Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information for

Rhodium-catalyzed Asymmetric Hydrogenation of Tetrasubstituted *β*-acetoxy-*α*-enamido Esters and Efficient Synthesis of Droxidopa

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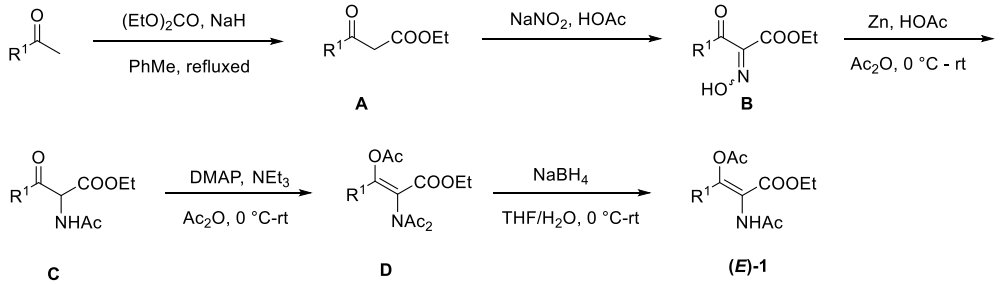
Contents

1. General Information

Unless otherwise noted, all reagents and solvents were purchased from commercial suppliers and used without further purification. NMR spectra were recorded on Bruker ADVANCE III (400 MHz) spectrometers for ^{1}H NMR and ^{13}C NMR. $CDCl_3$ was the solvent used for the NMR analysis, with tetramethylsilane as the internal standard. Chemical shifts were reported upfield to TMS (0.00 ppm) for ^{1}H NMR and relative to $CDCl_3$ (77.0 ppm) for ^{13}C NMR. Optical rotation was determined using a Perkin Elmer 343 polarimeter. HPLC analysis was conducted on an Agilent 1260 Series instrument. Column Chromatography was performed with silica gel Merck 60 (300-400 mesh). All new products were further characterized by HRMS. A positive ion mass spectrum of sample was acquired on a Thermo LTQ-FT mass spectrometer with an electrospray ionization source.

2. General Procedure for the Synthesis of Compound 1



To a stirred solution of ketones (25 mmol) in toluene (120 mL) was added ethyl dicarbonate (75 mmol) and NaH (50 mmol, 60%). The reaction mixture was refluxed until TLC indicated the total consumption of the ketones. After cooling, the reaction mixture was quenched by ice water, acidified with 3 M HCl to pH 2~3 and extracted with EtOAc (100 mL × 3). The combined organic layer was dried over sodium sulfate and evaporated under reduced pressure. The obtained-keto ester **A** was used directly for the next step without further purifications.[1]

To a solution of *β*-keto ester **A** (20 mmol) in HOAc (5.7 mL, 100 mmol) was added $NaNO_2$ (2.07 g, 30 mmol) in H_2O (6 mL) at 0 °C. The reaction was monitored by TLC. When the reaction was completed, the reaction mixture was treated with H_2O (50 mL) and CH_2Cl_2 (50 mL), the mixture was stirred for 10 minutes and the phases were separated. The aqueous layer was extracted with CH_2Cl_2 (3×30 mL). The combined organic solution was washed with saturated $NaHCO_3$ (150 mL) and brine

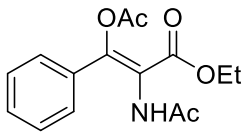
(150 mL), then dried over Na_2SO_4. After evaporation of the solvent, the residue was subjected to chromatography or crystallization to afford the desired α-hydroxyimino-β-ketoester **B**, yielding 95-99%.[2]

To a stirred ice-cold solution of **B** (10 mmol) in Ac_2O (15-30 mL) was added Zn powder (3.25 g, 10 mmol) in small portion. Then HOAc (5.7 mL, 100 mmol) was added drop-wise from the dropping funnel to the heterogeneous solution at 0 °C. The reaction. After completion of the reaction, the mixture was filtered and the residue was washed with CH_2Cl_2 (100 mL). The filtrate was treated with saturated $NaHCO_3$ and separated. The organic phase was washed with brine (100 mL), then dried over Na_2SO_4. After evaporation of the solvent, the residue was subject to chromatography (PE/EA 2/1) to get the desired product α-acetamido-β-ketoester **C** in 70-92% yield.[3]

To a stirred solution of **C** (4.26 mmol) in acetic anhydride (16 mL) was added Et_3N (1.85 mL, 13.4 mmol), and DMAP (0.0052 g, 0.043 mmol) at 0 °C. After 0.5 h at 0 °C, the reaction was kept at room temperature and monitored by TLC. The reaction mixture was treated with saturated $NaHCO_3$ and separated. The organic phase was washed with brine (50 mL), then dried over Na_2SO_4. After evaporation of the solvent, the residue was subject to chromatography (PE/EA 8/1) to get the desired product α-acetamido-β-ketoester **D**, yielding 78-90%. [4]

Sodium borohydride (76.5 mg, 2.0 mmol) in water (0.6 mL) was added dropwise to a stirred solution of **D** (1 mmol) in tetrahydrofuran (40 mL) at 0 °C. After 20 min, the mixture was allowed to warm up to room temperature and then stirring was continued for 1 h. The reaction was quenched with brine (2 mL), and the resulting mixture was diluted with ethyl acetate (200 mL). The organic layer was washed with brine and dried over Na_2SO_4. Concentration of the solvent in vacuo gave a residue, which was purified by column chromatography (PE/EA 1/1) to give (***E*)-1** as a white solid, yielding 40-70%. [5]

(*E*)-ethyl-2-acetamido-3-acetoxy-3-phenylacrylate ((*E*)-1a)



White solid; m. p. 142-145 □; 150 mg; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.48-7.50 (m, 2H), 7.40-7.42 (m, 3H), 6.99 (s, 1H), 4.28 (q, *J* = 7.1 Hz, 1H), 2.23 (s, 3H), 2.00 (s, 3H), 1.33 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 169.22,

168.36, 163.41, 148.52, 133.16, 130.09, 128.68, 127.88, 119.03, 61.59, 22.81, 20.81, 14.14. ESI-HRMS calculated for $C_{15}H_{18}NO_5^+$ ([M+H]$^+$): 292.1179, found 292.1175. The structure of **(*E*)-1a** was determined by X-Ray (figure 1).

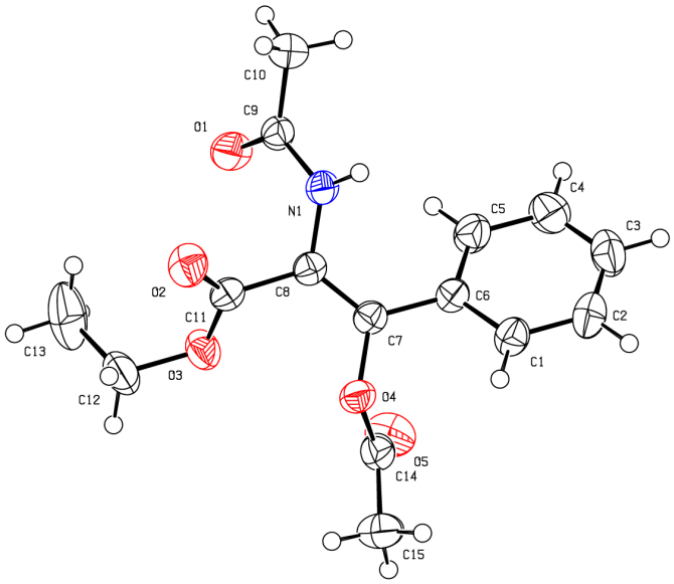
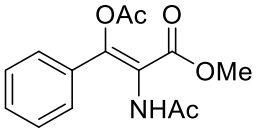


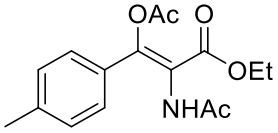
Figure 1. the X-ray structure of **(*E*)-1a**.

(*E*)-methyl-2-acetamido-3-acetoxy-3-phenylacrylate ((*E*)-1b)



White solid; m. p. 183-186 □;130 mg; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.46-7.49 (m, 2H), 7.39-7.43 (m, 3H), 6.73 (s, 1H), 3.81 (s, 3H), 2.23 (s, 3H), 2.01 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 169.14, 168.48, 163.78, 149.02, 133.12, 130.24, 128.78, 127.94, 118.65, 52.60, 22.94, 20.83. ESI-HRMS calculated for $C_{14}H_{16}NO_5^+$ ([M+H]$^+$): 278.1023, found 278.1016.

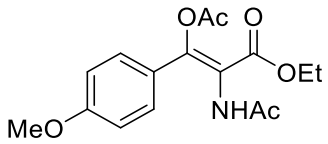
(*E*)-ethyl-2-acetamido-3-acetoxy-3-(*p*-tolyl)acrylate ((*E*)-1c)



White solid; m. p. 174-176 □; 145 mg; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.36-7.38 (m, 2H), 7.19-7.21 (m, 2H), 6.72 (s, 1H), 4.27 (q, *J* = 7.1 Hz, 2H), 2.37 (s, 3H), 2.21 (s, 3H), 2.01 (s, 3H), 1.31 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ =

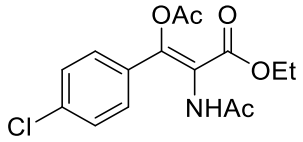
169.30, 168.39, 163.54, 148.87, 140.41, 130.24, 129.38, 127.78, 118.48, 61.50, 22.77, 21.47, 20.82, 14.14. ESI-HRMS calculated for $C_{16}H_{20}NO_5^+$ ($[M+H]^+$): 306.1336, found 306.1333.

(*E*)-ethyl-2-acetamido-3-acetoxy-3-(4-methoxyphenyl)acrylate ((*E*)-1d)



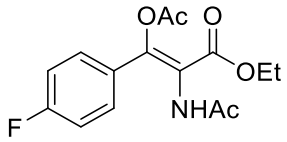
White solid; m. p. 161-164 □; 155 mg; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.42-7.44 (m, 2H), 6.72-6.91 (m, 2H), 6.72 (s, 1H), 4.26 (q, *J* = 7.1 Hz, 2H), 3.83 (s, 3H), 2.22 (s, 3H), 2.02 (s, 3H), 1.31 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 169.10, 168.44, 163.55, 160.91, 149.32, 129.55, 125.40, 117.72, 114.16, 61.53, 55.41, 22.99, 20.92, 14.20. ESI-HRMS calculated for $C_{16}H_{20}NO_6^+$ ($[M+H]^+$): 322.1285, found 322.1282.

(*E*)-ethyl-2-acetamido-3-acetoxy-3-(4-chlorophenyl)acrylate ((*E*)-1e)



White solid; m. p. 188-190 □; 150 mg; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.41-7.43 (m, 2H), 7.35-7.38 (m, 2H), 4.27 (q, *J* = 7.1 Hz, 2H), 2.22 (s, 3H), 2.01 (s, 3H), 1.32 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 168.97, 168.34, 163.24, 148.28, 136.14, 131.89, 129.25, 129.06, 119.04, 61.80, 23.01, 20.84, 14.18. ESI-HRMS calculated for $C_{15}H_{15}NO_5Cl^-$ ($[M-H]^-$): 324.0644, found 324.06447.

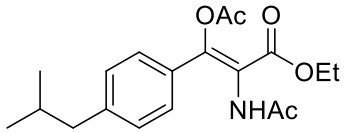
(*E*)-ethyl-2-acetamido-3-acetoxy-3-(4-fluorophenyl)acrylate ((*E*)-1f)



White solid; m. p. 135-137 □; 132 mg; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.46-7.50 (m, 2H), 7.06-7.10 (m, 2H), 6.73 (s, 1H), 4.27 (q, *J* = 7.1 Hz, 2H), 2.22 (s, 3H), 2.00 (s, 3H), 1.32 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 169.02, 168.35, 164.66, 163.31, 148.62, 130.13, 130.04, 129.50, 118.74, 116.06, 115.84, 61.73, 22.99,

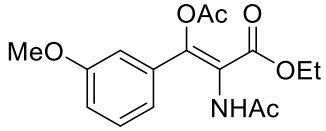
20.86, 14.18. ESI-HRMS calculated for $C_{15}H_{17}NO_5F^+$ ([M+H]$^+$): 310.1085, found 310.1077.

(*E*)-ethyl-2-acetamido-3-acetoxy-3-(4-isobutylphenyl)acrylate ((*E*)-1g)



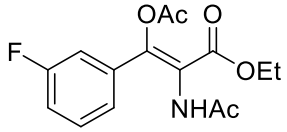
White solid; m. p. 127-130 □; 152 mg; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.38 (d, *J* = 8.2 Hz, 2H), 7.17 (d, *J* = 8.2 Hz, 2H), 6.73 (s, 1H), 4.27 (q, *J* = 7.1 Hz, 2H), 2.48 (d, *J* = 7.2 Hz, 2H), 2.22 (s, 3H), 2.02 (s, 3H), 1.85-1.90 (m, 1H), 1.31 (t, *J* = 7.1 Hz, 3H), 0.91 (d, *J* = 6.6 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 169.12, 168.42, 163.47, 148.78, 144.29, 130.41, 129.47, 127.70, 118.55, 61.55, 45.30, 30.13, 22.93, 22.44, 20.90, 14.18. ESI-HRMS calculated for $C_{19}H_{26}NO_5^+$ ([M+H]$^+$): 348.1805, found 348.1797.

(*E*)-ethyl-2-acetamido-3-acetoxy-3-(3-methoxyphenyl)acrylate ((*E*)-1h)



White solid; m. p. 123-125 □; 128 mg; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.30-7.34 (m, 1H), 7.05-7.07 (m, 1H), 7.00 -7.01 (m, 1H), 6.91-6.94 (m, 1H), 4.28 (q, *J* = 7.1 Hz, 2H), 3.80 (s, 3H), 2.21 (s, 3H), 2.02 (s, 3H), 1.32 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 168.91, 168.35, 163.33, 159.68, 147.73, 134.40, 129.91, 120.23, 119.25, 115.78, 113.36, 61.67, 55.40, 22.94, 20.83, 14.18. ESI-HRMS calculated for $C_{16}H_{20}NO_6^+$ ([M+H]$^+$): 322.1285, found 322.1283.

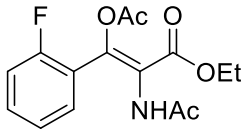
(*E*)-ethyl-2-acetamido-3-acetoxy-3-(3-fluorophenyl)acrylate ((*E*)-1i)



White solid; m. p. 112-115 □; 123 mg; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.35-7.40 (m, 1H), 7.28-7.30 (m, 1H), 7.17-7.20 (m, 1H), 7.05-7.09 (m, 1H), 6.73 (s, 1H), 4.29 (q, *J* = 7.1 Hz, 2H), 2.23 (s, 3H), 2.03 (s, 3H), 1.32 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101

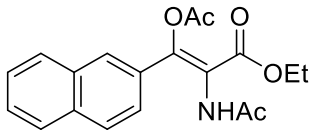
MHz, $CDCl_3$) δ = 168.82, 168.29, 163.17, 161.43, 130.49, 130.41, 123.62, 117.28, 117.07, 114.99, 114.76, 61.84, 23.05, 20.82, 14.18. ESI-HRMS calculated for $C_{15}H_{15}NO_5F^-$ ($[M-H]^-$): 308.0940, found 308.0942.

(*E*)-ethyl-2-acetamido-3-acetoxy-3-(2-fluorophenyl)acrylate ((*E*)-1j)



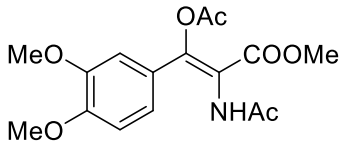
White solid; m. p. 137-139 □; 117 mg; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.39-7.44 (m, 2H), 7.10-7.21 (m, 2H), 6.73 (s, 1H), 4.28 (q, *J* = 7.1 Hz, 2H), 2.20 (s, 3H), 1.97 (s, 3H), 1.32 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 168.78, 168.08, 162.82, 160.63, 158.13, 144.12, 132.10, 132.01, 130.19, 130.17, 124.52, 124.49, 121.28, 121.02, 116.38, 116.16, 61.72, 22.85, 20.76, 14.14. ESI-HRMS calculated for $C_{15}H_{17}NO_5F^+$ ($[M+H]^+$): 310.1085, found 310.1077.

(*E*)-ethyl-2-acetamido-3-acetoxy-3-(naphthalen-2-yl)acrylate ((*E*)-1k)



White solid; m. p. 162-164 □; 238 mg; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.95 (s, 1H), 7.83-7.86 (m, 3H), 7.52-7.56 (m, 3H), 6.79 (s, 1H), 4.30 (q, *J* = 7.1 Hz, 2H), 2.24 (s, 3H), 2.00 (s, 3H), 1.33 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 169.03, 168.45, 163.42, 148.63, 133.78, 132.86, 130.62, 128.62, 128.53, 128.10, 127.81, 127.59, 126.85, 124.64, 119.19, 61.70, 22.96, 20.90, 14.21. ESI-HRMS calculated for $C_{19}H_{20}NO_5^+$ ($[M+H]^+$): 342.1336, found 342.1332.

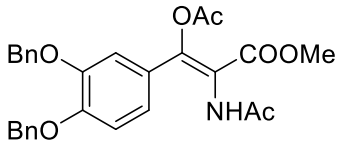
(*E*)-methyl-2-acetamido-3-acetoxy-3-(3,4-dimethoxyphenyl)acrylate ((*E*)-1l)



White solid; m. p. 174-176 □; 150 mg; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.06-7.09 (m, 1H), 7.00 (d, *J* = 2.0 Hz, 1H), 6.87 (d, *J* = 8.4 Hz, 1H), 6.79 (s, 1H), 3.90 (s, 3H), 3.85 (s, 3H), 3.81 (s, 3H), 2.23 (s, 3H), 2.03 (s, 3H); ^{13}C NMR (101

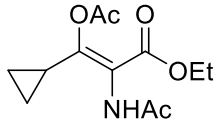
MHz, $CDCl_3$) δ = 169.16, 168.57, 163.88, 150.60, 149.50, 148.82, 125.44, 121.29, 117.56, 110.94, 110.66, 55.97, 52.54, 22.94, 20.87. ESI-HRMS calculated for $C_{16}H_{20}NO_7^+$ ($[M+H]^+$): 338.1232, found 338.1229.

(*E*)-methyl-2-acetamido-3-acetoxy-3-(3,4-bis(benzyloxy)phenyl)acrylate ((*E*)-1m)



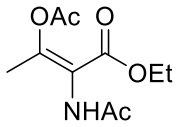
White solid; m. p. 122-125 □; 250 mg; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49-7.28 (m, 10 H), 7.02 (s, 1H), 6.91 (d, *J* = 8.9 Hz, 1H), 6.66 (s, 1H), 5.18 (s, 2H), 5.14 (s, 2H), 3.78 (s, 3H), 2.13 (s, 3H), 1.90 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.08, 168.46, 163.85, 150.53, 148.86, 148.50, 136.83, 136.64, 128.67, 128.08, 128.01, 127.21, 127.17, 125.80, 121.86, 117.66, 114.21, 113.88, 71.20, 70.91, 52.51, 22.89, 20.76. ESI-HRMS calculated for $C_{28}H_{28}NO_7^+$ ($[M+H]^+$): 490.1858, found 490.1855.

(*E*)-ethyl-2-acetamido-3-acetoxy-3-cyclopropylacrylate ((*E*)-1n)



White solid; 120 mg; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 6.83 (s, 1H), 4.16 (q, *J* = 7.1 Hz, 2H), 2.19 (s, 3H), 2.14 (s, 3H), 1.89-1.93 (m, 1H), 1.25 (t, *J* = 7.1 Hz, 3H), 0.80-0.98 (m, 4H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 169.61, 168.29, 162.74, 159.08, 116.06, 61.17, 23.21, 20.85, 14.19, 12.87, 6.76. ESI-HRMS calculated for $C_{12}H_{18}NO_5^+$ ($[M+H]^+$): 256.1179, found 256.1177.

(*E*)-ethyl-2-acetamido-3-acetoxybut-2-enoate ((*E*)-1o)



White solid; 133 mg; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 6.76 (s, 1H), 4.20 (q, *J* = 7.1 Hz, 2H), 2.22 (s, 3H), 2.12 (s, 3H), 2.01 (s, 3H), 1.28 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 168.78, 168.37, 162.93, 155.78, 117.24, 61.46, 23.30, 20.99, 19.28, 14.19. ESI-HRMS calculated for $C_{10}H_{16}NO_5^+$ ($[M+H]^+$): 230.1023, found 230.1018.

3. General Procedure for Asymmetric Hydrogenation of Compound 1

A stock solution was made by mixing $[Rh(COD)_2]BF_4$ with (R_C,S_P)-DuanPhos in a 1:1.1 molar ratio in CH_2Cl_2 at room temperature for 30 min in a nitrogen-filled glovebox. An aliquot of the catalyst solution (0.5 mL, 0.001 mmol) was transferred by syringe into the vials charged with different substrates (0.05 mmol for each). The vials were subsequently transferred into an autoclave into which hydrogen gas was charged. The reaction was then stirred under H_2 (30 atm) at room temperature for 24 h. The hydrogen gas was released slowly and carefully. The solution was concentrated and passed through a short column of silica gel (eluent: EtOAc) to remove the metal complex. The ee values of all compounds **2** were determined by HPLC analysis on a chiral stationary phase.

Procedure for Gram-scale Asymmetric Hydrogenation of (*E*)-1a

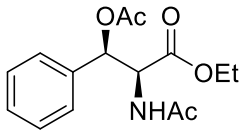
A stock solution was made by mixing $[Rh(COD)_2]BF_4$ with (R_C,S_P)-DuanPhos in a 1:1.1 molar ratio in CH_2Cl_2 at room temperature for 45 min in a nitrogen-filled glovebox. An aliquot of the catalyst solution (2 mL, 0.068 mmol) was transferred by syringe into the vial charged with **(*E*)-1a** (3.4 mmol) in anhydrous CH_2Cl_2 (5 mL). The vials were subsequently transferred into an autoclave into which hydrogen gas was charged. The reaction was then stirred under H_2 (30 atm) at room temperature for 30 h. The hydrogen gas was released slowly and carefully. The solution was concentrated and passed through a short column of silica gel (eluent: EtOAc) to remove the metal complex. The ee values of **2a** were determined by HPLC analysis on a chiral stationary phase.

Procedure for TON study of Asymmetric Hydrogenation of (*E*)-1a

A stock solution was made by mixing $[Rh(COD)_2]BF_4$ with (R_C,S_P)-DuanPhos in a 1:1.1 molar ratio in CH_2Cl_2 at room temperature for 45 min in a nitrogen-filled glovebox. An aliquot of the catalyst solution (0.2 mL, 0.0004 mmol) was transferred by syringe into the vial charged with **(*E*)-1a** (0.2 mmol) in anhydrous CH_2Cl_2 (2 mL). The vials were subsequently transferred into an autoclave into which hydrogen gas was charged. The reaction was then stirred under H_2 (70 atm) at 60 °C for 24 h. The hydrogen gas was released slowly and carefully. The solution was concentrated and passed through a short column of silica gel (eluent: EtOAc) to remove the metal

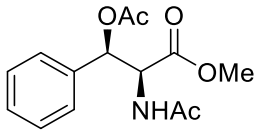
complex. The ee values of **2a** were determined by HPLC analysis on a chiral stationary phase.

(2*S*,3*R*)-ethyl-2-acetamido-3-acetoxy-3-phenylpropanoate (2a)



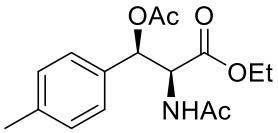
White solid; m. p. 68-70 □; 14.6 mg, 99% yield; >99% ee; $[\alpha]_D^{20}$ = -36.70 (c = 1.00, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralpak OJ-H column, hexane: isopropanol = 95:5; flow rate = 1.0 mL/min; UV detection at 210 nm; t_R = 21.2 min (major), 29.7 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.32-7.39 (m, 5H), 6.30 (d, *J* = 4.2 Hz, 1H), 6.18 (d, *J* = 9.2 Hz, 1H), 5.09 (dd, *J* = 9.2 Hz, 4.2 Hz, 1H), 4.17 (q, *J* = 7.1 Hz, 2H), 2.16 (s, 3H), 1.97 (s, 3H), 1.22 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.85, 169.56, 169.53, 135.94, 128.66, 128.55, 126.29, 74.69, 61.97, 56.38, 23.00, 20.89, 14.02. ESI-HRMS calculated for $C_{15}H_{20}NO_5^+$ ($[M+H]^+$): 294.1336, found 294.1334.

(2*S*,3*R*)-methyl-2-acetamido-3-acetoxy-3-phenylpropanoate (2b)



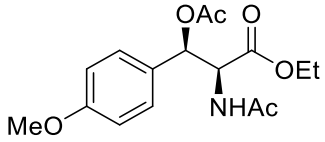
White solid; m. p. 135-136 □; 13.9 mg; 99% yield; >99% ee; $[\alpha]_D^{20}$ = -29.33 (c = 0.60, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralpak OJ-H column, hexane: isopropanol = 95:5; flow rate = 1.0 mL/min; UV detection at 210 nm; t_R = 39.4 min (major), 54.8 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.28-7.37 (m, 5H), 6.28 (d, *J* = 3.9 Hz, 1H), 6.13 (d, *J*= 9.2 Hz, 1H), 5.07 (dd, *J* = 9.3 Hz, 3.9 Hz, 1H), 3.71 (s, 3H), 2.14 (s, 3H), 1.95 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 170.10, 169.83, 169.53, 135.91, 128.68, 128.60, 126.16, 74.53, 56.29, 52.80, 23.01, 20.92. ESI-HRMS calculated for $C_{14}H_{18}NO_5^+$ ($[M+H]^+$): 280.1179, found 280.1173.

(2*S*,3*R*)-ethyl-2-acetamido-3-acetoxy-3-(*p*-tolyl)propanoate (2c)



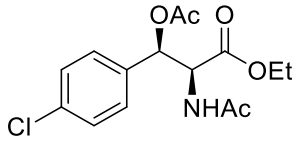
White solid; m. p. 123-158 □; 15.1 mg; 98% yield; >99% ee; $[\alpha]_D^{20}$ = -14.08 (c = 0.71, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralpak OJ-H column, hexane: isopropanol = 95:5; flow rate = 1.0 mL/min; UV detection at 210 nm; t_R = 17.6 min (major), 23.9 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.13-7.20 (m, 4H), 6.24 (d, *J* = 4.2 Hz, 1H), 6.13 (d, *J* = 9.3 Hz, 1H), 5.04 (dd, *J* = 9.3 Hz, 4.2 Hz, 1H), 4.15 (q, *J* = 7.1 Hz, 2H), 2.33 (s, 3H), 2.12 (s, 3H), 1.96 (s, 3H), 1.20 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 169.88, 169.63, 169.58, 138.47, 132.93, 129.25, 126.22, 74.62, 61.93, 56.37, 23.05, 21.23, 20.92, 14.02. ESI-HRMS calculated for $C_{16}H_{22}NO_5^+$ ($[M+H]^+$): 308.1492, found 308.1491.

(2*S*,3*R*)-ethyl-2-acetamido-3-acetoxy-3-(4-methoxyphenyl)propanoate (2d)



White solid; m. p. 129-131 □; 15.7 mg; 97% yield; >99% ee; $[\alpha]_D^{20}$ = -2.96 (c = 0.30, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralpak OJ-H column, hexane: isopropanol = 94:6; flow rate = 1.0 mL/min; UV detection at 210 nm; t_R = 34.9 min (major), 49.1 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.22-7.26 (m, 2H), 6.85 – 6.88 (m, 2H), 6.21 (d, *J* = 4.5 Hz, 1H), 6.17 (d, *J* = 9.3 Hz, 1H), 5.03 (dd, *J* = 9.3 Hz, 4.5 Hz, 1H), 4.13 (q, *J* = 7.1 Hz, 2H), 3.79 (s, 3H), 2.11 (s, 3H), 1.97 (s, 3H), 1.19 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 169.85, 169.62, 159.73, 127.95, 127.75, 113.89, 74.42, 61.87, 56.42, 55.26, 23.04, 20.92, 14.01. ESI-HRMS calculated for $C_{16}H_{22}NO_6^+$ ($[M+H]^+$): 324.1442, found 324.1437.

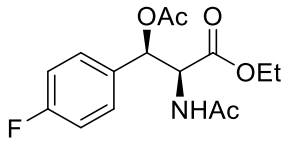
(2*S*,3*R*)-ethyl-2-acetamido-3-acetoxy-3-(4-chlorophenyl)propanoate (2e)



White solid; 16.1 mg; 98% yield; >99% ee; $[\alpha]_D^{20}$ = -14.61 (c = 0.70, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralpak OD-H column,

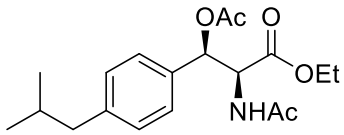
hexane: isopropanol = 95:5; flow rate = 1.0 mL/min; UV detection at 210 nm; t_R = 21.8 min (major), 29.3 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.30-7.33 (m, 2H), 7.22-7.25 (m, 2H), 6.24 (d, *J* = 4.0 Hz, 1H), 6.11 (d, *J* = 9.3 Hz, 1H), 5.05 (dd, *J* = 9.4 Hz, 4.0 Hz, 1H), 4.16 (q, *J* = 7.1 Hz, 2H), 2.12 (s, 3H), 1.95 (s, 3H), 1.22 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 169.83, 169.38, 169.35, 134.57, 134.55, 128.79, 127.69, 74.14, 62.14, 56.05, 23.02, 20.85, 14.06. ESI-HRMS calculated for $C_{15}H_{19}NO_5Cl^+$ ($[M+H]^+$): 328.0946, found 328.0943.

(2*S*,3*R*)-ethyl-2-acetamido-3-acetoxy-3-(4-fluorophenyl)propanoate (2f)



White solid; m. p. 167-169 □; 15.4 mg; 99% yield; >99% ee; $[\alpha]_D^{20}$ = -17.43 (c = 0.70, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralpak OJ-H column, hexane: isopropanol = 95:5; flow rate = 1.0 mL/min; UV detection at 210 nm; t_R = 23.6 min (major), 32.7 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.34 – 7.27 (m, 1H), 7.15 – 6.98 (m, 1H), 6.25 (d, *J*=4.2, 1H), 6.13 (d, *J*=9.3, 1H), 5.05 (dd, *J*=9.4, 4.2, 1H), 4.16 (q, *J*=7.1, 1H), 2.13 (s, 1H), 1.96 (s, 1H), 1.21 (t, *J*=7.1, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 169.80, 169.45, 169.42, 163.96, 161.50, 131.87, 131.84, 128.22, 128.13, 115.68, 115.46, 74.15, 62.07, 56.23, 23.01, 20.87, 14.04. ESI-HRMS calculated for $C_{15}H_{19}NO_5F^+$ ($[M+H]^+$): 312.1242, found 312.1233.

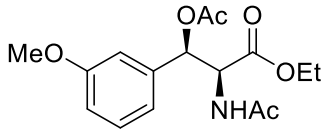
(2*S*,3*R*)-ethyl-2-acetamido-3-acetoxy-3-(4-isobutylphenyl)propanoate (2g)



White solid; 16.8 mg; 96% yield; >99% ee; $[\alpha]_D^{20}$ = -10.32 (c = 0.53, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralpak OJ-H column, hexane: isopropanol = 97:3; flow rate = 1.0 mL/min; UV detection at 210 nm; t_R = 19.1 min (major), 23.5 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.21 (d, *J* = 8.1 Hz, 2H), 7.11 (d, *J* = 8.1 Hz, 2H), 6.25 (d, *J* = 4.5 Hz, 1H), 6.12 (d, *J* = 9.2 Hz, 1H), 5.04 (dd, *J* = 9.3 Hz, 4.5 Hz, 1H), 4.12 (q, *J* = 7.1 Hz, 2H), 2.45 (d, *J* = 7.2 Hz, 2H), 2.13 (s, 3H), 1.95 (s, 3H), 1.81-1.85 (m, 1H), 1.18 (t, *J* = 7.1 Hz, 1H), 0.87-0.89 (m, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 169.84, 169.67, 142.28, 133.12, 129.27,

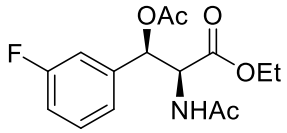
126.16, 74.63, 61.89, 56.46, 45.11, 30.20, 23.05, 22.39, 22.35, 20.95, 14.01. ESI-HRMS calculated for $C_{19}H_{28}NO_5^+$ ($[M+H]^+$): 350.1962, found 350.1954.

(2*S*,3*R*)-ethyl-2-acetamido-3-acetoxy-3-(3-methoxyphenyl)propanoate (2h)



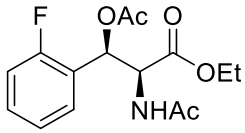
White solid; m. p. 134-135 □; 15.7 mg; 97% yield; >99% ee; $[\alpha]_D^{20}$ = -16.13 (c = 0.50, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralpak OJ-H column, hexane: isopropanol = 95:5; flow rate = 1.0 mL/min; UV detection at 210 nm; t_R = 31.1 min (major), 37.4 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.24-7.28 (m, 1H), 6.83-6.90 (m, 3H), 6.25 (d, *J* = 4.0 Hz, 1H), 6.10 (d, *J* = 9.3 Hz, 1H), 5.06 (dd, *J* = 9.3 Hz, 4.0 Hz, 1H), 4.16 (q, *J* = 7.1 Hz, 2H), 3.80 (s, 3H), 2.13 (s, 3H), 1.96 (s, 3H), 1.22 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 168.78, 168.46, 168.41, 158.59, 136.45, 128.60, 117.47, 112.66, 111.12, 73.41, 60.92, 55.24, 54.25, 21.99, 19.82, 12.98. ESI-HRMS calculated for $C_{16}H_{22}NO_6^+$ ($[M+H]^+$): 324.1442, found 324.1439.

(2*S*,3*R*)-ethyl-2-acetamido-3-acetoxy-3-(3-fluorophenyl)propanoate (2i)



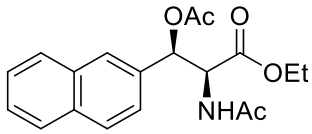
Pale oil; 14.9 mg; 96% yield; >99% ee; $[\alpha]_D^{20}$ = -27.87 (c = 0.93, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralpak OD-H column, hexane: isopropanol = 95:5; flow rate = 1.0 mL/min; UV detection at 210 nm; t_R = 23.4 min (major), 32.2 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.29-7.33 (m, 1H), 7.10 (d, *J* = 7.8 Hz, 1H), 6.99-7.09 (m, 2H), 6.27 (d, *J* = 3.8 Hz, 1H), 6.14 (d, *J* = 9.3 Hz, 1H), 5.07 (dd, *J* = 9.4 Hz, 3.8 Hz, 1H), 4.18 (q, *J* = 7.1 Hz, 2H), 2.14 (s, 3H), 1.96 (s, 3H), 1.24 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 169.85, 169.31, 163.97, 161.52, 138.63, 138.56, 130.26, 130.18, 122.01, 121.98, 115.68, 115.47, 113.43, 113.20, 74.04, 62.14, 56.09, 22.97, 20.81, 14.05. ESI-HRMS calculated for $C_{15}H_{19}NO_5F^+$ ($[M+H]^+$): 312.1242, found 312.1238.

(2*S*,3*R*)-ethyl-2-acetamido-3-acetoxy-3-(2-fluorophenyl)propanoate (2j)



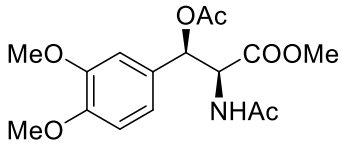
Pale oil; 15.1 mg; 97% yield; >99% ee; $[\alpha]_D^{20}$ = -26.43 (c = 0.70, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralpak OJ-H column, hexane: isopropanol = 95:5; flow rate = 1.0 mL/min; UV detection at 210 nm; t_R = 25.5 min (major), 30.6 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.25-7.29 (m, 2H), 7.06-7.13 (m, 2H), 6.55 (d, *J* = 3.6 Hz, 1H), 6.16 (d, *J* = 9.3 Hz, 1H), 5.15 (dd, *J* = 9.4 Hz, 3.6 Hz, 1H), 4.20 (q, *J* = 7.1 Hz, 2H), 2.13 (s, 3H), 1.92 (s, 3H), 1.25 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 169.74, 169.29, 169.13, 161.00, 158.54, 130.34, 130.26, 127.28, 127.25, 124.04, 124.01, 123.69, 123.56, 115.79, 115.58, 69.85, 69.83, 62.12, 55.06, 22.94, 20.76, 14.07. ESI-HRMS calculated for $C_{15}H_{19}NO_5F^+$ ($[M+H]^+$): 312.1242, found 312.1233.

(2*S*,3*R*)-ethyl-2-acetamido-3-acetoxy-3-(naphthalen-2-yl)propanoate (2k)



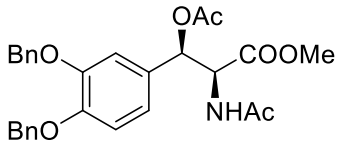
White solid; 17.1 mg; 99% yield; >99% ee; $[\alpha]_D^{20}$ = -4.88 (c = 0.47, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralpak OD-H column, hexane: isopropanol = 95:5; flow rate = 1.0 mL/min; UV detection at 210 nm; t_R = 32.6 min (major), 46.9 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.81-7.85 (m, 3H), 7.75 (s, 1H), 7.42-7.50 (m, 3H), 6.45 (d, *J* = 4.2 Hz, 1H), 6.18 (d, *J* = 9.3 Hz, 1H), 5.18 (dd, *J* = 9.3 Hz, 4.2 Hz, 1H), 4.14 (q, *J* = 7.1 Hz, 2H), 2.18 (s, 3H), 1.93 (s, 3H), 1.16 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 169.88, 169.59, 169.58, 133.34, 133.26, 132.96, 128.52, 128.05, 127.81, 126.50, 125.61, 123.78, 74.81, 62.01, 56.33, 23.03, 20.95, 14.00. ESI-HRMS calculated for $C_{19}H_{22}NO_5^+$ ($[M+H]^+$): 344.1492, found 344.1490.

(2*S*,3*R*)-methyl-2-acetamido-3-acetoxy-3-(3,4-dimethoxyphenyl)propanoate (2l)



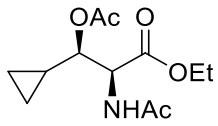
White solid; m. p. 139-140 □; 16.6 mg; 98% yield; >99% ee; $[\alpha]_D^{20}$ = -7.75 (c = 0.80, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralpak OJ-H column, hexane: isopropanol = 80:20; flow rate = 1.0 mL/min; UV detection at 210 nm; t_R = 23.0 min (minor), 35.8 min (major). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.81-6.87 (m, 3H), 6.20 (d, *J* = 4.6 Hz, 1H), 6.17 (d, *J* = 9.4 Hz, 1H), 5.06 (dd, *J* = 9.3, 4.6 Hz, 1H), 3.88 (s, 3H), 3.87 (s, 3H), 3.68 (s, 3H), 2.13 (s, 3H), 1.99 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.16, 169.86, 169.71, 149.24, 148.97, 128.24, 118.93, 110.85, 109.48, 74.38, 56.32, 55.99, 55.86, 52.74, 23.06, 20.95. ESI-HRMS calculated for $C_{16}H_{22}NO_7^+$ ($[M+H]^+$): 340.1388, found 340.1386.

(2*S*,3*R*)-methyl -2-acetamido-3-acetoxy-3-(3,4-bis(benzyloxy)phenyl)propanoate (2m)



White solid; m. p. 89-94 □; 24.3 mg; 99% yield; >99% ee; $[\alpha]_D^{20}$ = -4.60 (c = 1.00, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralpak OD-H column, hexane: isopropanol = 80:20; flow rate = 1.0 mL/min; UV detection at 210 nm; t_R = 19.8 min (major), 36.3 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42-7.45 (m, 4H), 7.34-7.39 (m, 4H), 7.30-7.32 (m, 2H), 6.87-6.89 (m, 2H), 6.81 (dd, *J* = 8.4, 1.9 Hz, 1H), 6.14 (d, *J* = 4.5 Hz, 1H), 6.08 (d, *J* = 9.2 Hz, 1H), 5.14 (s, 1H), 5.00 (dd, *J* = 9.2, 4.5 Hz, 1H), 3.63 (s, 3H), 2.06 (s, 3H), 1.93 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.11, 169.85, 169.59, 149.21, 148.80, 137.08, 137.01, 128.87, 128.56, 128.54, 127.91, 127.89, 127.35, 127.28, 119.72, 114.44, 113.30, 74.17, 71.46, 71.07, 56.31, 52.70, 23.04, 20.89. ESI-HRMS calculated for $C_{28}H_{30}NO_7^+$ ($[M+H]^+$): 492.2014, found 492.2012.

