Supporting Information for:

Highly Enantioselective Synthesis of Non-natural Aliphatic α-Amino Acids via Asymmetric Hydrogenation

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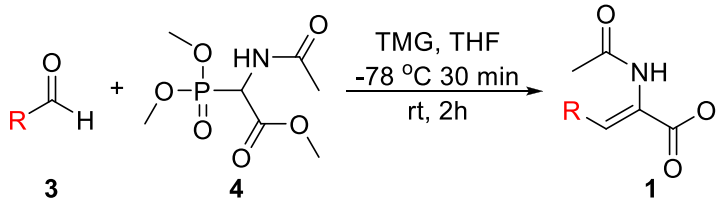
Contents

Experimental Section

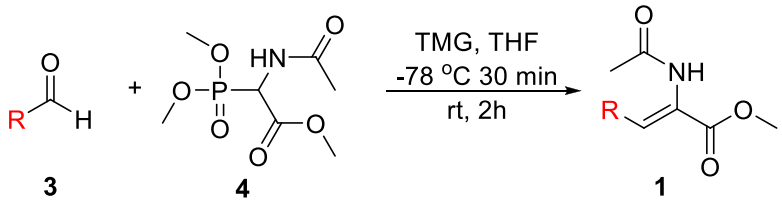
General Information Unless otherwise noted, all reagents and solvents were purchased from commercial suppliers and used without further purification. NMR spectra were recorded on Bruker Advance III (400 MHz) spectrometers for ^{1}H NMR and ^{13}C NMR. $CDCl_3$ was used as the solvent for the NMR analysis, 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.

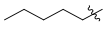
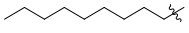
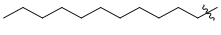
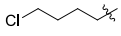
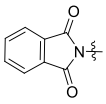
General procedure for the synthesis of *β*-alkyl (*Z*)-N-acetyldehydroamino esters



Tetramethylguanidine (13.5 mmol) was added to a solution of the phosphonate **4** (10 mmol) in distilled THF (35 mL) at -78℃. After 15 min, aldehyde **3** (12 mmol) was added and the resulting solution was stirred at -78 ℃ for 2 h then allowed to warm to 25 ℃ over a period of 2 h. The mixture was quenched with water and extracted with EA (3×20 mL). The organic layer was combined and dried with $MgSO_4$ and concentrated under reduced pressure to give an oil. Purification by flash chromatography on silica gel using ethyl acetate and petroleum ether (1:1) gave the (Z)-esters **1** as a white solid.[1] Results of the preparation of **1** were summarized in Table 1.

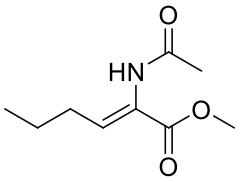
Table 1. Preparation of β-alkyl (Z)-N-acetyldihydroamino esters [a]



Entry	R	Product	Yield (%)[b]
1		**1a**	88
2		**1b**	90
3		**1c**	85
4		**1d**	82
5		**1e**	97
6		**1f**	93
7		**1g**	85
8		**1h**	50
9		**1i**	82
10		**1j**	80
11		**1k**	57

[a] Unless otherwise mentioned, all reactions were carried out in THF at atemperature from -78 ℃ to rt for 2 h, all the product was determined as Z-configuration; [b] isolated yield.

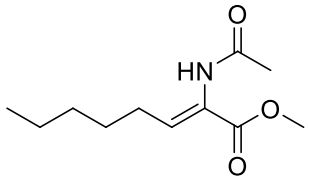
(Z)-methyl 2-acetamidohex-2-enoate, 1a



1a

White solid; Yield: 80 %;^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (br, 1H), 6.70 - 6.68 (m, 1H), 3.76 (s, 3H), 2.16 – 2.10 (m, 5H), 1.52 – 1.43 (m, 2H), 0.95 – 0.91 (m, 3H); ^{13}C NMR(101 MHz, $CDCl_3$) δ 168.98, 165.05, 139.04, 125.17, 52.03, 30.35, 22.85, 21.24, 13.68. ESI-HRMS Calculated for $C_9H_{15}NO_3^+$([M+H]$^+$): 86.1125, found: 86.1127.

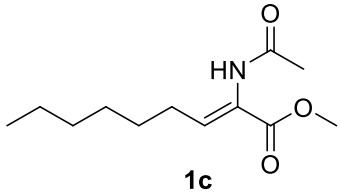
(*Z*)-methyl 2-acetamidooct-2-enoate, 1b



1b

White solid; Yield: 90%; ^{1}H NMR(400 MHz, $CDCl_3$)δ 7.71 (s, 1H), 6.70 – 6.66 (m, 1H), 3.75 (s, 3H), 3.71 – 3.66 (m, 1H), 2.15 – 2.10 (m, 5H), 1.46 – 1.43 (m, 2H), 1.31 - 1.19 (m, 6H), 0.90 – 0.87 (m, 3H); ^{13}C NMR(101MHz, $CDCl_3$) δ 168.97, 165.03, 139.37, 125.00, 57.71, 51.97, 51.87, 31.29, 28.33, 27.62, 22.77, 22.17, 18.01, 13.67. ESI-HRMS Calculated for $C_{11}H_{20}NO_3^+$([M+H]$^+$): 214.1438, found: 214.1442.

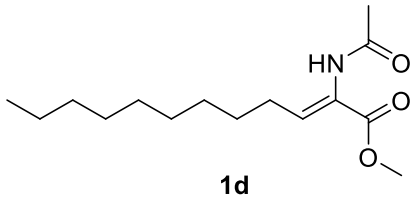
(Z)-methyl 2-acetamidonon-2-enoate, 1c



1c

White solid; Yield: 84%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.89 (br, 1H), 6.73 – 6.69 (m, 1H), 3.77 (s, 3H), 2.18 – 2.13 (m, 5H), 1.52 - 1.37 (m, 2H), 1.36 - 1.16 (m, 6H), 0.90 – 0.86 (m, 3H).; ^{13}C NMR (101 MHz, $CDCl_3$)δ168.35, 165.19, 139.46, 124.56, 52.33, 31.56, 29.07, 29.00, 28.14, 23.41, 22.51, 14.02. ESI-HRMS Calculated for $C_{11}H_{22}NO_3^+$([M+H]$^+$): 228.1594, found: 228.1595.

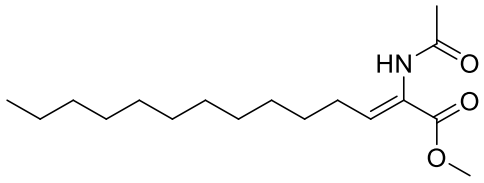
(Z)-methyl 2-acetamidododec-2-enoate, 1d



1d

White solid; Yield: 93 %;^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70 (br, 1H), 6.69 – 6.67 (m, 1H), 3.76 (s, 3H), 2.99 – 2.97 (m, 2H), 2.12 – 2.11 (m, 5H), 1.45 – 1.43(m, 2H), 1.31 – 1.21 (m, 9H), 0.89 – 0.87 (m, 3H); ^{13}C NMR(101 MHz, $CDCl_3$) δ 168.88, 165.21, 139.42, 125.00, 52.13, 39.52, 31.70, 29.27, 29.13, 28.58, 28.07, 24.66, 23.01, 22.50, 13.95. ESI-HRMS Calculated for $C_{15}H_{27}NO_3^+$([M+H]$^+$): 270.2064, found: 270.2067.

(Z)-methyl 2-acetamidotetradec-2-enoate, 1e

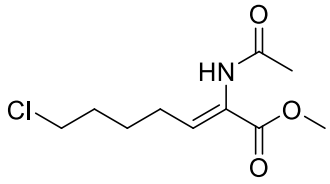


1e

White solid; Yield: 97 %;^{1}H NMR (400 MHz, $CDCl_3$) δ 7.15 (br, 1H), 6.72 – 6.68 (m, 1H), 3.77 (s, 3H), 2.17 – 2.12 (m, 5H), 1.46 – 1.43 (m, 2H), 1.33 – 1.21 (m, 17H), 0.90 – 0.86 (m, 3H); ^{13}C NMR(101

MHz, $CDCl_3$) δ 168.51, 165.19, 139.48, 124.66, 52.27, 39.61, 31.83, 29.54, 29.47, 29.37, 29.36, 29.26, 28.88, 28.16, 23.29, 22.61, 14.05. ESI-HRMS Calculated for $C_{17}H_{31}NO_3^+$([M+H]$^+$):298.2377, found: 298.2367.

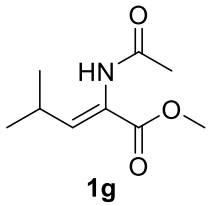
(Z)-methyl 2-acetamido-7-chlorohept-2-enoate, 1f



1f

White solid; Yield: 82 %;^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 (br, 1H), 6.69 – 6.65 (m, 1H), 3.77 (s, 3H), 3.56 – 3.53 (m, 2H), 2.22 – 2.12 (m, 5H), 1.82 – 1.78 (m, 2H), 1.64 – 1.60 (m, 2H); ^{13}C NMR(101 MHz, $CDCl_3$) δ168.56, 164.95, 137.95, 125.12, 52.29, 44.54, 31.98, 27.95, 25.29, 23.21.. ESI-HRMS Calculated for $C_{10}H_{16}ClNO_3^+$([M+H]$^+$):234.0891, found: 234.0897.

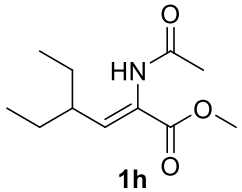
(Z)-methyl 2-acetamido-4-methylpent-2-enoate, 1g



1g

White solid; Yield: 87%; ^{1}H NMR (400 MHz, $CDCl_3$) δ7.10 (br, 1H), 6.54 – 6.51 (m, 1H), 3.76 (s, 3H), 2.62 – 2.58 (m, 1H), 2.12 (s, 3H), 1.07 -1.02 (m, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ169.17, 165.39, 145.87, 123.03, 52.30, 27.96, 23.16, 21.51.ESI-HRMS Calculated for $C_{11}H_{20}NO_3^+$ ([M+H]$^+$): 186.1125, found: 186.1127.

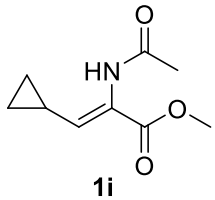
(Z)-methyl 2-acetamido-4-ethylhex-2-enoate, 1h



1h

White solid; Yield: 87%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.81 (br, 1H), 6.49 - 6.46 (m, 1H), 3.81 (s, 3H), 2.26 – 2.23 (m, 1H), 2.11 (s, 3H), 1.53 – 1.50 (m, 2H), 1.38 – 1.31 (m, 2H), 0.88 – 0.84 (m, 6H); ^{13}C NMR (101 MHz, $CDCl_3$)δ169.11, 165.15, 143.85, 125.75, 52.28, 41.42, 26.65, 23.14, 11.73, 11.60.ESI-HRMS Calculated for $C_{11}H_{20}NO_3^+$([M+H]$^+$): 214.1438, found: 214.1442.

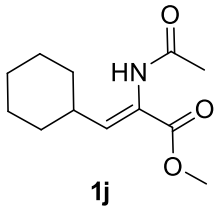
(Z)-methyl 2-acetamido-3-cyclopropylacrylate, 1i



1i

White solid; Yield: 85%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.96 (br, 1H), 6.15 – 6.14 (m, 1H), 3.75 (s, 3H), 2.15 (s, 3H), 1.61 – 1.59 (m, 1H), 1.08 – 1.02 (m, 2H), 0.70 – 0.68 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ168.73, 164.98, 144.82, 123.14, 52.20, 23.41, 12.21, 8.84.ESI-HRMS Calculated for $C_{11}H_{20}NO_3^+$([M+H]$^+$): 184.0968, found: 184.0976.

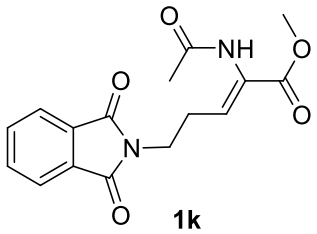
(Z)-methyl 2-acetamido-3-cyclohexylacrylate, 1j



1j

White solid; Yield: 85%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.84 (br, 1H), 6.55 - 6.53 (m, 1H), 3.76 (s, 3H), 2.31 – 2.29 (m, 1H), 2.13 (s, 3H), 1.82 - 1.65 (m, 5H), 1.30 – 1.12 (m, 5H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.28, 165.44, 144.33, 123.44, 52.30, 37.58, 37.39, 32.96, 31.36, 25.75, 25.31, 23.16. ESI-HRMS Calculated for $C_{12}H_{20}NO_3^+$ ([M+H]$^+$): 226.1438, found: 226.1436.

(Z)-methyl 2-acetamido-5-(1,3-dioxoisoindolin-2-yl)pent-2-enoate, 1k



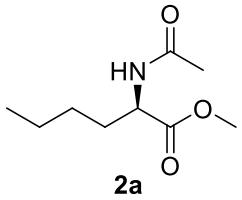
1k

Light yellow solid; Yield: 57 %;^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 -7.85(m, 2H), 7.75 – 7.32(m, 2H), 7.27 (br, 1H), 6.69 – 6.65 (m, 1H), 3.85 – 3.80(m, 2H), 3.76 (s, 3H), 2.60 – 2.55 (m, 2H), 2.11 (s, 3H); ^{13}C NMR(101 MHz, $CDCl_3$) δ168.39, 168.10, 164.71, 133.94, 132.80, 131.83, 126.76, 123.17, 52.46, 36.20, 28.30, 23.28. ESI-HRMS Calculated for $C_6H_{16}N_2O_5^+$([M+H]$^+$):317.1132, found: 317.1132.

General Procedure for Asymmetric Hydrogenation of compound 1

In a nitrogen-filled glove box, $[Rh(NBD)_2]BF_4$ (0.01 mmol) and Duanphos (0.011 mmol) were dissolved in MeOH (1 mL) and stirred for 30 min. 0.1 mL of the resulting solution was transferred by syringe into the vials charged with different substrates (0.1 mmol for each). Additional MeOH was added to bring the total reaction volume to 1 mL. The vials were subsequently transferred into an autoclave which was charged with hydrogen (100 psi). The reaction was then stirred at rt for 2 h. The hydrogen gas was released slowly and carefully. The solution was passed through a short column of silica gel (eluent: EtOAc) to remove the metal complex and concentrated to give compounds **2**. The *ee* values of compounds **2** were then determined by HPLC analysis on a chiral stationary phase.

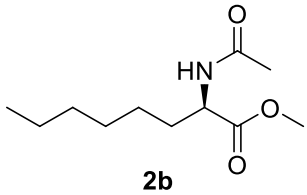
(*R*)-methyl 2-acetamidohexanoate, 2a



2a

White solid; 99.0% *ee*; $[\alpha]_D^{20}$ = -26.60 (c = 0.5, CH_2Cl_2); The enantiomeric excess was determined by HPLC on Chiralpak AD-H column, hexane: isopropanol = 90:10; flow rate = 1.0 mL/min; UV detection at 205 nm; t_R = 5.5 min (major), 7.2 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.16 (br, 1H), 4.63 – 4.58 (m, 1H), 3.75 (s, 3H), 2.03 (s, 3H), 1.83 – 1.80 (m, 1H), 1.67 – 1.65 (m, 1H), 1.33 - 1.26 (m, 4H), 0.91 – 0.88 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ173.27, 169.79, 52.26, 52.07, 32.14, 27.25, 23.09

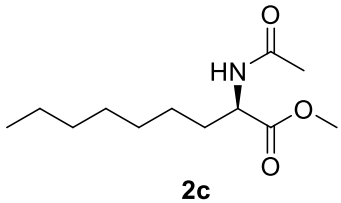
(*R*)-methyl 2-acetamidooctanoate, 2b



2b

White solid; 99.2% *ee*; $[\alpha]_D^{20}$= -29.80 (c = 0.5, CH_2Cl_2); The enantiomeric excess was determined by HPLC on Chiralpak AD-H column, hexane: isopropanol = 90:10; flow rate = 1.0 mL/min; UV detection at 205 nm; t_R = 5.4 min (major), 6.7 min (minor).^{1}H NMR(400 MHz, $CDCl_3$) δ6.12 (br, 1H), 4.63 – 4.58 (m, 1H), 3.75(s, 3H), 2.03(s, 3H), 1.84- 1.79(m, 1H), 1.69 – 1.63(m,1H), 1.29 – 1.26(m, 8H), 0.89 – 0.86(m, 3H); ^{13}C NMR(101MHz, $CDCl_3$) δ173.28, 169.77, 52.28, 52.09, 32.44, 31.51, 28.81, 25.09, 23.13, 22.47, 13.98. ESI-HRMS Calculated for $C_{11}H_{21}NO_3^+$([M+H]$^+$): 216.1600, found: 216.1589

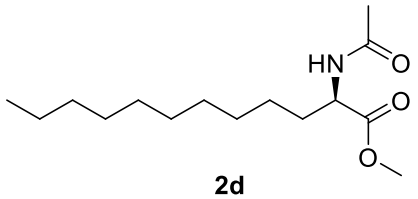
(*R*)-methyl 2-acetamidononanoate, 2c



2c

White solid; 99.5% *ee*; $[\alpha]_D^{20}$ = -26.00 (c = 0.5, CH_2Cl_2); The enantiomeric excess was determined by HPLC on Chiralpak AD-H column, hexane: isopropanol = 90:10; flow rate = 1.0 mL/min; UV detection at 205 nm; t_R = 5.2 min (major), 6.2 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.09 (br, 1H), 4.63 – 4.58 (m, 1H), 3.75 (s, 3H), 2.03 (s, 3H), 1.84 – 1.79 (m, 1H), 1.68 – 1.64 (m, 1H), 1.38 – 1.19 (m, 10H), 0.89 – 0.86(m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 173.28, 169.73, 52.27, 52.11, 32.46, 31.66, 29.11, 29.00, 25.14, 23.14, 22.55, 14.02. ESI-HRMS Calculated for $C_{12}H_{23}NO_3^+$ ([M+H]$^+$): 230.1756, found: 230.1744.

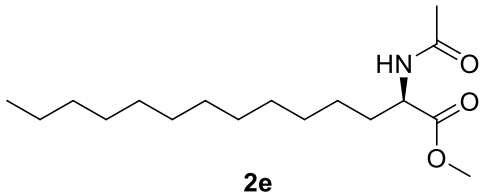
(*R*)-methyl 2-acetamidododecanoate, 2d



2d

White solid; 99.2% *ee*; $[\alpha]_D^{20}$ = -49.40 (c = 0.5, CH_2Cl_2); The enantiomeric excess was determined by HPLC on Chiralpak AD-H column, hexane: isopropanol = 95:5; flow rate = 1.0 mL/min; UV detection at 205 nm; t_R = 6.9 min (major), 8.2 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.38 (br, 1H), 4.61 – 4.59 (m, 1H), 3.74 (s, 3H), 2.03 (s, 3H), 1.84 – 1.78 (m, 1H), 1.68 – 1.63 (m, 1H), 1.30 – 1.17 (m, 16H), 0.89 – 0.86 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 173.29, 169.85, 77.32, 77.00, 76.68, 52.16, 52.05, 32.30, 31.77, 29.44, 29.40, 29.27, 29.19, 29.09, 25.13, 22.96, 22.55, 13.99. ESI-HRMS Calculated for $C_{15}H_{29}NO_3^+$ ($[M+H]^+$): 272.2226, found 272.2213.

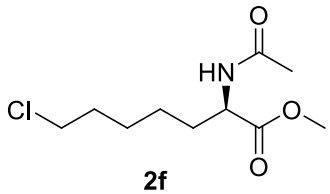
(*R*)-methyl 2-acetamidotetradecanoate, 2e



2e

White solid; 99.5% *ee*; $[\alpha]_D^{20}$ = -36.60 (c = 0.5, CH_2Cl_2); The enantiomeric excess was determined by HPLC on Chiralpak AD-H column, hexane: isopropanol = 90:10; flow rate = 1.0 mL/min; UV detection at 205 nm; t_R = 4.5 min (major), 5.0 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.29 (br, 1H),4.63 - 4.58 (m, 1H), 3.74 (s, 3H), 2.03 (s, 3H), 1.84 - 1.79 (d, *J* = 8.3 Hz, 1H), 1.70 - 1.61 (s, 1H), 1.32 – 1.25 (m, 20H), 0.90 - 0.86 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ173.29, 169.81, 52.20, 52.07, 32.35, 31.82, 29.56, 29.54, 29.52, 29.44, 29.30, 29.26, 29.12, 25.14, 23.02, 22.60, 14.03. ESI-HRMS Calculated for $C_{11}H_{15}N_2O_3^+$ ($[M+H]^+$):300.2539, found: 300.2526.

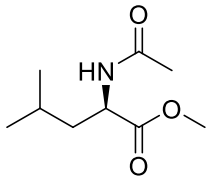
(*R*)-methyl 2-acetamido-7-chloroheptanoate, 2f



2f

White solid; 99.7% *ee*; $[\alpha]_D^{20}$ = -64.80 (c = 0.5, CH_2Cl_2); The enantiomeric excess was determined by HPLC on Chiralpak AD-H column, hexane: isopropanol = 90:10; flow rate = 1.0 mL/min; UV detection at 205 nm; t_R = 8.7 min (major), 10.0 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.27 (br, 1H), 6.64 – 4.59 (m, 1H), 3.75 (s, 3H), 3.54 - 3.51 (m, 2H), 2.03 (s, 3H), 1.85 – 1.66 (m, 4H), 1.48 – 1.32 (m, 4H); ^{13}C NMR (101 MHz, $CDCl_3$) δ173.03, 169.86, 52.31, 51.90, 44.76, 32.25, 32.16, 26.32, 24.47, 23.04. ESI-HRMS Calculated for $C_{10}H_{18}ClNO_3^+$ ($[M+H]^+$): 236.1053, found: 236.1042.

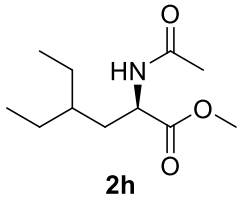
(*R*)-methyl 2-acetamido-4-methylpentanoate, 2g



2g

White solid; 98.5% *ee*; $[\alpha]_D^{20}$ = -4.00 (c = 0.5, CH_2Cl_2); The enantiomeric excess was determined by HPLC on Chiralpak AD-H column, hexane: isopropanol = 90:10; flow rate = 1.0 mL/min; UV detection at 205 nm; t_R = 5.0 min (major), 6.7 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.10 (br, 1H), 4.65 – 4.63 (m, 1H), 3.74 (s, 3H), 2.02 (s, 3H), 1.67 – 1.60 (m, 2H), 1.55 – 1.50 (m, 1H), 0.95 – 0.93 (m, 6H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 173.75, 169.96, 52.23, 50.65, 41.57, 24.79, 23.04, 22.72, 21.89.. ESI-HRMS Calculated for $C_9H_{17}NO_3^+$([M+H]$^+$): 188.1287, found: 188.1275.

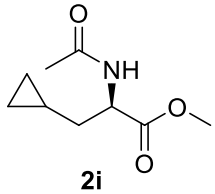
(*R*)-methyl 2-acetamido-4-ethylhexanoate, 2h



2h

White solid; 96.5% *ee*; $[\alpha]_D^{20}$ = -3.80 (c = 0.5, CH_2Cl_2); The enantiomeric excess was determined by HPLC on Chiralpak AD-H column, hexane: isopropanol = 90:10; flow rate = 1.0 mL/min; UV detection at 205 nm; t_R = 5.0 min (major), 7.2 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.07 (br, 1H), 4.66 – 4.61 (m, 1H), 3.74 (s, 3H), 2.02 (s, 3H), 1.70 – 1.64 (d, *J* = 6.7 Hz, 1H), 1.56 – 1.50 (m, 1H), 1.39 - 1.26 (m, 5H), 0.87 – 0.82 (m, 6H); ^{13}C NMR (101 MHz, $CDCl_3$)δ 173.89, 169.89, 52.21, 50.46, 36.61, 36.18, 25.28, 24.55, 23.06, 10.60, 10.10. ESI-HRMS Calculated for $C_{11}H_{21}NO_3^+$ ([M+H]$^+$): 216.1600, found: 216.1587.

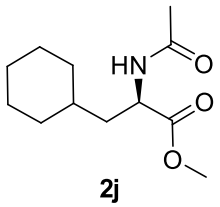
(R)-methyl 2-acetamido-3-cyclopropylpropanoate, 2i



2i

White solid; 99.7% *ee*; $[\alpha]_D^{20}$ = -19.4 (c = 0.5, CH_2Cl_2); The enantiomeric excess was determined by HPLC on Chiralpak AD-H column, hexane: isopropanol = 90:10; flow rate = 1.0 mL/min; UV detection at 205 nm; t_R = 6.3 min (major), 8.2 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.29 (s, 1H), 4.72 – 4.67 (m, 1H), 3.76 (s, 3H), 2.04 (s, 3H), 1.76 – 1.65 (m, 2H), 0.70 – 0.66 (m, 1H), 0.49 –0.46 (m, 2H), 0.08 – 0.06 (m, 2H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 173.06, 169.67, 52.51, 52.21, 37.00, 23.08, 6.78, 4.09, 3.97.. ESI-HRMS Calculated for $C_9H_{15}NO_3^+$([M+H]$^+$): 186.1130, found: 186.1118.

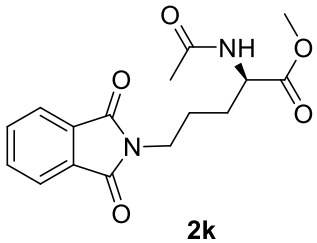
(*R*)-methyl 2-acetamido-3-cyclohexylpropanoate, 2j



2j

White solid; 99.5% *ee*; $[\alpha]_D^{20}$ = -2.80(c = 0.5, CH_2Cl_2); The enantiomeric excess was determined by HPLC on Chiralpak AD-H column, hexane: isopropanol = 90:10; flow rate = 1.0 mL/min; UV detection at 205 nm; t_R = 6.9 min (major), 10.7 min (minor). ^{1}H NMR(400 MHz, $CDCl_3$) δ 6.04 (br, 1H), 4.67 – 4.64 (m, 1H), 3.73 (s, 3H), 2.03 (s, 3H), 1.71 – 1.65 (m, 6H), 1.54 – 1.51 (m, 1H), 1.26 – 1.14 (m, 4H), 0.96 – 0.88 (m, 2H); ^{13}C NMR(101 MHz, $CDCl_3$) δ173.83, 169.87, 52.22, 50.04, 40.10, 34.03, 33.41, 32.49, 26.28, 26.10, 25.92, 23.09. ESI-HRMS Calculated for $C_{12}H_{21}NO_3^+$ ($[M+H]^+$):228.1600, found: 228.1584.

(*R*)-methyl 2-acetamido-7-chloroheptanoate, 2k

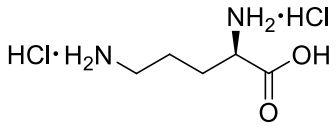


2k

White solid; 99.8% *ee*; $[\alpha]_D^{20}$ = -21.75 (c = 0.4, CH_2Cl_2); The enantiomeric excess was determined by HPLC on Chiralpak AD-H column, hexane: isopropanol = 80:20; flow rate = 1.0 mL/min; UV detection at 205 nm; t_R = 14.1 min (major), 22.3 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 – 7.83 (m, 2H),7.75 – 7.72 (m, 2H), 6.33 (br, 1H), 3.74 – 3.68 (m, 5H), 2.03 (s, 3H), 1.90 – 1.88 (m, 1H), 1.77 – 1.69 (m, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.68, 169.92, 168.28, 133.97, 131.86, 123.18, 52.43, 51.71, 37.32, 29.54, 24.64, 23.06.. ESI-HRMS Calculated for $C_{16}H_{18}N_2O_5^+$($[M+H]^+$): 319.1294, found: 319.1285.

