Supporting Information for

Copper-Catalyzed Asymmetric Propargylic Substitution with Salicylaldehyde-Derived Imine Esters

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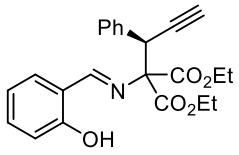
I. General Remarks

^{1}H NMR spectra were recorded on a Bruker 400 MHz spectrometer in $CDCl_3$. Chemical shifts are reported in ppm with the internal TMS signal at 0.0 ppm as a standard. ^{13}C NMR spectra were recorded on a Bruker 100 MHz spectrometer in $CDCl_3$. Chemical shifts are reported in ppm with the internal chloroform signal at 77.0 ppm as a standard. ^{19}F NMR spectra were recorded on a Bruker 376 MHz spectrometer in $CDCl_3$. Chemical shifts are reported in ppm with the internal CF_3COOH signal at -76.55 ppm. The data are reported as (s = single, d = double, t = triple, q = quarter, m = multiple or unresolved, br s = broad single, coupling constant(s) in Hz, integration). Commercially obtained reagents were used without further purification. Solvents were purified prior to use according to the standard methods. Unless otherwise noted, all reactions were carried out under nitrogen atmosphere. The enantiomeric excesses (ee) of the products were determined by high-performance liquid chromatography (HPLC) analysis performed on Agilent 1260 Series chromatographs using a Diacel chiral column (25 cm). Optical rotations were measured on a Rudolph Research Analytical Autopol VI polarimeter with [α]D values reported in degrees; concentration (c) is in g/100 mL. All reactions were reacted under Ar_2 atmosphere. The absolute configuration of **9** was determined by comparing the result of previous report[1], and absolute configurations of other adducts were deduced on the basis of these results.

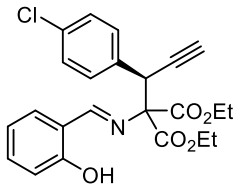
II. General Procedure for the synthesis of 3

In a 10 mL Schlenk tube was placed $Cu(CH_3CN)_4BF_4$ (0.01 mmol) and **L4** (0.01 mmol) under Ar. Anhydrous DCM (1.0 mL) was added, and the mixture was magnetically stirred at room temperature for 30 min. Then the reaction flask was placed in a cool bath of -10 ºC, followed by the addition of **1a** (0.2 mmol), **2** (0.2 mmol), 4-methylmorpholine (0.2 mmol) and anhydrous DCM (1.0 mL) sequentially, and monitored by TLC analysis. After completion, the reaction was quenched with H_2O (3 mL). The aqueous layer was extracted three times with ethyl acetate (6 mL ×3). The combined organic layer was dried over Na_2SO_4. The volatile solvent was removed under reduced pressure. The residue was purified by flash chromatography on silica gel to afford pure **3**.



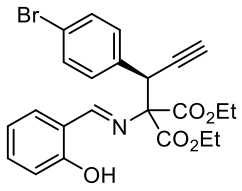
3a

diethyl (*R*,*E*)-2-((2-hydroxybenzylidene)amino)-2-(1-phenylprop-2-yn-1-yl)malonate (3a): 67.4 mg, 86% yield, yellow oil; $[\alpha]^{32}_{D}$ = -48.77 (*c* 1.06, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ 13.06 (s, 1H), 8.45 (s, 1H), 7.40 – 7.26 (m, 7H), 7.02 (d, *J* = 9.0 Hz, 1H), 6.91 (m, 1H), 4.87 (d, *J* = 2.5 Hz, 1H), 4.39 – 4.29 (m, 2H), 4.13 – 4.03 (m, 2H), 2.42 (d, *J* = 2.5 Hz, 1H), 1.31 (t, *J* = 7.1 Hz, 3H), 1.14 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.2, 166.9, 166.5, 161.1, 135.1, 133.3, 132.6, 129.6, 128.21, 128.17, 118.9, 118.7, 117.4, 81.9, 78.7, 74.3, 62.7, 62.4, 44.7, 14.0, 13.8; HRMS (ESI+) Calcd. For $C_{23}H_{24}NO_5^+$ ($[M+H]^+$): 394.1649, found: 394.1652. The product was analyzed by HPLC to determine the enantiomeric excess: 92% ee (Chiralpak IE, *i*-propanol/hexane = 2/98 flow rate 1.0 mL/min, λ = 220 nm); t_r = 19.12 and 21.61 min.



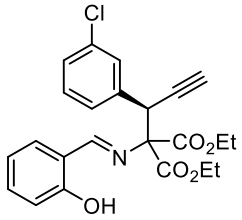
3b

diethyl (*R*,*E*)-2-(1-(4-chlorophenyl)prop-2-yn-1-yl)-2-((2-hydroxybenzylidene)amino)-malonate (3b): 68.4 mg, 80% yield, yellow oil; $[\alpha]^{32}_{D}$ = -76.92 (*c* 1.05, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.96 (s, 1H), 8.46 (s, 1H), 7.43 – 7.34 (m, 1H), 7.33 – 7.27 (m, 3H), 7.24 (m, 2H), 7.02 (d, *J* = 8.2 Hz, 1H), 6.92 (m, 1H), 4.84 (d, *J* = 2.5 Hz, 1H), 4.43 – 4.27 (m, 2H), 4.17 – 3.98 (m, 2H), 2.43 (d, *J* = 2.5 Hz, 1H), 1.31 (t, *J* = 7.1 Hz, 3H), 1.17 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.4, 166.6, 166.4, 161.1, 134.2, 133.7, 133.4, 132.7, 131.0, 128.4, 118.84, 118.76, 117.4, 81.5, 78.5, 74.6, 62.8, 62.5, 44.1, 13.9, 13.8; HRMS (ESI+) Calcd. For $C_{23}H_{23}ClNO_5^+$ ($[M+H]^+$): 428.1259, found: 428.1259. The product was analyzed by HPLC to determine the enantiomeric excess: 94% ee (Chiralpak AD-H, *i*-propanol/hexane = 5/95 flow rate 1.0 mL/min, λ = 262 nm); t_r = 15.49 and 17.68 min.



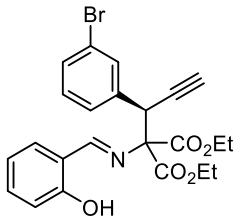
3c

diethyl (*R*,*E*)-2-(1-(4-bromophenyl)prop-2-yn-1-yl)-2-((2-hydroxybenzylidene)amino)-malonate (3c): 78.4 mg, 83% yield, yellow oil; $[\alpha]^{32}_D$ = -87.46 (*c* 1.34, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.96 (s, 1H), 8.46 (s, 1H), 7.42 – 7.35 (m, 3H), 7.30 (dd, *J* = 7.7, 1.7 Hz, 1H), 7.23 (d, *J* = 8.5 Hz, 2H), 7.02 (d, *J* = 7.4 Hz, 1H), 6.94 – 6.90 (m, 1H), 4.83 (d, *J* = 2.5 Hz, 1H), 4.40 – 4.27 (m, 2H), 4.17 – 4.05 (m, 2H), 2.43 (d, *J* = 2.4 Hz, 1H), 1.31 (t, *J* = 7.1 Hz, 3H), 1.17 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.4, 166.6, 166.4, 161.0, 134.2, 133.5, 132.7, 131.33, 131.31, 122.4, 118.9, 118.7, 117.4, 81.4, 78.4, 74.6, 62.9, 62.5, 44.1, 13.9, 13.8; HRMS (ESI+) Calcd. For $C_{23}H_{23}BrNO_5^+$ ($[M+H]^+$): 472.0574, found: 472.0566. The product was analyzed by HPLC to determine the enantiomeric excess: 90% ee (Chiralpak AD-H, *i*-propanol/hexane = 5/95 flow rate 1.0 mL/min, λ = 220 nm); t_r = 16.91 and 18.45 min.



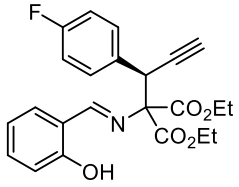
3d

diethyl (*R*,*E*)-2-(1-(3-chlorophenyl)prop-2-yn-1-yl)-2-((2-hydroxybenzylidene)amino)-malonate (3d): 68.3 mg, 80% yield, yellow oil; $[\alpha]^{32}_D$ = -65.74 (*c* 1.01, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.91 (s, 1H), 8.46 (s, 1H), 7.42 – 7.25 (m, 4H), 7.26 – 7.17 (m, 2H), 7.02 (d, *J* = 7.8 Hz, 1H), 6.91 (t, *J* = 7.5 Hz, 1H), 4.84 (d, *J* = 2.5 Hz, 1H), 4.41 – 4.27 (m, 2H), 4.17 – 4.09 (m, 2H), 2.45 (d, *J* = 2.5 Hz, 1H), 1.31 (t, *J* = 7.1 Hz, 3H), 1.18 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.5, 166.6, 166.4, 161.1, 137.1, 133.9, 133.5, 132.7, 129.8, 129.5, 128.3, 127.9, 118.82, 118.77, 117.4, 81.2, 78.5, 74.7, 62.9, 62.6, 44.3, 14.0, 13.8; Calcd. For $C_{23}H_{23}ClNO_5^+$ ($[M+H]^+$): 428.1258, found: 428.1259. The product was analyzed by HPLC to determine the enantiomeric excess: 87% ee (Chiralpak AD-H, *i*-propanol/hexane = 5/95 flow rate 1.0 mL/min, λ = 264 nm); t_r = 13.89 and 20.14 min.



3e

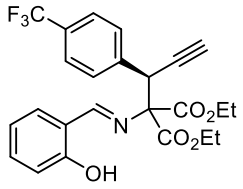
diethyl (*R*,*E*)-2-(1-(3-bromophenyl)prop-2-yn-1-yl)-2-((2-hydroxybenzylidene)amino)-malonate (3e): 79.1 mg, 84% yield, yellow oil; $[\alpha]^{32}_{D}$ = -60.40 (*c* 1.01, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.91 (s, 1H), 8.46 (s, 1H), 7.46 (t, *J* = 1.9 Hz, 1H), 7.42 – 7.29 (m, 4H), 7.16 (t, *J* = 7.9 Hz, 1H), 7.04 – 6.99 (m, 1H), 6.91 (m, 1H), 4.82 (d, *J* = 2.5 Hz, 1H), 4.40 – 4.29 (m, 2H), 4.17 – 4.09 (m, 2H), 2.44 (d, *J* = 2.5 Hz, 1H), 1.31 (t, *J* = 7.1 Hz, 3H), 1.19 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.5, 166.6, 166.3, 161.1, 137.3, 133.5, 132.70, 132.67, 131.2, 129.8, 128.3, 122.0, 118.82, 118.75, 117.4, 81.2, 78.5, 74.7, 62.9, 62.6, 44.3, 14.0, 13.8; HRMS (ESI+) Calcd. For $C_{23}H_{23}BrNO_5^+$ ($[M+H]^+$): 472.0571, found: 472.0566. The product was analyzed by HPLC to determine the enantiomeric excess: 87% ee (Chiralpak AD-H, *i*-propanol/hexane = 5/95 flow rate 1.0 mL/min, λ = 220 nm); t_r = 14.14 and 22.15 min.



3f

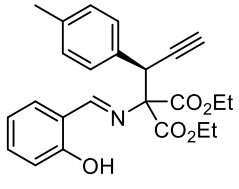
diethyl (*R*,*E*)-2-(1-(4-fluorophenyl)prop-2-yn-1-yl)-2-((2-hydroxybenzylidene)amino)-malonate (3f): 72.3 mg, 88% yield, yellow oil; $[\alpha]^{32}_{D}$ = -48.81 (*c* 1.18, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ 13.00 (s, 1H), 8.45 (s, 1H), 7.41 – 7.36 (m, 1H), 7.34 – 7.29 (m, 3H), 7.02 (d, *J* = 8.3 Hz, 1H), 6.99 – 6.89 (m, 3H), 4.85 (d, *J* = 2.5 Hz, 1H), 4.38 – 4.29 (m, 2H), 4.15 – 4.06 (m, 2H), 2.43 (d, *J* = 2.5 Hz, 1H), 1.31 (t, *J* = 7.1 Hz, 3H), 1.16 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.4, 166.7, 166.5, 162.5 (d, *J* = 245.9 Hz), 161.1, 133.4, 132.7, 131.3 (d, *J* = 8.1 Hz), 130.9 (d, *J* = 3.1 Hz), 118.9 (d, *J* = 8.0 Hz), 117.4, 115.3, 115.0, 81.7, 78.6, 74.5, 62.8, 62.5, 43.9, 14.0, 13.8; ^{19}F NMR (376 MHz, $CDCl_3$) δ -113.82; HRMS (ESI+) Calcd. For $C_{23}H_{23}FNO_5^+$ ($[M+H]^+$): 412.1555, found: 412.1553. The product was analyzed by HPLC

to determine the enantiomeric excess: 90% ee (Chiralpak IE, *i*-propanol/hexane = 5/95 flow rate 1.0 mL/min, λ = 254 nm); t_r = 11.28 and 12.72 min.



3g

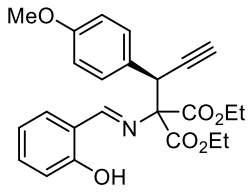
diethyl (*R*,*E*)-2-((2-hydroxybenzylidene)amino)-2-(1-(4-(trifluoromethyl)phenyl)prop-2-yn-1-yl)malonate (3g): 82.9 mg, 90% yield, yellow oil; $[\alpha]^{32}_D$ = -57.19 (*c* 1.14, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.92 (s, 1H), 8.48 (s, 1H), 7.56 – 7.47 (m, 4H), 7.42 – 7.37 (m, 1H), 7.31 (dd, *J* = 7.7, 1.7 Hz, 1H), 7.02 (d, *J* = 8.3 Hz, 1H), 6.95 – 6.91 (m, 1H), 4.92 (d, *J* = 2.5 Hz, 1H), 4.41 – 4.29 (m, 2H), 4.15 – 4.05 (m, 2H), 2.46 (d, *J* = 2.5 Hz, 1H), 1.32 (t, *J* = 7.1 Hz, 3H), 1.14 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.6, 166.6, 166.4, 161.1, 139.3, 133.6, 132.7, 130.4 (q, *J* = 32.3 Hz), 130.1, 125.1 (q, *J* = 4.0 Hz), 123.9 (q, *J* = 272.7 Hz), 118.9, 118.7, 117.4, 81.1, 78.5, 74.9, 63.0, 62.6, 44.4, 13.9, 13.7; ^{19}F NMR (376 MHz, $CDCl_3$) δ -62.66; HRMS (ESI+) Calcd. For $C_{24}H_{23}F_3NO_5^+$ ($[M+H]^+$): 462.1523, found: 462.1522. The product was analyzed by HPLC to determine the enantiomeric excess: 92% ee (Chiralpak IE, *i*-propanol/hexane = 5/95 flow rate 1.0 mL/min, λ = 254 nm); t_r = 7.52 and 8.27 min.