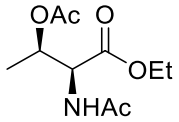
(2*S*,3*R*)-ethyl-2-acetamido-3-acetoxy-3-cyclopropylpropanoate (2n)



White solid; 12.5 mg; 97% yield; >99% ee; $[\alpha]_D^{20}$ = -61.80 (c = 0.23, $CHCl_3$); The enantiomeric excess was determined by GC on Gamma-Dex 225 column; flow rate = 2.0 mL/min; 160 ℃ isothermal; t_R = 33.4 min (major), 33.7 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.22 (d, *J* = 9.1 Hz, 1H), 4.97 (dd, *J* = 9.4 Hz, 3.0 Hz, 1H),

4.65 (dd, *J* = 9.5, 3.0 Hz, 1H), 4.16 (q, *J* = 7.2 Hz, 2H), 2.11 (s, 3H), 2.06 (s, 3H), 1.25 (t, *J* = 7.1 Hz, 3H), 1.05-1.11 (m, 1H), 0.39-0.66 (m, 4H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.28, 169.97, 169.95, 78.02, 61.91, 55.28, 23.26, 20.91, 14.08, 12.79, 4.26, 3.47. ESI-HRMS calculated for $C_{12}H_{20}NO_5^+$ ($[M+H]^+$): 258.1336, found 258.1335.

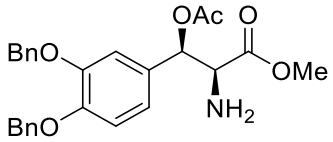
ethyl *N,O*-diacetyl-$_L$-threoninate (2o)



Yellow oil; 11.3 mg; 98% yield; >99% ee; $[\alpha]_D^{20}$ = -62.33 (c = 0.65, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralpak OJ-H column, hexane: isopropanol = 95:5; flow rate = 1.0 mL/min; UV detection at 210 nm; t_R = 14.1 min (major), 16.5 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.17 (d, *J* = 8.9 Hz, 1H), 5.45 (m, 1H), 4.79 (dd, *J* = 9.4 Hz, 2.6 Hz, 1H), 4.19 (q, *J* = 7.1 Hz, 2H), 2.11 (s, 3H), 2.01 (s, 3H), 1.25-1.28 (m, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ = 170.42, 169.95, 169.71, 70.56, 61.91, 55.40, 23.18, 20.92, 17.07, 14.07. MS calculated for $C_{10}H_{18}NO_5^+$ ($[M+H]^+$): 232.1, found 232.1.

4. Procedure for the Synthesis of Compound 4

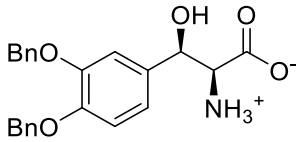
(2*S*,3*R*)-methyl -3-acetoxy-2-amino-3-(3,4-bis(benzyloxy)phenyl)propanoate (3)



A solution of **2m** (147.5 mg, 0.3 mmol) in dry CH_2Cl_2 (2.5 mL) was treated with trimethyloxonium tetrafluoroborate (49 mg, 0.33 mmol) in one portion and the resulting homogeneous colorless solution was stirred at room temperature for 7 h. Evaporation of the solution to obtain the pale solid, then treatment of a chloroform solution of the solid with cold aqueous sodium bicarbonate and processing the organic phase gave the crude free base. After flash column chromatography (PE/EA 1/1), the product **3** (90 mg) was obtained, yielding 66% yield.[6] $[\alpha]_D^{20}$ = -84.00 (c = 0.90, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44 (m, 4H), 7.30-7.38 (m, 6H), δ 6.91 (d, *J* = 8.2 Hz, 1H), 6.87 (d, *J* = 1.9 Hz, 1H), 6.84 (dd, *J* = 8.3, 1.9 Hz, 1H), 5.57 (d, *J* = 7.6 Hz, 1H), 5.16 (s, 4H), 4.54 (dd, *J* = 7.6, 1.3 Hz, 1H), 3.80 (s, 3H), 2.09 (d, *J* = 1.3 Hz,

3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.45, 166.93, 149.27, 149.21, 137.02, 136.97, 132.44, 128.56, 127.95, 127.91, 127.42, 127.24, 119.08, 114.84, 112.50, 83.03, 76.19, 71.42, 71.19, 52.80, 14.16. ESI-HRMS calculated for $C_{26}H_{27}NO_6Na^+$ ($[M+Na]^+$): 472.1731, found 472.1715.

(2*S*,3*R*)-2-ammonio-3-(3,4-bis(benzyloxy)phenyl)-3-hydroxypropanoate (4)



To a solution of **3** (37 mg, 0.08 mmol) in MeOH (2.0 mL) was added dropwise 2 N NaOH (2.0 mL) at 0 □. The heterogeneous milky reaction mixture was stirred overnight at 40 □. Then the mixture was cooled to 0 □, and neutralized to pH 6-7 by slowly addition of 1 N HCl. The resulting white precipitate was filtered and washed with cold water and ether to give 30 mg (92%) of **4**. [7] $[\alpha]_D^{25}$ = +2.00 (c = 0.20, DMSO). ^{1}H NMR (400 MHz, DMSO-*d*$_6$) δ 10.08 (d, *J* = 10.2 Hz, 1H), 7.46 (m, 4H), 7.38 (m, 6H), 7.08 (d, *J* = 1.8 Hz, 1H), 6.95 (d, *J* = 8.3 Hz, 1H), 6.81 (dd, *J* = 8.3, 1.8 Hz, 1H), 6.02 (d, *J* = 14.7 Hz, 1H), 5.15 (s, 2H), 5.10 (s, 2H), 5.09 (s, 1H); ^{13}C NMR (101 MHz, DMSO) δ 167.59, 149.00, 147.28, 137.87, 137.84, 130.54, 128.94, 128.83, 128.19, 128.03, 127.93, 122.91, 118.91, 115.24, 111.43, 111.19, 70.56, 70.42, 23.18, 22.89. ESI-HRMS calculated for $C_{23}H_{24}NO_5^+$ ($[M+H]^+$): 394.1649, found 394.1637.

Compared the optical rotation of **2m** with the literature[7], the absolute configuration of **2** is determined as (2*S,*3*R*).

5. References

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[3] H. Ryu, H. Kuriyama, H. Miyazato, S. Minakata, M. Komatsu, J. Y. Yoon, S. Kim, *Bull. Chem. Soc. Jpn.* **2004**, *77*, 1407.

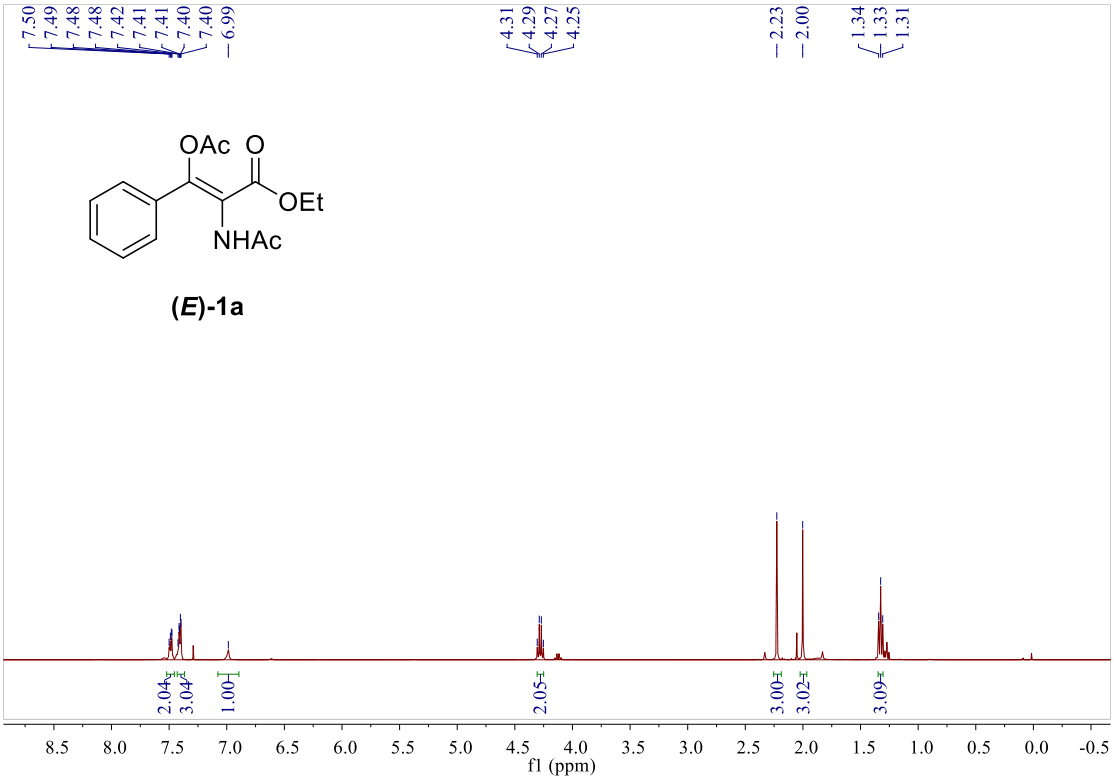
[4] J. P. Burkhart, J. R. Koehl, S. Mehdi, S. L. Durham, M. J. Janusz, E. W. Huber, et al. *J. Med. Chem.* **1995,** *38*, 223-233.

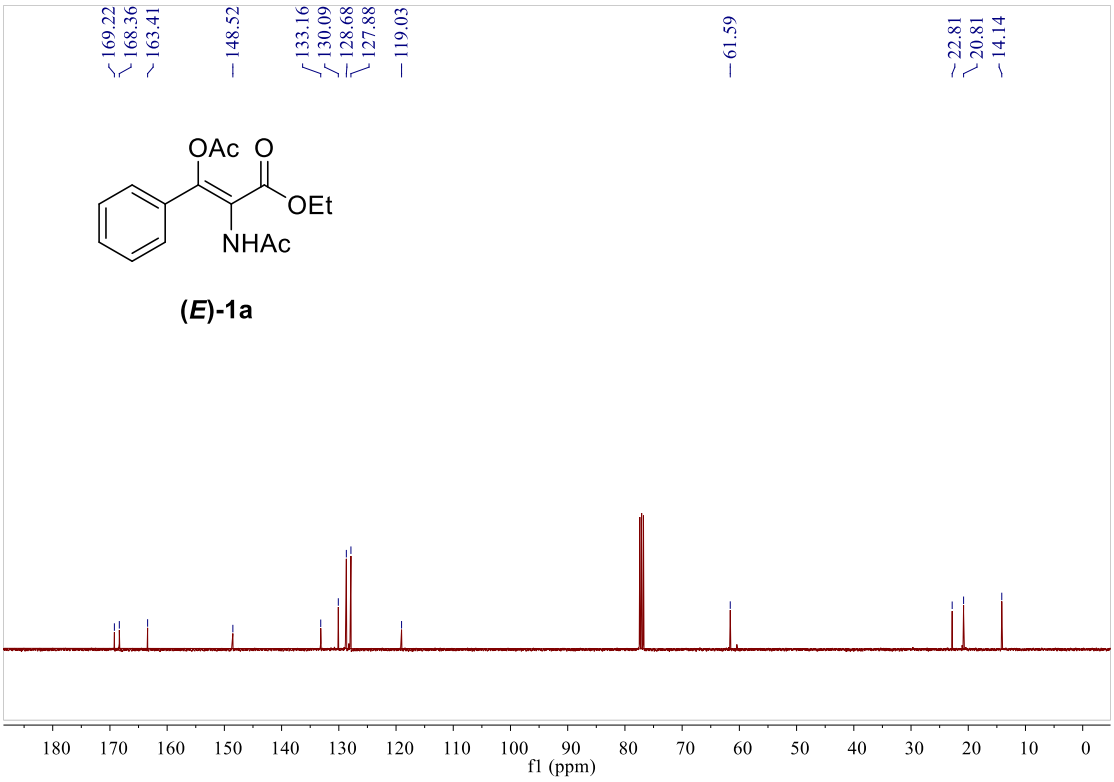
[5] T. Katoh, E. ltoh, T. Yoshino, S. Terashima, *Tetrahedron*, **1997**, *53*, 10229-10238.

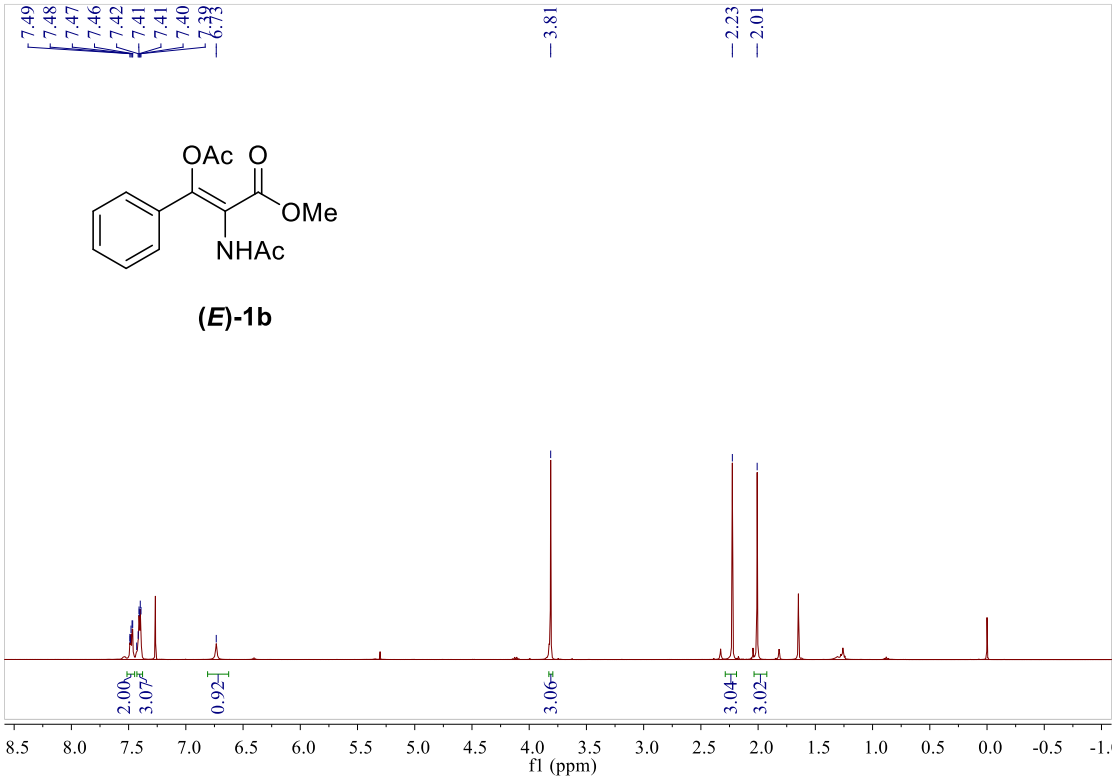
[6] S. Hanessian, *Tetrahedron Lett*. **1967**, 1549-1552.

[7] B. Herbert, I. H. Kim and K. L. Kirk, *J. Org. Chem*. **2001**, *66*, 4892-4897.

6. NMR spectra of (*E*)-1, 2 and 3, HPLC spectra or GC spectra of 2



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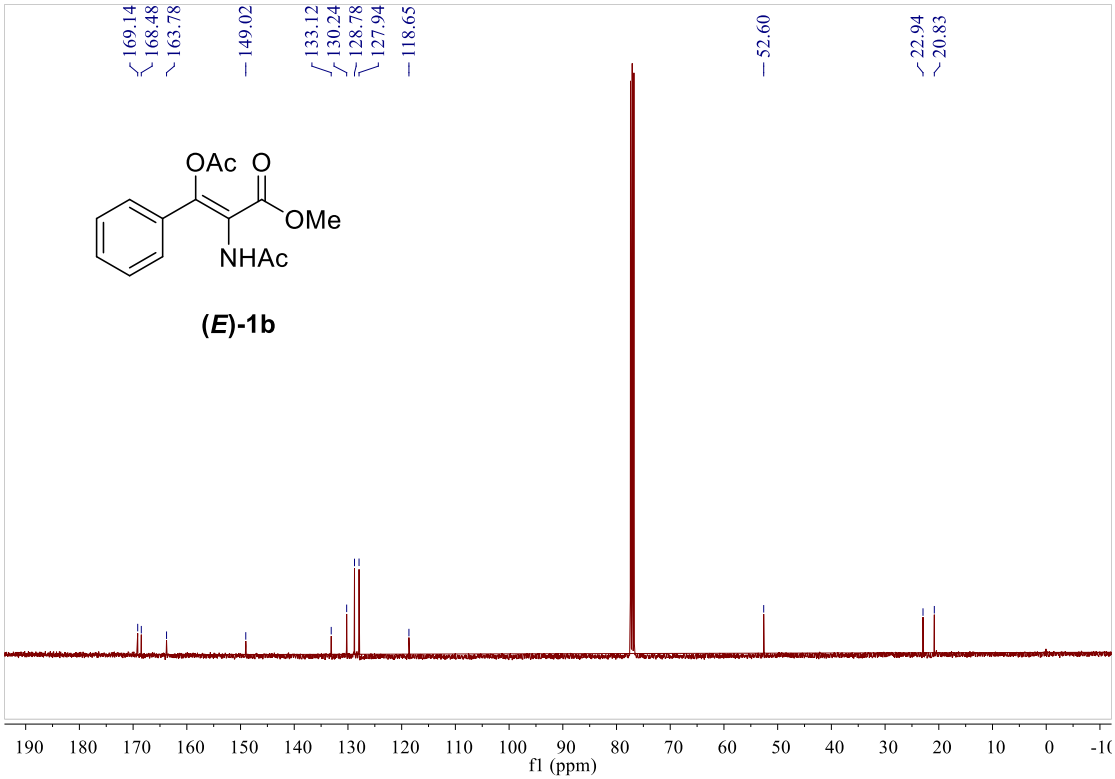
7.46
7.42
7.41
7.41
7.40
7.39
6.73
3.81
2.23
2.01
OAc
O
OMe
NHAc
(E)-1b
2.00
3.07
0.92
3.06
3.04
3.02
f1 (ppm)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52.60
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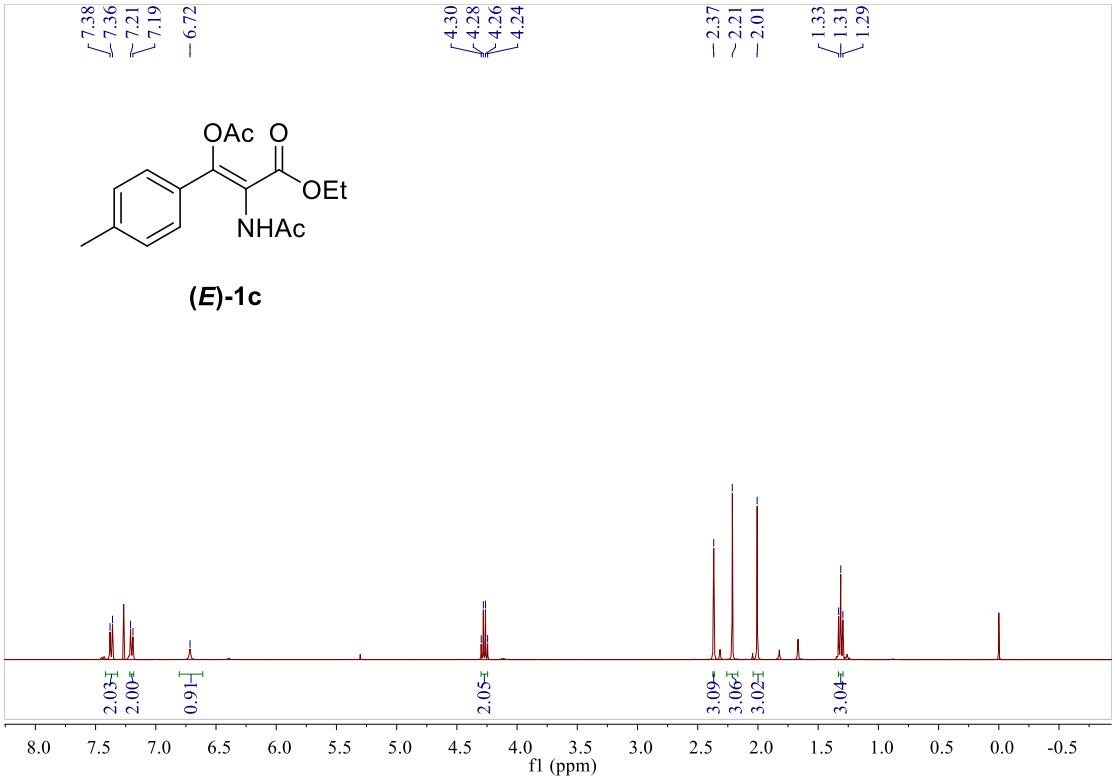
OAc
O
OMe
NHAc
(E)-1b
f1 (ppm)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1.33
1.31
1.29
OAc
O
OEt
NHAc
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2.00
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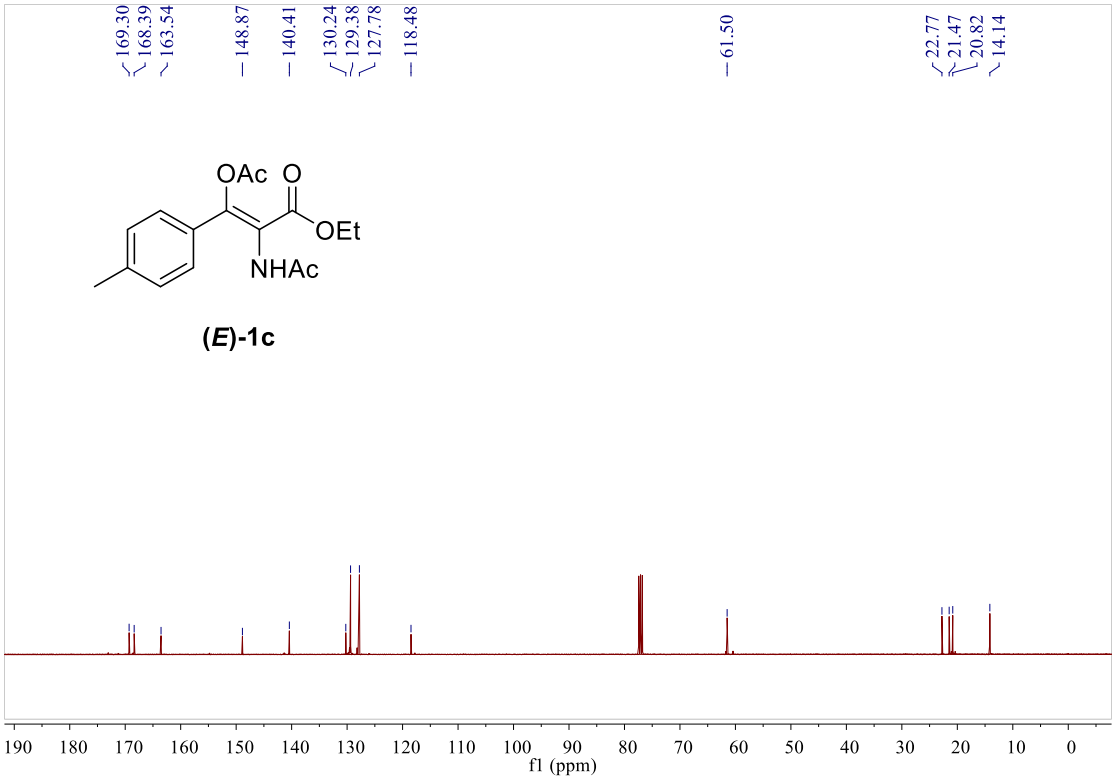
2.05
3.09
3.06
3.02
3.04
f1 (ppm)

(*E*)-1c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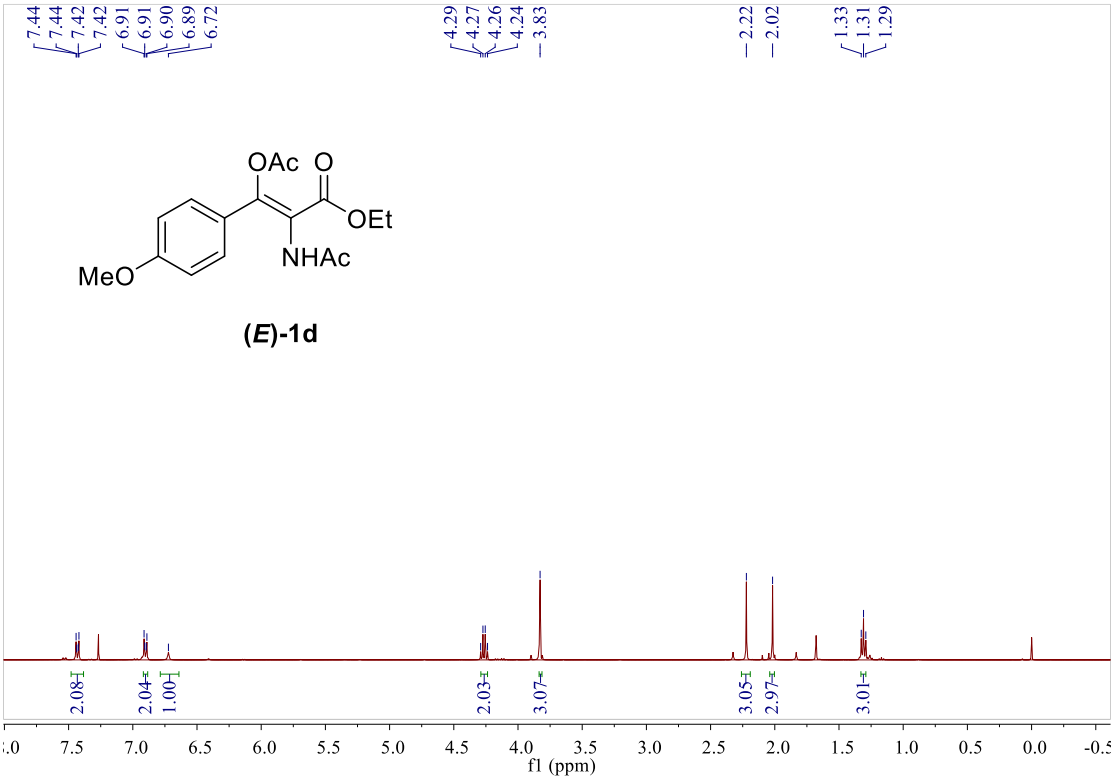
OAc
O
OEt
NHAc
f1 (ppm)

(*E*)-1c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7.42
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4.27
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4.24
3.83
2.22
2.02
1.33
1.31
1.29
OAc
O
OEt
NHAc
MeO
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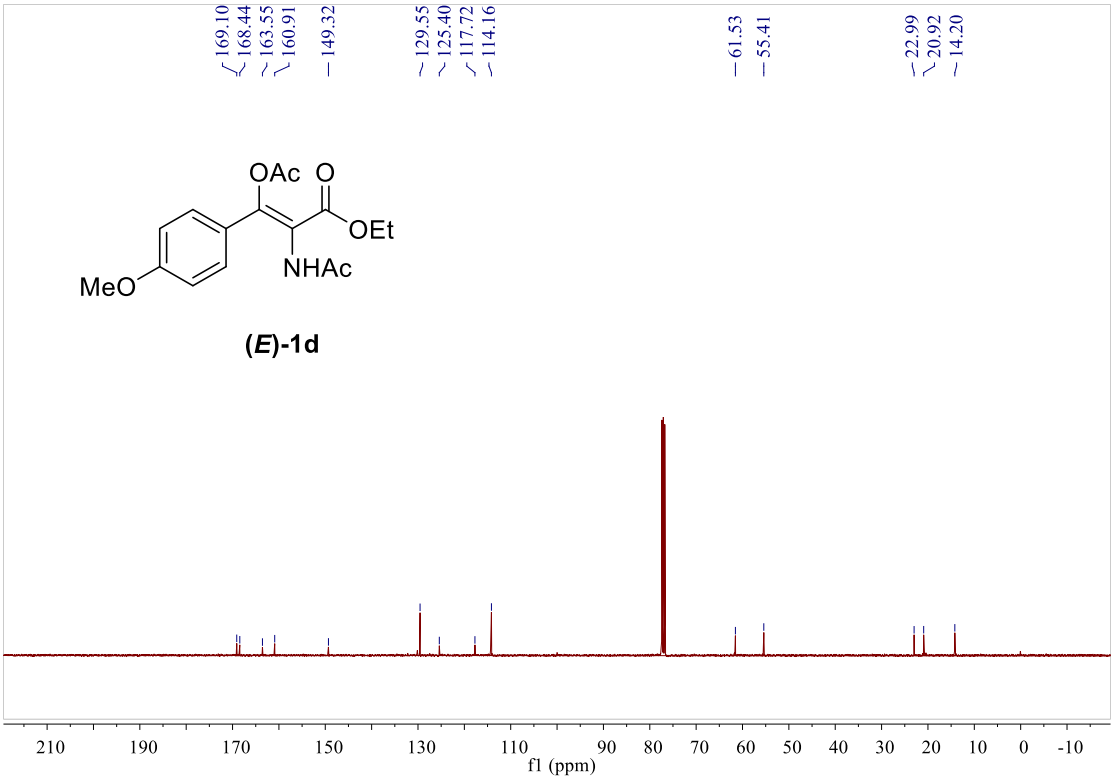
2.04
1.00
2.03
3.07
3.05
2.97
3.01
f1 (ppm)

(*E*)-1d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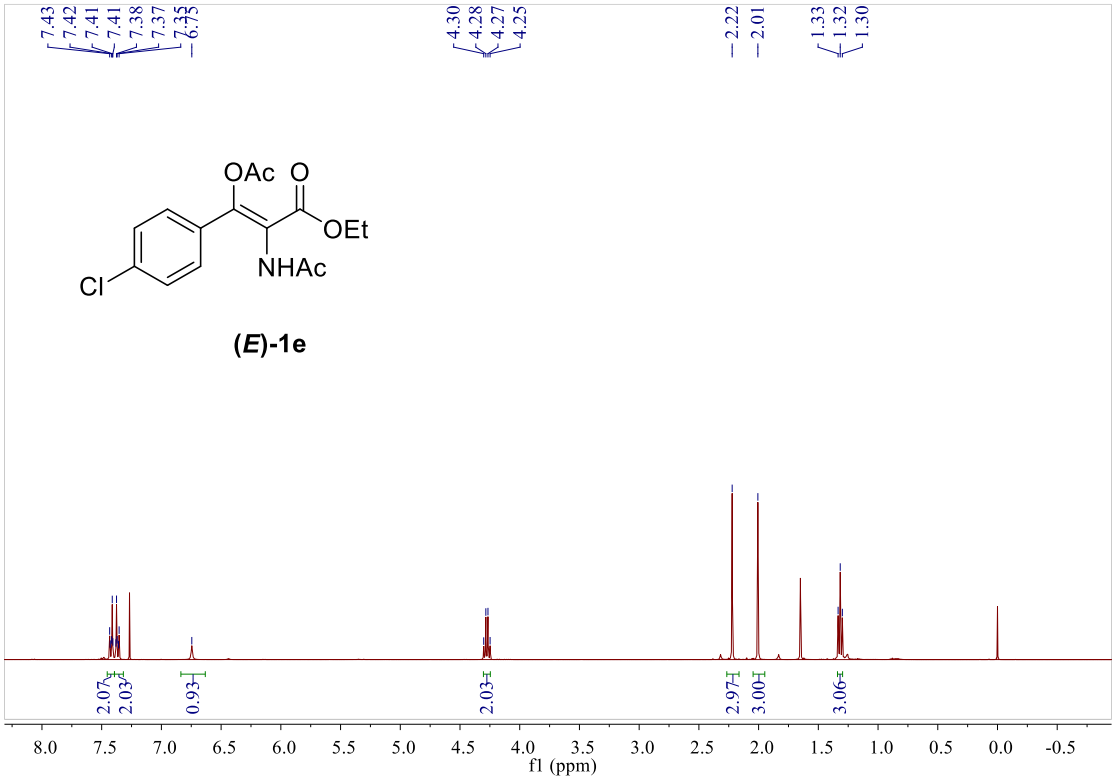
NHAc
MeO
f1 (ppm)

