Electronic Supplementary Material (ESI) for ChemComm.

Enantioselective synthesis of β-substituted Chiral Allylic Amines *via* Rh-Catalyzed Asymmetric Hydrogenation

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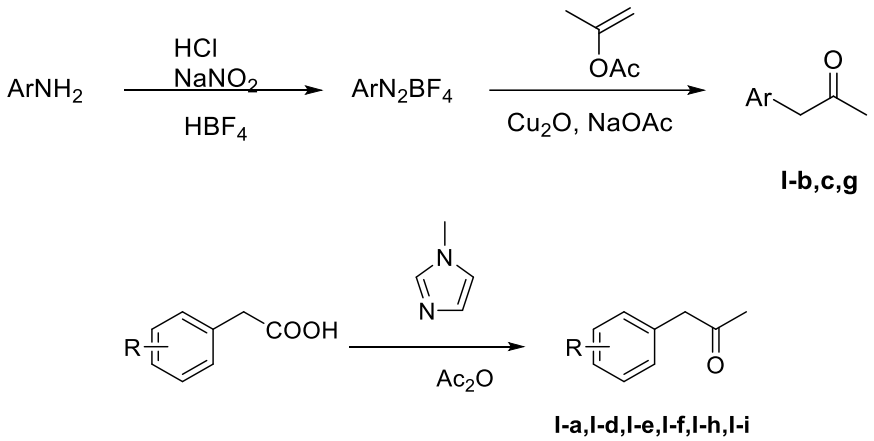
1. General Information

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 Ia-i [1, 2]



Preparation of I-b, c, g

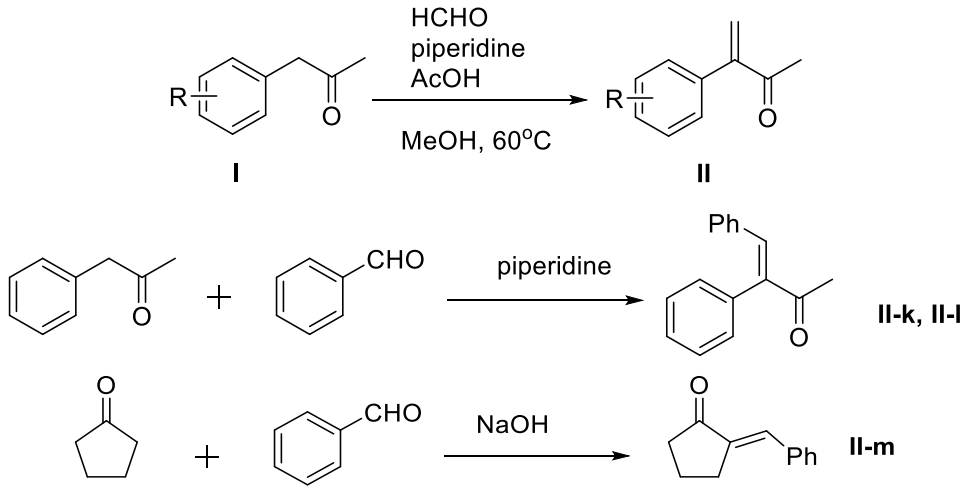
To a round bottom flask equipped with a magnetic stir bar was charged concentrated hydrochloric acid （38 mL）, H_2O (55 mL), and freshly distilled arylamine (150 mmol)was added dropwise. The reaction mixture was then cooled to 0°C and a solution of sodium nitrite (10.6 g in 7 mL H_2O) was added dropwise and keep the internal temperature did not exceed 5°C. After stirring at 0°C for 40 min, 17.04 gram fluoboric acid (40%) was added to the reaction mixture in 15 min. After stirring at 0°C for 30 min, the reaction mixture was quiescent at that temperature for 2 h. The reaction mixture was then filtered, the filter cake was washed with 30 mL of ethanol (95%) and anhydrous ether (30×2 mL). Then it was dried in vacuum oven to give aromaticdiazonium tetrafluoroborate without further purification.

To a round bottom flask equipped with a magnetic stir bar was charged anhydrous sodium acetate (12.4 g), cuprous oxide (0.8 g), isopropenyl acetate (40 mL), aromaticdiazonium tetrafluoroborate (54 mmol) was added slowly. The reaction mixture was then warmed at 40-65 °C for 5-8 h. The precipitation was separated through filter and the filter residue was washed with ethyl acetate thoroughly. Combined the filtrate and washed with water and saturated sodium bicarbonate solution. The organic layer was dried over $NaSO_4$, filtered. Removing the solvent by rotary evaporation, the crude material was purified by flash chromatography (*n*-hexane : ethyl acetate 50:1) to afford the target product.

Preparation of I-a, d, e, f, h, i

In a typical reaction, an aryl carboxylic acid (0.05 mol, 1 equiv.) was dissolved in Ac_2O (0.25 mol, 25mL, 5 equiv.) at room temperature, and the solution was stirred and purged with N_2 for several minutes. The reaction was initiated by the addition of 1-Methylimidazole (0.025 mol, 0.5 equiv.), and the reaction was continuously purged with a slow flow of N_2 at room temperature until the starting material was completely disappear (by TLC). After completion, water (10 mL) was added to the reaction flask to hydrolyze Ac_2O. The reaction mixture was extracted with ethyl acetate (3× 20mL) , and the extracts combined and washed with saturated Na_2HCO_3 followed by water , then dried over $MgSO_4$ and filtered . Removing the solvent by rotary evaporation gave the product mixture, then purified by flash chromatography (*n*-hexane /ethyl acetate: 20:1-10:1) to give the desired products.

3. General procedure for the synthesis of compound IIa-m [3, 4]



Preparation of IIa - j

A mixture composed of the 1-aryl-2-propanone (1. 0 equiv.), 37% formaldehyde solution (1.2 equiv.), piperidine (10 mol%), and AcOH (10 mol%) was heated in anhydrous MeOH (1.4 M) at 60°C for 10 h. Upon completion, the reaction mixture was cooled down to ambient temperature, and then diluted with water. The aqueous layer was extracted several times with diethyl ether. The combined organic layers were dried over anhydrous Na_2SO_4, filtered and concentrated under reduced pressure. The crude residue was purified by flash chromatography on silica gel, using an appropriate *n*-hexane/ ethyl acetate (20:1-10:1) mixture as eluent to give the pure products II.

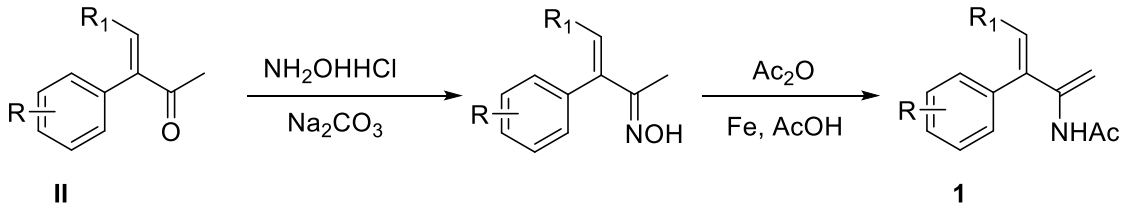
Preparation of II-k-l

A mixture of phenylacetone (30 mmol), benzaldehyde (30 mmol) and piperidine (0.8 mmol) in toluene (50 mL) was refluxed for 12 h using a Dean-Stark water separator. Then the reaction mixture was concentrated in vacuo and the residue was subjected to silica gel column chromatography (penatane /EtOAc, 20/1-10/1) to give the desired product.

Preparation of II-m

A mixture of aldehyde (30 mmol), cycloakanone (36 mmol), diethyl ether (30 mL), and 1N NaOH solution (30 mL) was stirred at room temperature for 72 h. After reaction, the mixture was diluted with 50 mL of diethyl ether and the aqueous layer was separated and extracted with ether (3×20 mL). The combined ether solution was washed to neutral with water and dried over Na_2SO_4. The solvent was evaporated and the product was purified by silica gel column chromatography with petroleum/ ethyl acetate to yield the desired product.

4. General procedure for the synthesis of compound 1[3,5]



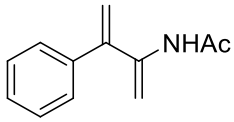
II 1

Preparation of 1

A methanol solution of the α,β-unsaturated ketone (1.0 equiv.), hydroxylamine hydrochloride (1.1 equiv.), and Na_2CO_3 (1.2 equiv.) was stirred at ambient temperature for 12 h. Then diluted with water. The resultant layer was extracted with

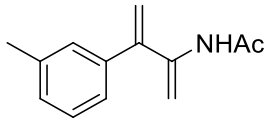
diethyl ether several times. The combined organic layer was dried over anhydrous Na_2SO_4 , filtered and concentrated under reduced pressure. The crude residue was purified by flash chromatography on silica gel (*n*-hexane/EtOAc = 5/1-2/1) to give oxime as a white solid.

Acetic anhydride (3.0 equiv.) was added, in portions, to a solution of oxime (1.0 equiv.) obtained from first step in toluene (1.3 M) under a nitrogen atmosphere. Acetic acid (3.0 equiv.) was then added, followed by Fe powder (2.0 equiv.). The mixture was then heated to 70 °C overnight. The reaction was then cooled to room temperature and filtered through celite to remove solid residues, which were then washed with ethyl acetate. The combined filtrate was washed with brine. The organic phase was separated, dried by Na_2SO_4 and evaporated. The crude material was purified by flash chromatography (*n*-hexane/ ethyl acetate 5:1-2:1) to afford the product **1** as white solid.



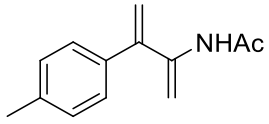
***N*-(3-phenylbuta-1,3-dien-2-yl)acetamide (1a)**

White solid, 0.56g, 60% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32-7.48 (m, 5H), 6.74 (brs, 1H), 5.93 (s, 1H), 5.44 (s, 1H), 5.34 (s, 1H) 4.96 (s, 1H), 2.02 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.9, 147.4, 140.3, 138.4, 128.4, 128.3, 128.0, 114.9, 105.3, 24.5; ESI-HRMS Calculated for $C_{12}H_{13}NNaO^+$ ($[M+Na]^+$): 210.0889, found: 210.08894.



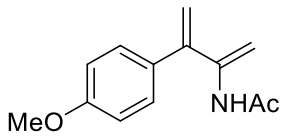
***N*-(3-m-tolylbuta-1,3-dien-2-yl)acetamide (1b)**

White solid, 0.52g, 51% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.14-7.30 (m, 5H), 6.67 (brs, 1H), 5.96 (s, 1H), 5.43 (s, 1H), 5.35 (s, 1H) 4.96 (s, 1H), 2.38 (s, 3H), 2.03 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.8, 147.5, 140.4, 138.2, 138.1, 129.1, 128.6, 128.3, 125.1, 114.8, 105.0, 24.6, 21.4; ESI-HRMS Calculated for $C_{13}H_{16}NO^+$ ($[M+H]^+$): 202.1226, found: 202.12240.



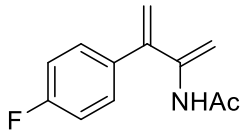
***N*-(3-p-tolylbuta-1,3-dien-2-yl)acetamide (1c)**

White solid, 0.72g, 71% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26-7.33 (m, 2H), 7.14-7.19 (m, 2H), 6.77 (brs, 1H), 5.93 (s, 1H), 5.40 (s, 1H), 5.34 (s, 1H) 4.96 (s, 1H), 2.38 (s, 3H), 2.02 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.9, 147.2, 140.5, 138.2, 135.4, 129.1, 127.8, 114.3, 104.9, 24.5, 21.2; ESI-HRMS Calculated for $C_{13}H_{15}NNaO^+$ ($[M+Na]^+$): 224.1046, found: 224.10461.



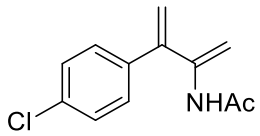
***N*-(3-(4-methoxyphenyl)buta-1,3-dien-2-yl)acetamide (1d)**

White solid , 0.49g, 45% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27-7.36 (m, 2H), 6.81-6.92 (m, 2H), 6.50 (brs, 1H), 5.94 (s, 1H), 5.34 (s, 1H), 5.28 (s, 1H), 4.95 (s, 1H), 3.82 (s, 3H), 2.01 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.76, 159.73, 146.84, 140.54, 130.59, 129.18, 113.81, 113.61, 104.58, 55.33, 24.64; ESI-HRMS Calculated for $C_{13}H_{16}NO_2^+$ ($[M+H]^+$): 218.1176, found: 218.1175.



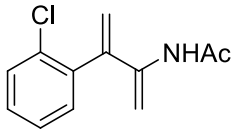
***N*-(3-(4-fluorophenyl)buta-1,3-dien-2-yl)acetamide (1e)**

White solid, 0.31g, 30% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27-7.40 (m, 2H), 6..96-7.11 (m, 2H), 6.55 (brs, 1H), 5.90 (s, 1H), 5.40 (s, 1H), 5.30 (s, 1H), 4.91 (s, 1H), 2.03 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.71, 163.99, 161.52, 146.39, 140.13, 134.42, 129.77, 129.69, 115.48, 115.27, 114.78, 105.52, 24.58; ESI-HRMS Calculated for $C_{12}H_{13F}NO^+$ ($[M+H]^+$): 206.0976, found: 206.0975.



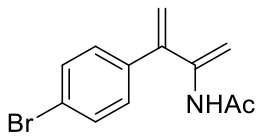
***N*-(3-(4-chlorophenyl)buta-1,3-dien-2-yl)acetamide (1f)**

White solid, 0.40g, 35% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28-7.35 (m, 5H), 6.55 (brs, 1H), 5.89 (s, 1H), 5.42 (s, 1H), 5.33 (s, 1H), 4.91 (s, 1H), 2.02 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.71, 146.33, 139.95, 136.87, 134.23, 129.32, 128.64, 115.27, 105.71, 24.57; ESI-HRMS Calculated for $C_{12}H_{13}ClNO^+$ ($[M+H]^+$): 222.0680, found: 222.0679.



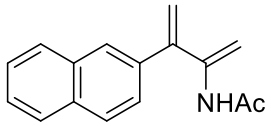
***N*-(3-(2-chlorophenyl)buta-1,3-dien-2-yl)acetamide (1g)**

White solid, 0.50g, 42% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36-7.46 (m, 1H), 7.25-7.32 (m, 3H), 6.87 (brs, 1H), 5.80 (s, 1H), 5.59 (s, 1H), 5.21 (s, 1H) 4.66 (s, 1H), 2.15 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.9, 145.2, 138.6, 138.55, 133.2, 131.4, 129.6, 129.3, 126.7, 115.4, 106.8, 24.5; ESI-HRMS Calculated for $C_{12}H_{12}ClNNaO^+$ ($[M+Na]^+$): 244.0500, found: 244.04996.



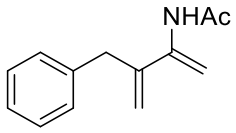
***N*-(3-(4-bromophenyl)buta-1,3-dien-2-yl)acetamide (1h)**

White solid, 0.40g, 30% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42-7.53 (m, 2H), 7.38 – 7.13 (m, 2H), 6.53 (brs, 1H), 5.90 (s, 1H), 5.44 (s, 1H), 5.35 (s, 1H), 4.92 (s, 1H), 2.03 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.69, 146.39, 139.86, 137.34, 131.61, 129.64, 122.45, 115.32, 105.75, 24.58; ESI-HRMS Calculated for $C_{12}H_{13}BrNO^+$ ($[M+H]^+$): 266.0175, found: 266.0175, 268.0152.



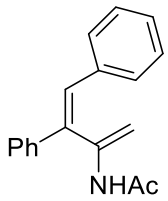
***N*-(3-(naphthalen-2-yl)buta-1,3-dien-2-yl)acetamide (1i)**

White solid, 0.45g, 38% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.73-8.04 (m, 4H), 7.41-7.57 (m, 3H), 6.57 (brs, 1H), 6.02 (s, 1H), 5.53 (s, 1H), 5.49 (s, 1H), 5.01 (s, 1H), 2.00 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.81, 147.42, 140.35, 135.61, 133.24, 133.16, 128.24, 128.10, 127.65, 127.19, 126.44, 125.76, 115.56, 105.08, 24.67; ESI-HRMS Calculated for $C_{16}H_{16}NO^+$ ($[M+H]^+$): 238.1226, found: 238.1225.



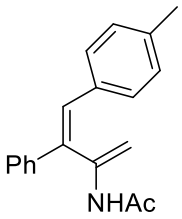
***N*-(3-benzylbuta-1,3-dien-2-yl)acetamide (1j)**

White solid, 0.48g, 48% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27-7.36 (m, 2H), 7.16-7.25 (m, 3H), 6.52 (brs, 1H), 5.66 (s, 1H), 5.30 (s, 1H), 5.10 (s, 1H), 4.97 (s, 1H), 3.60 (s, 2H), 2.04 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.68, 144.29, 139.29, 138.95, 128.78, 128.53, 126.45, 114.20, 104.96, 40.13, 24.49; ESI-HRMS Calculated for $C_{13}H_{16}NO^+$ ($[M+H]^+$): 202.1226, found : 202.1225.



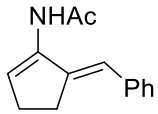
N-(3,4-diphenylbuta-1,3-dien-2-yl)acetamide (1k)

White solid, 0.39g, 30% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27-7.44 (m, 3H), 7.16-7.24 (m, 2H), 7.12 – 7.05 (m, 3H), .686-6.99 (m, 2H), 6.83 (s, 1H), 6.64 (brs, 1H), 5.83 (s, 1H), 4.92 (s, 1H), 2.04 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.75, 142.12, 139.75, 137.32, 136.26, 130.16, 129.66, 128.80, 128.04, 127.85, 127.28, 106.99, 24.51; ESI-HRMS Calculated for $C_{18}H_{18}NO^+$ ($[M+H]^+$): 264.1383, found : 264.1381.



N-(3-phenyl-4-(p-tolyl)buta-1,3-dien-2-yl)acetamide (1l)

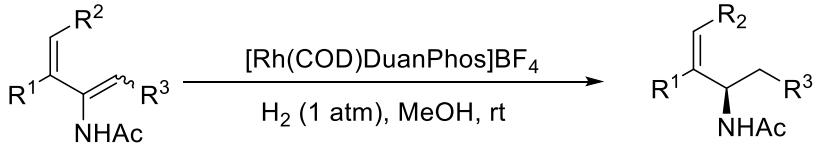
White solid, 0.40g, 26% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27-7.41 (m, 3H), 7.15-7.24 (m, 2H), 6.88 –6.96 (m, 2H), 6.71-6.87 (m, 3H), 6.54 (brs, 1H), 5.82 (s, 1H), 4.90 (s, 1H), 2.25 (s, 3H), 2.05 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.78, 142.14, 138.82, 137.54, 137.24, 133.32, 130.15, 129.58, 128.81, 127.93, 127.75, 106.75, 24.54, 21.20; ESI-HRMS Calculated for $C_{19}H_{20}NO^+$ ($[M+H]^+$): 278.1539, found : 278.1538.



(*E*)-N-(5-benzylidenecyclopent-1-en-1-yl)acetamide (1m)

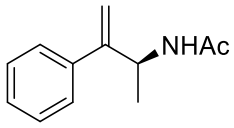
Dark red solid, 0.38g, 36% yield; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39-7.44 (m, 4H), 7.15-7.24 (m, 1H), 6.96 (brs, 1H), 6.73 (t, *J* = 2.7 Hz, 1H), 6.11 (s, 1H), 2.93 – 2.79 (m, 2H), 2.58-2.66 (m, 2H), 2.21 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.40, 143.42, 137.51, 136.33, 128.49, 128.19, 126.32, 123.56, 114.30, 29.64, 28.60, 24.41; ESI-HRMS Calculated for $C_{14}H_{16}NO^+$ ($[M+H]^+$): 214.1226, found : 214.1226.

5. General Procedure for Asymmetric Hydrogenation of compound 1



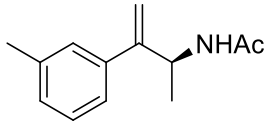
1 **2**

A stock solution was made by mixing $[Rh(cod)_2]BF_4$ with (*S*c,*R*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 (0.5mL, 0.0005 mmol) was transferred by syringe into the vials charged with different substrates (0.05 mmol for each) in anhydrous MeOH (0.5 mL). The vials were subsequently transferred into an autoclave into which hydrogen gas was charged. The reaction was then stirred under H_2 (1 atm) at room temperature for 6 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 yield the desired products. The ee values of compounds **2** were determined by HPLC analysis on a chiral stationary phase.



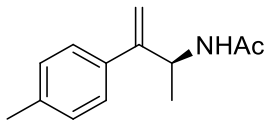
(*S*)-*N*-(3-phenylbut-3-en-2-yl)acetamide (2a)

White solid, 9.3 mg, 97% yield, 95% ee; $[\alpha]_D^{20}$ = -26.0 (c = 0.5, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralpak OD-H column, hexane: isopropanol = 97: 3; flow rate = 1.0 mL/min; UV detection at 220 nm; t_R = 16.8 min (major), 21.6 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.38-7.46 (m, 2H), 7.29-7.38 (m, 3H), 5.67 (br, 1H), 5.35 (s, 1H), 5.24 (s, 1H), 5.03-5.19 (m, 1H), 1.96 (s, 3H), 1.33 (d, *J* = 6.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 169.2, 150.5, 139.9, 128.4, 127.8, 126.6, 112.2, 47.5, 23.4, 20.3. ESI-HRMS Calculated for $C_{12}H_{15}NNaO^+$ ($[M+Na]^+$): 212.1046, found: 212.10463.



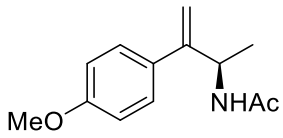
(*S*)-*N*-(3-m-tolylbut-3-en-2-yl)acetamide (2b)

Yellow oil, 9.6 mg, 94% yield, 95% ee; $[\alpha]_D^{20}$ = -21.0 (c = 0.4, CH_2Cl_2); 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 254 nm; t_R = 11.7 min (minor), 13.7 min (major). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.18-7.27 (m, 3H), 7.09-7.16 (m, 1H), 5.60 (br, 1H), 5.32 (s, 1H), 5.21 (s, 1H), 5.02-5.16 (m, 1H), 2.37 (s, 3H), 1.97 (s, 3H), 1.33 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 169.2, 150.7, 139.9, 137.9, 128.5, 128.3, 127.4, 123.8, 111.9, 47.6, 23.4, 21.5, 20.4. ESI-HRMS Calculated for $C_{13}H_{17}NNaO^+$ ($[M+Na]^+$): 226.1202, found: 226.12024.



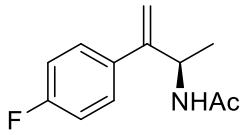
(*S*)-*N*-(3-p-tolylbut-3-en-2-yl)acetamide (2c)

White solid, 9.5 mg, 93% yield, 94% ee; $[\alpha]_D^{20}$ = -22.7 (c = 0.4, CH_2Cl_2); 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 254 nm; t_R = 13.5 min (minor), 15.7 min (major). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.29-7.36 (m, 2H), 7.13-7.18 (m, 2H), 5.61 (br, 1H), 5.33 (s, 1H), 5.20 (s, 1H), 5.04-5.17 (m, 1H), 2.36 (s, 3H), 1.96 (s, 3H), 1.33 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 169.2, 150.5, 137.6, 136.9, 129.1, 126.5, 111.5, 47.4, 23.4, 21.1, 20.3. ESI-HRMS Calculated for $C_{13}H_{17}NNaO^+$ ($[M+Na]^+$): 226.1202, found: 226.12044.



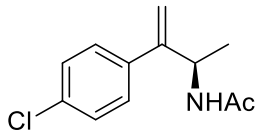
(*R*)-N-(3-(4-methoxyphenyl)but-3-en-2-yl)acetamide (2d)

White solid, 10.5 mg, 95% yield, 91% ee; $[\alpha]_D^{20}$= + 25.3 (c = 1.0, $CHCl_3$); 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 254 nm; t_R = 19.9 min (major), 29.7 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 – 7.30 (m, 2H), 6.99 – 6.72 (m, 2H), 5.56 (br s, 1H), 5.27 (s, 1H), 5.14 (s, 1H), 5.01-5.11 (m, 1H), 3.80 (s, 3H), 1.93 (s, 3H), 1.31 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.17, 159.29, 149.66, 132.18, 127.70, 113.72, 110.83, 55.28, 47.27, 23.45, 20.28; ESI-HRMS Calculated for $C_{13}H_{18}NO_2^+$ ($[M+H]^+$): 220.1332, found : 220.1330.



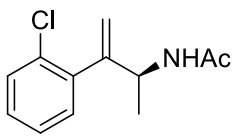
(*R*)-N-(3-(4-fluorophenyl)but-3-en-2-yl)acetamide (2e)

White solid, 10.0 mg, 96% yield, 86% ee; $[\alpha]_D^{20}$ = +21.9 (c = 1.0, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralpak OD-H column, hexane: isopropanol = 95: 5; flow rate = 0.5 mL/min; UV detection at 220 nm; t_R = 22.4 min (major), 26.4 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.45 – 7.30 (m, 2H), 7.07 – 6.94 (m, 2H), 5.56 (brs, 1H), 5.28 (s, 1H), 5.20 (s, 1H), 5.00-5.11 (m, 1H), 1.94 (s, 3H), 1.30 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.18, 163.69, 161.24, 149.55, 135.96, 135.92, 128.33, 128.25, 115.35, 115.14, 112.20, 47.42, 23.40, 20.24; ESI-HRMS Calculated for $C_{12}H_{15}FNO^+$ ($[M+H]^+$): 208.1132, found : 208.1131.



(*R*)-N-(3-(4-chlorophenyl)but-3-en-2-yl)acetamide (2f)

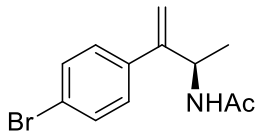
White solid, 10.9 mg, 97% yield, 97% ee; $[\alpha]_D^{20}$= +18.4 (c = 1.0, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralpak OD-H column, hexane: isopropanol = 95: 5; flow rate = 1.0 mL/min; UV detection at 220 nm; t_R = 23.9 min (major), 28.2 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 – 7.31 (m, 2H), 7.31 – 7.27 (m, 2H), 5.52 (brs, 1H), 5.31 (s, 1H), 5.23 (s, 1H), 5.00-5.10 (m, 1H), 1.94 (s, 3H), 1.30 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.18, 149.46, 138.34, 133.64, 128.55, 127.97, 112.68, 47.25, 23.40, 20.22; ESI-HRMS Calculated for $C_{12}H_{15}ClNO^+$ ($[M+H]^+$): 224.0837, found : 224.0836.



(*S*)-*N*-(3-(2-chlorophenyl)but-3-en-2-yl)acetamide (2g)

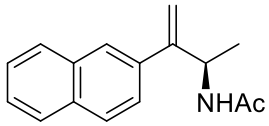
Yellow oil, 11.0 mg, 98% ee; $[\alpha]_D^{20}$ = -20.5 (c = 0.4, CH_2Cl_2); The enantiomeric

excess was determined by HPLC on Chiralpak OJ-H column, hexane: isopropanol = 99: 1; flow rate = 1.0 mL/min; UV detection at 210 nm; t_R = 42.7 min (major), 48.4 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.38-7.42 (m, 1H), 7.21-7.27 (m, 3H), 5.60 (br, 1H), 5.45 (s, 1H), 5.12 (s, 1H), 4.87-5.02 (m, 1H), 1.96 (s, 3H), 1.29 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ = 159.1, 148.3, 139.7, 132.6, 130.5, 129.6, 128.7, 126.5, 115.5, 49.3, 23.4, 19.8. ESI-HRMS Calculated for $C_{12}H_{14}ClNNaO^+$ ($[M+Na]^+$): 246.0656, found 246.06561.



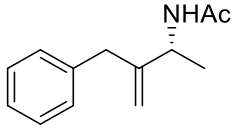
(*R*)-N-(3-(4-bromophenyl)but-3-en-2-yl)acetamide (2h)

White solid, 13.4 mg, 99% yield, 90% ee; $[\alpha]_D^{20}$ = +11.3 (c=1.0, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralpak AD-H column, hexane: isopropanol = 95: 5; flow rate = 0.5 mL/min; UV detection at 220 nm; t_R = 30.5 min (major), 35.9 min (minor); H NMR (400 MHz, $CDCl_3$) δ 7.59 – 7.38 (m, 2H), 7.33 – 7.17 (m, 2H), 5.48 (brs, 1H), 5.33 (s, 1H), 5.24 (s, 1H), 5.01-5.11 (m, 1H), 1.95 (s, 3H), 1.31 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.15, 149.51, 138.80, 131.51, 128.32, 121.84, 112.75, 76.74, 47.20, 23.42, 20.22; ESI-HRMS Calculated for $C_{12}H_{15}BrNO^+$ ($[M+H]^+$): 268.0332, found : 268.0332, 270.0332.



(*R*)-N-(3-(naphthalen-2-yl)but-3-en-2-yl)acetamide (2i)

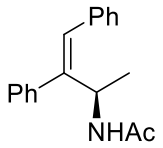
White solid, 10.4 mg, 86% yield, 84% ee; $[\alpha]_D^{20}$ = -0.9 (c = 1.0, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralpak AD-H column, hexane: isopropanol = 95: 5; flow rate = 0.5 mL/min; UV detection at 220 nm; t_R = 32.1 min (major), 35.8 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 – 7.72 (m, 4H), 7.52-7.58 (m, 1H), 7.43-7.50 (m, 2H), 5.54 (br s, 1H), 5.47 (s, 1H), 5.32 (s, 1H), 5.20-5.29 (m, 1H), 1.95 (s, 3H), 1.37 (d, *J* = 6.7 Hz, 3H); ^{3}C NMR (101 MHz, $CDCl_3$) δ 169.18, 150.42, 137.20, 133.28, 132.93, 128.26, 127.98, 127.55, 126.25, 126.07, 125.45, 124.98, 112.66, 47.48, 23.48, 20.43; ESI-HRMS Calculated for $C_{16}H_{18}NO^+$ ($[M+H]^+$): 240.1383, found : 240.1381.



(*R*)-N-(3-benzylbut-3-en-2-yl)acetamide (2j)

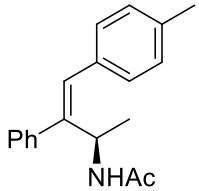
White solid, 10.0 mg, 98%yield, 96% ee; $[\alpha]_D^{20}$ = +53.6 (c = 0.5, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralpak OJ-H column, hexane: isopropanol = 95: 5; flow rate = 0.5 mL/min; UV detection at 220 nm; t_R = 33.8 min (major), 41.0 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.24 (m, 2H), 7.24 –

7.14 (m, 3H), 5.25 (brs, 1H), 5.07 (s, 1H), 4.84 (s, 1H), 4.66 – 4.45 (m, 1H), 3.39 (s, 2H), 1.85 (s, 3H), 1.24 (d, *J* = 6.9 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.09, 149.67, 139.21, 129.00, 128.47, 126.33, 112.30, 48.53, 40.76, 23.41, 19.88; ESI-HRMS Calculated for $C_{13}H_{18}NO^+$ ($[M+H]^+$): 204.1383, found : 204.1382.



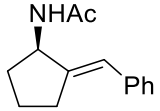
(*R*)-N-(3,4-diphenylbut-3-en-2-yl)acetamide (2k)

White solid, 13.0 mg, 98% yield, 97% ee; $[\alpha]_D^{20}$ = +17.3 (c=1.0, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralpak OD-H column, hexane: isopropanol = 90: 10; flow rate = 1.0 mL/min; UV detection at 254 nm; t_R = 19.1 min (major), 7.8 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27 – 7.44 (m, 3H), 7.14-7.20 (m, 2H), 6.95 –7.12 (m, 3H), 6.93 – 6.75 (m, 2H), 6.57 (s, 1H), 5.47 (d, *J* = 8.2 Hz, 1H), 5.03 – 4.73 (m, 1H), 1.96 (s, 3H), 1.31 (d, *J* = 6.9 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.00, 143.24, 138.54, 136.40, 129.25, 129.17, 128.79, 127.88, 127.48, 126.77, 126.74, 51.89, 23.53, 20.60 ESI-HRMS Calculated for $C_{18}H_{20}NO^+$ ($[M+H]^+$): 266.1539, found : 266.1537.



