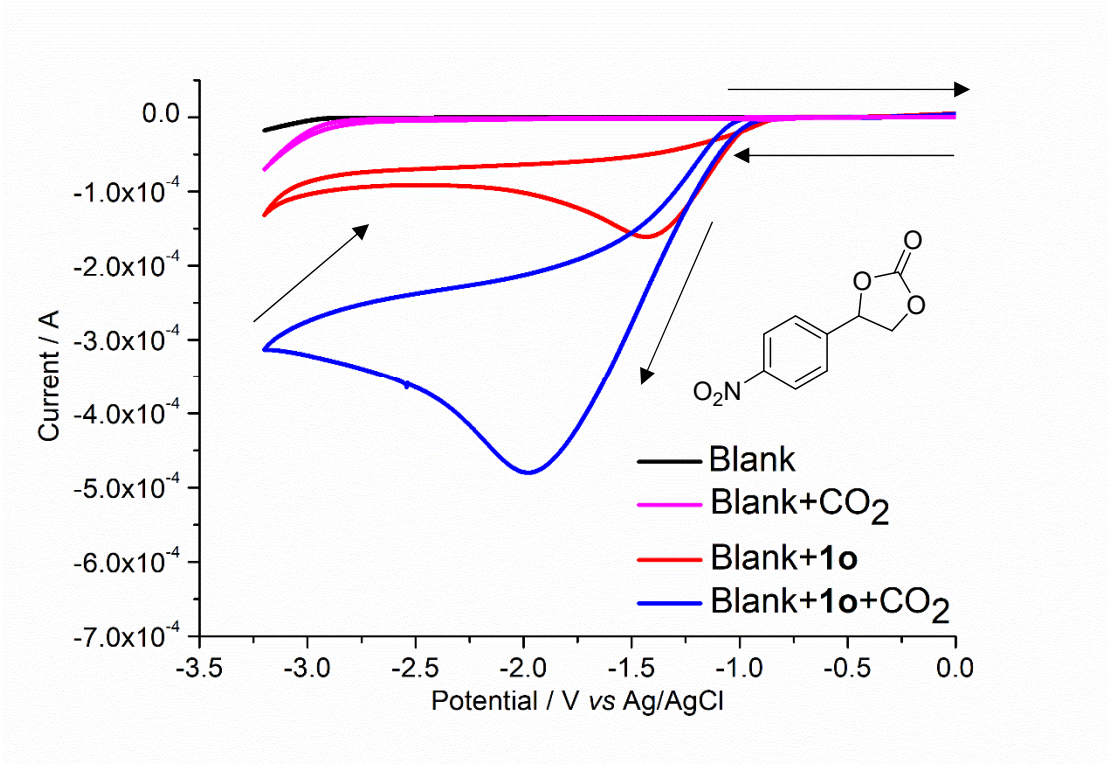


Figure S14. Cyclic voltammograms recorded on a Glass Carbon electrode (d = 3 mm). $TBABF_4$ (0.3 mmol), DMF (5 mL), **1o** (0.3 mmol). (maintaining IUPAC convention)

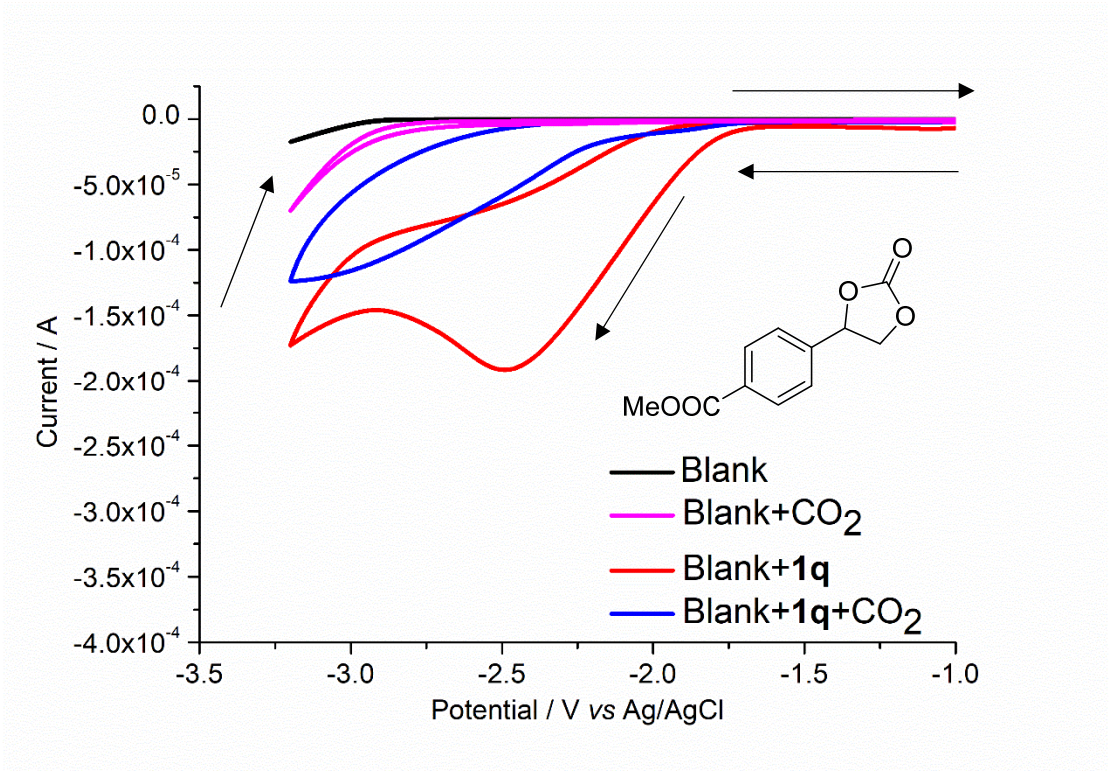


Figure S15. Cyclic voltammograms recorded on a Glass Carbon electrode (d = 3 mm). $TBABF_4$ (0.3 mmol), DMF (5 mL), **1q** (0.3 mmol). (maintaining IUPAC convention)

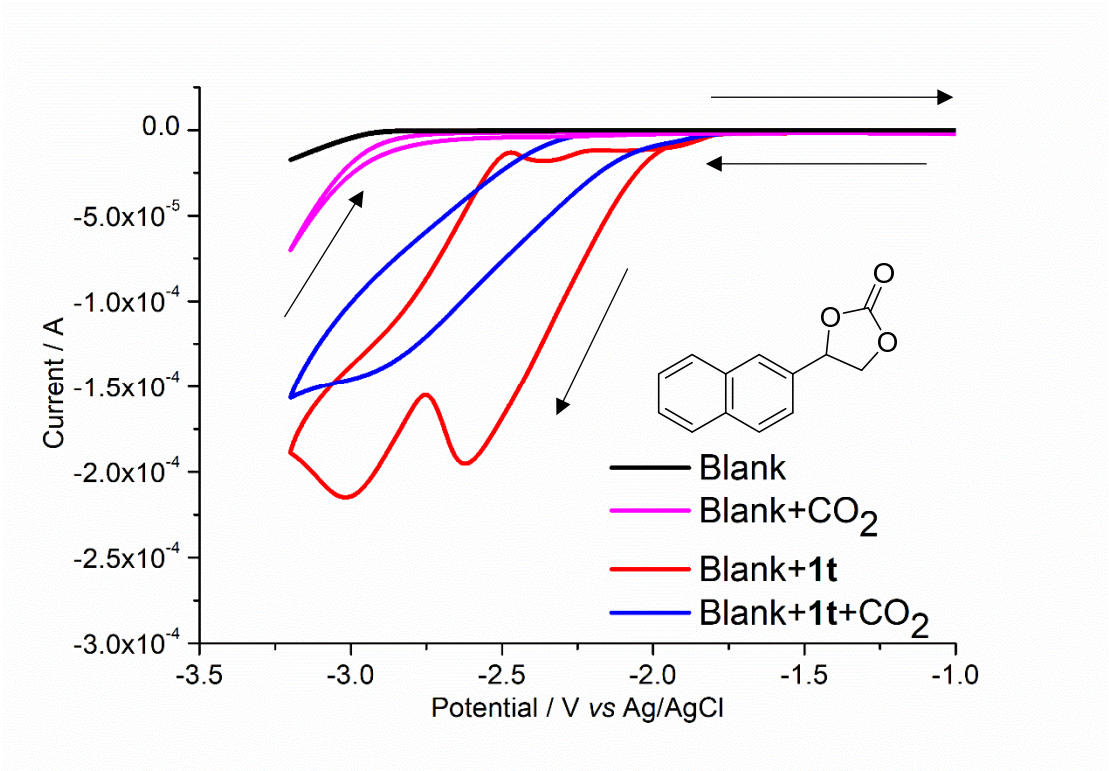


Figure S16. Cyclic voltammograms recorded on a Glass Carbon electrode (d = 3 mm). $TBABF_4$ (0.3 mmol), DMF (5 mL), **1t** (0.3 mmol). (maintaining IUPAC convention)

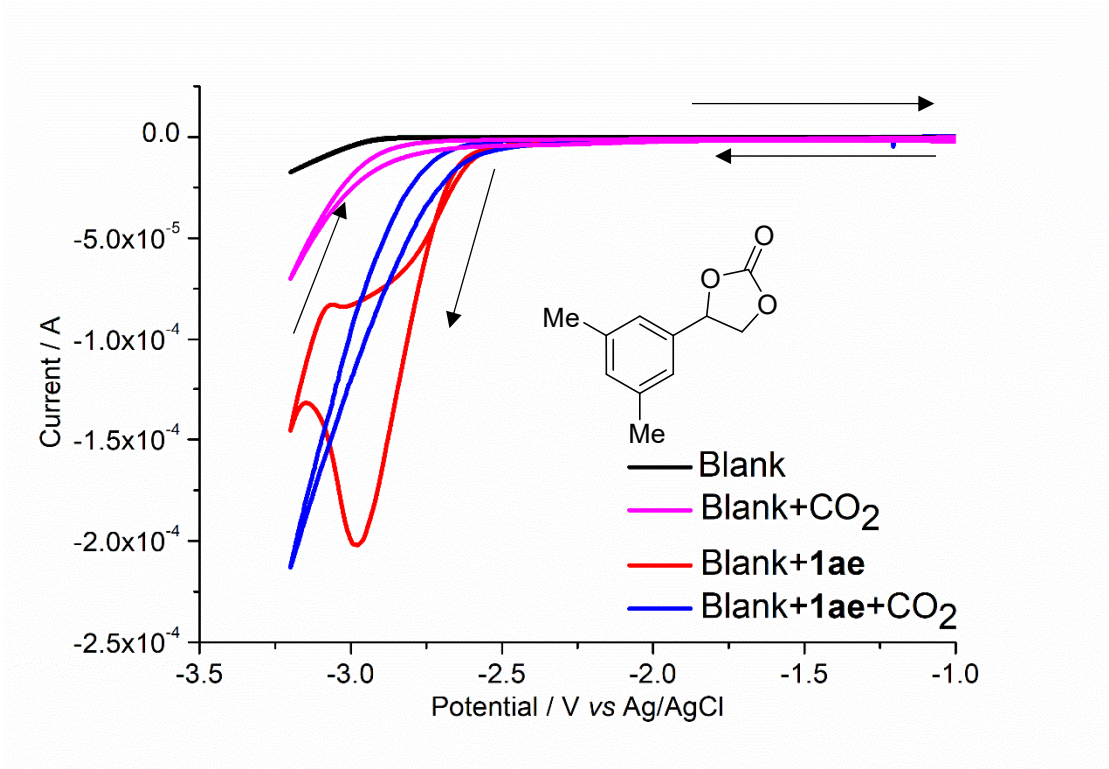


Figure S17. Cyclic voltammograms recorded on a Glass Carbon electrode (d = 3 mm). $TBABF_4$ (0.3 mmol), DMF (5 mL), **1ae** (0.3 mmol). (maintaining IUPAC convention)

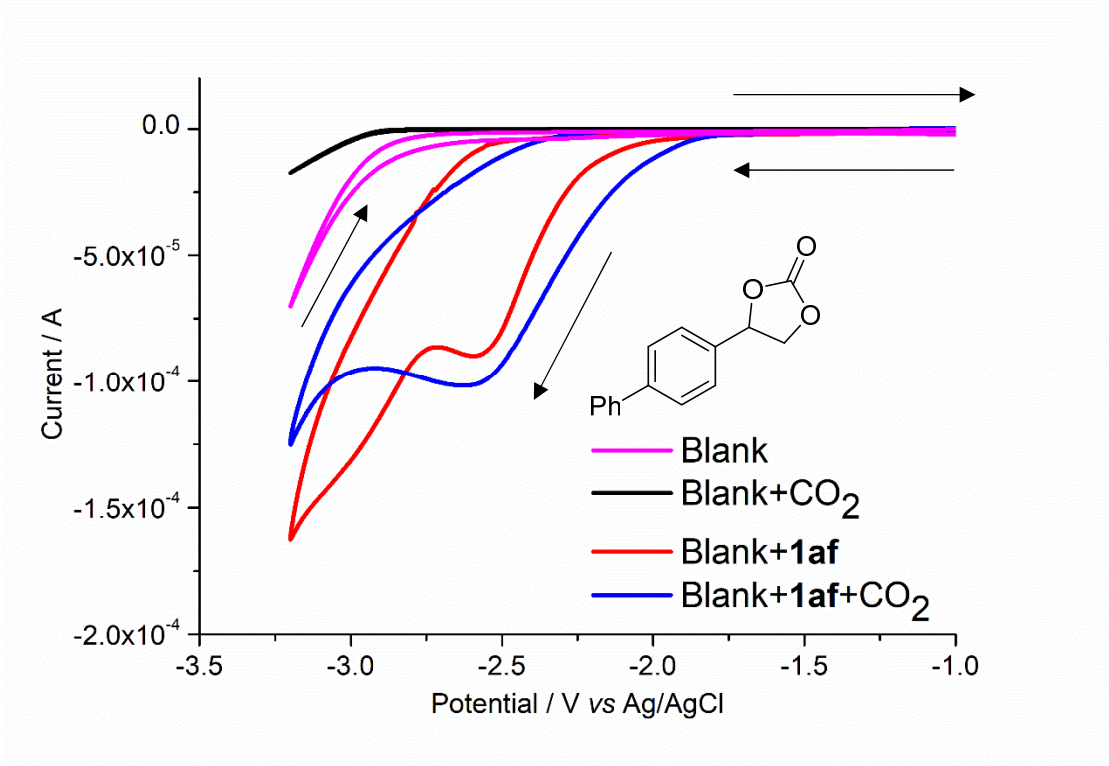
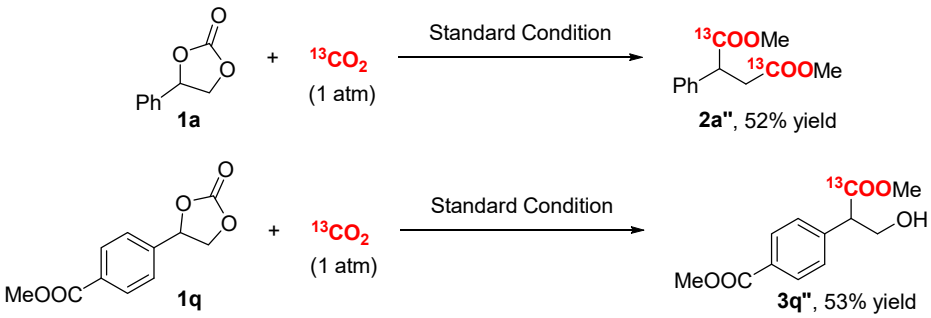


Figure S18. Cyclic voltammograms recorded on a Glass Carbon electrode (d = 3 mm). $TBABF_4$ (0.3 mmol), DMF (5 mL), **1af** (0.3 mmol). (maintaining IUPAC convention)

9.3 $^{13}CO_2$ isotope labeling experiment



A 50 mL oven-dried electrochemical cell containing a stir bar was evacuated and back-filled under Ar flow for at least 3 times. Cyclic carbonate (0.3 mmol), $TBABF_4$ (0.3 mmol) and DMF (5 mL) were added to the cell and the cell was installed with a Pt cathode and Mg anode, and the cell was closed under Ar atmosphere. Then, the cell was evacuated and back-filled under $^{13}CO_2$. The reaction mixture was electrolyzed under a constant current of 10 mA for 6 h. The reaction mixture was carefully quenched with HCl (1M, 10 mL) and extracted three times with ethyl acetate (3 × 20 mL). The combined organic layers were washed with brine (2 × 50 mL) and dried over Na_2SO_4. The solvents were removed under reduced pressure and the residue was methylated at 0°C with $TMSCHN_2$ (2 M in hexane, 0.6 mL) in 2 mL of ether/methanol (1:1 v/v) solvent. The crude residue was purified by flash column chromatography (petroleum ether/ethyl acetate 7/1 to 3/1) to give desired product.

The calculation for incorporation rates of ^{13}C by mass spectrometry

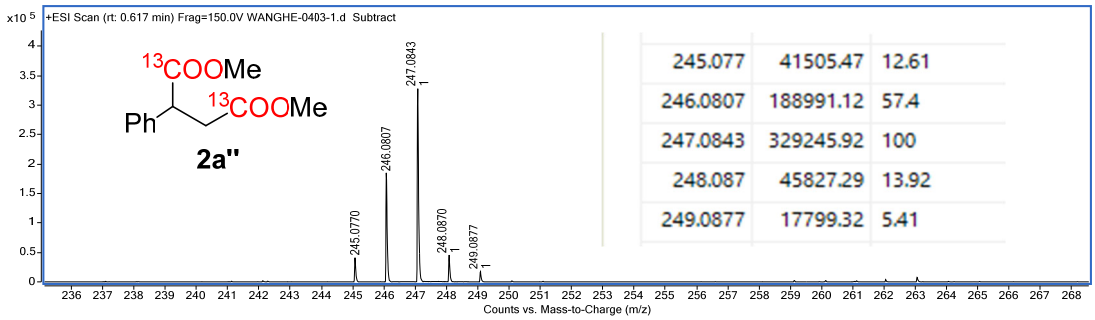
The theoretical value for the unlabelled product **2a** and ^{13}C labeled product **2a"**

	m/z	Abund (% largest)	Abund (% sum)	Abund (% first)
▶	245.0784	100	86.9	100
	246.0818	13.29	11.55	13.29
	247.0839	1.64	1.42	1.64
	248.0866	0.14	0.12	0.14
	249.089	0.01	0.01	0.01
	250.0915	0	0	0
*				

	m/z	Abund (% largest)	Abund (% sum)	Abund (% first)
▶	246.0818	100	87.84	100
	247.0852	12.21	10.73	12.21
	248.0872	1.5	1.32	1.5
	249.0899	0.12	0.11	0.12
	250.0922	0.01	0.01	0.01
	251.0947	0	0	0
*				

	m/z	Abund (% largest)	Abund (% sum)	Abund (% first)
▶	247.0851	100	88.79	100
	248.0885	11.13	9.88	11.13
	249.0904	1.38	1.23	1.38
	250.0932	0.11	0.1	0.11
	251.0955	0.01	0.01	0.01
	252.0979	0	0	0

The measured spectrum for **2a"**



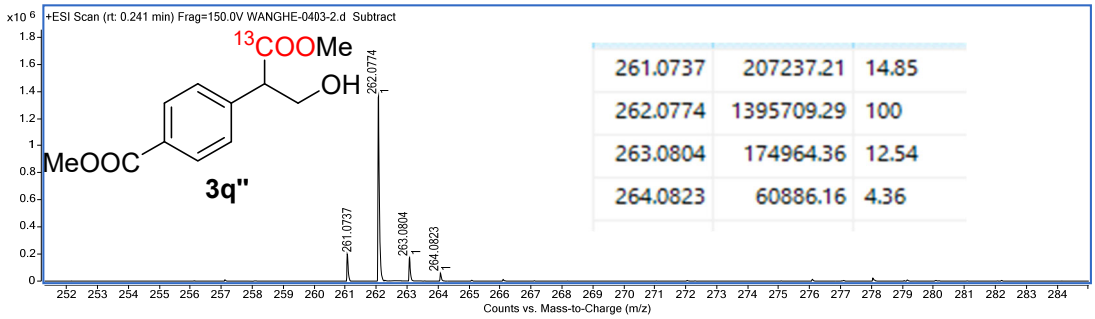
245.077	41505.47	12.61
246.0807	188991.12	57.4
247.0843	329245.92	100
248.087	45827.29	13.92
249.0877	17799.32	5.41

The theoretical value for the unlabelled product **3q** and ^{13}C labeled product **3q"**

	m/z	Abund (% largest)	Abund (% sum)	Abund (% first)
▶	261.0733	100	86.69	100
	262.0767	13.33	11.56	13.33
	263.0787	1.85	1.6	1.85
	264.0815	0.17	0.14	0.17
	265.0837	0.01	0.01	0.01
	266.0862	0	0	0
*				

	m/z	Abund (% largest)	Abund (% sum)	Abund (% first)
▶	262.0767	100	87.62	100
	263.0801	12.25	10.73	12.25
	264.082	1.71	1.5	1.71
	265.0848	0.15	0.13	0.15
	266.087	0.01	0.01	0.01
	267.0895	0	0	0
*				

The measured spectrum for **3q"**

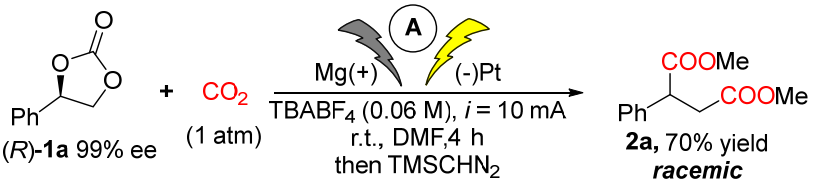


261.0737	207237.21	14.85
262.0774	1395709.29	100
263.0804	174964.36	12.54
264.0823	60886.16	4.36

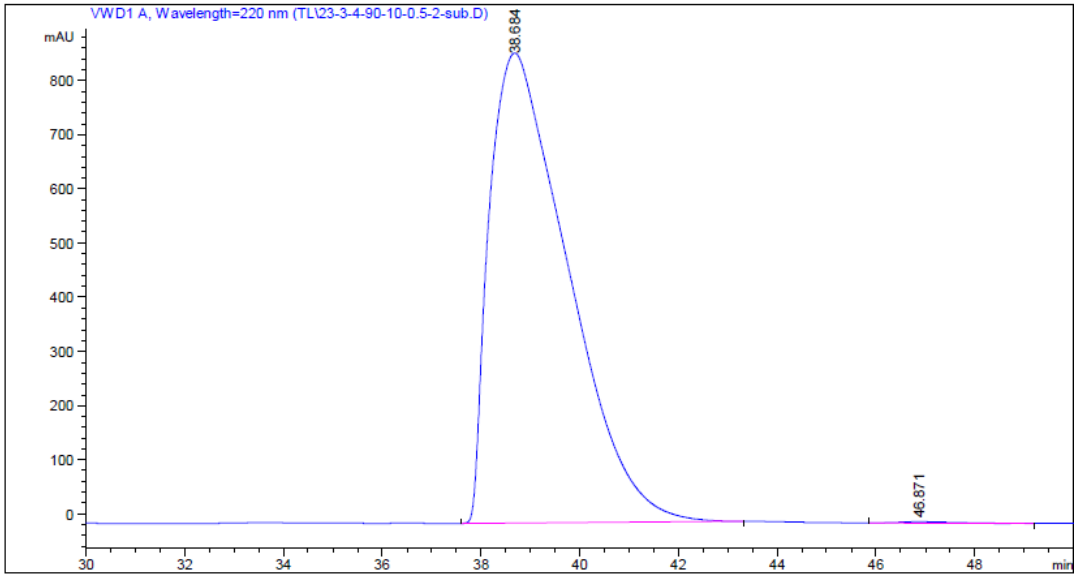
We use **3q"** as an example to calculate the ^{13}C labeling ratio. The method is as follows: According to the table, it is observed that the monoisotopic peak (A) of the unlabelled product has an *m/z* of 261.0733, while the A+1 peak has an *m/z* of 262.0767. The ratio of the monoisotopic peak to its A+1 peak is 100/13.33, indicating when the monoisotopic peak has an abundance of 100, the contribution of its A+1 peak is 13.33. In our measured spectrum, the relative height of the unlabelled monoisotopic peak A is observed to be 14.85. Therefore, the contribution of its A+1 peak is calculated as 14.85 * 13.33 / 100 = 1.975. Consequently, the contribution of the ^{13}C-labelled compound to the A+1 peak is 100 - 1.975 = 98.025. Thus, the ^{13}C incorporation ratio is estimated to be approximately 98.025 / (98.025 + 14.85) = 87%. To verify the accuracy of our calculation method, we applied the same method to deduce the experimental spectrum data. According to the table, when the relative abundance of the monoisotopic peak A of the ^{13}C-labeled product is 100, its contribution to the A+1 peak is 12.54. In the measured spectrum, the relative abundance of the monoisotopic peak labeled with ^{13}C is

98.025. Therefore, the contribution of its A+1 peak is calculated as 98.025 * 12.54 / 100 = 12.29. This calculated value is close to the measured value, indicating the feasibility of this method. Applying the same method, for the **2a''** peak, the proportion of singly labeled isotopes is approximately 35%, while the proportion of doubly labeled isotopes is approximately 58%.

9.4 Stereochemical study



A 50 mL oven-dried electrochemical cell containing a stir bar was evacuated and back-filled under CO_2 flow for at least 3 times. (*R*)-**1a** (0.3 mmol), $TBABF_4$ (0.3 mmol) and DMF (5 mL) were added to the cell and the cell was installed with a Pt cathode and Mg anode and the cell was closed under CO_2 atmosphere. Then, the reaction mixture was electrolyzed under a constant current of 10 mA for 4 h. Then the mixture was carefully quenched with HCl (1 M, 10 mL) and extracted three times with ethyl acetate (3 × 20 mL). The combined extracts were washed with brine (2 × 50 mL) and dried over Na_2SO_4. The solvents were removed under reduced pressure and the crude residue was dissolved in THF (1 mL) and methanol (1 mL) under Ar. $TMSCHN_2$ (0.6 mL, 2 M in Hexane) was added dropwise at 0 ºC for 0.5 h. The mixture was quenched with water, extracted with ethyl acetated and dried over Na_2SO_4. The solvents were removed under reduced pressure and the crude residue was purified by prepared TLC. CHIRALPAK OD-H, 25 ºC, ^{i}PrOH-hexanes 10.0/90.0, 0.5 mL/min, 220 nm. t_R = 38.6 min, *ts* = 46.8 min.Chiral HPLC: CHIRALPAK OD-H, 25 ºC, ^{i}PrOH-hexanes 5.0/95.0, 0.25 mL/min, 220 nm. t_R = 38.6 min, t_S = 52.1 min.



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Area Percent Report

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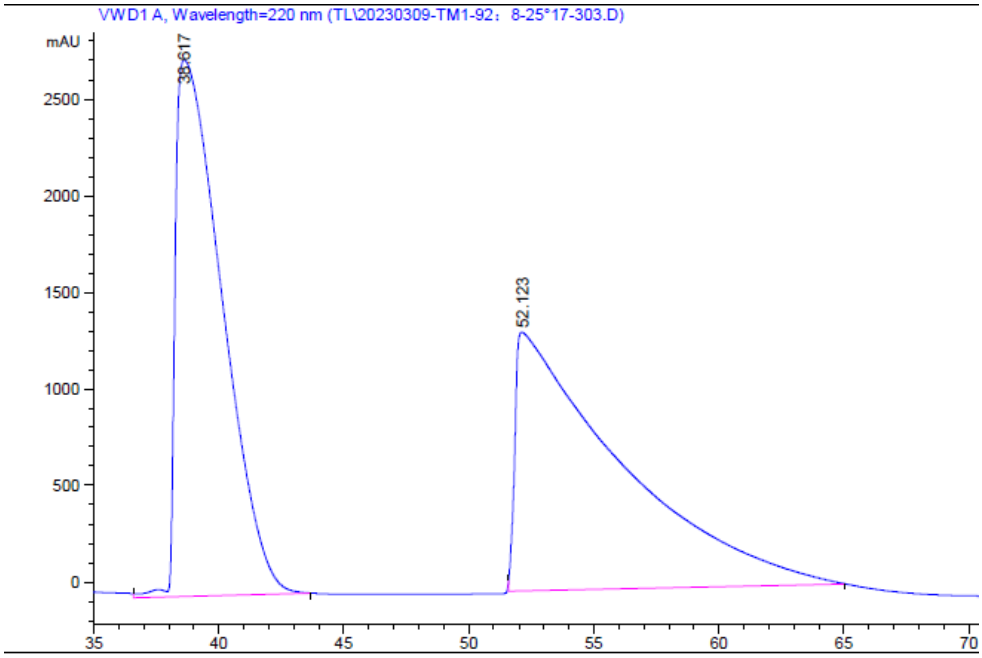
Sorted by : Signal
Multiplier : 1.0000
Dilution : 1.0000
Used multiplier & dilution factor

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	38.684	BB	1.6166	9.56472e4	867.31287	99.7739
2	46.871	BB	1.0908	216.77664	2.51446	0.2261

Figure S19. HPLC of (*R*)-**1a**



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Area Percent Report

==========

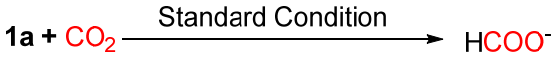
Sorted by : Signal
Multiplier : 1.0000
Dilution : 1.0000
Used multiplier & dilution factor

Signal 1: VWD1 A, Wavelength=220 nm

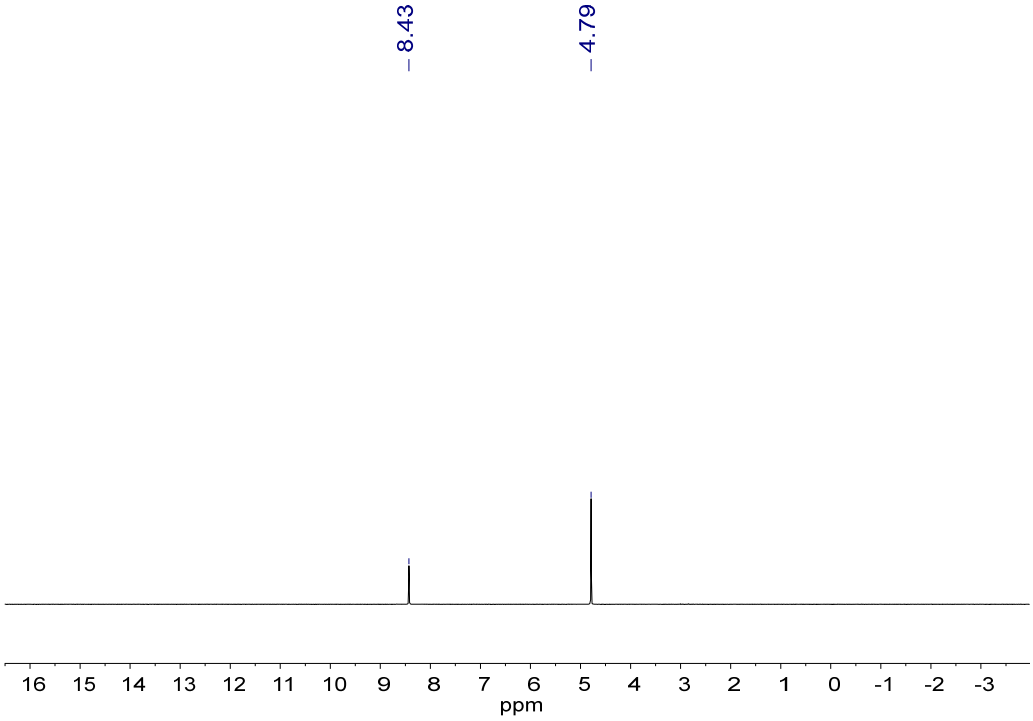
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	38.617	MM	2.1612	3.60265e5	2778.25269	48.0222
2	52.123	MM	4.8539	3.89939e5	1338.91602	51.9778

Figure S20. HPLC of racemic **2a**

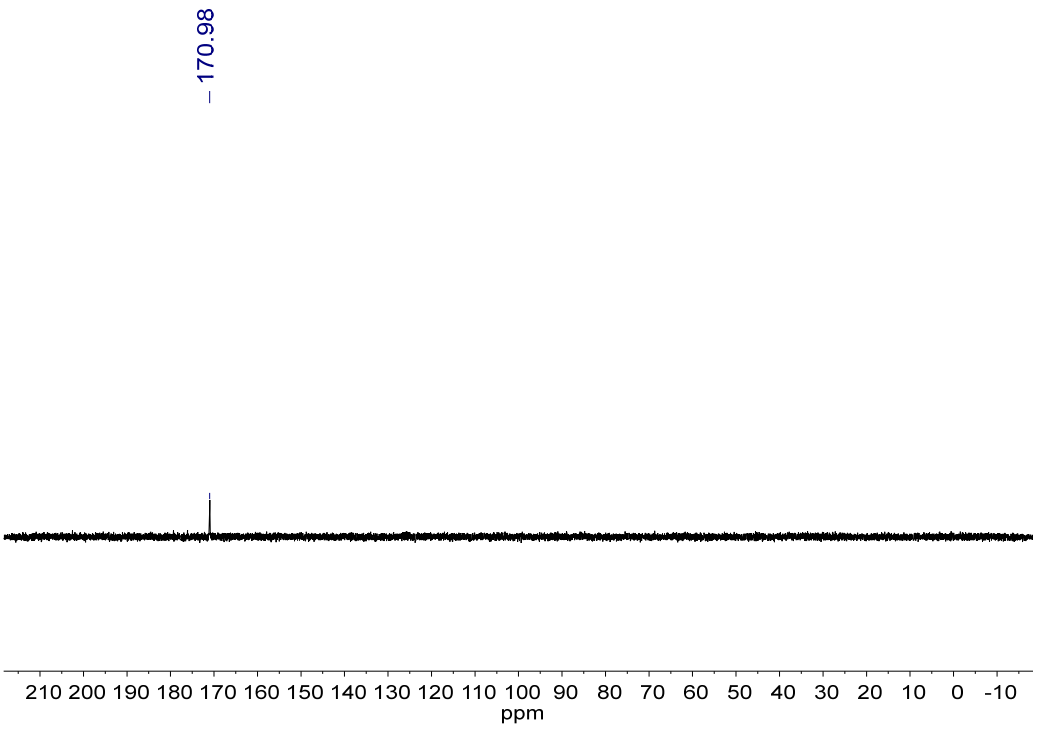
9.5 Formation of CO_2 radical anion.



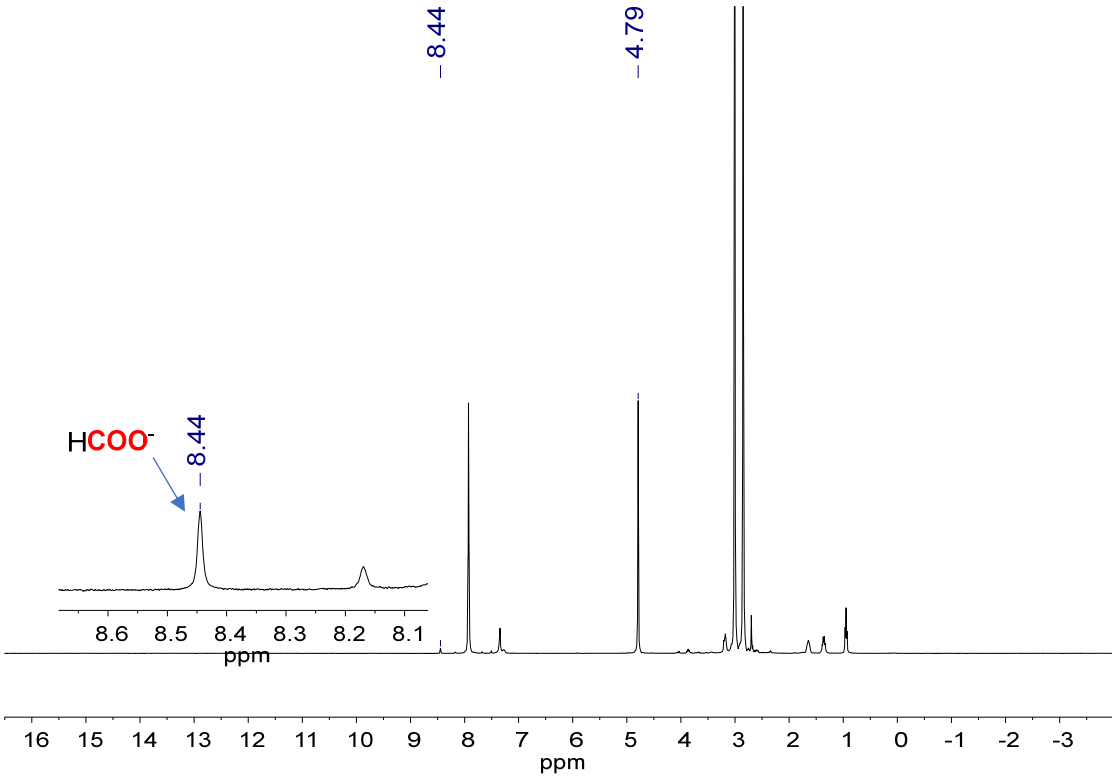
A 50 mL oven-dried electrochemical cell containing a stir bar was evacuated and back-filled under CO_2 flow for at least 3 times. **1a** (0.3 mmol), $TBABF_4$ (0.3 mmol) and DMF (5 mL) were added to the cell and the cell was installed with a Ni(foam) cathode and Mg anode. The reaction mixture was electrolyzed under a constant current of 10 mA for 4 h. After concentrating in vacuo carefully, 2 mL D_2O was added to make the mixture dissolved sufficiently, the $HCOO^-$ was detected by NMR. Therefore, the CO_2 radical anion might be generated through CO_2 reduction on cathode



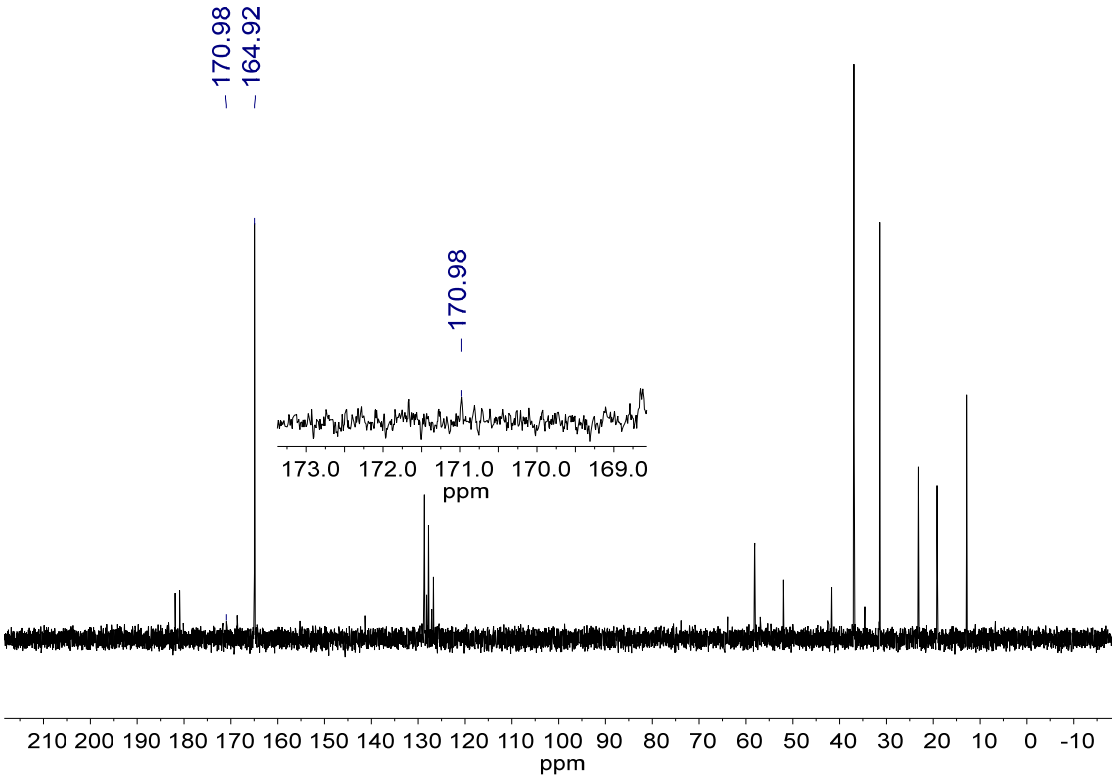
^{1}H NMR spectrum (400 MHz, Deuterium Oxide) of HCOOMg



^{13}C NMR spectrum (101 MHz, Deuterium Oxide) of HCOOMg

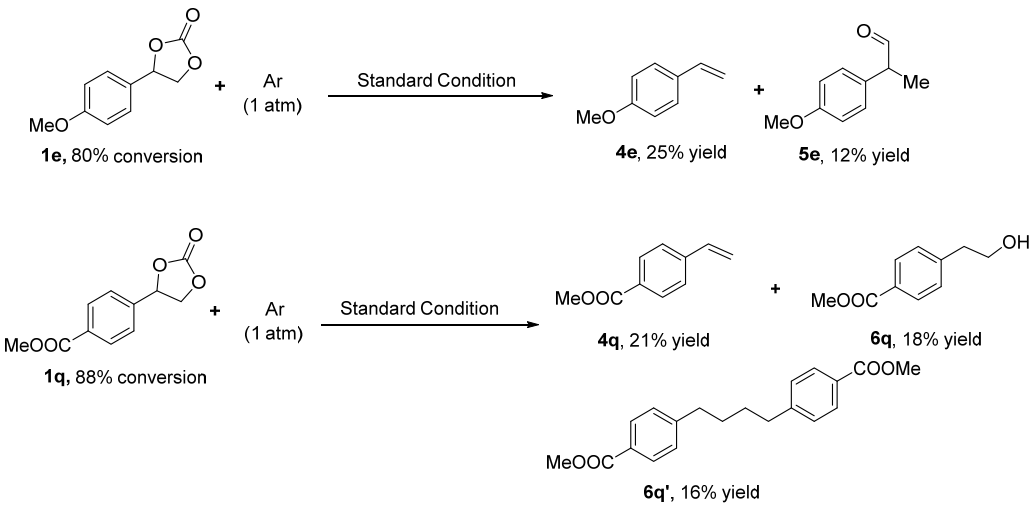


^{1}H NMR spectrum (400 MHz, Deuterium Oxide) of HCOO^{-}

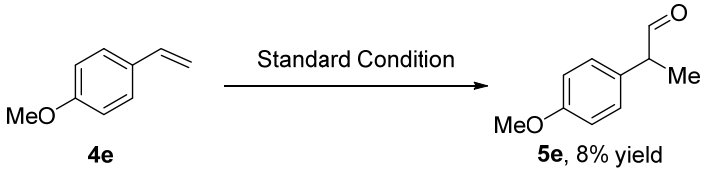


^{13}C NMR spectrum (101 MHz, Deuterium Oxide) of $HCOO^-$

9.6 Control experiments



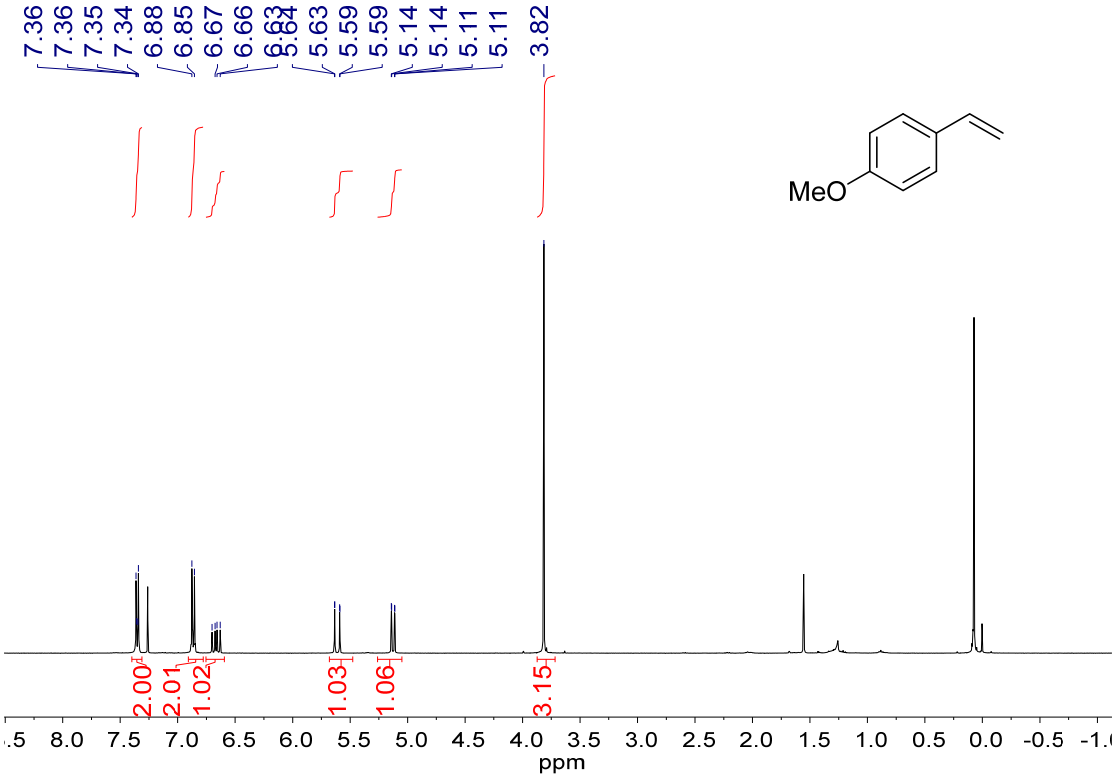
A 50 mL oven-dried electrochemical cell containing a stir bar was evacuated and back-filled under Ar flow for at least 3 times. Cyclic carbonate (0.3 mmol), $TBABF_4$ (0.3 mmol), and DMF (5 mL) were added to the cell and the cell was installed with a Pt cathode and Mg anode and closed under Ar atmosphere. Then, the reaction mixture was electrolyzed under a constant current of 10 mA for 4 h. The mixture was carefully quenched with HCl (1M, 10 mL) and extracted three times with ethyl acetate (3 × 20 mL). The combined extracts were washed with brine (2 × 50 mL) and dried over Na_2SO_4. The residue was methylated at 0 °C with $TMSCHN_2$ (2 M in hexane, 0.6 mL) in 2 mL of ether/methanol (1:1 v/v) solvent. Finally, the corresponding methylation product was purified by column chromatography (10:1-3:1 v/v petroleum ether/ethyl acetate). (For the reaction under Ar atmosphere, **4e**, white solid, 10 mg, 25% yield. **5e**, white solid, 6 mg, 12% yield. **4q**, white solid, 10 mg, 21% yield, **6q**, white solid, 10 mg, 18% yield, **6q'**, white solid, 18 mg, 16% yield.)



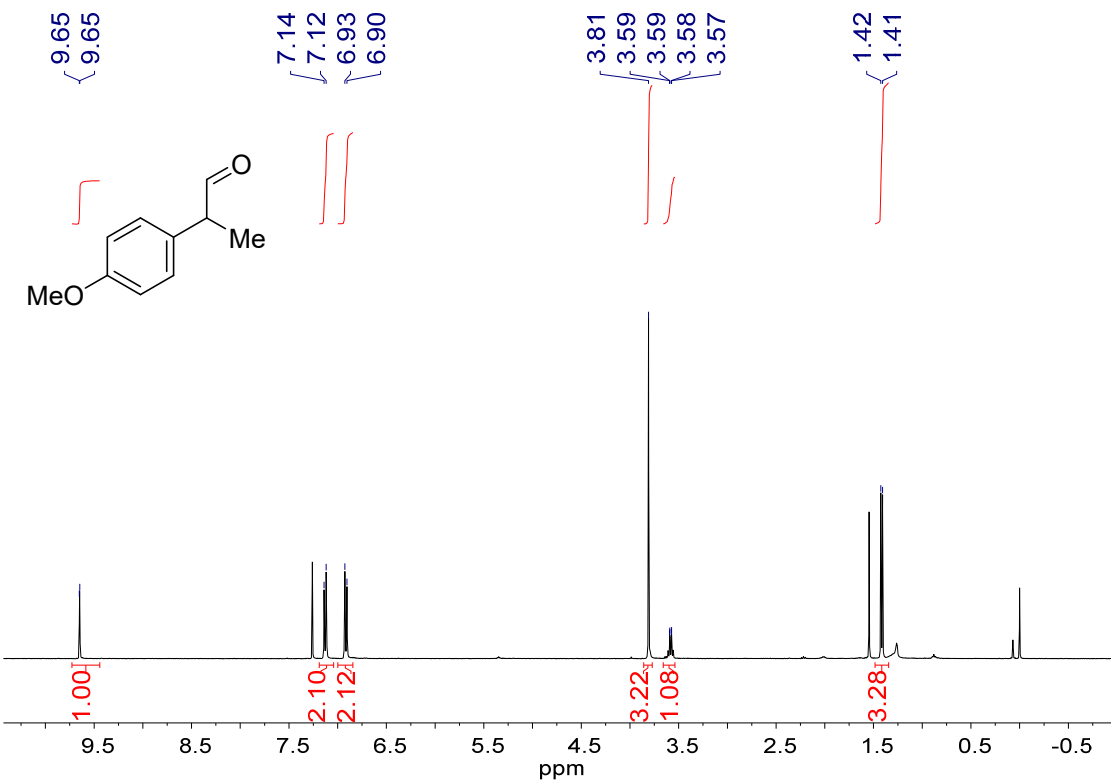
A 50 mL oven-dried electrochemical cell containing a stir bar was evacuated and back-filled under Ar flow for at least 3 times. **4e** (0.3 mmol), $TBABF_4$ (0.3 mmol), and DMF (5 mL) were added to the cell and the cell was installed with a Pt cathode and Mg anode and closed under Ar atmosphere. Then, the reaction mixture was electrolyzed under a constant current of 10 mA for 4 h. The mixture was quenched with water (10 mL) and extracted three times with ethyl acetate (3 × 20 mL). The combined extracts were washed with brine (2 × 50 mL) and dried over Na_2SO_4. The solvents were removed under reduced pressure and the crude residue

was purified by flash column chromatography (10:1 v/v petroleum ether/ethyl acetate) to get desired product **5e**.

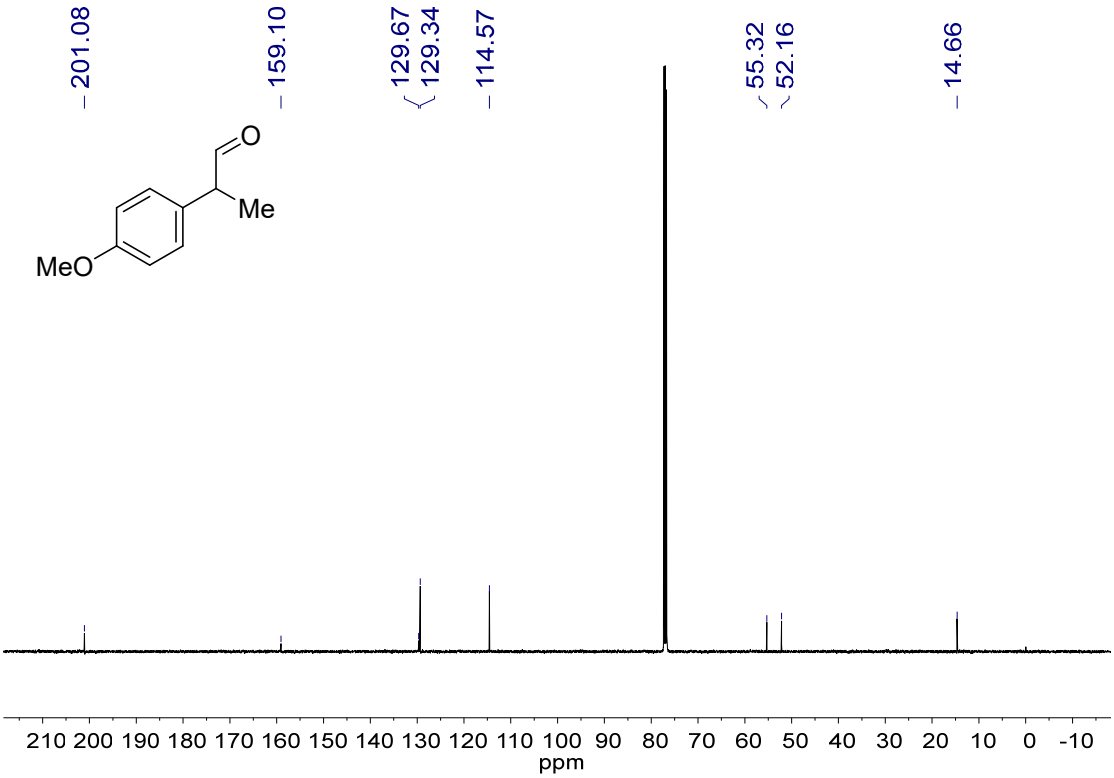
2-(4-Methoxyphenyl) propanal (5e). White solid, 3.9 mg, 8% yield. **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 9.65 (d, J = 1.4 Hz, 1H), 7.13 (d, J = 8.6 Hz, 1H), 6.92 (d, J = 8.6 Hz, 1H), 3.81 (s, 2H), 3.58 (dd, J = 7.1, 1.4 Hz, 0H), 1.42 (d, J = 7.0 Hz, 2H). **^{13}C NMR** (126 MHz, Chloroform-*d*) δ 201.0, 159.1, 129.6, 129.3, 114.5, 55.3, 52.1, 14.6. **HRMS** (ESI-TOF) *m*/*z*: $[M+Na]^+$ Calcd for $C_{10}H_{12}NaO_2$ 187.0735; Found 187.0734.



