

Pd(*(R)*-DTBM-SEGphos)Cl₂-catalyzed kinetic resolution of tertiary propargylic alcohols

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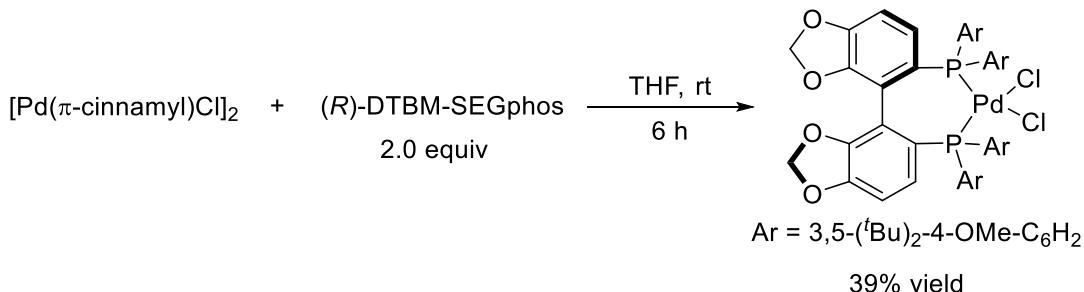
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General Information. NMR spectra were taken with Bruker Avance III spectrometer (400 MHz for ¹H NMR, 100 MHz for ¹³C NMR) in CDCl₃. All ¹H NMR experiments were measured with tetramethylsilane (0 ppm) in CDCl₃ as the internal reference; ¹³C NMR experiments were measured in relative to the signal of CDCl₃ (77.0 ppm). All reactions were carried out in Schlenk tubes. (R)- or (S)-DTBM-SEGphos was purchased from Strem Chemicals Inc.; (PhO)₂POOH was purchased from Energy Chemical, acidified with 1 N HCl under stirring, and extracted with dichloromethane, then the solvent was removed under vacuum. Petroleum ether (b.p. 60~90°C) was purchased from Shanghai Titan Scientific Co., Ltd. Toluene was used as received without further purification. The reaction should be conducted in a hood working efficiently with a CO detector due to the toxicity of CO gas. All the temperatures are referred to the oil baths used. Recoveries of substrates were determined by ¹H NMR analysis using dibromomethane as the internal standard. The tertiary propargylic alcohols¹ and racemic 2,3-allenoic acids² were prepared according to the literature methods. The optically active tertiary propargylic alcohols (S)-**1a**-(S)-**1f**, (S)-**1h**, (S)-**1j**-(S)-**1l**, (S)-**1o**-(S)-**1t** (S)-**1j**-(S)-**1l**³ and the chiral 2,3-allenoic acids (S)-**2a**-(S)-**2v**⁴ are known compounds.

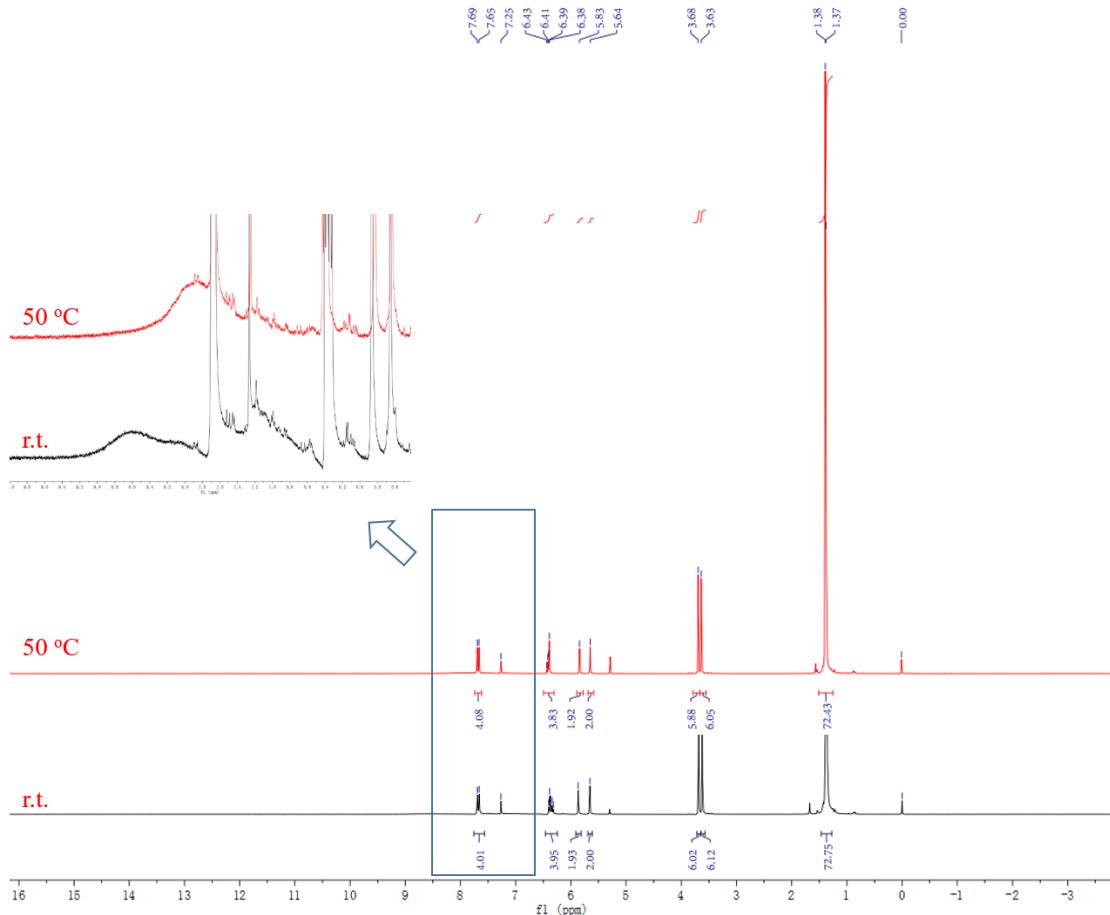
Experimental details and analytical data

Synthesis of palladium complex

(1) Preparation of Pd((R)-DTBM-SEGphos)Cl₂ (zwf-6-135)⁵

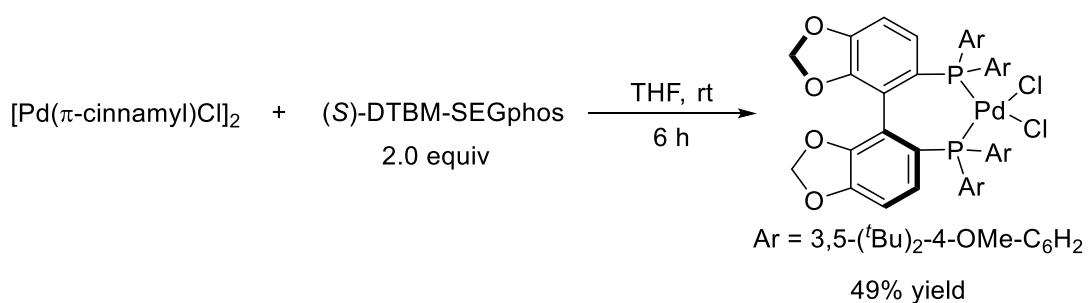


Typical Procedure I: To a dry flask were added [Pd(π -cinnamyl)Cl]₂ (518.3 mg, 1 mmol) and (*R*)-DTBM-SEGphos (2.4077 g, 2 mmol). Then THF (50 mL) were added under nitrogen atmosphere. After being stirred at rt for 6 h, removal of THF via evaporation and recrystallization from CH₂Cl₂/*n*-hexane afforded Pd((*R*)-DTBM-SEGphos)Cl₂ (1.0473 g, 39%). $[\alpha]_{\text{D}}^{25} = +263.1$ (*c* = 1.14, CHCl₃); solid; m.p. > 220 °C; **¹H NMR** (rt, 400 MHz, CDCl₃): δ = 9.31-6.52 (m, 8 H, Ar-H), 6.43-6.29 (m, 4 H, Ar-H), 5.87 (s, 2 H, CH₂), 5.66 (s, 2 H, CH₂), 3.69 (s, 6 H, 2 x CH₃), 3.62 (s, 6 H, 2 x CH₃), 1.43-1.29 (m, 72 H, 24 x CH₃); **¹³C NMR** (100 MHz, CDCl₃): δ = 162.6 (d, *J* = 2.4 Hz), 161.5 (d, *J* = 2.4 Hz), 149.7 (d, *J* = 2.4 Hz), 146.2 (d, *J* = 12.6 Hz), 143.8 (d, *J* = 14.2 Hz), 142.6 (d, *J* = 11.8 Hz), 134.3 (d, *J* = 12.6 Hz), 129.4 (d, *J* = 8.7 Hz), 124.1 (d, *J* = 5.5 Hz), 124.0 (d, *J* = 64.0 Hz), 123.6 (d, *J* = 5.6 Hz), 119.2 (d, *J* = 56.1 Hz), 117.3 (d, *J* = 2.4 Hz), 117.2 (d, *J* = 3.2 Hz), 108.0 (d, *J* = 11.8 Hz), 100.8, 64.3 (d, *J* = 2.3 Hz), 36.0, 35.9, 32.0, 31.8; **³¹P NMR** (162 MHz, CDCl₃): δ = 29.1; **IR** (neat): ν = 2959, 1440, 1393, 1264, 1227, 1207, 1182, 1140, 1115, 1046, 1007 cm⁻¹; **MS** (MALDI) *m/z* (%): 1319 (M(³⁵Cl)-Cl)⁺; Anal. Calcd. for C₇₀H₁₀₀Cl₂O₈P₂Pd: C 65.50, H 7.43; found: C 65.66, H 7.31. (Variable temperature ¹H-NMR spectra was given for explaining the hidden proton)



Supplementary Figure 1. $^1\text{H-NMR}$ (400 MHz) spectra of Pd(*(R)*-DTBM-SEGphos)Cl₂ in CDCl₃ at different temperature.

(2) Preparation of Pd((S)-DTBM-SEGphos)Cl₂ (zwf-7-124)

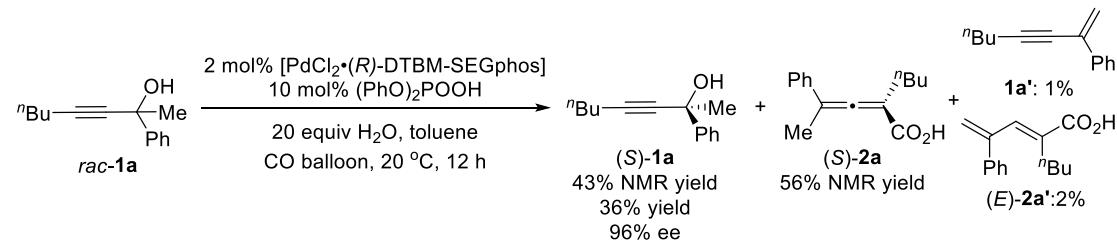


Following Typical Procedure I: the reaction of $[\text{Pd}(\pi\text{-cinnamyl})\text{Cl}]_2$ (518.6 mg, 1 mmol), (*S*)-DTBM-SEGphos (2.4083 g, 2 mmol) and THF (50 mL) afforded $\text{Pd}((S)\text{-DTBM-SEGphos})\text{Cl}_2$ (1.3242 g, 49%). $[\alpha]_D^{26} = -260.3$ ($c = 1.24, \text{CHCl}_3$); solid; m.p. > 220 °C; **$^1\text{H NMR}$** (rt, 400 MHz, CDCl_3): $\delta = 9.30\text{--}6.51$ (m, 8 H, Ar-H), 6.42–6.29 (m, 4 H, Ar-H), 5.87 (s, 2 H, CH_2), 5.66 (s, 2 H, CH_2), 3.69 (s, 6 H, 2 x CH_3), 3.62 (s, 6 H, 2

$\times \text{CH}_3$), 1.42-1.30 (m, 72 H, 24 $\times \text{CH}_3$); **^{13}C NMR** (100 MHz, CDCl_3): δ = 162.6 (d, J = 2.4 Hz), 161.5 (d, J = 2.4 Hz), 149.7 (d, J = 2.4 Hz), 146.2 (d, J = 12.6 Hz), 143.8 (d, J = 12.7 Hz), 142.6 (d, J = 11.8 Hz), 134.3 (d, J = 11.8 Hz), 129.4 (d, J = 8.7 Hz), 124.1 (d, J = 5.5 Hz), 124.0 (d, J = 64.0 Hz), 123.6 (d, J = 4.8 Hz), 119.2 (d, J = 56.1 Hz), 117.3 (d, J = 2.4 Hz), 117.2 (d, J = 2.4 Hz), 108.0 (d, J = 11.8 Hz), 100.8, 64.3 (d, J = 2.3 Hz), 36.0, 35.9, 32.0, 31.8; **^{31}P NMR** (162 MHz, CDCl_3): δ = 29.0; **IR** (neat): ν = 2955, 2940, 1440, 1393, 1264, 1226, 1182, 1140, 1115, 1046, 1006 cm^{-1} ; **MS** (MALDI) m/z (%): 1319 ($\text{M}^{(35)\text{Cl}}\text{-Cl}$) $^+$; Anal. Calcd. for $\text{C}_{70}\text{H}_{100}\text{Cl}_2\text{O}_8\text{P}_2\text{Pd}$: C 65.50, H 7.43; found: C 65.71, H 7.30.

Synthesis of chiral tertiary propargylic alcohols

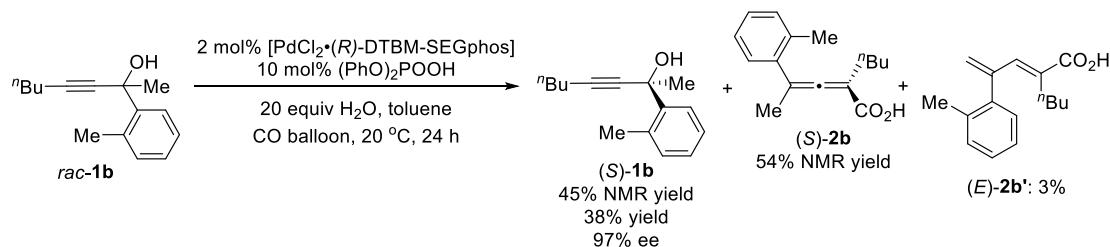
(1) Preparation of (*S*)-2-phenyloct-3-yn-2-ol ((*S*)-1a) (wj-1-015-1)



Typical Procedure II: To a Schlenk flask (25 mL) were added $[\text{PdCl}_2\bullet(R)\text{-DTBM-SEGphos}]$ (13.4 mg, 0.01 mmol) and $(\text{PhO})_2\text{PO}_2\text{H}$ (12.7 mg, 0.05 mmol). After addition, the flask was degassed and refilled with Ar for three times to ensure the complete exclusion of air. Then *rac*-1a (100.3 mg, 0.5 mmol)/toluene (1.5 mL) and H_2O (180 μL , d = 1.0 g/mL, 180.0 mg, 10 mmol)/toluene (1.0 mL) were added sequentially. After that, the Ar gas line was closed. The resulting mixture was then frozen with a liquid nitrogen bath, degassed to remove the argon inside completely, and refilled with CO by a balloon of CO (about 1 L) for three times. Then the liquid nitrogen bath was removed and the resulting mixture was allowed to stand until completely thawed, vigorously stirred at 20 °C with a balloon of CO for 12 h, warmed up to room temperature, diluted with 5 mL of ethyl acetate, filtered through a short column silica gel (3 cm) eluted with ethyl acetate (20 mL), and concentrated. The crude product was analyzed by ^1H NMR with CH_2Br_2 (35 μL , 2.477 g/mL, 0.5 mmol) as the internal standard: 56% NMR yield of

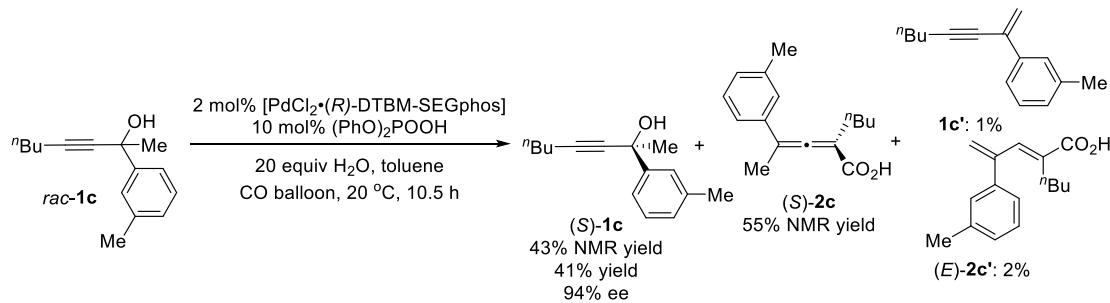
(*S*)-**2a**, 1% of **1a'**, and 2% of (*E*)-**2a'** were formed with 43% of (*S*)-**1a** remained. The residue was purified by chromatography on silica gel to afford the product (*S*)-**1a** (36.4 mg, 36%) [eluent: petroleum ether / ethyl ether / dichloromethane = 30/1/1 (480mL), then petroleum ether / ethyl acetate = 10/1 (440 mL)]: 96% ee (HPLC conditions: AS-H column, hexane/*i*PrOH = 98/2, 1.0 mL/min, λ = 214 nm, t_R (minor) = 7.2 min, t_R (major) = 11.0 min); oil; **1H NMR** (400 MHz, CDCl₃): δ = 7.65 (d, J = 7.6 Hz, 2 H, Ar-H), 7.35 (t, J = 7.6 Hz, 2 H, Ar-H), 7.30-7.23 (m, 1 H, Ar-H), 2.38 (d, J = 8.8 Hz, 1 H, OH), 2.27 (t, J = 7.0 Hz, 2 H, CH₂), 1.74 (s, 3 H, CH₃), 1.58-1.48 (m, 2 H, CH₂), 1.48-1.38 (m, 2 H, CH₂), 0.92 (t, J = 7.2 Hz, 3 H, CH₃); **13C NMR** (100 MHz, CDCl₃): δ = 146.2, 128.1, 127.4, 124.9, 85.6, 83.7, 70.0, 33.5, 30.7, 21.9, 18.4, 13.5.

(2) Preparation of (*S*)-2-(2-methylphenyl)oct-3-yn-2-ol ((*S*)-**1b**) (wj-1-017-1)



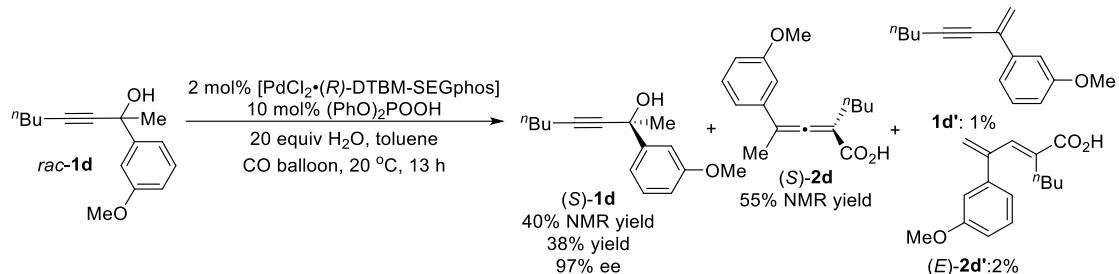
Following Typical Procedure II, the reaction of [PdCl₂•(*R*)-DTBM-SEGphos] (13.6 mg, 0.01 mmol), (PhO)₂PO₂H (12.6 mg, 0.05 mmol), *rac*-**1b** (108.7 mg, 0.5 mmol), and H₂O (180 μ L, d = 1.0 g/mL, 180.0 mg, 10.0 mmol) in toluene (2.5 mL) afforded the product (*S*)-**1b** (41.2 mg, 38%) (54% NMR yield of (*S*)-**2b**, and 3% of (*E*)-**2b'** were formed with 45% of (*S*)-**1b** remained based on **1H NMR** analysis of the crude product) [eluent: petroleum ether / diethyl ether / DCM = 30/1/1 (448 mL), then petroleum ether/ethyl acetate = 10/1 (440 mL)]: 97% ee (HPLC conditions: AS-H column, hexane/*i*PrOH = 98/2, 1.0 mL/min, λ = 214 nm, t_R (minor) = 6.5 min, t_R (major) = 8.5 min); oil; **1H NMR** (400 MHz, CDCl₃): δ = 7.73-7.65 (m, 1 H, Ar-H), 7.21-7.10 (m, 3 H, Ar-H), 2.62 (s, 3 H, CH₃), 2.39-2.30 (m, 1 H, OH), 2.23 (t, J = 7.0 Hz, 2 H, CH₂), 1.80 (s, 3 H, CH₃), 1.56-1.45 (m, 2 H, CH₂), 1.45-1.34 (m, 2 H, CH₂), 0.90 (t, J = 7.2 Hz, 3 H, CH₃); **13C NMR** (100 MHz, CDCl₃): δ = 142.8, 135.6, 132.2, 127.4, 125.6, 124.9, 85.2, 84.1, 69.7, 31.1, 30.6, 22.0, 21.2, 18.4, 13.5.

(3) Preparation of (*S*)-2-(3-methylphenyl)oct-3-yn-2-ol ((*S*)-1c) (wj-1-020-1)



Following Typical Procedure II, the reaction of [PdCl₂•(R)-DTBM-SEGphos] (13.8 mg, 0.01 mmol), (PhO)₂PO₂H (12.5 mg, 0.05 mmol), *rac*-1c (109.6 mg, 0.5 mmol), and H₂O (180 μL, d = 1.0 g/mL, 180.0 mg, 10.0 mmol) in toluene (2.5 mL) afforded the product (*S*)-1c (44.4 mg, 41%) (55% NMR yield of (*S*)-2c, 1% of 1c', and 2% of (*E*)-2c' were formed with 43% of (*S*)-1c remained based on ¹H NMR analysis of the crude product) [eluent: petroleum ether / diethyl ether / DCM = 30/1/1 (480 mL), then petroleum ether/ethyl acetate = 10/1 (440 mL)]: 94% ee (HPLC conditions: AS-H column, hexane/ⁱPrOH = 98/2, 1.0 mL/min, λ = 214 nm, t_R (minor) = 6.6 min, t_R (major) = 8.8 min); oil; ¹H NMR (400 MHz, CDCl₃): δ = 7.51-7.39 (m, 2 H, Ar-H), 7.28-7.19 (m, 1 H, Ar-H), 7.12-7.02 (m, 1 H, Ar-H), 2.46-2.33 (m, 4 H, CH₃ and OH), 2.27 (t, *J* = 7.0 Hz, 2 H, CH₂) 1.73 (s, 3 H, CH₃), 1.58-1.38 (m, 4 H, 2 x CH₂), 0.93 (t, *J* = 7.2 Hz, 3 H, CH₃); ¹³C NMR (100 MHz, CDCl₃): δ = 146.2, 137.7, 128.2, 128.1, 125.6, 122.0, 85.5, 83.9, 70.0, 33.5, 30.7, 21.9, 21.5, 18.4, 13.5.

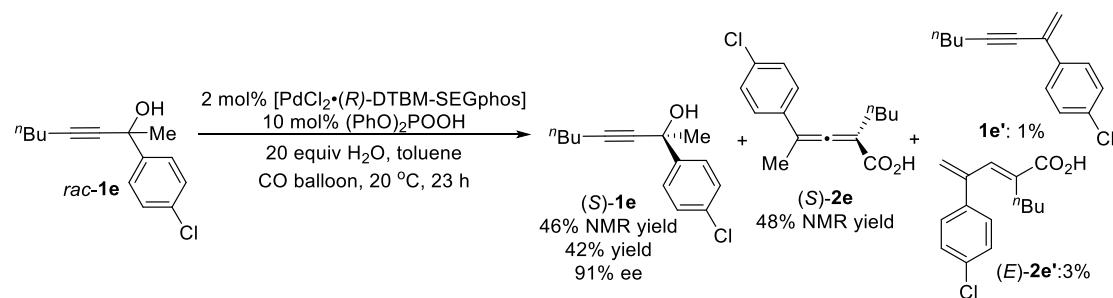
(4) Preparation of (*S*)-2-(3-methoxyphenyl)oct-3-yn-2-ol ((*S*)-1d) (wj-1-024-1)



Following Typical Procedure II, the reaction of [PdCl₂•(R)-DTBM-SEGphos] (13.6 mg, 0.01 mmol), (PhO)₂PO₂H (12.4 mg, 0.05 mmol), *rac*-1d (115.7 mg, 0.5 mmol), and H₂O (180 μL, d = 1.0 g/mL, 180.0 mg, 10 mmol) in toluene (2.5 mL) afforded the product (*S*)-1d (44.5 mg, 38%) (55% NMR yield of (*S*)-2d, 1% of 1d', and

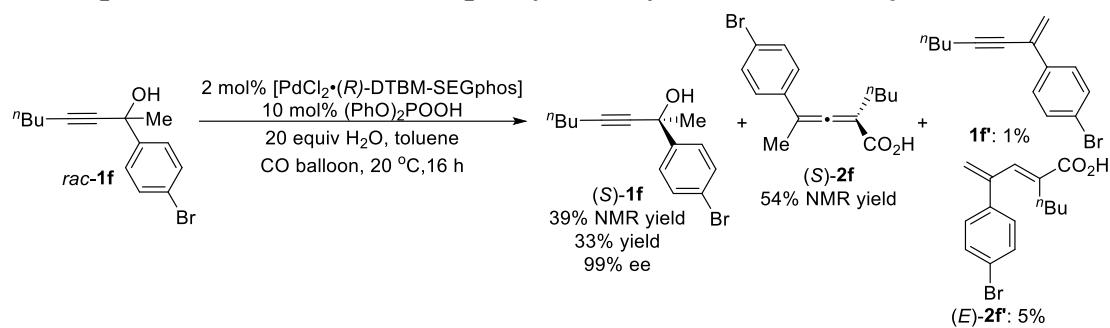
2% of (*E*)-**2d'** were formed with 40% of (*S*)-**1d** remained based on ¹H NMR analysis of the crude product) [eluent: petroleum ether / ethyl ether / dichloromethane = 30/1/1 (480 mL), then petroleum ether / ethyl acetate = 10/1 (660 mL)]: 97% ee (HPLC conditions: AS-H column, hexane/*i*PrOH = 98/2, 1.0 mL/min, λ = 214 nm, t_R (minor) = 13.0 min, t_R (major) = 18.8 min); oil; **1H NMR** (400 MHz, CDCl₃): δ = 7.29-7.18 (m, 3 H, Ar-H), 6.85-6.76 (m, 1 H, Ar-H), 3.81 (s, 3 H, CH₃), 2.46 (s, 1 H, OH), 2.27 (t, J = 7.0 Hz, 2 H, CH₂), 1.73 (s, 3 H, CH₃), 1.58-1.49 (m, 2 H, CH₂), 1.49-1.38 (m, 2 H, CH₂), 0.92 (t, J = 7.2 Hz, 3 H, CH₃); **13C NMR** (100 MHz, CDCl₃): δ = 159.4, 148.0, 129.2, 117.3, 112.9, 110.7, 85.5, 83.7, 69.9, 55.2, 33.5, 30.7, 21.9, 18.4, 13.5.

(5) Preparation of (*S*)-2-(4-chlorophenyl)oct-3-yn-2-ol ((*S*)-**1e**) (wj-1-027-1)



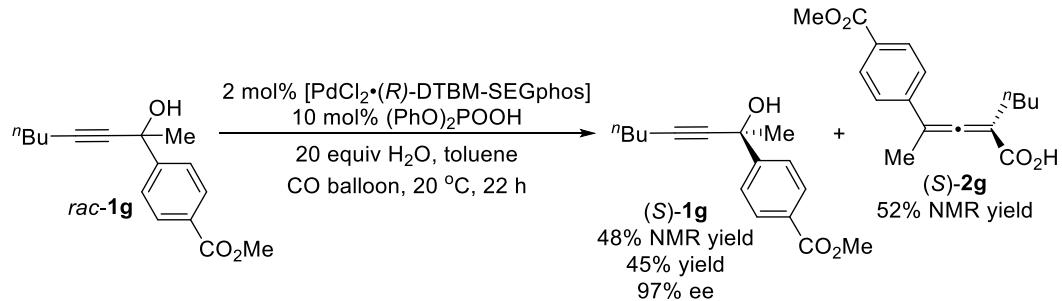
Following Typical Procedure II, the reaction of [PdCl₂•(R)-DTBM-SEGphos] (13.6 mg, 0.01 mmol), (PhO)₂PO₂H (12.4 mg, 0.05 mmol), *rac*-**1e** (118.0 mg, 0.5 mmol), and H₂O (180 μ L, d = 1.0 g/mL, 180.0 mg, 10 mmol) in toluene (2.5 mL) afforded the product (*S*)-**1e** (49.1 mg, 42%) (48% NMR yield of (*S*)-**2e**, 1% of **1e'**, and 3% of (*E*)-**2e'** were formed with 46% of (*S*)-**1e** remained based on ¹H NMR analysis of the crude product) [eluent: petroleum ether / ethyl ether / dichloromethane = 30/1/1 (480 mL), then petroleum ether / ethyl acetate = 10/1 (550 mL)]: 91% ee (HPLC conditions: AS-H column, hexane/*i*PrOH = 98/2, 1.0 mL/min, λ = 214 nm, t_R (minor) = 8.1 min, t_R (major) = 11.1 min); oil; **1H NMR** (400 MHz, CDCl₃): δ = 7.57 (d, J = 8.8 Hz, 2 H, Ar-H), 7.30 (d, J = 8.4 Hz, 2 H, Ar-H), 2.48-2.38 (m, 1 H, OH), 2.26 (t, J = 7.0 Hz, 2 H, CH₂), 1.71 (s, 3 H, CH₃), 1.58-1.48 (m, 2 H, CH₂), 1.48-1.37 (m, 2 H, CH₂), 0.92 (t, J = 7.2 Hz, 3 H, CH₃); **13C NMR** (100 MHz, CDCl₃): δ = 144.8, 133.2, 128.2, 126.5, 86.0, 83.3, 69.6, 33.6, 30.6, 21.9, 18.3, 13.5.

(6) Preparation of (*S*)-2-(4-bromophenyl)oct-3-yn-2-ol ((*S*)-1f) (wj-1-028-1)



Following Typical Procedure II, the reaction of $[\text{PdCl}_2\bullet(\text{R})\text{-DTBM-SEGphos}]$ (13.5 mg, 0.01 mmol), $(\text{PhO})_2\text{PO}_2\text{H}$ (12.4 mg, 0.05 mmol), *rac-1f* (141.7 mg, 0.5 mmol), and H_2O (180 μL , d = 1.0 g/mL, 180.0 mg, 10 mmol) in toluene (2.5 mL) afforded the product (*S*)-1f (47.0 mg, 33%) (54% NMR yield of (*S*)-2f, 1% of 1f, and 5% of (*E*)-2f were formed with 39% of (*S*)-1f remained based on ^1H NMR analysis of the crude product) [eluent: petroleum ether / ethyl ether / dichloromethane = 30/1/1 (480 mL), then petroleum ether/ethyl acetate = 10/1 (440 mL)]: 99% ee (HPLC conditions: AS-H column, hexane/ $^i\text{PrOH}$ = 98/2, 1.0 mL/min, λ = 214 nm, t_R (minor) = 8.3 min, t_R (major) = 11.0 min); oil; **$^1\text{H NMR}$** (400 MHz, CDCl_3): δ = 7.52 (d, J = 8.4 Hz, 2 H, Ar-H), 7.46 (d, J = 8.8 Hz, 2 H, Ar-H), 2.45-2.33 (m, 1 H, OH), 2.26 (t, J = 7.0 Hz, 2 H, CH_2), 1.70 (s, 3 H, CH_3), 1.57-1.48 (m, 2 H, CH_2), 1.48-1.36 (m, 2 H, CH_2), 0.92 (t, J = 7.2 Hz, 3 H, CH_3); **$^{13}\text{C NMR}$** (100 MHz, CDCl_3): δ = 145.4, 131.2, 126.9, 121.4, 86.0, 83.3, 69.6, 33.6, 30.6, 21.9, 18.3, 13.5.

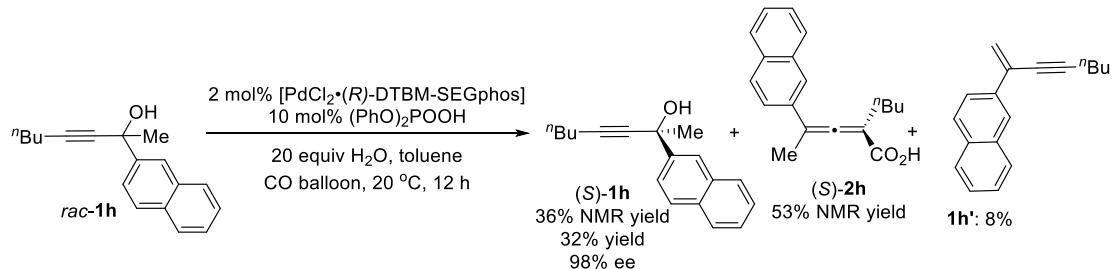
(7) Preparation of (*S*)-2-(4-methoxycarbonyl)phenyl)oct-3-yn-2-ol ((*S*)-1g) (wj-1-032-1)



Following Typical Procedure II, the reaction of $[\text{PdCl}_2\bullet(\text{R})\text{-DTBM-SEGphos}]$ (13.5 mg, 0.01 mmol), $(\text{PhO})_2\text{PO}_2\text{H}$ (12.5 mg, 0.05 mmol), *rac-1g* (130.0 mg, 0.5 mmol), and H_2O (180 μL , d = 1.0 g/mL, 180.0 mg, 10.0 mmol) in toluene (2.5 mL)

afforded the product (*S*)-**1g** (58.4 mg, 45%) (52% NMR yield of (*S*)-**2g** was formed with 48% of (*S*)-**1g** remained based on ^1H NMR analysis of the crude product) [eluent: petroleum ether / diethyl ether / dichloromethane = 30/1/1 (480 mL), then petroleum ether / ethyl acetate = 20/1(840 mL), 10/1 (550 mL)]: 97% ee (HPLC conditions: AS-H column, hexane/ $^i\text{PrOH}$ = 90/10, 1.0 mL/min, λ = 214 nm, t_R (major) = 6.8 min, t_R (minor) = 7.5 min); $[\alpha]_D^{25} = +4.8$ ($c = 1.17$, CHCl_3); oil; $^1\text{H NMR}$ (400 MHz, CDCl_3): $\delta = 8.00$ (d, $J = 8.4$ Hz, 2 H, Ar-H), 7.71 (d, $J = 8.0$ Hz, 2 H, Ar-H), 3.91 (s, 3 H, CH_3), 2.64 (s, 1 H, OH), 2.27 (t, $J = 7.0$ Hz, 2 H, CH_2), 1.73 (s, 3 H, CH_3), 1.58-1.48 (m, 2 H, CH_2), 1.48-1.37 (m, 2 H, CH_2), 0.92 (t, $J = 7.2$ Hz, 3 H, CH_3); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): $\delta = 166.9, 151.2, 129.5, 129.2, 125.0, 86.1, 83.2, 69.8, 52.0, 33.6, 30.6, 21.9, 18.3, 13.5$; IR (neat): $\nu = 3461, 2956, 2932, 2873, 2243, 1723, 1707, 1610, 1437, 1407, 1278, 1184, 1113, 1059, 1019$ cm $^{-1}$; MS (70 eV, EI) m/z (%): 245 ((M- CH_3) $^+$, 100); HRMS (EI) calcd m/z for $\text{C}_{15}\text{H}_{17}\text{O}_3$ [M- CH_3] $^+$: 245.1172, found: 245.1172.

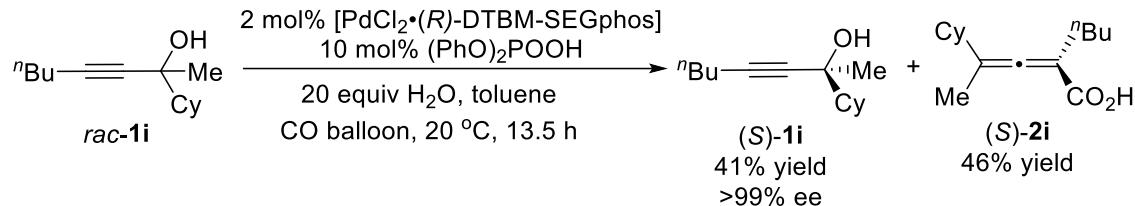
(8) Preparation of (*S*)-2-(2-naphthyl)oct-3-yn-2-ol ((*S*)-**1h**) (wj-4-134)



Following Typical Procedure II, the reaction of $[\text{PdCl}_2 \bullet (R)\text{-DTBM-SEGphos}]$ (13.6 mg, 0.01 mmol), $(\text{PhO})_2\text{PO}_2\text{H}$ (12.5 mg, 0.05 mmol), *rac*-**1h** (126.3 mg, 0.5 mmol), and H_2O (180 μL , d = 1.0 g/mL, 180.0 mg, 10.0 mmol) in toluene (2.5 mL) afforded (*S*)-**1h** (39.9 mg, 32%) (53% NMR yield of (*S*)-**2h** and 8% of **1h'** were formed with 36% of (*S*)-**1h** remained based on ^1H NMR analysis of the crude product) [eluent: petroleum ether / ethyl ether / dichloromethane = 30/1/1 (640 mL)]: 98% ee (HPLC conditions: AS-H column, hexane/ $^i\text{PrOH}$ = 95/5, 1.0 mL/min, λ = 214 nm, t_R (minor) = 7.1 min, t_R (major) = 8.4 min); oil; $^1\text{H NMR}$ (400 MHz, CDCl_3): $\delta = 8.15\text{-}8.09$ (m, 1 H, Ar-H), 7.89-7.78 (m, 3 H, Ar-H), 7.77-7.70 (m, 1 H, Ar-H), 7.52-7.42 (m, 2 H, Ar-H), 2.47 (s, 1 H, OH), 2.31 (t, $J = 7.2$ Hz, 2 H, CH_2), 1.82 (s, 3 H, CH_3), 1.60-1.51 (m, 2 H,

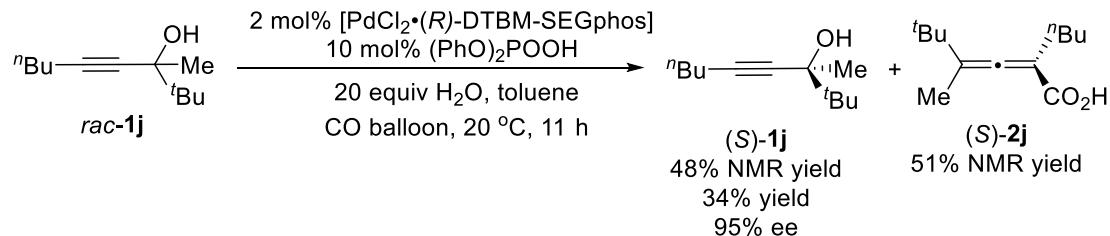
CH_2), 1.51-1.40 (m, 2 H, CH_2), 0.94 (t, $J = 7.2$ Hz, 3 H, CH_3); ^{13}C NMR (100 MHz, CDCl_3): $\delta = 143.5, 133.0, 132.8, 128.3, 128.0, 127.5, 126.1, 125.9, 123.6, 123.3, 85.9, 83.8, 70.1, 33.4, 30.7, 22.0, 18.4, 13.6$.

(9) Preparation of (*S*)-2-cyclohexyloct-3-yn-2-ol ((*S*)-1*i*) (wj-1-038-1)



Following Typical Procedure II, the reaction of $[\text{PdCl}_2\bullet(R)\text{-DTBM-SEGphos}]$ (13.7 mg, 0.01 mmol), $(\text{PhO})_2\text{PO}_2\text{H}$ (12.6 mg, 0.05 mmol), *rac*-1*i* (103.7 mg, 0.5 mmol), and H_2O (180 μL , d = 1.0 g/mL, 180.0 mg, 10.0 mmol) in toluene (2.5 mL) afforded the product (*S*)-1*i* (42.5 mg, 41%) (Feature peak overlap based on ^1H NMR analysis of the crude product) [eluent: petroleum ether / ethyl ether / dichloromethane = 30/1/1 (480 mL), then petroleum ether / ethyl acetate = 10/1 (330 mL)]: >99% ee (HPLC conditions: AS-H column, hexane/ $^i\text{PrOH}$ = 98/2, 1.0 mL/min, $\lambda = 214$ nm, t_R (major) = 6.0 min); $[\alpha]_D^{25} = +6.6$ ($c = 1.25$, CHCl_3); oil; ^1H NMR (400 MHz, CDCl_3): $\delta = 2.20$ (t, $J = 6.8$ Hz, 2 H, CH_2), 2.00-1.75 (m, 5 H, 2 x CH_2 and OH), 1.54-1.03 (m, 14 H, CH and 5 x CH_2 and CH_3), 0.91 (t, $J = 7.0$ Hz, 3 H, CH_3); ^{13}C NMR (100 MHz, CDCl_3): $\delta = 84.3, 83.4, 71.1, 48.9, 30.9, 27.9, 27.6, 27.3, 26.34, 26.31, 26.2, 21.9, 18.3, 13.5$; IR (neat): $\nu = 3419, 2926, 2854, 1751, 1450, 1370, 1328, 1247, 1191, 1145, 1111, 1068, 1046 \text{ cm}^{-1}$; MS (ESI) m/z (%): 209 ($\text{M}+\text{H}^+$)); HRMS (ESI) calcd for $\text{C}_{14}\text{H}_{25}\text{O}$ [$\text{M}+\text{H}^+$]: 209.1900, found: 209.1896.

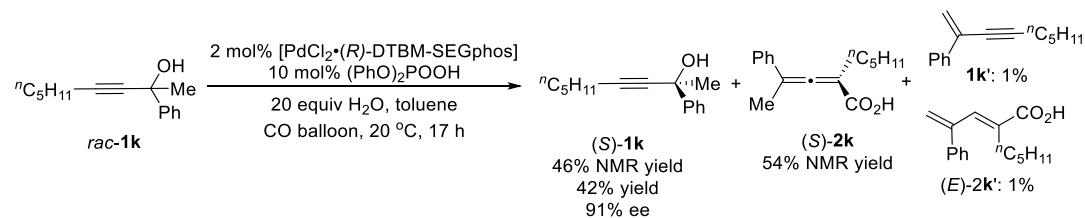
(10) Preparation of (*S*)-2,2,3-trimethylnon-4-yn-3-ol ((*S*)-1*j*) (wj-1-055-1)



Following Typical Procedure II, the reaction of $[\text{PdCl}_2\bullet(R)\text{-DTBM-SEGphos}]$ (13.6

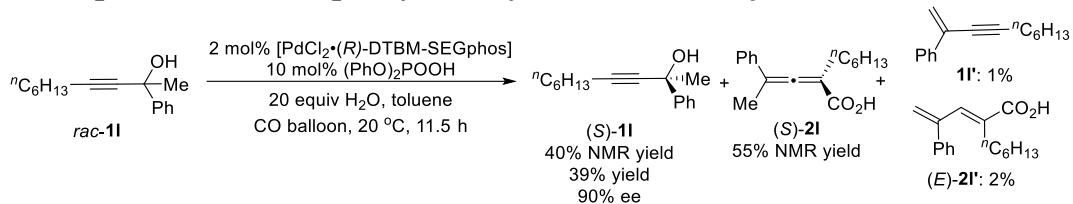
mg, 0.01 mmol), $(\text{PhO})_2\text{PO}_2\text{H}$ (12.5 mg, 0.05 mmol), *rac*-**1j** (92.4 mg, 0.5 mmol), and H_2O (180 μL , d = 1.0 g/mL, 180.0 mg, 10.0 mmol) in toluene (2.5 mL) afforded the product (*S*)-**1j** (31.0 mg, 34%) (51% NMR yield of (*S*)-**2j** with 48% of (*S*)-**1j** remained based on ^1H NMR analysis of the crude product) [eluent: petroleum ether / diethyl ether / DCM = 30/1/1 (320 mL), then petroleum ether / ethyl acetate = 10/1 (330 mL)]: 95% ee (HPLC conditions: IC column, hexane/ $i\text{PrOH}$ = 98/2, 1.0 mL/min, λ = 214 nm, t_R (minor) = 4.2 min, t_R (major) = 4.5 min); oil; **1H NMR** (400 MHz, CDCl_3): δ = 2.20 (t, J = 6.8 Hz, 2 H, CH_2), 1.82 (s, 1 H, OH), 1.54-1.37 (m, 7 H, 2 x CH_2 and CH_3), 1.03 (s, 9 H, 3 x CH_3), 0.91 (t, J = 7.2 Hz, 3 H, CH_3); **13C NMR** (100 MHz, CDCl_3): δ = 84.2, 83.8, 74.0, 38.2, 30.9, 25.1, 25.0, 21.9, 18.3, 13.5.

(11) Preparation of (*S*)-2-phenylnon-3-yn-2-ol ((*S*)-**1k**) (wj-1-051-1)



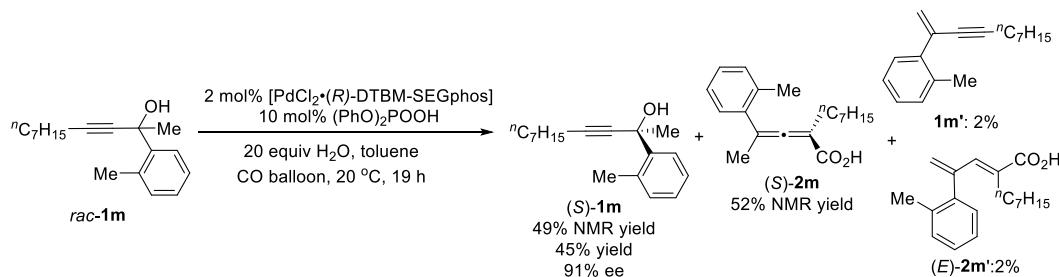
Following Typical Procedure II, the reaction of $[\text{PdCl}_2\bullet(\text{R})\text{-DTBM-SEGphos}]$ (13.7 mg, 0.01 mmol), $(\text{PhO})_2\text{PO}_2\text{H}$ (12.5 mg, 0.05 mmol), *rac*-**1k** (108.5 mg, 0.5 mmol), and H_2O (180 μL , d = 1.0 g/mL, 180.0 mg, 10.0 mmol) in toluene (2.5 mL) afforded the product (*S*)-**1k** (45.6 mg, 42%) (54% NMR yield of (*S*)-**2k**, 1% of **1k'**, and 1% of (*E*)-**2k'** were formed with 46% of (*S*)-**1k** remained based on ^1H NMR analysis of the crude product) [eluent: petroleum ether / ethyl ether / dichloromethane = 30/1/1 (480 mL), then petroleum ether / ethyl acetate = 10/1 (440 mL)]: 91% ee (HPLC conditions: AS-H column, hexane/ $i\text{PrOH}$ = 98/2, 1.0 mL/min, λ = 214 nm, t_R (minor) = 6.5 min, t_R (major) = 9.4 min); oil; **1H NMR** (400 MHz, CDCl_3): δ = 7.65 (d, J = 7.6 Hz, 2 H, Ar-H), 7.34 (t, J = 7.4 Hz, 2 H, Ar-H), 7.30-7.23 (m, 1 H, Ar-H), 2.38 (m, 1 H, OH), 2.26 (t, J = 7.2 Hz, 2 H, CH_2), 1.74 (s, 3 H, CH_3), 1.55 (quint, J = 7.2 Hz, 2 H, CH_2), 1.44-1.27 (m, 4 H, 2 x CH_2), 0.91 (t, J = 7.0 Hz, 3 H, CH_3); **13C NMR** (100 MHz, CDCl_3): δ = 146.2, 128.1, 127.4, 125.0, 85.7, 83.8, 70.0, 33.5, 31.1, 28.3, 22.1, 18.7, 13.9.

(12) Preparation of (*S*)-2-phenyldec-3-yn-2-ol ((*S*)-1l**) (wj-1-054-1)**



Following Typical Procedure II, the reaction of $[\text{PdCl}_2\bullet(R)\text{-DTBM-SEGphos}]$ (13.5 mg, 0.01 mmol), $(\text{PhO})_2\text{PO}_2\text{H}$ (12.4 mg, 0.05 mmol), *rac-1l* (116.0 mg, 0.5 mmol), and H_2O (180 μL , d = 1.0 g/mL, 180.0 mg, 10.0 mmol) in toluene (2.5 mL) afforded the product (*S*)-**1l** (44.8 mg, 39%) (55% NMR yield of (*S*)-**2l**, 1% of **1l'**, and 2% of (*E*)-**2l'** were formed with 40% of (*S*)-**1l** remained based on ^1H NMR analysis of the crude product) [eluent: petroleum ether / ethyl ether / dichloromethane = 30/1/1 (480 mL), then petroleum ether / ethyl acetate = 10/1 (440 mL)]: 90% ee (HPLC conditions: AS-H column, hexane/ $i\text{PrOH}$ = 98/2, 1.0 mL/min, $\lambda = 214$ nm, t_{R} (minor) = 6.0 min, t_{R} (major) = 8.5 min); oil; **1H NMR** (400 MHz, CDCl_3): $\delta = 7.65$ (d, $J = 7.6$ Hz, 2 H, Ar-H), 7.34 (t, $J = 7.4$ Hz, 2 H, Ar-H), 7.29-7.23 (m, 1 H, Ar-H), 2.43-2.33 (m, 1 H, OH), 2.26 (t, $J = 7.2$ Hz, 2 H, CH_2), 1.74 (s, 3 H, CH_3), 1.55 (quint, $J = 7.2$ Hz, 2 H, CH_2), 1.47-1.36 (m, 2 H, CH_2), 1.35-1.24 (m, 4 H, 2 x CH_2), 0.89 (t, $J = 6.8$ Hz, 3 H, CH_3); **13C NMR** (100 MHz, CDCl_3): $\delta = 146.2, 128.2, 127.5, 125.0, 85.7, 83.8, 70.0, 33.5, 31.3, 28.6, 28.5, 22.5, 18.7, 14.0$.

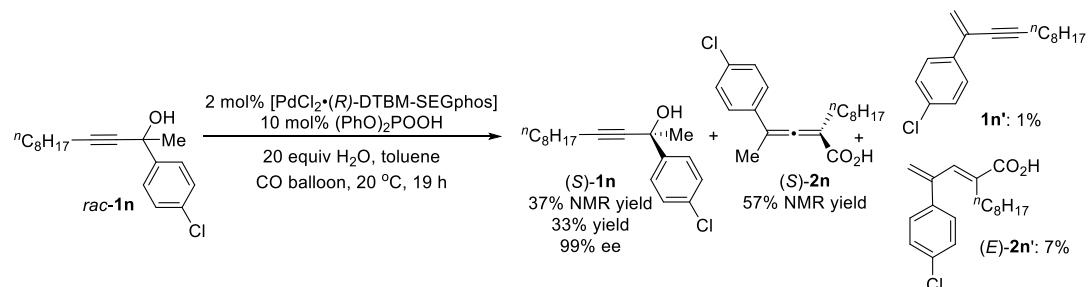
(13) Preparation of (*S*)-2-(o-tolyl)undec-3-yn-2-ol ((*S*)-1m**) (wj-1-072-1)**



Following Typical Procedure II, the reaction of $[\text{PdCl}_2\bullet(R)\text{-DTBM-SEGphos}]$ (13.7 mg, 0.01 mmol), $(\text{PhO})_2\text{PO}_2\text{H}$ (12.5 mg, 0.05 mmol), *rac-1m* (129.1 mg, 0.5 mmol), and H_2O (180 μL , d = 1.0 g/mL, 180.0 mg, 10.0 mmol) in toluene (2.5 mL) afforded the product (*S*)-**1m** (57.9 mg, 45%) (52% NMR yield of (*S*)-**2m**, 2% of **1m'**, and 2% of (*E*)-**2m'** were formed with 49% of (*S*)-**1m** remained based on ^1H NMR analysis of the

crude product) [eluent: petroleum ether / ethyl ether / dichloromethane = 30/1/1 (416 mL), then petroleum ether / ethyl acetate = 8/1 (360 mL)]: 91% ee (HPLC conditions: AS-H column, hexane/*i*PrOH = 98/2, 1.0 mL/min, λ = 214 nm, t_R (minor) = 4.9 min, t_R (major) = 5.9 min); $[\alpha]_D^{26} = -2.4$ ($c = 1.02$, CHCl₃); oil; **1H NMR** (400 MHz, CDCl₃): $\delta = 7.73\text{-}7.65$ (m, 1 H, Ar-H), 7.21-7.07 (m, 3 H, Ar-H), 2.62 (s, 3 H, CH₃), 2.31 (s, 1 H, OH), 2.23 (t, $J = 7.2$ Hz, 2 H, CH₂), 1.80 (s, 3 H, CH₃), 1.52 (quint, $J = 7.2$ Hz, 2 H, CH₂), 1.42-1.20 (m, 8 H, 4 x CH₂), 0.88 (t, $J = 6.8$ Hz, 3 H, CH₃); **13C NMR** (100 MHz, CDCl₃): $\delta = 142.9, 135.6, 132.2, 127.5, 125.6, 125.0, 85.3, 84.1, 69.7, 31.7, 31.1, 28.8, 28.7, 28.5, 22.6, 21.2, 18.7, 14.0$; **IR** (neat): $\nu = 3428, 2928, 2856, 2240, 1456, 1367, 1329, 1221, 1078, 1059, 1047\text{cm}^{-1}$; **MS** (70 eV, EI) *m/z* (%): 258 (M⁺, 3.42), 243 (100); **HRMS** (EI) calcd for C₁₇H₂₃O [M-CH₃]⁺: 243.1743, found: 243.1742.

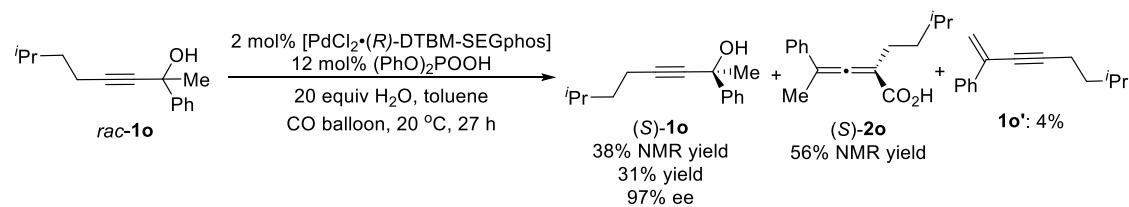
(14) Preparation of (*S*)-2-(4-chlorophenyl)dodec-3-yn-2-ol ((*S*)-1n) (wj-1-069-1)



Following Typical Procedure II, the reaction of [PdCl₂•(R)-DTBM-SEGphos] (13.7 mg, 0.01 mmol), (PhO)₂PO₂H (12.4 mg, 0.05 mmol), *rac*-1n (146.3 mg, 0.5 mmol), and H₂O (180 μ L, d = 1.0 g/mL, 180.0 mg, 10.0 mmol) in toluene (2.5 mL) afforded the product (*S*)-1n (48.0 mg, 33%) (57% NMR yield of (*S*)-2n, 1% of 1n', and 7% of (*E*)-2n' were formed with 37% of (*S*)-1n remained based on ¹H NMR analysis of the crude product) [eluent: petroleum ether / ethyl ether / dichloromethane = 30/1/1 (480 mL), then petroleum ether / ethyl acetate = 10/1 (660 mL)]: 99% ee (HPLC conditions: AS-H column, hexane/*i*PrOH = 98/2, 1.0 mL/min, λ = 214 nm, t_R (minor) = 5.4 min, t_R (major) = 6.6 min); $[\alpha]_D^{27} = -2.5$ ($c = 1.16$, CHCl₃); oil; **1H NMR** (400 MHz, CDCl₃): $\delta = 7.58$ (d, $J = 8.0$ Hz, 2 H, Ar-H), 7.30 (d, $J = 8.0$ Hz, 2 H, Ar-H), 2.39 (s, 1 H, OH), 2.25 (t, $J = 7.0$ Hz, 2 H, CH₂), 1.71 (s, 3 H, CH₃), 1.59-1.47 (m, 2 H, CH₂), 1.45-1.34 (m, 2 H, CH₂), 1.34-1.16 (m, 8 H, 4 x CH₂), 0.94-0.78 (m, 3 H, CH₃); **13C NMR** (100

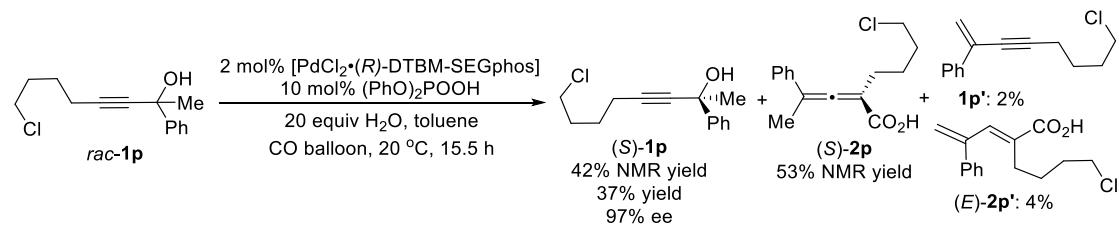
MHz, CDCl₃): δ = 144.8, 133.2, 128.2, 126.5, 86.1, 83.3, 69.6, 33.6, 31.8, 29.2, 29.0, 28.9, 28.5, 22.6, 18.7, 14.1; **IR** (neat): ν = 3393, 2926, 2855, 2243, 1489, 1466, 1400, 1368, 1329, 1229, 1173, 1092, 1015cm⁻¹; **MS** (70 eV, EI) *m/z* (%): 294 (M⁺(³⁷Cl), 0.76), 292 (M⁺(³⁵Cl), 2.04), 277 (100); **HRMS** (EI) calcd for C₁₇H₂₂³⁵ClO [M-CH₃]⁺: 277.1354, found: 277.1357.

(15) Preparation of (*S*)-7-methyl-2-phenyloct-3-yn-2-ol ((*S*)-1o) (wj-1-120-1)



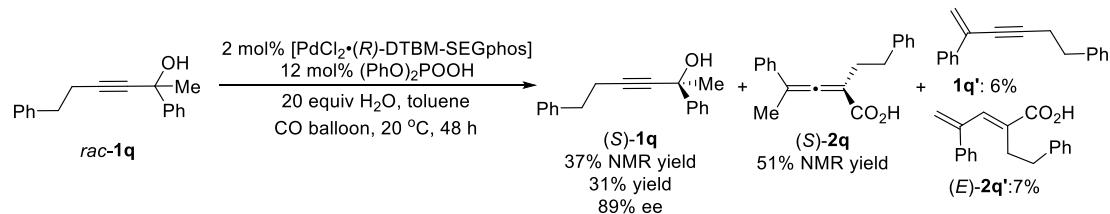
Following Typical Procedure II, the reaction of [PdCl₂•(*R*)-DTBM-SEGphos] (13.5 mg, 0.01 mmol), (PhO)₂PO₂H (15.1 mg, 0.06 mmol), *rac*-1o (107.0 mg, 0.5 mmol), and H₂O (180 μL, d = 1.0 g/mL, 180.0 mg, 10.0 mmol) in toluene (2.5 mL) afforded the product (*S*)-1o (33.2 mg, 31%) (56% NMR yield of (*S*)-2o and 4% of 1o') were formed with 38% of (*S*)-1o remained based on ¹H NMR analysis of the crude product [eluent: petroleum ether / ethyl ether / dichloromethane = 30/1/1 (448 mL), then petroleum ether / ethyl acetate = 8/1 (450 mL)]: 97% ee (HPLC conditions: AS-H column, hexane/ⁱPrOH = 98/2, 1.0 mL/min, λ = 214 nm, t_R (minor) = 6.7 min, t_R (major) = 9.2 min); oil; **1H NMR** (400 MHz, CDCl₃): δ = 7.70-7.62 (m, 2 H, Ar-H), 7.39-7.31 (m, 2 H, Ar-H), 7.31-7.24 (m, 1 H, Ar-H), 2.35 (s, 1 H, OH), 2.28 (t, *J* = 7.4 Hz, 2 H, CH₂), 1.81-1.64 (m, 4 H, CH and CH₃), 1.45 (q, *J* = 7.3 Hz, 2 H, CH₂), 0.91 (d, *J* = 6.4 Hz, 6 H, 2 x CH₃); **13C NMR** (100 MHz, CDCl₃): δ = 146.2, 128.1, 127.4, 124.9, 85.7, 83.6, 70.0, 37.6, 33.5, 27.3, 22.1, 16.7.

(16) Preparation of (*S*)-8-chloro-2-phenyloct-3-yn-2-ol ((*S*)-1p) (wj-1-057-1)



Following Typical Procedure II, the reaction of $[\text{PdCl}_2\bullet(R)\text{-DTBM-SEGphos}]$ (13.5 mg, 0.01 mmol), $(\text{PhO})_2\text{PO}_2\text{H}$ (12.5 mg, 0.05 mmol), *rac*-**1p** (118.7 mg, 0.5 mmol), and H_2O (180 μL , d = 1.0 g/mL, 180.0 mg, 10.0 mmol) in toluene (2.5 mL) afforded the product (*S*)-**1p** (44.4 mg, 37%) (53% NMR yield of (*S*)-**2p**, 2% of **1p'**, and 4% of (*E*)-**2p'** were formed with 42% of (*S*)-**1p** remained based on ^1H NMR analysis of the crude product) [eluent: petroleum ether / ethyl ether / dichloromethane = 30/1/1 (704 mL), then petroleum ether / ethyl acetate = 8/1 (630 mL)]: 97% ee (HPLC conditions: AS-H column, hexane/ $i\text{PrOH}$ = 95/5, 1.0 mL/min, λ = 214 nm, t_R (minor) = 9.1 min, t_R (major) = 15.8 min); oil; ^1H NMR (400 MHz, CDCl_3): δ = 7.64 (d, J = 7.6 Hz, 2 H, Ar-H), 7.35 (t, J = 7.4 Hz, 2 H, Ar-H), 7.31-7.23 (m, 1 H, Ar-H), 3.56 (t, J = 6.6 Hz, 2 H, CH_2), 2.49-2.14 (m, 3 H, OH and CH_2), 1.91 (quint, J = 7.0 Hz, 2 H, CH_2), 1.77-1.64 (m, 5 H, CH_2 and CH_3); ^{13}C NMR (100 MHz, CDCl_3): δ = 146.0, 128.2, 127.5, 124.9, 84.6, 84.5, 70.0, 44.4, 33.4, 31.6, 25.7, 18.0.

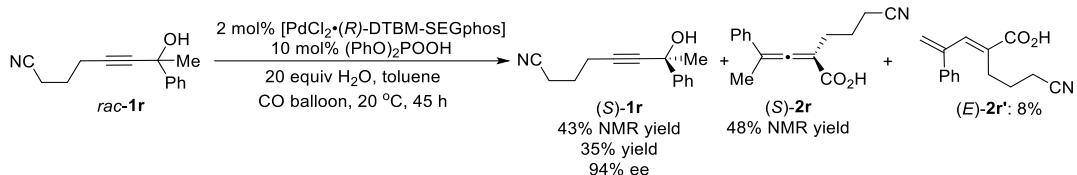
(17) Preparation of (*S*)-2,6-diphenylhex-3-yn-2-ol ((*S*)-**1q**) (wj-1-111-1)



Following Typical Procedure II, the reaction of $[\text{PdCl}_2\bullet(R)\text{-DTBM-SEGphos}]$ (13.6 mg, 0.01 mmol), $(\text{PhO})_2\text{PO}_2\text{H}$ (15.1 mg, 0.06 mmol), *rac*-**1q** (125.0 mg, 0.5 mmol), and H_2O (180 μL , d = 1.0 g/mL, 180.0 mg, 10.0 mmol) in toluene (2.5 mL) afforded the product (*S*)-**1q** (39.0 mg, 31%) (51% NMR yield of (*S*)-**2q**, 6% of **1q'**, and 7% of (*E*)-**2q'** were formed with 37% of (*S*)-**1q** remained based on ^1H NMR analysis of the crude product) [eluent: petroleum ether / ethyl ether / dichloromethane = 30/1/1 (790 mL), then petroleum ether / ethyl acetate = 8/1 (450 mL)]: 89% ee (HPLC conditions: AS-H column, hexane/ $i\text{PrOH}$ = 98/2, 1.0 mL/min, λ = 214 nm, t_R (minor) = 12.9 min, t_R (major) = 16.6 min); oil; ^1H NMR (400 MHz, CDCl_3): δ = 7.59-7.52 (m, 2 H, Ar-H), 7.37-7.17 (m, 8 H, Ar-H), 2.86 (t, J = 7.4 Hz, 2 H, CH_2), 2.57 (t, J = 7.4 Hz, 2 H, CH_2), 2.30 (s, 1 H, OH), 1.71 (s, 3 H, CH_3); ^{13}C NMR (100 MHz, CDCl_3): δ =

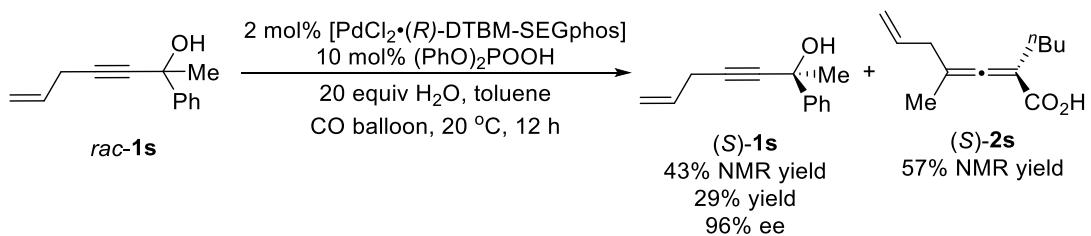
145.9, 140.5, 128.5, 128.4, 128.1, 127.5, 126.3, 124.9, 84.7, 84.6, 69.9, 34.9, 33.4, 20.9.

(18) Preparation of (*S*)-7-cyano-2-phenylhept-3-yn-2-ol ((*S*)-1r) (wj-1-082-1)



Following Typical Procedure II, the reaction of $[\text{PdCl}_2\bullet(R)\text{-DTBM-SEGphos}]$ (13.6 mg, 0.01 mmol), $(\text{PhO})_2\text{PO}_2\text{H}$ (12.5 mg, 0.05 mmol), *rac-1r* (106.0 mg, 0.5 mmol), and H_2O (180 μL , d = 1.0 g/mL, 180.0 mg, 10.0 mmol) in toluene (2.5 mL) afforded the product (*S*)-1r (36.8 mg, 35%) (48% NMR yield of (*S*)-2r and 8% of (*E*)-2r') were formed with 43% of (*S*)-1r remained based on ^1H NMR analysis of the crude product [eluent: petroleum ether / ethyl acetate = 8/1 (1080 mL) to 5/1(840 mL)]: 94% ee (HPLC conditions: AS-H column, hexane/ $^i\text{PrOH}$ = 90/10, 1.0 mL/min, λ = 214 nm, t_{R} (minor) = 17.1 min, t_{R} (major) = 29.4 min); oil; **$^1\text{H NMR}$** (400 MHz, CDCl_3): δ = 7.62 (d, J = 7.2 Hz, 2 H, Ar-H), 7.36 (t, J = 7.4 Hz, 2 H, Ar-H), 7.32-7.24 (m, 1 H, Ar-H), 2.75-2.33 (m, 5 H, OH and 2 x CH_2), 1.89 (quint, J = 6.9 Hz, 2 H, CH_2), 1.75 (s, 3 H, CH_3); **$^{13}\text{C NMR}$** (100 MHz, CDCl_3): δ = 145.7, 128.2, 127.6, 124.7, 119.1, 85.9, 82.3, 69.8, 33.3, 24.3, 17.9, 16.2.

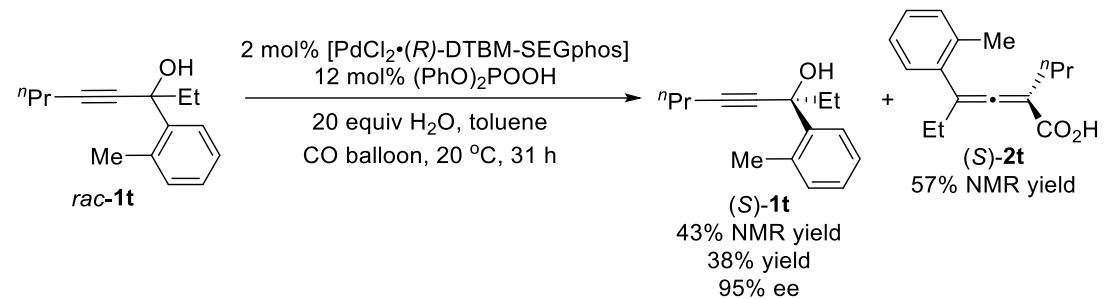
(19) Preparation of (*S*)-2-phenylhept-6-en-3-yn-2-ol ((*S*)-1s) (wj-1-134)



Following Typical Procedure II, the reaction of $[\text{PdCl}_2\bullet(R)\text{-DTBM-SEGphos}]$ (13.6 mg, 0.01 mmol), $(\text{PhO})_2\text{PO}_2\text{H}$ (12.6 mg, 0.05 mmol), *rac-1s* (93.1 mg, 0.5 mmol), and H_2O (180 μL , d = 1.0 g/mL, 180.0 mg, 10.0 mmol) in toluene (2.5 mL) afforded the product (*S*)-1s (27.3 mg, 29%) (57% NMR yield of (*S*)-2s was formed with 43% of (*S*)-1s remained based on ^1H NMR analysis of the crude product) [eluent: petroleum ether / ethyl ether / dichloromethane = 30/1/1 (960 mL): 96% ee (HPLC conditions: AS-H

column, hexane/*i*PrOH = 98/2, 1.0 mL/min, λ = 214 nm, t_R (minor) = 10.0 min, t_R (major) = 12.4 min); oil; **1H NMR** (400 MHz, CDCl₃): δ = 7.66 (d, J = 7.6 Hz, 2 H, Ar-H), 7.36 (t, J = 7.6 Hz, 2 H, Ar-H), 7.31-7.23 (m, 1 H, Ar-H), 5.91-5.78 (m, 1 H, CH), 5.35 (dd, J_1 = 16.8 Hz, J_2 = 1.6 Hz, 1 H, one proton of =CH₂), 5.14 (dd, J_1 = 10.0 Hz, J_2 = 1.6 Hz, 1 H, one proton of =CH₂), 3.10-3.03 (m, 2 H, CH₂), 2.38 (s, 1 H, OH), 1.77 (s, 3 H, CH₃); **13C NMR** (100 MHz, CDCl₃): δ = 145.9, 132.3, 128.2, 127.6, 124.9, 116.3, 86.2, 82.0, 70.0, 33.5, 23.0.

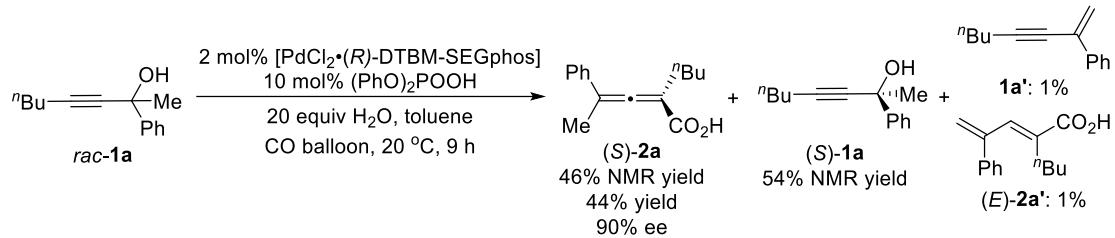
(20) Preparation of (*S*)-3-(o-tolyl)oct-4-yn-3-ol ((*S*)-1t) (wj-1-122-1)



Following Typical Procedure II, the reaction of [PdCl₂•(*R*)-DTBM-SEGphos] (13.7 mg, 0.01 mmol), (PhO)₂PO₂H (14.9 mg, 0.06 mmol), *rac*-1t (107.7 mg, 0.5 mmol), and H₂O (180 μ L, d = 1.0 g/mL, 180.0 mg, 10.0 mmol) in toluene (2.5 mL) afforded the product (*S*)-1t (40.8 mg, 38%) (57% NMR yield of (*S*)-2t was formed with 43% of (*S*)-1t remained based on ¹H NMR analysis of the crude product) [eluent: petroleum ether / ethyl ether / dichloromethane = 50/1/1 (520 mL), then petroleum ether / ethyl acetate = 8/1 (360 mL)]; 95% ee (HPLC conditions: AS-H column, hexane/*i*PrOH = 98/2, 1.0 mL/min, λ = 214 nm, t_R (minor) = 5.5 min, t_R (major) = 7.0 min); oil; **1H NMR** (400 MHz, CDCl₃): δ = 7.76-7.69 (m, 1 H, Ar-H), 7.21-7.12 (m, 3 H, Ar-H), 2.58 (s, 3 H, CH₃), 2.28-2.20 (m, 3 H, CH₂ and OH), 2.09-1.90 (m, 2 H, CH₂), 1.63-1.51 (m, 2 H, CH₂), 1.06-0.92 (m, 6 H, 2 x CH₃); **13C NMR** (100 MHz, CDCl₃): δ = 142.0, 135.4, 132.2, 127.3, 126.2, 125.4, 86.5, 83.1, 73.8, 35.2, 22.0, 21.3, 20.8, 13.6, 8.9.

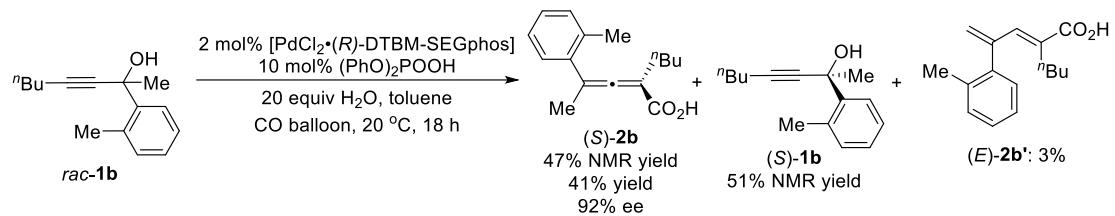
Synthesis of chiral 2,3-allenoic acids

(1) Preparation of (*S*)-2-butyl-4-phenyl-2,3-pentadienoic acid ((*S*)-2a) (wj-1-013-2)



Following Typical Procedure II, the reaction of [PdCl₂•(*R*)-DTBM-SEGphos] (13.5 mg, 0.01 mmol), (PhO)₂PO₂H (12.4 mg, 0.05 mmol), *rac*-1a (102.5 mg, 0.5 mmol), and H₂O (180 μL, d = 1.0 g/mL, 18.0 mg, 10 mmol) in toluene (2.5 mL) afforded the product (*S*)-2a (51.2 mg, 44%) (46% NMR yield of (*S*)-2a, 1% of 1a', and 1% of (*E*)-2a' were formed with 54% of (*S*)-1a remained based on ¹H NMR analysis of the crude product) [eluent: petroleum ether / ethyl ether / dichloromethane = 30/1/1 (640 mL), then petroleum ether / ethyl acetate = 10/1 (440 mL)]: 90% ee (HPLC conditions: AS-H column, hexane/ⁱPrOH = 98/2, 1.0 mL/min, λ = 214 nm, t_R (major) = 8.6 min, t_R (minor) = 11.6 min); solid; ¹H NMR (400 MHz, CDCl₃): δ = 7.42-7.29 (m, 4 H, Ar-H), 7.28-7.21 (m, 1 H, Ar-H), 2.32 (t, J = 7.6 Hz, 2 H, CH₂), 2.19 (s, 3 H, CH₃), 1.52-1.42 (m, 2 H, CH₂), 1.40-1.29 (m, 2 H, CH₂), 0.88 (t, J = 7.2 Hz, 3 H, CH₃); ¹³C NMR (100 MHz, CDCl₃): δ = 212.6, 172.8, 135.0, 128.5, 127.6, 126.1, 105.2, 101.8, 30.2, 28.3, 22.2, 16.3, 13.8.

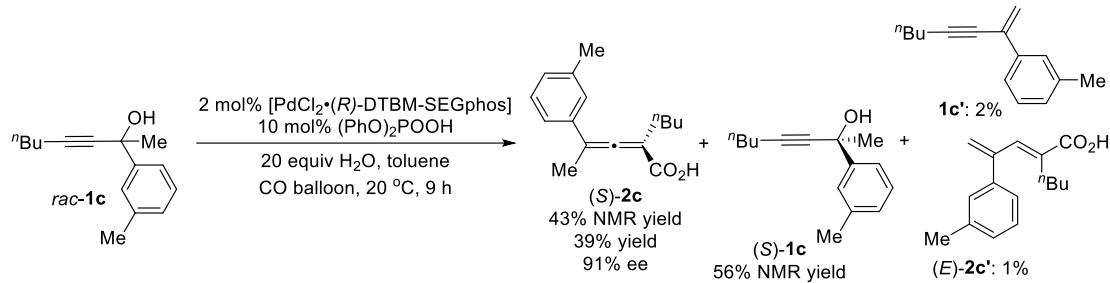
(2) Preparation of (*S*)-2-butyl-4-(2-methylphenyl)-2,3-pentadienoic acid ((*S*)-2b) (wj-1-016-2)



Following Typical Procedure II, the reaction of [PdCl₂•(*R*)-DTBM-SEGphos] (13.4 mg, 0.01 mmol), and (PhO)₂PO₂H (12.6 mg, 0.05 mmol), *rac*-1b (108.1 mg, 0.5 mmol), and H₂O (180 μL, d = 1.0 g/mL, 18.0 mg, 10 mmol) in toluene (2.5 mL) afforded the product (*S*)-2b (50.2 mg, 41%) (47% NMR yield of (*S*)-2b and 3% of (*E*)-2b' were

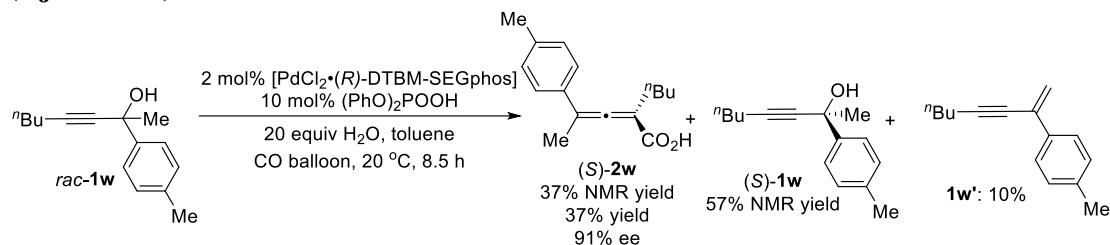
formed with 51% of (*S*)-**1b** remained based on ^1H NMR analysis of the crude product) [eluent: petroleum ether / ethyl ether / dichloromethane = 30/1/1 (480 mL), then petroleum ether / ethyl acetate = 10/1 (440 mL)]: 92% ee (HPLC conditions: AS-H column, hexane/ $i\text{PrOH}$ = 98/2, 1.0 mL/min, λ = 214 nm, t_{R} (major) = 7.1 min, t_{R} (minor) = 10.1 min); oil; ^1H NMR (400 MHz, CDCl_3): δ = 7.29-7.23 (m, 1 H, Ar-H), 7.21-7.14 (m, 3 H, Ar-H), 2.40 (s, 3 H, CH_3), 2.35-2.08 (m, 5 H, CH_2 and CH_3), 1.52-1.41 (m, 2 H, CH_2), 1.40-1.28 (m, 2 H, CH_2), 0.90 (t, J = 7.4 Hz, 3 H, CH_3); ^{13}C NMR (100 MHz, CDCl_3): δ = 210.1, 173.4, 136.2, 136.0, 130.6, 127.9, 127.6, 125.9, 104.6, 98.9, 30.1, 28.1, 22.2, 20.3, 19.9, 13.8.

(3) Preparation of (*S*)-2-butyl-4-(3-methylphenyl)-2,3-pentadienoic acid ((*S*)-**2c**) (wj-1-019-2)



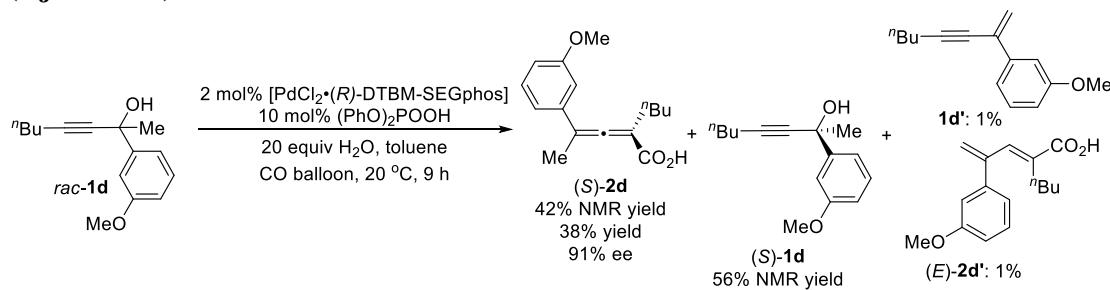
Following Typical Procedure II, the reaction of $[\text{PdCl}_2 \bullet (\text{R})\text{-DTBM-SEGphos}]$ (13.7 mg, 0.01 mmol), $(\text{PhO})_2\text{PO}_2\text{H}$ (12.5 mg, 0.05 mmol), **rac-1c** (109.0 mg, 0.5 mmol), and H_2O (180 μL , d = 1.0 g/mL, 180.0 mg, 10 mmol) in toluene (2.5 mL) afforded the product (*S*)-**2c** (48.5 mg, 39%) (43% NMR yield of (*S*)-**2c**, 2% of **1c'**, and 1% of (*E*)-**2c'** were formed with 56% of (*S*)-**1c** remained based on ^1H NMR analysis of the crude product) [eluent: petroleum ether / ethyl ether / dichloromethane = 30/1/1 (480 mL), then petroleum ether / ethyl acetate = 10/1 (440 mL)]: 91% ee (HPLC conditions: AS-H column, hexane/ $i\text{PrOH}$ = 98/2, 1.0 mL/min, λ = 214 nm, t_{R} (major) = 7.3 min, t_{R} (minor) = 9.3 min); solid; ^1H NMR (400 MHz, CDCl_3): δ = 7.26-7.12 (m, 3 H, Ar-H), 7.06 (d, J = 7.2 Hz, 1 H, Ar-H), 2.40-2.26 (m, 5 H, CH_2 and CH_3), 2.18 (s, 3 H, CH_3), 1.53-1.41 (m, 2 H, CH_2), 1.40-1.29 (m, 2 H, CH_2), 0.88 (t, J = 7.2 Hz, 3 H, CH_3); ^{13}C NMR (100 MHz, CDCl_3): δ = 212.6, 172.8, 138.1, 134.9, 128.42, 128.38, 126.8, 123.2, 105.2, 101.6, 30.2, 28.3, 22.2, 21.4, 16.4, 13.8.

(4) Preparation of (S)-2-butyl-4-(4-methylphenyl)-2,3-pentadienoic acid ((S)-2w) (w.j-1-022-2)



Following Typical Procedure II, the reaction of [PdCl₂•(R)-DTBM-SEGphos] (13.5 mg, 0.01 mmol), (PhO)₂PO₂H (12.6 mg, 0.05 mmol), *rac*-**1w** (108.7 mg, 0.5 mmol), and H₂O (180 μL, d = 1.0 g/mL, 180.0 mg, 10 mmol) in toluene (2.5 mL) afforded the product (*S*)-**2w** (45.7 mg, 37%) (37% NMR yield of (*S*)-**2w** and 10% of **1w'** were formed with 57% of (*S*)-**1w** remained based on ¹H NMR analysis of the crude product) [eluent: petroleum ether / ethyl ether / dichloromethane = 30/1/1 (480 mL), then petroleum ether / ethyl acetate = 10/1 (440 mL)]: 91% ee (HPLC conditions: AS-H column, hexane/ⁱPrOH = 98/2, 1.0 mL/min, λ = 214 nm, *t*_R (major) = 9.4 min, *t*_R (minor) = 10.7 min); solid; **1H NMR** (400 MHz, CDCl₃): δ = 7.27 (d, *J* = 8.0 Hz, 2 H, Ar-H), 7.14 (d, *J* = 7.6 Hz, 2 H, Ar-H), 2.37-2.25 (m, 5 H, CH₂ and CH₃), 2.17 (s, 3 H, CH₃), 1.50-1.40 (m, 2 H, CH₂), 1.40-1.28 (m, 2 H, CH₂), 0.87 (t, *J* = 7.2 Hz, 3 H, CH₃); **13C NMR** (100 MHz, CDCl₃): δ = 212.4, 172.6, 137.4, 132.0, 129.3, 126.0, 105.1, 101.6, 30.2, 28.4, 22.2, 21.1, 16.3, 13.8.

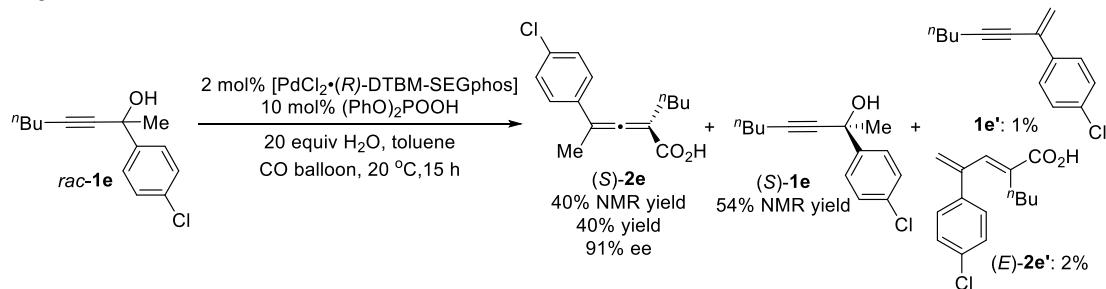
(5) Preparation of (*S*)-2-butyl-4-(3-methoxyphenyl)-2,3-pentadienoic acid ((*S*)-2d) (w/j-1-030-2)



Following Typical Procedure II, the reaction of $[\text{PdCl}_2\bullet(R)\text{-DTBM-SEGphos}]$ (13.7 mg, 0.01 mmol), $(\text{PhO})_2\text{PO}_2\text{H}$ (12.6 mg, 0.05 mmol), ***rac-1d*** (118.0 mg, 0.5 mmol), and H_2O (180 μL , d = 1.0 g/mL, 180.0 mg, 10 mmol) in toluene (2.5 mL) afforded the product ***(S)-2d*** (50.6 mg, 38%) (42% NMR yield of ***(S)-2d***, 1% of ***1d'***, and 1% of ***(E)-2d***).

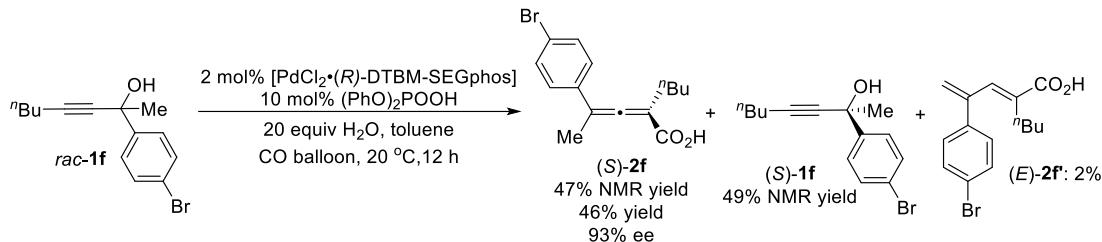
2d' were formed with 56% of (*S*)-**1d** remained based on ^1H NMR analysis of the crude product) [eluent: petroleum ether / ethyl ether / dichloromethane = 30/1/1 (480 mL), then petroleum ether / ethyl acetate = 10/1 (660 mL)]: 91% ee (HPLC conditions: AS-H column, hexane/*i*PrOH = 98/2, 1.0 mL/min, λ = 214 nm, t_R (major) = 12.0 min, t_R (minor) = 16.2 min); solid; **1H NMR** (400 MHz, CDCl_3): δ = 7.26 (t, J = 8.0 Hz, 1 H, Ar-H), 6.98 (d, J = 8.0 Hz, 1 H, Ar-H), 6.92 (s, 1 H, Ar-H), 6.80 (dd, J_1 = 8.0 Hz, J_2 = 2.0 Hz, 1 H, Ar-H), 3.80 (s, 3 H, OCH₃), 2.32 (t, J = 7.6 Hz, 2 H, CH₂), 2.18 (s, 3 H, CH₃), 1.51-1.40 (m, 2 H, CH₂), 1.40-1.29 (m, 2 H, CH₂), 0.88 (t, J = 7.2 Hz, 3 H, CH₃); **13C NMR** (100 MHz, CDCl_3): δ = 212.6, 172.5, 159.8, 136.6, 129.5, 118.6, 112.9, 112.0, 105.2, 101.8, 55.2, 30.2, 28.3, 22.3, 16.4, 13.8.

(6) Preparation of (*S*)-2-butyl-4-(4-chlorophenyl)-2,3-pentadienoic acid ((*S*)-**2e**) (wj-1-025-2)



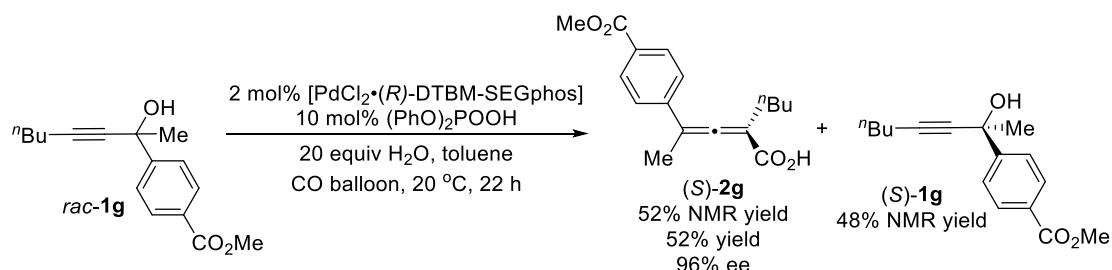
Following Typical Procedure II, the reaction of [PdCl₂•(*R*)-DTBM-SEGphos] (13.5 mg, 0.01 mmol), (PhO)₂PO₂H (12.6 mg, 0.05 mmol), **rac-1e** (117.4 mg, 0.5 mmol), and H₂O (180 μ L, d = 1.0 g/mL, 180.0 mg, 10 mmol) in toluene (2.5 mL) afforded the product (*S*)-**2e** (53.1 mg, 40%) (40% NMR yield of (*S*)-**2e**, 1% of **1e'**, and 2% of (*E*)-**2e'** were formed with 54% of (*S*)-**1e** remained based on ^1H NMR analysis of the crude product) [eluent: petroleum ether / ethyl ether / dichloromethane = 30/1/1 (480mL), then petroleum ether / ethyl acetate = 10/1 (550 mL)]: 91% ee (HPLC conditions: AS-H column, hexane/*i*PrOH = 98/2, 1.0 mL/min, λ = 214 nm, t_R (major) = 9.3 min, t_R (minor) = 12.4 min); solid; **1H NMR** (400 MHz, CDCl_3): δ = 7.30 (s, 4 H, Ar-H), 2.32 (t, J = 7.4 Hz, 2 H, CH₂), 2.17 (s, 3 H, CH₃), 1.50-1.40 (m, 2 H, CH₂), 1.39-1.28 (m, 2 H, CH₂), 0.88 (t, J = 7.2 Hz, 3 H, CH₃); **13C NMR** (100 MHz, CDCl_3): δ = 212.4, 172.2, 133.6, 133.4, 128.7, 127.3, 104.4, 102.1, 30.2, 28.3, 22.2, 16.3, 13.8.

(7) Preparation of (*S*)-2-butyl-4-(4-bromophenyl)-2,3-pentadienoic acid ((*S*)-2f) (wj-1-031-2)



Following Typical Procedure II, the reaction of $[\text{PdCl}_2\bullet(R)\text{-DTBM-SEGphos}]$ (13.6 mg, 0.01 mmol), $(\text{PhO})_2\text{PO}_2\text{H}$ (12.5 mg, 0.05 mmol), **rac-1f** (140.1 mg, 0.5 mmol), and H_2O (180 μL , d = 1.0 g/mL, 180.0 mg, 10 mmol) in toluene (2.5 mL) afforded the product **(S)-2f** (70.9 mg, 46%) (47% NMR yield of **(S)-2f** and 2% of **(E)-2f** were formed with 49% of **(S)-1f** remained based on ^1H NMR analysis of the crude product) [eluent: petroleum ether / ethyl ether / dichloromethane = 30/1/1 (480 mL), petroleum ether / ethyl acetate = 10/1 (440 mL)]: 93% ee (HPLC conditions: AS-H column, hexane/ $i\text{PrOH}$ = 98/2, 1.0 mL/min, λ = 214 nm, t_{R} (major) = 10.5 min, t_{R} (minor) = 15.0 min); solid; **$^1\text{H NMR}$** (400 MHz, CDCl_3): δ = 7.46 (d, J = 8.4 Hz, 2 H, Ar-H), 7.24 (d, J = 8.4 Hz, 2 H, Ar-H), 2.32 (t, J = 7.4 Hz, 2 H, CH_2), 2.17 (s, 3 H, CH_3), 1.50-1.40 (m, 2 H, CH_2), 1.39-1.28 (m, 2 H, CH_2), 0.88 (t, J = 7.2 Hz, 3 H, CH_3); **$^{13}\text{C NMR}$** (100 MHz, CDCl_3): δ = 212.3, 171.9, 134.1, 131.7, 127.6, 121.6, 104.5, 102.1, 30.2, 28.3, 22.2, 16.3, 13.8.

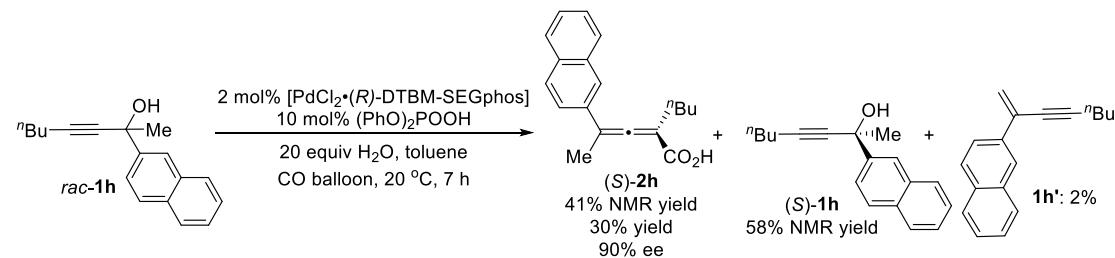
(8) Preparation of (*S*)-2-butyl-4-(4-(methoxycarbonyl)phenyl)-2,3-pentadienoic acid ((*S*)-2g) (wj-1-032-2)



Following Typical Procedure II, the reaction of $[\text{PdCl}_2\bullet(R)\text{-DTBM-SEGphos}]$ (13.5 mg, 0.01 mmol), $(\text{PhO})_2\text{PO}_2\text{H}$ (12.5 mg, 0.05 mmol), **rac-1g** (130.0 mg, 0.5 mmol), and H_2O (180 μL , d = 1.0 g/mL, 180.0 mg, 10 mmol) in toluene (2.5 mL) afforded the product **(S)-2g** (74.6 mg, 52%) (52% NMR yield of **(S)-2g** was formed with 48% of

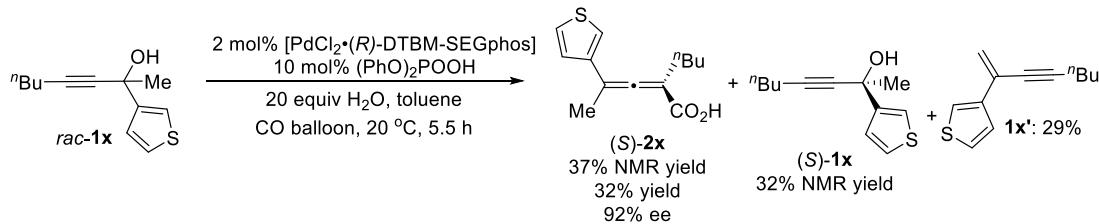
(*S*)-**1g** remained based on ^1H NMR analysis of the crude product [eluent: petroleum ether / ethyl ether / dichloromethane = 30/1/1 (480 mL), then petroleum ether / ethyl acetate = 20/1 (840 mL), 10/1(550 mL)]: 96% ee (HPLC conditions: AS-H column, hexane/ $i\text{PrOH}$ = 95/5, 1.0 mL/min, λ = 214 nm, t_{R} (major) = 13.9 min, t_{R} (minor) = 17.4 min); solid; **1H NMR** (400 MHz, CDCl_3): δ = 8.00 (d, J = 8.4 Hz, 2 H, Ar-H), 7.44 (d, J = 8.0 Hz, 2 H, Ar-H), 3.92 (s, 3 H, OCH_3), 2.34 (t, J = 7.4 Hz, 2 H, CH_2), 2.21 (s, 3 H, CH_3), 1.52-1.41 (m, 2 H, CH_2), 1.40-1.28 (m, 2 H, CH_2), 0.88 (t, J = 7.2 Hz, 3 H, CH_3); **^{13}C NMR** (100 MHz, CDCl_3): δ = 213.1, 172.3, 166.8, 139.9, 129.8, 129.0, 125.9, 104.8, 102.3, 52.1, 30.1, 28.2, 22.2, 16.2, 13.8.

(9) Preparation of (*S*)-2-butyl-4-(2-naphthyl)-2,3-pentadienoic acid ((*S*)-**2h**) (wj-1-047-2)



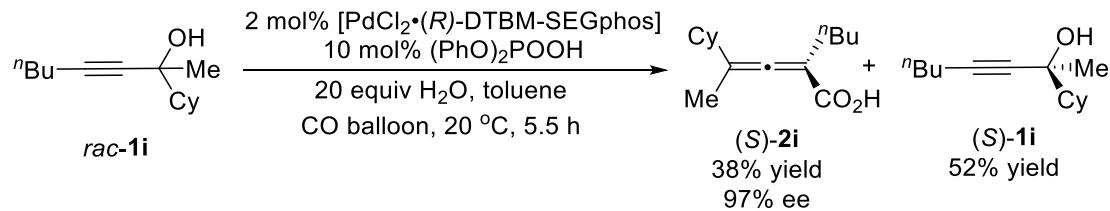
Following Typical Procedure II, the reaction of $[\text{PdCl}_2\bullet(R)\text{-DTBM-SEGphos}]$ (13.5 mg, 0.01 mmol), $(\text{PhO})_2\text{PO}_2\text{H}$ (12.5 mg, 0.05 mmol), *rac*-**1h** (127.0 mg, 0.5 mmol), and H_2O (180 μL , d = 1.0 g/mL, 180.0 mg, 10 mmol) in toluene (2.5 mL) afforded the product (*S*)-**2h** (42.5 mg, 30%) (41% NMR yield of (*S*)-**2h** and 2% of **1h'** were formed with 58% of (*S*)-**1h** remained based on ^1H NMR analysis of the crude product) [eluent: petroleum ether / ethyl ether / dichloromethane = 30/1/1 (480 mL), petroleum ether / ethyl acetate = 10/1 (660 mL)]: 90% ee (HPLC conditions: AS-H column, hexane/ $i\text{PrOH}$ = 95/5, 1.0 mL/min, λ = 214 nm, t_{R} (major) = 6.0 min, t_{R} (minor) = 7.3 min); solid; **1H NMR** (400 MHz, CDCl_3): δ = 7.86-7.64 (m, 4 H, Ar-H), 7.55-7.34 (m, 3 H, Ar-H), 2.37 (t, J = 7.4 Hz, 2 H, CH_2), 2.30 (s, 3 H, CH_3), 1.56-1.44 (m, 2 H, CH_2), 1.43-1.31 (m, 2 H, CH_2), 0.88 (t, J = 7.2 Hz, 3 H, CH_3); **^{13}C NMR** (100 MHz, CDCl_3): δ = 213.2, 172.7, 133.5, 132.8, 132.4, 128.09, 128.06, 127.6, 126.3, 126.1, 124.8, 124.2, 105.5, 102.1, 30.2, 28.4, 22.3, 16.3, 13.8.

(10) Preparation of (*S*)-2-butyl-4-(thiophen-3-yl)-2,3-pentadienoic acid ((*S*)-2x) (wj-1-036-2)



Following Typical Procedure II, the reaction of $[\text{PdCl}_2\bullet(\text{R})\text{-DTBM-SEGphos}]$ (13.7 mg, 0.01 mmol), $(\text{PhO})_2\text{PO}_2\text{H}$ (12.4 mg, 0.05 mmol), *rac-1x* (104.5 mg, 0.5 mmol), and H_2O (180 μL , d = 1.0 g/mL, 180.0 mg, 10 mmol) in toluene (2.5 mL) afforded the product (*S*)-2x (37.6 mg, 32%) (37% NMR yield of (*S*)-2x and 29% of *1x'* were formed with 32% of (*S*)-1x remained based on ^1H NMR analysis of the crude product) [eluent: petroleum ether / ethyl ether / dichloromethane = 30/1/1 (480 mL), then petroleum ether / ethyl acetate = 10/1 (440 mL)]: 92% ee (HPLC conditions: AS-H column, hexane/ $i\text{PrOH}$ = 98/2, 1.0 mL/min, λ = 214 nm, t_{R} (major) = 11.7 min, t_{R} (minor) = 16.3 min); solid; **$^1\text{H NMR}$** (400 MHz, CDCl_3): δ = 7.31-7.22 (m, 1 H, one proton of thiaryl), 7.18-7.12 (m, 1 H, one proton of thiaryl), 7.04 (d, J = 4.8 Hz, 1 H, one proton of thiaryl), 2.31 (t, J = 7.6 Hz, 2 H, CH_2), 2.17 (s, 3 H, CH_3), 1.53-1.41 (m, 2 H, CH_2), 1.40-1.30 (m, 2 H, CH_2), 0.88 (t, J = 7.2 Hz, 3 H, CH_3); **$^{13}\text{C NMR}$** (100 MHz, CDCl_3): δ = 212.8, 172.3, 136.6, 126.3, 125.9, 120.6, 101.4, 101.3, 30.3, 28.4, 22.2, 16.7, 13.8.

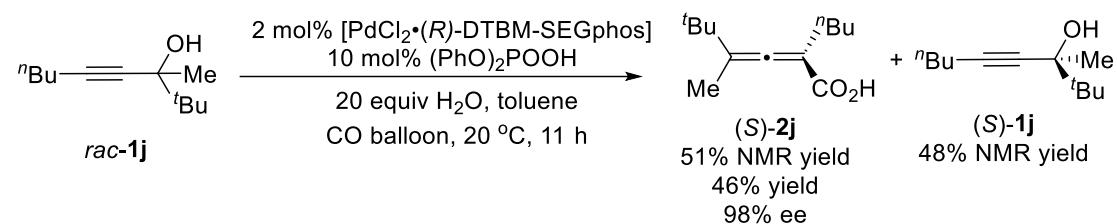
(11) Preparation of (*S*)-2-butyl-4-cyclohexyl-2,3-pentadienoic acid ((*S*)-2i) (wj-1-050-2)



Following Typical Procedure II, the reaction of $[\text{PdCl}_2\bullet(\text{R})\text{-DTBM-SEGphos}]$ (13.7 mg, 0.01 mmol), $(\text{PhO})_2\text{PO}_2\text{H}$ (12.4 mg, 0.05 mmol), *rac-1i* (104.5 mg, 0.5 mmol), and H_2O (180 μL , d = 1.0 g/mL, 180.0 mg, 10 mmol) in toluene (2.5 mL) afforded the product (*S*)-2i (45.6 mg, 38%) [eluent: petroleum ether / diethyl ether / dichloromethane

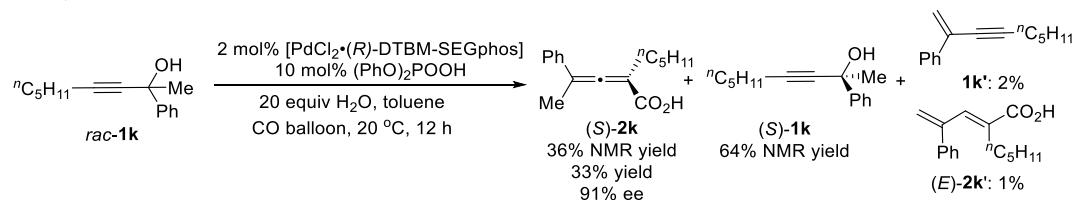
= 30/1/1 (480 mL), then petroleum ether / ethyl acetate = 10/1 (330 mL): 97% ee (HPLC conditions: AS-H column, hexane/*i*PrOH = 98/2, 1.0 mL/min, λ = 214 nm, t_R (major) = 5.4 min, t_R (minor) = 7.2 min); oil; **1H NMR** (400 MHz, CDCl₃): δ = 2.27-2.10 (m, 2 H, CH₂), 1.92-1.81 (m, 2 H, CH₂), 1.81-1.70 (m, 5 H, CH₂ and CH₃), 1.70-1.61 (m, 1 H, CH), 1.47-1.03 (m, 10 H, 5 x CH₂), 0.90 (t, J = 7.0 Hz, 3 H, CH₃); **13C NMR** (100 MHz, CDCl₃): δ = 208.6, 174.0, 109.3, 99.9, 41.8, 31.8, 31.6, 30.4, 28.1, 26.32, 26.26, 26.2, 22.3, 16.4, 13.9.

(12) Preparation of (*S*)-2-butyl-4,5,5-trimethyl-2,3-hexadienoic acid ((*S*)-2j) (wj-1-055-2)



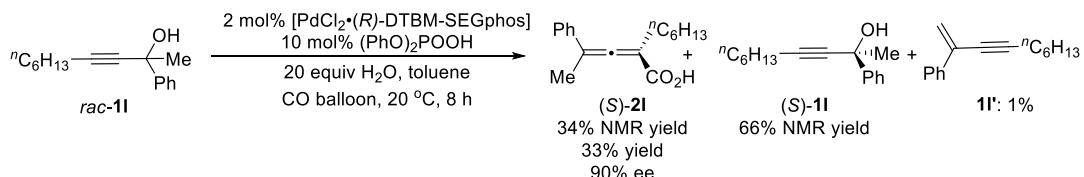
Following Typical Procedure II, the reaction of [PdCl₂•(*R*)-DTBM-SEGphos] (13.6 mg, 0.01 mmol), (PhO)₂PO₂H (12.5 mg, 0.05 mmol), *rac*-1j (92.4 mg, 0.5 mmol), and H₂O (180 μ L, d = 1.0 g/mL, 180.0 mg, 10 mmol) in toluene (2.5 mL) afforded the product (*S*)-2j (48.6 mg, 46%) (51% NMR yield of (*S*)-2j with 48% of (*S*)-1j remained based on 1H NMR analysis of the crude product) [eluent: petroleum ether / ethyl ether / dichloromethane = 30/1/1 (320 mL), then petroleum ether / ethyl acetate = 10/1 (330 mL): 98% ee (HPLC conditions: AD-H column, hexane/*i*PrOH = 98/2, 1.0 mL/min, λ = 214 nm, t_R (minor) = 7.0 min, t_R (major) = 7.5 min); solid; **1H NMR** (400 MHz, CDCl₃): δ = 2.25-2.10 (m, 2 H, CH₂), 1.77 (s, 3 H, CH₃), 1.48-1.29 (m, 4 H, 2 x CH₂), 1.10 (s, 9 H, 3 x CH₃), 0.90 (t, J = 7.0 Hz, 3 H, CH₃); **13C NMR** (100 MHz, CDCl₃): δ = 208.1, 174.2, 112.9, 99.5, 34.2, 30.4, 28.8, 28.0, 22.3, 14.0, 13.9.

(13) Preparation of (*S*)-2-pentyl-4-phenyl-2,3-pentadienoic acid ((*S*)-2k) (wj-1-046-2)



Following Typical Procedure II, the reaction of [PdCl₂•(R)-DTBM-SEGphos] (13.6 mg, 0.01 mmol), (PhO)₂PO₂H (12.5 mg, 0.05 mmol), *rac*-**1k** (107.4 mg, 0.5 mmol), and H₂O (180 μL, d = 1.0 g/mL, 180.0 mg, 10 mmol) in toluene (2.5 mL) afforded the product (*S*)-**2k** (40.6 mg, 33%) (36% NMR yield of (*S*)-**2k**, 2% of **1k'**, and 1% of (*E*)-**2k'** were formed with 64% of (*S*)-**1k** remained based on ¹H NMR analysis of the crude product) [eluent: petroleum ether / ethyl ether / dichloromethane = 30/1/1 (480 mL), then petroleum ether / ethyl acetate = 10/1 (440 mL)]: 91% ee (HPLC conditions: AS-H column, hexane/ⁱPrOH = 98/2, 1.0 mL/min, λ = 214 nm, *t*_R (major) = 7.8 min, *t*_R (minor) = 12.4 min); solid; **1H NMR** (400 MHz, CDCl₃): δ = 7.43-7.30 (m, 4 H, Ar-H), 7.29-7.23 (m, 1 H, Ar-H), 2.32 (t, *J* = 7.6 Hz, 2 H, CH₂), 2.20 (s, 3 H, CH₃), 1.54-1.42 (m, 2 H, CH₂), 1.35-1.22 (m, 4 H, 2 x CH₂), 0.84 (t, *J* = 6.8 Hz, 3 H, CH₃); **13C NMR** (100 MHz, CDCl₃): δ = 212.6, 172.7, 135.1, 128.5, 127.6, 126.1, 105.2, 101.8, 31.3, 28.6, 27.7, 22.4, 16.3, 14.0.

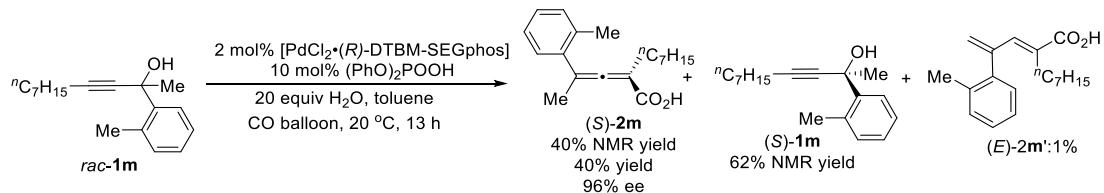
(14) Preparation of (*S*)-2-hexyl-4-phenyl-2,3-pentadienoic acid ((*S*)-2l**) (wj-1-094-2)**



Following Typical Procedure II, the reaction of [PdCl₂•(R)-DTBM-SEGphos] (13.7 mg, 0.01 mmol), (PhO)₂PO₂H (12.6 mg, 0.05 mmol), *rac*-**1l** (115.0 mg, 0.5 mmol), and H₂O (180 μL, d = 1.0 g/mL, 180.0 mg, 10 mmol) in toluene (2.5 mL) afforded the product (*S*)-**2l** (42.0 mg, 33%) (34% NMR yield of (*S*)-**2l** and 1% of **1l'** were formed with 66% of (*S*)-**1l** remained based on ¹H NMR analysis of the crude product) [eluent: petroleum ether / ethyl ether / dichloromethane = 30/1/1 (448 mL), then petroleum ether / ethyl acetate = 10/1 (660 mL)]: 90% ee (HPLC conditions: AS-H column, hexane/ⁱPrOH = 98/2, 1.0 mL/min, λ = 214 nm, *t*_R (major) = 7.5 min, *t*_R (minor) = 13.4 min); solid; **1H NMR** (400 MHz, CDCl₃): δ = 7.44-7.29 (m, 4 H, Ar-H), 7.28-7.22 (m, 1 H, Ar-H), 2.32 (t, *J* = 7.4 Hz, 2 H, CH₂), 2.19 (s, 3 H, CH₃), 1.55-1.41 (m, 2 H, CH₂), 1.37-1.18 (m, 6 H, 3 x CH₂), 0.84 (t, *J* = 6.6 Hz, 3 H, CH₃); **13C NMR** (100 MHz,

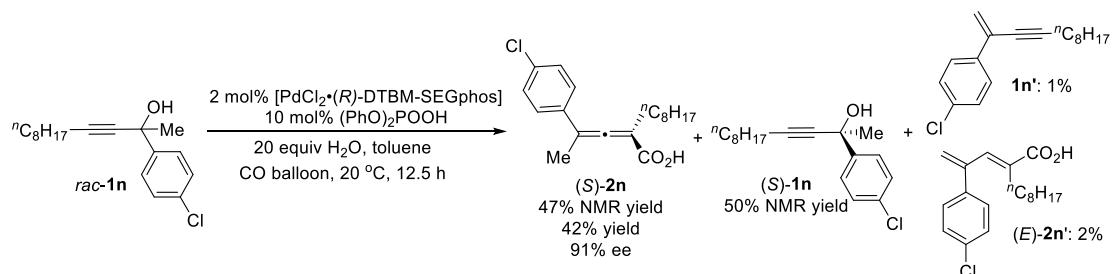
CDCl_3): $\delta = 212.5, 172.5, 135.0, 128.5, 127.6, 126.1, 105.2, 101.8, 31.6, 28.8, 28.6, 28.0, 22.6, 16.3, 14.0$.

**(15) Preparation of (*S*)-2-heptyl-4-(2-methylphenyl)-2,3-pentadienoic acid ((*S*)-2m)
(wj-1-070-2)**



Following Typical Procedure II, the reaction of $[\text{PdCl}_2^\bullet(\text{R})\text{-DTBM-SEGphos}]$ (13.5 mg, 0.01 mmol), $(\text{PhO})_2\text{PO}_2\text{H}$ (12.4 mg, 0.05 mmol), **rac-1m** (129.6 mg, 0.5 mmol), and H_2O (180 μL , d = 1.0 g/mL, 180.0 mg, 10 mmol) in toluene (2.5 mL) afforded the product **(S)-2m** (56.9 mg, 40%) (40% NMR yield of **(S)-2m** and 2% of **(E)-2m'**) were formed with 62% of **(S)-1m** remained based on ^1H NMR analysis of the crude product) [eluent: petroleum ether / ethyl ether / dichloromethane = 30/1/1 (~420 mL), then petroleum ether / ethyl acetate = 8/1 (360 mL)]: 96% ee (HPLC conditions: AS-H column, hexane/ $i\text{PrOH}$ = 98/2, 1.0 mL/min, $\lambda = 214 \text{ nm}$, t_R (major) = 6.2 min, t_R (minor) = 10.3 min); oil; **$^1\text{H NMR}$** (400 MHz, CDCl_3): $\delta = 7.29\text{-}7.22$ (m, 1 H, Ar-H), 7.21-7.10 (m, 3 H, Ar-H), 2.40 (s, 3 H, CH_3), 2.34-2.15 (m, 2 H, CH_2), 2.13 (s, 3 H, CH_3), 1.54-1.40 (m, 2 H, CH_2), 1.35-1.15 (m, 8 H, 4 x CH_2), 0.87 (t, $J = 6.8 \text{ Hz}$, 3 H, CH_3); **$^{13}\text{C NMR}$** (100 MHz, CDCl_3): $\delta = 210.2, 173.5, 136.3, 136.0, 130.6, 127.9, 127.6, 126.0, 104.6, 98.9, 31.8, 29.11, 29.06, 28.4, 28.0, 22.6, 20.3, 19.9, 14.1$.

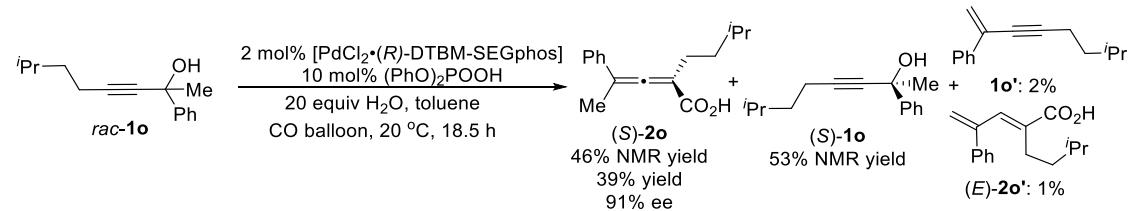
**(16) Preparation of (*S*)-2-octyl-4-(4-chlorophenyl)-2,3-pentadienoic acid ((*S*)-2n)
(wj-1-074-2)**



Following Typical Procedure II, the reaction of $[\text{PdCl}_2^\bullet(\text{R})\text{-DTBM-SEGphos}]$

(13.6 mg, 0.01 mmol), $(\text{PhO})_2\text{PO}_2\text{H}$ (12.4 mg, 0.05 mmol), *rac*-**1n** (146.1 mg, 0.5 mmol), and H_2O (180 μL , d = 1.0 g/mL, 180.0 mg, 10 mmol) in toluene (2.5 mL) afforded the product (*S*)-**2n** (68.0 mg, 42%) (47% NMR yield of (*S*)-**2n**, 1% of **1n'**, and 2% of (*E*)-**2n'** were formed with 50% of (*S*)-**1n** remained based on ^1H NMR analysis of the crude product) [eluent: petroleum ether / ethyl ether / dichloromethane = 30/1/1 (480 mL), then petroleum ether / ethyl acetate = 8/1 (630 mL)]: 91% ee (HPLC conditions: AS-H column, hexane/ $i\text{PrOH}$ = 98/2, 1.0 mL/min, λ = 214 nm, t_R (major) = 6.4 min, t_R (minor) = 9.8 min); solid; **1H NMR** (400 MHz, CDCl_3): δ = 7.30 (s, 4 H, Ar-H), 2.31 (t, J = 7.4 Hz, 2 H, CH_2), 2.16 (s, 3 H, CH_3), 1.53-1.40 (m, 2 H, CH_2), 1.34-1.17 (m, 10 H, 5 x CH_2), 0.86 (t, J = 7.0 Hz, 3 H, CH_3); **13C NMR** (100 MHz, CDCl_3): δ = 212.5, 172.5, 133.6, 133.4, 128.7, 127.3, 104.4, 102.1, 31.8, 29.3, 29.2, 29.1, 28.5, 28.0, 22.6, 16.3, 14.0.

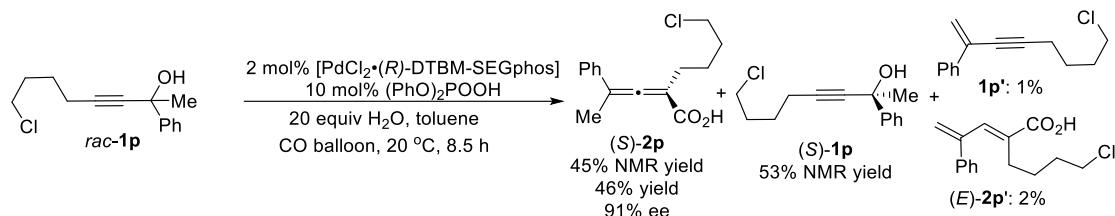
(17) Preparation of (*S*)-2-(3-methylbutyl)-4-phenyl-2,3-pentadienoic acid ((*S*)-**2o**) (wj-1-066-2)



Following Typical Procedure II, the reaction of $[\text{PdCl}_2\bullet(\text{R})\text{-DTBM-SEGphos}]$ (13.7 mg, 0.01 mmol), $(\text{PhO})_2\text{PO}_2\text{H}$ (12.6 mg, 0.05 mmol), *rac*-**1o** (107.9 mg, 0.5 mmol), and H_2O (180 μL , d = 1.0 g/mL, 180.0 mg, 10 mmol) in toluene (2.5 mL) afforded the product (*S*)-**2o** (47.3 mg, 39%) (46% NMR yield of (*S*)-**2o**, 2% of **1o'**, and 1% of (*E*)-**2o'** were formed with 53% of (*S*)-**1o** remained based on ^1H NMR analysis of the crude product) [eluent: petroleum ether / ethyl ether / dichloromethane = 30/1/1 (384 mL), then petroleum ether / ethyl acetate = 10/1 (660 mL)]: 91% ee (HPLC conditions: AS-H column, hexane/ $i\text{PrOH}$ = 98/2, 1.0 mL/min, λ = 214 nm, t_R (major) = 7.8 min, t_R (minor) = 11.0 min); solid; **1H NMR** (400 MHz, CDCl_3): δ = 7.42-7.29 (m, 4 H, Ar-H), 7.29-7.21 (m, 1 H, Ar-H), 2.33 (t, J = 7.8 Hz, 2 H, CH_3), 2.19 (s, 3 H, CH_3), 1.65-1.52 (m, 1 H, CH), 1.40-1.31 (m, 2 H, CH_2), 0.87 (t, J = 6.0 Hz, 6 H, 2 x CH_3);

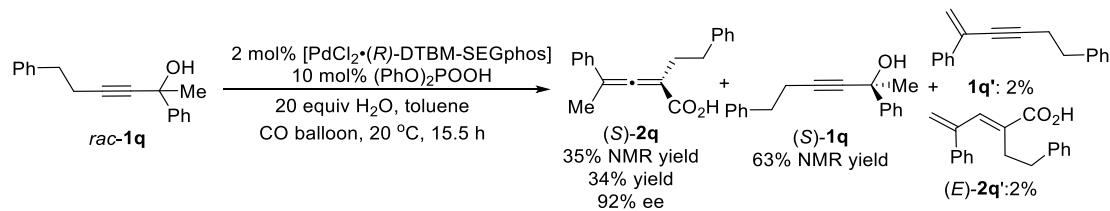
¹³C NMR (100 MHz, CDCl₃): δ = 212.5, 172.8, 135.0, 128.5, 127.6, 126.1, 105.2, 102.0, 37.1, 27.6, 26.6, 22.44, 22.40, 16.3.

(18) Preparation of (S)-2-(4-chlorobutyl)-4-phenyl-2,3-pentadienoic acid ((S)-2p) (wj-1-064-2)



Following Typical Procedure II, the reaction of [PdCl₂•(R)-DTBM-SEGphos] (13.6 mg, 0.01 mmol), (PhO)₂PO₂H (12.4 mg, 0.05 mmol), *rac*-1p (117.7 mg, 0.5 mmol), and H₂O (180 μL, d = 1.0 g/mL, 180.0 mg, 10 mmol) in toluene (2.5 mL) afforded the product (S)-2p (60.2 mg, 46%) (45% NMR yield of (S)-2p, 1% of 1p', and 2% of (E)-2p' were formed with 53% of (S)-1p remained based on ¹H NMR analysis of the crude product) [eluent: petroleum ether / ethyl ether / dichloromethane = 30/1/1 (960 mL), then petroleum ether / ethyl acetate = 8/1 (900 mL)]; 91% ee (HPLC conditions: AS-H column, hexane/ⁱPrOH = 95/5, 1.0 mL/min, λ = 214 nm, t_R (major) = 8.0 min, t_R (minor) = 10.7 min); solid; **¹H NMR** (400 MHz, CDCl₃): δ = 7.49-7.30 (m, 4 H, Ar-H), 7.30-7.22 (m, 1 H, Ar-H), 3.50 (t, J = 6.6 Hz, 2 H, CH₂), 2.36 (t, J = 7.4 Hz, 2 H, CH₂), 2.20 (s, 3 H, CH₃), 1.88-1.75 (m, 2 H, CH₂), 1.70-1.58 (m, 2 H, CH₂); **¹³C NMR** (100 MHz, CDCl₃): δ = 212.5, 172.5, 134.7, 128.6, 127.8, 126.1, 105.7, 101.1, 44.6, 32.0, 27.8, 25.3, 16.3.

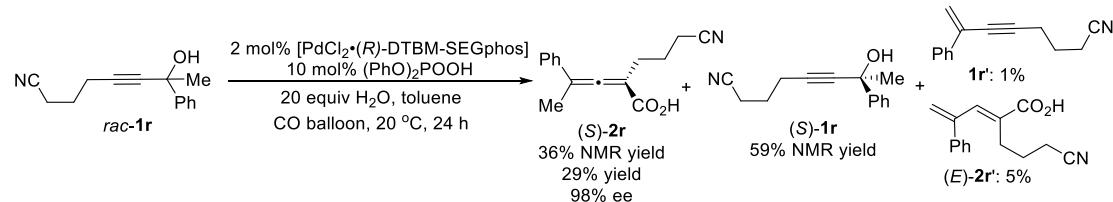
(19) Preparation of (S)-2-phenethyl-4-phenyl-2,3-pentadienoic acid ((S)-2q) (wj-1-056-2)



Following Typical Procedure II, the reaction of [PdCl₂•(R)-DTBM-SEGphos] (13.7 mg, 0.01 mmol), (PhO)₂PO₂H (12.5 mg, 0.05 mmol), *rac*-1q (125.1 mg, 0.5

mmol), and H₂O (180 μL, d = 1.0 g/mL, 180.0 mg, 10 mmol) in toluene (2.5 mL) afforded the product (*S*)-**2q** (47.5 mg, 34%) (35% NMR yield of (*S*)-**2q**, 2% of **1q'**, and 2% of (*E*)-**2p'** were formed with 63% of (*S*)-**1q** remained based on ¹H NMR analysis of the crude product) [eluent: petroleum ether / ethyl ether / dichloromethane = 22/1/1 (480 mL), then petroleum ether / ethyl acetate = 8/1 (450 mL)]: 92% ee (HPLC conditions: AS-H column, hexane/ⁱPrOH = 98/2, 1.0 mL/min, λ = 214 nm, *t*_R (major) = 14.3 min, *t*_R (minor) = 24.7 min); solid; **1H NMR** (400 MHz, CDCl₃): δ = 7.35-7.19 (m, 7 H, Ar-H), 7.19-7.11 (m, 3 H, Ar-H), 2.83 (t, *J* = 7.4 Hz, 2 H, CH₃), 2.75-2.60 (m, 2 H, CH₂), 2.02 (s, 3 H, CH₃); **13C NMR** (100 MHz, CDCl₃): δ = 212.9, 172.5, 141.1, 134.7, 128.5, 128.3, 127.6, 126.1, 125.9, 105.6, 100.7, 34.1, 30.3, 16.1.

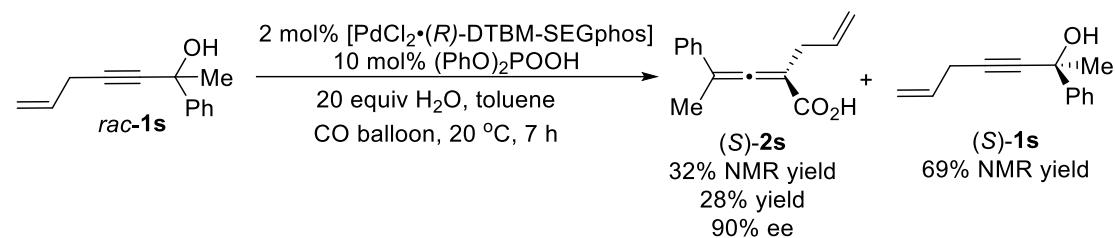
(20) Preparation of (*S*)-2-(3-cyanopropyl)-4-phenyl-2,3-pentadienoic acid ((*S*)-**2r**) (wj-1-073-2)



To a Schlenk flask (25 mL) were added [PdCl₂•(R)-DTBM-SEGphos] (13.6 mg, 0.01 mmol) and (PhO)₂PO₂H (12.5 mg, 0.05 mmol). After addition, the flask was degassed and refilled with Ar for three times to ensure the complete exclusion of air. Then *rac*-**1r** (106.4 mg, 0.5 mmol)/toluene (1.5 mL) and H₂O (180 μL, d = 1.0 g/mL, 180.0 mg, 10 mmol)/toluene (1.0 mL) were added sequentially. After that, the Ar gas line was closed. The resulting mixture was then frozen with a liquid nitrogen bath, degassed to remove the argon inside completely, and refilled with CO by a balloon of CO (about 1 L) for three times. Then the liquid nitrogen bath was removed and the resulting mixture was allowed to stand until completely thawed, vigorously stirred at 20 °C with a balloon of CO for 24 h, warmed up to room temperature. And the crude product treated with H₂O₂ (20 μL, d = 1.13 g/mL, 30 wt. % in H₂O) for 30 min before stopping the reaction, diluted with 5 mL of ethyl acetate, filtered through a short column silica gel (3 cm) eluted with ethyl acetate (20 mL), and concentrated. The crude product

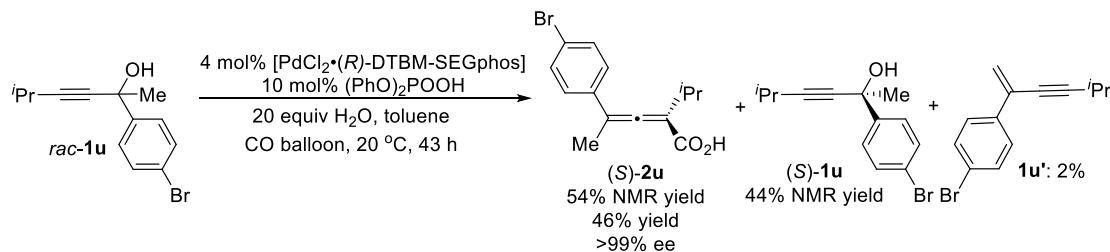
was analyzed by ^1H NMR with CH_2Br_2 (35 μL , 2.477 g/mL, 0.5 mmol) as the internal standard: 36% NMR yield of (*S*)-**2r**, 1% of **1r'**, and 5% of (*E*)-**2r'** were formed with 59% of (*S*)-**1r** remained. The residue was purified by chromatography on silica gel to afford the product (*S*)-**2r** (35.8 mg, 29%, purity: 97%) [eluent: petroleum ether / ethyl acetate = 6/1 (490 mL), 5/1 (1080 mL)]: 98% ee (HPLC conditions: AS-H column, hexane/ $i\text{PrOH}$ = 90/10, 1.0 mL/min, λ = 214 nm, t_{R} (major) = 13.9 min, t_{R} (minor) = 16.7 min); solid; ^1H NMR (400 MHz, CDCl_3): δ = 7.41-7.23 (m[, 5 H, Ar-H], 2.48 (t, J = 7.4 Hz, 2 H, CH_2), 2.35 (t, J = 7.2 Hz, 2 H, CH_2), 2.22 (s, 3 H, CH_3), 1.86 (quint, J = 7.3 Hz, 2 H, CH_2); ^{13}C NMR (100 MHz, CDCl_3): δ = 212.4, 171.9, 134.3, 128.7, 128.0, 126.1, 119.2, 106.4, 99.7, 27.7, 24.0, 16.5, 16.4.