Experimental Procedure for the Synthesis of (R)-piperidin-3-amine chloride 7k

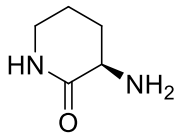
(*R*)-2,5-diaminopentanoic acid chloride, 5k



5k

6 M HCl (40 ml) was added to a stirred solution of (*R*)-methyl 2-acetamido-7-chloroheptanoate (2.8 g, 8.8 mmol). The reaction mixture was heated to reflux for 10 h. After the solution was cooled to room temperature, insoluble solid was filtered off, and washed with 6M HCl three times. The solvent was evaporated and dried in vacuum 24 h to give the product, 1.5 g ; Yield: 85%; $[\alpha]_D^{20}$ = -13.40 (c = 0.5, H_2O); ^{1}H NMR (400 MHz, D_2O) δ 3.95 – 3.92(t, *J*=8 Hz, 4 Hz, 1H), 2.93 – 2.90 (t, *J*=8 Hz, 4Hz, 2H), 1.91 – 1.83 (m, 2H), 1.76 – 1.64 (m, 2H); ^{13}C NMR(101 MHz, D_2O) δ 171.81, 52.43, 38.62, 26.78, 22.60

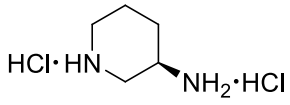
(*R*)-3-aminopiperidin-2-one[2], 6k



6k

(*R*)-2,5-diaminopentanoic acid chloride (10 g, 0.059 mol) was added to a stirred solution of sodium hydroxide pellets (2.38 g, 0.059 mol) in water (100 ml) at 25℃. After 15 min, this solution was added to a stirred mixture of alumina (30 g) and toluene (100 ml) and heated under reflux for 1.5 h. The water produced during the reaction was collected in a Dean-Stark trap. The reaction mixture was allowed to cool and the alumina was filtered off and washed with 10% MeOH/CH_2Cl_2(30 ml). The filtration was combined and the solvent was removed under vacuum to leave **6k,** 5.8 g, Yield: 86 %;$[\alpha]_D^{20}$ = -3.40 (c = 0.5, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20（br, 1H）3.29 – 3.23(m, 3H), 2.15– 2.14 (m, 1H), 1.87– 1.85 (m, 1H), 1.80– 1.72 (m, 3H), 1.58 – 1.49 (m, 1H); ^{13}C NMR (101 MHz, D_2O) δ 175.11, 51.21, 42.07, 29.53, 21.19

(*R*)-piperidin-3-amine chloride, 7k



7k

THF (27 ml) was added to lithium aluminum hydride (0.252 g, 6.64 mmol). To the resulting suspension, (*R*)-3-aminopiperidin-2-one (0.4 g, 2.66 mmol) was gradually added while ensuring the temperature range of 5 ℃ to 16 ℃. Ten minutes after the completion of the addition, the

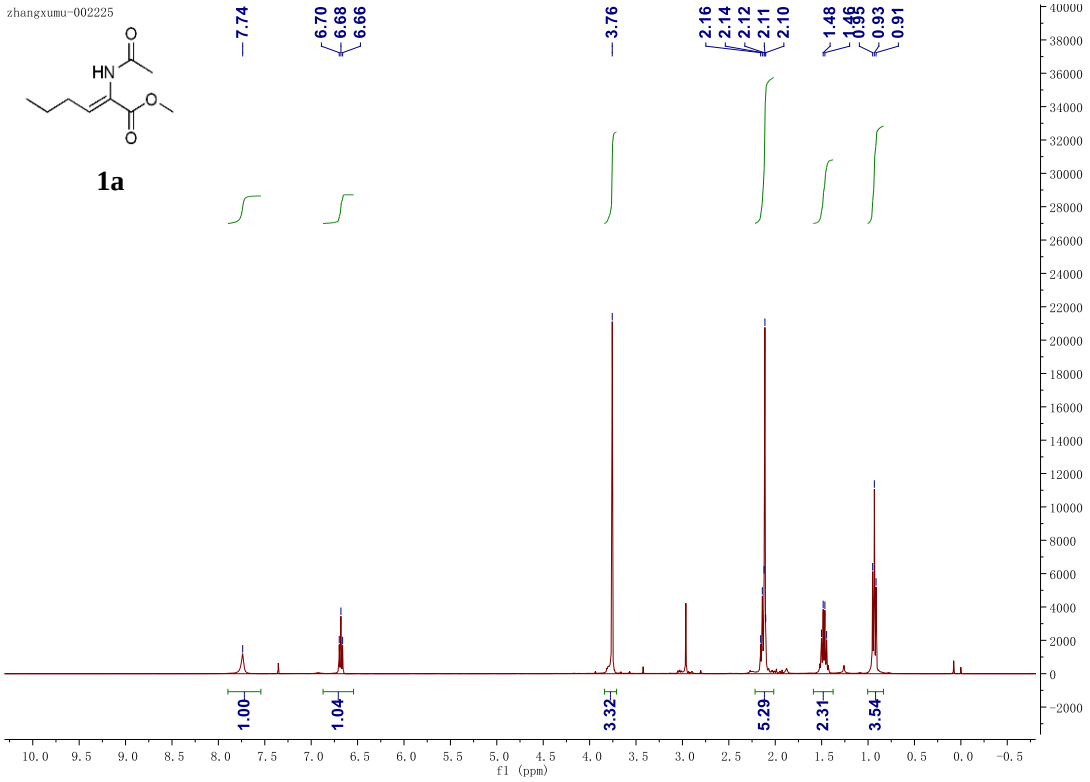
mixture was warmed to room temperature and further vigorously stirred for 3 h. Lithium aluminum hydride (20.2 mg, 0.532 mmol) was added to the mixture, and the mixture was stirred for 50 min. The mixture was ice cooled, and water (0.91 ml) was added to the cooled mixture. The mixture was warmed to room temperature and vigorously stirred for 1.5 h. The precipitated inorganic material was filtered through Celite and was then washed with THF. The filtration was combined and dried over anhydrous Na_2SO_4. Aqueous HCl (4 M) in AcOEt (1.33 ml, 5.3 mmol) was added to the mixture, and the solvent was removed by distillation under reduced pressure. The residue was subjected to azeotropic distillation with MeOH to give hydrochloride salt of **7K**, 0.26 g; Yield: 56 %;$[\alpha]_D^{20}$ = -0.80 (c = 0.5, CH_3OH); ^{1}H NMR (400 MHz, D_2O) δ 3.73(m, 2H), 3.47 – 3.44 (m, 1H), 3.20 – 3.17 (m, 1H), 3.05 – 3.04 (m, 1H), 2.30 – 2.28 (m, 1H), 2.14 – 2.10 (m, 1H), 1.97– 1.94 (m, 1H), 1.84– 1.82 (m, 1H); ^{13}C NMR(101 MHz, D_2O) δ 44.91, 44.55, 43.23, 26.28, 20.17.

References:

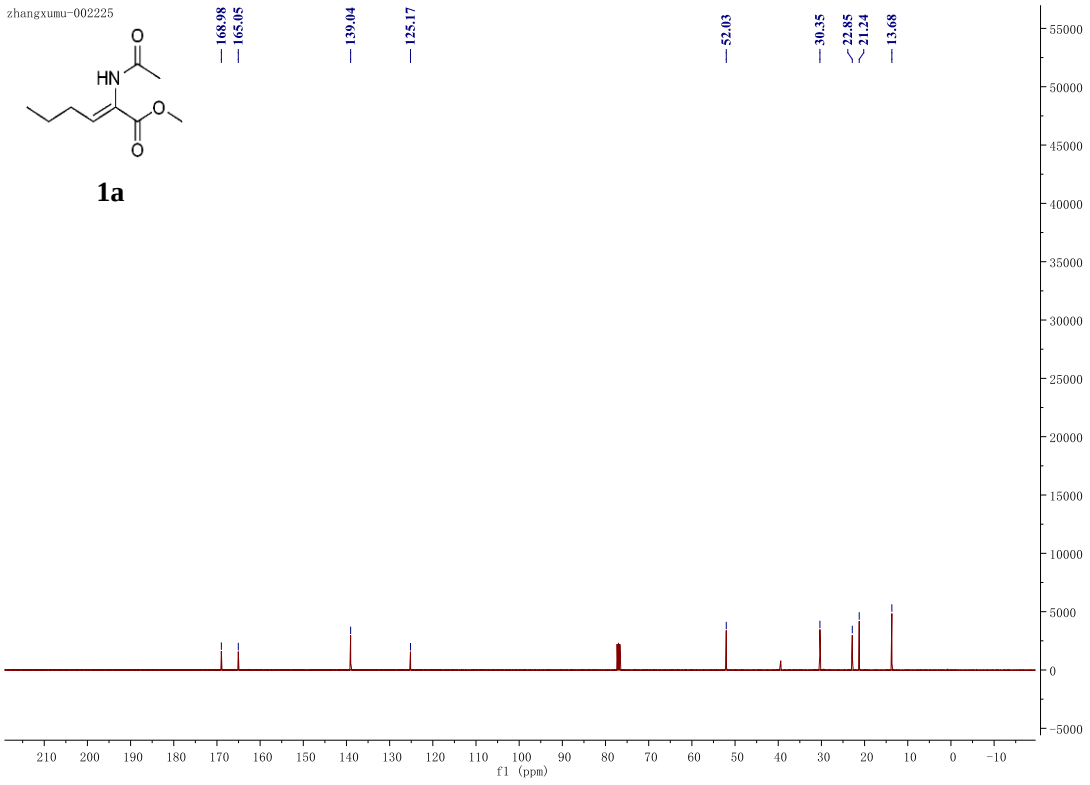
[1](a)H. Rodríguez-Solla, J. Concellón, C. Concellón, P. Tuya, *Synlett* **2008**, *2008*, 402-404;(b)E. Teoh, E. M. Campi, W. R. Jackson, A. J. Robinson, *New J. Chem.* **2003**, *27*, 387-394.

[2] Itans S. Hutchinson.; Stephen A. Matlin.; Antonio Mete. The synthesis and chemistry of 3-diazo-piperidin-2-one.*Tetrahedron,* **2002,** *58,* 3137-3143

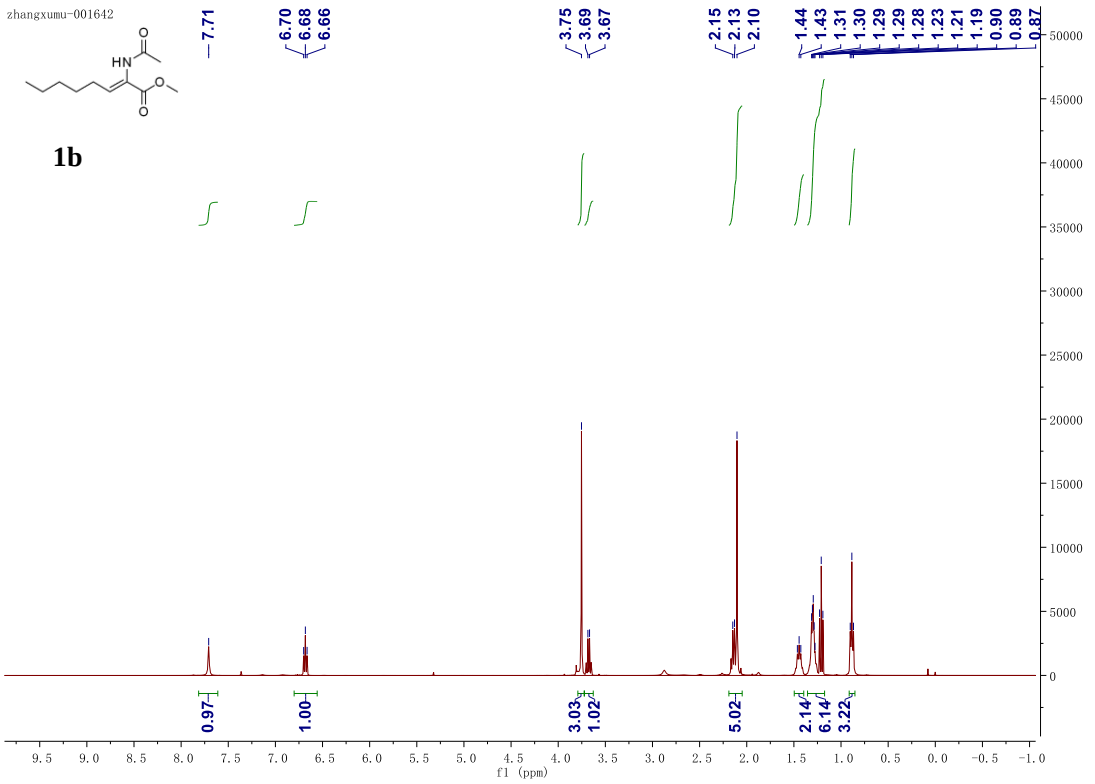
NMR Spectra



1a



1a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2.10
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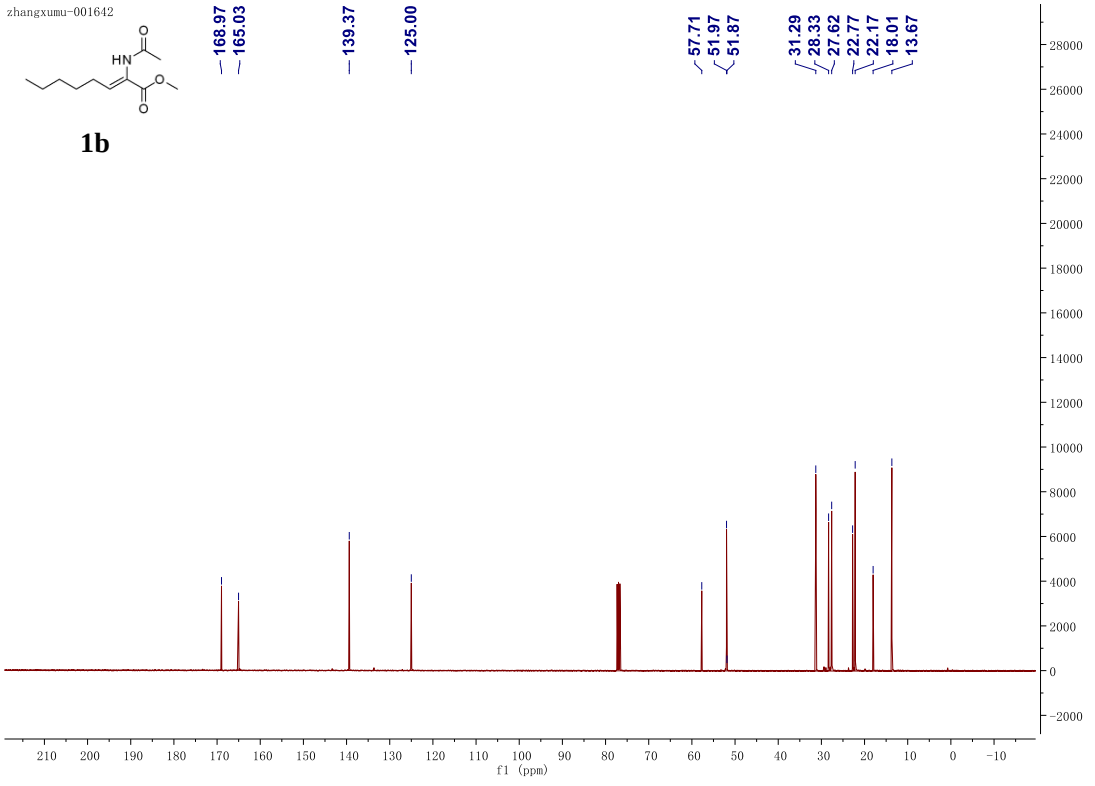
1.43
1.31
1.30
1.29
1.29
1.28
1.23
1.21
1.19
0.90
0.89
0.87
0.97
1.00
3.03
1.02
5.02
2.14
6.14
3.22
f1 (ppm)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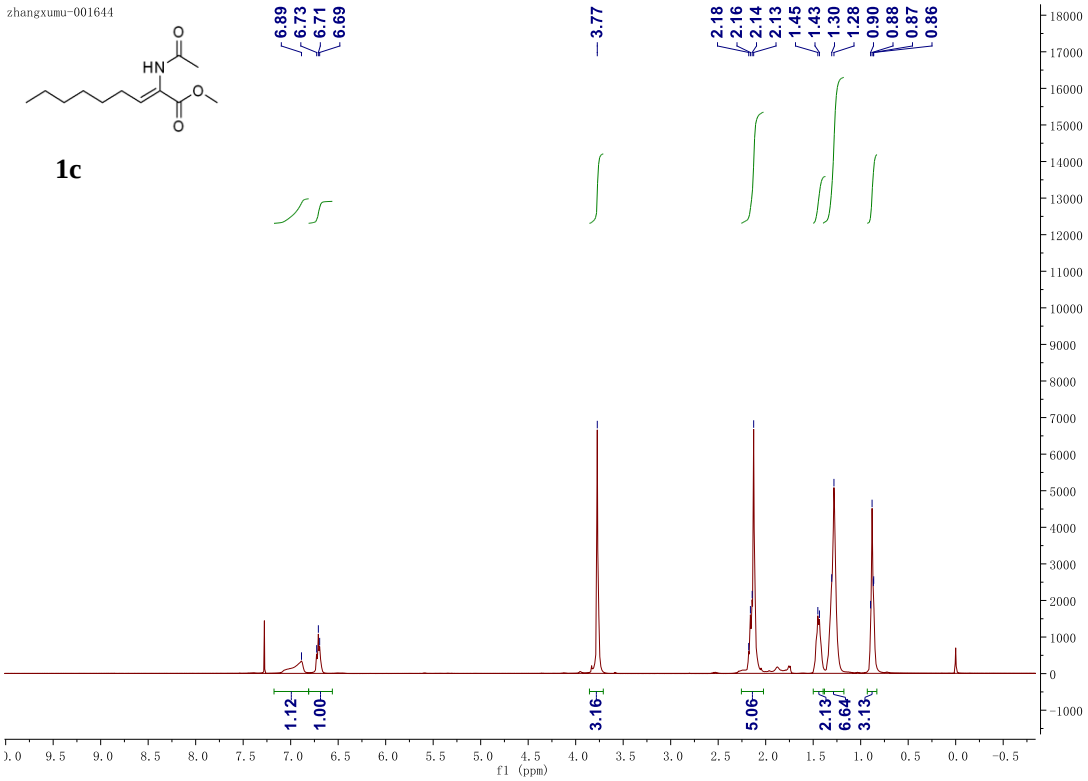
f1 (ppm)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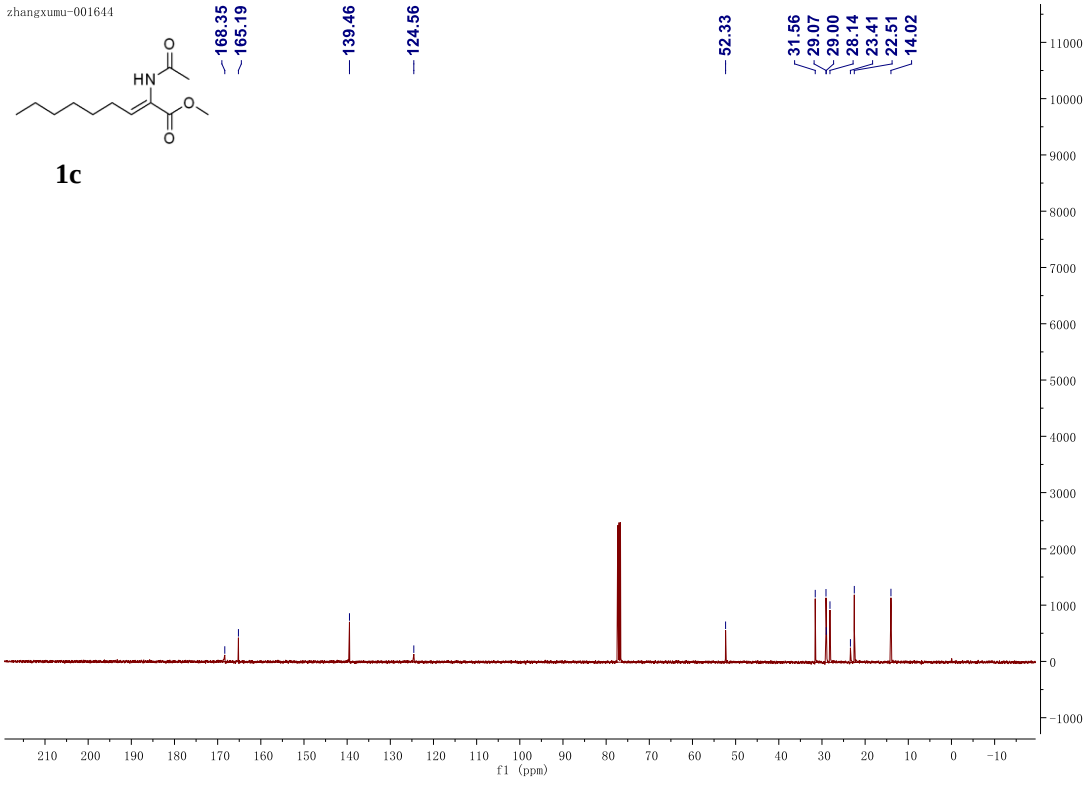
3.13
f1 (ppm)

1c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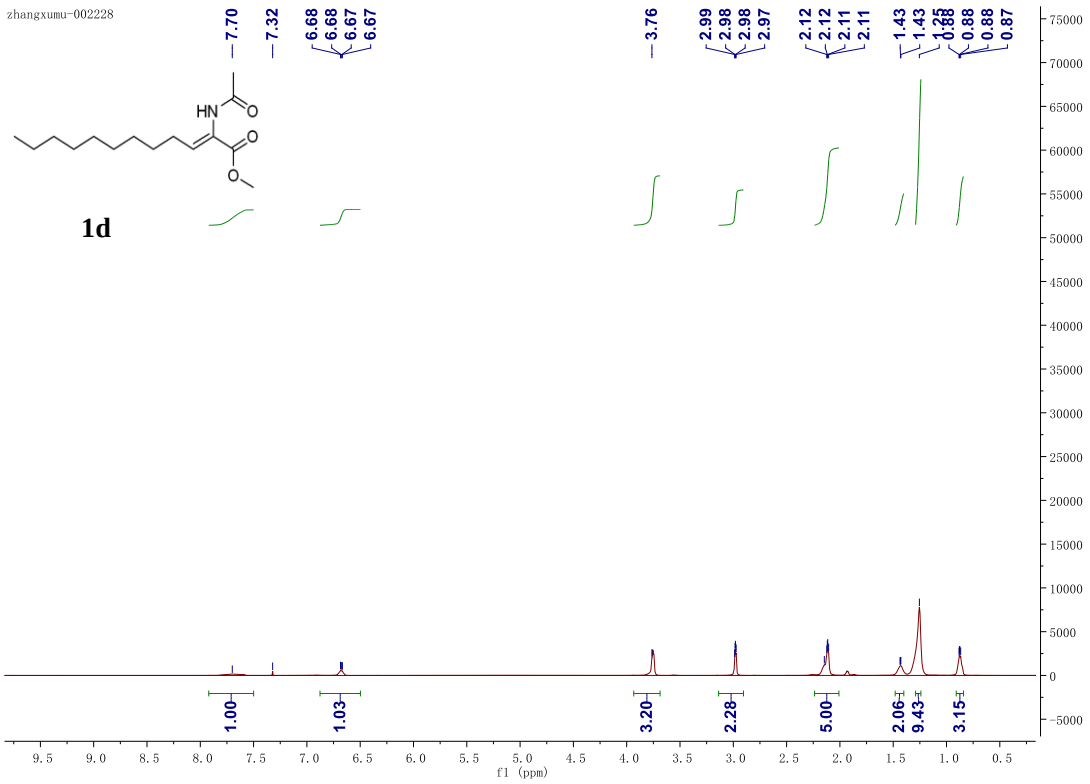
f1 (ppm)

