Rhodium-Catalyzed Enantioselective Hydrogenation of α-amino acrylonitriles: An Efficient Approach to Chiral α-amino nitriles

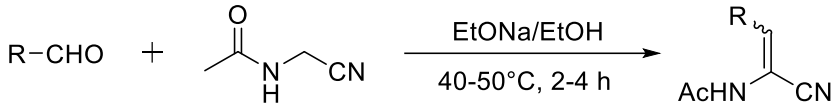
Xiuxiu Li,[+a] Cai You,[+a] Yusheng Yang,[a] Fangyuan Wang,[a] Shuailong Li,[a] Hui Lv,*[a] and Xumu Zhang*[ab]

Contents

1. General Information

Unless otherwise noted, all reagents and solvents were purchased from commercial suppliers and used without further purification. NMR spectra were recorded on Bruker ADVANCE III (400 MHz) spectrometers for ^{1}H NMR and ^{13}C NMR. $CDCl_3$ was the solvent used for the NMR analysis, with tetramethylsilane as the internal standard. Chemical shifts were reported upfield to TMS (0.00 ppm) for ^{1}H NMR and relative to $CDCl_3$ (77.3 ppm) for ^{13}C NMR. Optical rotation was determined using a Perkin Elmer 343 polarimeter. HPLC analysis was conducted on an Agilent 1260 Series instrument. Column Chromatography was performed with silica gel Merck 60 (300-400 mesh). All new products were further characterized by HRMS. A positive ion mass spectrum of sample was acquired on a Thermo LTQ-FT mass spectrometer with an electrospray ionization source.

2. General procedure for the synthesis of compound 1

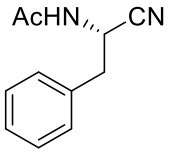


1

Preparation of **1** according to the literature [1]: To a stirred solution of sodium (270 mg, 12 mmol) in ethanol (7.0 mL) was added acetylaminoacetonitrile (980 mg, 10 mmol) at 40-50℃. Five minutes later, aldehyde (10 mmol) was added and the mixture was stirred for another 2-4 h (monitored by TLC). After it was cooled to room temperature, the mixture was poured into water (30 mL), acidified with 3 N aq. HC1 and allowed to stand overnight. The precipitate was collected, washed with 20% aq. ethanol (2 x 30 mL). The residue was redissolved in EtOAc, and then dried over sodium sulfate. Removing the solvent by rotary evaporation, the product mixture was purified by flash column chromatography using 20-50% ethyl acetate/hexane to give the desired products (total 55-87% yield).

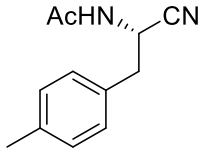
3. General Procedure for Asymmetric Hydrogenation of compound 1

A stock solution was made by mixing $[Rh(NBD)_2]BF_4$ with (*S, S*)-Me-DuPhos in a 1:1.1 molar ratio in trifluoroethanol at room temperature for 30 min in a nitrogen-filled glovebox. An aliquot of the catalyst solution (0.1 mL, 0.001 mmol) was transferred by syringe into the vials charged with different substrates (0.1 mmol for each) in anhydrous trifluoroethanol (1.0 mL). The vials were subsequently transferred into an autoclave into which hydrogen gas was charged. The reaction was then stirred under H_2 (30 atm) at room temperature for 12 h. The hydrogen gas was released slowly and carefully. The solution was concentrated and passed through a short column of silica gel (eluant: ethyl acetate) to remove the metal complex. The *ee* values of all compounds **2** were determined by HPLC analysis on a chiral stationary phase.



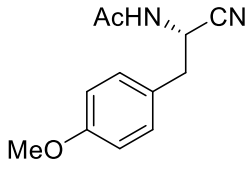
2a

(*S*)-N-(1-cyano-2-phenylethyl)acetamide (2a): White solid; >99% *ee*; $[\alpha]_D^{25}$ = -56.0 (c = 0.77, EtOH), Lit [2]: $[\alpha]_D$ = 45.1 (c = 0.78, EtOH); The enantiomeric excess was determined by HPLC on Chiralpak AD column, hexane: isopropanol = 95: 5; flow rate = 1.0 mL/min; UV detection at 220 nm; t_R = 17.4 min (major), 18.9 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 – 7.31 (m, 3H), 7.29 – 7.24 (m, 2H), 6.56 (d, *J* = 8.2 Hz, 1H), 5.14-5.08 (m, 1H), 3.18 – 2.96 (m, 2H), 1.97 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.0, 134.2, 129.6, 129.2, 128.2, 118.5, 41.9, 38.9, 23.0 ppm. **HRMS** calculated $[M+H]^+$ for $C_{11}H_{13}ON_2$ = 189.1022, found: 189.1021.



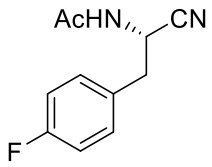
2b

(*S*)-N-(1-cyano-2-(p-tolyl)ethyl)acetamide (2b): White solid; >99% *ee*; $[\alpha]_D^{25}$ = -43.8 (c = 0.80, EtOH); The enantiomeric excess was determined by HPLC on Chiralpak AD column, hexane: isopropanol = 95: 5; flow rate = 1.0 mL/min; UV detection at 220 nm; t_R = 17.0 min (major), 19.2 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.23 – 7.13 (m, 4H), 6.10 (d, *J* = 8.2 Hz, 1H), 5.15-5.09 (m, 1H), 3.10-2.97 (m, 2H), 2.35 (s, 3H), 1.98 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.7, 138.0, 131.0, 130.0, 129.6, 118.5, 41.8, 38.6, 23.1, 21.4 ppm. **HRMS** calculated $[M+H]^+$ for $C_{12}H_{15}ON_2$ = 203.1179, found: 203.1177.



2c

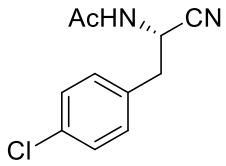
(*S*)-N-(1-cyano-2-(4-methoxyphenyl)ethyl)acetamide (2c): White solid; >99% *ee*; $[\alpha]_D^{25}$ = -36.8 (c = 0.85, EtOH); The enantiomeric excess was determined by HPLC on Chiralpak AD column, hexane: isopropanol = 95: 5; flow rate = 1.0 mL/min; UV detection at 220 nm; t_R = 26.2 min (major), 30.0 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20 (d, *J* = 8.6 Hz, 2H), 6.93 – 6.85 (m, 2H), 6.16 (d, *J* = 8.1 Hz, 1H), 5.12-5.07 (m, 1H), 3.80 (s, 3H), 3.08-2.95 (m, 2H), 1.99 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.7, 159.5, 130.8, 126.0, 118.5, 114.7, 55.6, 42.0, 38.2, 23.1 ppm. **HRMS** calculated $[M+H]^+$ for $C_{12}H_{15}O_2N_2$ = 219.1128, found: 219.1127.



2d

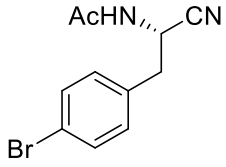
(*S*)-N-(1-cyano-2-(4-fluorophenyl)ethyl)acetamide (2d): White solid; >99% *ee*; $[\alpha]_D^{25}$ = -43.9 (c = 0.35, EtOH); The enantiomeric excess was determined by HPLC on Chiralpak AD column, hexane: isopropanol = 95: 5; flow rate = 1.0 mL/min; UV detection at 220 nm; t_R = 19.2 min (major), 20.0 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28 – 7.23 (m, 2H), 7.10-7.03 (m, 2H), 6.13 (d, *J* = 8.0 Hz, 1H), 5.14-5.09 (m, 1H), 3.11-3.00 (m, 2H), 2.00 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.7, 164.0, 161.5, 131.4, 131.3, 129.9, 129.9, 118.3, 116.4, 116.1, 41.9, 38.3, 23.1 ppm. **HRMS** calculated

$[M+H]^+$ for $C_{11}H_{12}ON_2F$ = 207.0928, found: 207.0926.



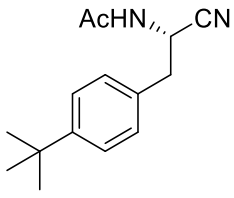
2e

(*S*)-N-(2-(4-chlorophenyl)-1-cyanoethyl)acetamide (2e): White solid; >99% *ee*; $[\alpha]_D^{25}$ = -32.6 (c = 0.67, EtOH); The enantiomeric excess was determined by HPLC on Chiralpak AD column, hexane: isopropanol = 93:7; flow rate = 0.5 mL/min; UV detection at 220 nm; t_R = 26.4 min (major), 36.1min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 (d, *J* = 8.4 Hz, 2H), 7.23 (d, *J* = 8.4 Hz, 2H), 5.86 (d, *J* = 8.3 Hz, 1H), 5.20-5.12 (m, 1H), 3.12-3.00 (m, 2H), 2.01 (s, 3H);^{13}C NMR (100 MHz, $CDCl_3$) δ 169.5, 134.4, 132.5, 131.1, 129.5, 118.1, 41.6, 38.5, 23.1 ppm. **HRMS** calculated $[M+H]^+$ for $C_{11}H_{12}ON_2Cl$ = 223.0633, found: 223.0630.



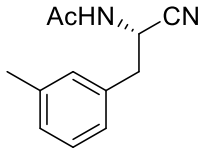
2f

(*S*)-N-(2-(4-bromophenyl)-1-cyanoethyl)acetamide (2f): White solid; >99% *ee*; $[\alpha]_D^{25}$ = -29.9 (c = 0.73, EtOH); The enantiomeric excess was determined by HPLC on Chiralpak AD column, hexane: isopropanol = 93:7; flow rate = 0.6 mL/min; UV detection at 220 nm; t_R = 23.2 min (major), 31.7 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 (d, *J* = 8.4 Hz, 2H), 7.23 (d, *J* = 8.4 Hz, 2H), 5.86 (d, *J* = 8.3 Hz, 1H), 5.20-5.12 (m, 1H), 3.12-2.99 (m, 2H), 2.01 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.6, 133.1, 132.4, 131.4, 122.4, 118.1, 41.6, 38.5, 23.1 ppm. **HRMS** calculated $[M+H]^+$ for $C_{11}H_{12}ON_2Br$ = 267.0128, found: 267.0125.



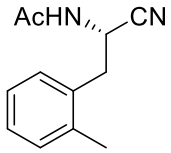
2g

(*S*)-N-(2-(4-(tert-butyl)phenyl)-1-cyanoethyl)acetamide (2g): Yellow oil; 95% *ee*; $[\alpha]_D^{25}$ = -23.7 (c = 0.83, EtOH); The enantiomeric excess was determined by HPLC on Chiralpak AD column, hexane: isopropanol = 95: 5; flow rate = 1.0 mL/min; UV detection at 220 nm; t_R = 11.0 min (major), 12.8 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 – 7.34 (m, 2H), 7.23-7.19 (m, 2H), 6.20 (d, *J* = 8.4 Hz, 1H), 5.17-5.10 (m, 1H), 3.12-2.98 (m, 2H), 1.99 (s, 3H), 1.31 (s, 9H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.7, 151.2, 131.0, 129.4, 126.2, 118.6, 41.8, 38.4, 34.8, 31.5, 23.1 ppm. **HRMS** calculated $[M+H]^+$ for $C_{15}H_{21}ON_2$ = 245.1648, found: 245.1645.



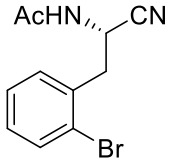
2h

(*S*)-N-(1-cyano-2-(m-tolyl)ethyl)acetamide (2h): White solid; >99% *ee*; $[\alpha]_D^{25}$ = -53.1 (c = 0.47, EtOH); The enantiomeric excess was determined by HPLC on Chiralpak AD column, hexane: isopropanol = 95: 5; flow rate = 1.0 mL/min; UV detection at 220 nm; t_R = 18.4 min (major), 20.5 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28-7.23 (m, 1H), 7.17 – 7.04 (m, 3H), 6.42 – 6.18 (m, 1H), 5.14-5.09 (m, 1H), 3.11-2.97 (m, 2H), 2.36 (s, 3H), 1.99 (s, 3H);^{13}C NMR (100 MHz, $CDCl_3$) δ 169.8, 139.0, 134.1, 134.0, 129.1, 129.0, 126.7, 118.5, 41.8, 38.9, 23.0, 21.6 ppm. **HRMS** calculated $[M+H]^+$ for $C_{12}H_{15}ON_2$ = 203.1179, found: 203.1177.



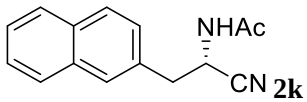
2i

(*S*)-N-(1-cyano-2-(o-tolyl)ethyl)acetamide (2i): White solid; >99% *ee*; $[\alpha]_D^{25}$ = -47.1 (c = 0.33, EtOH); The enantiomeric excess was determined by HPLC on Chiralpak AD column, hexane: isopropanol = 97:3; flow rate = 0.5 mL/min; UV detection at 220 nm; t_R = 62.0 min (minor), 65.5 min (major); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 – 7.16 (m, 4H), 6.19 (s, 1H), 5.15-5.09(m, 1H), 3.12 (d, *J* = 7.3 Hz, 2H), 2.38 (s, 3H), 2.00 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.7, 137.0, 132.5, 131.2, 130.4, 128.3, 126.7, 118.6, 100.2, 41.2, 36.5, 23.1, 19.7 ppm. **HRMS** calculated $[M+H]^+$ for $C_{12}H_{15}ON_2$ = 203.1179, found: 203.1178.



2j

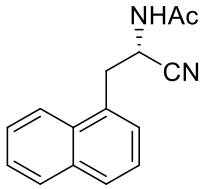
(*S*)-N-(2-(2-bromophenyl)-1-cyanoethyl)acetamide (2j): White solid; 98% *ee*; $[\alpha]_D^{25}$ = -38.1 (c = 0.69, EtOH); The enantiomeric excess was determined by HPLC on Chiralpak AD column, hexane: isopropanol = 95: 5; flow rate = 1.0 mL/min; UV detection at 220 nm; t_R = 24.4 min (major), 26.3 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56 (dd, *J* = 8.0, 1.1 Hz, 1H), 7.41 – 7.26 (m, 3H), 7.21 – 7.12 (m, 1H), 5.15 (q, *J* = 7.8 Hz, 1H), 3.34-3.22 (m, 2H), 1.97 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.4, 133.9, 133.2, 131.8, 129.7, 128.0, 124.7, 118.1, 40.6, 38.7, 22.8 ppm. **HRMS** calculated $[M+H]^+$ for $C_{11}H_{12}ON_2Br$ = 267.0128, found: 267.0125.



2k

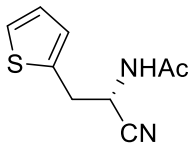
(*S*)-N-(1-cyano-2-(naphthalen-2-yl)ethyl)acetamide (2k): White solid; >99% *ee*; $[\alpha]_D^{25}$ = -34.5 (c = 0.67, EtOH); The enantiomeric excess was determined by HPLC on Chiralpak AD column, hexane: isopropanol = 95: 5; flow rate = 1.0 mL/min; UV detection at 220 nm; t_R = 24.2 min (major), 28.3 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85-7.80 (m, 3H), 7.71 (s, 1H), 7.51-7.47 (m, 2H),

7.37 (d, *J* = 8.4 Hz, 1H), 6.35 (d, *J* = 8.3 Hz, 1H), 5.22-5.17(m, 1H), 3.27-3.14 (m, 2H), 1.93 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.9, 133.6, 133.0, 131.6, 129.1, 128.8, 128.0, 127.3, 126.8, 126.5, 118.5, 41.8, 39.1, 23.0 ppm. **HRMS** calculated $[M+H]^+$ for $C_{15}H_{15}ON_2$ = 239.1179, found: 239.1176.



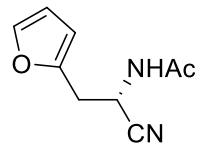
2l

(*S*)-N-(1-cyano-2-(naphthalen-1-yl)ethyl)acetamide (2l): Yellow solid; 93% *ee*; $[\alpha]_D^{25}$ = -30.1 (c = 0.71, EtOH); The enantiomeric excess was determined by HPLC on Chiralpak AD column, hexane: isopropanol = 95: 5; flow rate = 1.0 mL/min; UV detection at 220 nm; t_R = 18.9 min (major), 21.3 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (d, *J* = 8.4 Hz, 1H), 7.88 (d, *J* = 7.9 Hz, 1H), 7.85 – 7.80 (m, 1H), 7.59 – 7.49 (m, 2H), 7.48 – 7.41 (m, 2H), 6.39 (d, *J* = 8.1 Hz, 1H), 5.32-5.24 (m, 1H), 3.64-3.45 (m, 2H), 1.94 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.0, 134.2, 132.0, 130.3, 129.4, 129.1, 128.6, 127.1, 126.3, 125.8, 123.2, 118.4, 41.9, 36.3, 23.0 ppm. **HRMS** calculated $[M+H]^+$ for $C_{15}H_{15}ON_2$ = 239.1179, found: 239.1176.



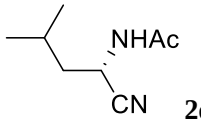
2m

(*S*)-N-(1-cyano-2-(thiophen-2-yl)ethyl)acetamide (2m): White solid; >99% *ee*; $[\alpha]_D^{25}$ = -36.9 (c = 0.57, EtOH); The enantiomeric excess was determined by HPLC on Chiralpak AD column, hexane: isopropanol = 94:6; flow rate = 0.6 mL/min; UV detection at 220 nm; t_R = 26.1 min (major), 27.8 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29 – 7.26 (m, 1H), 7.09 – 6.99 (m, 2H), 6.39 (s, 1H), 5.18-5.11 (m, 1H), 3.41 – 3.22 (m, 2H), 2.01 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.8, 135.4, 128.0, 127.8, 126.0, 118.2, 41.8, 33.2, 23.1 ppm. **HRMS** calculated $[M+H]^+$ for $C_9H_{11}ON_2S$ = 195.0587, found: 195.0586.



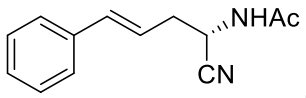
. **2n**

(*S*)-N-(1-cyano-2-(furan-2-yl)ethyl)acetamide (2n): colorless oil; >99% *ee*; $[\alpha]_D^{25}$ = -19.2 (c = 0.31, EtOH); The enantiomeric excess was determined by HPLC on Chiralpak AD column, hexane: isopropanol = 95: 5; flow rate = 1.0 mL/min; UV detection at 220 nm; t_R = 19.8 min (major), 24.1 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 (d, *J* = 1.7 Hz, 1H), 6.39-6.36 (m, 1H), 6.30 (d, *J* = 3.2 Hz, 1H), 6.12 (d, *J* = 7.3 Hz, 1H), 5.21-5.16 (m, 1H), 3.18-3.14 (m, 2H), 2.04 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.6, 148.6, 143.2, 117.9, 111.1, 109.5, 40.2, 31.7, 23.2 ppm. **HRMS** calculated $[M+H]^+$ for $C_9H_{11}O_2N_2$ = 179.0815, found: 179.0813.



2o

(*S*)-N-(1-cyano-3-methylbutyl)acetamide (2o): colorless oil; 97% *ee*; $[\alpha]_D^{25}$ = -36.5 (c = 0.46, EtOH); The enantiomeric excess was determined by HPLC on Chiralpak OJ column, hexane: isopropanol = 95: 5; flow rate = 1.0 mL/min; UV detection at 210 nm; t_R = 11.6 min (minor), 13.5 min (major); ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.02 (d, *J* = 7.6 Hz, 1H), 4.92 (q, *J* = 8.2 Hz, 1H), 2.04 (s, 3H), 1.86-1.71 (m, 2H), 1.67-1.60 (m, 1H), 0.98 (dd, *J* = 6.5, 1.9 Hz, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.7, 119.1, 42.1, 39.2, 25.1, 23.1, 22.5, 22.1 ppm. **HRMS** calculated $[M+H]^+$ for $C_8H_{15}ON_2$ = 155.1179, found: 155.1177.



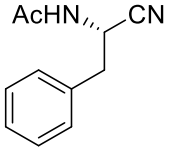
2p

(*S*,*E*)-N-(1-cyano-4-phenylbut-3-en-1-yl)acetamide (2p): Yellow solid; >99% *ee*; $[\alpha]_D^{25}$ = -13.6 (c = 0.70, EtOH); The enantiomeric excess was determined by HPLC on Chiralpak AD column, hexane: isopropanol = 95: 5; flow rate = 1.0 mL/min; UV detection at 220 nm; t_R = 18.5 min (major), 20.1 min (minor); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 – 7.25 (m, 6H), 6.59 (d, *J* = 15.8 Hz, 1H), 6.26 – 6.11 (m, 2H), 5.07-5.01 (m, 1H), 2.80 – 2.60 (m, 2H), 2.02 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 180.8, 169.8, 136.3, 136.3, 129.0, 128.4, 126.7, 121.5, 118.5, 40.4, 36.7, 23.1 ppm. **HRMS** calculated $[M+H]^+$ for $C_{13}H_{15}ON_2$ = 215.1179, found: 215.1178.

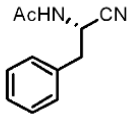
References

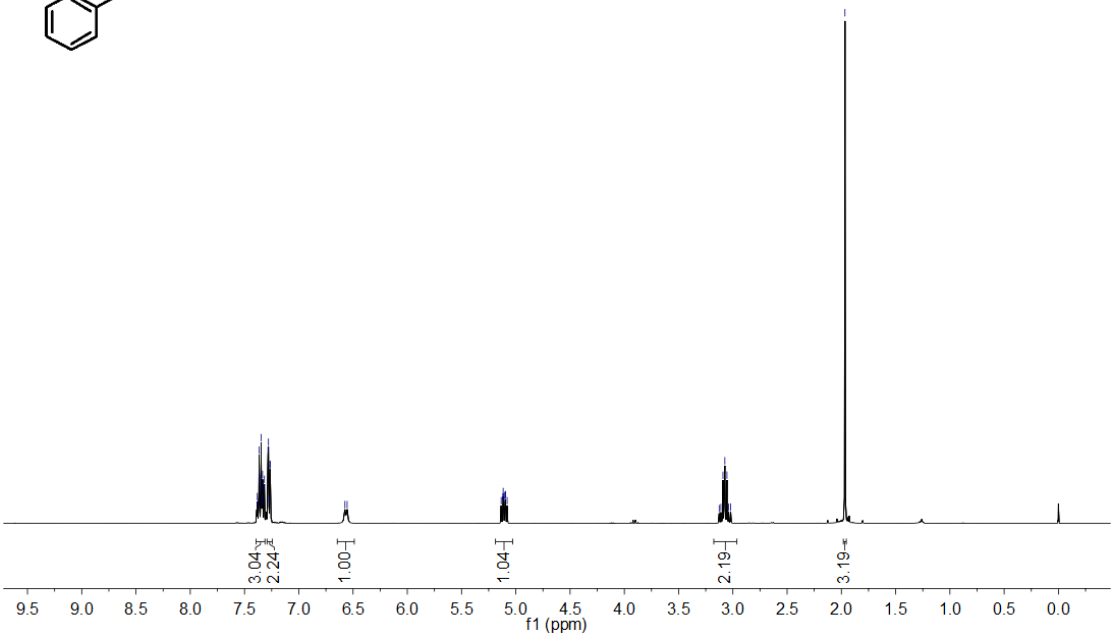
1. P. Zeng, Y. Hu, H. Hu. *Synth. Commun*., **1995**, *25*, 1167-1172;
2. R. S. Atkinson, M. P. Coogan, I. S. T. Lochrie. *J. Chem. Soc*., *Perkin Trans. 1*, **1997**, 897-900.

