Supplementary Information for:

Highly Enantioselective Ni-Catalyzed Asymmetric Hydrogenation of *β*, *β*-Disubstituted Acrylic Acids

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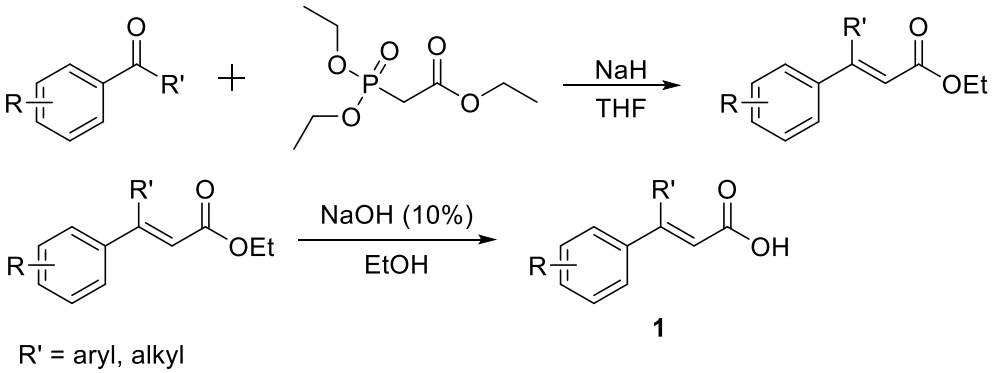
1. Experimental Section

General Information: All the air or moisture sensitive reactions and manipulations were performed by using standard Schlenk techniques and in a nitrogen-filled glovebox. ^{1}H NMR and ^{13}C NMR spectra were recorded on JEOL JNM-ECX600P and JNM-ECS600 (400 MHz or 600 MHz) spectrometers. ($CDC1_3$ was the solvent used for the NMR analysis, with TMS as the internal standard). Optical rotation was determined using Autopol III Automatic polarimeter (Rudolph research Analyical). HPLC analysis was conducted on Agilent 1260 series instrument. SFC analysis was conducted on Agilent 1260 series instrument. GC analysis was conducted on Agilent 7890 series instrument. HRMS were recorded on a Waters LCT Premier XE mass spectrometer with TOF.

2. General procedure for the synthesis of substrates.[1]

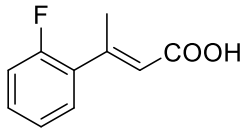


NaH (45 mmol, 1.5 eq, 70% in mineral oil) was placed in an oven-dried 250 mL three-neck round bottom flask. Then THF (50 mL) was added dropwise with stirring under nitrogen, and the mixture was cooled to 0 °C. Triethyl phosphonoacetate (45 mmol, 1.5 eq) was added dropwise to the suspension at 0 °C, followed by stirring for 1 h at room temperature. Ketone (30 mmol, 1.0 eq) in THF (50 mL) was added dropwise. After that，the solution was stirred at room temperature until no starting material was detected by TLC. The reaction was quenched by the dropwise addition of a saturated saturated aqueous NH_4Cl solution (50 mL) and the product extracted with EtOAc, dried over Na_2SO_4, filtered and concentrated in vacuo. The crude residue was purified by flash chromatography (EtOAc: Petroleum ether = 1:50 to 1:20) to give the (*E*)-ester or (*Z*)-ester as a colourless oil (yield: 18-82%).

$NaOH_{aq}$ (10%, 20 mL) was added to a solution of the corresponding (*E*)-ester or (*Z*)-ester (5.0 mmol, 1.0 eq) in EtOH (20 mL) at room temperature. The reaction was stirred at room temperature until no starting material was detected by TLC, after which the volatiles were removed in vacuo and the residue slowly acidified with 2 M HCl_{aq}. The product was extracted with ethyl acetate, dried over Na_2SO_4 and concentrated in vacuo. The crude product was recrystallised from boiling ethyl acetate and hexane to give **1** as white needles.

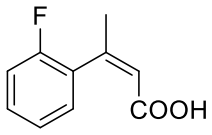
3. NMR and HRMS data of substrates

(*E*)-3-(2-fluorophenyl)but-2-enoic acid (**1a**)



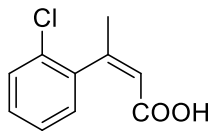
Purification by recrystallization (PE : EA = 5 : 1) afforded the product as a white solid; 0.87 g, yield: 97%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.37-7.27 (m, 2H), 7.17-7.11 (m, 1H), 7.12-7.06 (m, 1H), 6.07-6.04 (m, 1H), 2.62-2.43 (m, 3H). MP: 91-93 ℃. The analytical data are consistent with the literature.[1]

(*Z*)-3-(2-fluorophenyl)but-2-enoic acid (**1b**)



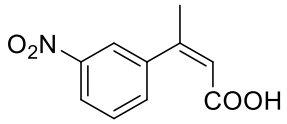
Purification by recrystallization (PE : EA = 5 : 1) afforded the product as a white solid; 0.85 g, yield: 95%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.31-7.21 (m, 1H), 7.15-7.08 (m, 2H), 7.06-6.96 (m, 1H), 6.01-5.96 (m, 1H), 2.18 (s, 3H). ^{13}C NMR (150 MHz, Chloroform-*d*) δ 170.5, 158.4 (d, *J* = 246.3 Hz), 152.1, 129.6 (d, *J* = 8.4 Hz), 128.7 (d, *J* = 3.6 Hz), 128.1 (d, *J* = 16.2 Hz), 123.8 (d, *J* = 3.0 Hz), 119.5, 115.5 (d, *J* = 22.1 Hz), 26.7. MP: 124-126 ℃. The analytical data are consistent with the literature.[2]

(*Z*)-3-(2-chlorophenyl)but-2-enoic acid (**1c**)



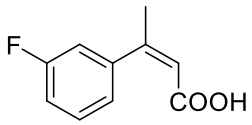
Purification by recrystallization (PE : EA = 5 : 1) afforded the product as a white solid; 0.95 g, yield: 97%; ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.38-7.33 (m, 1H), 7.24-7.19 (m, 2H), 7.05-7.02 (m, 1H), 5.97 (q, *J* = 1.3 Hz, 1H), 2.16 (d, *J* = 1.5 Hz, 3H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 170.3, 156.2, 139.9, 130.5, 129.4, 128.7, 127.8, 126.7, 119.2, 26.5. MP: 96-98 °C. The analytical data are consistent with the literature.[3]

(*Z*)-3-(3-nitrophenyl)but-2-enoic acid (**1d**)



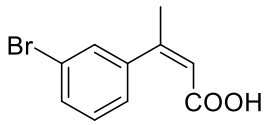
Purification by recrystallization (PE : EA = 5 : 1) afforded the product as a white solid; 0.98 g, yield: 95%; ^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.179-8.14 (m, 1H), 8.04-8.03 (m, 1H), 7.51-7.48 (m, 2H), 5.97 (d, *J* = 1.4 Hz, 1H), 2.21 (d, *J* = 1.4 Hz, 3H). ^{13}C NMR (150 MHz, Chloroform-*d*) δ 169.9, 155.9, 148.0, 141.9, 133.2, 129.1, 122.9, 122.0, 118.6, 27.4. MP: 106-108 ℃. APCI-HRMS calcd for $C_{10}H_{10}NO_4^+$ [M+H$^+$] 208.0304, found 208.0308.

(*Z*)-3-(3-fluorophenyl)but-2-enoic acid (**1e**)



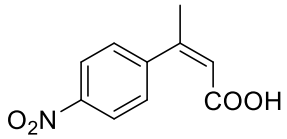
Purification by recrystallization (PE : EA = 5 : 1) afforded the product as a white solid; 0.85 g, yield: 95%; ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.31-7.26 (m, 1H), 7.02-6.97 (m, 1H), 6.97-6.94 (m, 1H), 6.91-6.87 (m, 1H), 5.90 (q, *J* = 1.4 Hz, 1H), 2.17 (d, *J* = 1.5 Hz, 3H). ^{13}C NMR (150 MHz, Chloroform-*d*) δ 170.8, 162.5 (d, *J* = 245.9 Hz), 156.9, 142.5 (d, *J* = 8.2 Hz), 129.7 (d, *J* = 8.1 Hz), 122.7, 117.7, 114.9 (d, *J* = 21.3 Hz), 114.1 (d, *J* = 22.6 Hz), 27.5. MP: 98-100 °C. The analytical data are consistent with the literature.[2]

(*Z*)-3-(3-bromophenyl)but-2-enoic acid (**1f**)



Purification by recrystallization (PE : EA = 5 : 1) afforded the product as a white solid; 1.1 g, yield: 94%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.45-7.41 (m, 1H), 7.32 (t, *J* = 1.8 Hz, 1H), 7.20 (t, *J* = 7.8 Hz, 1H), 7.14-7.08 (m, 1H), 5.91-5.87 (m, 1H), 2.16 (d, *J* = 1.4 Hz, 3H). ^{13}C NMR (150 MHz, Chloroform-*d*) δ 170.8, 156.7, 142.4, 131.0, 129.8, 129.7, 125.7, 122.1, 117.8, 27.6. MP: 112-114 °C. The analytical data are consistent with the literature.[4]

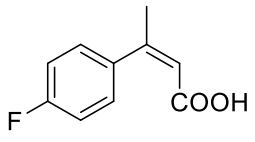
(*Z*)-3-(4-nitrophenyl)but-2-enoic acid (**1g**)



Purification by recrystallization (PE : EA = 5 : 1) afforded the product as a white solid; 0.99 g, yield: 96%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.22-8.15 (m, 2H), 7.36 -7.30 (m, 2H), 5.98 (d, *J* = 1.4 Hz, 1H), 2.20 (d, *J* = 1.7 Hz, 3H). ^{13}C NMR (150 MHz, Chloroform-*d*) δ 169.4, 156.3, 147.5, 147.4, 127.8, 123.5, 118.4, 27.3. MP: 156-157 °C. APCI-HRMS

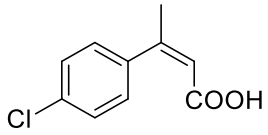
calcd for $C_{10}H_{10}NO_4^+$ [M+H$^+$] 208.0304, found 208.0308.

(*Z*)-3-(4-fluorophenyl)but-2-enoic acid (**1h**)



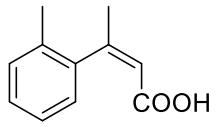
Purification by recrystallization (PE : EA = 5 : 1) afforded the product as a white solid; 0.83 g, yield: 93%; ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.21-7.14 (m, 2H), 7.04-6.96 (m, 2H), 5.92-5.87 (m, 1H), 2.17 (d, *J* = 1.2 Hz, 3H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 171.0, 162.6 (d, *J* = 246.8 Hz), 157.4, 136.0 (d, *J* = 3.2 Hz), 128.9 (d, *J* = 8.2 Hz), 117.2, 115.1 (d, *J* = 21.5 Hz), 27.7. MP: 99-101 °C. The analytical data are consistent with the literature.[2]

(*Z*)-3-(4-chlorophenyl)but-2-enoic acid (**1i**)



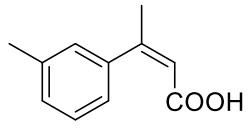
Purification by recrystallization (PE : EA = 5 : 1) afforded the product as a white solid; 0.93 g, yield: 95%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.32-7.28 (m, 2H), 7.16-7.10 (m, 2H), 5.90 (q, *J* = 1.4 Hz, 1H), 2.17 (d, *J* = 1.4 Hz, 3H). ^{13}C NMR (150 MHz, Chloroform-*d*) δ 171.1, 157.2, 138.6, 134.1, 128.4, 128.3, 117.5, 27.5. MP: 111-113 °C. The analytical data are consistent with the literature.[5]

(*Z*)-3-(*o*-tolyl)but-2-enoic acid (**1j**)



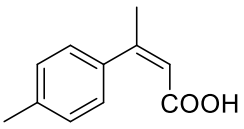
Purification by recrystallization (PE : EA = 5 : 1) afforded the product as a white solid; 0.83g, yield: 95%; ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.19-7.15 (m, 3H), 6.97-6.91 (m, 1H), 5.96-5.93 (m, 1H), 2.19 (s, 3H), 2.11 (d, *J* = 1.4 Hz, 3H). ^{13}C NMR (150 MHz, Chloroform-*d*) δ 169.6, 158.9, 140.6, 133.4, 129.9, 127.4, 125.8, 125.7, 118.1, 27.6, 19.1. MP: 134-136 °C. The analytical data are consistent with the literature.[6]

(*Z*)-3-(*m*-tolyl)but-2-enoic acid (**1k**)



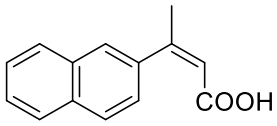
Purification by recrystallization (PE : EA = 5 : 1) afforded the product as a white solid; 0.81 g, yield: 93%; ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.22 (t, *J* = 7.7 Hz, 1H), 7.12 (d, 1H), 7.00 (d, *J* = 7.8 Hz, 2H), 5.89-5.85 (m, 1H), 2.34 (s, 3H), 2.18 (d, *J* = 1.3 Hz, 3H). ^{13}C NMR (150 MHz, Chloroform-*d*) δ 170.7, 158.3, 140.2, 137.7, 128.9, 127.9, 127.5, 124.1, 116.8, 27.7, 21.5. MP: 90-92 °C. The analytical data are consistent with the literature.[6]

(*Z*)-3-(*p*-tolyl)but-2-enoic acid (**1l**)



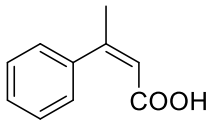
Purification by recrystallization (PE : EA = 5 : 1) afforded the product as a white solid; 0.83 g, yield: 95%; ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.17-7.08 (m, 4H), 5.91-5.81 (m, 1H), 2.35 (s, 3H), 2.18 (d, *J* = 1.2 Hz, 3H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 170.6, 158.2, 138.1, 137.2, 128.8, 127.0, 116.6, 27.6, 21.4. MP: 108-110 °C. The analytical data are consistent with the literature.[6]

(*Z*)-3-(naphthalen-2-yl)but-2-enoic acid (**1m**)



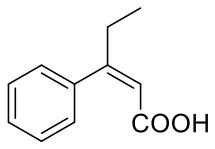
Purification by recrystallization (PE : EA = 5 : 1) afforded the product as a white solid; 0.98 g, yield: 93%; ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.83-7.75 (m, 3H), 7.67-7.64 (m, 1H), 7.49-7.44 (m, 2H), 7.31 (dd, *J* = 8.4, 1.7 Hz, 1H), 5.97 (d, *J* = 1.4 Hz, 1H), 2.27 (d, *J* = 1.4 Hz, 3H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 170.1, 158.2, 137.8, 133.0, 133.0, 128.3, 127.8, 127.6, 126.3, 126.2, 125.8, 125.5, 117.2, 27.8. MP: 129-131 °C. The analytical data are consistent with the literature.[6]

(*Z*)-3-phenylbut-2-enoic acid (**1n**)



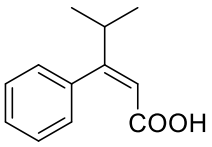
Purification by recrystallization (PE : EA = 5 : 1) afforded the product as a white solid; 0.77 g, yield: 96%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.35-7.29 (m, 3H), 7.22-7.16 (m, 2H), 5.88 (q, *J* = 1.3 Hz, 1H), 2.19 (d, *J* = 1.4 Hz, 3H). ^{13}C NMR (150 MHz, Chloroform-*d*) δ 171.1, 158.3, 140.3, 128.1, 128.1, 126.9, 117.0, 27.7. MP: 120-122 ℃. MP: 128-130 ℃. The analytical data are consistent with the literature.[7]

(*Z*)-3-phenylpent-2-enoic acid (**1o**)



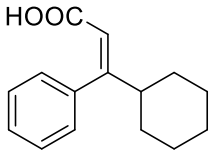
Purification by recrystallization (PE : EA = 5 : 1) afforded the product as a white solid; 0.82 g, yield: 95%; ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.34-7.27 (m, 3H), 7.15-7.10 (m, 2H), 5.83 (t, *J* = 1.3 Hz, 1H), 2.49-2.40 (m, 2H), 1.02 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 171.5, 163.8, 139.9, 128.0, 127.9, 127.1, 115.6, 33.9, 12.2. MP: 90-92 ℃. The analytical data are consistent with the literature.[7]

(*Z*)-4-methyl-3-phenylpent-2-enoic acid (**1p**)



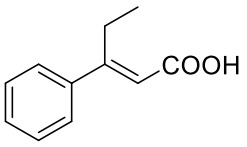
Purification by recrystallization (PE : EA = 5 : 1) afforded the product as a white solid; 0.93 g, yield: 93%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.35-7.27 (m, 3H), 7.10-7.05 (m, 2H), 5.82 (d, *J* = 0.9 Hz, 1H), 2.72-2.59 (m, 1H), 1.06 (d, *J* = 6.8 Hz, 6H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 167.9, 139.7, 127.8, 127.6, 127.2, 115.0, 37.7, 21.2. MP: 88-90 °C. The analytical data are consistent with the literature.[6]

(*Z*)-3-cyclohexyl-3-phenylacrylic acid (**1q**)



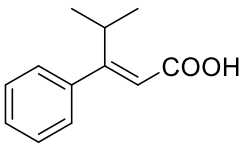
Purification by recrystallization (PE : EA = 5 : 1) afforded the product as a white solid; 1.1 g, yield: 96%; ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.34-7.27 (m, 3H), 7.10-7.02 (m, 2H), 5.81-5.75 (m, 1H), 2.28-2.21 (m, 1H), 1.80-1.73 (m, 4H), 1.69-1.63 (m, 1H), 1.28-1.20 (m, 2H), 1.18-1.10 (m, 3H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 171.8, 167.1, 140.1, 127.8, 127.5, 127.2, 115.3, 47.8, 31.7, 26.5, 26.1. MP: 140-142 °C. The analytical data are consistent with the literature.[8]

(*E*)-3-phenylpent-2-enoic acid (**1r**)



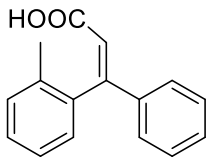
Purification by recrystallization (PE : EA = 5 : 1) afforded the product as a white solid; 0.89 g, yield: 94%; ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.48-7.45 (m, 2H), 7.41-7.38 (m, 3H), 6.05 (s, 1H), 3.13 (q, *J* = 7.5 Hz, 2H), 1.09 (t, *J* = 7.5 Hz, 3H). MP: 95-97 °C. The analytical data are consistent with the literature.[1]

(*E*)-4-methyl-3-phenylpent-2-enoic acid (**1s**)



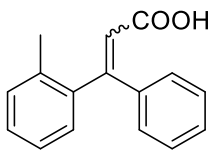
Purification by recrystallization (PE : EA = 5 : 1) afforded the product as a white solid; 0.81 g, yield: 93%; ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.36-7.32 (m, 3H), 7.23-7.20 (m, 2H), 5.75 (s, 1H), 4.19-4.12 (m, 1H), 1.10 (dd, *J* = 7.0, 1.2 Hz, 6H). MP: 97-97 °C. The analytical data are consistent with the literature.[1]

(*Z*)-3-phenyl-3-(*o*-tolyl)acrylic acid (**1t**)



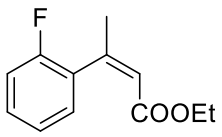
Purification by recrystallization (PE : EA = 3 : 1) afforded the product as a white solid; 1.0 g, yield: 91%; ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.38-7.27 (m, 6H), 7.22 (d, *J* = 7.4 Hz, 2H), 7.06 (d, *J* = 7.4 Hz, 1H), 6.49 (s, 1H), 2.06 (s, 3H). ^{13}C NMR (150 MHz, Chloroform-*d*) δ 170.6, 158.4, 139.3, 138.1, 135.4, 130.1, 129.9, 128.7, 128.5, 128.1, 127.7, 125.6, 116.9, 19.7. MP: 110-112 °C. APCI-HRMS calcd for $C_{16}H_{15}O_2^+$ [M+H$^+$] 239.1067, found 239.1070.

3-phenyl-3-(*o*-tolyl)acrylic acid (**1u**)



Purification by recrystallization (PE : EA = 3 : 1) afforded the product as a white solid (*E*:*Z* =8:1); 1.1 g, yield: 93%; ^{1}H NMR (400 MHz, Chloroform-*d*; *E* isomer) δ 7.35-7.24 (m, 6H), 7.21-7.17 (m, 2H), 7.15 (d, *J* = 7.3 Hz, 1H), 6.00 (s, 1H), 2.05 (s, 3H). ^{13}C NMR (100 MHz, Chloroform-*d*; *E* isomer) δ 171.4, 159.6, 141.9, 138.3, 136.1, 130.8, 129.6, 129.4, 128.9, 128.8, 127.9, 125.83, 119.0, 20.4. MP: 132-134 °C. APCI-HRMS calcd for $C_{16}H_{15}O_2^+$ [M+H$^+$] 239.1067, found 239.1070.

ethyl (*Z*)-3-(2-fluorophenyl)but-2-enoate (**1v**)



Purification by column chromatography (PE : EA = 50:1 to 20:1) afforded the product as a colourless oil; 2.1 g, yield: 34%; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.34-7.23 (m, 1H), 7.14-7.10 (m, 2H), 7.08-7.02 (m, 1H), 6.01 (q, *J* = 1.2 Hz, 1H), 4.00 (q, *J* = 7.1 Hz, 2H), 2.17 (d, *J* = 1.4 Hz, 3H), 1.08 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (150 MHz, Chloroform-*d*) δ 165.4, 158.6 (d, *J* = 245.8 Hz), 149.3, 129.4 (d, *J* = 8.1 Hz), 128.8 (d, *J* = 3.3 Hz), 128.7 (d, *J* = 16.6 Hz), 123.8 (d, *J* = 2.7 Hz), 120.2, 115.4 (d, *J* = 22.3 Hz), 59.9, 26.4, 14.0. The analytical data are consistent with the literature.[9]

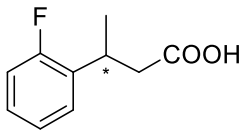
4. General procedure for asymmetric hydrogenation of 1.

A stock solution was made by mixing $Ni(OAc)_2 \cdot H_2O$ with (*S*,*S*)-Ph-BPE in a 1:1.1 molar ratio in solvent (CF_3CH_2OH) at room temperature for 12 hours in a nitrogen-filled glovebox. An aliquot of the catalyst solution (1.0 mL, 0.00125 mmol) was transferred by syringe into the vials charged with different substrates (0.125 mmol for

each) and added additive HOAc. The vials were subsequently transferred into an autoclave which hydrogen gas was charged. The reaction was then stirred under H_2 (80 atm) at r.t. for 12-24 h. The hydrogen gas was released slowly and carefully. The solution was passed through a short column of silica gel to remove the metal complex. The conversion of products were determined by GC or ^{1}H NMR analysis. The crude products were concentrated and purified by flash column chromatography and the ee values were determined by HPLC, SFC analysis on a chiral stationary phase.

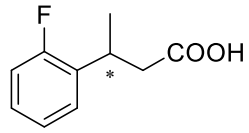
5. NMR, GC or HPLC, optical rotation and HRMS Data of compound 2.

(+)-3-(2-fluorophenyl)butanoic acid (**2a**)



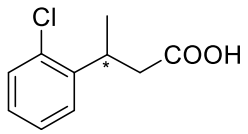
Purification by flash column chromatography (silica gel, PE : EA = 1 : 1) afforded the product as light yellow oil; 22.0 mg, yield: 97%; 93% ee; $[\alpha]_D^{20}$ = +13.2 (c = 1.1, CH_2Cl_2); SFC conditions (Lux 5u Amylose-1, column temperature: 37.0 ℃, MeOH/CO_2 = 5/95, flow rate = 3.0 mL/min, l = 210 nm) t_R = 2.5 min (major), 2.8 min (minor); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.69 (s, 1H), 7.25-7.17 (m, 2H), 7.11-7.05 (m, 1H), 7.05-6.99 (m, 1H), 3.75-3.46 (m, 1H), 2.84-2.51 (m, 2H), 1.34 (d, *J* = 7.0 Hz, 3H). The analytical data are consistent with the literature.[1]

(-)-3-(2-fluorophenyl)butanoic acid (**2b**)



Purification by flash column chromatography (silica gel, PE : EA = 1 : 1) afforded the product as light yellow oil; 22.3 mg, yield: 98%; 99% ee; $[\alpha]_D^{20}$ = -22.9 (c = 1.1, CH_2Cl_2); SFC conditions (Lux 5u Amylose-1, column temperature: 37.0 ℃, MeOH/CO_2 = 5/95, flow rate = 3.0 mL/min, l = 210 nm) t_R = 2.8 min (minor), 3.2 min (major); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.69 (s, 1H), 7.25-7.17 (m, 2H), 7.11-7.05 (m, 1H), 7.05-6.99 (m, 1H), 3.75-3.46 (m, 1H), 2.84-2.51 (m, 2H), 1.34 (d, *J* = 7.0 Hz, 3H). The analytical data are consistent with the literature.[1]

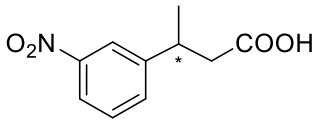
(-)-3-(2-chlorophenyl)butanoic acid (**2c**)



Purification by flash column chromatography (silica gel, PE : EA = 1 : 1) afforded the product as light yellow oil; 23.7 mg, yield: 96%; 90% ee; $[\alpha]_D^{20}$ = -17.1 (c = 1.2, CH_2Cl_2); SFC conditions

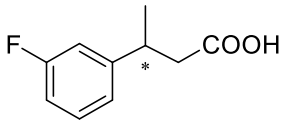
(Lux 5u Amylose-1, column temperature: 37.0 °C, MeOH/CO_2 = 5/95, flow rate = 3.0 mL/min, l = 210 nm) t_R = 2.3 min (major), 2.6 min (minor); ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.35 (d, *J* = 7.9 Hz, 1H), 7.25-7.21 (m, 2H), 7.16-7.11 (m, 1H), 3.86-3.70 (m, 1H), 2.96-2.37 (m, 2H), 1.31 (d, *J* = 6.7 Hz, 3H). The analytical data are consistent with the literature.[1]

(-)-3-(3-nitrophenyl)butanoic acid (**2d**)



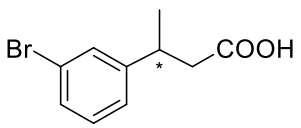
Purification by flash column chromatography (silica gel, PE : EA = 1 : 1) afforded the product as light yellow soild; 25.1 mg, yield: 96%; 94% ee; $[\alpha]_D^{20}$ = -22.9 (c = 1.3, CH_2Cl_2); SFC conditions (Lux 5u Amylose-1, column temperature: 37.0 °C, MeOH/CO_2 = 5/95, flow rate = 3.0 mL/min, l = 210 nm) t_R = 14.8 min (minor), 16.4 min (major); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.14-8.04 (m, 2H), 7.61-7.53 (m, 1H), 7.48 (t, *J* = 7.8 Hz, 1H), 3.40 (q, *J* = 7.2 Hz, 1H), 2.78-2.58 (m, 2H), 1.37 (d, *J* = 7.0 Hz, 3H). MP: 163-165 °C. The analytical data are consistent with the literature.[1]

(-)-3-(3-fluorophenyl)butanoic acid (**2e**)



Purification by flash column chromatography (silica gel, PE : EA = 1 : 1) afforded the product as light yellow oil; 22.3 mg, yield: 98%; 98% ee; $[\alpha]_D^{20}$ = -27.0 (c = 1.1, CH_2Cl_2); The hydrogenated product was dissolved in CH_2Cl_2, and then aniline, EDC, and DMAP were added at 0 °C. The solution was stirred for 4 h. The residue was purified by chromatography; The ee values of the amides were determined by SFC analysis on a chiral stationary phase. SFC conditions (Lux 5u Cellulose-1, column temperature: 37.0 °C, MeOH/CO_2 = 10/90, flow rate = 3.0 mL/min, l = 210 nm) t_R = 5.0 min (major), 5.5 min (minor); ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.24 (d, *J* = 6.3 Hz, 1H), 6.99 (d, *J* = 7.6 Hz, 1H), 6.92-6.88 (m, 2H), 3.30-3.21 (m, 1H), 2.66-2.51 (m, 2H), 1.30 (d, *J* = 6.9 Hz, 3H). The analytical data are consistent with the literature.[1]

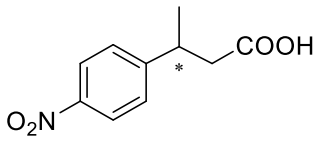
(-)-3-(3-bromophenyl)butanoic acid (**2f**)



Purification by flash column chromatography (silica gel, PE : EA = 1 : 1) afforded the product as light yellow oil; 29.3 mg, yield: 97%; 96% ee; $[\alpha]_D^{20}$ = -25.1 (c = 1.5, CH_2Cl_2); The

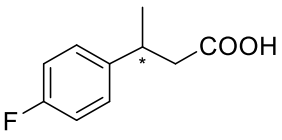
hydrogenated product was dissolved in CH_2Cl_2, and then aniline, EDC, and DMAP were added at 0 °C. The solution was stirred for 4 h. The residue was purified by chromatography. The ee values of the amides were determined by SFC analysis on a chiral stationary phase; SFC conditions (Lux 5u Cellulose-3, column temperature: 37.0 ℃, MeOH/CO_2 = 15/85, flow rate = 3.0 mL/min, l = 210 nm) t_R = 5.1 min (major), 5.4 min (minor); ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.40-7.31 (m, 2H), 7.21-7.11 (m, 2H), 3.24 (s, 1H), 2.88-2.46 (m, 2H), 1.31 (d, *J* = 6.6 Hz, 3H). The analytical data are consistent with the literature.[1]

(-)-3-(4-nitrophenyl)butanoic acid (**2g**)



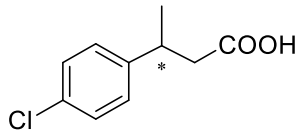
Purification by flash column chromatography (silica gel, PE : EA = 1 : 1) afforded the product as light yellow soild; 25.6 mg, yield: 98%; 94% ee; $[\alpha]_D^{20}$ = -39.2 (c = 1.3, CH_2Cl_2); The hydrogenated product was dissolved in CH_2Cl_2, and then aniline, EDC, and DMAP were added at 0 °C. The solution was stirred for 4 h. The residue was purified by chromatography. The ee values of the amides were determined by SFC analysis on a chiral stationary phase; SFC conditions (Lux 5u Cellulose-3, column temperature: 37.0 ℃, MeOH/CO_2 = 20/80, flow rate = 3.0 mL/min, l = 210 nm) t_R = 5.6 min (major), 6.1 min (minor); ^{1}H NMR (600 MHz, Chloroform-*d*) δ 8.16 (d, *J* = 8.4 Hz, 2H), 7.39 (d, *J* = 8.4 Hz, 2H), 3.39 (q, *J* = 6.7 Hz, 1H), 2.78-2.55 (m, 2H), 1.35 (d, *J* = 6.8 Hz, 3H). MP: 123-125 °C. The analytical data are consistent with the literature.[1]

(-)-3-(4-fluorophenyl)butanoic acid (**2h**)



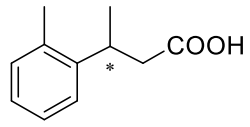
Purification by flash column chromatography (silica gel, PE : EA = 1 : 1) afforded the product as light yellow oil; 22.3 mg, yield: 98%; 93% ee; $[\alpha]_D^{20}$ = -33.5 (c = 1.1, $CHCl_2$); SFC conditions (Lux 5u Cellulose-1, column temperature: 37.0 ℃, MeOH/CO_2 = 10/90, flow rate = 3.0 mL/min, l = 210 nm) t_R = 5.1 min (major), 5.7 min (minor); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.22-7.13 (m, 2H), 7.02-6.93 (m, 2H), 3.34-3.19 (m, 1H), 2.67-2.51 (m, 2H), 1.30 (d, *J* = 7.0 Hz, 3H). The analytical data are consistent with the literature.[10]

(-)-3-(4-chlorophenyl)butanoic acid (**2i**)



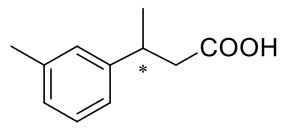
Purification by flash column chromatography (silica gel, PE : EA = 1 : 1) afforded the product as light yellow soild; 24.2 mg, yield: 98%; 95% ee; $[\alpha]_D^{20}$ = -49.5 (c = 1.2, CH_2Cl_2); SFC conditions (Lux 5u Amylose-3, column temperature: 37.0 ℃, MeOH/CO_2 = 10/90, flow rate = 3.0 mL/min, l = 210 nm) t_R = 4.0 min (minor), 4.2 min (major); ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.28-7.25 (m, 2H), 7.17-7.13 (m, 2H), 3.28-3.21 (m, 1H), 2.66-2.54 (m, 2H), 1.30 (d, *J* = 7.0 Hz, 3H). MP: 63-65 °C. The analytical data are consistent with the literature.[1]

(-)-3-(*o*-tolyl)butanoic acid (**2j**)



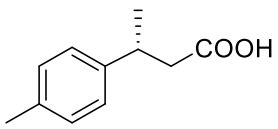
Purification by flash column chromatography (silica gel, PE : EA = 1 : 1) afforded the product as light yellow soild; 22.8 mg, yield: 98%; 97% ee; $[\alpha]_D^{20}$ = -17.3 (c = 1.1, CH_2Cl_2); SFC conditions (Lux 5u Cellulose-1, column temperature: 37.0 ℃, MeOH/CO_2 = 5/95, flow rate = 3.0 mL/min, l = 210 nm) t_R = 4.6 min (major), 7.5 min (minor); ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.18 (d, *J* = 3.4 Hz, 2H), 7.15 (d, *J* = 7.2 Hz, 1H), 7.13-7.09 (m, 1H), 3.50-3.56 (m, *J* = 6.9 Hz, 1H), 2.72-2.53 (m, 2H), 2.37 (s, 3H), 1.28 (d, *J* = 6.9 Hz, 3H). MP: 40-42 °C. The analytical data are consistent with the literature.[1]

(-)-3-(*m*-tolyl)butanoic acid (**2k**)



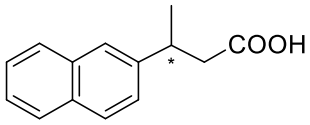
Purification by flash column chromatography (silica gel, PE : EA = 1 : 1) afforded the product as light yellow oil; 22.8 mg, yield: 98%; 98% ee; $[\alpha]_D^{20}$ = -44.3 (c = 1.1, $CHCl_3$); SFC conditions (Lux 5u Cellulose-1, column temperature: 37.0 ℃, MeOH/CO_2 = 5/95, flow rate = 3.0 mL/min, l = 210 nm) t_R = 6.2 min (minor), 6.9 min (major); ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.19 (t, *J* = 7.4 Hz, 1H), 7.03 (d, *J* = 8.0 Hz, 3H), 3.30-3.18 (m, 1H), 2.74-2.49 (m, 2H), 2.34 (s, 3H), 1.31 (d, *J* = 6.8 Hz, 3H). The analytical data are consistent with the literature.[10]

(*R*)-3-(*p*-tolyl)butanoic acid (**2l**)



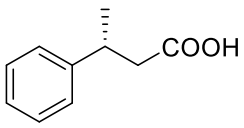
Purification by flash column chromatography (silica gel, PE : EA = 1 : 1) afforded the product as light yellow soild; 21.4 mg, yield: 96%; 98% ee; $[\alpha]_D^{20}$ = -45.5 (c = 1.2, $CHCl_3$); SFC conditions (Lux 5u Cellulose-4, column temperature: 37.0 ℃, MeOH/CO_2 = 5/95, flow rate = 3.0 mL/min, l = 210 nm) t_R = 4.7 min (minor), = 5.0 min (major); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.18-7.00 (m, 4H), 3.34-3.14 (m, 1H), 2.72-2.49 (m, 2H), 2.32 (s, 3H), 1.30 (d, *J* = 7.0 Hz, 3H). MP: 97-99 °C. The absolute configuration of (*R*)-**2l** was determined by comparison with optical rotation data for the reported literature.[11]

(-)-3-(naphthalen-2-yl)butanoic acid (**2m**)



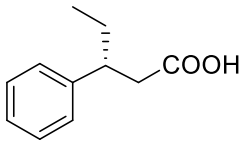
Purification by flash column chromatography (silica gel, PE : EA = 1 : 1) afforded the product as light yellow soild; 25.6 mg, yield: 96%; 93% ee; $[\alpha]_D^{20}$ = -22.8 (c = 1.3, ethanol); SFC conditions (Lux 5u Amylose-1, column temperature: 37.0 ℃, MeOH/CO_2 = 15/85, flow rate = 3.0 mL/min, l = 210 nm) t_R = 3.7 min (minor), 4.4 min (major); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.80 (d, *J* = 8.2 Hz, 3H), 7.66 (s, 1H), 7.49-7.41 (m, 2H), 7.37 (d, *J* = 8.5 Hz, 1H), 3.55-3.36 (m, 1H), 2.81-2.65 (m, 2H), 1.40 (d, *J* = 6.9 Hz, 3H). MP: 109-111 °C. The analytical data are consistent with the literature.[10]

(*R*)-3-phenylbutanoic acid (**2n**)



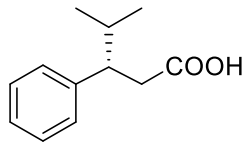
Purification by flash column chromatography (silica gel, PE : EA = 1 : 1) afforded the product as light yellow oil; 20.0 mg, yield: 98%; 98% ee; $[\alpha]_D^{20}$ = -37.3 (c = 1.0, $CHCl_3$); SFC conditions (Lux 5u Amylose-1, column temperature: 37.0 ℃, MeOH/CO_2 = 5/95, flow rate = 3.0 mL/min, l = 210 nm) t_R = 3.6 min (minor), 4.0 min (major); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.36-7.26 (m, 2H), 7.25-7.15 (m, 3H), 3.27 (h, *J* = 7.0 Hz, 1H), 2.85-2.43 (m, 2H), 1.32 (d, *J* = 6.9 Hz, 3H). The absolute configuration of (*R*)-**2n** was determined by comparison with optical rotation data for the reported literature.[12]

(*R*)-3-phenylpentanoic acid (**2o**)



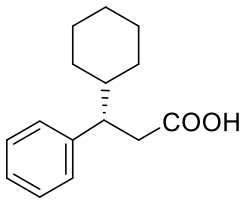
Purification by flash column chromatography (silica gel, PE : EA = 1 : 1) afforded the product as light yellow oil; 21.4 mg, yield: 97%; 98% ee; $[\alpha]_D^{20}$ = -29.5 (c = 1.1, CH_2Cl_2); SFC conditions (Lux 5u Amylose-1, column temperature: 37.0 ℃, MeOH/CO_2 = 5/95, flow rate = 3.0 mL/min, l = 210 nm) t_R = 3.2 min (minor), 3.7 min (major); ^{1}H NMR (600 MHz, Chloroform-*d*) δ 10.69 (s, 1H), 7.28 (t, *J* = 7.4 Hz, 2H), 7.19 (dd, *J* = 17.3, 7.4 Hz, 3H), 3.03-2.92 (m, 1H), 2.72-2.50 (m, 2H), 1.82-1.68 (m, 1H), 1.66-1.53 (m, 1H), 0.79 (t, *J* = 7.2 Hz, 3H). The absolute configuration of (*R*)-**2o** was determined by comparison with optical rotation data for the reported literature.[1]

(*S*)-4-methyl-3-phenylpentanoic acid (**2p**)



Purification by flash column chromatography (silica gel, PE : EA = 1 : 1) afforded the product as light yellow oil; 23.5 mg, yield: 98%; 99% ee; $[\alpha]_D^{20}$ = -32.3 (c = 1.2, CH_2Cl_2); SFC conditions (Lux 5u Cellulose-1, column temperature: 37.0 ℃, MeOH/CO_2 = 10/90, flow rate = 3.0 mL/min, l = 210 nm) t_R = 2.1 min (major), 2.3 min (minor); ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.27-7.21 (m, 2H), 7.18 (t, *J* = 7.2 Hz, 1H), 7.12 (d, *J* = 7.3 Hz, 2H), 2.89-2.83 (m, 1H), 2.81-2.74 (m, 1H), 2.67-2.55 (m, 1H), 1.91-1.79 (m, 1H), 0.92 (d, *J* = 6.5 Hz, 3H), 0.74 (d, *J* = 6.6 Hz, 3H). The absolute configuration of (*S*)-**2p** was determined by comparison with optical rotation data for the reported literature.[1]

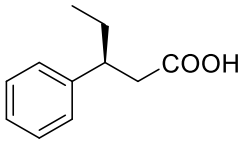
(*S*)-3-cyclohexyl-3-phenylpropanoic acid (**2q**)



Purification by flash column chromatography (silica gel, PE : EA = 1 : 1) afforded the product as light yellow soild; 28.0 mg, yield: 97%; 99% ee; $[\alpha]_D^{20}$ = -56.6 (c = 1.4, CH_2Cl_2); SFC conditions (Lux 5u Cellulose-4, column temperature: 37.0 ℃, MeOH/CO_2 = 10/90, flow rate = 3.0 mL/min, l = 210 nm) t_R = 3.9 min (minor), 5.0 min (major); ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.28-7.24 (m, 2H), 7.21-7.17 (m, 1H), 7.14-7.09 (m, 2H), 2.90-2.85 (m, 1H), 2.84-2.78 (m, 1H), 2.61-2.54 (m, 1H), 1.82-1.71 (m, 2H), 1.65-1.57 (m, 2H), 1.49-1.41 (m, 2H), 1.26-1.18 (m, 1H), 1.14-1.03 (m, 2H), 0.97-0.90 (m, 1H), 0.83-0.75 (m, 1H). MP: 90-92 °C. The absolute configuration of (*S*)-**2q** was

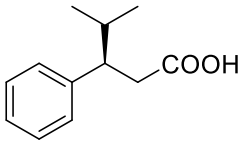
determined by comparison with optical rotation data for the reported literature.[1]

(*S*)-3-phenylpentanoic acid (**2r**)



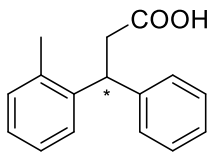
Purification by flash column chromatography (silica gel, PE : EA = 1 : 1) afforded the product as light yellow oil; 21.6 mg, yield: 97%; 91% ee; $[\alpha]_D^{20}$ = +20.5 (c = 1.1, CH_2Cl_2); SFC conditions (Lux 5u Amylose-1, column temperature: 37.0 ℃, MeOH/CO_2 = 5/95, flow rate = 3.0 mL/min, l = 210 nm) t_R = 3.1 min (major), 3.5 min (minor); ^{1}H NMR (600 MHz, Chloroform-*d*) δ 10.69 (s, 1H), 7.28 (t, *J* = 7.4 Hz, 2H), 7.19 (dd, *J* = 17.3, 7.4 Hz, 3H), 3.03-2.92 (m, 1H), 2.72-2.50 (m, 2H), 1.82-1.68 (m, 1H), 1.66-1.53 (m, 1H), 0.79 (t, *J* = 7.2 Hz, 3H). The absolute configuration of (*S*)-**2r** was determined by comparison with optical rotation data for the reported literature.[1]

(*R*)-3-phenylpentanoic acid (**2s**)



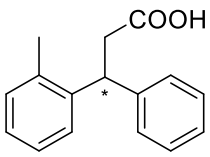
Purification by flash column chromatography (silica gel, PE : EA = 1 : 1) afforded the product as light yellow oil; 23.0 mg, yield: 96%; 90% ee; $[\alpha]_D^{20}$ = +21.3 (c = 1.0, CH_2Cl_2); SFC conditions (Lux 5u Cellulose-1, column temperature: 37.0 ℃, MeOH/CO_2 = 10/90, flow rate = 3.0 mL/min, l = 210 nm) t_R =2 .9 min (minor), 3.2 min (major); ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.27-7.21 (m, 2H), 7.18 (t, *J* = 7.2 Hz, 1H), 7.12 (d, *J* = 7.3 Hz, 2H), 2.89-2.83 (m, 1H), 2.81-2.74 (m, 1H), 2.67-2.55 (m, 1H), 1.91-1.79 (m, 1H), 0.92 (d, *J* = 6.5 Hz, 3H), 0.74 (d, *J* = 6.6 Hz, 3H). The absolute configuration of (*R*)-**2s** was determined by comparison with optical rotation data for the reported literature.[1]

(-)-3-phenyl-3-(*o*-tolyl)propanoic acid (**2t**)



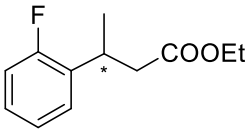
Purification by flash column chromatography (silica gel, PE : EA = 1 : 1) afforded the product as light yellow soild; 29.1 mg, yield: 97%; 83% ee; $[\alpha]_D^{20}$ = -15.7 (c = 1.5, CH_2Cl_2); SFC conditions (Lux 5u Cellulose-4, column temperature: 37.0 ℃, MeOH/CO_2 = 10/90, flow rate = 3.0 mL/min, l = 210 nm) t_R = 3.1 min (minor), 4.0 min (major); ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.26 (d, *J* = 5.2 Hz, 6H), 7.22-7.15 (m, 4H), 7.16-7.11 (m, 2H), 4.72 (t, *J* = 7.9 Hz, 1H), 3.12-3.01 (m, 2H), 2.28 (s, 3H). MP: 121-123 °C. The analytical data are consistent with the literature.[13]

(+)-3-phenyl-3-(*o*-tolyl)propanoic acid (**2u**)



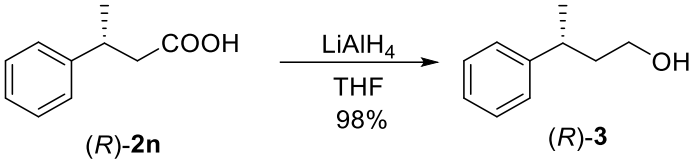
Purification by flash column chromatography (silica gel, PE : EA = 1 : 1) afforded the product as light yellow soild; 29.3 mg, yield: 98%; 79% ee; $[\alpha]_D^{20}$ = +14.3 (c = 1.5, CH_2Cl_2); SFC conditions (Lux 5u Cellulose-4, column temperature: 37.0 ℃, MeOH/CO_2 = 10/90, flow rate = 3.0 mL/min, l = 210 nm) t_R = 3.4 min (major), 4.5 min (minor); ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.26 (d, *J* = 5.2 Hz, 6H), 7.22-7.15 (m, 4H), 7.16-7.11 (m, 2H), 4.72 (t, *J* = 7.9 Hz, 1H), 3.12-3.01 (m, 2H), 2.28 (s, 3H). MP: 121-123 °C. The analytical data are consistent with the literature.[13]

(-)-ethyl 3-(2-fluorophenyl)butanoate (**2v**)



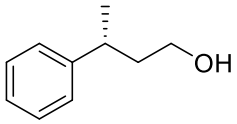
Purification by flash column chromatography (silica gel, PE : EA = 20 : 1) afforded the product as light yellow oil; 25.2 mg, yield: 96%; 98% ee; $[\alpha]_D^{20}$ = -22.7 (c = 1.3, CH_2Cl_2); HPLC conditions (Lux 5u Cellulose-3, ipa/hex = 10/90, flow rate = 1.0 mL/min, l = 210 nm) t_R = 4.3 min (major), 4.6 min (minor); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.25-7.13 (m, 2H), 7.11-7.04 (m, 1H), 7.03-6.97 (m, 1H), 4.08 (q, *J* = 7.1 Hz, 2H), 3.63-3.48 (m, 1H), 2.74-2.52 (m, 2H), 1.32 (d, *J* = 7.0 Hz, 3H), 1.18 (t, *J* = 7.1 Hz, 3H). The analytical data are consistent with the literature.[14]