(*E*)-1d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1.33
1.32
1.30
OAc
O
OEt
NHAc
Cl
(E)-1e
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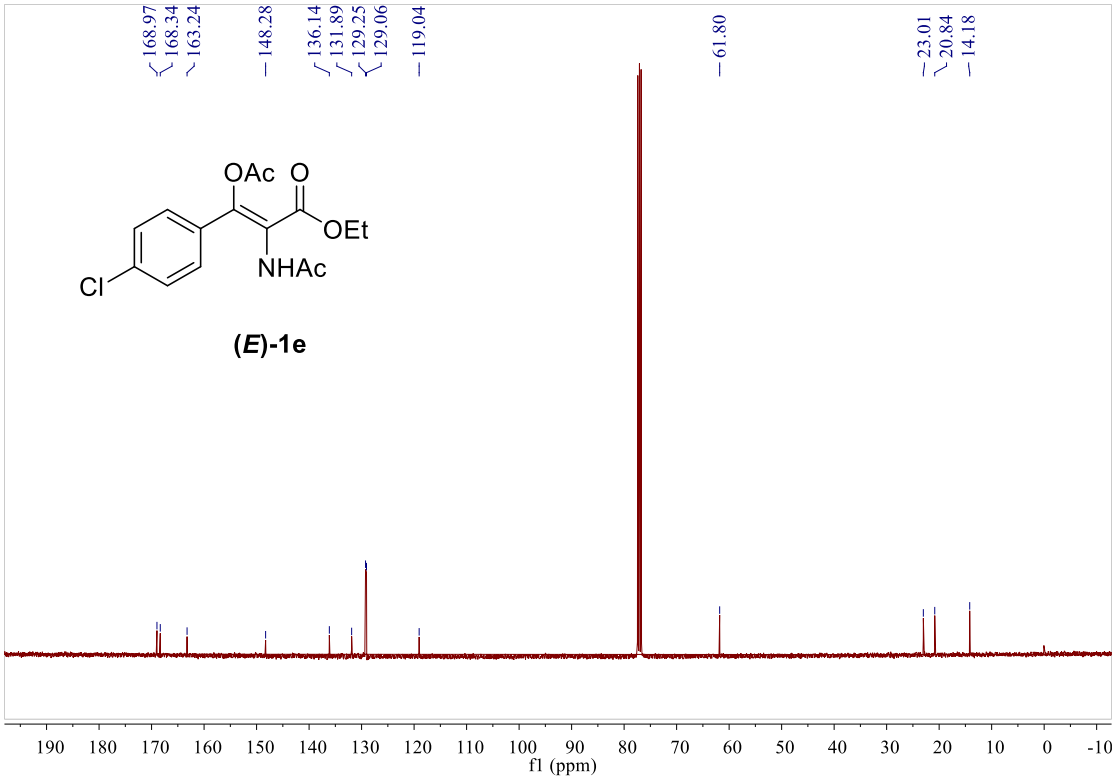
2.97
3.00
3.06
f1 (ppm)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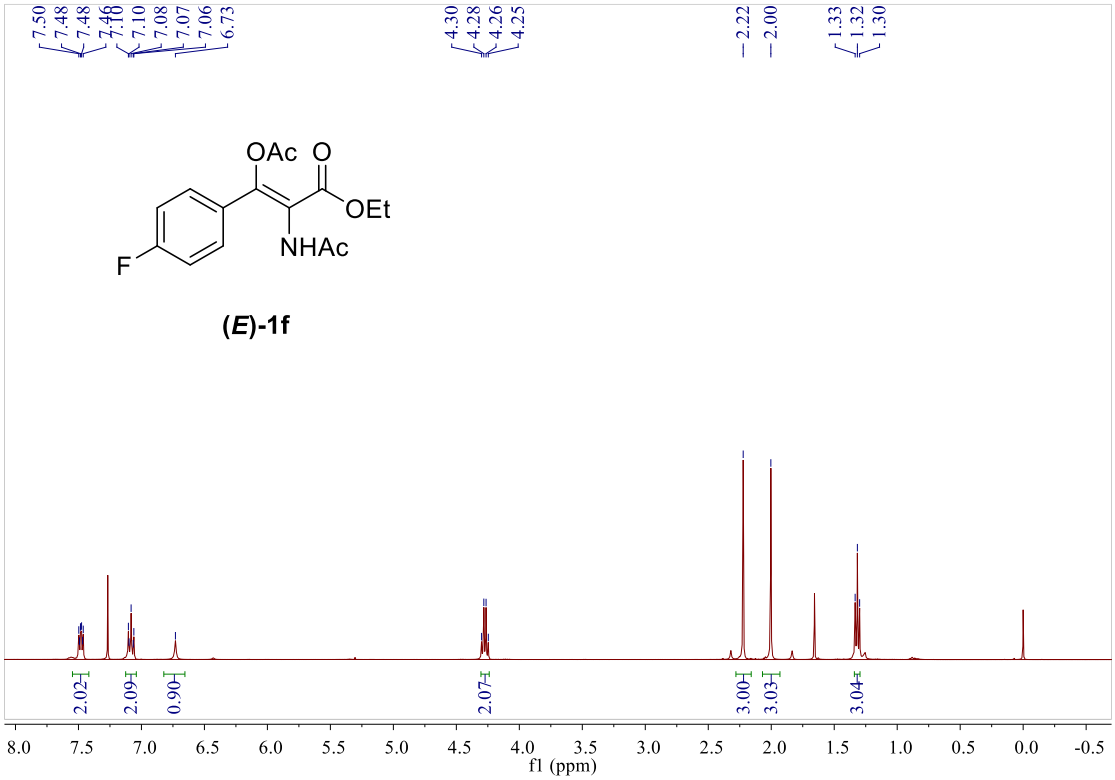
OEt
NHAc
Cl
(E)-1e
f1 (ppm)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1.32
1.30
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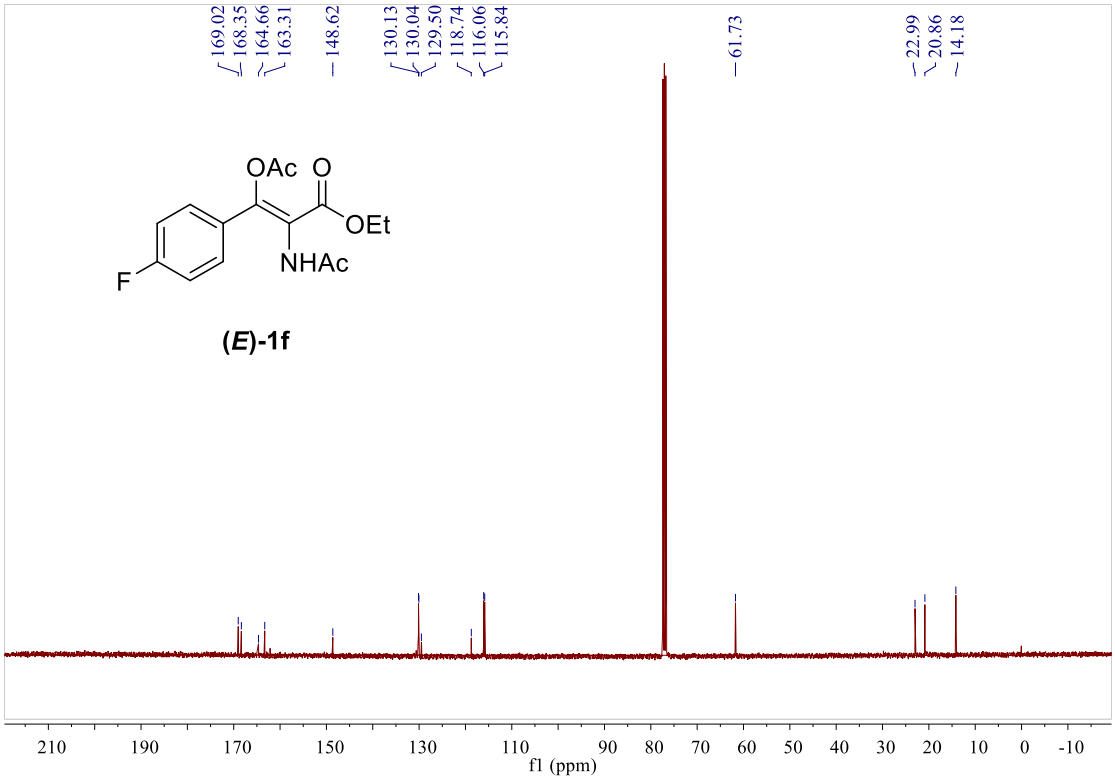
f1 (ppm)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F
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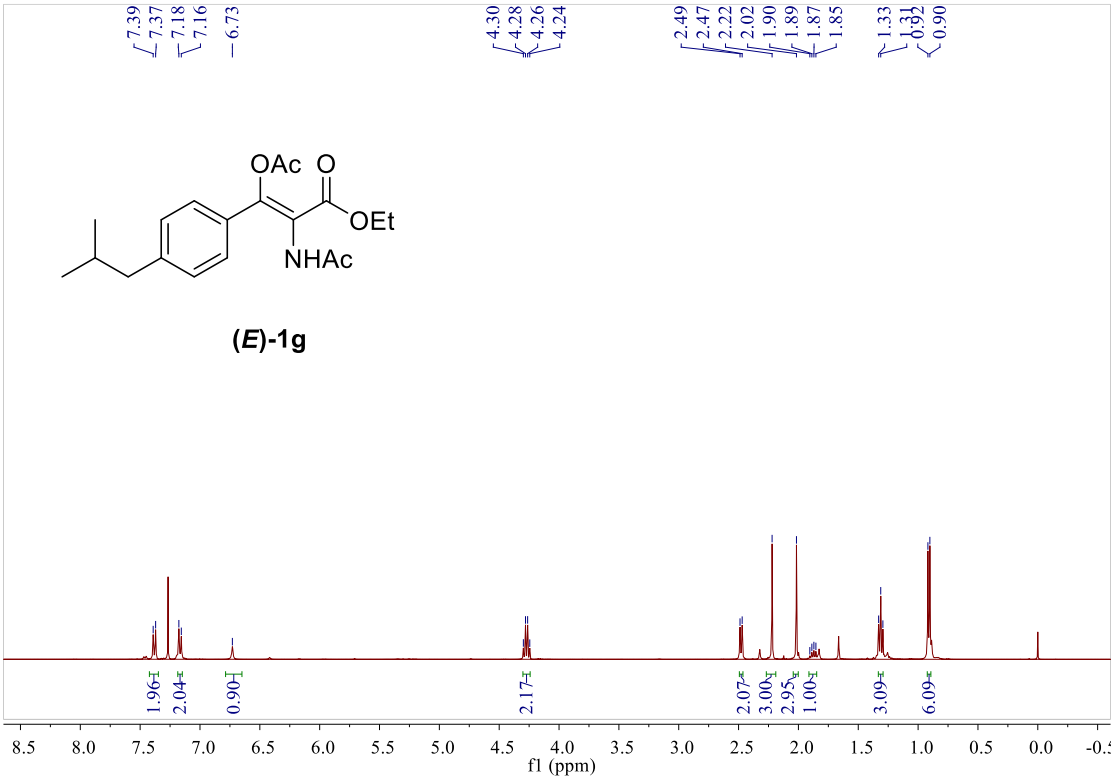
f1 (ppm)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2.02
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1.89
1.87
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0.90
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OEt
NHAc
(E)-1g
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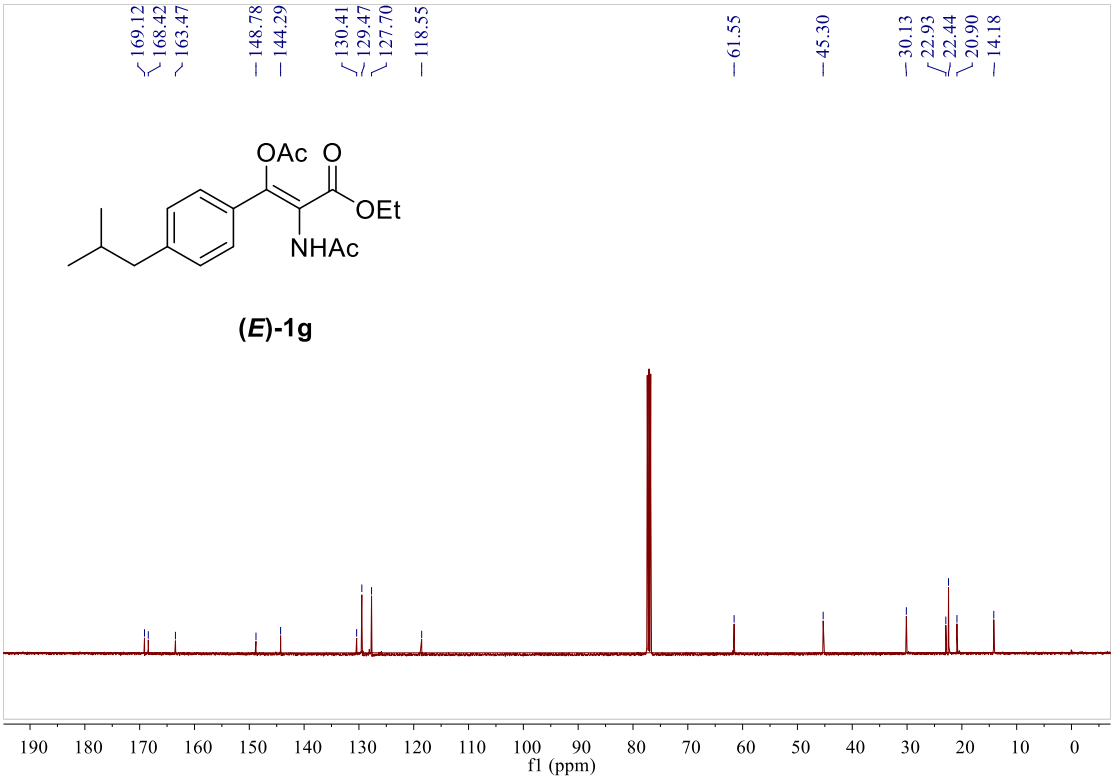
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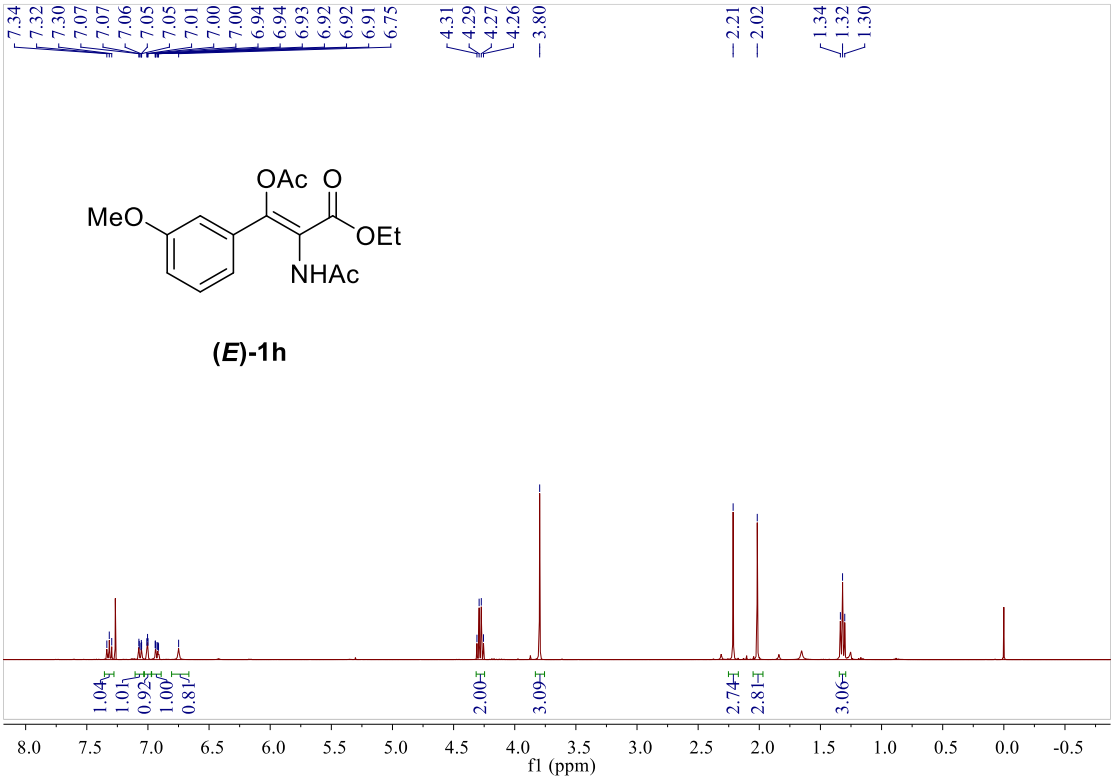
f1 (ppm)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3.06
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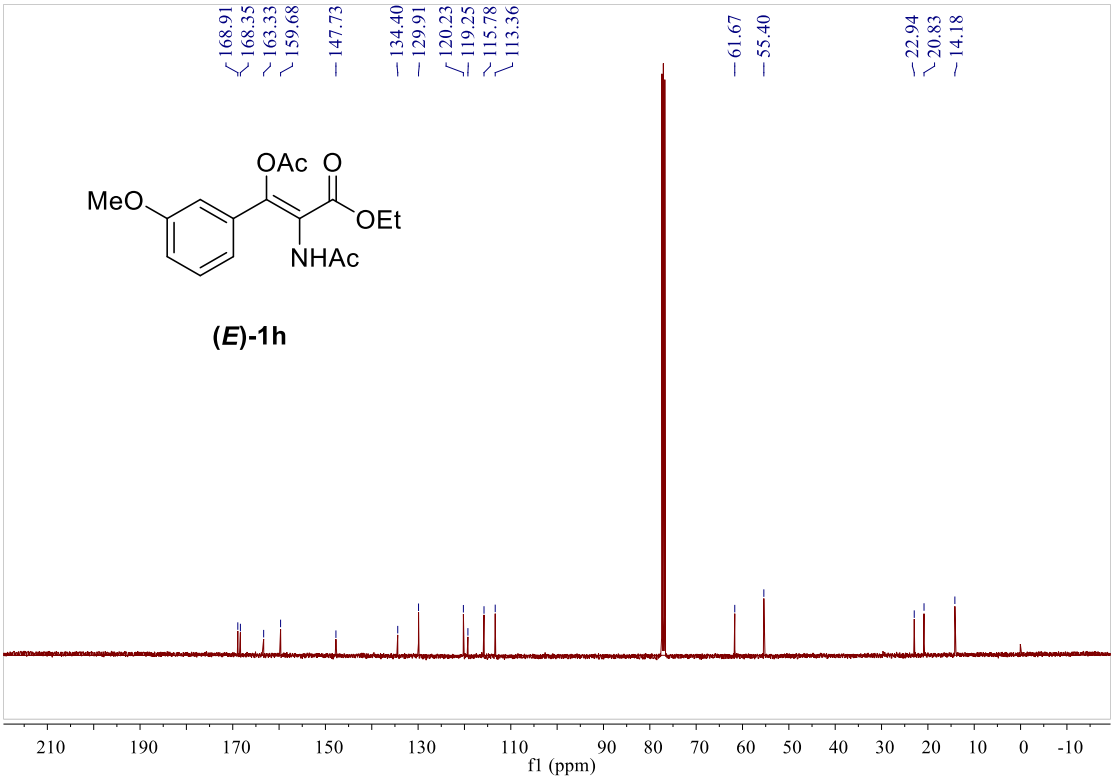
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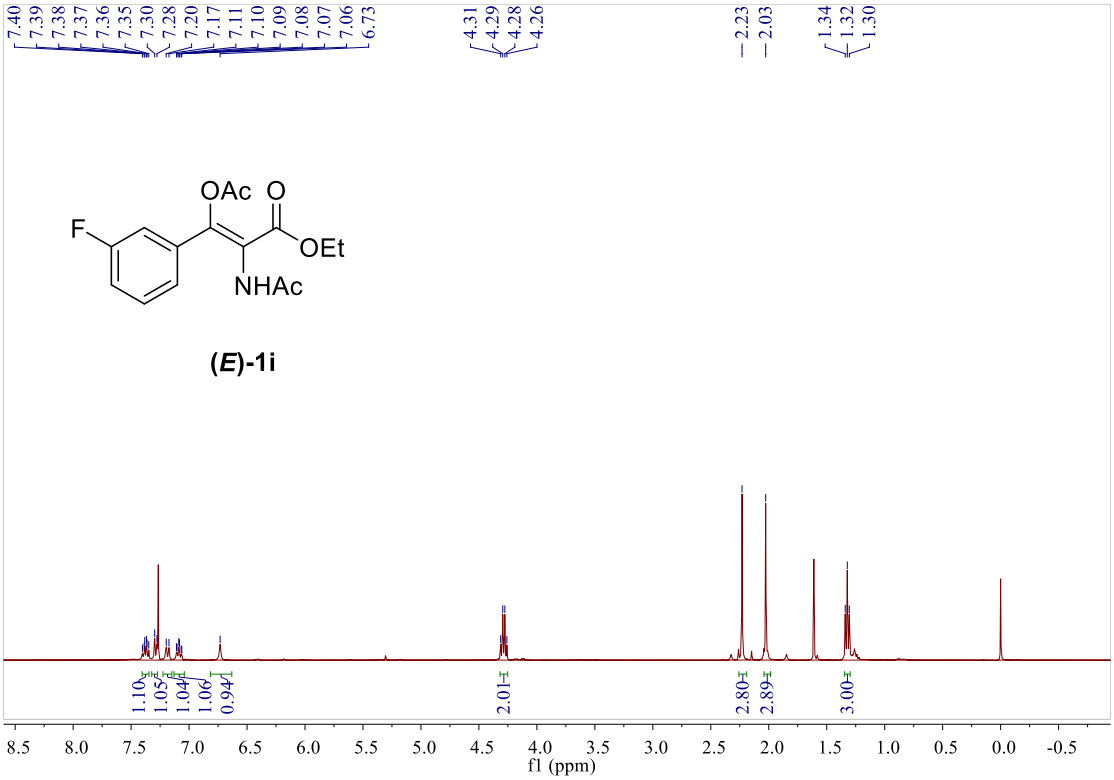
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1.06
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2.80
2.89
3.00
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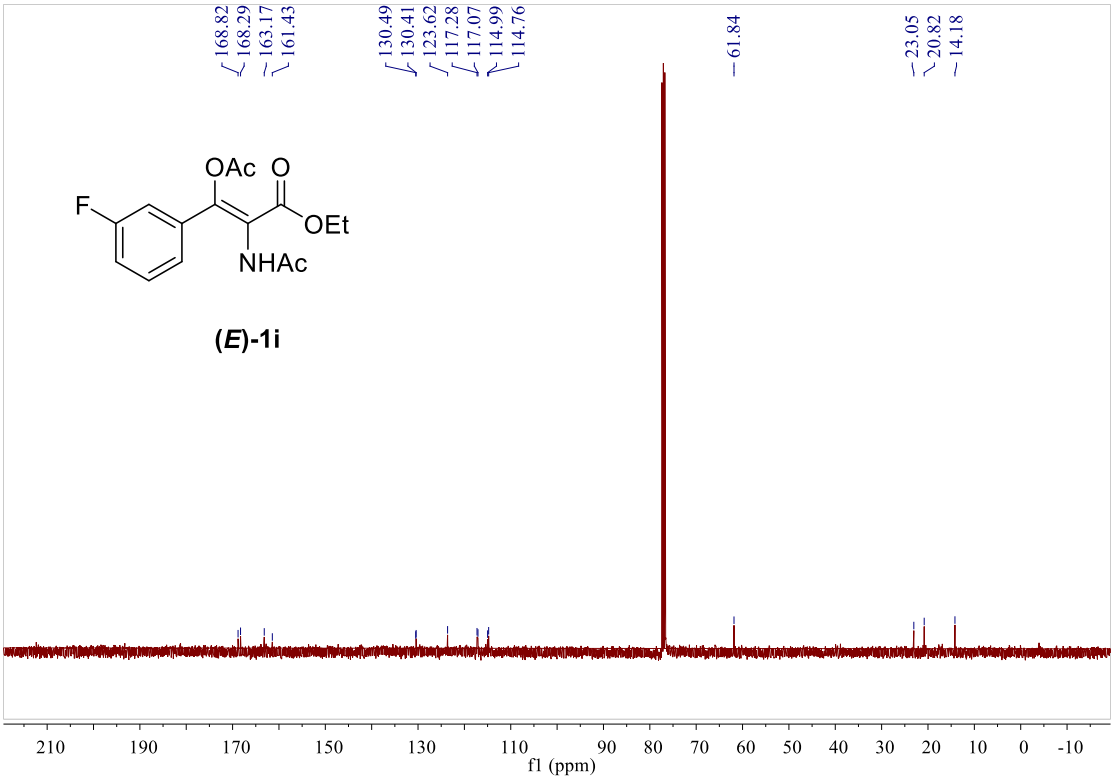
f1 (ppm)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61.84
23.05
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OAc O
F
OEt
NHAc
(E)-1i
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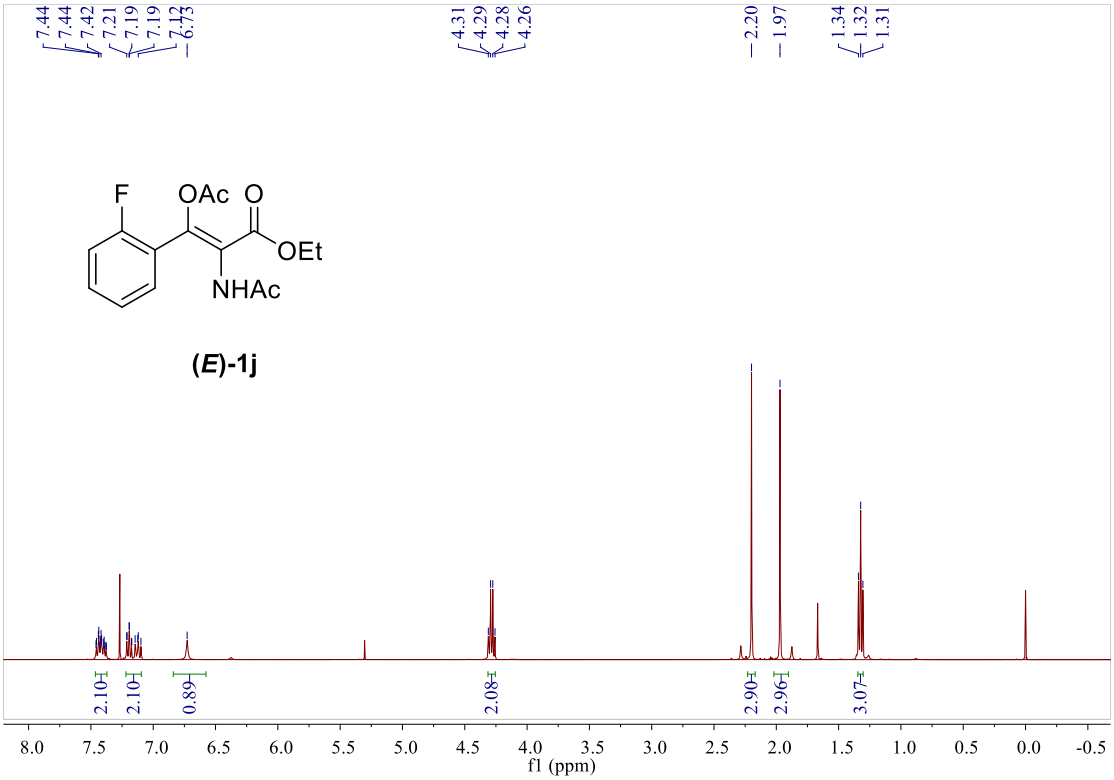
f1 (ppm)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1.97
1.34
1.32
1.31
F
OAc
O
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(E)-1j
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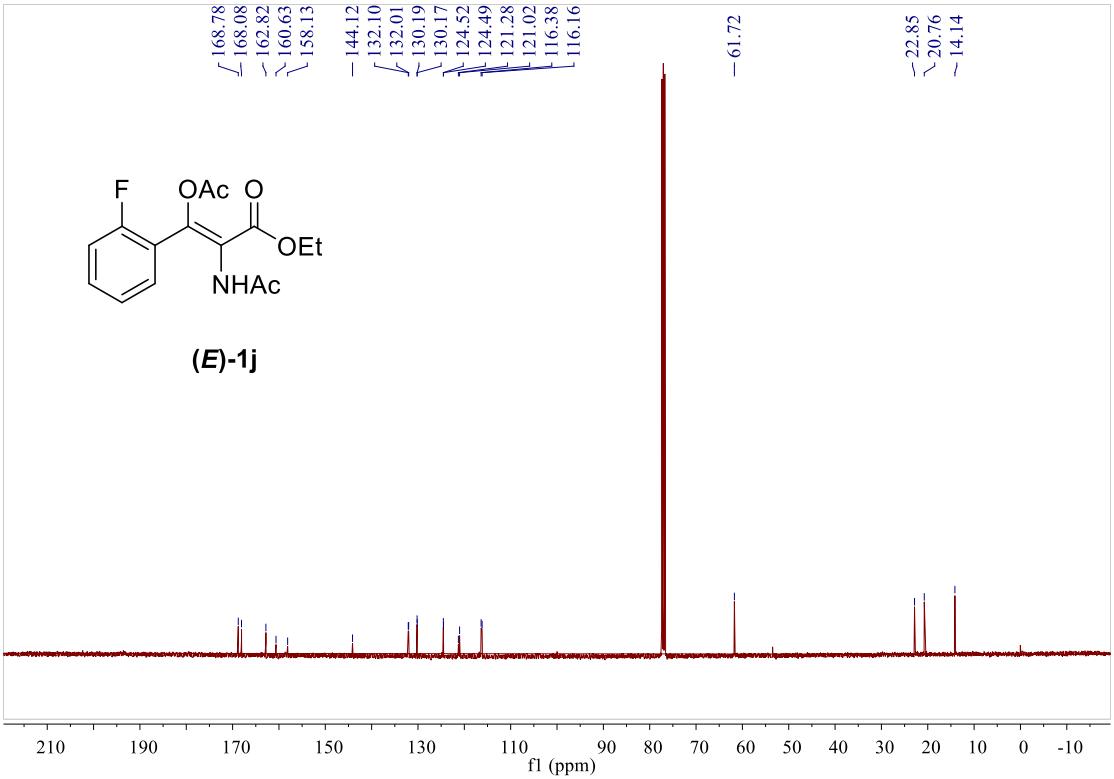
3.07
f1 (ppm)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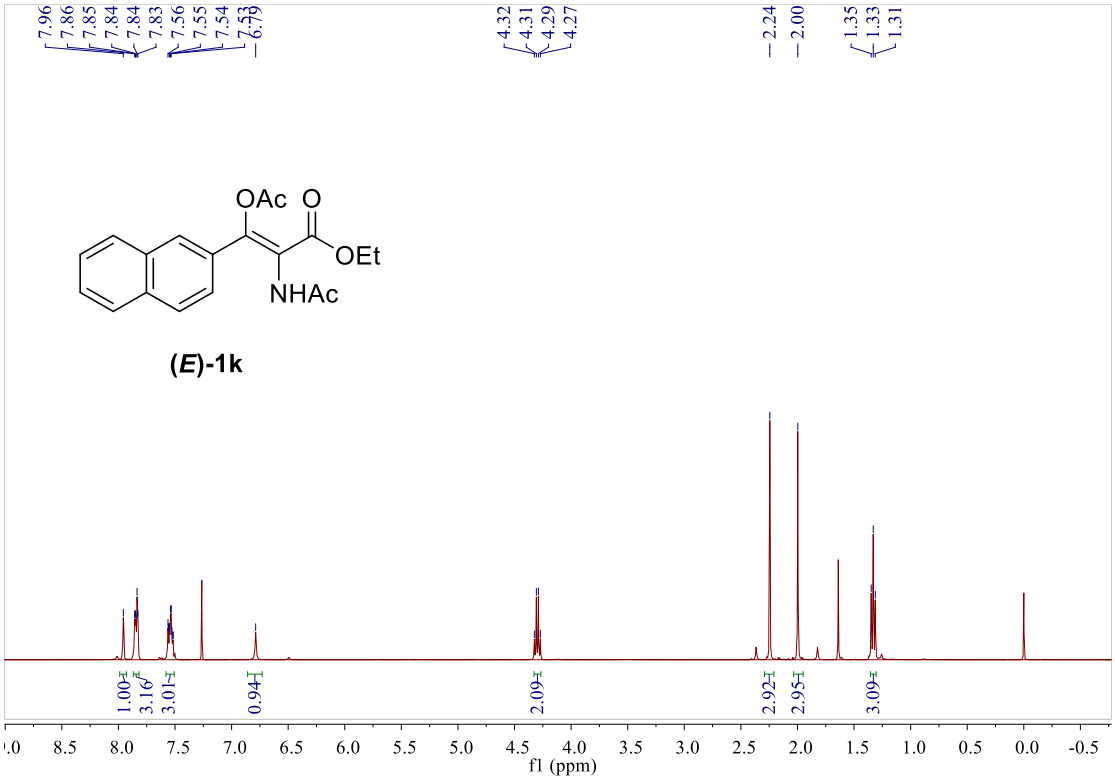
(E)-1j
f1 (ppm)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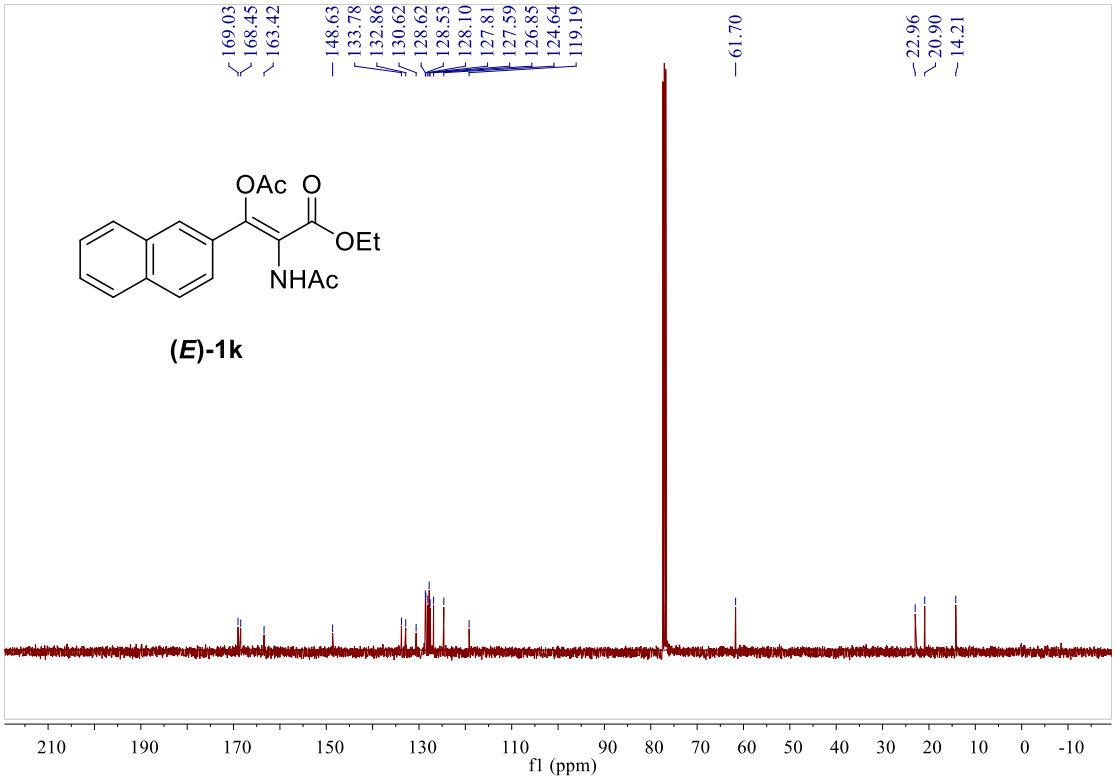
1.35
1.33
1.31
OAc
O
OEt
NHAc
(E)-1k
1.00
3.16
3.01
0.94
2.09
2.92
2.95
3.09
f1 (ppm)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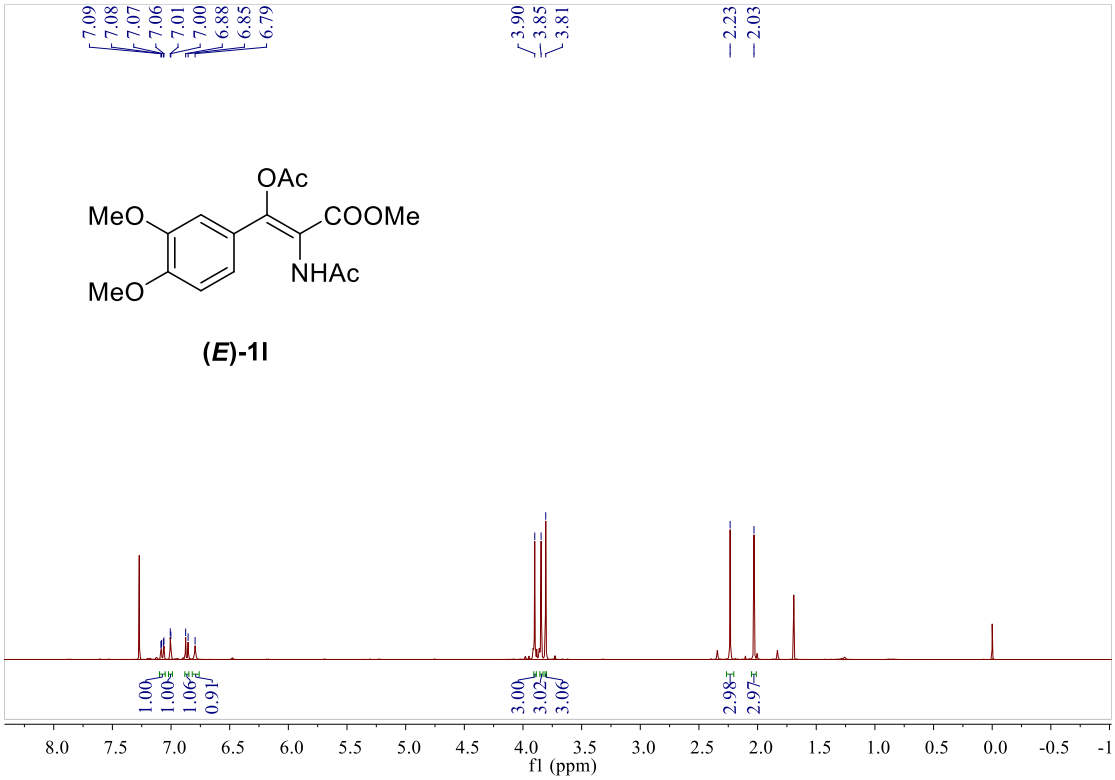
168.45
163.42
148.63
133.78
132.86
130.62
128.62
128.53
128.10
127.81
127.59
126.85
124.64
119.19
61.70
22.96
20.90
14.21
OAc
O
OEt
NHAc
(E)-1k
f1 (ppm)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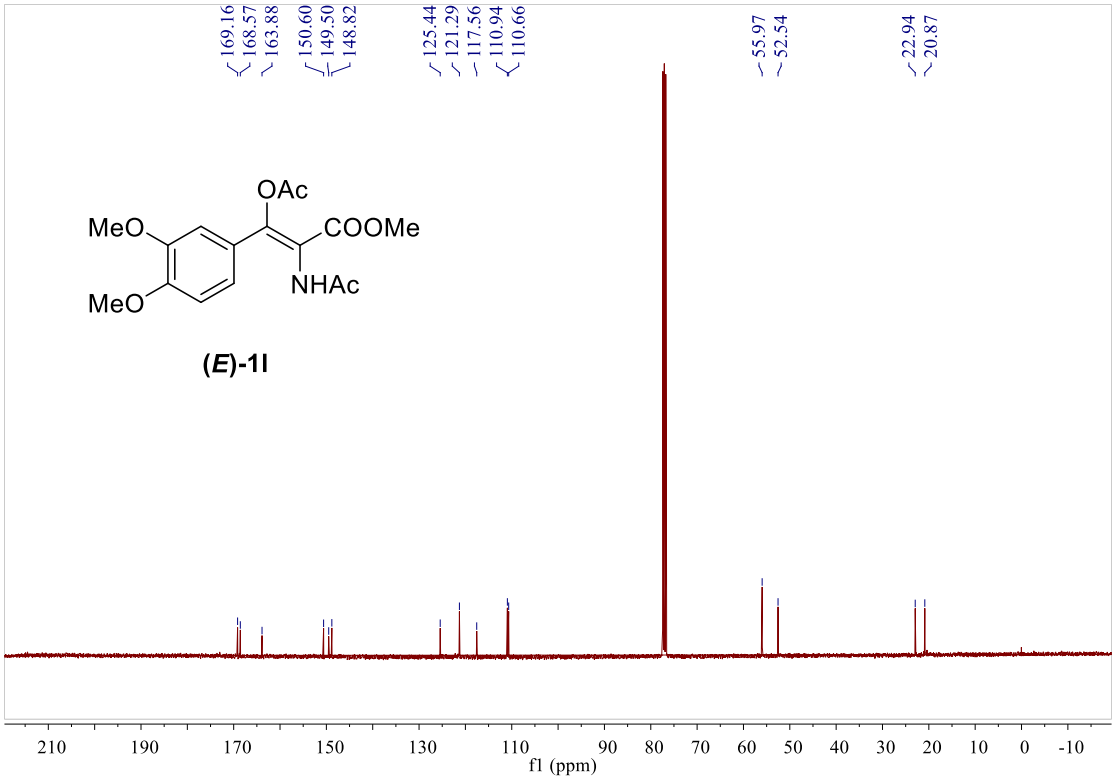
7.08
7.07
7.06
7.01
7.00
6.88
6.85
6.79
3.90
3.85
3.81
2.23
2.03
OAc
MeO
COOMe
NHAc
MeO
(E)-1l
1.00
1.00
1.06
0.91
3.00
3.02
3.06
2.98
2.97
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1
f1 (ppm)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168.57
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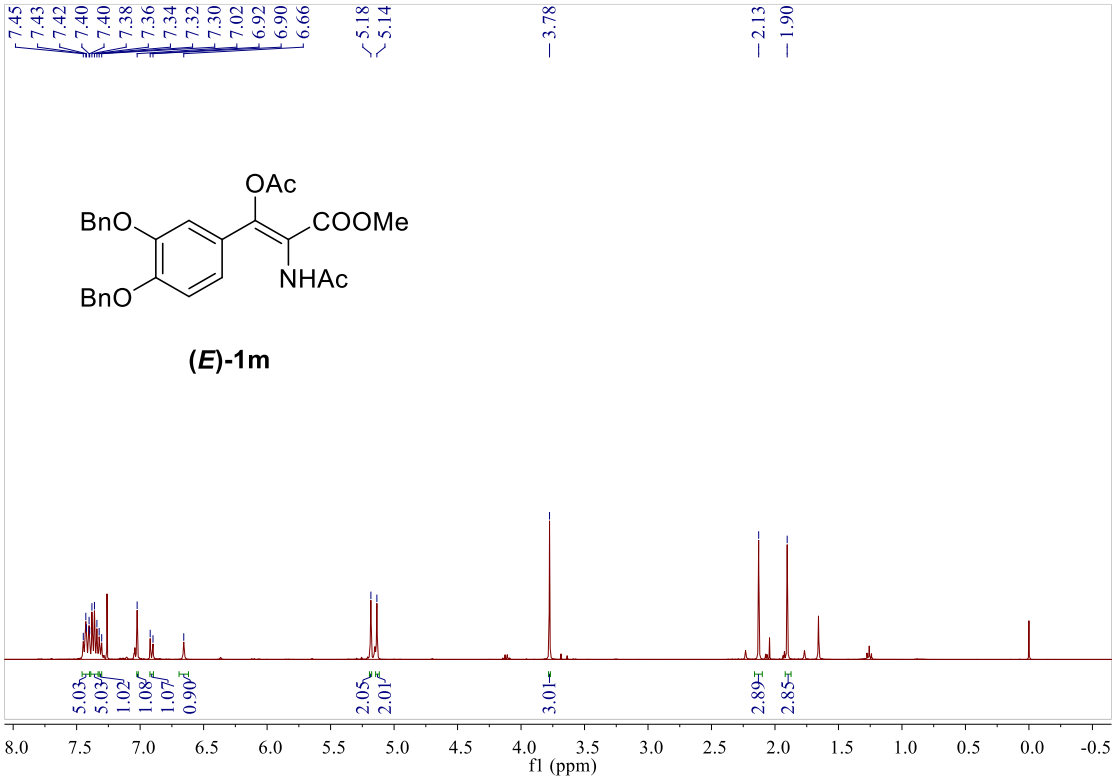
150.60
149.50
148.82
125.44
121.29
117.56
110.94
110.66
55.97
52.54
22.94
20.87
OAc
MeO
COOMe
NHAc
MeO
(E)-1l
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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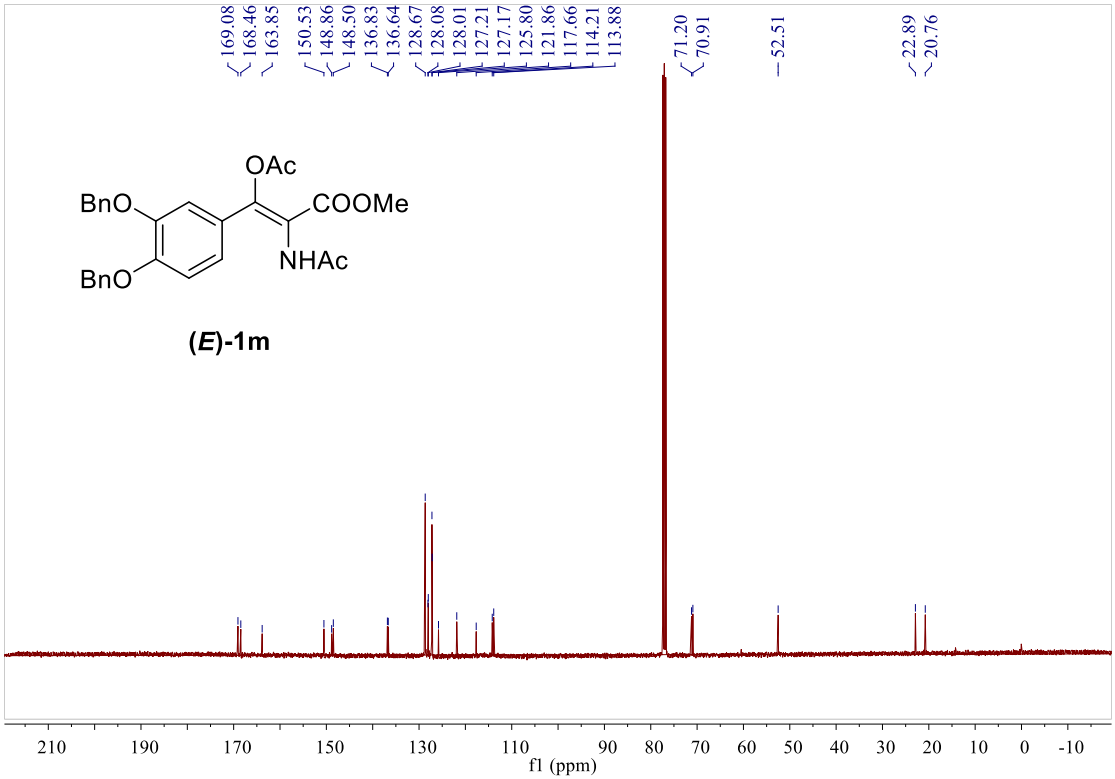
7.43
7.42
7.40
7.40
7.38
7.36
7.34
7.32
7.30
7.02
6.92
6.90
6.66
5.18
5.14
3.78
2.13
1.90
OAc
BnO
COOMe
NHAc
BnO
(E)-1m
5.03
5.03
1.02
1.08
1.07
0.90
2.05
2.01
3.01
2.89
2.85
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)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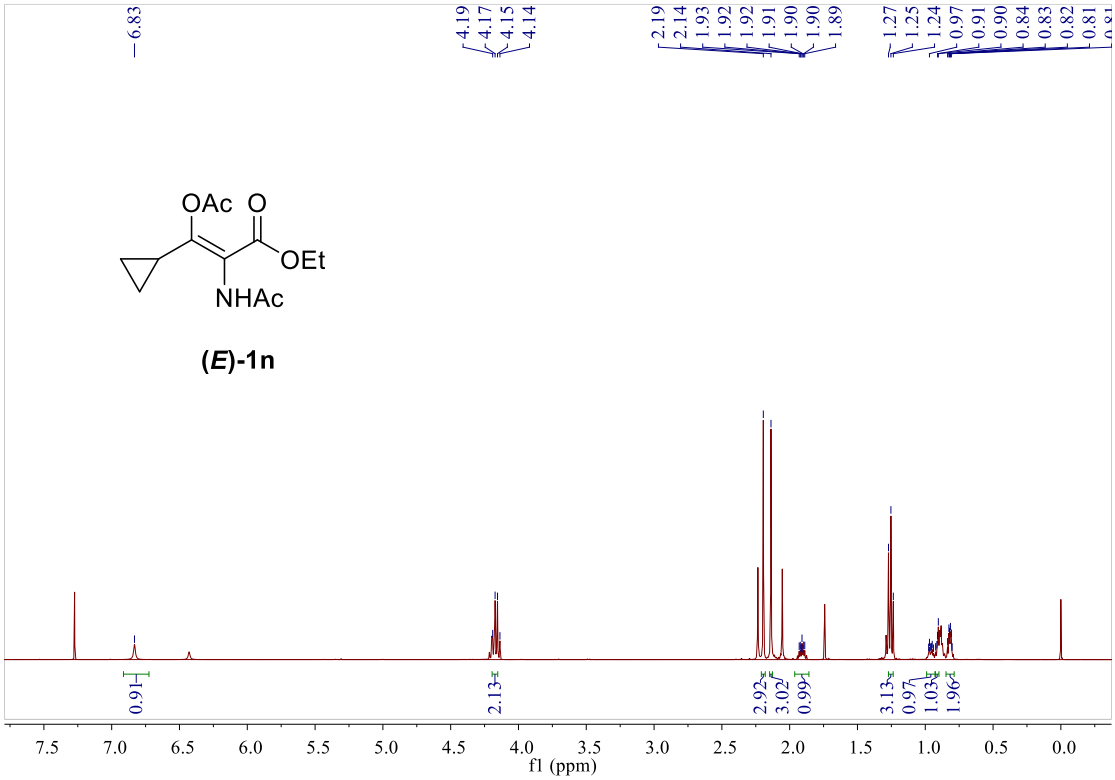
163.85
150.53
148.86
148.50
136.83
136.64
128.67
128.08
128.01
127.21
127.17
125.80
121.86
117.66
114.21
113.88
71.20
70.91
52.51
22.89
20.76
OAc
BnO
COOMe
NHAc
BnO
(E)-1m
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


