Supporting Information for

A new ferrocenyl bisphosphorus ligand for asymmetric hydrogenation of α-methylene-γ-keto-carboxylic acids

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Contents

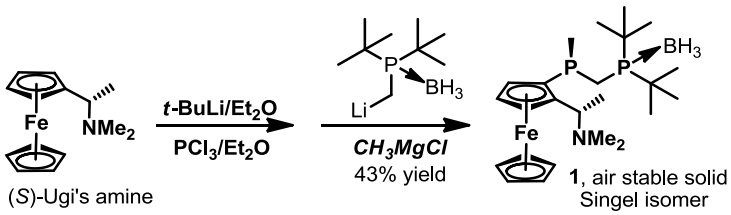
General remarks

All reactions were performed in the argon-filled glovebox or under nitrogen using standard Schlenk techniques, unless otherwise noted. Solvents were dried with standard procedures and degassed with N_2. Column chromatography was performed using 200~400 mesh silica gel. Thin layer chromatography (TLC) was performed on EM reagents 0.25 mm silica 60-F plates. ^{1}H, ^{13}C, and ^{31}P NMR spectrum were recorded on Bruker-400, with $CDCl_3$ as the solvent and tetramethylsilane (TMS) as the internal standard. Chemical shifts were reported in ppm, upfield to TMS (0.00 ppm) for and relative to $CDCl_3$ (7.26 ppm, 77.3 ppm) for ^{1}H NMR and ^{13}C NMR. Data are reported as: multiplicity (s = singlet, d = doublet, dd = double of doublet, t = triplet, dt = double of triplet, q = quartet, m = multiplet), coupling constant in hertz (Hz) and signal area integration in natural numbers. ^{13}C NMR and ^{31}P NMR analysis were run with decoupling. HPLC analysis was conducted on an Agilent 1260 Series instrument. High resolution mass spectrum was obtained on Thermo LTQ XL Orbitrap. Unless otherwise noted, all reagents and solvents were purchased from commercial suppliers and used without further purification.

The substrates for asymmetric hydrogenation were prepared according to the literature procedures. [1] The absolute configuration of products **3** was determined by comparison of optical data with those reported by the literature. [1c]

Synthesis of ligand *t*-Bu-Wudaphos

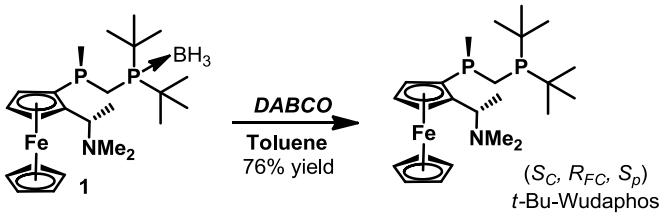
1. Synthesis of the intermediate **1**



To an oven dried Schlenk flask (100 mL) was added (*S*)-Ugi's amine (10 mmol, 2.57 g) and 20 mL of dry Et_2O under N_2 atmosphere. The resulting solution was cooled to -78 ℃ and *t*-BuLi (11 mmol, 1.5 M in pentane, 7.6 mL) was added carefully and dropwise. After the addition, the solution was allowed to warm to room

temperature (rt) and stirred for 1.5 h. The Schlenk flask was cooed to -78 ℃ and PCl_3 (10 mmol, 1.0 mL) was added in one portion. The yellow suspension was allowed to warm to rt and stirred for 1.5 h. The Schlenk flask was cooled to -78 ℃ and borane protected ((di-tert-butylphosphino)methyl)lithium (11 mmol, prepared by treating borane protected di-tert-butyl(methyl)phosphine[2] with *s*-BuLi in an 1:1.1 molar ratio under 0 ℃ for 1.0 h in Et_2O) was added dropwise and the resulting suspension was allowed to warm to rt and stirred for 1.5 h. The Schlenk flask was cooled to -78 ℃ again and CH_3MgCl (3.0 M in Et_2O, 3.7 mL) was added dropwise. The resulting yellow suspension was allowed to warm to rt and stirred for 3 h. Water (20 mL) was added into the Schlenk flask and the solution was stirred for 10 min. The organic phase was separated and the aqueous phase was extracted by ethyl acetate (30 mL X 3). The organic phases were combined, dried and concentrated under reduced pressure. The residue was purified by column chromatography to give the desired product **1** as a yellow solid (2.04g, 43% yield). $[\alpha]_D^{20}$ = 22.000 (c 0.100, $CHCl_3$); HRMS (ESI) calculated for $C_{24}H_{45}BFeNP_2^+$ [M + H^+]: 476.2464; found: 476.2462. ^{1}H NMR (400 MHz, $CDCl_3$): δ 4.31 (s, 1H), 4.26 (s, 1H), 4.22 (d, *J* = 0.9 Hz, 1H), 4.15 (dd, *J* = 11.6, 4.8 Hz, 1H), 4.12 – 4.03 (m, 5H), 2.30 (t, *J* = 14.8 Hz, 2H), 2.06 (s, 6H), 1.61 (d, *J* = 4.1 Hz, 3H), 1.42 – 1.34 (m, 9H), 1.25 (d, *J* = 6.5 Hz, 3H), 1.18 (d, *J* = 12.4 Hz, 9H), 0.66 (brs, 1H), 0.40 (brs, 1H), 0.21 (brs, 1H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$) δ 95.92, 69.84 (d, *J* = 3.6 Hz), 69.56, 68.14, 67.82 (d, *J* = 5.3 Hz), 56.71 (d, *J* = 7.6 Hz), 39.73, 32.72 (dd, *J* = 51.9, 26.0 Hz), 28.25 (d, *J* = 6.5 Hz), 27.72, 25.19, 21.85 (dd, *J* = 32.7, 18.7 Hz), 12.29 (d, *J* = 12.7 Hz), 7.21 ppm; ^{31}P NMR (162 MHz, $CDCl_3$): δ 43.02 (s), -48.34 (d, *J* = 25.8 Hz) ppm.

2. Synthesis of the *t*-Bu-Wudaphos

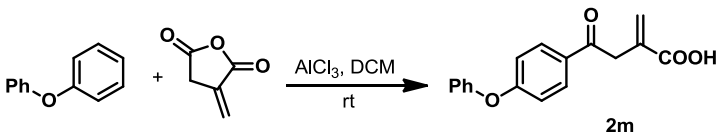


To an oven dried Schlenk flask (100 mL) was added the intermediate **1** (4 mmol,

1.90 g), DABCO (4.8 mmol, 540 mg) and 50 mL of dry toluene under N_2 atmosphere. The reaction system was heated to 80 ℃ and stirred for 8 h. The resulting solution was concentrated under reduced pressure. The residue was purified by column chromatography to give the desired product *t*-Bu-Wudaphos (air stable yellow solid, 1.40 g, 76% yield). $[\alpha]_D^{20}$ = 25.385 (c 0.130, $CHCl_3$); HRMS (ESI) calculated for $C_{24}H_{42}FeNP_2^+$ [M + H^+]: 462.2136; found: 462.2130. ^{1}H NMR (400 MHz, $CDCl_3$): δ 4.29 (brs, 1H), 4.26 – 4.20 (m, 2H), 4.15 (qd, *J* = 6.7, 2.5 Hz, 1H), 4.08 (s, 5H), 2.08 (s, 6H), 2.06 – 2.03 (m, 1H), 1.75 – 1.61 (m, 1H), 1.47 (d, *J* = 4.1 Hz, 3H), 1.24 (d, *J* = 6.8 Hz, 3H), 1.21 (d, *J* = 11.2 Hz, 9H), 1.06 (d, *J* = 11.1 Hz, 9H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$): δ 96.31 (d, *J* = 22.3 Hz), 81.47 (dd, *J* = 16.4, 15.0 Hz), 70.16, 69.41, 67.67, 67.33 (d, *J* = 5.9 Hz), 56.88 (d, *J* = 7.3 Hz), 39.69, 31.86 (dd, *J* = 20.8, 2.6 Hz), 31.33 (dd, *J* = 22.0, 8.5 Hz), 29.78 (dd, *J* = 13.1, 4.4 Hz), 29.52 (d, *J* = 13.5 Hz), 25.10 (dd, *J* = 33.5, 18.1 Hz), 10.35 (dd, *J* = 11.0, 6.9 Hz), 7.51 ppm; ^{31}P NMR (162 MHz, $CDCl_3$): δ 20.29 (d, *J* = 107.5 Hz), -49.86 (d, *J* = 107.6 Hz) ppm.

Preparation of compounds 2m and 8

1. Preparation of compound **2m**

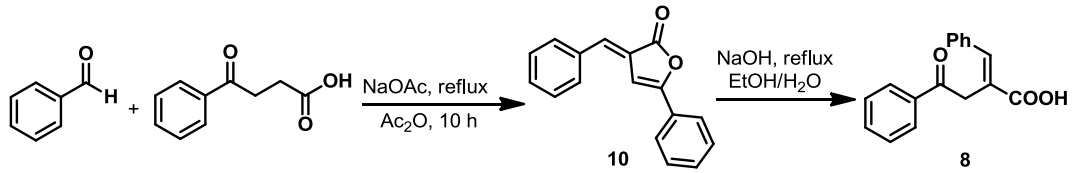


2m

The following is a modified literature procedure. [1c] To an oven dried Schlenk flask with DCM (5 mL) was added $AlCl_3$ (20 mmol, 2.67 g) portion-wise under argon atmosphere. 3-methylenedihydrofuran-2,5-dione (10 mmol, 1.12 g) was then added and the oxydibenzene (15 mmol, 2.55 g) was added dropwise. The resulting suspension was stirred at rt for 2 h. After the reaction, the suspension was poured into ice water (20 mL) and conc. HCl aqueous solution (5 mL) was added dropwise. Hexane (30 mL) was then added and the mixture was then stirred for 20 min and filtered. The filtrate was washed with 3M HCl solution (30 mL X 1), PE (30 mL X 2) and dried. The crude product was further recrystallized with PE/EA as the solvent to

give the pure product **2m** as a white solid (2.54 g, 90% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01–7.93 (m, 2H), 7.41 (dd, *J* = 8.4, 7.5 Hz, 2H), 7.25–7.18 (m, 1H), 7.12–7.06 (m, 2H), 7.05–6.98 (m, 2H), 6.53 (s, 1H), 5.81 (s, 1H), 3.97 (s, 2H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ 195.51, 171.53, 162.51, 155.63, 134.33, 131.18, 131.09, 130.88, 130.36, 124.97, 120.52, 117.59, 41.36 ppm.

2. Preparation of compound **8**

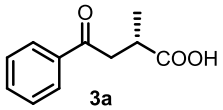


The compound **10** was synthesized according to the reported procedure.[1d] A mixture of 10 mmol of benzaldehyde, 10 mmol of 3-benzoylpropionic acid, 10 mmol of sodium acetate and 5 mL of acetic anhydride was heated under reflux for 10 h until crystals separated. After cooling, the reaction was poured into water, and the solid product was filtered, washed with water and finally recrystallized from 95% ethanol. Pure product was easily obtained by filtration (compound **10**, 1.32 g, 53% yield). The compound **8** was synthesized according to a modified literature procedure.[1e] A mixture of 0.21 g (5.3 mmol) of sodium hydroxide, 5.3 mmol of the compound **10** and 30 mL of ethanol/water (V/V = 2:1) was heated under reflux for 6 hours and then allowed to cool room temperature. After evaporation of ethanol, HCl (20%) was added until the mixture was acidified (pH 1). The aqueous layer was extracted with ethyl acetate (3×10 mL). The combined extracts were washed with a saturated solution of NaCl (20 mL), dried over Na_2SO_4, and evaporated in vacuo. The residue was purified by column chromatography (eluant: DCM/MeOH (V/V) = 15:1) to give the product **8** (a white solid, 1.27 g, 90% yield). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.13 (s, 1H), 8.01 (d, *J* = 7.4 Hz, 2H), 7.59 (t, *J* = 7.4 Hz, 1H), 7.48 (t, *J* = 7.6 Hz, 2H), 7.36-7.30 (m, 5H), 4.21 (s, 2H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ 197.40, 172.64, 144.64, 136.75, 135.23, 133.64, 129.38, 129.17, 128.95, 128.59, 126.50, 38.15 ppm.

General procedure for the asymmetric hydrogenation

In an argon-filled glove box, $[Rh(NBD)_2]BF_4$ (0.01 mmol) and *t*-Bu-Wudaphos (0.011 mmol) were dissolved in THF (1.0 mL) and stirred for 45 min. 0.1 mL of the resulting solution was transferred by syringe into the vials charged with different substrates (0.1 mmol for each). Additional THF was added to bring the total reaction volume to 1.0 mL. The vials were subsequently transferred into an autoclave which was charged with hydrogen (10 bar). The reaction was then stirred at rt for 12 h. The hydrogen gas was released slowly and carefully in a well-ventilated hood. The solution was passed through a short column of silica gel (eluent: EtOAc) to remove the metal complex and concentrated to give compounds **3**. The ee values of compounds **3** were then determined by HPLC analysis on a chiral stationary phase.

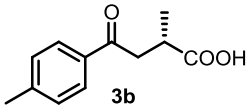
The following products can be obtained following the procedures for asymmetric hydrogenation.



3a

> 99% Conv.
98% ee

>99% conv., 98% *ee*, white solid; $[\alpha]_D^{20}$ = −32.9 (c 0.240, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.98-7.96 (m, 2H), 7.59-7.55 (m, 1H), 7.47 (t, *J* = 8.0 Hz, 2H), 3.48 (dd, *J* = 17.6, 7.6 Hz, 1H), 3.21-3.12 (m, 1H), 3.06 (dd, *J* = 17.6, 5.2 Hz, 1H), 1.32 (d, *J* = 7.2 Hz, 3H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$): δ 198.16, 182.28, 136.71, 133.59, 128.90, 128.32, 41.97, 35.06, 17.36 ppm. The enantiomeric excess of **3a** was determined by chiral HPLC analysis on Chiralpak OJ-H column. Conditions: hexane/isopropanol = 95 :5, flow rate = 1.0 mL/min, uv-vis detection at λ = 230 nm, t_R = 17.9 min (major), 22.6 min (minor).

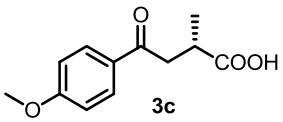


3b

> 99% Conv.
98% ee

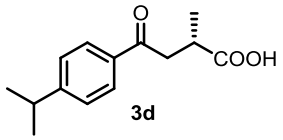
>99% conv., 98% *ee*, white solid; $[\alpha]_D^{20}$ = −42.6 (c 0.385, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.87 (d, *J* = 8.0 Hz, 2H), 7.26 (d, *J* = 8.0 Hz, 2H), 3.45 (dd, *J* =

18.0, 7.6 Hz, 1H), 3.19 – 3.11 (m, 1H), 3.04 (dd, *J* = 17.6, 5.6 Hz, 1H), 2.42 (s, 3H), 1.31 (d, *J* = 6.8 Hz, 3H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$): δ 197.87, 182.20, 144.41, 134.27, 129.56, 128.45, 41.88, 35.12, 21.94, 17.37 ppm. The enantiomeric excess of **3b** was determined by chiral HPLC analysis on Chiralpak OJ-H column. Conditions: hexane/isopropanol = 95 :5, flow rate = 1.0 mL/min, uv-vis detection at λ = 254 nm, t_R = 17.0 min (major), 22.8 min (minor).



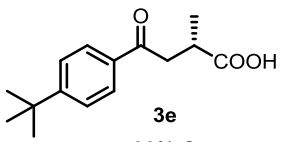
3c
> 99% Conv.
> 99% ee

>99% conv., > 99% *ee*, white solid; $[\alpha]_D^{20}$ = −33.9 (c 0.440, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.95 (d, *J* = 8.9 Hz, 2H), 6.93 (d, *J* = 8.9 Hz, 2H), 3.87 (s, 3H), 3.42 (dd, *J* = 17.5, 7.7 Hz, 1H), 3.19 – 3.09 (m, 1H), 3.02 (dd, *J* = 17.6, 5.5 Hz, 1H), 1.31 (d, *J* = 7.1 Hz, 3H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$): δ 196.74, 182.19, 163.89, 130.62, 129.83, 114.01, 55.76, 41.63, 35.13, 17.38 ppm. The enantiomeric excess of **3c** was determined by chiral HPLC analysis on Chiralpak OJ-H column. Conditions: hexane/isopropanol = 95 :5, flow rate = 1.0 mL/min, uv-vis detection at λ = 254 nm, t_R = 46.3 min (major), 60.4 min (minor).



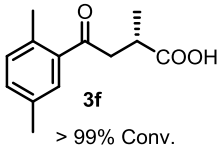
3d
> 99% Conv.
98% ee

>99% conv., 98% *ee*, white solid; $[\alpha]_D^{20}$ = −39.3 (c 0.450, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.90 (d, *J* = 8.3 Hz, 2H), 7.31 (d, *J* = 8.2 Hz, 2H), 3.45 (dd, *J* = 17.6, 7.5 Hz, 1H), 3.21 – 3.09 (m, 1H), 3.09 – 2.99 (m, 1H), 2.98-2.91 (m, 1H), 1.30 (d, *J* = 7.1 Hz, 3H), 1.26 (d, *J* = 6.9 Hz, 6H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$): δ 197.86, 182.34, 155.11, 134.63, 128.60, 126.97, 41.89, 35.13, 34.52, 23.93, 17.35 ppm. The enantiomeric excess of **3d** was determined by chiral HPLC analysis on Chiralpak OJ-H column. Conditions: hexane/isopropanol = 95 :5, flow rate = 1.0 mL/min, uv-vis detection at λ = 254 nm, t_R = 12.9 min (major), 16.9 min (minor).



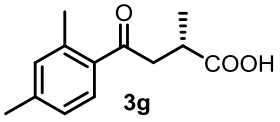
3e
> 99% Conv.
97% ee

>99% conv., 97% *ee*, white solid; $[\alpha]_D^{20}$ = −36.2 (c 0.425, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.92 (d, *J* = 8.5 Hz, 2H), 7.48 (d, *J* = 8.5 Hz, 2H), 3.46 (dd, *J* = 17.6, 7.5 Hz, 1H), 3.22 – 3.10 (m, 1H), 3.04 (dd, *J* = 17.6, 5.7 Hz, 1H), 1.34 (s, 9H), 1.31 (d, *J* = 7.1 Hz, 3H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$): δ 197.88, 182.45, 157.30, 134.19, 128.31, 125.83, 41.89, 35.38, 35.15, 31.33, 17.35 ppm. The enantiomeric excess of **3e** was determined by chiral HPLC analysis on Chiralpak OJ-H column. Conditions: hexane/isopropanol = 95 :5, flow rate = 1.0 mL/min, uv-vis detection at λ = 230 nm, t_R = 10.3 min (major), 15.2 min (minor).



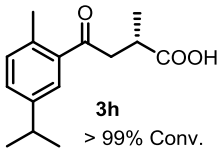
3f
> 99% Conv.
98% ee

>99% conv., 98% *ee*, white solid; $[\alpha]_D^{20}$ = −31.5 (c 0.425, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.47 (s, 1H), 7.20 – 7.10 (m, 2H), 3.39 (dd, *J* = 17.8, 8.0 Hz, 1H), 3.18 – 3.08 (m, 1H), 2.96 (dd, *J* = 17.8, 5.4 Hz, 1H), 2.43 (s, 3H), 2.36 (s, 3H), 1.30 (d, *J* = 7.2 Hz, 3H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$): δ 202.21, 182.31, 137.44, 135.46, 132.50, 132.17, 129.41, 44.76, 35.32, 21.19, 21.14, 17.30 ppm. The enantiomeric excess of **3f** was determined by chiral HPLC analysis on Chiralpak OJ-H column. Conditions: hexane/isopropanol = 95 :5, flow rate = 1.0 mL/min, uv-vis detection at λ = 254 nm, t_R = 10.9 min (major), 16.7 min (minor).



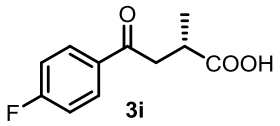
3g
> 99% Conv.
> 99% ee

>99% conv., > 99% *ee*, white solid; $[\alpha]_D^{20}$ = −34.2 (c 0.503, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.63 (d, *J* = 8.5 Hz, 1H), 7.06 (d, *J* = 7.1 Hz, 2H), 3.38 (dd, *J* = 17.6, 8.0 Hz, 1H), 3.16 – 3.06 (m, 1H), 2.96 (dd, *J* = 17.6, 5.4 Hz, 1H), 2.48 (s, 3H), 2.35 (s, 3H), 1.29 (d, *J* = 7.2 Hz, 3H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$) δ 201.32, 182.32, 142.48, 139.22, 134.47, 133.20, 129.44, 126.59, 44.44, 35.39, 21.90, 21.65, 17.31 ppm. The enantiomeric excess of **3g** was determined by chiral HPLC analysis on Chiralpak OJ-H column. Conditions: hexane/isopropanol = 95 :5, flow rate = 1.0 mL/min, uv-vis detection at λ = 254 nm, t_R = 14.5 min (major), 26.2 min (minor).



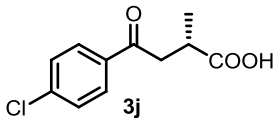
3h
> 99% Conv.
91% ee

>99% conv., 91% *ee*, white solid; $[\alpha]_D^{20}$ = −35.1 (c 0.305, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.50 (d, *J* = 1.6 Hz, 1H), 7.27 – 7.14 (m, 2H), 3.40 (dd, *J* = 17.7, 7.9 Hz, 1H), 3.19 – 3.09 (m, 1H), 3.03 – 2.86 (m, 2H), 2.44 (s, 3H), 1.31 (d, *J* = 7.2 Hz, 3H), 1.26 (d, *J* = 6.9 Hz, 6H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$): δ 202.33, 182.41, 146.59, 137.59, 135.84, 132.26, 129.79, 126.89, 44.80, 35.35, 33.96, 24.25, 21.15, 17.29 ppm. The enantiomeric excess of **3h** was determined by chiral HPLC analysis on Chiralpak OJ-H column. Conditions: hexane/isopropanol = 95 :5, flow rate = 1.0 mL/min, uv-vis detection at λ = 230 nm, t_R = 6.8 min (major), 7.9 min (minor).



3i
> 99% Conv.
98% ee

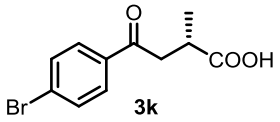
>99% conv., 98% *ee*, white solid; $[\alpha]_D^{20}$ = −36.9 (c 0.325, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.02 – 7.97 (m, 2H), 7.16 – 7.10 (m, 2H), 3.44 (dd, *J* = 17.8, 7.9 Hz, 1H), 3.19 – 3.10 (m, 1H), 3.01 (dd, *J* = 17.8, 5.3 Hz, 1H), 1.32 (d, *J* = 7.2 Hz, 3H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$): δ 196.56, 182.33, 166.12 (d, *J* = 255.0 Hz), 133.16 (d, *J* = 3.0 Hz), 130.97 (d, *J* = 9.4 Hz), 116.01 (d, *J* = 21.9 Hz), 41.86, 35.09, 17.37 ppm. The enantiomeric excess of **3i** was determined by chiral HPLC analysis on Chiralpak OJ-H column. Conditions: hexane/isopropanol = 95 :5, flow rate = 1.0 mL/min, uv-vis detection at λ = 210 nm, t_R = 19.5 min (major), 22.6 min (minor).



3j
> 99% Conv
98% ee

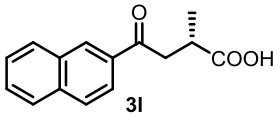
>99% conv., 98% *ee*, white solid; $[\alpha]_D^{20}$ = −41.0 (c 0.305, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.90 (d, *J* = 8.6 Hz, 2H), 7.43 (d, *J* = 8.6 Hz, 2H), 3.44 (dd, *J* = 17.8, 7.9 Hz, 1H), 3.19 – 3.09 (m, 1H), 3.00 (dd, *J* = 17.8, 5.2 Hz, 1H), 1.31 (d, *J* = 7.2 Hz, 3H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$): δ 196.98, 182.21, 140.03, 135.02,

129.74, 129.22, 41.92, 35.07, 17.37 ppm. The enantiomeric excess of **3j** was determined by chiral HPLC analysis on Chiralpak OJ-H column. Conditions: hexane/isopropanol = 95 :5, flow rate = 1.0 mL/min, uv-vis detection at λ = 254 nm, t_R = 19.3 min (major), 26.0 min (minor).



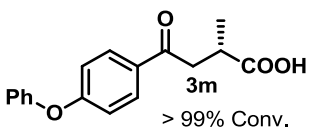
3k
> 99% Conv.
96% ee

>99% conv., 96% *ee*, white solid; $[\alpha]_D^{20}$ = −35.0 (c 0.400, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.76 (d, *J* = 8.6 Hz, 2H), 7.54 (d, *J* = 8.6 Hz, 2H), 3.36 (dd, *J* = 17.8, 8.0 Hz, 1H), 3.13 – 3.03 (m, 1H), 2.93 (dd, *J* = 17.8, 5.2 Hz, 1H), 1.25 (d, *J* = 7.2 Hz, 3H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$): δ 197.15, 181.87, 135.43, 132.23, 129.85, 128.80, 41.90, 34.99, 17.38 ppm. The enantiomeric excess of **3k** was determined by chiral HPLC analysis on Chiralpak OJ-H column. Conditions: hexane/isopropanol = 95 :5, flow rate = 1.0 mL/min, uv-vis detection at λ = 254 nm, t_R = 22.6 min (major), 32.2 min (minor).



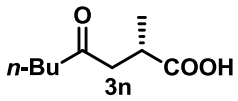
3l
> 99% Conv.
98% ee

>99% conv., 98% *ee*, white solid; $[\alpha]_D^{20}$ = −61.7 (c 0.360, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.43 (s, 1H), 7.96 (dd, *J* = 8.6, 1.5 Hz, 1H), 7.89 (d, *J* = 8.0 Hz, 1H), 7.81 (t, *J* = 7.9 Hz, 2H), 7.57 – 7.45 (m, 2H), 3.60 – 3.50 (m, 1H), 3.19 – 3.10 (m, 2H), 1.30 (d, *J* = 7.0 Hz, 3H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$): δ 198.13, 181.67, 135.95, 134.08, 132.73, 130.13, 129.86, 128.84, 128.78, 128.06, 127.10, 123.99, 42.07, 35.13, 17.46 ppm. The enantiomeric excess of **3l** was determined by chiral HPLC analysis on Chiralpak OJ-H column. Conditions: hexane/isopropanol = 95 :5, flow rate = 1.0 mL/min, uv-vis detection at λ = 230 nm, t_R = 42. 2 min (major), 67.0 min (minor).



3m
> 99% Conv.
96% ee

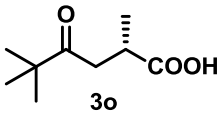
>99% conv., 96% *ee*, white solid; $[\alpha]_D^{20}$ = -6.8 (c 0.340, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.98–7.91 (m, 2H), 7.44–7.36 (m, 2H), 7.24–7.17 (m, 1H), 7.07 (dt, *J* = 9.0, 1.8 Hz, 2H), 7.03–6.97 (m, 2H), 3.43 (dd, *J* = 17.7, 7.8 Hz, 1H), 3.24–3.08 (m, 1H), 3.01 (dd, *J* = 17.7, 5.4 Hz, 1H), 1.31 (d, *J* = 7.2 Hz, 3H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ 196.74, 181.87, 162.43, 155.65, 131.40, 130.63, 130.34, 124.93, 120.47, 117.57, 41.77, 35.09, 17.40 ppm. The enantiomeric excess of **3m** was determined by chiral HPLC analysis on Chiralpak AD-H column. Conditions: hexane/isopropanol = 95 :5, flow rate = 1.0 mL/min, uv-vis detection at λ = 210 nm, t_R = 41.0 min (minor), 45.6 min (major).



3n

> 99% Conv.
93% ee

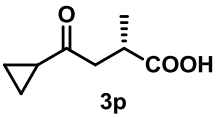
>99% conv., 93% *ee*, white solid; $[\alpha]_D^{20}$ = −6.6 (c 0.52, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.01 – 2.85 (m, 2H), 2.47 – 2.41 (m, 3H), 1.61 – 1.53 (m, 2H), 1.35 –1.29 (m, 2H), 1.22 (d, *J* = 7.1 Hz, 3H), 0.91 (t, *J* = 7.3 Hz, 3H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$): δ 209.50, 182.08, 45.68, 42.86, 34.81, 26.06, 22.54, 17.16, 14.11 ppm. The enantiomeric excess of **3n** was determined by chiral HPLC analysis on Chiralpak OJ-H column after esterification with BnOH in the presence of DCC and DMAP. Conditions: hexane/isopropanol = 99 :1, flow rate = 1.0 mL/min, uv-vis detection at λ = 210 nm, t_R = 18.0 min (minor), 19.8 min (major).



3o

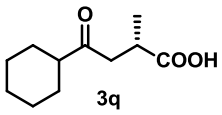
> 99% Conv.
95% ee

>99% conv., 95% *ee*, colorless oil; $[\alpha]_D^{20}$ = −11.5 (c 0.900, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.99 – 2.93 (m, 2H), 2.62 – 2.55 (m, 1H), 1.21 (d, *J* = 6.7 Hz, 3H), 1.15 (s, 9H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$): δ 214.30, 182.32, 44.17, 40.29, 34.90, 26.64, 17.17 ppm. The enantiomeric excess of **3o** was determined by chiral HPLC analysis on Chiralpak OJ-H column after esterification with BnOH in the presence of DCC and DMAP. Conditions: hexane/isopropanol = 99 :1, flow rate = 1.0 mL/min, uv-vis detection at λ = 210 nm, t_R = 10.2 min (minor), 11.8 min (major).



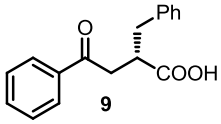
3p
> 99% Conv.
96% ee

>99% conv., 96% *ee*, colorless oil; $[\alpha]_D^{20}$ = −13.5 (c 0.635, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.08 – 2.95 (m, 2H), 2.67 (dd, *J* = 17.2, 5.0 Hz, 1H), 1.96 – 1.90 (m, 1H), 1.23 (d, *J* = 7.0 Hz, 3H), 1.05 (dd, *J* = 4.5, 2.9 Hz, 2H), 0.92 – 0.88 (m, 2H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$): δ 209.19, 181.60, 46.38, 34.84, 20.90, 17.18, 11.23, 11.16 ppm. The enantiomeric excess of **3p** was determined by chiral HPLC analysis on Chiralpak OJ-H column after esterification with BnOH in the presence of DCC and DMAP. Conditions: hexane/isopropanol = 99 :1, flow rate = 1.0 mL/min, uv-vis detection at λ = 210 nm, t_R = 25.1 min (minor), 32.2 min (major).



3q
> 99% Conv.
95% ee

>99% conv., 95% *ee*, colorless oil; $[\alpha]_D^{20}$ = −11.9 (c 0.655, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.99 – 2.89 (m, 2H), 2.52 (dd, *J* = 17.3, 4.8 Hz, 1H), 2.37 – 2.31 (m, 1H), 1.87 – 1.77 (m, 4H), 1.68 – 1.66 (m, 1H), 1.38 – 1.17 (m, 8H) ppm; ^{13}C NMR (101 MHz, $CDCl_3$): δ 212.34, 182.14, 51.00, 43.77, 34.74, 28.61, 28.58, 26.07, 25.89, 25.84, 17.20 ppm. The enantiomeric excess of **3q** was determined by chiral HPLC analysis on Chiralpak OJ-H column after esterification with BnOH in the presence of DCC and DMAP. Conditions: hexane/isopropanol = 99 :1, flow rate = 1.0 mL/min, uv-vis detection at λ = 205 nm, t_R = 15.3 min (minor), 19.7 min (major).