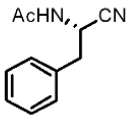
4. NMR spectra of 2

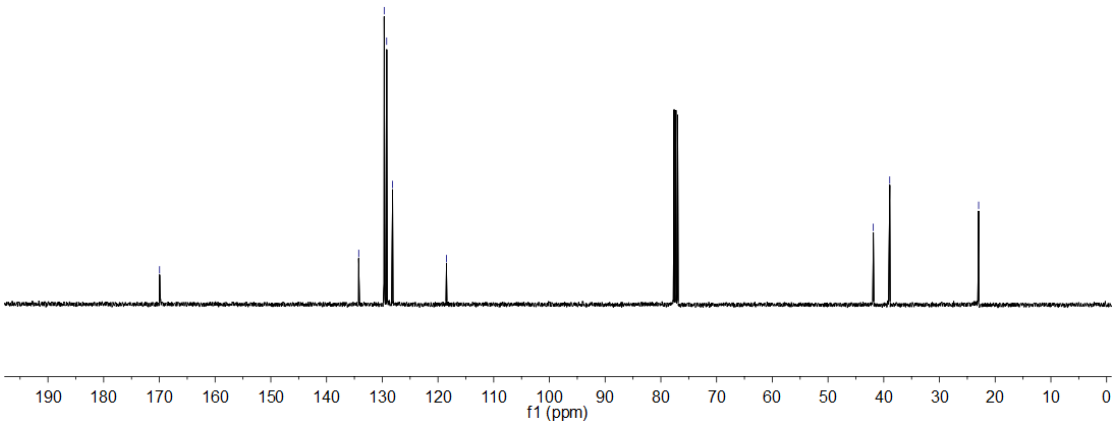


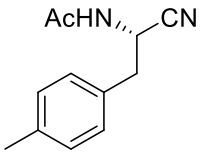
2a










AcHN
CN

2b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7.15
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5.11
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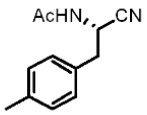
1.98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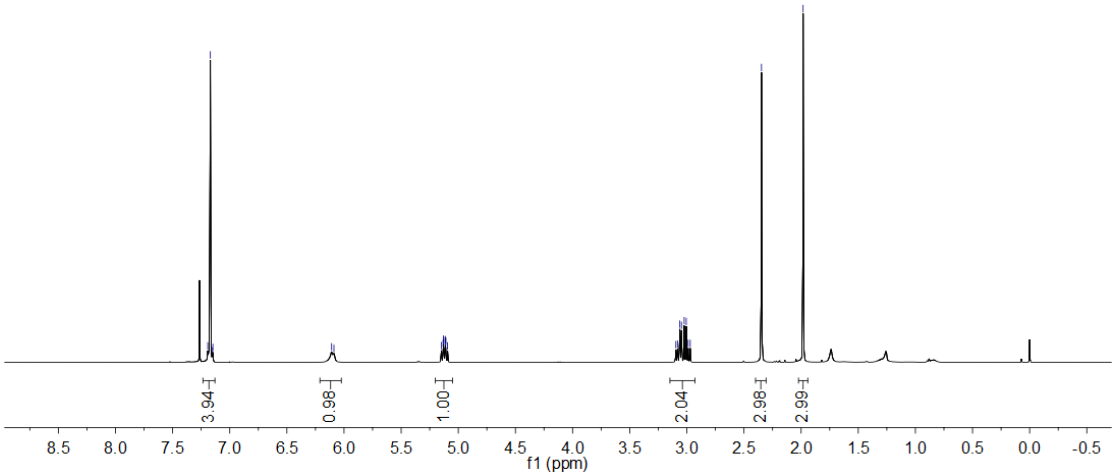
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4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
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f1 (ppm)


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137.99
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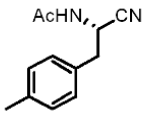
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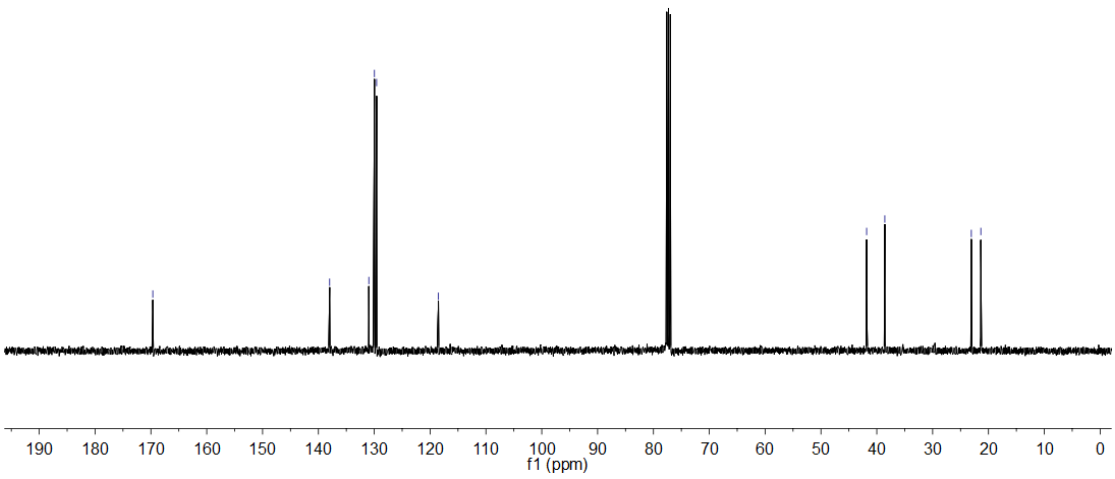
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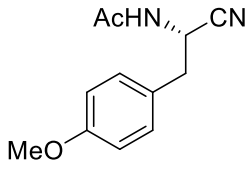
f1 (ppm)


AcHN
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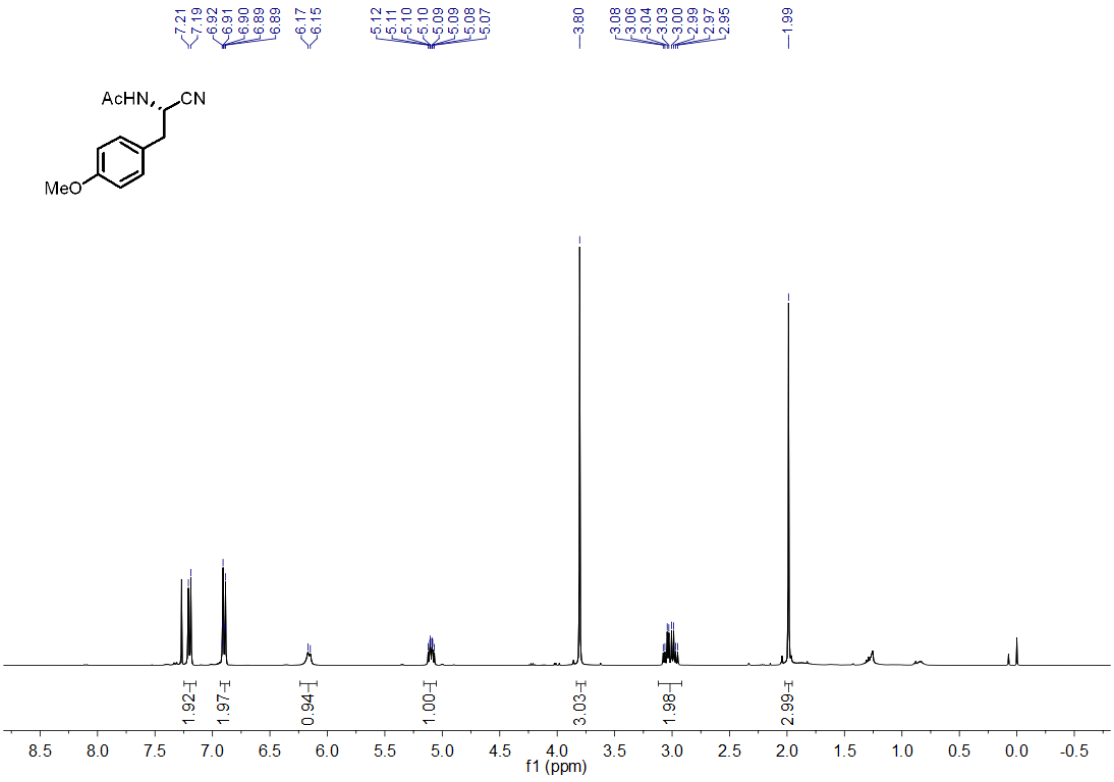
MeO

2c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2.97
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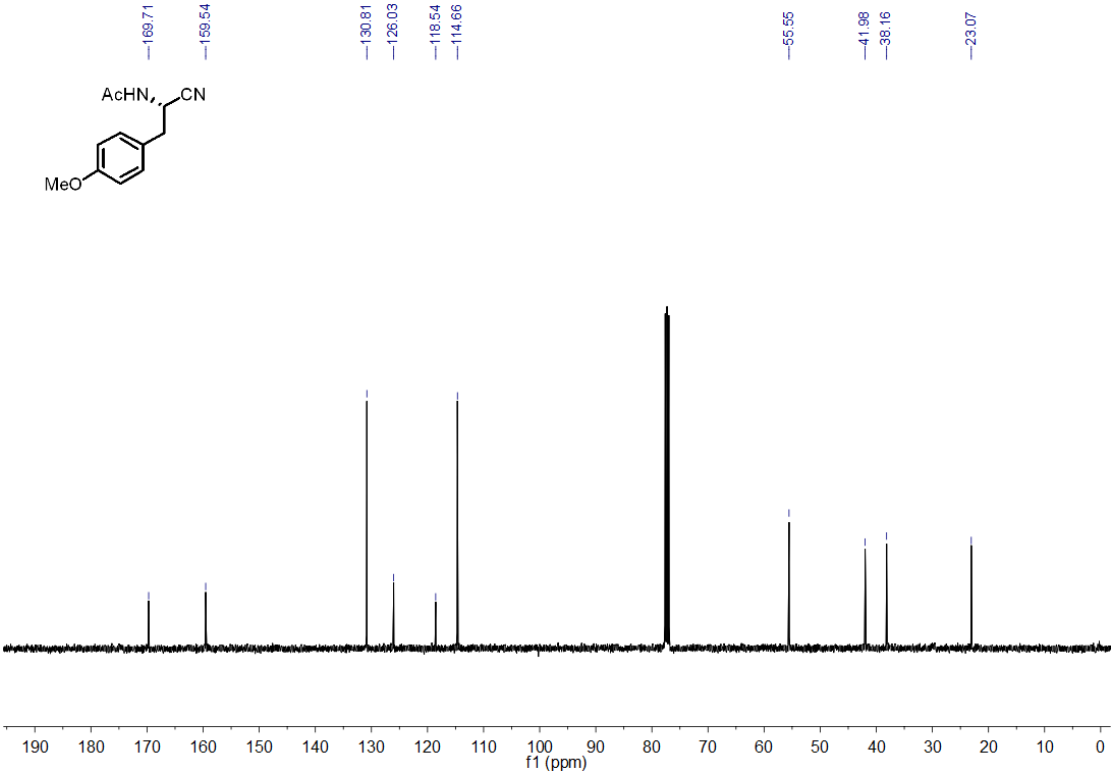
f1 (ppm)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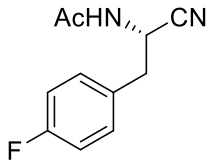
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CN
F

2d


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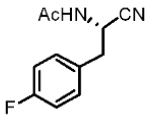
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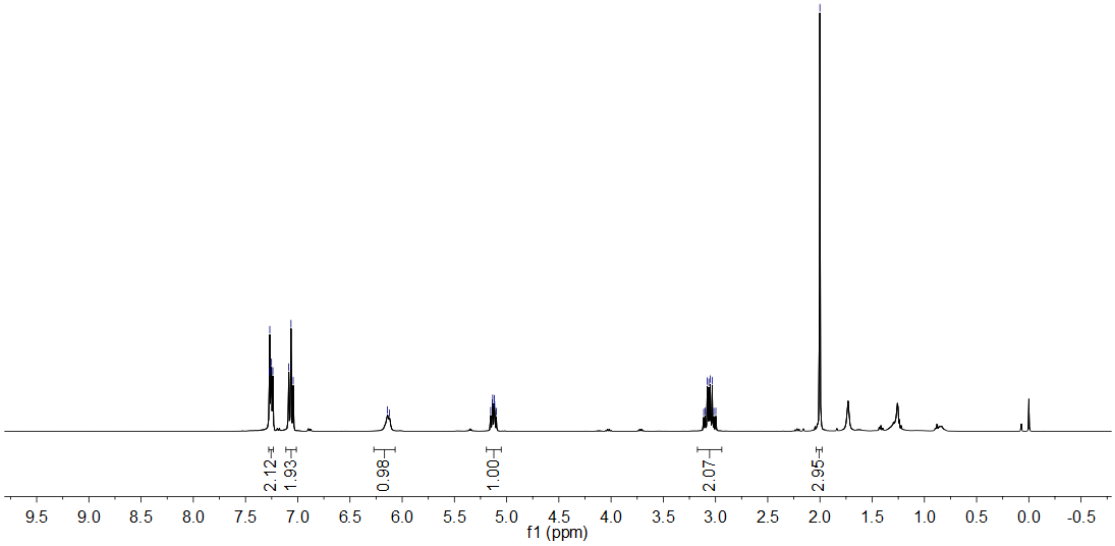
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f1 (ppm)


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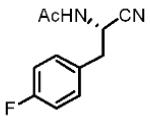
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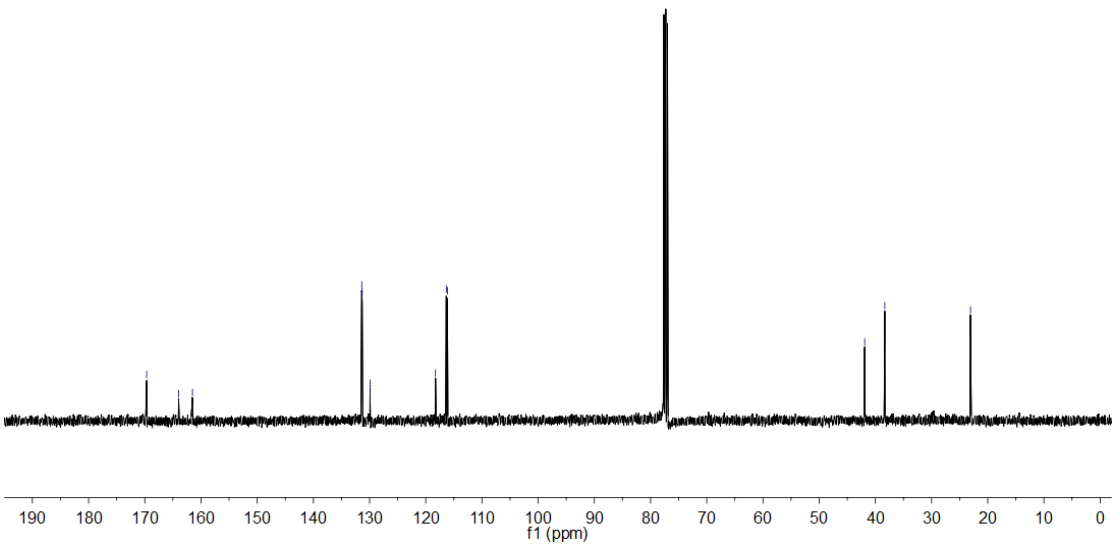
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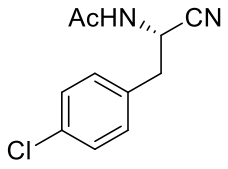
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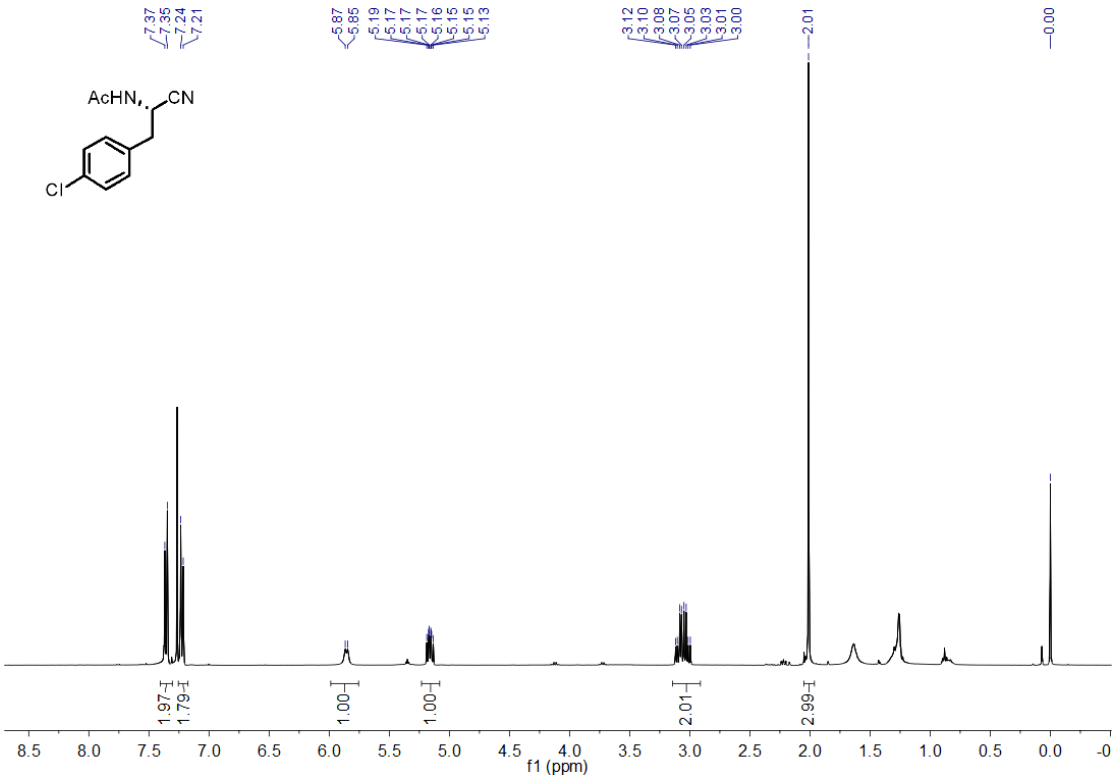
CN
Cl