3h

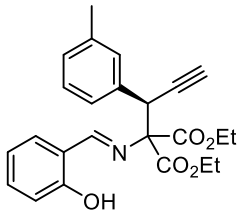
diethyl (*R*,*E*)-2-((2-hydroxybenzylidene)amino)-2-(1-(p-tolyl)prop-2-yn-1-yl)malonate (3h): 65.2 mg, 80% yield, yellow oil; $[\alpha]^{32}_D$ = -56.79 (*c* 1.12, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ 13.10 (s, 1H), 8.46 (s, 1H), 7.37 (m, 1H), 7.29 (dd, *J* = 7.7, 1.7 Hz, 1H), 7.24 – 7.19 (m, 2H), 7.07 (d, *J* = 7.8 Hz, 2H), 7.03 – 7.00 (m, 1H), 6.93 – 6.89 (m, 1H), 4.83 (d, *J* = 2.5 Hz, 1H), 4.38 – 4.28 (m, 2H), 4.14 – 4.04 (m, 2H), 2.40 (d, *J* = 2.5 Hz, 1H), 2.29 (s, 3H), 1.31 (t, *J* = 7.1 Hz, 3H), 1.16 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.2, 166.9, 166.5,

161.1, 137.9, 133.2, 132.6, 132.0, 129.4, 128.9, 118.9, 118.7, 117.4, 82.1, 78.7, 74.1, 62.7, 62.3, 44.4, 21.1, 14.0, 13.8; HRMS (ESI+) Calcd. For $C_{24}H_{26}NO_5^+$ ($[M+H]^+$): 408.1805, found: 408.1811. The product was analyzed by HPLC to determine the enantiomeric excess: 94% ee (Chiralpak AD-H, *i*-propanol/hexane = 5/95 flow rate 1.0 mL/min, λ = 262 nm); t_r = 17.79 and 20.48 min.



3i

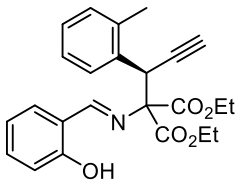
diethyl (*R,E*)-2-((2-hydroxybenzylidene)amino)-2-(1-(4-methoxyphenyl)prop-2-yn-1-yl)malonate (3i): 80.1 mg, 95% yield, yellow oil; $[\alpha]^{32}_D$ = -90.80 (*c* 1.38, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ 13.11 (s, 1H), 8.44 (s, 1H), 7.40 – 7.35 (m, 1H), 7.32 – 7.25 (m, 3H), 7.02 (d, *J* = 8.2 Hz, 1H), 6.91 (t, *J* = 7.5 Hz, 1H), 6.80 (d, *J* = 8.7 Hz, 2H), 4.82 (d, *J* = 2.5 Hz, 1H), 4.38 – 4.28 (m, 2H), 4.15 – 4.05 (m, 2H), 3.75 (s, 3H), 2.41 (d, *J* = 2.4 Hz, 1H), 1.31 (t, *J* = 7.1 Hz, 3H), 1.17 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.2, 166.9, 166.6, 161.1, 159.4, 133.3, 132.6, 130.7, 127.1, 118.9, 118.7, 117.4, 113.6, 82.2, 78.8, 74.1, 62.7, 62.4, 55.2, 44.0, 14.0, 13.9; HRMS (ESI+) Calcd. For $C_{24}H_{26}NO_6^+$ ($[M+H]^+$): 424.1755, found: 424.1753. The product was analyzed by HPLC to determine the enantiomeric excess: 92% ee (Chiralpak AD-H, *i*-propanol/hexane = 2/98 flow rate 1.0 mL/min, λ = 254 nm); t_r = 38.30 and 46.27 min.



3j

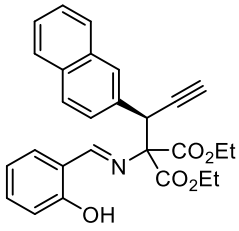
diethyl (*R,E*)-2-((2-hydroxybenzylidene)amino)-2-(1-(m-tolyl)prop-2-yn-1-yl)malonate (3j): 69.3 mg, 85% yield, yellow oil; $[\alpha]^{32}_D$ = -54.85 (*c* 1.36, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ 13.05 (s, 1H), 8.42 (s, 1H), 7.39 – 7.34 (m, 1H), 7.30 – 7.26 (m, 1H), 7.14 (t, *J* = 6.4 Hz, 3H), 7.07 (s, 1H), 7.02 (d, *J* = 8.2 Hz, 1H), 6.92 – 6.88 (m, 1H), 4.83 (d, *J* = 2.5 Hz, 1H),

4.40 – 4.28 (m, 2H), 4.13 – 4.04 (m, 2H), 2.41 (d, *J* = 2.5 Hz, 1H), 2.27 (s, 3H), 1.31 (t, *J* = 7.1 Hz, 3H), 1.15 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.9, 166.9, 166.6, 161.2, 137.8, 134.9, 133.2, 132.6, 130.4, 128.9, 128.1, 126.6, 118.9, 118.7, 117.4, 82.0, 78.7, 74.2, 62.7, 62.3, 44.7, 21.3, 14.0, 13.8; HRMS (ESI+) Calcd. For $C_{24}H_{26}NO_5^+$ ($[M+H]^+$): 408.1805, found: 408.1806. The product was analyzed by HPLC to determine the enantiomeric excess: 92% ee (Chiralpak AD-H, *i*-propanol/hexane = 5/95 flow rate 1.0 mL/min, λ = 220 nm); t_r = 14.09 and 17.76 min.



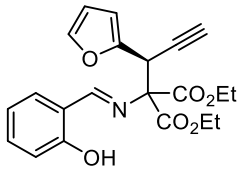
3k

diethyl (*R*,*E*)-2-((2-hydroxybenzylidene)amino)-2-(1-(o-tolyl)prop-2-yn-1-yl)malonate (3k): 61.1 mg, 75% yield, yellow oil; $[\alpha]^{32}_D$ = -50.00 (*c* 1.17, CH_2Cl_2); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 13.16 (s, 1H), 8.54 (s, 1H), 7.47 – 7.42 (m, 1H), 7.39 (ddd, *J* = 8.6, 7.3, 1.7 Hz, 1H), 7.34 (dd, *J* = 7.7, 1.7 Hz, 1H), 7.15 – 7.08 (m, 3H), 7.04 (dd, *J* = 8.3, 1.0 Hz, 1H), 6.95 – 6.91 (m, 1H), 5.16 (d, *J* = 2.5 Hz, 1H), 4.42 – 4.27 (m, 2H), 4.06 – 3.87 (m, 2H), 2.39 (s, 3H), 2.38 (d, *J* = 2.4 Hz, 1H), 1.30 (t, *J* = 7.1 Hz, 3H), 0.98 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 168.9, 167.1, 166.8, 161.1, 135.9, 134.3, 133.3, 132.6, 130.3, 129.5, 127.9, 126.3, 118.9, 118.8, 117.4, 82.4, 78.0, 73.9, 62.8, 62.3, 39.4, 19.7, 13.9, 13.5; HRMS (ESI+) Calcd. For $C_{24}H_{26}NO_5^+$ ($[M+H]^+$): 408.1805, found: 408.1802. The product was analyzed by HPLC to determine the enantiomeric excess: 76% ee (Chiralpak AD-H, *i*-propanol /hexane = 5/95 flow rate 1.0 mL/min, λ = 220 nm); t_r = 13.02 and 14.71 min.



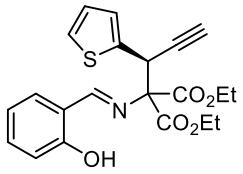
3l

diethyl (*R*,*E*)-2-((2-hydroxybenzylidene)amino)-2-(1-(naphthalen-2-yl)prop-2-yn-1-yl)malonate (3l): 80.7 mg, 91% yield, yellow oil; $[\alpha]^{32}_D$ = -90.87 (*c* 1.15, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ 13.11 (s, 1H), 8.47 (s, 1H), 7.82 – 7.73 (m, 4H), 7.51 – 7.36 (m, 4H), 7.30 – 7.26 (m, 1H), 7.04 (d, *J* = 8.3 Hz, 1H), 6.93 – 6.89 (m, 1H), 5.04 (d, *J* = 2.5 Hz, 1H), 4.41 – 4.30 (m, 2H), 4.04 (q, *J* = 7.1 Hz, 2H), 2.47 (d, *J* = 2.5 Hz, 1H), 1.32 (t, *J* = 7.1 Hz, 3H), 1.05 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.2, 166.9, 166.5, 161.2, 133.3, 133.0, 132.9, 132.7, 132.6, 129.0, 127.93, 127.90, 127.6, 127.1, 126.2, 126.1, 118.9, 118.8, 117.4, 81.9, 78.9, 74.5, 62.8, 62.4, 44.9, 14.0, 13.7; HRMS (ESI+) Calcd. For $C_{27}H_{26}NO_5^+$ ($[M+H]^+$): 444.1805, found: 444.1805. The product was analyzed by HPLC to determine the enantiomeric excess: 91% ee (Chiralpak AD-H, *i*-propanol /hexane = 5/95 flow rate 1.0 mL/min, λ = 220 nm); t_r = 27.07 and 28.87 min.



3m

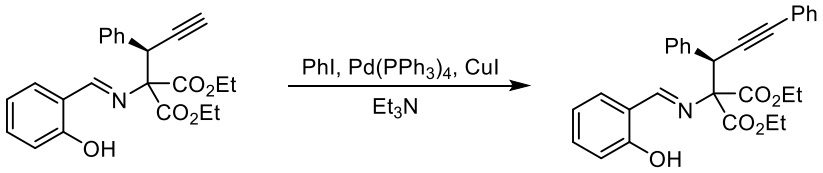
diethyl (*S*,*E*)-2-(1-(furan-2-yl)prop-2-yn-1-yl)-2-((2-hydroxybenzylidene)amino)-malonate (3m): 66.6 mg, 87% yield, yellow oil; $[\alpha]^{32}_D$ = -33.00 (*c* 1.00, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.90 (s, 1H), 8.55 (s, 1H), 7.37 – 7.29 (m, 3H), 6.97 (d, *J* = 8.3 Hz, 1H), 6.89 (m, 1H), 6.40 (dt, *J* = 3.3, 0.8 Hz, 1H), 6.31 (dd, *J* = 3.2, 1.9 Hz, 1H), 5.05 (d, *J* = 2.6 Hz, 1H), 4.39 – 4.30 (m, 2H), 4.26 (q, *J* = 7.1 Hz, 2H), 2.38 (d, *J* = 2.5 Hz, 1H), 1.33 (t, *J* = 7.1 Hz, 3H), 1.27 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.3, 166.3, 166.2, 161.1, 148.7, 142.5, 133.2, 132.7, 119.0, 118.7, 117.3, 110.7, 110.1, 79.2, 77.2, 73.6, 62.9, 62.8, 39.1, 14.0, 13.9; HRMS (ESI+) Calcd. For $C_{21}H_{22}NO_6^+$ ($[M+H]^+$): 384.1442, found: 384.1440. The product was analyzed by HPLC to determine the enantiomeric excess: 93% ee (Chiralpak IE, *i*-propanol/hexane = 5/95 flow rate 1.0 mL/min, λ = 254 nm); t_r = 18.62 and 21.27 min.



3n

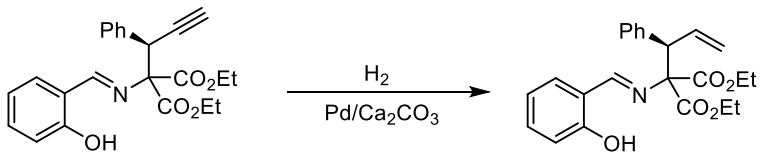
diethyl (*S*,*E*)-2-((2-hydroxybenzylidene)amino)-2-(1-(thiophen-2-yl)prop-2-yn-1-yl)-malonate (3n): 70.3 mg, 88% yield, yellow oil; $[\alpha]^{32}_{D}$ = -45.98 (*c* 1.02, CH_2Cl_2); ^{1}H NMR (400 MHz, $CDCl_3$) δ 12.92 (s, 1H), 8.55 (s, 1H), 7.39 – 7.34 (m, 1H), 7.31 – 7.29 (m, 1H), 7.22 – 7.20 (m, 1H), 7.07 – 6.99 (m, 2H), 6.92 – 6.87 (m, 2H), 5.21 (d, *J* = 2.5 Hz, 1H), 4.39 – 4.29 (m, 2H), 4.25 – 4.13 (m, 2H), 2.45 (d, *J* = 2.5 Hz, 1H), 1.32 (t, *J* = 7.1 Hz, 3H), 1.23 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.3, 166.4, 166.2, 161.0, 137.2, 133.3, 132.8, 128.2, 126.3, 118.9, 118.7, 117.3, 81.3, 78.4, 74.1, 62.9, 62.7, 40.2, 13.9, 13.8; HRMS (ESI+) Calcd. For $C_{21}H_{22}NO_5S^+$ ($[M+H]^+$): 400.1213, found: 400.1209. The product was analyzed by HPLC to determine the enantiomeric excess: 90% ee (Chiralpak AD-H, *i*-propanol/hexane = 5/95 flow rate 1.0 mL/min, λ = 220 nm); t_r = 26.81 and 33.63 min.

III. Synthetic Transformation



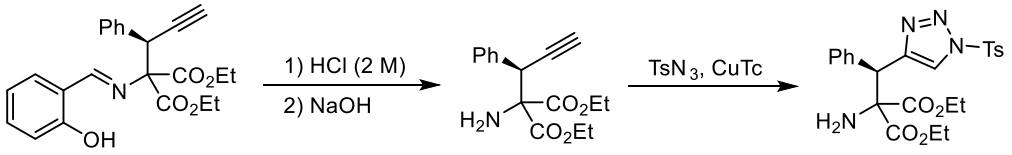
3a **4**, 99% yield, 92% ee

A mixture of **3a** (0.2 mmol, 92% ee), iodobenzene (0.40 mmol), $Pd(PPh_3)_4$ (0.02 mmol), and CuI (0.04 mmol) in Et_3N (2 mL) was stirred at 25 ºC for 20 h.[2,3] After the reaction was completed, the crude reaction mixture was filtrated with celite and washed with EtOAc. The solvents were removed under reduced pressure. Then the residue was purified by silica gel column chromatography to afford the desired product **4**.



3a **5**, 90% yield, 93% ee

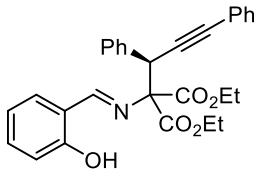
To a solution of **3a** (0.20 mmol) in EtOH (2 mL) under nitrogen, then 10 mg Pd-$CaCO_3$ was added. The reaction mixture was stirred under H_2 atmosphere (1 atm) at 25 ºC for 3d.[2] After the reaction was completed (monitored by TLC), the crude reaction mixture was filtered over a short pad of celite and washed with EtOAc. The solvents were removed under reduced pressure. Then the residue was purified by silica gel column chromatography to afford the desired product **5**.