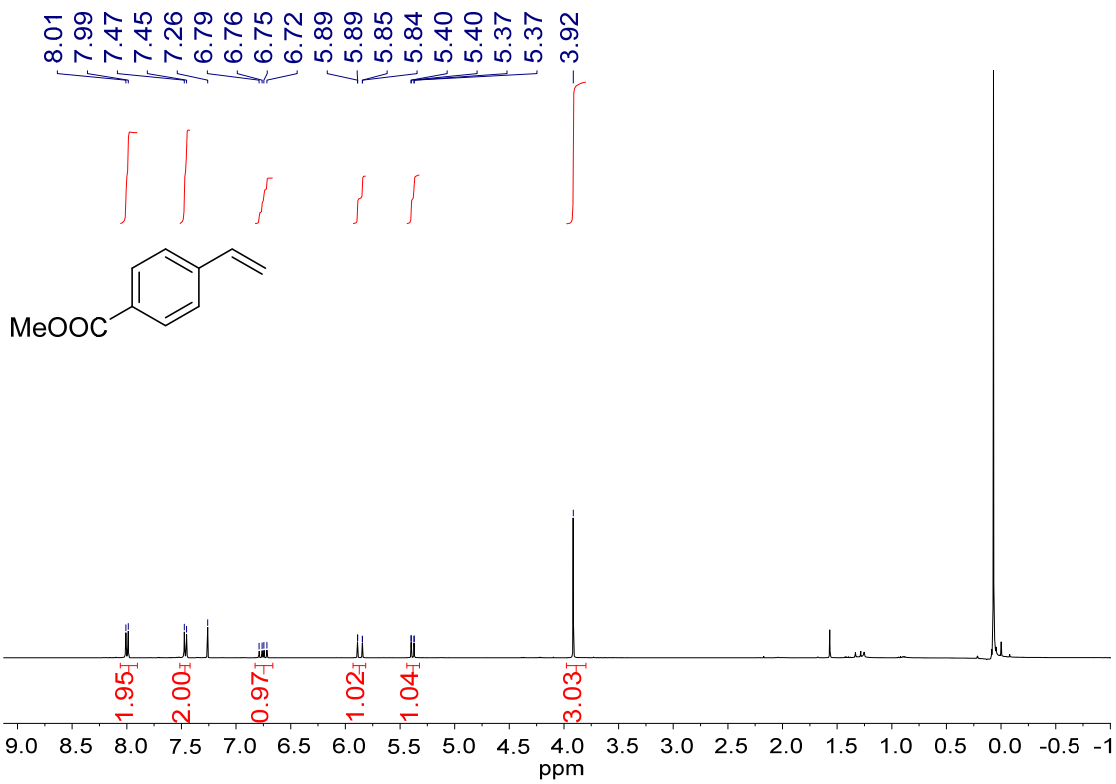
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 4e



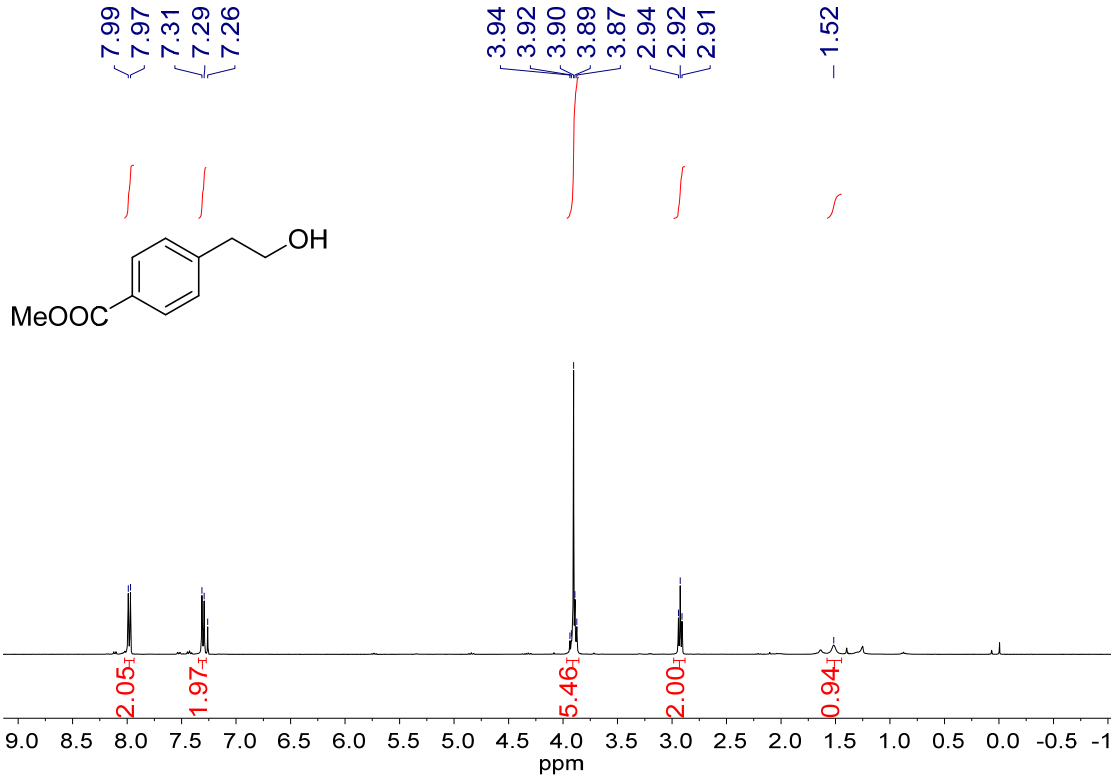
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 5e



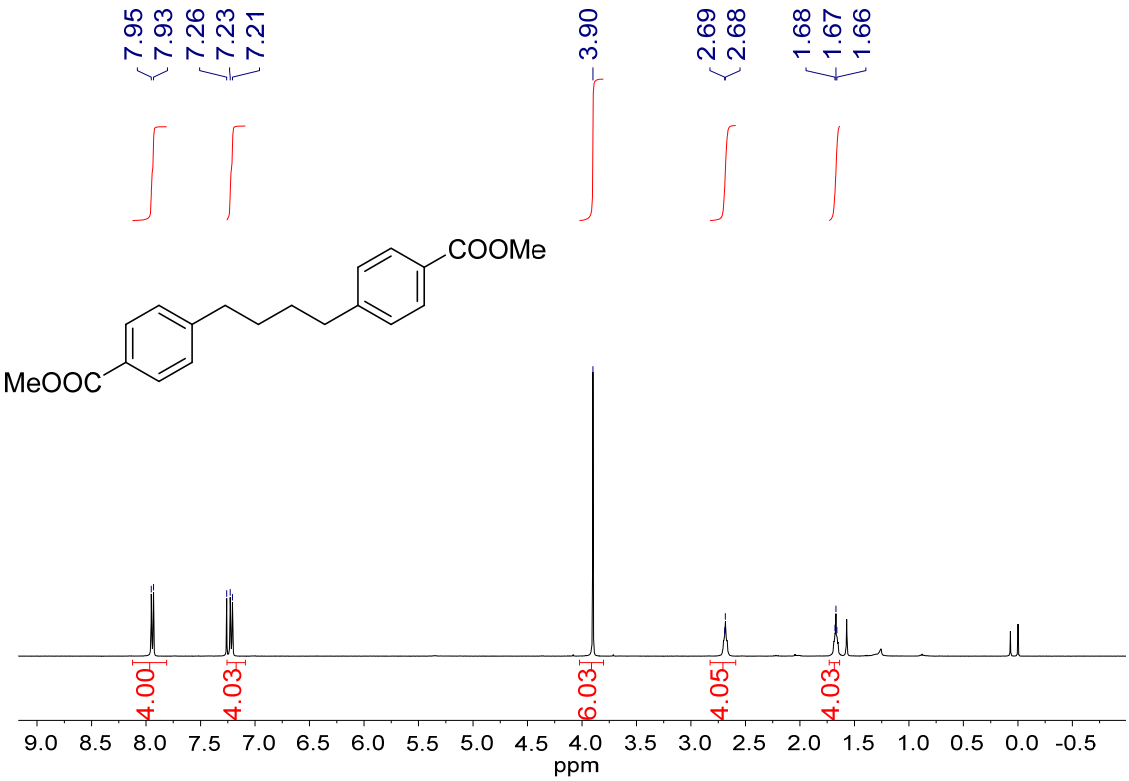
^{13}C{^{1}H} NMR spectrum (126 MHz, Chloroform-*d*) of 5e



^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 4q

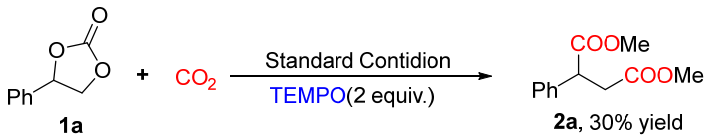


^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 6q

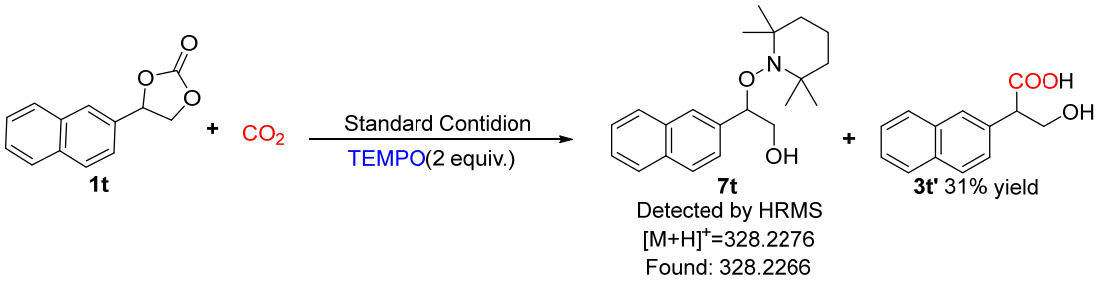


^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 6q'

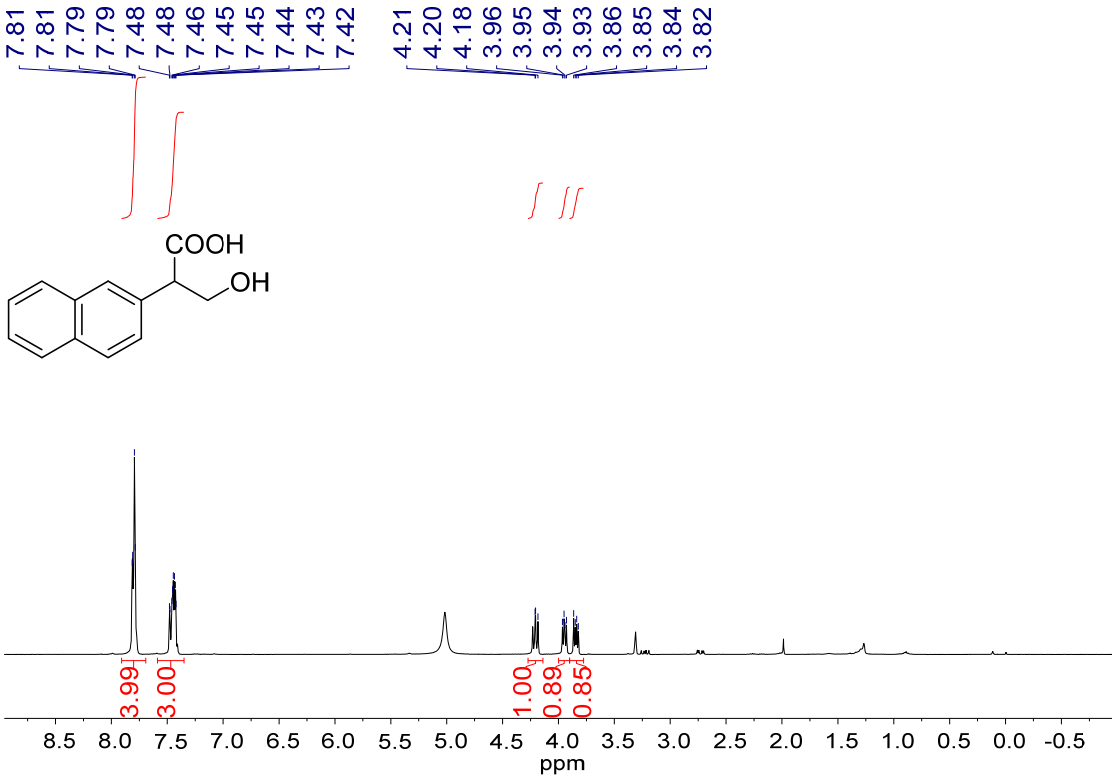
9.7 Radical trap experiment



A 50 mL oven-dried electrochemical cell containing a stir bar was evacuated and back-filled under CO_2 flow for at least 3 times. **1a** (0.3 mmol), $TBABF_4$ (0.3 mmol), TEMPO (0.6 mmol) and DMF (5 mL) were added to the cell and the cell was installed with a Ni cathode and Mg anode and the cell was closed under CO_2 atmosphere. Then, the reaction mixture was electrolyzed under a constant current of 10 mA for 4 h. Then the mixture was carefully quenched with HCl (1M, 10 mL) and extracted three times with ethyl acetate (3 × 20 mL). The combined extracts were washed with brine (2 × 50 mL) and dried over Na_2SO_4. The residue was methylated at 0 °C with $TMSCHN_2$ (2 M in hexane, 0.6 mL) in 2 mL of ether/methanol (1:1 v/v) solvent. Finally, the corresponding methylation product was purified by column chromatography (10:1-3:1 v/v petroleum ether/ethyl acetate).



A 50 mL oven-dried electrochemical cell containing a stir bar was evacuated and back-filled under CO_2 flow for at least 3 times. **1t** (0.3 mmol), $TBABF_4$ (0.3 mmol), TEMPO (0.6 mmol) and DMF (5 mL) were added to the cell and the cell was installed with a Ni cathode and Mg anode and the cell was closed under CO_2 atmosphere. Then, the reaction mixture was electrolyzed under a constant current of 10 mA for 4 h. Then the mixture was carefully quenched with HCl (1M, 10 mL) and extracted three times with ethyl acetate (3 × 20 mL). The combined extracts were washed with brine (2 × 50 mL) and dried over Na_2SO_4. The solvents were removed under reduced pressure and the crude residue was purified by flash column chromatography (petroleum ether/ethyl acetate/AcOH 10/1/0-3/1/0-3/2/0.005) to achieve the product **3t'**, white solid, 20 mg, 31% yield. The corresponding TEMPO-adduct **7t** was trace and detected by HRMS.



^{1}H NMR spectrum (400 MHz, Methanol-d_4) of 3t'

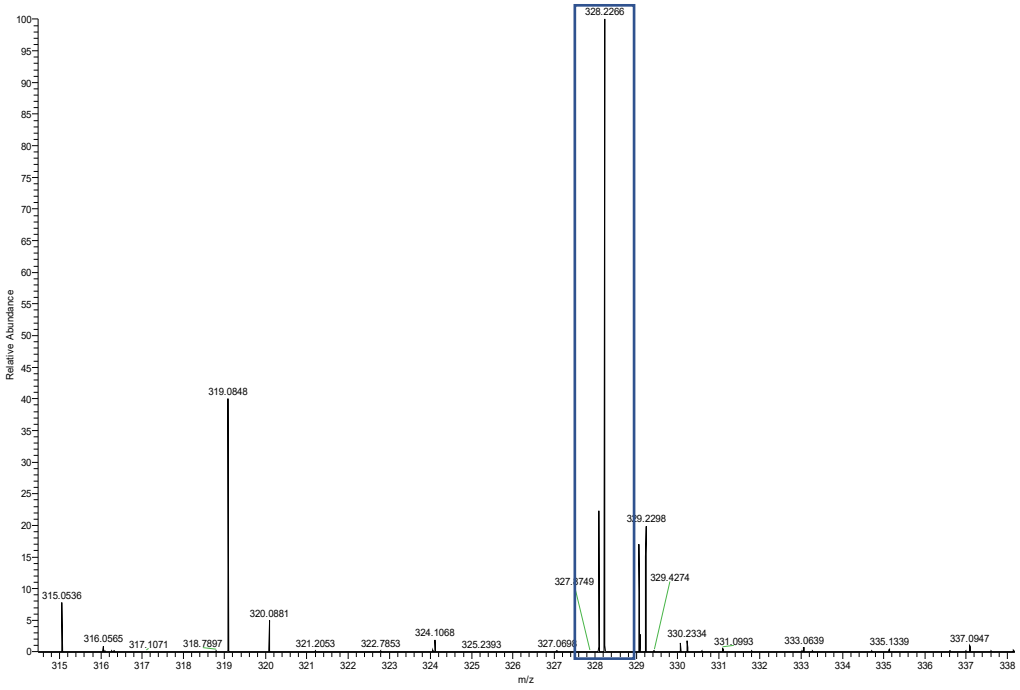
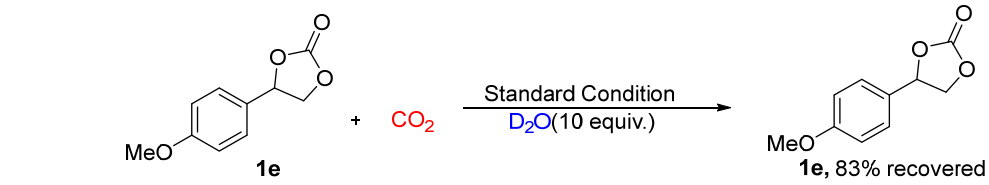
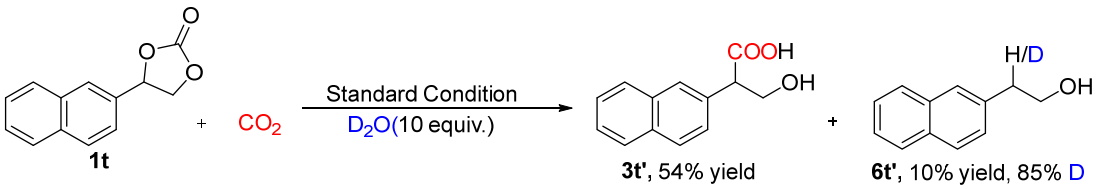


Figure S21. HRMS of 7t

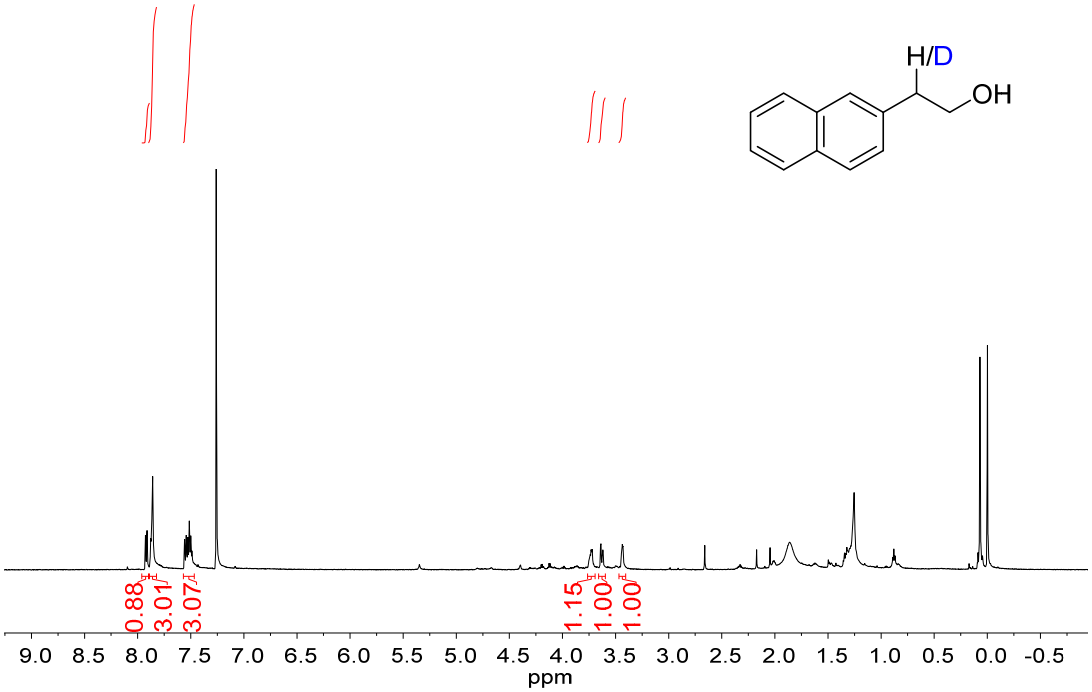
9.8 Deuteration experiment



A 50 mL oven-dried electrochemical cell containing a stir bar was evacuated and back-filled under CO_2 for at least 3 times. **1e** (0.3 mmol), $TBABF_4$ (0.3 mmol), D_2O (3 mmol) and DMF (5 mL) were added to the cell and the cell was installed with a Pt cathode and Mg anode and the cell was closed under CO_2 atmosphere. Then, the reaction mixture was electrolyzed under a constant current of 10 mA for 4 h. Only 17% **1e** was converted.

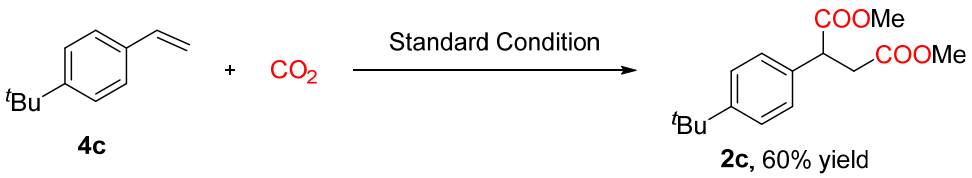


A 50 mL oven-dried electrochemical cell containing a stir bar was evacuated and back-filled under CO_2 for at least 3 times. **1t** (0.3 mmol), $TBABF_4$ (0.3 mmol), D_2O (3 mmol) and DMF (5 mL) were added to the cell and the cell was installed with a Pt cathode and Mg anode and the cell was closed under CO_2 atmosphere. Then, the reaction mixture was electrolyzed under a constant current of 10 mA for 4 h. Then the mixture was carefully quenched with HCl (1M, 10 mL) and extracted three times with ethyl acetate (3 × 20 mL). The combined extracts were washed with brine (2 × 50 mL) and dried over Na_2SO_4. The solvents were removed under reduced pressure and the crude residue was purified by flash column chromatography (petroleum ether/ethyl acetate/AcOH 2/1/0-3/2/0.005) to give product **3t'** as white solid (40 mg, 60% yield) and **6t'** as white solid (5mg, 10% yield, 85% D).

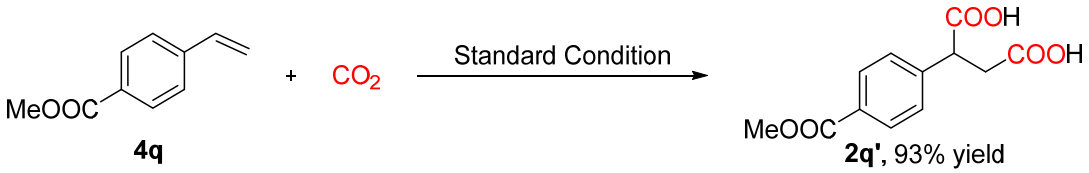


^{1}H NMR spectrum (600 MHz, Chloroform-*d*) of 6t'

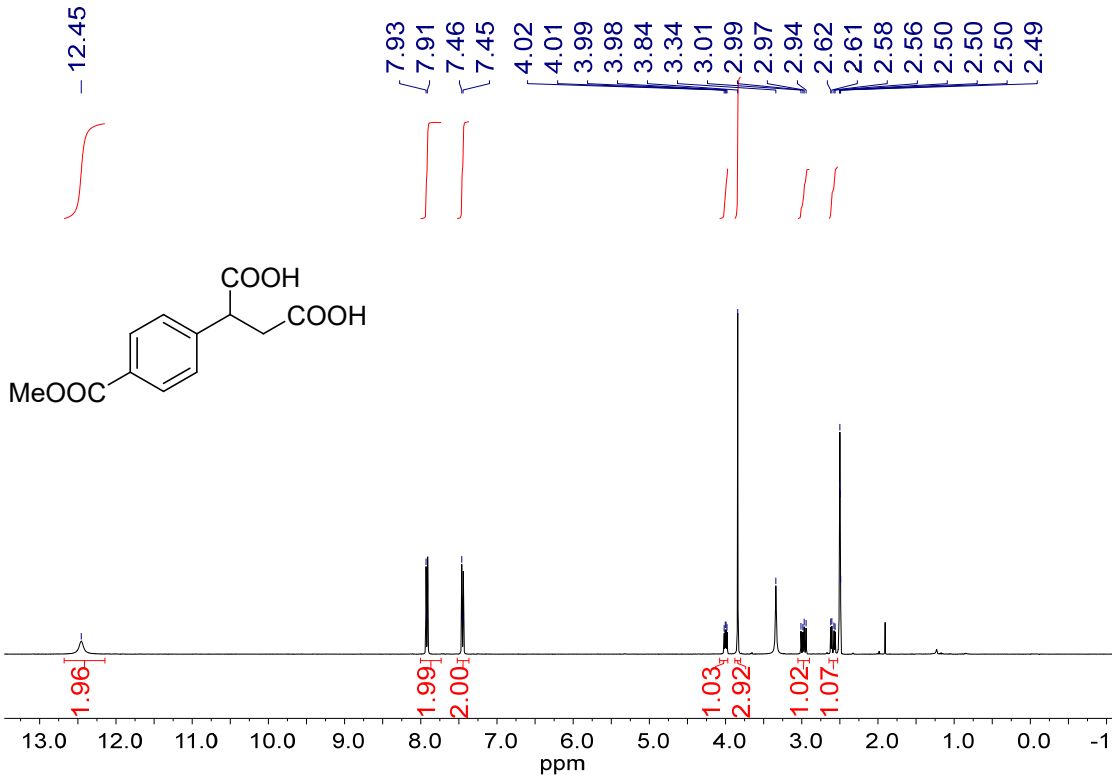
9.9 Control electrocarboxylation reaction of styrene substates with CO_2.



A 50 mL oven-dried electrochemical cell containing a stir bar was evacuated and back-filled under CO_2 flow for at least 3 times. **4c** (0.3 mmol), $TBABF_4$ (0.3 mmol) and DMF (5 mL) were added to the cell and the cell was installed with a Pt cathode and Mg anode, and the cell was closed under CO_2 atmosphere. Then, the reaction mixture was electrolyzed under a constant current of 10 mA for 4 h. The reaction mixture was carefully quenched with HCl (1M, 10 mL) and extracted three times with ethyl acetate (3 × 20 mL). The combined organic phase was dried by anhydrous $MgSO_4$ and evaporated in vacuum. The residue was methylated at 0°C with $TMSCHN_2$ (2M, 0.6 mL) in 2 mL of ether/methanol (1:1 v/v) solvent. Finally, the corresponding methylation product **2c** (52 mg, 60% yield) was purified by column chromatography (10:1 v/v petroleum ether/ethyl acetate).

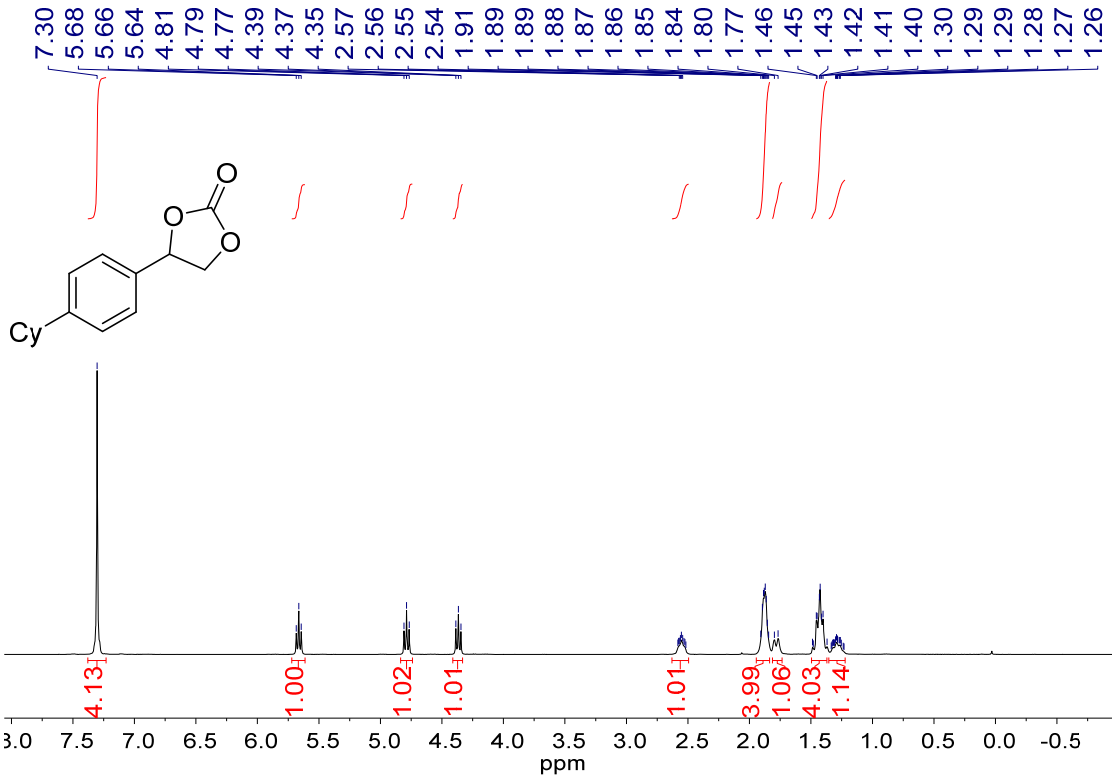


A 50 mL oven-dried electrochemical cell containing a stir bar was evacuated and back-filled under CO_2 flow for at least 3 times. **4q** (0.3 mmol), $TBABF_4$ (0.3 mmol) and DMF (5 mL) were added to the cell and the cell was installed with a Pt cathode and Mg anode, and the cell was closed under CO_2 atmosphere. Then, the reaction mixture was electrolyzed under a constant current of 10 mA for 4 h. The reaction mixture was carefully quenched with HCl (1M, 10 mL) and extracted three times with ethyl acetate (3 × 20 mL). The combined organic phase was dried by anhydrous $NaSO_4$. The solvents were removed under reduced pressure and the crude residue was purified by flash column chromatography (petroleum ether/ethyl acetate/AcOH 1/1/0.005) to give desired product **2q'**, white solid, 70 mg, 93% yield.

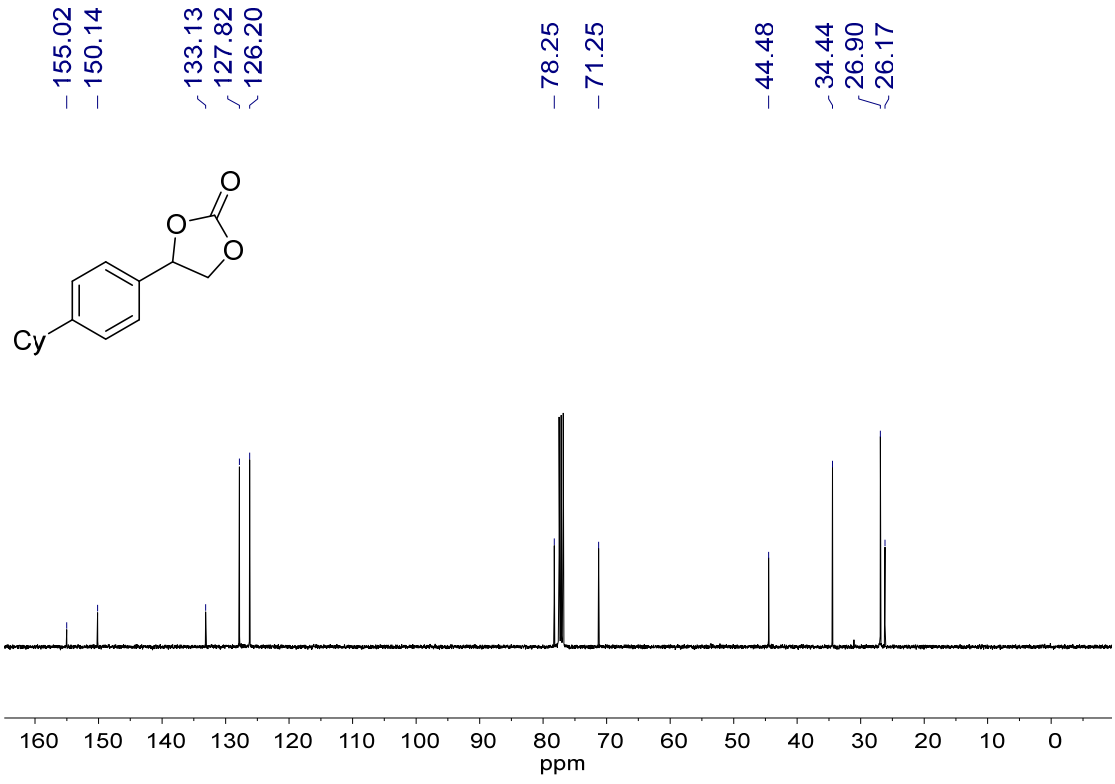


^{1}H NMR spectrum (400 MHz, DMSO-d_6) of 2q'

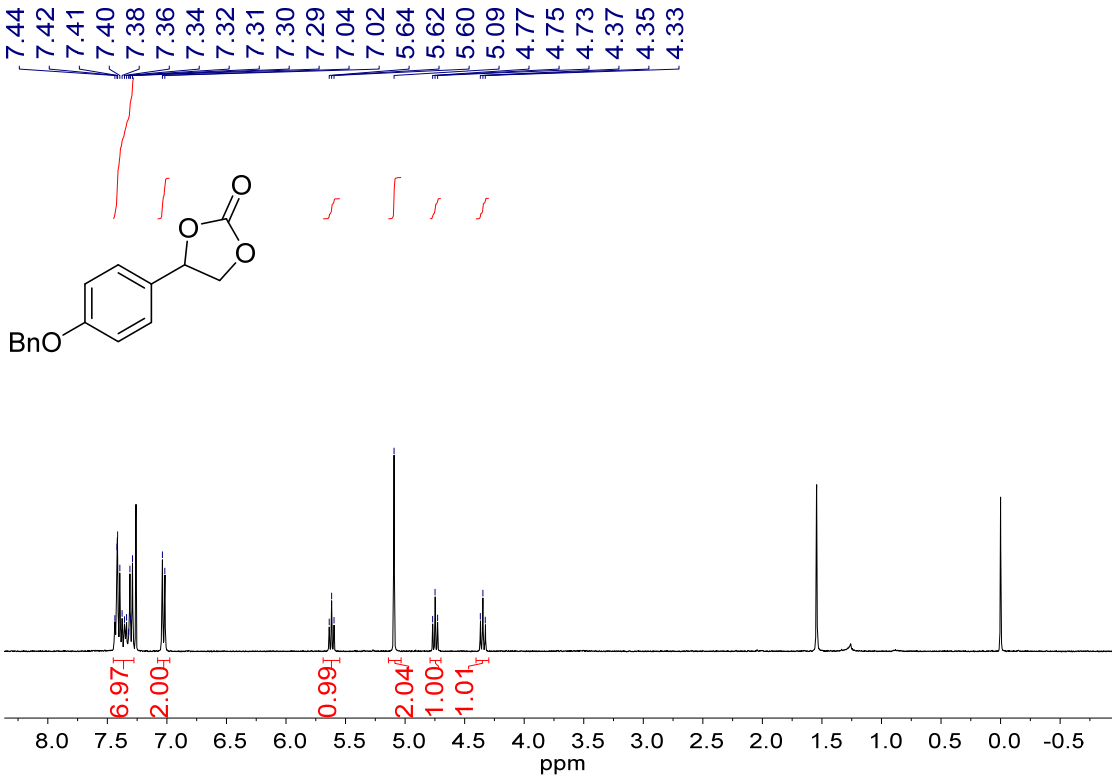
10. NMR spectra of substrates and all products



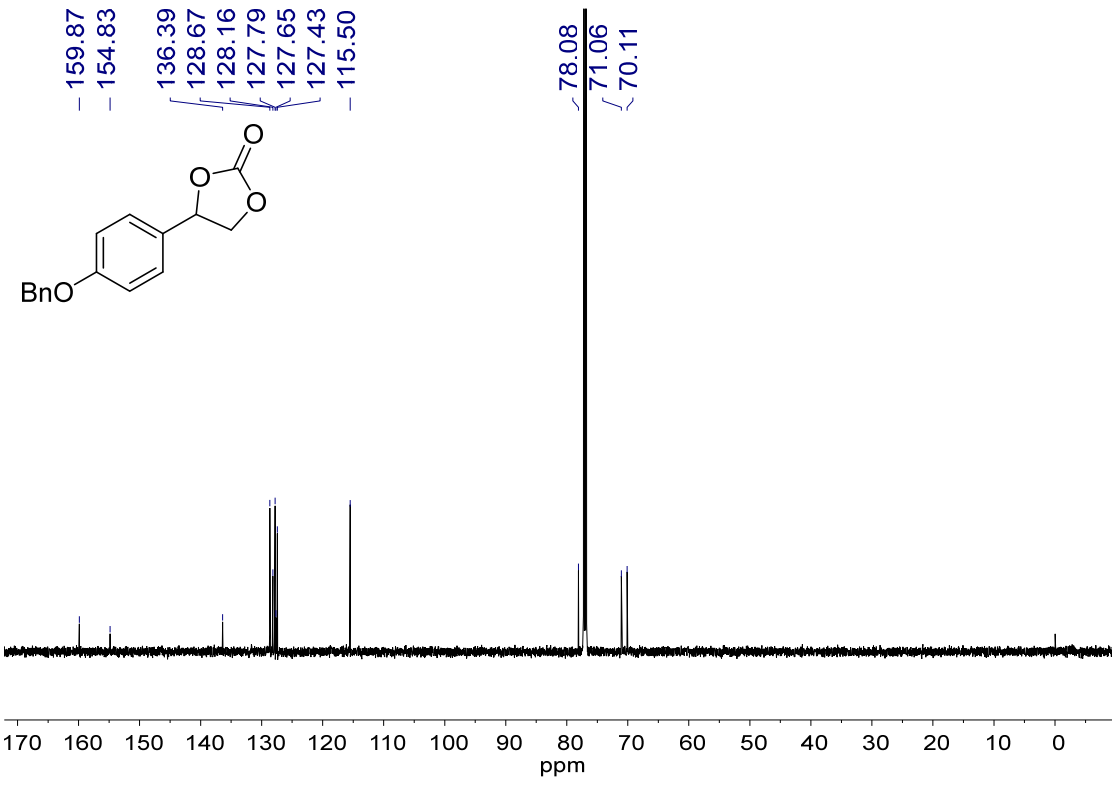
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 1d



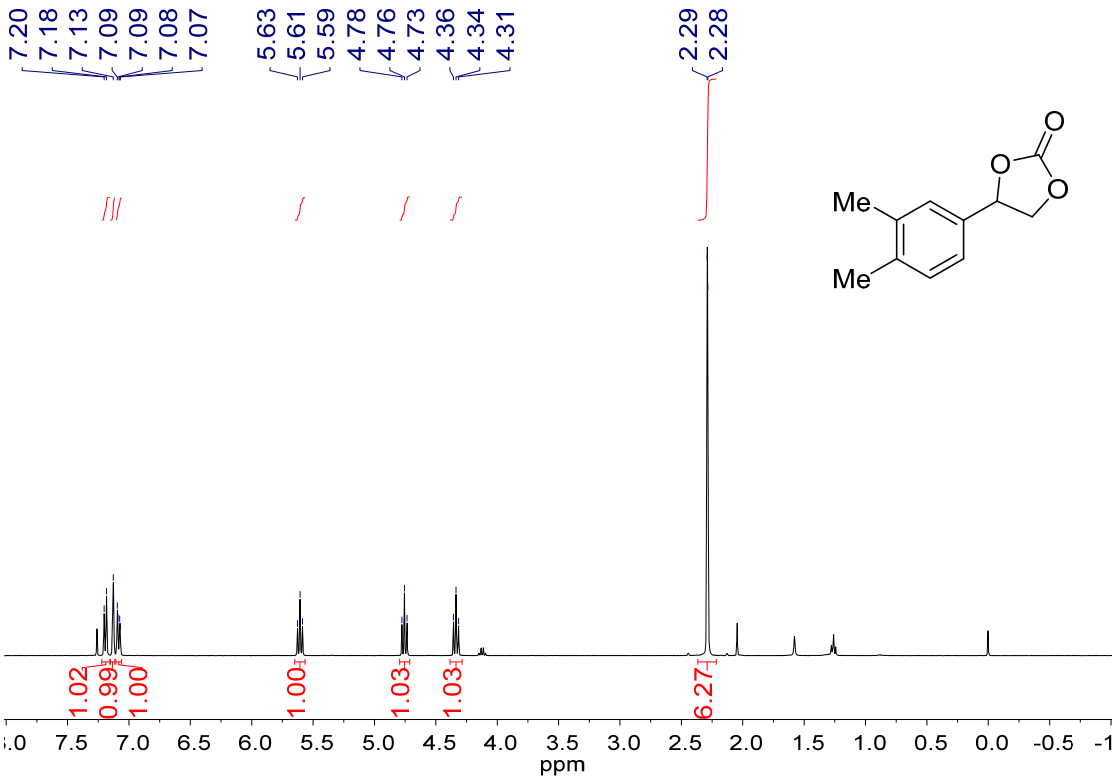
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 1d



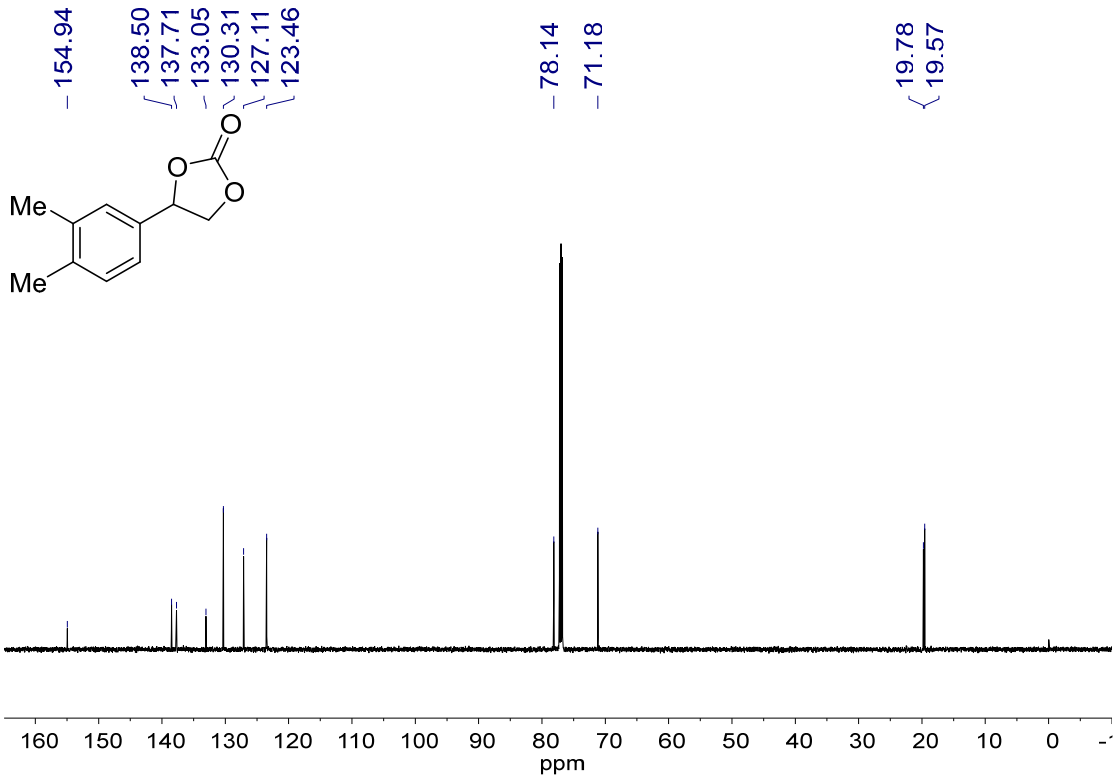
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 1f



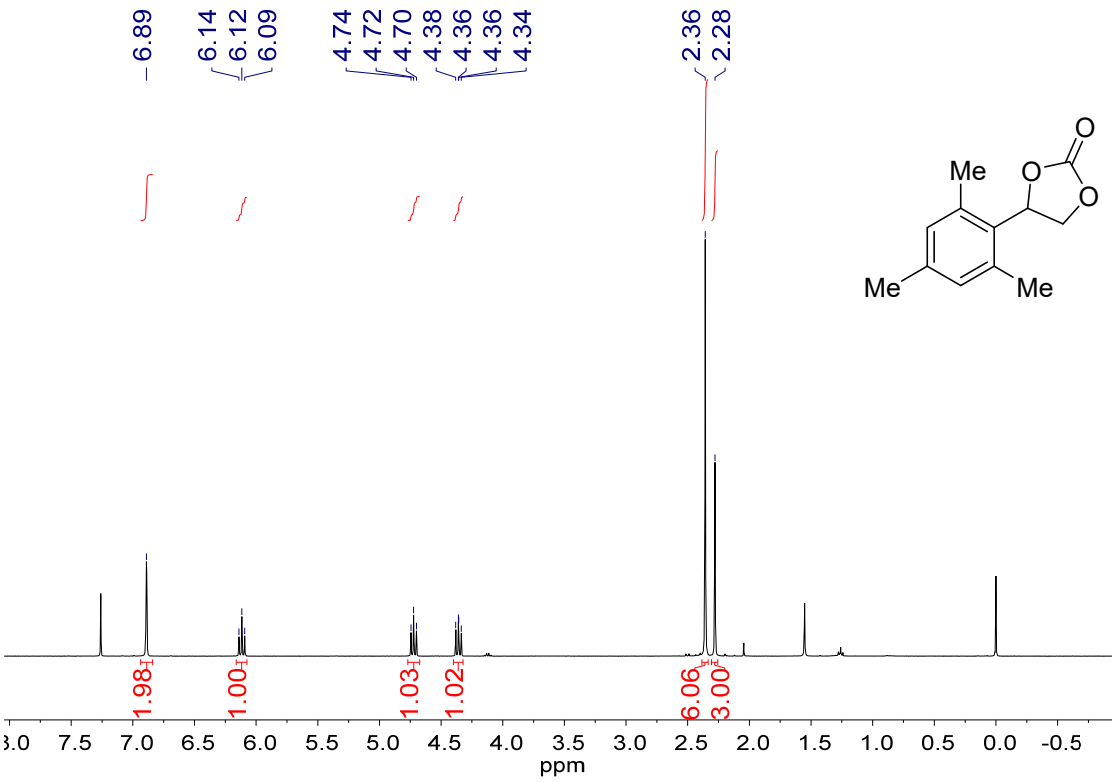
^{13}C{^{1}H} NMR spectrum (151 MHz, Chloroform-*d*) of 1f



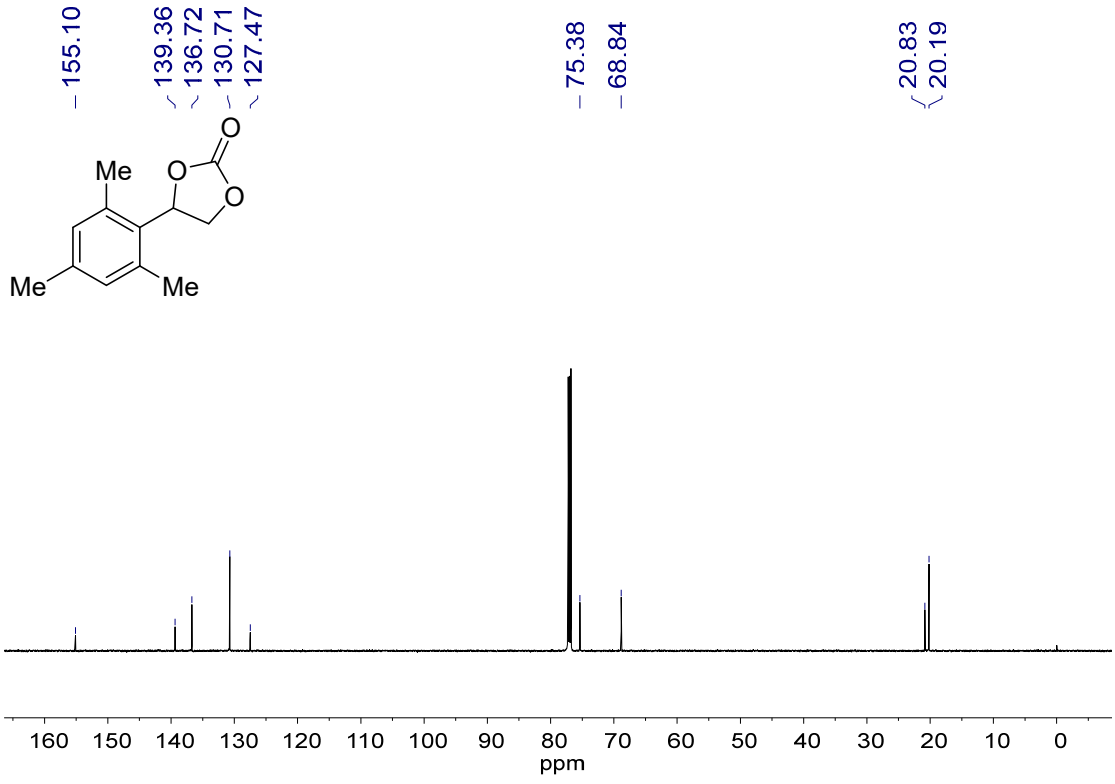
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 1h



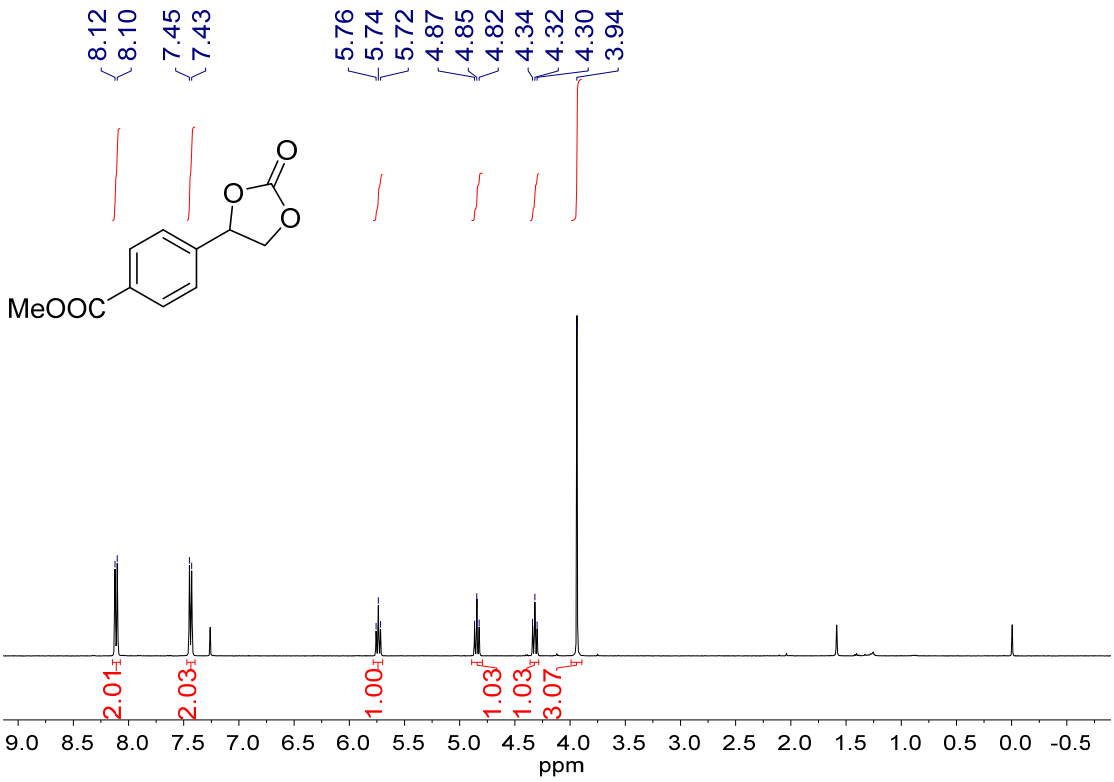
^{13}C{^{1}H} NMR spectrum (151 MHz, Chloroform-*d*) of 1h



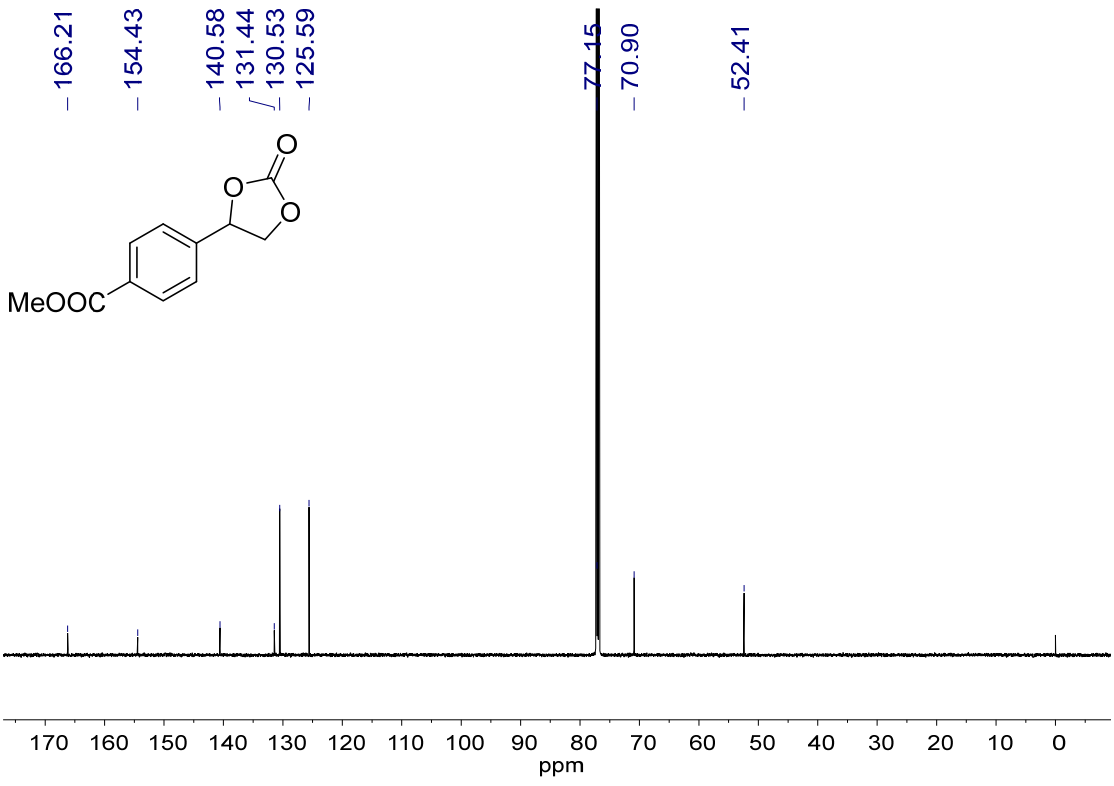
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 1j



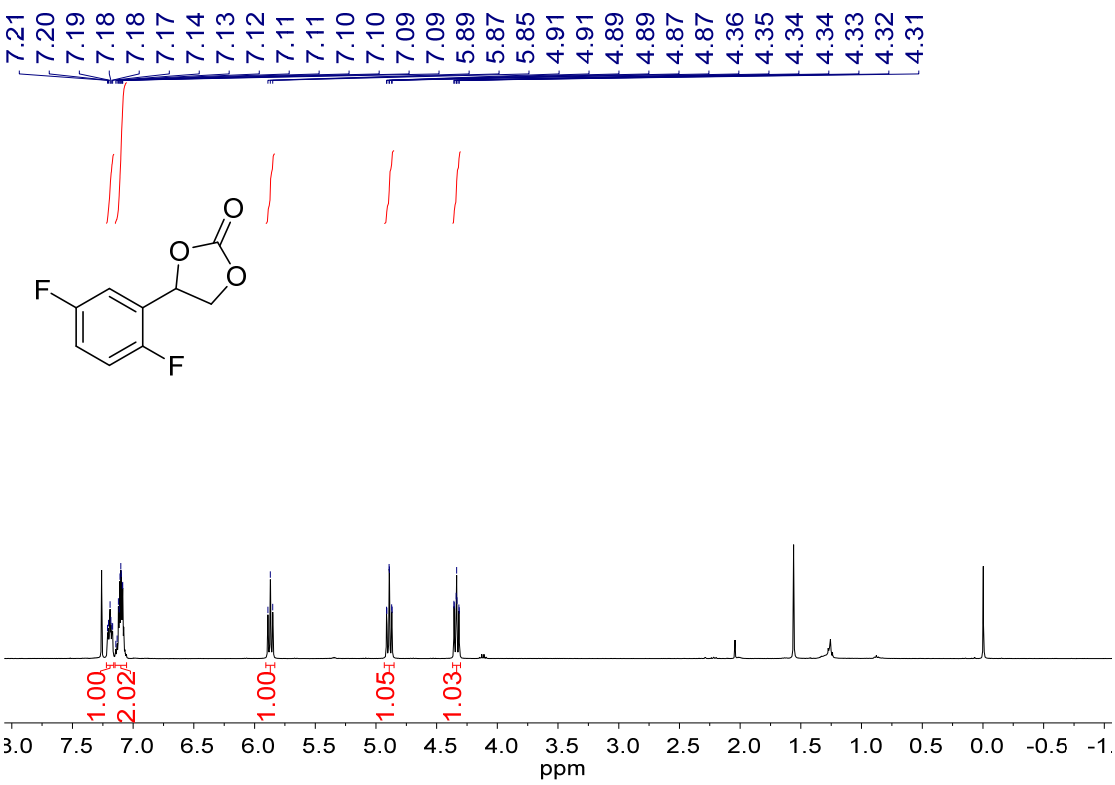
^{13}C{^{1}H} NMR spectrum (151 MHz, Chloroform-*d*) of 1j



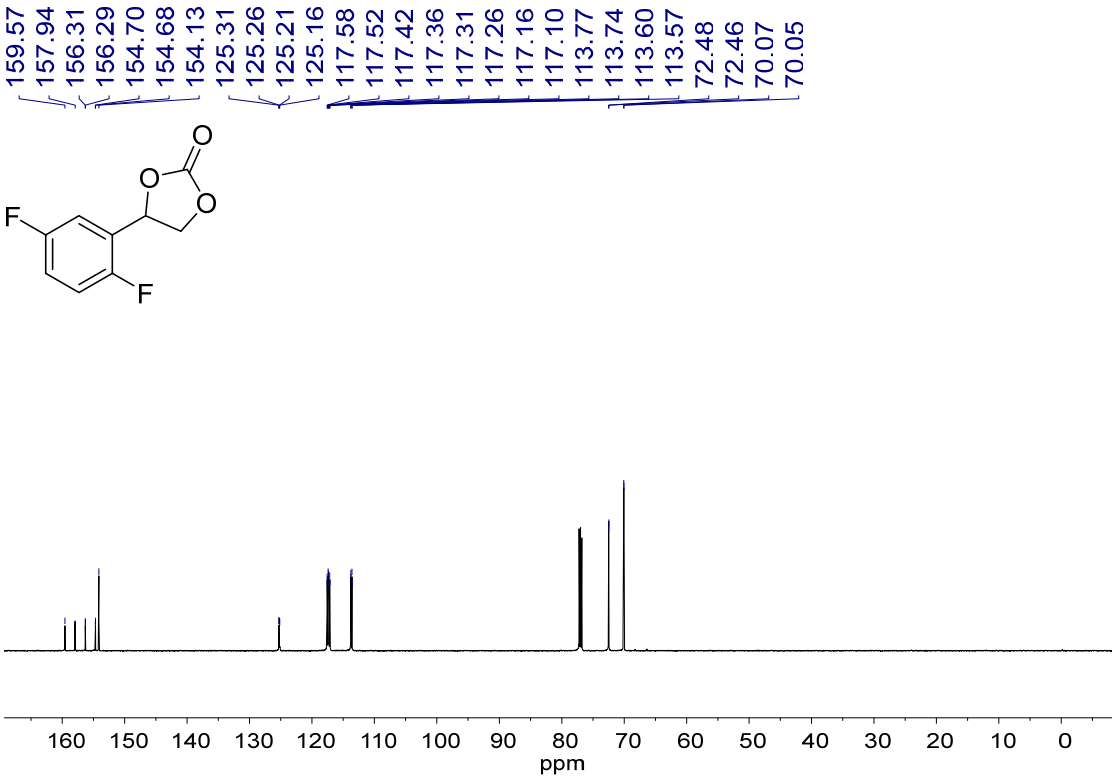
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 1q



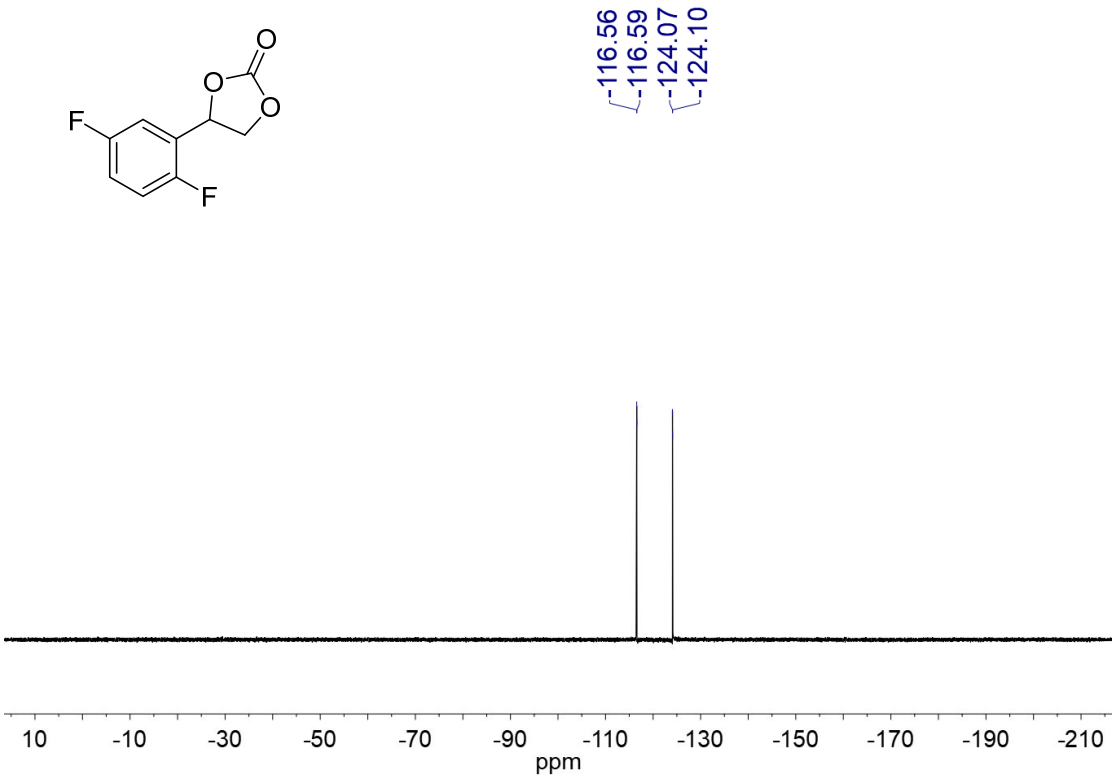
^{13}C{^{1}H} NMR spectrum (151 MHz, Chloroform-*d*) of 1q



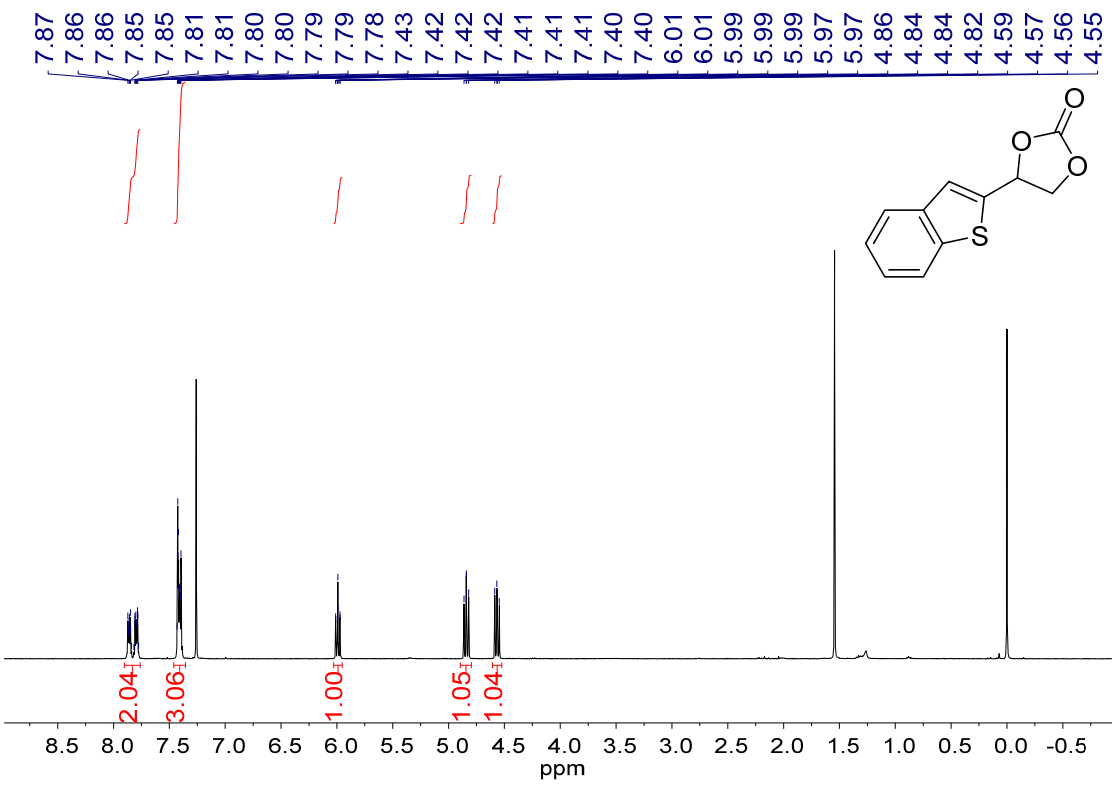
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 1s