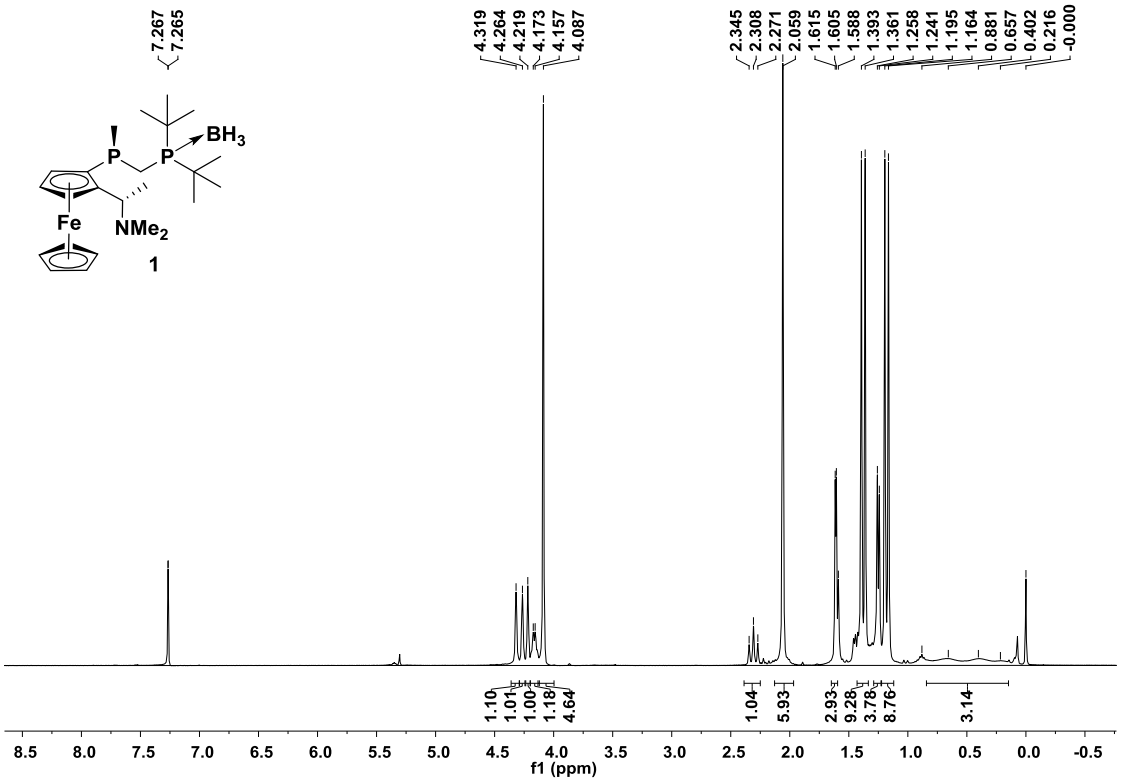


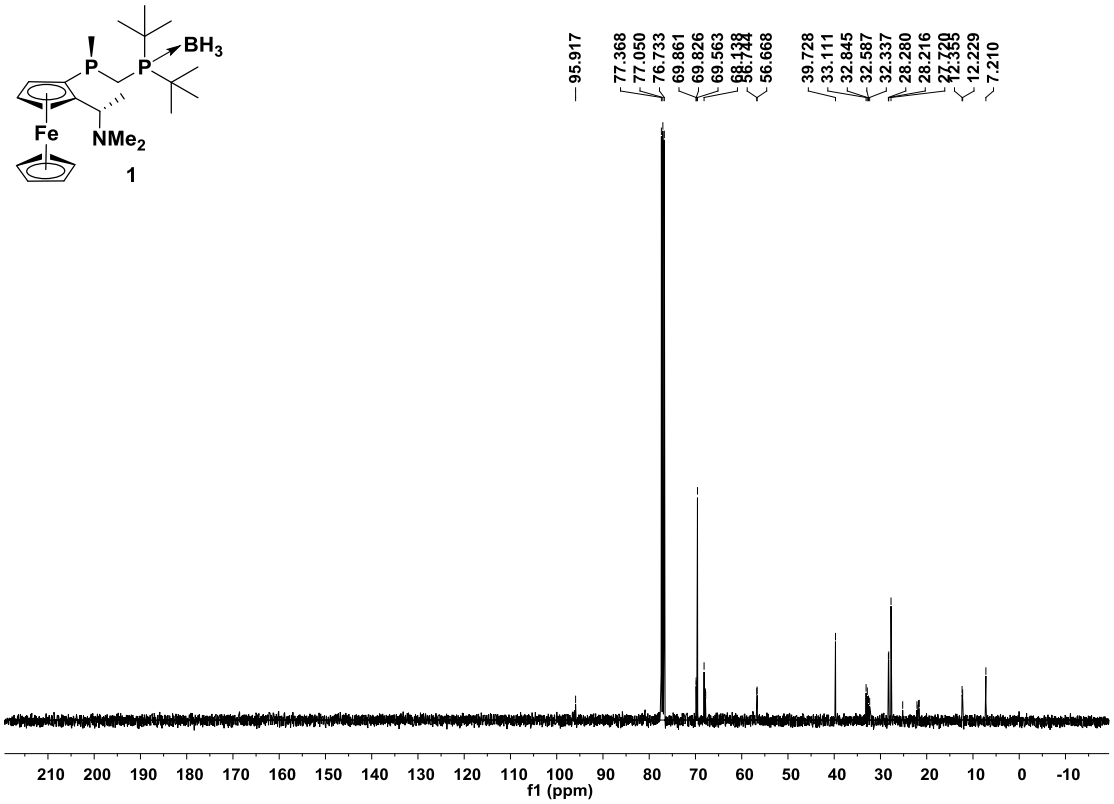
9
> 99% conv.
98% ee

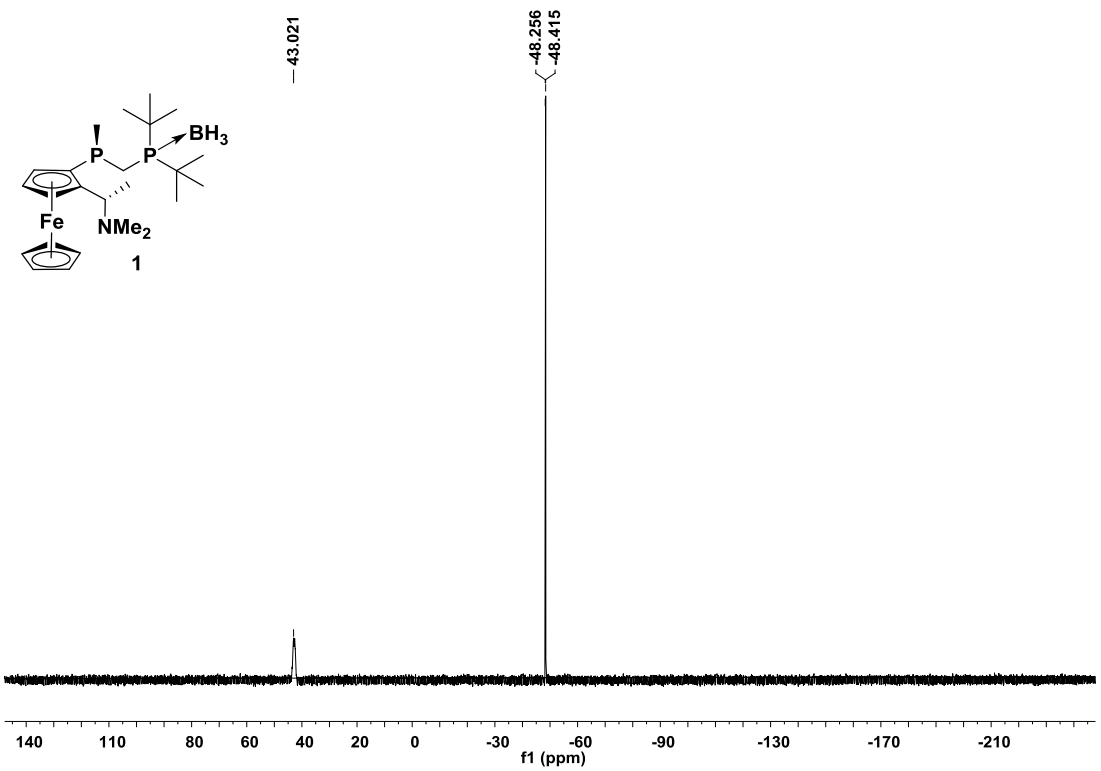
>99% conv., 98% *ee*, white solid; $[\alpha]_D^{20}$ = 3.7 (c 0.295, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.87 (d, *J* = 7.5 Hz, 2H), 7.53 (t, *J* = 7.3 Hz, 1H), 7.40 (t, *J* = 7.4 Hz, 2H), 7.33 – 7.26 (m, 2H), 7.21 (d, *J* = 7.3 Hz, 3H), 3.48 – 3.28 (m, 2H), 3.20 (dd, *J* = 13.7, 4.1 Hz, 1H), 2.98 (dd, *J* = 21.2, 7.6 Hz, 1H), 2.84 (dd, *J* = 13.3, 8.1 Hz, 1H)

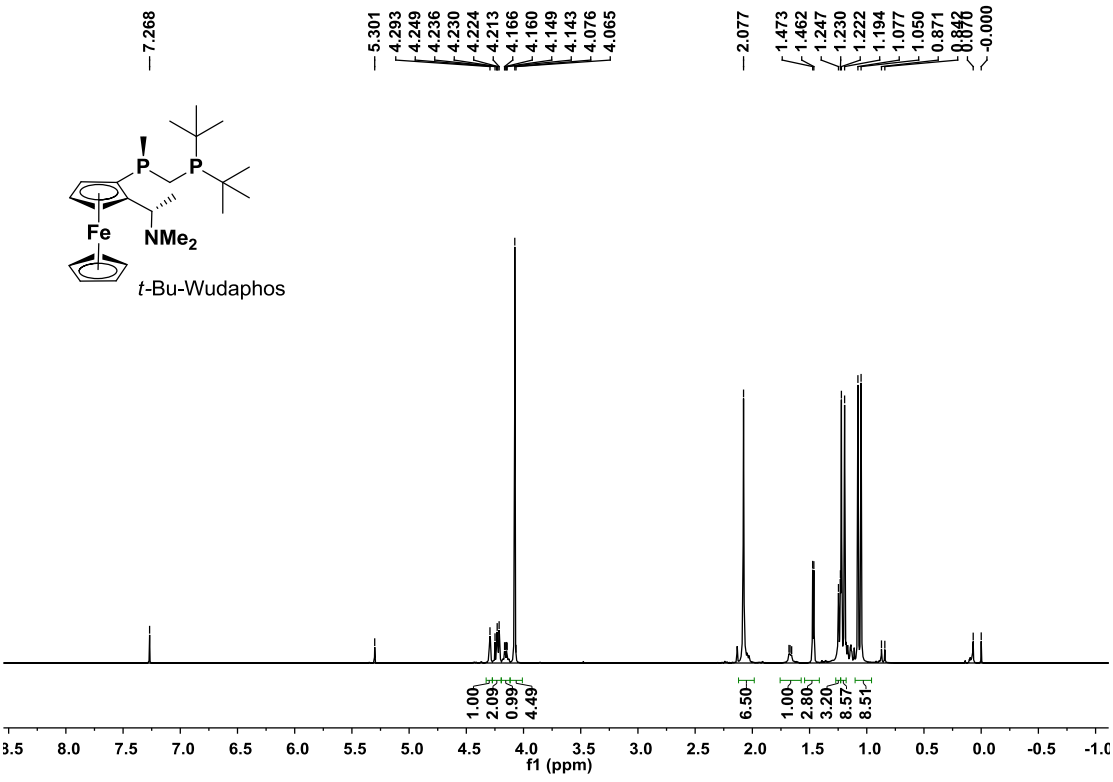
ppm. ^{13}C NMR (101 MHz, $CDCl_3$): δ 198.50, 180.81, 138.67, 136.65, 133.55, 129.35, 128.89, 128.82, 128.32, 126.97, 42.36, 39.21, 37.65 ppm. The enantiomeric excess of **9** was determined by chiral HPLC analysis on Chiralpak AD-H column. Conditions: hexane/isopropanol = 95 :5, flow rate = 1.0 mL/min, uv-vis detection at λ = 210 nm, t_R = 35.8 min (minor), 41.9 min (major).

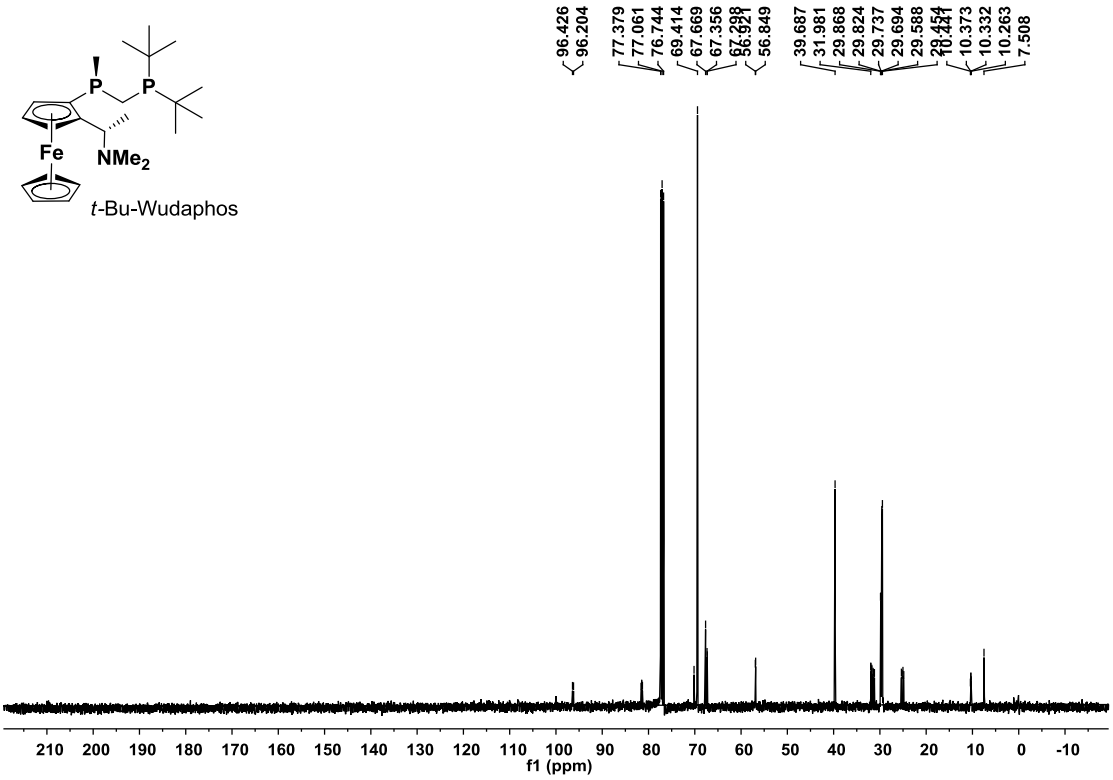
NMR spectra

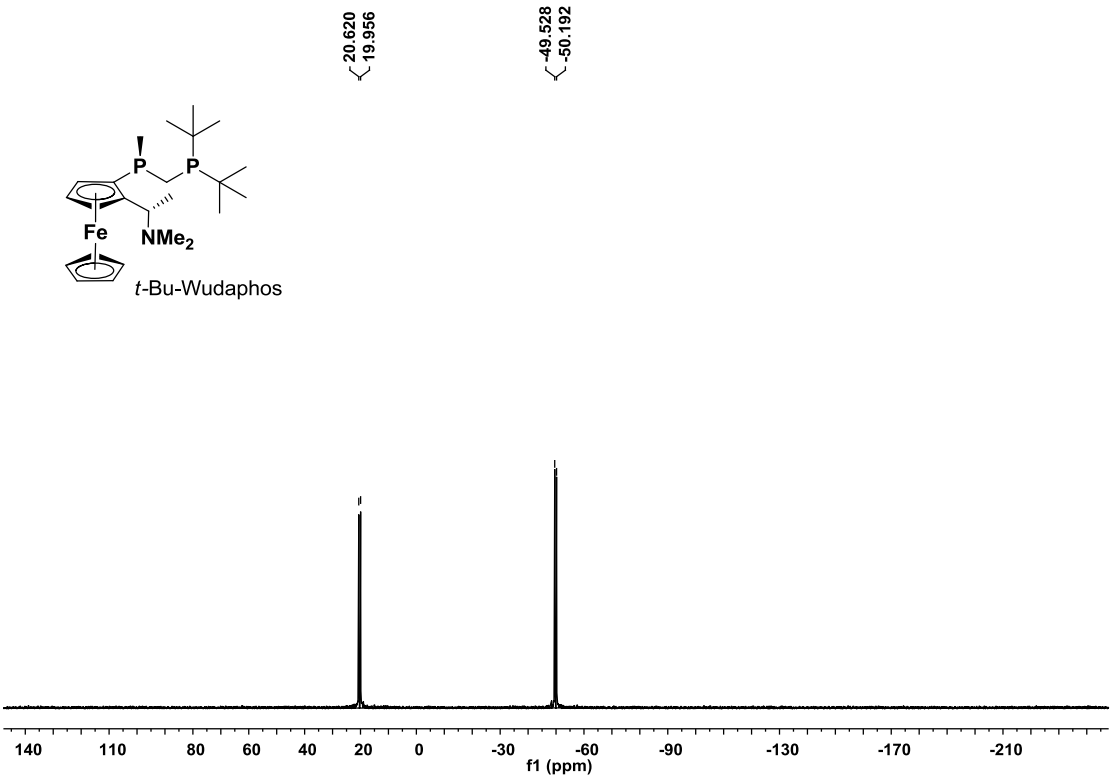

7.267
7.265
4.319
4.264
4.219
4.173
4.157
4.087
2.345
2.308
2.271
2.059
1.615
1.605
1.588
1.393
1.361
1.258
1.241
1.195
1.164
0.881
0.657
0.402
0.216
-0.000
BH3
P
P
Fe
NMe2
1
1.10
1.01
1.00
1.18
4.64
1.04
5.93
2.93
9.28
3.78
8.76
3.14
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

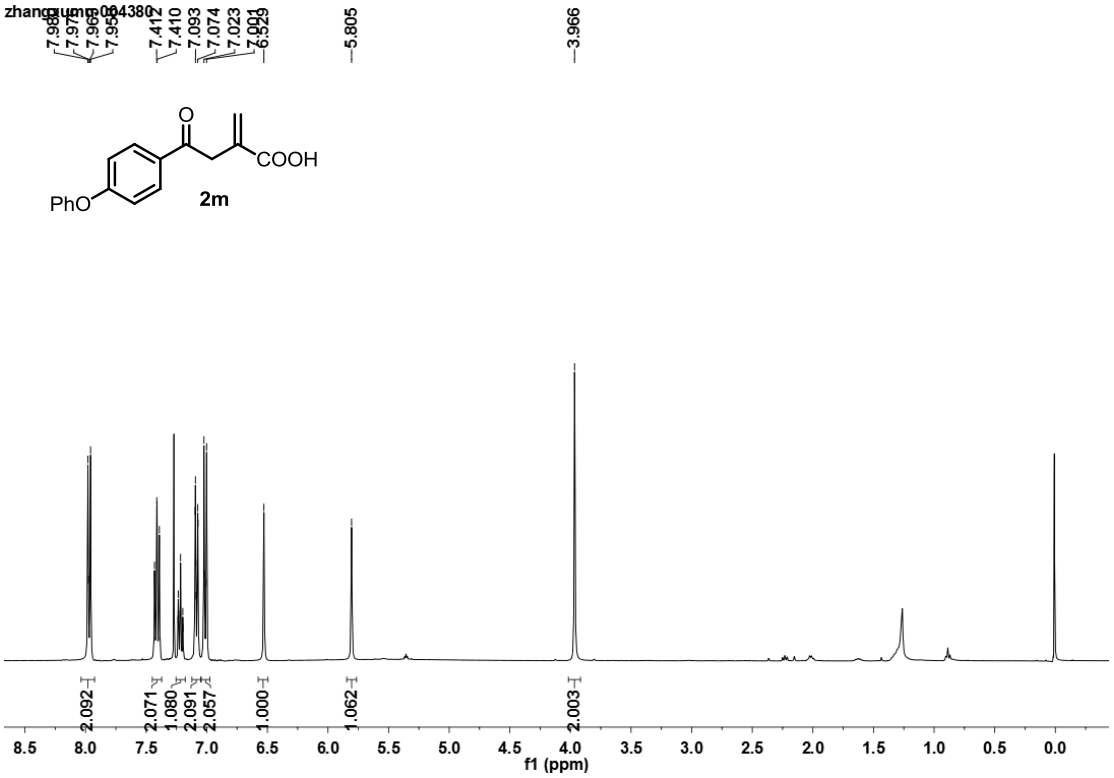

BH3
P
P
Fe
NMe2
1
95.917
77.368
77.050
76.733
69.861
69.826
69.563
56.668
39.728
33.111
32.845
32.587
32.337
28.280
28.216
12.355
12.229
7.210
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

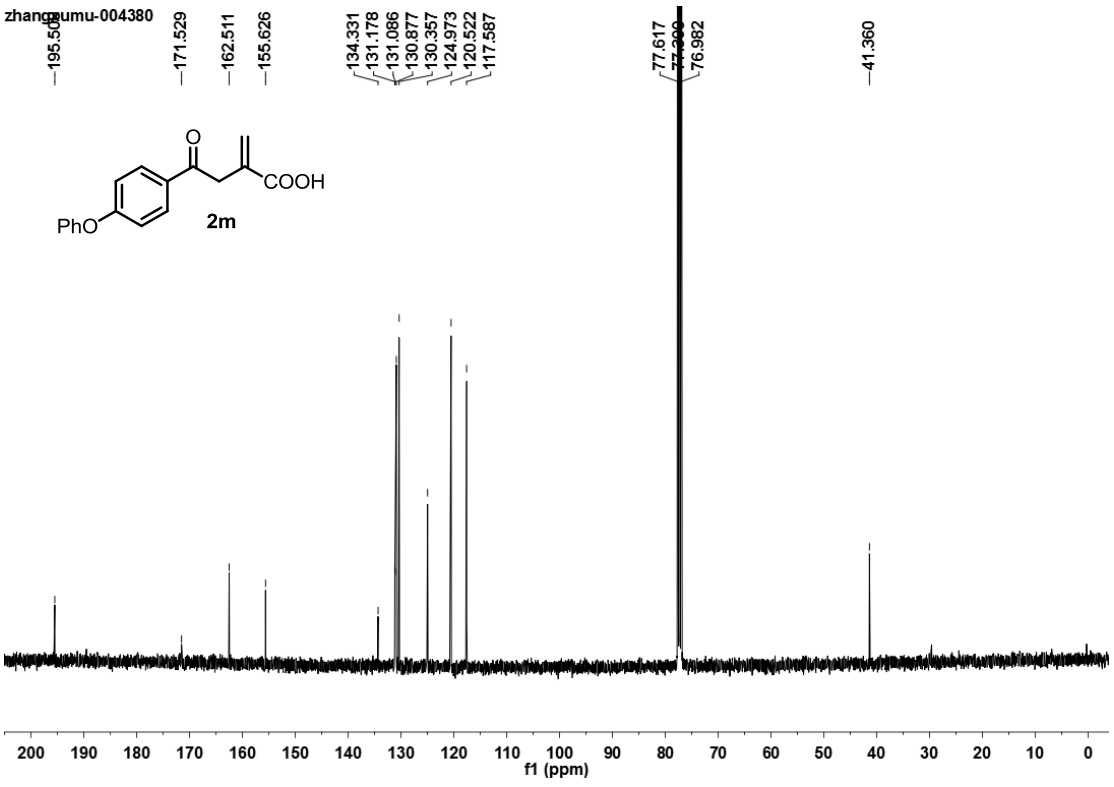

43.021
-48.256
-48.415
BH3
P
P
Fe
NMe2
1
140
110
80
60
40
20
0
-30
-60
-90
-130
-170
-210
f1 (ppm)

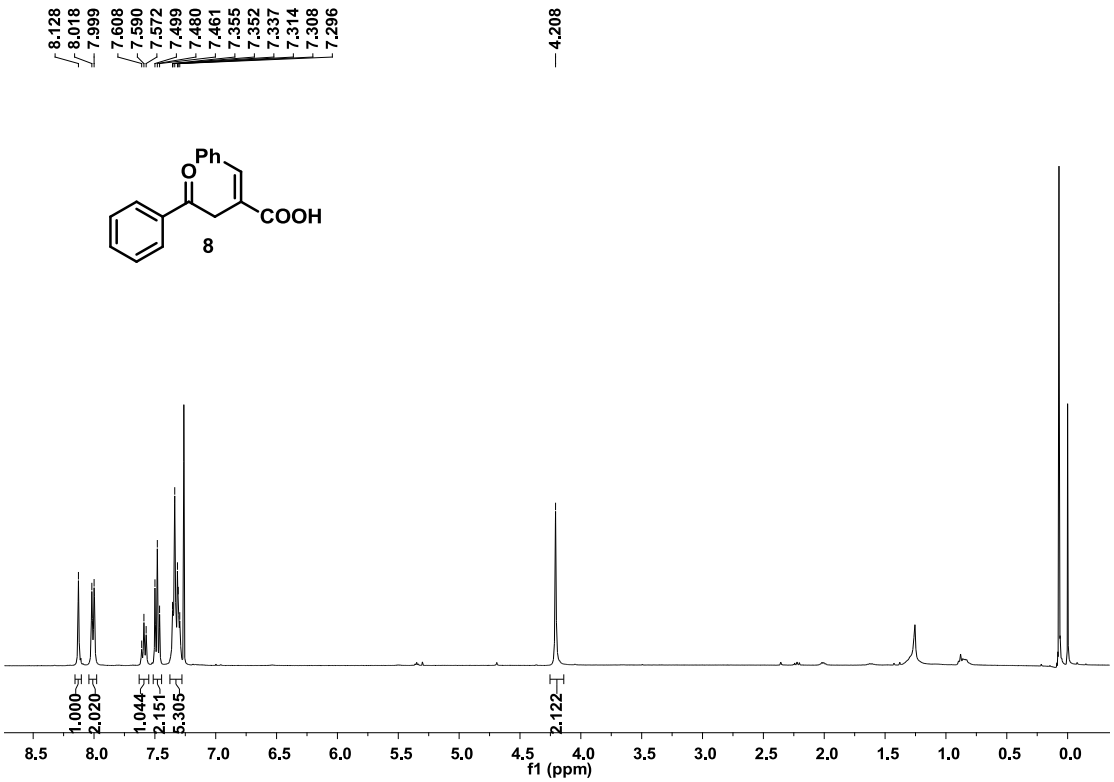

7.268
5.301
4.293
4.249
4.236
4.230
4.224
4.213
4.166
4.160
4.149
4.143
4.076
4.065
2.077
1.473
1.462
1.247
1.230
1.222
1.194
1.077
1.050
0.871
-0.000
P
P
Fe
NMe2
t-Bu-Wudaphos
1.00
2.09
0.99
4.49
6.50
1.00
2.80
3.20
8.57
8.51
f1 (ppm)

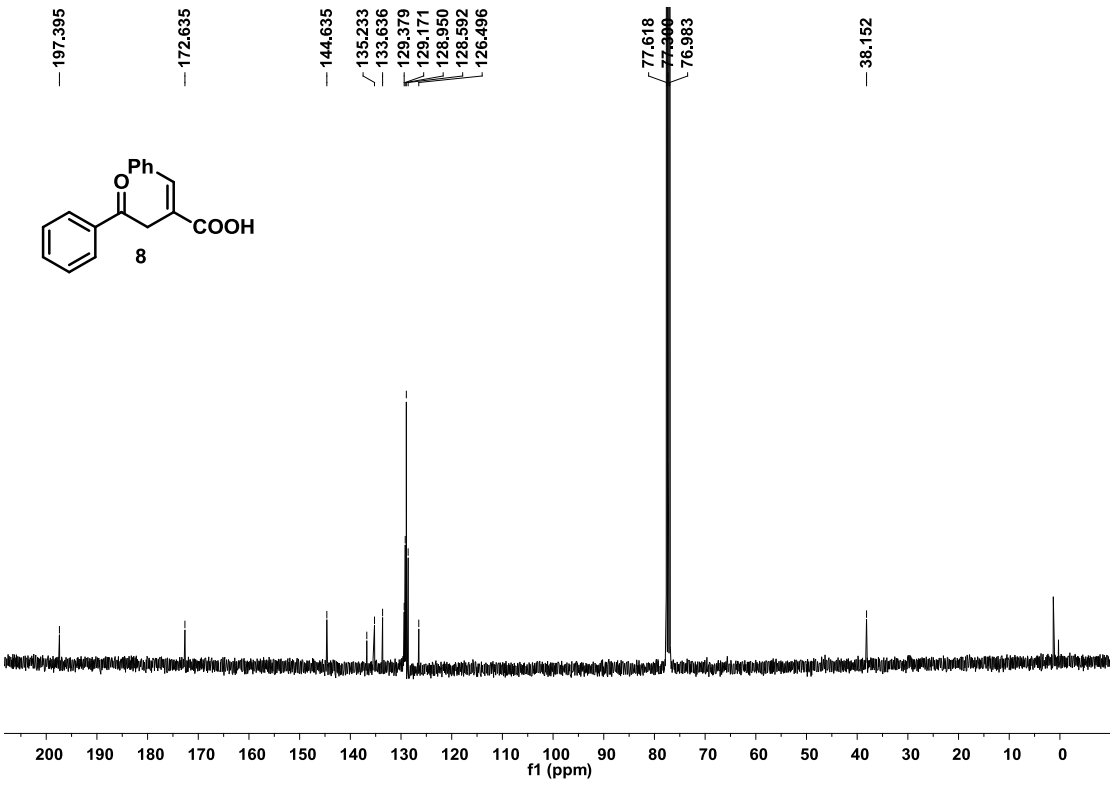

96.426
96.204
77.379
77.061
76.744
69.414
67.669
67.356
56.849
39.687
31.981
29.868
29.824
29.737
29.694
29.588
10.373
10.332
10.263
7.508
P
P
Fe
NMe2
t-Bu-Wudaphos
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

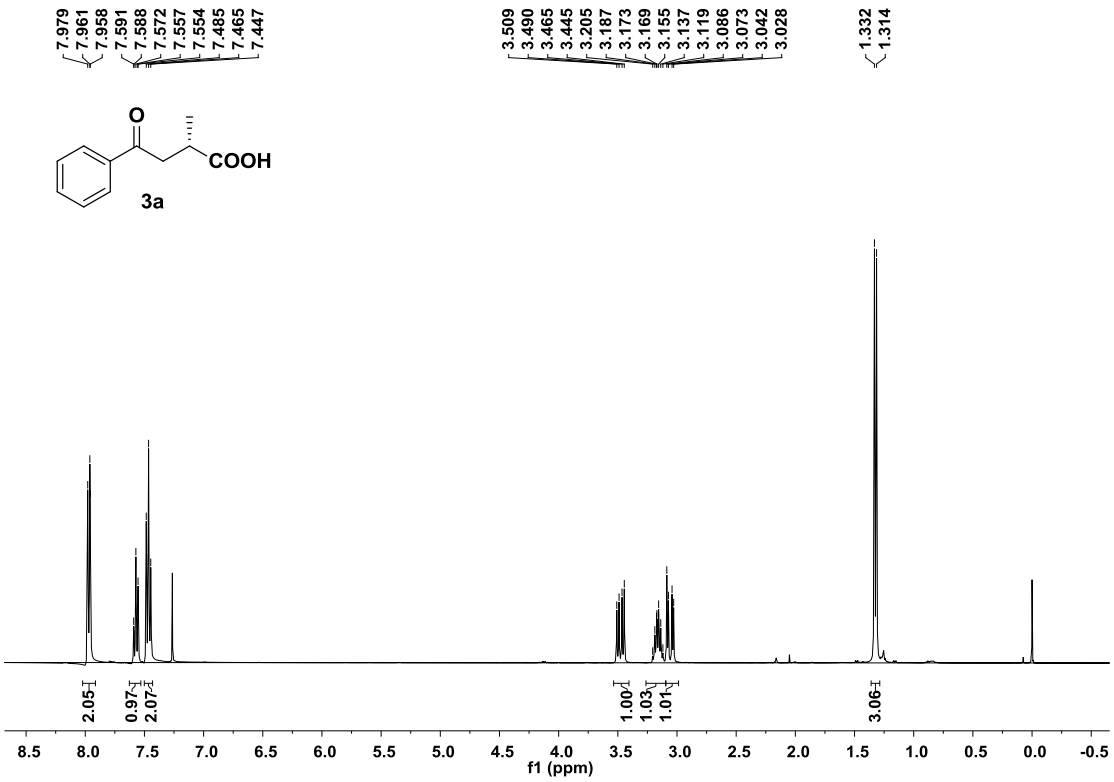

20.620
19.956
-49.528
-50.192
P
P
Fe
NMe2
t-Bu-Wudaphos
140 110 80 60 40 20 0 -30 -60 -90 -130 -170 -210
f1 (ppm)


zhangqumu-004380
5.805
3.966
O
COOH
PhO
2m
2.092
2.071
1.080
2.091
2.057
1.000
1.062
2.003
f1 (ppm)


zhangqumu-004380
171.529
162.511
155.626
134.331
131.178
131.086
130.877
130.357
124.973
120.522
117.587
77.617
76.982
41.360
O
COOH
PhO
2m
f1 (ppm)


8.128
8.018
7.999
7.608
7.590
7.572
7.499
7.480
7.461
7.355
7.352
7.337
7.314
7.308
7.296
4.208
Ph
O
COOH
8
1.000
2.020
1.044
2.151
5.305
2.122
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


197.395
172.635
144.635
135.233
133.636
129.379
129.171
128.950
128.592
126.496
77.618
77.300
76.983
38.152
Ph
O
COOH
8
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.979
7.961
7.958
7.591
7.588
7.572
7.557
7.554
7.485
7.465
7.447
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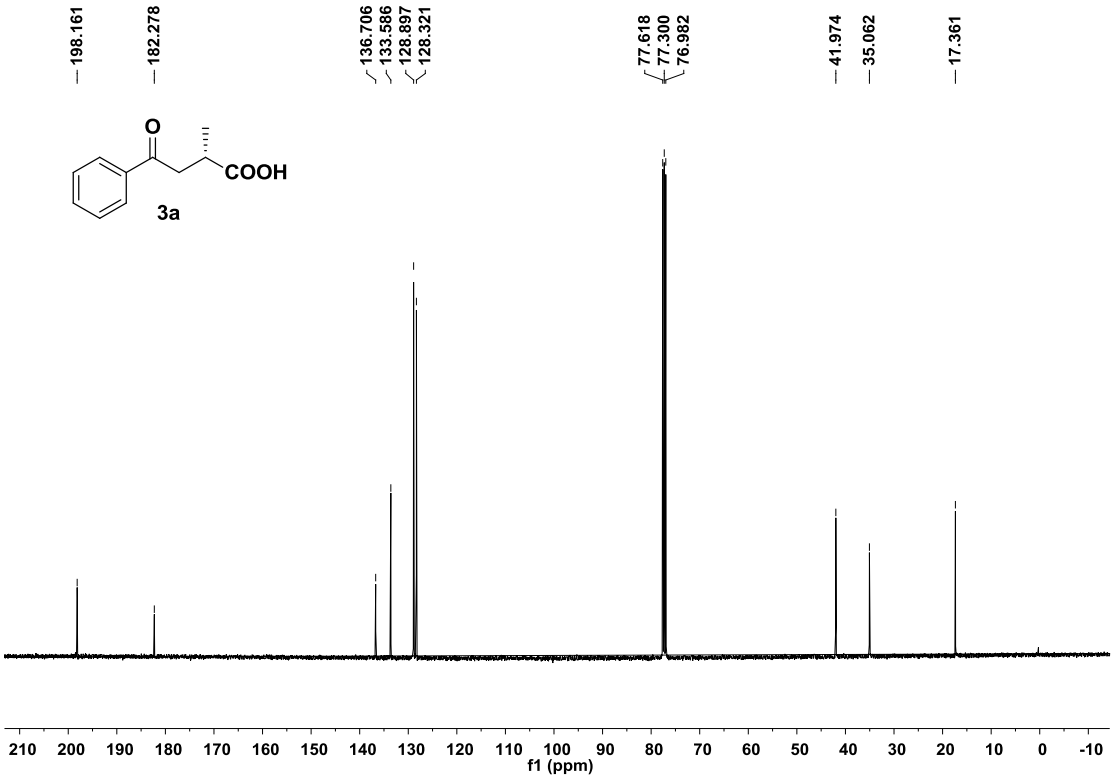
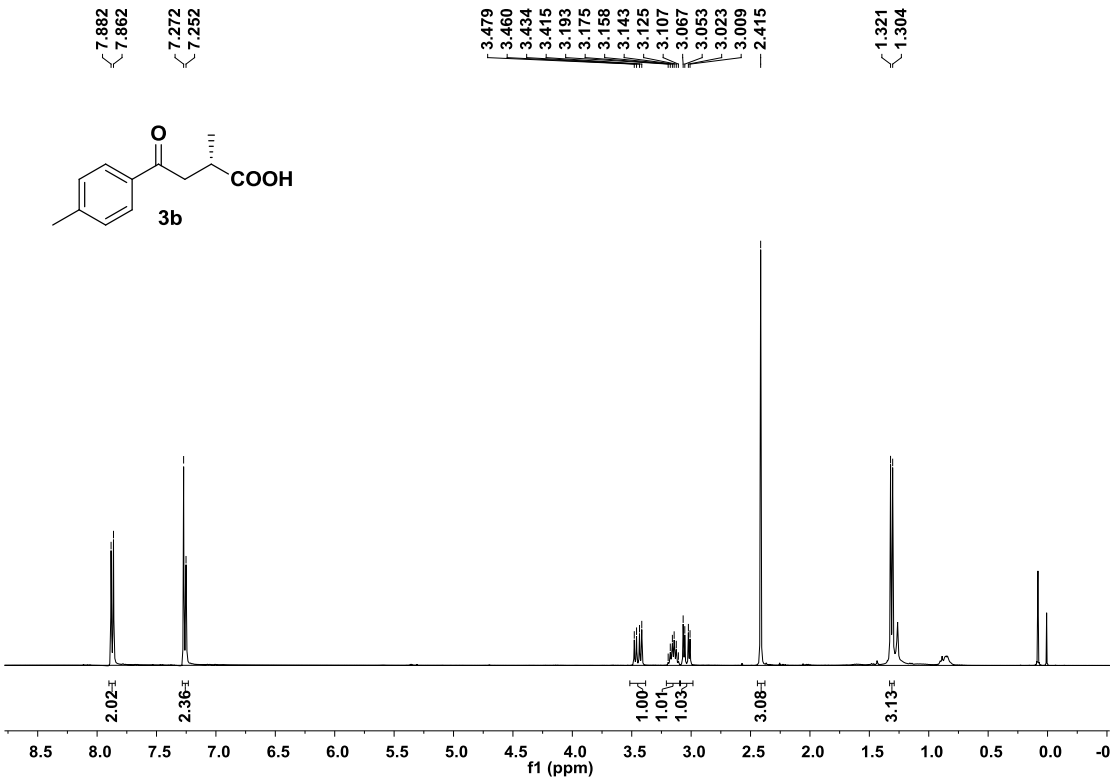
3.465
3.445
3.205
3.187
3.173
3.169
3.155
3.137
3.119
3.086
3.073
3.042
3.028
1.332
1.314
O
COOH
3a
2.05
0.97
2.07
1.00
1.03
1.01
3.06
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