3a **6**, 80% yield **7**, 71% yield, 93% ee

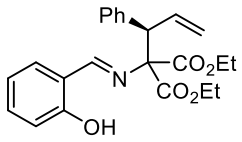
To a solution of **3a** (0.30 mmol) in THF (1 mL), then 1 mL 2 M HCl was added. The reaction mixture was stirred at 25 ºC for 2 h. After the reaction was complete (monitored by TLC), THF was removed under reduced pressure. The crude reaction mixture was extracted with EtOAc (3 × 3 mL) and the aqueous solution was added with 10% NaOH until pH = 10, then the crude reaction mixture was extracted with DCM (3 × 3 mL), the organics were combined and dried over Na_2SO_4 and concentrated under vacuum and purified by silica-gel flash chromatography to afford the desired product **6**.

A mixture of **6** (0.20 mmol), copper(I) thiophene-2-carboxylate (CuTc, 0.02 mmol) in anhydrous toluene (1.5 mL) was cooled in an ice-water bath. Subsequently, the tosyl azide (0.24 mmol) was added slowly, then the reaction mixture was allowed to warm to room temperature and stir until complete (monitored by TLC).[2,3] The reaction was quenched by saturated NH_4Cl aqueous solution (3 mL) and extracted into EtOAc (3 × 5 mL). The combined organic layers were dried over Na_2SO_4, filtrated and concentrated in vacuo. Then the residue was purified by silica gel column chromatography to afford the desired product **7**.



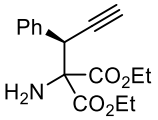
4

diethyl (*S*,*E*)-2-(1,3-diphenylprop-2-yn-1-yl)-2-((2-hydroxybenzylidene)amino)malonate (4): 93.0 mg, 99% yield, yellow oil; $[\alpha]^{32}_{D}$ = -8.59 (*c* 0.92, CH_2Cl_2); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 13.18 (s, 1H), 8.46 (s, 1H), 7.42 – 7.32 (m, 5H), 7.30 – 7.24 (m, 7H), 7.03 (dd, *J* = 8.4, 1.0 Hz, 1H), 6.91 – 6.87 (m, 1H), 5.09 (s, 1H), 4.42 – 4.26 (m, 2H), 4.14 – 4.05 (m, 2H), 1.30 (t, *J* = 7.1 Hz, 3H), 1.14 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 168.8, 167.0, 166.8, 161.3, 135.6, 133.2, 132.5, 131.7, 129.7, 128.2, 128.13, 128.07, 122.9, 118.9, 118.7, 117.4, 87.4, 86.4, 79.2, 62.6, 62.3, 45.6, 14.0, 13.8.; HRMS (ESI+) Calcd. For $C_{29}H_{28}NO_5^+$ ($[M+H]^+$): 470.1962, found: 470.1960. The product was analyzed by HPLC to determine the enantiomeric excess: 92% ee (Chiralpak IE, *i*-propanol/hexane = 5/95 flow rate 1.0 mL/min, λ = 254 nm); t_r = 11.88 and 14.01 min.



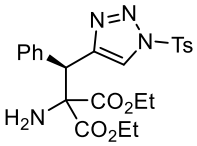
5

diethyl (*R*,*E*)-2-((2-hydroxybenzylidene)amino)-2-(1-phenylallyl)malonate (5): 71.2 mg, 90% yield, yellow oil; $[\alpha]^{32}_{D}$ = 14.3 (*c* 1.20, CH_2Cl_2); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 13.12 (s, 1H), 8.34 (s, 1H), 7.38 – 7.34 (m, 1H), 7.31 – 7.27 (m, 3H), 7.26 – 7.17 (m, 3H), 7.02 – 6.99 (dd, *J* = 8.4, 1.0 Hz, 1H), 6.92 – 6.88 (m, 1H), 6.35 – 6.26 (m, 1H), 5.24 (ddd, *J* = 10.2, 1.3, 1.2 Hz, 1H), 5.12 (ddd, *J* = 17.0, 1.4, 1.2 Hz, 1H), 4.54 – 4.52 (m, 1H), 4.34 – 4.21 (m, 2H), 4.11 – 3.99 (m, 2H), 1.28 (t, *J* = 7.1 Hz, 3H), 1.10 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 169.3, 167.6, 167.3, 161.1, 137.8, 136.4, 133.2, 132.5, 130.0, 128.1, 127.4, 118.94, 118.85, 118.8, 117.3, 79.1, 62.4, 62.1, 55.1, 14.0, 13.8; HRMS (ESI+) Calcd. For $C_{23}H_{26}NO_5^+$ ($[M+H]^+$): 396.1805, found: 396.1805. The product was analyzed by HPLC to determine the enantiomeric excess: 93% ee (Chiralpak IE, *i*-propanol/hexane = 5/95 flow rate 1.0 mL/min, λ = 254 nm); t_r = 8.83 and 10.33 min.



6

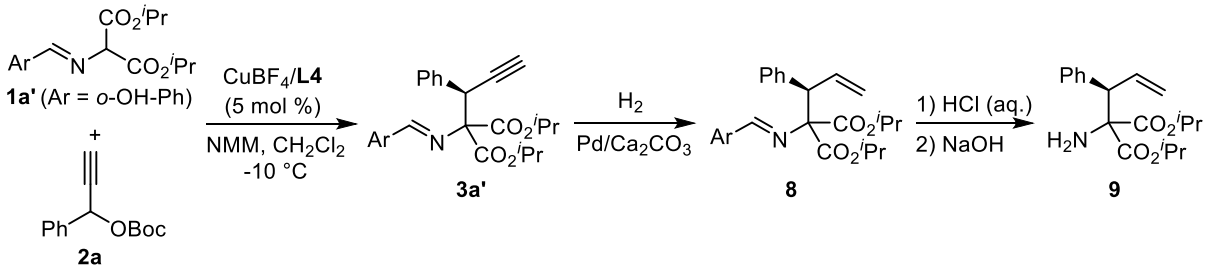
diethyl (*R*)-2-amino-2-(1-phenylprop-2-yn-1-yl)malonate (6): 69.4 mg, 80% yield, yellow oil; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.50 – 7.43 (m, 2H), 7.34 – 7.27 (m, 3H), 4.79 (d, *J* = 2.5 Hz, 1H), 4.40 – 4.26 (m, 2H), 4.15 – 4.02 (m, 2H), 2.33 (d, *J* = 2.5 Hz, 1H), 2.12 (brs, 2H), 1.34 (t, *J* = 7.1 Hz, 3H), 1.18 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 169.4, 168.6, 135.2, 129.5, 128.2, 128.0, 82.0, 72.7, 70.1, 62.5, 62.3, 43.5, 14.0, 13.8. HRMS (ESI+) Calcd. For $C_{16}H_{20}NO_4^+$ ([M+H]$^+$): 290.1388, found: 290.1385.



7

diethyl (*R*)-2-amino-2-(phenyl(1-tosyl-1H-1,2,3-triazol-4-yl)methyl)malonate (7): 69.1 mg, 71% yield, white solid, mp 88–90 °C; $[\alpha]^{32}_{D}$ = 77.00 (*c* 0.90, CH_2Cl_2); ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.30 (s, 1H), 8.00 – 7.90 (m, 2H), 7.37 – 7.34 (m, 4H), 7.27 – 7.24 (m, 2H), 5.35 (s, 1H), 4.12 – 4.01 (m, 4H), 2.44 (s, 3H), 1.17 (t, *J* = 7.1 Hz, 3H), 0.99 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 169.6, 169.5, 147.2, 147.1, 136.4, 133.1, 130.3, 129.7, 128.6, 128.4, 127.8, 122.6, 69.8, 62.5, 62.4, 47.6, 21.8, 13.9, 13.6. HRMS (ESI+) Calcd. For $C_{23}H_{27}N_4O_6S^+$ ([M+H]$^+$): 487.1646, found: 487.1646. The product was analyzed by HPLC to determine the enantiomeric excess: 93% ee (Chiralcel AS-H, *i*-propanol/hexane = 10/90 flow rate 1.0 mL/min, λ = 220 nm); t_r = 21.99 and 25.66 min.

IV. Determination of Absolute Configuration of 3a'

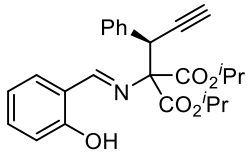


In a 10 mL Schlenk flask was placed $Cu(CH_3CN)_4BF_4$ (0.01 mmol) and **L4** (0.01 mmol) under Ar. Anhydrous DCM (1.0 mL) was added, and the mixture was magnetically stirred at room temperature for 30 min. Then the reaction flask was placed in a cool bath of -10 ºC, followed by addition of **1a'** (0.4 mmol), **2a** (0.4 mmol), 4-methylmorpholine (0.4 mmol) and Anhydrous DCM (1.0 mL) sequentially, and monitored by TLC analysis. After completion, the reaction was quenched with H_2O (3 mL). The aqueous layer was extracted three times with ethyl acetate (6 mL × 3). The combined organic layer was dried over Na_2SO_4. The volatile solvent was removed under reduced pressure. The residue was purified by flash chromatography on silica gel to afford pure **3a'**.

To a solution of **3a'** (0.30 mmol) in EtOH (3 mL) under nitrogen, then 10 mg Pd-$CaCO_3$ was added. The reaction mixture was stirred under H_2 atmosphere (1 atm) at 25 ºC for 3d.[2] After the reaction was completed (monitored by TLC), the crude reaction mixture was filtered over a short pad of celite and washed with EtOAc. The solvents were removed under reduced pressure. Then the residue was purified by silica gel column chromatography to afford the desired product **8**.

To a solution of **8** (0.20 mmol) in THF (1 mL), then 1 mL 2 M HCl was added. The reaction mixture was stirred at 25 ºC for 2h. After the reaction was complete (monitored by TLC), THF was removed under reduced pressure. The crude reaction mixture was extracted with EtOAc (3 × 3 mL) and the aqueous solution was added with 10% NaOH until pH = 10, then the crude reaction mixture was extracted with DCM (3 × 3 mL), the organics were combined and dried over Na_2SO_4 and concentrated under vacuum and purified by silica-gel flash chromatography to afford the desired product **9**; $[\alpha]^{32}_D$ = -50.6 (*c* 0.89, CH_2Cl_2).

Compared with the results in the literature[1], the absolute configuration of **9** is determined to be *R*.



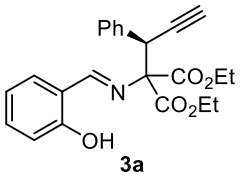
3a'

diisopropyl (*R*,*E*)-2-((2-hydroxybenzylidene)amino)-2-(1-phenylprop-2-yn-1-yl)malonate (3a'): 71% yield, yellow liquid; ^{1}H NMR (400 MHz, Chloroform-*d*) δ 13.12 (s, 1H), 8.45 (s, 1H), 7.40 – 7.32 (m, 3H), 7.32 – 7.22 (m, 4H), 7.03 – 7.01 (m, 1H), 6.93 – 6.89 (m, 1H), 5.24 – 5.15 (m, 1H), 4.95 – 4.86 (m, 1H), 4.84 (d, *J* = 2.5 Hz, 1H), 2.41 (d, *J* = 2.5 Hz, 1H), 1.30 (d, *J* = 6.0 Hz, 3H), 1.29 (d, *J* = 6.0 Hz, 3H), 1.21 (d, *J* = 6.3 Hz, 3H), 1.06 (d, *J* = 6.3 Hz, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 169.0, 166.4, 166.0, 161.1, 135.2, 133.2, 132.5, 129.8, 128.2, 128.1, 118.9, 118.7, 117.4, 82.2, 78.4, 74.1, 70.7, 70.5, 44.5, 21.6, 21.5, 21.4, 21.3. The product was analyzed by HPLC to determine the enantiomeric excess: 65% ee (Chiralpak AD-H, *i*-propanol/hexane = 5/95 flow rate 1.0 mL/min, λ = 262 nm); t_r = 16.01 and 19.99 min.