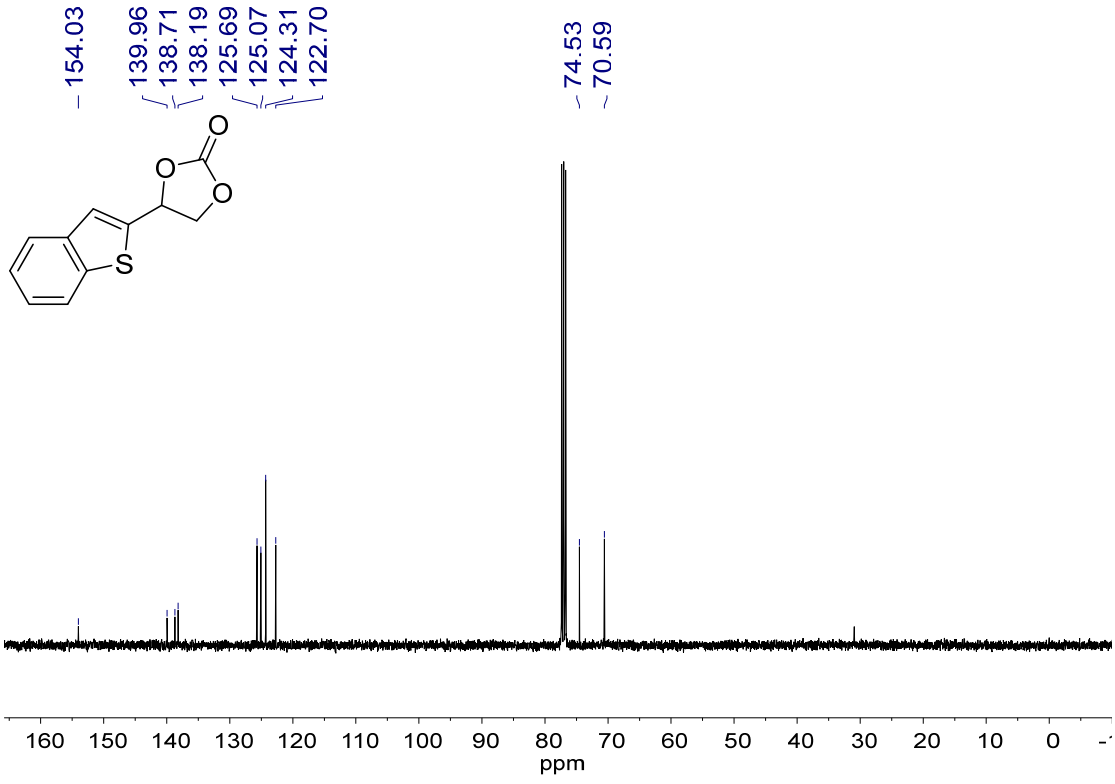
^{13}C{^{1}H} NMR spectrum (151 MHz, Chloroform-*d*) of 1s



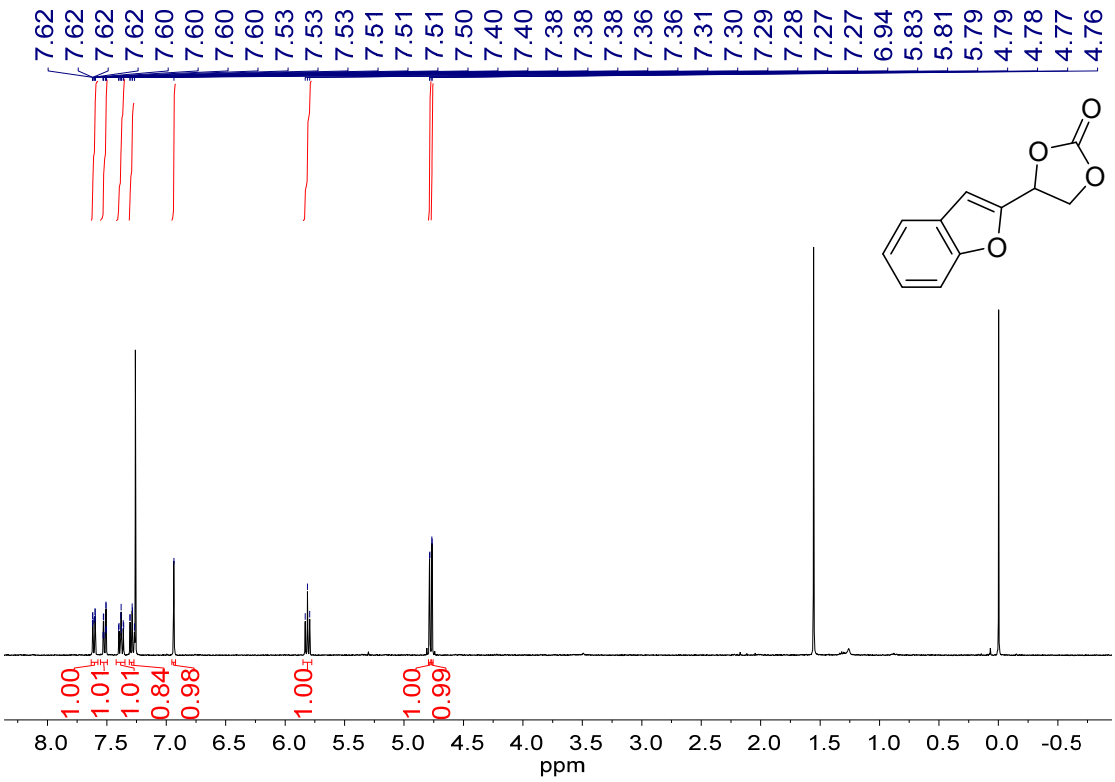
^{19}F NMR spectrum (376 MHz, Chloroform-*d*) of 1s



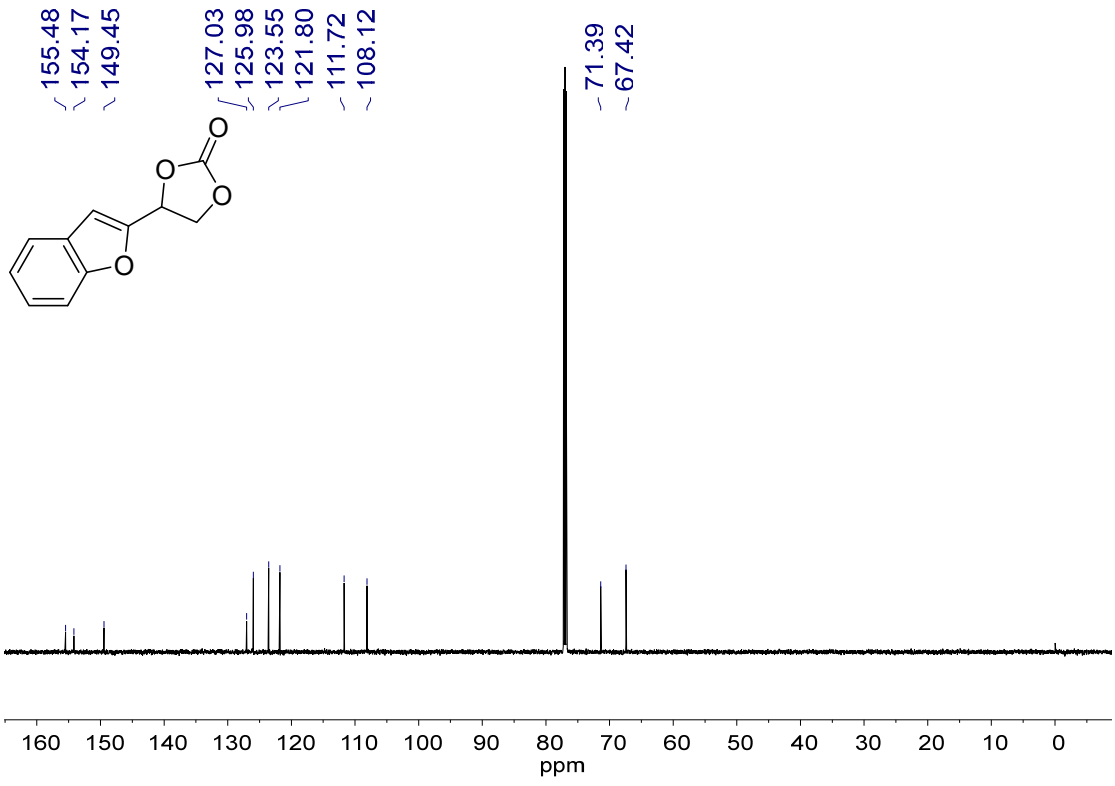
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 1v



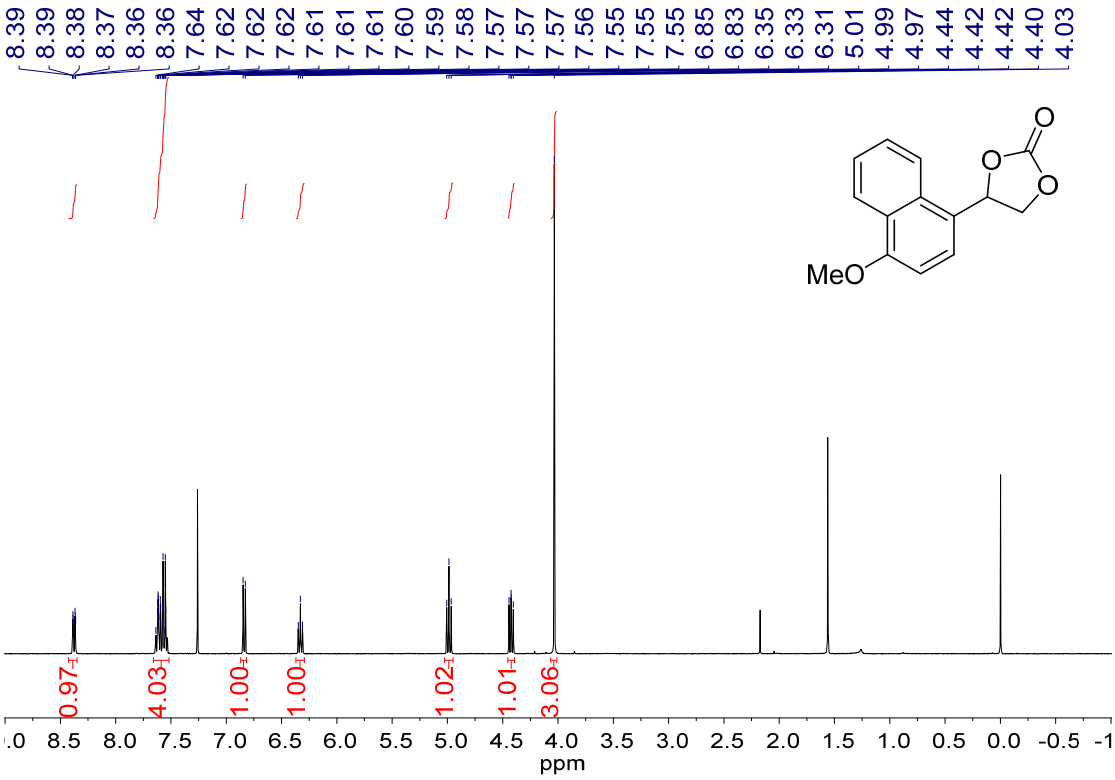
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 1v



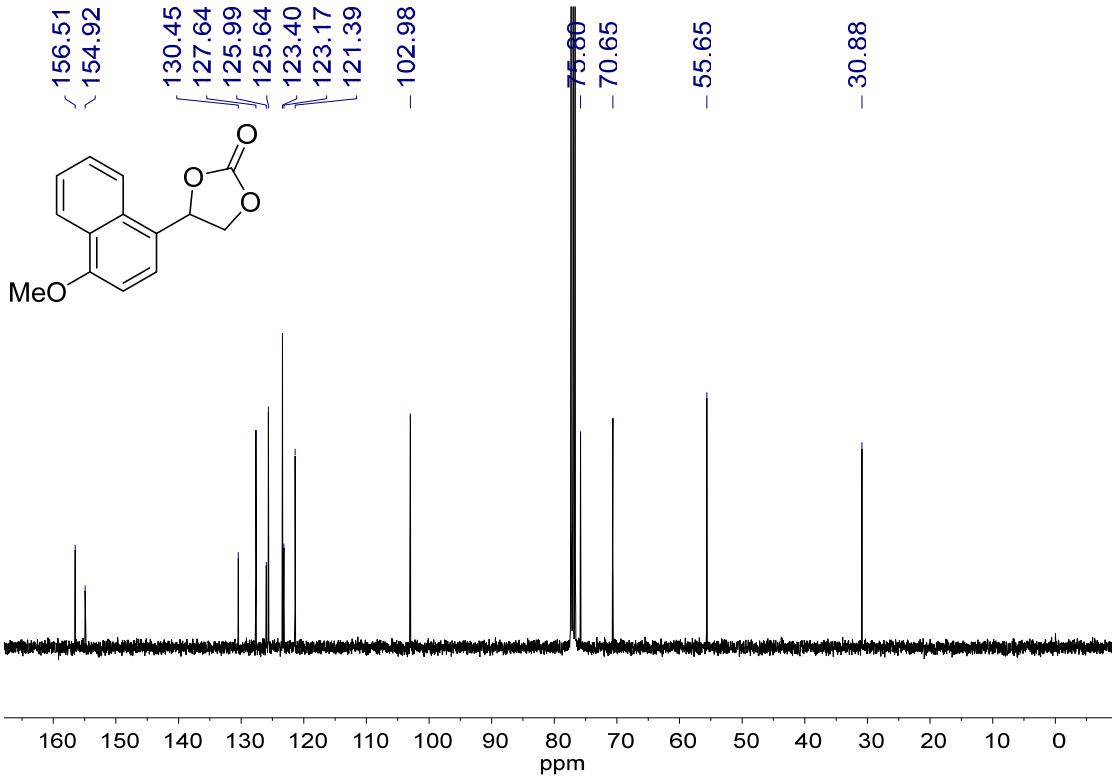
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 1w



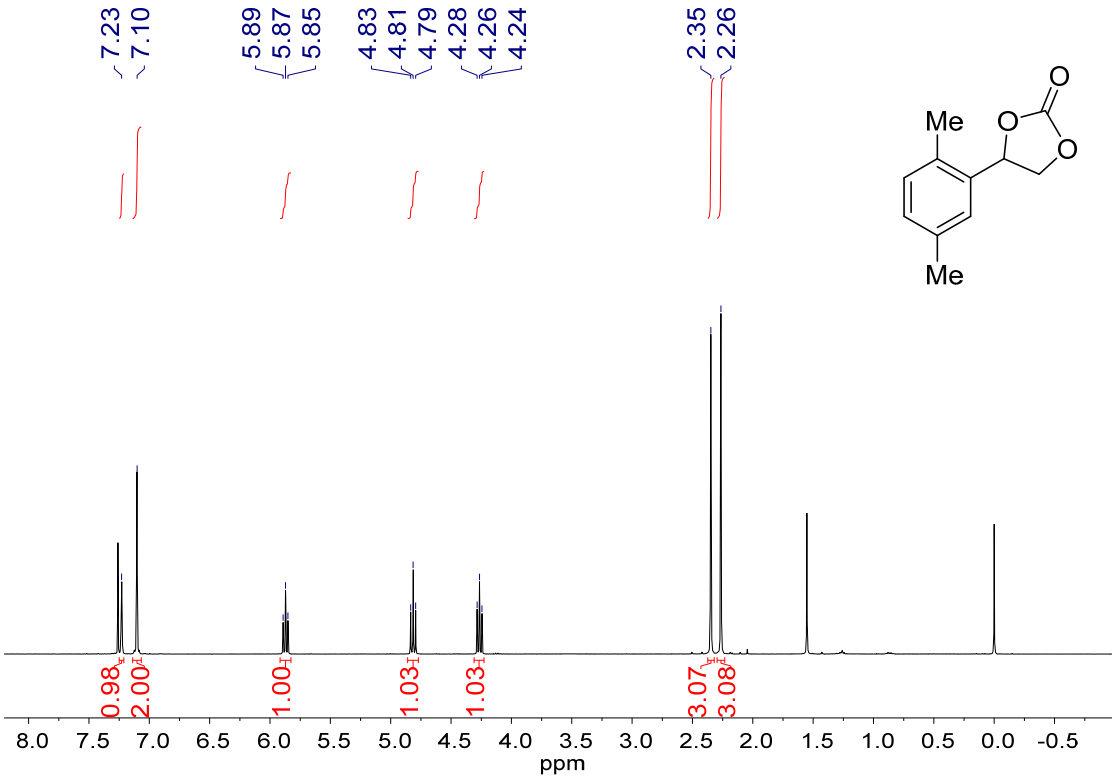
^{13}C{^{1}H} NMR spectrum (151 MHz, Chloroform-*d*) of 1w



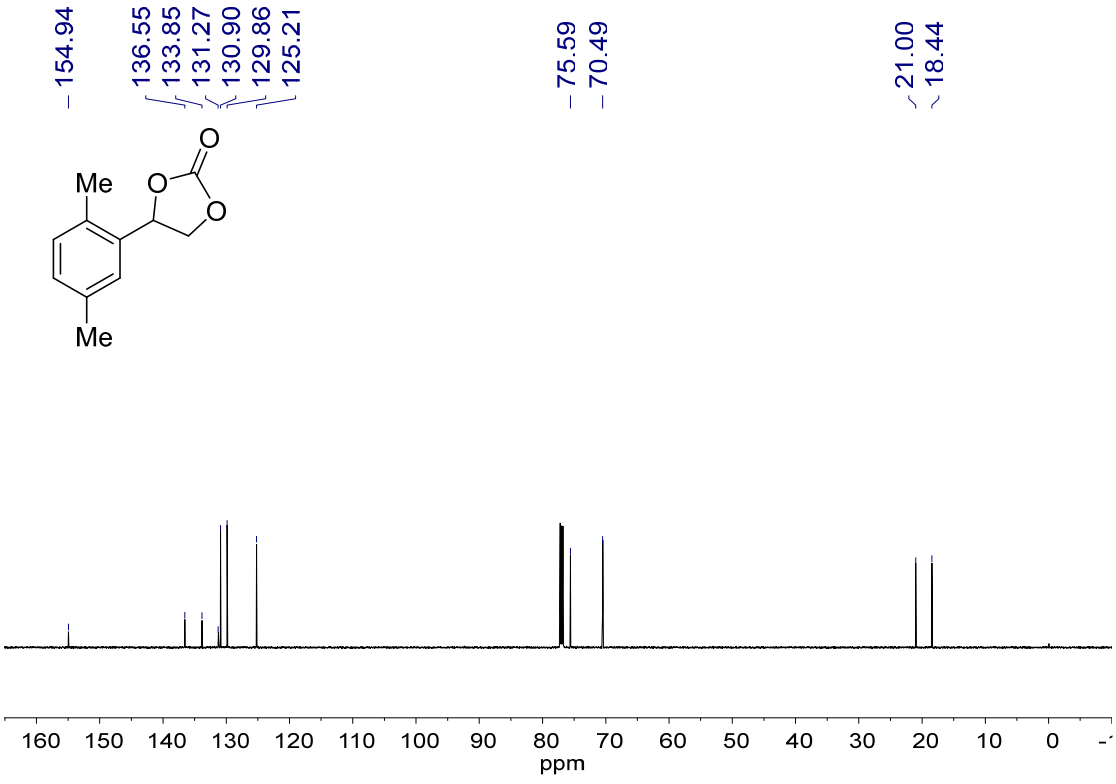
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 1x



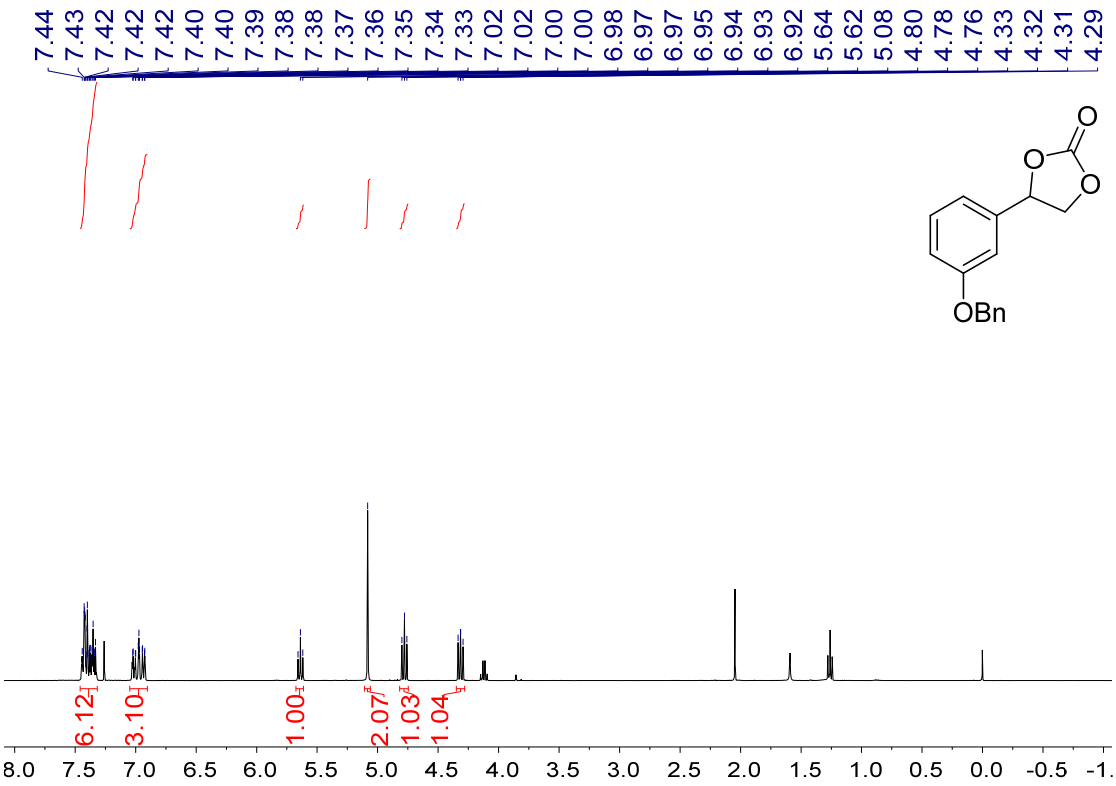
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 1x



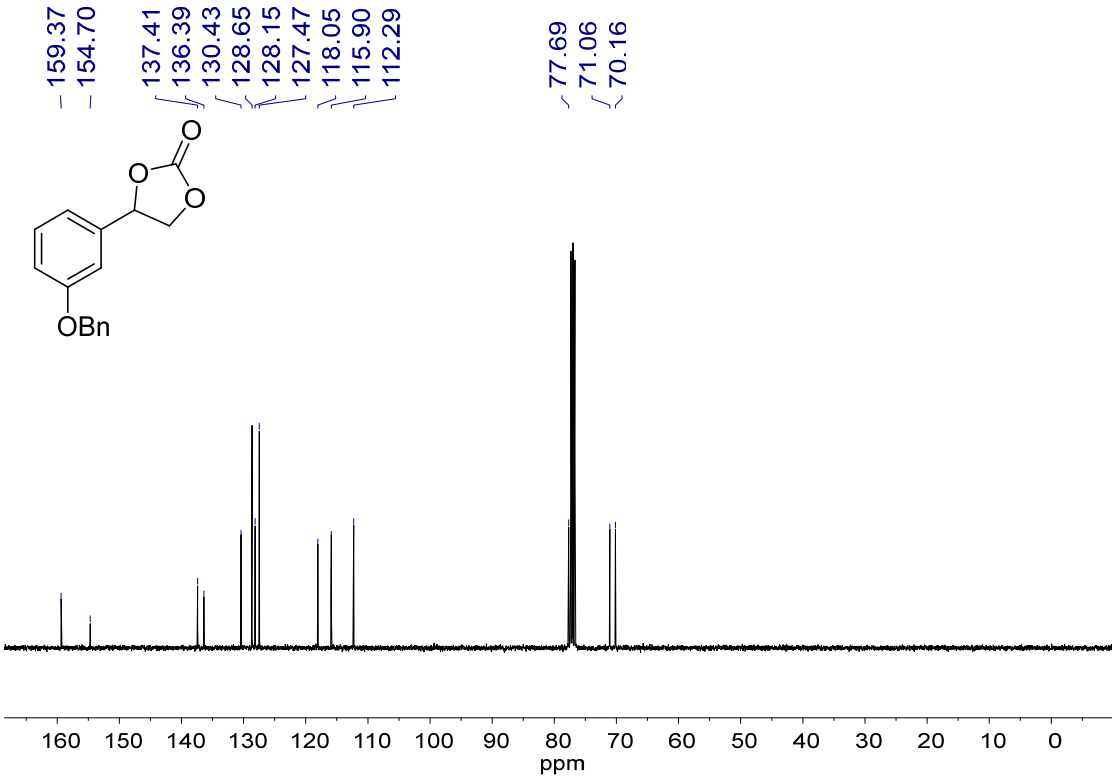
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 1ab



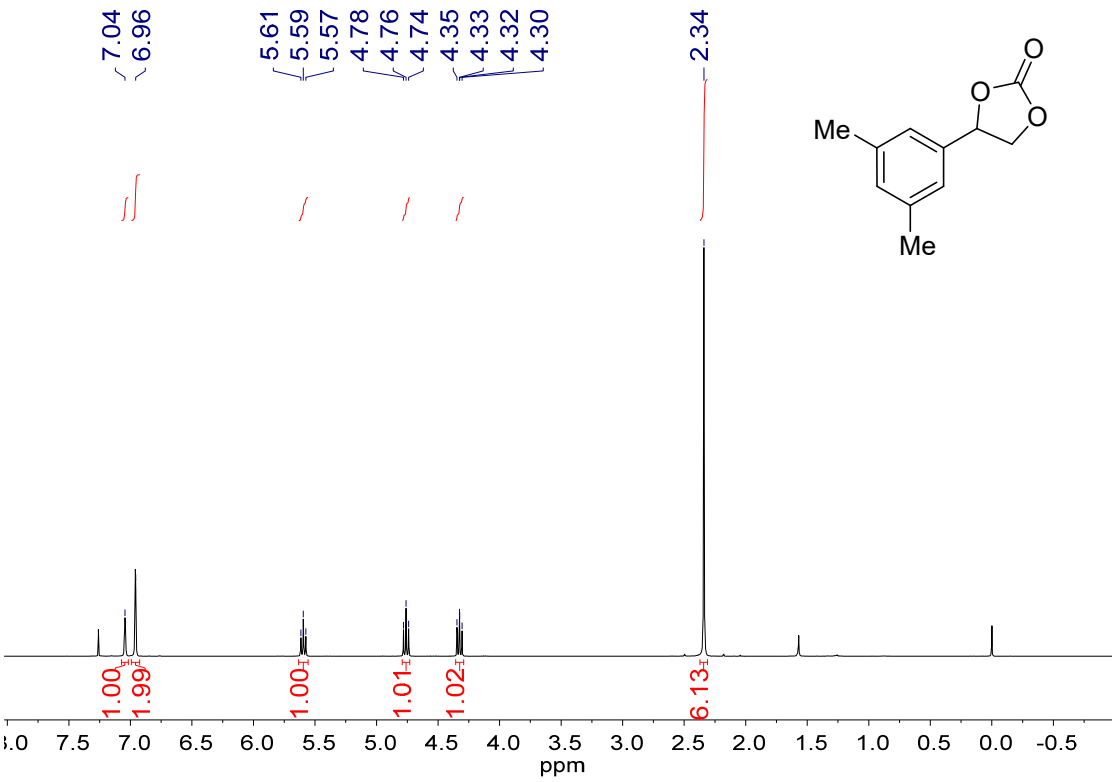
^{13}C{^{1}H} NMR spectrum (151 MHz, Chloroform-*d*) of 1ab



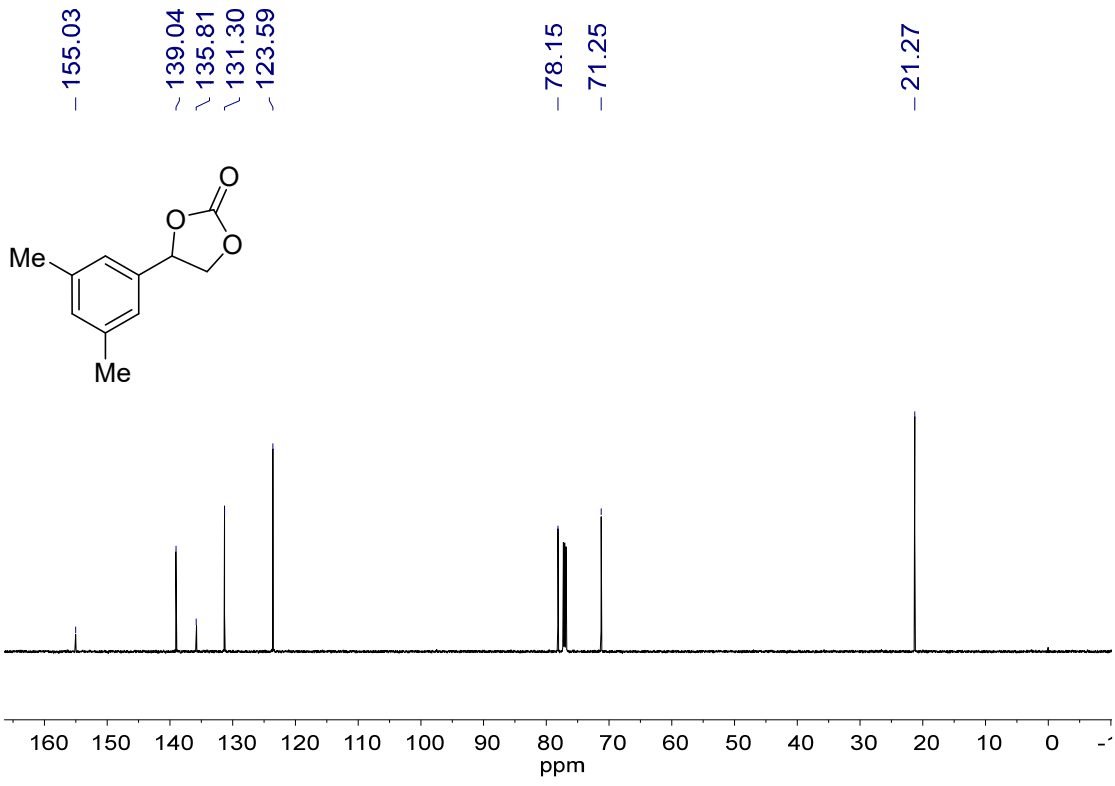
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 1ad



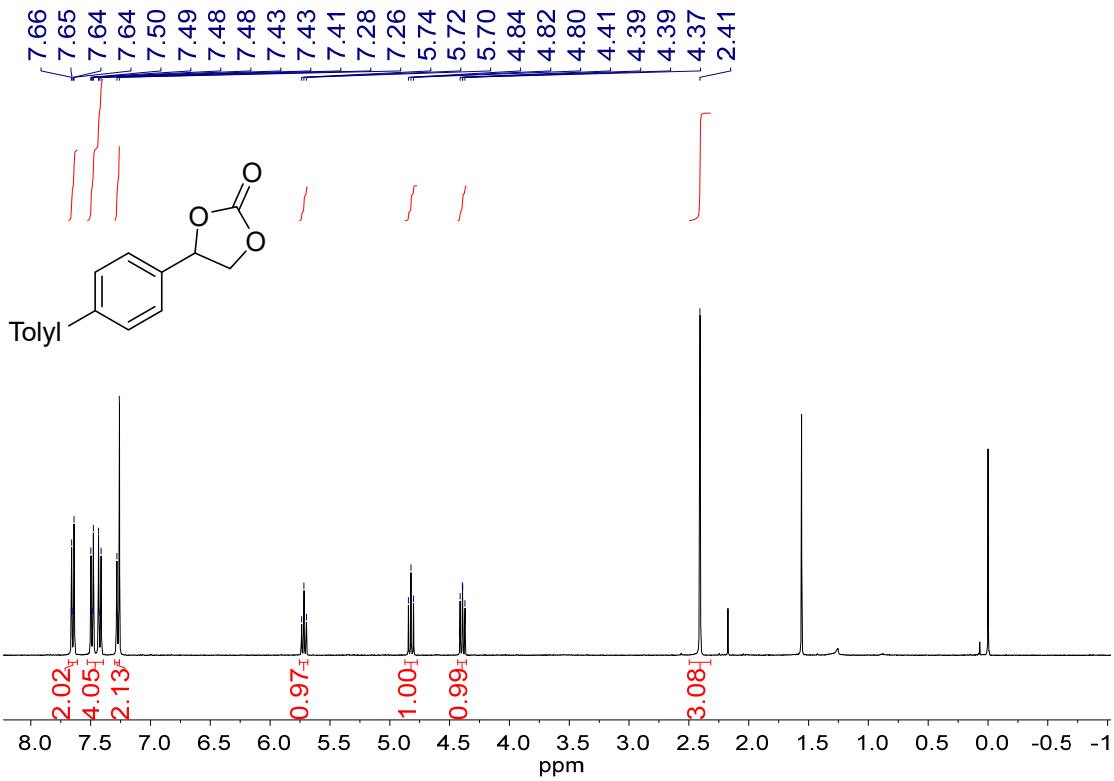
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 1ad



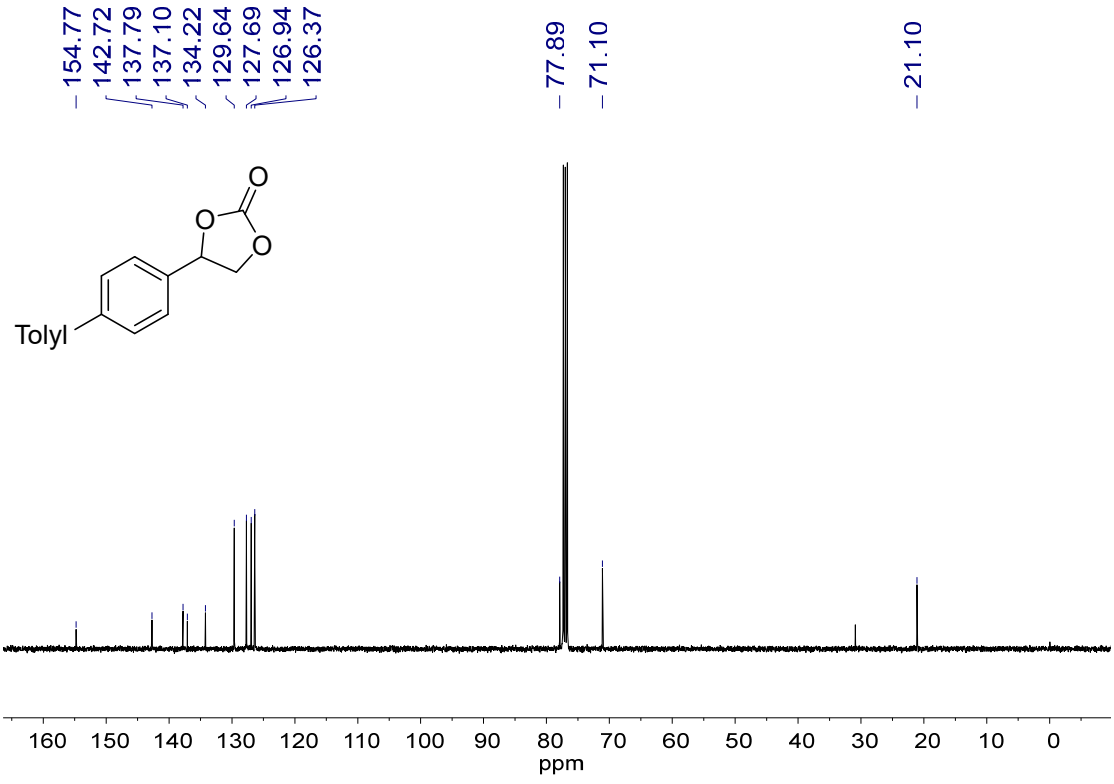
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 1ae



^{13}C{^{1}H} NMR spectrum (151 MHz, Chloroform-*d*) of 1ae



^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 1ag



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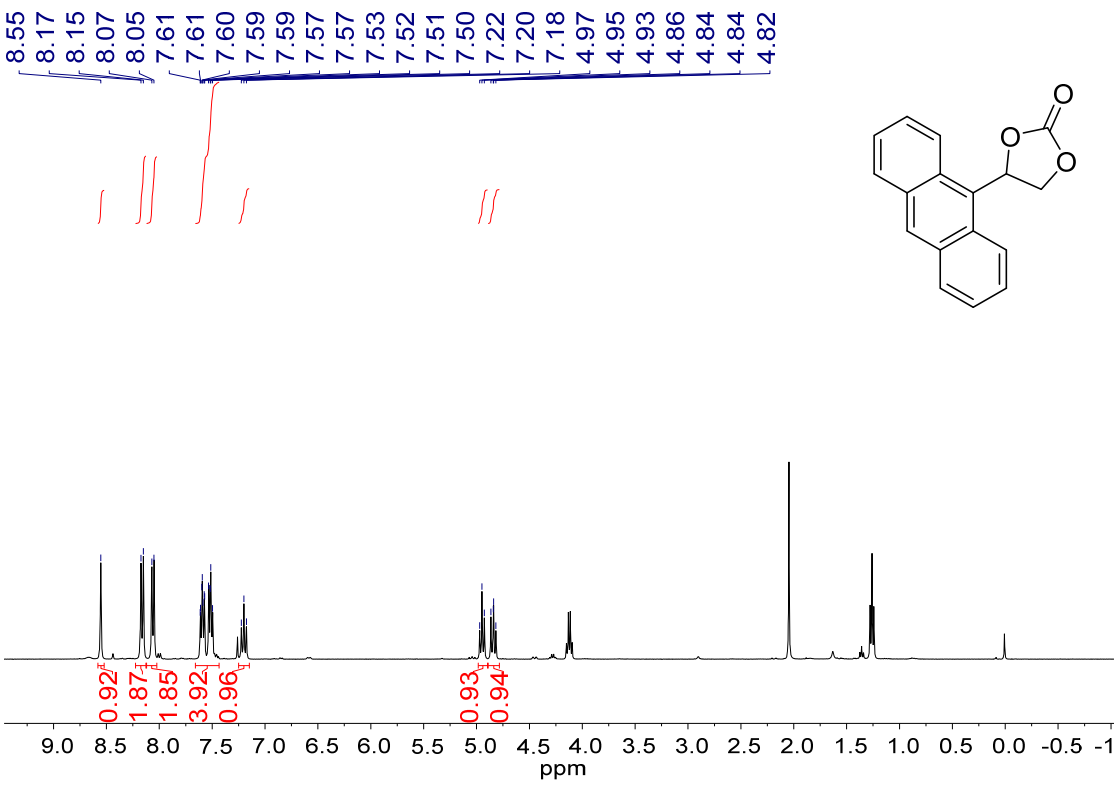
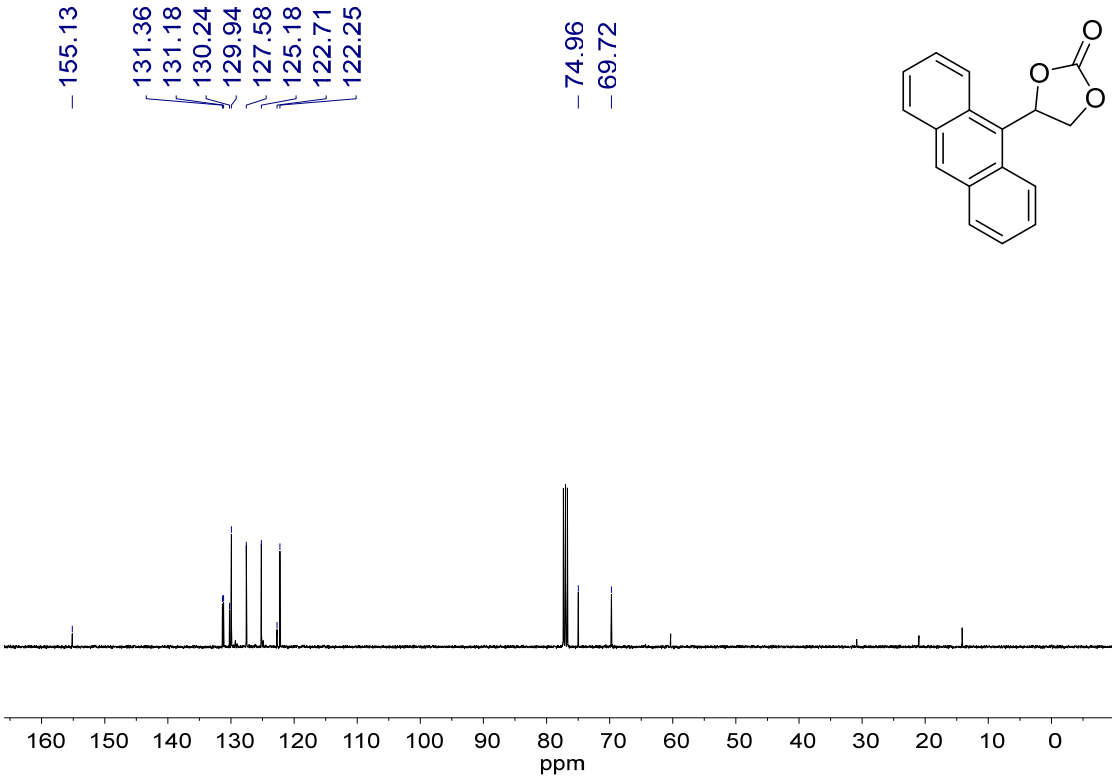
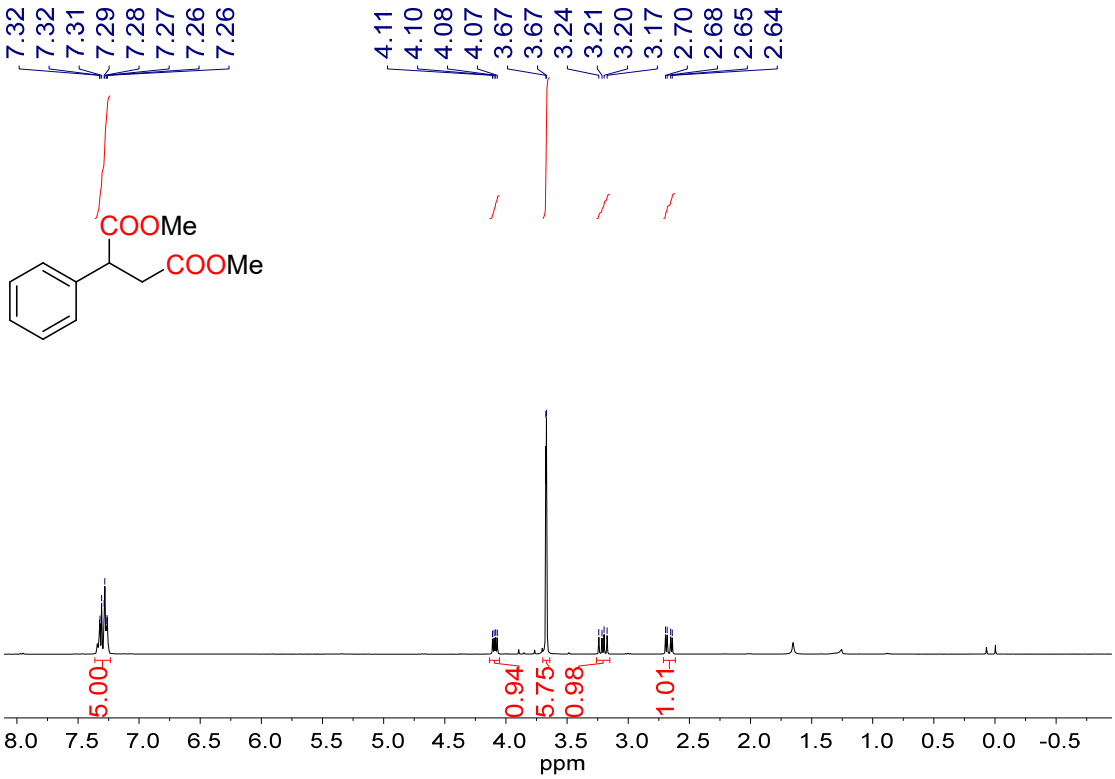


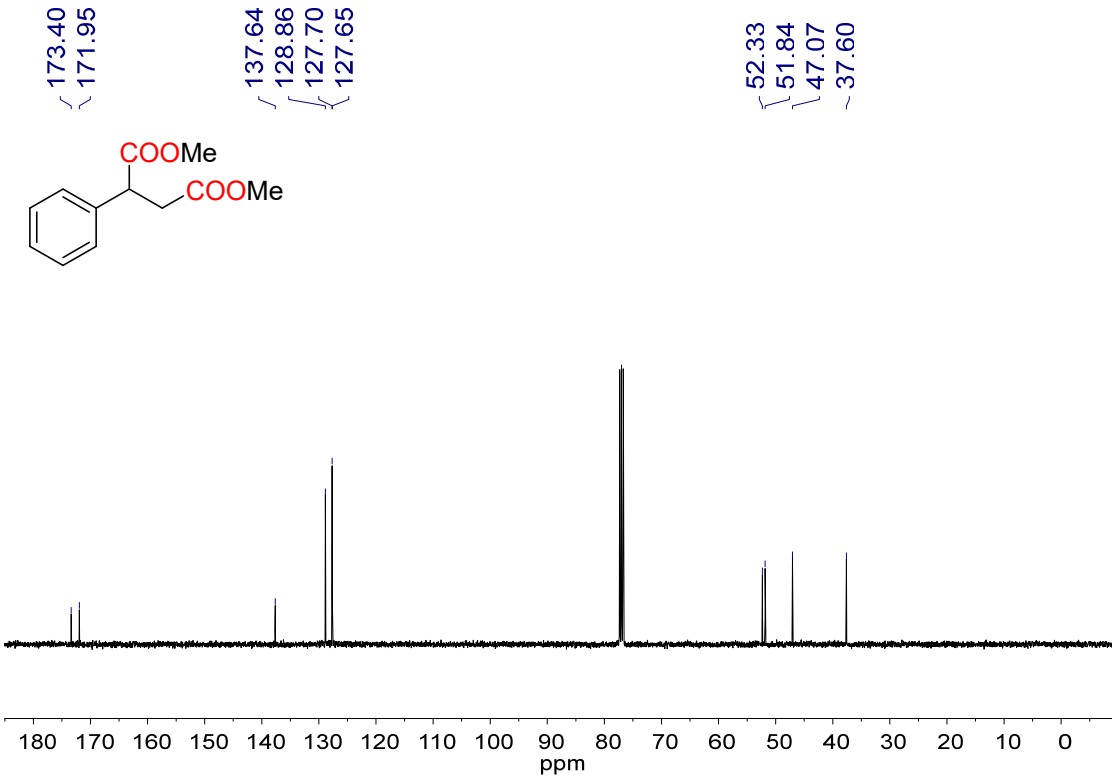
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 1ai



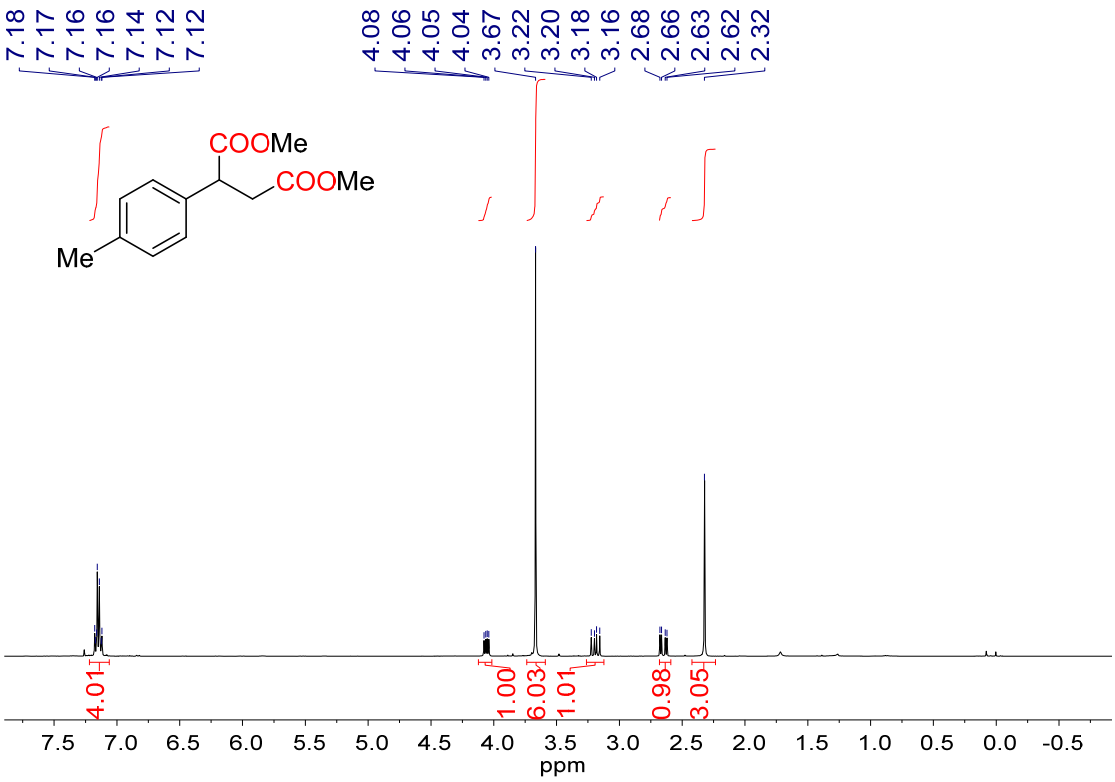
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 1ai



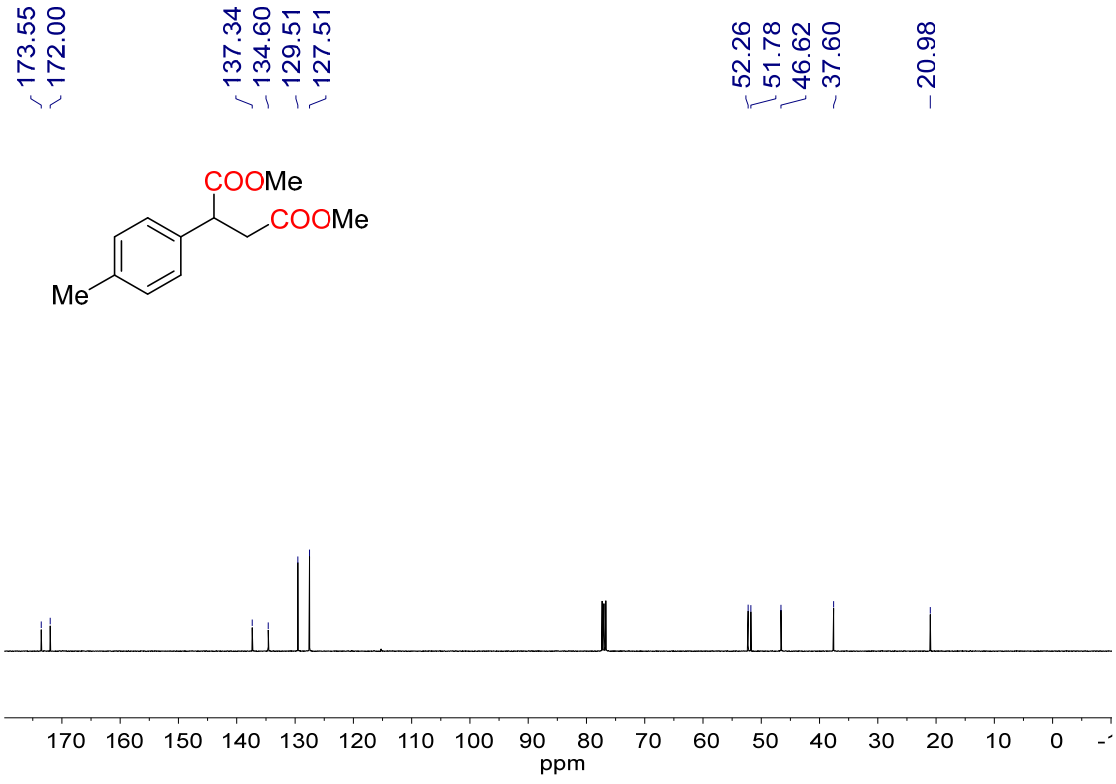
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 2a



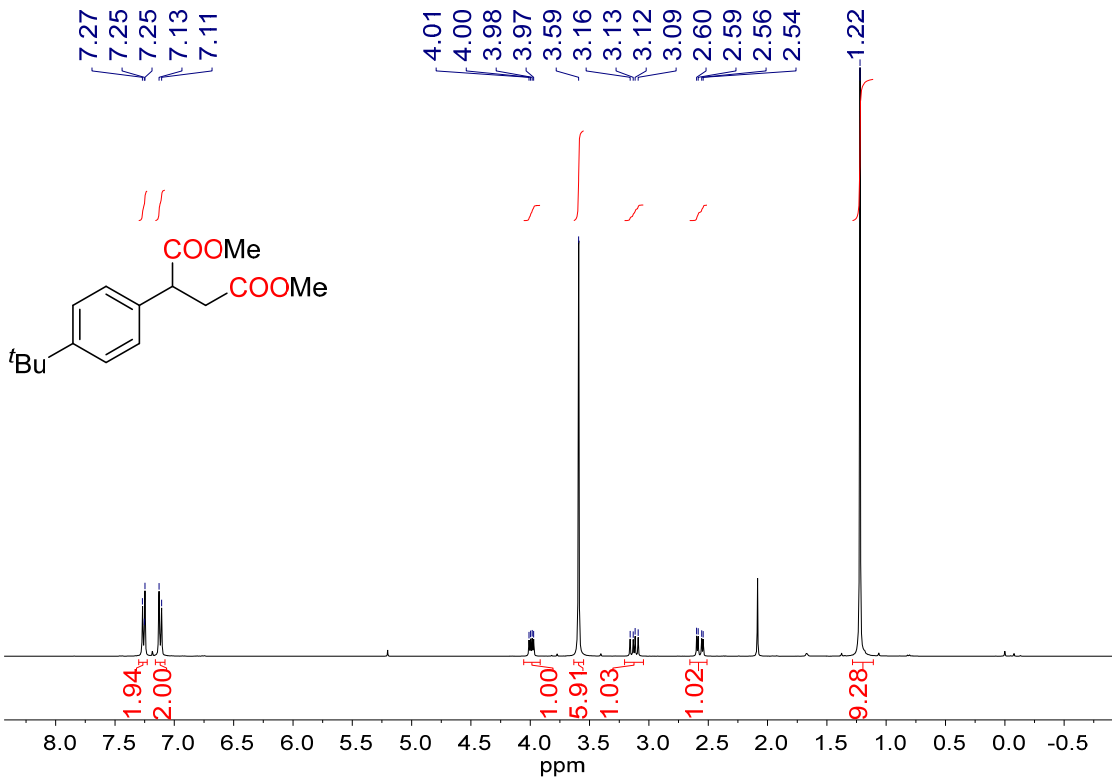
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 2a



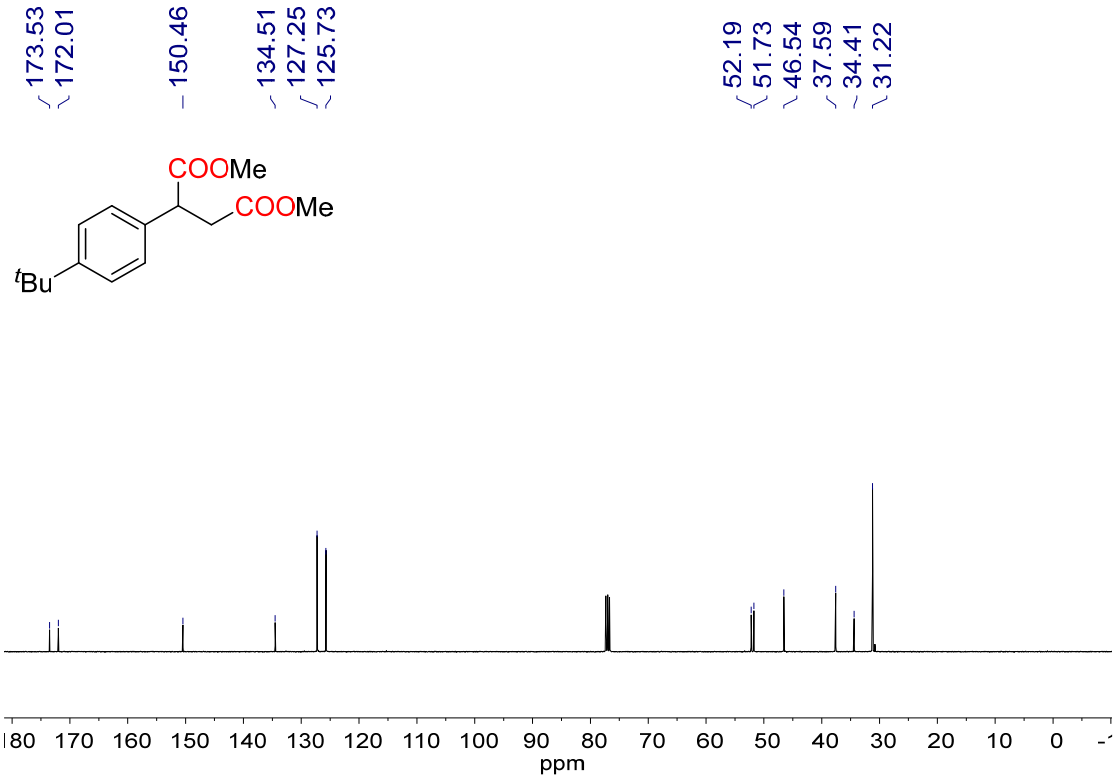
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 2b



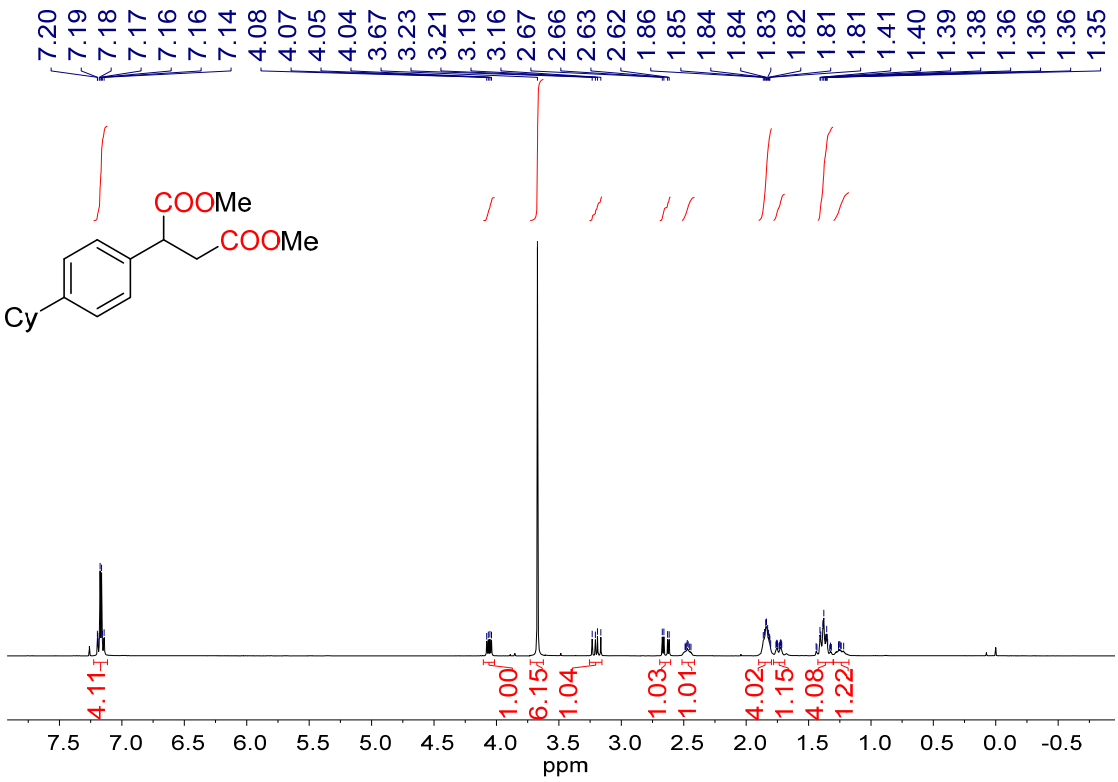
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 2b