198.161
182.278
136.706
133.586
128.897
128.321
77.618
77.300
76.982
41.974
35.062
17.361
O
COOH
3a
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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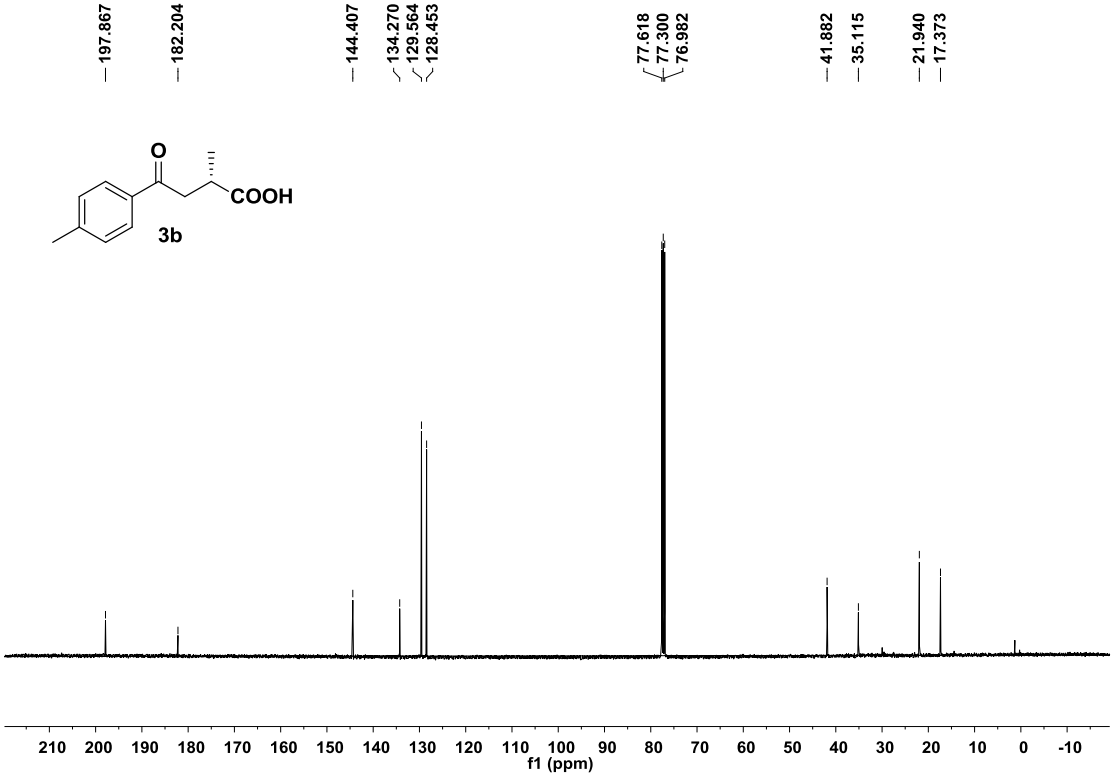
7.862
7.272
7.252
3.479
3.460
3.434
3.415
3.193
3.175
3.158
3.143
3.125
3.107
3.067
3.053
3.023
3.009
2.415
1.321
1.304
O
COOH
3b
2.02
2.36
1.00
1.01
1.03
3.08
3.13
f1 (ppm)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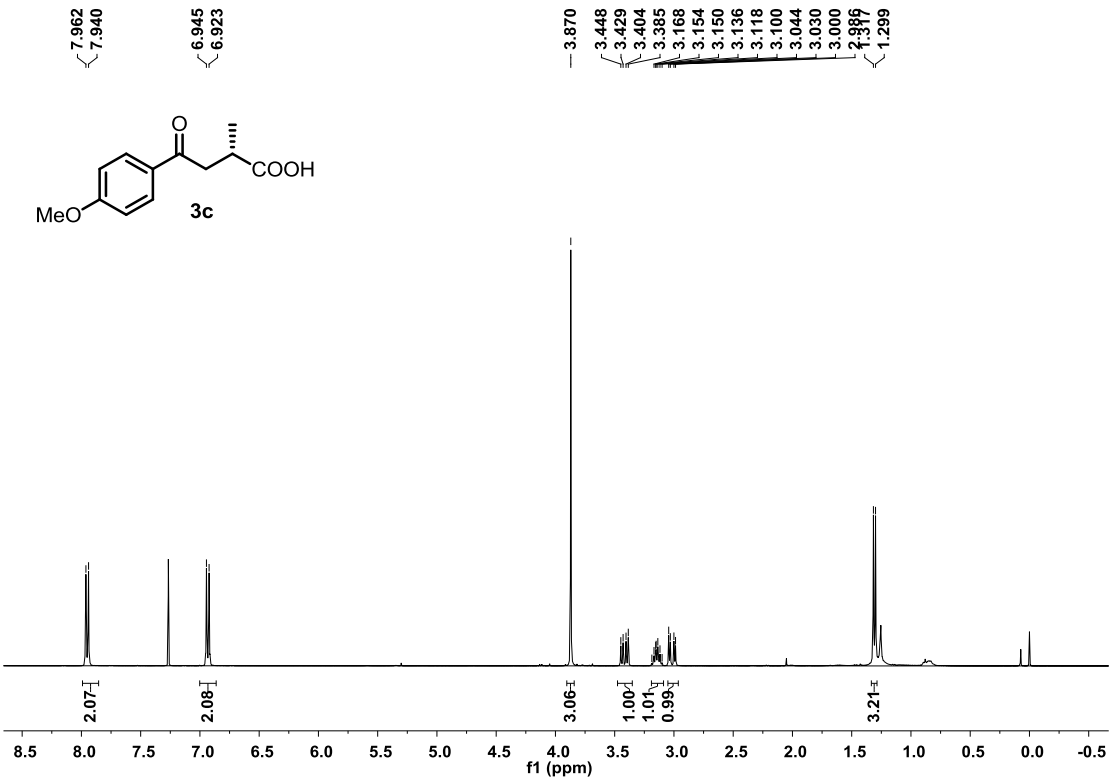
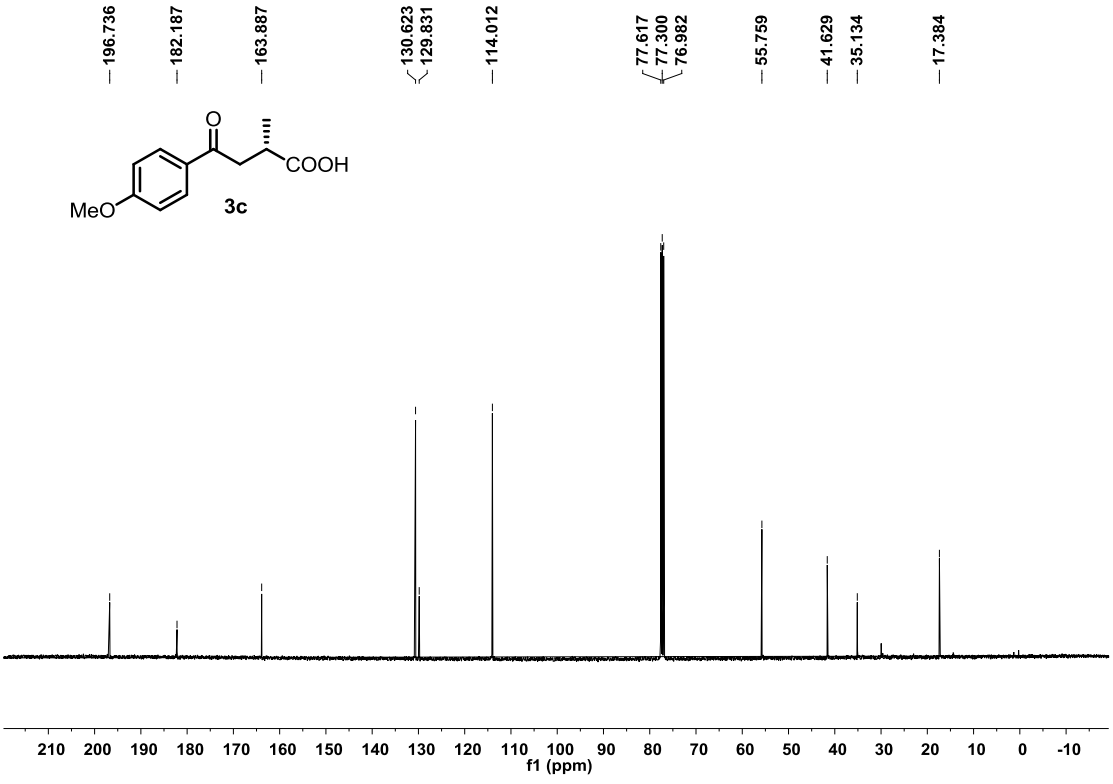
182.204
144.407
134.270
129.564
128.453
77.618
77.300
76.982
41.882
35.115
21.940
17.373
O
COOH
3b
f1 (ppm)


7.962
7.940
6.945
6.923
3.870
3.448
3.429
3.404
3.385
3.168
3.154
3.150
3.136
3.118
3.100
3.044
3.030
3.000
2.986
1.317
1.299
O
COOH
MeO
3c
2.07
2.08
3.06
1.00
1.01
0.99
3.21
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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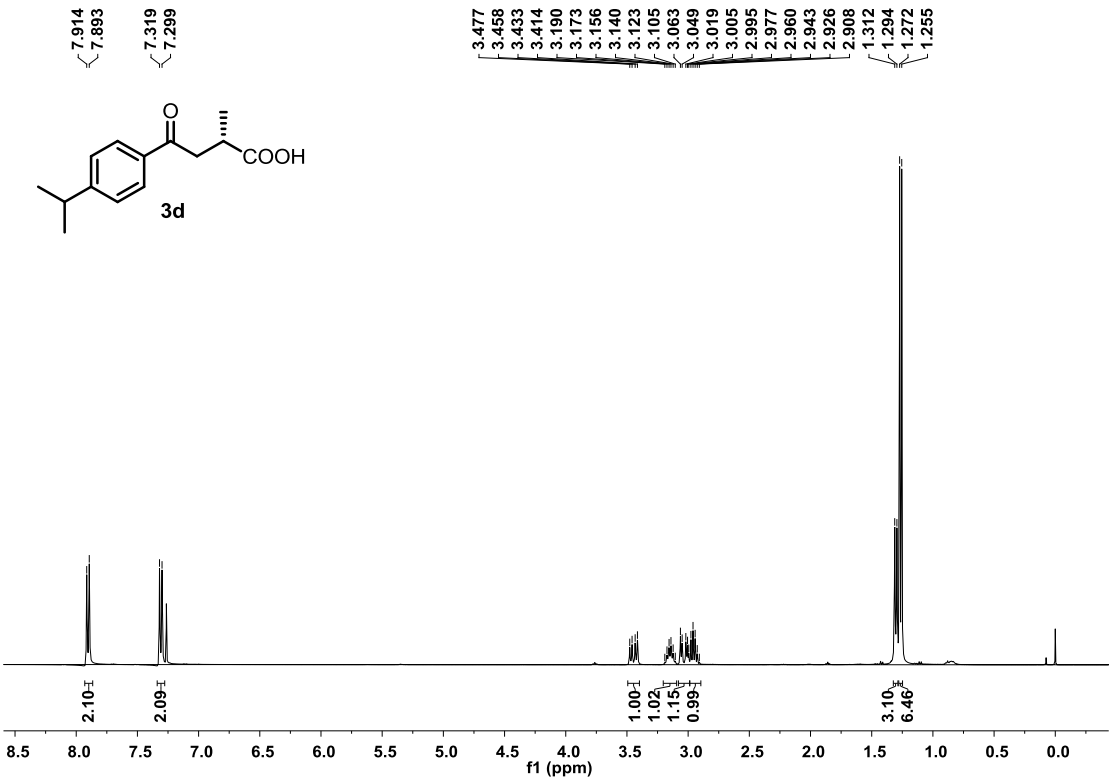
182.187
163.887
130.623
129.831
114.012
77.617
77.300
76.982
55.759
41.629
35.134
17.384
O
COOH
MeO
3c
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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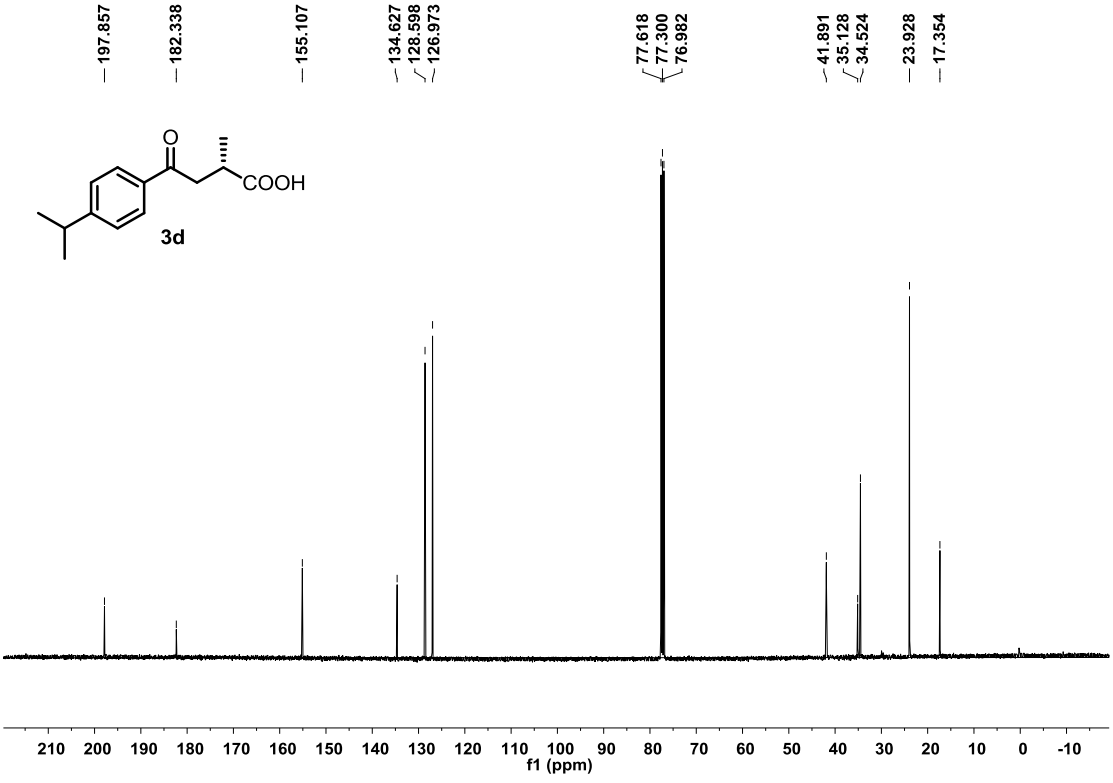
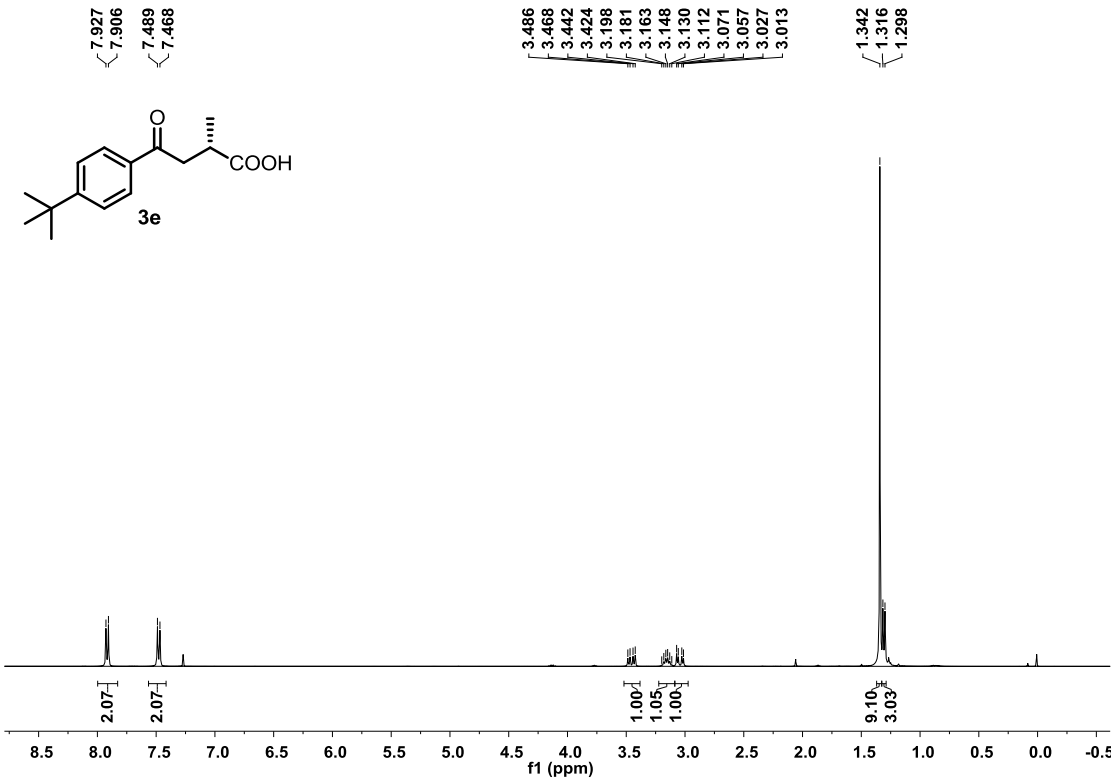
7.893
7.319
7.299
3.477
3.458
3.433
3.414
3.190
3.173
3.156
3.140
3.123
3.105
3.063
3.049
3.019
3.005
2.995
2.977
2.960
2.943
2.926
2.908
1.312
1.294
1.272
1.255
O
COOH
3d
2.10
2.09
1.00
1.02
1.15
0.99
3.10
6.46
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


197.857
182.338
155.107
134.627
128.598
126.973
77.618
77.300
76.982
41.891
35.128
34.524
23.928
17.354
O
COOH
3d
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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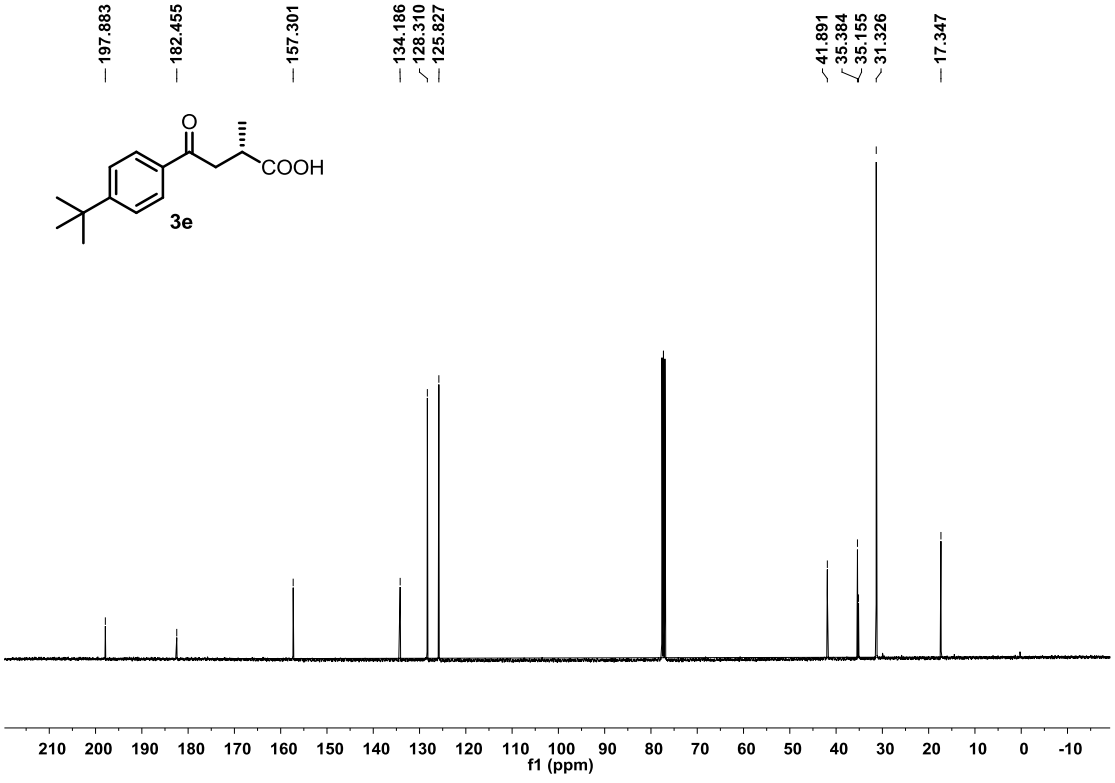
7.906
7.489
7.468
3.486
3.468
3.442
3.424
3.198
3.181
3.163
3.148
3.130
3.112
3.071
3.057
3.027
3.013
1.342
1.316
1.298
O
COOH
3e
2.07
2.07
1.00
1.05
1.00
9.10
3.03
f1 (ppm)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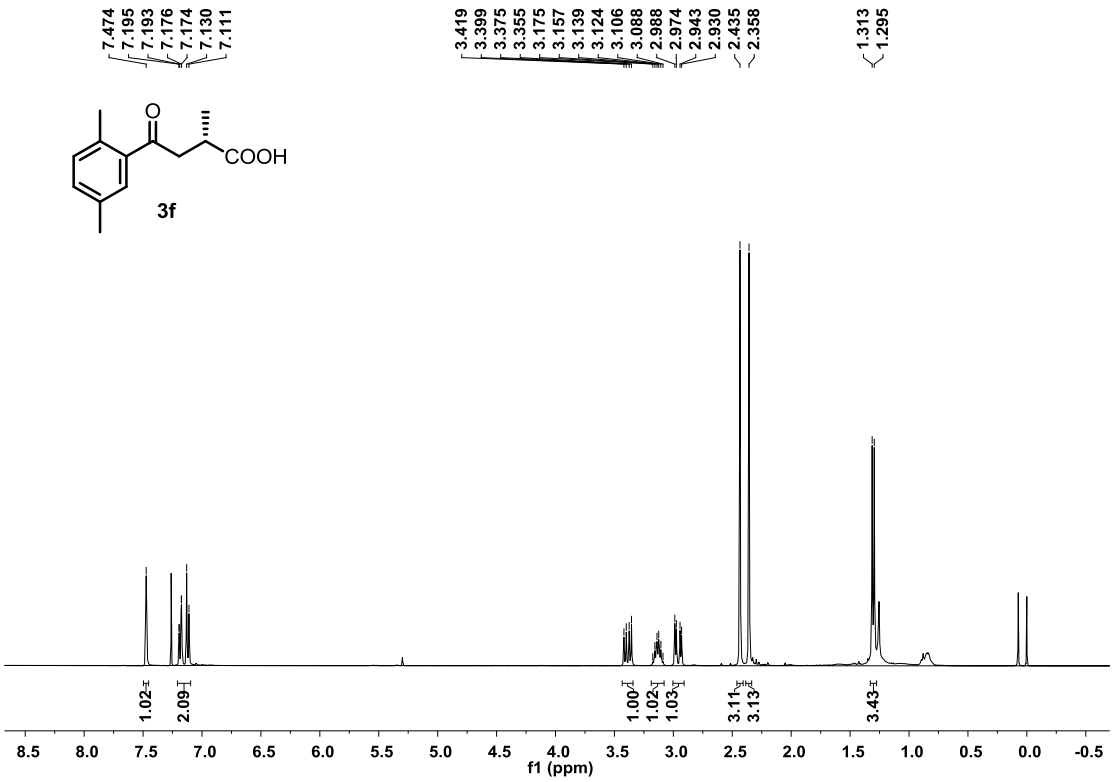
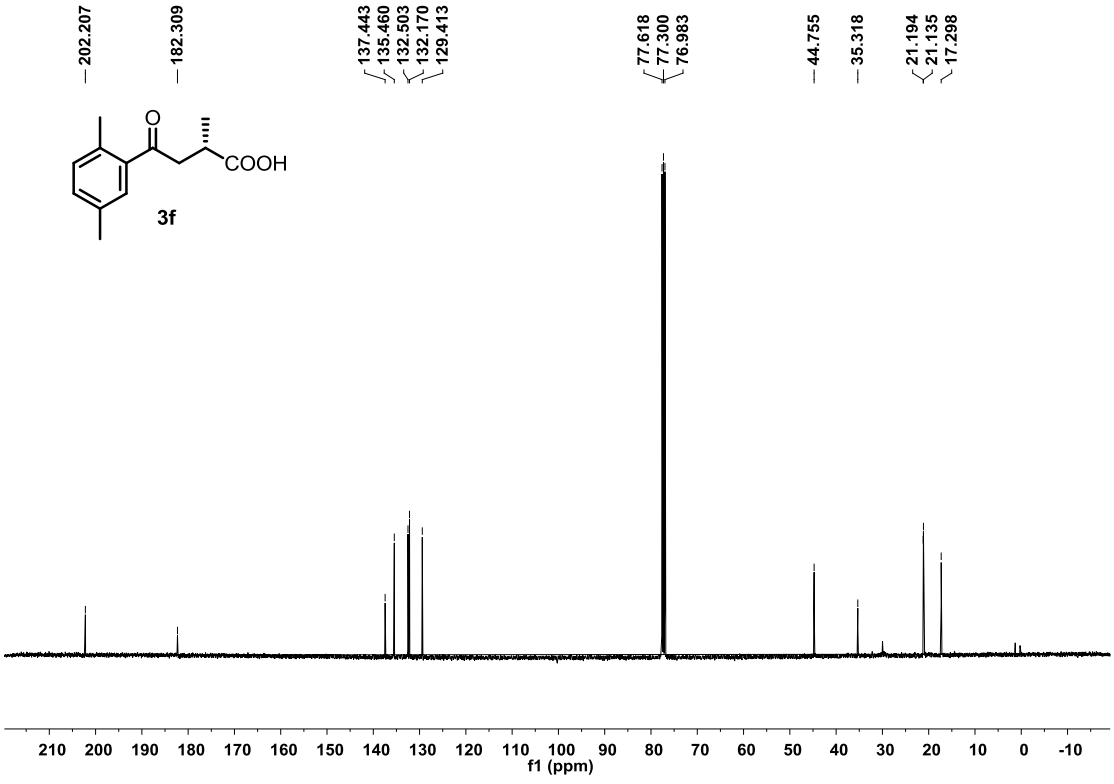
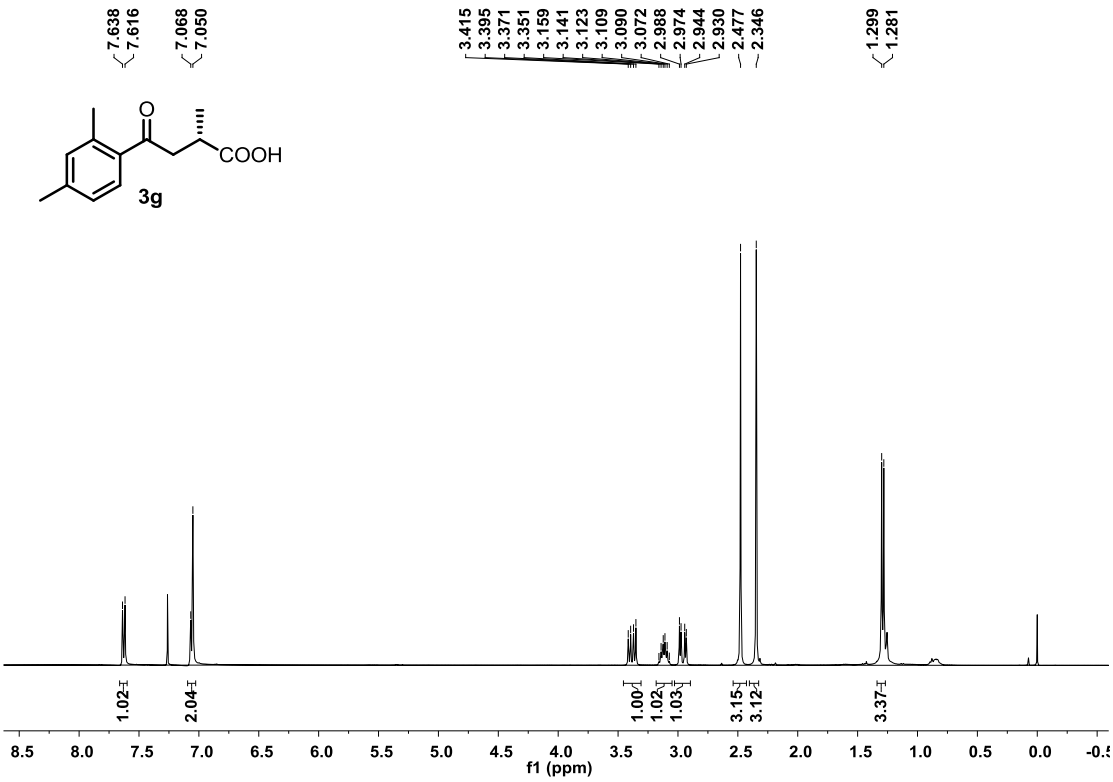
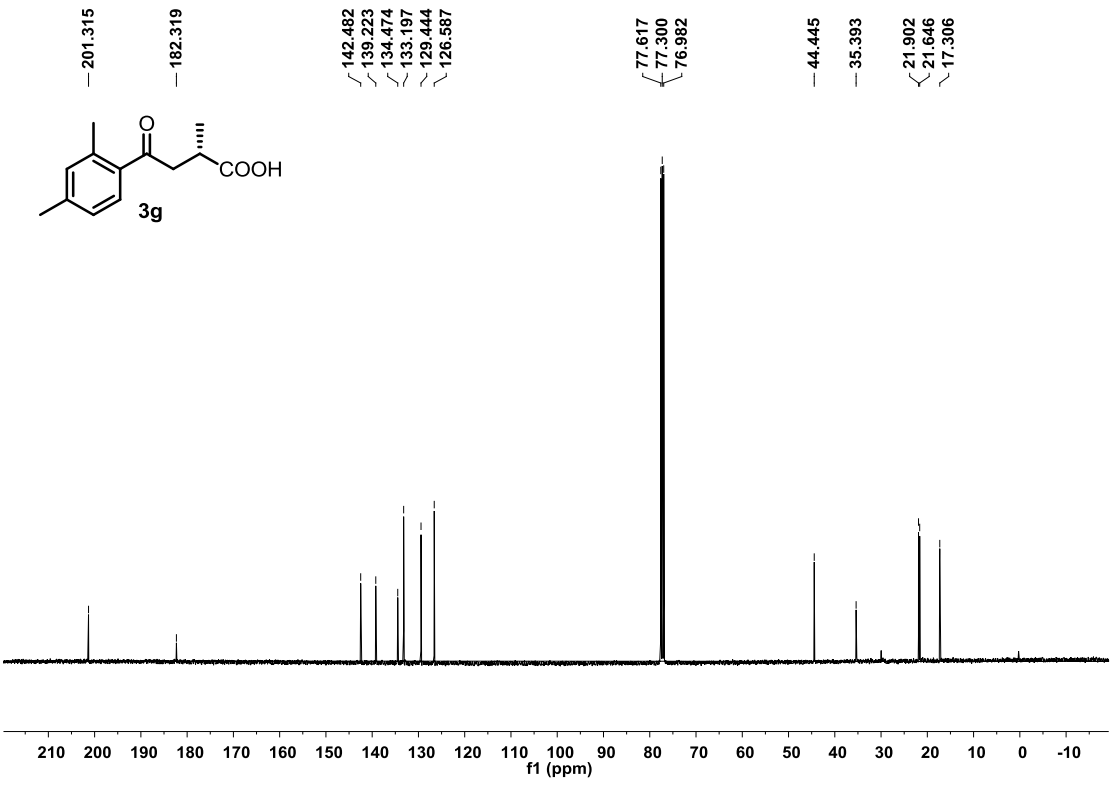
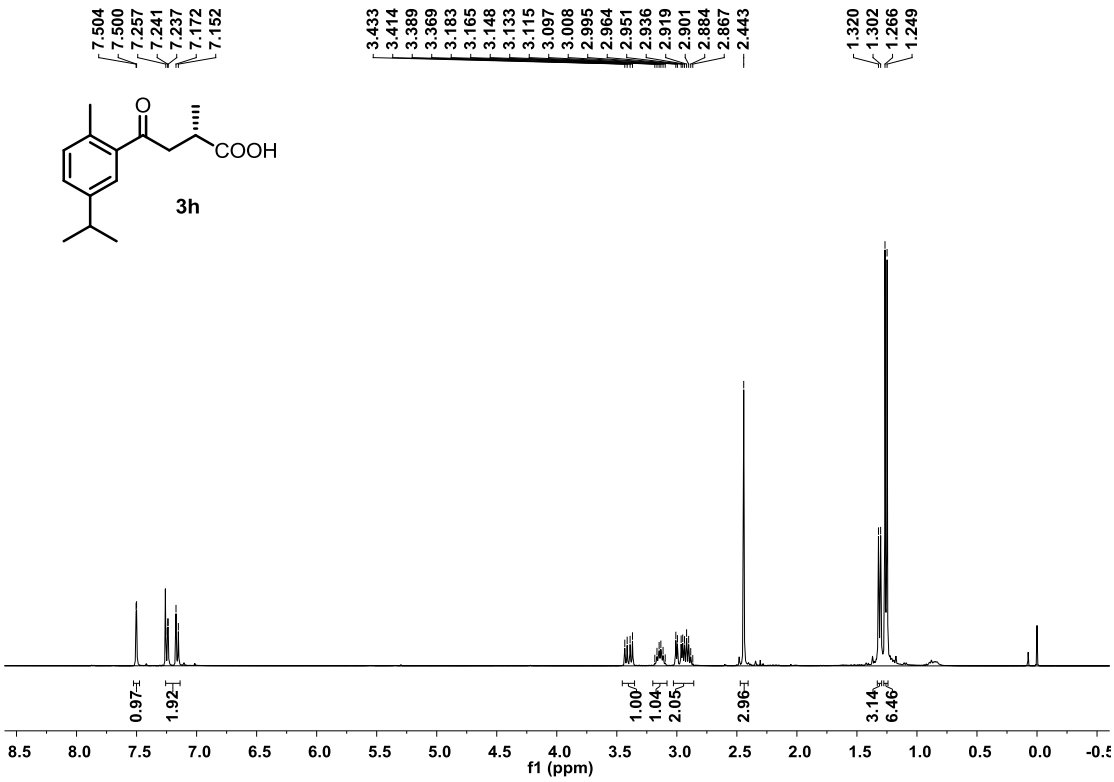
182.455
157.301
134.186
128.310
125.827
41.891
35.384
35.155
31.326
17.347
O
COOH
3e
f1 (ppm)