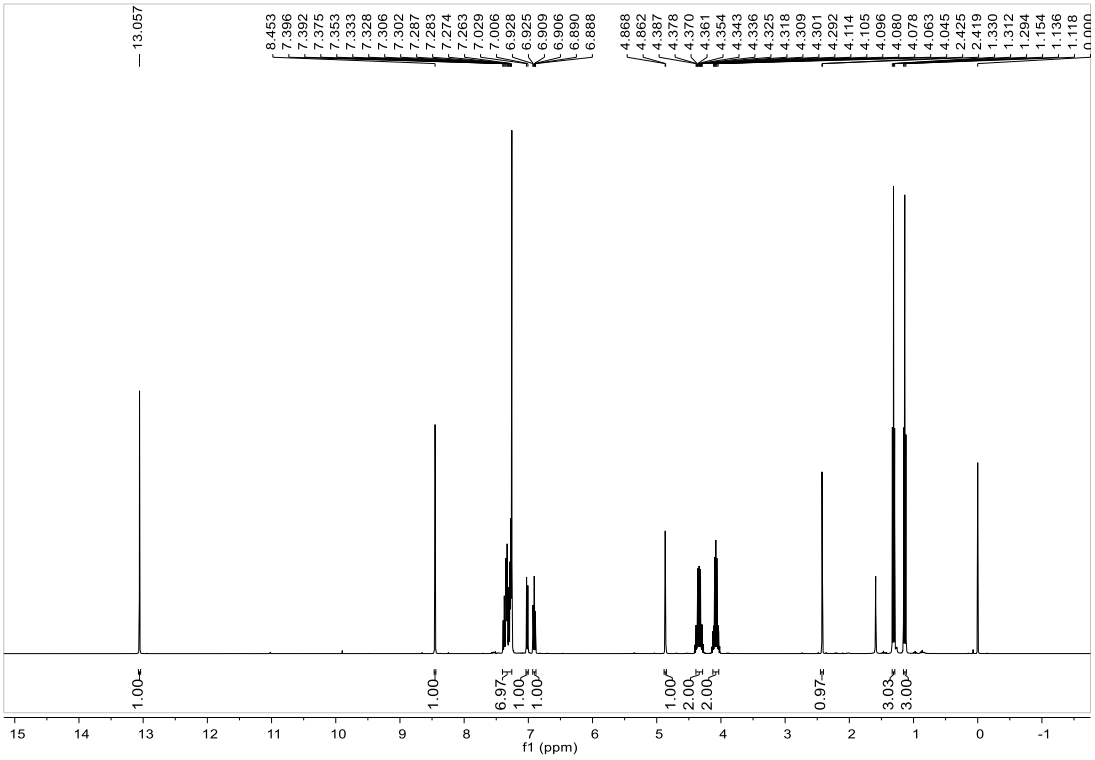
V. Reference

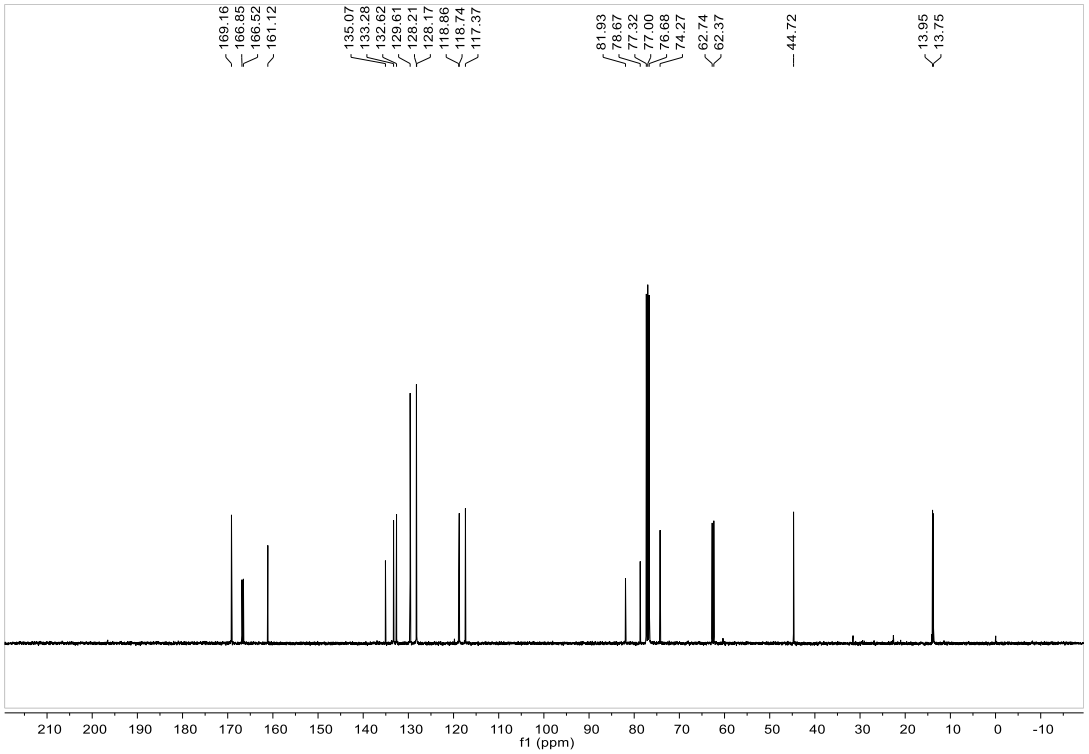
1. R.-Q. Wang, C. Shen, X. Cheng, Z.-F. Wang, H.-Y. Tao, X.-Q. Dong, C.-J. Wang, *Chin. J. Chem*. **2020**, *38*, 807.
2. W. Shao, H. Li, C. Liu, C.-J. Liu, S.-L. You, *Angew. Chem. Int. Ed*. **2015**, *54*, 7684.
3. Q. Zhu, B. Meng, C. Gu, Y. Xu, J. Chen, C. Lei, X. Wu, *Org. Lett*. **2019**, *21*, 9985.

VI. NMR and HPLC Spectra



3a





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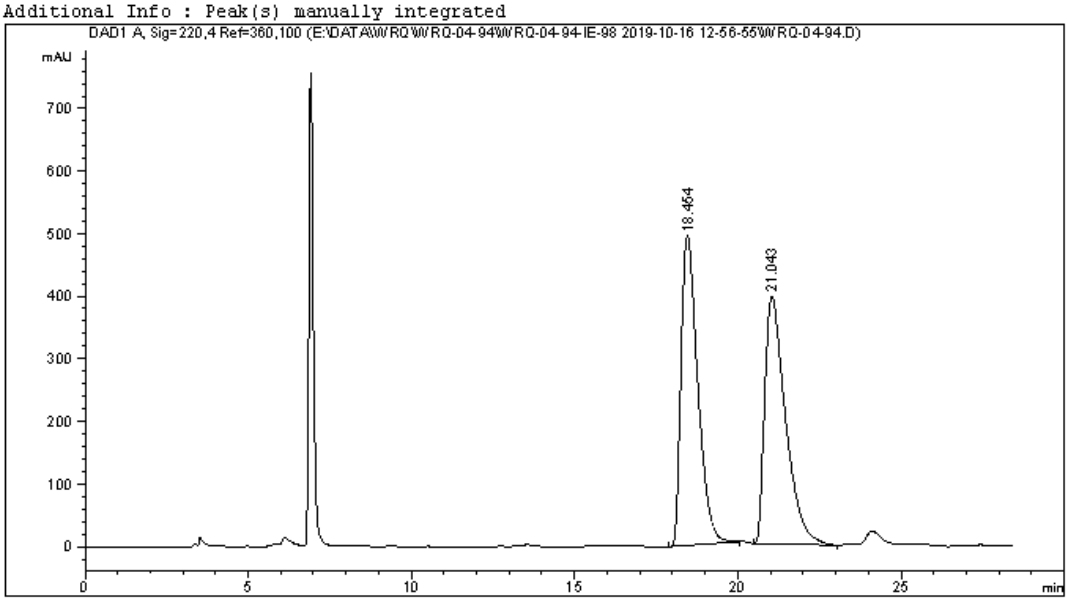


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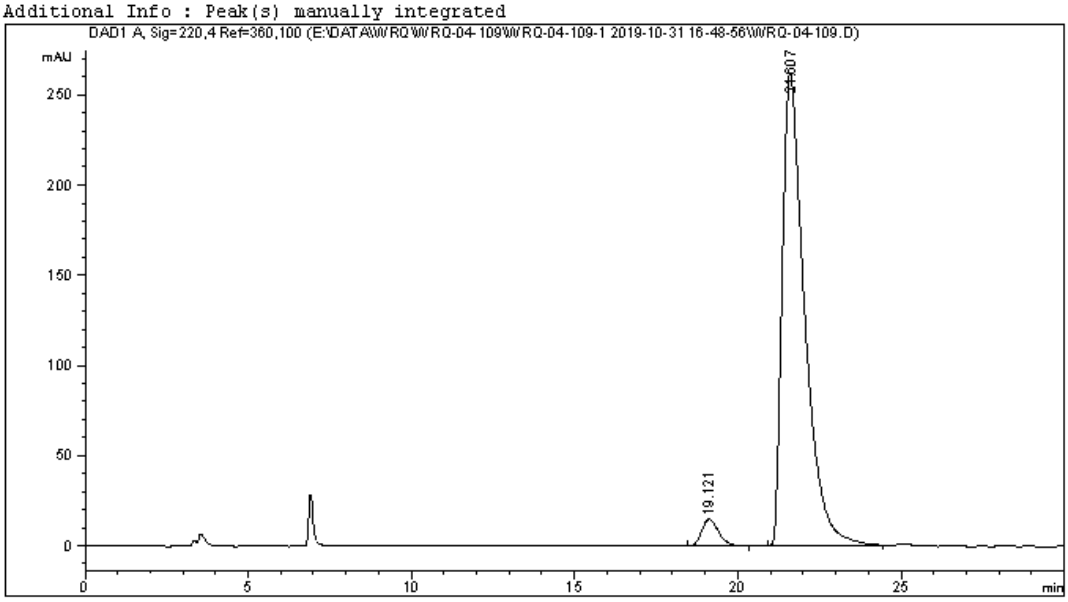
    Additional Info : Peak(s) manually integrated
```



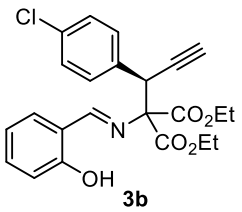

```
    ======================================================================
                           Area Percent Report
    ======================================================================

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

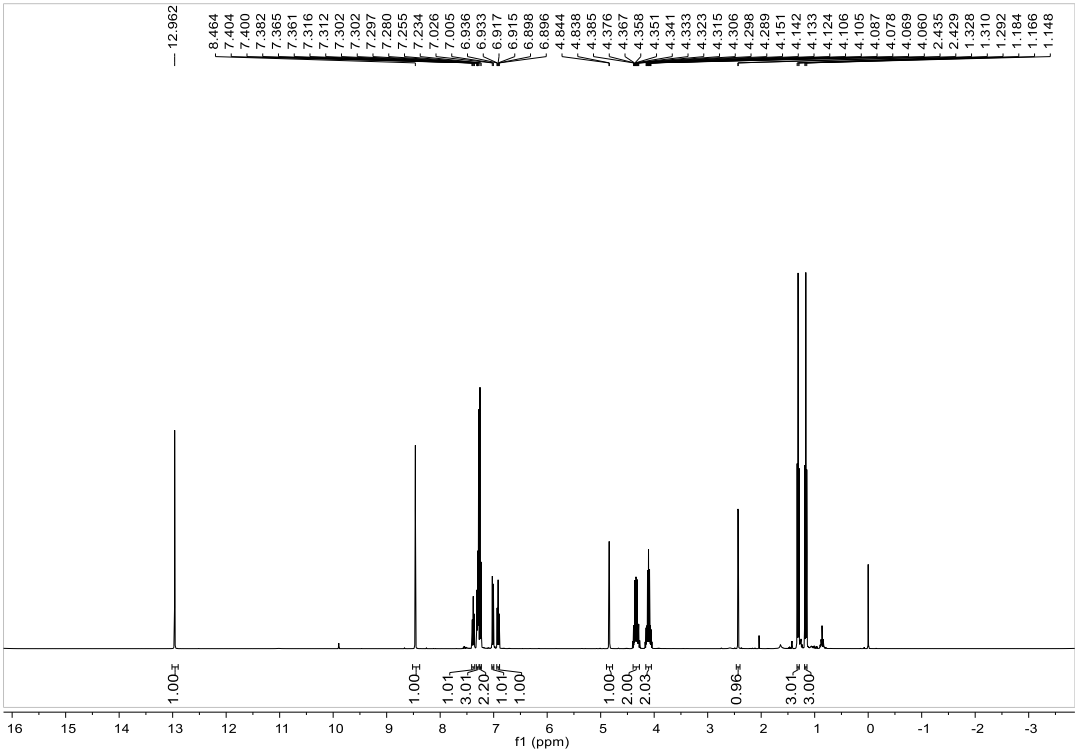

    Signal 1: DAD1 A, Sig=220,4 Ref=360,100

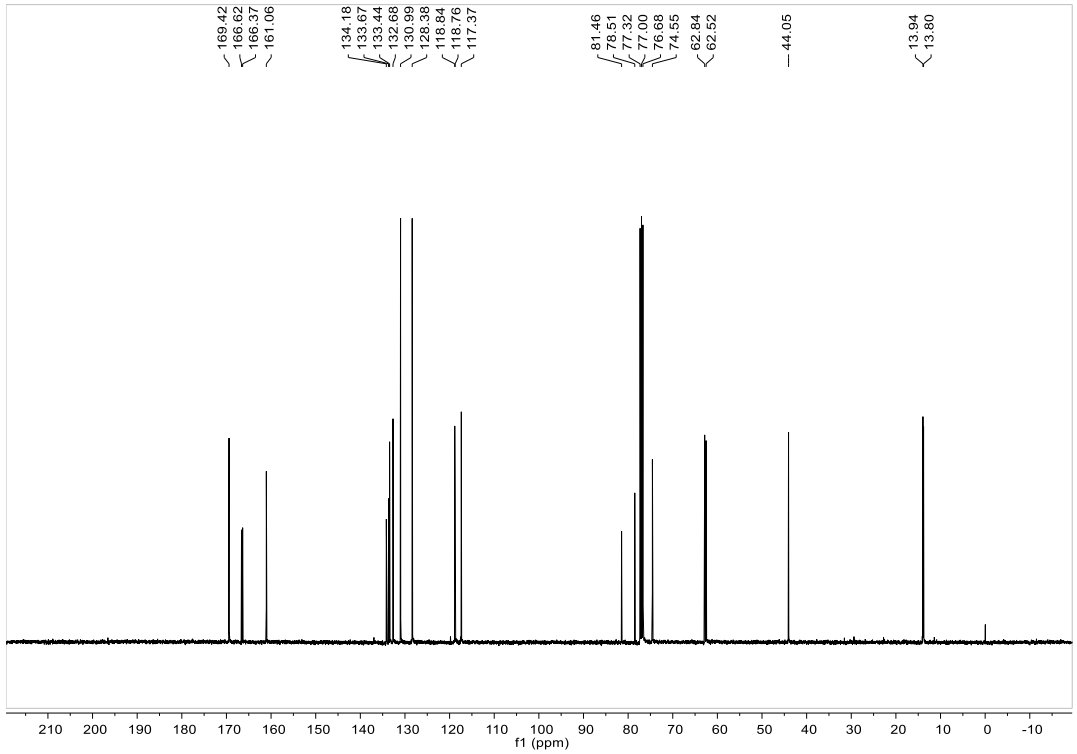
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  19.121 BB    0.4871  531.76917   14.59544   4.1155
       2  21.607 BB    0.6855 1.23893e4   261.62451  95.8845

    Totals :                  1.29210e4   276.21995
```


Cl
CO2Et
CO2Et
N
OH

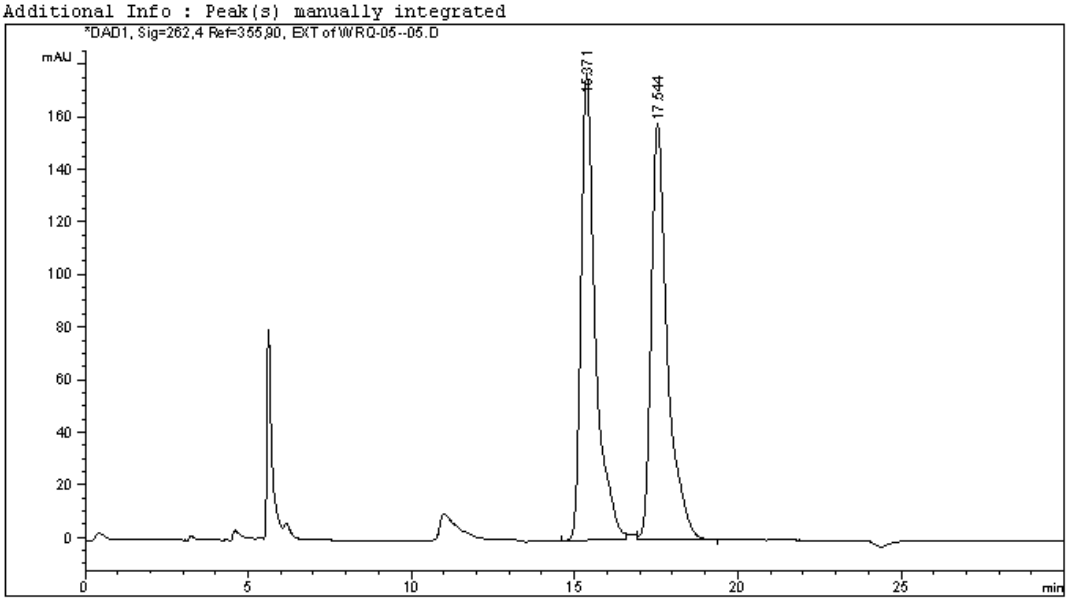
3b


12.962
8.464
7.404
7.400
7.382
7.365
7.361
7.316
7.312
7.302
7.302
7.297
7.280
7.255
7.234
7.026
7.005
6.936
6.933
6.917
6.915
6.898
6.896
4.844
4.838
4.385
4.376
4.367
4.358
4.351
4.341
4.333
4.323
4.315
4.306
4.298
4.289
4.151
4.142
4.133
4.124
4.106
4.105
4.087
4.078
4.069
4.060
2.435
2.429
1.328
1.310
1.292
1.184
1.166
1.148
1.00
1.00
1.01
3.01
2.20
1.01
1.00
1.00
2.00
2.03
0.96
3.01
3.00
f1 (ppm)


169.42
166.62
166.37
161.06
134.18
133.67
133.44
132.68
130.99
128.38
118.84
118.76
117.37
81.46
78.51
77.32
77.00
76.68
74.55
62.84
62.52
44.05
13.94
13.80
f1 (ppm)

Data File E:\DATA\WRQ\WRQ-05--05\WRQ-05-05-06-95-5 2019-12-18 01-01-11\WRQ-05--05.D
Sample Name: WRQ-05-05-Cl-rac