6. Procedure for the synthesis of compounds 3, 4 and 5.

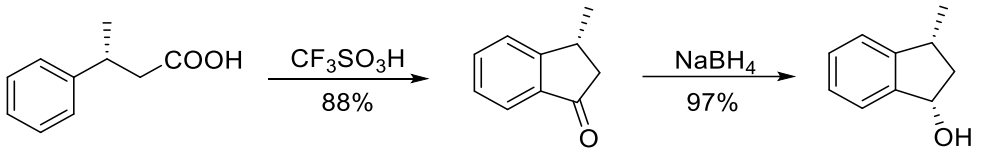


Under N_2 atmosphere, $LiAlH_4$ (57 mg, 1.5 mmol, 3.0 eq) was added into a solution of (*R*)-**2n** (82 mg, 0.5 mmol, 1.0 eq) in THF (10 mL) at 0 °C. After stirring at room temperature until no starting material was detected by TLC, the reaction was quenched by adding water slowly. The mixture was acidified with 2 M HCl_{aq}, and was extracted with EtOAc (3 × 50 mL) and then dried over anhydrous Na_2SO_4. The solvent was evaporated under vacuum, and the residue was purified by column chromatography on silica gel using EtOAc/petroleum ether (1:10 to 1:5) as the eluent giving the (*R*)-3-phenylbutan-1-ol (**3**) as a colorless oil (73 mg, yield: 98%).[15]

(*R*)-3-phenylbutan-1-ol (**3**)



Purification by flash column chromatography (silica gel, PE : EA = 10:1 to 5:1) afforded the product as colorless oil; 73 mg, yield: 98%; 98% ee; $[\alpha]_D^{20}$ = -32.3 (c = 1.5, $CHCl_3$); SFC conditions (Lux 5u Amylose-1, column temperature: 37.0 ℃, MeOH/CO_2 = 10/90, flow rate = 3.0 mL/min, l = 210 nm) t_R = 6.5 min (major), 7.5 min (minor); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.34-7.28 (m, 2H), 7.23-7.15 (m, 3H), 3.66-3.46 (m, 2H), 2.97-2.81 (m, 1H), 1.86 (q, *J* = 6.9 Hz, 2H), 1.28 (d, *J* = 7.0 Hz, 3H). The absolute configuration of (*R*)-**3** was determined by comparison with optical rotation data for the reported literature.[16]

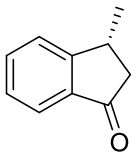


(*R*)-**2n** (*R*)-**4** (1*S*,3*R*)-**5**

(*R*)-**2n** (82 mg, 0.5 mmol, 1.0 eq) was dissolved in triflic acid (1 mL) and the solution was stirred for 12 h at 70 °C in an oil bath. After the reaction was complete, the solusion was poured over water (5 mL) and extracted with EtOAc (3 × 15 mL). The organic extracts were combined, washed with brine, and dried over anhydrous Na_2SO_4. The solvent was removed by vacuum evaporation, and crude product was purified by column chromatography on silica gel using EtOAc/petroleum ether (1:20) as eluent giving the (*R*)-**4** as a colorless oil (64 mg, yield: 88%).[15]

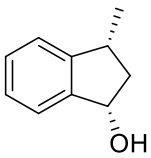
$NaBH_4$ (37 mg, 1 mmol, 3.3 eq) was added to a solution of (*R*)-**4** (44 mg, 0.3 mmol, 1.0 eq) in methanol (5 mL) at 0 °C. The reaction mixture was stirred for another 30 min until no starting material was detected by TLC, after which the volatiles were removed in vacuo and the residue slowly acidified with 2 M HCl_{aq}. The product was extracted with EtOAc (3 × 15 mL), dried over Na_2SO_4 and concentrated in vacuo. The crude product was purified by column chromatography on silica gel using EtOAc/petroleum ether (1:5) as eluent giving the **5** as a colorless soild (44 mg, yield: 97%).[17]

(*R*)-3-methyl-2,3-dihydro-1*H*-inden-1-one (**4**)



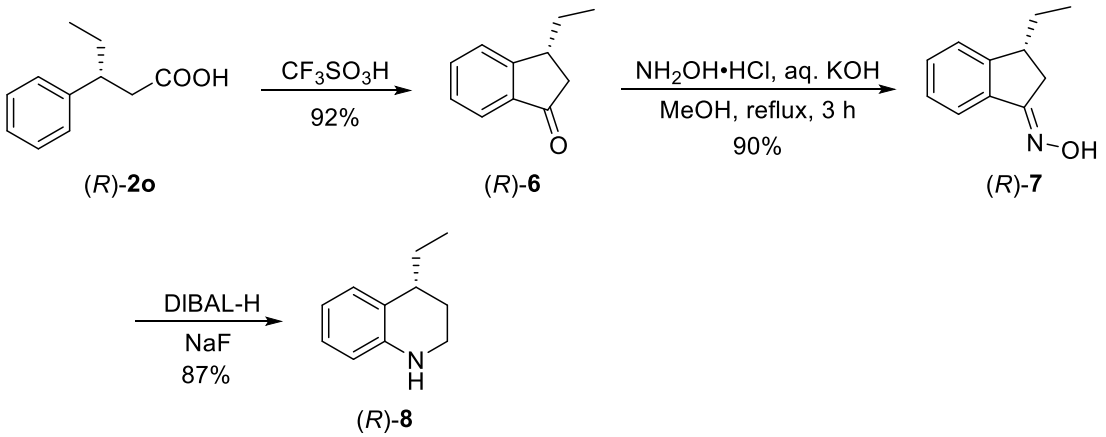
Purification by flash column chromatography (silica gel, PE : EA = 20 : 1) afforded the product as colorless oil; 64 mg, yield: 88%; 97% ee; $[\alpha]_D^{20}$ = -13.7 (c = 1.5, $CHCl_3$); HPLC conditions (Lux 5u Cellulose-1, ipa/hex = 5/95, flow rate = 1.0 mL/min, l = 210 nm) t_R = 9.8 min (minor), 10.3 min (major); ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.73 (d, *J* = 7.6 Hz, 1H), 7.65-7.57 (m, 1H), 7.53-7.49 (m, 1H), 7.40-7.34 (m, 1H), 3.49-3.42 (m, 1H), 2.96 (dd, *J* = 19.0, 7.5 Hz, 1H), 2.30 (dd, *J* = 19.0, 3.5 Hz, 1H), 1.41 (d, *J* = 7.2 Hz, 3H). The absolute configuration of (*R*)-**4** was determined by comparison with optical rotation data for the reported literature.[18]

(1*S*,3*R*)-3-methyl-2,3-dihydro-1*H*-inden-1-ol (**5**)



Purification by flash column chromatography (silica gel, PE : EA = 5 : 1) afforded the product as colorless solid; 43.0 mg, yield: 97%; 94:6 dr, 94% ee; $[\alpha]_D^{20}$ = +12.5 (c = 1.2, CH_2Cl_2); GC conditions: Supelco Alpha DexTM 120 column (30 m × 0.25 mm × 0.25 μm), N_2 1.0 mL/min, programmed 75 °C - 0.1 °C/min - 200 °C (hold 50 min); t_R = 28.1 min (major), 29.1 min (minor); ^{1}H NMR (600 MHz, Chloroform-*d*; *cis* isomer) δ 7.41 (d, *J* = 7.3 Hz, 1H), 7.32-7.26 (m, 2H), 7.23 (d, *J* = 7.1 Hz, 1H), 5.19 (t, *J* = 7.3 Hz, 1H), 3.03-3.08 (m, 1H), 2.79-2.75 (m, 1H), 1.78 (s, 1H), 1.49-1.45 (m, 1H), 1.37 (d, *J* = 6.9 Hz, 3H). M.P. = 74-76 °C. The absolute configuration of (1*S*,3*R*)-**5** was determined by comparison with optical rotation data for the reported literature.[17]

7. General procedure for the synthesis of 8 .



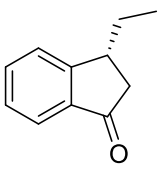
(*R*)-**2o** (*R*)-**6** (*R*)-**7** (*R*)-**8**

(*R*)-**2o** (267 mg, 1.5 mmol, 1.0 eq) was dissolved in triflic acid (3 mL) and the solution was stirred for 12 h at 70 °C in an oil bath. After the reaction was complete, the solusion was poured over water (15 mL) and extracted with EtOAc (3 × 20 mL). The organic extracts were combined, washed with brine, and dried over anhydrous Na_2SO_4. The solvent was removed by vacuum evaporation, and crude product was purified by column chromatography on silica gel using EtOAc/petroleum ether (1:20) as eluent giving the (*R*)-**6** as a colorless oil (220 mg, yield: 92%).[19]

To a solution of (*R*)-**6** (160 mg, 1.0 mmol, 1.0 eq) in methanol (5.0 mL) was added hydroxylamine hydrochloride (103 mg, 1.5 mmol, 1.5 eq) and aq. KOH (50%, 0.34 mL, 3.0 mmol, 3.0 eq). The mixture was refluxed in an oil bath for 3 h when TLC indicated complete conversion. After cooling to room temperature, the residue slowly neutralized with 2 M HCl_{aq}. The product was extracted with EtOAc (3 × 15 mL), dried over Na_2SO_4 and concentrated in vacuo. The crude product was purified by column chromatography on silica gel using EtOAc/petroleum ether (1:5) as eluent giving the (*R*)-**7** as a pale yellow solid (157 mg, yield: 90%).[19]

To a stirred solution of (*R*)-**7** (87 mg, 0.5 mmol, 1.0 eq) in anhydrous CH_2Cl_2 (56.0 mL) was added DIBAL-H (3.0 mL, 3.0 mmol, 6.0 eq, 1.0 M in *n*-hexane) at 0 °C under an argon atmosphere. The solution was stirred for 2 h at room temperature. NaF powder (0.6 g, 15 mmol, 30.0 eq) and water (0.5 mL) were added at 0 °C, and the resulting mixture was stirred for 30 min. The desired solution was then filtered through a pad of celite, followed by addition of Na_2SO_4 to remove water. The organic solutions were evaporated to give the crude product. Purification by column chromatography on a silica gel (EtOAc: petroleum ether = 1:3) yielded (*R*)-**8** (70 mg, yield: 87%).[20]

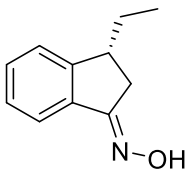
(*R*)-3-ethyl-2,3-dihydro-1*H*-inden-1-one (**6**)



Purification by flash column chromatography (silica gel, PE : EA = 20 : 1) afforded the product as colorless oil; 220.0 mg, yield: 92%; 98% ee; $[\alpha]_D^{20}$ = -20.2 (c = 1.4, ethanol); HPLC conditions (Lux 5u Cellulose-1, ipa/hex = 5/95, flow rate = 1.0 mL/min, l = 210 nm) t_R = 6.1 min (minor), 6.3 min (major); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.74 (d, *J* = 7.7 Hz, 1H), 7.63-7.57 (m, 1H), 7.51 (d, *J* = 7.7 Hz, 1H), 7.38 (t, *J* = 7.4 Hz, 1H), 3.37-3.29 (m, 1H), 2.85 (dd, *J* =

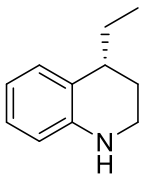
19.1, 7.5 Hz, 1H), 2.37 (dd, *J* = 19.1, 3.3 Hz, 1H), 2.03-1.92 (m, 1H), 1.60-1.51 (m, 1H), 0.98 (t, *J* = 7.4 Hz, 3H). The absolute configuration of (*R*)-**6** was determined by comparison with optical rotation data for the reported literature.[19]

(*R*)-3-ethyl-2,3-dihydro-1*H*-inden-1-one oxime (**7**)



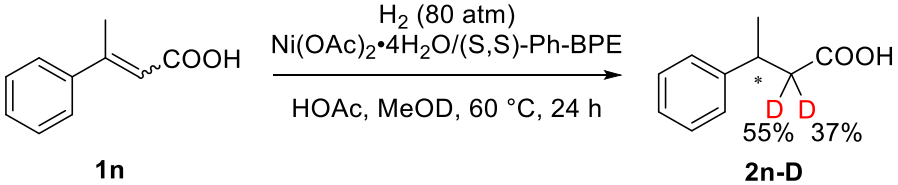
Purification by flash column chromatography (silica gel, PE : EA = 5 : 1) afforded the product as pale yellow solid; 157.0 mg, yield: 90%; 92% ee; $[\alpha]_D^{20}$ = +17.1 (c = 1.3, ethanol); SFC conditions (Lux 5u Cellulose-1, column temperature: 37.0 °C, MeOH/CO_2 = 10/90, flow rate = 3.0 mL/min, l = 210 nm) t_R = 3.5 min (minor), 4.0 min (major); ^{1}H NMR (400 MHz, Chloroform-*d*) ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.67 (d, *J* = 7.7 Hz, 1H), 7.40-7.36 (m, 1H), 7.33 (d, *J* = 7.6 Hz, 1H), 7.29-7.26 (m, 1H), 3.28-3.22 (m, 1H), 3.19-3.13 (m, 1H), 2.67-2.61 (m, 1H), 1.94-1.87 (m, 1H), 1.54-1.47 (m, 1H), 0.99 (t, *J* = 7.4 Hz, 3H). M.P. = 81-83 °C. The absolute configuration of (*R*)-**7** was determined by comparison with optical rotation data for the reported literature.[19]

(*R*)-4-ethyl-1,2,3,4-tetrahydroquinoline (**8**)



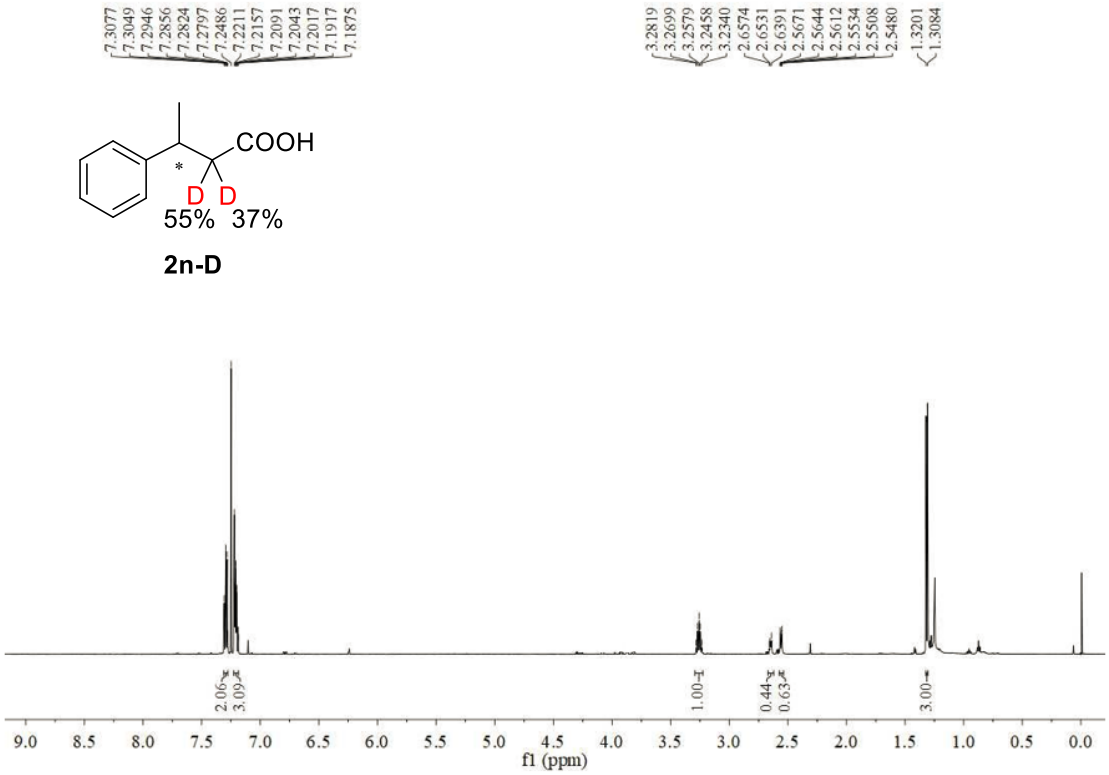
Purification by flash column chromatography (silica gel, PE : EA = 3 : 1) afforded the product as colorless oil; 70.0 mg, yield: 87%; 90% ee; $[\alpha]_D^{20}$ = +28.3 (c = 1.5, ethanol); SFC conditions (Lux 5u Cellulose-3, column temperature: 37.0 °C, MeOH/CO_2 = 10/90, flow rate = 3.0 mL/min, l = 210 nm) t_R = 4.5 min (major), 5.1 min (minor); ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.03 (d, *J* = 7.5 Hz, 1H), 6.97 (t, *J* = 7.6 Hz, 1H), 6.63 (t, *J* = 7.4 Hz, 1H), 6.48 (d, *J* = 7.9 Hz, 1H), 3.86 (s, 1H), 3.35-3.30 (m, 1H), 3.28-3.24 (m, 1H), 2.68-2.63 (m, 1H), 1.96-1.90 (m, 1H), 1.85-1.81 (m, 1H), 1.78-1.73 (m, 1H), 1.58-1.52 (m, 1H), 1.00 (t, *J* = 7.4 Hz, 3H). The absolute configuration of (*R*)-**8** was determined by comparison with optical rotation data for the reported literature.[19]

8. Deuterium labelling studies.



1n **2n-D**

A stock solution was made by mixing $Ni(OAc)_2 \cdot H_2O$ with (*S*, *S*)-Ph-BPE in a 5 : 5.1 molar ratio in deuterated methanol (1.0 mL) at room temperature for 12 hours in a nitrogen-filled glovebox. The catalyst solution was transferred by syringe into the vial charged with substrate (0.125 mmol) and added additive HOAc. The vial were subsequently transferred into an autoclave which hydrogen gas was charged. The reaction was then stirred under H_2 (80 atm) at 60 °C for 24 h. The hydrogen gas was released slowly and carefully. The solution was passed through a short column of silica gel to remove the metal complex. The conversion of products were determined by ^{1}H NMR analysis. ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.31-7.28 (m, 2H), 7.23-7.19 (m, 3H), 3.26 (p, *J* = 7.2 Hz, 1H), 2.67-2.62 (m, 0.44H), 2.57-2.54 (m, 0.63H), 1.31 (d, *J* = 7.0 Hz, 3H).



^{1}H NMR (600 MHz, Chloroform-*d*)

9. References

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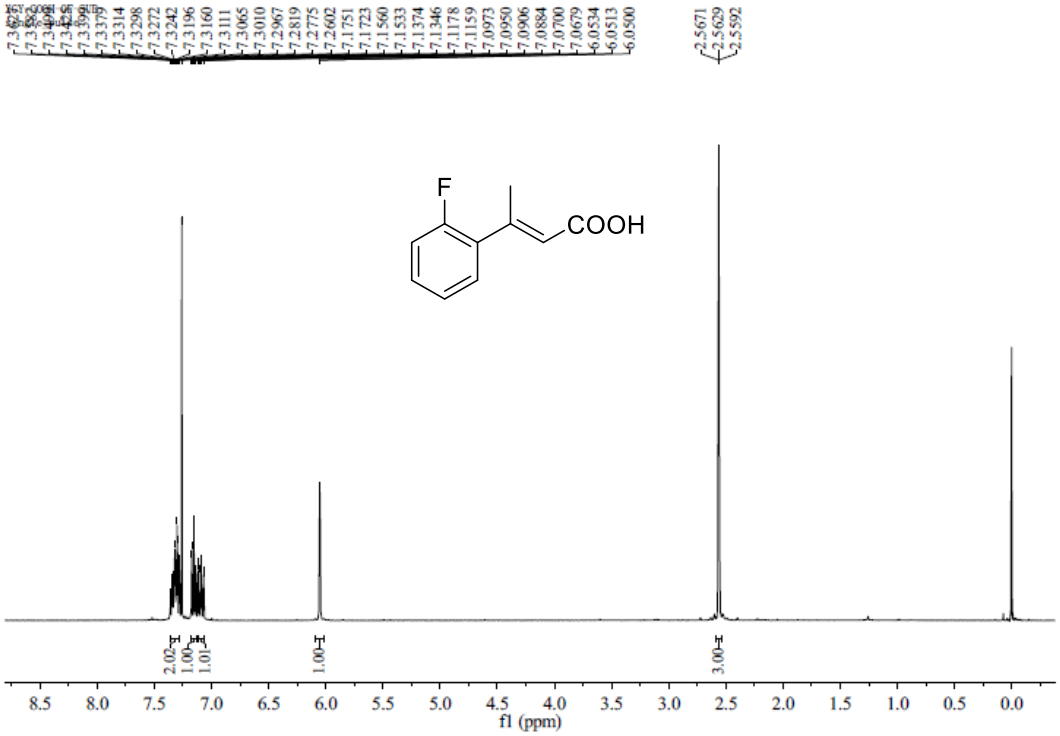
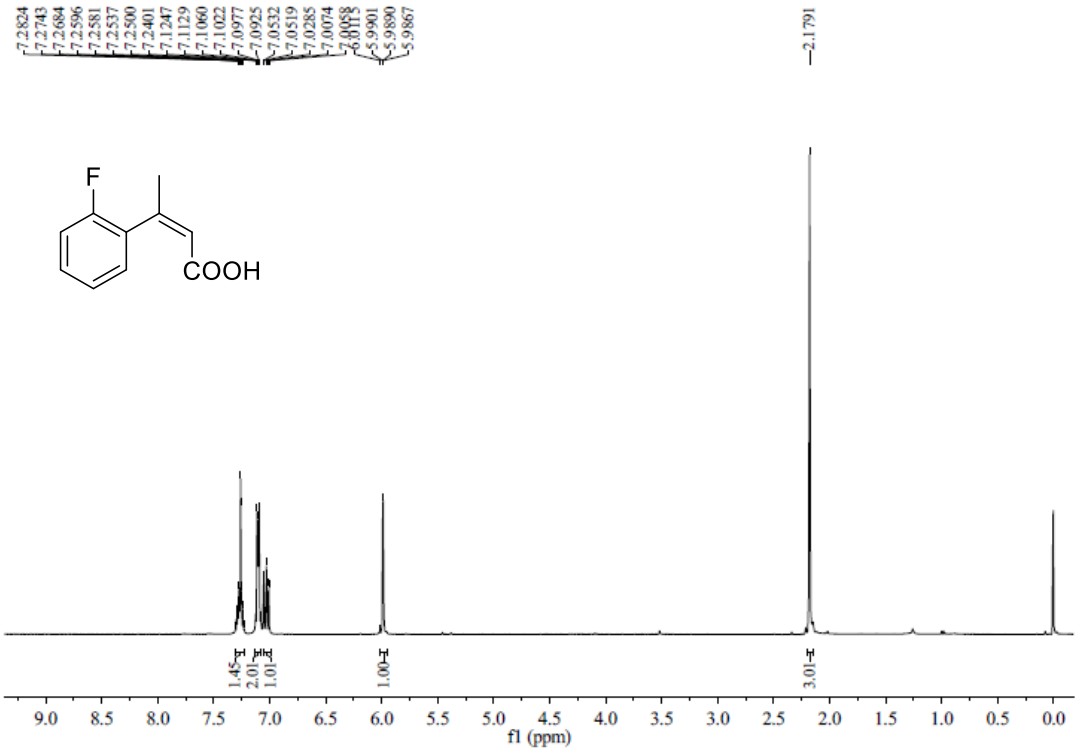
10. NMR, SFC and HPLC spectra

(*E*)-3-(2-fluorophenyl)but-2-enoic acid (**1a**)

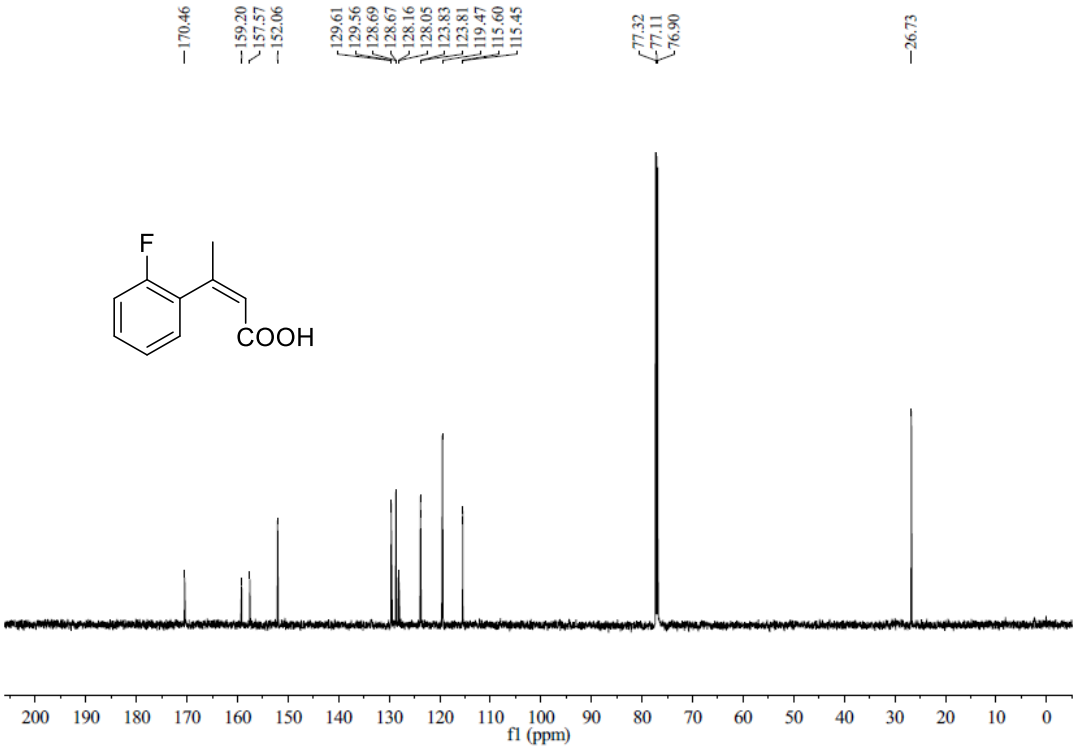


^{1}H NMR (400 MHz, Chloroform-*d*)

(*Z*)-3-(2-fluorophenyl)but-2-enoic acid (**1b**)

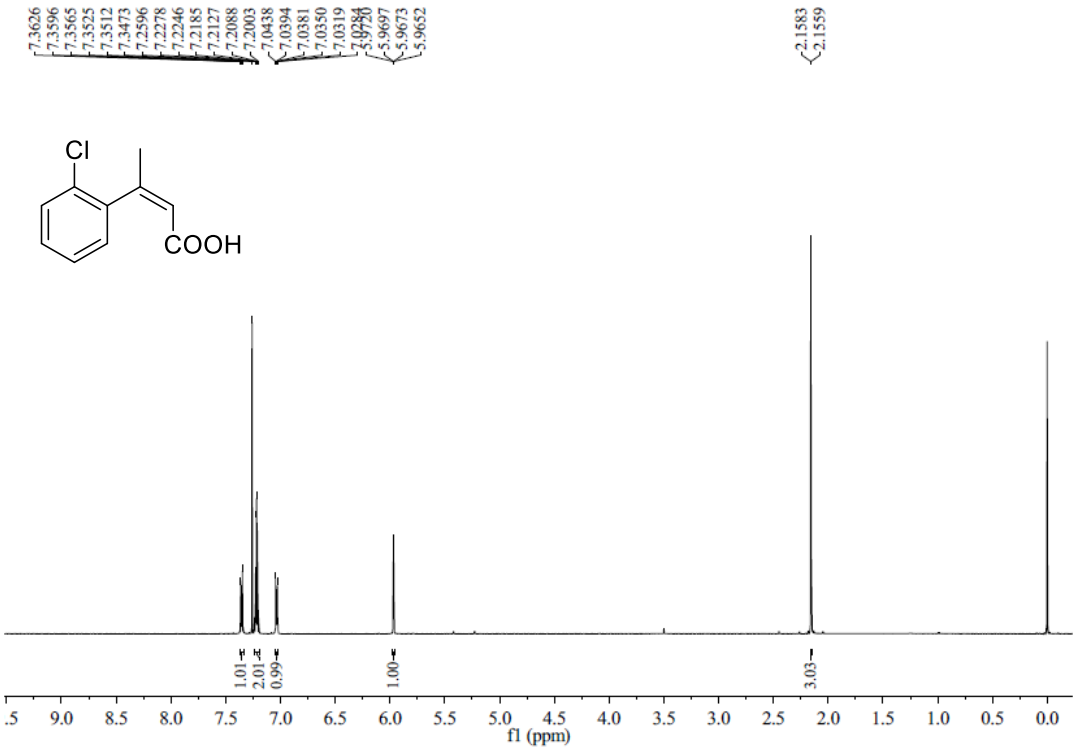


^{1}H NMR (400 MHz, Chloroform-*d*)

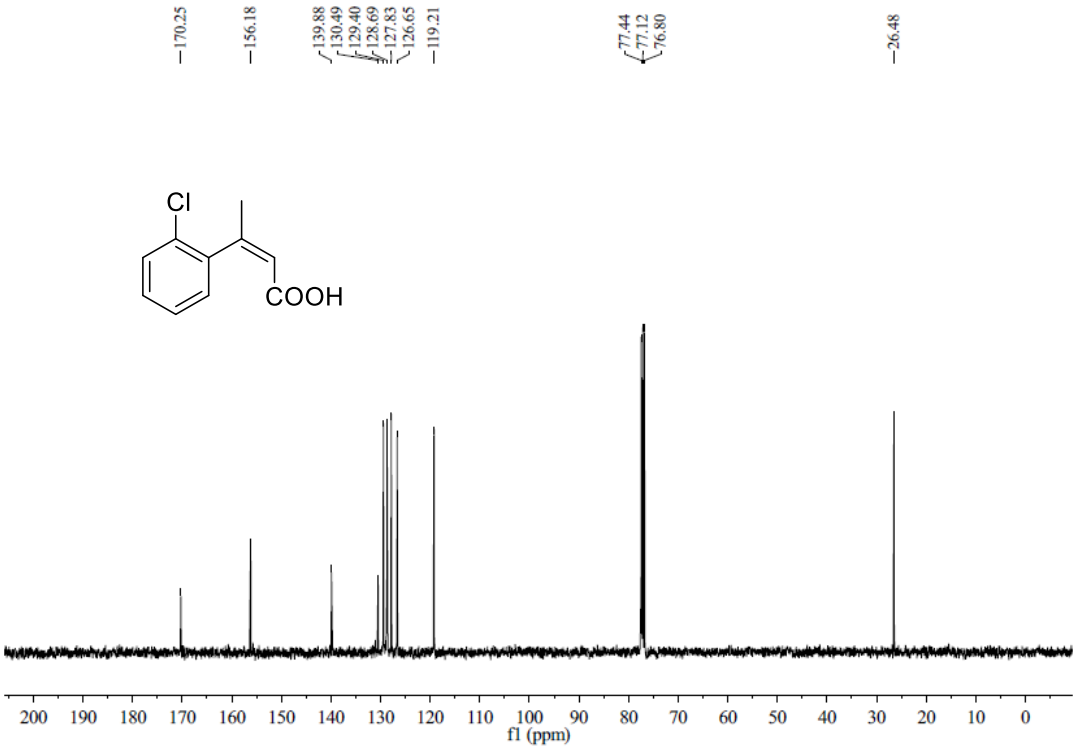


^{13}C NMR (150 MHz, Chloroform-*d*)

(*Z*)-3-(2-chlorophenyl)but-2-enoic acid (**1c**)

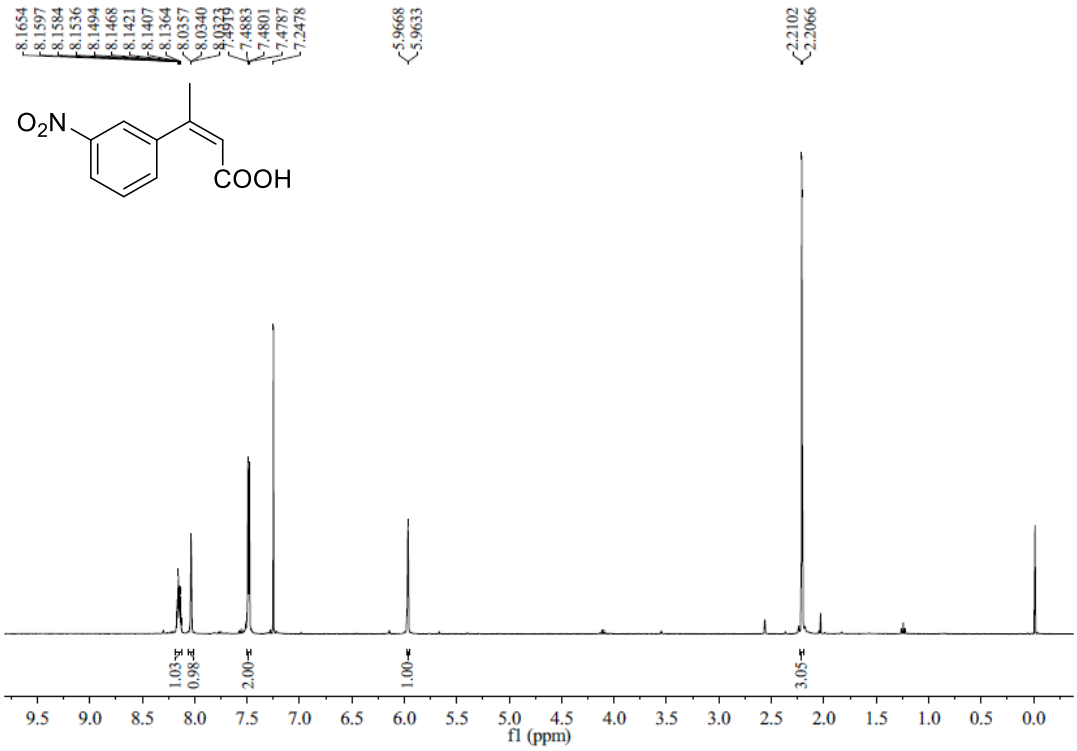


^{1}H NMR (600 MHz, Chloroform-*d*)

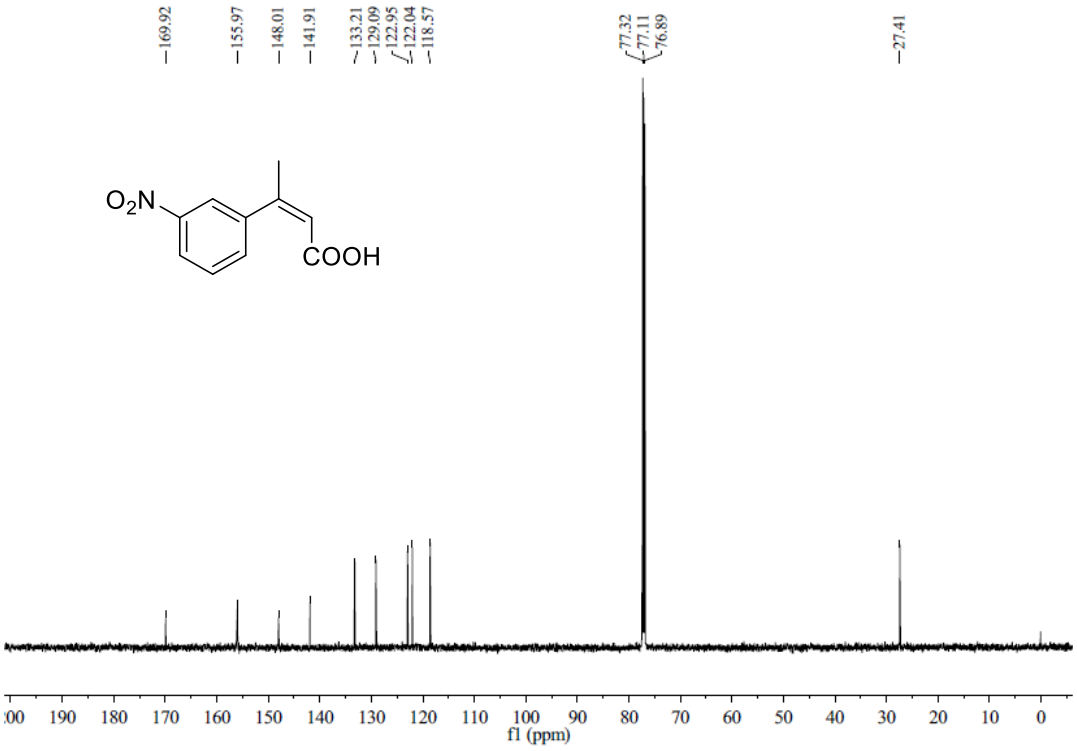


^{13}C NMR (100 MHz, Chloroform-*d*)

(*Z*)-3-(3-nitrophenyl)but-2-enoic acid (**1d**)

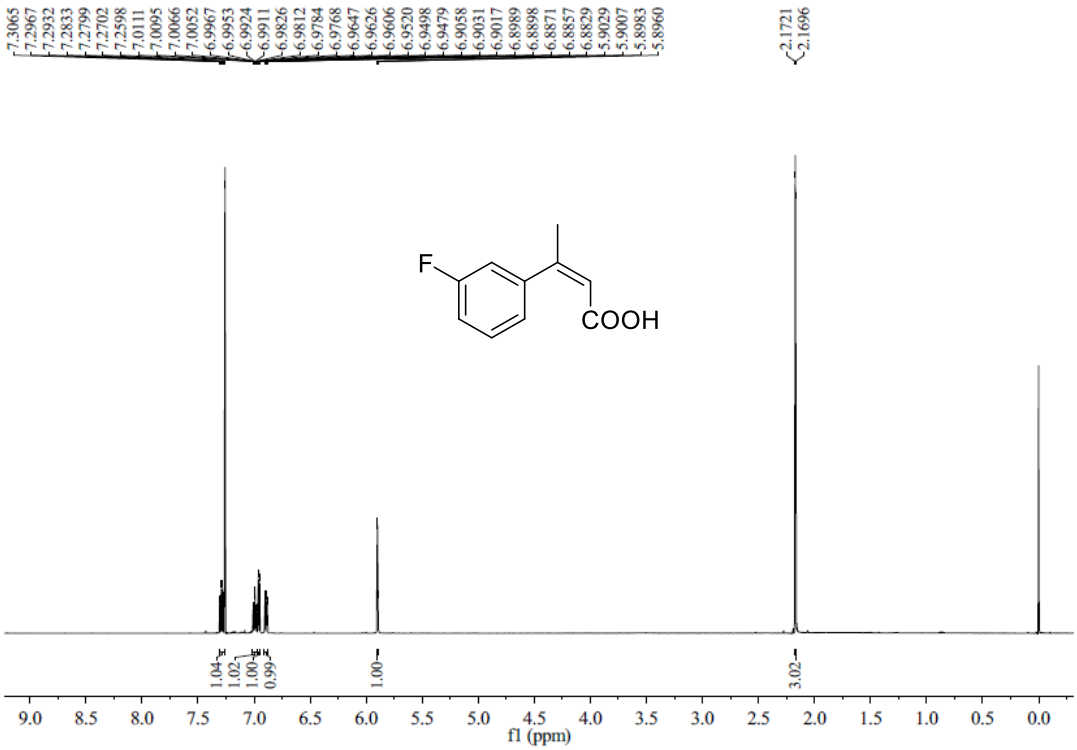


^{1}H NMR (600 MHz, Chloroform-*d*)

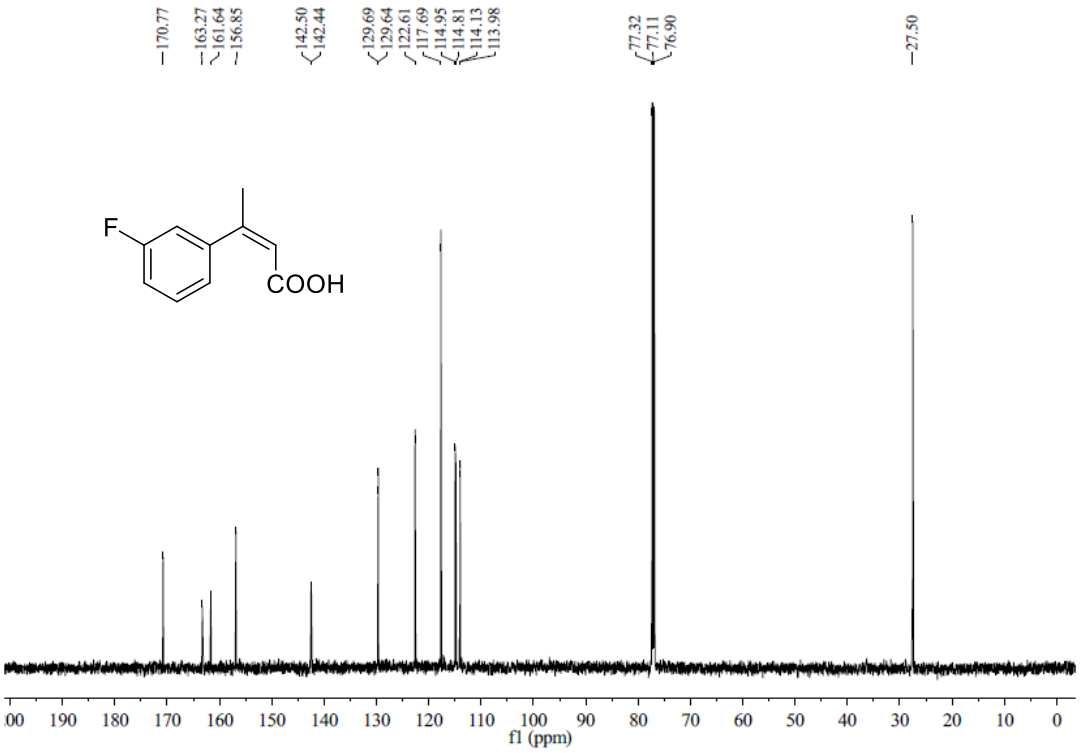


^{13}C NMR (150 MHz, Chloroform-*d*)

(*Z*)-3-(3-fluorophenyl)but-2-enoic acid (**1e**)

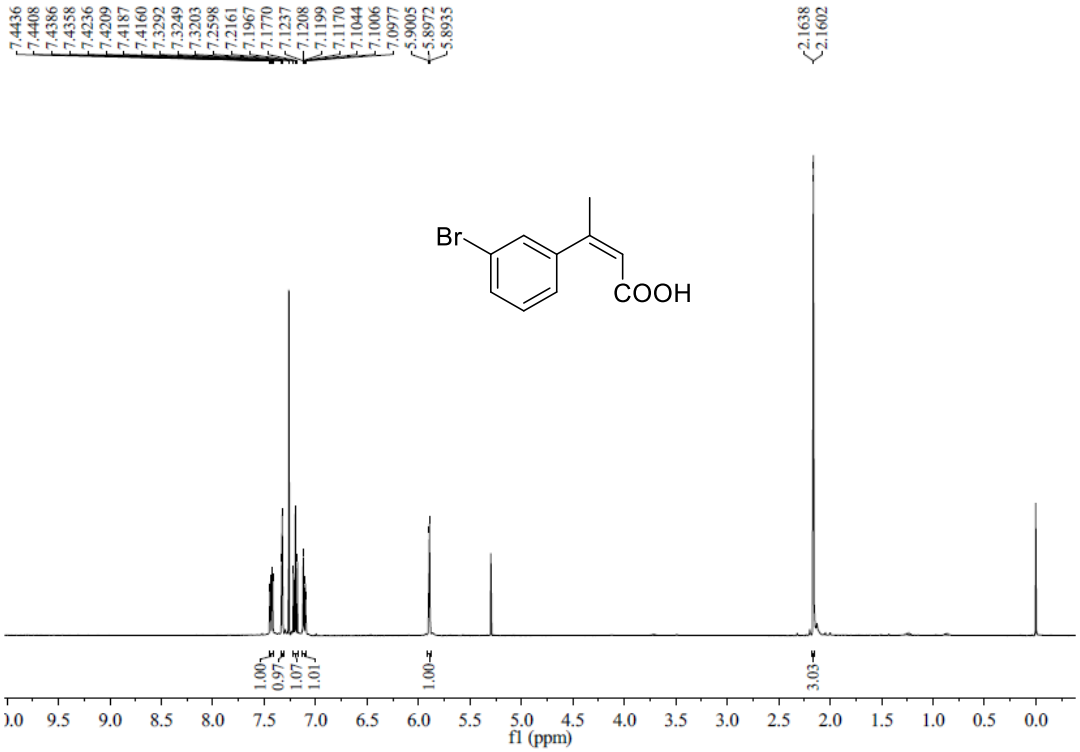


^{1}H NMR (600 MHz, Chloroform-*d*)

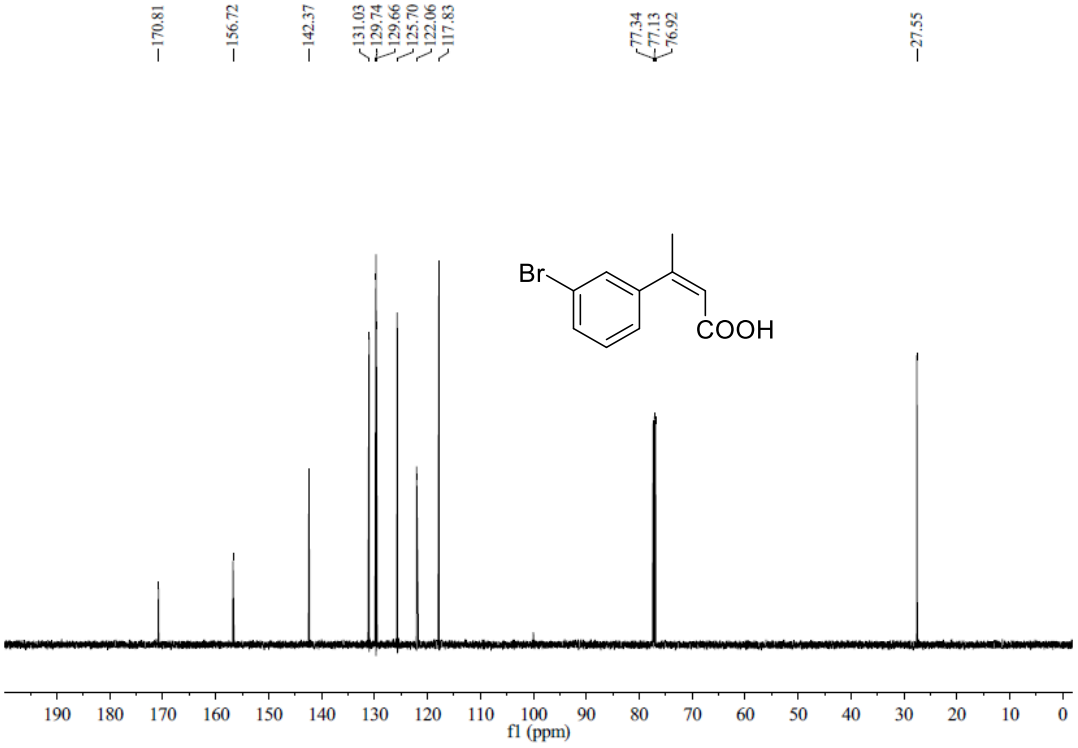


^{13}C NMR (150 MHz, Chloroform-*d*)

(*Z*)-3-(3-bromophenyl)but-2-enoic acid (**1f**)

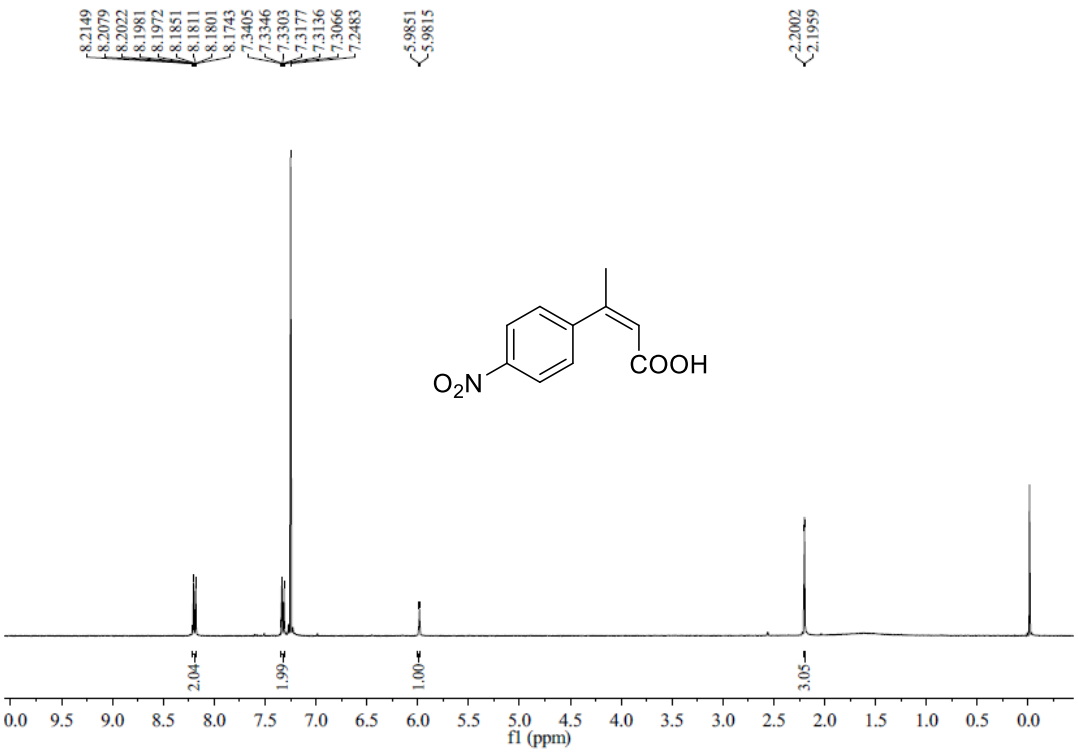


^{1}H NMR (400 MHz, Chloroform-*d*)