1c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2.12
2.11
2.11
1.43
1.43
0.88
0.88
0.87
HN
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3.20
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2.06
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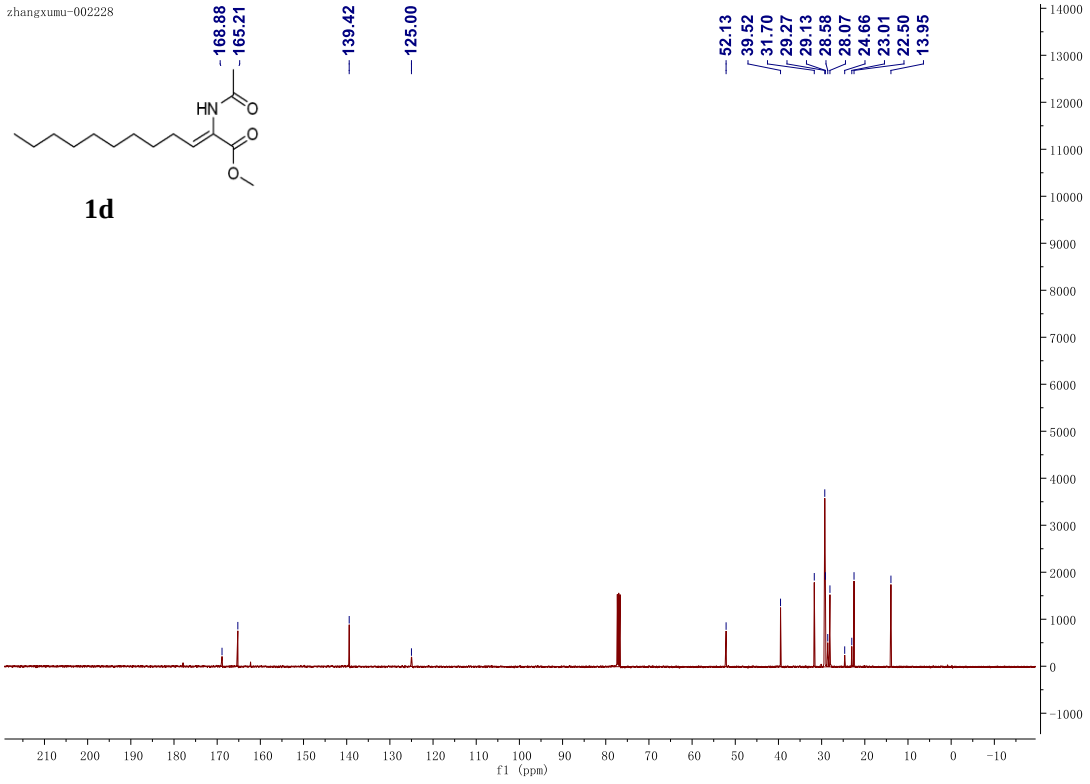
f1 (ppm)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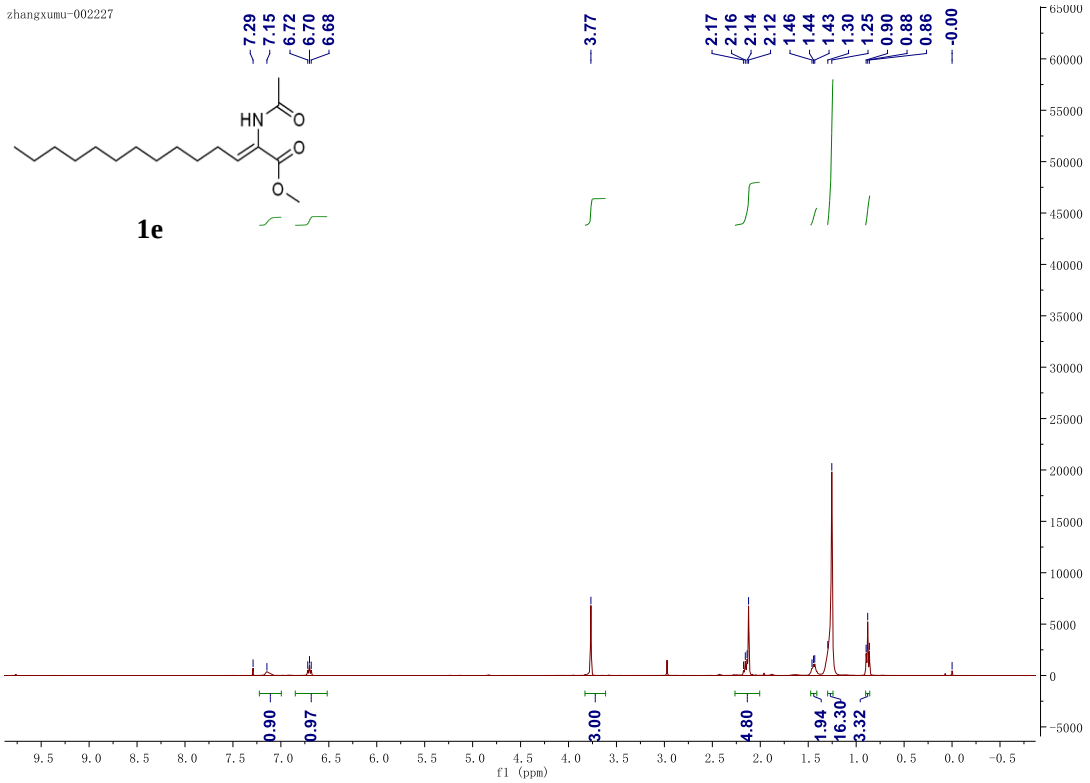
f1 (ppm)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1.43
1.30
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0.88
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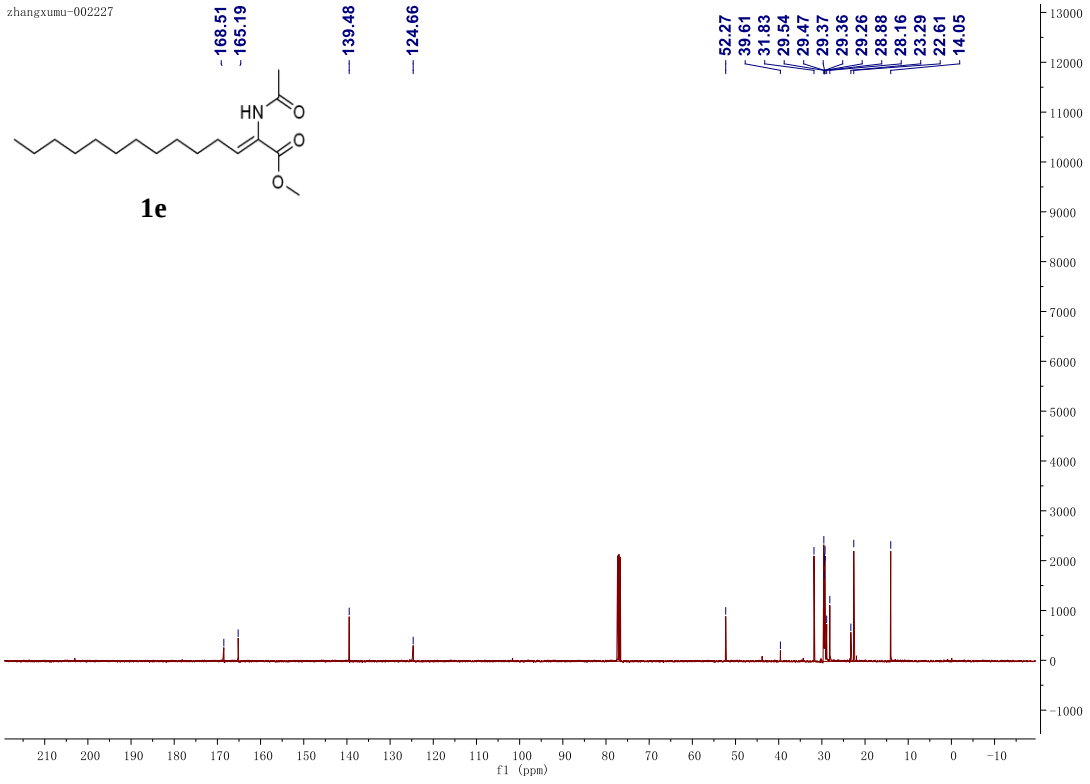
f1 (ppm)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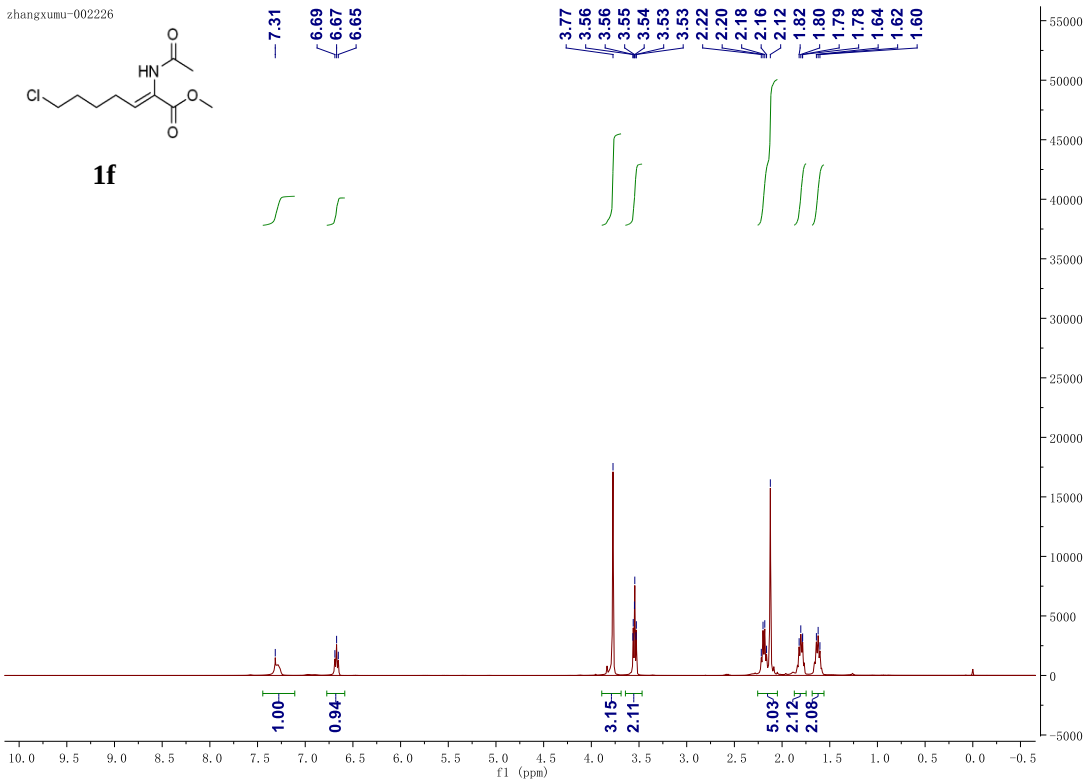
1e
f1 (ppm)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2.20
2.18
2.16
2.12
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1.80
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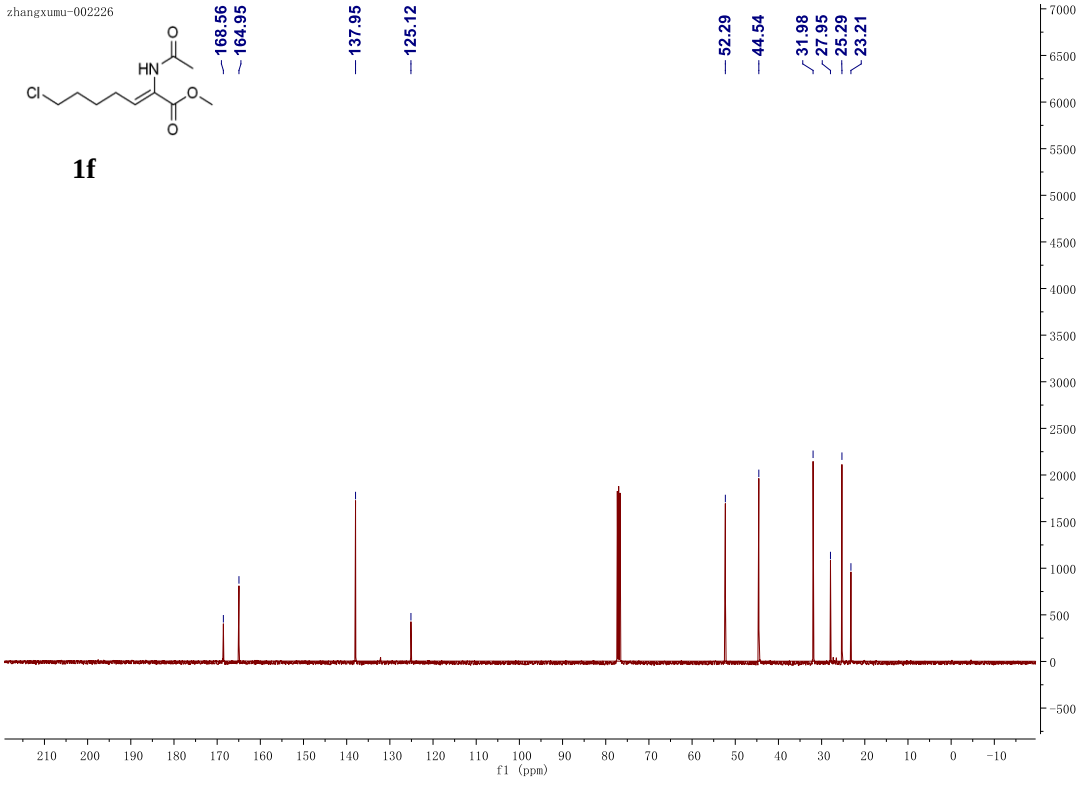
f1 (ppm)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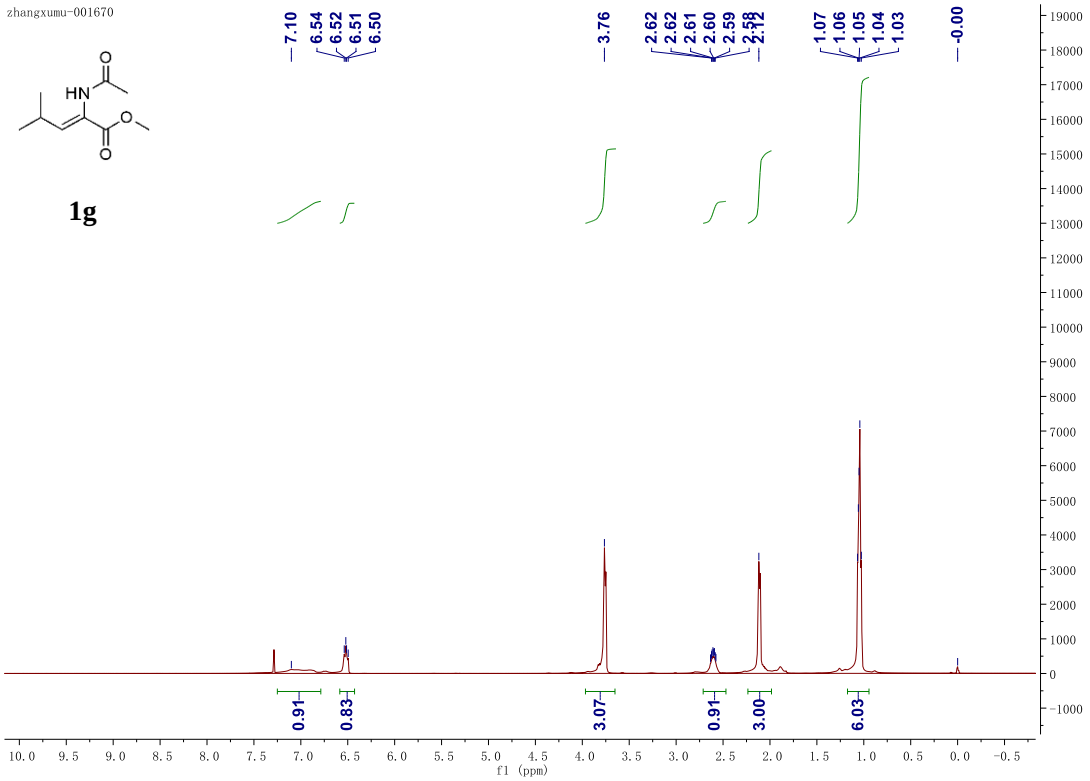
25.29
23.21
f1 (ppm)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2.59
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1.06
1.05
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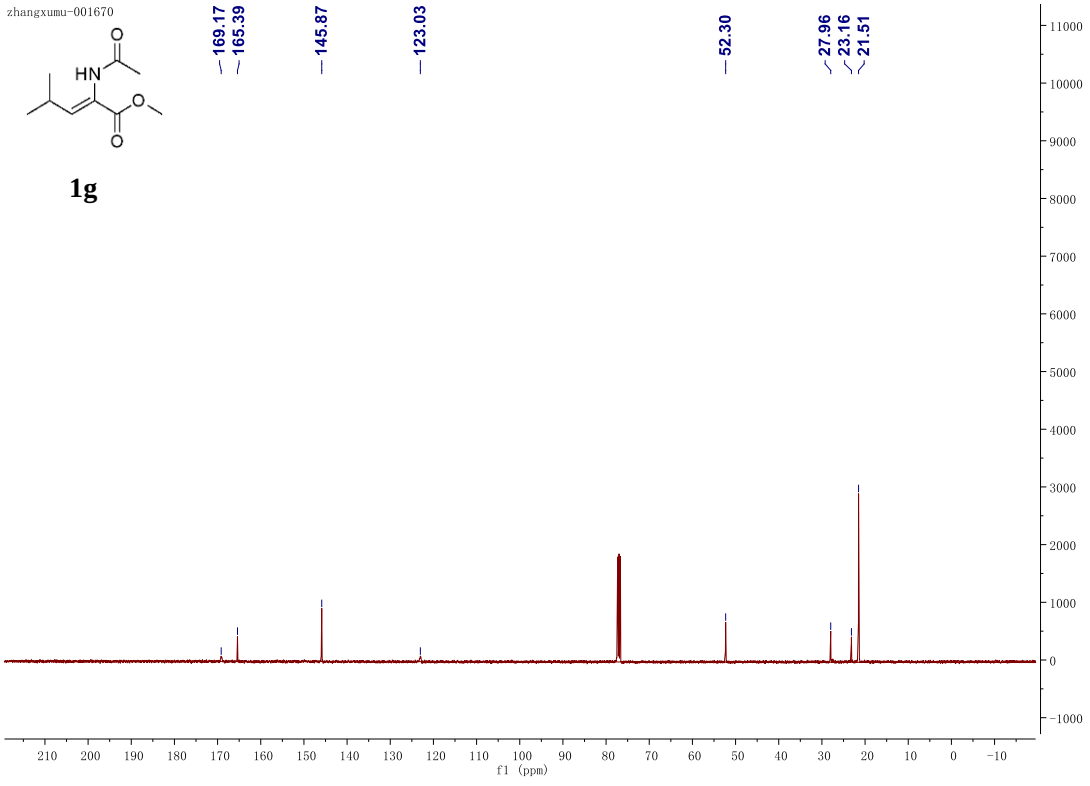
f1 (ppm)

1g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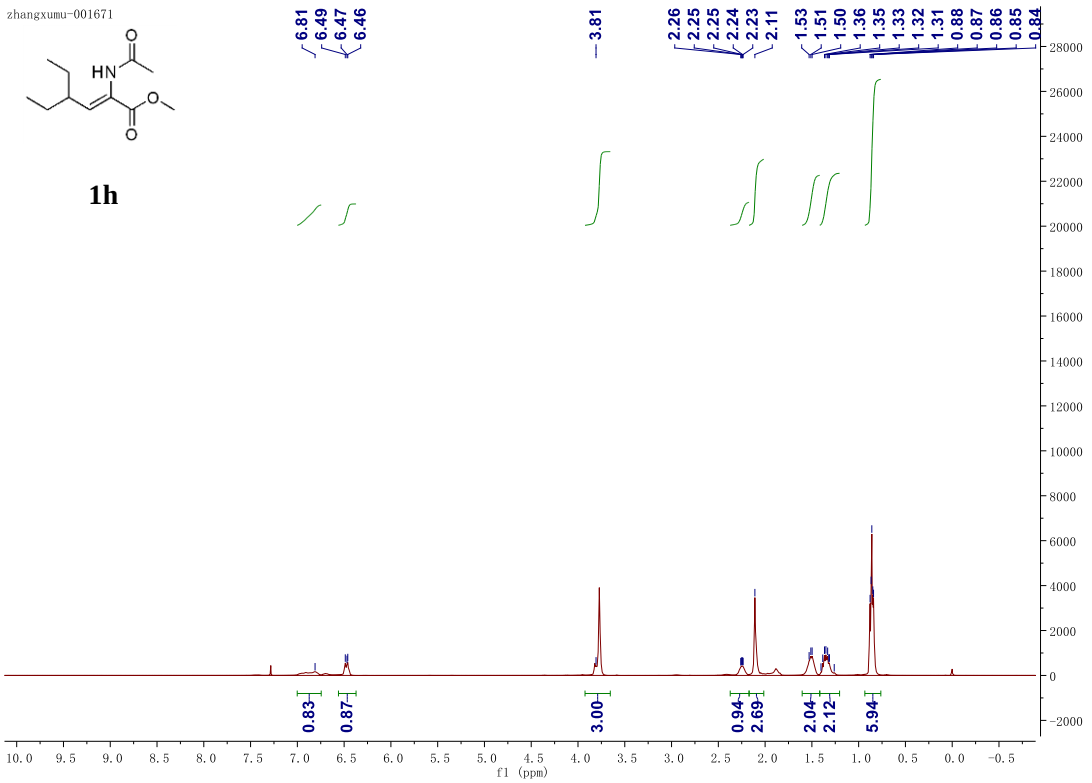
1g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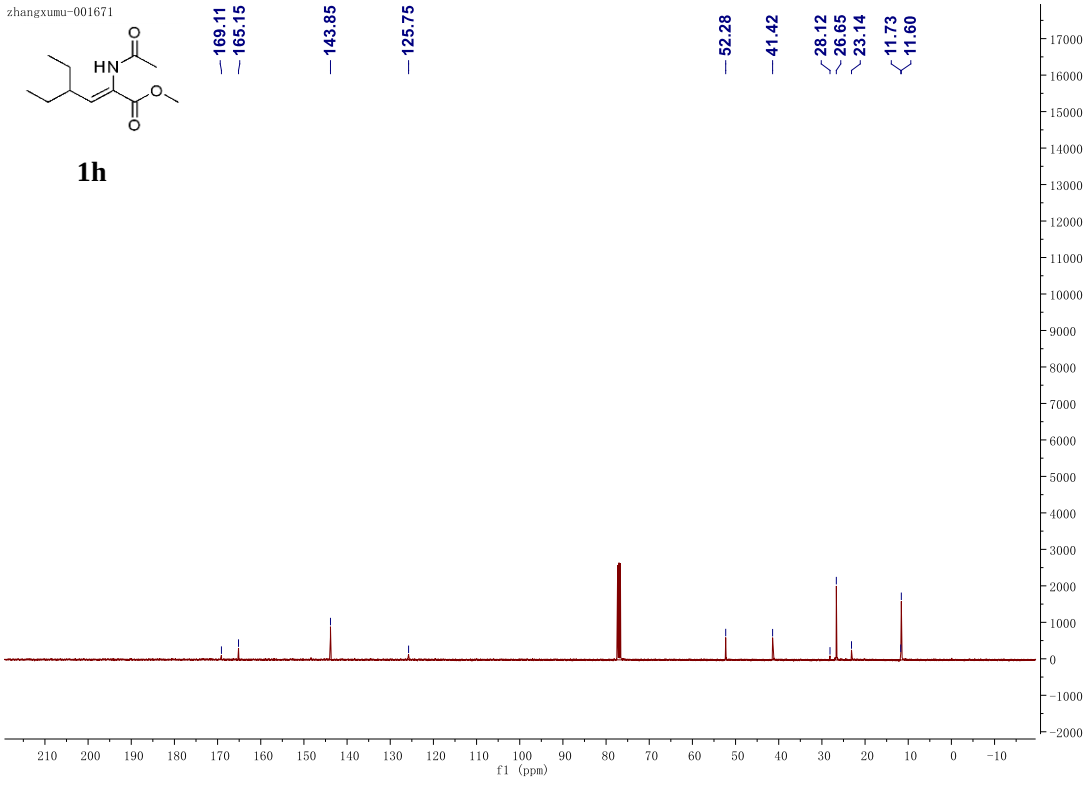
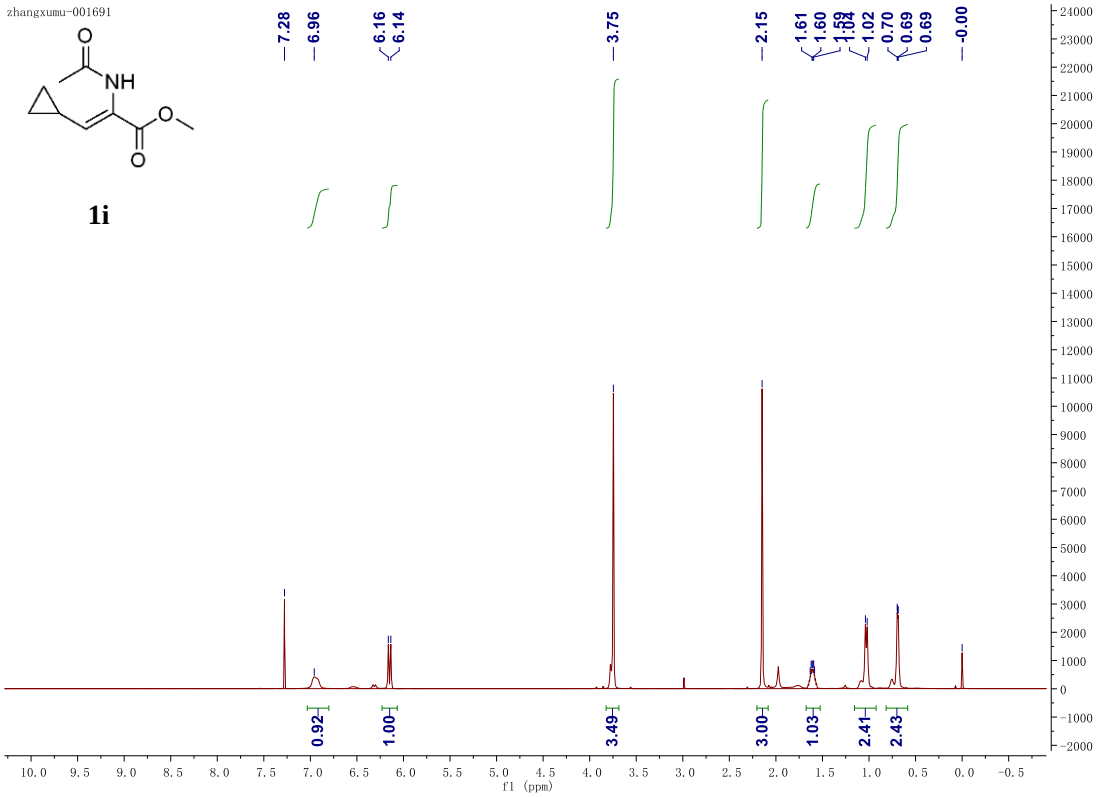
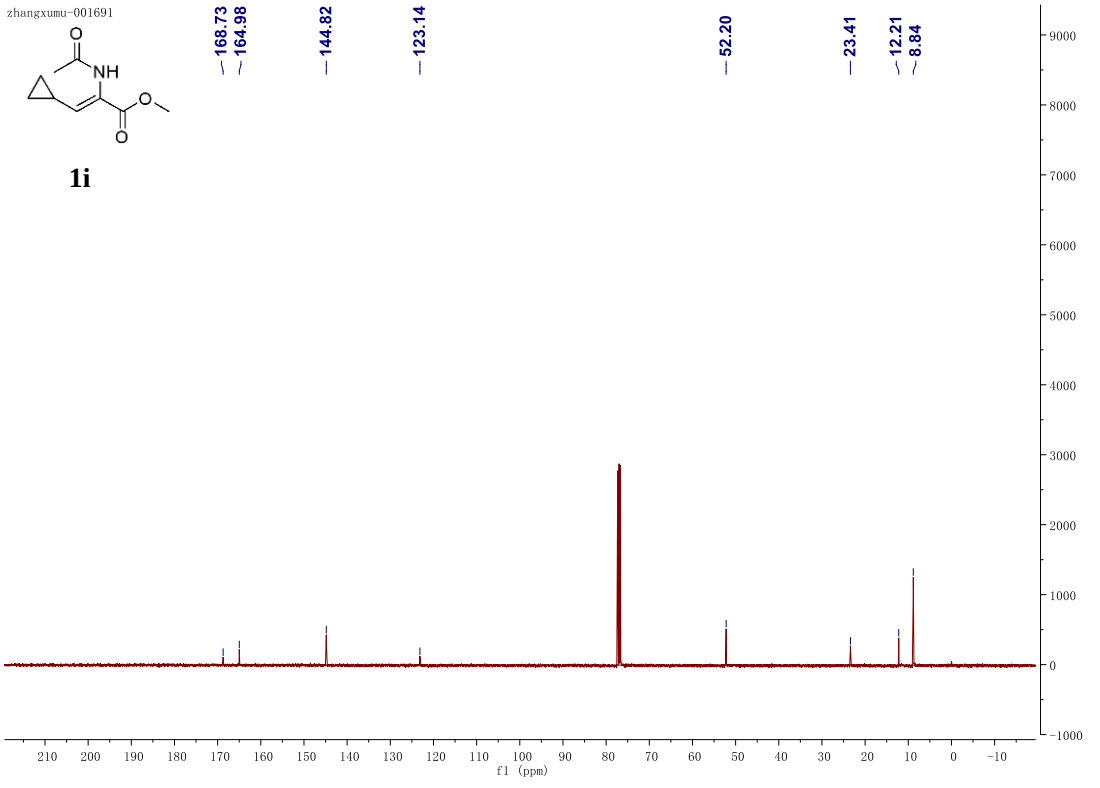
1.51
1.50
1.36
1.35
1.33
1.32
1.31
0.88
0.87
0.86
0.85
0.84
0.83
0.87
3.00
0.94
2.69
2.04
2.12
5.94
f1 (ppm)

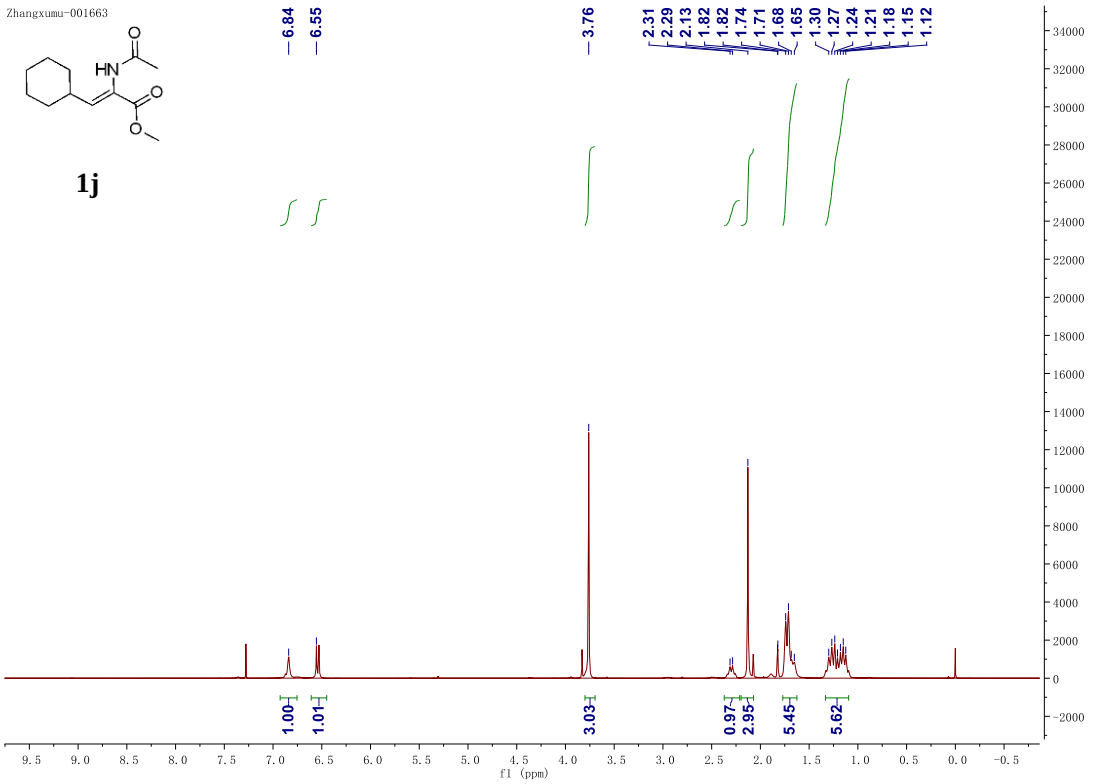
1h


zhangxumu-001671
169.11
165.15
143.85
125.75
52.28
41.42
28.12
26.65
23.14
11.73
11.60
f1 (ppm)

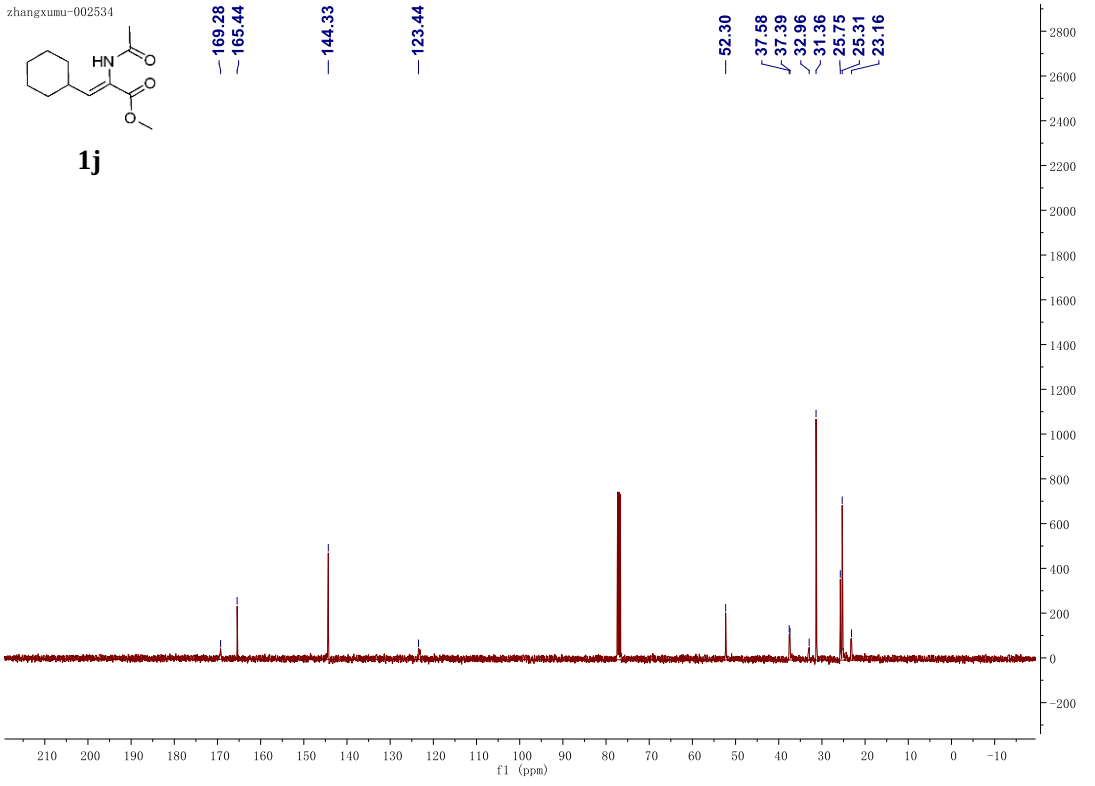
1h


zhangxumu-001691
NH
O
O
O
1i
7.28
6.96
6.16
6.14
3.75
2.15
1.61
1.60
1.59
1.04
1.02
0.70
0.69
0.69
-0.00
0.92
1.00
3.49
3.00
1.03
2.41
2.43
f1 (ppm)

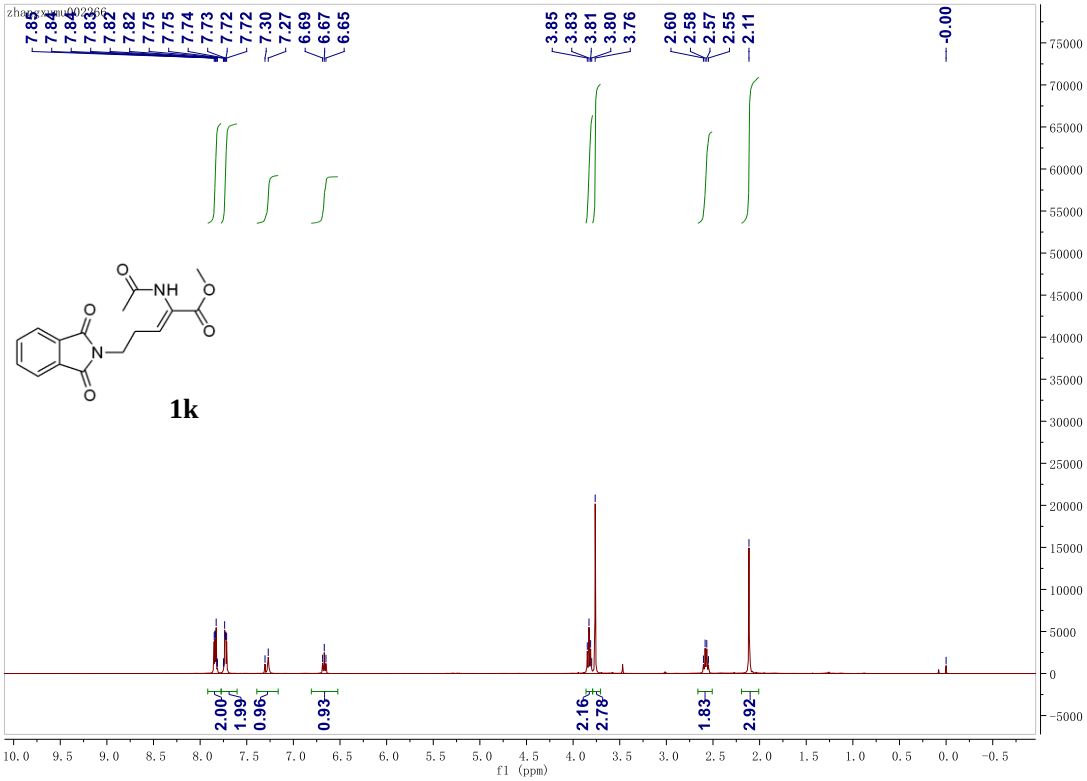

zhangxumu-001691
NH
O
O
O
1i
168.73
164.98
144.82
123.14
52.20
23.41
12.21
8.84
f1 (ppm)


Zhangxumu-001663
HN
O
O
O
6.84
6.55
3.76
2.31
2.29
2.13
1.82
1.74
1.71
1.68
1.65
1.30
1.27
1.24
1.21
1.18
1.15
1.12
1.00
1.01
3.03
0.97
2.95
5.45
5.62
f1 (ppm)