6.83
4.19
4.17
4.15
4.14
2.19
2.14
1.93
1.92
1.92
1.91
1.90
1.90
1.89
1.27
1.25
1.24
0.97
0.91
0.90
0.84
0.83
0.82
0.81
OAc
O
OEt
NHAc
(E)-1n
0.91
2.13
2.92
3.02
0.99
3.13
0.97
1.03
1.96
7.5
7.0
6.5
6.0
5.5
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4.5
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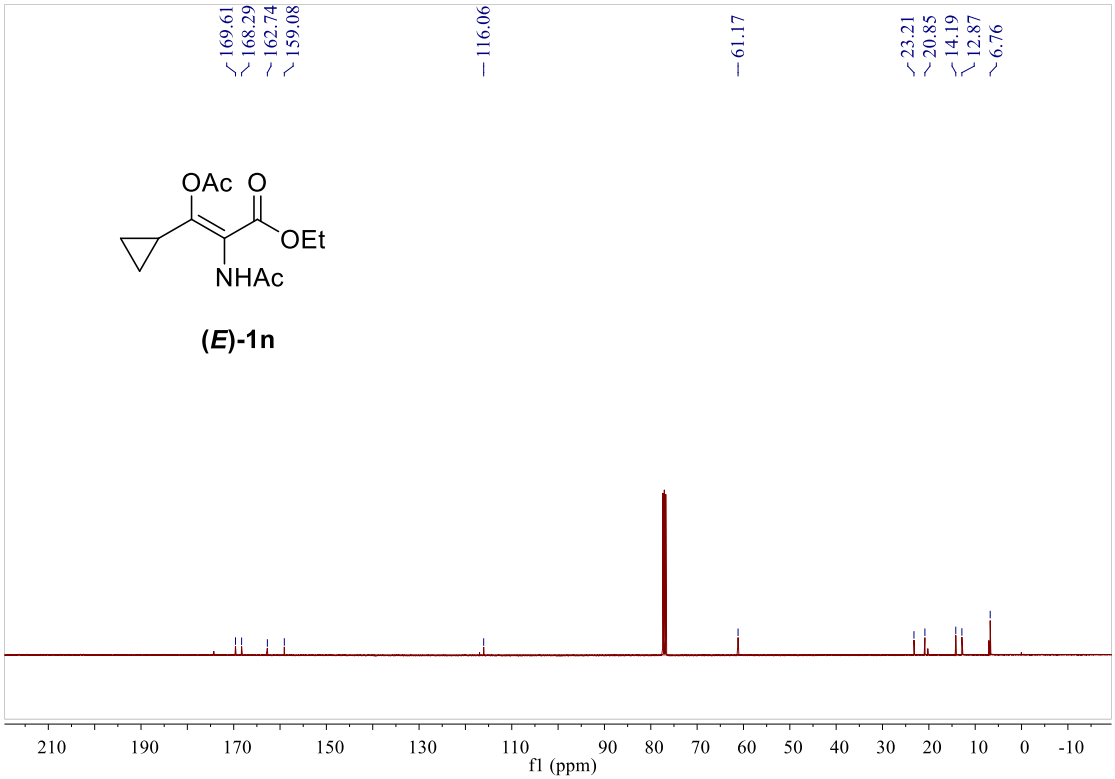
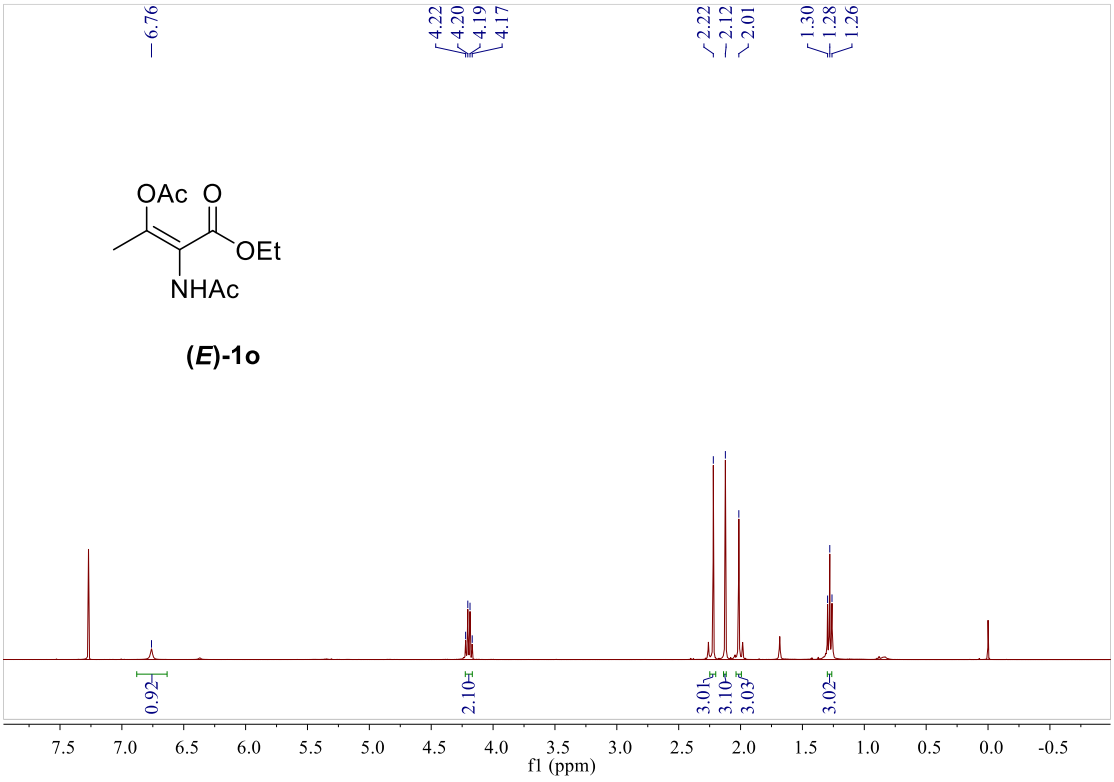
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


169.61
168.29
162.74
159.08
116.06
61.17
23.21
20.85
14.19
12.87
6.76
OAc
O
OEt
NHAc
(E)-1n
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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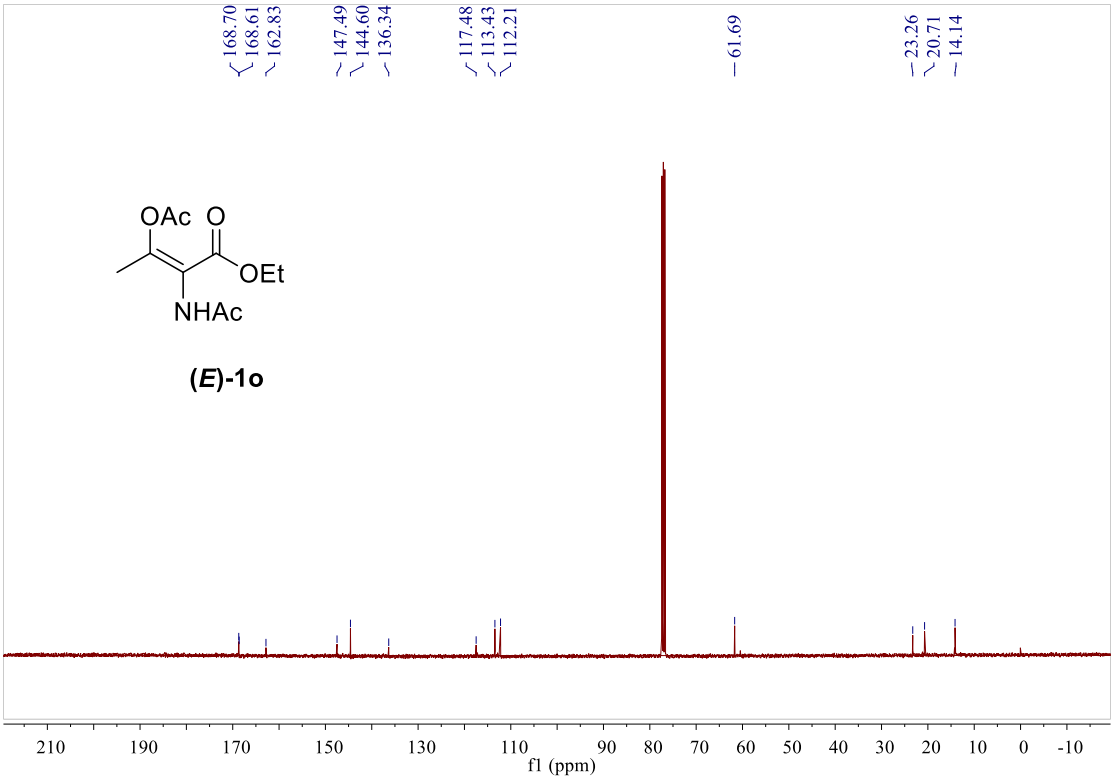
4.22
4.20
4.19
4.17
2.22
2.12
2.01
1.30
1.28
1.26
OAc
O
OEt
NHAc
(E)-1o
0.92
2.10
3.01
3.10
3.03
3.02
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)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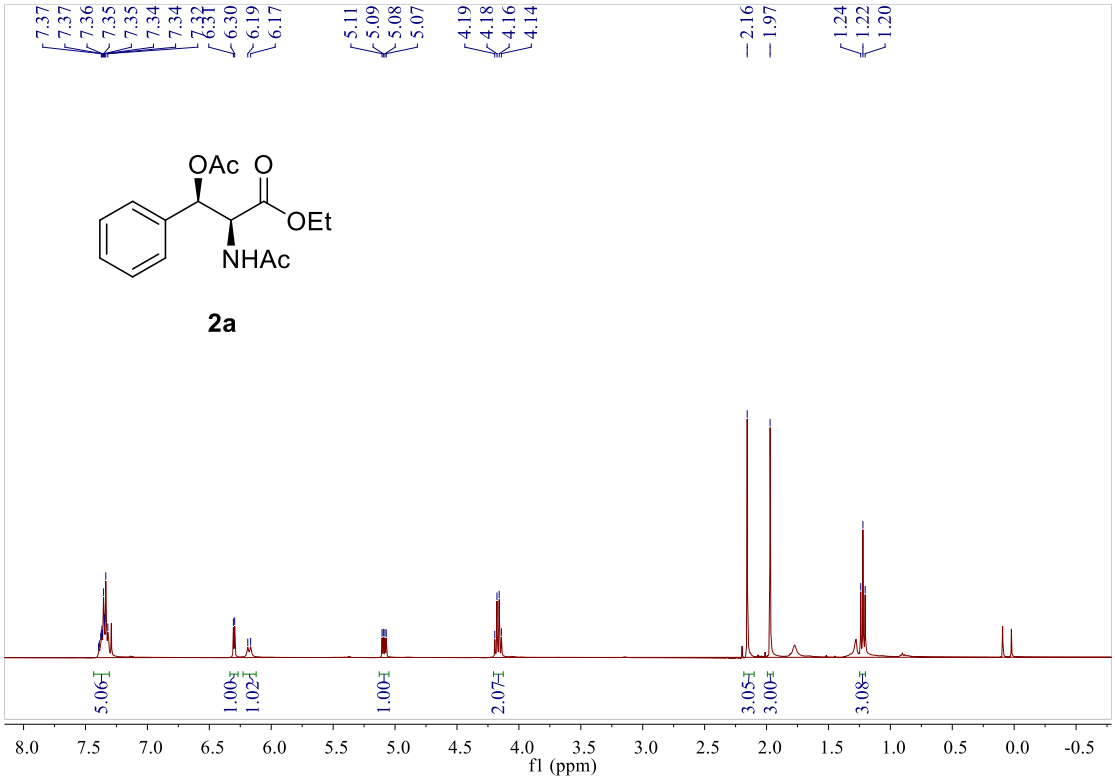
168.61
162.83
147.49
144.60
136.34
117.48
113.43
112.21
61.69
23.26
20.71
14.14
OAc
O
OEt
NHAc
(E)-1o
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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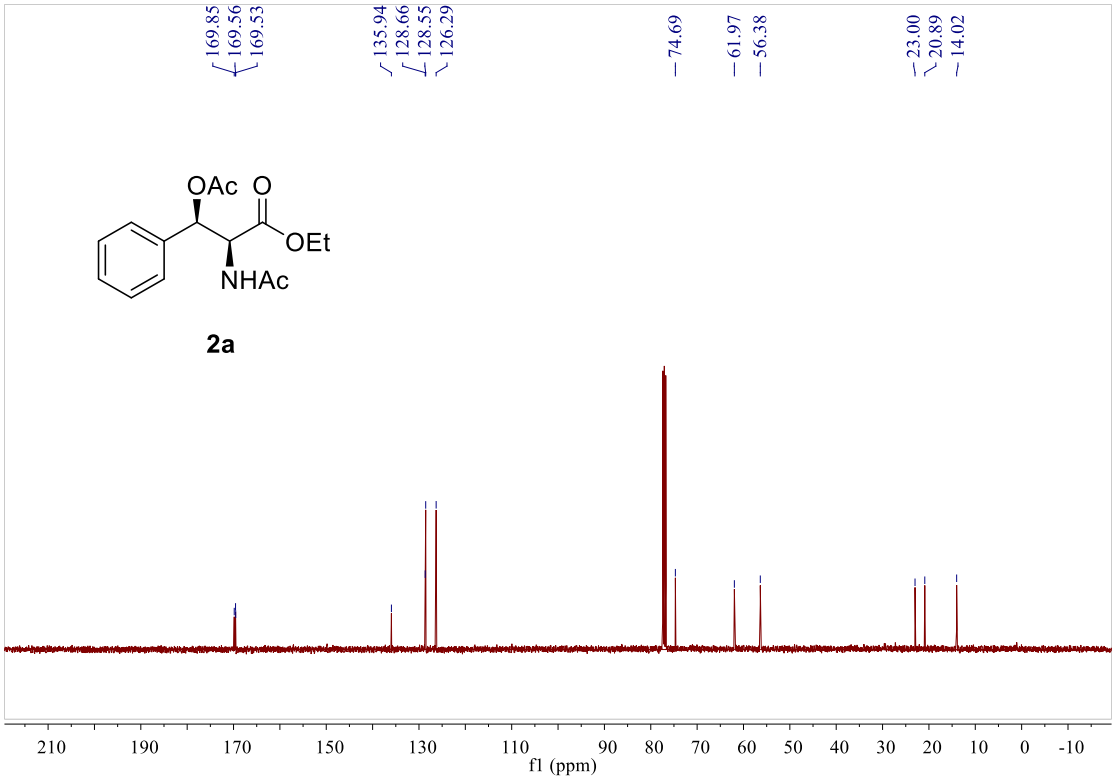
7.34
7.32
7.31
6.30
6.19
6.17
5.11
5.09
5.08
5.07
4.19
4.18
4.16
4.14
2.16
1.97
1.24
1.22
1.20
OAc O
OEt
NHAc
2a
5.06
1.00
1.02
1.00
2.07
3.05
3.00
3.08
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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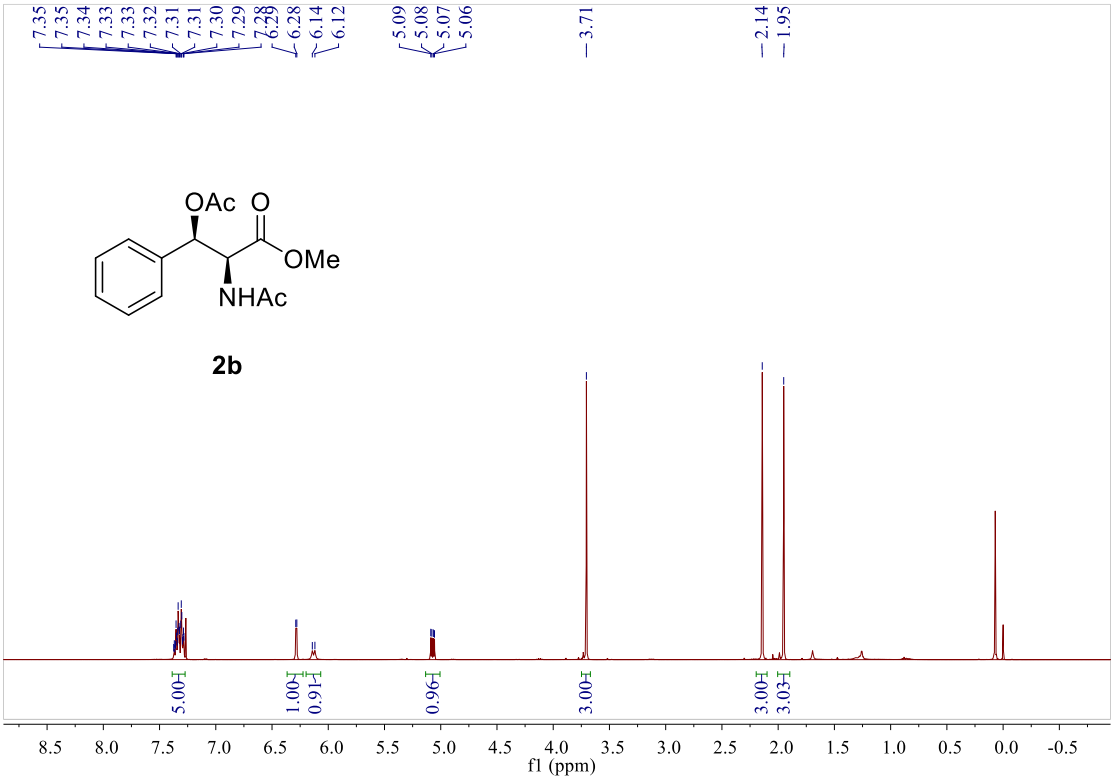
169.56
169.53
135.94
128.66
128.55
126.29
74.69
61.97
56.38
23.00
20.89
14.02
OAc O
OEt
NHAc
2a
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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7.35
7.34
7.33
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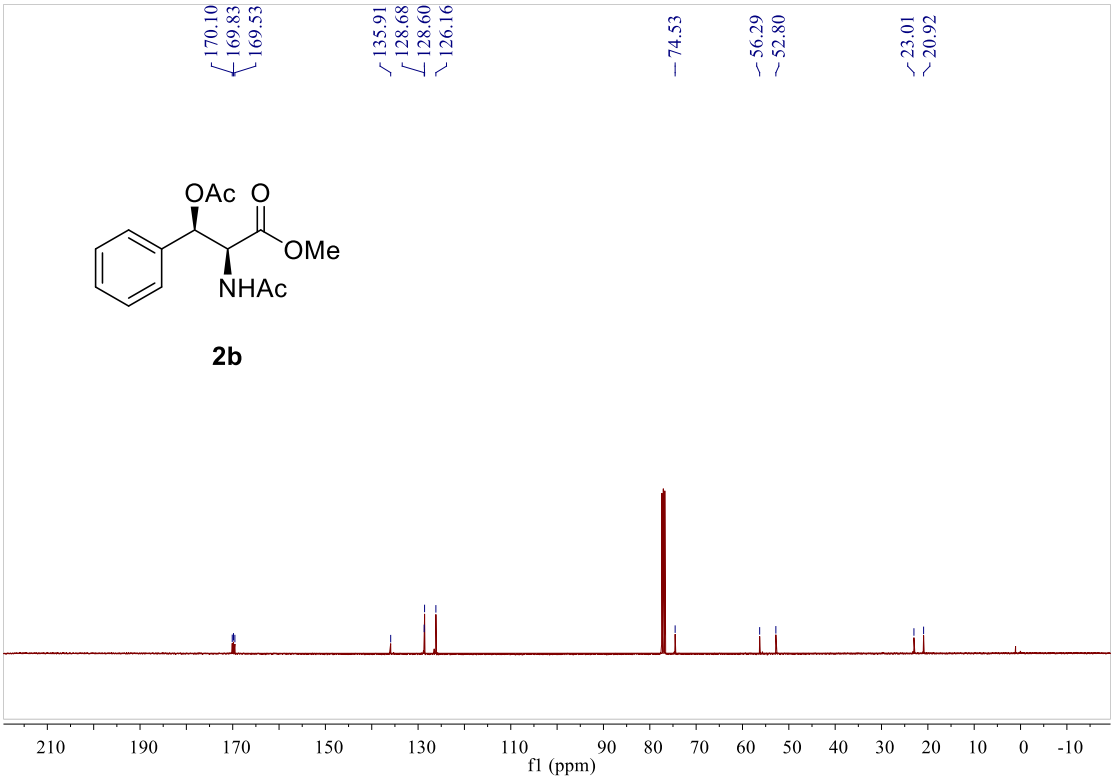
7.32
7.31
7.31
7.30
7.29
7.28
6.29
6.28
6.14
6.12
5.09
5.08
5.07
5.06
3.71
2.14
1.95
OAc O
OMe
NHAc
2b
5.00
1.00
0.91
0.96
3.00
3.00
3.03
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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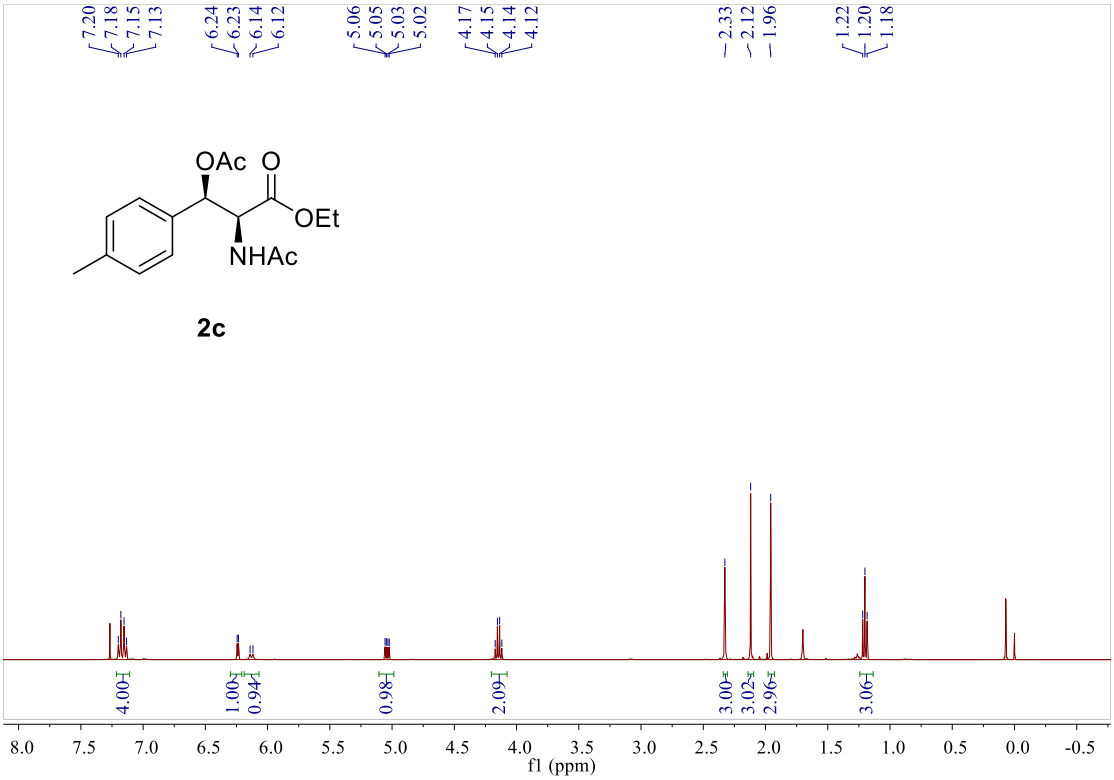
169.83
169.53
135.91
128.68
128.60
126.16
74.53
56.29
52.80
23.01
20.92
OAc O
OMe
NHAc
2b
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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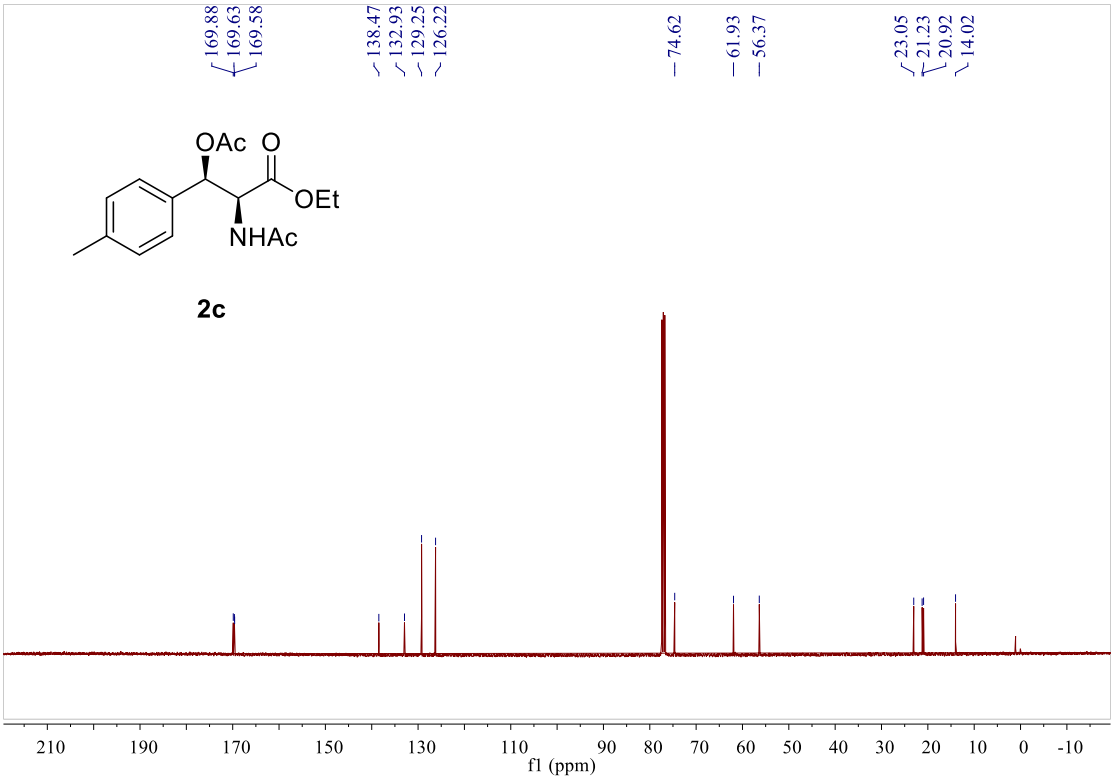
7.18
7.15
7.13
6.24
6.23
6.14
6.12
5.06
5.05
5.03
5.02
4.17
4.15
4.14
4.12
2.33
2.12
1.96
1.22
1.20
1.18
OAc O
OEt
NHAc
2c
4.00
1.00
0.94
0.98
2.09
3.00
3.02
2.96
3.06
f1 (ppm)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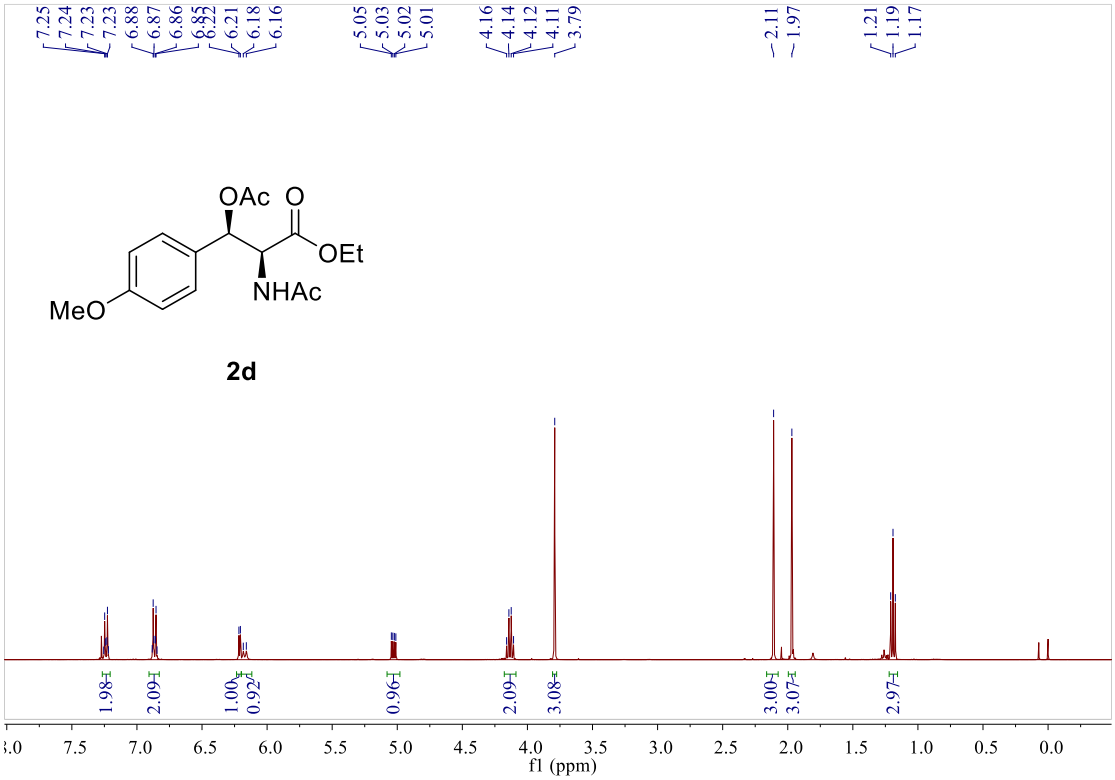
23.05
21.23
20.92
14.02
OAc O
OEt
NHAc
2c
f1 (ppm)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7.24
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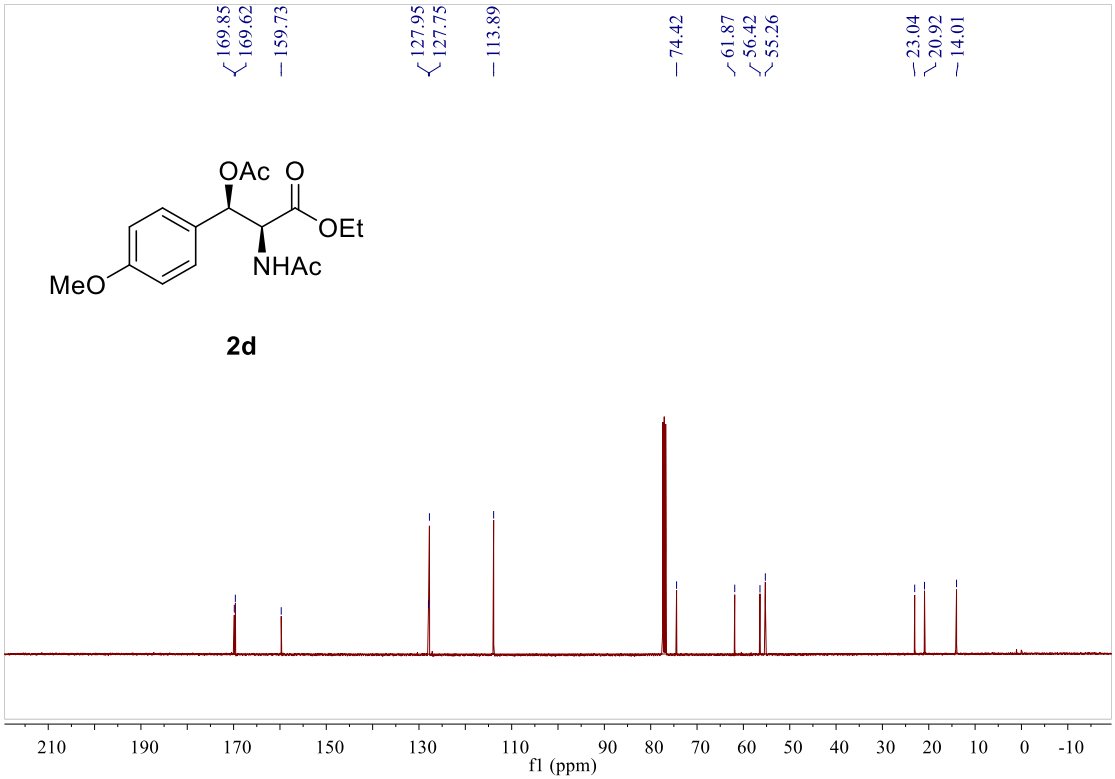
6.85
6.22
6.21
6.18
6.16
5.05
5.03
5.02
5.01
4.16
4.14
4.12
4.11
3.79
2.11
1.97
1.21
1.19
1.17
OAc
O
OEt
NHAc
MeO
2d
1.98
2.09
1.00
0.92
0.96
2.09
3.08
3.00
3.07
2.97
f1 (ppm)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113.89
74.42
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23.04
20.92
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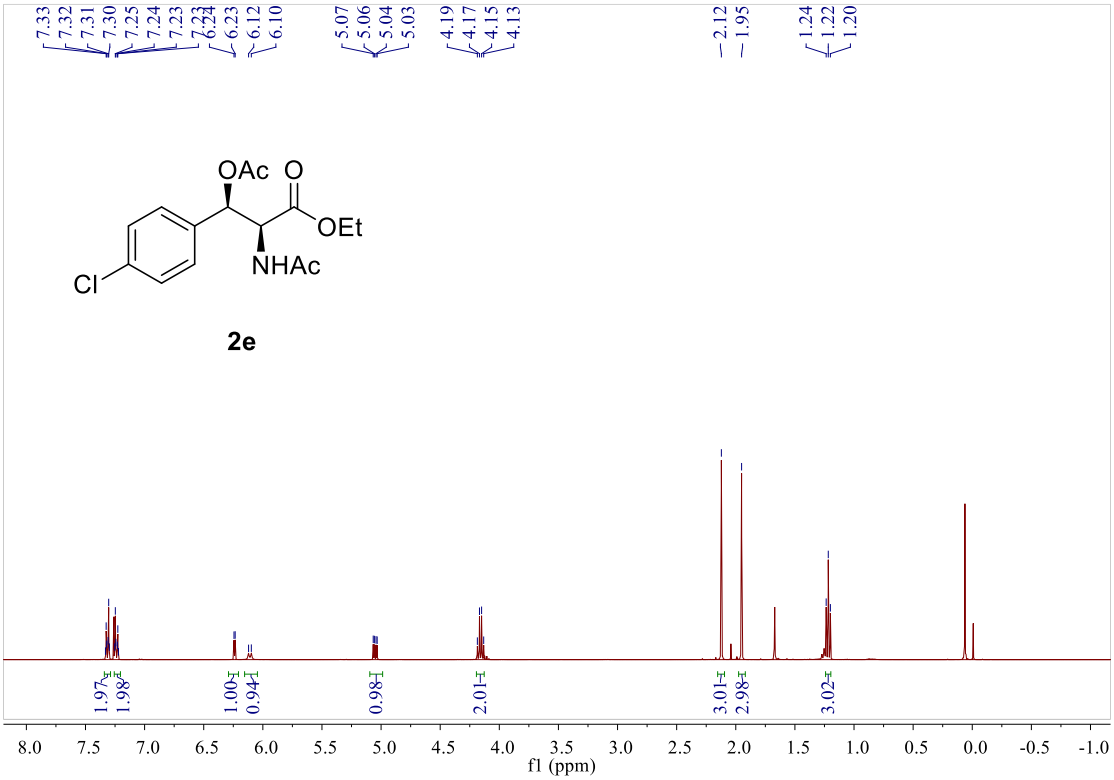
O
OEt
NHAc
MeO
2d
f1 (ppm)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7.23
6.24
6.23
6.12
6.10
5.07
5.06
5.04
5.03
4.19
4.17
4.15
4.13
2.12
1.95
1.24
1.22
1.20
OAc
O
OEt
NHAc
Cl
2e
1.97
1.98
1.00
0.94
0.98
2.01
3.01
2.98
3.02
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
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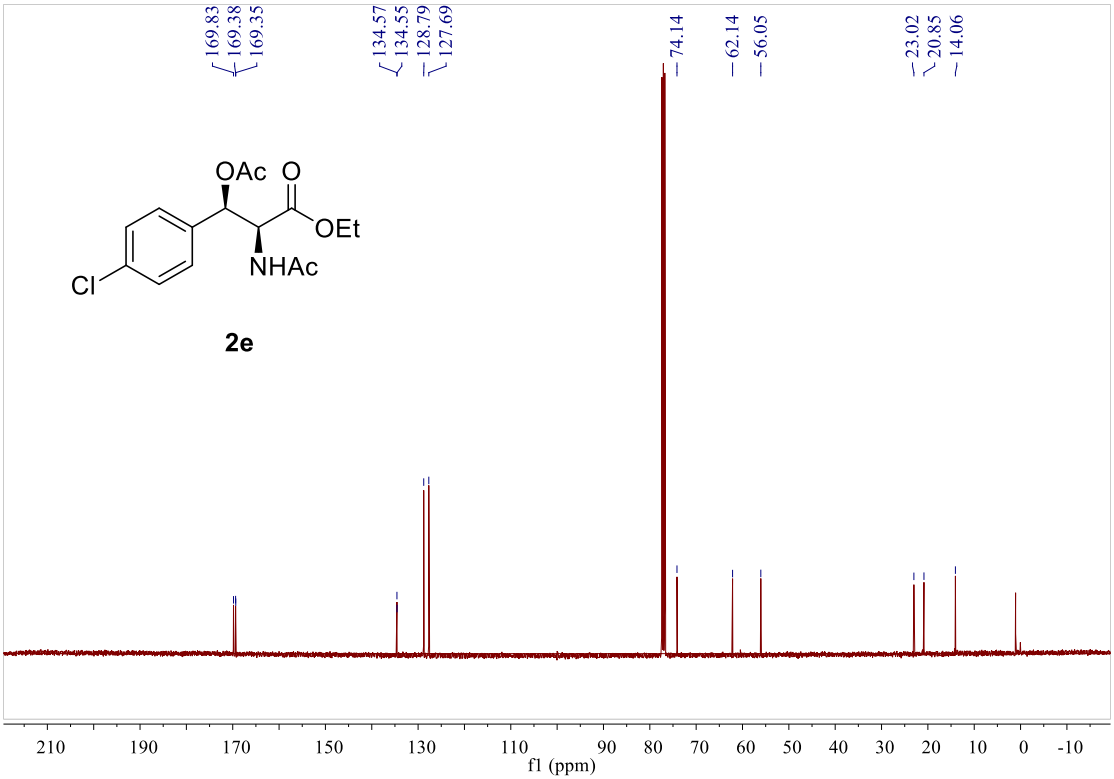
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)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134.57
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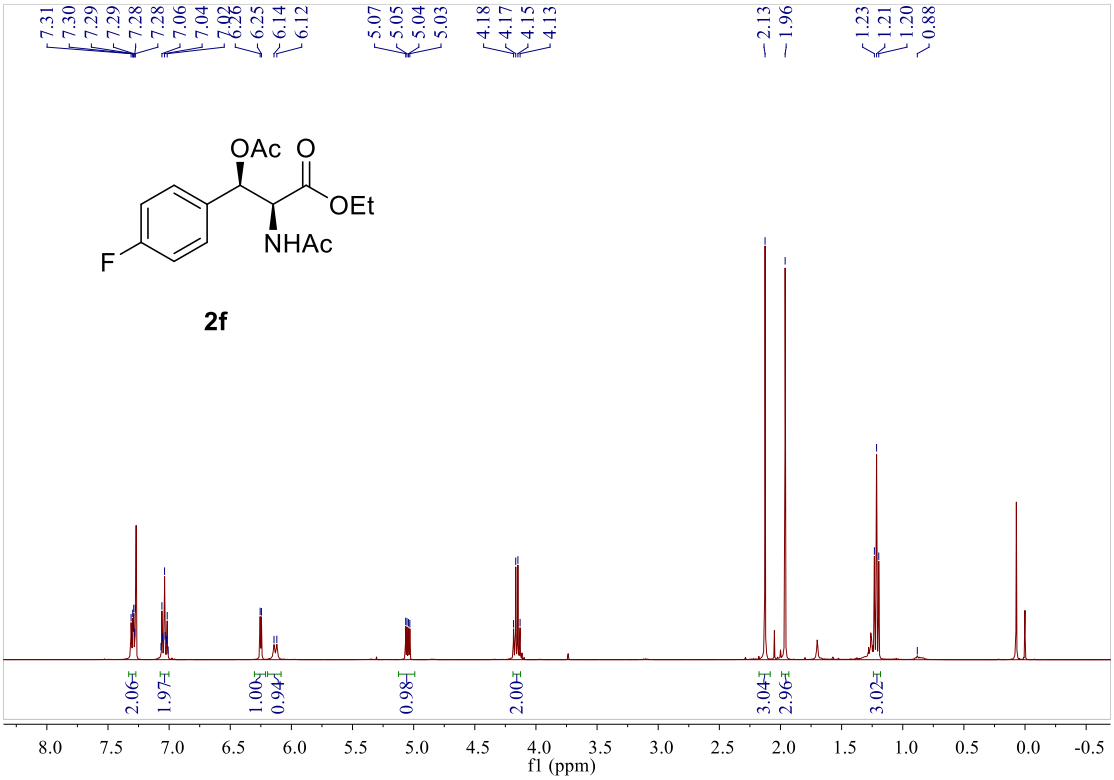
128.79
127.69
74.14
62.14
56.05
23.02
20.85
14.06
OAc
O
OEt
NHAc
Cl
2e
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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7.30
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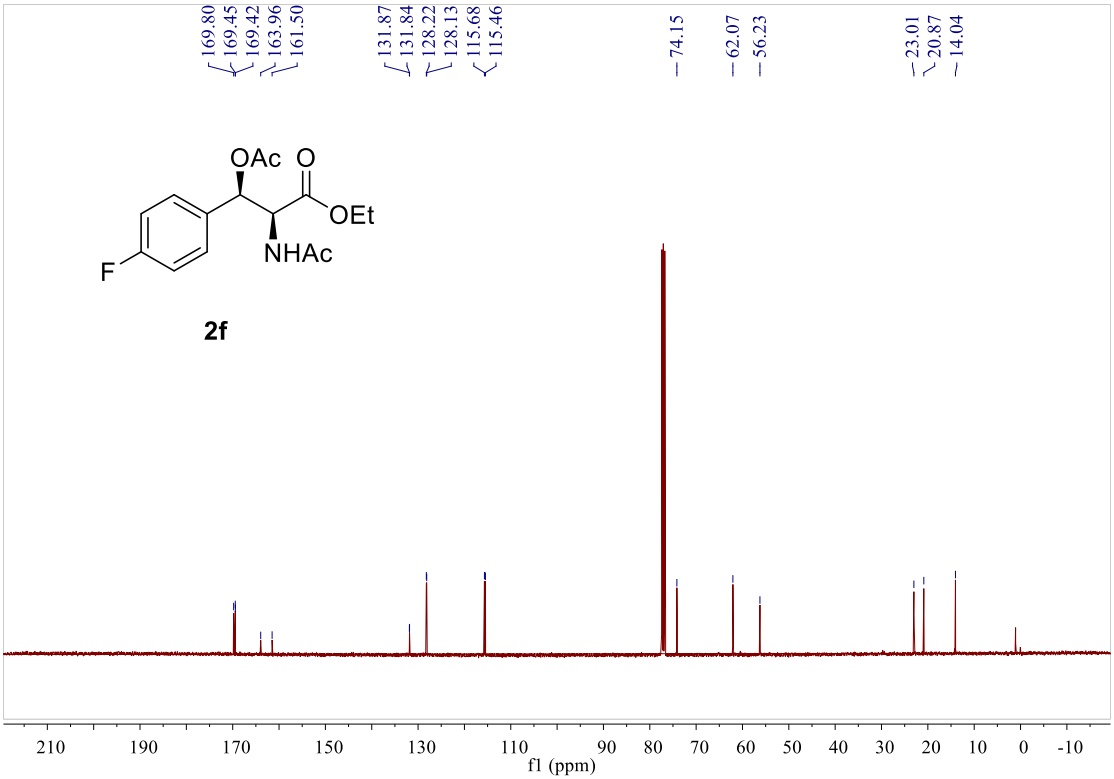
7.28
7.06
7.04
7.02
6.26
6.25
6.14
6.12
5.07
5.05
5.04
5.03
4.18
4.17
4.15
4.13
2.13
1.96
1.23
1.21
1.20
0.88
OAc O
OEt
NHAc
F
2f
2.06
1.97
1.00
0.94
0.98
2.00
3.04
2.96
3.02
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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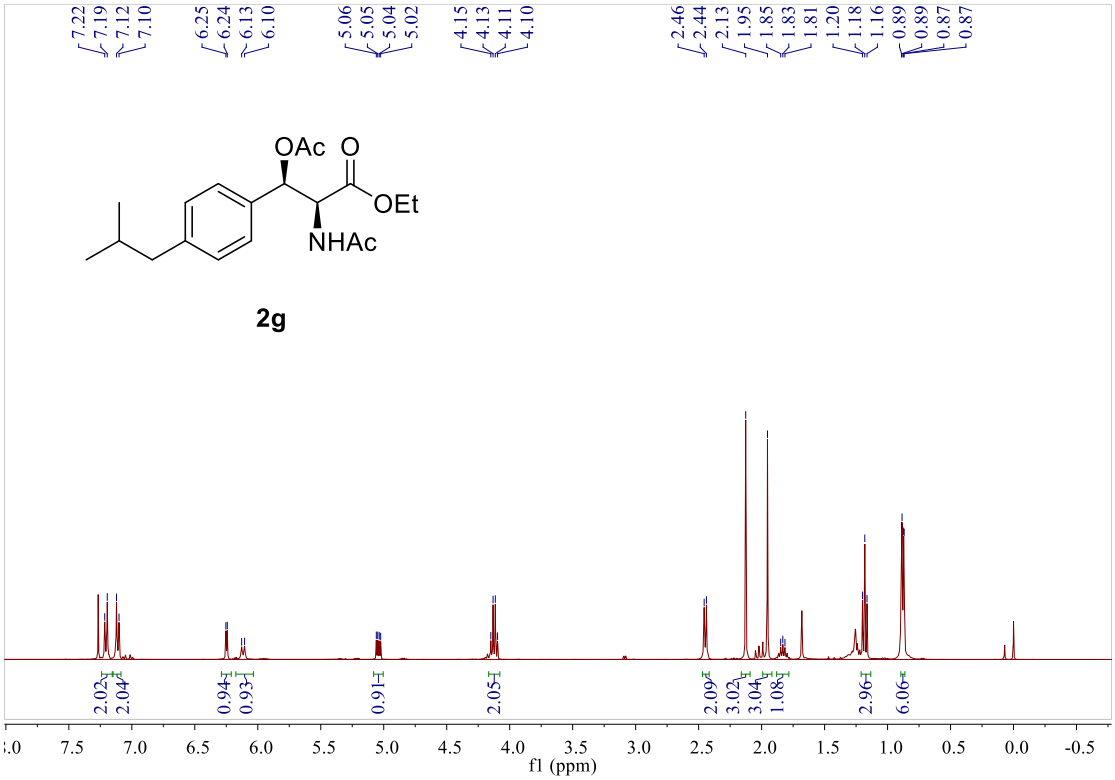
128.22
128.13
115.68
115.46
74.15
62.07
56.23
23.01
20.87
14.04
OAc O
OEt
NHAc
F
2f
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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7.10
6.25
6.24
6.13
6.10
5.06
5.05
5.04
5.02
4.15
4.13
4.11
4.10
2.46
2.44
2.13
1.95
1.85
1.83
1.81
1.20
1.18
1.16
0.89
0.89
0.87
0.87
OAc
O
OEt
NHAc
2g
2.02
2.04
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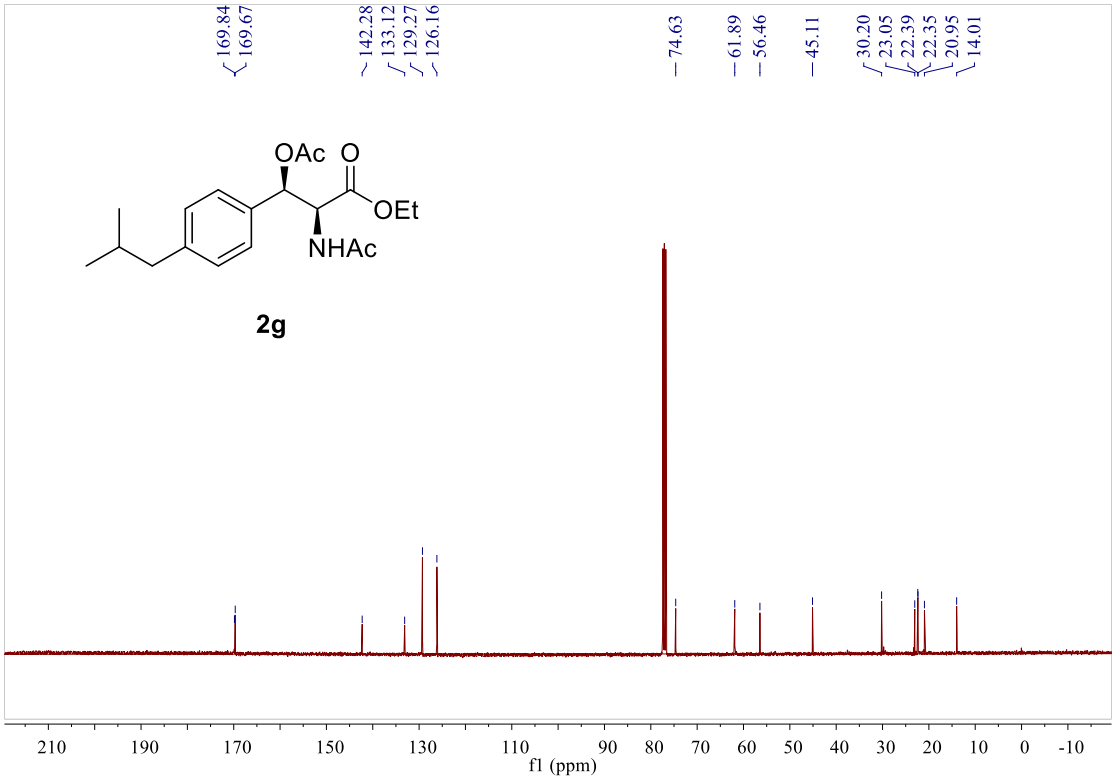
0.93
0.91
2.05
2.09
3.02
3.04
1.08
2.96
6.06
f1 (ppm)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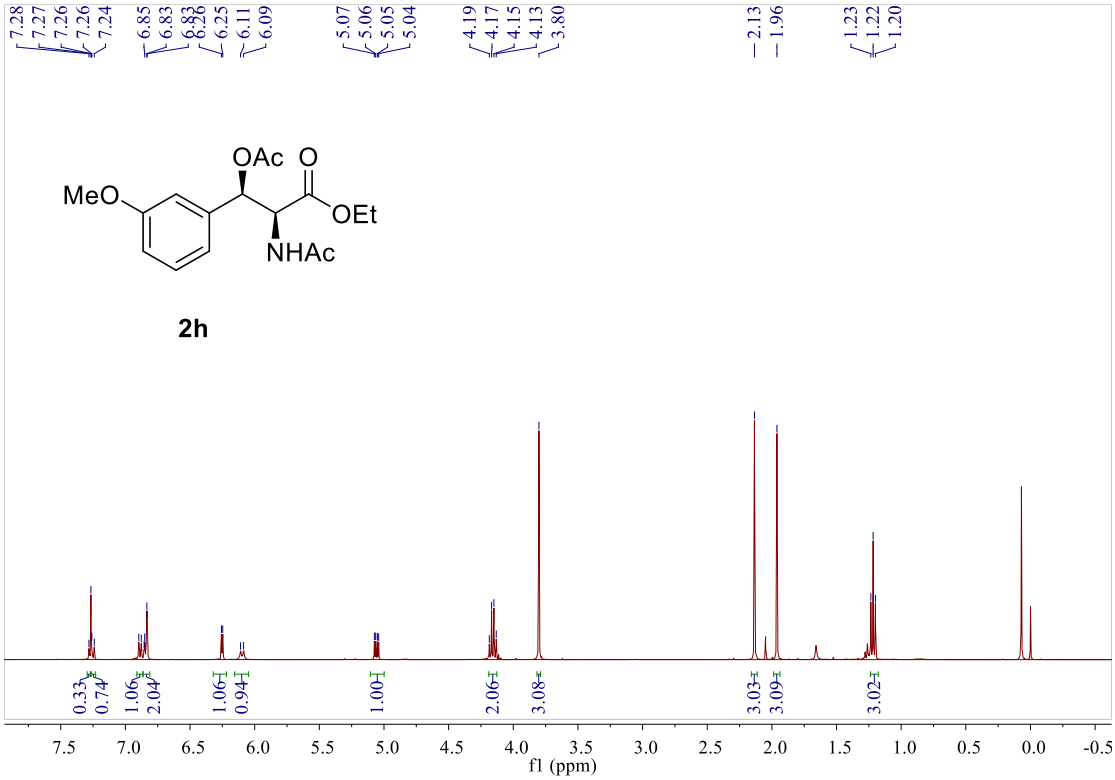
126.16
74.63
61.89
56.46
45.11
30.20
23.05
22.39
22.35
20.95
14.01
OAc
O
OEt
NHAc
2g
f1 (ppm)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6.85
6.83
6.83
6.26
6.25
6.11
6.09
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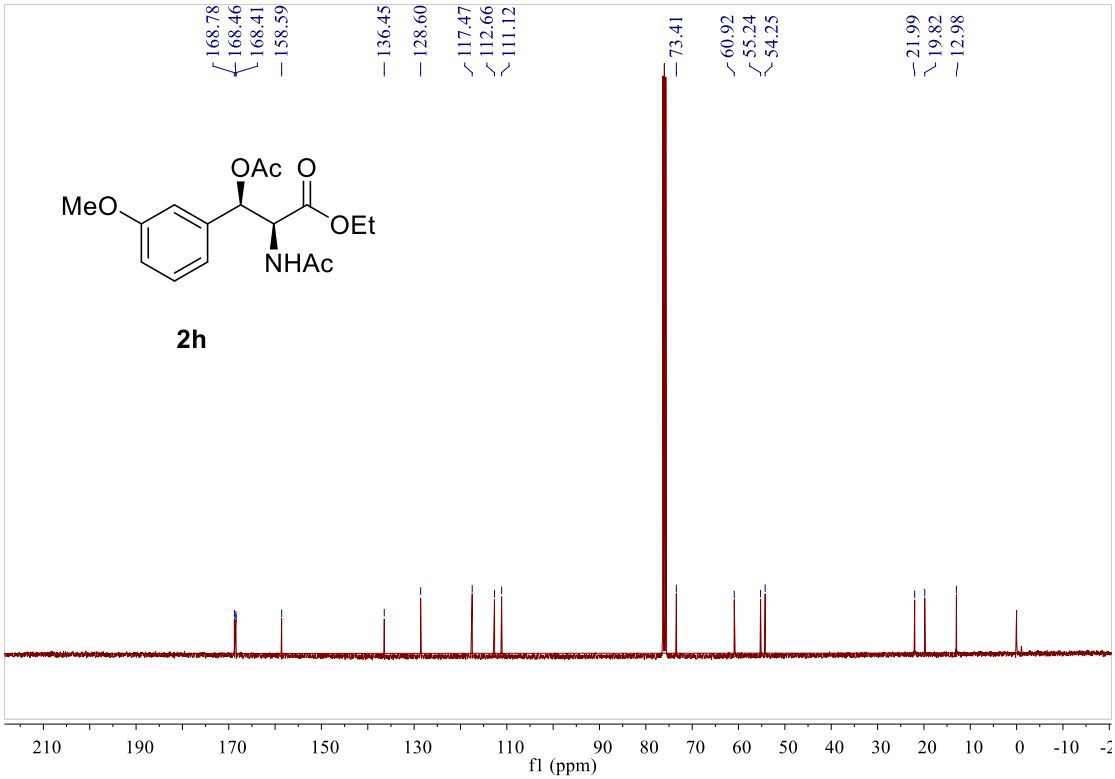
5.06
5.05
5.04
4.19
4.17
4.15
4.13
3.80
2.13
1.96
1.23
1.22
1.20
OAc O
MeO
OEt
NHAc
2h
0.33
0.74
1.06
2.04
1.06
0.94
1.00
2.06
3.08
3.03
3.09
3.02
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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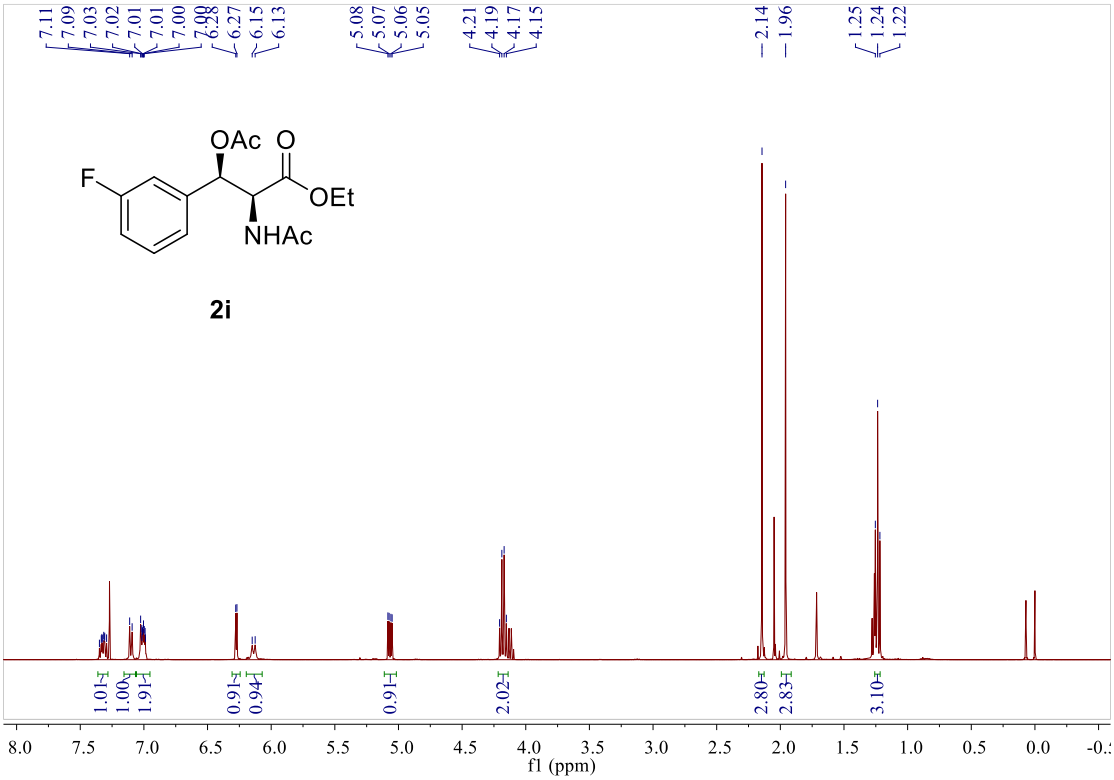
128.60
117.47
112.66
111.12
73.41
60.92
55.24
54.25
21.99
19.82
12.98
OAc O
MeO
OEt
NHAc
2h
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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7.02
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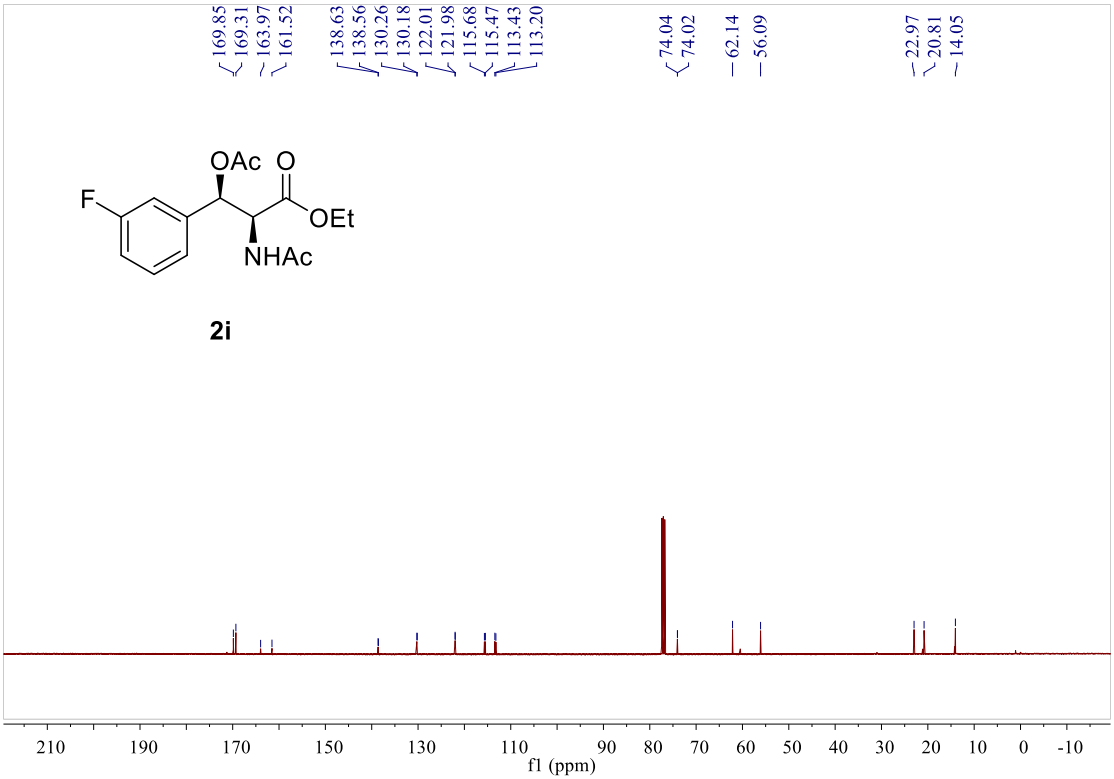
7.01
7.00
6.28
6.27
6.15
6.13
5.08
5.07
5.06
5.05
4.21
4.19
4.17
4.15
2.14
1.96
1.25
1.24
1.22
OAc O
F
OEt
NHAc
1.01
1.00
1.91
0.91
0.94
0.91
2.02
2.80
2.83
3.10
f1 (ppm)