(*R*)-N-(3-phenyl-4-(p-tolyl)but-3-en-2-yl)acetamide (2l)

White solid, 13.8 mg, 99% yield, 97% ee; $[\alpha]_D^{20}$ = +92.6 (c = 1.0, $CHCl_3$); 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 254 nm; t_R = 13.7 min (major), 19.9 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29-7.41 (m, 3H), 7.23 – 7.06 (m, 2H), 6.83-6.90 (m, 2H), 6.71-6.81 (m, 2H), 6.54 (s, 1H), 5.48 (d, *J* = 8.2 Hz, 1H), 4.86-5.00 (m, 1H), 2.22 (s, 3H), 1.95 (s, 3H), 1.30 (d, *J* = 6.9 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.98, 142.24, 138.75, 136.52, 133.49, 129.29, 129.06, 128.79, 128.63, 127.39, 126.72, 51.89, 23.54, 21.12, 20.63; ESI-HRMS Calculated for $C_{19}H_{22}NO^+$ ($[M+H]^+$): 280.1696, found : 280.1696.

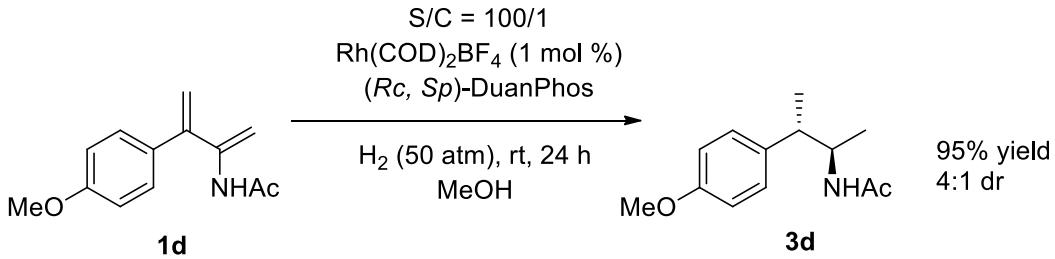


(*R*)-N-(2-benzylidenecyclopentyl)acetamide (2m)

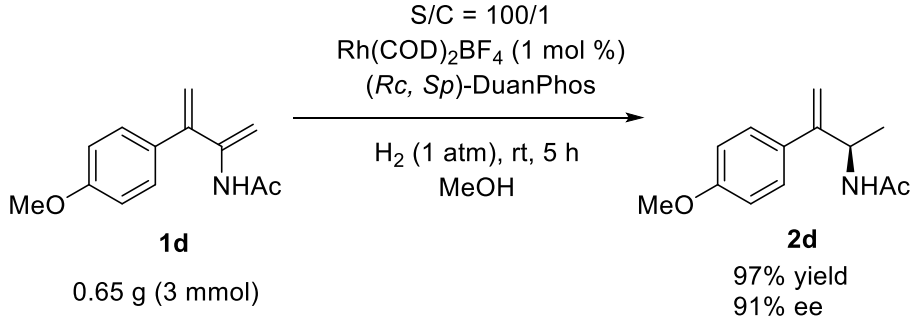
White solid, 10.1 mg, 94% yield, 84% ee; $[\alpha]_D^{20}$ = +53.6 (c=0.5, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralpak AD-H column, hexane: isopropanol = 95: 5; flow rate = 1.0 mL/min; UV detection at 220 nm; t_R = 18.4 min (major), 22.0 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 – 7.16 (m, 5H), 6.37 (s,

1H), 5.54 (brs, 1H), 4.83-4.93 (m, 1H), 2.78 – 2.56 (m, 2H), 2.25 – 2.14 (m, 1H), 2.07 (s, 3H), 1.93 – 1.81 (m, 1H), 1.76 – 1.61 (m, 1H), 1.48 – 1.36 (m, 1H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.87, 145.21, 137.63, 128.36, 128.26, 126.56, 122.71, 55.40, 33.62, 30.41, 23.64, 23.25; ESI-HRMS Calculated for $C_{14}H_{18}NO^+$ ($[M+H]^+$): 216.1383, found : 216.1382.

6. Procedure for the synthesis of compound 3d and hydrogenation of 1d and 1l with low catalyst loading

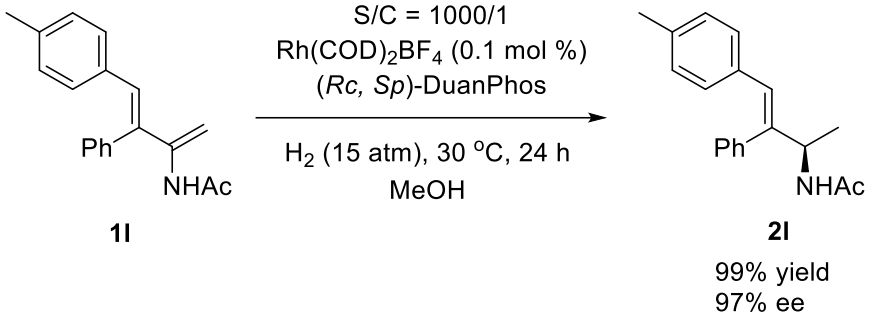


A stock solution was made by mixing $[Rh(cod)_2]BF_4$ with (*R*c,*S*p)-Duanphos in a 1:1.1 molar ratio in CH_2Cl_2 at room temperature for 30 min in a nitrogen-filled glovebox. An aliquot of the catalyst solution (0.5mL, 0.0005 mmol) was transferred by syringe into the vials charged with different substrates (0.05 mmol for each) in anhydrous MeOH (0.5 mL). The vials were subsequently transferred into an autoclave into which hydrogen gas was charged. The reaction was then stirred under H_2 (50 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 yield the desired products. The dr value of the resulting product was determined by ^{1}H NMR. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20 – 7.04 (m, 2H), 6.94 – 6.76 (m,2H), 5.27 – 4.97 (m, 1H), 4.42 – 4.06 (m, 1H), 3.83 (s, 3H), 3.00 – 2.51 (m, 1H), 1.91 (s, 3H), 1.29 (d, *J* = 7.0 Hz, 3H), 1.10 (d, *J* = 6.7 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.34, 158.24, 134.79, 129.05, 128.81, 113.75, 55.26, 49.60, 43.67, 23.56, 18.88, 17.66.



A stock solution was made by mixing $[Rh(cod)_2]BF_4$ with (Rc,Sp)-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 (0.03 mmol, 1 mol %) was transferred by syringe into the vials charged with **1d** (0.65g, 3 mmol) in anhydrous MeOH (3 mL). The vials were subsequently transferred into an autoclave into which hydrogen gas was charged. The reaction was then stirred under H_2 (1 atm) at room temperature for 5 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 yield the desired products **2d** as a white solid.



1l **2l**

99% yield
97% ee

A stock solution was made by mixing $[Rh(cod)_2]BF_4$ with (Rc,Sp)-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 (0.0001 mmol, 0.1 mol %) was transferred by syringe into the vials charged with **1l** (0.1 mmol) in anhydrous MeOH (1 mL). The vials were subsequently transferred into an autoclave into which hydrogen gas was charged. The reaction was then stirred under H_2 (15 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 yield the desired products **2l** as a white solid without no loss of yield and ee.

7. References

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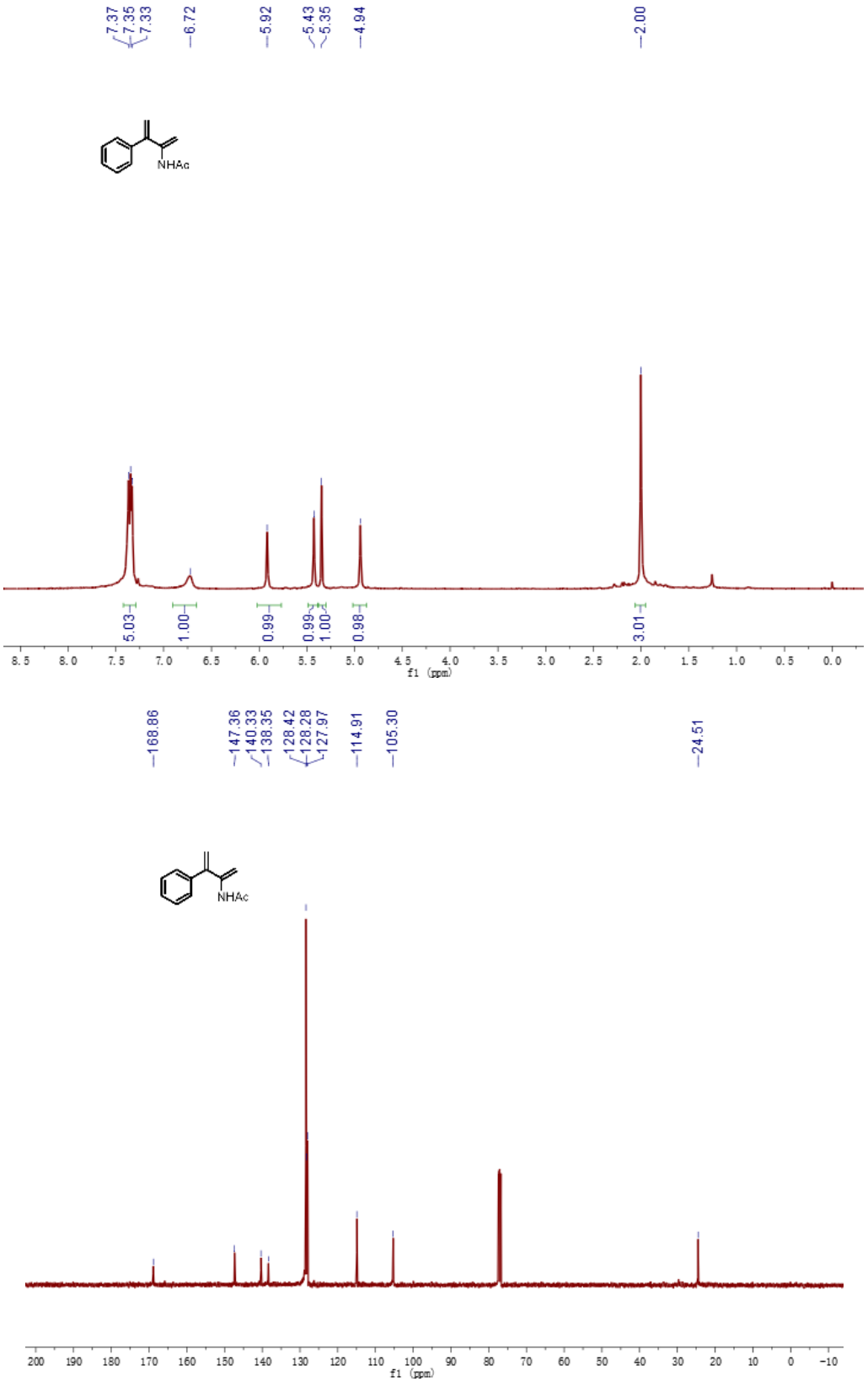
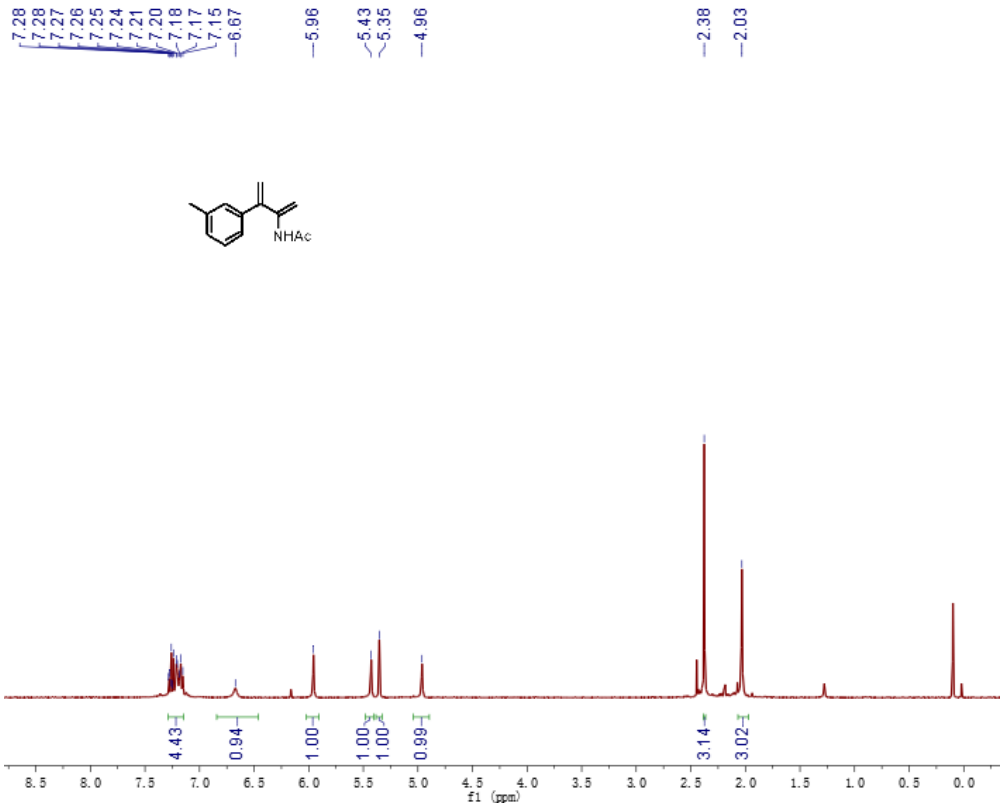
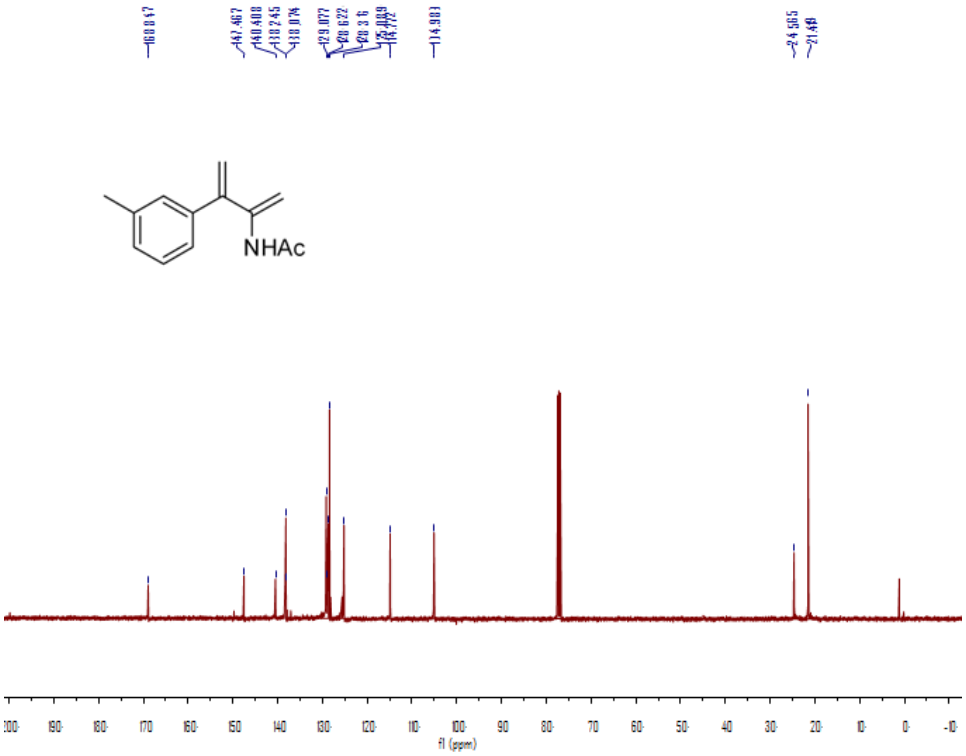
8. NMR spectra of 1 and 2, HPLC spectra of 2