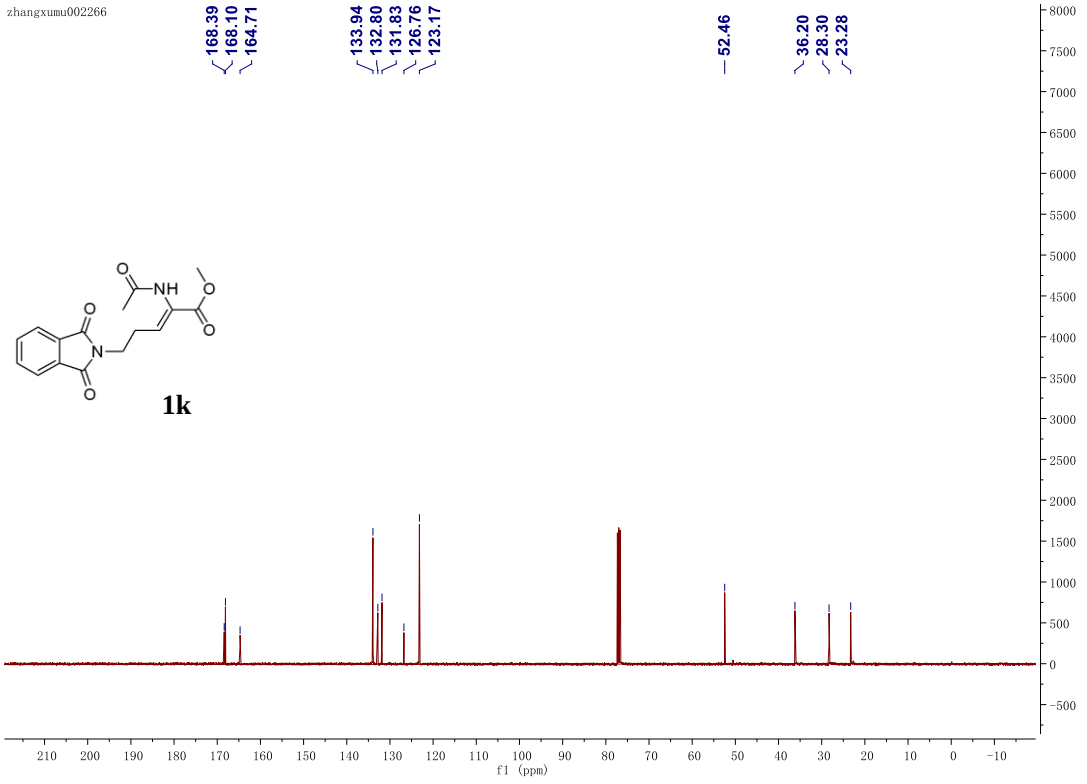
1j


zhangxumu-002534
HN
O
O
O
169.28
165.44
144.33
123.44
52.30
37.58
37.39
32.96
31.36
25.75
25.31
23.16
f1 (ppm)

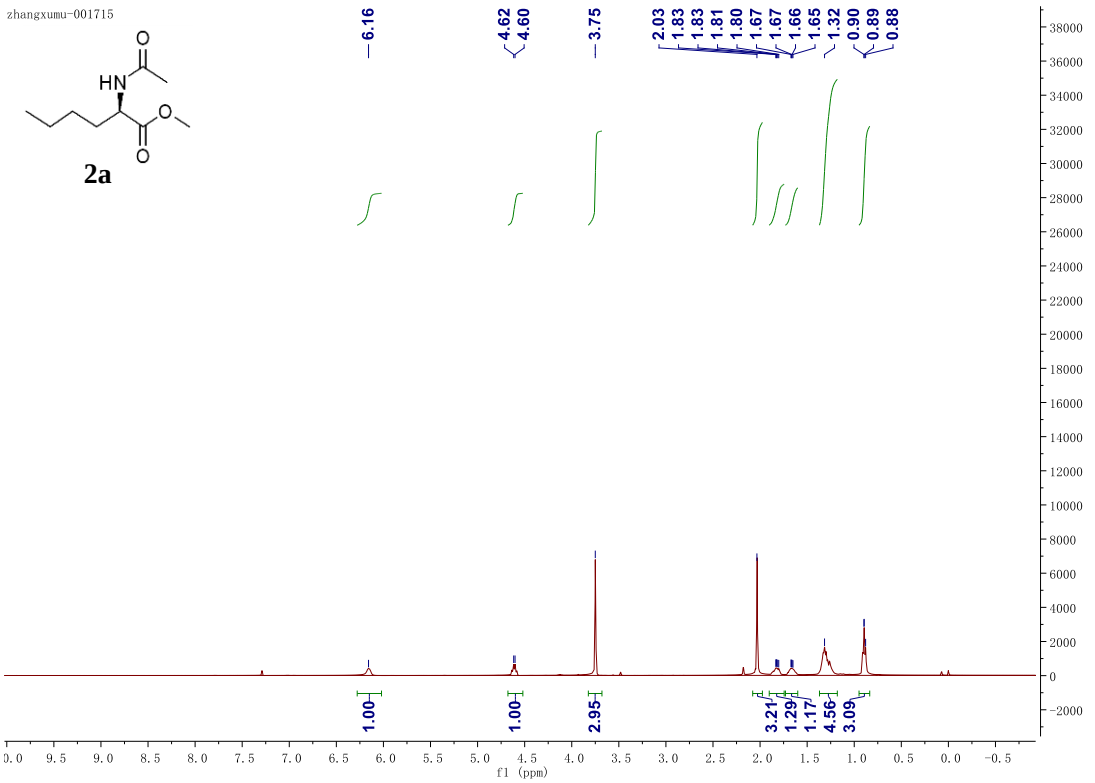
1j

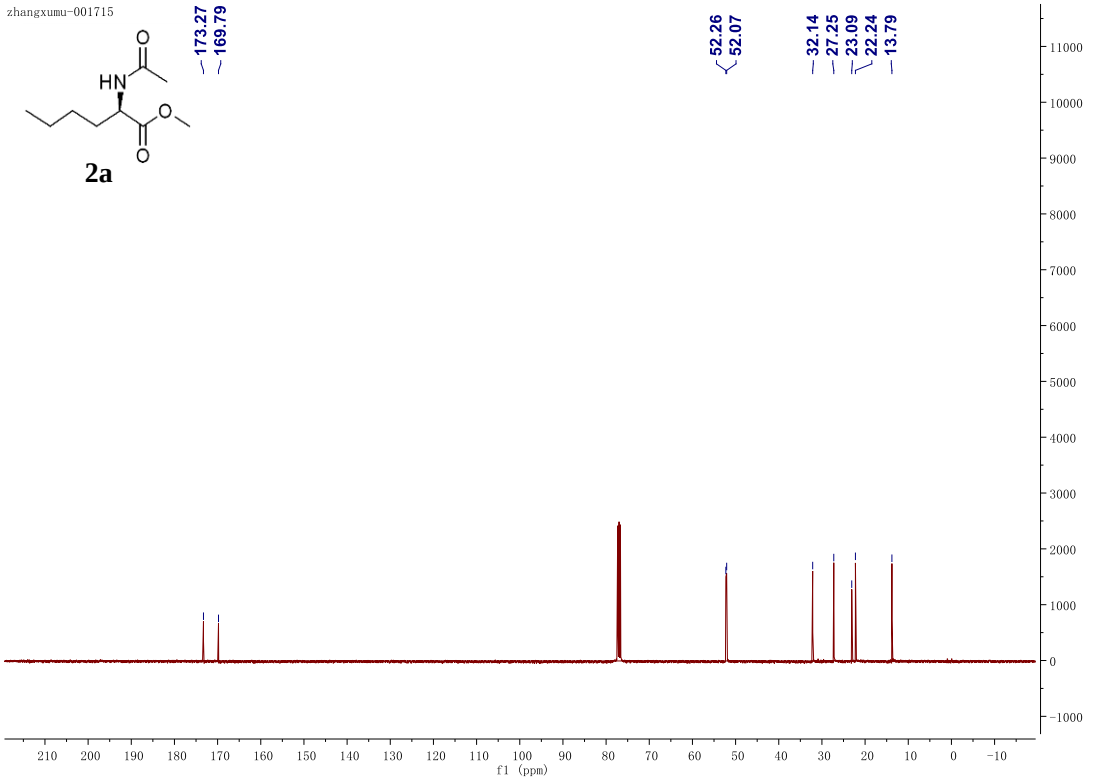

7.85
7.84
7.84
7.83
7.82
7.82
7.75
7.75
7.74
7.73
7.72
7.72
7.30
7.27
6.69
6.67
6.65
3.85
3.83
3.81
3.80
3.76
2.60
2.58
2.57
2.55
2.11
-0.00
NH
O
N
2.00
1.99
0.96
0.93
2.16
2.78
1.83
2.92
f1 (ppm)

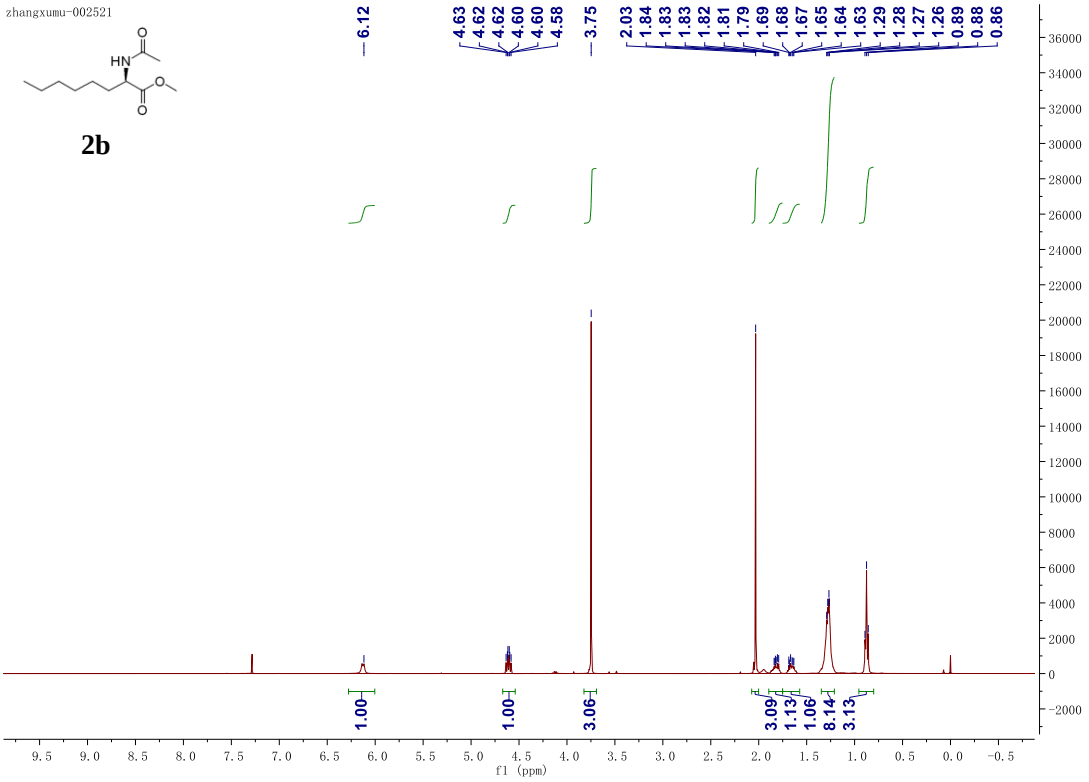
1k

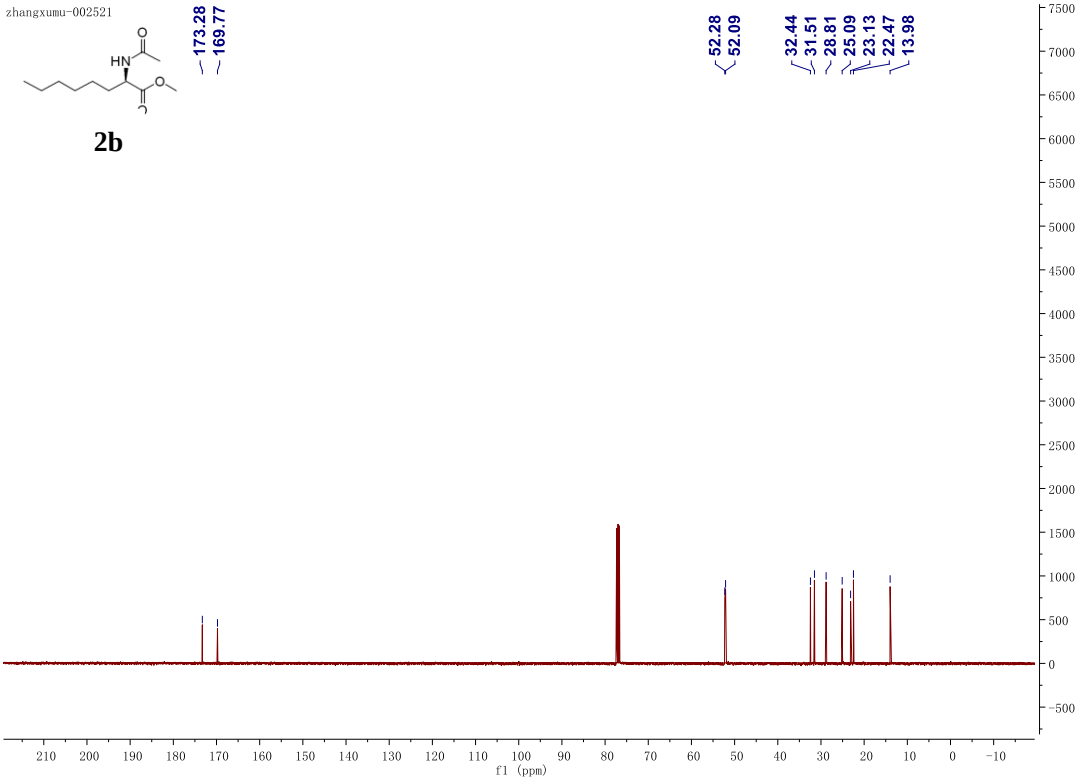

zhangxumu002266
168.39
168.10
164.71
133.94
132.80
131.83
126.76
123.17
52.46
36.20
28.30
23.28
f1 (ppm)

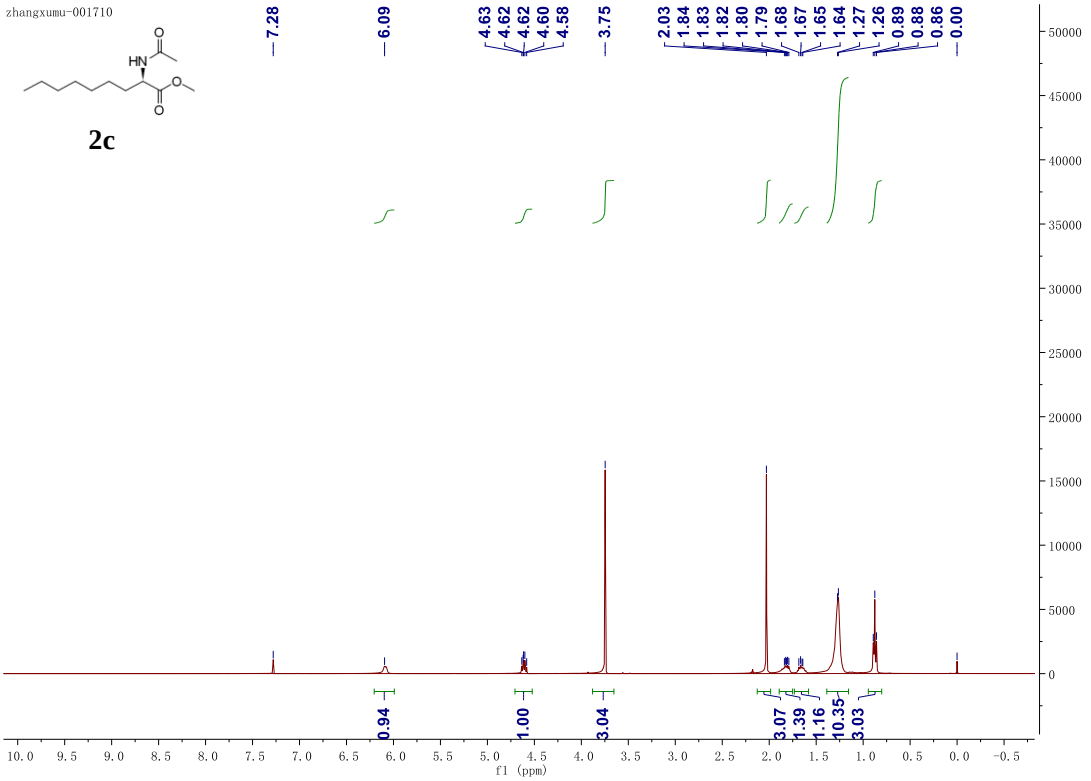
1k


zhangxumu-001715
2a
6.16
4.62
4.60
3.75
2.03
1.83
1.83
1.81
1.80
1.67
1.67
1.66
1.65
1.32
0.90
0.89
0.88
1.00
1.00
2.95
3.21
1.29
1.17
4.56
3.09
f1 (ppm)

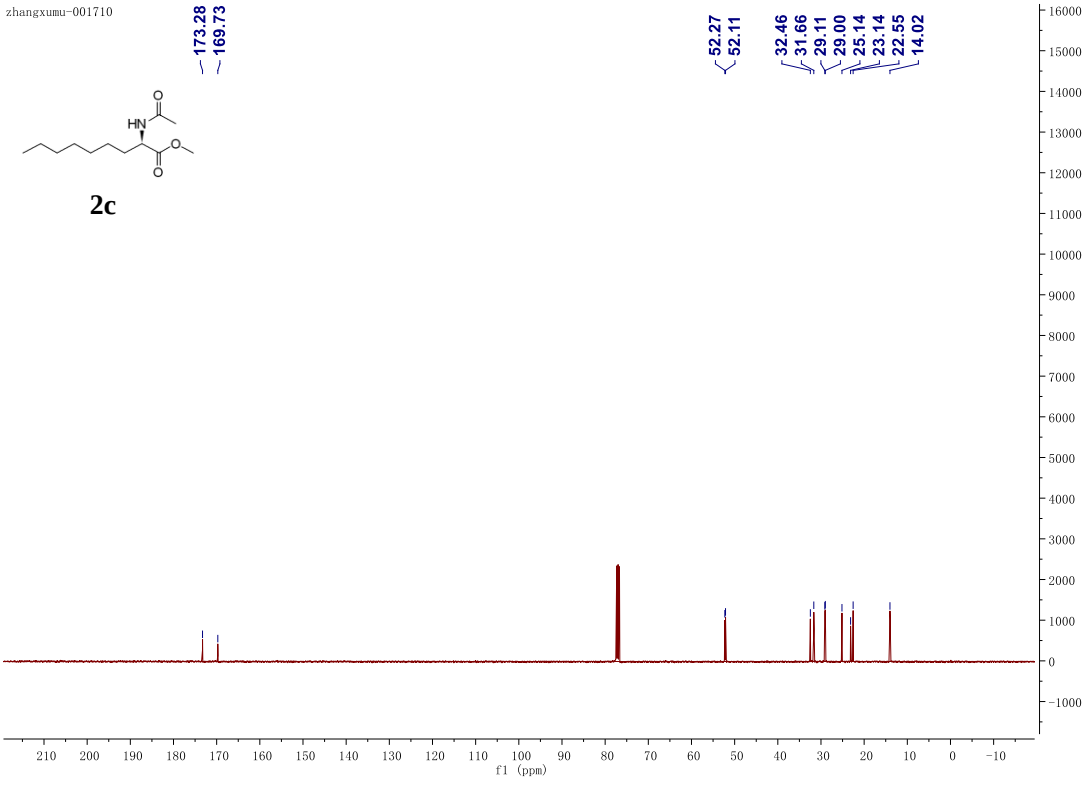

zhangxumu-001715
2a
173.27
169.79
52.26
52.07
32.14
27.25
23.09
22.24
13.79
f1 (ppm)


zhangxumu-002521
2b
6.12
4.63
4.62
4.62
4.60
4.60
4.58
3.75
2.03
1.84
1.83
1.83
1.82
1.81
1.79
1.69
1.68
1.67
1.65
1.64
1.63
1.29
1.28
1.27
1.26
0.89
0.88
0.86
1.00
1.00
3.06
3.09
1.13
1.06
8.14
3.13
f1 (ppm)

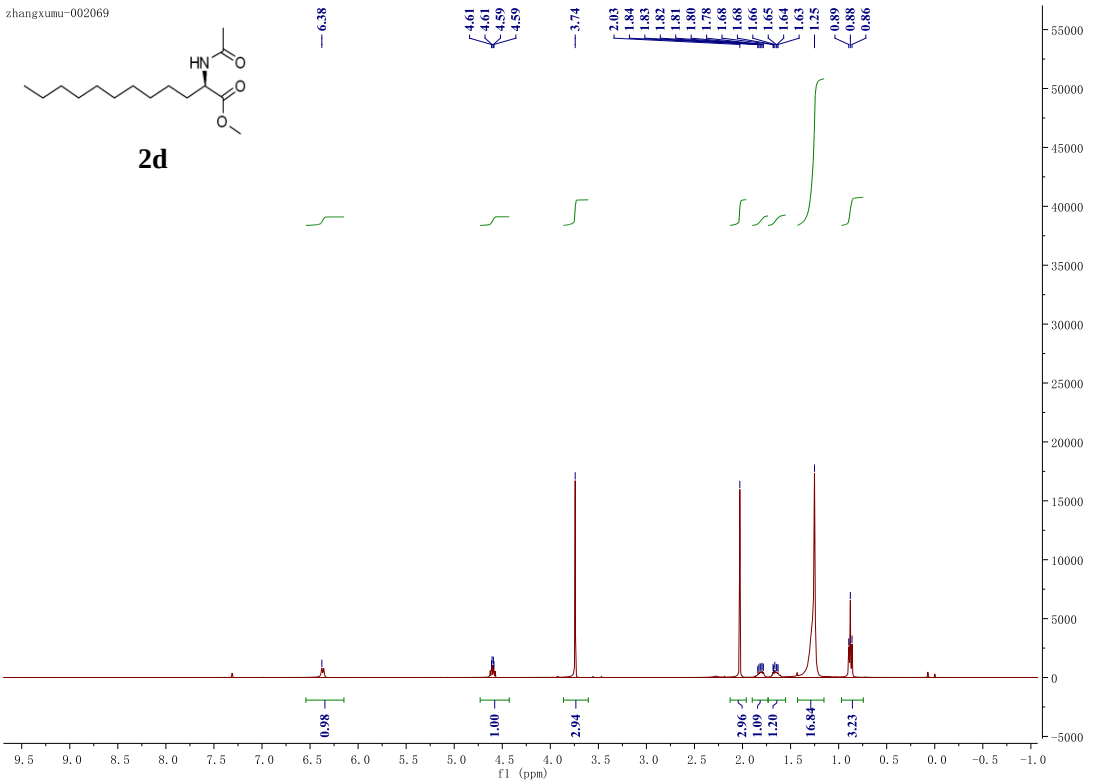

zhangxumu-002521
2b
173.28
169.77
52.28
52.09
32.44
31.51
28.81
25.09
23.13
22.47
13.98
f1 (ppm)

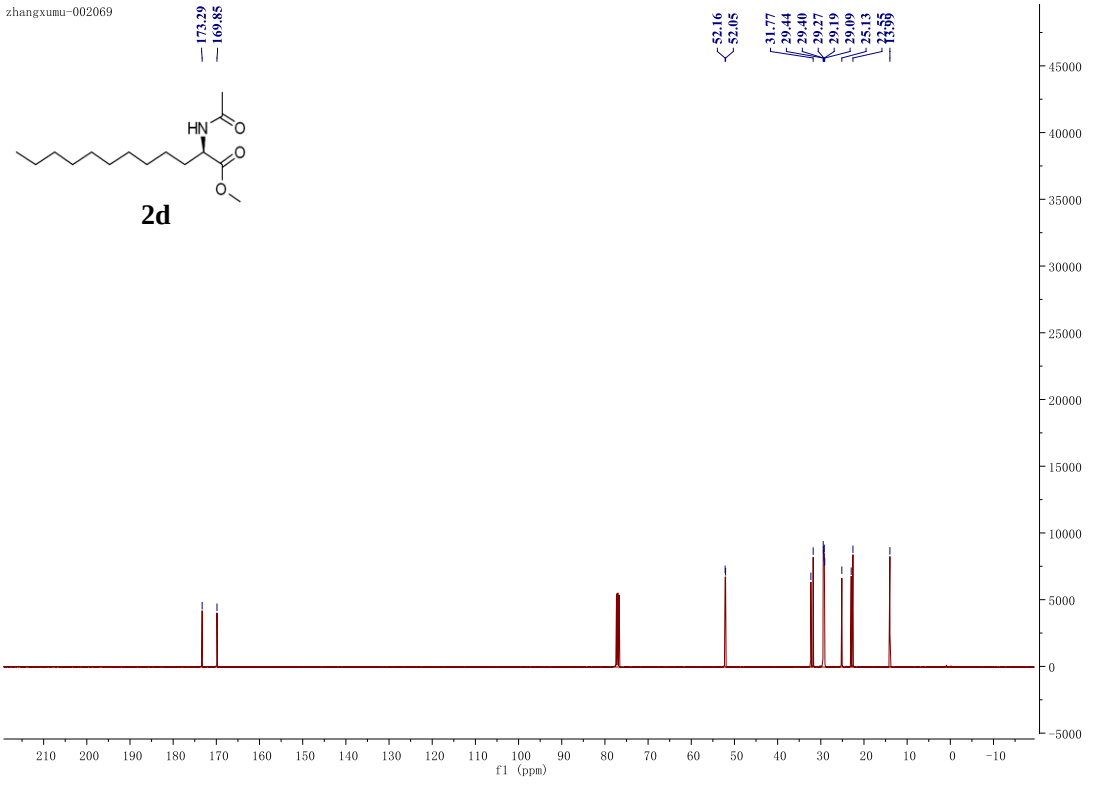

zhangxumu-001710
7.28
6.09
4.63
4.62
4.62
4.60
4.58
3.75
2.03
1.84
1.83
1.82
1.80
1.79
1.68
1.67
1.65
1.64
1.27
1.26
0.89
0.88
0.86
0.00
0.94
1.00
3.04
3.07
1.39
1.16
10.35
3.03
f1 (ppm)

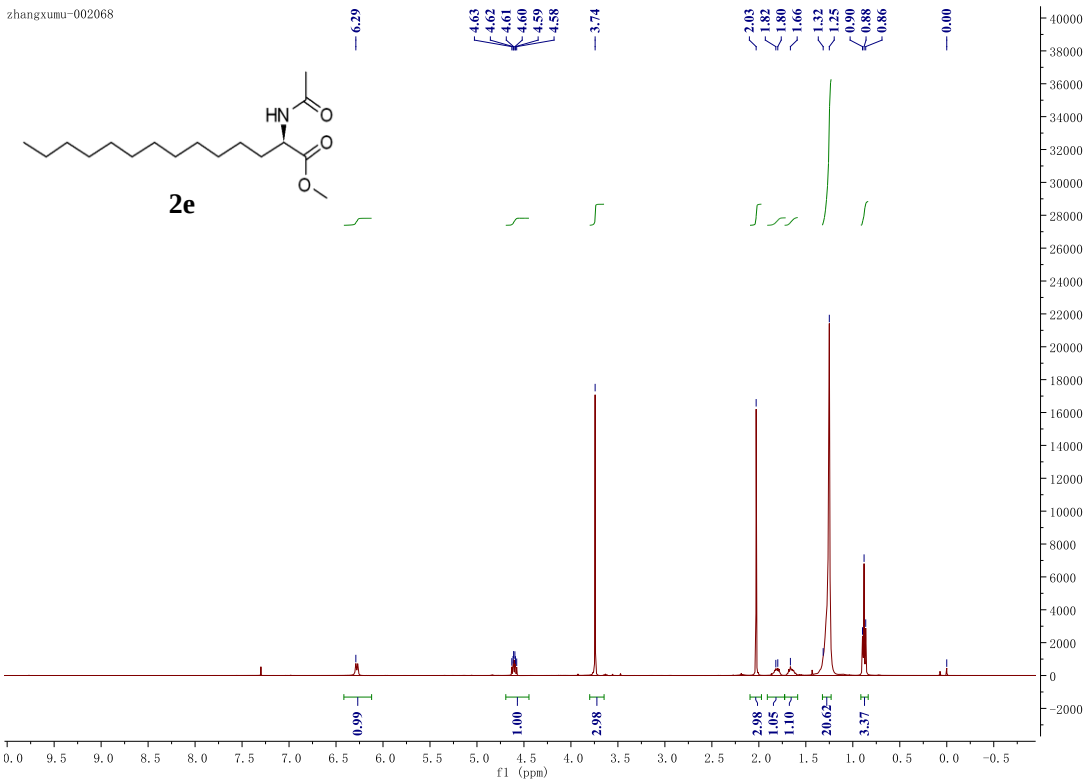
2c

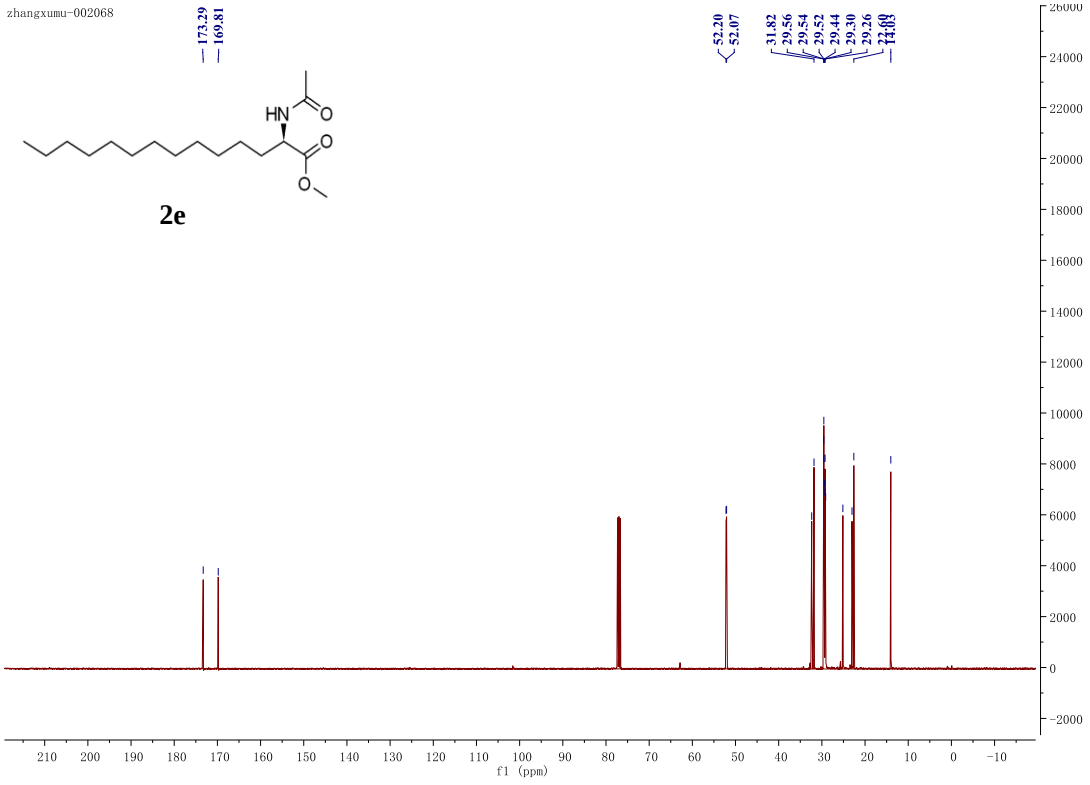

zhangxumu-001710
173.28
169.73
52.27
52.11
32.46
31.66
29.11
29.00
25.14
23.14
22.55
14.02
f1 (ppm)