2e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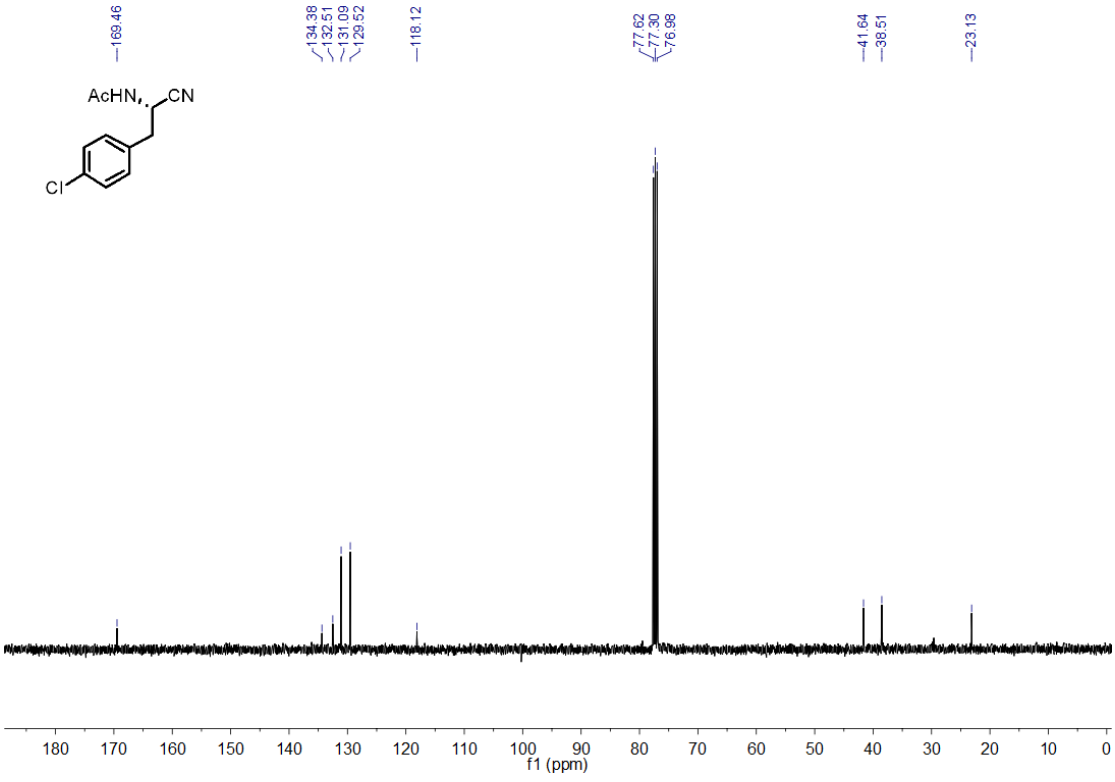
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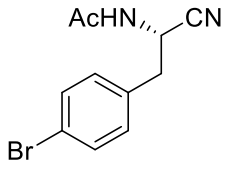
Cl
f1 (ppm)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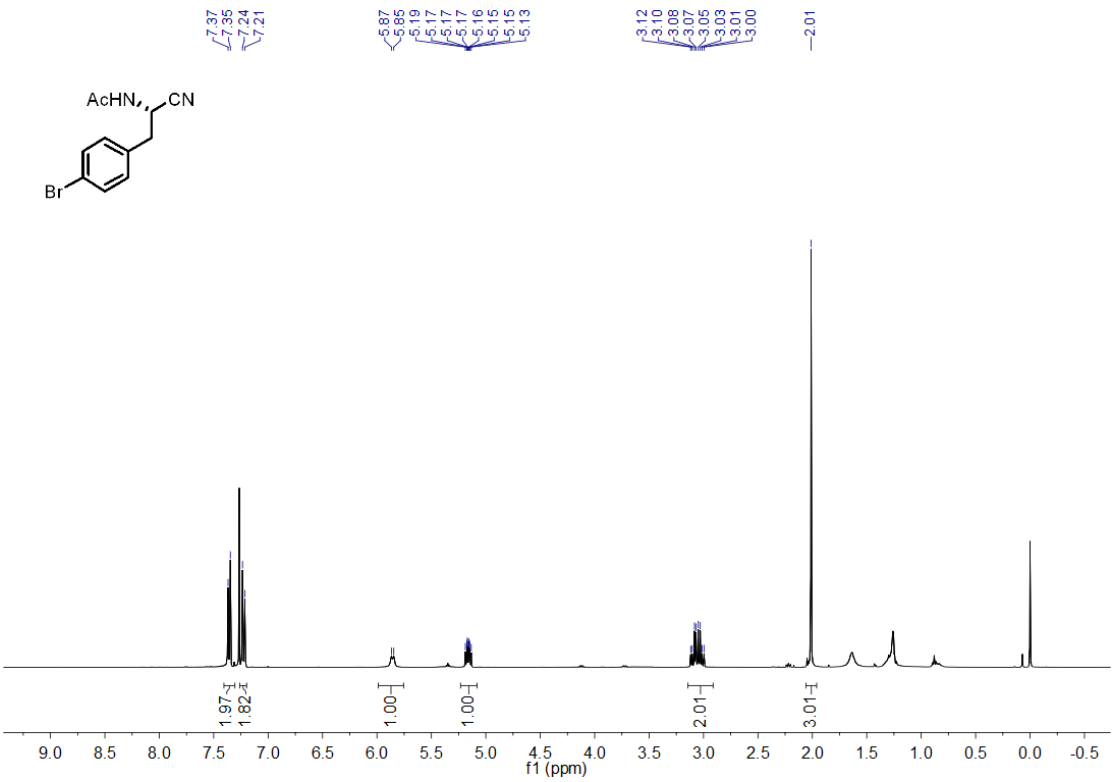
2f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3.00
2.01
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CN
Br
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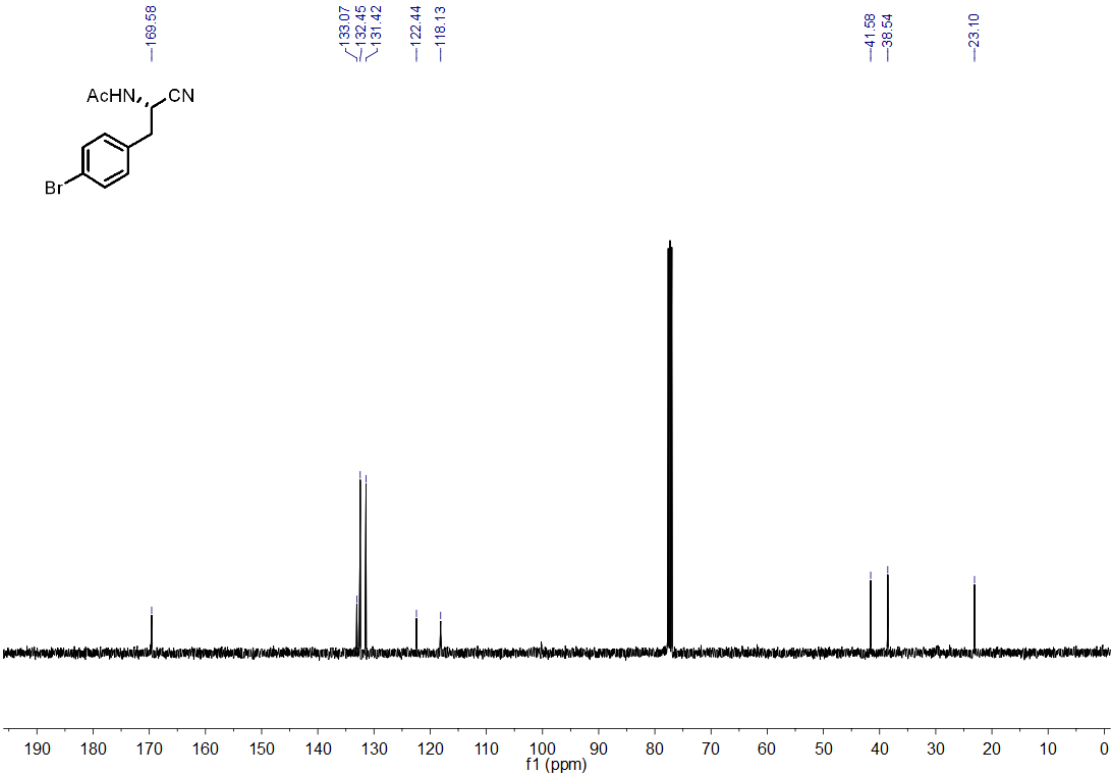
1.82
1.00
1.00
2.01
3.01
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)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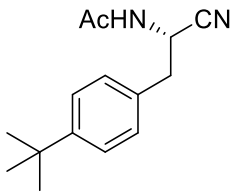
f1 (ppm)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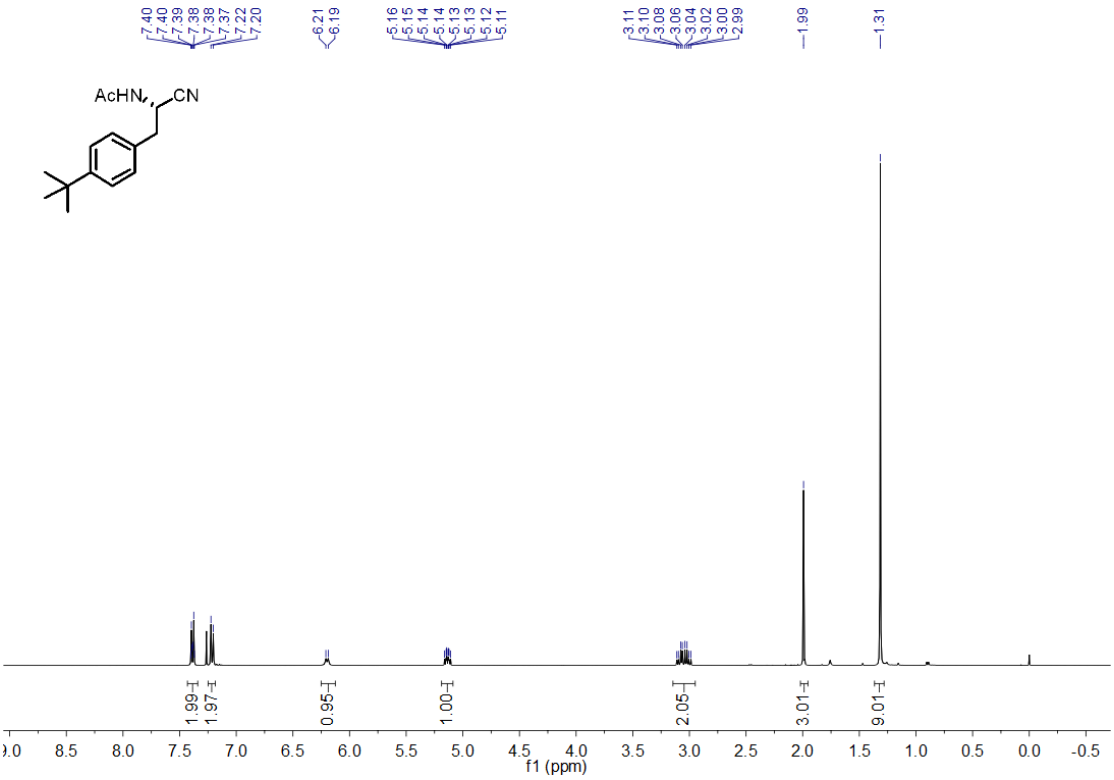
2g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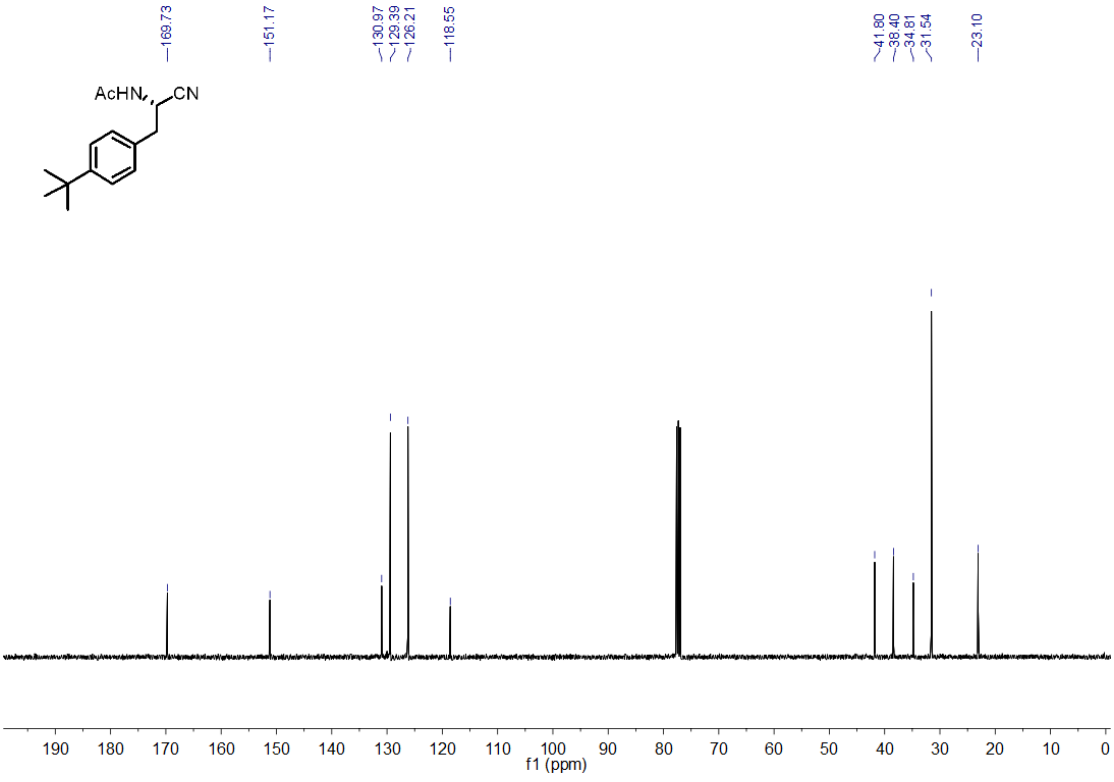
f1 (ppm)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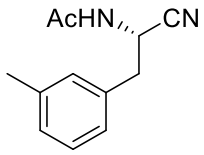
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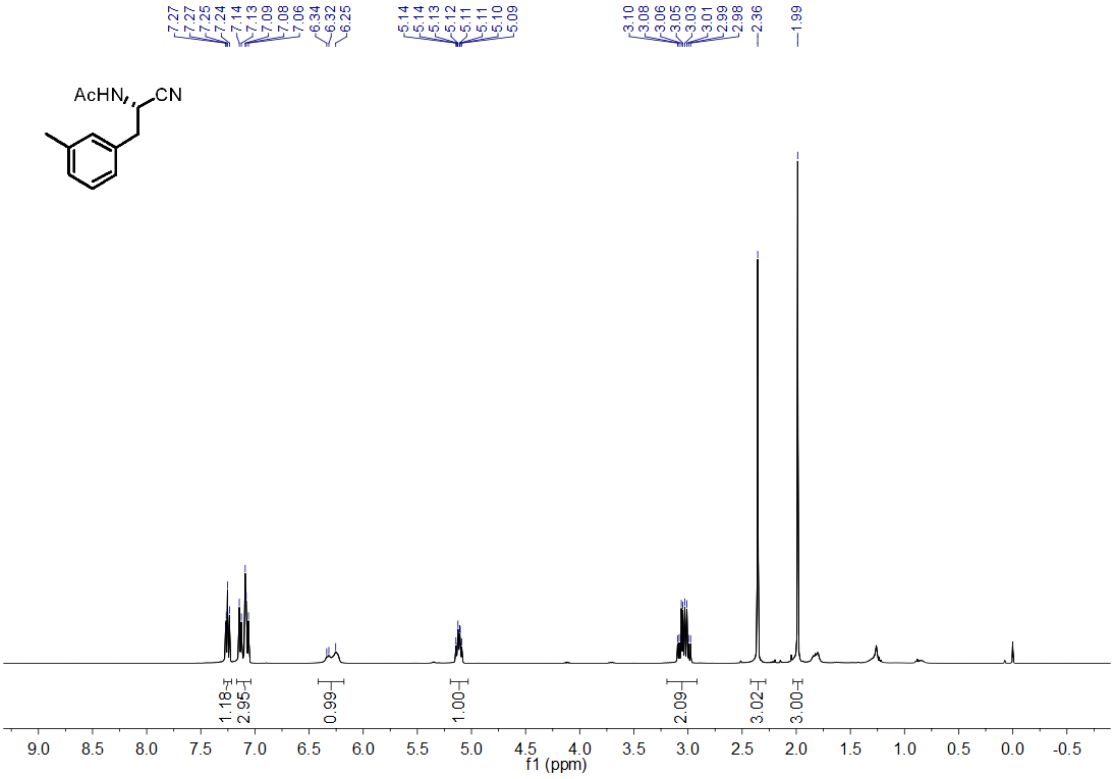
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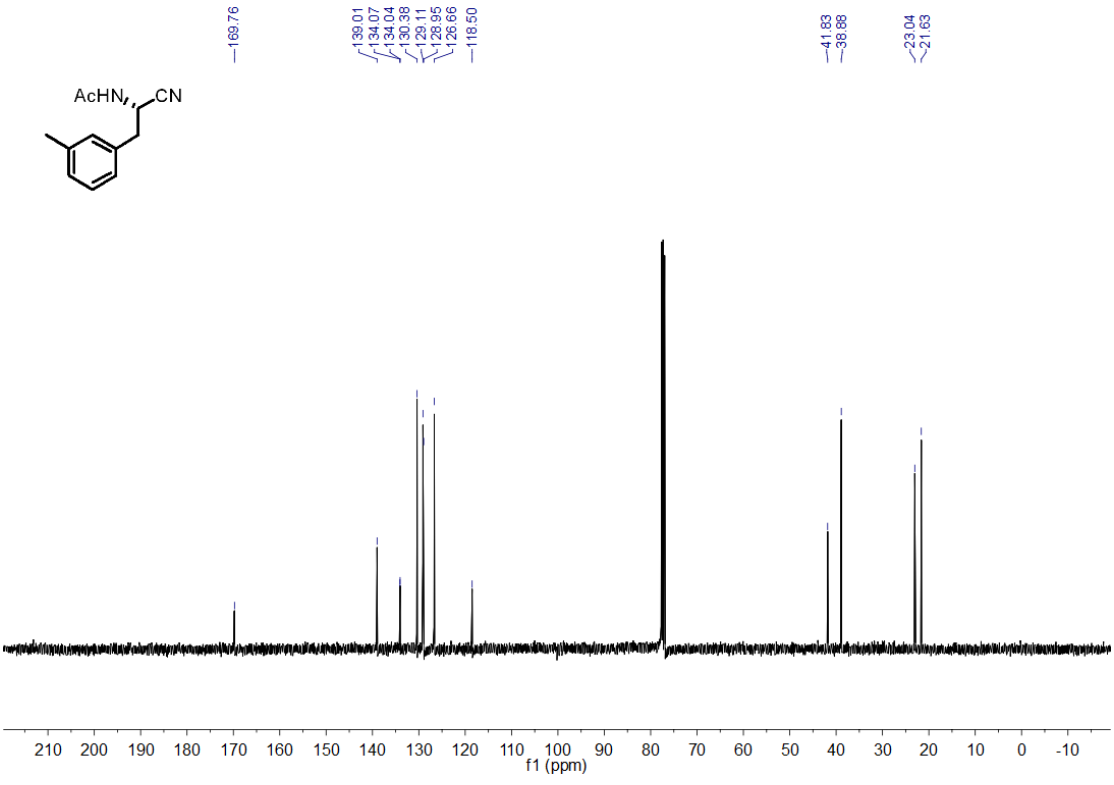
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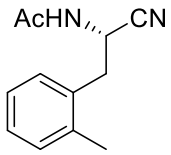
AcHN
CN

2i


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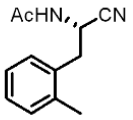
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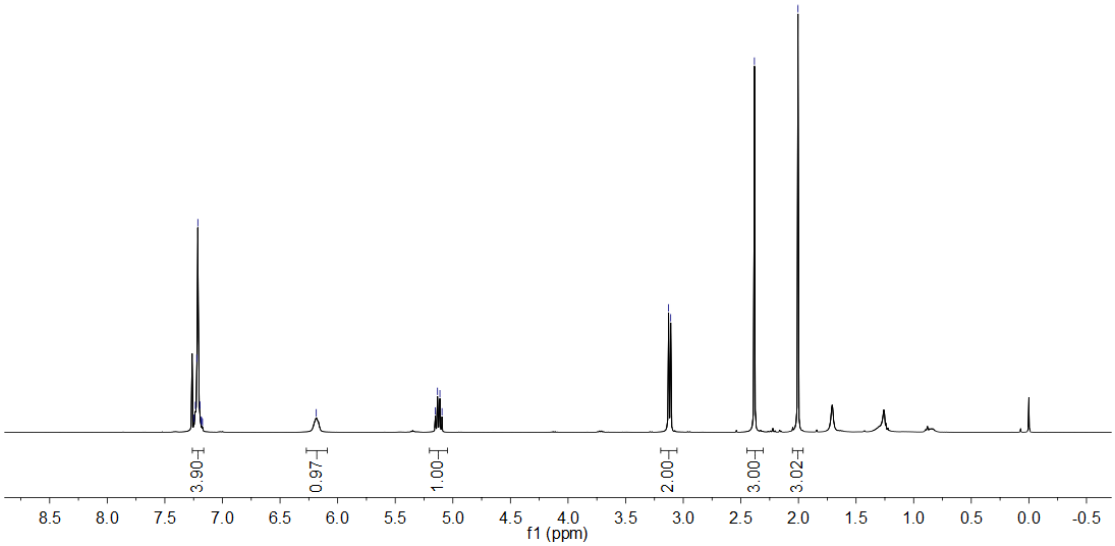
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f1 (ppm)


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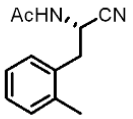
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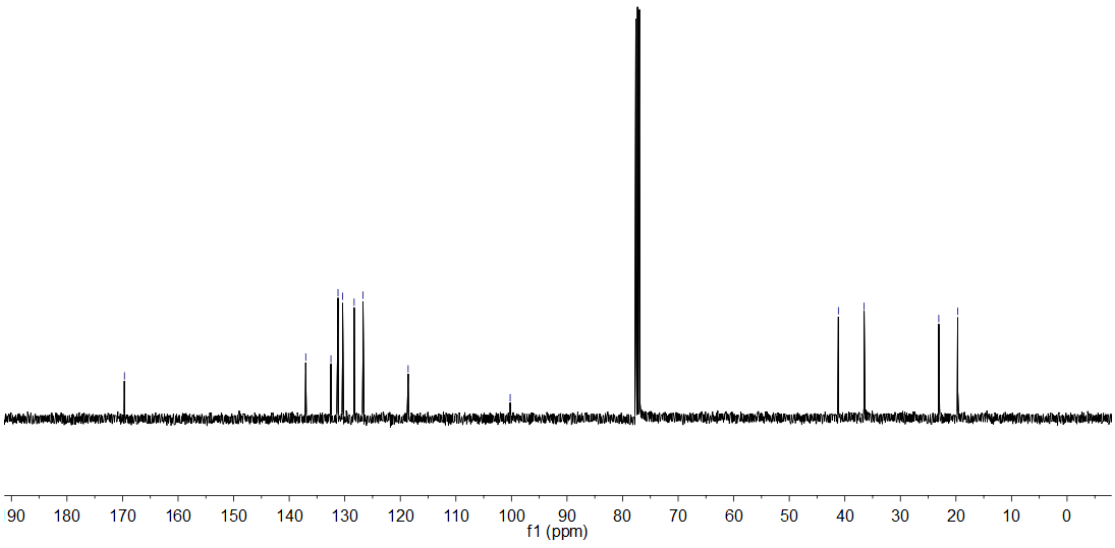
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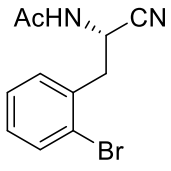
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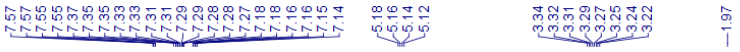
2j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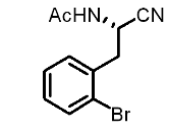
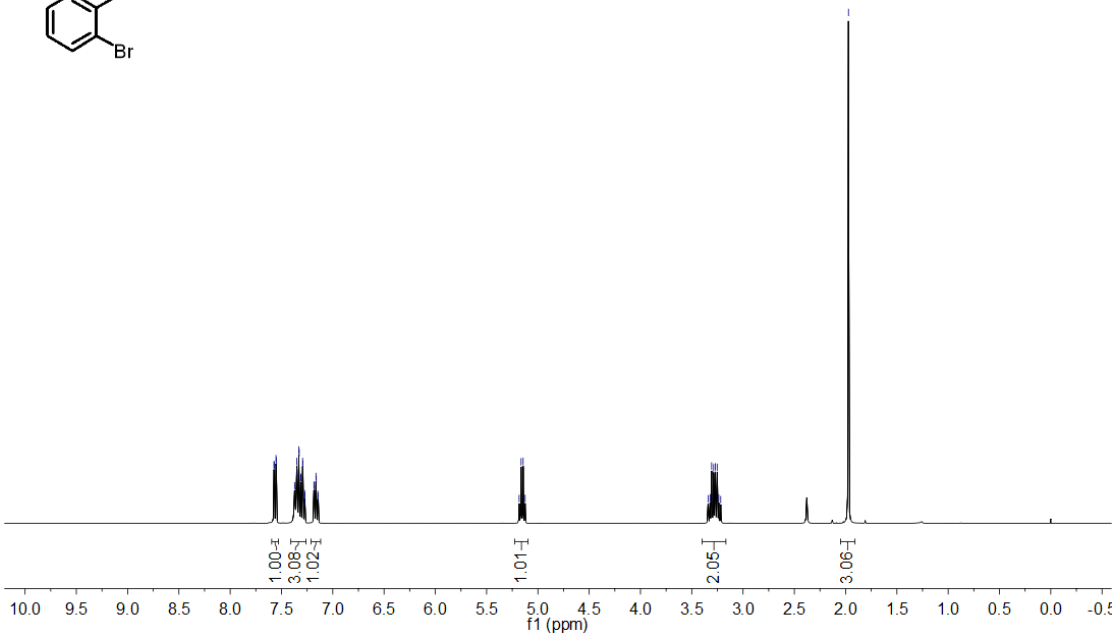
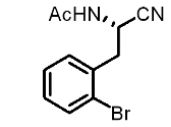
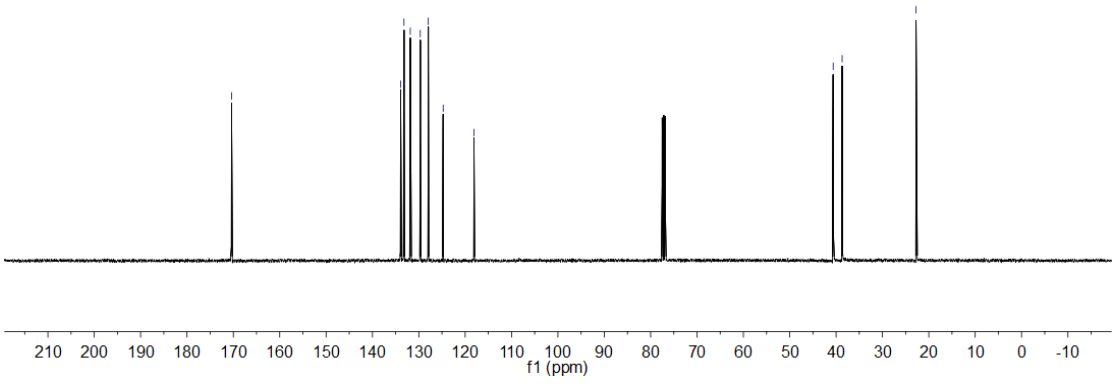
3.24
3.22
1.97

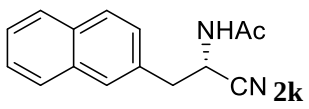

AcHN
CN
Br


1.00
3.08
1.02
1.01
2.05
3.06
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


170.39
133.92
133.20
131.84
129.68
127.96
124.74
118.10
40.63
38.69
22.75

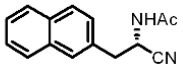

AcHN
CN
Br

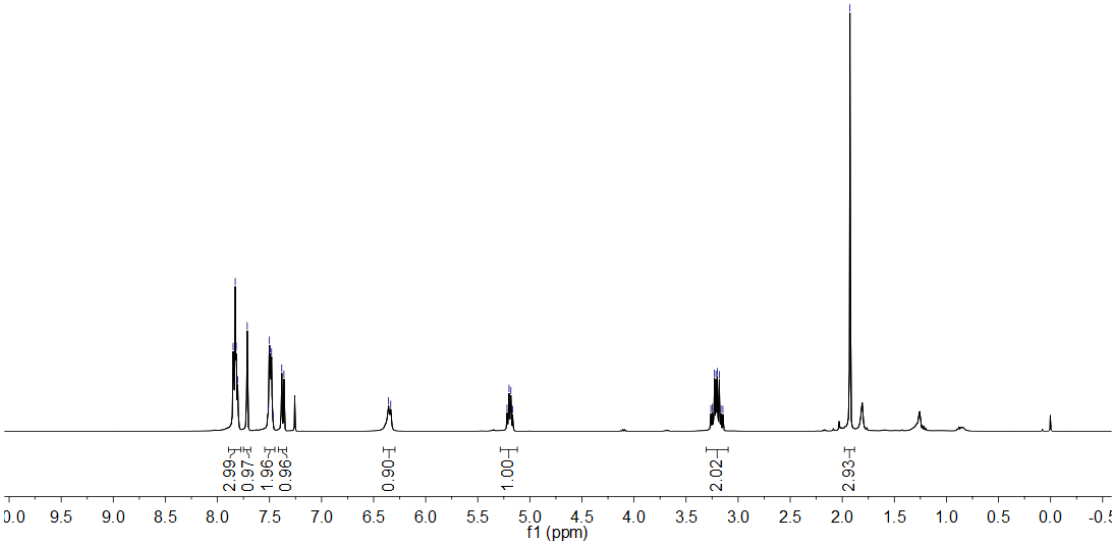

210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


NHAc
CN

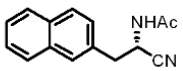
2k

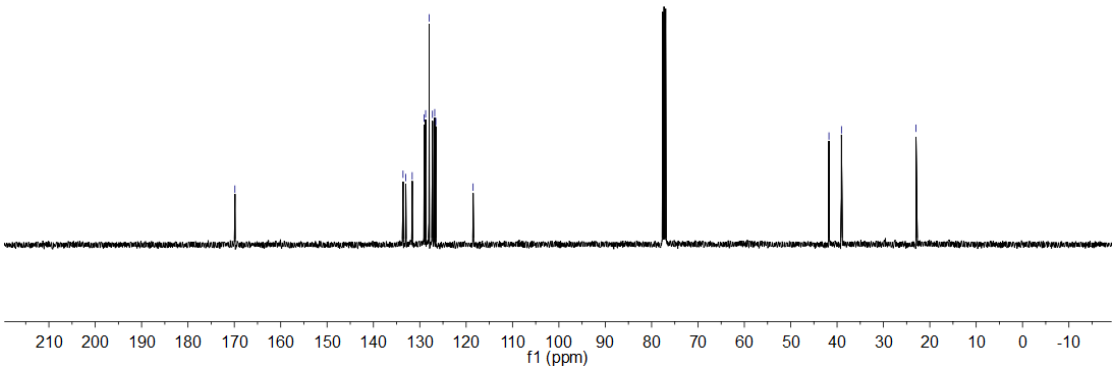

7.85
7.83
7.82
7.80
7.71
7.51
7.50
7.49
7.48
7.47
7.38
7.36
6.36
6.34
5.22
5.20
5.18
5.17
3.26
3.25
3.23
3.21
3.20
3.18
3.16
3.15
1.93

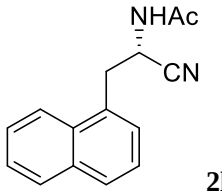

NHAc
CN


2.99
0.97
1.96
0.96
0.90
1.00
2.02
2.93
f1 (ppm)

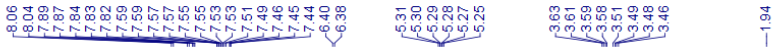

169.87
133.61
133.01
131.63
129.06
128.75
127.98
127.27
126.78
126.54
118.50
41.75
39.06
22.97

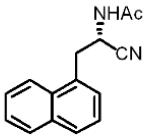

NHAc
CN

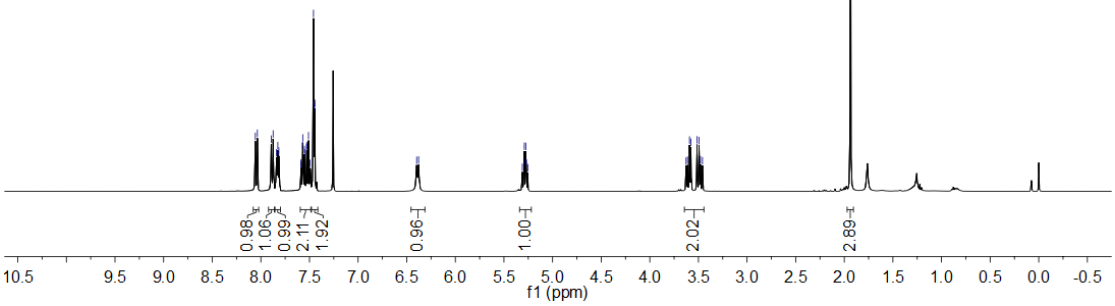

f1 (ppm)

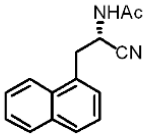

NHAc
CN

2l

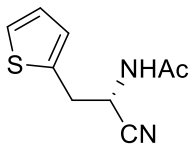

8.06
8.04
7.89
7.87
7.84
7.83
7.82
7.59
7.59
7.57
7.57
7.55
7.55
7.53
7.53
7.51
7.49
7.46
7.45
7.44
6.40
6.38
5.31
5.30
5.29
5.28
5.27
5.25
3.63
3.61
3.59
3.58
3.51
3.49
3.48
3.46
1.94