1a-^{1}H NMR and ^{13}C NMR

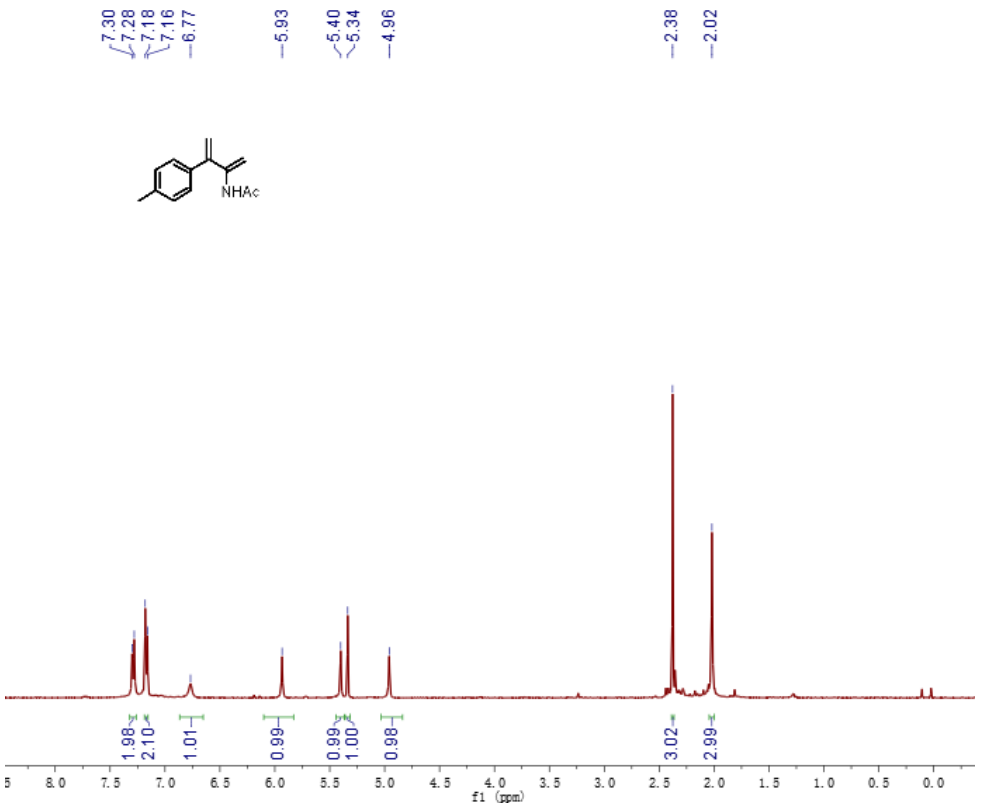


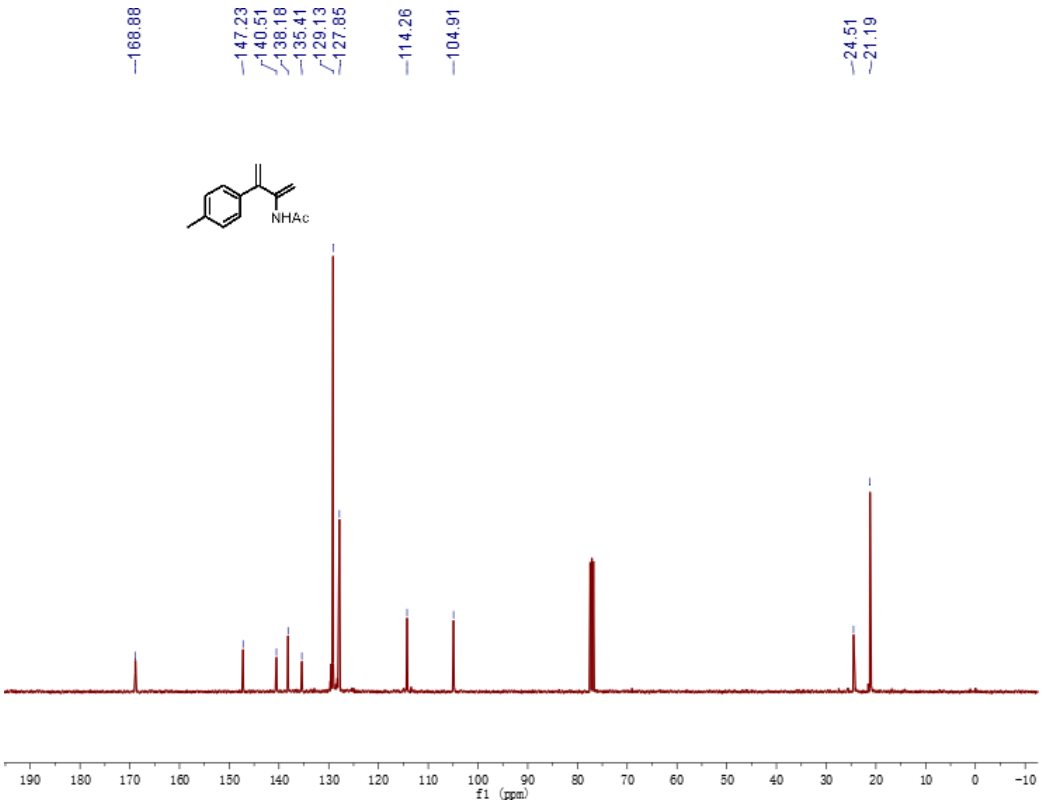
1b-^{1}H NMR and ^{13}C NMR



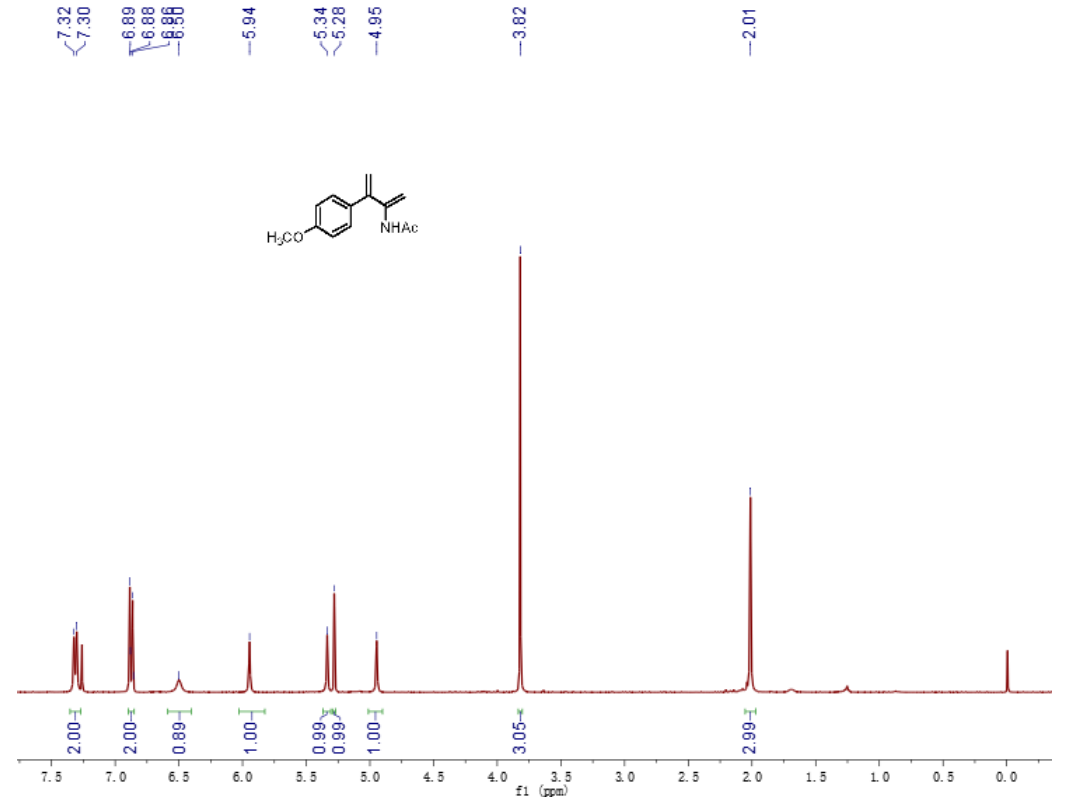


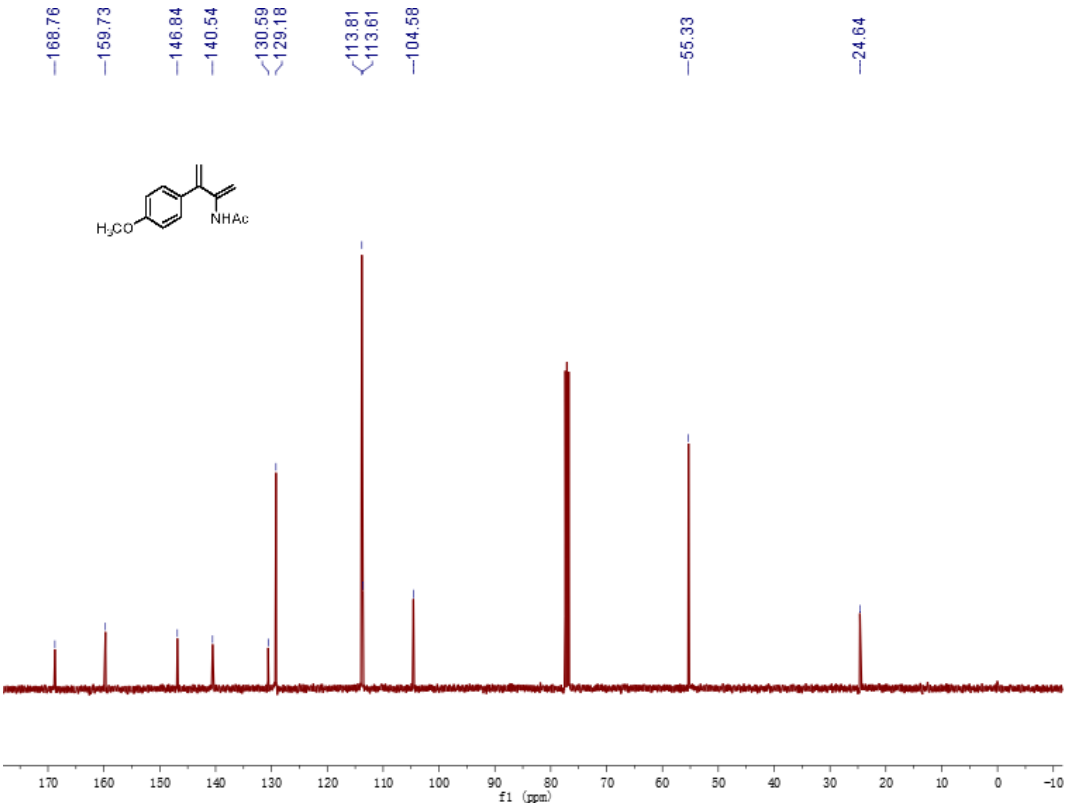
1c-^{1}HNMR and ^{13}C NMR



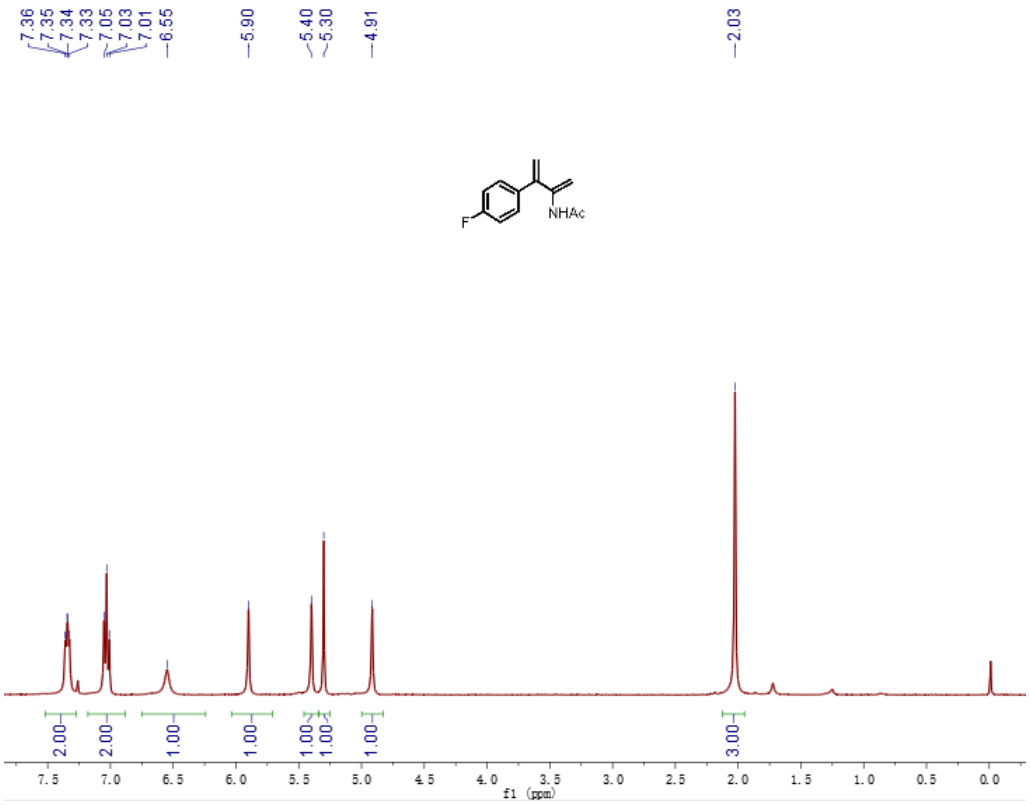


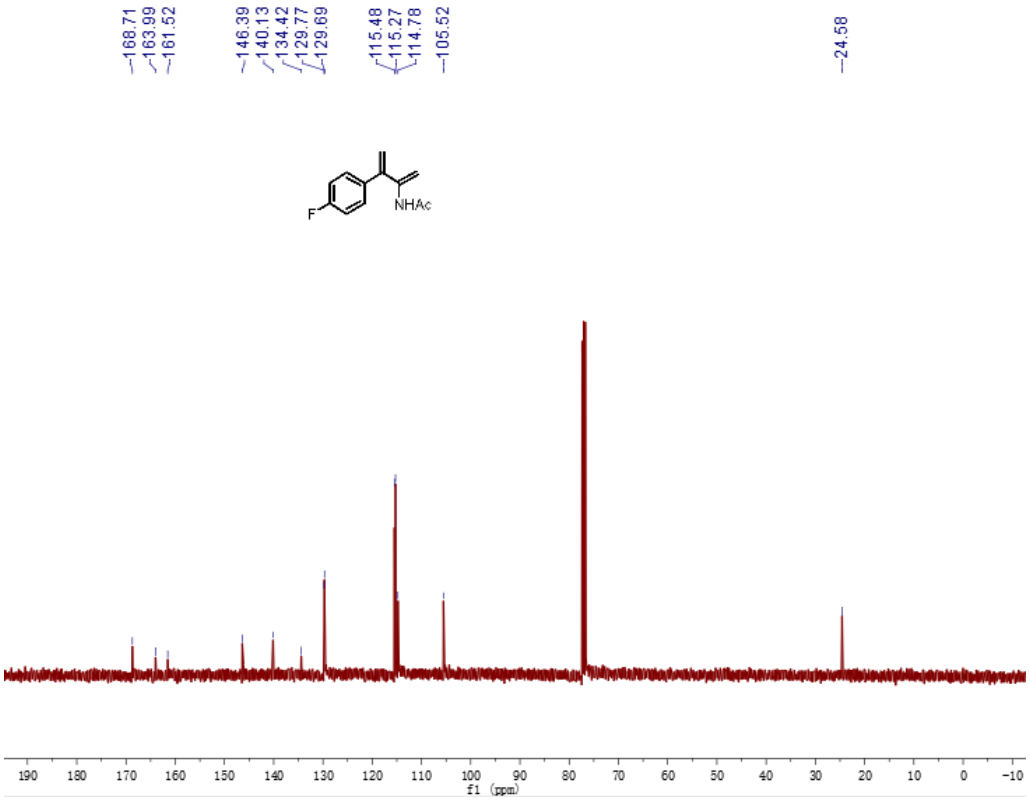
1d-^{1}H NMR and ^{13}C NMR



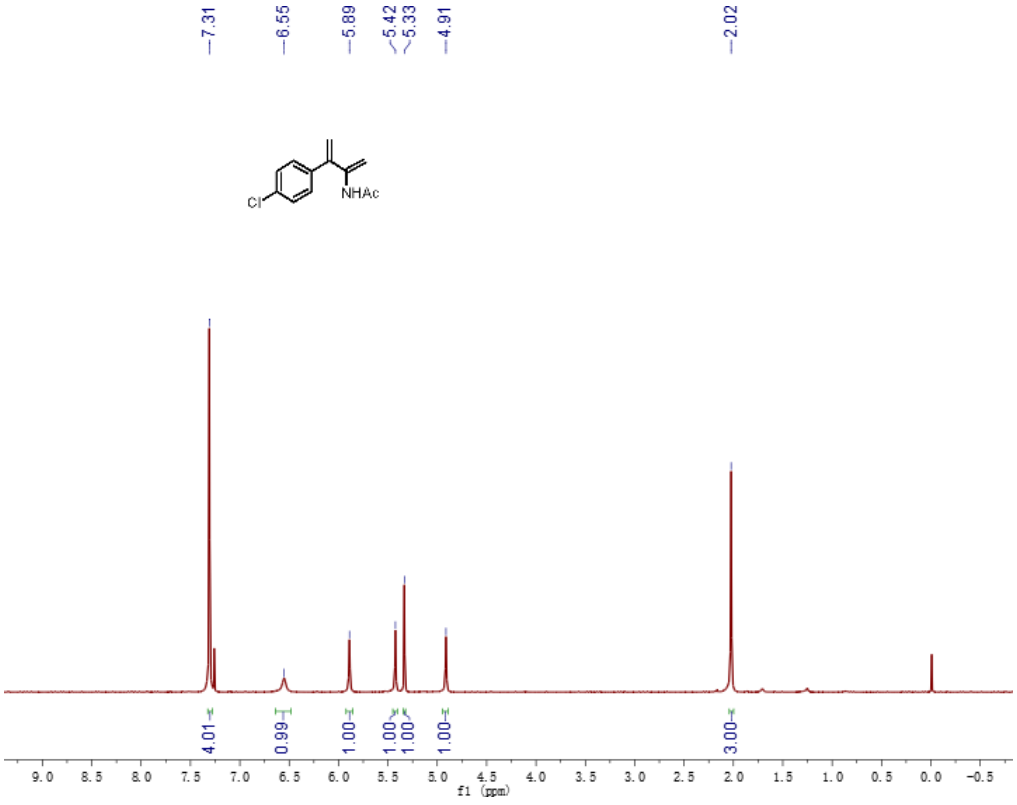


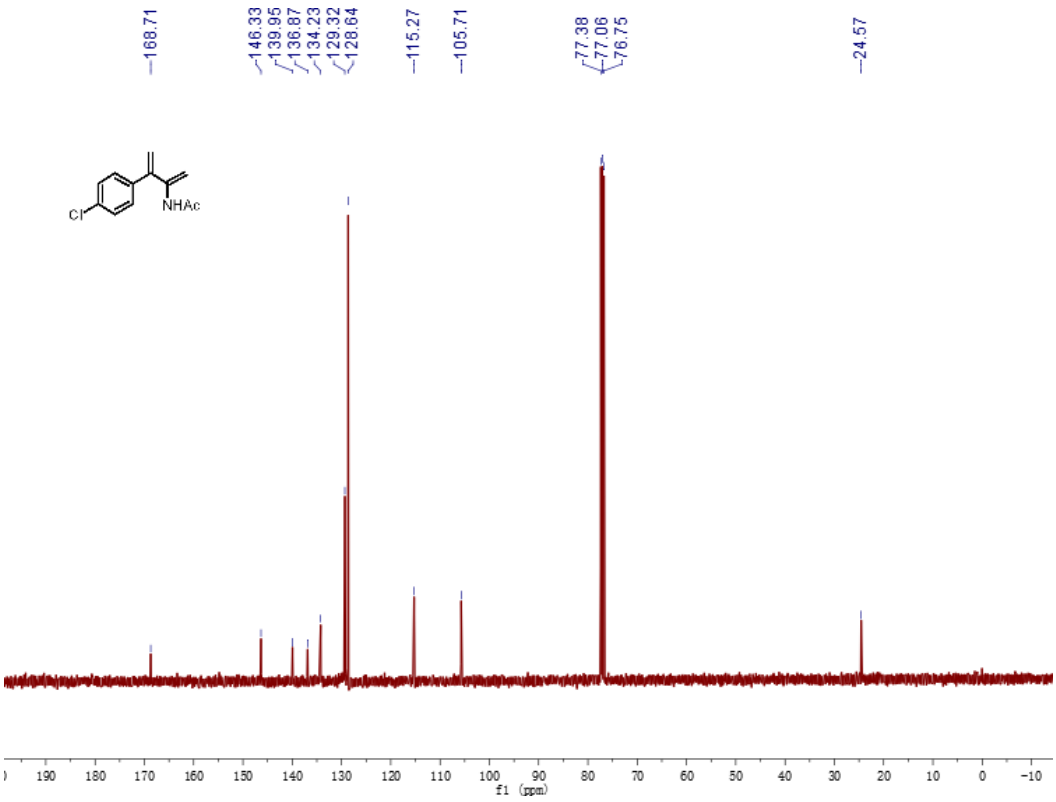
1e-^{1}H NMR and ^{13}C NMR



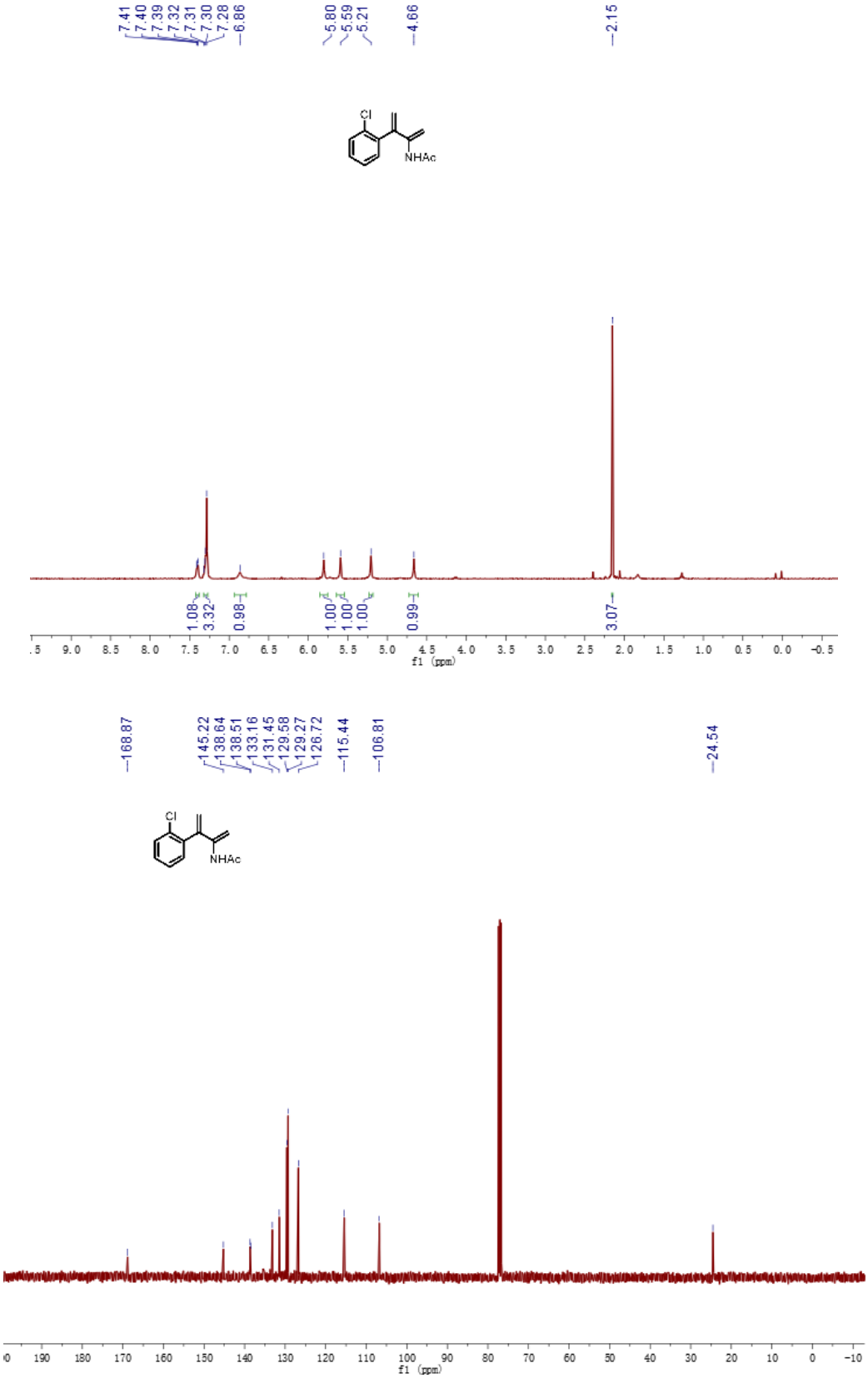


1f-^{1}H NMR and ^{13}C NMR

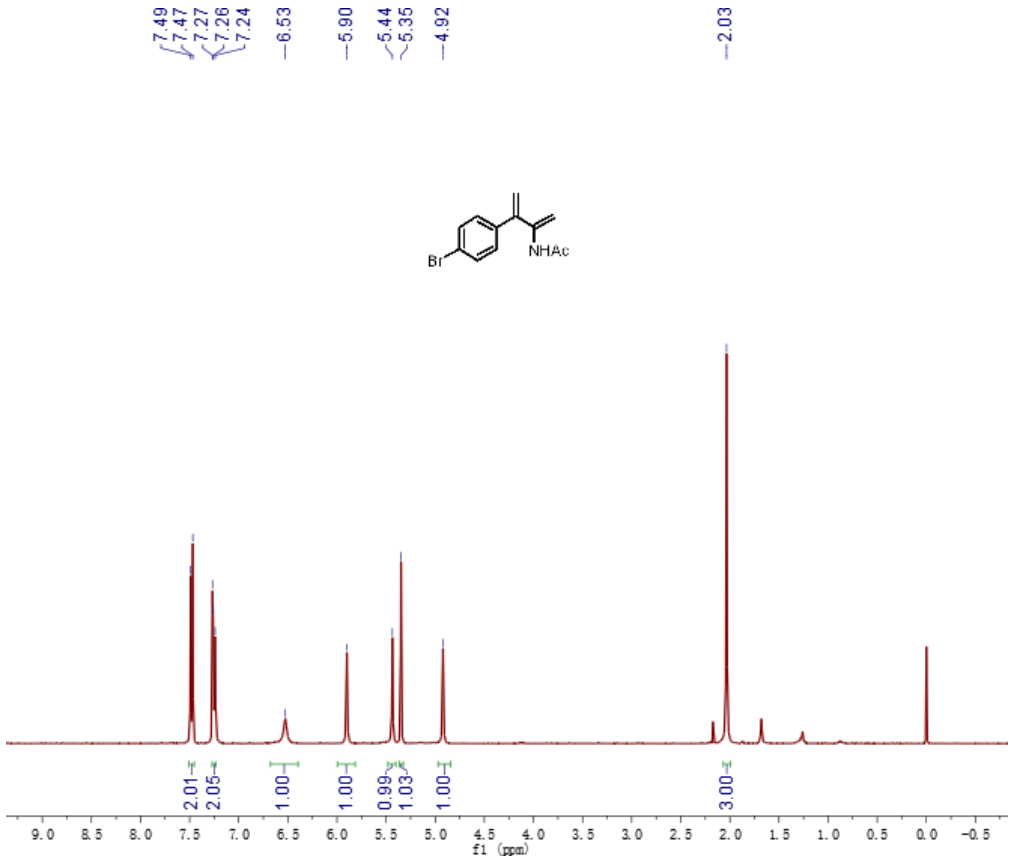


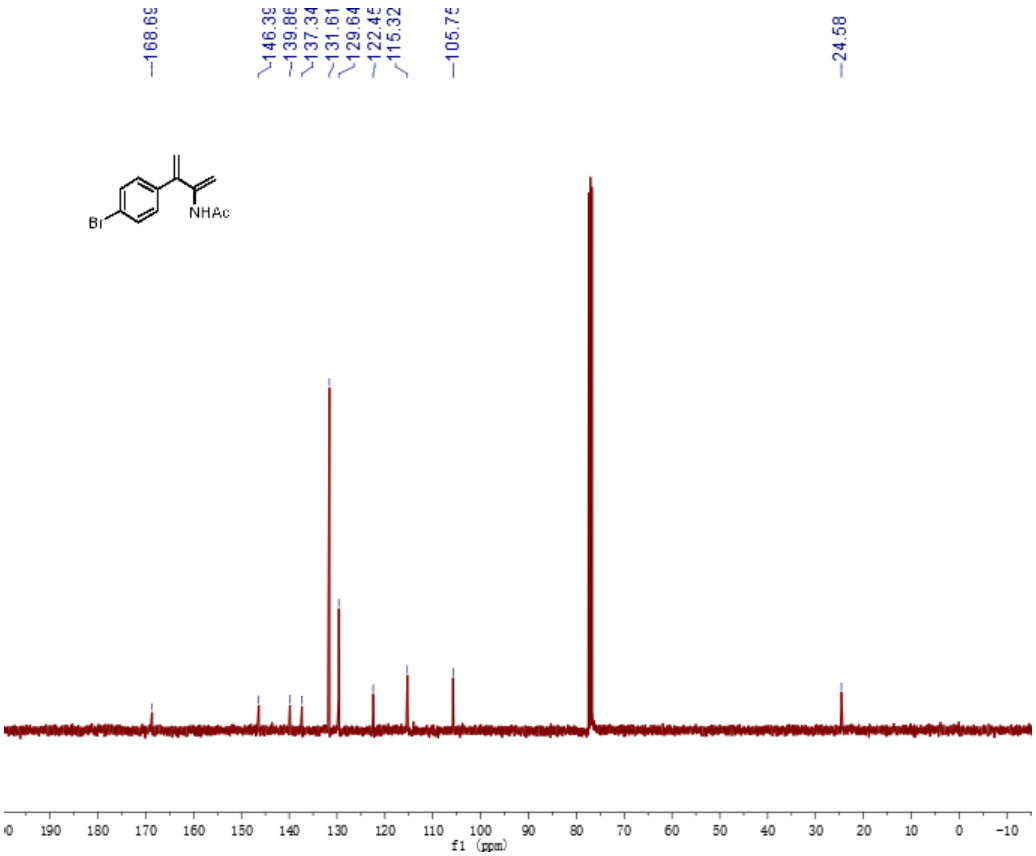


1g-^{1}H NMR and ^{13}C NMR

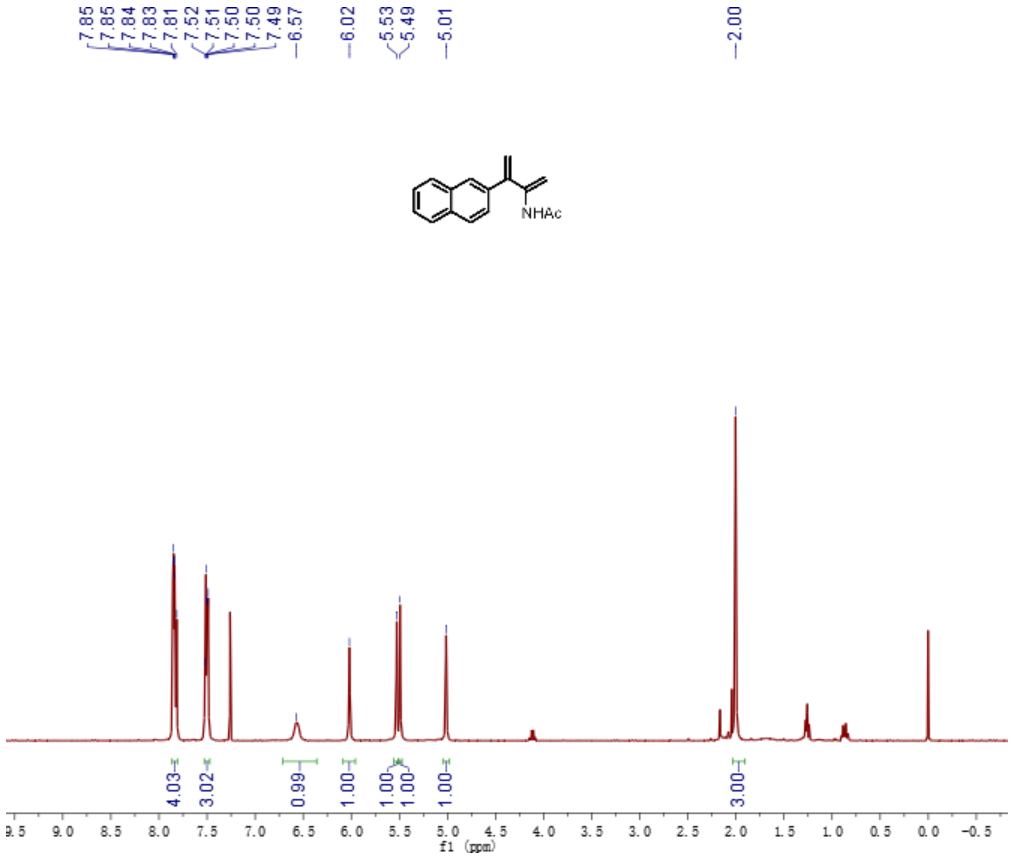


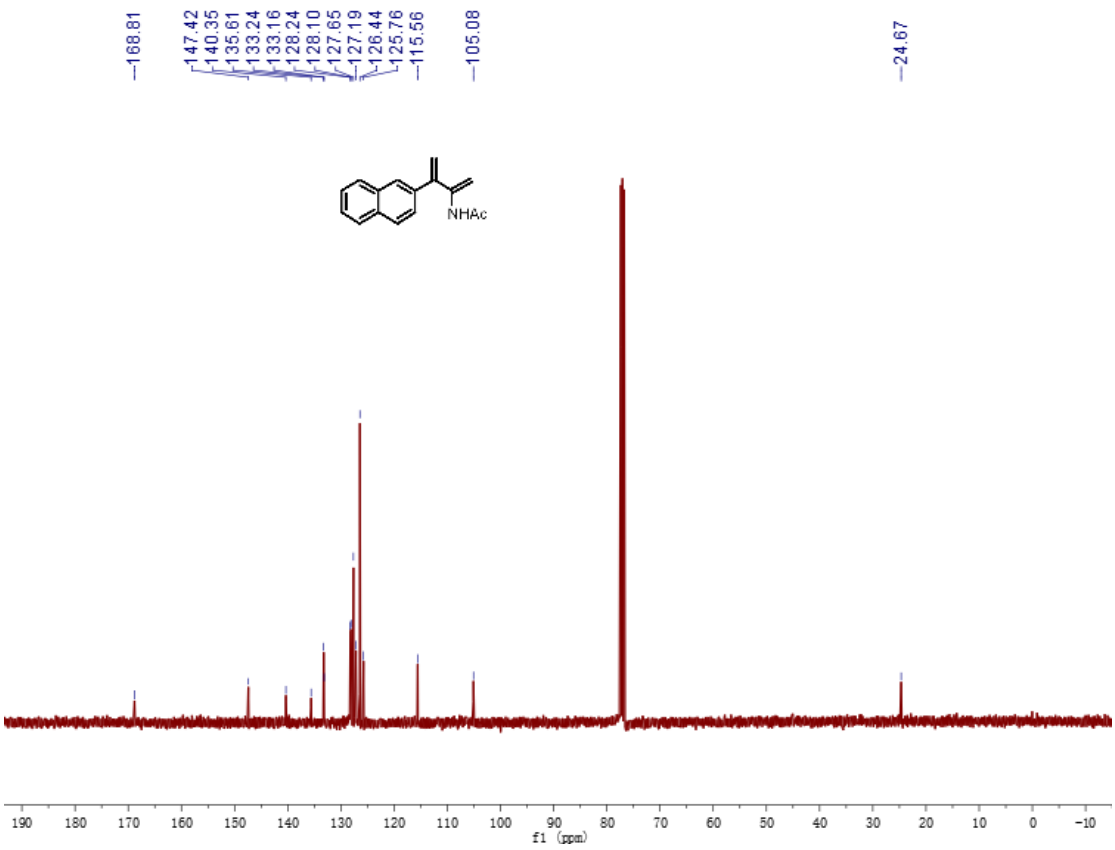
1h-^{1}H NMR and ^{13}C NMR



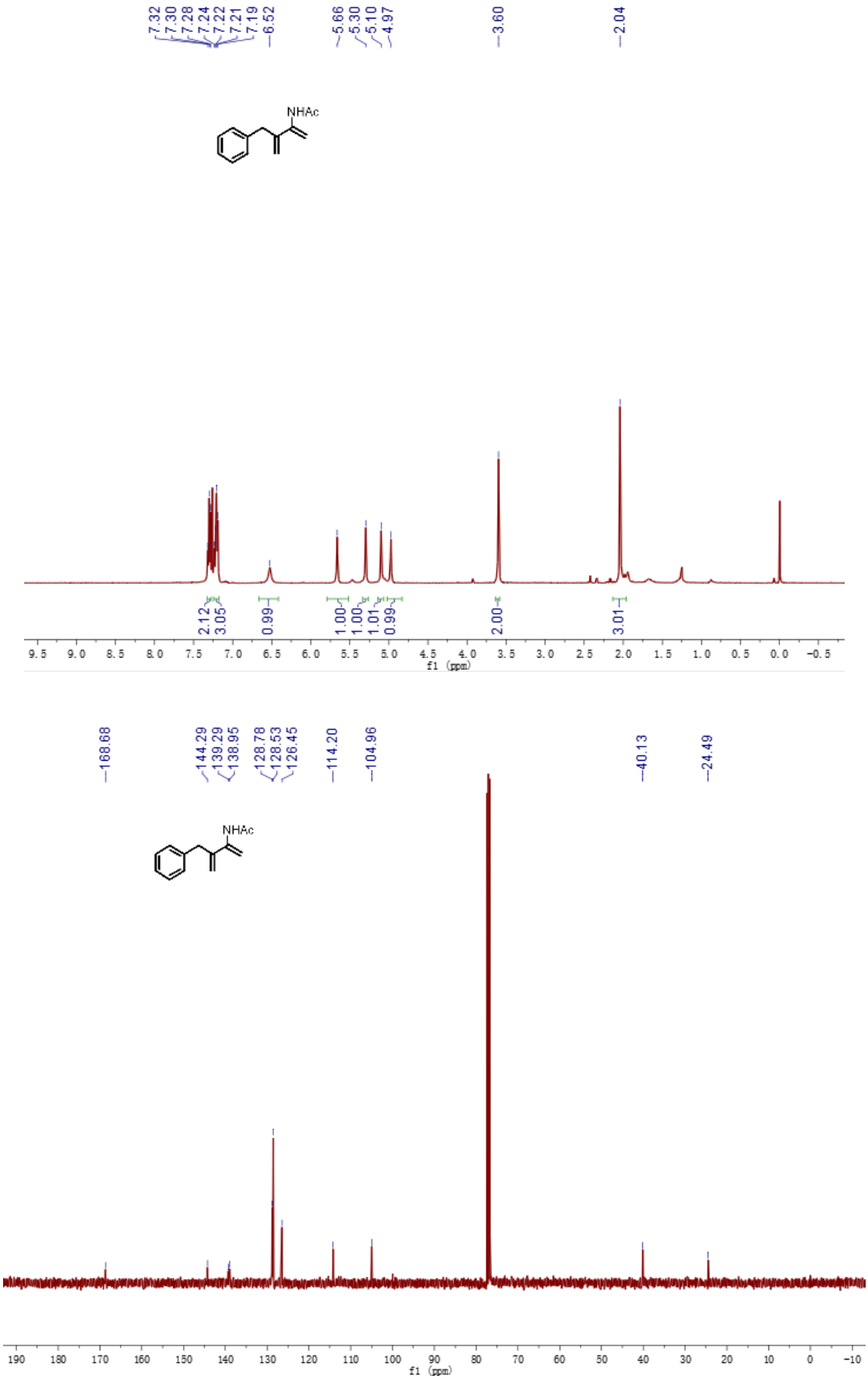


1i-^{1}H NMR and ^{13}C NMR

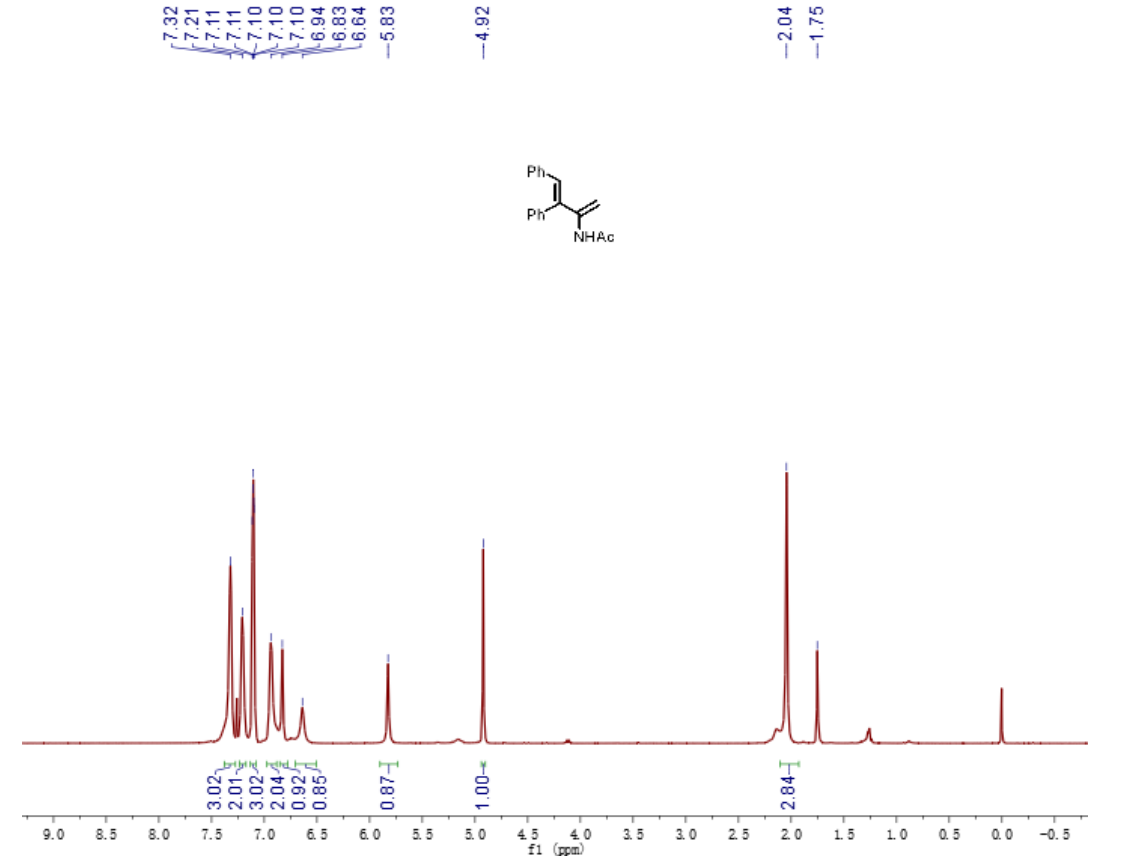


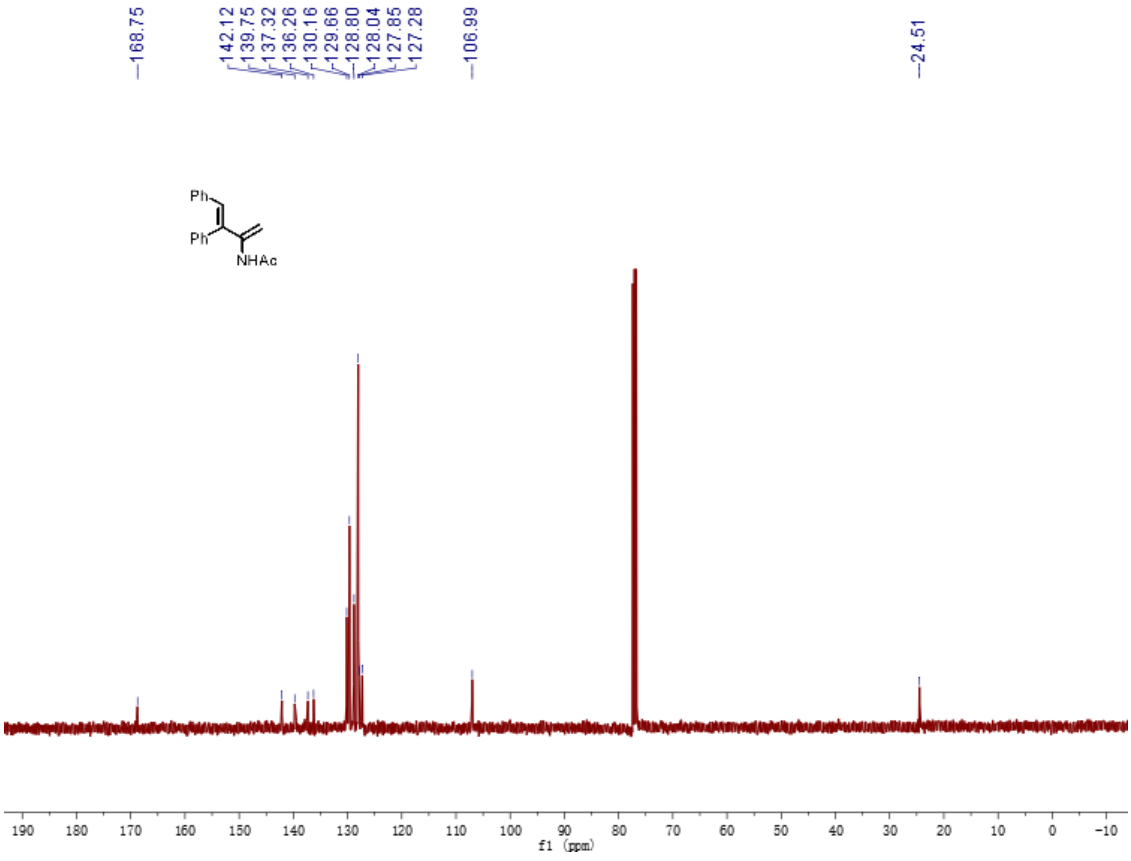


1j-^{1}H NMR and ^{13}C NMR

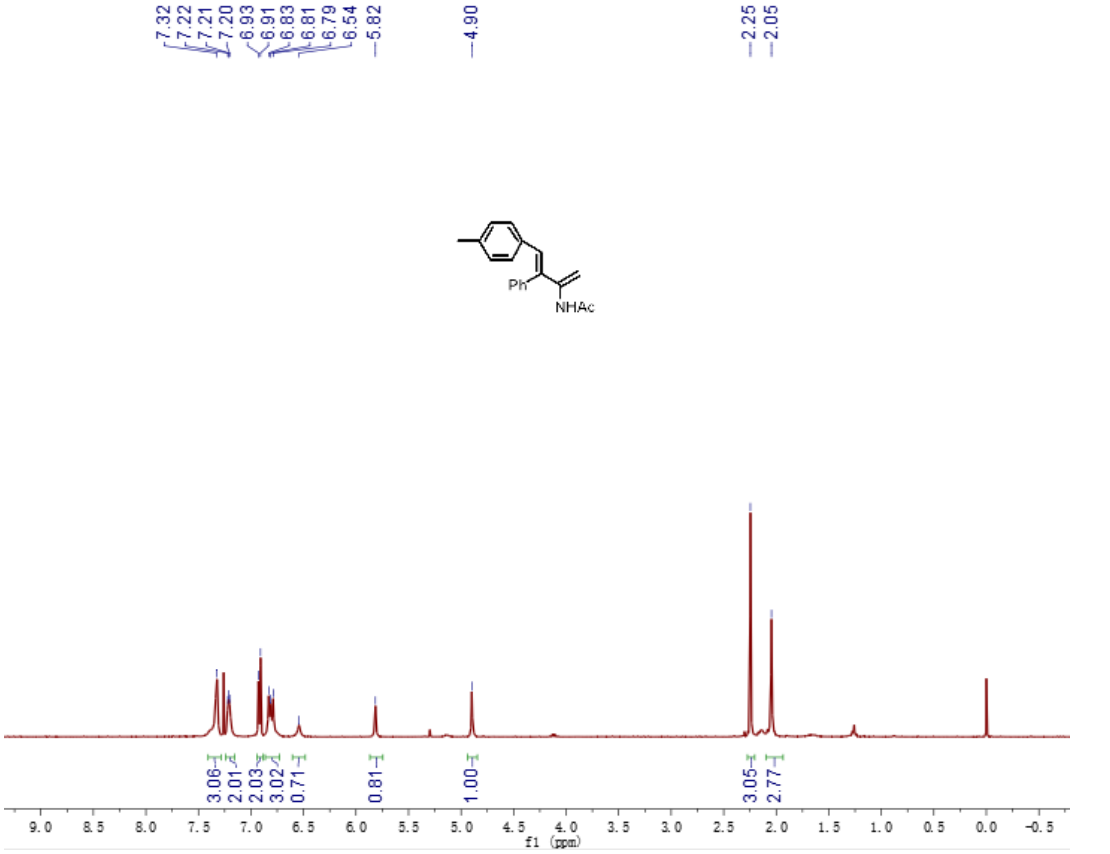


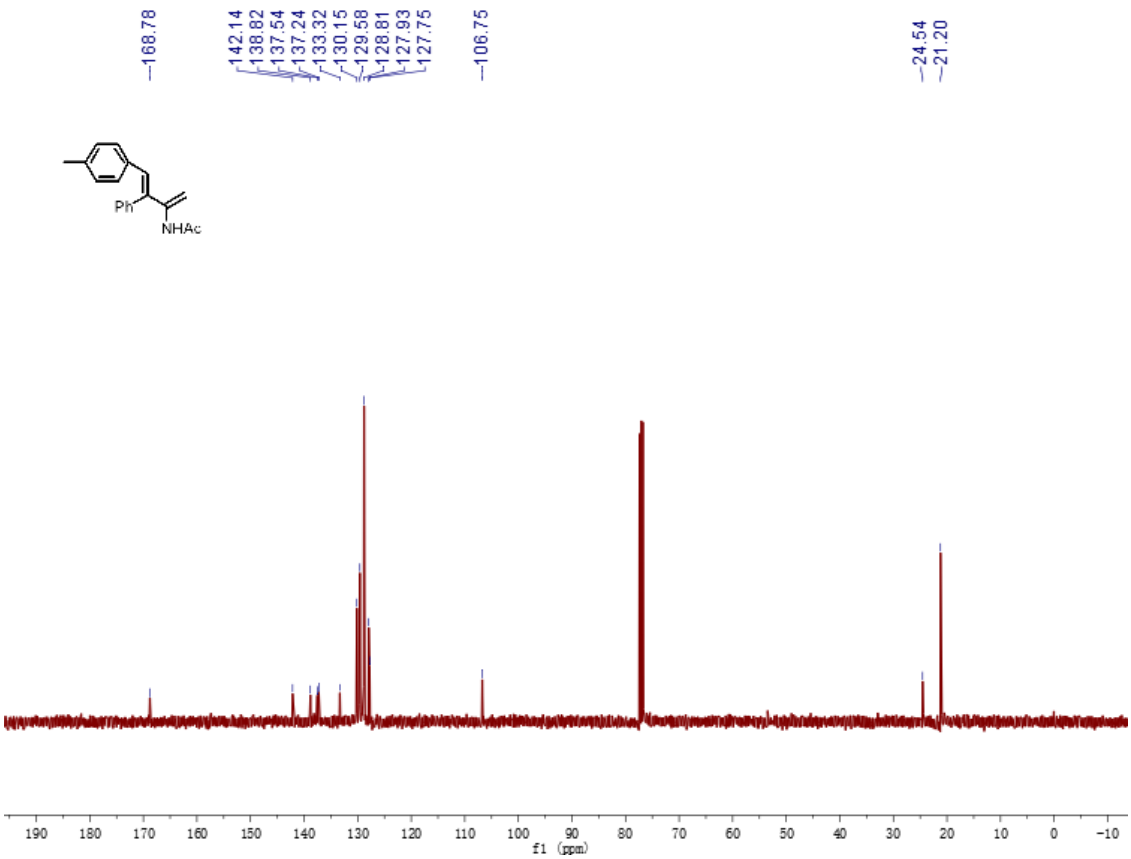
1k-^{1}H NMR and ^{13}C NMR



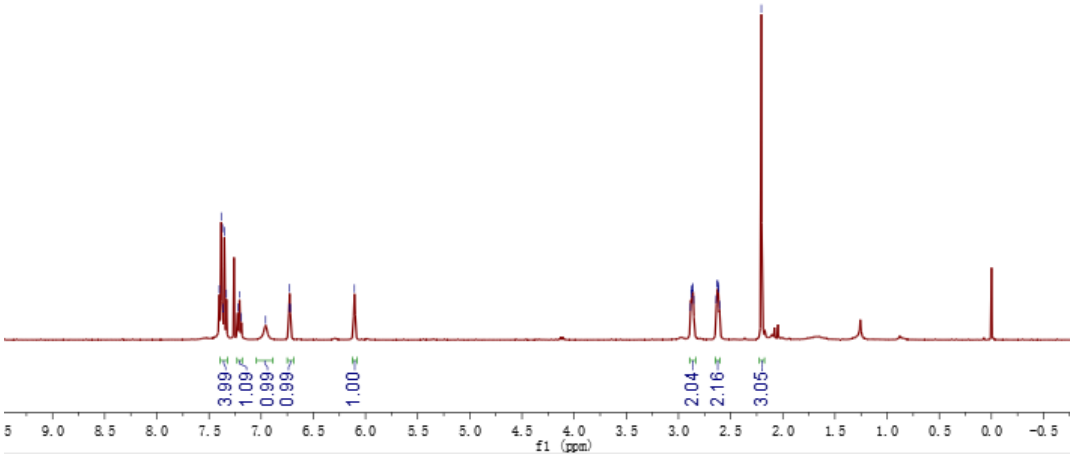


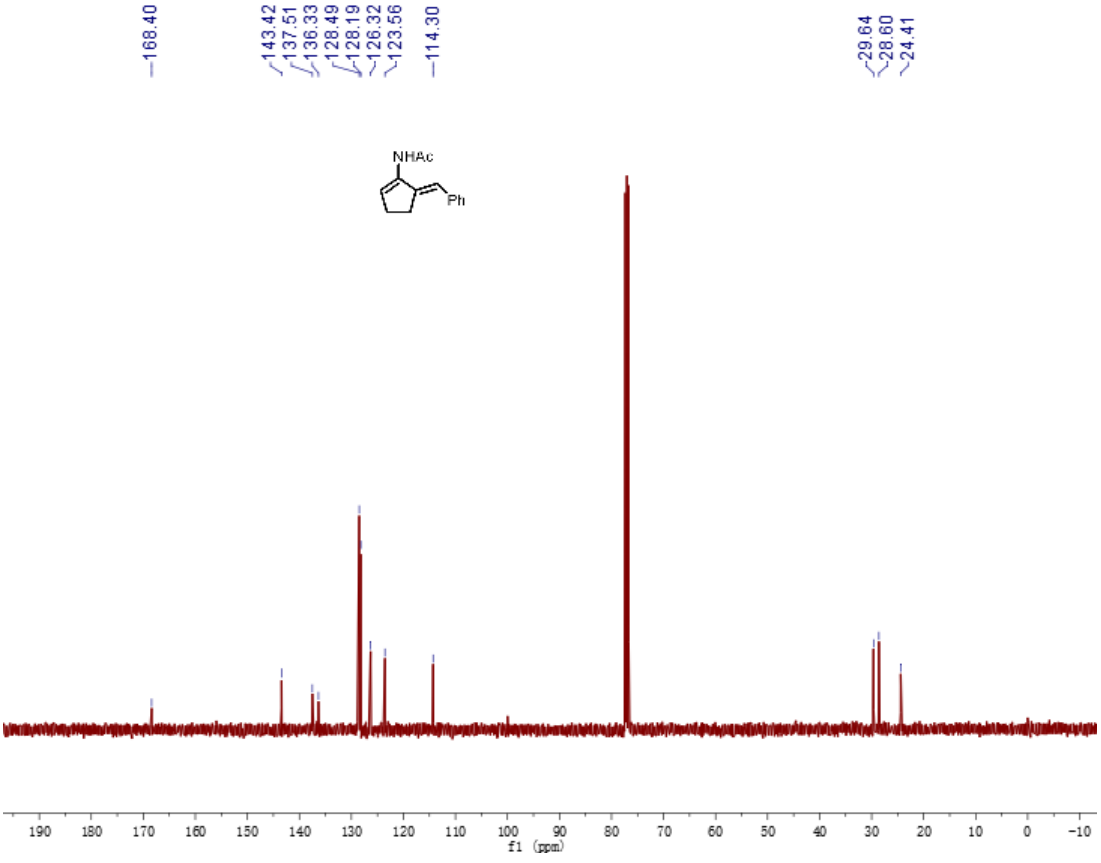
1l-^{1}H NMR and ^{13}C NMR



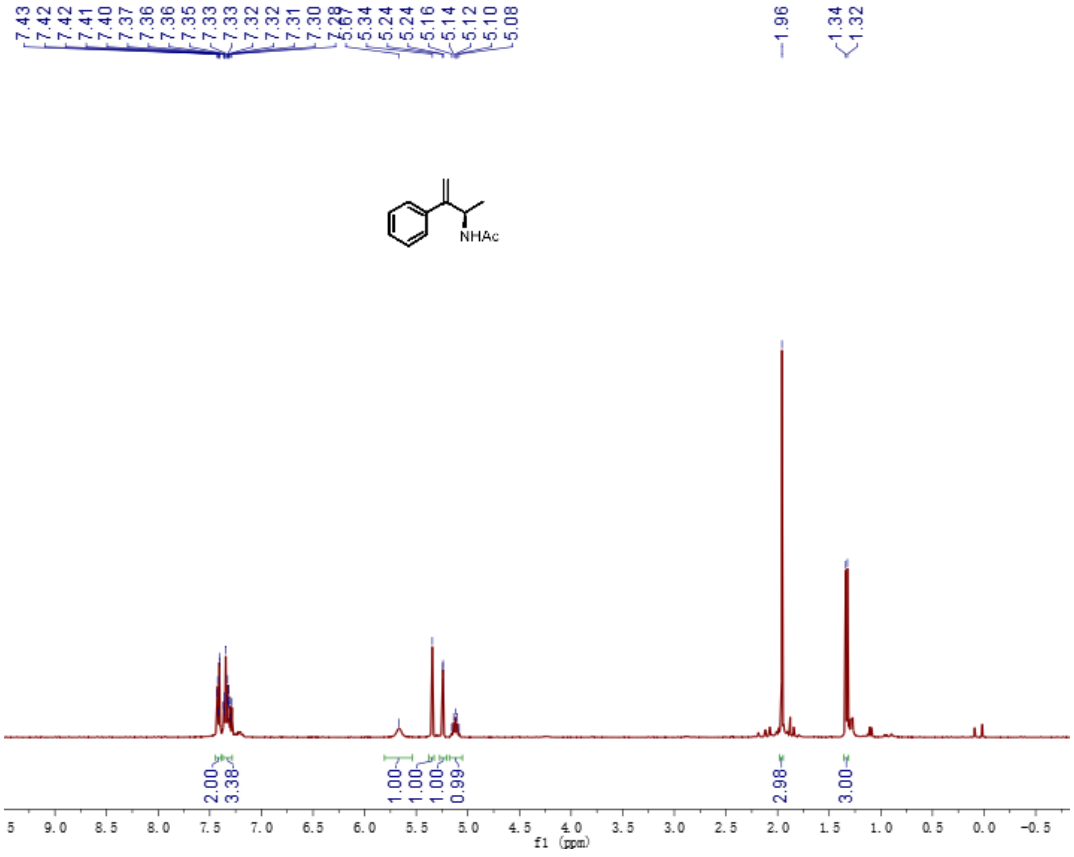


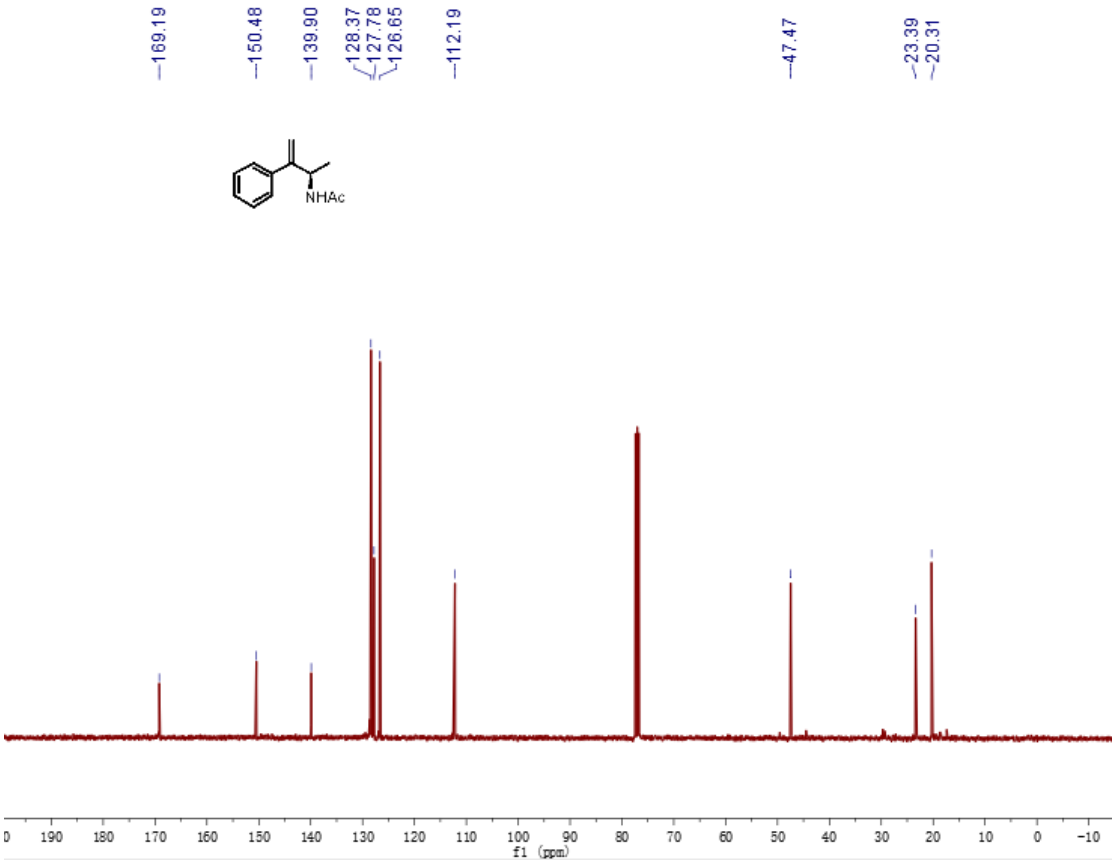
1m-^{1}H NMR and ^{13}C NMR



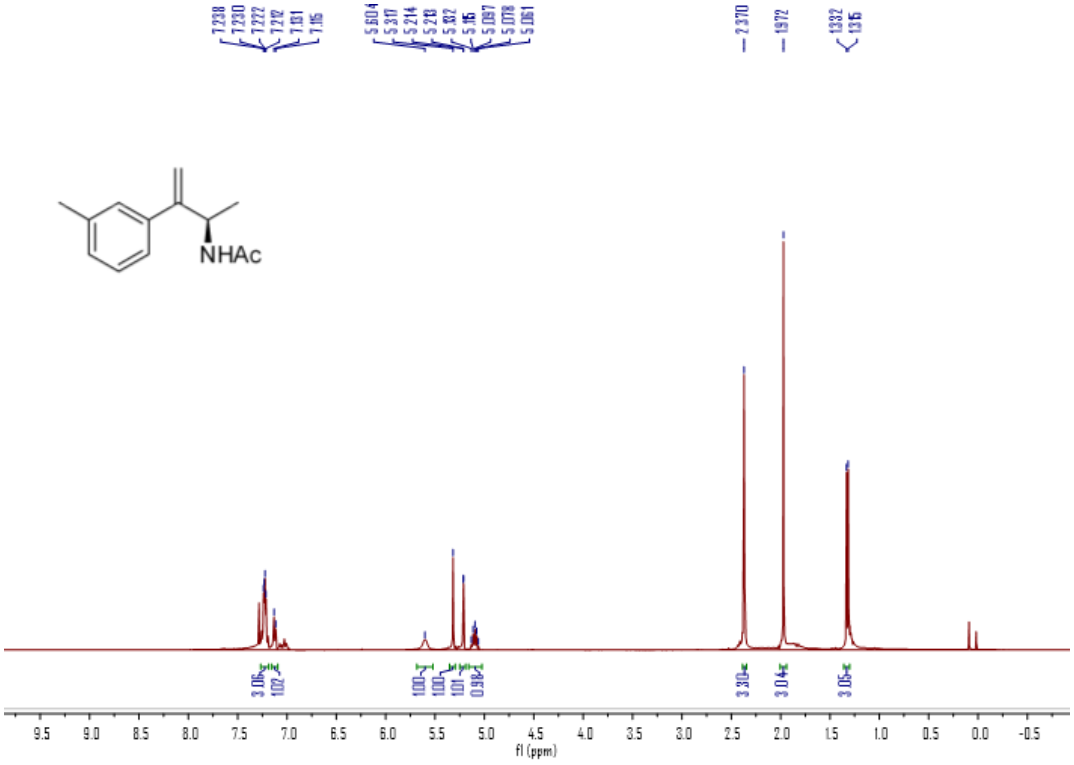


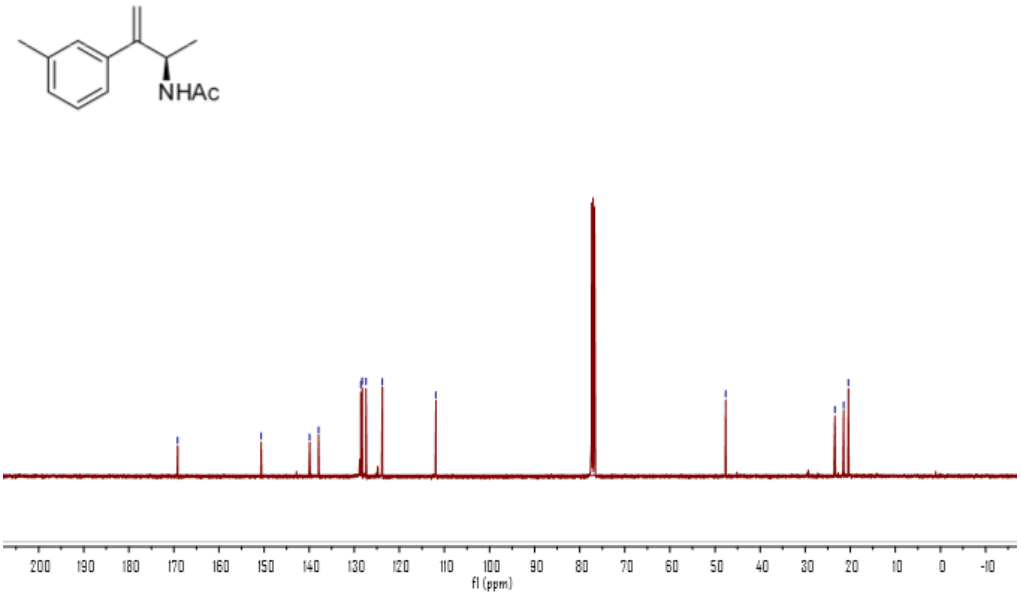
2a-^{1}H NMR and ^{13}C NMR



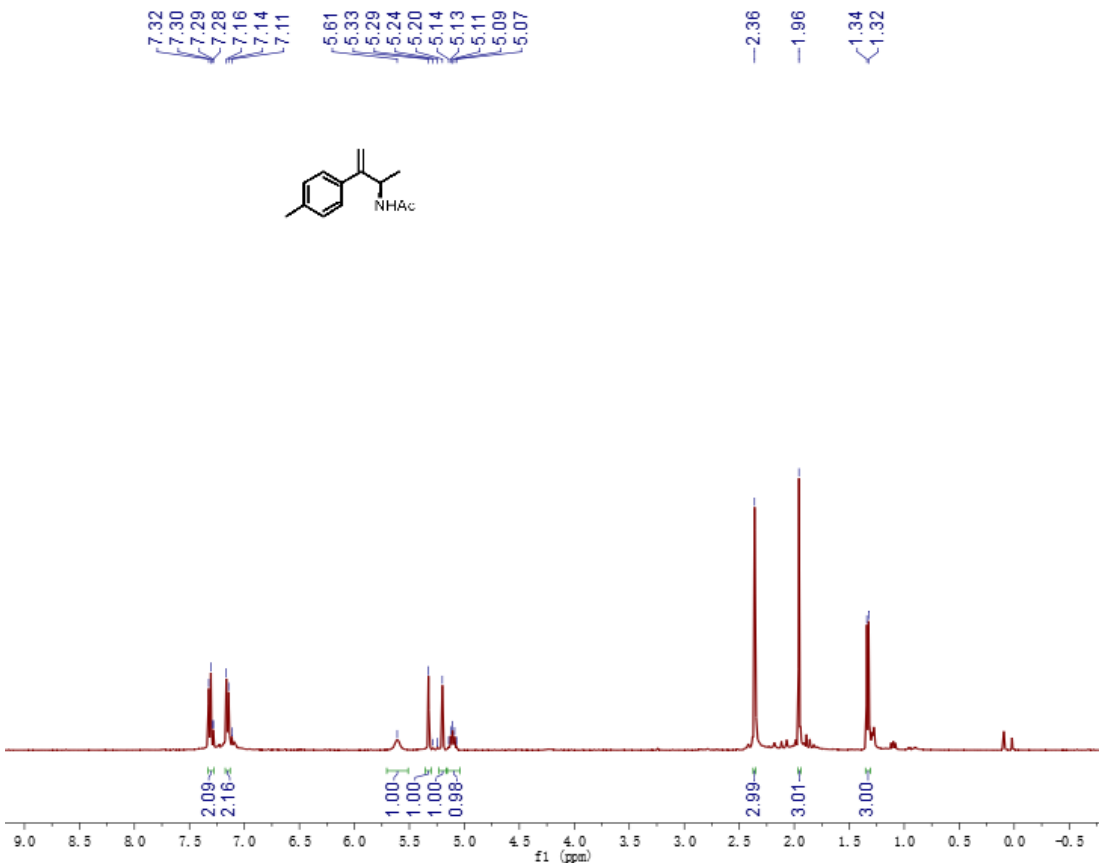


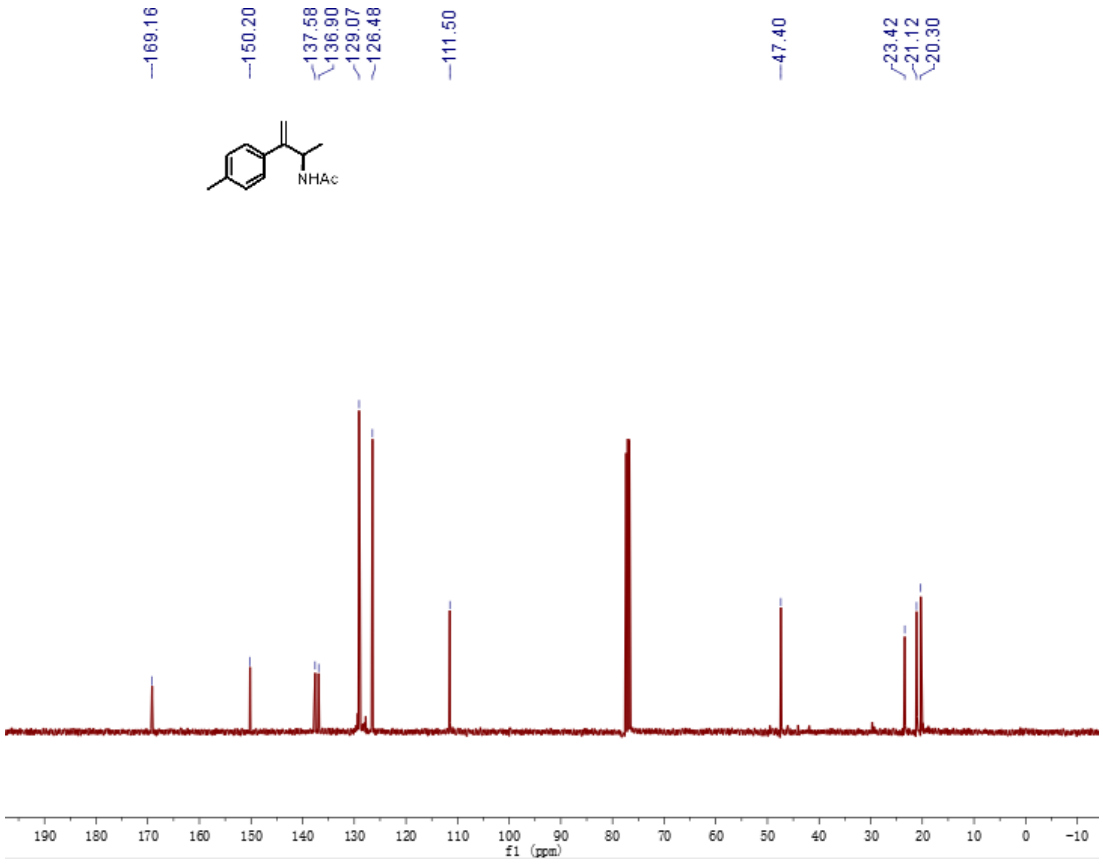
2b - ^{1}H NMR and ^{13}C NMR



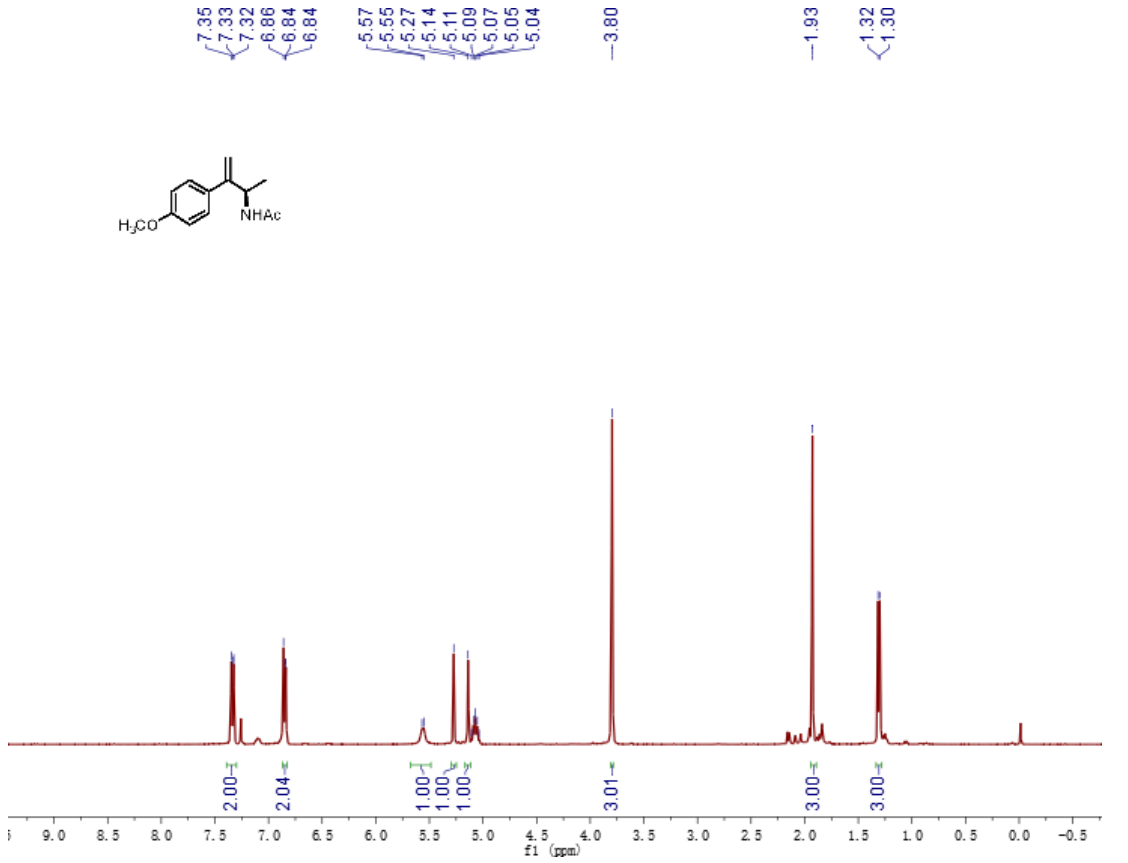


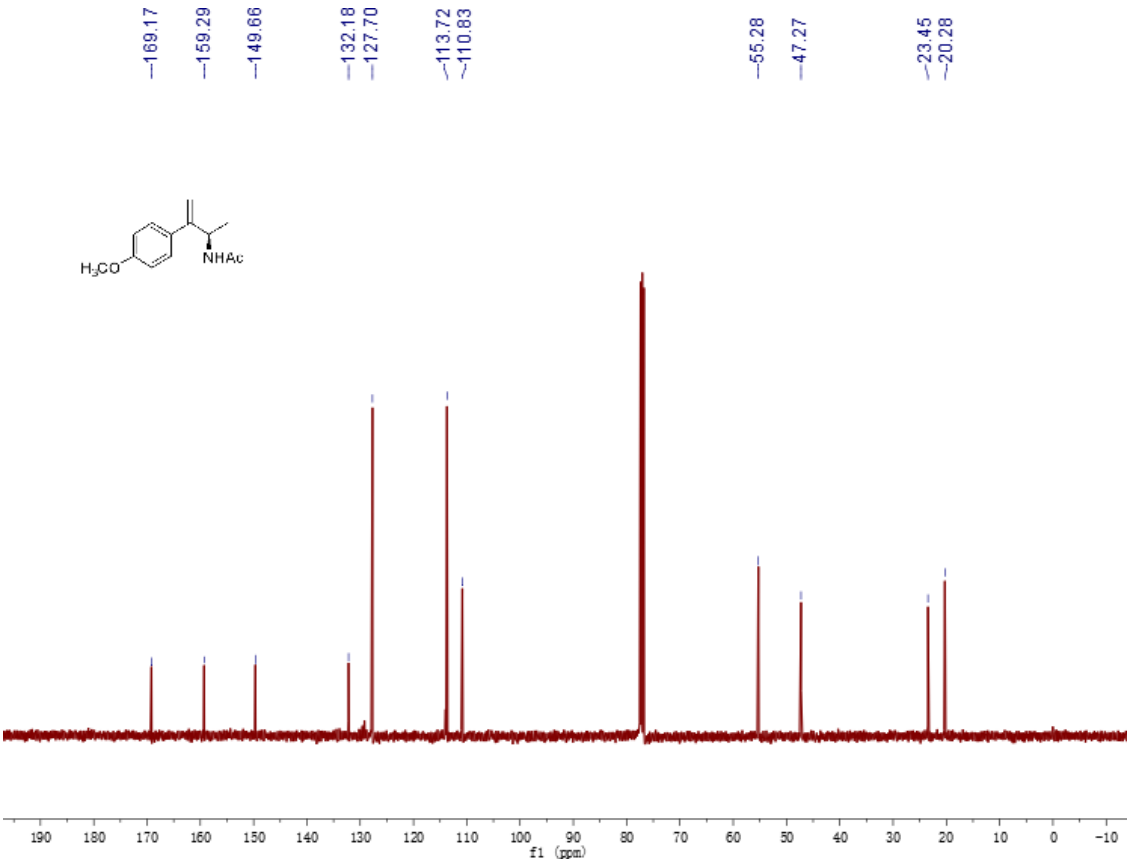
2c - ^{1}H NMR and ^{13}C NMR



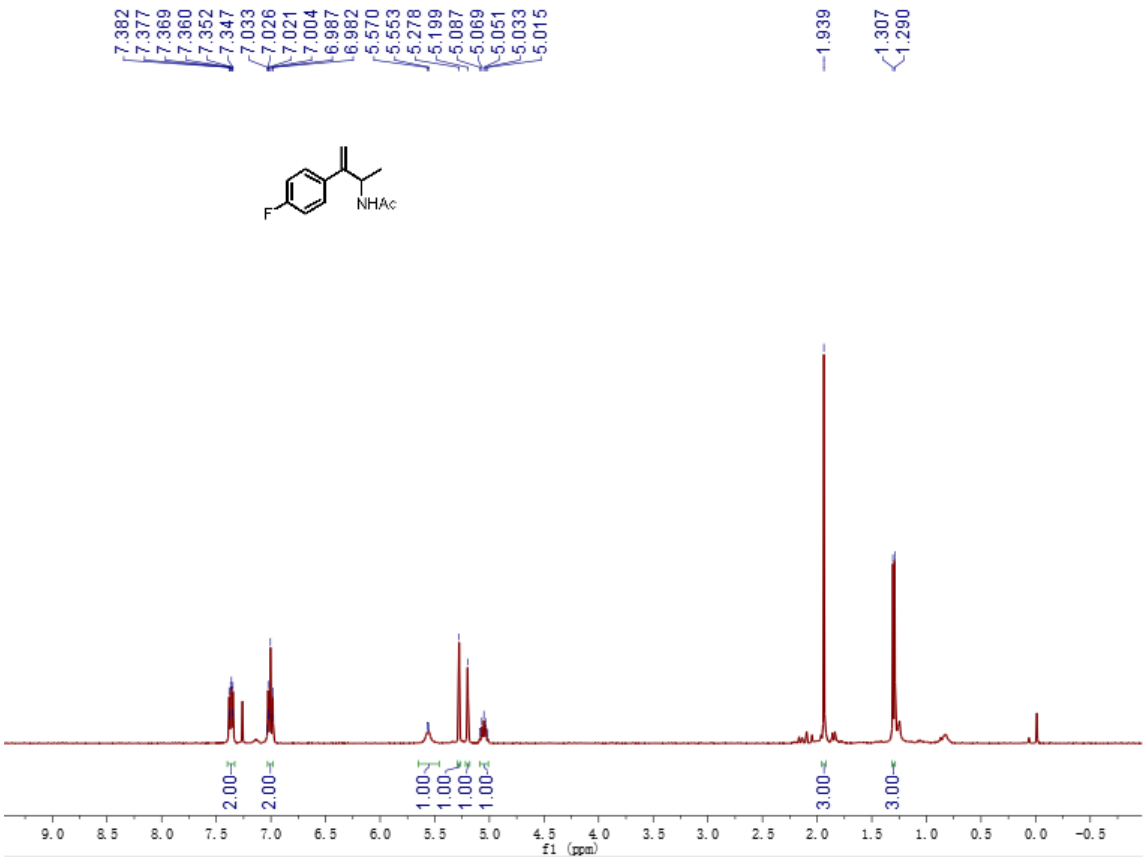


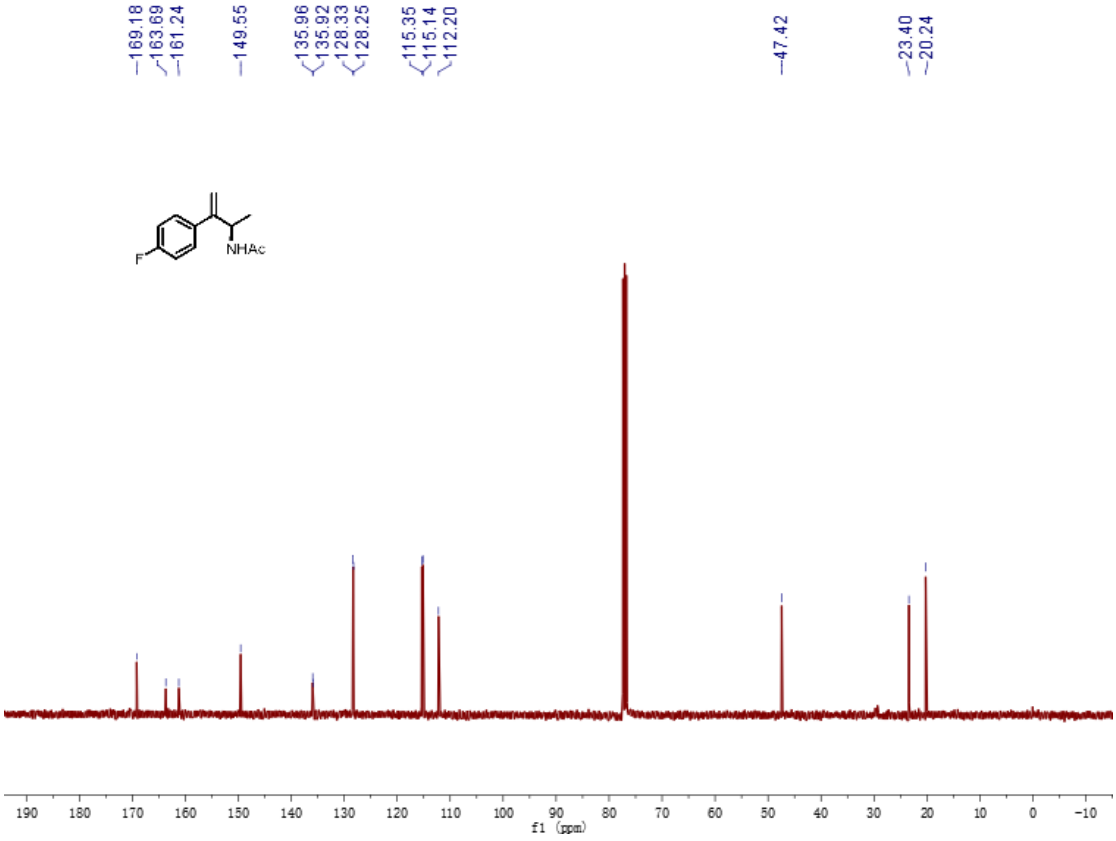
2d - ^{1}H NMR and ^{13}C NMR



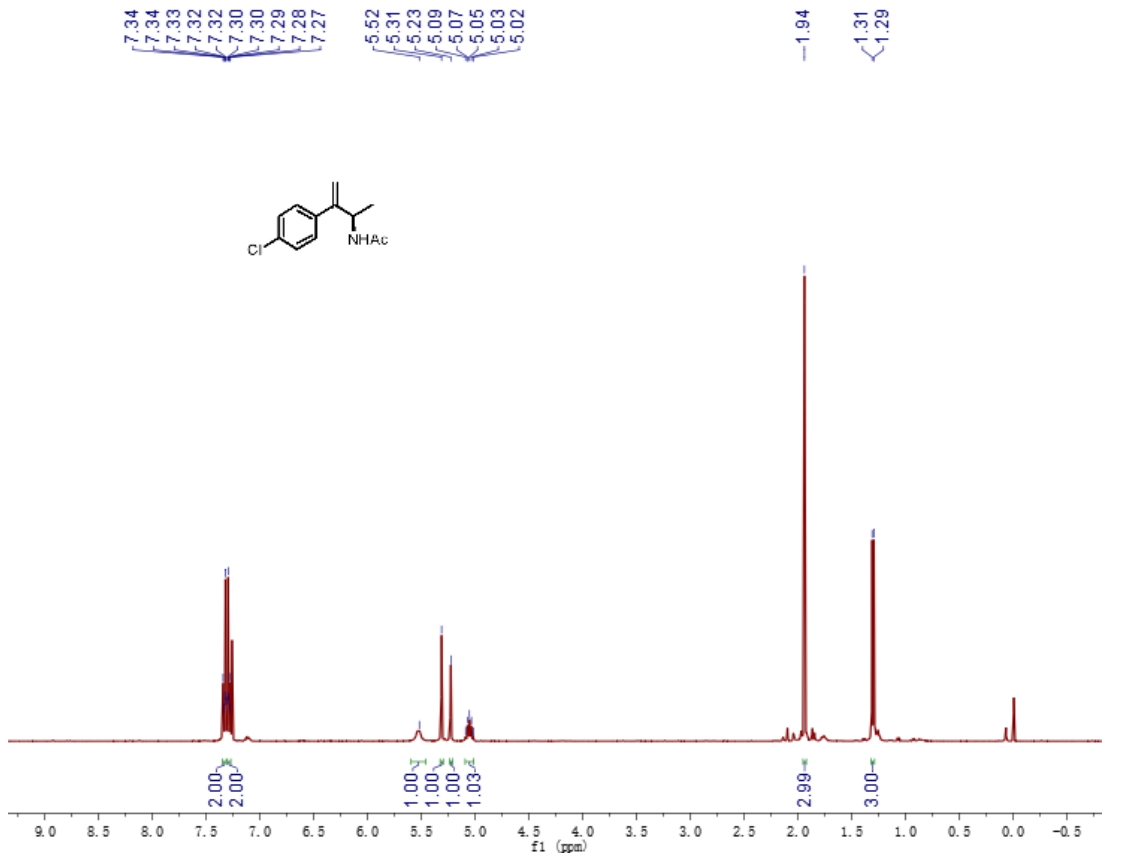


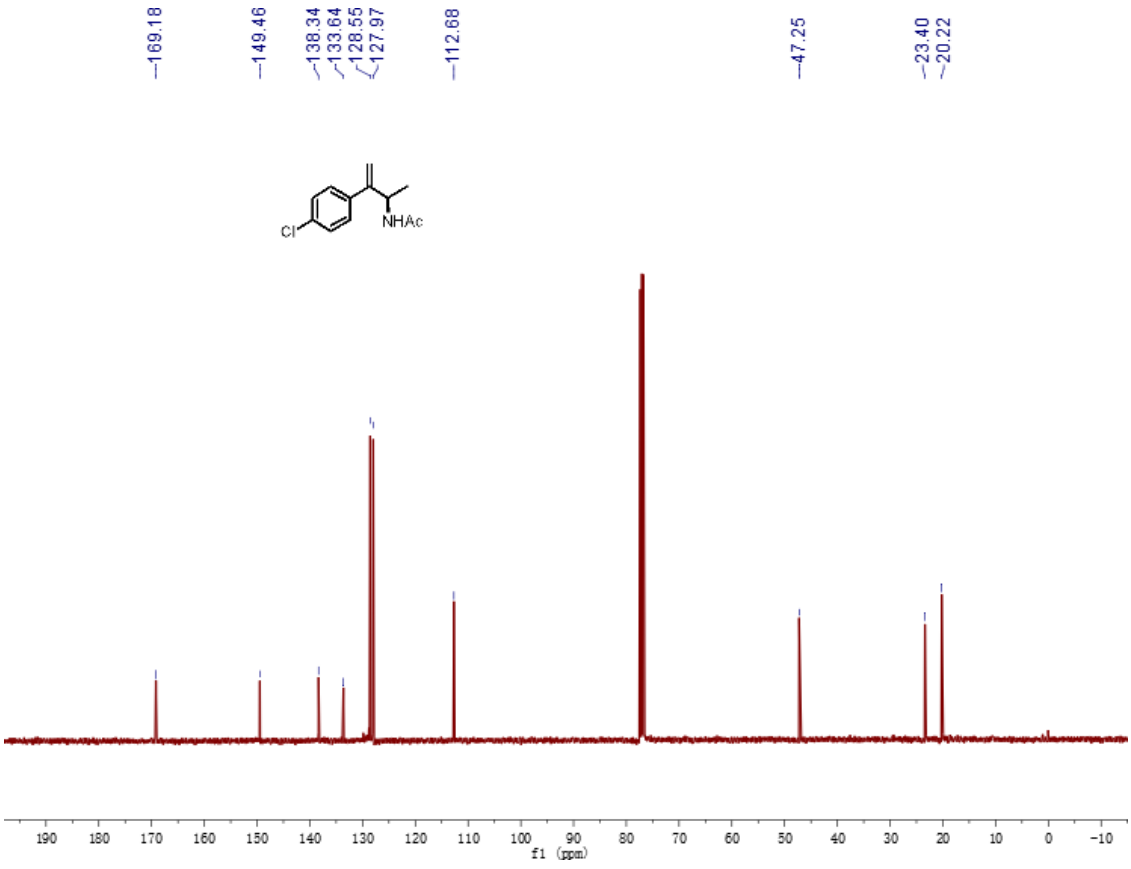
2e - ^{1}H NMR and ^{13}C NMR



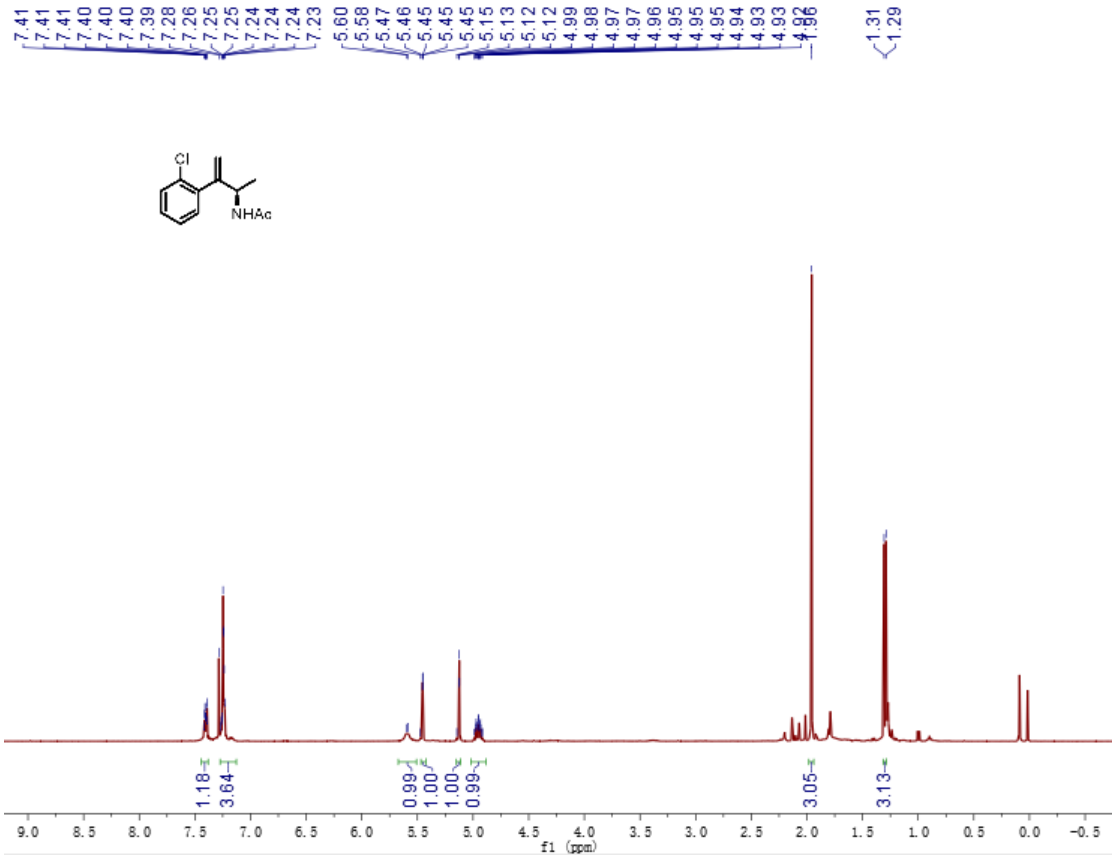


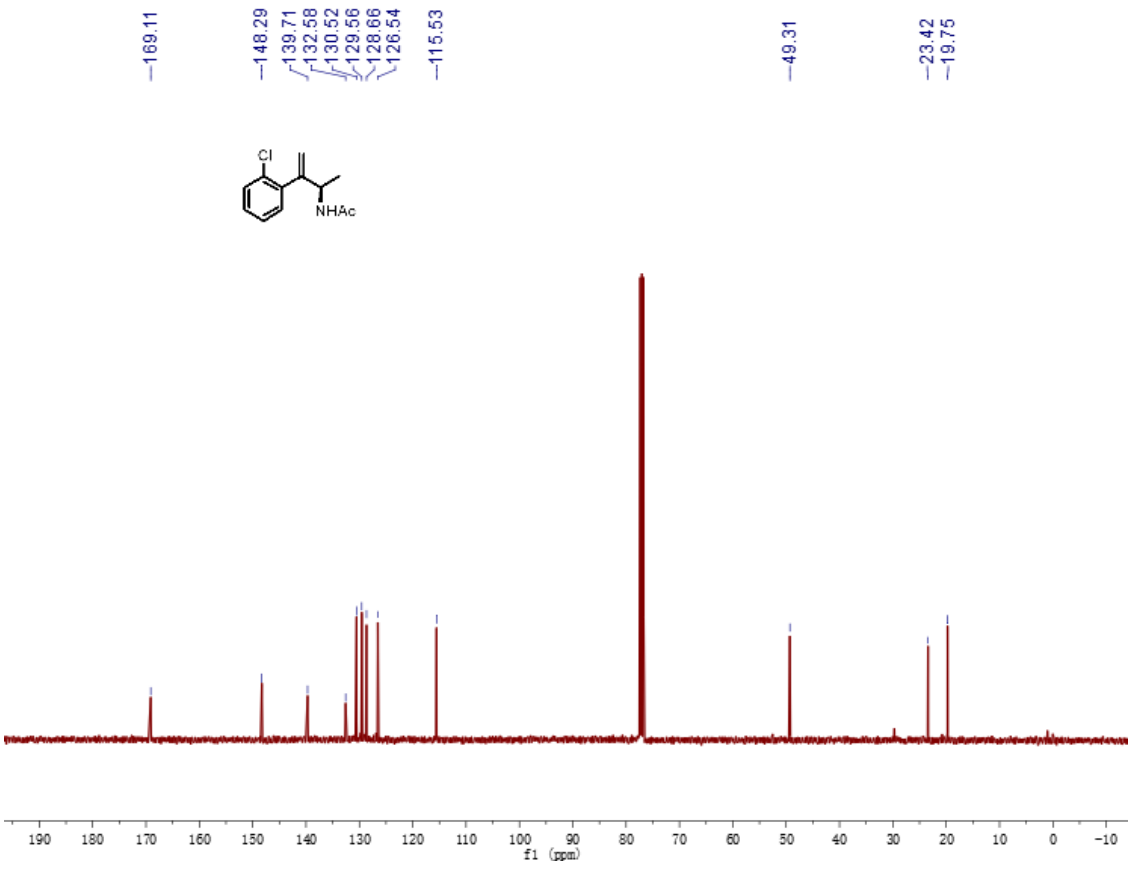
2f - ^{1}H NMR and ^{13}C NMR