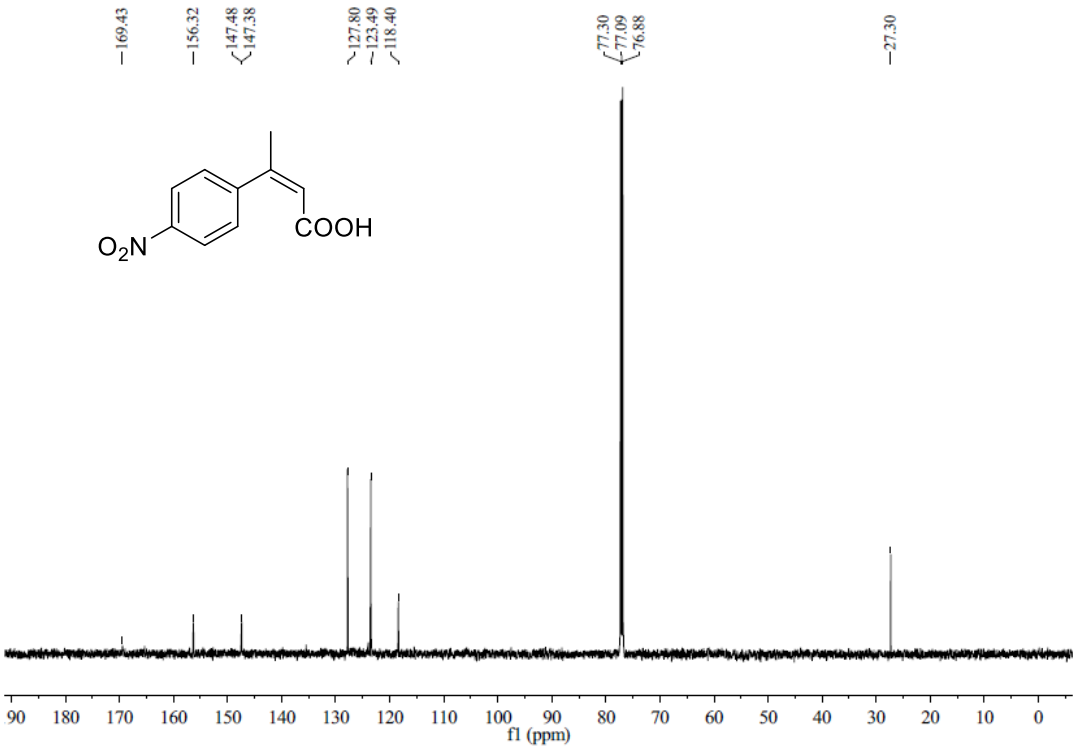


^{13}C NMR (150 MHz, Chloroform-*d*)

(*Z*)-3-(4-nitrophenyl)but-2-enoic acid (**1g**)

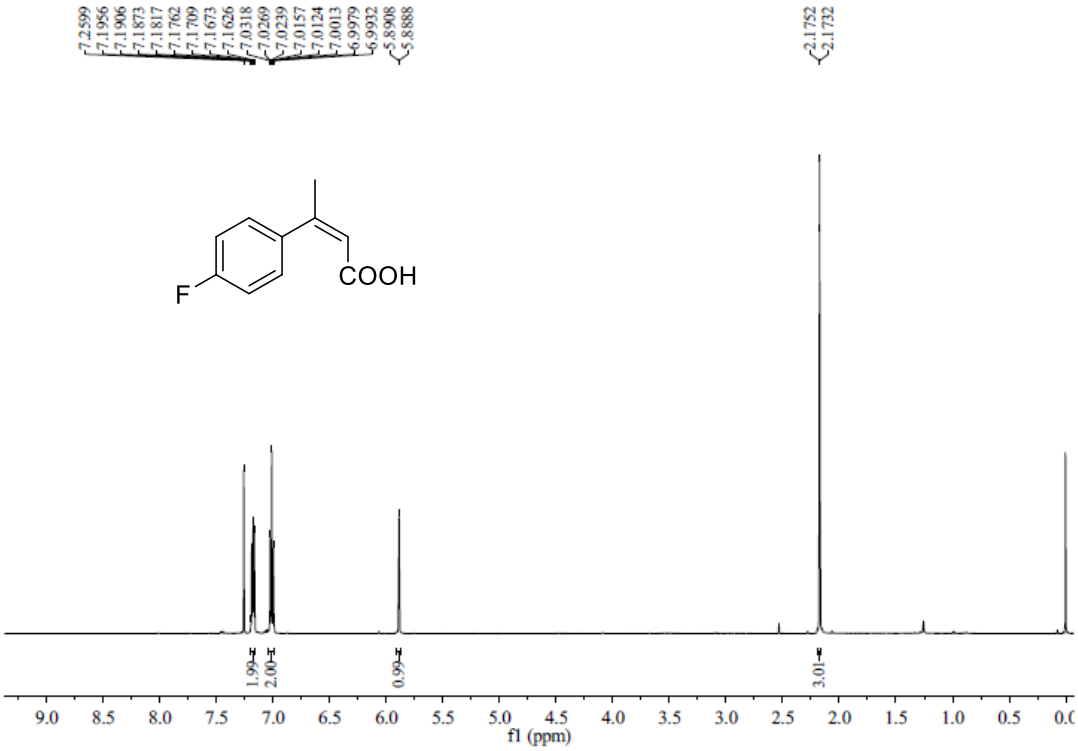


^{1}H NMR (400 MHz, Chloroform-*d*)

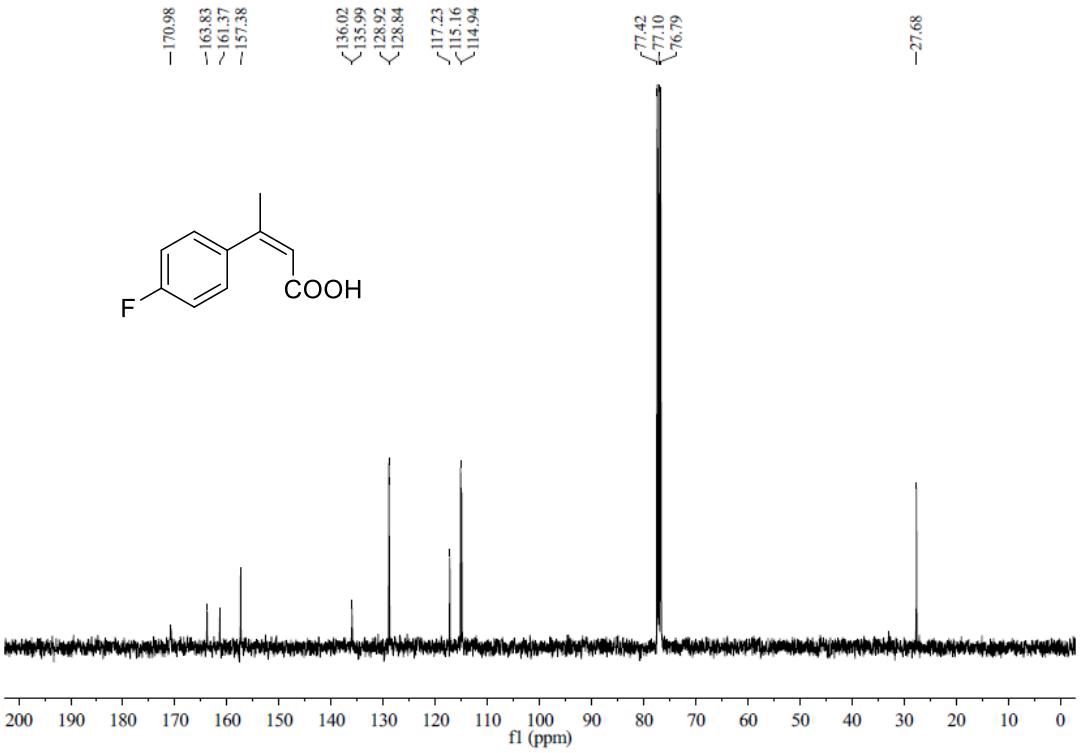


^{13}C NMR (150 MHz, Chloroform-*d*)

(*Z*)-3-(4-fluorophenyl)but-2-enoic acid (**1h**)

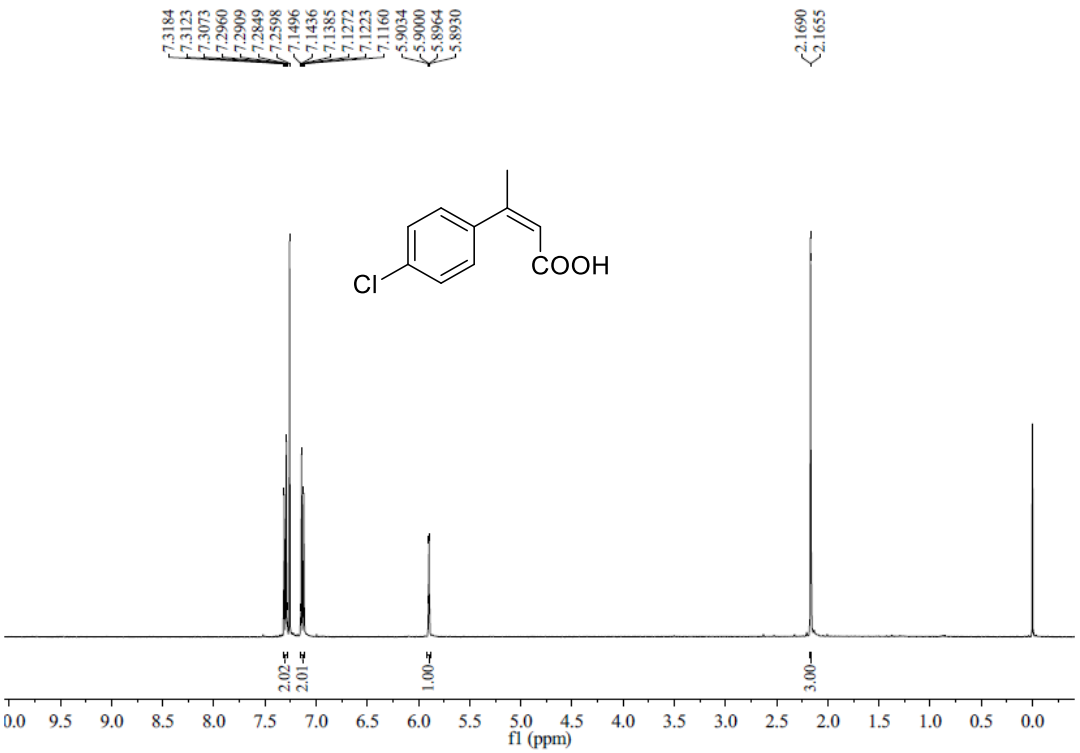


^{1}H NMR (600 MHz, Chloroform-*d*)

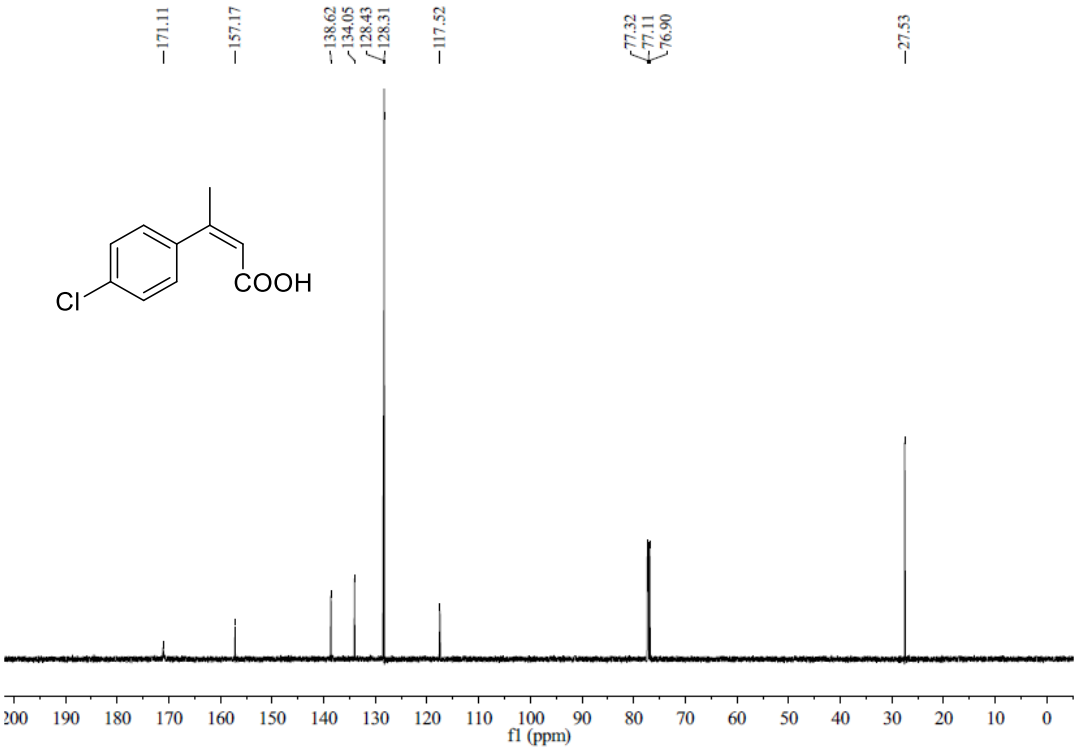


^{13}C NMR (100 MHz, Chloroform-*d*)

(*Z*)-3-(4-chlorophenyl)but-2-enoic acid (**1i**)

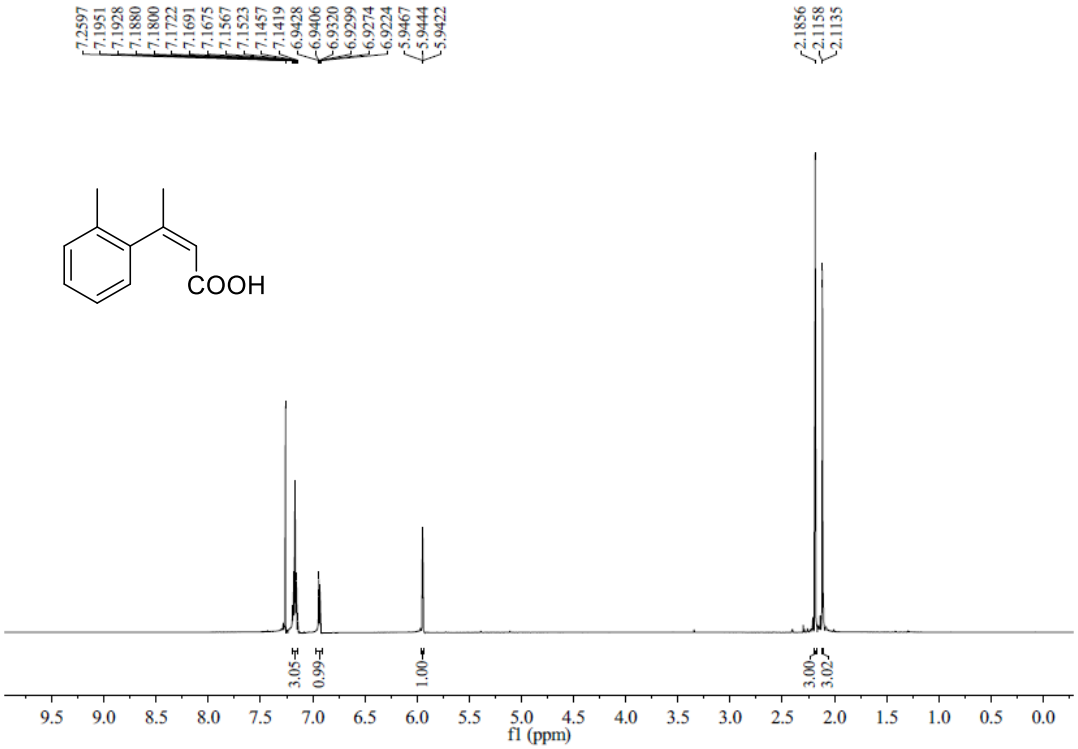


^{1}H NMR (400 MHz, Chloroform-*d*)

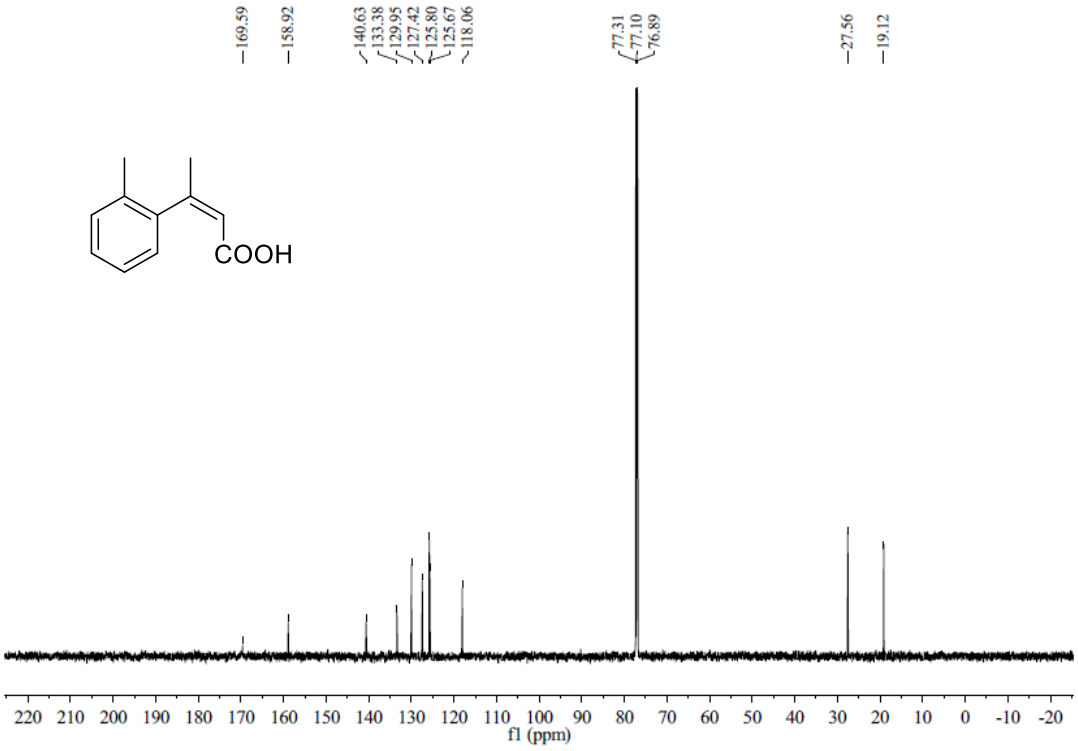


^{13}C NMR (150 MHz, Chloroform-*d*)

(*Z*)-3-(*o*-tolyl)but-2-enoic acid (**1j**)



^{1}H NMR (600 MHz, Chloroform-*d*)



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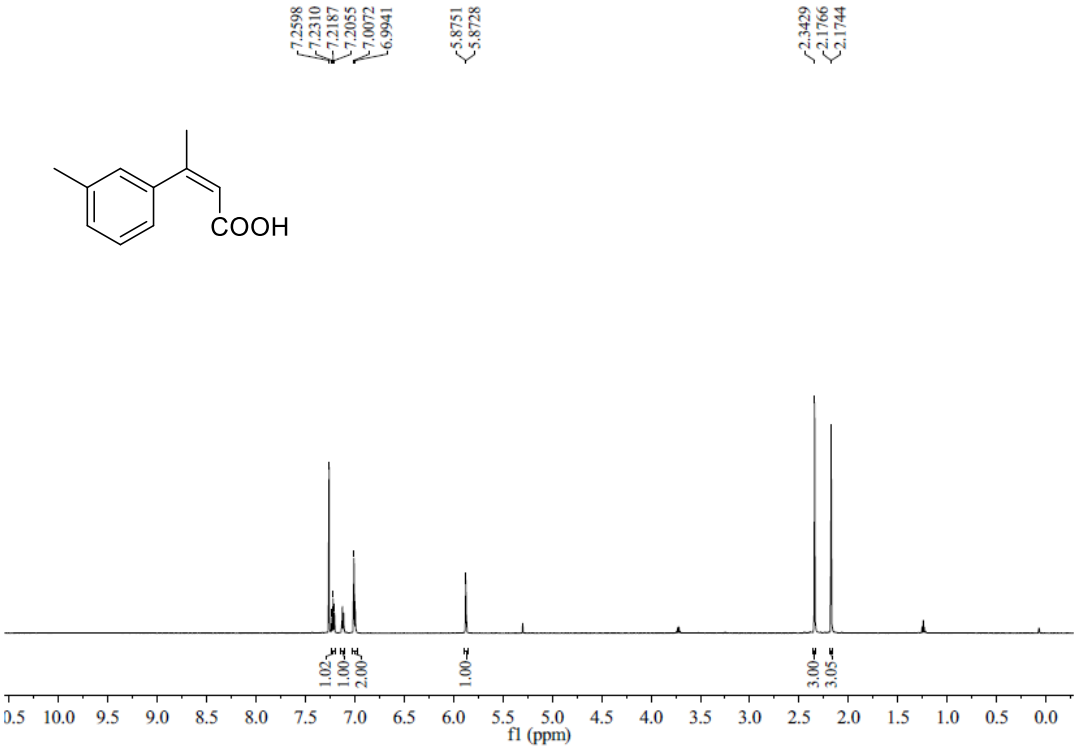
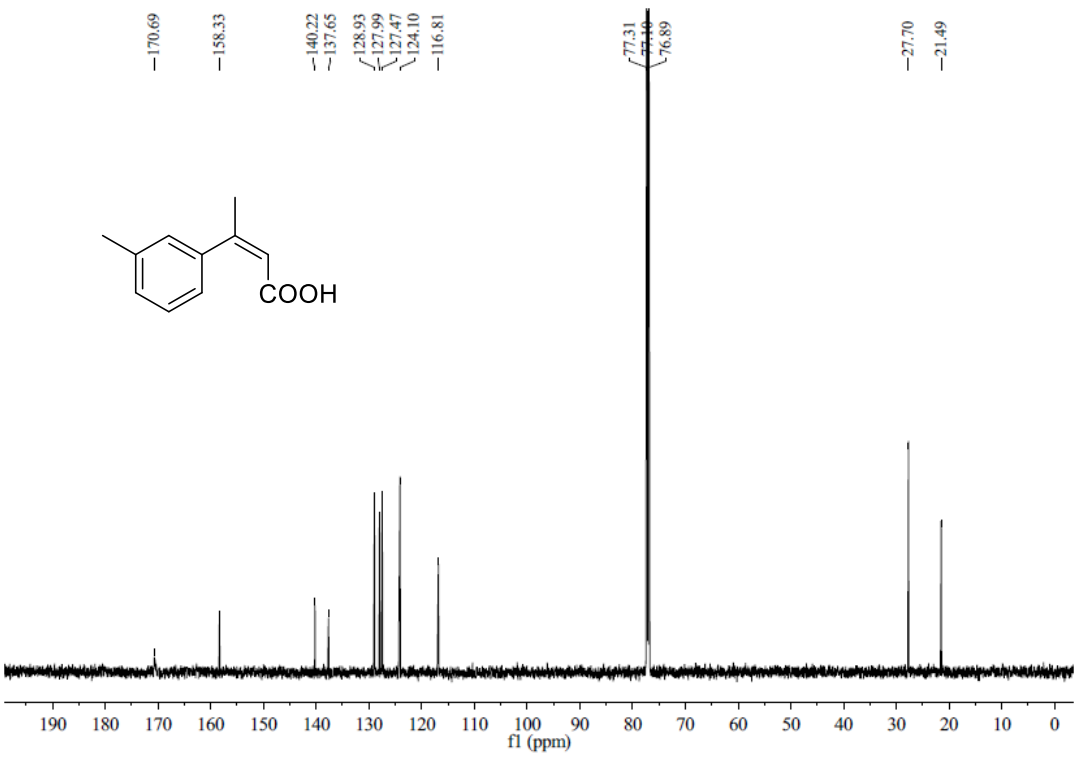
(*Z*)-3-(*m*-tolyl)but-2-enoic acid (**1k**)

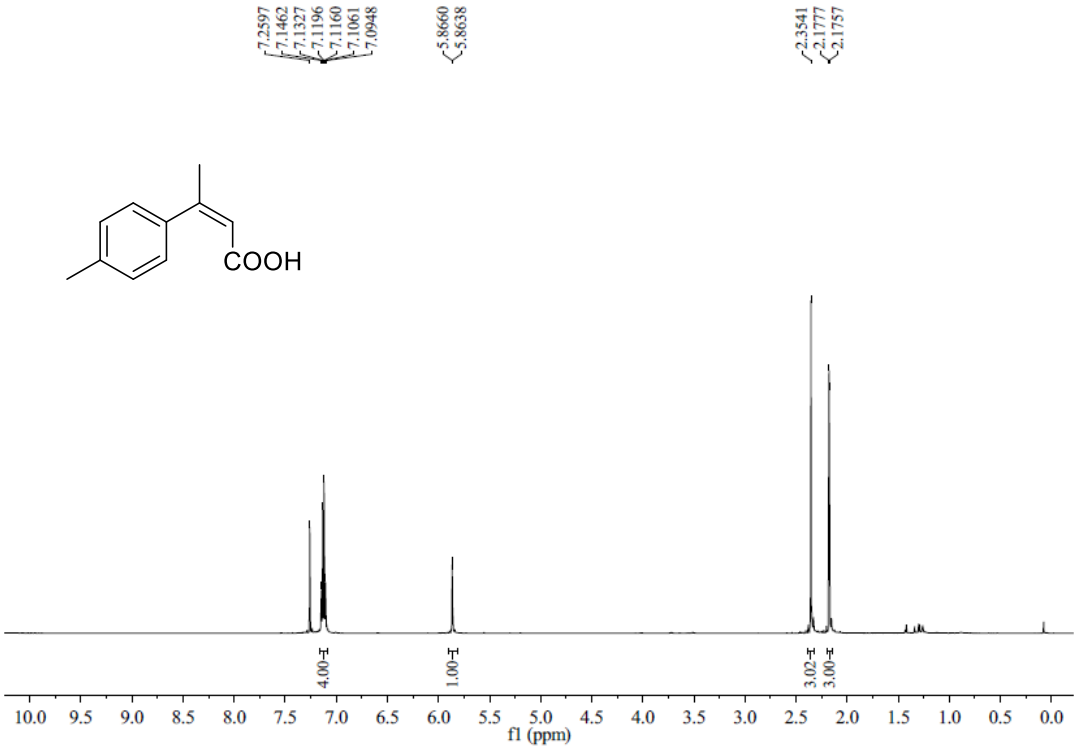


^{1}H NMR (600 MHz, Chloroform-*d*)

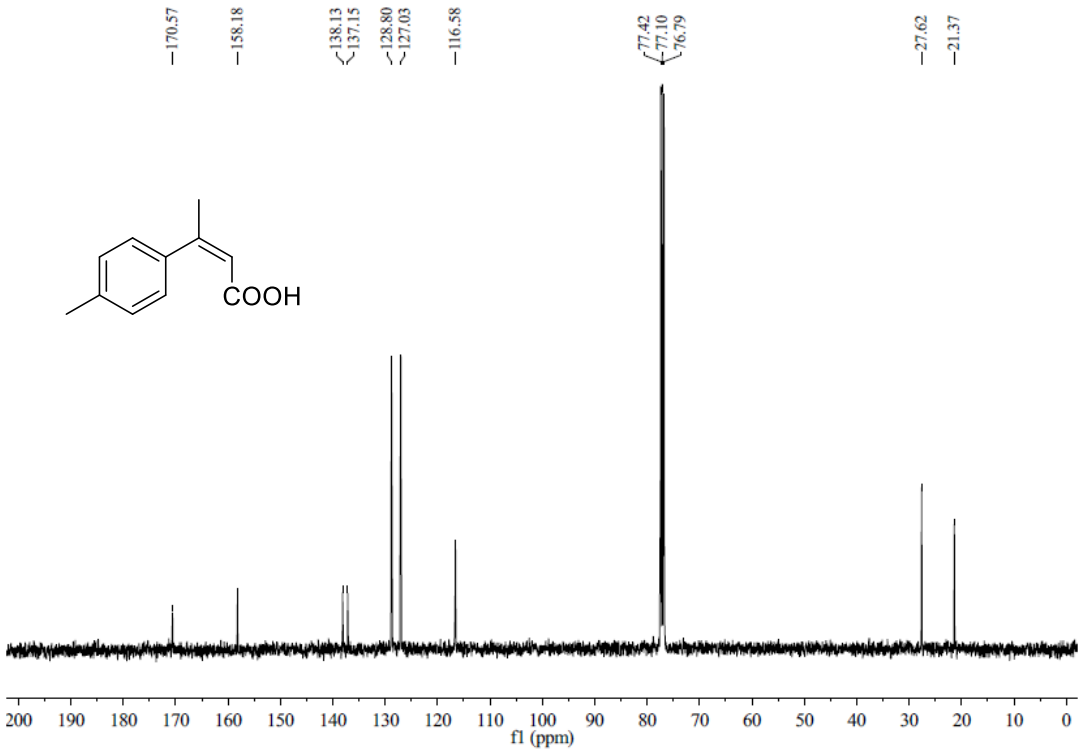


^{13}C NMR (150 MHz, Chloroform-*d*)

(*Z*)-3-(*p*-tolyl)but-2-enoic acid (**1l**)

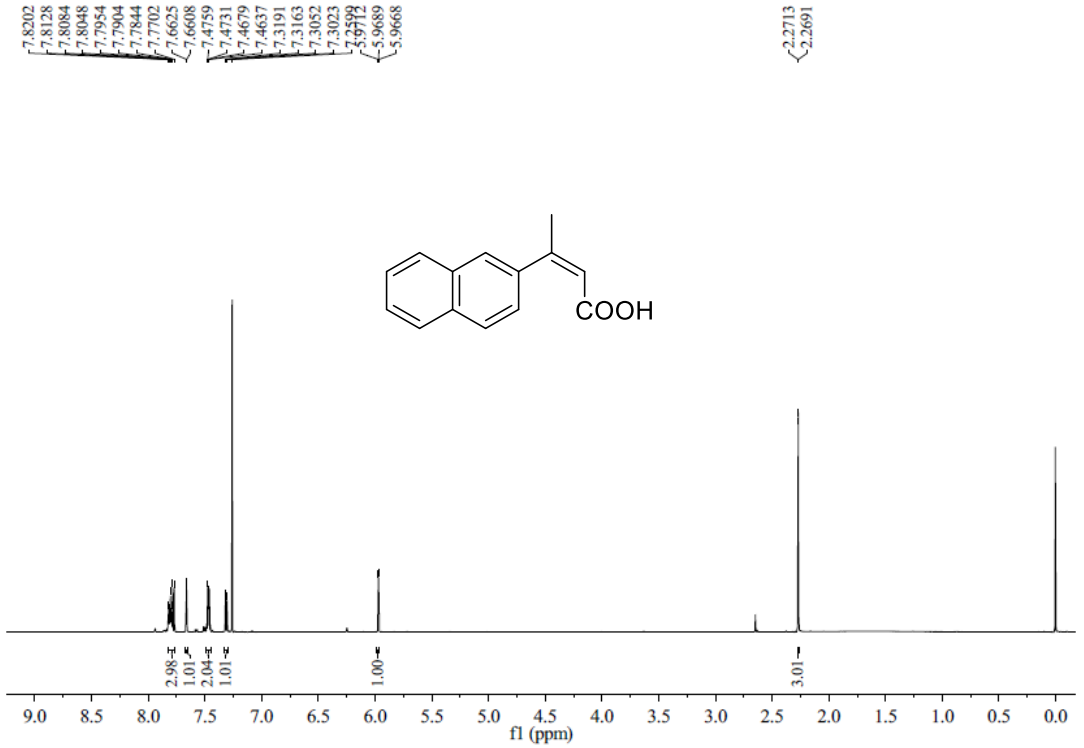


^{1}H NMR (600 MHz, Chloroform-*d*)

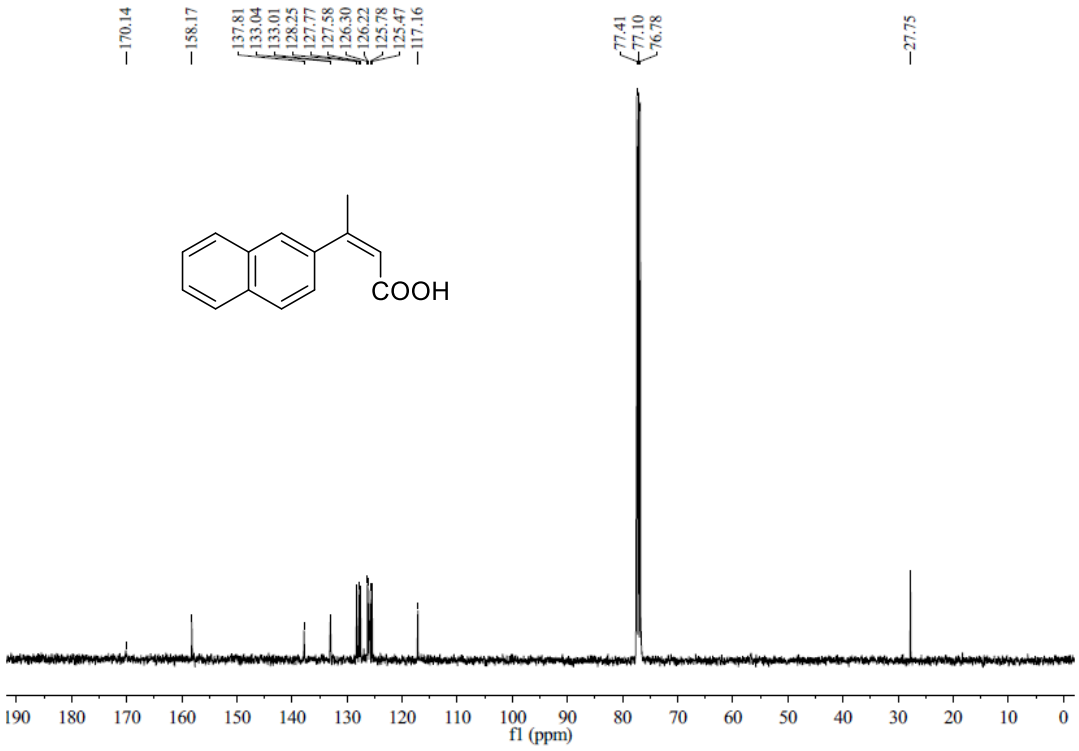


^{13}C NMR (100 MHz, Chloroform-*d*)

(*Z*)-3-(naphthalen-2-yl)but-2-enoic acid (**1m**)

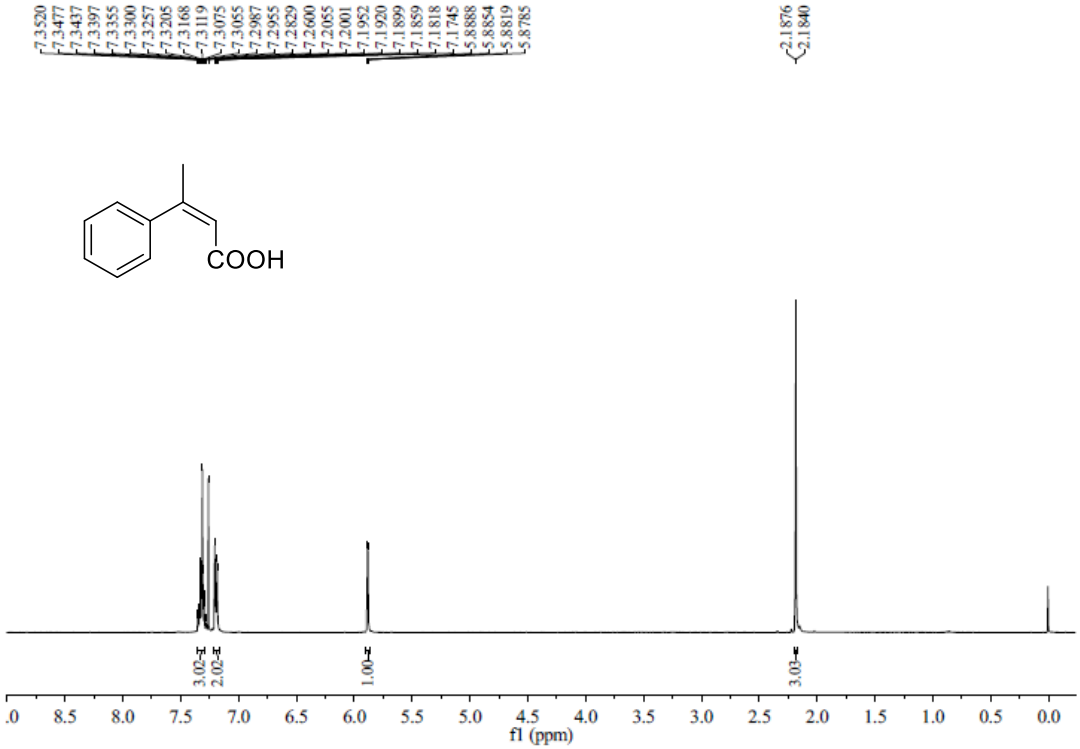


^{1}H NMR (600 MHz, Chloroform-*d*)

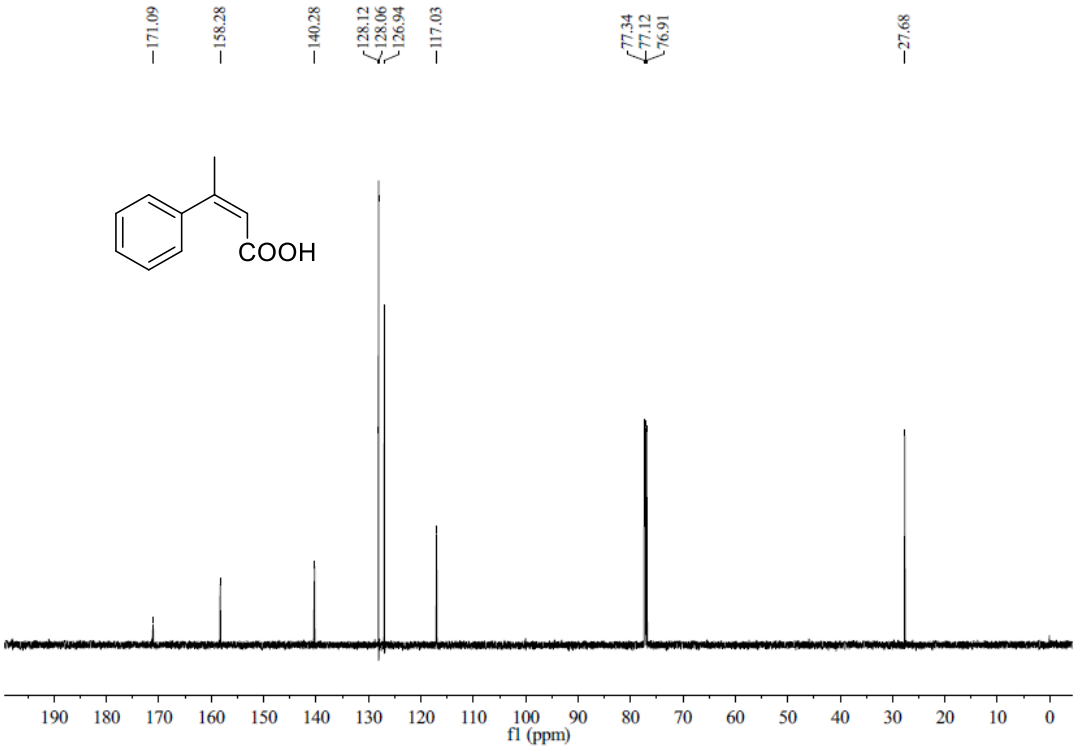


^{13}C NMR (100 MHz, Chloroform-*d*)

(*Z*)-3-phenylbut-2-enoic acid (**1n**)

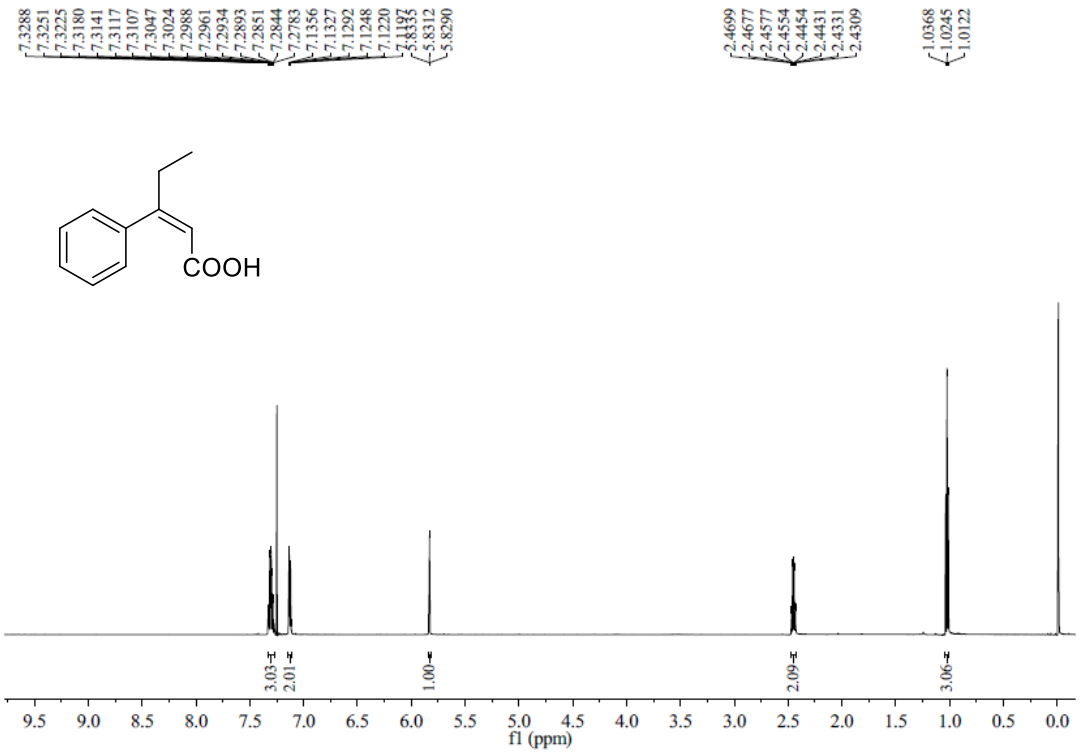


^{1}H NMR (400 MHz, Chloroform-*d*)

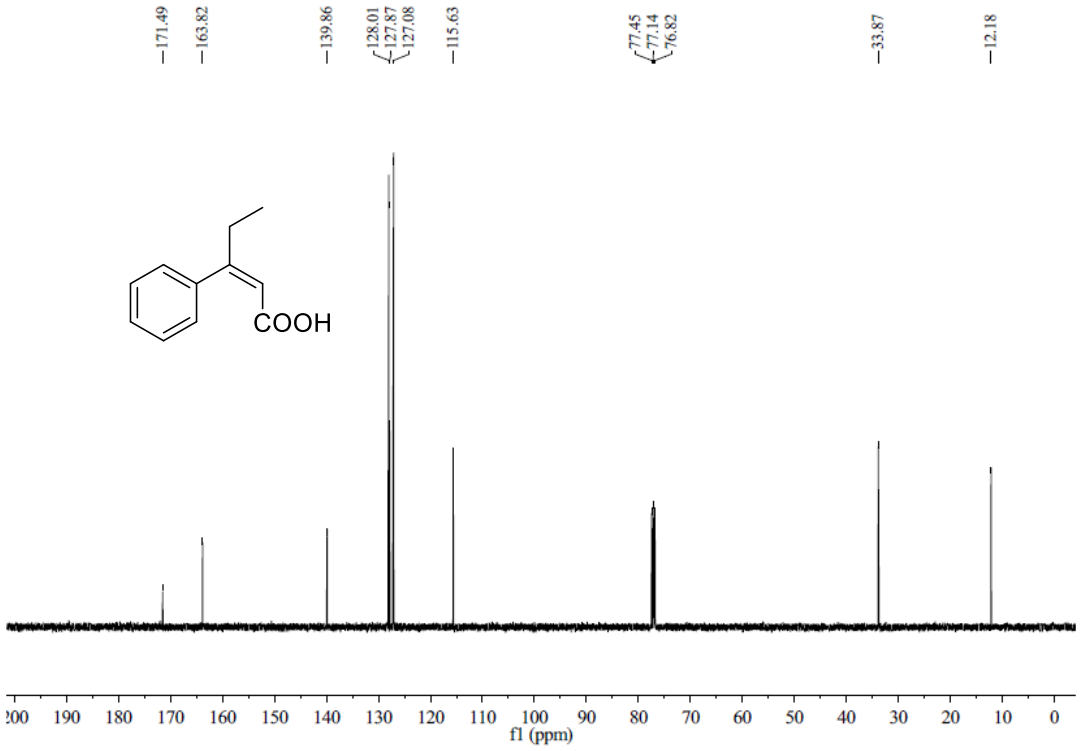


^{13}C NMR (150 MHz, Chloroform-*d*)

(*Z*)-3-phenylpent-2-enoic acid (**1o**)

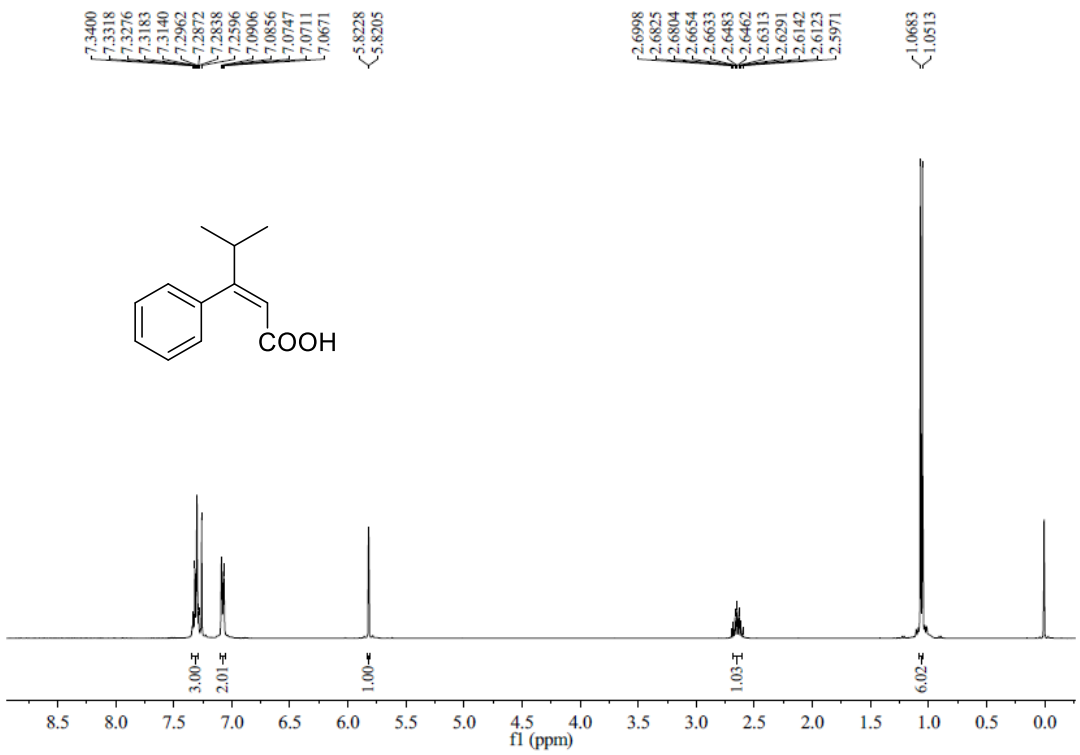


^{1}H NMR (600 MHz, Chloroform-*d*)