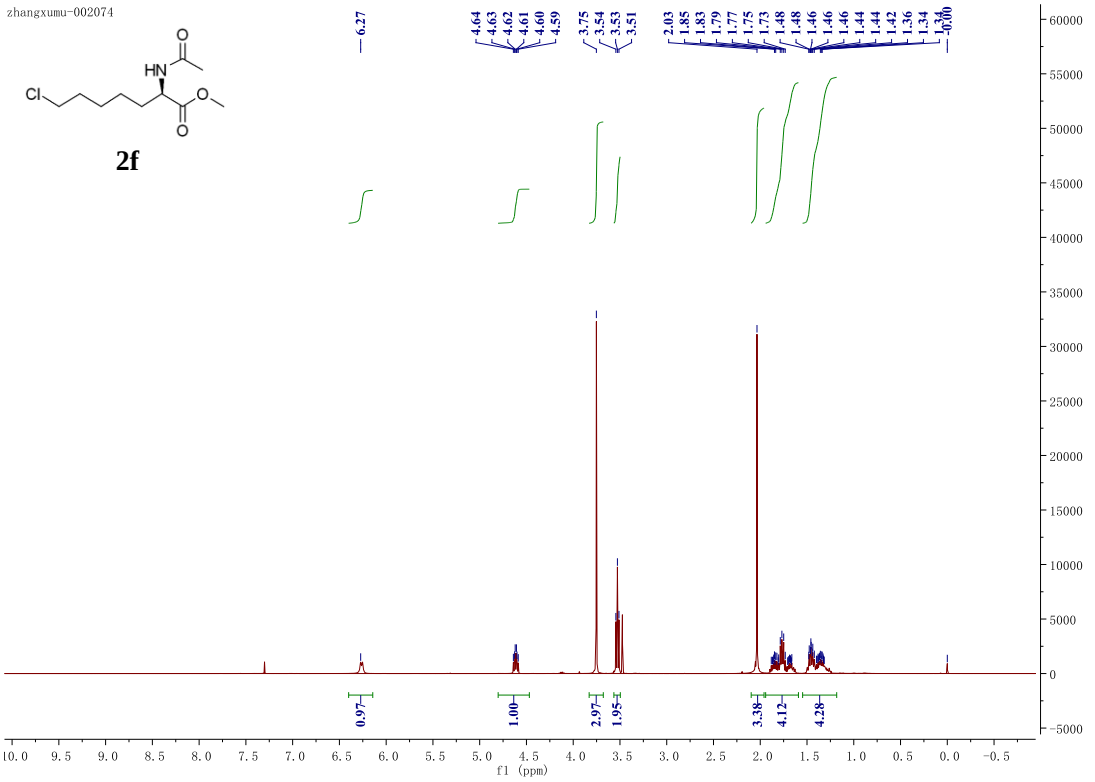
2c

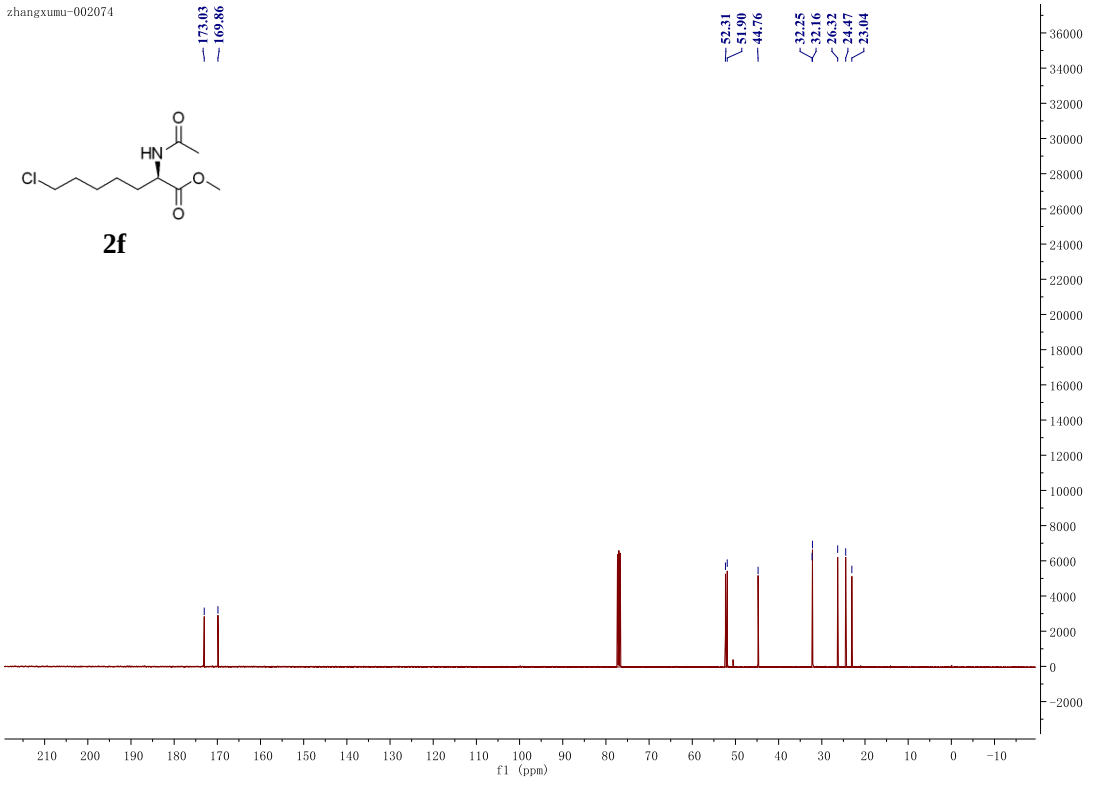

zhangxumu-002069
2d
6.38
4.61
4.61
4.59
4.59
3.74
2.03
1.84
1.83
1.82
1.81
1.80
1.78
1.68
1.68
1.66
1.65
1.64
1.63
1.25
0.89
0.88
0.86
0.98
1.00
2.94
2.96
1.09
1.20
16.84
3.23
f1 (ppm)

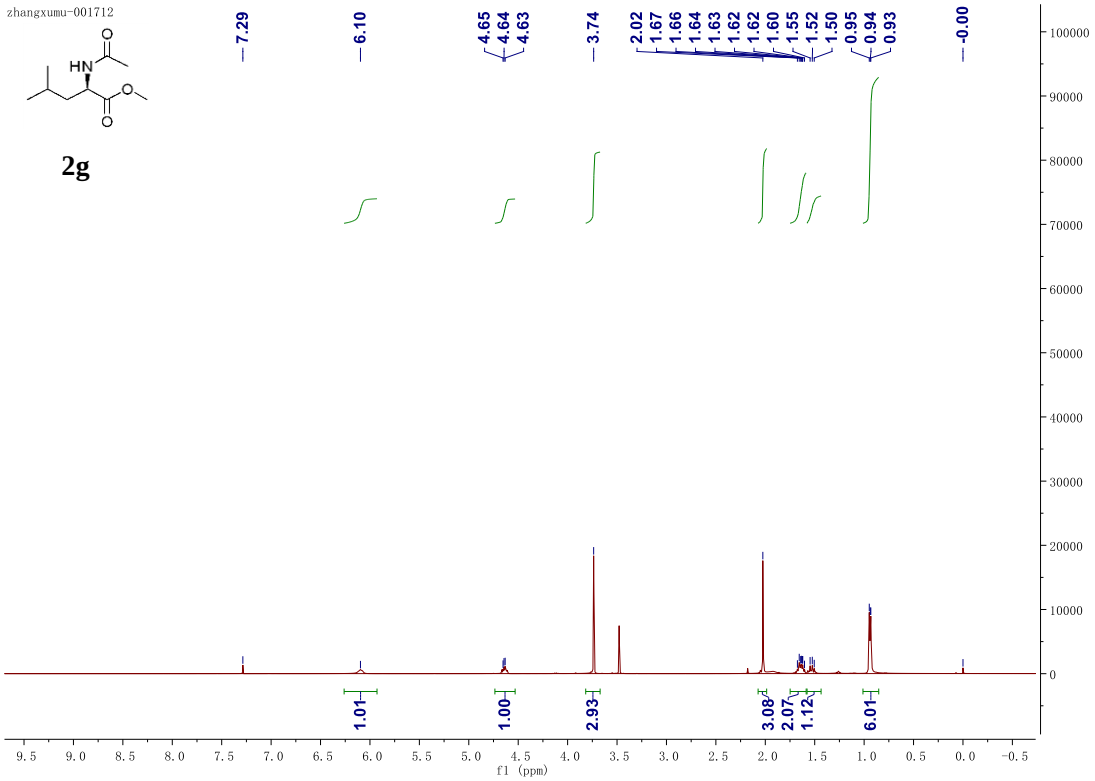

zhangxumu-002069
2d
173.29
169.85
52.16
52.05
31.77
29.44
29.40
29.27
29.19
29.09
25.13
22.55
13.99
f1 (ppm)

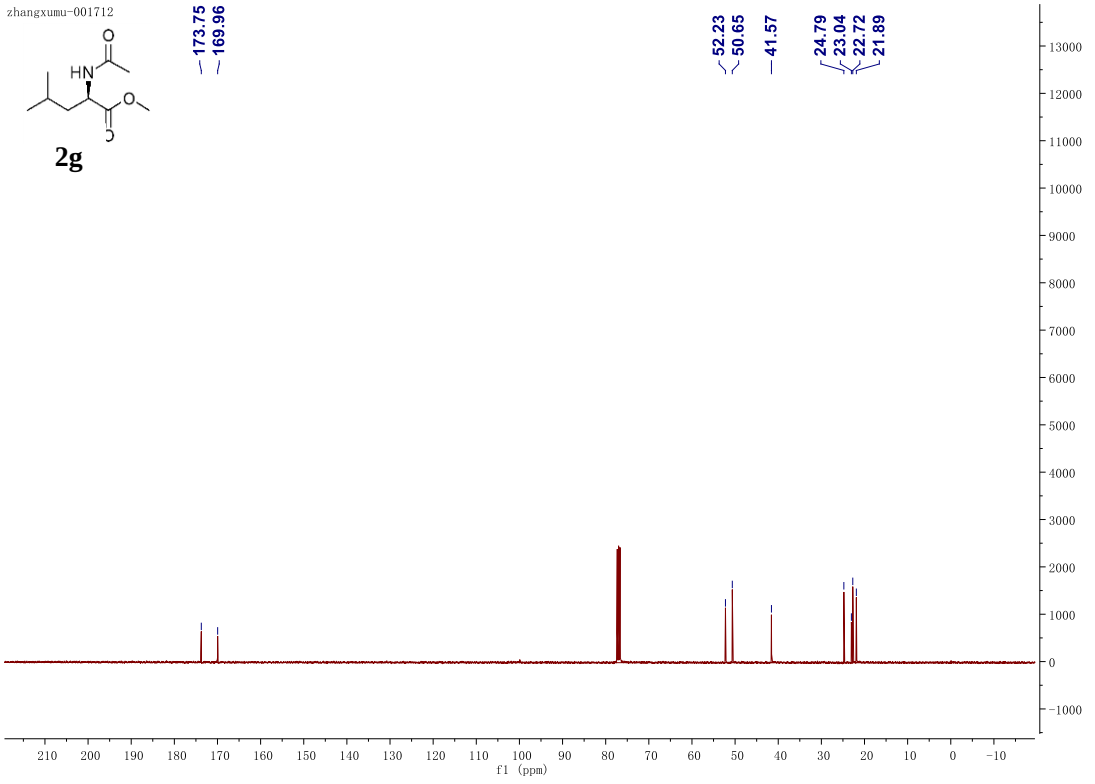

zhangxumu-002068
6.29
4.63
4.62
4.61
4.60
4.59
4.58
3.74
2.03
1.82
1.80
1.66
1.32
1.25
0.90
0.88
0.86
0.00
HN
O
O
O
2e
0.99
1.00
2.98
2.98
1.05
1.10
20.62
3.37
f1 (ppm)

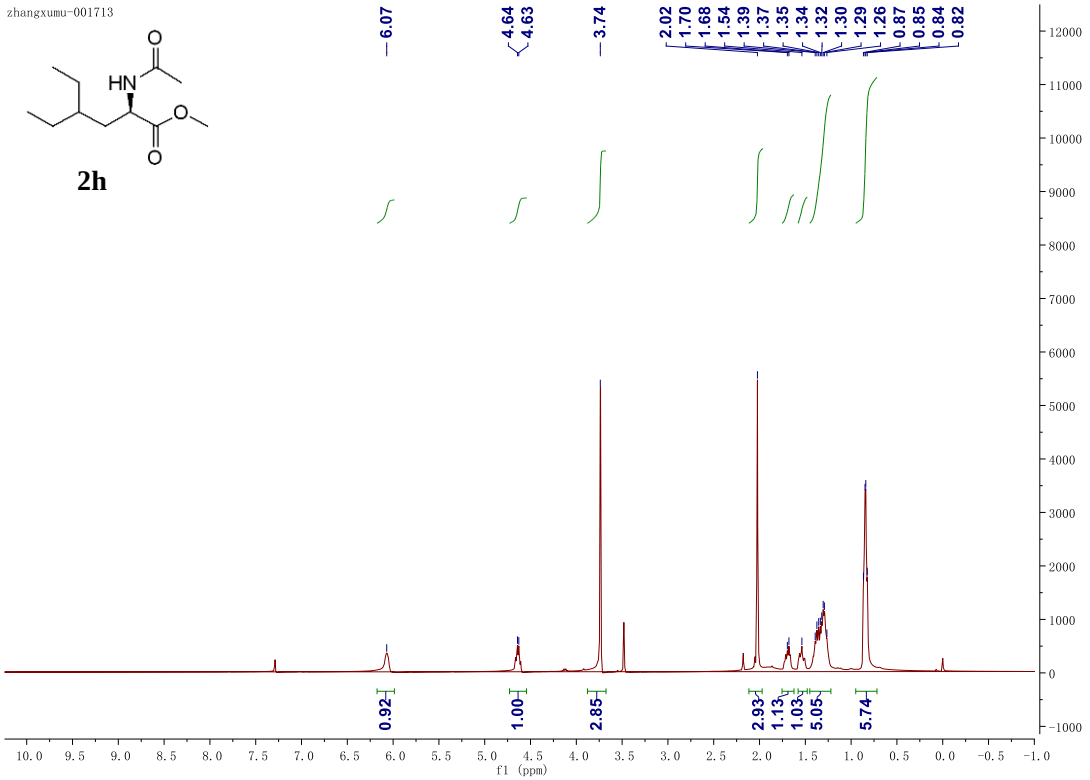

zhangxumu-002068
173.29
169.81
52.20
52.07
31.82
29.56
29.54
29.52
29.44
29.30
29.26
22.60
14.03
HN
O
O
O
2e
f1 (ppm)

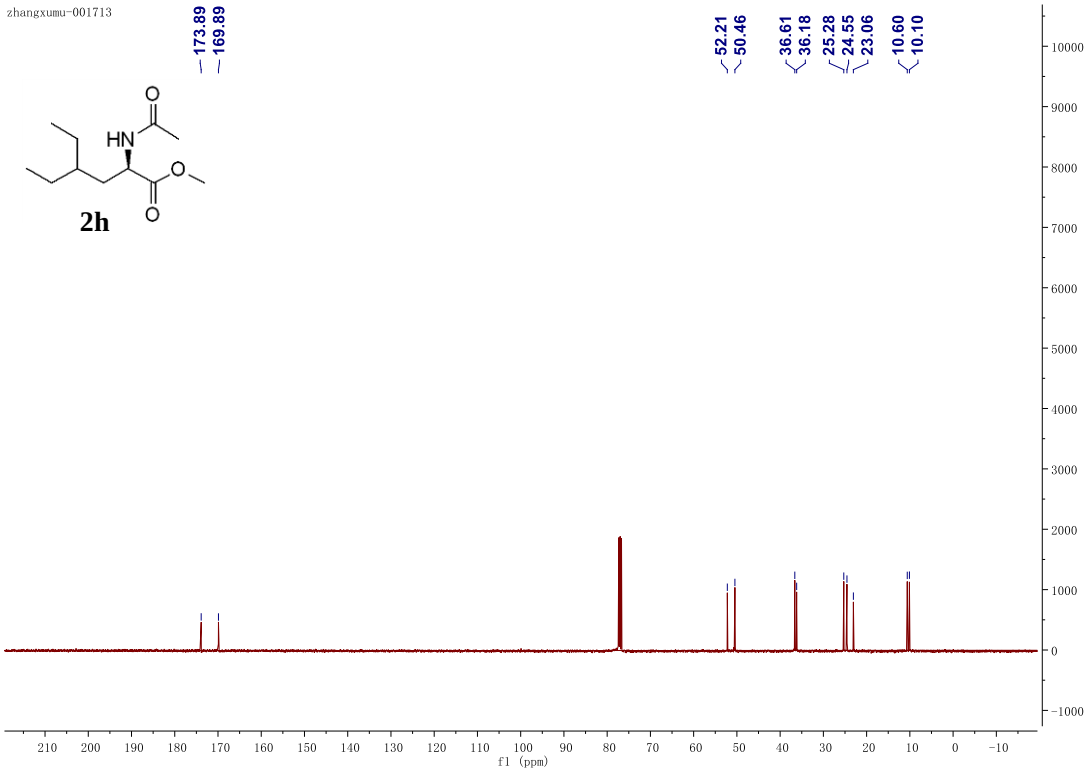

zhangxumu-002074
2f
6.27
4.64
4.63
4.62
4.61
4.60
4.59
3.75
3.54
3.53
3.51
2.03
1.85
1.83
1.79
1.77
1.75
1.73
1.48
1.46
1.46
1.44
1.44
1.42
1.36
1.34
1.34
0.00
0.97
1.00
2.97
1.95
3.38
4.12
4.28
f1 (ppm)

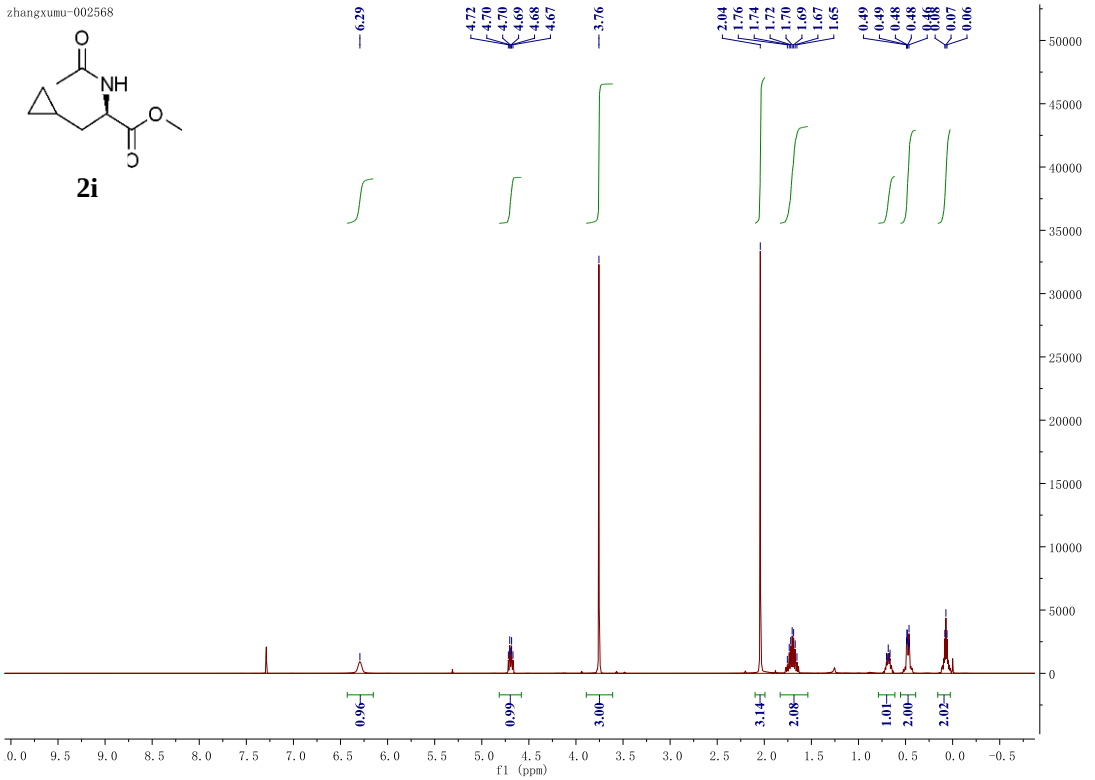

zhangxumu-002074
2f
173.03
169.86
52.31
51.90
44.76
32.25
32.16
26.32
24.47
23.04
f1 (ppm)

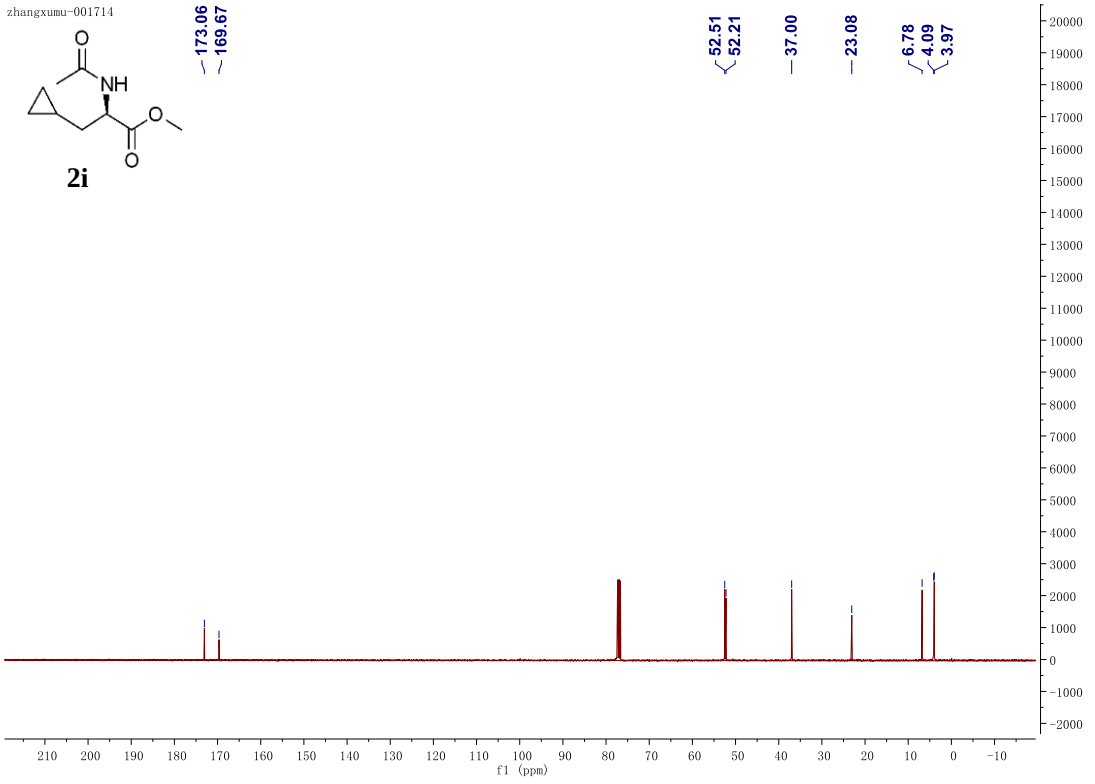

zhangxumu-001712
2g
7.29
6.10
4.65
4.64
4.63
3.74
2.02
1.67
1.66
1.64
1.63
1.62
1.62
1.60
1.55
1.52
1.50
0.95
0.94
0.93
0.00
1.01
1.00
2.93
3.08
2.07
1.12
6.01
f1 (ppm)

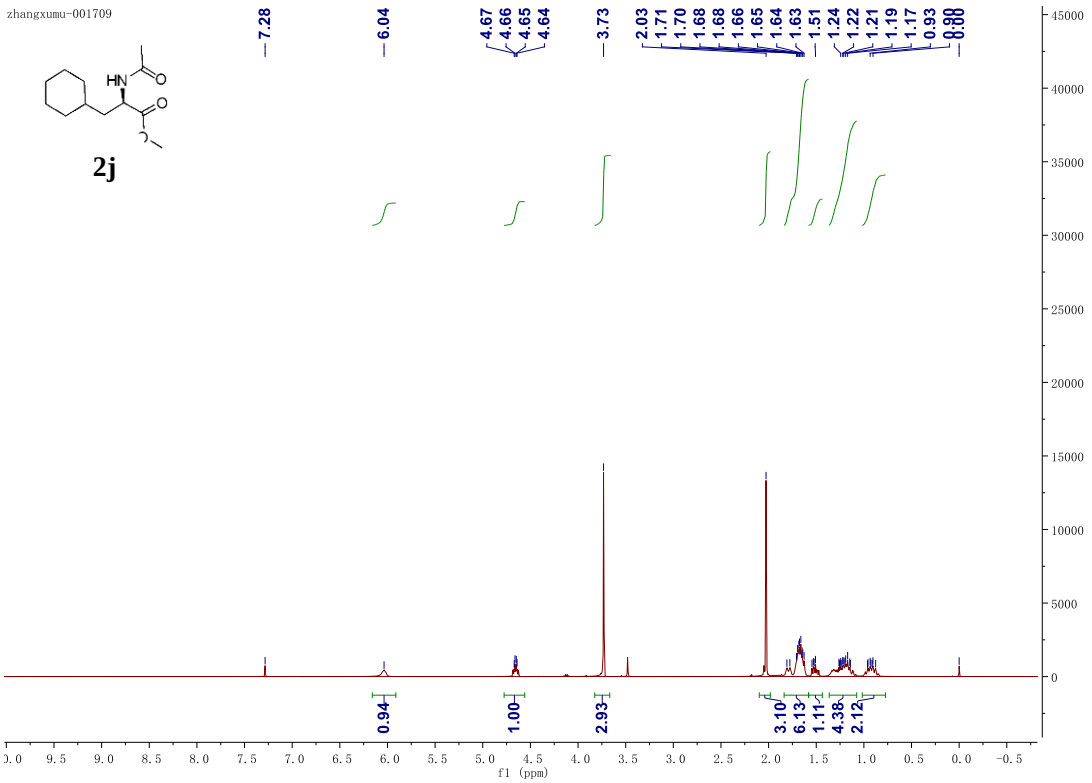

zhangxumu-001712
2g
173.75
169.96
52.23
50.65
41.57
24.79
23.04
22.72
21.89
f1 (ppm)

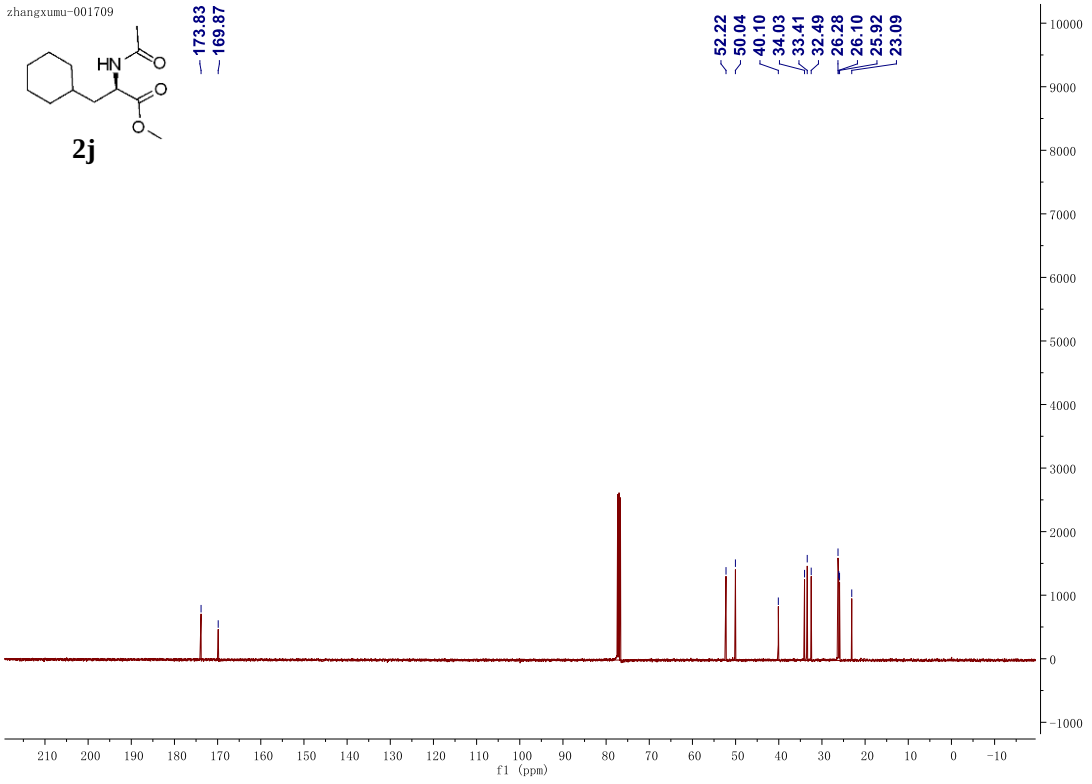

zhangxumu-001713
2h
6.07
4.64
4.63
3.74
2.02
1.70
1.68
1.54
1.39
1.37
1.35
1.34
1.32
1.30
1.29
1.26
0.87
0.85
0.84
0.82
0.92
1.00
2.85
2.93
1.13
1.03
5.05
5.74
f1 (ppm)