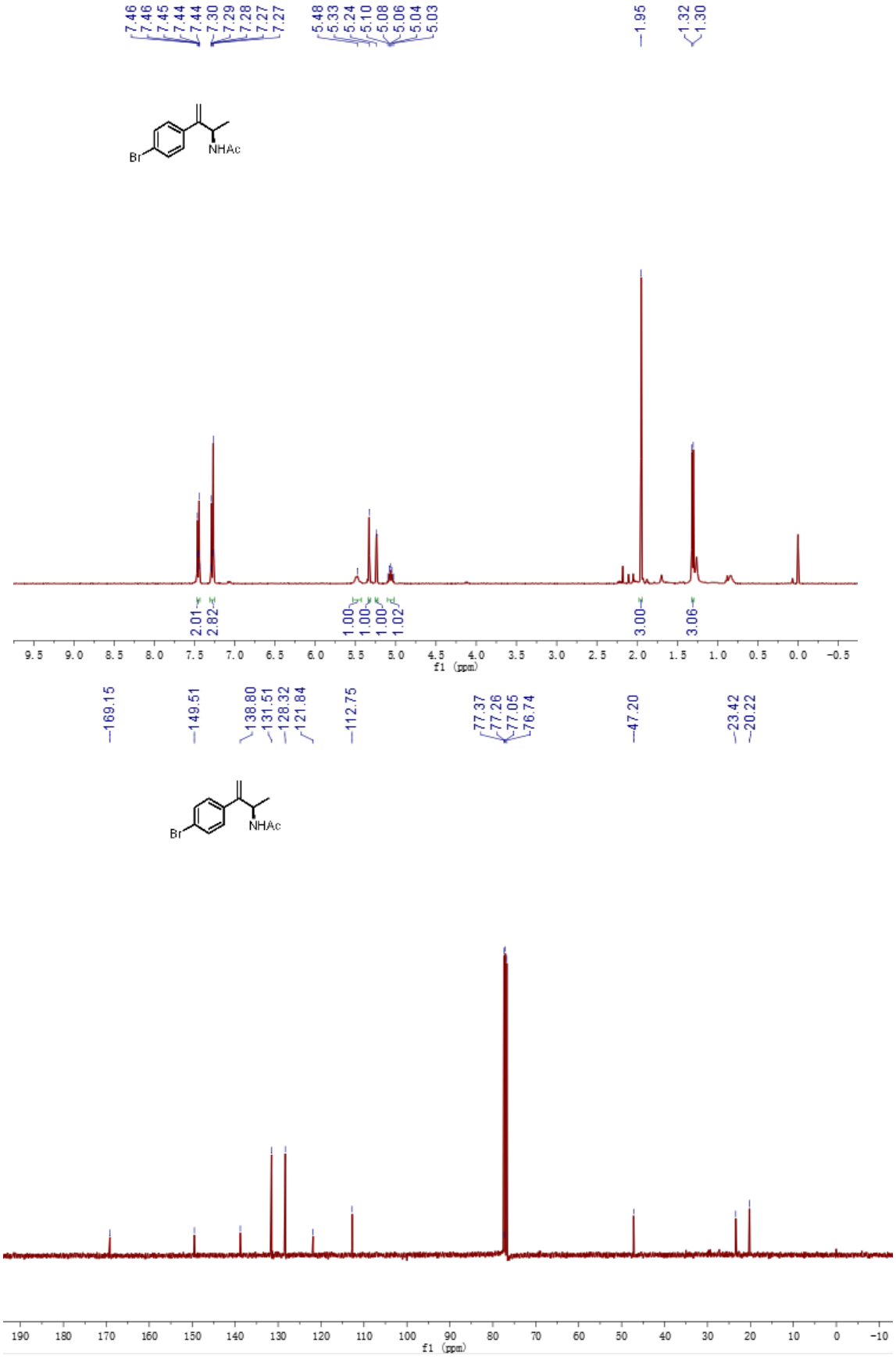


2g - ^{1}H NMR and ^{13}C NMR

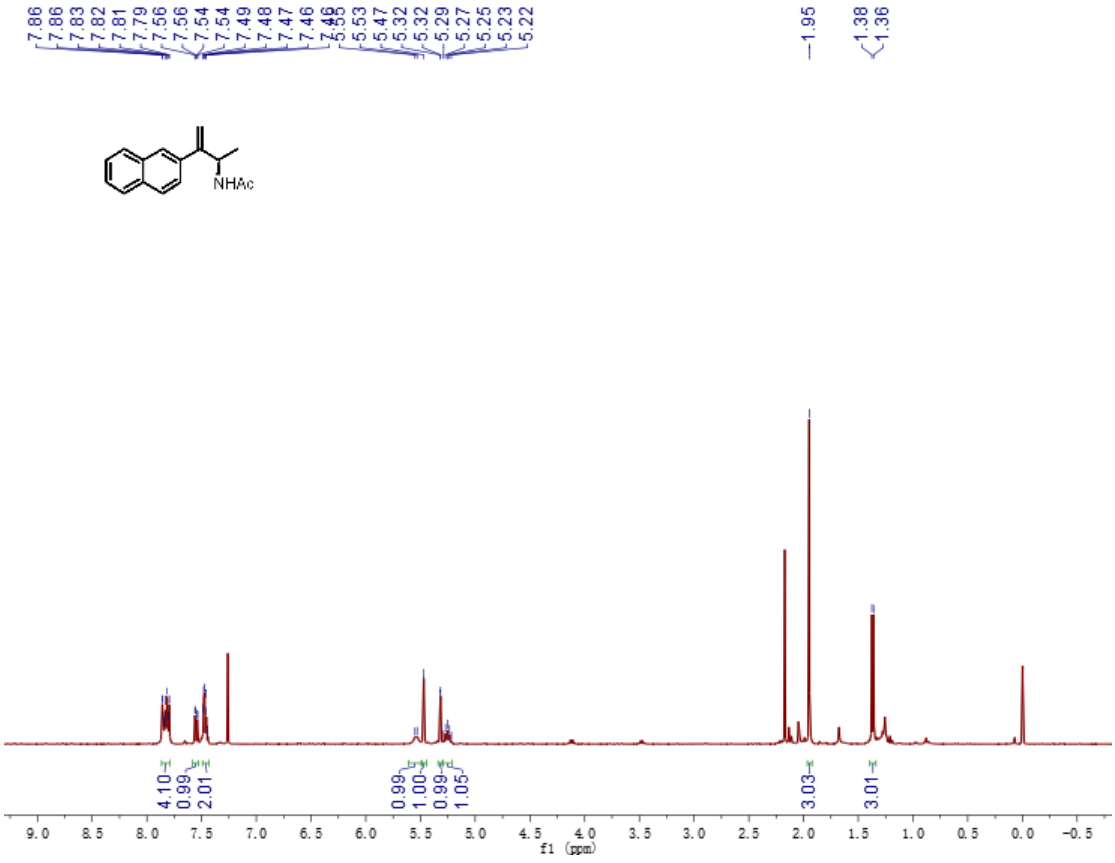


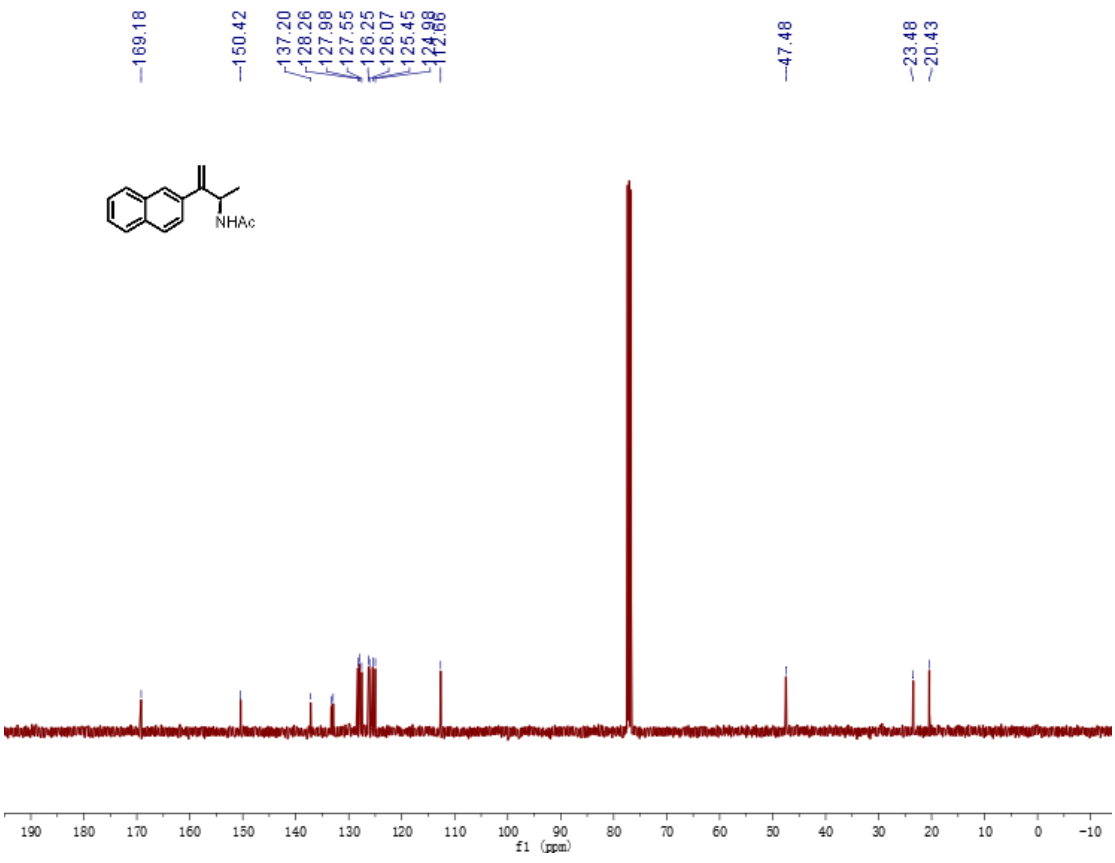


2h - ^{1}H NMR and ^{13}C NMR

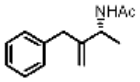


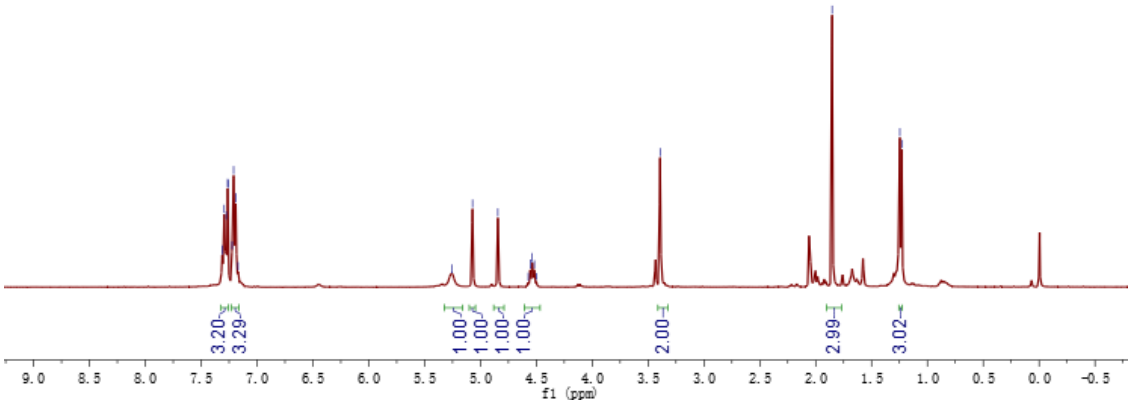
2i - ^{1}H NMR and ^{13}C NMR

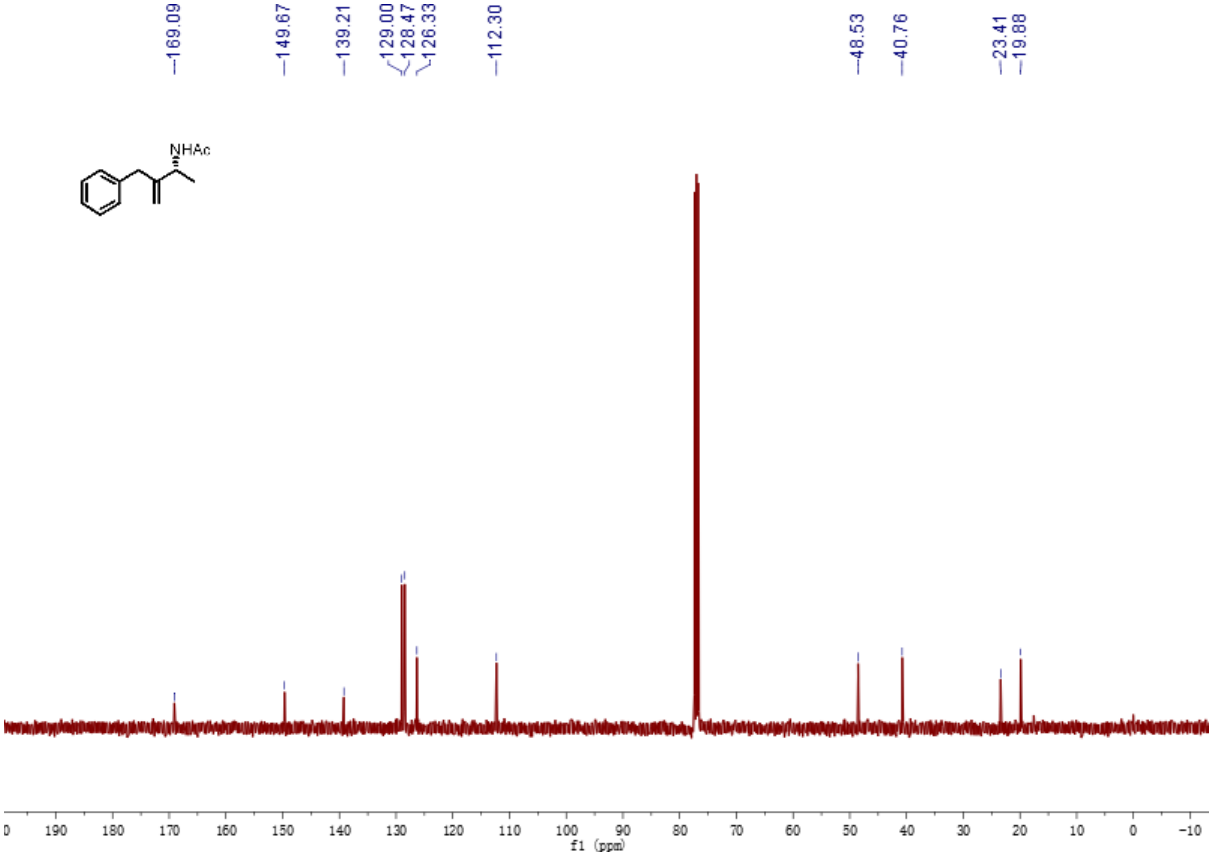




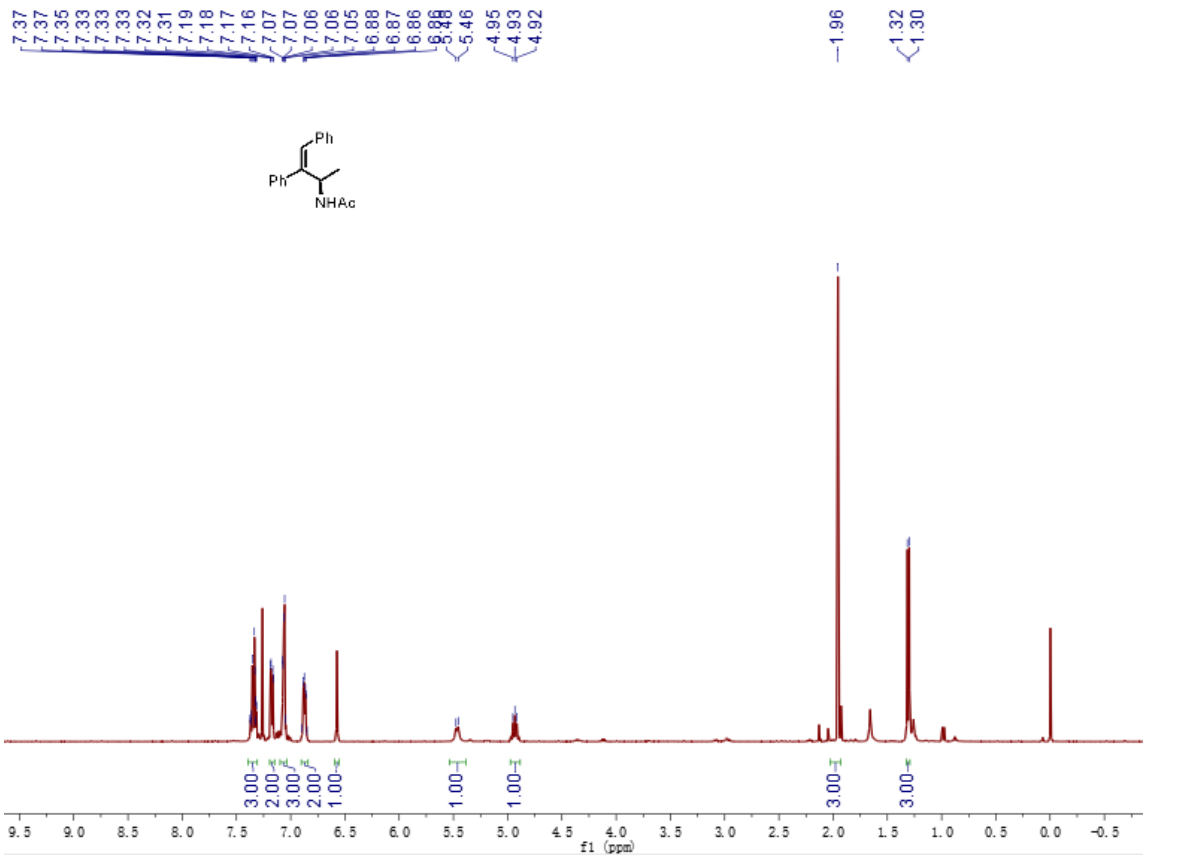
2j - ^{1}H NMR and ^{13}C NMR

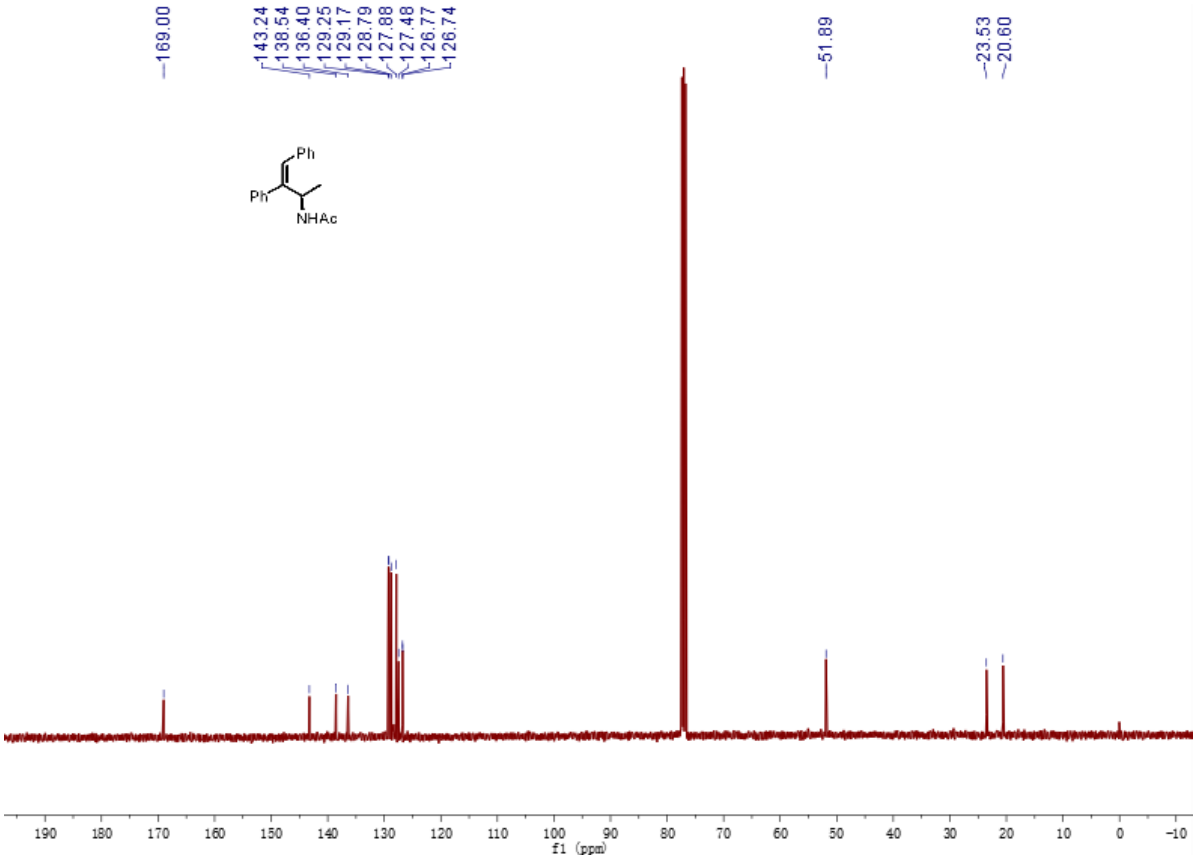




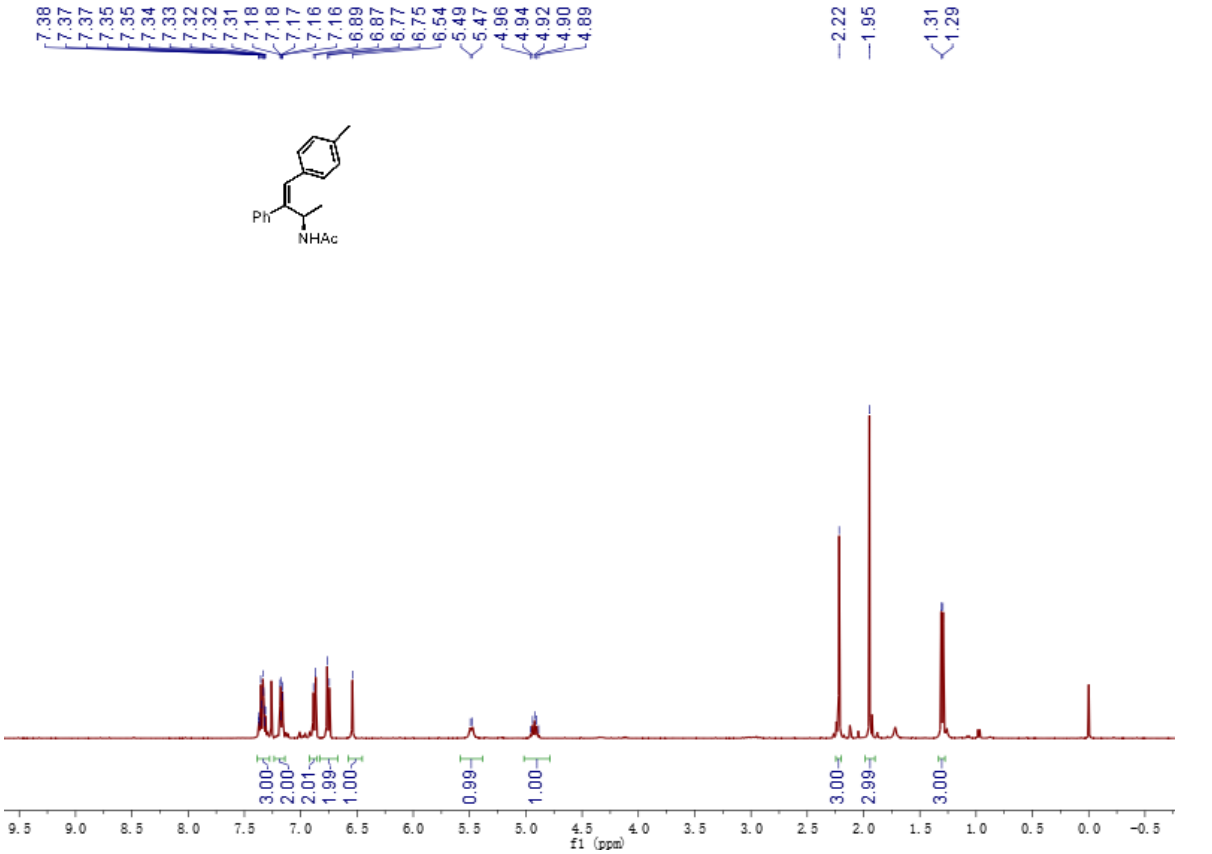


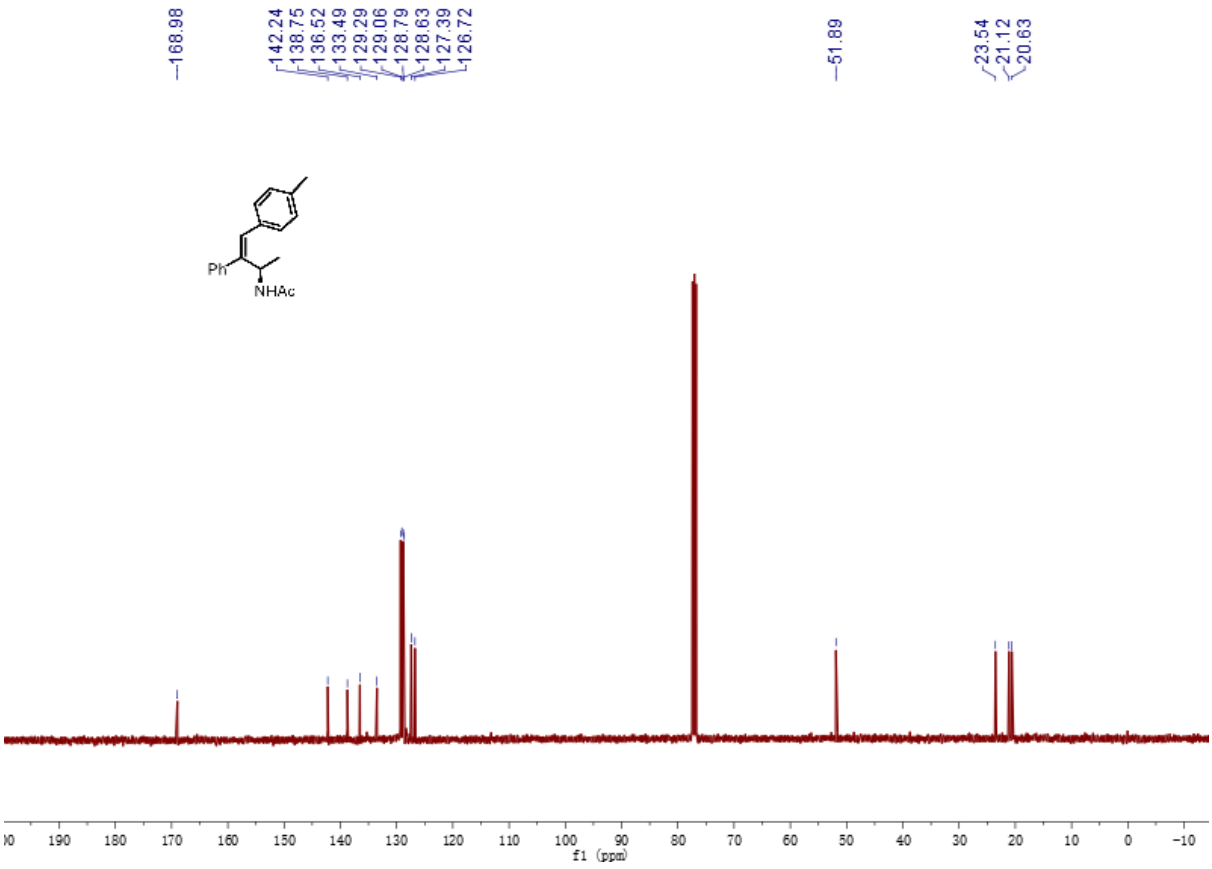
2k - ^{1}H NMR and ^{13}C NMR



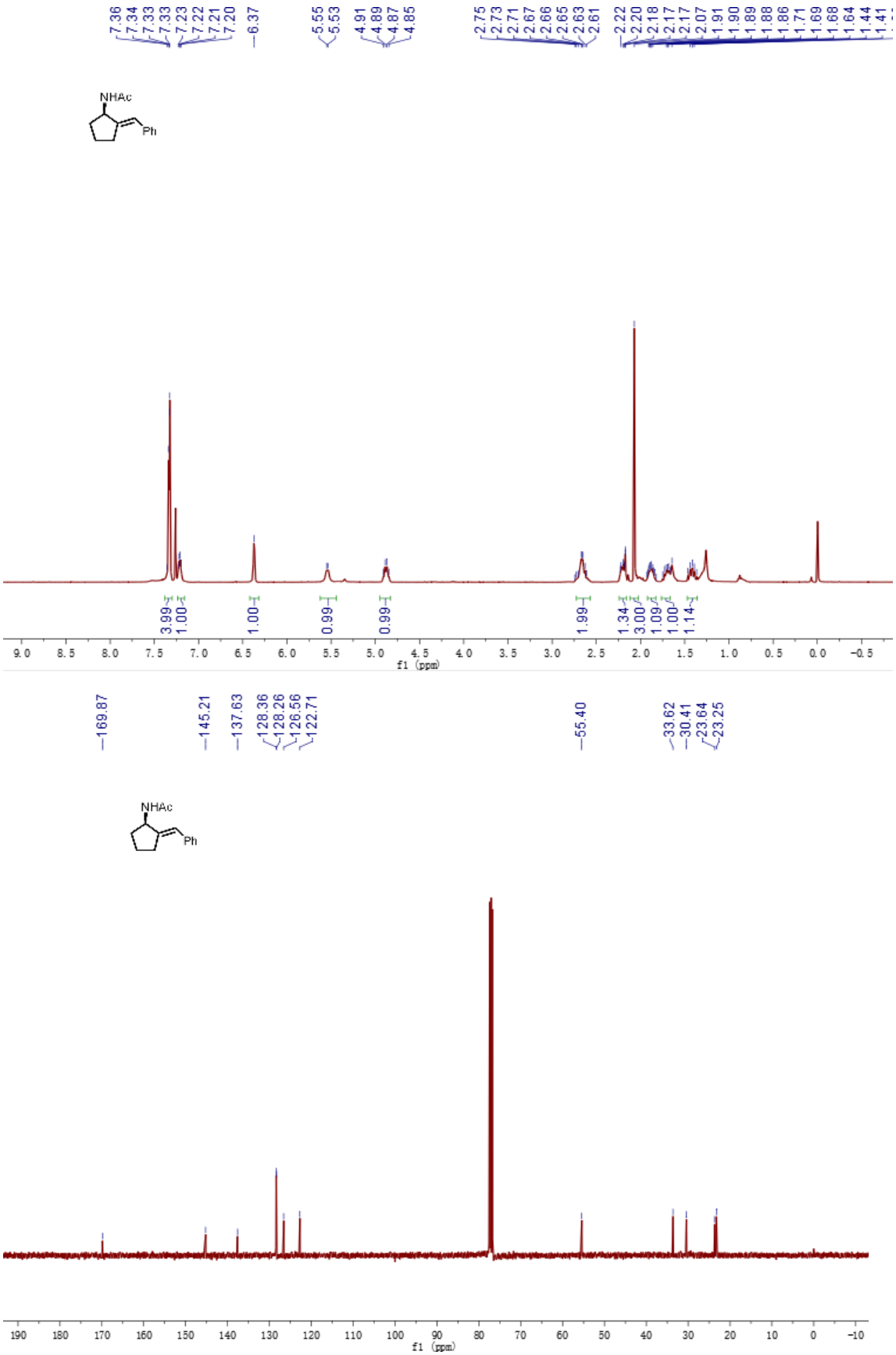


2l - ^{1}H NMR and ^{13}C NMR

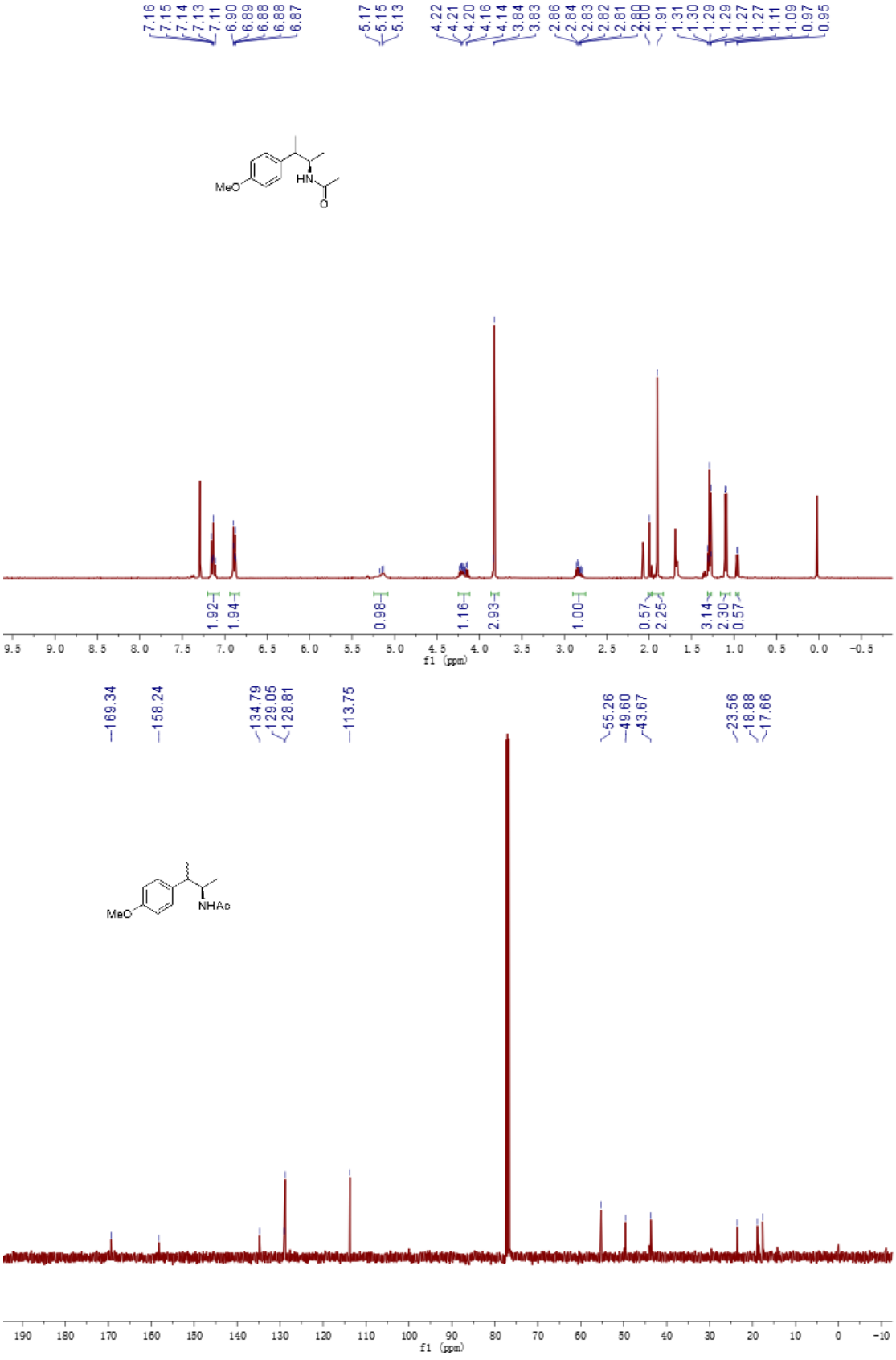




2m - ^{1}H NMR and ^{13}C NMR



3d - ^{1}H NMR and ^{13}C NMR



2a - HPLC

Data File E:\DATA\WQL\WQL-2-69-C\DAD-ODH-97-3-WQL-2-68-C 2013-05-14 16-26-27\043-0201.D
Sample Name: WQL-2-54-1

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    Injection Date  : 5/14/2013 4:38:25 PM                  Inj :   1
                                                    Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\WQL\WQL-2-69-C\DAD-ODH-97-3-WQL-2-68-C 2013-05-14 16-26-27\DAD-
                      ODH-95-5-1ML-40MIN(1-6).M
    Last changed    : 5/14/2013 4:26:27 PM by SYSTEM
    Analysis Method : E:\DATA\WQL\WQL-2-69-C\DAD-ODH-97-3-WQL-2-68-C 2013-05-14 16-26-27\DAD-
                      ODH-95-5-1ML-40MIN(1-6).M (Sequence Method)
    Last changed    : 4/29/2015 3:09:38 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
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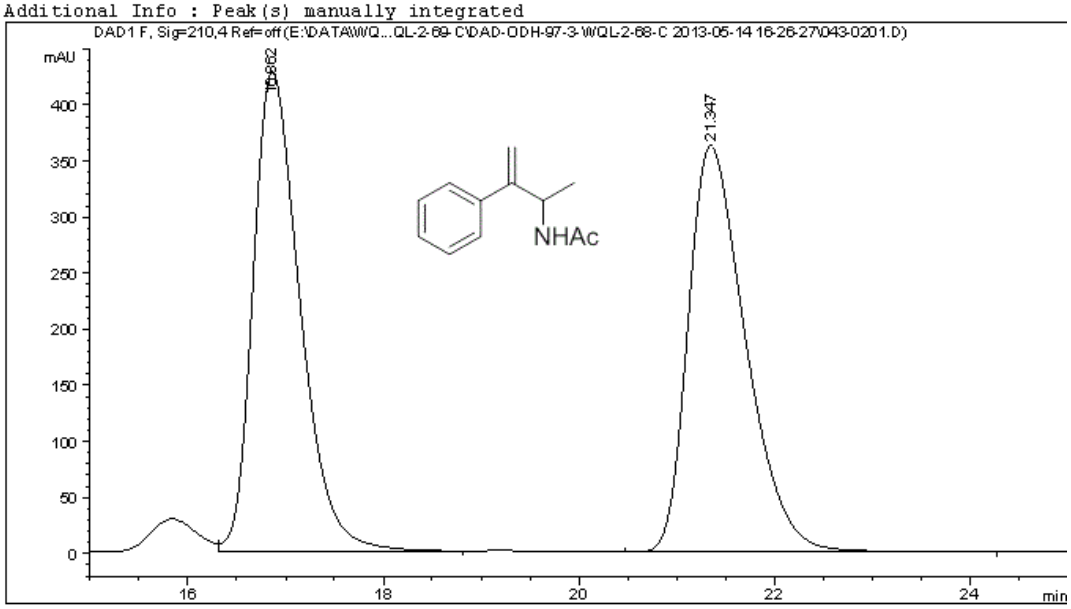


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    ======================================================================
                            Area Percent Report
    ======================================================================

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


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