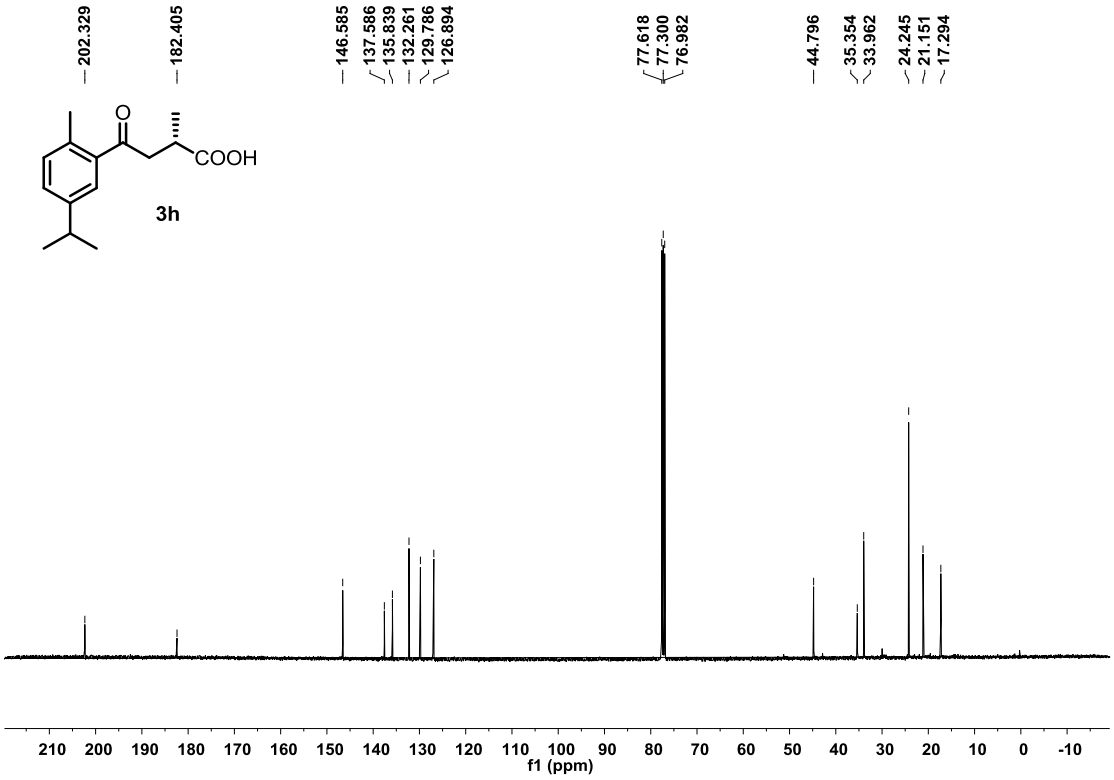

7.474
7.195
7.193
7.176
7.174
7.130
7.111
3.419
3.399
3.375
3.355
3.175
3.157
3.139
3.124
3.106
3.088
2.988
2.974
2.943
2.930
2.435
2.358
1.313
1.295
O
COOH
3f
1.02
2.09
1.00
1.02
1.03
3.11
3.13
3.43
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

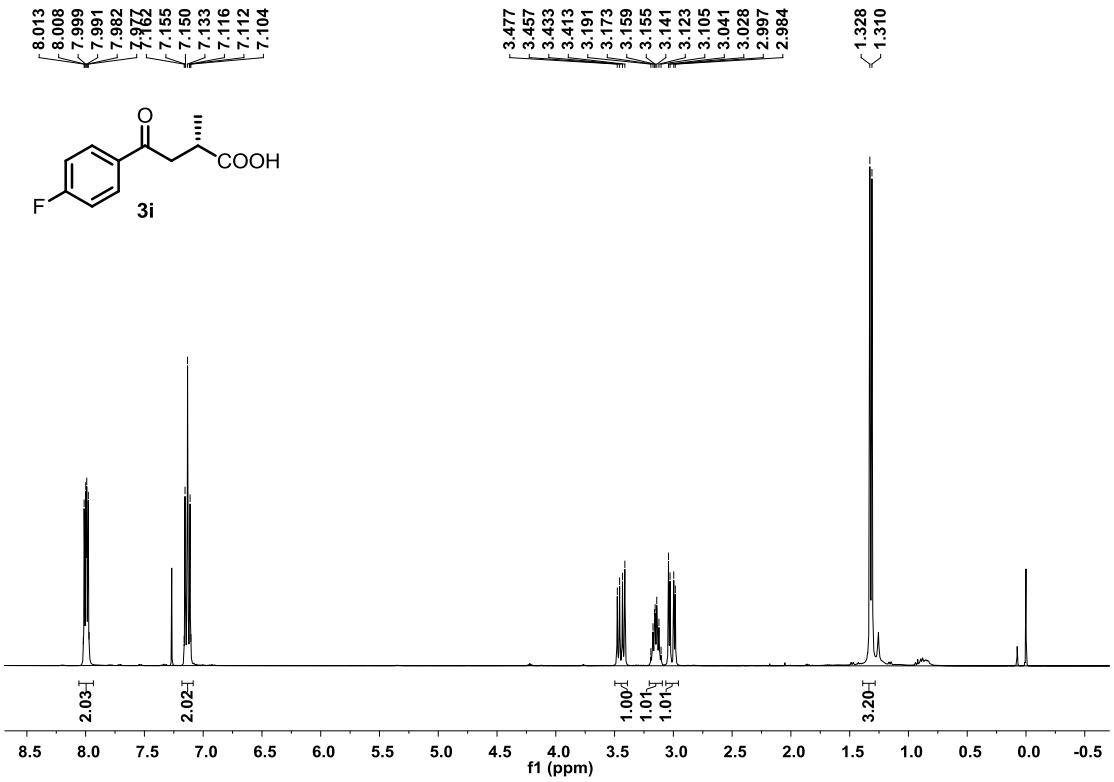

202.207
182.309
137.443
135.460
132.503
132.170
129.413
77.618
77.300
76.983
44.755
35.318
21.194
21.135
17.298
O
COOH
3f
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

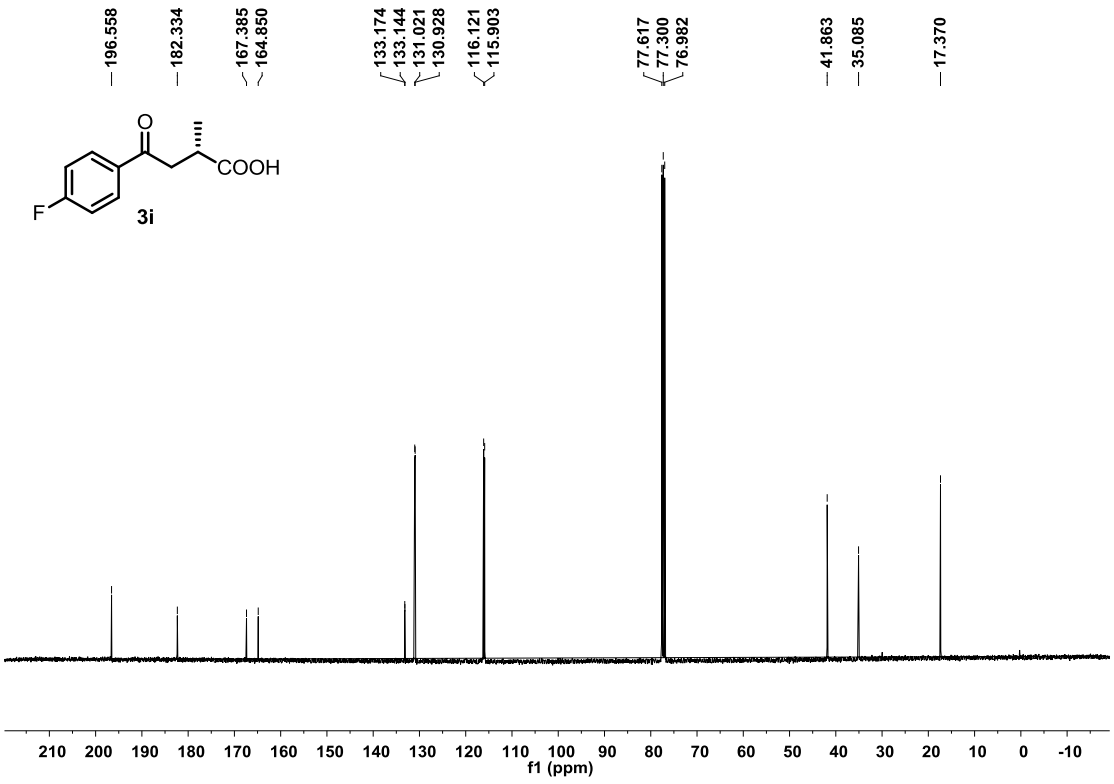

7.638
7.616
7.068
7.050
3.415
3.395
3.371
3.351
3.159
3.141
3.123
3.109
3.090
3.072
2.988
2.974
2.944
2.930
2.477
2.346
1.299
1.281
O
COOH
3g
1.02
2.04
1.00
1.02
1.03
3.15
3.12
3.37
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

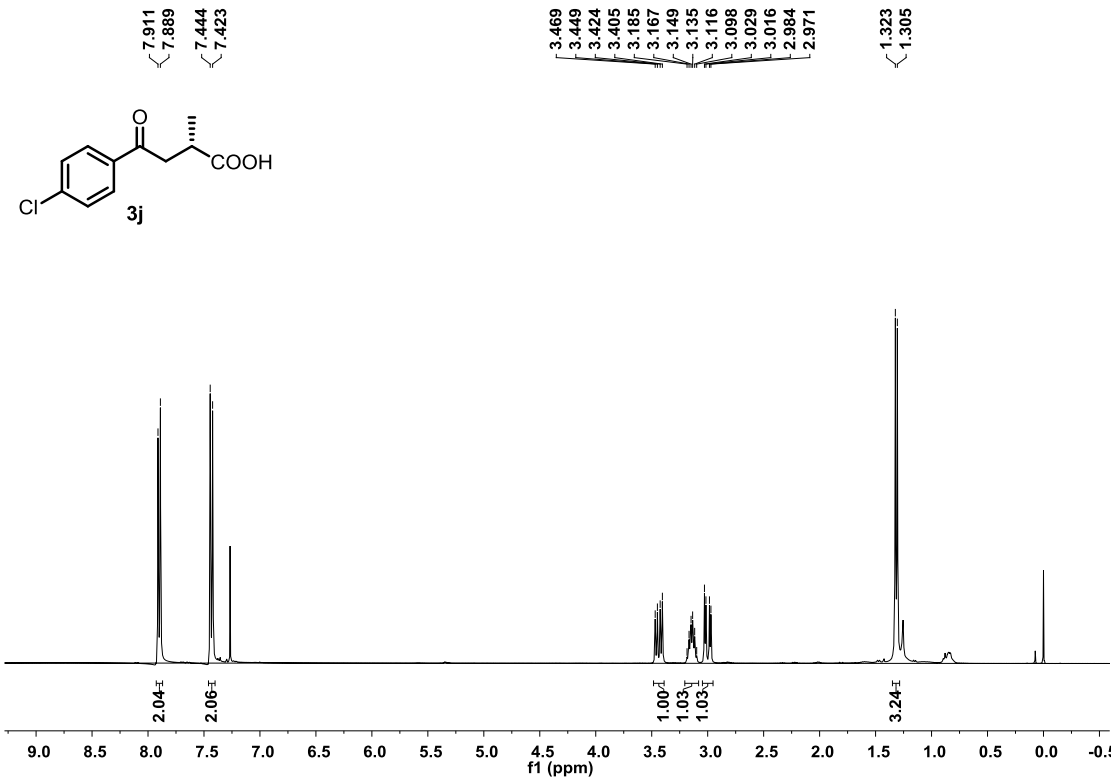

201.315
182.319
142.482
139.223
134.474
133.197
129.444
126.587
77.617
77.300
76.982
44.445
35.393
21.902
21.646
17.306
O
COOH
3g
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

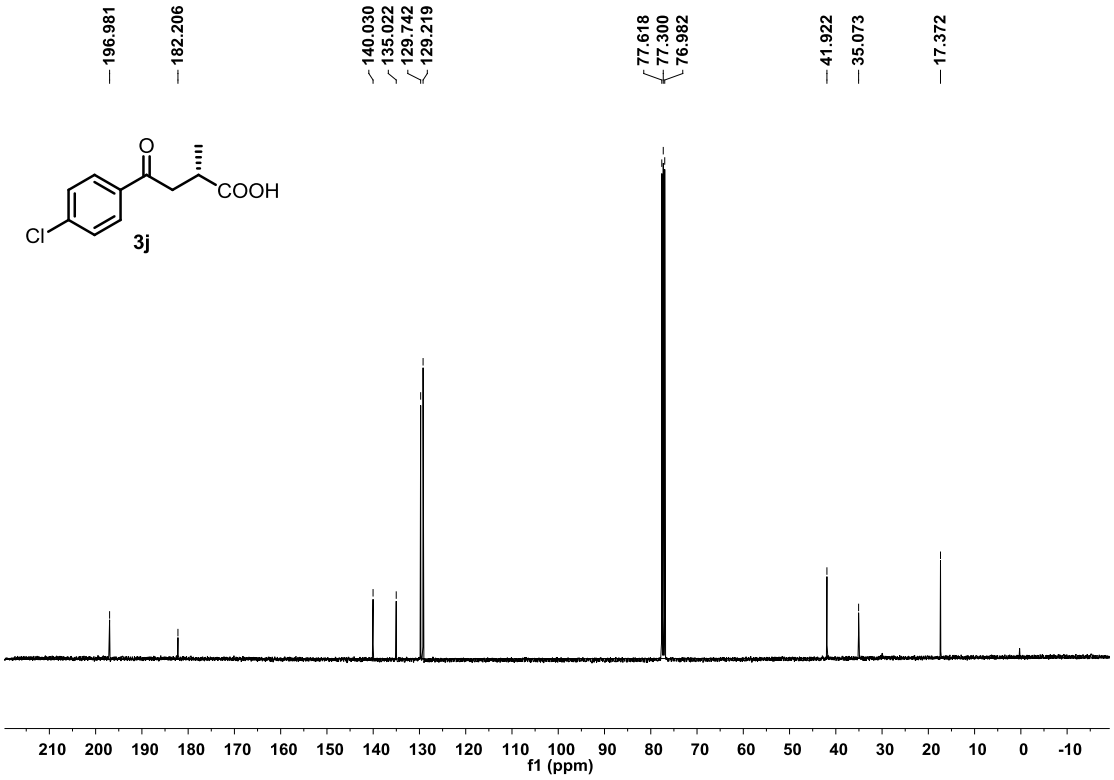

7.504
7.500
7.257
7.241
7.237
7.172
7.152
3.433
3.414
3.389
3.369
3.183
3.165
3.148
3.133
3.115
3.097
3.008
2.995
2.964
2.951
2.936
2.919
2.901
2.884
2.867
2.443
1.320
1.302
1.266
1.249
O
COOH
3h
0.97
1.92
1.00
1.04
2.05
2.96
3.14
6.46
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

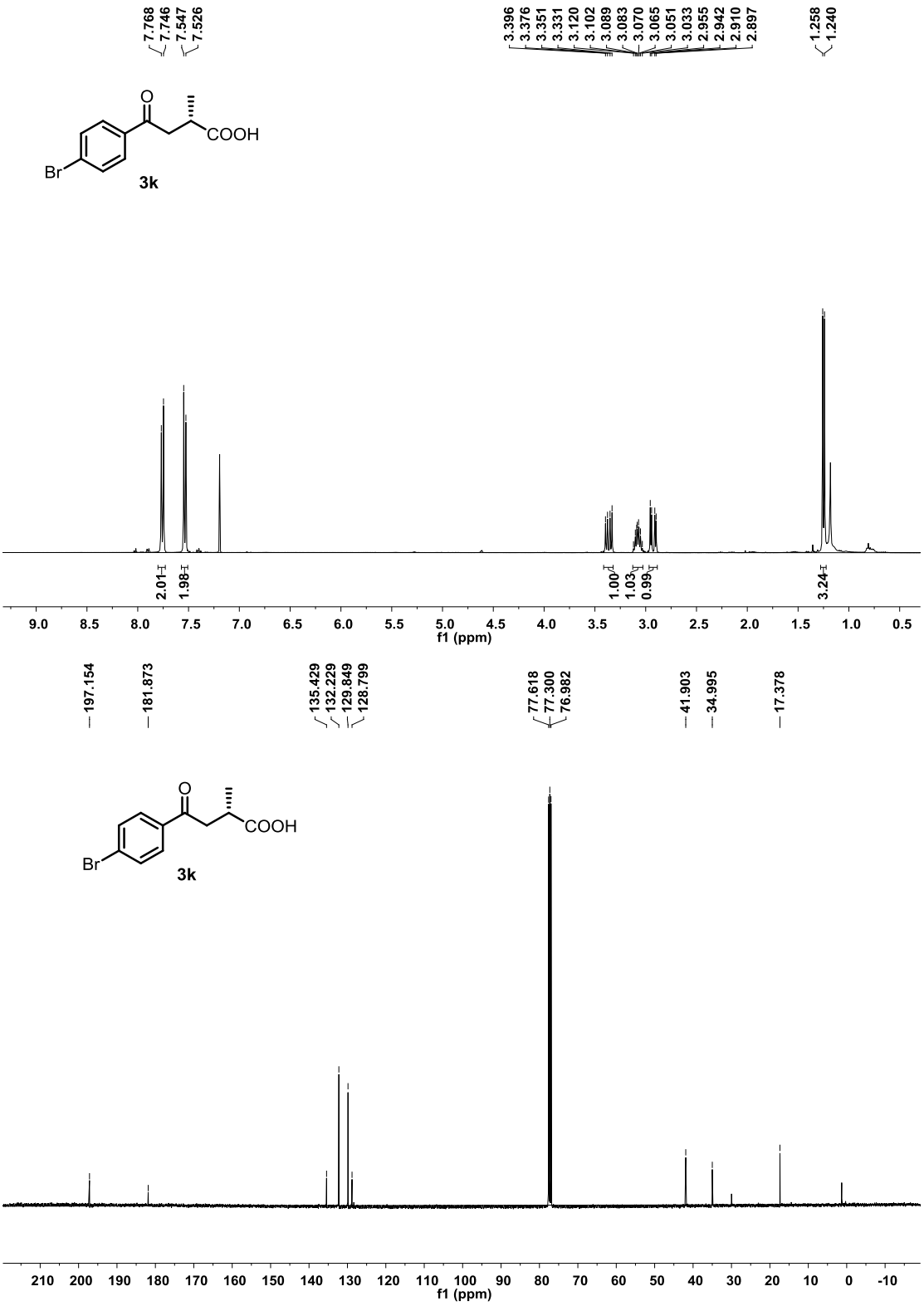

202.329
182.405
146.585
137.586
135.839
132.261
129.786
126.894
77.618
77.300
76.982
44.796
35.354
33.962
24.245
21.151
17.294
O
COOH
3h
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

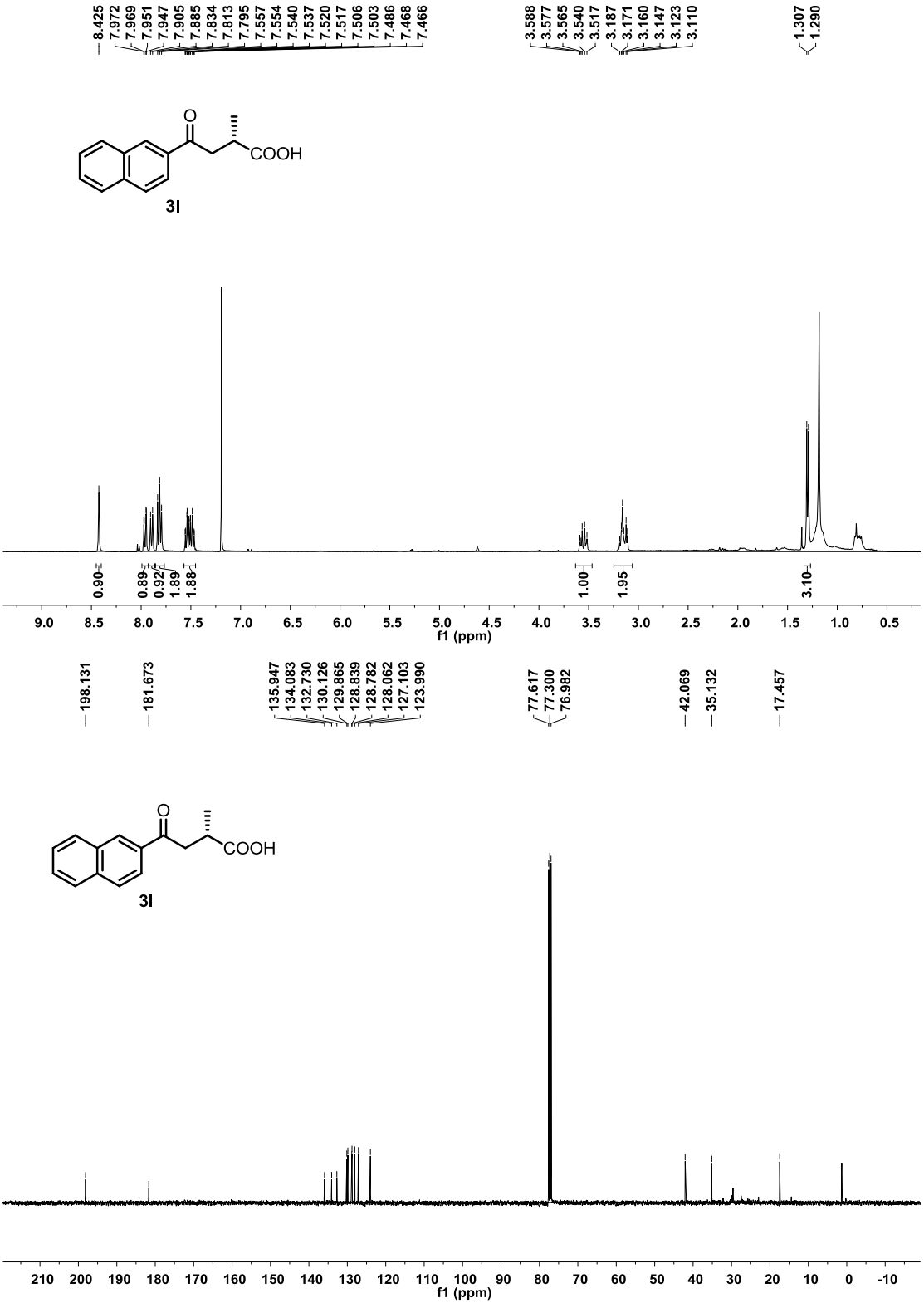

8.013
8.008
7.999
7.991
7.982
7.977
7.962
7.155
7.150
7.133
7.116
7.112
7.104
3.477
3.457
3.433
3.413
3.191
3.173
3.159
3.155
3.141
3.123
3.105
3.041
3.028
2.997
2.984
1.328
1.310
O
COOH
F
3i
2.03
2.02
1.00
1.01
1.01
3.20
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

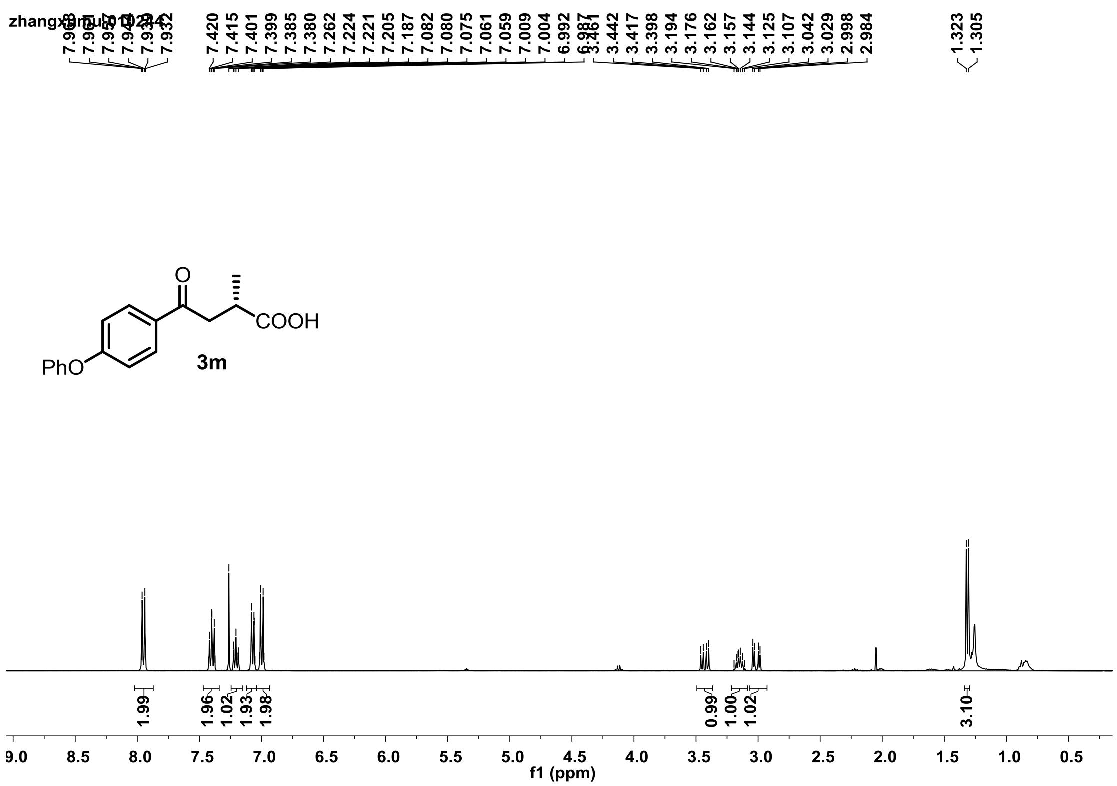

196.558
182.334
167.385
164.850
133.174
133.144
131.021
130.928
116.121
115.903
77.617
77.300
76.982
41.863
35.085
17.370
O
COOH
F
3i
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

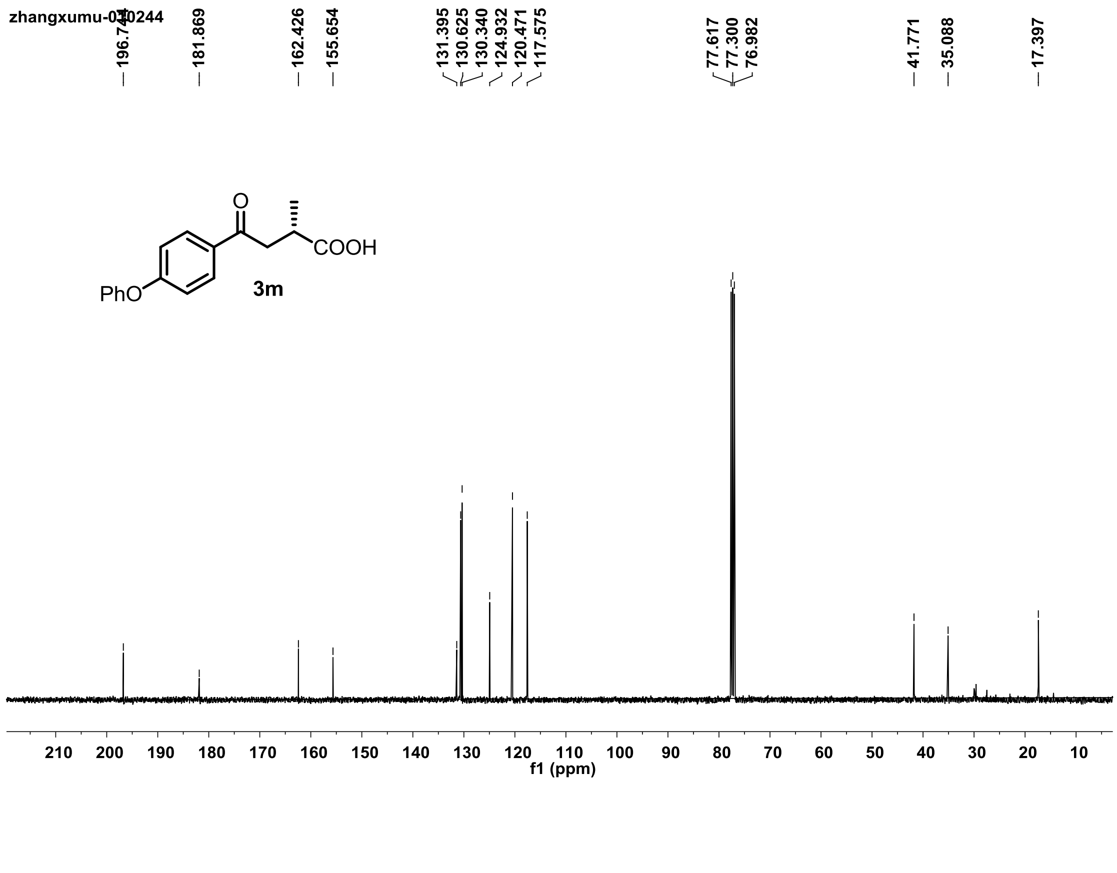

7.911
7.889
7.444
7.423
3.469
3.449
3.424
3.405
3.185
3.167
3.149
3.135
3.116
3.098
3.029
3.016
2.984
2.971
1.323
1.305
O
COOH
Cl
3j
2.04
2.06
1.00
1.03
1.03
3.24
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

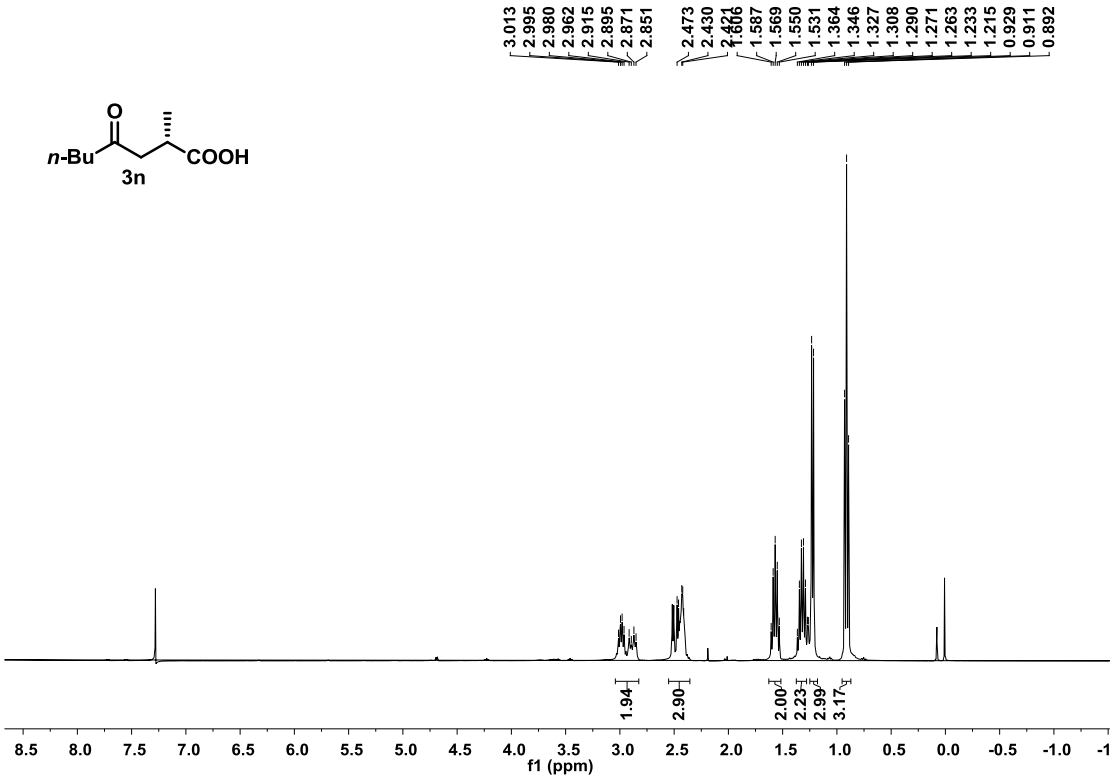

196.981
182.206
140.030
135.022
129.742
129.219
77.618
77.300
76.982
41.922
35.073
17.372
O
COOH
Cl
3j
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

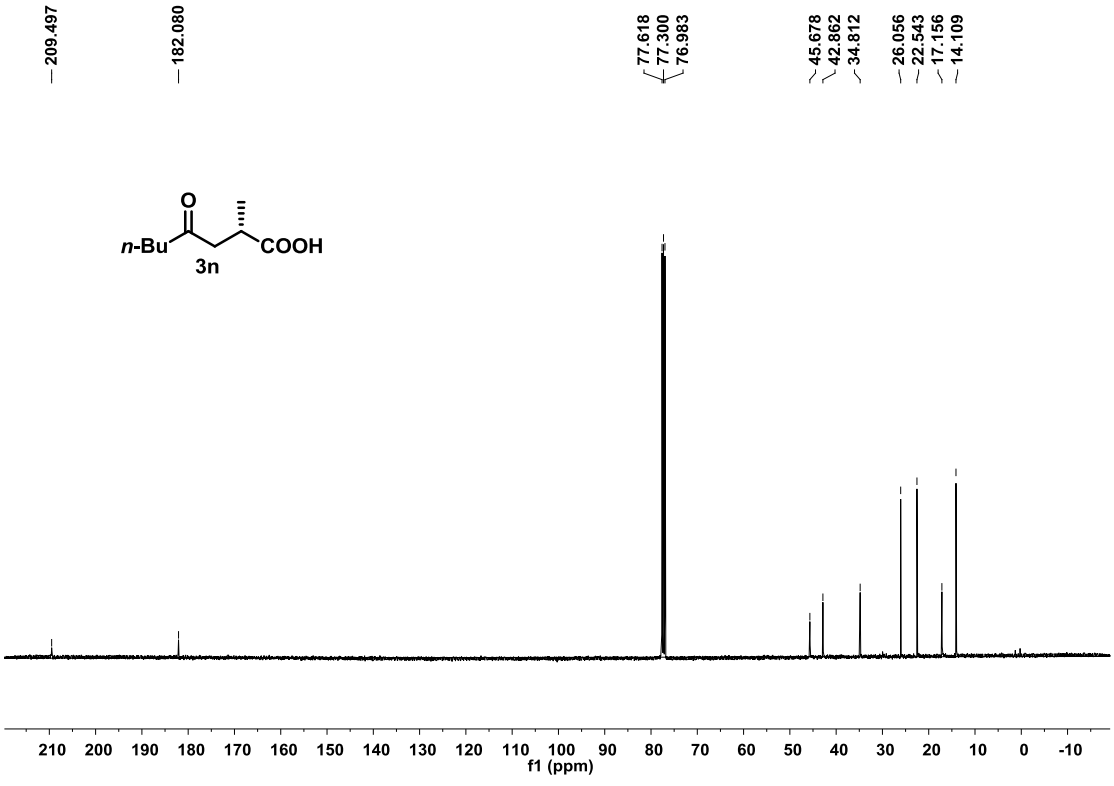

7.768
7.746
7.547
7.526
3.396
3.376
3.351
3.331
3.120
3.102
3.089
3.083
3.070
3.065
3.051
3.033
2.955
2.942
2.910
2.897
1.258
1.240
O
COOH
Br
3k
2.01
1.98
1.00
1.03
0.99
3.24
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)
197.154
181.873
135.429
132.229
129.849
128.799
77.618
77.300
76.982
41.903
34.995
17.378
O
COOH
Br
3k
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