(21) Preparation of (*S*)-2-allyl-4-phenyl-2,3-pentadienoic acid ((*S*)-**2s**) (wj-1-136)



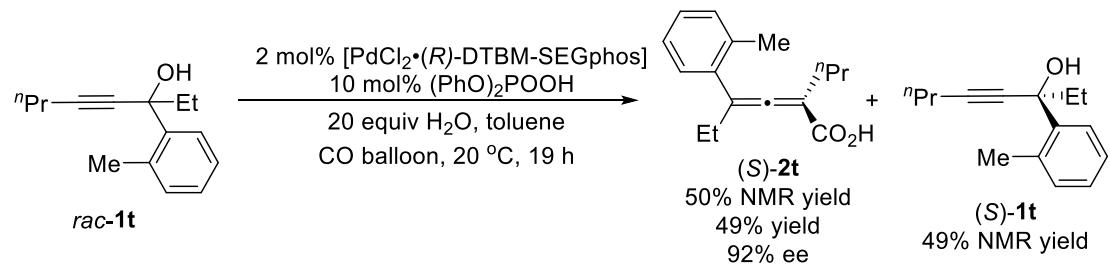
Following Typical Procedure II, the reaction of $[\text{PdCl}_2 \bullet (\text{R})\text{-DTBM-SEGphos}]$ (13.7 mg, 0.01 mmol), $(\text{PhO})_2\text{PO}_2\text{H}$ (12.4 mg, 0.05 mmol), *rac*-**1s** (93.2 mg, 0.5 mmol), and H_2O (180 μL , d = 1.0 g/mL, 180.0 mg, 10 mmol) in toluene (2.5 mL) afforded the product (*S*)-**2s** (29.9 mg, 28%) (32% NMR yield of (*S*)-**2s** was formed with 69% of (*S*)-**1s** remained based on ^1H NMR analysis of the crude product) [eluent: petroleum ether / ethyl acetate = 30/1 (940 mL), 10/1 (440 mL)]: 90% ee (HPLC conditions: AS-H column, hexane/ $i\text{PrOH}$ = 98/2, 1.0 mL/min, λ = 214 nm, t_{R} (major) = 11.1 min, t_{R} (minor) = 14.3 min); solid; ^1H NMR (400 MHz, CDCl_3): δ = 7.47-7.30 (m, 4 H, Ar-H), 7.30-7.22 (m, 1 H, Ar-H), 5.93-5.80 (m, 1 H, $=\text{CH}$), 5.13 (d, J = 17.2 Hz, 1 H, one proton of $=\text{CH}_2$), 5.03 (d, J = 10.0 Hz, 1 H, one proton of $=\text{CH}_2$), 3.09 (d, J = 6.4 Hz, 2 H, CH_2), 2.19 (s, 3 H, CH_3); ^{13}C NMR (100 MHz, CDCl_3): δ = 213.0, 172.2, 134.8, 134.7, 128.6, 127.7, 126.1, 116.5, 105.8, 100.2, 33.1, 16.2.

(22) Preparation of (*S*)-4-(4-bromophenyl)-2-isopropyl-2,3-pentadienoic acid ((*S*)-**2u**) (wj-1-115-2)



Following Typical Procedure II, the reaction of [PdCl₂•(*R*)-DTBM-SEGphos] (27.2 mg, 0.02 mmol), (PhO)₂PO₂H (12.4 mg, 0.05 mmol), *rac-1u* (133.6 mg, 0.5 mmol), and H₂O (180 μL, d = 1.0 g/mL, 180.0 mg, 10 mmol) in toluene (2.5 mL) afforded the product (*S*)-2u (68.3 mg, 46%) (54% NMR yield of (*S*)-2u and 2% of **1u'** were formed with 44% of (*S*)-1u remained based on ¹H NMR analysis of the crude product) [eluent: petroleum ether / ethyl ether / dichloromethane = 50/1/1 (832 mL), 8/1 (450 mL)]: >99% ee (HPLC conditions: AS-H column, hexane/*i*PrOH = 98/2, 1.0 mL/min, λ = 214 nm, *t*_R (major) = 11.5 min); solid; **1H NMR** (400 MHz, CDCl₃): δ = 7.46 (d, *J* = 8.8 Hz, 2 H, Ar-H), 7.25 (d, *J* = 8.8 Hz, 2 H, Ar-H), 2.85–2.74 (m, 1 H, CH), 2.18 (s, 3 H, CH₃), 1.09 (d, *J* = 6.8 Hz, 6 H, 2 × CH₃); **13C NMR** (100 MHz, CDCl₃): δ = 211.3, 172.0, 134.0, 131.7, 127.5, 121.5, 109.0, 105.9, 28.2, 22.13, 22.06, 16.3.

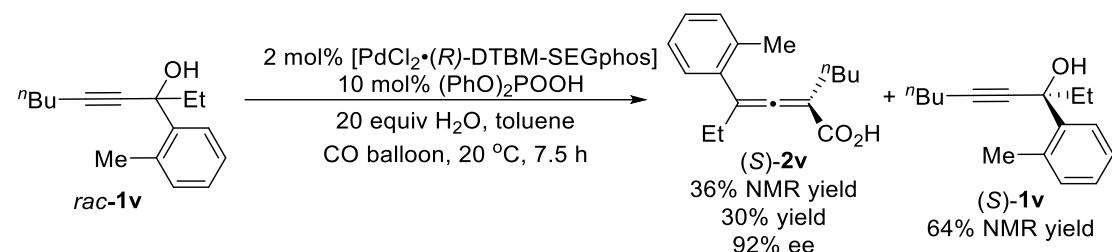
(23) Preparation of (*S*)-2-propyl-4-(2-methylphenyl)-2,3-hexadienoic acid ((*S*)-2t) (wj-1-083-2)



Following Typical Procedure II, the reaction of [PdCl₂•(*R*)-DTBM-SEGphos] (13.5 mg, 0.01 mmol), (PhO)₂PO₂H (12.6 mg, 0.05 mmol), *rac-1t* (108.0 mg, 0.5 mmol), and H₂O (180 μL, d = 1.0 g/mL, 180.0 mg, 10 mmol) in toluene (2.5 mL) afforded the product (*S*)-2t (59.8 mg, 49%) (50% NMR yield of (*S*)-2t was formed with 49% of (*S*)-1t remained based on ¹H NMR analysis of the crude product) [eluent: petroleum ether / ethyl ether / dichloromethane = 60/1/1 (868 mL), then petroleum ether

/ ethyl acetate = 20/1 (420 mL)]: 92% ee (HPLC conditions: AS-H column, hexane/*i*PrOH = 98/2, 1.0 mL/min, λ = 214 nm, t_R (major) = 6.4 min, t_R (minor) = 10.0 min); oil; **1H NMR** (400 MHz, CDCl₃): δ = 7.28-7.10 (m, 4 H, Ar-H), 2.50-2.33 (m, 5 H, CH₂ and CH₃), 2.33-2.12 (m, 2 H, CH₂), 1.60-1.42 (m, 2 H, CH₂), 1.12 (t, J = 7.2 Hz, 3 H, CH₃), 0.93 (t, J = 7.0 Hz, 3 H, CH₃); **13C NMR** (100 MHz, CDCl₃): δ = 209.1, 173.9, 136.1, 136.0, 130.4, 128.4, 127.5, 125.8, 111.2, 100.5, 30.5, 27.1, 21.4, 20.1, 13.8, 12.2.

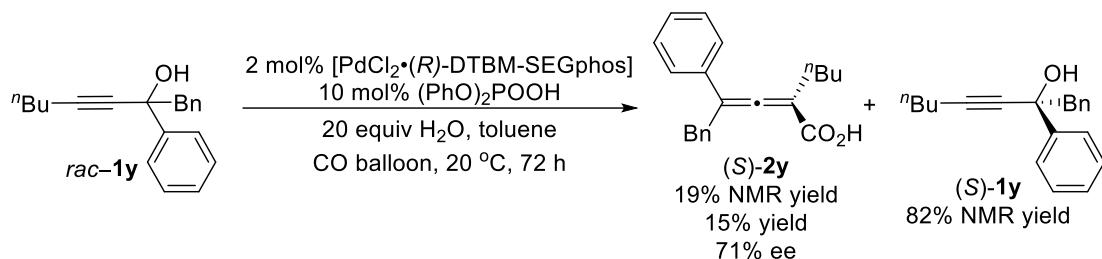
(24) Preparation of (*S*)-2-butyl-4-(2-methylphenyl)-2,3-hexadienoic acid ((*S*)-2v) (wj-1-112-2)



Following Typical Procedure II, the reaction of [PdCl₂•(*R*)-DTBM-SEGphos] (13.5 mg, 0.01 mmol), (PhO)₂PO₂H (12.5 mg, 0.05 mmol), *rac*-1v (115.2 mg, 0.5 mmol), and H₂O (180 μ L, d = 1.0 g/mL, 180.0 mg, 10 mmol) in toluene (2.5 mL) afforded the product (*S*)-2v (38.6 mg, 30%) (36% NMR yield of (*S*)-2v was formed with 64% of (*S*)-1v remained based on ¹H NMR analysis of the crude product) [eluent: petroleum ether / ethyl ether / dichloromethane = 40/1/1 (420 mL), then petroleum ether / ethyl acetate = 10/1 (440 mL)]: 92% ee (HPLC conditions: AS-H column, hexane/*i*PrOH = 98/2, 1.0 mL/min, λ = 214 nm, t_R (major) = 6.3 min, t_R (minor) = 10.5 min); oil; **1H NMR** (400 MHz, CDCl₃): δ = 7.29-7.12 (m, 4 H, Ar-H), 2.48-2.34 (m, 5 H, CH₂ and CH₃), 2.33-2.16 (m, 2 H, CH₂), 1.52-1.42 (m, 2 H, CH₂), 1.40-1.30 (m, 2 H, CH₂), 1.12 (t, J = 7.2 Hz, 3 H, CH₃), 0.90 (t, J = 7.2 Hz, 3 H, CH₃); **13C NMR** (100 MHz, CDCl₃): δ = 209.1, 173.8, 136.06, 135.03, 130.4, 128.4, 127.5, 125.8, 111.3, 100.7, 30.3, 28.1, 27.1, 22.3, 20.1, 13.9, 12.2.

(25) Preparation of (*S*)-2-butyl-4,5-diphenyl-2,3-pentadienoic acid ((*S*)-2y) (wj-1-

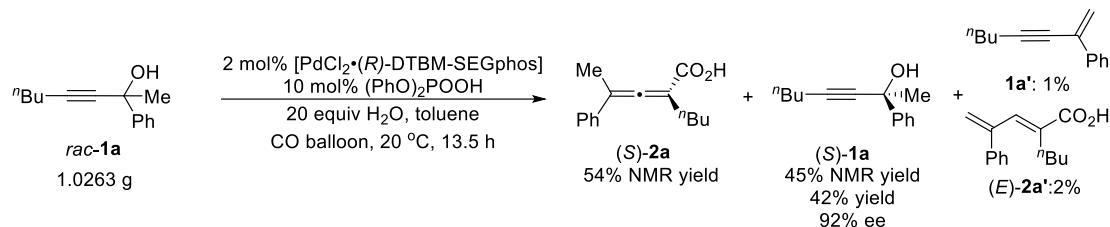
091-2)



Following Typical Procedure II, the reaction of $[\text{PdCl}_2\bullet(R)\text{-DTBM-SEGphos}]$ (13.5 g, 0.01 mmol), $(\text{PhO})_2\text{PO}_2\text{H}$ (12.5 mg, 0.05 mmol), **rac-1y** (138.4 mg, 0.5 mmol), and H_2O (180 μL , d = 1.0 g/mL, 18.0 mg, 10 mmol) in toluene (2.5 mL) afforded the product **(S)-2y** (23.4 mg, 15%) (19% NMR yield of **(S)-2y** was formed with 82% of **(S)-1y** remained based on ^1H NMR analysis of the crude product) [eluent: petroleum ether / ethyl ether / dichloromethane = 30/1/1 (384 mL), then petroleum ether / ethyl acetate = 8/1 (450 mL): 71% ee (HPLC conditions: AS-H column, hexane/ $i\text{PrOH}$ = 98/2, 1.0 mL/min, λ = 214 nm, t_{R} (major) = 10.0 min, t_{R} (minor) = 14.7 min); solid; m.p. 94.1–95.0 $^\circ\text{C}$ (petroleum ether/DCM); **$^1\text{H NMR}$** (400 MHz, CDCl_3): δ = 7.44–7.15 (m, 10 H, Ar-H), 4.00–3.77 (m, 2 H, CH_2), 2.29–2.10 (m, 2 H, CH_2), 1.38–1.19 (m, 4 H, 2 x CH_2), 0.84 (t, J = 6.8 Hz, 3 H, CH_3); **$^{13}\text{C NMR}$** (100 MHz, CDCl_3): δ = 213.5, 172.5, 138.1, 134.3, 128.9, 128.6, 128.3, 127.7, 126.6, 126.5, 109.8, 103.3, 36.9, 30.2, 28.3, 22.3, 13.8; **IR** (neat): ν = 3085, 3000, 2955, 2930, 2866, 2651, 2543, 1937, 1686, 1419, 1281 cm^{-1} ; **MS** (70 eV, EI) m/z (%): 306 (M^+ , 9.32), 91 (100); **HRMS** (EI) calcd for $\text{C}_{21}\text{H}_{22}\text{O}_2$ [M^+]: 306.1614, found: 306.1618.

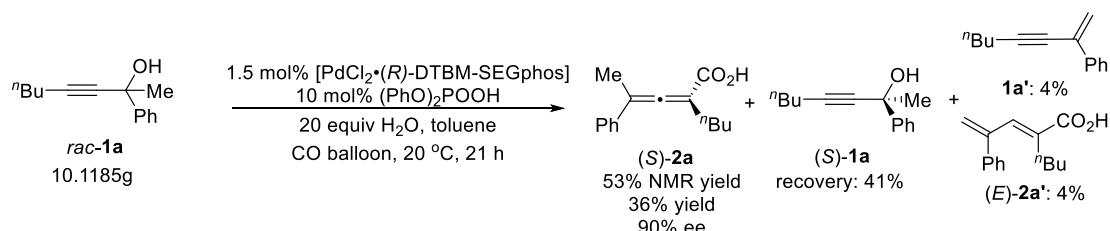
Gram scale reactions

(1) 1-gram scale synthesis preparation of (*S*)-2-phenyloct-3-yn-2-ol ((*S*)-1a) (wj-1-159)



Following Typical Procedure II, the reaction of [PdCl₂•(R)-DTBM-SEGphos] (135.9 mg, 0.1 mmol), (PhO)₂PO₂H (125.2 mg, 0.05 mmol), **rac-1a** (1.0263 g, 5.0 mmol), and H₂O (1.8 mL, d = 1.0 g/mL, 1.8 mg, 100.0 mmol) in toluene (25.0 mL) afforded the product (*S*)-**1a** (427.1 mg, 42%) (54% NMR yield of (*S*)-**2a**, 1% of enyne, and 2% of (*E*)-**2a**' were formed with 45% of (*S*)-**1a** remained based on ¹H NMR analysis of the crude product) [eluent: petroleum ether / ethyl ether / dichloromethane = 30/1/1 (2880 mL), then petroleum ether / ethyl acetate = 4/1 (750 mL)]: 92% ee (HPLC conditions: AS-H column, hexane/¹PrOH = 98/2, 1.0 mL/min, λ = 214 nm, t_R (minor) = 7.0 min, t_R (major) = 10.5 min); oil; **¹H NMR** (400 MHz, CDCl₃): δ = 7.68-7.62 (m, 2 H, Ar-H), 7.38-7.31 (m, 2 H, Ar-H), 7.30-7.24 (m, 1 H, Ar-H), 2.35 (s, 1 H, OH), 2.28 (t, J = 7.0 Hz, 2 H, CH₂), 1.74 (s, 3 H, CH₃), 1.58-1.49 (m, 2 H, CH₂), 1.49-1.38 (m, 2 H, CH₂), 0.93 (t, J = 7.2 Hz, 3 H, CH₃); **¹³C NMR** (100 MHz, CDCl₃): δ = 146.2, 128.2, 127.5, 124.9, 85.6, 83.7, 70.0, 33.5, 30.7, 22.0, 18.4, 13.6.

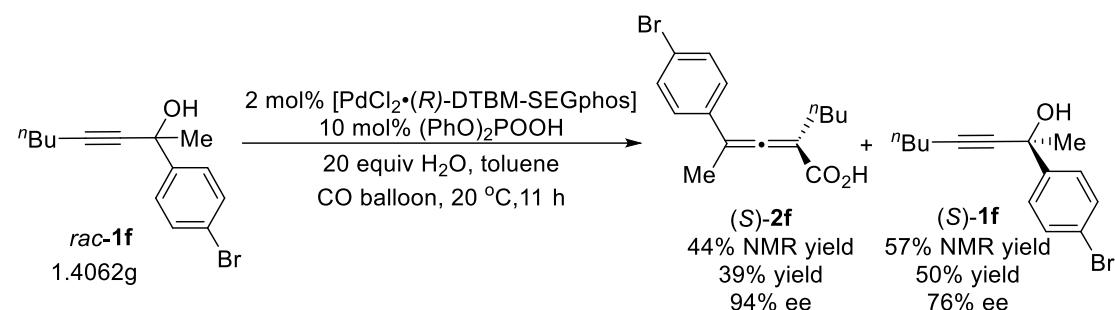
(2) 10-gram scale synthesis preparation of (*S*)-2-butyl-4-phenyl-2,3-pentadienoic acid ((*S*)-2a) (wj-1-178)



Following Typical Procedure II, the reaction of [PdCl₂•(R)-DTBM-SEGphos] (1.0421 g, 0.77 mmol), (PhO)₂PO₂H (1.2533 g, 5.0 mmol), **rac-1a** (10.1185 mg, 50.0 mmol), and H₂O (18.0 mL, d = 1.0 g/mL, 18.0 g, 1.0 mol) in toluene (250 mL) afforded

(*S*)-**2a** (4.2417 g, 36%, purity: 98%) (53% NMR yield of (*S*)-**2a**, 4% of enyne, and 4% of (*E*)-**2a'** were formed with 41% of (*S*)-**1a** remained based on ^1H NMR analysis of the crude product) [eluent: petroleum ether / ethyl ether / dichloromethane = 30/1/1 (4800 mL), then petroleum ether / ethyl acetate = 4/1 (1500 mL)]: 90% ee (HPLC conditions: AS-H column, hexane/*i*PrOH = 98/2, 1.0 mL/min, λ = 214 nm, t_{R} (major) = 8.5 min, t_{R} (minor) = 11.5 min); solid; **1H NMR** (400 MHz, CDCl_3): δ = 7.43-7.30 (m, 4 H, Ar-H), 7.28-7.23 (m, 1 H, Ar-H), 2.33 (t, J = 7.6 Hz, 2 H, CH_2), 2.20 (s, 3 H, CH_3), 1.55-1.42 (m, 2 H, CH_2), 1.41-1.28 (m, 2 H, CH_2), 0.88 (t, J = 7.2 Hz, 3 H, CH_3); **13C NMR** (100 MHz, CDCl_3): δ = 212.5, 172.4, 135.0, 128.6, 127.6, 126.1, 105.2, 101.8, 30.2, 28.3, 22.3, 16.3, 13.8.

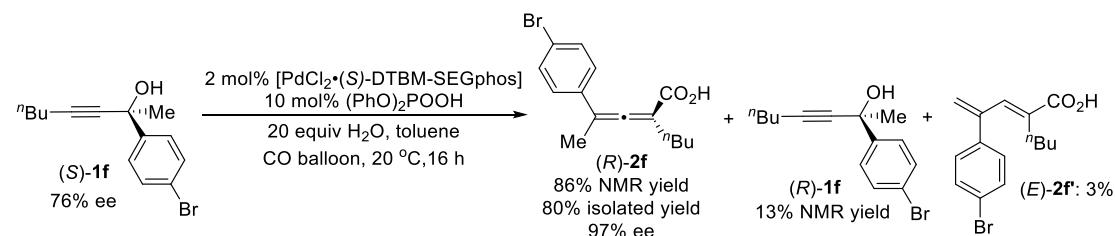
(3) 1-gram scale synthesis preparation of (*S*)-2-butyl-4-(4-bromophenyl)-2,3-pentadienoic acid ((*S*)-**2f**) (wj-2-115)



Following Typical Procedure II, the reaction of [PdCl₂•(R)-DTBM-SEGphos] (135.5 mg, 0.1 mmol), (PhO)₂PO₂H (125.2 mg, 0.5 mmol), **rac-1f** (1.4062 g, 5.0 mmol), and H₂O (1.8 mL, d = 1.0 g/mL, 1.8 g, 100 mmol) in toluene (25 mL) afforded the product (*S*)-**2f** (605.7 mg, 39%) (44% NMR yield of (*S*)-**2f** was formed with 57% of (*S*)-**1f** remained based on ^1H NMR analysis of the crude product) [eluent: petroleum ether / ethyl ether / dichloromethane = 30/1/1 (2400 mL), then petroleum ether / ethyl acetate = 10/1 (1100 mL) to 5/1(840 mL)]: 94% ee (HPLC conditions: AS-H column, hexane/*i*PrOH = 98/2, 1.0 mL/min, λ = 214 nm, t_{R} (major) = 10.5 min, t_{R} (minor) = 15.0 min); solid; **1H NMR** (400 MHz, CDCl_3): δ = 7.50-7.42 (m, 2 H, Ar-H), 7.27-7.20 (m, 2 H, Ar-H), 2.32 (t, J = 7.6 Hz, 2 H, CH_2), 2.17 (s, 3 H, CH_3), 1.49-1.39 (m, 2 H, CH_2), 1.39-1.28 (m, 2 H, CH_2), 0.88 (t, J = 7.2 Hz, 3 H, CH_3); **13C NMR** (100 MHz, CDCl_3):

δ = 212.4, 172.3, 134.1, 131.7, 127.6, 121.6, 104.5, 102.2, 30.2, 28.3, 22.2, 16.3, 13.8. And (*S*)-**1f** (707.1 mg, 50%): 76% ee (HPLC conditions: AS-H column, hexane/*i*PrOH = 98/2, 1.0 mL/min, λ = 214 nm, t_R (minor) = 8.6 min, t_R (major) = 11.3 min); oil; **1H NMR** (400 MHz, CDCl₃): δ = 7.55-7.49 (m, 2 H, Ar-H), 7.49-7.43 (m, 2 H, Ar-H), 2.31 (s, 1 H, OH), 2.27 (t, J = 7.0 Hz, 2 H, CH₂), 1.71 (s, 3 H, CH₃), 1.57-1.48 (m, 2 H, CH₂), 1.48-1.36 (m, 2 H, CH₂), 0.92 (t, J = 7.2 Hz, 3 H, CH₃); **13C NMR** (100 MHz, CDCl₃): δ = 145.3, 131.2, 126.9, 121.4, 86.0, 83.2, 69.6, 33.6, 30.6, 22.0, 18.4, 13.6.

(4) Successive kinetic resolutions preparation of (*R*)-2-butyl-4-(4-bromophenyl)-2,3-pentadienoic acid ((*R*)-**2f**) (wj-2-119)

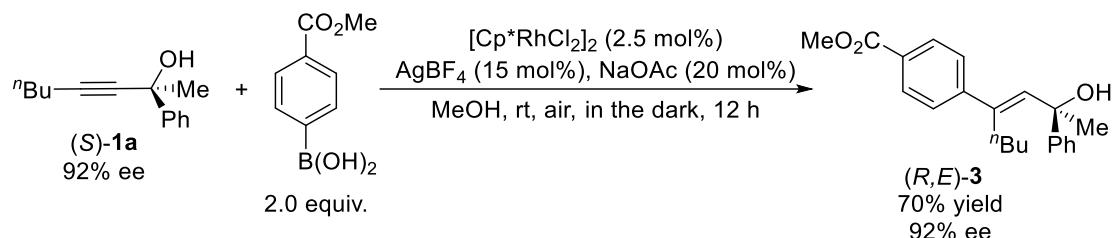


Following Typical Procedure II, the reaction of [PdCl₂•(S)-DTBM-SEGphos] (67.7 mg, 0.05 mmol), (PhO)₂PO₂H (62.6 mg, 0.25 mmol), (S)-**1f** (0.7035 g, 2.5 mmol, 76% ee), and H₂O (0.9 mL, d = 1.0 g/mL, 0.9 g, 50.0 mmol) in toluene (12.5 mL) afforded the product (*R*)-**2f** (615.6 mg, 80%) (86% NMR yield of (*R*)-**2f** and 3% of (*E*)-**2f'** were formed with 13% of (*R*)-**1f** remained based on **1H NMR** analysis of the crude product) [eluent: petroleum ether / ethyl ether / dichloromethane = 30/1/1 (960 mL), then petroleum ether / ethyl acetate = 15/1 (480 mL) to 10/1(550 mL)]: 97% ee (HPLC conditions: AS-H column, hexane/*i*PrOH = 98/2, 1.0 mL/min, λ = 214 nm, t_R (minor) = 10.9 min, t_R (major) = 14.2 min); $[\alpha]_D^{24} = -18.2$ (c = 1.07, CHCl₃); solid; m.p. 124.5-125.3 °C (petroleum ether/DCM); **1H NMR** (400 MHz, CDCl₃): δ = 7.46 (d, J = 8.0 Hz, 2 H, Ar-H), 7.24 (d, J = 8.0 Hz, 2 H, Ar-H), 2.32 (t, J = 7.2 Hz, 2 H, CH₂), 2.17 (s, 3 H, CH₃), 1.52-1.40 (m, 2 H, CH₂), 1.40-1.28 (m, 2 H, CH₂), 0.88 (t, J = 7.0 Hz, 3 H, CH₃); **13C NMR** (100 MHz, CDCl₃): δ = 212.4, 172.6, 134.1, 131.7, 127.6, 121.5, 104.5, 102.2, 30.2, 28.2, 22.2, 16.2, 13.8; **IR** (neat): ν = 3068, 2928, 2866, 2637, 2547, 1941, 1683, 1484, 1416, 1373, 1279, 1248, 1177, 1120, 1075, 1007 cm⁻¹; **MS** (70 eV, EI) *m/z* (%):

310 ($M(^{81}Br)^+$, 2.06), 308 ($M(^{79}Br)^+$, 1.83), 142 (100); Anal. Calcd. for $C_{15}H_{17}BrO_2$: C 58.27, H 5.54; found: C 57.98, H 5.54.

Synthetic transformations

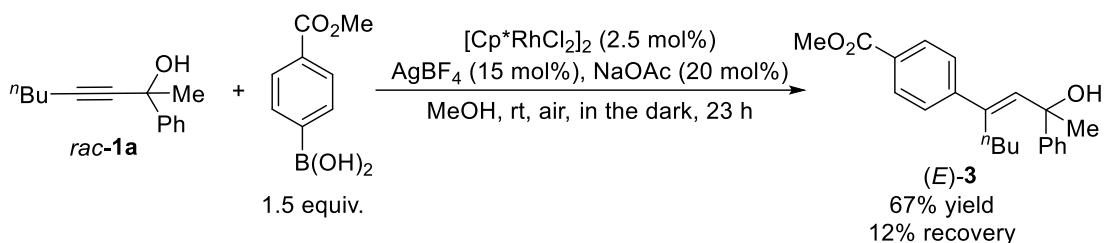
(1) Preparation of (*R,E*)-3-(4-methoxycarbonylphenyl)-2-phenyloct-3-en-2-ol ((*R,E*)-3) (wj-1-163)⁶



Typical Procedure III: To a Schlenk flask covered with aluminium foil paper was added AgBF_4 (14.7 mg, 0.075 mmol) in a glove box. After transferring out of the glove box, $[\text{Cp}^*\text{RhCl}_2]_2$ (7.7 mg, 0.012 mmol), NaOAc (8.4 mg, 0.10 mmol), $(4\text{-MeO}_2\text{C})\text{C}_6\text{H}_4\text{B}(\text{OH})_2$ (180.1 mg, 1.0 mmol), **(S)-1a** (100.9 mg, 0.5 mmol, 92% ee), and MeOH (2.5 mL) were sequentially added to the Schlenk flask without inert atmosphere protection. The resulting mixture was stirred at room temperature as monitored by TLC. After 12 h, the resulting mixture was filtered through a short column of celite (3 cm) eluted with MeOH (20 mL) and concentrated. The residue was purified by column chromatography on silica gel to afford the product **(*R,E*)-3** (118.3 mg, 70%) [eluent: petroleum ether/ethyl acetate = 40/1 (1230 mL) to 10/1 (220 mL)]: 92% ee (HPLC conditions: AS-H column, hexane/*i*PrOH = 95/5, 1.0 mL/min, λ = 214 nm, t_R (major) = 9.6 min, t_R (minor) = 10.8 min); $[\alpha]_D^{25} = -64.4$ ($c = 1.08$, CHCl_3); oil; **1H NMR** (400 MHz, CDCl_3) δ = 7.98 (d, $J = 8.4$ Hz, 2 H, Ar-H), 7.56-51 (m, 2 H, Ar-H), 7.42-7.30 (m, 4 H, Ar-H), 7.28-7.22 (m, 1 H, Ar-H), 6.18 (s, 1 H, =CH), 3.91 (s, 3 H, CH_3), 2.48-2.36 (m, 2 H, CH_2), 2.04 (s, 1 H, OH), 1.74 (s, 3 H, CH_3), 1.07-0.81 (m, 4 H, 2 x CH_2), 0.64 (t, $J = 7.0$ Hz, 3 H, CH_3); **13C NMR** (100 MHz, CDCl_3) δ = 167.0, 148.5, 147.8, 144.3, 137.2, 129.6, 128.8, 128.2, 126.7, 126.7, 125.1, 74.2, 52.0, 33.7, 29.88, 29.85, 22.6, 13.7; **IR** (neat) ν = 3509, 2955, 2928, 2859, 1720, 1706, 1605, 1435, 1276, 1180, 1105 cm^{-1} ; **MS** (ESI) m/z : 361 ($\text{M}+\text{Na}^+$); **HRMS** (ESI) calcd for $\text{C}_{22}\text{H}_{26}\text{O}_3\text{Na}$ [$\text{M}+\text{Na}^+$]: 361.1774, found: 361.1765.

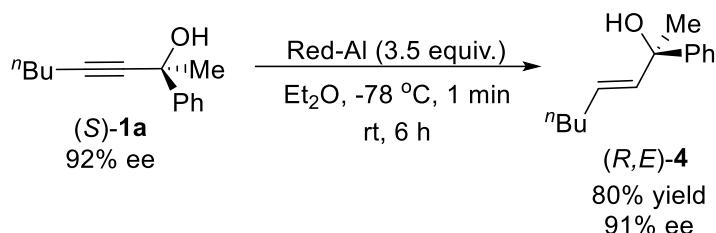
(2) Preparation of (*E*)-3-(4-methoxycarbonylphenyl)-2-phenyloct-3-en-2-ol ((*E*)-3)

(wj-1-139)



Following Typical Procedure III: the reaction of AgBF_4 (14.5 mg, 0.074 mmol), $[\text{Cp}^*\text{RhCl}_2]_2$ (7.6 mg, 0.012 mmol), NaOAc (8.2 mg, 0.10 mmol), (4- MeO_2C) $\text{C}_6\text{H}_4\text{B}(\text{OH})_2$ (135.1 mg, 0.75 mmol), *rac-1a* (101.2 mg, 0.5 mmol), and MeOH (2.5 mL) afforded the product *(E)-3* (112.7 mg, 67%) [eluent: petroleum ether/ethyl acetate = 50/1 (1020 mL) to 10/1 (440 mL)]: oil; **1H NMR** (400 MHz, CDCl_3) δ = 7.98 (d, J = 8.4 Hz, 2 H, Ar-H), 7.61-49 (m, 2 H, Ar-H), 7.44-7.30 (m, 4 H, Ar-H), 7.29-7.23 (m, 1 H, Ar-H), 6.17 (s, 1 H, =CH), 3.91 (s, 3 H, CH_3), 2.53-2.37 (m, 2 H, CH_2), 2.10-2.00 (m, 1 H, OH), 1.74 (s, 3 H, CH_3), 1.06-0.82 (m, 4 H, 2 x CH_2), 0.64 (t, J = 7.0 Hz, 3 H, CH_3); **13C NMR** (100 MHz, CDCl_3) δ = 167.0, 148.5, 147.8, 144.2, 137.3, 129.6, 128.7, 128.1, 126.7, 126.6, 125.1, 74.2, 52.0, 33.7, 29.9, 29.8, 22.6, 13.7; **IR** (neat) ν = 3501, 2955, 2928, 2860, 1720, 1705, 1605, 1435, 1276, 1180, 1105 cm^{-1} ; **MS** (ESI) m/z : 321 (M-OH^+); **HRMS** (ESI) calcd for $\text{C}_{22}\text{H}_{26}\text{O}_3\text{Na}$ [M+Na^+]: 361.1774, found: 361.1780.

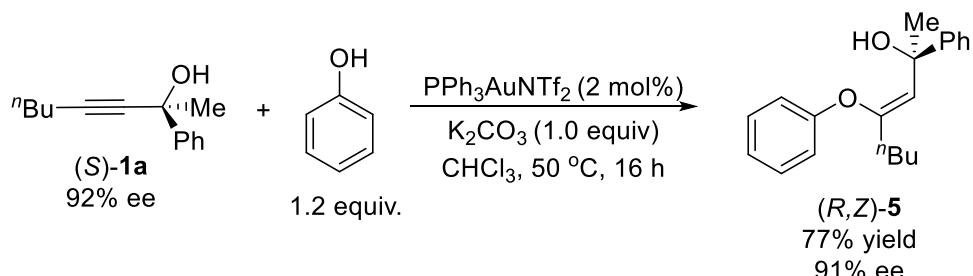
(3) Preparation of (*R,E*)-2-phenyloct-3-en-2-ol ((*R,E*)-4) (wj-1-181)



To a flame-dried Schlenk tube were added (*S*)-1a (40.6 mg, 0.2 mmol, 92% ee) and ethyl ether (1 mL) under argon. The resulting mixture was then cooled down to -78 $^\circ\text{C}$. To the reaction mixture was added a solution of Red-Al (70 wt% in toluene, 0.2 mL, 0.7 mmol) dropwise in 1 minute at -78 $^\circ\text{C}$. Then the reaction mixture was stirred for 6 h at room temperature and subsequently quenched by dropwise addition of MeOH (2

mL) at -78 °C. To the mixture was added a saturated aqueous solution of potassium sodium tartrate (Rochelle's salt) (2 mL). After extraction with ethyl acetate (2 mL x 3), the organic layer was washed with brine (5 mL) and dried over anhydrous Na₂SO₄. After filtration and concentration under reduced pressure, the crude product was purified by column chromatography on silica gel to afford the product (*R,E*)-**4**⁷ (33.0 mg, 80%) [eluent: petroleum ether/ethyl acetate = 40/1 (410 mL)]: 91% ee (HPLC conditions: AS-H column, hexane/ⁱPrOH = 98/2, 1.0 mL/min, λ = 214 nm, t_R (minor) = 6.1 min, t_R (major) = 6.9 min); oil; **1H NMR** (400 MHz, CDCl₃) δ = 7.50-7.43 (m, 2 H, Ar-H), 7.33 (t, J = 7.4 Hz, 2 H, Ar-H), 7.27-7.21 (m, 1 H, Ar-H), 5.77 (d, J = 15.6 Hz, 1 H, =CH), 5.67 (dt, J_1 = 15.6 Hz, J_2 = 6.5 Hz, 1 H, =CH), 2.06 (q, J = 6.8 Hz, 2 H, CH₂), 1.88 (s, 1 H, OH), 1.63 (s, 3 H, CH₃), 1.42-1.25 (m, 4 H, 2 x CH₂), 0.89 (t, J = 7.0 Hz, 3 H, CH₃); **13C NMR** (100 MHz, CDCl₃) δ = 147.3, 136.8, 129.2, 128.1, 126.7, 125.2, 74.4, 31.9, 31.4, 29.9, 22.2, 13.9.

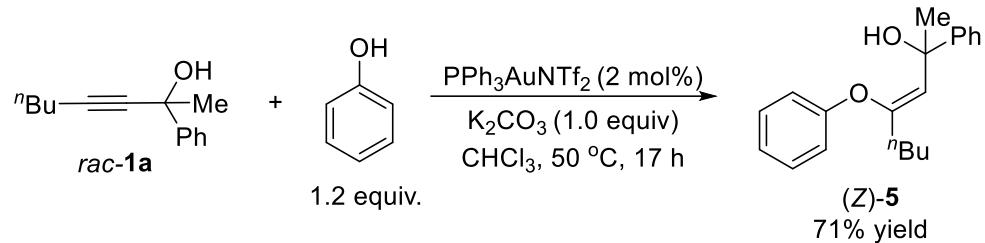
(4) Preparation of (*R,Z*)-4-phenoxy-2-phenyloct-3-en-2-ol ((*R,Z*)-**5**) (wj-1-166)⁸



Typical Procedure IV: To a Schlenk flask were added PPh₃AuNTf₂ (7.5 mg, 0.01 mmol), K₂CO₃ (69.3 mg, 0.5 mmol), and PhOH(58.0 mg, 0.6 mmol). After addition, the flask was degassed and refilled with Ar for three times to ensure the complete exclusion of air. Then (S)-**1a** (101.1 mg, 0.5 mmol, 92% ee) and CHCl₃ (0.3 mL) were added sequentially. The reaction was then stirred at 50 °C as monitored by TLC. After 16 h, the resulting mixture was diluted with 5 mL of ethyl acetate, filtered through a short column silica gel (3 cm) eluted with ethyl acetate (20 mL), and concentrated. The residue was purified by column chromatography on silica gel to afford the product (*R,Z*)-**5** (113.5 mg, 77%) [eluent: petroleum ether/ethyl acetate = 50/1 (510 mL)]: 91% ee (HPLC conditions: IC column, hexane/ⁱPrOH = 99.5/0.5, 0.5 mL/min, λ = 214 nm,

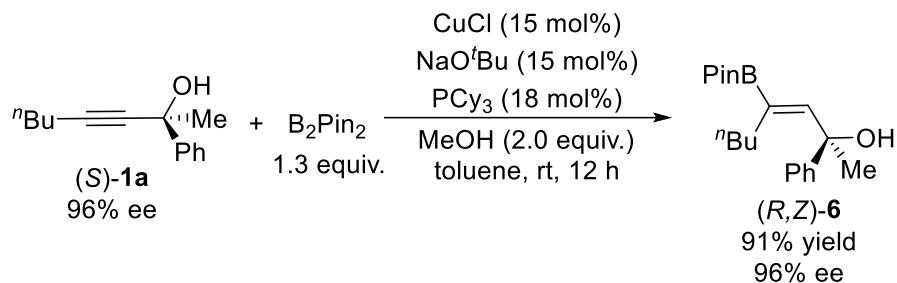
t_R (major) = 22.9 min, t_R (minor) = 24.3 min; $[\alpha]_D^{25} = +9.4$ ($c = 1.36$, CHCl_3); oil; **1H NMR** (400 MHz, CDCl_3) δ = 7.52-7.45 (m, 2 H, Ar-H), 7.31-7.23 (m, 2 H, Ar-H), 7.23-7.13 (m, 3 H, Ar-H), 7.02-6.95 (m, 1 H, Ar-H), 6.80-6.72 (m, 2 H, Ar-H), 5.47 (s, 1 H, =CH), 3.85 (s, 1 H, OH), 2.13-1.98 (m, 2 H, CH_2), 1.65 (s, 3 H, CH_3), 1.40 (quint, $J = 7.5$ Hz, 2 H, CH_2), 1.32-1.21 (m, 2 H, CH_2), 0.84 (t, $J = 7.2$ Hz, 3 H, CH_3); **13C NMR** (100 MHz, CDCl_3) δ = 154.8, 152.3, 149.1, 129.3, 127.9, 126.3, 124.8, 122.8, 121.7, 118.0, 73.6, 31.7, 31.3, 28.9, 21.9, 13.8; **IR** (neat) ν = 3562, 3059, 3027, 2956, 2930, 2871, 1676, 1594, 1489, 1446, 1375, 1329, 1211, 1162, 1089, 1071, 1025 cm^{-1} ; **MS** (ESI) m/z : 319 ($\text{M}+\text{Na}^+$); **HRMS** (ESI) calcd for $\text{C}_{20}\text{H}_{24}\text{O}_2\text{Na}$ [$\text{M}+\text{Na}^+$]: 319.1669, found: 319.1661.

(5) Preparation of (Z)-4-phenoxy-2-phenyloct-3-en-2-ol ((Z)-5) (wj-1-156)



Following Typical Procedure IV, the reaction of $\text{PPh}_3\text{AuNTf}_2$ (8.2 mg, 0.01 mmol), K_2CO_3 (70.4 mg, 0.5 mmol), PhOH (56.9 mg, 0.60 mmol), *rac*-1a (101.6 mg, 0.5 mmol), and CHCl_3 (0.3 mL) afforded the product (Z)-5 (106.2 mg, 71%) [eluent: petroleum ether/ethyl acetate = 50/1 (510 mL)]; oil; **1H NMR** (400 MHz, CDCl_3) δ = 7.53-7.45 (m, 2 H, Ar-H), 7.31-7.23 (m, 2 H, Ar-H), 7.23-7.14 (m, 3 H, Ar-H), 7.02-6.96 (m, 1 H, Ar-H), 6.80-6.73 (m, 2 H, Ar-H), 5.47 (s, 1 H, =CH), 3.86 (s, 1 H, OH), 2.12-1.99 (m, 2 H, CH_2), 1.65 (s, 3 H, CH_3), 1.40 (quint, $J = 7.5$ Hz, 2 H, CH_2), 1.32-1.21 (m, 2 H, CH_2), 0.84 (t, $J = 7.2$ Hz, 3 H, CH_3); **13C NMR** (100 MHz, CDCl_3) δ = 154.8, 152.3, 149.1, 129.3, 127.9, 126.4, 124.8, 122.8, 121.7, 118.0, 73.6, 31.7, 31.3, 28.9, 21.9, 13.8; **IR** (neat) ν = 3554, 3441, 3060, 3027, 2956, 2930, 2871, 1676, 1595, 1489, 1446, 1376, 1329, 1212, 1162, 1089, 1071, 1025 cm^{-1} ; **MS** (ESI) m/z : 319 ($\text{M}+\text{Na}^+$); **HRMS** (ESI) calcd for $\text{C}_{20}\text{H}_{24}\text{O}_2\text{Na}$ [$\text{M}+\text{Na}^+$]: 319.1669, found: 319.1658.

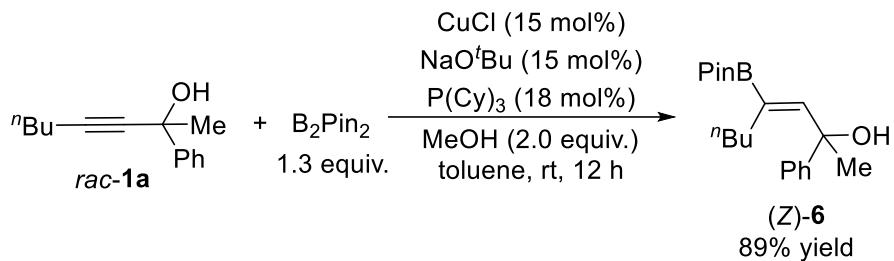
(6) Preparation of (*R,Z*)-2-phenyl-4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)oct-3-en-2-ol ((*R,Z*)-6) (wj-1-179, wj-2-193)⁹



Typical Procedure V: To an oven-dried Schlenk flask were sequentially added B_2Pin_2 (66.1 mg, 0.26 mmol), CuCl (3.0 mg, 0.03 mmol), $\text{NaO}'\text{Bu}$ (2.9 mg, 0.03 mmol), and PCy_3 (10.1 mg, 0.036 mmol) in a glove box. After addition, the flask was degassed and refilled with Ar for three times to ensure the complete exclusion of air. Then (S)-1a (40.3 mg, 0.2 mmol, 96% ee)/toluene (0.5 mL), MeOH (16.2 μL , d = 0.791 g/mL, 0.4 mmol)/toluene (0.1 mL) were added sequentially. And the reaction was stirred at room temperature for 12 h as monitored by TLC, quenched with MeOH (2 mL), filtered through a short column Celite (3 cm) eluted with ethyl acetate (10 mL), and concentrated. The residue was purified by column chromatography on silica gel to afford the product (*R,Z*)-6 (60.1 mg, 91%) [eluent: petroleum ether/ethyl acetate = 30/1 (465 mL) to 20:1 (420 mL)]: 96% ee (HPLC conditions: AD-H column, hexane/ $i\text{PrOH}$ = 98/2, 1.0 mL/min, λ = 214 nm, t_R (major) = 6.9 min, t_R (minor) = 7.7 min); $[\alpha]_D^{28}$ = +8.9 (c = 1.50, CHCl_3); oil; **$^1\text{H NMR}$** (400 MHz, CDCl_3) δ = 7.49 (d, J = 7.6 Hz, 2 H, Ar-H), 7.30 (t, J = 7.6 Hz, 2 H, Ar-H), 7.24-7.18 (m, 1 H, Ar-H), 6.67 (s, 1 H, =CH), 2.04 (t, J = 7.4 Hz, 2 H, CH_2), 1.96 (s, 1 H, OH), 1.67 (s, 3 H, CH_3), 1.26 (s, 12 H, 4 x CH_3), 1.16-0.97 (m, 4 H, 2 x CH_2), 0.72 (t, J = 7.0 Hz, 3 H, CH_3); **$^{13}\text{C NMR}$** (100 MHz, CDCl_3) δ = 149.5, 148.3, 128.0, 126.5, 125.2, 83.3, 74.9, 32.6, 31.6, 29.4, 24.70, 24.67, 22.9, 13.9; **IR** (neat) ν = 3485, 2977, 2929, 1621, 1446, 1371, 1341, 1307, 1213, 1139, 1090, 1064, 1028 cm^{-1} ; **MS** (70 eV, EI) m/z : 330 (M^+), 217 (100); **HRMS** (EI) calcd for $\text{C}_{20}\text{H}_{31}\text{O}_3^{10}\text{B}$ [M^+]: 329.2397, found: 329.2400.

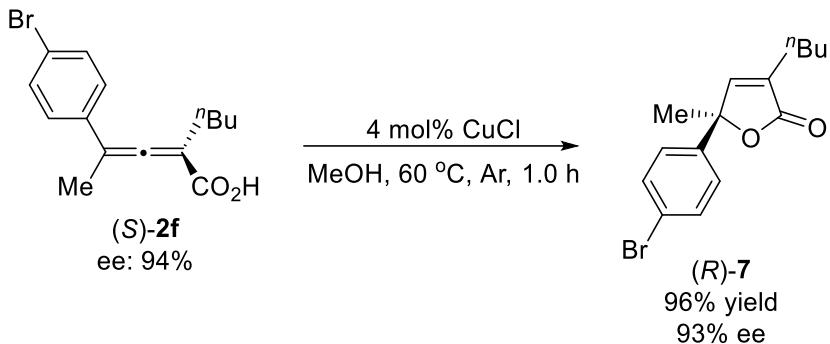
(7) Preparation of (*Z*)-2-phenyl-4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-

yl)oct-3-en-2-ol ((Z)-6) (wj-1-168, wj-2-176)



Following Typical Procedure V, the reaction of B_2Pin_2 (165.0 mg, 0.65 mmol), CuCl (7.4 mg, 0.075 mmol), $\text{NaO}'\text{Bu}$ (7.3 mg, 0.076 mmol), and $\text{P}(\text{Cy})_3$ (25.3 mg, 0.09 mmol), **rac-1a** (101.3 mg, 0.5 mmol)/toluene (1.0 mL), MeOH (40.5 μL , d = 0.791 g/mL, 1.0 mmol)/toluene (0.5 mL) afforded the product **(Z)-6** (147.0 mg, 89%) [eluent: petroleum ether/ethyl acetate = 40/1 (410 mL) to 30:1 (620 mL)]; oil; **$^1\text{H NMR}$** (400 MHz, CDCl_3) δ = 7.49 (d, J = 7.6 Hz, 2 H, Ar-H), 7.31 (t, J = 7.6 Hz, 2 H, Ar-H), 7.24–7.18 (m, 1 H, Ar-H), 6.67 (s, 1 H, =CH), 2.04 (t, J = 7.4 Hz, 2 H, CH_2), 1.94 (s, 1 H, OH), 1.68 (s, 3 H, CH_3), 1.26 (s, 12 H, 4 x CH_3), 1.18–0.97 (m, 4 H, 2 x CH_2), 0.72 (t, J = 7.0 Hz, 3 H, CH_3); **$^{13}\text{C NMR}$** (100 MHz, CDCl_3) δ = 149.5, 148.3, 128.0, 126.5, 125.2, 83.3, 74.9, 32.6, 31.6, 29.4, 24.70, 24.67, 22.9, 13.9; **IR** (neat) ν = 3491, 2977, 2870, 1621, 1446, 1371, 1341, 1307, 1214, 1138, 1090, 1063, 1028 cm^{-1} ; **MS** (70 eV, EI) m/z : 330 (M^+ , 9.75), 217 (100); **HRMS** (EI) calcd for $\text{C}_{20}\text{H}_{31}\text{O}_3^{10}\text{B} [\text{M}^+]$: 329.2397, found: 329.2398.

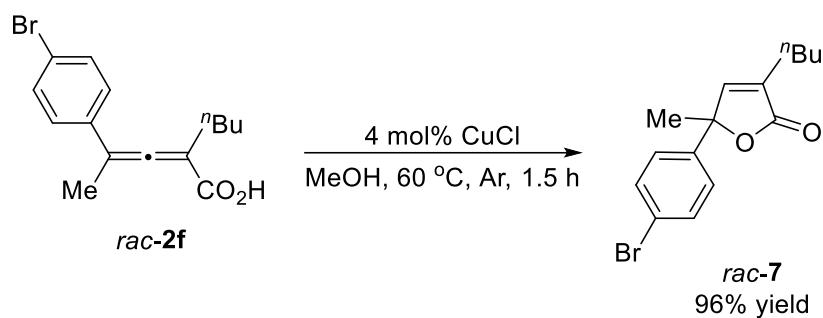
(8) Preparation of (*R*)-5-(4-bromophenyl)-3-butyl-5-methylfuran-2(5*H*)-one ((*R*)-7) (wj-2-131)¹⁰



Typical Procedure VI: To an oven-dried Schlenk flask were added **(S)-2f** (154.7 mg, 0.5 mmol, 94% ee), and CuCl (2.1 mg, 0.02 mmol) in a glove box. After the flask

was degassed and refilled with Ar for three times to ensure the complete exclusion of air, MeOH (5.0 mL) was added sequentially. The resulting mixture was vigorously stirred at 60 °C for 1 h as monitored by TLC, diluted with ethyl acetate (2 mL), filtered through a short column of silica gel (3 cm) eluted with ethyl acetate (20 mL), and concentrated. The residue was purified by chromatography on silica gel to afford the product (*R*)-7 (148.1 mg, 96%) [eluent: petroleum ether / ethyl acetate = 15/1 (320 mL)]: 93% ee (HPLC conditions: OD-H column, hexane/*i*PrOH = 99.5/0.5, 1.0 mL/min, λ = 214 nm, t_R (major) = 13.3 min, t_R (minor) = 14.7 min); $[\alpha]_D^{25} = +124.0$ ($c = 1.09$, CHCl₃); oil; **1H NMR** (400 MHz, CDCl₃): δ = 7.53-7.45 (m, 2 H, Ar-H), 7.30-7.22 (m, 2 H, Ar-H), 7.16 (t, $J = 1.6$ Hz, 1 H, =CH), 2.35-2.22 (m, 2 H, CH₂), 1.76 (s, 3 H, CH₃), 1.59-1.49 (m, 2 H, CH₂), 1.42-1.30 (m, 2 H, CH₂), 0.92 (t, $J = 7.2$ Hz, 3 H, CH₃); **13C NMR** (100 MHz, CDCl₃): δ = 172.9, 151.6, 139.3, 132.8, 131.8, 126.5, 122.1, 86.0, 29.4, 26.7, 24.7, 22.2, 13.7; **IR** (neat): ν = 3075, 2957, 2929, 2866, 1752, 1487, 1459, 1395, 1283, 1248, 1225, 1121, 1078, 1043, 1007 cm⁻¹; **MS** (70 eV, EI) *m/z* (%): 310 (M(⁸¹Br)⁺, 11.02), 308 (M(⁷⁹Br)⁺, 11.24), 267 (100); **HRMS** (EI) calcd for C₁₅H₁₇⁷⁹BrO₂ [M⁺]: 308.0406, found: 308.0406.

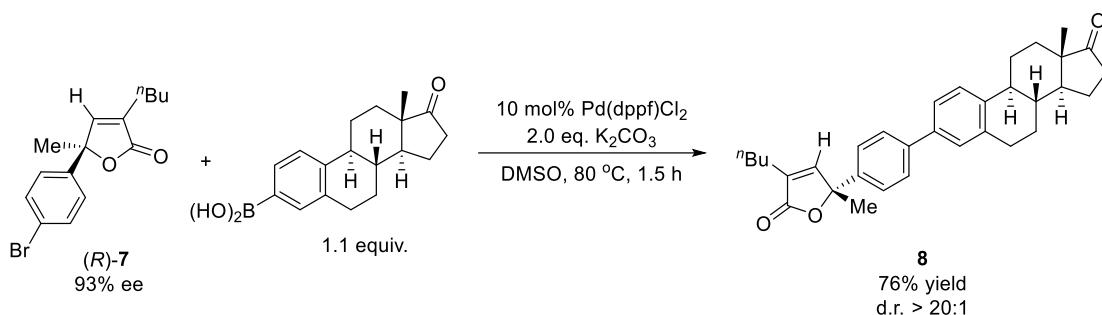
(9) Preparation of 5-(4-bromophenyl)-3-butyl-5-methylfuran-2(5*H*)-one (*rac*-7) (wj-2-130)



Following Typical Procedure VI, the reaction of *rac*-2f (154.6 mg, 0.5 mmol), CuCl (2.1 mg, 0.02 mmol), and MeOH (5.0 mL) afforded the product *rac*-7 (148.2 mg, 96%) [eluent: petroleum ether / ethyl acetate = 15/1 (320 mL)]; oil; **1H NMR** (400 MHz, CDCl₃): δ = 7.54-7.46 (m, 2 H, Ar-H), 7.30-7.22 (m, 2 H, Ar-H), 7.16 (t, $J = 1.6$ Hz, 1 H, =CH), 2.34-2.22 (m, 2 H, CH₂), 1.76 (s, 3 H, CH₃), 1.60-1.49 (m, 2 H, CH₂), 1.43-

1.30 (m, 2 H, CH₂), 0.92 (t, *J* = 7.2 Hz, 3 H, CH₃); ¹³C NMR (100 MHz, CDCl₃): δ = 172.9, 151.6, 139.3, 132.8, 131.8, 126.5, 122.1, 86.0, 29.4, 26.7, 24.7, 22.2, 13.7; IR (neat): ν = 3082, 2957, 2929, 2866, 1752, 1487, 1460, 1395, 1283, 1248, 1224, 1121, 1078, 1043, 1007 cm⁻¹; MS (70 eV, EI) *m/z* (%): 310 (M(⁸¹Br)⁺, 8.75), 308 (M(⁷⁹Br)⁺, 8.44), 265 (100); HRMS (EI) calcd for C₁₅H₁₇⁷⁹BrO₂ [M⁺]: 308.0406, found: 308.0408.

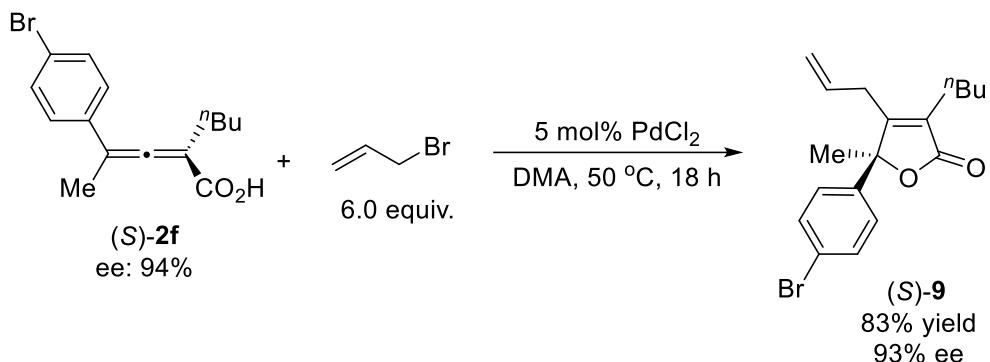
(10) Preparation of 8 (wj-2-134)¹¹



To an oven-dried Schlenk flask (25 mL) were added boronic acid (65.7 mg, 0.22 mmol), Pd(dppf)Cl₂ (14.6 mg, 0.02 mmol), and K₂CO₃ (55.3 mg, 0.4 mmol). After the flask was degassed and refilled with Ar for three times to ensure the complete exclusion of air, (R)-7 (61.8 mg, 0.2 mmol, 93% ee) and DMSO (2 mL) were added sequentially. The resulting mixture was stirred at 80 °C for 1.5 h as monitored by TLC, quenched with 2 mL of H₂O, and extracted with ethyl acetate (5 mL x 3). The combined organic layer was washed with brine, dried over anhydrous Na₂SO₄, filtered, and concentrated. The residue was purified by chromatography on silica gel to afford the product 8 (73.1 mg, 76%) [eluent: petroleum ether / ethyl acetate = 10/1 (440 mL)]; [α]_D²³ = +132.5 (*c* = 1.02, CHCl₃); solid; m.p. 100.8-101.7 °C (petroleum ether/DCM); ¹H NMR (400 MHz, CDCl₃): δ = 7.57 (d, *J* = 8.0 Hz, 2 H, Ar-H), 7.42 (d, *J* = 8.4 Hz, 2 H, Ar-H), 7.37 (s, 2 H, Ar-H), 7.31 (s, 1 H, Ar-H), 7.23 (s, 1 H, =CH), 3.00 (dd, *J*₁ = 9.0 Hz, *J*₂ = 4.0 Hz, 2 H, CH₂), 2.58-2.42 (m, 2 H, CH₂), 2.42-2.25 (m, 3 H, CH and CH₂), 2.22-1.95 (m, 4 H, 2 x CH₂), 1.82 (s, 3 H, CH₃), 1.71-1.45 (m, 8 H, 2 x CH and 3 x CH₂), 1.42-32 (m, 2 H, CH₂), 0.97-0.90 (m, 6 H, 2 x CH₃); ¹³C NMR (100 MHz, CDCl₃): δ = 220.8, 173.2, 152.1, 140.8, 139.2, 138.9, 137.8, 137.0, 132.5, 127.6, 127.2, 125.9, 125.2, 124.4, 86.5, 50.4, 47.9, 44.3, 38.1, 35.8, 31.5, 29.5, 29.4, 26.8, 26.4, 25.7, 24.8, 22.3, 21.5,

13.8, 13.7; **IR** (neat): ν = 3026, 2927, 2861, 1739, 1492, 1457, 1372, 1338, 1255, 1115, 1041, 1008 cm⁻¹; **MS** (70 eV, EI) *m/z* (%): 482 (M⁺, 33.77), 117 (100); **HRMS** (EI) calcd for C₃₃H₃₈O₃ [M⁺]: 482.2815, found: 482.2816.

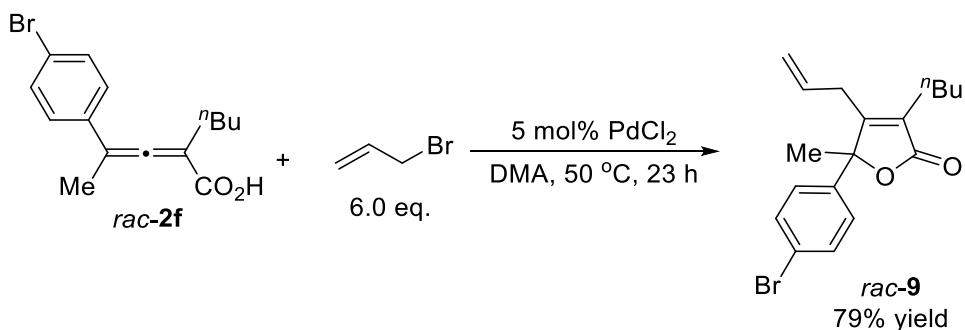
(11) Preparation of (*S*)-4-allyl-5-(4-bromophenyl)-3-butyl-5-methylfuran-2(5*H*)-one ((*S*)-9) (wj-2-129)¹²



Typical Procedure VII: To an oven-dried Schlenk flask were added (S)-2f (154.5 mg, 0.5 mmol, 94% ee) and PdCl₂ (4.4 mg, 0.025 mmol). After the flask was degassed and refilled with Ar for three times to ensure the complete exclusion of air, DMA (3 mL) and allyl bromide (260 uL, d = 1.398 g/mL, 363.5 mg, 3.0 mmol) were added sequentially via a syringe under a flow of argon. The resulting mixture was vigorously stirred at 50 °C for 18 h as monitored by TLC, quenched with 5 mL of H₂O, and extracted with Et₂O (10 mL x 3). The combined organic layer was washed with brine, dried over anhydrous Na₂SO₄, filtered, and concentrated. The residue was purified by chromatography on silica gel to afford the product (S)-9 (144.9 mg, 83%) [eluent: petroleum ether / ethyl acetate = 20/1 (420 mL)]: 93% ee (HPLC conditions: OD-H column, hexane/ⁱPrOH = 99.5/0.5, 1.0 mL/min, λ = 214 nm, *t*_R (major) = 12.6 min, *t*_R (minor) = 14.4 min); $[\alpha]_D^{24} = +155.7$ (*c* = 1.33, CHCl₃); oil; **1H NMR** (400 MHz, CDCl₃): δ = 7.52-7.43 (m, 2 H, Ar-H), 7.19-7.11 (m, 2 H, Ar-H), 5.55-5.42 (m, 1 H, =CH), 5.07-4.96 (m, 2 H, =CH₂), 2.99 (dd, *J*₁ = 15.6 Hz, *J*₂ = 5.6 Hz, 1 H, one proton of CH₂), 2.85 (dd, *J*₁ = 15.6 Hz, *J*₂ = 7.2 Hz, 1 H, one proton of CH₂); 2.29 (t, *J* = 7.8 Hz, 2 H, CH₂), 1.81(s, 3 H, CH₃), 1.58-1.46 (m, 2 H, CH₂), 1.40-1.28 (m, 2 H, CH₂), 0.93 (t, *J* = 7.4 Hz, 3 H, CH₃); **13C NMR** (100 MHz, CDCl₃): δ = 173.5, 163.8, 137.6,

132.2, 131.8, 127.9, 127.3, 122.6, 117.9, 87.3, 30.6, 30.1, 23.5, 23.3, 22.6, 13.8; **IR** (neat): ν = 3081, 2957, 2930, 2864, 1749, 1668, 1638, 1489, 1453, 1396, 1257, 1207, 1080, 1043, 1006 cm⁻¹; **MS** (70 eV, EI) *m/z* (%): 350 ($M(^{81}Br)^+$, 25.73), 25.40), 307 (100); **HRMS** (EI) calcd for $C_{18}H_{21}^{79}BrO_2$ [M⁺]: 348.0719, found: 348.0718.

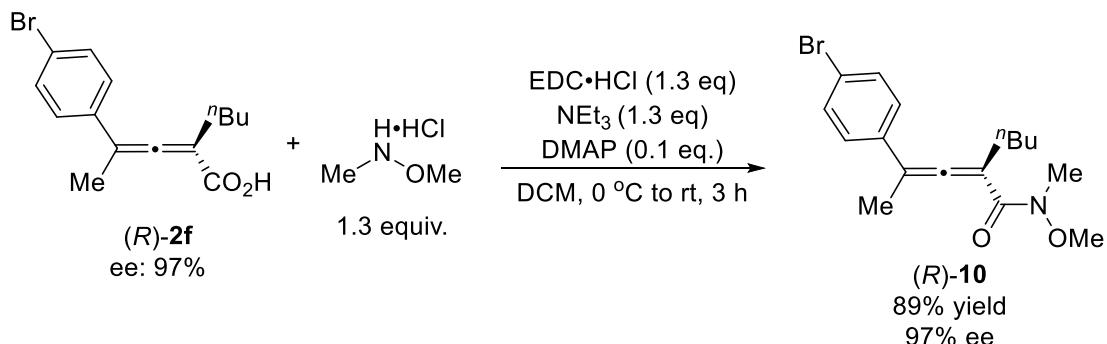
(12) Preparation of 4-allyl-5-(4-bromophenyl)-3-butyl-5-methylfuran-2(5*H*)-one (*rac*-9**) (wj-2-126)**



Following Typical Procedure VII: the reaction of *rac*-2f (154.7 mg, 0.5 mmol), PdCl₂ (4.4 mg, 0.025 mmol), DMA (3 mL), and allyl bromide (259.6 uL, d = 1.398 g/mL, 362.9 mg, 3.0 mmol) afforded *rac*-9 (137.7 mg, 79%) [eluent: petroleum ether / ethyl acetate = 20/1 (420 mL)]; oil; **1H NMR** (400 MHz, CDCl₃): δ = 7.52-7.44 (m, 2 H, Ar-H), 7.18-7.12 (m, 2 H, Ar-H), 5.56-5.41 (m, 1 H, =CH), 5.07-4.97 (m, 2 H, =CH₂), 2.99 (ddt, J_1 = 15.6 Hz, J_2 = 5.6 Hz, J_3 = 1.6 Hz, 1 H, one proton of CH₂), 2.85 (dd, J_1 = 15.6 Hz, J_2 = 7.2 Hz, 1 H, one proton of CH₂); 2.29 (t, J = 7.8 Hz, 2 H, CH₂), 1.81(s, 3 H, CH₃), 1.59-1.46 (m, 2 H, CH₂), 1.40-1.28 (m, 2 H, CH₂), 0.92 (t, J = 7.4 Hz, 3 H, CH₃); **13C NMR** (100 MHz, CDCl₃): δ = 173.5, 163.8, 137.6, 132.2, 131.8, 127.9, 127.3, 122.6, 117.9, 87.3, 30.6, 30.1, 23.5, 23.3, 22.6, 13.8; **IR** (neat): ν = 3085, 2958, 2931, 2864, 1749, 1668, 1637, 1490, 1453, 1396, 1256, 1207, 1079, 1043, 1006 cm⁻¹; **MS** (70 eV, EI) *m/z* (%): 350 ($M(^{81}Br)^+$, 26.45), 348 ($M(^{79}Br)^+$, 26.02), 183 (100); **HRMS** (EI) calcd for $C_{18}H_{21}^{79}BrO_2$ [M⁺]: 348.0719, found: 348.0723.

(13) Preparation of (*R*)-*N*-methoxy-*N*-methyl-2-butyl-4-(4-bromophenyl)-2,3-

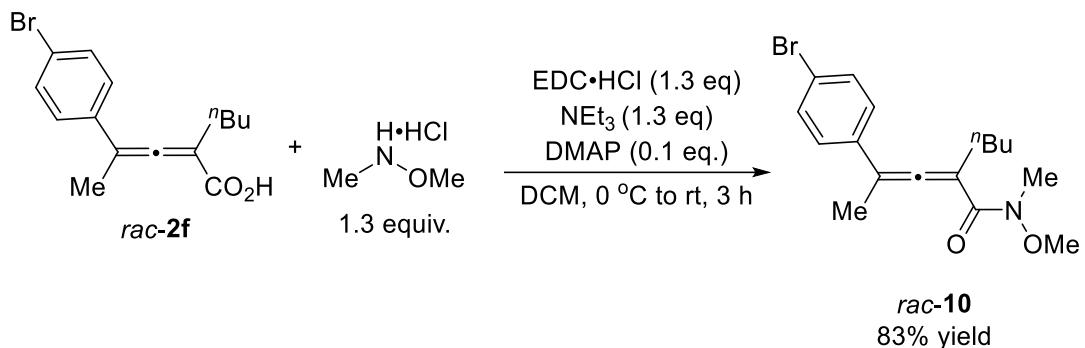
pentadienamide ((R)-10) (wj-2-128)¹³



Typical Procedure VIII: To an oven-dried Schlenk flask were added (*R*)-**2f** (92.9 mg, 0.3 mmol, 97% ee), EDC•HCl (74.8 mg, 0.39 mmol), methyl methoxylamine hydrochloride (38.0 mg, 0.39 mmol), and DMAP (3.8 mg, 0.03 mmol). After the flask was degassed and refilled with Ar for three times to ensure the complete exclusion of air, NEt₃ (54.2 uL, d = 0.728 g/mL, 39.5 mg, 0.39 mmol) and DCM (1 mL) were added sequentially. The resulting mixture was vigorously stirred at 0 °C for 1 h, gradually warmed up to room temperature and stirred at room temperature for 2 h as monitored by TLC, diluted with ethyl acetate (2 mL), and washed with saturated NH₄Cl (10 mL x 3). The combined organic layer was washed with brine, dried over anhydrous Na₂SO₄, filtered, and concentrated. The residue was purified by chromatography on silica gel to afford the product (*R*)-**10** (94.0 mg, 89%) [eluent: petroleum ether / ethyl acetate = 10/1 (220 mL)]: 97% ee (HPLC conditions: OD-H column, hexane/PrOH = 99/1, 1.0 mL/min, λ = 214 nm, t_R (major) = 8.0 min, t_R (minor) = 9.4 min); $[\alpha]_D^{25} = -131.4$ ($c = 0.99$, CHCl₃); oil; **1H NMR** (400 MHz, CDCl₃): δ = 7.49-7.42 (m, 2 H, Ar-H), 7.30-7.20 (m, 2 H, Ar-H), 3.50 (s, 3 H, CH₃), 3.23 (s, 3 H, CH₃), 2.41 (t, J = 7.2 Hz, 2 H, CH₂), 2.14 (s, 3 H, CH₃), 1.49-1.31 (m, 4 H, 2 x CH₂), 0.89 (t, J = 7.0 Hz, 3 H, CH₃); **13C NMR** (100 MHz, CDCl₃): δ = 206.2, 167.8, 135.1, 131.5, 127.4, 120.9, 101.4, 101.3, 61.1, 33.7, 30.1, 30.0, 22.3, 16.4, 13.9; **IR** (neat): ν = 2956, 2929, 2860, 1942, 1637, 1484, 1458, 1408, 1372, 1183, 1148, 1105, 1076, 1006 cm⁻¹; **MS** (ESI) *m/z* (%): 354 (M(⁸¹Br)+H⁺), 352 (M(⁷⁹Br)+H⁺); **HRMS** (ESI) calcd for C₁₇H₂₃⁷⁹BrO₂N [M+H⁺]: 352.0907, found: 352.0904.

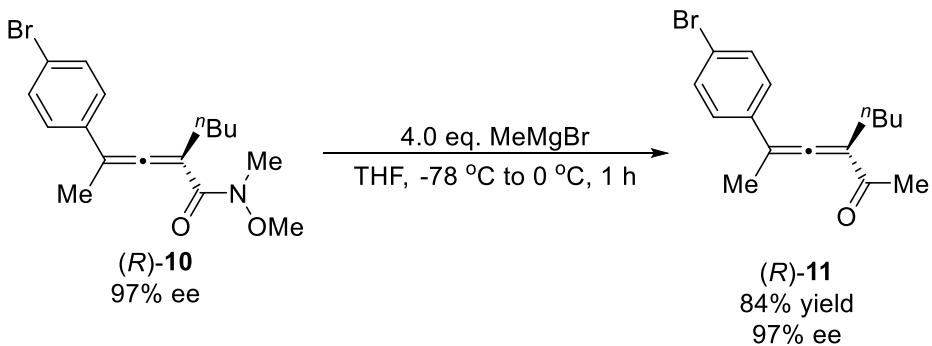
(14) Preparation of N-methoxy-N-methyl-2-butyl-4-(4-bromophenyl)-2,3-

pentadienamide (*rac*-10) (wj-2-127)



Following Typical Procedure VIII, the reaction of *rac*-2f (92.7 mg, 0.3 mmol), EDC·HCl (74.8 mg, 0.39 mmol), methyl methoxylamine hydrochloride (38.0 mg, 0.39 mmol), DMAP (3.8 mg, 0.03 mmol), NEt₃ (54.2 uL, d = 0.728 g/mL, 39.5 mg, 0.39 mmol), and DCM (1 mL) afforded the product *rac*-10 (88.0 mg, 83%) [eluent: petroleum ether / ethyl acetate = 10/1 (220 mL)]; oil; ¹H NMR (400 MHz, CDCl₃): δ = 7.48-7.42 (m, 2 H, Ar-H), 7.30-7.23 (m, 2 H, Ar-H), 3.50 (s, 3 H, CH₃), 3.22 (s, 3 H, CH₃), 2.41 (t, J = 7.4 Hz, 2 H, CH₂), 2.14 (s, 3 H, CH₃), 1.50-1.30 (m, 4 H, 2 x CH₂), 0.89 (t, J = 7.0 Hz, 3 H, CH₃); ¹³C NMR (100 MHz, CDCl₃): δ = 206.2, 167.8, 135.1, 131.5, 127.4, 120.9, 101.4, 101.3, 61.1, 33.7, 30.1, 30.0, 22.3, 16.4, 13.9; IR (neat): ν = 2957, 2929, 2859, 1942, 1637, 1484, 1458, 1408, 1372, 1183, 1148, 1105, 1076, 1006 cm⁻¹; MS (ESI) m/z (%): 354 (M(⁸¹Br)+H⁺), 352 (M(⁷⁹Br)+H⁺); HRMS (ESI) calcd for C₁₇H₂₃⁷⁹BrO₂N [M+H⁺]: 352.0907, found: 352.0906.

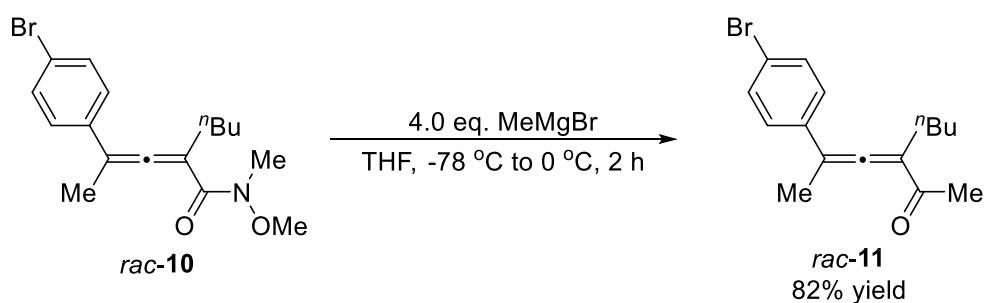
(15) Preparation of (*R*)-3-butyl-5-(4-bromophenyl)-3,4-hexadien-2-one ((*R*)-11) (wj-2-136)



Typical Procedure IX: To an oven-dried Schlenk flask were added Weinreb amide (*R*)-10 (70.7 mg, 0.2 mmol, 97% ee) and THF (2 mL) under Ar atmosphere and

cooled to -78 °C. Then MeMgBr (0.27 mL, 3.0 M in Et₂O, 0.8 mmol) was added into the solution dropwise with stirring. The resulting mixture was warmed up to 0 °C gradually, monitored by TLC, quenched with 2 mL of H₂O and extracted with ethyl acetate (10 mL x 3). The combined organic layer was washed with brine, dried over anhydrous Na₂SO₄, filtered, and concentrated. The residue was purified by chromatography on silica gel to afford the product (*R*)-11 (51.5 mg, 84%) [eluent: petroleum ether / ethyl acetate = 15/1 (100 mL)]: 97% ee (HPLC conditions: OD-H column, hexane/ⁱPrOH = 99.5/0.5, 1.0 mL/min, λ = 214 nm, t_R (major) = 6.3 min, t_R (minor) = 7.4 min); $[\alpha]_D^{23} = -16.0$ (c = 1.20, CHCl₃); oil; **¹H NMR** (400 MHz, CDCl₃): δ = 7.48 (dt, J_1 = 8.4 Hz, J_2 = 2.2 Hz, 2 H, Ar-H), 7.29-7.21 (m, 2 H, Ar-H), 2.30 (t, J = 7.4 Hz, 2 H, CH₂), 2.24 (s, 3 H, CH₃), 2.21 (s, 3 H, CH₃), 1.45-1.28 (m, 4 H, 2 x CH₂), 0.88 (t, J = 7.0 Hz, 3 H, CH₃); **¹³C NMR** (100 MHz, CDCl₃): δ = 213.6, 198.5, 134.0, 131.8, 127.2, 121.5, 111.7, 104.2, 30.1, 27.3, 26.6, 22.4, 16.3, 13.8; **IR** (neat): ν = 2956, 2926, 2859, 1931, 1678, 1486, 1461, 1355, 1228, 1117, 1075, 1007 cm⁻¹; **MS** (70 eV, EI) m/z (%): 308 (M(⁸¹Br)⁺, 6.39), 306 (M(⁷⁹Br)⁺, 6.71), 263 (100); **HRMS** (EI) calcd for C₁₆H₁₉⁷⁹BrO [M⁺]: 306.0614, found: 306.0617.

(16) Preparation of 3-butyl-5-(4-bromophenyl)-3,4-hexadien-2-one (*rac*-11) (wj-2-135)

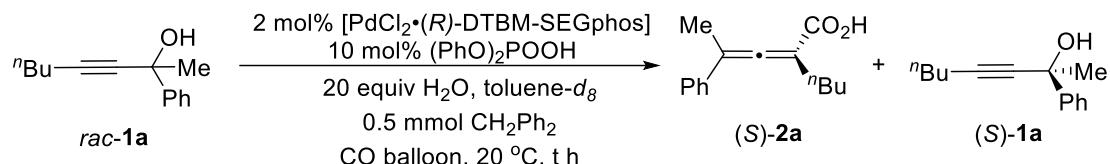


Following Typical Procedure IX: the reaction of Weinreb amide **rac-10** (70.6 mg, 0.2 mmol), THF (2 mL) and MeMgBr (0.27 mL, 3.0 M in Et₂O, 0.8 mmol) afforded the product **rac-11** (50.4 mg, 82%) [eluent: petroleum ether / ethyl acetate = 15/1 (100 mL); oil; **¹H NMR** (400 MHz, CDCl₃): δ = 7.49 (dt, *J*₁ = 8.8 Hz, *J*₂ = 2.2 Hz, 2 H, Ar-H), 7.28-7.21 (m, 2 H, Ar-H), 2.3 (t, *J* = 7.4 Hz, 2 H, CH₂), 2.24 (s, 3 H, CH₃), 2.21 (s, 3 H,

CH_3), 1.45-1.28 (m, 4 H, 2 x CH_2), 0.88 (t, $J = 7.0$ Hz, 3 H, CH_3); **^{13}C NMR** (100 MHz, CDCl_3): $\delta = 213.6, 198.5, 134.0, 131.8, 127.2, 121.5, 111.7, 104.2, 30.1, 27.3, 26.6, 22.4, 16.3, 13.9$; **IR** (neat): $\nu = 2956, 2926, 2860, 1931, 1678, 1486, 1461, 1355, 1228, 1117, 1075, 1007 \text{ cm}^{-1}$; **MS** (70 eV, EI) m/z (%): 308 ($\text{M}^{(81\text{Br})^+}$, 5.44), 306 ($\text{M}^{(79\text{Br})^+}$, 5.66), 263 (100); **HRMS** (EI) calcd for $\text{C}_{16}\text{H}_{19}^{79}\text{BrO} [\text{M}^+]$: 306.0614, found: 306.0618.

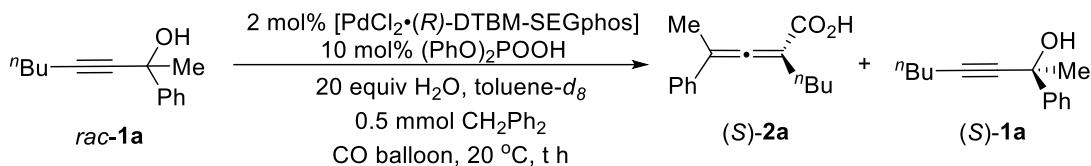
Monitoring Experiment⁴

¹H NMR Monitoring Experiment of the kinetic resolution reaction (wj-3-121)

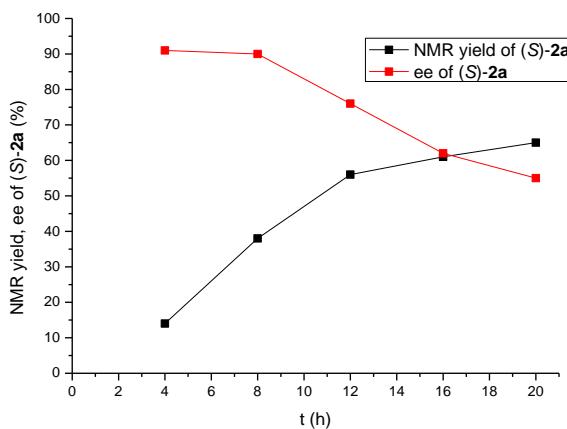


To a Schlenk tube (25 mL) were added [PdCl₂•(R)-DTBM-SEGphos] (27.2 mg, 0.02 mmol), (PhO)₂PO₂H (24.9 mg, 0.1 mmol). After addition, the flask was degassed and refilled with Ar for three times to ensure the complete exclusion of air. Then *rac*-1a (202.4 mg, 1 mmol)/toluene-*d*₈ (3 mL), H₂O (360 μL, d = 1.0 g/mL, 360.0 mg, 20 mmol)/toluene-*d*₈ (2 mL) and CH₂Ph₂ (83.6 μL, d = 1.006 g/mL, 84.1 mg, 0.5 mmol) were added sequentially under argon. After that, the Ar gas line was closed. The resulting mixture was then frozen with a liquid nitrogen bath, degassed to remove the argon inside completely, and refilled with CO by a balloon of CO (about 1 L) for three times. Then the liquid nitrogen bath was removed and the mixture was allowed to stand until completely thawed, vigorously stirred at 20 °C with a balloon of CO. 0.2 mL each of the aliquot was taken with an Ar-purged syringes after 4 h, 8 h, 12 h, 16 h, 20 h. Then the mixture was analyzed by ¹H NMR spectra with CH₂Ph₂ as the internal standard to determine the yield of (S)-2a and the recoveries of 1a. The residues were purification by the preparative TLC (petroleum ether / ethyl acetate = 5/1) and the ee value were determined by HPLC analysis. And the data acquired were analyzed by Origin 9.

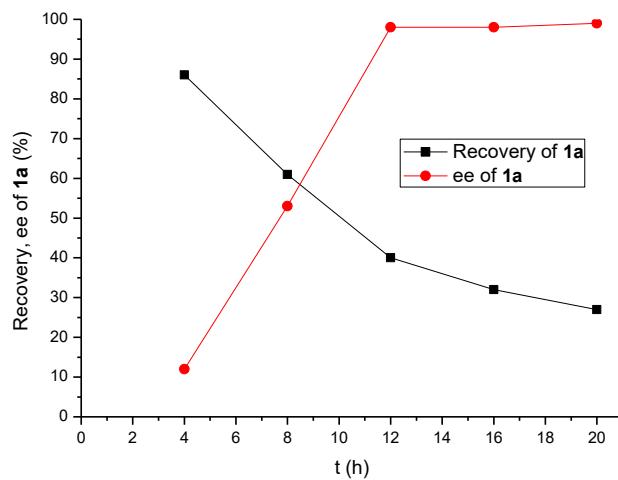
Supplementary Table 1. ¹H NMR Monitoring Experiment of the kinetic resolution reaction



entry	time / h	NMR yield of (S)-2a, ee of (S)-2a / %	Recovery of 1a, ee of 1a / %
1	4	14, 91	86, 12
2	8	38, 90	61, 53
3	12	56, 76	40, 98
4	16	61, 62	32, 98
5	20	65, 55	27, 99



Supplementary Figure 2. The effect in the formation of (S)-2a via the kinetic resolution reaction.



Supplementary Figure 3. The effect in the recoveries of 1a via the kinetic resolution reaction.

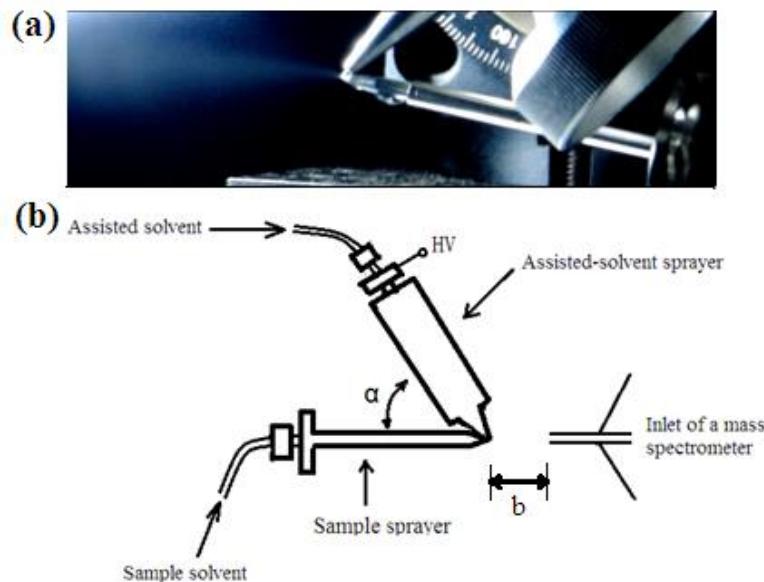
SAESI-MS studies¹⁴

SAESI-MS conditions

SAESI-MS spectra were recorded on a Thermo TSQ Quantum Access triple-quadrupole mass spectrometer (Thermo Fisher Scientific, Waltham, MA) equipped with a home-made SAESI ion source in positive mode. The basic SAESI conditions were: vacuum, 2.8×10^{-6} torr; spray voltage, 4000 V; capillary temperature, 275 °C; sheath gas pressure of two sprayers, 3 arb. units; the collision energy of CID, 20 eV. Data acquisition and analysis were done with the Xcalibur (version 2.0, Thermo Fisher Scientific) software package.

In solvent-assisted electrospray ionization mass spectrometric experiment, the angle (α) between the two sprayers is 45° and the distance (b) between the tip of sprayers and the inlet to the mass is 6 mm. The chemical solutions were injected by a 500-μL air-tight syringe with a speed at 5 μL/min to SAESI-MS. The assisted solvent of methanol was injected by another 500-μL air-tight syringe with a speed at 5 μL/min.

SAESI-MS device.



Supplementary Figure 4. (a) Photographic image of SAESI apparatus. (b) Schematic representation of the SAESI. The angle (α) between the two sprayers is 45° and the distance (b) between the tip of sprayers and the inlet to the mass is 6 mm.

References:

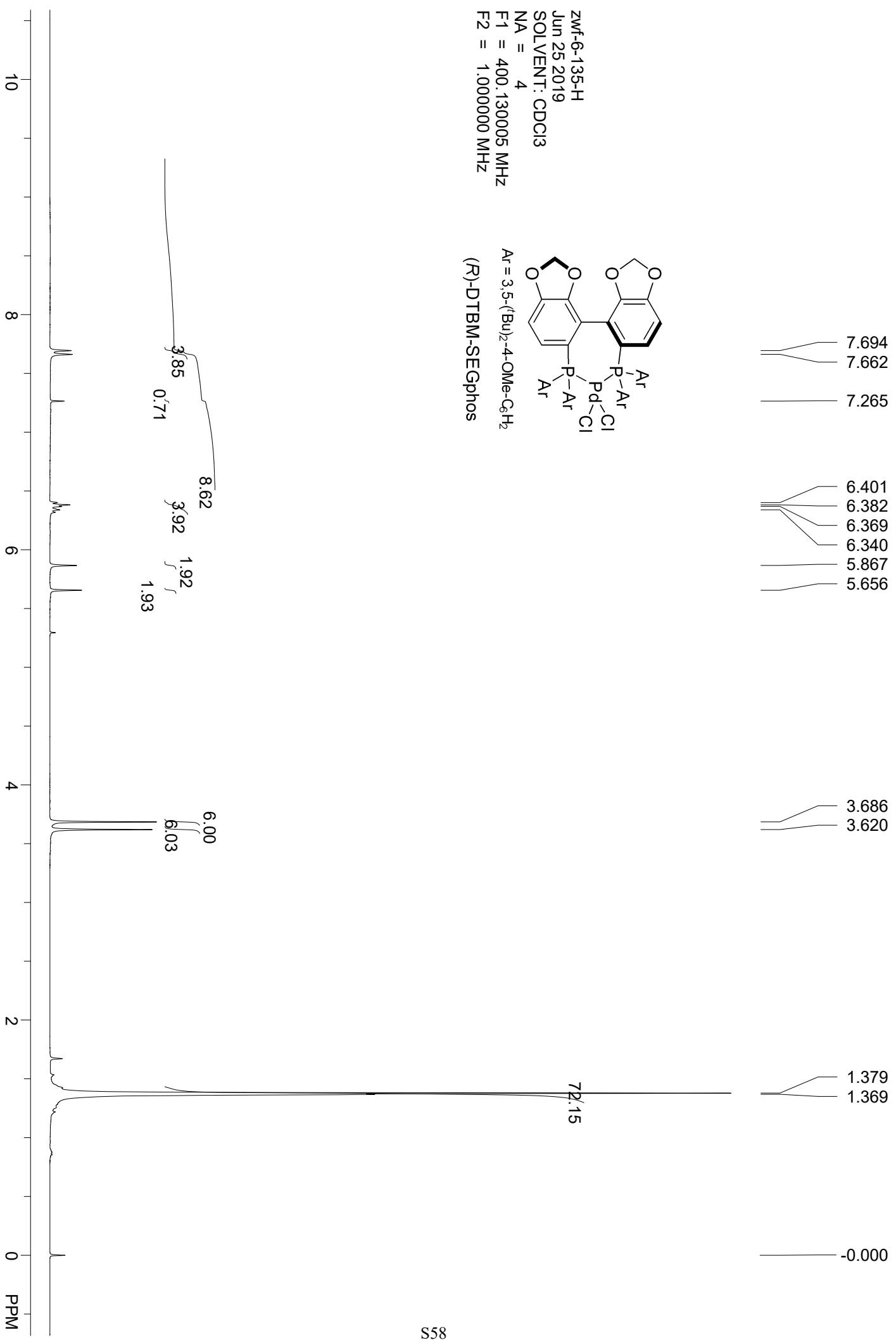
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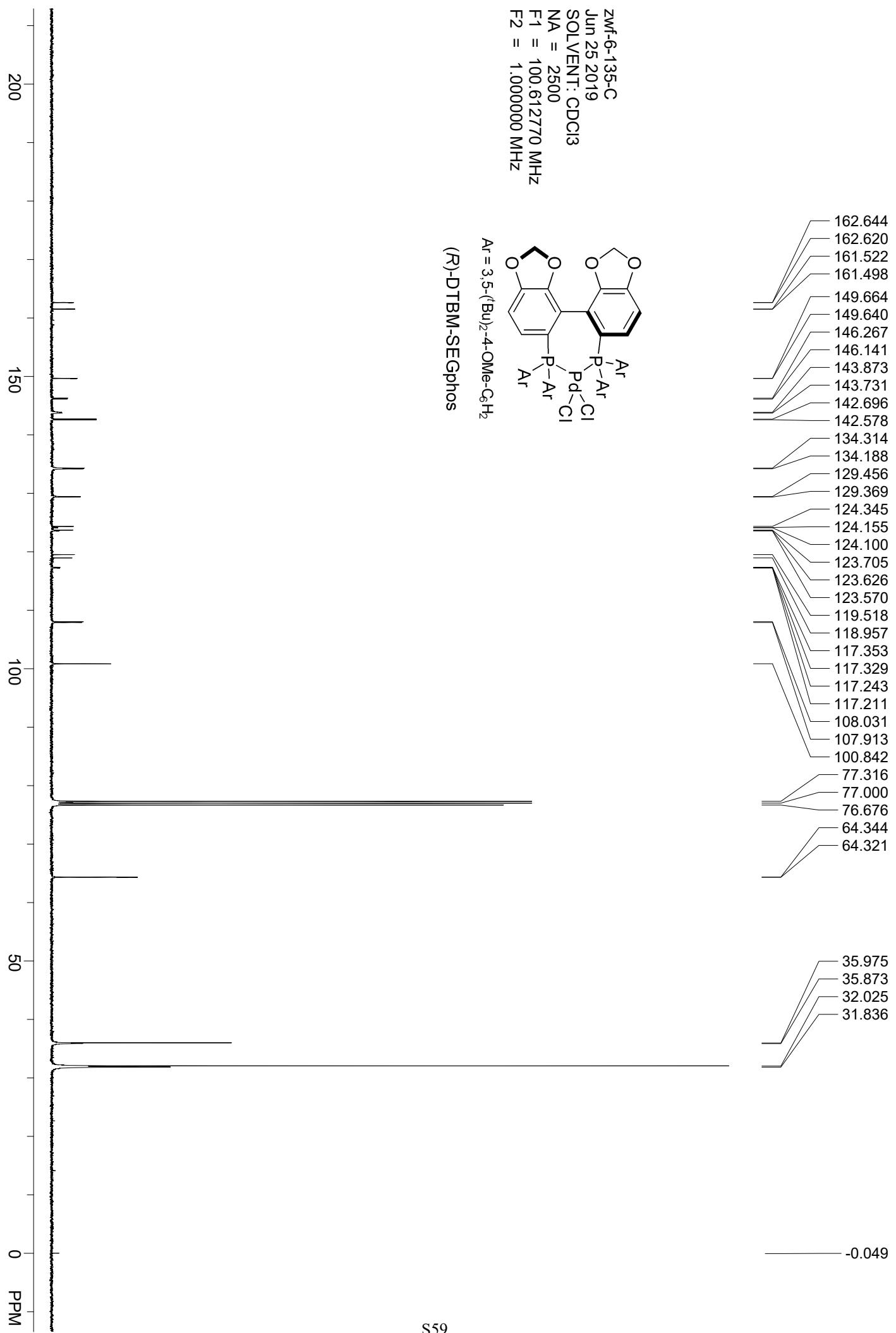
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JUN 25 2010

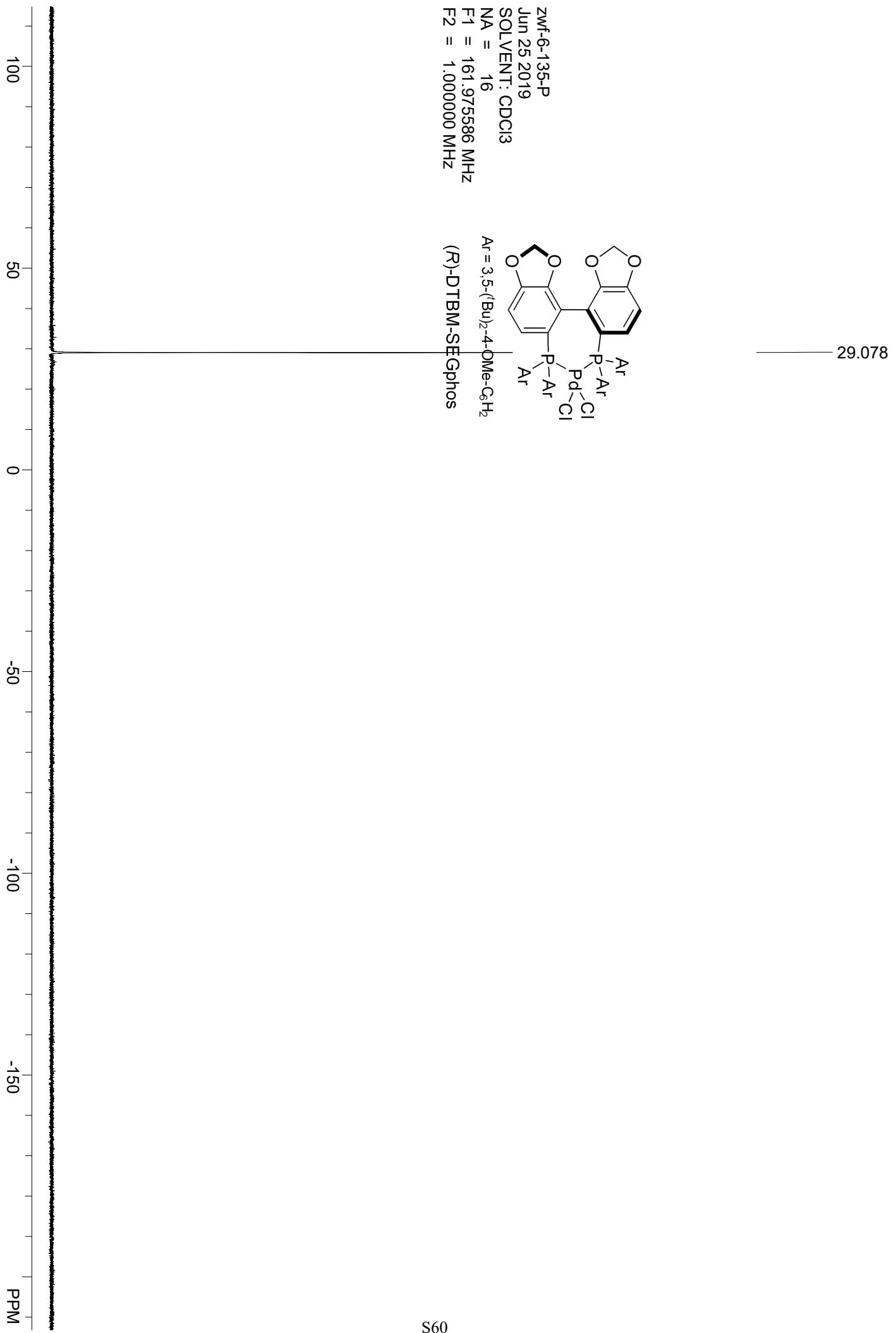
JUL 25 2019
SOLVENT: C

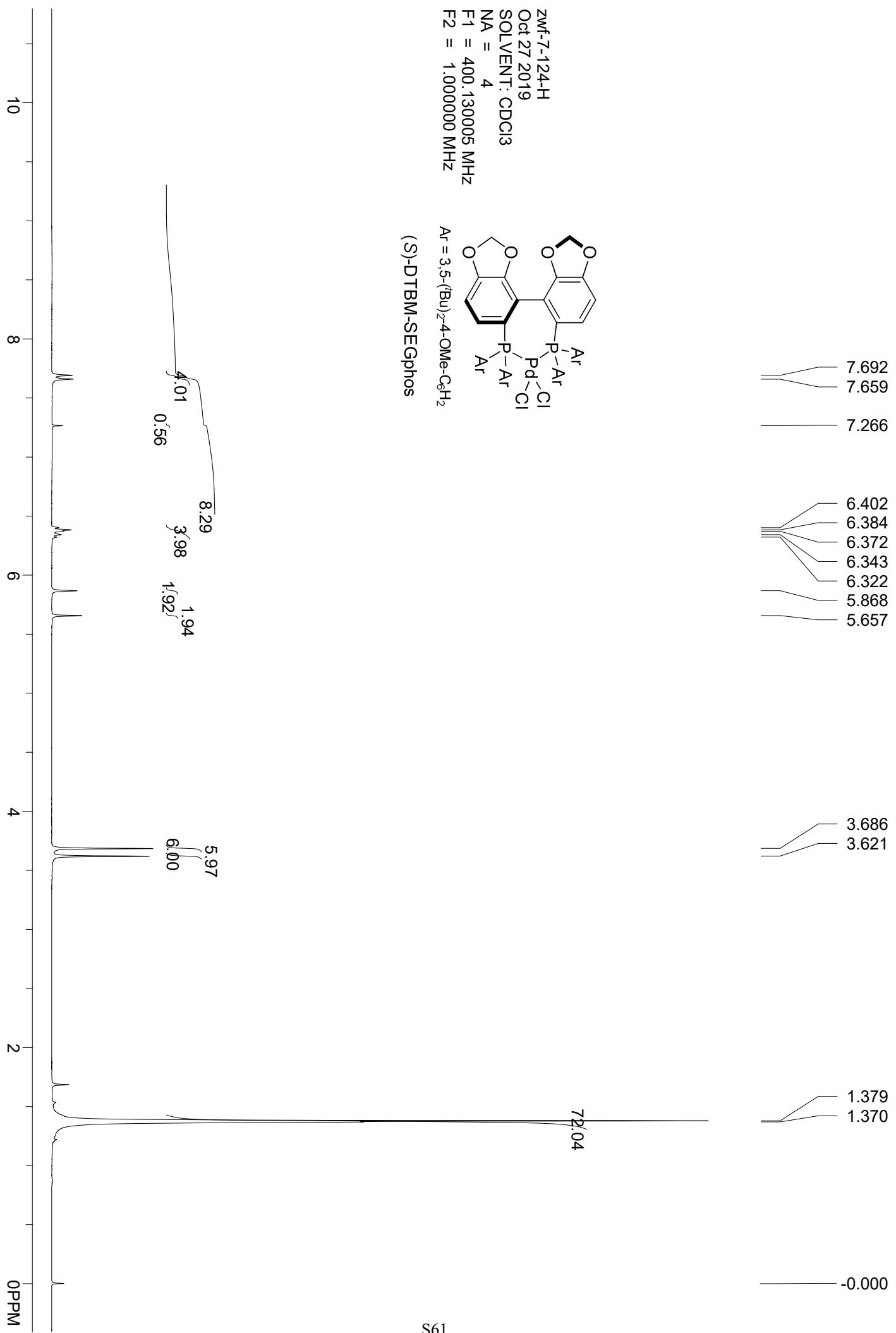
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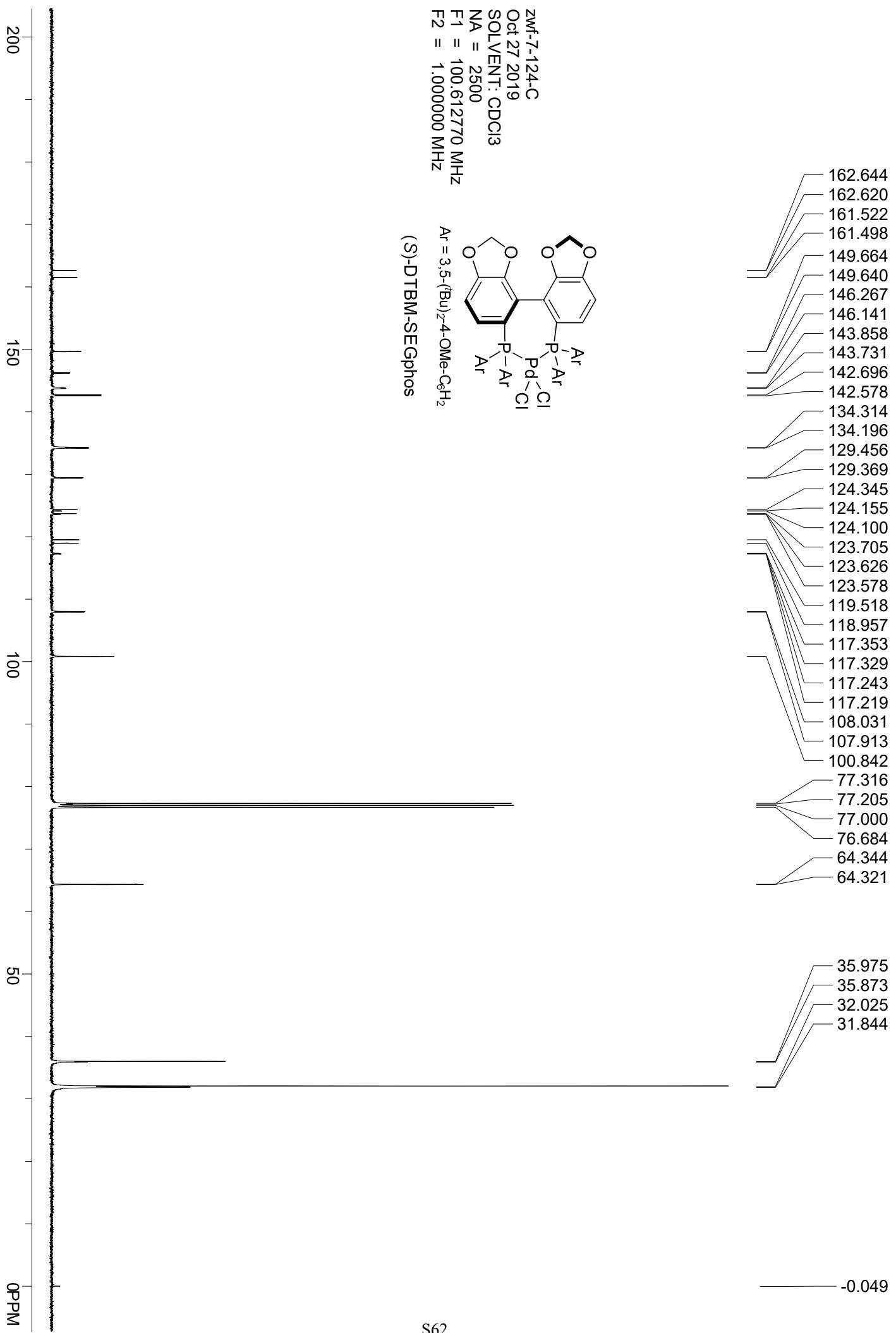
$$F_1 = 400.130003 \text{ MHz}$$

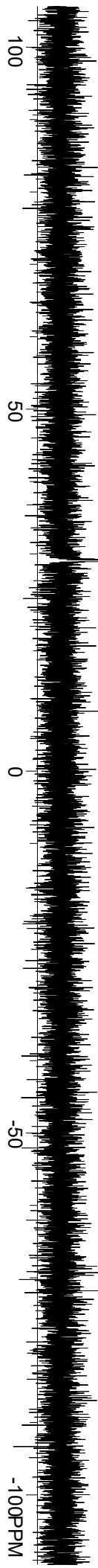


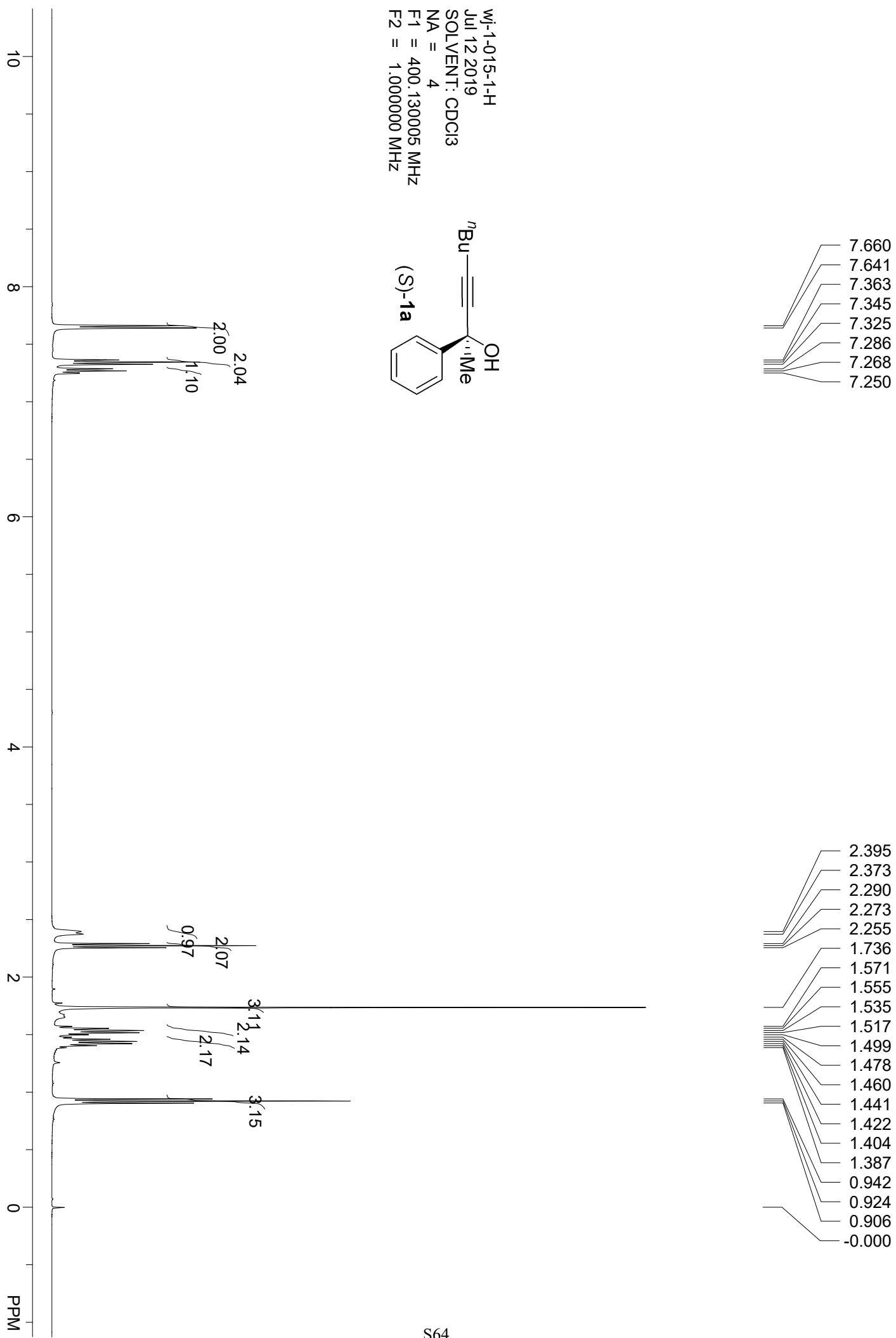


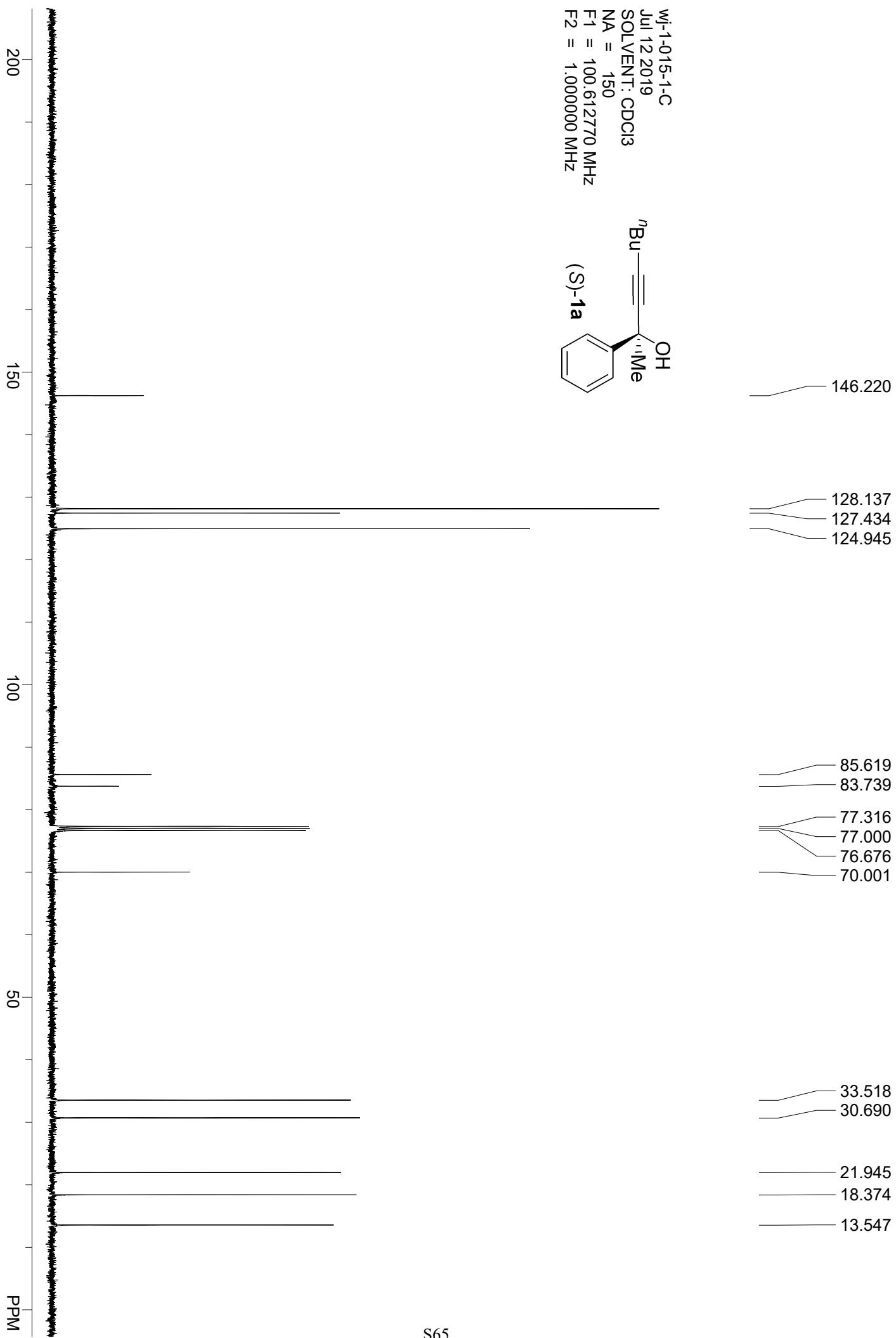












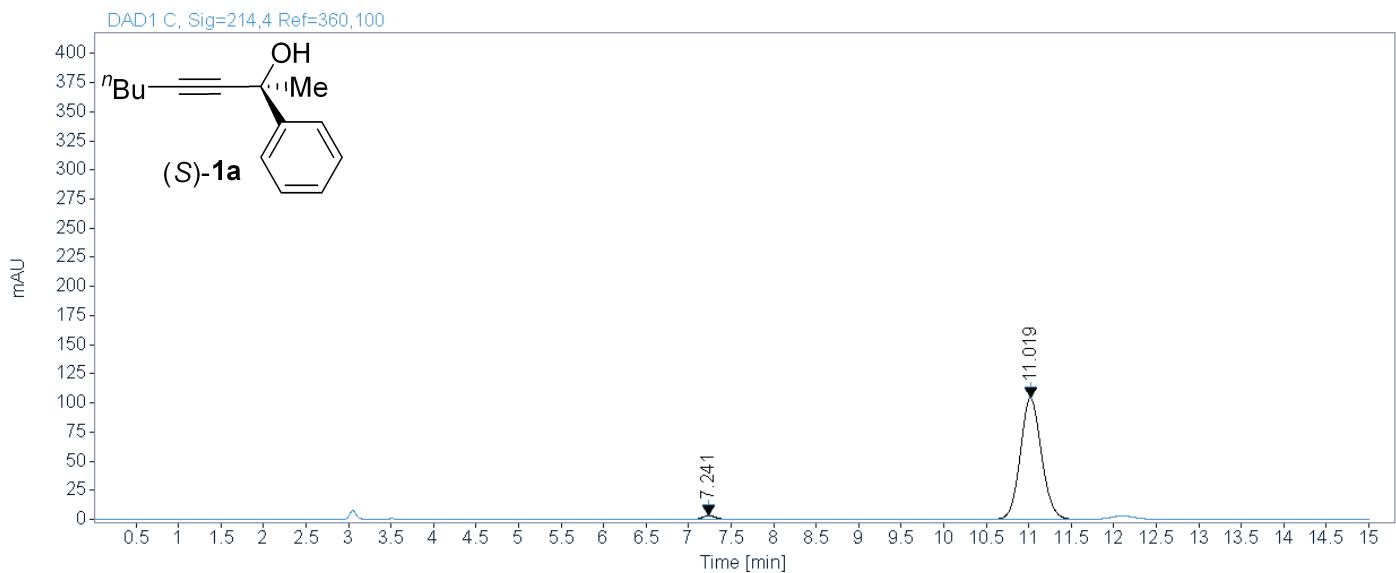
Area Percent Report



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Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

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11.019	0.2683	104.3075	1808.7644	98.1504
Sum		104.3075	1842.8500	100.0000

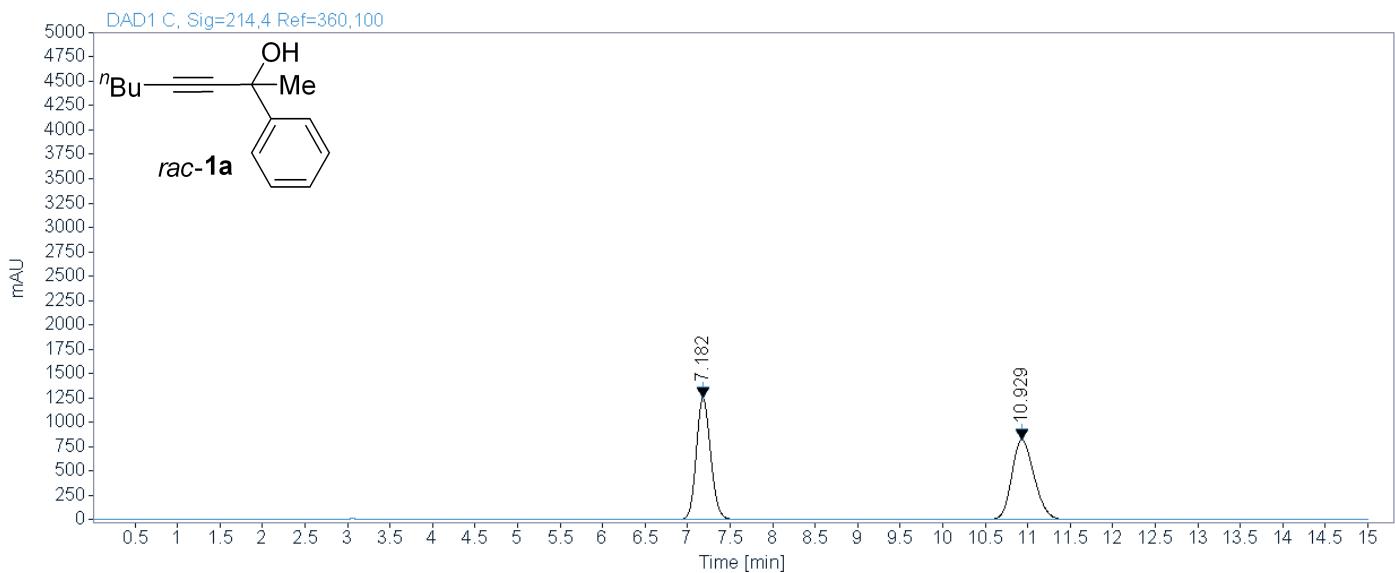
Area Percent Report



sample wj-1-015-1-rac-AS-H-98-2-1.0-
214

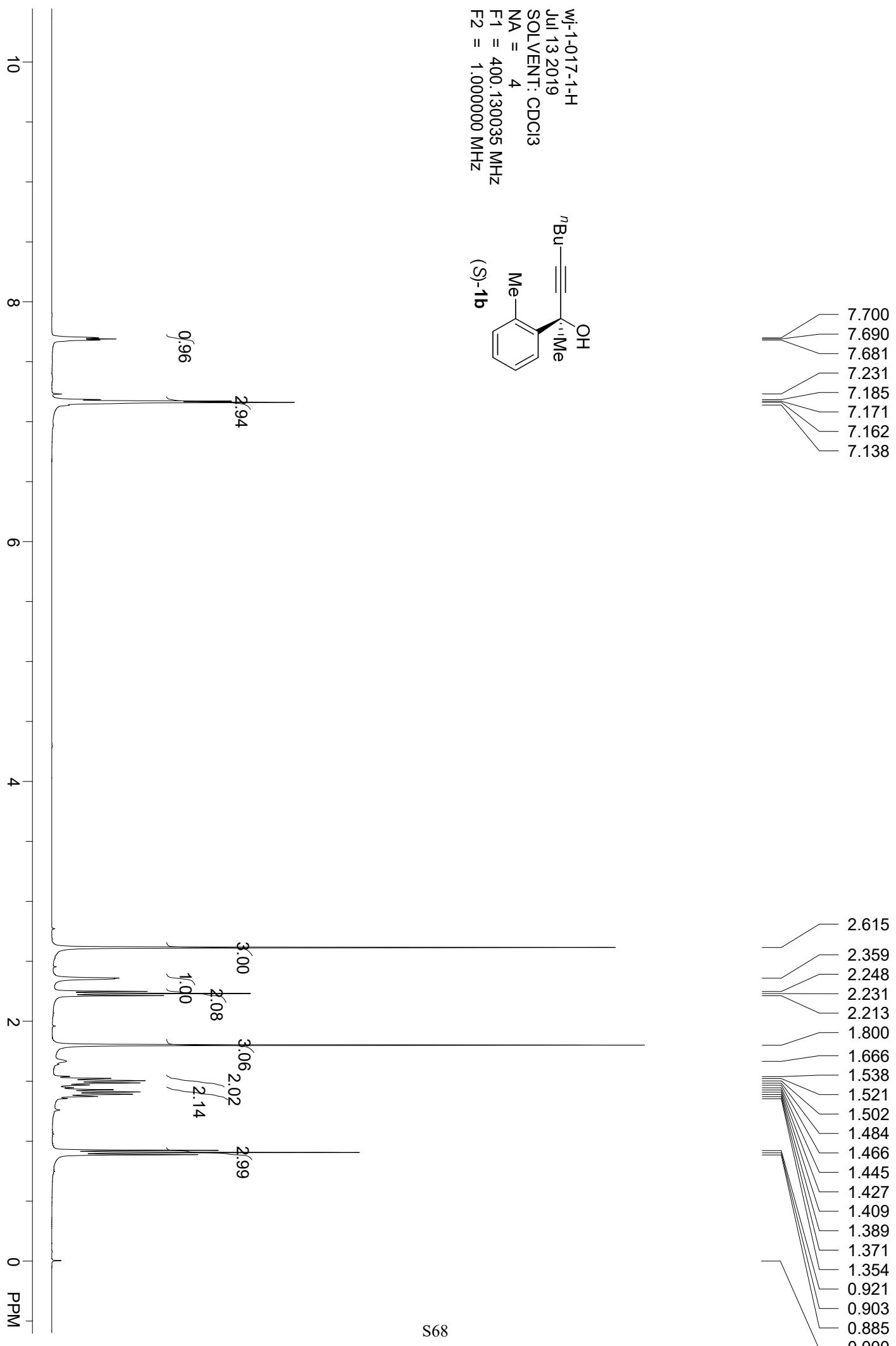
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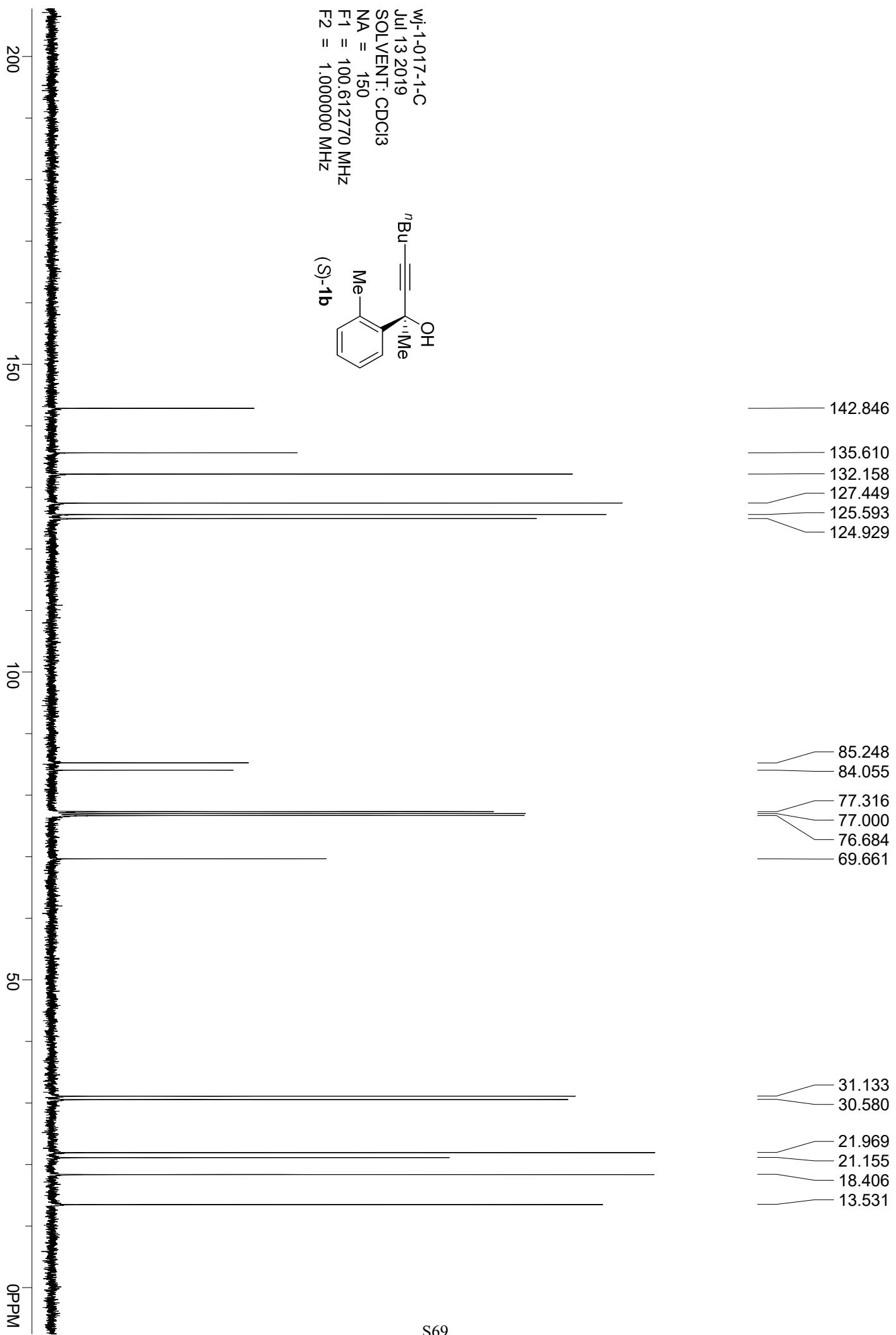
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Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
7.182	0.1838	1251.3541	14780.0400	49.4825
10.929	0.2866	820.3699	15089.1592	50.5175
Sum		29869.1992	100.0000	





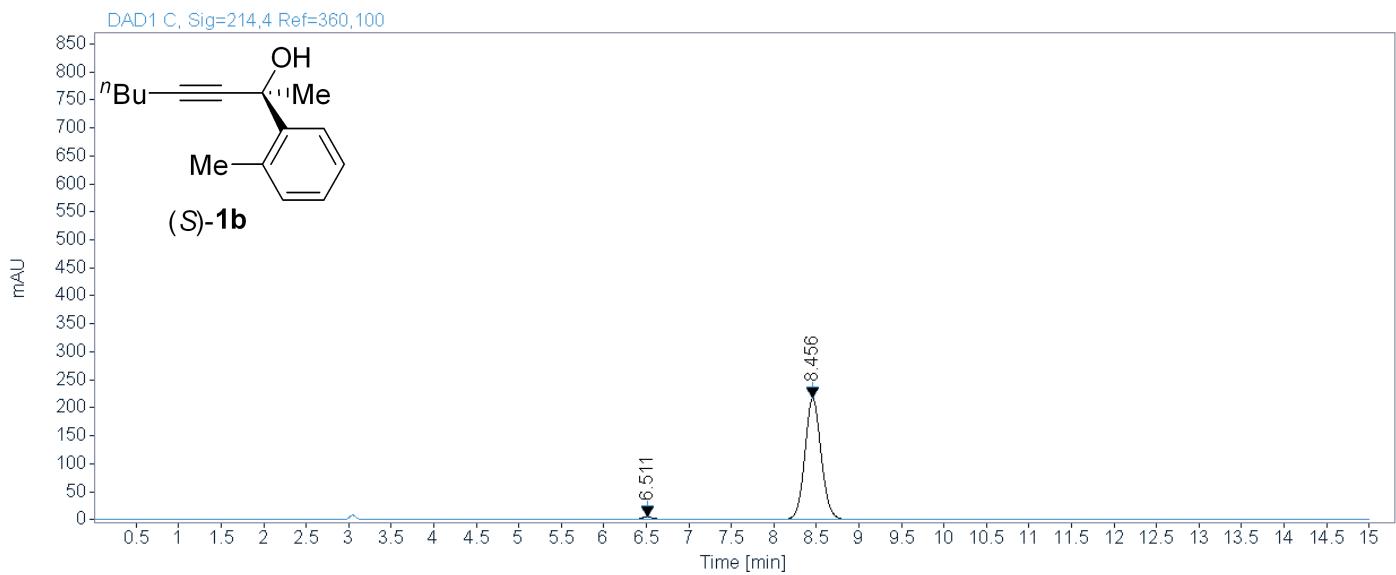
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sample wj-1-017-1-AS-H-98-2-1.0-214

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Acquisition Data:



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RT [min]	Width [min]	Height	Area	Area%
6.511	0.1597	4.5076	47.0272	1.5953
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		Sum	2947.7741	100.0000

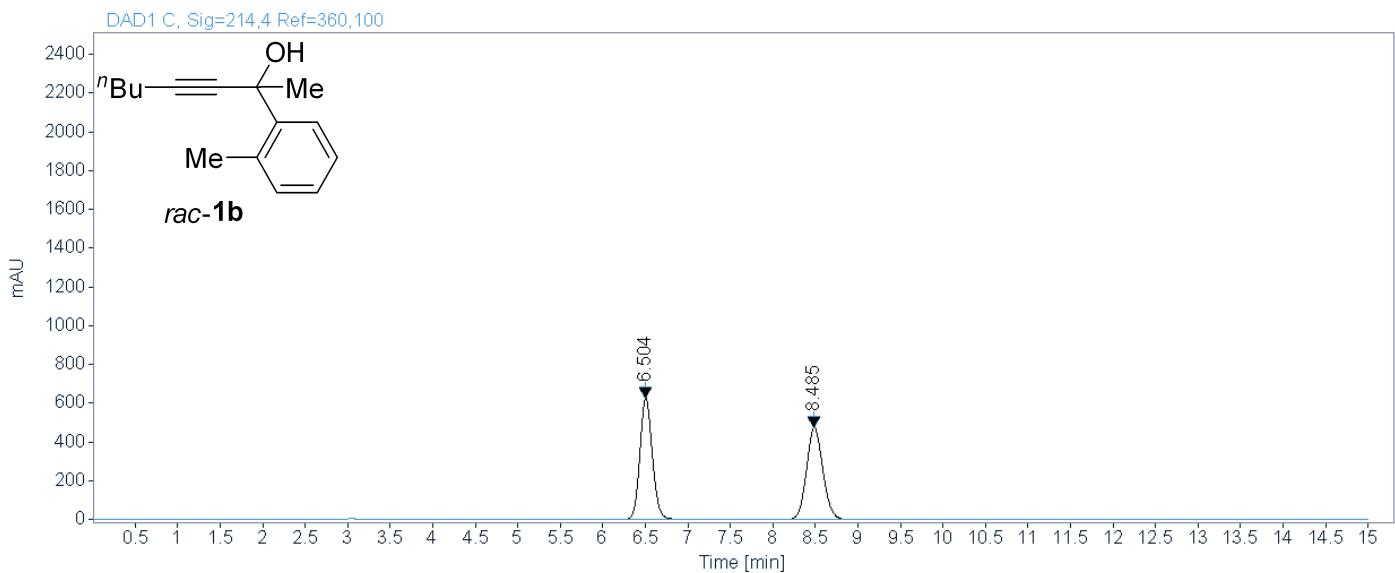
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214