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  16.862 VB    0.5145 1.43278e4   428.00723  49.1943
       2  21.347 BB    0.6222 1.47972e4   362.55991  50.8057

    Totals :                  2.91250e4   790.56714


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

1260HPLC-VWD 4/29/2015 3:09:48 PM SYSTEM                    Page 1 of 1
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Data File E:\DATA\WQL\WQL-2-69-C\DAD-ODH-97-3-WQL-2-68-C 2013-05-14 16-26-27\047-0501.D
Sample Name: WQL-2-69-7

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                                                   Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\WQL\WQL-2-69-C\DAD-ODH-97-3-WQL-2-68-C 2013-05-14 16-26-27\DAD-
                      ODH-95-5-1ML-40MIN(1-6).M
    Last changed    : 5/14/2013 4:26:27 PM by SYSTEM
    Analysis Method : E:\DATA\WQL\WQL-2-69-C\DAD-ODH-97-3-WQL-2-68-C 2013-05-14 16-26-27\DAD-
                      ODH-95-5-1ML-40MIN(1-6).M (Sequence Method)
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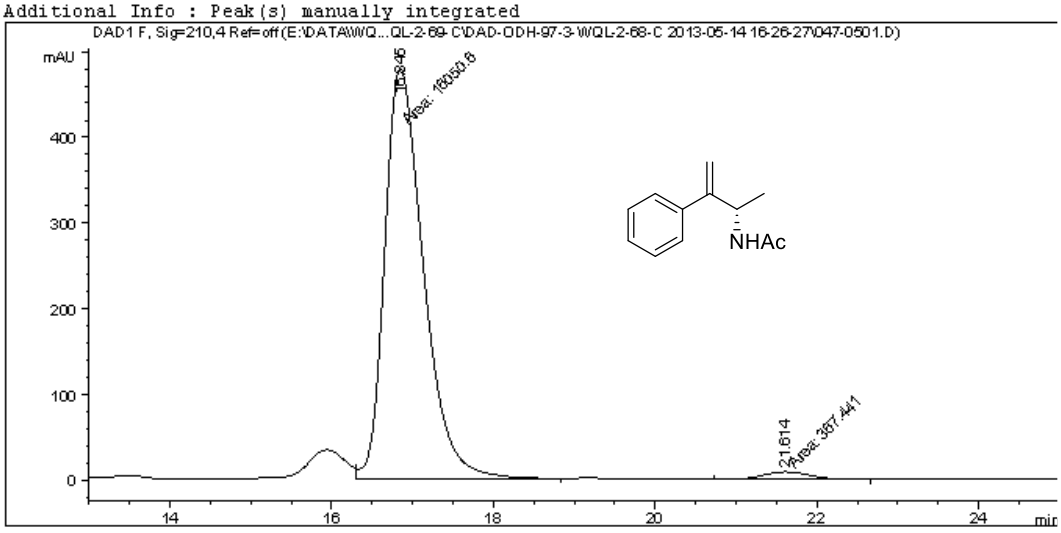


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    ==================================================================
                            Area Percent Report
    ==================================================================

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


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

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  16.845 FM    0.5580 1.60506e4   479.42480  97.7620
       2  21.614 MM    0.6807  367.44070    8.99603   2.2380

    Totals :                  1.64180e4   488.42083


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2b – HPLC

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Data File E:\DATA\WQL\WQL-2-78-A\VWD-OJH--95-5-WQL-2-78-A 2013-06-14 21-02-41\023-0401.D
Sample Name: WQL-2-74-3

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                                                   Inj Volume : 5.000 µl
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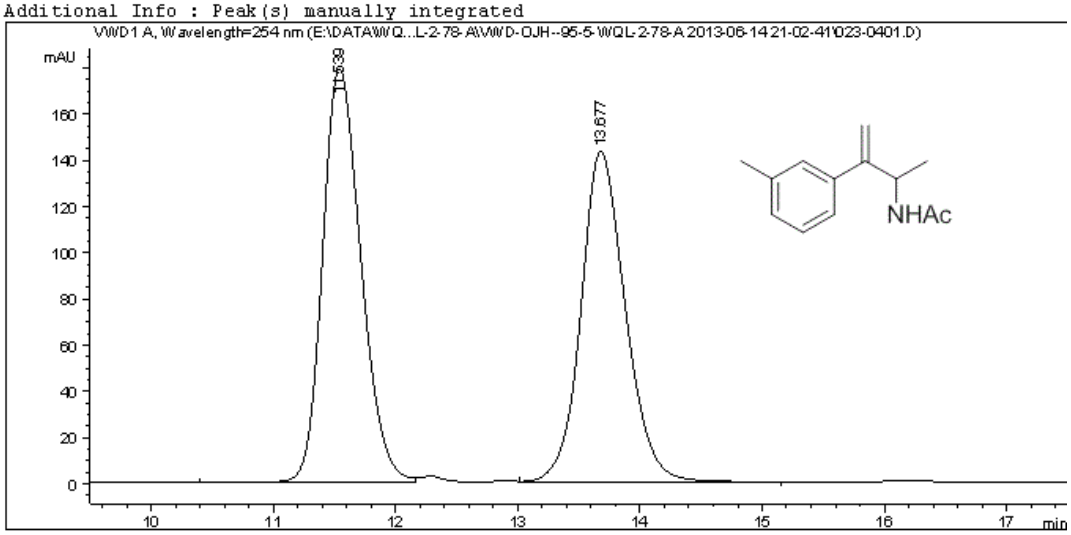