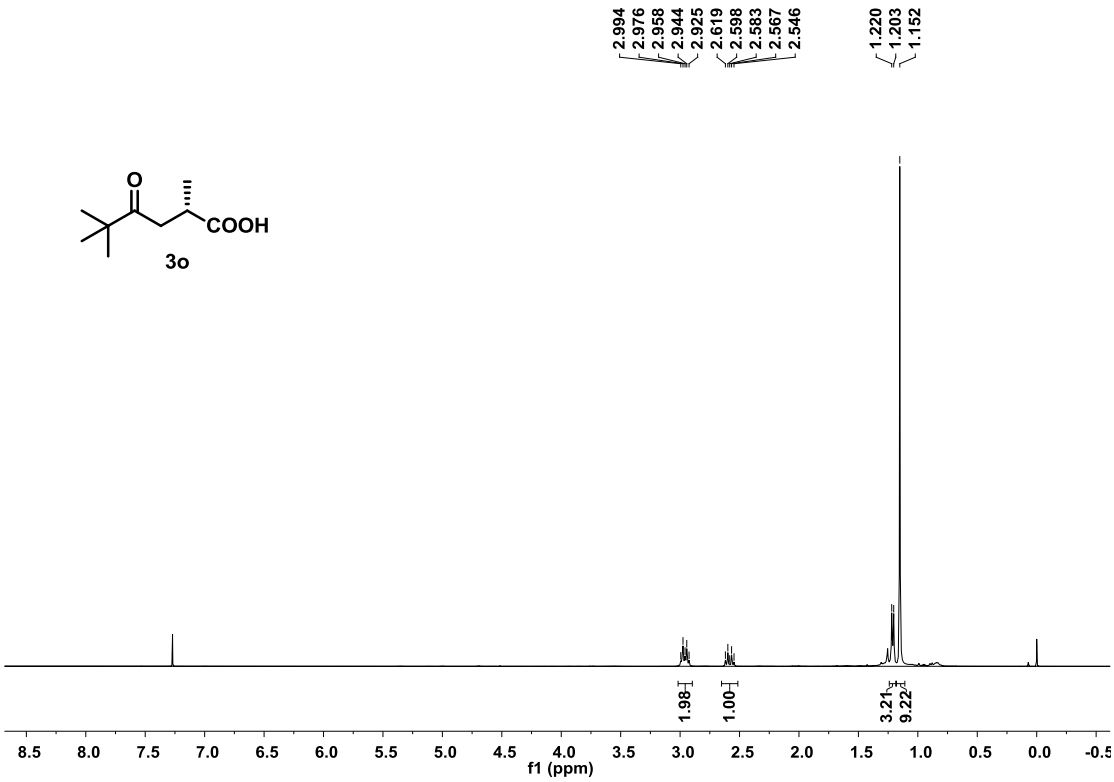

8.425
7.972
7.969
7.951
7.947
7.905
7.885
7.834
7.813
7.795
7.557
7.554
7.540
7.537
7.520
7.517
7.506
7.503
7.486
7.468
7.466
3.588
3.577
3.565
3.540
3.517
3.187
3.171
3.160
3.147
3.123
3.110
1.307
1.290
O
COOH
3l
0.90
0.89
0.92
1.89
1.88
1.00
1.95
3.10
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)
198.131
181.673
135.947
134.083
132.730
130.126
129.865
128.839
128.782
128.062
127.103
123.990
77.617
77.300
76.982
42.069
35.132
17.457
O
COOH
3l
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

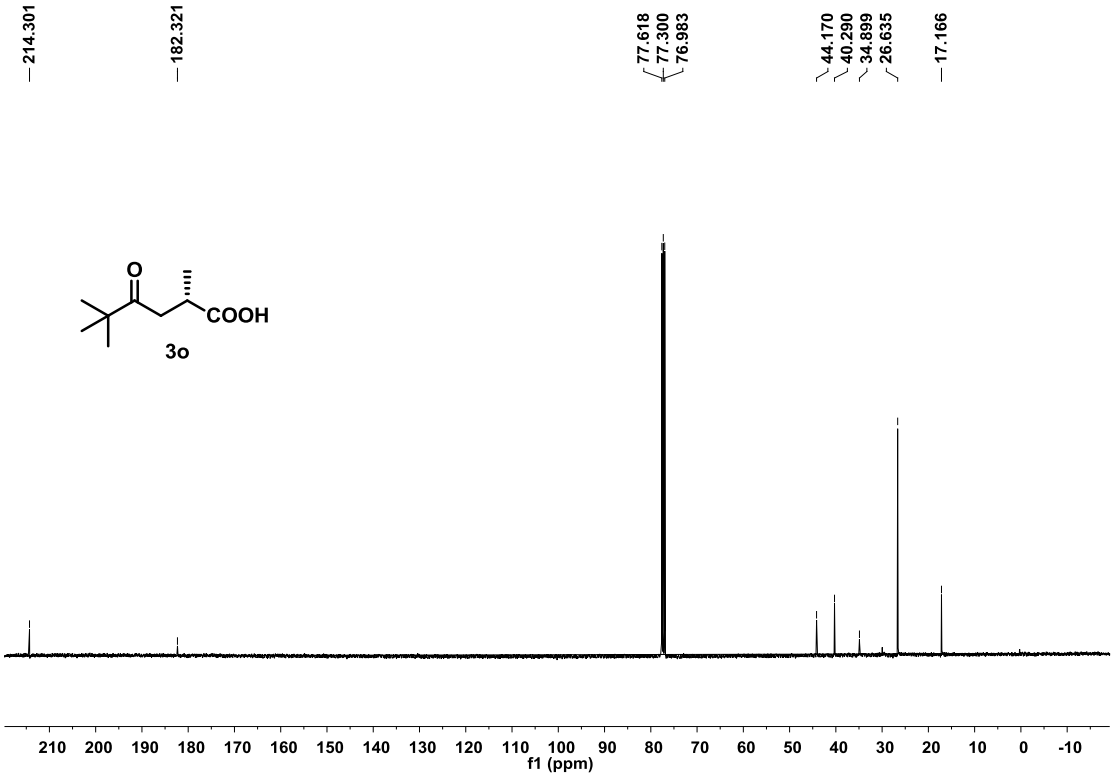

7.420
7.415
7.401
7.399
7.385
7.380
7.262
7.224
7.221
7.205
7.187
7.082
7.080
7.075
7.061
7.059
7.009
7.004
6.992
6.987
3.461
3.442
3.417
3.398
3.194
3.176
3.162
3.157
3.144
3.125
3.107
3.042
3.029
2.998
2.984
1.323
1.305
O
COOH
PhO
3m
1.99
1.96
1.02
1.93
1.98
0.99
1.00
1.02
3.10
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

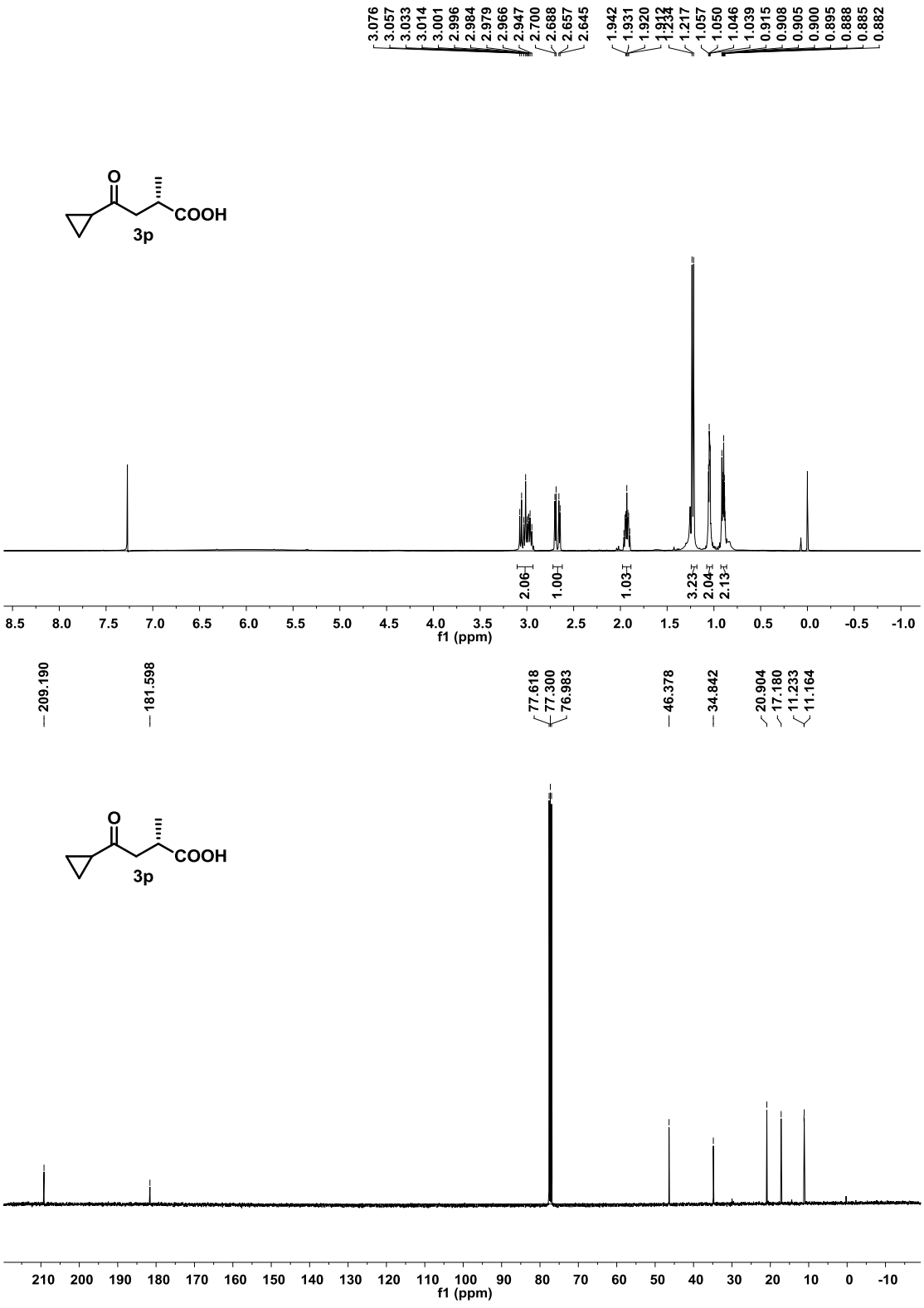

181.869
162.426
155.654
131.395
130.625
130.340
124.932
120.471
117.575
77.617
77.300
76.982
41.771
35.088
17.397
O
COOH
PhO
3m
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

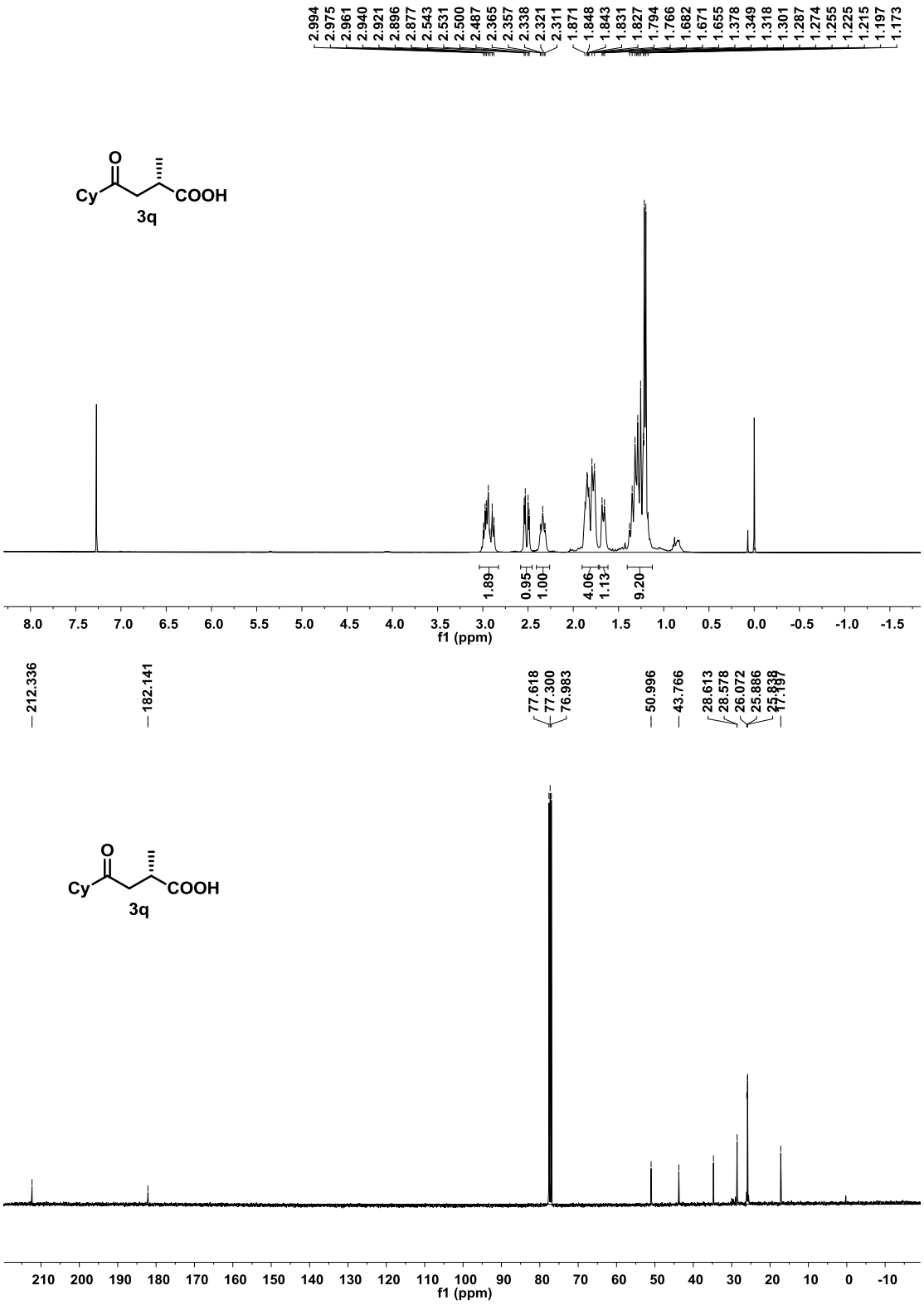

3.013
2.995
2.980
2.962
2.915
2.895
2.871
2.851
2.473
2.430
2.421
1.606
1.587
1.569
1.550
1.531
1.364
1.346
1.327
1.308
1.290
1.271
1.263
1.233
1.215
0.929
0.911
0.892
n-Bu
COOH
3n
1.94
2.90
2.00
2.23
2.99
3.17
f1 (ppm)


209.497
182.080
77.618
77.300
76.983
45.678
42.862
34.812
26.056
22.543
17.156
14.109
n-Bu
COOH
3n
f1 (ppm)

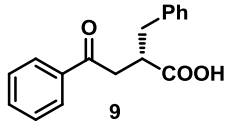

2.994
2.976
2.958
2.944
2.925
2.619
2.598
2.583
2.567
2.546
1.220
1.203
1.152
O
COOH
3o
1.98
1.00
3.21
9.22
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

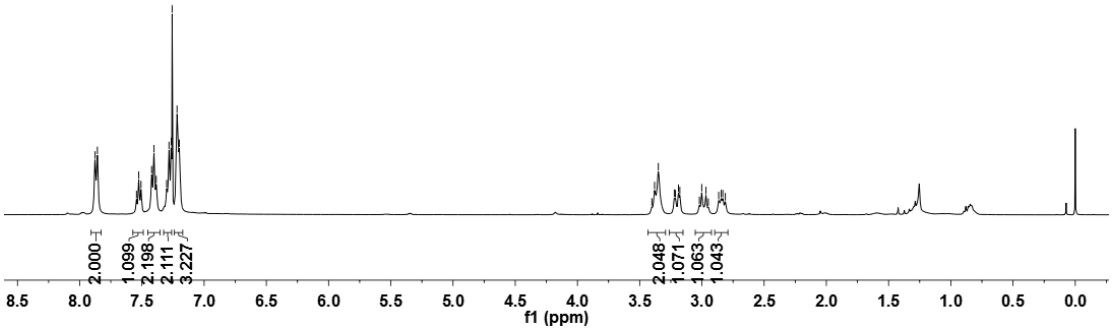

214.301
182.321
77.618
77.300
76.983
44.170
40.290
34.899
26.635
17.166
O
COOH
3o
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

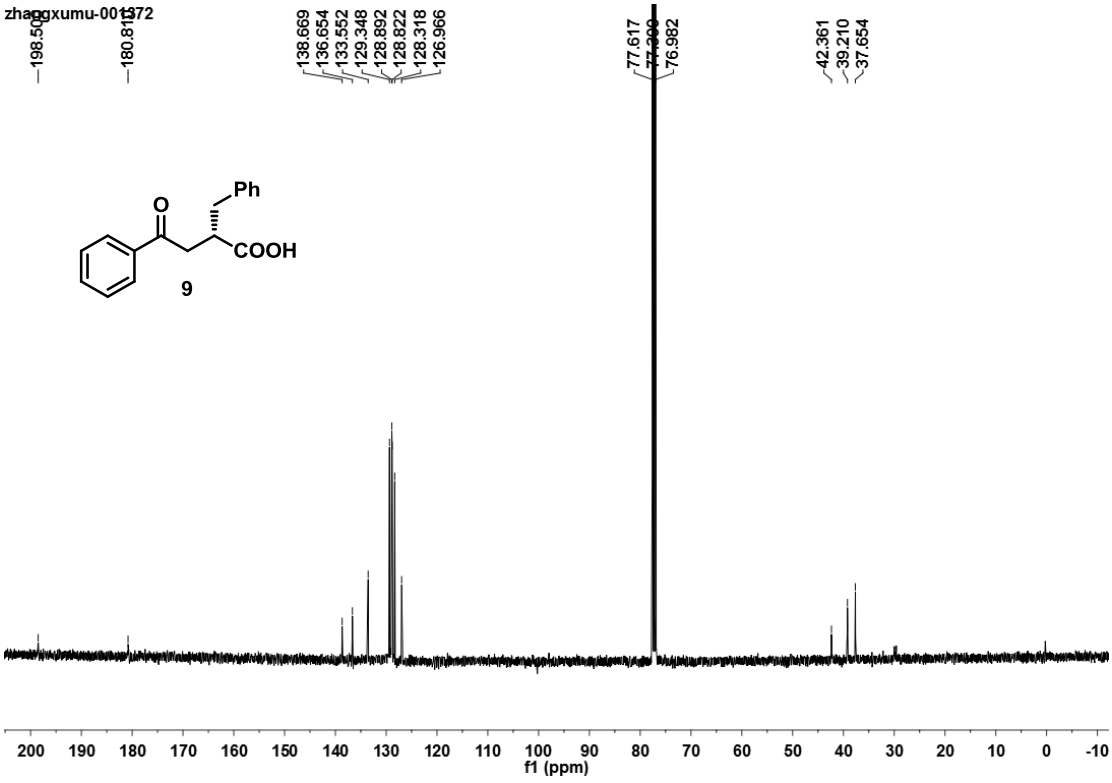

3.076
3.057
3.033
3.014
3.001
2.996
2.984
2.979
2.966
2.947
2.700
2.688
2.657
2.645
1.942
1.931
1.920
1.912
1.234
1.217
1.057
1.050
1.046
1.039
0.915
0.908
0.905
0.900
0.895
0.888
0.885
0.882
O
COOH
3p
2.06
1.00
1.03
3.23
2.04
2.13
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)
209.190
181.598
77.618
77.300
76.983
46.378
34.842
20.904
17.180
11.233
11.164
O
COOH
3p
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


2.994
2.975
2.961
2.940
2.921
2.896
2.877
2.543
2.531
2.500
2.487
2.365
2.357
2.338
2.321
2.311
1.871
1.848
1.843
1.831
1.827
1.794
1.766
1.682
1.671
1.655
1.378
1.349
1.318
1.301
1.287
1.274
1.255
1.225
1.215
1.197
1.173
O
Cy
COOH
3q
1.89
0.95
1.00
4.06
1.13
9.20
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5
f1 (ppm)
212.336
182.141
77.618
77.300
76.983
50.996
43.766
28.613
28.578
26.072
25.886
O
Cy
COOH
3q
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


zhangxumu-001372
7.877
7.856
7.541
7.526
7.507
7.422
7.403
7.385
7.301
7.282
7.265
7.257
7.218
7.199
3.402
3.380
3.350
3.222
3.212
3.188
3.177
3.020
3.001
2.967
2.948
2.865
2.845
2.832
2.812


Ph
O
COOH
9

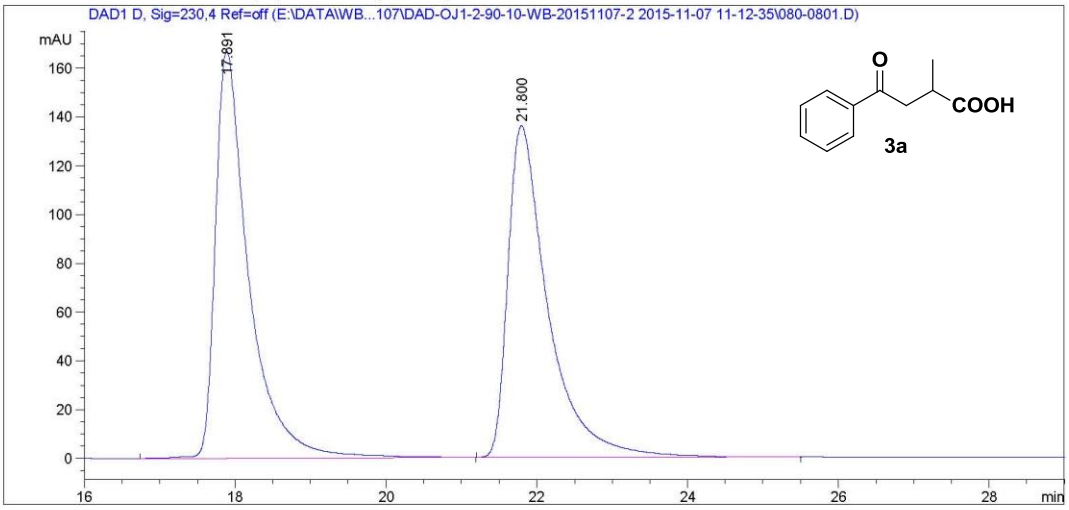

2.000
1.099
2.198
2.111
3.227
2.048
1.071
1.063
1.043
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


zhangxumu-001372
198.500
180.811
138.669
136.654
133.552
129.348
128.892
128.822
128.318
126.966
77.617
77.200
76.982
42.361
39.210
37.654
Ph
O
COOH
9
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

HPLC spectra

Data File E:\DATA\WB...151107\DAD-OJ1-2-90-10-WB-20151107-2 2015-11-07 11-12-35\080-0801.D
Sample Name: CCY-6-94-5-RAC

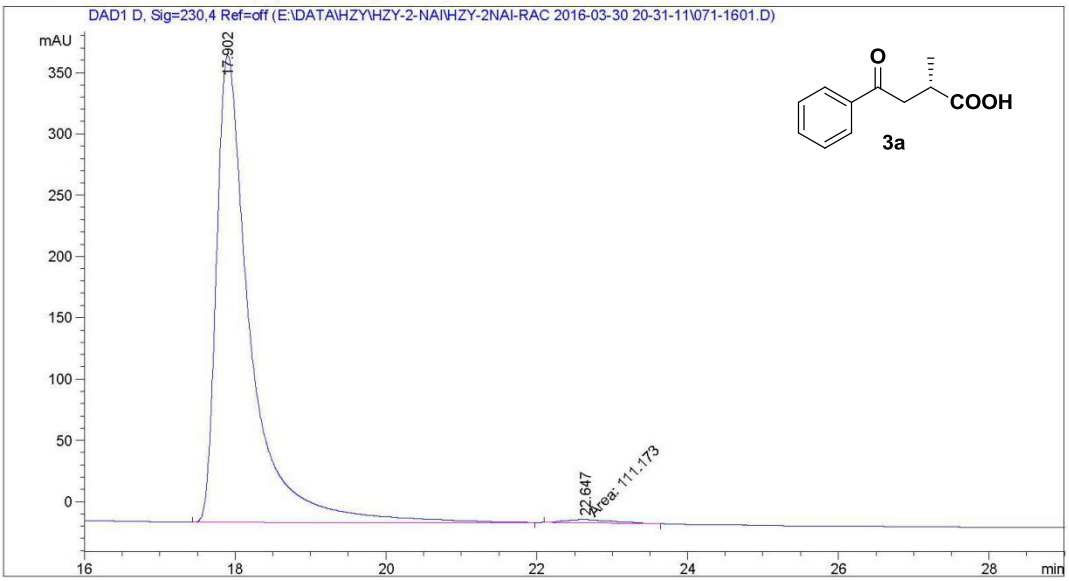
```
==================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   8
Acq. Instrument : 1260HPLC-DAD                  Location : Vial 80
Injection Date  : 11/7/2015 2:31:30 PM               Inj :   1
                                               Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\WB\WEIB-20151107\DAD-OJ1-2-90-10-WB-20151107-2 2015-11-07 11-12-35
                  \DAD-OJ(1-6)-95-5-1.0ML-ALLNM-45MIN.M
Last changed    : 11/7/2015 12:28:53 PM by SYSTEM
Analysis Method : E:\DATA\WB\WEIB-20151107\DAD-OJ1-2-90-10-WB-20151107-2 2015-11-07 11-12-35
                  \DAD-OJ(1-6)-95-5-1.0ML-ALLNM-45MIN.M (Sequence Method)
Last changed    : 9/5/2016 9:45:28 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 D, Sig=230,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  17.891 BB    0.4479 5121.19678  166.95232  50.4726
   2  21.800 BB    0.5410 5025.28809  136.01506  49.5274

Totals :                  1.01465e4   302.96738

==================================================================
                        *** End of Report ***
```

Data File E:\DATA\HZY\HZY-2-NAI\HZY-2NAI-RAC 2016-03-30 20-31-11\071-1601.D
Sample Name: wsw-1-75-1

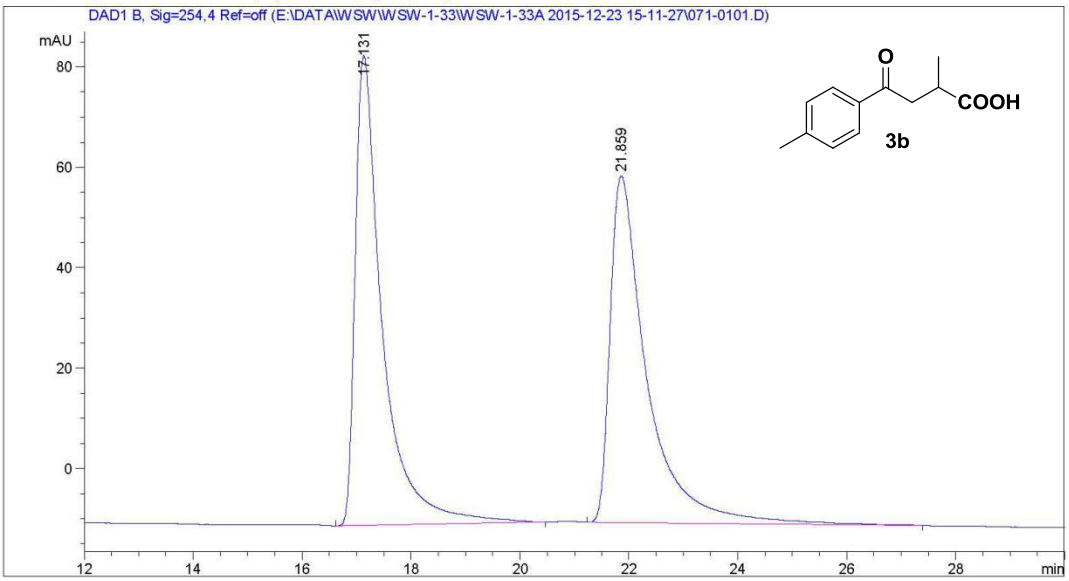
```
==================================================================
Acq. Operator   : SYSTEM                        Seq. Line :  16
Acq. Instrument : 1260HPLC-DAD                   Location : Vial 71
Injection Date  : 3/31/2016 5:56:27 AM                Inj :   1
                                                Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\HZY\HZY-2-NAI\HZY-2NAI-RAC 2016-03-30 20-31-11\DAD-OJ(1-6)-95-5-1.
                  0ML-ALL-45MIN.M
Last changed    : 3/30/2016 10:58:50 PM by SYSTEM
Analysis Method : E:\DATA\HZY\HZY-2-NAI\HZY-2NAI-RAC 2016-03-30 20-31-11\DAD-OJ(1-6)-95-5-1.
                  0ML-ALL-45MIN.M (Sequence Method)
Last changed    : 2/10/2017 8:03:40 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==================================================================
                      Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 D, Sig=230,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  17.902 BB    0.4515 1.18591e4   380.66229  99.0713
   2  22.647 MM    0.7386  111.17309    2.50857   0.9287

Totals :                  1.19703e4   383.17087
```

Data File E:\DATA\WSW\WSW-1-33\WSW-1-33A 2015-12-23 15-11-27\071-0101.D
Sample Name: WSW-1-33-1

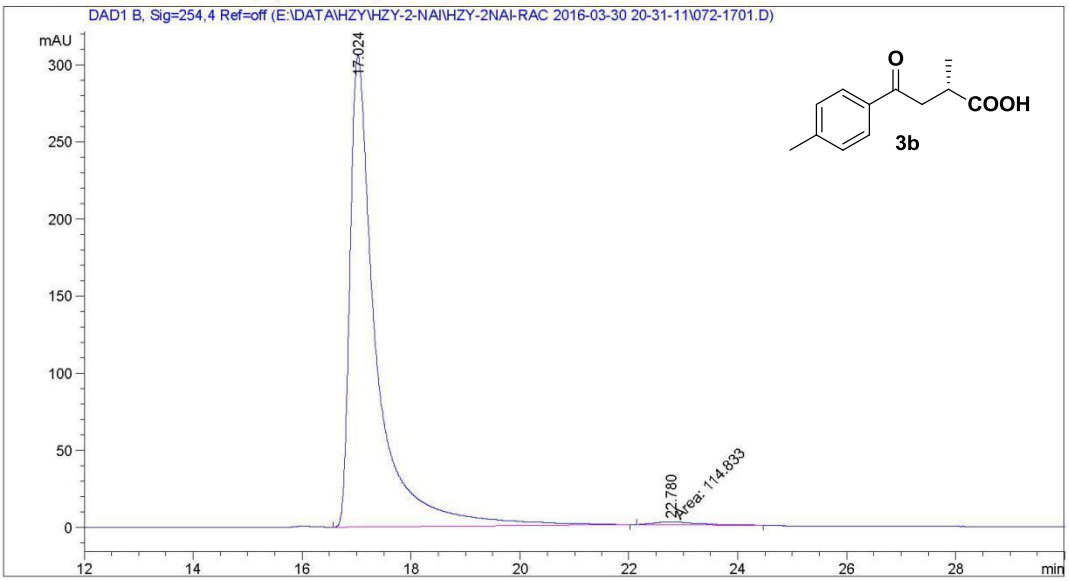
```
=====================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   1
Acq. Instrument : 1260HPLC-DAD                   Location : Vial 71
Injection Date  : 12/23/2015 3:12:19 PM               Inj :   1
                                                Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\WSW\WSW-1-33\WSW-1-33A 2015-12-23 15-11-27\DAD-OJ(1-6)-95-5-1.0ML-
                  ALL-45MIN.M
Last changed    : 12/23/2015 3:11:27 PM by SYSTEM
Analysis Method : E:\DATA\WSW\WSW-1-33\WSW-1-33A 2015-12-23 15-11-27\DAD-OJ(1-6)-95-5-1.0ML-
                  ALL-45MIN.M (Sequence Method)
Last changed    : 9/3/2016 7:27:20 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
=====================================================================
                        Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  17.131 BB    0.4999 3239.95850   93.59347  49.9162
   2  21.859 BB    0.6791 3250.84058   69.07588  50.0838

Totals :                  6490.79907  162.66935
```

Data File E:\DATA\HZY\HZY-2-NAI\HZY-2NAI-RAC 2016-03-30 20-31-11\072-1701.D
Sample Name: wsw-1-75-2

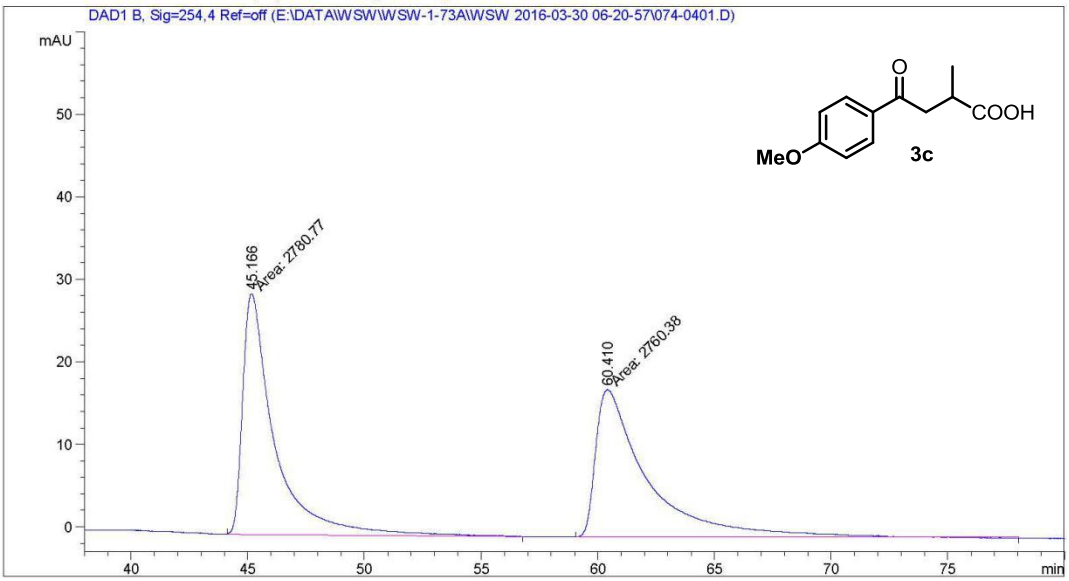
```
==================================================================
Acq. Operator   : SYSTEM                        Seq. Line :  17
Acq. Instrument : 1260HPLC-DAD                   Location : Vial 72
Injection Date  : 3/31/2016 6:42:27 AM                Inj :   1
                                                Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\HZY\HZY-2-NAI\HZY-2NAI-RAC 2016-03-30 20-31-11\DAD-OJ(1-6)-95-5-1.
                  0ML-ALL-45MIN.M
Last changed    : 3/30/2016 10:58:50 PM by SYSTEM
Analysis Method : E:\DATA\HZY\HZY-2-NAI\HZY-2NAI-RAC 2016-03-30 20-31-11\DAD-OJ(1-6)-95-5-1.
                  0ML-ALL-45MIN.M (Sequence Method)
Last changed    : 2/10/2017 8:52:56 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==================================================================
                      Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  17.024 BB    0.4770 1.01971e4   305.81003  98.8864
   2  22.780 MM    1.0717  114.83280    1.78584   1.1136

Totals :                  1.03119e4   307.59587
```