2i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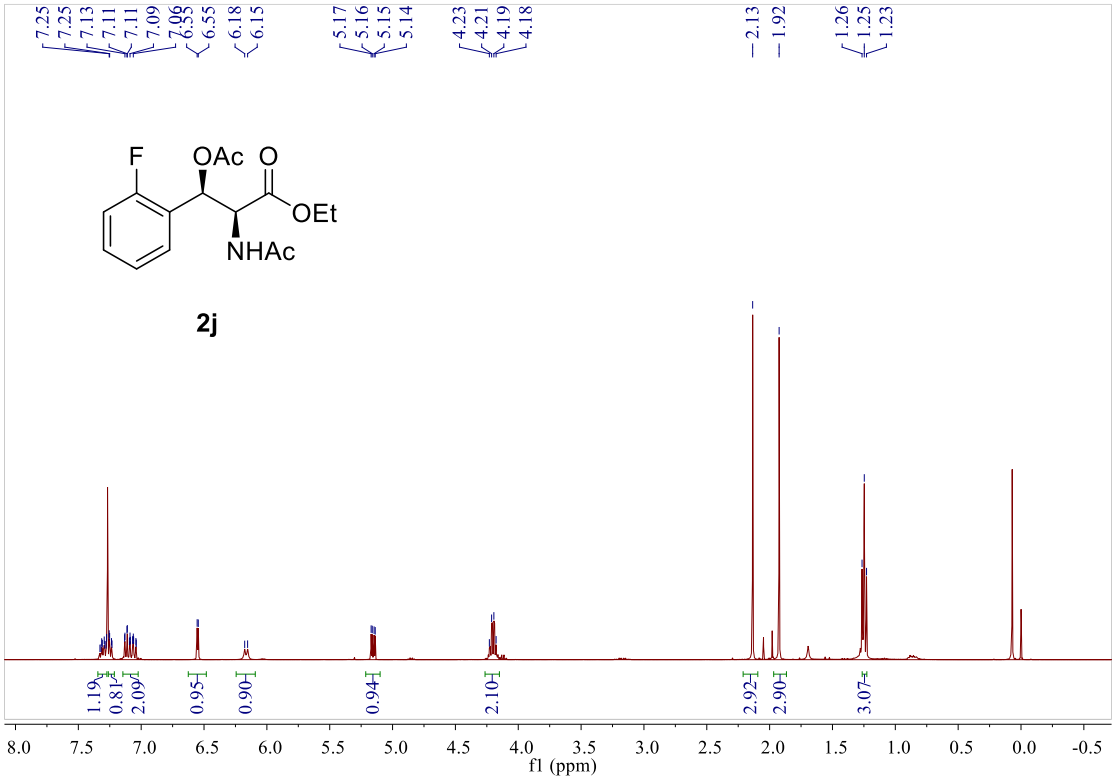
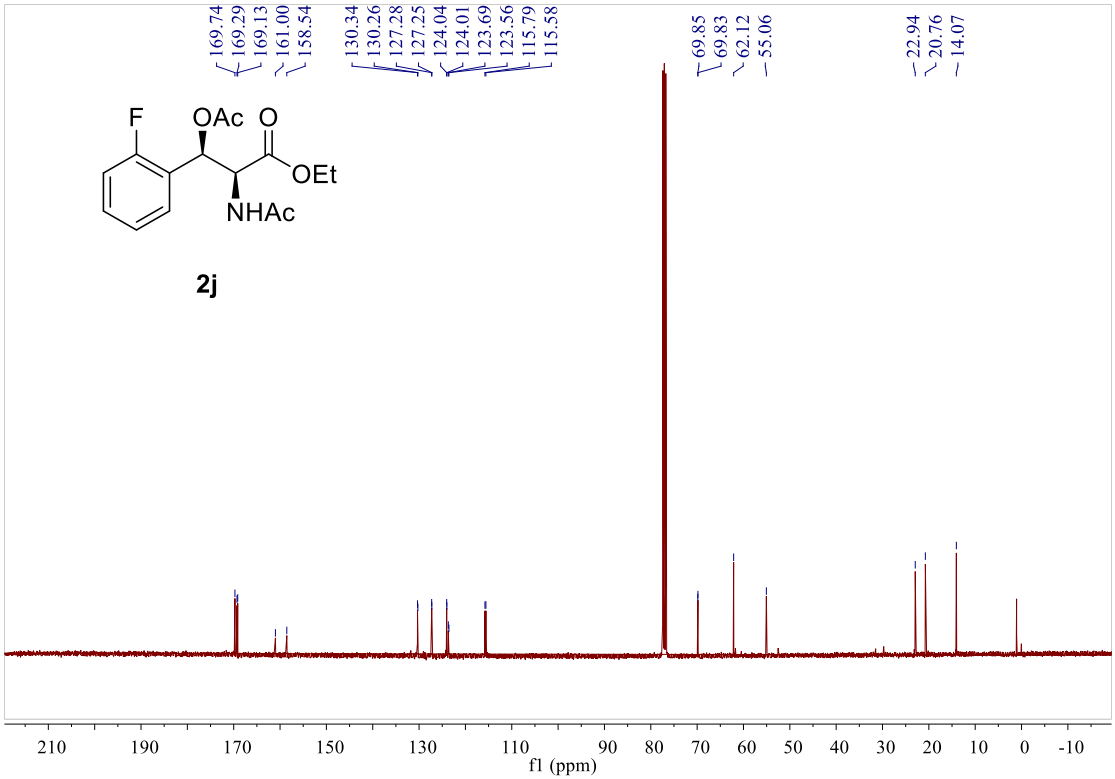
169.31
163.97
161.52
138.63
138.56
130.26
130.18
122.01
121.98
115.68
115.47
113.43
113.20
74.04
74.02
62.14
56.09
22.97
20.81
14.05
OAc O
F
OEt
NHAc
f1 (ppm)

2i


7.25
7.25
7.13
7.11
7.11
7.09
7.06
6.55
6.55
6.18
6.15
5.17
5.16
5.15
5.14
4.23
4.21
4.19
4.18
2.13
1.92
1.26
1.25
1.23
F
OAc
O
OEt
NHAc
2j
1.19
0.81
2.09
0.95
0.90
0.94
2.10
2.92
2.90
3.07
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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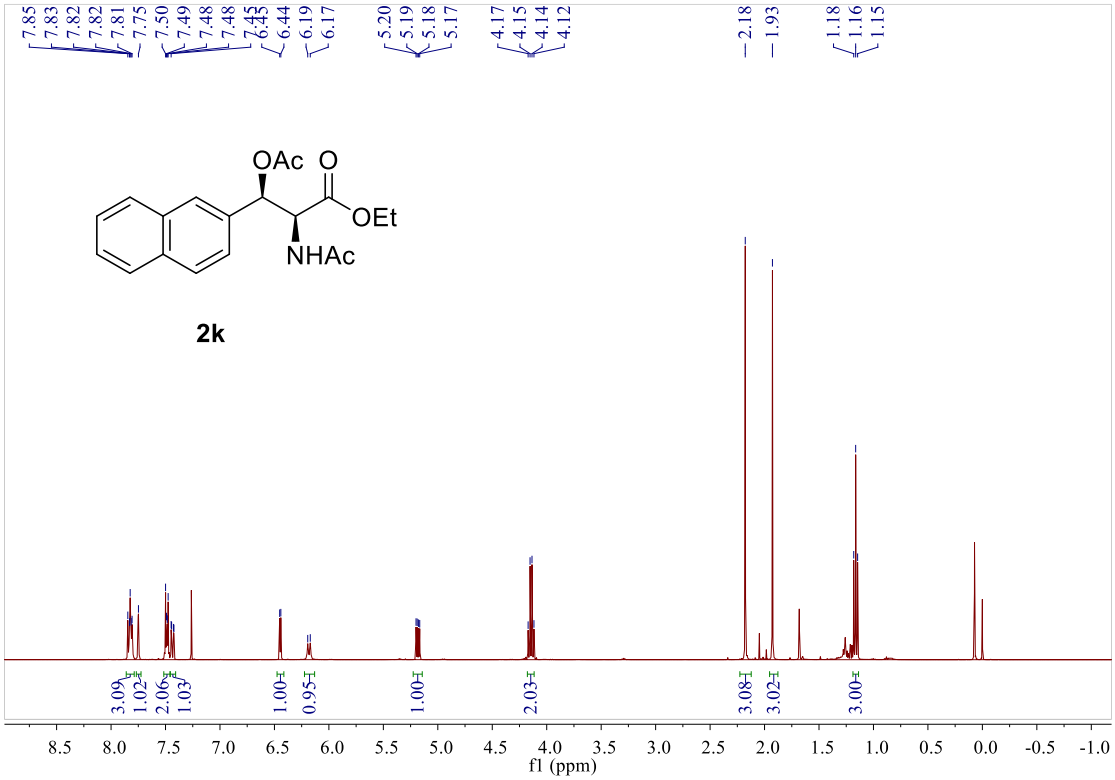
169.29
169.13
161.00
158.54
130.34
130.26
127.28
127.25
124.04
124.01
123.69
123.56
115.79
115.58
69.85
69.83
62.12
55.06
22.94
20.76
14.07
F
OAc
O
OEt
NHAc
2j
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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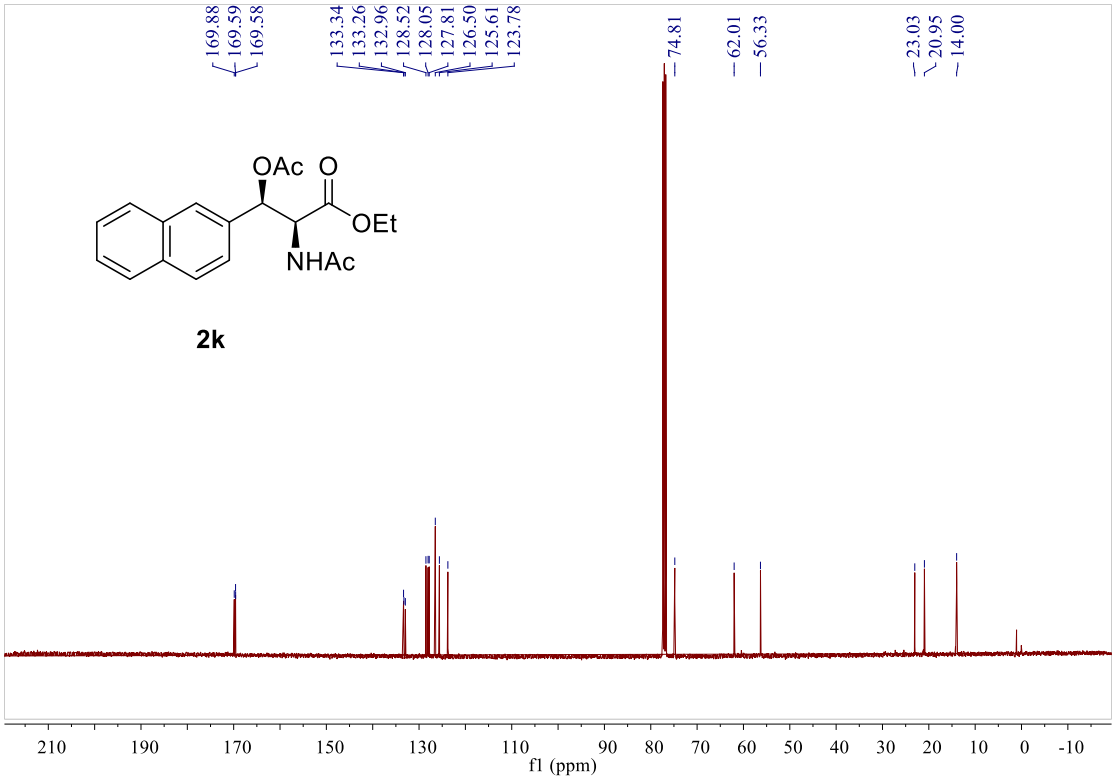
7.83
7.82
7.82
7.81
7.75
7.50
7.49
7.48
7.48
7.45
6.45
6.44
6.19
6.17
5.20
5.19
5.18
5.17
4.17
4.15
4.14
4.12
2.18
1.93
1.18
1.16
1.15
OAc O
OEt
NHAc
2k
3.09
1.02
2.06
1.03
1.00
0.95
1.00
2.03
3.08
3.02
3.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)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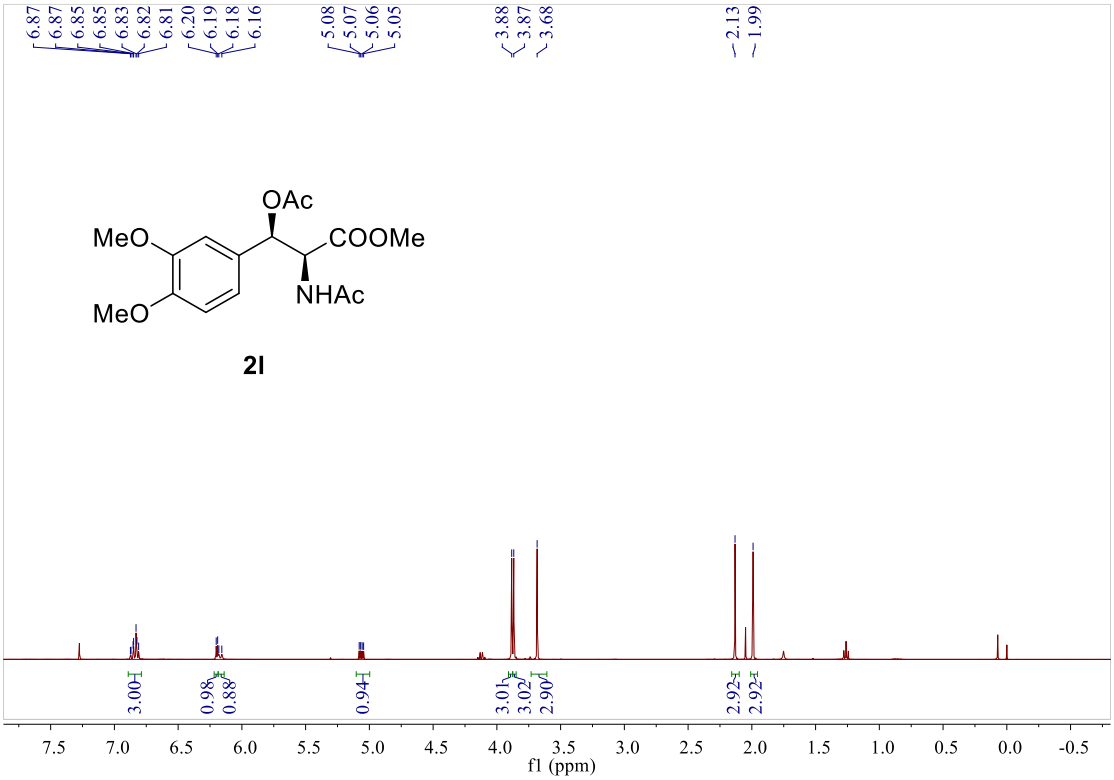
169.59
169.58
133.34
133.26
132.96
128.52
128.05
127.81
126.50
125.61
123.78
74.81
62.01
56.33
23.03
20.95
14.00
OAc O
OEt
NHAc
2k
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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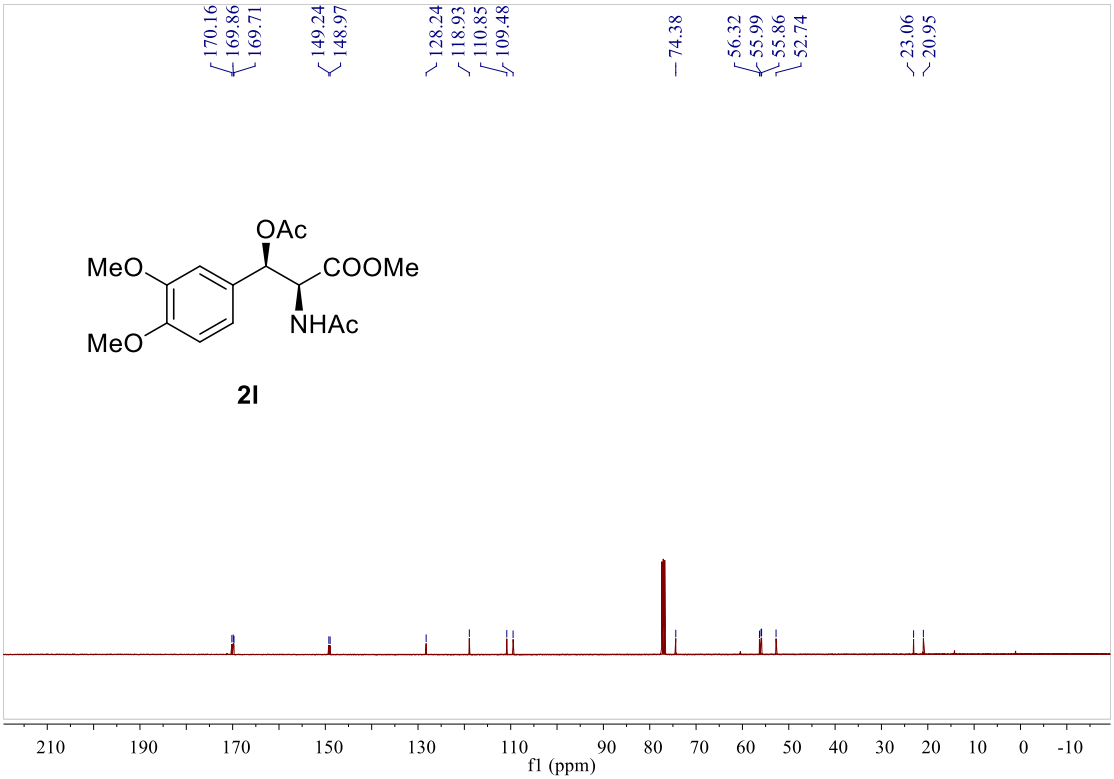
6.87
6.85
6.85
6.83
6.82
6.81
6.20
6.19
6.18
6.16
5.08
5.07
5.06
5.05
3.88
3.87
3.68
2.13
1.99
OAc
MeO
COOMe
NHAc
MeO
3.00
0.98
0.88
0.94
3.01
3.02
2.90
2.92
2.92
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