                      OJH-95-5-1ML-254NM-40MIN(1-2).M
    Last changed    : 6/14/2013 9:02:42 PM by SYSTEM
    Analysis Method : E:\DATA\WQL\WQL-2-78-A\VWD-OJH--95-5-WQL-2-78-A 2013-06-14 21-02-41\VWD-
                      OJH-95-5-1ML-254NM-40MIN(1-2).M (Sequence Method)
    Last changed    : 4/29/2015 3:43:42 PM by SYSTEM
                      (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=254 nm

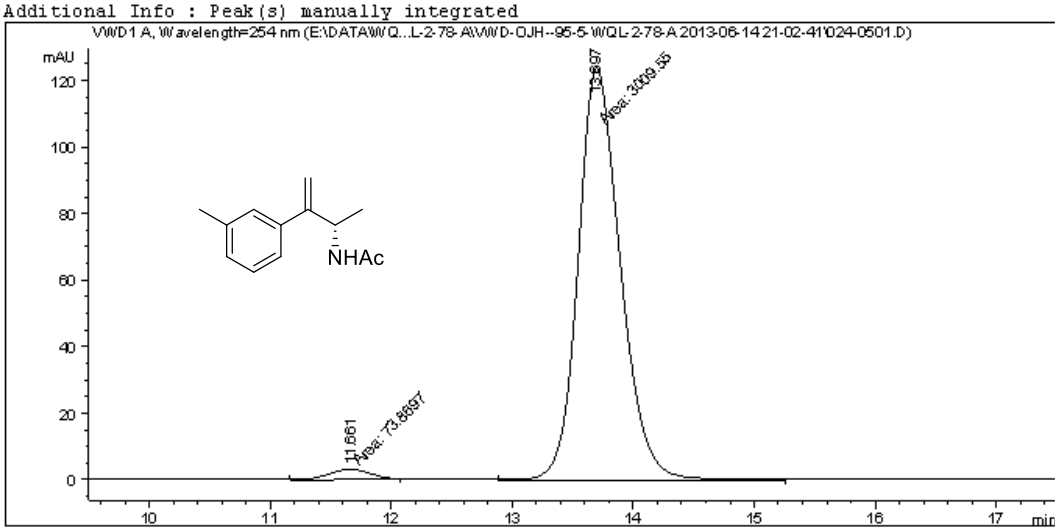
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  11.539 BV    0.3371 3901.96606  179.13626  51.8904
       2  13.677 VB    0.3849 3617.66992  143.12558  48.1096

    Totals :                  7519.63599  322.26184


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

Data File E:\DATA\WQL\WQL-2-78-A\VWD-OJH--95-5-WQL-2-78-A 2013-06-14 21-02-41\024-0501.D
Sample Name: WQL-2-78-6

```
==================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   5
Acq. Instrument : 1260HPLC-VWD                   Location : Vial 24
Injection Date  : 6/14/2013 11:16:25 PM              Inj :   1
                                                Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\WQL\WQL-2-78-A\VWD-OJH--95-5-WQL-2-78-A 2013-06-14 21-02-41\VWD-
                  OJH-95-5-1ML-254NM-40MIN(1-2).M
Last changed    : 6/14/2013 9:02:42 PM by SYSTEM
Analysis Method : E:\DATA\WQL\WQL-2-78-A\VWD-OJH--95-5-WQL-2-78-A 2013-06-14 21-02-41\VWD-
                  OJH-95-5-1ML-254NM-40MIN(1-2).M (Sequence Method)
Last changed    : 4/29/2015 3:43:42 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=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  11.661 MM    0.4038   73.86974    3.04892   2.3957
   2  13.697 MM    0.4052 3009.54663  123.80196  97.6043

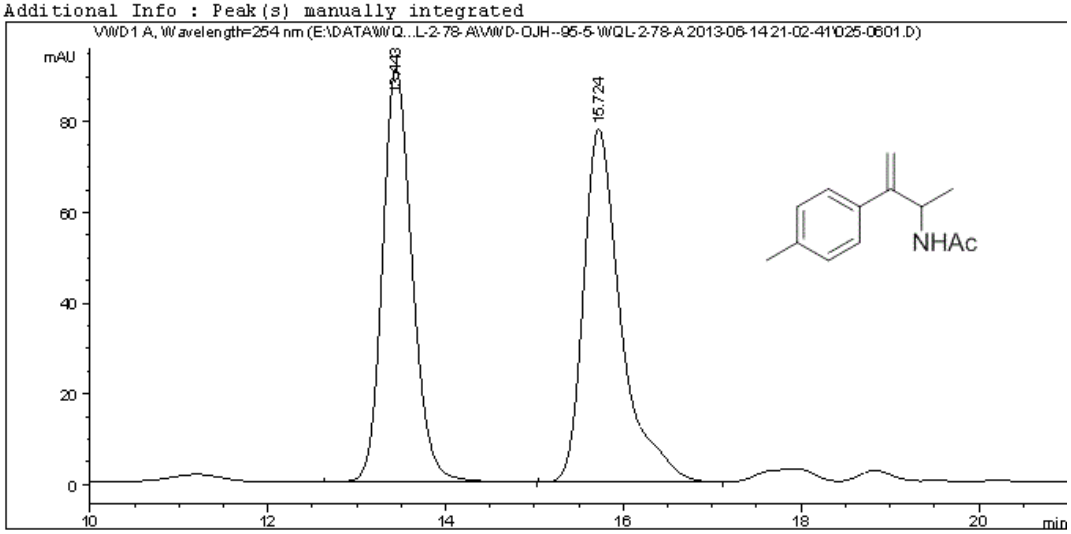
Totals :                  3083.41637  126.85088


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


2c - HPLC

Data File E:\DATA\WQL\WQL-2-78-A\VWD-OJH--95-5-WQL-2-78-A 2013-06-14 21-02-41\025-0601.D
Sample Name: WQL-2-78-4