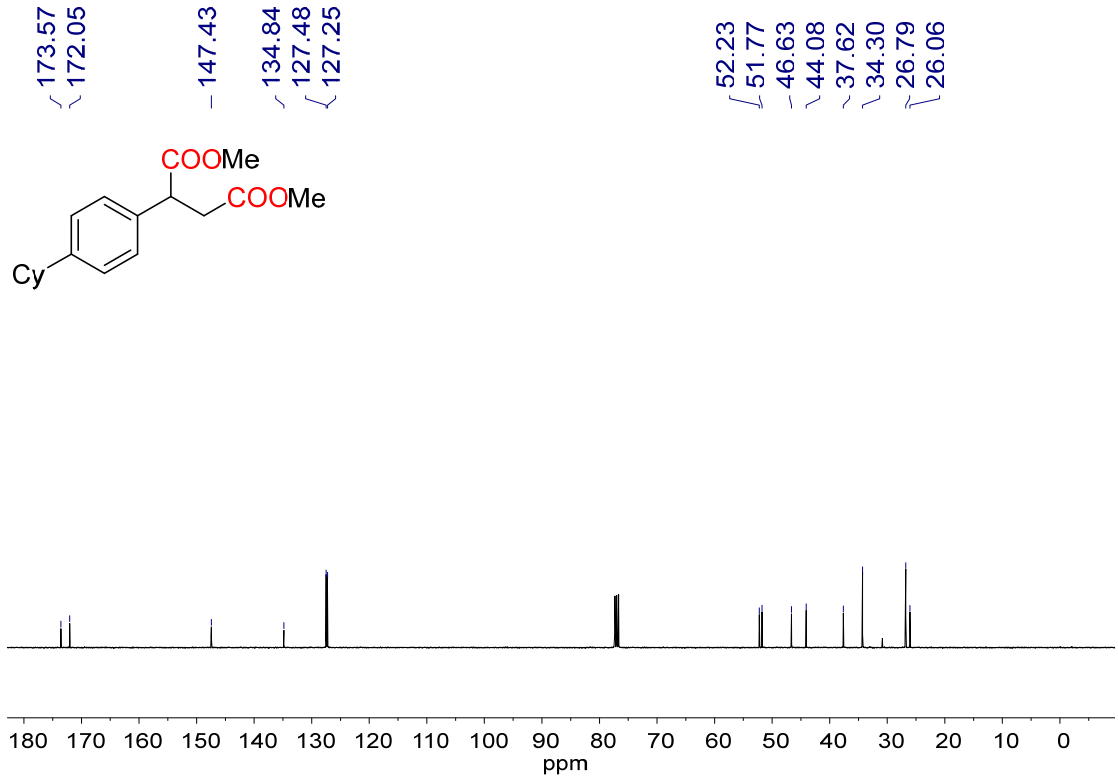
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 2c



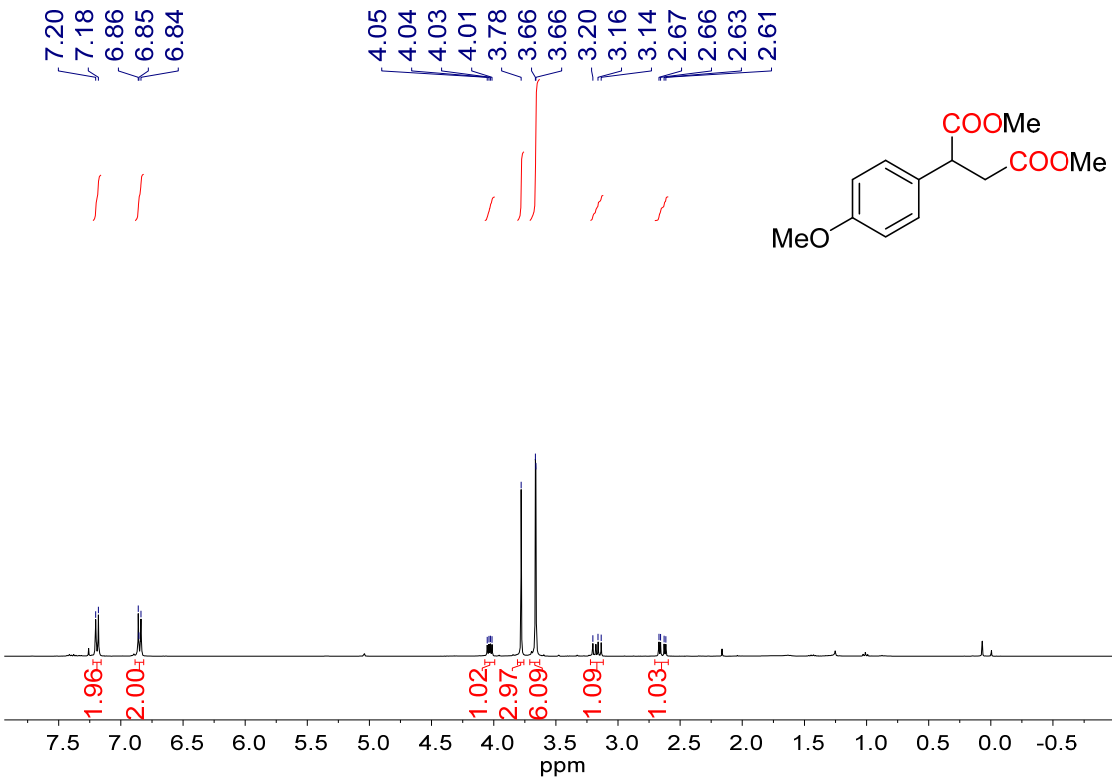
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 2c



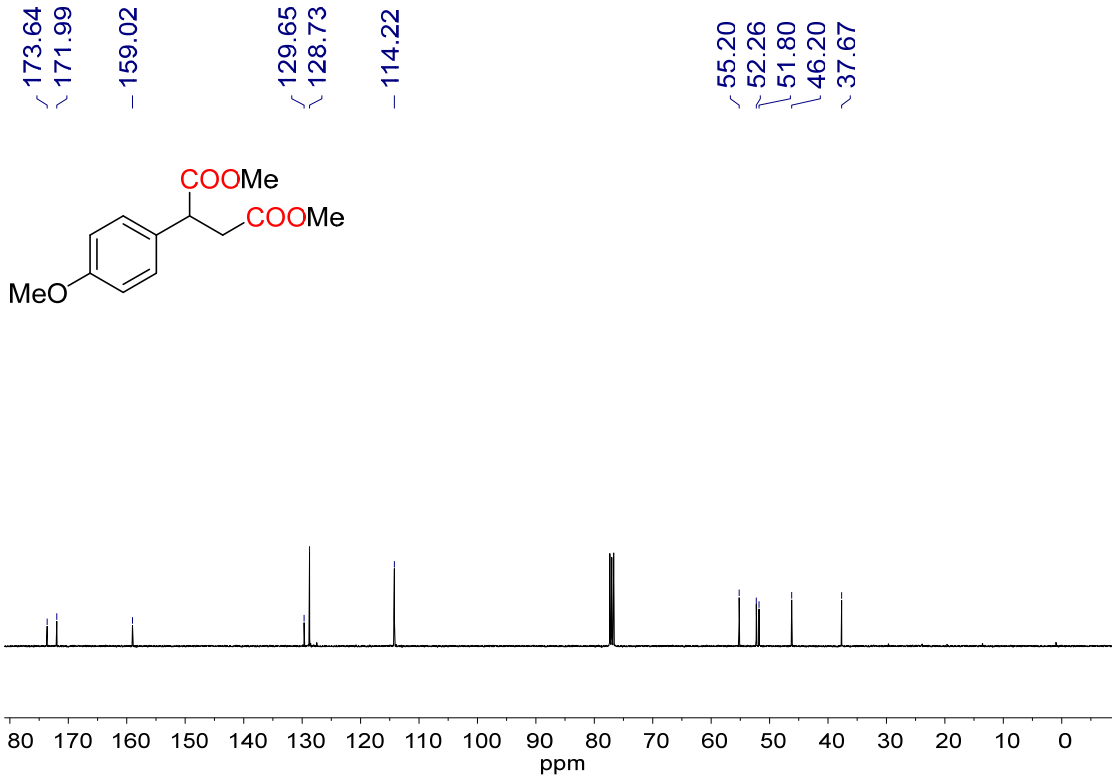
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 2d



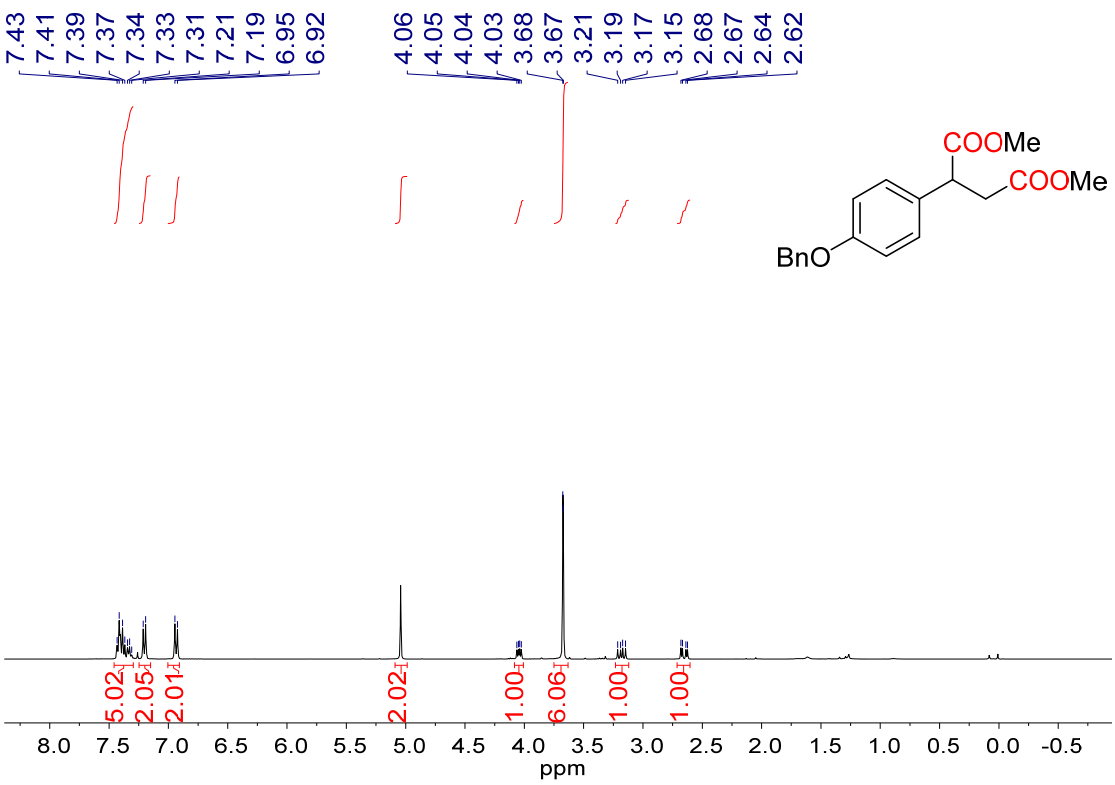
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 2d



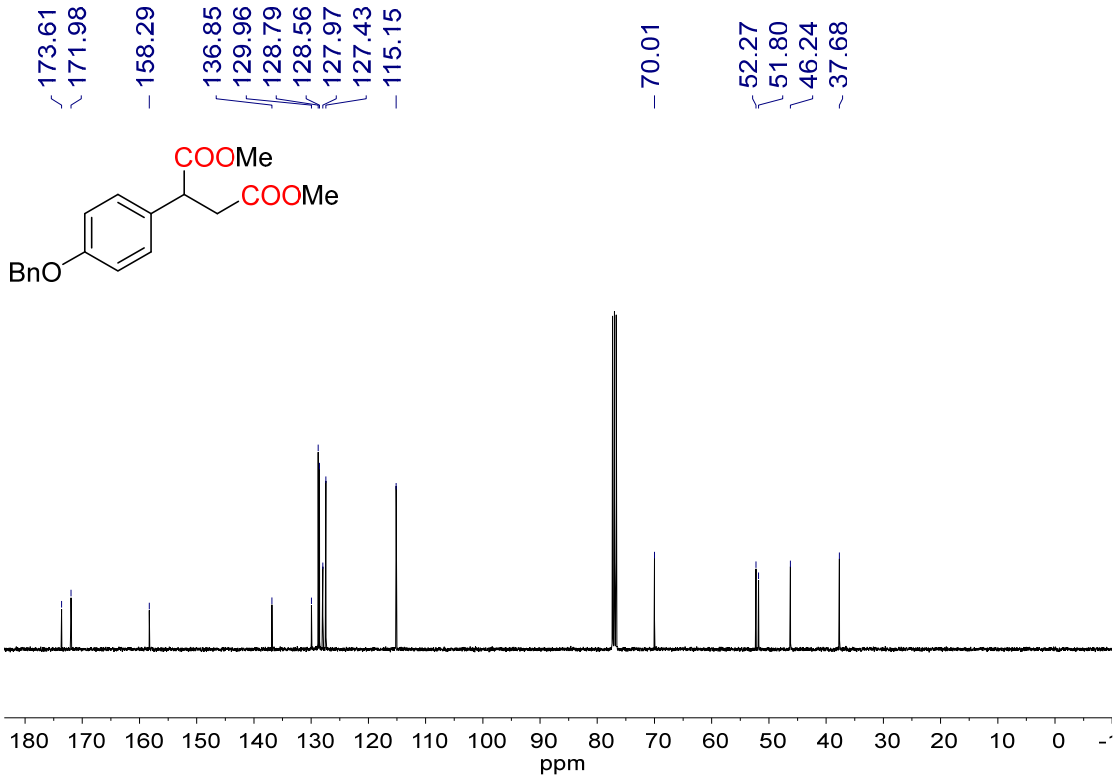
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 2e



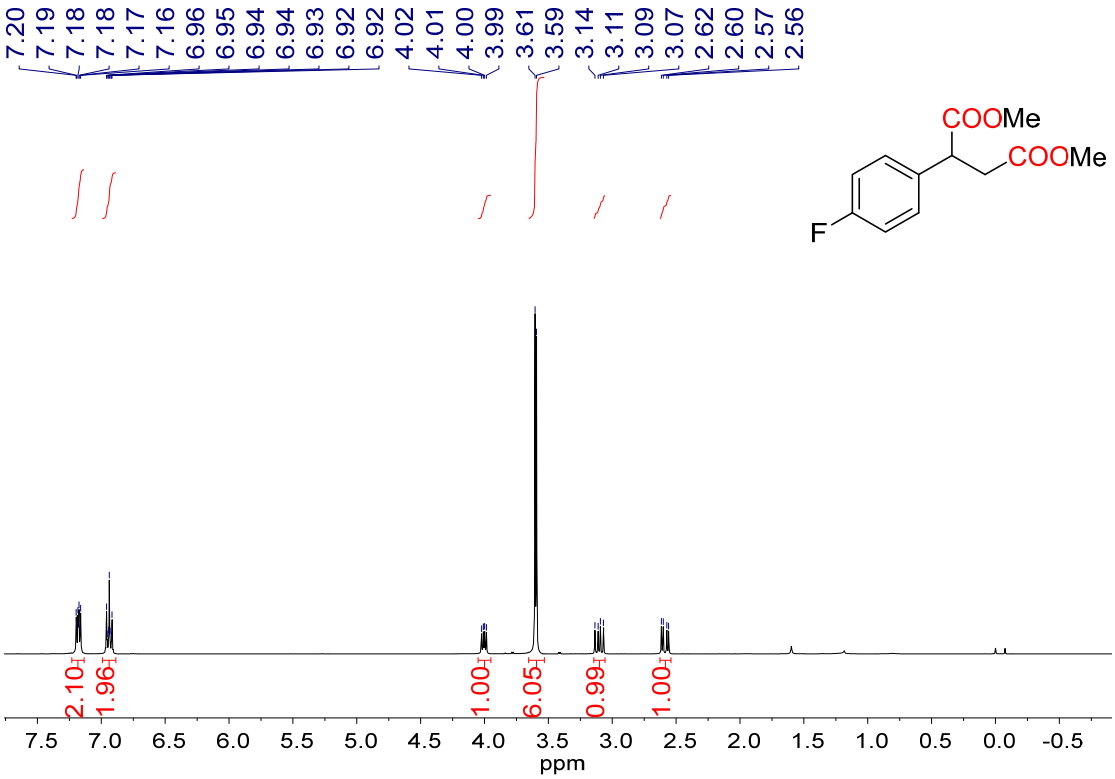
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 2e



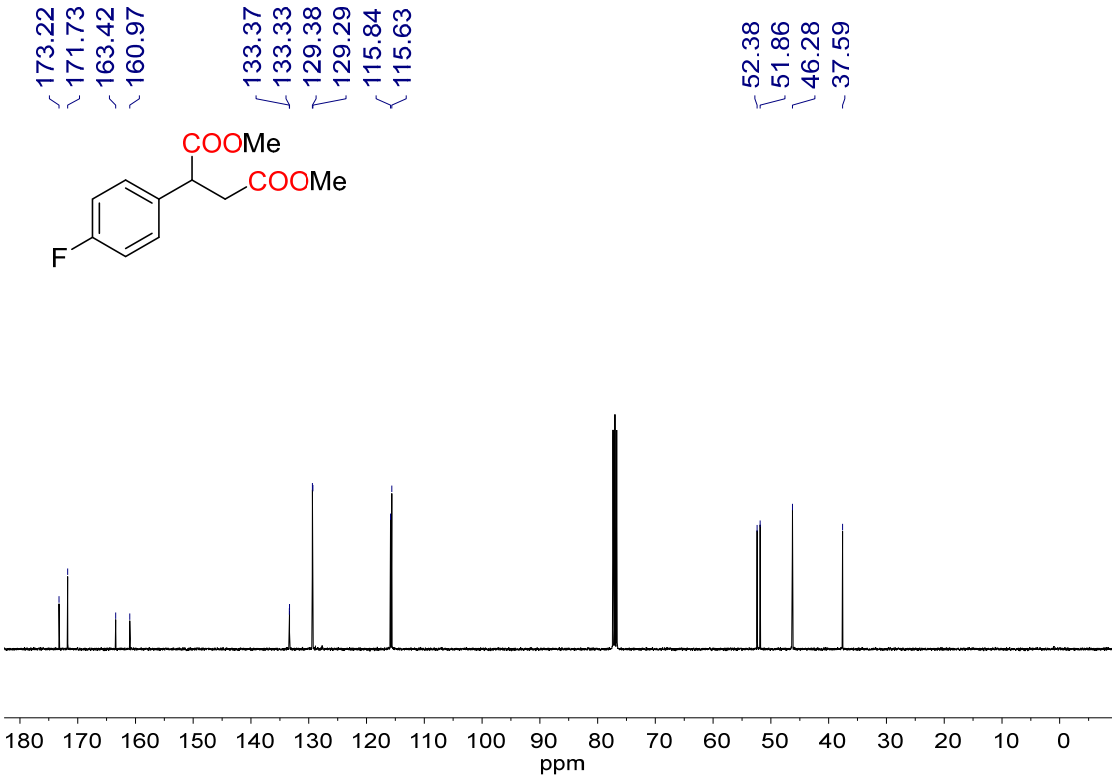
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 2f



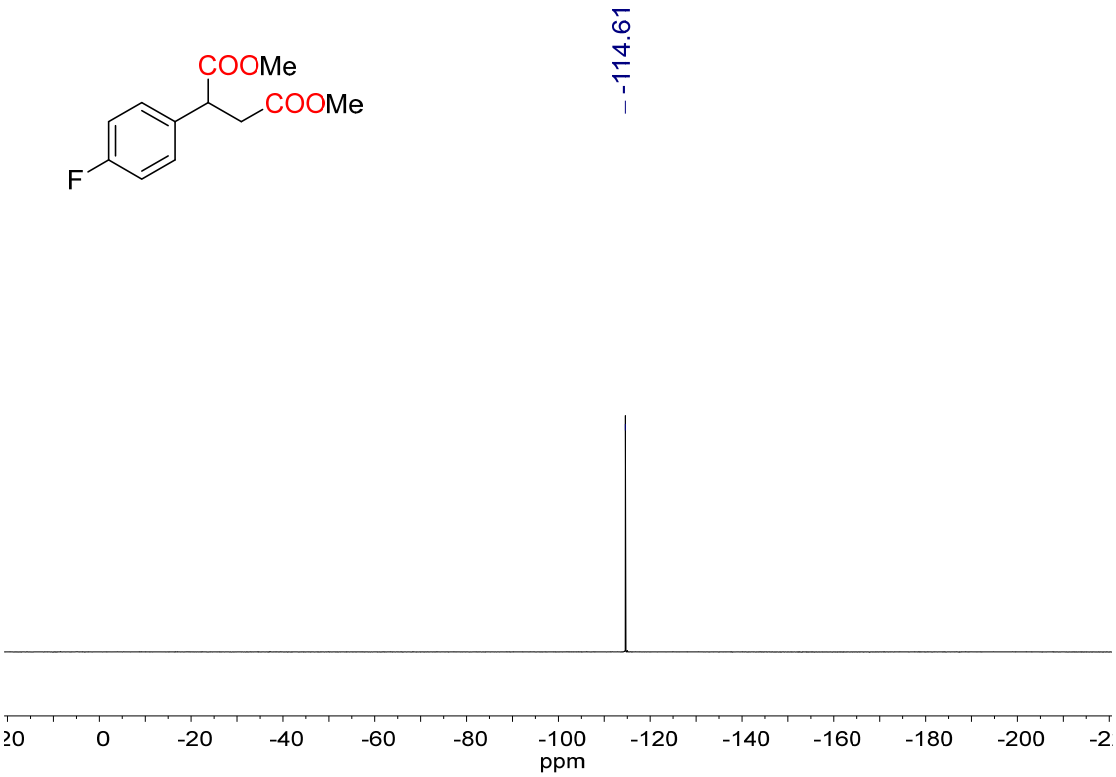
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 2f



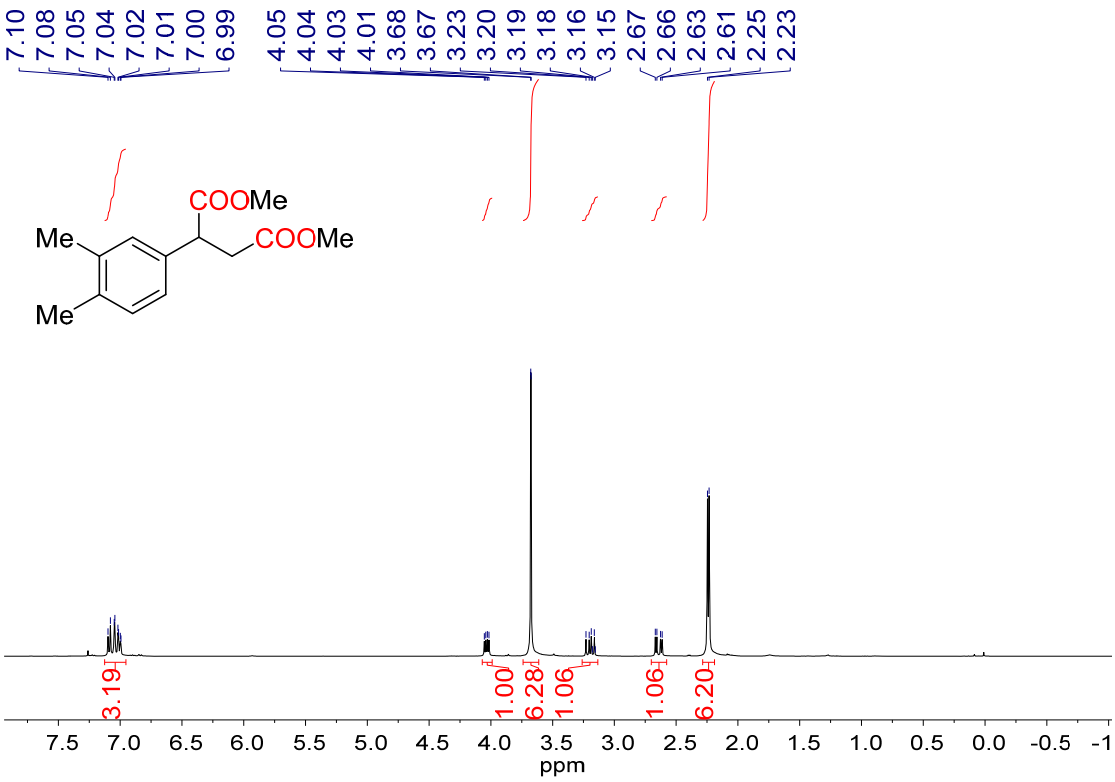
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 2g



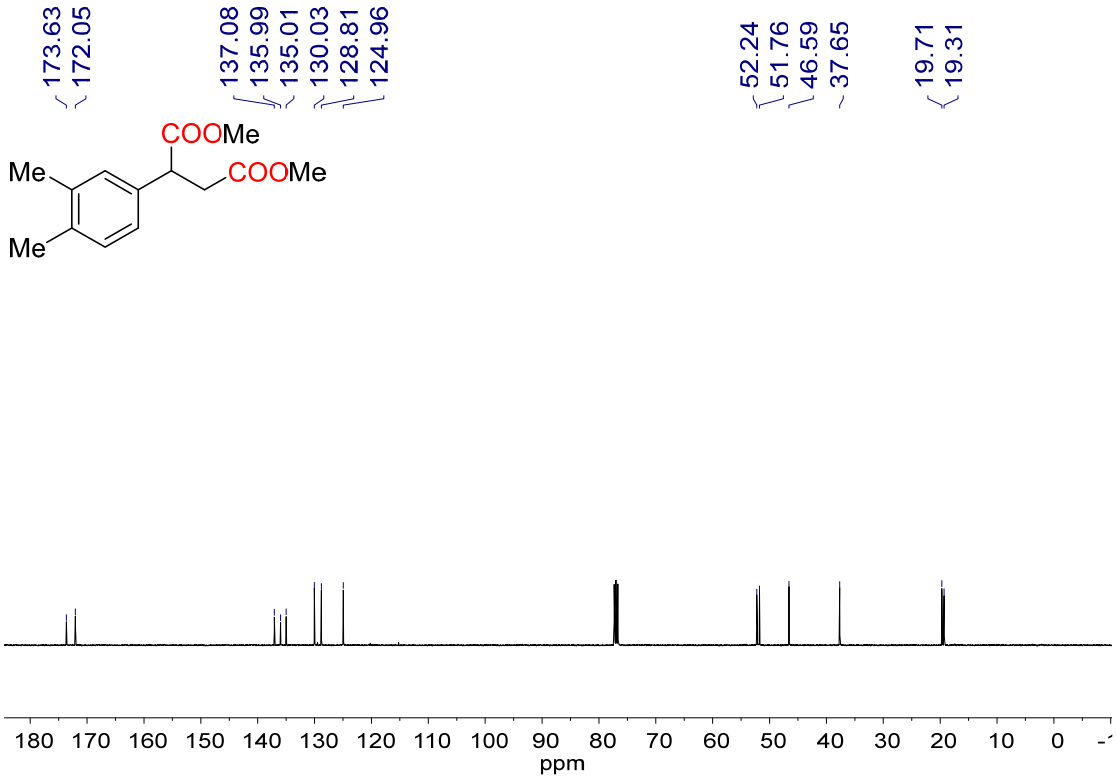
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 2g



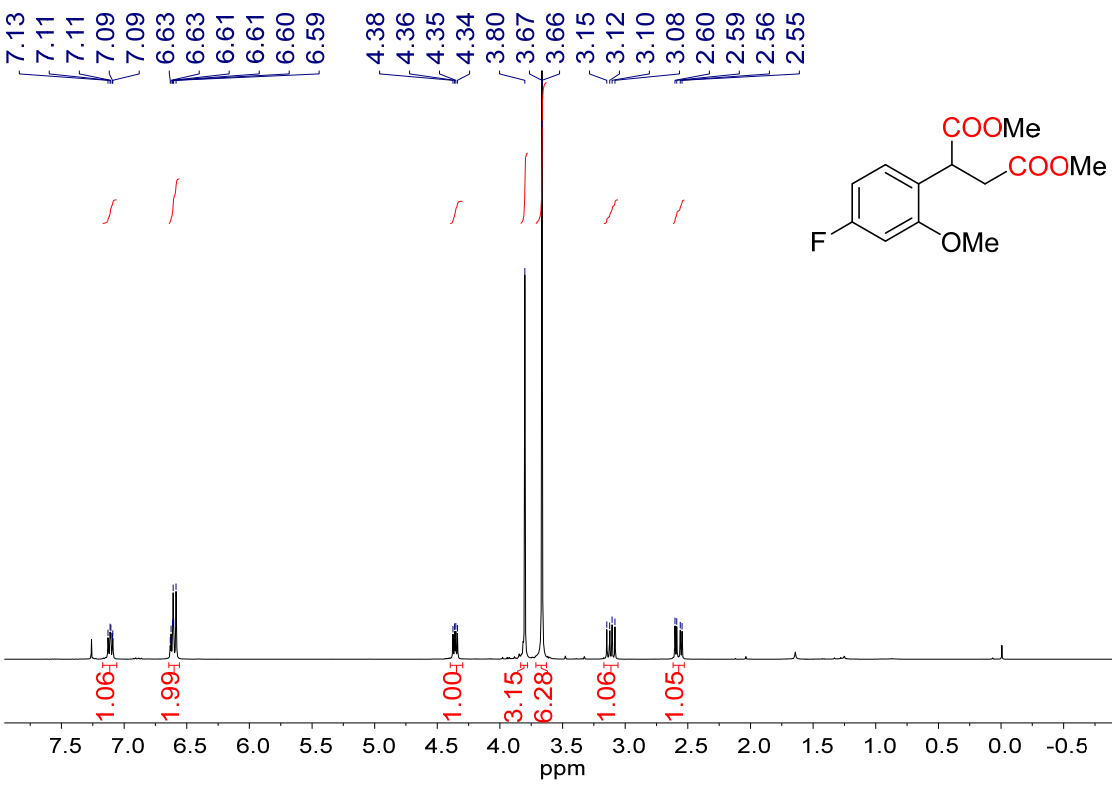
^{19}F NMR spectrum (376 MHz, Chloroform-*d*) of 2g



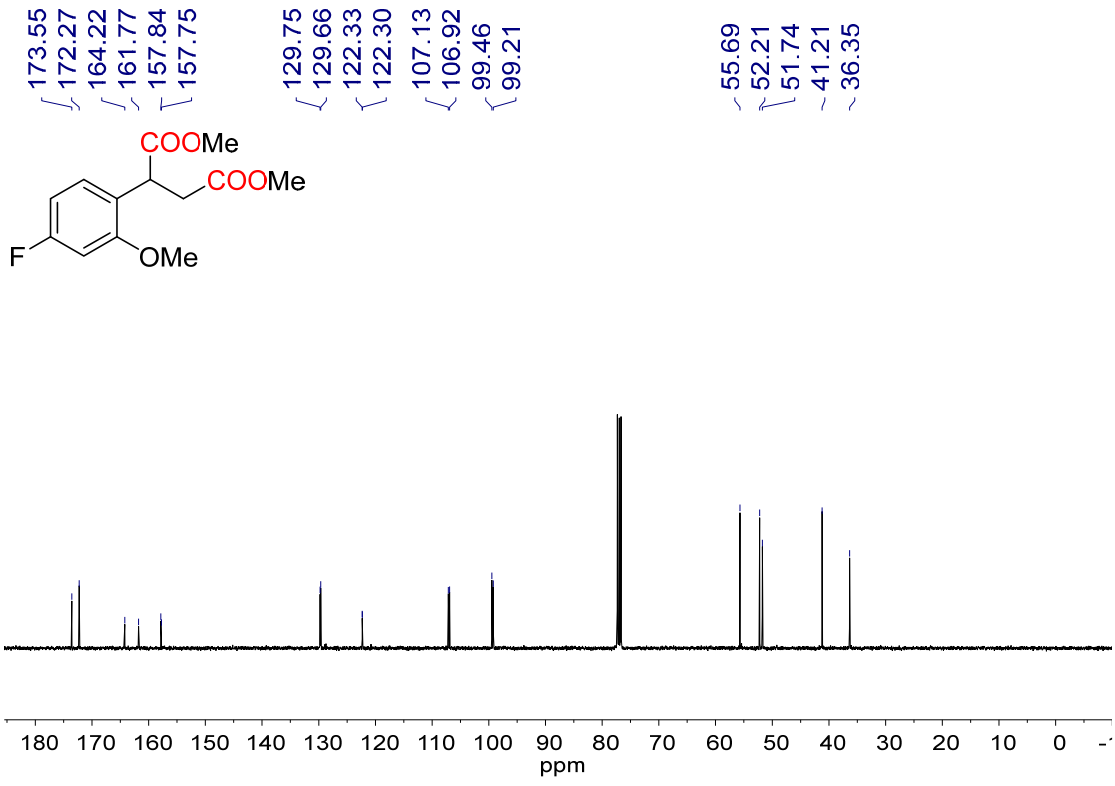
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 2h



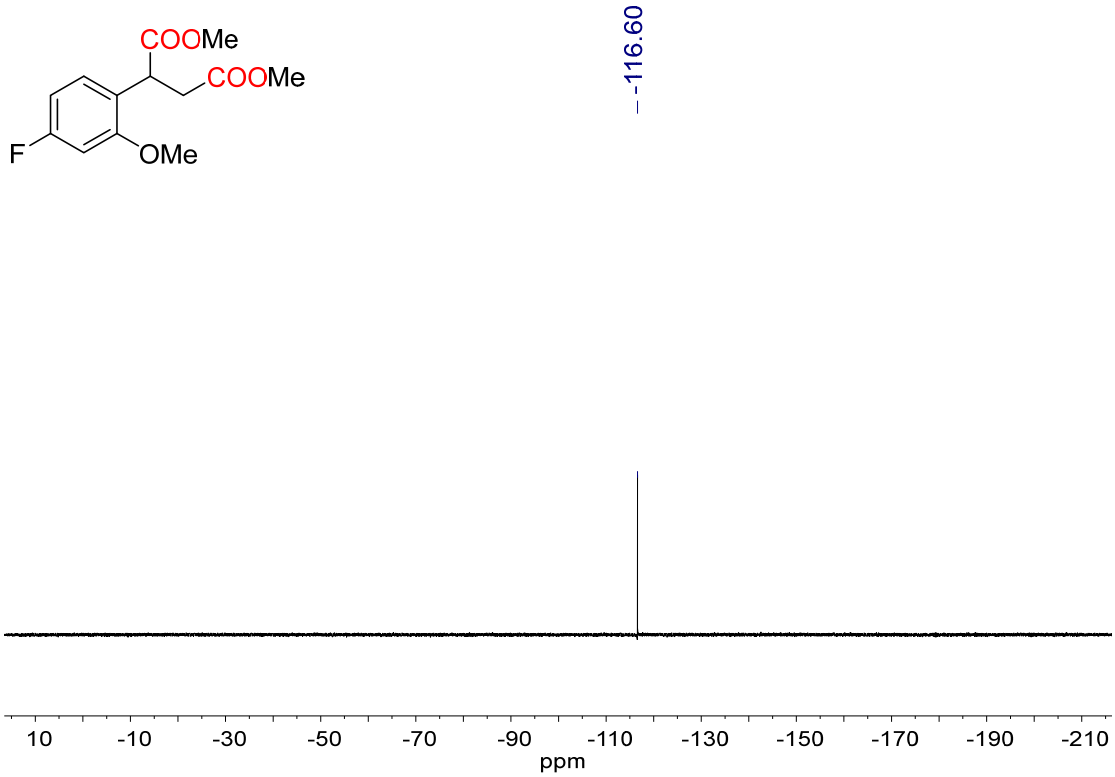
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 2h



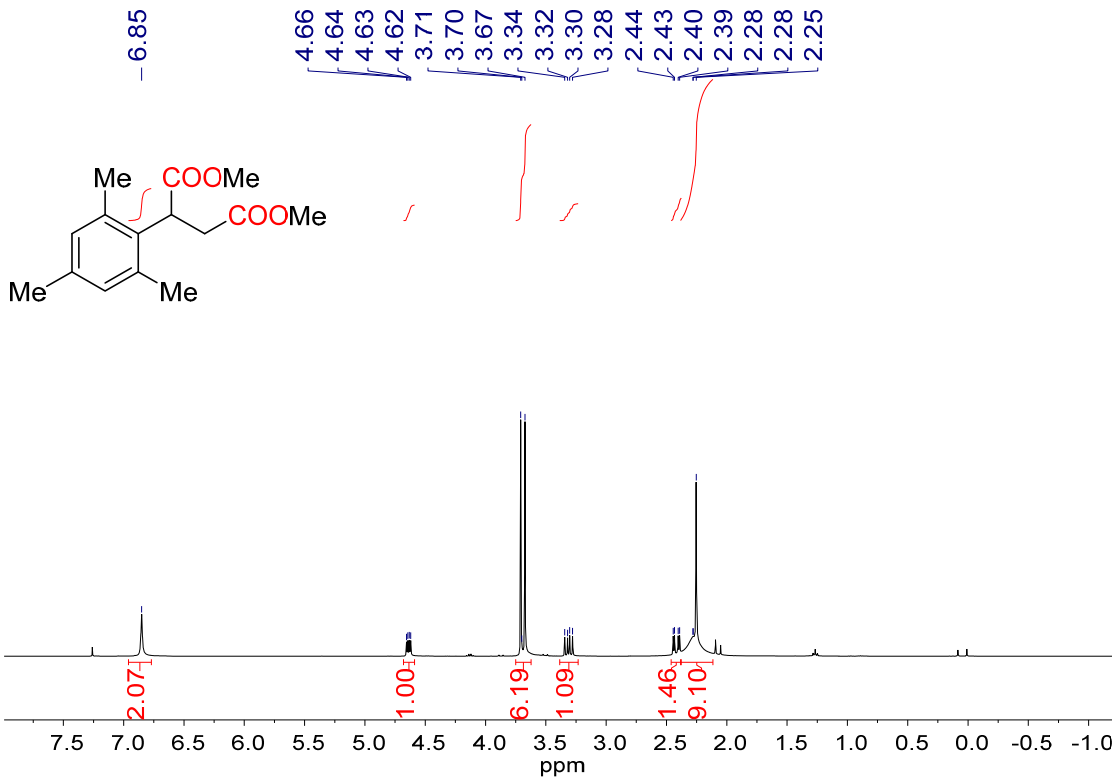
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 2i



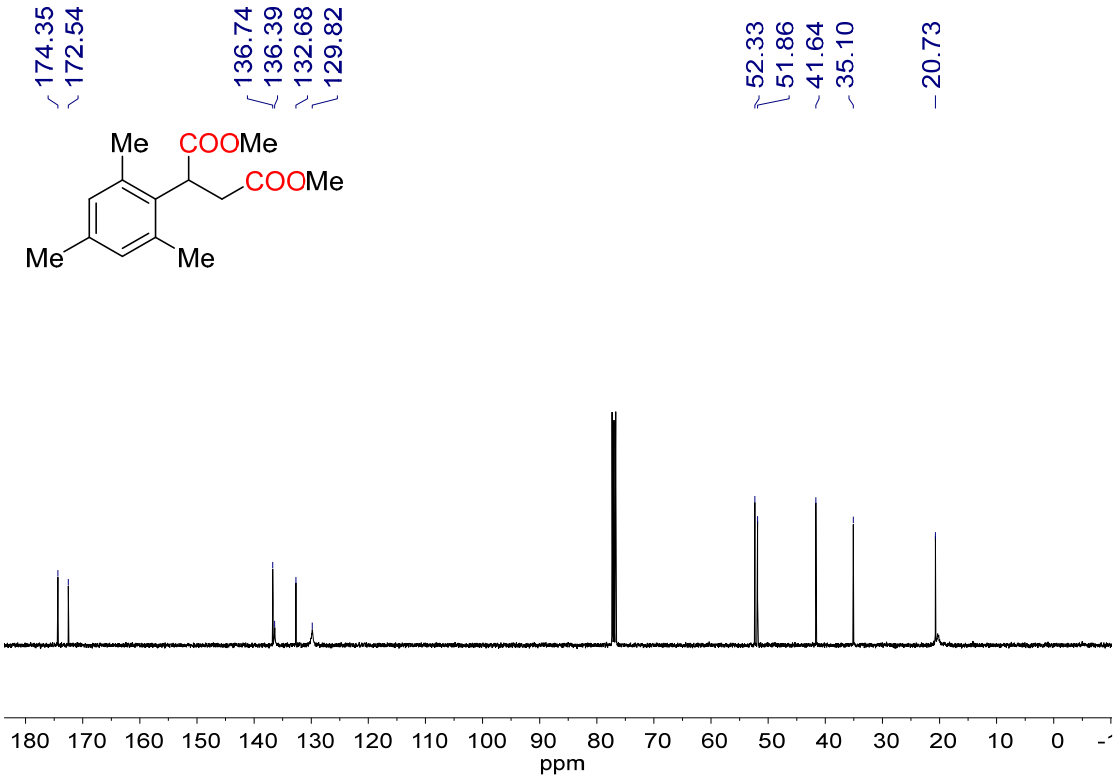
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 2i



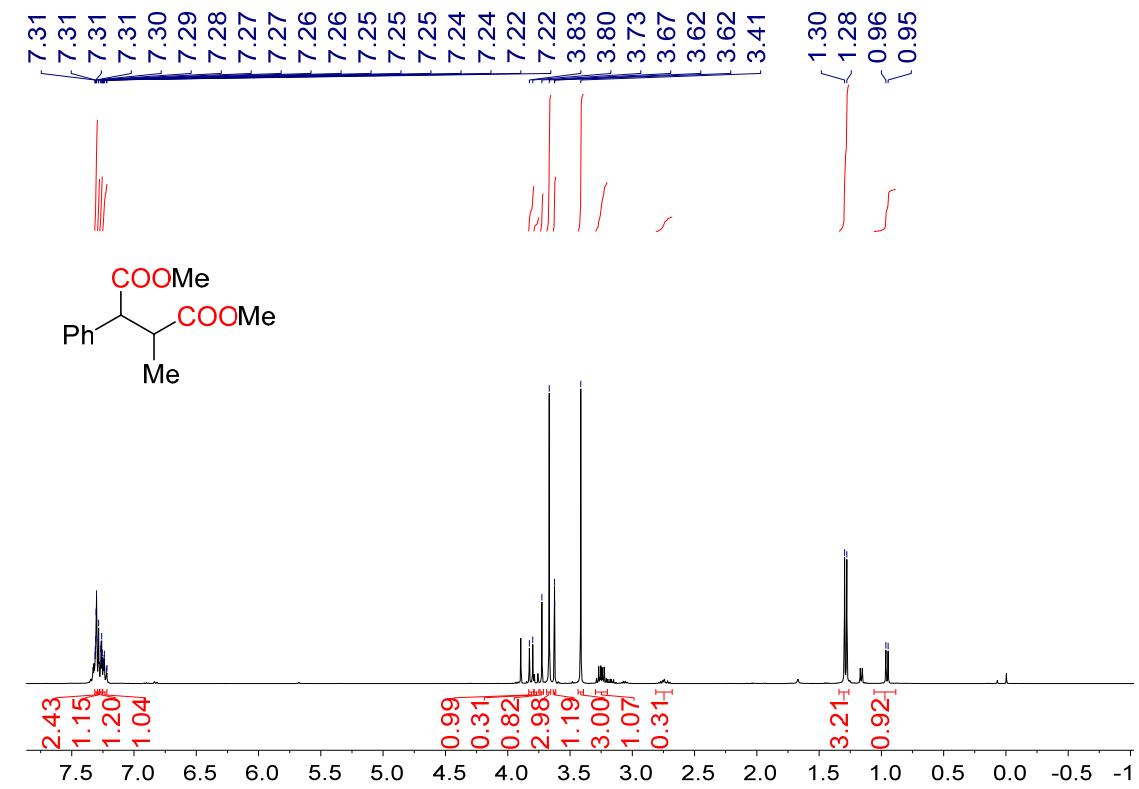
^{19}F NMR spectrum (376 MHz, Chloroform-*d*) of 2i



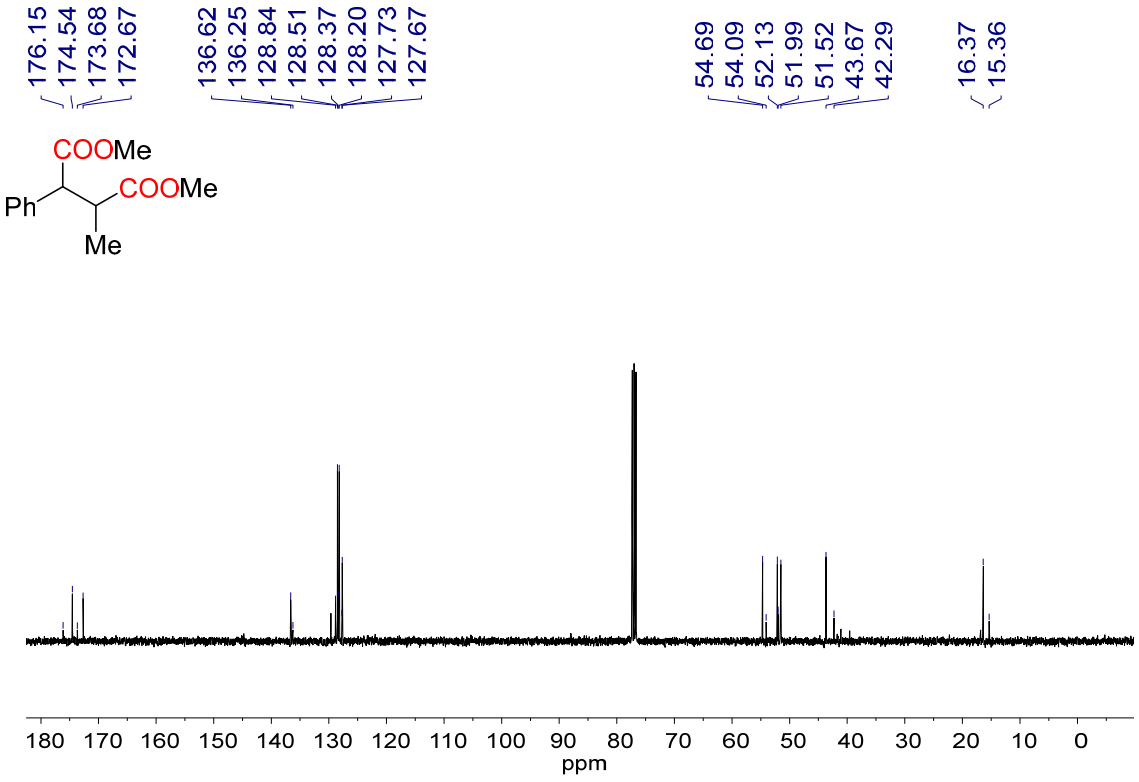
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 2j



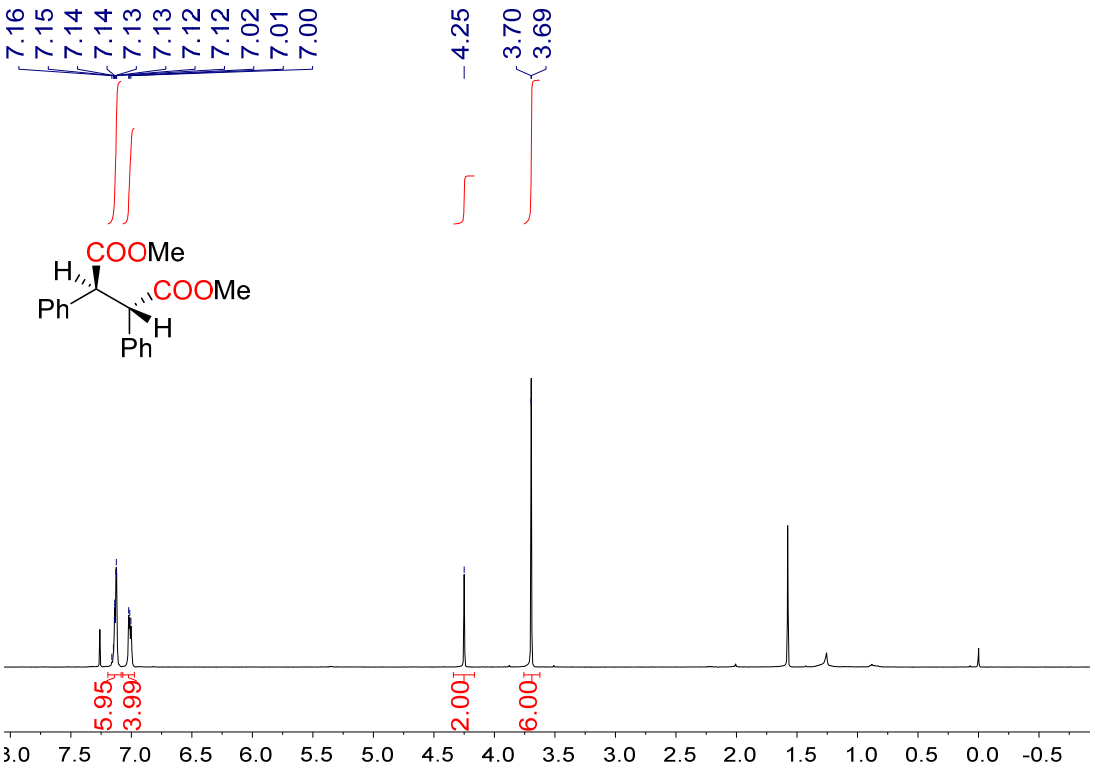
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 2j



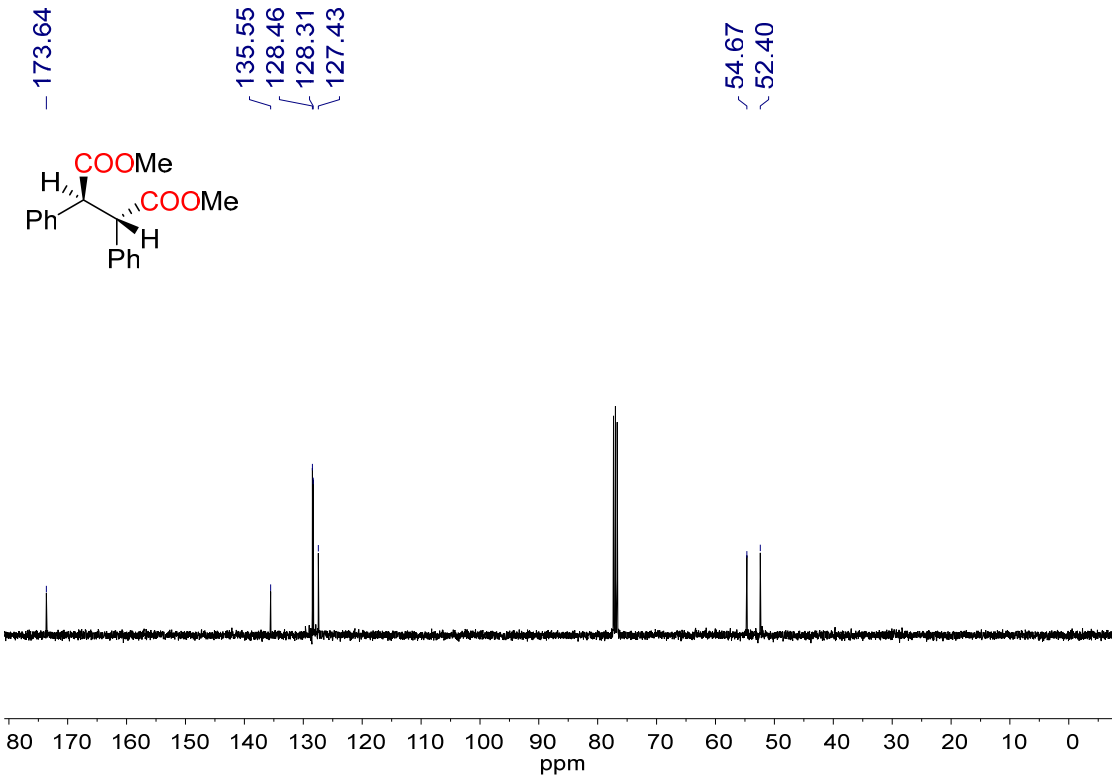
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 2k



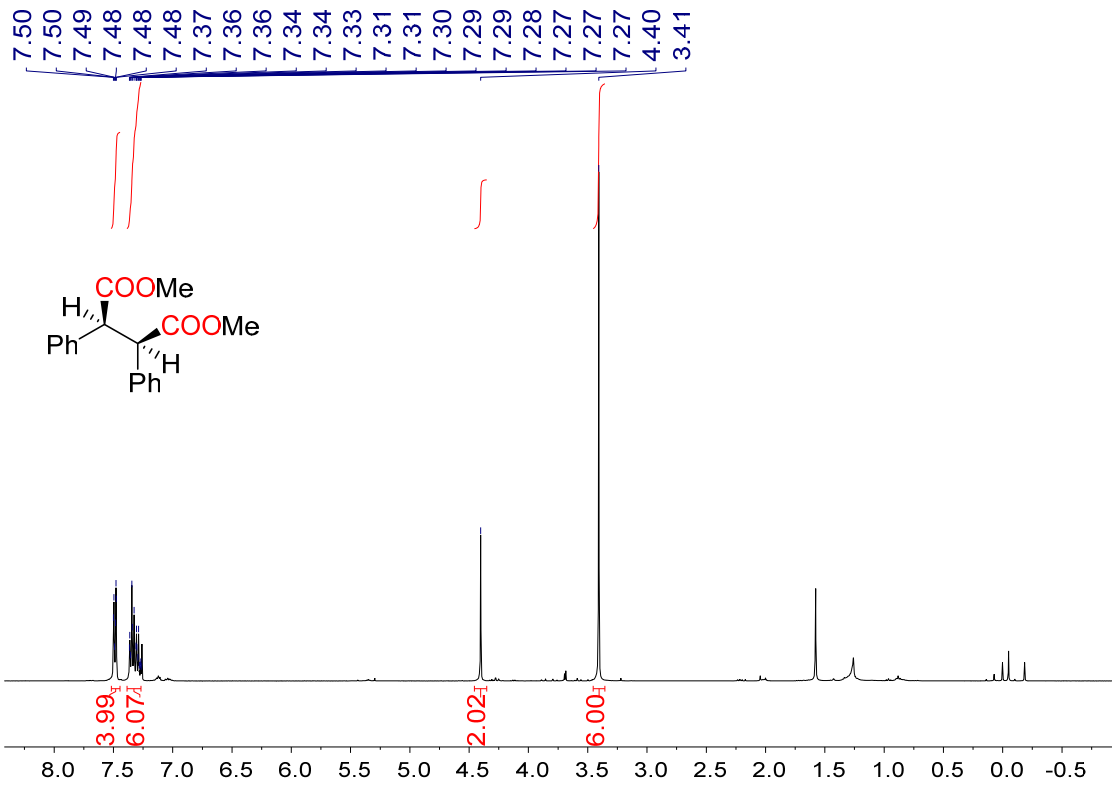
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 2k



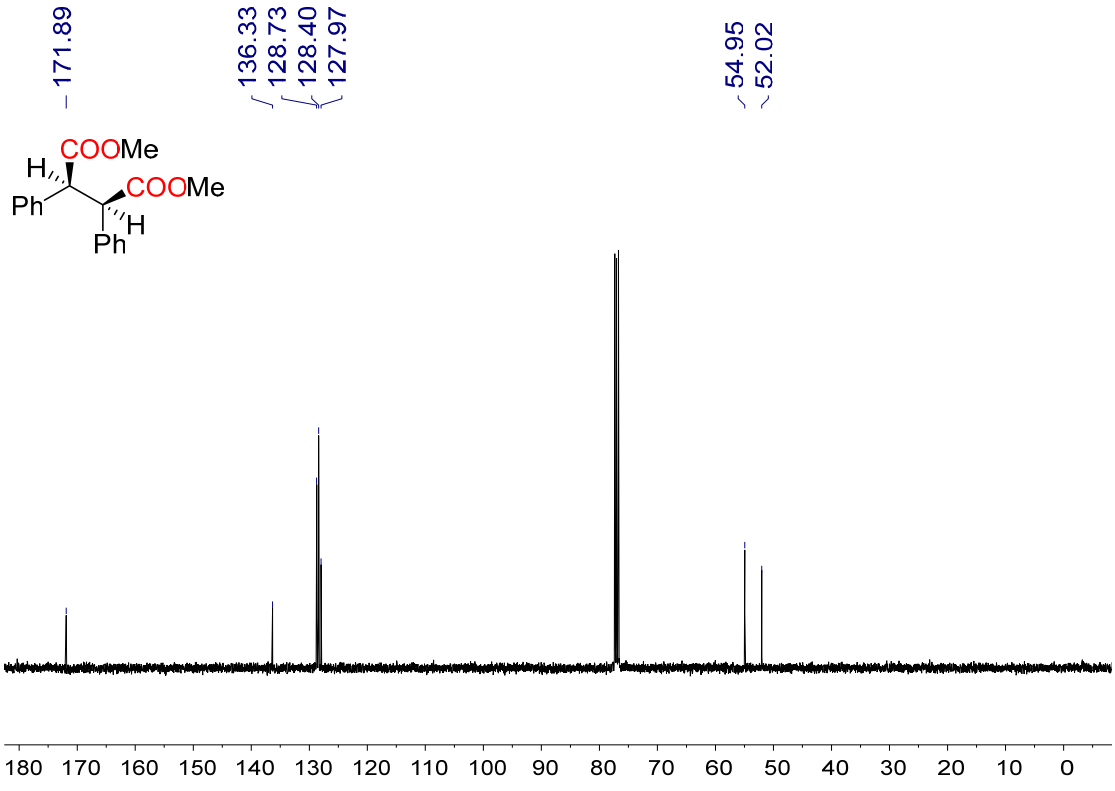
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of *trans*-2l



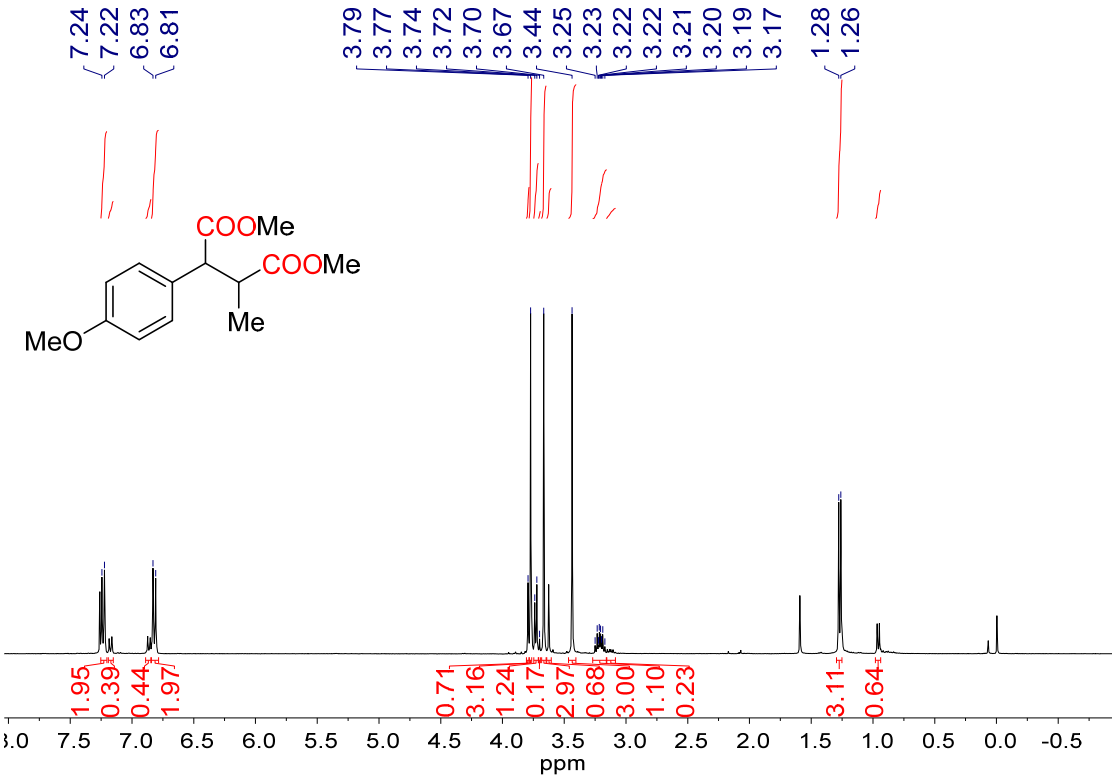
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of *trans*-2l