2l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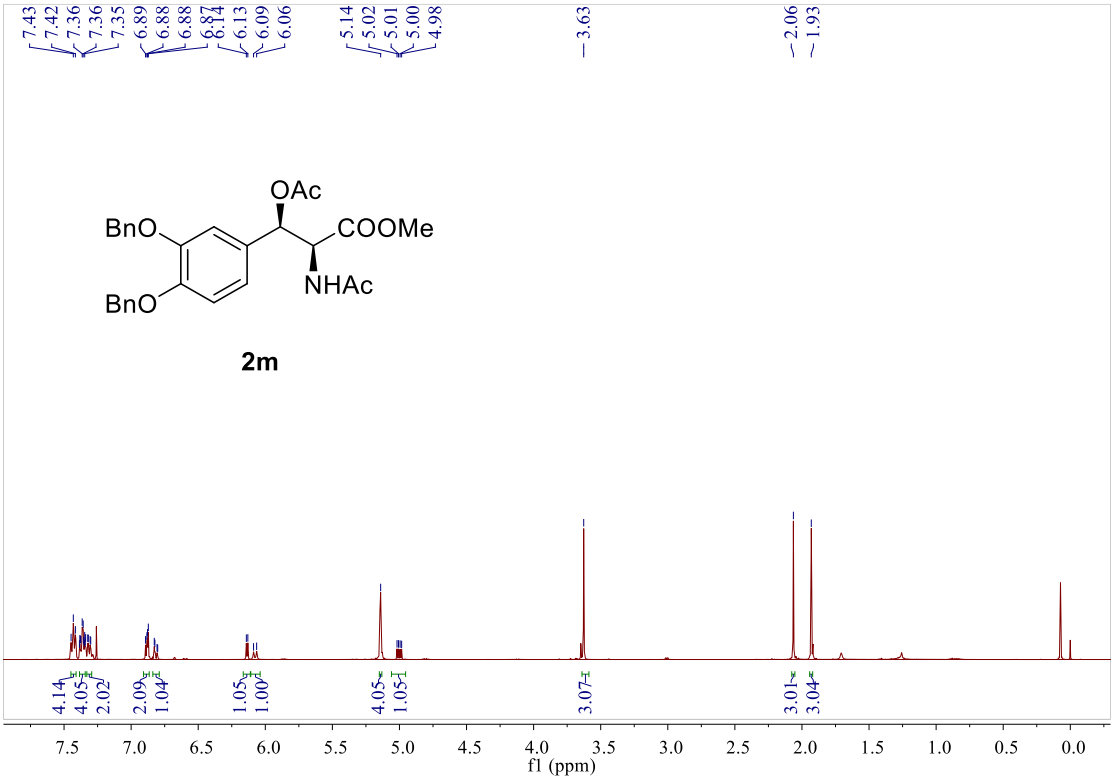
169.86
169.71
149.24
148.97
128.24
118.93
110.85
109.48
74.38
56.32
55.99
55.86
52.74
23.06
20.95
OAc
MeO
COOMe
NHAc
MeO
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

2l


7.43
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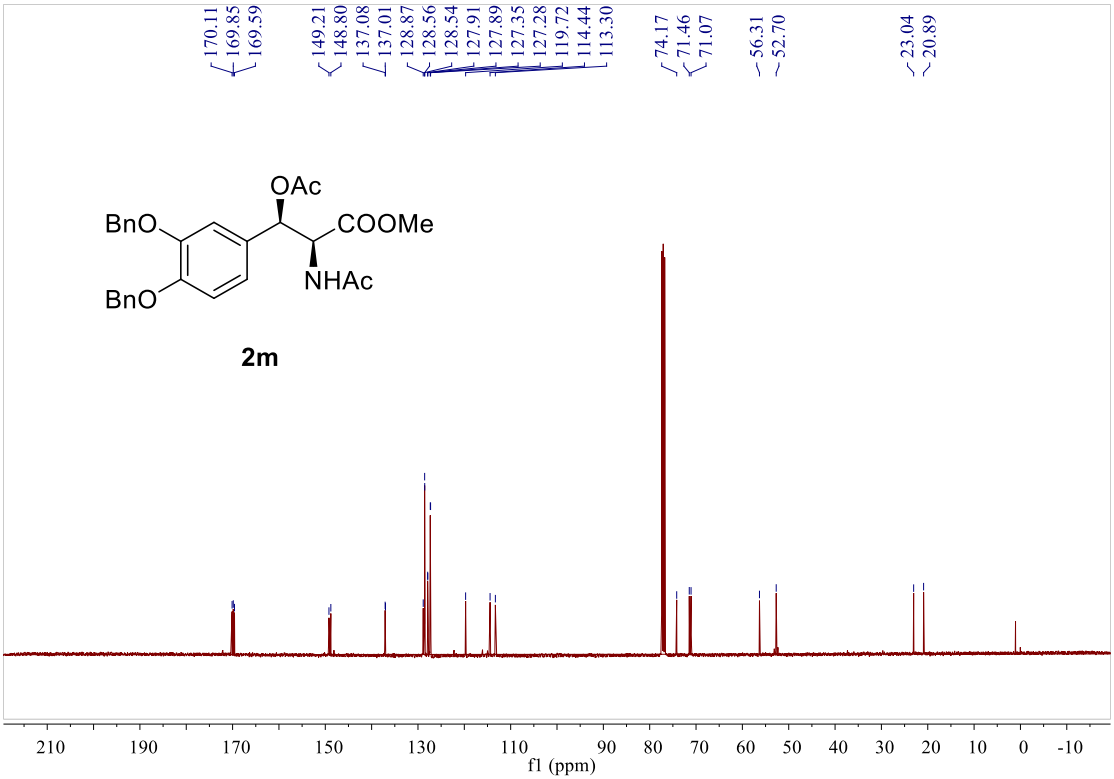
7.36
7.35
6.89
6.88
6.88
6.87
6.14
6.13
6.09
6.06
5.14
5.02
5.01
5.00
4.98
3.63
2.06
1.93
OAc
BnO
COOMe
NHAc
BnO
2m
4.14
4.05
2.02
2.09
1.04
1.05
1.00
4.05
1.05
3.07
3.01
3.04
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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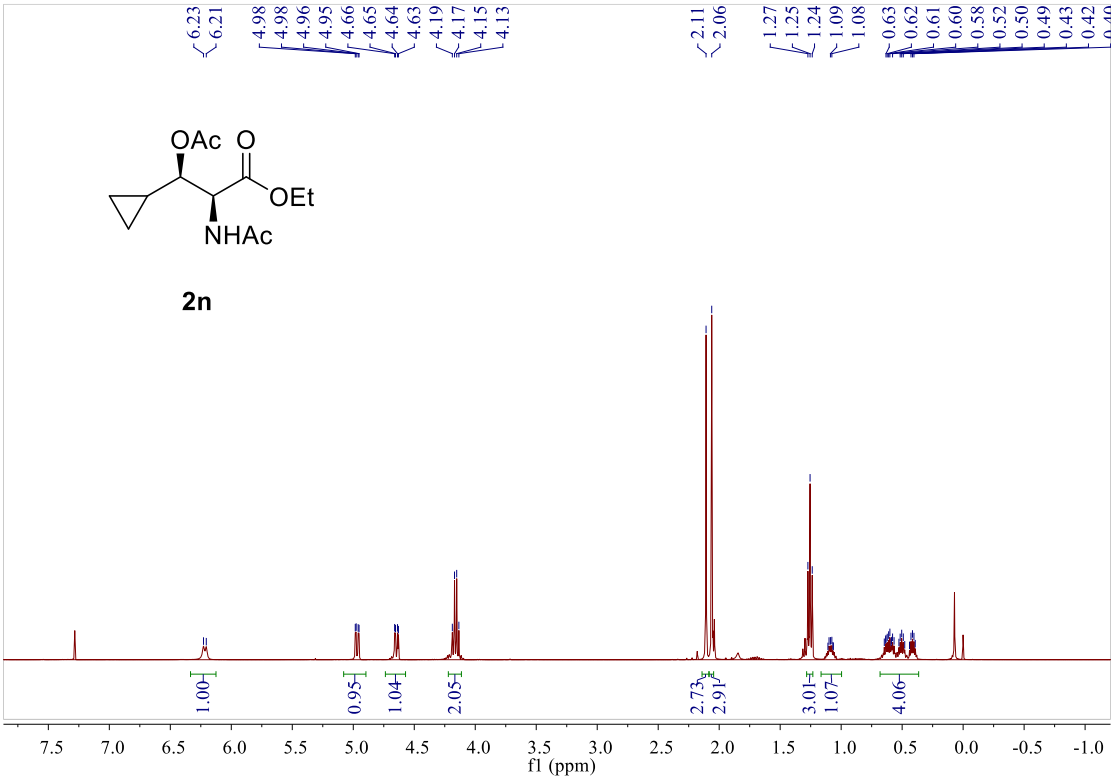
149.21
148.80
137.08
137.01
128.87
128.56
128.54
127.91
127.89
127.35
127.28
119.72
114.44
113.30
74.17
71.46
71.07
56.31
52.70
23.04
20.89
OAc
BnO
COOMe
NHAc
BnO
2m
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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4.96
4.95
4.66
4.65
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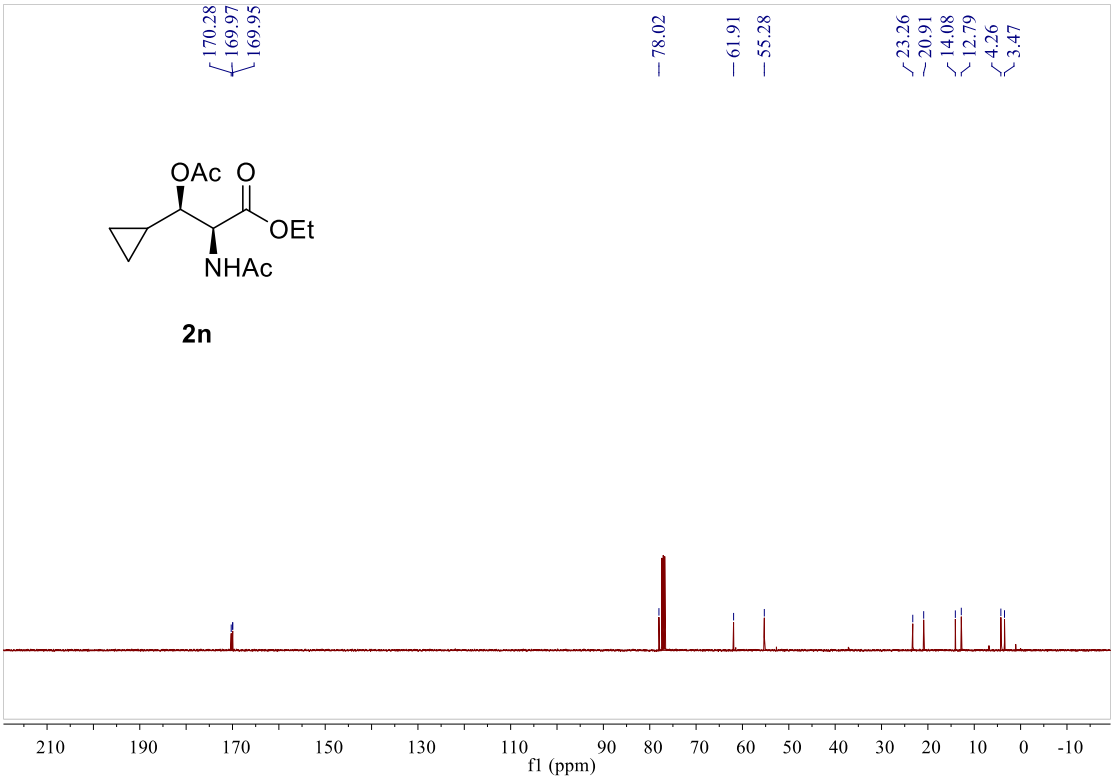
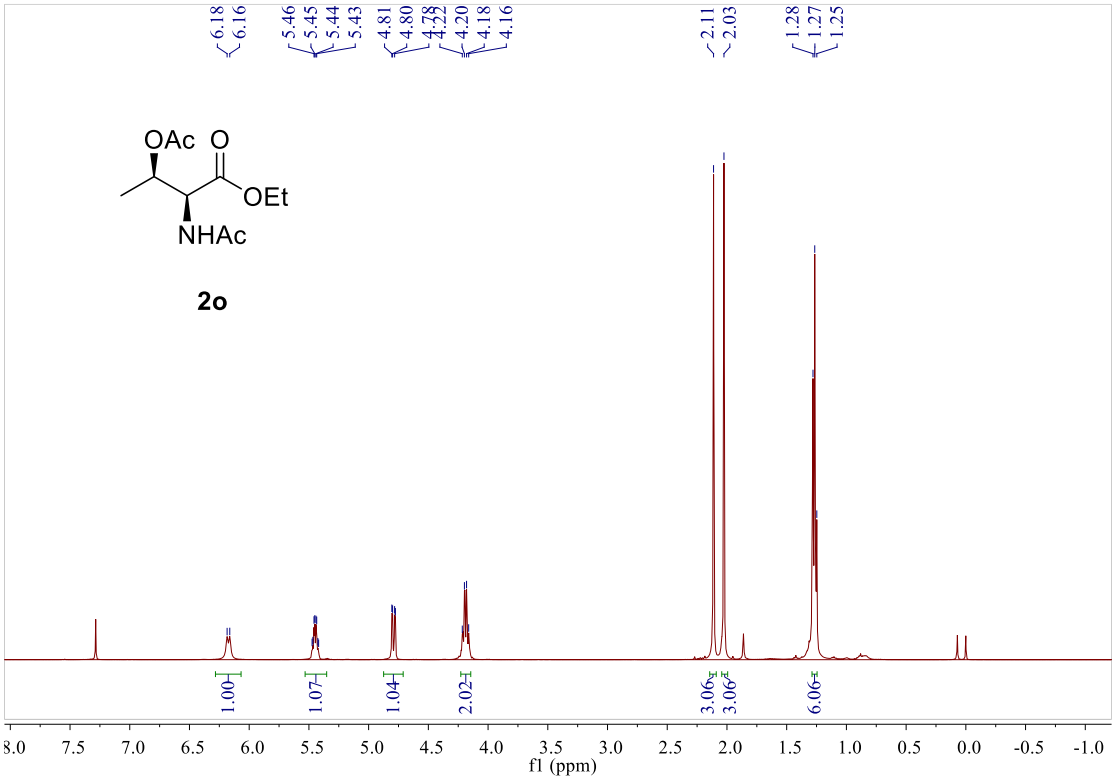
4.63
4.19
4.17
4.15
4.13
2.11
2.06
1.27
1.25
1.24
1.09
1.08
0.63
0.62
0.61
0.60
0.58
0.52
0.50
0.49
0.43
0.42
OAc O
OEt
NHAc
2n
1.00
0.95
1.04
2.05
2.73
2.91
3.01
1.07
4.06
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


170.28
169.97
169.95
78.02
61.91
55.28
23.26
20.91
14.08
12.79
4.26
3.47
OAc O
OEt
NHAc
2n
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


6.18
6.16
5.46
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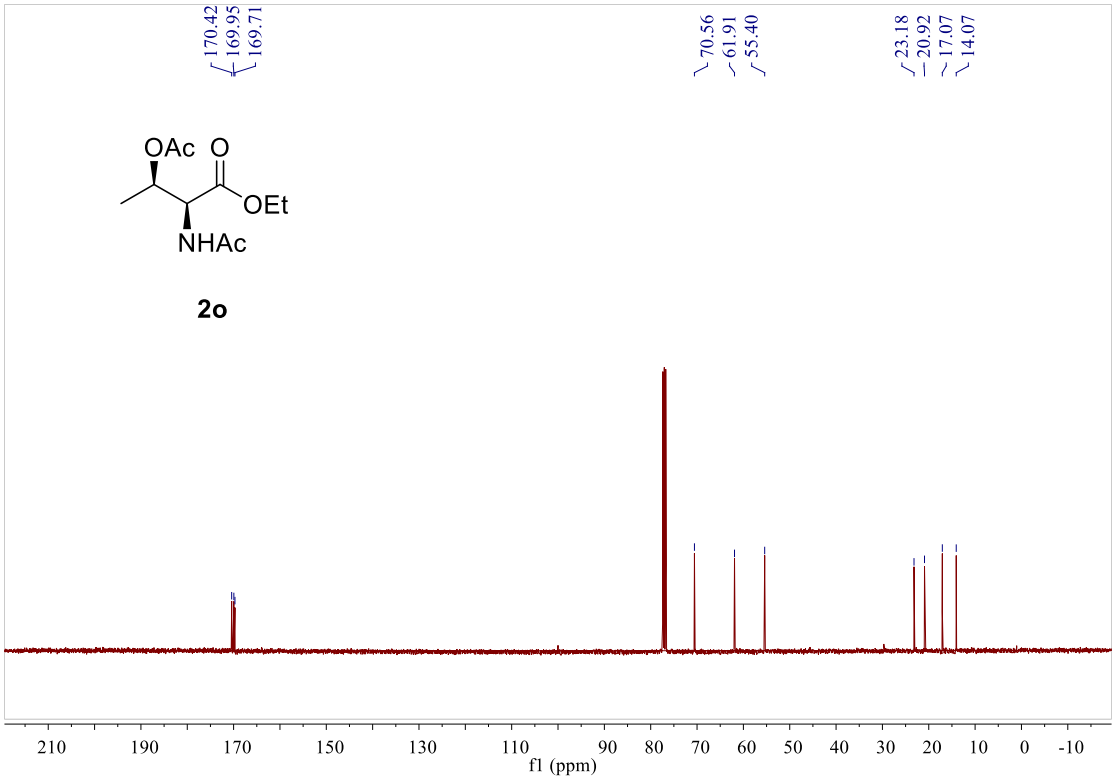
5.44
5.43
4.81
4.80
4.78
4.22
4.20
4.18
4.16
2.11
2.03
1.28
1.27
1.25
OAc O
OEt
NHAc
2o
1.00
1.07
1.04
2.02
3.06
3.06
6.06
f1 (ppm)


170.42
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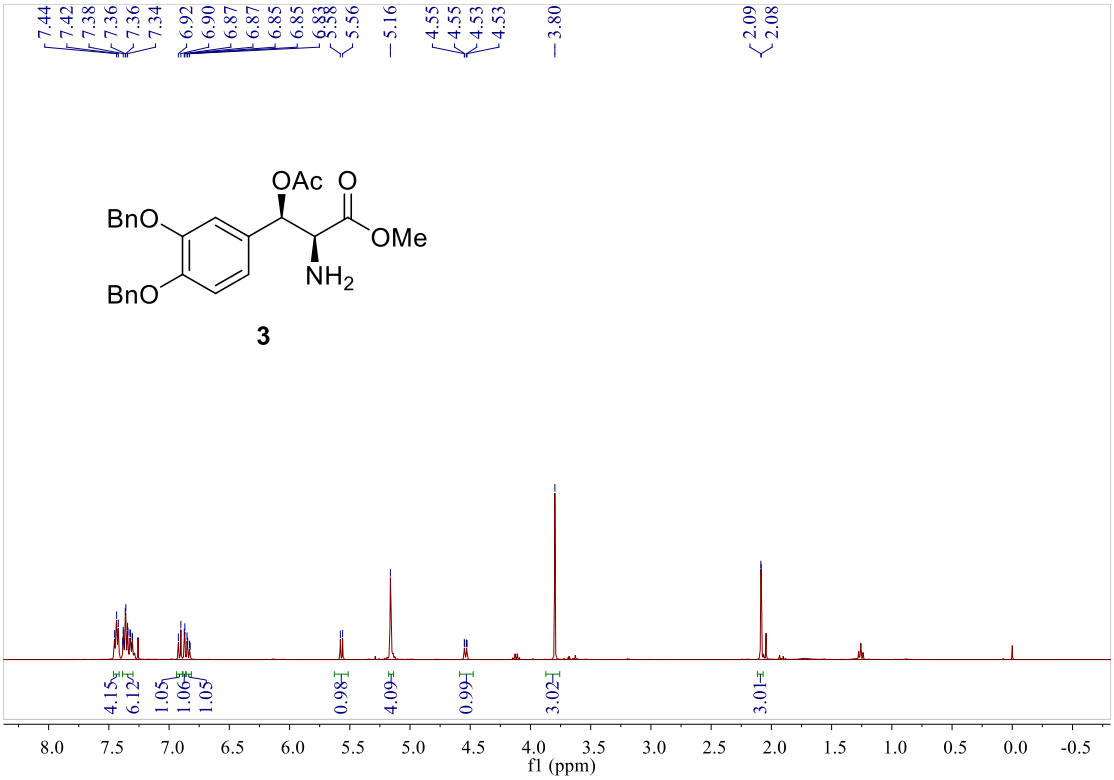
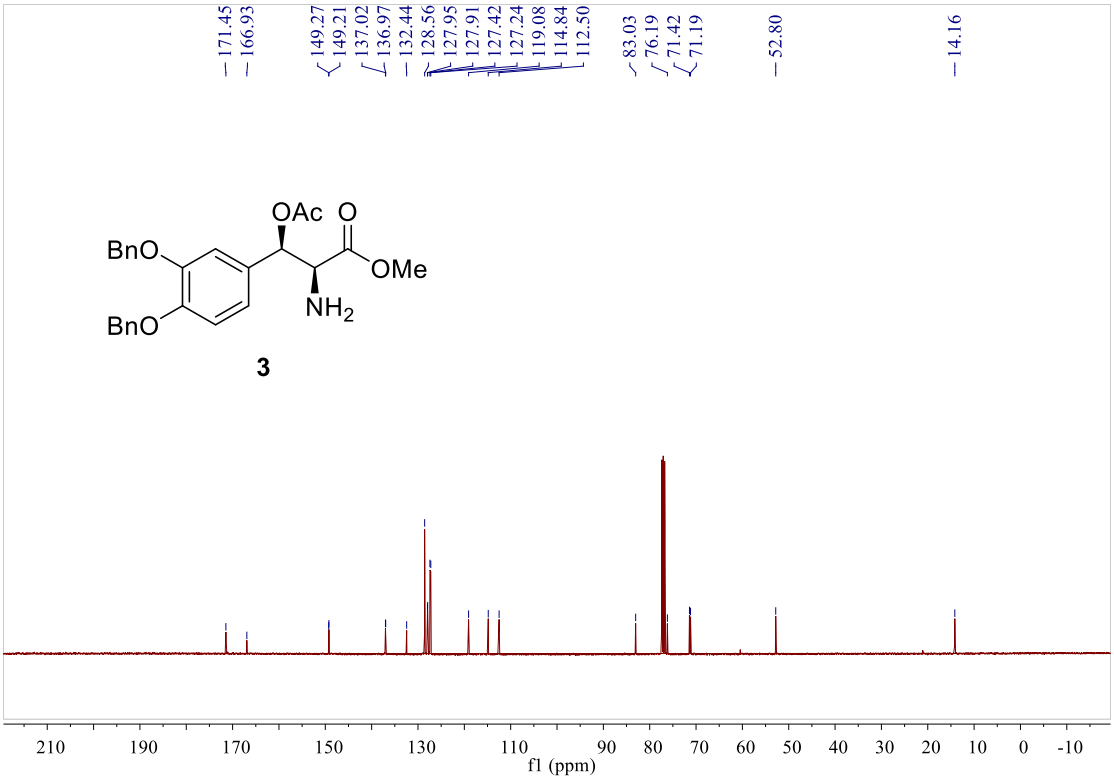
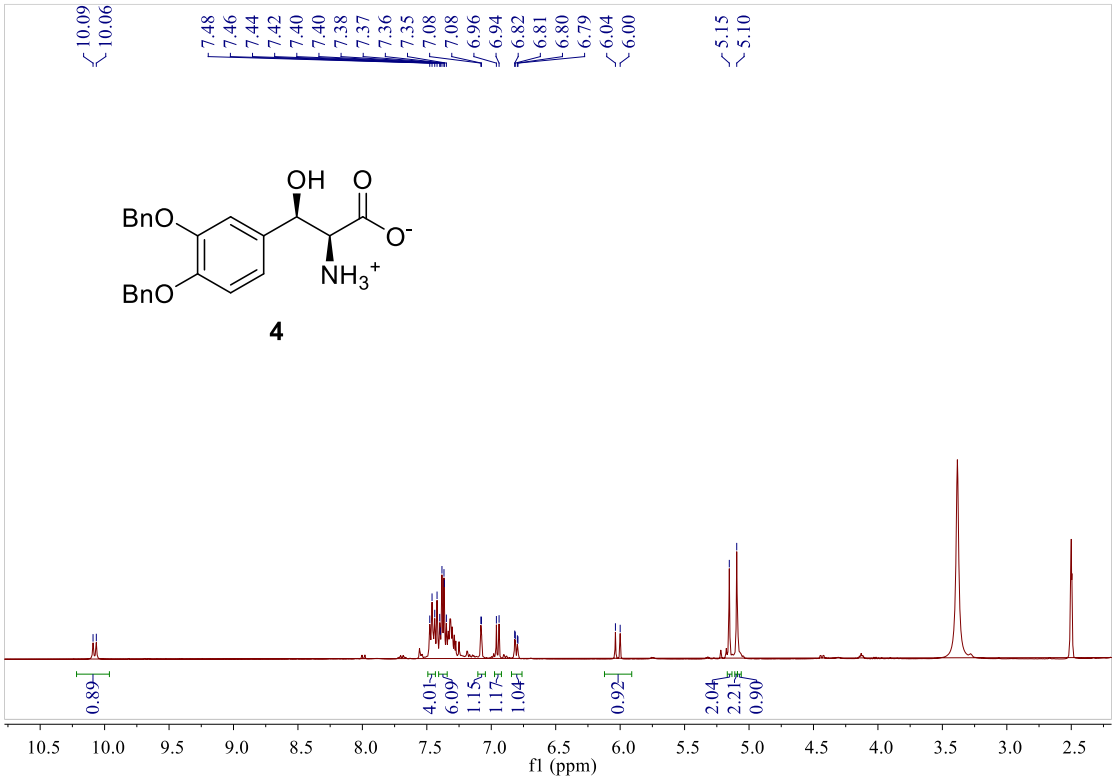
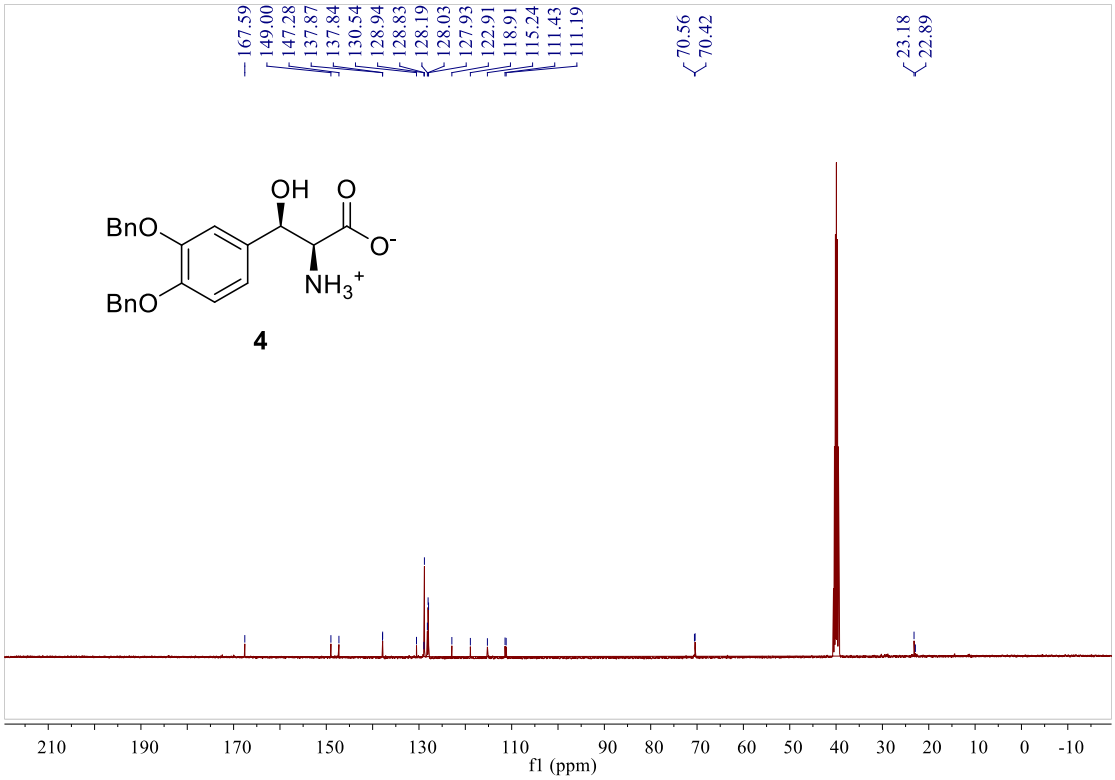
169.71
70.56
61.91
55.40
23.18
20.92
17.07
14.07
OAc O
OEt
NHAc
2o
f1 (ppm)


7.44
7.42
7.38
7.36
7.36
7.34
6.92
6.90
6.87
6.87
6.85
6.85
6.83
5.58
5.56
5.16
4.55
4.55
4.53
4.53
3.80
2.09
2.08
OAc
O
BnO
OMe
NH2
BnO
3
4.15
6.12
1.05
1.06
1.05
0.98
4.09
0.99
3.02
3.01
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)


171.45
166.93
149.27
149.21
137.02
136.97
132.44
128.56
127.95
127.91
127.42
127.24
119.08
114.84
112.50
83.03
76.19
71.42
71.19
52.80
14.16
OAc
O
BnO
OMe
NH2
BnO
3
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


10.09
10.06
7.48
7.46
7.44
7.42
7.40
7.40
7.38
7.37
7.36
7.35
7.08
7.08
6.96
6.94
6.82
6.81
6.80
6.79
6.04
6.00
5.15
5.10
OH
O
BnO
O-
NH3+
BnO
0.89
4.01
6.09
1.15
1.17
1.04
0.92
2.04
2.21
0.90
10.5
10.0
9.5
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
f1 (ppm)

4


167.59
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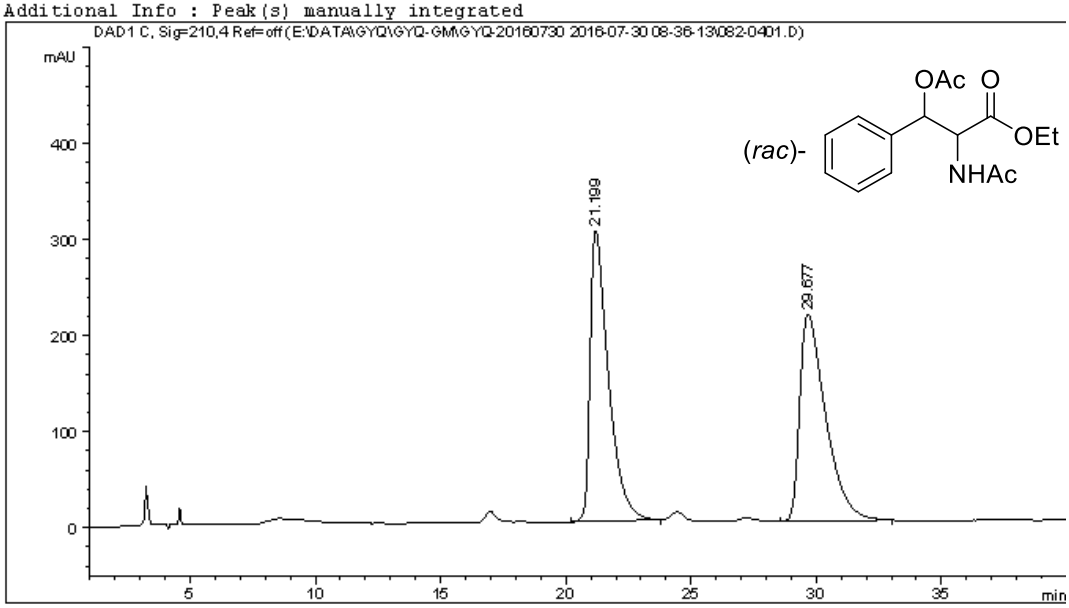
147.28
137.87
137.84
130.54
128.94
128.83
128.19
128.03
127.93
122.91
118.91
115.24
111.43
111.19
70.56
70.42
23.18
22.89
OH
O
BnO
O-
NH3+
BnO
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

4

Data File E:\DATA\GYQ\GYQ-GM\GYQ-20160730 2016-07-30 08-36-13\082-0401.D
Sample Name: Ph-rac

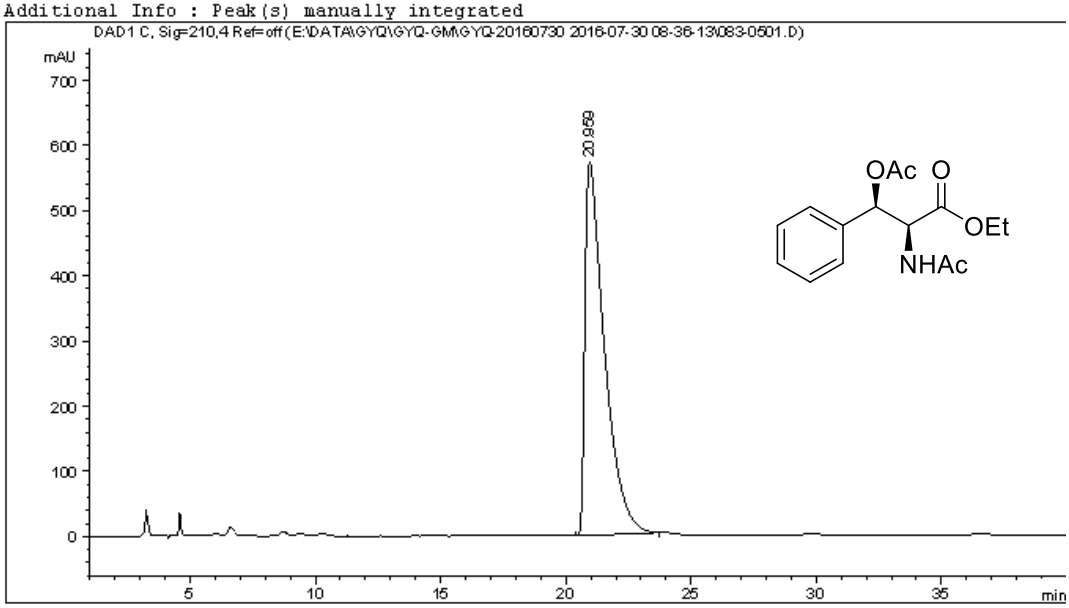
```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   4
    Acq. Instrument : 1260HPLC-DAD                   Location : Vial 82
    Injection Date  : 7/30/2016 9:59:02 AM               Inj :   1
                                                   Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\GYQ\GYQ-GM\GYQ-20160730 2016-07-30 08-36-13\DAD-0J(1-6)-95-5-1.
                      0ML-5-210NM-50MIN.M
    Last changed    : 7/30/2016 9:58:07 AM by SYSTEM
    Analysis Method : E:\DATA\GYQ\GYQ-GM\GYQ-20160730 2016-07-30 08-36-13\DAD-0J(1-6)-95-5-1.
                      0ML-5-210NM-50MIN.M (Sequence Method)
    Last changed    : 6/10/2017 8:57:32 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  21.199 BB    0.7367 1.53169e4   302.53458  49.7010
       2  29.677 BB    1.0322 1.55012e4   215.25539  50.2990

    Totals :                  3.08182e4   517.78996


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

1260HPLC-VWD 6/10/2017 8:57:45 PM SYSTEM                    Page 1 of 1
```

Data File E:\DATA\GYQ\GYQ-GM\GYQ-20160730 2016-07-30 08-36-13\083-0501.D
Sample Name: 30 bar

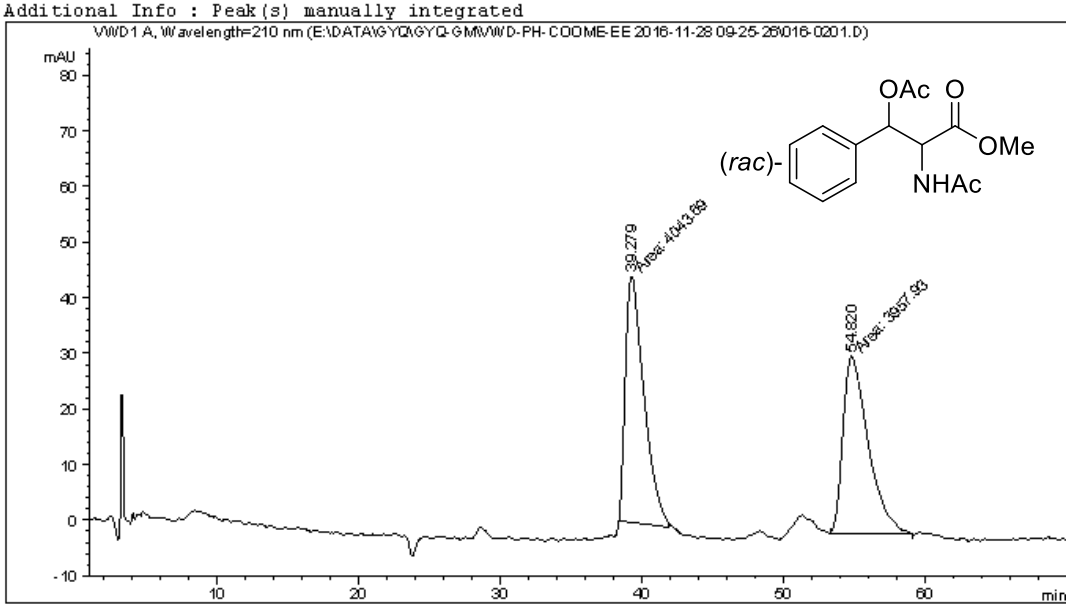
```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   5
    Acq. Instrument : 1260HPLC-DAD                   Location : Vial 83
    Injection Date  : 7/30/2016 10:47:56 AM             Inj :   1
                                                   Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\GYQ\GYQ-GM\GYQ-20160730 2016-07-30 08-36-13\DAD-0J(1-6)-95-5-1.
                      0ML-5-210NM-50MIN.M
    Last changed    : 7/30/2016 9:58:07 AM by SYSTEM
    Analysis Method : E:\DATA\GYQ\GYQ-GM\GYQ-20160730 2016-07-30 08-36-13\DAD-0J(1-6)-95-5-1.
                      0ML-5-210NM-50MIN.M (Sequence Method)
    Last changed    : 6/10/2017 9:01:32 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  20.959 BB    0.7764 3.09458e4   572.71411 100.0000

    Totals :                  3.09458e4   572.71411


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

Data File E:\DATA\GYQ\GYQ-GM\VWD-PH-COOME-EE 2016-11-28 09-25-26\016-0201.D
Sample Name: PH-COOME-RAC

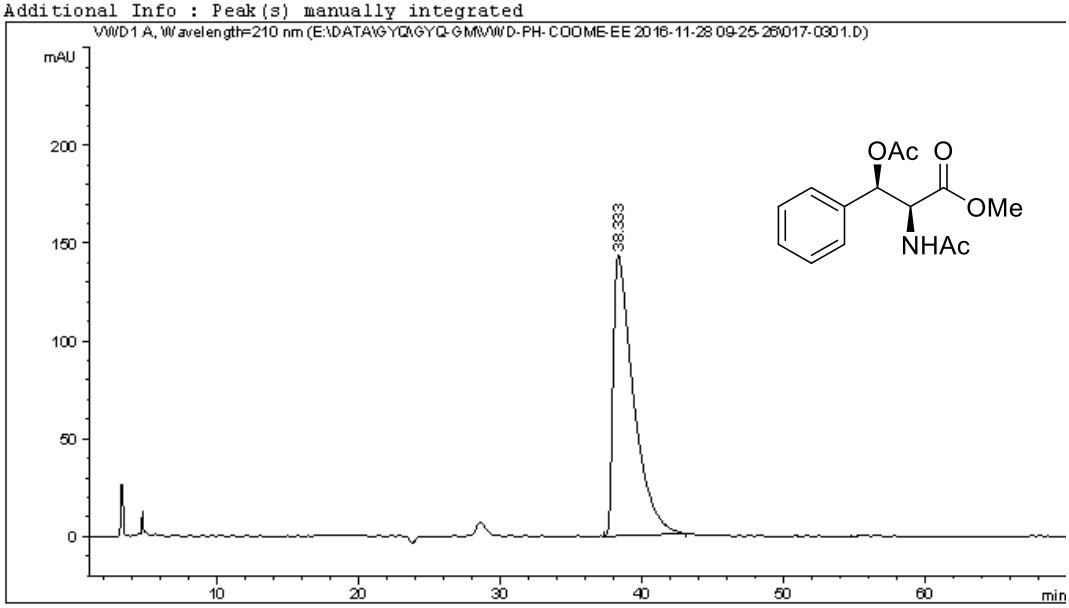
```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   2
    Acq. Instrument : 1260HPLC-VWD                   Location : Vial 16
    Injection Date  : 11/28/2016 9:46:59 AM             Inj :   1
                                                   Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\GYQ\GYQ-GM\VWD-PH-COOME-EE 2016-11-28 09-25-26\VWD-0J(1-6)-95-5-
                      1.0ML-210NM-75MIN.M
    Last changed    : 11/28/2016 9:25:26 AM by SYSTEM
    Analysis Method : E:\DATA\GYQ\GYQ-GM\VWD-PH-COOME-EE 2016-11-28 09-25-26\VWD-0J(1-6)-95-5-
                      1.0ML-210NM-75MIN.M (Sequence Method)
    Last changed    : 6/10/2017 9:04:41 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  39.279 MM    1.5213 4043.68604   44.30155  50.5359
       2  54.820 MM    2.0652 3957.92822   31.94175  49.4641