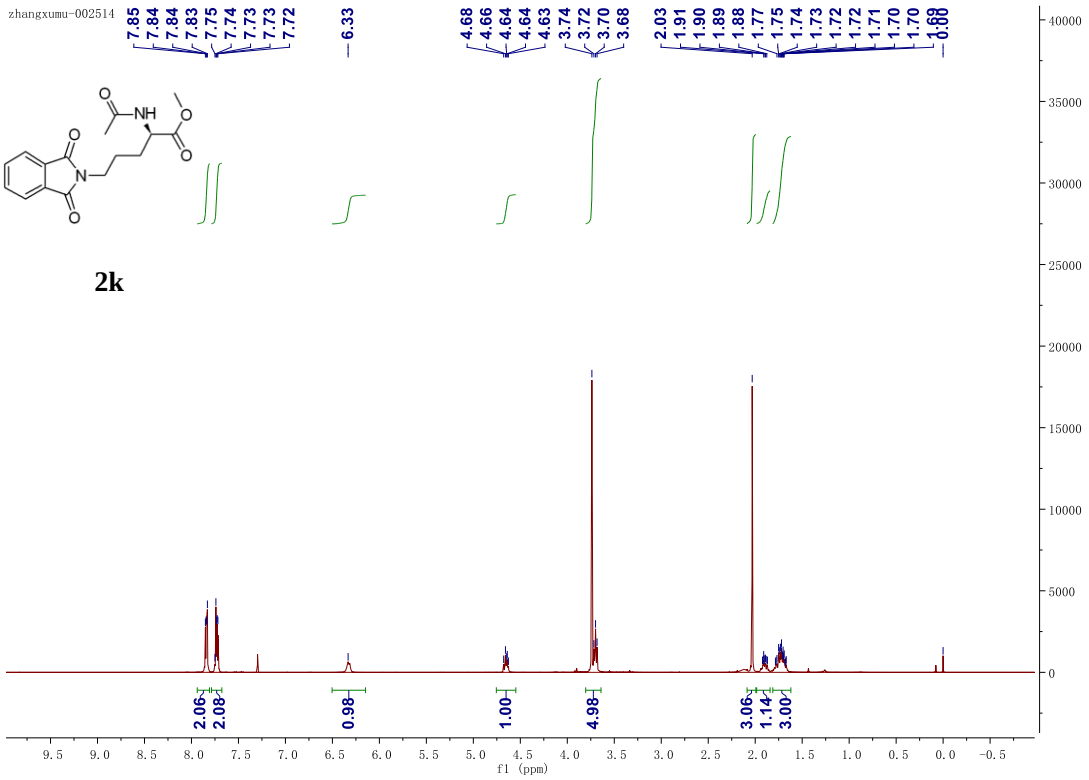

zhangxumu-001713
2h
173.89
169.89
52.21
50.46
36.61
36.18
25.28
24.55
23.06
10.60
10.10
f1 (ppm)


zhangxumu-002568
2i
6.29
4.72
4.70
4.70
4.69
4.68
4.67
3.76
2.04
1.76
1.74
1.72
1.70
1.69
1.67
1.65
0.49
0.49
0.48
0.48
0.07
0.06
0.96
0.99
3.00
3.14
2.08
1.01
2.00
2.02
f1 (ppm)

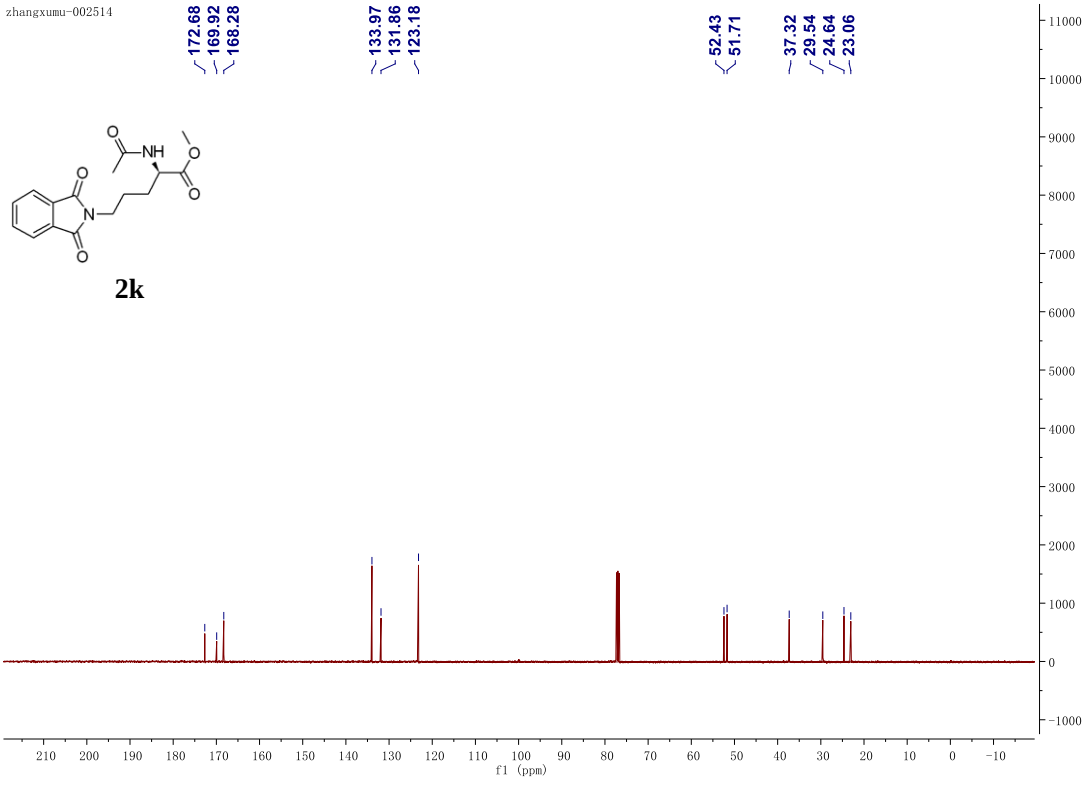

zhangxumu-001714
2i
173.06
169.67
52.51
52.21
37.00
23.08
6.78
4.09
3.97
f1 (ppm)


zhangxumu-001709
2j
7.28
6.04
4.67
4.66
4.65
4.64
3.73
2.03
1.71
1.70
1.68
1.68
1.66
1.65
1.64
1.63
1.51
1.24
1.22
1.21
1.19
1.17
0.93
0.90
0.88
0.94
1.00
2.93
3.10
6.13
1.11
4.38
2.12
f1 (ppm)

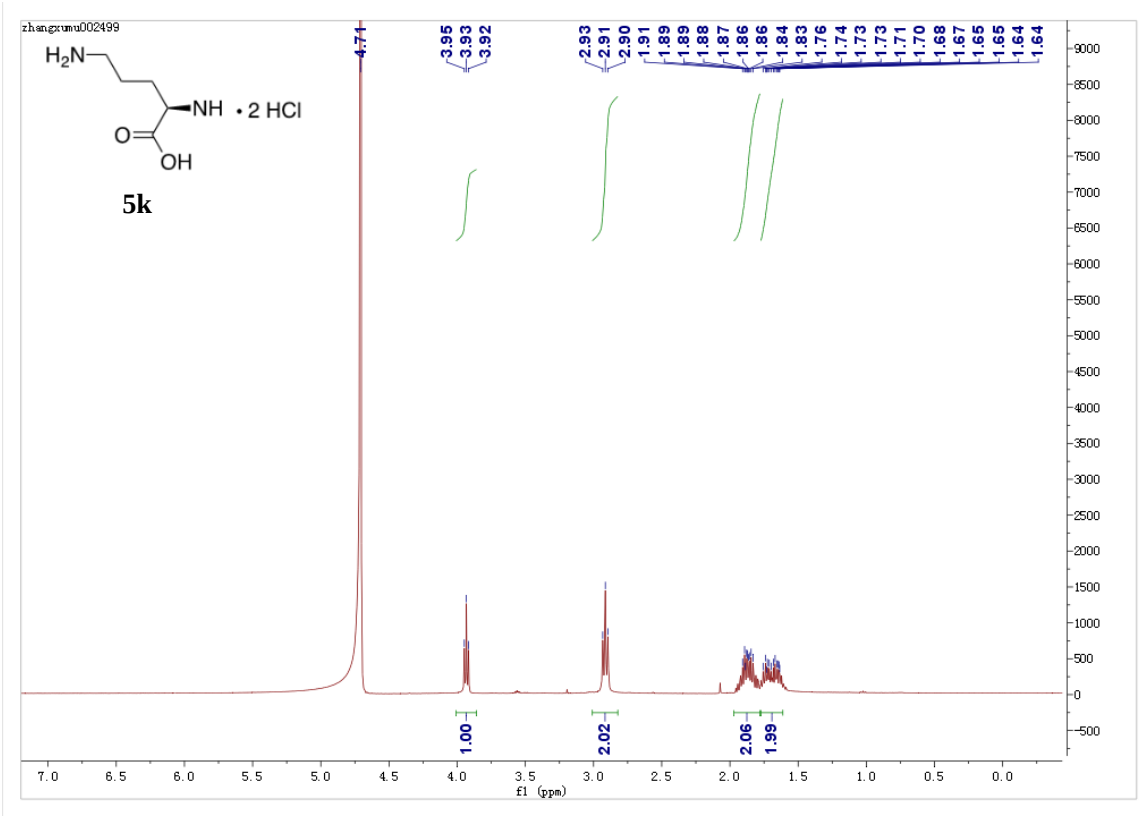

zhangxumu-001709
2j
173.83
169.87
52.22
50.04
40.10
34.03
33.41
32.49
26.28
26.10
25.92
23.09
f1 (ppm)

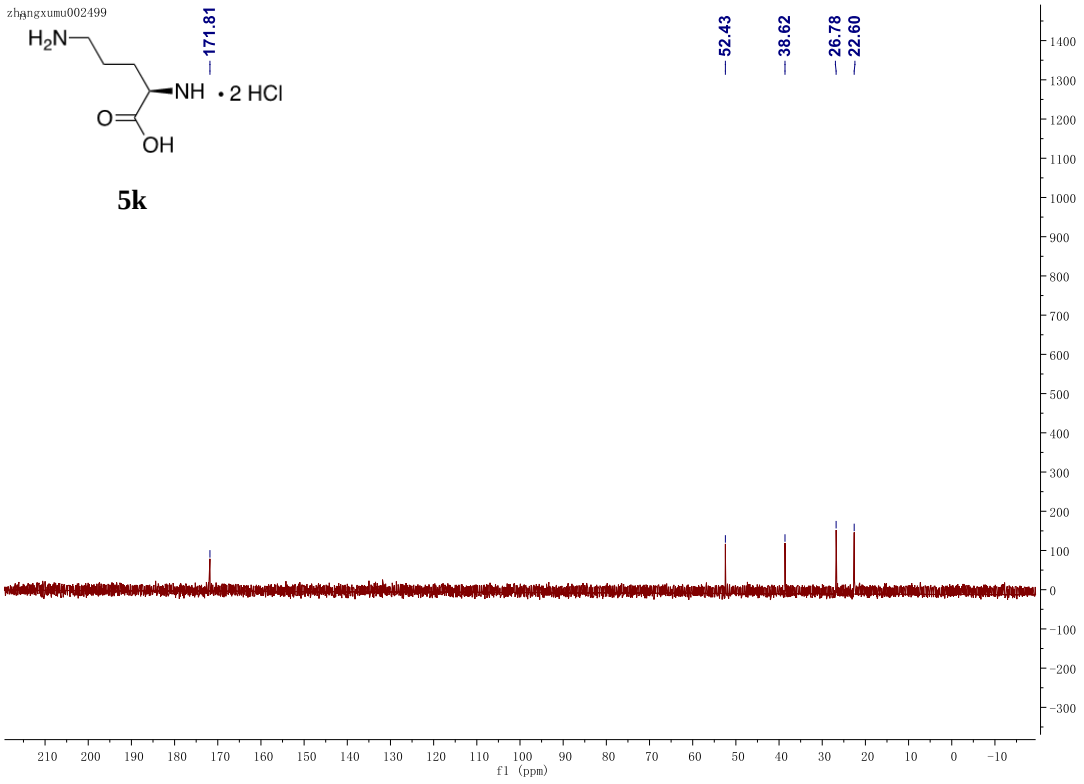

zhangxumu-002514
7.85
7.84
7.84
7.83
7.75
7.74
7.73
7.73
7.72
6.33
4.68
4.66
4.64
4.64
4.63
3.74
3.72
3.70
3.68
2.03
1.91
1.90
1.89
1.88
1.77
1.75
1.74
1.73
1.72
1.72
1.71
1.70
1.70
1.69
1.68
NH
O
N
2.06
2.08
0.98
1.00
4.98
3.06
1.14
3.00
f1 (ppm)

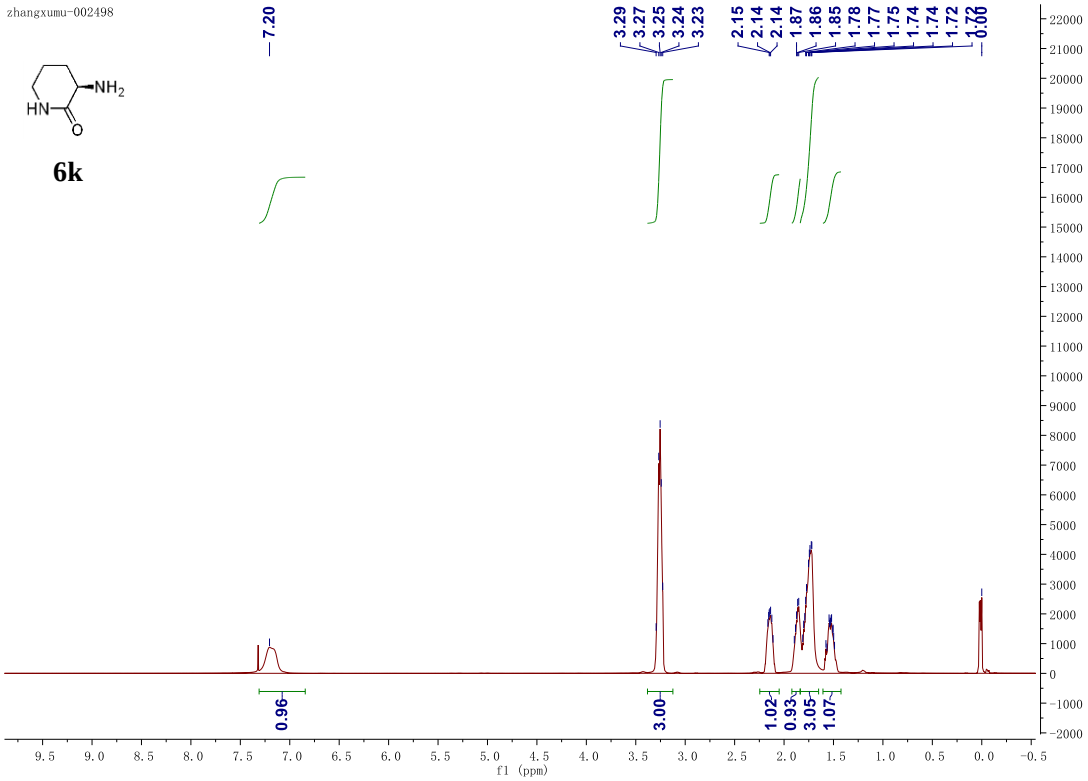
2k

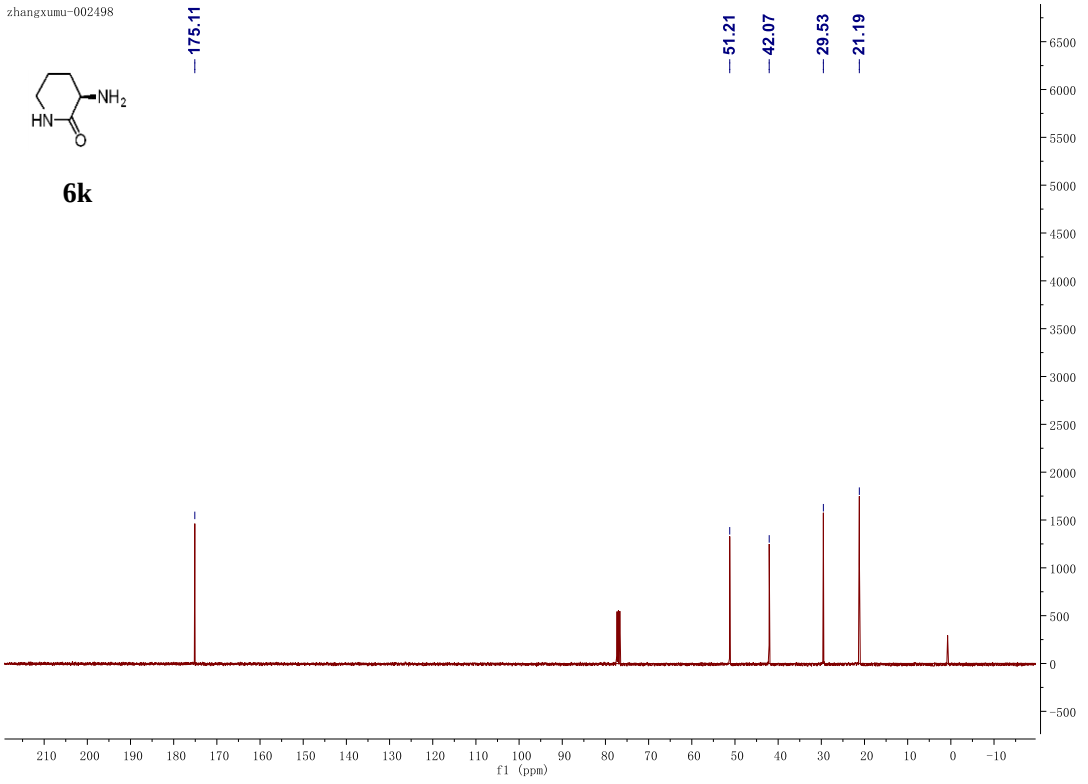

zhangxumu-002514
172.68
169.92
168.28
133.97
131.86
123.18
52.43
51.71
37.32
29.54
24.64
23.06
NH
O
N
f1 (ppm)

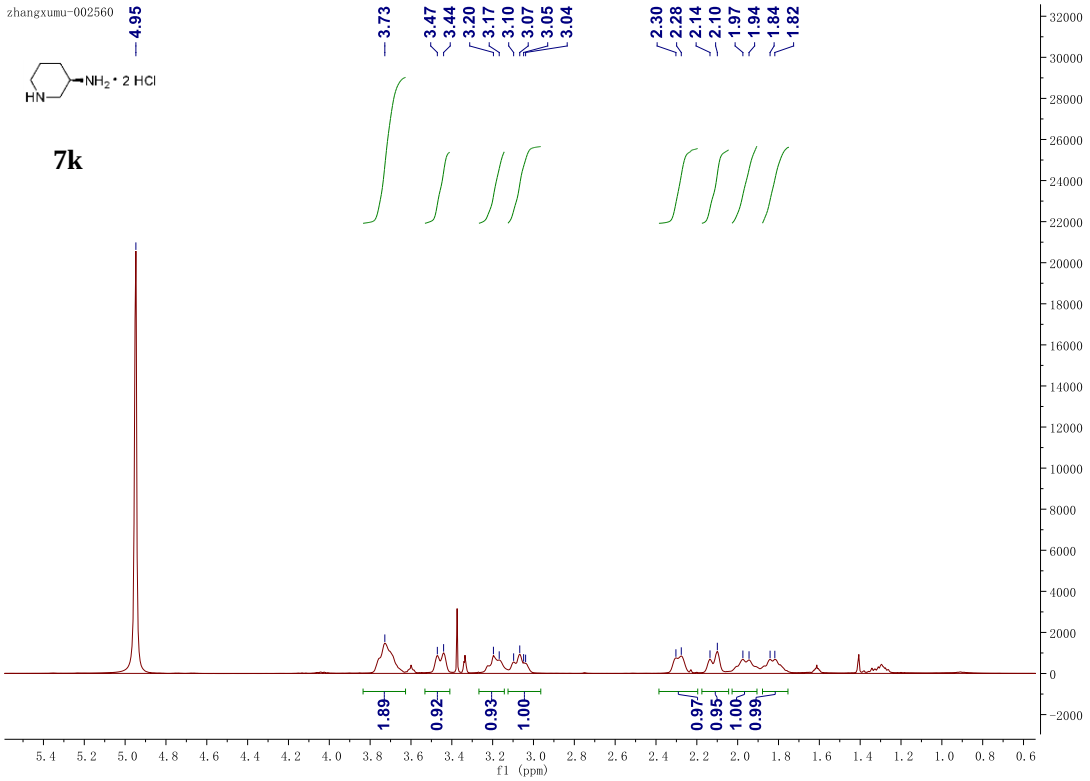
2k

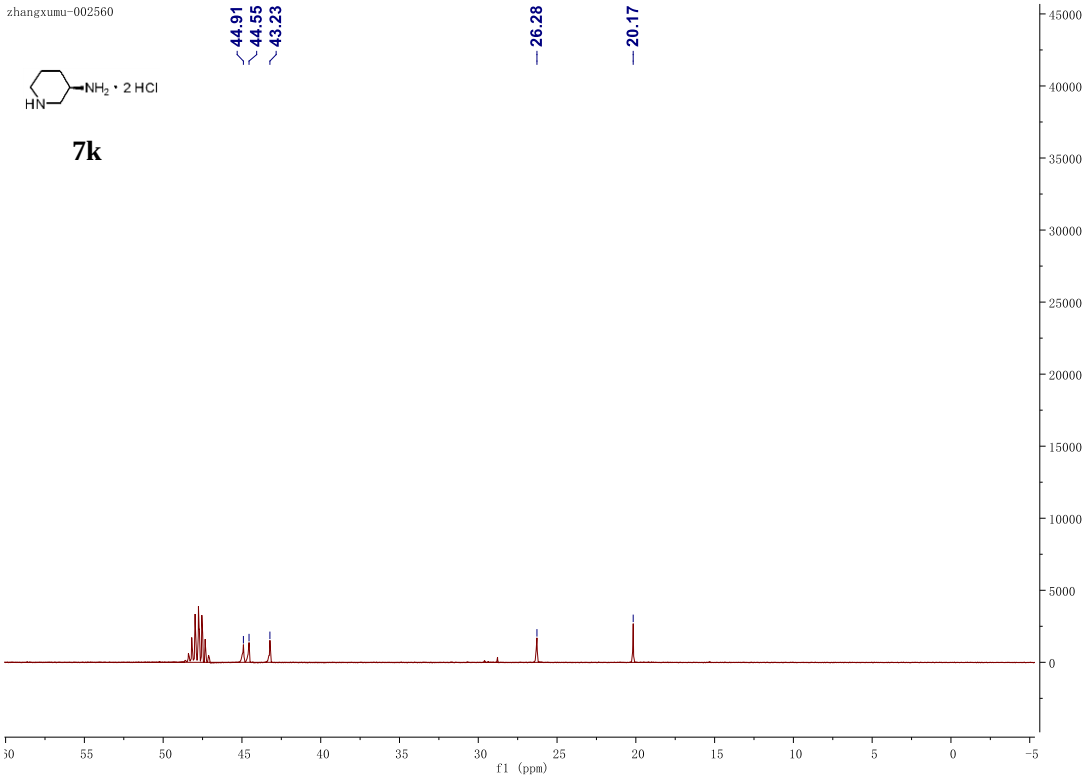

zhangxumu002499
H2N
NH • 2 HCl
O
OH
5k
4.71
3.95
3.93
3.92
2.93
2.91
2.90
1.91
1.89
1.89
1.88
1.87
1.86
1.86
1.84
1.83
1.76
1.74
1.73
1.73
1.71
1.70
1.68
1.67
1.65
1.65
1.64
1.64
1.00
2.02
2.06
1.99
f1 (ppm)


zhangxumu002499
H2N
NH • 2 HCl
O
OH
5k
171.81
52.43
38.62
26.78
22.60
f1 (ppm)


zhangxumu-002498
6k
7.20
3.29
3.27
3.25
3.24
3.23
2.15
2.14
2.14
1.87
1.86
1.85
1.78
1.77
1.75
1.74
1.74
1.72
0.00
0.96
3.00
1.02
0.93
3.05
1.07
f1 (ppm)

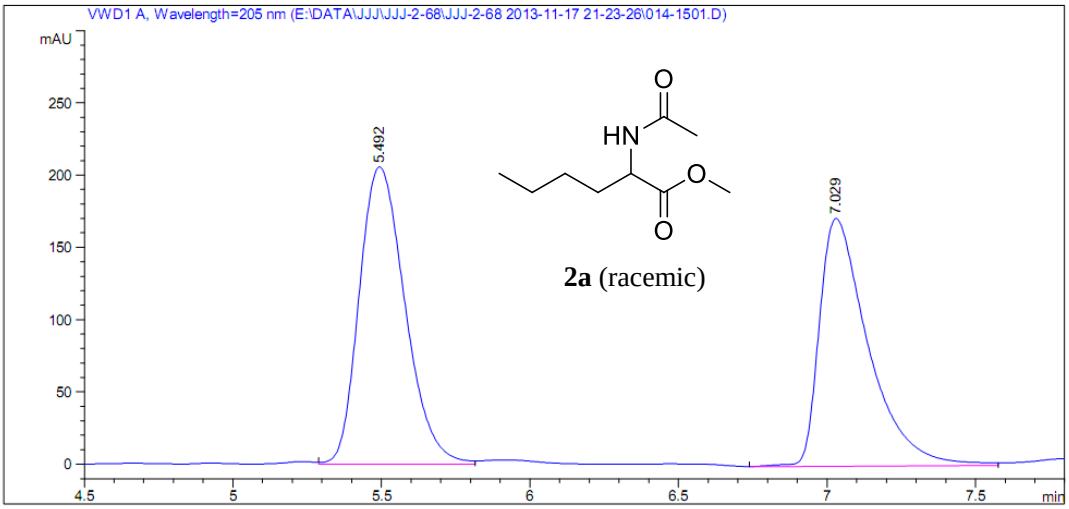

zhangxumu-002498
6k
175.11
51.21
42.07
29.53
21.19
f1 (ppm)


zhangxumu-002560
4.95
3.73
3.47
3.44
3.20
3.17
3.10
3.07
3.05
3.04
2.30
2.28
2.14
2.10
1.97
1.94
1.84
1.82
NH2 • 2 HCl
HN
7k
1.89
0.92
0.93
1.00
0.97
0.95
1.00
0.99
f1 (ppm)


zhangxumu-002560
44.91
44.55
43.23
26.28
20.17
NH2 • 2 HCl
HN
7k
f1 (ppm)

HPLC results for determining the enantioselectivity

```
Data File E:\DATA\JJJ\JJJ-2-68\JJJ-2-68 2013-11-17 21-23-26\014-1501.D
Sample Name: JJJ-2-57-7

    ======================================================================
    Acq. Operator   : SYSTEM                       Seq. Line :  15
    Acq. Instrument : 1260HPLC-VWD                  Location : Vial 14
    Injection Date  : 11/18/2013 1:00:46 AM              Inj :  1
                                                  Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\JJJ\JJJ-2-68\JJJ-2-68 2013-11-17 21-23-26\VWD-ADH-90-10-1ML-205NM-
                      15MIN.M
    Last changed    : 11/17/2013 9:23:26 PM by SYSTEM
    Analysis Method : E:\DATA\JJJ\JJJ-2-68\JJJ-2-68 2013-11-17 21-23-26\VWD-ADH-90-10-1ML-205NM-
                      15MIN.M (Sequence Method)
    Last changed    : 5/5/2014 11:03:13 AM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```

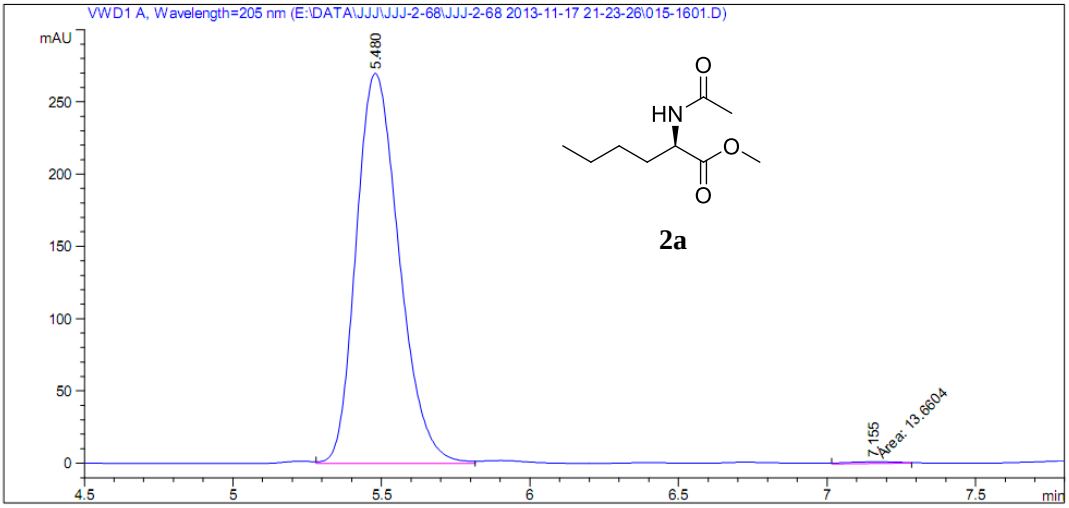


2a (racemic)

```
    ======================================================================
                              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=205 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]    [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   5.492 VV    0.1680 2202.27515  205.54398  52.8199
       2   7.029 BV    0.1718 1967.13171  171.47842  47.1801