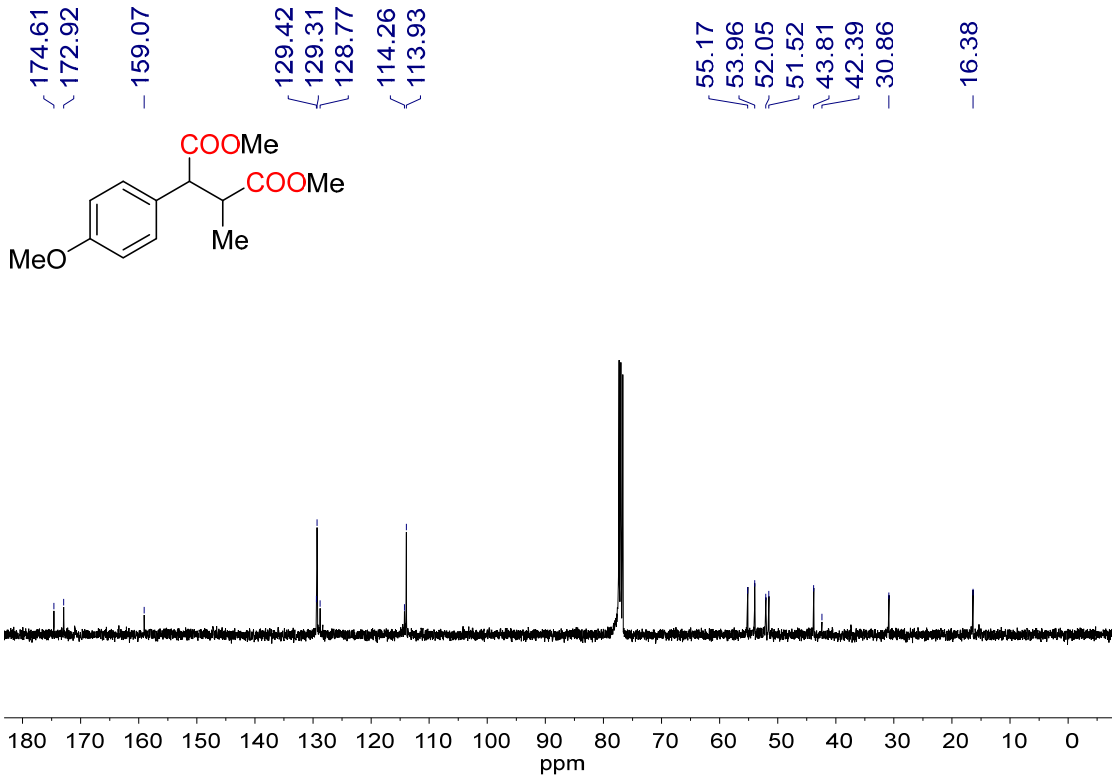
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of *cis*-2l



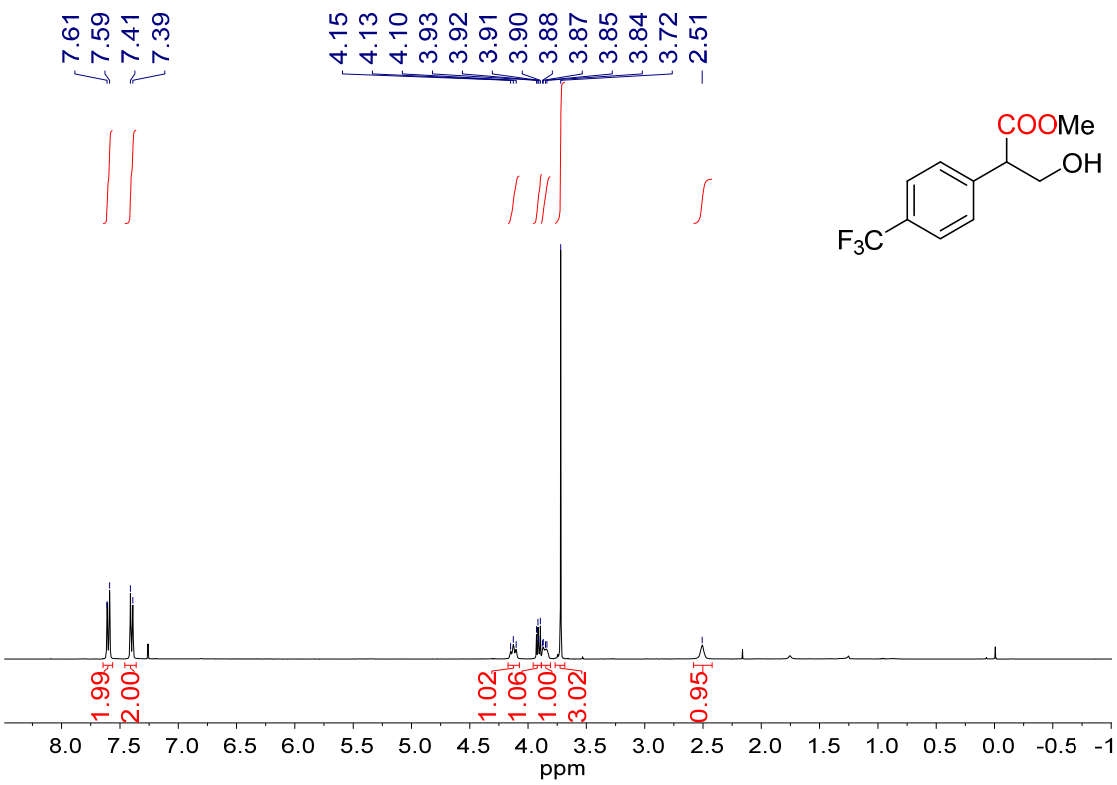
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of *cis*-2l



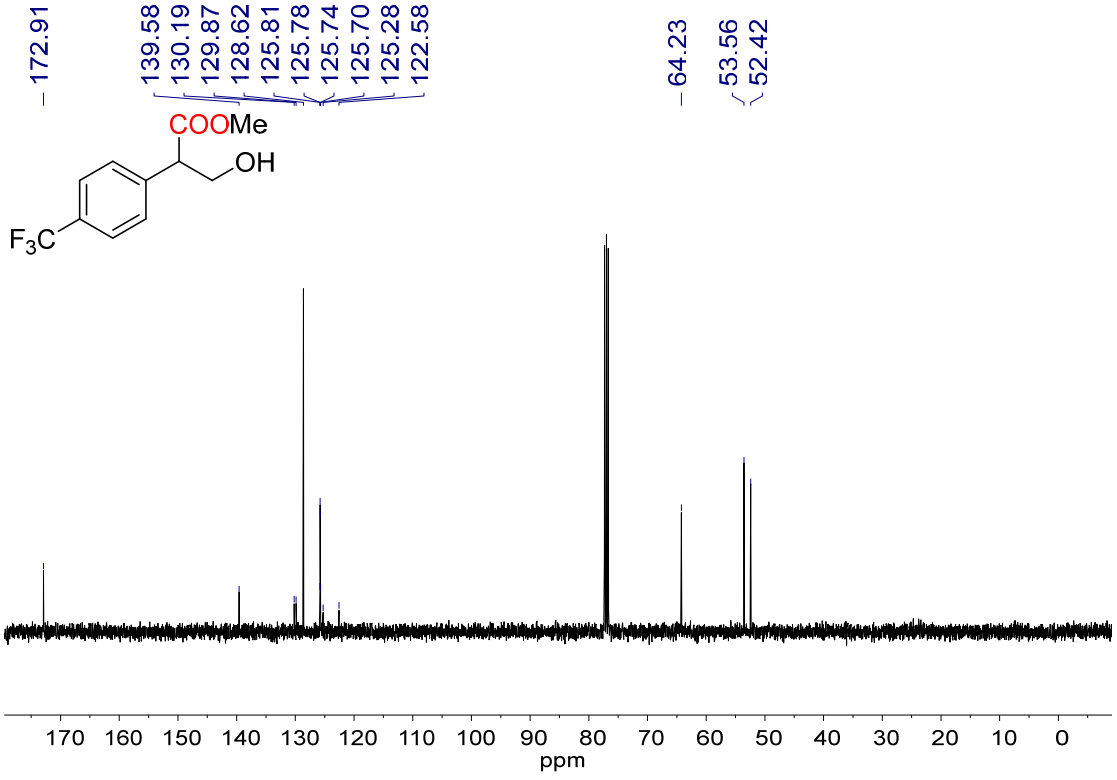
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 2m



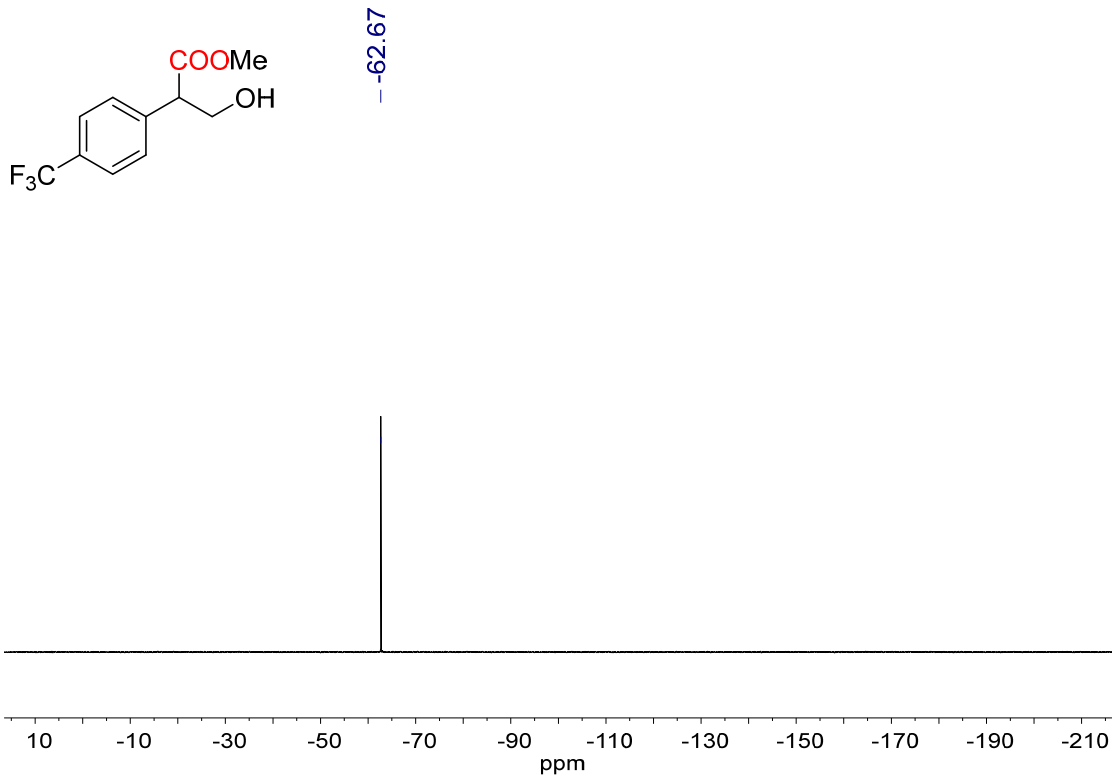
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 2m



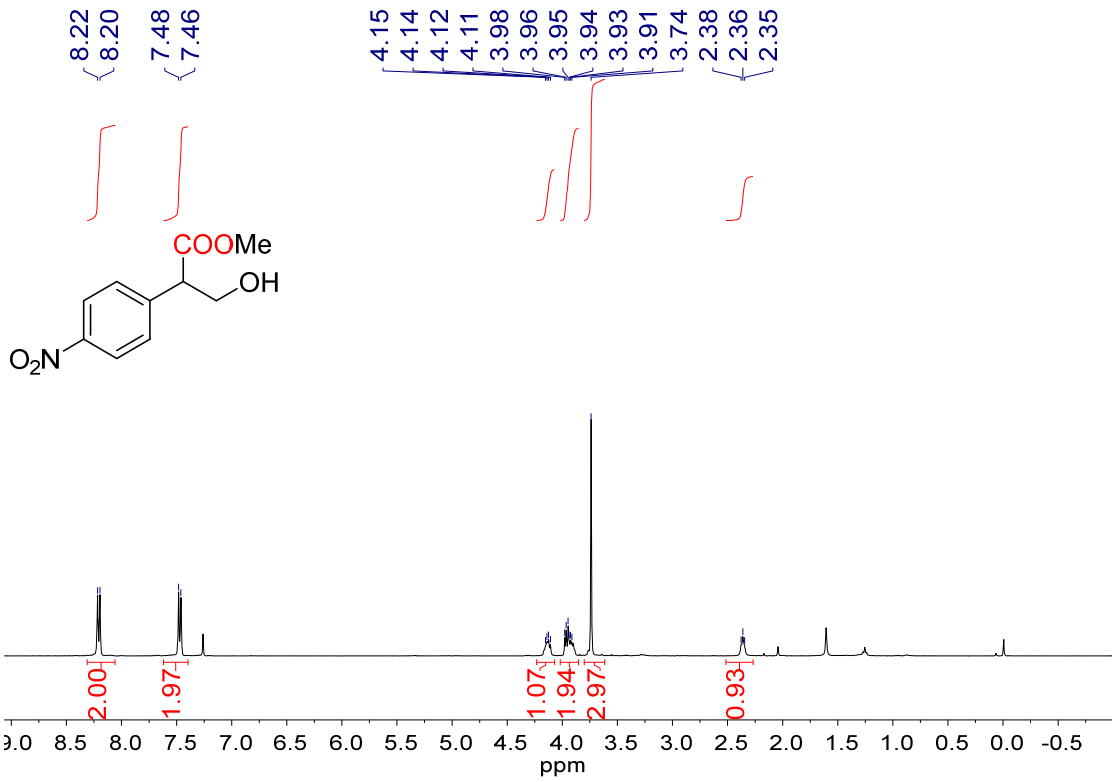
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 3n



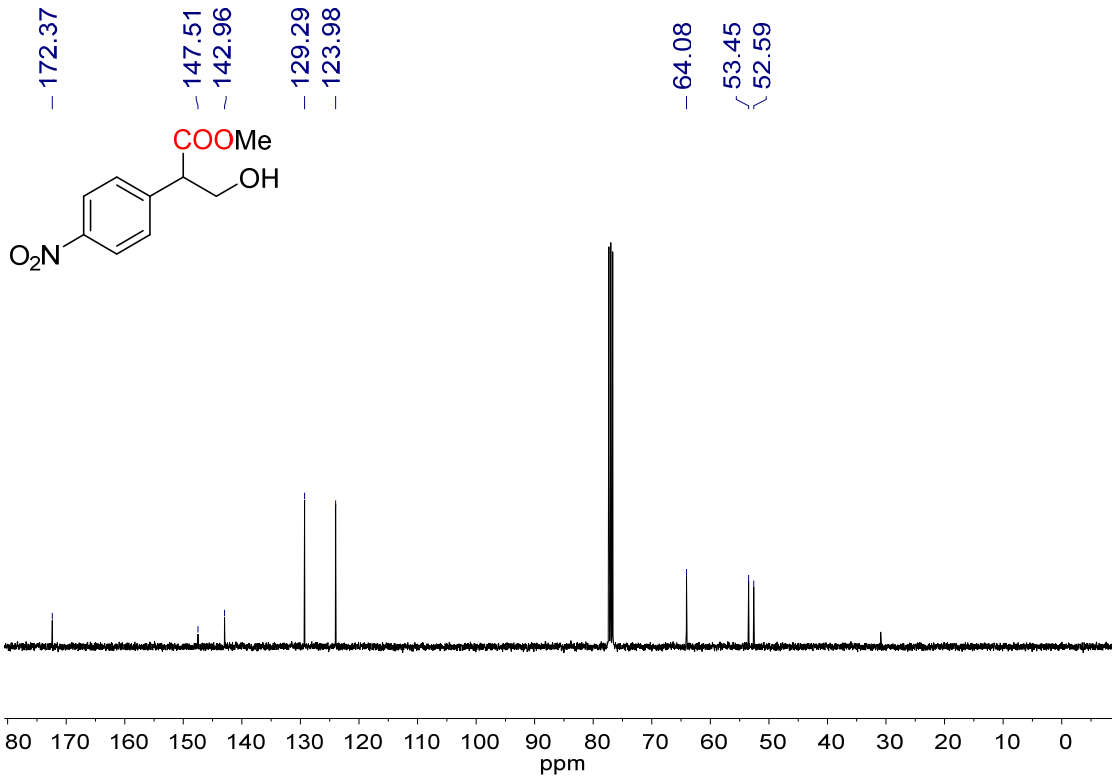
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 3n



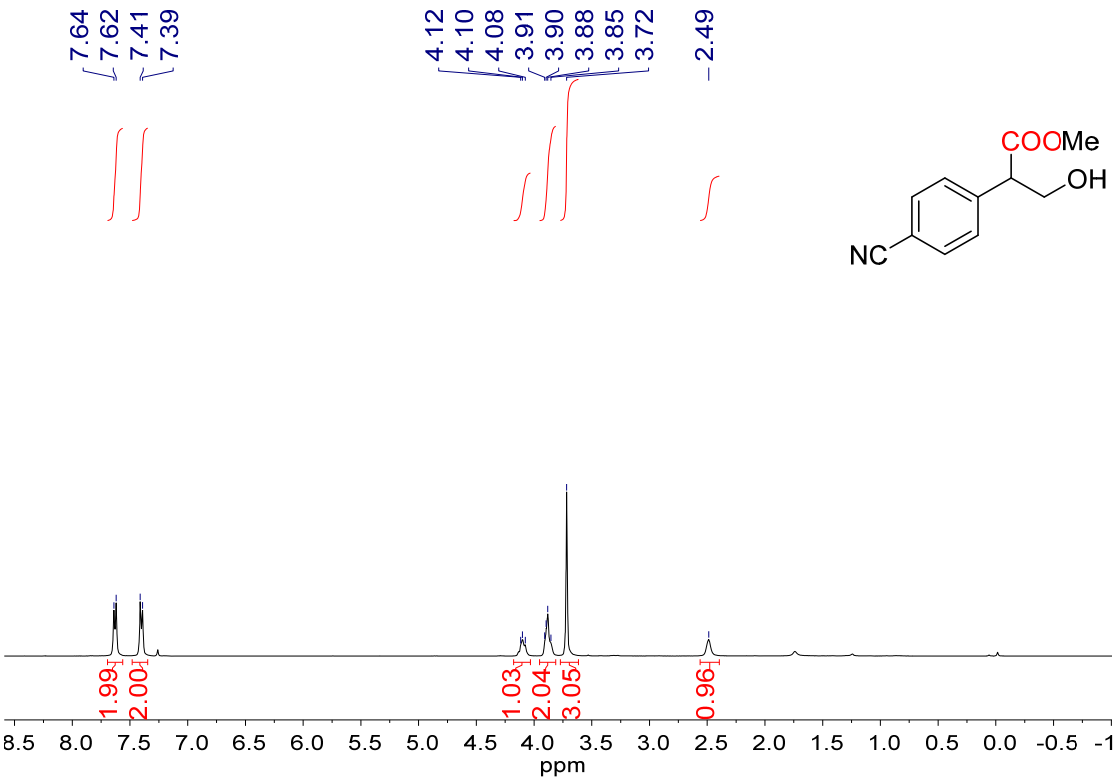
^{19}F NMR spectrum (376 MHz, Chloroform-*d*) of 3n



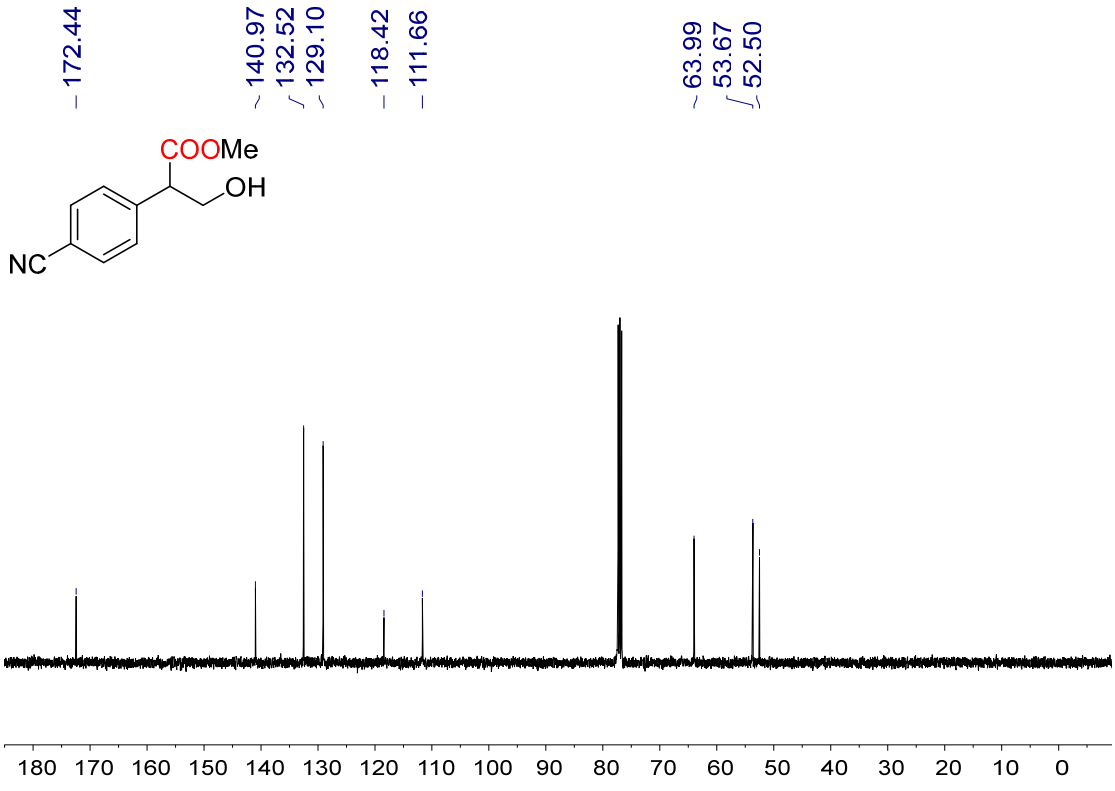
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 3o



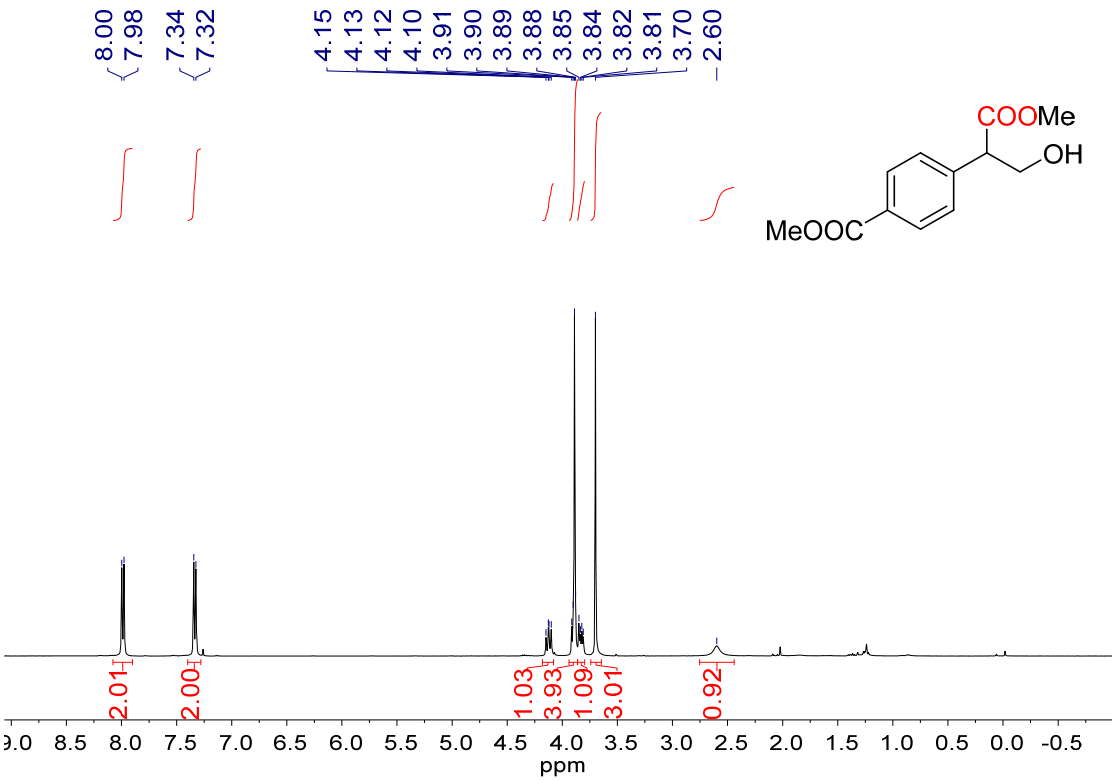
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 3o



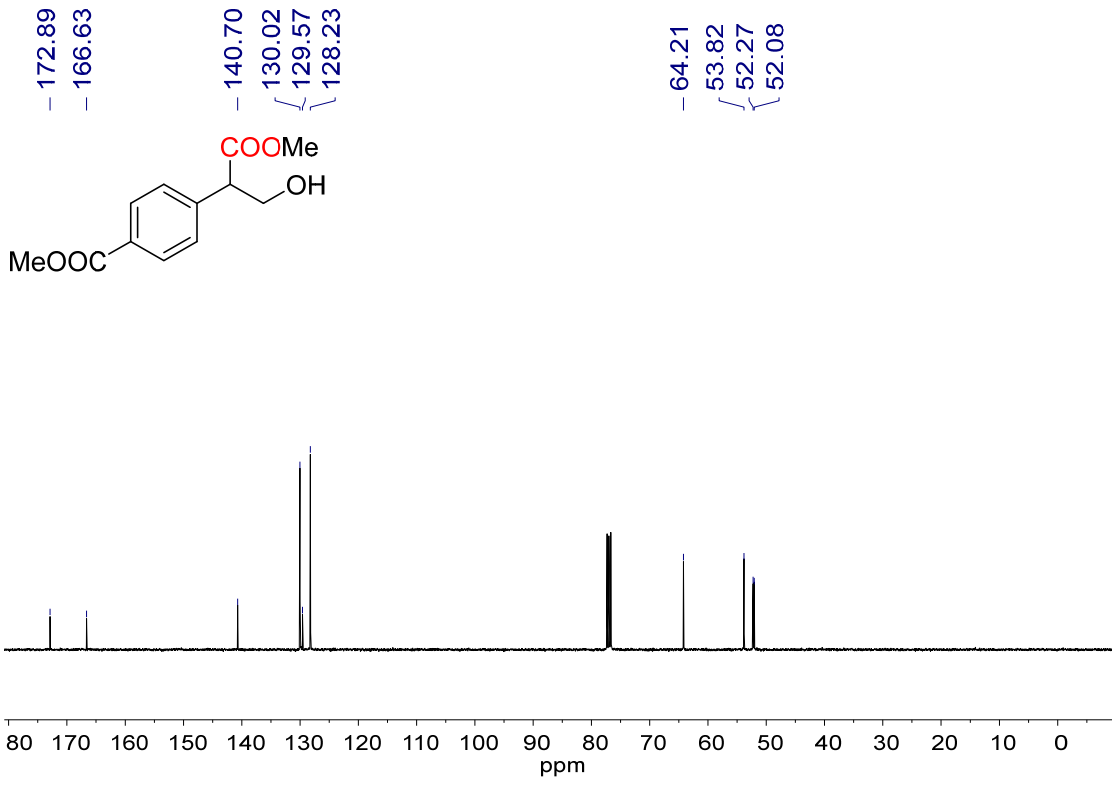
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 3p



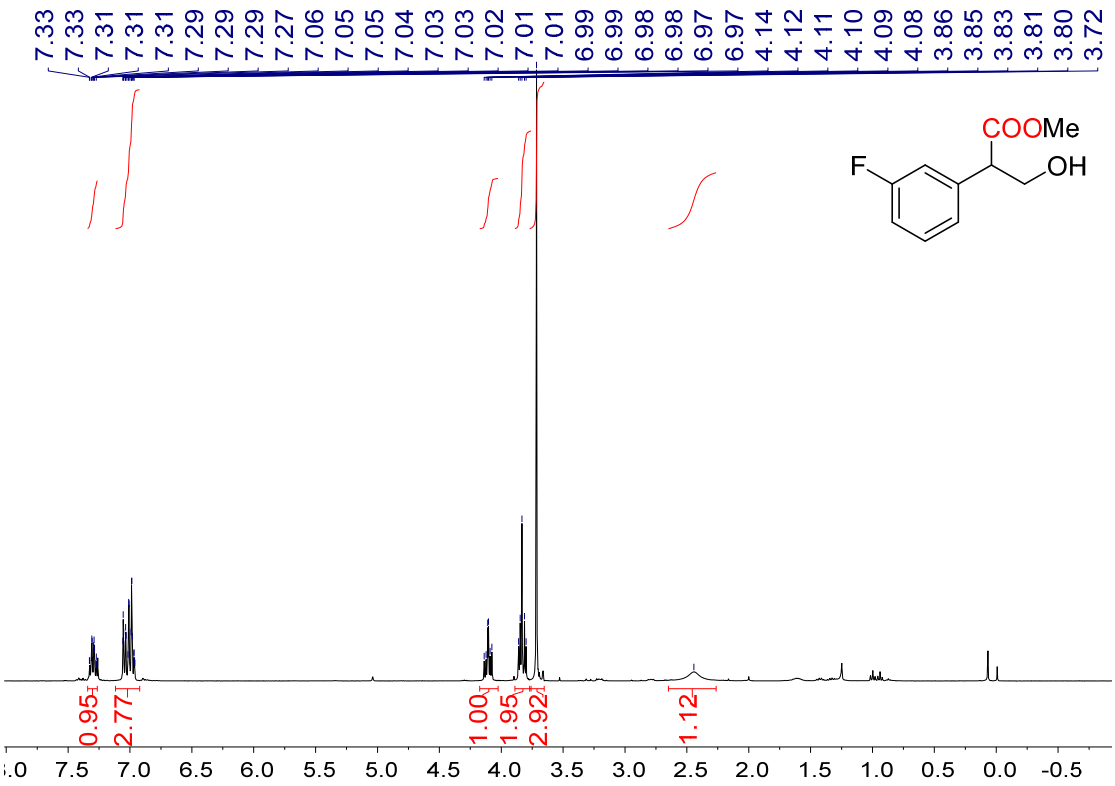
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 3p



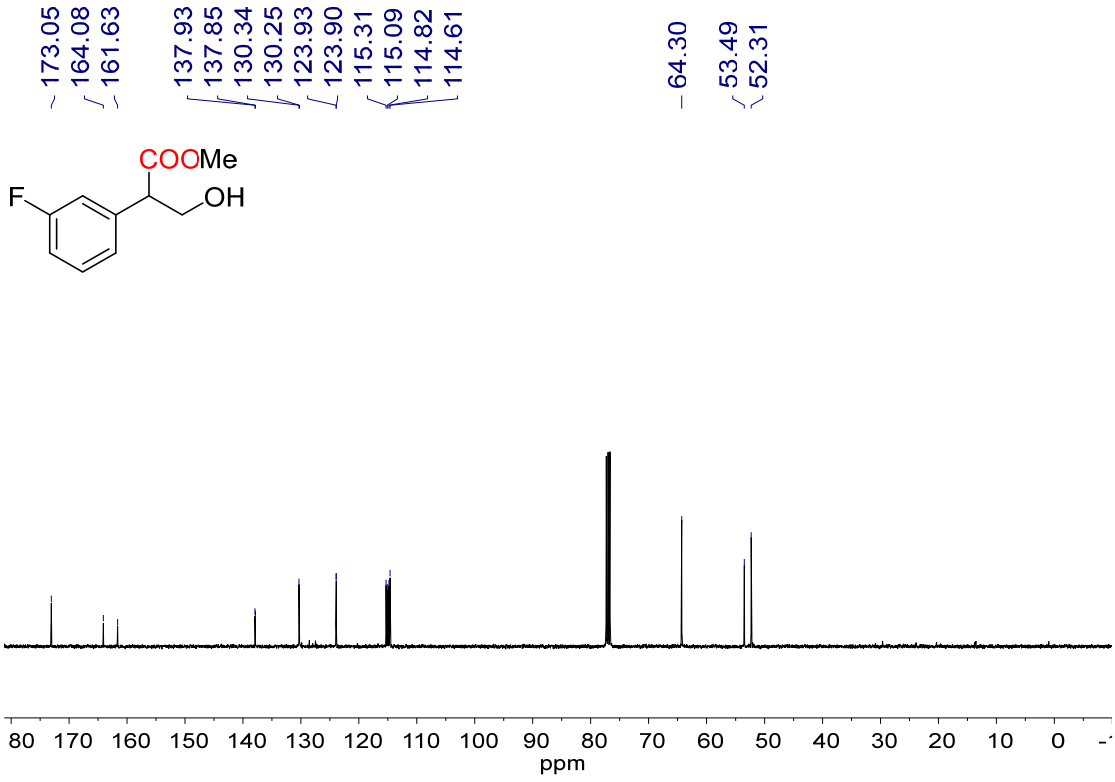
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 3q



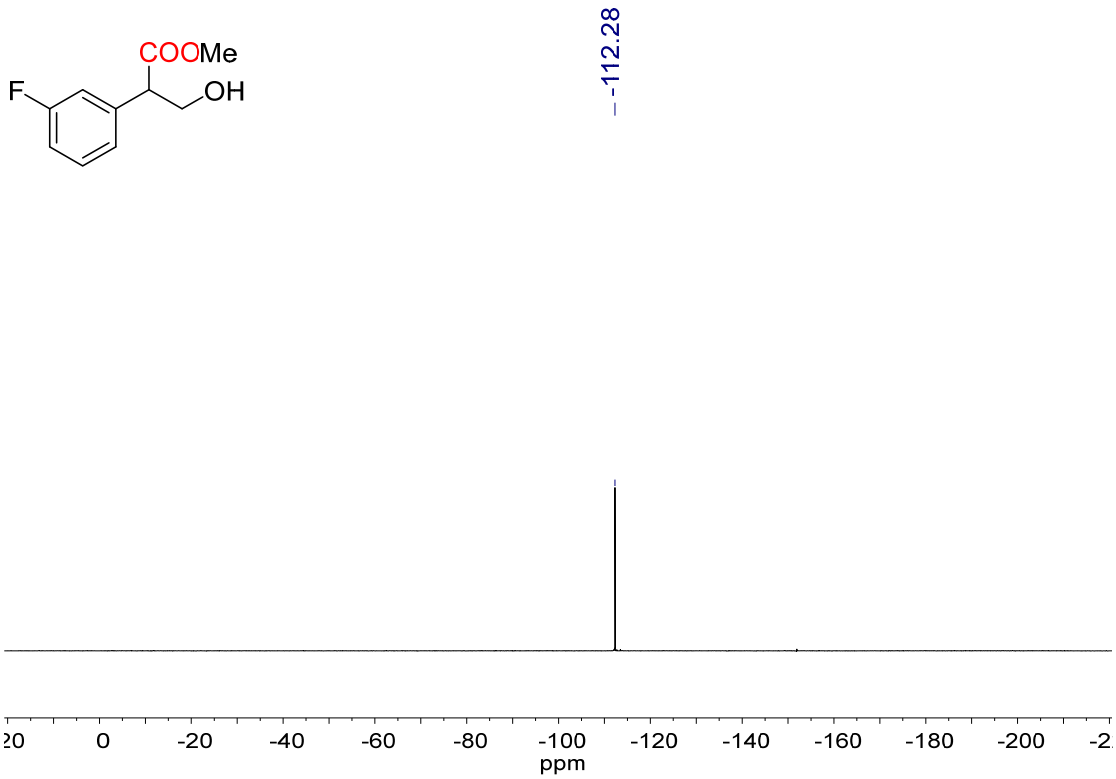
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 3q



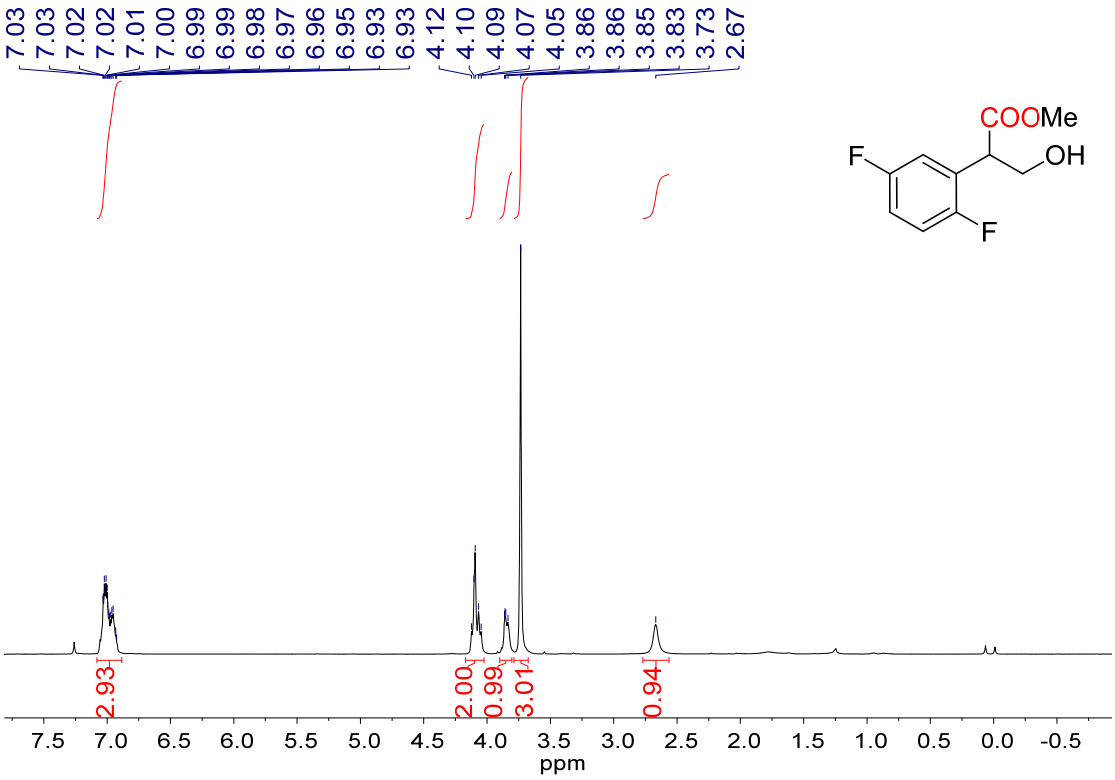
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 3r



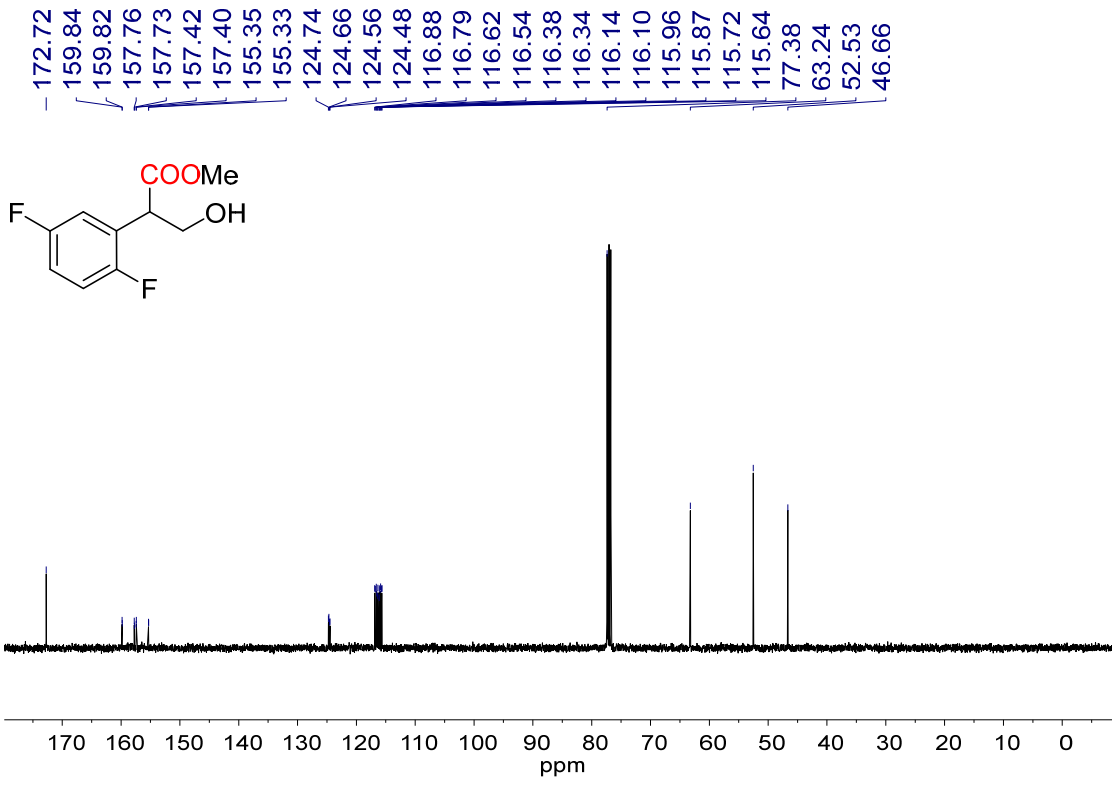
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 3r



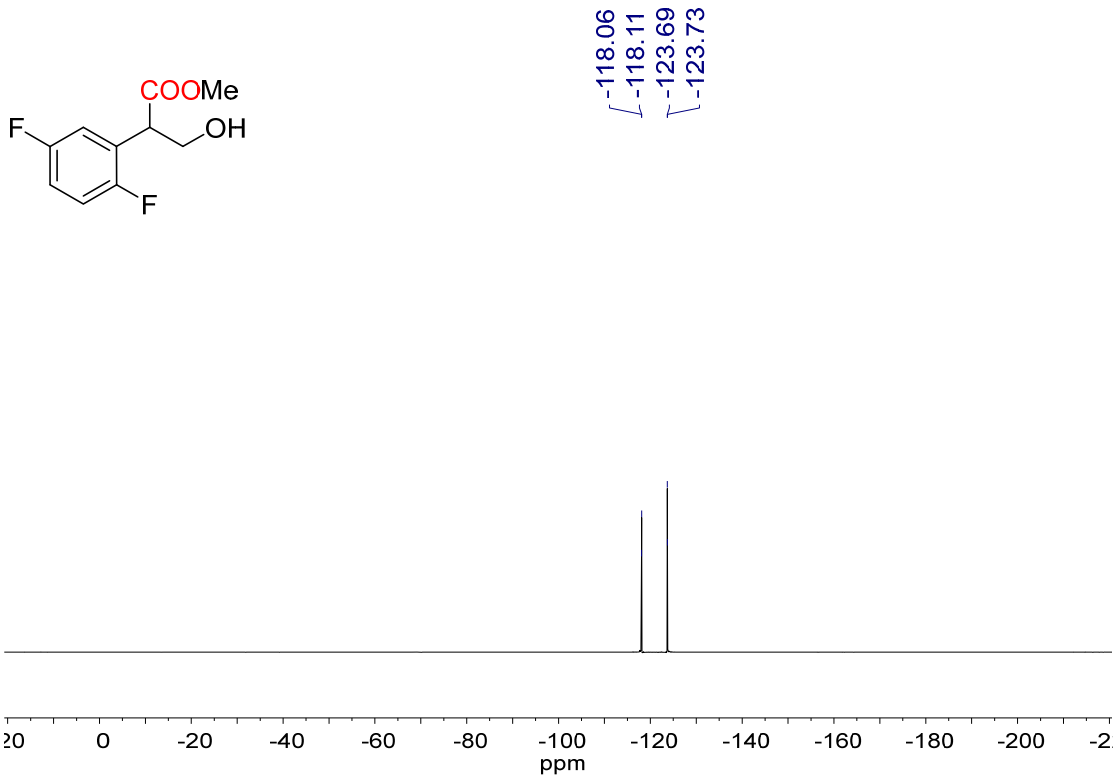
^{19}F NMR spectrum (376 MHz, Chloroform-*d*) of 3r



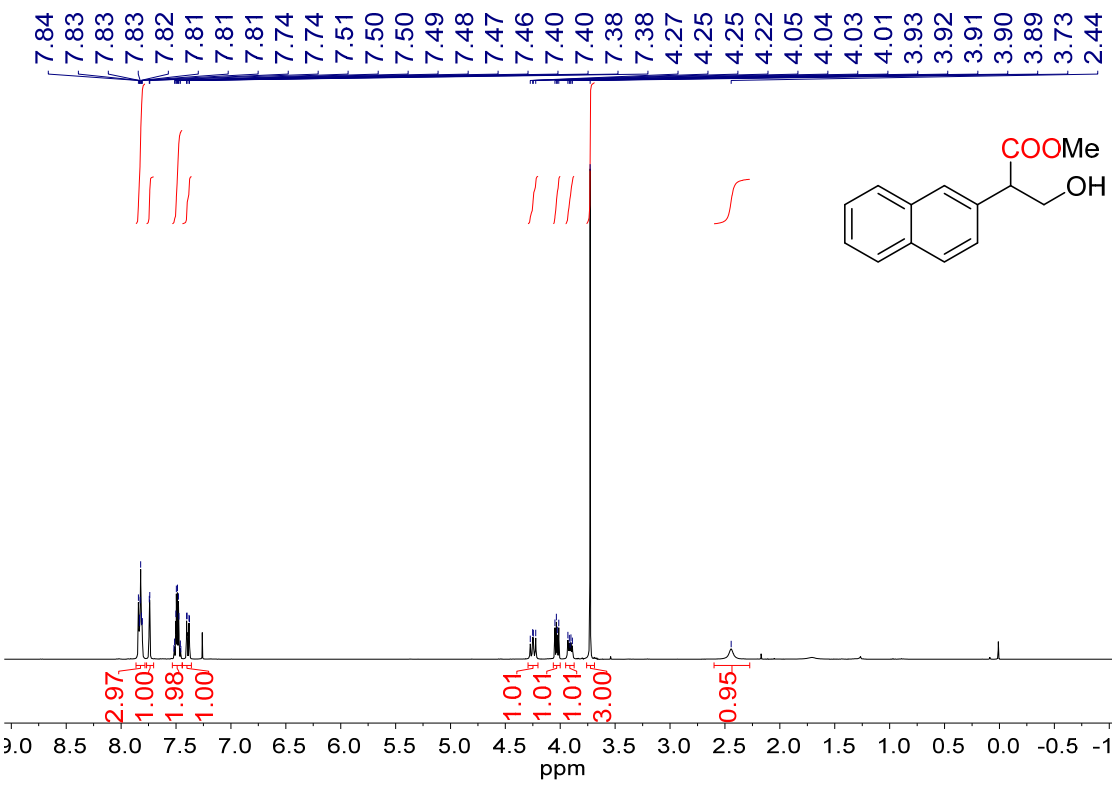
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 3s



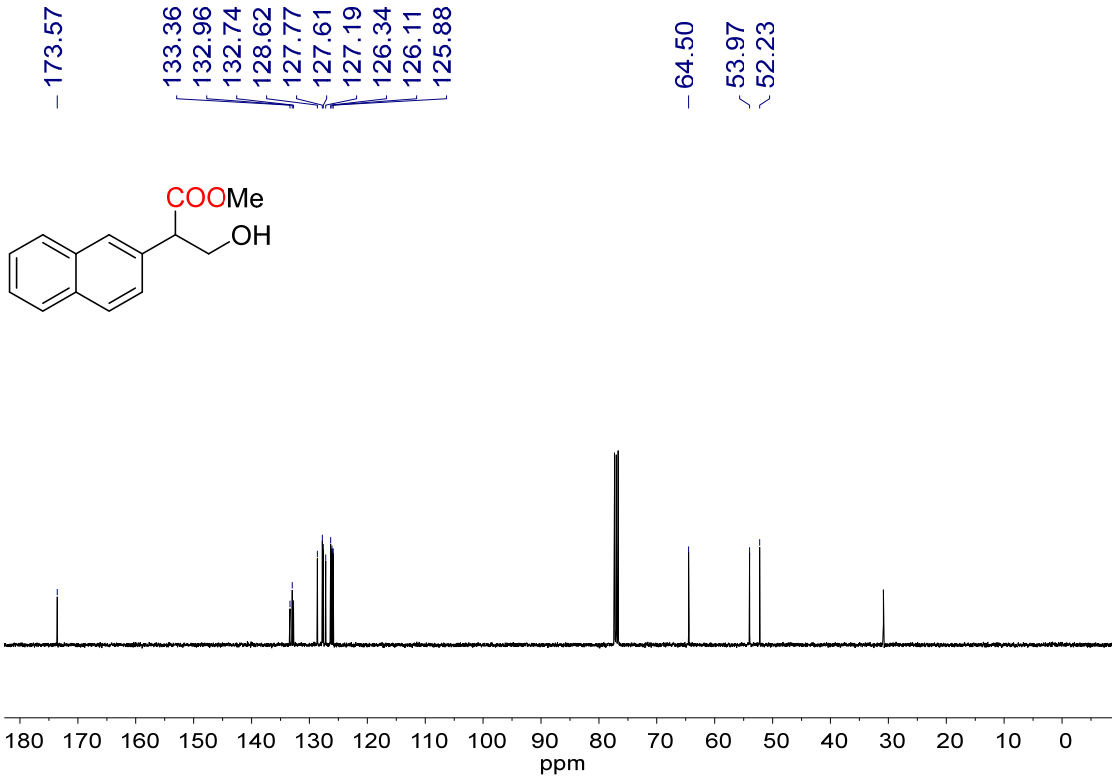
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 3s



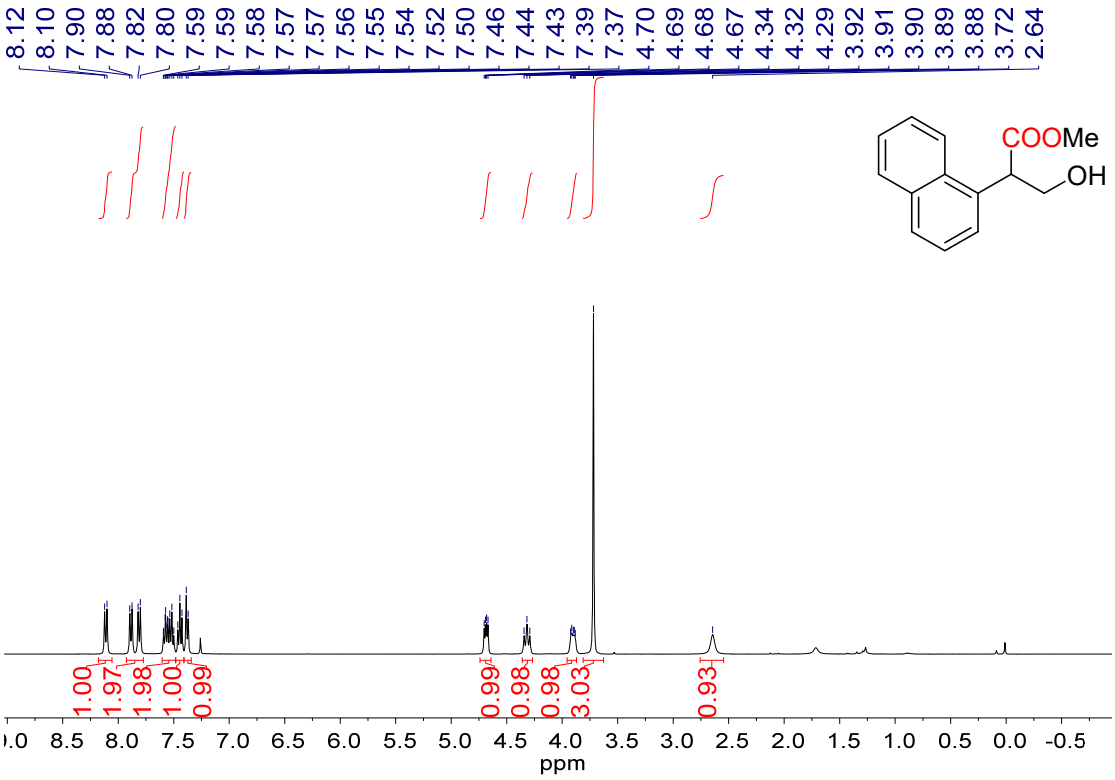
^{19}F NMR spectrum (376 MHz, Chloroform-*d*) of 3s



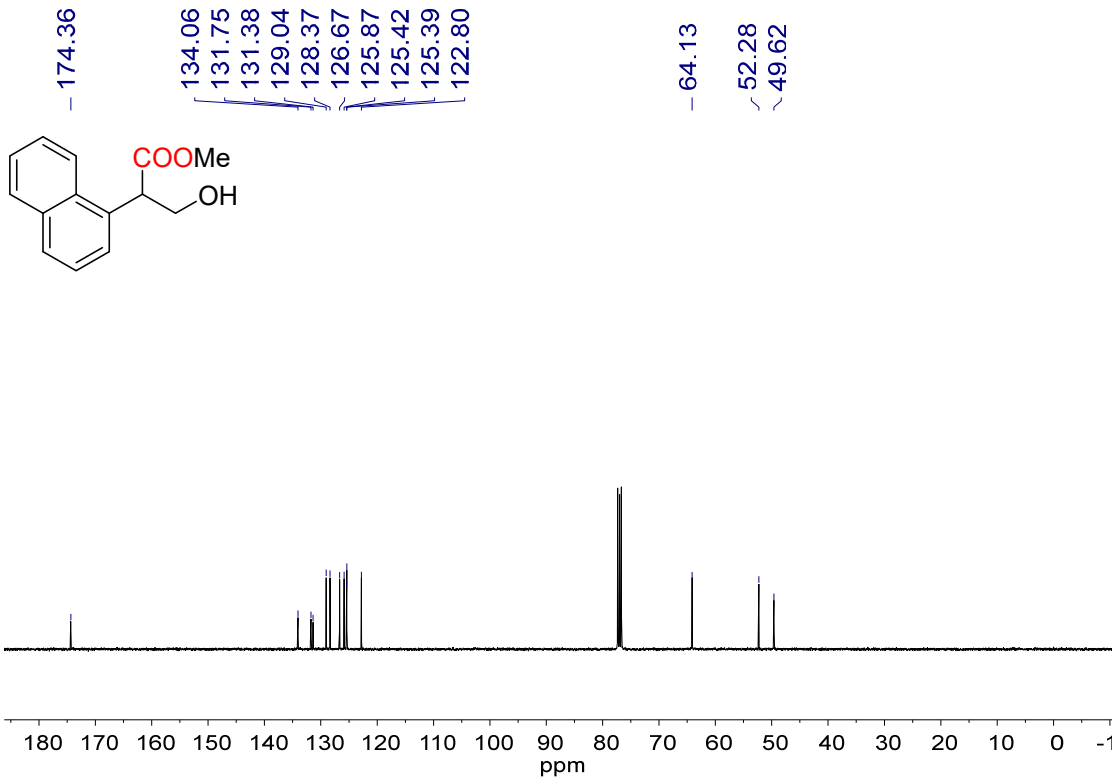
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 3t



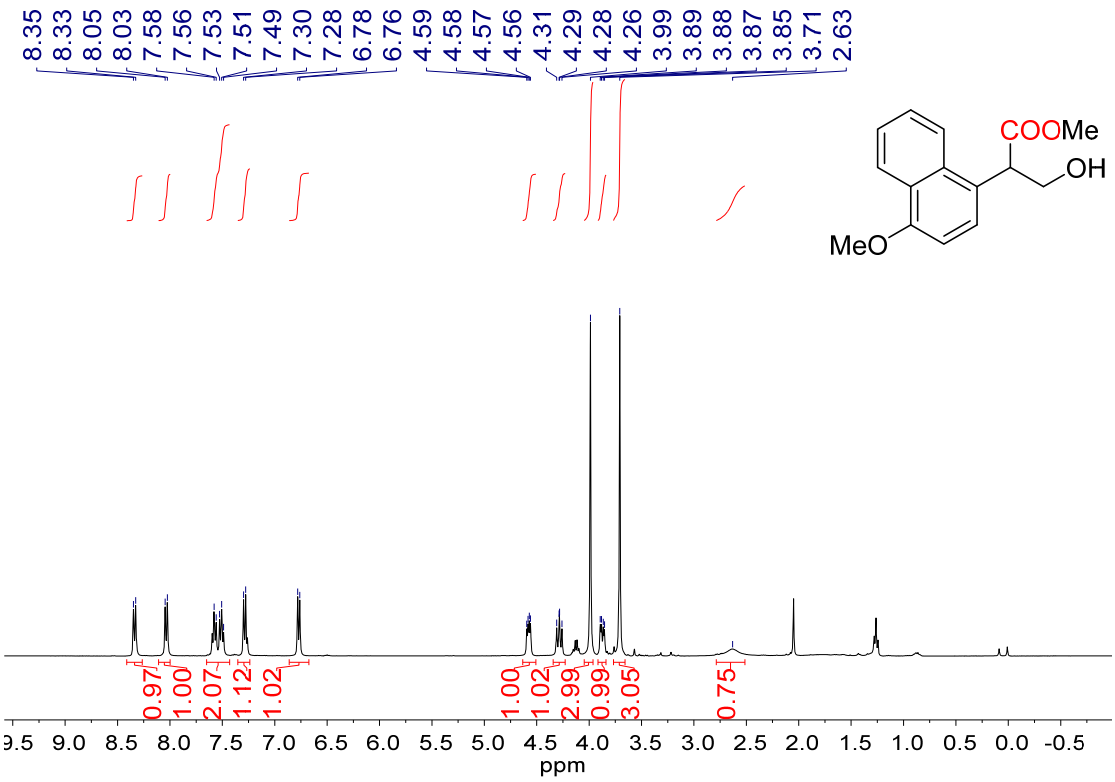
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 3t



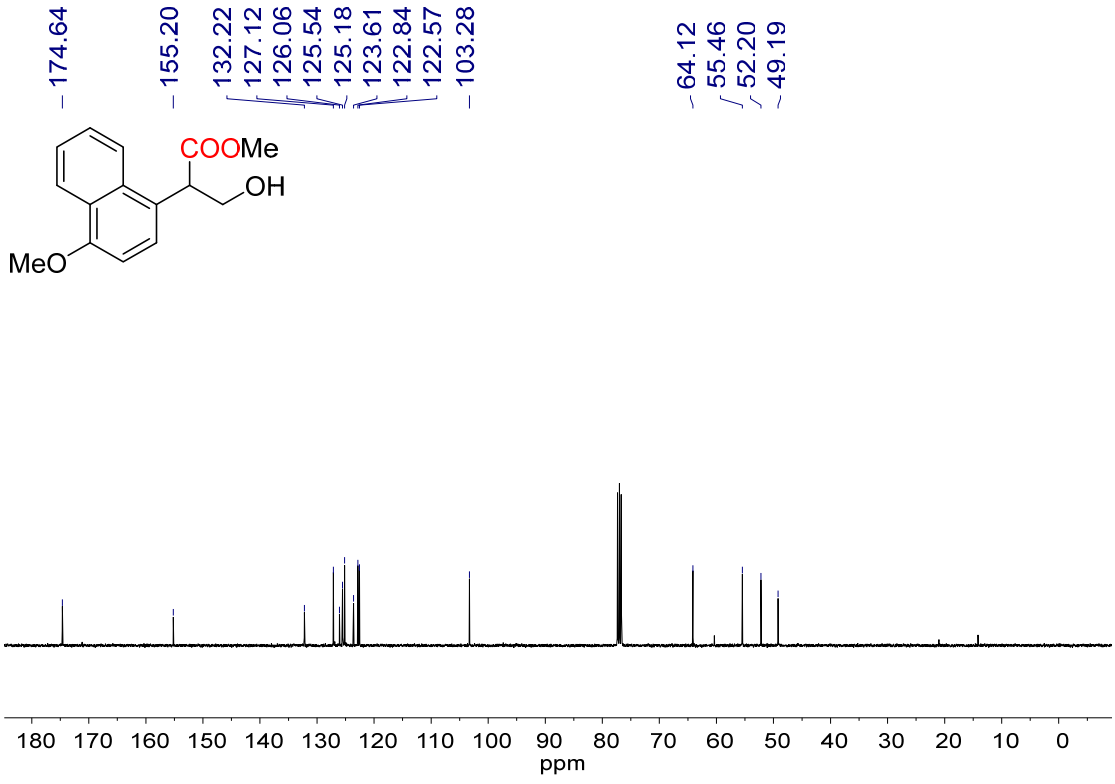
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 3u