    Totals :                  8001.61426   76.24330


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

Data File E:\DATA\GYQ\GYQ-GM\VWD-PH-COOME-EE 2016-11-28 09-25-26\017-0301.D
Sample Name: PH-COOME-EE

```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   3
    Acq. Instrument : 1260HPLC-VWD                   Location : Vial 17
    Injection Date  : 11/28/2016 11:02:44 AM              Inj :   1
                                                   Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\GYQ\GYQ-GM\VWD-PH-COOME-EE 2016-11-28 09-25-26\VWD-0J(1-6)-95-5-
                      1.0ML-210NM-75MIN.M
    Last changed    : 11/28/2016 9:25:26 AM by SYSTEM
    Analysis Method : E:\DATA\GYQ\GYQ-GM\VWD-PH-COOME-EE 2016-11-28 09-25-26\VWD-0J(1-6)-95-5-
                      1.0ML-210NM-75MIN.M (Sequence Method)
    Last changed    : 6/10/2017 9:08:02 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```

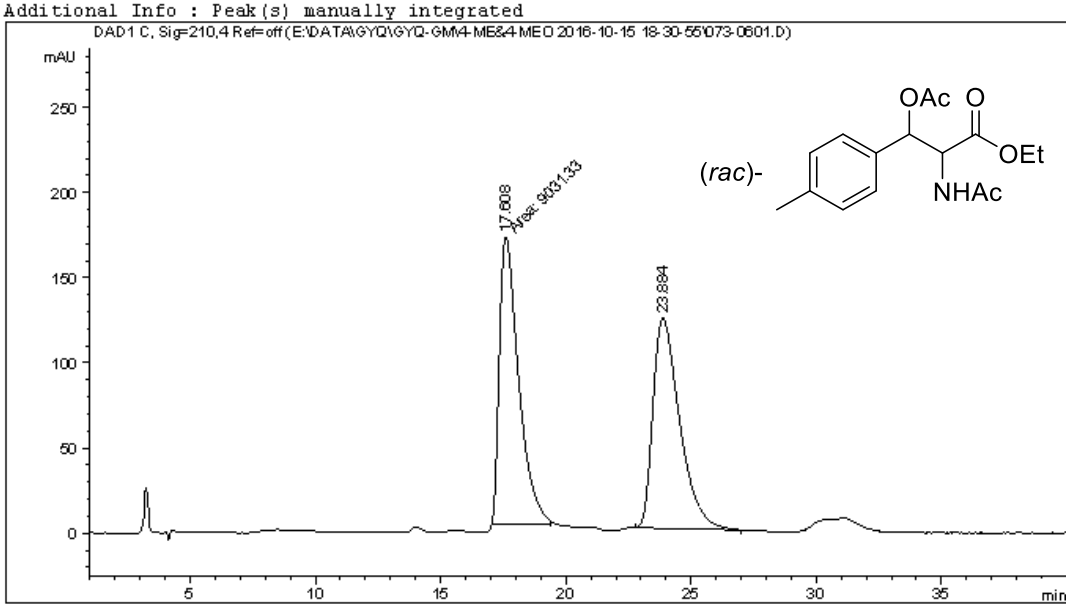



```
    ======================================================================
                              Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    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  38.333 BB    1.4611 1.43183e4   143.24849 100.0000

    Totals :                  1.43183e4   143.24849


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

1260HPLC-VWD 6/10/2017 9:08:08 PM SYSTEM Page 1 of 1

Data File E:\DATA\GYQ\GYQ-GM\4-ME&4-MEO 2016-10-15 18-30-55\073-0601.D
Sample Name: 4-Me-rac

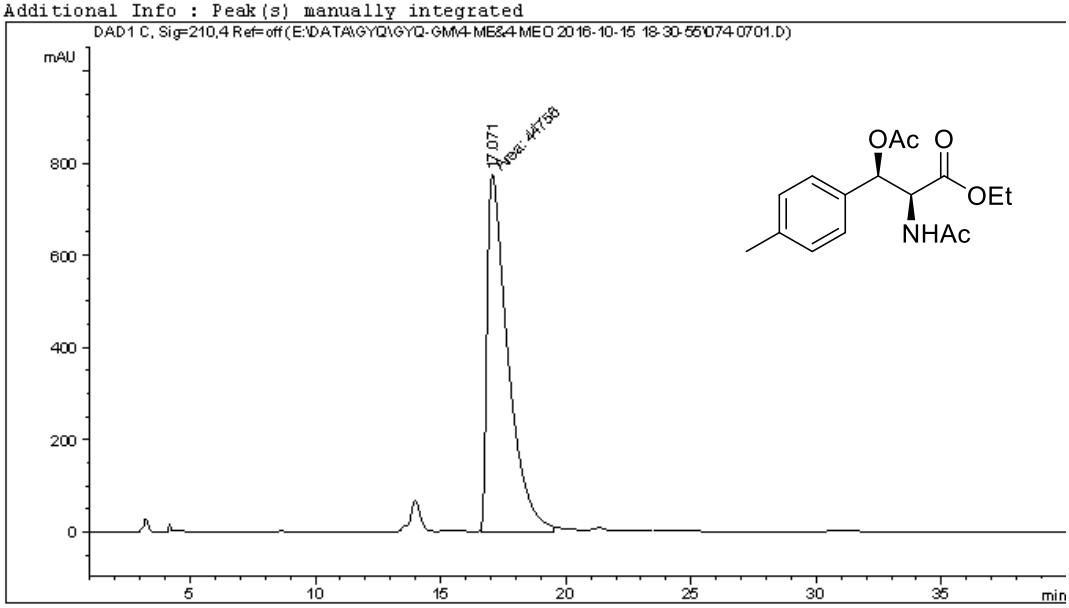
```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   6
    Acq. Instrument : 1260HPLC-DAD                   Location : Vial 73
    Injection Date  : 10/15/2016 10:33:42 PM              Inj :   1
                                                   Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\GYQ\GYQ-GM\4-ME&4-MEO 2016-10-15 18-30-55\DAD-0J(1-6)-95-5-1.0ML
                      -5-210NM-50MIN.M
    Last changed    : 10/15/2016 6:30:56 PM by SYSTEM
    Analysis Method : E:\DATA\GYQ\GYQ-GM\4-ME&4-MEO 2016-10-15 18-30-55\DAD-0J(1-6)-95-5-1.0ML
                      -5-210NM-50MIN.M (Sequence Method)
    Last changed    : 6/10/2017 9:10:36 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  17.608 MM    0.8916 9031.32617  168.82851  49.6320
       2  23.884 BB    0.9673 9165.26660  123.57682  50.3680

    Totals :                  1.81966e4   292.40533


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

Data File E:\DATA\GYQ\GYQ-GM\4-ME&4-MEO 2016-10-15 18-30-55\074-0701.D
Sample Name: 4-Me-ee

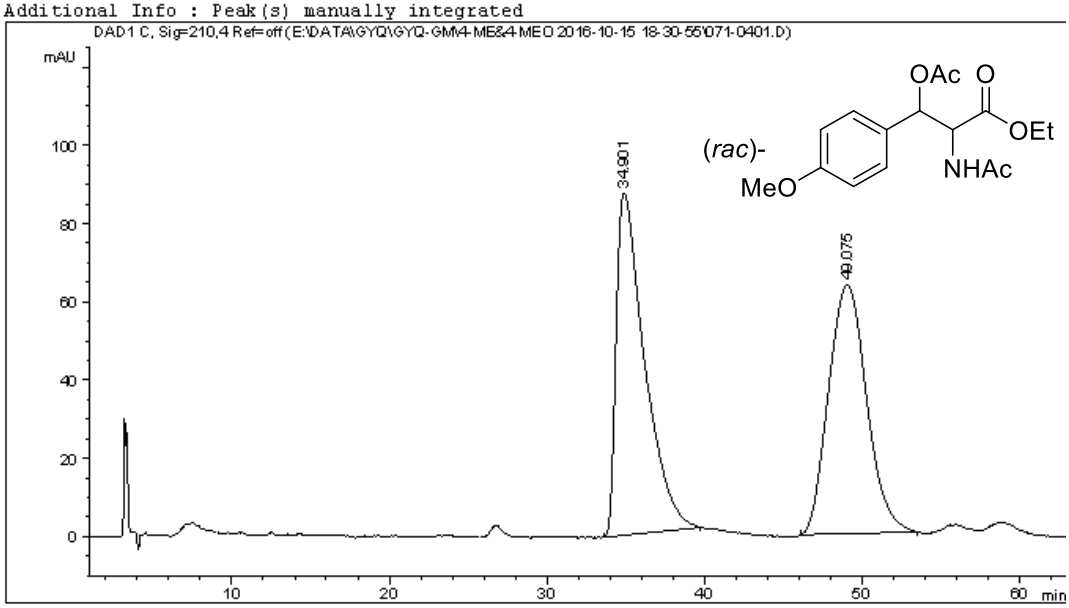
```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   7
    Acq. Instrument : 1260HPLC-DAD                   Location : Vial 74
    Injection Date  : 10/15/2016 11:24:41 PM              Inj :   1
                                                   Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\GYQ\GYQ-GM\4-ME&4-MEO 2016-10-15 18-30-55\DAD-0J(1-6)-95-5-1.0ML
                      -5-210NM-50MIN.M
    Last changed    : 10/15/2016 6:30:56 PM by SYSTEM
    Analysis Method : E:\DATA\GYQ\GYQ-GM\4-ME&4-MEO 2016-10-15 18-30-55\DAD-0J(1-6)-95-5-1.0ML
                      -5-210NM-50MIN.M (Sequence Method)
    Last changed    : 6/10/2017 9:12:41 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  17.071 MM    0.9642 4.47560e4   773.66003 100.0000

    Totals :                  4.47560e4   773.66003


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

Data File E:\DATA\GYQ\GYQ-GM\4-ME&4-ME0 2016-10-15 18-30-55\071-0401.D
Sample Name: 4-Me0-rac

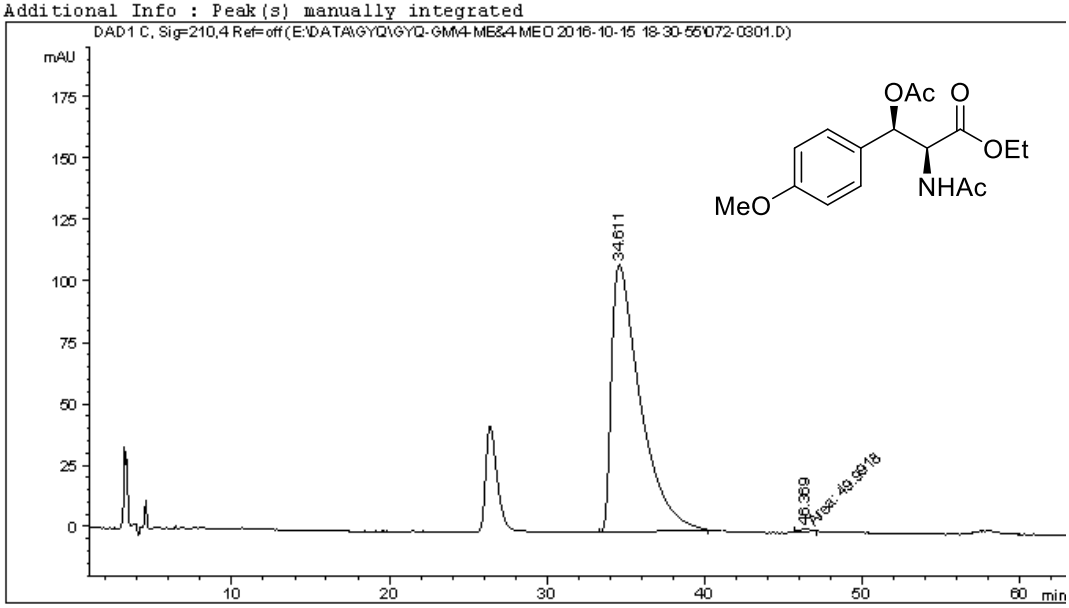
```
    ====================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   4
    Acq. Instrument : 1260HPLC-DAD                   Location : Vial 71
    Injection Date  : 10/15/2016 9:06:45 PM             Inj :   1
                                                   Inj Volume : 10.000 µl
    Acq. Method     : E:\DATA\GYQ\GYQ-GM\4-ME&4-ME0 2016-10-15 18-30-55\DAD-0J(1-6)-94-6-1.0ML
                      -5-210NM-65MIN.M
    Last changed    : 10/15/2016 6:30:55 PM by SYSTEM
    Analysis Method : E:\DATA\GYQ\GYQ-GM\4-ME&4-ME0 2016-10-15 18-30-55\DAD-0J(1-6)-94-6-1.0ML
                      -5-210NM-65MIN.M (Sequence Method)
    Last changed    : 6/10/2017 9:16:27 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  34.901 BB    1.4598 1.0925le4    87.55154  50.5613
       2  49.075 BB    1.9706 1.06826e4    63.63345  49.4387

    Totals :                  2.16077e4   151.18500


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

Data File E:\DATA\GYQ\GYQ-GM\4-ME&4-MEO 2016-10-15 18-30-55\072-0301.D
Sample Name: 4-MeO-ee

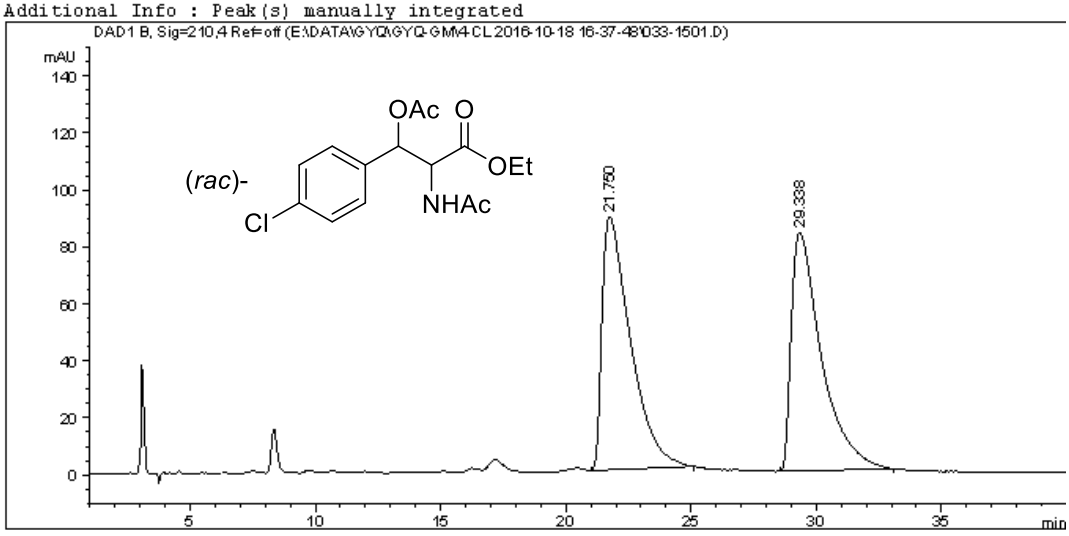
```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   3
    Acq. Instrument : 1260HPLC-DAD                   Location : Vial 72
    Injection Date  : 10/15/2016 8:00:47 PM             Inj :   1
                                                   Inj Volume : 10.000 µl
    Acq. Method     : E:\DATA\GYQ\GYQ-GM\4-ME&4-MEO 2016-10-15 18-30-55\DAD-0J(1-6)-94-6-1.0ML
                      -5-210NM-65MIN.M
    Last changed    : 10/15/2016 6:30:55 PM by SYSTEM
    Analysis Method : E:\DATA\GYQ\GYQ-GM\4-ME&4-MEO 2016-10-15 18-30-55\DAD-0J(1-6)-94-6-1.0ML
                      -5-210NM-65MIN.M (Sequence Method)
    Last changed    : 6/10/2017 9:17:51 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  34.611 BB    1.4914 1.37987e4   108.81527  99.6390
       2  46.369 MM    0.8820   49.99180 9.44638e-1   0.3610

    Totals :                  1.38487e4   109.75991


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

Data File E:\DATA\GYQ\GYQ-GM\4-CL 2016-10-18 16-37-48\033-1501.D
Sample Name: 4-Cl-rac

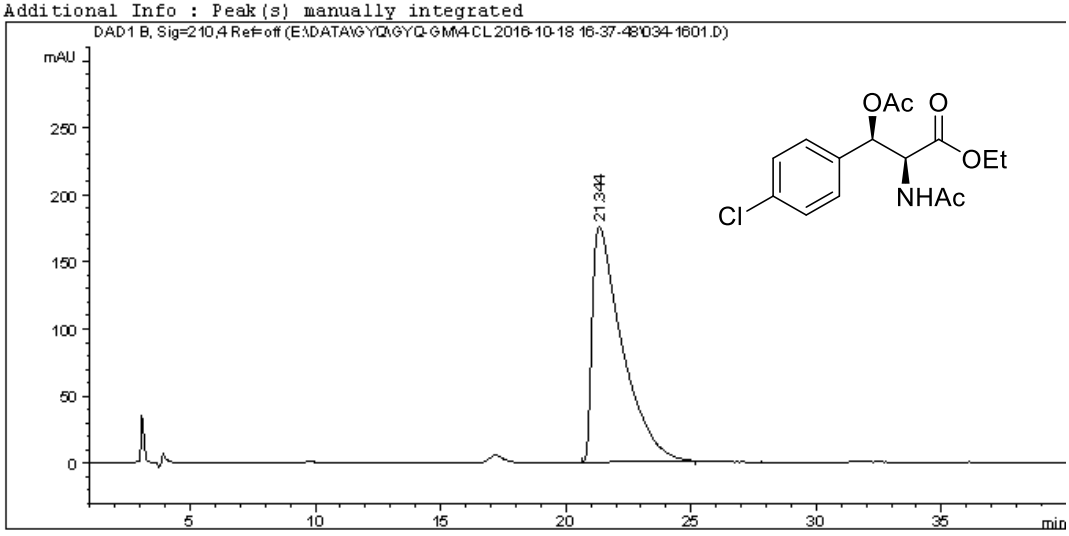
```
    ======================================================================
    Acq. Operator   : SYSTEM                          Seq. Line :  15
    Acq. Instrument : 1260HPLC-DAD                     Location : Vial 33
    Injection Date  : 10/19/2016 12:32:34 AM               Inj :   1
                                                     Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\GYQ\GYQ-GM\4-CL 2016-10-18 16-37-48\DAD-OD(1-2)-95-5-1ML-50MIN-
                      210.M
    Last changed    : 10/18/2016 4:37:50 PM by SYSTEM
    Analysis Method : E:\DATA\GYQ\GYQ-GM\4-CL 2016-10-18 16-37-48\DAD-OD(1-2)-95-5-1ML-50MIN-
                      210.M (Sequence Method)
    Last changed    : 6/10/2017 9:20:10 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 B, Sig=210,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  21.750 BB    0.9250 6998.43799   88.84822  49.9765
       2  29.338 BB    1.0107 7005.03320   83.84902  50.0235

    Totals :                  1.40035e4   172.69724

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

Data File E:\DATA\GYQ\GYQ-GM\4-CL 2016-10-18 16-37-48\034-1601.D
Sample Name: 4-Cl-ee

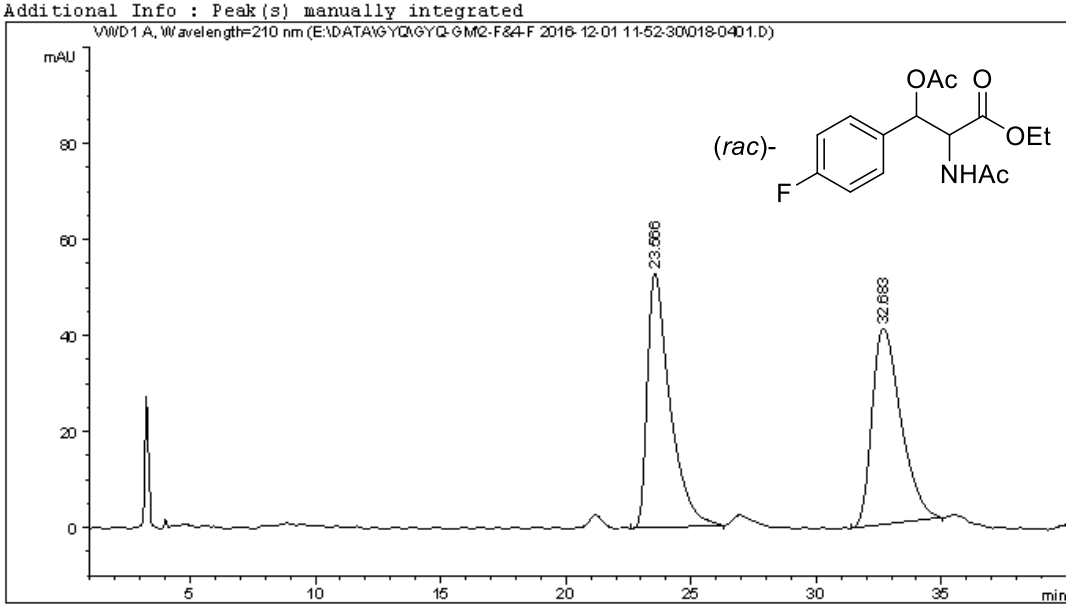
```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :  16
    Acq. Instrument : 1260HPLC-DAD                   Location : Vial 34
    Injection Date  : 10/19/2016 1:23:28 AM               Inj :  1
                                                   Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\GYQ\GYQ-GM\4-CL 2016-10-18 16-37-48\DAD-OD(1-2)-95-5-1ML-50MIN-
                      210.M
    Last changed    : 10/18/2016 4:37:50 PM by SYSTEM
    Analysis Method : E:\DATA\GYQ\GYQ-GM\4-CL 2016-10-18 16-37-48\DAD-OD(1-2)-95-5-1ML-50MIN-
                      210.M (Sequence Method)
    Last changed    : 6/10/2017 9:22:13 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 B, Sig=210,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  21.344 BB    1.0863 1.46163e4   175.73535 100.0000

    Totals :                  1.46163e4   175.73535

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

Data File E:\DATA\GYQ\GYQ-GM\2-F&4-F 2016-12-01 11-52-30\018-0401.D
Sample Name: 4-F-RAC

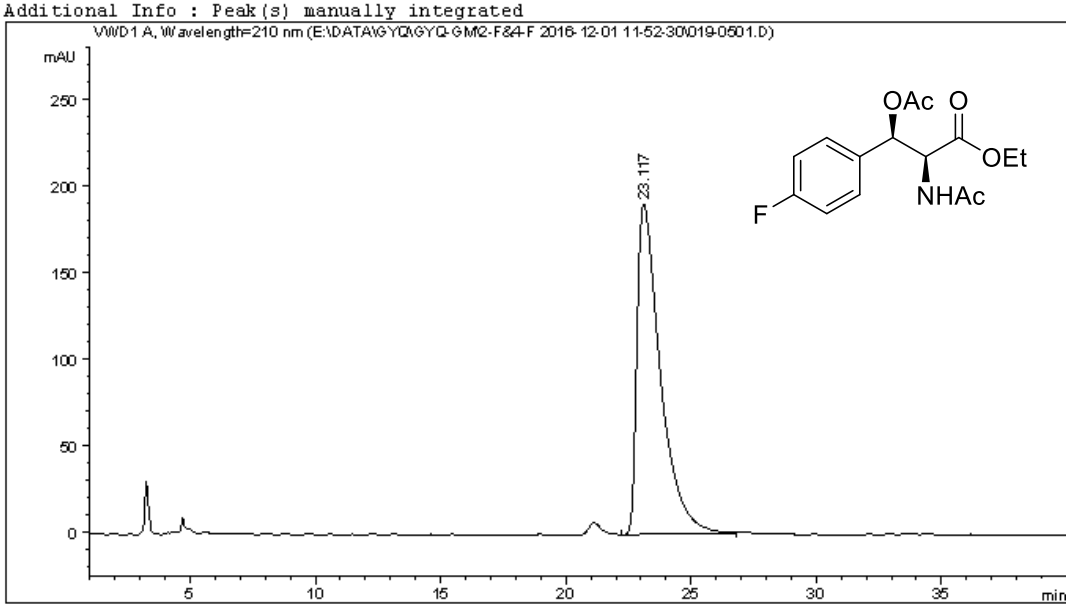
```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   4
    Acq. Instrument : 1260HPLC-VWD                   Location : Vial 18
    Injection Date  : 12/1/2016 1:56:34 PM               Inj :   1
                                                   Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\GYQ\GYQ-GM\2-F&4-F 2016-12-01 11-52-30\VWD-0J(1-2)-95-5-1.0ML-
                      5UL-210NM-40MIN.M
    Last changed    : 12/1/2016 11:52:30 AM by SYSTEM
    Analysis Method : E:\DATA\GYQ\GYQ-GM\2-F&4-F 2016-12-01 11-52-30\VWD-0J(1-2)-95-5-1.0ML-
                      5UL-210NM-40MIN.M (Sequence Method)
    Last changed    : 6/10/2017 9:28:49 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    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  23.566 BB    0.9158 3328.55859   53.06331  50.3793
       2  32.683 BB    1.2065 3278.43726   40.71347  49.6207

    Totals :                  6606.99585   93.77678


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

Data File E:\DATA\GYQ\GYQ-GM\2-F&4-F 2016-12-01 11-52-30\019-0501.D
Sample Name: 4-F-EE

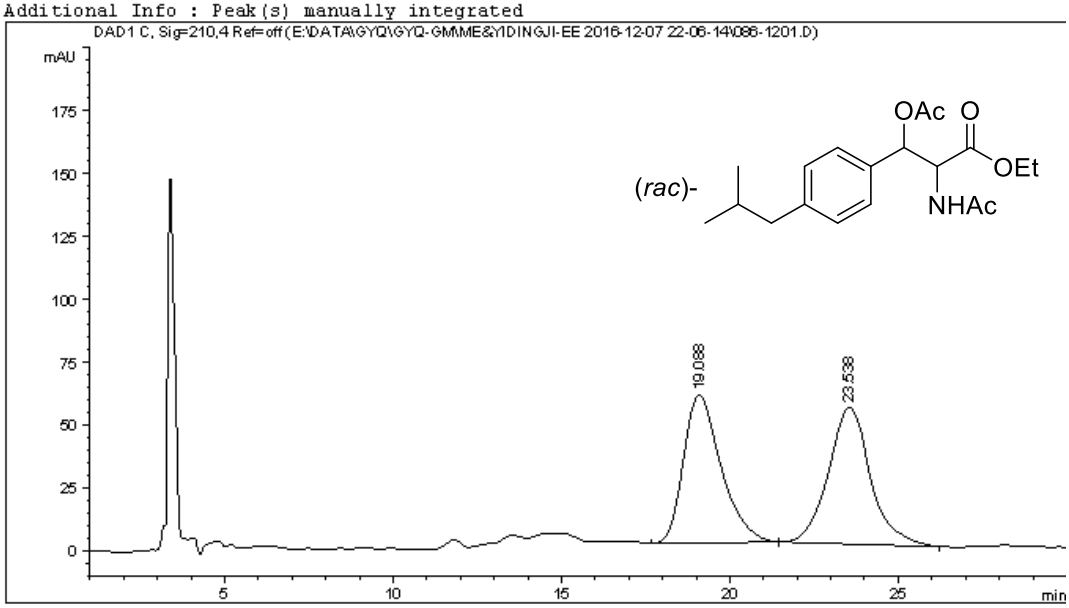
```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   5
    Acq. Instrument : 1260HPLC-VWD                   Location : Vial 19
    Injection Date  : 12/1/2016 2:37:21 PM               Inj :   1
                                                   Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\GYQ\GYQ-GM\2-F&4-F 2016-12-01 11-52-30\VWD-0J(1-2)-95-5-1.0ML-
                      5UL-210NM-40MIN.M
    Last changed    : 12/1/2016 11:52:30 AM by SYSTEM
    Analysis Method : E:\DATA\GYQ\GYQ-GM\2-F&4-F 2016-12-01 11-52-30\VWD-0J(1-2)-95-5-1.0ML-
                      5UL-210NM-40MIN.M (Sequence Method)
    Last changed    : 6/10/2017 9:30:30 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  23.117 BB    0.9716 1.23525e4   190.71989 100.0000

    Totals :                  1.23525e4   190.71989


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

Data File E:\DATA\GYQ\GYQ-GM\ME&YIDINGJI-EE 2016-12-07 22-06-14\086-1201.D
Sample Name: yidingji-rac

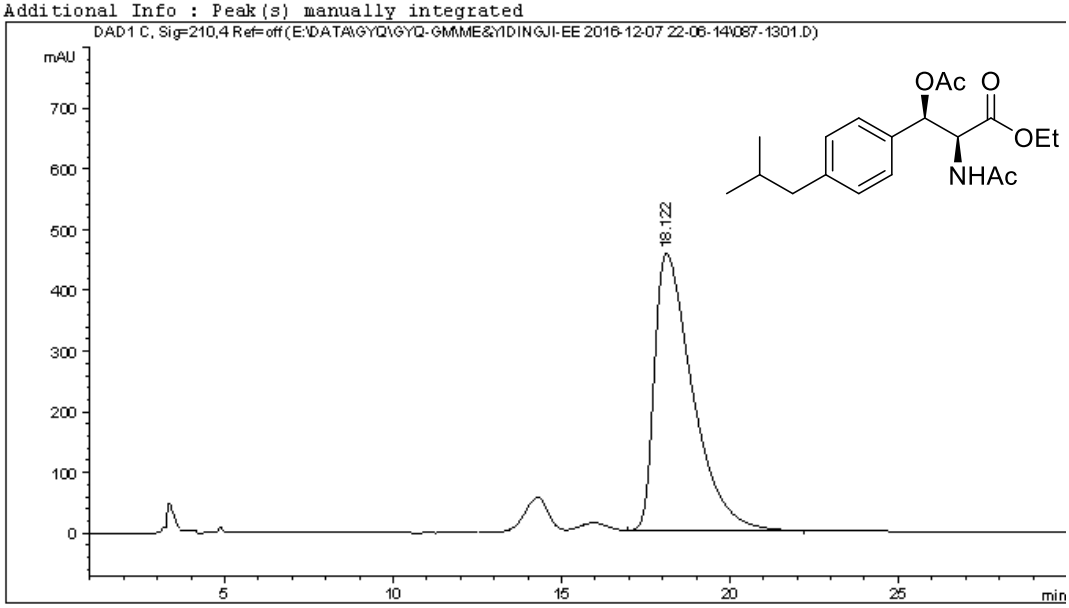
```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :  12
    Acq. Instrument : 1260HPLC-DAD                   Location : Vial 86
    Injection Date  : 12/8/2016 4:19:51 AM                Inj :  1
                                                  Inj Volume : 10.000 µl
    Acq. Method     : E:\DATA\GYQ\GYQ-GM\ME&YIDINGJI-EE 2016-12-07 22-06-14\DAD-0J(1-6)-97-3-1
                      .OML-10-210NM-75MIN.M
    Last changed    : 12/7/2016 10:06:16 PM by SYSTEM
    Analysis Method : E:\DATA\GYQ\GYQ-GM\ME&YIDINGJI-EE 2016-12-07 22-06-14\DAD-0J(1-6)-97-3-1
                      .OML-10-210NM-75MIN.M (Sequence Method)
    Last changed    : 6/10/2017 9:45:48 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    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  19.088 BB    1.1227 4571.60693   58.95211  49.1674
       2  23.538 BB    1.1568 4726.43604   54.33568  50.8326

    Totals :                  9298.04297  113.28780


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

Data File E:\DATA\GYQ\GYQ-GM\ME&YIDINGJI-EE 2016-12-07 22-06-14\087-1301.D
Sample Name: yidingji-ee

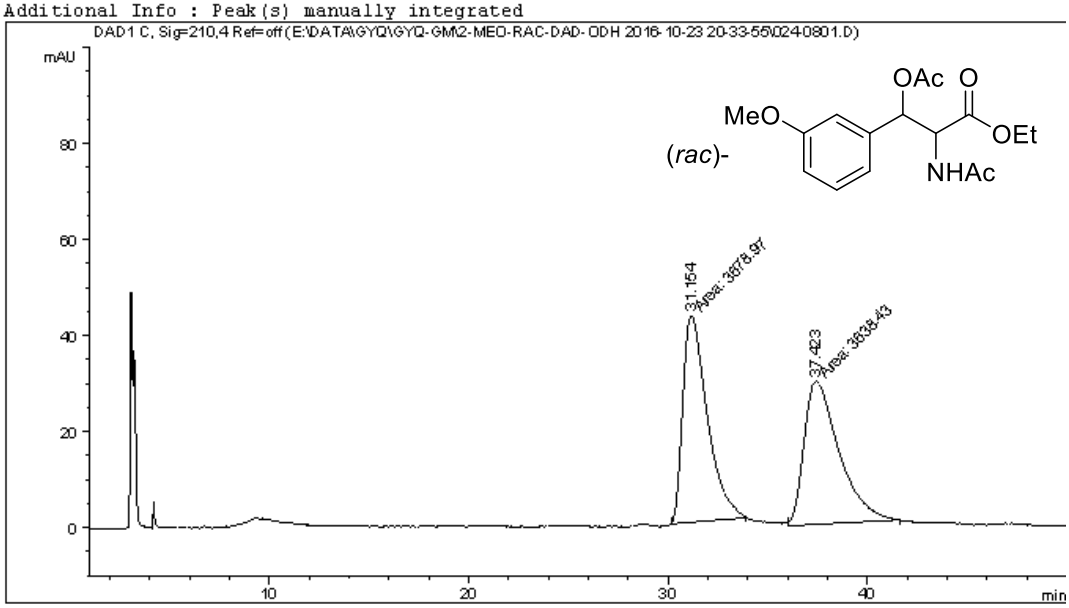
```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :  13
    Acq. Instrument : 1260HPLC-DAD                   Location : Vial 87
    Injection Date  : 12/8/2016 5:35:51 AM               Inj :   1
                                                   Inj Volume : 10.000 µl
    Acq. Method     : E:\DATA\GYQ\GYQ-GM\ME&YIDINGJI-EE 2016-12-07 22-06-14\DAD-0J(1-6)-97-3-1
                      .OML-10-210NM-75MIN.M
    Last changed    : 12/7/2016 10:06:16 PM by SYSTEM
    Analysis Method : E:\DATA\GYQ\GYQ-GM\ME&YIDINGJI-EE 2016-12-07 22-06-14\DAD-0J(1-6)-97-3-1
                      .OML-10-210NM-75MIN.M (Sequence Method)
    Last changed    : 6/10/2017 9:47:02 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    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  18.122 BB    1.1764 3.58620e4   456.55618 100.0000

    Totals :                  3.58620e4   456.55618


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

Data File E:\DATA\GYQ\GYQ-GM\2-MEO-RAC-DAD-ODH 2016-10-23 20-33-55\024-0801.D
Sample Name: 3-MeO-rac

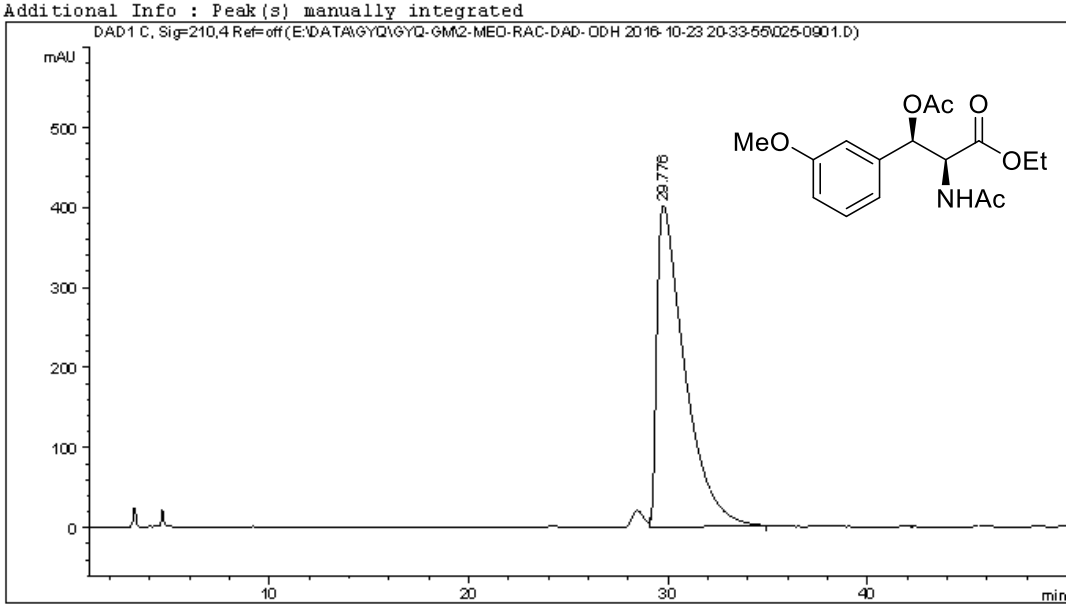
```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   8
    Acq. Instrument : 1260HPLC-DAD                   Location : Vial 24
    Injection Date  : 10/24/2016 12:38:22 AM              Inj :   1
                                                   Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\GYQ\GYQ-GM\2-MEO-RAC-DAD-ODH 2016-10-23 20-33-55\DAD-OJ(1-6)-95-
                      5-1.0ML-5-210NM-50MIN.M
    Last changed    : 10/23/2016 10:10:49 PM by SYSTEM
    Analysis Method : E:\DATA\GYQ\GYQ-GM\2-MEO-RAC-DAD-ODH 2016-10-23 20-33-55\DAD-OJ(1-6)-95-
                      5-1.0ML-5-210NM-50MIN.M (Sequence Method)
    Last changed    : 6/10/2017 9:49:21 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    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  31.154 MM    1.4238 3678.96729   43.06536  50.2770
       2  37.423 MM    2.0392 3638.43311   29.73787  49.7230

    Totals :                  7317.40039   72.80323


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

Data File E:\DATA\GYQ\GYQ-GM\2-MEO-RAC-DAD-ODH 2016-10-23 20-33-55\025-0901.D
Sample Name: 3-MeO-ee

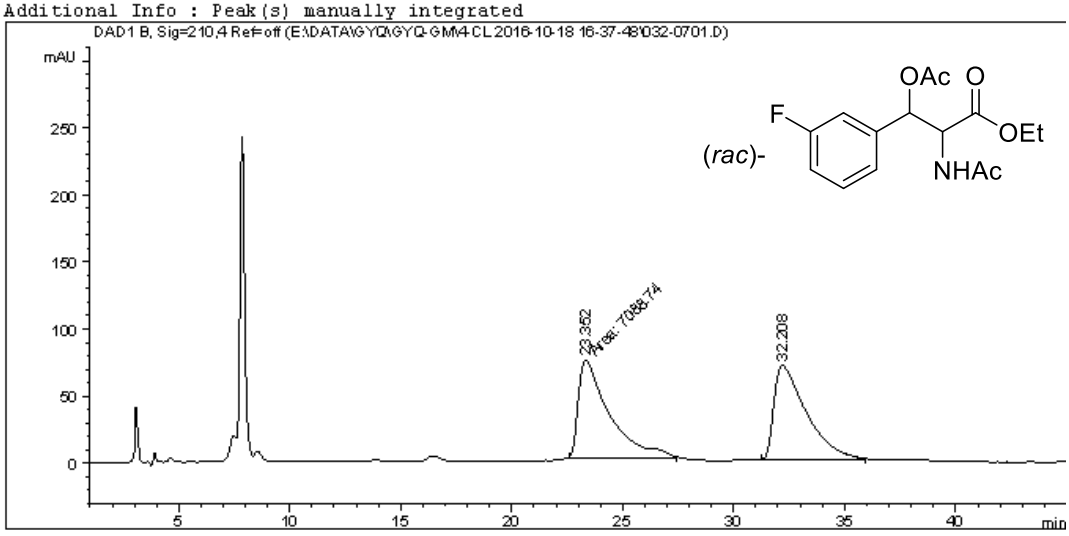
```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   9
    Acq. Instrument : 1260HPLC-DAD                   Location : Vial 25
    Injection Date  : 10/24/2016 1:29:17 AM             Inj :   1
                                                   Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\GYQ\GYQ-GM\2-MEO-RAC-DAD-ODH 2016-10-23 20-33-55\DAD-OJ(1-6)-95-
                      5-1.0ML-5-210NM-50MIN.M
    Last changed    : 10/23/2016 10:10:49 PM by SYSTEM
    Analysis Method : E:\DATA\GYQ\GYQ-GM\2-MEO-RAC-DAD-ODH 2016-10-23 20-33-55\DAD-OJ(1-6)-95-
                      5-1.0ML-5-210NM-50MIN.M (Sequence Method)
    Last changed    : 6/10/2017 9:50:49 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    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  29.776 VB    1.2468 3.88495e4   401.51578 100.0000