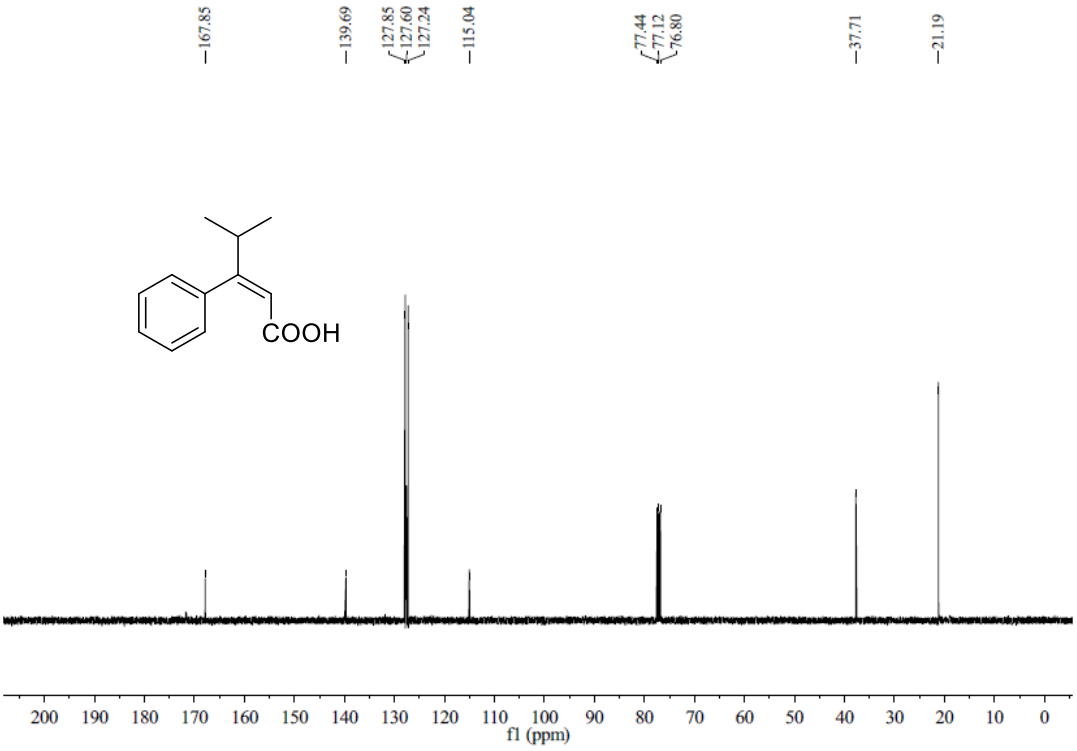


^{13}C NMR (100 MHz, Chloroform-*d*)

(*Z*)-4-methyl-3-phenylpent-2-enoic acid (**1p**)

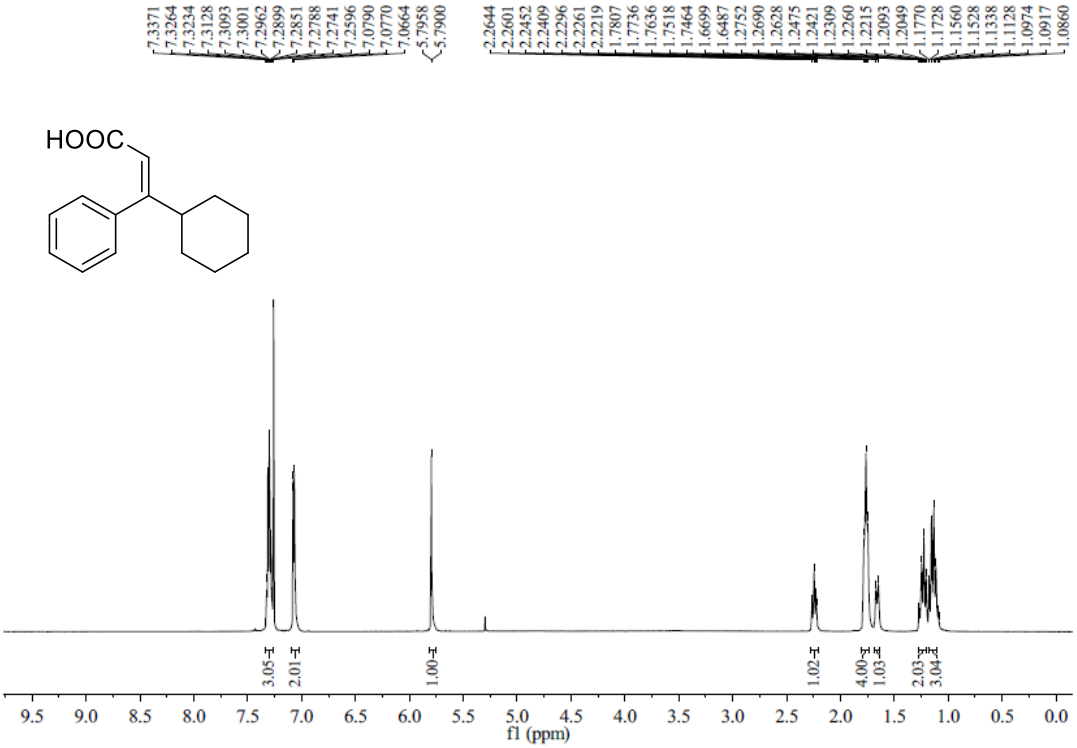


^{1}H NMR (400 MHz, Chloroform-*d*)



^{13}C NMR (100 MHz, Chloroform-*d*)

(*Z*)-3-cyclohexyl-3-phenylacrylic acid (**1q**)



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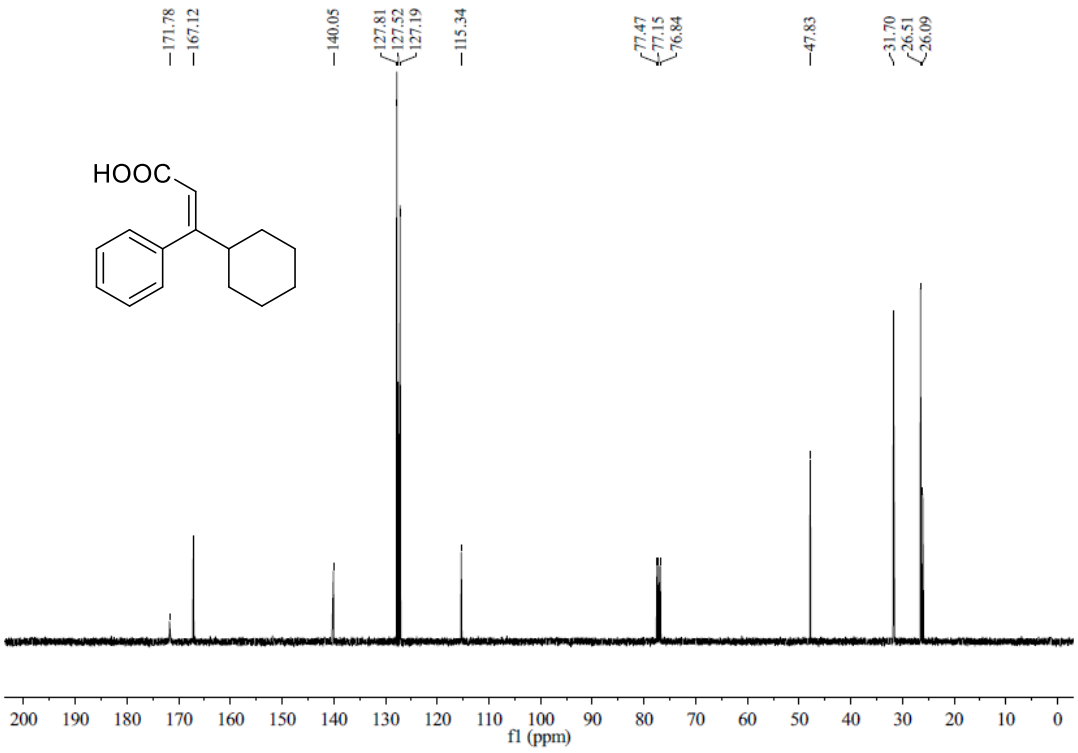
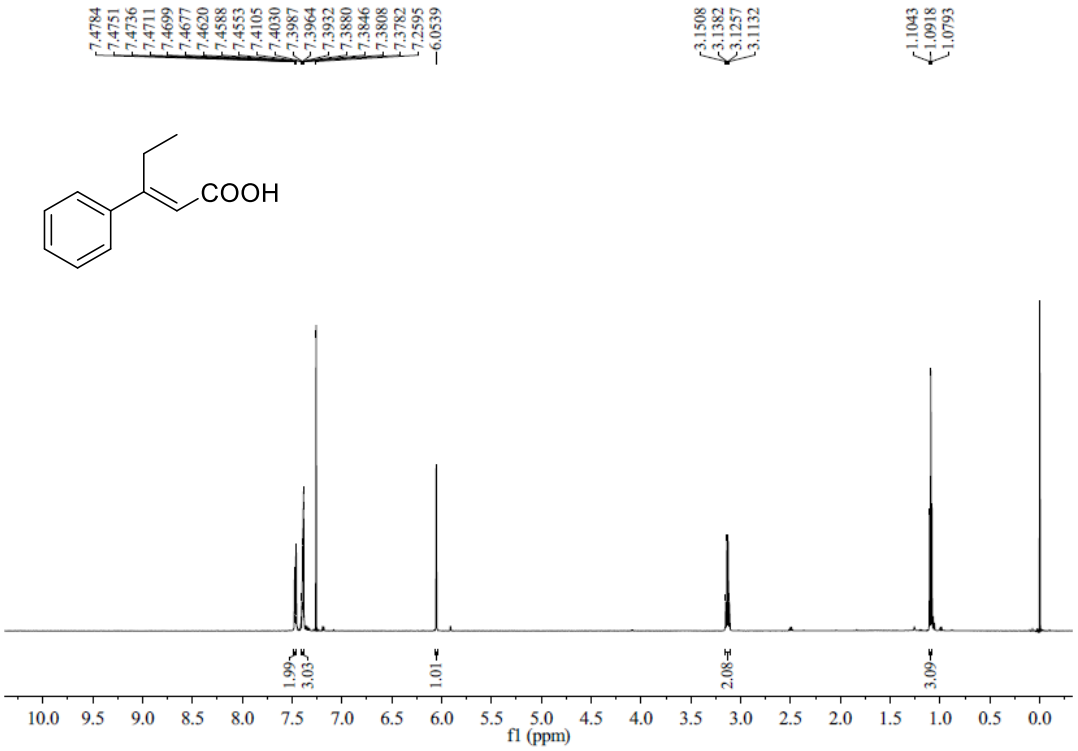


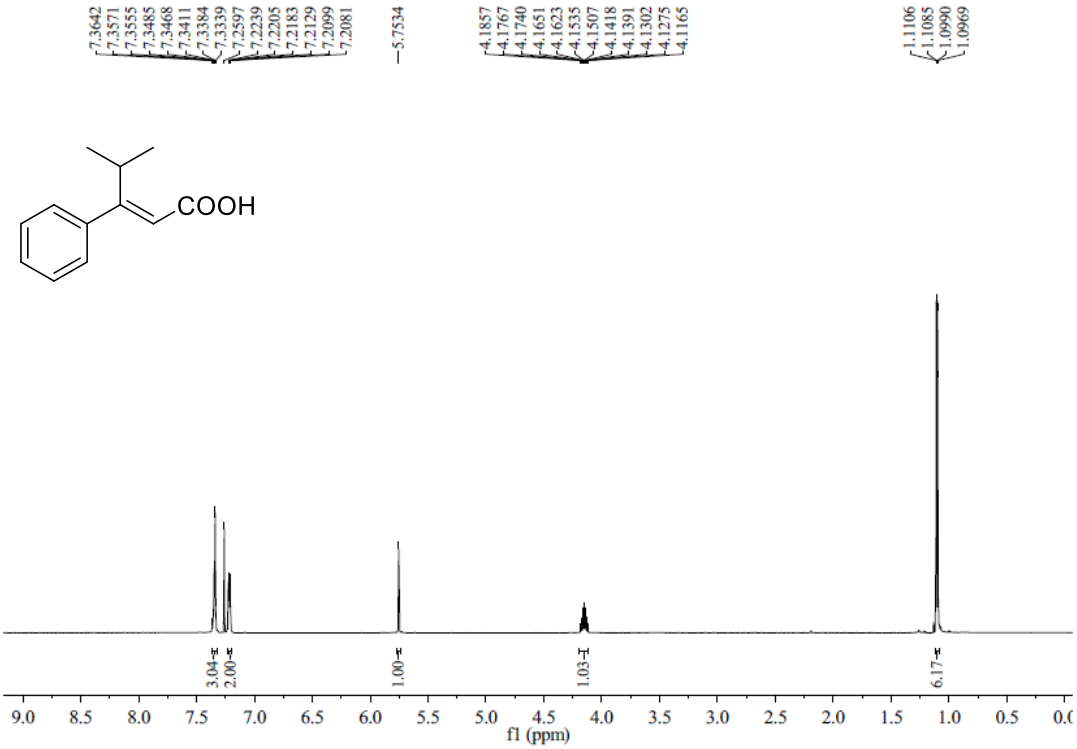
^{13}C NMR (100 MHz, Chloroform-*d*)

(*E*)-3-phenylpent-2-enoic acid (**1r**)



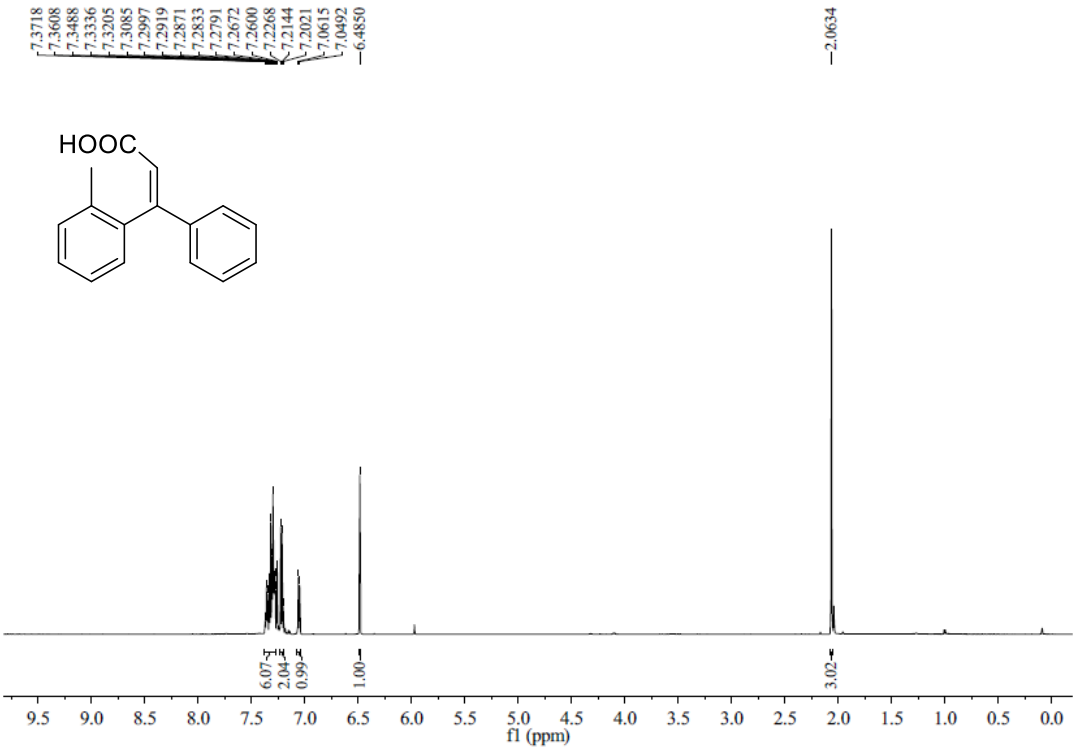
^{1}H NMR (600 MHz, Chloroform-*d*)

(*E*)-4-methyl-3-phenylpent-2-enoic acid (**1s**)

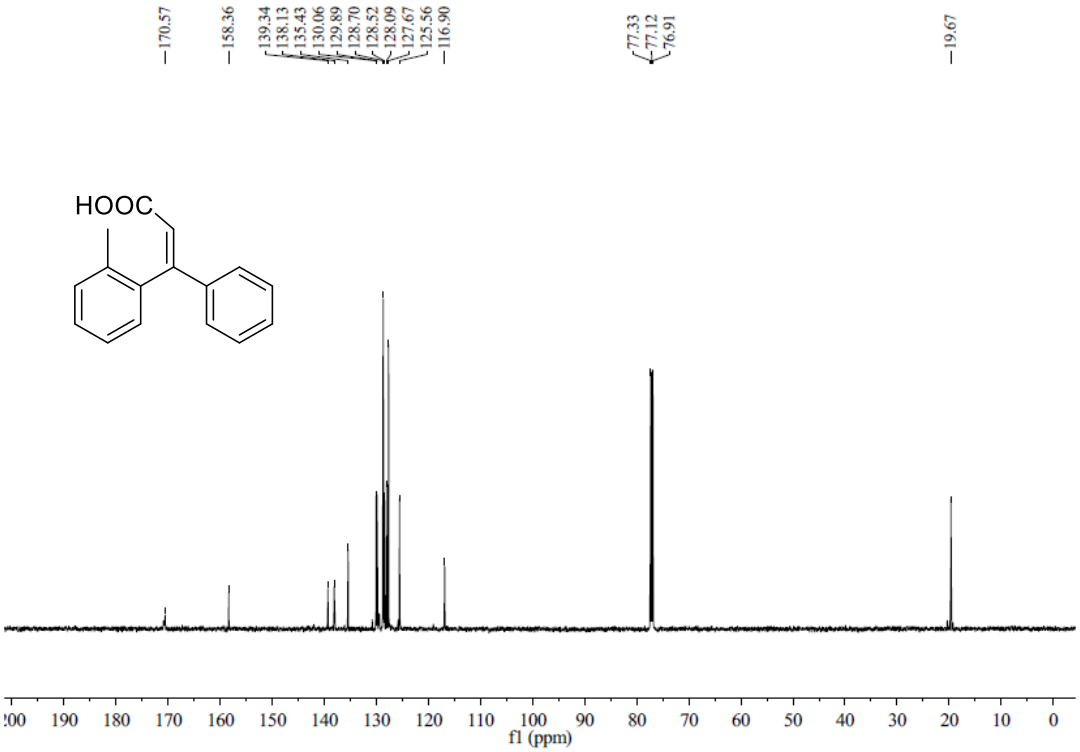


^{1}H NMR (600 MHz, Chloroform-*d*)

(Z)-3-phenyl-3-(*o*-tolyl)acrylic acid (**1t**)

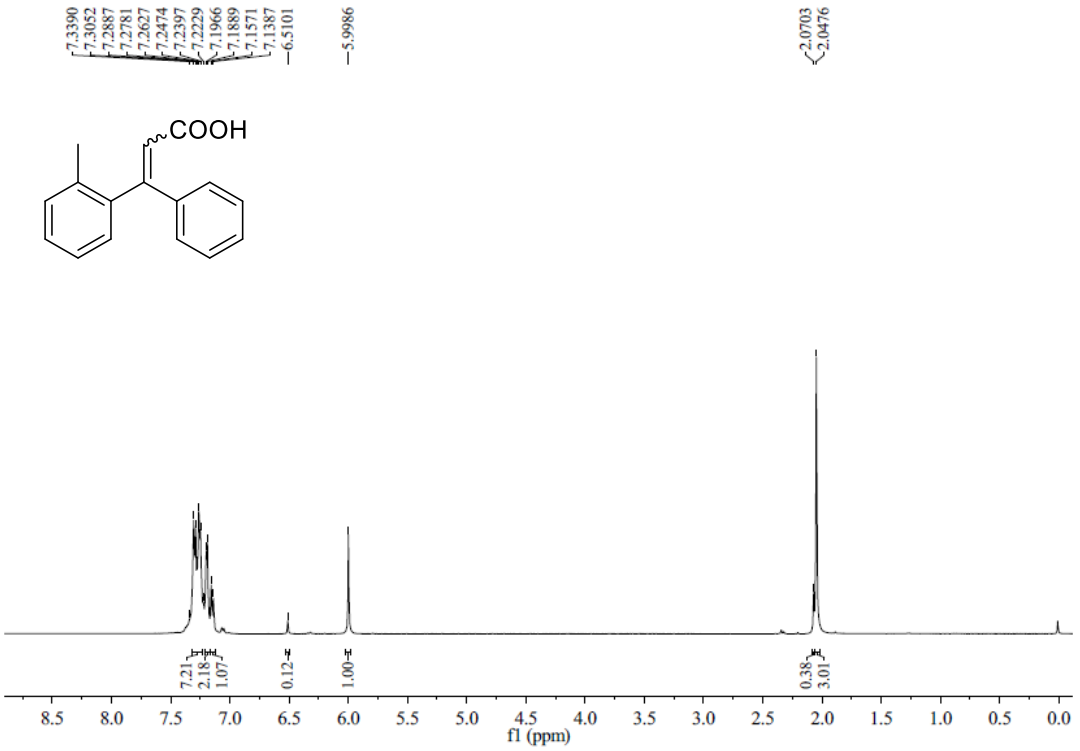


^{1}H NMR (600 MHz, Chloroform-*d*)

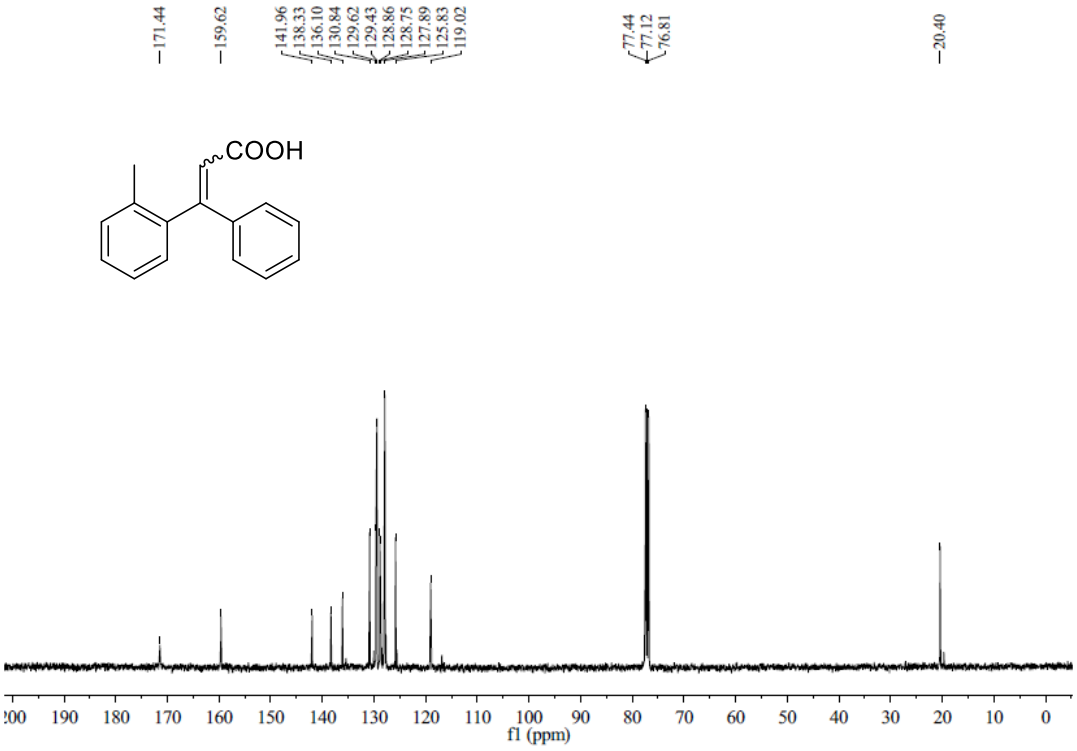


^{13}C NMR (150 MHz, Chloroform-*d*)

3-phenyl-3-(*o*-tolyl)acrylic acid (**1u**) (*E*:*Z* =8:1)

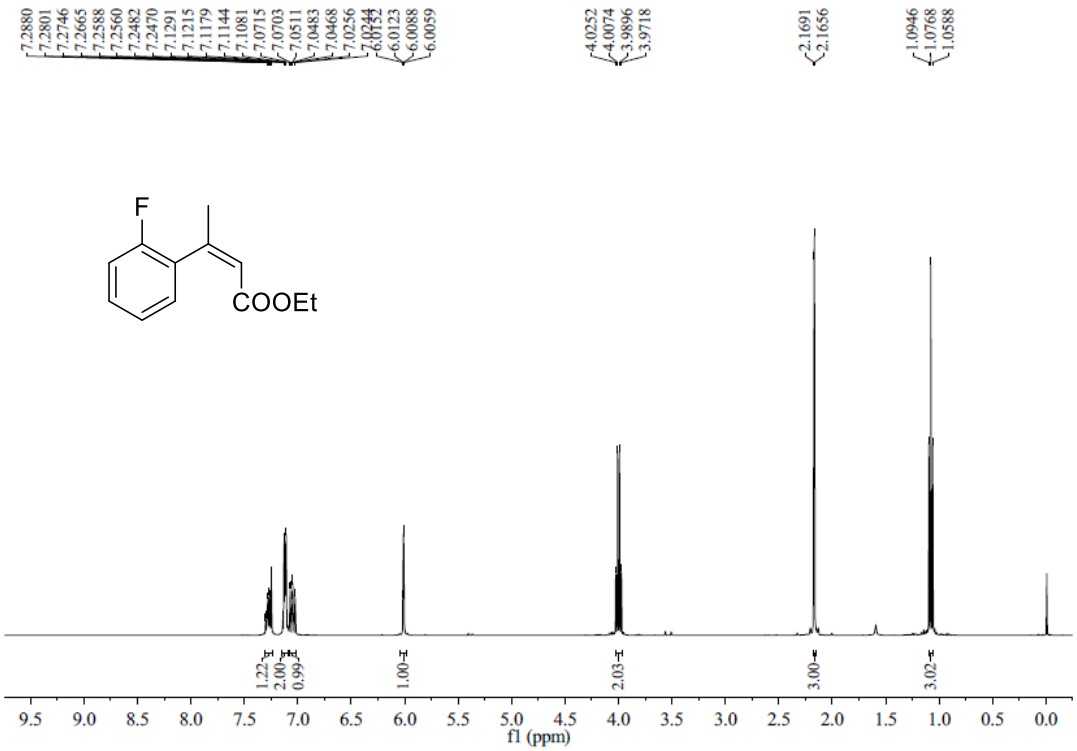


^{1}H NMR (400 MHz, Chloroform-*d*)

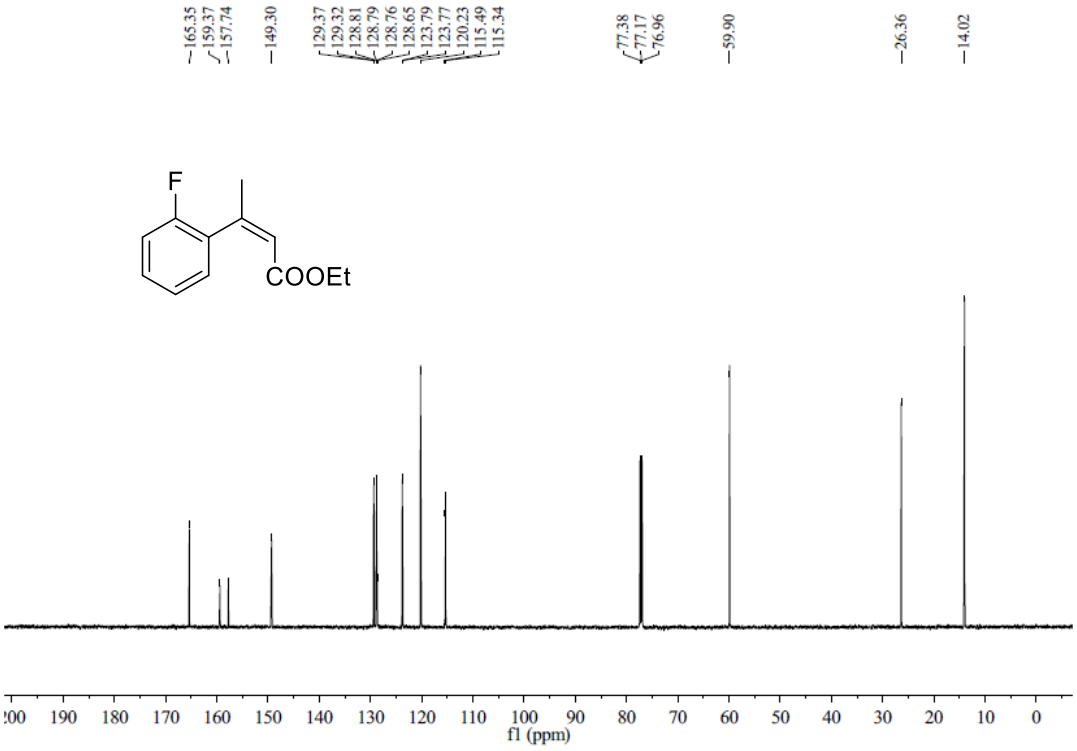


^{13}C NMR (150 MHz, Chloroform-*d*)

ethyl (Z)-(2-fluorophenyl)but-2-enoate (**1v**)

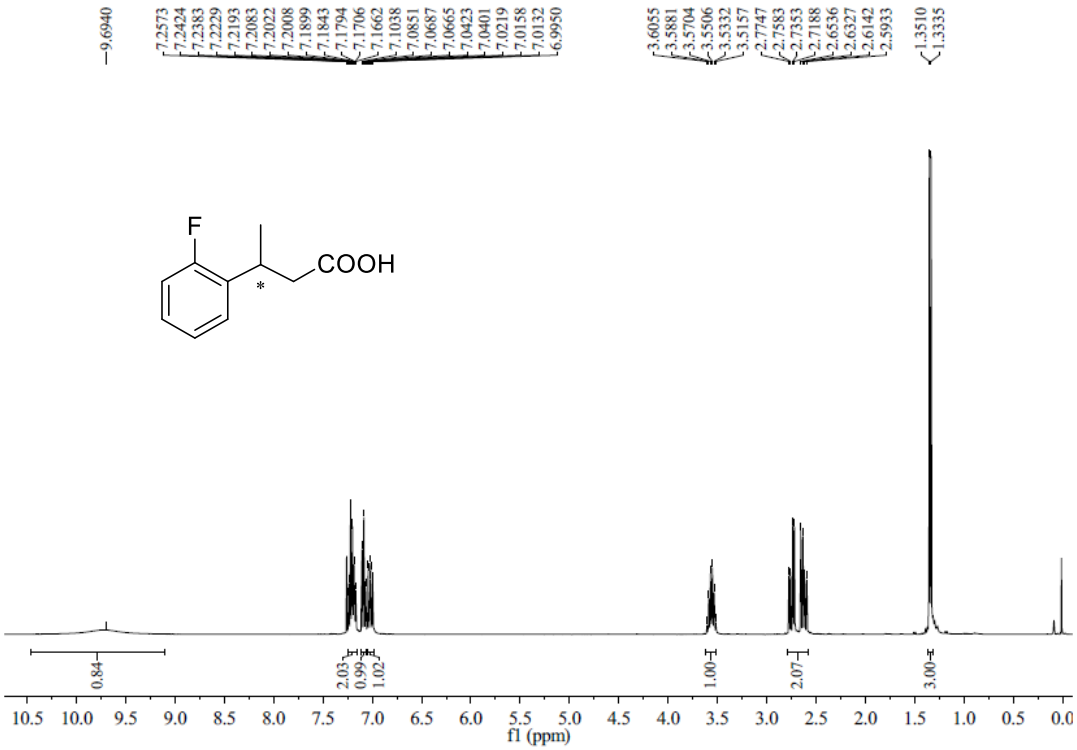


^{1}H NMR (400 MHz, Chloroform-*d*)



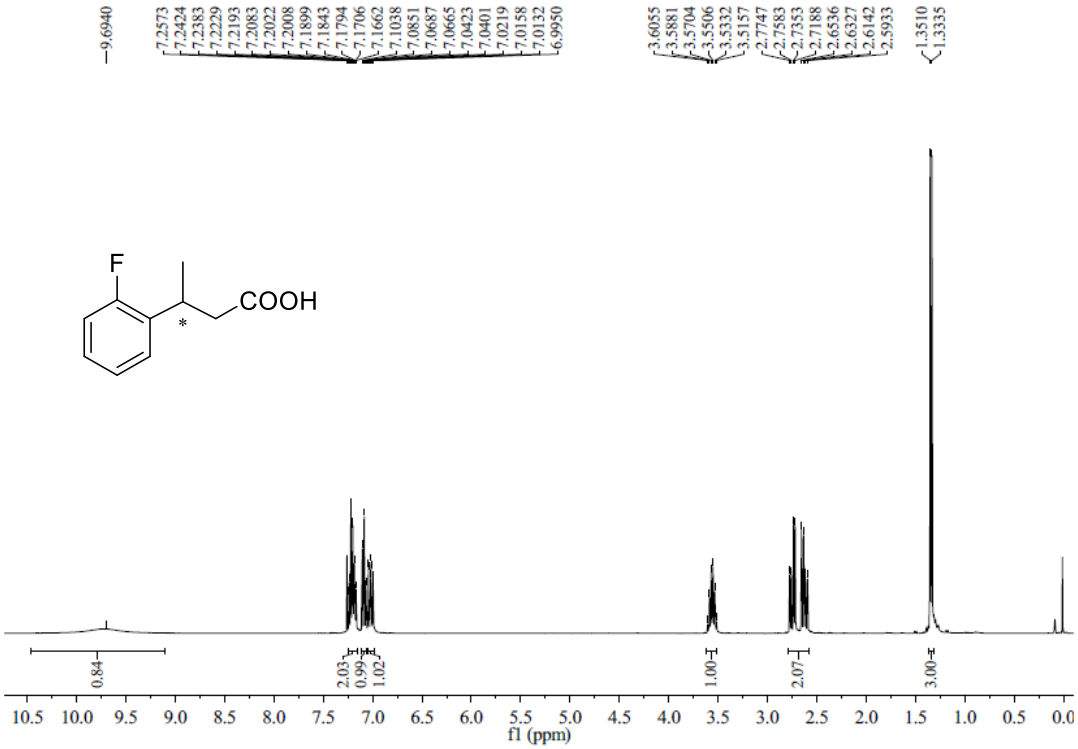
^{13}C NMR (150 MHz, Chloroform-*d*)

(+)-3-(2-fluorophenyl)butanoic acid (**2a**)



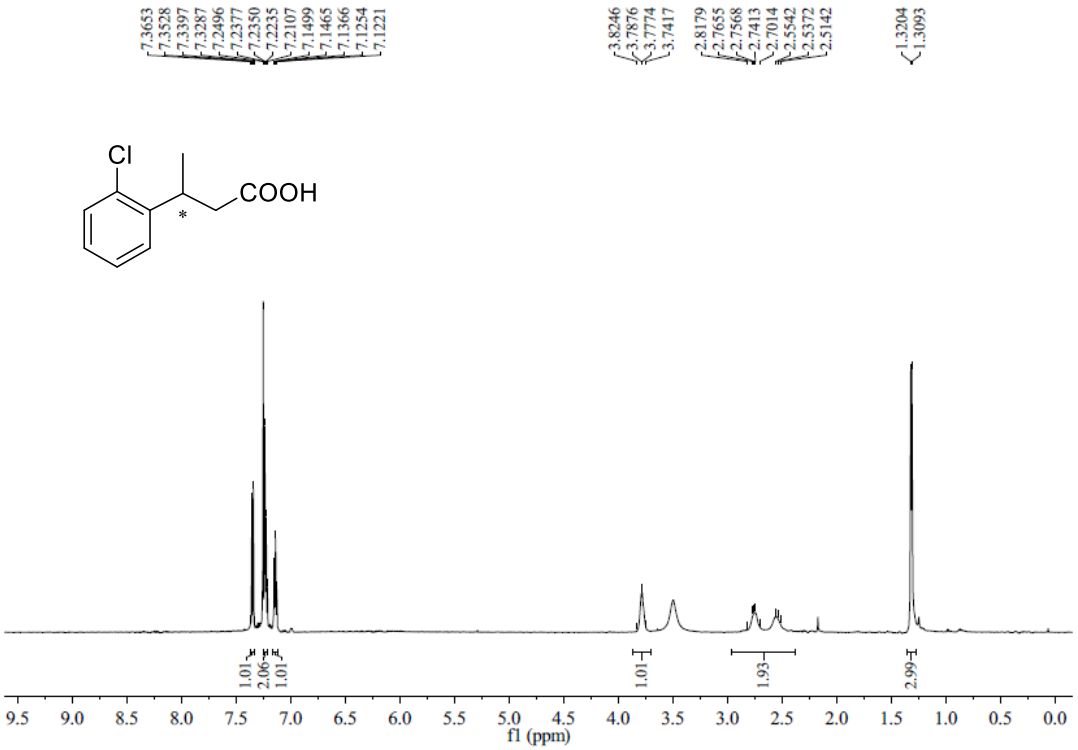
^{1}H NMR (400 MHz, Chloroform-*d*)

(-)-3-(2-fluorophenyl)butanoic acid (**2b**)



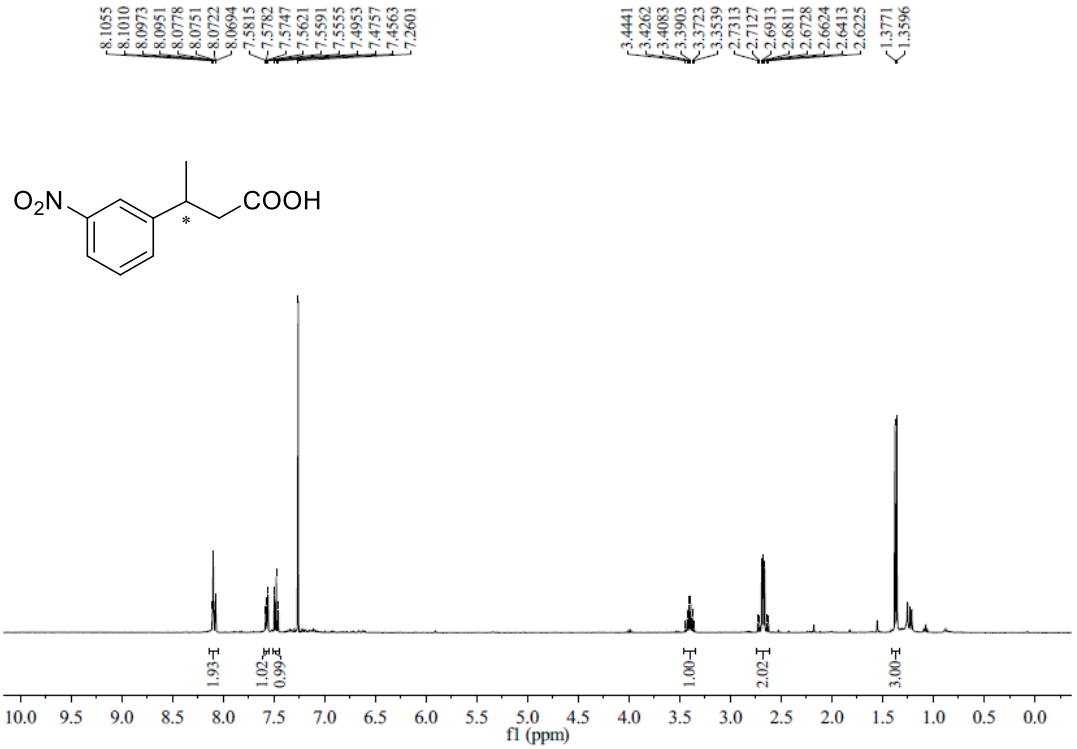
^{1}H NMR (400 MHz, Chloroform-*d*)

(-)-3-(2-chlorophenyl)butanoic acid (**2c**)



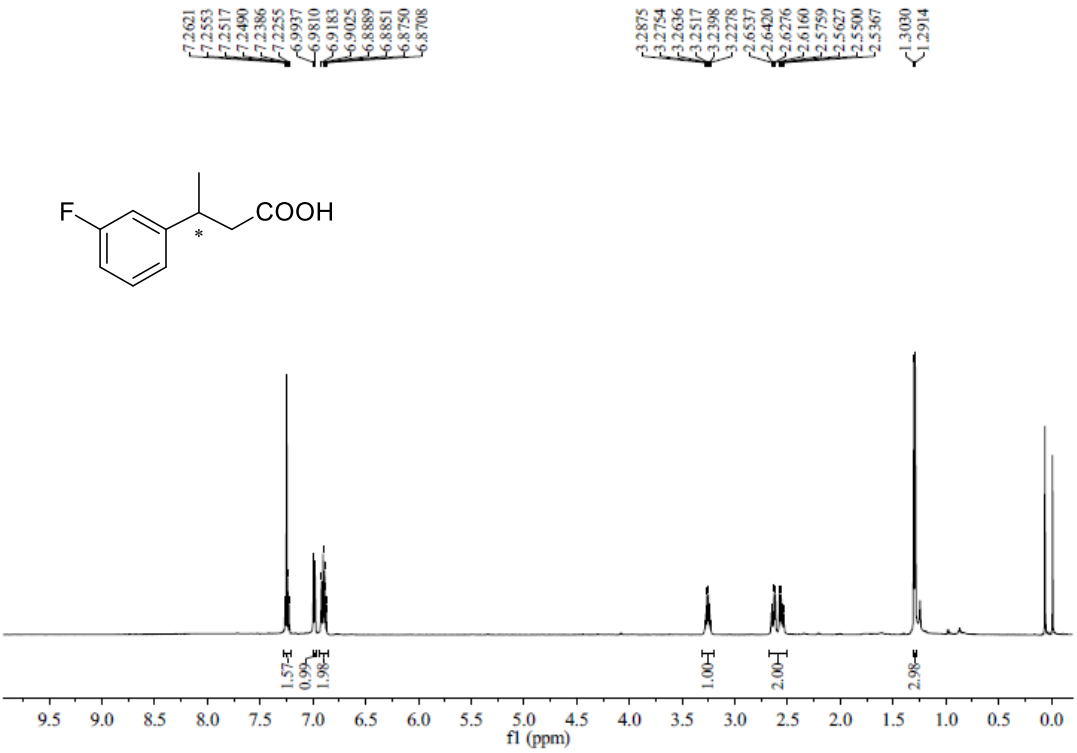
^{1}H NMR (600 MHz, Chloroform-*d*)

(-)-3-(3-nitrophenyl)butanoic acid (**2d**)



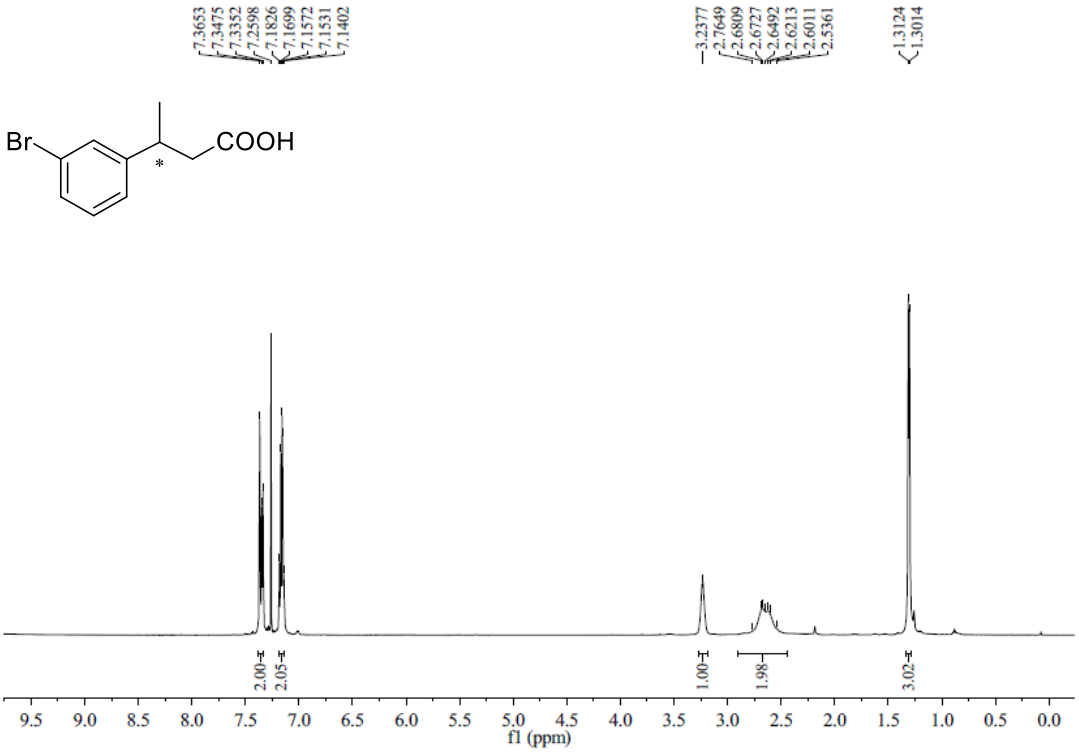
^{1}H NMR (400 MHz, Chloroform-*d*)

(-)-3-(3-fluorophenyl)butanoic acid (**2e**)



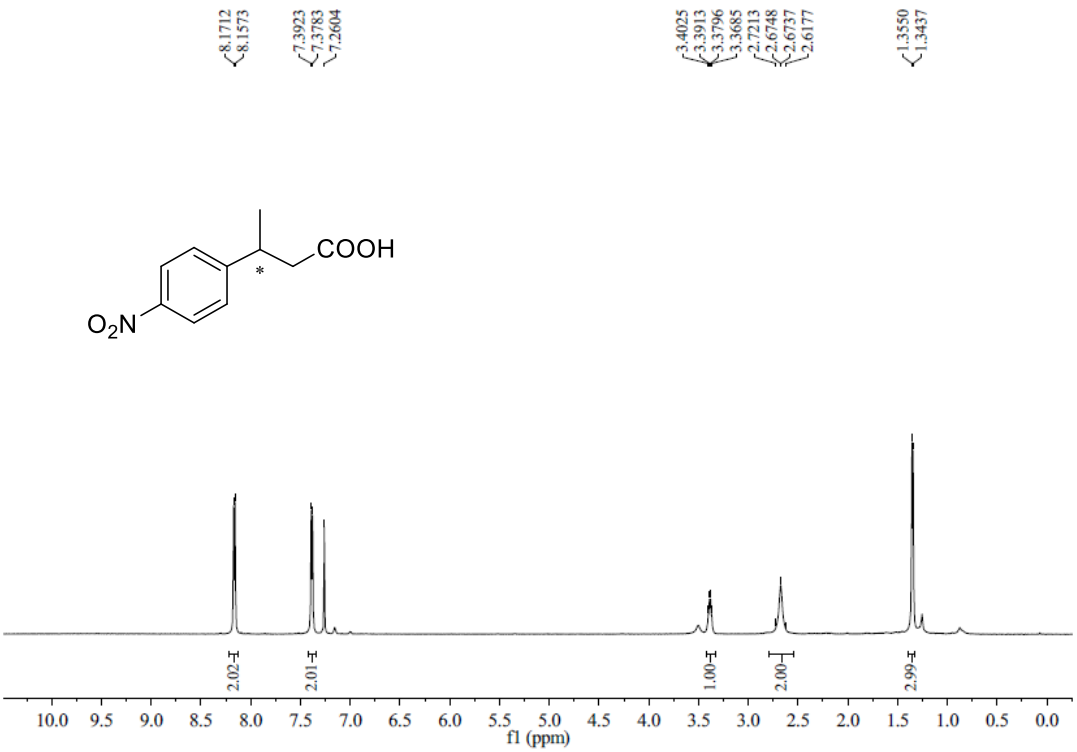
^{1}H NMR (600 MHz, Chloroform-*d*)

(-)-3-(3-bromophenyl)butanoic acid (**2f**)