```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   1
    Acq. Instrument : 1260                          Location :   51
    Injection Date  : 12/18/2019 1:02:38 AM              Inj :   1
                                                  Inj Volume : 10.000 µl
    Acq. Method     : E:\DATA\WRQ\WRQ-05--05\WRQ-05-05-06-95-5 2019-12-18 01-01-11\WRQ-2-95-5-DAD
                      -1ML-60MIN.M
    Last changed    : 12/18/2019 1:01:11 AM by SYSTEM
    Analysis Method : E:\DATA\WRQ\WRQ-05--05\WRQ-05-05-06-95-5 2019-12-18 01-01-11\WRQ-2-95-5-DAD
                      -1ML-60MIN.M (Sequence Method)
    Last changed    : 7/21/2020 8:07:05 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

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

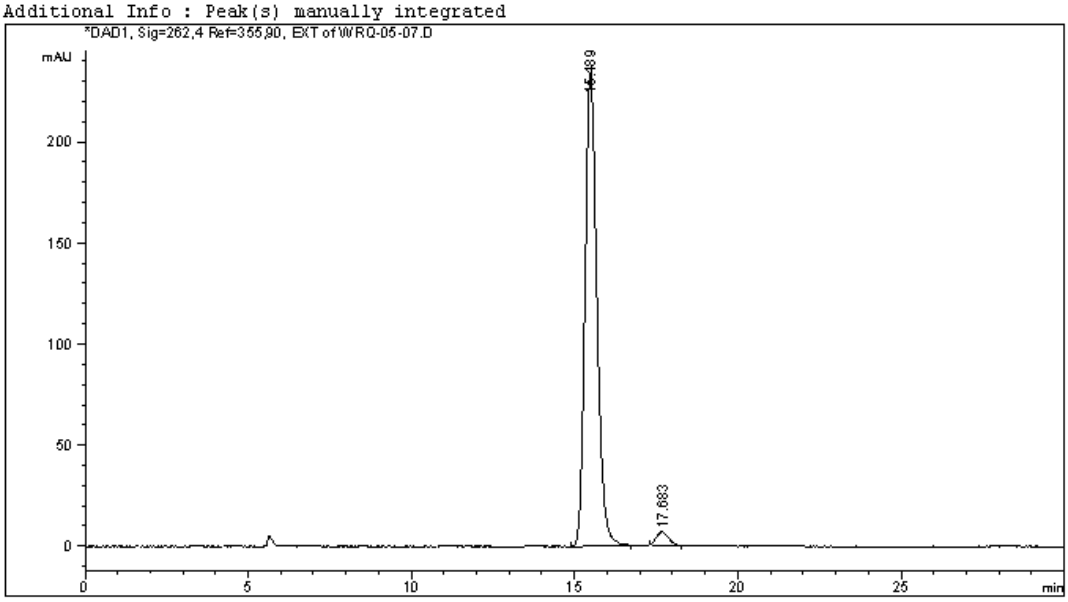

    Signal 1: DAD1, Sig=262,4 Ref=355,90, EXT
     Signal has been modified after loading from rawdata file!

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  15.371 MF R  0.5119 5449.81299  177.45215  50.0770
       2  17.544 FM R  0.5726 5433.06006  158.14549  49.9230

    Totals :                  1.08829e4   335.59764
```

Data File E:\DATA\WRQ\WRQ-05--07\WRQ-05-07-8-9-10 2019-12-19 23-03-29\WRQ-05-07.D
Sample Name: WRQ-05-07

```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   1
    Acq. Instrument : 1260                          Location :   53
    Injection Date  : 12/19/2019 11:04:59 PM             Inj :   1
                                                  Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\WRQ\WRQ-05--07\WRQ-05-07-8-9-10 2019-12-19 23-03-29\WRQ-2-95-5-DAD-
                      1ML-30MIN.M
    Last changed    : 12/19/2019 11:03:29 PM by SYSTEM
    Analysis Method : E:\DATA\WRQ\WRQ-05--07\WRQ-05-07-8-9-10 2019-12-19 23-03-29\WRQ-2-95-5-DAD-
                      1ML-30MIN.M (Sequence Method)
    Last changed    : 7/21/2020 8:10:39 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```



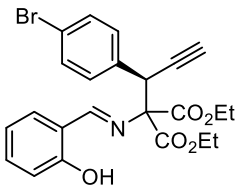

```
    ======================================================================
                           Area Percent Report
    ======================================================================

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

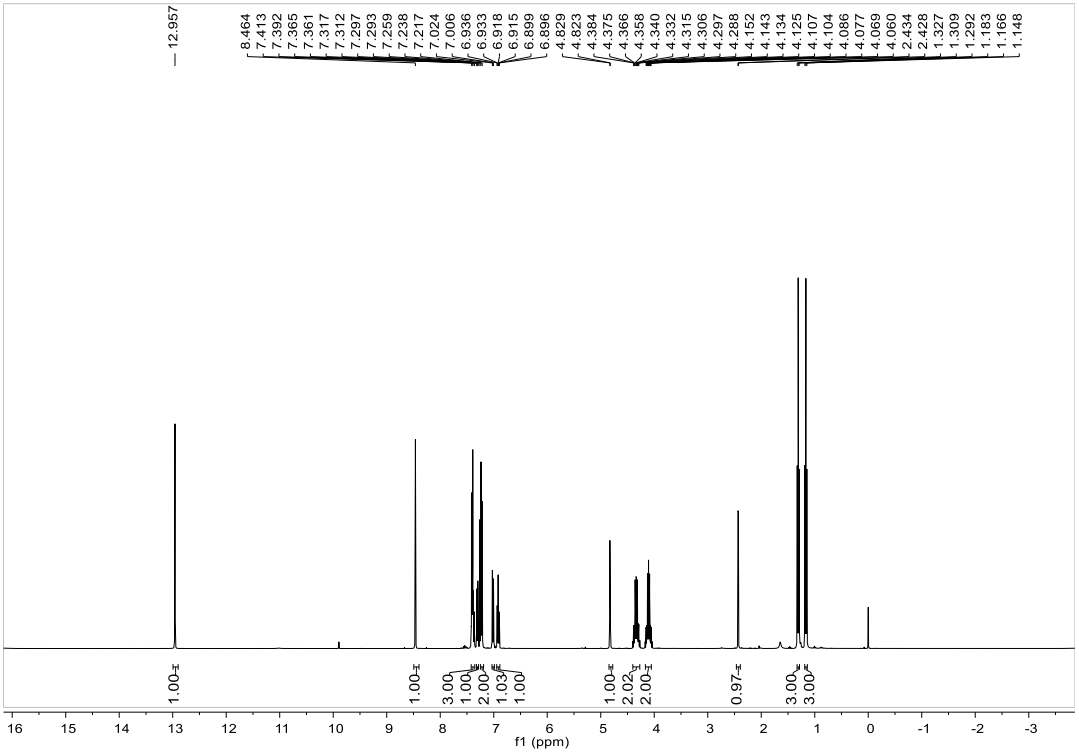

    Signal 1: DAD1, Sig=262,4 Ref=355,90, EXT
     Signal has been modified after loading from rawdata file!

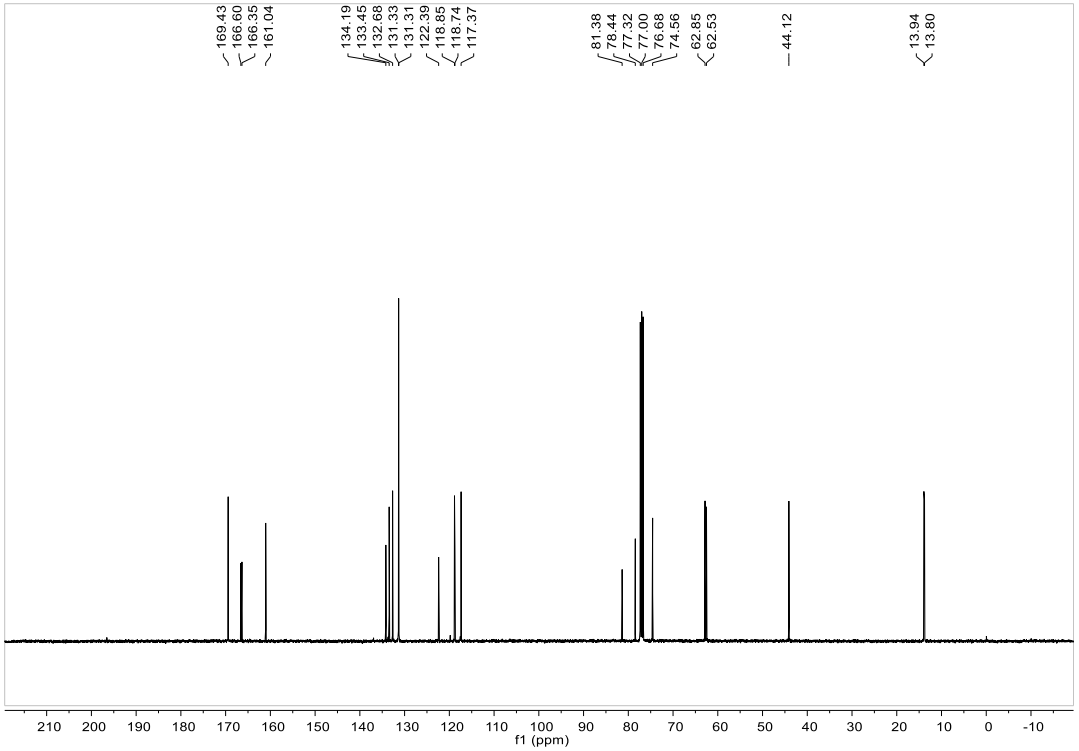
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  15.489 MM R  0.4238 5945.11084  233.81195  97.1726
       2  17.683 MM R  0.4397  172.98402    6.55733   2.8274

    Totals :                  6118.09486  240.36928
```


Br
CO2Et
CO2Et
N
OH

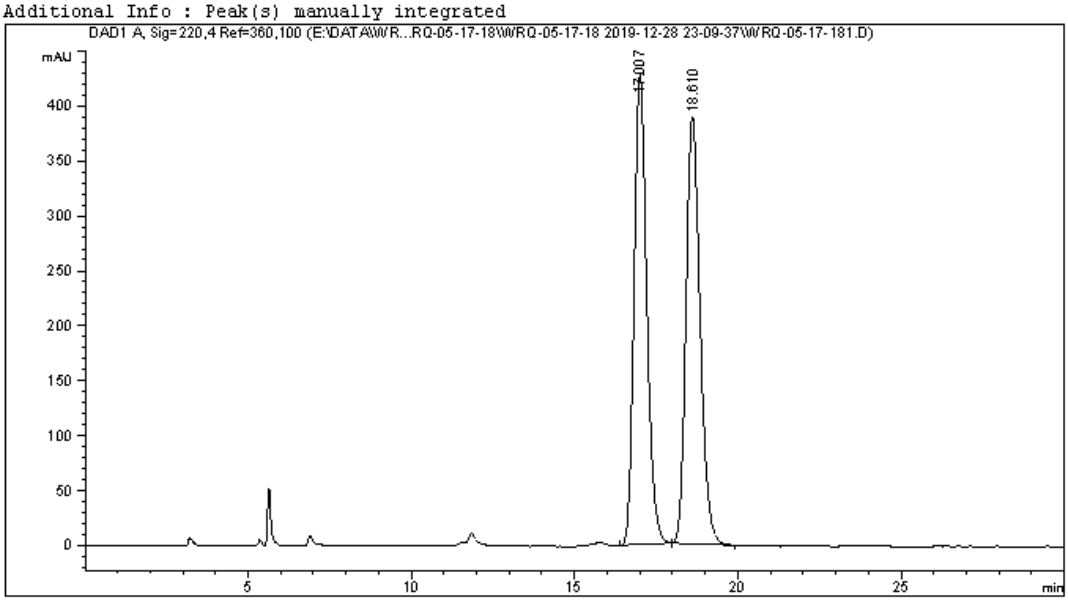
3c


12.957
8.464
7.413
7.392
7.365
7.361
7.317
7.312
7.297
7.293
7.259
7.238
7.217
7.024
7.006
6.936
6.933
6.918
6.915
6.899
6.896
4.829
4.823
4.384
4.375
4.366
4.358
4.340
4.332
4.315
4.306
4.297
4.288
4.152
4.143
4.134
4.125
4.107
4.104
4.086
4.077
4.069
4.060
2.434
2.428
1.327
1.309
1.292
1.183
1.166
1.148
1.00
1.00
3.00
1.00
2.00
1.03
1.00
1.00
2.02
2.00
0.97
3.00
3.00
f1 (ppm)


169.43
166.60
166.35
161.04
134.19
133.45
132.68
131.33
131.31
122.39
118.85
118.74
117.37
81.38
78.44
77.32
77.00
76.68
74.56
62.85
62.53
44.12
13.94
13.80
f1 (ppm)