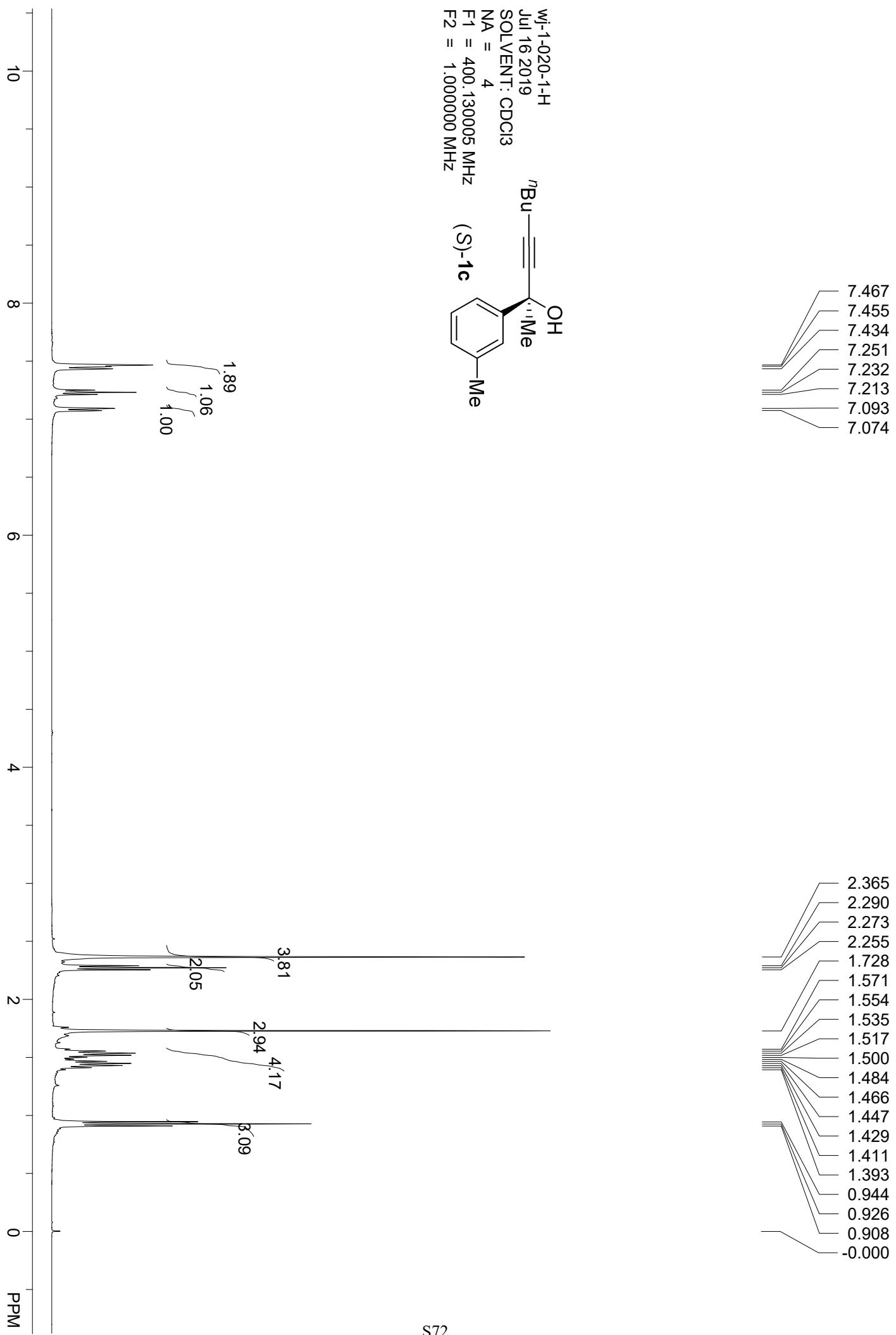
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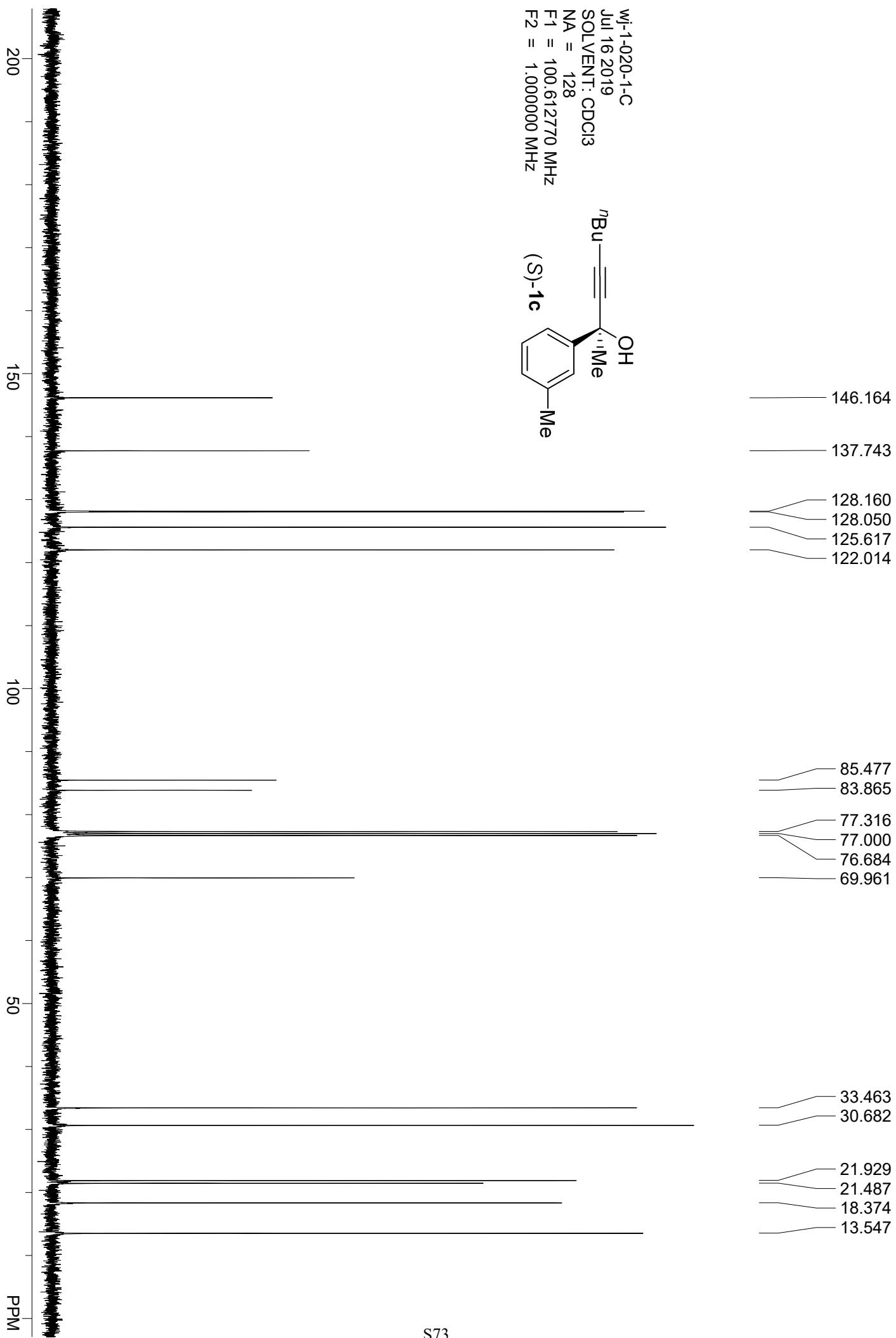
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Signal: DAD1 C, Sig=214,4 Ref=360,100

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8.485	0.2092	475.5550	6409.4746	49.9166
Sum		12840.3550	100.0000	





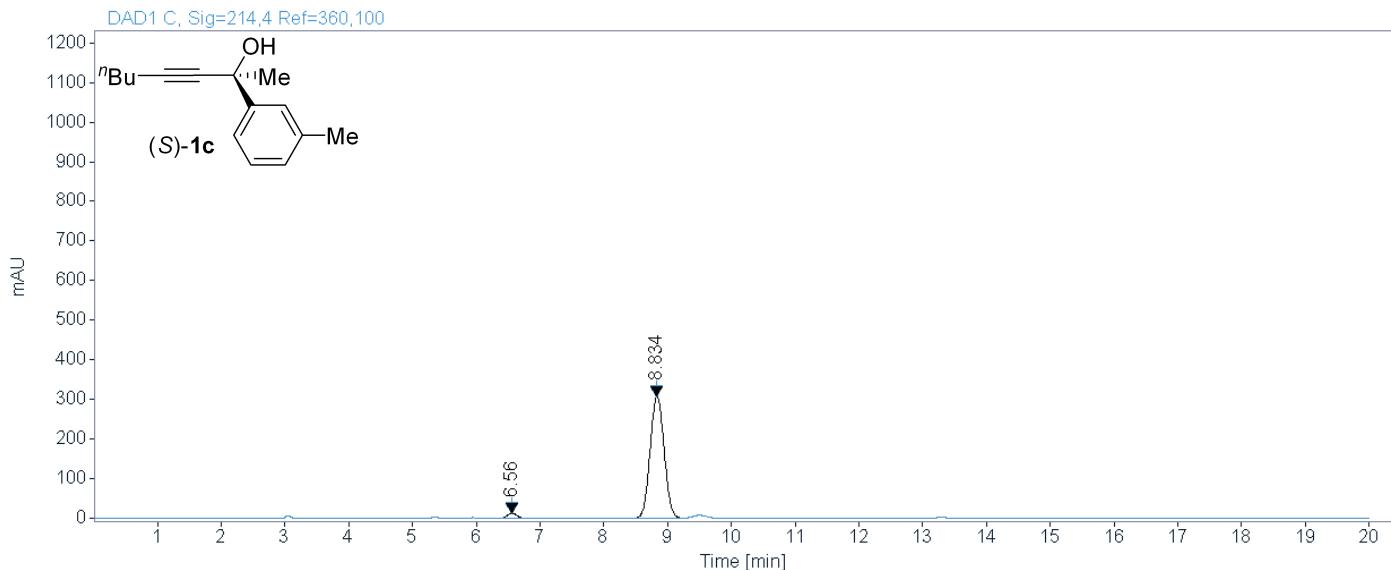
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sample wj-1-020-1-AS-H-98-2-1.0-214

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Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
6.560	0.1728	13.1455	145.0943	2.9778
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		Sum	4872.6104	100.0000

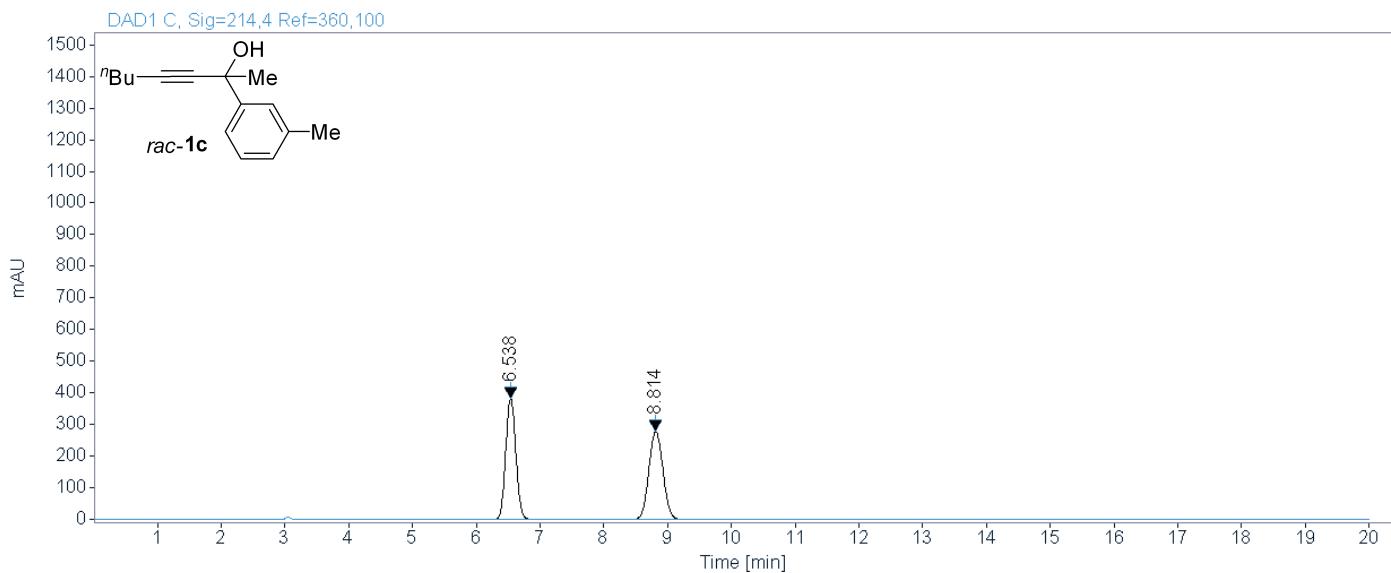
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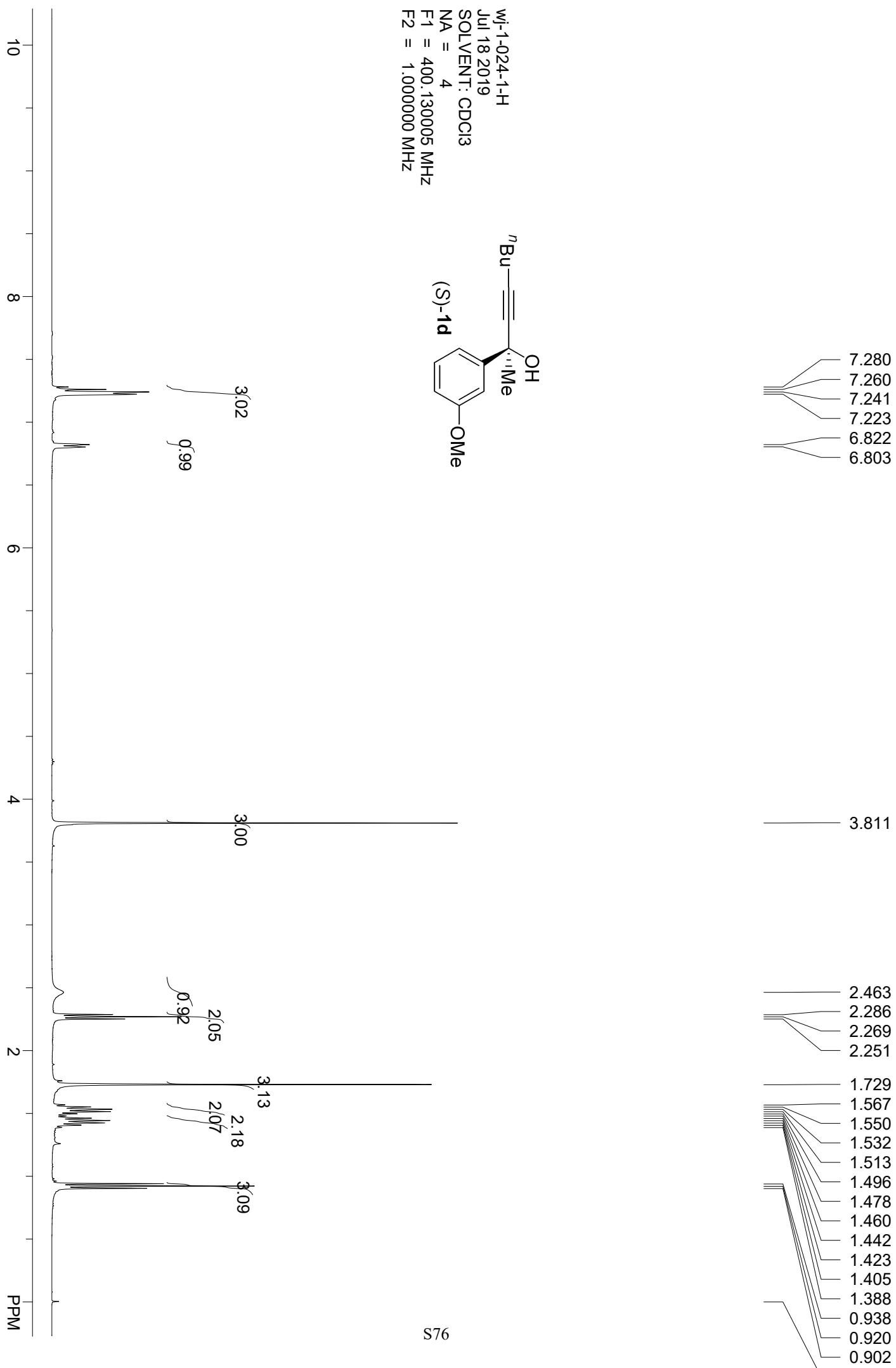
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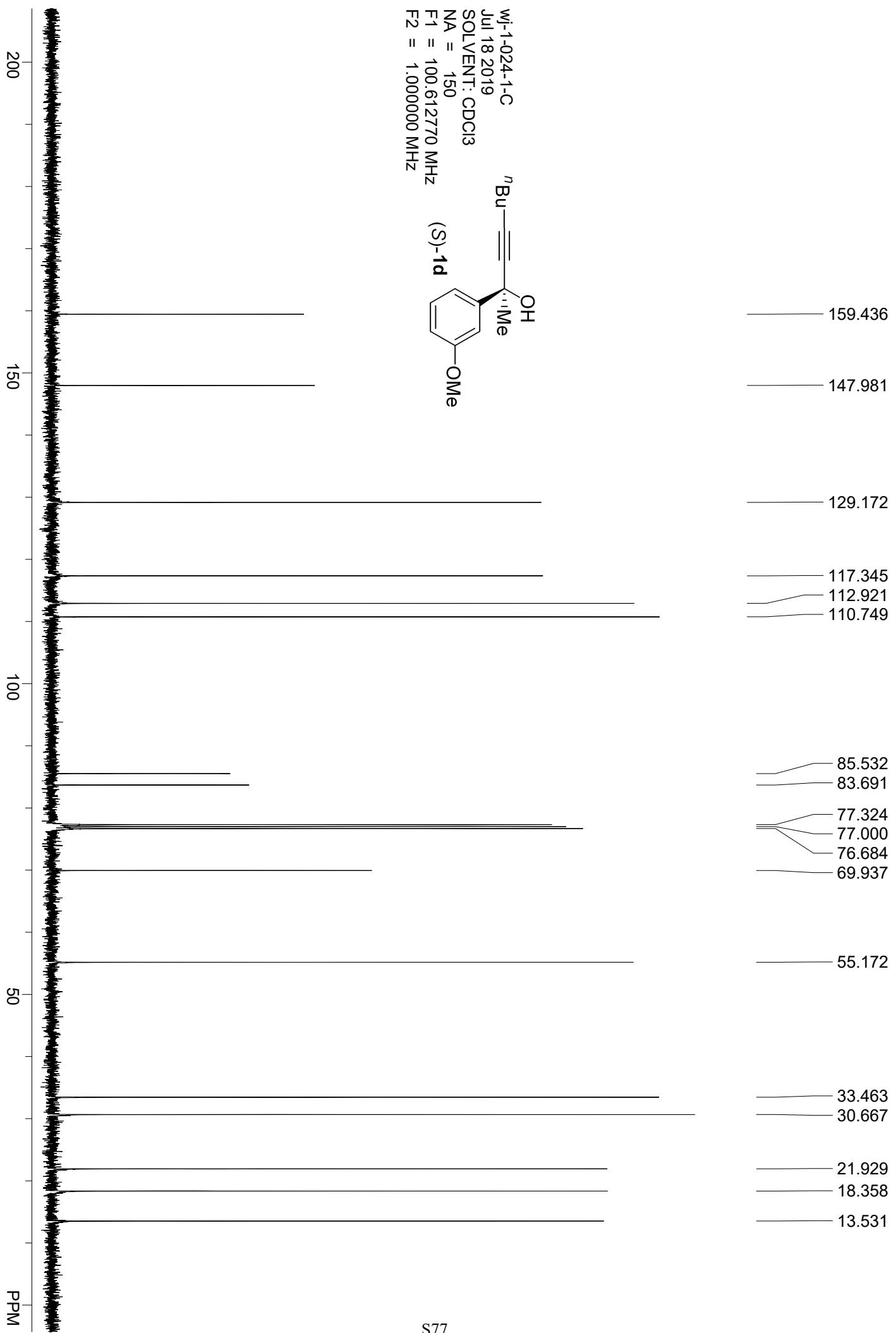
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RT [min]	Width [min]	Height	Area	Area%
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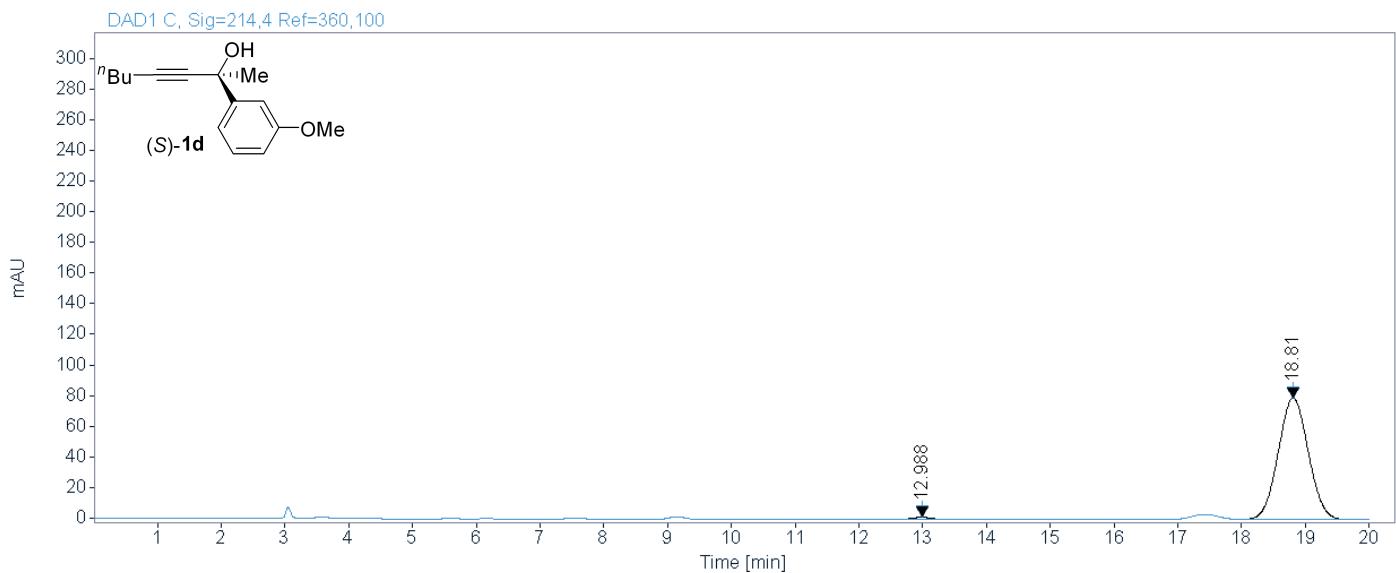
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Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
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18.810	0.5153	79.4234	2609.1577	98.7425
Sum			2642.3867	100.0000

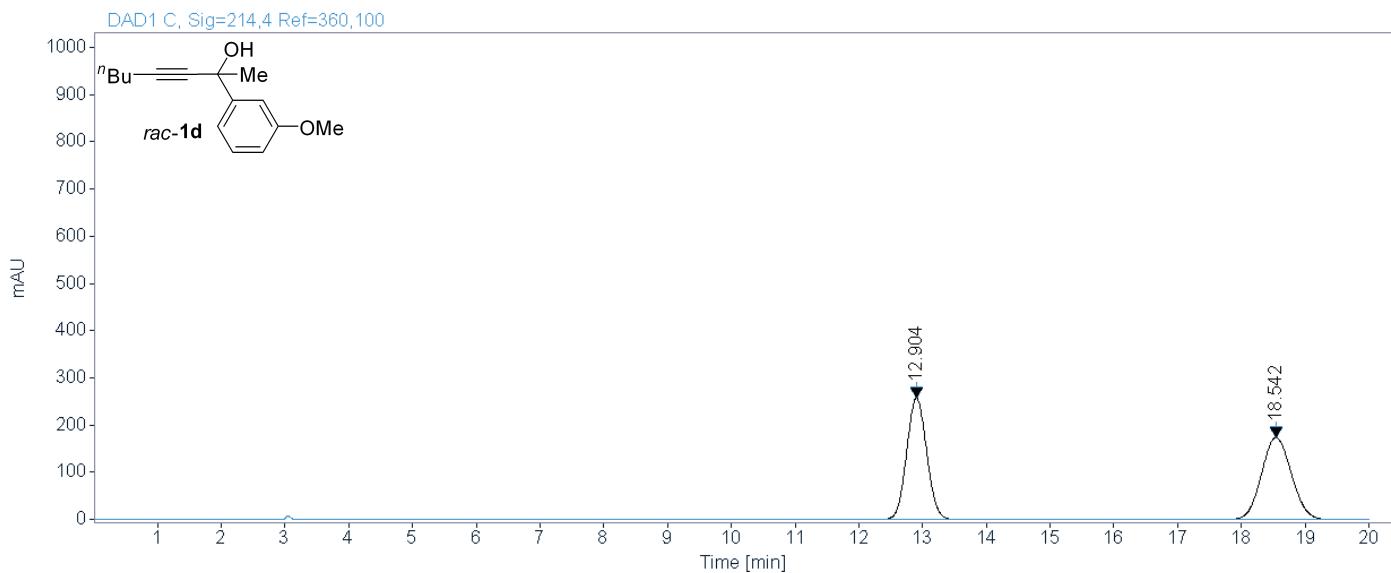
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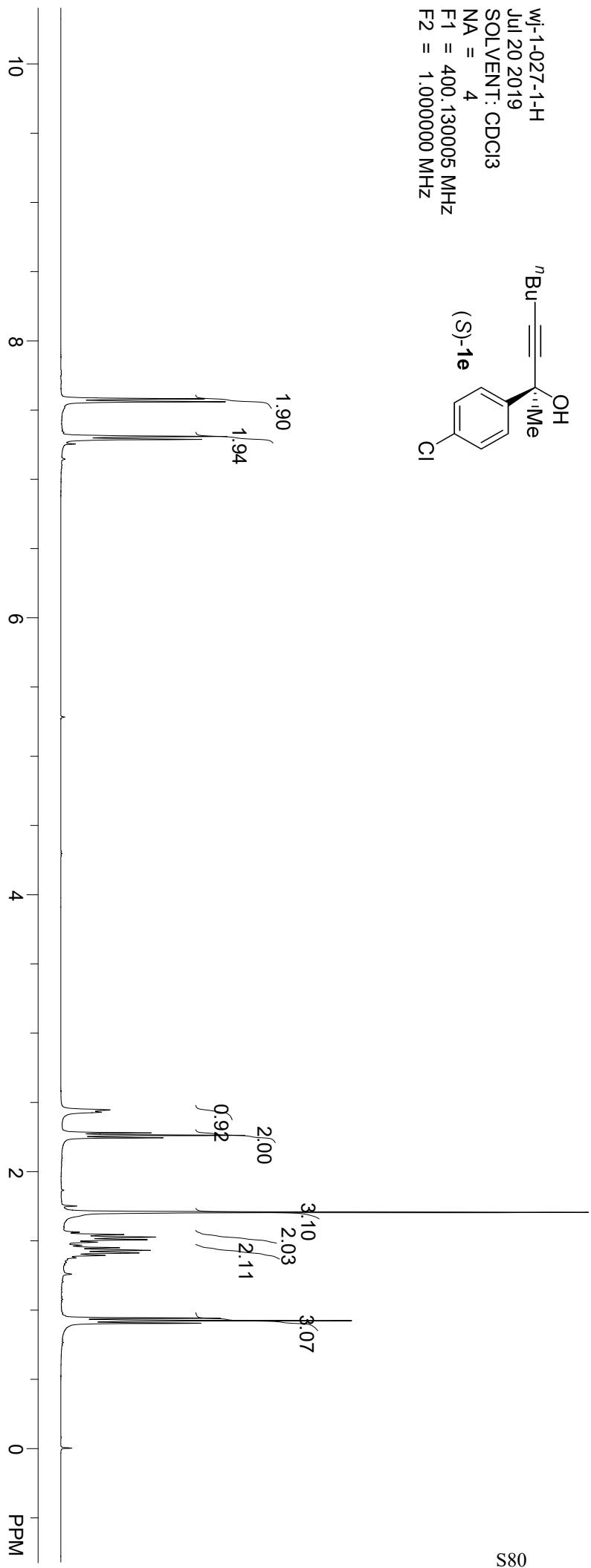
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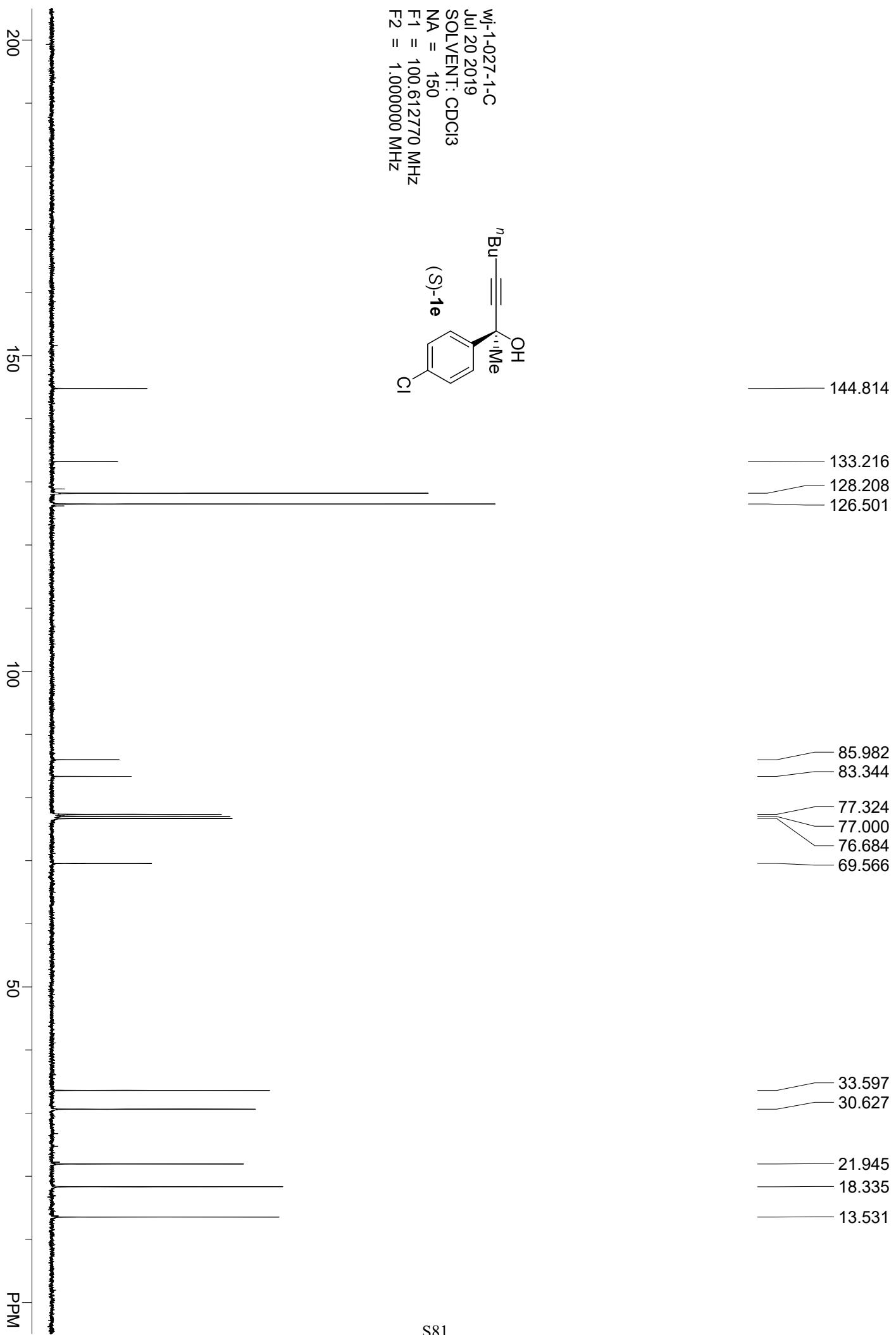
Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
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Sum		11297.1133	100.0000	





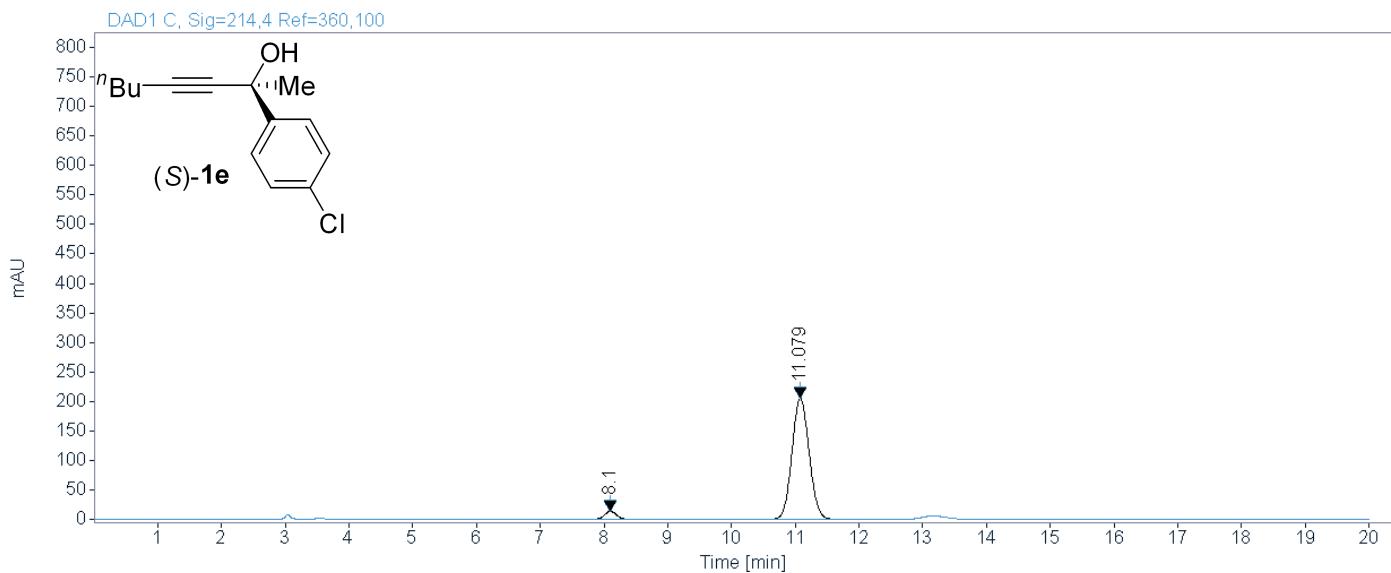
Area Percent Report



sample wj-1-027-1-AS-H-98-2-1.0-214

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Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
8.100	0.2085	13.5342	181.5527	4.4689
11.079	0.2951	206.6439	3881.0625	95.5311
Sum		4062.6152	100.0000	

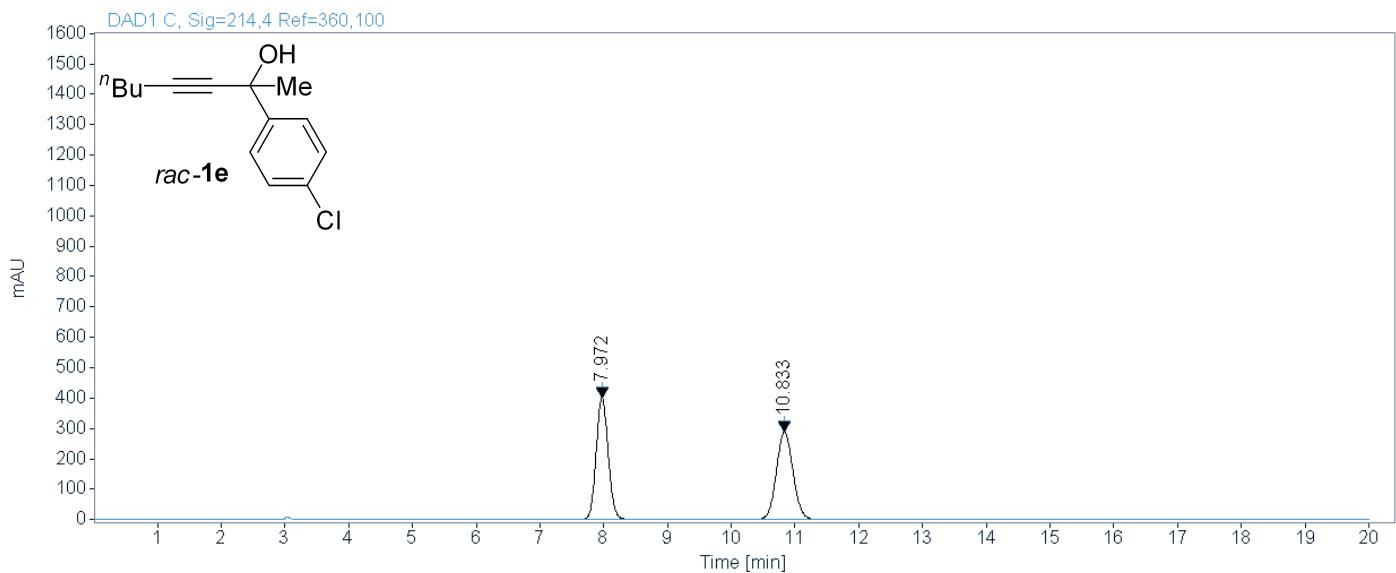
Area Percent Report



sample wj-1-027-1-rac-AS-H-98-2-1.0-214

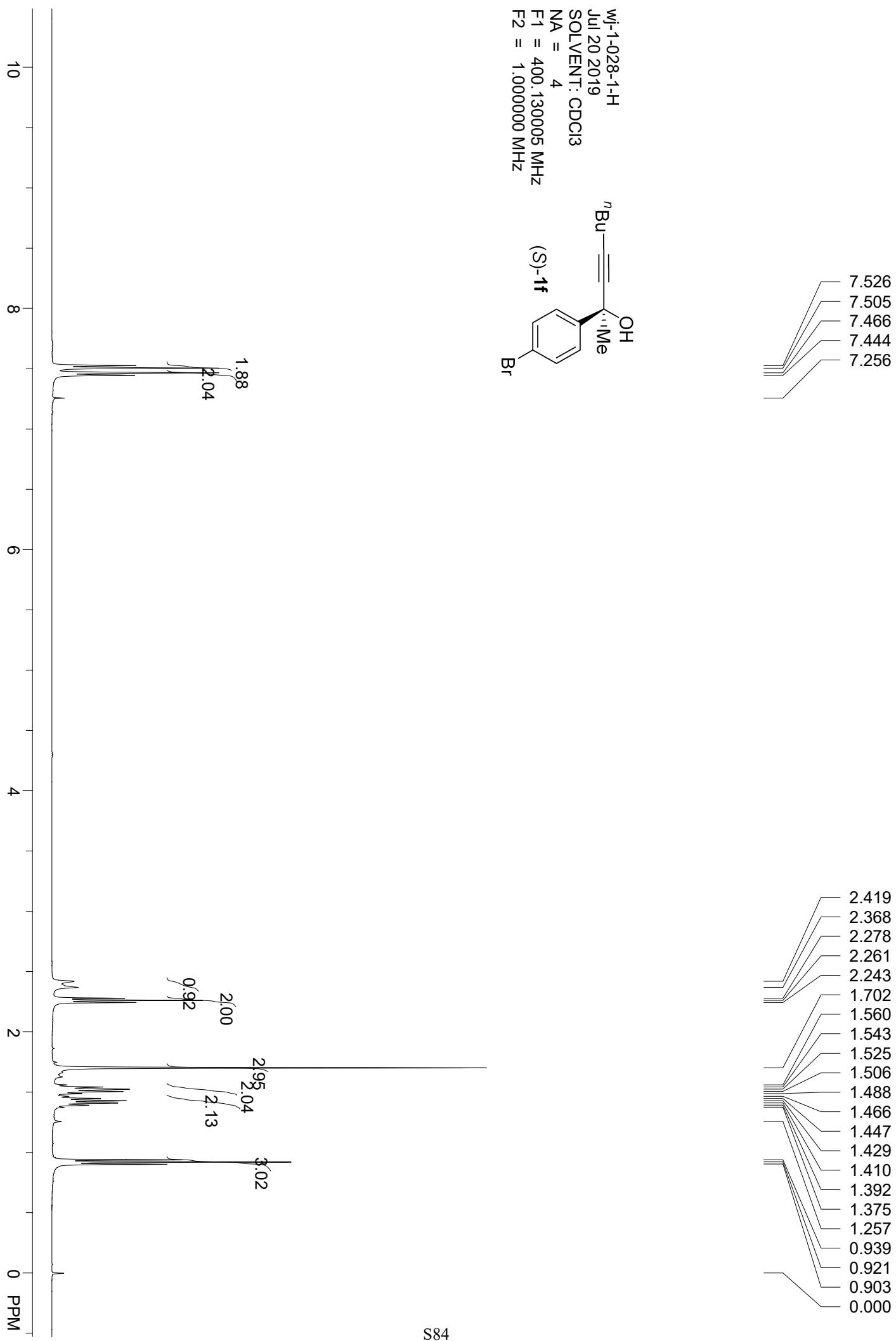
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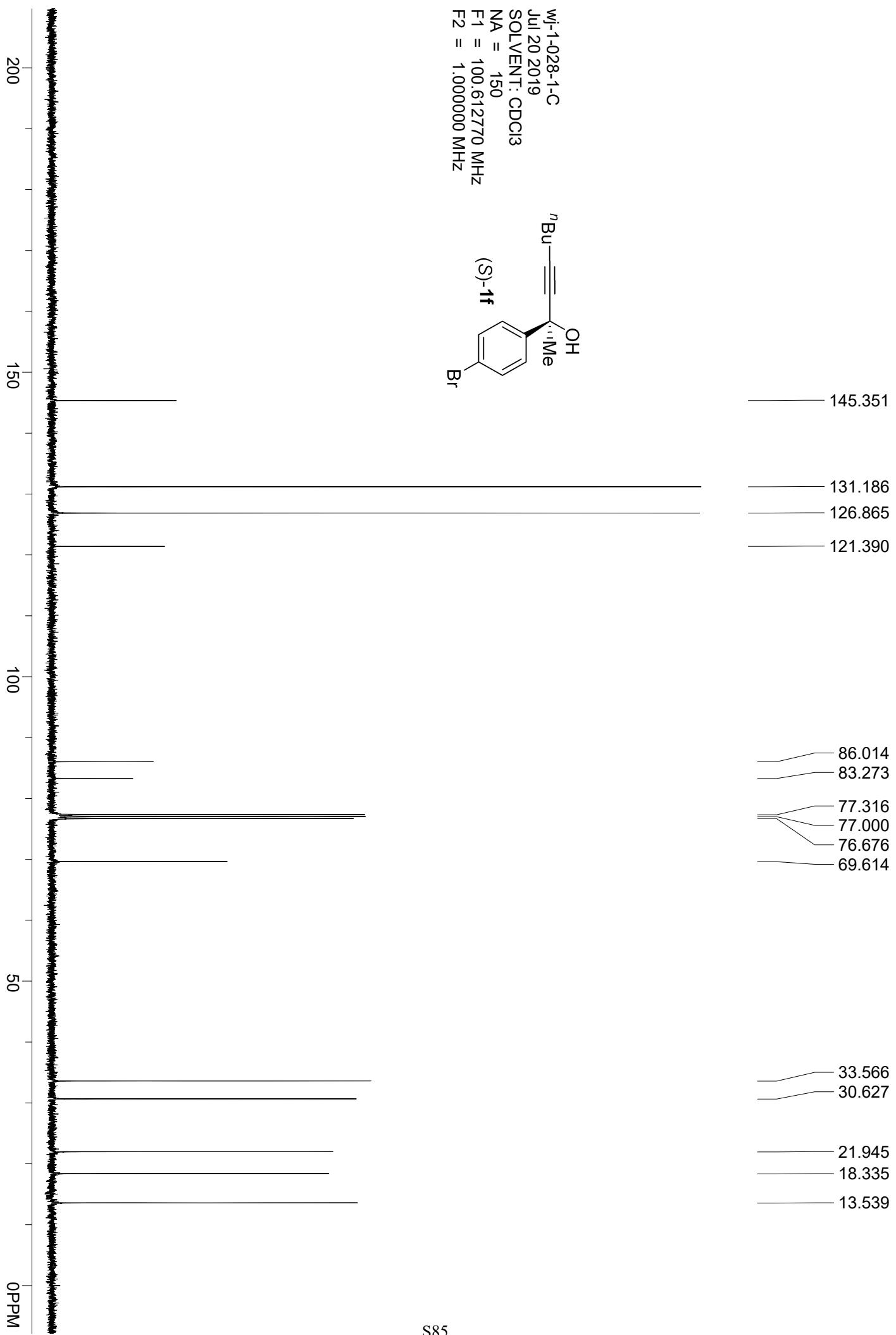
Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
7.972	0.2023	400.7166	5230.1724	49.9318
10.833	0.2865	287.8897	5244.4502	50.0682
Sum		10474.6226	100.0000	





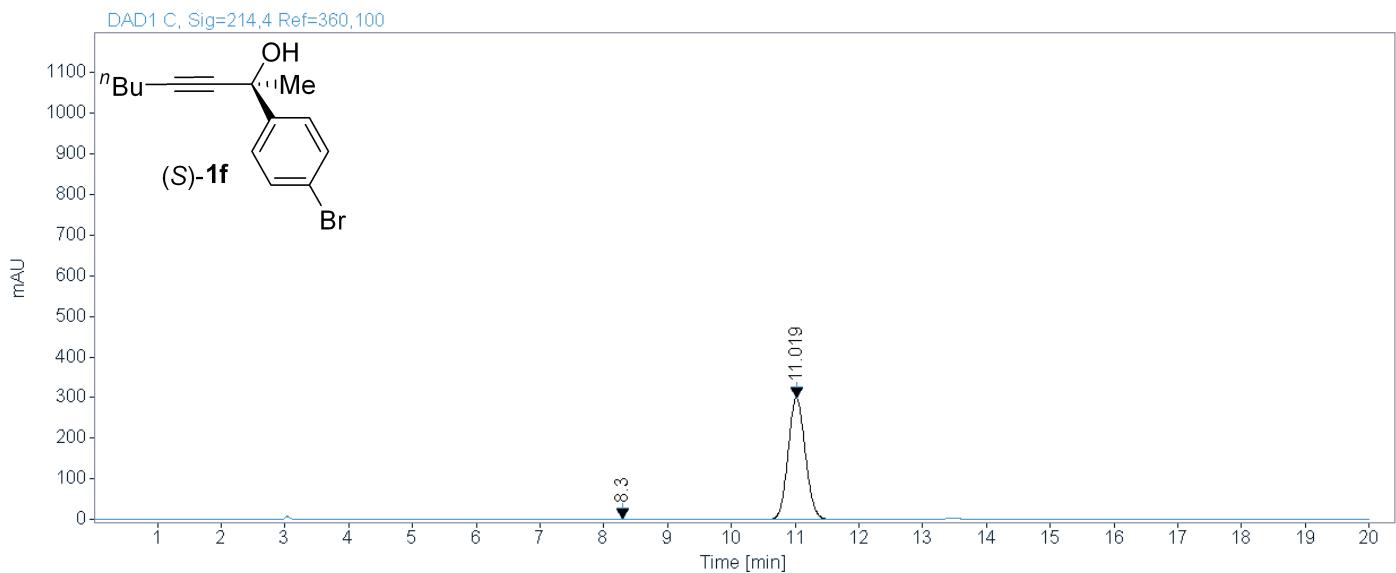
Area Percent Report



sample wj-1-028-1-AS-H-98-2-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\WJ_LC 2019-07-20 19-12-56\022-P2-C1-wj-1-028-1.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
8.300	0.2195	1.1975	16.9838	0.2998
11.019	0.2957	299.9573	5648.5952	99.7002
Sum		5665.5790	100.0000	

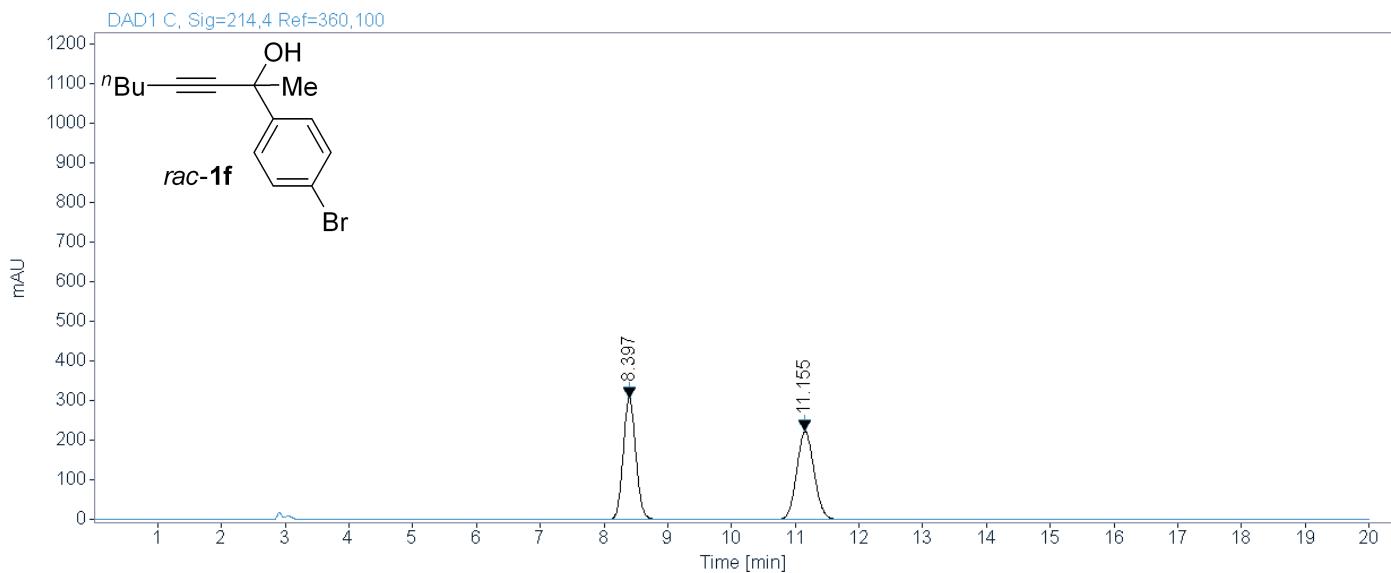
Area Percent Report



sample wj-1-028-1-rac-AS-H-98-2-1.0-214

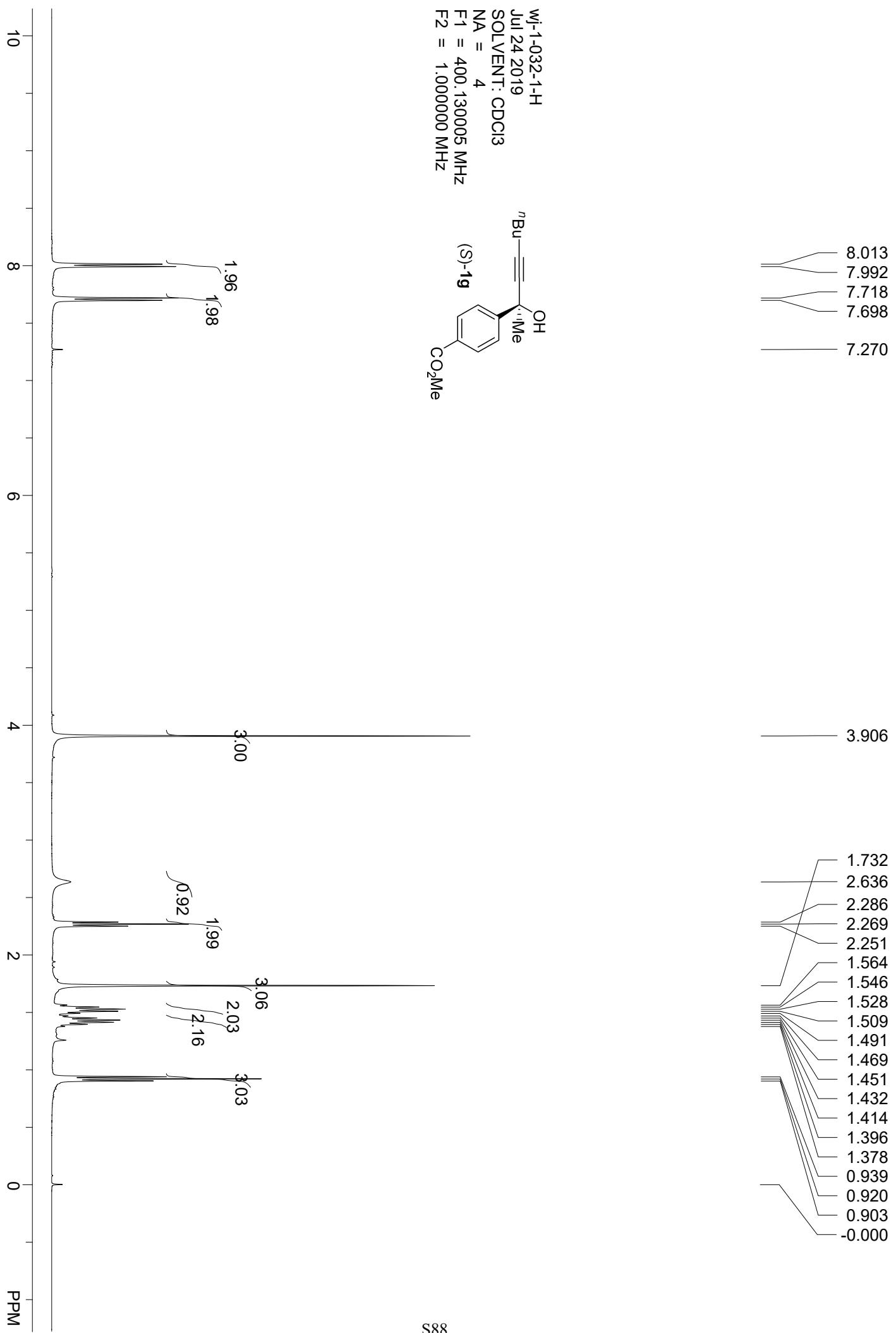
Data file: C:\Users\Public\Documents\ChemStation\1\Data\WJ_LC 2019-07-20 19-12-56\023-P2-C2-wj-1-028-1-rac.D

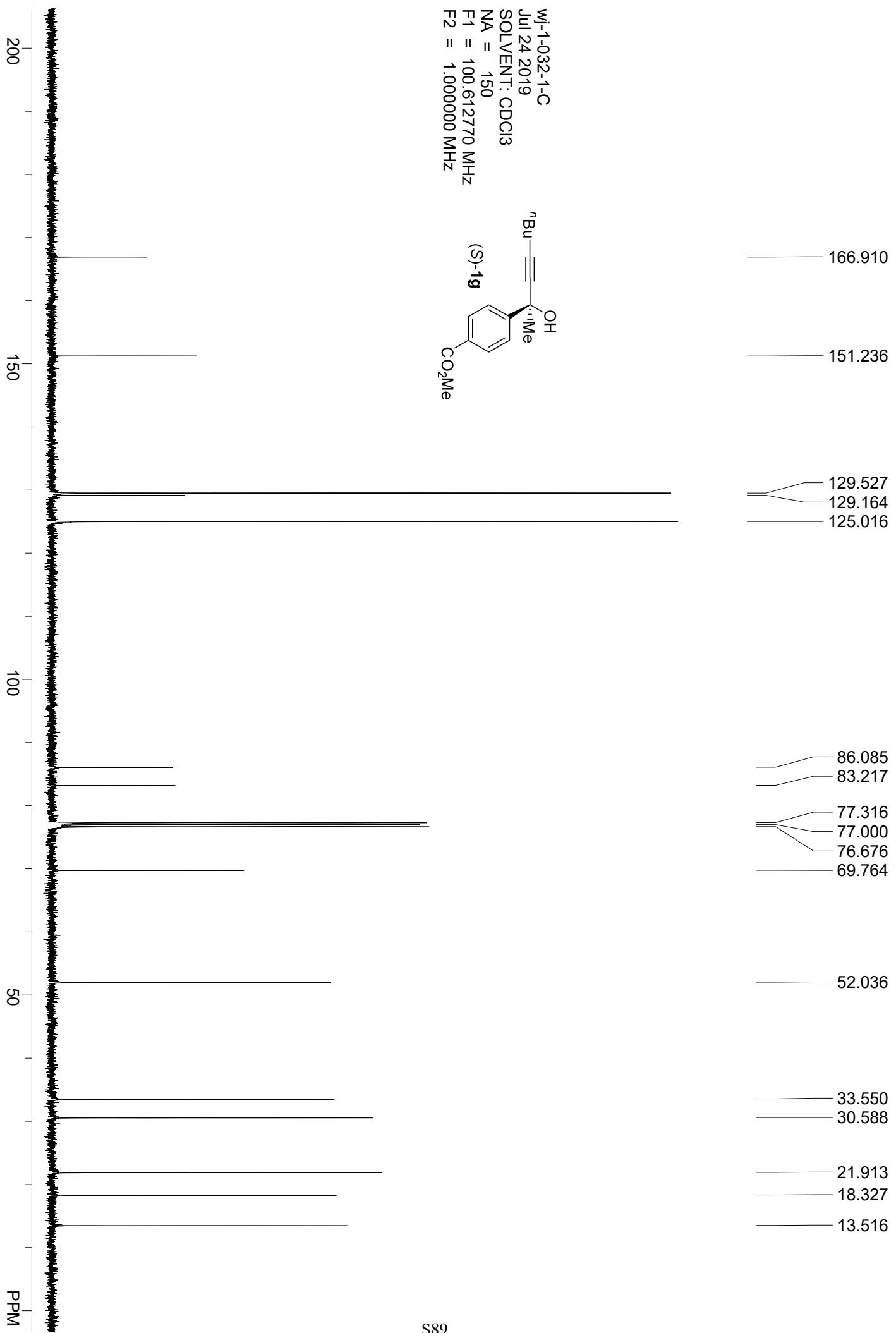
Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
8.397	0.2152	307.5428	4248.5869	49.9716
11.155	0.2989	222.6530	4253.4209	50.0284
Sum		8502.0078	100.0000	





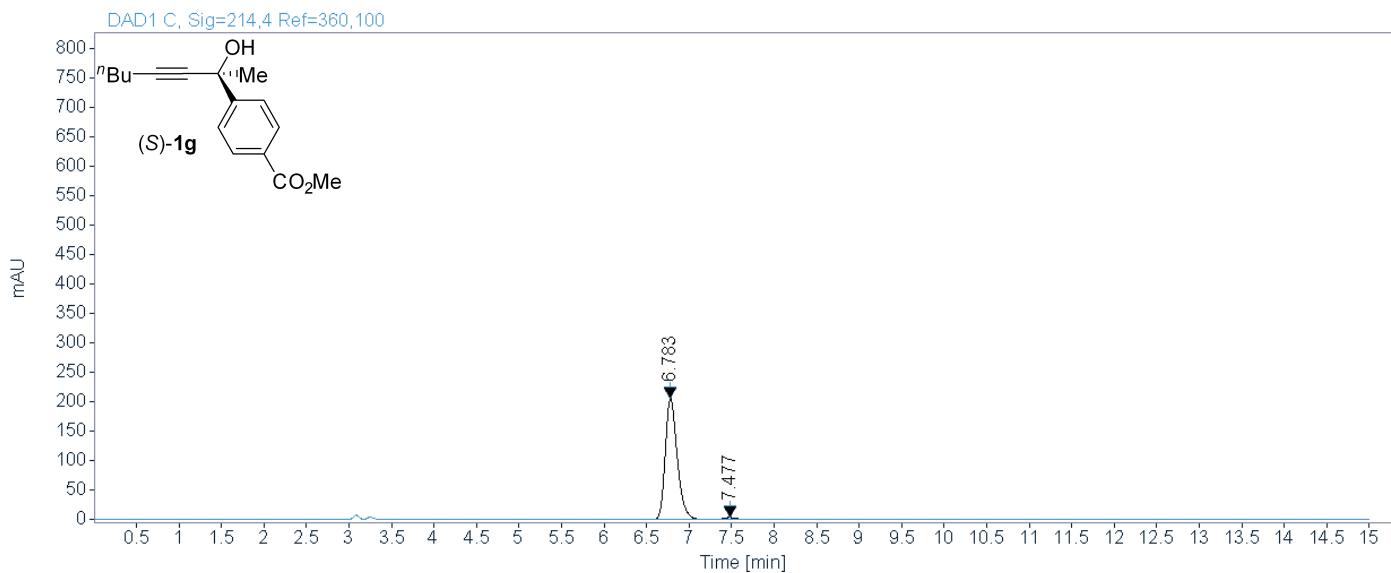
Area Percent Report



sample wj-1-032-1-AS-H-90-10-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\WJ_LC 2019-07-24 08-34-00\030-P2-C3-wj-1-032-1.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
6.783	0.1484	206.8252	2027.9703	98.4971
7.477	0.1532	3.1326	30.9439	1.5029
Sum		2058.9142	100.0000	

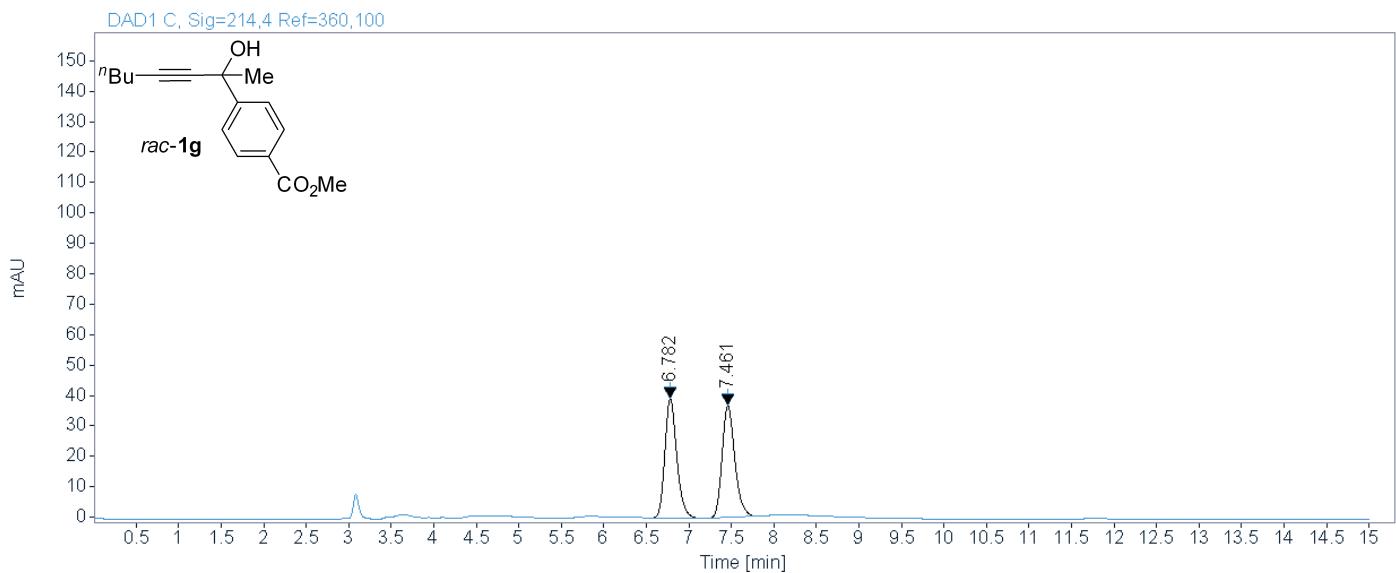
Area Percent Report



sample wj-1-032-1-rac-AS-H-90-10-1.0-214

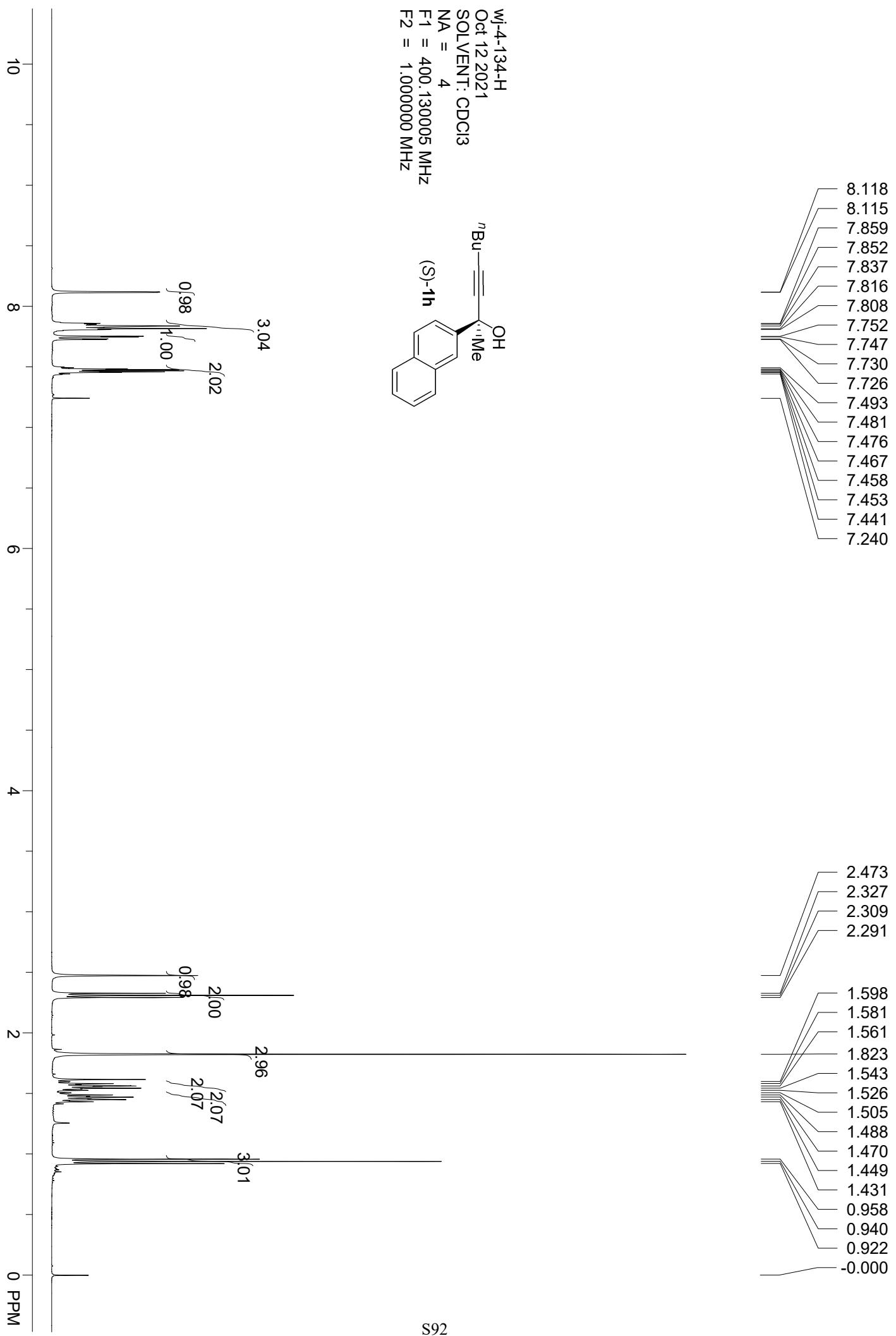
Data file: C:\Users\Public\Documents\ChemStation\1\Data\WJ_LC 2019-07-24 08-34-00\031-P2-C4-wj-1-032-1-rac.D

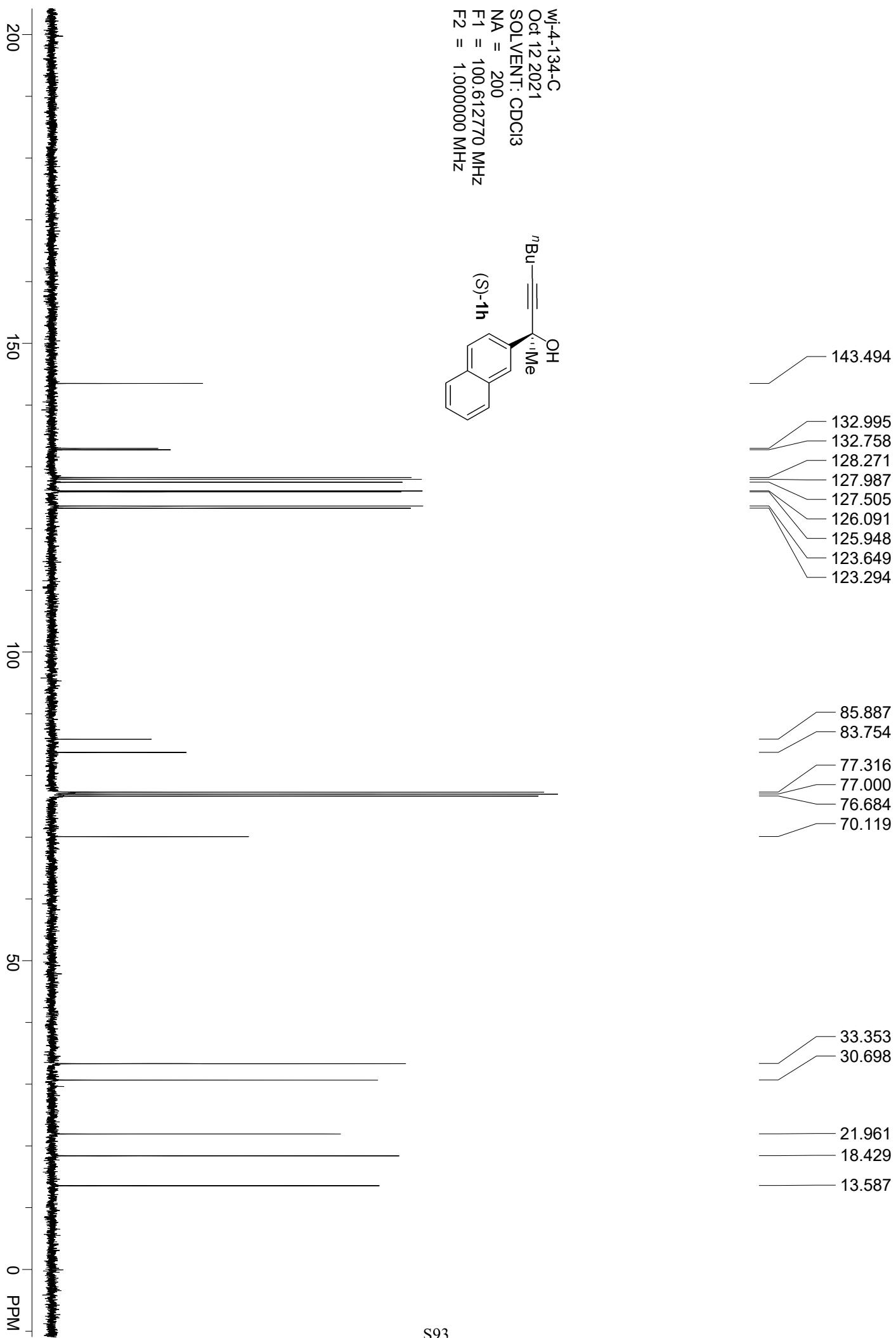
Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
6.782	0.1502	39.5373	386.8459	50.1070
7.461	0.1618	36.8955	385.1939	49.8930
Sum		772.0398	100.0000	





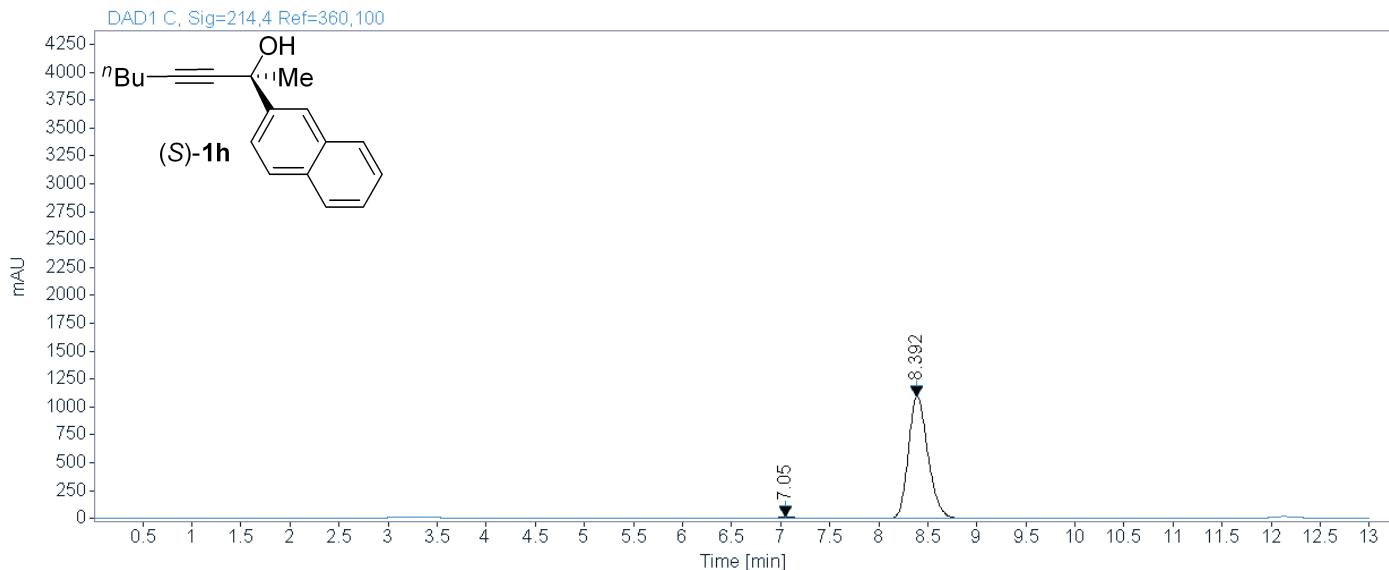
Area Percent Report



sample wj-4-134-AS-H-95-5-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\WJ_LC 2021-10-12 15-26-33\002-P2-C1-wj-4-134.D

Acquisition Data:



Signal: DAD1 C, Sig=214.4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
7.050	0.1655	15.7134	171.7532	1.1175
8.392	0.2140	1094.6259	15198.0674	98.8825
Sum		15369.8206	100.0000	

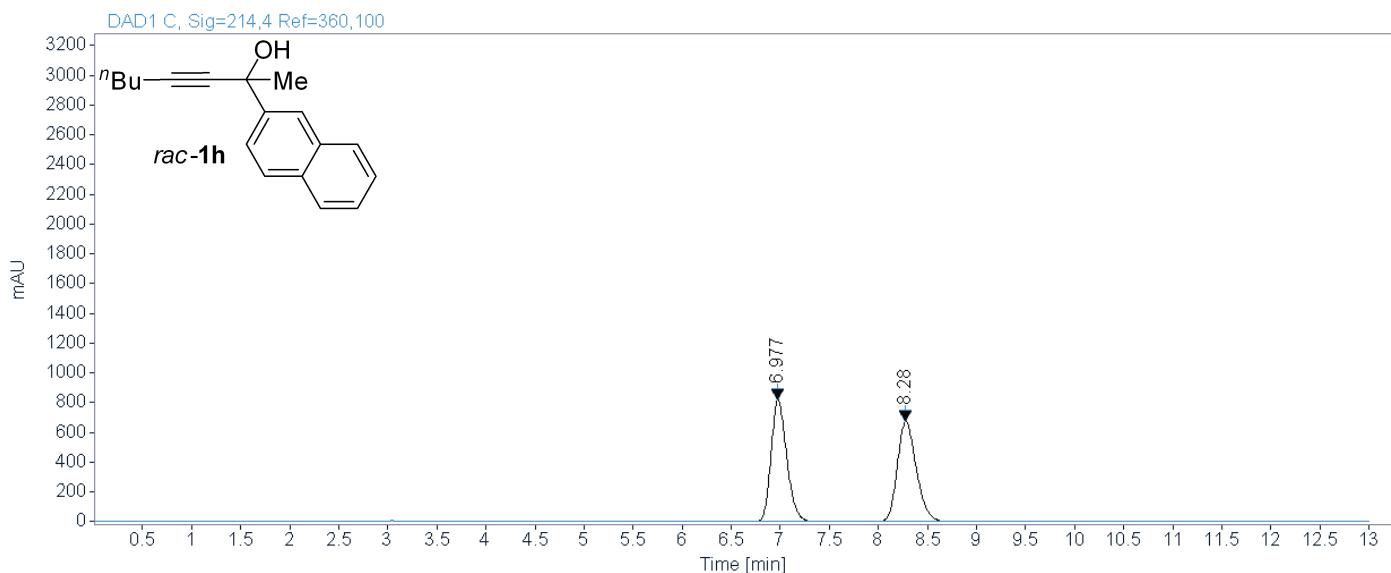
Area Percent Report



sample wj-4-134-rac-AS-H-95-5-1.0-214

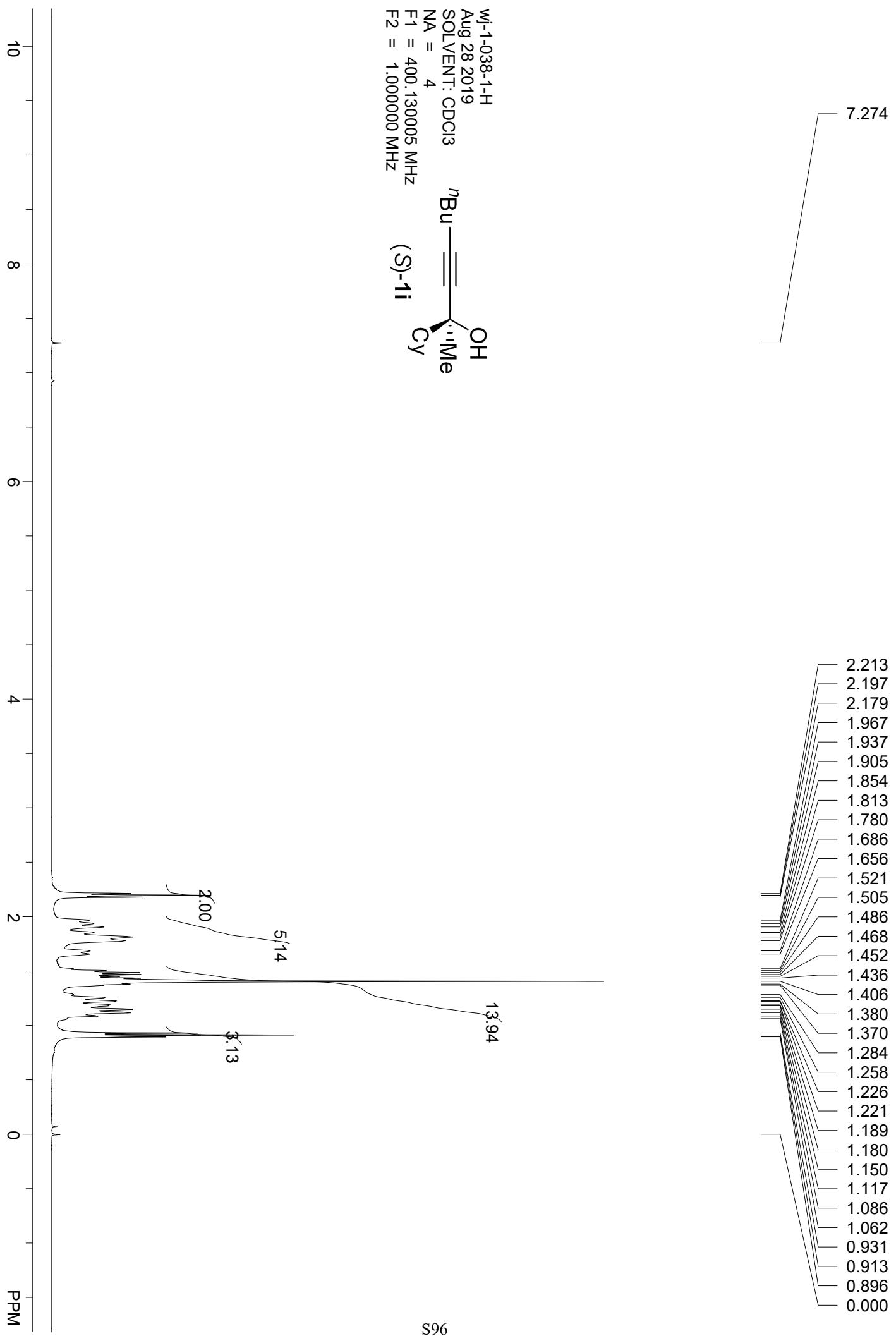
Data file: C:\Users\Public\Documents\ChemStation\1\Data\WJ_LC 2021-10-12 15-26-33\003-P2-C2-wj-4-134--rac.D

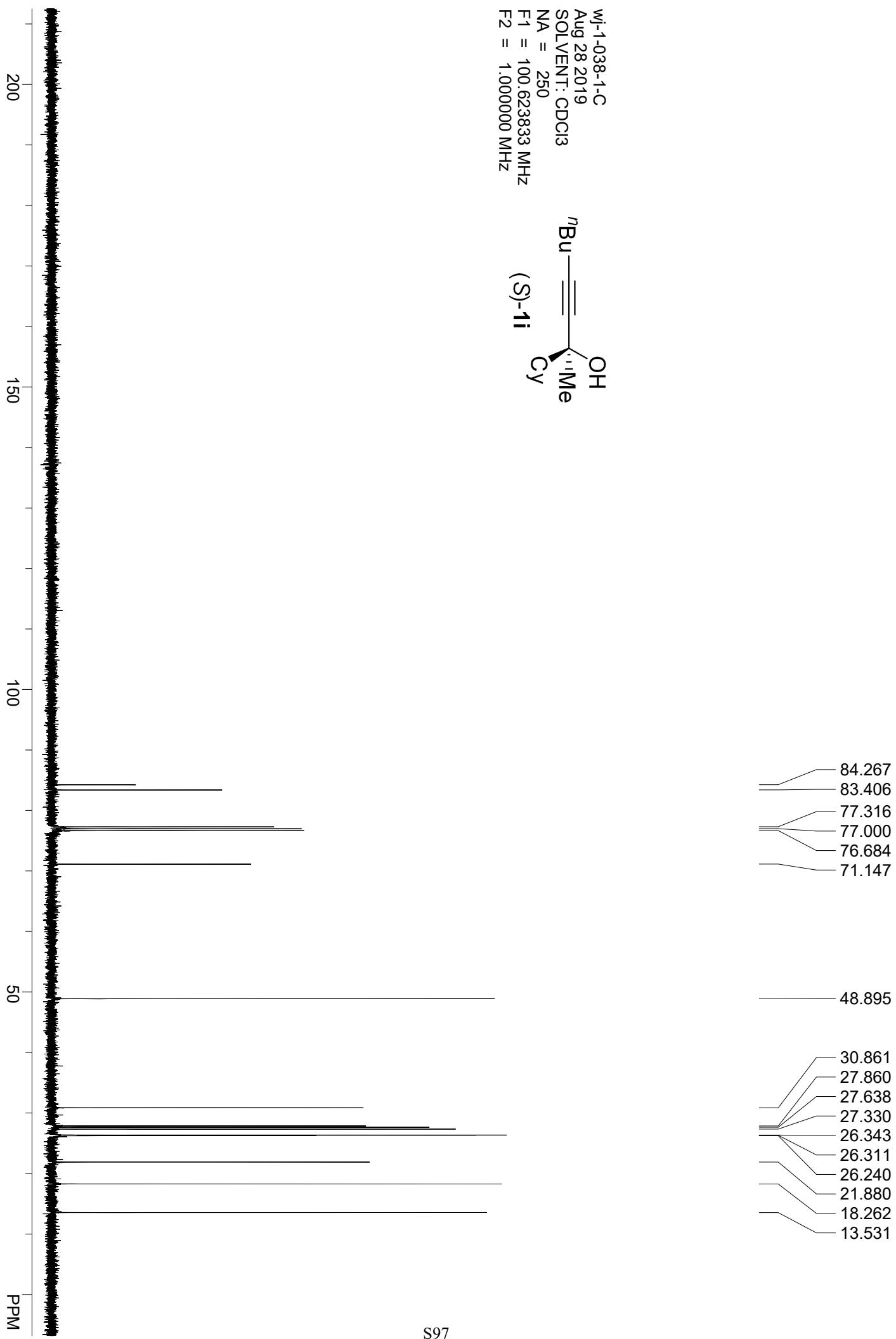
Acquisition Data:



Signal: DAD1 C, Sig=214.4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
6.977	0.1845	821.0001	9090.6621	50.0233
8.280	0.2071	674.5023	9082.1826	49.9767
Sum		18172.8447	100.0000	





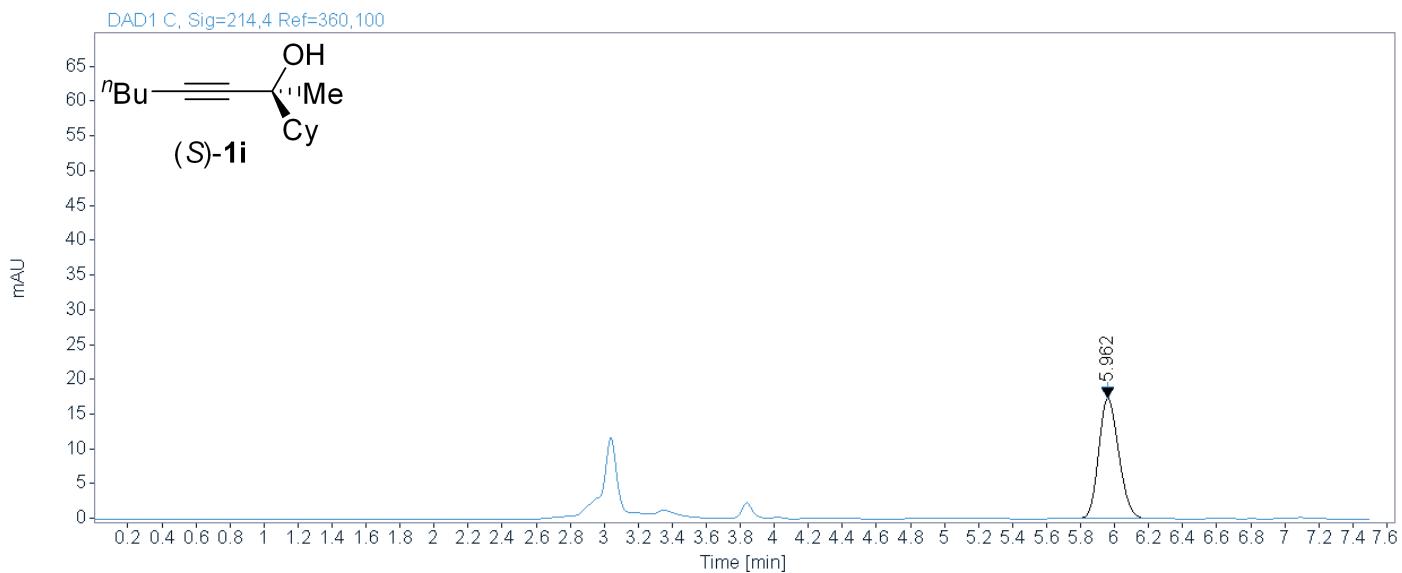
Area Percent Report



sample wj-1-038-1-AS-H-98-2-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\DEF_LC 2019-09-04 09-57-05\035-P2-C1-wj-1-038-1.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
5.962	0.1273	17.3202	141.5171	100.0000
		Sum	141.5171	100.0000

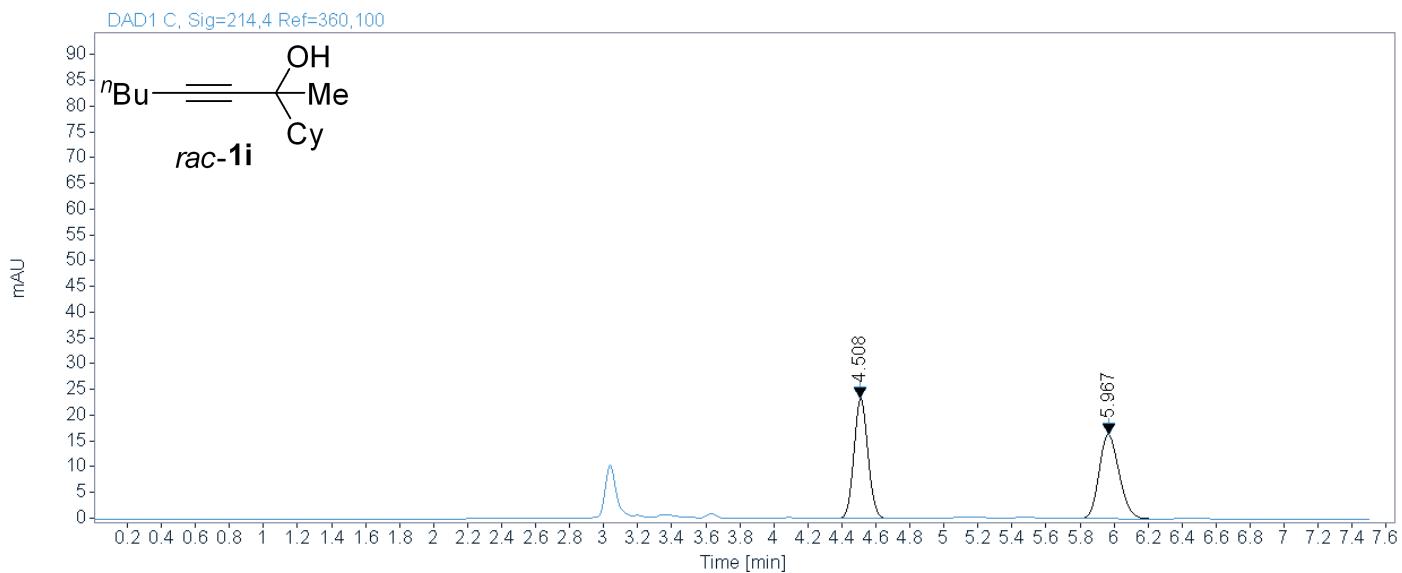
Area Percent Report



sample wj-1-038-1-rac-AS-H-98-2-1.0-214

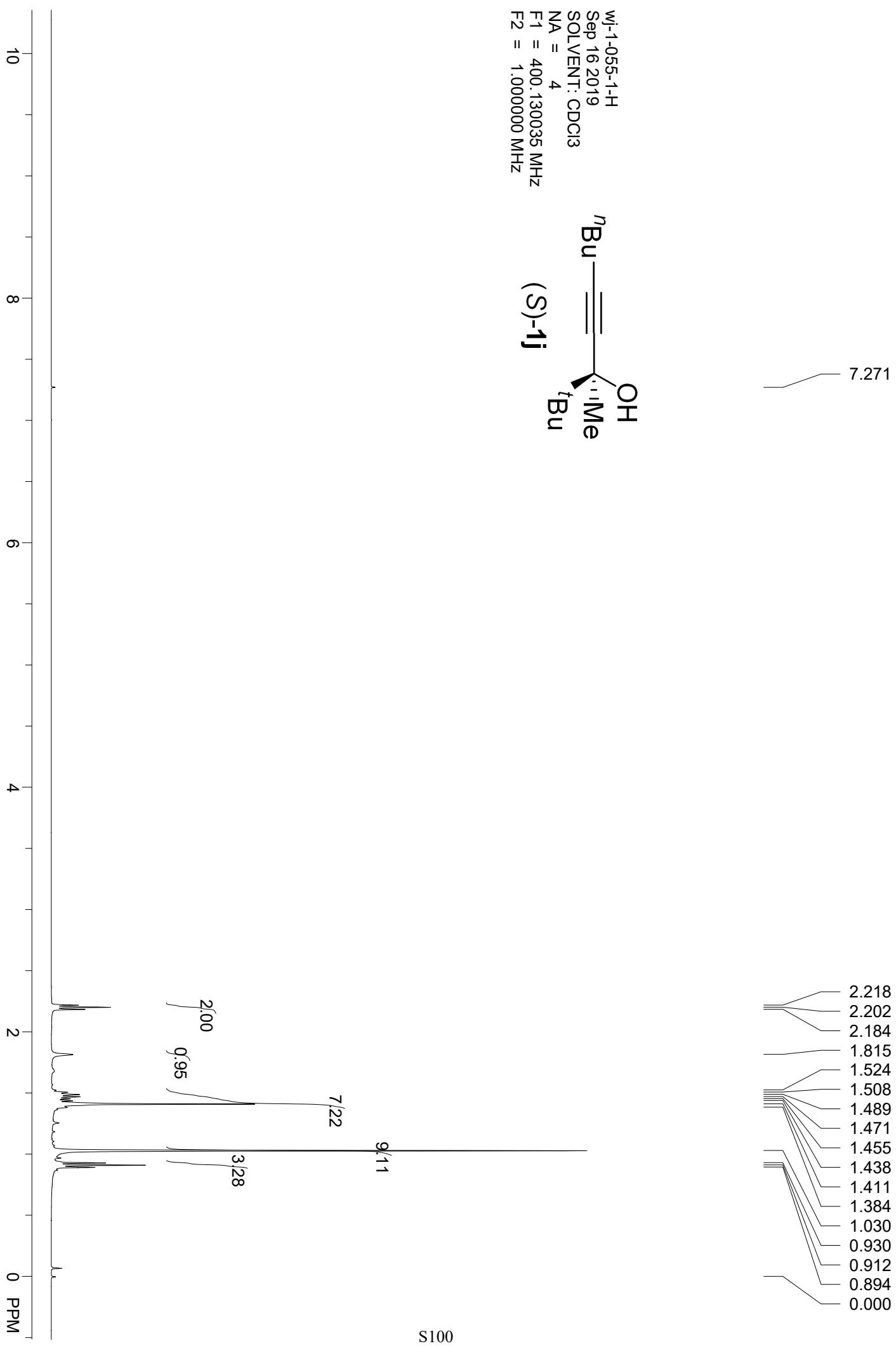
Data file: C:\Users\Public\Documents\ChemStation\1\Data\DEF_LC 2019-09-04 09-57-05\037-P2-C2-wj-1-038-1-rac.D

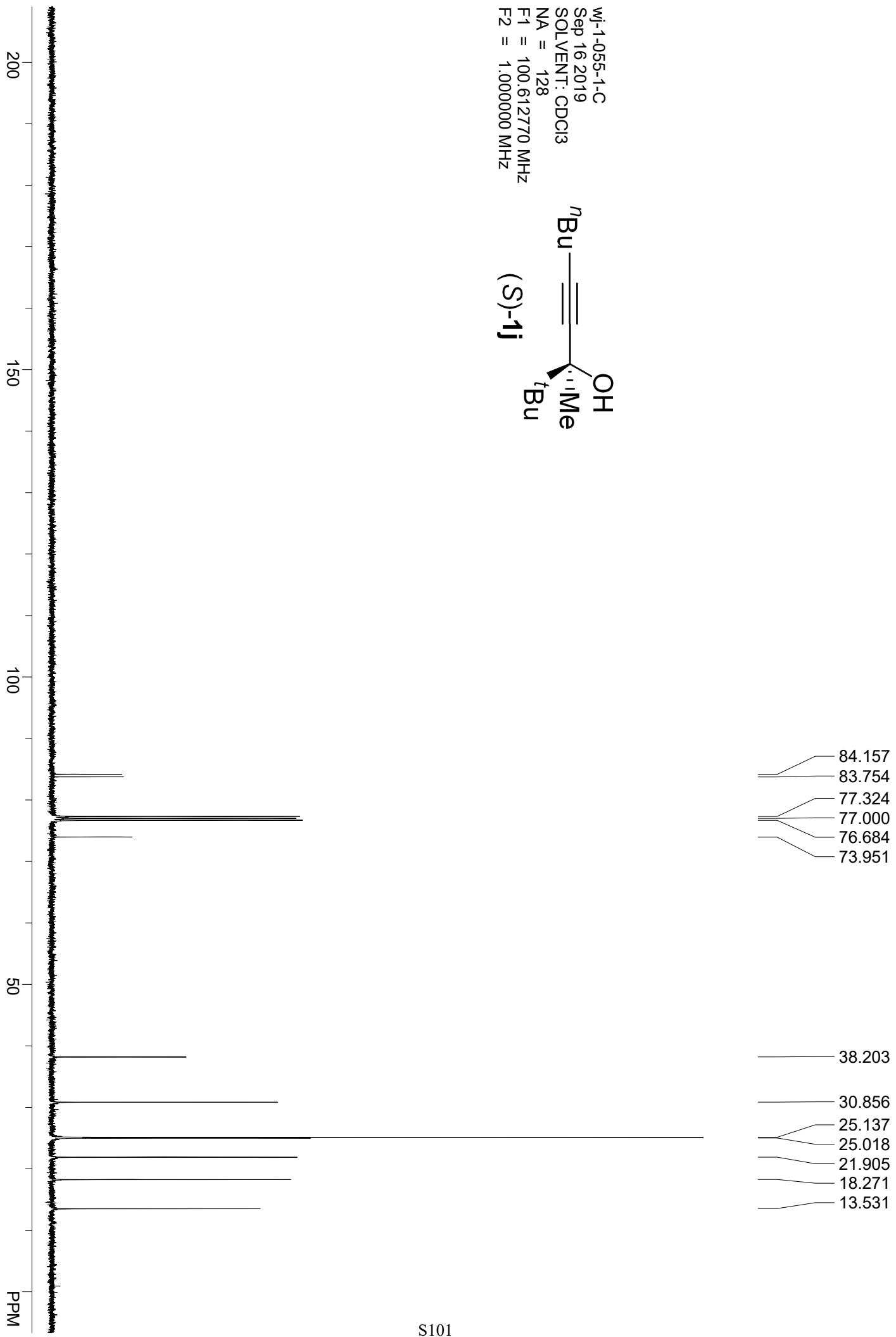
Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
4.508	0.0888	23.4767	134.3593	49.9786
5.967	0.1279	16.3616	134.4742	50.0214
		Sum	268.8335	100.0000





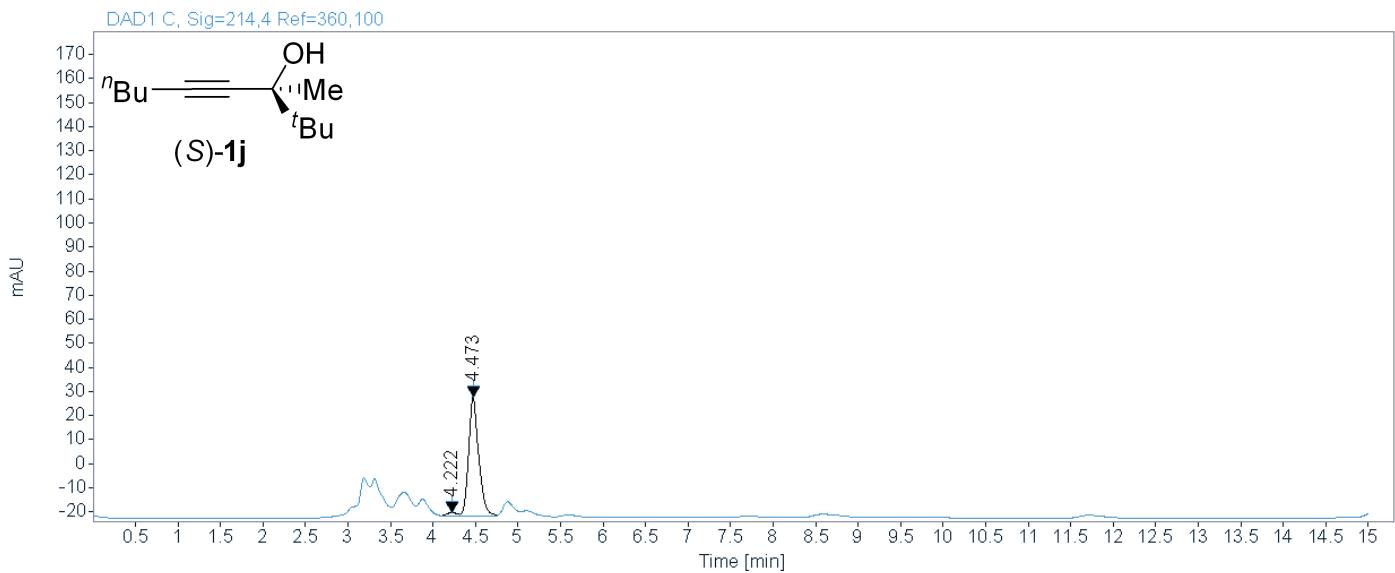
Area Percent Report



sample wj-1-055-1-IC-98-2-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\wgl 2019-09-17 09-02-49\060-P2-C5-wj-1-055-1.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
4.222	0.1355	1.2791	10.3955	2.4737
4.473	0.1379	49.5485	409.8443	97.5263
Sum		420.2397	100.0000	

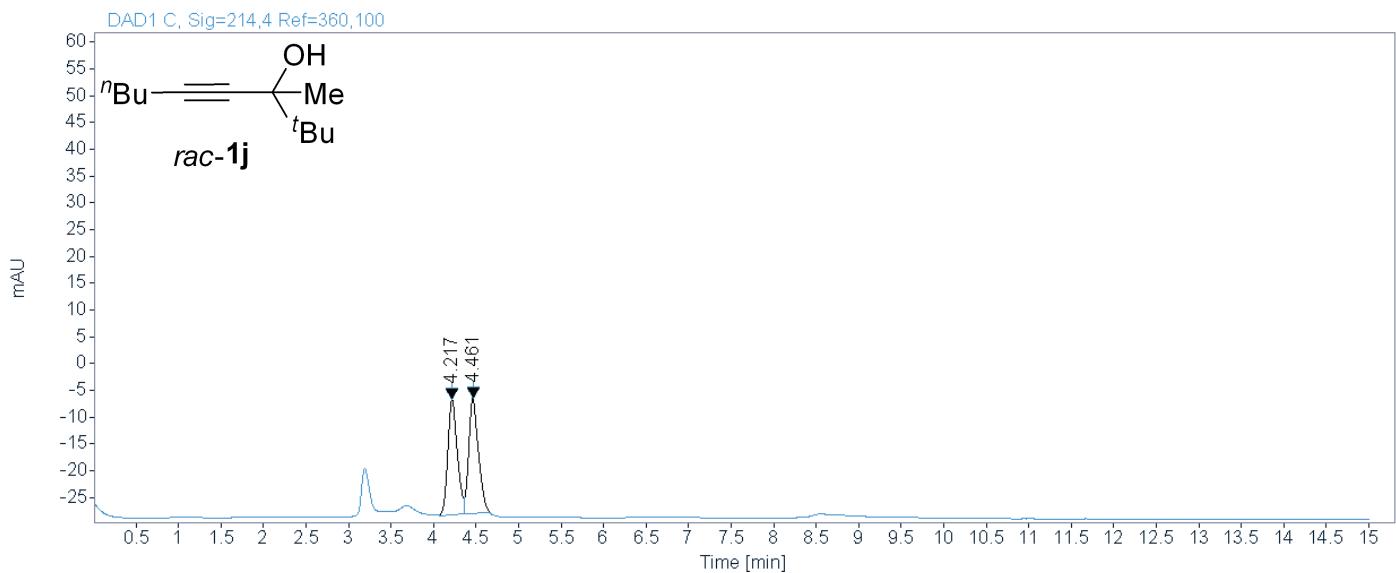
Area Percent Report



sample wj-1-055-1-rac-IC-98-2-1.0-214

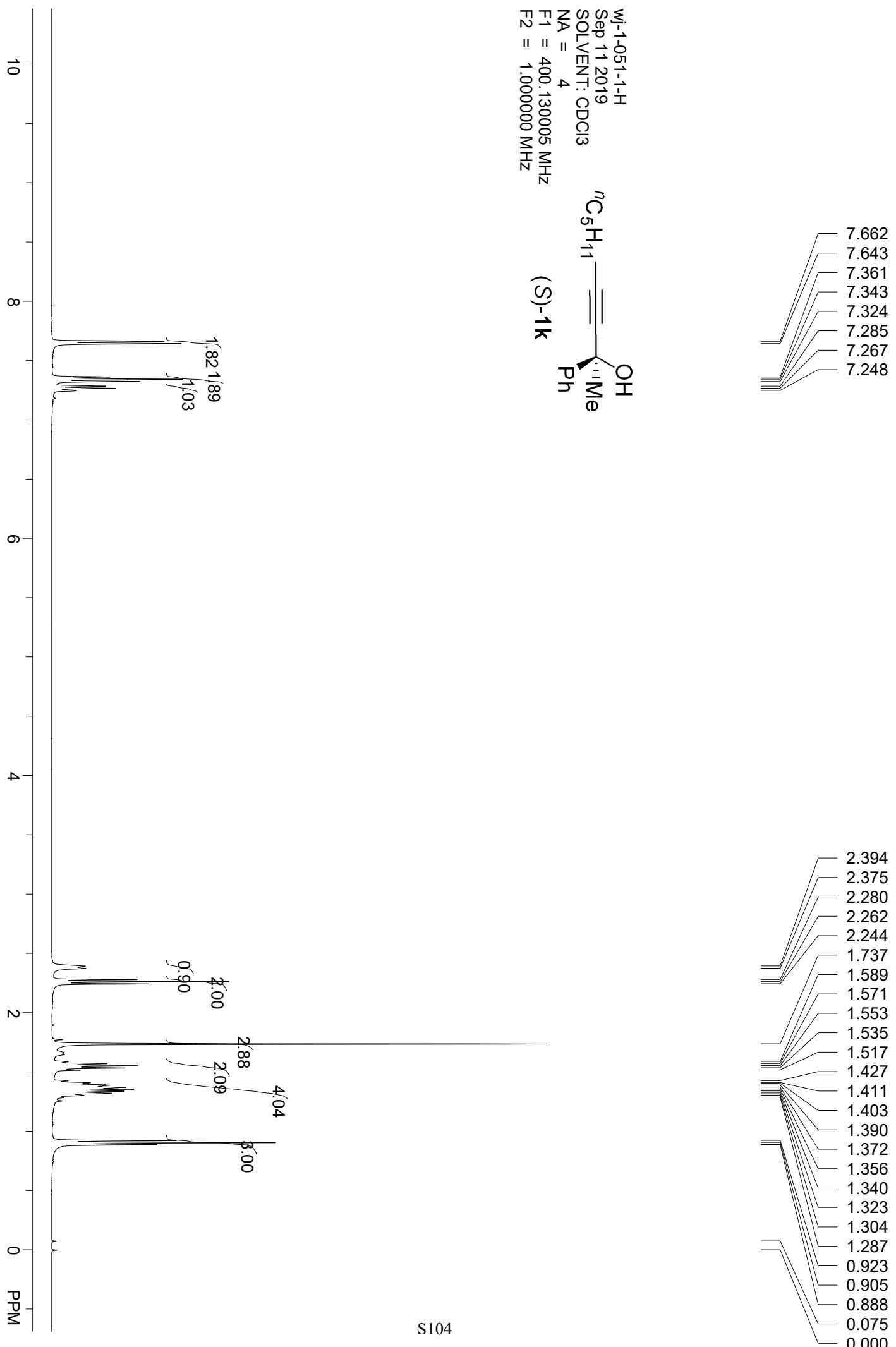
Data file: C:\Users\Public\Documents\ChemStation\1\Data\wgl 2019-09-17 09-02-49\061-P2-C6-wj-1-055-1-rac.D

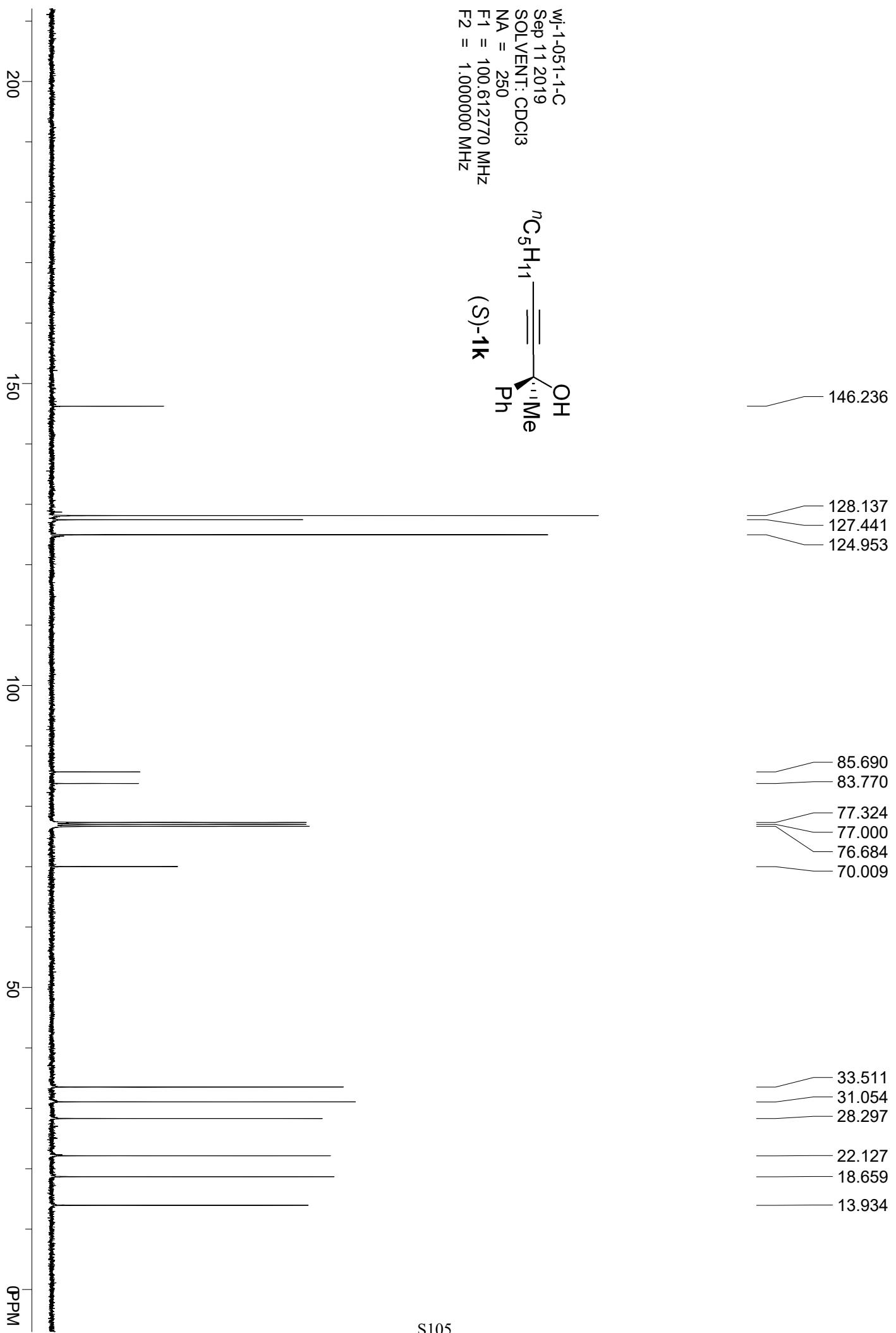
Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
4.217	0.1301	21.6970	169.3124	49.7421
4.461	0.1315	21.6804	171.0677	50.2579
Sum		340.3801	100.0000	





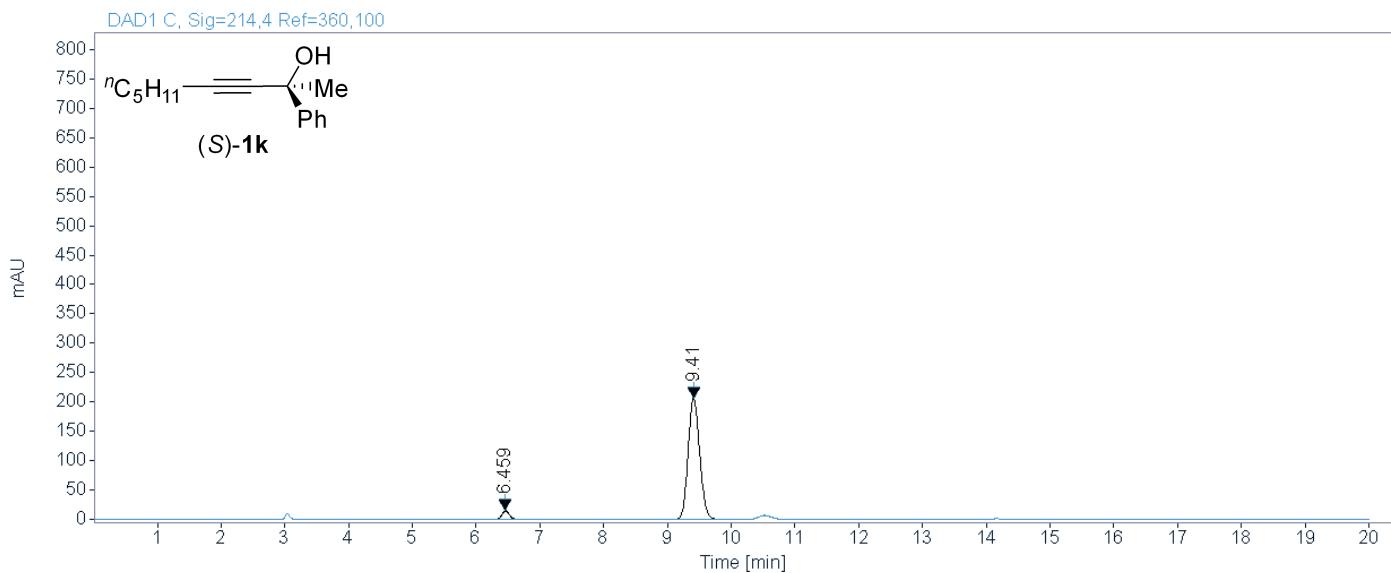
Area Percent Report



sample wj-1-051-1-AS-H-98-2-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\WJ_LC 2019-09-12 22-38-10\003-P2-C1-wj-1-051-1.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
6.459	0.1237	14.7959	118.8316	4.3368
9.410	0.1953	207.6289	2621.2363	95.6632
Sum		2740.0679	100.0000	

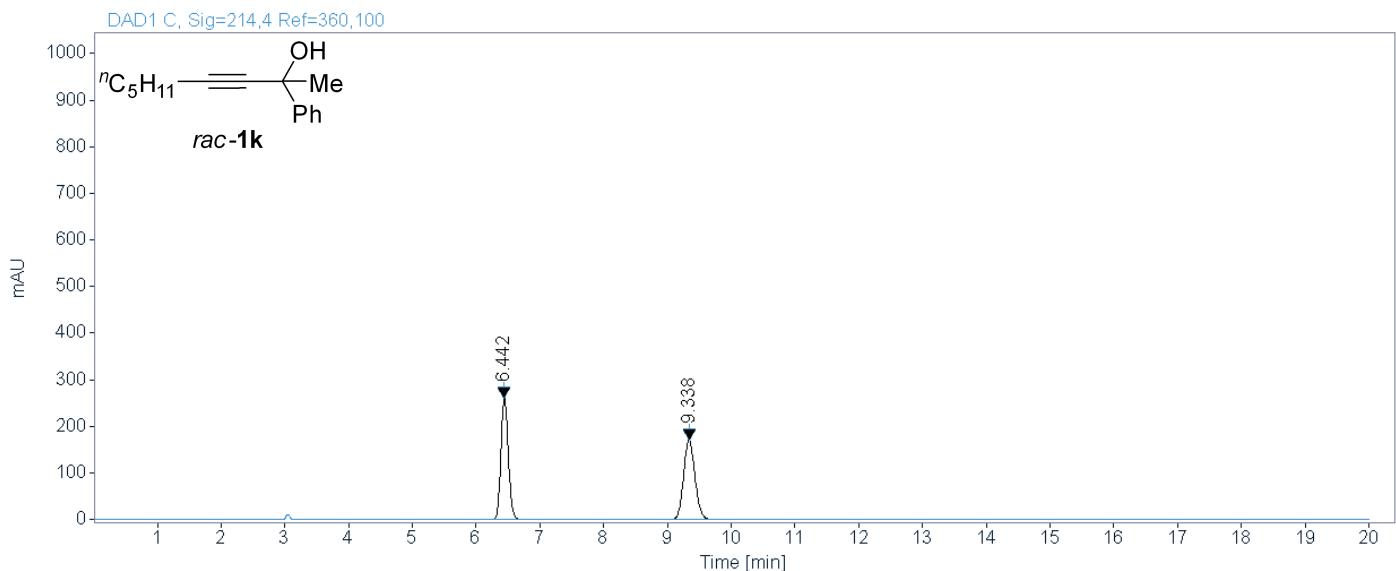
Area Percent Report



sample wj-1-051-1-rac-AS-H-98-2-1.0-214

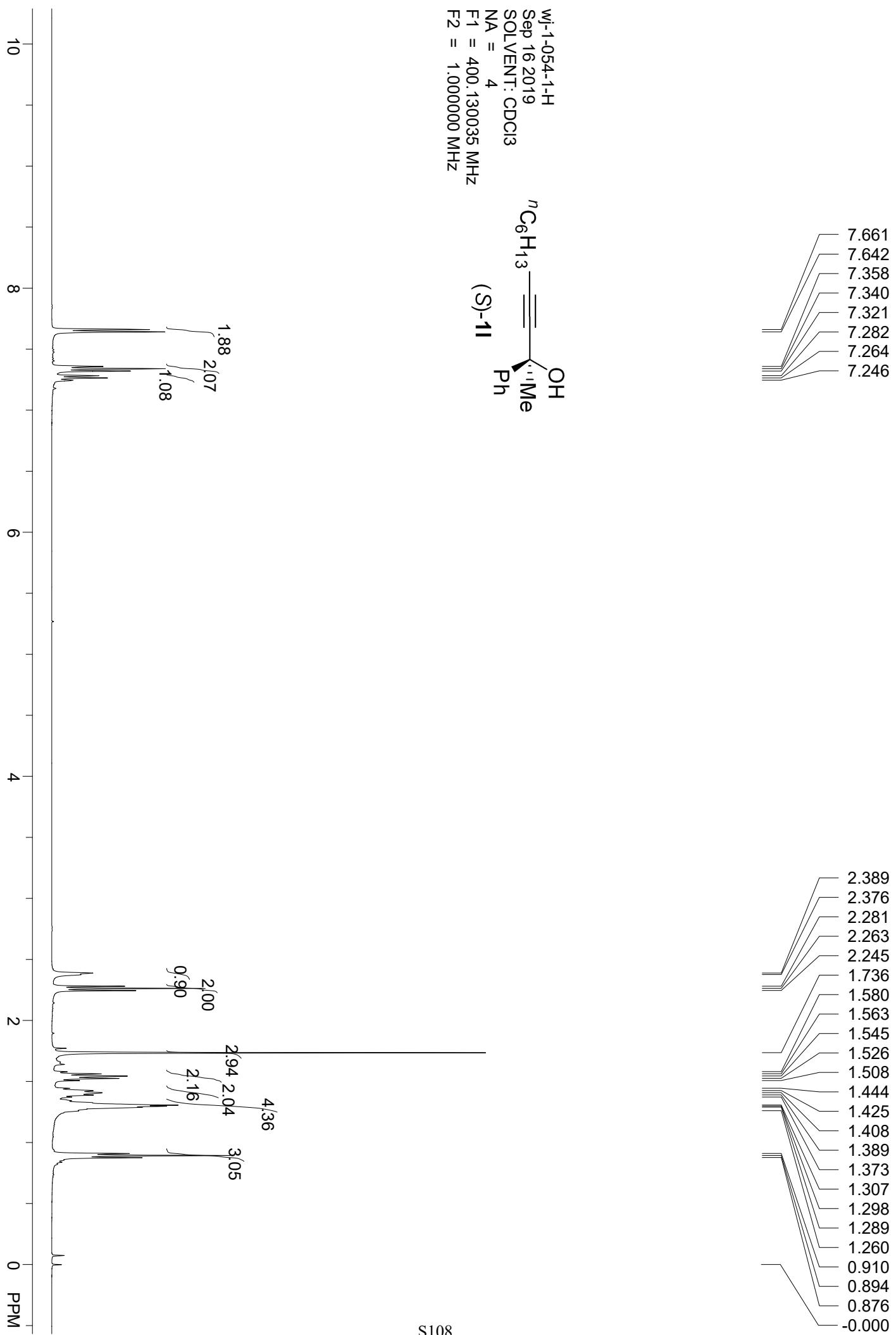
Data file: C:\Users\Public\Documents\ChemStation\1\Data\WJ_LC 2019-09-12 22-38-10\004-P2-C2-wj-1-051-1-rac.D

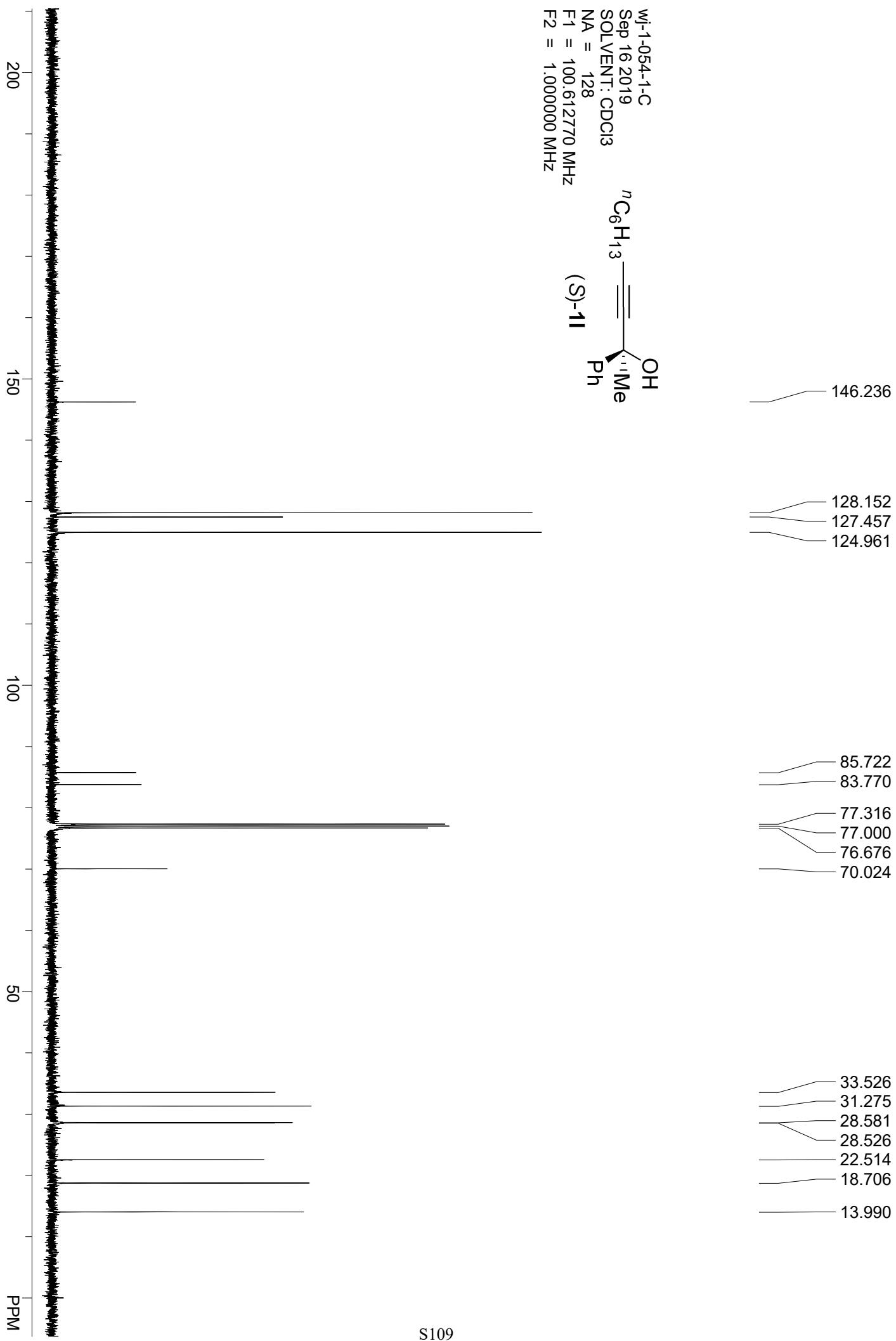
Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
6.442	0.1259	261.4917	2104.3892	49.7532
9.338	0.1935	170.4534	2125.2681	50.2468
Sum		4229.6572	100.0000	





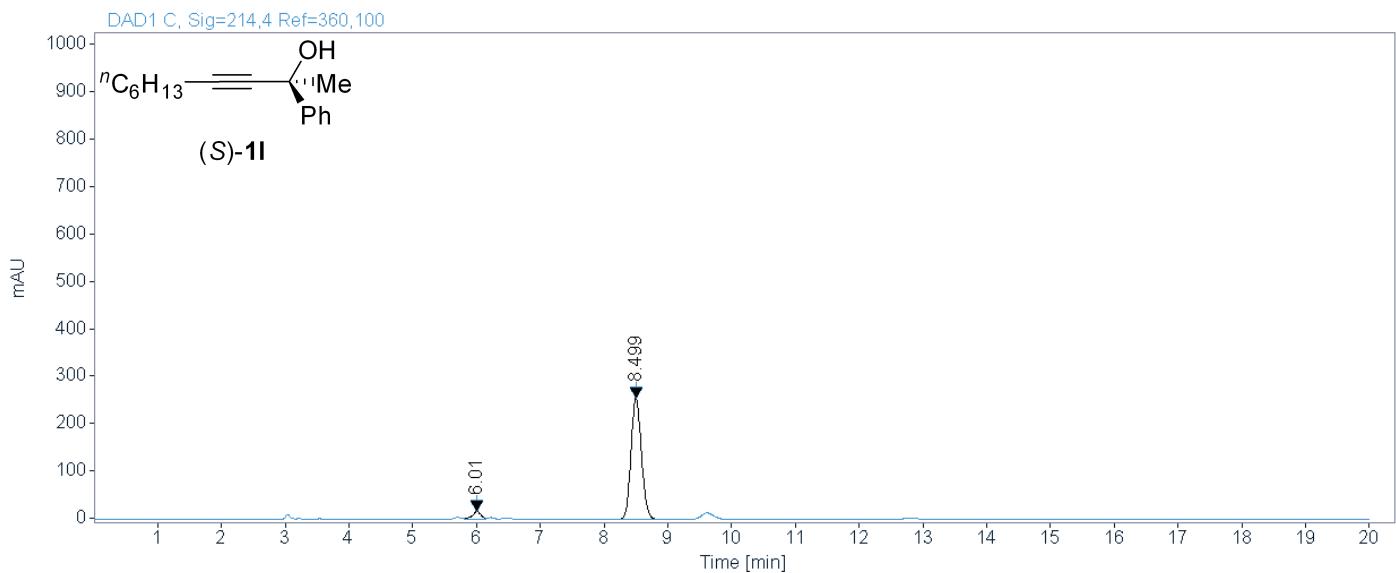
Area Percent Report



sample wj-1-054-1-AS-H-98-2-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\WJ_LC 2019-09-16 20-02-18\003-P2-C1-wj-1-054-1.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
6.010	0.1346	16.3880	149.7057	4.8551
8.499	0.1798	255.7857	2933.7458	95.1449
Sum		3083.4516	100.0000	

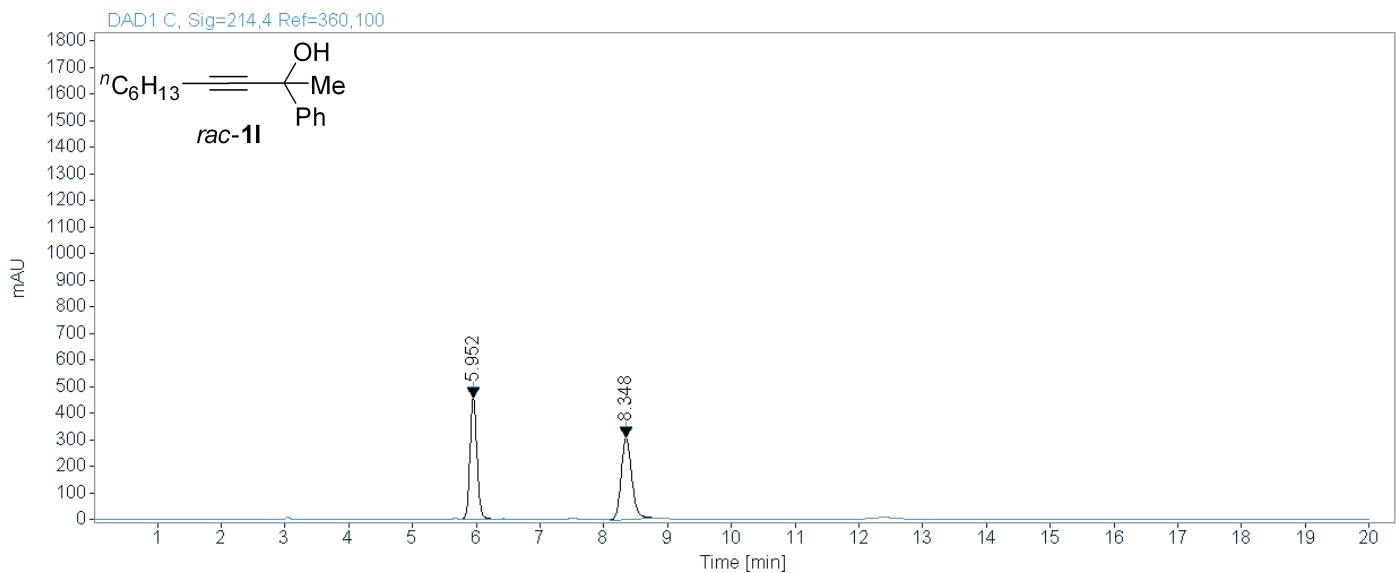
Area Percent Report



sample wj-1-054-1-rac-AS-H-98-2-1.0-214

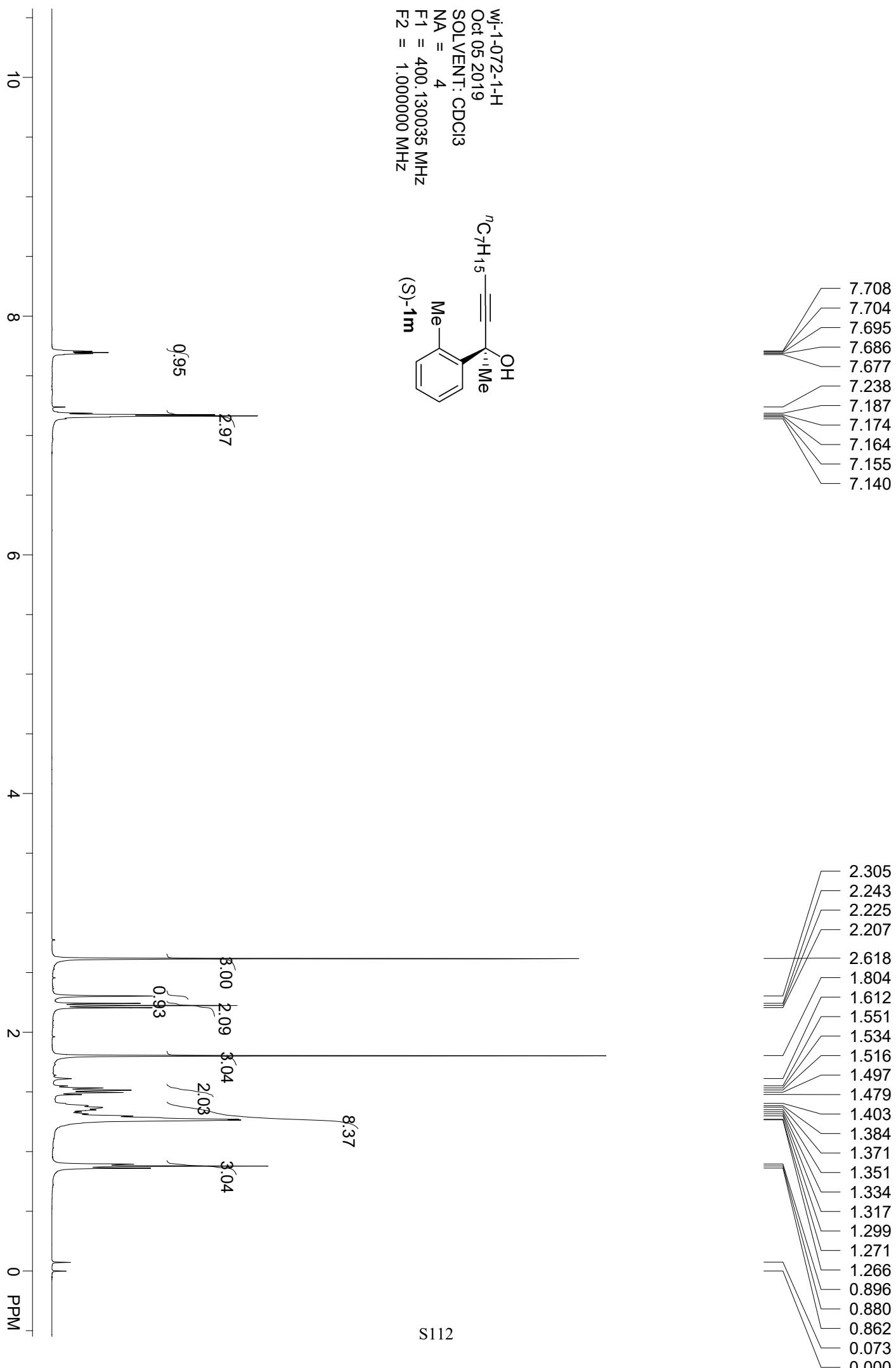
Data file: C:\Users\Public\Documents\ChemStation\1\Data\WJ_LC 2019-09-16 20-02-18\004-P2-C2-wj-1-054-1-rac.D