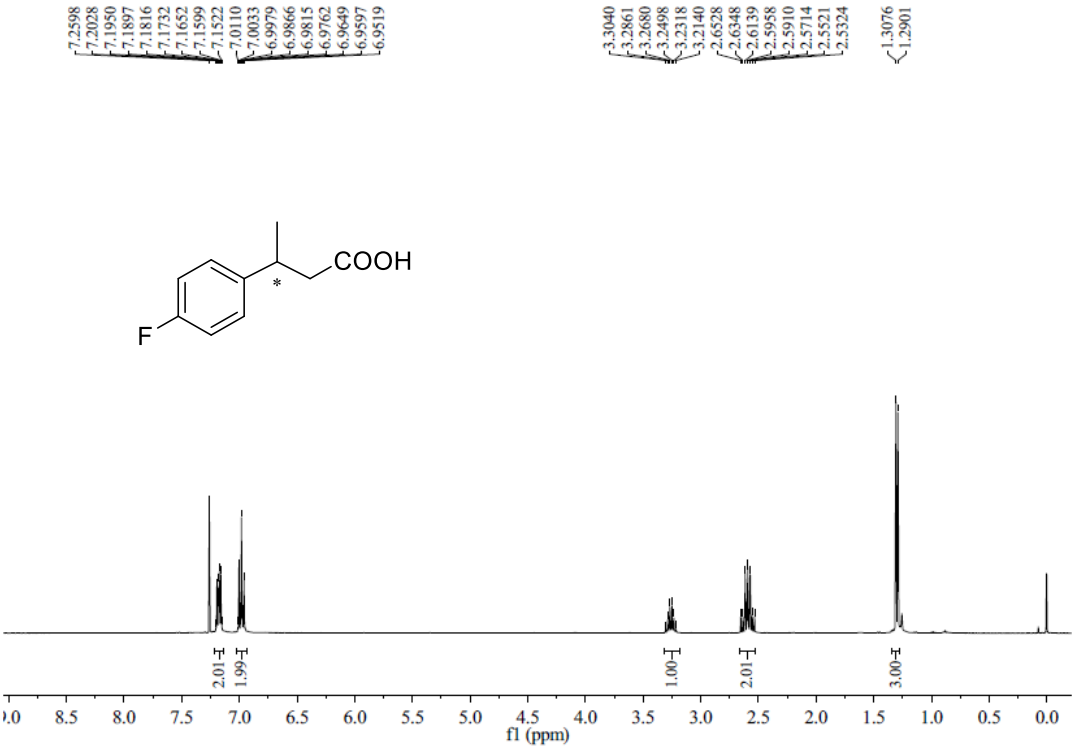
^{1}H NMR (600 MHz, Chloroform-*d*)

(-)-3-(4-nitrophenyl)butanoic acid (**2g**)



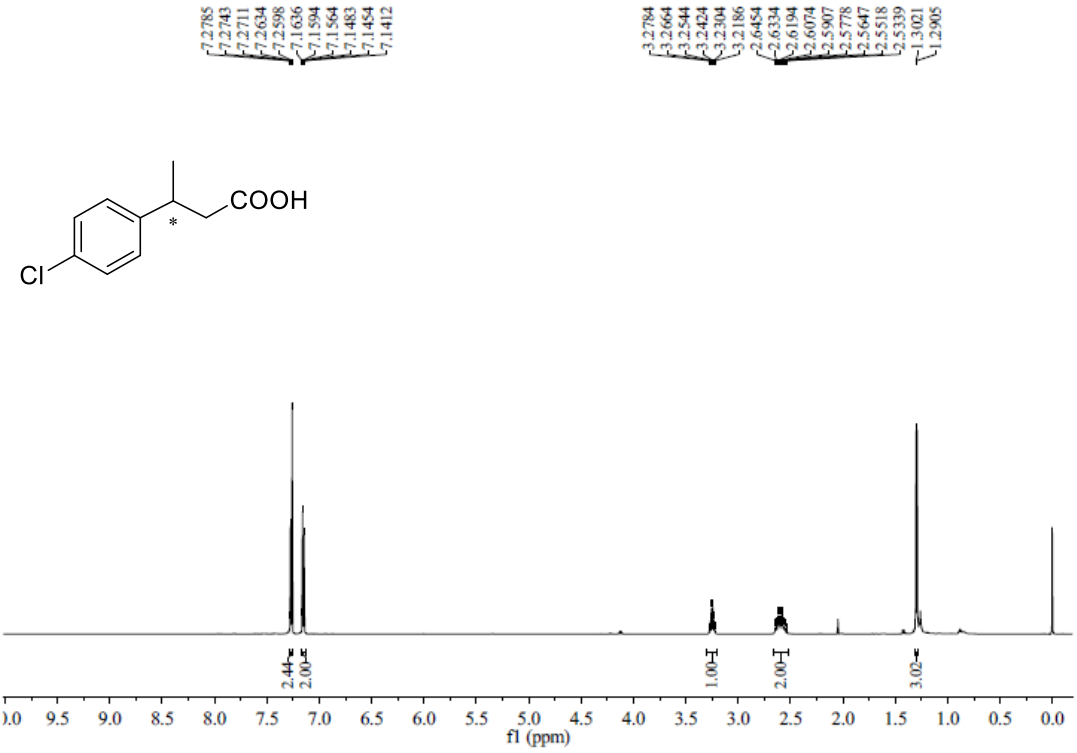
^{1}H NMR (600 MHz, Chloroform-*d*)

(-)-3-(4-fluorophenyl)butanoic acid (**2h**)



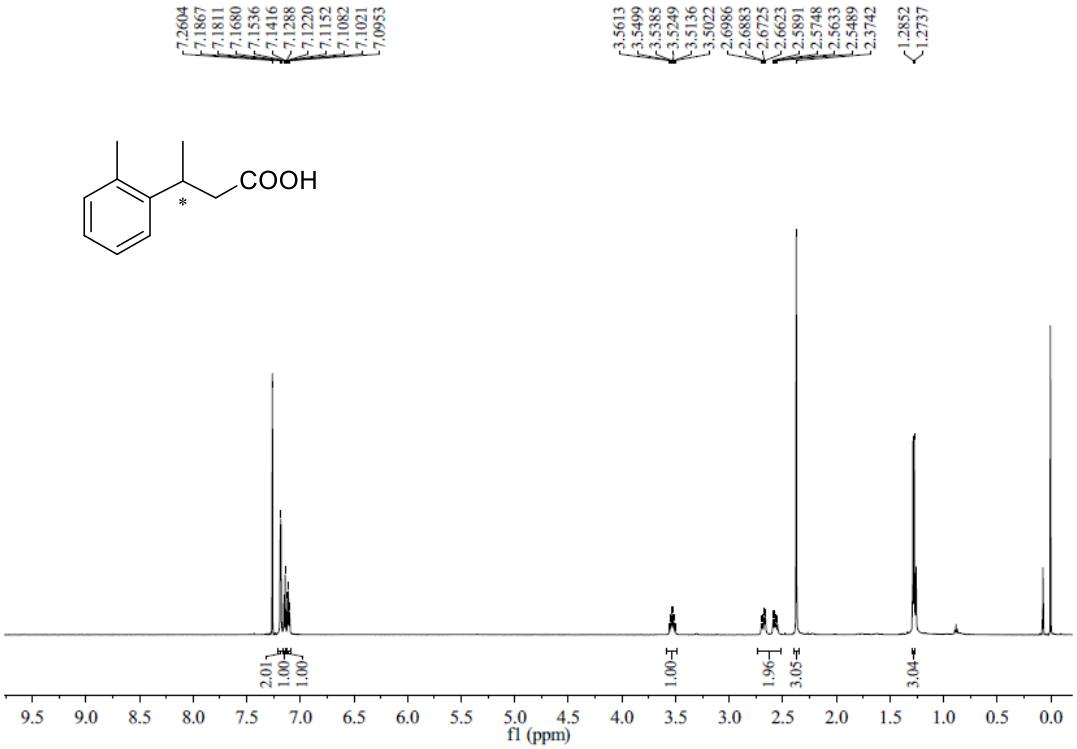
^{1}H NMR (400 MHz, Chloroform-*d*)

(-)-3-(4-chlorophenyl)butanoic acid (**2i**)



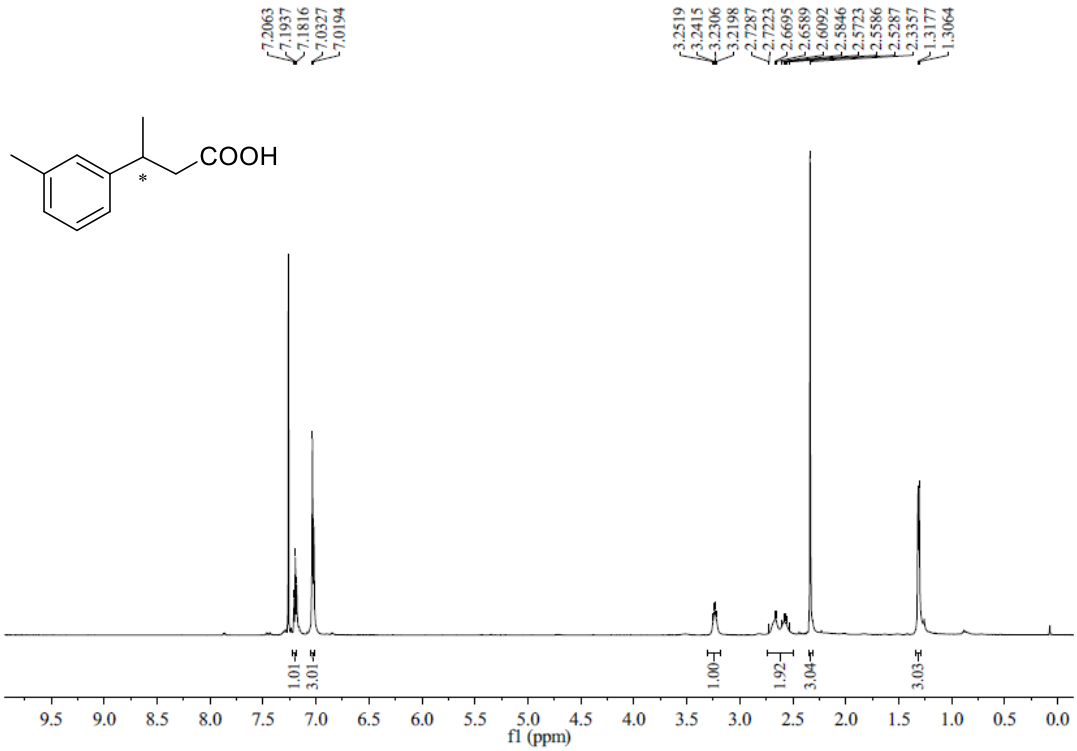
^{1}H NMR (600 MHz, Chloroform-*d*)

(-)-3-(*o*-tolyl)butanoic acid (**2j**)



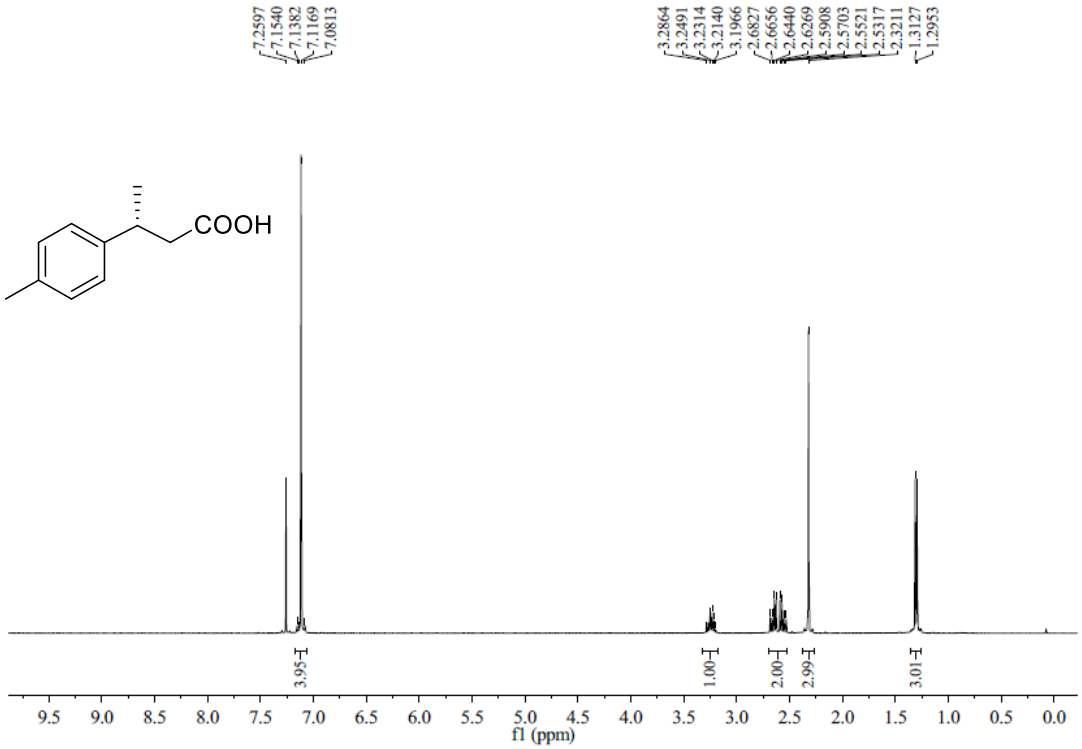
^{1}H NMR (600 MHz, Chloroform-*d*)

(-)-3-(*m*-tolyl)butanoic acid (**2k**)



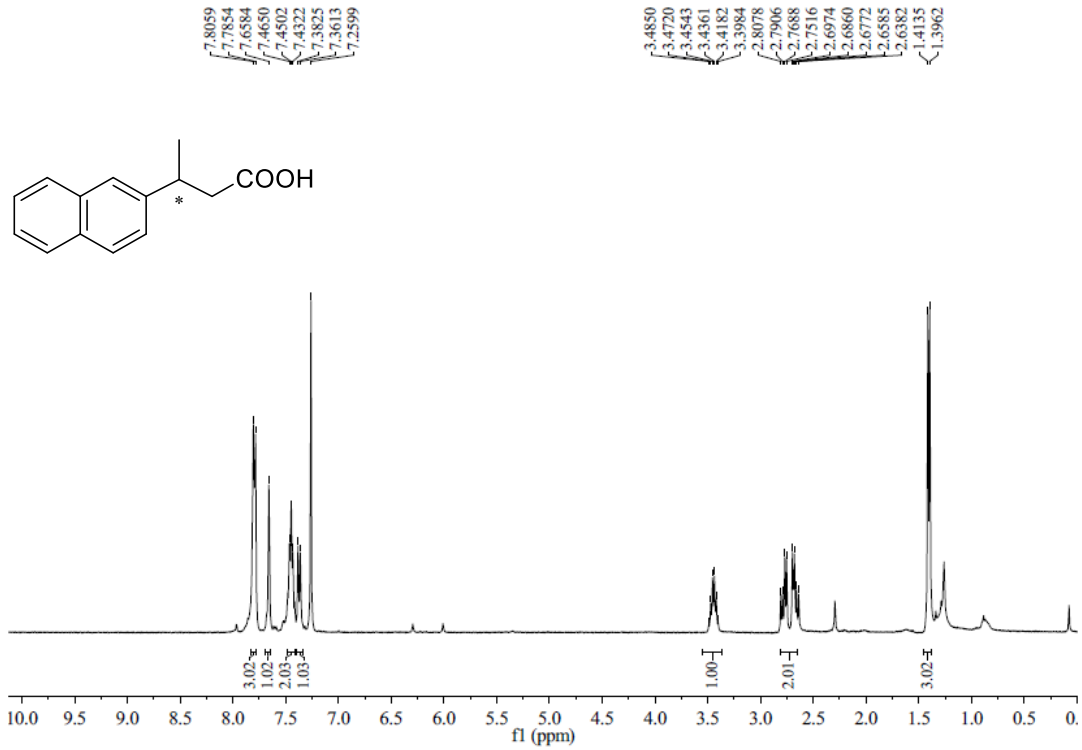
^{1}H NMR (600 MHz, Chloroform-*d*)

(*R*)-3-(*p*-tolyl)butanoic acid (**2l**)



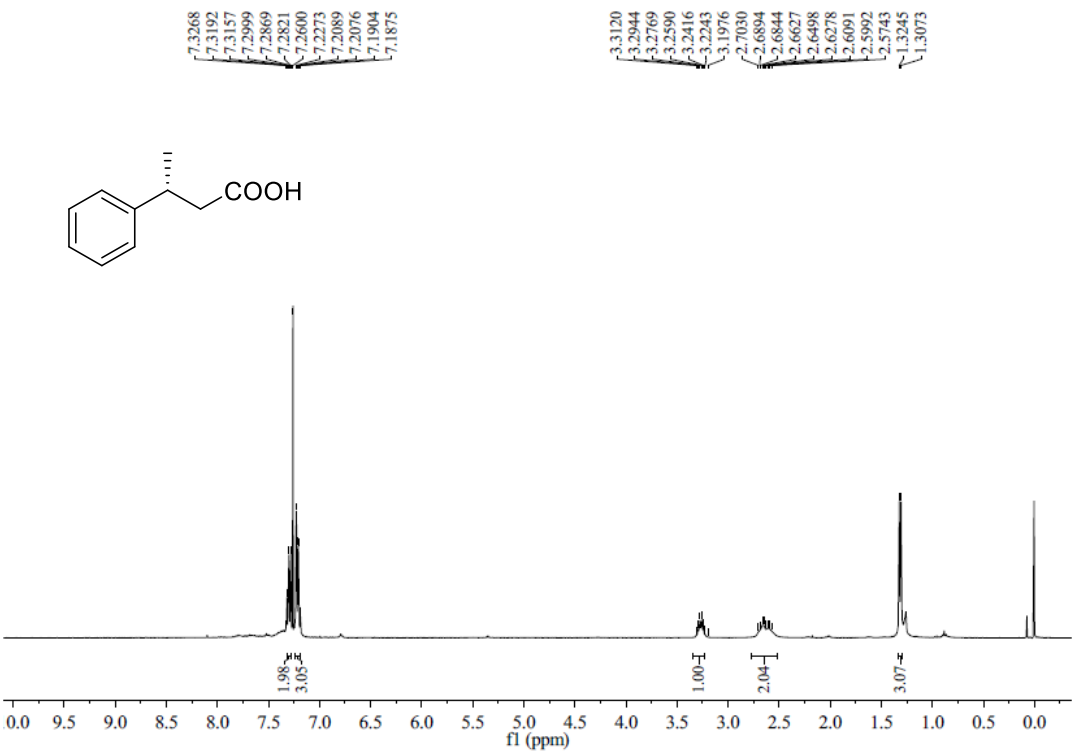
^{1}H NMR (400 MHz, Chloroform-*d*)

(-)-3-(naphthalen-2-yl)butanoic acid (**2m**)



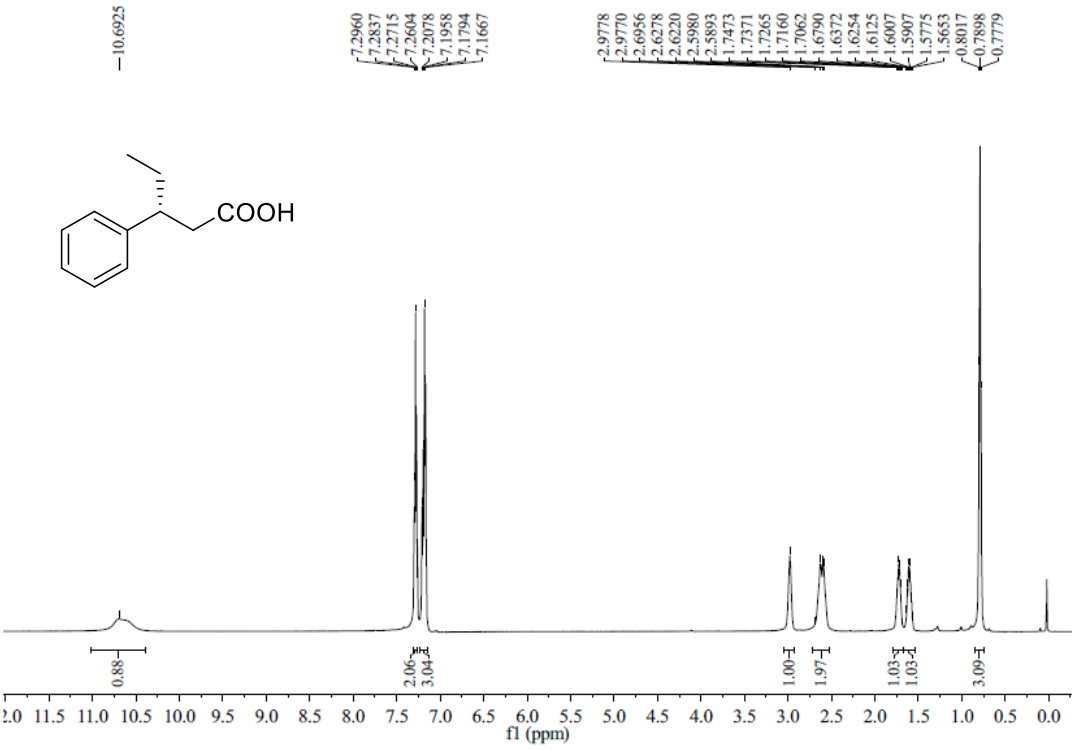
^{1}H NMR (400 MHz, Chloroform-*d*)

(*R*)-3-phenylbutanoic acid (**2n**)



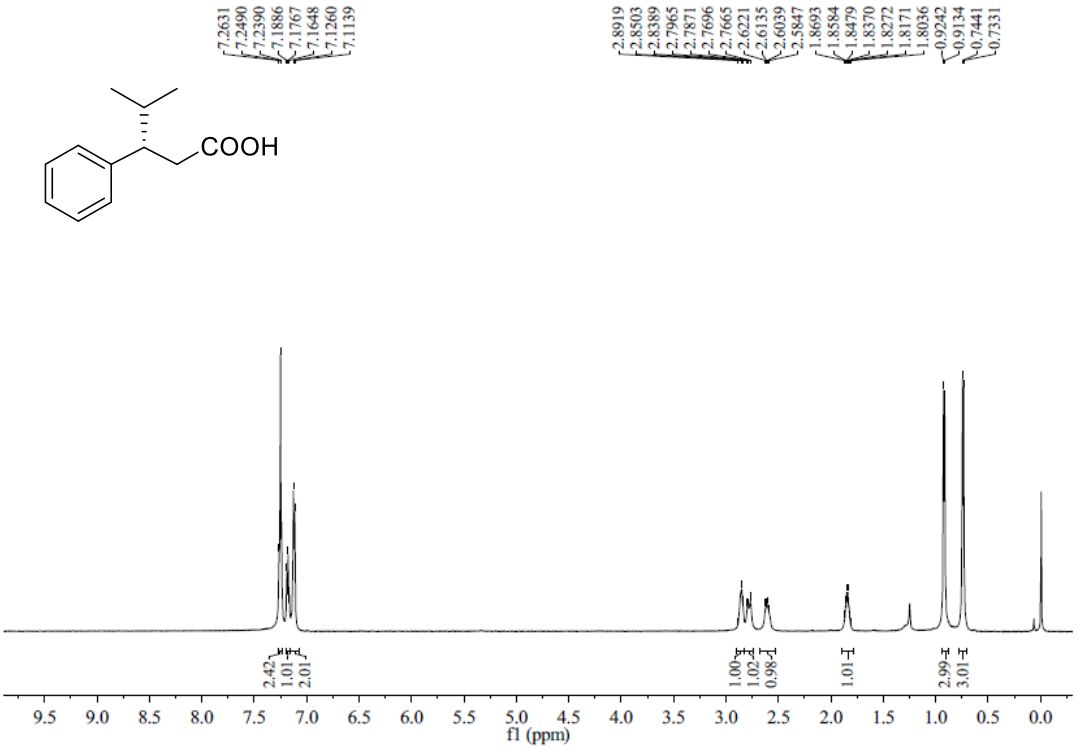
^{1}H NMR (400 MHz, Chloroform-*d*)

(*R*)-3-phenylpentanoic acid (**2o**)



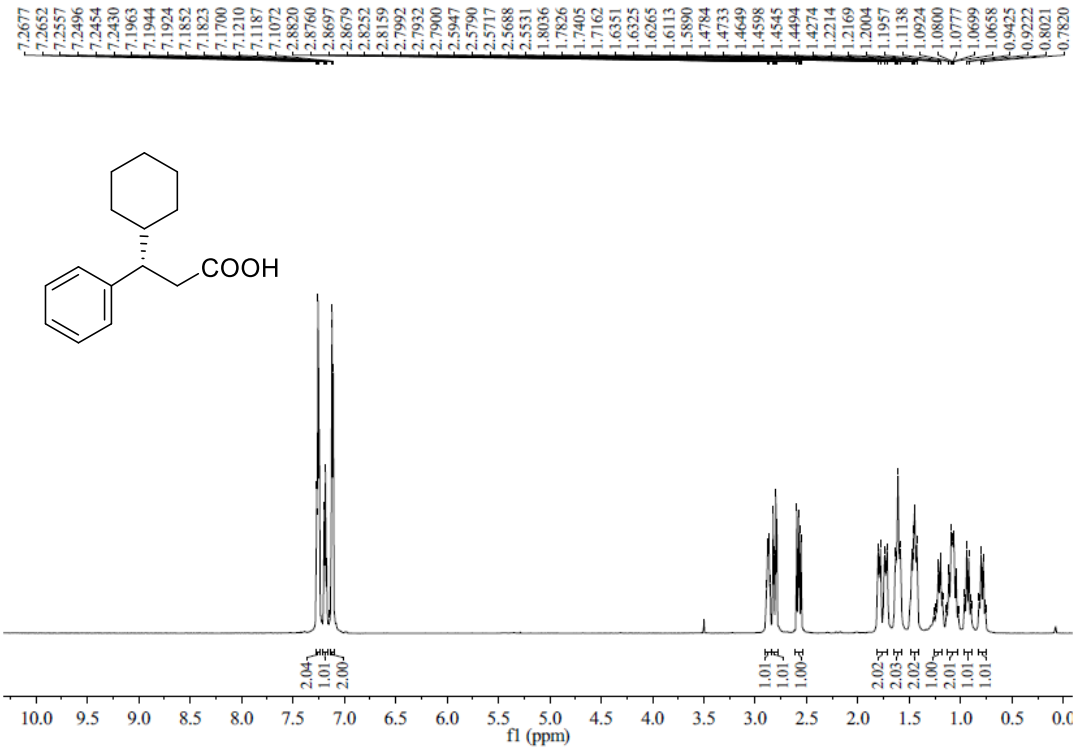
^{1}H NMR (600 MHz, Chloroform-*d*)

(*S*)-4-methyl-3-phenylpentanoic acid (**2p**)



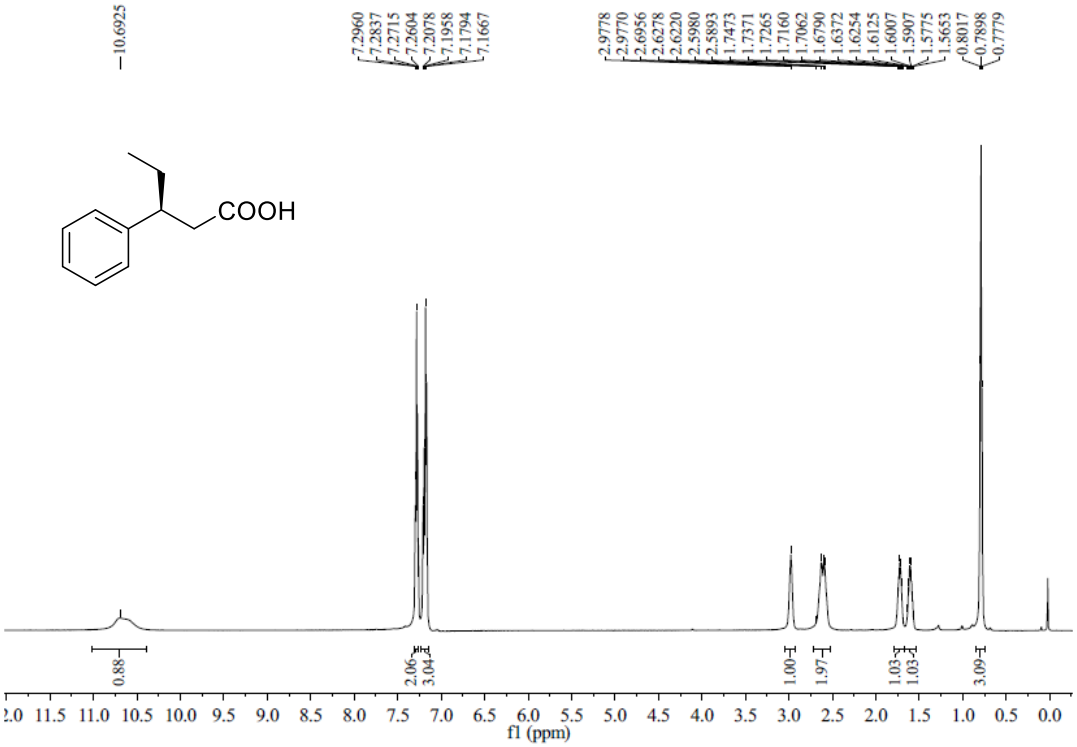
^{1}H NMR (600 MHz, Chloroform-*d*)

(*S*)-3-cyclohexyl-3-phenylpropanoic acid (**2q**)



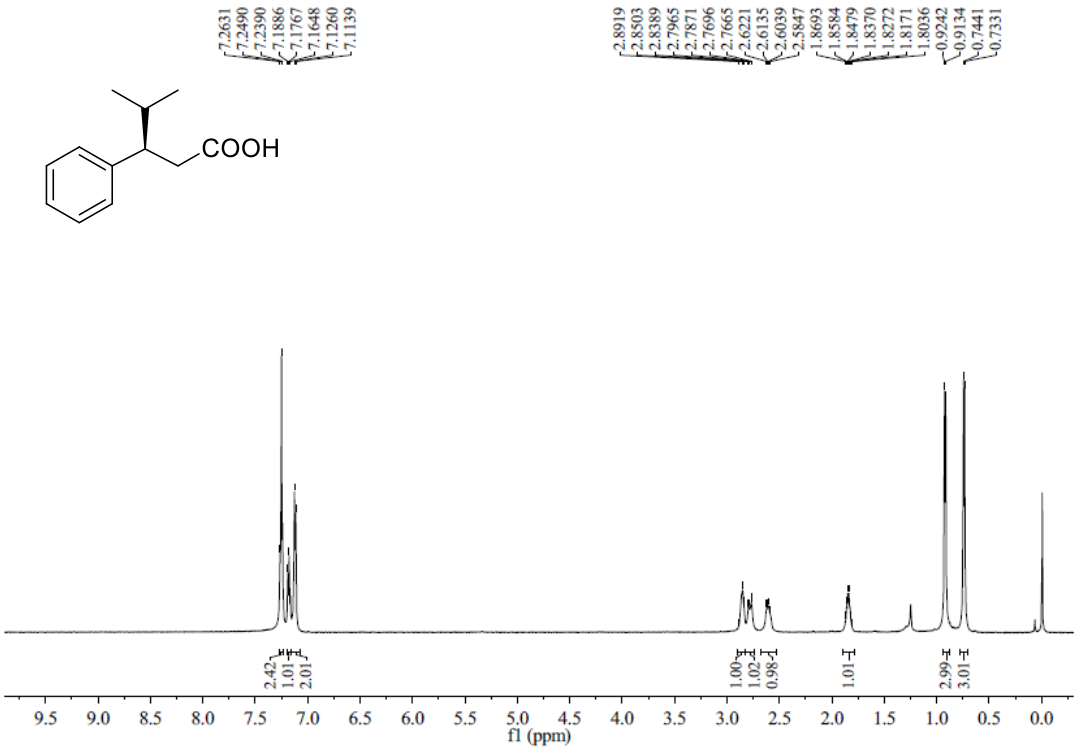
^{1}H NMR (600 MHz, Chloroform-*d*)

(*S*)-3-phenylpentanoic acid (**2r**)



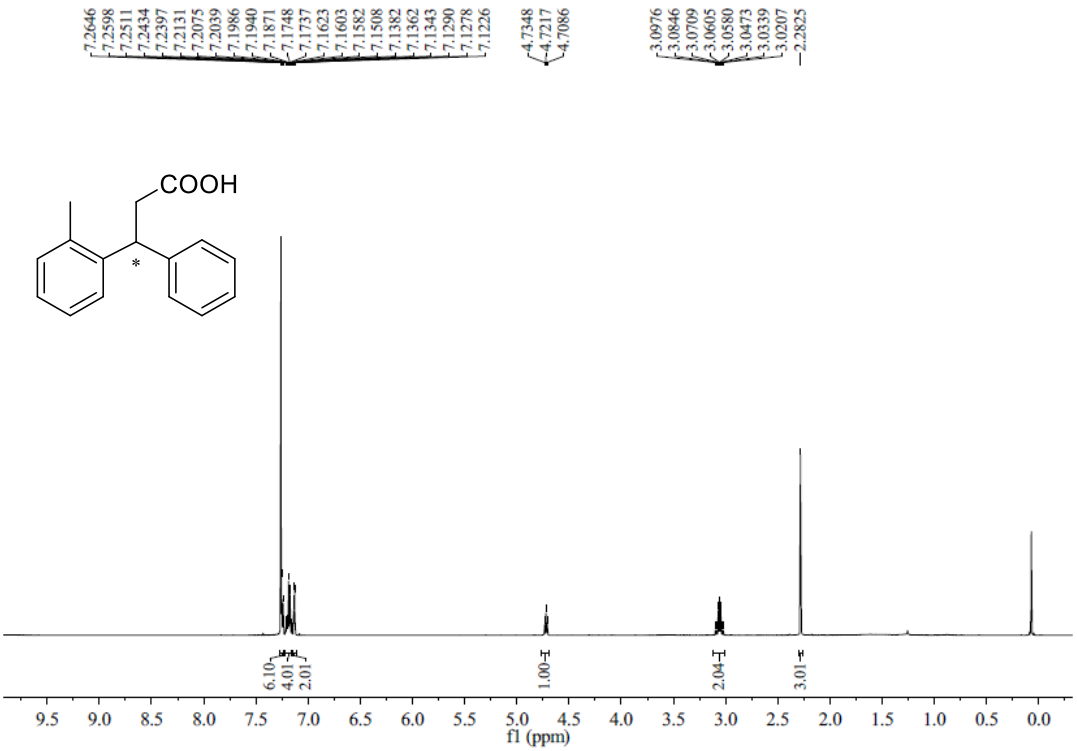
^{1}H NMR (600 MHz, Chloroform-*d*)

(*R*)-4-methyl-3-phenylpentanoic acid (**2s**)



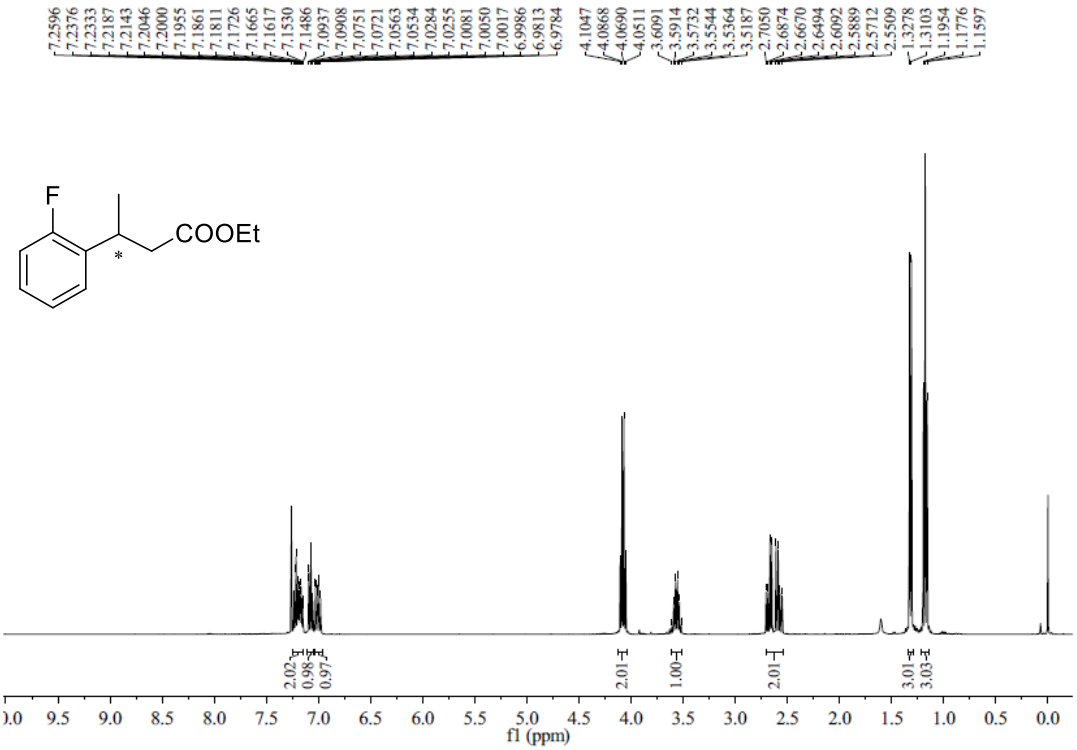
^{1}H NMR (600 MHz, Chloroform-*d*)

(-)-3-phenyl-3-(*o*-tolyl)propanoic acid (**2t**)



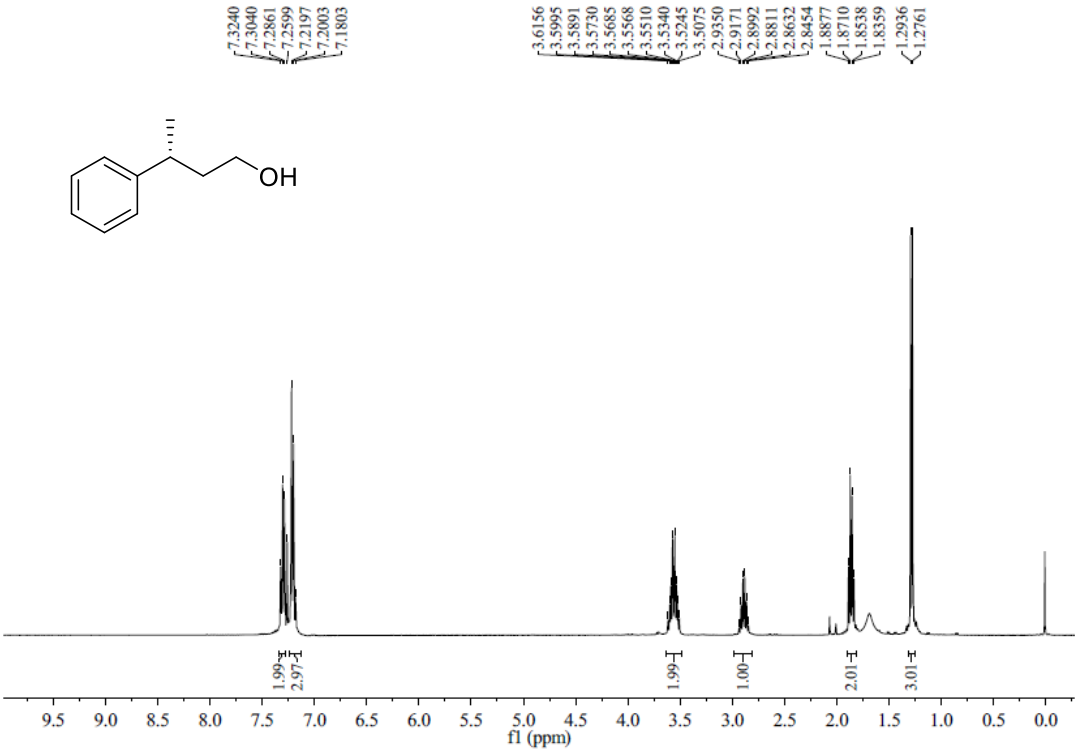
^{1}H NMR (600 MHz, Chloroform-*d*)

(-)-ethyl 3-(2-fluorophenyl)butanoate (**2u**)



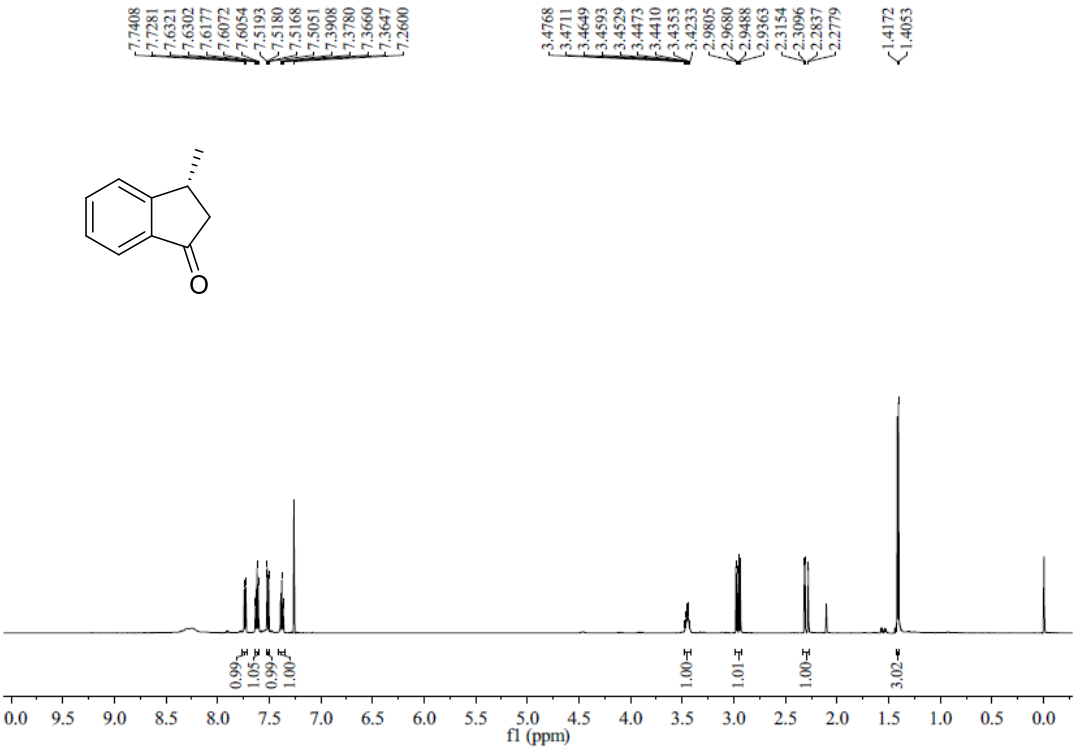
^{1}H NMR (400 MHz, Chloroform-*d*)

(*R*)-3-phenylbutan-1-ol (**3**)



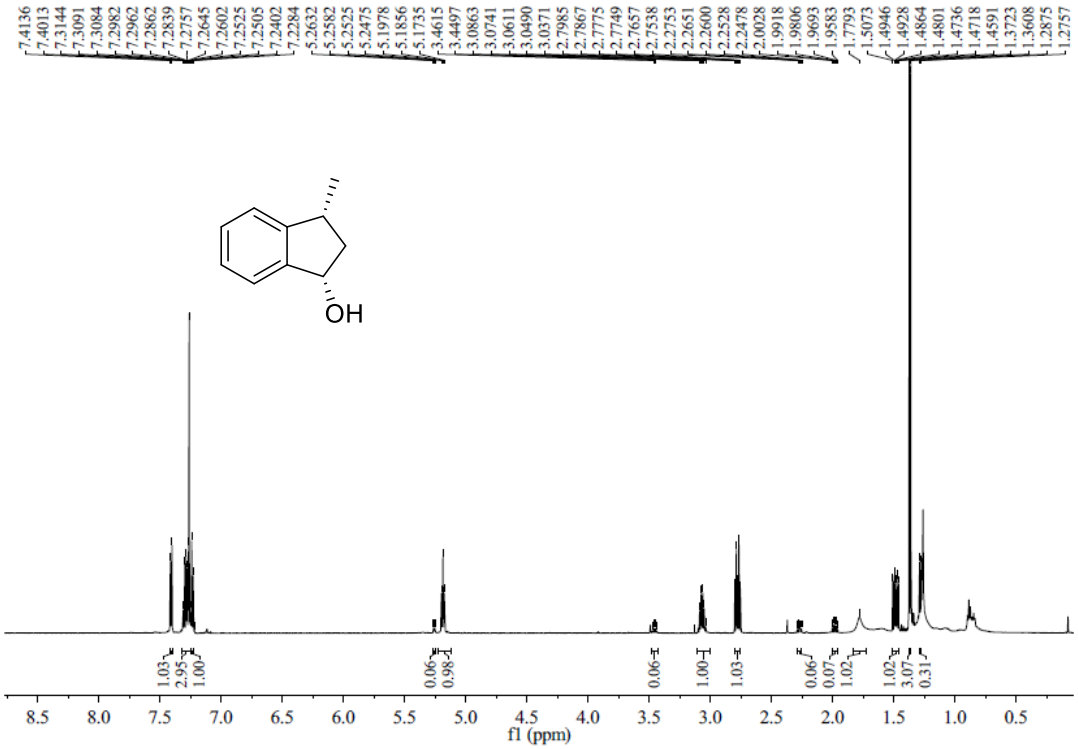
^{1}H NMR (400 MHz, Chloroform-*d*)

(*R*)-3-methyl-2,3-dihydro-1*H*-inden-1-one (**4**)



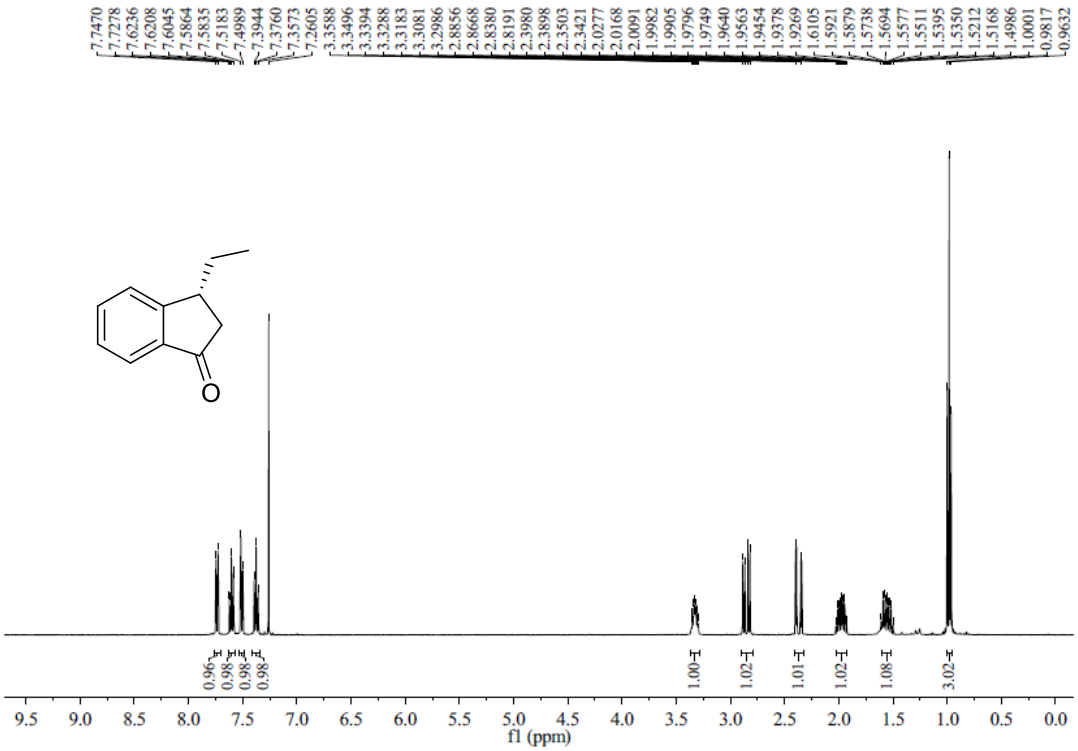
^{1}H NMR (600 MHz, Chloroform-*d*)

(1*S*,3*R*)-3-methyl-2,3-dihydro-1*H*-inden-1-ol (**5**)