    Totals :                   4169.40686  377.02240

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

1260HPLC-DAD 5/5/2014 11:03:17 AM SYSTEM                          Page   1 of 1
```

Data File E:\DATA\JJJ\JJJ-2-68\JJJ-2-68 2013-11-17 21-23-26\015-1601.D
Sample Name: JJJ-2-68-8

```
==========================================================================
Acq. Operator   : SYSTEM                        Seq. Line :  16
Acq. Instrument : 1260HPLC-VWD                  Location : Vial 15
Injection Date  : 11/18/2013 1:16:32 AM              Inj :   1
                                               Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\JJJ\JJJ-2-68\JJJ-2-68 2013-11-17 21-23-26\VWD-ADH-90-10-1ML-205NM-
                  15MIN.M
Last changed    : 11/17/2013 9:23:26 PM by SYSTEM
Analysis Method : E:\DATA\JJJ\JJJ-2-68\JJJ-2-68 2013-11-17 21-23-26\VWD-ADH-90-10-1ML-205NM-
                  15MIN.M (Sequence Method)
Last changed    : 5/5/2014 11:03:13 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```

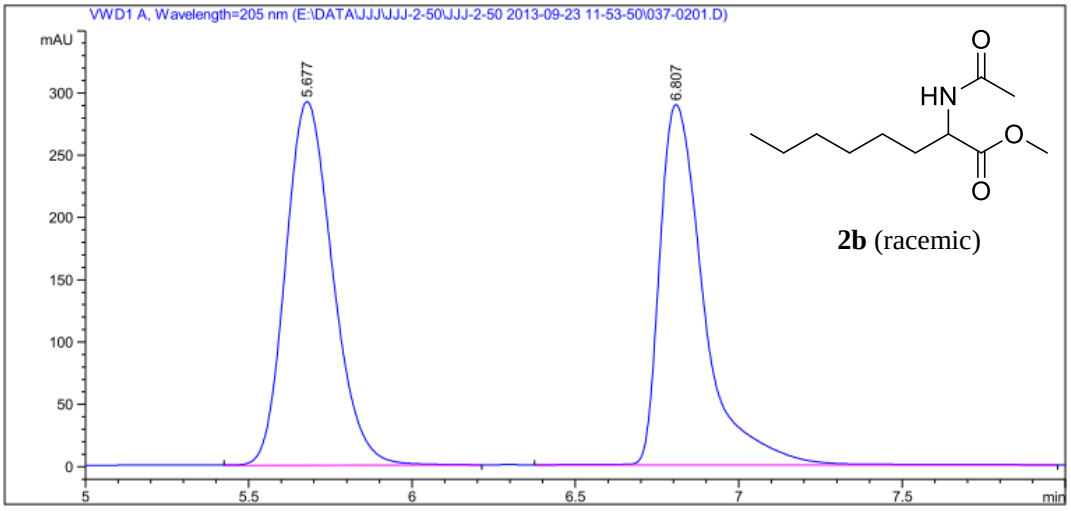


2a

```
==========================================================================
                        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=205 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   5.480 VV    0.1577 2724.71558  270.10306  99.5012
   2   7.155 MM T  0.1990   13.66038    1.14437   0.4988

Totals :                  2738.37595  271.24743


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

Data File E:\DATA\JJJ\JJJ-2-50\JJJ-2-50 2013-09-23 11-53-50\037-0201.D
Sample Name: JJJ-2-50

```
==========================================================================
Acq. Operator   : SYSTEM                          Seq. Line :   2
Acq. Instrument : 1260HPLC-VWD                    Location : Vial 37
Injection Date  : 9/23/2013 12:05:29 PM              Inj :   1
                                                  Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\JJJ\JJJ-2-50\JJJ-2-50 2013-09-23 11-53-50\VWD-ADH-90-10-1ML-205NM-
                  25MIN.M
Last changed    : 9/23/2013 11:53:50 AM by SYSTEM
Analysis Method : E:\DATA\JJJ\JJJ-2-50\JJJ-2-50 2013-09-23 11-53-50\VWD-ADH-90-10-1ML-205NM-
                  25MIN.M (Sequence Method)
Last changed    : 8/16/2014 12:22:33 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```

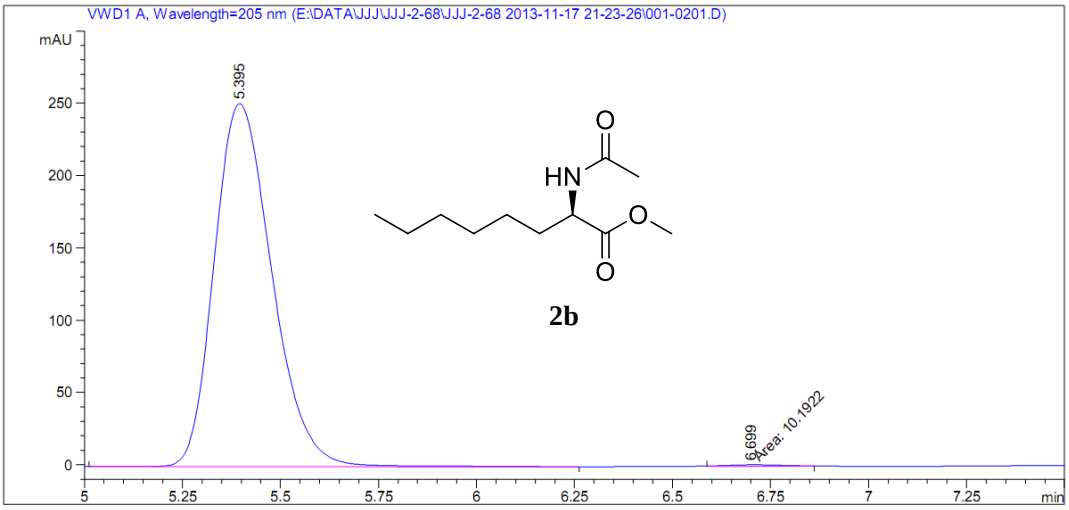


2b (racemic)

```
==========================================================================
                        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=205 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   5.677 VB    0.1547 2920.13965  291.84036  51.8225
   2   6.807 BB    0.1465 2714.74512  289.32983  48.1775

Totals :                  5634.88477  581.17020


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

Data File E:\DATA\JJJ\JJJ-2-68\JJJ-2-68 2013-11-17 21-23-26\001-0201.D
Sample Name: JJJ-2-68-1

```
======================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   2
Acq. Instrument : 1260HPLC-VWD                   Location : Vial 1
Injection Date  : 11/17/2013 9:35:59 PM               Inj :   1
                                                Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\JJJ\JJJ-2-68\JJJ-2-68 2013-11-17 21-23-26\VWD-ADH-90-10-1ML-205NM-
                  15MIN.M
Last changed    : 11/17/2013 9:23:26 PM by SYSTEM
Analysis Method : E:\DATA\JJJ\JJJ-2-68\JJJ-2-68 2013-11-17 21-23-26\VWD-ADH-90-10-1ML-205NM-
                  15MIN.M (Sequence Method)
Last changed    : 1/21/2014 8:33:14 PM by SYSTEM
Additional Info : Peak(s) manually integrated
```

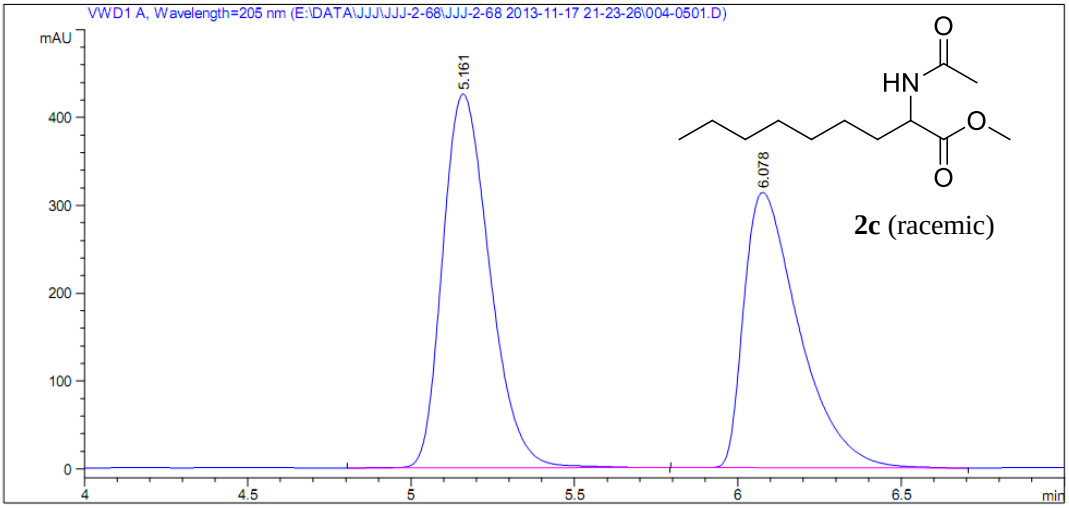


2b

```
======================================================================
                        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=205 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   5.395 BB    0.1602 2563.71948  251.02618  99.6040
   2   6.699 MM T  0.1646   10.19225    1.03179   0.3960

Totals :                  2573.91173  252.05797

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

Data File E:\DATA\JJJ\JJJ-2-68\JJJ-2-68 2013-11-17 21-23-26\004-0501.D
Sample Name: JJJ-2-57-2

```
==================================================================
Acq. Operator   : SYSTEM                       Seq. Line :   5
Acq. Instrument : 1260HPLC-VWD                  Location : Vial 4
Injection Date  : 11/17/2013 10:23:12 PM             Inj :   1
                                               Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\JJJ\JJJ-2-68\JJJ-2-68 2013-11-17 21-23-26\VWD-ADH-90-10-1ML-205NM-
                  15MIN.M
Last changed    : 11/17/2013 9:23:26 PM by SYSTEM
Analysis Method : E:\DATA\JJJ\JJJ-2-68\JJJ-2-68 2013-11-17 21-23-26\VWD-ADH-90-10-1ML-205NM-
                  15MIN.M (Sequence Method)
Last changed    : 5/5/2014 10:55:44 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```

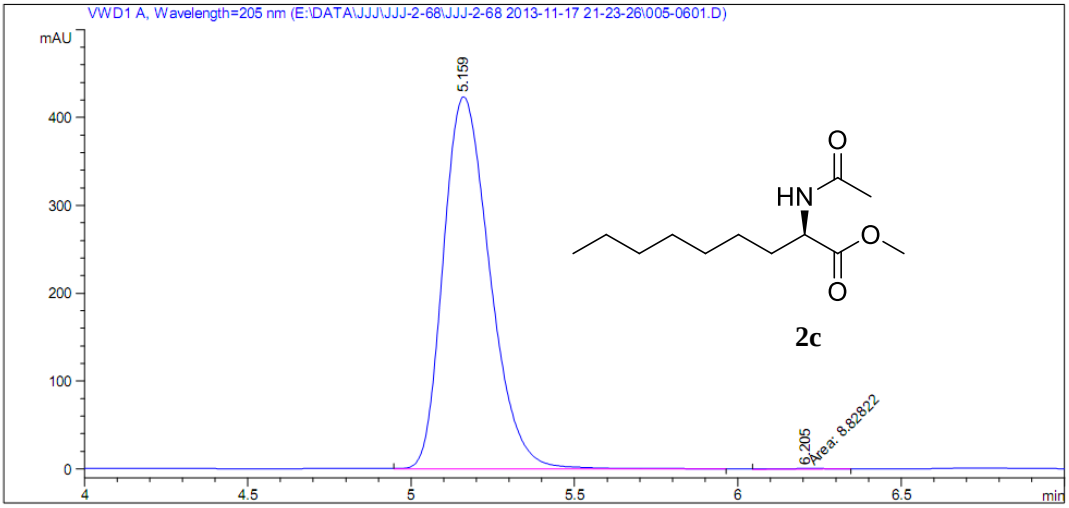


2c (racemic)

```
==================================================================
                      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=205 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   5.161 BB    0.1568 4262.05908  425.60657  53.6589
   2   6.078 BB    0.1780 3680.81787  313.50177  46.3411

Totals :                  7942.87695  739.10834

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

Data File E:\DATA\JJJ\JJJ-2-68\JJJ-2-68 2013-11-17 21-23-26\005-0601.D
Sample Name: JJJ-2-68-3

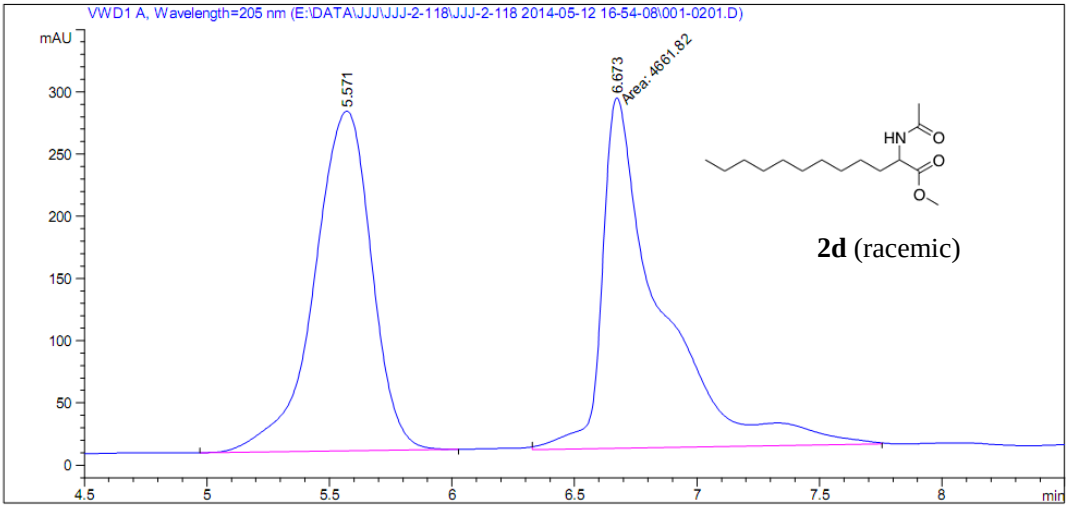
```
==========================================================================
Acq. Operator   : SYSTEM                          Seq. Line :   6
Acq. Instrument : 1260HPLC-VWD                    Location : Vial 5
Injection Date  : 11/17/2013 10:38:57 PM               Inj :   1
                                                  Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\JJJ\JJJ-2-68\JJJ-2-68 2013-11-17 21-23-26\VWD-ADH-90-10-1ML-205NM-
                  15MIN.M
Last changed    : 11/17/2013 9:23:26 PM by SYSTEM
Analysis Method : E:\DATA\JJJ\JJJ-2-68\JJJ-2-68 2013-11-17 21-23-26\VWD-ADH-90-10-1ML-205NM-
                  15MIN.M (Sequence Method)
Last changed    : 5/5/2014 10:55:44 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: VWD1 A, Wavelength=205 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   5.159 VB    0.1554 4191.18945  423.63522  99.7898
   2   6.205 MM T  0.2339    8.82822 6.20920e-1   0.2102

Totals :                  4200.01767  424.25614


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

Data File E:\DATA\JJJ\JJJ-2-118\JJJ-2-118 2014-05-12 16-54-08\001-0201.D
Sample Name: JJJ-2-118-2

```
==========================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   2
Acq. Instrument : 1260HPLC-VWD                  Location : Vial 1
Injection Date  : 5/12/2014 5:07:42 PM               Inj :   1
                                               Inj Volume : 10.000 µl
Acq. Method     : E:\DATA\JJJ\JJJ-2-118\JJJ-2-118 2014-05-12 16-54-08\VWD-ADH-90-10-1ML-205NM
                  -15MIN.M
Last changed    : 5/12/2014 4:54:08 PM by SYSTEM
Analysis Method : E:\DATA\JJJ\JJJ-2-118\JJJ-2-118 2014-05-12 16-54-08\VWD-ADH-90-10-1ML-205NM
                  -15MIN.M (Sequence Method)
Last changed    : 5/12/2014 6:58:13 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```

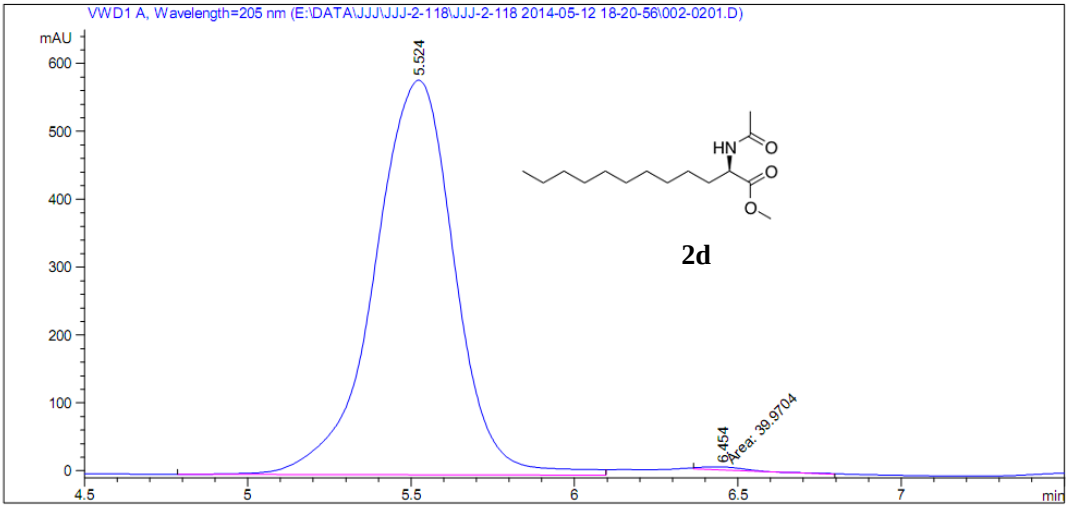


2d (racemic)

```
==========================================================================
                        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=205 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   5.571 BB    0.2550 4448.60303  273.11478  48.8298
   2   6.673 MM T  0.2757 4661.82471  281.82275  51.1702

Totals :                   9110.42773  554.93753


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

Data File E:\DATA\JJJ\JJJ-2-118\JJJ-2-118 2014-05-12 18-20-56\002-0201.D
Sample Name: JJJ-2-118-1

```
==========================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   2
Acq. Instrument : 1260HPLC-VWD                  Location : Vial 2
Injection Date  : 5/12/2014 6:37:30 PM               Inj :   1
                                               Inj Volume : 10.000 µl
Acq. Method     : E:\DATA\JJJ\JJJ-2-118\JJJ-2-118 2014-05-12 18-20-56\VWD-ADH-90-10-1ML-205NM
                  -15MIN.M
Last changed    : 5/12/2014 6:20:56 PM by SYSTEM
Analysis Method : E:\DATA\JJJ\JJJ-2-118\JJJ-2-118 2014-05-12 18-20-56\VWD-ADH-90-10-1ML-205NM
                  -15MIN.M (Sequence Method)
Last changed    : 5/12/2014 6:56:24 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```

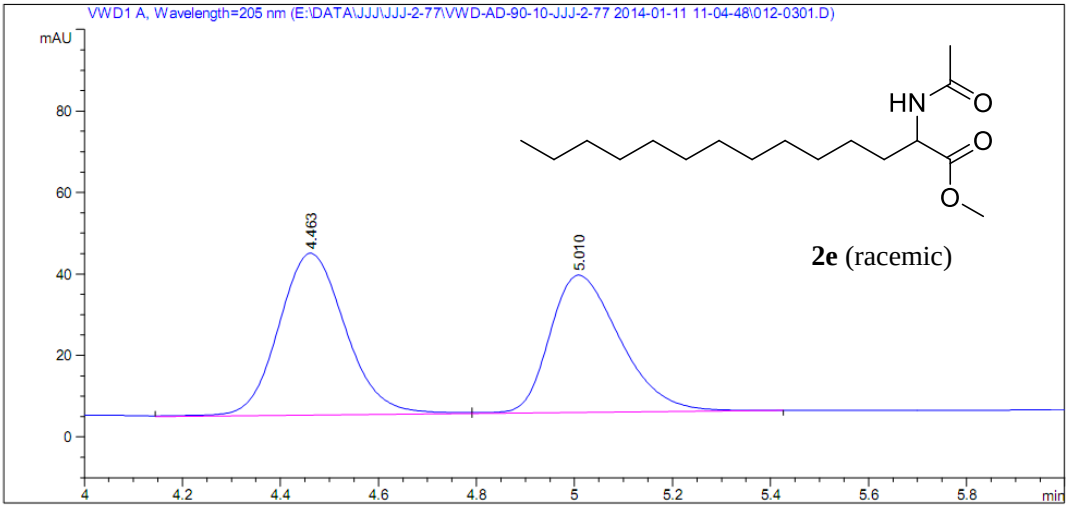


2d

```
==========================================================================
                        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=205 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   5.524 BV    0.2676 9944.33203  581.38959  99.5997
   2   6.454 MM T  0.1586   39.97039    4.19943   0.4003

Totals :                  9984.30242  585.58901


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

Data File E:\DATA\JJJ\JJJ-2-77\VWD-AD-90-10-JJJ-2-77 2014-01-11 11-04-48\012-0301.D
Sample Name: JJJ-2-77-6

```
    ======================================================================
    Acq. Operator   : SYSTEM                       Seq. Line :   3
    Acq. Instrument : 1260HPLC-VWD                  Location : Vial 12
    Injection Date  : 1/11/2014 11:33:06 AM             Inj :   1
                                                   Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\JJJ\JJJ-2-77\VWD-AD-90-10-JJJ-2-77 2014-01-11 11-04-48\VWD-ADH-90-
                      10-1ML-205NM-15MIN.M
    Last changed    : 1/11/2014 11:04:48 AM by SYSTEM
    Analysis Method : E:\DATA\JJJ\JJJ-2-77\VWD-AD-90-10-JJJ-2-77 2014-01-11 11-04-48\VWD-ADH-90-
                      10-1ML-205NM-15MIN.M (Sequence Method)
    Last changed    : 5/5/2014 11:09:06 AM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```

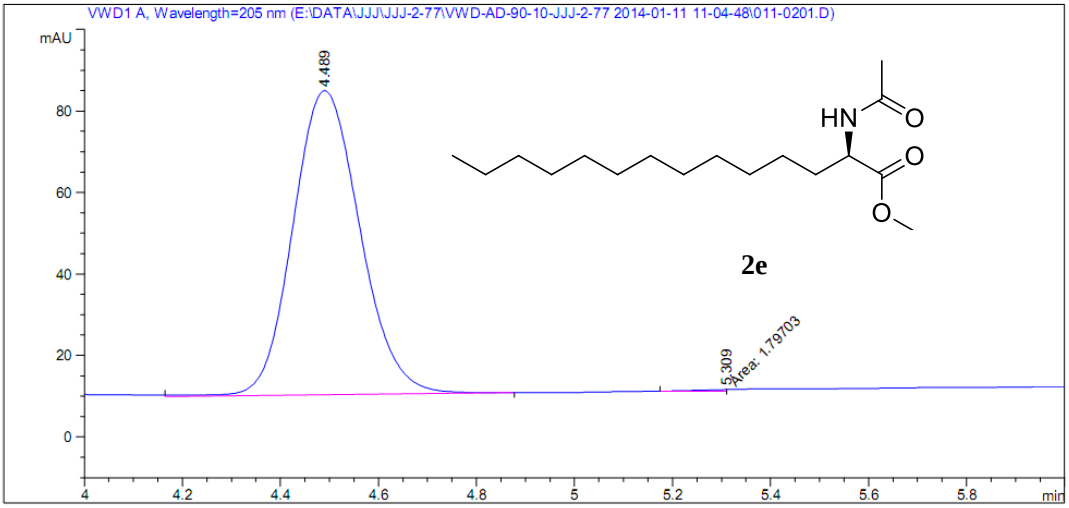


2e (racemic)

```
    ======================================================================
                           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=205 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]    [mAU*s]    [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   4.463 VV    0.1480  378.15582   39.74978  52.8982
       2   5.010 VB    0.1545  336.71829   33.72126  47.1018

    Totals :                    714.87411   73.47104


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

Data File E:\DATA\JJJ\JJJ-2-77\VWD-AD-90-10-JJJ-2-77 2014-01-11 11-04-48\011-0201.D
Sample Name: JJJ-2-77-2

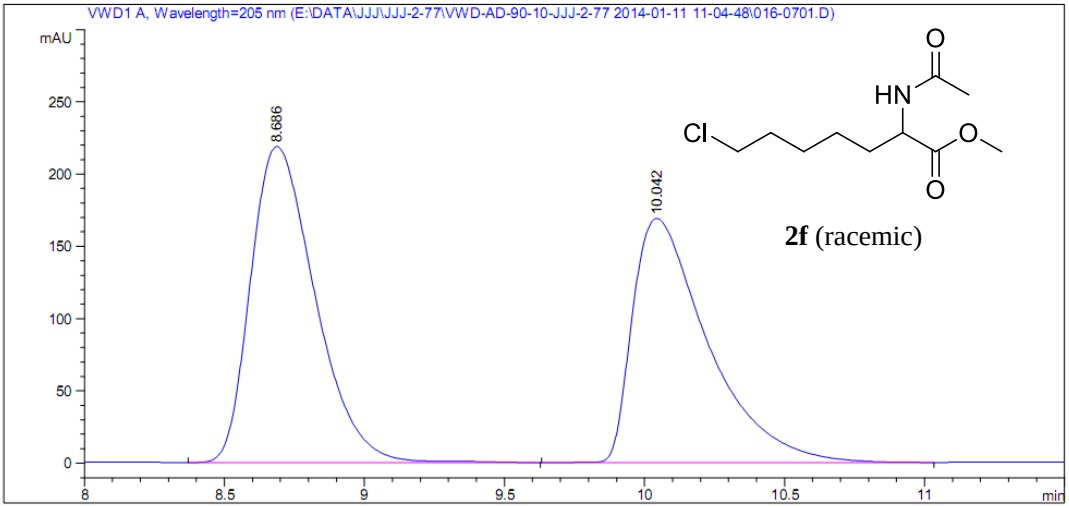
```
=====================================================================
Acq. Operator   : SYSTEM                       Seq. Line :   2
Acq. Instrument : 1260HPLC-VWD                  Location : Vial 11
Injection Date  : 1/11/2014 11:17:22 AM              Inj :   1
                                               Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\JJJ\JJJ-2-77\VWD-AD-90-10-JJJ-2-77 2014-01-11 11-04-48\VWD-ADH-90-
                  10-1ML-205NM-15MIN.M
Last changed    : 1/11/2014 11:04:48 AM by SYSTEM
Analysis Method : E:\DATA\JJJ\JJJ-2-77\VWD-AD-90-10-JJJ-2-77 2014-01-11 11-04-48\VWD-ADH-90-
                  10-1ML-205NM-15MIN.M (Sequence Method)
Last changed    : 5/5/2014 11:06:24 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: VWD1 A, Wavelength=205 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   4.489 VB    0.1488  709.08209   74.65593  99.7472
   2   5.309 MM T  0.0713    1.79703 4.19863e-1   0.2528

Totals :                   710.87913   75.07579


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

Data File E:\DATA\JJJ\JJJ-2-77\VWD-AD-90-10-JJJ-2-77 2014-01-11 11-04-48\016-0701.D
Sample Name: JJJ-2-77-8

```
=====================================================================
Acq. Operator   : SYSTEM                       Seq. Line :   7
Acq. Instrument : 1260HPLC-VWD                  Location : Vial 16
Injection Date  : 1/11/2014 12:36:06 PM              Inj :   1
                                               Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\JJJ\JJJ-2-77\VWD-AD-90-10-JJJ-2-77 2014-01-11 11-04-48\VWD-ADH-90-
                  10-1ML-205NM-15MIN.M
Last changed    : 1/11/2014 11:04:48 AM by SYSTEM
Analysis Method : E:\DATA\JJJ\JJJ-2-77\VWD-AD-90-10-JJJ-2-77 2014-01-11 11-04-48\VWD-ADH-90-
                  10-1ML-205NM-15MIN.M (Sequence Method)
Last changed    : 5/5/2014 11:24:58 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```

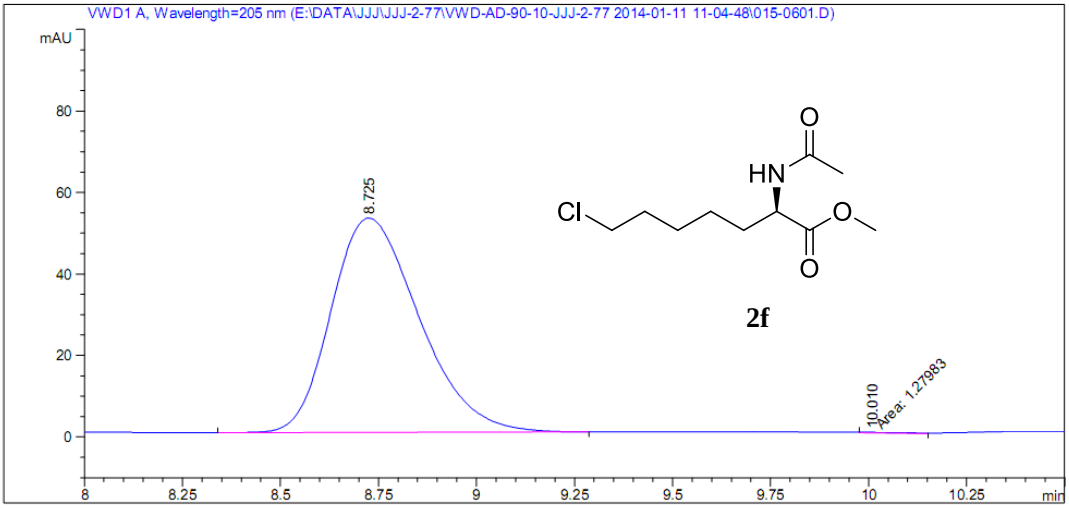


2f (racemic)

```
=====================================================================
                        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=205 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   8.686 VB    0.2478 3503.48950  218.82393  52.5610
   2  10.042 BB    0.2814 3162.08496  168.91719  47.4390

Totals :                  6665.57446  387.74112


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

1260HPLC-DAD 5/5/2014 11:25:14 AM SYSTEM                    Page   1 of 1
```

Data File E:\DATA\JJJ\JJJ-2-77\VWD-AD-90-10-JJJ-2-77 2014-01-11 11-04-48\015-0601.D
Sample Name: JJJ-2-77-4