```
    ====================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   6
    Acq. Instrument : 1260HPLC-VWD                   Location : Vial 25
    Injection Date  : 6/14/2013 11:57:13 PM              Inj :   1
                                                   Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\WQL\WQL-2-78-A\VWD-OJH--95-5-WQL-2-78-A 2013-06-14 21-02-41\VWD-
                      OJH-95-5-1ML-254NM-40MIN(1-2).M
    Last changed    : 6/14/2013 9:02:42 PM by SYSTEM
    Analysis Method : E:\DATA\WQL\WQL-2-78-A\VWD-OJH--95-5-WQL-2-78-A 2013-06-14 21-02-41\VWD-
                      OJH-95-5-1ML-254NM-40MIN(1-2).M (Sequence Method)
    Last changed    : 4/29/2015 3:50:38 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

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


    Signal 1: VWD1 A, Wavelength=254 nm

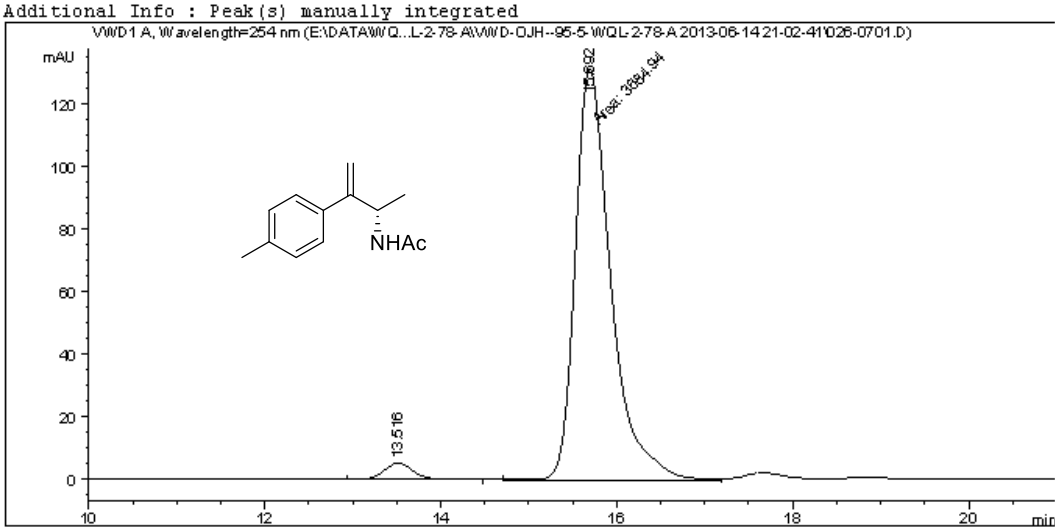
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  13.443 BB    0.3566 2116.04980   91.23906  48.1864
       2  15.724 BB    0.4393 2275.33154   77.78905  51.8136

    Totals :                  4391.38135  169.02811

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

Data File E:\DATA\WQL\WQL-2-78-A\VWD-OJH--95-5-WQL-2-78-A 2013-06-14 21-02-41\026-0701.D
Sample Name: WQL-2-78-7

```
==================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   7
Acq. Instrument : 1260HPLC-VWD                   Location : Vial 26
Injection Date  : 6/15/2013 12:37:57 AM               Inj :   1
                                                Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\WQL\WQL-2-78-A\VWD-OJH--95-5-WQL-2-78-A 2013-06-14 21-02-41\VWD-
                  OJH-95-5-1ML-254NM-40MIN(1-2).M
Last changed    : 6/14/2013 9:02:42 PM by SYSTEM
Analysis Method : E:\DATA\WQL\WQL-2-78-A\VWD-OJH--95-5-WQL-2-78-A 2013-06-14 21-02-41\VWD-
                  OJH-95-5-1ML-254NM-40MIN(1-2).M (Sequence Method)
Last changed    : 4/29/2015 3:50:38 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




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

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


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  13.516 BB    0.3529  119.95254    5.24414   3.1526
   2  15.692 MM    0.4668 3684.94287  131.57304  96.8474

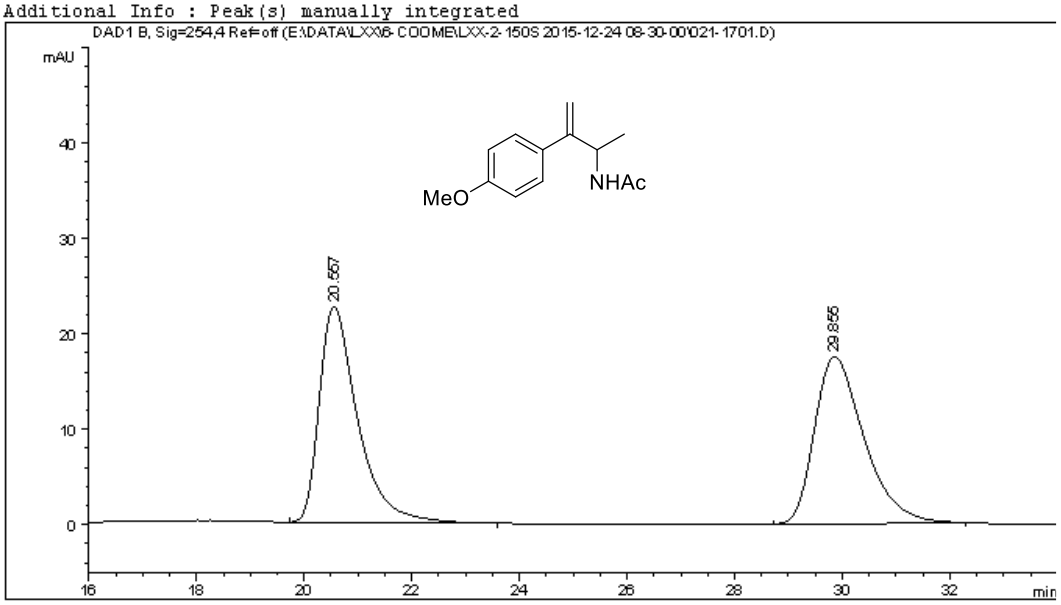
Totals :                  3804.89541  136.81718


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


2d - HPLC

Data File E:\DATA\LXX\6-COOME\LXX-2-150S 2015-12-24 08-30-00\021-1701.D
Sample Name: gwc-wai

```
    ==================================================================
    Acq. Operator   : SYSTEM                          Seq. Line :  17
    Acq. Instrument : 1260HPLC-DAD                     Location : Vial 21
    Injection Date  : 12/24/2015 5:53:22 PM                 Inj :  1
                                                      Inj Volume : 1.000 µl
    Acq. Method     : E:\DATA\LXX\6-COOME\LXX-2-150S 2015-12-24 08-30-00\DAD-OD(1-6)-95-5-1.
                      OML-ALL-40MIN.M
    Last changed    : 12/24/2015 12:26:54 PM by SYSTEM
    Analysis Method : E:\DATA\LXX\6-COOME\LXX-2-150S 2015-12-24 08-30-00\DAD-OD(1-6)-95-5-1.
                      OML-ALL-40MIN.M (Sequence Method)
    Last changed    : 2/23/2016 9:31:52 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=254,4 Ref=off

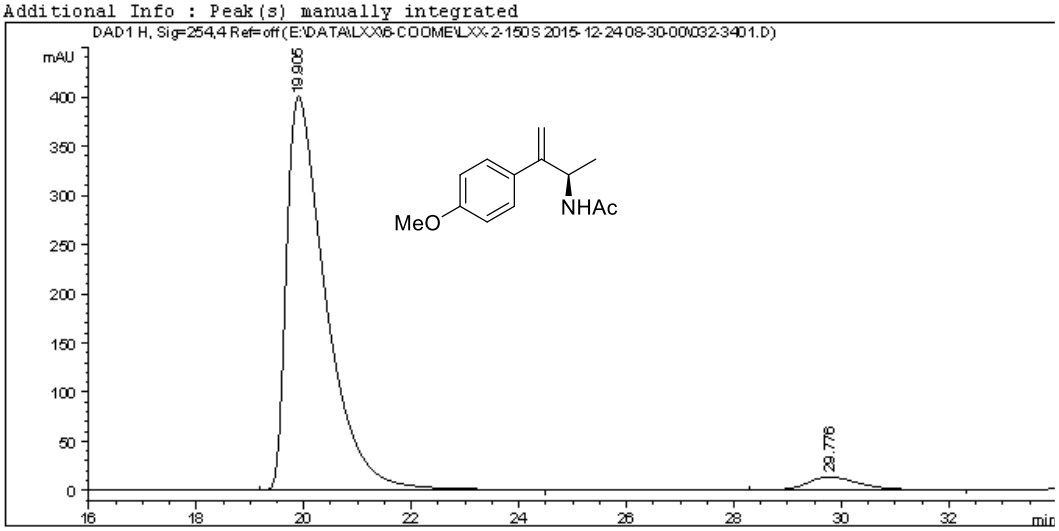
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]        %
    ----|-------|----|-------|----------|----------|--------|
       1  20.557 BB    0.7262 1122.28625   22.64534  49.9401
       2  29.855 BB    0.9046 1124.98010   17.53318  50.0599

    Totals :                  2247.26636   40.17852


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

Data File E:\DATA\LXX\6-COOME\LXX-2-150S 2015-12-24 08-30-00\032-3401.D
Sample Name: gwc-16-1-2

```
    ==================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :  34
    Acq. Instrument : 1260HPLC-DAD                   Location : Vial 32
    Injection Date  : 12/25/2015 3:31:46 AM               Inj :  1
                                                  Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\LXX\6-COOME\LXX-2-150S 2015-12-24 08-30-00\DAD-ODH-95-5-1ML-220-
                      254NM-40MIN(1-2).M
    Last changed    : 12/24/2015 7:53:30 PM by SYSTEM
    Analysis Method : E:\DATA\LXX\6-COOME\LXX-2-150S 2015-12-24 08-30-00\DAD-ODH-95-5-1ML-220-
                      254NM-40MIN(1-2).M (Sequence Method)
    Last changed    : 2/23/2016 9:34:57 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

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


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

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  19.905 BB    0.7508 2.02663e4   400.18912  95.7518
       2  29.776 BB    0.8075  899.15448   13.38812   4.2482

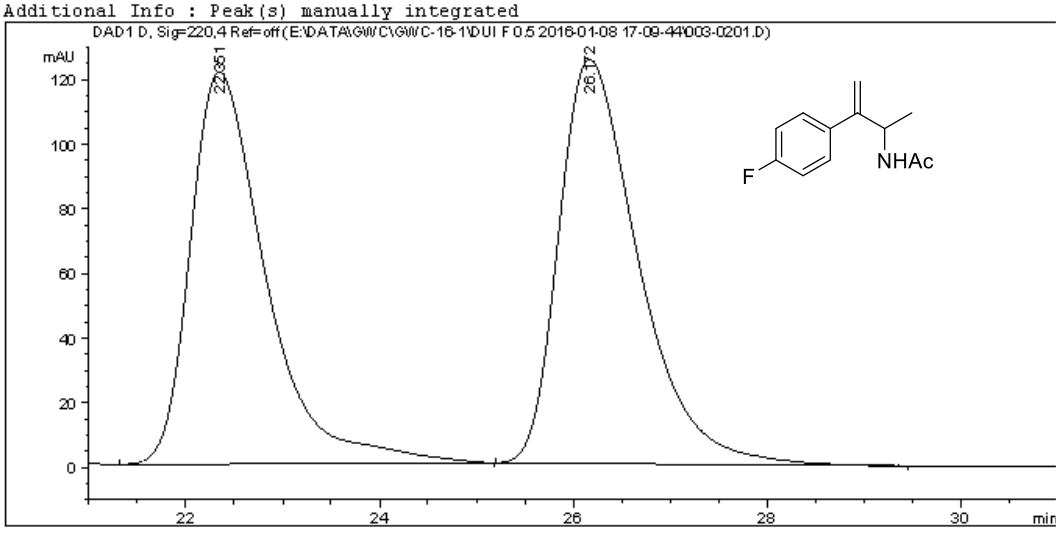
    Totals :                  2.11655e4   413.57723


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

2e - HPLC

Data File E:\DATA\GWC\GWC-16-1\DUI F 0.5 2016-01-08 17-09-44\003-0201.D
Sample Name: 16-1-3

```
    ==================================================================
    Acq. Operator   : SYSTEM                       Seq. Line :   2
    Acq. Instrument : 1260HPLC-DAD                  Location : Vial 3
    Injection Date  : 1/8/2016 5:21:36 PM                Inj :   1
                                                  Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\GWC\GWC-16-1\DUI F 0.5 2016-01-08 17-09-44\DAD-OD(1-2)-95-5-0.
                      5ML-220NM-254NM-50MIN.M
    Last changed    : 1/8/2016 5:48:01 PM by SYSTEM
                      (modified after loading)
    Analysis Method : E:\DATA\GWC\GWC-16-1\DUI F 0.5 2016-01-08 17-09-44\DAD-OD(1-2)-95-5-0.
                      5ML-220NM-254NM-50MIN.M (Sequence Method)
    Last changed    : 2/22/2016 10:33:13 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 D, Sig=220,4 Ref=off

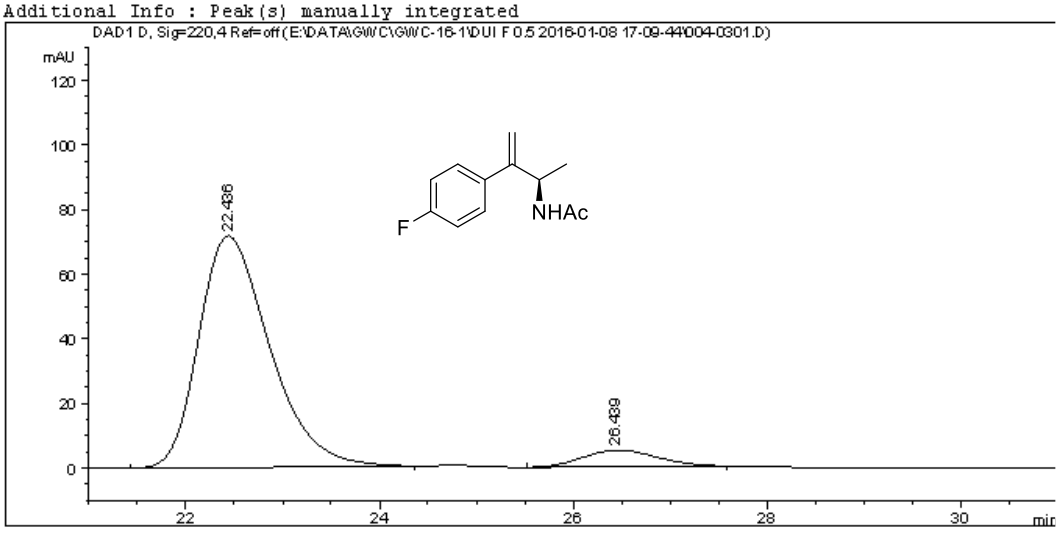
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  22.351 BB    0.8191 6582.78223  121.29909  47.9349
       2  26.172 BB    0.8550 7149.95996  125.06830  52.0651

    Totals :                  1.37327e4   246.36739


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

Data File E:\DATA\GWC\GWC-16-1\DUI F 0.5 2016-01-08 17-09-44\004-0301.D
Sample Name: 16--4

```
    ======================================================================
    Acq. Operator   : SYSTEM                          Seq. Line :   3
    Acq. Instrument : 1260HPLC-DAD                     Location : Vial 4
    Injection Date  : 1/8/2016 5:57:30 PM                   Inj :   1
                                                    Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\GWC\GWC-16-1\DUI F 0.5 2016-01-08 17-09-44\DAD-0D(1-2)-95-5-0.
                      5ML-220NM-254NM-50MIN.M
    Last changed    : 1/8/2016 5:48:01 PM by SYSTEM
    Analysis Method : E:\DATA\GWC\GWC-16-1\DUI F 0.5 2016-01-08 17-09-44\DAD-0D(1-2)-95-5-0.
                      5ML-220NM-254NM-50MIN.M (Sequence Method)
    Last changed    : 2/22/2016 10:36:02 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 D, Sig=220,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  22.436 BB    0.7599 3649.48804   71.69084  93.0517
       2  26.439 BB    0.6259  272.51062    5.12842   6.9483

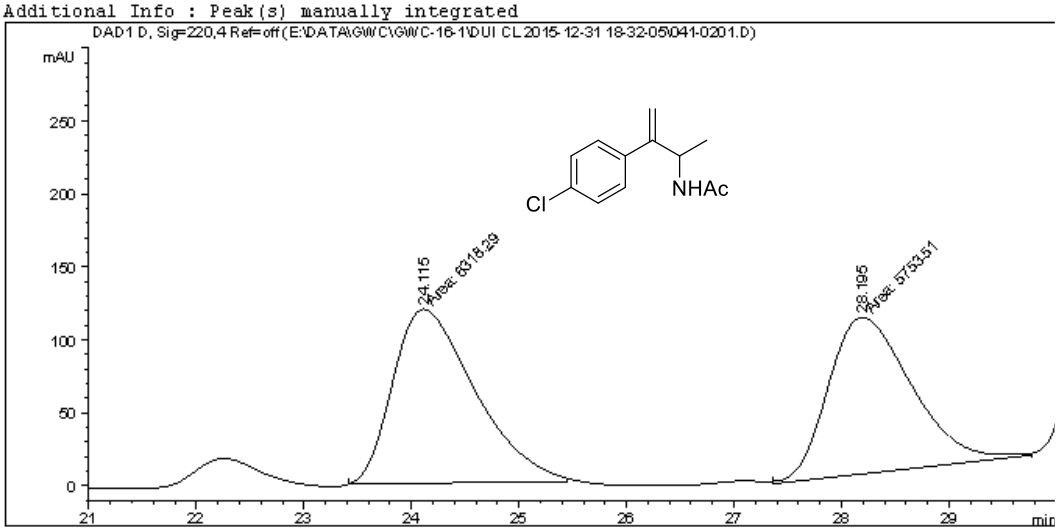
    Totals :                  3921.99866   76.81926


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

2f - HPLC

```
Data File E:\DATA\GWC\GWC-16-1\DUI CL 2015-12-31 18-32-05\041-0201.D
Sample Name: gwc-16-1-5

    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   2
    Acq. Instrument : 1260HPLC-DAD                   Location : Vial 41
    Injection Date  : 12/31/2015 6:43:57 PM               Inj :   1
                                                   Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\GWC\GWC-16-1\DUI CL 2015-12-31 18-32-05\DAD-OD(1-2)-95-5-0.5ML-
                      220NM-254NM-50MIN.M
    Last changed    : 12/31/2015 6:32:05 PM by SYSTEM
    Analysis Method : E:\DATA\GWC\GWC-16-1\DUI CL 2015-12-31 18-32-05\DAD-OD(1-2)-95-5-0.5ML-
                      220NM-254NM-50MIN.M (Sequence Method)
    Last changed    : 2/22/2016 10:48:00 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: DAD1 D, Sig=220,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  24.115 MM    0.8840 6318.28906  119.12337  52.3393
       2  28.195 MM    0.8933 5753.50732  107.34454  47.6607

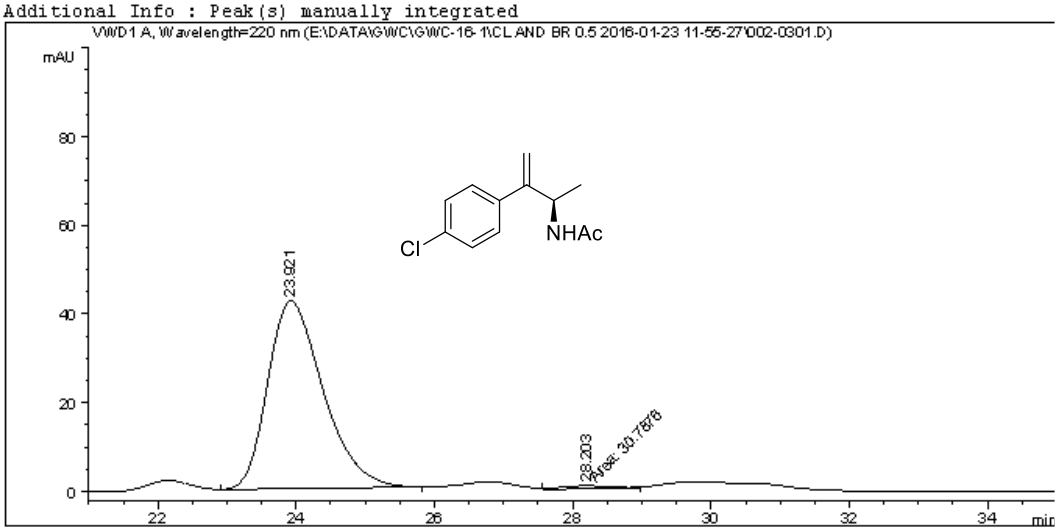
    Totals :                  1.20718e4   226.46790


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


1260HPLC-DAD 2/22/2016 10:48:56 AM SYSTEM                    Page 1 of 1
```

Data File E:\DATA\GWC\GWC-16-1\CL AND BR 0.5 2016-01-23 11-55-27\002-0301.D
Sample Name: Cl cui

```
    ======================================================================
    Acq. Operator   : SYSTEM                          Seq. Line :   3
    Acq. Instrument : 1260HPLC-VWD                     Location : Vial 2
    Injection Date  : 1/23/2016 12:47:46 PM                 Inj :   1
                                                     Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\GWC\GWC-16-1\CL AND BR 0.5 2016-01-23 11-55-27\VWD-ODH-95-5-
                      220NM--0.5-40MIN(1-2).M
    Last changed    : 1/23/2016 11:55:27 AM by SYSTEM
    Analysis Method : E:\DATA\GWC\GWC-16-1\CL AND BR 0.5 2016-01-23 11-55-27\VWD-ODH-95-5-
                      220NM--0.5-40MIN(1-2).M (Sequence Method)
    Last changed    : 2/22/2016 10:43:51 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=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  23.921 BB    0.8510 2356.60718   42.29798  98.7104
       2  28.203 MM    0.7995   30.78764 6.41784e-1   1.2896

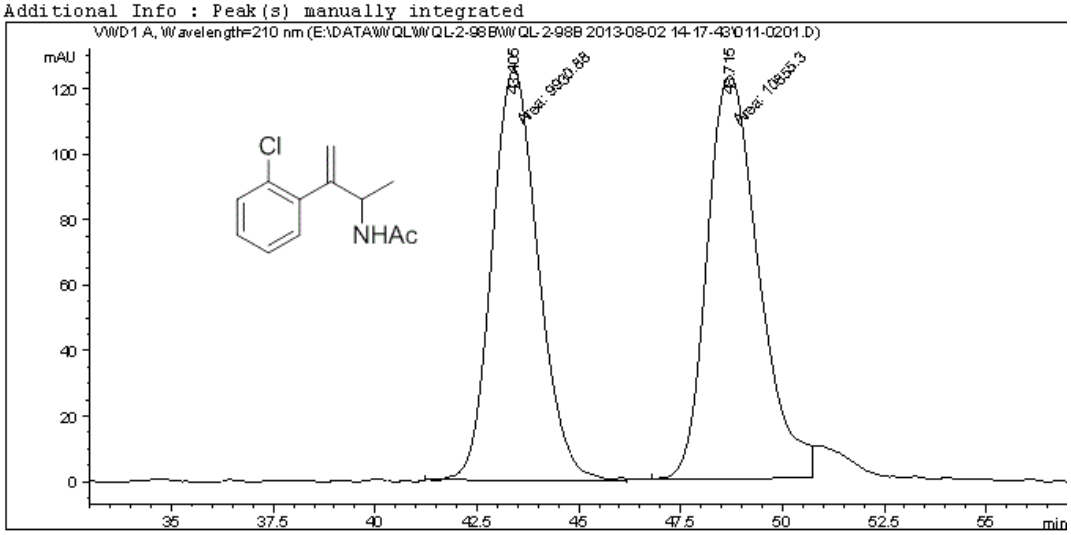
    Totals :                  2387.39482   42.93977


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

2g - HPLC

Data File E:\DATA\WQL\WQL-2-98B\WQL-2-98B 2013-08-02 14-17-43\011-0201.D
Sample Name: wql-2-98-1

```
    ==================================================================
    Acq. Operator   : SYSTEM                          Seq. Line :   2
    Acq. Instrument : 1260HPLC-VWD                     Location : Vial 11
    Injection Date  : 8/2/2013 2:29:16 PM                   Inj :   1
                                                    Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\WQL\WQL-2-98B\WQL-2-98B 2013-08-02 14-17-43\VWD-0JH-99-1-1ML-
                      210NM-60MIN(1-2).M
    Last changed    : 8/2/2013 2:17:43 PM by SYSTEM
    Analysis Method : E:\DATA\WQL\WQL-2-98B\WQL-2-98B 2013-08-02 14-17-43\VWD-0JH-99-1-1ML-
                      210NM-60MIN(1-2).M (Sequence Method)
    Last changed    : 4/29/2015 3:17:27 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: VWD1 A, Wavelength=210 nm

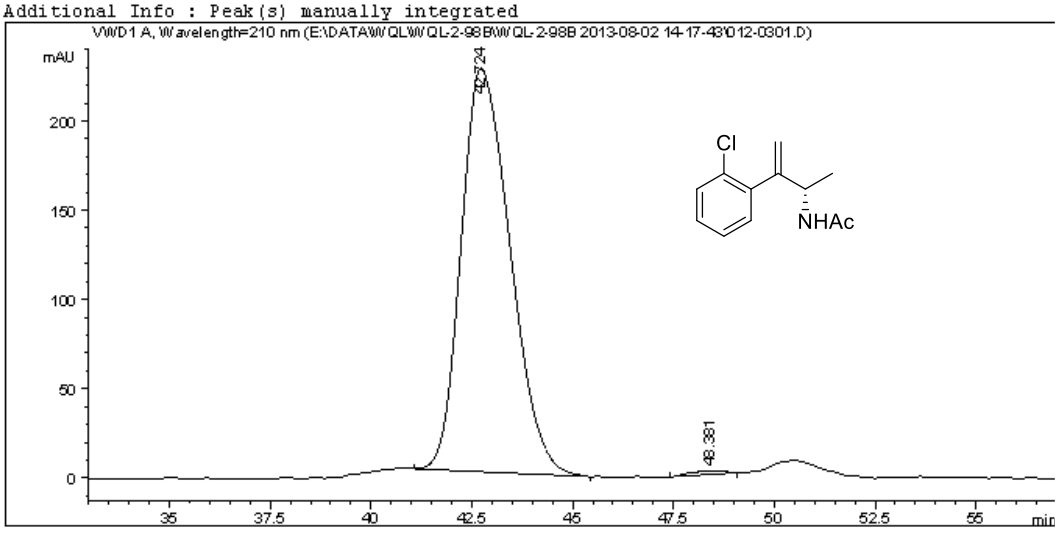
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  43.405 MM    1.3162 9930.87695  125.75005  47.7763
       2  48.715 MF    1.4712 1.08553e4   122.97532  52.2237

    Totals :                  2.07862e4   248.72537


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

Data File E:\DATA\WQL\WQL-2-98B\WQL-2-98B 2013-08-02 14-17-43\012-0301.D
Sample Name: wql-2-98-2

```
    ======================================================================
    Acq. Operator   : SYSTEM                          Seq. Line :   3
    Acq. Instrument : 1260HPLC-VWD                     Location : Vial 12
    Injection Date  : 8/2/2013 3:30:04 PM                  Inj :   1
                                                     Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\WQL\WQL-2-98B\WQL-2-98B 2013-08-02 14-17-43\VWD-0JH-99-1-1ML-
                      210NM-60MIN(1-2).M
    Last changed    : 8/2/2013 2:17:43 PM by SYSTEM
    Analysis Method : E:\DATA\WQL\WQL-2-98B\WQL-2-98B 2013-08-02 14-17-43\VWD-0JH-99-1-1ML-
                      210NM-60MIN(1-2).M (Sequence Method)
    Last changed    : 4/29/2015 3:17:27 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: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  42.724 BB    1.3405 1.9097le4   226.07219  99.3688
       2  48.381 BB    0.7205  121.31303    2.15591   0.6312

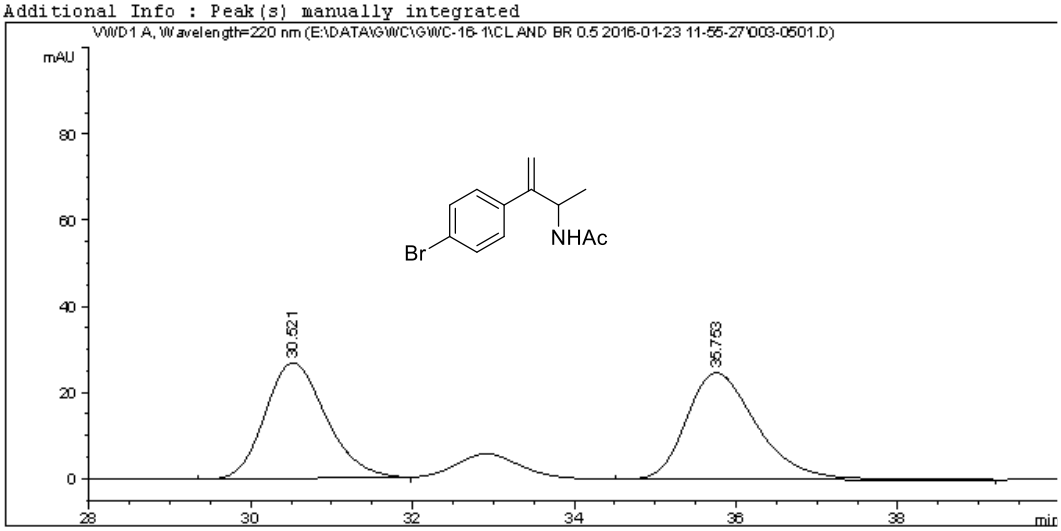
    Totals :                  1.92184e4   228.22810

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

2h - HPLC

Data File E:\DATA\GWC\GWC-16-1\CL AND BR 0.5 2016-01-23 11-55-27\003-0501.D
Sample Name: Br wai

```
    ======================================================================
    Acq. Operator   : SYSTEM                       Seq. Line :   5
    Acq. Instrument : 1260HPLC-VWD                  Location : Vial 3
    Injection Date  : 1/23/2016 1:39:25 PM               Inj :   1
                                                  Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\GWC\GWC-16-1\CL AND BR 0.5 2016-01-23 11-55-27\VWD-ADH(1-6)-95-5
                      -220NM--0.5-40MIN.M
    Last changed    : 1/23/2016 11:55:27 AM by SYSTEM
    Analysis Method : E:\DATA\GWC\GWC-16-1\CL AND BR 0.5 2016-01-23 11-55-27\VWD-ADH(1-6)-95-5
                      -220NM--0.5-40MIN.M (Sequence Method)
    Last changed    : 2/22/2016 10:38:39 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=220 nm

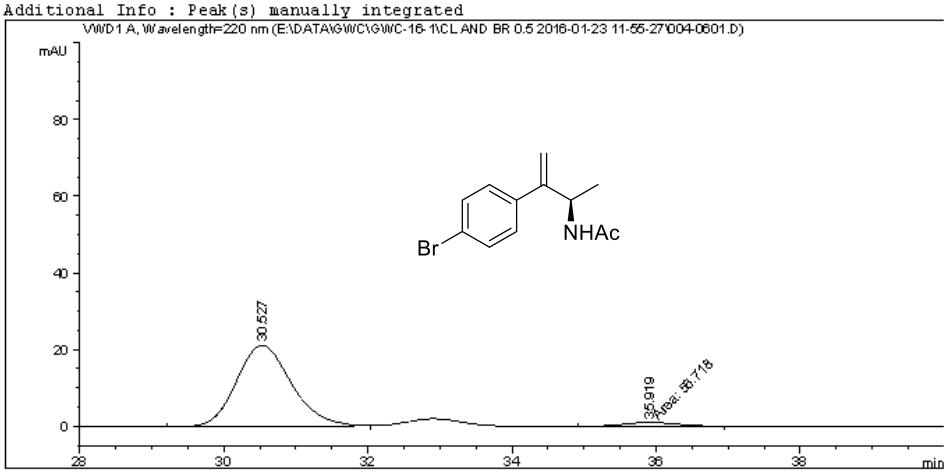
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]       [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  30.521 BB    0.8054 1401.24109   26.82582  48.2459
       2  35.753 BB    0.9296 1503.12976   24.78430  51.7541

    Totals :                  2904.37085   51.61012


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

Data File E:\DATA\GWC\GWC-16-1\CL AND BR 0.5 2016-01-23 11-55-27\004-0601.D
Sample Name: Br cui