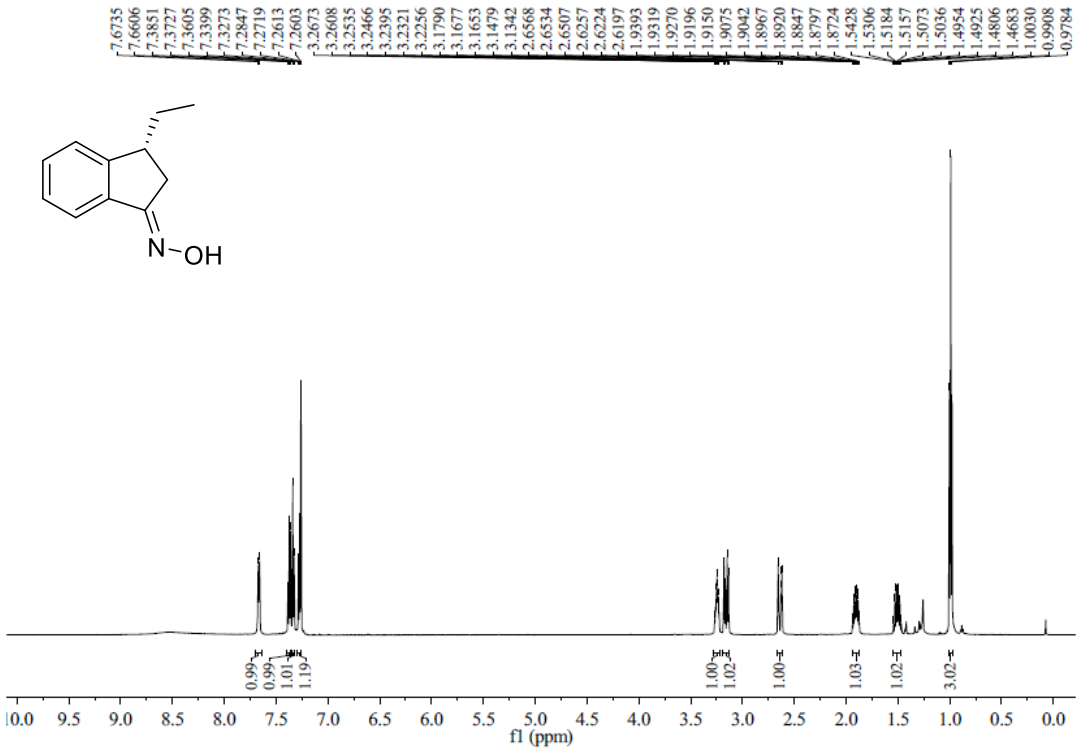
^{1}H NMR (600 MHz, Chloroform-*d*)

(*R*)-3-ethyl-2,3-dihydro-1*H*-inden-1-one (**6**)



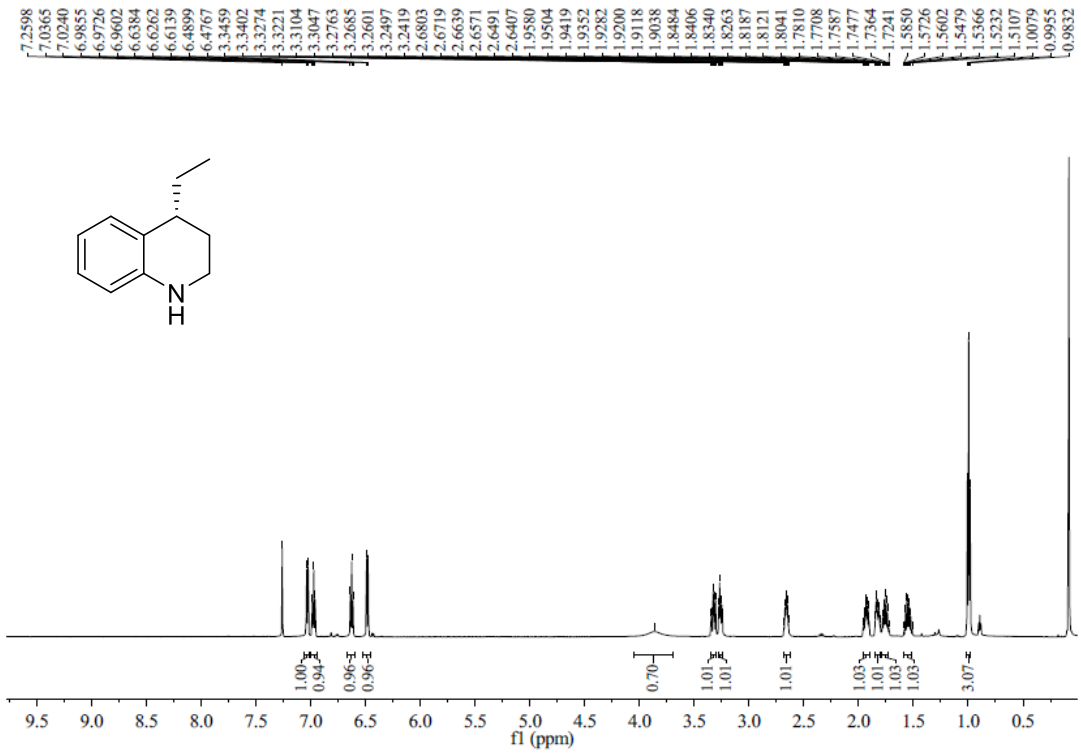
^{1}H NMR (400 MHz, Chloroform-*d*)

(*R*)-3-ethyl-2,3-dihydro-1*H*-inden-1-one oxime (**7**)



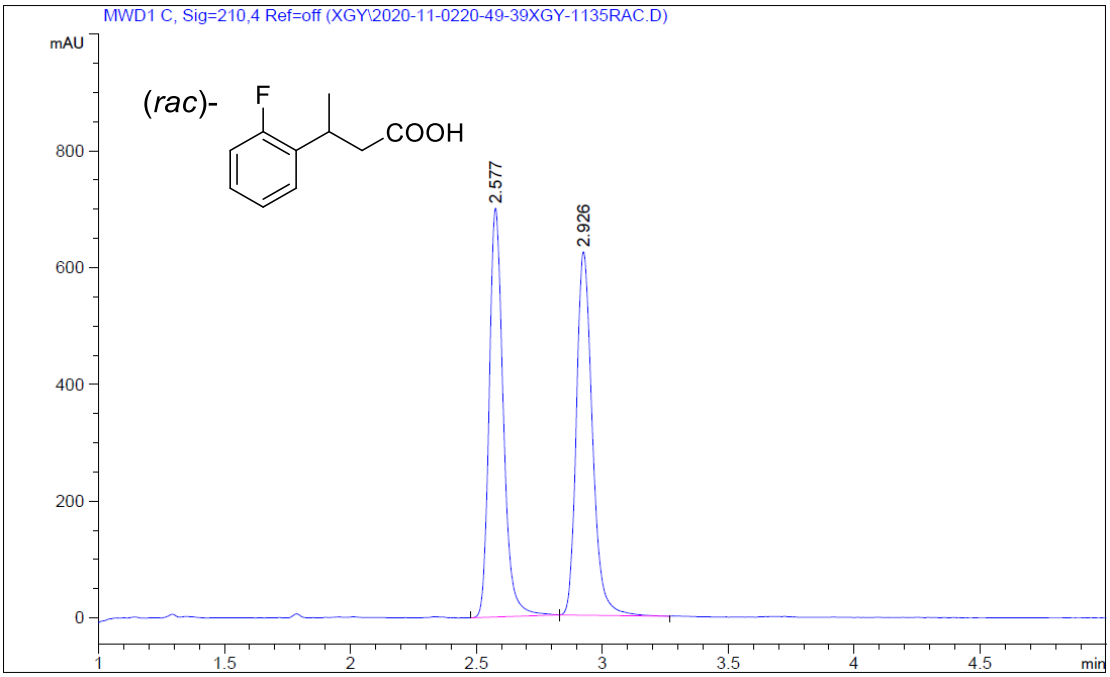
^{1}H NMR (400 MHz, Chloroform-*d*)

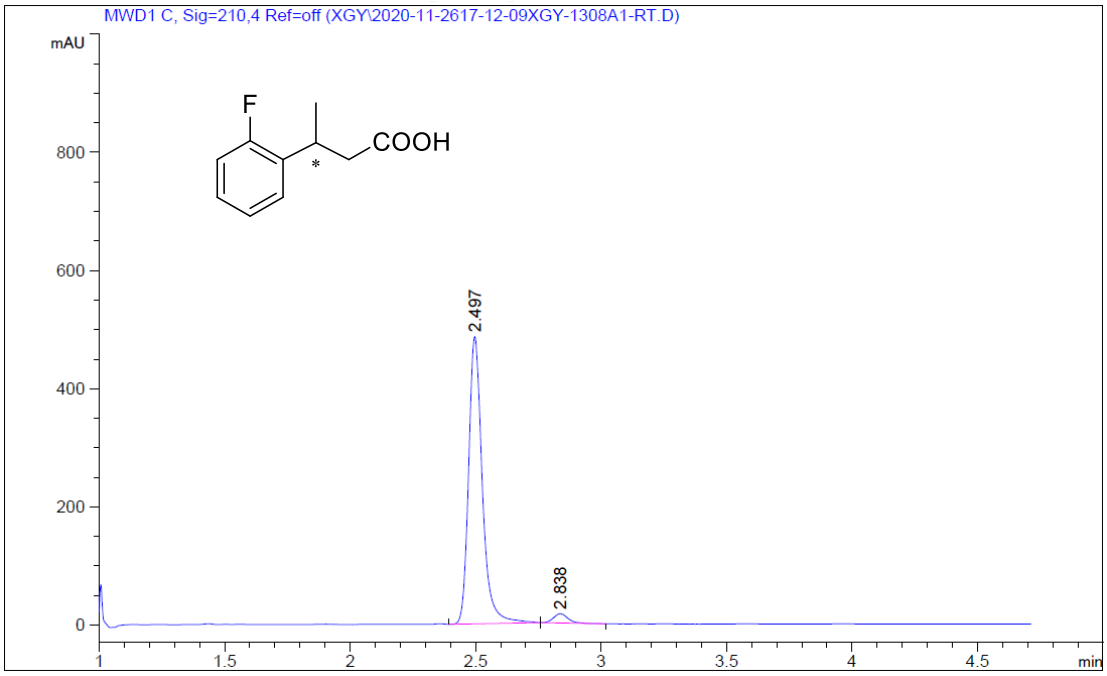
(*R*)-4-ethyl-1,2,3,4-tetrahydroquinoline (**8**)



^{1}H NMR (600 MHz, Chloroform-*d*)

(+)-3-(2-fluorophenyl)butanoic acid (**2a**)

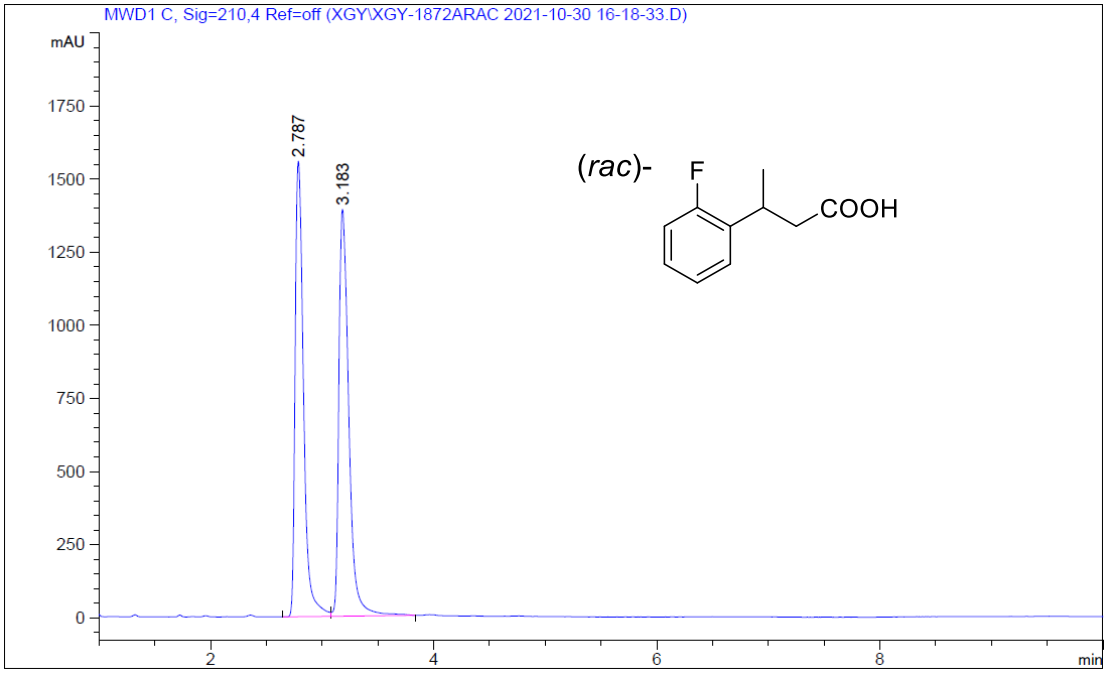


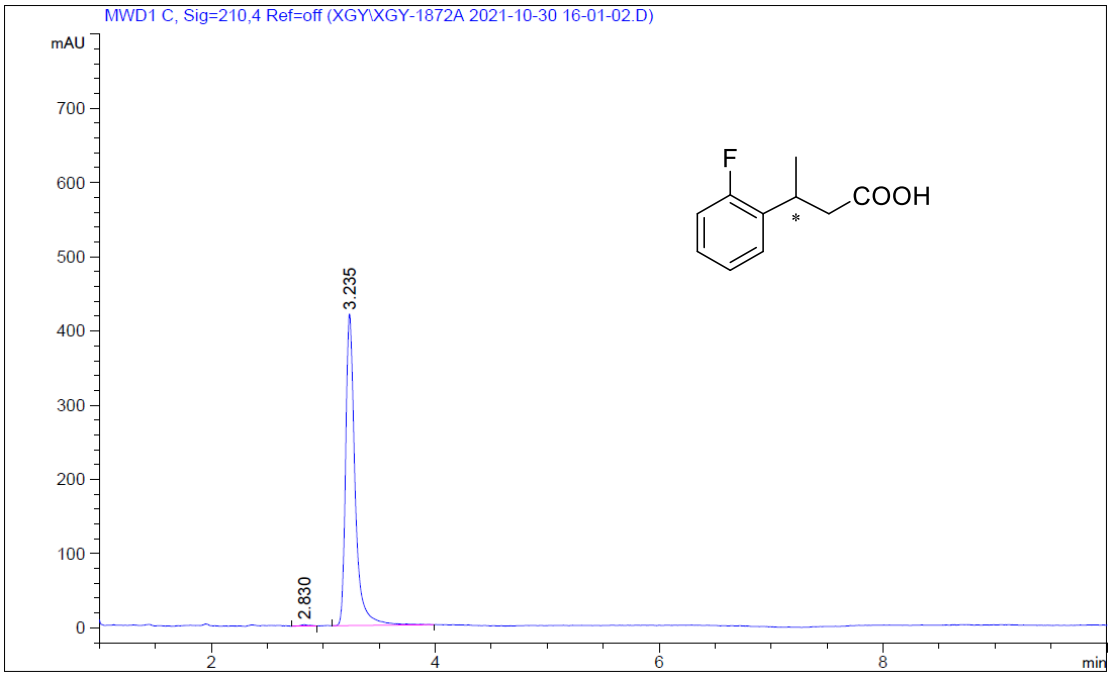


Signal 1: MWD1 C, Sig=210,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	2.497	BB	0.0578	1845.43042	486.19901	96.6801
2	2.838	BB	0.0620	63.36998	15.57936	3.3199

(-)-3-(2-fluorophenyl)butanoic acid (**2b**)

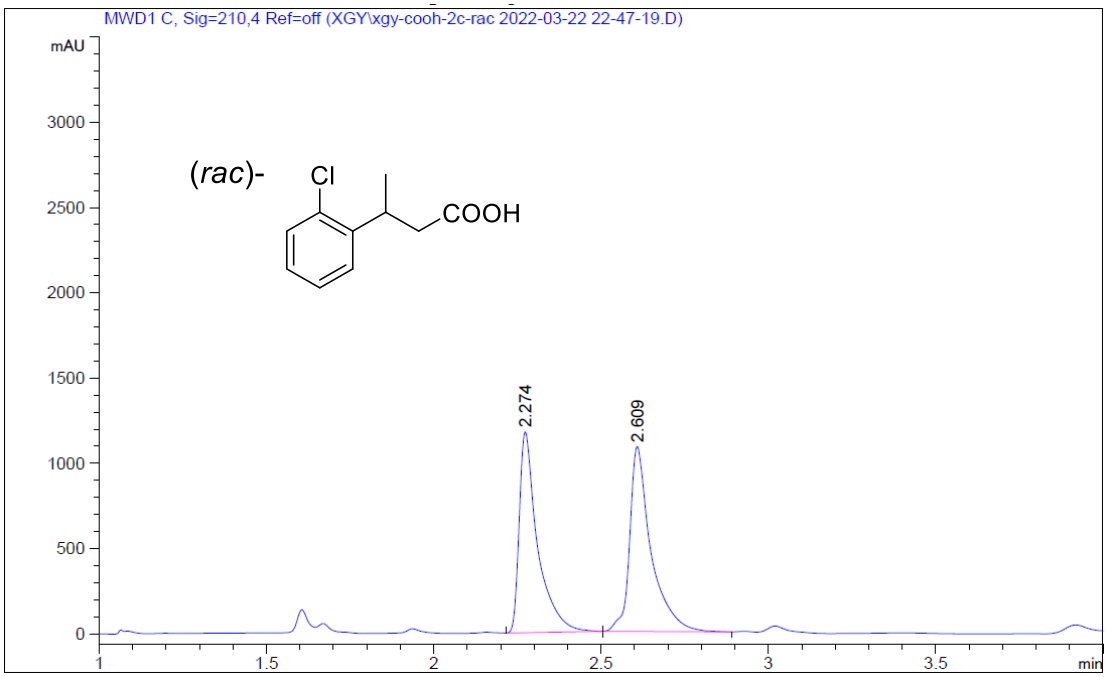


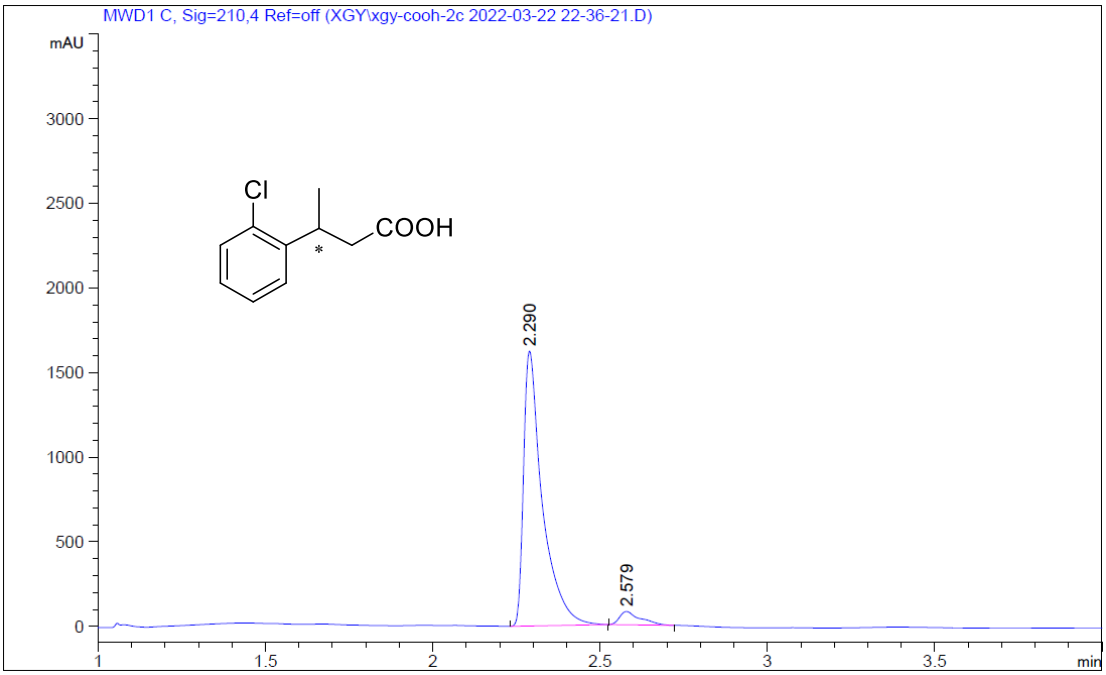


Signal 1: MWD1 C, Sig=210,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	2.830	BB	0.0738	9.57644	1.86971	0.3867
2	3.235	BB	0.0887	2466.88428	419.02966	99.6133

(-)3-(2-chlorophenyl)butanoic acid (**2c**)

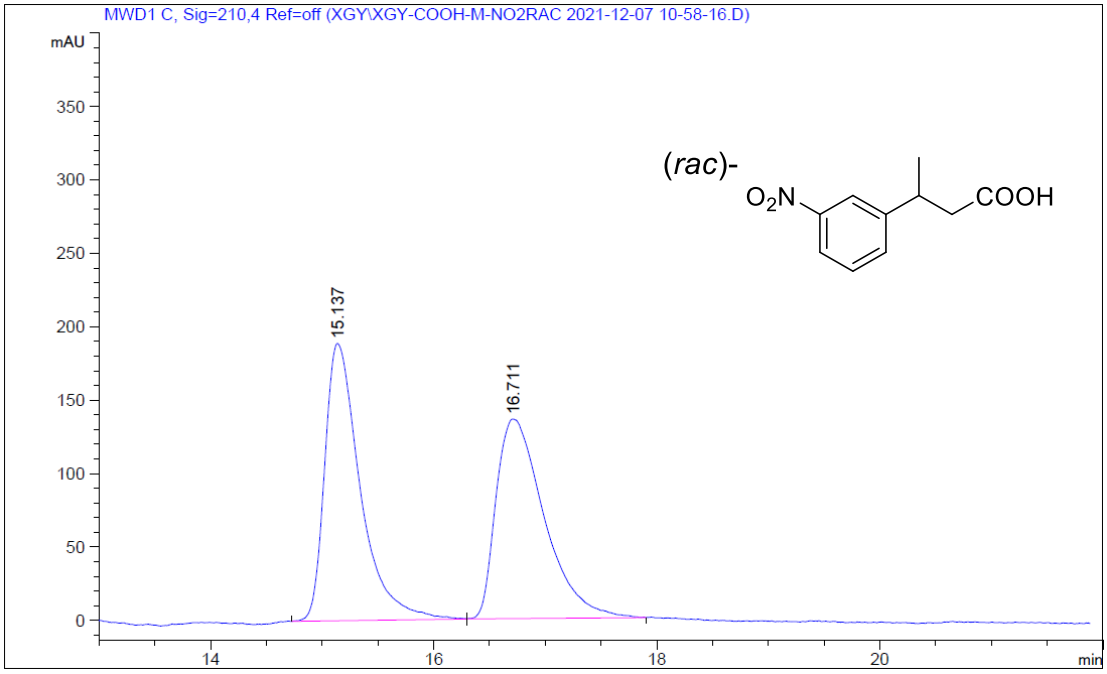


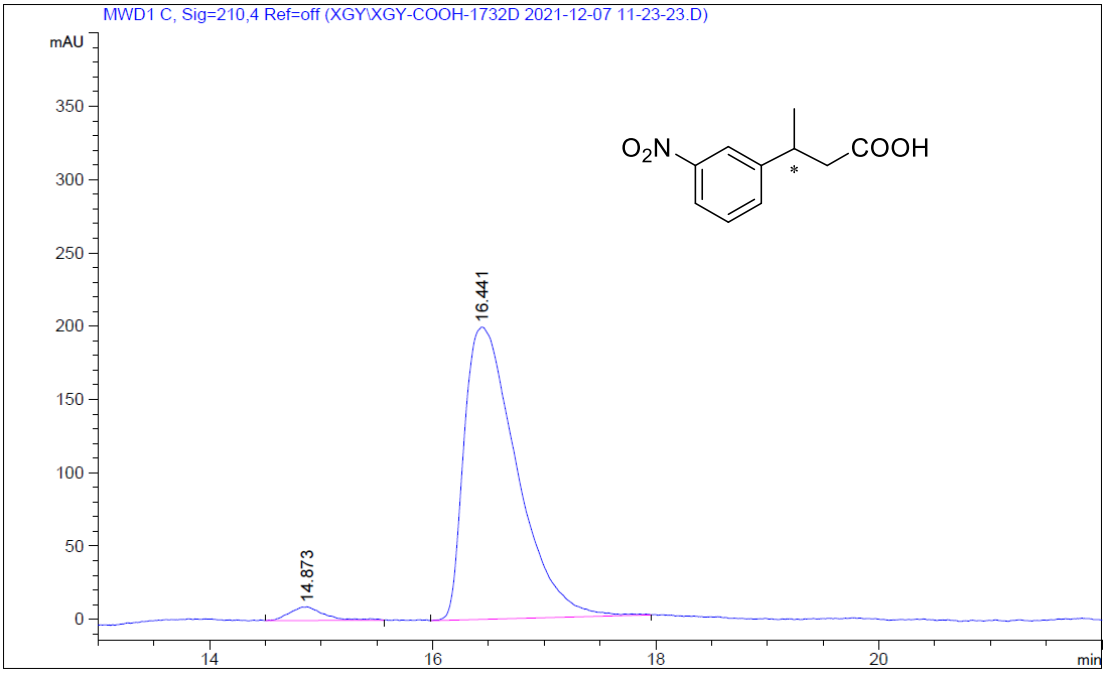


Signal 1: MWD1 C, Sig=210,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	2.290	BB	0.0566	6347.92188	1625.02527	94.9215
2	2.579	BB	0.0607	339.62582	78.87069	5.0785

(-)-3-(3-nitrophenyl)butanoic acid (**2d**)

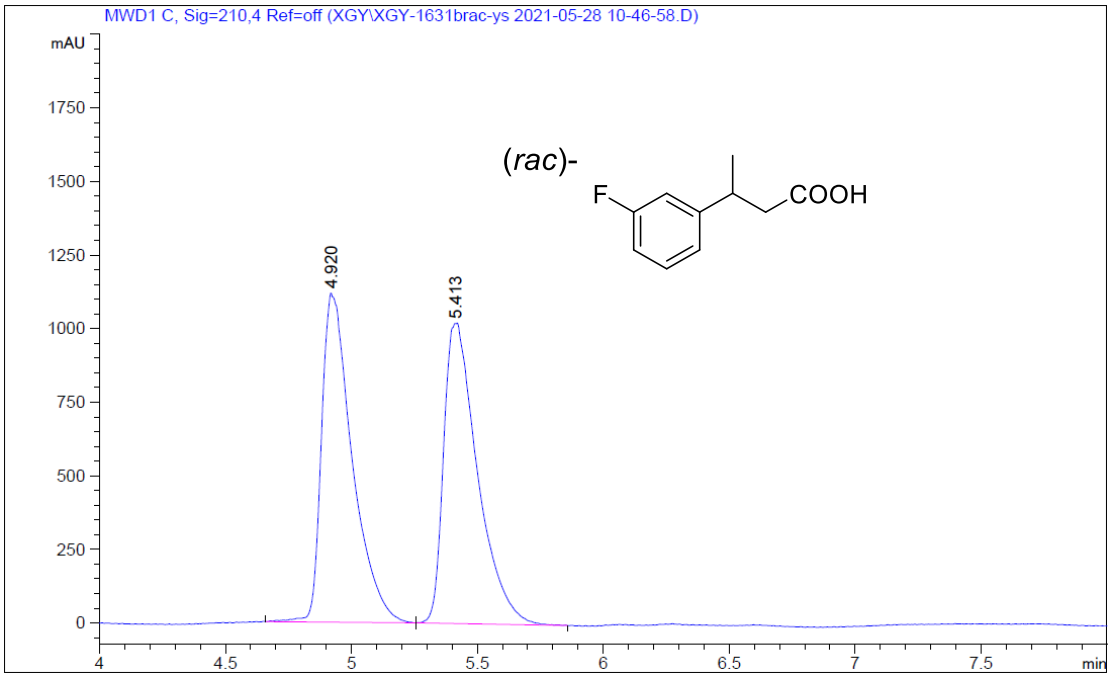


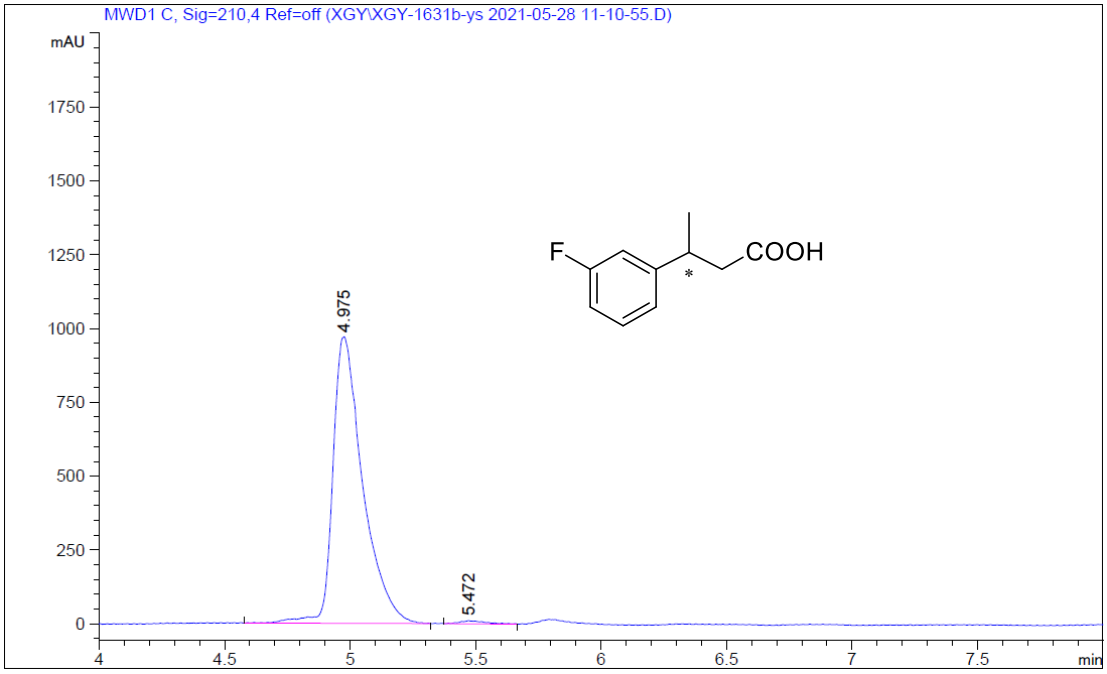


Signal 1: MWD1 C, Sig=210,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.873	VV R	0.2560	201.89615	9.34520	3.0055
2	16.441	BV R	0.3936	6515.66162	199.44106	96.9945

(-)-3-(3-fluorophenyl)butanoic acid (**2e**)

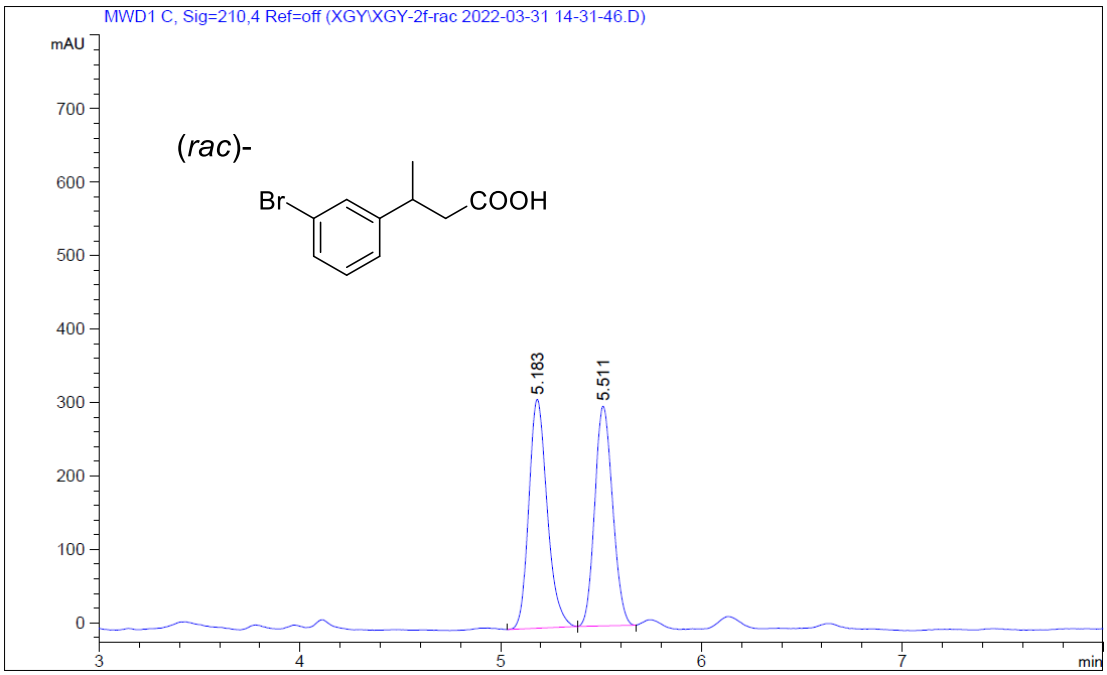


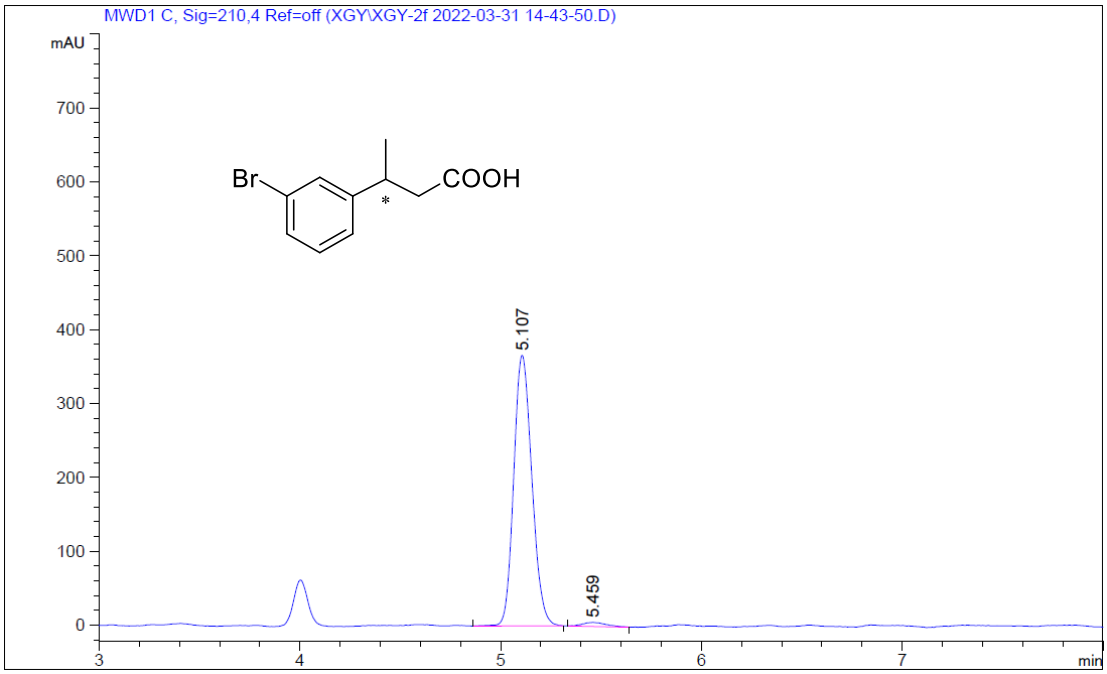


Signal 1: MWD1 C, Sig=210,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.975	VB R	0.1156	8038.21484	970.13336	99.0175
2	5.472	BB	0.0872	79.75824	11.05405	0.9825

(-)-3-(3-bromophenyl)butanoic acid (**2f**)

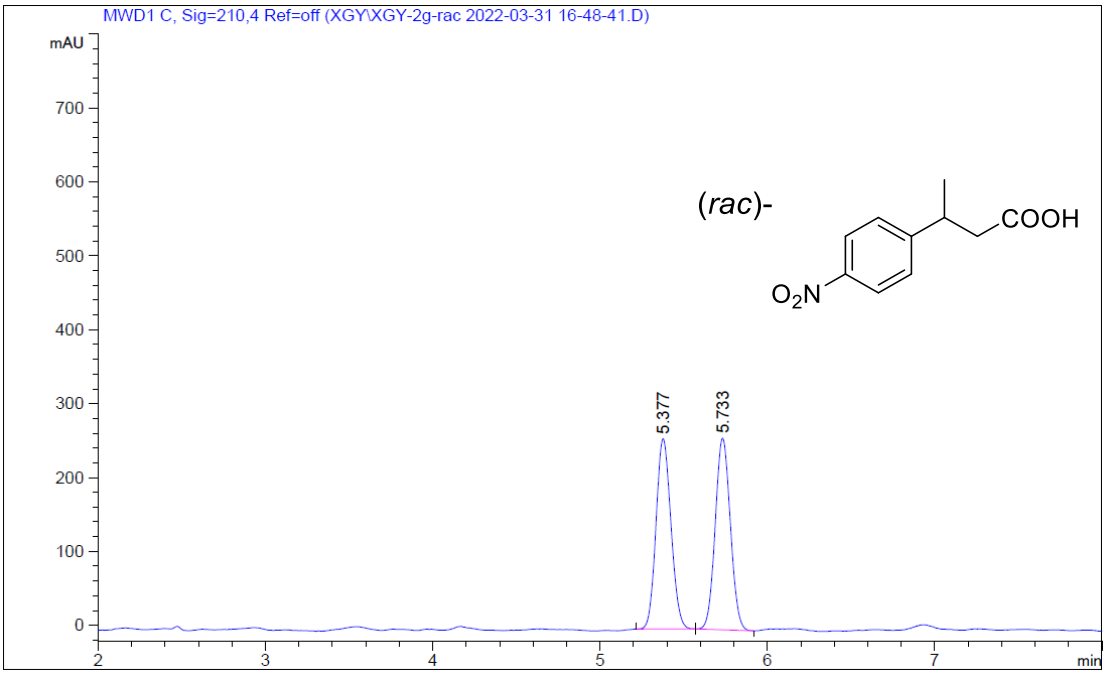


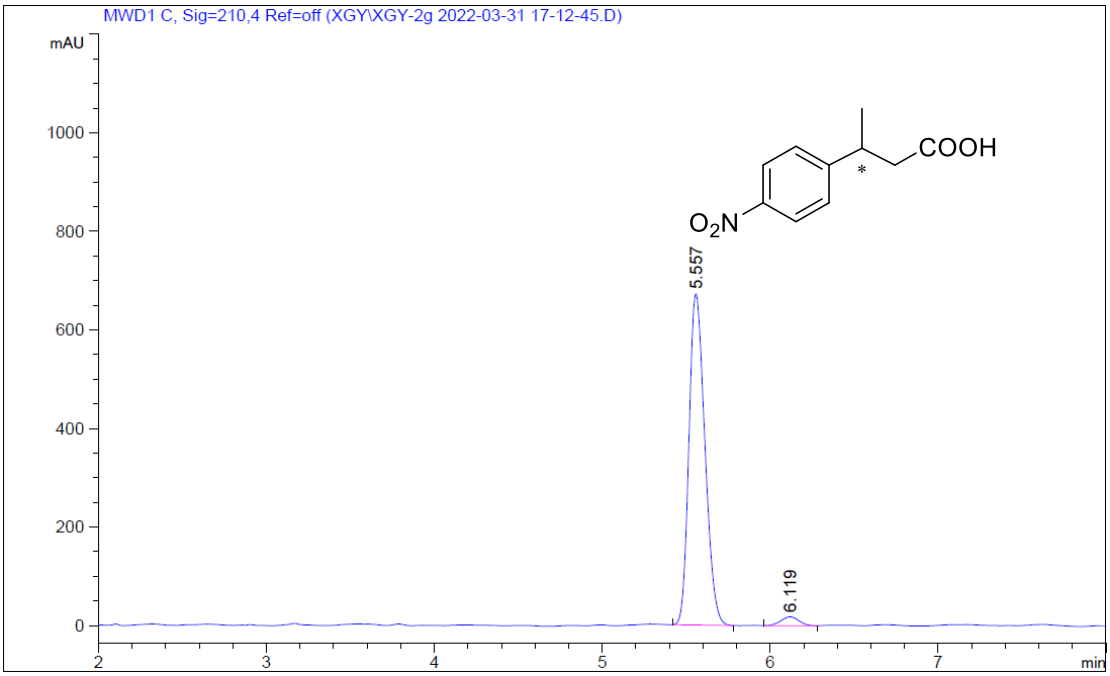


Signal 1: MWD1 C, Sig=210,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.107	VV R	0.0969	2303.96924	366.36096	98.0535
2	5.459	VV R	0.0976	45.73698	5.52417	1.9465

(-)-3-(4-nitrophenyl)butanoic acid (**2g**)

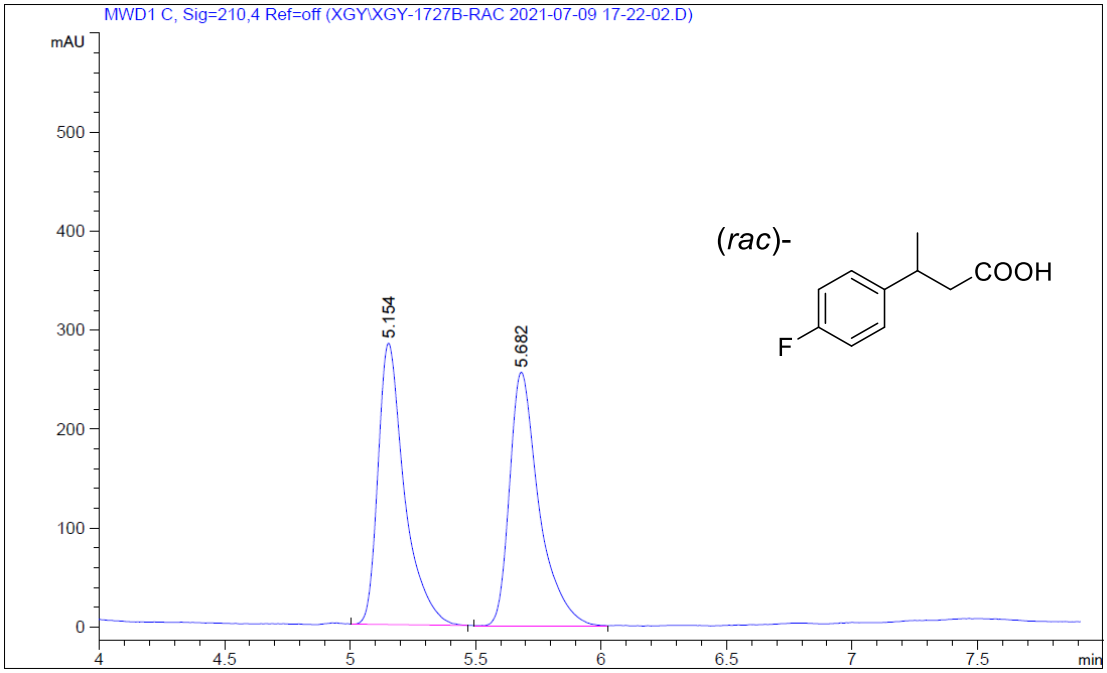


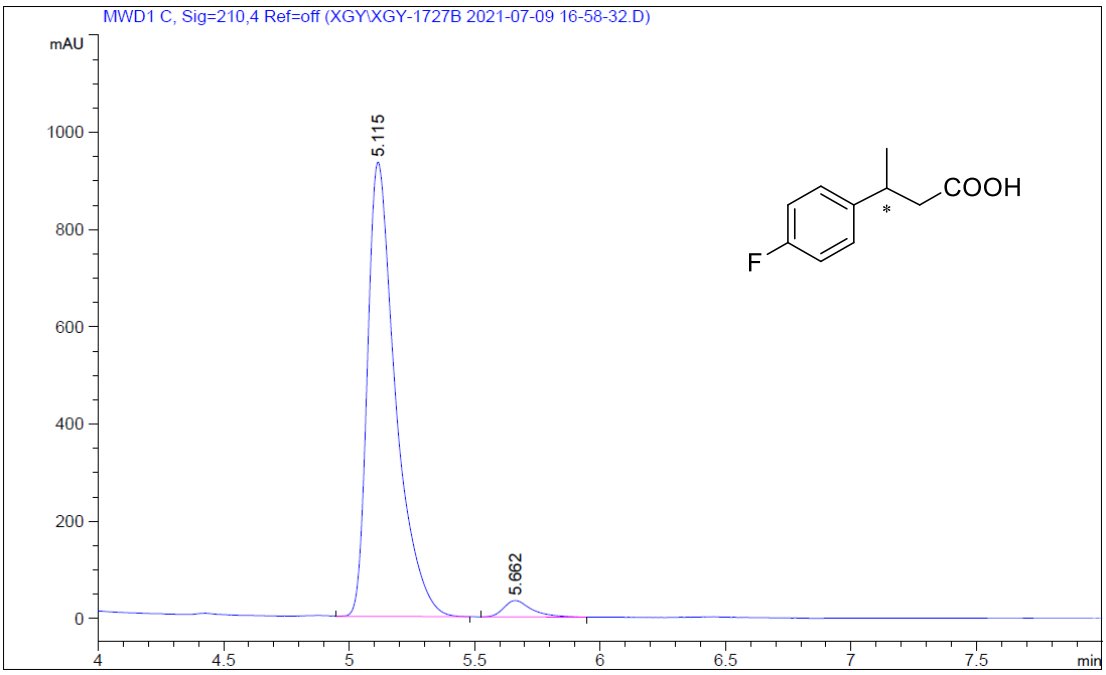


Signal 1: MWD1 C, Sig=210,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.557	BB	0.1034	4430.71729	672.00006	97.0722
2	6.119	BB	0.0993	133.63306	18.06862	2.9278

(-)-3-(4-fluorophenyl)butanoic acid (**2h**)