Data File E:\DATA\WSW\WSW-1-73A\WSW 2016-03-30 06-20-57\074-0401.D
Sample Name: WSW-1-73-4

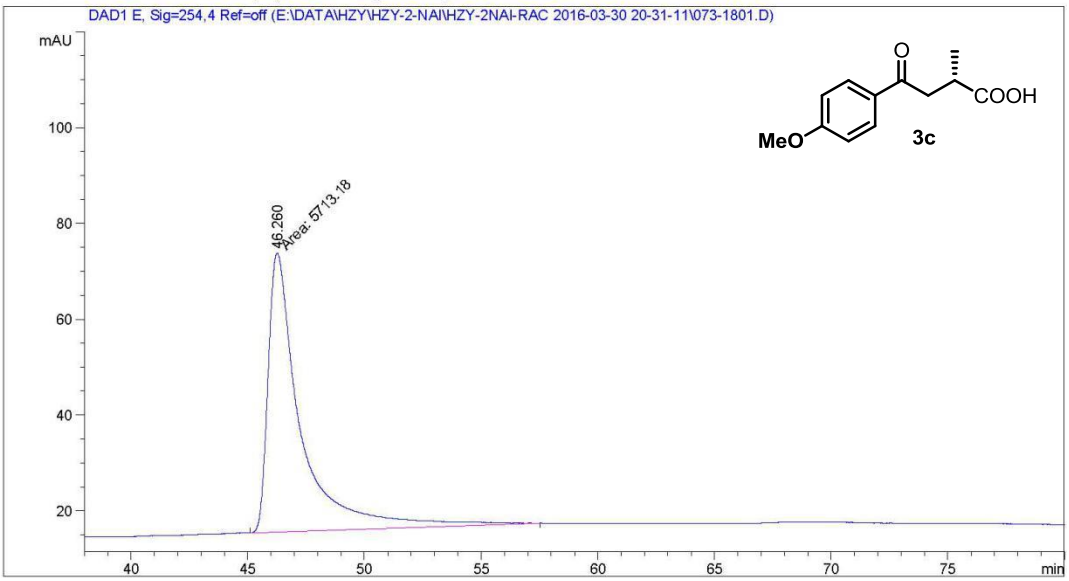
```
==========================================================================
Acq. Operator   : SYSTEM                          Seq. Line :   4
Acq. Instrument : 1260HPLC-DAD                    Location : Vial 74
Injection Date  : 3/30/2016 9:14:59 AM                 Inj :   1
                                                  Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\WSW\WSW-1-73A\WSW 2016-03-30 06-20-57\DAD-OJ(1-6)-95-5-1.0ML-ALL-
                  45MIN.M
Last changed    : 3/30/2016 6:42:23 AM by SYSTEM
Analysis Method : E:\DATA\WSW\WSW-1-73A\WSW 2016-03-30 06-20-57\DAD-OJ(1-6)-95-5-1.0ML-ALL-
                  45MIN.M (Sequence Method)
Last changed    : 9/5/2016 9:33:18 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  45.166 MM    1.5877 2780.76855   29.18990  50.1840
   2  60.410 MM    2.5733 2760.37842   17.87862  49.8160

Totals :                 5541.14697   47.06852
```

Data File E:\DATA\HZY\HZY-2-NAI\HZY-2NAI-RAC 2016-03-30 20-31-11\073-1801.D
Sample Name: wsw-1-75-3

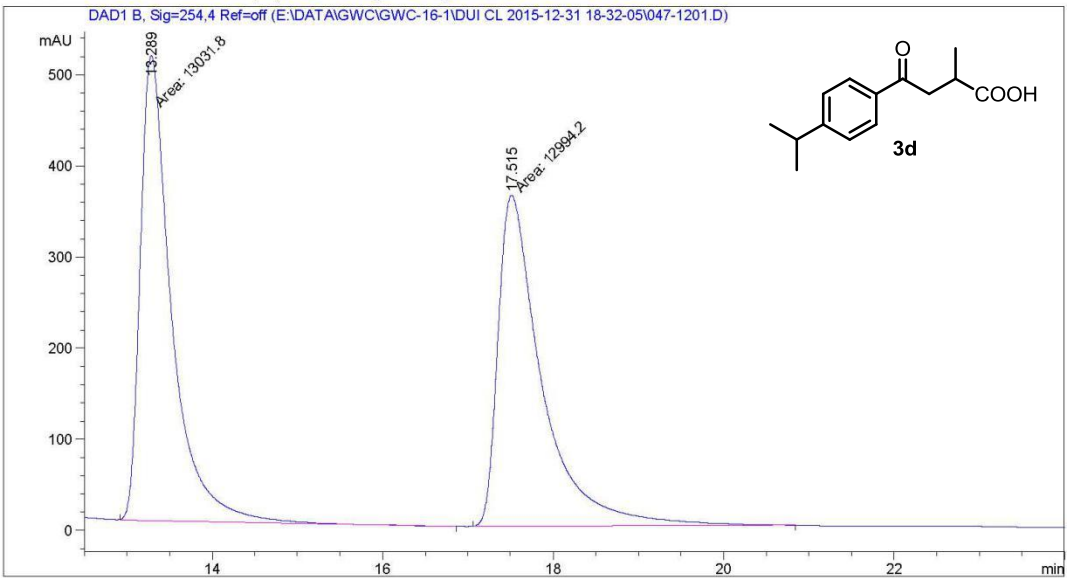
```
==========================================================================
Acq. Operator   : SYSTEM                        Seq. Line :  18
Acq. Instrument : 1260HPLC-DAD                  Location : Vial 73
Injection Date  : 3/31/2016 7:28:29 AM               Inj :   1
                                               Inj Volume : 1.000 µl
Acq. Method     : E:\DATA\HZY\HZY-2-NAI\HZY-2NAI-RAC 2016-03-30 20-31-11\DAD-OJ(1-6)-95-5-1ML
                  -ALL-90MIN.M
Last changed    : 3/30/2016 11:07:04 PM by SYSTEM
Analysis Method : E:\DATA\HZY\HZY-2-NAI\HZY-2NAI-RAC 2016-03-30 20-31-11\DAD-OJ(1-6)-95-5-1ML
                  -ALL-90MIN.M (Sequence Method)
Last changed    : 9/5/2016 9:31:12 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 E, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  46.260 MM    1.6337 5713.18262   58.28351 100.0000

Totals :                  5713.18262   58.28351
```

Data File E:\DATA\GWC\GWC-16-1\DUI CL 2015-12-31 18-32-05\047-1201.D
Sample Name: wsw-1-38-5

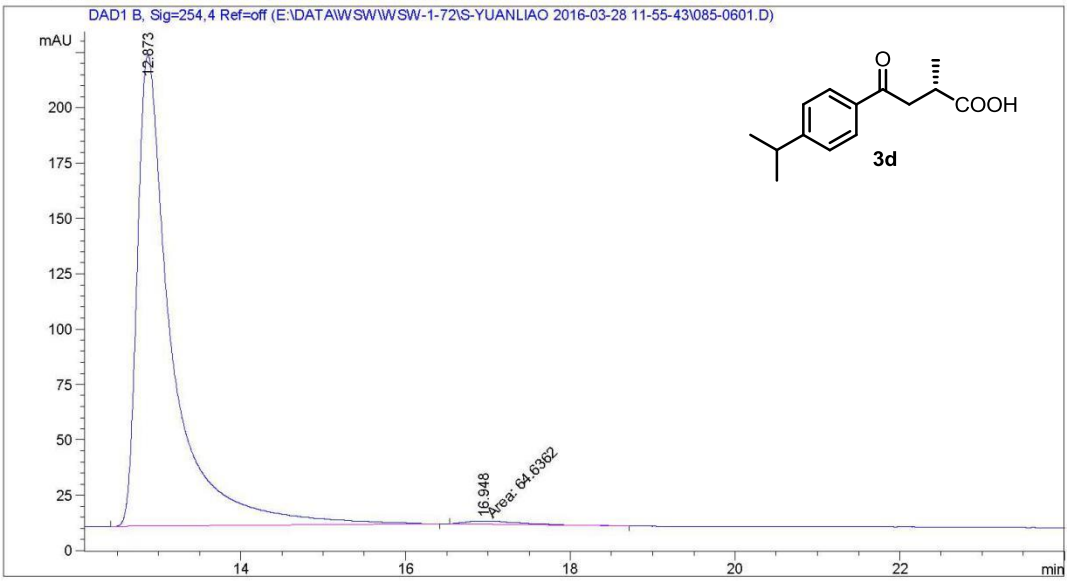
```
======================================================================
Acq. Operator   : SYSTEM                          Seq. Line :  12
Acq. Instrument : 1260HPLC-DAD                     Location : Vial 47
Injection Date  : 1/1/2016 12:28:37 AM                  Inj :   1
                                                  Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\GWC\GWC-16-1\DUI CL 2015-12-31 18-32-05\DAD-OJ(1-6)-95-5-1.0ML-ALL-
                  45MIN.M
Last changed    : 12/31/2015 7:06:20 PM by SYSTEM
Analysis Method : E:\DATA\GWC\GWC-16-1\DUI CL 2015-12-31 18-32-05\DAD-OJ(1-6)-95-5-1.0ML-ALL-
                  45MIN.M (Sequence Method)
Last changed    : 9/4/2016 5:08:38 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  13.289 MM    0.4254 1.30318e4   510.57037  50.0723
   2  17.515 MM    0.5967 1.29942e4   362.93323  49.9277

Totals :                  2.60260e4   873.50360
```

Data File E:\DATA\WSW\WSW-1-72\S-YUANLIAO 2016-03-28 11-55-43\085-0601.D
Sample Name: WSW-1-72-1-1

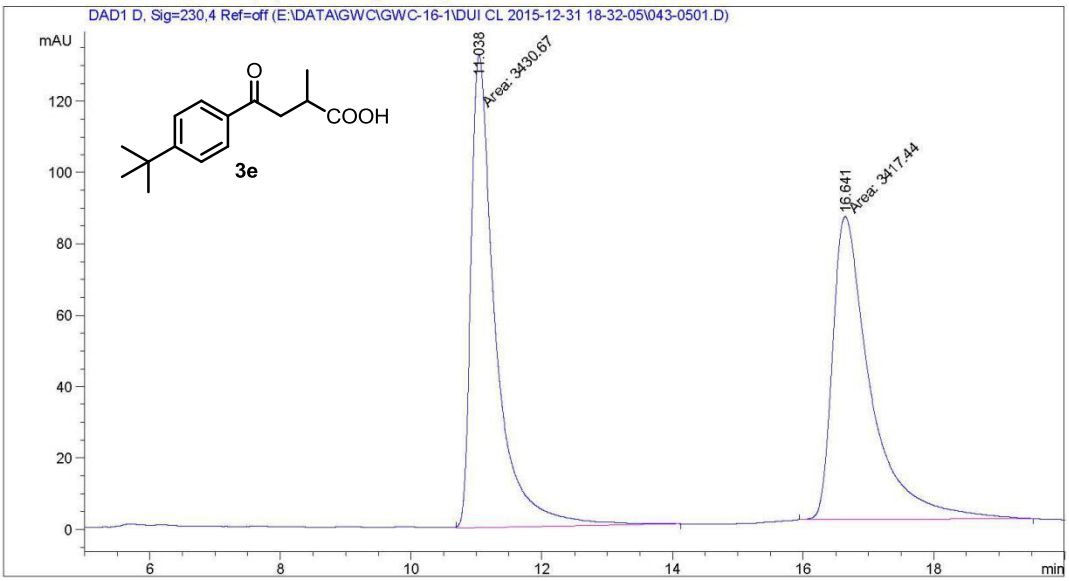
```
==========================================================================
Acq. Operator   : SYSTEM                          Seq. Line :   6
Acq. Instrument : 1260HPLC-DAD                    Location : Vial 85
Injection Date  : 3/28/2016 3:26:32 PM                 Inj :   1
                                                 Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\WSW\WSW-1-72\S-YUANLIAO 2016-03-28 11-55-43\DAD-OJ(1-6)-95-5-1.0ML-
                  ALL-45MIN.M
Last changed    : 3/28/2016 4:01:19 PM by SYSTEM
                  (modified after loading)
Analysis Method : E:\DATA\WSW\WSW-1-72\S-YUANLIAO 2016-03-28 11-55-43\DAD-OJ(1-6)-95-5-1.0ML-
                  ALL-45MIN.M (Sequence Method)
Last changed    : 9/5/2016 9:25:34 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  12.873 BB    0.4150 6215.80518  212.66885  98.9708
   2  16.948 MM    0.7306   64.63621    1.47440   1.0292

Totals :                  6280.44138  214.14326
```

Data File E:\DATA\GWC\GWC-16-1\DUI CL 2015-12-31 18-32-05\043-0501.D
Sample Name: wsw-1-38-2

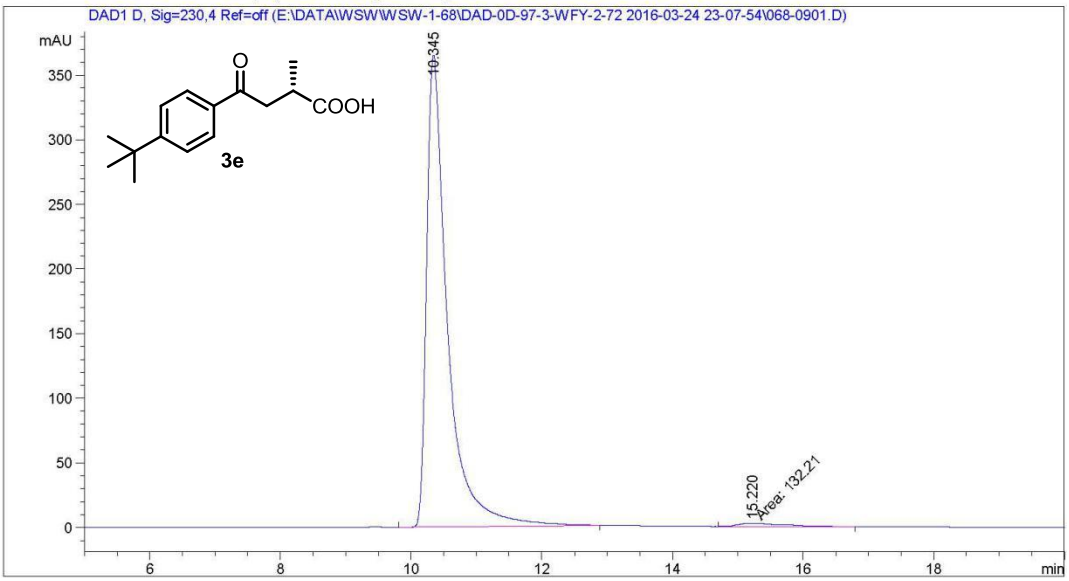
```
==========================================================================
Acq. Operator   : SYSTEM                          Seq. Line :   5
Acq. Instrument : 1260HPLC-DAD                     Location : Vial 43
Injection Date  : 12/31/2015 8:36:50 PM                 Inj :   1
                                                  Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\GWC\GWC-16-1\DUI CL 2015-12-31 18-32-05\DAD-OJ(1-6)-95-5-1.0ML-ALL-
                  45MIN.M
Last changed    : 12/31/2015 7:06:20 PM by SYSTEM
Analysis Method : E:\DATA\GWC\GWC-16-1\DUI CL 2015-12-31 18-32-05\DAD-OJ(1-6)-95-5-1.0ML-ALL-
                  45MIN.M (Sequence Method)
Last changed    : 9/5/2016 10:09:47 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 D, Sig=230,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  11.038 MM    0.4322 3430.66821  132.30887  50.0966
   2  16.641 MM    0.6717 3417.44312   84.79080  49.9034

Totals :                  6848.11133  217.09967
```

Data File E:\DATA\WSW\WSW-1-68\DAD-0D-97-3-WFY-2-72 2016-03-24 23-07-54\068-0901.D
Sample Name: wsw-1-68-8

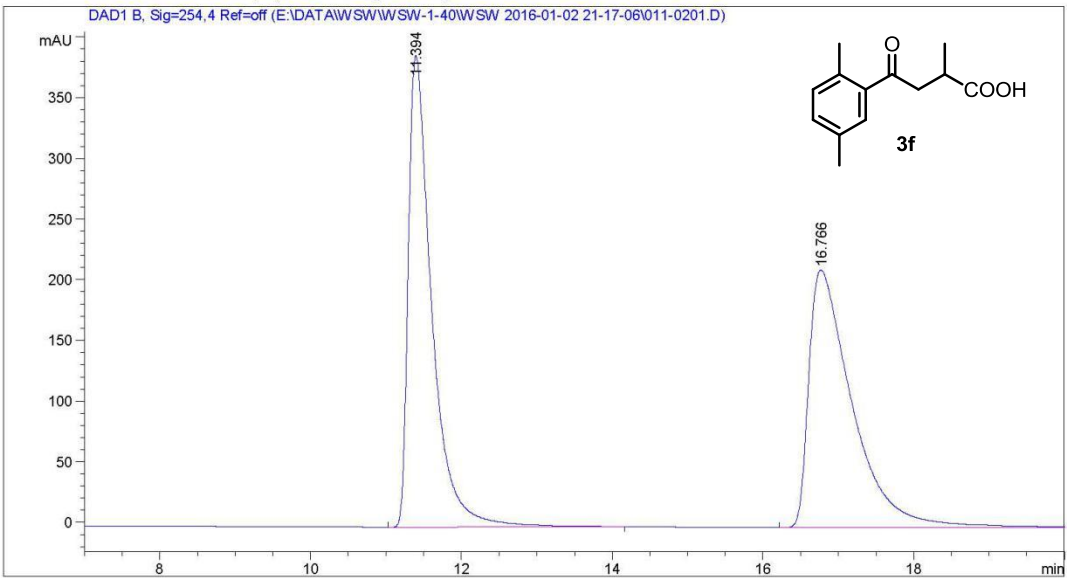
```
==========================================================================
Acq. Operator   : SYSTEM                          Seq. Line :   9
Acq. Instrument : 1260HPLC-DAD                   Location : Vial 68
Injection Date  : 3/25/2016 4:43:22 AM                Inj :   1
                                                 Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\WSW\WSW-1-68\DAD-0D-97-3-WFY-2-72 2016-03-24 23-07-54\DAD-OJ(1-6)-
                  95-5-1.0ML-ALL-45MIN.M
Last changed    : 3/24/2016 11:07:54 PM by SYSTEM
Analysis Method : E:\DATA\WSW\WSW-1-68\DAD-0D-97-3-WFY-2-72 2016-03-24 23-07-54\DAD-OJ(1-6)-
                  95-5-1.0ML-ALL-45MIN.M (Sequence Method)
Last changed    : 9/5/2016 9:35:03 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 D, Sig=230,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  10.345 BB    0.3224 8082.22900  364.90659  98.3905
   2  15.220 MM    0.8103  132.21048    2.71943   1.6095

Totals :                  8214.43948  367.62602
```

Data File E:\DATA\WSW\WSW-1-40\WSW 2016-01-02 21-17-06\011-0201.D
Sample Name: wsw-1-40-1

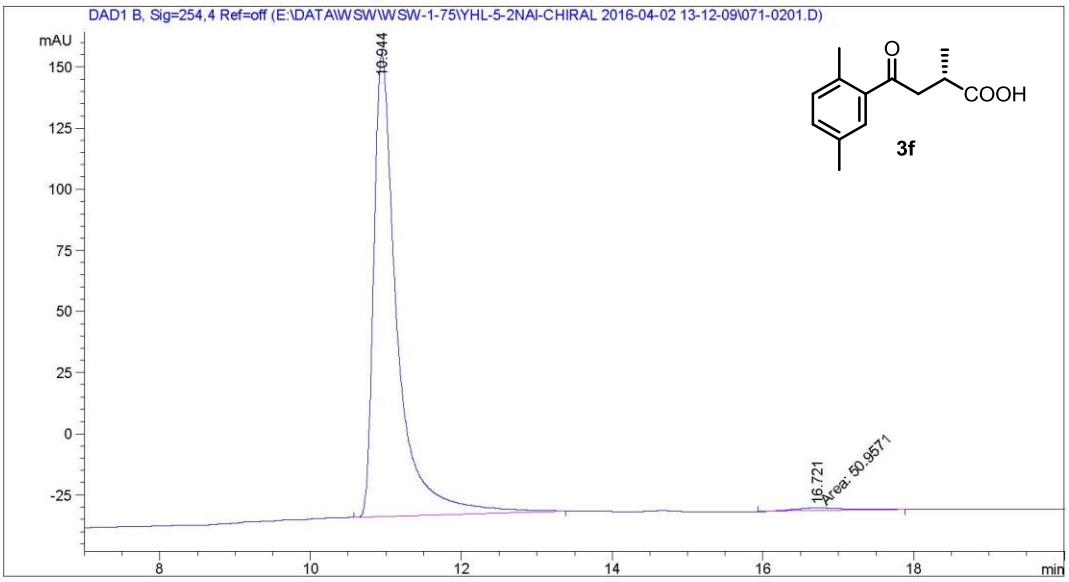
```
=====================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   2
Acq. Instrument : 1260HPLC-DAD                   Location : Vial 11
Injection Date  : 1/2/2016 9:29:43 PM                 Inj :   1
                                                Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\WSW\WSW-1-40\WSW 2016-01-02 21-17-06\DAD-OJ(1-6)-95-5-1.0ML-ALL-
                  45MIN.M
Last changed    : 1/2/2016 9:17:06 PM by SYSTEM
Analysis Method : E:\DATA\WSW\WSW-1-40\WSW 2016-01-02 21-17-06\DAD-OJ(1-6)-95-5-1.0ML-ALL-
                  45MIN.M (Sequence Method)
Last changed    : 9/3/2016 7:14:43 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
=====================================================================
                        Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  11.394 BB    0.3196 8385.75000  388.85330  49.6792
   2  16.766 BB    0.5895 8494.04297  211.98248  50.3208

Totals :                 1.68798e4   600.83578
```

Data File E:\DATA\WSW\WSW-1-75\YHL-5-2NAI-CHIRAL 2016-04-02 13-12-09\071-0201.D
Sample Name: WSW-1-75-4

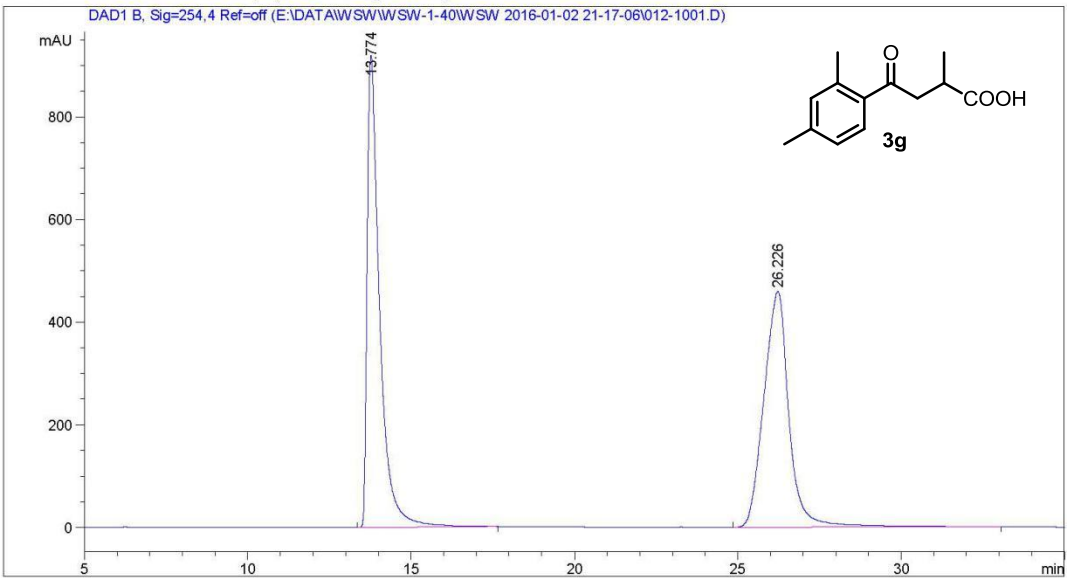
```
Acq. Operator   : SYSTEM                          Seq. Line :   2
Acq. Instrument : 1260HPLC-DAD                     Location : Vial 71
Injection Date  : 4/2/2016 1:29:04 PM                  Inj :   1
                                                  Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\WSW\WSW-1-75\YHL-5-2NAI-CHIRAL 2016-04-02 13-12-09\DAD-OJ(1-6)-95-5
                  -1.0ML-ALL-45MIN.M
Last changed    : 4/2/2016 2:07:23 PM by SYSTEM
                  (modified after loading)
Analysis Method : E:\DATA\WSW\WSW-1-75\YHL-5-2NAI-CHIRAL 2016-04-02 13-12-09\DAD-OJ(1-6)-95-5
                  -1.0ML-ALL-45MIN.M (Sequence Method)
Last changed    : 2/10/2017 8:57:02 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
=====================================================================
                        Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  10.944 BB    0.3162 4077.70337  188.63388  98.7658
   2  16.721 MM    0.8238   50.95708    1.03097   1.2342

Totals :                  4128.66045  189.66485
```

Data File E:\DATA\WSW\WSW-1-40\WSW 2016-01-02 21-17-06\012-1001.D
Sample Name: wsw-1-40-2

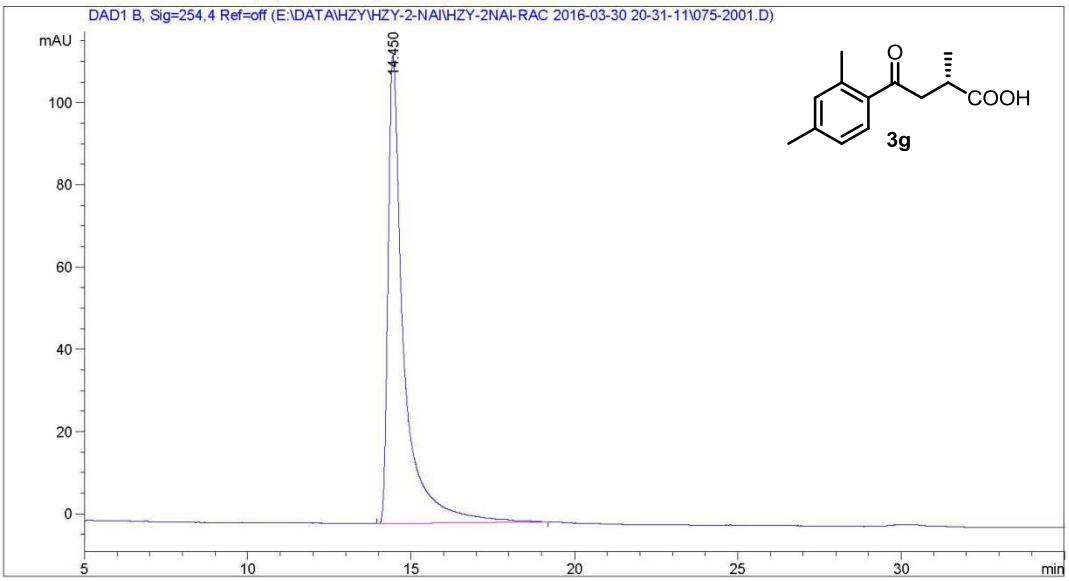
```
==========================================================================
Acq. Operator   : SYSTEM                          Seq. Line :  10
Acq. Instrument : 1260HPLC-DAD                    Location : Vial 12
Injection Date  : 1/2/2016 11:37:28 PM                 Inj :   1
                                                  Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\WSW\WSW-1-40\WSW 2016-01-02 21-17-06\DAD-OJ(1-6)-95-5-1.0ML-ALL-
                  45MIN.M
Last changed    : 1/2/2016 9:17:06 PM by SYSTEM
Analysis Method : E:\DATA\WSW\WSW-1-40\WSW 2016-01-02 21-17-06\DAD-OJ(1-6)-95-5-1.0ML-ALL-
                  45MIN.M (Sequence Method)
Last changed    : 9/3/2016 8:01:05 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  13.774 BB    0.3882 2.42904e4   919.49597  49.7527
   2  26.226 BB    0.8077 2.45319e4   458.88672  50.2473

Totals :                  4.88224e4  1378.38269
```

Data File E:\DATA\HZY\HZY-2-NAI\HZY-2NAI-RAC 2016-03-30 20-31-11\075-2001.D
Sample Name: wsw-1-75-5

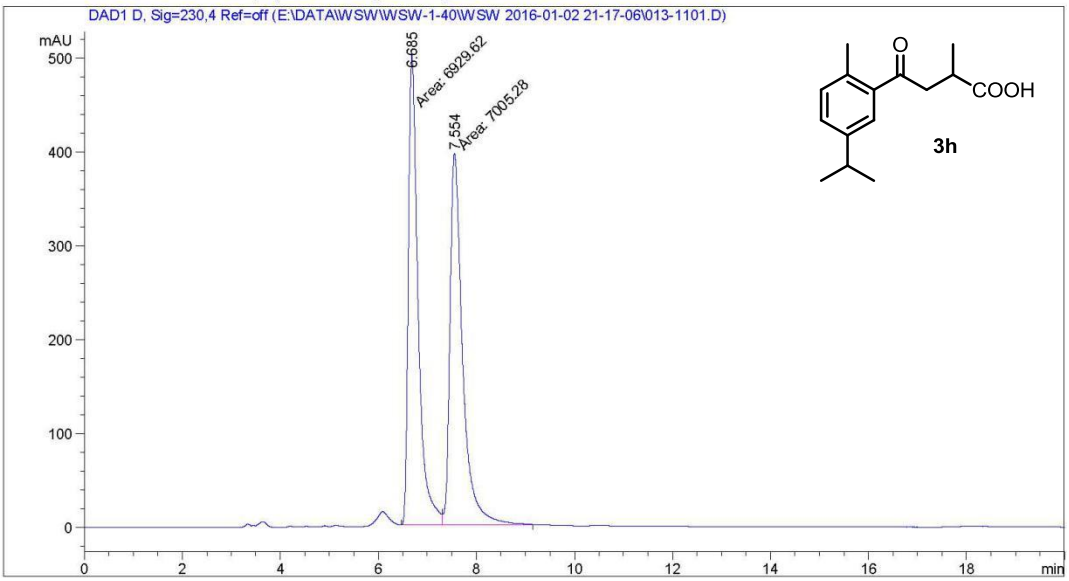
```
==================================================================
Acq. Operator   : SYSTEM                        Seq. Line :  20
Acq. Instrument : 1260HPLC-DAD                   Location : Vial 75
Injection Date  : 3/31/2016 9:45:35 AM                Inj :   1
                                                Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\HZY\HZY-2-NAI\HZY-2NAI-RAC 2016-03-30 20-31-11\DAD-OJ(1-6)-95-5-1.
                  0ML-ALL-45MIN.M
Last changed    : 3/30/2016 10:58:50 PM by SYSTEM
Analysis Method : E:\DATA\HZY\HZY-2-NAI\HZY-2NAI-RAC 2016-03-30 20-31-11\DAD-OJ(1-6)-95-5-1.
                  0ML-ALL-45MIN.M (Sequence Method)
Last changed    : 9/3/2016 8:02:21 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  14.450 BB    0.4801 3828.53345  113.91030 100.0000

Totals :                  3828.53345  113.91030
```

Data File E:\DATA\WSW\WSW-1-40\WSW 2016-01-02 21-17-06\013-1101.D
Sample Name: wsw-1-40-3

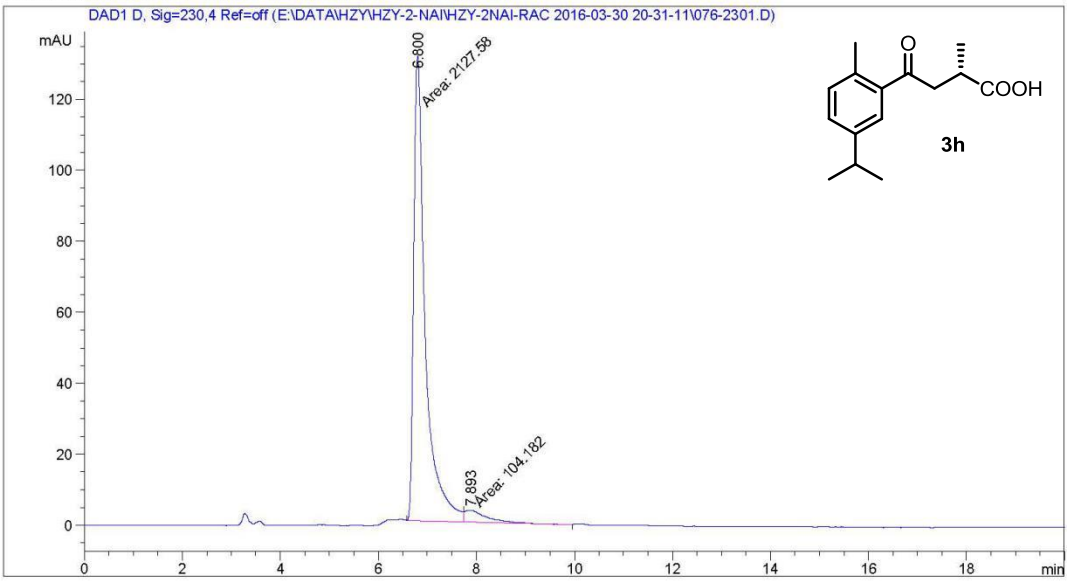
```
==========================================================================
Acq. Operator   : SYSTEM                          Seq. Line :  11
Acq. Instrument : 1260HPLC-DAD                    Location : Vial 13
Injection Date  : 1/3/2016 12:23:23 AM                 Inj :   1
                                                  Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\WSW\WSW-1-40\WSW 2016-01-02 21-17-06\DAD-OJ(1-6)-95-5-1.0ML-ALL-
                  45MIN.M
Last changed    : 1/2/2016 9:17:06 PM by SYSTEM
Analysis Method : E:\DATA\WSW\WSW-1-40\WSW 2016-01-02 21-17-06\DAD-OJ(1-6)-95-5-1.0ML-ALL-
                  45MIN.M (Sequence Method)
Last changed    : 9/4/2016 4:16:17 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 D, Sig=230,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   6.685 MF    0.2310 6929.62451  499.92773  49.7285
   2   7.554 FM    0.2955 7005.27734  395.04926  50.2715

Totals :                  1.39349e4   894.97699
```

Data File E:\DATA\HZY\HZY-2-NAI\HZY-2NAI-RAC 2016-03-30 20-31-11\076-2301.D
Sample Name: wsw-1-75-6