NHAc
CN

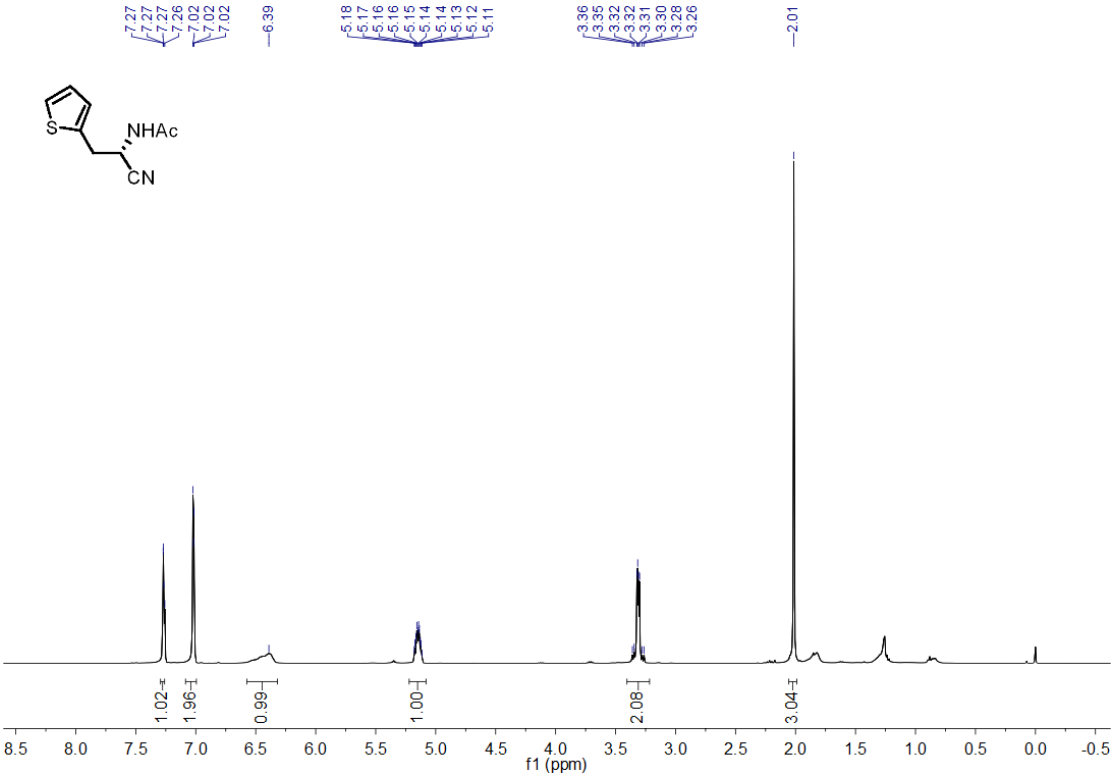

0.98
1.06
0.99
2.11
1.92
0.96
1.00
2.02
2.89
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

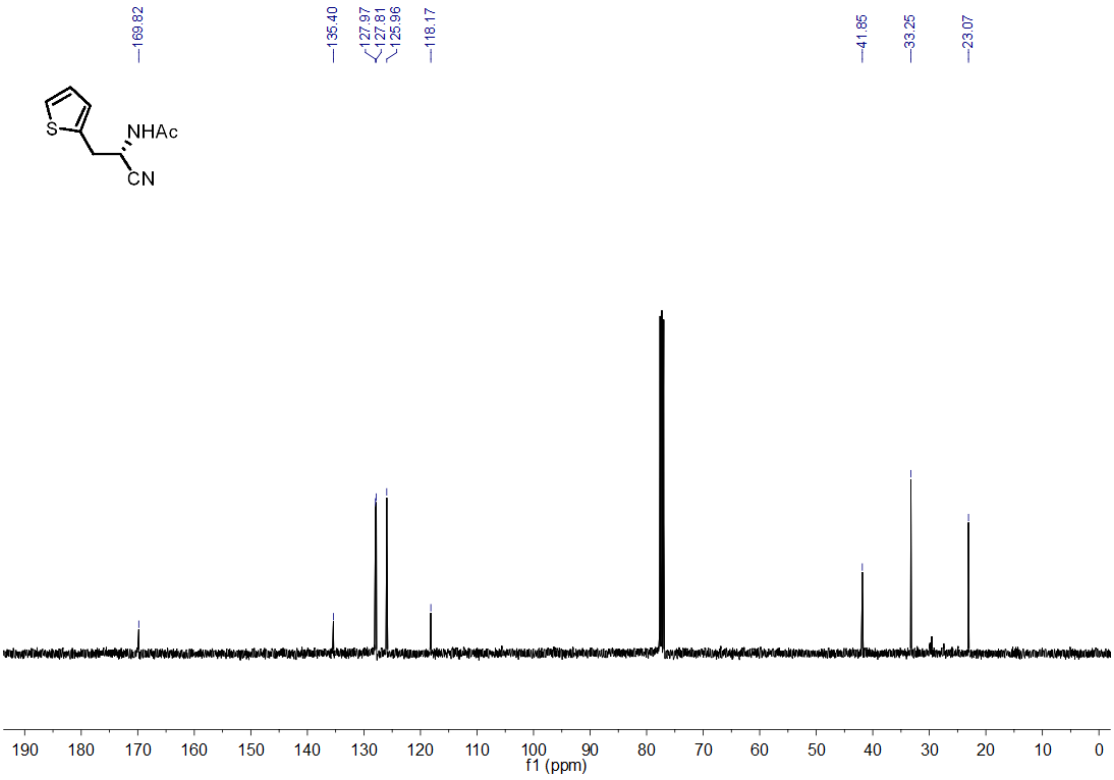

NHAc
CN

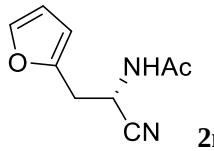
169.96
134.18
132.03
130.30
129.36
129.06
128.59
127.08
126.34
125.75
123.16
118.44
41.94
36.26
23.02
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


S
NHAc
CN

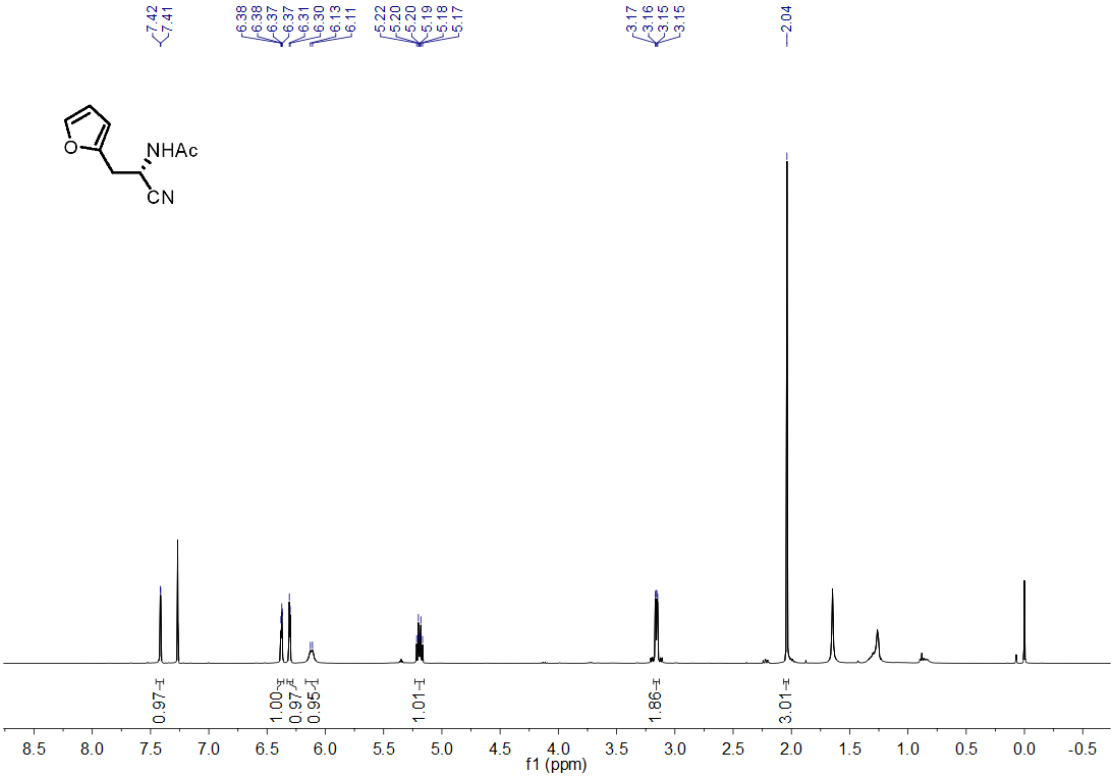
2m

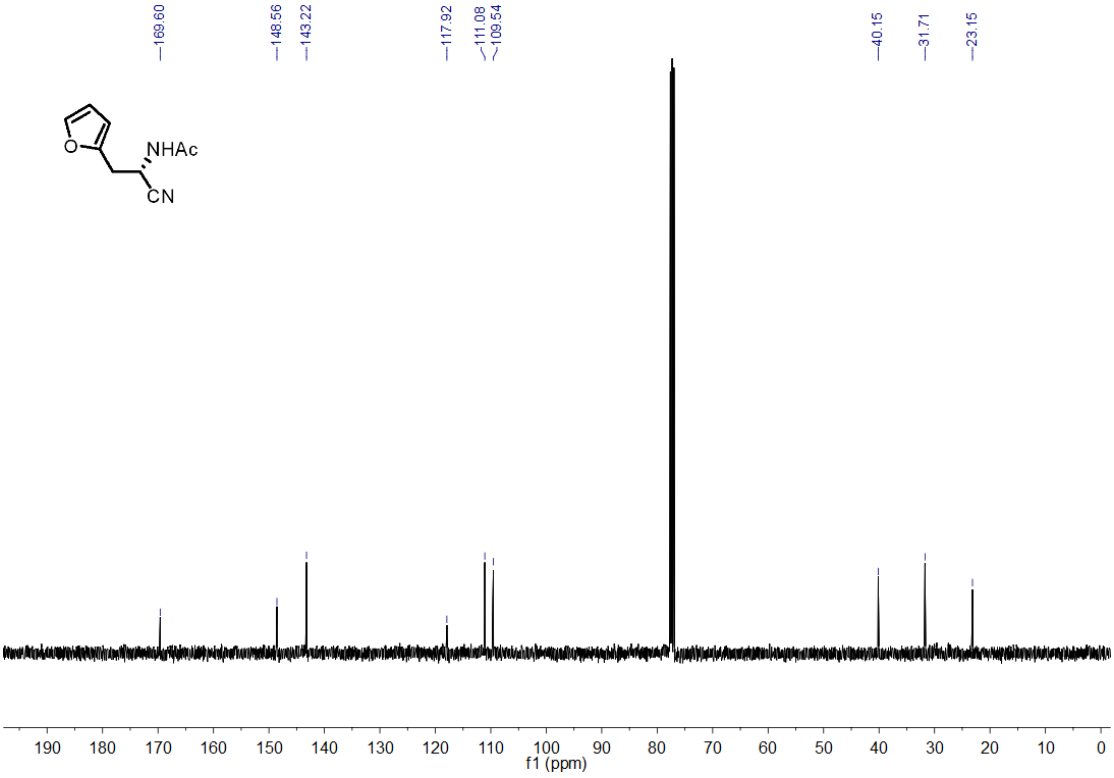

7.27
7.27
7.27
7.26
7.02
7.02
7.02
6.39
5.18
5.17
5.16
5.16
5.15
5.14
5.14
5.13
5.12
5.11
3.36
3.35
3.32
3.32
3.31
3.30
3.28
3.26
2.01
S
NHAc
CN
1.02
1.96
0.99
1.00
2.08
3.04
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

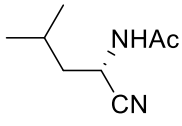

169.82
135.40
127.97
127.81
125.96
118.17
41.85
33.25
23.07
S
NHAc
CN
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


NHAc
CN

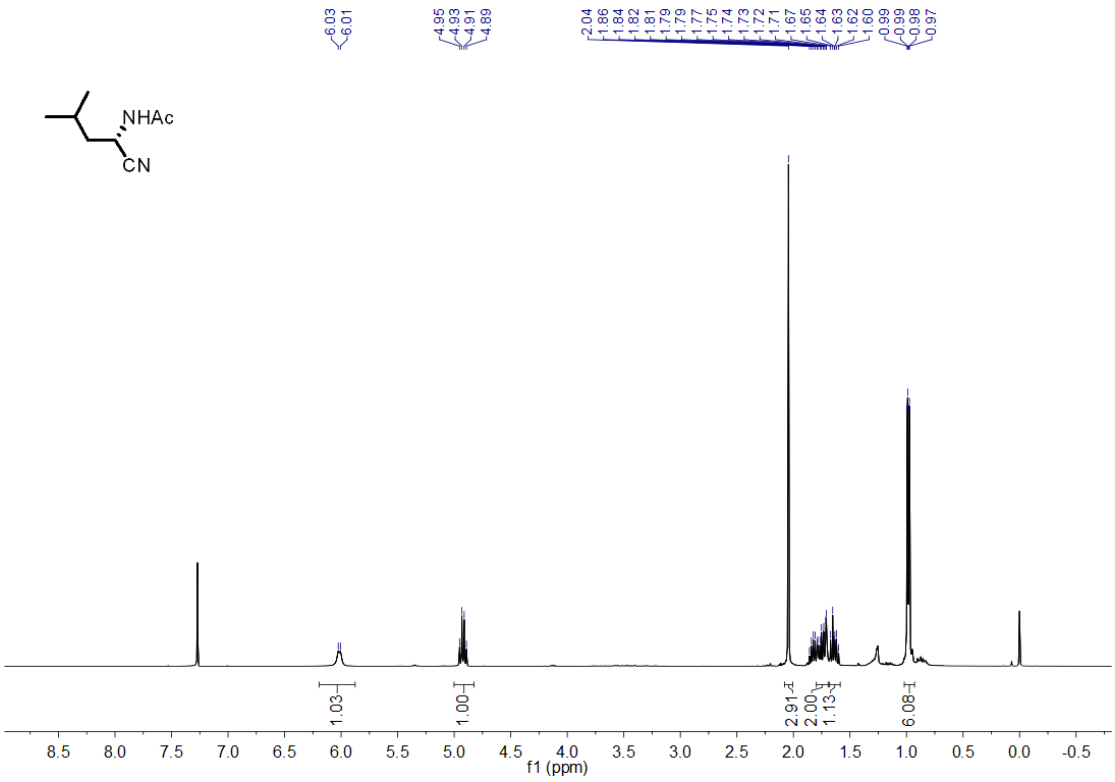
2n

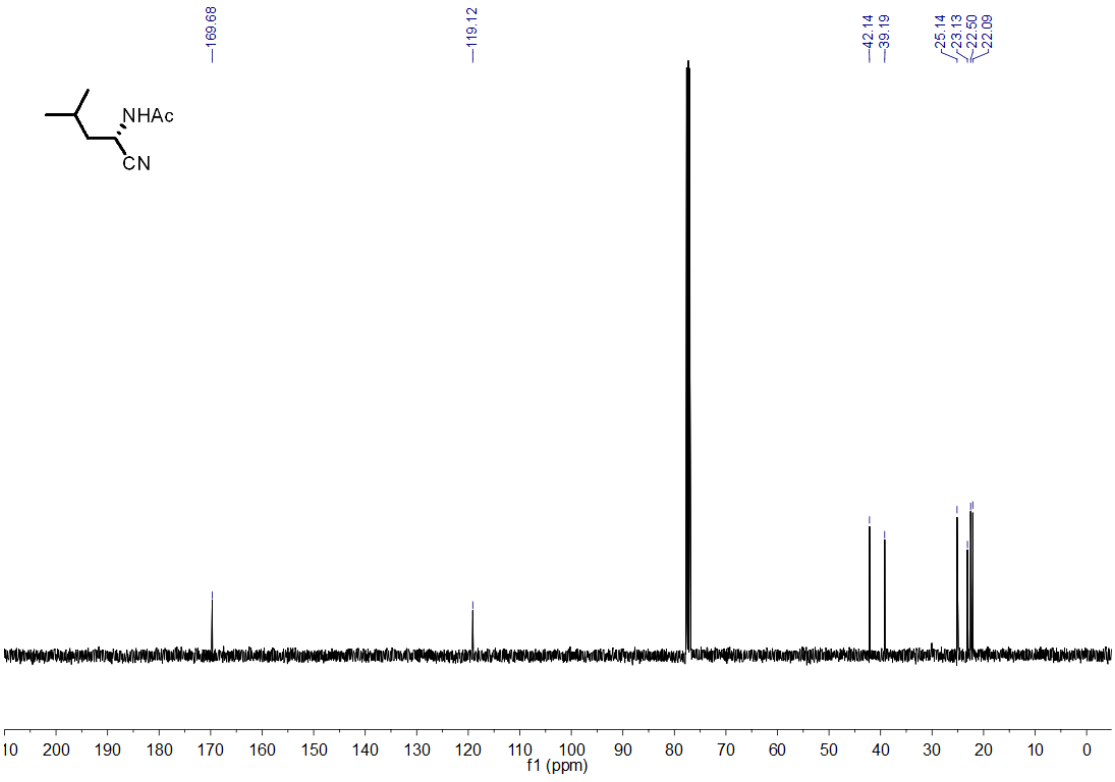

7.42
7.41
6.38
6.38
6.37
6.37
6.31
6.30
6.13
6.11
5.22
5.20
5.20
5.19
5.18
5.17
3.17
3.16
3.15
3.15
2.04
NHAc
CN
0.97
1.00
0.97
0.95
1.01
1.86
3.01
f1 (ppm)

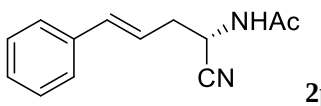

169.60
148.56
143.22
117.92
111.08
109.54
40.15
31.71
23.15
NHAc
CN
f1 (ppm)


NHAc
CN

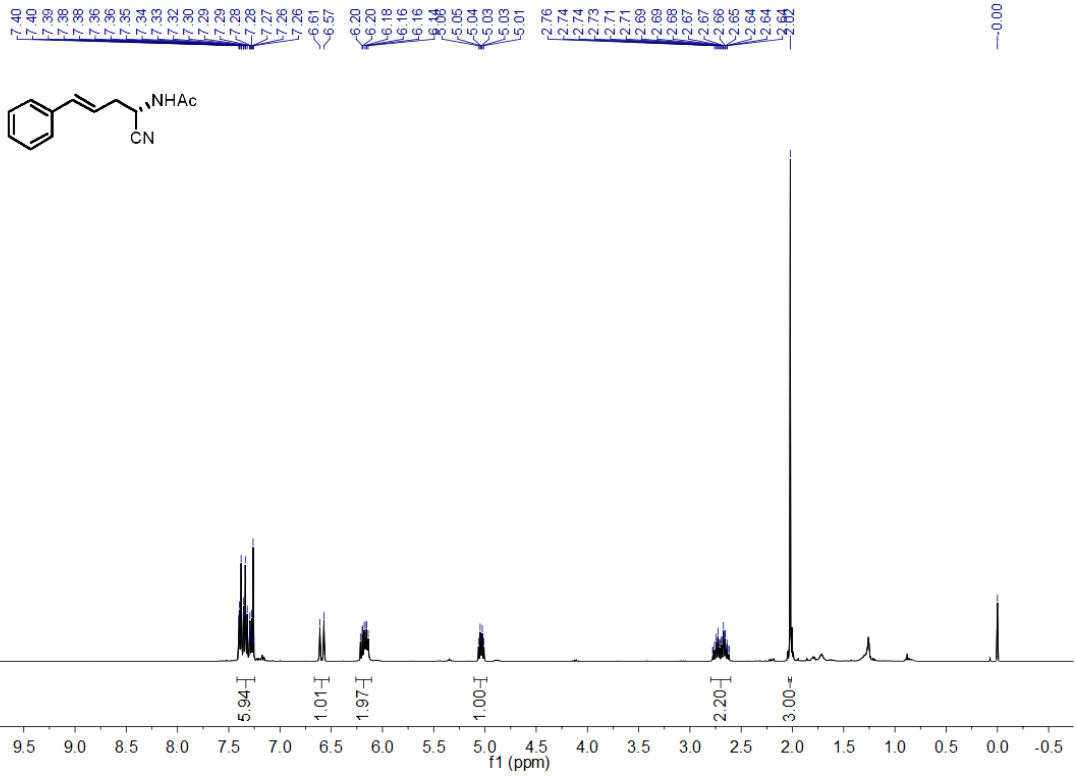
2o

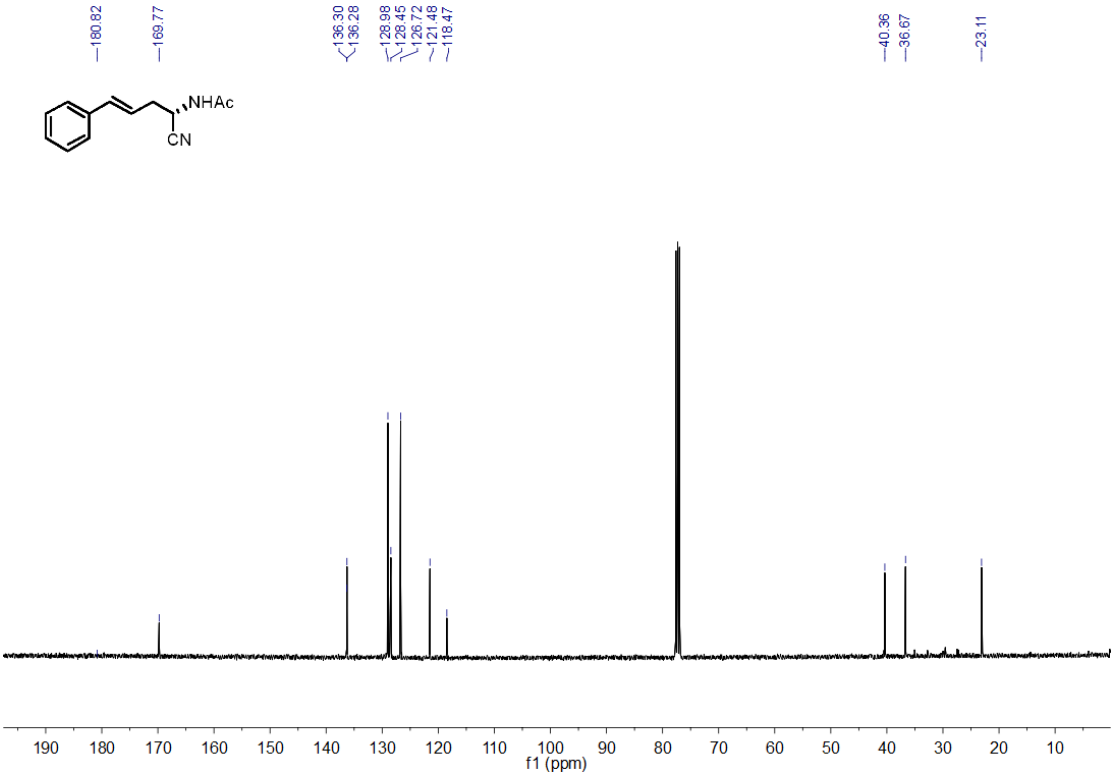

6.03
6.01
4.95
4.93
4.91
4.89
2.04
1.86
1.84
1.82
1.81
1.79
1.79
1.77
1.75
1.74
1.73
1.72
1.71
1.67
1.65
1.64
1.63
1.62
1.60
0.99
0.99
0.98
0.97
NHAc
CN
1.03
1.00
2.91
2.00
1.13
6.08
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


169.68
119.12
42.14
39.19
25.14
23.13
22.50
22.09
NHAc
CN
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

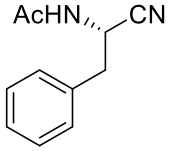

NHAc
CN

2p


7.40
7.40
7.39
7.38
7.38
7.36
7.36
7.35
7.34
7.33
7.32
7.30
7.29
7.29
7.28
7.28
7.27
7.26
7.26
6.61
6.57
6.20
6.20
6.18
6.16
6.16
6.14
6.08
5.05
5.04
5.03
5.03
5.01
2.76
2.74
2.74
2.73
2.71
2.71
2.69
2.69
2.68
2.67
2.67
2.66
2.65
2.64
2.64
2.64
2.62
0.00
NHAc
CN
5.94
1.01
1.97
1.00
2.20
3.00
f1 (ppm)


180.82
169.77
136.30
136.28
128.98
128.45
126.72
121.48
118.47
40.36
36.67
23.11
NHAc
CN
f1 (ppm)

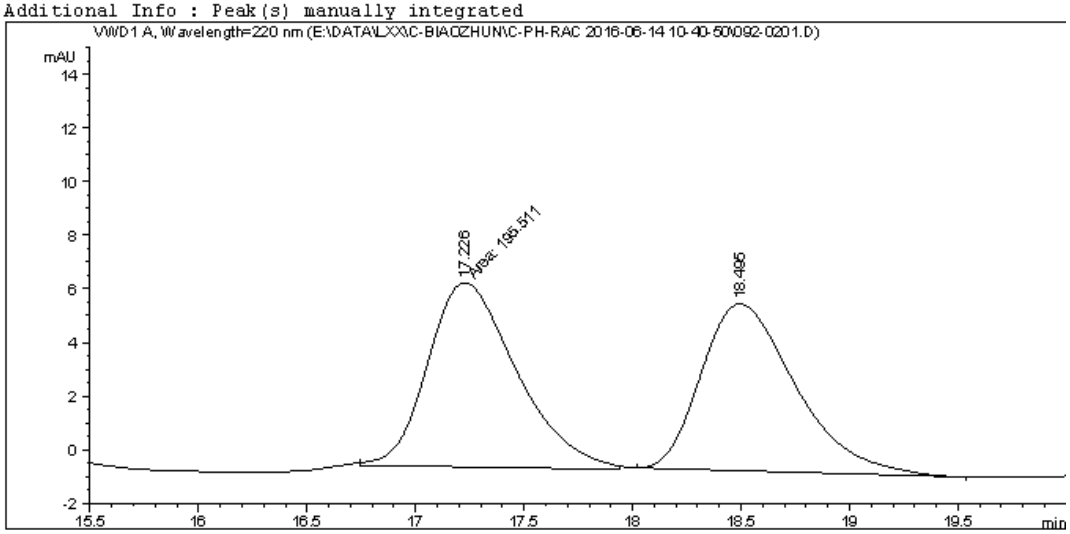
5. HPLC spectra of 2



2a

Data File E:\DATA\LXX\C-BIAOZHUN\C-PH-RAC 2016-06-14 10-40-50\092-0201.D
Sample Name: C-Ph-Rac