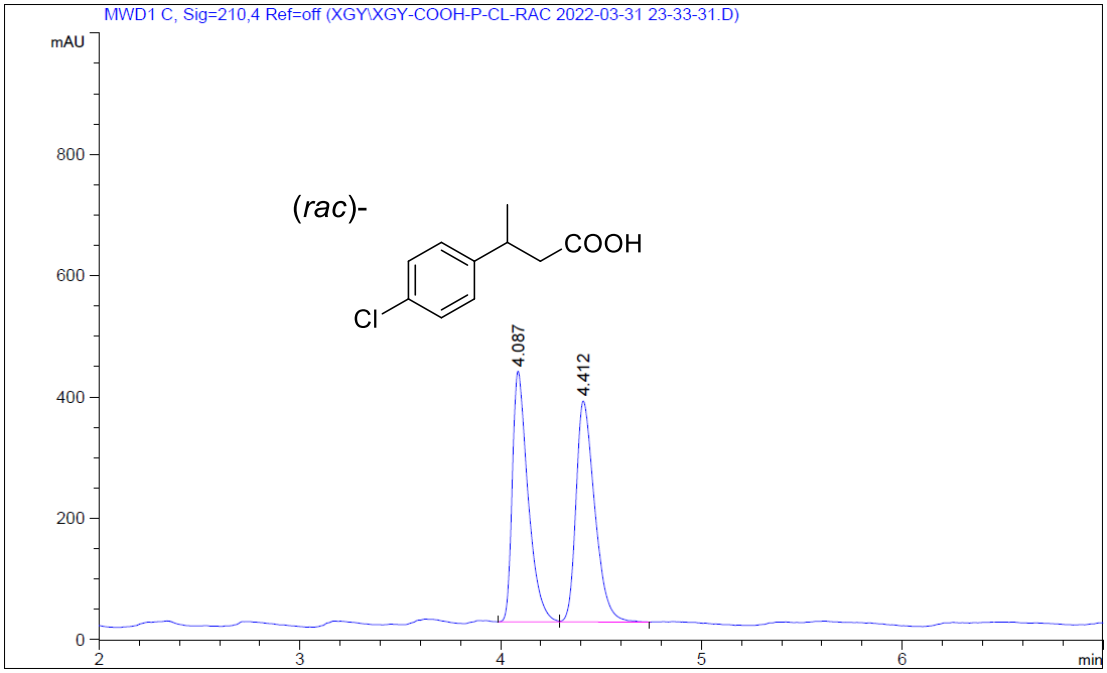


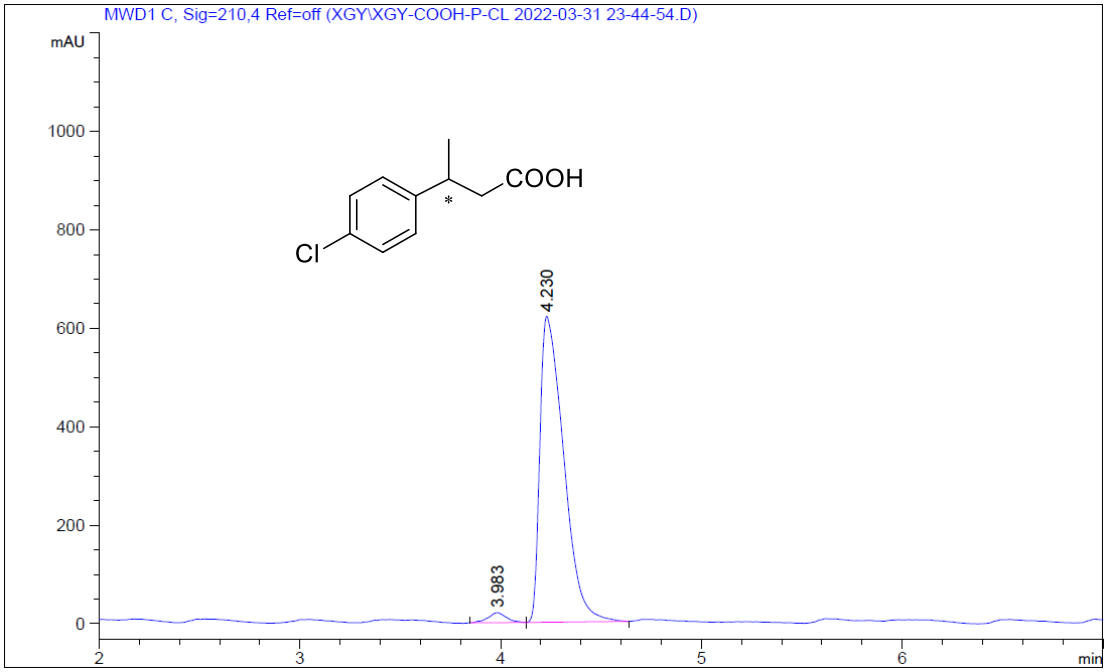


Signal 1: MWD1 C, Sig=210,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.115	BV R	0.1162	7268.42871	934.46948	96.5317
2	5.662	BV R	0.1119	261.14706	33.89094	3.4683

(-)-3-(4-chlorophenyl)butanoic acid (**2i**)

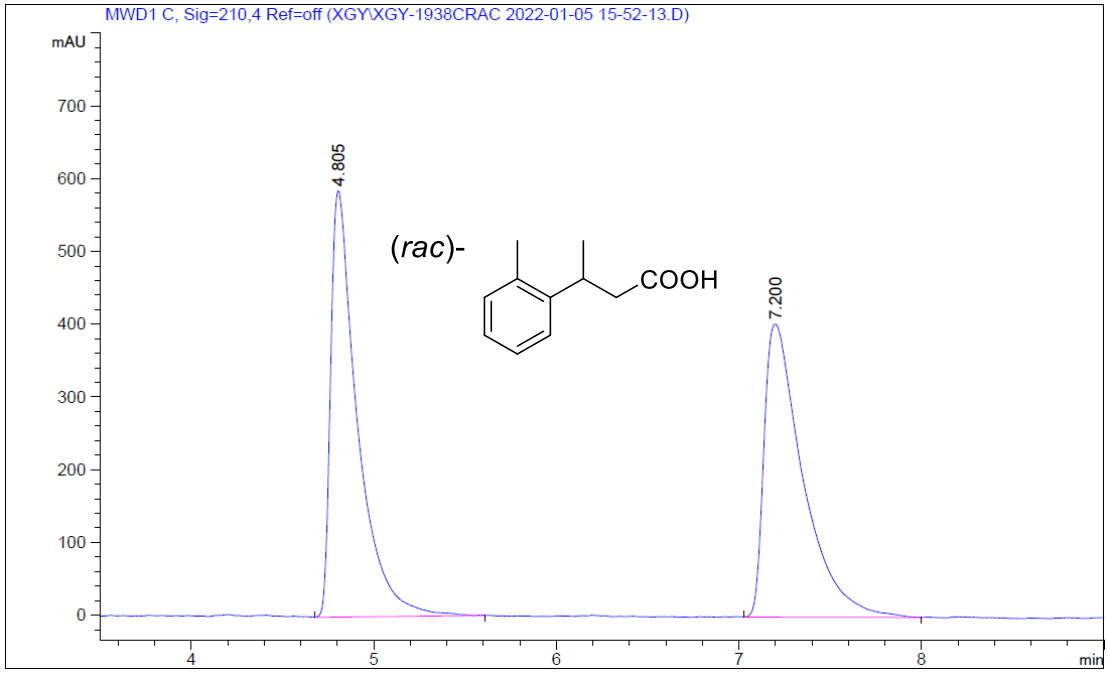


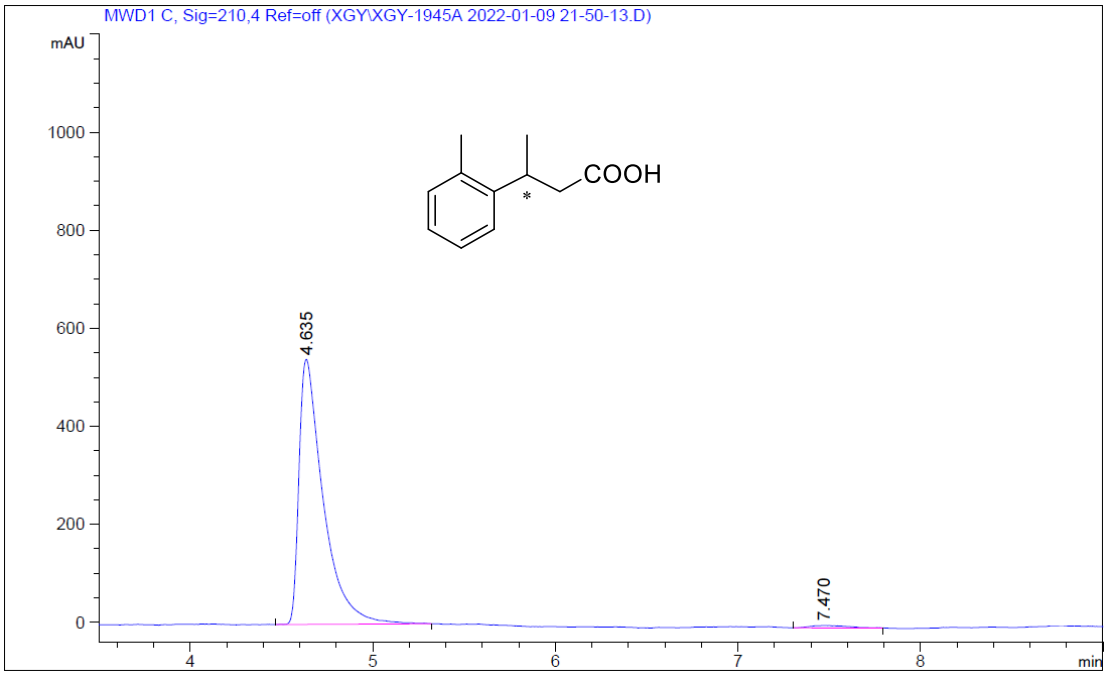


Signal 1: MWD1 C, Sig=210,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.983	BB	0.0882	126.88873	20.37037	2.4259
2	4.230	BB	0.1203	5103.78125	621.33539	97.5741

(-)-3-(*o*-tolyl)butanoic acid (**2j**)

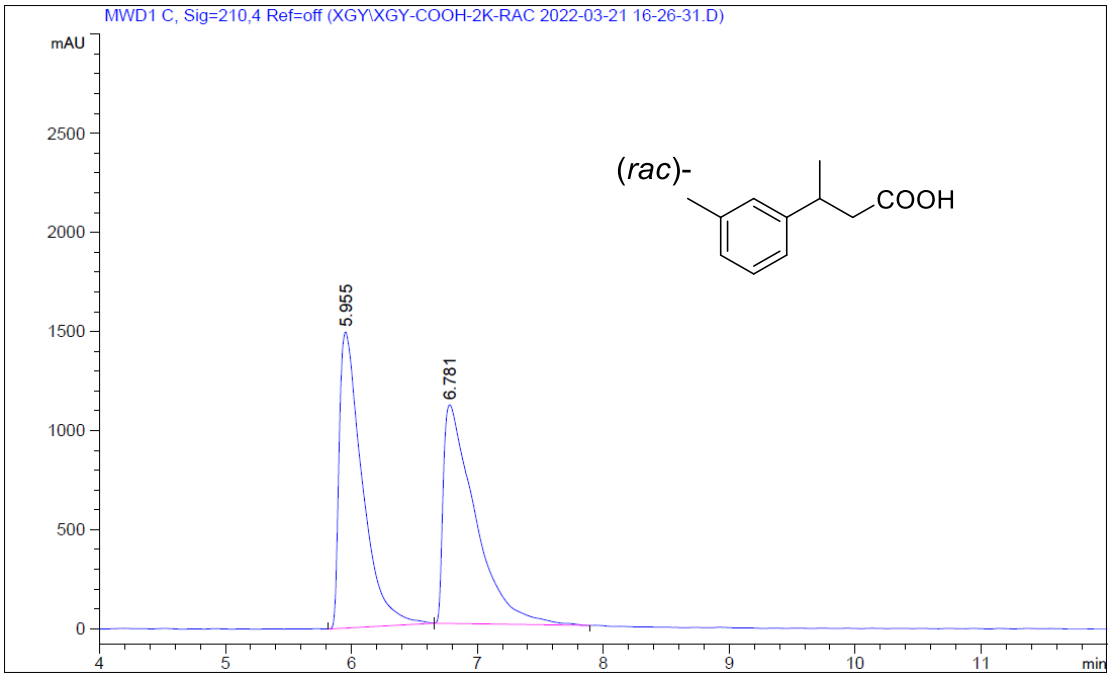


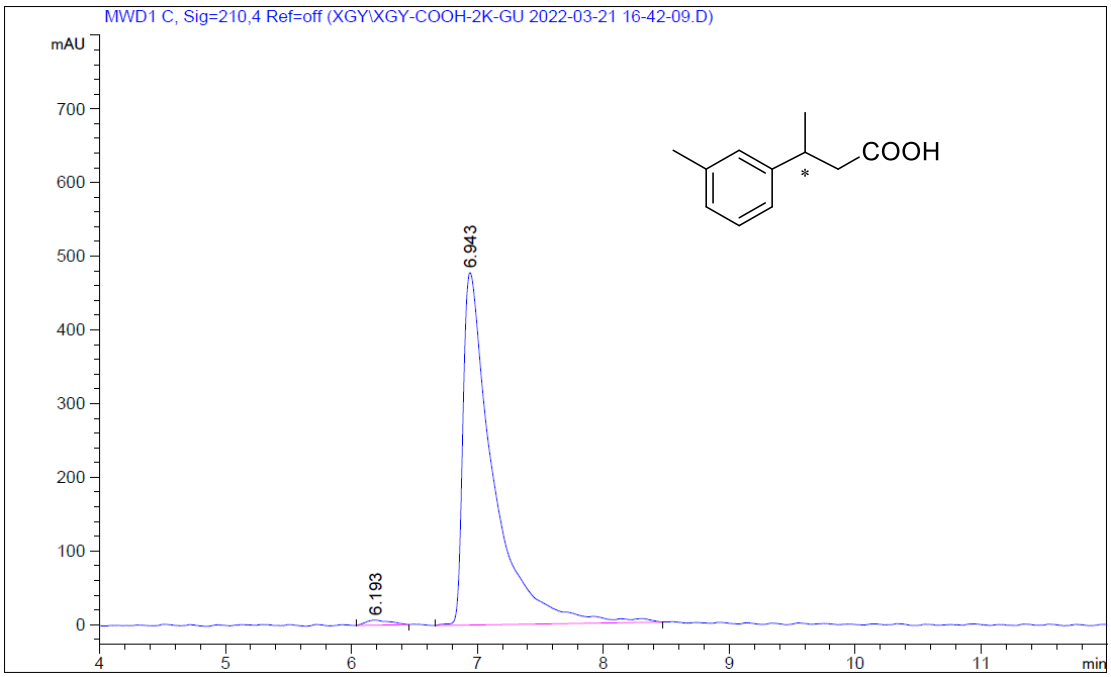


Signal 1: MWD1 C, Sig=210,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.635	VB R	0.1327	4988.40332	540.54468	98.5776
2	7.470	BB	0.1683	71.98016	5.14191	1.4224

(-)-3-(*m*-tolyl)butanoic acid (**2k**)

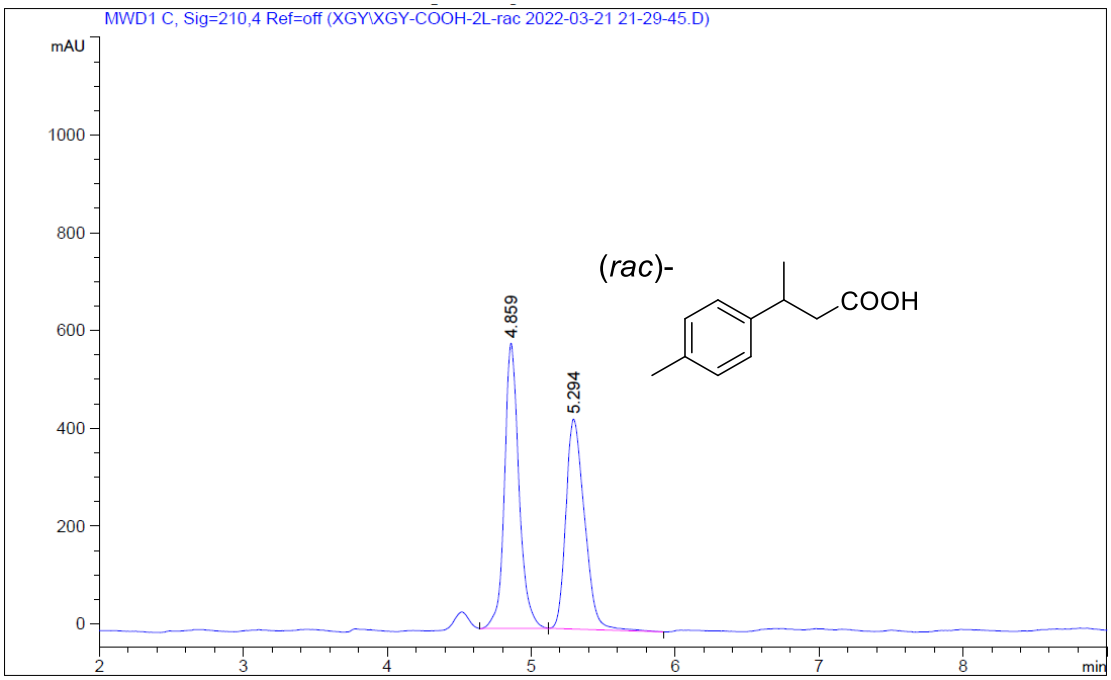


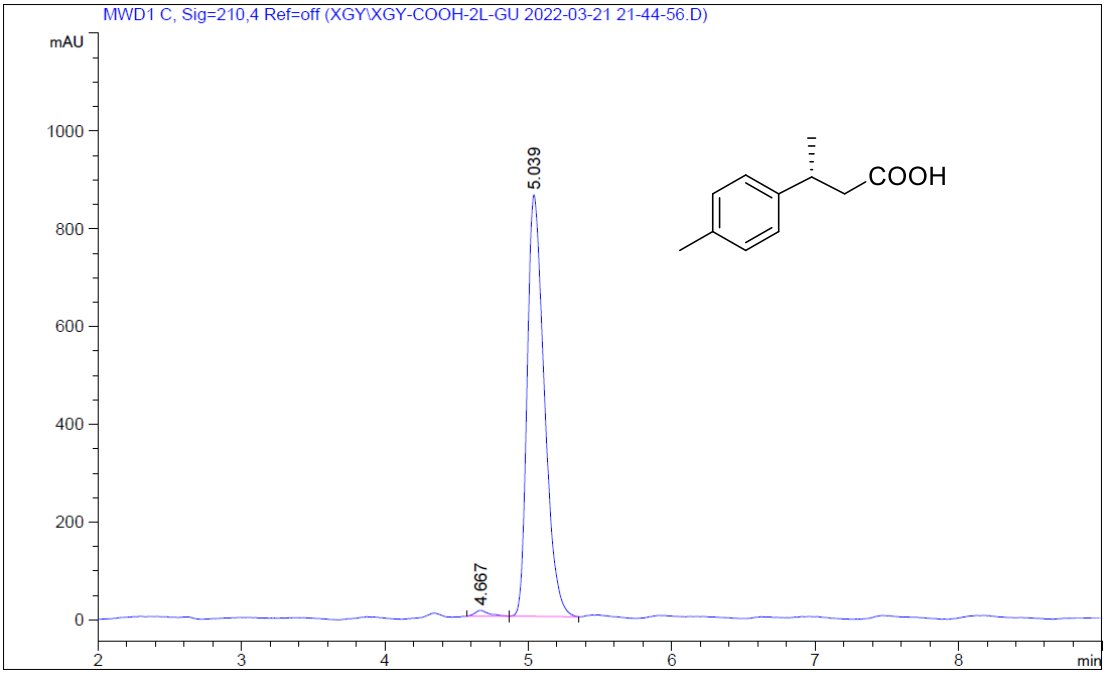


Signal 1: MWD1 C, Sig=210,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.193	BB	0.1568	91.70895	6.97732	1.1350
2	6.943	BV R	0.2294	7988.40869	478.13507	98.8650

(*R*)-3-(*p*-tolyl)butanoic acid (**2l**)

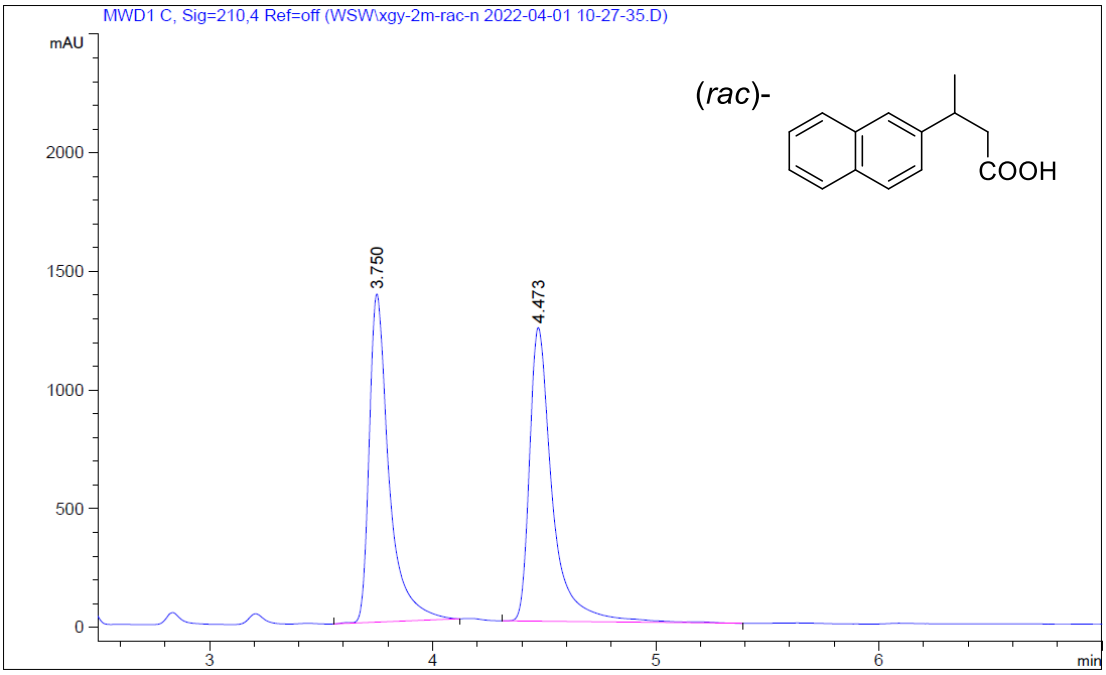


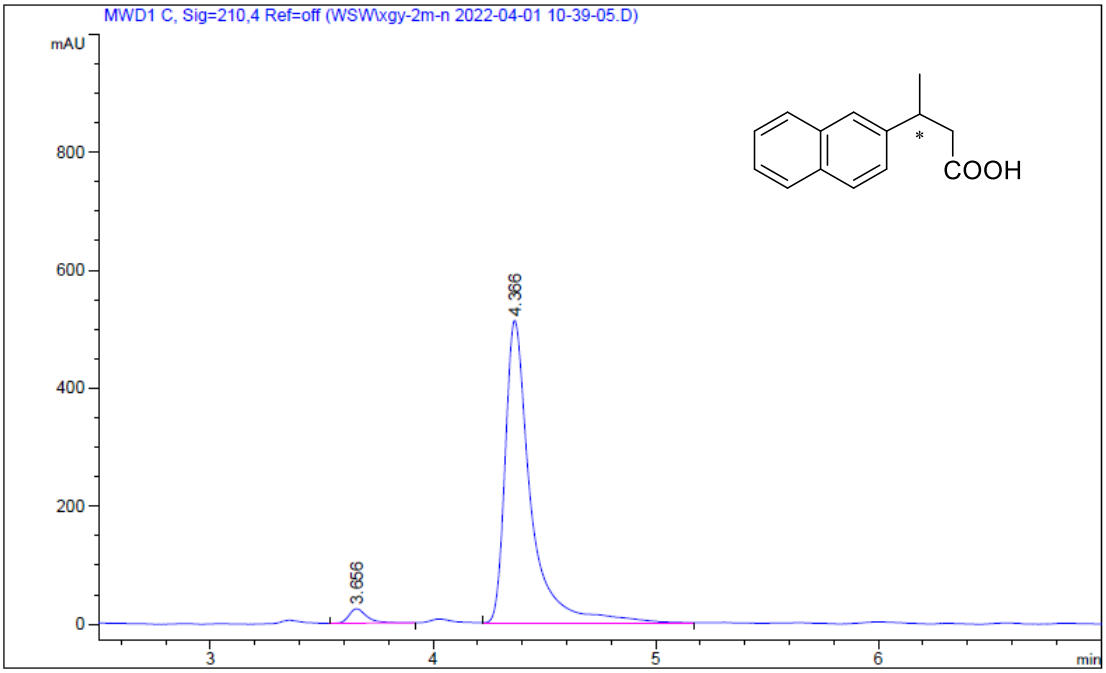


Signal 1: MWD1 C, Sig=210,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.667	BB	0.0844	80.88847	12.04179	1.1136
2	5.039	BB	0.1247	7182.53320	861.82855	98.8864

(-)-3-(naphthalen-2-yl)butanoic acid (**2m**)

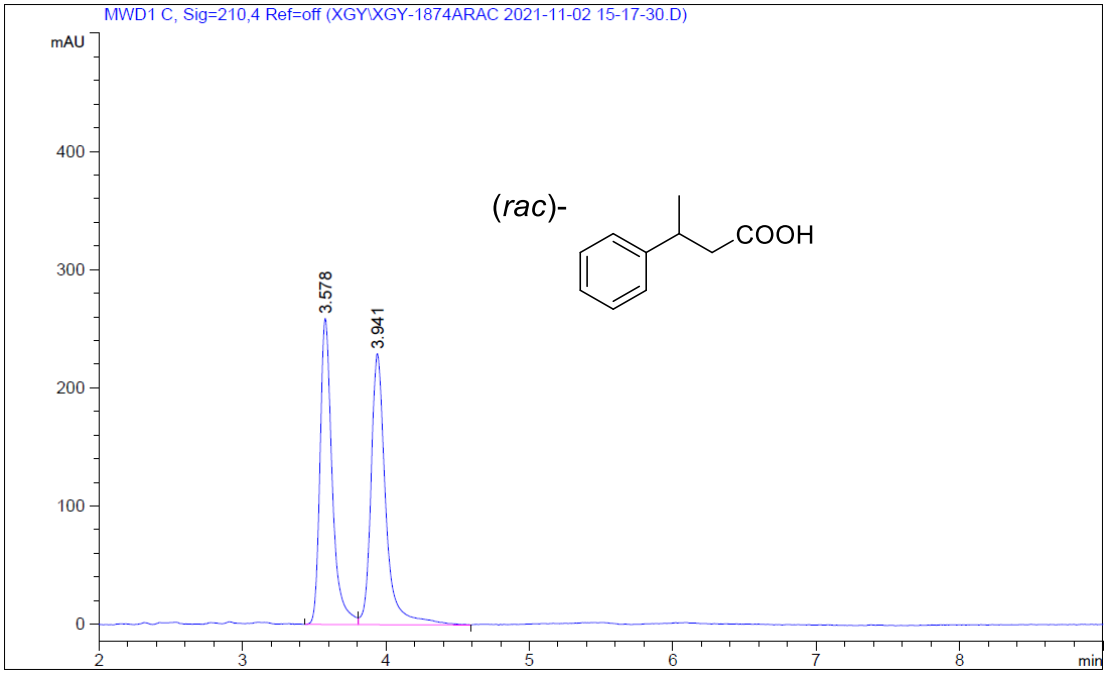


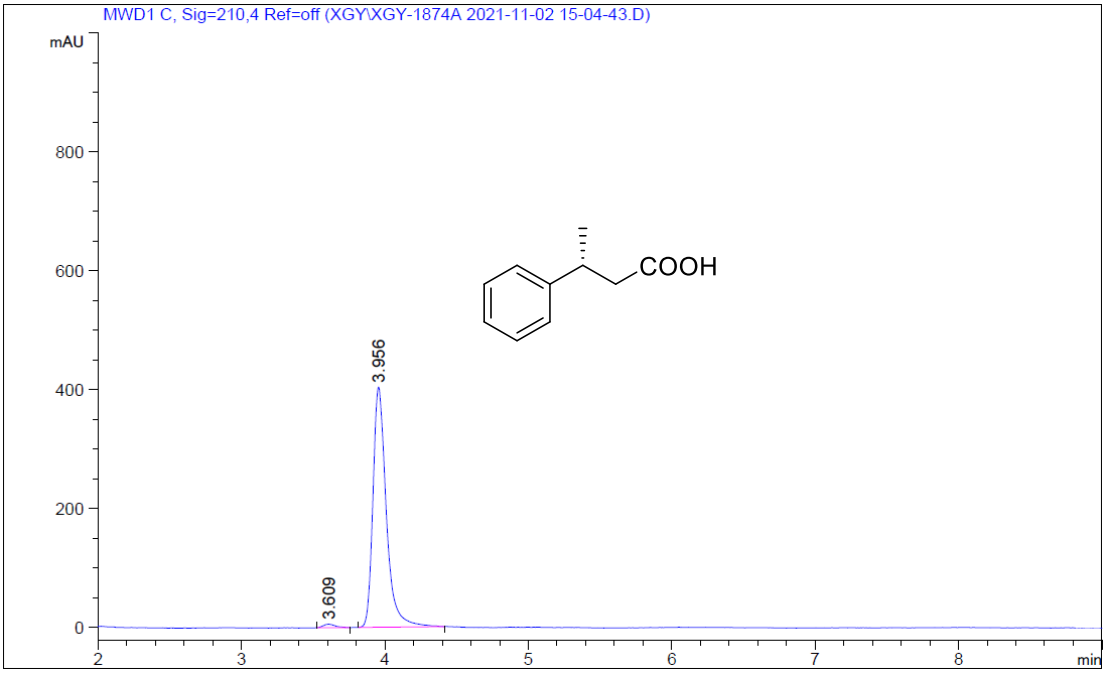


Signal 1: MWD1 C, Sig=210,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.656	BB	0.0829	138.97630	24.61253	3.3062
2	4.366	BB	0.1156	4064.57178	511.90485	96.6938

(*R*)-3-phenylbutanoic acid (**2n**)

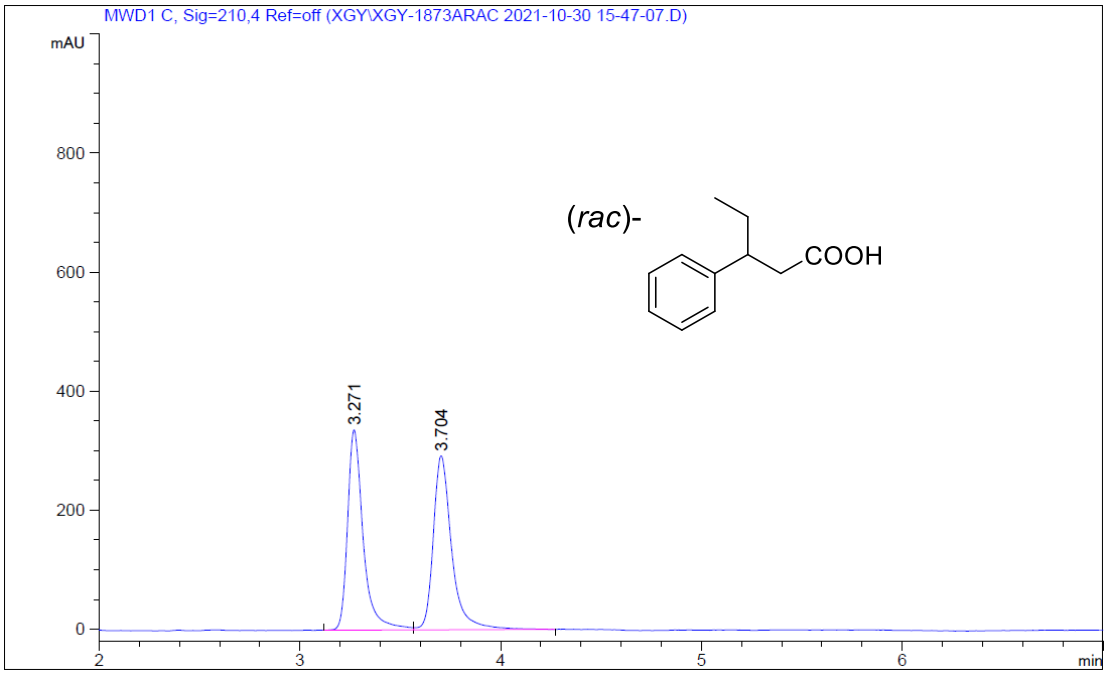


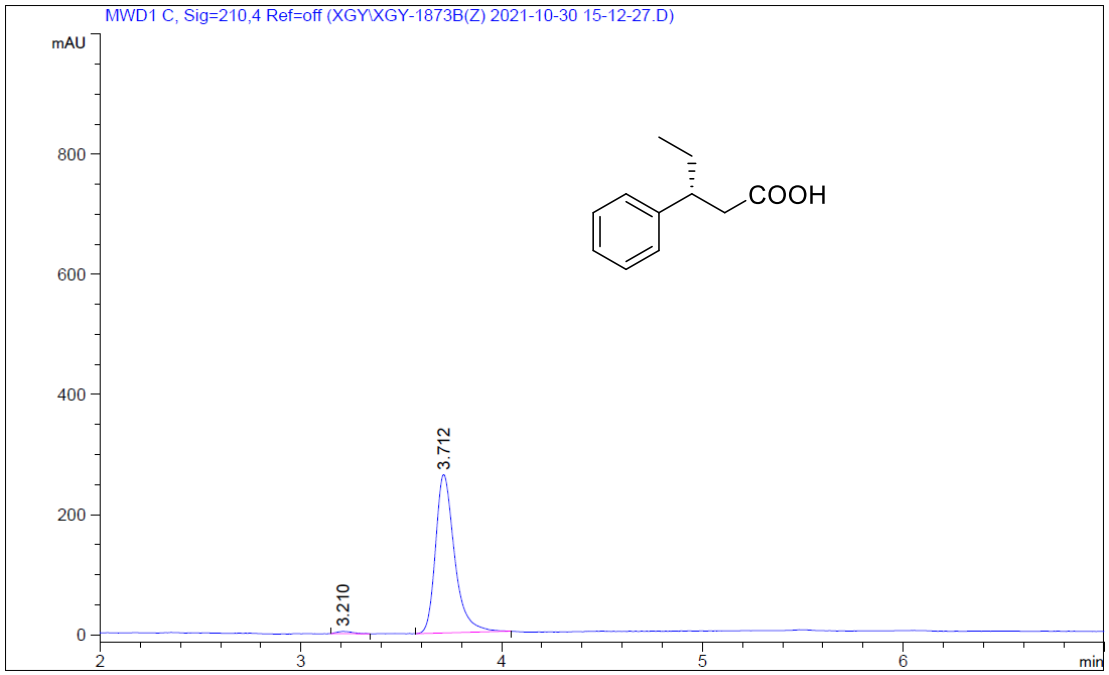


Signal 1: MWD1 C, Sig=210,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.609	BB	0.0689	33.49699	5.79442	1.2641
2	3.956	BB	0.0976	2616.37158	403.87708	98.7359

(*R*)-3-phenylpentanoic acid (**2o**)

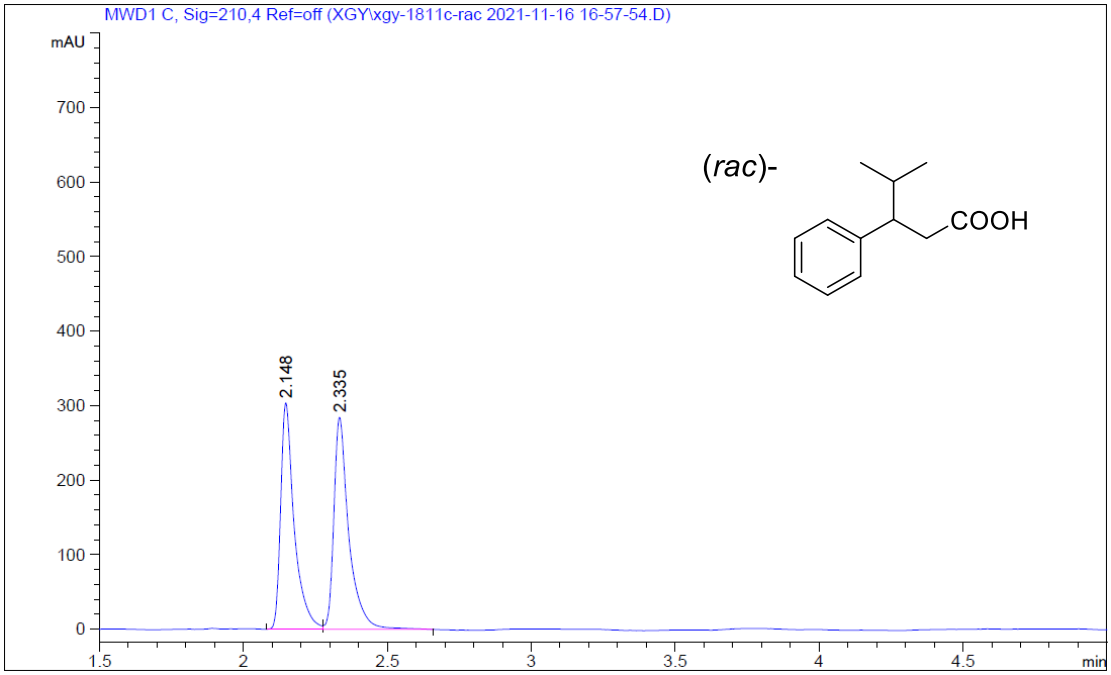


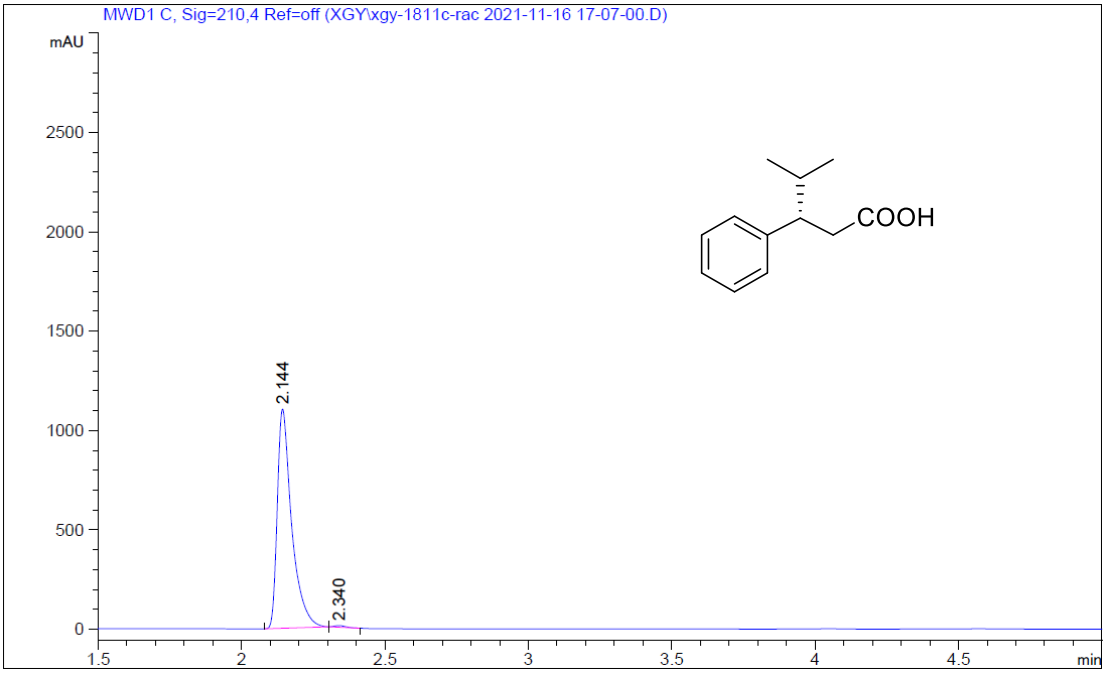


Signal 1: MWD1 C, Sig=210,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.210	BB	0.0693	20.82004	3.86967	1.1851
2	3.712	BB	0.0993	1735.92993	263.71954	98.8149

(*S*)-4-methyl-3-phenylpentanoic acid (**2p**)

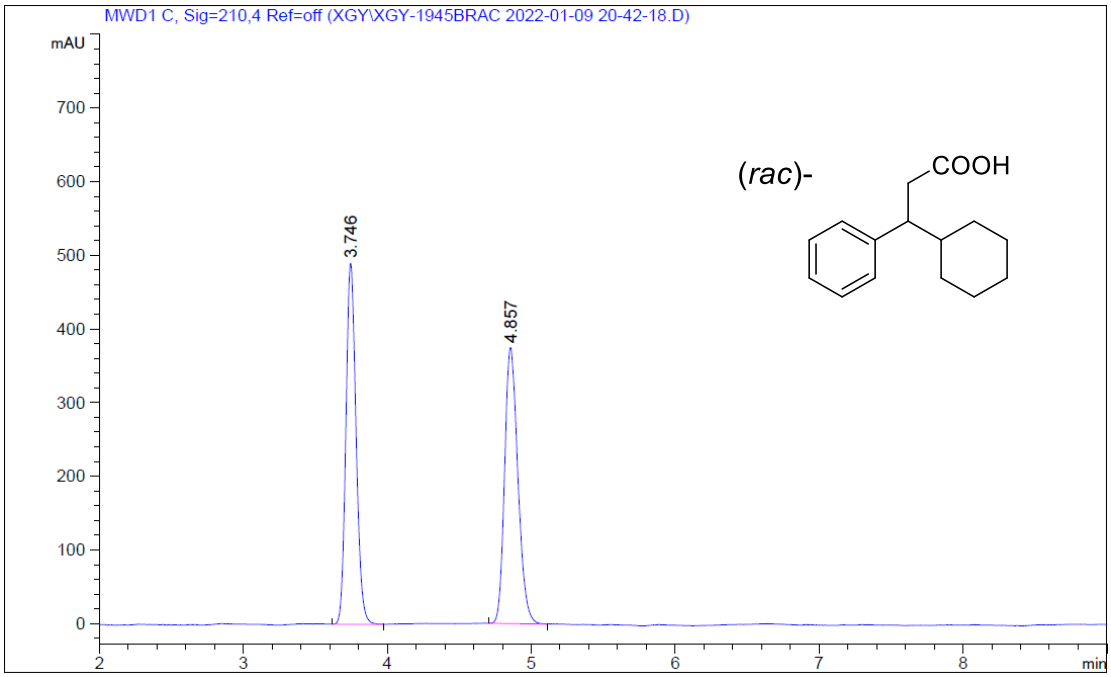


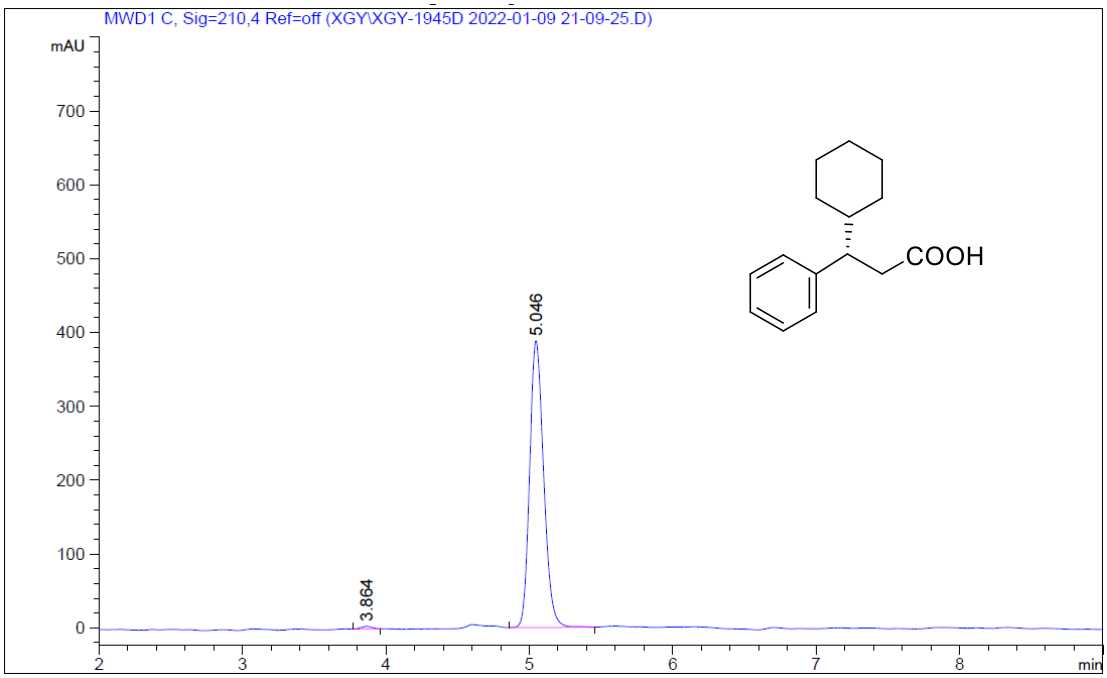


Signal 1: MWD1 C, Sig=210,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	2.144	BB	0.0517	3847.97534	1103.20752	99.4162
2	2.340	BB	0.0424	22.59569	8.53228	0.5838

(*S*)-3-cyclohexyl-3-phenylpropanoic acid (**2q**)

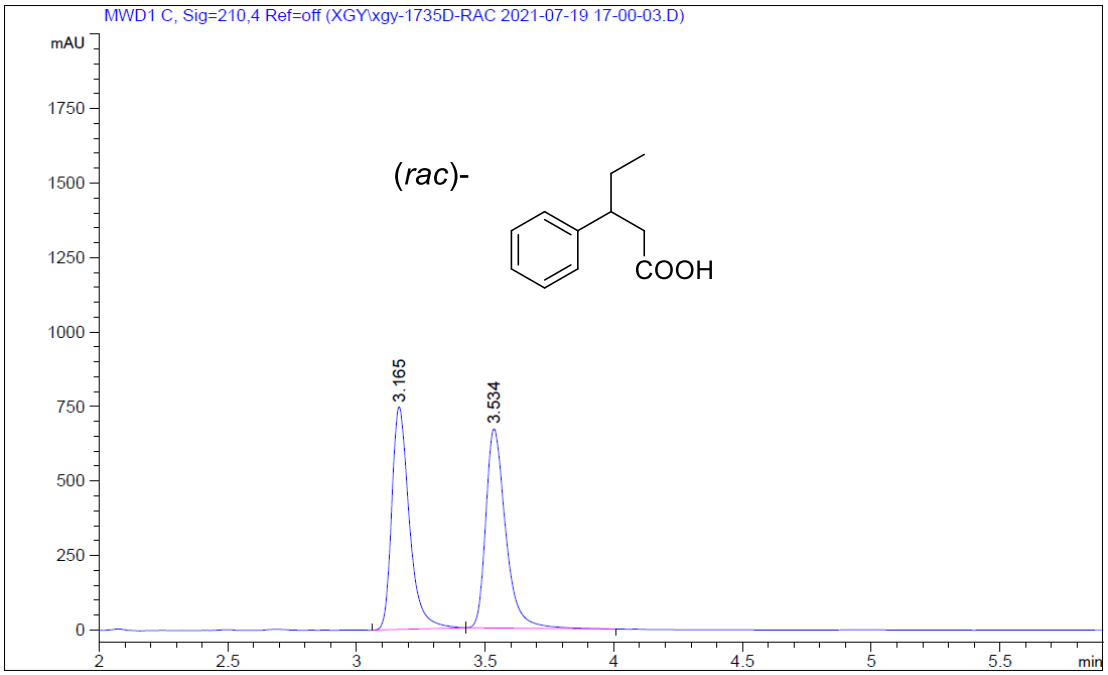


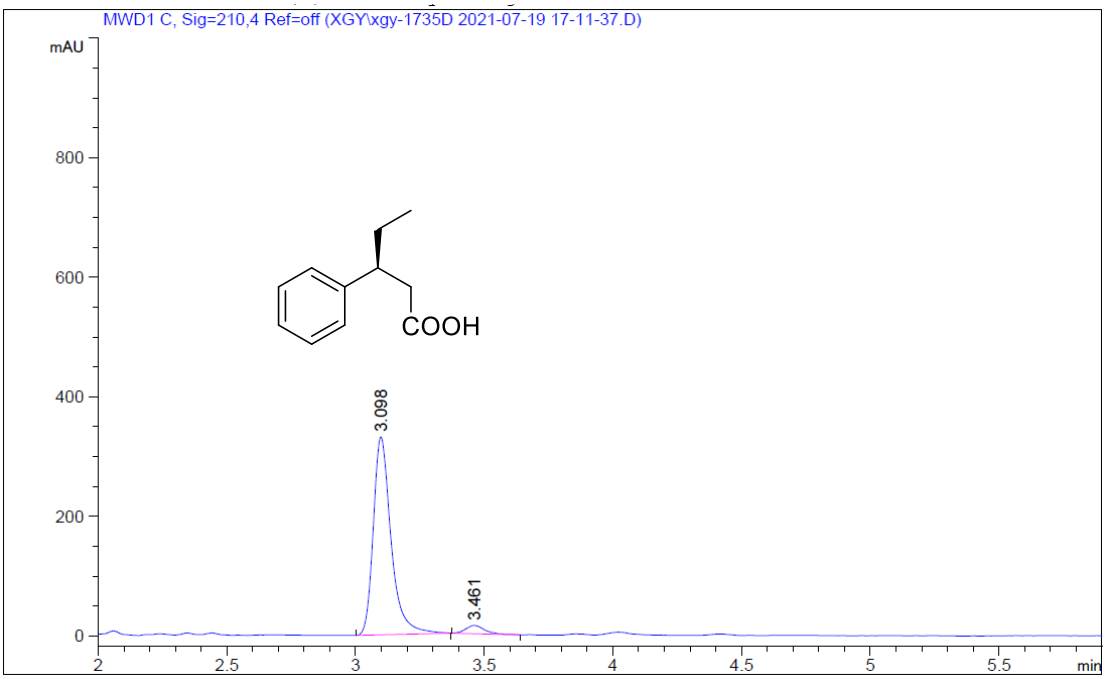


Signal 1: MWD1 C, Sig=210,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.864	VV R	0.0603	18.17768	3.73043	0.6833
2	5.046	BV R	0.1039	2642.14722	388.31042	99.3167