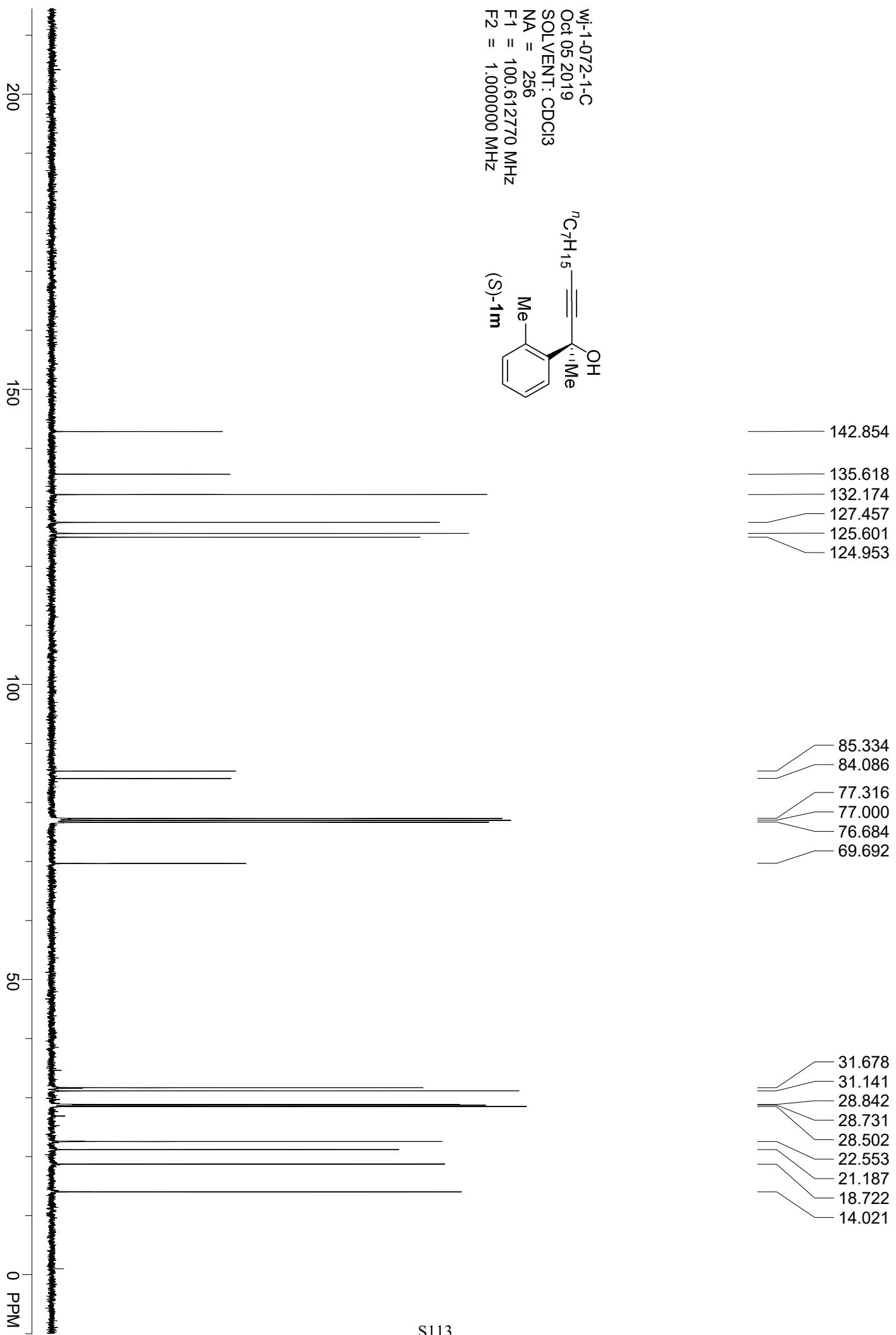
Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
5.952	0.1286	459.4753	3544.4277	49.8856
8.348	0.1940	305.9164	3560.6772	50.1144
		Sum	7105.1050	100.0000





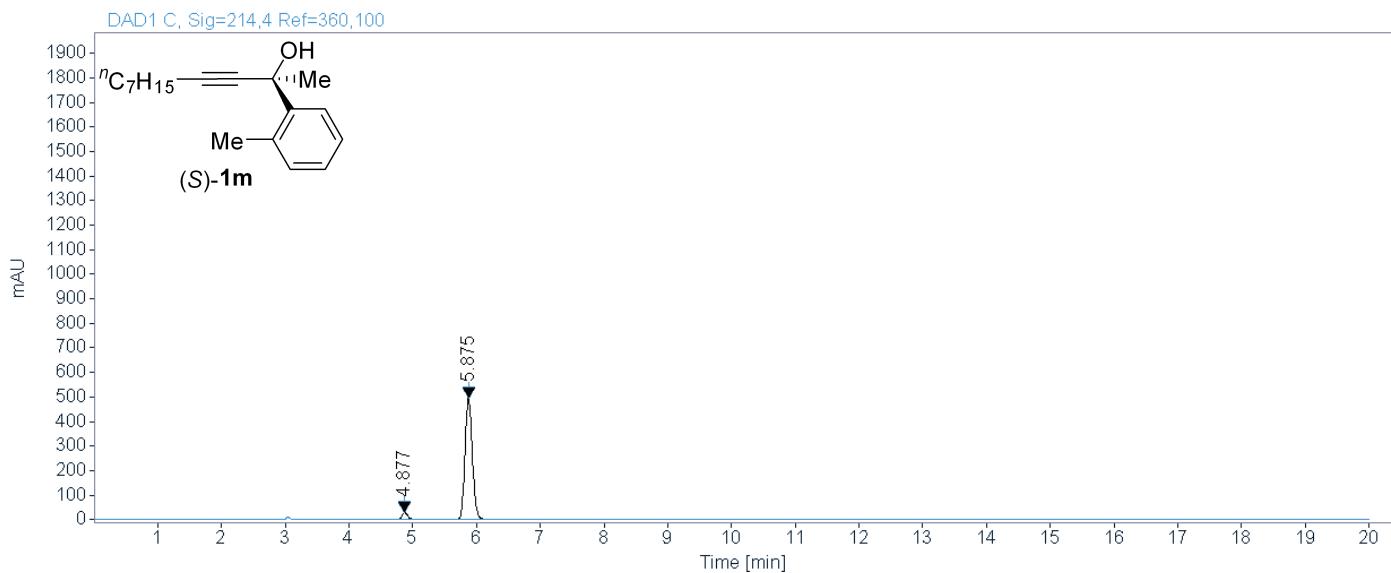
Area Percent Report



sample wj-1-072-1-AS-H-98-2-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\zwf-allenioic acid_LC 2019-10-05 11-37-07\036-P2-C1-wj-1-072-1.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
4.877	0.0977	28.5560	180.2966	4.3083
5.875	0.1241	496.5209	4004.5703	95.6917
Sum		4184.8669	100.0000	

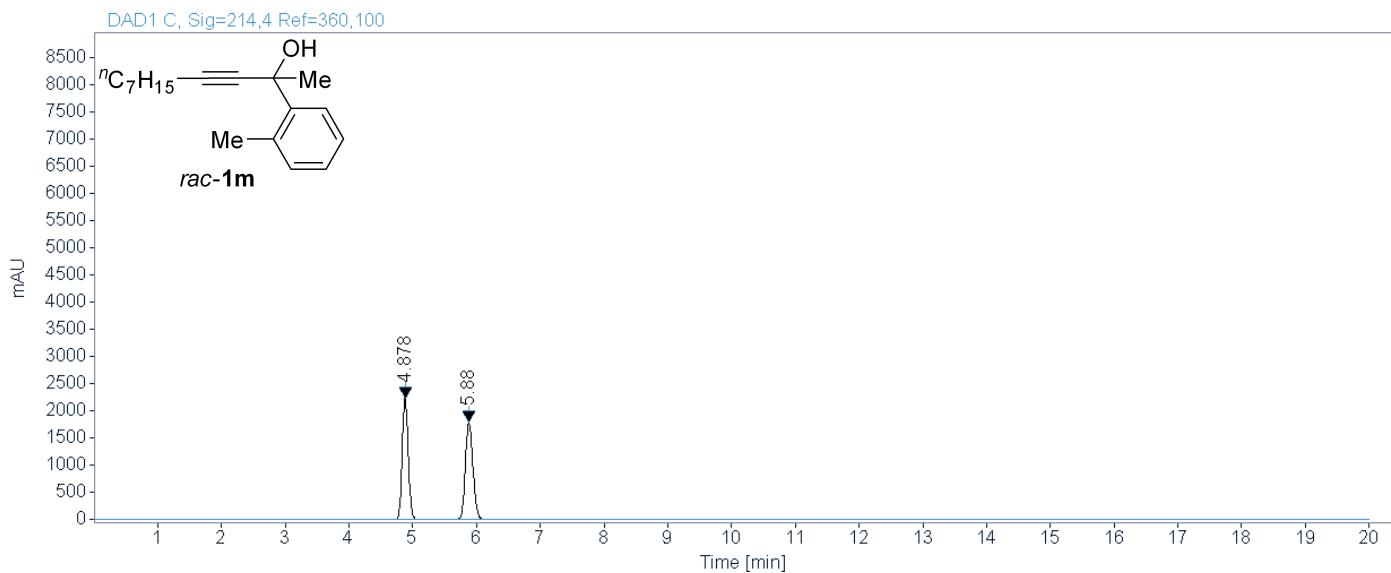
Area Percent Report



sample wj-1-072-1-rac-AS-H-98-2-1.0-214

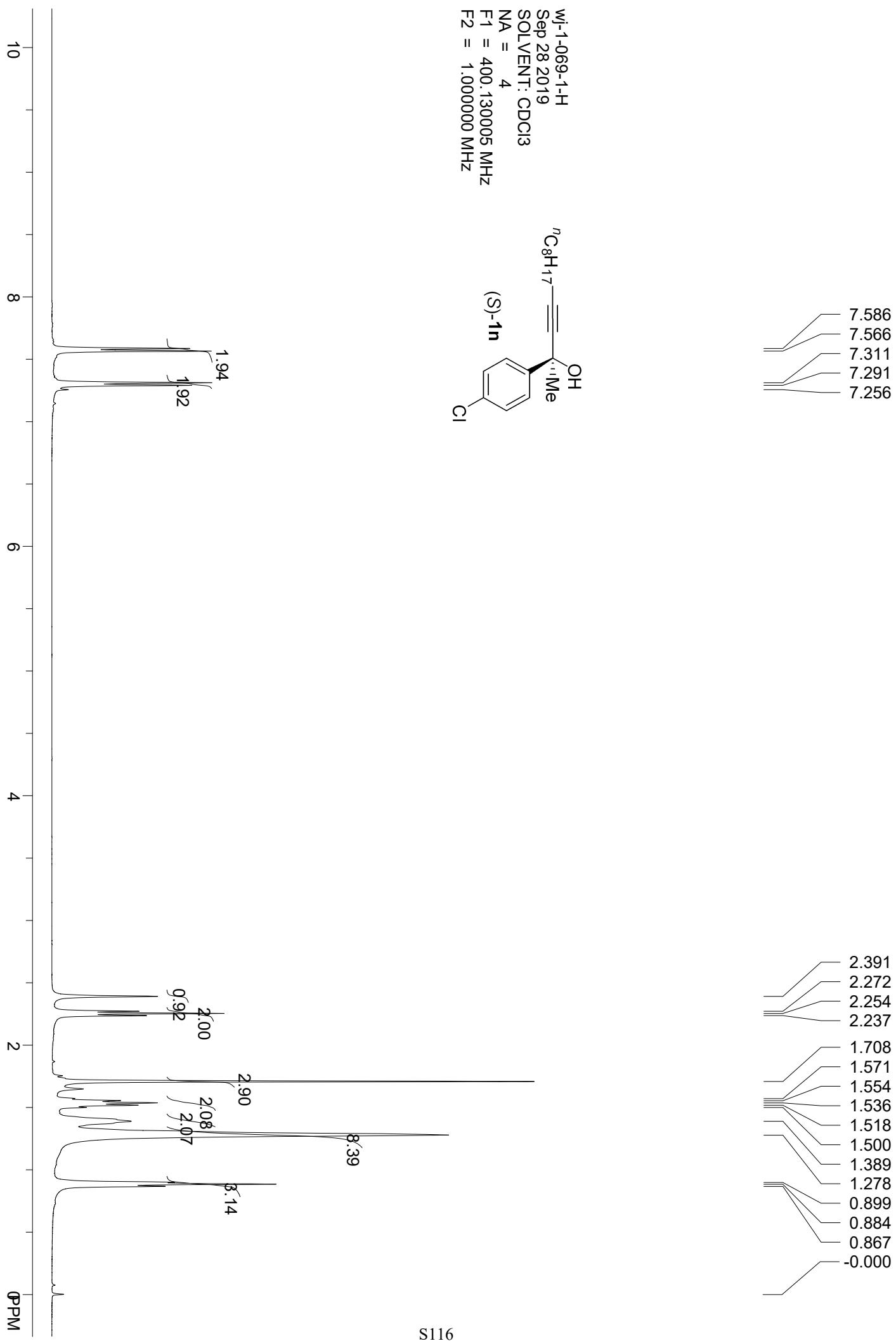
Data file: C:\Users\Public\Documents\ChemStation\1\Data\zwf-alleniac acid_LC 2019-10-05 11-37-07\037-P2-C2-wj-1-072-1-rac.D

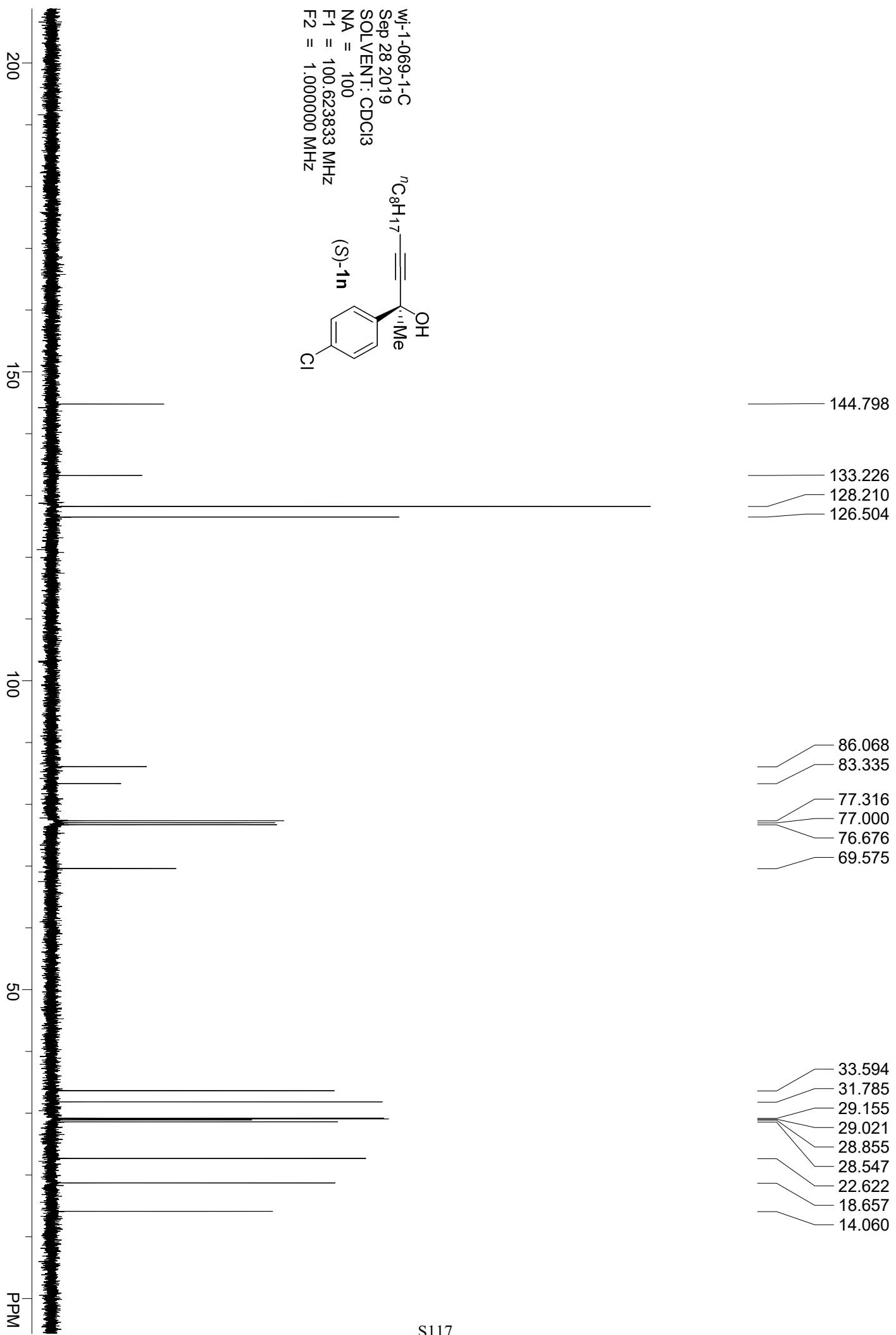
Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
4.878	0.1031	2244.5510	14895.3457	49.6865
5.880	0.1317	1795.0894	15083.3193	50.3135
Sum		29978.6650	100.0000	





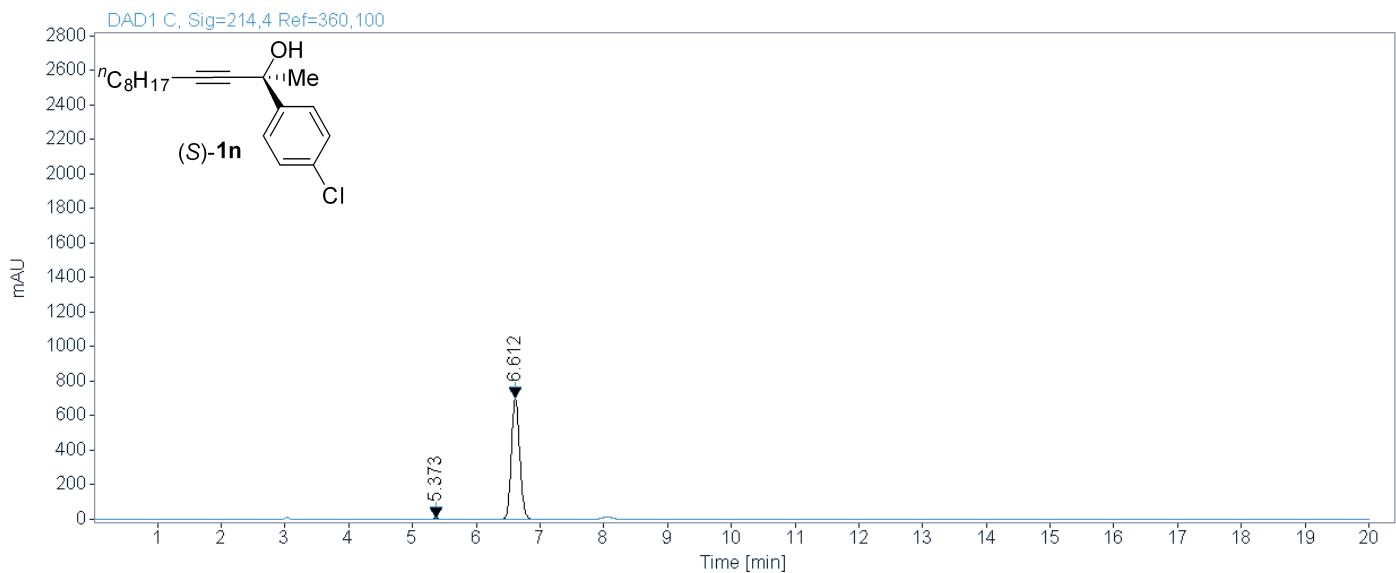
Area Percent Report



sample wj-1-069-1-AS-H-98-2-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\WJ_LC 2019-09-28 15-09-29\003-P2-C1-wj-1-069-1.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
5.373	0.1068	5.2197	36.1348	0.5599
6.612	0.1403	704.2572	6417.6631	99.4401
Sum		6453.7979	100.0000	

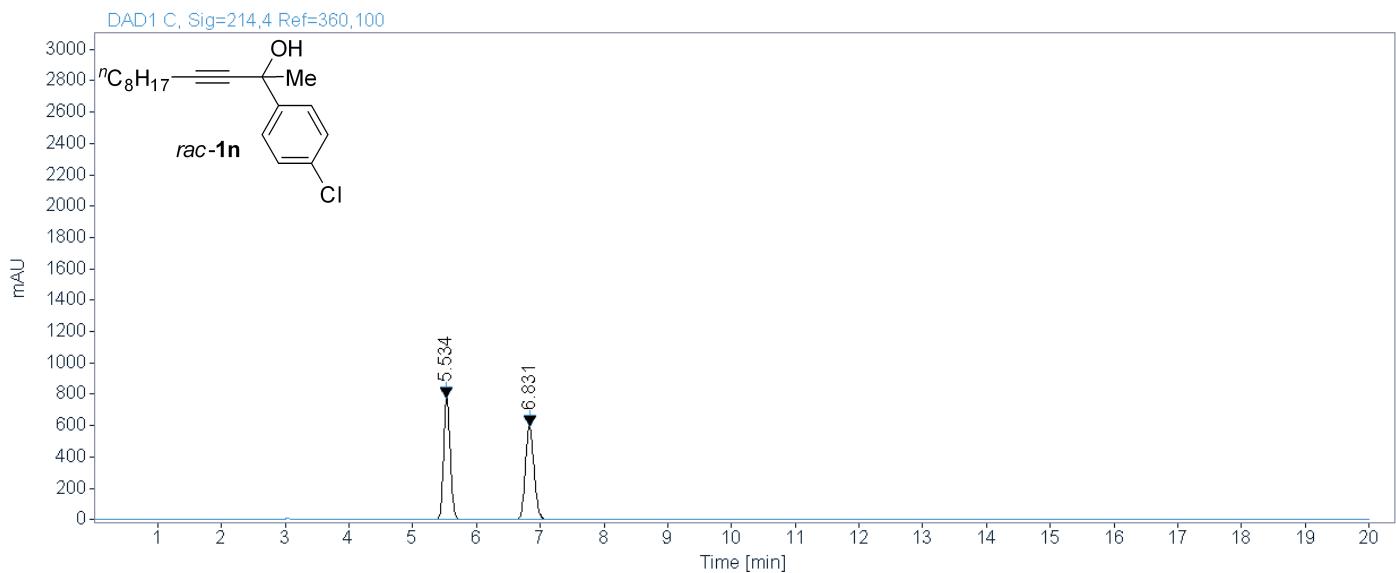
Area Percent Report



sample wj-1-069-1-rac-AS-H-98-2-1.0-214

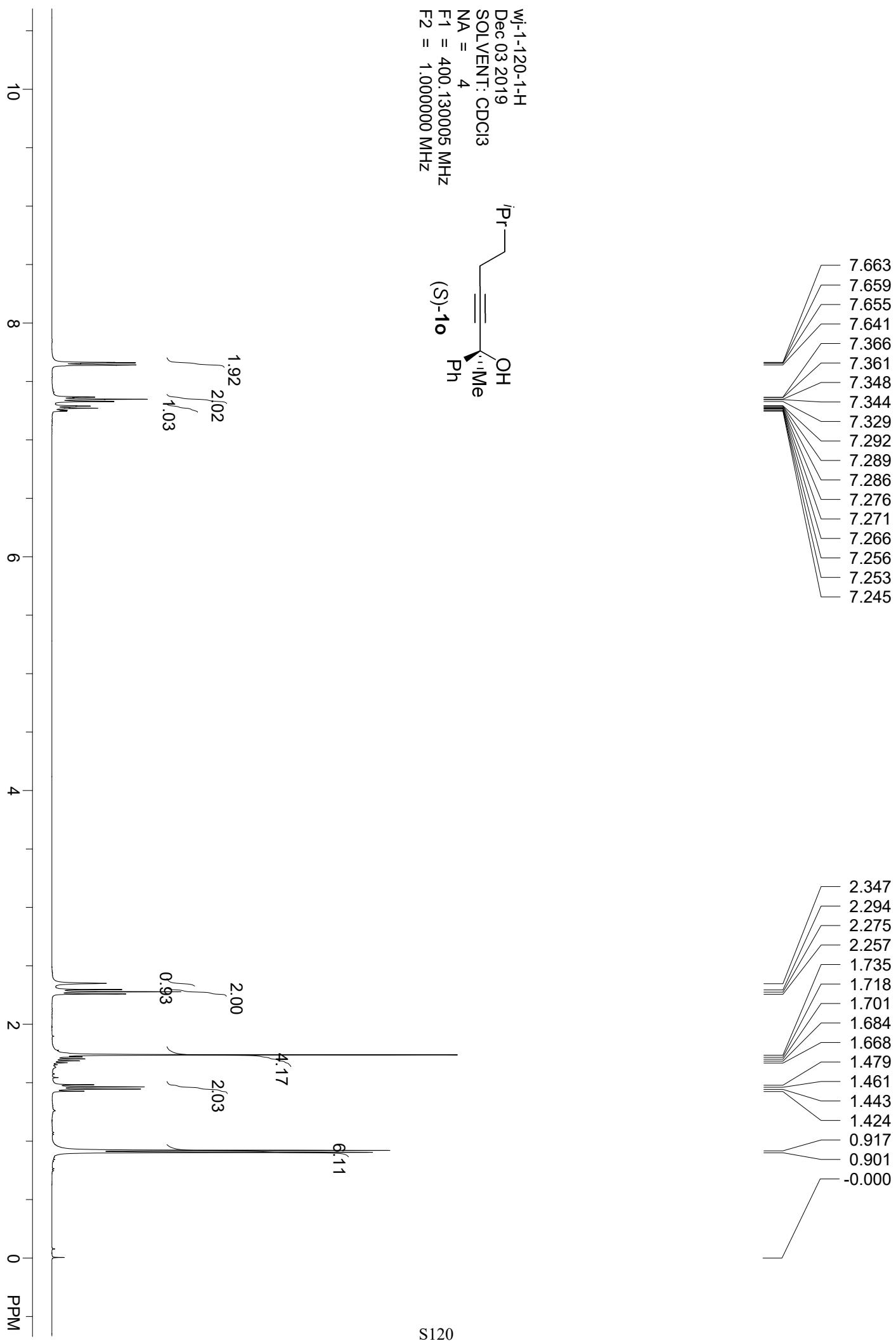
Data file: C:\Users\Public\Documents\ChemStation\1\Data\WJ_LC 2019-09-28 15-09-29\004-P2-C2-wj-1-069-1-rac.D

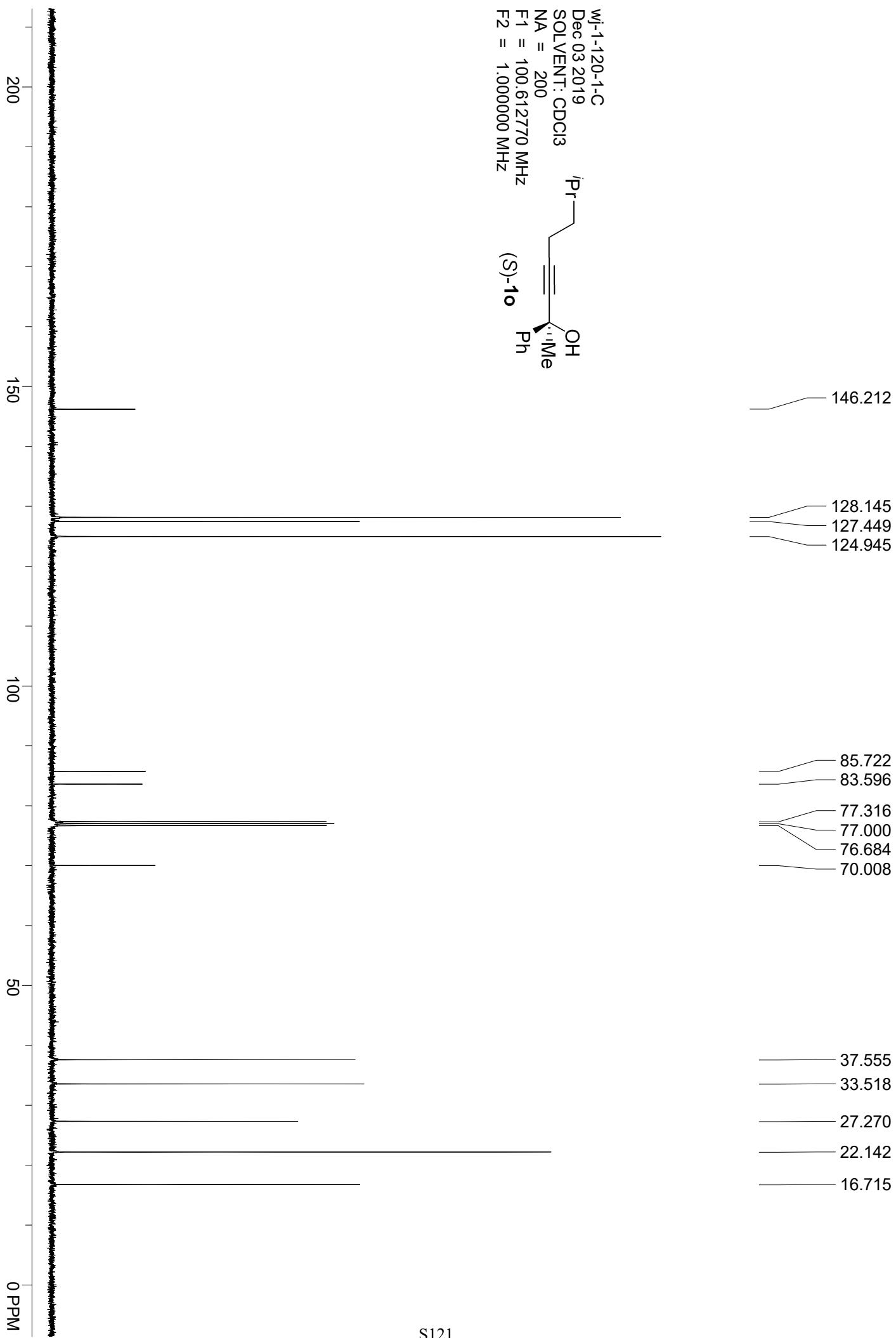
Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
5.534	0.1128	777.6021	5654.7524	49.9115
6.831	0.1470	596.2778	5674.8145	50.0885
Sum		11329.5669	100.0000	





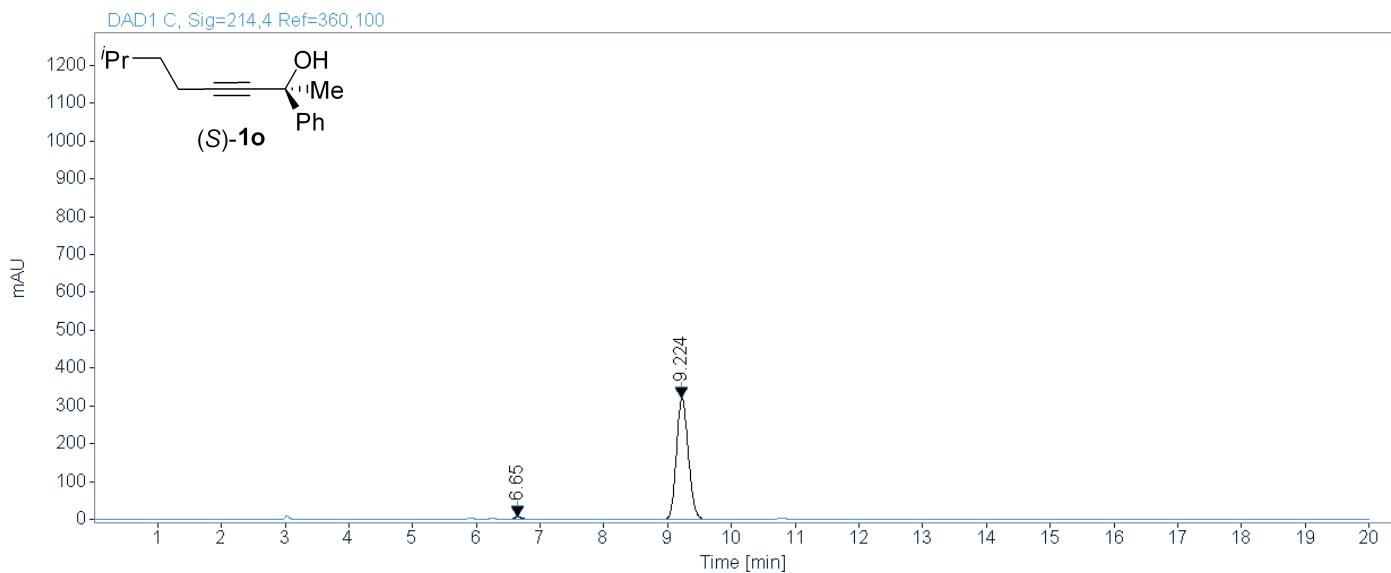
Area Percent Report



sample wj-1-120-1-AS-H-98-2-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\zwf-alleniac acid_LC 2019-12-03 19-19-43\002-P2-C1-wj-1-120-1.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
6.650	0.1378	6.9647	61.9364	1.4505
9.224	0.2045	321.9935	4208.0449	98.5495
Sum		4269.9813	100.0000	

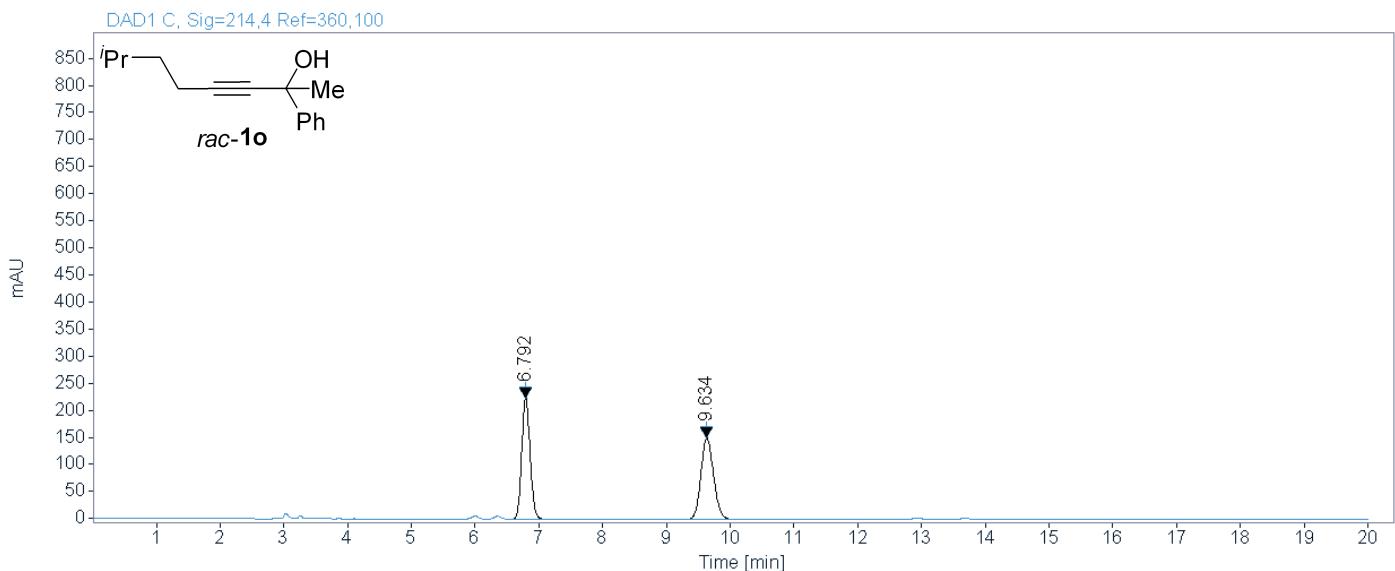
Area Percent Report



sample wj-1-120-1-rac-AS-H-98-2-1.0-214

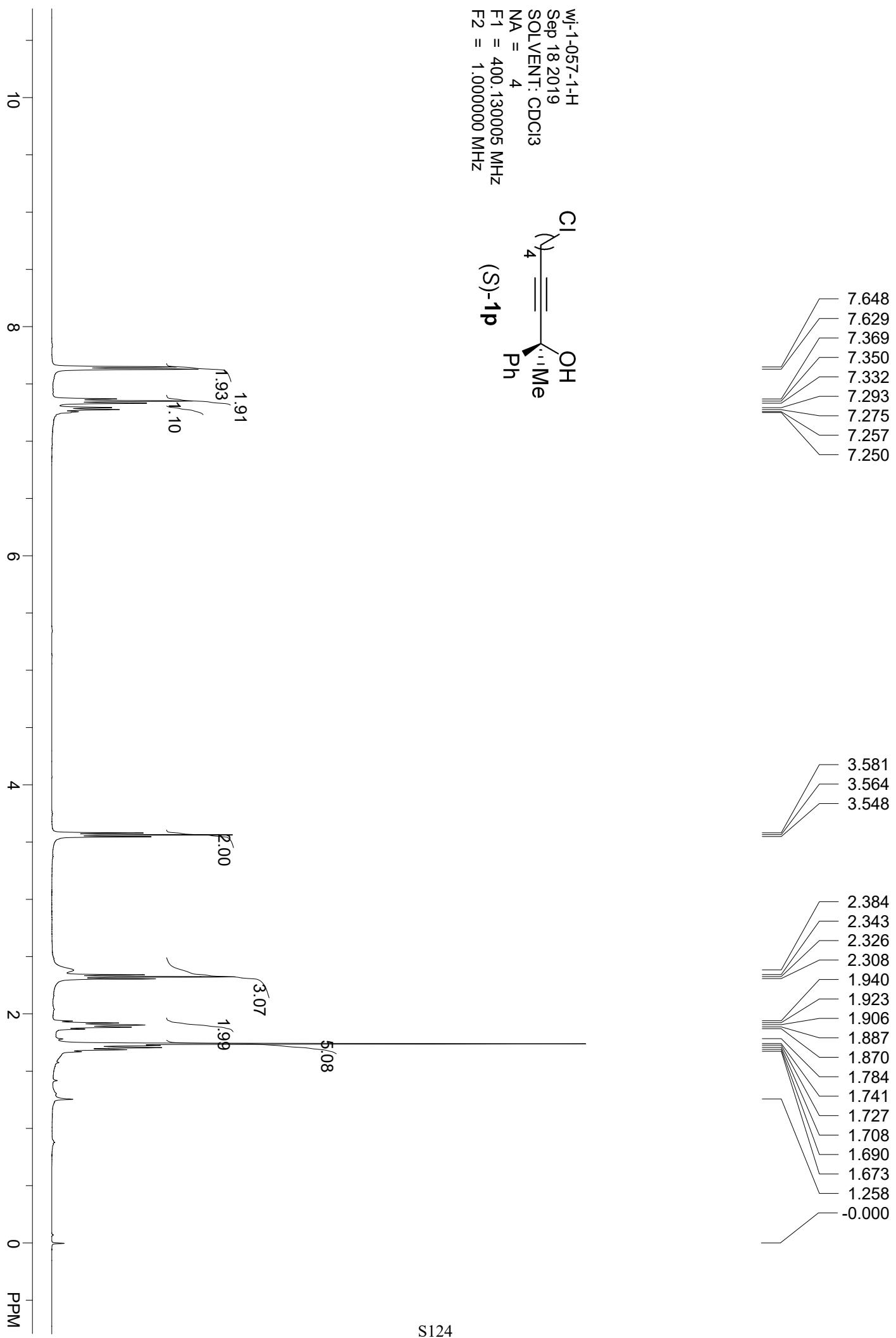
Data file: C:\Users\Public\Documents\ChemStation\1\Data\zwf-alleniac acid_LC 2019-12-03 19-19-43\003-P2-C2-wj-1-120-1-rac.D

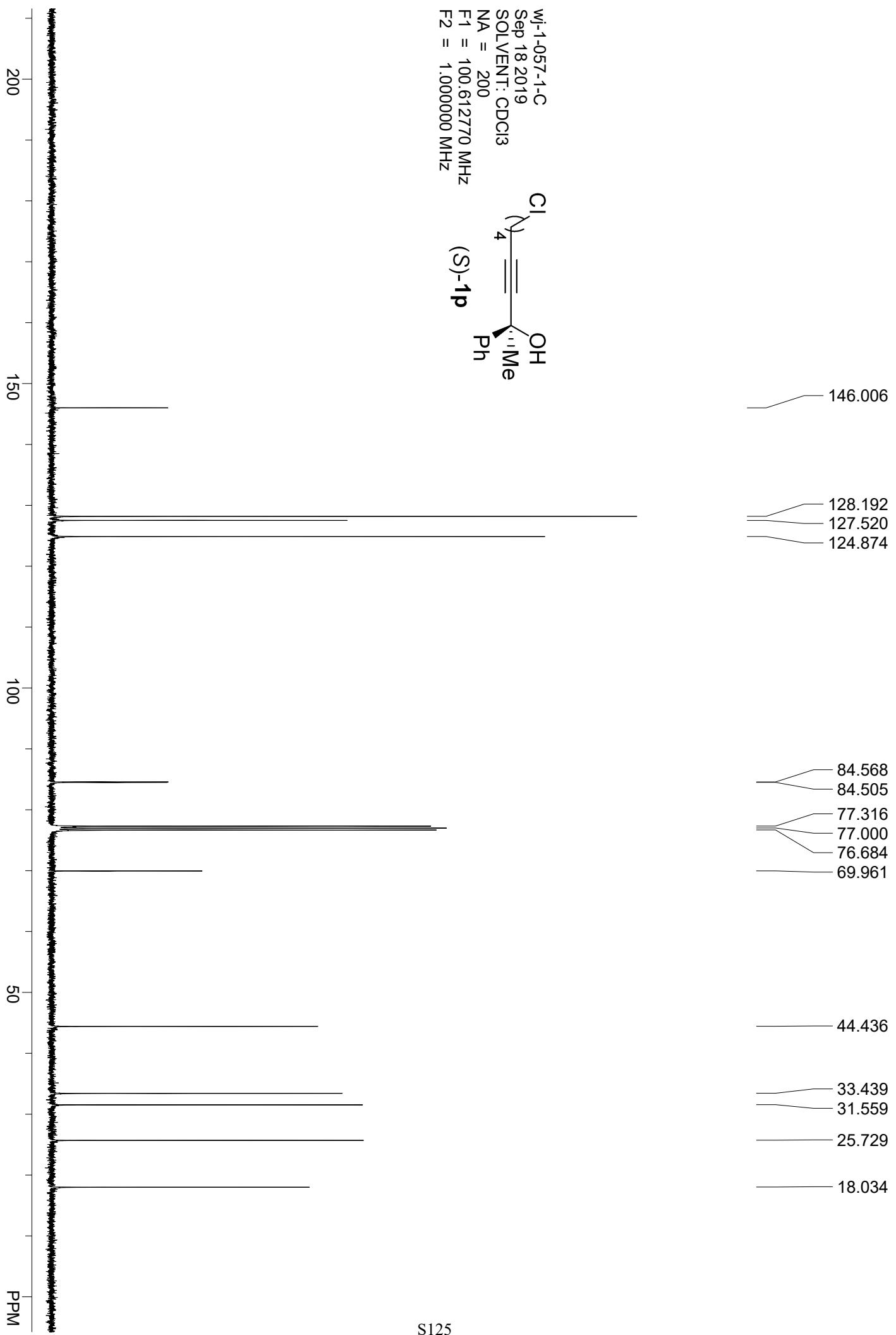
Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
6.792	0.1431	224.1575	2058.3005	49.8814
9.634	0.2146	150.2552	2068.0857	50.1186
Sum		4126.3862	100.0000	





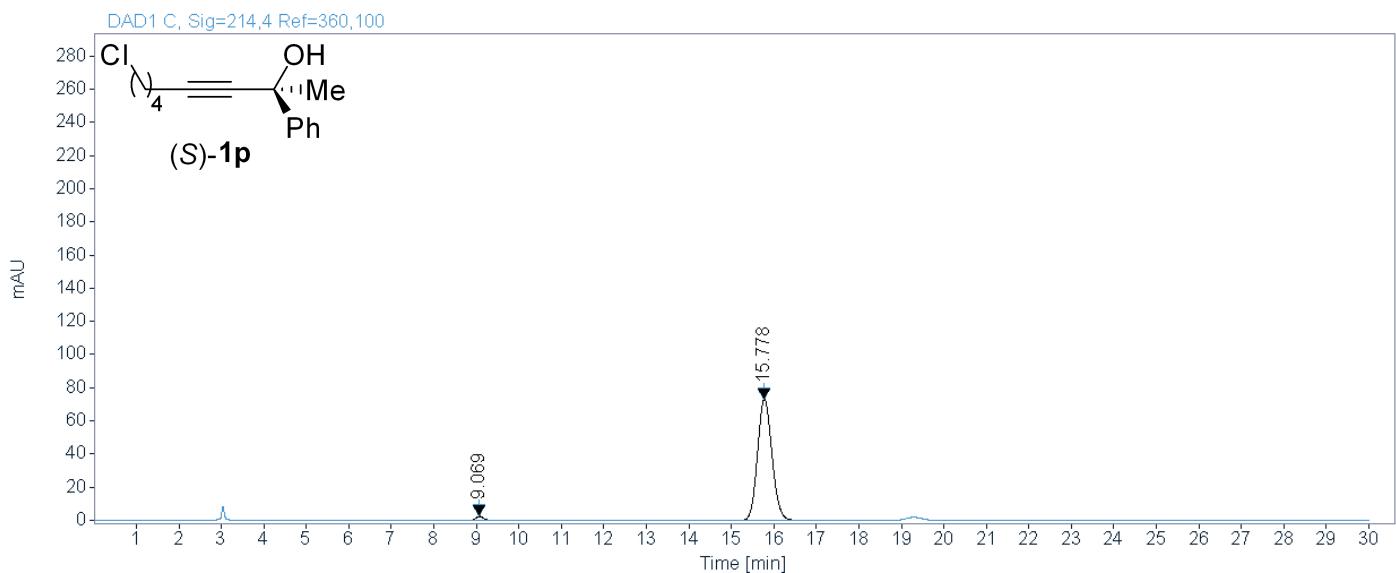
Area Percent Report



sample wj-1-057-1-AS-H-95-5-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\wgl 2019-09-18 08-46-24\053-P2-C1-wj-1-057-1.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
9.069	0.1916	2.4201	29.7949	1.6814
15.778	0.3692	73.3359	1742.2300	98.3186
		Sum	1772.0249	100.0000

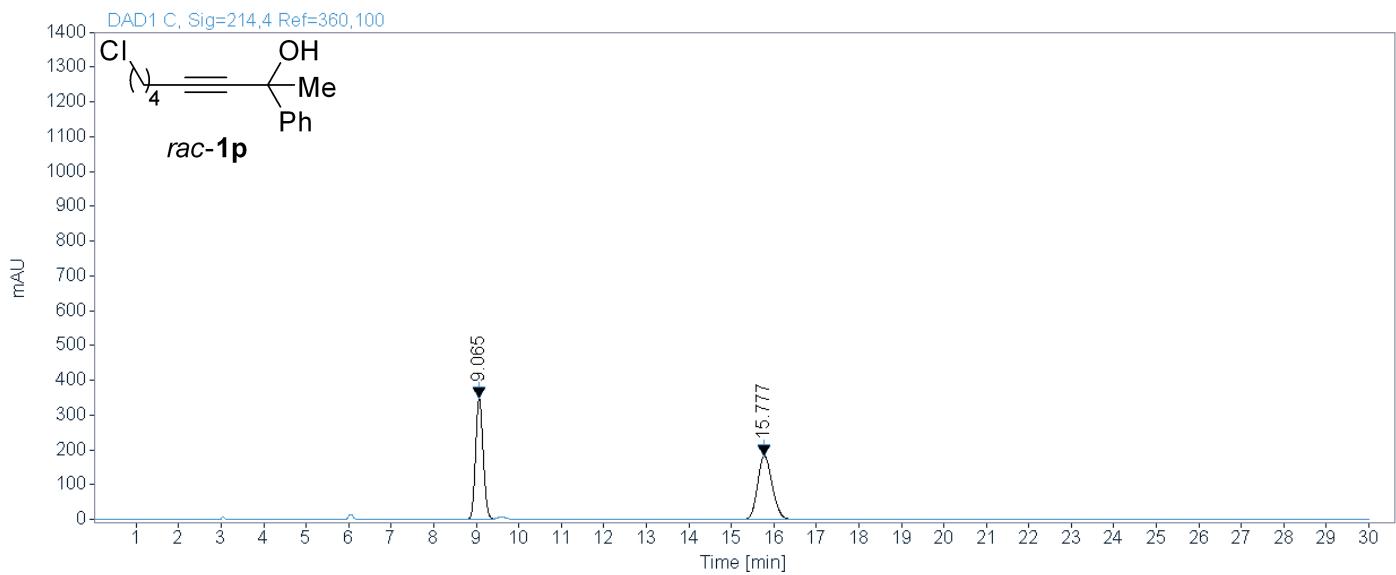
Area Percent Report



sample wj-1-057-1-rac-AS-H-95-5-1.0-214

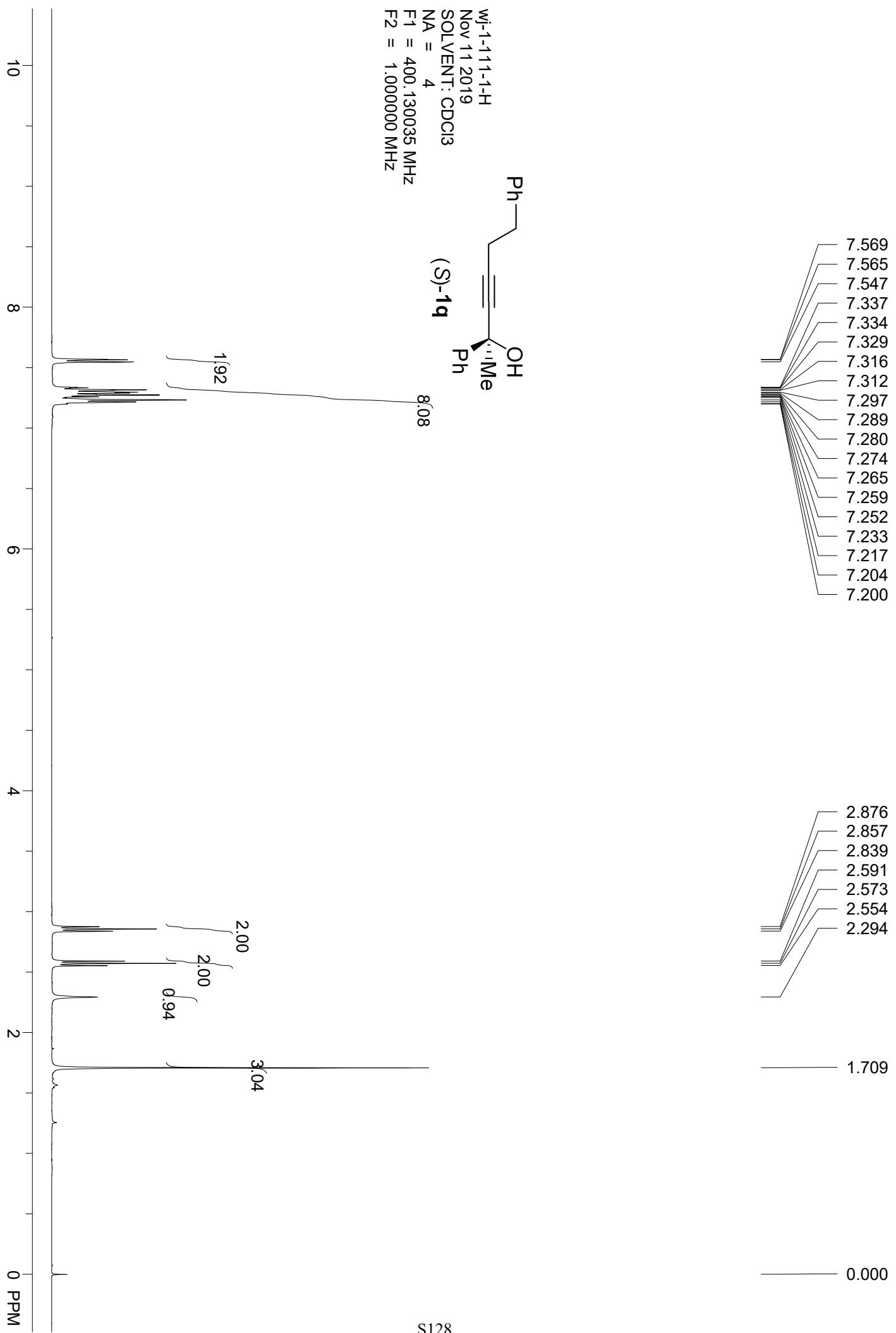
Data file: C:\Users\Public\Documents\ChemStation\1\Data\wgl 2019-09-18 08-46-24\054-P2-C2-wj-1-057-1-rac.D

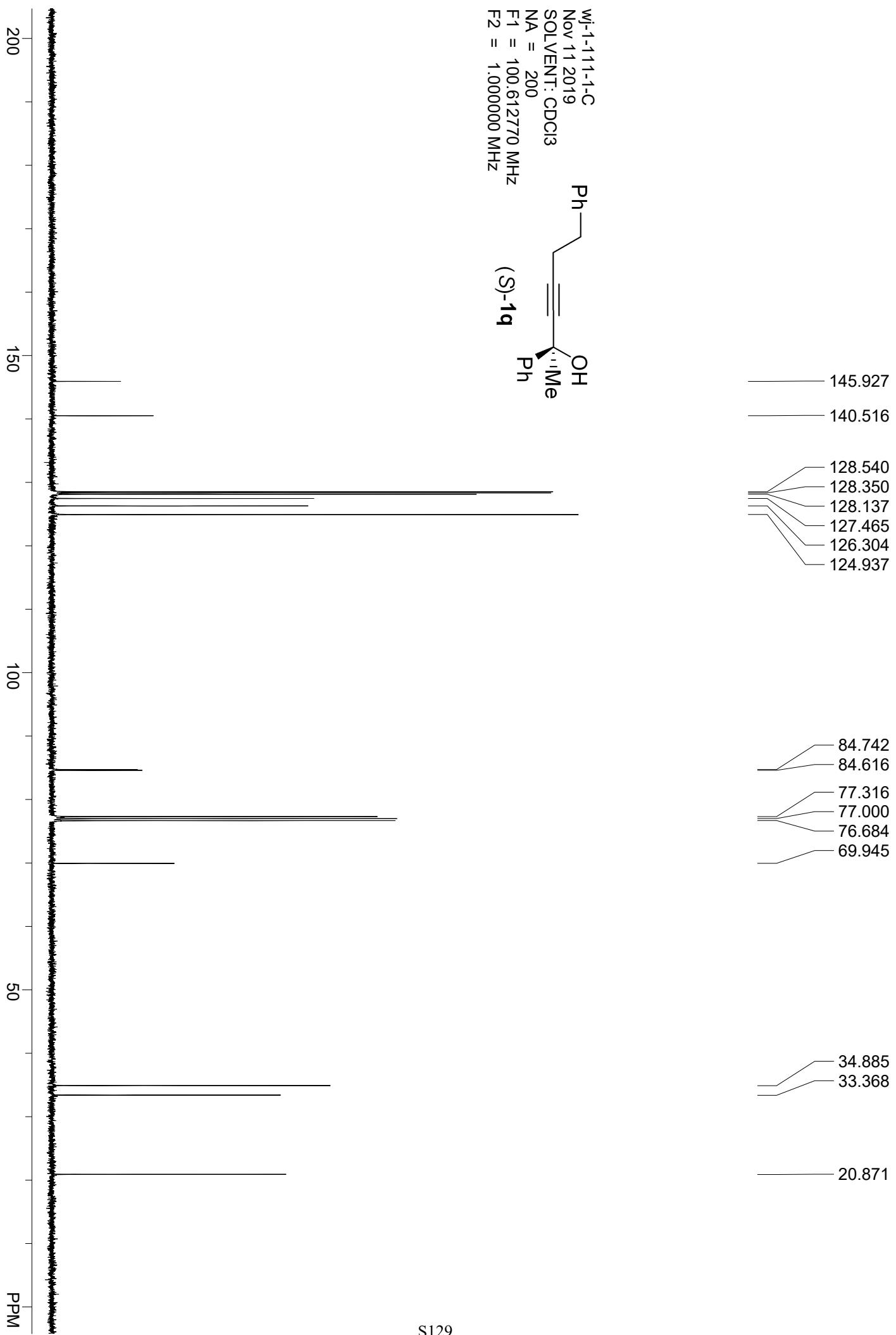
Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
9.065	0.2079	349.9830	4366.1230	49.8629
15.777	0.3709	183.6236	4390.1318	50.1371
Sum		8756.2549	100.0000	





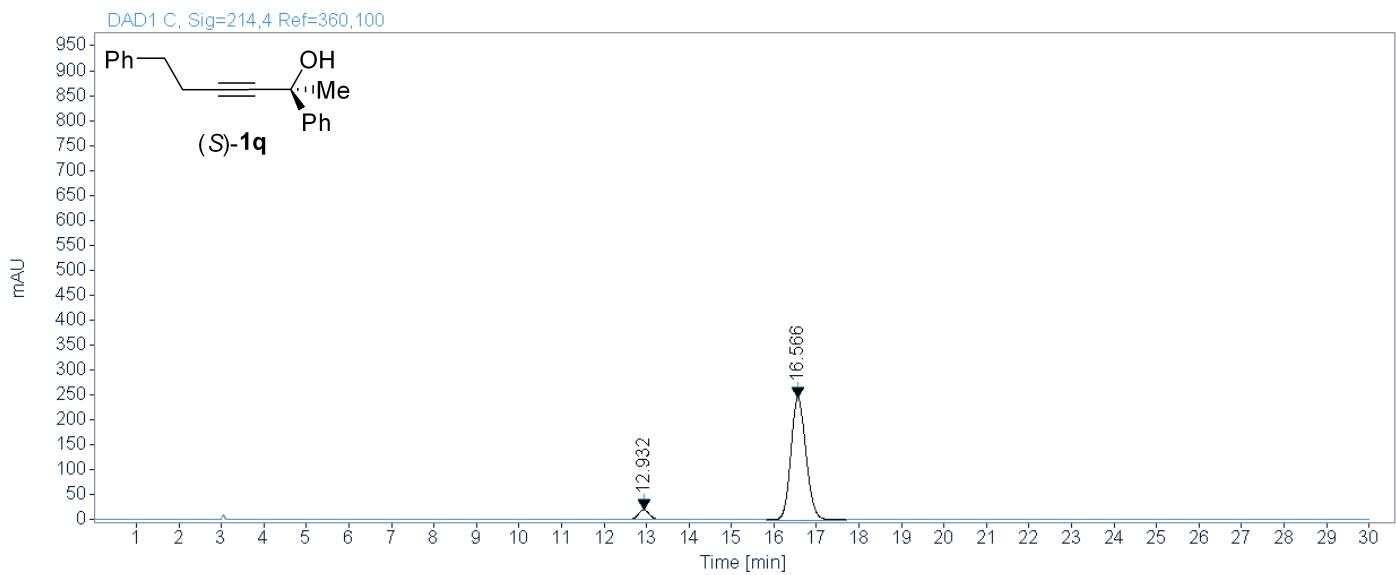
Area Percent Report



sample wj-1-111-1-AS-H-98-2-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\YuanYuan 2019-11-11 18-50-01\017-P2-C1-wj-1-111-1.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
12.932	0.3035	19.3830	353.0218	5.5040
16.566	0.4122	245.0556	6060.9170	94.4960
		Sum	6413.9388	100.0000

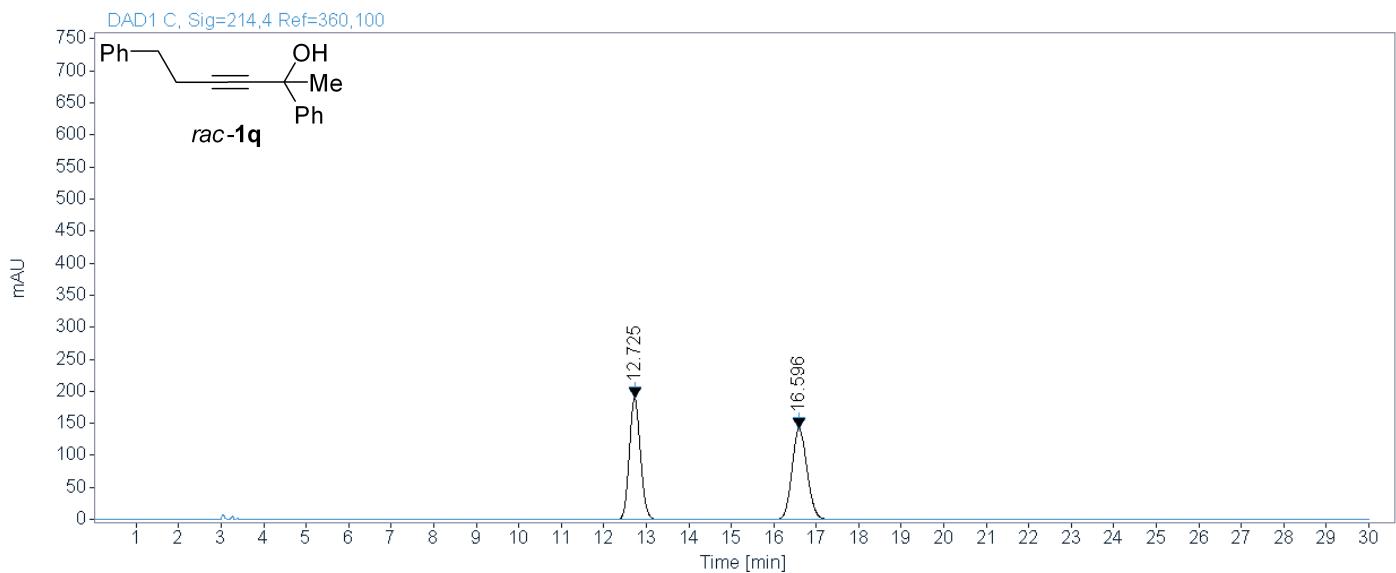
Area Percent Report



sample wj-1-111-1-rac-AS-H-98-2-1.0-214

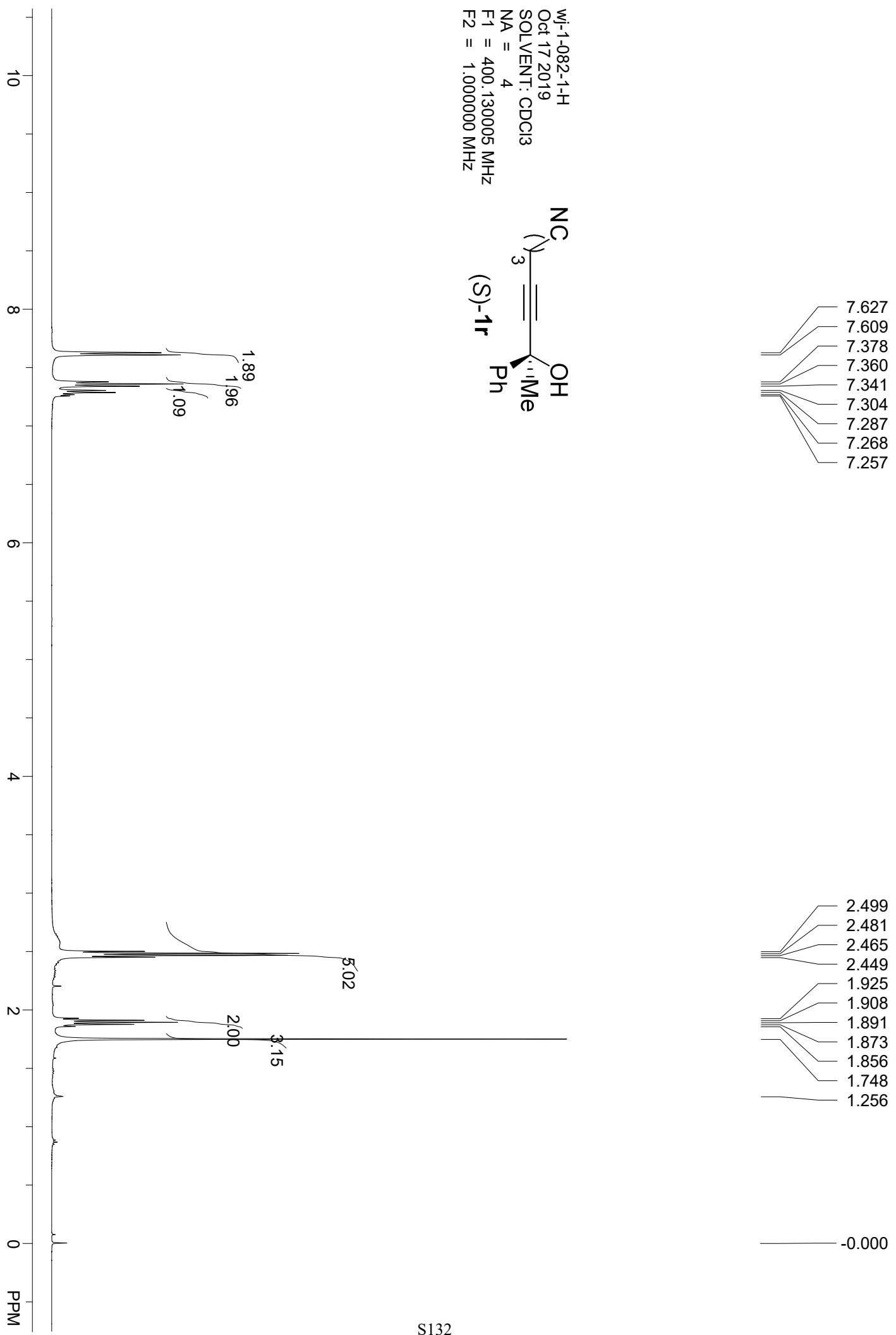
Data file: C:\Users\Public\Documents\ChemStation\1\Data\YuanYuan 2019-11-11 18-50-01\018-P2-C2-wj-1-111-1-rac.D

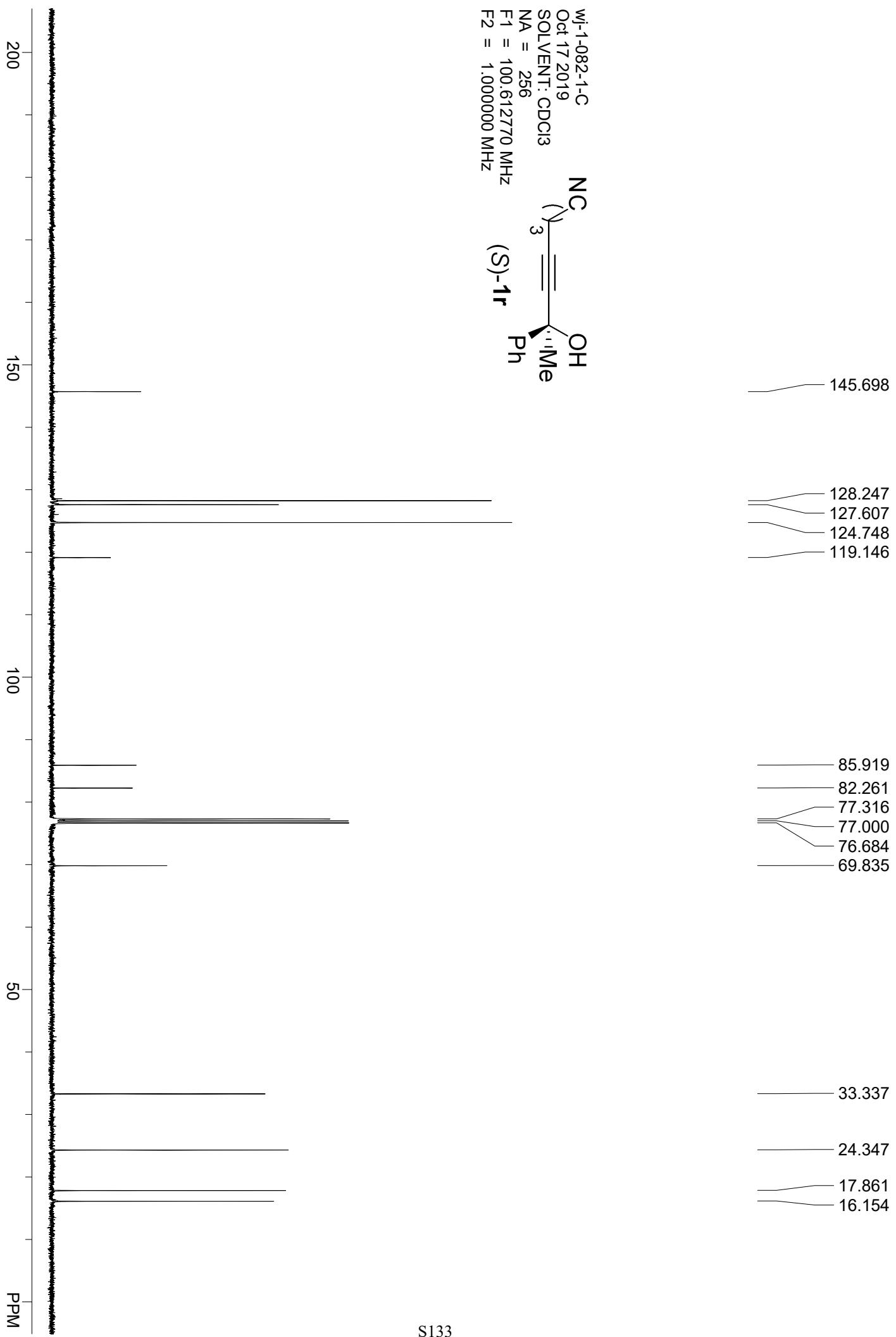
Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
12.725	0.2820	189.8749	3450.9958	49.9081
16.596	0.3812	141.6962	3463.6985	50.0919
		Sum	6914.6943	100.0000





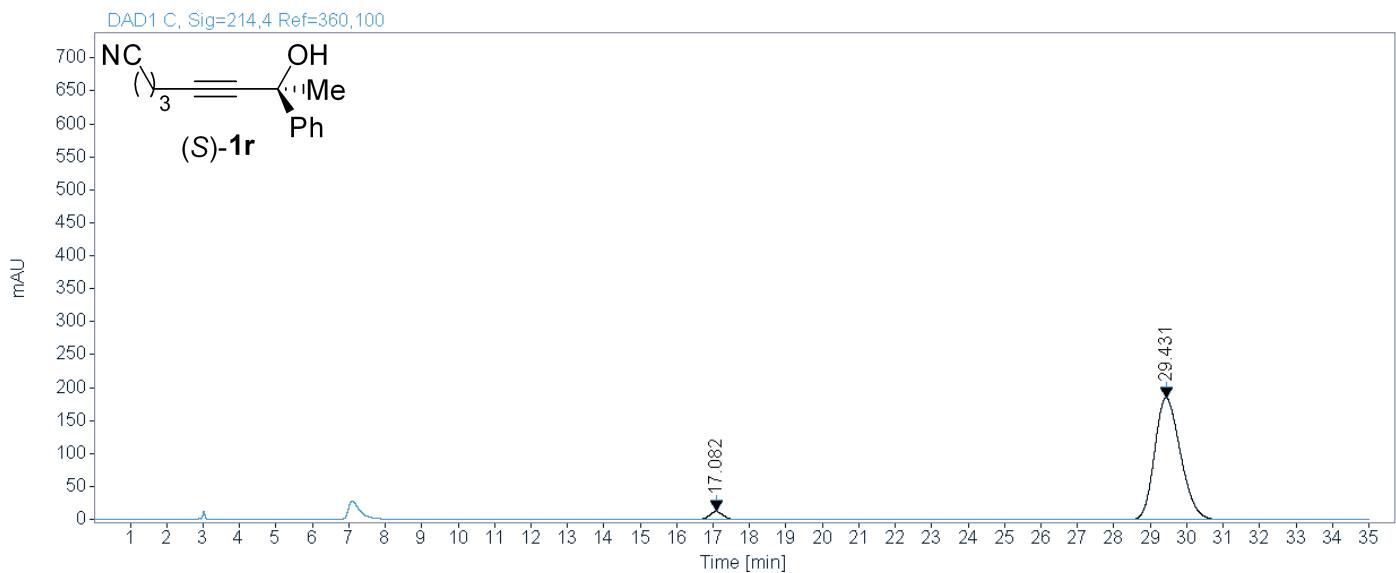
Area Percent Report



sample wj-1-082-1-AS-H-90-10-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\WJ_LC 2019-10-17 17-55-02\004-P2-C1-wj-1-082-1.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
17.082	0.4154	11.7621	293.1832	3.1193
29.431	0.7669	184.7821	9105.9668	96.8807
Sum		184.7821	9399.1500	100.0000

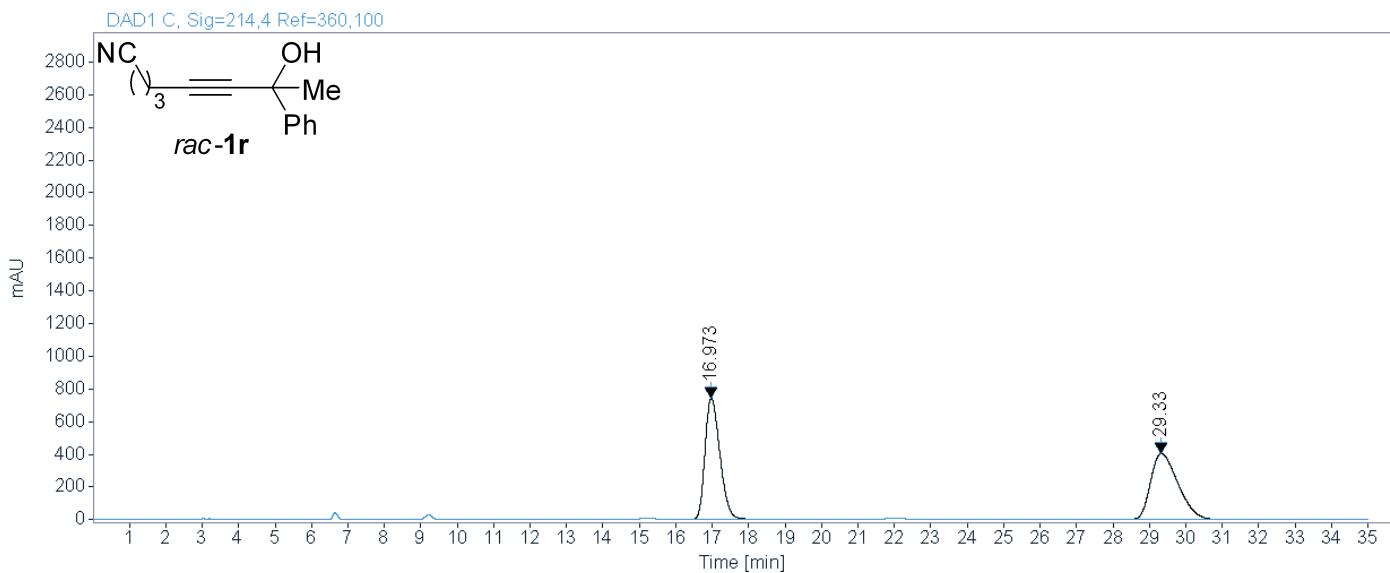
Area Percent Report



sample wj-1-082-1-rac-AS-H-90-10-1.0-
214

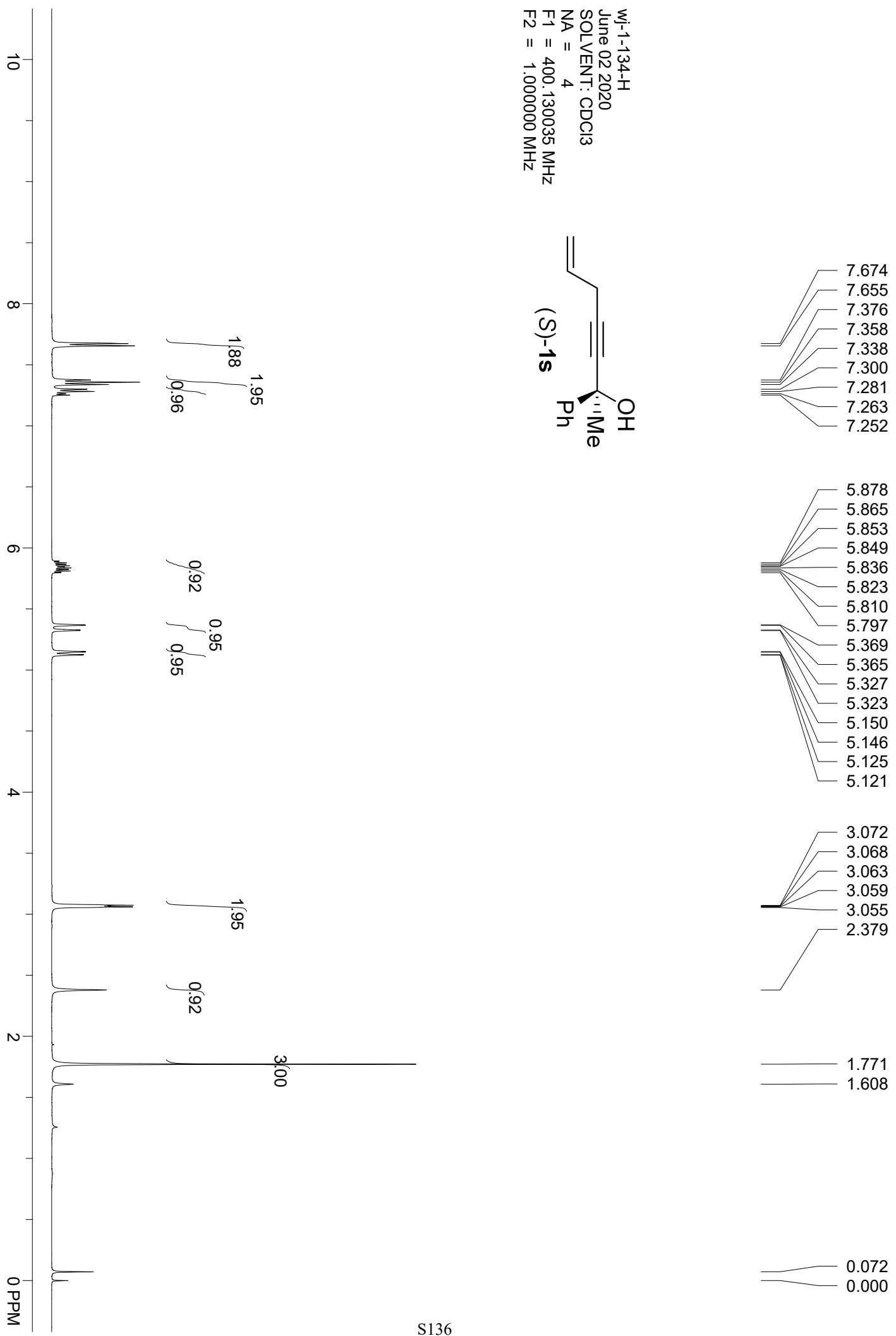
Data file: C:\Users\Public\Documents\ChemStation\1\Data\WJ_LC 2019-10-17 17-55-02\005-P2-C2-wj-1-082-1-rac.D

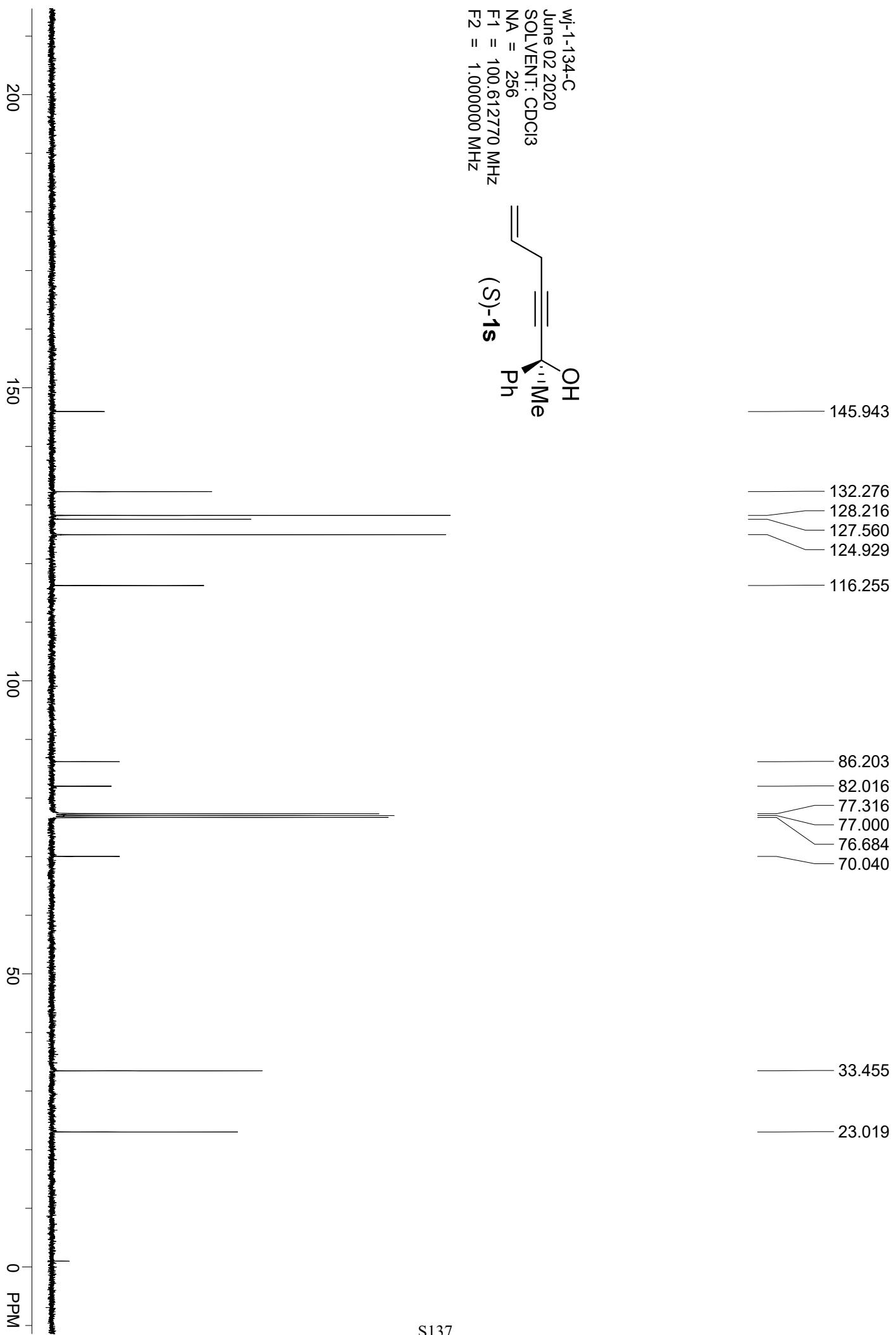
Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
16.973	0.4430	745.3535	21145.2012	49.8992
29.330	0.8164	405.8014	21230.6484	50.1008
		Sum	42375.8496	100.0000





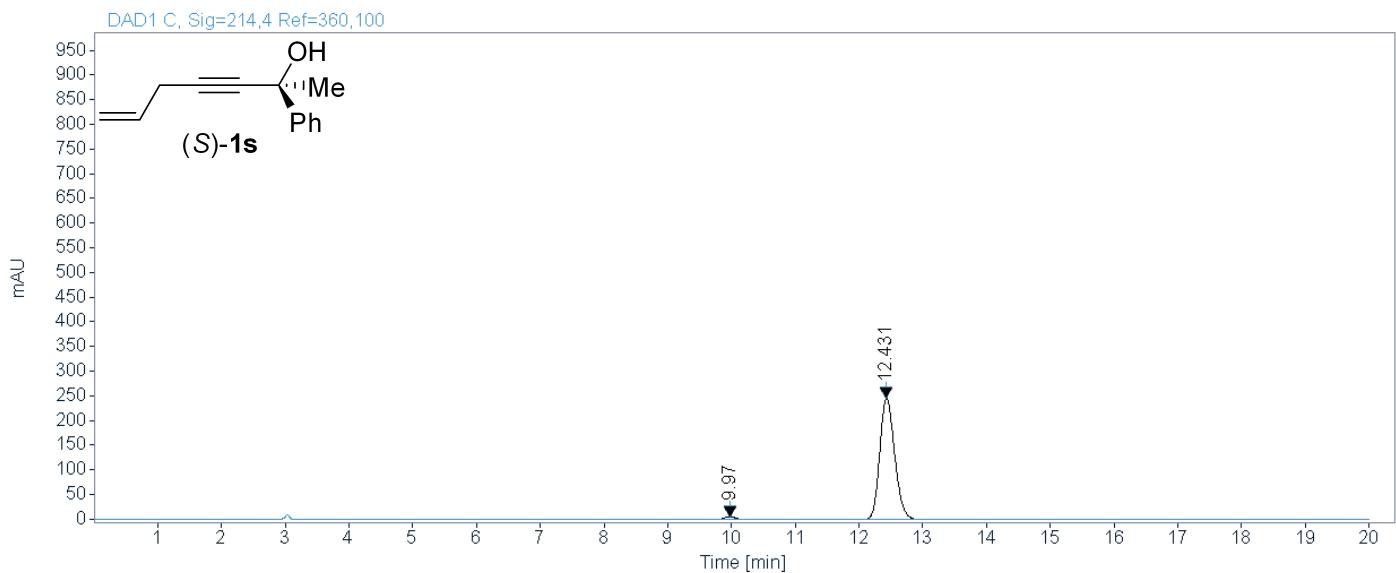
Area Percent Report



sample wj-1-134-AS-H-98-2-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\WJ_LC 2020-06-02 12-57-04\002-P2-C1-wj-1-134.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
9.970	0.1894	6.0447	74.3090	1.8280
12.431	0.2487	246.7380	3990.7153	98.1720
		Sum	4065.0243	100.0000

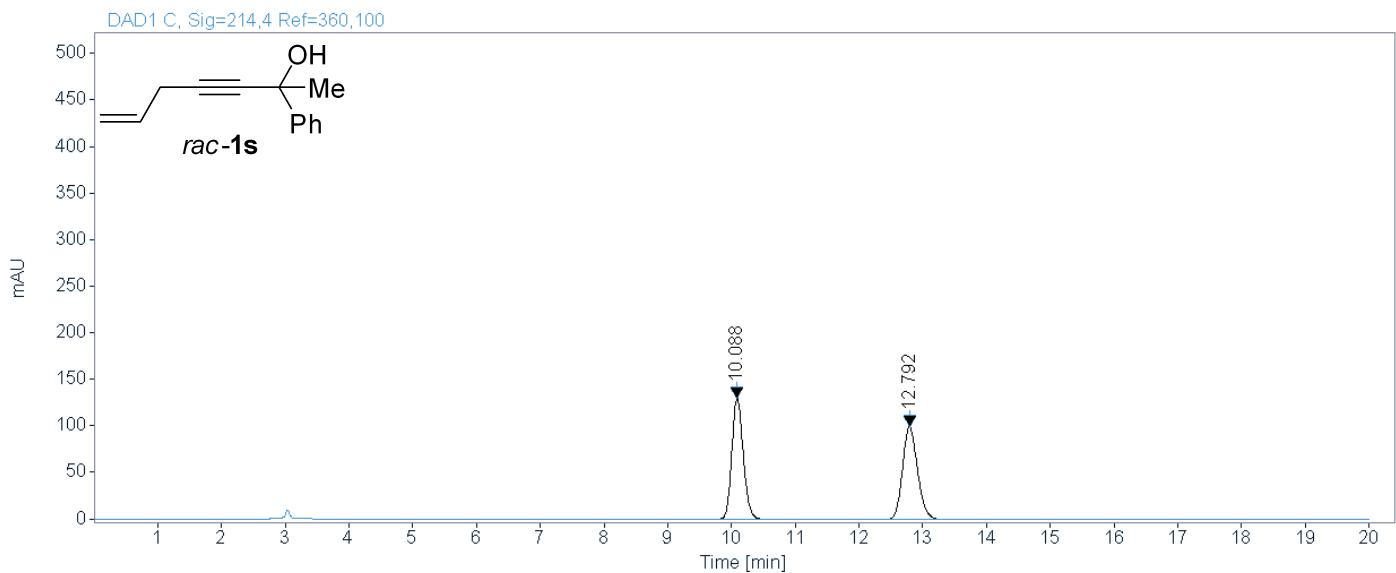
Area Percent Report



sample wj-1-134-rac-AS-H-98-2-1.0-214

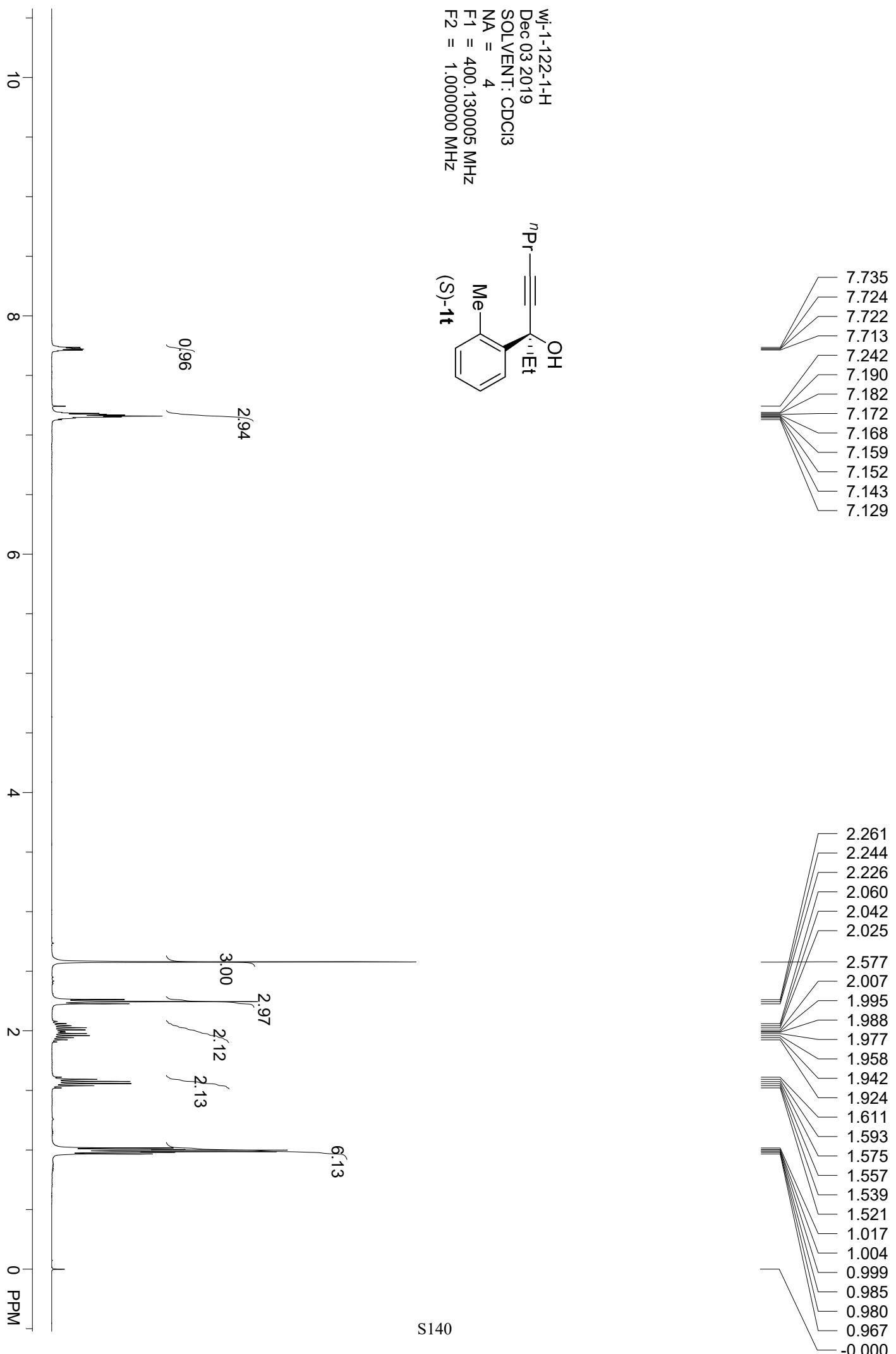
Data file: C:\Users\Public\Documents\ChemStation\1\Data\WJ_LC 2020-06-02 12-57-04\003-P2-C2-wj-1-134-rac.D

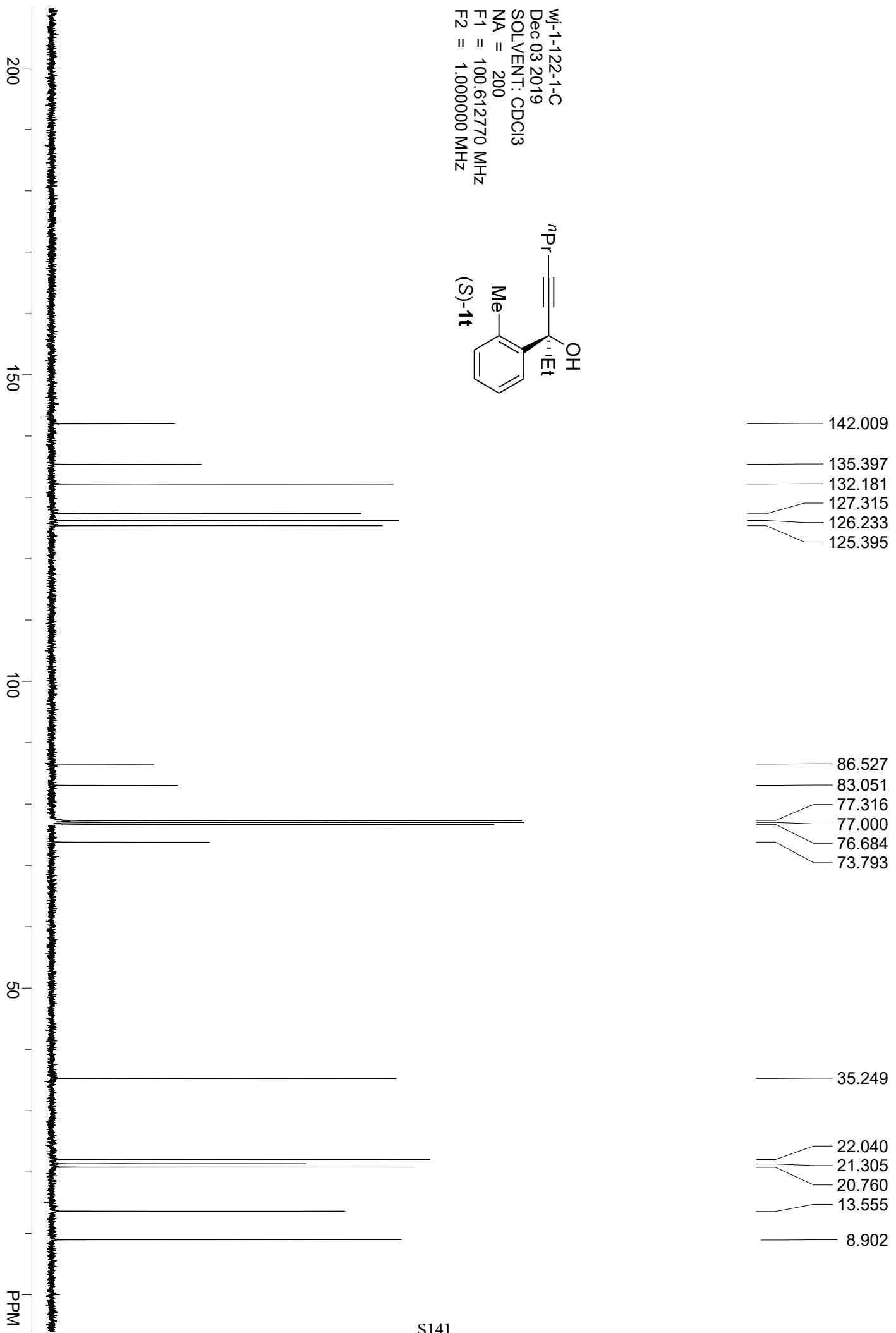
Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
10.088	0.1920	130.3764	1631.1051	49.9835
12.792	0.2531	99.6525	1632.1830	50.0165
Sum		3263.2881	100.0000	





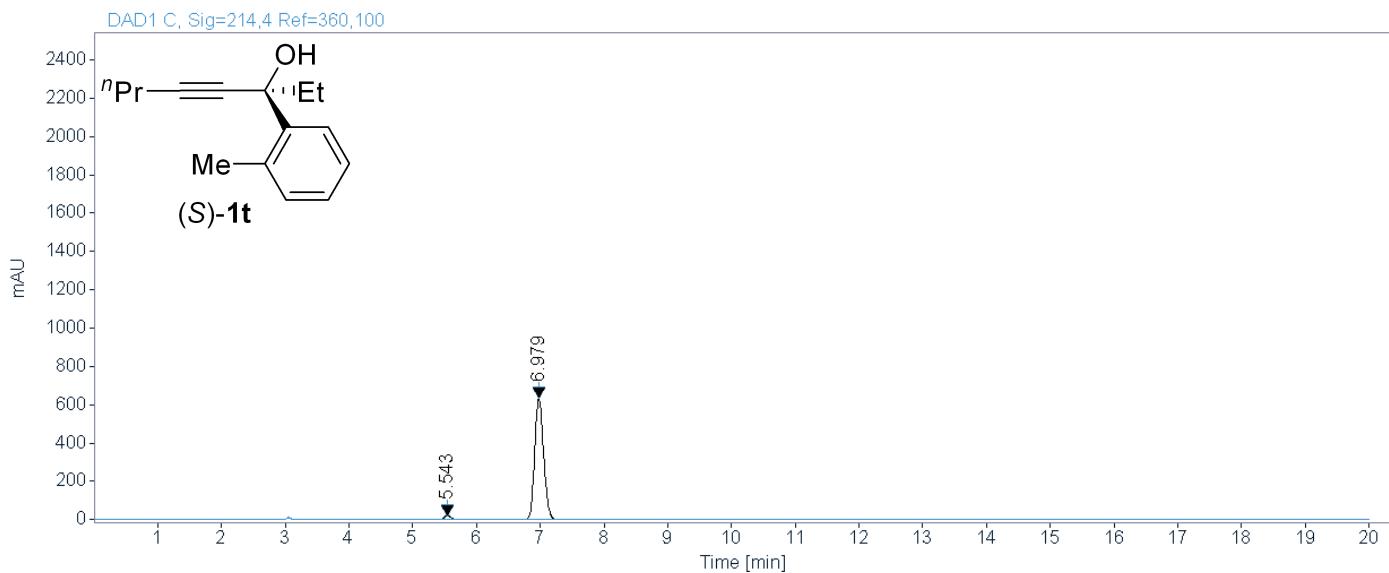
Area Percent Report



sample wj-1-122-1-AS-H-98-2-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\zwf-allenioc acid_LC 2019-12-03 19-19-43\010-P2-C1-wj-1-122-1.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
5.543	0.1144	21.9182	162.2364	2.5587
6.979	0.1512	636.6016	6178.2861	97.4413
Sum		6340.5225	100.0000	

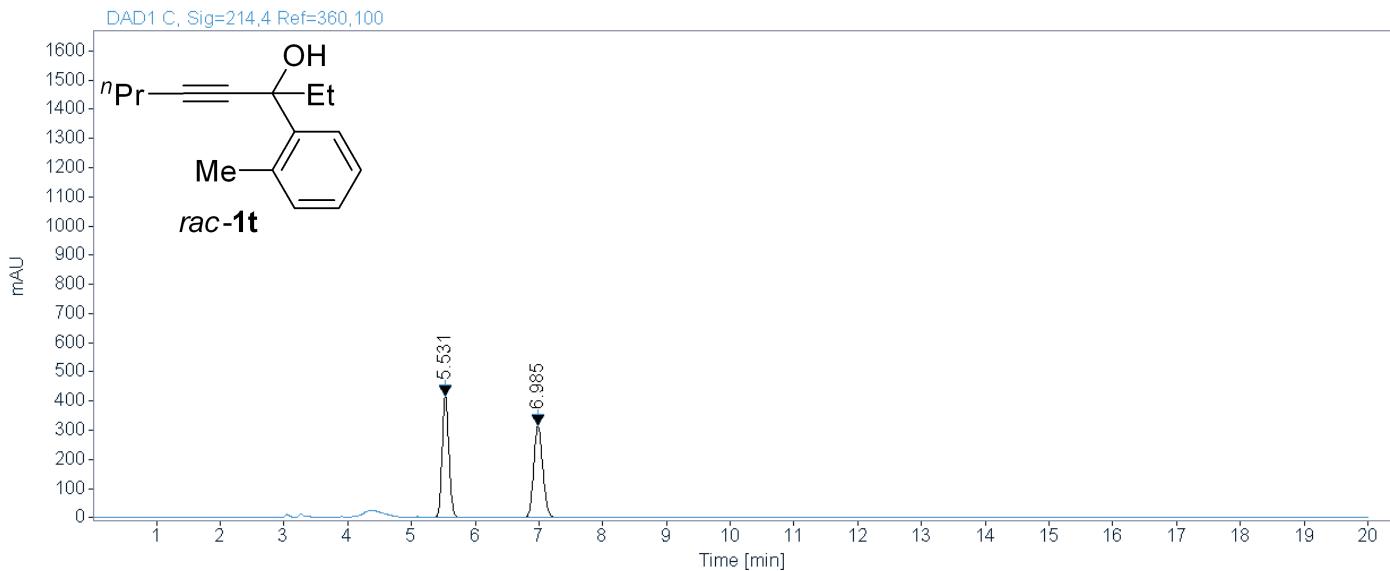
Area Percent Report



sample wj-1-122-1-rac-AS-H-98-2-1.0-
214

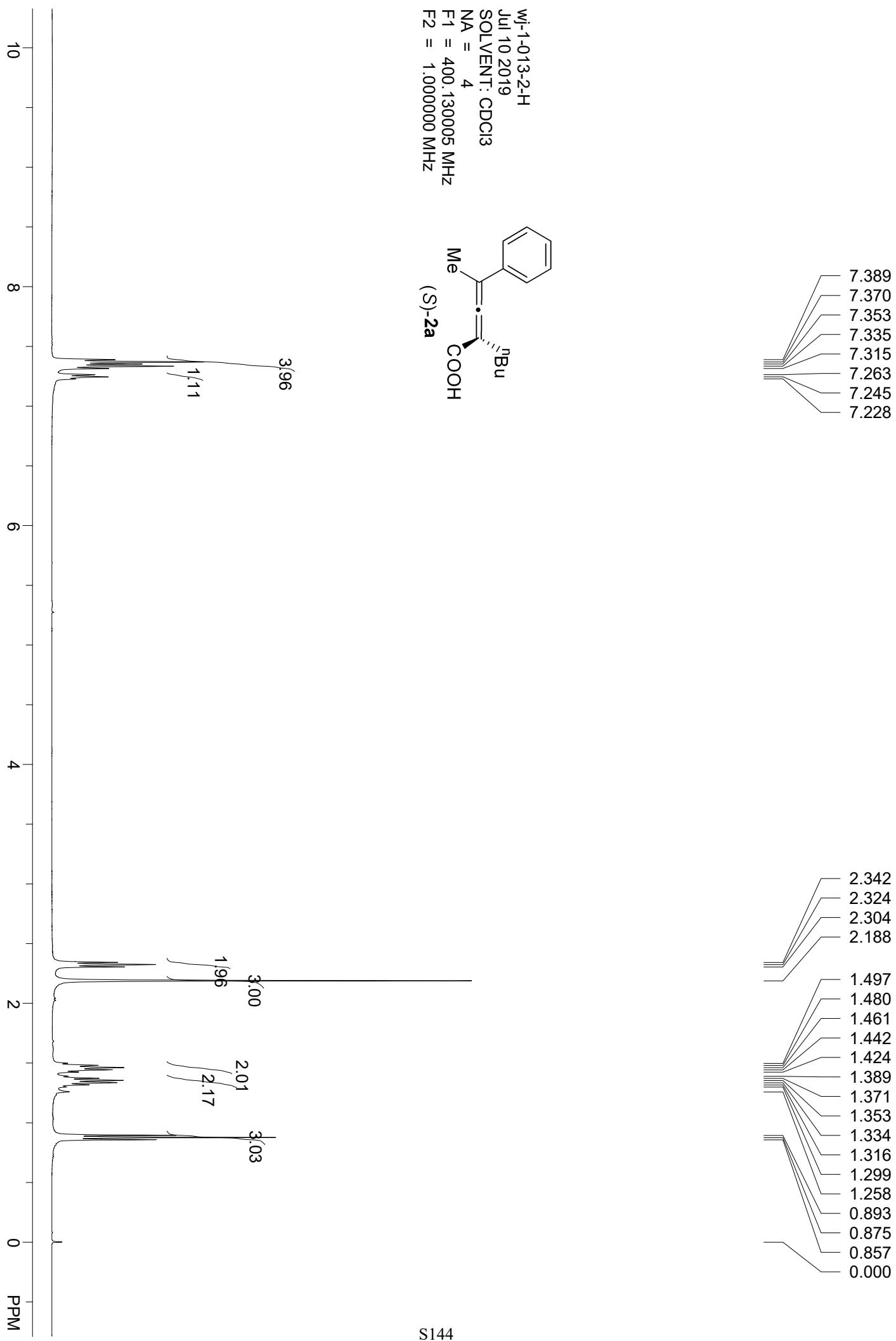
Data file: C:\Users\Public\Documents\ChemStation\1\Data\zwf-allenioc acid_LC 2019-12-03 19-19-43\011-P2-C2-wj-1-122-1-rac.D

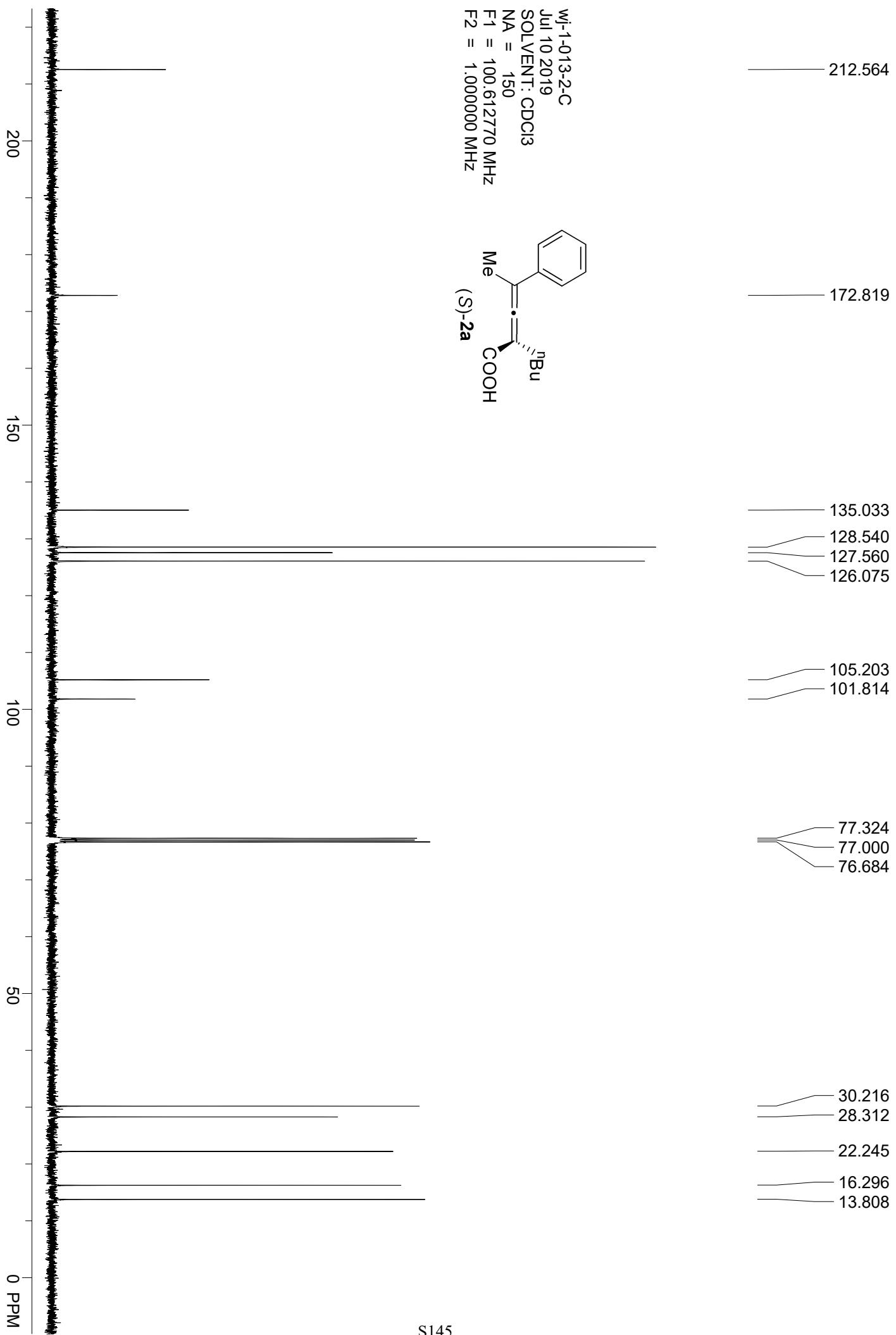
Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
5.531	0.1147	417.5157	3102.4727	49.8900
6.985	0.1533	315.4018	3116.1555	50.1100
Sum		6218.6282	100.0000	





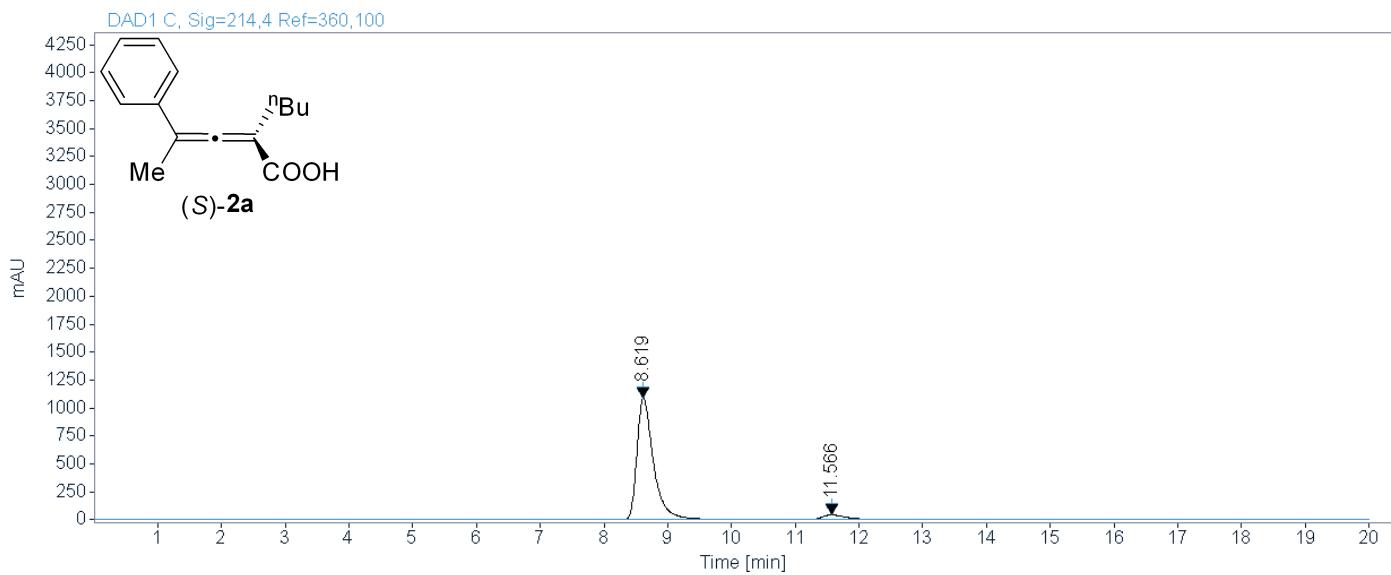
Area Percent Report



sample wj-1-013-2-AS-H-98-2-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\WJ_LC 2019-07-11 09-14-49\006-P2-C4-wj-1-013-2.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
8.619	0.2713	1090.7281	19793.5664	95.0068
11.566	0.4164	41.6374	1040.2765	4.9932
		Sum	20833.8429	100.0000

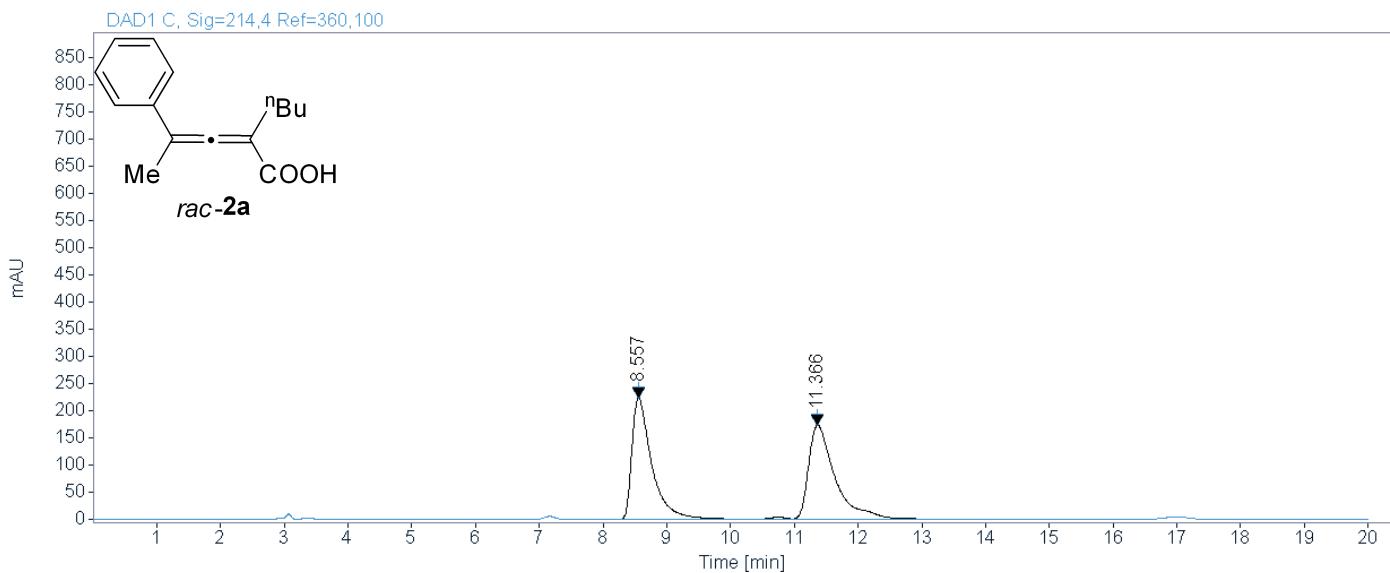
Area Percent Report



sample wj-1-013-2-rac-AS-H-98-2-
1.0-214

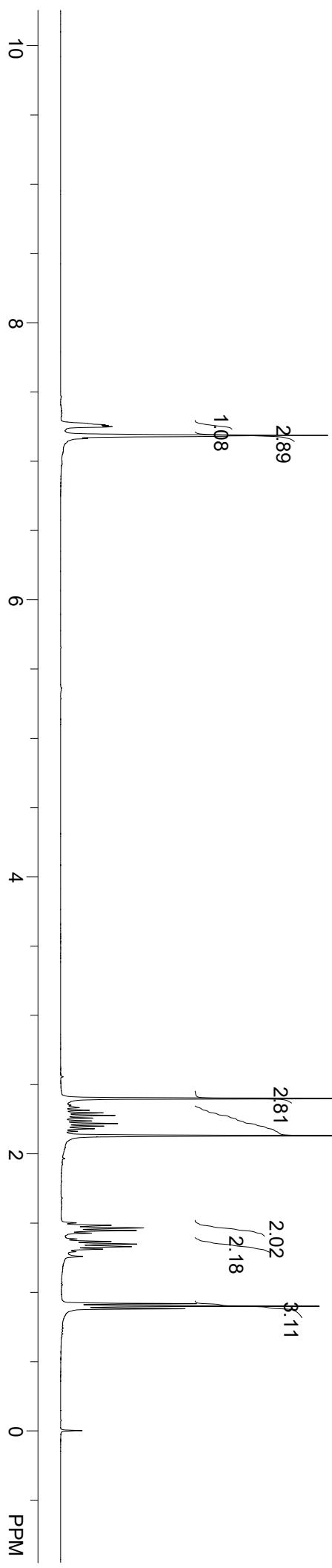
Data file: C:\Users\Public\Documents\ChemStation\1\Data\WJ_LC 2019-07-10 19-09-25\008-P2-C6-wj-1-013-2-rac.D

Acquisition Data:

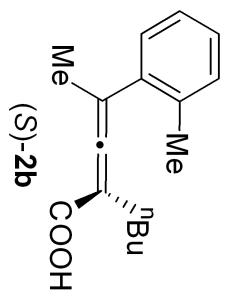


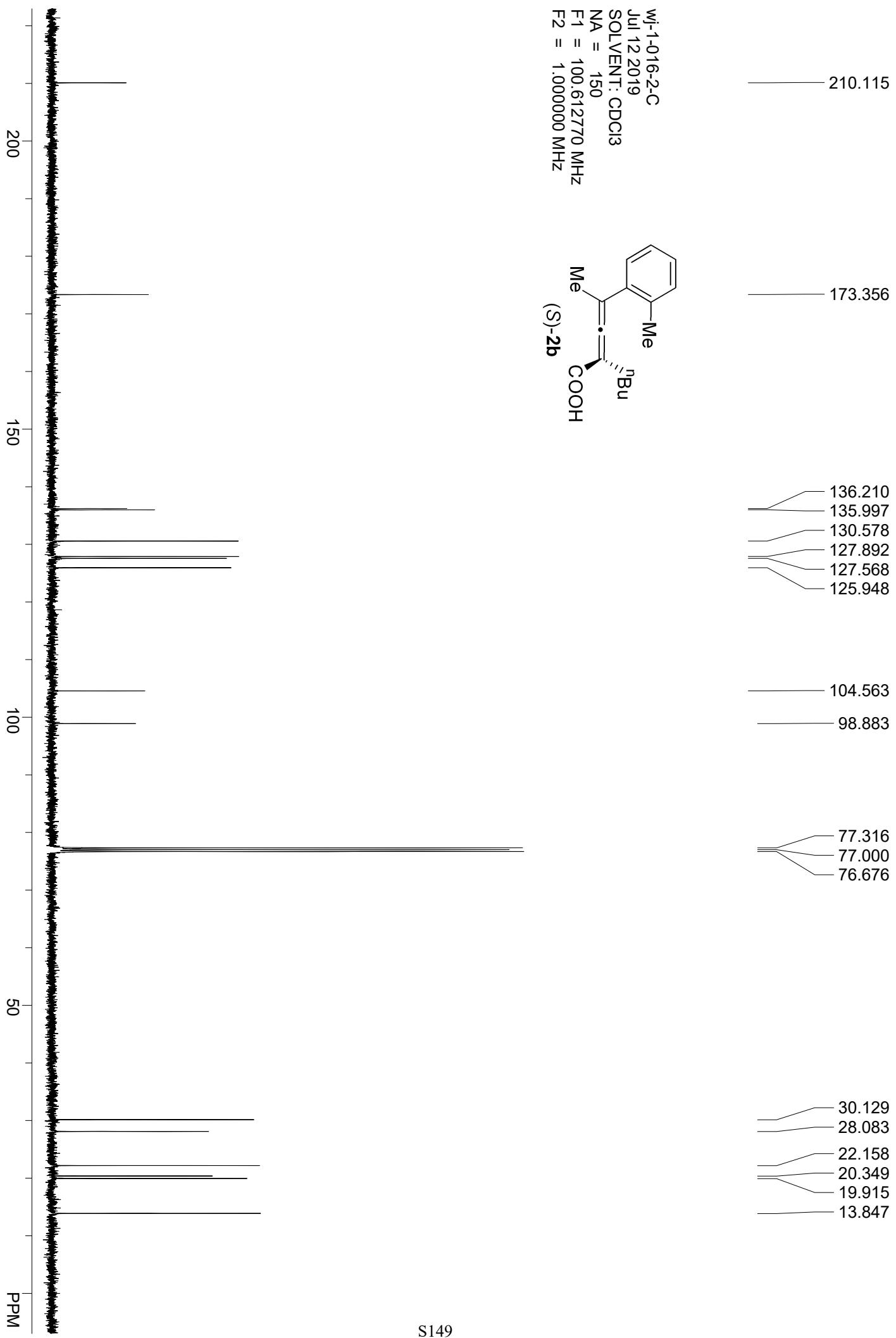
Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
8.557	0.3203	223.4749	4833.5288	48.7991
11.366	0.4277	173.1143	5071.4165	51.2009
Sum		9904.9453	100.0000	



wj-1-016-2-H
Jul 12 2019
SOLVENT: CDCl₃
NA = 4
F1 = 400.130005 MHz
F2 = 1.000000 MHz





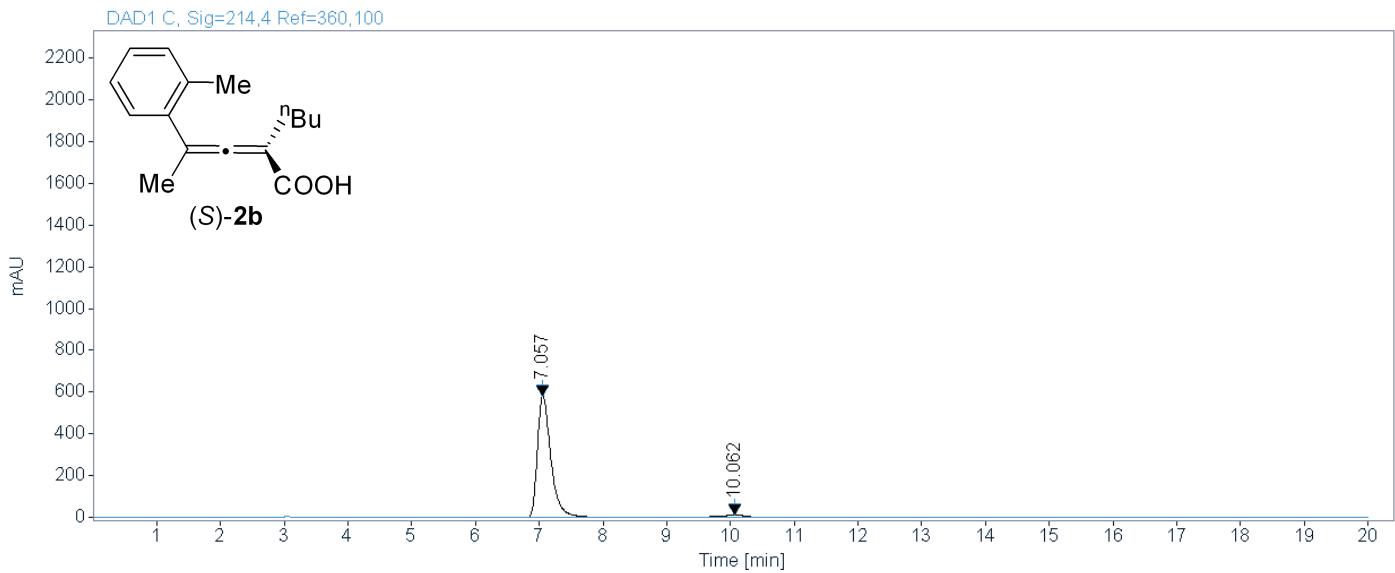
Area Percent Report



sample wj-1-016-2-AS-H-98-2-1.0-
214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\WJ_LC 2019-07-12 22-04-29\009-P2-C7-wj-1-016-2.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
7.057	0.2239	582.6659	8584.3232	95.9713
10.062	0.4107	12.6379	360.3499	4.0287
Sum		8944.6731	100.0000	

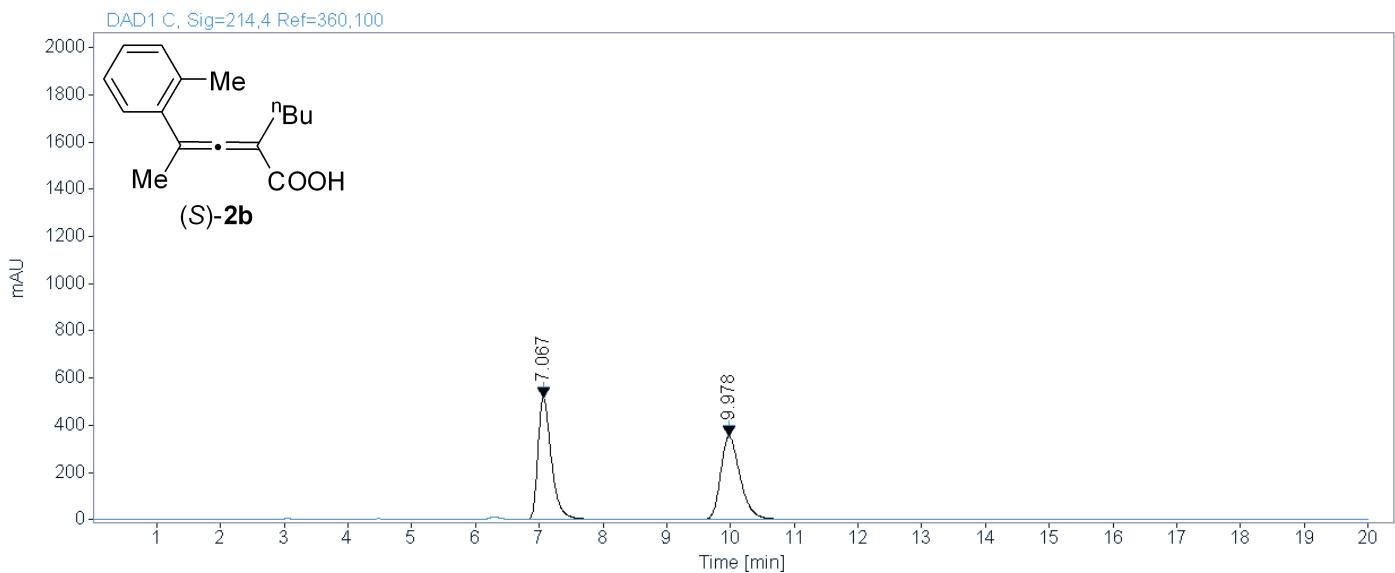
Area Percent Report



sample wj-1-016-2-rac-AS-H-98-2-
1.0-214

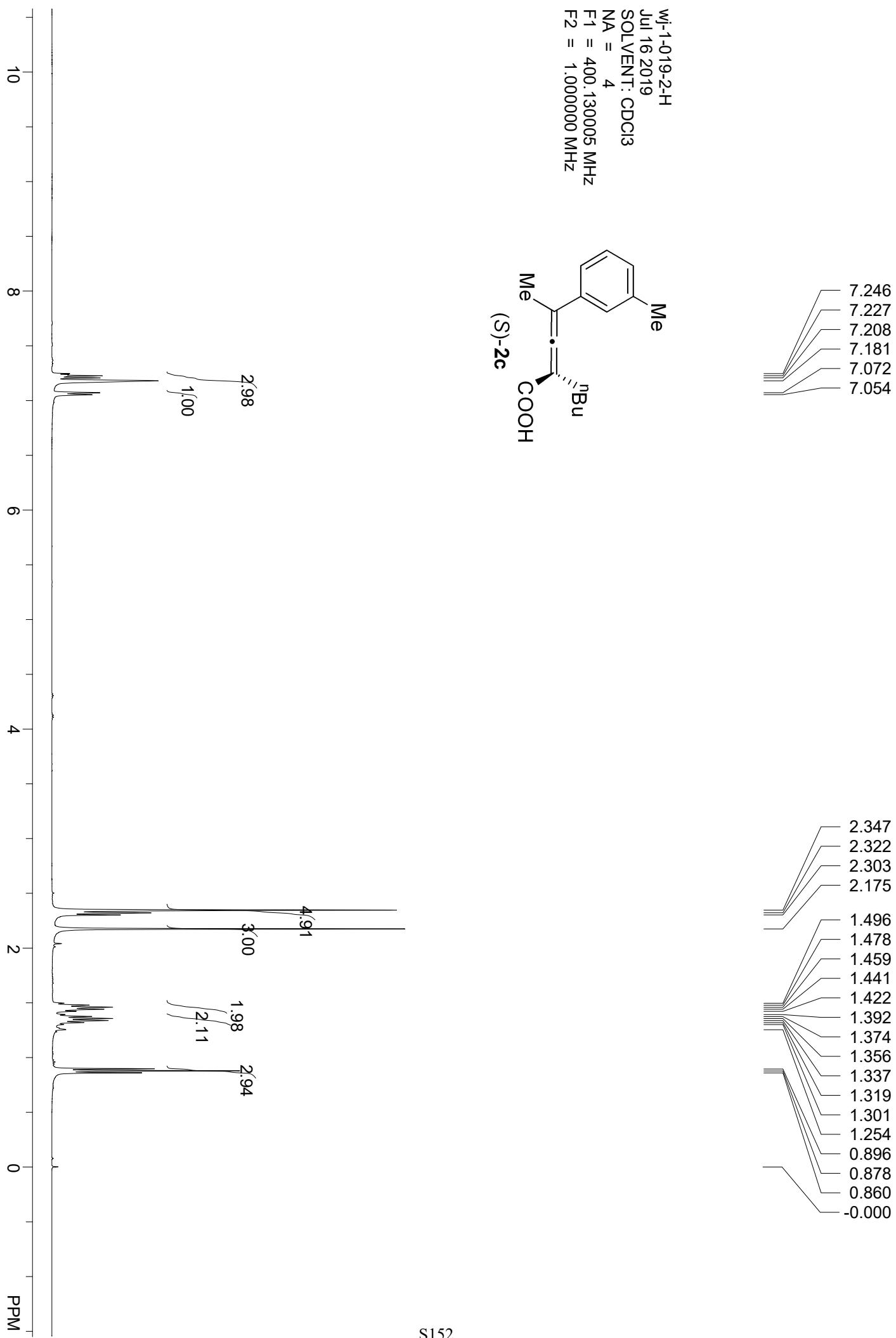
Data file: C:\Users\Public\Documents\ChemStation\1\Data\WJ_LC 2019-07-12 22-04-29\010-P2-C8-wj-1-016-2-rac.D