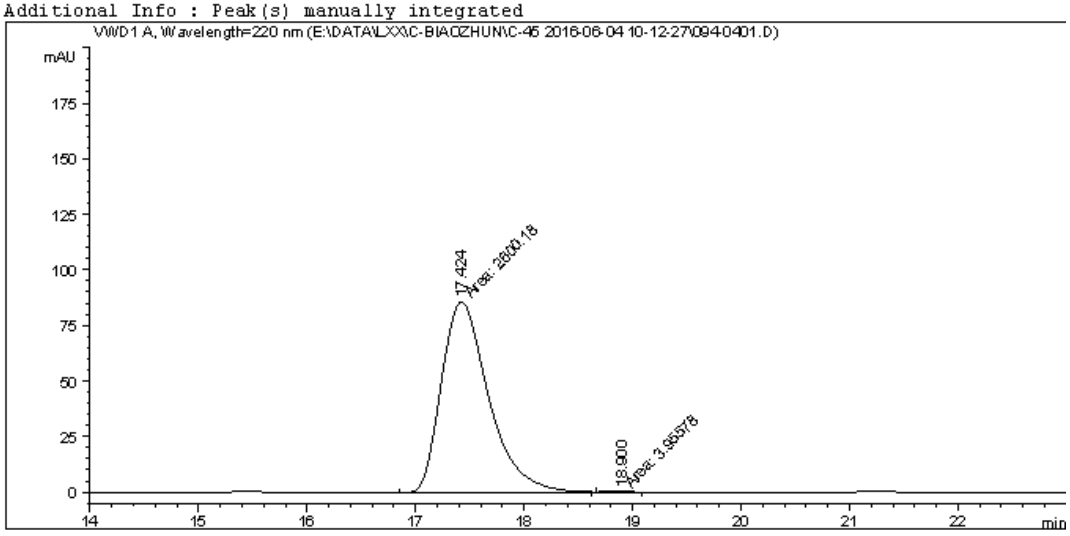
```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   2
    Acq. Instrument : 1260HPLC-VWD                   Location : Vial 92
    Injection Date  : 6/14/2016 11:09:55 AM              Inj :   1
                                                   Inj Volume : 3.000 µl
    Acq. Method     : E:\DATA\LXX\C-BIAOZHUN\C-PH-RAC 2016-06-14 10-40-50\VWD-AD(1-6)-95-5-1ML
                      -3UL-220-40MIN.M
    Last changed    : 6/14/2016 10:40:51 AM by SYSTEM
    Analysis Method : E:\DATA\LXX\C-BIAOZHUN\C-PH-RAC 2016-06-14 10-40-50\VWD-AD(1-6)-95-5-1ML
                      -3UL-220-40MIN.M (Sequence Method)
    Last changed    : 6/14/2016 1:27:44 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```



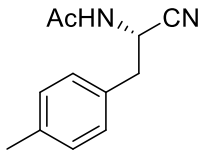

```
    ======================================================================
                              Area Percent Report
    ======================================================================

    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  17.226 MM    0.4737  195.51149    6.87861  50.9462
       2  18.495 BB    0.4676  188.24924    6.22795  49.0538

    Totals :                   383.76073   13.10656


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

Data File E:\DATA\LXX\C-BIAOZHUN\C-45 2016-06-04 10-12-27\094-0401.D
Sample Name: c-45-3

```
==========================================================================
Acq. Operator   : SYSTEM                          Seq. Line :   4
Acq. Instrument : 1260HPLC-VWD                     Location : Vial 94
Injection Date  : 6/4/2016 11:36:29 AM                Inj :   1
                                                  Inj Volume : 3.000 µl
Acq. Method     : E:\DATA\LXX\C-BIAOZHUN\C-45 2016-06-04 10-12-27\VWD-AD(1-6)-95-5-1.0ML-
                  3UL-220-30MIN.M
Last changed    : 6/4/2016 10:12:28 AM by SYSTEM
Analysis Method : E:\DATA\LXX\C-BIAOZHUN\C-45 2016-06-04 10-12-27\VWD-AD(1-6)-95-5-1.0ML-
                  3UL-220-30MIN.M (Sequence Method)
Last changed    : 6/6/2016 9:16:06 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



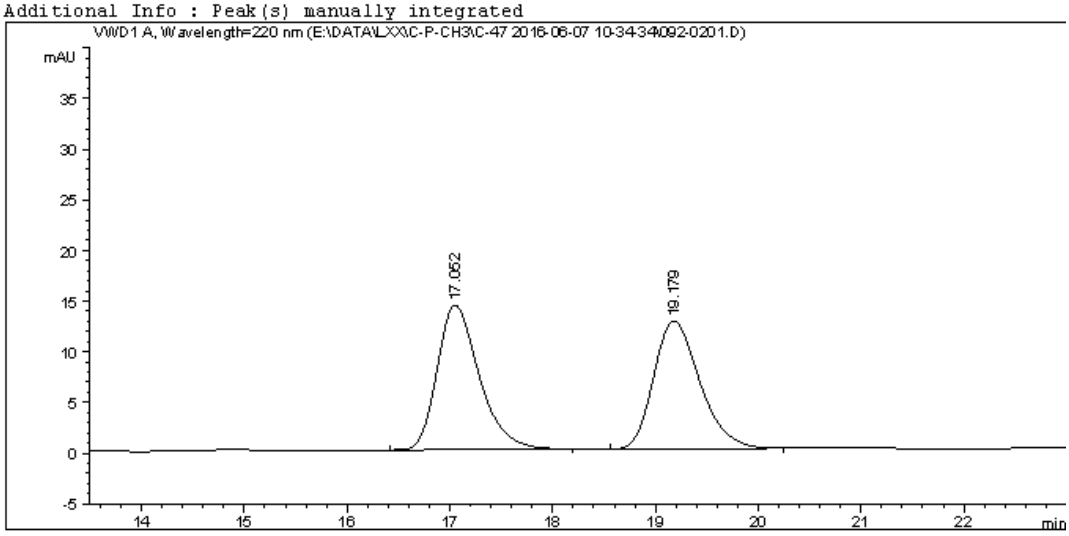

```
==========================================================================
                          Area Percent Report
==========================================================================

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  17.424 MM    0.5045 2600.18091   85.90569  99.8481
   2  18.900 MM    0.3078    3.95578 2.14172e-1   0.1519

Totals :                  2604.13669   86.11986

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



2b

Data File E:\DATA\LXX\C-P-CH3\C-47 2016-06-07 10-34-34\092-0201.D
Sample Name: p-Me-Rac

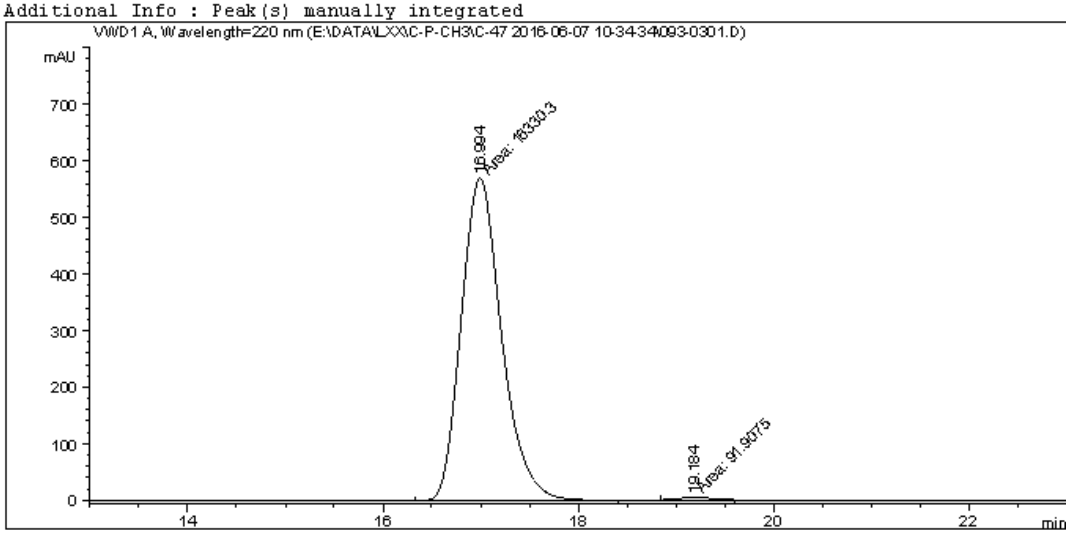
```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   2
    Acq. Instrument : 1260HPLC-VWD                   Location : Vial 92
    Injection Date  : 6/7/2016 10:46:05 AM                Inj :   1
                                                   Inj Volume : 3.000 µl
    Acq. Method     : E:\DATA\LXX\C-P-CH3\C-47 2016-06-07 10-34-34\VWD-AD(1-6)-95-5-1ML-3UL-
                      50MIN-220NM-.M
    Last changed    : 6/7/2016 10:34:34 AM by SYSTEM
    Analysis Method : E:\DATA\LXX\C-P-CH3\C-47 2016-06-07 10-34-34\VWD-AD(1-6)-95-5-1ML-3UL-
                      50MIN-220NM-.M (Sequence Method)
    Last changed    : 6/7/2016 12:46:44 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```



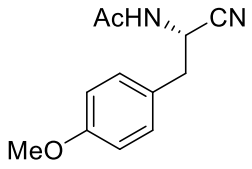

```
    ======================================================================
                            Area Percent Report
    ======================================================================

    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  17.052 BB    0.4346  405.46860   14.26752  50.7149
       2  19.179 BB    0.4789  394.03778   12.63250  49.2851

    Totals :                   799.50638   26.90002


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

Data File E:\DATA\LXX\C-P-CH3\C-47 2016-06-07 10-34-34\093-0301.D
Sample Name: c-47

```
==================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   3
Acq. Instrument : 1260HPLC-VWD                   Location : Vial 93
Injection Date  : 6/7/2016 11:36:49 AM                Inj :   1
                                                Inj Volume : 3.000 µl
Acq. Method     : E:\DATA\LXX\C-P-CH3\C-47 2016-06-07 10-34-34\VWD-AD(1-6)-95-5-1ML-3UL-
                  50MIN-220NM-.M
Last changed    : 6/7/2016 10:34:34 AM by SYSTEM
Analysis Method : E:\DATA\LXX\C-P-CH3\C-47 2016-06-07 10-34-34\VWD-AD(1-6)-95-5-1ML-3UL-
                  50MIN-220NM-.M (Sequence Method)
Last changed    : 6/7/2016 12:44:42 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



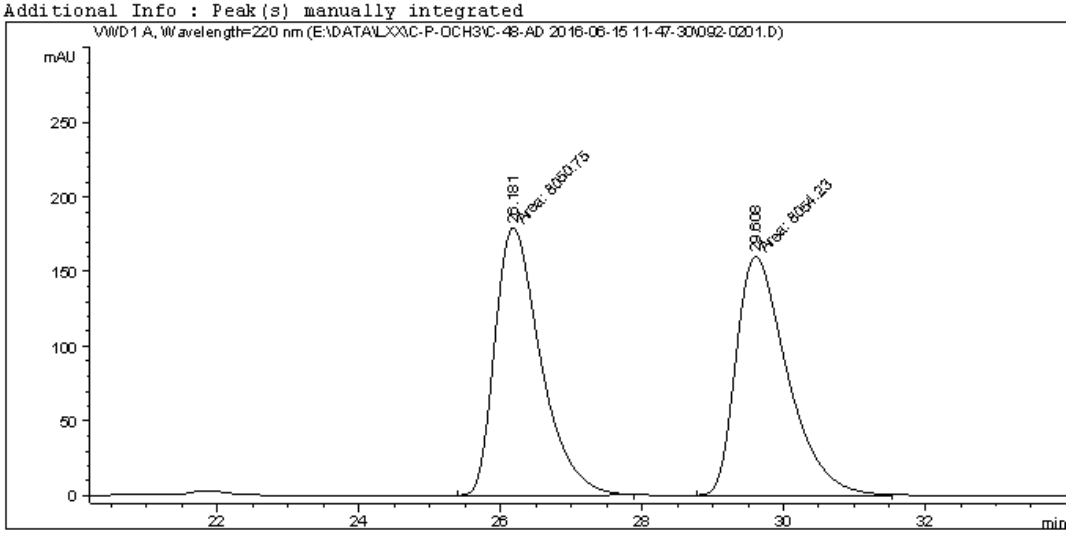

```
==================================================================
                        Area Percent Report
==================================================================

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


Signal 1: VWD1 A, Wavelength=220 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  16.994 MM    0.4782 1.63303e4   569.18378  99.4403
   2  19.184 MM    0.4122   91.90754    3.71598   0.5597

Totals :                  1.64222e4   572.89976

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



2c

Data File E:\DATA\LXX\C-P-OCH3\C-48-AD 2016-06-15 11-47-30\092-0201.D
Sample Name: C-48-1-Rac

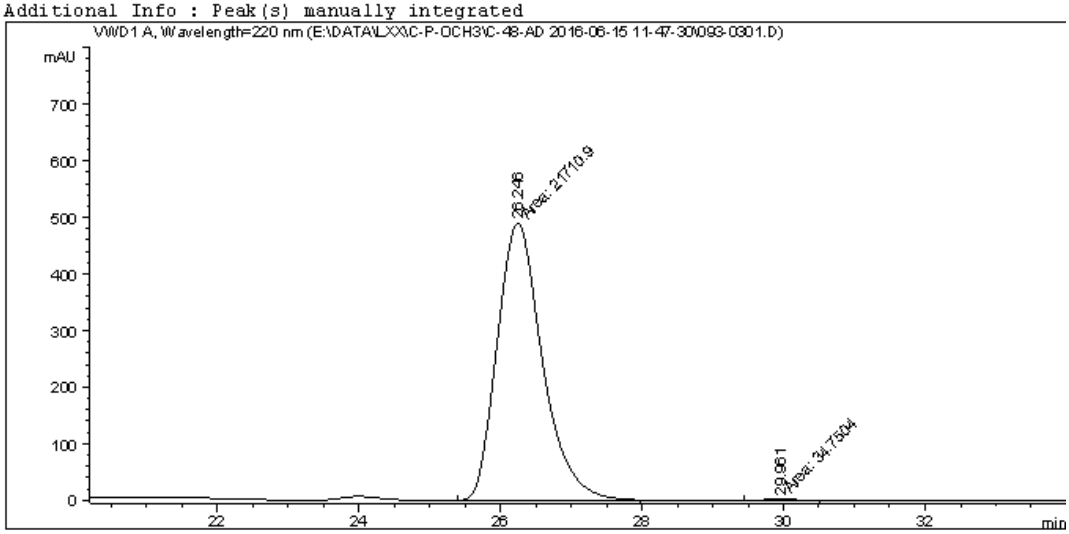
```
    ======================================================================
    Acq. Operator   : SYSTEM                          Seq. Line :   2
    Acq. Instrument : 1260HPLC-VWD                     Location : Vial 92
    Injection Date  : 6/15/2016 11:59:04 AM                 Inj :   1
                                                    Inj Volume : 3.000 µl
    Acq. Method     : E:\DATA\LXX\C-P-OCH3\C-48-AD 2016-06-15 11-47-30\VWD-AD(1-6)-95-5-1ML-
                      3UL-220-40MIN.M
    Last changed    : 6/15/2016 11:47:30 AM by SYSTEM
    Analysis Method : E:\DATA\LXX\C-P-OCH3\C-48-AD 2016-06-15 11-47-30\VWD-AD(1-6)-95-5-1ML-
                      3UL-220-40MIN.M (Sequence Method)
    Last changed    : 6/15/2016 1:38:53 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```



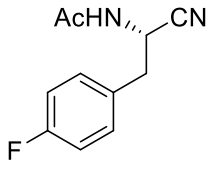

```
    ======================================================================
                              Area Percent Report
    ======================================================================

    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  26.181 MM    0.7486 8050.75098  179.23079  49.9892
       2  29.608 MM    0.8399 8054.23486  159.83328  50.0108

    Totals :                  1.61050e4   339.06407


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

Data File E:\DATA\LXX\C-P-OCH3\C-48-AD 2016-06-15 11-47-30\093-0301.D
Sample Name: C-48-2

```
=====================================================================
Acq. Operator   : SYSTEM                          Seq. Line :   3
Acq. Instrument : 1260HPLC-VWD                     Location : Vial 93
Injection Date  : 6/15/2016 12:39:48 PM                 Inj :   1
                                                  Inj Volume : 3.000 µl
Acq. Method     : E:\DATA\LXX\C-P-OCH3\C-48-AD 2016-06-15 11-47-30\VWD-AD(1-6)-95-5-1ML-
                  3UL-220-40MIN.M
Last changed    : 6/15/2016 11:47:30 AM by SYSTEM
Analysis Method : E:\DATA\LXX\C-P-OCH3\C-48-AD 2016-06-15 11-47-30\VWD-AD(1-6)-95-5-1ML-
                  3UL-220-40MIN.M (Sequence Method)
Last changed    : 6/15/2016 1:40:04 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



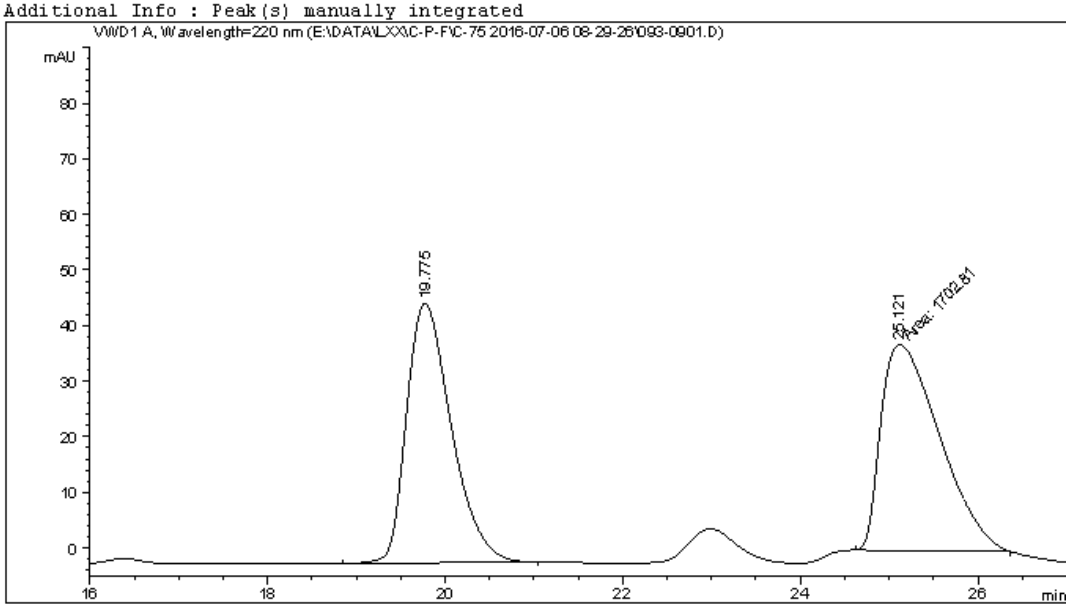

```
=====================================================================
                        Area Percent Report
=====================================================================

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  26.246 MM    0.7392 2.17109e4   489.53763  99.8402
   2  29.961 MM    0.5834   34.75039 9.92680e-1   0.1598

Totals :                  2.17456e4   490.53031

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



2d

Data File E:\DATA\LXX\C-P-F\C-75 2016-07-06 08-29-26\093-0901.D
Sample Name: p-F-Rac

Acq. Operator	: SYSTEM
Seq. Line	: 9
Acq. Instrument	: 1260HPLC-VWD
Location	: Vial 93
Injection Date	: 7/6/2016 1:09:32 PM
Inj	: 1
	Inj Volume : 5.000 µl
Acq. Method	: E:\DATA\LXX\C-P-F\C-75 2016-07-06 08-29-26\VWD-AD(1-6)-95-5-1ML-5UL-220NM-40MIN.M
Last changed	: 7/6/2016 1:01:40 PM by SYSTEM
Analysis Method	: E:\DATA\LXX\C-P-F\C-75 2016-07-06 08-29-26\VWD-AD(1-6)-95-5-1ML-5UL-220NM-40MIN.M (Sequence Method)
Last changed	: 7/6/2016 9:05:20 PM by SYSTEM (modified after loading)
Additional Info	: Peak(s) manually integrated



Area Percent Report

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

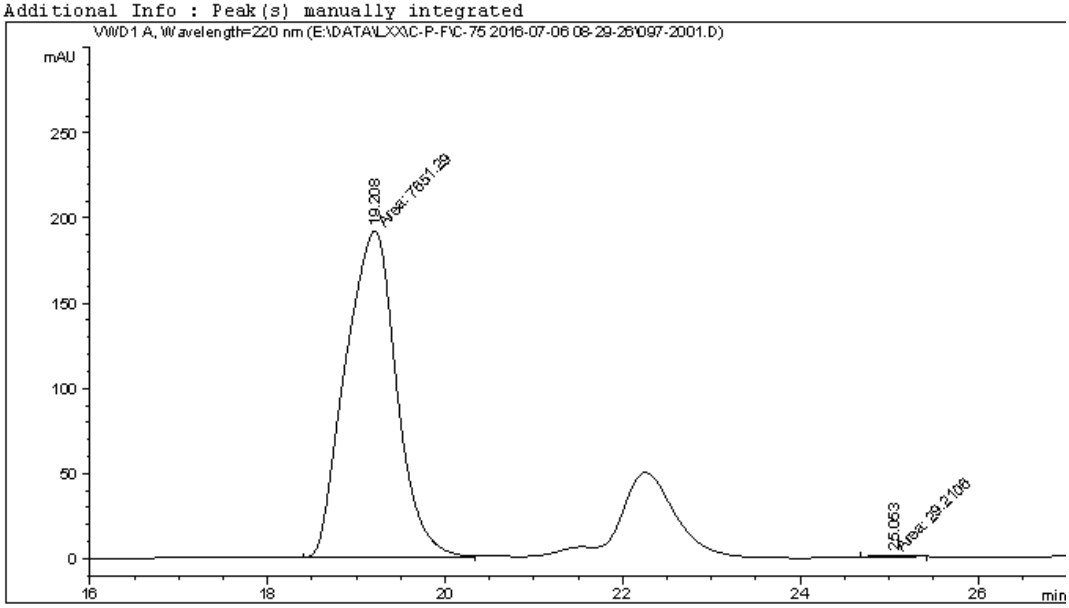
Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.775	BB	0.5414	1634.88477	46.70587	48.9824
2	25.121	MM	0.7655	1702.81262	37.07634	51.0176
Totals :				3337.69739	83.78221	

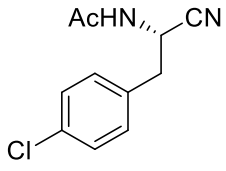
*** End of Report ***

Data File E:\DATA\LXX\C-P-F\C-75 2016-07-06 08-29-26\097-2001.D
Sample Name: C-75-3

```
    ======================================================================
    Acq. Operator   : SYSTEM                          Seq. Line :  20
    Acq. Instrument : 1260HPLC-VWD                     Location : Vial 97
    Injection Date  : 7/6/2016 8:23:38 PM                   Inj :  1
                                                    Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\LXX\C-P-F\C-75 2016-07-06 08-29-26\VWD-AD(1-6)-95-5-1ML-5UL-
                      220NM-40MIN.M
    Last changed    : 7/6/2016 1:01:40 PM by SYSTEM
    Analysis Method : E:\DATA\LXX\C-P-F\C-75 2016-07-06 08-29-26\VWD-AD(1-6)-95-5-1ML-5UL-
                      220NM-40MIN.M (Sequence Method)
    Last changed    : 7/6/2016 9:34:06 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```



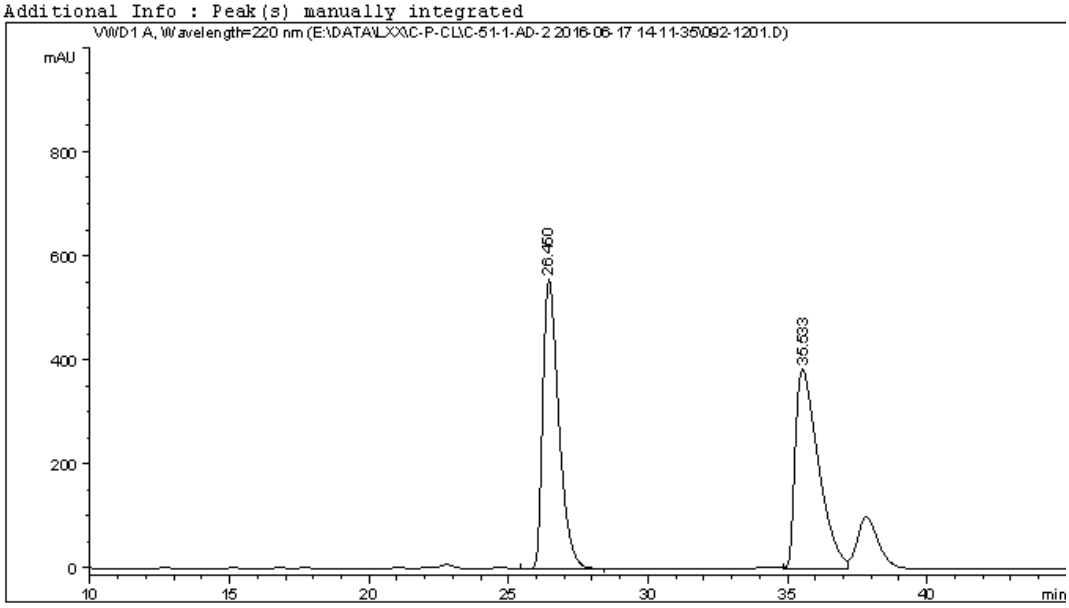

```
    ======================================================================
                              Area Percent Report
    ======================================================================

    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  19.208 MM    0.6645 7651.29248  191.89857  99.6197
       2  25.053 MM    0.4620   29.21063    1.05372   0.3803

    Totals :                  7680.50311  192.95229


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



2e

Data File E:\DATA\LXX\C-P-CL\C-51-1-AD-2 2016-06-17 14-11-35\092-1201.D
Sample Name: C-51-1