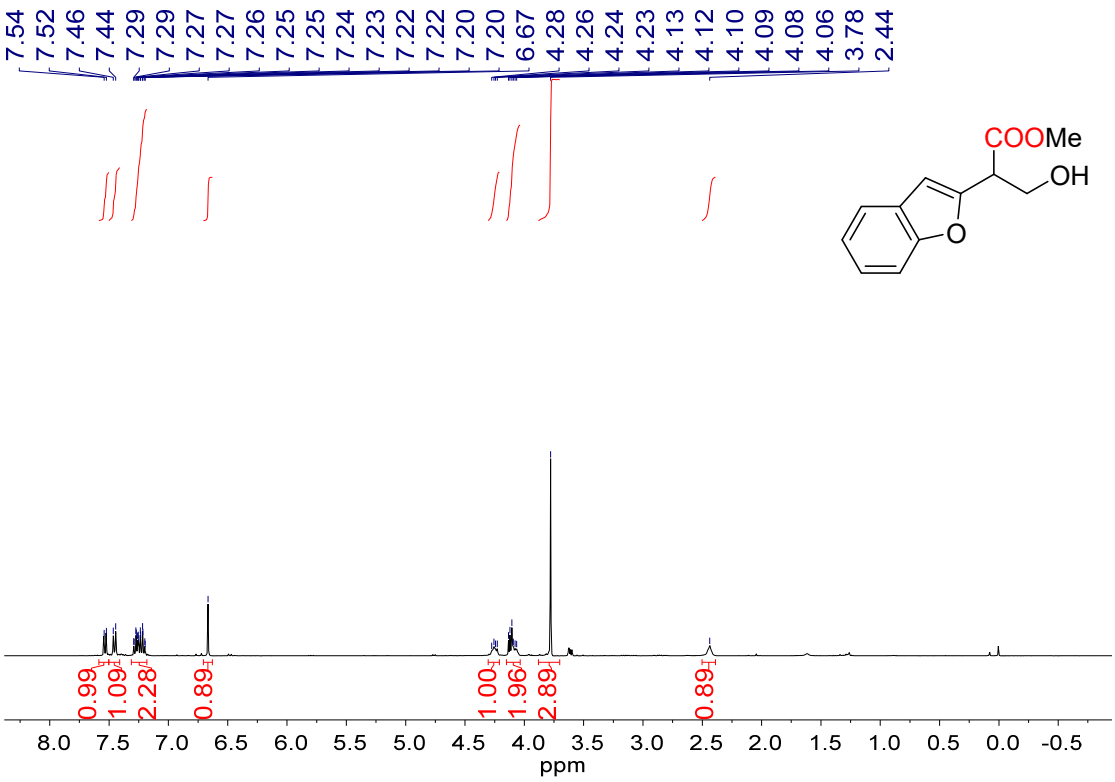
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 3u



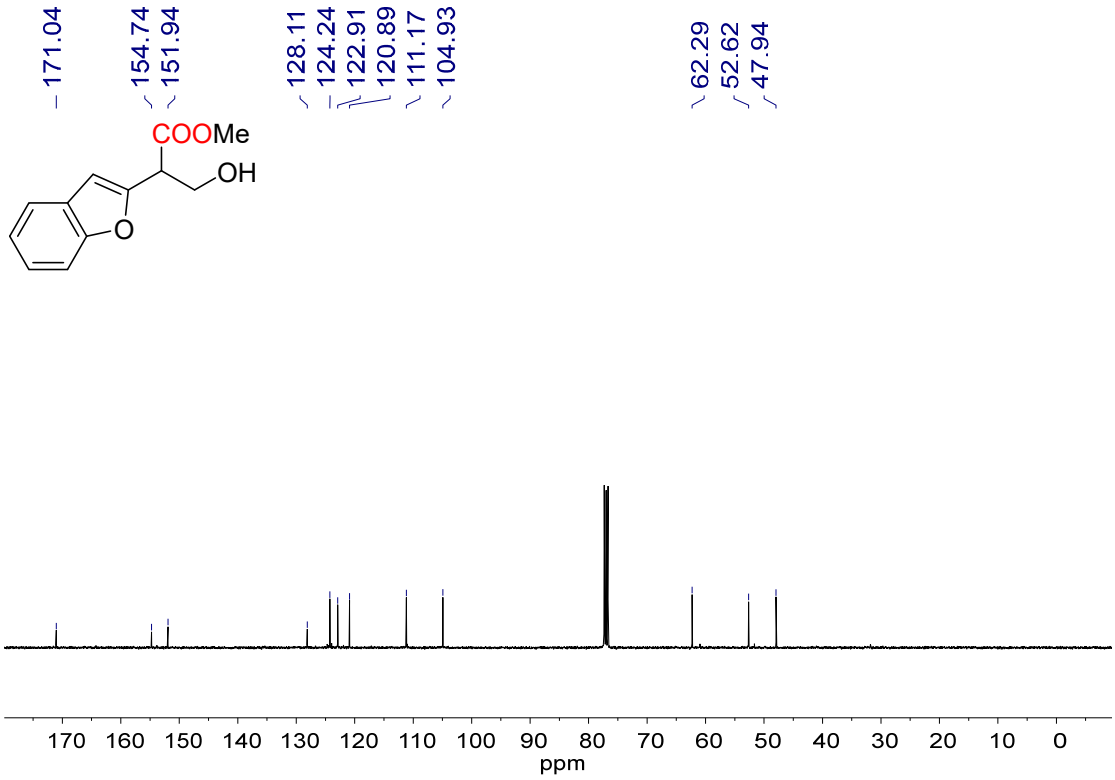
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 3v



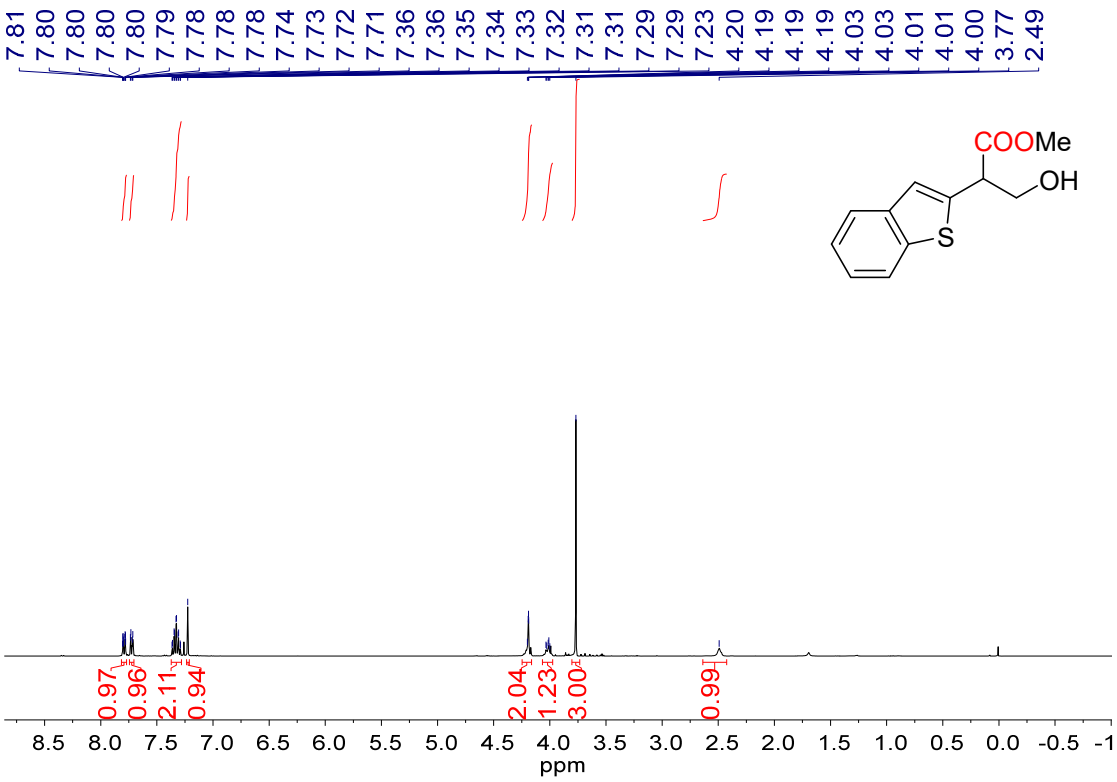
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 3v



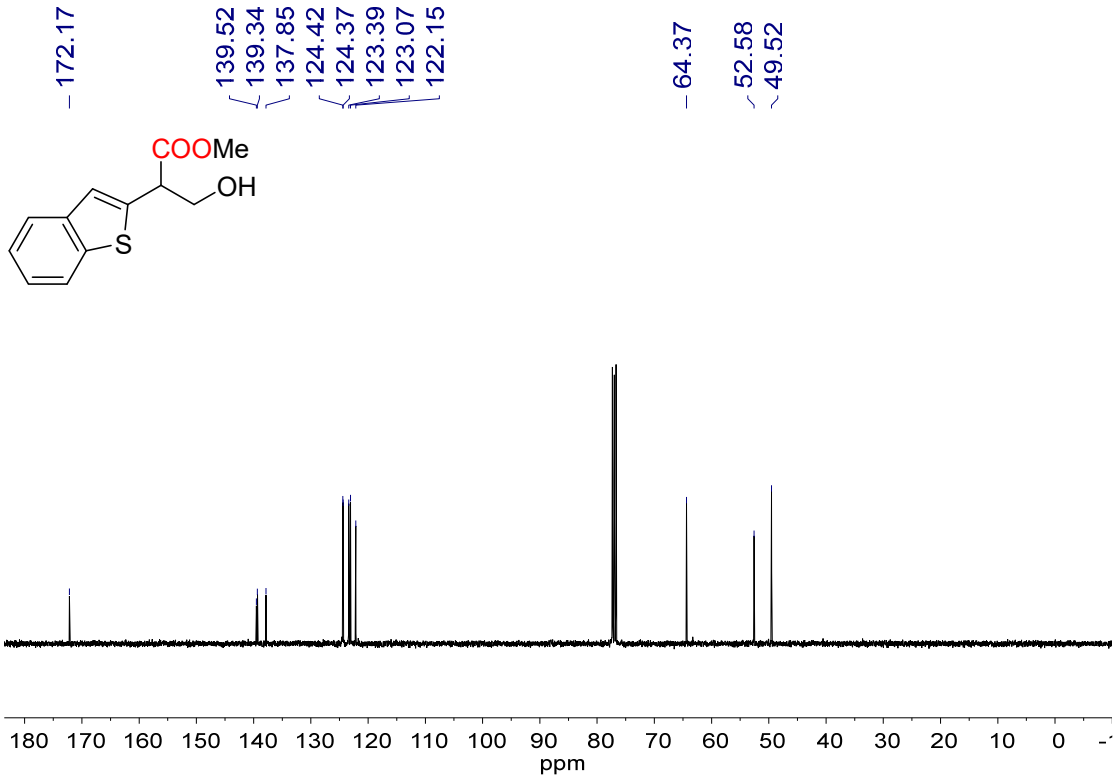
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 3w



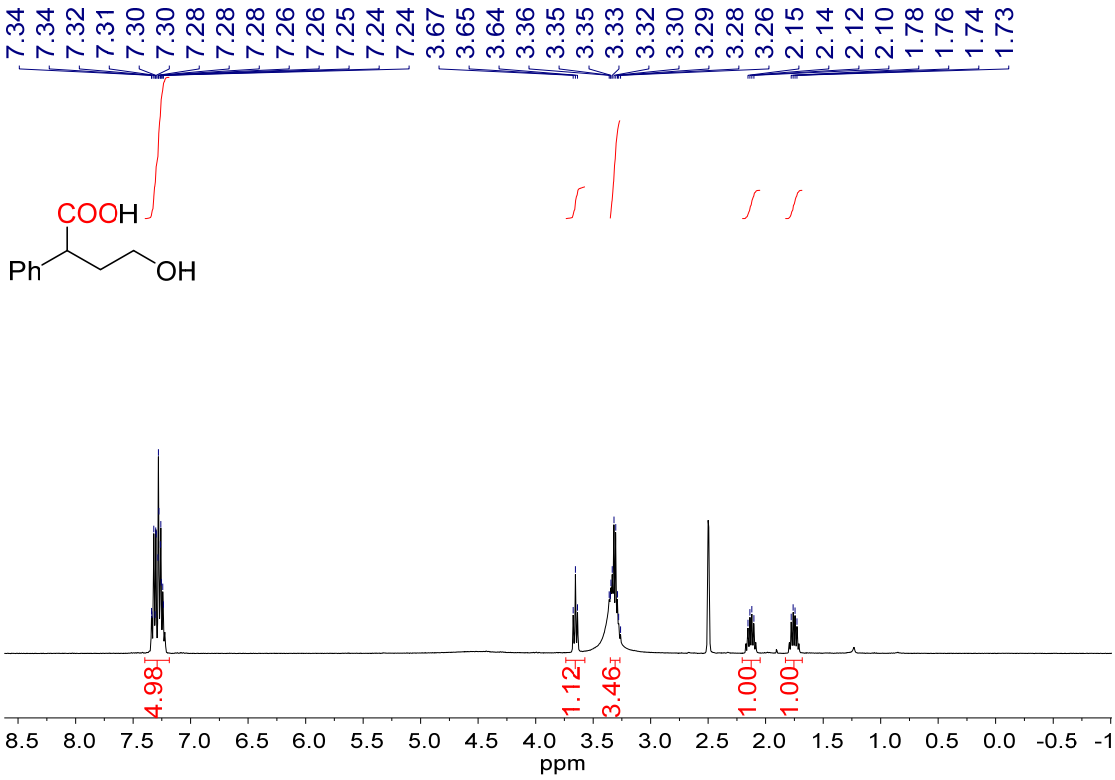
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 3w



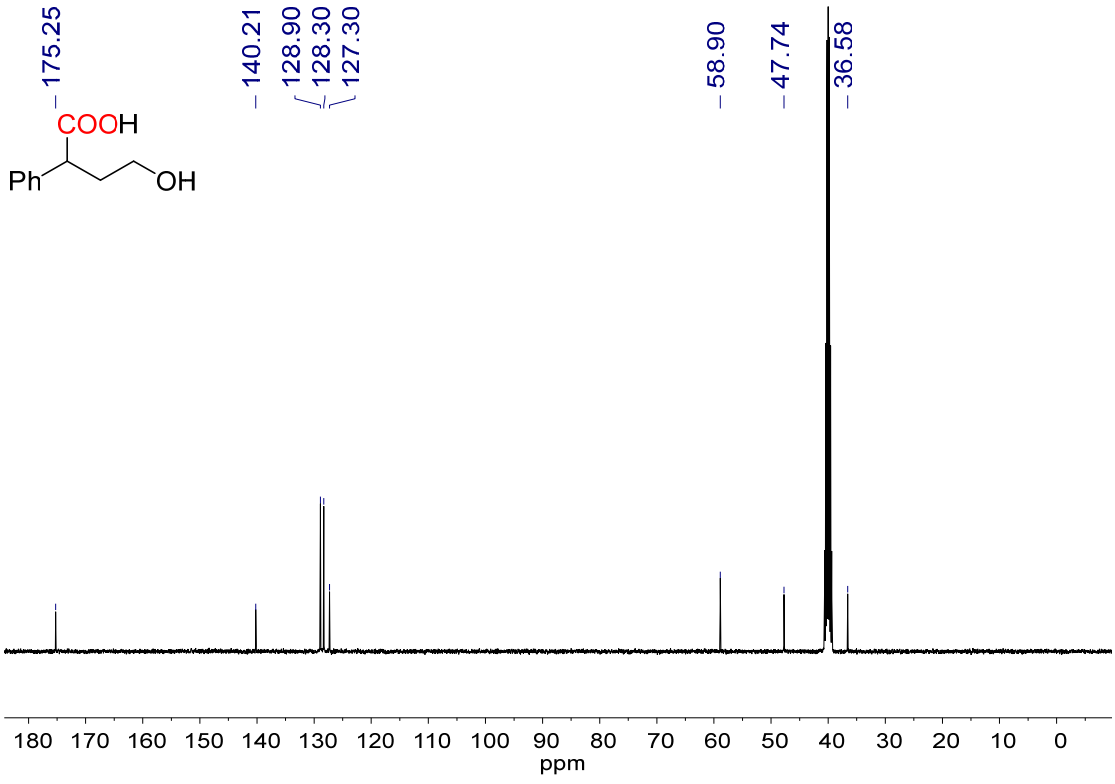
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 3x



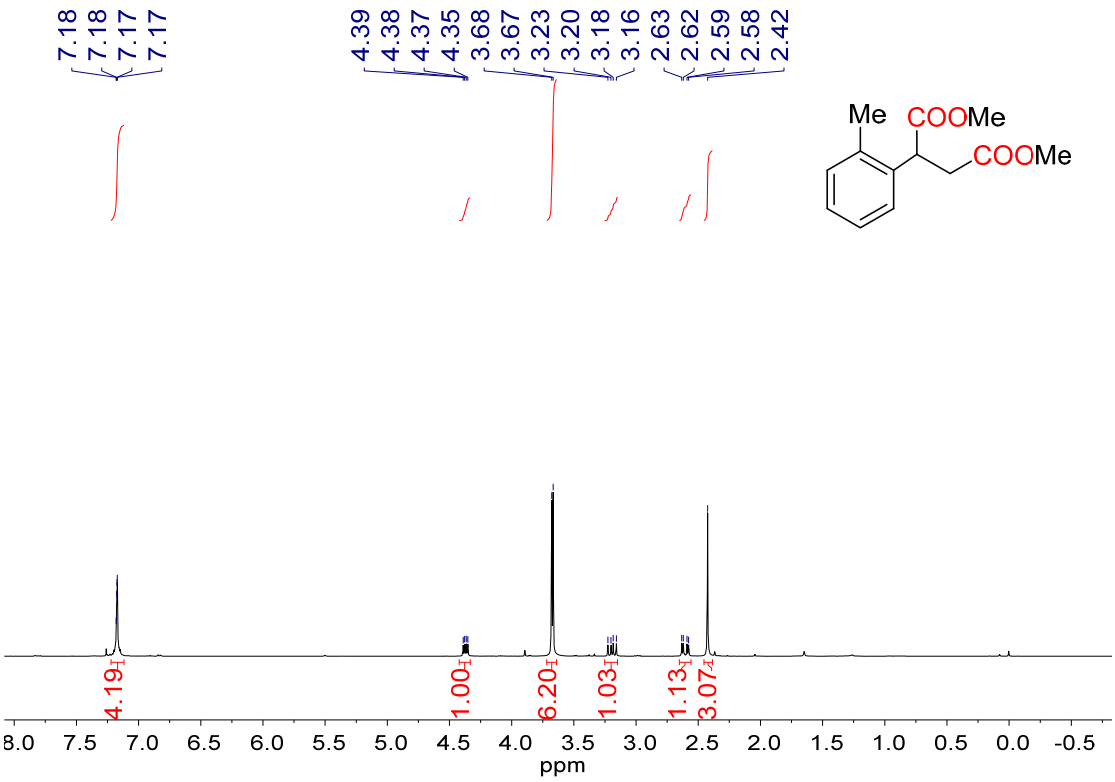
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 3x



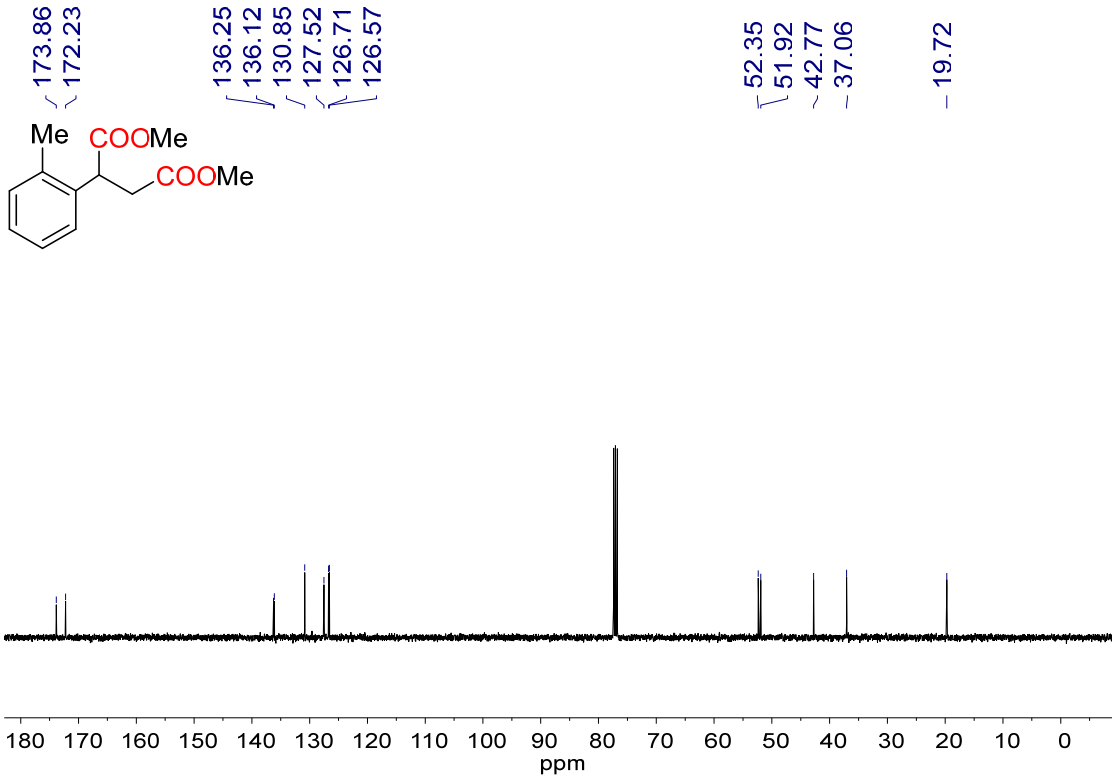
^{1}H NMR spectrum (400 MHz, DMSO-d_6) of 3y



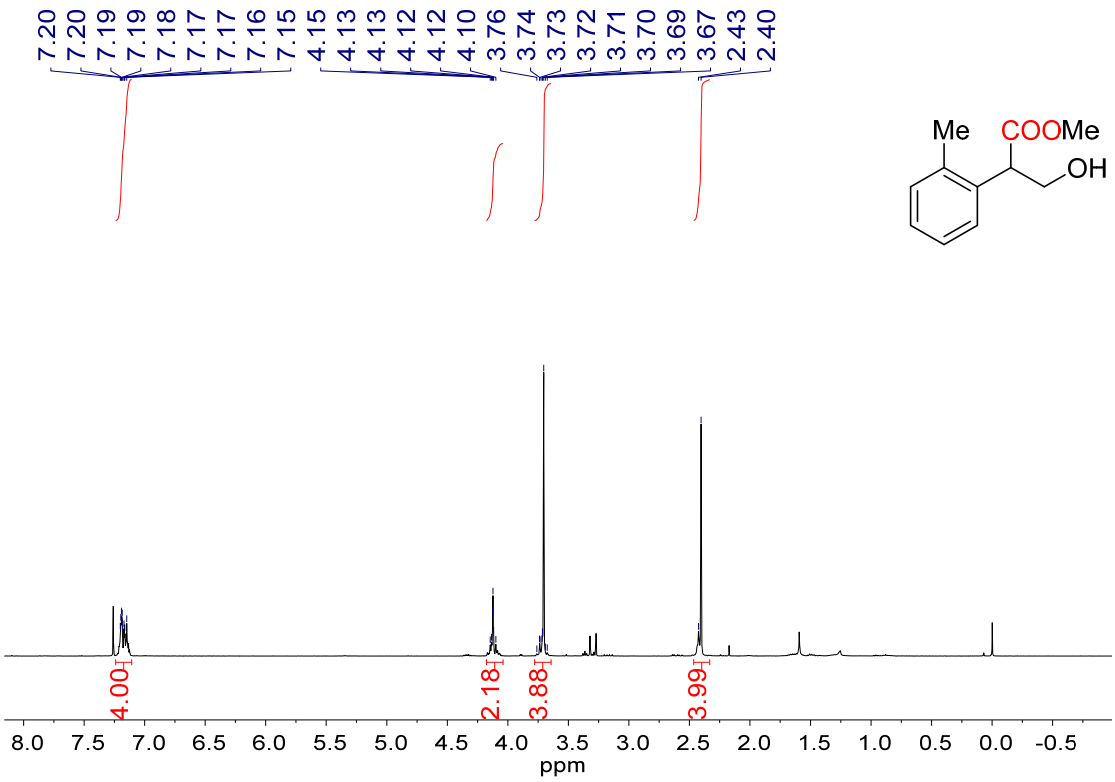
^{13}C{^{1}H} NMR spectrum (101 MHz, DMSO-d_6) of 3y



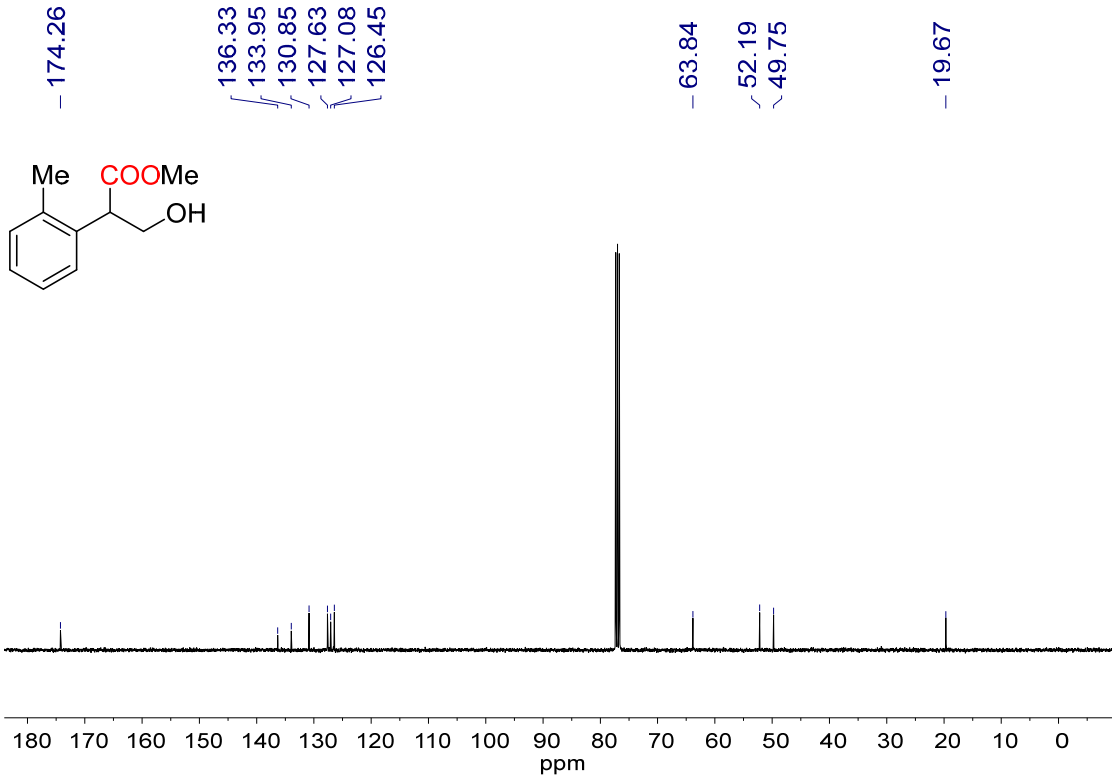
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 2z



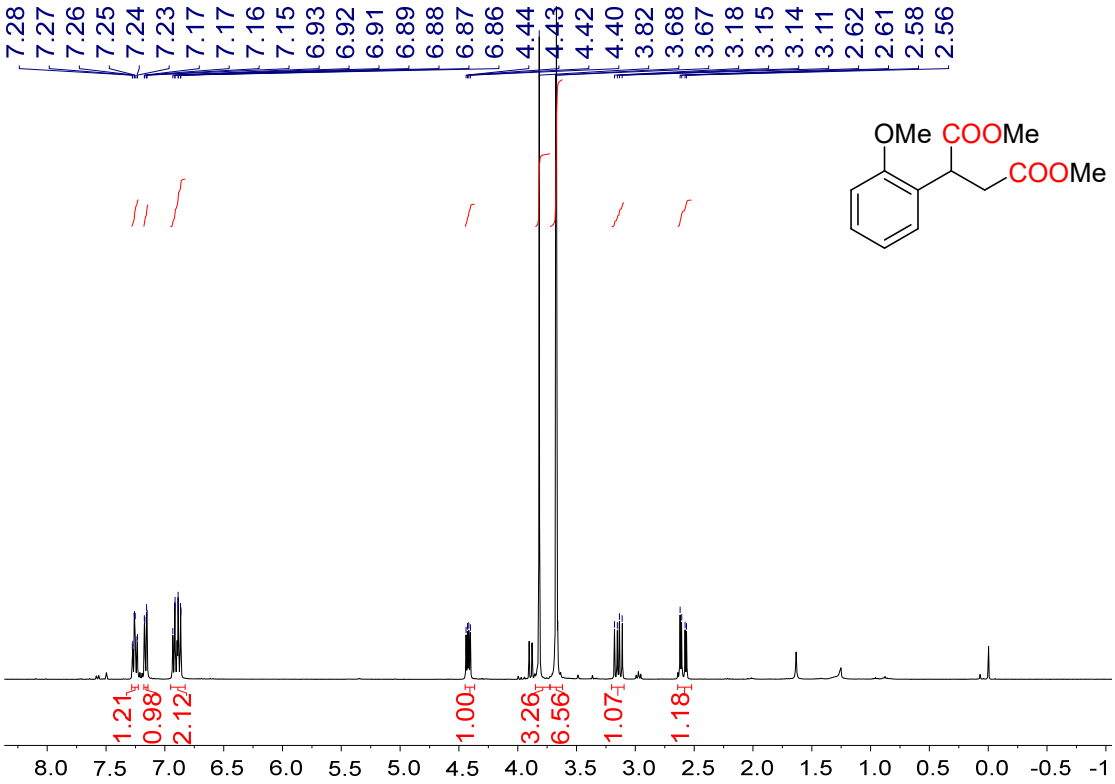
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 2z



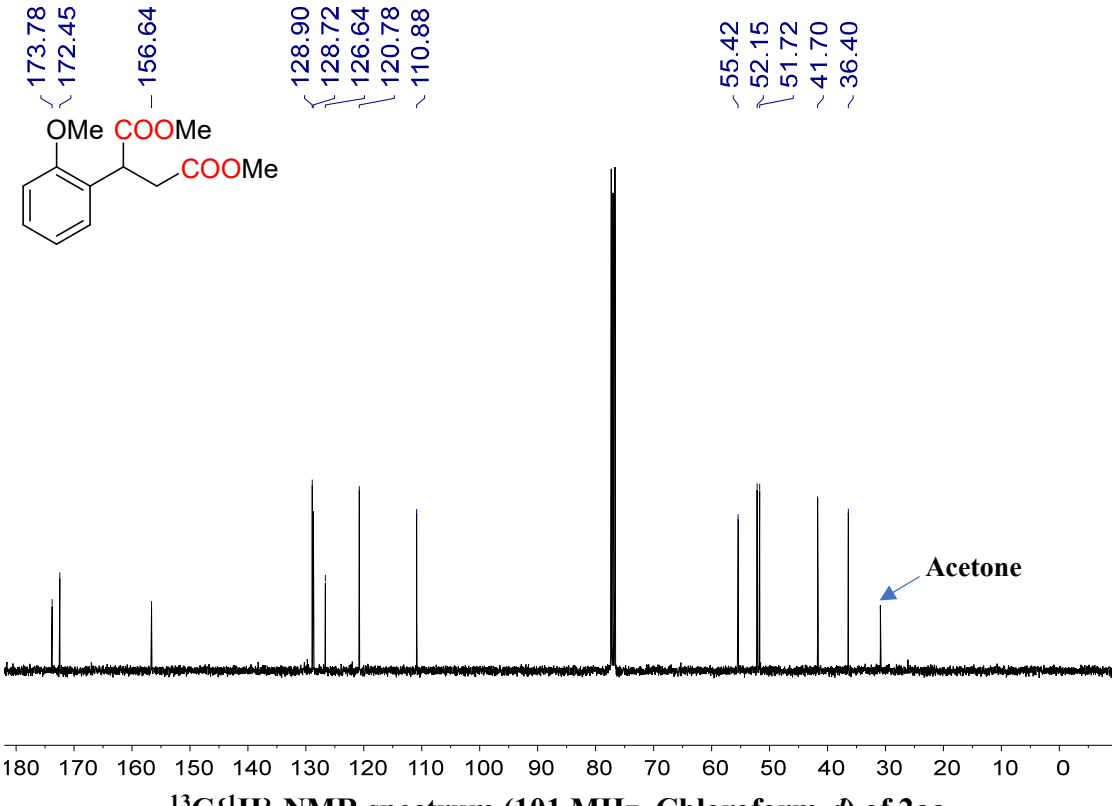
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 3z



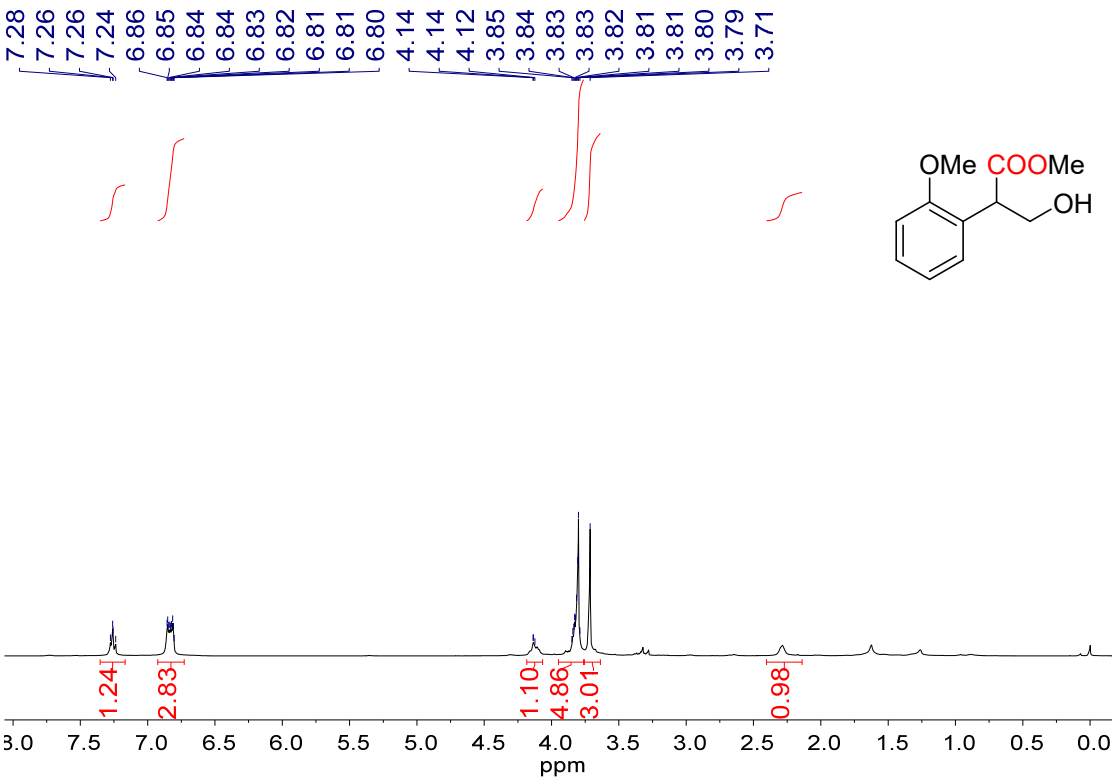
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 3z



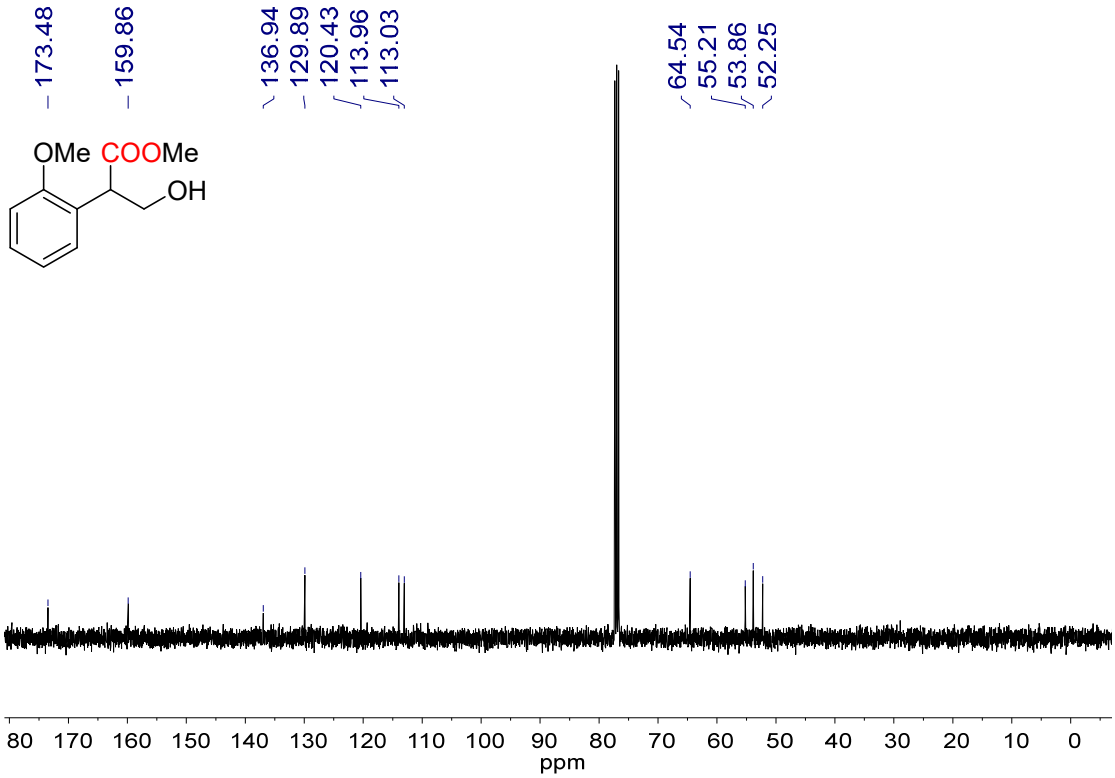
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 2aa



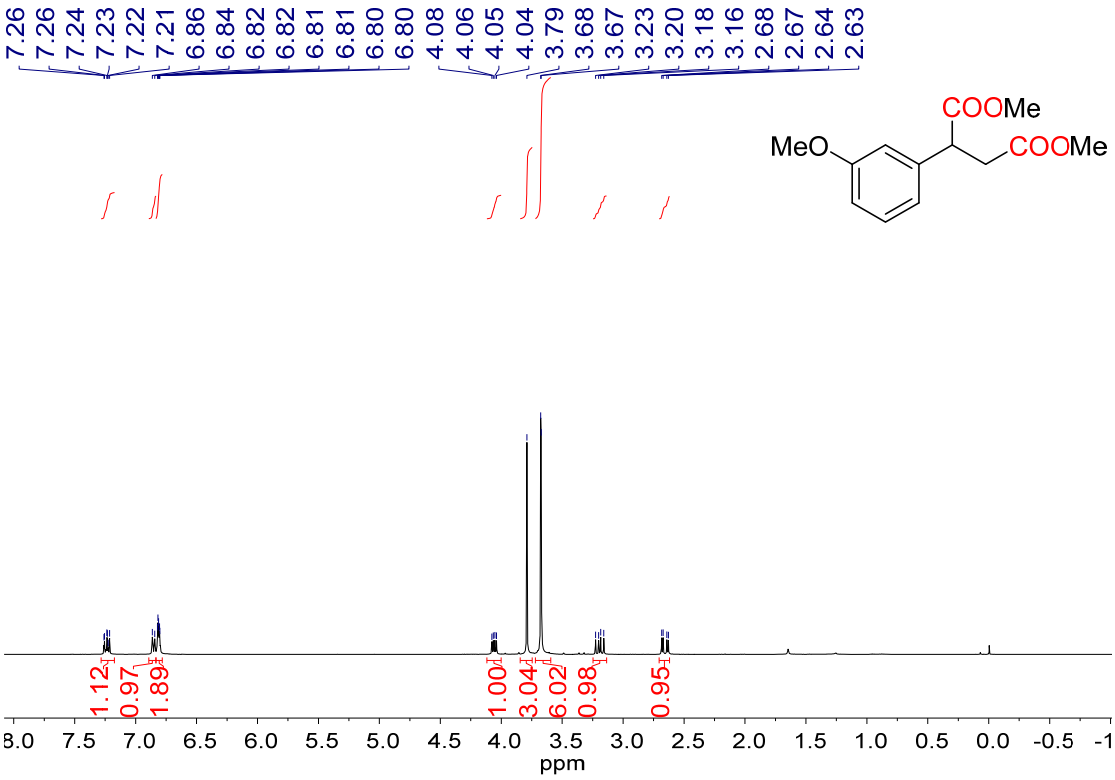
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 2aa



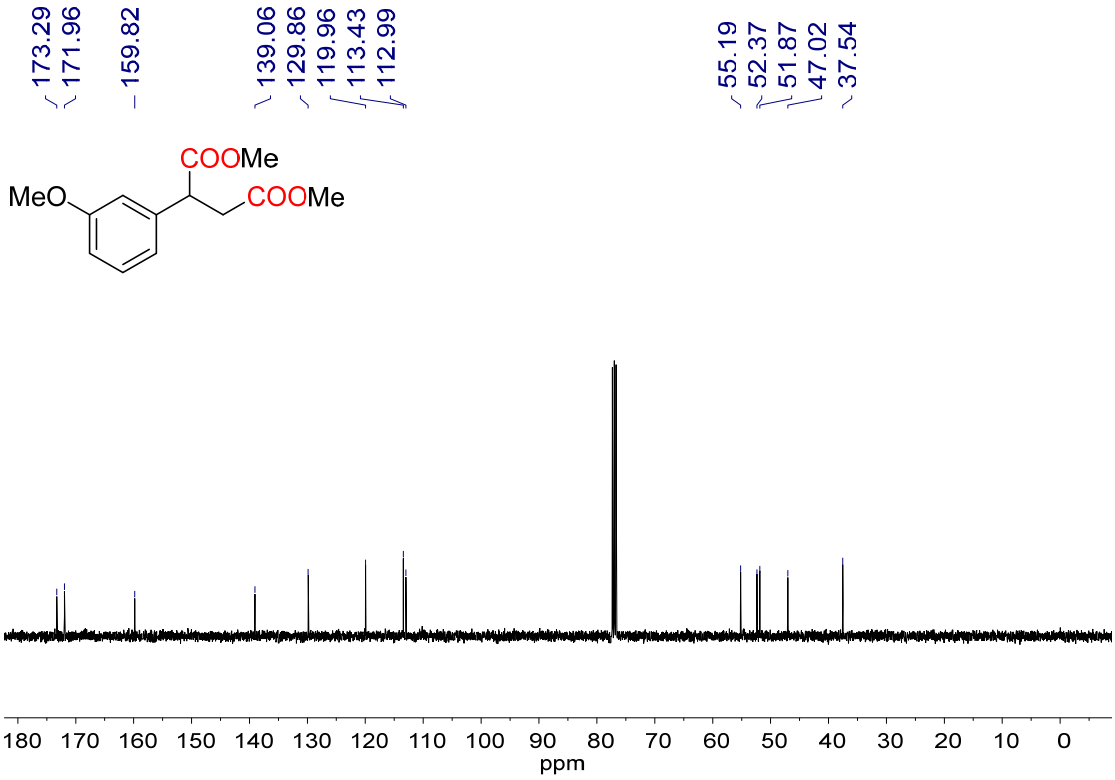
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 3aa



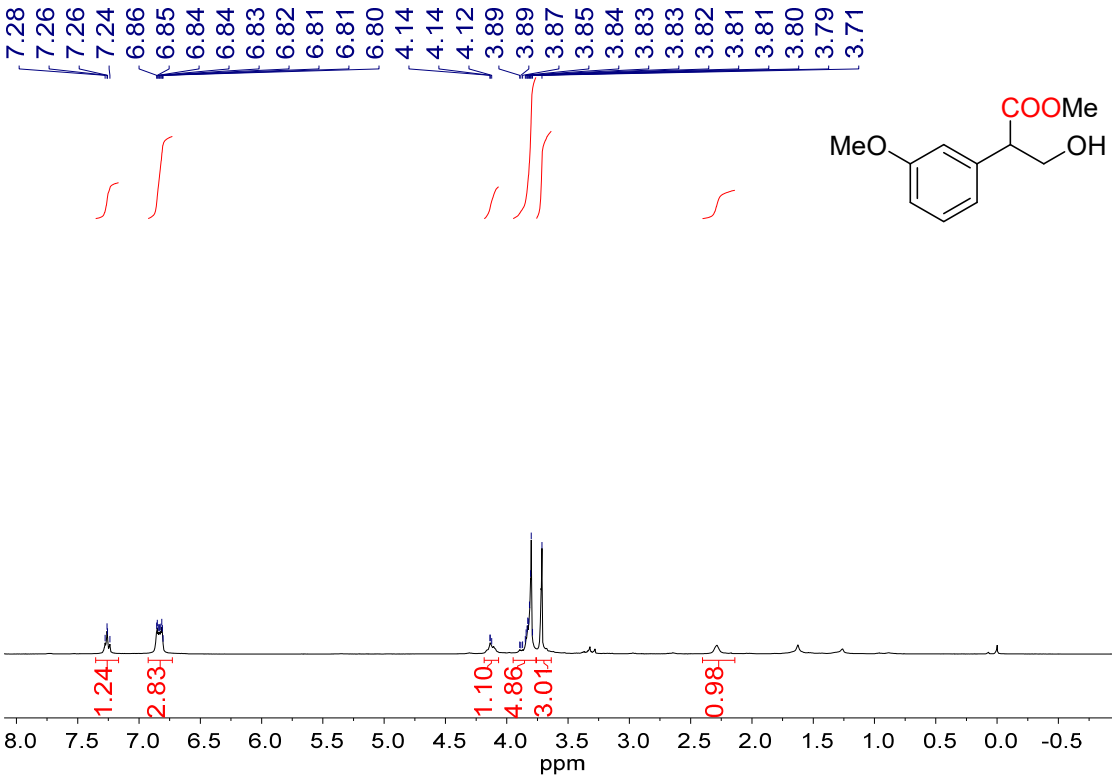
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 3aa



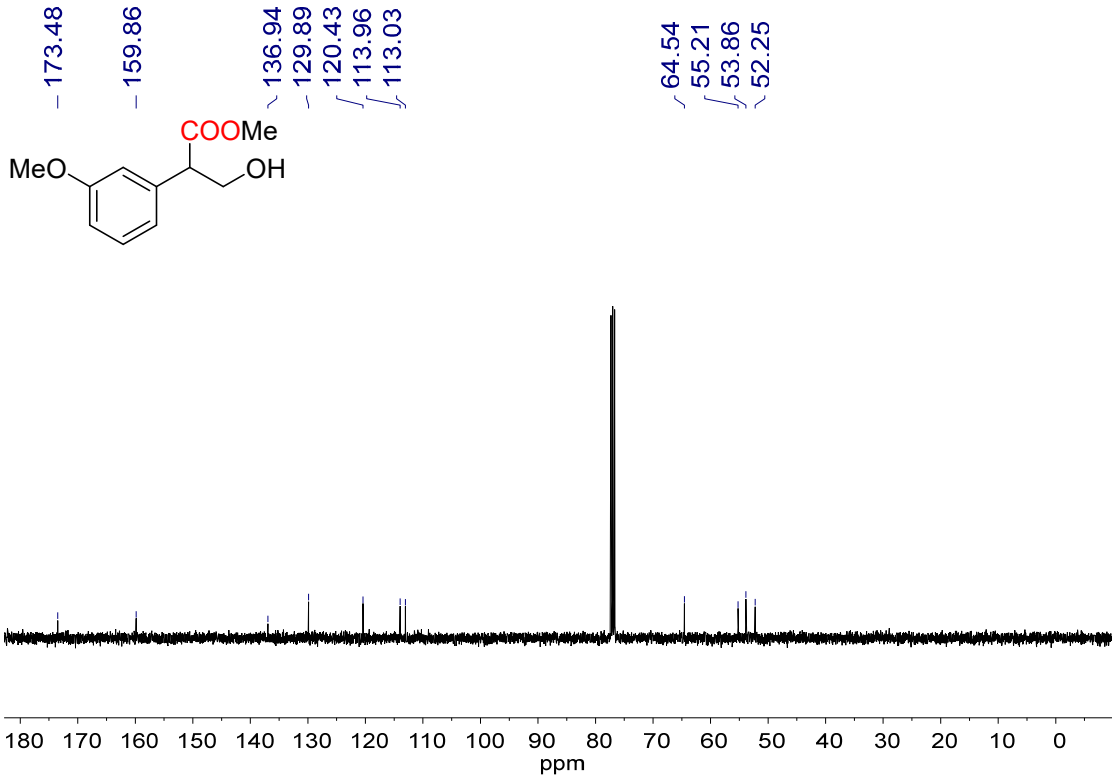
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 2ab



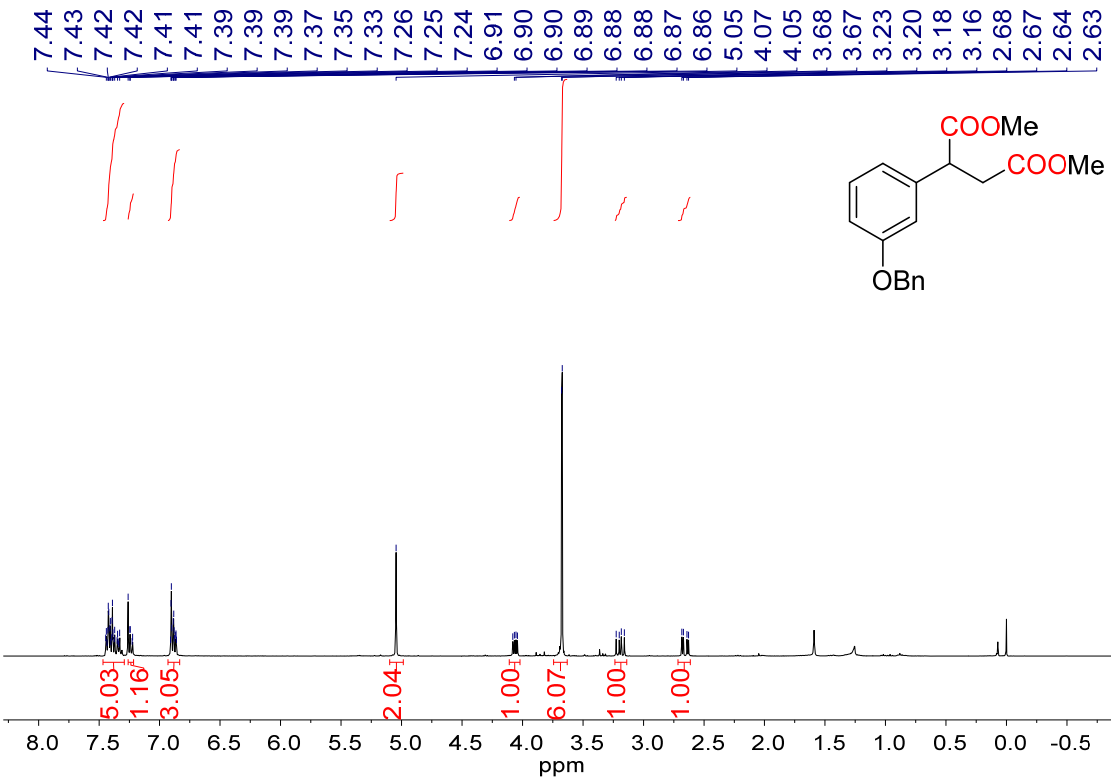
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 2ab



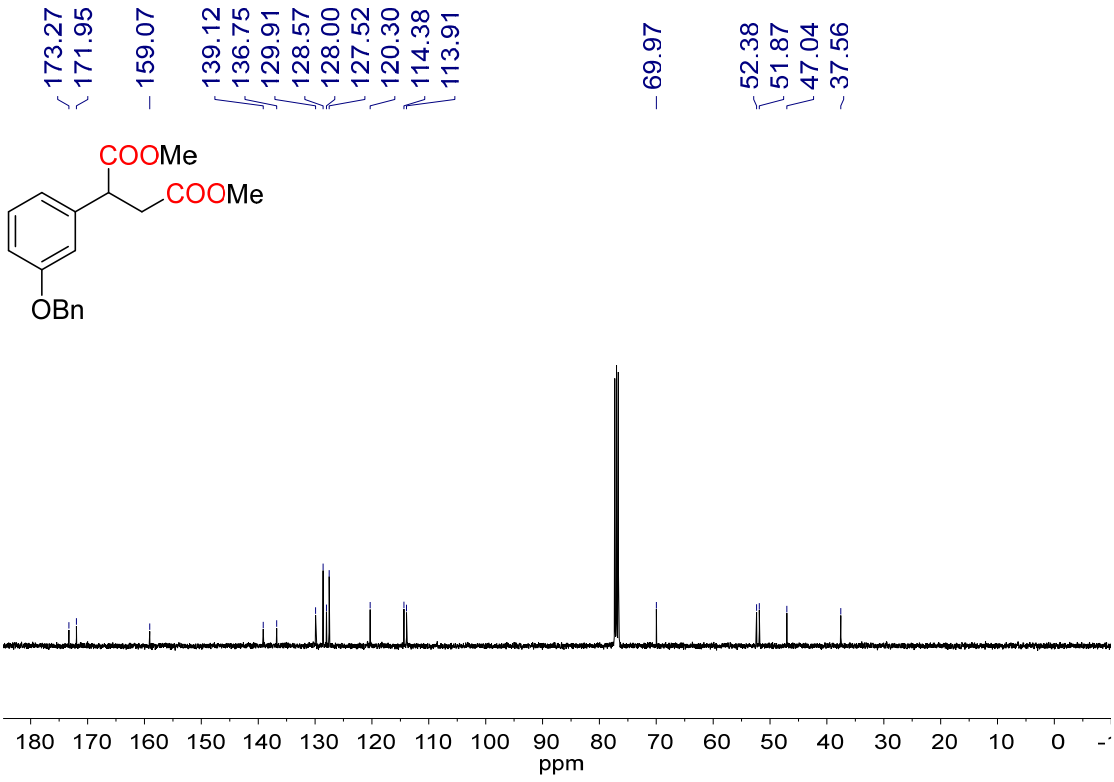
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 3ab



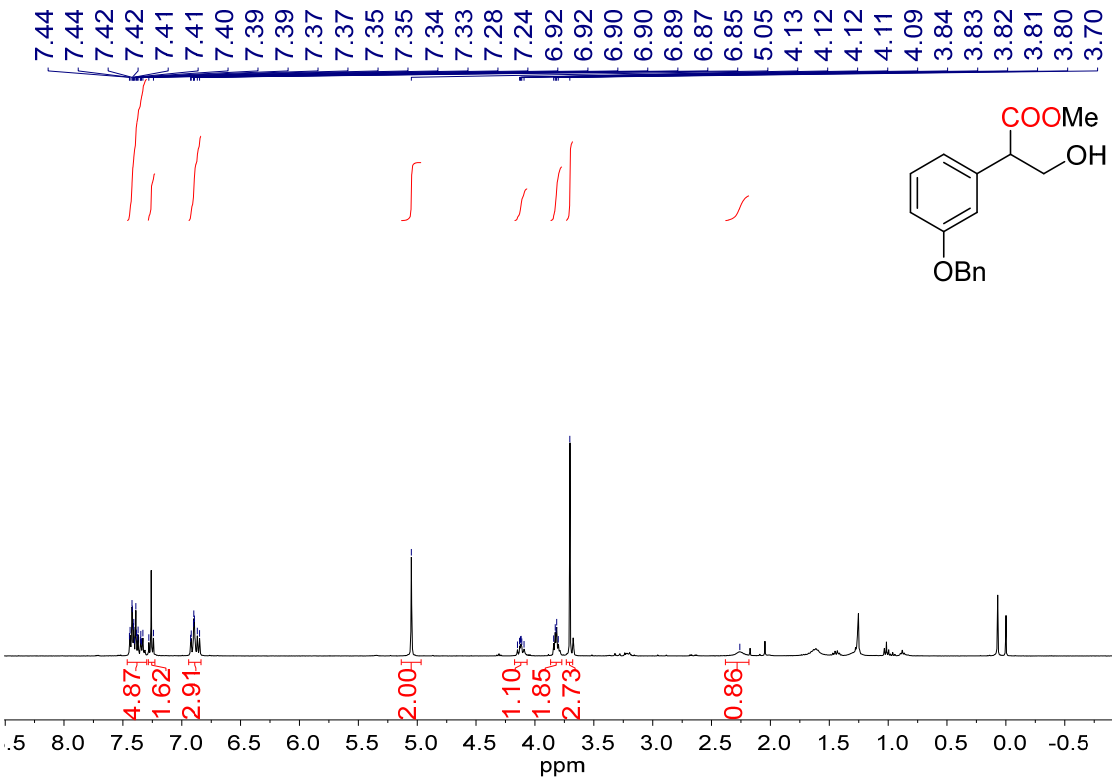
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 3ab



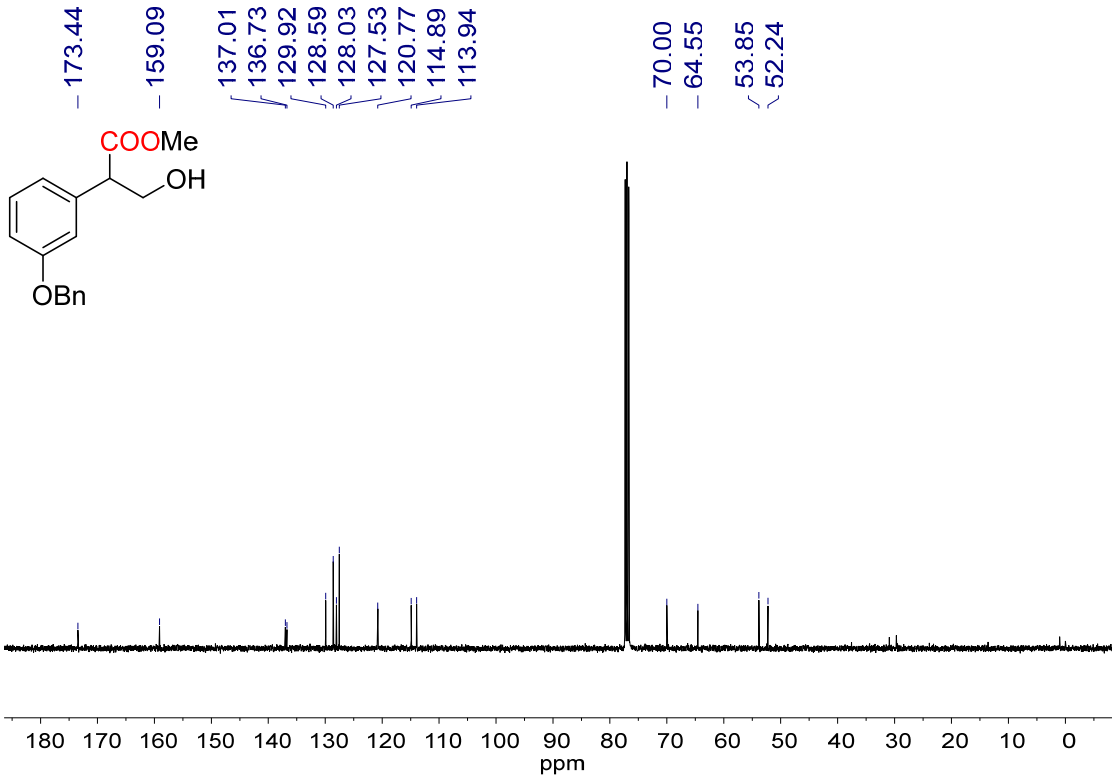
^{1}H NMR spectrum (600 MHz, Chloroform-*d*) of 2ac