(*S*)-3-phenylpentanoic acid (**2r**)

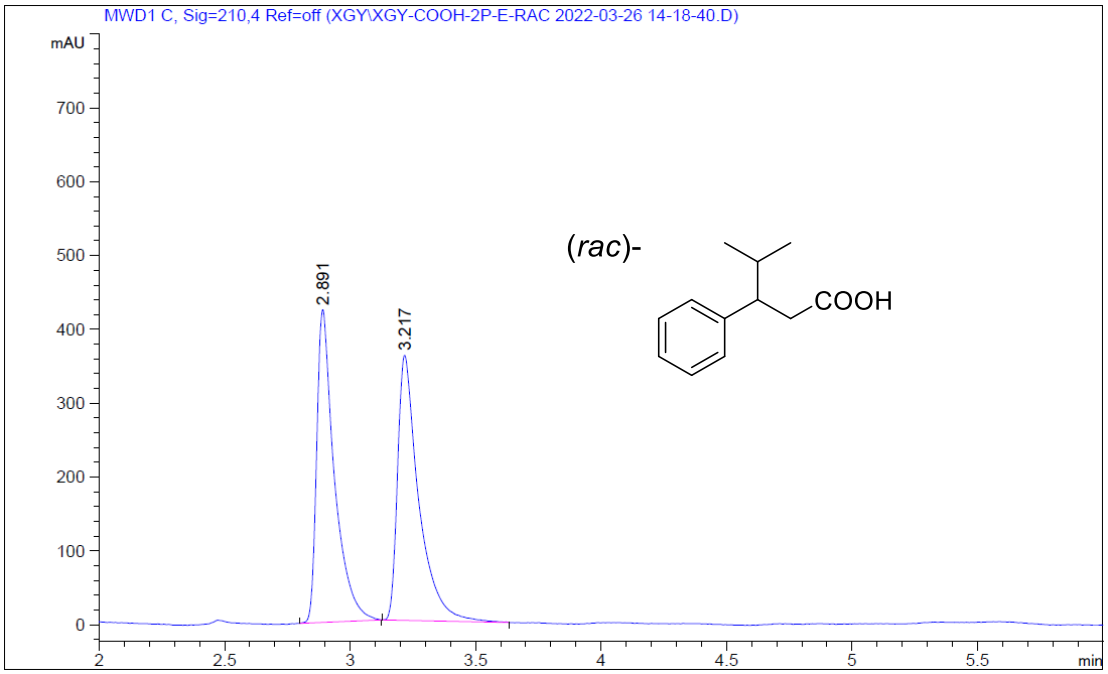


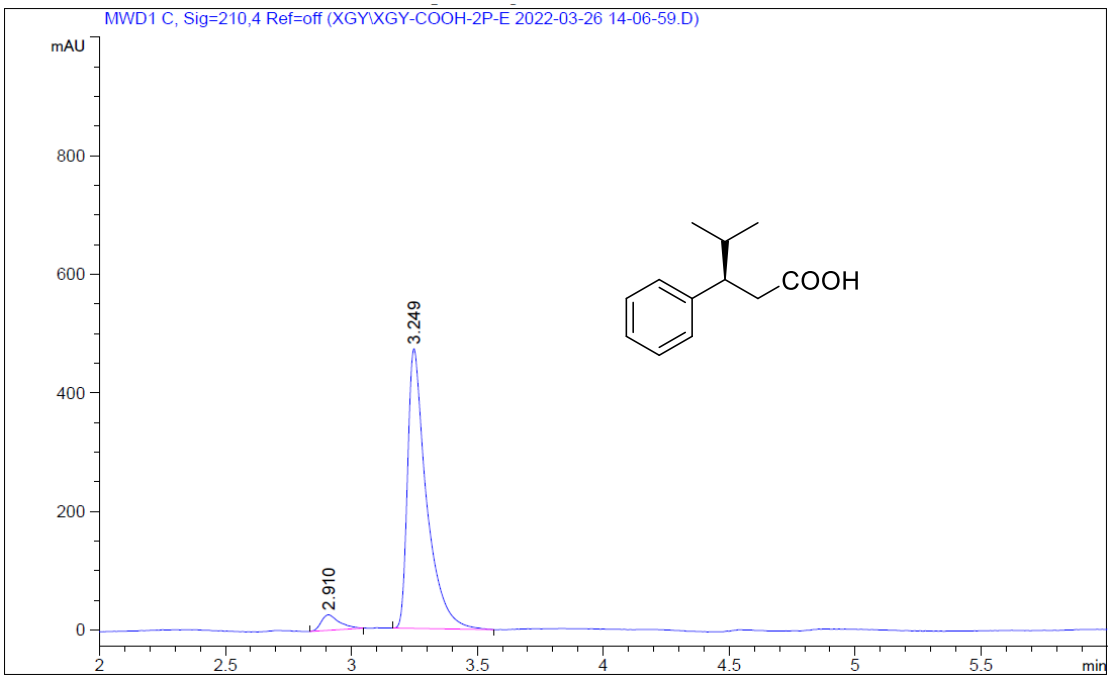


Signal 1: MWD1 C, Sig=210,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.098	BB	0.0736	1592.53467	331.01434	95.6464
2	3.461	BB	0.0686	72.48919	14.17848	4.3536

(*R*)-3-phenylpentanoic acid (**2s**)

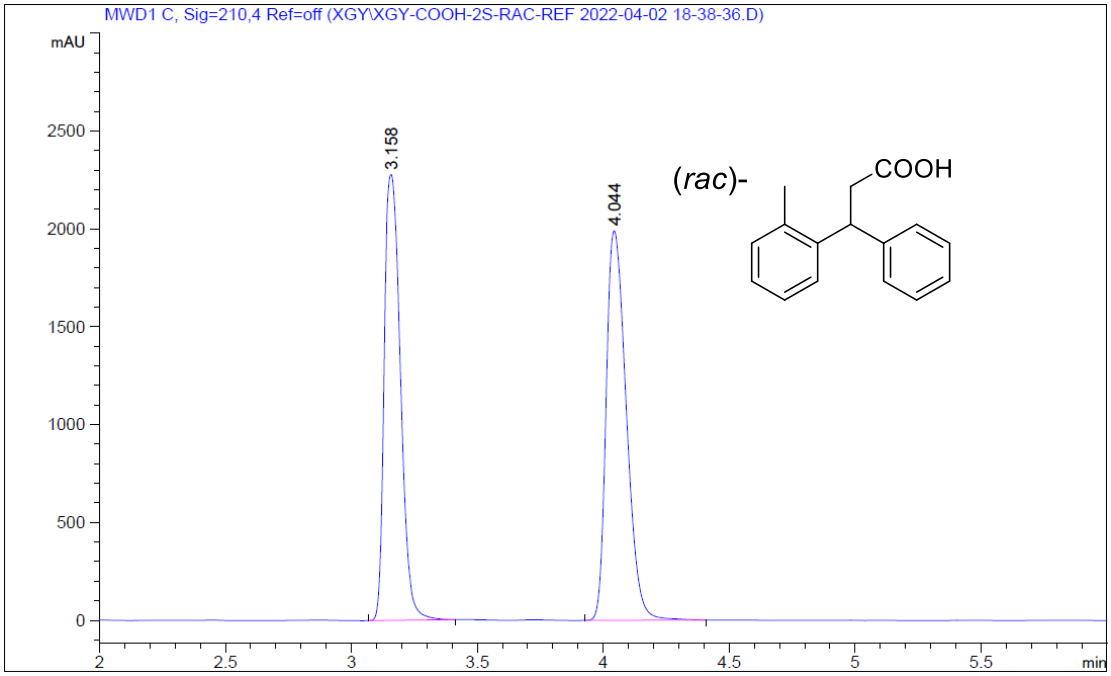


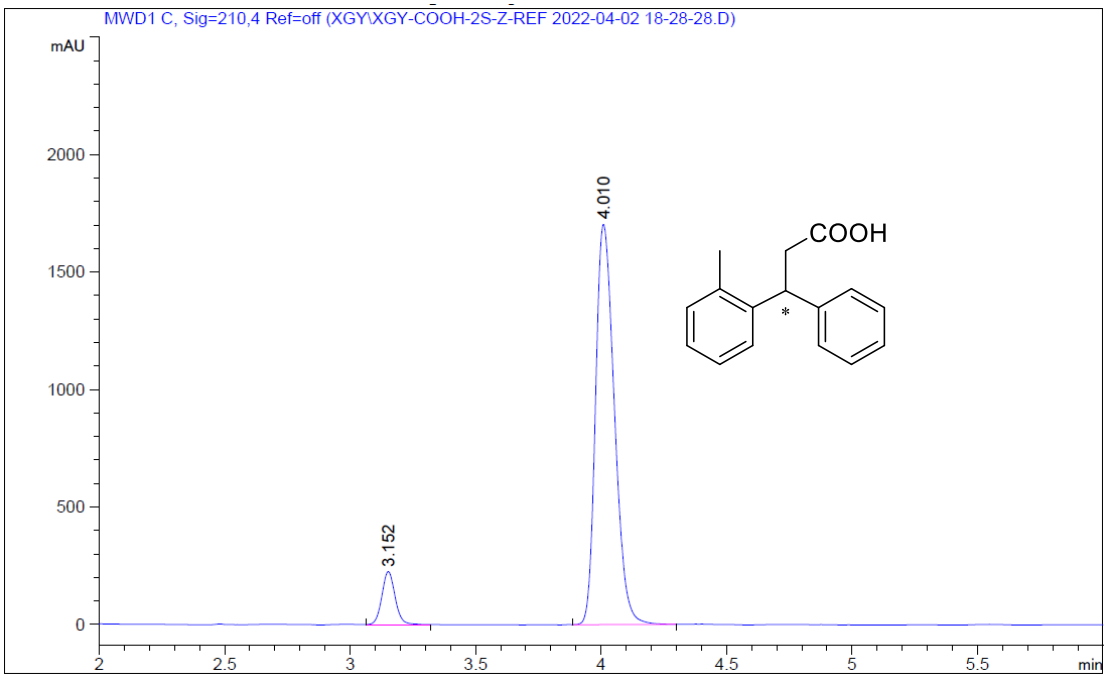


Signal 1: MWD1 C, Sig=210,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	2.910	BB	0.0704	130.26912	26.23104	5.0232
2	3.249	BB	0.0764	2463.08789	471.69113	94.9768

(-)-3-phenyl-3-(*o*-tolyl)propanoic acid (**2t**)

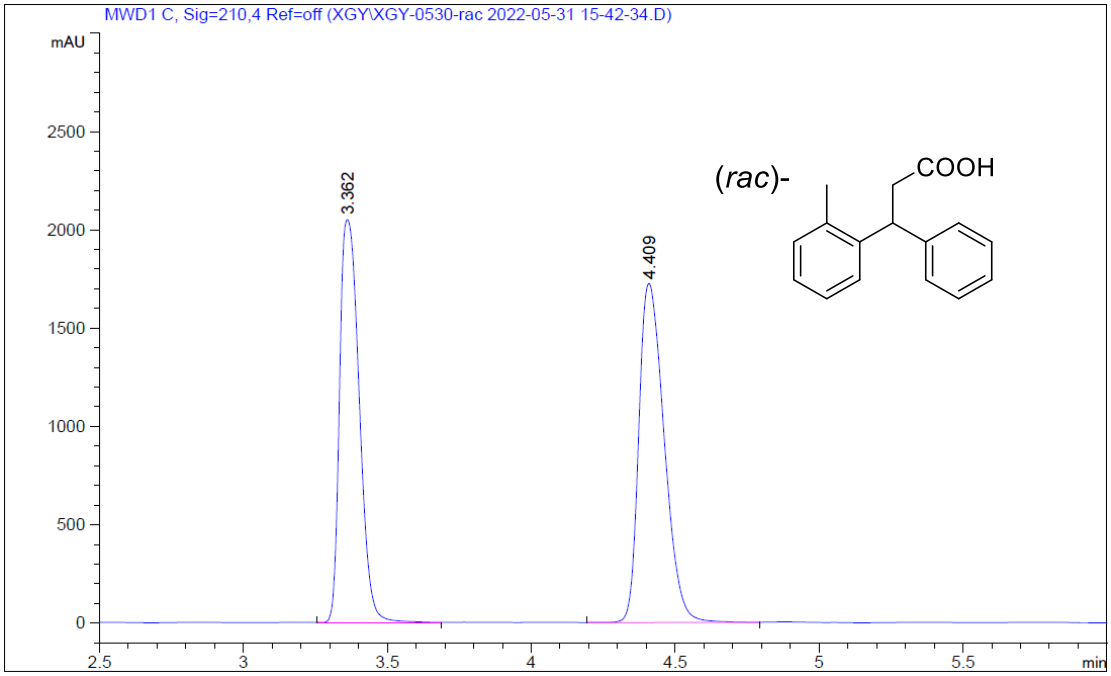


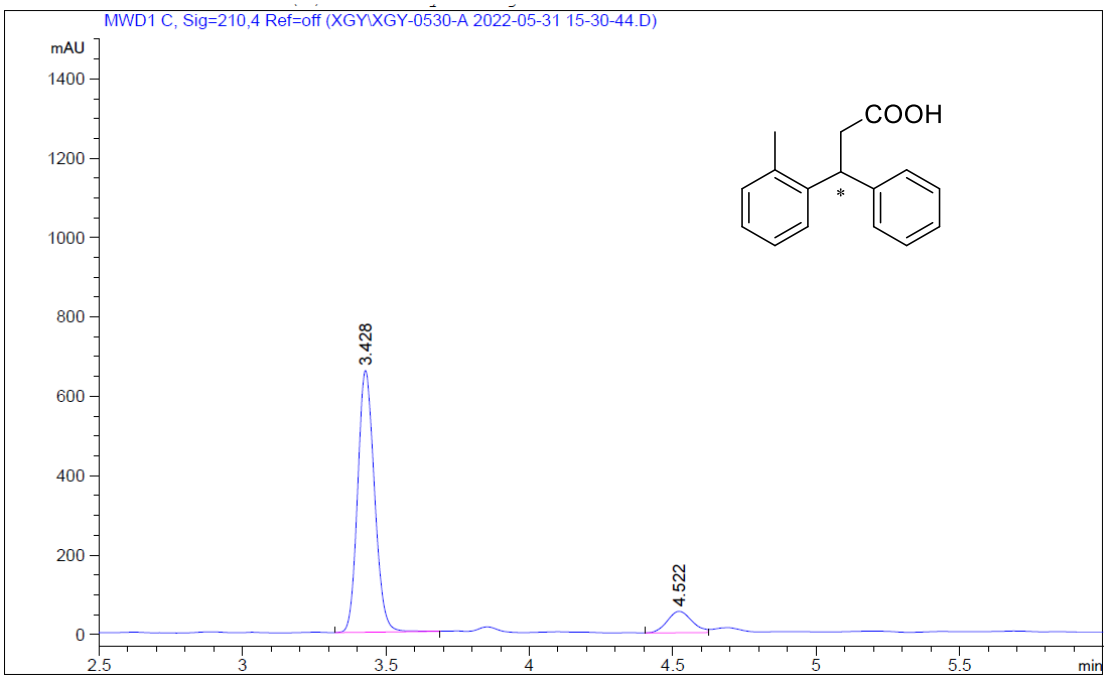


Signal 1: MWD1 C, Sig=210,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.152	BB	0.0559	830.93750	226.03180	8.5639
2	4.010	BB	0.0822	8871.81445	1704.39001	91.4361

(-)-3-phenyl-3-(*o*-tolyl)propanoic acid (**2u**)

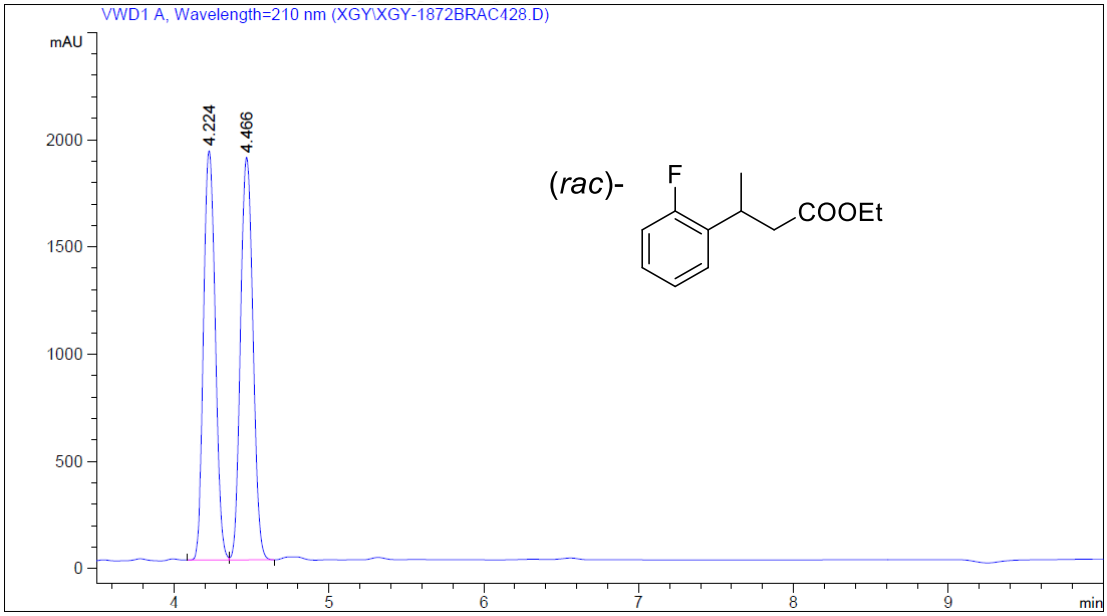


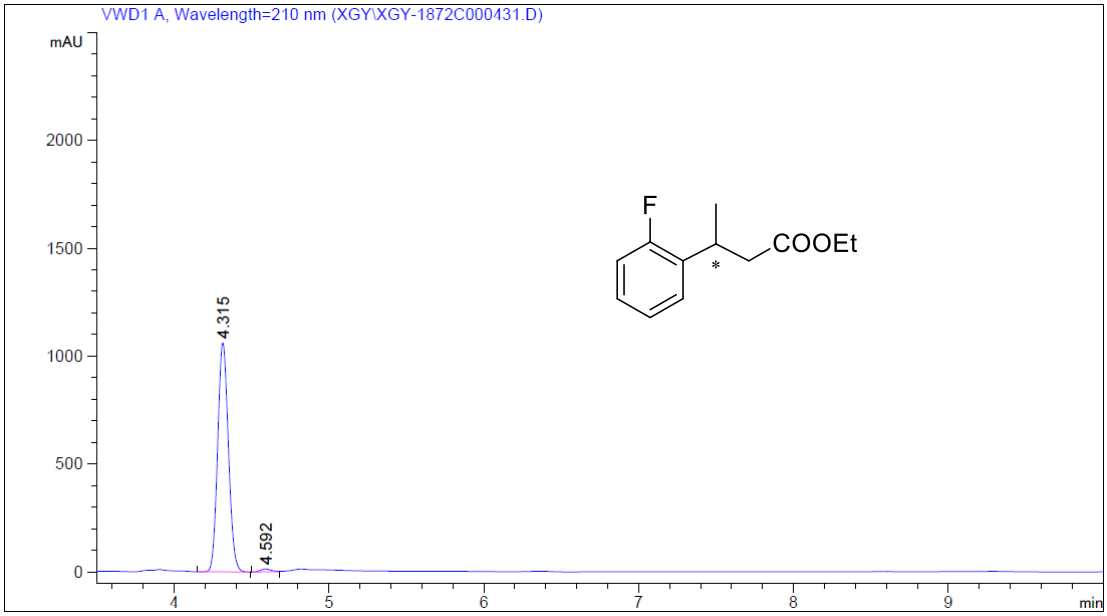


Signal 1: MWD1 C, Sig=210,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.428	BB	0.0636	2688.08936	659.51123	89.2509
2	4.522	BV	0.0921	323.74454	53.86615	10.7491

(-)-ethyl 3-(2-fluorophenyl)butanoate (**2v**)

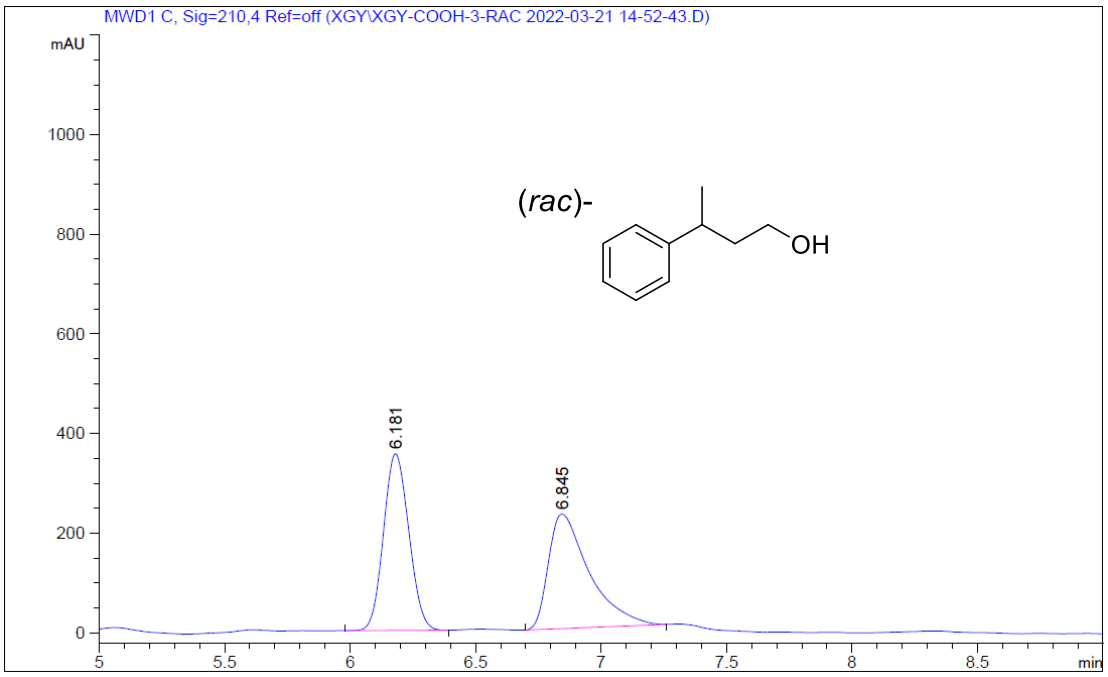


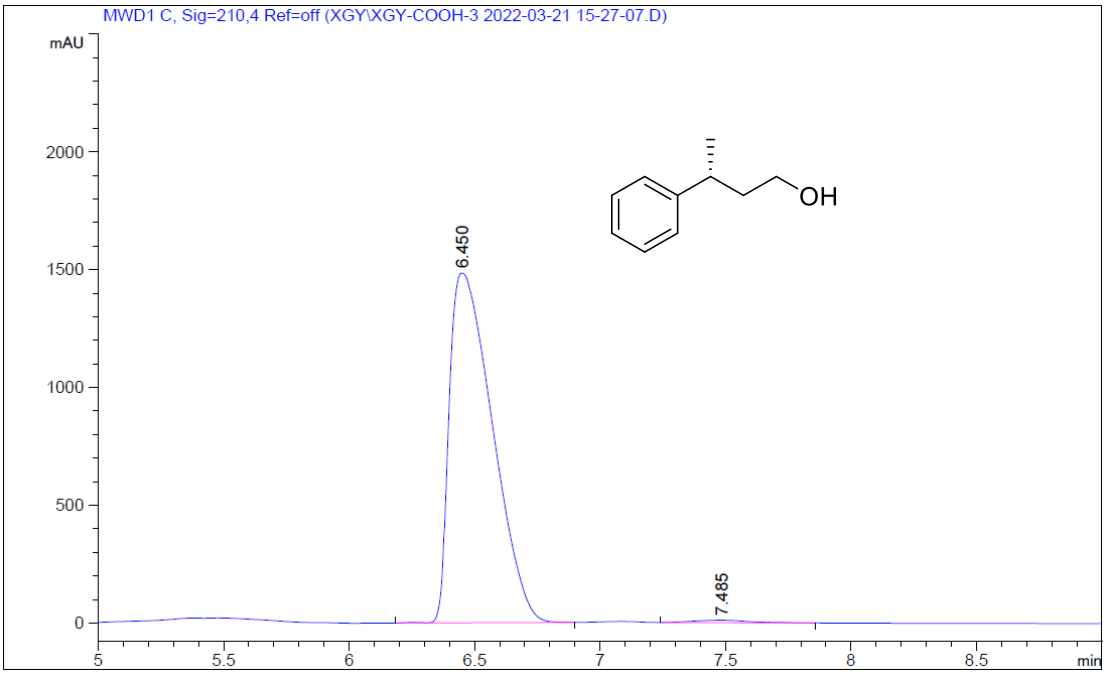


Signal 1: VWD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.315	BB	0.0750	5099.10742	1061.46033	98.9269
2	4.592	BB	0.0719	55.31410	12.19748	1.0731

(*R*)-3-phenylbutan-1-ol (**3**)

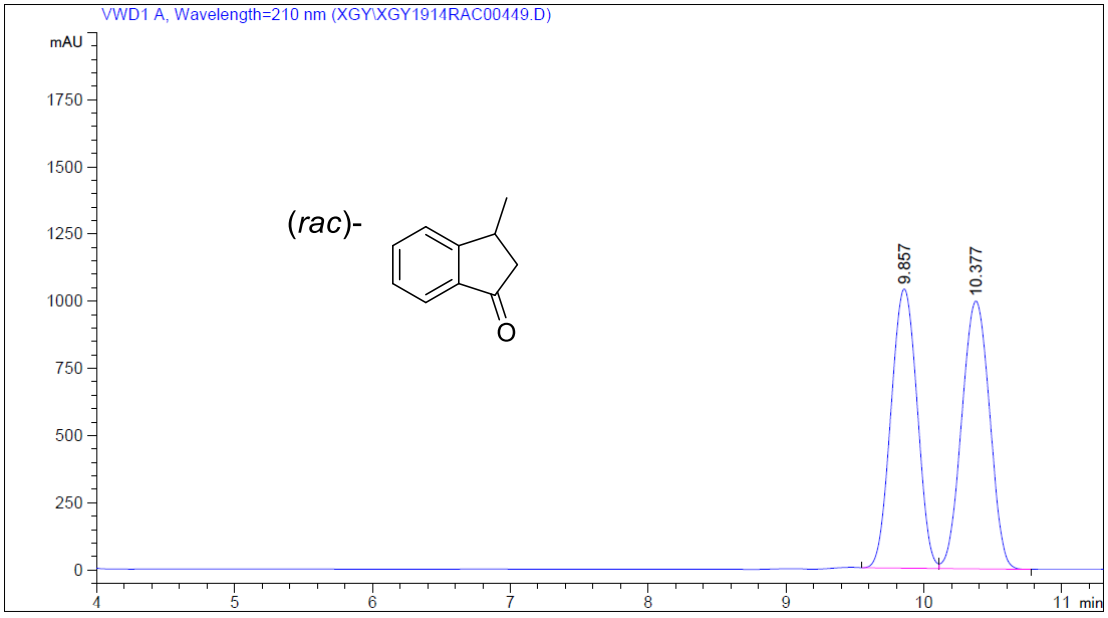


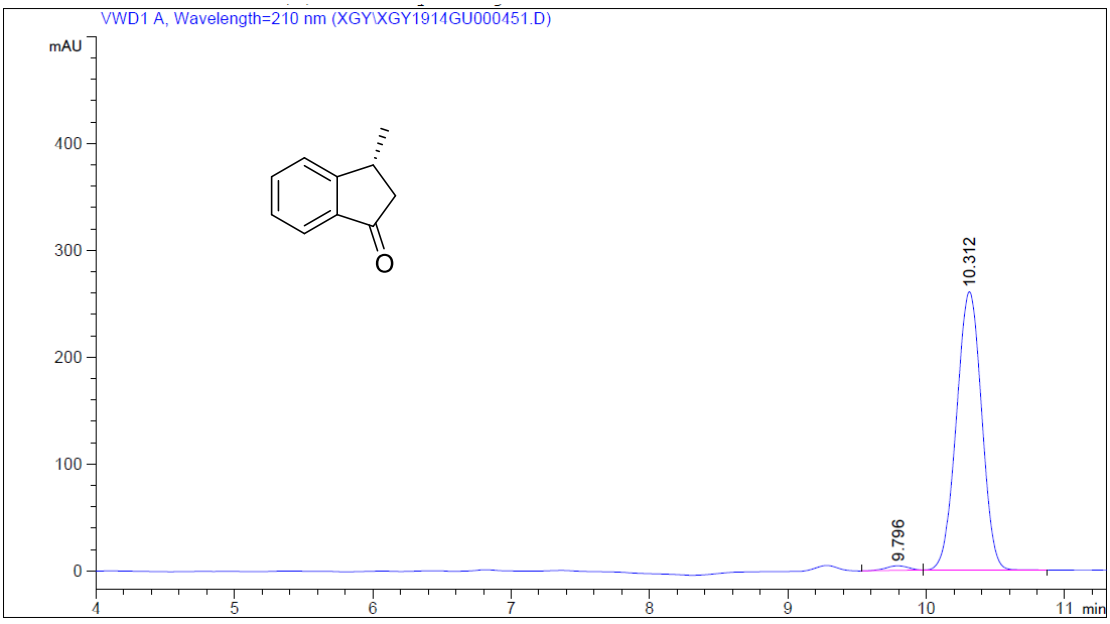


Signal 1: MWD1 C, Sig=210,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.450	VB R	0.1644	1.76845e4	1484.54358	99.0370
2	7.485	BB	0.1844	171.96130	11.08819	0.9630

(*R*)-3-methyl-2,3-dihydro-1*H*-inden-1-one (**4**)

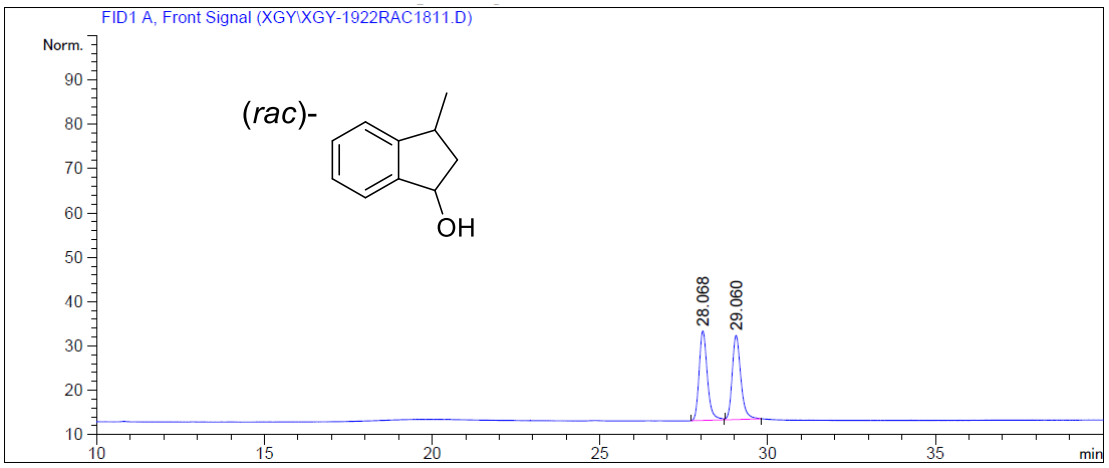


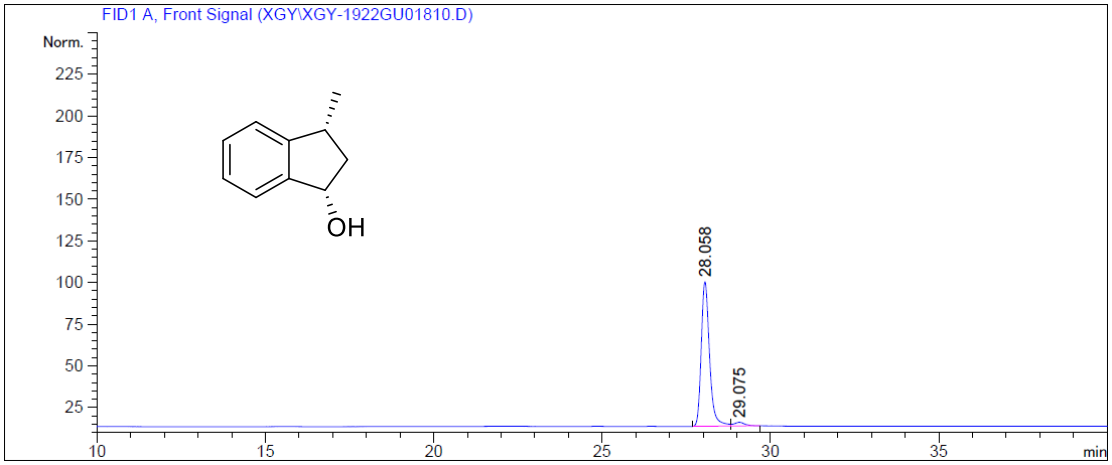


Signal 1: VWD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.796	BB	0.1744	47.77046	4.30690	1.4289
2	10.312	BB	0.1997	3295.41553	260.33994	98.5711

(1*S*,3*R*)-3-methyl-2,3-dihydro-1*H*-inden-1-ol (**5**)

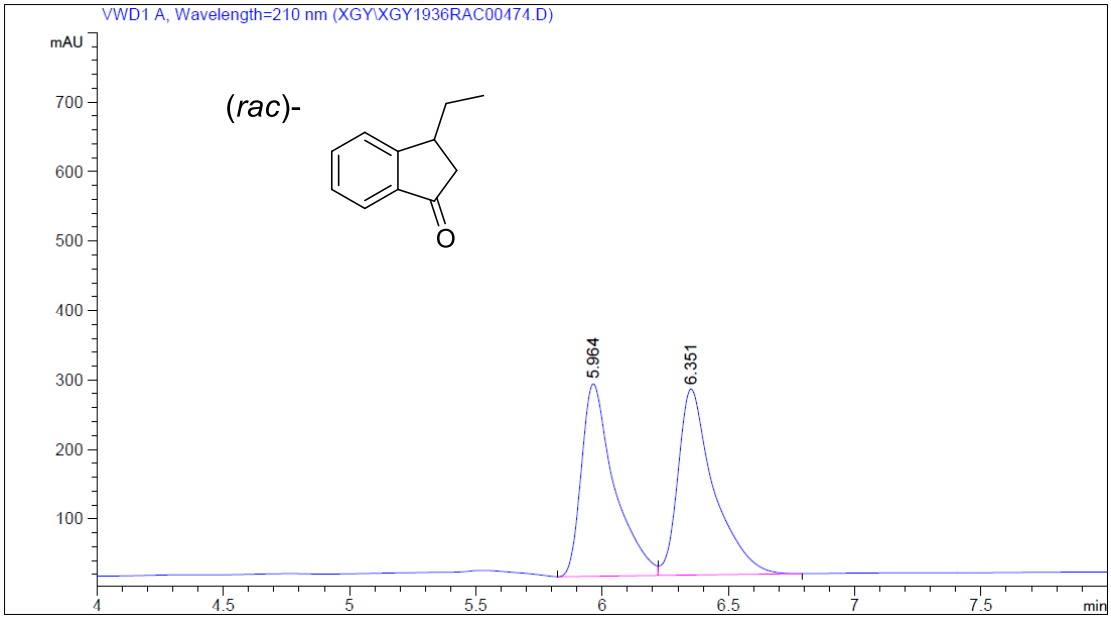


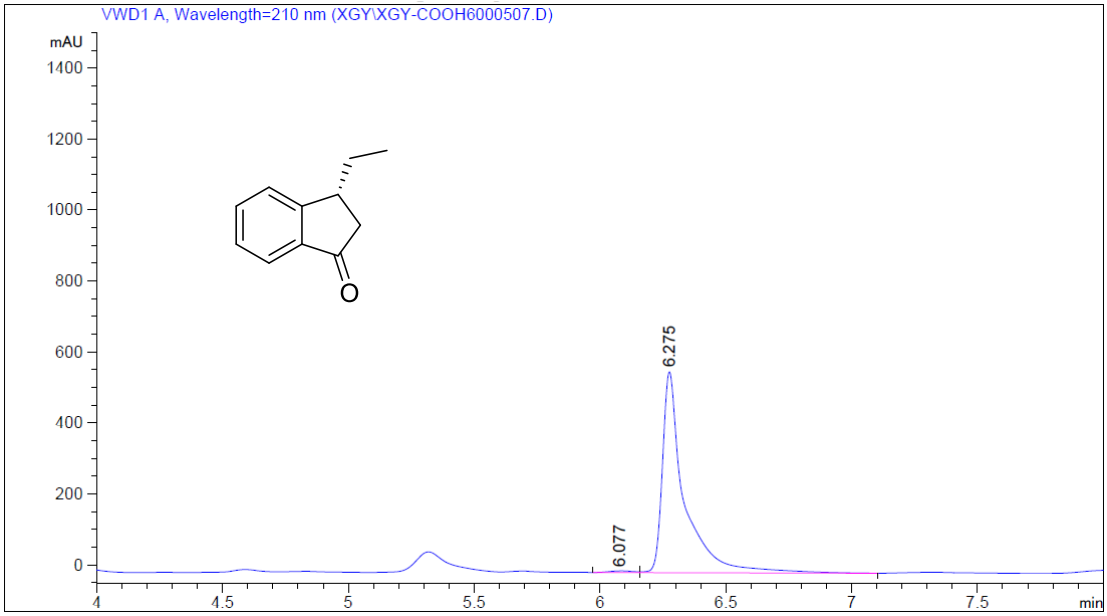


Signal 1: FID1 A, Front Signal

Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	28.058	BV	0.2600	1480.17676	86.67854	96.74832
2	29.075	VB	0.2673	49.74823	2.21756	3.25168

(*R*)-3-ethyl-2,3-dihydro-1*H*-inden-1-one (**6**)

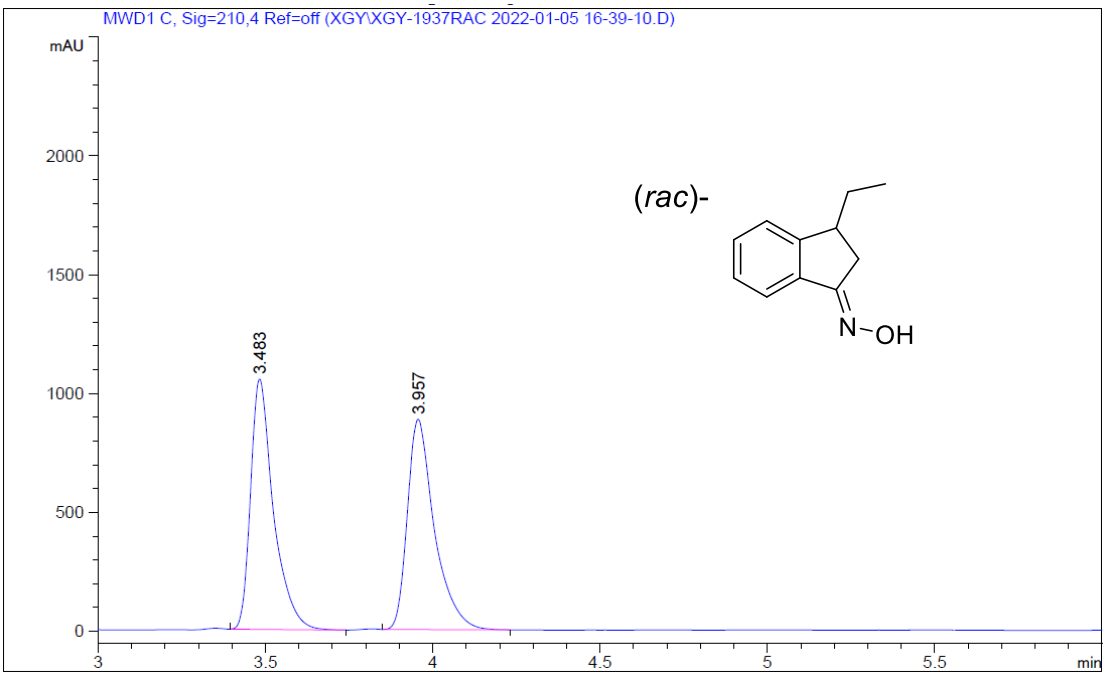


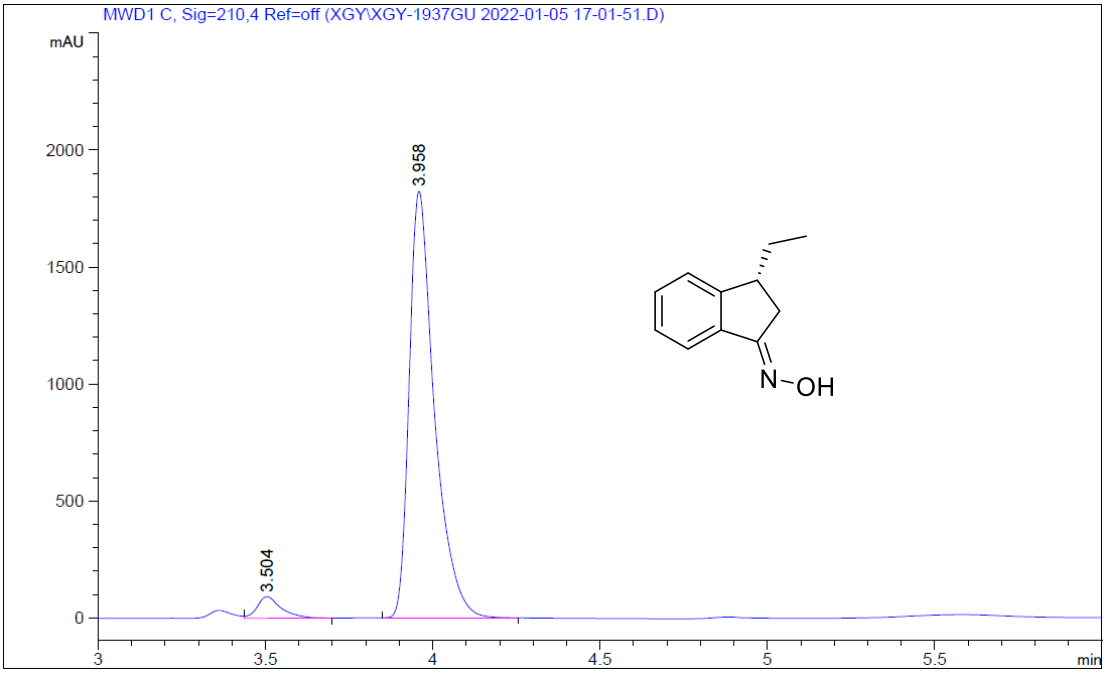


Signal 1: VWD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.077	BV	0.0940	29.93592	4.78182	0.8594
2	6.275	VB	0.0852	3453.41553	566.07141	99.1406

(*R*)-3-ethyl-2,3-dihydro-1*H*-inden-1-one oxime (**7**)

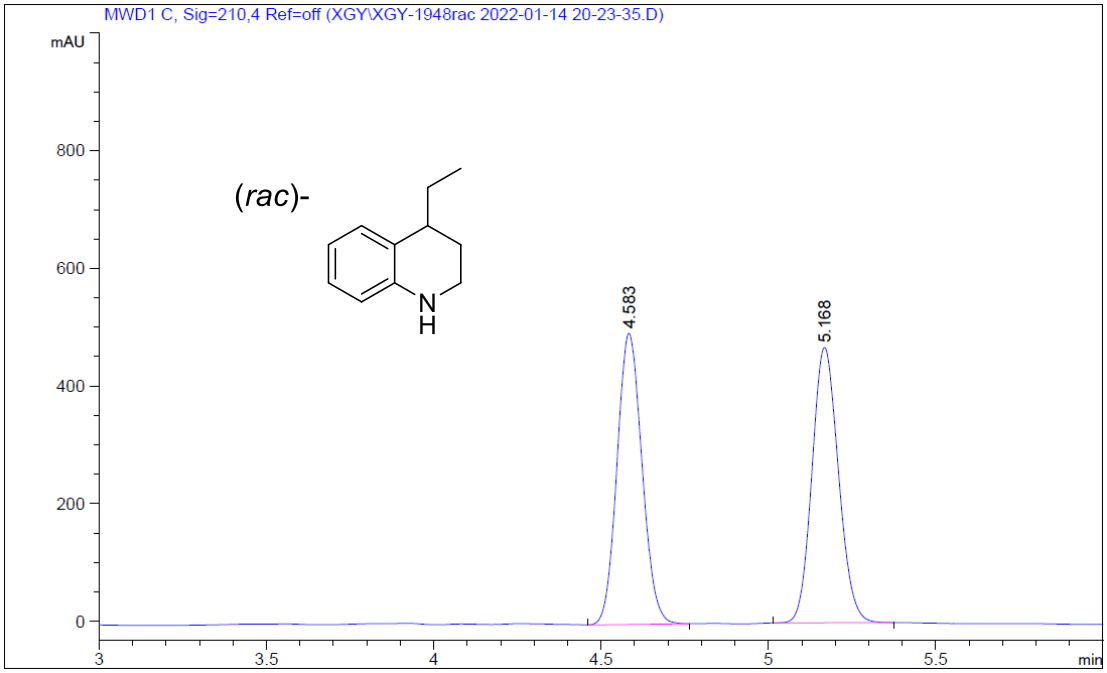


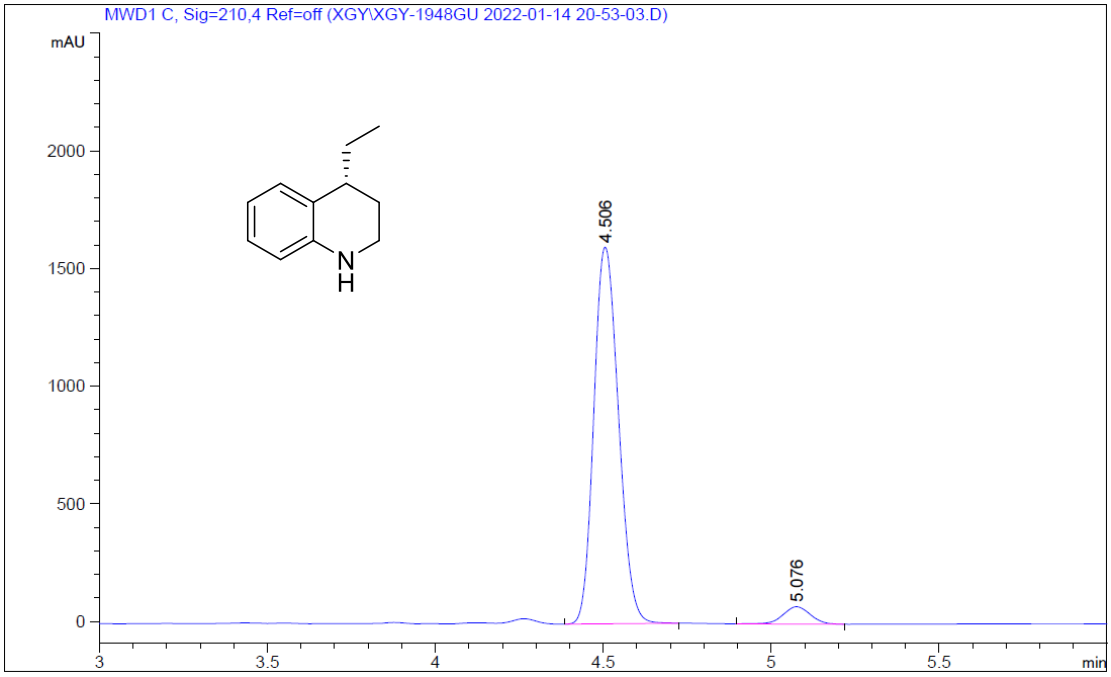


Signal 1: MWD1 C, Sig=210,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.504	BB	0.0670	388.37949	85.49287	3.8990
2	3.958	BB	0.0782	9572.69922	1823.70300	96.1010

(*R*)-4-ethyl-1,2,3,4-tetrahydroquinoline (**8**)





Signal 1: MWD1 C, Sig=210,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.506	BB	0.0811	8172.56982	1600.52576	95.1602
2	5.076	BB	0.0859	415.65088	73.60310	4.8398