Data File E:\DATA\WRQ\WRQ-05-17-18\WRQ-05-17-18 2019-12-28 23-09-37\WRQ-05-17-181.D
Sample Name: WRQ-05-18

```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   2
    Acq. Instrument : 1260                          Location :   52
    Injection Date  : 12/28/2019 11:42:31 PM             Inj :   1
                                                  Inj Volume : 7.000 µl
    Acq. Method     : E:\DATA\WRQ\WRQ-05-17-18\WRQ-05-17-18 2019-12-28 23-09-37\WRQ-2-95-5-DAD-
                      1ML-30MIN.M
    Last changed    : 12/28/2019 11:09:37 PM by SYSTEM
    Analysis Method : E:\DATA\WRQ\WRQ-05-17-18\WRQ-05-17-18 2019-12-28 23-09-37\WRQ-2-95-5-DAD-
                      1ML-30MIN.M (Sequence Method)
    Last changed    : 7/21/2020 8:56:14 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

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

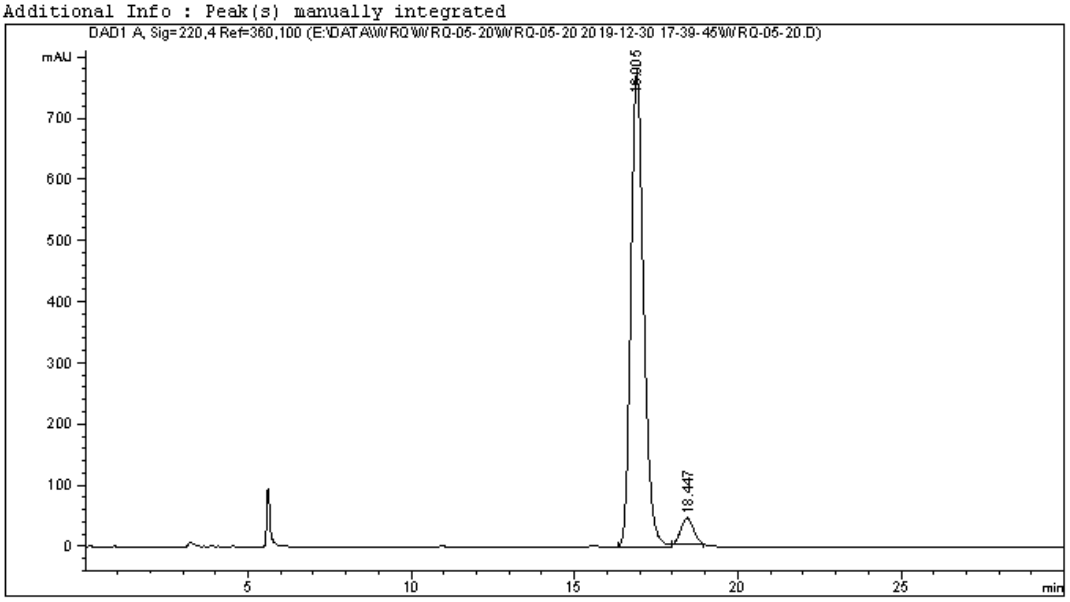

    Signal 1: DAD1 A, Sig=220,4 Ref=360,100

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  17.007 BB    0.4115 1.15967e4   428.87823  49.8109
       2  18.610 BB    0.4627 1.16848e4   388.71231  50.1891

    Totals :                  2.32815e4   817.59055
```

Data File E:\DATA\WRQ\WRQ-05-20\WRQ-05-20 2019-12-30 17-39-45\WRQ-05-20.D
Sample Name: WRQ-05-20

```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   1
    Acq. Instrument : 1260                          Location :   54
    Injection Date  : 12/30/2019 5:41:13 PM              Inj :   1
                                                  Inj Volume : 7.000 µl
    Acq. Method     : E:\DATA\WRQ\WRQ-05-20\WRQ-05-20 2019-12-30 17-39-45\WRQ-2-95-5-DAD-1ML-
                      30MIN.M
    Last changed    : 12/30/2019 5:39:45 PM by SYSTEM
    Analysis Method : E:\DATA\WRQ\WRQ-05-20\WRQ-05-20 2019-12-30 17-39-45\WRQ-2-95-5-DAD-1ML-
                      30MIN.M (Sequence Method)
    Last changed    : 7/21/2020 8:52:29 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```



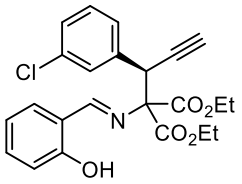

```
    ======================================================================
                           Area Percent Report
    ======================================================================

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

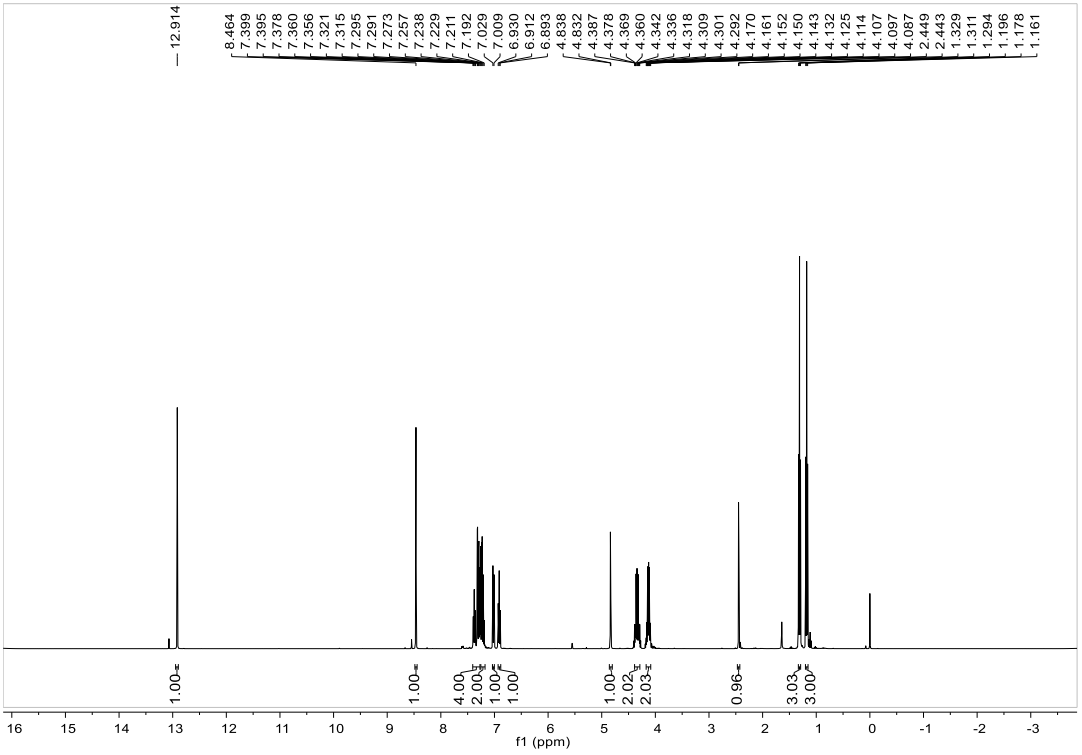

    Signal 1: DAD1 A, Sig=220,4 Ref=360,100

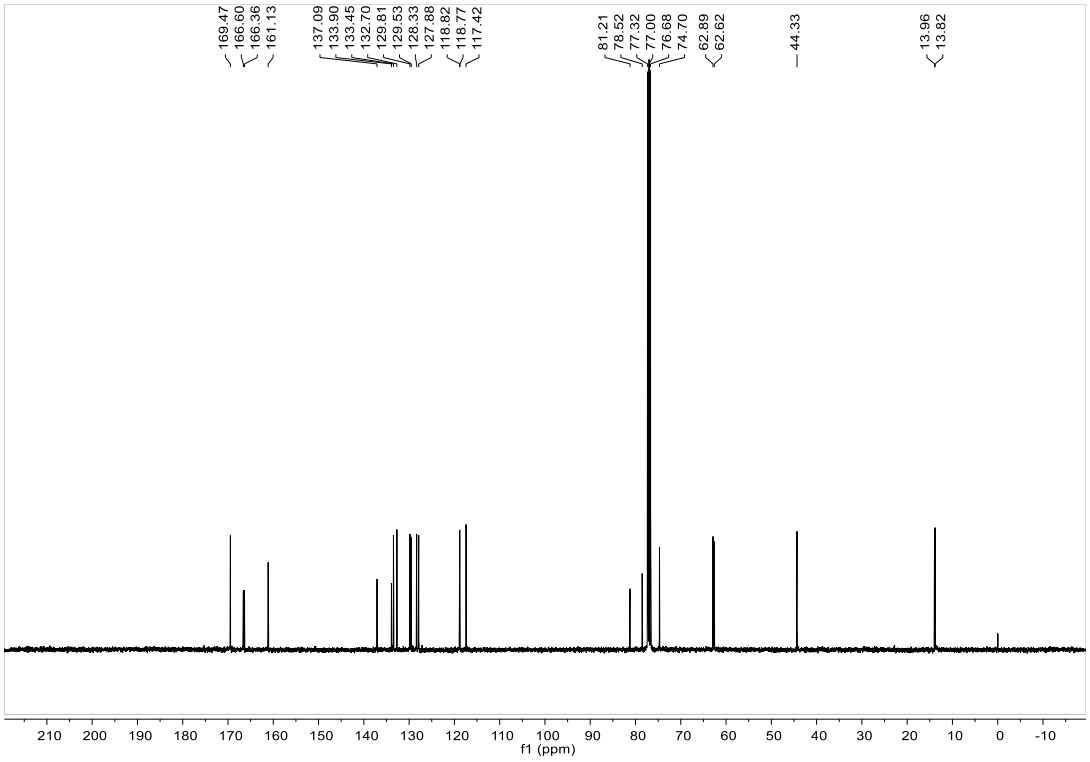
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  16.905 MM R  0.4571 2.12663e4   775.46625  94.8088
       2  18.447 MM R  0.4508 1164.42810   43.04722   5.1912

    Totals :                  2.24307e4   818.51347
```


Cl
N
CO2Et
CO2Et
OH

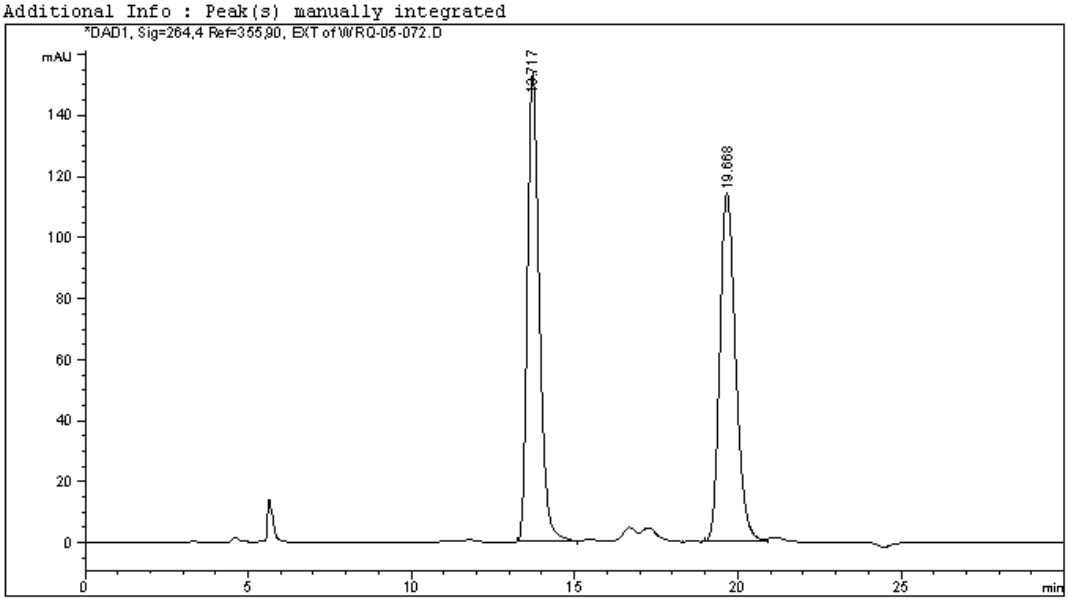
3d


12.914
8.464
7.399
7.395
7.378
7.360
7.356
7.321
7.315
7.295
7.291
7.273
7.257
7.238
7.229
7.211
7.192
7.029
7.009
6.930
6.912
6.893
4.838
4.832
4.387
4.378
4.369
4.360
4.342
4.336
4.318
4.309
4.301
4.292
4.170
4.161
4.152
4.150
4.143
4.132
4.125
4.114
4.107
4.097
4.087
2.449
2.443
1.329
1.311
1.294
1.196
1.178
1.161
1.00
1.00
4.00
2.00
1.00
1.00
1.00
2.02
2.03
0.96
3.03
3.00
f1 (ppm)


169.47
166.60
166.36
161.13
137.09
133.90
133.45
132.70
129.81
129.53
128.33
127.88
118.82
118.77
117.42
81.21
78.52
77.32
77.00
76.68
74.70
62.89
62.62
44.33
13.96
13.82
f1 (ppm)

Data File E:\DATA\WRQ\WRQ-05--07\WRQ-05-07-8-9-10 2019-12-19 23-03-29\WRQ-05-072.D
Sample Name: WRQ-05-09

```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   3
    Acq. Instrument : 1260                          Location :   55
    Injection Date  : 12/20/2019 12:07:55 AM             Inj :   1
                                                  Inj Volume : 10.000 µl
    Acq. Method     : E:\DATA\WRQ\WRQ-05--07\WRQ-05-07-8-9-10 2019-12-19 23-03-29\WRQ-2-95-5-DAD-
                      1ML-60MIN.M
    Last changed    : 12/19/2019 11:03:29 PM by SYSTEM
    Analysis Method : E:\DATA\WRQ\WRQ-05--07\WRQ-05-07-8-9-10 2019-12-19 23-03-29\WRQ-2-95-5-DAD-
                      1ML-60MIN.M (Sequence Method)
    Last changed    : 7/21/2020 8:29:58 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

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

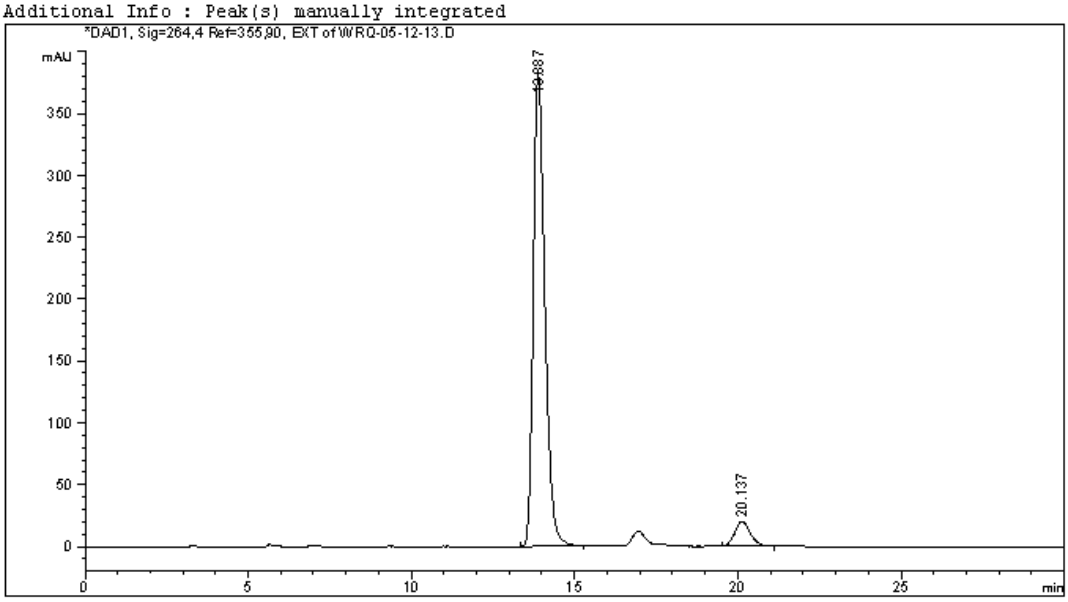

    Signal 1: DAD1, Sig=264,4 Ref=355,90, EXT
     Signal has been modified after loading from rawdata file!