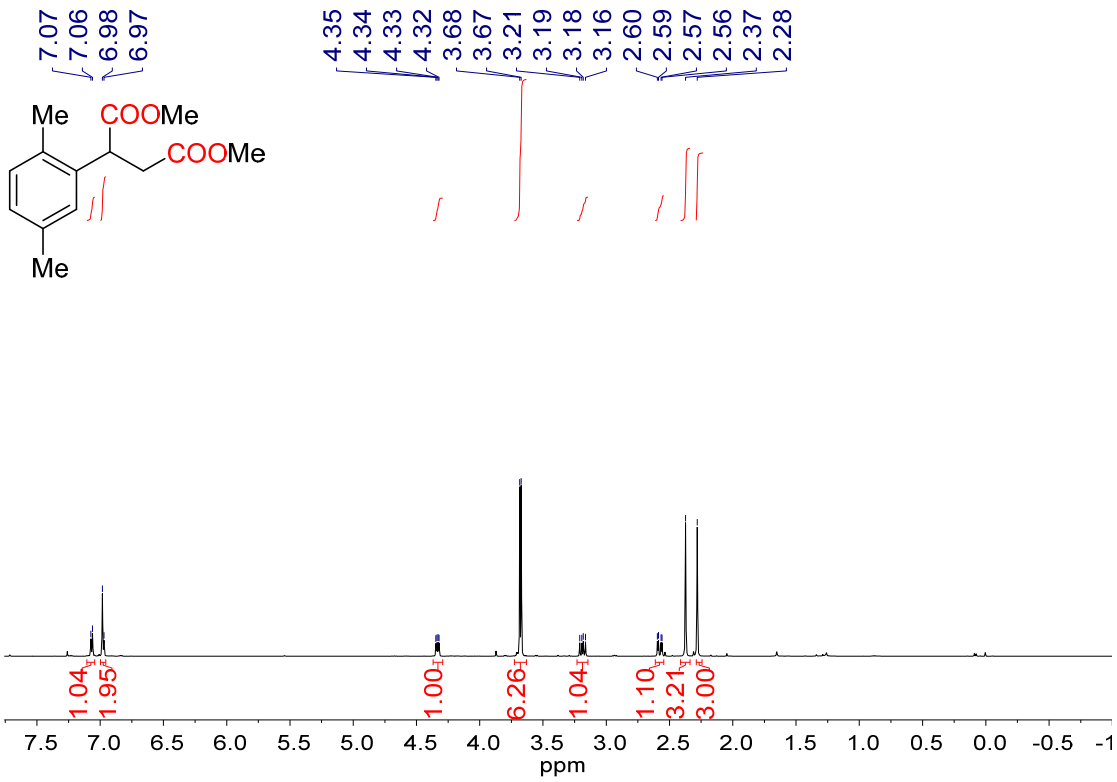
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 2ac



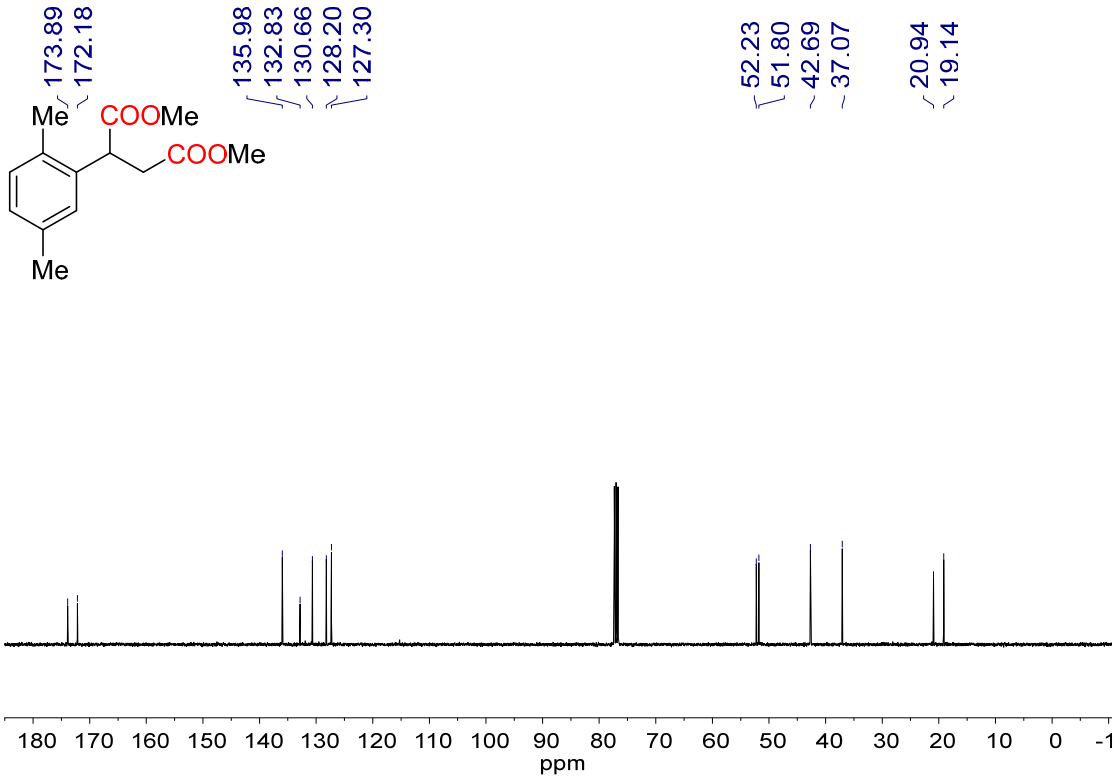
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 3ac



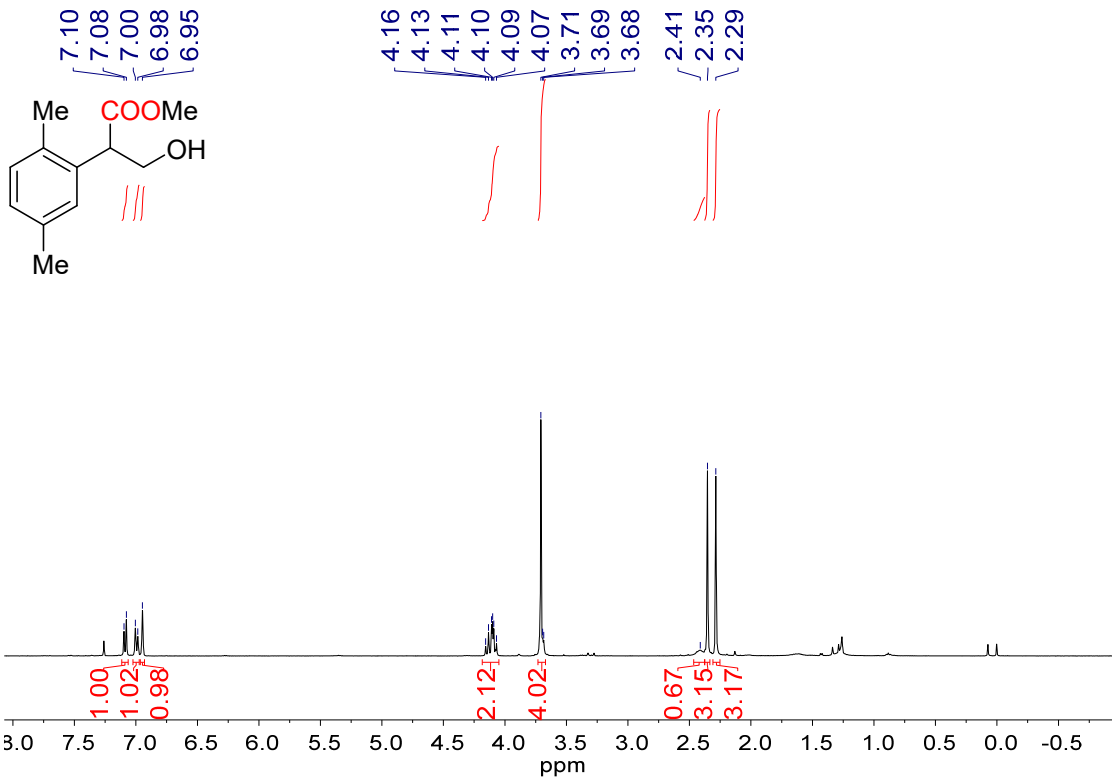
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 3ac



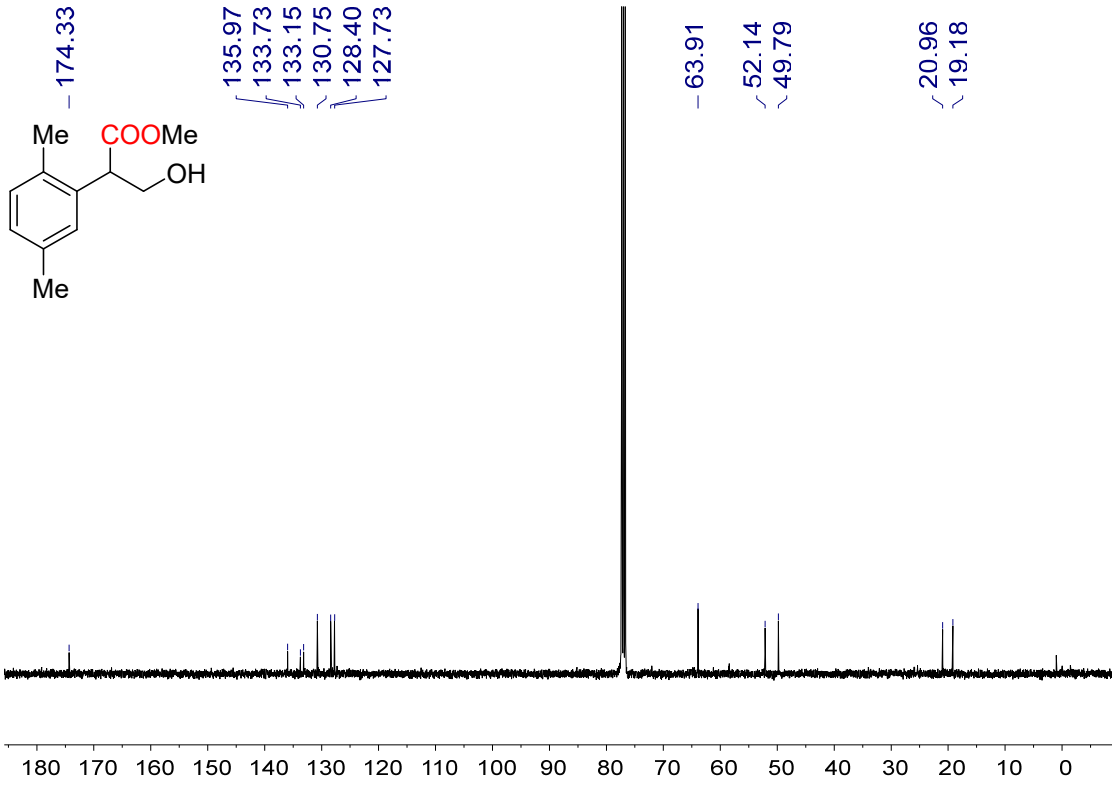
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 2ad



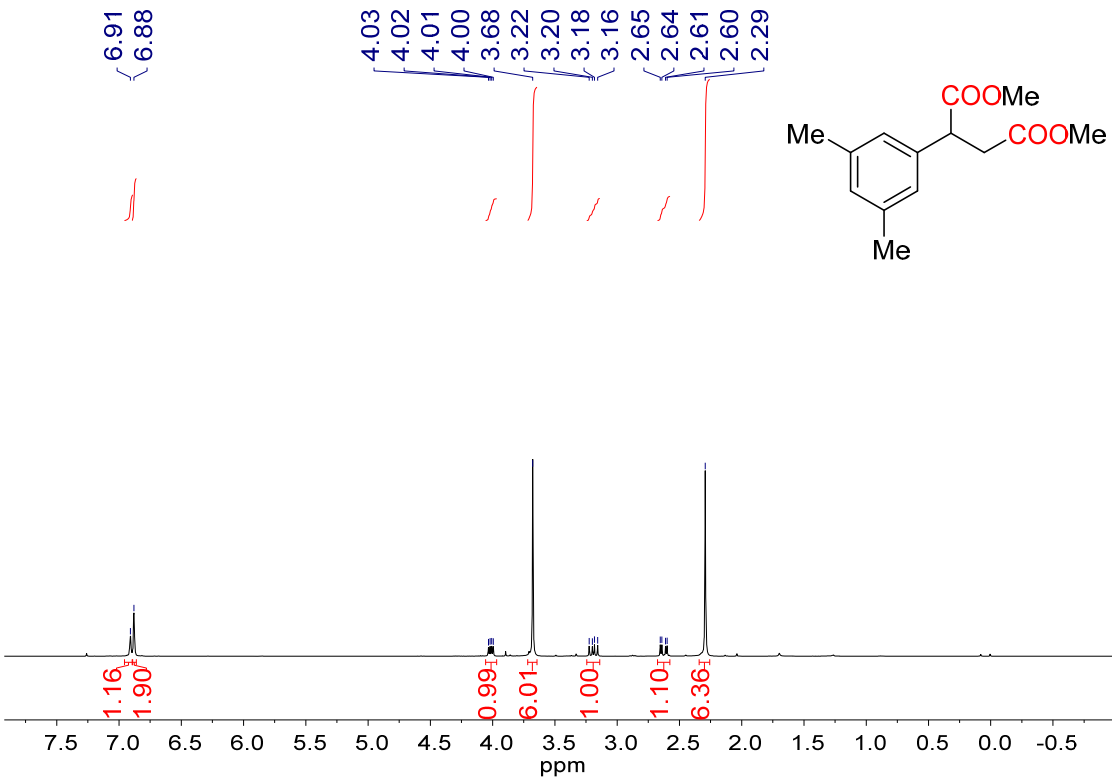
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 2ad



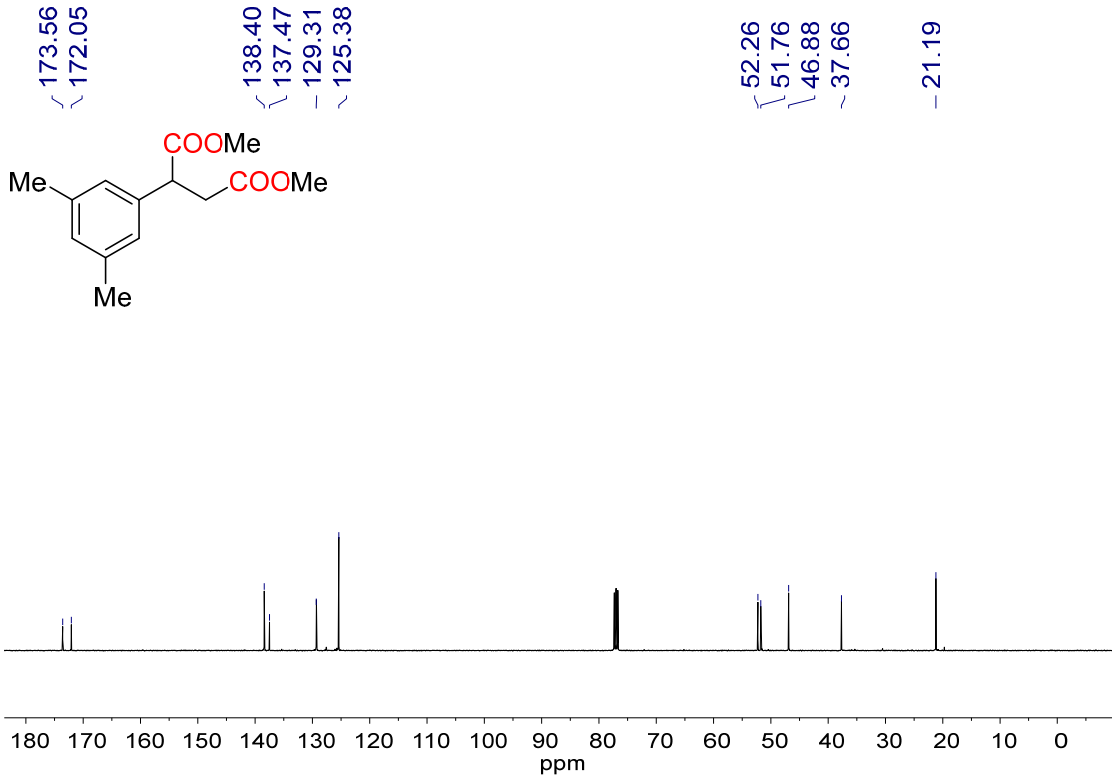
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 3ad



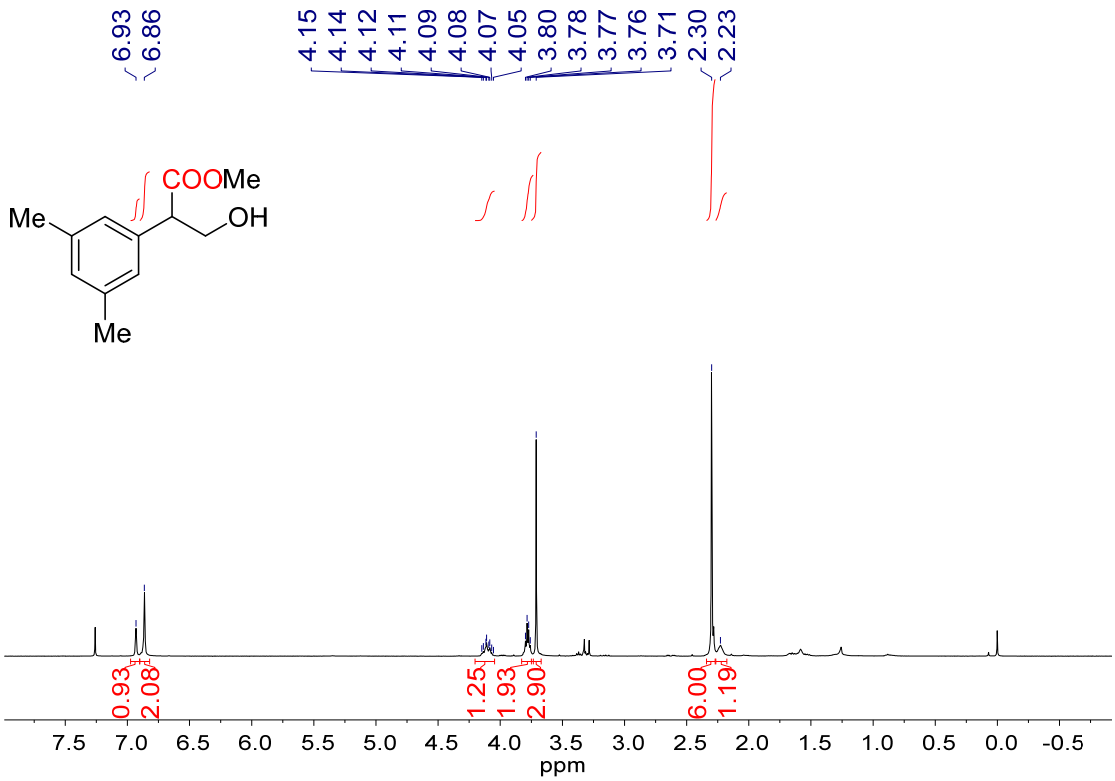
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 3ad



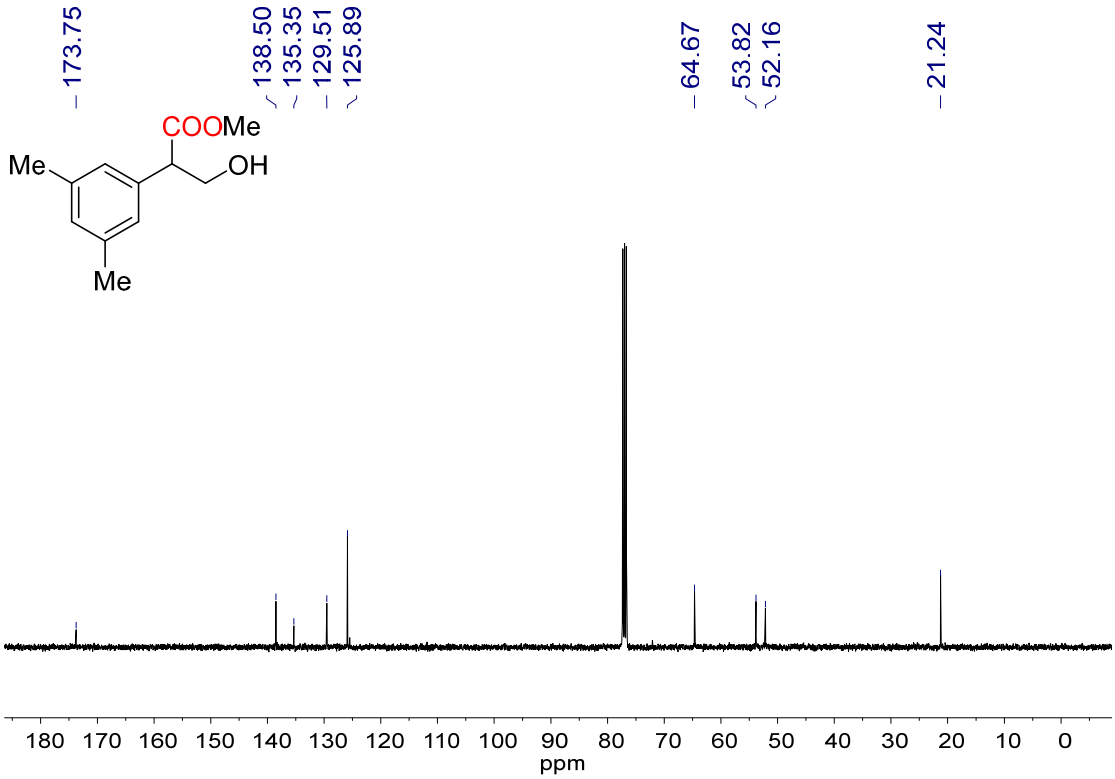
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 2ae



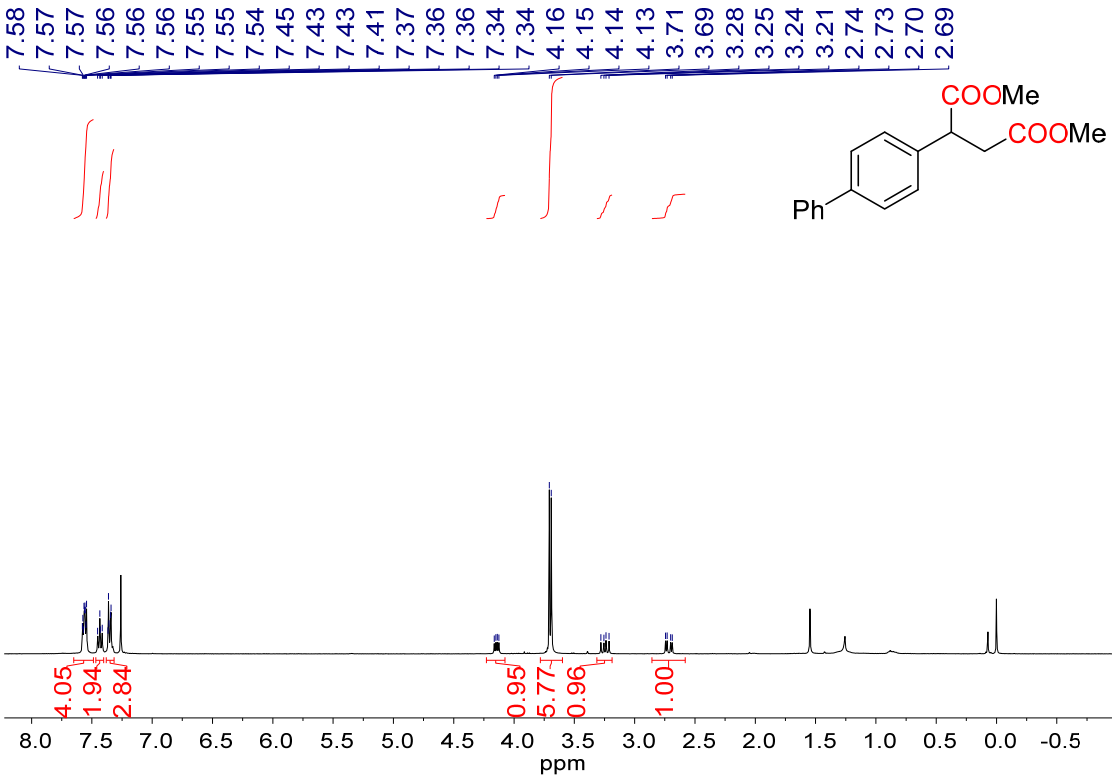
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 2ae



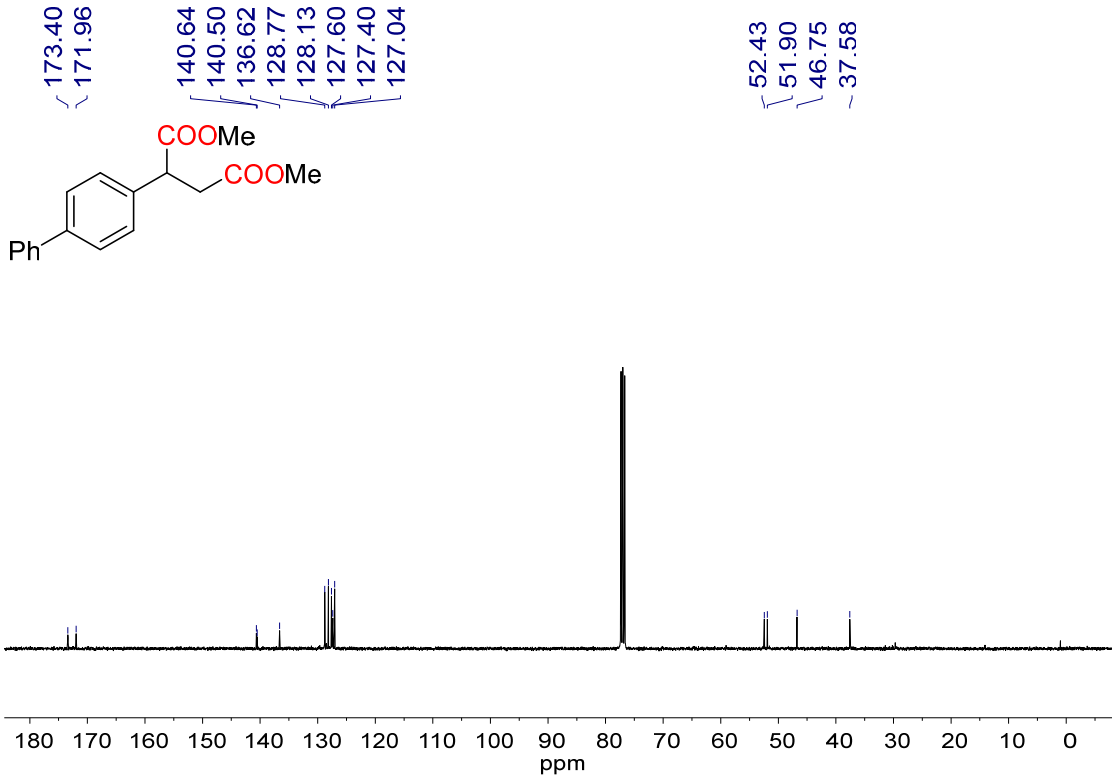
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 3ae



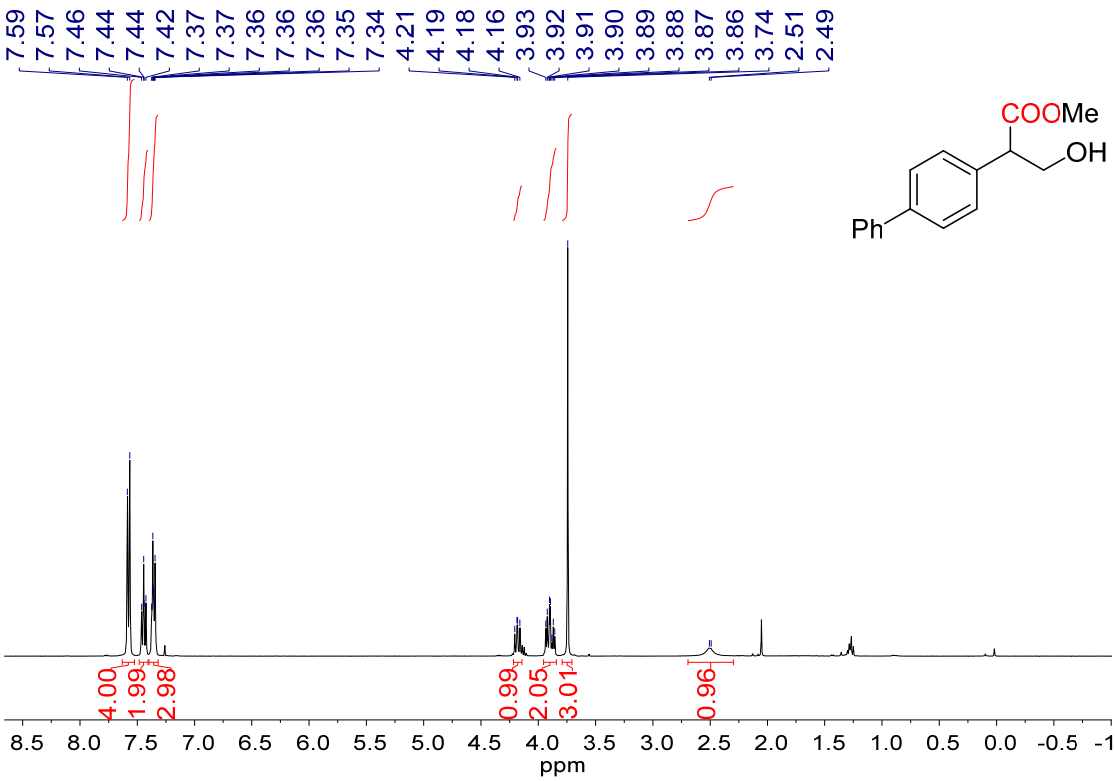
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 3ae



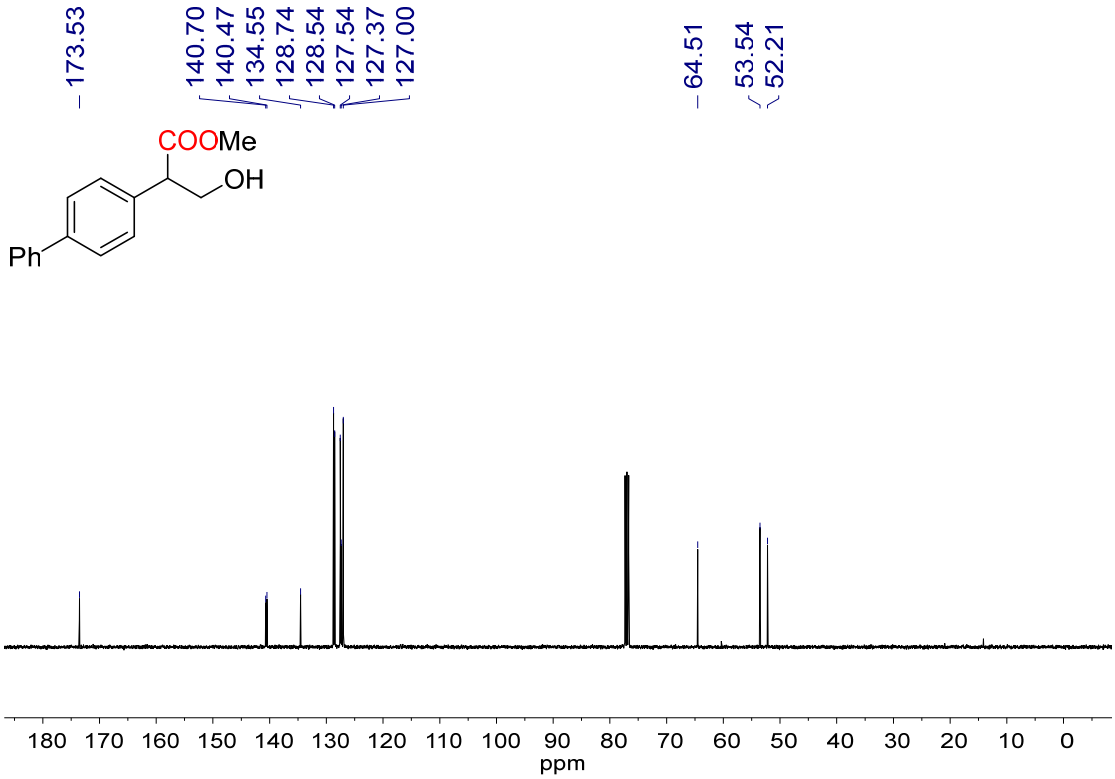
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 2af



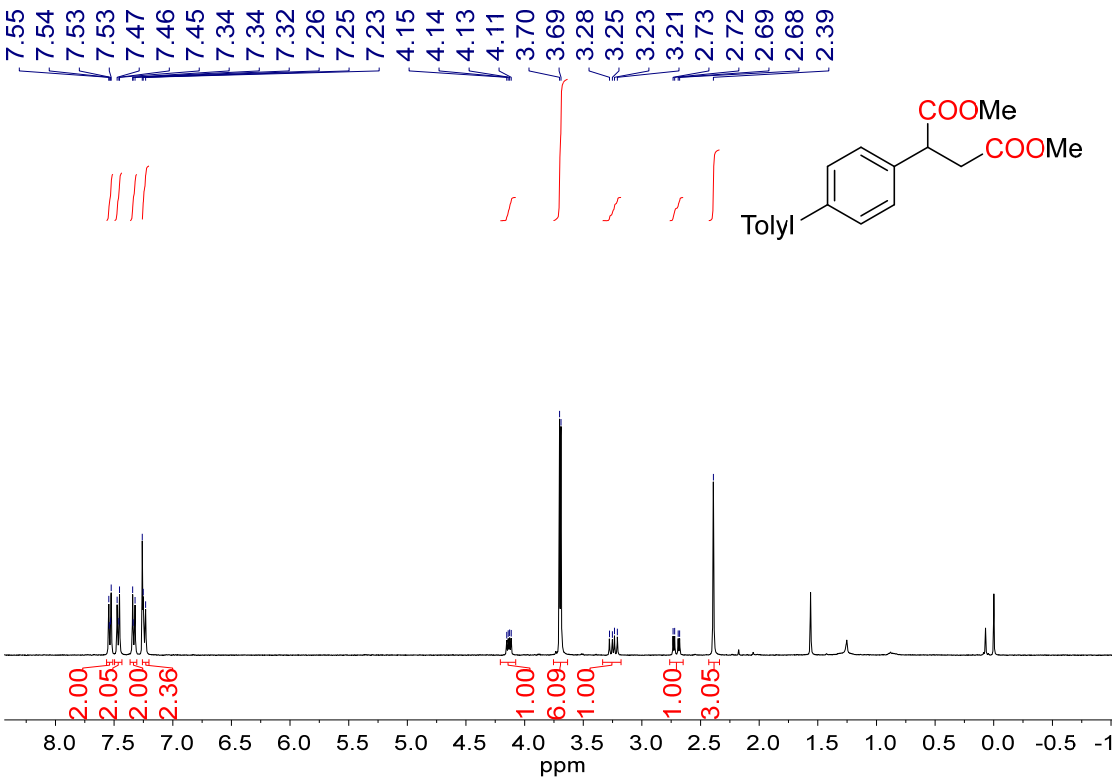
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 2af



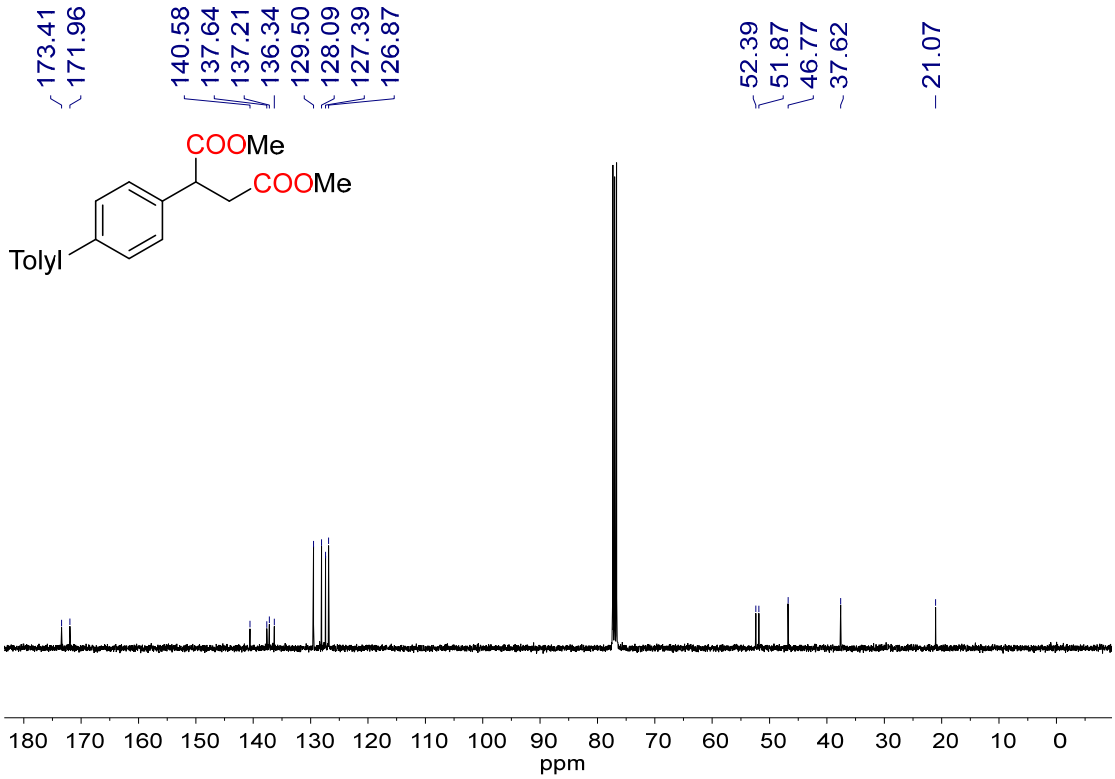
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 3af



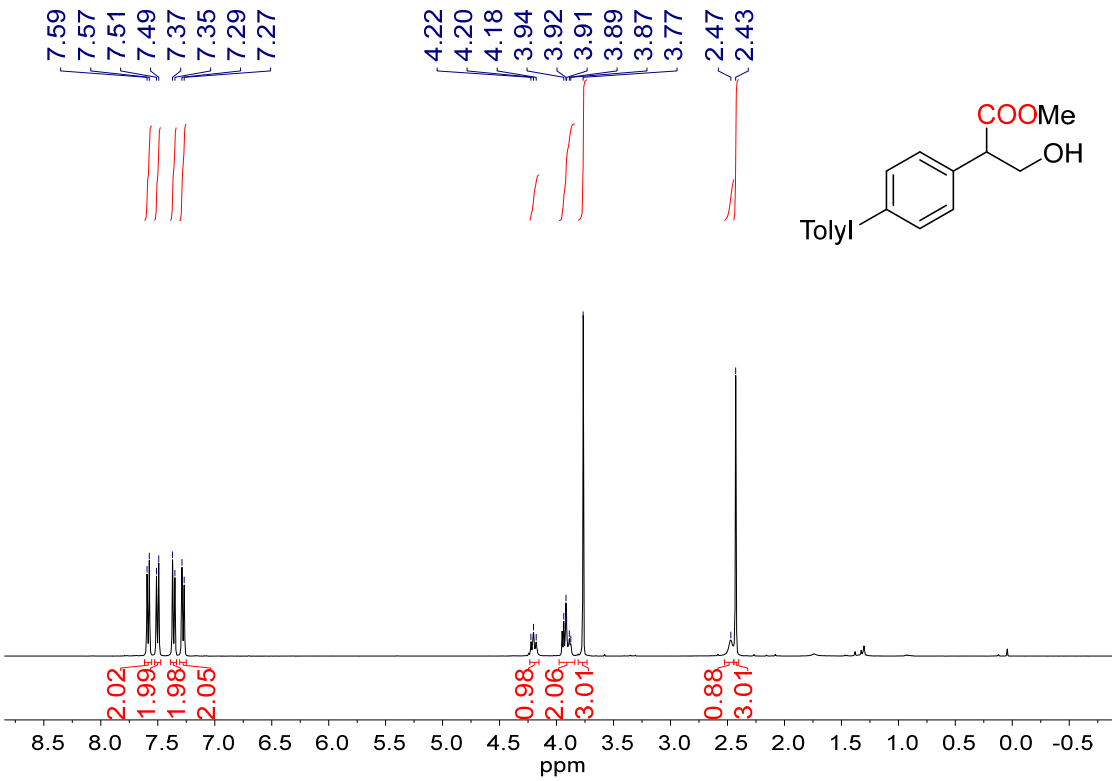
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 3af



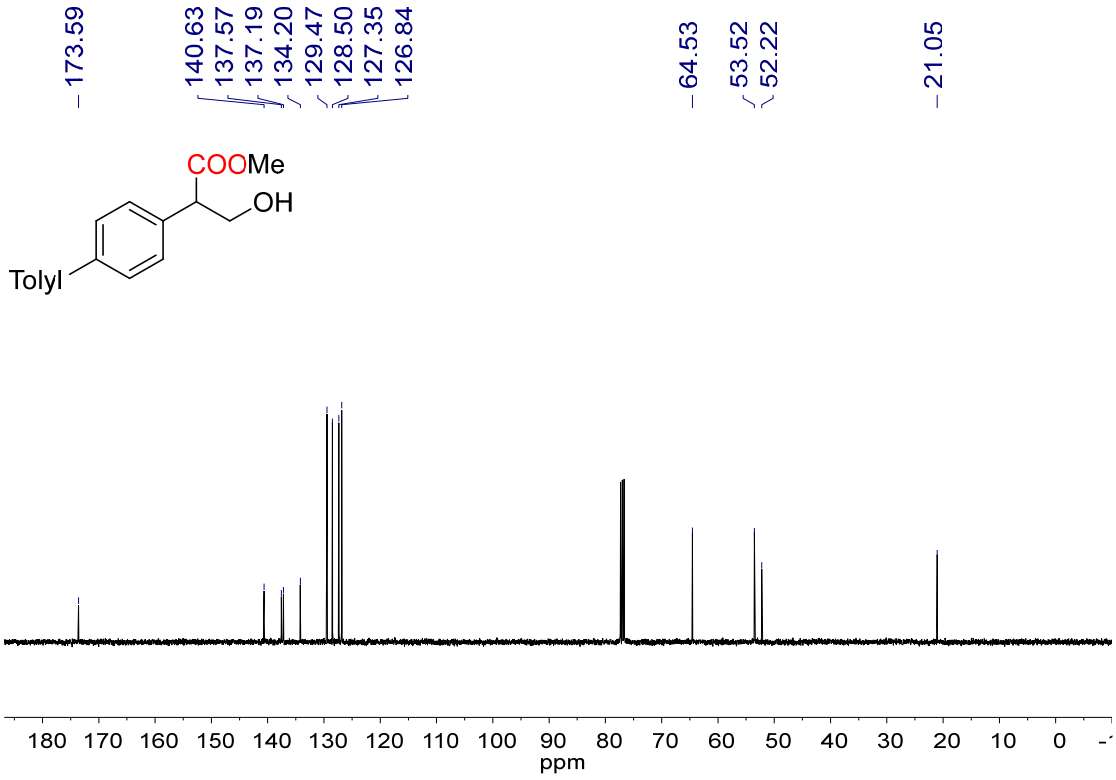
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 2ag



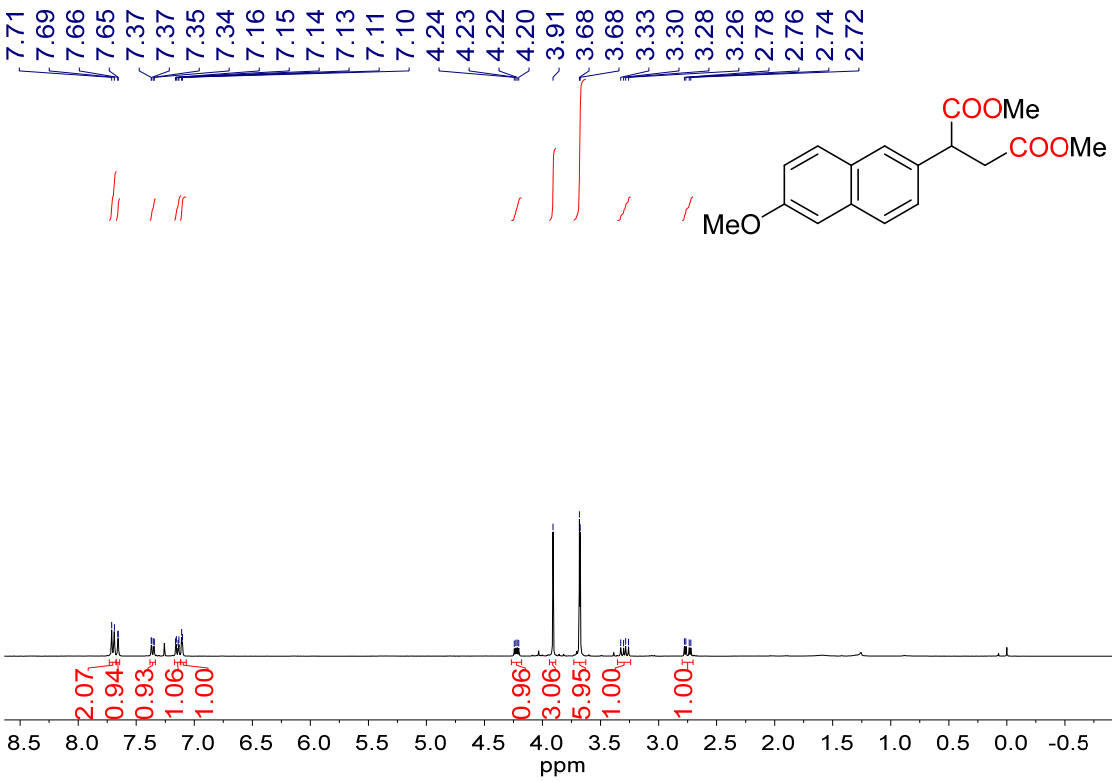
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 2ag



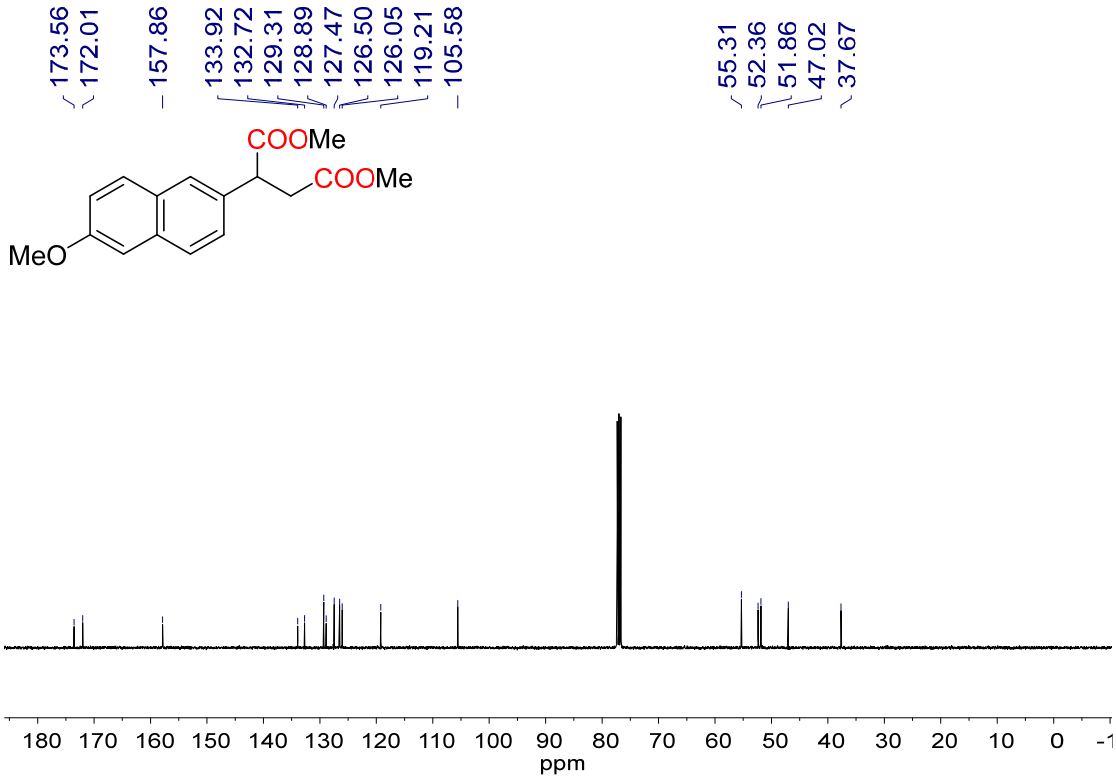
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 3ag



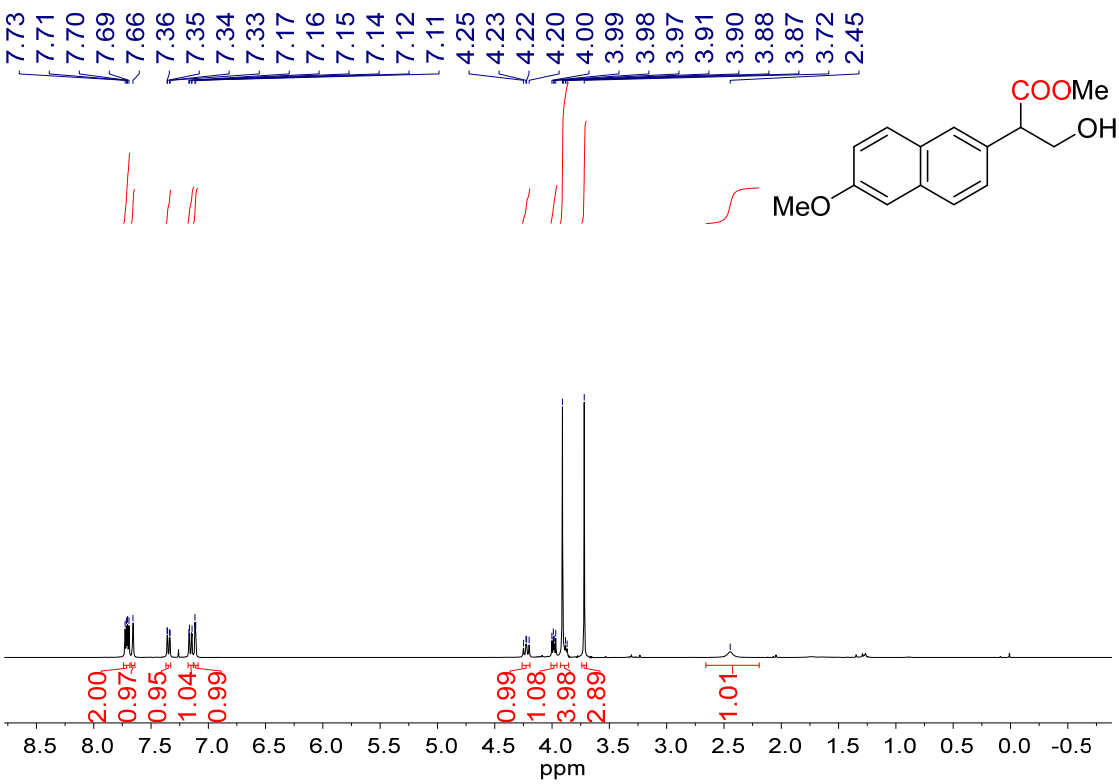
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 3ag



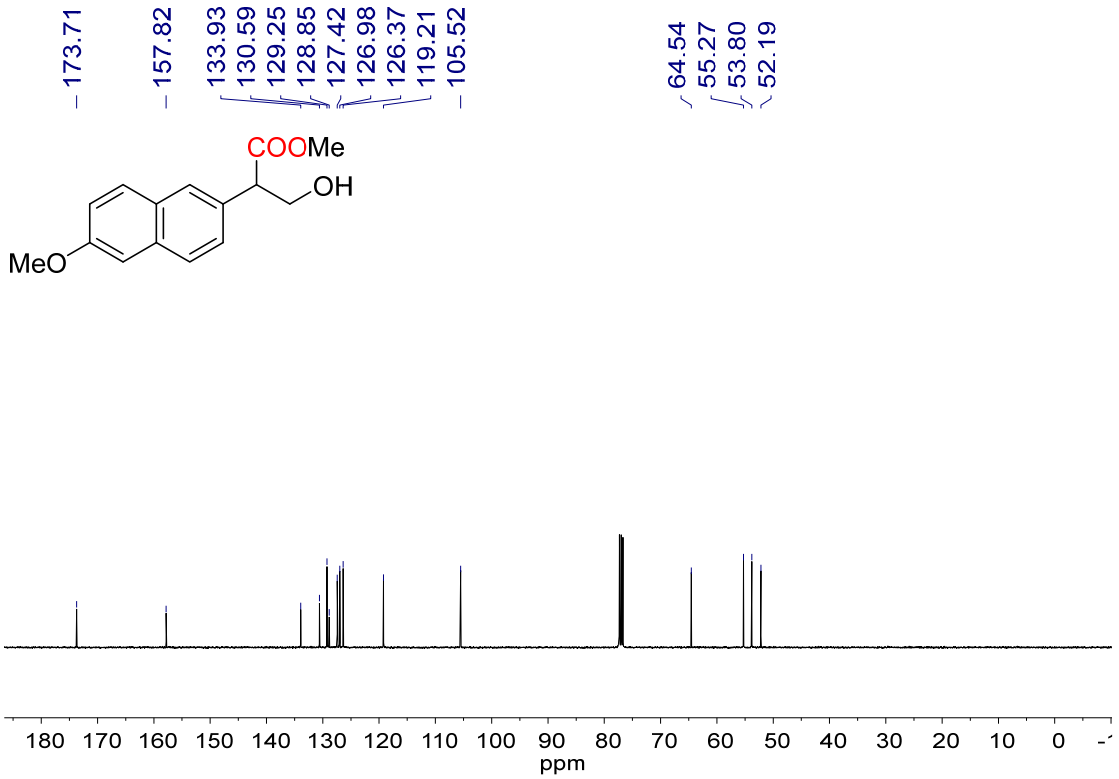
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 2ah



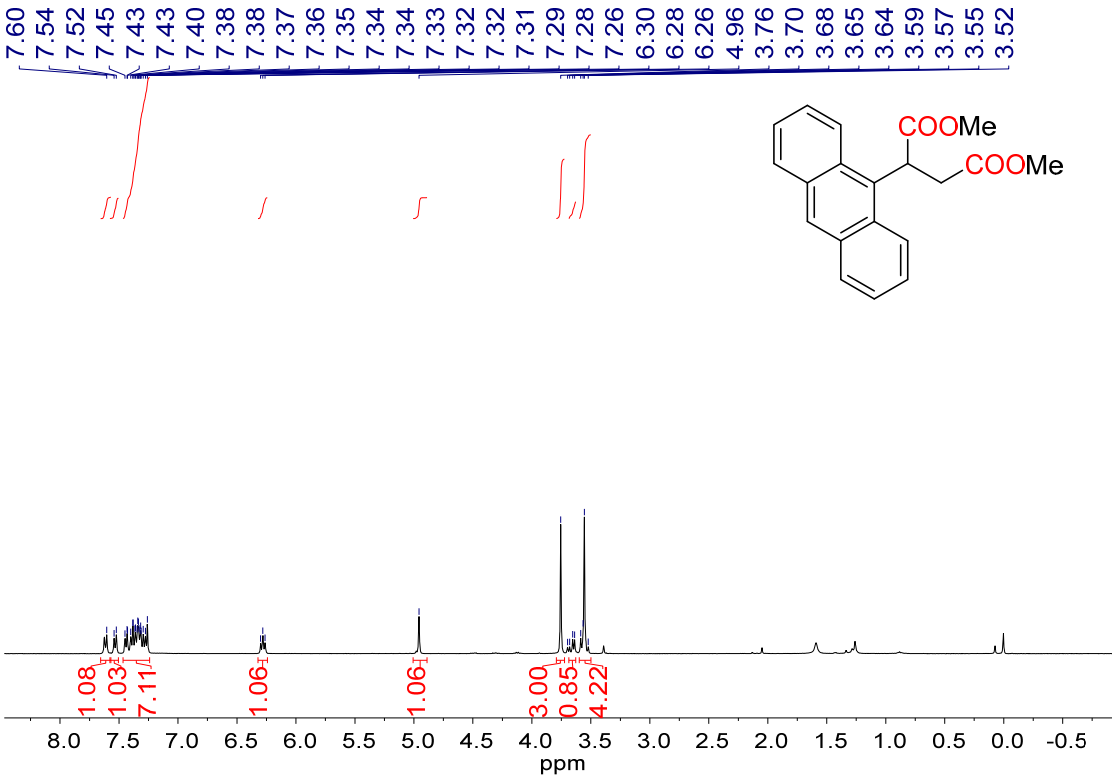
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 2ah



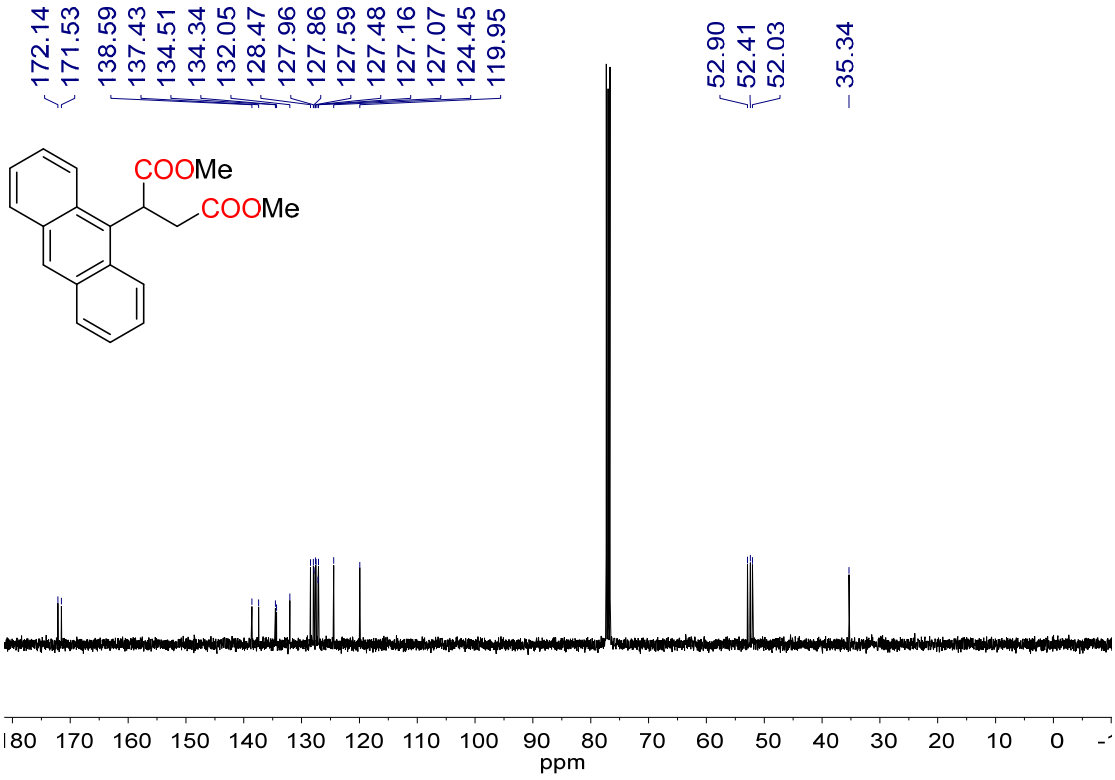
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 3ah