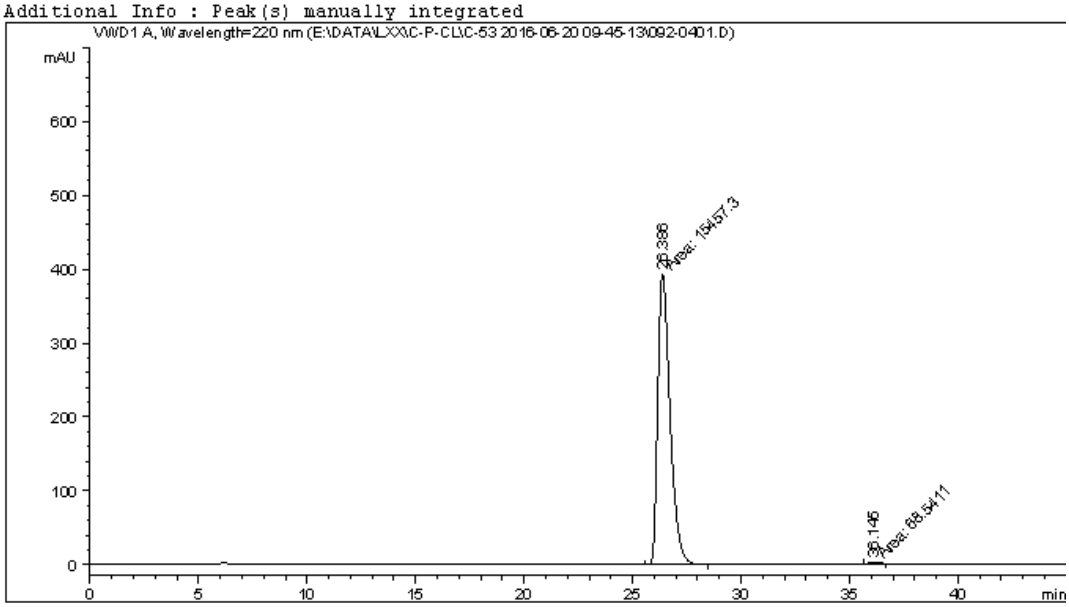
```
    ======================================================================
    Acq. Operator   : SYSTEM                          Seq. Line :  12
    Acq. Instrument : 1260HPLC-VWD                     Location : Vial 92
    Injection Date  : 6/17/2016 8:39:19 PM                  Inj :  1
                                                    Inj Volume : 3.000 µl
    Acq. Method     : E:\DATA\LXX\C-P-CL\C-51-1-AD-2 2016-06-17 14-11-35\VWD-AD(1-6)-93-7-0.
                      5ML-3UL-220-60MIN.M
    Last changed    : 6/17/2016 9:39:09 PM by SYSTEM
                      (modified after loading)
    Analysis Method : E:\DATA\LXX\C-P-CL\C-51-1-AD-2 2016-06-17 14-11-35\VWD-AD(1-6)-93-7-0.
                      5ML-3UL-220-60MIN.M (Sequence Method)
    Last changed    : 6/17/2016 9:51:30 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```



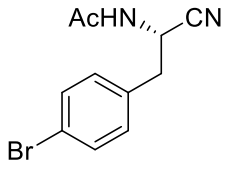

```
    ======================================================================
                           Area Percent Report
    ======================================================================

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


    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  26.450 BB    0.6113 2.20937e4   555.21405  49.9703
       2  35.533 VV    0.8777 2.21199e4   383.03159  50.0297

    Totals :                  4.42136e4   938.24564

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

Data File E:\DATA\LXX\C-P-CL\C-53 2016-06-20 09-45-13\092-0401.D
Sample Name: C-53

```
    ======================================================================
    Acq. Operator   : SYSTEM                          Seq. Line :   4
    Acq. Instrument : 1260HPLC-VWD                     Location : Vial 92
    Injection Date  : 6/20/2016 11:28:22 AM                 Inj :   1
                                                     Inj Volume : 3.000 µl
    Acq. Method     : E:\DATA\LXX\C-P-CL\C-53 2016-06-20 09-45-13\VWD-AD(1-6)-93-7-0.5ML-3UL-
                      220-60MIN.M
    Last changed    : 6/20/2016 11:32:12 AM by SYSTEM
                      (modified after loading)
    Analysis Method : E:\DATA\LXX\C-P-CL\C-53 2016-06-20 09-45-13\VWD-AD(1-6)-93-7-0.5ML-3UL-
                      220-60MIN.M (Sequence Method)
    Last changed    : 6/20/2016 12:34:52 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```



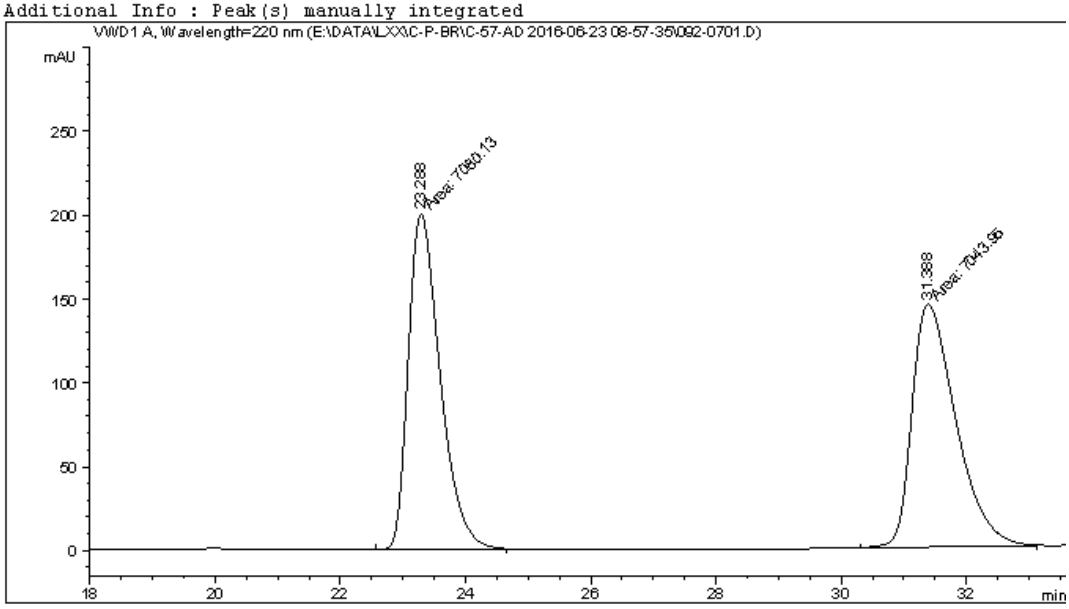

```
    ======================================================================
                              Area Percent Report
    ======================================================================

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


    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  26.386 MM    0.6558 1.54573e4   392.85995  99.5585
       2  36.145 MM    0.5956   68.54111    1.91814   0.4415

    Totals :                  1.55259e4   394.77809


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



2f

Data File E:\DATA\LXX\C-P-BR\C-57-AD 2016-06-23 08-57-35\092-0701.D
Sample Name: C-57-1-Rac

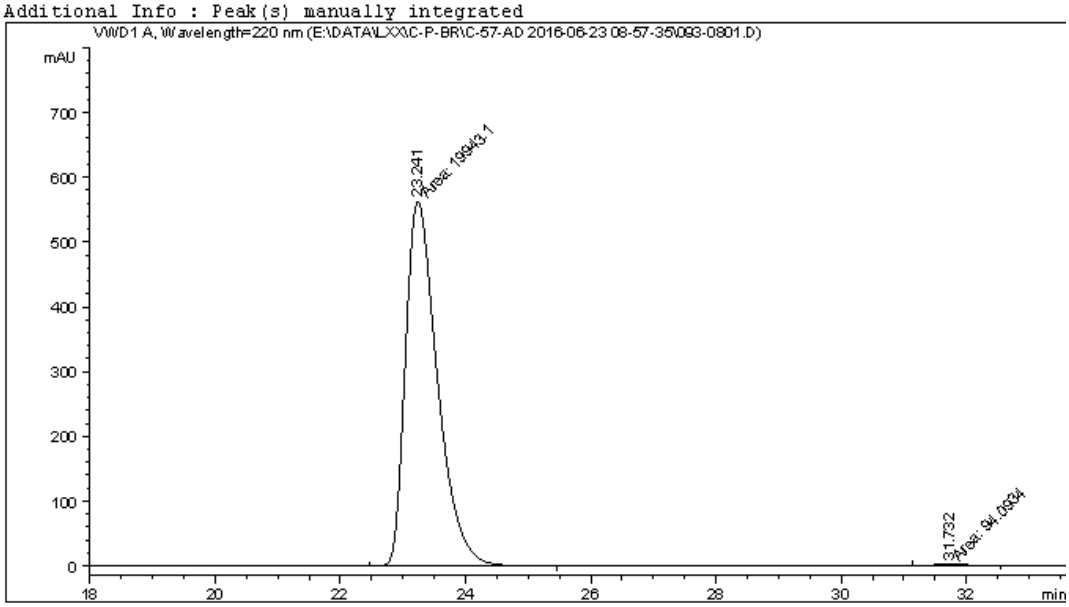
```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   7
    Acq. Instrument : 1260HPLC-VWD                   Location : Vial 92
    Injection Date  : 6/23/2016 12:03:52 PM              Inj :   1
                                                   Inj Volume : 3.000 µl
    Acq. Method     : E:\DATA\LXX\C-P-BR\C-57-AD 2016-06-23 08-57-35\VWD-AD(1-6)-93-7-0.6ML-
                      3UL-220-60MIN.M
    Last changed    : 6/23/2016 11:30:34 AM by SYSTEM
    Analysis Method : E:\DATA\LXX\C-P-BR\C-57-AD 2016-06-23 08-57-35\VWD-AD(1-6)-93-7-0.6ML-
                      3UL-220-60MIN.M (Sequence Method)
    Last changed    : 6/23/2016 1:48:40 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```



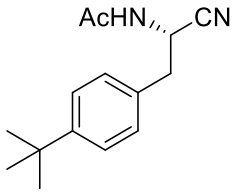

```
    ======================================================================
                              Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    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  23.288 MM    0.5897 7080.12891  200.10556  50.1281
       2  31.388 MM    0.8100 7043.94824  144.94562  49.8719

    Totals :                  1.4124le4   345.05118

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

Data File E:\DATA\LXX\C-P-BR\C-57-AD 2016-06-23 08-57-35\093-0801.D
Sample Name: C-57-2

```
    ======================================================================
    Acq. Operator   : SYSTEM                          Seq. Line :   8
    Acq. Instrument : 1260HPLC-VWD                     Location : Vial 93
    Injection Date  : 6/23/2016 1:04:36 PM                 Inj :   1
                                                     Inj Volume : 3.000 µl
    Acq. Method     : E:\DATA\LXX\C-P-BR\C-57-AD 2016-06-23 08-57-35\VWD-AD(1-6)-93-7-0.6ML-
                      3UL-220-60MIN.M
    Last changed    : 6/23/2016 1:43:06 PM by SYSTEM
                      (modified after loading)
    Analysis Method : E:\DATA\LXX\C-P-BR\C-57-AD 2016-06-23 08-57-35\VWD-AD(1-6)-93-7-0.6ML-
                      3UL-220-60MIN.M (Sequence Method)
    Last changed    : 6/23/2016 1:50:44 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```



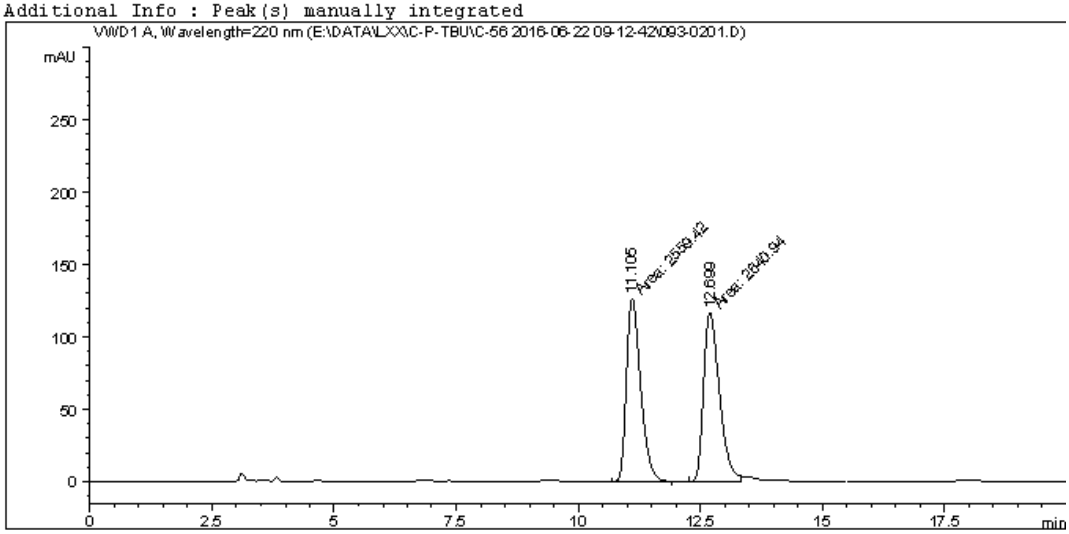

```
    ======================================================================
                              Area Percent Report
    ======================================================================

    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  23.241 MM    0.5906 1.99431e4   562.81189  99.5304
       2  31.732 MM    0.7049   94.09338    2.22470   0.4696

    Totals :                  2.00372e4   565.03659


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



2g

Data File E:\DATA\LXX\C-P-TBU\C-56 2016-06-22 09-12-42\093-0201.D
Sample Name: C-54-1-Rac

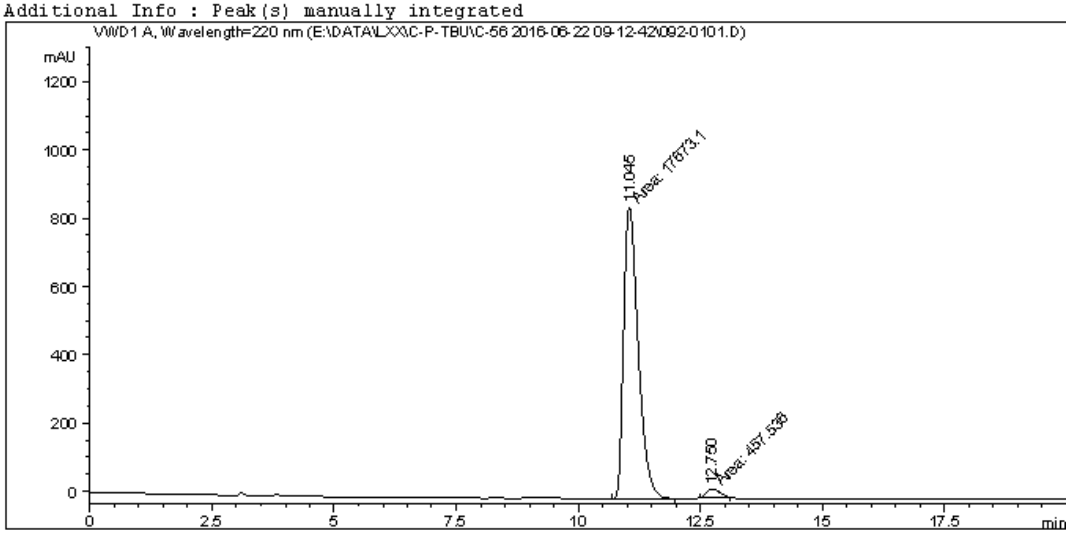
```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   2
    Acq. Instrument : 1260HPLC-VWD                   Location : Vial 93
    Injection Date  : 6/22/2016 9:55:11 AM                Inj :   1
                                                  Inj Volume : 3.000 µl
    Acq. Method     : E:\DATA\LXX\C-P-TBU\C-56 2016-06-22 09-12-42\VWD-AD(1-6)-95-5-1ML-3UL-
                      220-40MIN.M
    Last changed    : 6/22/2016 9:12:42 AM by SYSTEM
    Analysis Method : E:\DATA\LXX\C-P-TBU\C-56 2016-06-22 09-12-42\VWD-AD(1-6)-95-5-1ML-3UL-
                      220-40MIN.M (Sequence Method)
    Last changed    : 6/22/2016 10:40:04 AM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```



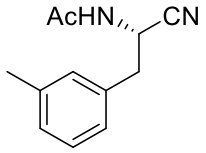

```
    ======================================================================
                            Area Percent Report
    ======================================================================

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


    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]        %
    ----|-------|----|-------|----------|----------|--------|
       1  11.105 MM    0.3374 2559.41797  126.42804  49.2161
       2  12.699 MF    0.3778 2640.94482  116.50204  50.7839

    Totals :                  5200.36279  242.93008


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

Data File E:\DATA\LXX\C-P-TBU\C-56 2016-06-22 09-12-42\092-0101.D
Sample Name: C-56

```
==================================================================
Acq. Operator   : SYSTEM                        Seq. Line :   1
Acq. Instrument : 1260HPLC-VWD                   Location : Vial 92
Injection Date  : 6/22/2016 9:14:27 AM               Inj :   1
                                                Inj Volume : 3.000 µl
Acq. Method     : E:\DATA\LXX\C-P-TBU\C-56 2016-06-22 09-12-42\VWD-AD(1-6)-95-5-1ML-3UL-
                  220-40MIN.M
Last changed    : 6/22/2016 9:12:42 AM by SYSTEM
Analysis Method : E:\DATA\LXX\C-P-TBU\C-56 2016-06-22 09-12-42\VWD-AD(1-6)-95-5-1ML-3UL-
                  220-40MIN.M (Sequence Method)
Last changed    : 6/22/2016 10:04:26 AM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



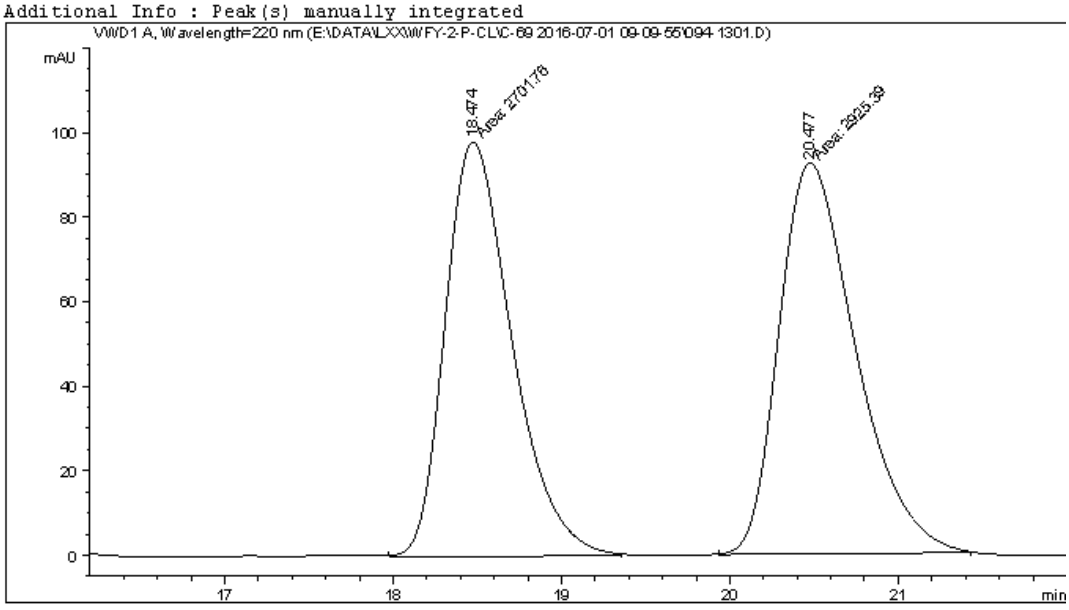

```
==================================================================
                        Area Percent Report
==================================================================

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  11.045 MM    0.3443 1.76731e4   855.56940  97.4765
   2  12.750 MM    0.3244  457.53632   23.50762   2.5235

Totals :                  1.81307e4   879.07701

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



2h

Data File E:\DATA\LXX\WFY-2-P-CL\C-69 2016-07-01 09-09-55\094-1301.D
Sample Name: m-CH3-Rac

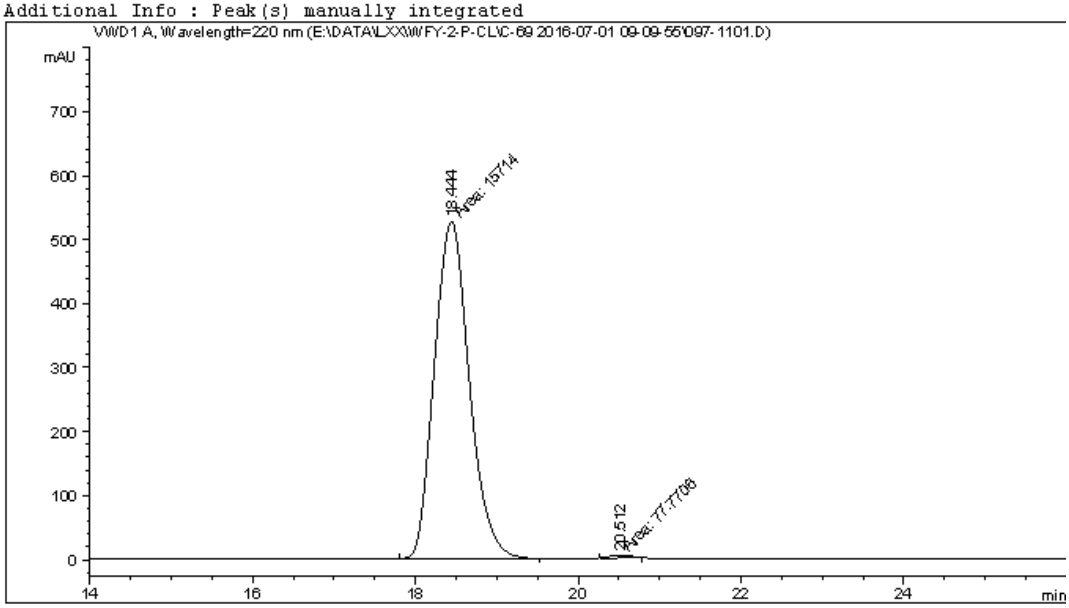
```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :  13
    Acq. Instrument : 1260HPLC-VWD                   Location : Vial 94
    Injection Date  : 7/1/2016 5:35:11 PM                 Inj :  1
                                                   Inj Volume : 3.000 µl
    Acq. Method     : E:\DATA\LXX\WFY-2-P-CL\C-69 2016-07-01 09-09-55\VWD-AD(1-6)-94-6-0.
                      6ML3UL-220-60MIN.M
    Last changed    : 7/1/2016 6:18:01 PM by SYSTEM
                      (modified after loading)
    Analysis Method : E:\DATA\LXX\WFY-2-P-CL\C-69 2016-07-01 09-09-55\VWD-AD(1-6)-94-6-0.
                      6ML3UL-220-60MIN.M (Sequence Method)
    Last changed    : 7/1/2016 7:27:34 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```



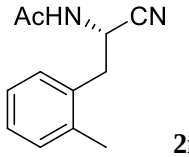

```
    ======================================================================
                              Area Percent Report
    ======================================================================

    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  18.474 MM    0.4593 2701.75952   98.04378  48.0129
       2  20.477 MM    0.5279 2925.39233   92.35935  51.9871

    Totals :                  5627.15186  190.40313


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

Data File E:\DATA\LXX\WFY-2-P-CL\C-69 2016-07-01 09-09-55\097-1101.D
Sample Name: C-69-3

```
    ======================================================================
    Acq. Operator   : SYSTEM                          Seq. Line :  11
    Acq. Instrument : 1260HPLC-VWD                     Location : Vial 97
    Injection Date  : 7/1/2016 4:18:33 PM                  Inj :  1
                                                     Inj Volume : 3.000 µl
    Acq. Method     : E:\DATA\LXX\WFY-2-P-CL\C-69 2016-07-01 09-09-55\VWD-AD(1-6)-94-6-0.
                      6ML3UL-220-60MIN.M
    Last changed    : 7/1/2016 12:14:50 PM by SYSTEM
    Analysis Method : E:\DATA\LXX\WFY-2-P-CL\C-69 2016-07-01 09-09-55\VWD-AD(1-6)-94-6-0.
                      6ML3UL-220-60MIN.M (Sequence Method)
    Last changed    : 7/1/2016 7:30:09 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```



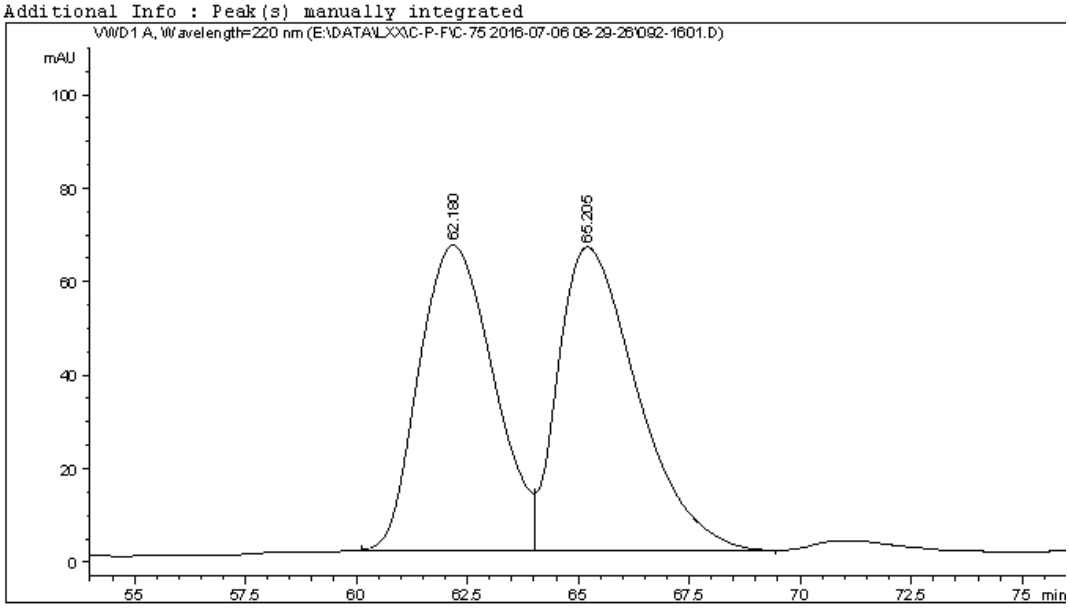