```
    ==================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   6
    Acq. Instrument : 1260HPLC-VWD                   Location : Vial 4
    Injection Date  : 1/23/2016 2:20:10 PM               Inj :   1
                                                   Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\GWC\GWC-16-1\CL AND BR 0.5 2016-01-23 11-55-27\VWD-ADH(1-6)-95-5
                      -220NM--0.5-40MIN.M
    Last changed    : 1/23/2016 11:55:27 AM by SYSTEM
    Analysis Method : E:\DATA\GWC\GWC-16-1\CL AND BR 0.5 2016-01-23 11-55-27\VWD-ADH(1-6)-95-5
                      -220NM--0.5-40MIN.M (Sequence Method)
    Last changed    : 2/22/2016 10:40:23 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: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  30.527 BB    0.8207 1114.62036   21.05220  95.1578
       2  35.919 MM    0.9285   56.71799    1.01808   4.8422

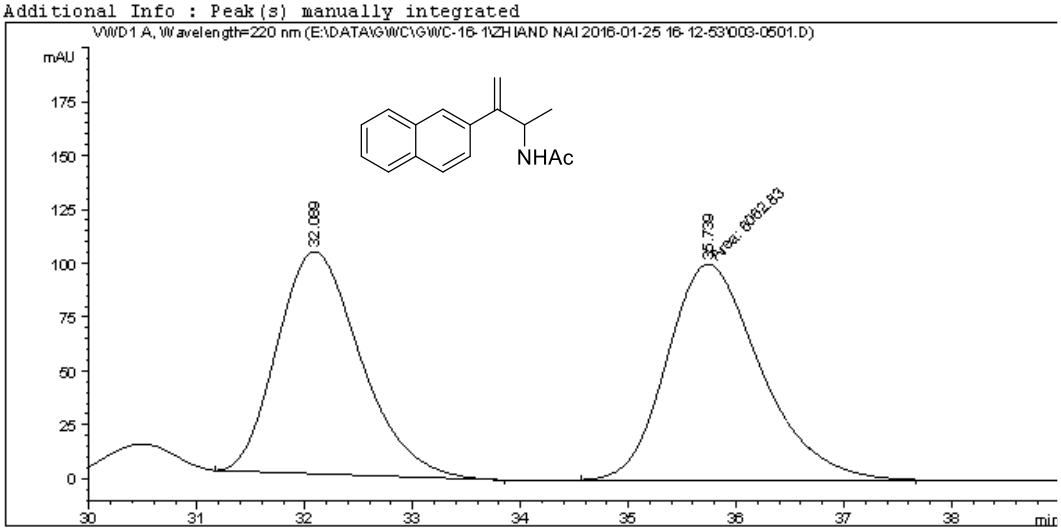
    Totals :                  1171.33835   22.07028


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

2i - HPLC

Data File E:\DATA\GWC\GWC-16-1\ZHIAND NAI 2016-01-25 16-12-53\003-0501.D
Sample Name: NAI WAI

```
    ======================================================================
    Acq. Operator   : SYSTEM                       Seq. Line :   5
    Acq. Instrument : 1260HPLC-VWD                  Location : Vial 3
    Injection Date  : 1/25/2016 6:59:23 PM               Inj :   1
                                                  Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\GWC\GWC-16-1\ZHIAND NAI 2016-01-25 16-12-53\VWD-ADH(1-6)-95-5-
                      220NM--0.5-40MIN.M
    Last changed    : 1/25/2016 4:12:54 PM by SYSTEM
    Analysis Method : E:\DATA\GWC\GWC-16-1\ZHIAND NAI 2016-01-25 16-12-53\VWD-ADH(1-6)-95-5-
                      220NM--0.5-40MIN.M (Sequence Method)
    Last changed    : 2/22/2016 10:54:38 AM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

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


    Signal 1: VWD1 A, Wavelength=220 nm

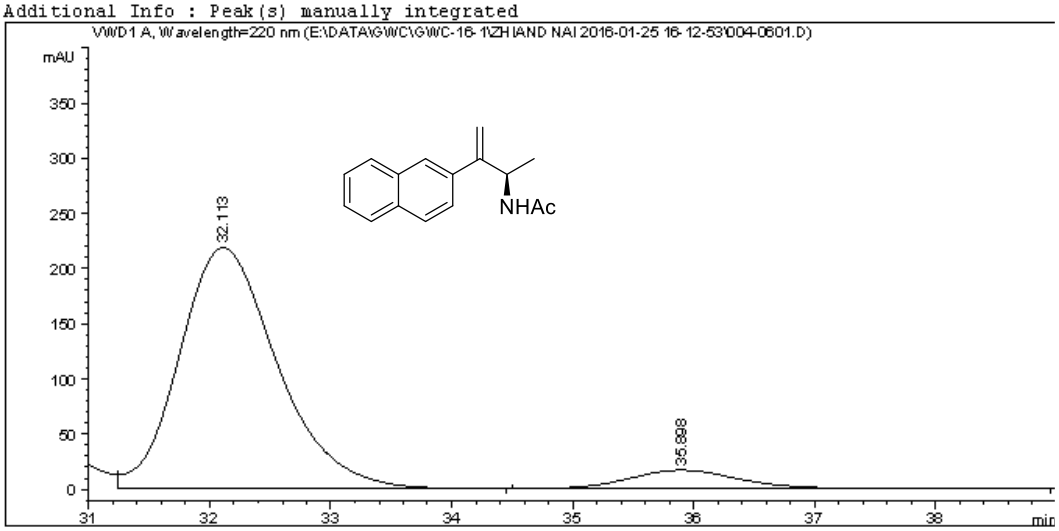
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]       [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  32.089 BB    0.8365 5530.81592  102.99131  47.7056
       2  35.739 MM    1.0085 6062.83252  100.19076  52.2944

    Totals :                  1.15936e4   203.18207


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

Data File E:\DATA\GWC\GWC-16-1\ZHIAND NAI 2016-01-25 16-12-53\004-0601.D
Sample Name: NAI CUI

```
    ======================================================================
    Acq. Operator   : SYSTEM                          Seq. Line :   6
    Acq. Instrument : 1260HPLC-VWD                     Location : Vial 4
    Injection Date  : 1/25/2016 7:40:08 PM                  Inj :   1
                                                    Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\GWC\GWC-16-1\ZHIAND NAI 2016-01-25 16-12-53\VWD-ADH(1-6)-95-5-
                      220NM--0.5-40MIN.M
    Last changed    : 1/25/2016 4:12:54 PM by SYSTEM
    Analysis Method : E:\DATA\GWC\GWC-16-1\ZHIAND NAI 2016-01-25 16-12-53\VWD-ADH(1-6)-95-5-
                      220NM--0.5-40MIN.M (Sequence Method)
    Last changed    : 2/22/2016 10:55:47 AM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

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


    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  32.113 VB    0.8641 1.22601e4   218.34422  91.8003
       2  35.898 BB    0.9762 1095.08386   17.03093   8.1997

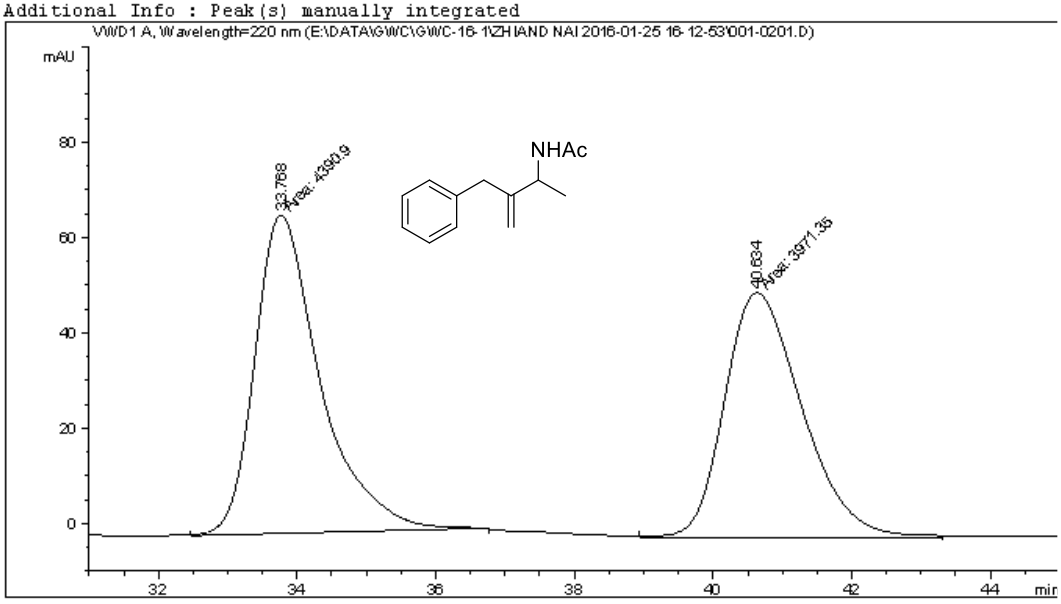
    Totals :                  1.33552e4   235.37515


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

2j - HPLC

Data File E:\DATA\GWC\GWC-16-1\ZHIAND NAI 2016-01-25 16-12-53\001-0201.D
Sample Name: ZHI WAI

```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   2
    Acq. Instrument : 1260HPLC-VWD                   Location : Vial 1
    Injection Date  : 1/25/2016 4:24:27 PM                Inj :   1
                                                   Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\GWC\GWC-16-1\ZHIAND NAI 2016-01-25 16-12-53\VWD-0J(1-2)-95-5-0.
                      5ML-60MIN-220NM.M
    Last changed    : 1/25/2016 5:15:29 PM by SYSTEM
                      (modified after loading)
    Analysis Method : E:\DATA\GWC\GWC-16-1\ZHIAND NAI 2016-01-25 16-12-53\VWD-0J(1-2)-95-5-0.
                      5ML-60MIN-220NM.M (Sequence Method)
    Last changed    : 2/23/2016 9:03:26 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=220 nm

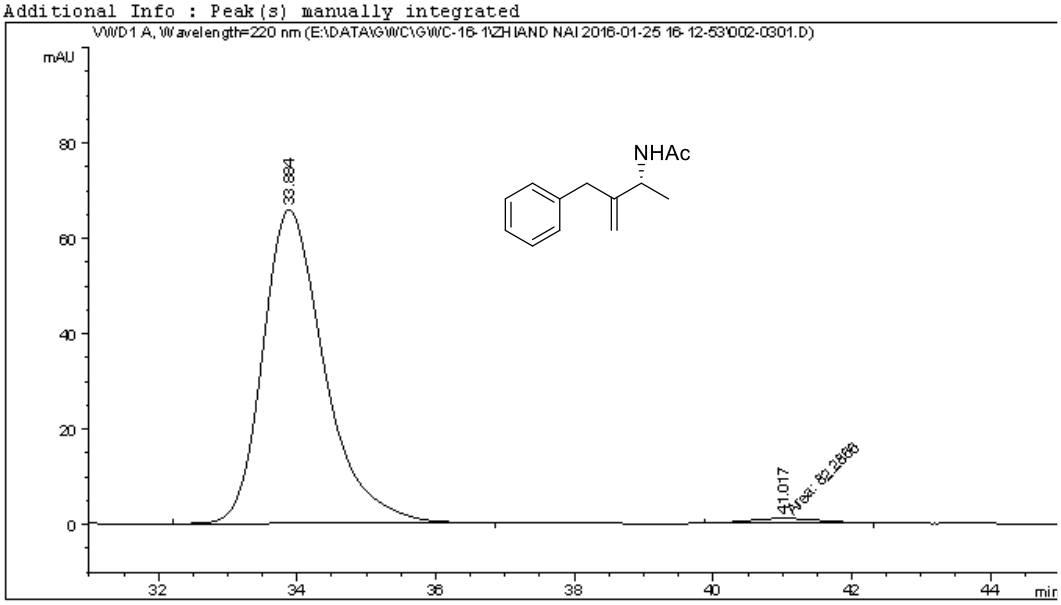
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  33.768 MM    1.0958 4390.90186   66.78225  52.5086
       2  40.634 MM    1.2913 3971.35083   51.25767  47.4914

    Totals :                  8362.25269  118.03992

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

Data File E:\DATA\GWC\GWC-16-1\ZHIAND NAI 2016-01-25 16-12-53\002-0301.D
Sample Name: ZHI CUI

```
    ======================================================================
    Acq. Operator   : SYSTEM                          Seq. Line :   3
    Acq. Instrument : 1260HPLC-VWD                     Location : Vial 2
    Injection Date  : 1/25/2016 5:35:12 PM                  Inj :   1
                                                    Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\GWC\GWC-16-1\ZHIAND NAI 2016-01-25 16-12-53\VWD-0J(1-2)-95-5-0.
                      5ML-60MIN-220NM.M
    Last changed    : 1/25/2016 5:15:29 PM by SYSTEM
    Analysis Method : E:\DATA\GWC\GWC-16-1\ZHIAND NAI 2016-01-25 16-12-53\VWD-0J(1-2)-95-5-0.
                      5ML-60MIN-220NM.M (Sequence Method)
    Last changed    : 2/23/2016 9:04:09 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=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]        %
    ----|-------|----|-------|----------|----------|--------|
       1  33.884 BB    0.9414 4054.80493   65.84998  98.0110
       2  41.017 MM    1.2843   82.28664    1.06783   1.9890

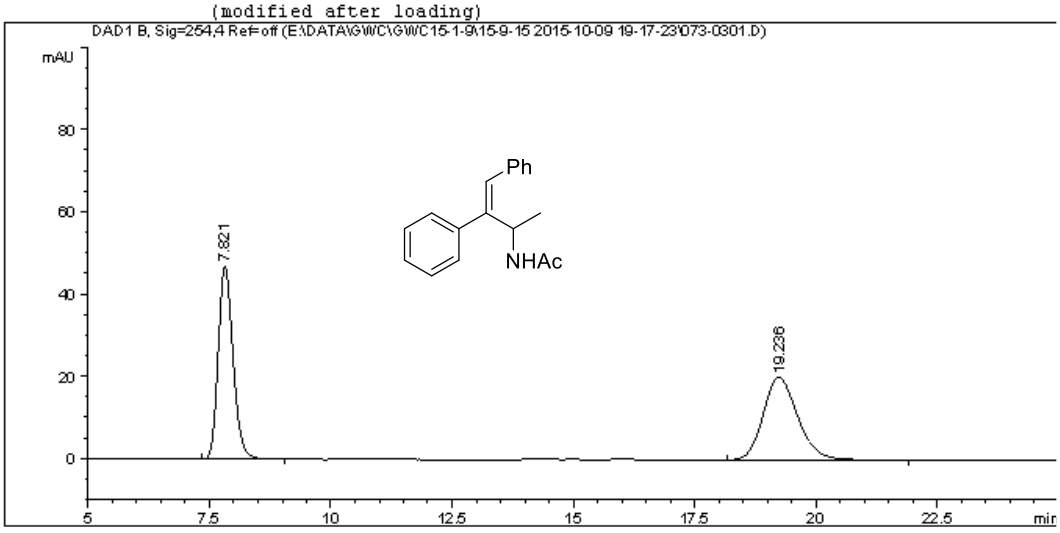
    Totals :                  4137.09158   66.91781


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

2k - HPLC

Data File E:\DATA\GWC\GWC15-1-9\15-9-15 2015-10-09 19-17-23\073-0301.D
Sample Name: wai

```
==================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   3
Acq. Instrument : 1260HPLC-DAD                   Location : Vial 73
Injection Date  : 10/9/2015 7:55:09 PM                Inj :   1
                                                Inj Volume : 5.000 µl
Acq. Method     : E:\DATA\GWC\GWC15-1-9\15-9-15 2015-10-09 19-17-23\DAD-ODH-90-10-1ML-
                  40MIN(1-2).M
Last changed    : 10/9/2015 7:52:27 PM by SYSTEM
Analysis Method : E:\DATA\GWC\GWC15-1-9\15-9-15 2015-10-09 19-17-23\DAD-ODH-90-10-1ML-
                  40MIN(1-2).M (Sequence Method)
Last changed    : 2/23/2016 7:42:31 PM by SYSTEM
                  (modified after loading)
```




```
==================================================================
                        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=254,4 Ref=off

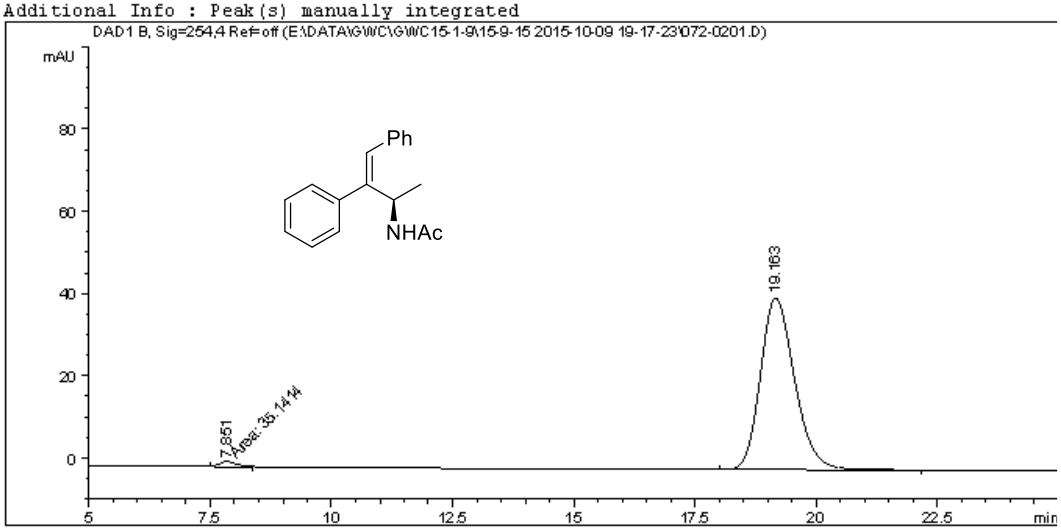
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1   7.821 BB    0.3256  995.52374   46.90849  49.9797
   2  19.236 BB    0.7527  996.33215   20.01796  50.0203

Totals :                  1991.85590   66.92645

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

Data File E:\DATA\GWC\GWC15-1-9\15-9-15 2015-10-09 19-17-23\072-0201.D
Sample Name: 15-9-15

```
    ======================================================================
    Acq. Operator   : SYSTEM                          Seq. Line :   2
    Acq. Instrument : 1260HPLC-DAD                     Location : Vial 72
    Injection Date  : 10/9/2015 7:29:15 PM                  Inj :   1
                                                    Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\GWC\GWC15-1-9\15-9-15 2015-10-09 19-17-23\DAD-0DH-90-10-1ML-
                      40MIN(1-2).M
    Last changed    : 10/9/2015 7:52:27 PM by SYSTEM
                      (modified after loading)
    Analysis Method : E:\DATA\GWC\GWC15-1-9\15-9-15 2015-10-09 19-17-23\DAD-0DH-90-10-1ML-
                      40MIN(1-2).M (Sequence Method)
    Last changed    : 2/23/2016 7:43:46 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=254,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   7.851 MM    0.4481   35.14143    1.30715   1.6841
       2  19.163 BB    0.7590 2051.52808   41.77021  98.3159

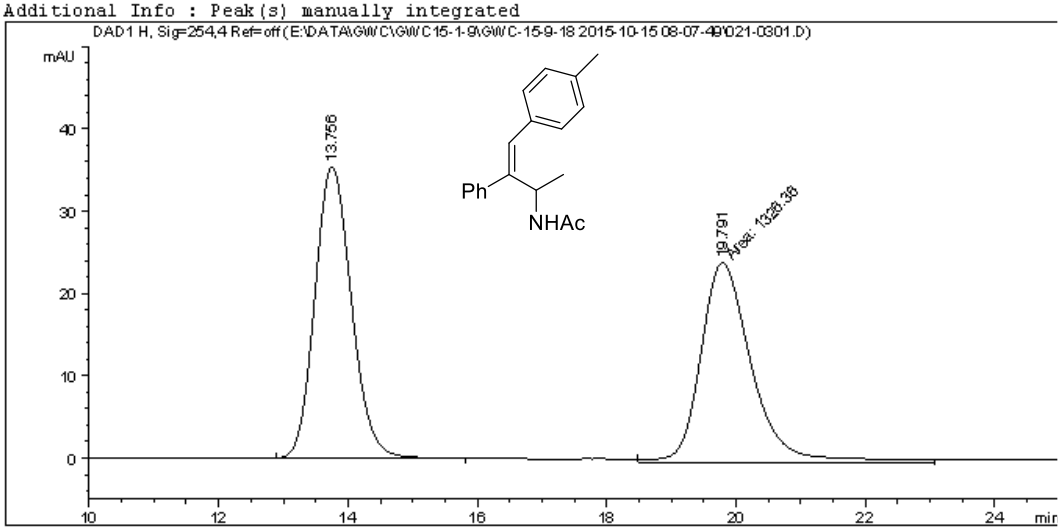
    Totals :                  2086.66951   43.07736

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

2l - HPLC

Data File E:\DATA\GWC\GWC15-1-9\GWC-15-9-18 2015-10-15 08-07-49\021-0301.D
Sample Name: 15-9-19-2

```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   3
    Acq. Instrument : 1260HPLC-DAD                  Location : Vial 21
    Injection Date  : 10/15/2015 8:56:04 AM              Inj :   1
                                                   Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\GWC\GWC15-1-9\GWC-15-9-18 2015-10-15 08-07-49\DAD-ODH-95-5-1ML-
                      220-254NM-40MIN(1-2).M
    Last changed    : 10/15/2015 9:26:39 AM by SYSTEM
                      (modified after loading)
    Analysis Method : E:\DATA\GWC\GWC15-1-9\GWC-15-9-18 2015-10-15 08-07-49\DAD-ODH-95-5-1ML-
                      220-254NM-40MIN(1-2).M (Sequence Method)
    Last changed    : 2/23/2016 7:56:24 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 H, Sig=254,4 Ref=off

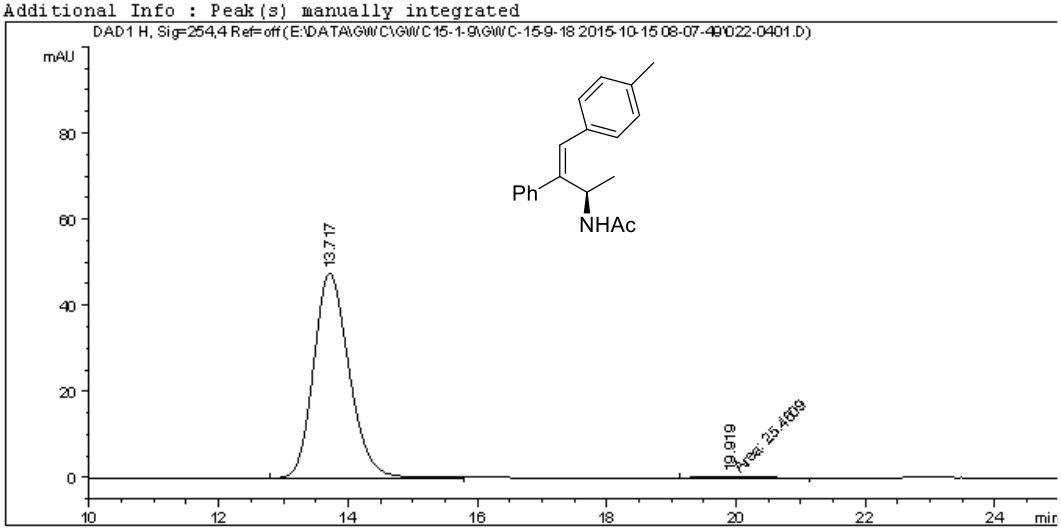
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]        %
    ----|-------|----|-------|----------|----------|--------|
       1  13.756 BB    0.6139 1391.82678   35.45882  51.2043
       2  19.791 MM    0.9116 1326.35547   24.24839  48.7957

    Totals :                  2718.18225   59.70722


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

Data File E:\DATA\GWC\GWC15-1-9\GWC-15-9-18 2015-10-15 08-07-49\022-0401.D
Sample Name: 15-9-18

```
    ====================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   4
    Acq. Instrument : 1260HPLC-DAD                  Location : Vial 22
    Injection Date  : 10/15/2015 9:27:57 AM              Inj :   1
                                                   Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\GWC\GWC15-1-9\GWC-15-9-18 2015-10-15 08-07-49\DAD-ODH-95-5-1ML-
                      220-254NM-40MIN(1-2).M
    Last changed    : 10/15/2015 9:28:31 AM by SYSTEM
                      (modified after loading)
    Analysis Method : E:\DATA\GWC\GWC15-1-9\GWC-15-9-18 2015-10-15 08-07-49\DAD-ODH-95-5-1ML-
                      220-254NM-40MIN(1-2).M (Sequence Method)
    Last changed    : 2/23/2016 7:57:36 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 H, Sig=254,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  13.717 BB    0.5831 1802.31653   47.45339  98.6070
       2  19.919 MM    0.9870   25.46087 4.29932e-1   1.3930

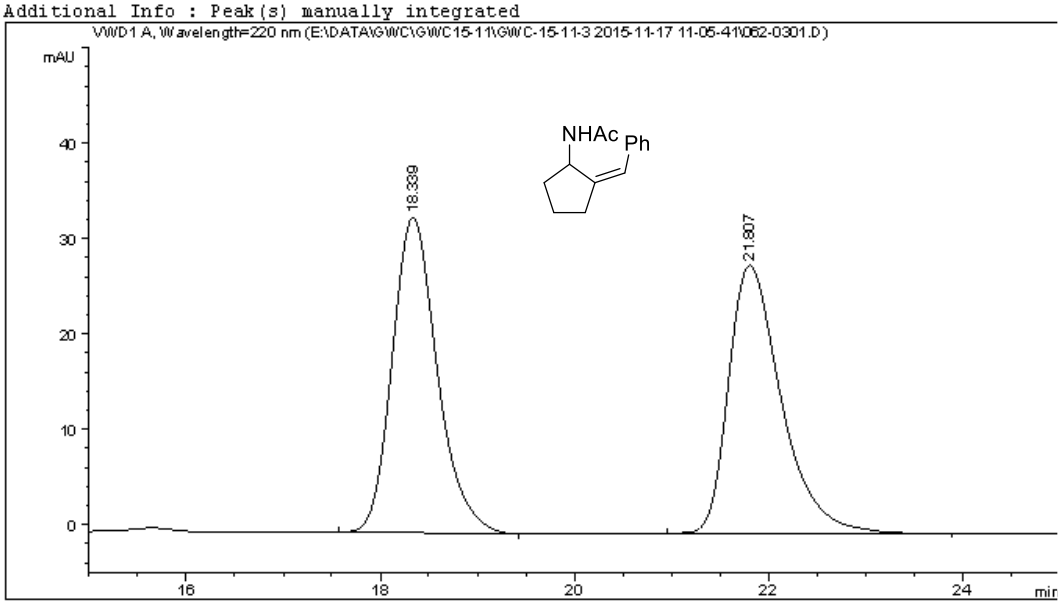
    Totals :                  1827.77740   47.88332

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

2m - HPLC

Data File E:\DATA\GWC\GWC15-11\GWC-15-11-3 2015-11-17 11-05-41\062-0301.D
Sample Name: SLY-3-70-1-J

```
    ======================================================================
    Acq. Operator   : SYSTEM                          Seq. Line :   3
    Acq. Instrument : 1260HPLC-VWD                     Location : Vial 62
    Injection Date  : 11/17/2015 11:48:05 AM               Inj :   1
                                                     Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\GWC\GWC15-11\GWC-15-11-3 2015-11-17 11-05-41\VWD-ADH(1-2)-95-5-
                      220-254NM-40MIN.M
    Last changed    : 11/17/2015 11:42:01 AM by SYSTEM
    Analysis Method : E:\DATA\GWC\GWC15-11\GWC-15-11-3 2015-11-17 11-05-41\VWD-ADH(1-2)-95-5-
                      220-254NM-40MIN.M (Sequence Method)
    Last changed    : 2/23/2016 8:59:45 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=220 nm

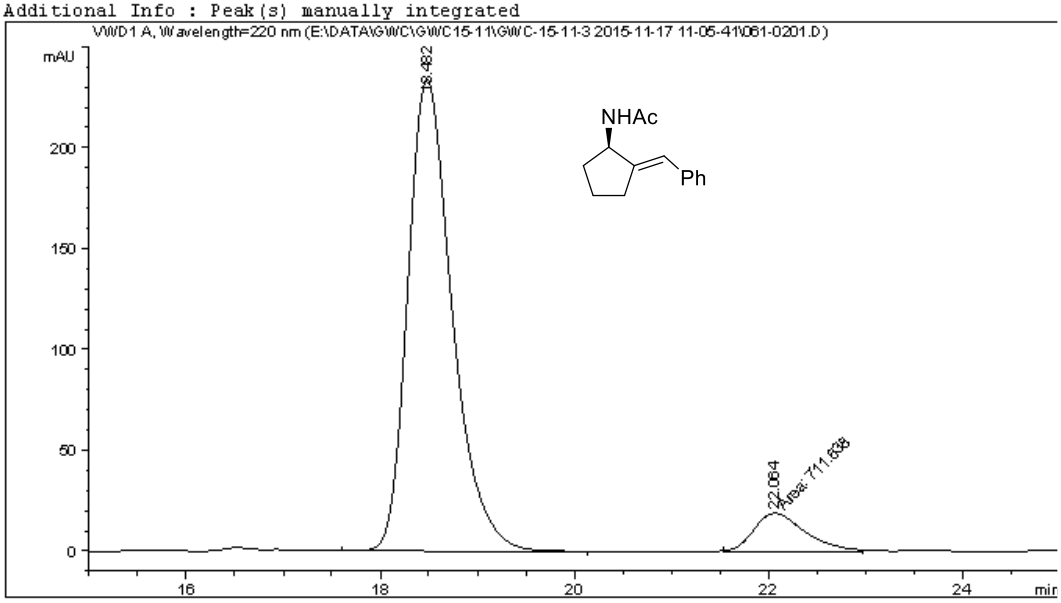
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  18.339 BB    0.4951 1067.27368   33.10320  50.2909
       2  21.807 BB    0.5744 1054.92639   28.07599  49.7091

    Totals :                  2122.20007   61.17919


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

Data File E:\DATA\GWC\GWC15-11\GWC-15-11-3 2015-11-17 11-05-41\061-0201.D
Sample Name: SLY-3-70-3-J

```
    ====================================================================
    Acq. Operator   : SYSTEM                          Seq. Line :   2
    Acq. Instrument : 1260HPLC-VWD                     Location : Vial 61
    Injection Date  : 11/17/2015 11:17:19 AM               Inj :   1
                                                    Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\GWC\GWC15-11\GWC-15-11-3 2015-11-17 11-05-41\VWD-ADH(1-2)-95-5-
                      220-254NM-40MIN.M
    Last changed    : 11/17/2015 11:42:01 AM by SYSTEM
                      (modified after loading)
    Analysis Method : E:\DATA\GWC\GWC15-11\GWC-15-11-3 2015-11-17 11-05-41\VWD-ADH(1-2)-95-5-
                      220-254NM-40MIN.M (Sequence Method)
    Last changed    : 2/23/2016 9:01:10 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: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  18.482 BB    0.4863 7425.51709  233.97545  91.2545
       2  22.064 MM    0.6197  711.63751   19.13794   8.7455

    Totals :                  8137.15460  253.11339


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