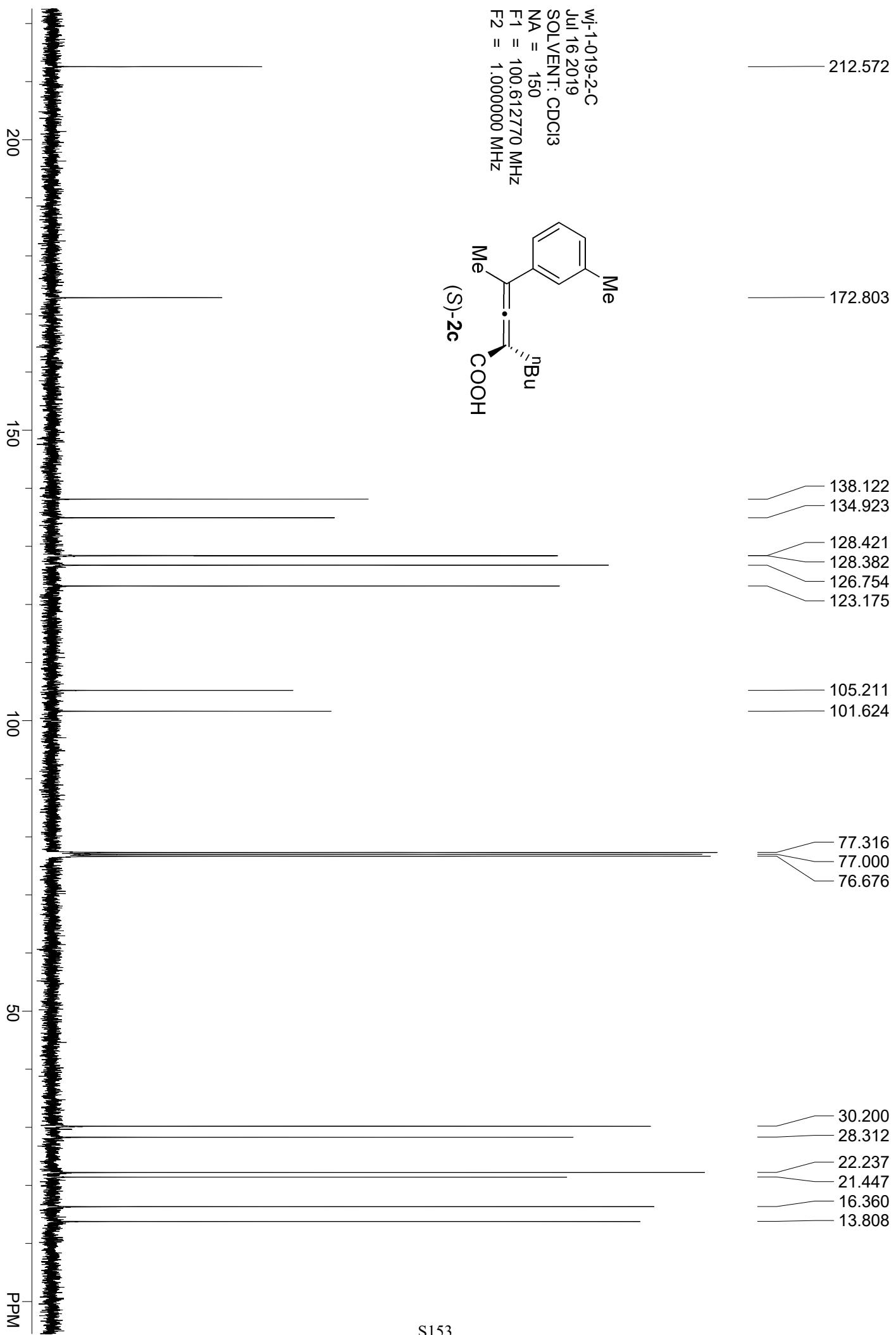
Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
7.067	0.2220	514.9291	7503.5029	49.8546
9.978	0.3301	352.0159	7547.2827	50.1454
Sum		15050.7856	100.0000	





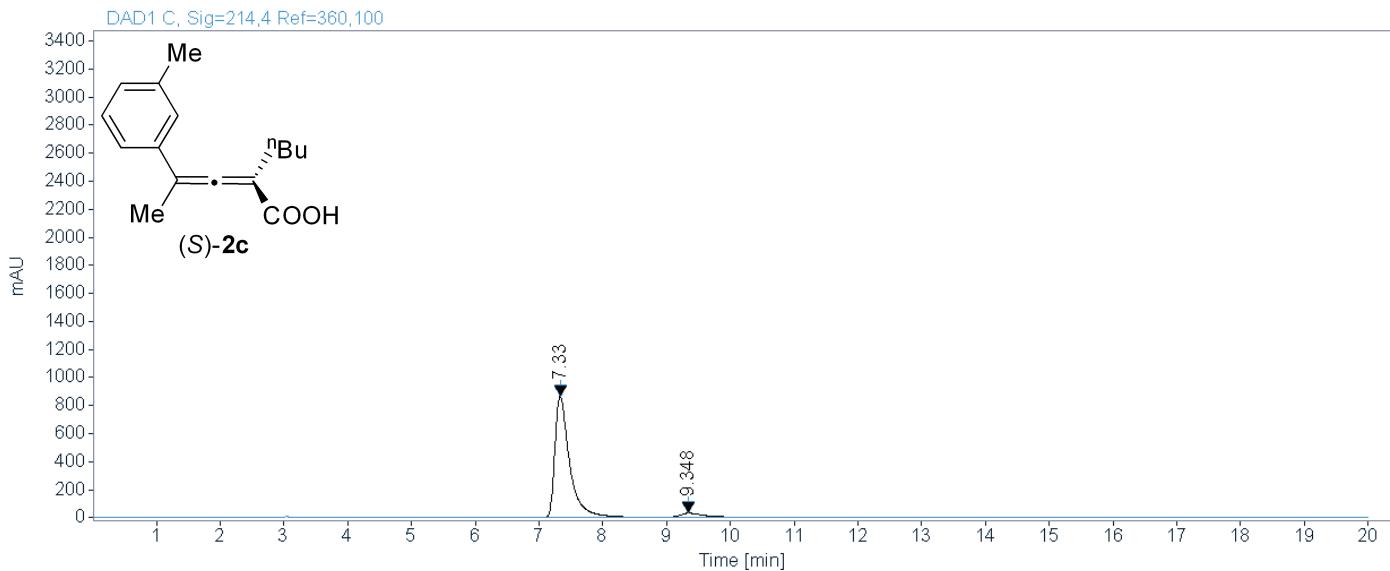
Area Percent Report



sample wj-1-019-2-AS-H-98-2-
1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\zwf-allenioc acid_LC 2019-07-15 22-12-25\005-P2-C3-wj-1-019-2.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
7.330	0.2404	868.6541	14024.9150	95.6821
9.348	0.3852	27.3880	632.9089	4.3179
Sum		14657.8240	100.0000	

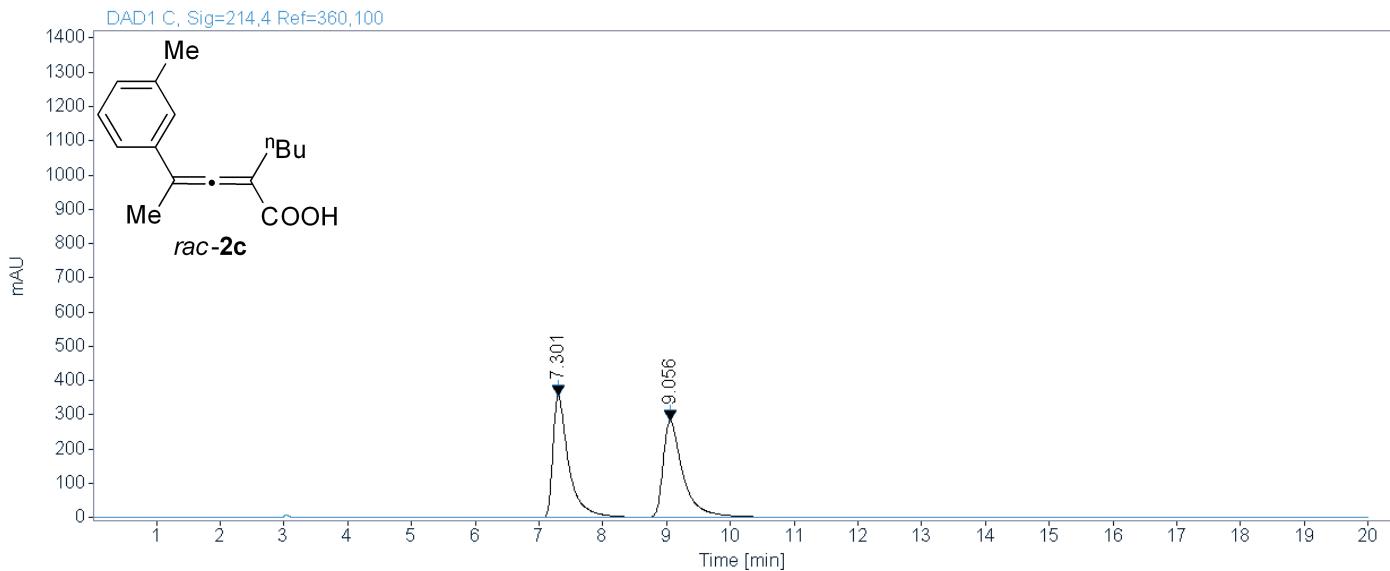
Area Percent Report



sample wj-1-019-2-rac-AS-H-98-
2-1.0-214

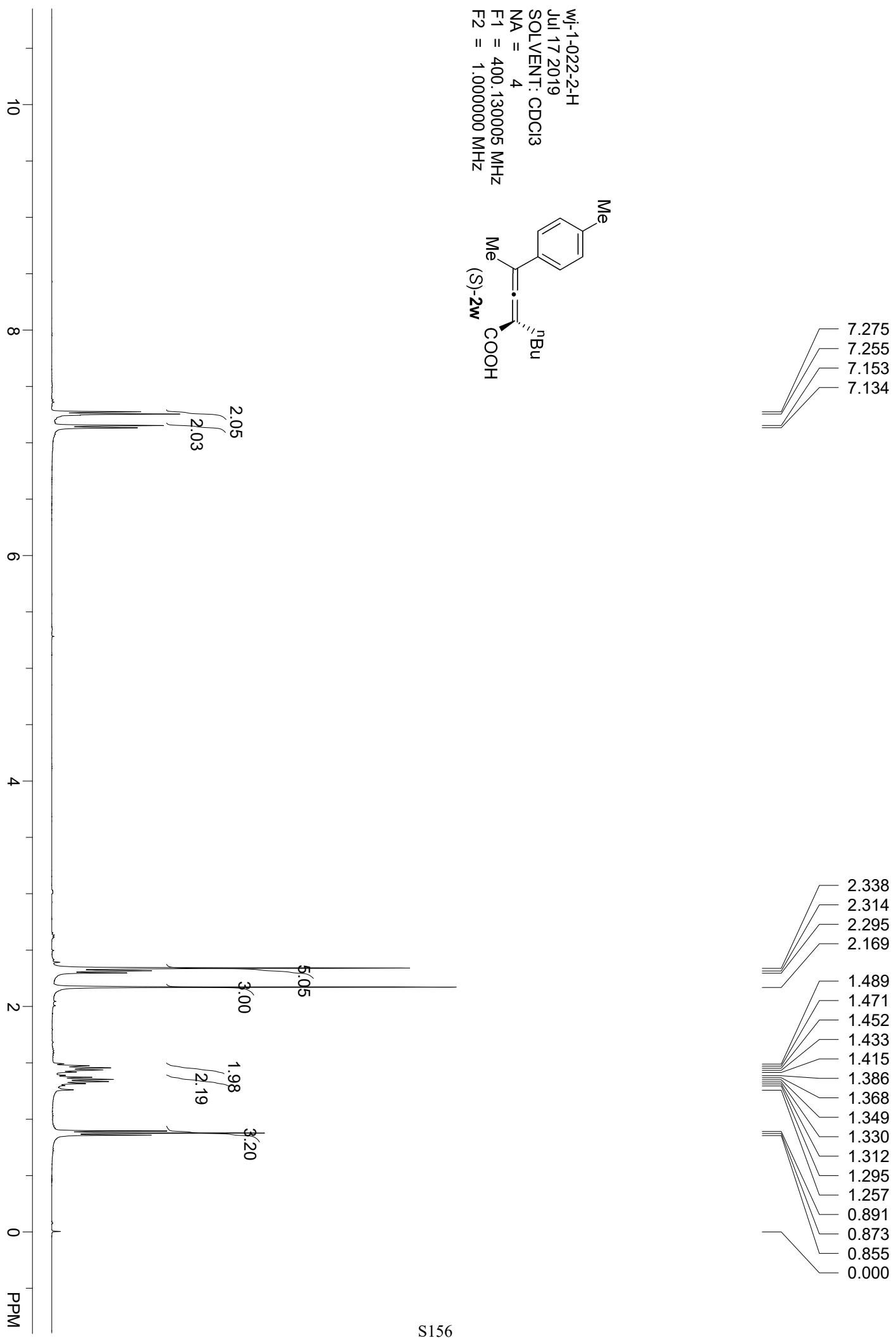
Data file: C:\Users\Public\Documents\ChemStation\1\Data\zwf-allenioc acid_LC 2019-07-15 22-12-25\006-P2-C4-wj-1-019-2-rac.D

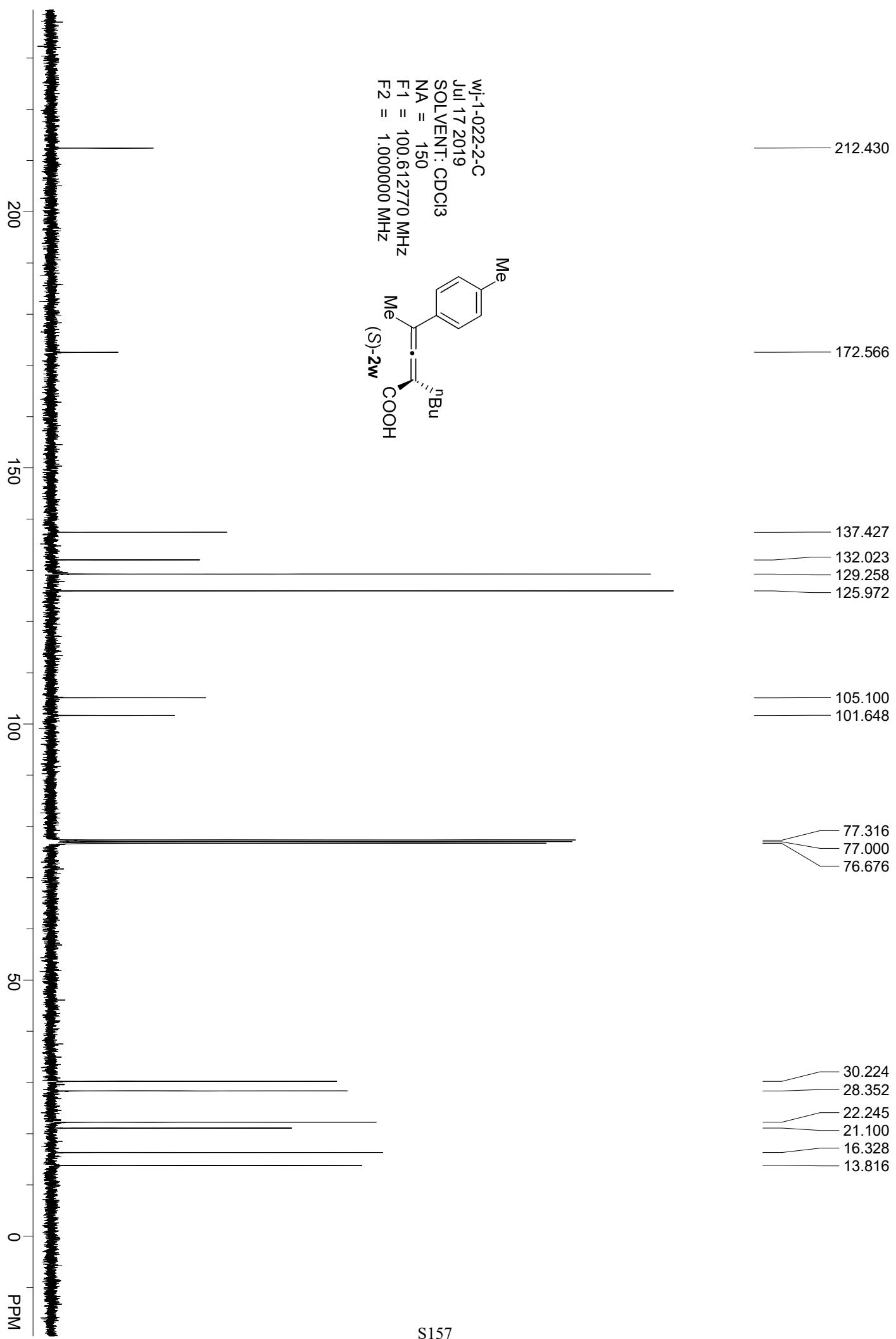
Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
7.301	0.2540	355.2681	6151.0254	49.7808
9.056	0.3249	281.8253	6205.1973	50.2192
Sum		12356.2227	12356.2227	100.0000





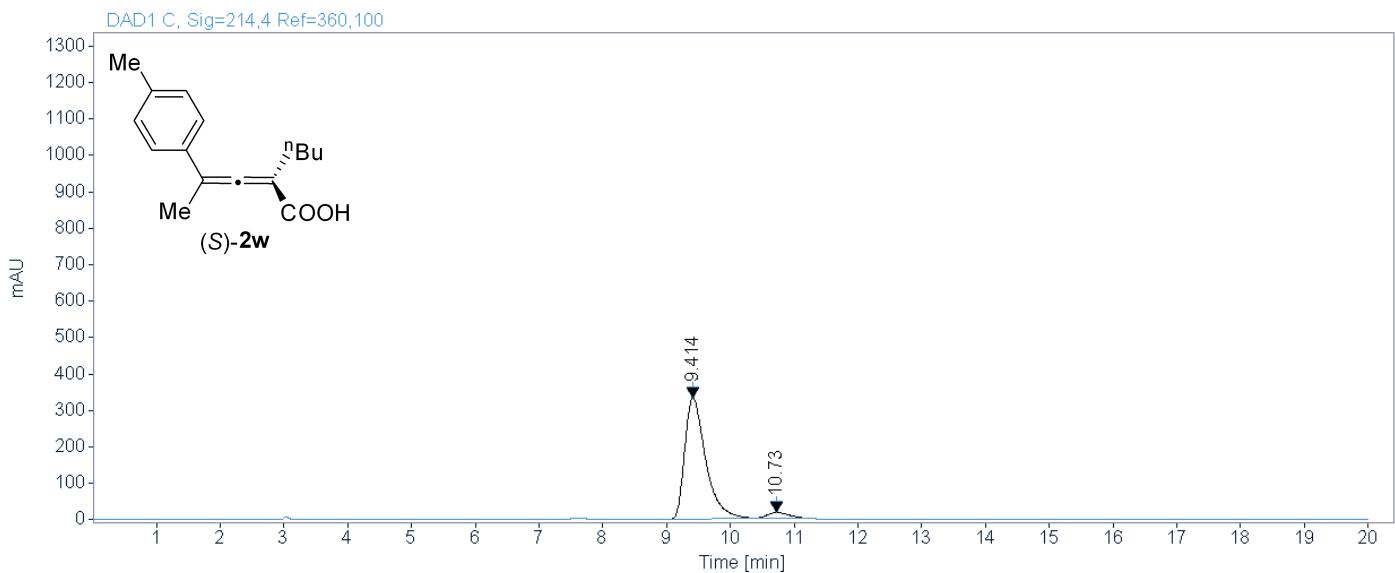
Area Percent Report



sample wj-1-022-2-AS-H-98-2-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\WJ_LC 2019-07-18 08-40-27\005-P2-C3-wj-1-022-2.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
9.414	0.3818	333.5311	7639.8374	95.3541
10.730	0.4053	15.3062	372.2325	4.6459
Sum		8012.0699	100.0000	

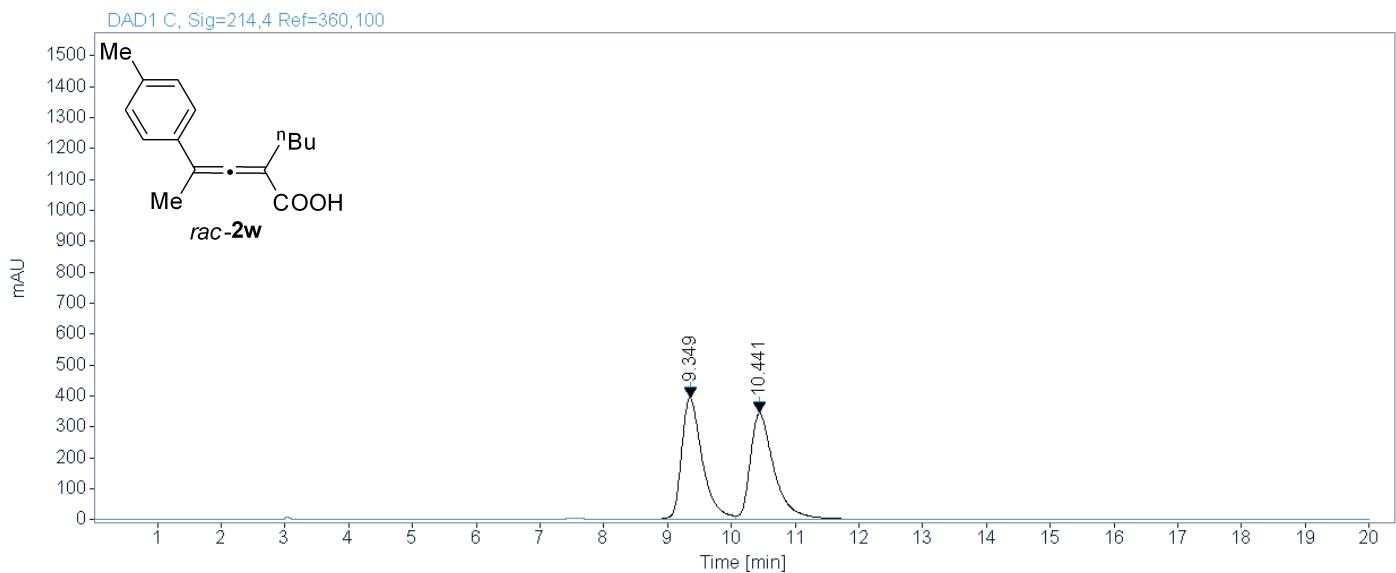
Area Percent Report



sample wj-1-022-2-rac-AS-H-98-2-1.0-214

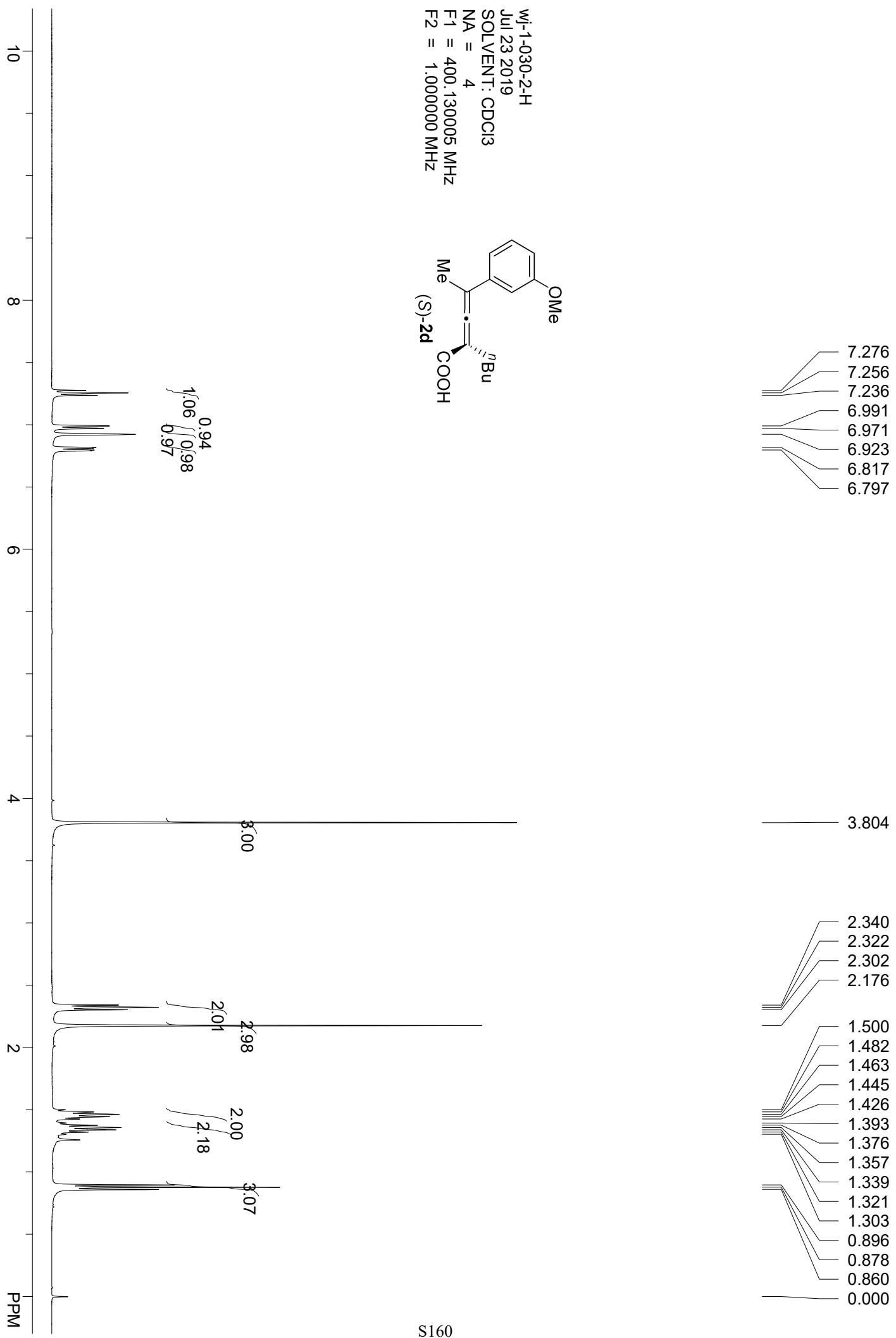
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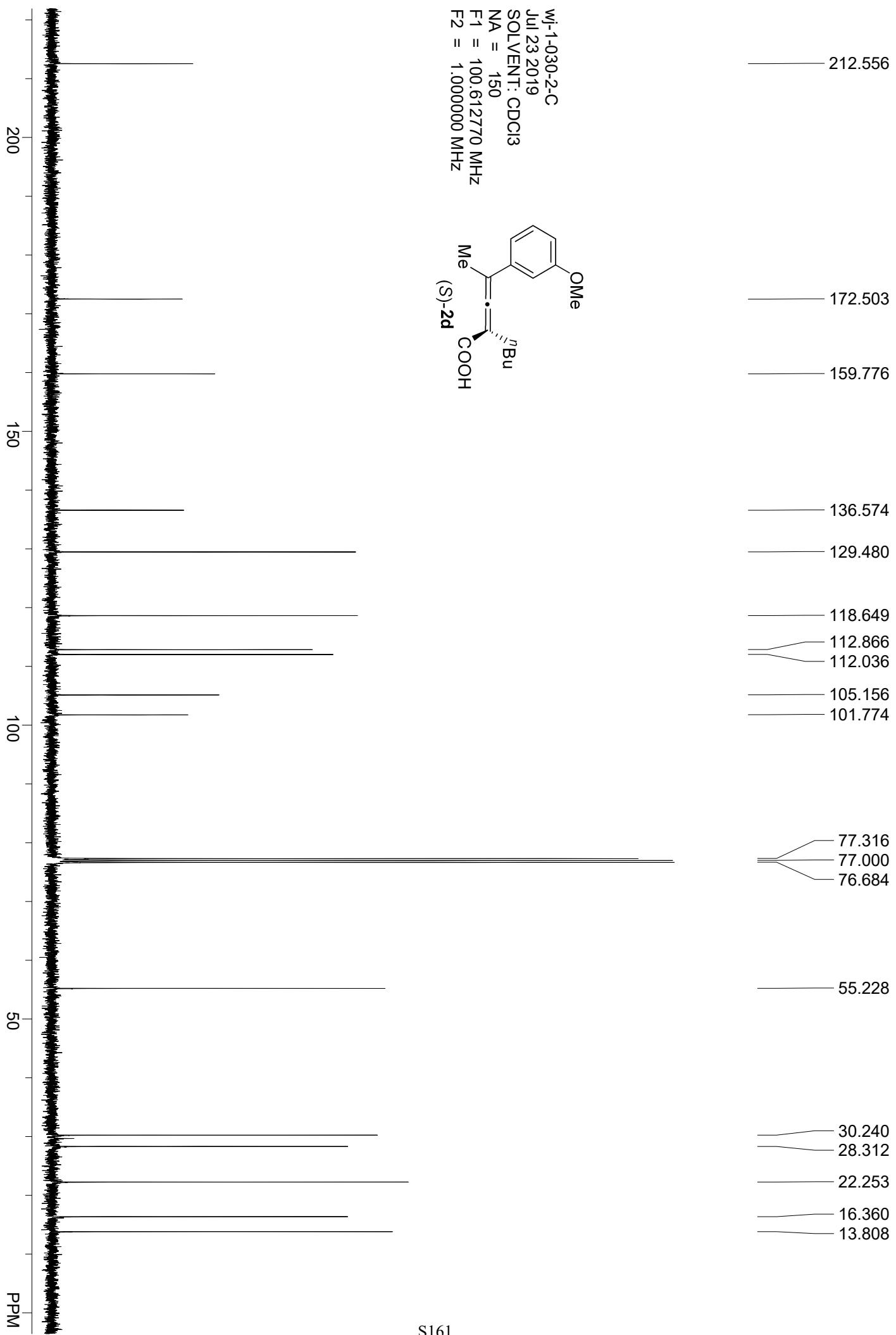
Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
9.349	0.3457	393.8154	8968.2070	49.6367
10.441	0.3968	343.7471	9099.5010	50.3633
		Sum	18067.7080	100.0000





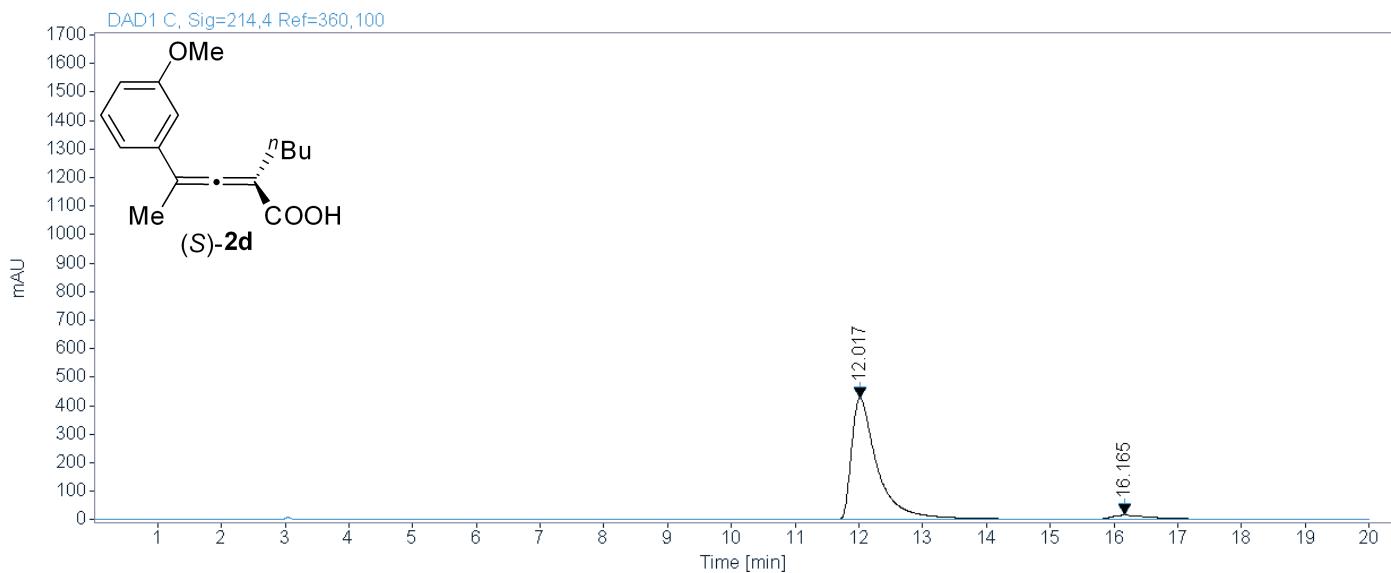
Area Percent Report



sample wj-1-030-2-AS-H-98-2-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\WJ_LC 2019-07-23 14-03-17\041-P2-C3-wj-1-030-2.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
12.017	0.4208	427.2359	12259.2998	95.2932
16.165	0.6995	14.4275	605.5215	4.7068
Sum		12864.8213	100.0000	

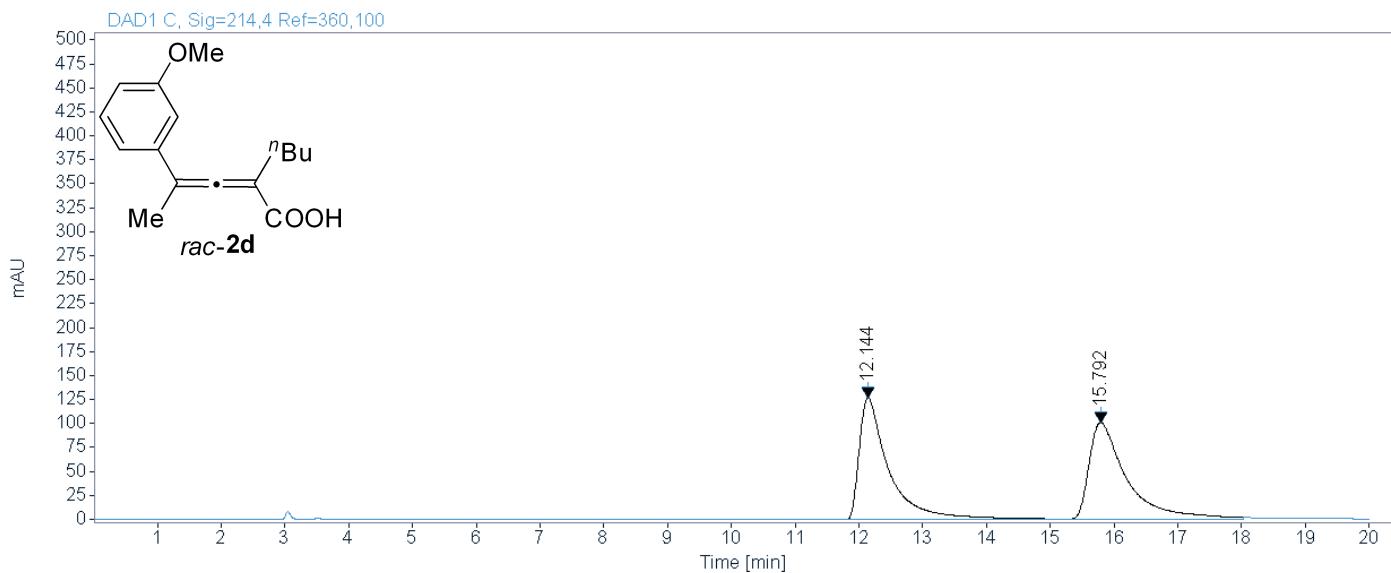
Area Percent Report



sample wj-1-030-2-rac-AS-H-98-2-1.0-214

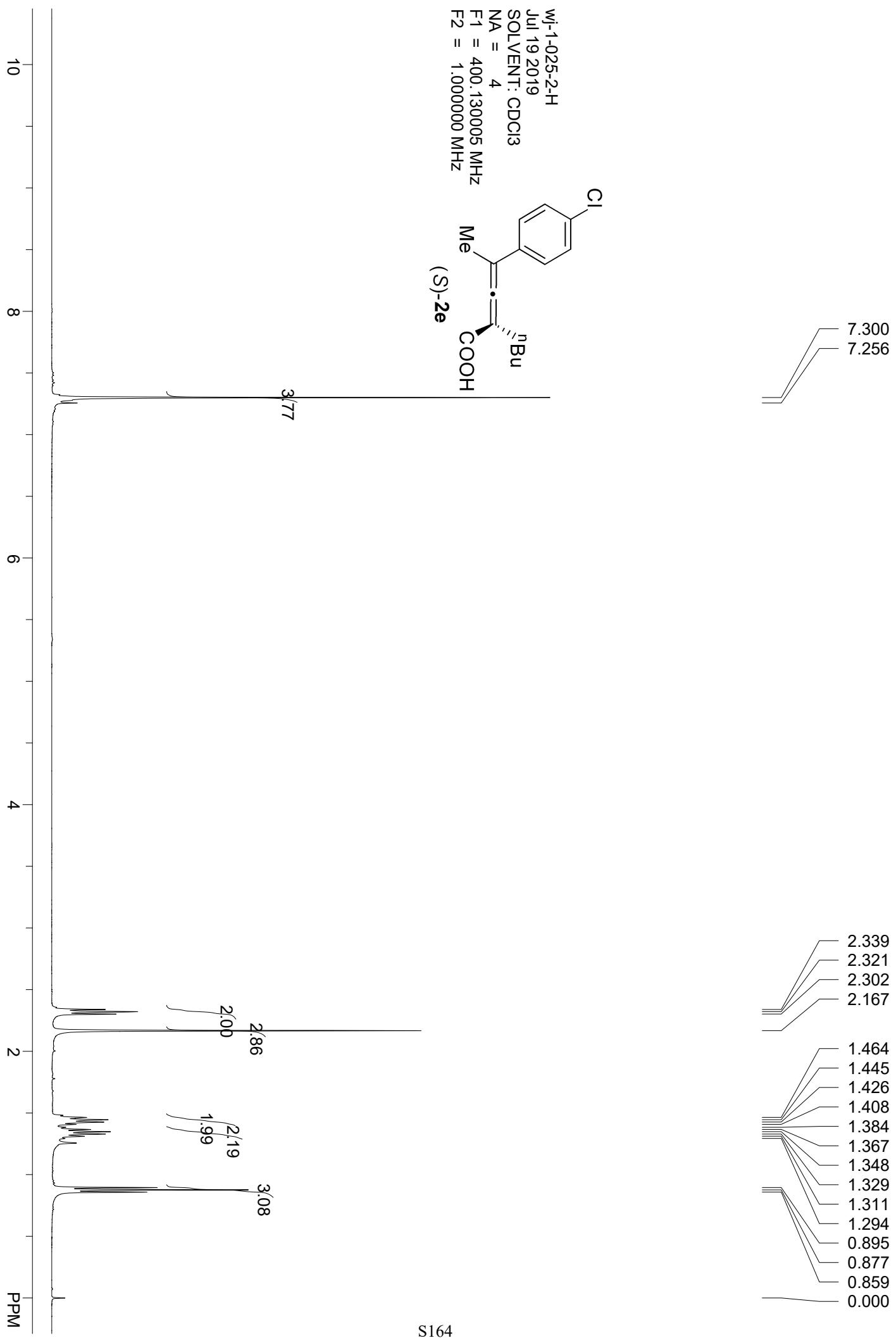
Data file: C:\Users\Public\Documents\ChemStation\1\Data\WJ_LC 2019-07-23 14-03-17\042-P2-C4-wj-1-030-2-rac.D

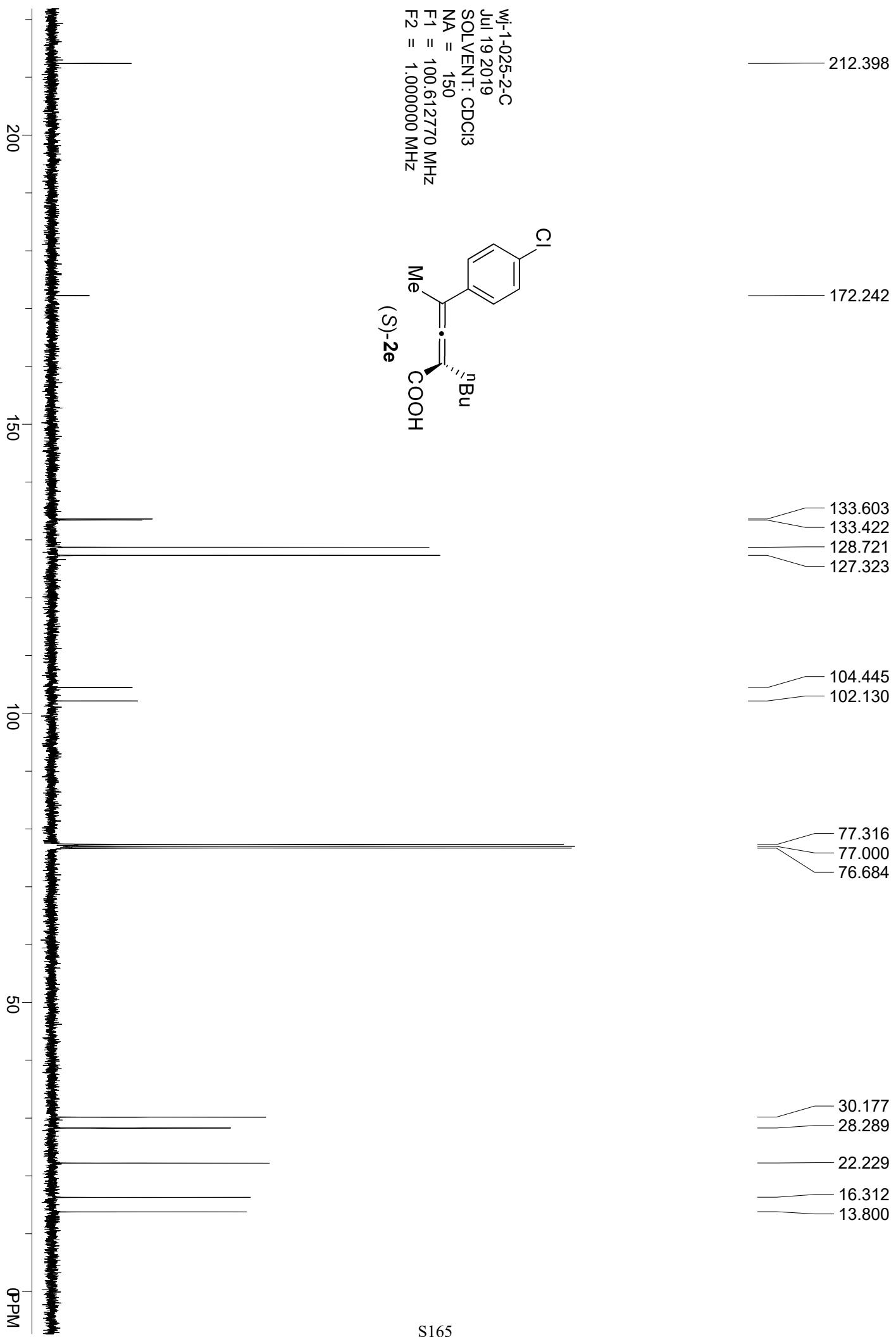
Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
12.144	0.4617	127.0507	4090.9783	49.9490
15.792	0.6793	100.5813	4099.3257	50.0510
Sum		8190.3040	100.0000	





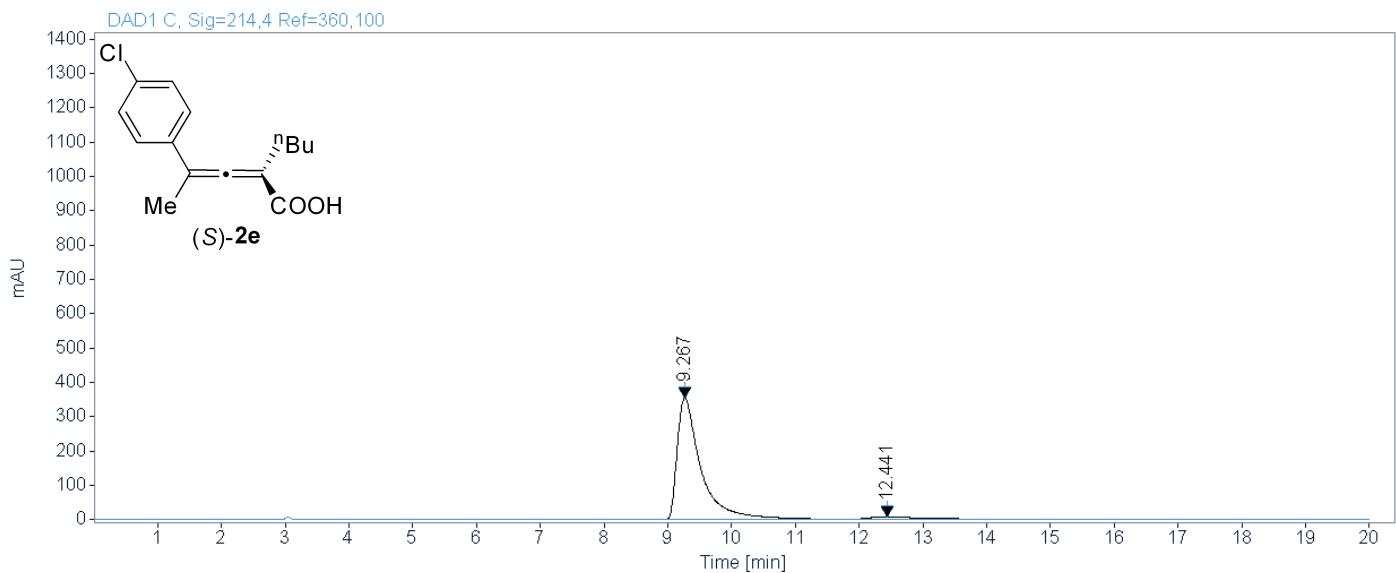
Area Percent Report



sample wj-1-025-2-AS-H-98-2-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\WJ_LC 2019-07-19 19-25-49\005-P2-C3-wj-1-025-2.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
9.267	0.3819	354.4984	9233.2666	95.5965
12.441	0.8294	7.4346	425.3109	4.4035
		Sum	9658.5775	100.0000

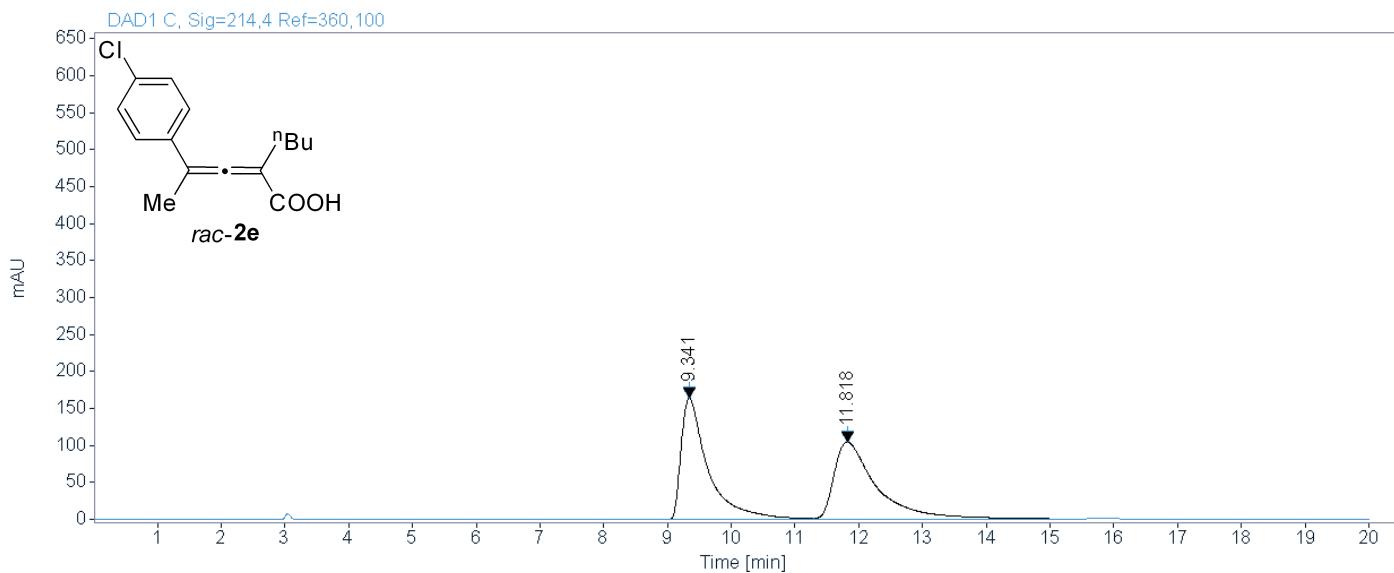
Area Percent Report



sample wj-1-025-2-rac-AS-H-98-2-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\WJ_LC 2019-07-19 19-25-49\006-P2-C4-wj-1-025-2-rac.D

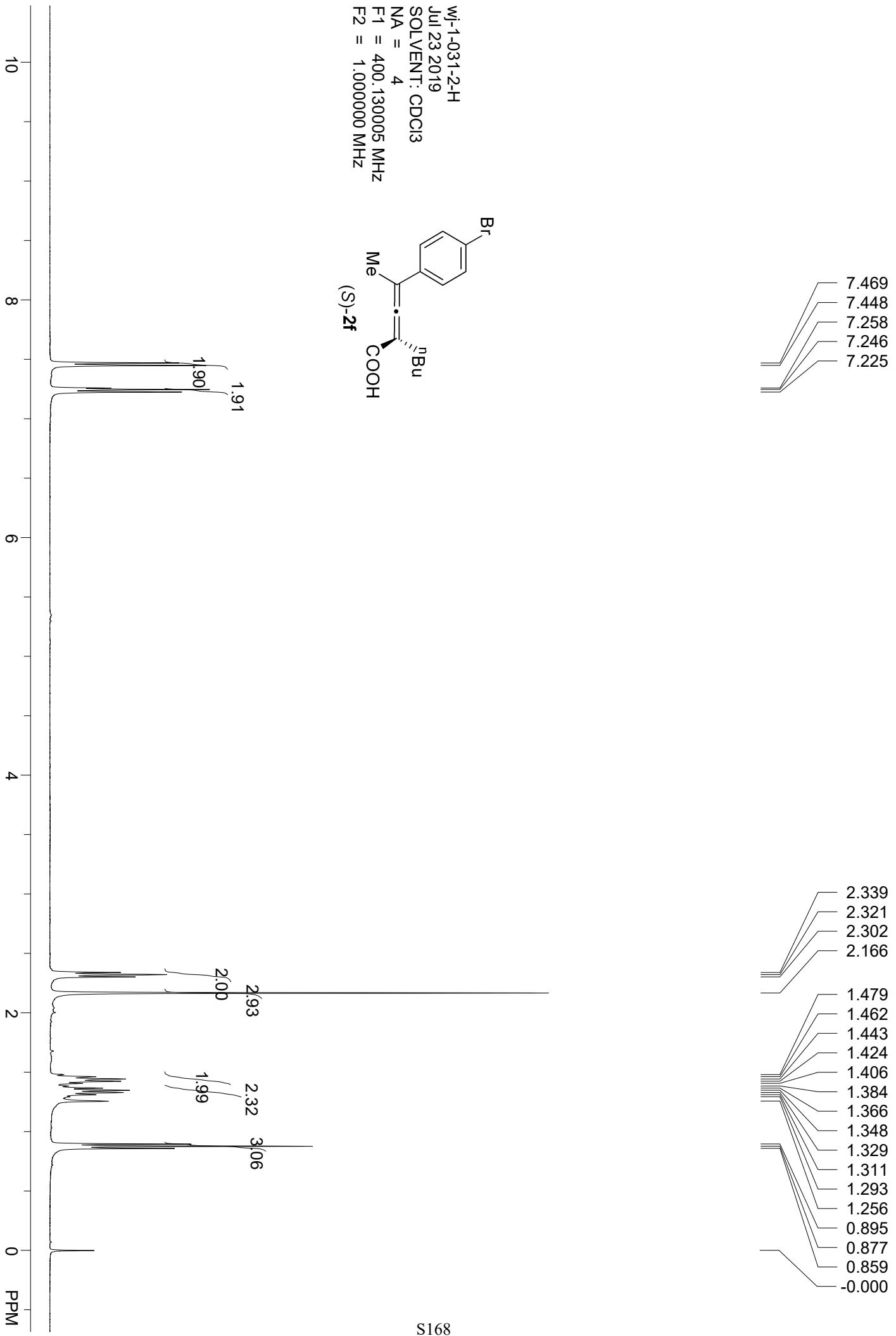
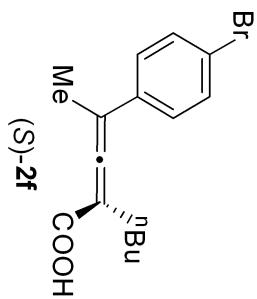
Acquisition Data:

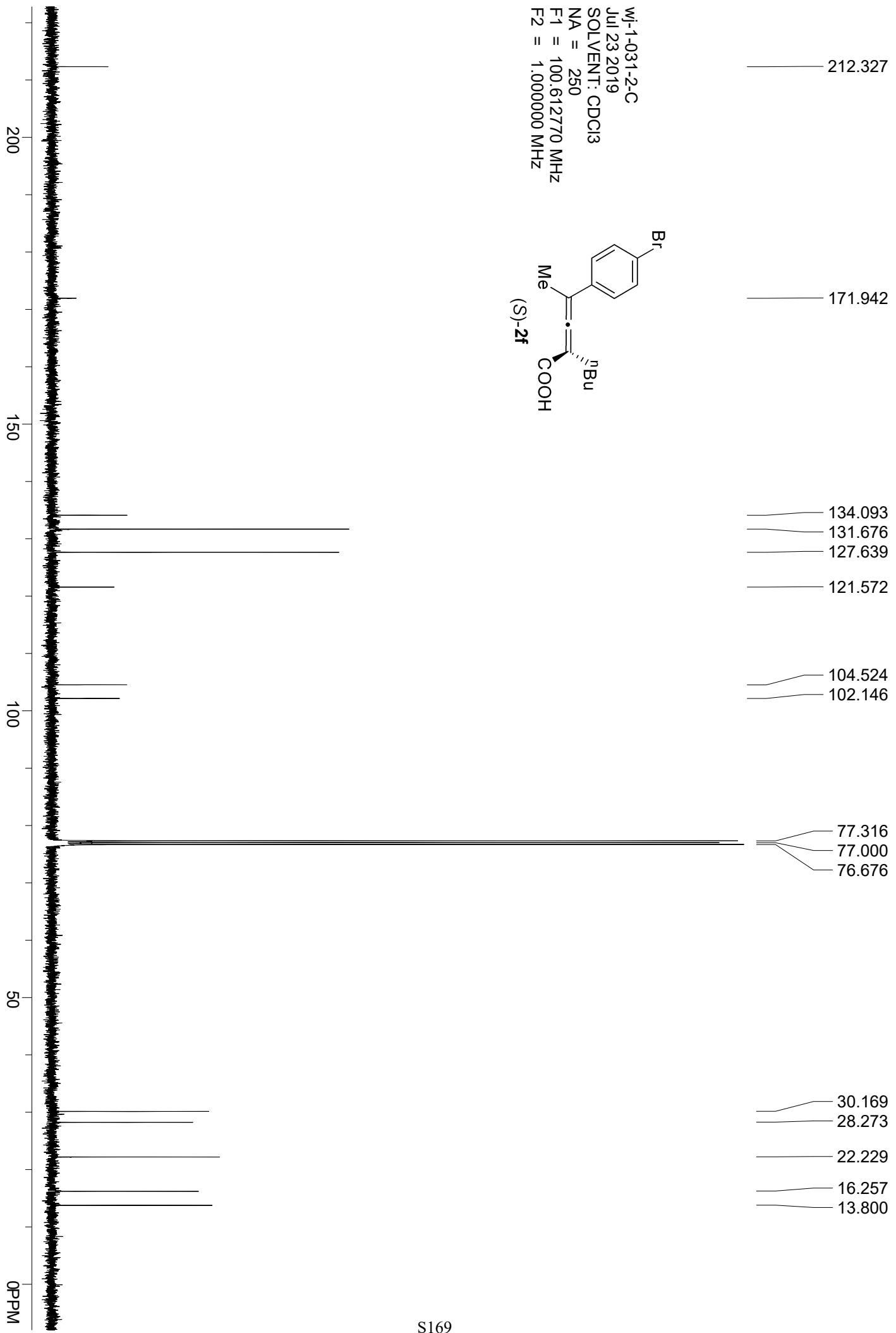


Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
9.341	0.4286	164.6044	4861.3979	49.8970
11.818	0.6826	104.5655	4881.4761	50.1030
Sum			9742.8740	100.0000

wj-1-031-12-H
Jul 23 2019
SOLVENT: CDCl₃
NA = 4
F1 = 400.130005 MHz
F2 = 1.000000 MHz





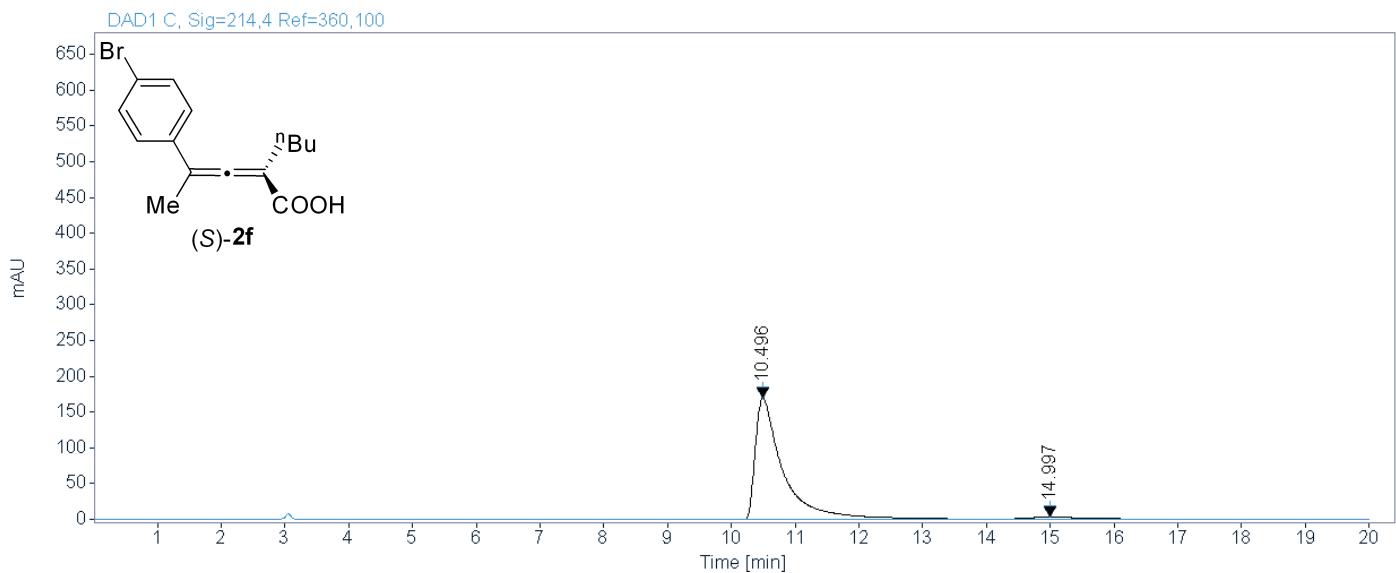
Area Percent Report



sample wj-1-031-2-AS-H-98-2-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\WJ_LC 2019-07-24 08-34-00\022-P2-C1-wj-1-031-2.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
10.496	0.4347	170.1617	5202.1748	96.5075
14.997	1.1338	2.7674	188.2598	3.4925
Sum		5390.4346	100.0000	

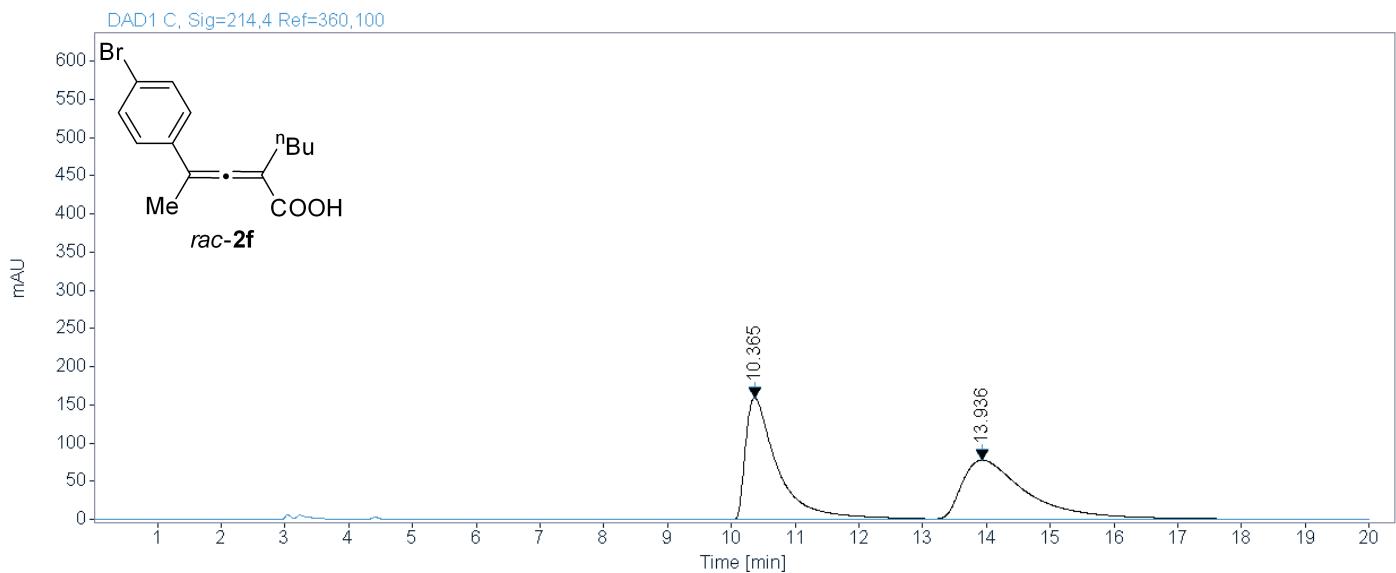
Area Percent Report



sample wj-1-031-2-rac-AS-H-98-2-1.0-214

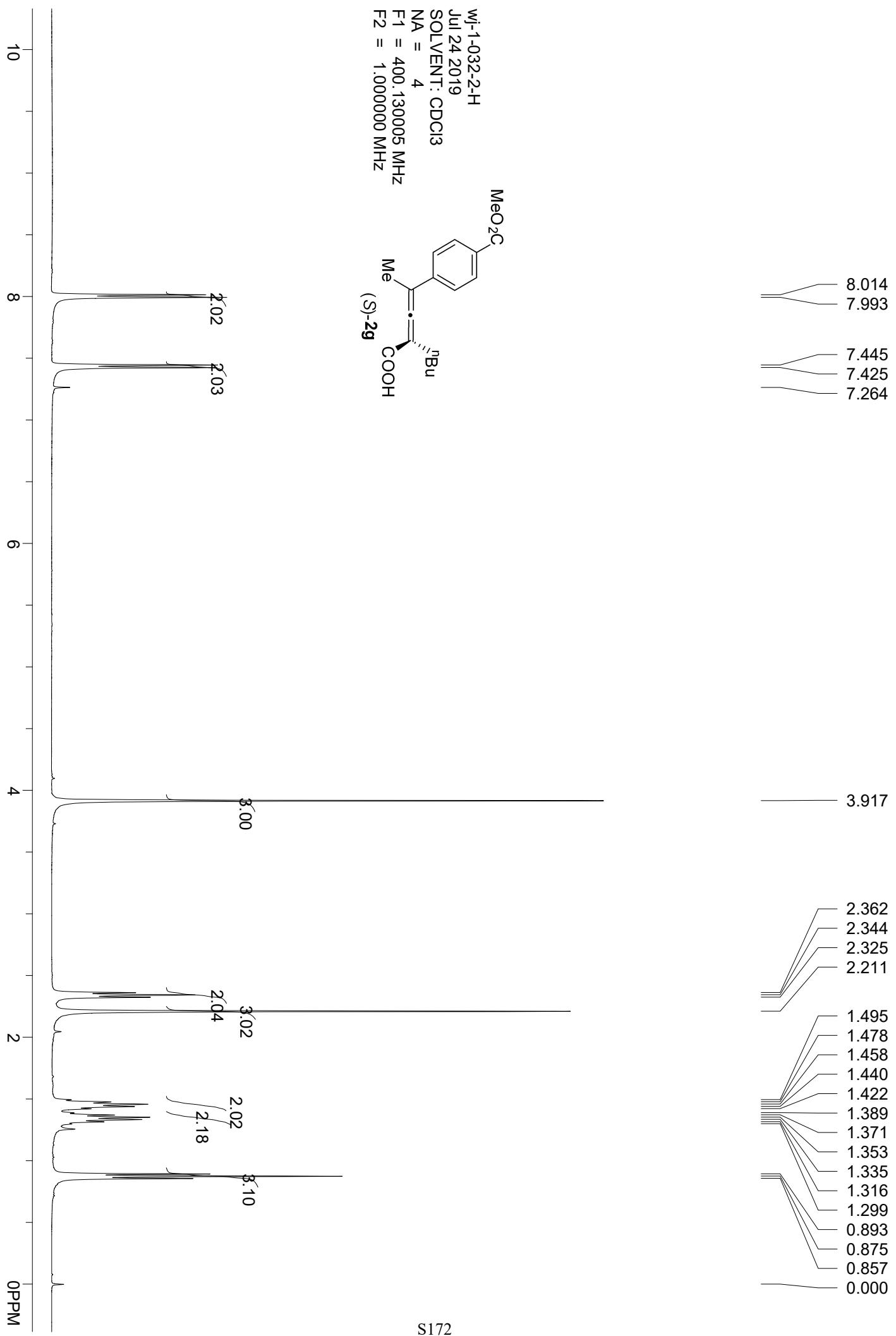
Data file: C:\Users\Public\Documents\ChemStation\1\Data\WJ_LC 2019-07-24 08-34-00\023-P2-C2-wj-1-031-2-rac.D

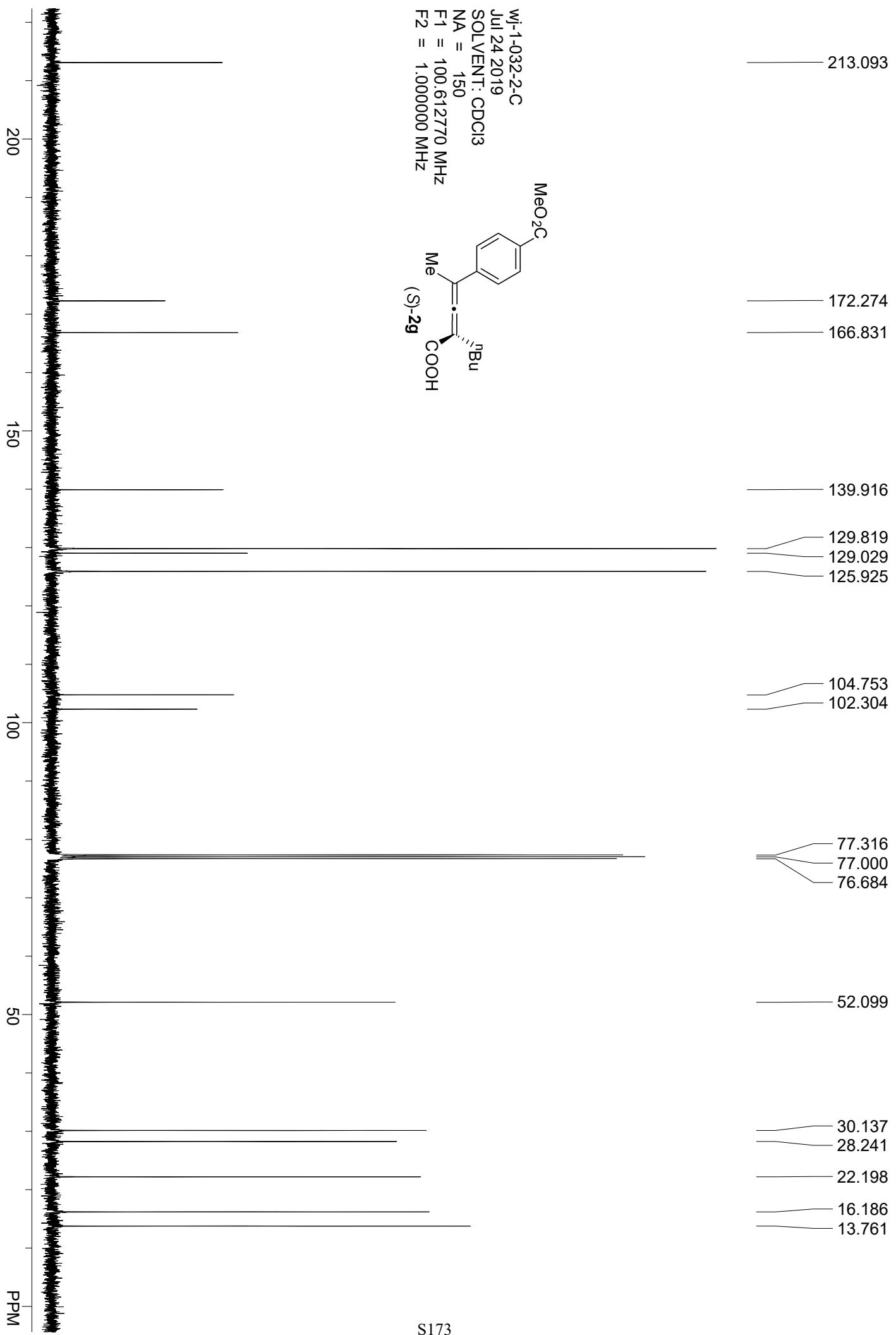
Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
10.365	0.5807	159.1867	5546.7344	50.4681
13.936	1.0391	77.1657	5443.8462	49.5319
Sum		10990.5806	100.0000	





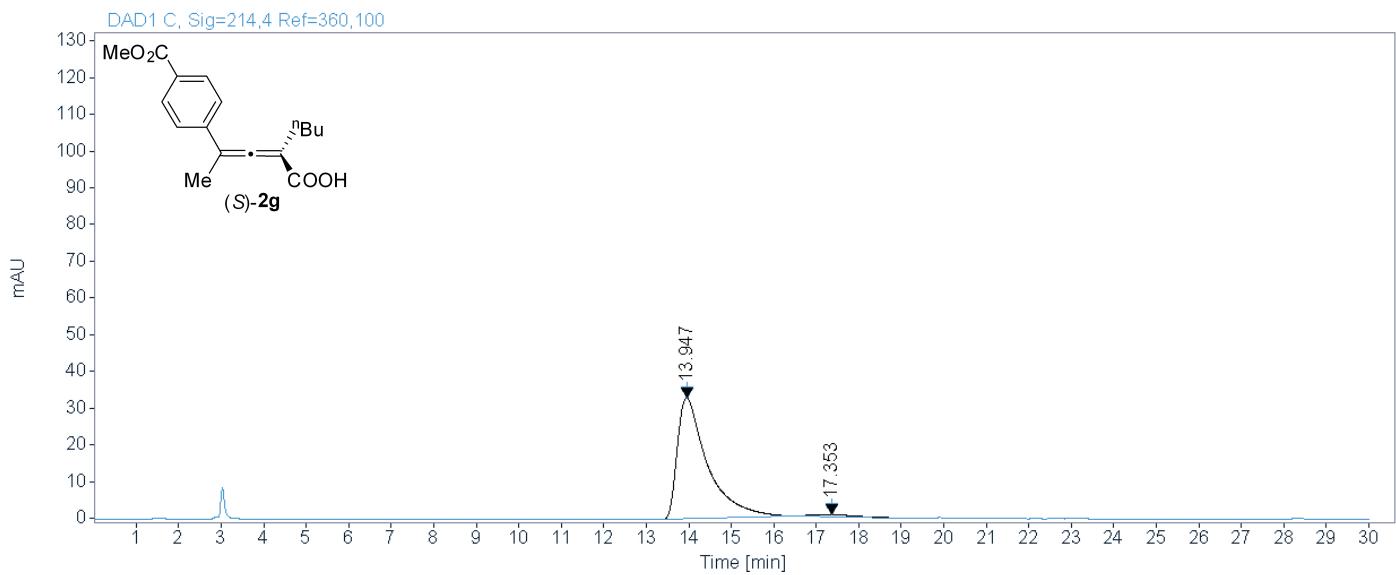
Area Percent Report



sample wj-1-032-2-AS-H-95-5-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\WJ_LC 2019-07-24 08-34-00\028-P2-C5-wj-1-032-2.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
13.947	0.7116	32.9281	1613.4130	97.8007
17.353	1.1921	0.5072	36.2815	2.1993
Sum		1649.6944	100.0000	

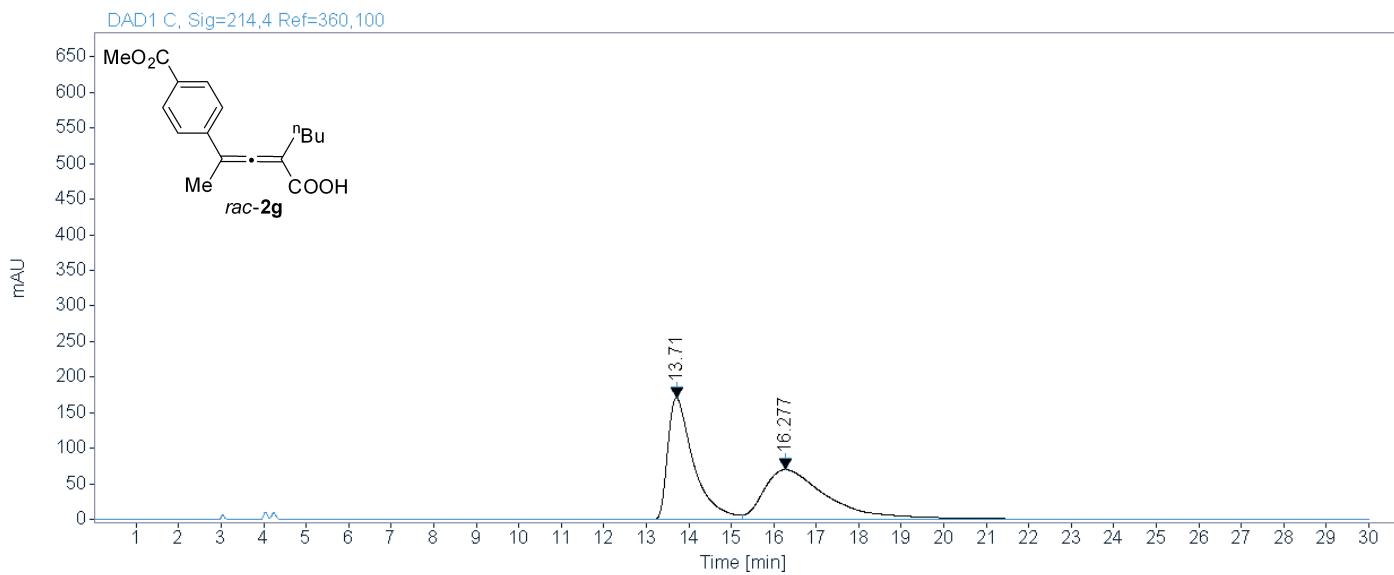
Area Percent Report



sample wj-1-032-2-rac-AS-H-95-5-1.0-214

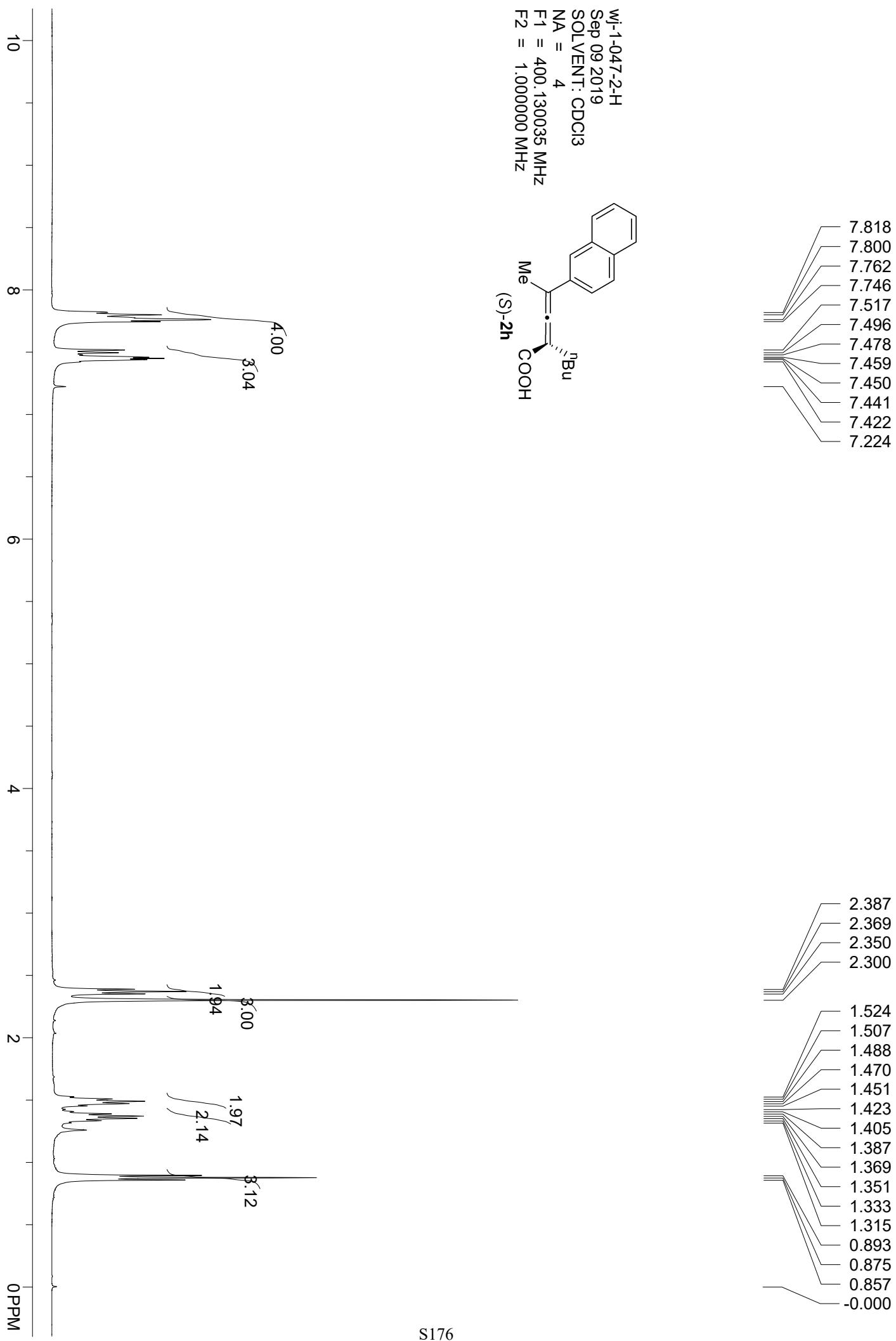
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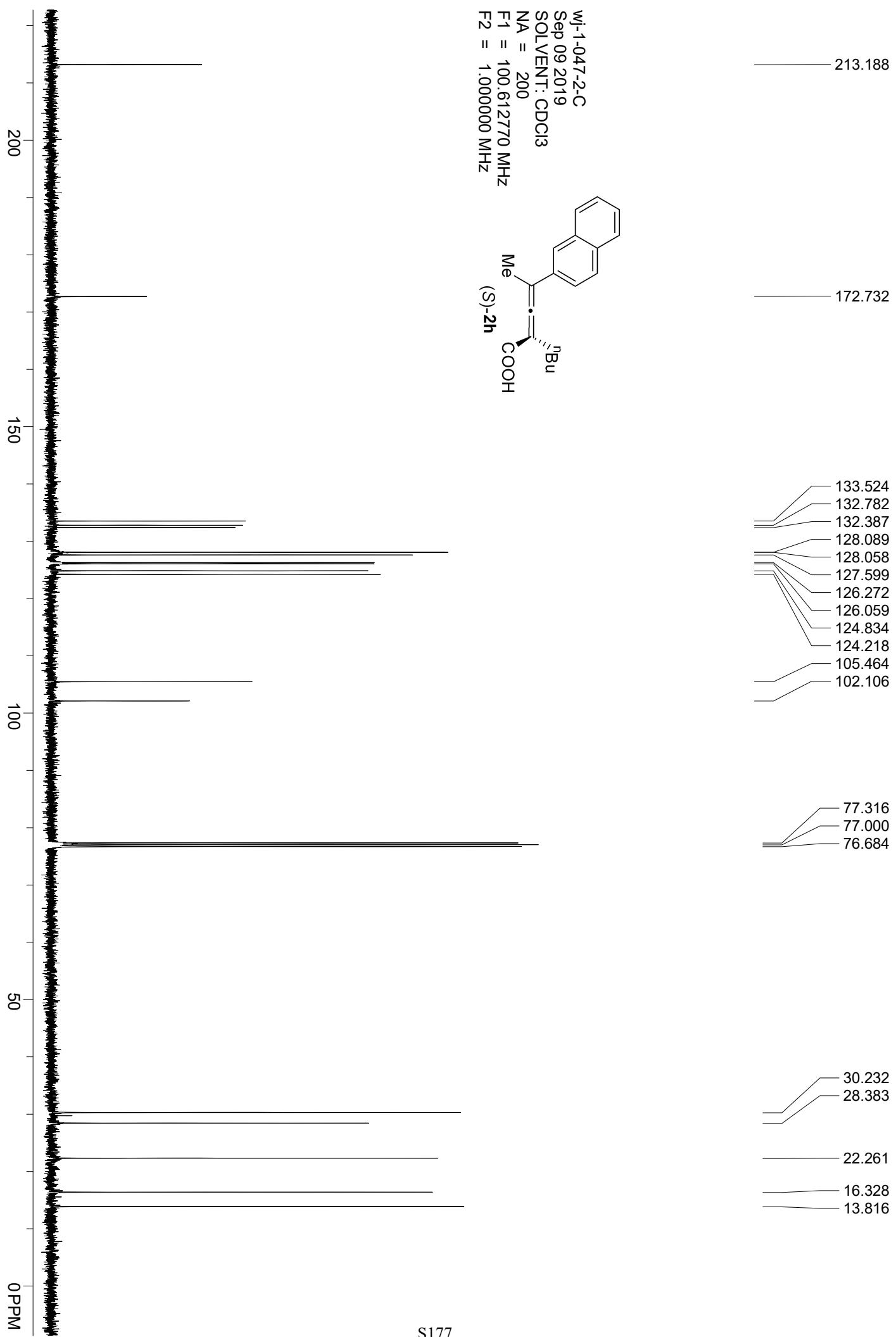
Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
13.710	0.6978	170.9039	7155.1748	49.7312
16.277	1.7256	69.8552	7232.5112	50.2688
Sum		14387.6860	100.0000	





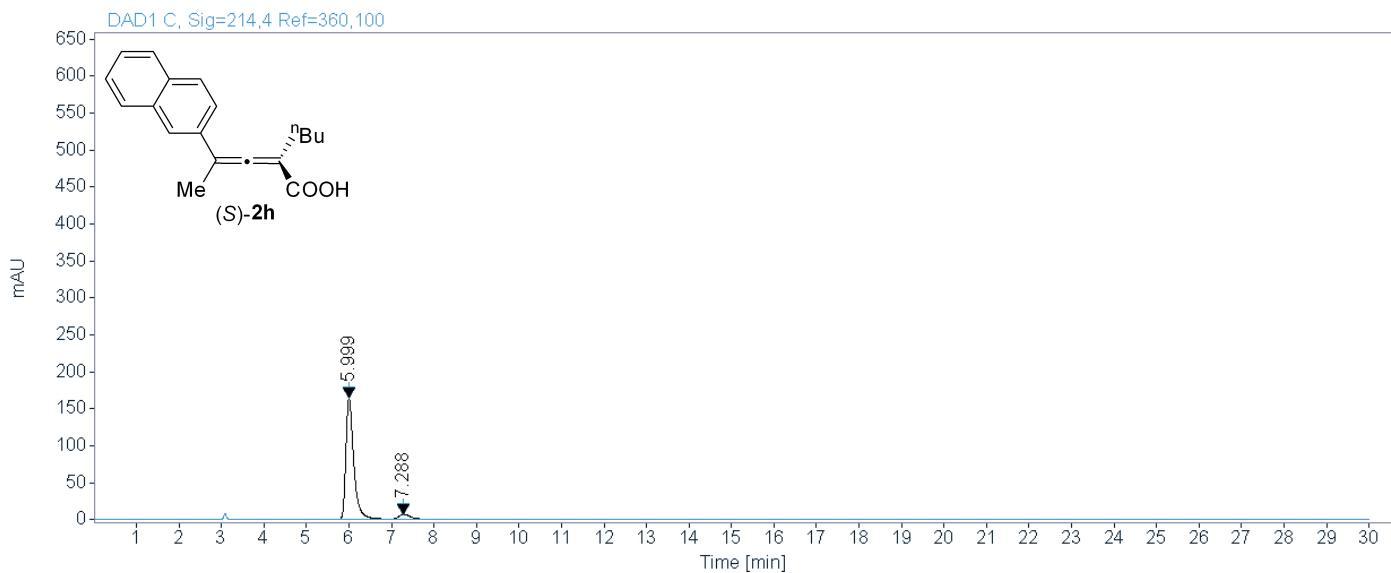
Area Percent Report



sample wj-1-047-2-AS-H-95-5-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\zwf-allenioic acid_LC 2019-09-09 16-35-10\036-P2-C7-wj-1-047-2.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
5.999	0.1945	164.5141	2122.4004	94.9958
7.288	0.2962	6.2918	111.8043	5.0042
		Sum	2234.2047	100.0000

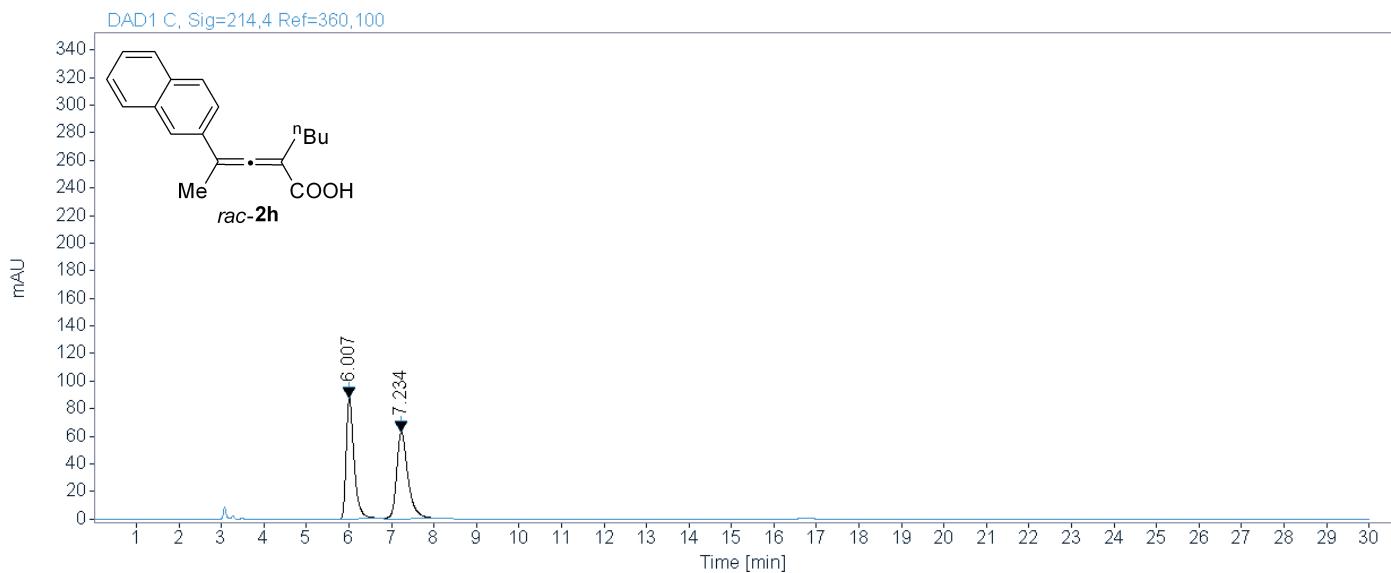
Area Percent Report



sample wj-1-047-2-rac-AS-H-95-5-1.0-214

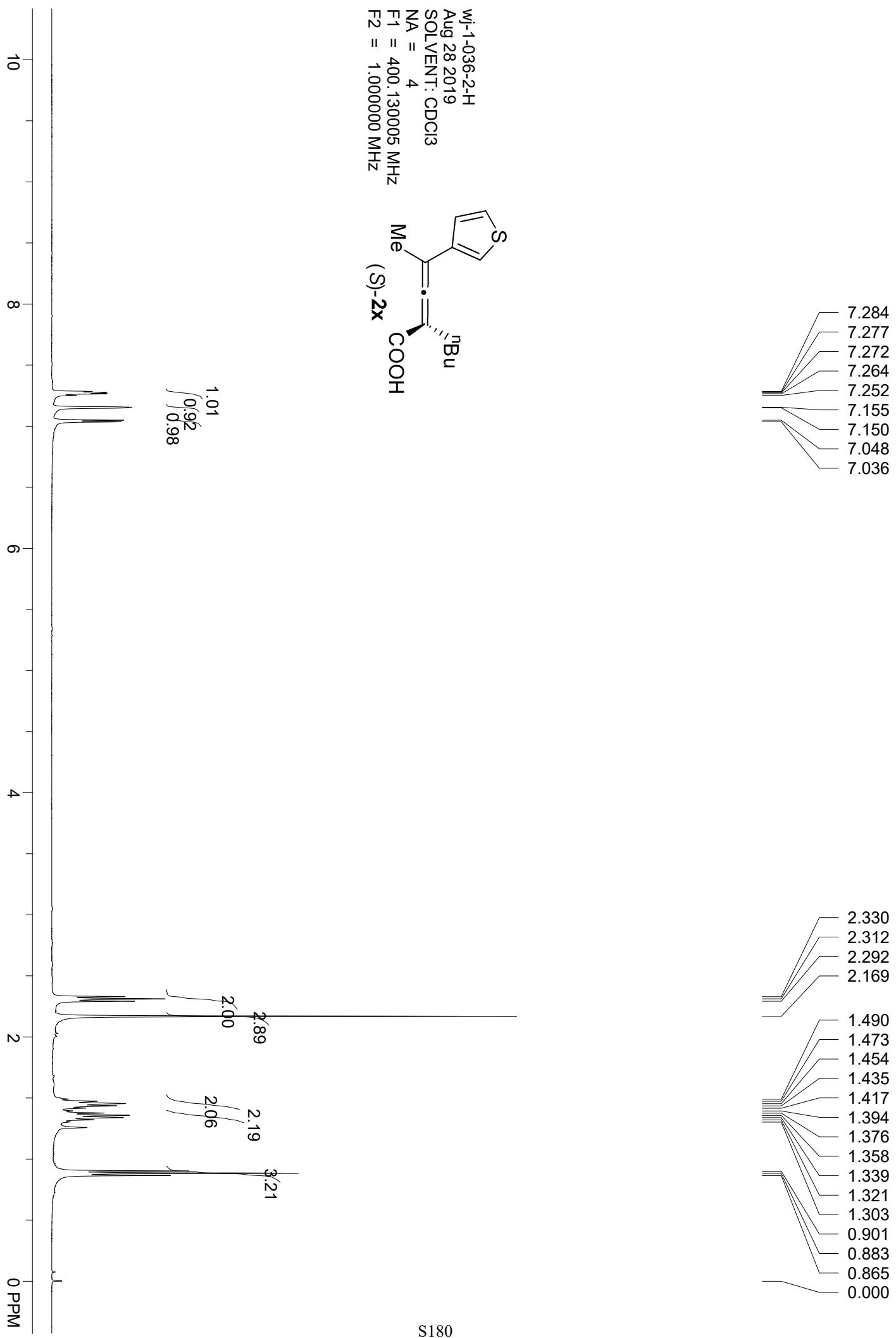
Data file: C:\Users\Public\Documents\ChemStation\1\Data\zwf-allenioic acid_LC 2019-09-09 16-35-10\037-P2-C8-wj-1-047-2-rac.D

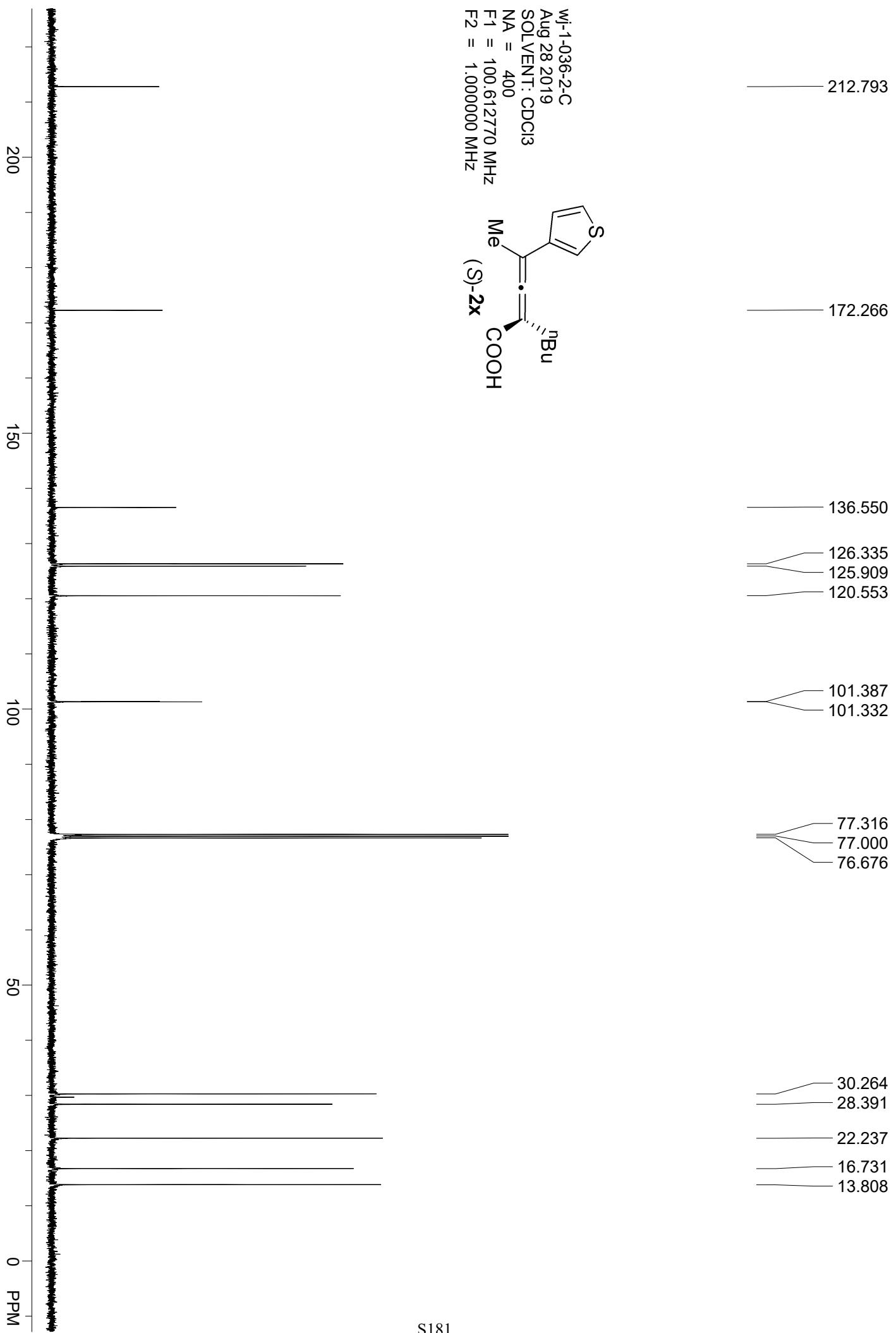
Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
6.007	0.2178	88.2336	1152.8596	49.5780
7.234	0.3097	63.0879	1172.4857	50.4220
		Sum	2325.3453	100.0000





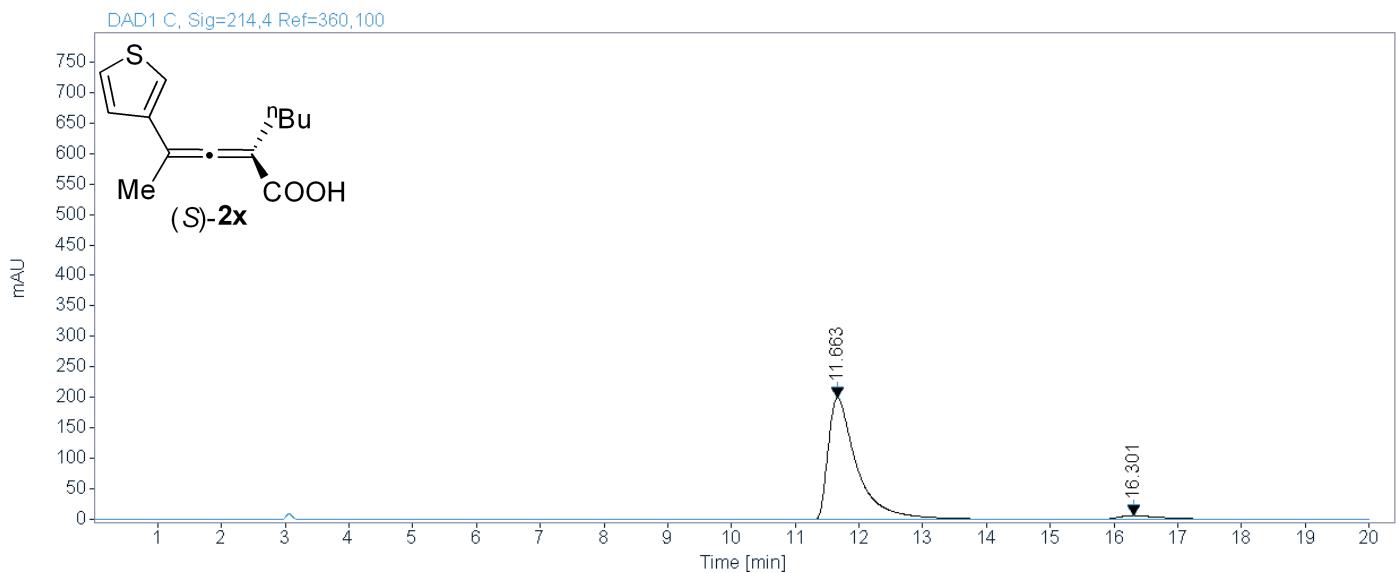
Area Percent Report



sample wj-1-036-2-AS-H-98-2-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\WJ_LC 2019-08-31 15-28-15\007-P2-C4-wj-1-036-2.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
11.663	0.5359	200.1053	6433.8291	95.7610
16.301	0.7643	6.2105	284.8026	4.2390
		Sum	6718.6317	100.0000

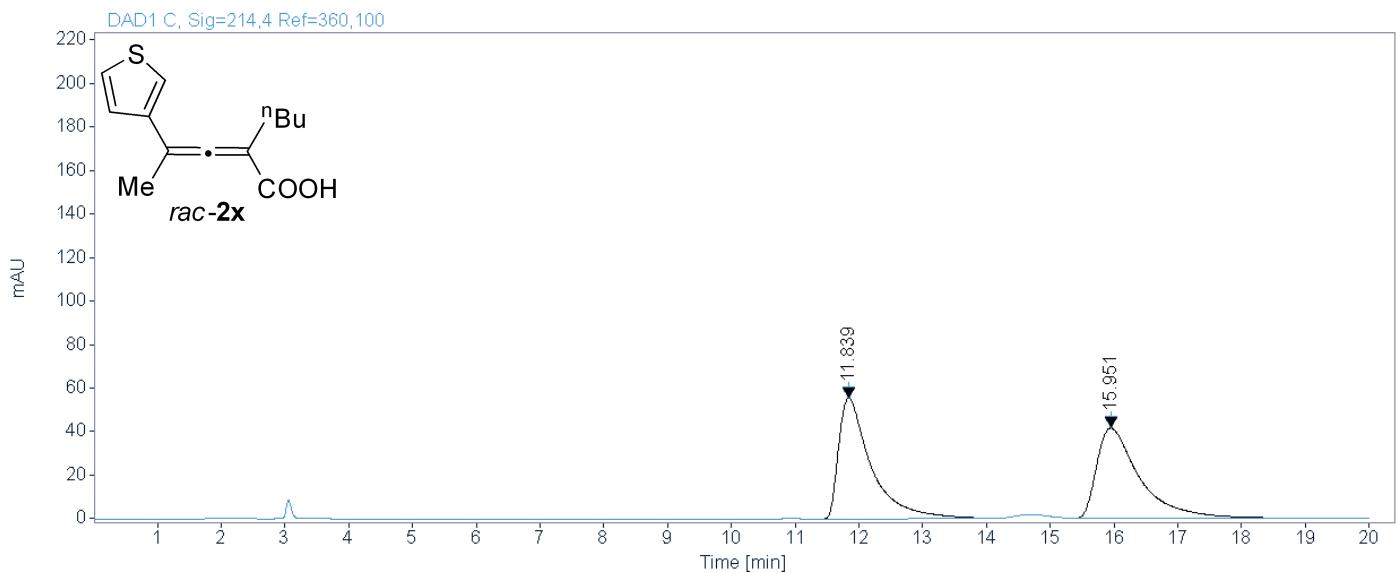
Area Percent Report



sample wj-1-036-2-rac-AS-H-98-2-1.0-214

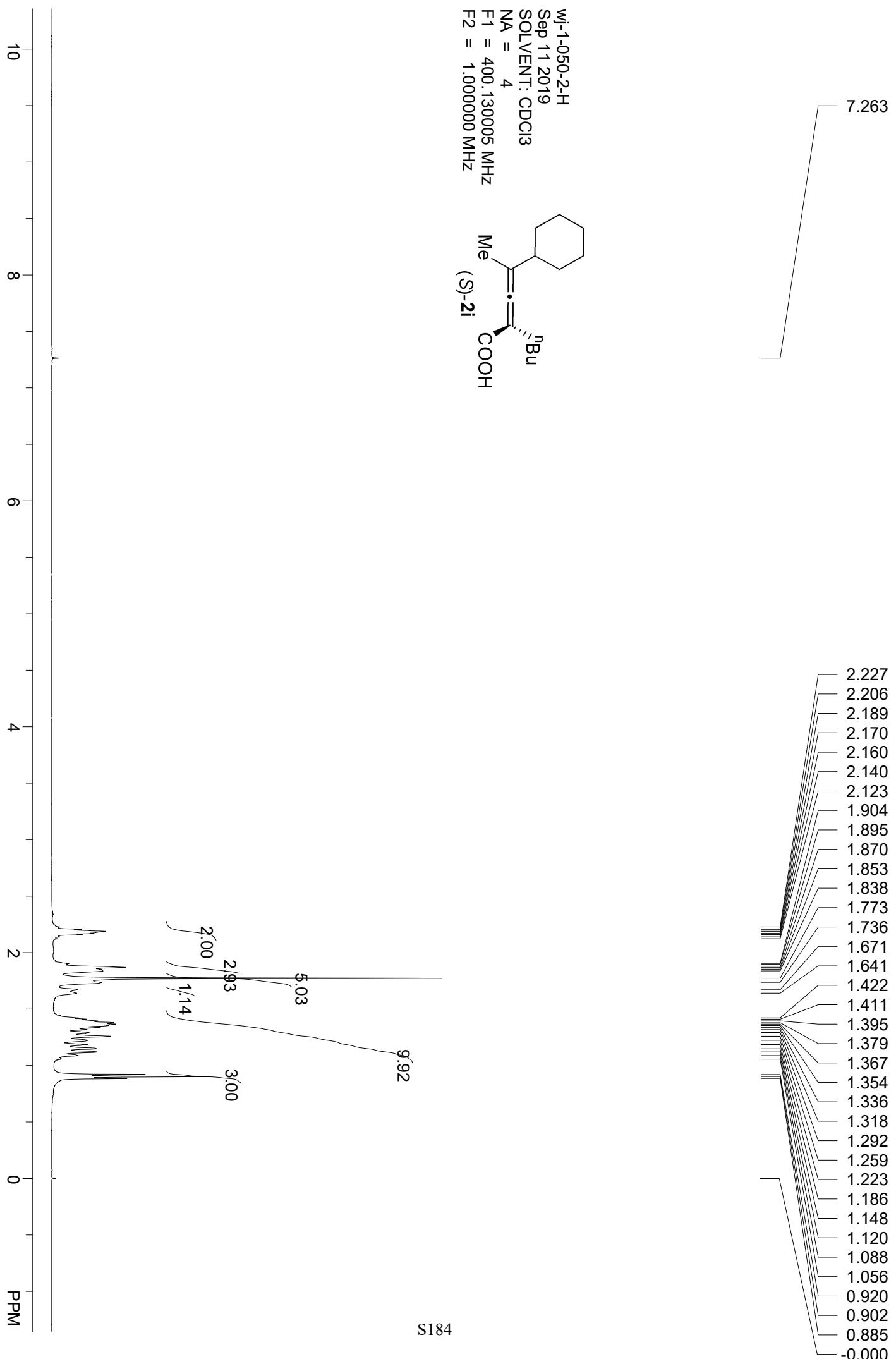
Data file: C:\Users\Public\Documents\ChemStation\1\Data\WJ_LC 2019-08-31 15-28-15\008-P2-C5-wj-1-036-2-rac.D

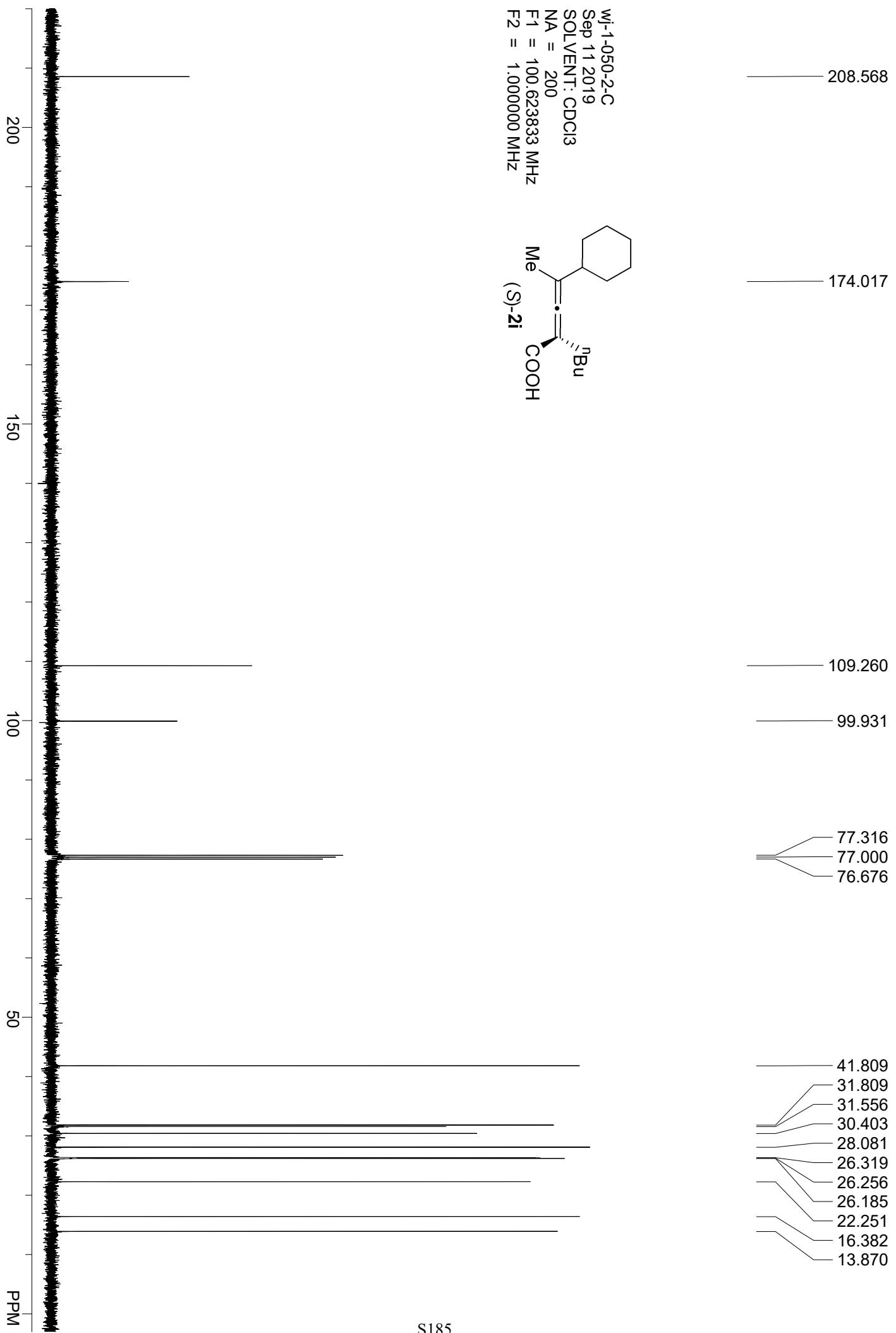
Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
11.839	0.5186	55.6528	1977.6501	50.1687
15.951	0.7023	41.4911	1964.3499	49.8313
		Sum	3942.0000	100.0000





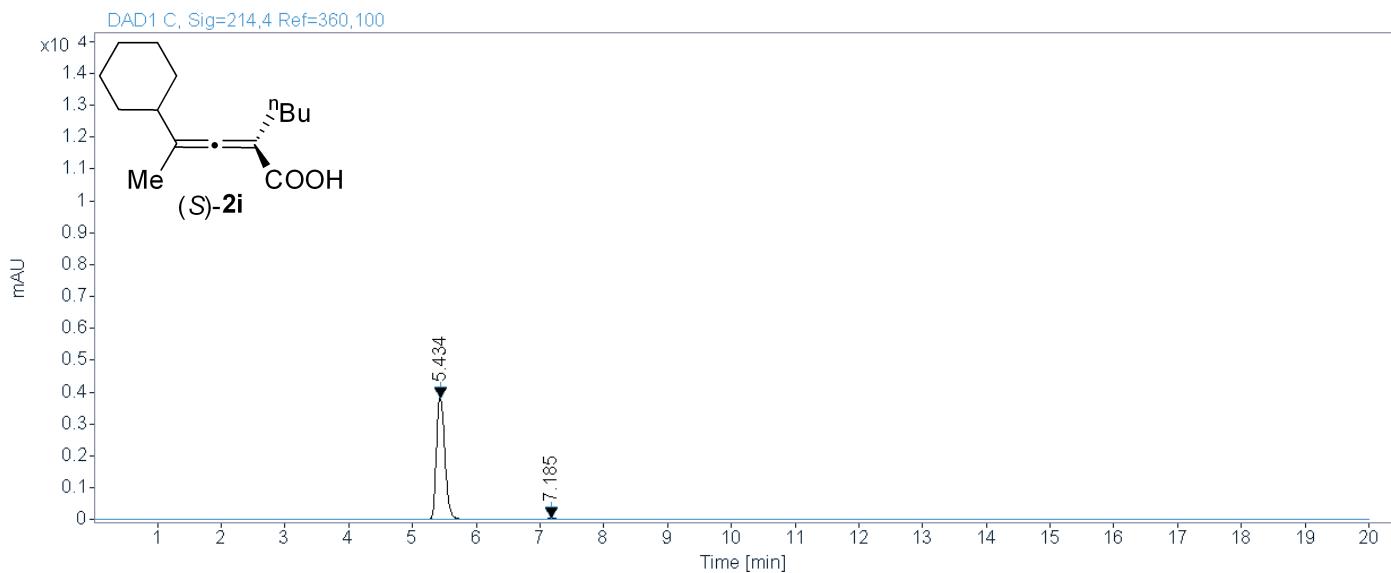
Area Percent Report



sample wj-1-050-2-AS-H-98-2-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\zwf-allenioc acid_LC 2019-09-11 17-12-23\012-P2-C3-wj-1-050-2.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
5.434	0.1454	3825.0781	35883.6367	98.5244
7.185	0.2418	37.0453	537.4363	1.4756
Sum		36421.0730	100.0000	

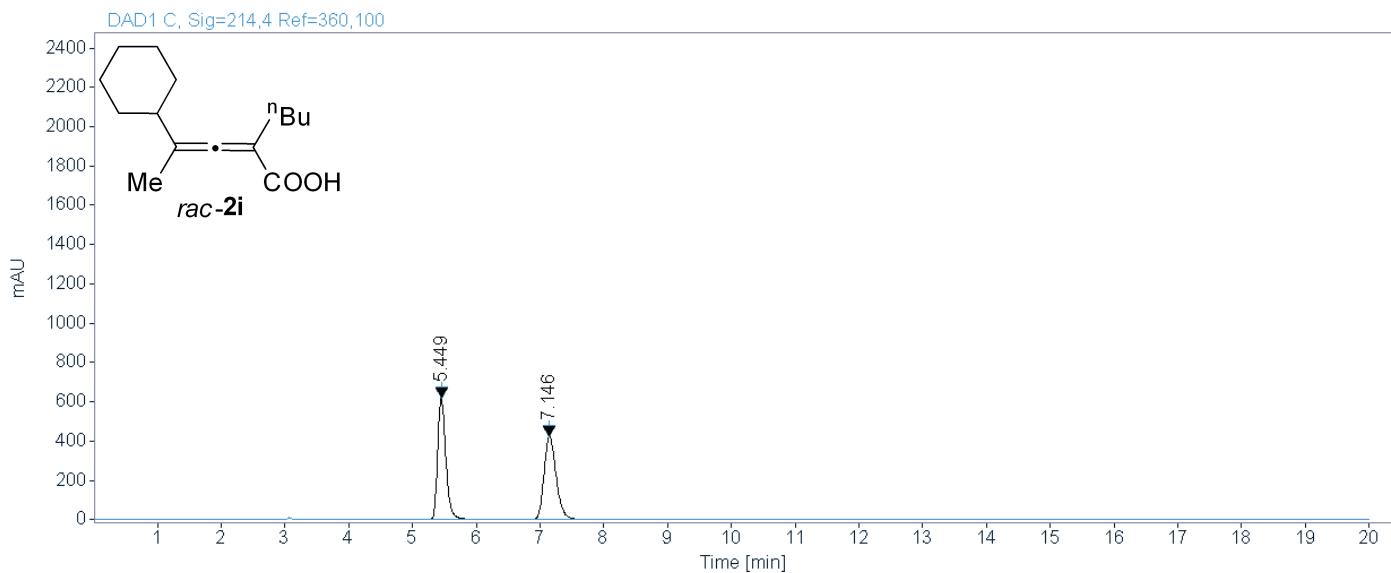
Area Percent Report



sample wj-1-050-2-rac-AS-H-98-2-1.0-214

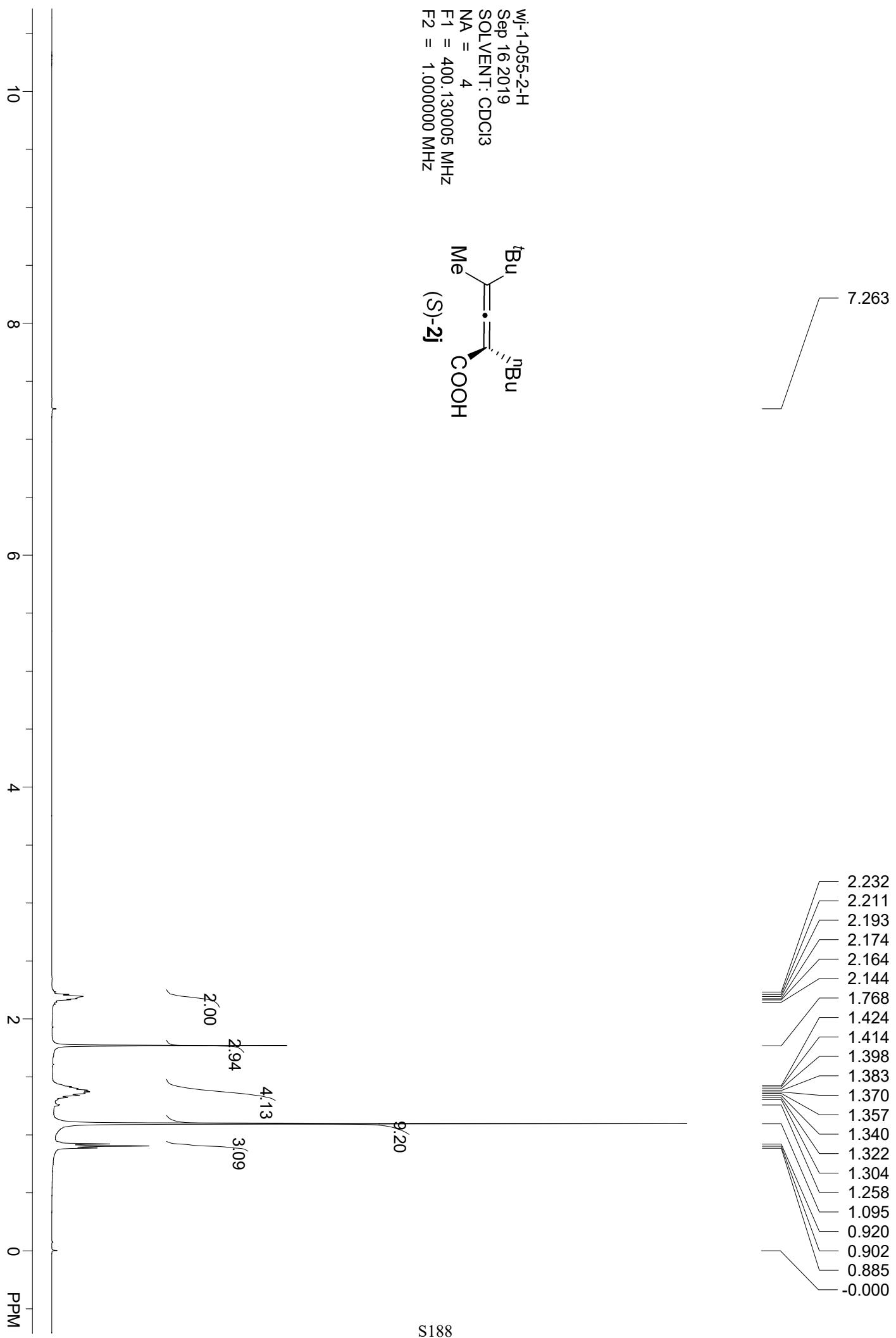
Data file: C:\Users\Public\Documents\ChemStation\1\Data\zwf-allenioic acid_LC 2019-09-11 17-12-23\013-P2-C4-wj-1-050-2-rac.D

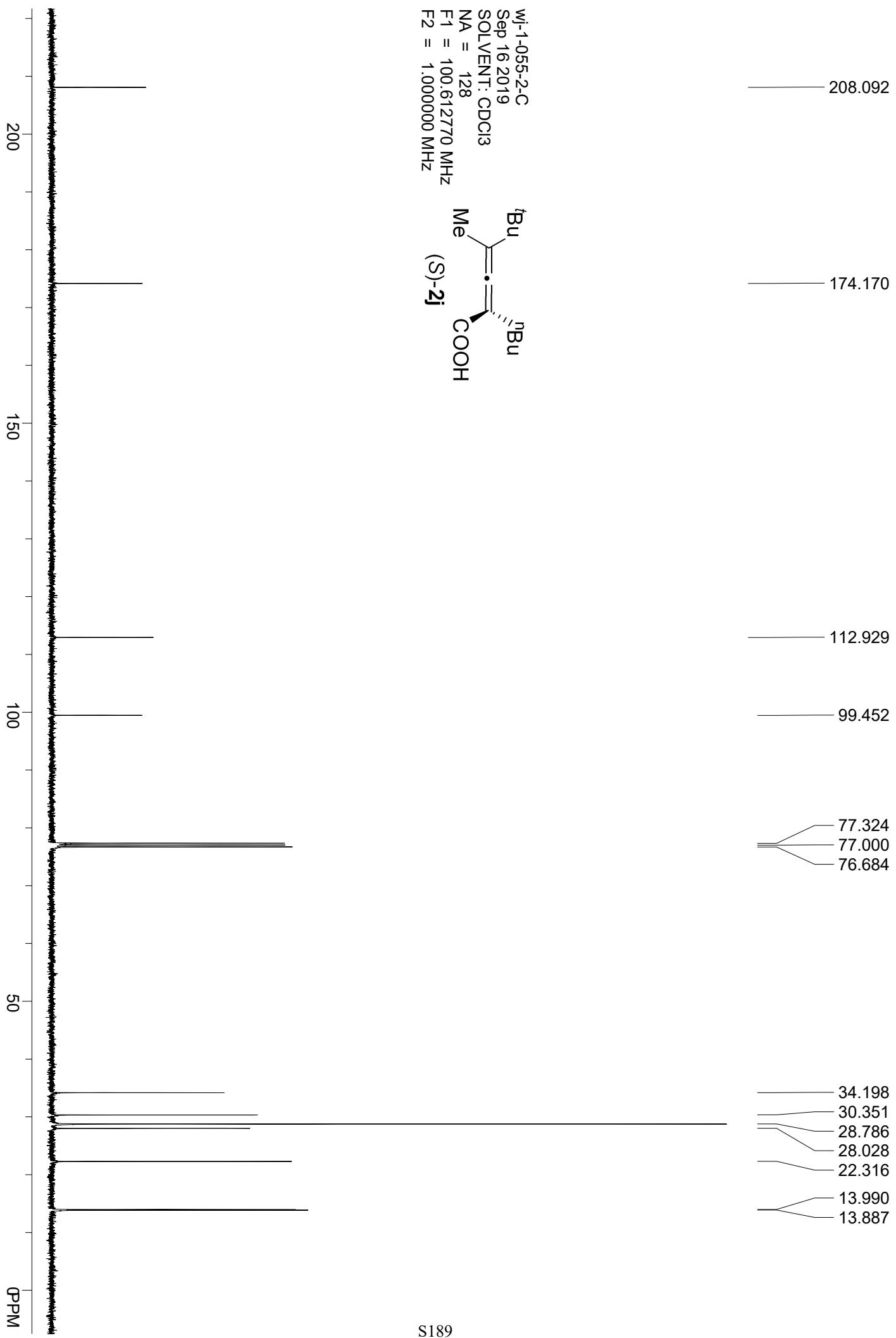
Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
5.449	0.1365	619.4649	5548.2568	49.8322
7.146	0.2035	424.4063	5585.6196	50.1678
		Sum	11133.8765	100.0000





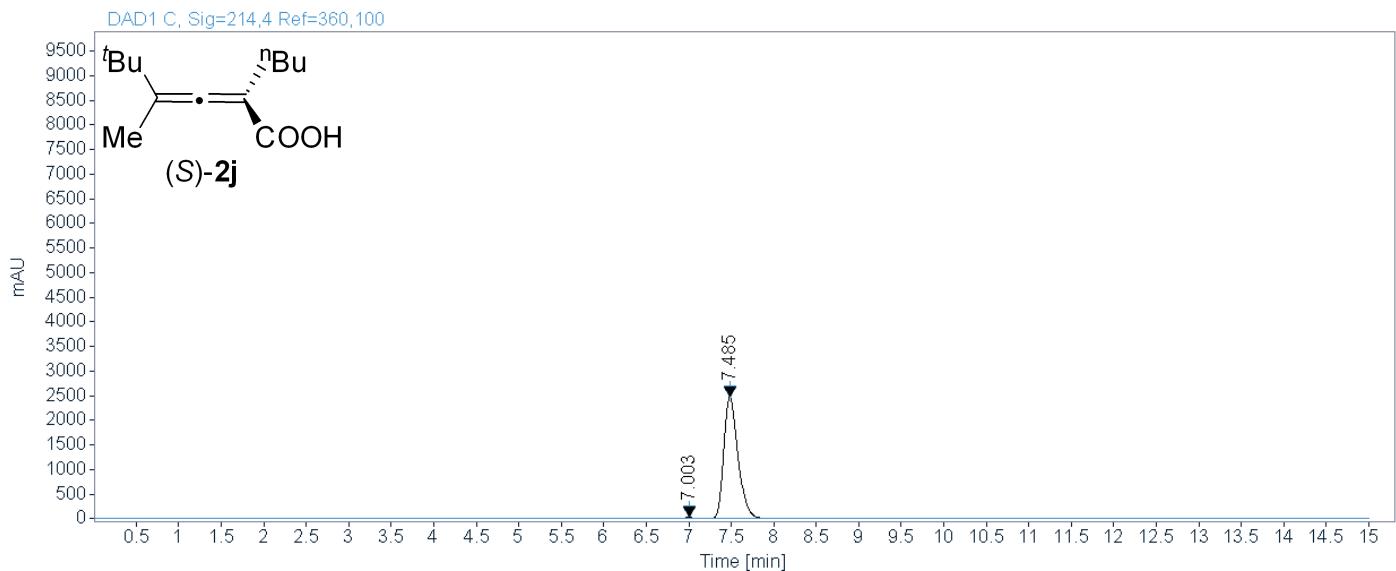
Area Percent Report



sample wj-1-055-2-AD-H-98-2-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\wgl 2019-09-17 09-02-49\034-P2-C7-wj-1-055-2.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
7.003	0.1652	22.3828	240.1547	0.8288
7.485	0.1773	2475.7034	28737.6777	99.1712
		Sum	28977.8324	100.0000

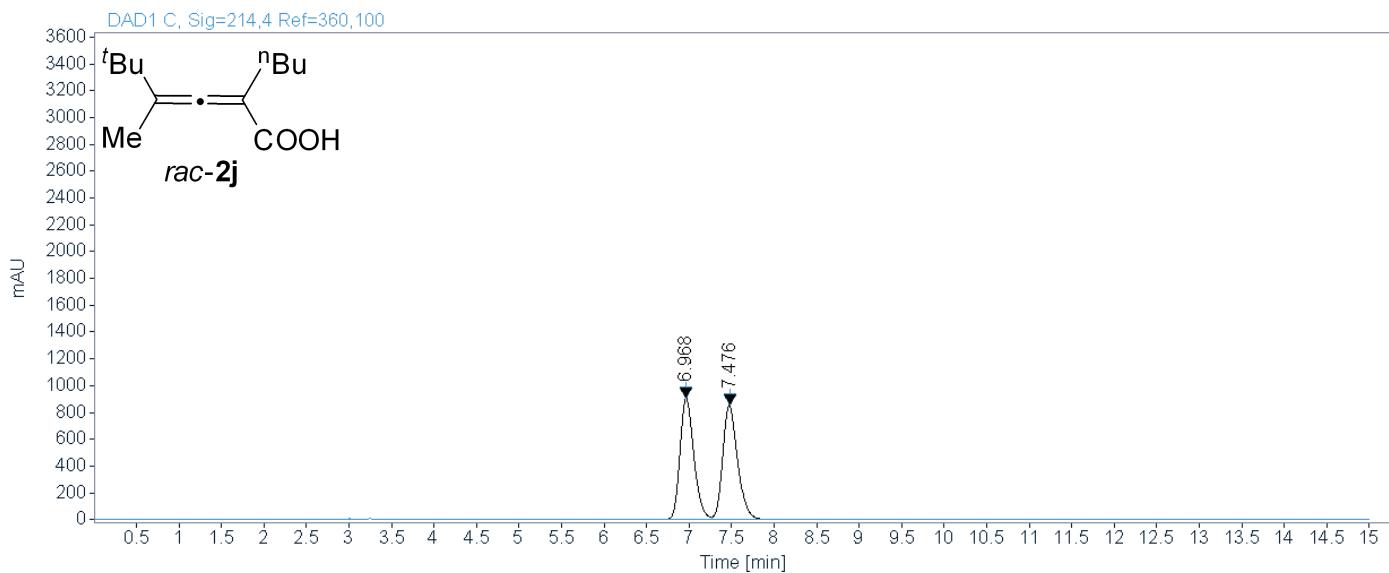
Area Percent Report



sample wj-1-055-2-rac-AD-H-98-2-1.0-214

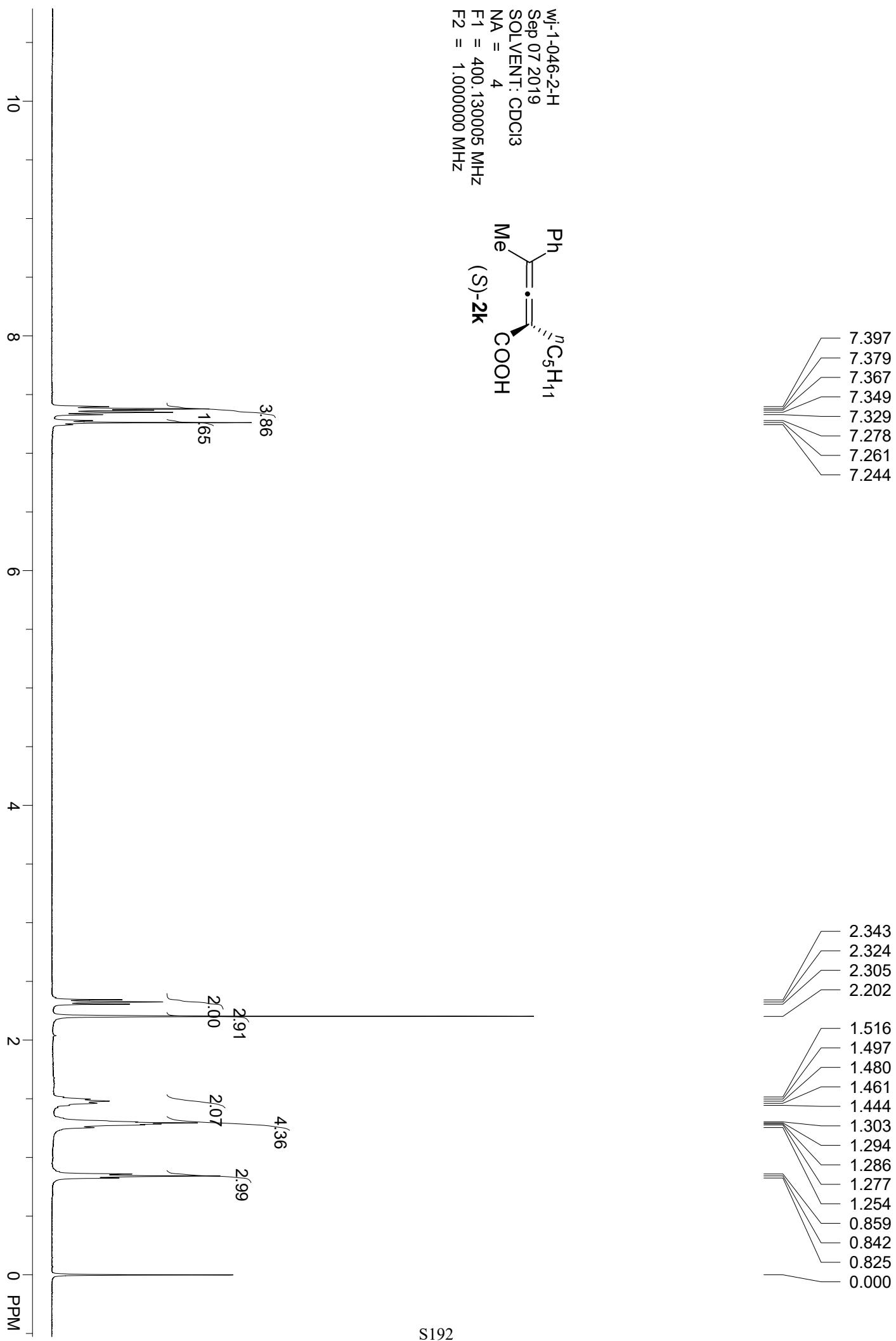
Data file: C:\Users\Public\Documents\ChemStation\1\Data\wgl 2019-09-17 09-02-49\035-P2-C8-wj-1-055-2-rac.D