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  13.717 MM R  0.4244 3907.79834  153.47763  50.2725
       2  19.668 MM R  0.5642 3865.44116  114.17921  49.7275

    Totals :                  7773.23950  267.65684
```

Data File E:\DATA\WRQ\WRQ-05-12-13\WRQ-05-12-13 2019-12-25 15-43-54\WRQ-05-12-13.D
Sample Name: WRQ-05-12-C1

```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   1
    Acq. Instrument : 1260                          Location :   51
    Injection Date  : 12/25/2019 3:45:22 PM             Inj :   1
                                                  Inj Volume : 7.000 µl
    Acq. Method     : E:\DATA\WRQ\WRQ-05-12-13\WRQ-05-12-13 2019-12-25 15-43-54\WRQ-2-95-5-DAD-
                      1ML-30MIN.M
    Last changed    : 12/25/2019 3:43:54 PM by SYSTEM
    Analysis Method : E:\DATA\WRQ\WRQ-05-12-13\WRQ-05-12-13 2019-12-25 15-43-54\WRQ-2-95-5-DAD-
                      1ML-30MIN.M (Sequence Method)
    Last changed    : 7/21/2020 8:33:26 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```



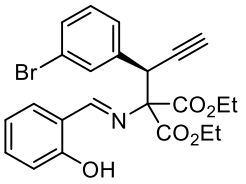

```
    ======================================================================
                           Area Percent Report
    ======================================================================

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

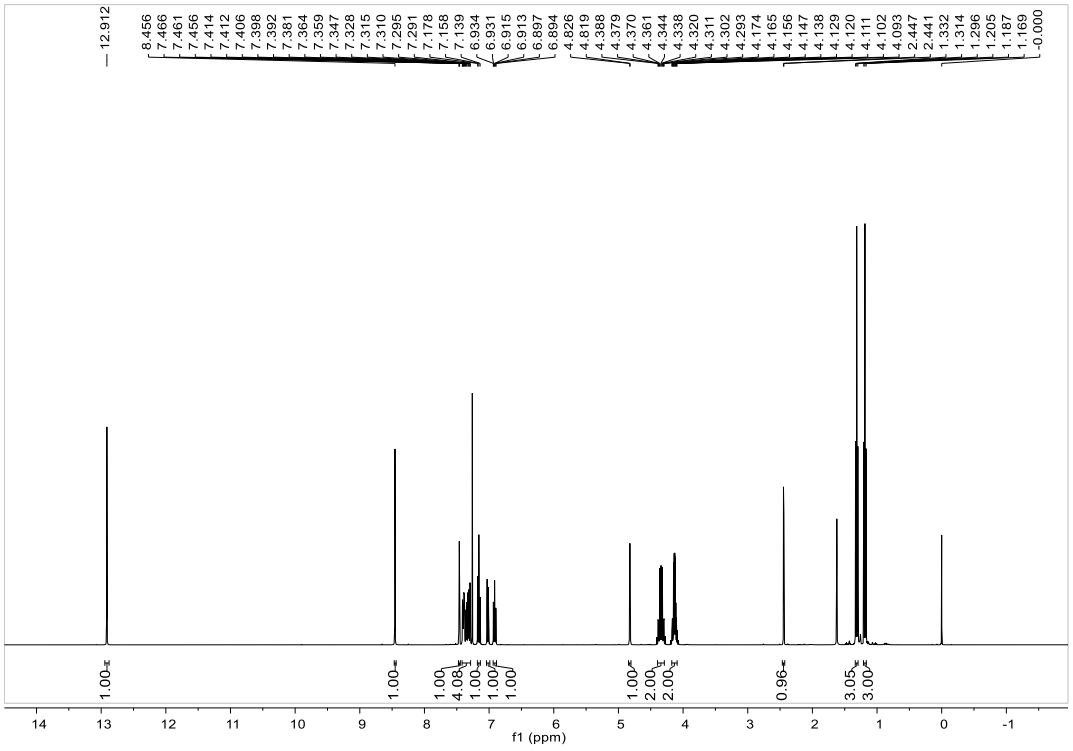

    Signal 1: DAD1, Sig=264,4 Ref=355,90, EXT
     Signal has been modified after loading from rawdata file!

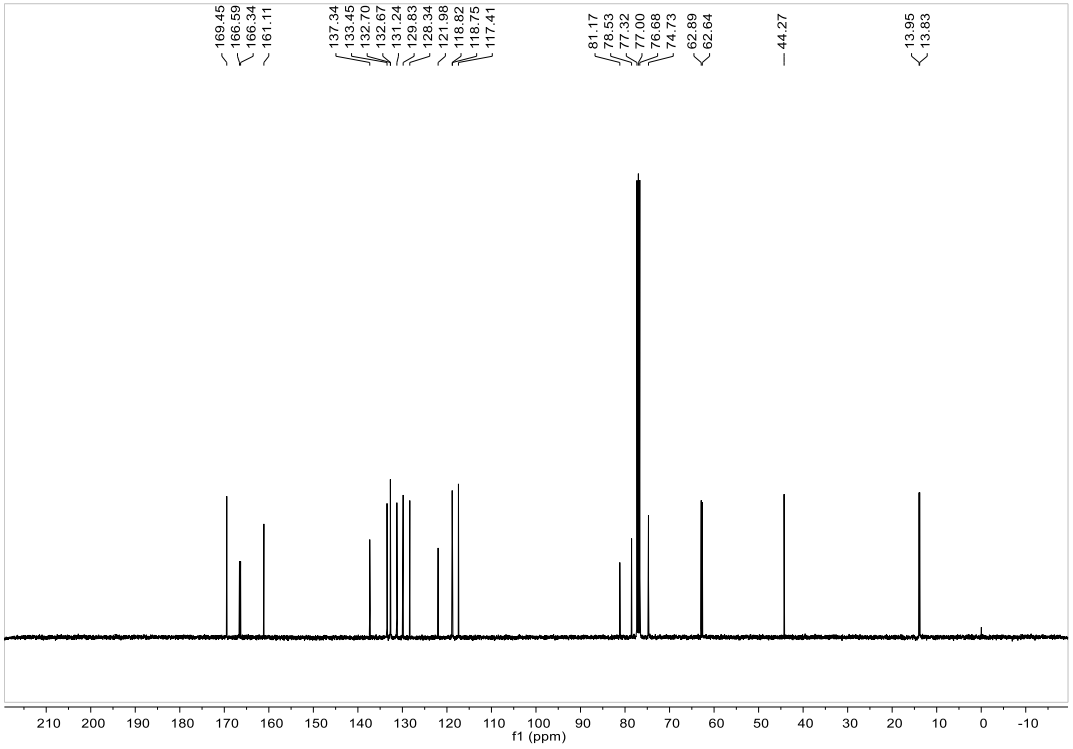
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  13.887 BB    0.3774 9416.22852  382.17908  93.5279
       2  20.137 BB    0.4078  651.60229   19.90847   6.4721

    Totals :                  1.00678e4   402.08755
```


Br
N
CO2Et
CO2Et
OH

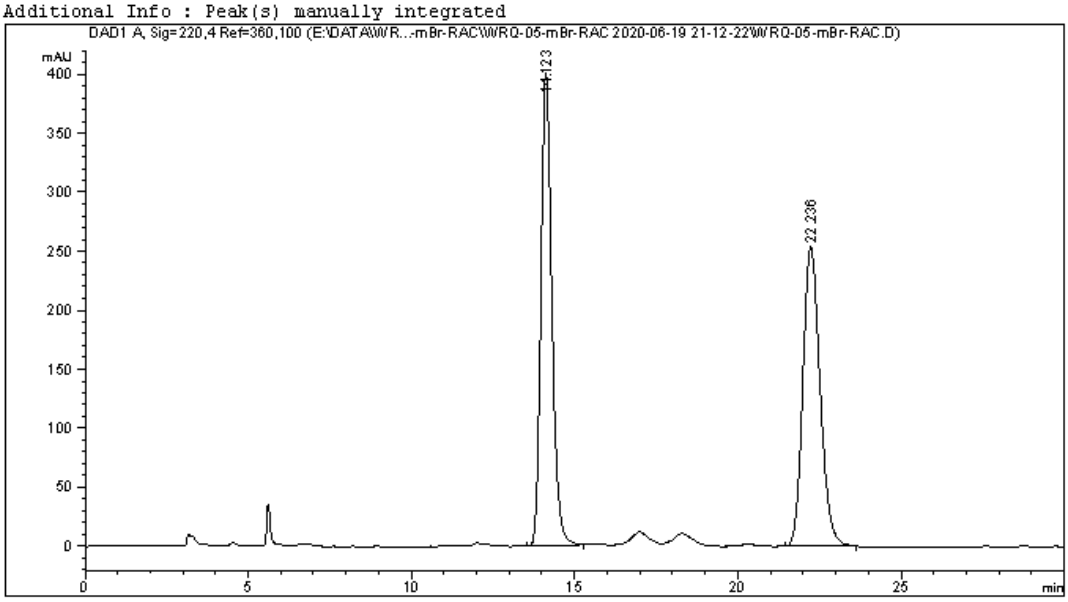
3e


12.912
8.456
7.466
7.461
7.456
7.414
7.412
7.406
7.398
7.392
7.381
7.364
7.359
7.347
7.328
7.315
7.310
7.295
7.291
7.178
7.158
7.139
6.934
6.931
6.915
6.913
6.897
6.894
4.826
4.819
4.388
4.379
4.370
4.361
4.344
4.338
4.320
4.311
4.302
4.293
4.174
4.165
4.156
4.147
4.138
4.129
4.120
4.111
4.102
4.093
2.447
2.441
1.332
1.314
1.296
1.205
1.187
1.169
-0.000
1.00
1.00
1.00
4.08
1.00
1.00
1.00
1.00
2.00
2.00
0.96
3.05
3.00
f1 (ppm)


169.45
166.59
166.34
161.11
137.34
133.45
132.70
132.67
131.24
129.83
128.34
121.98
118.82
118.75
117.41
81.17
78.53
77.32
77.00
76.68
74.73
62.89
62.64
44.27
13.95
13.83
f1 (ppm)

Data File E:\DATA\WRQ\WRQ-05-mBr-RAC\WRQ-05-mBr-RAC 2020-06-19 21-12-22\WRQ-05-mBr-RAC.D
Sample Name: WRQ-05-mBr-RAC

```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   1
    Acq. Instrument : 1260                           Location :   65
    Injection Date  : 6/19/2020 9:13:57 PM               Inj :   1
                                                  Inj Volume : 10.000 µl
    Acq. Method     : E:\DATA\WRQ\WRQ-05-mBr-RAC\WRQ-05-mBr-RAC 2020-06-19 21-12-22\WRQ-2-95-5-
                      DAD-1ML-40MIN.M
    Last changed    : 6/19/2020 9:12:22 PM by SYSTEM
    Analysis Method : E:\DATA\WRQ\WRQ-05-mBr-RAC\WRQ-05-mBr-RAC 2020-06-19 21-12-22\WRQ-2-95-5-
                      DAD-1ML-40MIN.M (Sequence Method)
    Last changed    : 7/20/2020 8:30:01 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

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

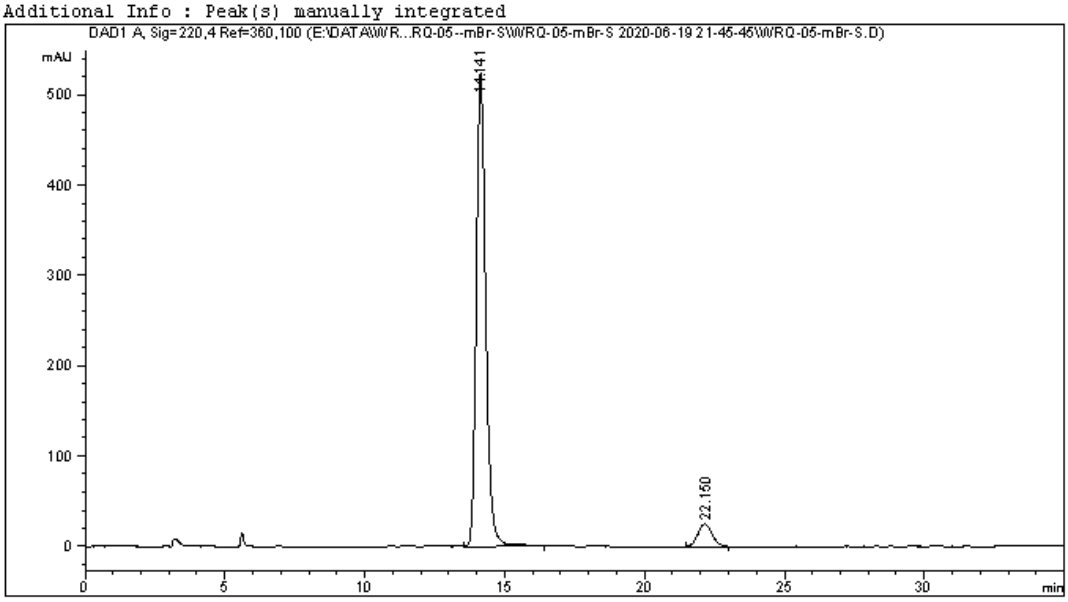

    Signal 1: DAD1 A, Sig=220,4 Ref=360,100

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  14.123 BB    0.3503 9274.55566  400.37326  49.9855
       2  22.236 BB    0.5483 9279.94336  253.95900  50.0145

    Totals :                  1.85545e4   654.33226
```

Data File E:\DATA\WRQ\WRQ-05--mBr-S\WRQ-05-mBr-S 2020-06-19 21-45-45\WRQ-05-mBr-S.D
Sample Name: WRQ-05-mBr-S

```
    ======================================================================
    Acq. Operator   : SYSTEM                          Seq. Line :   1
    Acq. Instrument : 1260                             Location :   66
    Injection Date  : 6/19/2020 9:47:22 PM                  Inj :   1
                                                    Inj Volume : 10.000 µl
    Acq. Method     : E:\DATA\WRQ\WRQ-05--mBr-S\WRQ-05-mBr-S 2020-06-19 21-45-45\WRQ-2-95-5-DAD-
                      1ML-40MIN.M
    Last changed    : 6/19/2020 9:45:45 PM by SYSTEM
    Analysis Method : E:\DATA\WRQ\WRQ-05--mBr-S\WRQ-05-mBr-S 2020-06-19 21-45-45\WRQ-2-95-5-DAD-
                      1ML-40MIN.M (Sequence Method)
    Last changed    : 7/20/2020 8:28:13 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```



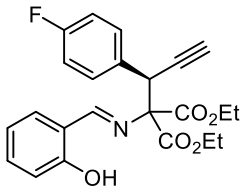

```
    ======================================================================
                           Area Percent Report
    ======================================================================