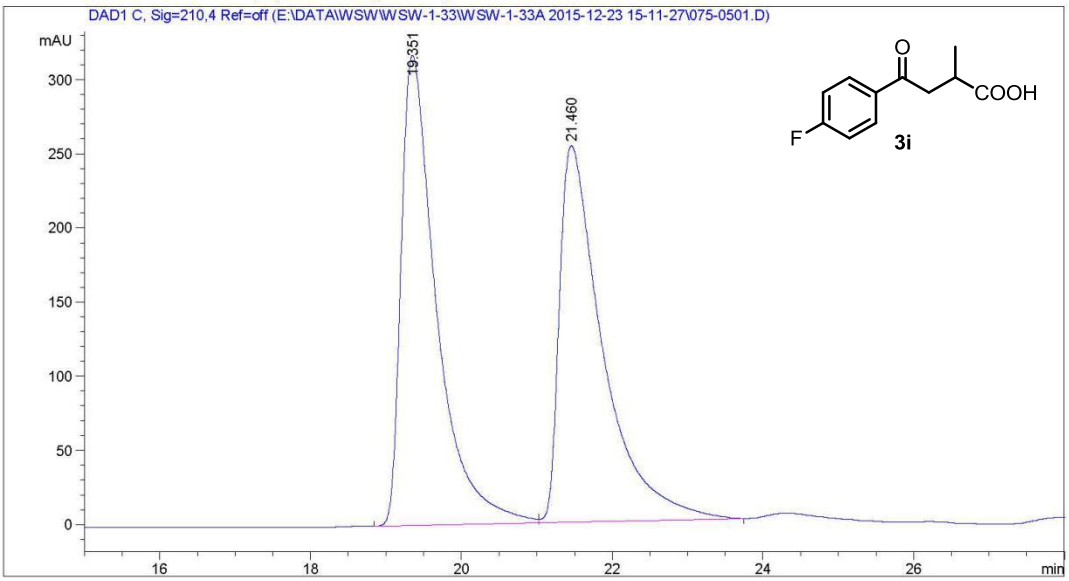
```
==================================================================
Acq. Operator   : SYSTEM                        Seq. Line :  23
Acq. Instrument : 1260HPLC-DAD                   Location : Vial 76
Injection Date  : 3/31/2016 1:34:00 PM                Inj :   1
                                                Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\HZY\HZY-2-NAI\HZY-2NAI-RAC 2016-03-30 20-31-11\DAD-OJ(1-6)-95-5-1.
                  0ML-ALL-45MIN.M
Last changed    : 3/31/2016 1:46:38 PM by SYSTEM
                  (modified after loading)
Analysis Method : E:\DATA\HZY\HZY-2-NAI\HZY-2NAI-RAC 2016-03-30 20-31-11\DAD-OJ(1-6)-95-5-1.
                  0ML-ALL-45MIN.M (Sequence Method)
Last changed    : 9/4/2016 4:08:40 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 D, Sig=230,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   6.800 MF    0.2703 2127.58252  131.17767  95.3318
   2   7.893 FM    0.5215  104.18213    3.32926   4.6682

Totals :                  2231.76465  134.50693
```

Data File E:\DATA\WSW\WSW-1-33\WSW-1-33A 2015-12-23 15-11-27\075-0501.D
Sample Name: WSW-1-33-5

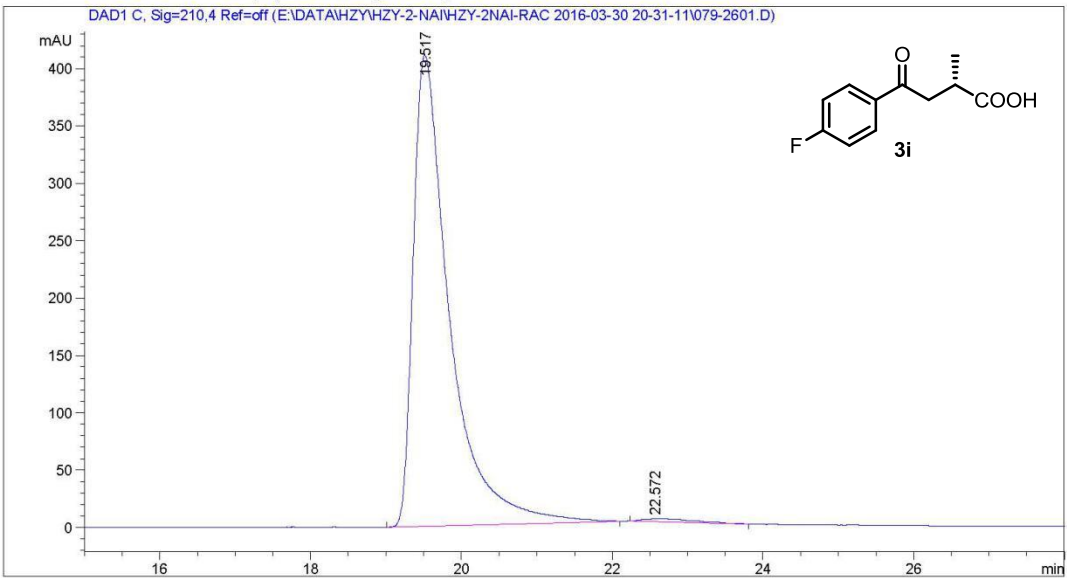
```
======================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   5
Acq. Instrument : 1260HPLC-DAD                   Location : Vial 75
Injection Date  : 12/23/2015 6:16:07 PM               Inj :   1
                                                Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\WSW\WSW-1-33\WSW-1-33A 2015-12-23 15-11-27\DAD-OJ(1-6)-95-5-1.0ML-
                  ALL-45MIN.M
Last changed    : 12/23/2015 3:11:27 PM by SYSTEM
Analysis Method : E:\DATA\WSW\WSW-1-33\WSW-1-33A 2015-12-23 15-11-27\DAD-OJ(1-6)-95-5-1.0ML-
                  ALL-45MIN.M (Sequence Method)
Last changed    : 9/5/2016 10:20:38 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  19.351 BV    0.4783 1.02811e4   317.07803  50.3718
   2  21.460 VB    0.5774 1.01293e4   253.94756  49.6282

Totals :                  2.04105e4   571.02559
```

Data File E:\DATA\HZY\HZY-2-NAI\HZY-2NAI-RAC 2016-03-30 20-31-11\079-2601.D
Sample Name: wsw-1-75-9

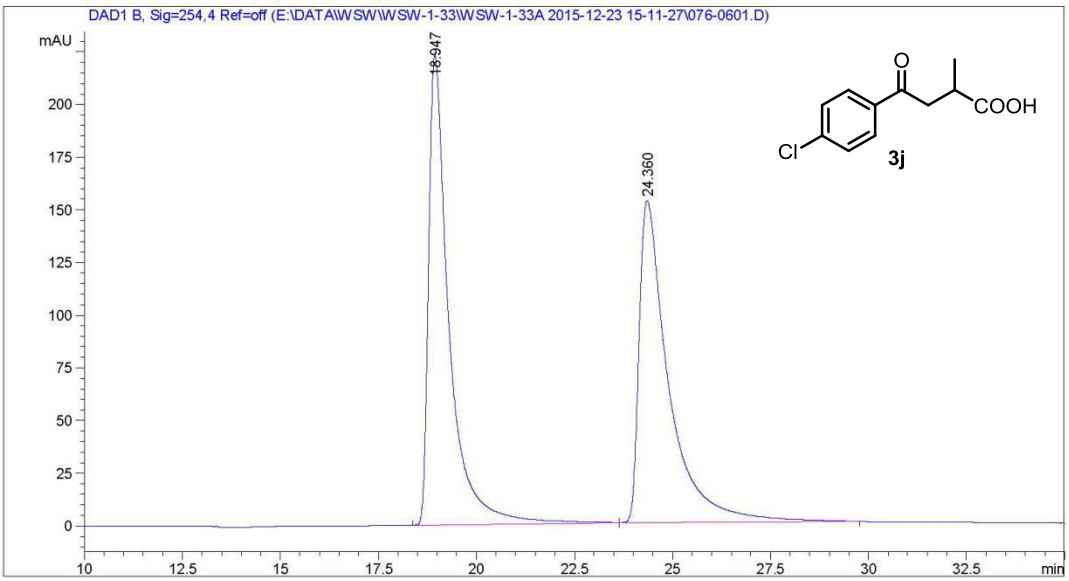
```
==================================================================
Acq. Operator   : SYSTEM                        Seq. Line :  26
Acq. Instrument : 1260HPLC-DAD                   Location : Vial 79
Injection Date  : 3/31/2016 3:52:05 PM                Inj :   1
                                                Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\HZY\HZY-2-NAI\HZY-2NAI-RAC 2016-03-30 20-31-11\DAD-OJ(1-6)-95-5-1.
                  0ML-ALL-45MIN.M
Last changed    : 3/31/2016 1:46:38 PM by SYSTEM
Analysis Method : E:\DATA\HZY\HZY-2-NAI\HZY-2NAI-RAC 2016-03-30 20-31-11\DAD-OJ(1-6)-95-5-1.
                  0ML-ALL-45MIN.M (Sequence Method)
Last changed    : 9/5/2016 10:18:37 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  19.517 BB    0.4873 1.37680e4   410.51605  99.0316
   2  22.572 BB    0.5940  134.63878    2.69914   0.9684

Totals :                  1.39027e4   413.21520
```

Data File E:\DATA\WSW\WSW-1-33\WSW-1-33A 2015-12-23 15-11-27\076-0601.D
Sample Name: WSW-1-33-6

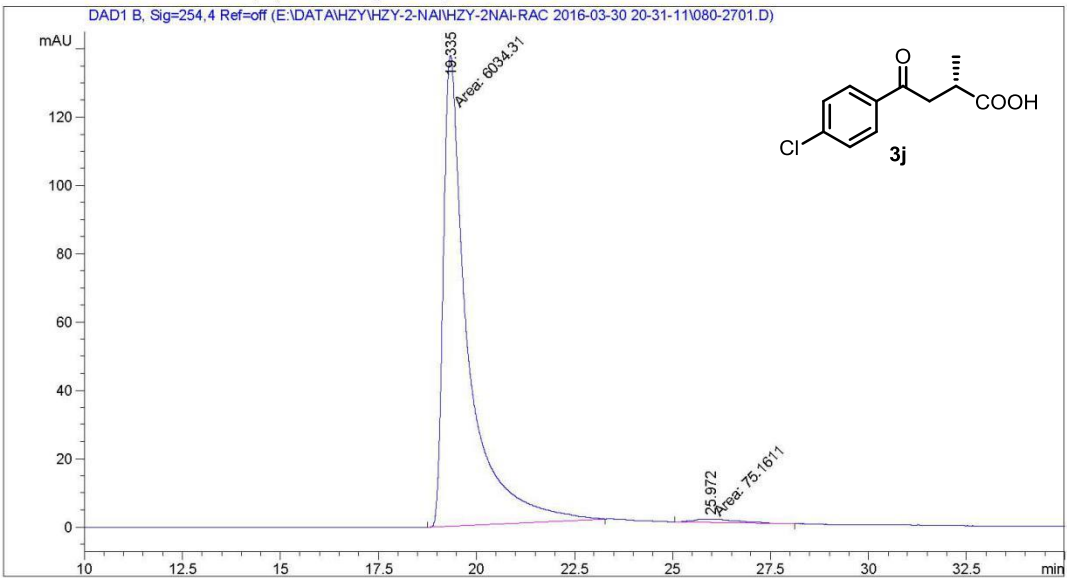
```
==========================================================================
Acq. Operator   : SYSTEM                          Seq. Line :   6
Acq. Instrument : 1260HPLC-DAD                    Location : Vial 76
Injection Date  : 12/23/2015 7:02:06 PM               Inj :   1
                                                  Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\WSW\WSW-1-33\WSW-1-33A 2015-12-23 15-11-27\DAD-OJ(1-6)-95-5-1.0ML-
                  ALL-45MIN.M
Last changed    : 12/23/2015 3:11:27 PM by SYSTEM
Analysis Method : E:\DATA\WSW\WSW-1-33\WSW-1-33A 2015-12-23 15-11-27\DAD-OJ(1-6)-95-5-1.0ML-
                  ALL-45MIN.M (Sequence Method)
Last changed    : 9/5/2016 10:32:49 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  18.947 BB    0.5248 8079.64014  222.92558  50.2066
   2  24.360 BB    0.7503 8013.15137  152.68410  49.7934

Totals :                 1.60928e4   375.60968
```

Data File E:\DATA\HZY\HZY-2-NAI\HZY-2NAI-RAC 2016-03-30 20-31-11\080-2701.D
Sample Name: wsw-1-75-10

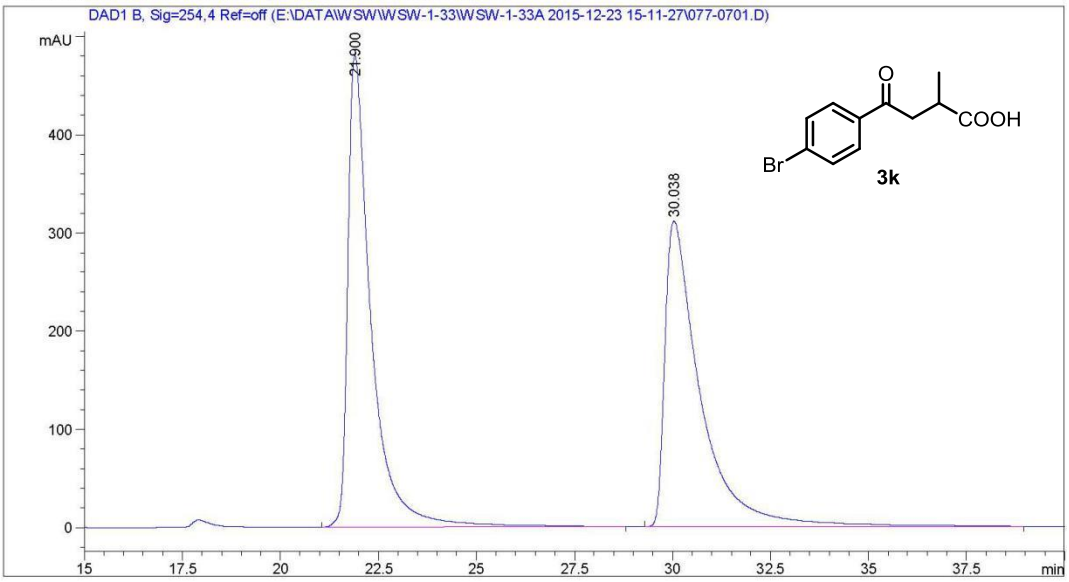
```
==========================================================================
Acq. Operator   : SYSTEM                          Seq. Line :  27
Acq. Instrument : 1260HPLC-DAD                    Location : Vial 80
Injection Date  : 3/31/2016 4:38:07 PM                 Inj :  1
                                                  Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\HZY\HZY-2-NAI\HZY-2NAI-RAC 2016-03-30 20-31-11\DAD-OJ(1-6)-95-5-1.
                  0ML-ALL-45MIN.M
Last changed    : 3/31/2016 1:46:38 PM by SYSTEM
Analysis Method : E:\DATA\HZY\HZY-2-NAI\HZY-2NAI-RAC 2016-03-30 20-31-11\DAD-OJ(1-6)-95-5-1.
                  0ML-ALL-45MIN.M (Sequence Method)
Last changed    : 9/5/2016 10:31:01 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                          Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  19.335 MM    0.7295 6034.31055  137.85857  98.7698
   2  25.972 MM    1.3019   75.16106 9.62180e-1   1.2302

Totals :                  6109.47161  138.82075
```

Data File E:\DATA\WSW\WSW-1-33\WSW-1-33A 2015-12-23 15-11-27\077-0701.D
Sample Name: WSW-1-33-7

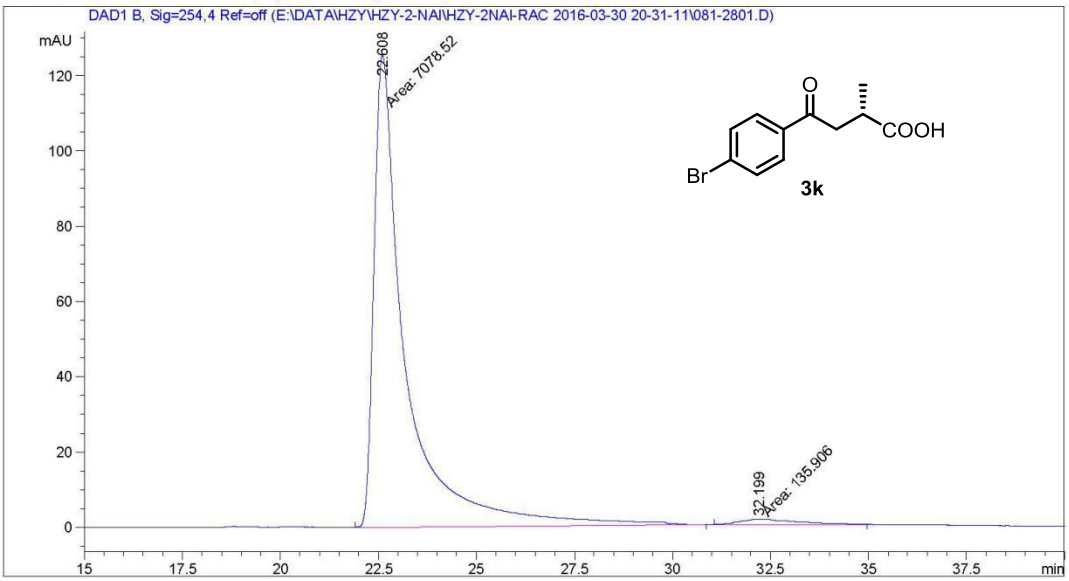
```
==========================================================================
Acq. Operator   : SYSTEM                          Seq. Line :   7
Acq. Instrument : 1260HPLC-DAD                    Location : Vial 77
Injection Date  : 12/23/2015 7:48:05 PM               Inj :   1
                                                  Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\WSW\WSW-1-33\WSW-1-33A 2015-12-23 15-11-27\DAD-OJ(1-6)-95-5-1.0ML-
                  ALL-45MIN.M
Last changed    : 12/23/2015 8:27:34 PM by SYSTEM
                  (modified after loading)
Analysis Method : E:\DATA\WSW\WSW-1-33\WSW-1-33A 2015-12-23 15-11-27\DAD-OJ(1-6)-95-5-1.0ML-
                  ALL-45MIN.M (Sequence Method)
Last changed    : 9/5/2016 10:41:11 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  21.900 BB    0.5987 1.97965e4   480.42813  50.3200
   2  30.038 BB    0.8950 1.95447e4   311.06342  49.6800

Totals :                  3.93412e4   791.49155
```

Data File E:\DATA\HZY\HZY-2-NAI\HZY-2NAI-RAC 2016-03-30 20-31-11\081-2801.D
Sample Name: wsw-1-75-11

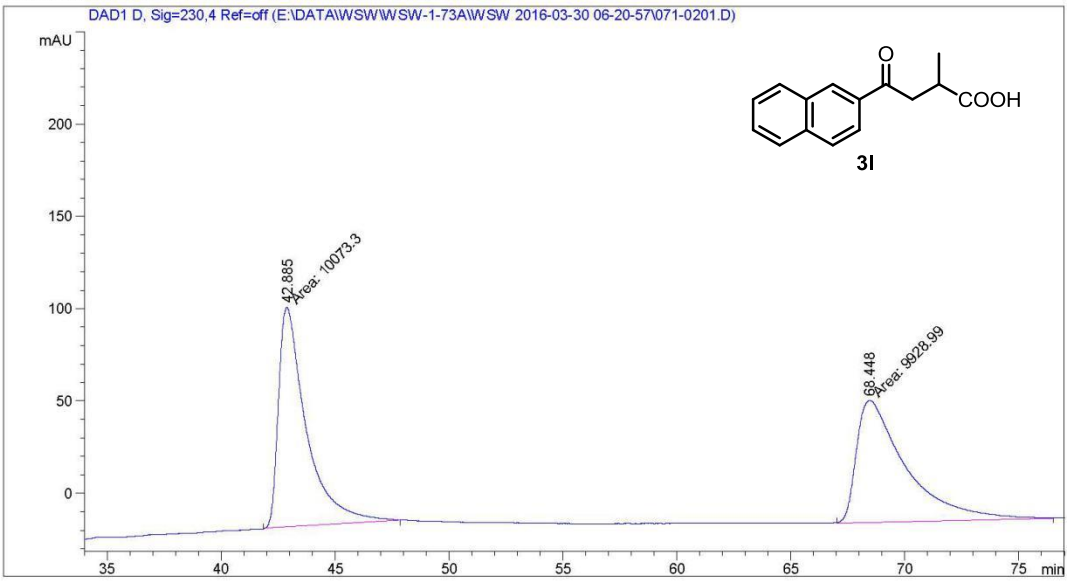
```
==================================================================
Acq. Operator   : SYSTEM                        Seq. Line :  28
Acq. Instrument : 1260HPLC-DAD                   Location : Vial 81
Injection Date  : 3/31/2016 5:24:09 PM                Inj :   1
                                                Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\HZY\HZY-2-NAI\HZY-2NAI-RAC 2016-03-30 20-31-11\DAD-OJ(1-6)-95-5-1.
                  0ML-ALL-45MIN.M
Last changed    : 3/31/2016 1:46:38 PM by SYSTEM
Analysis Method : E:\DATA\HZY\HZY-2-NAI\HZY-2NAI-RAC 2016-03-30 20-31-11\DAD-OJ(1-6)-95-5-1.
                  0ML-ALL-45MIN.M (Sequence Method)
Last changed    : 9/5/2016 10:39:23 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=254,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  22.608 MM    0.9406 7078.52197  125.42750  98.1162
   2  32.199 MM    1.7115  135.90642    1.32345   1.8838

Totals :                  7214.42839  126.75095
```

Data File E:\DATA\WSW\WSW-1-73A\WSW 2016-03-30 06-20-57\071-0201.D
Sample Name: WSW-1-73-1

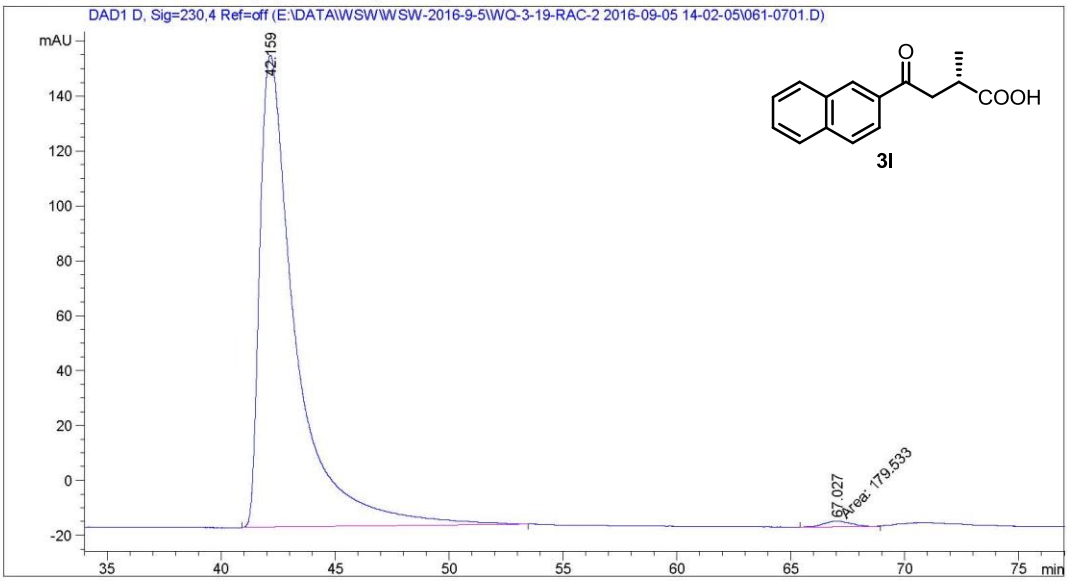
```
=====================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   2
Acq. Instrument : 1260HPLC-DAD                   Location : Vial 71
Injection Date  : 3/30/2016 6:32:48 AM               Inj :   1
                                                Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\WSW\WSW-1-73A\WSW 2016-03-30 06-20-57\DAD-OJ(1-6)-95-5-1.0ML-ALL-
                  45MIN.M
Last changed    : 3/30/2016 6:42:23 AM by SYSTEM
                  (modified after loading)
Analysis Method : E:\DATA\WSW\WSW-1-73A\WSW 2016-03-30 06-20-57\DAD-OJ(1-6)-95-5-1.0ML-ALL-
                  45MIN.M (Sequence Method)
Last changed    : 9/5/2016 8:30:45 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
=====================================================================
                        Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 D, Sig=230,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  42.885 MM    1.4131 1.00733e4   118.81017  50.3606
   2  68.448 MM    2.5070 9928.98828   66.00892  49.6394

Totals :                  2.00022e4   184.81909
```

Data File E:\DATA\WSW\WSW-2016-9-5\WQ-3-19-RAC-2 2016-09-05 14-02-05\061-0701.D
Sample Name: wsw-1-175-12

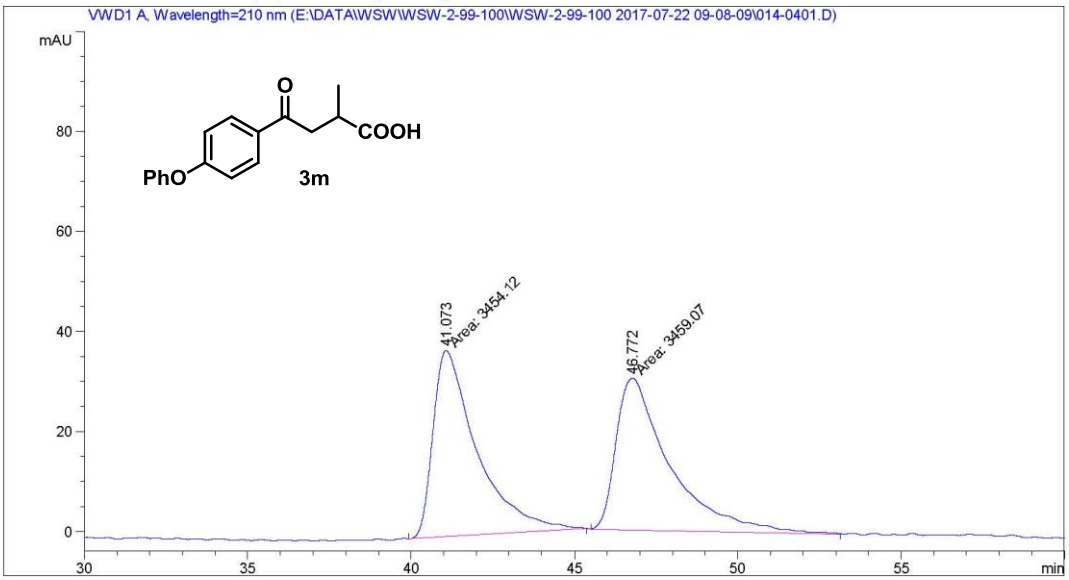
```
==================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   7
Acq. Instrument : 1260HPLC-DAD                  Location : Vial 61
Injection Date  : 9/5/2016 4:14:58 PM               Inj :   1
                                               Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\WSW\WSW-2016-9-5\WQ-3-19-RAC-2 2016-09-05 14-02-05\DAD-OJ(1-6)-95-5
                  -1.0ML-ALL-45MIN.M
Last changed    : 9/5/2016 4:33:49 PM by SYSTEM
                  (modified after loading)
Analysis Method : E:\DATA\WSW\WSW-2016-9-5\WQ-3-19-RAC-2 2016-09-05 14-02-05\DAD-OJ(1-6)-95-5
                  -1.0ML-ALL-45MIN.M (Sequence Method)
Last changed    : 2/10/2017 9:05:17 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 D, Sig=230,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  42.159 BB    1.5528 1.87795e4   171.85948  99.0530
   2  67.027 MM    1.4412  179.53259    2.07624   0.9470

Totals :                  1.89590e4   173.93572
```

Data File E:\DATA\WSW\WSW-2-99-100\WSW-2-99-100 2017-07-22 09-08-09\014-0401.D
Sample Name: WSW-2-100-1

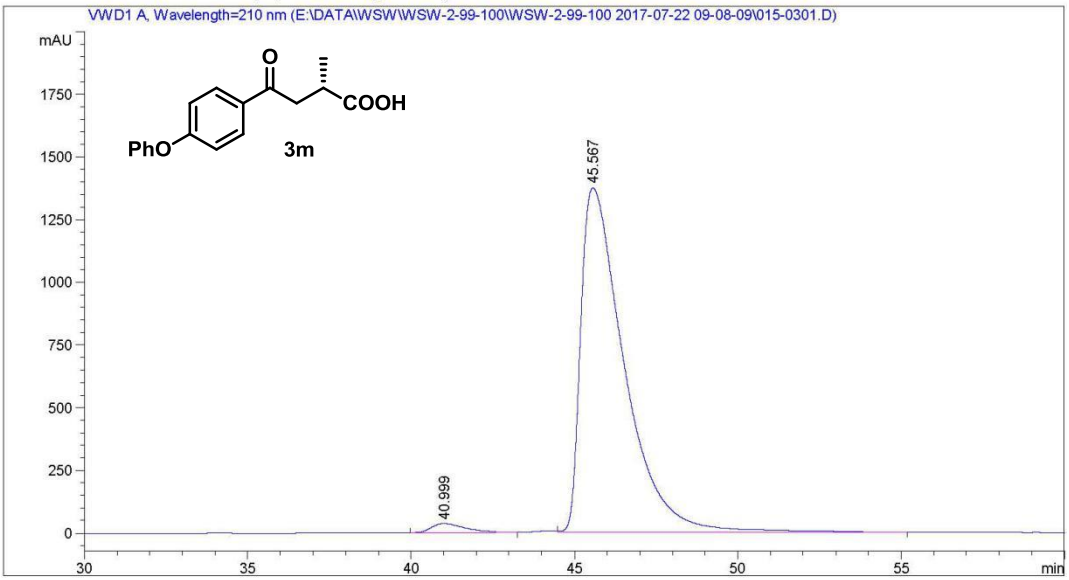
```
======================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   4
Acq. Instrument : 1260HPLC-VWD                  Location : Vial 14
Injection Date  : 7/22/2017 11:21:24 AM              Inj :   1
                                               Inj Volume : 10.000 µl
Acq. Method     : E:\DATA\WSW\WSW-2-99-100\WSW-2-99-100 2017-07-22 09-08-09\VWD-AD(1-2)-95-5-
                  1ML-10UL-210NM-60MIN.M
Last changed    : 7/22/2017 9:08:09 AM by SYSTEM
Analysis Method : E:\DATA\WSW\WSW-2-99-100\WSW-2-99-100 2017-07-22 09-08-09\VWD-AD(1-2)-95-5-
                  1ML-10UL-210NM-60MIN.M (Sequence Method)
Last changed    : 7/22/2017 4:17:36 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=210 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  41.073 MM    1.5479 3454.11890   37.19158  49.9642
   2  46.772 MM    1.8979 3459.06543   30.37634  50.0358

Totals :                 6913.18433   67.56792
```

Data File E:\DATA\WSW\WSW-2-99-100\WSW-2-99-100 2017-07-22 09-08-09\015-0301.D
Sample Name: WSW-2-99-2

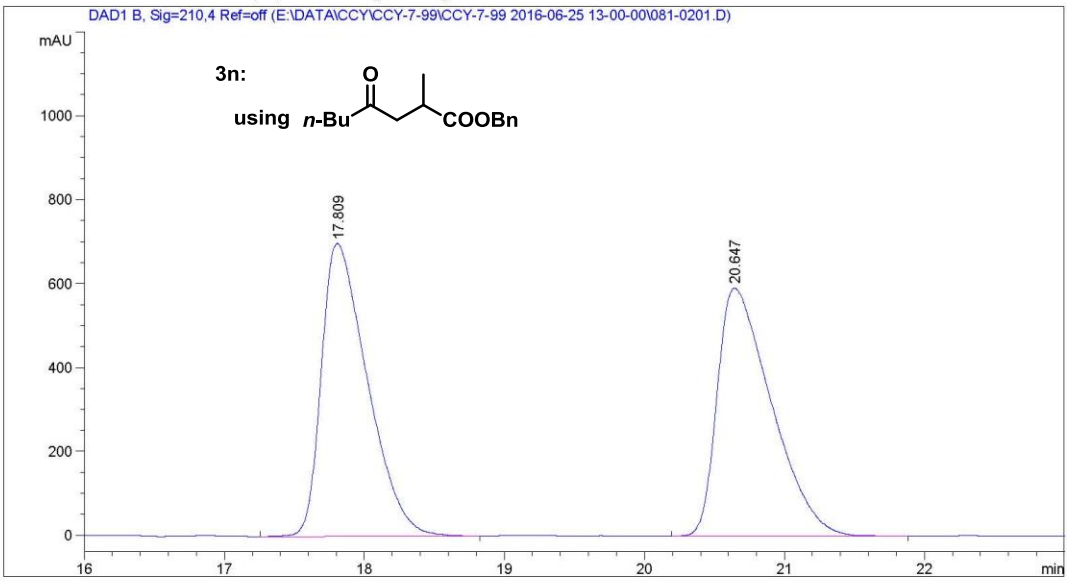
```
=====================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   3
Acq. Instrument : 1260HPLC-VWD                   Location : Vial 15
Injection Date  : 7/22/2017 10:20:35 AM              Inj :   1
                                                Inj Volume : 10.000 µl
Acq. Method     : E:\DATA\WSW\WSW-2-99-100\WSW-2-99-100 2017-07-22 09-08-09\VWD-AD(1-2)-95-5-
                  1ML-10UL-210NM-60MIN.M
Last changed    : 7/22/2017 9:08:09 AM by SYSTEM
Analysis Method : E:\DATA\WSW\WSW-2-99-100\WSW-2-99-100 2017-07-22 09-08-09\VWD-AD(1-2)-95-5-
                  1ML-10UL-210NM-60MIN.M (Sequence Method)
Last changed    : 7/22/2017 4:16:05 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
=====================================================================
                        Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=210 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  40.999 BB    1.0526 2599.48291   36.55566   2.0022
   2  45.567 VB    1.3483 1.27229e5  1373.98499  97.9978

Totals :                 1.29828e5  1410.54065
```

Data File E:\DATA\CCY\CCY-7-99\CCY-7-99 2016-06-25 13-00-00\081-0201.D
Sample Name: ccy-7-99-1

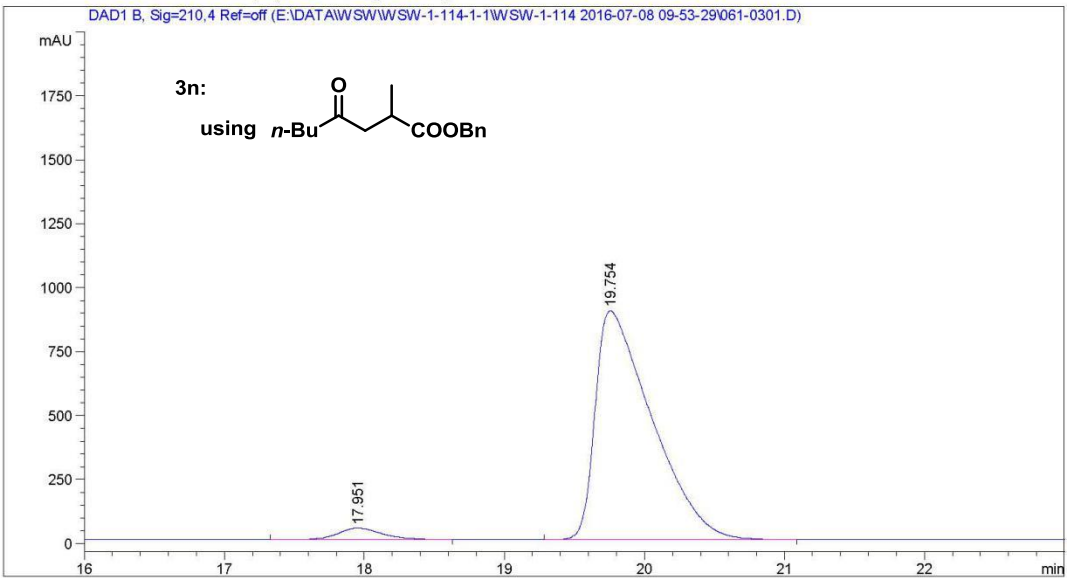
```
==========================================================================
Acq. Operator   : SYSTEM                          Seq. Line :   2
Acq. Instrument : 1260HPLC-DAD                    Location : Vial 81
Injection Date  : 6/25/2016 1:11:56 PM                 Inj :   1
                                                  Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\CCY\CCY-7-99\CCY-7-99 2016-06-25 13-00-00\DAD-OJ(1-6)-99-1-1.0ML-
                  5UL-45MIN-ALL.M
Last changed    : 6/25/2016 1:00:00 PM by SYSTEM
Analysis Method : E:\DATA\CCY\CCY-7-99\CCY-7-99 2016-06-25 13-00-00\DAD-OJ(1-6)-99-1-1.0ML-
                  5UL-45MIN-ALL.M (Sequence Method)
Last changed    : 9/5/2016 8:52:56 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=210,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  17.809 BB    0.3456 1.58933e4   698.08386  50.0459
   2  20.647 BB    0.4090 1.58641e4   591.29150  49.9541

Totals :                  3.17575e4  1289.37537
```