```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   6
    Acq. Instrument : 1260HPLC-VWD                   Location : Vial 15
    Injection Date  : 1/11/2014 12:20:20 PM              Inj :   1
                                                   Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\JJJ\JJJ-2-77\VWD-AD-90-10-JJJ-2-77 2014-01-11 11-04-48\VWD-ADH-90-
                      10-1ML-205NM-15MIN.M
    Last changed    : 1/11/2014 11:04:48 AM by SYSTEM
    Analysis Method : E:\DATA\JJJ\JJJ-2-77\VWD-AD-90-10-JJJ-2-77 2014-01-11 11-04-48\VWD-ADH-90-
                      10-1ML-205NM-15MIN.M (Sequence Method)
    Last changed    : 5/5/2014 11:10:22 AM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```

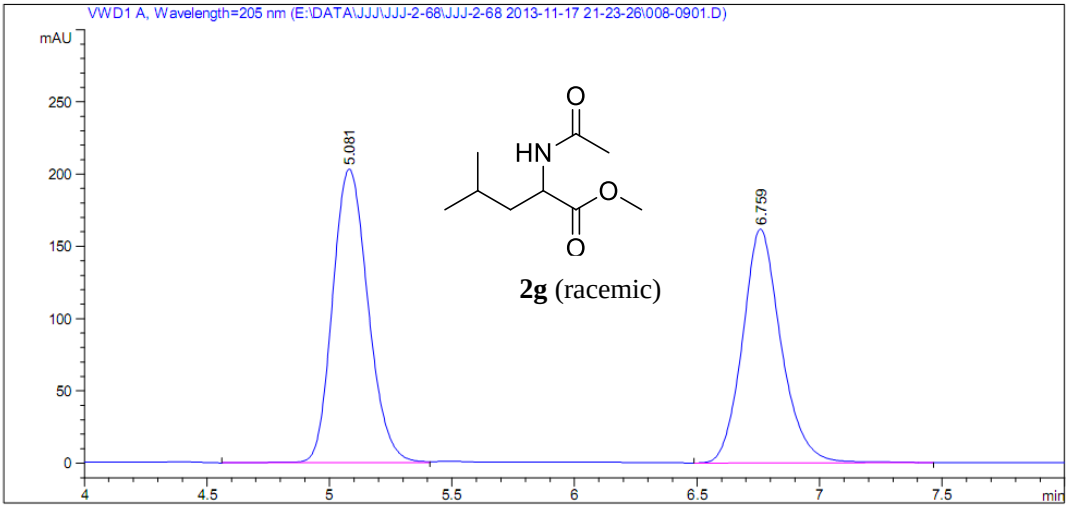


2f

```
    ======================================================================
                           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=205 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]    [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   8.725 BB    0.2433  822.72766   52.65119  99.8447
       2  10.010 MM T  0.1576    1.27983 1.35367e-1   0.1553

    Totals :                   824.00749   52.78656


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

1260HPLC-DAD 5/5/2014 11:10:28 AM SYSTEM                        Page   1 of 1
```

Data File E:\DATA\JJJ\JJJ-2-68\JJJ-2-68 2013-11-17 21-23-26\008-0901.D
Sample Name: JJJ-2-57-4

```
==================================================================
Acq. Operator   : SYSTEM                       Seq. Line :   9
Acq. Instrument : 1260HPLC-VWD                  Location : Vial 8
Injection Date  : 11/17/2013 11:26:16 PM             Inj :   1
                                               Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\JJJ\JJJ-2-68\JJJ-2-68 2013-11-17 21-23-26\VWD-ADH-90-10-1ML-205NM-
                  15MIN.M
Last changed    : 11/17/2013 9:23:26 PM by SYSTEM
Analysis Method : E:\DATA\JJJ\JJJ-2-68\JJJ-2-68 2013-11-17 21-23-26\VWD-ADH-90-10-1ML-205NM-
                  15MIN.M (Sequence Method)
Last changed    : 5/5/2014 10:58:20 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```

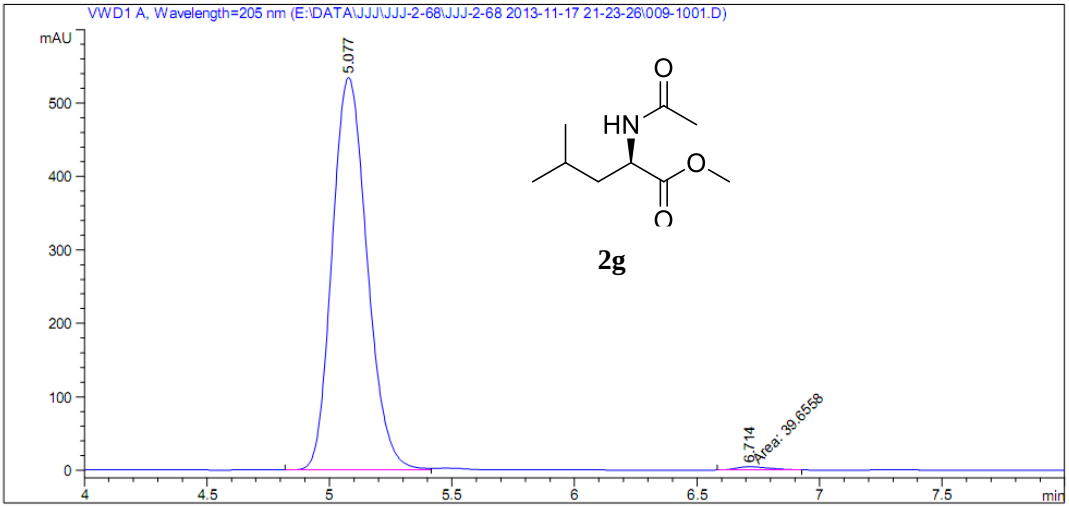


2g (racemic)

```
==================================================================
                        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=205 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   5.081 BV    0.1571 2039.38867  203.11372  53.5222
   2   6.759 BB    0.1627 1770.96899  161.81169  46.4778

Totals :                  3810.35767  364.92542


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

Data File E:\DATA\JJJ\JJJ-2-68\JJJ-2-68 2013-11-17 21-23-26\009-1001.D
Sample Name: JJJ-2-68-5

```
==========================================================================
Acq. Operator   : SYSTEM                        Seq. Line :  10
Acq. Instrument : 1260HPLC-VWD                  Location : Vial 9
Injection Date  : 11/17/2013 11:42:01 PM             Inj :  1
                                               Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\JJJ\JJJ-2-68\JJJ-2-68 2013-11-17 21-23-26\VWD-ADH-90-10-1ML-205NM-
                  15MIN.M
Last changed    : 11/17/2013 9:23:26 PM by SYSTEM
Analysis Method : E:\DATA\JJJ\JJJ-2-68\JJJ-2-68 2013-11-17 21-23-26\VWD-ADH-90-10-1ML-205NM-
                  15MIN.M (Sequence Method)
Last changed    : 5/5/2014 10:59:13 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```

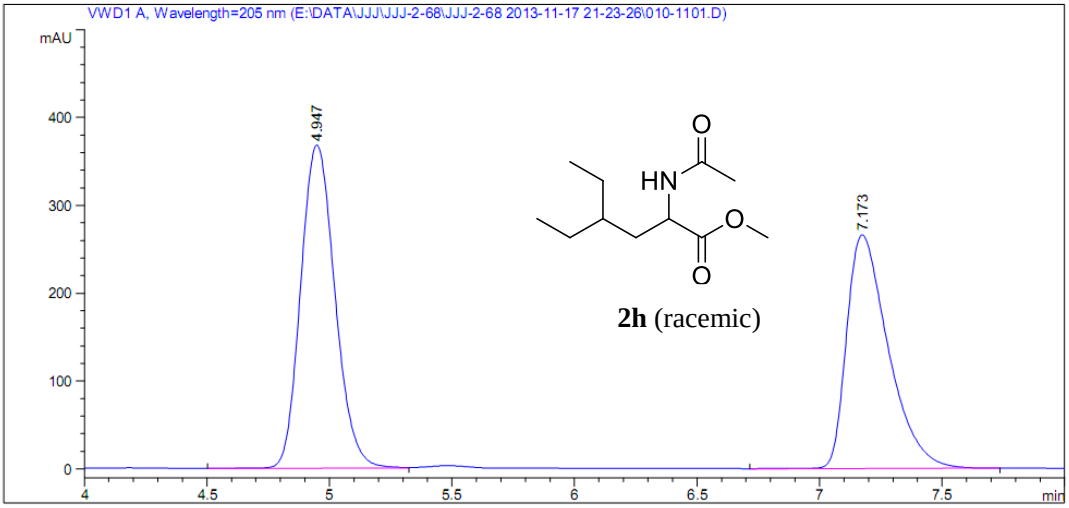


2g

```
==========================================================================
                        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=205 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   5.077 BV    0.1556 5292.67139  534.18994  99.2563
   2   6.714 MM T  0.1546   39.65582    4.27532   0.7437

Totals :                  5332.32720  538.46526


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

Data File E:\DATA\JJJ\JJJ-2-68\JJJ-2-68 2013-11-17 21-23-26\010-1101.D
Sample Name: JJJ-2-57-5

```
    ====================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :  11
    Acq. Instrument : 1260HPLC-VWD                   Location : Vial 10
    Injection Date  : 11/17/2013 11:57:47 PM              Inj :   1
                                                   Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\JJJ\JJJ-2-68\JJJ-2-68 2013-11-17 21-23-26\VWD-ADH-90-10-1ML-205NM-
                      15MIN.M
    Last changed    : 11/17/2013 9:23:26 PM by SYSTEM
    Analysis Method : E:\DATA\JJJ\JJJ-2-68\JJJ-2-68 2013-11-17 21-23-26\VWD-ADH-90-10-1ML-205NM-
                      15MIN.M (Sequence Method)
    Last changed    : 5/5/2014 10:59:48 AM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```

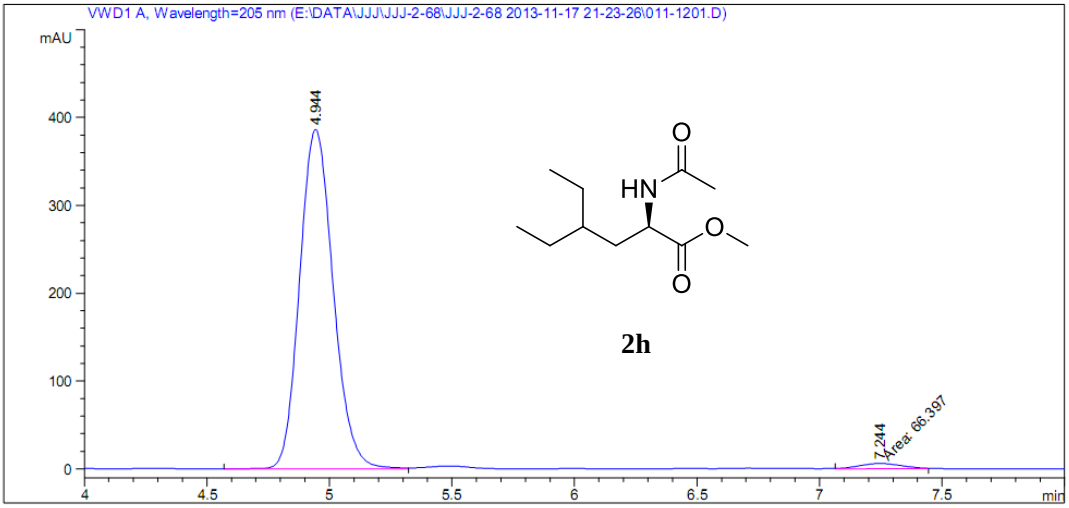


2h (racemic)

```
    ====================================================================
                            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=205 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]    [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   4.947 BV    0.1527 3587.48047  368.09924  53.3337
       2   7.173 BB    0.1807 3138.99536  265.95334  46.6663

    Totals :                   6726.47583  634.05258


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

Data File E:\DATA\JJJ\JJJ-2-68\JJJ-2-68 2013-11-17 21-23-26\011-1201.D
Sample Name: JJJ-2-68-6

```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :  12
    Acq. Instrument : 1260HPLC-VWD                   Location : Vial 11
    Injection Date  : 11/18/2013 12:13:32 AM              Inj :   1
                                                   Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\JJJ\JJJ-2-68\JJJ-2-68 2013-11-17 21-23-26\VWD-ADH-90-10-1ML-205NM-
                      15MIN.M
    Last changed    : 11/17/2013 9:23:26 PM by SYSTEM
    Analysis Method : E:\DATA\JJJ\JJJ-2-68\JJJ-2-68 2013-11-17 21-23-26\VWD-ADH-90-10-1ML-205NM-
                      15MIN.M (Sequence Method)
    Last changed    : 5/5/2014 10:59:48 AM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```

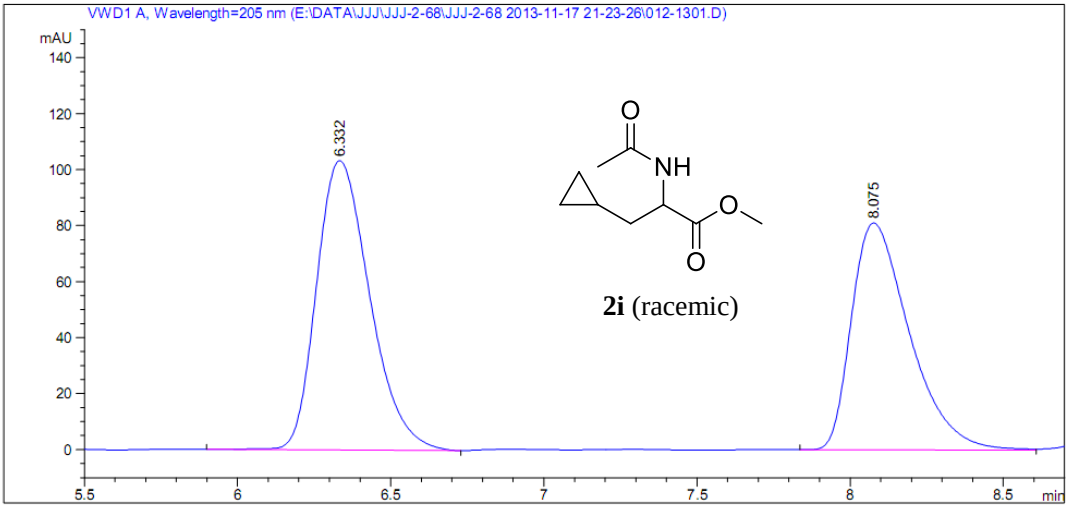


2h

```
    ======================================================================
                           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=205 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   4.944 BV    0.1490 3675.92871  386.27914  98.2258
       2   7.244 MM T  0.1928   66.39699    5.74056   1.7742

    Totals :                  3742.32570  392.01970


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

Data File E:\DATA\JJJ\JJJ-2-68\JJJ-2-68 2013-11-17 21-23-26\012-1301.D
Sample Name: JJJ-2-57-6

```
=====================================================================
Acq. Operator   : SYSTEM                        Seq. Line :  13
Acq. Instrument : 1260HPLC-VWD                  Location : Vial 12
Injection Date  : 11/18/2013 12:29:16 AM             Inj :   1
                                               Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\JJJ\JJJ-2-68\JJJ-2-68 2013-11-17 21-23-26\VWD-ADH-90-10-1ML-205NM-
                  15MIN.M
Last changed    : 11/17/2013 9:23:26 PM by SYSTEM
Analysis Method : E:\DATA\JJJ\JJJ-2-68\JJJ-2-68 2013-11-17 21-23-26\VWD-ADH-90-10-1ML-205NM-
                  15MIN.M (Sequence Method)
Last changed    : 5/5/2014 11:01:23 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```

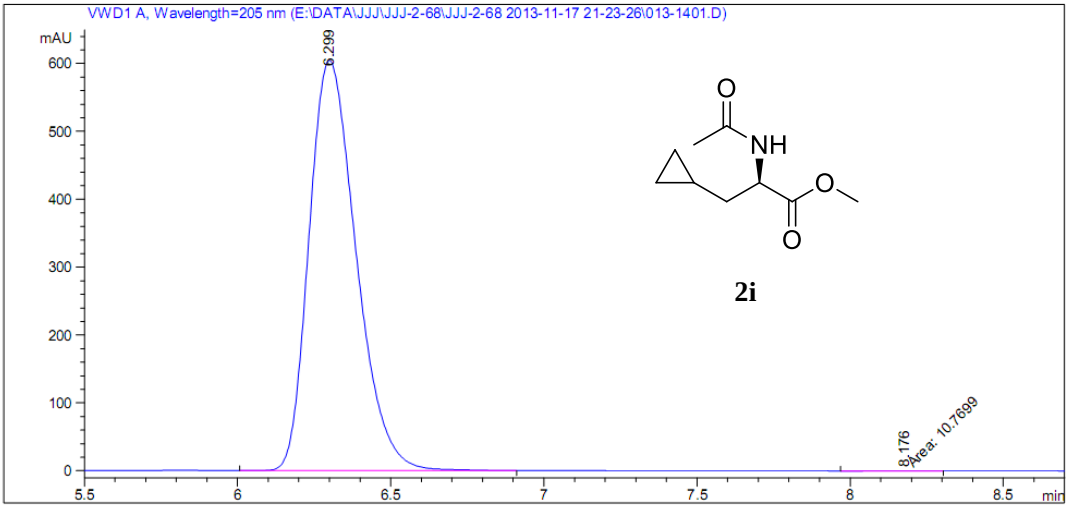


2i (racemic)

```
=====================================================================
                        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=205 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   6.332 BB    0.1878 1247.94849  103.37258  53.9388
   2   8.075 BV    0.2014 1065.68896   81.05479  46.0612

Totals :                  2313.63745  184.42737


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

Data File E:\DATA\JJJ\JJJ-2-68\JJJ-2-68 2013-11-17 21-23-26\013-1401.D
Sample Name: JJJ-2-68-7

```
==========================================================================
Acq. Operator   : SYSTEM                          Seq. Line :  14
Acq. Instrument : 1260HPLC-VWD                    Location : Vial 13
Injection Date  : 11/18/2013 12:45:00 AM               Inj :   1
                                                  Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\JJJ\JJJ-2-68\JJJ-2-68 2013-11-17 21-23-26\VWD-ADH-90-10-1ML-205NM-
                  15MIN.M
Last changed    : 11/17/2013 9:23:26 PM by SYSTEM
Analysis Method : E:\DATA\JJJ\JJJ-2-68\JJJ-2-68 2013-11-17 21-23-26\VWD-ADH-90-10-1ML-205NM-
                  15MIN.M (Sequence Method)
Last changed    : 5/5/2014 11:02:04 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```

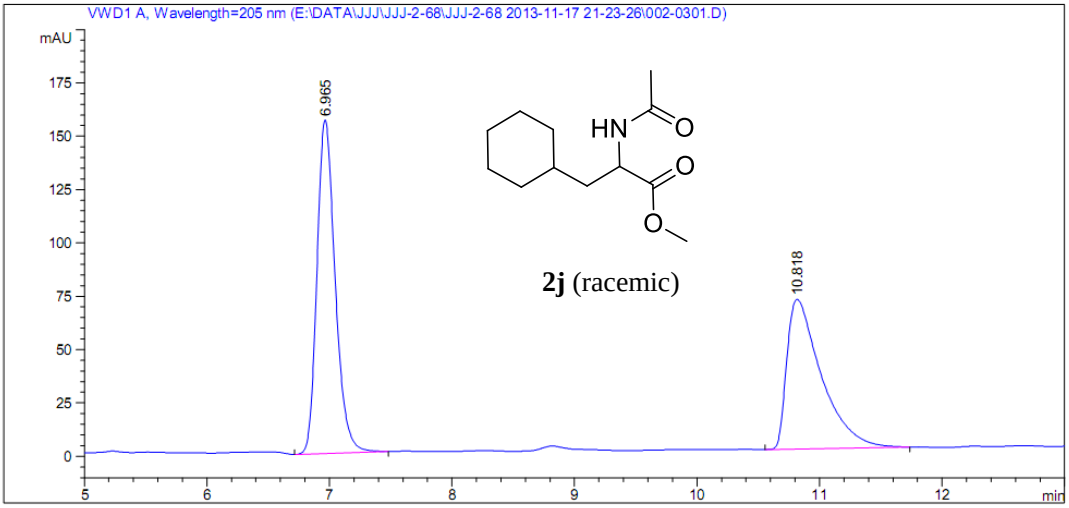


2i

```
==========================================================================
                        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=205 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   6.299 BB    0.1656 6456.27588  604.72894  99.8335
   2   8.176 MM T  0.2524   10.76987 7.34851e-1   0.1665

Totals :                  6467.04575  605.46379


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

Data File E:\DATA\JJJ\JJJ-2-68\JJJ-2-68 2013-11-17 21-23-26\002-0301.D
Sample Name: JJJ-2-57-1

```
==========================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   3
Acq. Instrument : 1260HPLC-VWD                  Location : Vial 2
Injection Date  : 11/17/2013 9:51:44 PM              Inj :   1
                                               Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\JJJ\JJJ-2-68\JJJ-2-68 2013-11-17 21-23-26\VWD-ADH-90-10-1ML-205NM-
                  15MIN.M
Last changed    : 11/17/2013 9:23:26 PM by SYSTEM
Analysis Method : E:\DATA\JJJ\JJJ-2-68\JJJ-2-68 2013-11-17 21-23-26\VWD-ADH-90-10-1ML-205NM-
                  15MIN.M (Sequence Method)
Last changed    : 5/5/2014 10:50:10 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```

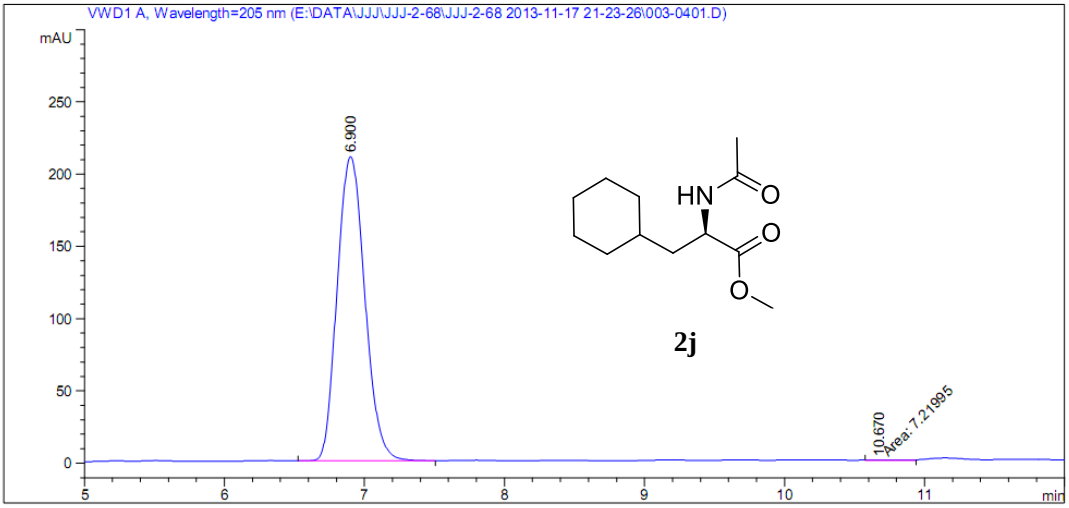


2j (racemic)

```
==========================================================================
                        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=205 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   6.965 BB    0.1593 1611.87964  156.35672  54.1062
   2  10.818 BB    0.2875 1367.22571   70.11797  45.8938

Totals :                  2979.10535  226.47469


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

Data File E:\DATA\JJJ\JJJ-2-68\JJJ-2-68 2013-11-17 21-23-26\003-0401.D
Sample Name: JJJ-2-68-2

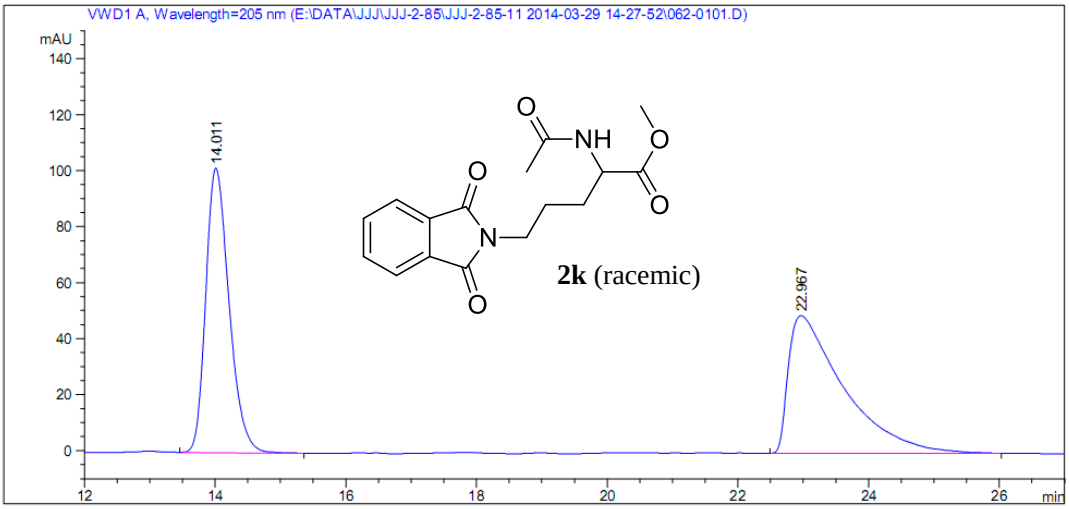
```
==========================================================================
Acq. Operator   : SYSTEM                          Seq. Line :   4
Acq. Instrument : 1260HPLC-VWD                    Location : Vial 3
Injection Date  : 11/17/2013 10:07:28 PM             Inj :   1
                                                  Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\JJJ\JJJ-2-68\JJJ-2-68 2013-11-17 21-23-26\VWD-ADH-90-10-1ML-205NM-
                  15MIN.M
Last changed    : 11/17/2013 9:23:26 PM by SYSTEM
Analysis Method : E:\DATA\JJJ\JJJ-2-68\JJJ-2-68 2013-11-17 21-23-26\VWD-ADH-90-10-1ML-205NM-
                  15MIN.M (Sequence Method)
Last changed    : 5/5/2014 10:53:00 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: VWD1 A, Wavelength=205 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   6.900 BB    0.2180 2890.20972  210.69270  99.7508
   2  10.670 MM T  0.2679    7.21995 4.49148e-1   0.2492

Totals :                  2897.42967  211.14185


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

Data File E:\DATA\JJJ\JJJ-2-85\JJJ-2-85-11 2014-03-29 14-27-52\062-0101.D
Sample Name: JJJ-2-87-1

```
==================================================================
Acq. Operator   : SYSTEM                       Seq. Line :   1
Acq. Instrument : 1260HPLC-VWD                  Location : Vial 62
Injection Date  : 3/29/2014 2:28:34 PM               Inj :   1
                                               Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\JJJ\JJJ-2-85\JJJ-2-85-11 2014-03-29 14-27-52\VWD-AD-80-20-1ML-5UL-
                  205NM-35MIN.M
Last changed    : 3/29/2014 2:57:01 PM by SYSTEM
                  (modified after loading)
Analysis Method : E:\DATA\JJJ\JJJ-2-85\JJJ-2-85-11 2014-03-29 14-27-52\VWD-AD-80-20-1ML-5UL-
                  205NM-35MIN.M (Sequence Method)
Last changed    : 5/5/2014 11:30:08 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```

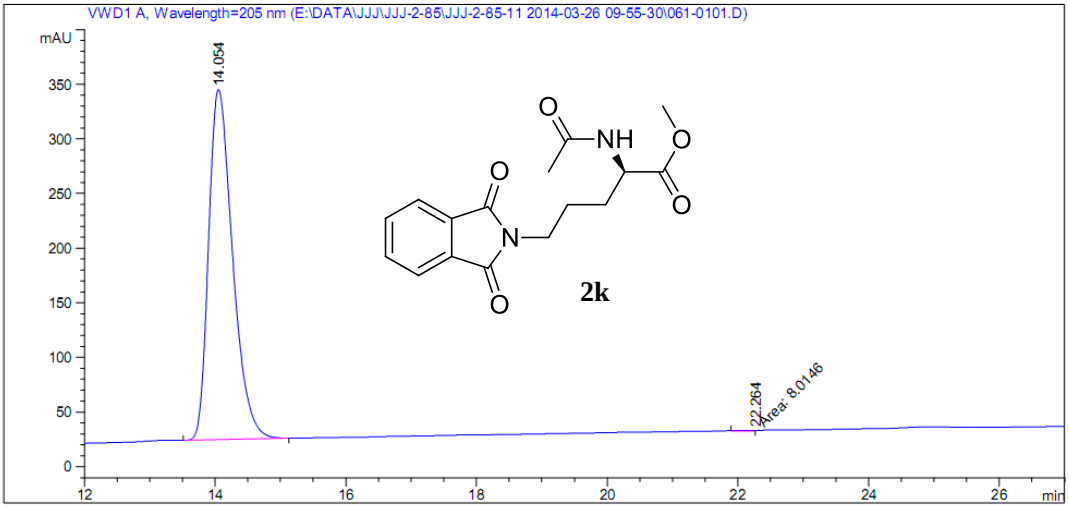


2k (racemic)

```
==================================================================
                        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=205 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  14.011 BB    0.3819 2517.44092  101.66602  46.6356
   2  22.967 BB    0.8249 2880.66455   49.14392  53.3644

Totals :                  5398.10547  150.80994

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

Data File E:\DATA\JJJ\JJJ-2-85\JJJ-2-85-11 2014-03-26 09-55-30\061-0101.D
Sample Name: JJJ-2-85-1

```
==========================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   1
Acq. Instrument : 1260HPLC-VWD                  Location : Vial 61
Injection Date  : 3/26/2014 9:56:13 AM               Inj :   1
                                               Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\JJJ\JJJ-2-85\JJJ-2-85-11 2014-03-26 09-55-30\VWD-AD-80-20-1ML-5UL-
                  205NM-35MIN.M
Last changed    : 3/26/2014 9:55:30 AM by SYSTEM
Analysis Method : E:\DATA\JJJ\JJJ-2-85\JJJ-2-85-11 2014-03-26 09-55-30\VWD-AD-80-20-1ML-5UL-
                  205NM-35MIN.M (Sequence Method)
Last changed    : 5/5/2014 11:31:04 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: VWD1 A, Wavelength=205 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  14.054 BB    0.3925 8170.12256  320.44247  99.9020
   2  22.264 MM T  0.2791    8.01460 4.78639e-1   0.0980

Totals :                  8178.13715  320.92111


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