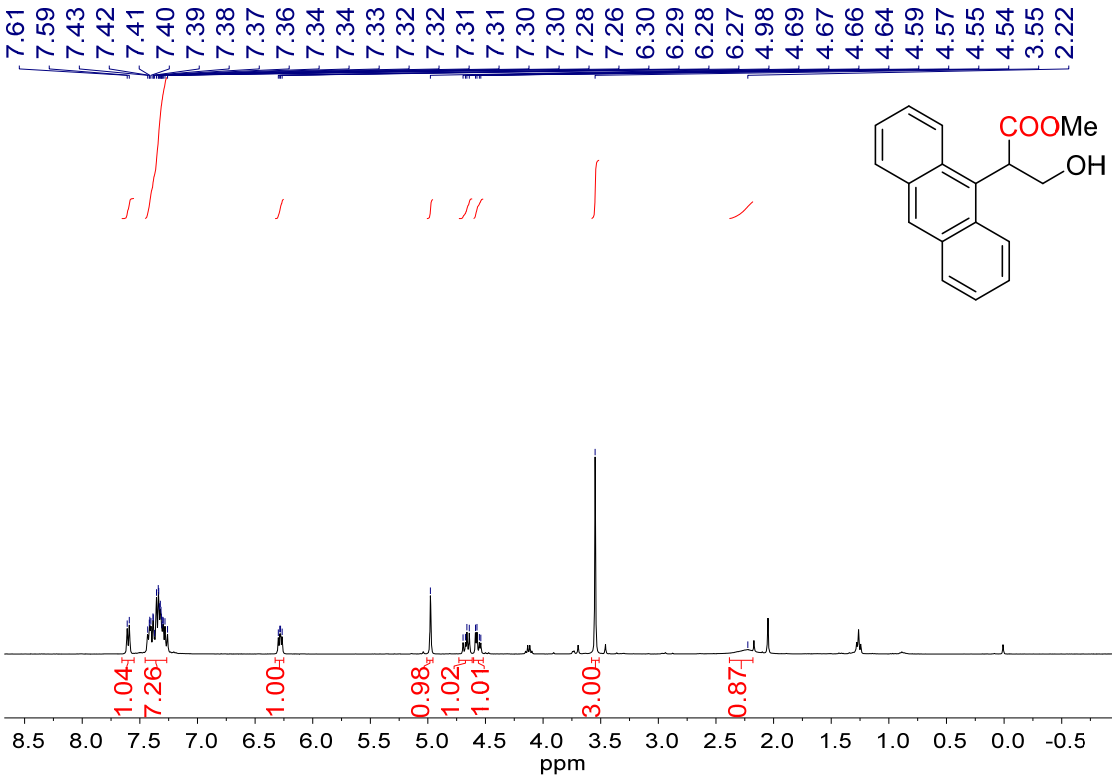
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 3ah



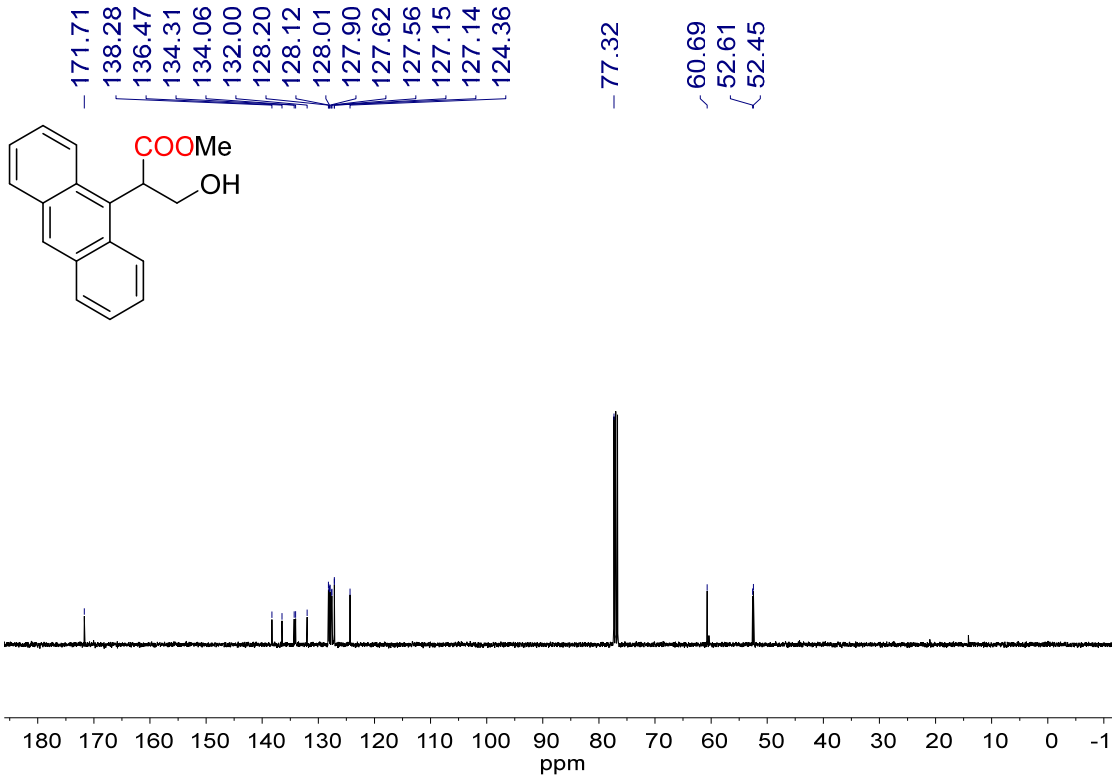
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 2ai



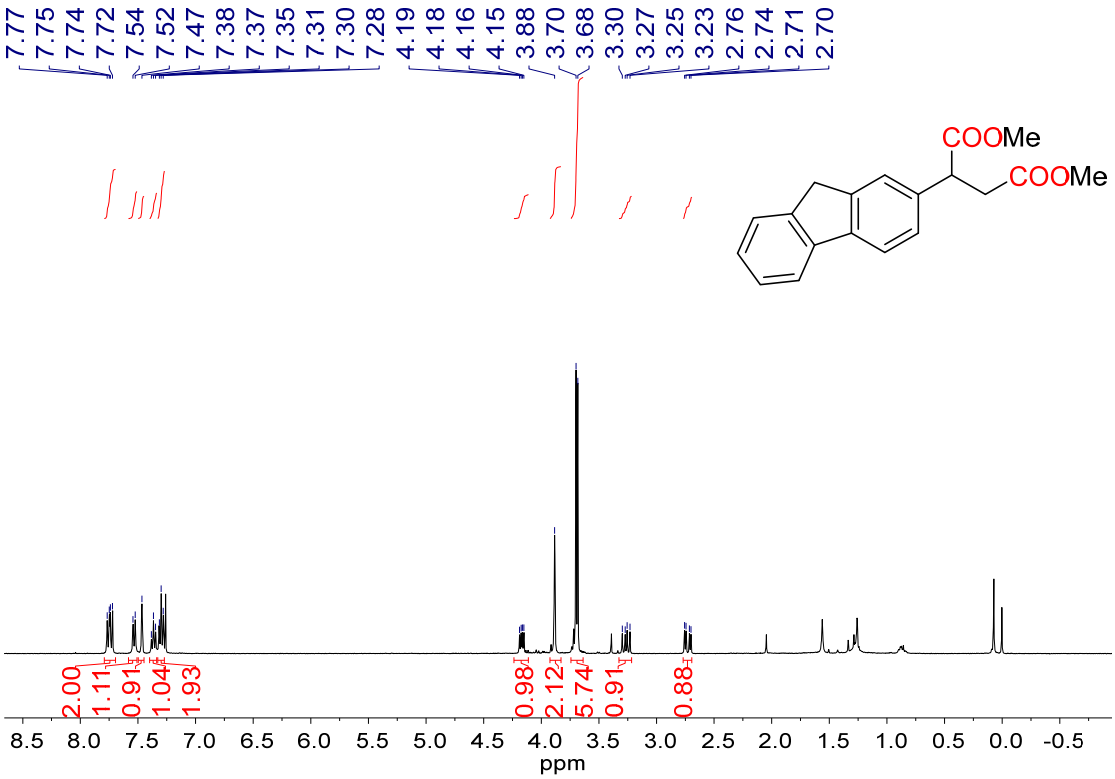
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 2ai



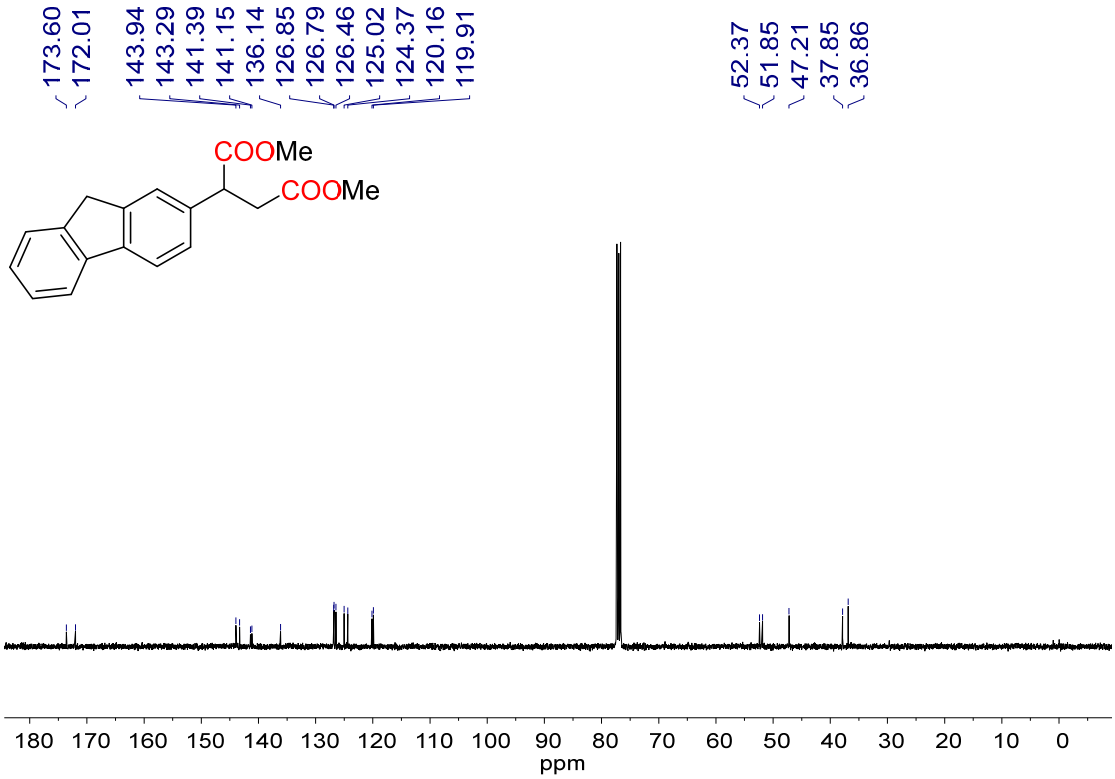
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 3ai



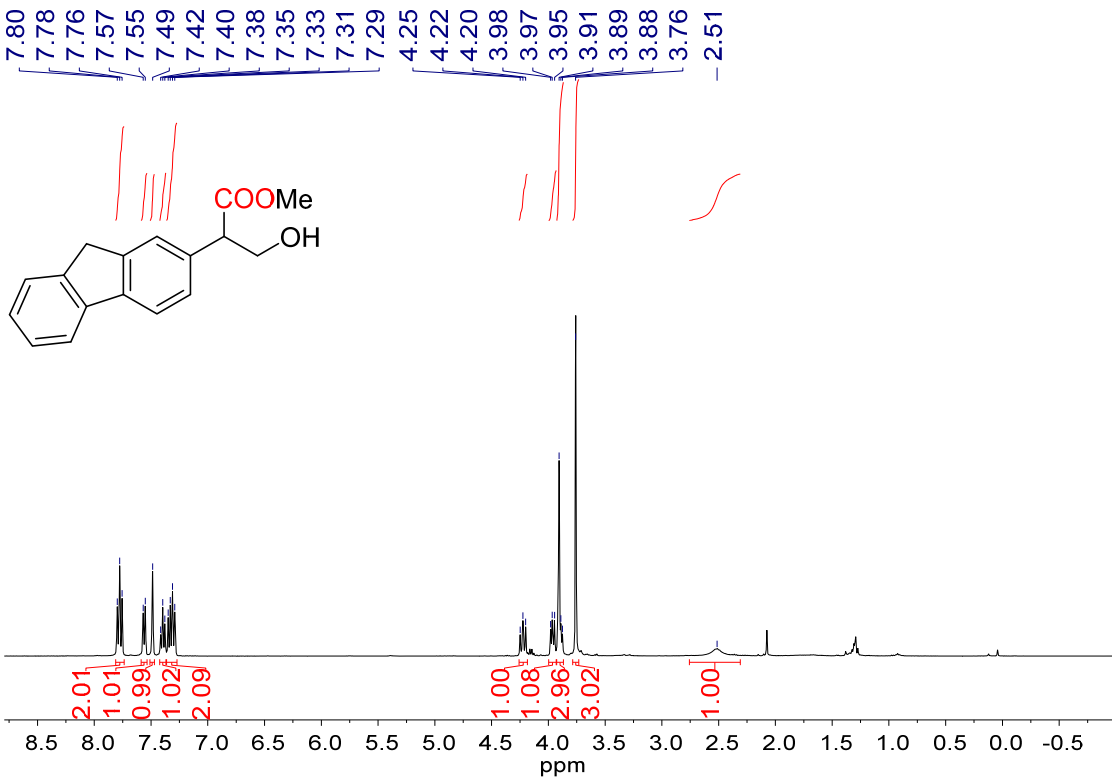
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 3ai



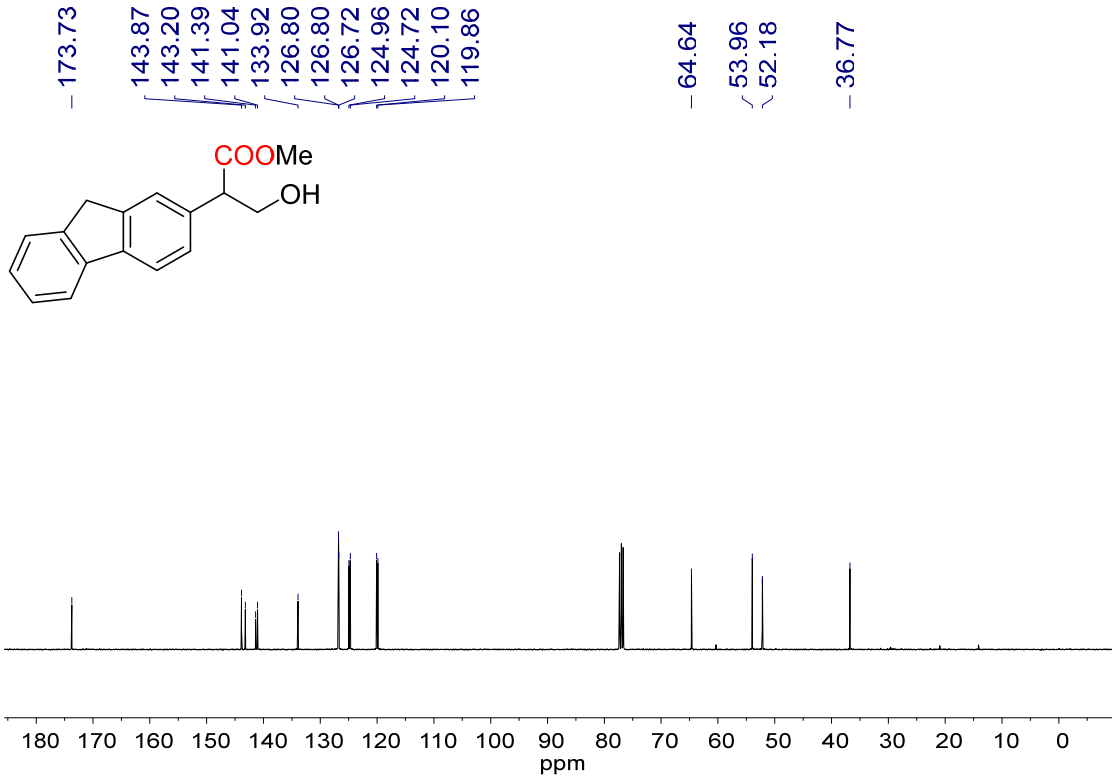
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 2aj



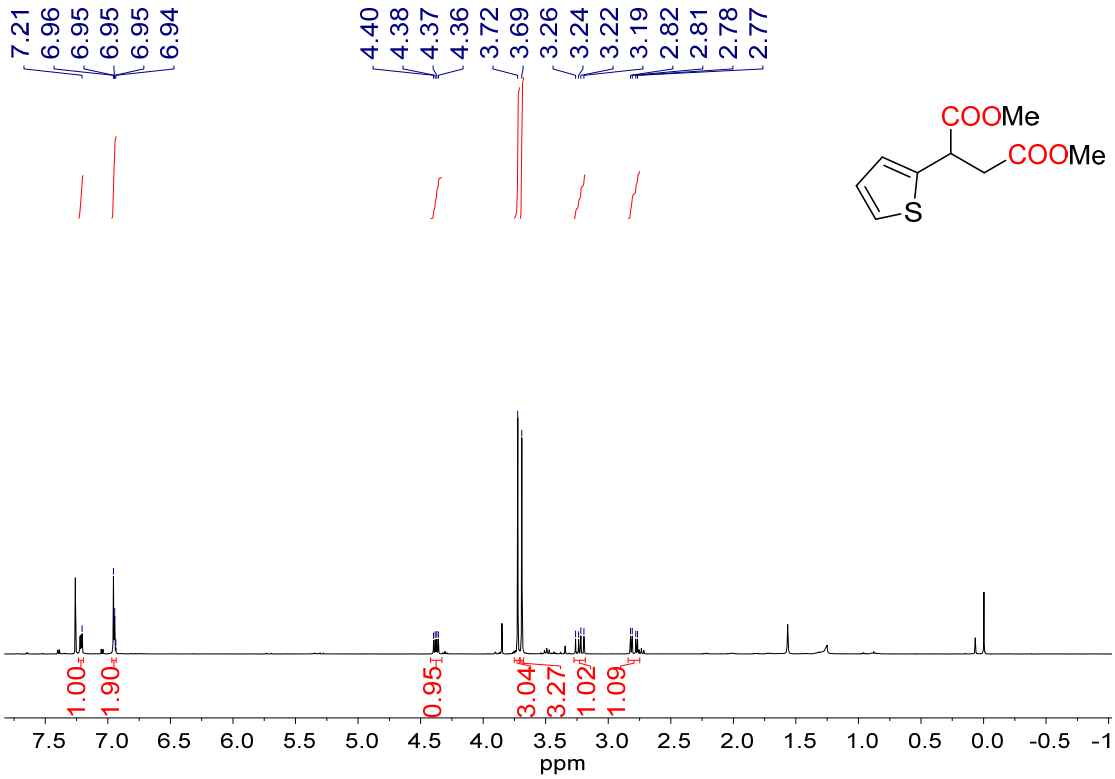
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 2aj



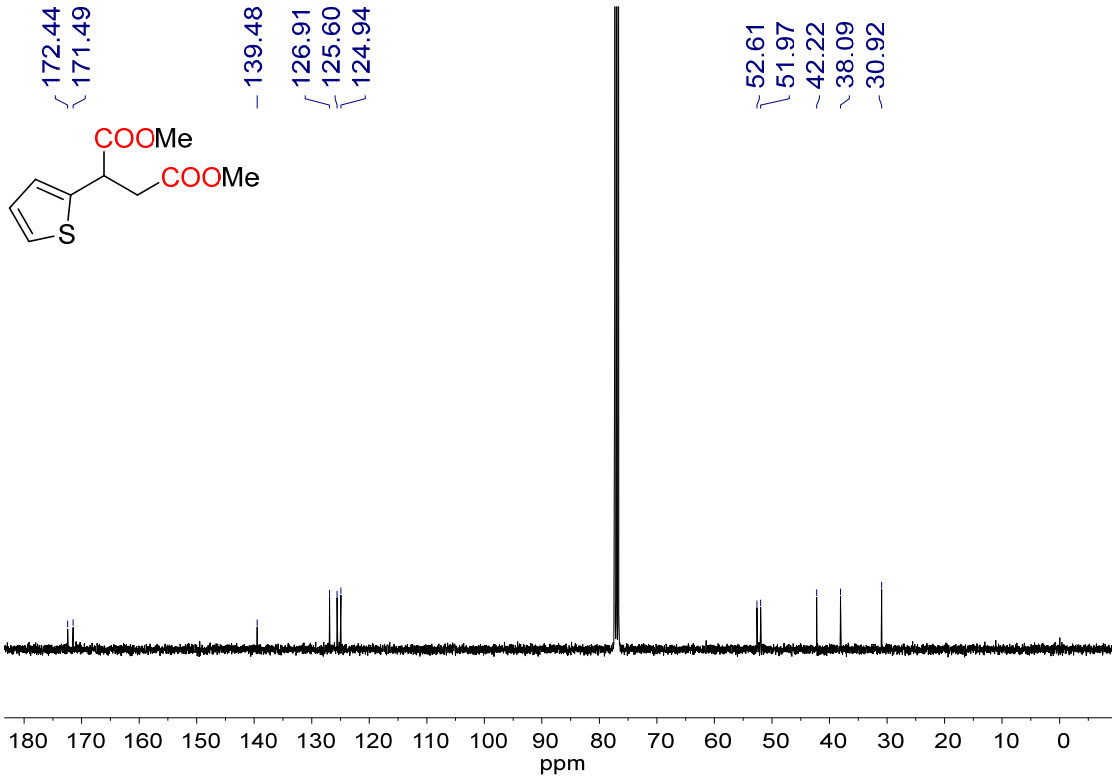
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 3aj



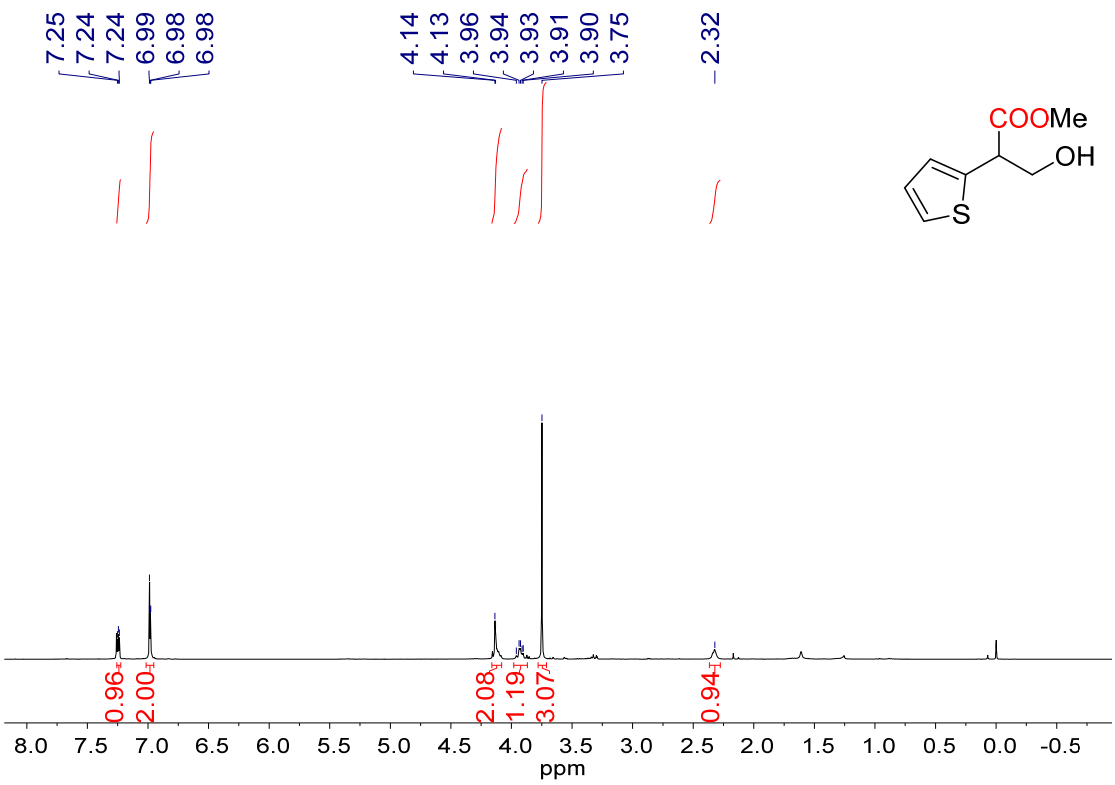
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 3aj



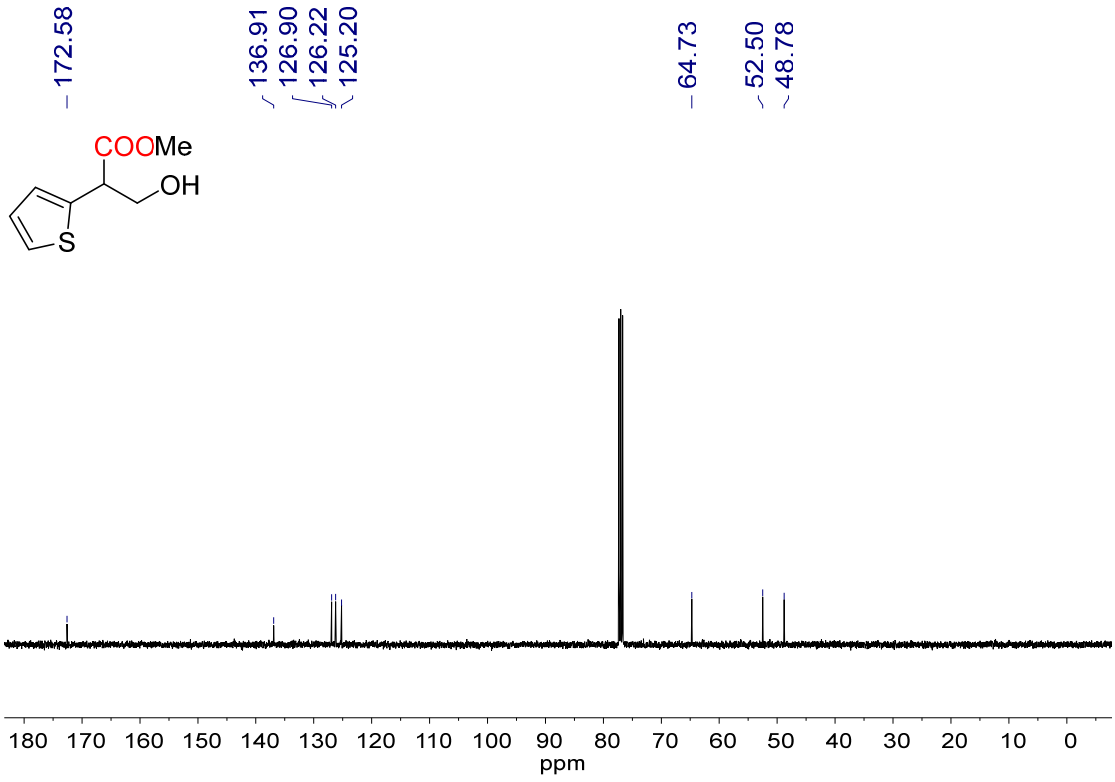
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 2ak



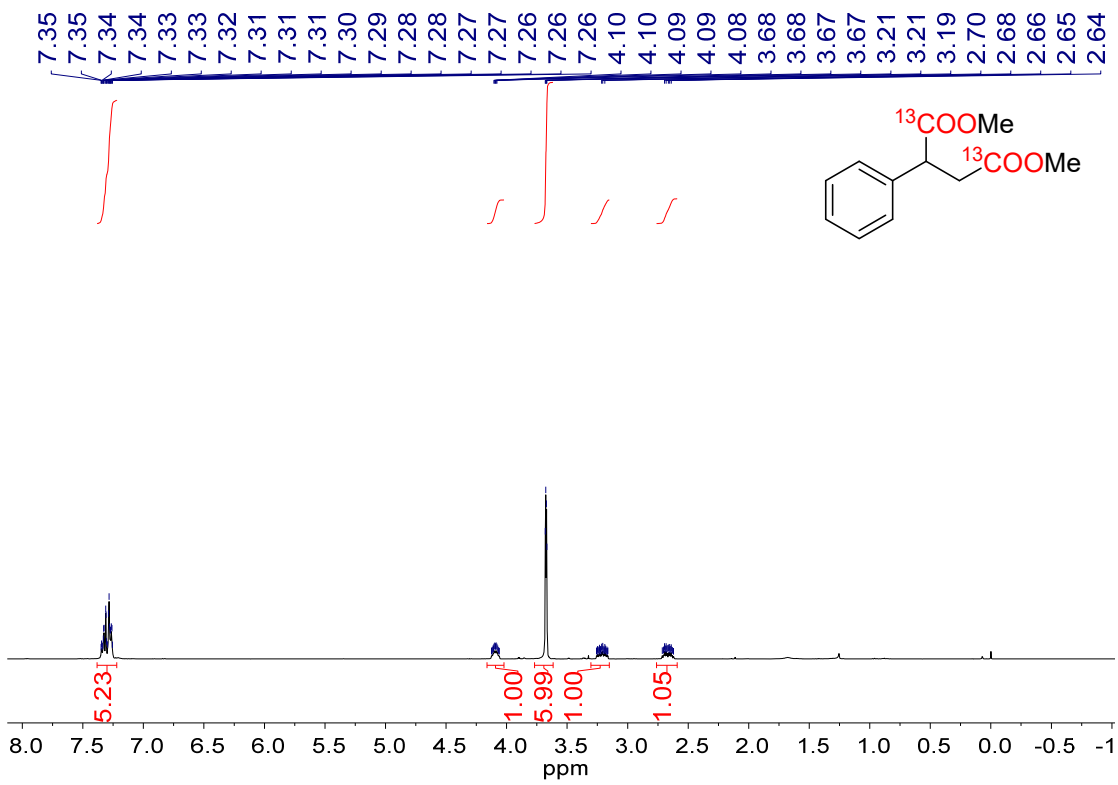
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 2ak



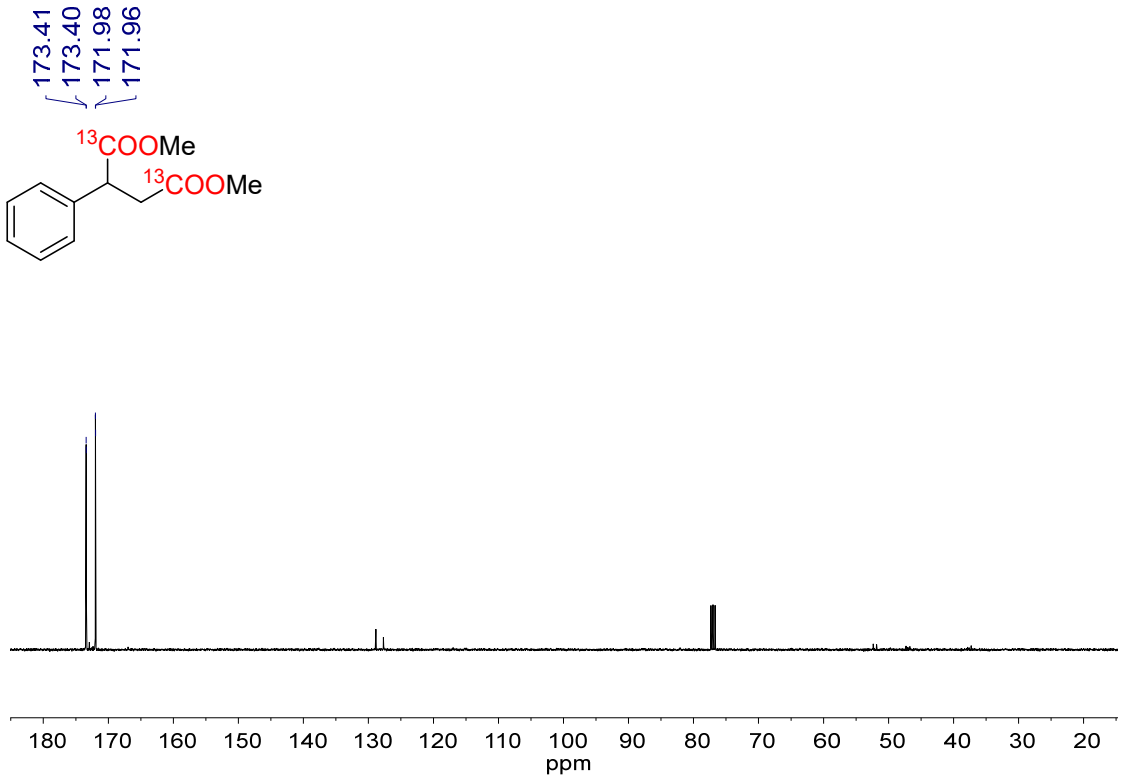
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 3ak



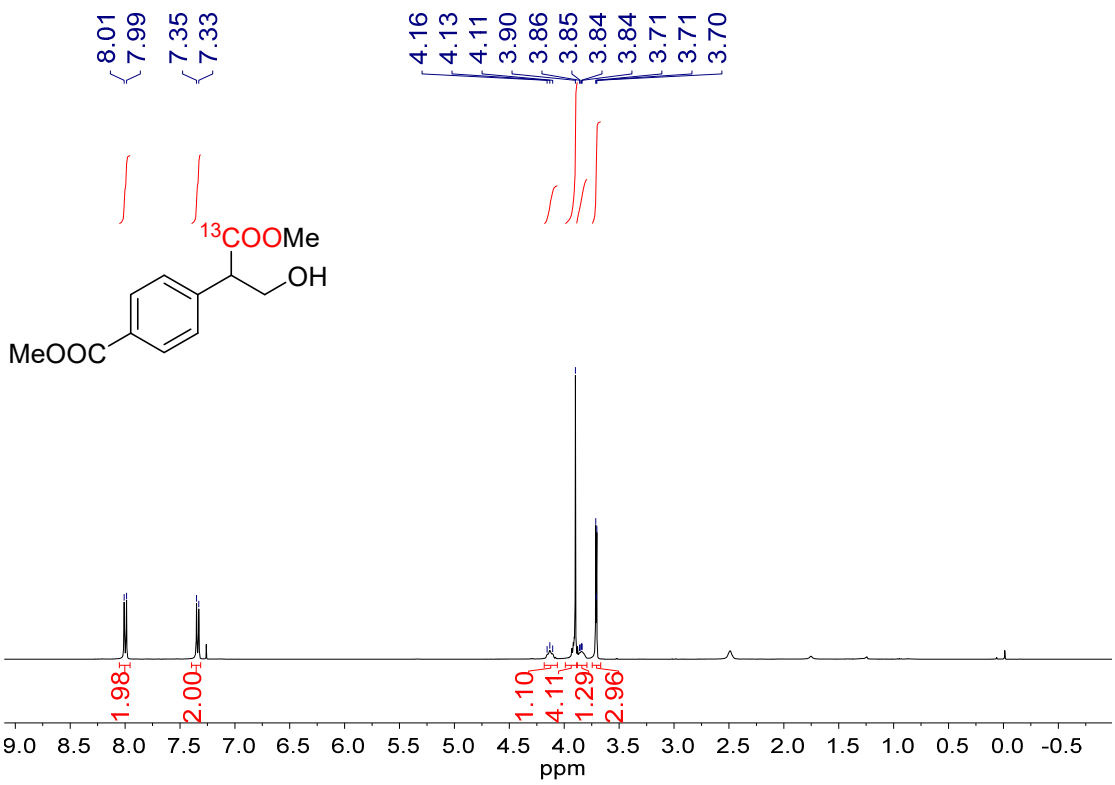
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 3ak



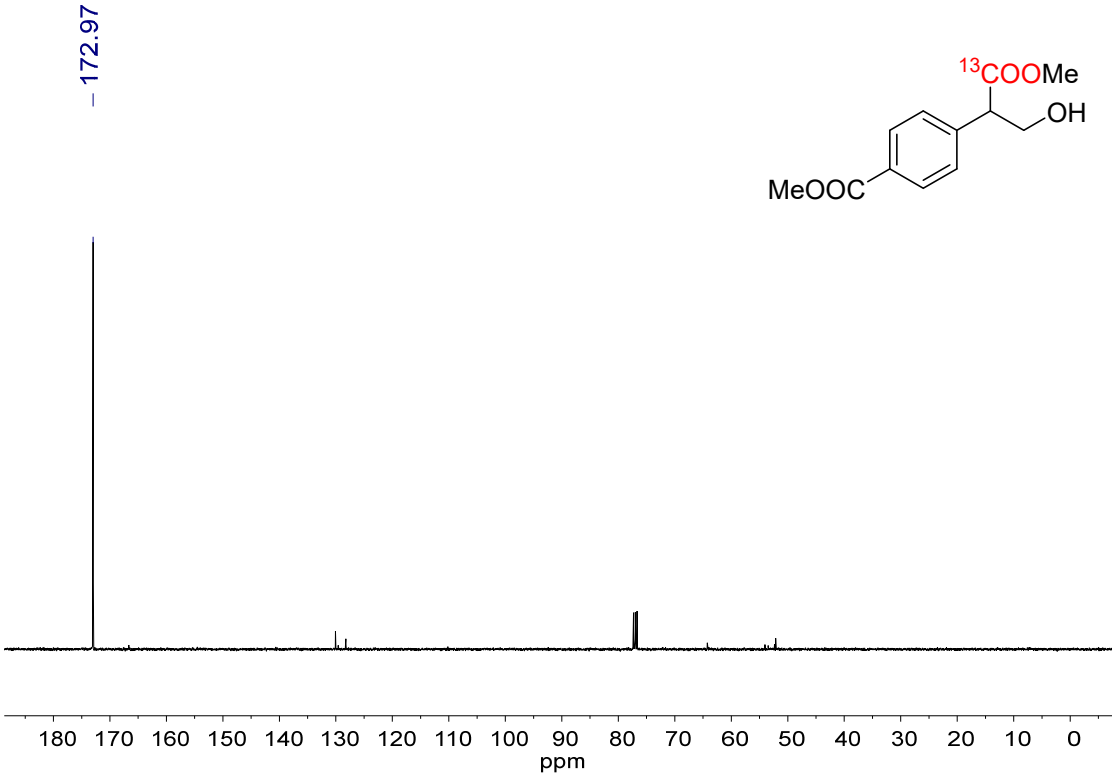
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 2a”



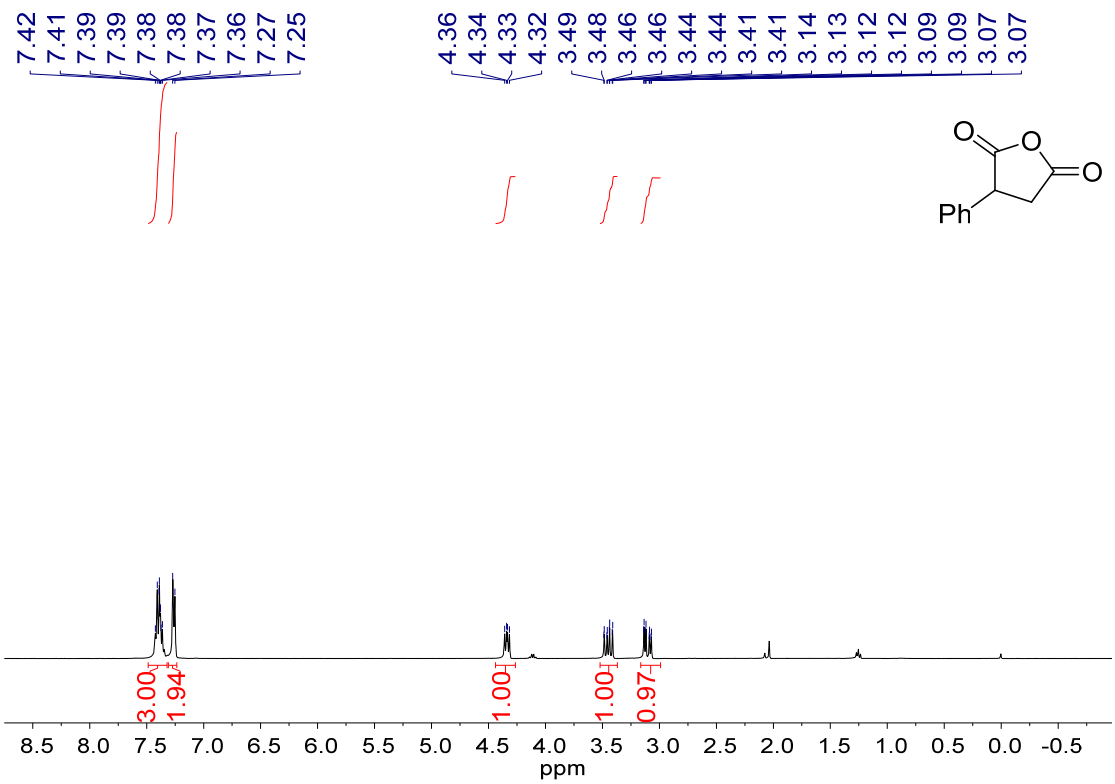
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 2a”



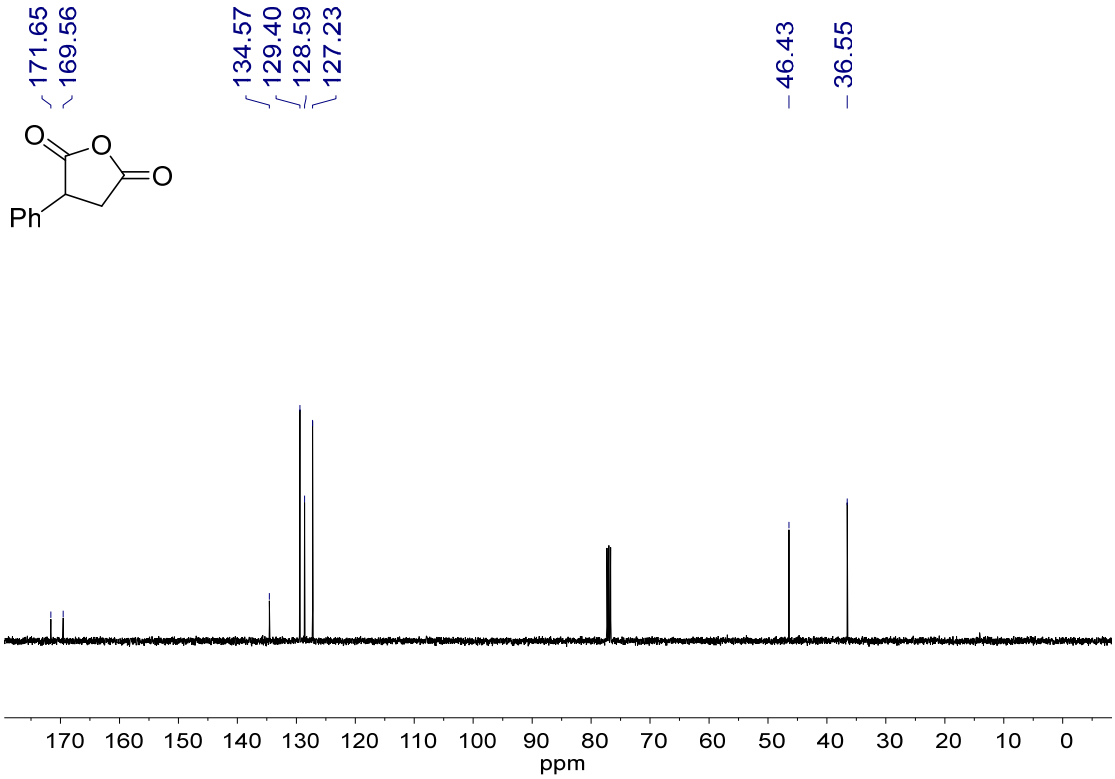
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 3q'



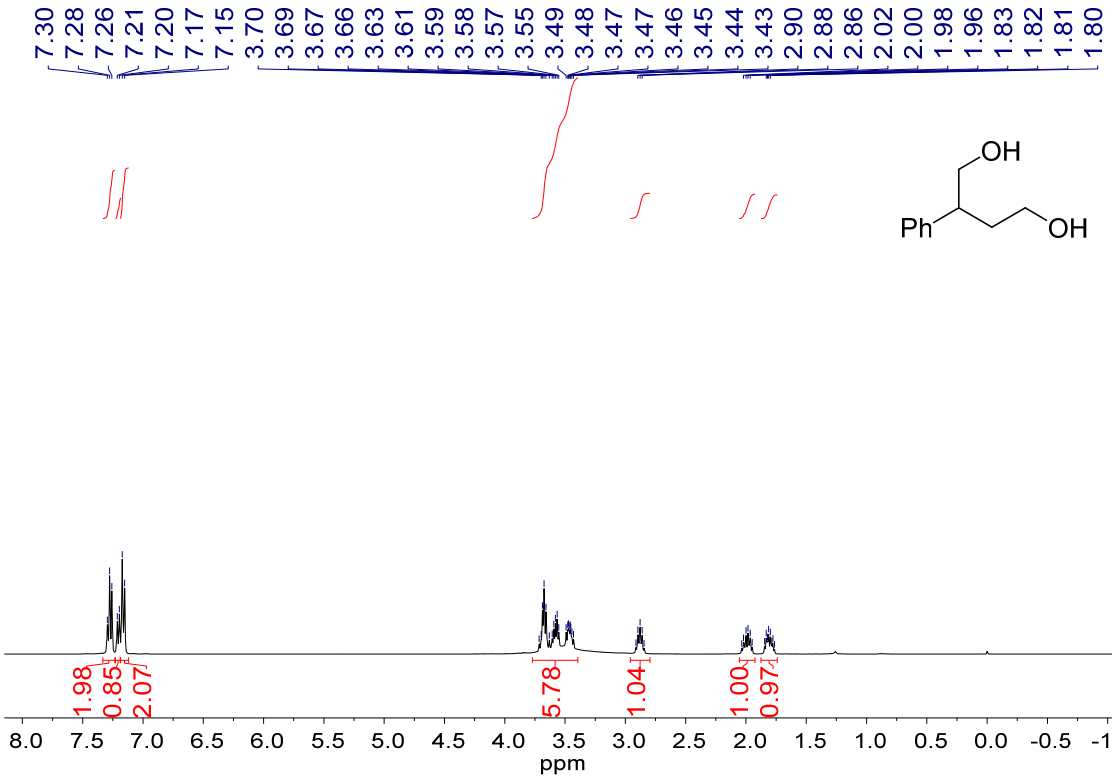
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 3q'



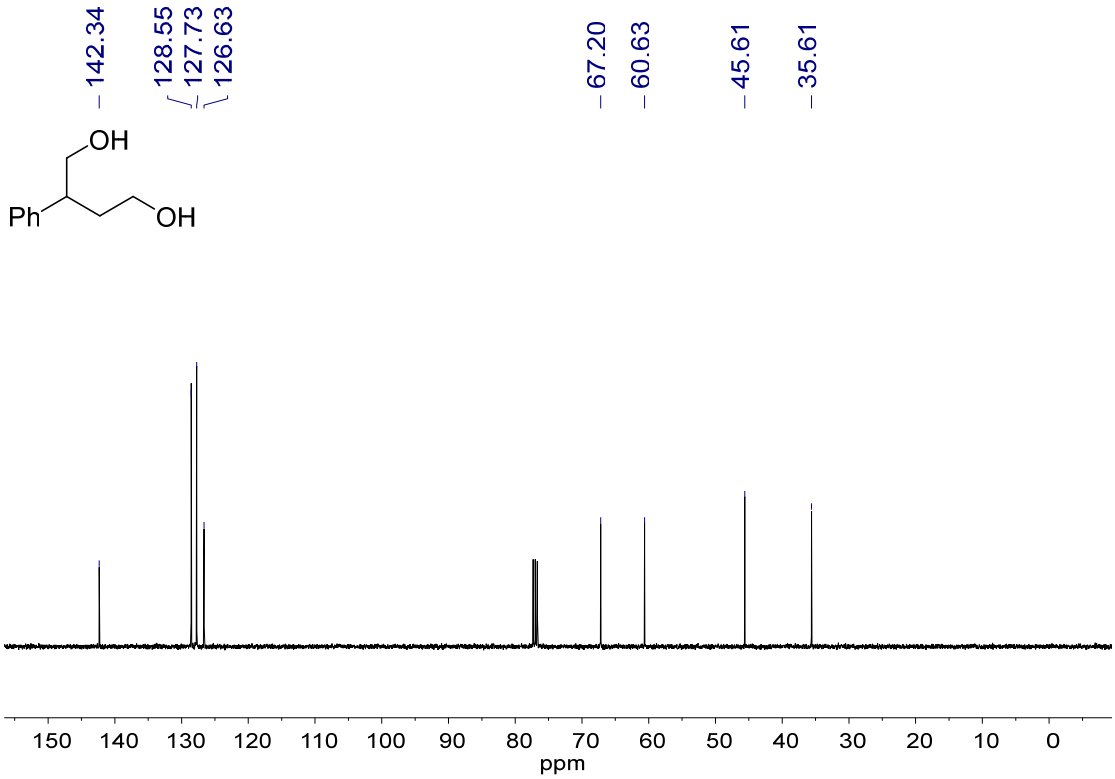
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 4a



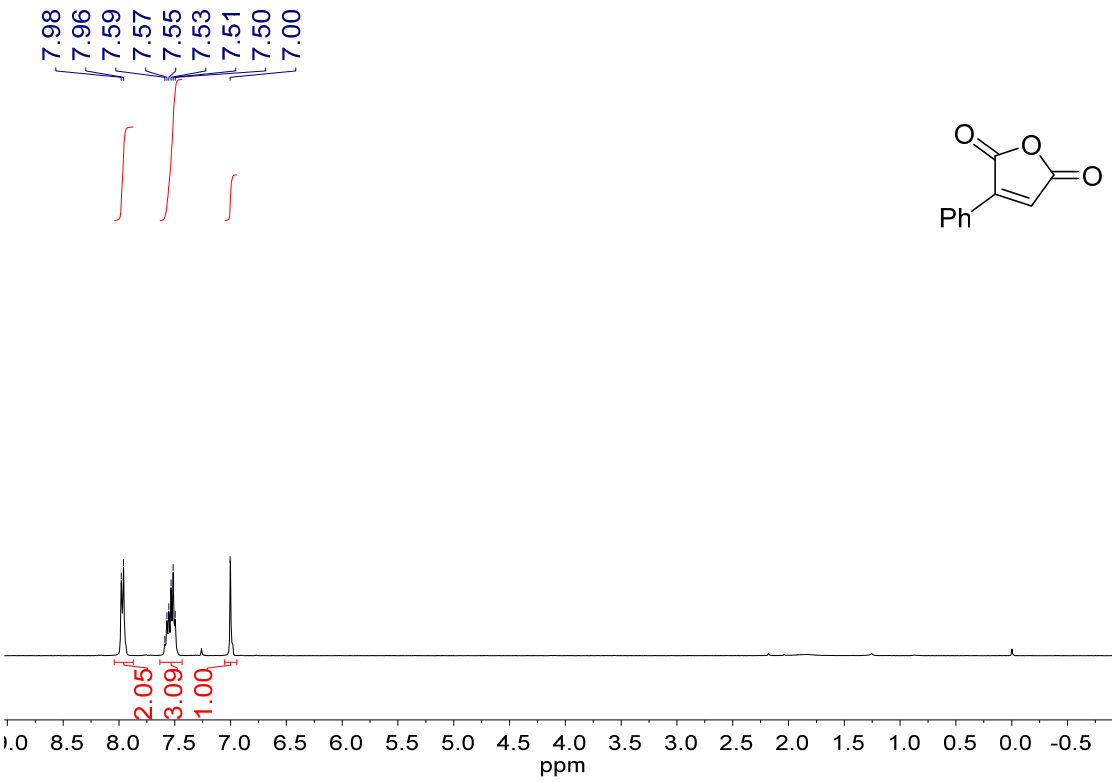
^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 4a



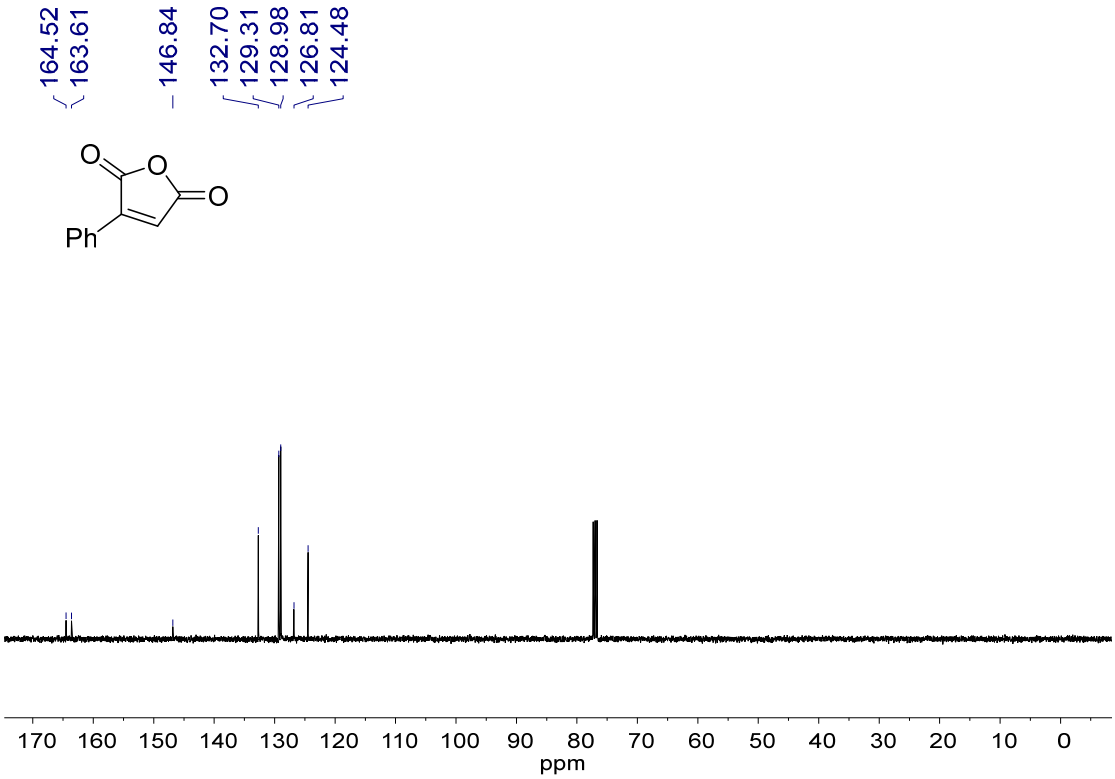
^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 5a



^{13}C{^{1}H} NMR spectrum (101 MHz, Chloroform-*d*) of 5a



^{1}H NMR spectrum (400 MHz, Chloroform-*d*) of 6a



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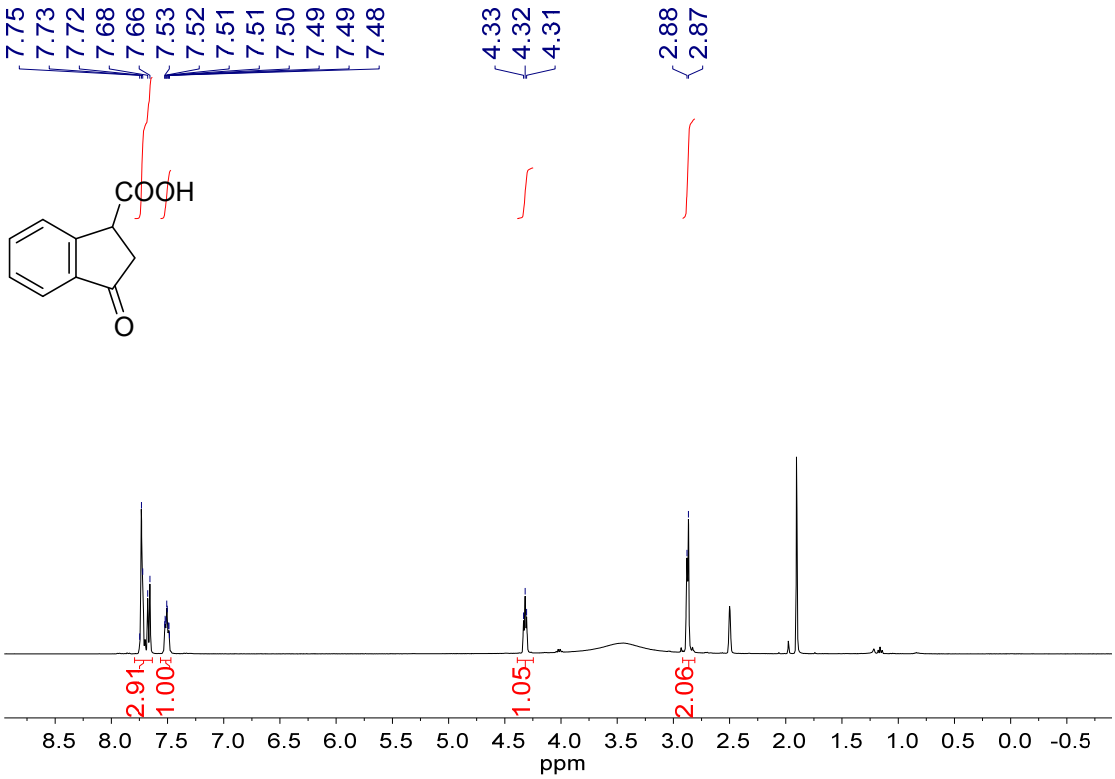
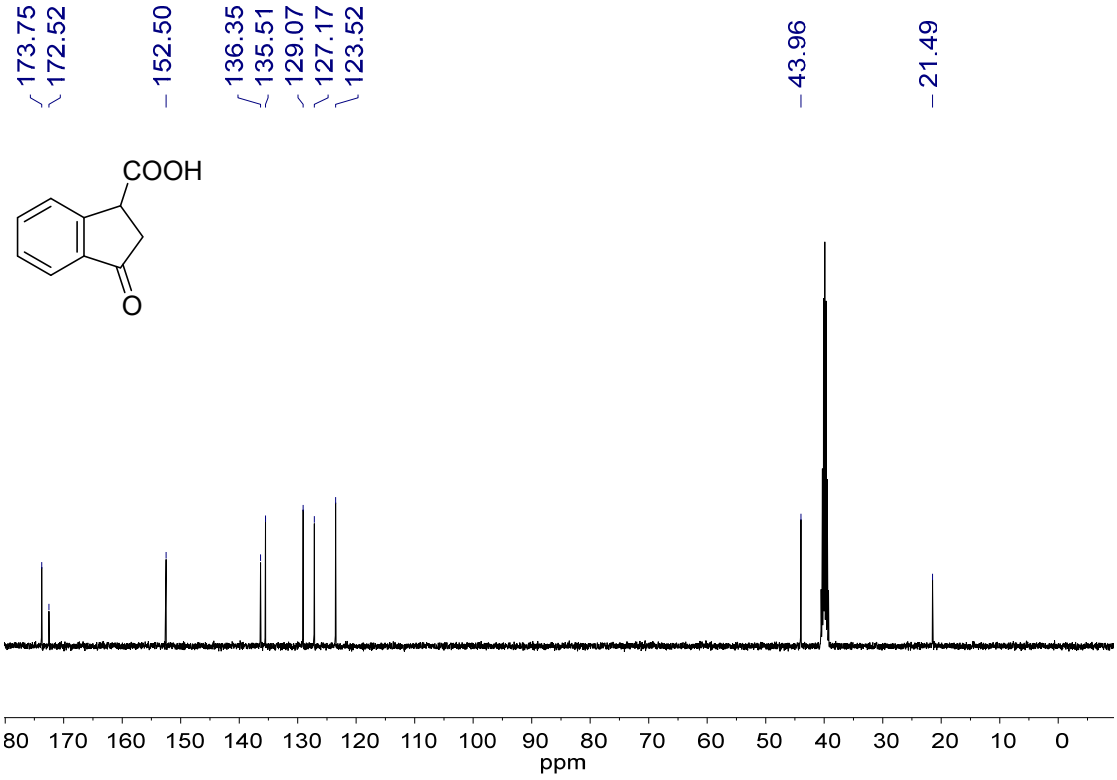


^{1}H NMR spectrum (400 MHz, DMSO-d_6) of 7a



^{13}C{^{1}H} NMR spectrum (101 MHz, DMSO-d_6) of 7a

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