    Totals :                  3.88495e4   401.51578


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

Data File E:\DATA\GYQ\GYQ-GM\4-CL 2016-10-18 16-37-48\032-0701.D
Sample Name: 3-F-rac

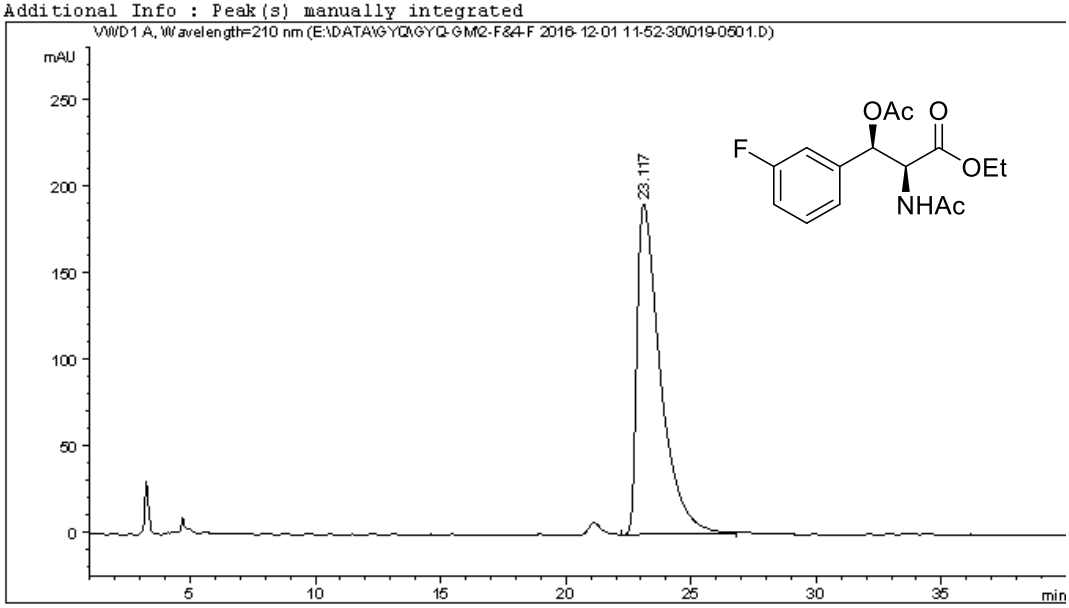
```
    ======================================================================
    Acq. Operator   : SYSTEM                          Seq. Line :   7
    Acq. Instrument : 1260HPLC-DAD                     Location : Vial 32
    Injection Date  : 10/18/2016 8:14:45 PM               Inj :   1
                                                     Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\GYQ\GYQ-GM\4-CL 2016-10-18 16-37-48\DAD-OD(1-2)-95-5-1ML-50MIN-
                      210.M
    Last changed    : 10/18/2016 4:37:50 PM by SYSTEM
    Analysis Method : E:\DATA\GYQ\GYQ-GM\4-CL 2016-10-18 16-37-48\DAD-OD(1-2)-95-5-1ML-50MIN-
                      210.M (Sequence Method)
    Last changed    : 6/10/2017 9:55:48 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Do not 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  23.352 MM    1.6241 7088.74121   72.74491  50.2422
       2  32.208 BB    1.1787 7020.39209   69.84410  49.7578

    Totals :                  1.4109le4   142.58901

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

Data File E:\DATA\GYQ\GYQ-GM\2-F&4-F 2016-12-01 11-52-30\019-0501.D
Sample Name: 4-F-EE

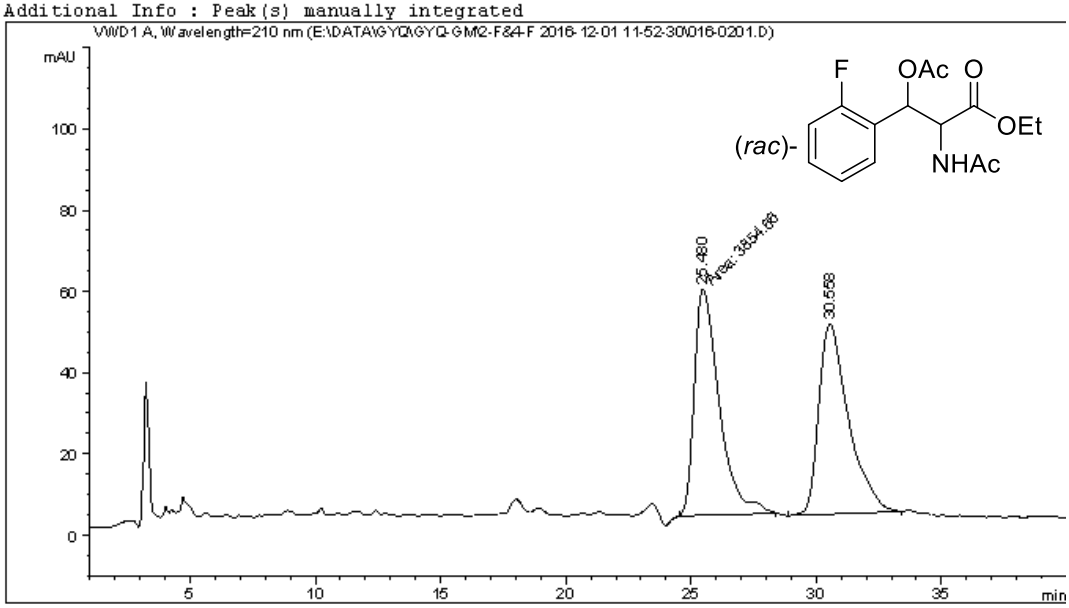
```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   5
    Acq. Instrument : 1260HPLC-VWD                   Location : Vial 19
    Injection Date  : 12/1/2016 2:37:21 PM               Inj :   1
                                                   Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\GYQ\GYQ-GM\2-F&4-F 2016-12-01 11-52-30\VWD-0J(1-2)-95-5-1.0ML-
                      5UL-210NM-40MIN.M
    Last changed    : 12/1/2016 11:52:30 AM by SYSTEM
    Analysis Method : E:\DATA\GYQ\GYQ-GM\2-F&4-F 2016-12-01 11-52-30\VWD-0J(1-2)-95-5-1.0ML-
                      5UL-210NM-40MIN.M (Sequence Method)
    Last changed    : 6/10/2017 9:30:30 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  23.117 BB    0.9716 1.23525e4   190.71989 100.0000

    Totals :                  1.23525e4   190.71989


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

Data File E:\DATA\GYQ\GYQ-GM\2-F&4-F 2016-12-01 11-52-30\016-0201.D
Sample Name: 2-F-RAC

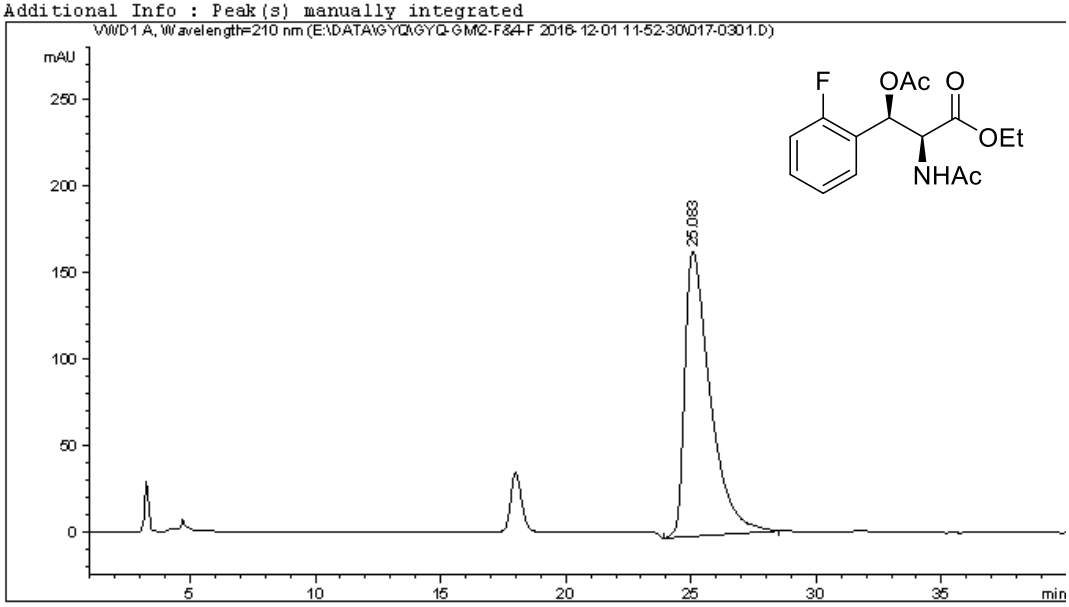
```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   2
    Acq. Instrument : 1260HPLC-VWD                   Location : Vial 16
    Injection Date  : 12/1/2016 12:15:02 PM             Inj :   1
                                                   Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\GYQ\GYQ-GM\2-F&4-F 2016-12-01 11-52-30\VWD-0J(1-2)-95-5-1.0ML-
                      3UL-210NM-60MIN.M
    Last changed    : 12/1/2016 11:52:30 AM by SYSTEM
    Analysis Method : E:\DATA\GYQ\GYQ-GM\2-F&4-F 2016-12-01 11-52-30\VWD-0J(1-2)-95-5-1.0ML-
                      3UL-210NM-60MIN.M (Sequence Method)
    Last changed    : 6/10/2017 9:33:13 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  25.480 MM    1.1541 3854.65820   55.66693  49.8791
       2  30.558 BB    1.2099 3873.35205   46.73795  50.1209

    Totals :                  7728.01025  102.40487


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

Data File E:\DATA\GYQ\GYQ-GM\2-F&4-F 2016-12-01 11-52-30\017-0301.D
Sample Name: 2-F-EE

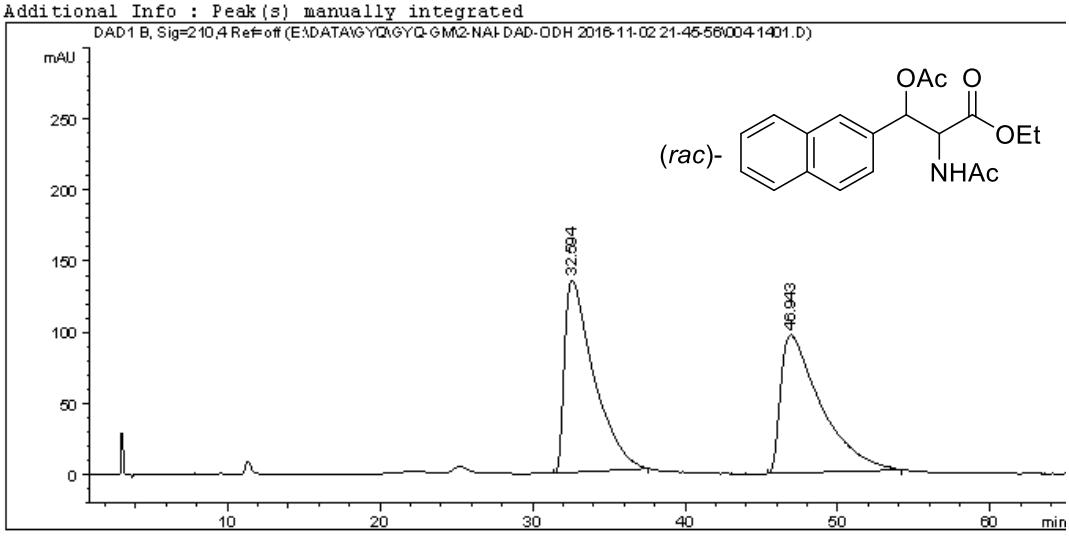
```
    ======================================================================
    Acq. Operator   : SYSTEM                          Seq. Line :   3
    Acq. Instrument : 1260HPLC-VWD                     Location : Vial 17
    Injection Date  : 12/1/2016 1:15:49 PM                 Inj :   1
                                                     Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\GYQ\GYQ-GM\2-F&4-F 2016-12-01 11-52-30\VWD-0J(1-2)-95-5-1.0ML-
                      5UL-210NM-40MIN.M
    Last changed    : 12/1/2016 11:52:30 AM by SYSTEM
    Analysis Method : E:\DATA\GYQ\GYQ-GM\2-F&4-F 2016-12-01 11-52-30\VWD-0J(1-2)-95-5-1.0ML-
                      5UL-210NM-40MIN.M (Sequence Method)
    Last changed    : 6/10/2017 9:32:28 PM by SYSTEM
    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  25.083 BB    1.0454 1.16220e4   164.27574 100.0000

    Totals :                  1.16220e4   164.27574


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

Data File E:\DATA\GYQ\GYQ-GM\2-NAI-DAD-ODH 2016-11-02 21-45-56\004-1401.D
Sample Name: 2-nai-rac

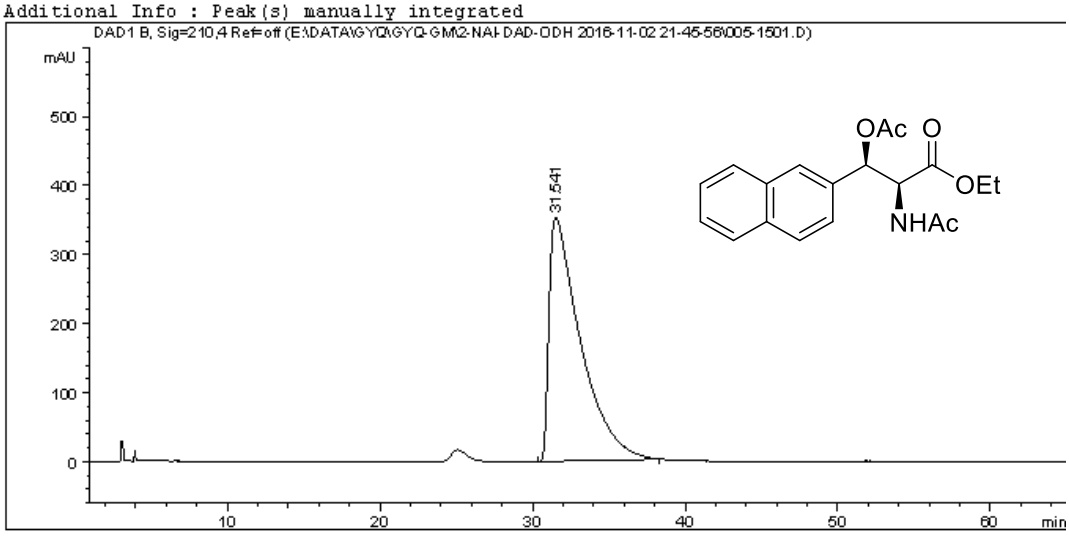
```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :  14
    Acq. Instrument : 1260HPLC-DAD                   Location : Vial 4
    Injection Date  : 11/3/2016 2:58:41 PM               Inj :  1
                                                   Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\GYQ\GYQ-GM\2-NAI-DAD-ODH 2016-11-02 21-45-56\DAD-OD(1-2)-95-5-
                      1ML-75MIN-210-254.M
    Last changed    : 11/3/2016 4:09:32 PM by SYSTEM
                      (modified after loading)
    Analysis Method : E:\DATA\GYQ\GYQ-GM\2-NAI-DAD-ODH 2016-11-02 21-45-56\DAD-OD(1-2)-95-5-
                      1ML-75MIN-210-254.M (Sequence Method)
    Last changed    : 6/10/2017 10:03:39 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: DAD1 B, Sig=210,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  32.594 BB    1.5638 1.80568e4   135.23212  50.3901
       2  46.943 BB    2.1466 1.77773e4    96.89047  49.6099

    Totals :                  3.5834le4   232.12259


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

Data File E:\DATA\GYQ\GYQ-GM\2-NAI-DAD-ODH 2016-11-02 21-45-56\005-1501.D
Sample Name: 2-nai-ee

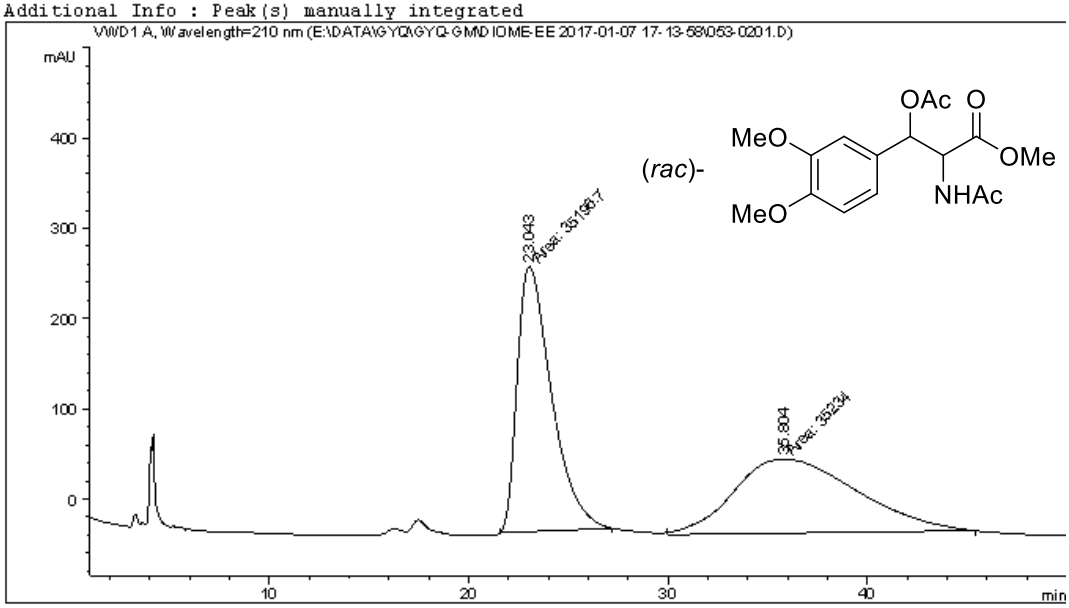
```
    ==================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :  15
    Acq. Instrument : 1260HPLC-DAD                   Location : Vial 5
    Injection Date  : 11/3/2016 4:10:27 PM                Inj :   1
                                                   Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\GYQ\GYQ-GM\2-NAI-DAD-ODH 2016-11-02 21-45-56\DAD-OD(1-2)-95-5-
                      1ML-75MIN-210-254.M
    Last changed    : 11/3/2016 5:15:06 PM by SYSTEM
                      (modified after loading)
    Analysis Method : E:\DATA\GYQ\GYQ-GM\2-NAI-DAD-ODH 2016-11-02 21-45-56\DAD-OD(1-2)-95-5-
                      1ML-75MIN-210-254.M (Sequence Method)
    Last changed    : 6/10/2017 10:04:50 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 B, Sig=210,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  31.541 BB    1.6780 5.03139e4   352.76291 100.0000

    Totals :                  5.03139e4   352.76291


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

Data File E:\DATA\GYQ\GYQ-GM\DIOME-EE 2017-01-07 17-13-58\053-0201.D
Sample Name: GYQ-DiOMe-rac

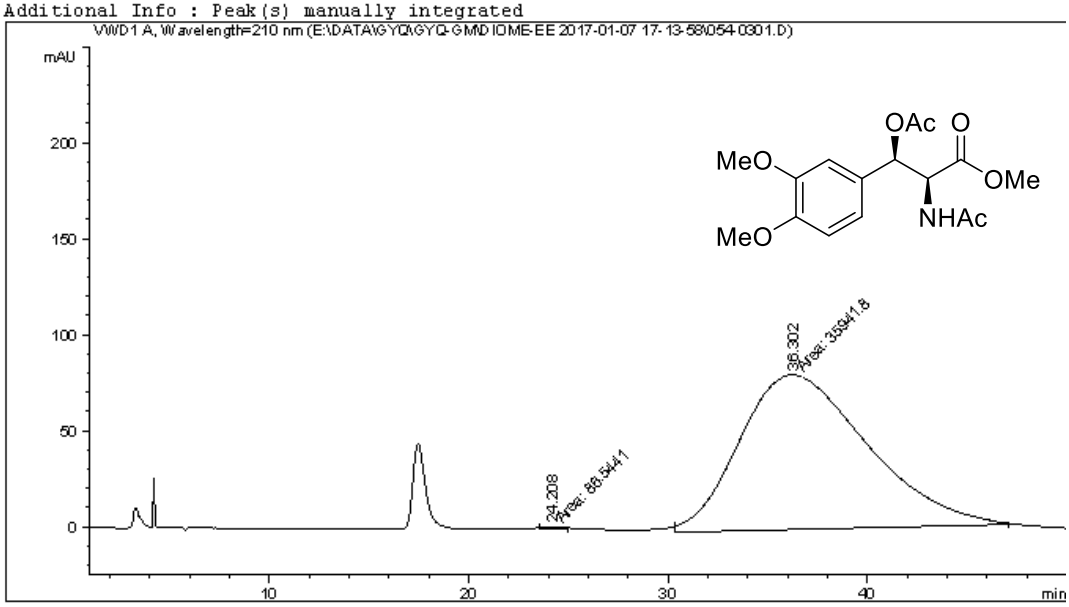
```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   2
    Acq. Instrument : 1260HPLC-VWD                   Location : Vial 53
    Injection Date  : 1/7/2017 5:30:36 PM                 Inj :   1
                                                   Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\GYQ\GYQ-GM\DIOME-EE 2017-01-07 17-13-58\VWD-0J(1-6)-80-20-1.0ML-
                      210NM-50MIN.M
    Last changed    : 1/7/2017 5:13:58 PM by SYSTEM
    Analysis Method : E:\DATA\GYQ\GYQ-GM\DIOME-EE 2017-01-07 17-13-58\VWD-0J(1-6)-80-20-1.0ML-
                      210NM-50MIN.M (Sequence Method)
    Last changed    : 6/10/2017 10:06:45 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    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  23.043 MM    1.9981 3.51967e4   293.58615  49.9735
       2  35.804 MM    7.1639 3.52340e4    81.97152  50.0265

    Totals :                  7.04308e4   375.55767


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

Data File E:\DATA\GYQ\GYQ-GM\DIOME-EE 2017-01-07 17-13-58\054-0301.D
Sample Name: GYQ-DiOMe-ee

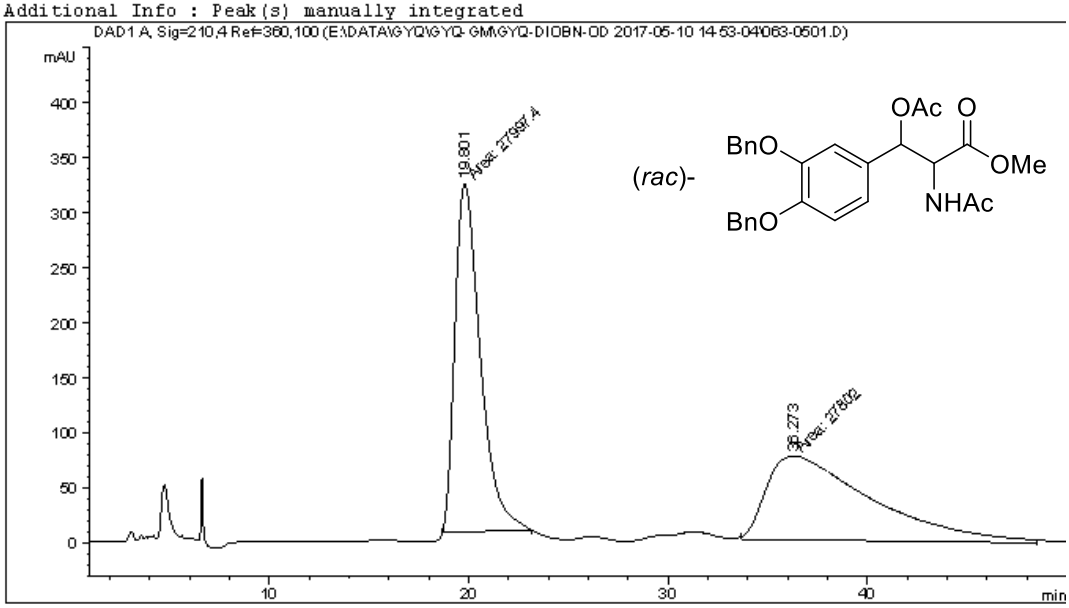
```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   3
    Acq. Instrument : 1260HPLC-VWD                   Location : Vial 54
    Injection Date  : 1/7/2017 6:21:24 PM                 Inj :   1
                                                  Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\GYQ\GYQ-GM\DIOME-EE 2017-01-07 17-13-58\VWD-0J(1-6)-80-20-1.0ML-
                      210NM-50MIN.M
    Last changed    : 1/7/2017 5:13:58 PM by SYSTEM
    Analysis Method : E:\DATA\GYQ\GYQ-GM\DIOME-EE 2017-01-07 17-13-58\VWD-0J(1-6)-80-20-1.0ML-
                      210NM-50MIN.M (Sequence Method)
    Last changed    : 6/10/2017 10:11:50 PM by SYSTEM
    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  24.208 MM    1.1890   86.54407    1.21313   0.2402
       2  36.302 MM    7.4595 3.59418e4    80.30405  99.7598

    Totals :                  3.60284e4    81.51717


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

Data File E:\DATA\GYQ\GYQ-GM\GYQ-DIOBN-OD 2017-05-10 14-53-04\063-0501.D
Sample Name: GYQ-DIOBN-RAC

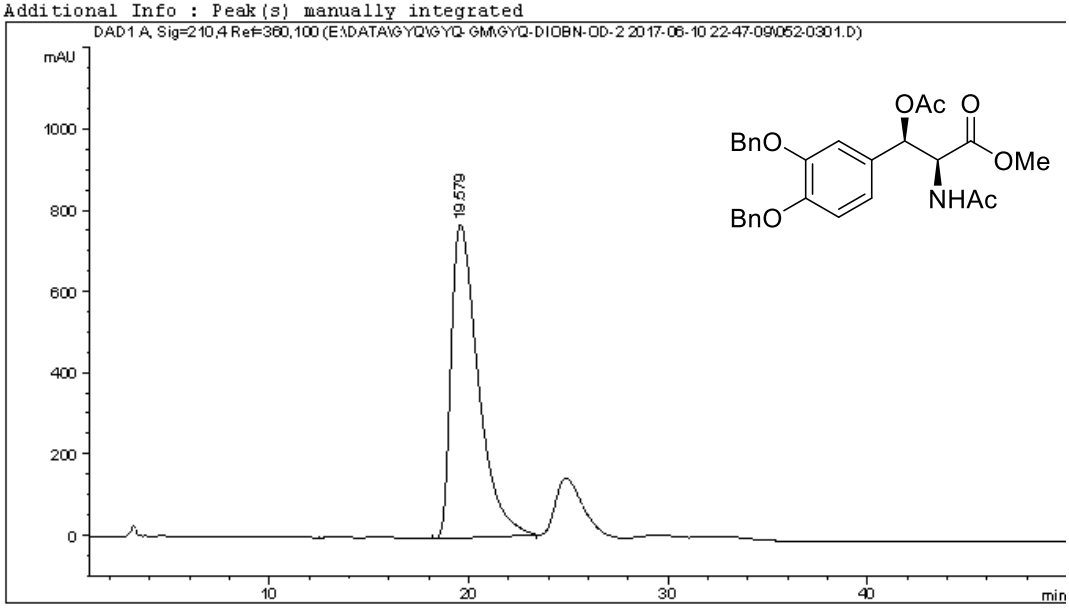
```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   5
    Acq. Instrument : 1260HPLC-DAD                   Location : Vial 63
    Injection Date  : 5/10/2017 5:47:55 PM               Inj :   1
                                                   Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\GYQ\GYQ-GM\GYQ-DIOBN-OD 2017-05-10 14-53-04\DAD-OD(1-2)-80-20-
                      1ML-5UL- ALL-65MIN.M
    Last changed    : 5/10/2017 5:08:19 PM by SYSTEM
    Analysis Method : E:\DATA\GYQ\GYQ-GM\GYQ-DIOBN-OD 2017-05-10 14-53-04\DAD-OD(1-2)-80-20-
                      1ML-5UL- ALL-65MIN.M (Sequence Method)
    Last changed    : 6/11/2017 8:08:23 AM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

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


    Signal 1: DAD1 A, Sig=210,4 Ref=360,100

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  19.801 MM    1.4744 2.79974e4   316.48984  50.1751
       2  36.273 MM    6.0796 2.78020e4    76.21697  49.8249

    Totals :                  5.57993e4   392.70681


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

1260HPLC-DAD 6/11/2017 8:08:38 AM SYSTEM                       Page 1 of 1
```

Data File E:\DATA\GYQ\GYQ-GM\GYQ-DIOBN-OD-2 2017-06-10 22-47-09\052-0301.D
Sample Name: GYQ-DIOBN-EE

```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   3
    Acq. Instrument : 1260HPLC-DAD                   Location : Vial 52
    Injection Date  : 6/11/2017 12:04:17 AM             Inj :   1
                                                   Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\GYQ\GYQ-GM\GYQ-DIOBN-OD-2 2017-06-10 22-47-09\DAD-OD(1-2)-80-20-
                      1ML-5UL- ALL-55MIN.M
    Last changed    : 6/10/2017 10:47:10 PM by SYSTEM
    Analysis Method : E:\DATA\GYQ\GYQ-GM\GYQ-DIOBN-OD-2 2017-06-10 22-47-09\DAD-OD(1-2)-80-20-
                      1ML-5UL- ALL-55MIN.M (Sequence Method)
    Last changed    : 6/11/2017 8:03:37 AM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

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


    Signal 1: DAD1 A, Sig=210,4 Ref=360,100

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  19.579 BV    1.1123 7.29634e4   770.80939 100.0000

    Totals :                  7.29634e4   770.80939


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

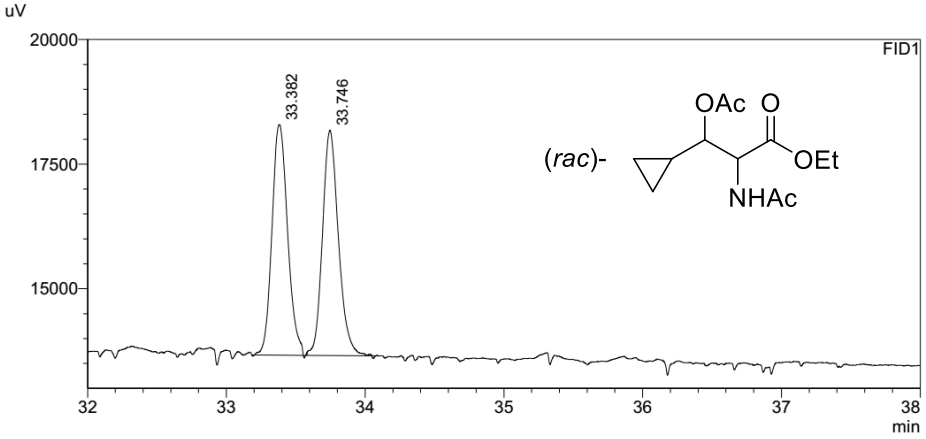
SHIMADZU LabSolutions

Analysis Report

<Sample Information>

Sample Name	: gyq-cyclo-rac		
Sample ID	: L/RhRu,2/2,0.15,i-Pr/MeOH		
Data Filename	: gyq-cyclo-rac-1.gcd		
Method Filename	: gamma dex-225-250-100(0)-2-160(20)-260-50min.gcm		
Batch Filename	: gyq-cyclo.gcb		
Vial #	: 1	Sample Type	: Unknown
Injection Volume	: 1 uL		
Date Acquired	: 2016-11-1 16:55:03	Acquired by	: System Administrator
Date Processed	: 2016-11-1 19:32:58	Processed by	: System Administrator

<Chromatogram>



<Peak Table>

FID1

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	33.382	36228	4620	50.079		M	
2	33.746	36114	4519	49.921		V M	
Total		72342	9139				

D:\DATA FILE\gyq\data\gyq-cyclo\gyq-cyclo-rac-1.gcd

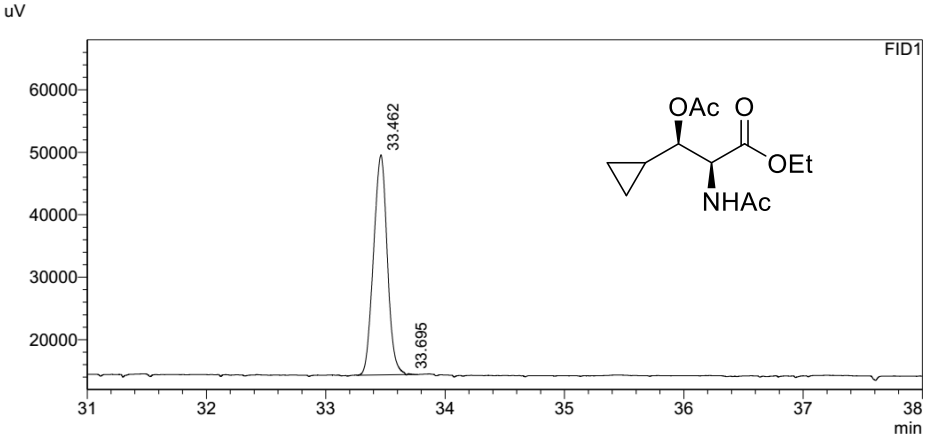
SHIMADZU LabSolutions

Analysis Report

<Sample Information>

Sample Name	: gyq-cyclo-ee		
Sample ID	: L/Rh,2/2,0.15,i-Pr/TFE		
Data Filename	: gyq-cyclo-ee.gcd		
Method Filename	: gamma dex-225-250-100(0)-2-160(20)-260-50min.gcm		
Batch Filename	: gyq-cyclo-ee.gcb		
Vial #	: 2	Sample Type	: Unknown
Injection Volume	: 1 uL		
Date Acquired	: 2016-11-2 16:47:55	Acquired by	: System Administrator
Date Processed	: 2016-11-3 11:26:04	Processed by	: System Administrator

<Chromatogram>



<Peak Table>

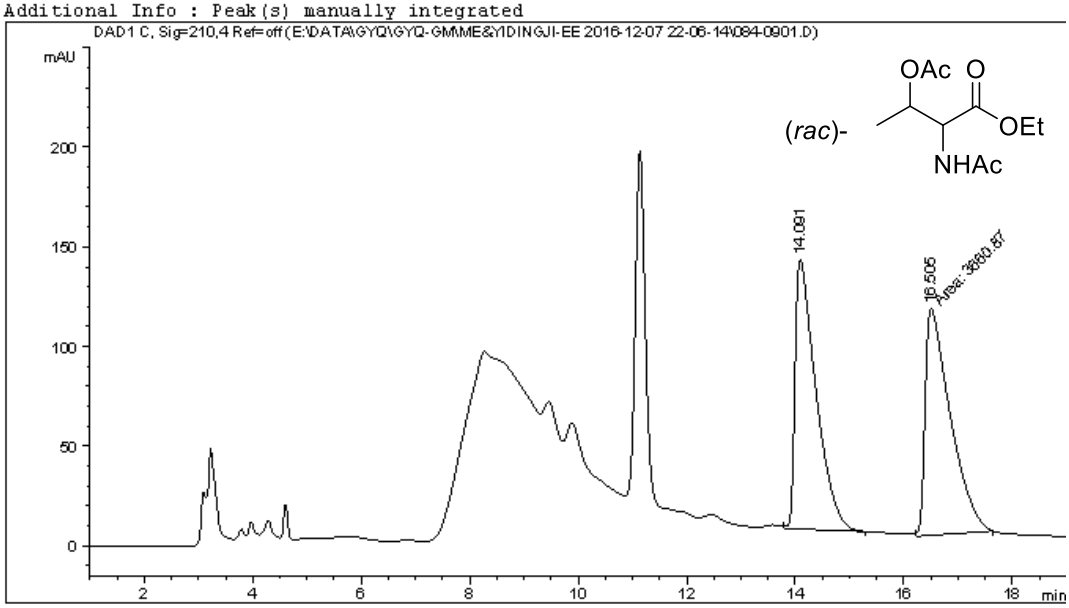
FID1

Peak#	Ret. Time	Area	Height	Conc.	Unit	Mark	Name
1	33.462	282372	35203	99.940		M	
2	33.695	170	125	0.060		M	
Total		282542	35327				

D:\DATA FILE\gyq\data\gyq-cyclo\gyq-cyclo-ee.gcd

Data File E:\DATA\GYQ\GYQ-GM\ME&YIDINGJI-EE 2016-12-07 22-06-14\084-0901.D
Sample Name: Me-rac

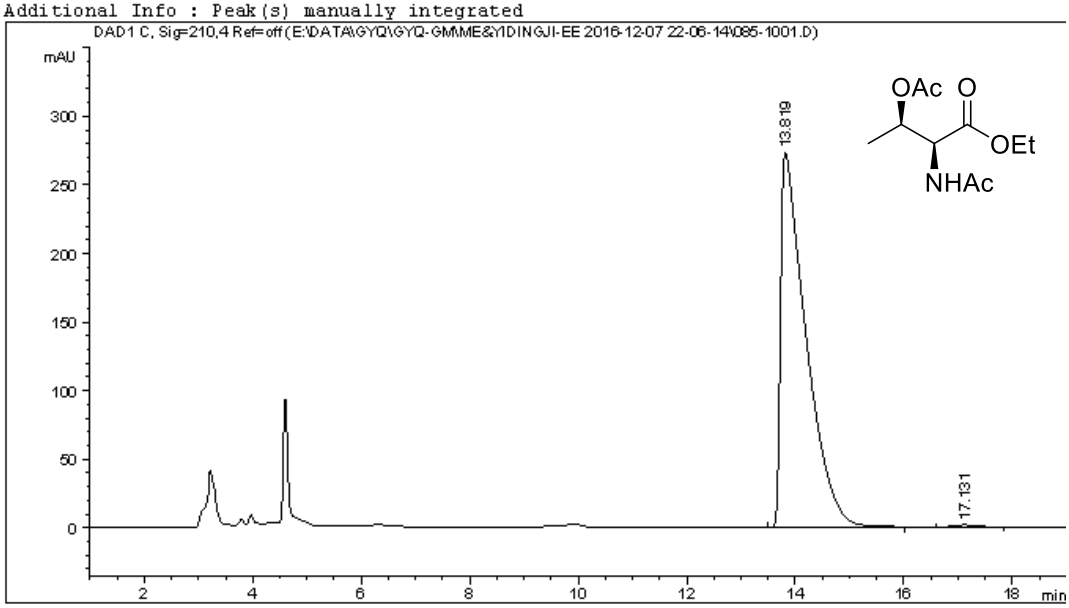
```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   9
    Acq. Instrument : 1260HPLC-DAD                   Location : Vial 84
    Injection Date  : 12/8/2016 2:16:41 AM                Inj :   1
                                                  Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\GYQ\GYQ-GM\ME&YIDINGJI-EE 2016-12-07 22-06-14\DAD-0J(1-6)-95-5-1
                      .OML-5-210NM-50MIN.M
    Last changed    : 12/7/2016 10:06:16 PM by SYSTEM
    Analysis Method : E:\DATA\GYQ\GYQ-GM\ME&YIDINGJI-EE 2016-12-07 22-06-14\DAD-0J(1-6)-95-5-1
                      .OML-5-210NM-50MIN.M (Sequence Method)
    Last changed    : 6/10/2017 9:41:25 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  14.091 VB    0.3868 3606.98047  135.41585  49.6293
       2  16.505 MM    0.5364 3660.87134  113.74592  50.3707

    Totals :                  7267.85181  249.16177


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

Data File E:\DATA\GYQ\GYQ-GM\ME&YIDINGJI-EE 2016-12-07 22-06-14\085-1001.D
Sample Name: Me-EE

```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :  10
    Acq. Instrument : 1260HPLC-DAD                   Location : Vial 85
    Injection Date  : 12/8/2016 3:07:45 AM                Inj :   1
                                                  Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\GYQ\GYQ-GM\ME&YIDINGJI-EE 2016-12-07 22-06-14\DAD-0J(1-6)-95-5-1
                      .OML-5-210NM-50MIN.M
    Last changed    : 12/7/2016 10:06:16 PM by SYSTEM
    Analysis Method : E:\DATA\GYQ\GYQ-GM\ME&YIDINGJI-EE 2016-12-07 22-06-14\DAD-0J(1-6)-95-5-1
                      .OML-5-210NM-50MIN.M (Sequence Method)
    Last changed    : 6/10/2017 9:43:20 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    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  13.819 BB    0.4619 8835.72461  272.76071  99.5346
       2  17.131 BB    0.3341   41.31002    1.49612   0.4654

    Totals :                  8877.03463  274.25683


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