```
    ======================================================================
                              Area Percent Report
    ======================================================================

    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  18.444 MM    0.4971 1.57140e4   526.88458  99.5075
       2  20.512 MM    0.3311   77.77057    3.91464   0.4925

    Totals :                  1.57917e4   530.79923


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



2i

Data File E:\DATA\LXX\C-P-F\C-75 2016-07-06 08-29-26\092-1601.D
Sample Name: o-CH3-Rac

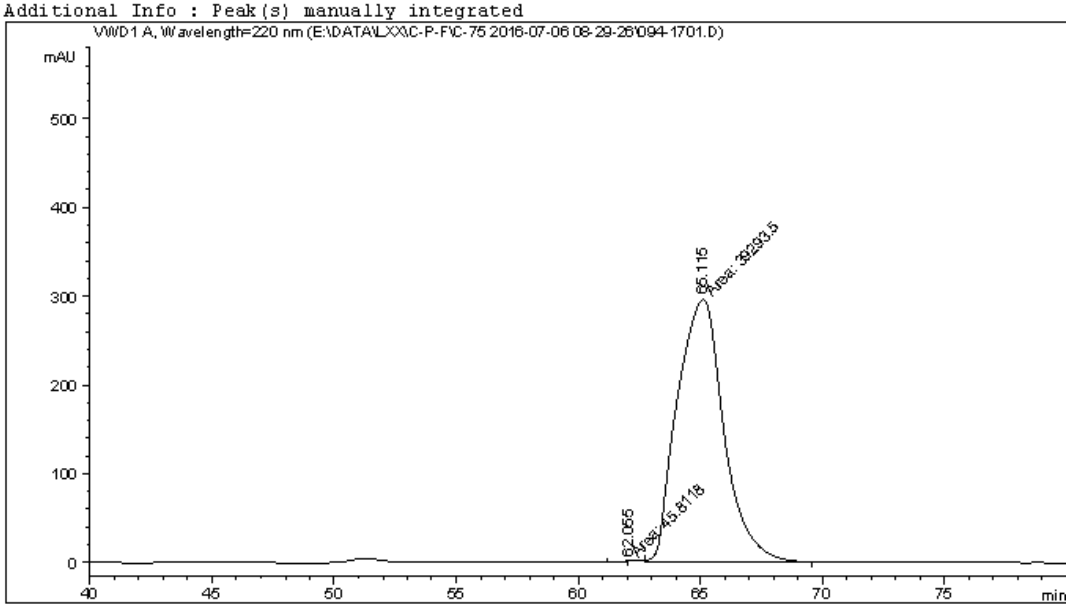
```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :  16
    Acq. Instrument : 1260HPLC-VWD                   Location : Vial 92
    Injection Date  : 7/6/2016 4:40:23 PM                 Inj :  1
                                                   Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\LXX\C-P-F\C-75 2016-07-06 08-29-26\VWD-AD(1-6)-97-3-0.5ML-5UL-
                      220NM-70MIN.M
    Last changed    : 7/6/2016 5:49:03 PM by SYSTEM
                      (modified after loading)
    Analysis Method : E:\DATA\LXX\C-P-F\C-75 2016-07-06 08-29-26\VWD-AD(1-6)-97-3-0.5ML-5UL-
                      220NM-70MIN.M (Sequence Method)
    Last changed    : 7/6/2016 7:38:58 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```



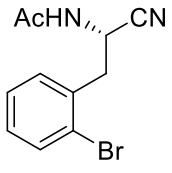

```
    ======================================================================
                              Area Percent Report
    ======================================================================

    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  62.180 BV    1.7259 7588.88672   65.24884  48.5403
       2  65.205 VB    1.8922 8045.31006   64.85965  51.4597

    Totals :                  1.56342e4   130.10849


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

Data File E:\DATA\LXX\C-P-F\C-75 2016-07-06 08-29-26\094-1701.D
Sample Name: C-75-4

```
    ======================================================================
    Acq. Operator   : SYSTEM                          Seq. Line :  17
    Acq. Instrument : 1260HPLC-VWD                     Location : Vial 94
    Injection Date  : 7/6/2016 6:01:09 PM                  Inj :  1
                                                     Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\LXX\C-P-F\C-75 2016-07-06 08-29-26\VWD-AD(1-6)-97-3-0.5ML-5UL-
                      220NM-70MIN.M
    Last changed    : 7/6/2016 5:49:03 PM by SYSTEM
    Analysis Method : E:\DATA\LXX\C-P-F\C-75 2016-07-06 08-29-26\VWD-AD(1-6)-97-3-0.5ML-5UL-
                      220NM-70MIN.M (Sequence Method)
    Last changed    : 7/6/2016 7:37:28 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```



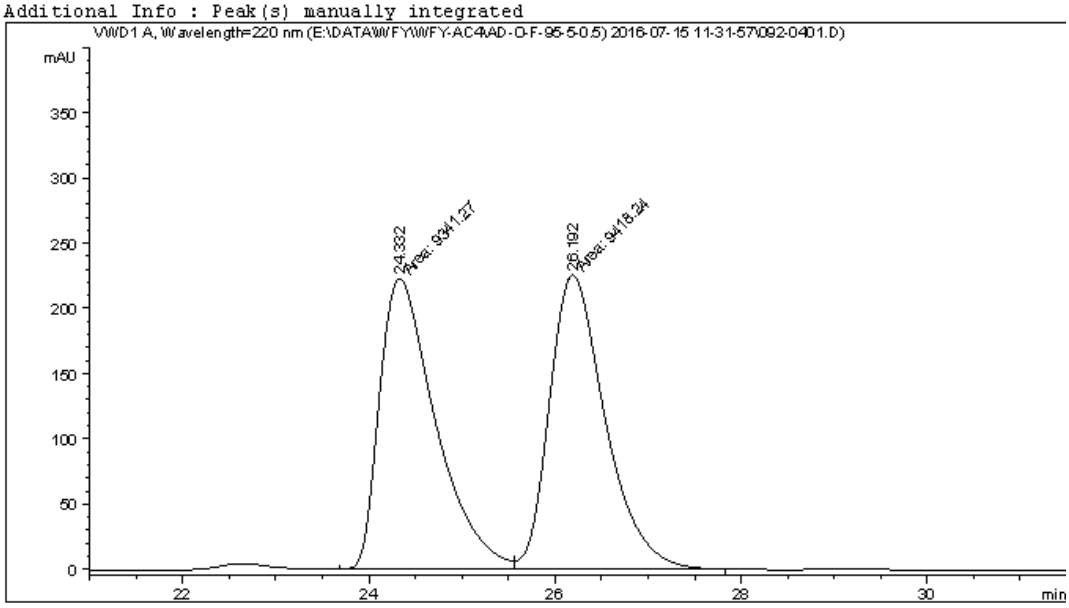

```
    ======================================================================
                              Area Percent Report
    ======================================================================

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


    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  62.055 MM    0.4912   45.81175    1.55428   0.1165
       2  65.115 FM    2.2110 3.92935e4   296.19366  99.8835

    Totals :                  3.93393e4   297.74794


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



2j

Data File E:\DATA\WFY\WFY-AC4\AD-0-F-95-5-0.5) 2016-07-15 11-31-57\092-0401.D
Sample Name: C-o-Br-Rac

Acq. Operator	: SYSTEM	Seq. Line	: 4
Acq. Instrument	: 1260HPLC-VWD	Location	: Vial 92
Injection Date	: 7/15/2016 1:16:52 PM	Inj	: 1
		Inj Volume	: 3.000 µl
Acq. Method	: E:\DATA\WFY\WFY-AC4\AD-0-F-95-5-0.5) 2016-07-15 11-31-57\VWD-AD(1-6)-95-5-0.8ML-3UL-220NM-35MIN.M		
Last changed	: 7/15/2016 1:54:31 PM by SYSTEM (modified after loading)		
Analysis Method	: E:\DATA\WFY\WFY-AC4\AD-0-F-95-5-0.5) 2016-07-15 11-31-57\VWD-AD(1-6)-95-5-0.8ML-3UL-220NM-35MIN.M (Sequence Method)		
Last changed	: 7/15/2016 2:38:59 PM by SYSTEM (modified after loading)		
Additional Info	: Peak(s) manually integrated		



Area Percent Report

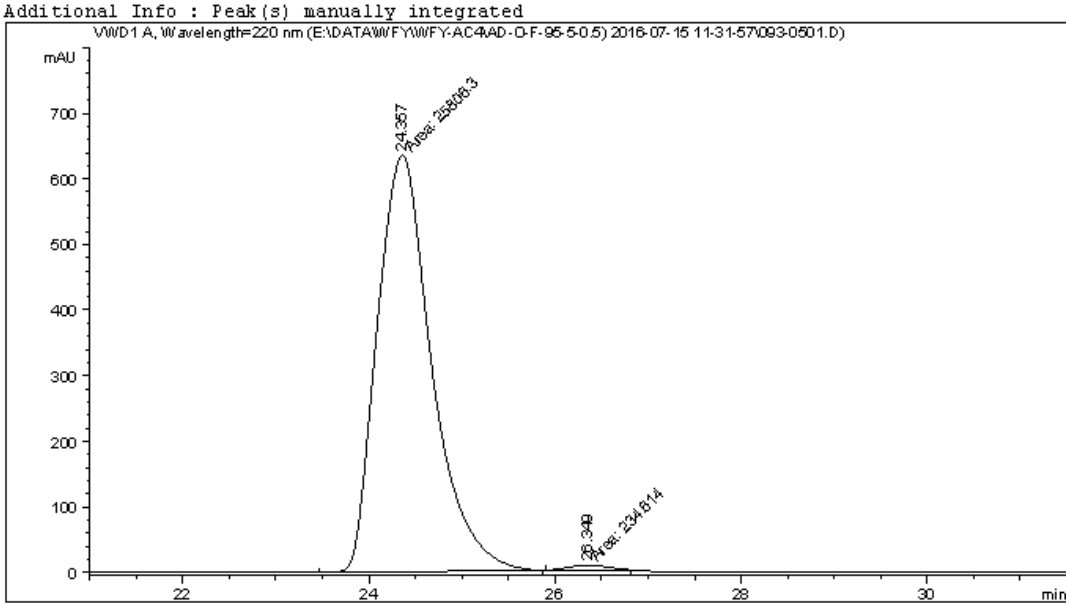
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=220 nm

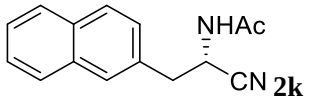
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	24.332	MF	0.6978	9341.26563	223.11790	49.7948
2	26.192	FM	0.6951	9418.24121	225.81535	50.2052
Totals :				1.87595e4	448.93326	

Data File E:\DATA\WFY\WFY-AC4\AD-0-F-95-5-0.5) 2016-07-15 11-31-57\093-0501.D
Sample Name: C-88-3

```
    ======================================================================
    Acq. Operator   : SYSTEM                          Seq. Line :   5
    Acq. Instrument : 1260HPLC-VWD                     Location : Vial 93
    Injection Date  : 7/15/2016 1:55:15 PM                  Inj :   1
                                                    Inj Volume : 3.000 µl
    Acq. Method     : E:\DATA\WFY\WFY-AC4\AD-0-F-95-5-0.5) 2016-07-15 11-31-57\VWD-AD(1-6)-95-
                      5-0.8ML-3UL-220NM-35MIN.M
    Last changed    : 7/15/2016 2:04:36 PM by SYSTEM
                      (modified after loading)
    Analysis Method : E:\DATA\WFY\WFY-AC4\AD-0-F-95-5-0.5) 2016-07-15 11-31-57\VWD-AD(1-6)-95-
                      5-0.8ML-3UL-220NM-35MIN.M (Sequence Method)
    Last changed    : 7/15/2016 2:42:09 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```



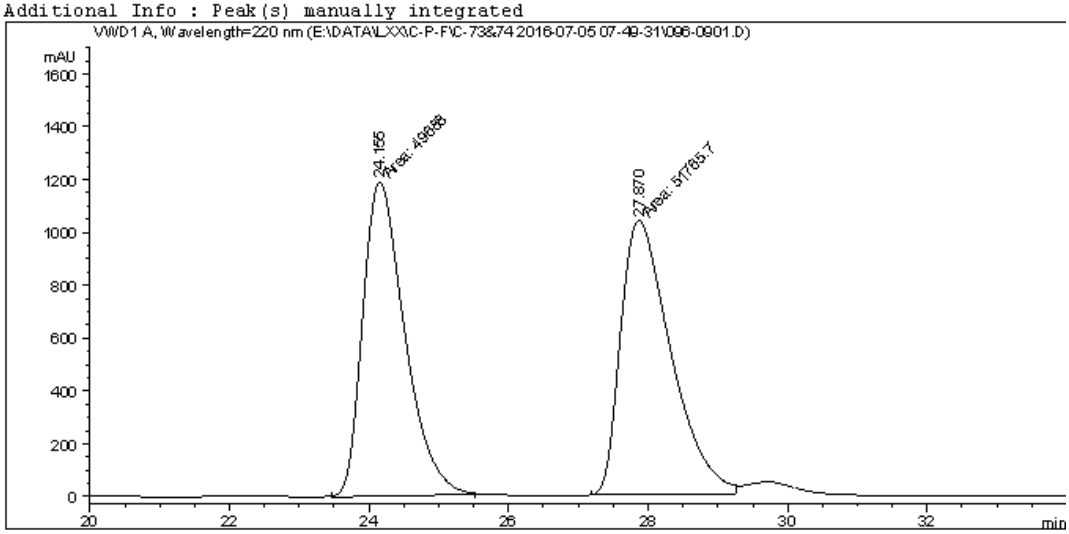

```
    ======================================================================
                              Area Percent Report
    ======================================================================

    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  24.357 MM    0.6774 2.58063e4   634.96436  99.0991
       2  26.349 MM    0.5011  234.61411    7.80281   0.9009

    Totals :                  2.60409e4   642.76717


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



2k

Data File E:\DATA\LXX\C-P-F\C-73&74 2016-07-05 07-49-31\096-0901.D
Sample Name: 2-NAI-Rac

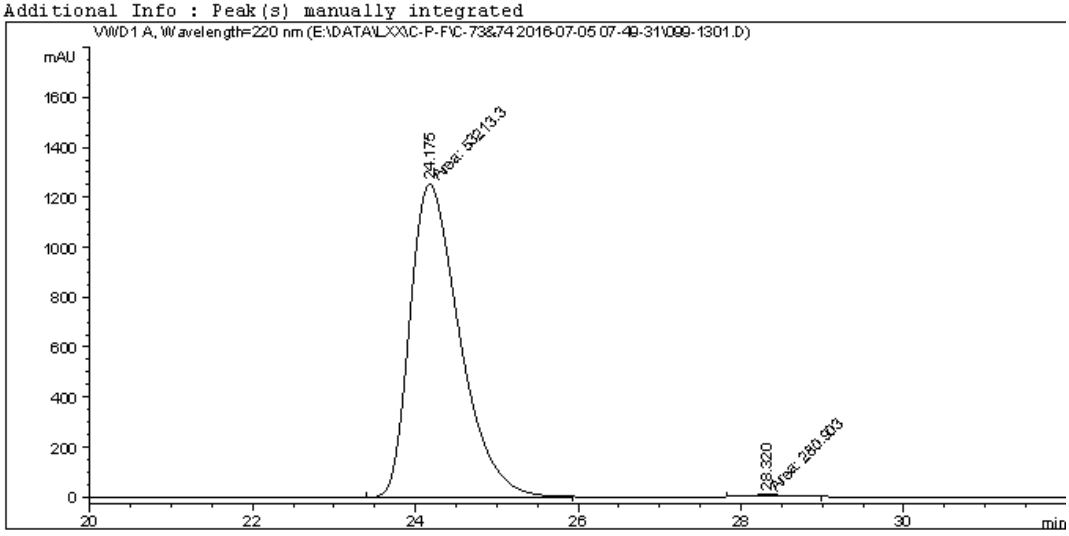
```
    ======================================================================
    Acq. Operator   : SYSTEM                          Seq. Line :   9
    Acq. Instrument : 1260HPLC-VWD                     Location : Vial 96
    Injection Date  : 7/5/2016 1:14:51 PM                  Inj :   1
                                                    Inj Volume : 3.000 µl
    Acq. Method     : E:\DATA\LXX\C-P-F\C-73&74 2016-07-05 07-49-31\VWD-AD(1-6)-95-5-1ML-3UL-
                      220-40MIN.M
    Last changed    : 7/5/2016 7:49:31 AM by SYSTEM
    Analysis Method : E:\DATA\LXX\C-P-F\C-73&74 2016-07-05 07-49-31\VWD-AD(1-6)-95-5-1ML-3UL-
                      220-40MIN.M (Sequence Method)
    Last changed    : 7/5/2016 2:46:13 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```



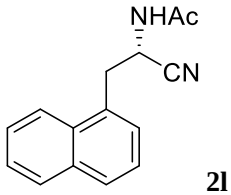

```
    ======================================================================
                            Area Percent Report
    ======================================================================

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


    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  24.155 MM    0.6965 4.96880e4  1189.06067  48.9760
       2  27.870 MM    0.8320 5.17657e4  1036.96191  51.0240

    Totals :                  1.01454e5  2226.02258


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

Data File E:\DATA\LXX\C-P-F\C-73&74 2016-07-05 07-49-31\099-1301.D
Sample Name: C-74-2

```
=====================================================================
Acq. Operator   : SYSTEM                          Seq. Line :  13
Acq. Instrument : 1260HPLC-VWD                     Location : Vial 99
Injection Date  : 7/5/2016 3:58:00 PM                   Inj :  1
                                                  Inj Volume : 3.000 µl
Acq. Method     : E:\DATA\LXX\C-P-F\C-73&74 2016-07-05 07-49-31\VWD-AD(1-6)-95-5-1ML-3UL-
                  220-40MIN.M
Last changed    : 7/5/2016 7:49:31 AM by SYSTEM
Analysis Method : E:\DATA\LXX\C-P-F\C-73&74 2016-07-05 07-49-31\VWD-AD(1-6)-95-5-1ML-3UL-
                  220-40MIN.M (Sequence Method)
Last changed    : 7/5/2016 4:43:51 PM by SYSTEM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```



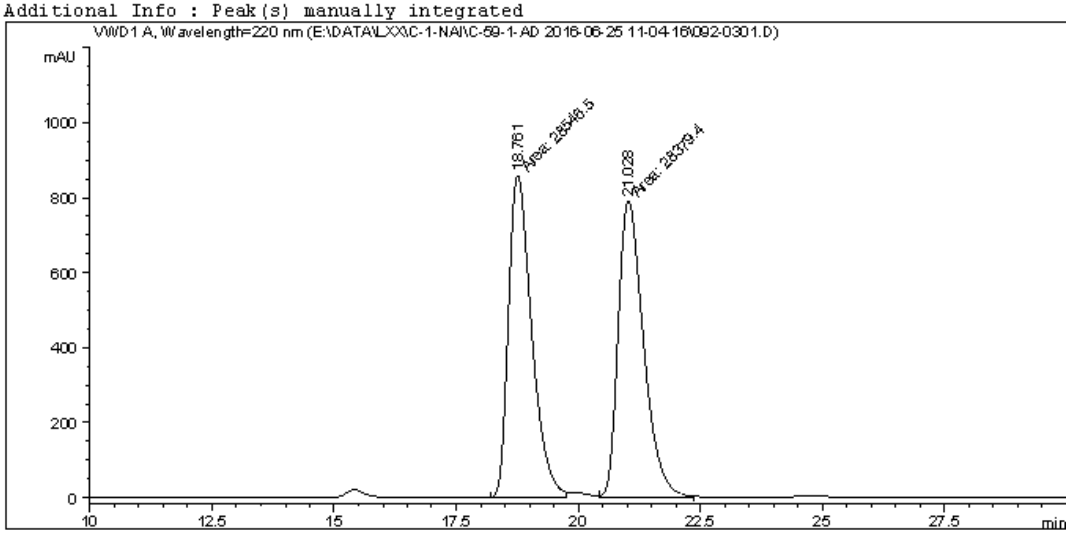

```
=====================================================================
                        Area Percent Report
=====================================================================

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


Signal 1: VWD1 A, Wavelength=220 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  24.175 MM    0.7068 5.32133e4  1254.77527  99.4749
   2  28.320 MM    0.6518  280.90329    7.18264   0.5251

Totals :                  5.34942e4  1261.95791

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



2l

Data File E:\DATA\LXX\C-1-NAI\C-59-1-AD 2016-06-25 11-04-16\092-0301.D
Sample Name: C-59-1

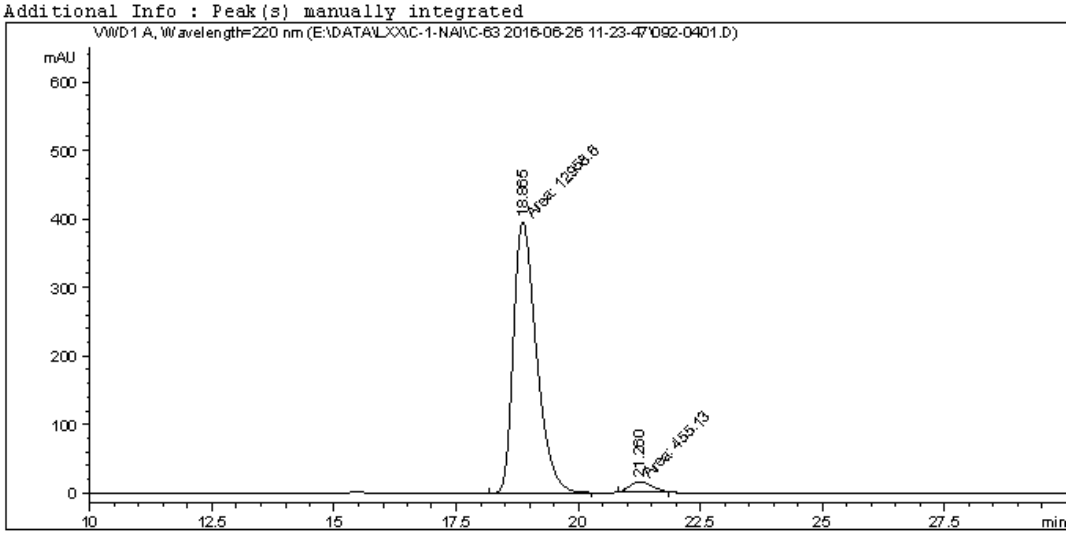
```
    ==================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   3
    Acq. Instrument : 1260HPLC-VWD                   Location : Vial 92
    Injection Date  : 6/25/2016 11:36:36 AM               Inj :   1
                                                  Inj Volume : 3.000 µl
    Acq. Method     : E:\DATA\LXX\C-1-NAI\C-59-1-AD 2016-06-25 11-04-16\VWD-AD(1-6)-95-5-1ML-
                      3UL-220-40MIN.M
    Last changed    : 6/25/2016 11:04:17 AM by SYSTEM
    Analysis Method : E:\DATA\LXX\C-1-NAI\C-59-1-AD 2016-06-25 11-04-16\VWD-AD(1-6)-95-5-1ML-
                      3UL-220-40MIN.M (Sequence Method)
    Last changed    : 6/25/2016 12:21:03 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```



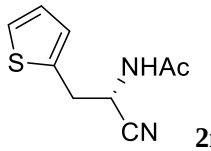

```
    ==================================================================
                          Area Percent Report
    ==================================================================

    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  18.761 MM    0.5531 2.85465e4   860.14276  50.1468
       2  21.028 MM    0.6000 2.83794e4   788.28717  49.8532

    Totals :                  5.69259e4  1648.42993


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

Data File E:\DATA\LXX\C-1-NAI\C-63 2016-06-26 11-23-47\092-0401.D
Sample Name: C-63-1

```
    ======================================================================
    Acq. Operator   : SYSTEM                          Seq. Line :   4
    Acq. Instrument : 1260HPLC-VWD                     Location : Vial 92
    Injection Date  : 6/26/2016 1:06:48 PM                 Inj :   1
                                                     Inj Volume : 3.000 µl
    Acq. Method     : E:\DATA\LXX\C-1-NAI\C-63 2016-06-26 11-23-47\VWD-AD(1-6)-95-5-1ML-3UL-
                      220-40MIN.M
    Last changed    : 6/26/2016 1:45:49 PM by SYSTEM
                      (modified after loading)
    Analysis Method : E:\DATA\LXX\C-1-NAI\C-63 2016-06-26 11-23-47\VWD-AD(1-6)-95-5-1ML-3UL-
                      220-40MIN.M (Sequence Method)
    Last changed    : 6/26/2016 1:55:44 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```



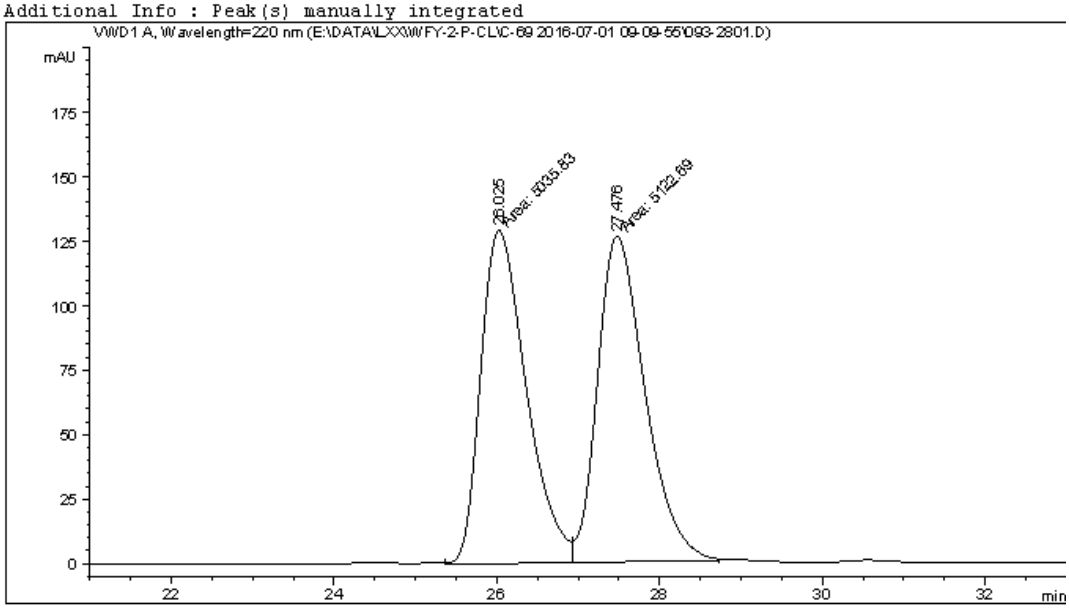