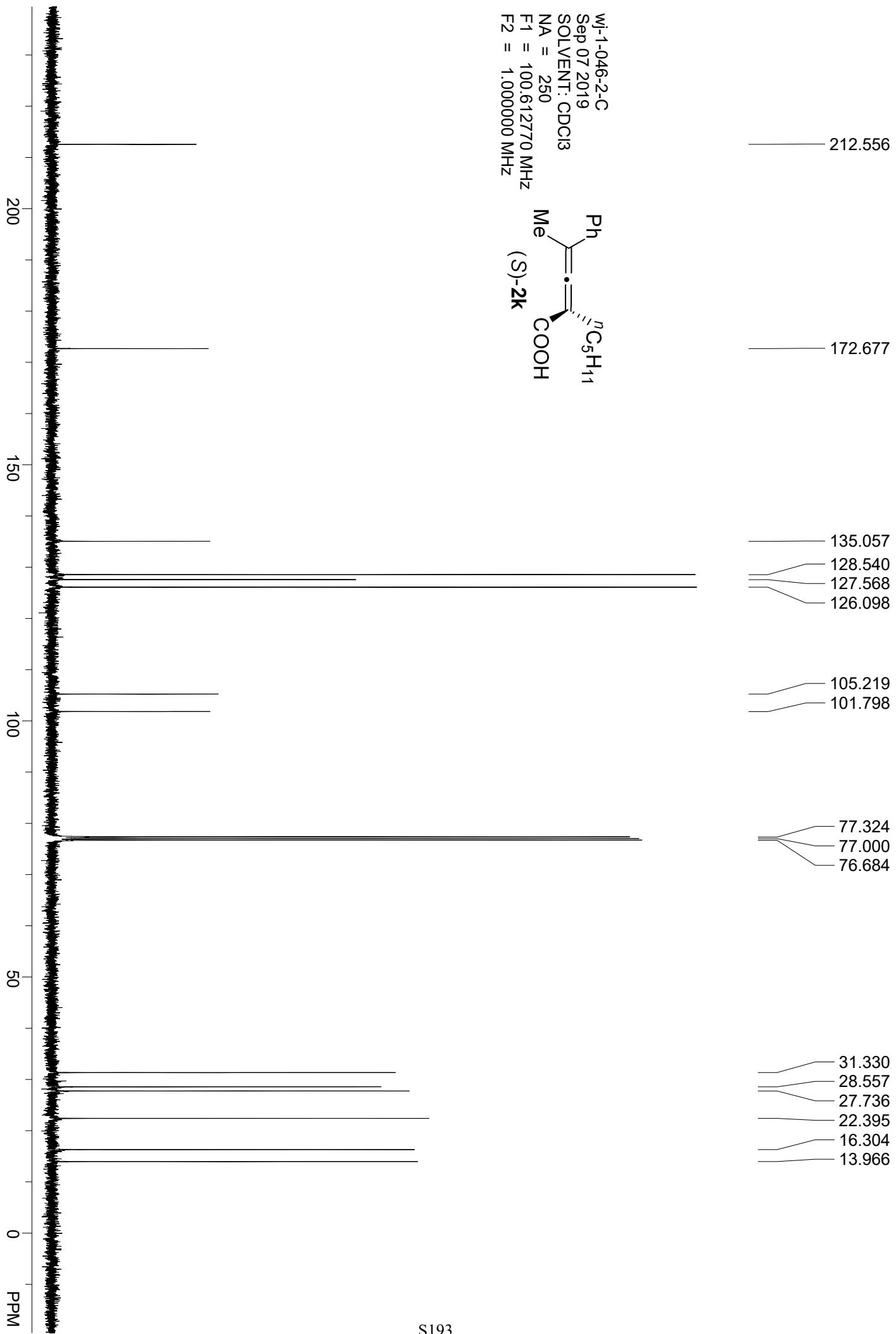
Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
6.968	0.1743	907.0036	10285.4258	49.8380
7.476	0.1880	850.8357	10352.2822	50.1620
		Sum	20637.7080	100.0000





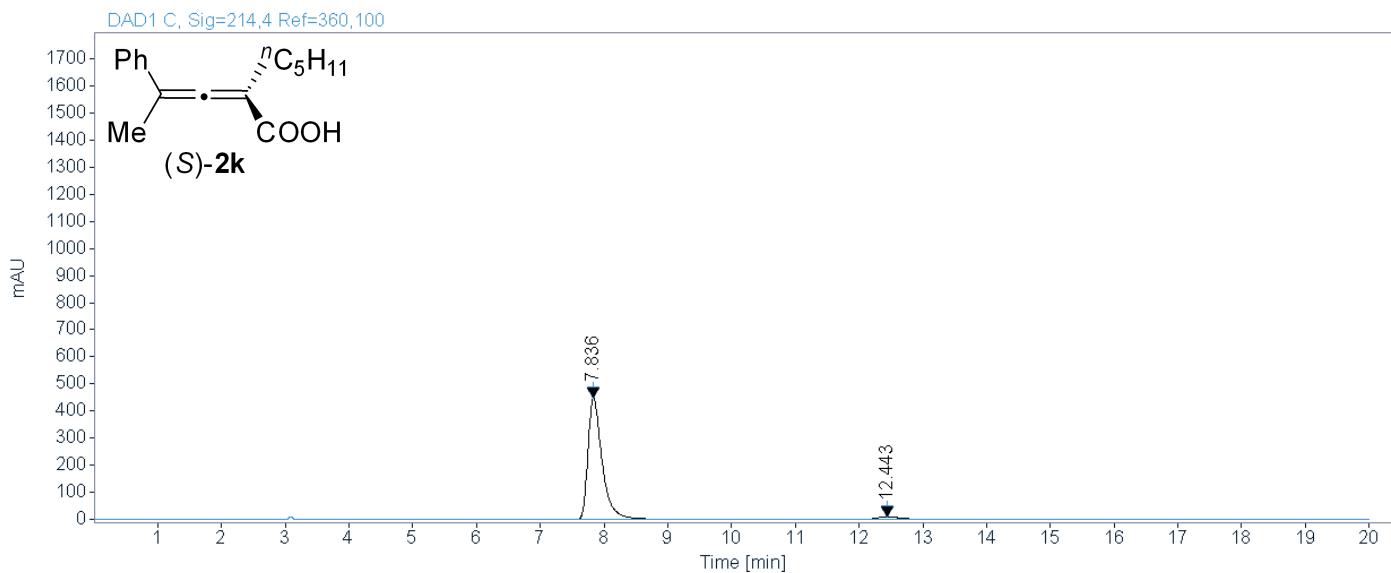
Area Percent Report



sample wj-1-046-2-AS-H-98-2-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\zwf-allenioic acid_LC 2019-09-09 16-35-10\031-P2-C3-wj-1-046-2.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
7.836	0.2358	449.1552	7014.2578	95.4400
12.443	0.4584	10.8478	335.1310	4.5600
Sum		460.0000	7349.3888	100.0000

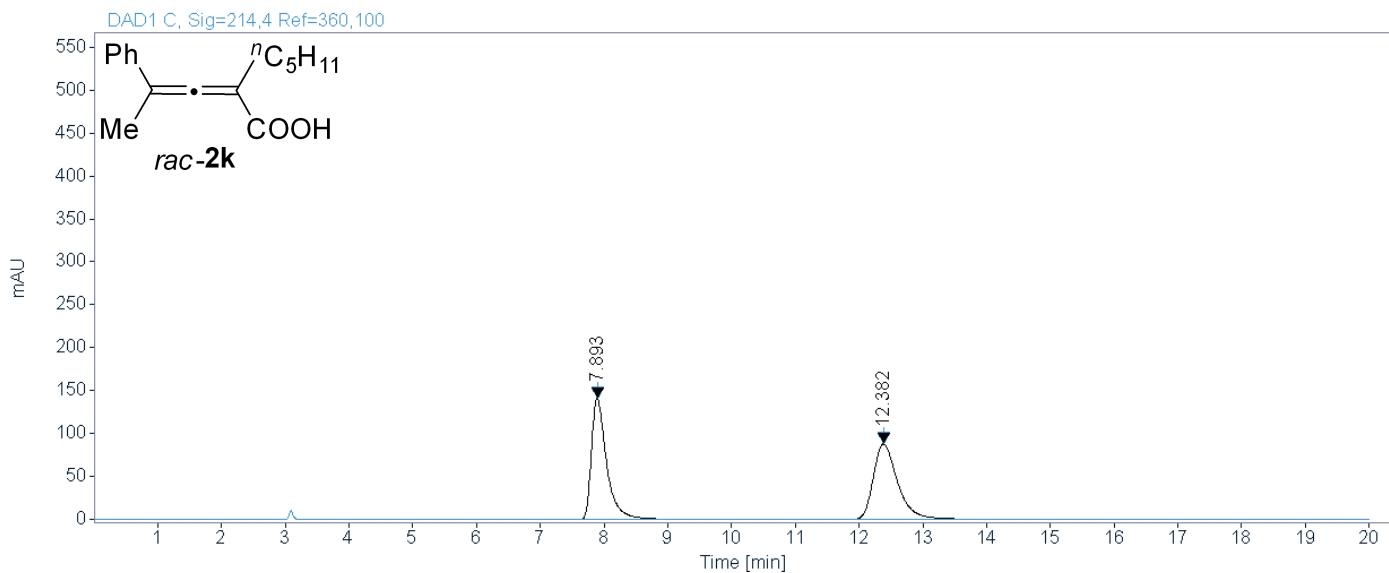
Area Percent Report



sample wj-1-046-2-rac-AS-H-98-2-1.0-214

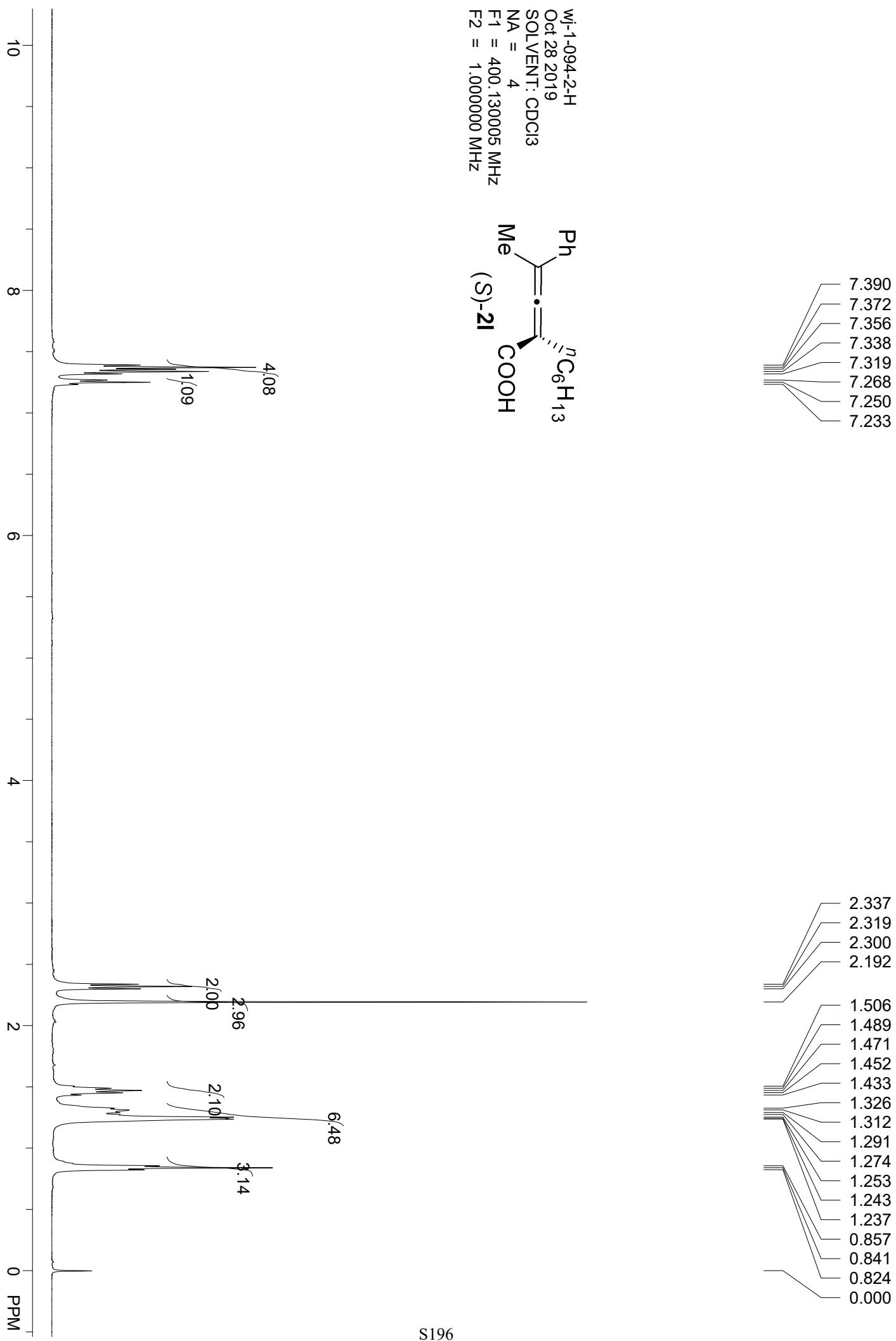
Data file: C:\Users\Public\Documents\ChemStation\1\Data\zwf-allenioic acid_LC 2019-09-09 16-35-10\032-P2-C4-wj-1-046-2-rac.D

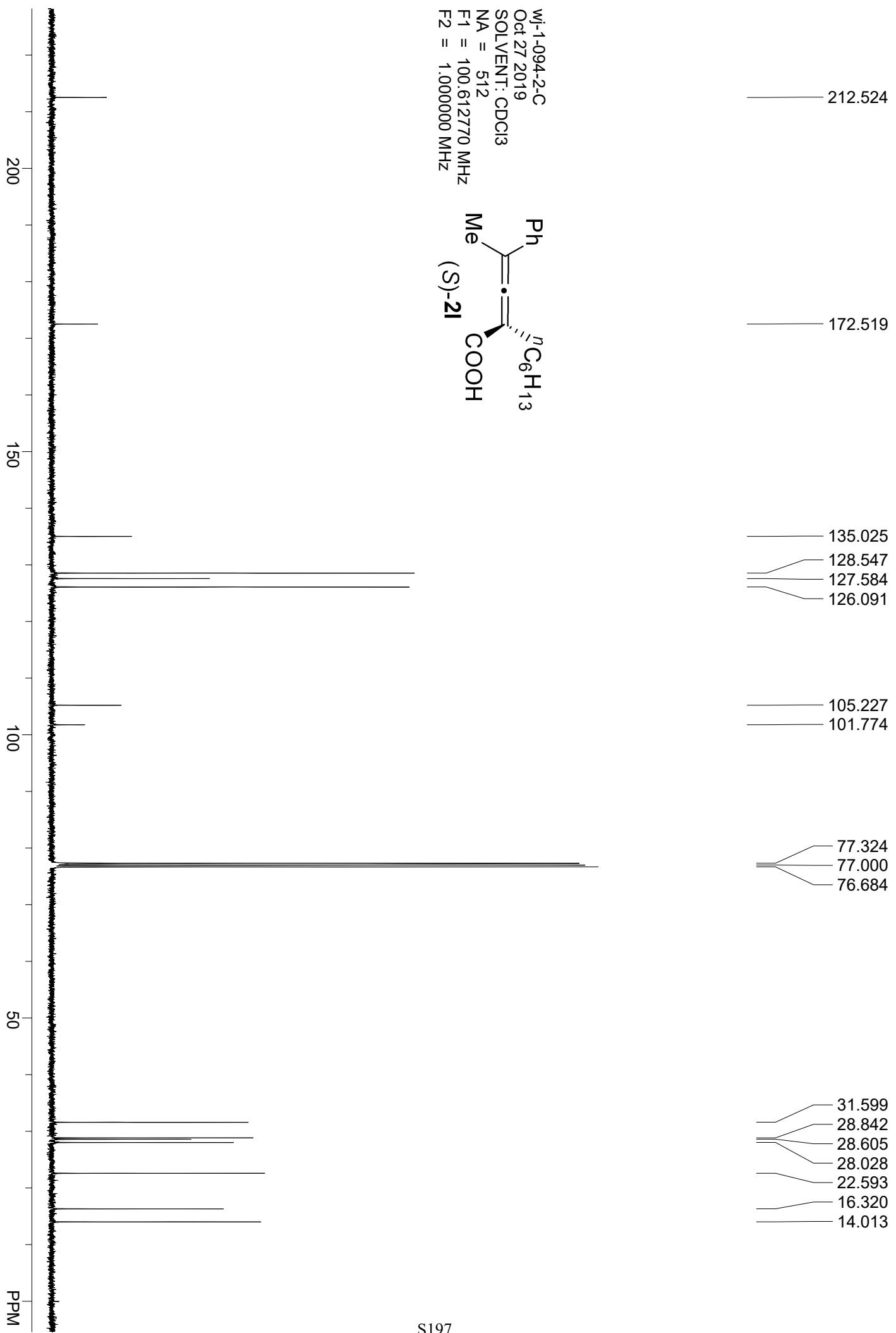
Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
7.893	0.2465	141.8907	2340.6135	50.0324
12.382	0.4040	87.9829	2337.5791	49.9676
Sum		4678.1926	100.0000	





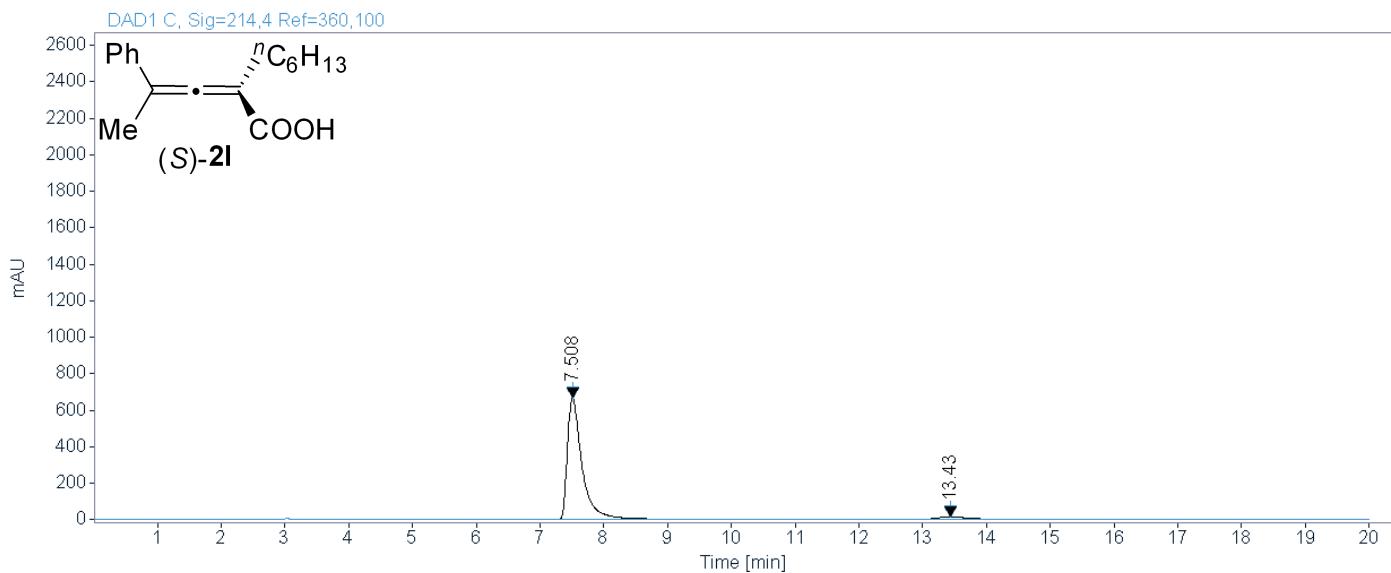
Area Percent Report



sample wj-1-094-2-AS-H-98-2-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\WJ_LC 2019-10-26 22-43-36\003-P2-C1-wj-1-094-2.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
7.508	0.2425	667.7341	10924.6270	94.8889
13.430	0.6316	15.5273	588.4440	5.1111
		Sum	11513.0709	100.0000

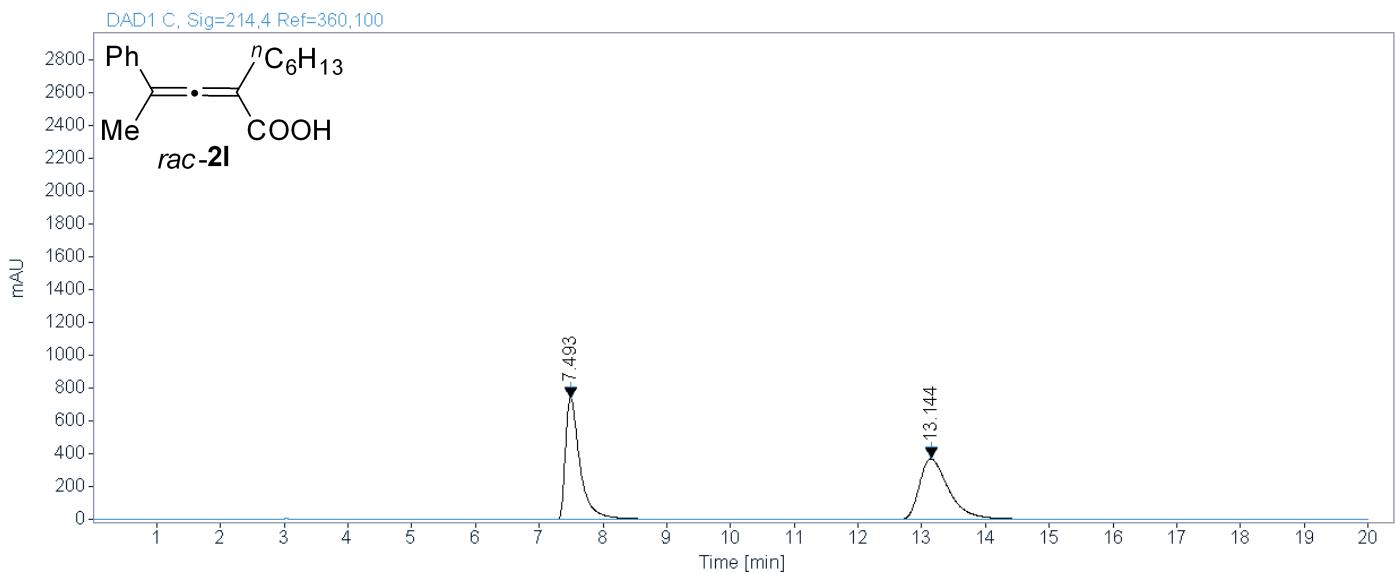
Area Percent Report



sample wj-1-094-2-rac-AS-H-98-2-1.0-214

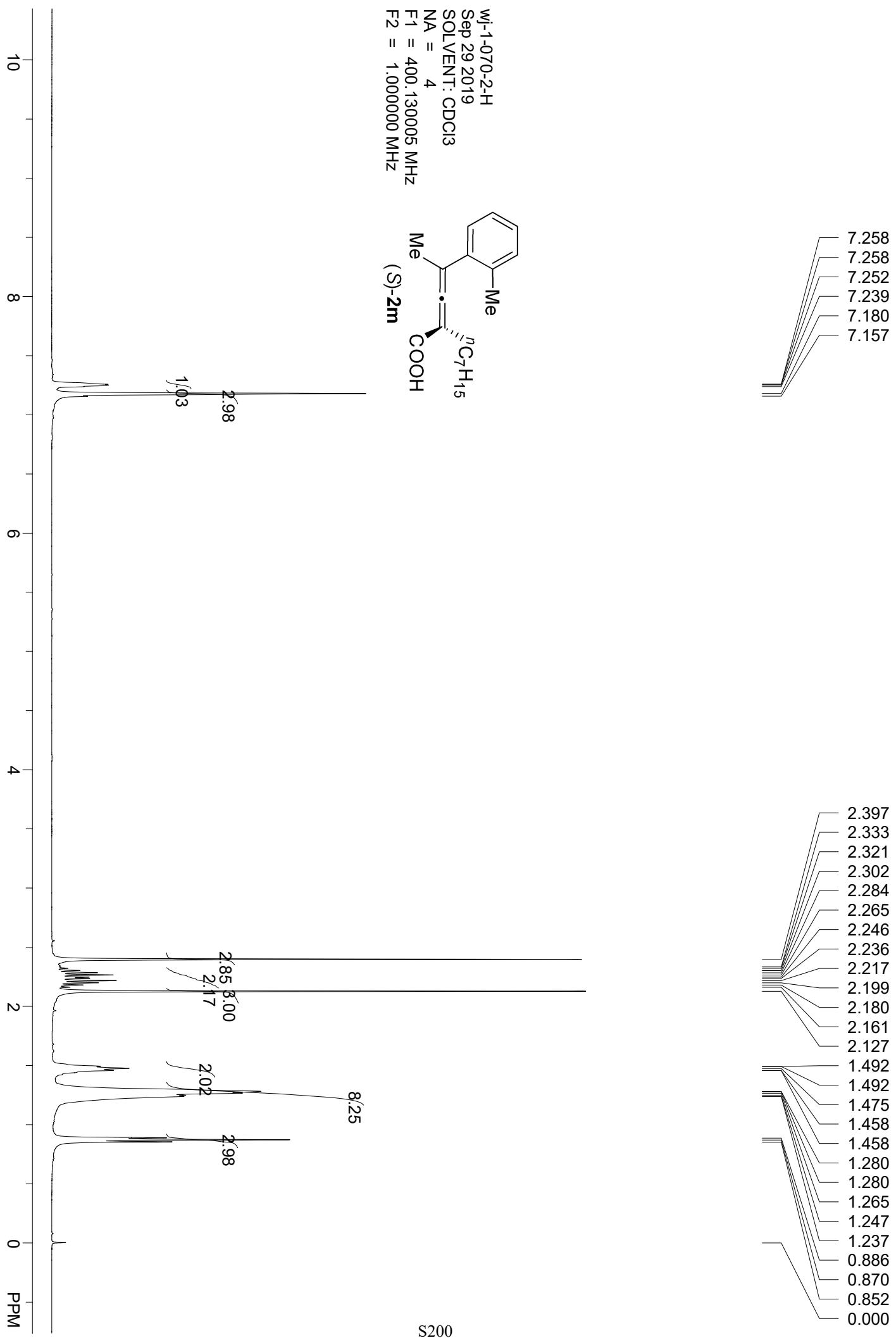
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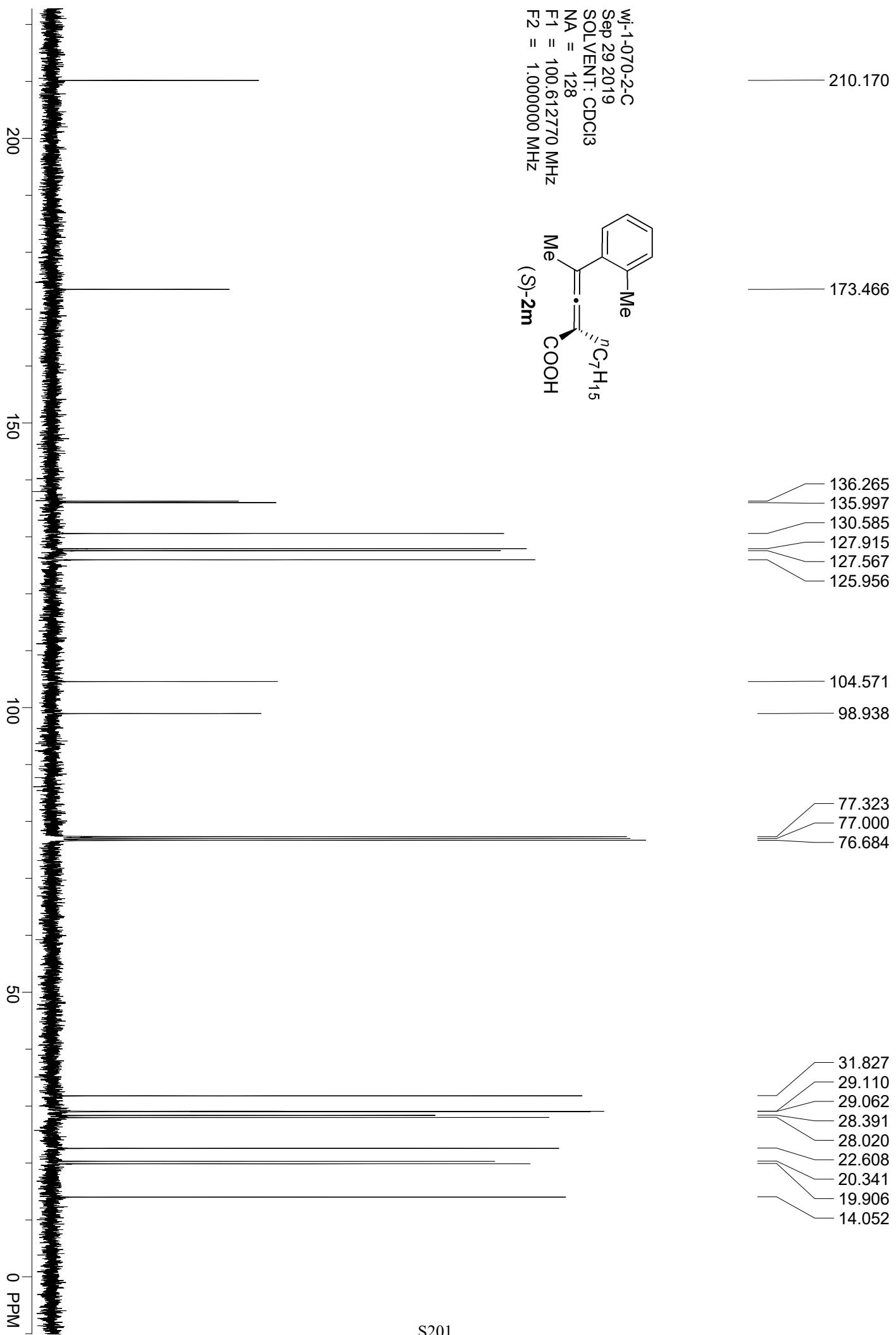
Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
7.493	0.2413	742.0991	12042.5449	49.7878
13.144	0.4960	370.9405	12145.1973	50.2122
Sum		24187.7422	100.0000	





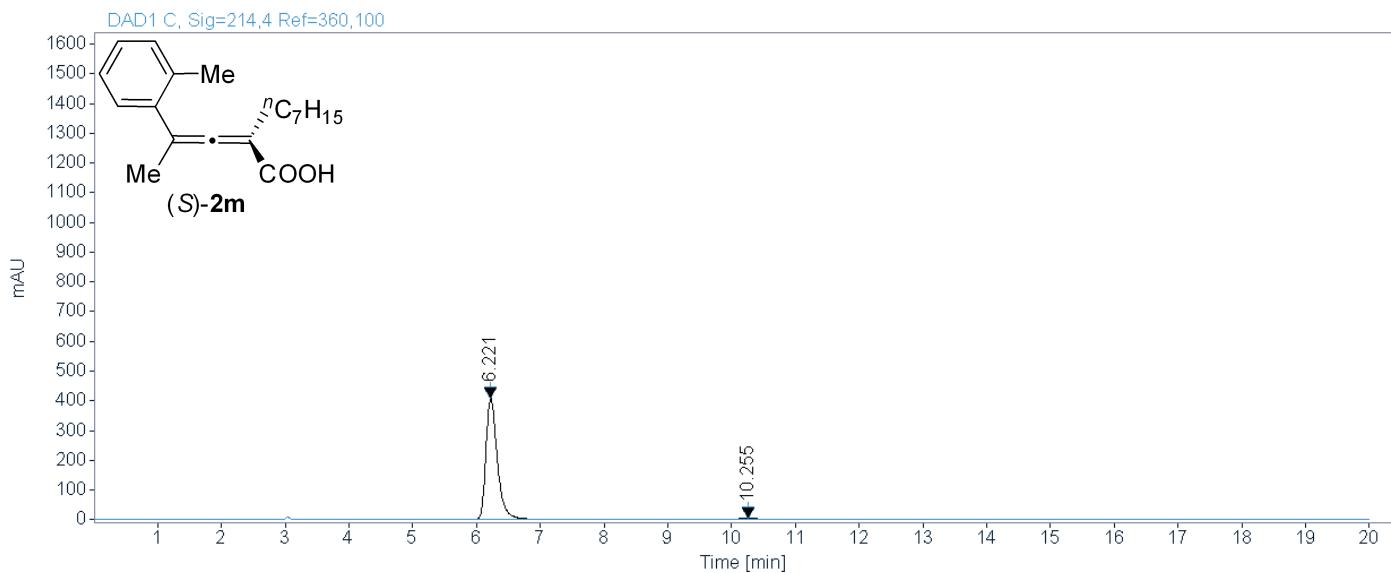
Area Percent Report



sample wj-1-070-2-AS-H-98-2-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\QAN 2019-09-29 18-30-35\016-P2-C3-wj-1-070-2.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
6.221	0.1867	409.8192	5083.7461	97.8153
10.255	0.4338	4.3622	113.5469	2.1847
Sum		5197.2930	100.0000	

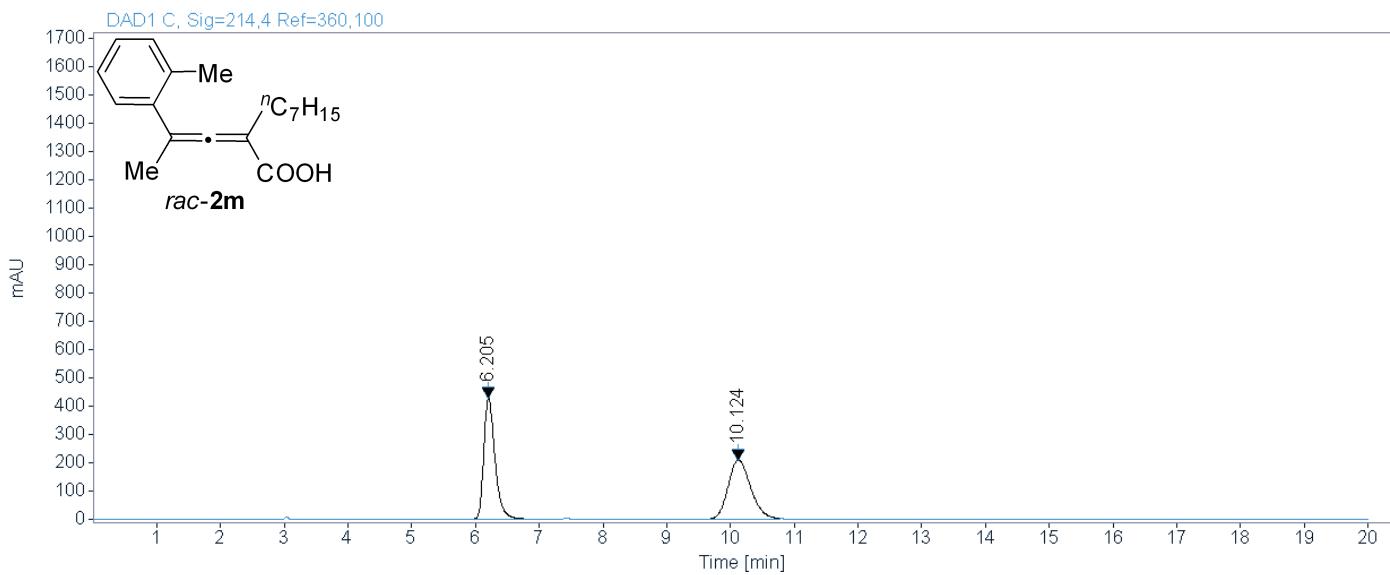
Area Percent Report



sample wj-1-070-2-rac-AS-H-98-2-1.0-214

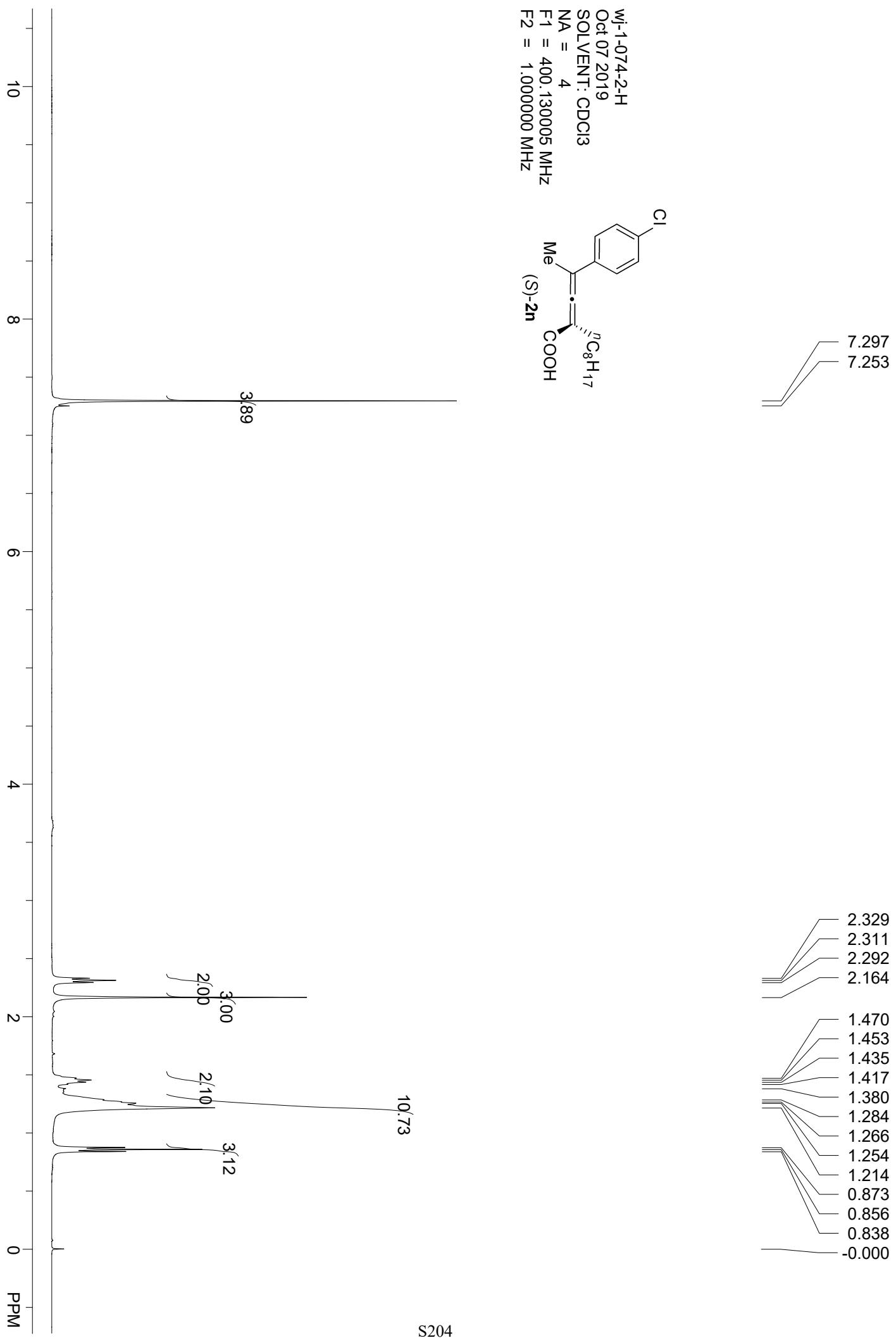
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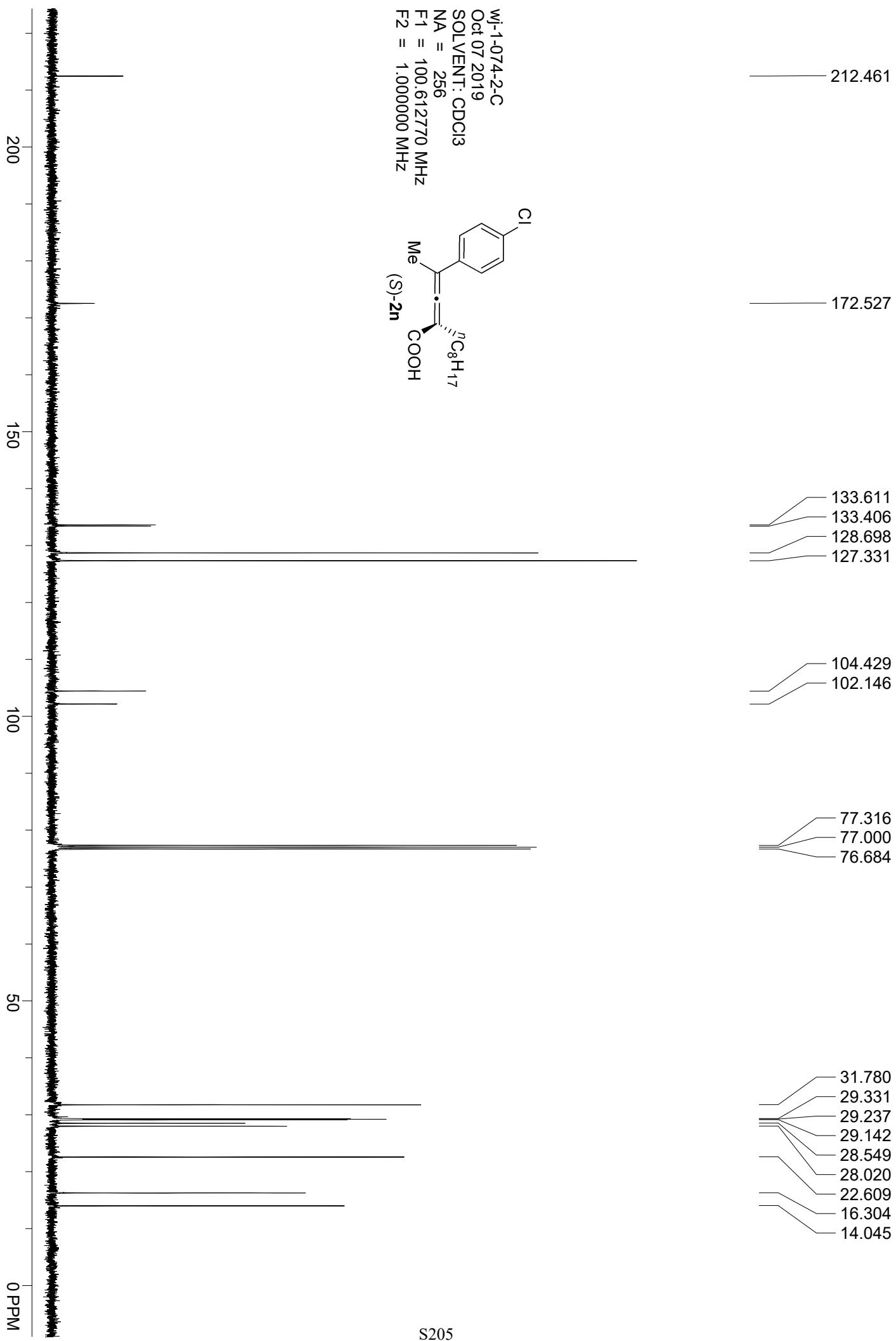
Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
6.205	0.1860	429.8355	5230.5894	50.1663
10.124	0.4128	209.7661	5195.9199	49.8337
Sum		10426.5093	100.0000	





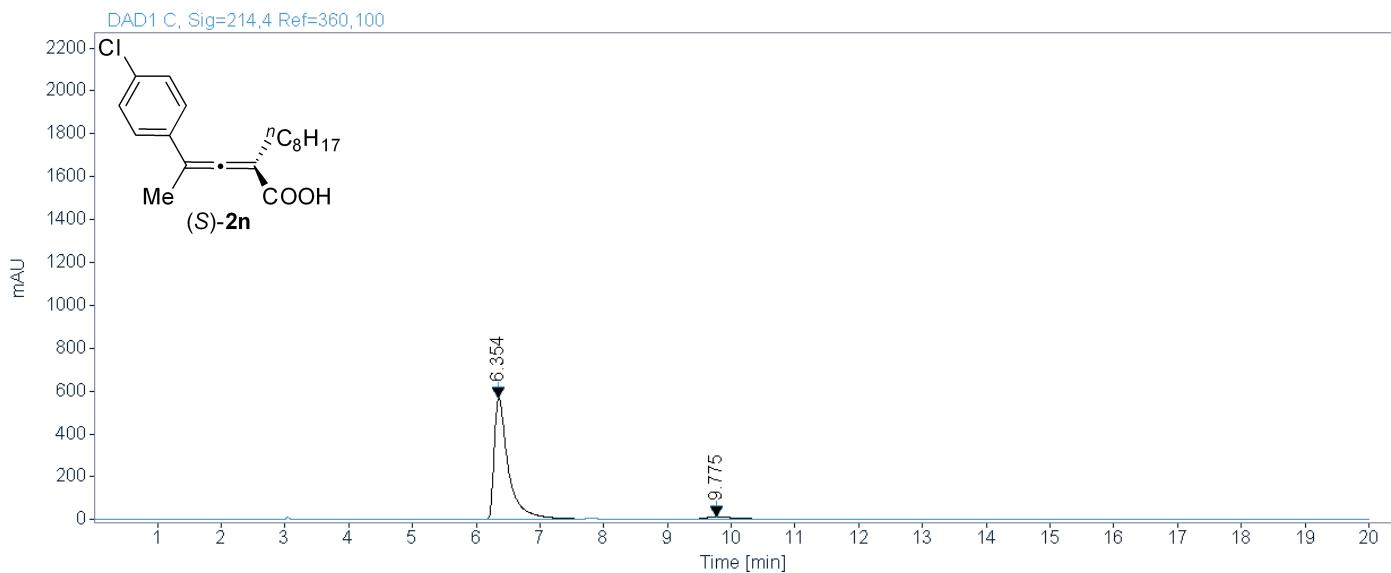
Area Percent Report



sample wj-1-074-2-AS-H-98-2-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\zwf-allenioic acid_LC 2019-10-06 14-14-49\027-P2-C7-wj-1-074-2.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
6.354	0.2598	568.2360	8858.5625	95.4554
9.775	0.5934	11.8454	421.7574	4.5446
		Sum	9280.3199	100.0000

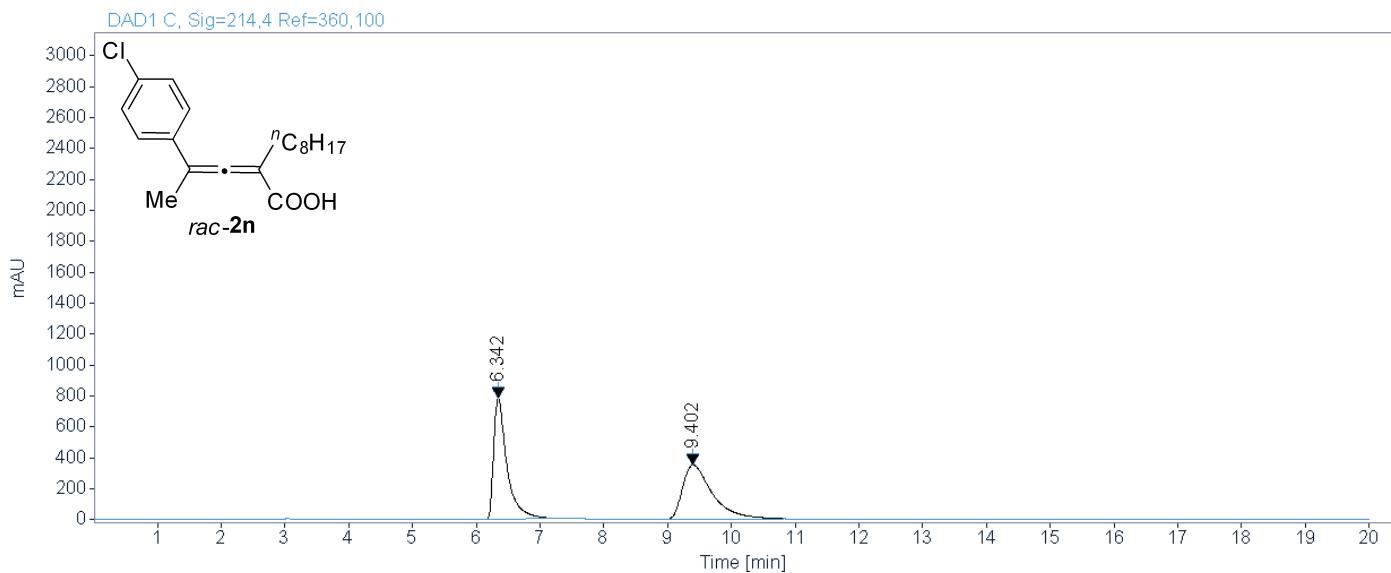
Area Percent Report



sample wj-1-074-2-rac-AS-H-98-2-1.0-214

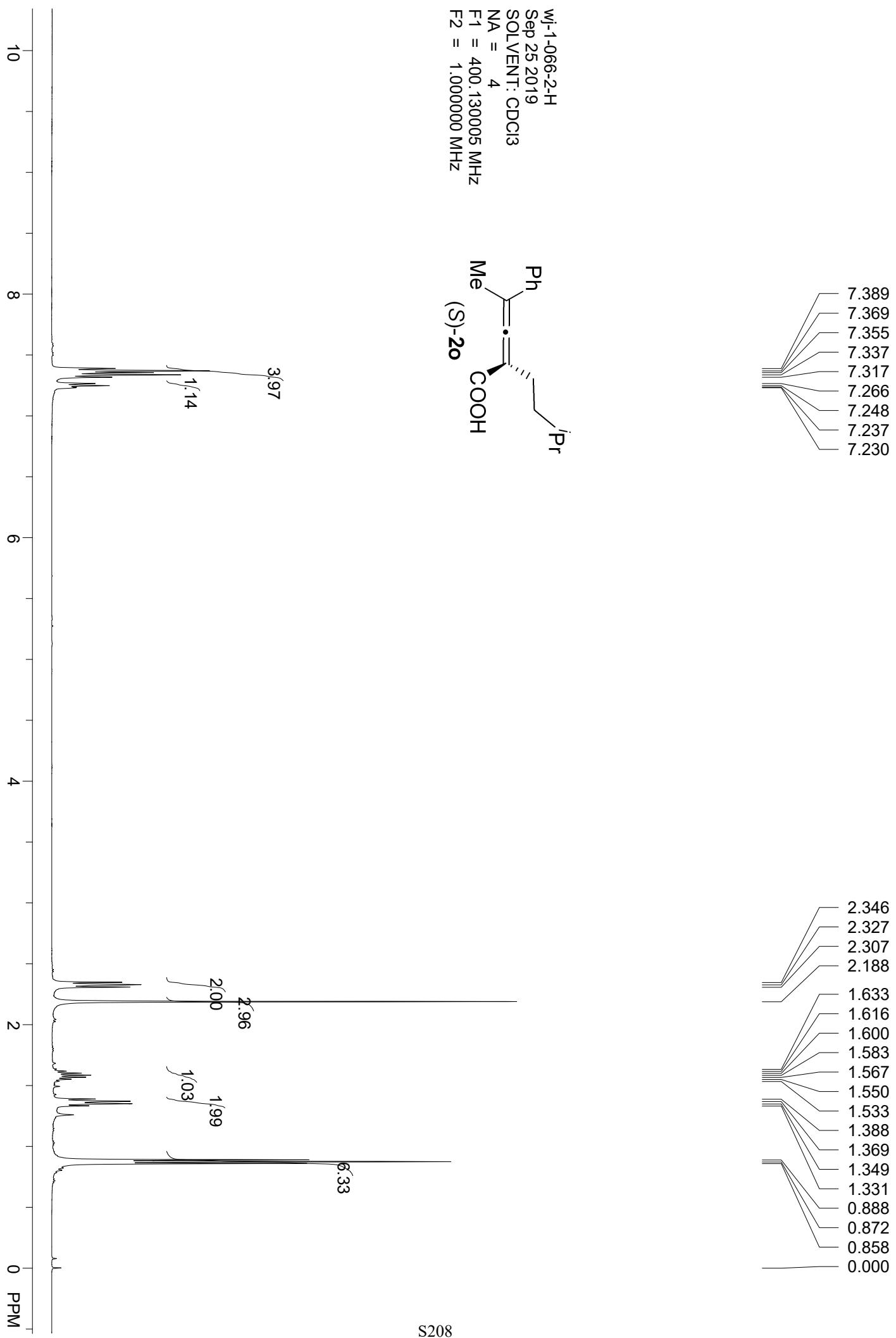
Data file: C:\Users\Public\Documents\ChemStation\1\Data\zwf-alleniac acid_LC 2019-10-06 14-14-49\028-P2-C8-wj-1-074-2-rac.D

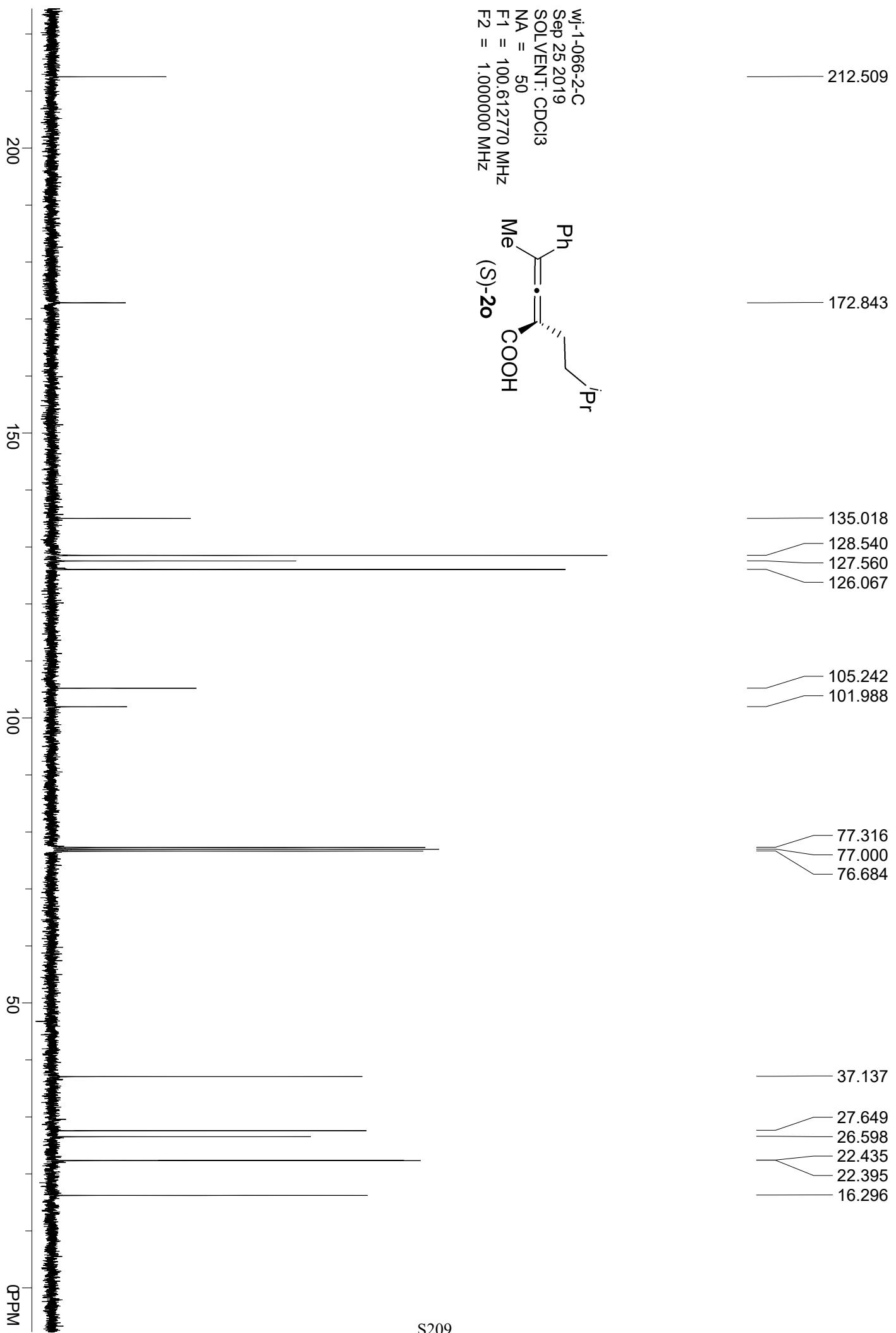
Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
6.342	0.2174	786.3198	11554.1123	50.0759
9.402	0.5432	353.4627	11519.1074	49.9241
		Sum	23073.2197	100.0000





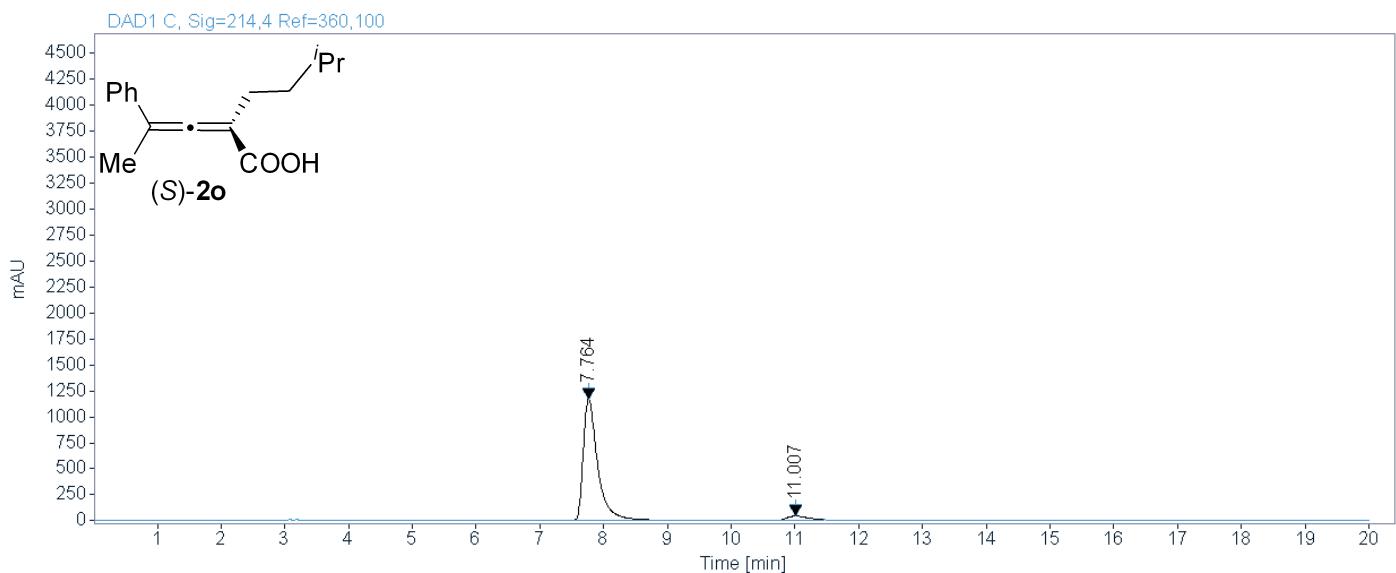
Area Percent Report



sample wj-1-066-2-AS-H-98-2-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\wgl 2019-09-25 09-43-22\039-P2-C1-wj-1-066-2.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
7.764	0.2391	1173.9525	18828.7598	95.3185
11.007	0.3785	40.7234	924.7599	4.6815
Sum		19753.5197	100.0000	

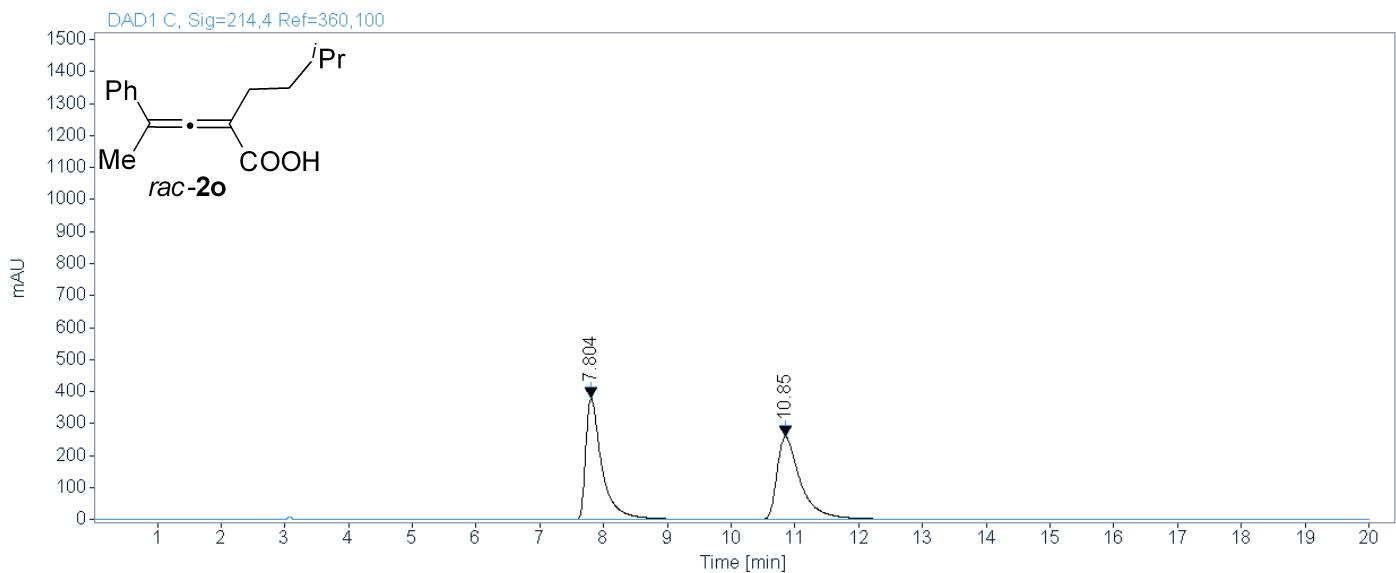
Area Percent Report



sample wj-1-066-2-rac-AS-H-98-2-1.0-214

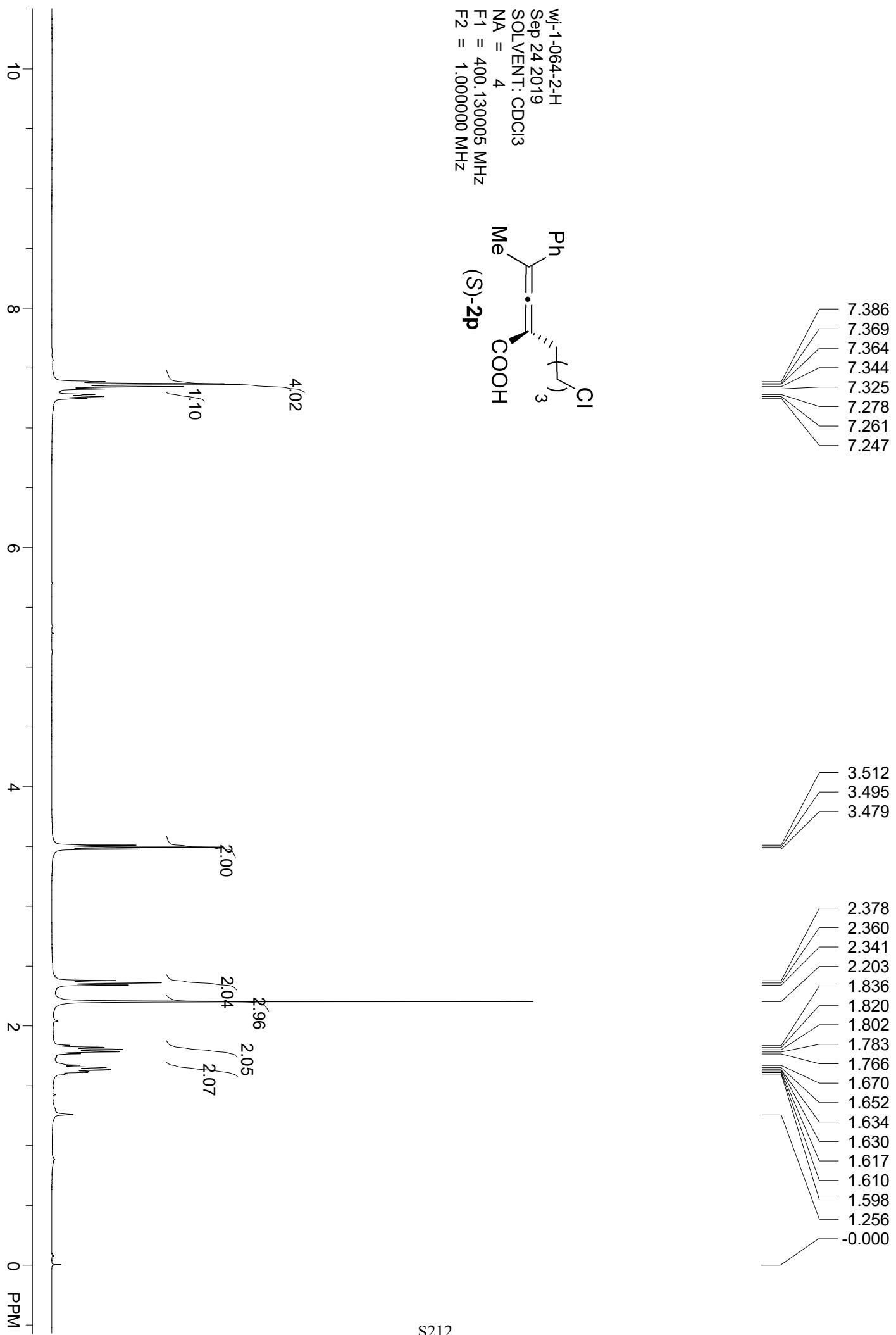
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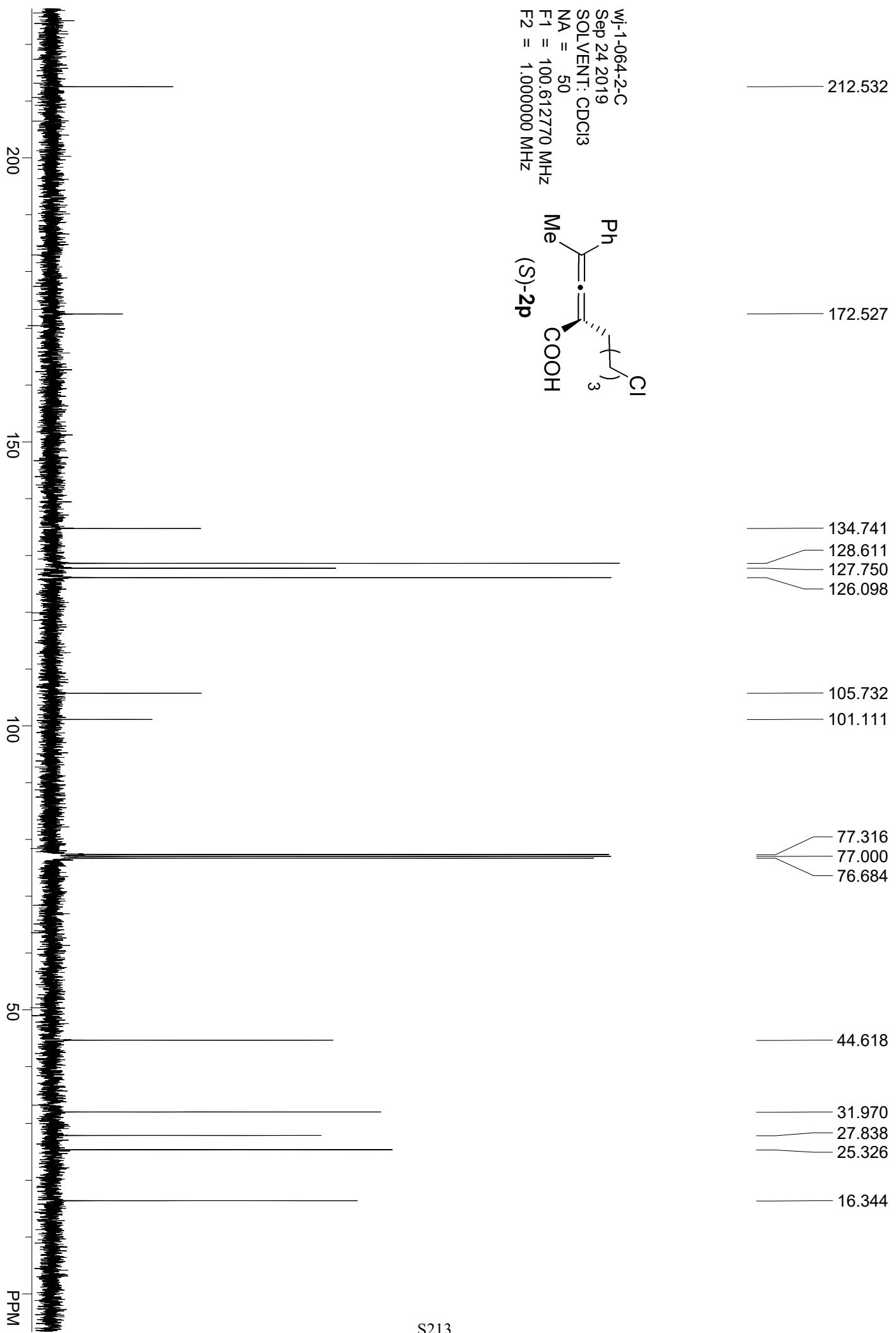
Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
7.804	0.2621	380.7250	6792.5049	50.0949
10.850	0.3886	259.1956	6766.7642	49.9051
Sum		13559.2690	100.0000	





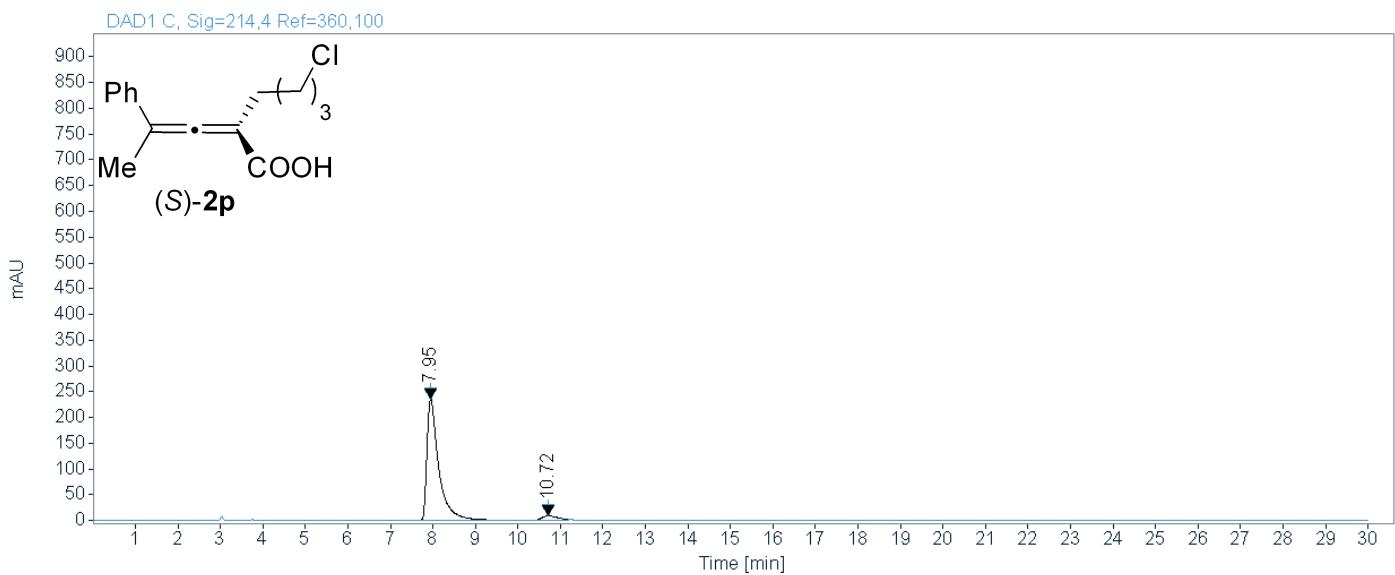
Area Percent Report



sample wj-1-064-2-AS-H-95-5-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\wgl 2019-09-24 10-35-10\033-P2-C5-wj-1-064-2.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
7.950	0.2844	236.0499	4580.5767	95.5169
10.720	0.4204	8.5227	214.9886	4.4831
		Sum	4795.5653	100.0000

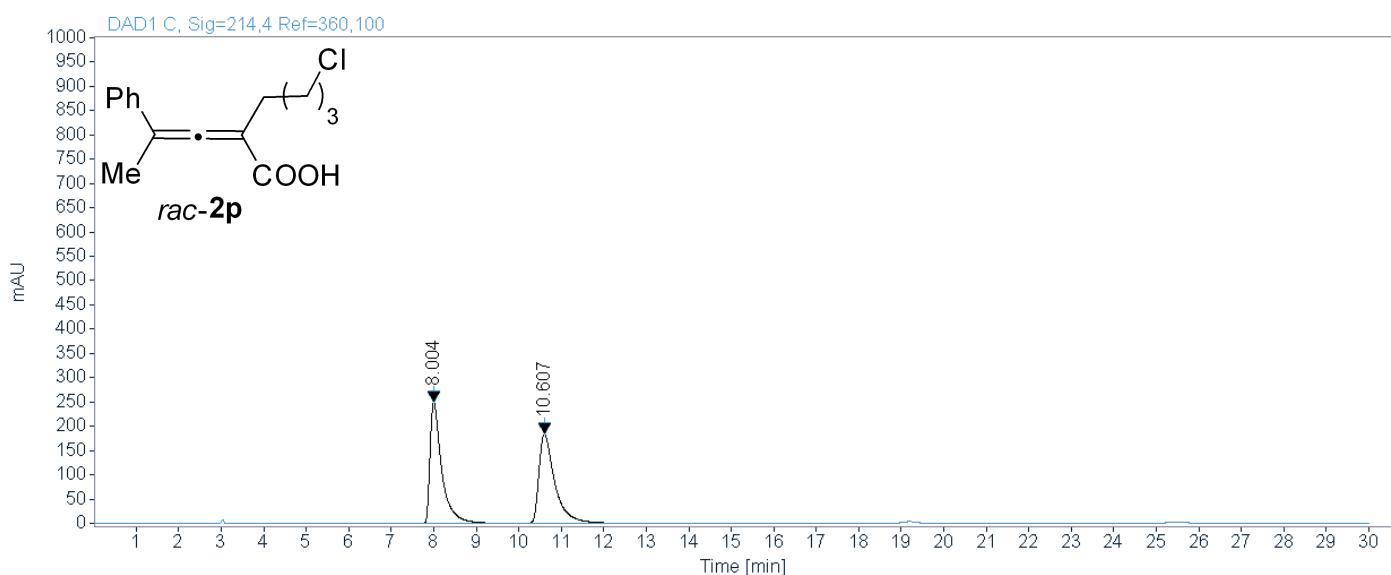
Area Percent Report



sample wj-1-064-2-rac-AS-H-95-5-1.0-214

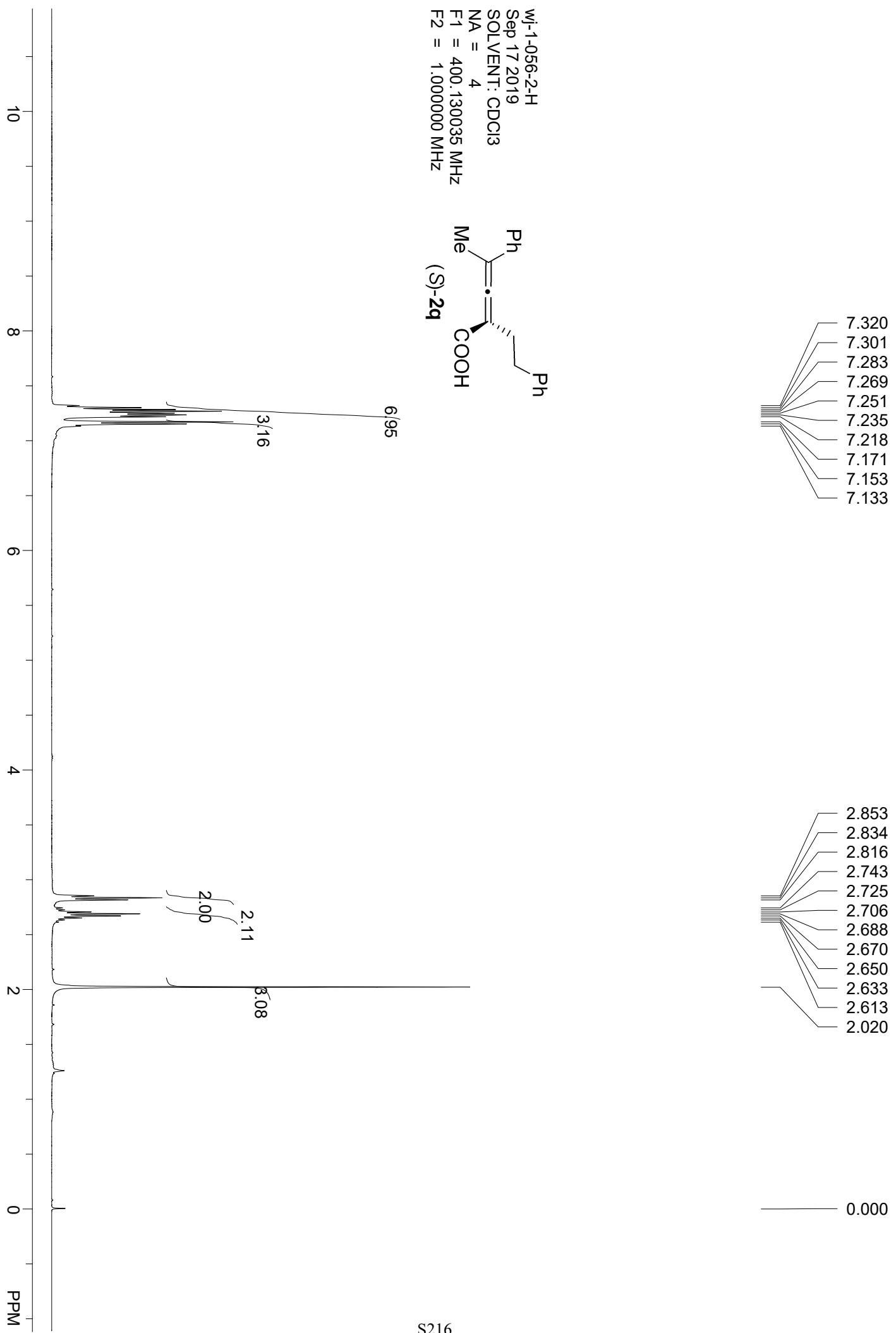
Data file: C:\Users\Public\Documents\ChemStation\1\Data\wgl 2019-09-24 10-35-10\034-P2-C6-wj-1-064-2-rac.D

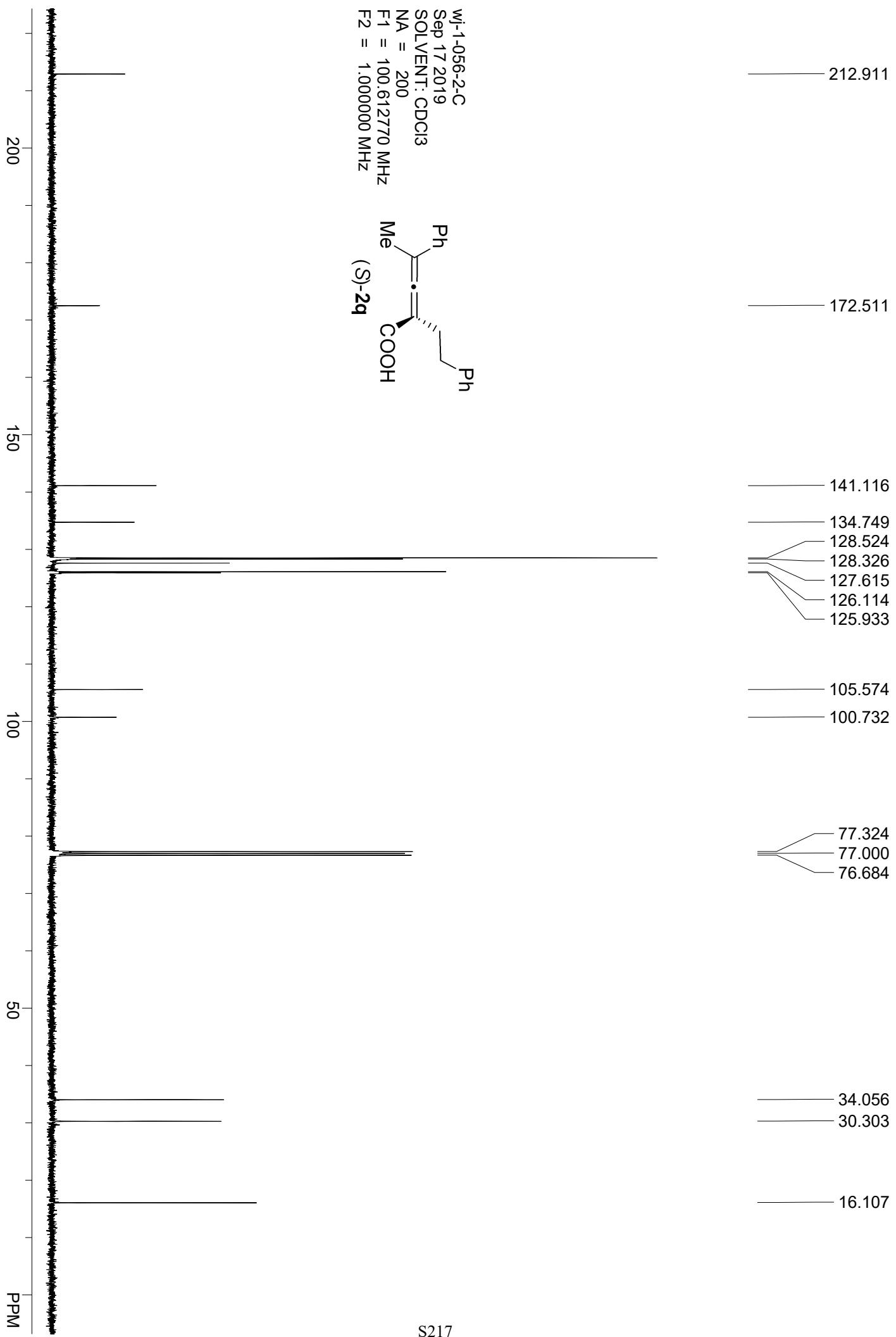
Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
8.004	0.2830	250.5520	4832.7207	49.8970
10.607	0.3901	183.7673	4852.6724	50.1030
Sum		9685.3931	100.0000	





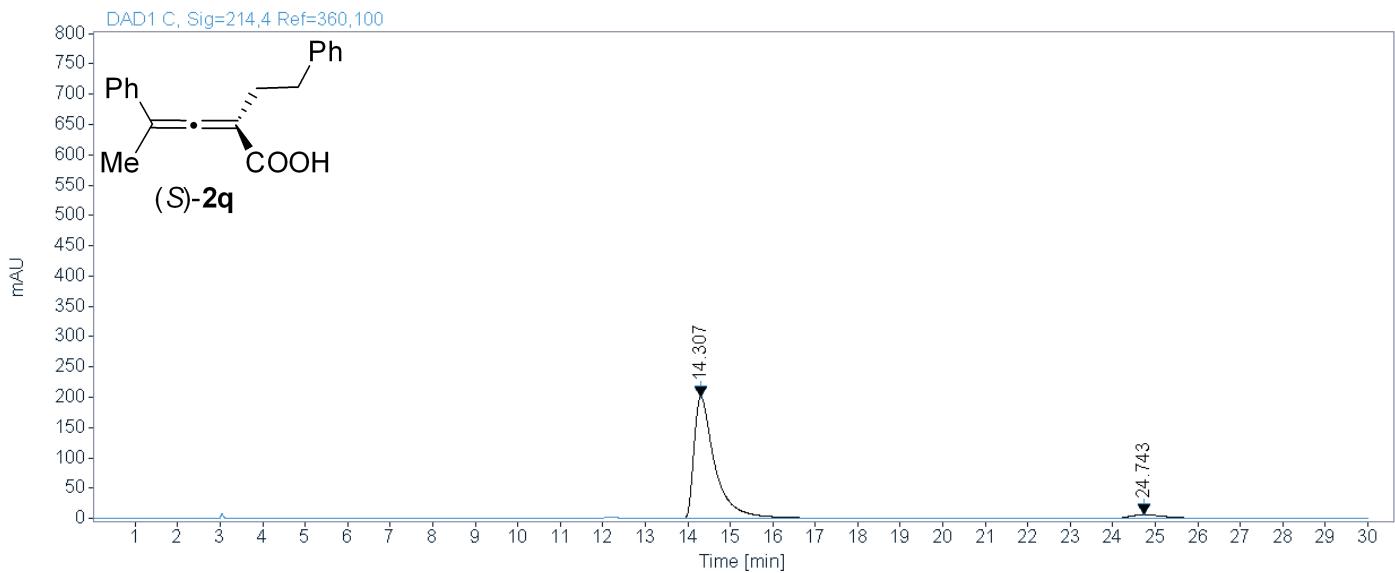
Area Percent Report



sample wj-1-056-2-AS-H-98-2-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\wgl 2019-09-17 09-02-49\055-P2-C3-wj-1-056-2.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
14.307	0.5110	200.8281	6936.7827	95.8457
24.743	0.8684	5.7703	300.6647	4.1543
		Sum	7237.4474	100.0000

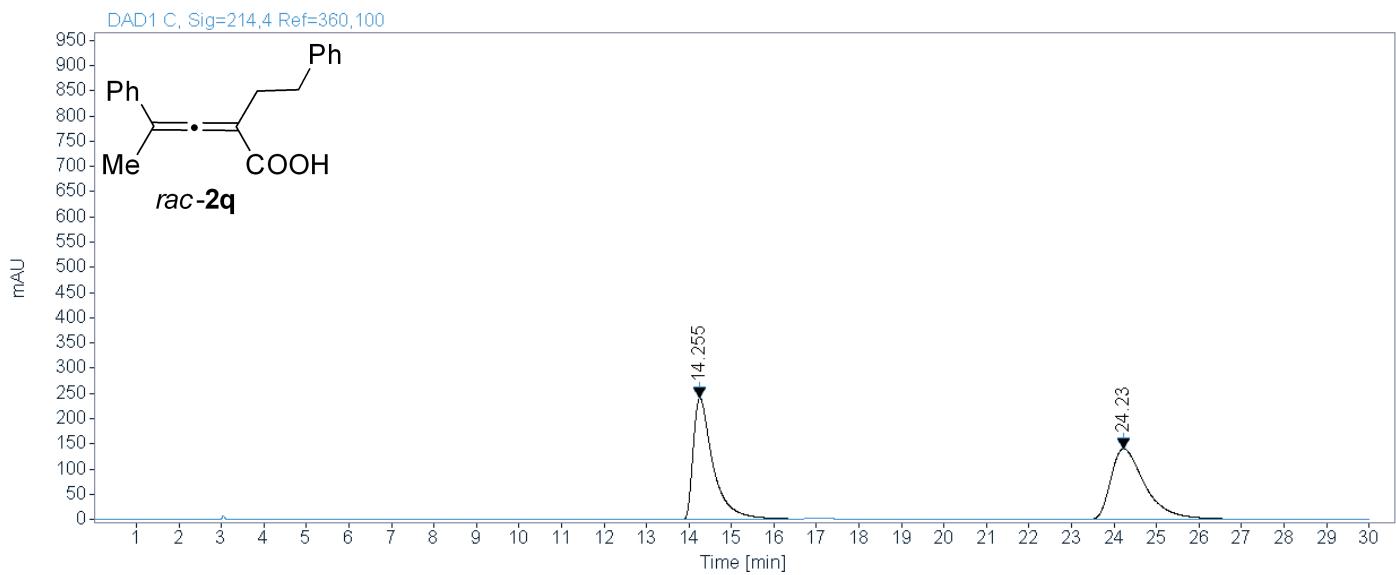
Area Percent Report



sample wj-1-056-2-rac-AS-H-98-2-1.0-214

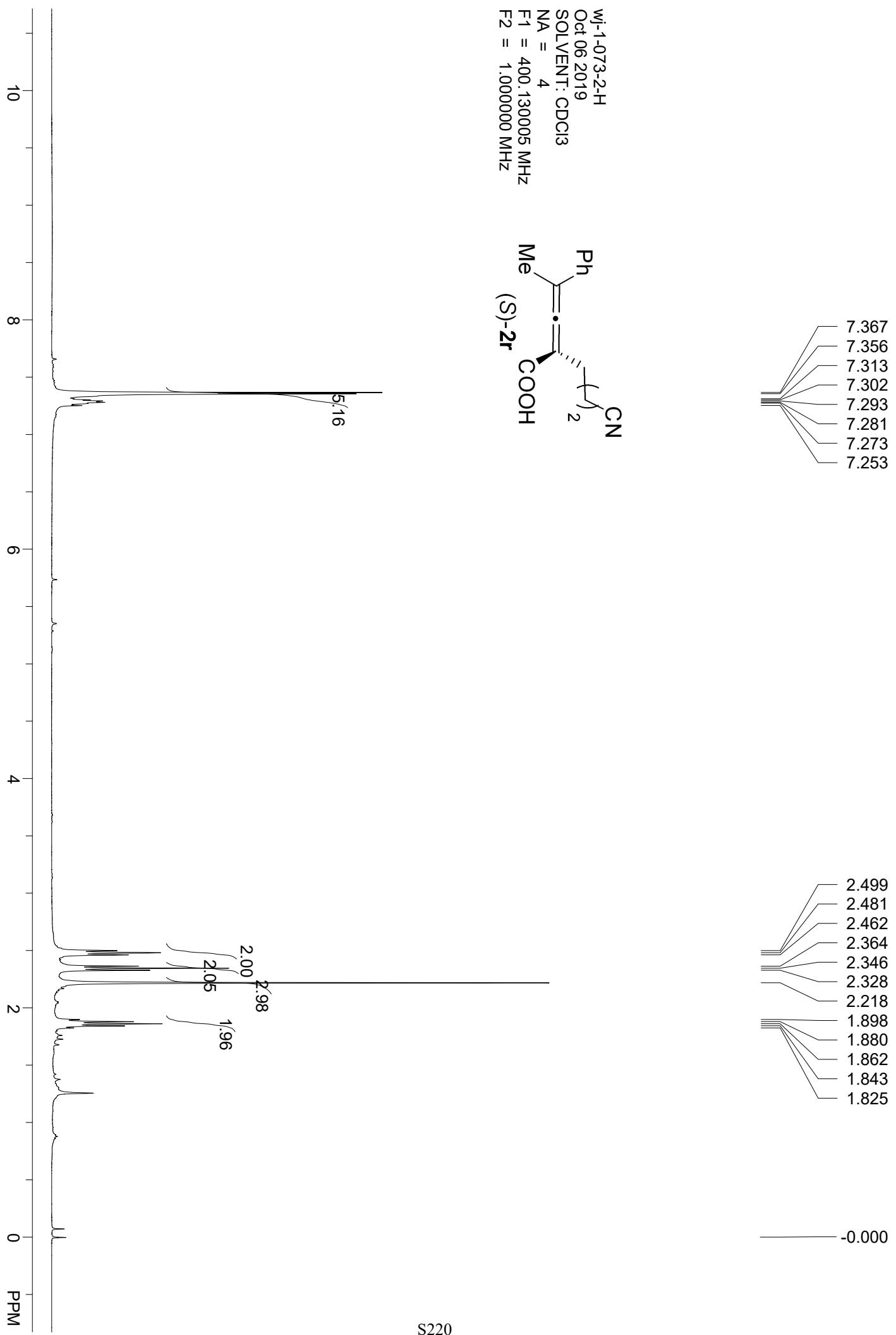
Data file: C:\Users\Public\Documents\ChemStation\1\Data\wgl 2019-09-17 09-02-49\056-P2-C4-wj-1-056-2-rac.D

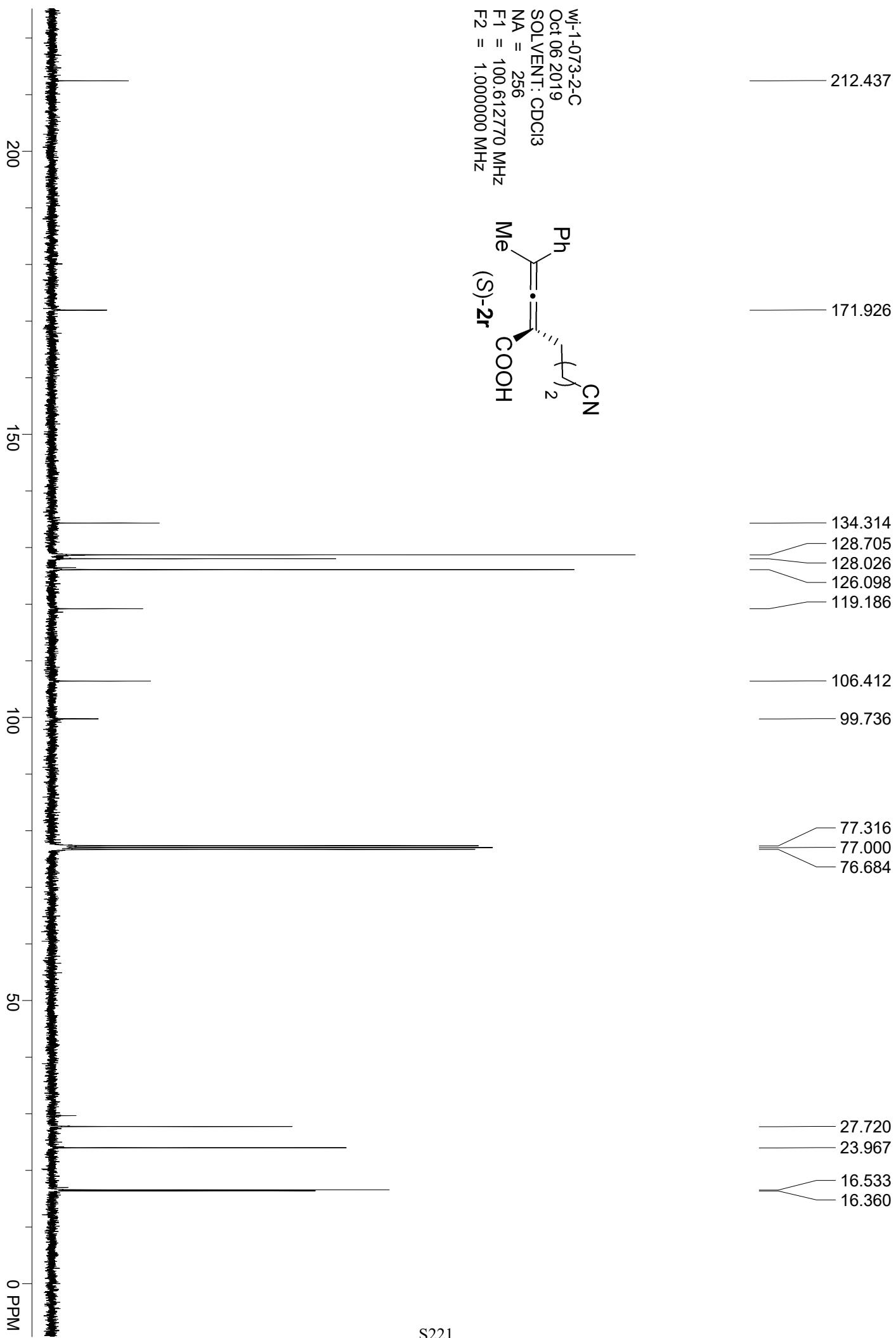
Acquisition Data:

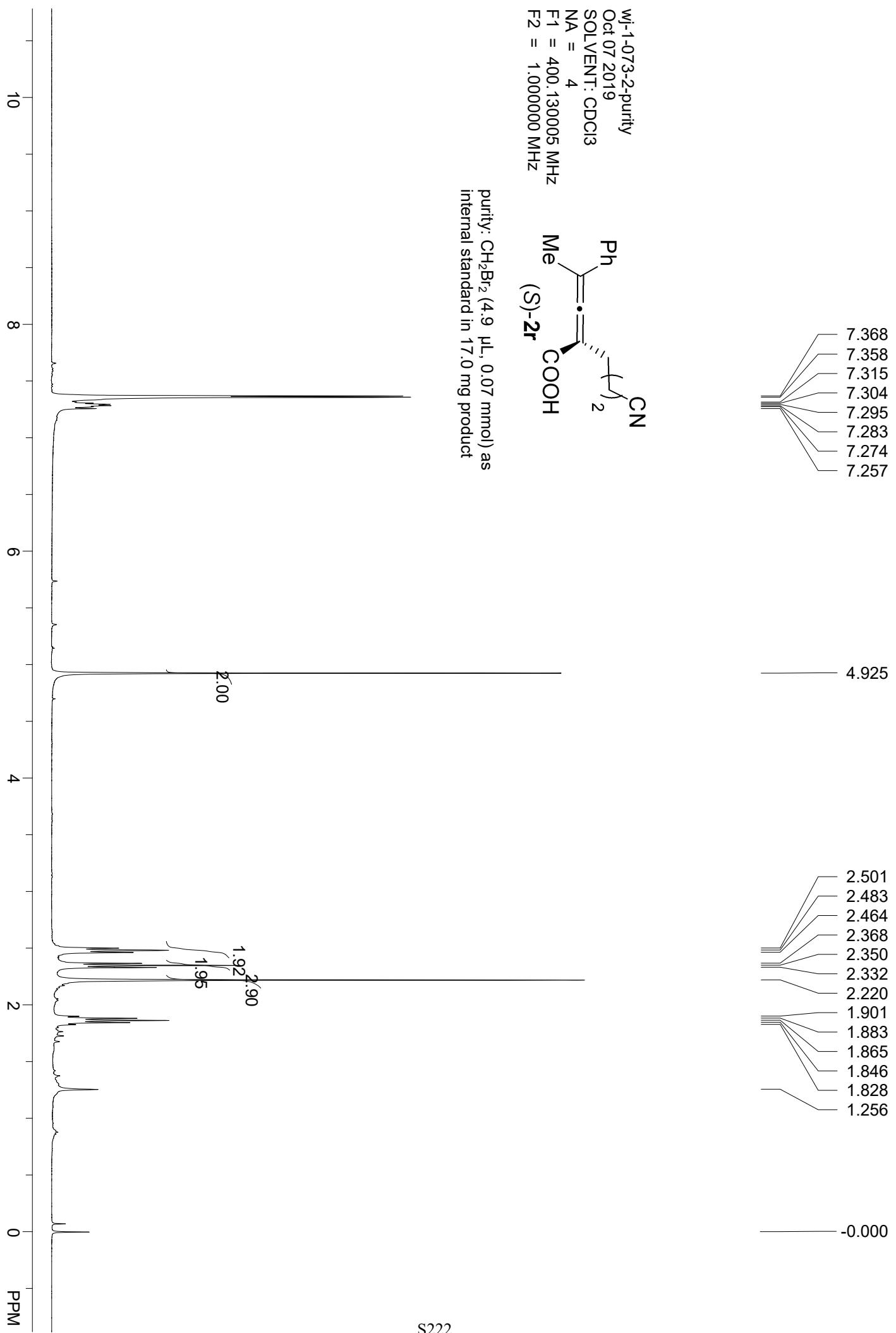


Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
14.255	0.4842	241.2527	7861.6177	49.6767
24.230	0.8614	140.4720	7963.9458	50.3233
		Sum	15825.5635	100.0000







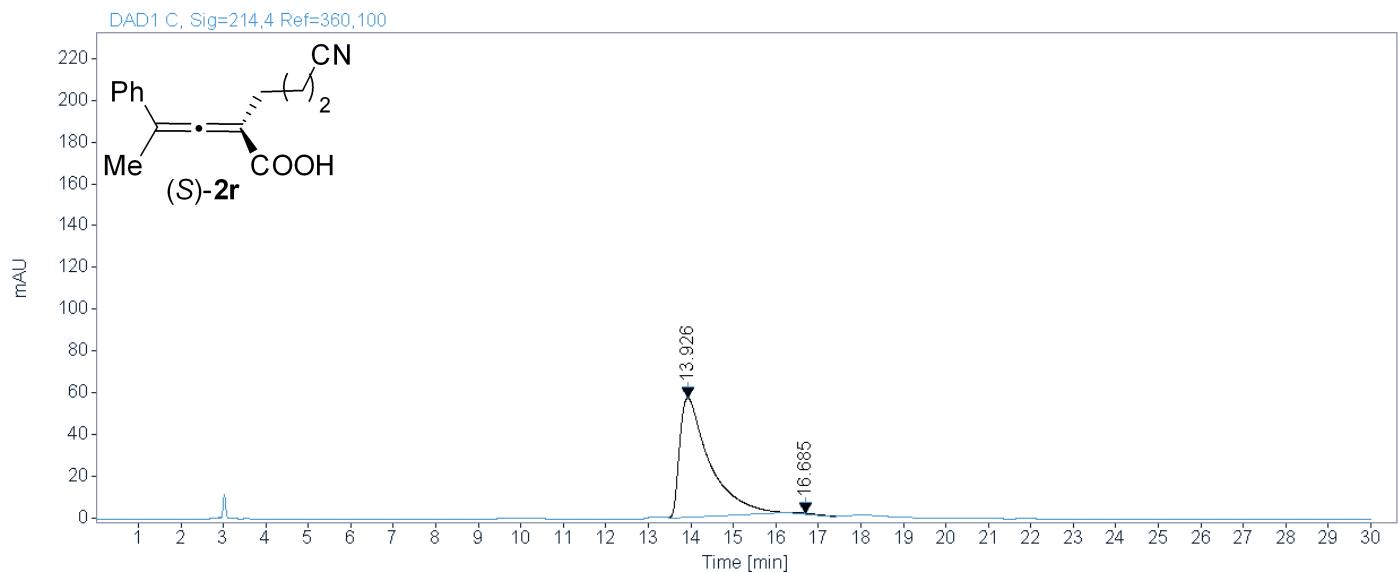
Area Percent Report



sample wj-1-073-2-AS-H-90-10-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\zwf-allenioc acid_LC 2019-10-06 14-14-49\022-P2-C3-wj-1-073-2.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
13.926	0.8154	57.1405	2795.6130	99.0068
16.685	0.8372	0.5583	28.0436	0.9932
		Sum	2823.6566	100.0000

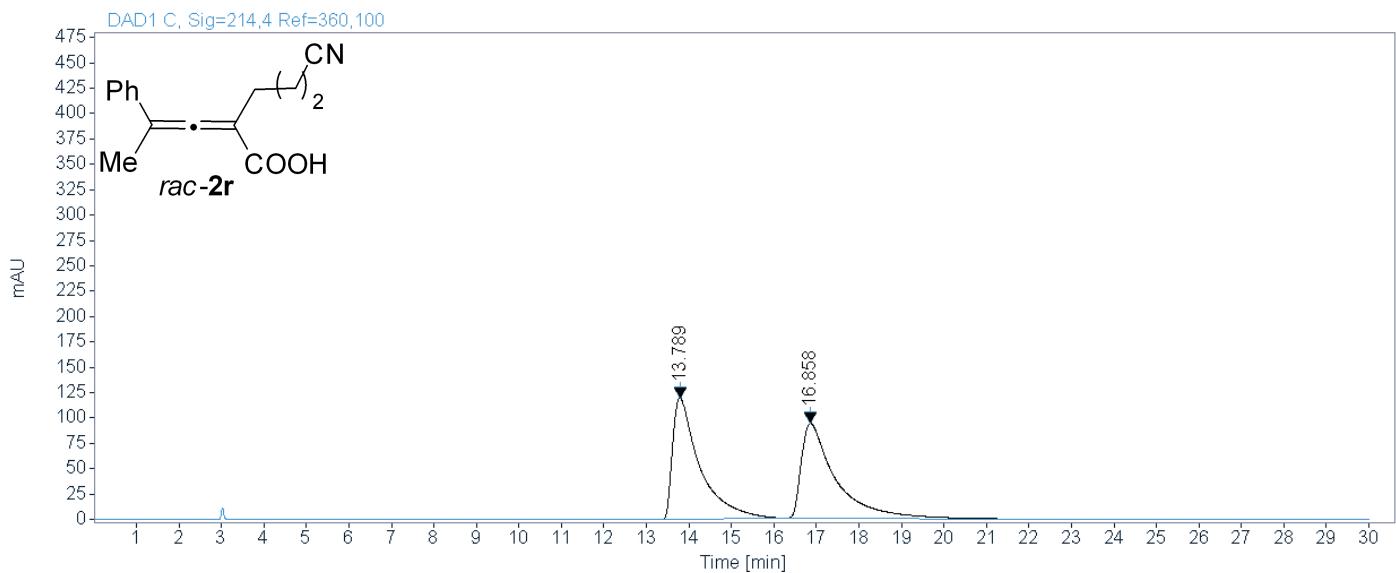
Area Percent Report



sample wj-1-073-2-rac-AS-H-90-10-1.0-214

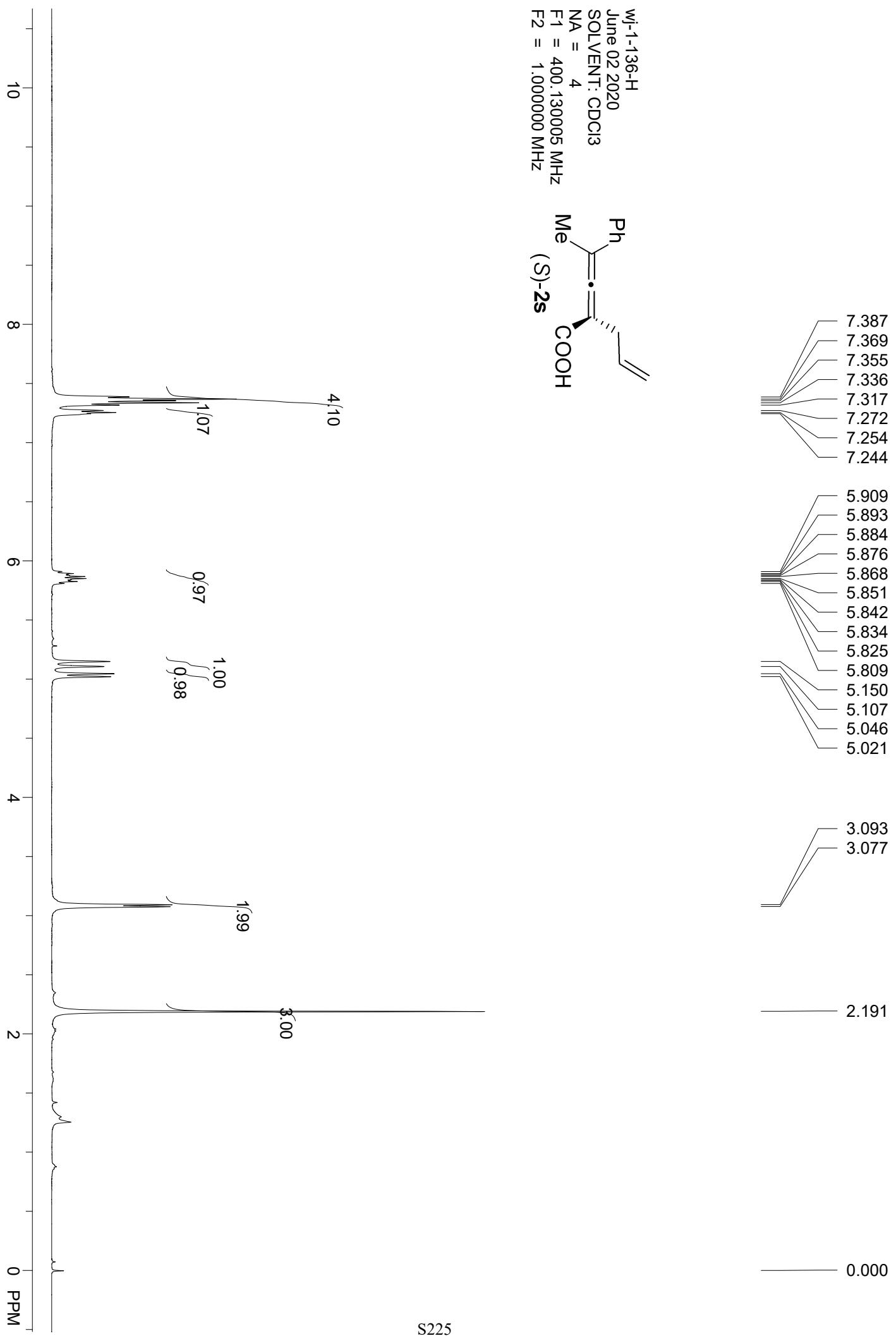
Data file: C:\Users\Public\Documents\ChemStation\1\Data\zwf-allenioc acid_LC 2019-10-06 14-14-49\023-P2-C4-wj-1-073-2-rac.D

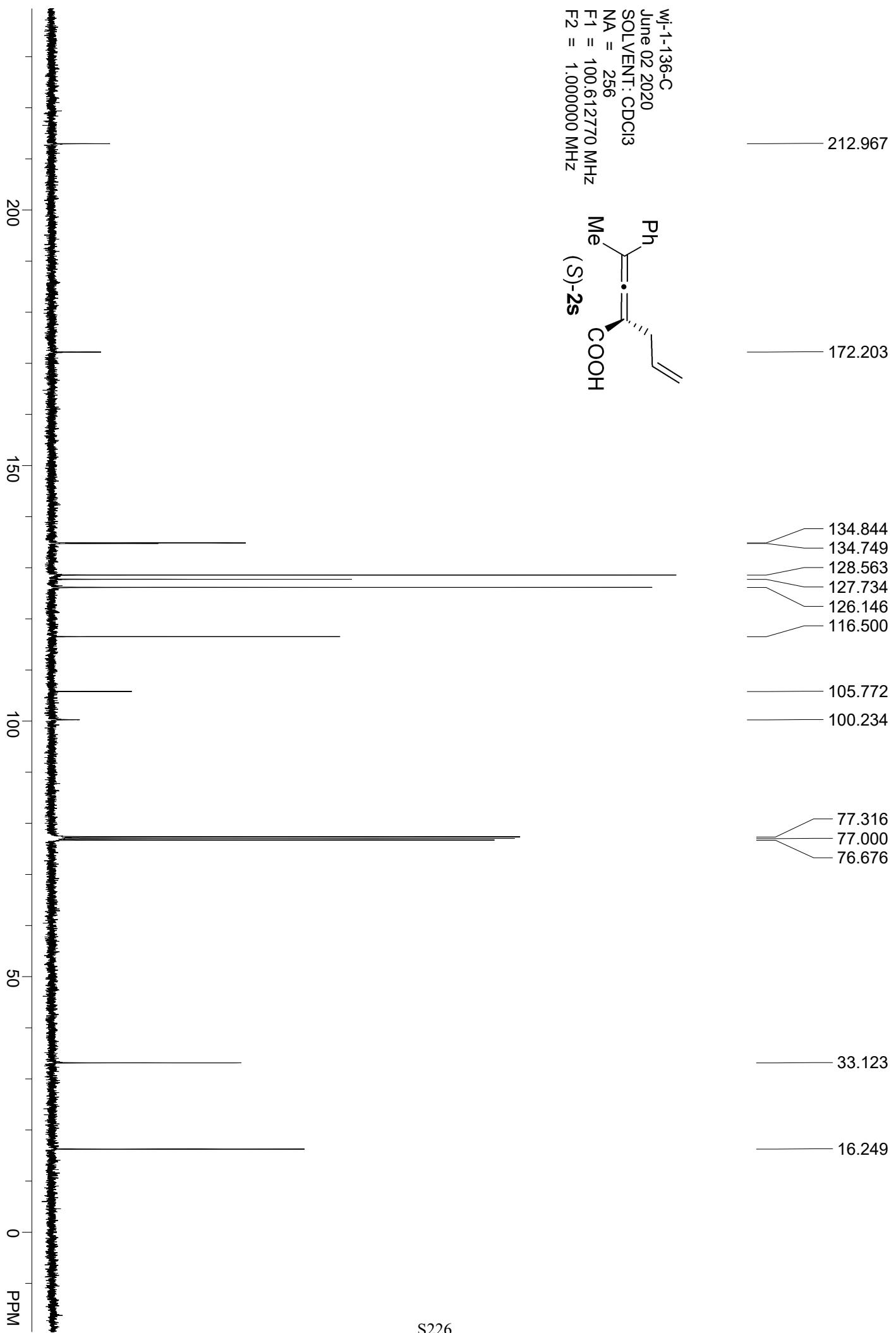
Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
13.789	0.6321	119.7780	5298.2300	50.0652
16.858	0.8062	93.3867	5284.4390	49.9348
Sum		10582.6689	100.0000	





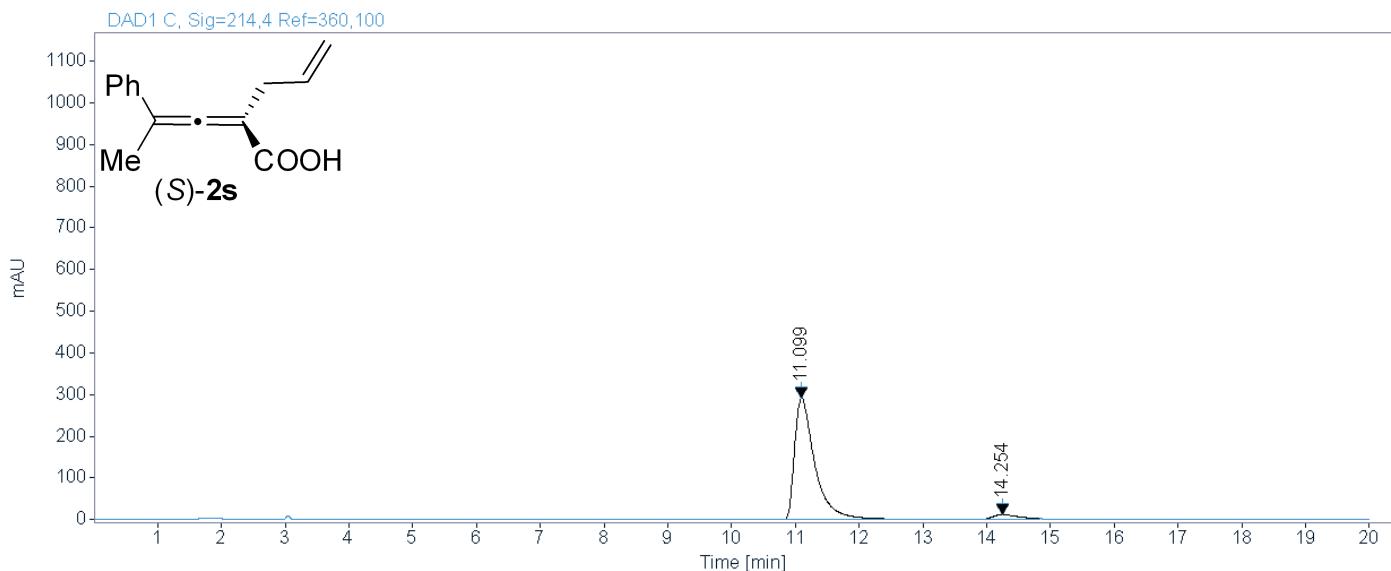
Area Percent Report



sample wj-1-136-AS-H-98-2-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\zwf-allenioc acid_LC 2020-06-01 19-44-43\010-P2-C4-wj-1-136.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
11.099	0.3323	292.3181	6522.3804	94.8461
14.254	0.5026	11.7540	354.4257	5.1539
Sum		292.3181	6876.8061	100.0000

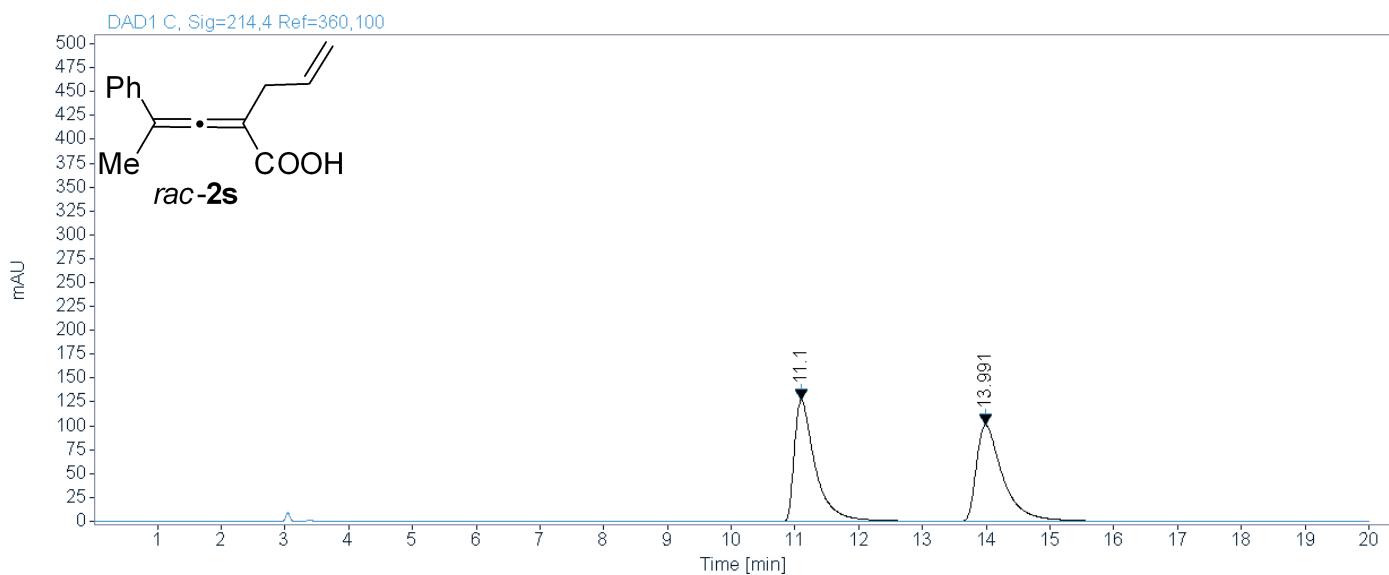
Area Percent Report



sample wj-1-136-rac-AS-H-98-2-1.0-214

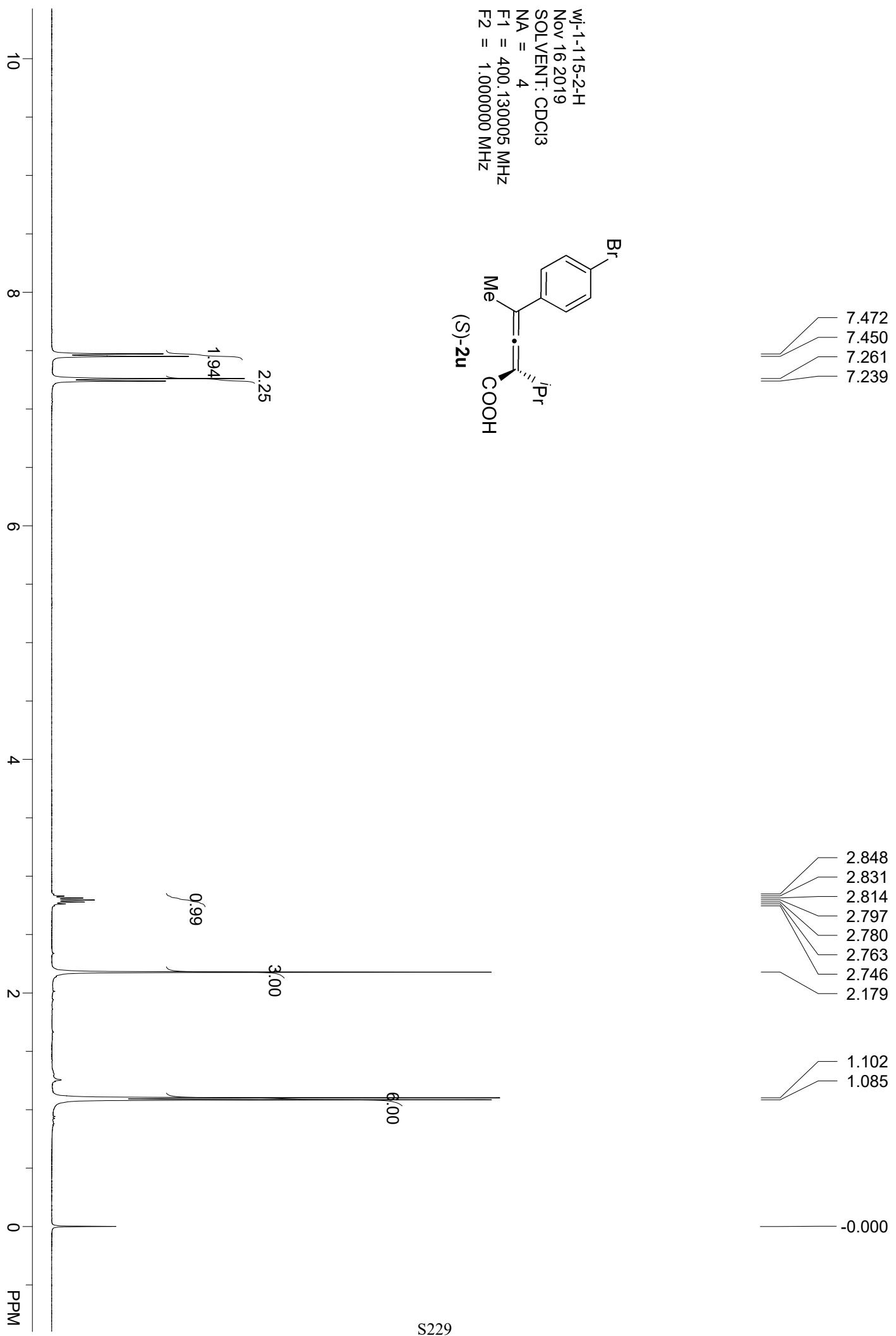
Data file: C:\Users\Public\Documents\ChemStation\1\Data\zwf-allenioc acid_LC 2020-06-01 19-44-43\011-P2-C5-wj-1-136-rac.D

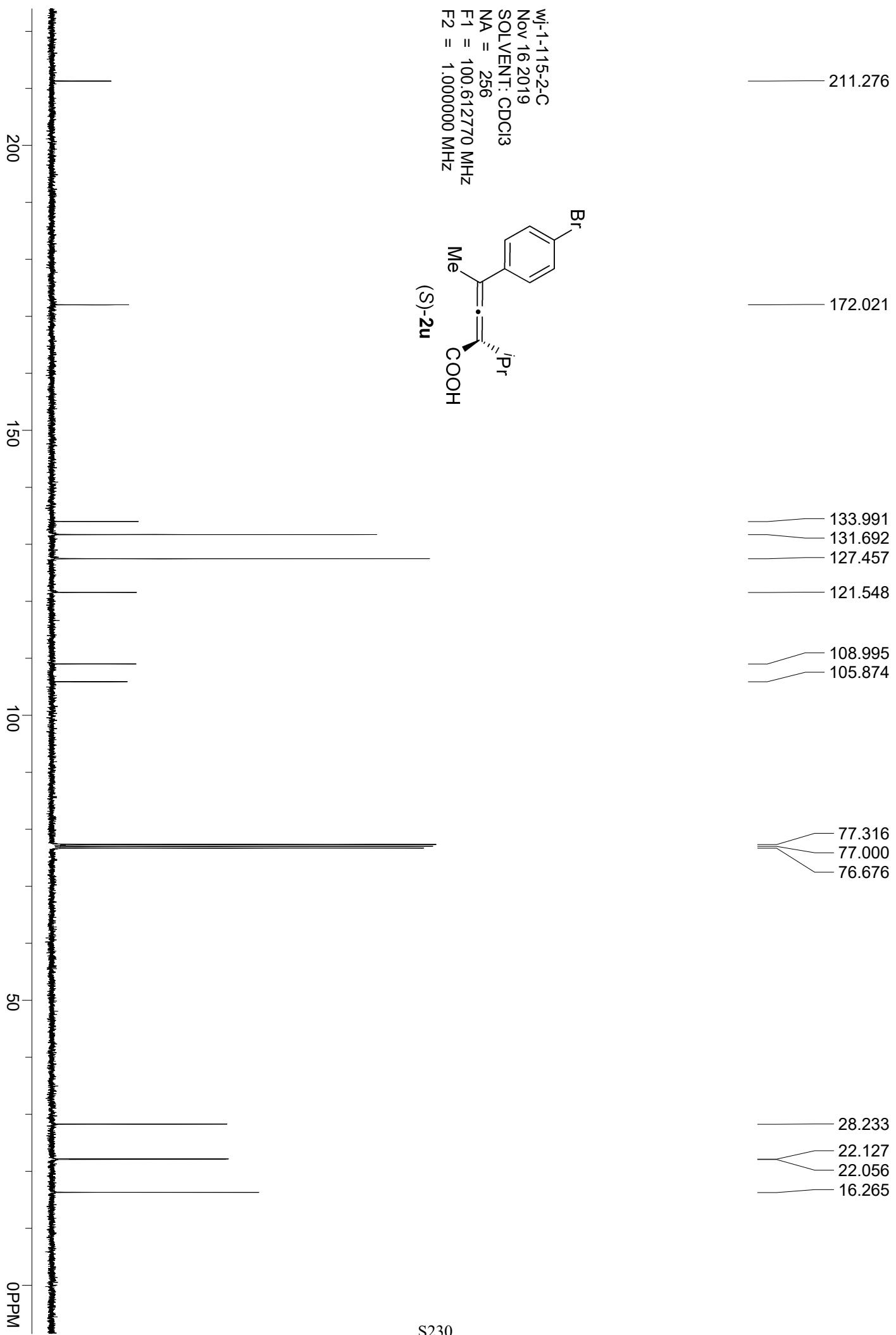
Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
11.100	0.3417	127.4330	2945.4487	50.4505
13.991	0.4312	100.6969	2892.8433	49.5495
Sum		5838.2920	100.0000	





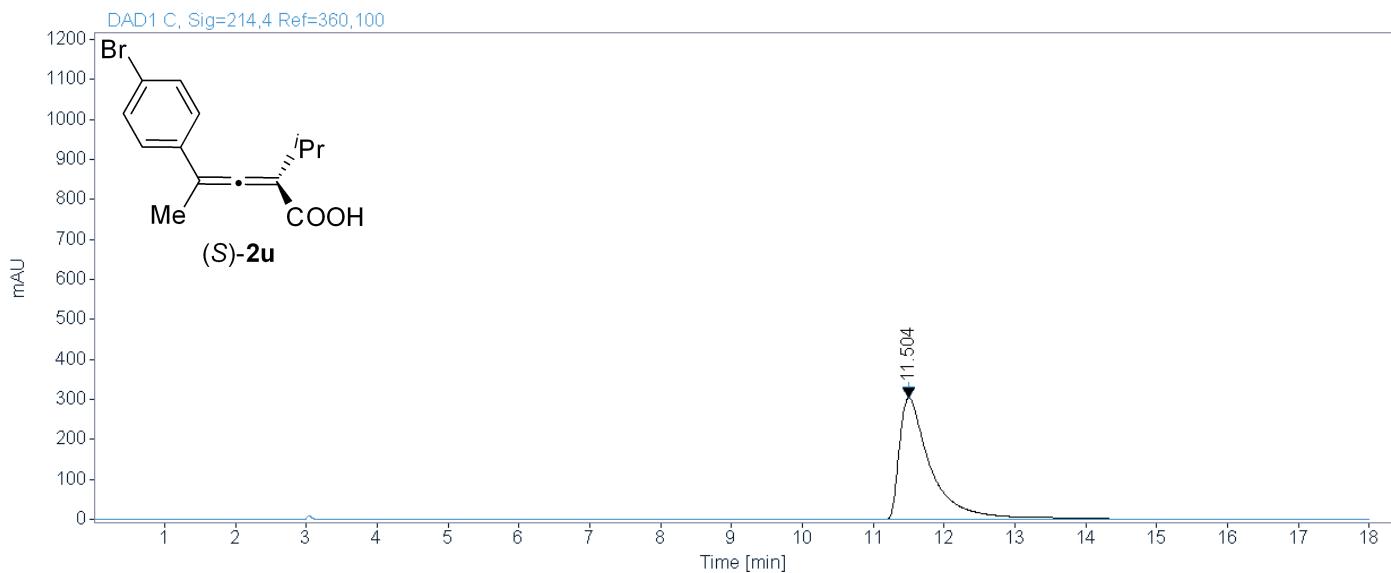
Area Percent Report



sample wj-1-115-2-AS-H-98-2-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\zwf-allenioc acid_LC 2019-11-15 20-57-54\012-P2-C5-wj-1-115-2.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
11.504	0.4649	304.5253	9734.5674	100.0000
		Sum	9734.5674	100.0000