Data File E:\DATA\WSW\WSW-1-114-1-1\WSW-1-114 2016-07-08 09-53-29\061-0301.D
Sample Name: WSW-1-114-1-1A

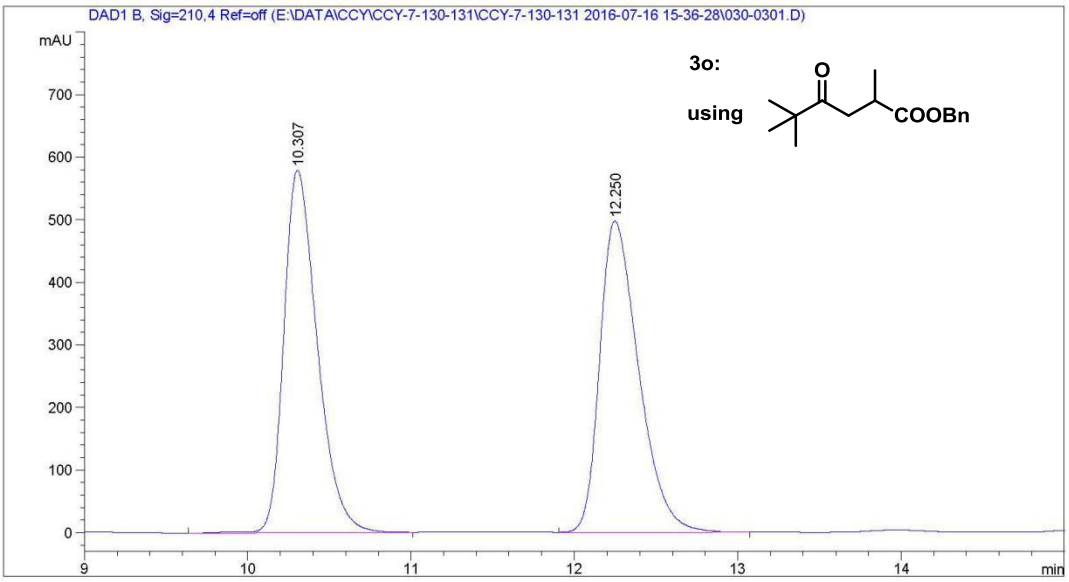
```
=====================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   3
Acq. Instrument : 1260HPLC-DAD                  Location : Vial 61
Injection Date  : 7/8/2016 10:44:42 AM               Inj :   1
                                               Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\WSW\WSW-1-114-1-1\WSW-1-114 2016-07-08 09-53-29\DAD-OJ(1-6)-99-1-1.
                  0ML-5UL-45MIN-ALL.M
Last changed    : 7/8/2016 10:43:44 AM by SYSTEM
Analysis Method : E:\DATA\WSW\WSW-1-114-1-1\WSW-1-114 2016-07-08 09-53-29\DAD-OJ(1-6)-99-1-1.
                  0ML-5UL-45MIN-ALL.M (Sequence Method)
Last changed    : 9/5/2016 8:48:49 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
=====================================================================
                        Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=210,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  17.951 BB    0.3263  965.45789   44.64465   3.7097
   2  19.754 BB    0.4153 2.50601e4   893.32635  96.2903

Totals :                  2.60256e4   937.97100
```

Data File E:\DATA\CCY\CCY-7-130-131\CCY-7-130-131 2016-07-16 15-36-28\030-0301.D
Sample Name: CCY-7-131-2

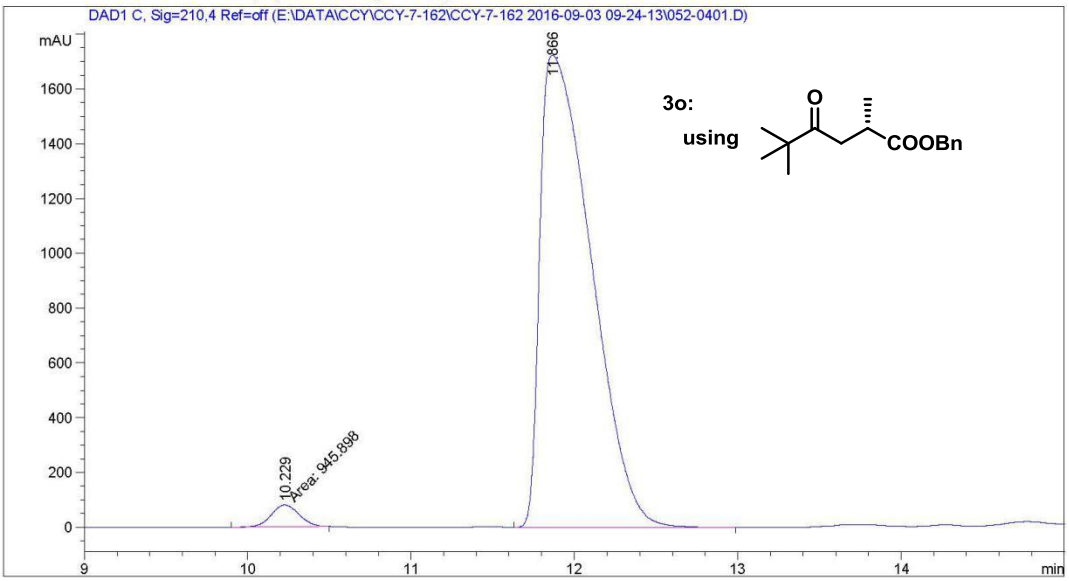
```
==========================================================================
Acq. Operator   : SYSTEM                          Seq. Line :   3
Acq. Instrument : 1260HPLC-DAD                    Location : Vial 30
Injection Date  : 7/16/2016 4:34:29 PM                 Inj :   1
                                                  Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\CCY\CCY-7-130-131\CCY-7-130-131 2016-07-16 15-36-28\DAD-OJ(1-6)-99-
                  1-1.0ML-5UL-45MIN-ALL.M
Last changed    : 7/16/2016 4:56:46 PM by SYSTEM
                  (modified after loading)
Analysis Method : E:\DATA\CCY\CCY-7-130-131\CCY-7-130-131 2016-07-16 15-36-28\DAD-OJ(1-6)-99-
                  1-1.0ML-5UL-45MIN-ALL.M (Sequence Method)
Last changed    : 9/3/2016 4:25:47 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=210,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  10.307 BB    0.2177 8224.67969  579.01904  49.9209
   2  12.250 BB    0.2535 8250.75488  497.45221  50.0791

Totals :                  1.64754e4  1076.47125
```

Data File E:\DATA\CCY\CCY-7-162\CCY-7-162 2016-09-03 09-24-13\052-0401.D
Sample Name: WSW-153-1

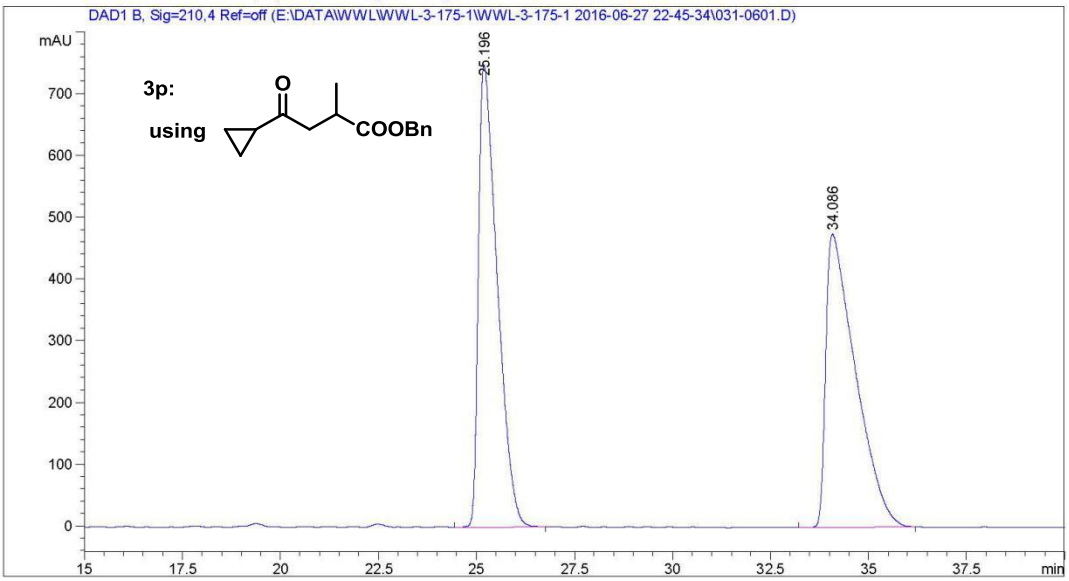
```
=====================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   4
Acq. Instrument : 1260HPLC-DAD                   Location : Vial 52
Injection Date  : 9/3/2016 10:28:06 AM                Inj :   1
                                                Inj Volume : 3.000 µl
Acq. Method     : E:\DATA\CCY\CCY-7-162\CCY-7-162 2016-09-03 09-24-13\DAD-OJ(1-6)-99-1-1.0ML-
                  5UL-30MIN.M
Last changed    : 9/3/2016 9:50:02 AM by SYSTEM
Analysis Method : E:\DATA\CCY\CCY-7-162\CCY-7-162 2016-09-03 09-24-13\DAD-OJ(1-6)-99-1-1.0ML-
                  5UL-30MIN.M (Sequence Method)
Last changed    : 9/3/2016 4:16:36 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
=====================================================================
                        Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 C, Sig=210,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  10.229 MM    0.1976  945.89850   79.79642   2.4638
   2  11.866 VB    0.3495 3.74461e4  1723.47534  97.5362

Totals :                  3.83920e4  1803.27177
```

Data File E:\DATA\WWL\WWL-3-175-1\WWL-3-175-1 2016-06-27 22-45-34\031-0601.D
Sample Name: ccy-7-99-2

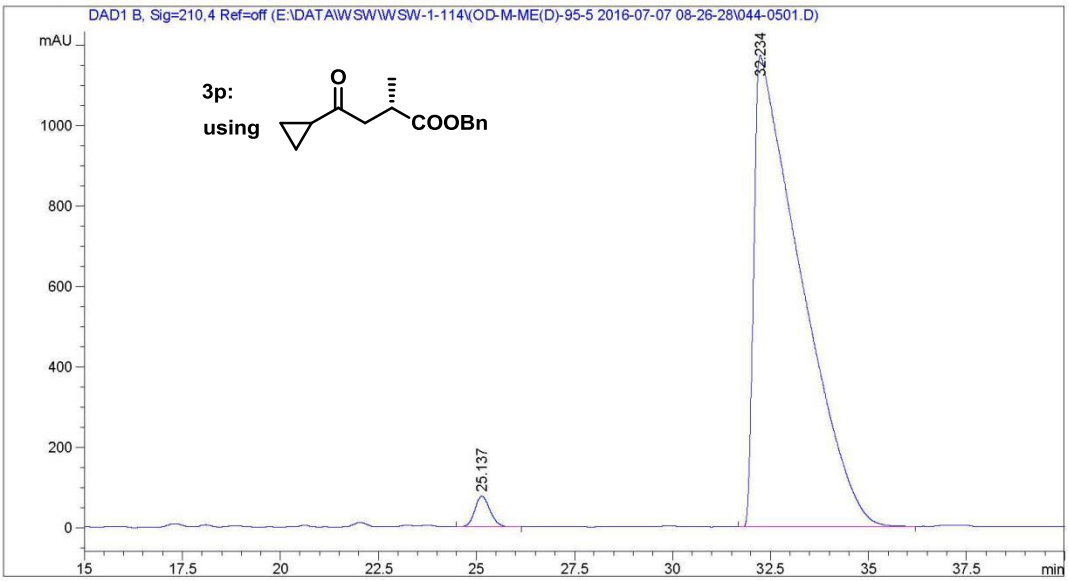
```
=====================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   6
Acq. Instrument : 1260HPLC-DAD                  Location : Vial 31
Injection Date  : 6/28/2016 1:31:32 AM               Inj :   1
                                               Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\WWL\WWL-3-175-1\WWL-3-175-1 2016-06-27 22-45-34\DAD-OJ(1-6)-99-1-1.
                  0ML-5UL-45MIN-ALL.M
Last changed    : 6/27/2016 10:46:58 PM by SYSTEM
Analysis Method : E:\DATA\WWL\WWL-3-175-1\WWL-3-175-1 2016-06-27 22-45-34\DAD-OJ(1-6)-99-1-1.
                  0ML-5UL-45MIN-ALL.M (Sequence Method)
Last changed    : 9/3/2016 9:00:48 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
=====================================================================
                        Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=210,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  25.196 BB    0.4999 2.49313e4   749.75635  49.9331
   2  34.086 BB    0.7400 2.49980e4   475.02005  50.0669

Totals :                  4.99293e4  1224.77640
```

Data File E:\DATA\WSW\WSW-1-114\(OD-M-ME(D)-95-5 2016-07-07 08-26-28\044-0501.D
Sample Name: WSW-1-114-3-1

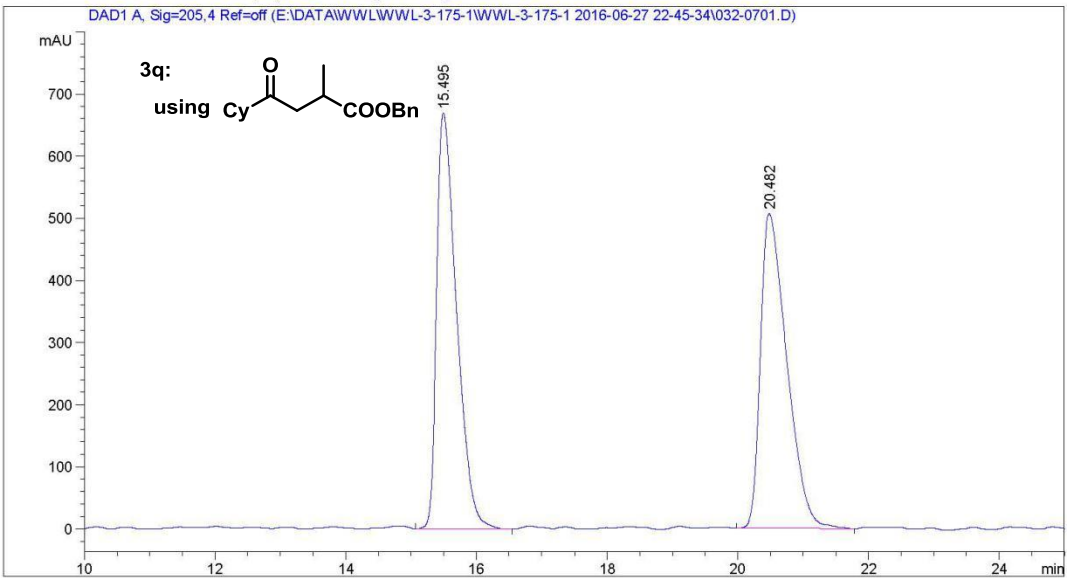
```
==========================================================================
Acq. Operator   : SYSTEM                          Seq. Line :   5
Acq. Instrument : 1260HPLC-DAD                    Location : Vial 44
Injection Date  : 7/7/2016 10:56:18 AM                 Inj :   1
                                                  Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\WSW\WSW-1-114\(OD-M-ME(D)-95-5 2016-07-07 08-26-28\DAD-OJ(1-6)-99-1
                  -1.0ML-5UL-45MIN-ALL.M
Last changed    : 7/7/2016 11:31:30 AM by SYSTEM
                  (modified after loading)
Analysis Method : E:\DATA\WSW\WSW-1-114\(OD-M-ME(D)-95-5 2016-07-07 08-26-28\DAD-OJ(1-6)-99-1
                  -1.0ML-5UL-45MIN-ALL.M (Sequence Method)
Last changed    : 9/3/2016 8:57:48 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 B, Sig=210,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  25.137 BB    0.4217 2085.11328   76.58176   2.1059
   2  32.234 BB    1.0624 9.69280e4  1172.51477  97.8941

Totals :                  9.90131e4  1249.09653
```

Data File E:\DATA\WWL\WWL-3-175-1\WWL-3-175-1 2016-06-27 22-45-34\032-0701.D
Sample Name: ccy-7-99-3

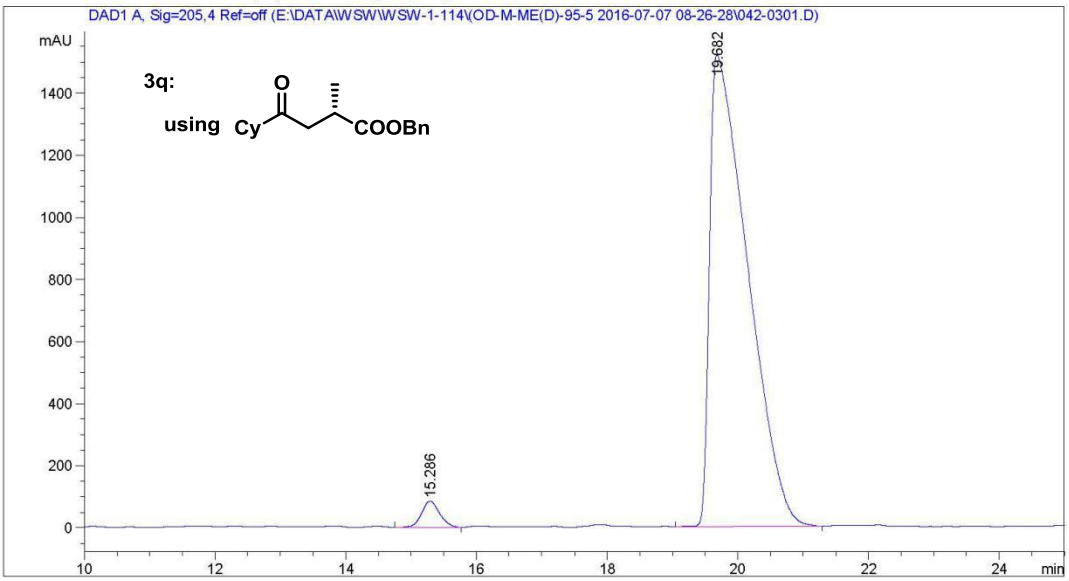
```
=====================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   7
Acq. Instrument : 1260HPLC-DAD                  Location : Vial 32
Injection Date  : 6/28/2016 2:17:32 AM               Inj :   1
                                               Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\WWL\WWL-3-175-1\WWL-3-175-1 2016-06-27 22-45-34\DAD-OJ(1-6)-99-1-1.
                  0ML-5UL-45MIN-ALL.M
Last changed    : 6/27/2016 10:46:58 PM by SYSTEM
Analysis Method : E:\DATA\WWL\WWL-3-175-1\WWL-3-175-1 2016-06-27 22-45-34\DAD-OJ(1-6)-99-1-1.
                  0ML-5UL-45MIN-ALL.M (Sequence Method)
Last changed    : 9/3/2016 5:27:22 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
=====================================================================
                        Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=205,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  15.495 VB    0.3197 1.41013e4   669.61841  49.9133
   2  20.482 BB    0.4220 1.41503e4   506.41763  50.0867

Totals :                  2.82516e4  1176.03604
```

Data File E:\DATA\WSW\WSW-1-114\(OD-M-ME(D)-95-5 2016-07-07 08-26-28\042-0301.D
Sample Name: WSW-1-114-2

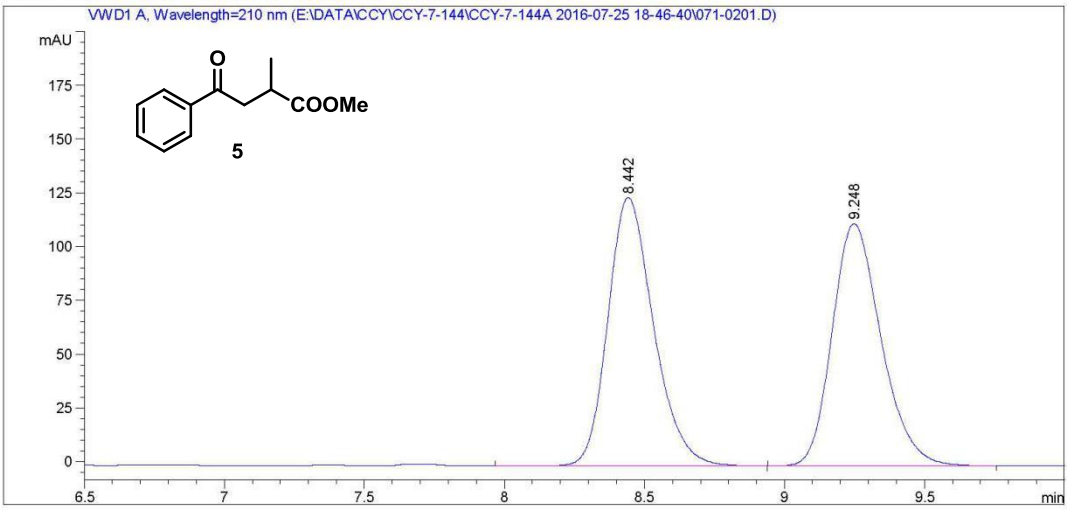
```
=====================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   3
Acq. Instrument : 1260HPLC-DAD                  Location : Vial 42
Injection Date  : 7/7/2016 9:24:22 AM                Inj :   1
                                               Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\WSW\WSW-1-114\(OD-M-ME(D)-95-5 2016-07-07 08-26-28\DAD-OJ(1-6)-99-1
                  -1.0ML-5UL-45MIN-ALL.M
Last changed    : 7/7/2016 8:26:28 AM by SYSTEM
Analysis Method : E:\DATA\WSW\WSW-1-114\(OD-M-ME(D)-95-5 2016-07-07 08-26-28\DAD-OJ(1-6)-99-1
                  -1.0ML-5UL-45MIN-ALL.M (Sequence Method)
Last changed    : 9/3/2016 5:24:57 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
=====================================================================
                        Area Percent Report
=====================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: DAD1 A, Sig=205,4 Ref=off

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  15.286 BB    0.2944 1601.35291   84.02046   2.5606
   2  19.682 VB    0.5467 6.09372e4  1516.76587  97.4394

Totals :                  6.25385e4  1600.78633
```

Data File E:\DATA\CCY\CCY-7-144\CCY-7-144A 2016-07-25 18-46-40\071-0201.D
Sample Name: ccy-7-144-2

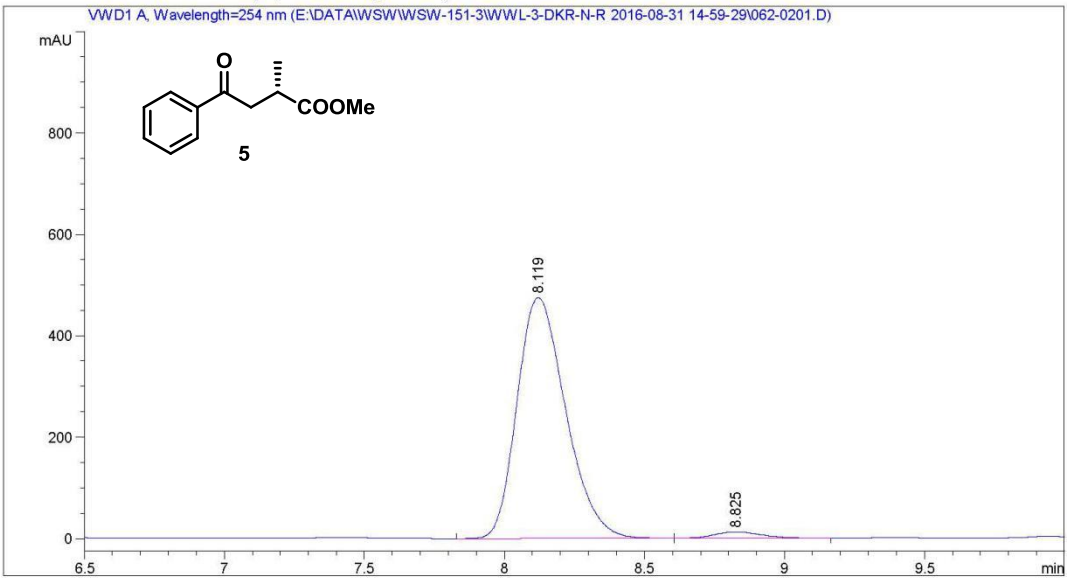
```
======================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   2
Acq. Instrument : 1260HPLC-VWD                   Location : Vial 71
Injection Date  : 7/25/2016 6:58:14 PM               Inj :   1
                                                Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\CCY\CCY-7-144\CCY-7-144A 2016-07-25 18-46-40\VWD-AD(1-6)-95-5-1ML-
                  5UL-210-60MIN.M
Last changed    : 7/25/2016 7:26:07 PM by SYSTEM
                  (modified after loading)
Analysis Method : E:\DATA\CCY\CCY-7-144\CCY-7-144A 2016-07-25 18-46-40\VWD-AD(1-6)-95-5-1ML-
                  5UL-210-60MIN.M (Sequence Method)
Last changed    : 7/19/2017 5:25:22 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=210 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.442 BB    0.1743 1414.93030  124.72693  50.9759
   2   9.248 BB    0.1861 1360.75659  112.49741  49.0241

Totals :                  2775.68689  237.22434


======================================================================
```

Data File E:\DATA\WSW\WSW-151-3\WWL-3-DKR-N-R 2016-08-31 14-59-29\062-0201.D
Sample Name: WSW-151-3

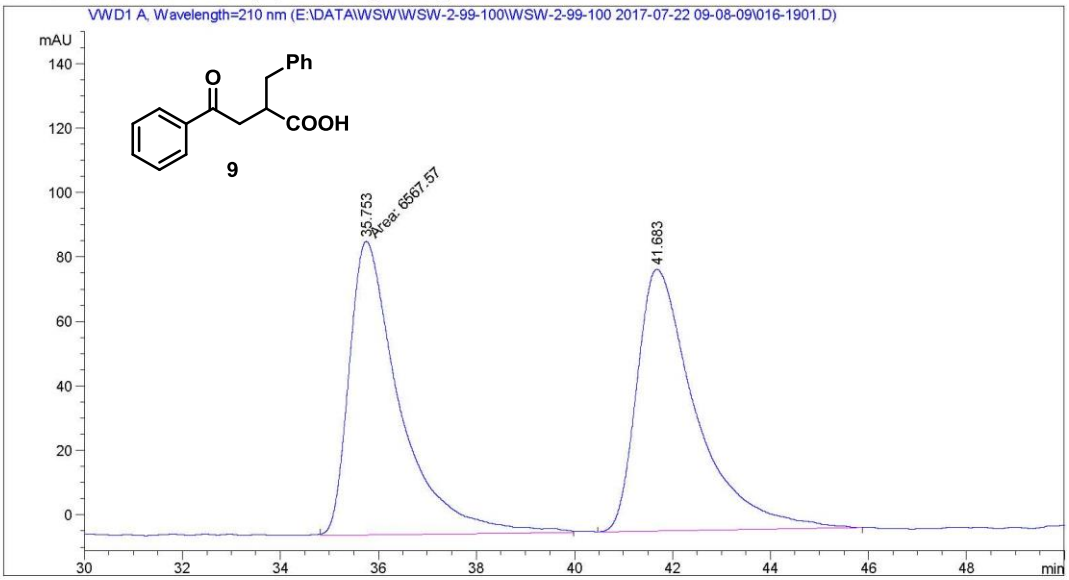
```
==========================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   2
Acq. Instrument : 1260HPLC-VWD                  Location : Vial 62
Injection Date  : 8/31/2016 3:11:10 PM               Inj :   1
                                               Inj Volume : 10.000 µl
Acq. Method     : E:\DATA\WSW\WSW-151-3\WWL-3-DKR-N-R 2016-08-31 14-59-29\VWD-AD(1-6)-95-5-1.
                  0ML-254NM-60MIN.M
Last changed    : 8/31/2016 2:59:29 PM by SYSTEM
Analysis Method : E:\DATA\WSW\WSW-151-3\WWL-3-DKR-N-R 2016-08-31 14-59-29\VWD-AD(1-6)-95-5-1.
                  0ML-254NM-60MIN.M (Sequence Method)
Last changed    : 7/19/2017 5:23:05 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
==========================================================================
                        Area Percent Report
==========================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.119 BV    0.1865 5720.54834  474.98645  97.4050
   2   8.825 VB    0.1847  152.40485   12.73159   2.5950

Totals :                  5872.95319  487.71804
```

Data File E:\DATA\WSW\WSW-2-99-100\WSW-2-99-100 2017-07-22 09-08-09\016-1901.D
Sample Name: WSW-2-100-2

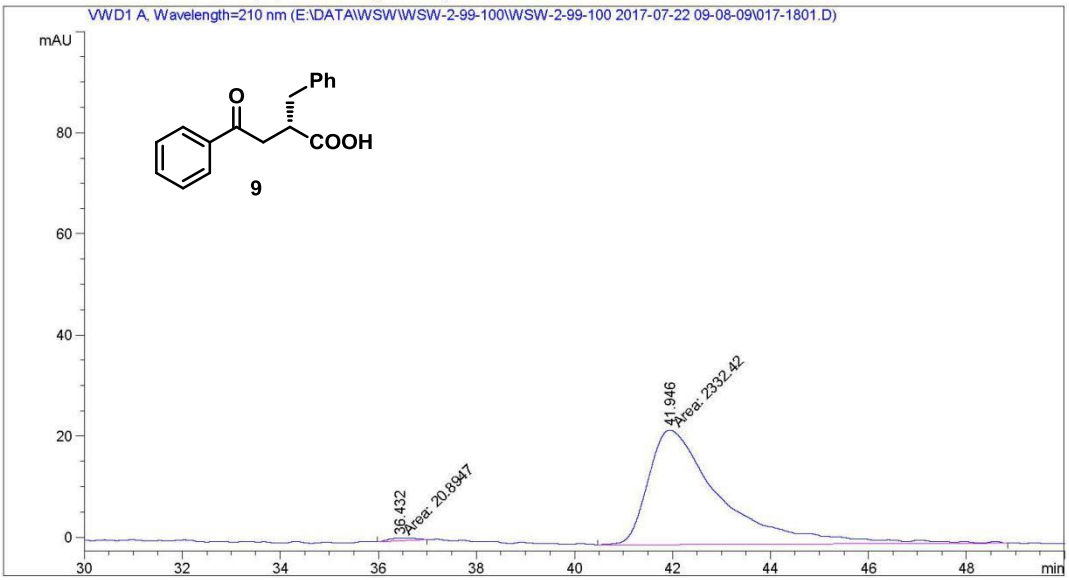
```
======================================================================
Acq. Operator   : SYSTEM                          Seq. Line :  19
Acq. Instrument : 1260HPLC-VWD                    Location : Vial 16
Injection Date  : 7/22/2017 8:04:12 PM                 Inj :   1
                                                  Inj Volume : 10.000 µl
Acq. Method     : E:\DATA\WSW\WSW-2-99-100\WSW-2-99-100 2017-07-22 09-08-09\VWD-AD(1-2)-95-5-
                  1ML-10UL-210NM-60MIN.M
Last changed    : 7/22/2017 8:53:16 PM by SYSTEM
                  (modified after loading)
Analysis Method : E:\DATA\WSW\WSW-2-99-100\WSW-2-99-100 2017-07-22 09-08-09\VWD-AD(1-2)-95-5-
                  1ML-10UL-210NM-60MIN.M (Sequence Method)
Last changed    : 7/22/2017 9:02:17 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=210 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  35.753 MM    1.2001 6567.56689   91.21033  49.2973
   2  41.683 BB    1.2326 6754.78906   81.18958  50.7027

Totals :                  1.33224e4   172.39992
```

Data File E:\DATA\WSW\WSW-2-99-100\WSW-2-99-100 2017-07-22 09-08-09\017-1801.D
Sample Name: WSW-2-99-1

```
======================================================================
Acq. Operator   : SYSTEM                        Seq. Line :  18
Acq. Instrument : 1260HPLC-VWD                   Location : Vial 17
Injection Date  : 7/22/2017 7:03:24 PM               Inj :   1
                                                Inj Volume : 10.000 µl
Acq. Method     : E:\DATA\WSW\WSW-2-99-100\WSW-2-99-100 2017-07-22 09-08-09\VWD-AD(1-2)-95-5-
                  1ML-10UL-210NM-60MIN.M
Last changed    : 7/22/2017 4:09:58 PM by SYSTEM
Analysis Method : E:\DATA\WSW\WSW-2-99-100\WSW-2-99-100 2017-07-22 09-08-09\VWD-AD(1-2)-95-5-
                  1ML-10UL-210NM-60MIN.M (Sequence Method)
Last changed    : 7/27/2017 10:26:39 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




```
======================================================================
                        Area Percent Report
======================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Do not use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=210 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  36.432 MM    0.4485   20.89468 5.54385e-1   0.8879
   2  41.946 MM    1.7180 2332.42212   22.62732  99.1121

Totals :                  2353.31680   23.18171
```

Reference

[1] a) T. Noguchi, A. Onodera, K. Tomisawa, S. Yokomori, *Chem. Pharm. Bull.* **2002**, *50*, 1407-1412; b) T. Noguchi, A. Onodera, M. Ito, M. Yoshida, S. Yokomoril, *Synth.Comm.*, **2003**, *33*, 2657–2670; c) C. Chen, Z. Zhang, S. Jin, X. Fan, M. Geng, S. Wen, Y. Zhou, X. Wang, L. W. Chung, X.-Q. Dong, X. Zhang. *Angew. Chem. Int. Ed.* **2017**, *56*, 6808-6812; d) G. Abbandonato, G. Signore, R. Nifosì, V. Voliani, R. Bizzarri, F. Beltram, *Eur. Biophys. J.* **2011**, *40*, 1205-1214; e) F., Robert and H., Lourdes M. *J. Am. Chem. Soc.* **1959**, *81*, 391-393. f) S. Dominique, M. Federica, PCT Int. APPI., 2013092979, 27 Jun 2013.

[2] Hoge, G.; Wu, H.-P.; Kissel, W. S.; Pflum, D. A.; Greene, D. J.; Bao, J. *J. Am. Chem. Soc.* **2004**, *126*, 5966-5967.