```
    ======================================================================
                              Area Percent Report
    ======================================================================

    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  18.865 MM    0.5455 1.29586e4   395.89621  96.6070
       2  21.260 MM    0.5098  455.12979   14.87798   3.3930

    Totals :                  1.34137e4   410.77419

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



2m

Data File E:\DATA\LXX\WFY-2-P-CL\C-69 2016-07-01 09-09-55\093-2801.D
Sample Name: Saifen-Rac

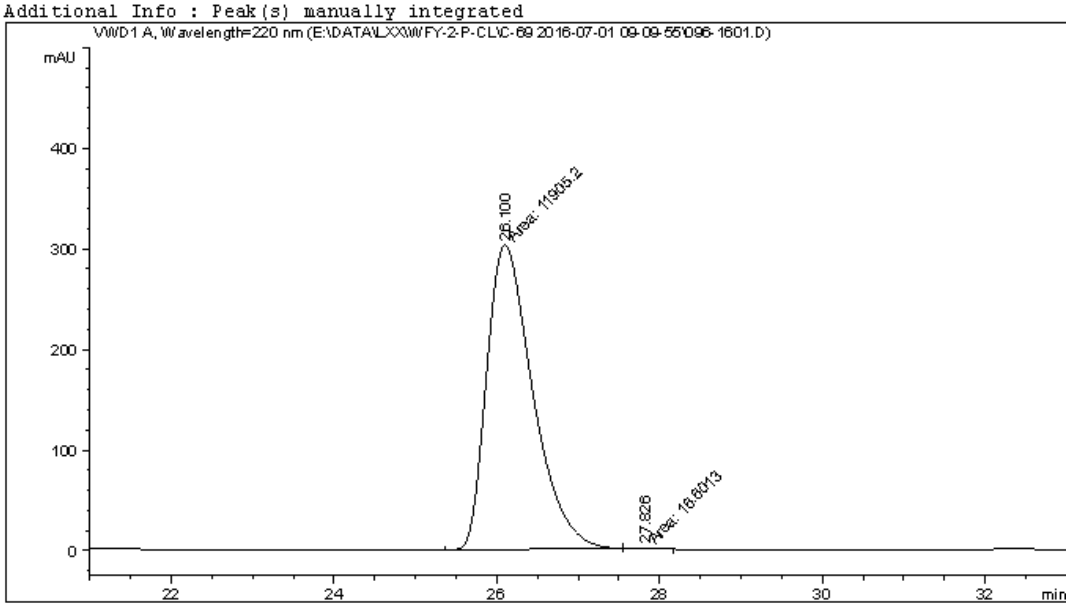
```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :  28
    Acq. Instrument : 1260HPLC-VWD                   Location : Vial 93
    Injection Date  : 7/2/2016 12:24:59 AM                Inj :  1
                                                   Inj Volume : 3.000 µl
    Acq. Method     : E:\DATA\LXX\WFY-2-P-CL\C-69 2016-07-01 09-09-55\VWD-AD(1-6)-94-6-0.
                      6ML3UL-220-60MIN.M
    Last changed    : 7/1/2016 12:14:50 PM by SYSTEM
    Analysis Method : E:\DATA\LXX\WFY-2-P-CL\C-69 2016-07-01 09-09-55\VWD-AD(1-6)-94-6-0.
                      6ML3UL-220-60MIN.M (Sequence Method)
    Last changed    : 7/2/2016 8:51:11 AM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```



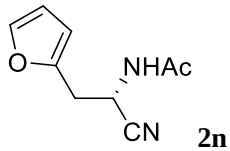

```
    ======================================================================
                              Area Percent Report
    ======================================================================

    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    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  26.025 MF    0.6494 5035.82959  129.25130  49.5725
       2  27.476 FM    0.6756 5122.69287  126.36832  50.4275

    Totals :                  1.01585e4   255.61961


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

1260HPLC-VWD 7/2/2016 8:52:05 AM SYSTEM                        Page 1 of 1
```

Data File E:\DATA\LXX\WFY-2-P-CL\C-69 2016-07-01 09-09-55\096-1601.D
Sample Name: C-69-2

```
    ======================================================================
    Acq. Operator   : SYSTEM                          Seq. Line :  16
    Acq. Instrument : 1260HPLC-VWD                     Location : Vial 96
    Injection Date  : 7/1/2016 7:35:23 PM                   Inj :  1
                                                     Inj Volume : 3.000 µl
    Acq. Method     : E:\DATA\LXX\WFY-2-P-CL\C-69 2016-07-01 09-09-55\VWD-AD(1-6)-94-6-0.
                      6ML3UL-220-60MIN.M
    Last changed    : 7/1/2016 7:36:30 PM by SYSTEM
                      (modified after loading)
    Analysis Method : E:\DATA\LXX\WFY-2-P-CL\C-69 2016-07-01 09-09-55\VWD-AD(1-6)-94-6-0.
                      6ML3UL-220-60MIN.M (Sequence Method)
    Last changed    : 7/2/2016 8:52:48 AM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```



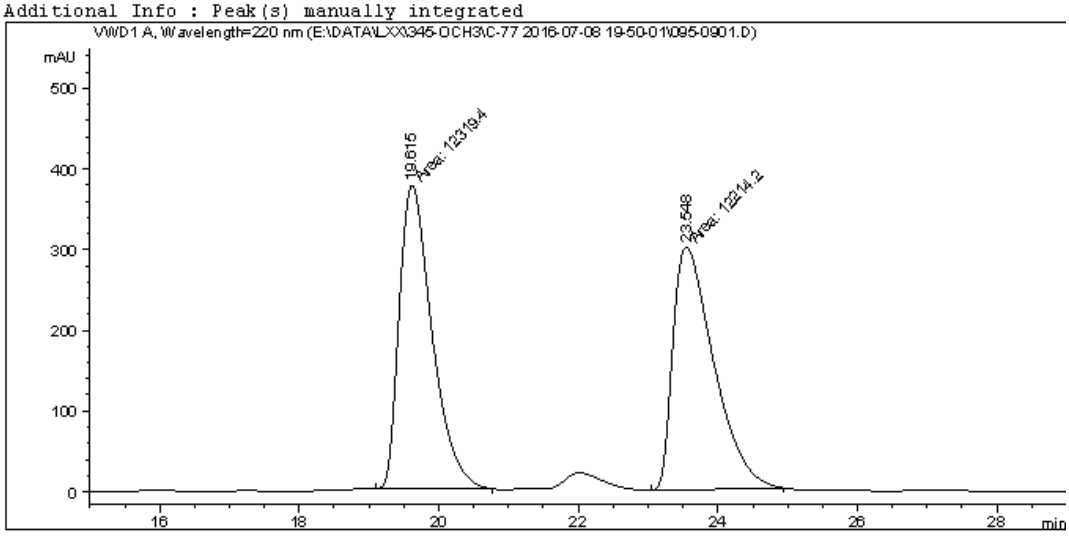

```
    ======================================================================
                              Area Percent Report
    ======================================================================

    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  26.100 MM    0.6554 1.19052e4   302.75143  99.8607
       2  27.826 MM    0.3748   16.60127 7.38190e-1   0.1393

    Totals :                  1.19218e4   303.48962


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



2n

Data File E:\DATA\LXX\345-OCH3\C-77 2016-07-08 19-50-01\095-0901.D
Sample Name: Funan-Rac

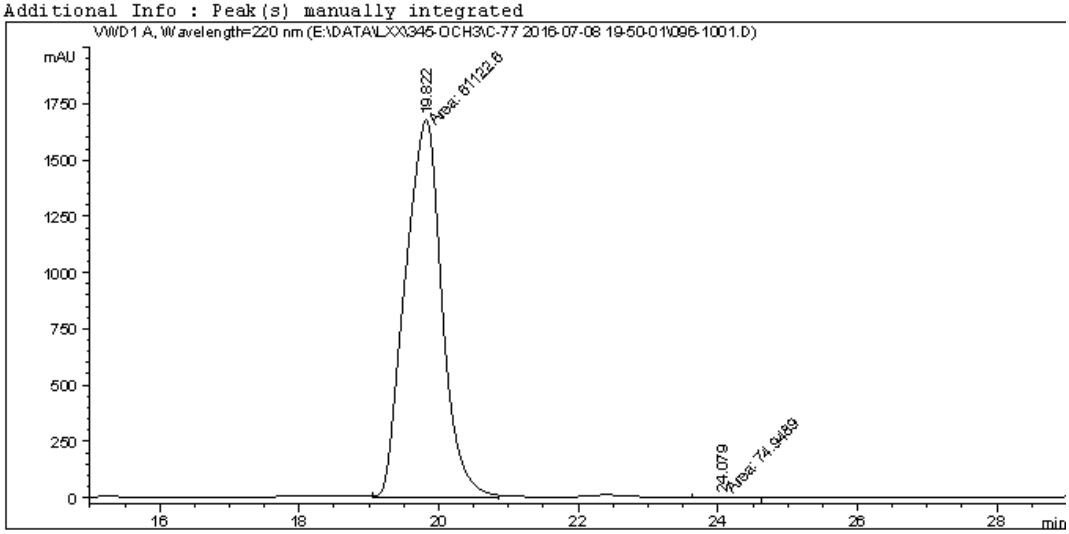
```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   9
    Acq. Instrument : 1260HPLC-VWD                   Location : Vial 95
    Injection Date  : 7/9/2016 12:12:21 AM                Inj :   1
                                                   Inj Volume : 3.000 µl
    Acq. Method     : E:\DATA\LXX\345-OCH3\C-77 2016-07-08 19-50-01\VWD-AD(1-6)-95-5-1ML-3UL-
                      220-40MIN.M
    Last changed    : 7/8/2016 7:50:02 PM by SYSTEM
    Analysis Method : E:\DATA\LXX\345-OCH3\C-77 2016-07-08 19-50-01\VWD-AD(1-6)-95-5-1ML-3UL-
                      220-40MIN.M (Sequence Method)
    Last changed    : 7/9/2016 8:20:04 AM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```



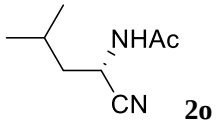

```
    ======================================================================
                            Area Percent Report
    ======================================================================

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


    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  19.615 MM    0.5457 1.23194e4   376.28650  50.2144
       2  23.548 MM    0.6764 1.22142e4   300.93906  49.7856

    Totals :                  2.45335e4   677.22556

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

Data File E:\DATA\LXX\345-OCH3\C-77 2016-07-08 19-50-01\096-1001.D
Sample Name: C-77-3

```
    ======================================================================
    Acq. Operator   : SYSTEM                          Seq. Line :  10
    Acq. Instrument : 1260HPLC-VWD                     Location : Vial 96
    Injection Date  : 7/9/2016 12:53:07 AM                  Inj :  1
                                                     Inj Volume : 3.000 µl
    Acq. Method     : E:\DATA\LXX\345-OCH3\C-77 2016-07-08 19-50-01\VWD-AD(1-6)-95-5-1ML-3UL-
                      220-40MIN.M
    Last changed    : 7/8/2016 7:50:02 PM by SYSTEM
    Analysis Method : E:\DATA\LXX\345-OCH3\C-77 2016-07-08 19-50-01\VWD-AD(1-6)-95-5-1ML-3UL-
                      220-40MIN.M (Sequence Method)
    Last changed    : 7/9/2016 8:18:38 AM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```



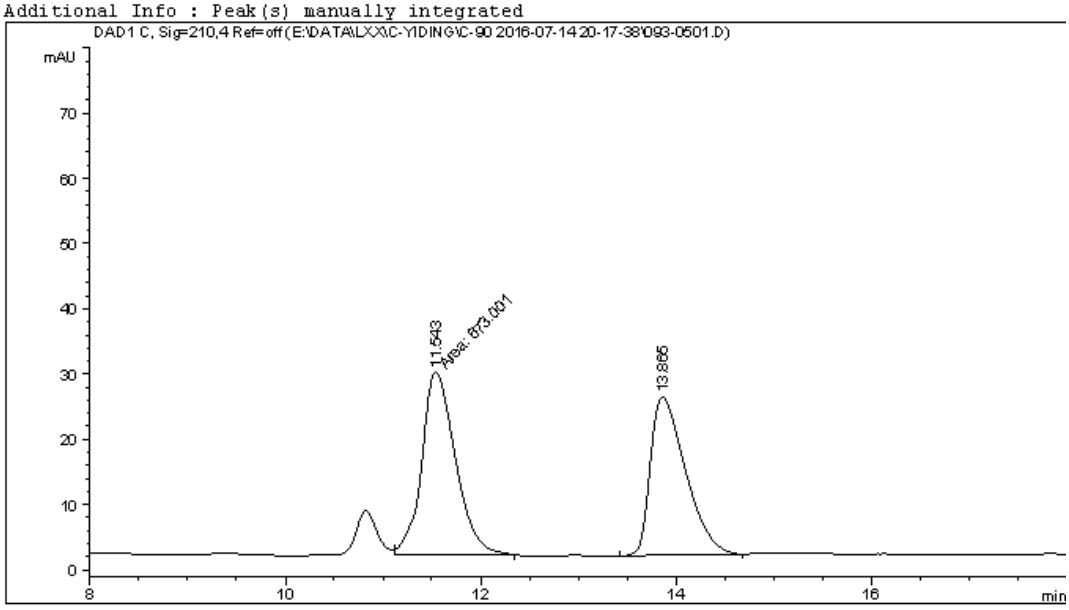

```
    ======================================================================
                            Area Percent Report
    ======================================================================

    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  19.822 MM    0.6077 6.11226e4  1676.25879  99.8775
       2  24.079 MM    0.5243   74.94890    2.38232   0.1225

    Totals :                  6.11975e4  1678.64111

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



2o

Data File E:\DATA\LXX\C-YIDING\C-90 2016-07-14 20-17-38\093-0501.D
Sample Name: C-89-1

Acq. Operator	: SYSTEM
Seq. Line	: 5
Acq. Instrument	: 1260HPLC-DAD
Location	: Vial 93
Injection Date	: 7/14/2016 9:47:21 PM
Inj	: 1
Inj Volume	: 5.000 µl
Acq. Method	: E:\DATA\LXX\C-YIDING\C-90 2016-07-14 20-17-38\DAD-OJ(1-6)-95-5-1.0ML-5-ALL-50MIN.M
Last changed	: 7/14/2016 8:54:13 PM by SYSTEM
Analysis Method	: E:\DATA\LXX\C-YIDING\C-90 2016-07-14 20-17-38\DAD-OJ(1-6)-95-5-1.0ML-5-ALL-50MIN.M (Sequence Method)
Last changed	: 7/15/2016 8:24:38 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

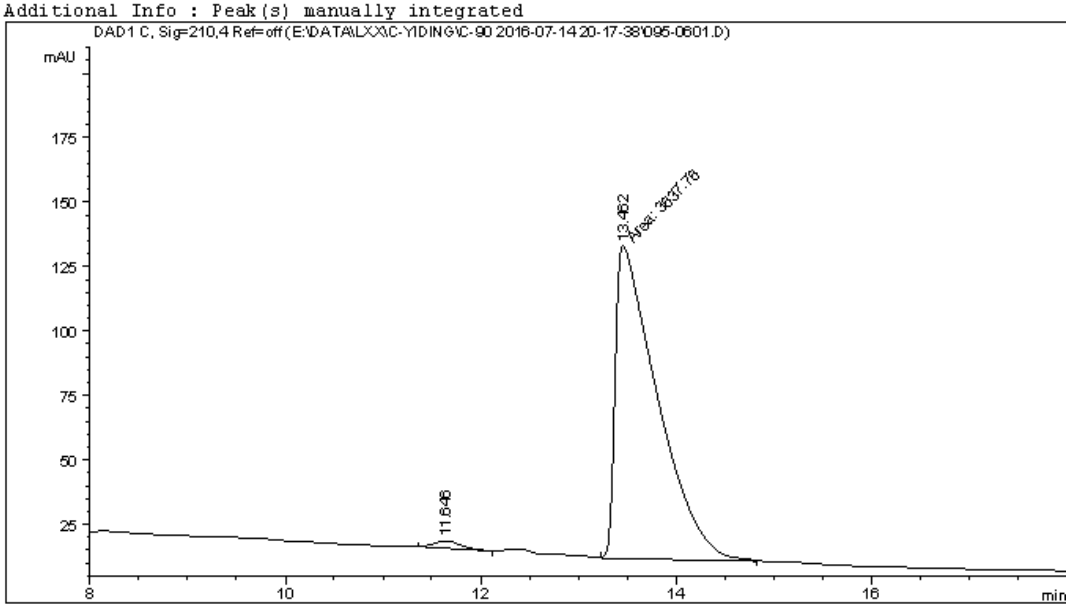
Signal 1: DAD1 C, Sig=210,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.543	FM	0.4003	673.00104	28.01959	52.6637
2	13.865	BB	0.3772	604.91992	24.23234	47.3363
Totals :				1277.92096	52.25193	

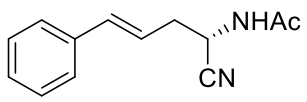
*** End of Report ***

Data File E:\DATA\LXX\C-YIDING\C-90 2016-07-14 20-17-38\095-0601.D
Sample Name: C-90

```
    ==================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   6
    Acq. Instrument : 1260HPLC-DAD                   Location : Vial 95
    Injection Date  : 7/14/2016 10:13:17 PM              Inj :   1
                                                   Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\LXX\C-YIDING\C-90 2016-07-14 20-17-38\DAD-0J(1-6)-95-5-1.0ML-5-
                      ALL-50MIN.M
    Last changed    : 7/14/2016 8:54:13 PM by SYSTEM
    Analysis Method : E:\DATA\LXX\C-YIDING\C-90 2016-07-14 20-17-38\DAD-0J(1-6)-95-5-1.0ML-5-
                      ALL-50MIN.M (Sequence Method)
    Last changed    : 7/15/2016 8:27:01 AM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```



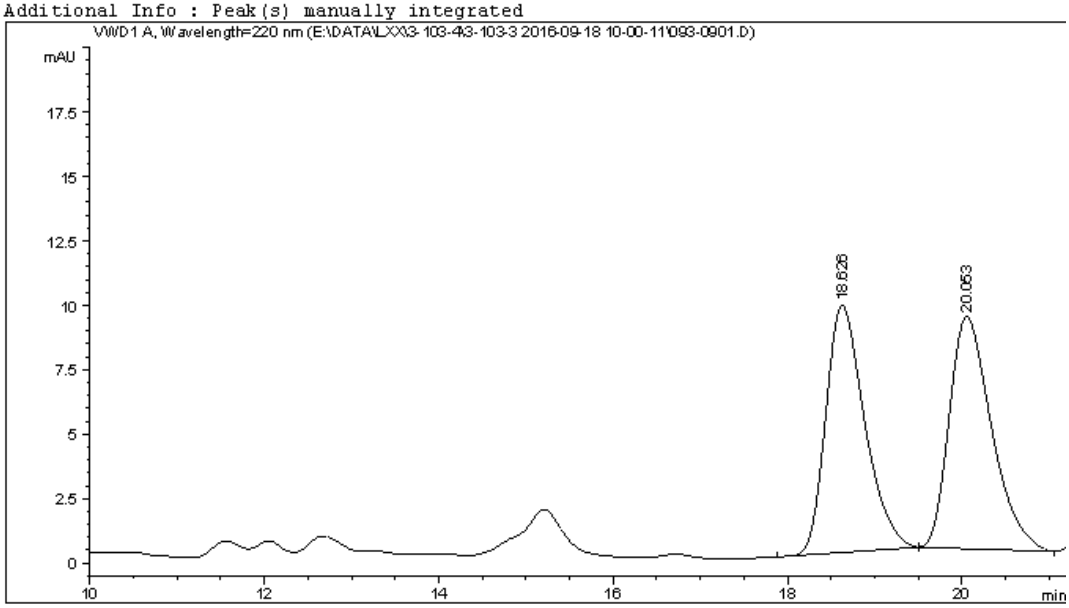

```
    ==================================================================
                            Area Percent Report
    ==================================================================

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


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

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]        %
    ----|-------|----|-------|----------|----------|--------|
       1  11.646 BB    0.2710   52.73094    2.75852   1.4288
       2  13.462 MM    0.4996 3637.76001  121.35647  98.5712

    Totals :                  3690.49095  124.11499


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



2p

Data File E:\DATA\LXX\3-103-4\3-103-3 2016-09-18 10-00-11\093-0901.D
Sample Name: C-102-1-Rac

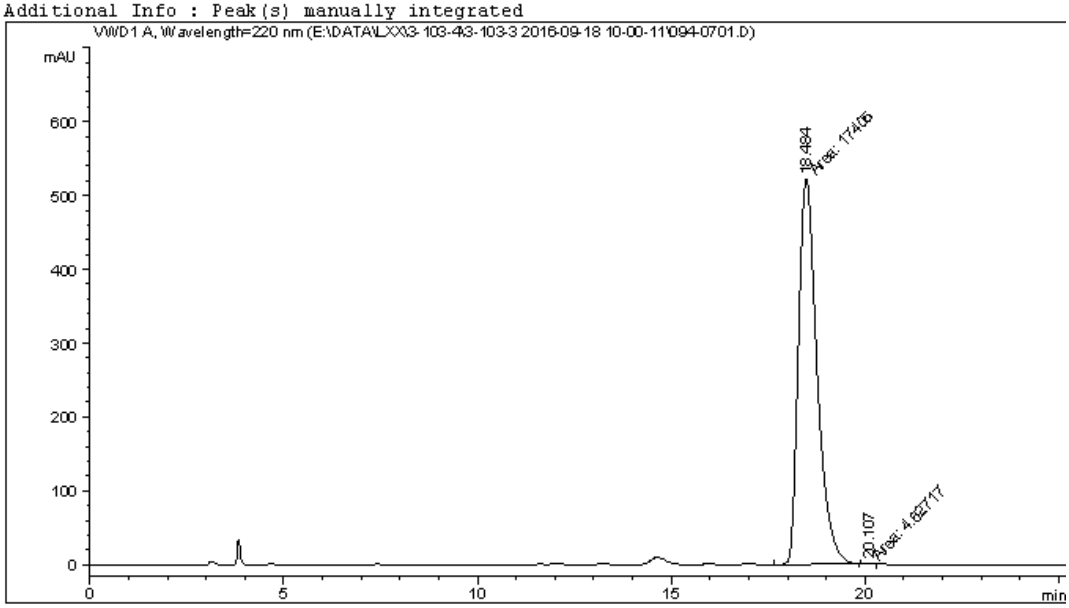
```
    ====================================================================
    Acq. Operator   : SYSTEM                          Seq. Line :   9
    Acq. Instrument : 1260HPLC-VWD                     Location : Vial 93
    Injection Date  : 9/18/2016 2:52:15 PM                  Inj :   1
                                                    Inj Volume : 3.000 µl
    Acq. Method     : E:\DATA\LXX\3-103-4\3-103-3 2016-09-18 10-00-11\VWD-AD(1-6)-95-5-1ML-3UL
                      -40MIN-220NM.M
    Last changed    : 9/18/2016 10:00:11 AM by SYSTEM
    Analysis Method : E:\DATA\LXX\3-103-4\3-103-3 2016-09-18 10-00-11\VWD-AD(1-6)-95-5-1ML-3UL
                      -40MIN-220NM.M (Sequence Method)
    Last changed    : 9/22/2016 7:55:02 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

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


    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  18.626 BB    0.4686  296.65326    9.59833  49.4413
       2  20.053 BB    0.5121  303.35776    9.00392  50.5587

    Totals :                   600.01102   18.60225

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

Data File E:\DATA\LXX\3-103-4\3-103-3 2016-09-18 10-00-11\094-0701.D
Sample Name: C-102-2

```
    ======================================================================
    Acq. Operator   : SYSTEM                          Seq. Line :   7
    Acq. Instrument : 1260HPLC-VWD                     Location : Vial 94
    Injection Date  : 9/18/2016 1:30:46 PM                 Inj :   1
                                                     Inj Volume : 3.000 µl
    Acq. Method     : E:\DATA\LXX\3-103-4\3-103-3 2016-09-18 10-00-11\VWD-AD(1-6)-95-5-1ML-3UL
                      -40MIN-220NM.M
    Last changed    : 9/18/2016 10:00:11 AM by SYSTEM
    Analysis Method : E:\DATA\LXX\3-103-4\3-103-3 2016-09-18 10-00-11\VWD-AD(1-6)-95-5-1ML-3UL
                      -40MIN-220NM.M (Sequence Method)
    Last changed    : 9/22/2016 7:53:19 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  18.484 MM    0.5561 1.74050e4   521.67749  99.9734
       2  20.107 MM    0.2456    4.62717 3.13968e-1   0.0266

    Totals :                  1.74096e4   521.99146


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