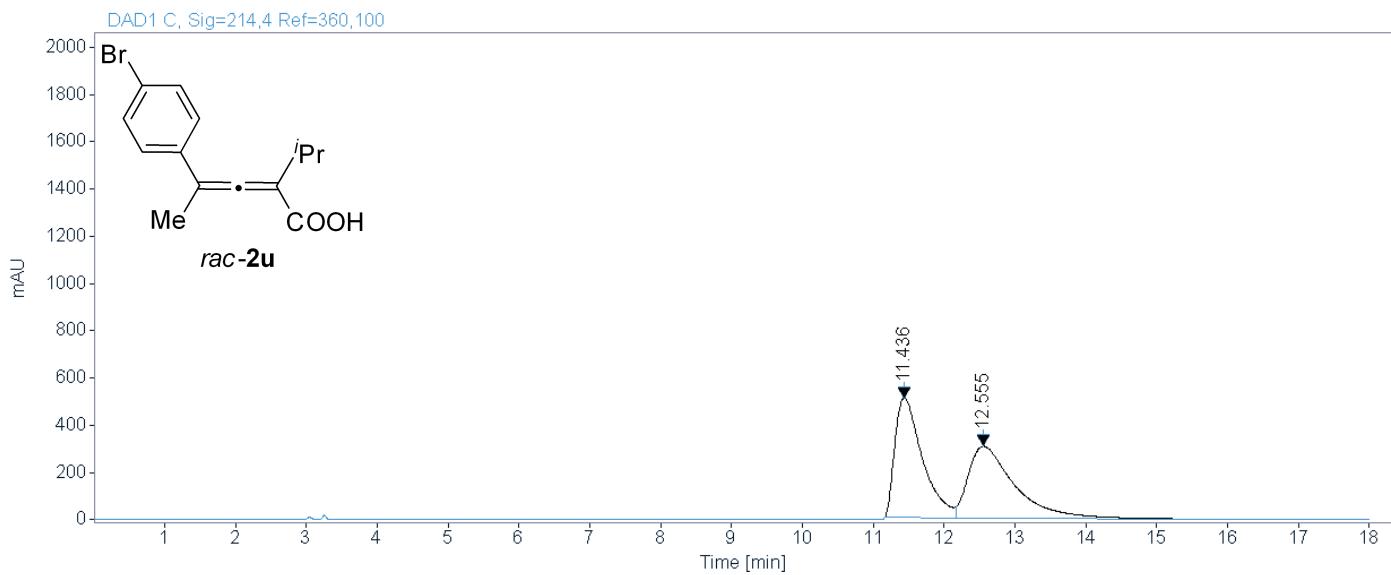
Area Percent Report



sample wj-1-115-2-rac-AS-H-98-2-1.0-214

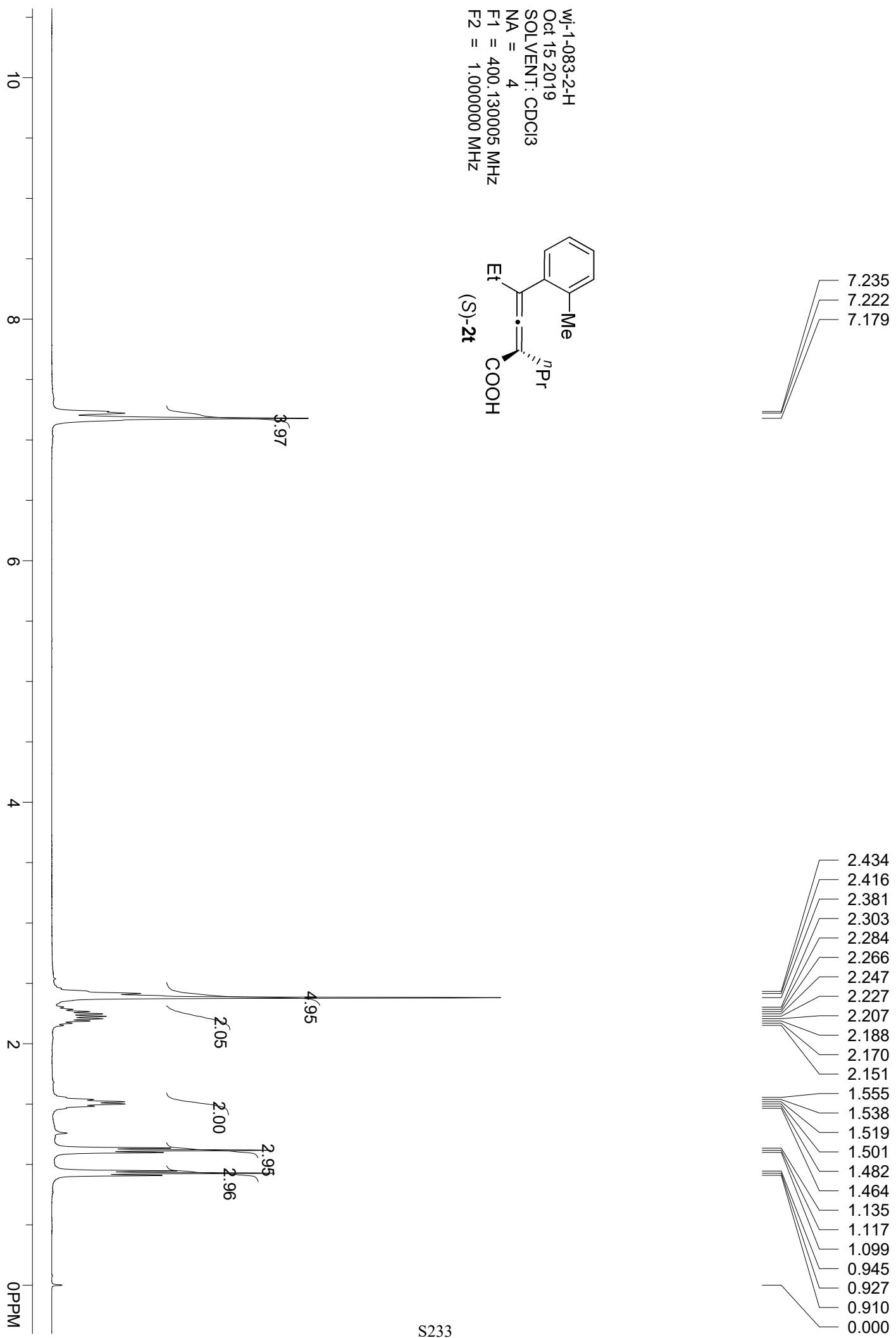
Data file: C:\Users\Public\Documents\ChemStation\1\Data\zwf-allenioc acid_LC 2019-11-15 20-57-54\013-P2-C6-wj-1-115-2-rac.D

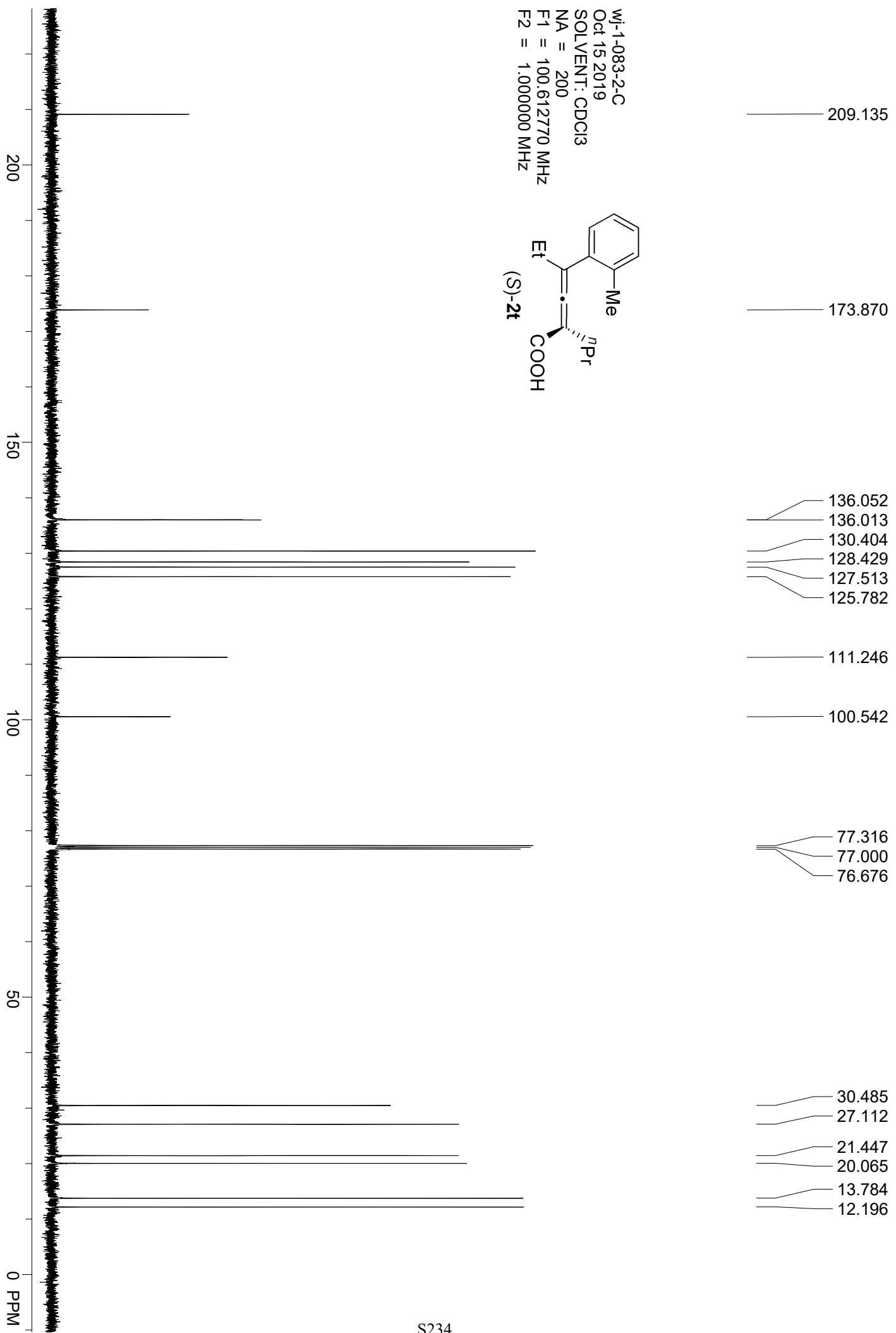
Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
11.436	0.4525	506.3639	13748.8301	49.6833
12.555	0.7652	303.2699	13924.1279	50.3167
Sum		27672.9580	100.0000	





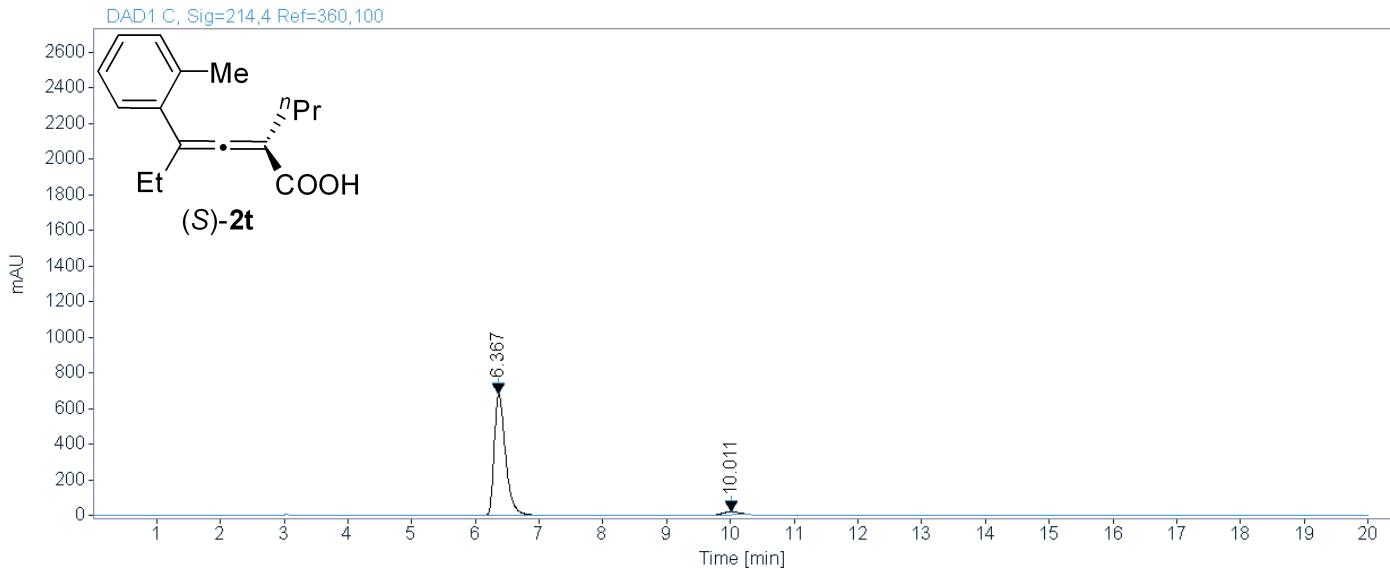
Area Percent Report



sample wj-1-083-2-AS-H-98-2-1.0-
214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\YuanYuan 2019-10-15 19-00-21\026-P2-C3-wj-1-083-2.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
6.367	0.2071	684.2971	8504.6465	95.9428
10.011	0.3225	18.5866	359.6418	4.0572
Sum		8864.2883	100.0000	

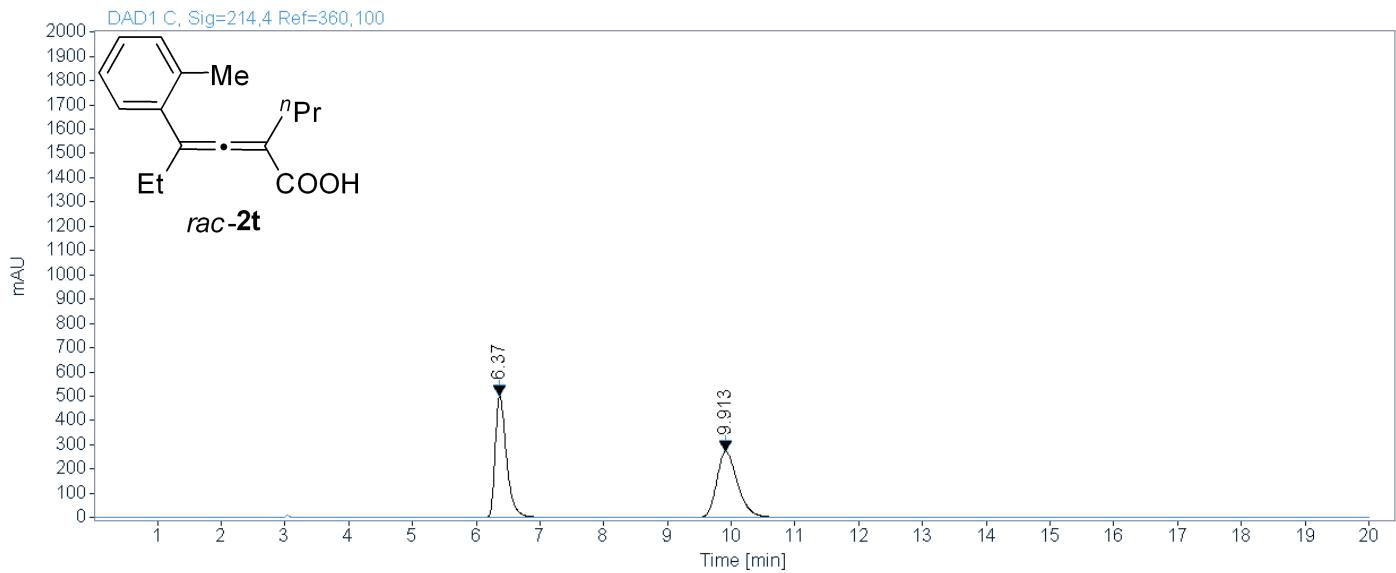
Area Percent Report



sample wj-1-083-2-rac-AS-H-98-2-
1.0-214

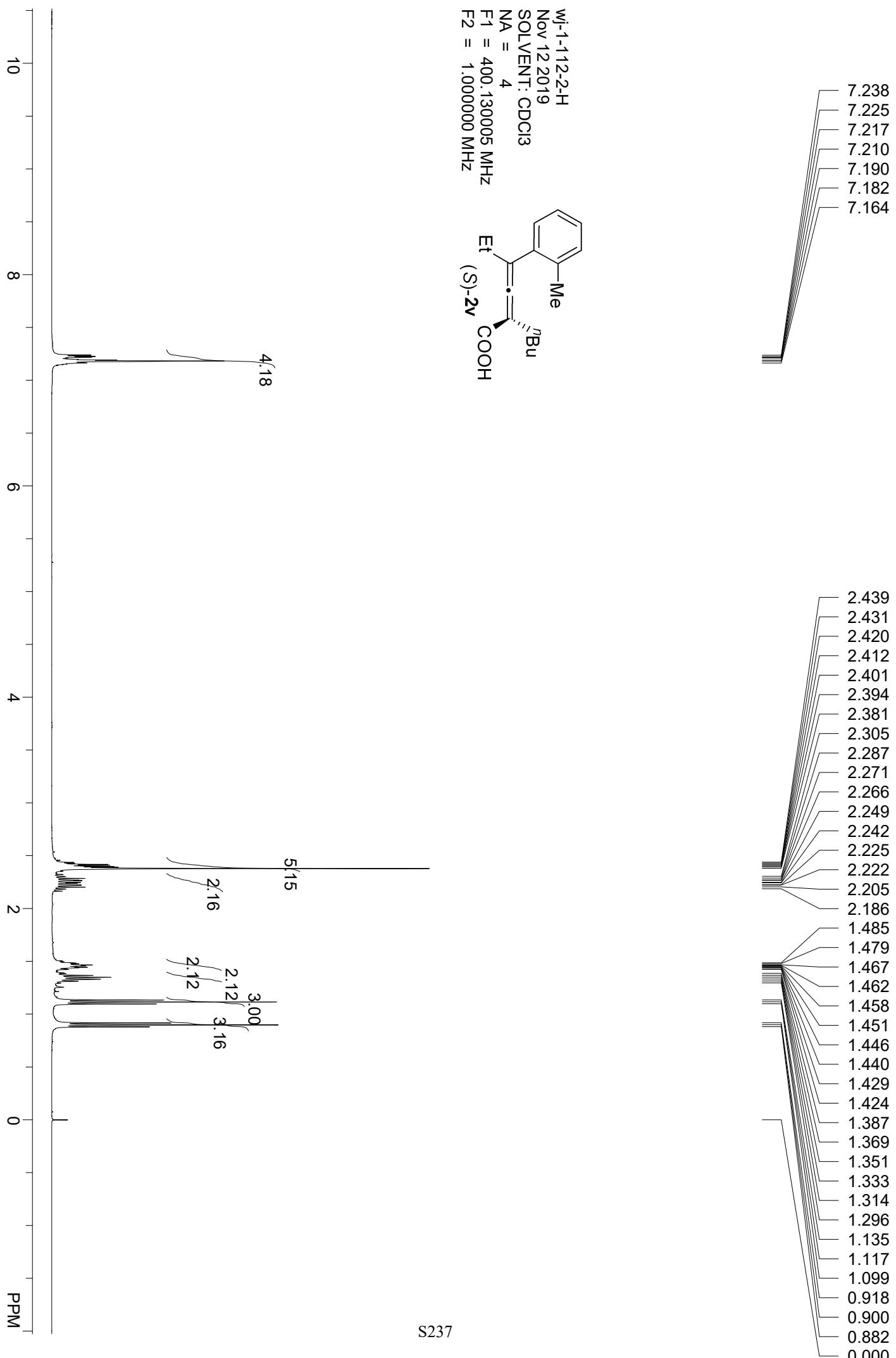
Data file: C:\Users\Public\Documents\ChemStation\1\Data\YuanYuan 2019-10-15 19-00-21\027-P2-C4-wj-1-083-2-rac.D

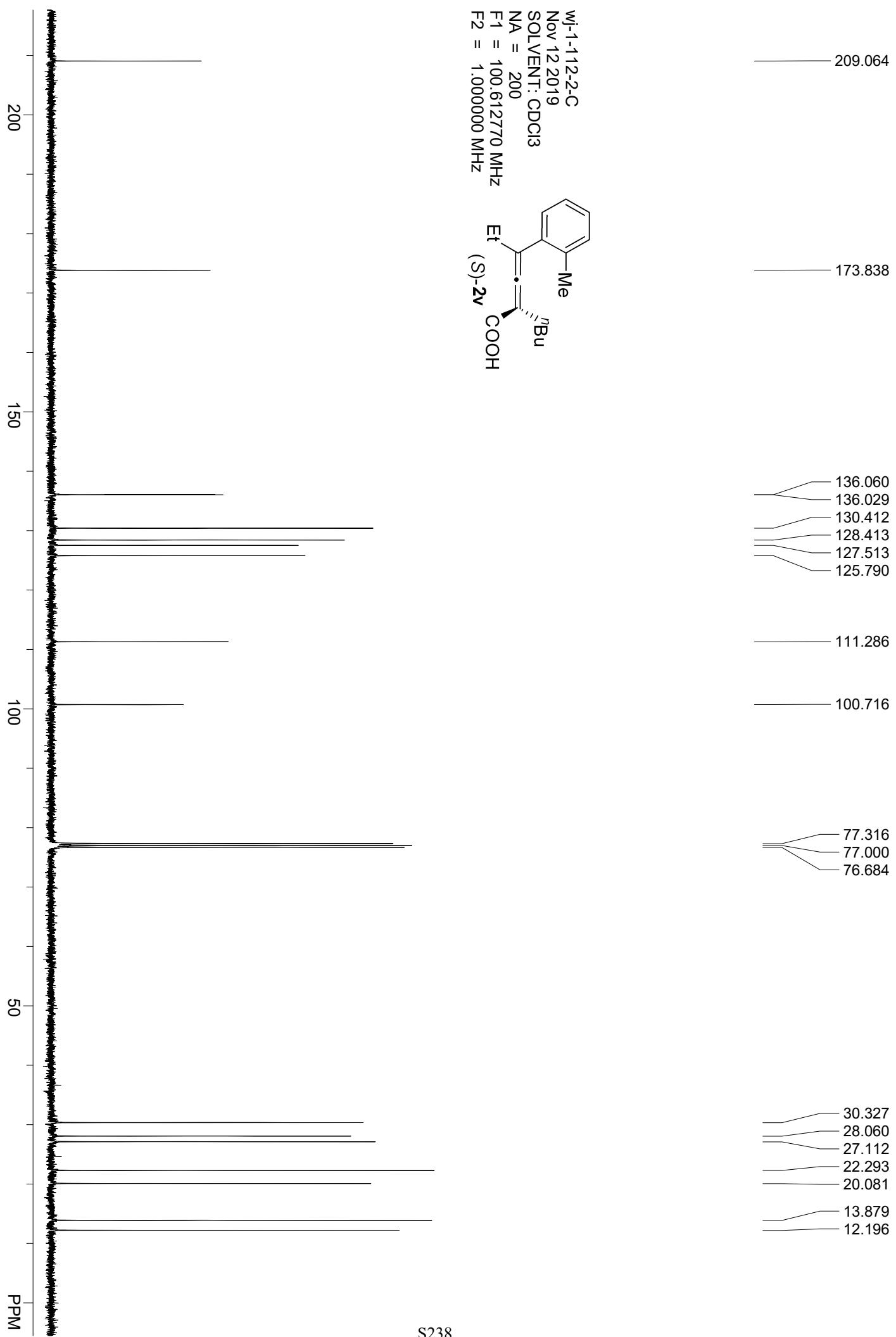
Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
6.370	0.1890	501.8834	6236.6787	50.1961
9.913	0.3523	271.1423	6187.9565	49.8039
Sum		12424.6353	100.0000	





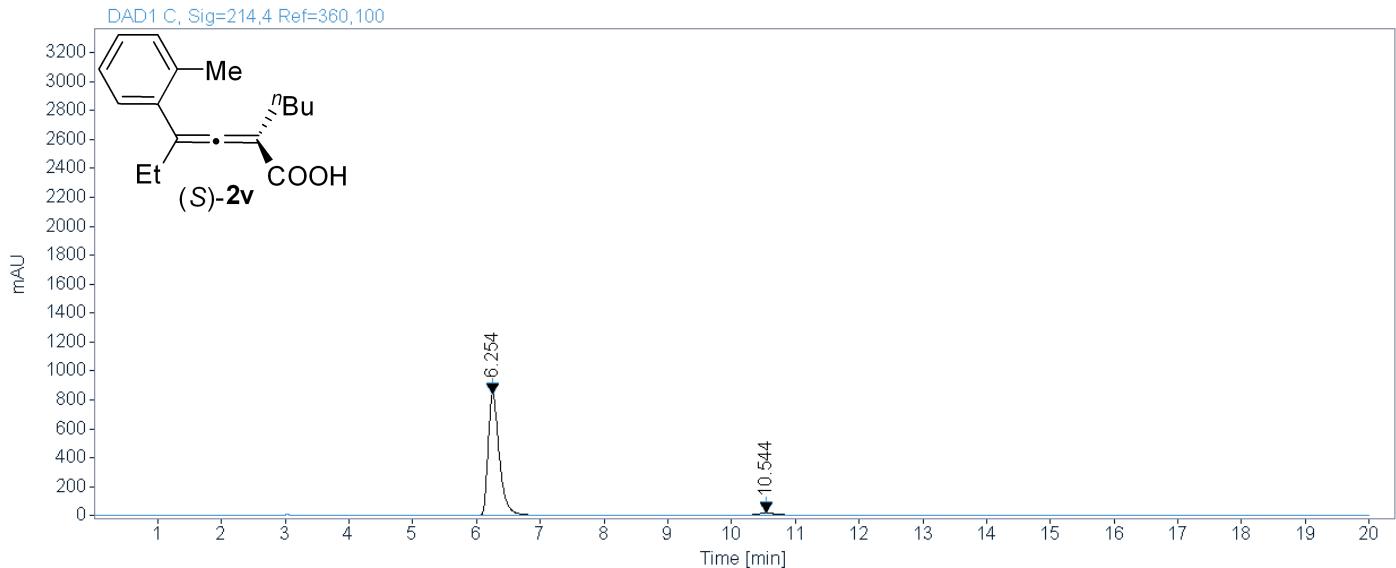
Area Percent Report



sample wj-1-112-2-AS-H-98-2-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\wgl 2019-11-12 21-55-01\006-P2-C1-wj-1-112-2.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
6.254	0.1877	842.0982	10373.5869	95.9732
10.544	0.4365	16.6175	435.2501	4.0268
Sum		10808.8370	100.0000	

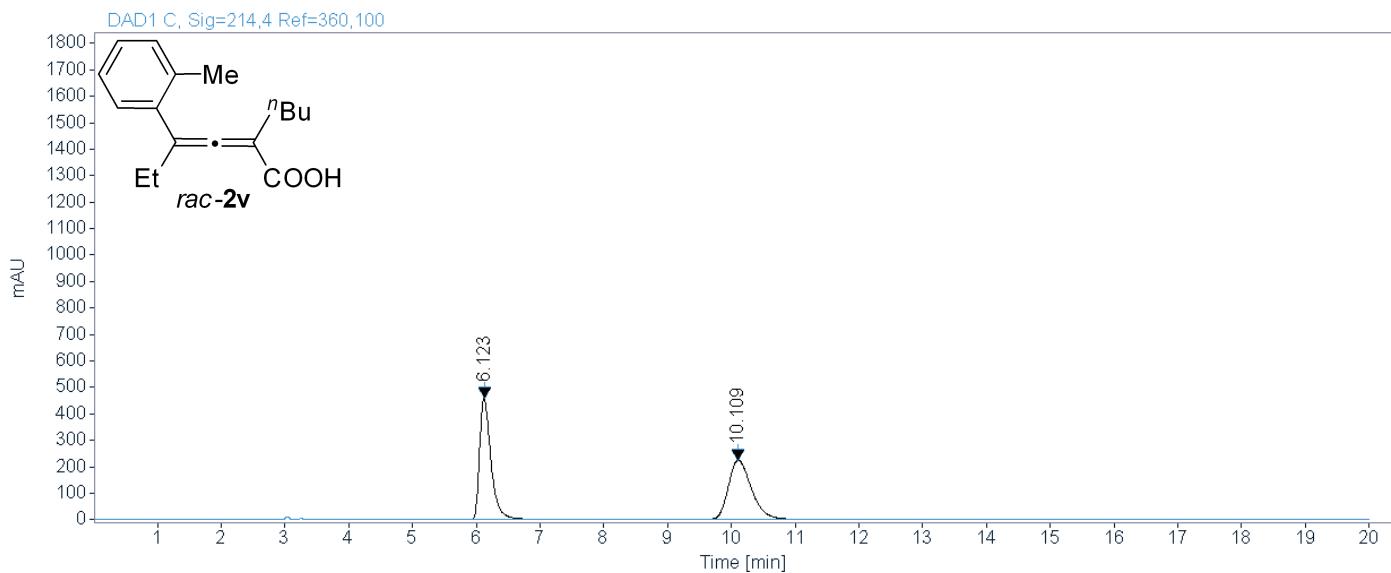
Area Percent Report



sample wj-1-112-2-rac-AS-H-98-2-1.0-214

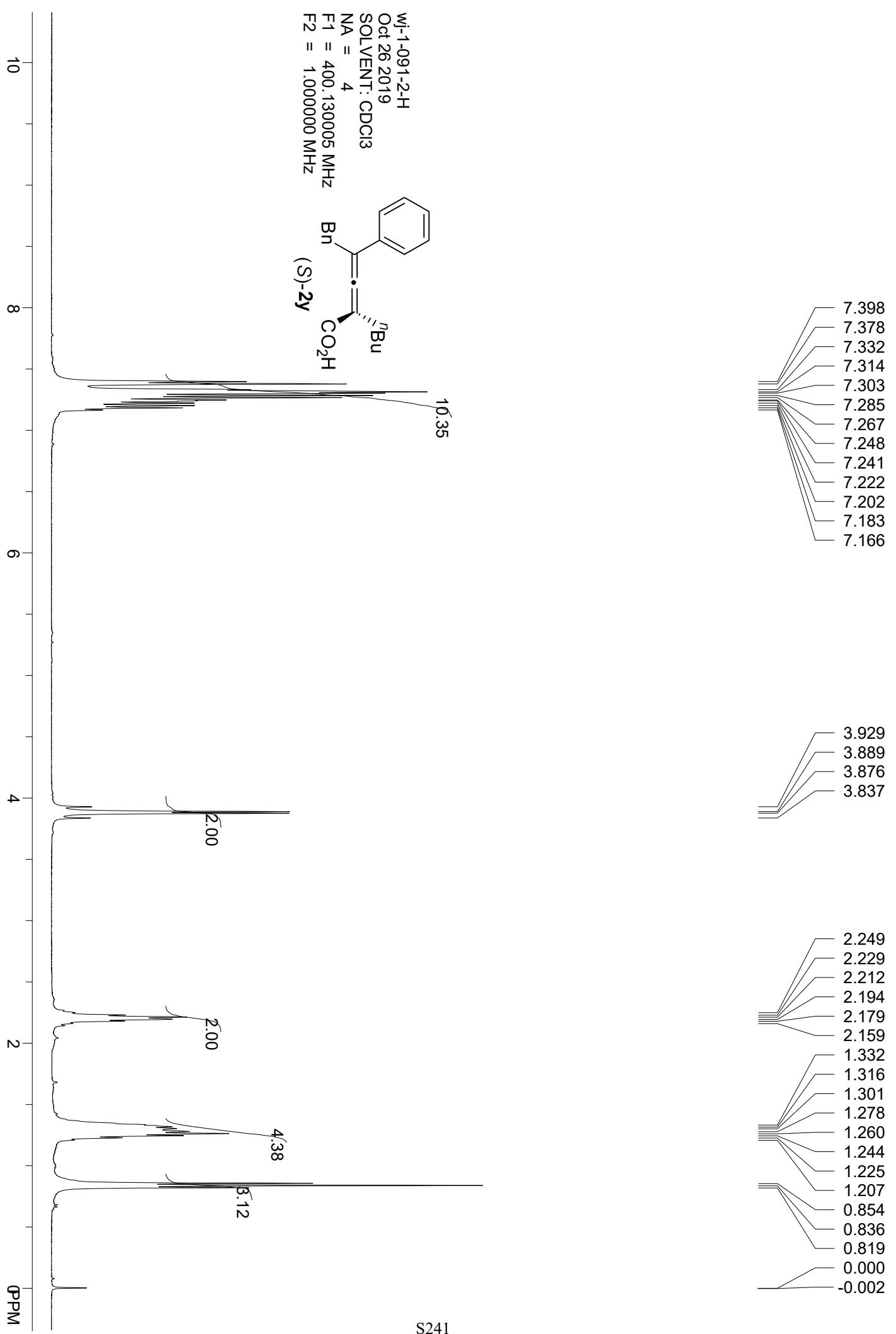
Data file: C:\Users\Public\Documents\ChemStation\1\Data\wgl 2019-11-12 21-55-01\007-P2-C2-wj-1-112-2-rac.D

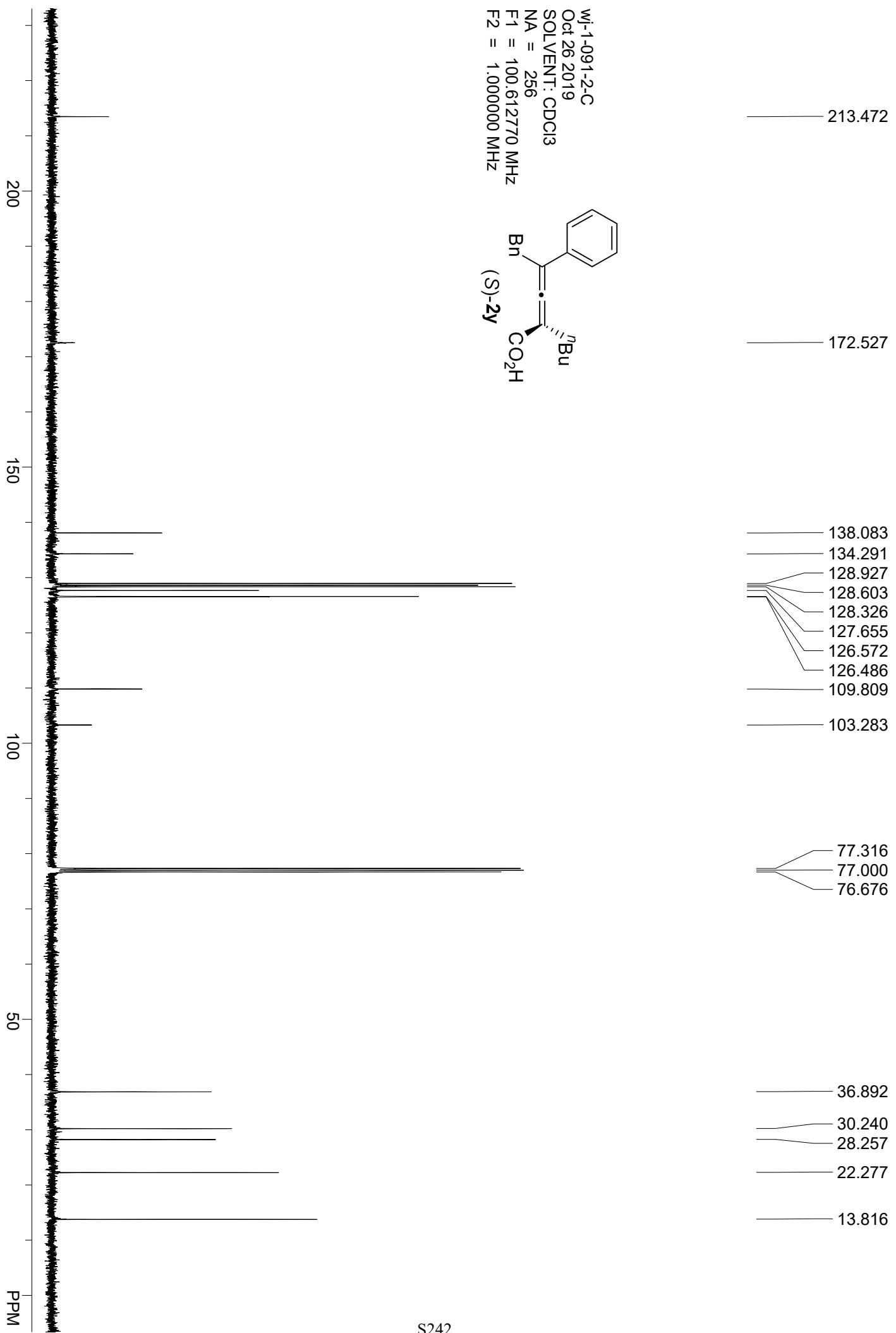
Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
6.123	0.1883	460.1762	5693.4077	49.8083
10.109	0.3920	224.6288	5737.2310	50.1917
Sum		11430.6387	100.0000	





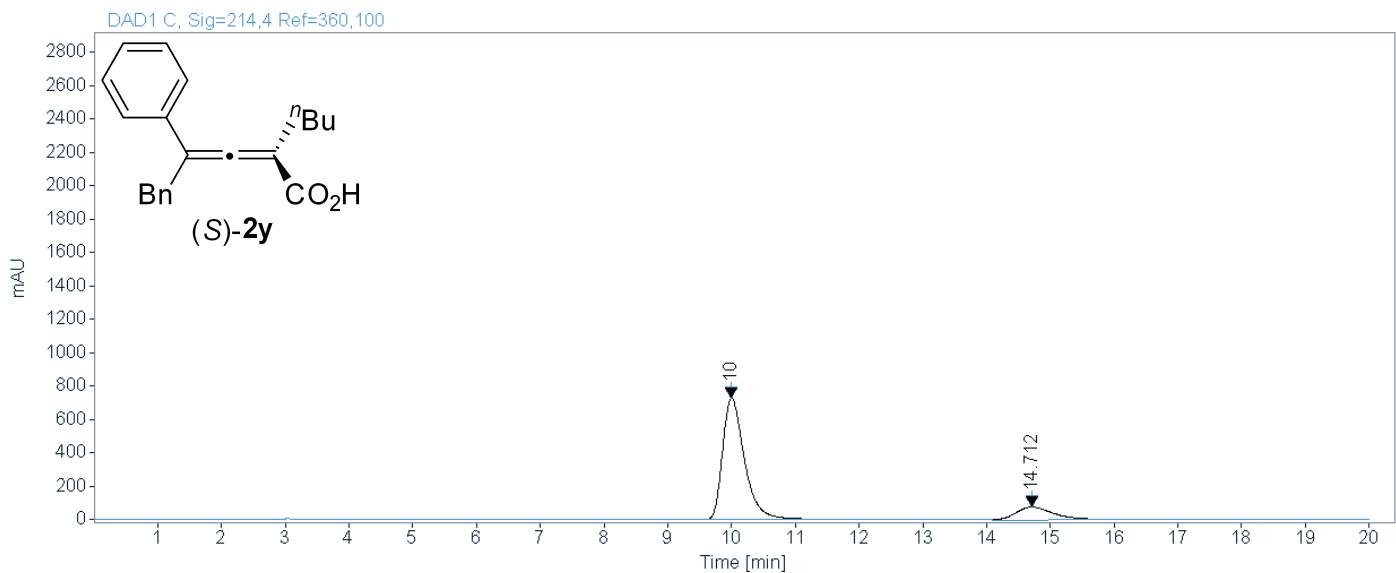
Area Percent Report



sample wj-1-091-2-AS-H-98-2-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\WJ_LC 2019-10-25 22-04-20\005-P2-C3-wj-1-091-2.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
10.000	0.3687	729.9028	17560.6309	85.4857
14.712	0.6611	75.1666	2981.5488	14.5143
		Sum	20542.1797	100.0000

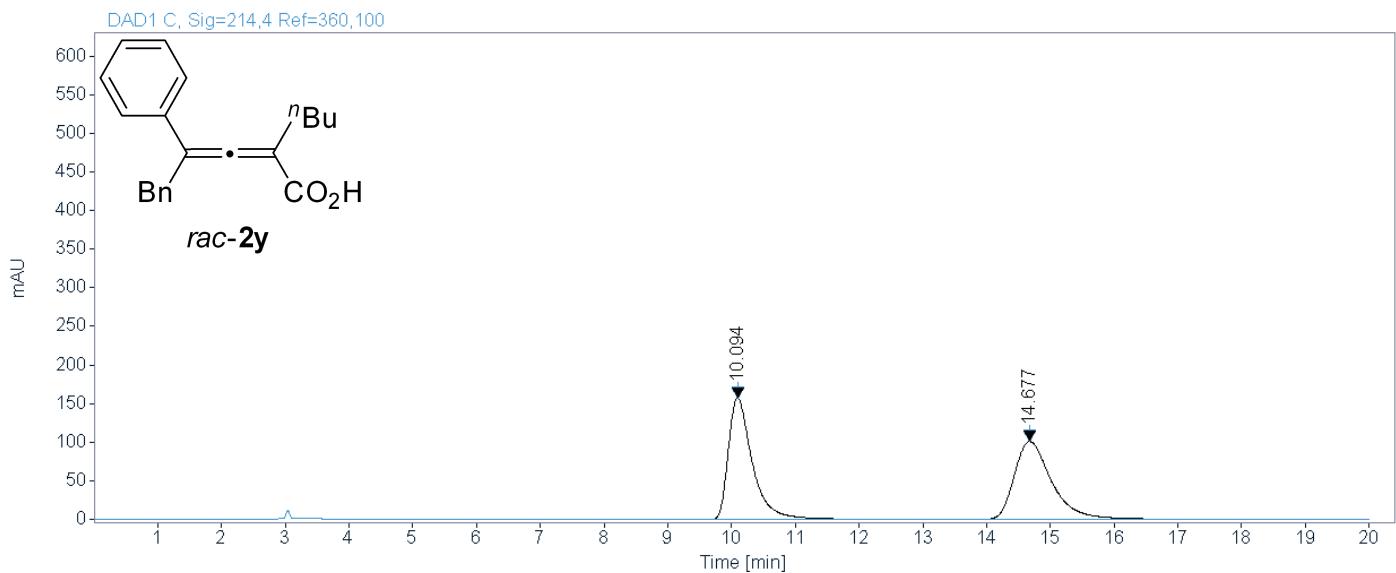
Area Percent Report



sample wj-1-091-2-rac-AS-H-98-2-1.0-214

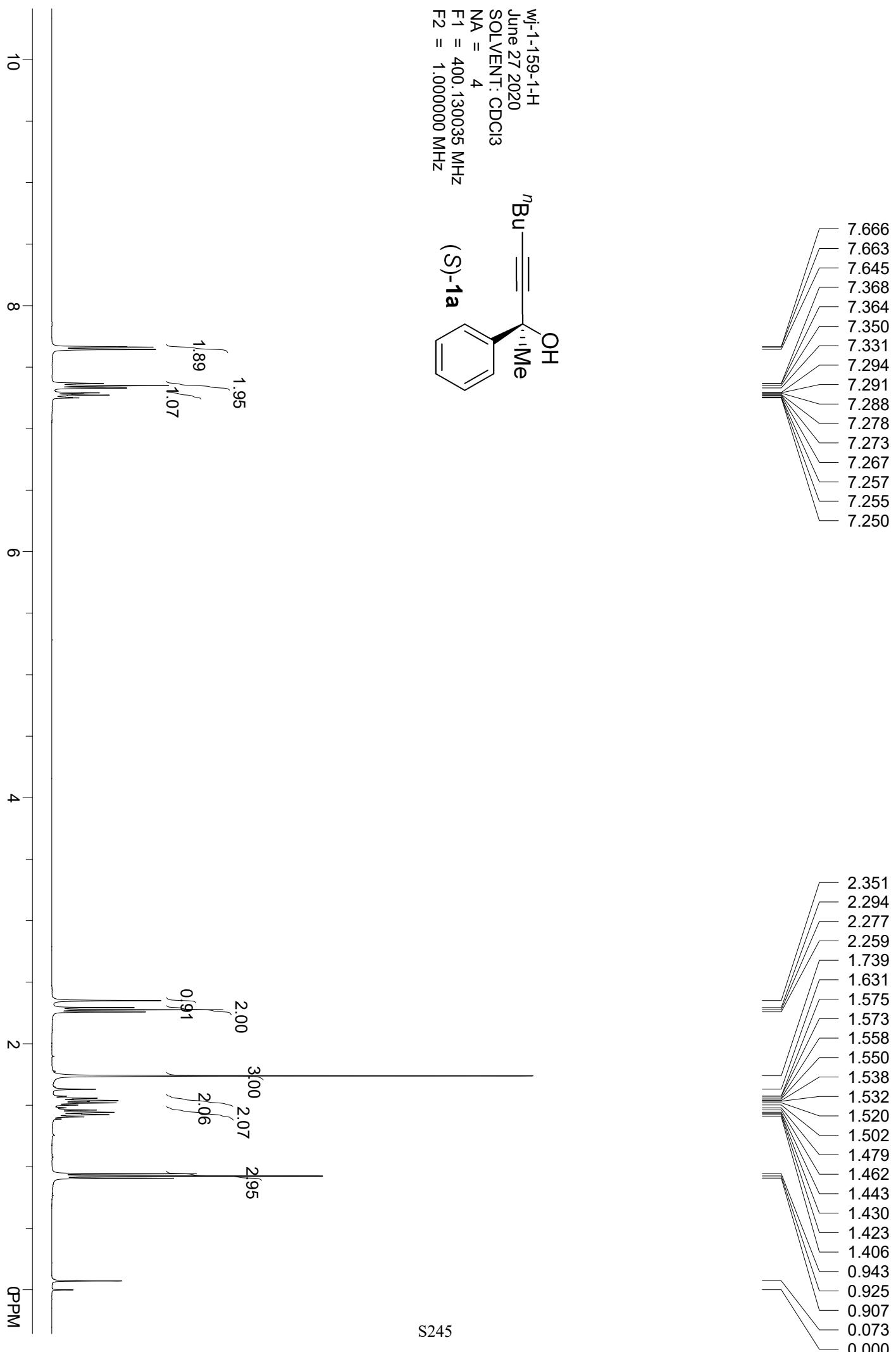
Data file: C:\Users\Public\Documents\ChemStation\1\Data\WJ_LC 2019-10-25 22-04-20\006-P2-C4-wj-1-091-2-rac.D

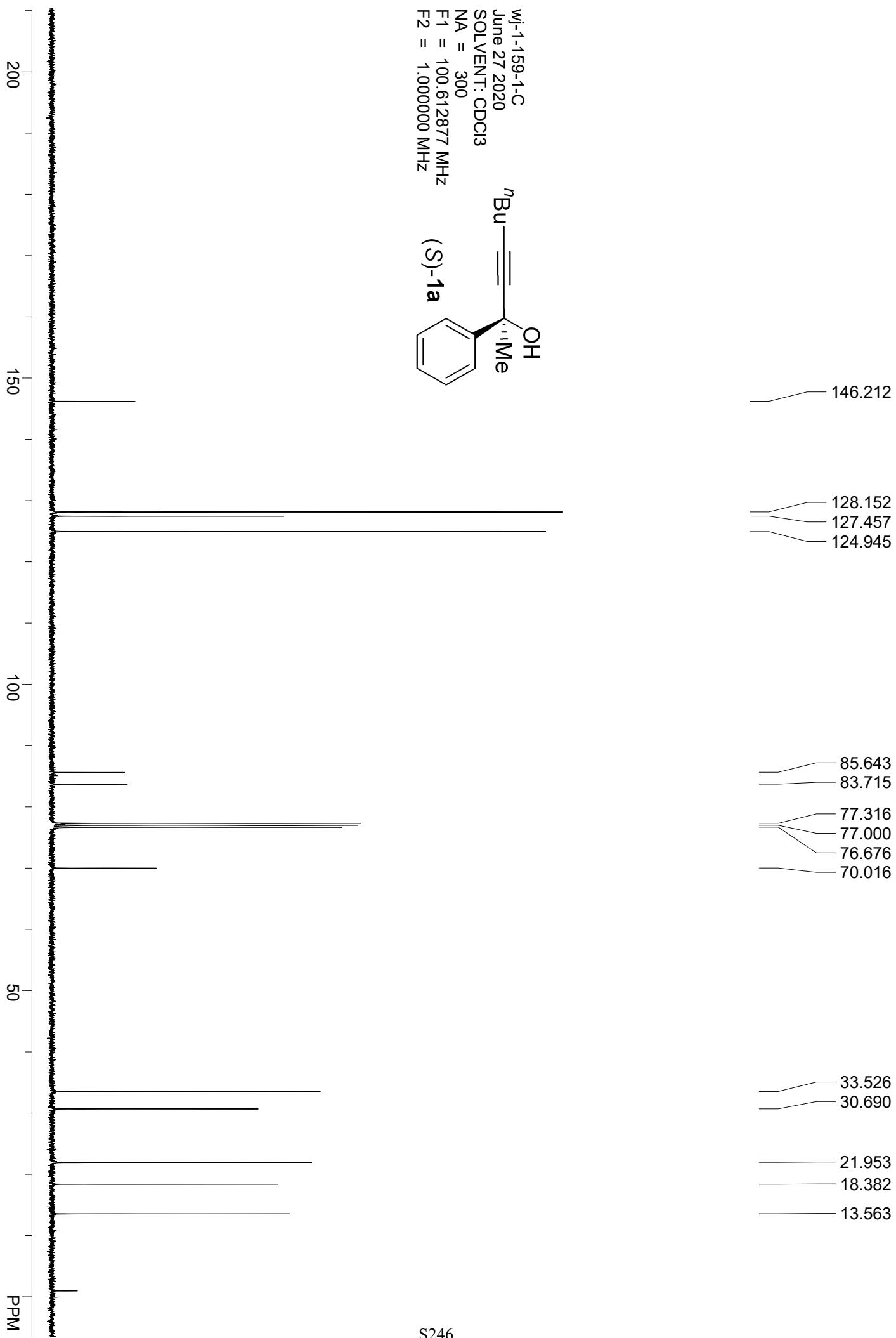
Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
10.094	0.3980	157.7003	4190.3838	50.0406
14.677	0.6270	101.0590	4183.5767	49.9594
Sum		8373.9604	100.0000	





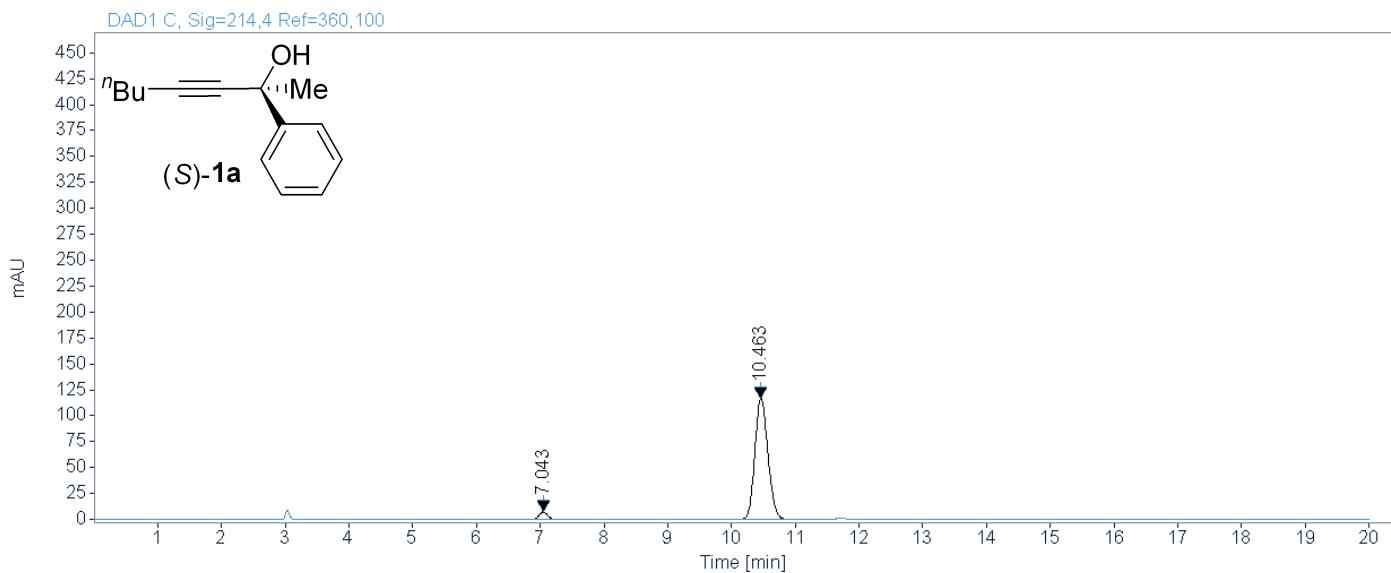
Area Percent Report



sample wj-1-159-1-AS-H-98-2-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\zwf-allenioc acid_LC 2020-06-27 10-15-53\027-P2-C1-wj-1-159-1.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
7.043	0.1404	7.4554	67.9760	3.8981
10.463	0.2225	117.4467	1675.8452	96.1019
		Sum	1743.8212	100.0000

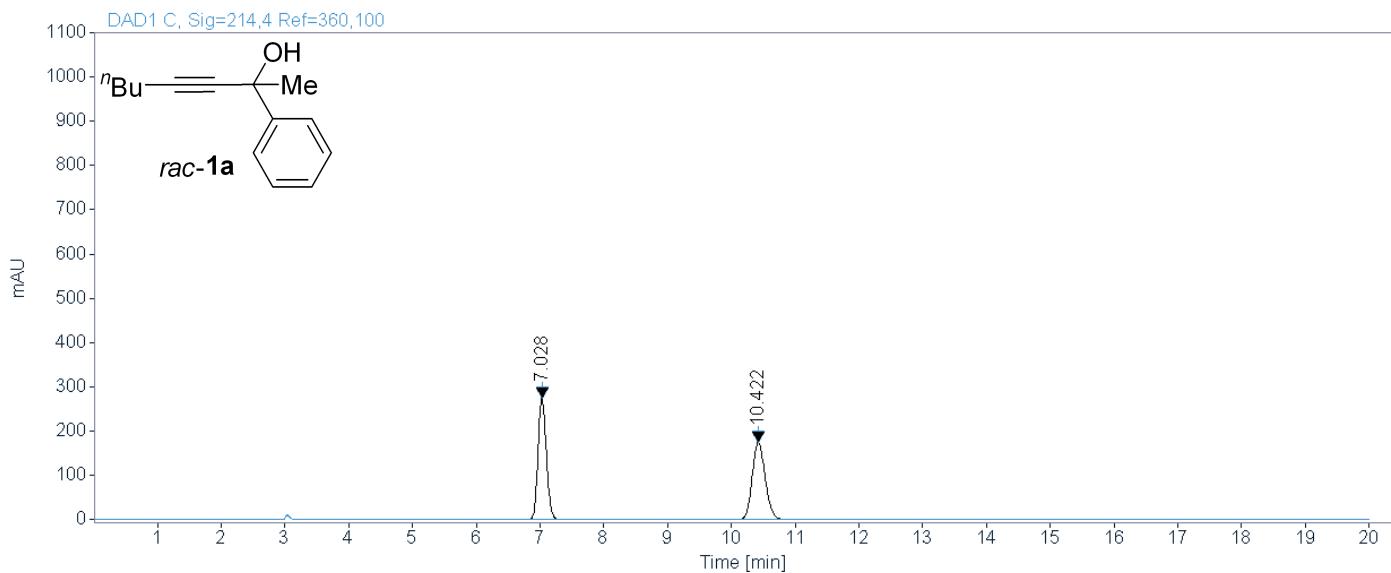
Area Percent Report



sample wj-1-159-1-rac-AS-H-98-2-1.0-214

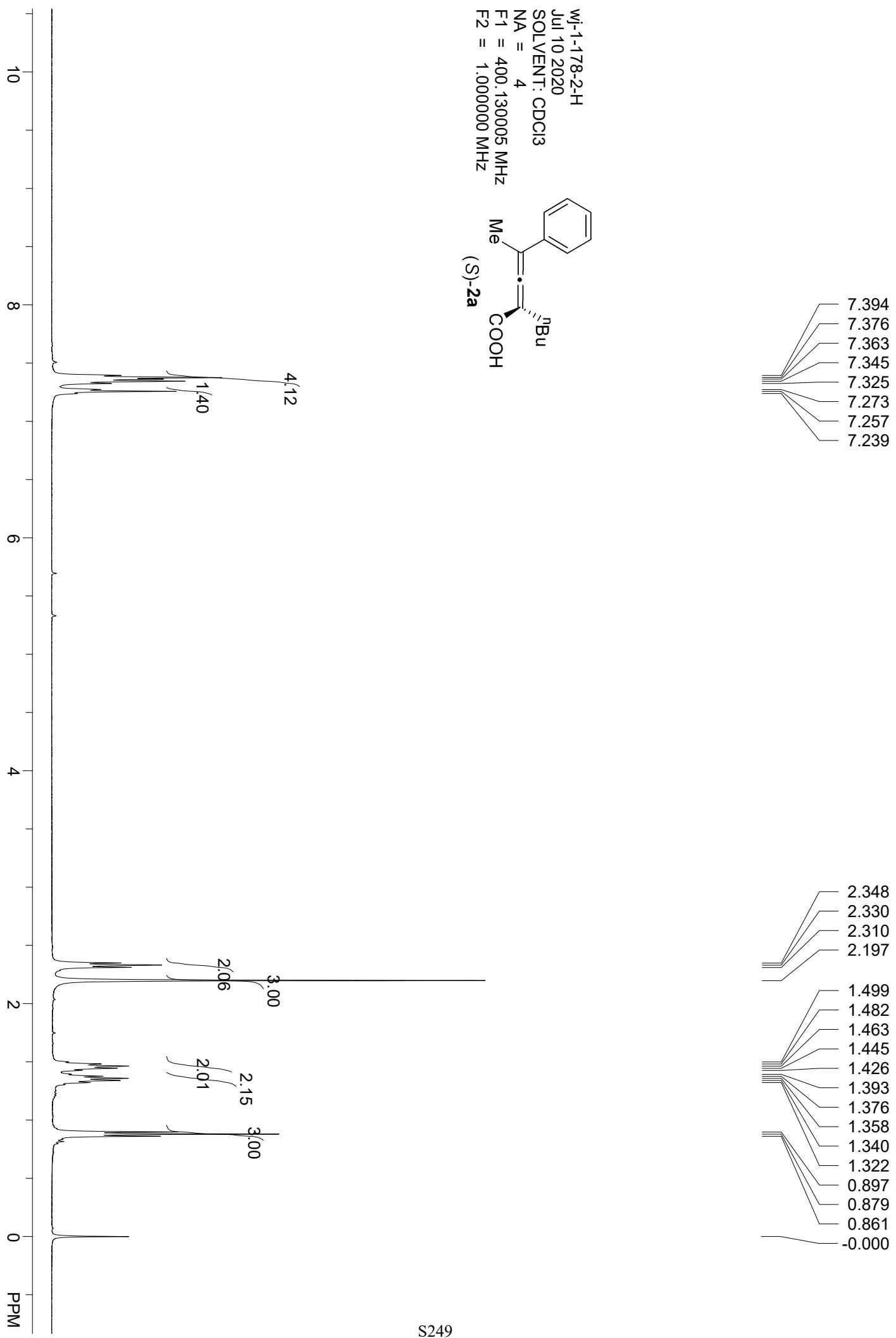
Data file: C:\Users\Public\Documents\ChemStation\1\Data\zwf-alleniac acid_LC 2020-06-27 10-15-53\028-P2-C2-wj-1-159-1-rac.D

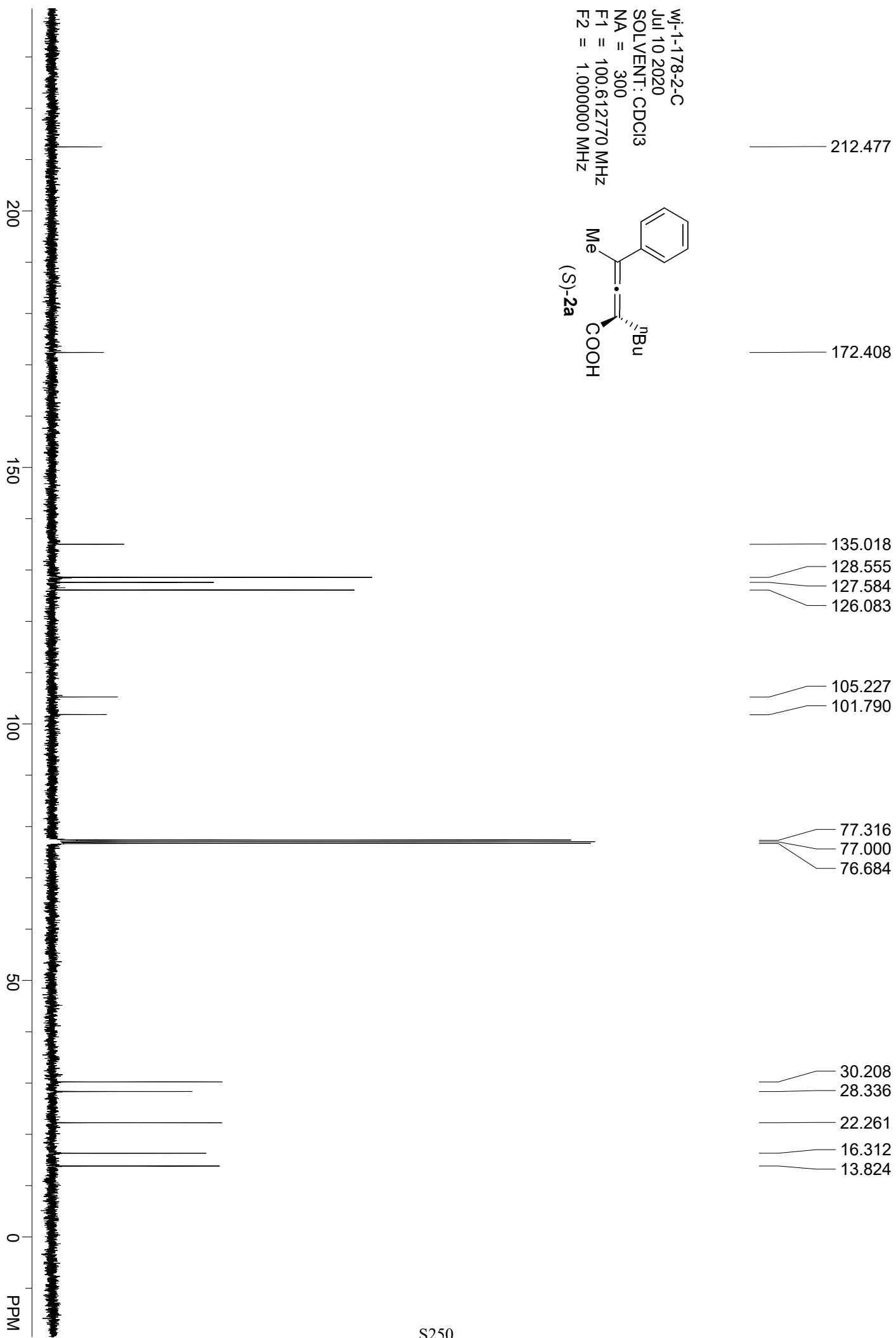
Acquisition Data:

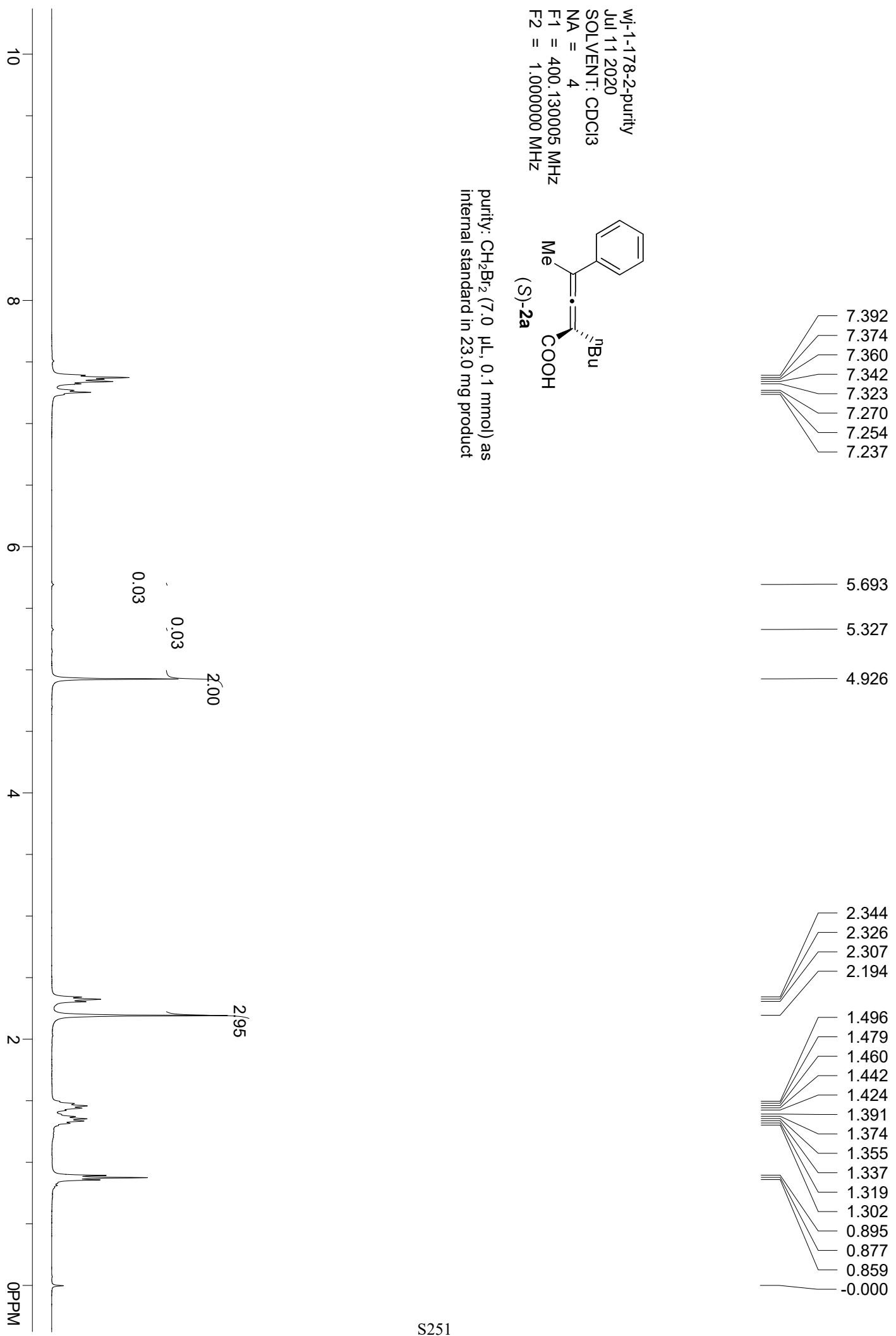


Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
7.028	0.1410	275.4709	2479.3728	49.8828
10.422	0.2223	174.7387	2491.0229	50.1172
Sum		4970.3958	100.0000	







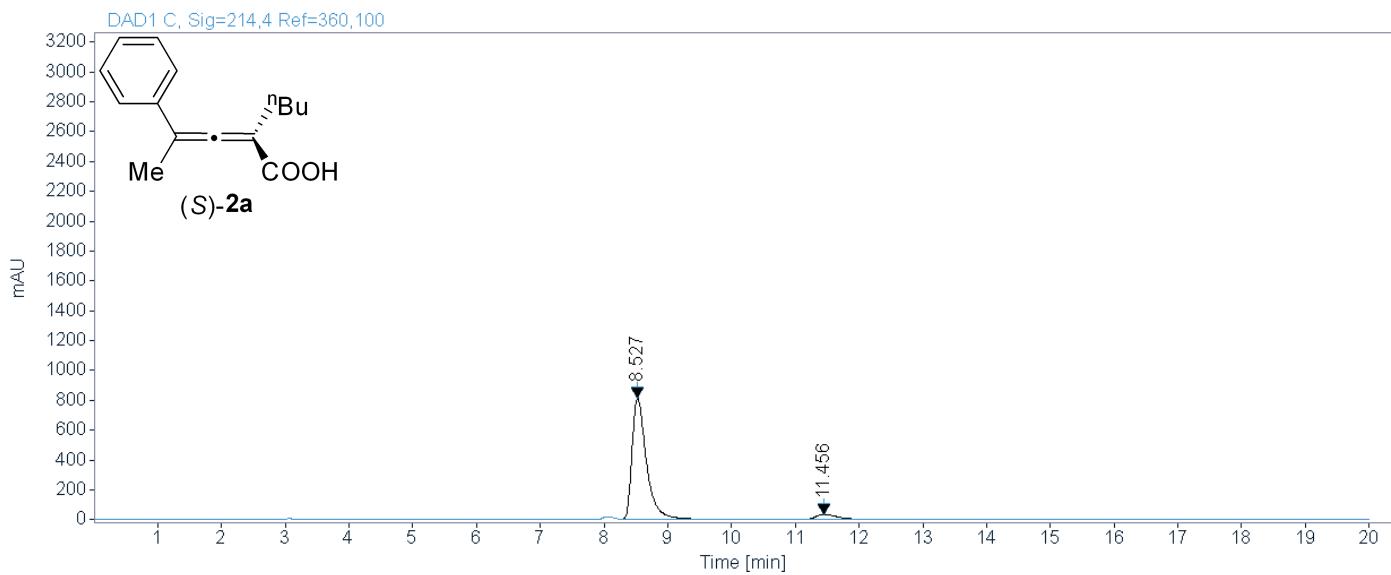
Area Percent Report



sample wj-1-178-2-AS-H-98-2-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\WJ_LC 2020-07-09 11-27-12\031-P2-C3-wj-1-178-2.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
8.527	0.2622	815.7829	12831.7021	94.9816
11.456	0.3532	31.9884	677.9717	5.0184
Sum		13509.6739	100.0000	

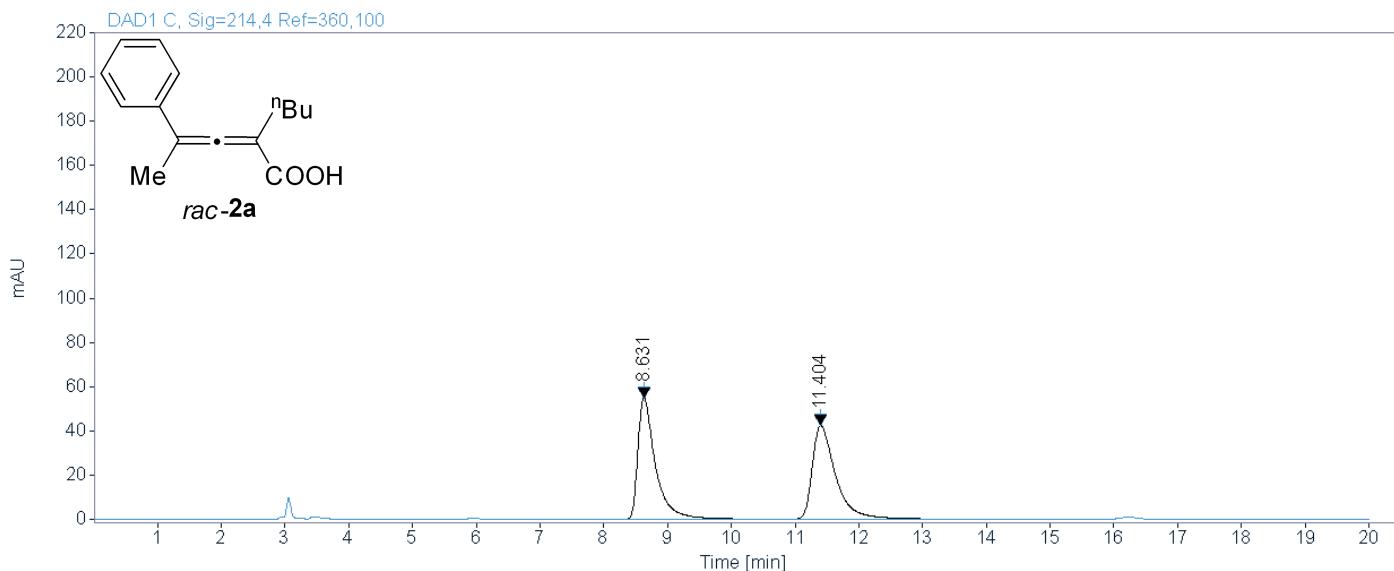
Area Percent Report



sample wj-1-178-2-rac-AS-H-98-2-1.0-214

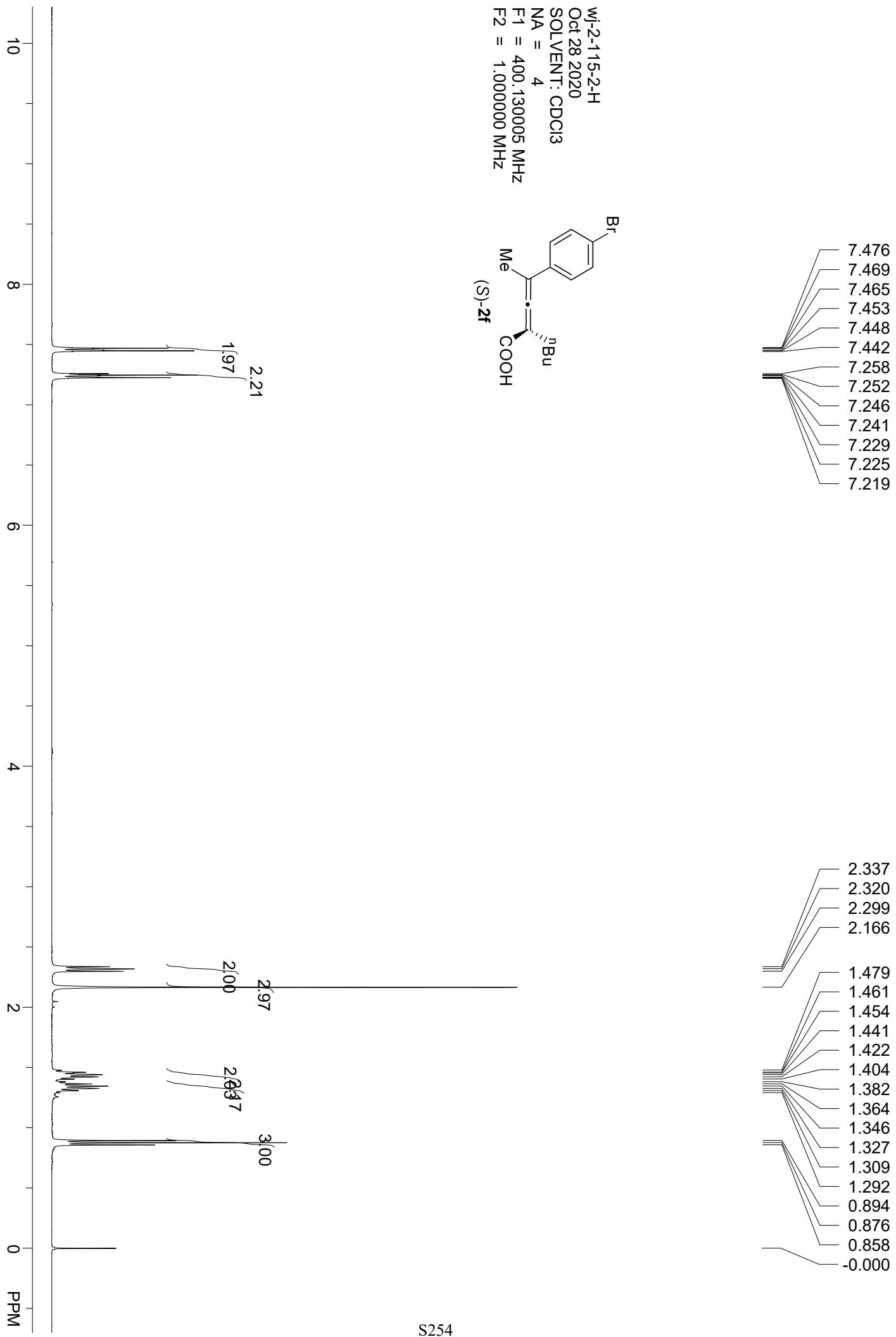
Data file: C:\Users\Public\Documents\ChemStation\1\Data\WJ_LC 2020-07-09 11-27-12\032-P2-C4-wj-1-178-2-rac.D

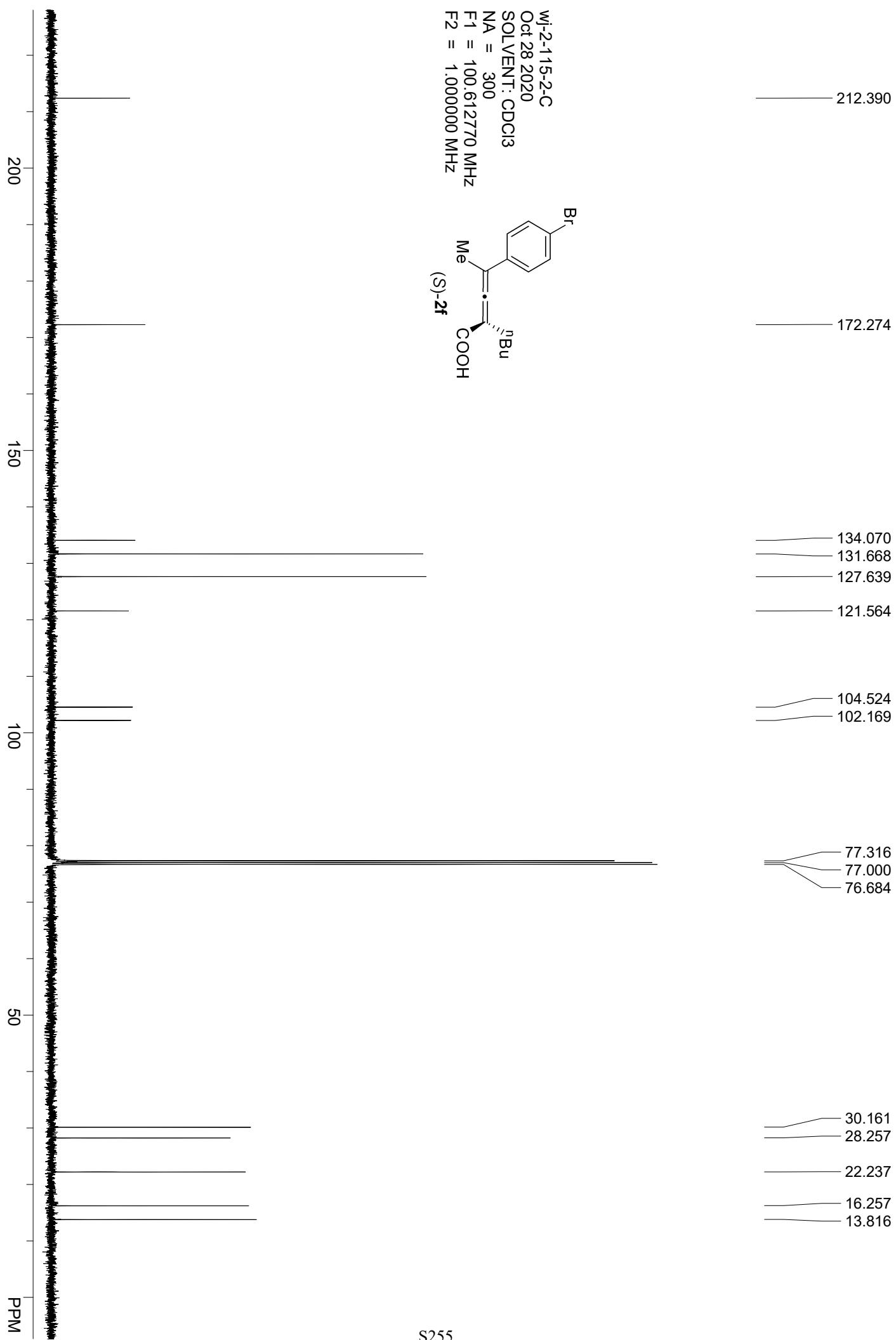
Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
8.631	0.2858	54.9920	1064.5034	49.6288
11.404	0.3785	42.5138	1080.4259	50.3712
Sum		2144.9293	100.0000	





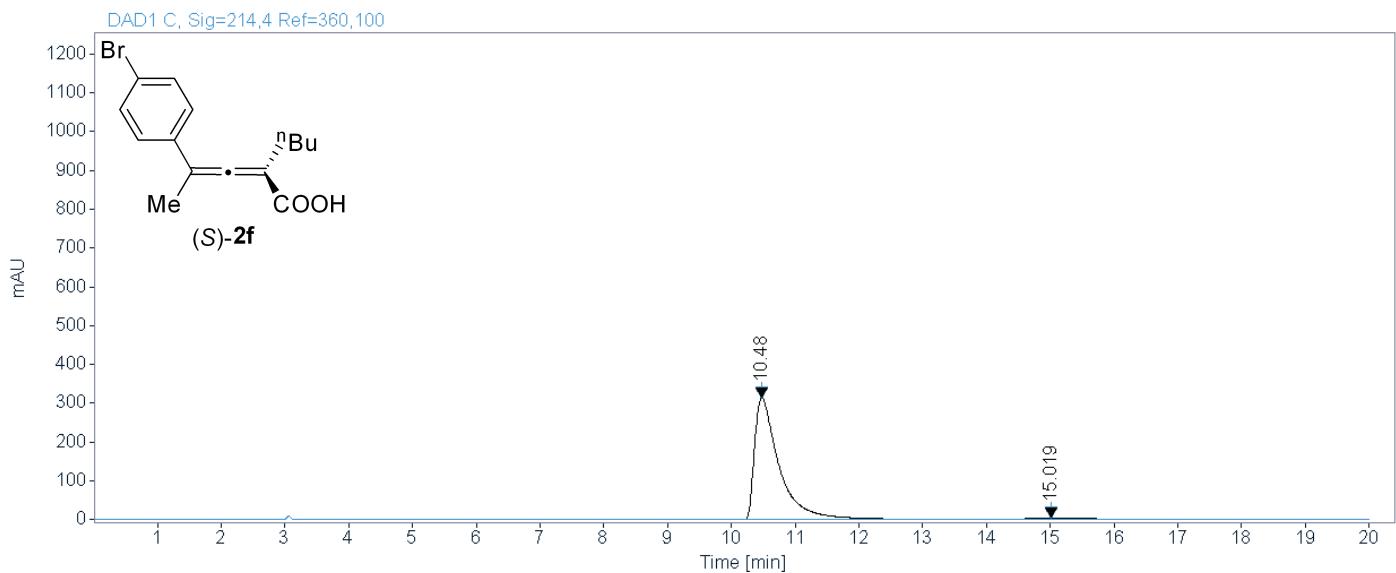
Area Percent Report



sample wj-2-115-2-AS-H-98-2-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\WHN 2020-10-27 22-39-12\036-P2-C3-wj-2-115-2.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
10.480	0.4499	314.0293	8475.9814	97.2368
15.019	1.0441	3.8450	240.8650	2.7632
Sum		314.0293	8716.8465	100.0000

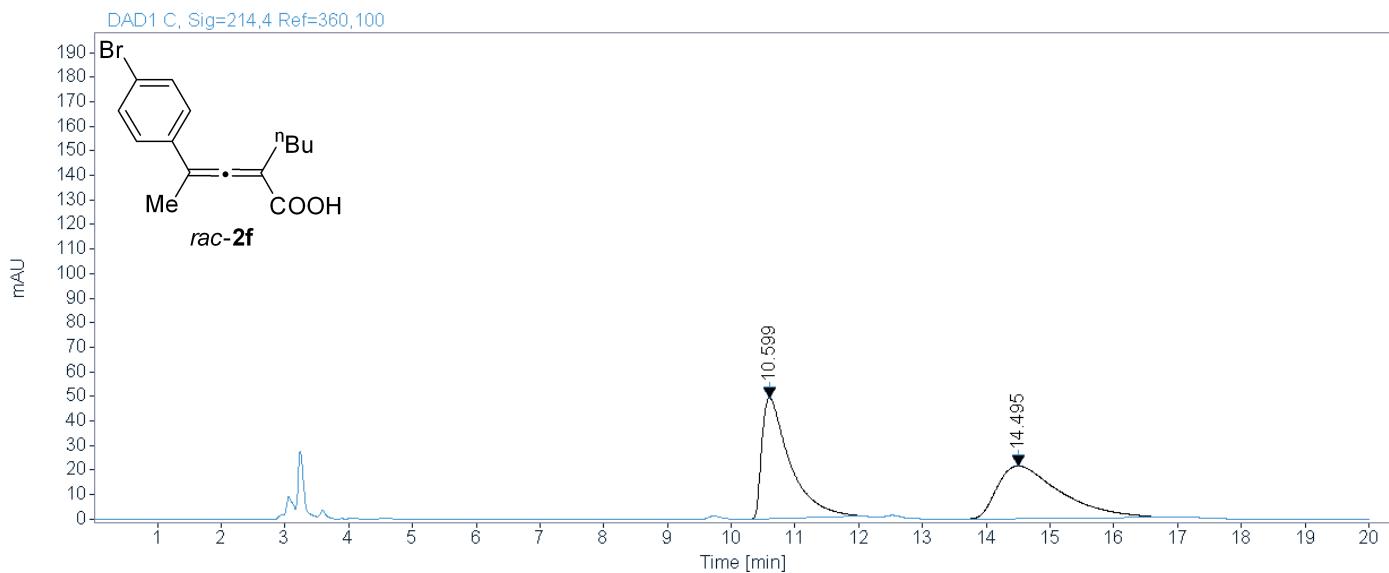
Area Percent Report



sample wj-2-115-2-rac-AS-H-98-2-1.0-214

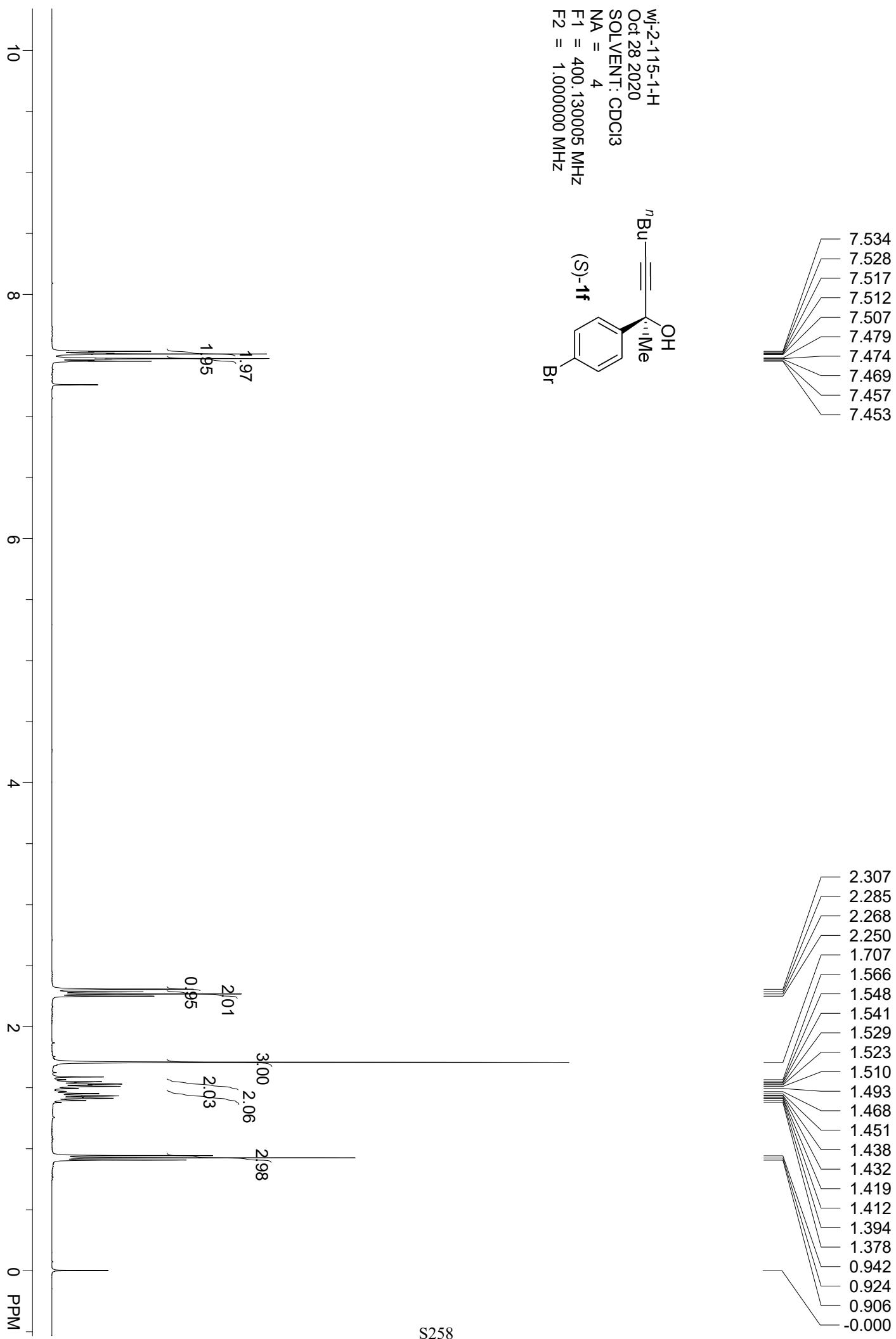
Data file: C:\Users\Public\Documents\ChemStation\1\Data\WHN 2020-10-27 22-39-12\037-P2-C4-wj-2-115-2-rac.D

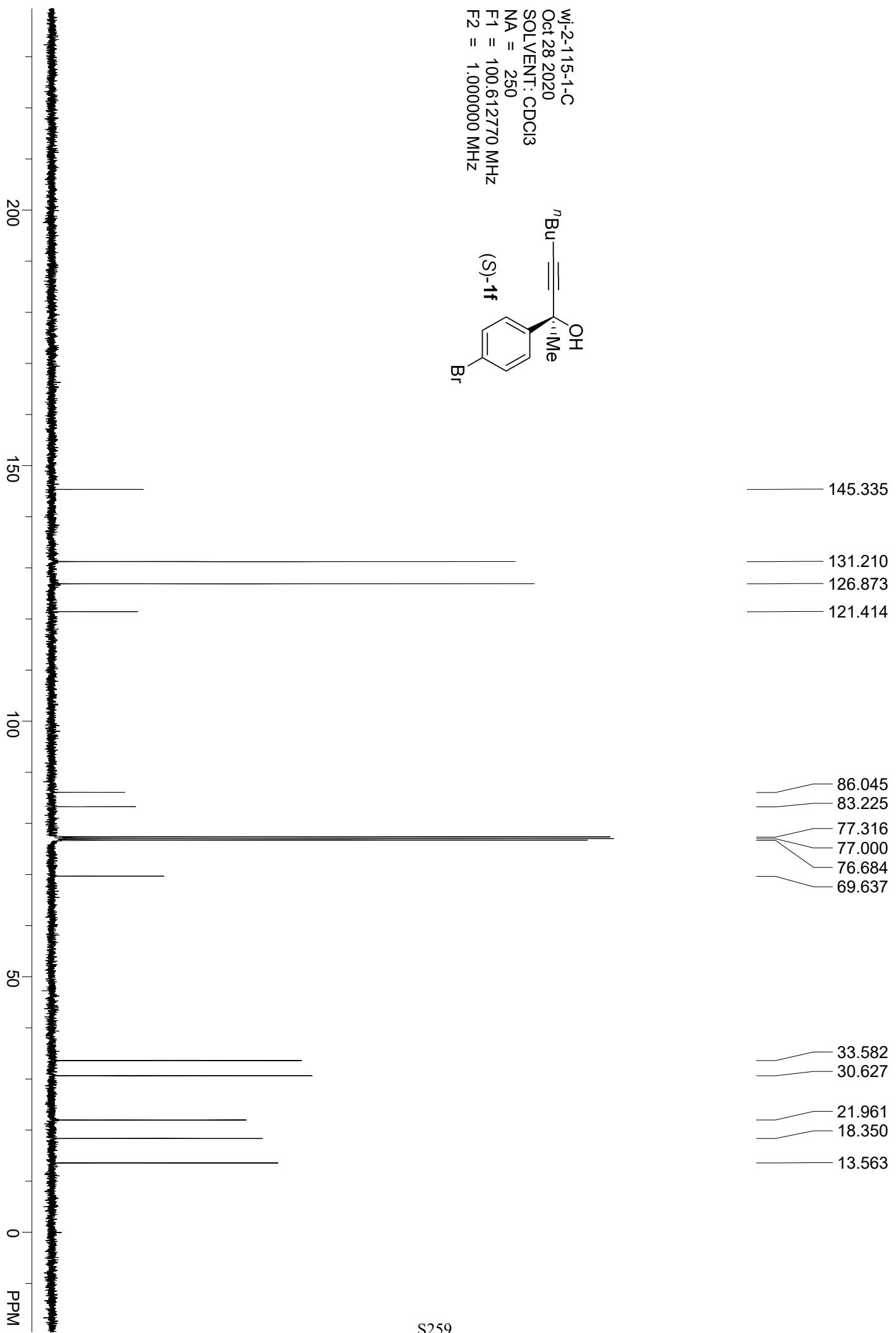
Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
10.599	0.5087	49.1622	1500.5134	50.1570
14.495	1.0269	21.4538	1491.1208	49.8430
Sum		2991.6343	100.0000	





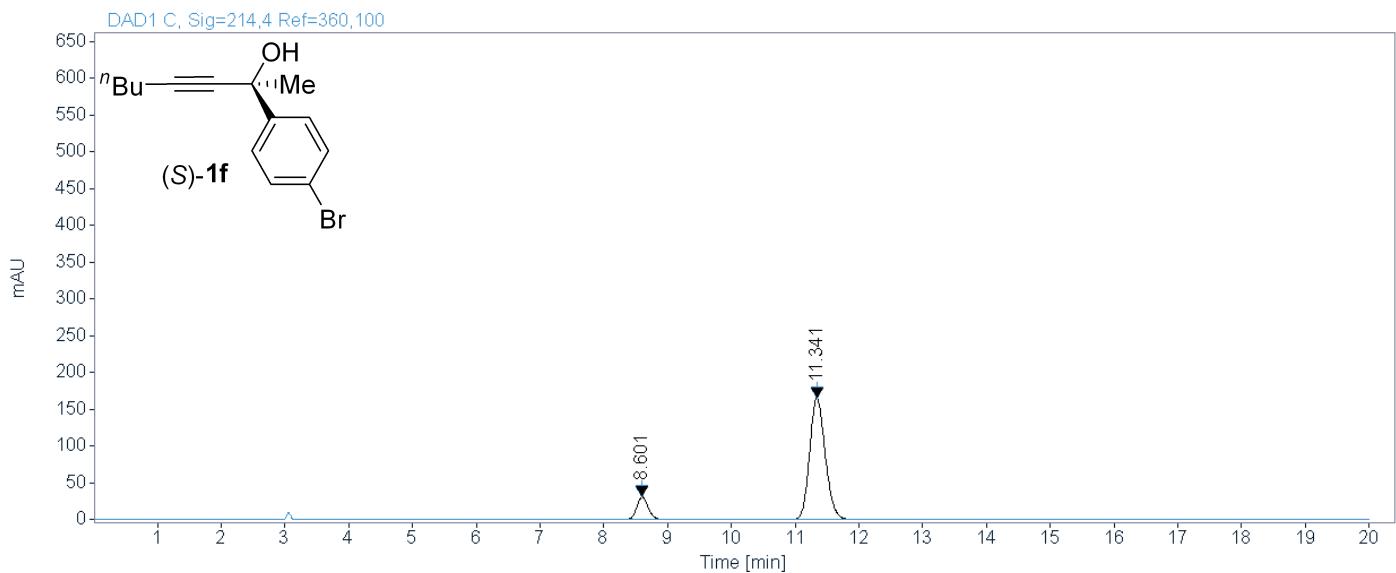
Area Percent Report



sample wj-2-115-1-AS-H-98-2-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\WHN 2020-10-27 22-39-12\034-P2-C1-wj-2-115-1.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
8.601	0.1969	30.7501	392.3292	12.1958
11.341	0.2630	165.6306	2824.5901	87.8042
Sum		3216.9193	100.0000	

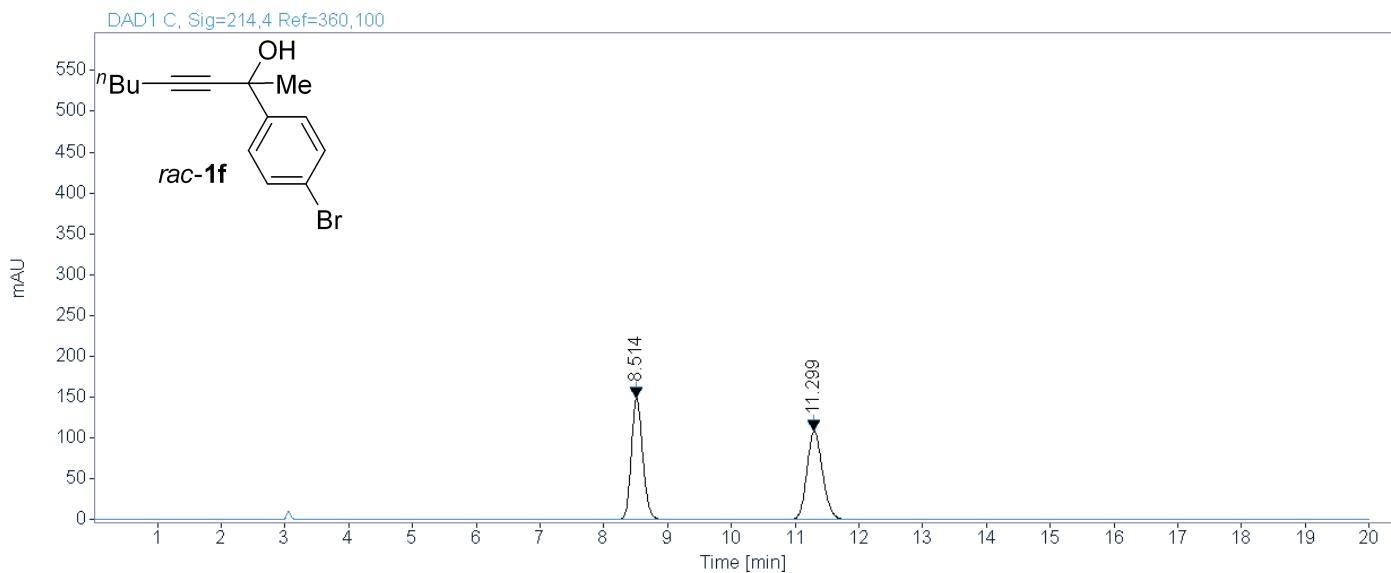
Area Percent Report



sample wj-2-115-1-rac-AS-H-98-2-1.0-214

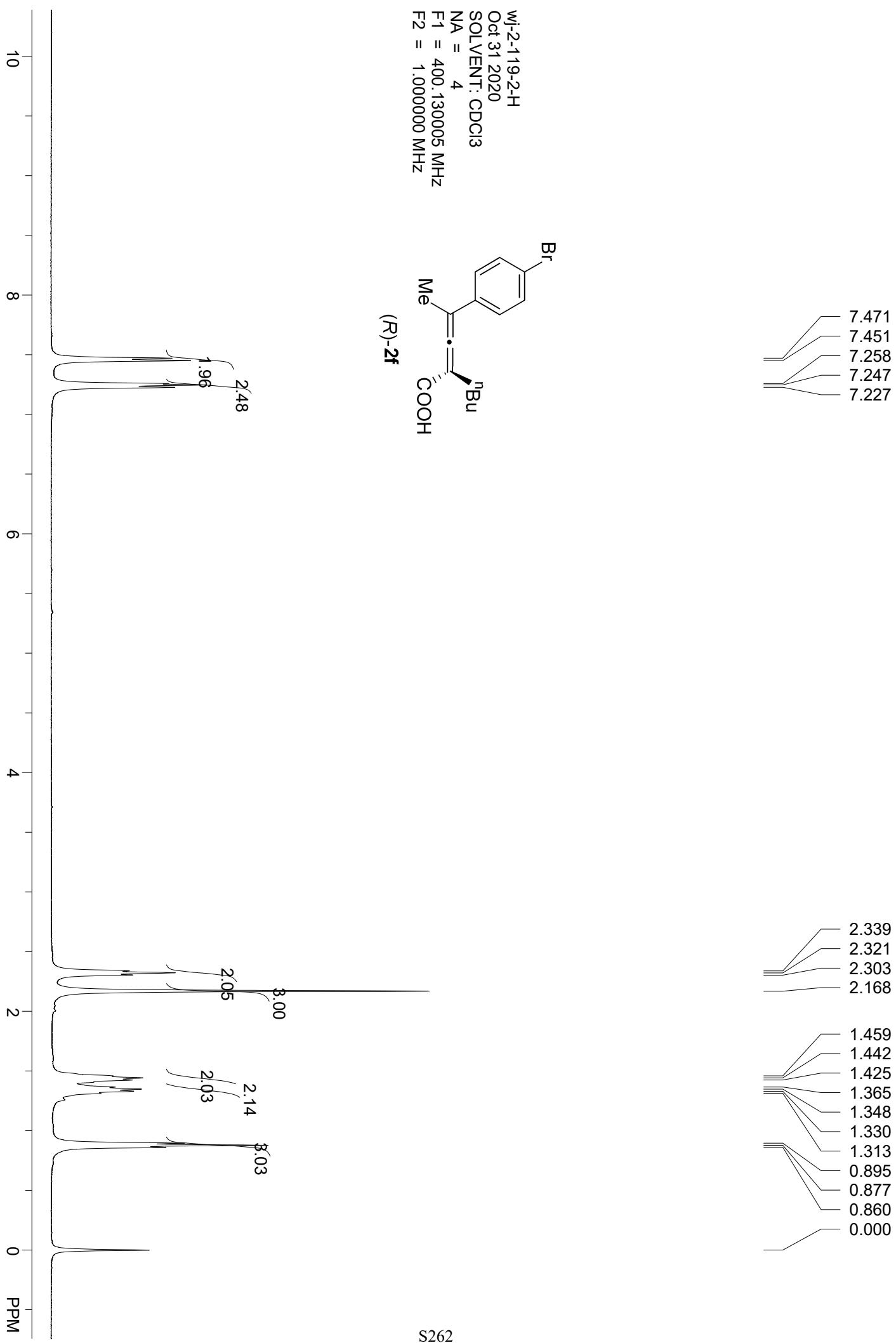
Data file: C:\Users\Public\Documents\ChemStation\1\Data\WHN 2020-10-27 22-39-12\035-P2-C2-wj-2-115-1.rac.D

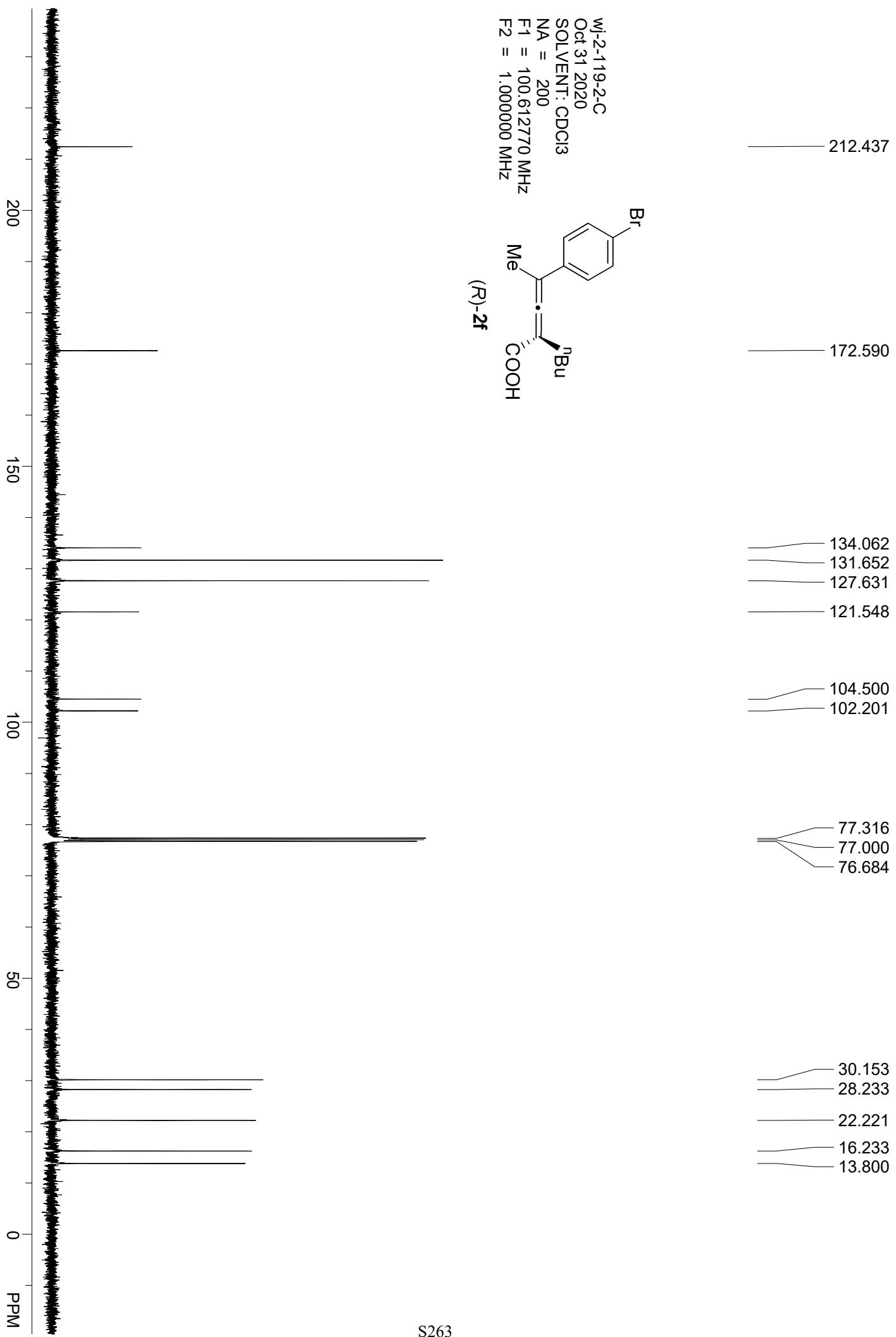
Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
8.514	0.1930	149.1293	1853.8256	50.0083
11.299	0.2633	108.4760	1853.2098	49.9917
Sum		3707.0354	100.0000	





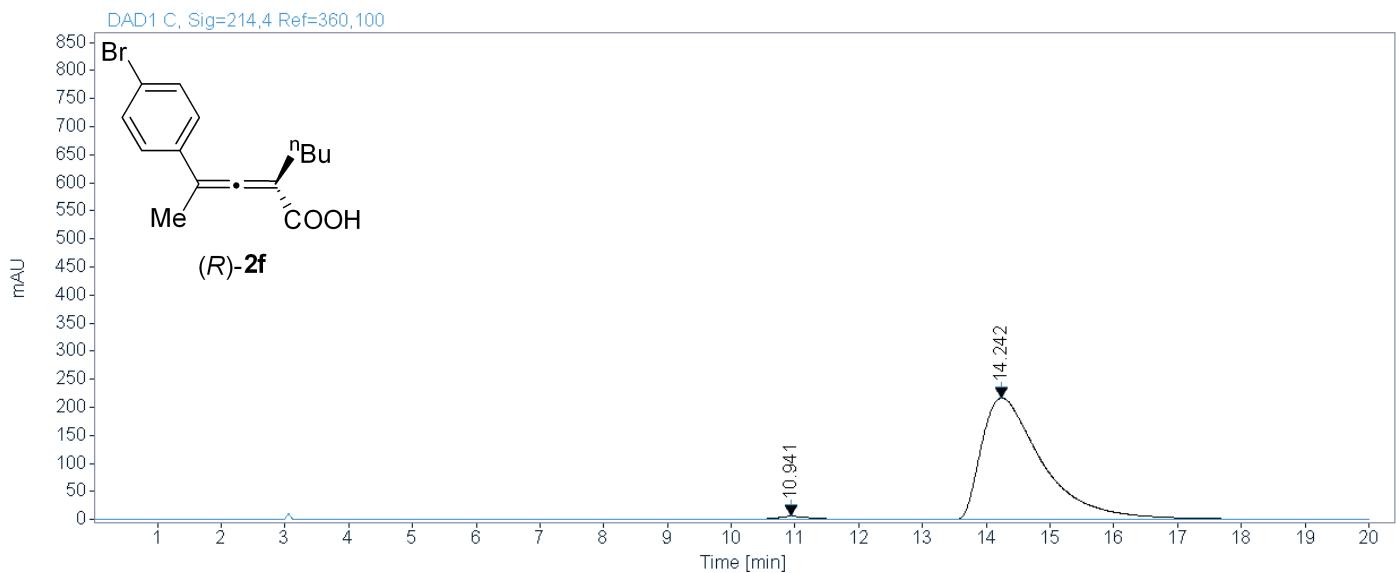
Area Percent Report



sample wj-2-119-2-AS-H-98-2-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\WJ_LC 2020-10-31 17-12-21\004-P2-C3-wj-2-119-2.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
10.941	0.5602	5.8755	235.7755	1.6035
14.242	1.0260	216.9615	14467.9209	98.3965
Sum		14703.6964	100.0000	

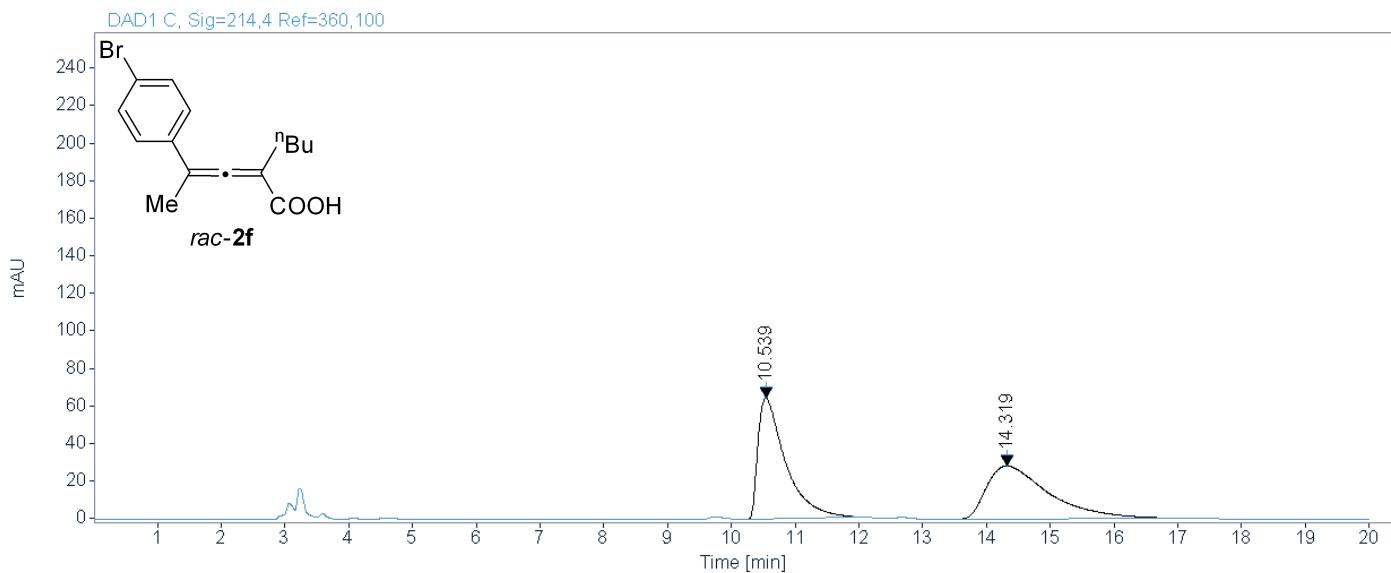
Area Percent Report



sample wj-2-119-2-rac-AS-H-98-2-1.0-214

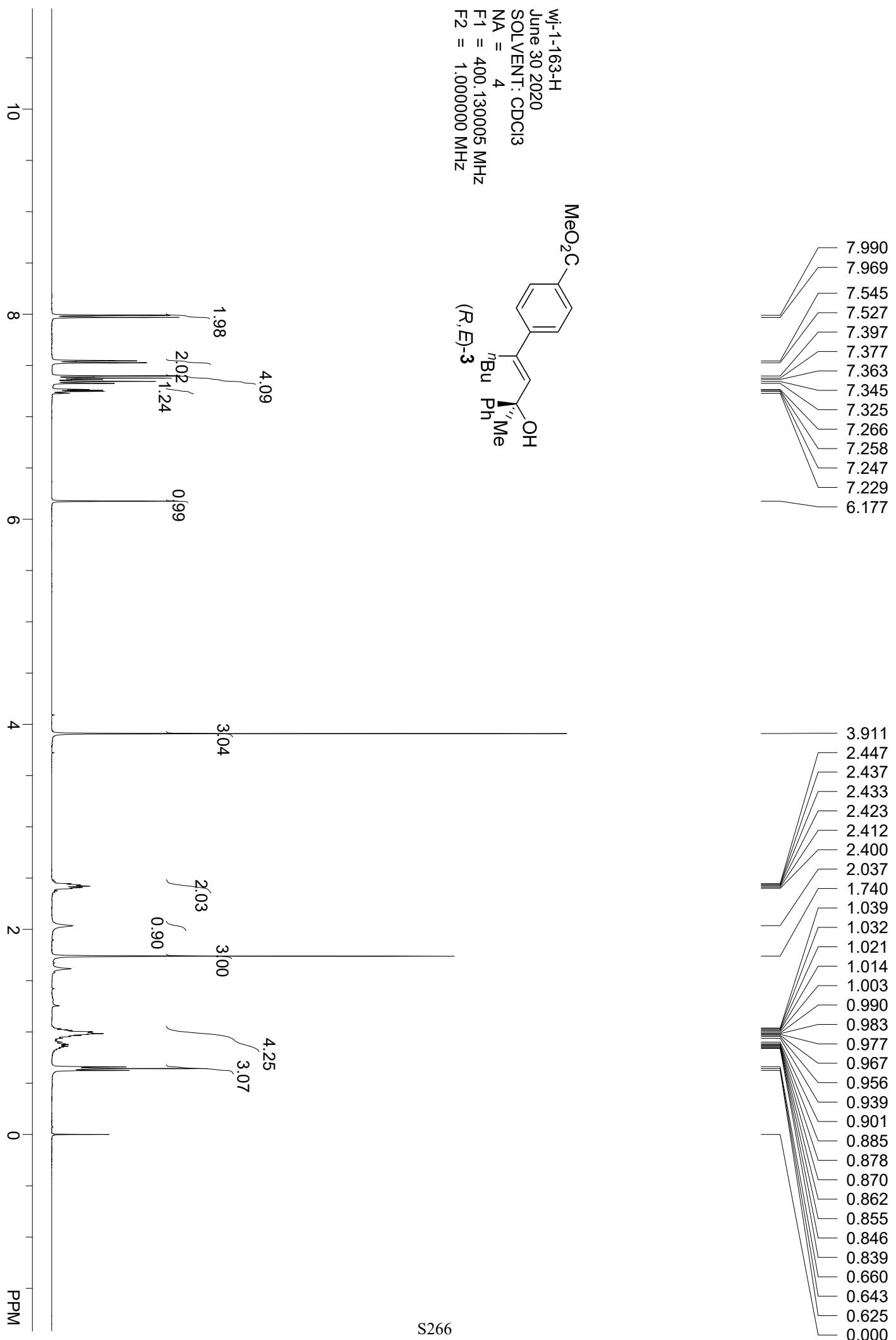
Data file: C:\Users\Public\Documents\ChemStation\1\Data\WJ_LC 2020-10-31 17-12-21\005-P2-C4-wj-2-119-2-rac.D

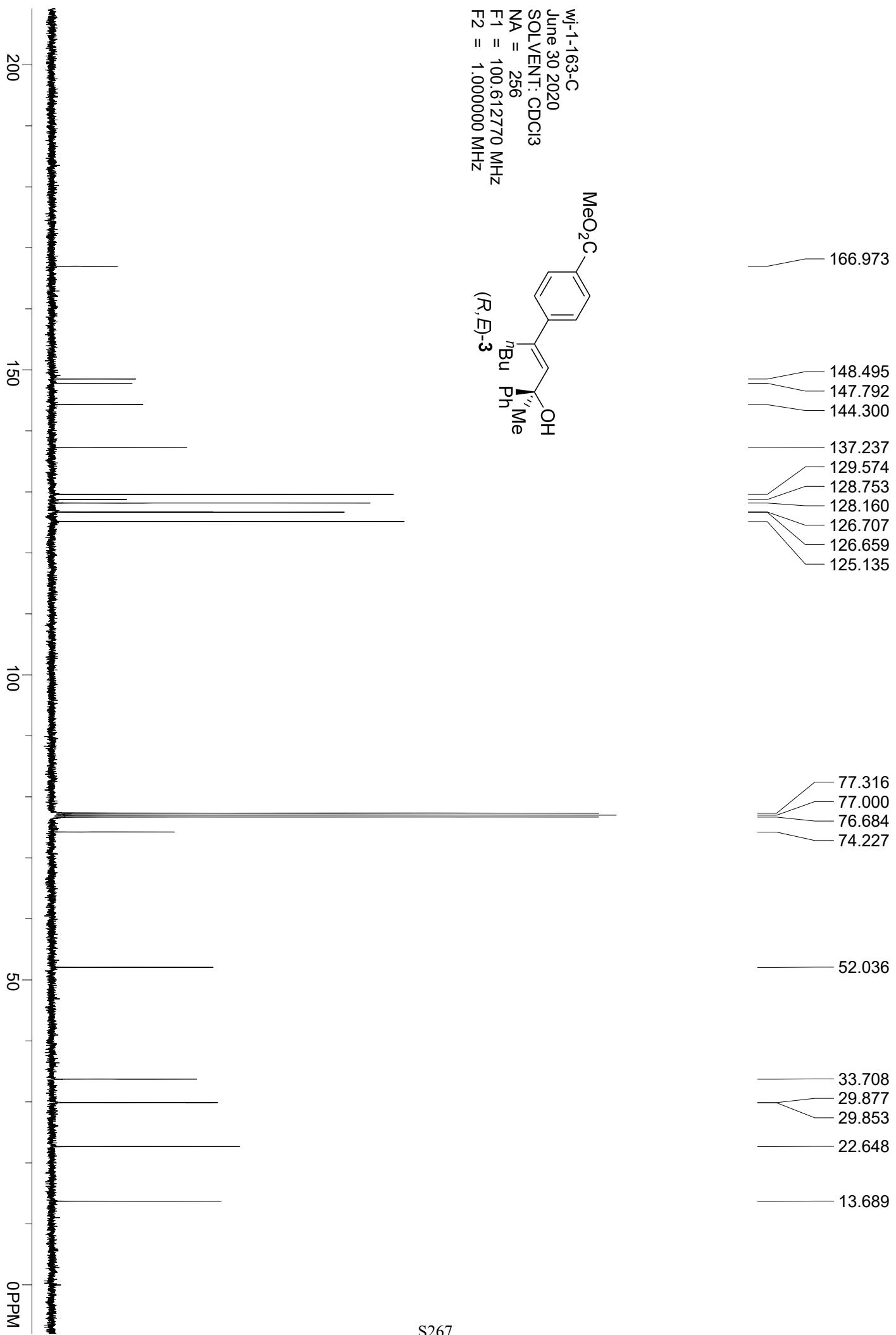
Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
10.539	0.4961	64.4313	1917.8356	49.8704
14.319	0.9769	28.2956	1927.8069	50.1296
Sum		3845.6425	100.0000	





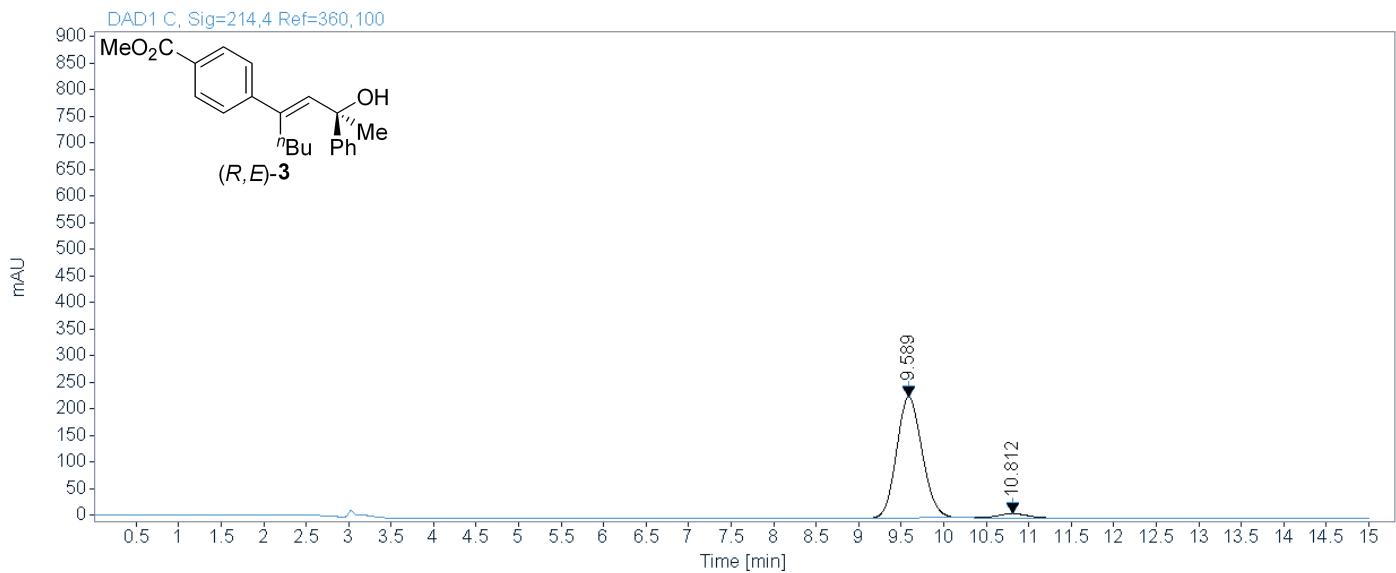
Area Percent Report



sample wj-1-163-AS-H-95-5-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\QAN 2020-06-29 08-41-07\085-P2-C1-wj-1-163.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
9.589	0.3267	227.6091	4774.8540	95.9432
10.812	0.4132	8.1444	201.8971	4.0568
Sum		4976.7511	100.0000	

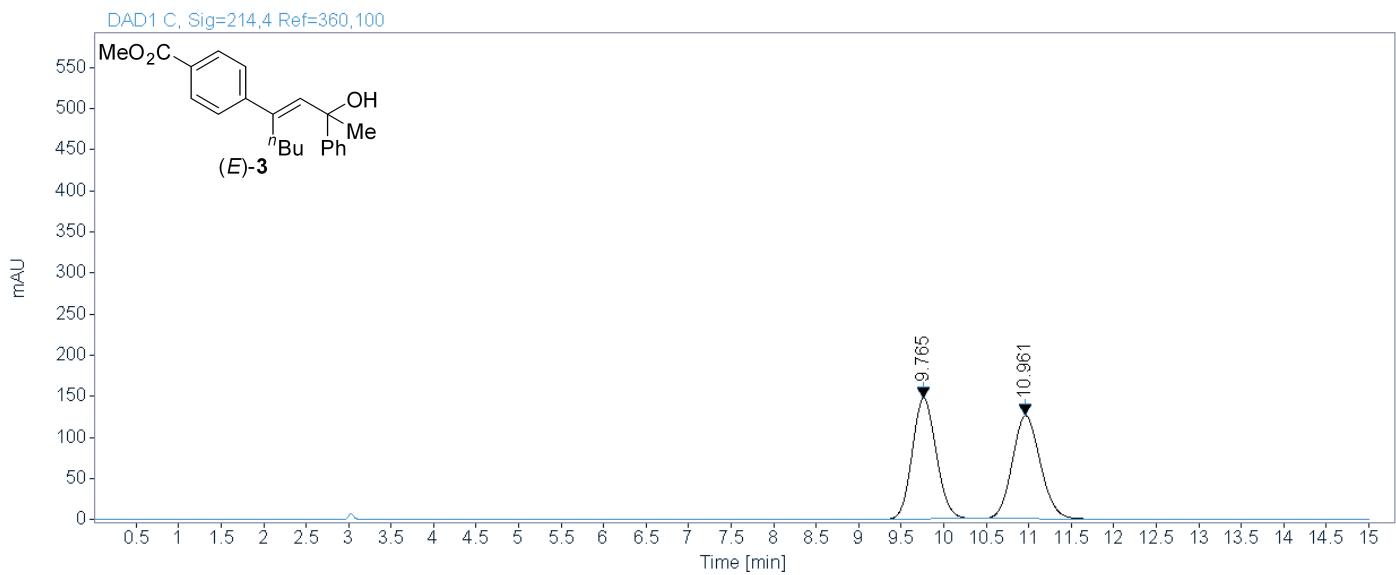
Area Percent Report



sample wj-1-163-rac-AS-H-95-5-1.0-214

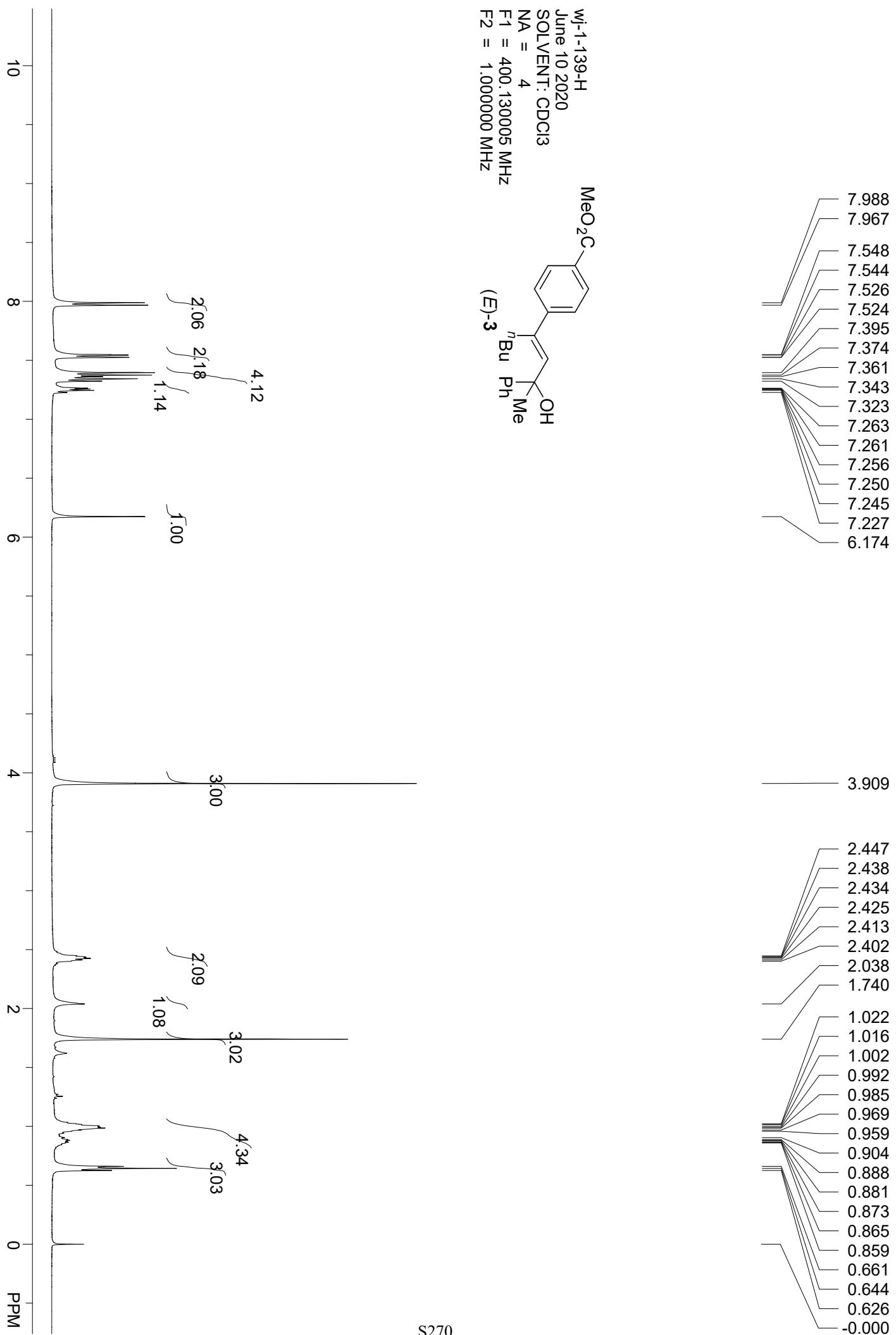
Data file: C:\Users\Public\Documents\ChemStation\1\Data\QAN 2020-06-29 08-41-07\086-P2-C2-wj-1-163-rac.D

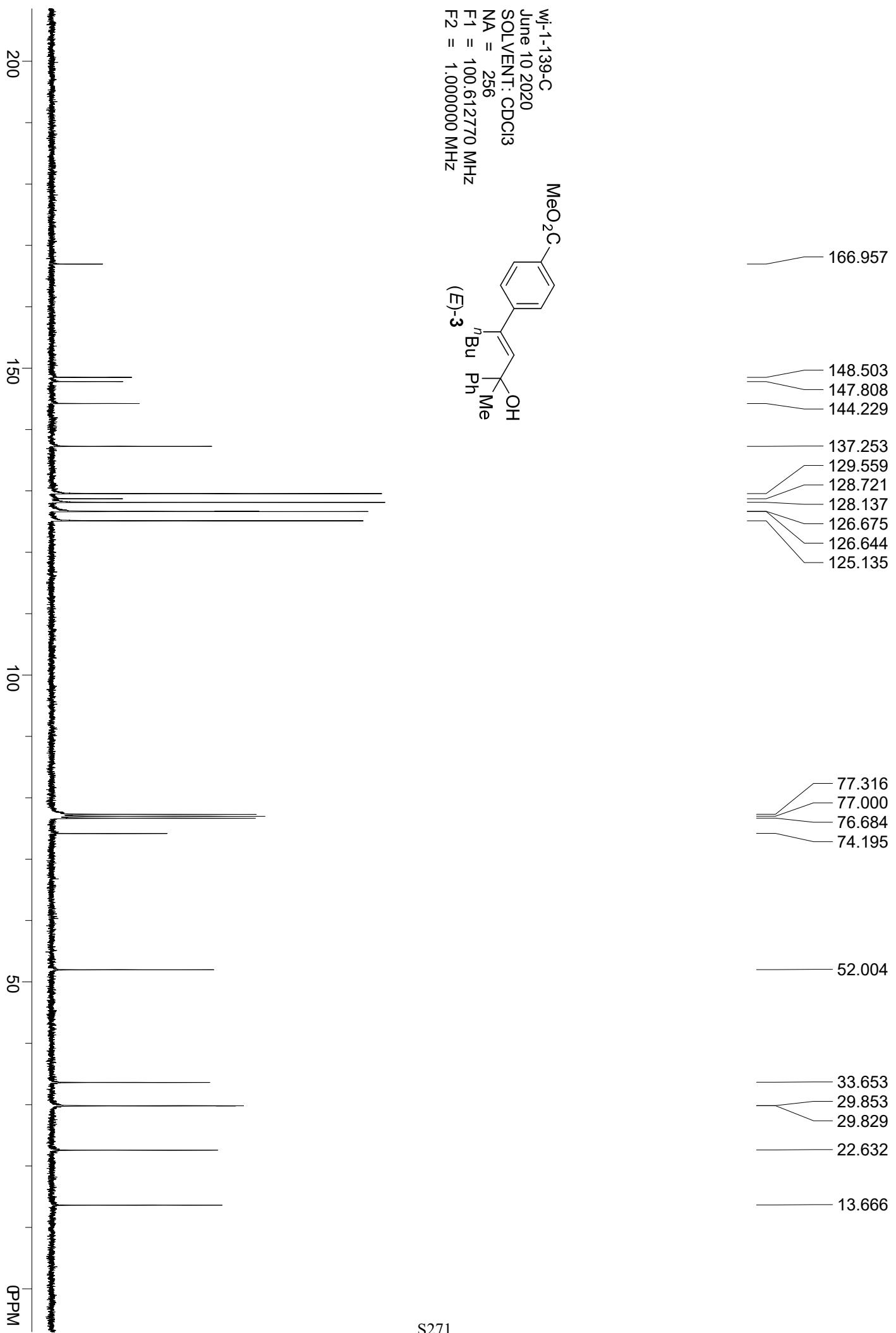
Acquisition Data:

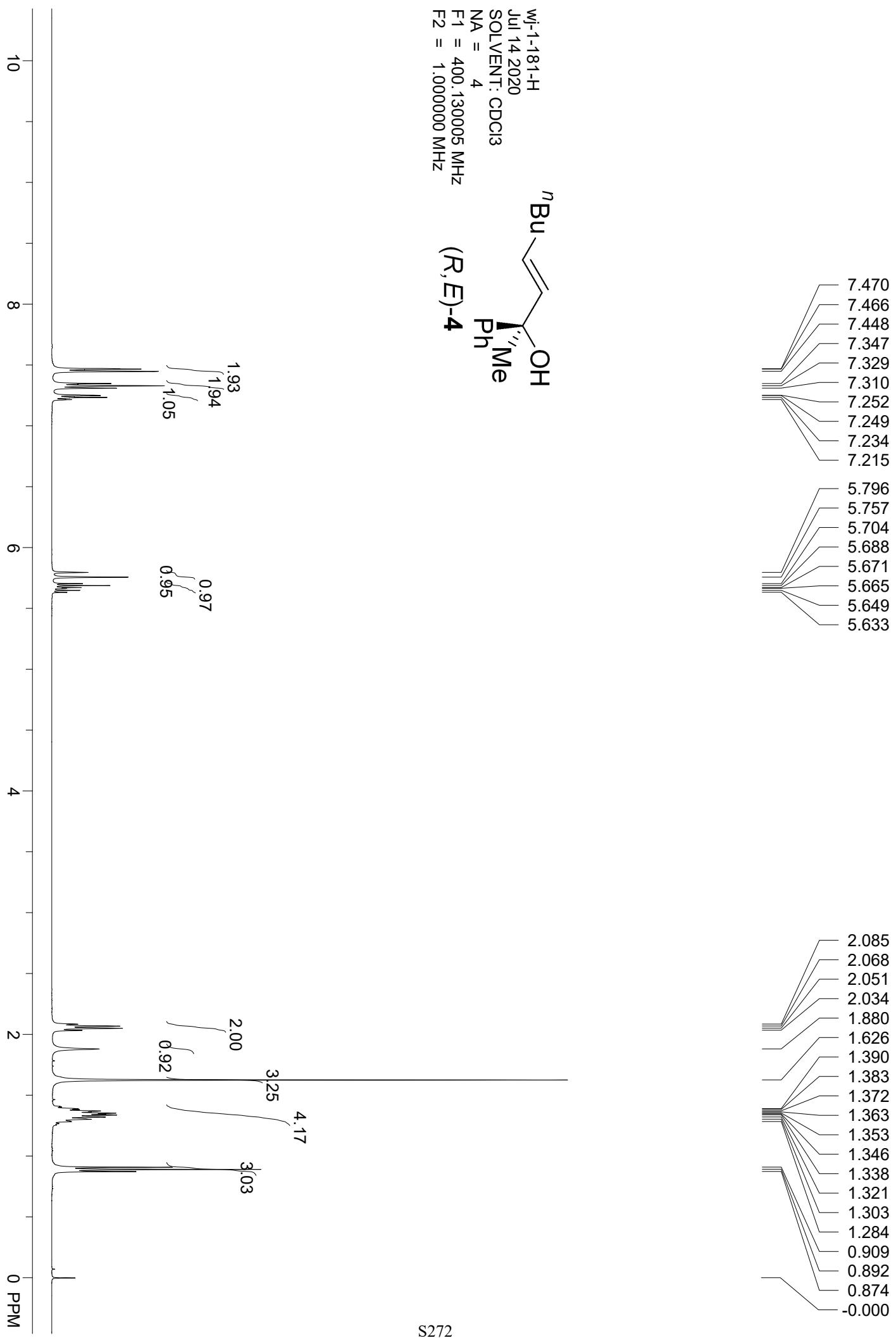


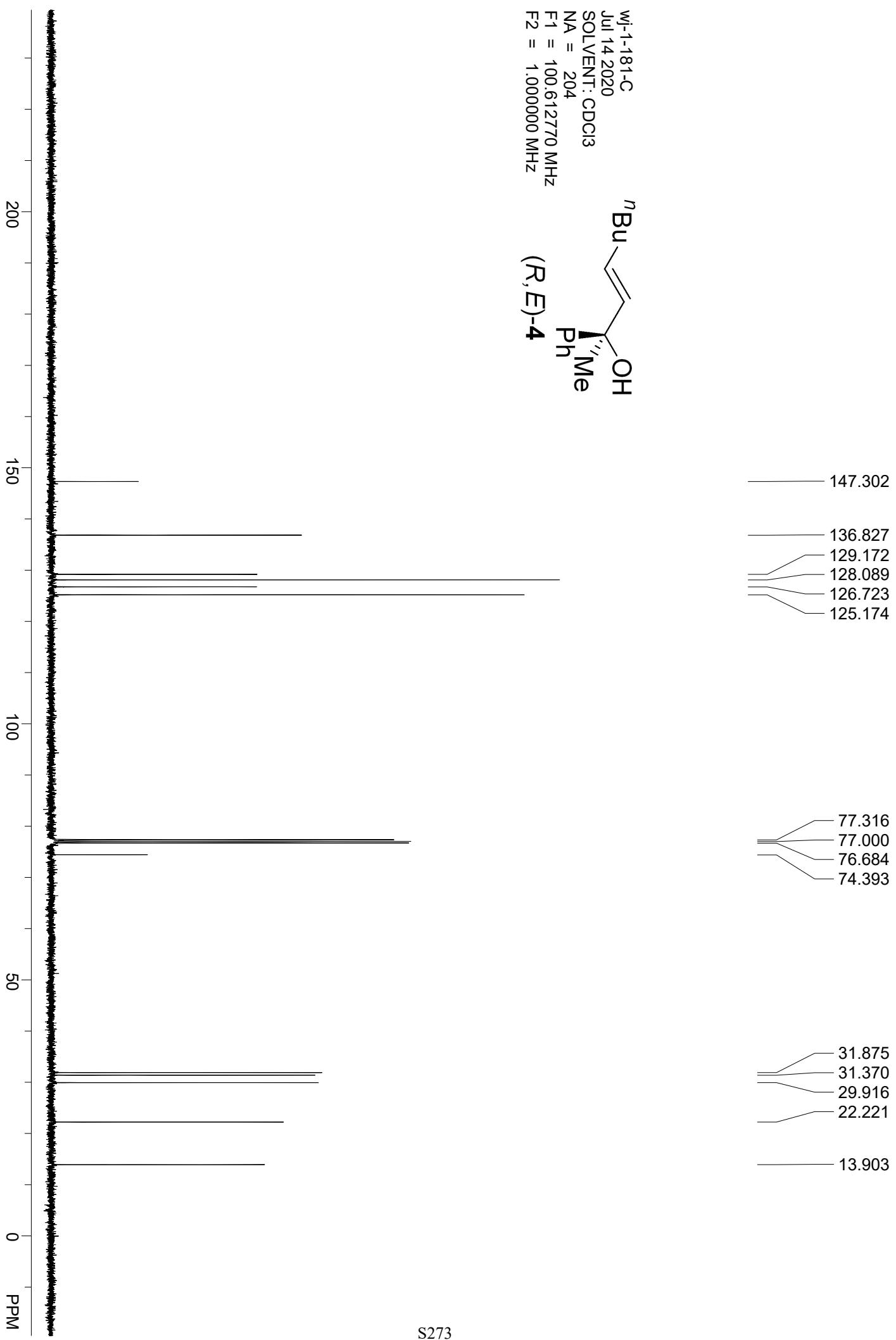
Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
9.765	0.3076	147.5005	2902.2227	50.0799
10.961	0.3587	125.6307	2892.9604	49.9201
Sum		5795.1831	100.0000	









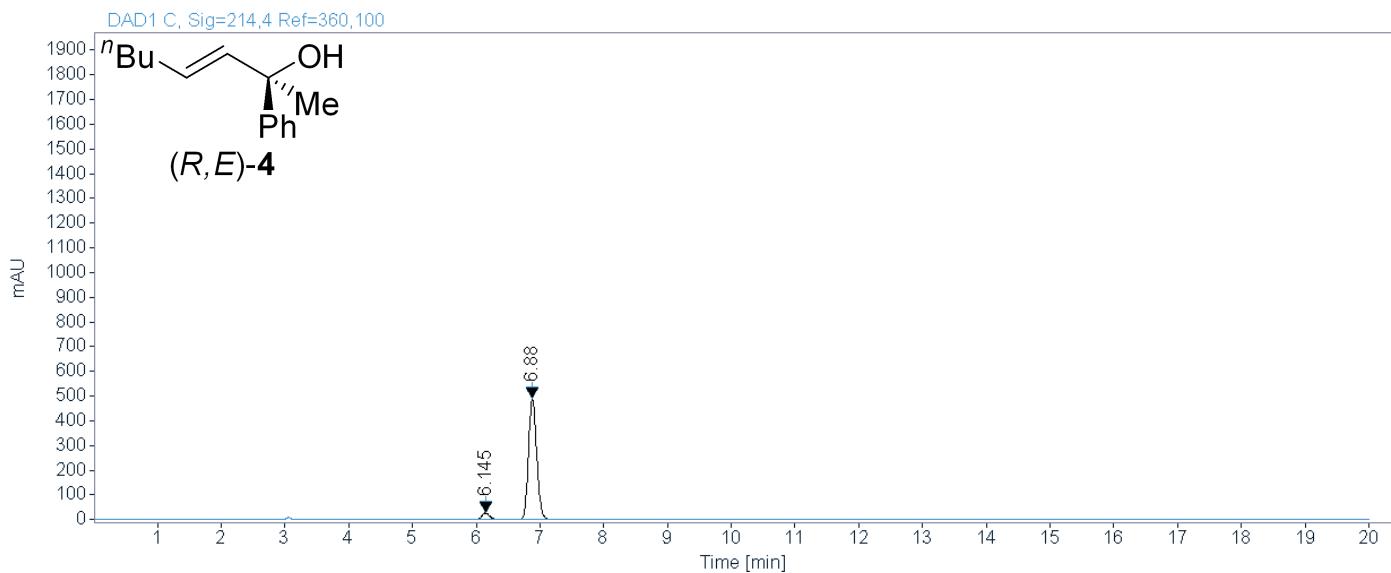
Area Percent Report



sample wj-1-181-AS-H-98-2-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\zwf-allenioc acid_LC 2020-07-14 08-35-46\040-P2-C1-wj-1-181.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
6.145	0.1237	27.4706	220.7224	4.7083
6.880	0.1507	494.0445	4467.1943	95.2917
		Sum	4687.9167	100.0000

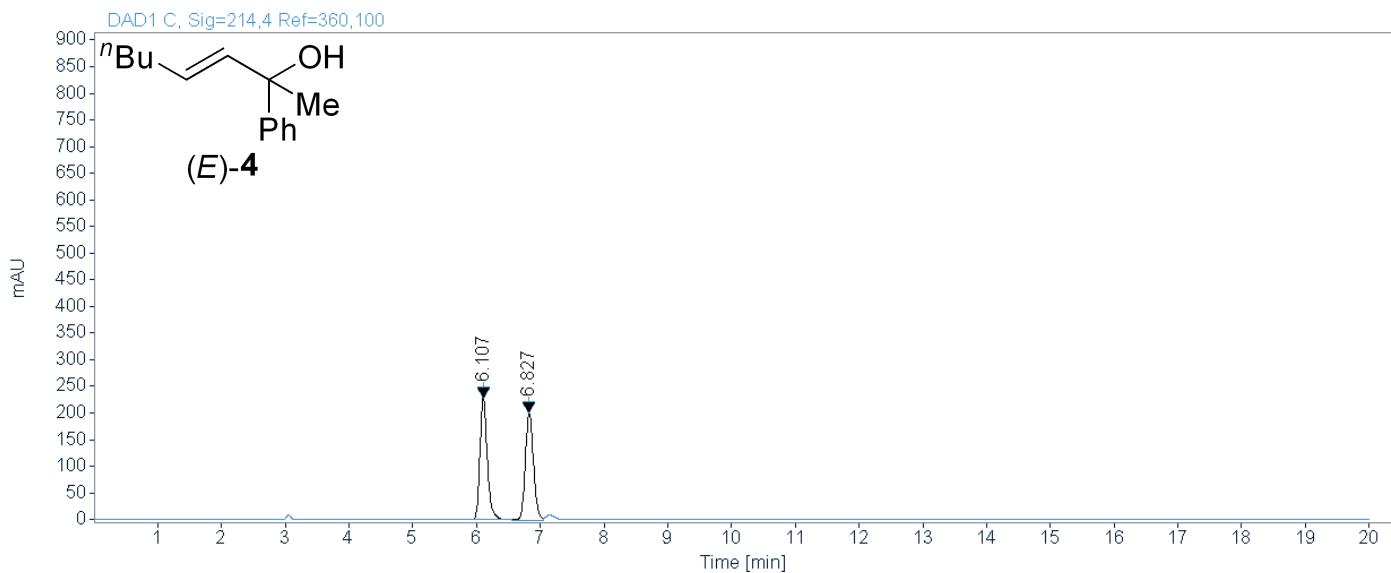
Area Percent Report



sample wj-1-181-rac-AS-H-98-2-1.0-214

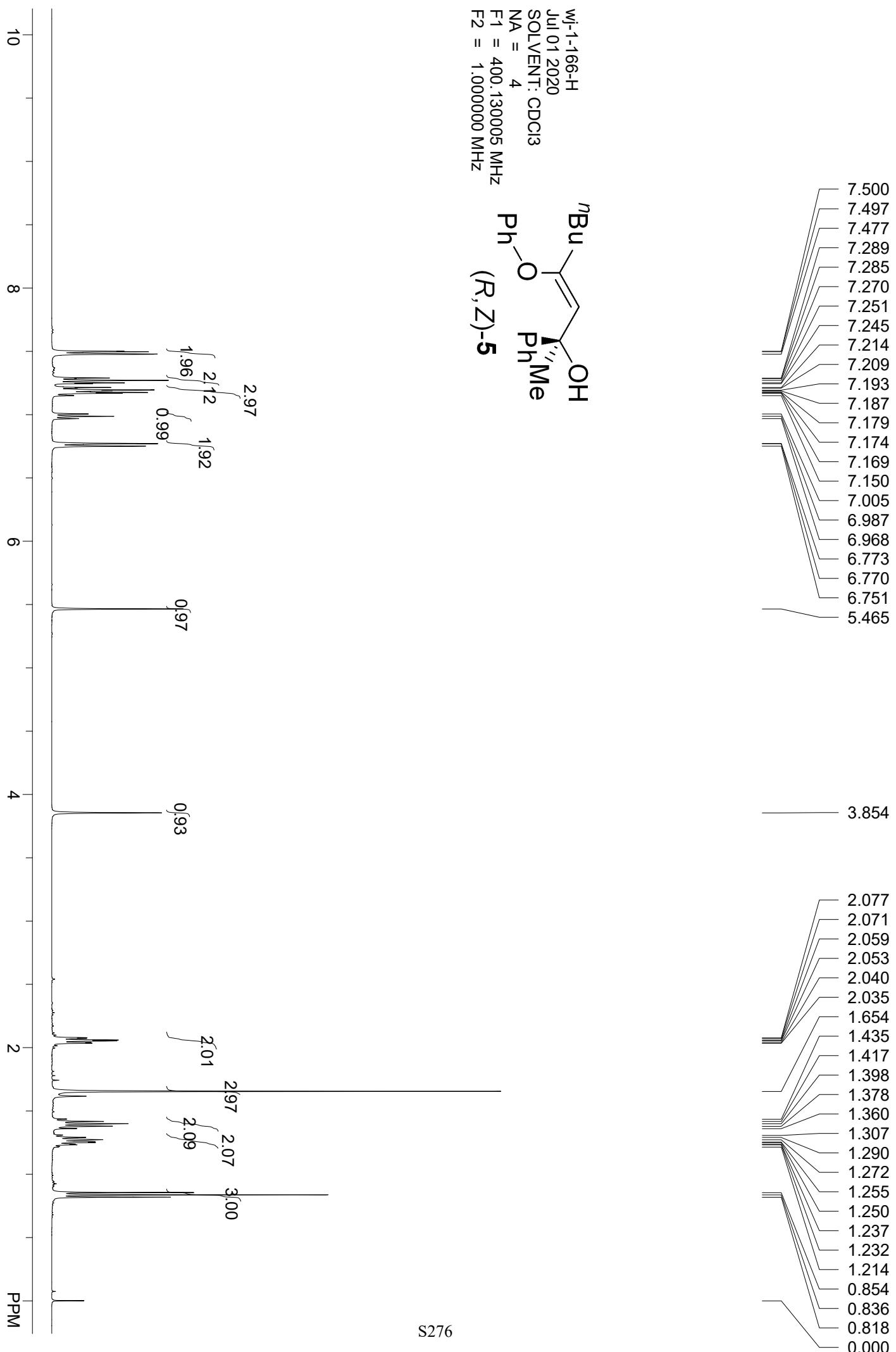
Data file: C:\Users\Public\Documents\ChemStation\1\Data\zwf-allenioc acid_LC 2020-07-14 08-35-46\041-P2-C2-wj-1-181-rac.D

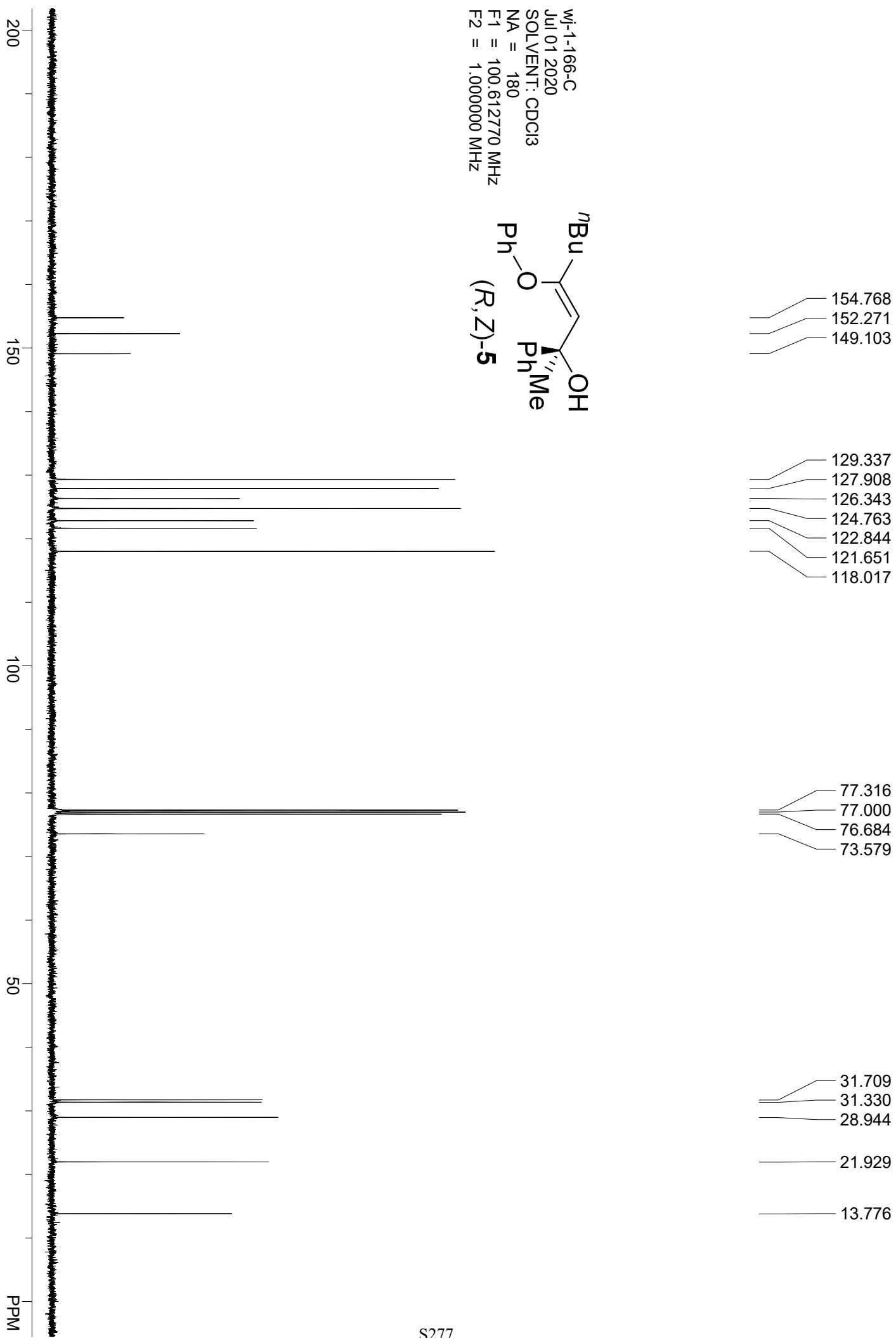
Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
6.107	0.1332	228.7032	1827.2728	50.3165
6.827	0.1488	202.0844	1804.2880	49.6835
Sum		3631.5608	100.0000	





Area Percent Report

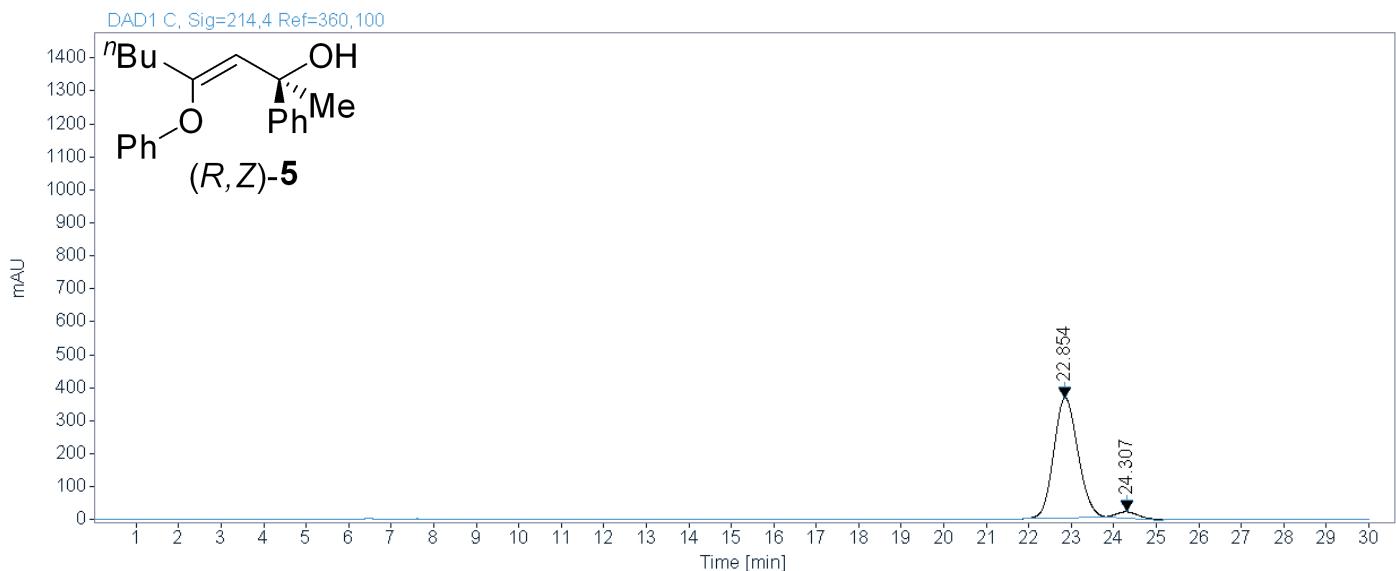


Agilent Technologies

sample wj-1-166-IC-99.5-0.5-0.5-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\WJ_LC 2020-07-02 11-33-30\009-P2-C1-wj-1-166.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
22.854	0.6605	364.1797	14432.0059	95.5562
24.307	0.5949	18.8043	671.1620	4.4438
Sum		15103.1679	100.0000	

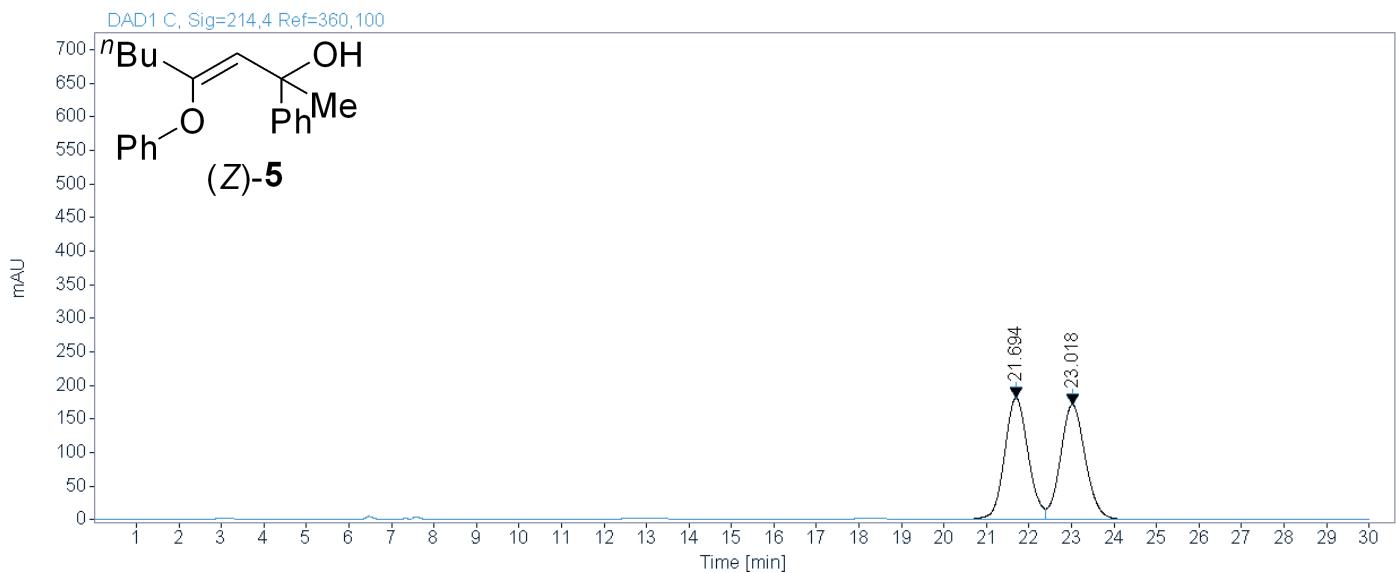
Area Percent Report



sample wj-1-166-rac-LC-99.5-0.5-0.5-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\WJ_LC 2020-07-02 11-33-30\008-P2-C2-wj-1-166-RAC.D

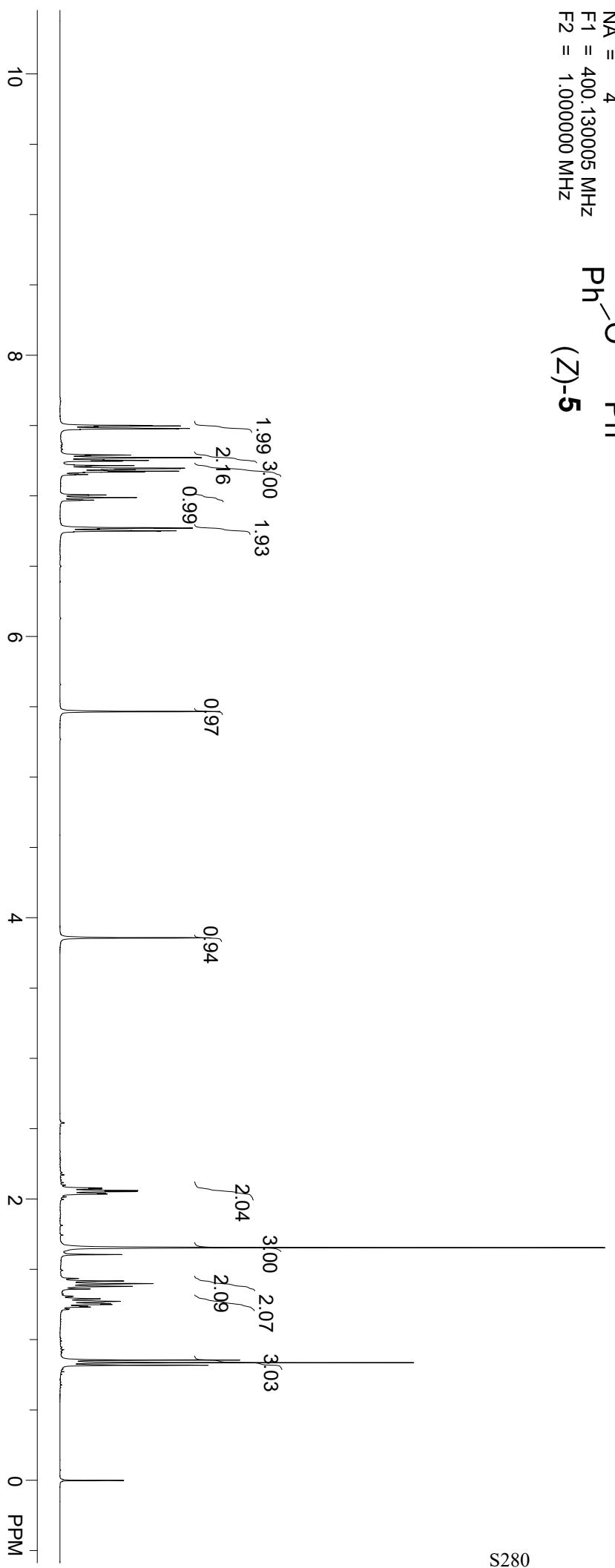
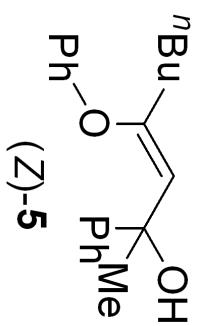
Acquisition Data:

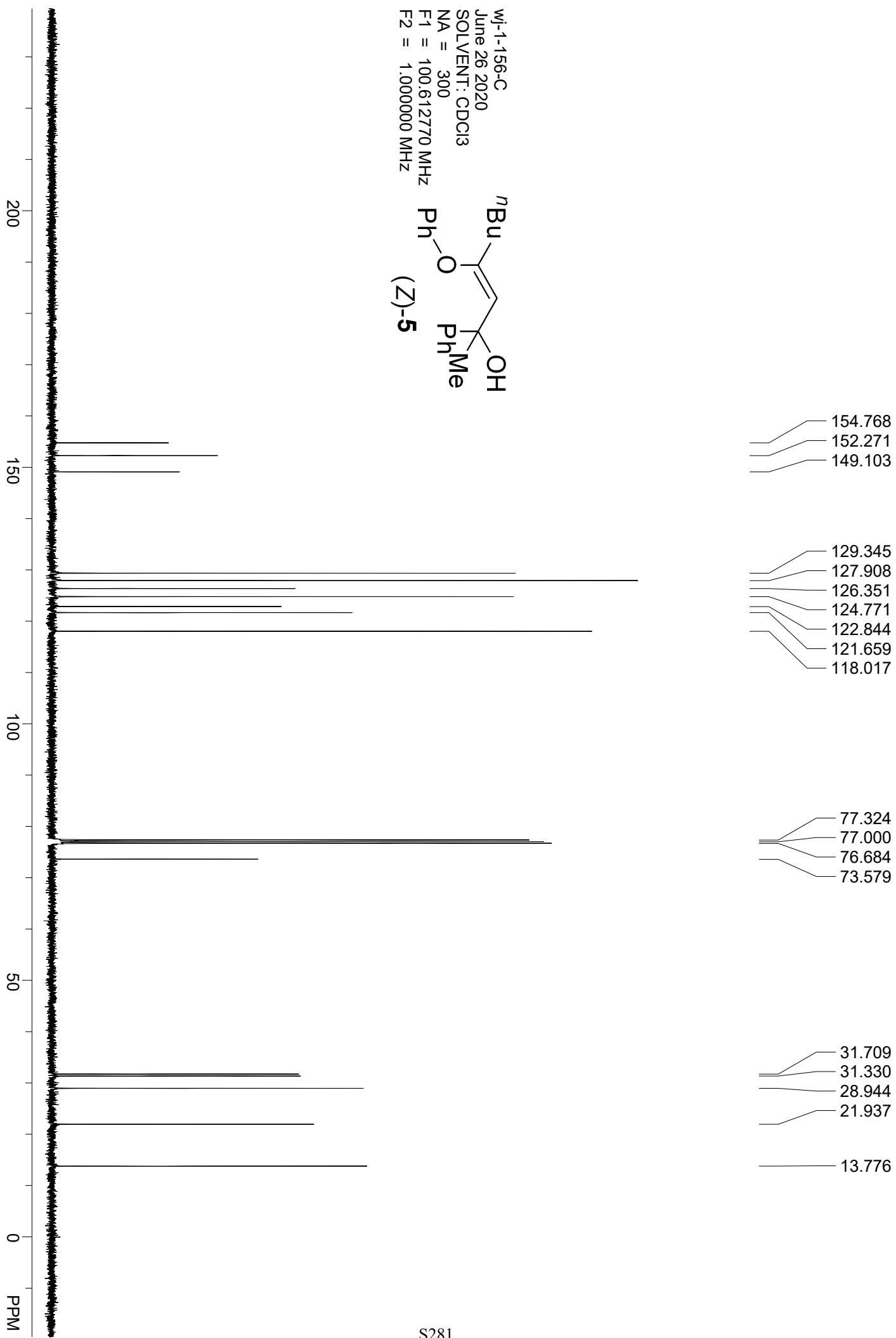


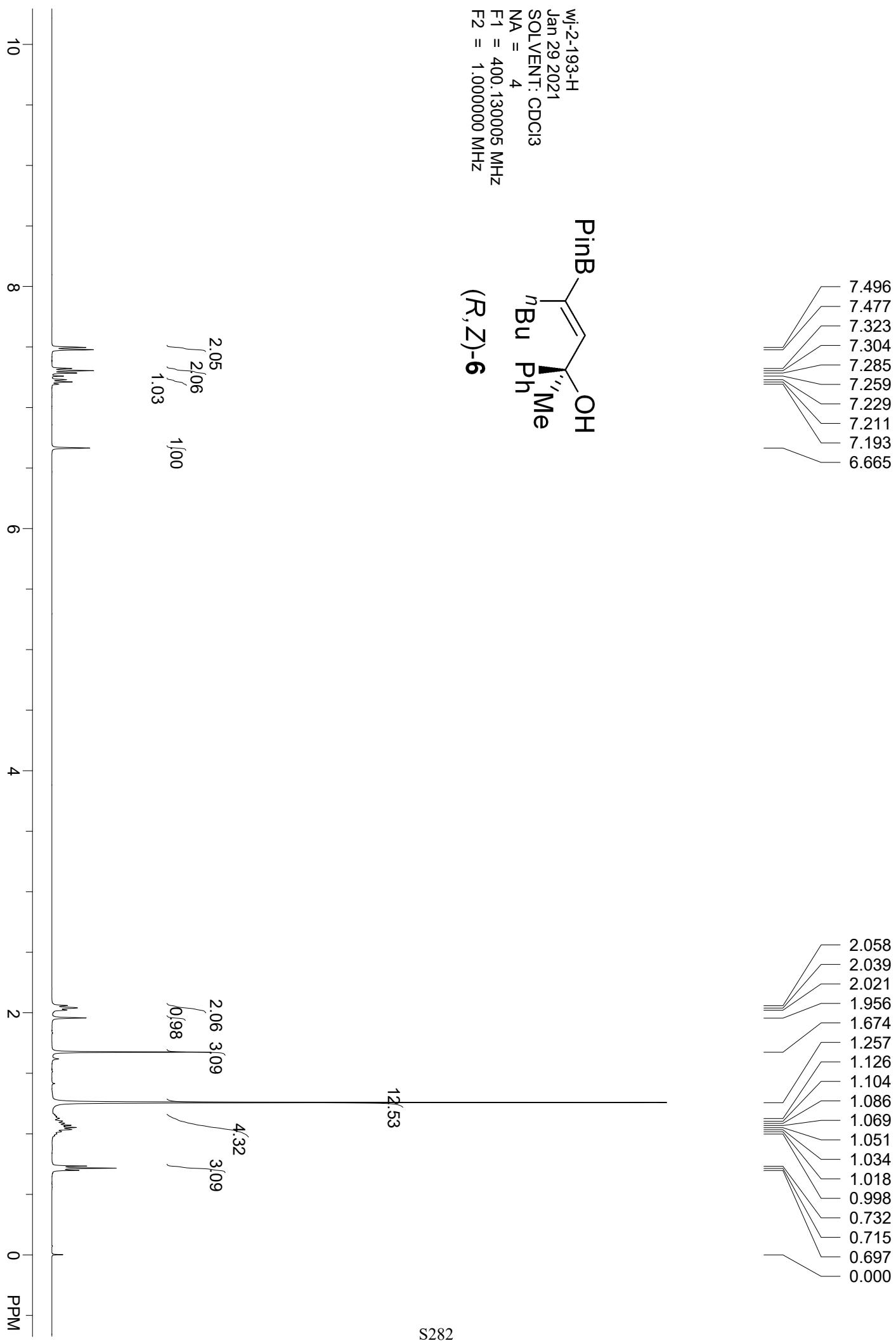
Signal: DAD1 C, Sig=214,4 Ref=360,100

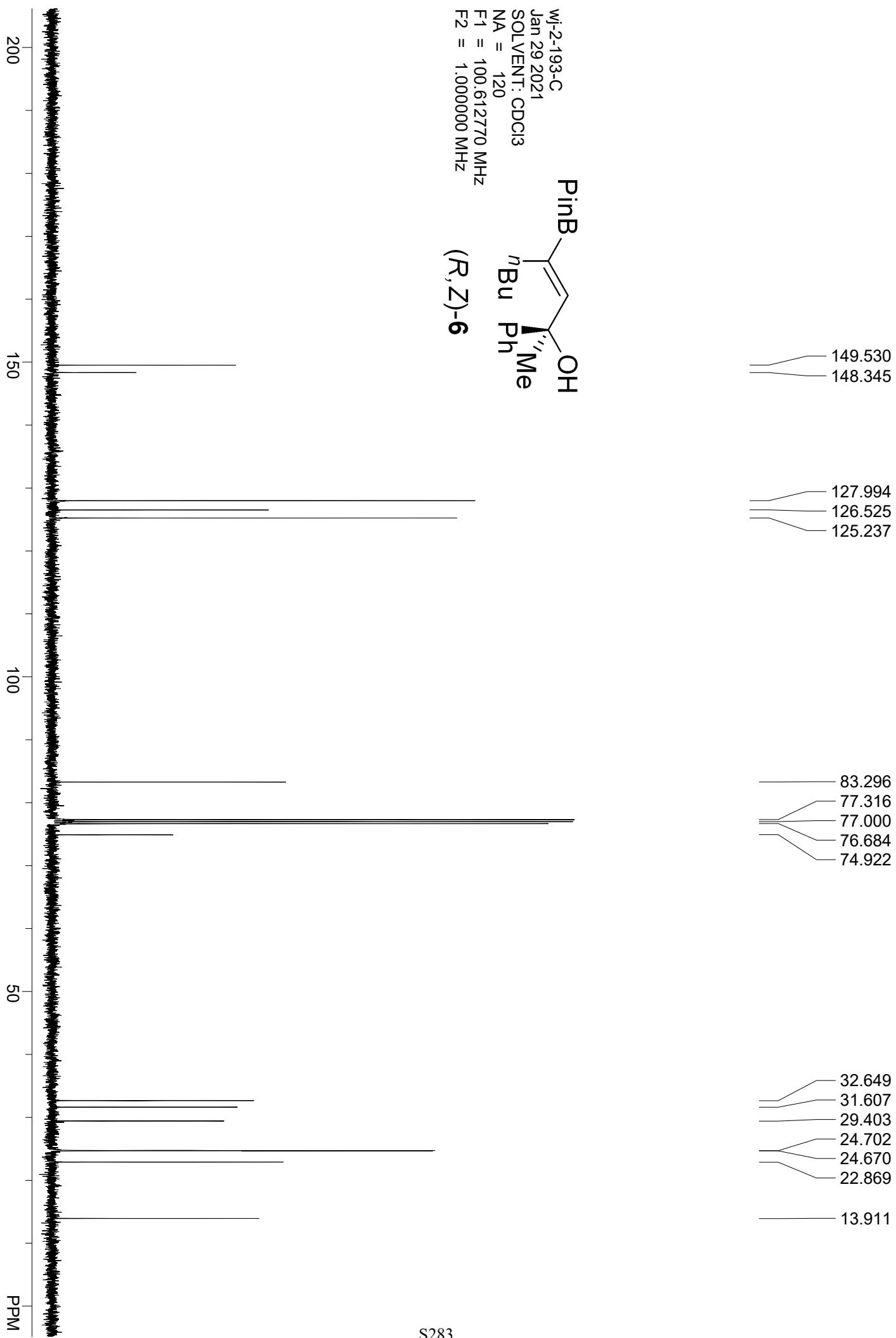
RT [min]	Width [min]	Height	Area	Area%
21.694	0.5962	181.2001	6961.1162	50.0641
23.018	0.6294	171.1311	6943.2949	49.9359
Sum		13904.4111	13904.4111	100.0000

wj-1-156-H
June 28 2020
SOLVENT: CDCl₃
NA = 4
F1 = 400.130005 MHz
F2 = 1.000000 MHz









Area Percent Report

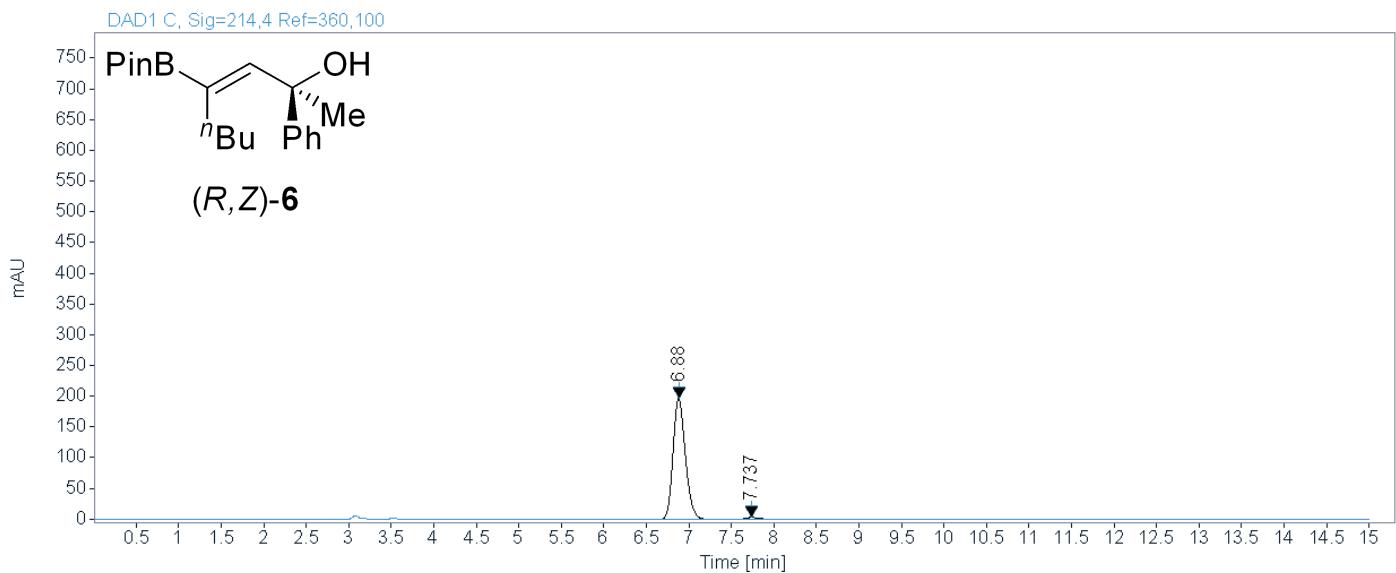


Agilent Technologies

sample wj-2-193-AD-H-98-2-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\WJ_LC 2021-01-29 15-35-00\005-P2-C3-wj-2-193.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
6.880	0.1676	197.8443	1989.7397	98.0664
7.737	0.2023	3.2314	39.2322	1.9336
Sum		2028.9720	100.0000	

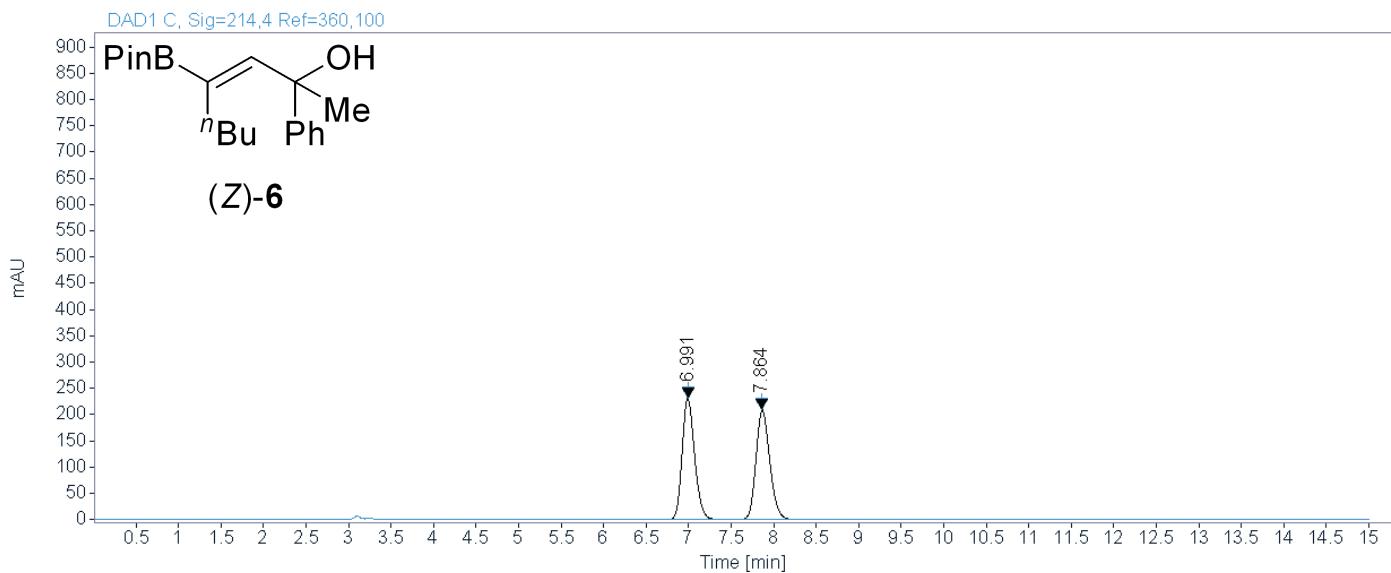
Area Percent Report



sample wj-2-193-rac-AD-H-98-2-1.0-214

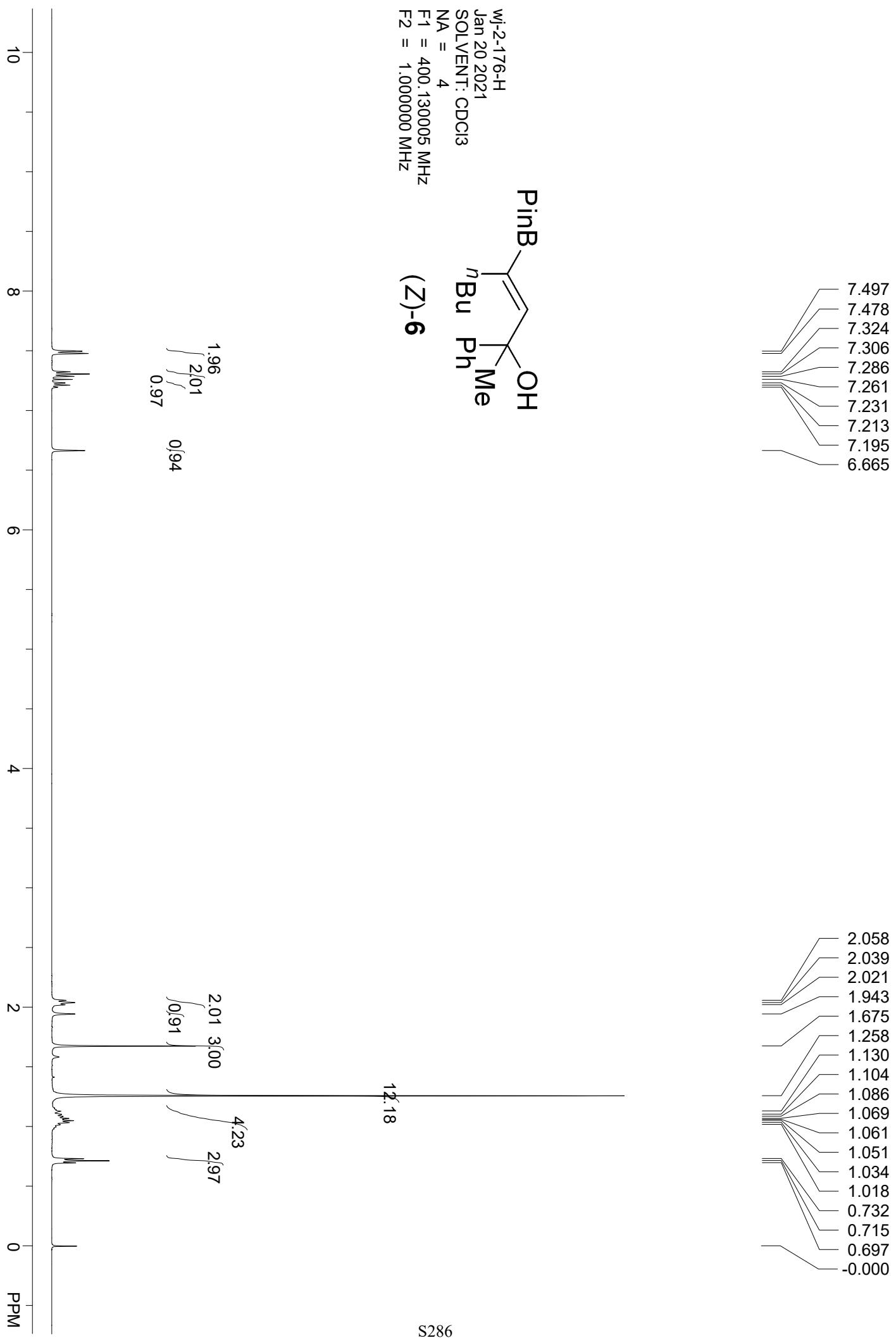
Data file: C:\Users\Public\Documents\ChemStation\1\Data\WJ_LC 2021-01-29 15-35-00\006-P2-C4-wj-2-193-rac.D

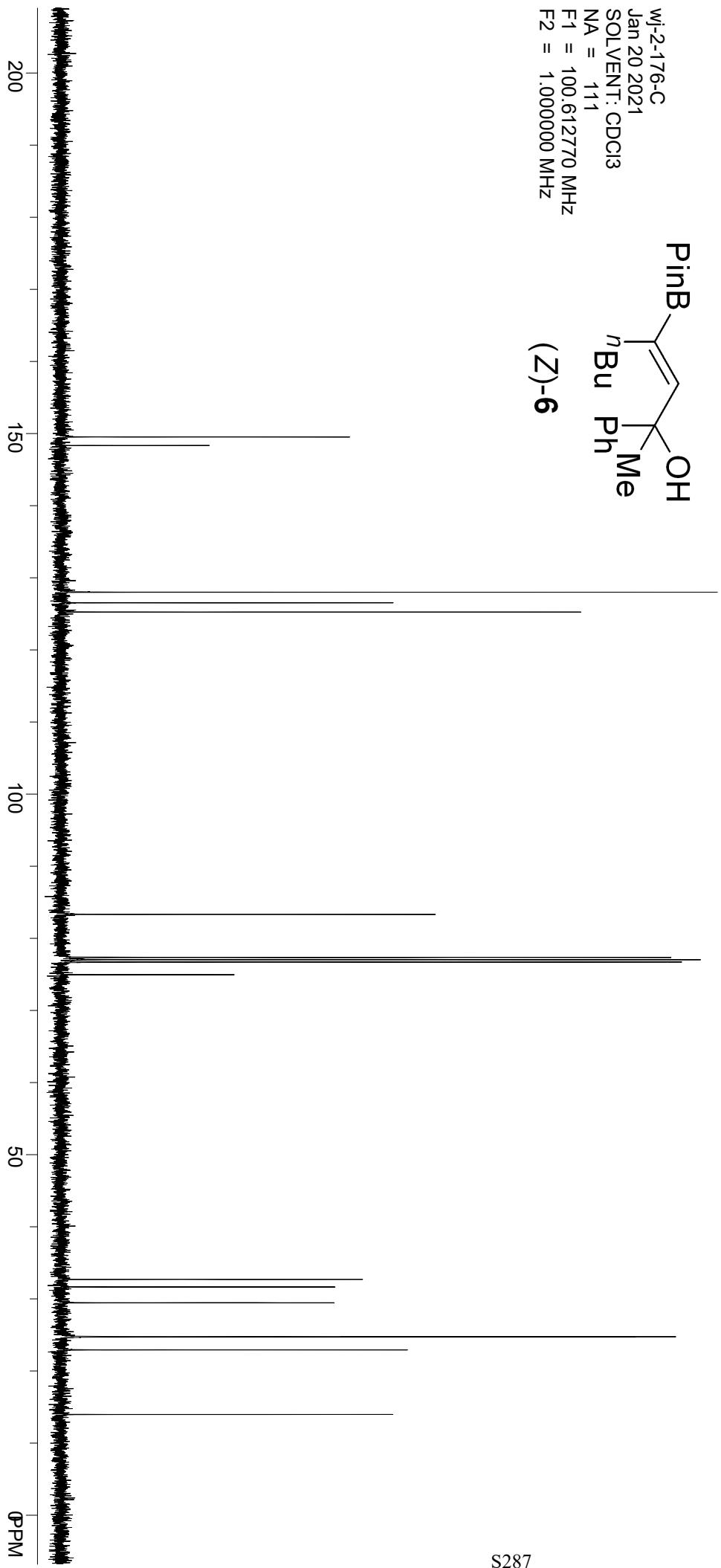
Acquisition Data:



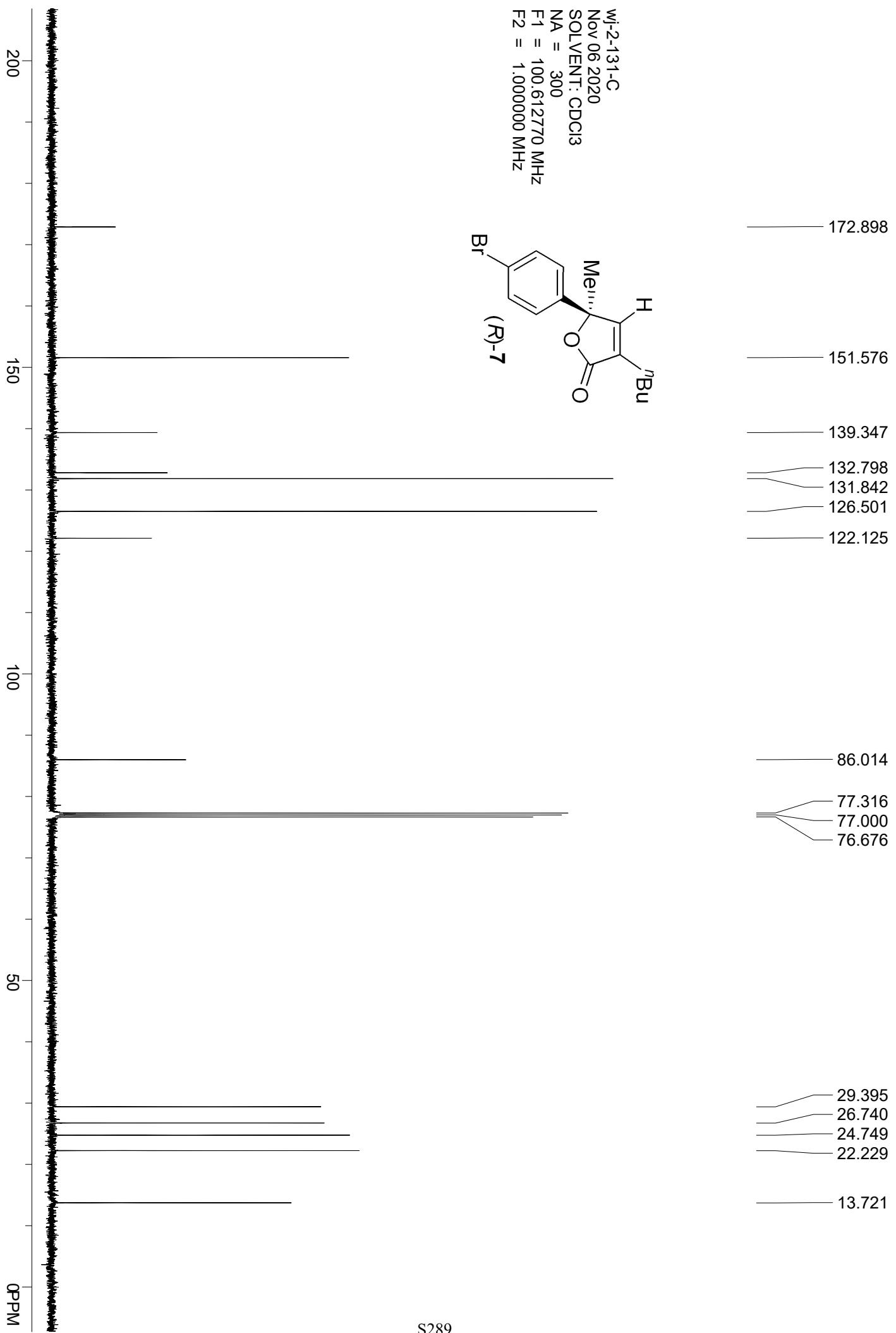
Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
6.991	0.1566	231.5414	2353.2109	49.9057
7.864	0.1763	208.3642	2362.1013	50.0943
Sum		4715.3123	100.0000	









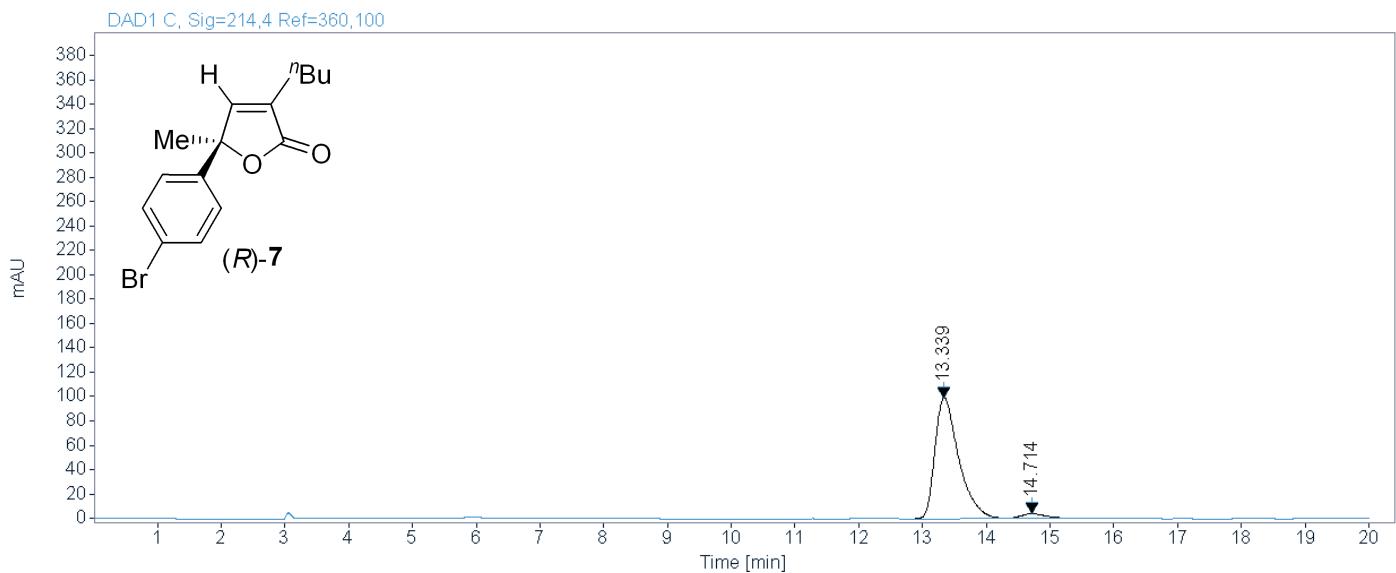
Area Percent Report



sample wj-2-131-OD-H-99.5-0.5-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\zhangq 2020-11-06 15-59-05\015-P2-C3-wj-2-131.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
13.339	0.3901	99.1396	2567.5222	96.3754
14.714	0.4001	3.6568	96.5632	3.6246
Sum		99.1396	2664.0854	100.0000

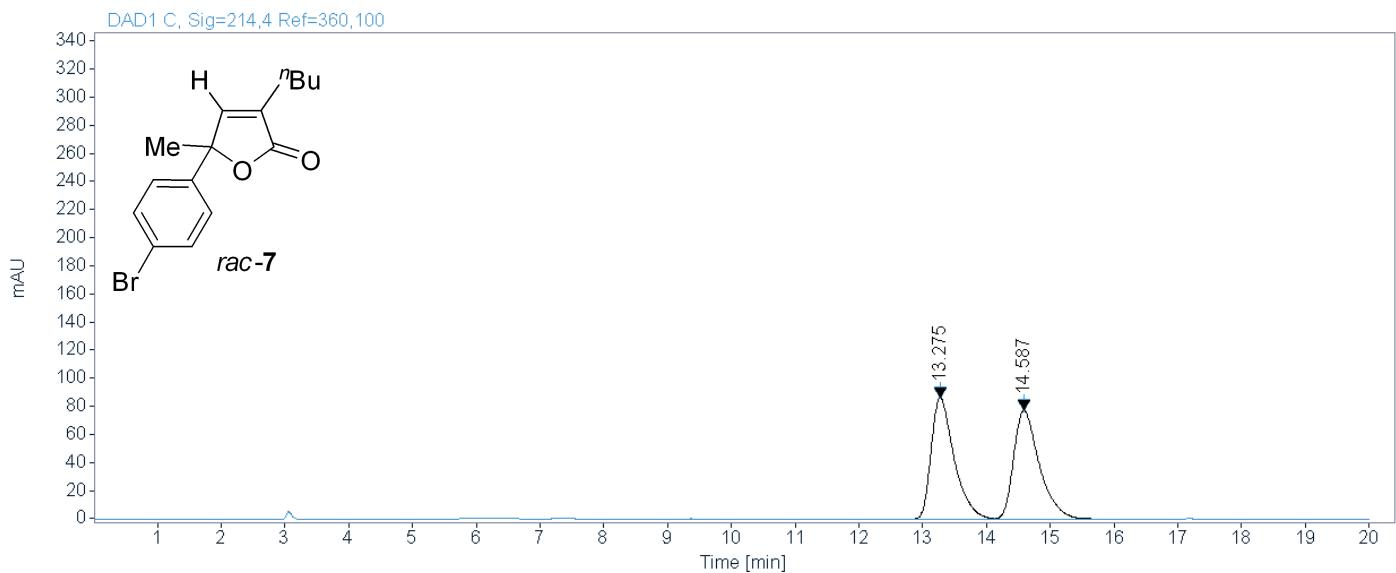
Area Percent Report



sample wj-2-131-rac-OD-H-99.5-0.5-1.0-214

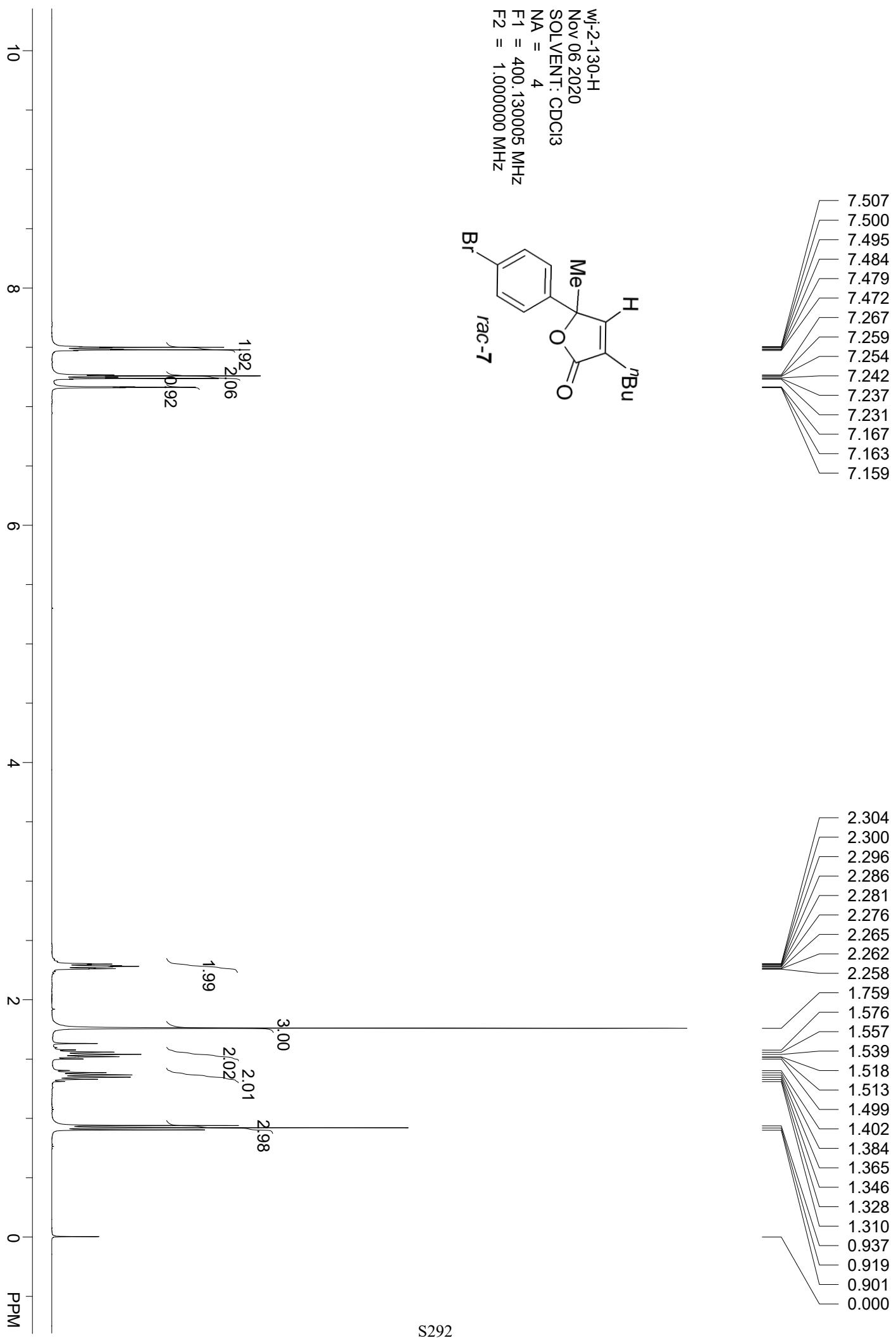
Data file: C:\Users\Public\Documents\ChemStation\1\Data\zhangq 2020-11-06 15-59-05\014-P2-C4-wj-2-131-rac.D

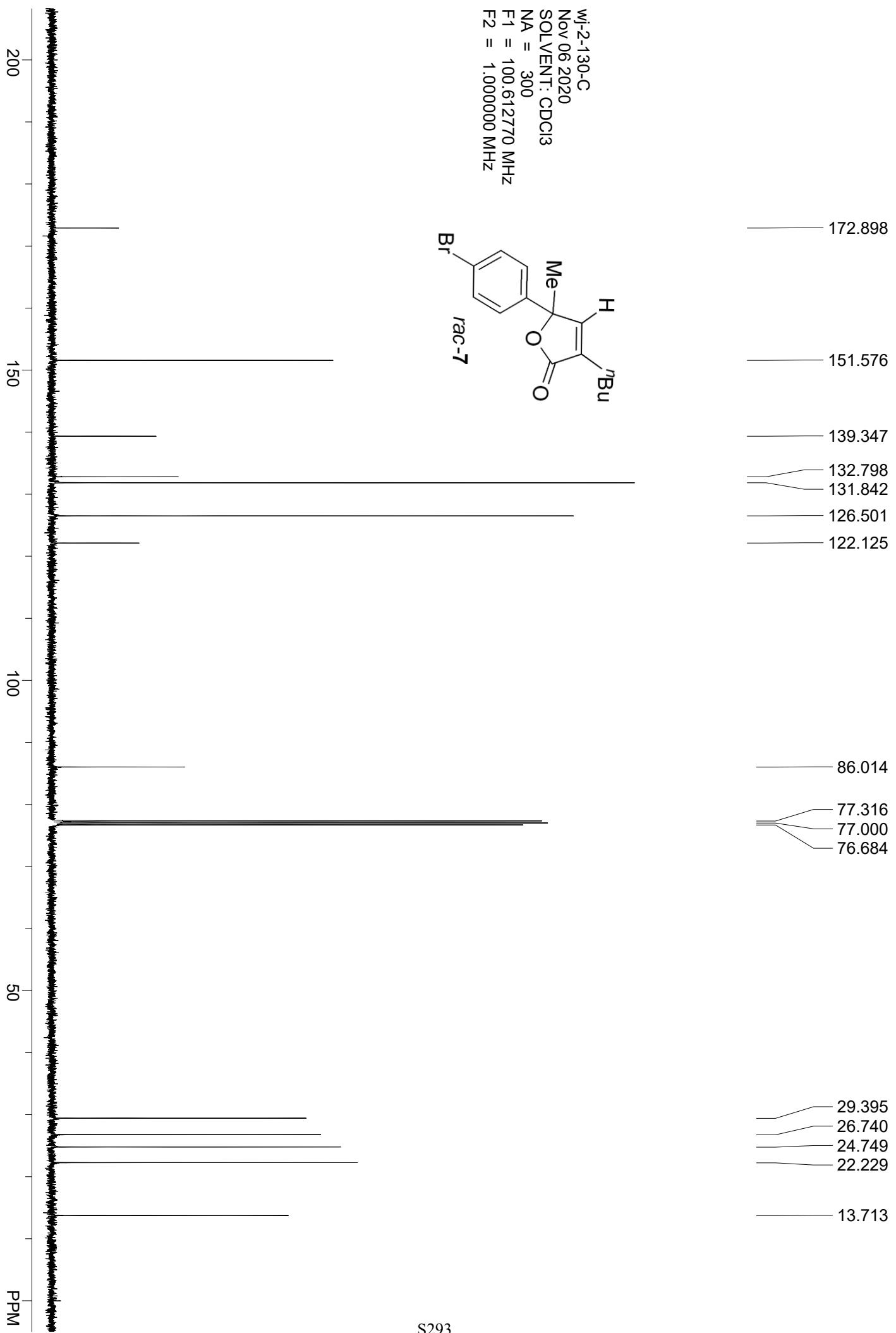
Acquisition Data:

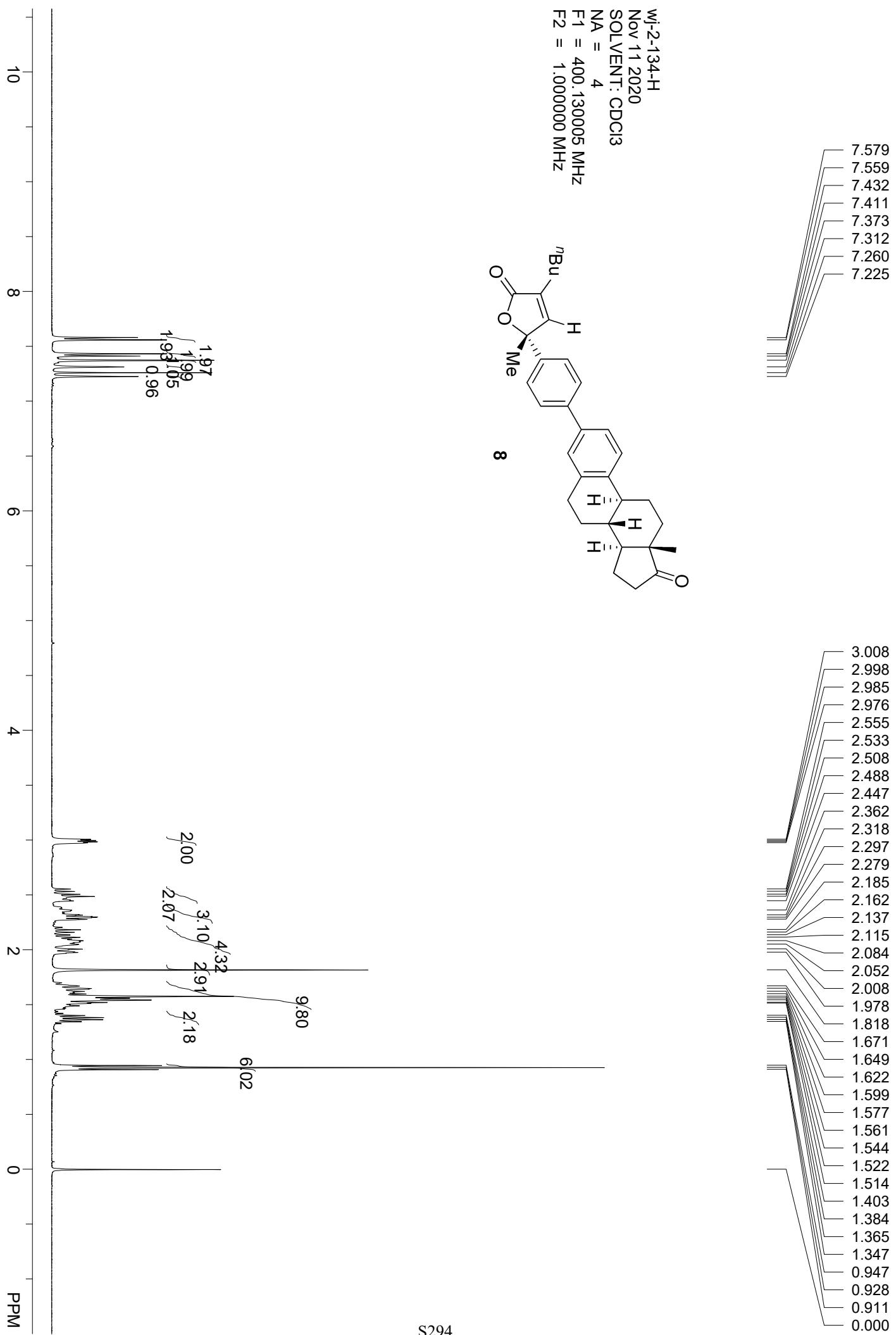


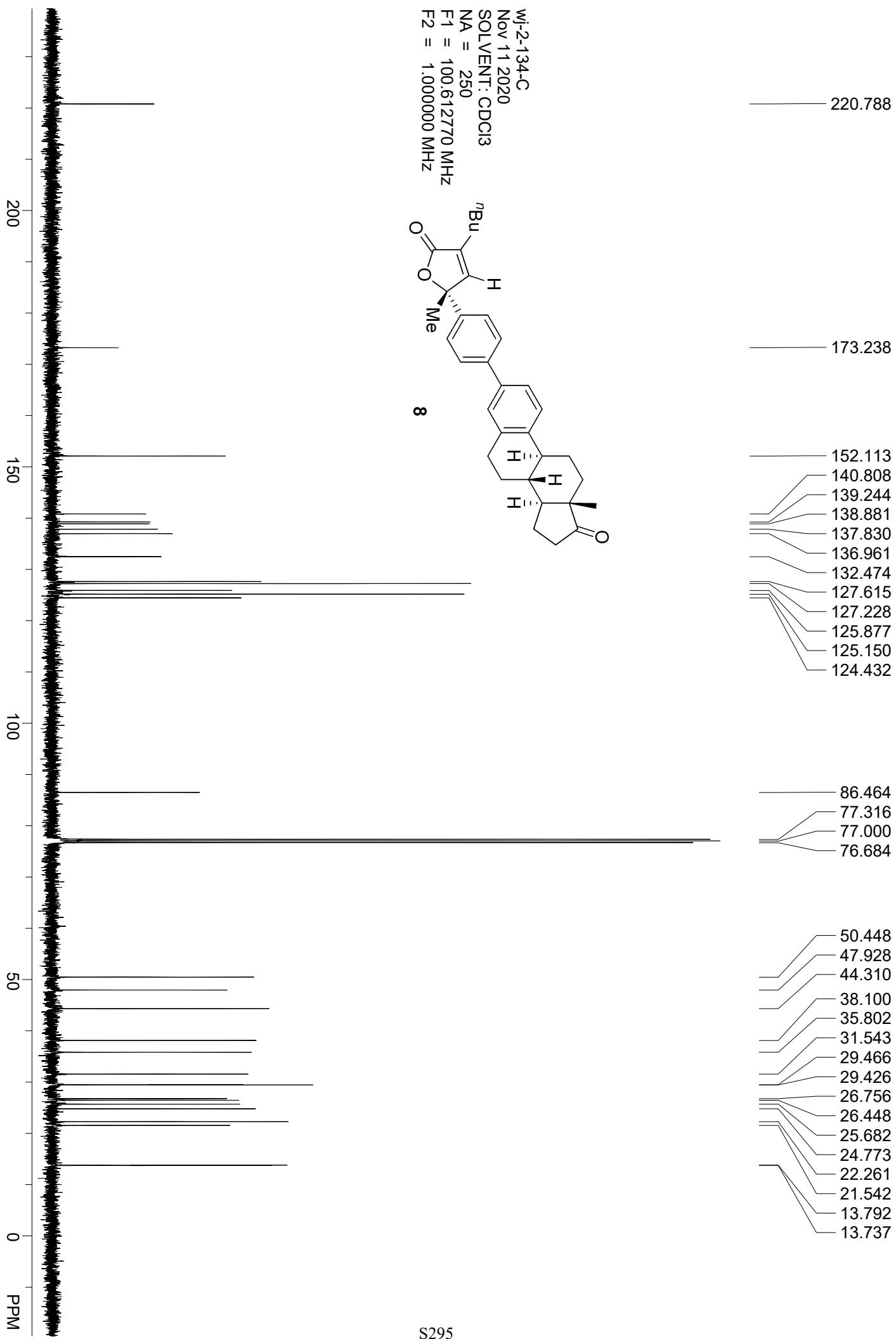
Signal: DAD1 C, Sig=214,4 Ref=360,100

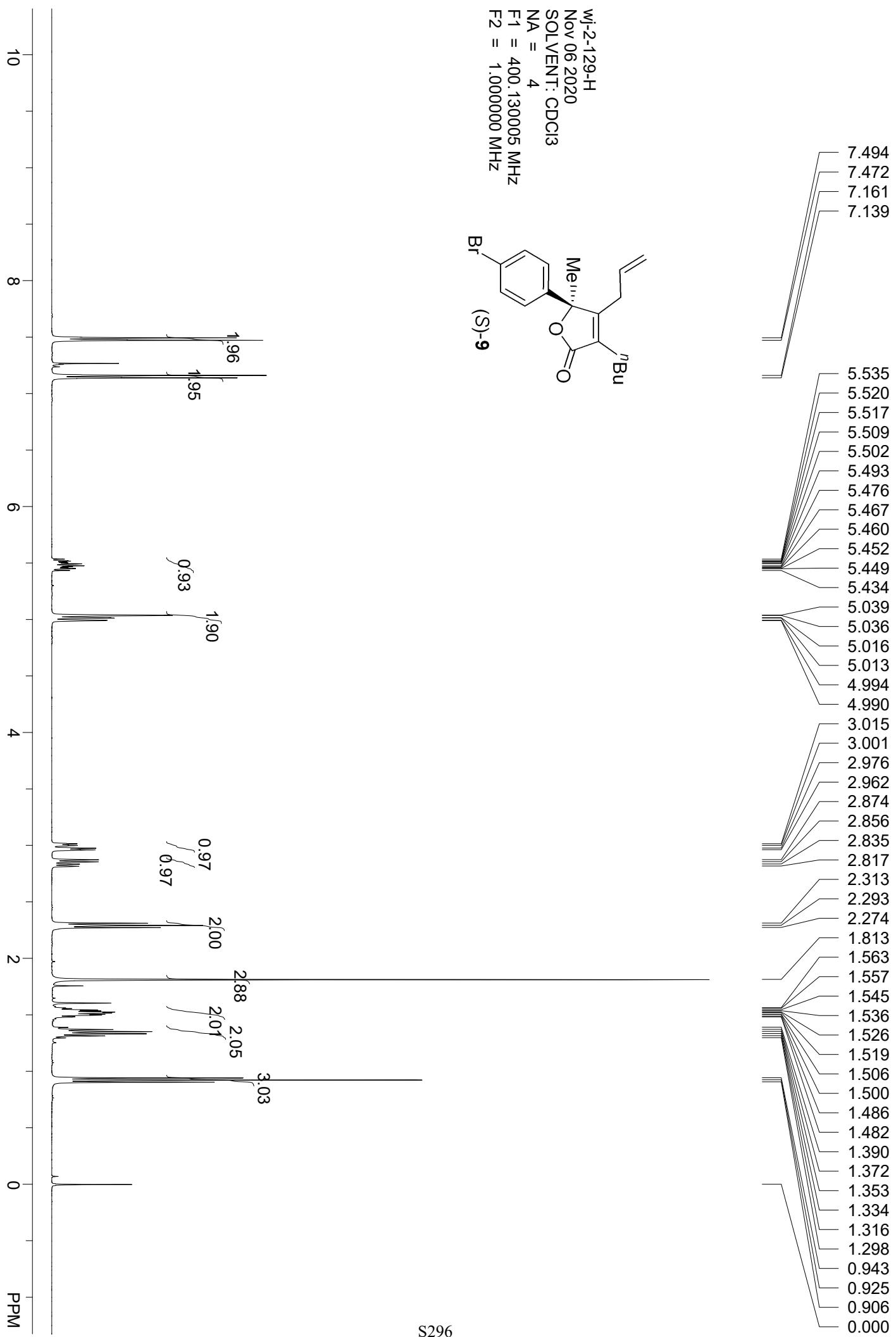
RT [min]	Width [min]	Height	Area	Area%
13.275	0.3792	86.3742	2170.4182	49.7857
14.587	0.4275	77.5067	2189.1057	50.2143
Sum		4359.5239	100.0000	

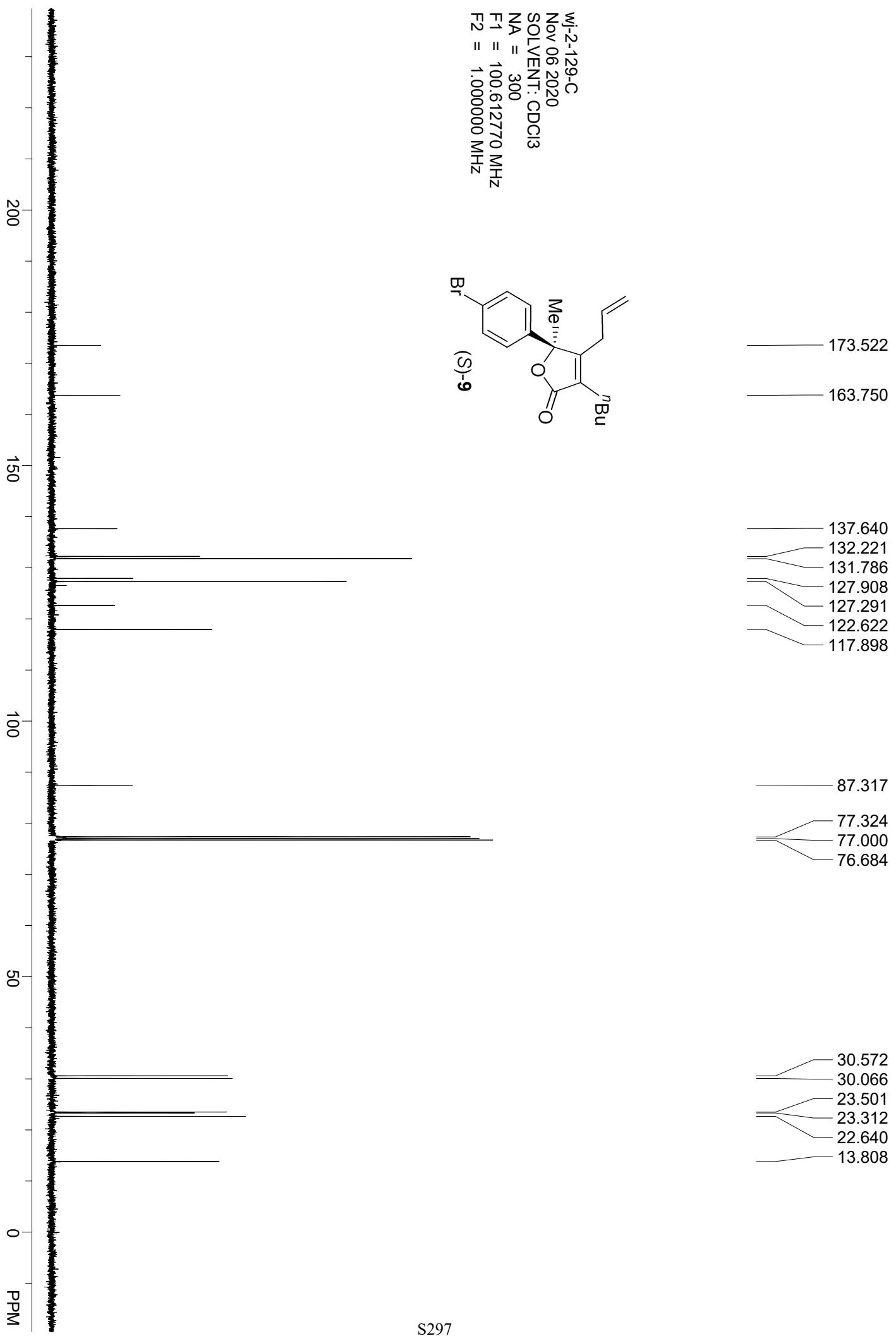












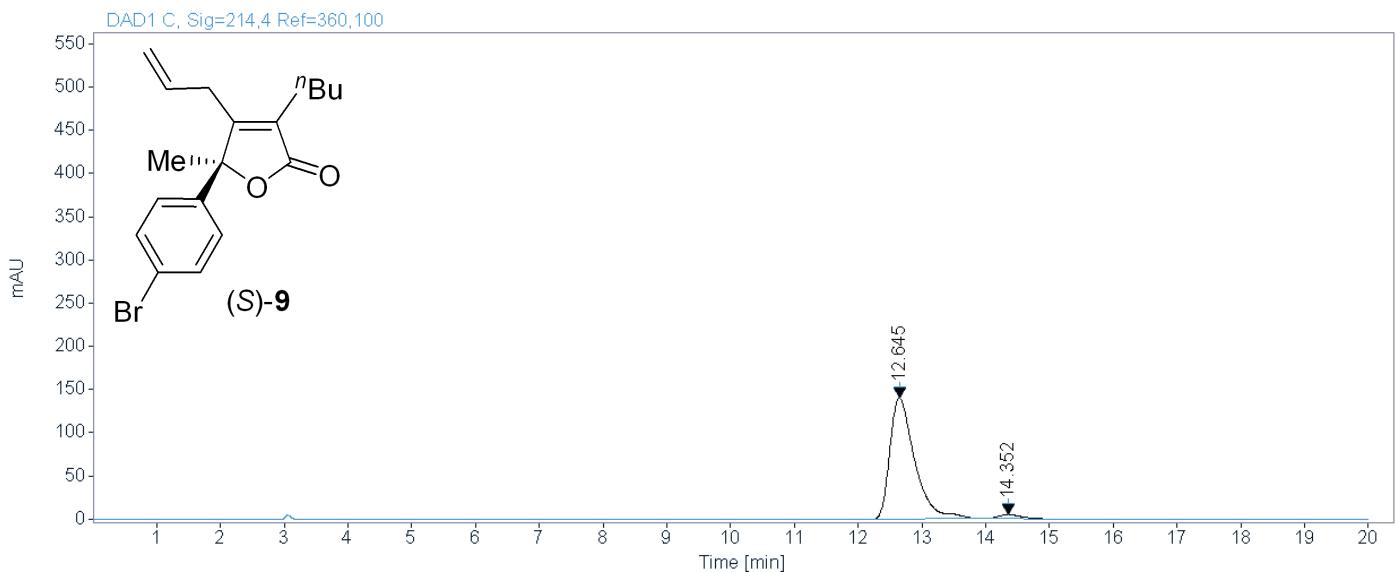
Area Percent Report



sample wj-2-129-OD-H-99.5-0.5-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\zhangq 2020-11-06 15-59-05\012-P2-C1-wj-2-129.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
12.645	0.4127	140.7526	3845.3459	96.6718
14.352	0.4185	4.8197	132.3864	3.3282
		Sum	3977.7323	100.0000

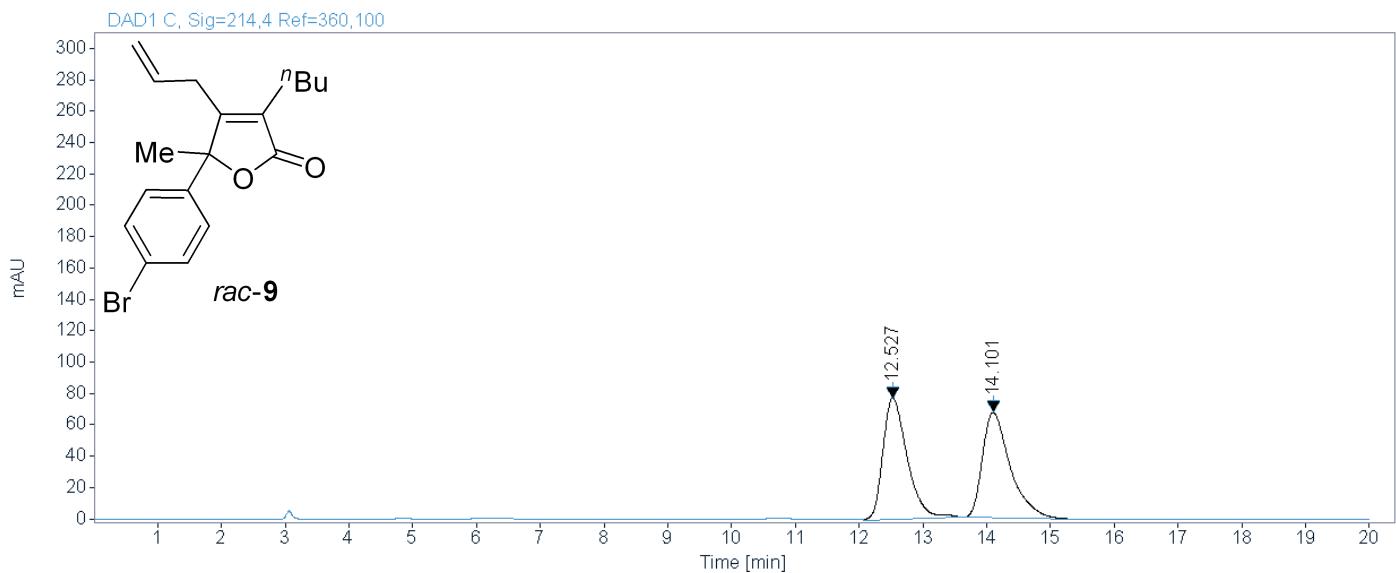
Area Percent Report



sample wj-2-129-rac-OD-H-99.5-0.5-1.0-214

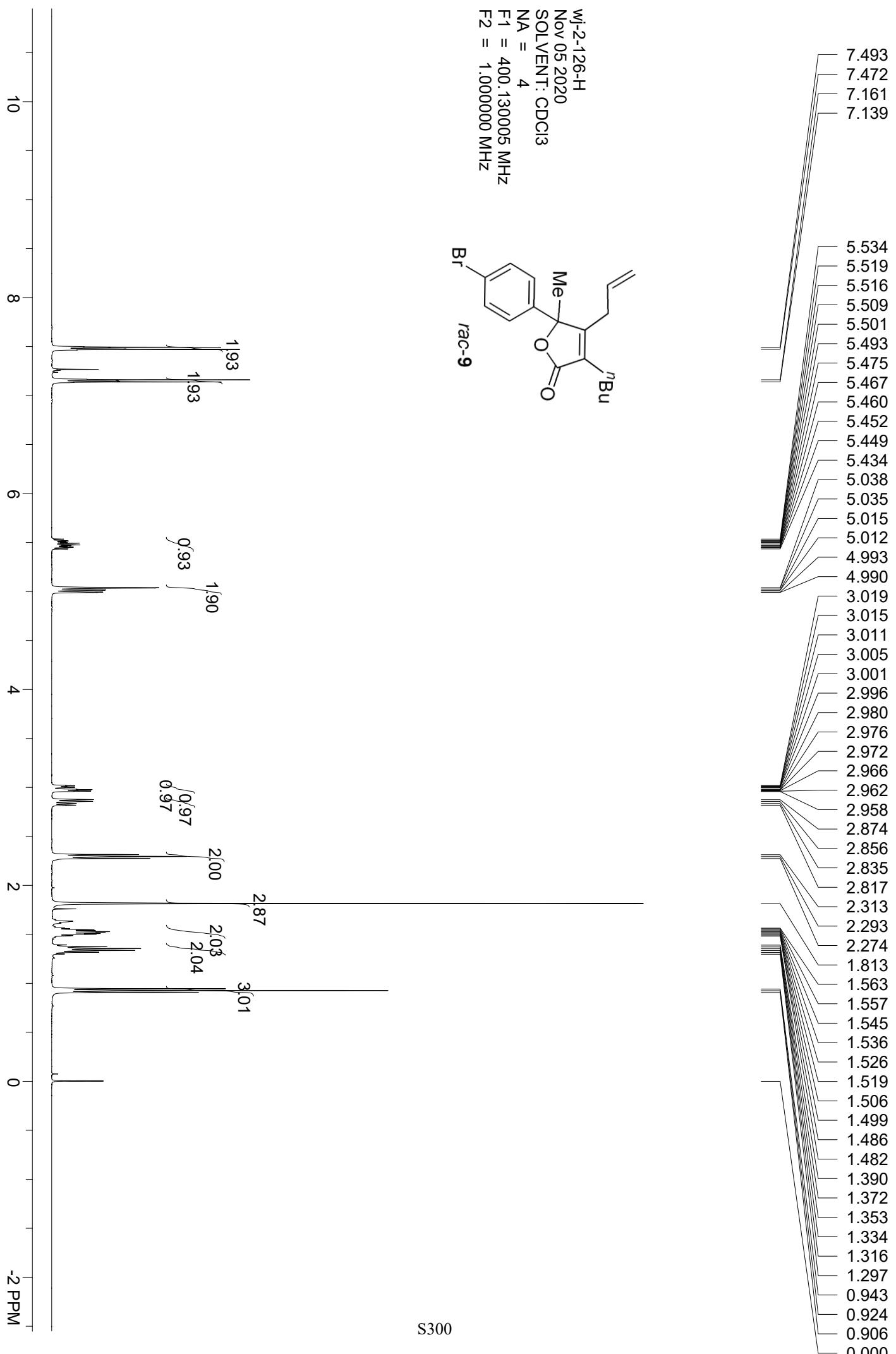
Data file: C:\Users\Public\Documents\ChemStation\1\Data\zhangq 2020-11-06 15-59-05\013-P2-C2-wj-2-129.rac.D

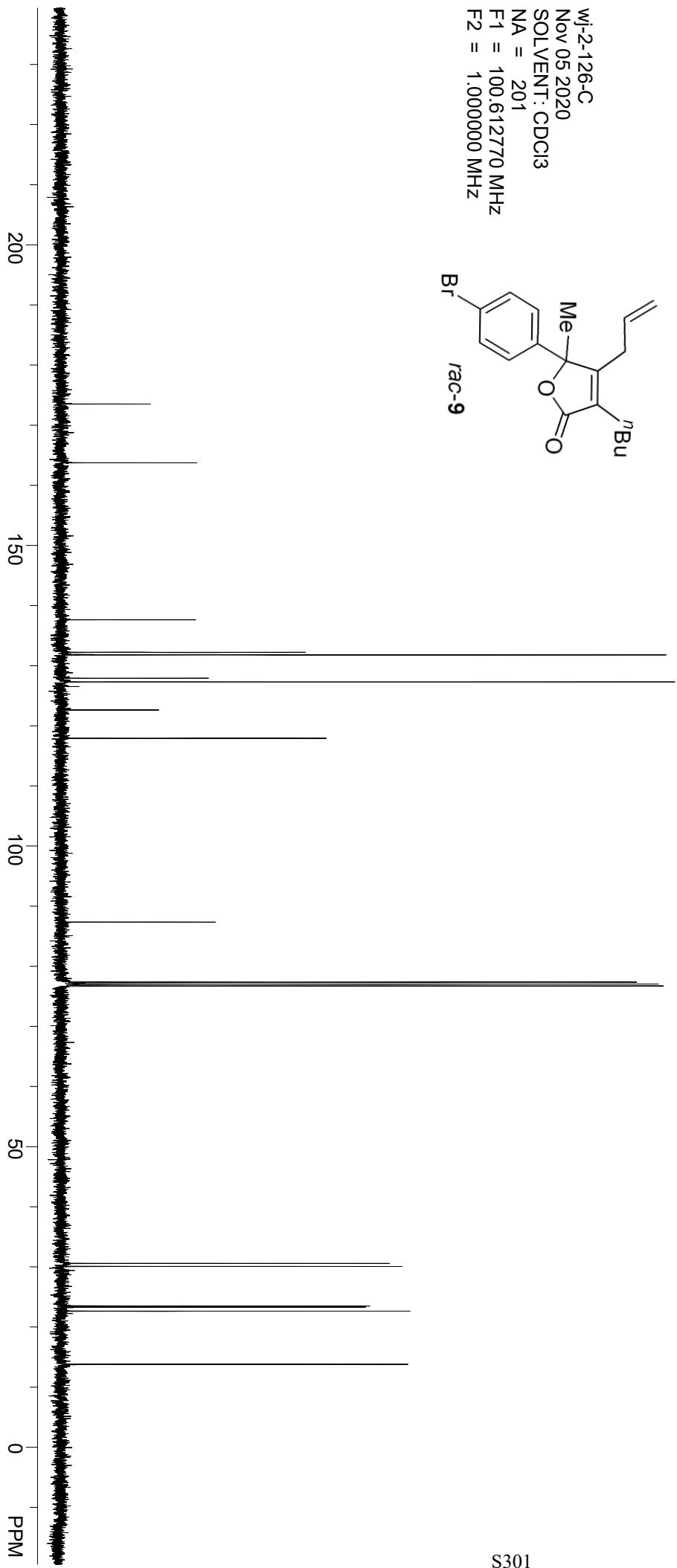
Acquisition Data:

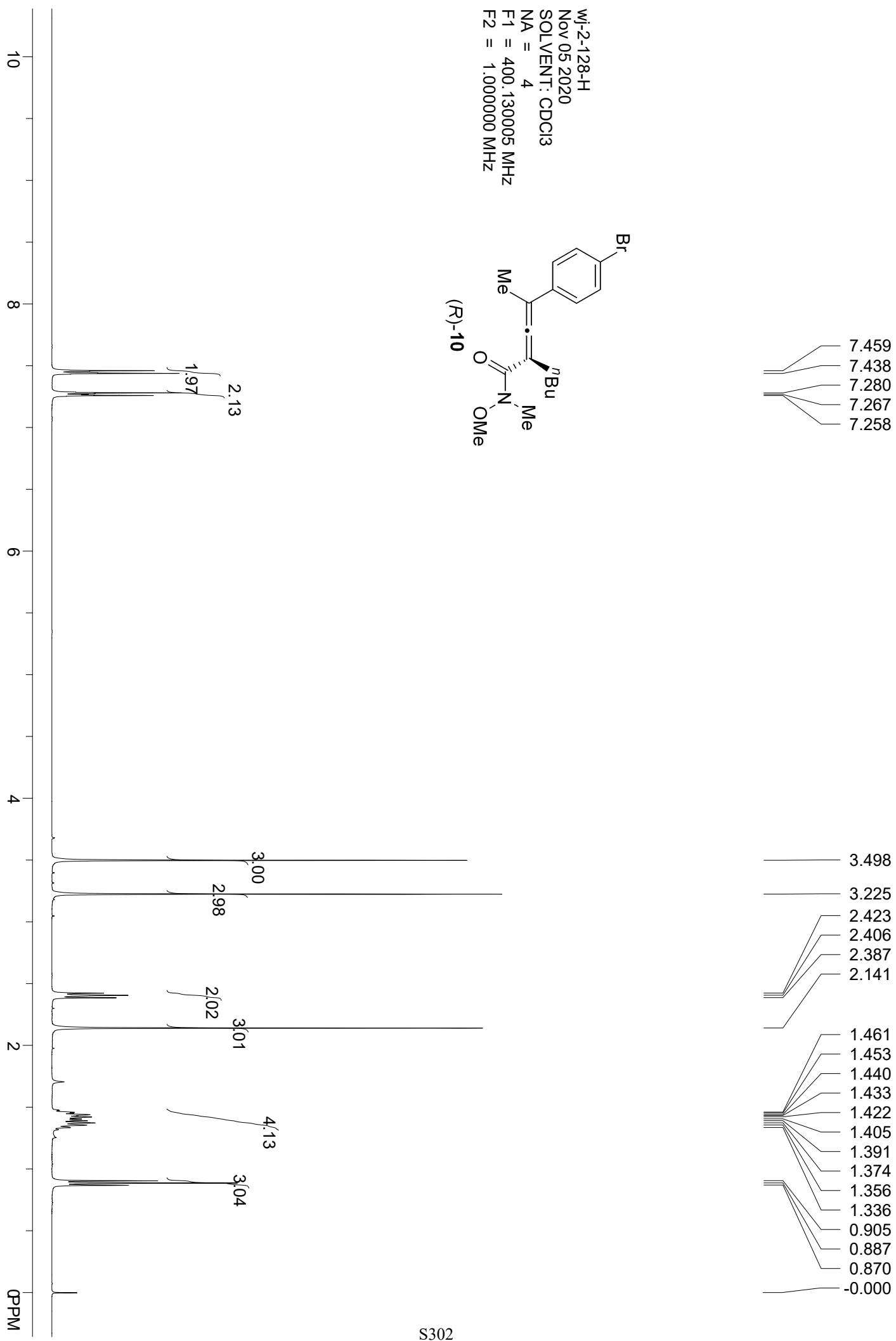


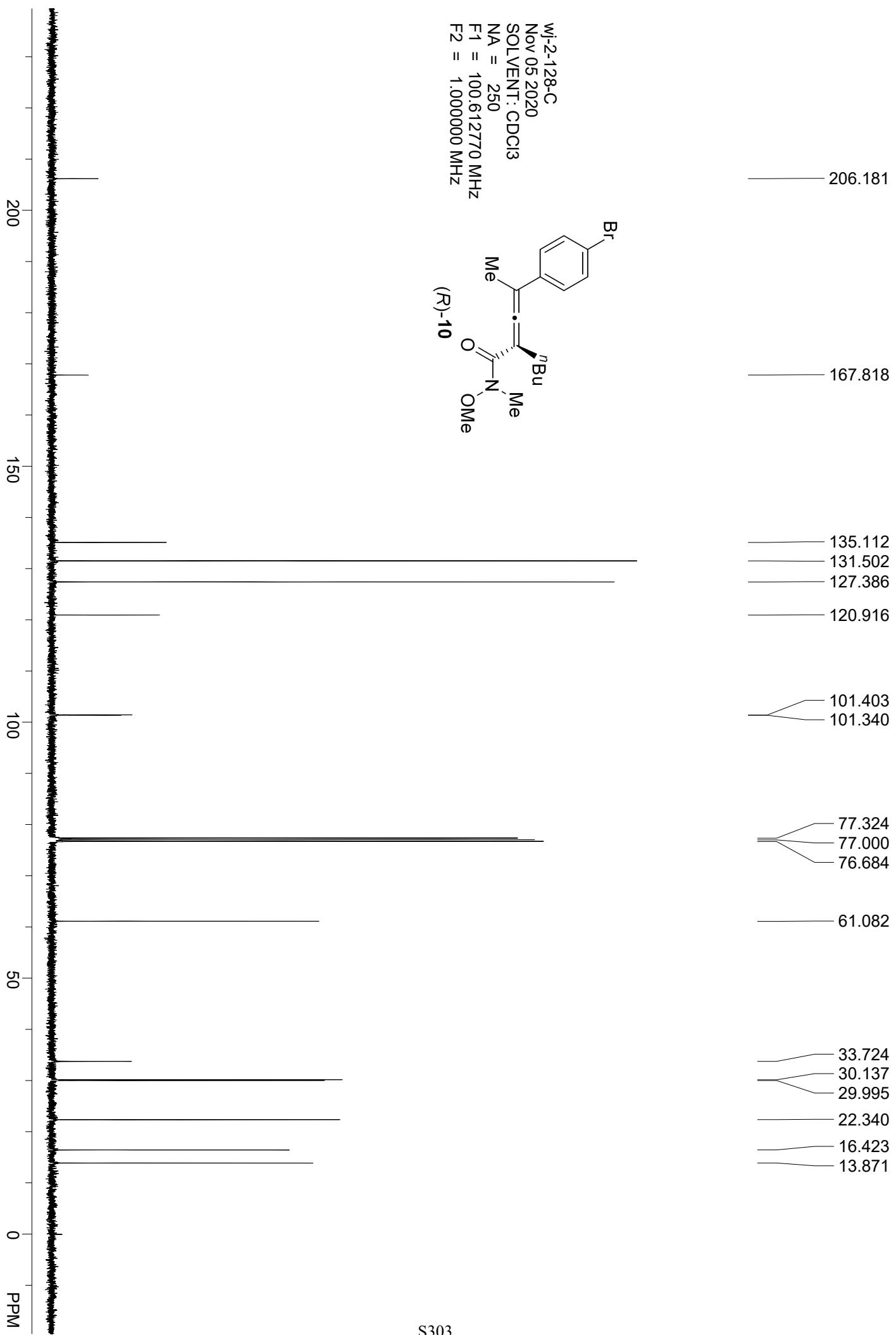
Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
12.527	0.4409	77.3684	2046.8362	50.0784
14.101	0.4582	67.2166	2040.4290	49.9216
Sum		4087.2651	100.0000	









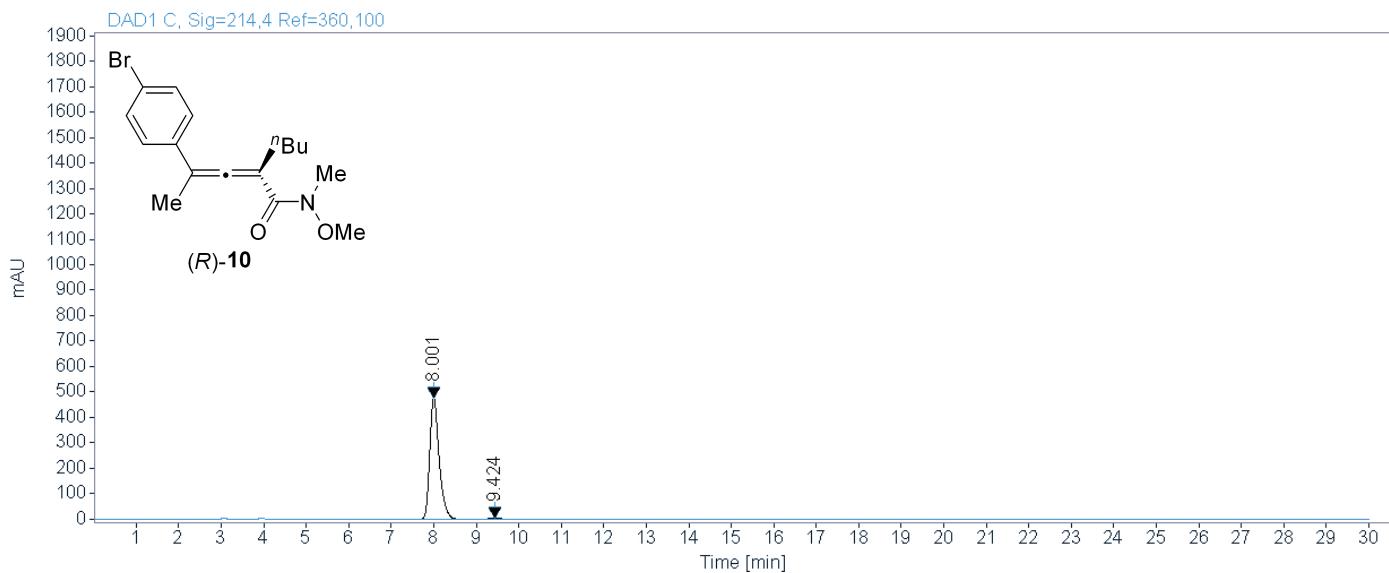
Area Percent Report



sample wj-2-128-OD-H-99-1-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\WJ_LC 2020-11-05 11-10-49\040-P2-C1-wj-2-128.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
8.001	0.2336	477.2816	7345.8462	98.5817
9.424	0.2756	5.7728	105.6875	1.4183
		Sum	7451.5337	100.0000

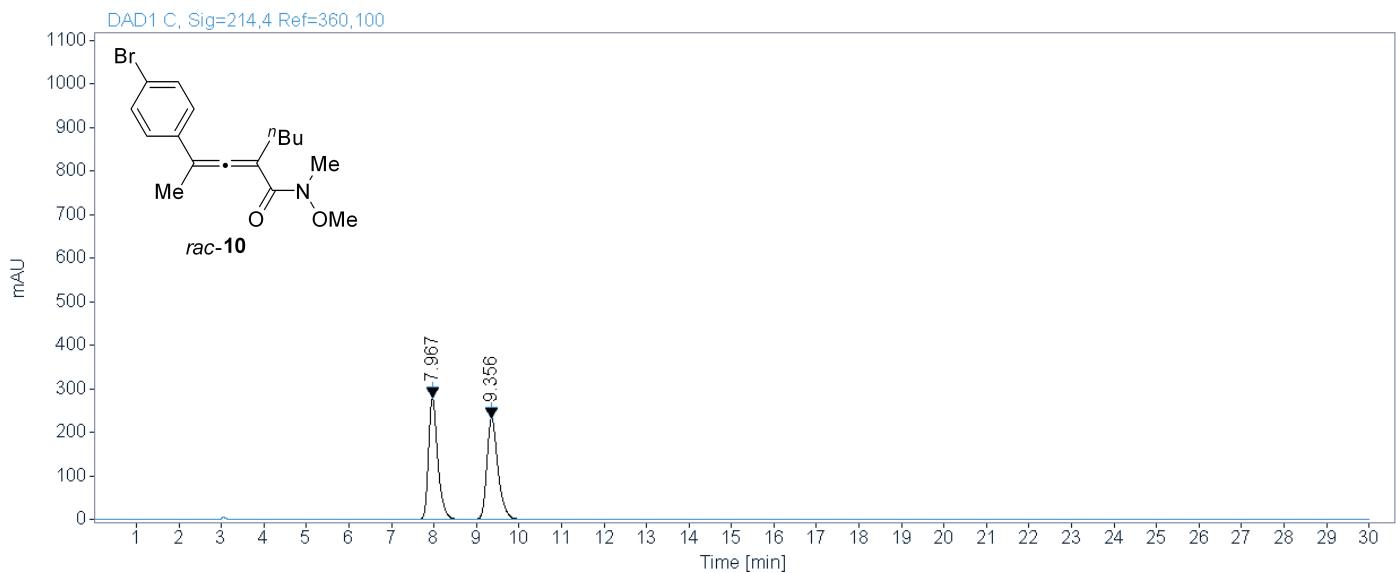
Area Percent Report



sample wj-2-128--rac-OD-H-99-1-1.0-214

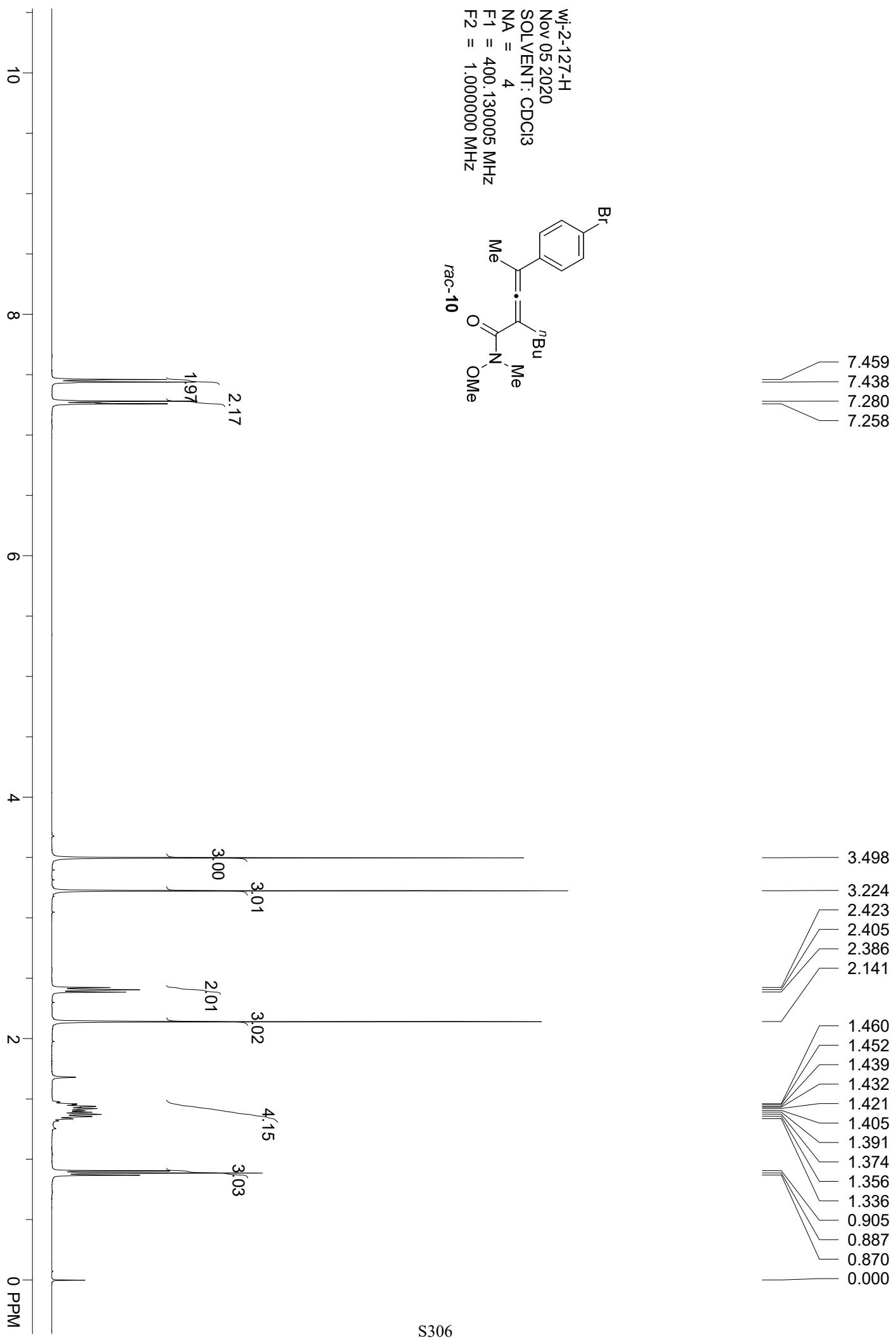
Data file: C:\Users\Public\Documents\ChemStation\1\Data\WJ_LC 2020-11-05 11-10-49\041-P2-C2-wj-2-128-rac.D

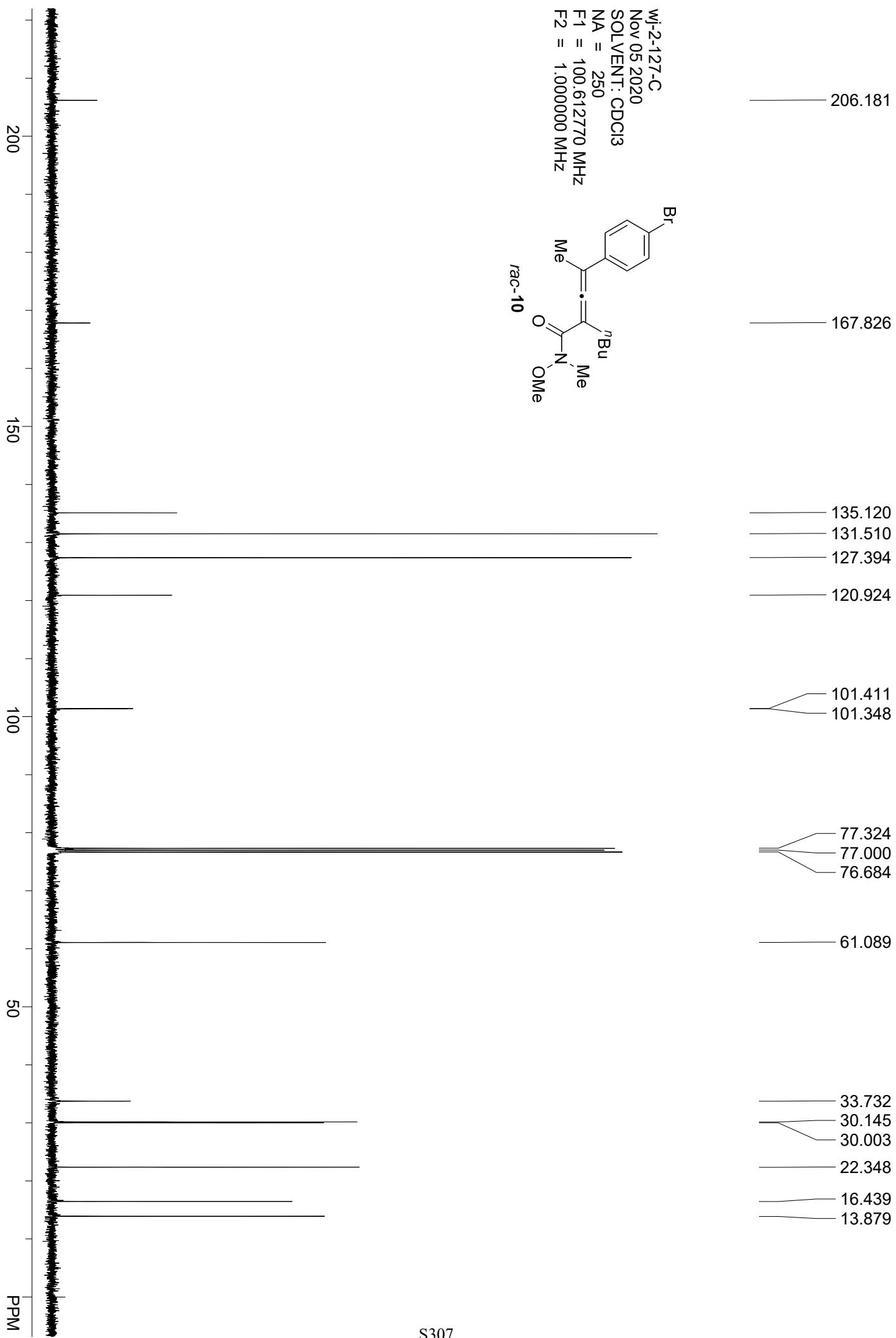
Acquisition Data:

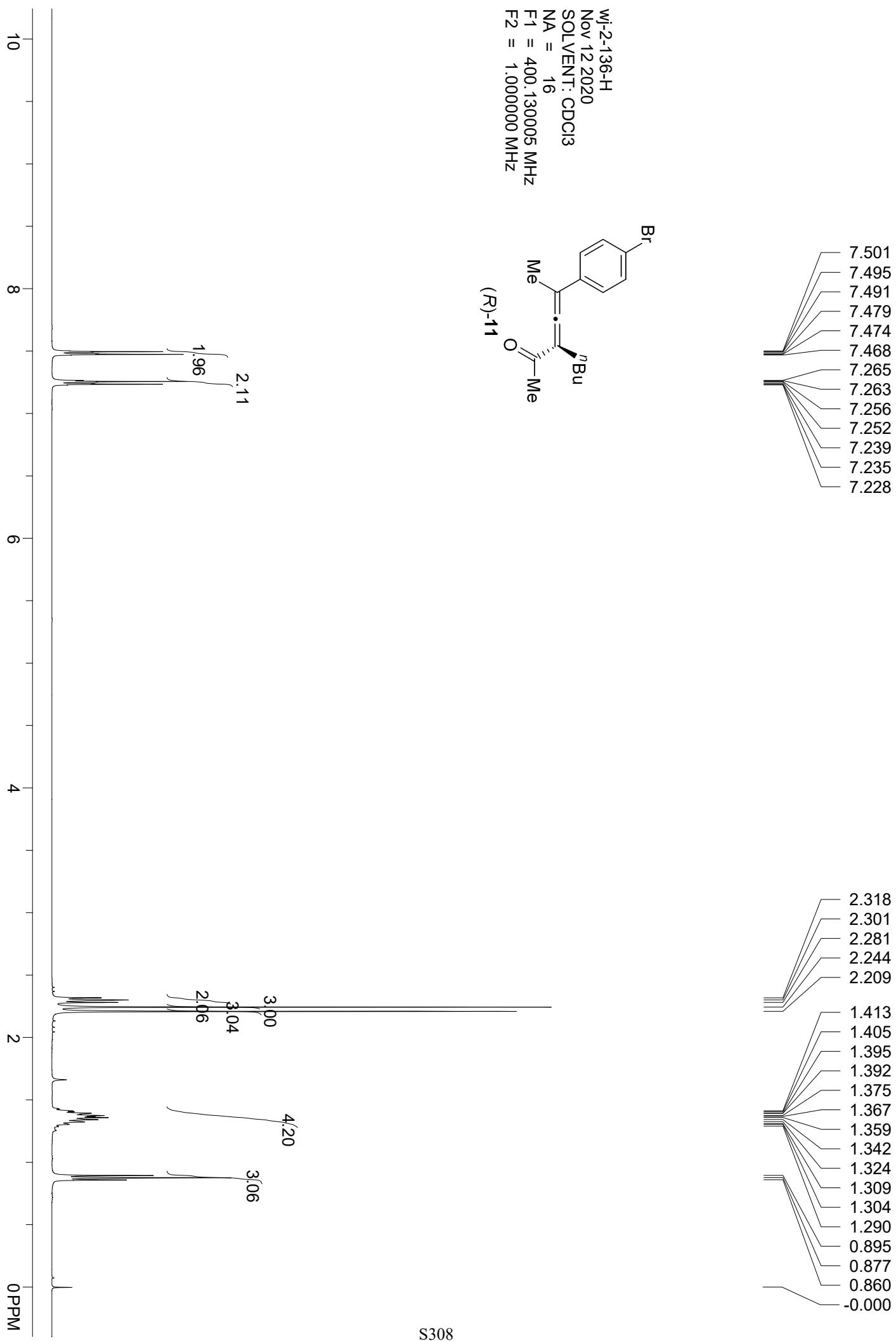


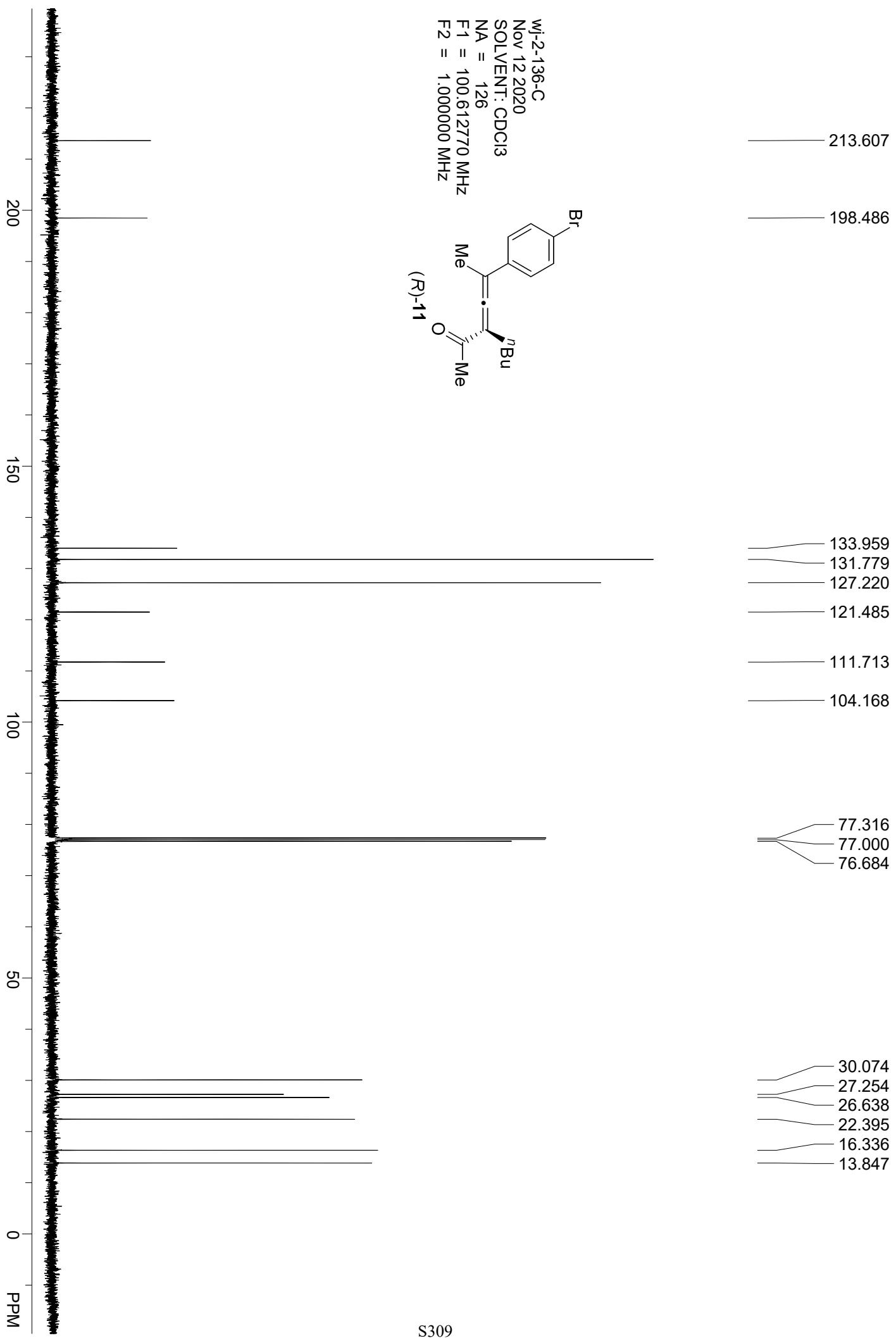
Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
7.967	0.2285	278.4149	4211.4351	50.0043
9.356	0.2778	231.9053	4210.7114	49.9957
Sum		8422.1465	100.0000	









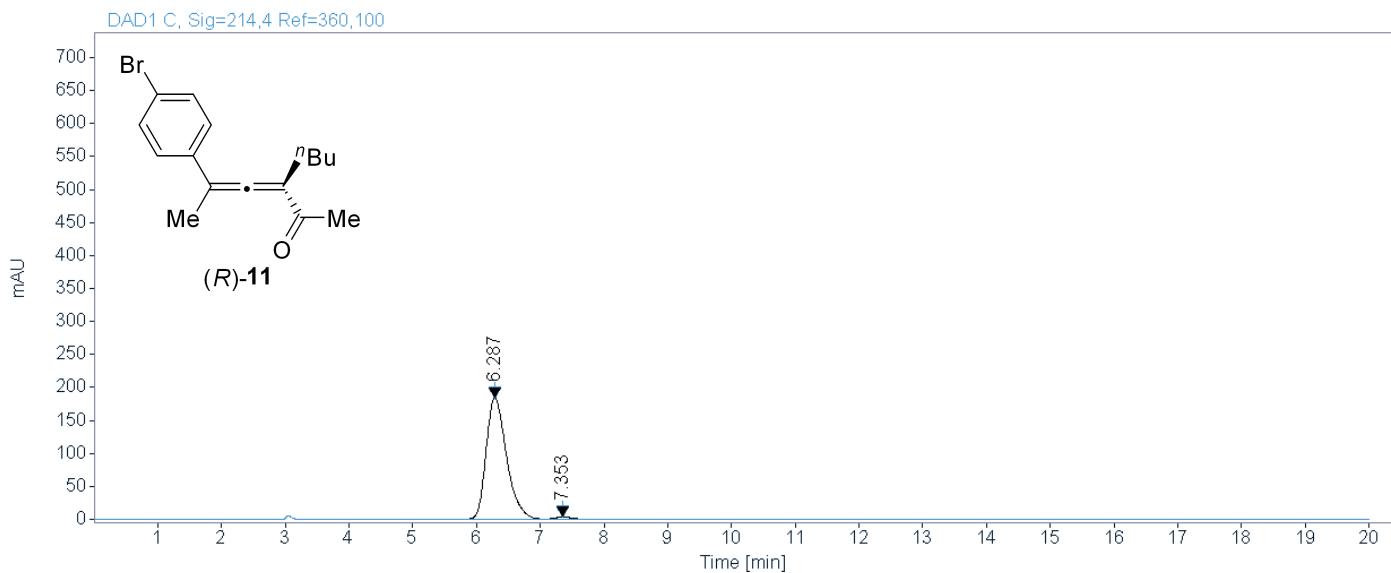
Area Percent Report



sample wj-2-136-OD-H-99.5-0.5-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\WJ_LC 2020-11-11 21-24-07\006-P2-C2-wj-2-136.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
6.287	0.3434	183.9776	4090.1533	98.5656
7.353	0.2664	3.4316	59.5220	1.4344
		Sum	4149.6753	100.0000

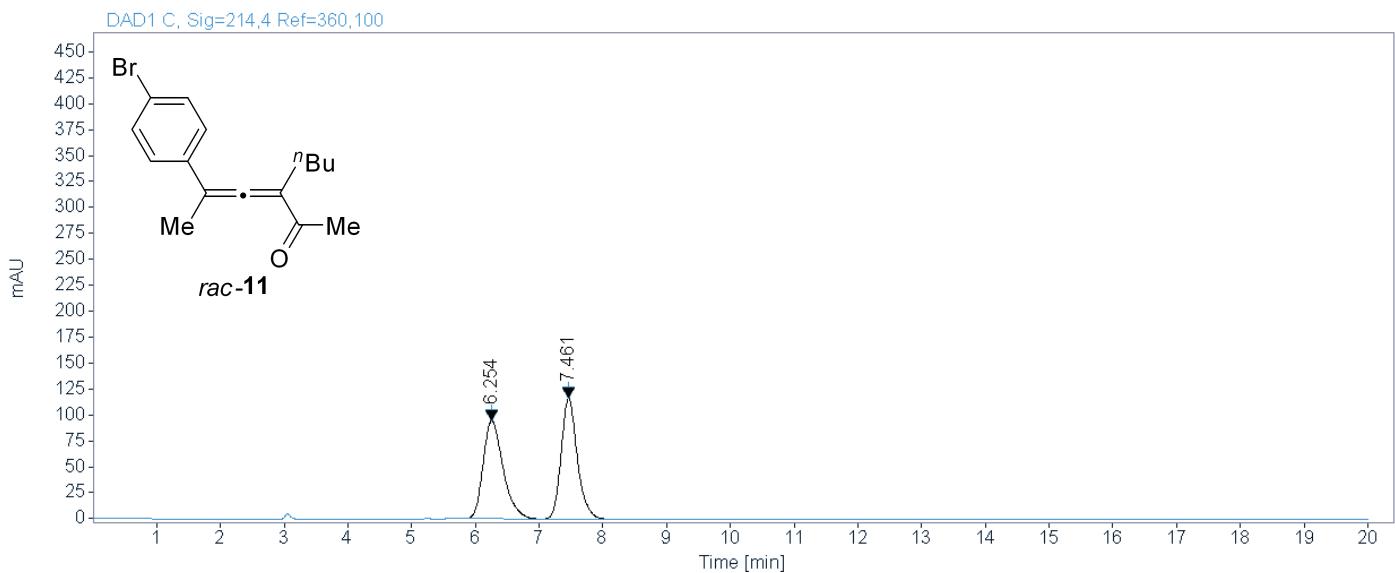
Area Percent Report



sample wj-2-136-rac-OD-H-99.5-0.5-1.0-214

Data file: C:\Users\Public\Documents\ChemStation\1\Data\WJ_LC 2020-11-11 21-24-07\005-P2-C1-wj-2-136-rac.D

Acquisition Data:



Signal: DAD1 C, Sig=214,4 Ref=360,100

RT [min]	Width [min]	Height	Area	Area%
6.254	0.3388	94.2691	2058.8628	49.9882
7.461	0.2697	116.8115	2059.8315	50.0118
Sum		4118.6943	100.0000	