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

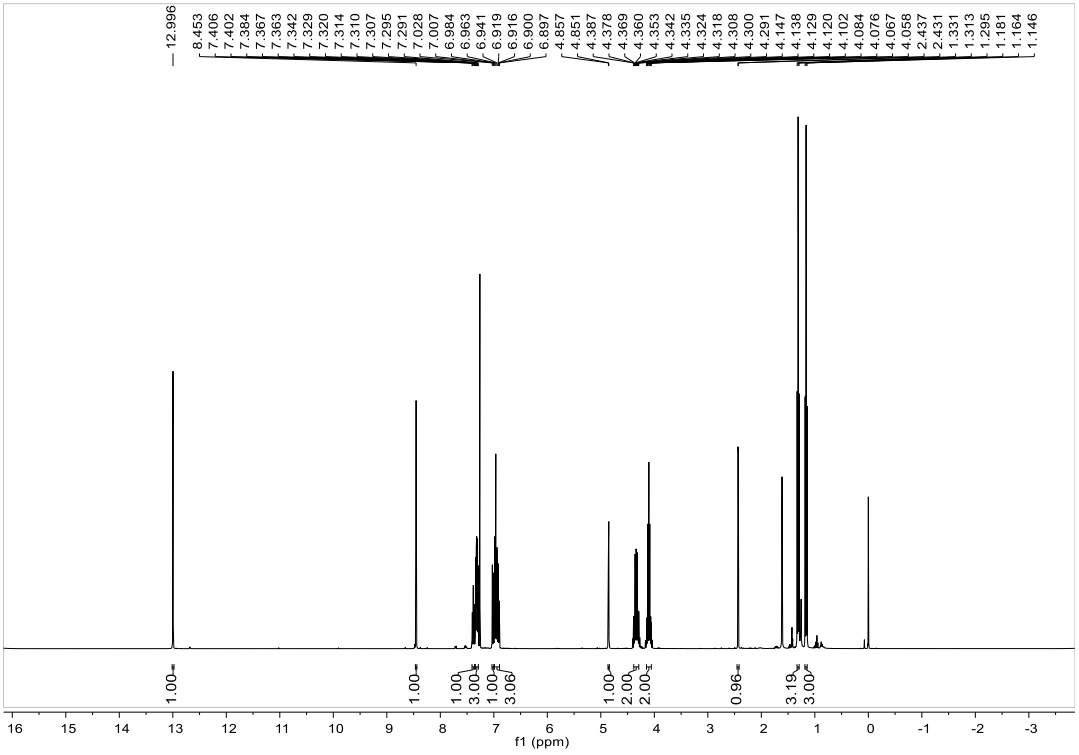

    Signal 1: DAD1 A, Sig=220,4 Ref=360,100

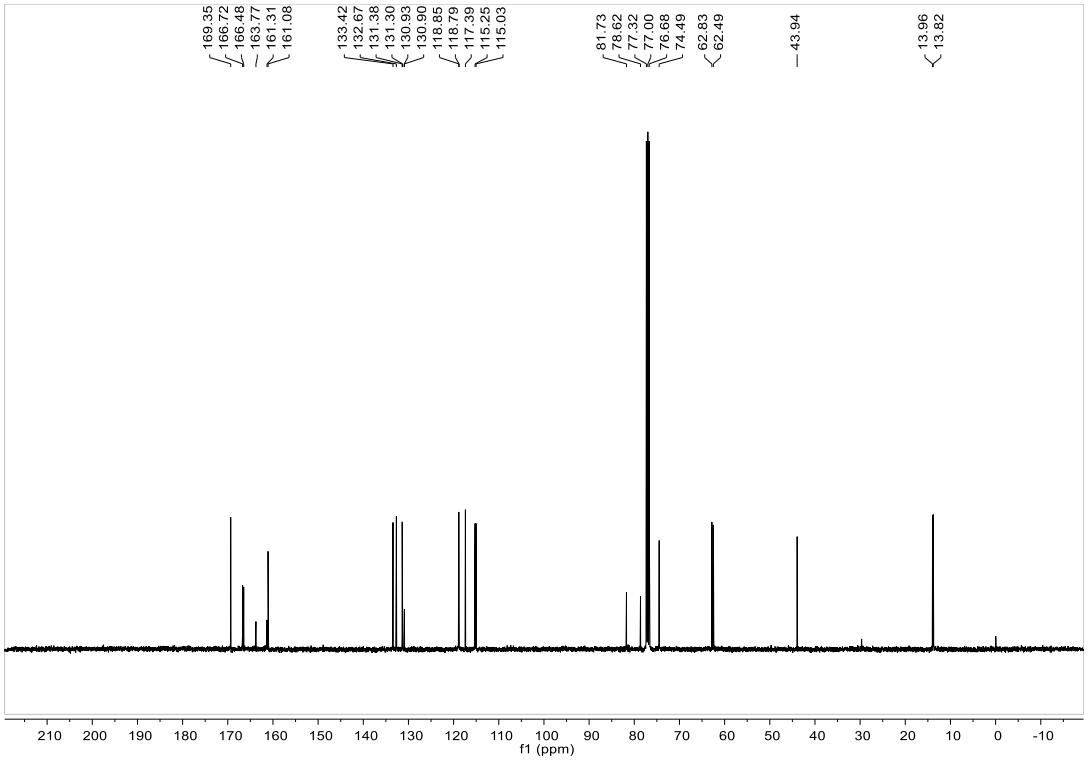
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  14.141 MM R  0.3950 1.24136e4   523.78461  93.3130
       2  22.150 MM R  0.6012  889.57928   24.66289   6.6870

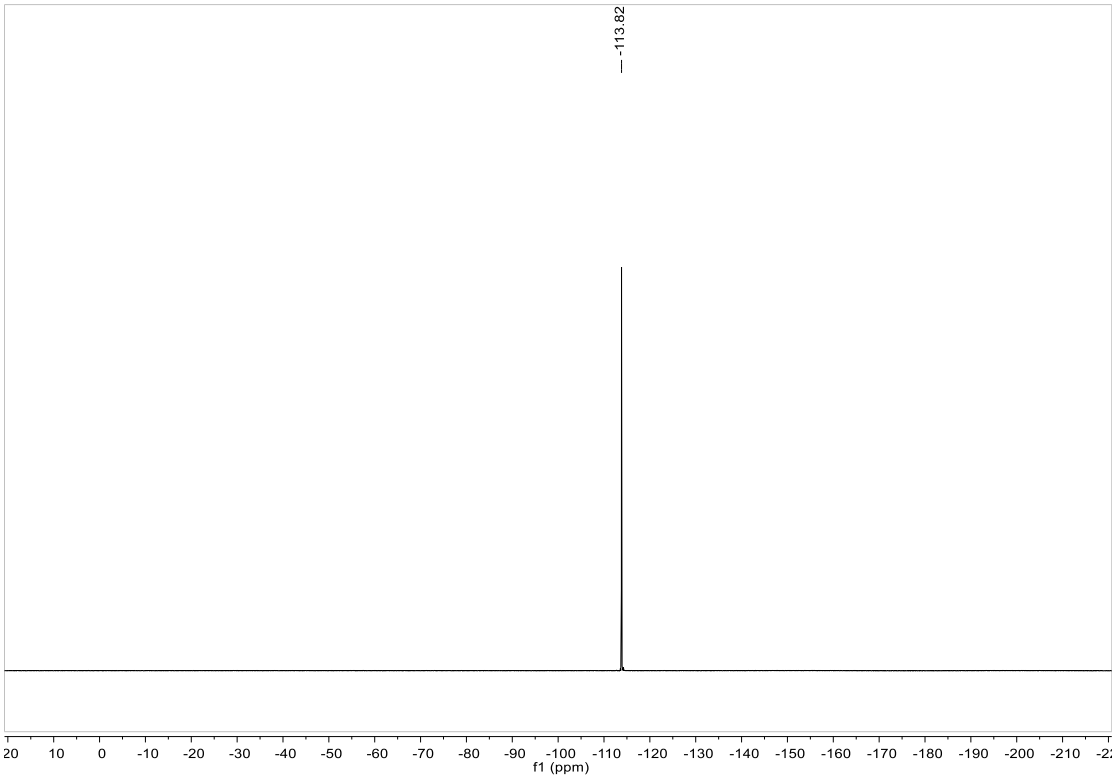
    Totals :                  1.33032e4   548.44749
```


F
CO2Et
CO2Et
N
OH

3f

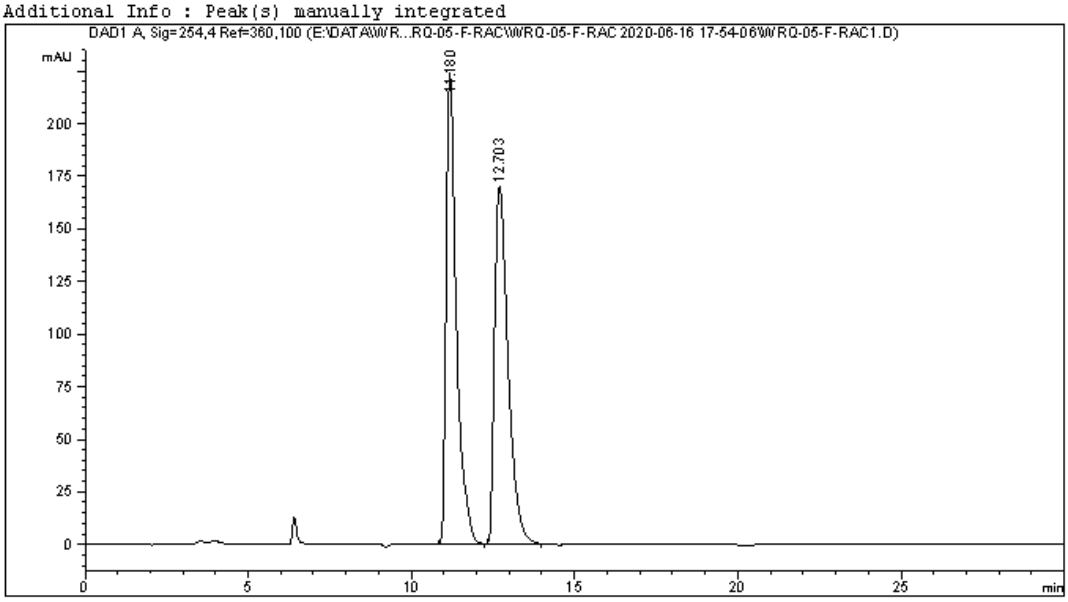

12.996
8.453
7.406
7.402
7.384
7.367
7.363
7.342
7.329
7.320
7.314
7.310
7.307
7.295
7.291
7.028
7.007
6.984
6.963
6.941
6.919
6.916
6.900
6.897
4.857
4.851
4.387
4.378
4.369
4.360
4.353
4.342
4.335
4.324
4.318
4.308
4.300
4.291
4.147
4.138
4.129
4.120
4.102
4.084
4.076
4.067
4.058
2.437
2.431
1.331
1.313
1.295
1.181
1.164
1.146
1.00
1.00
1.00
3.00
1.00
3.06
1.00
2.00
2.00
0.96
3.19
3.00
f1 (ppm)


169.35
166.72
166.48
163.77
161.31
161.08
133.42
132.67
131.38
131.30
130.93
130.90
118.85
118.79
117.39
115.25
115.03
81.73
78.62
77.32
77.00
76.68
74.49
62.83
62.49
43.94
13.96
13.82
f1 (ppm)


-113.82
20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

Data File E:\DATA\WRQ\WRQ-05-F-RAC\WRQ-05-F-RAC 2020-06-16 17-54-06\WRQ-05-F-RAC1.D
Sample Name: WRQ-05-F-RAC

```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   2
    Acq. Instrument : 1260                           Location :   61
    Injection Date  : 6/16/2020 6:27:01 PM               Inj :   1
                                                  Inj Volume : 10.000 µl
    Acq. Method     : E:\DATA\WRQ\WRQ-05-F-RAC\WRQ-05-F-RAC 2020-06-16 17-54-06\WRQ-4-IE-95-5-DAD
                      -1ML-30MIN.M
    Last changed    : 6/16/2020 5:54:06 PM by SYSTEM
    Analysis Method : E:\DATA\WRQ\WRQ-05-F-RAC\WRQ-05-F-RAC 2020-06-16 17-54-06\WRQ-4-IE-95-5-DAD
                      -1ML-30MIN.M (Sequence Method)
    Last changed    : 7/20/2020 8:40:11 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

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

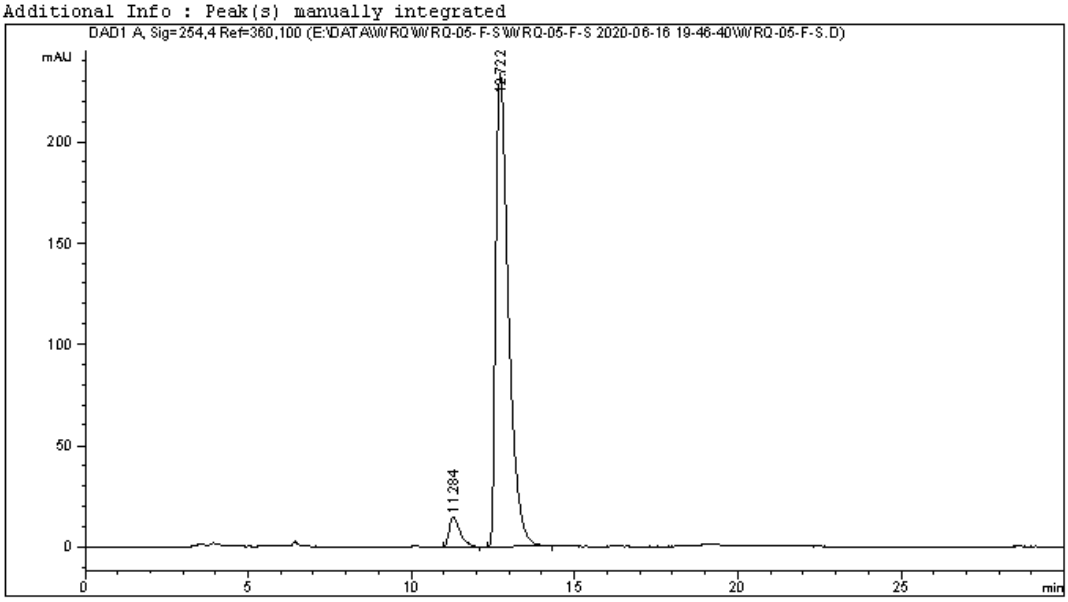

    Signal 1: DAD1 A, Sig=254,4 Ref=360,100

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  11.180 BB    0.3244 4918.21631  223.81128  50.2807
       2  12.703 BB    0.4325 4863.31055  169.64330  49.7193

    Totals :                  9781.52686  393.45457
```

Data File E:\DATA\WRQ\WRQ-05-F-S\WRQ-05-F-S 2020-06-16 19-46-40\WRQ-05-F-S.D
Sample Name: WRQ-05-F-S

```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   1
    Acq. Instrument : 1260                           Location :   62
    Injection Date  : 6/16/2020 7:48:15 PM                Inj :   1
                                                  Inj Volume : 10.000 µl
    Acq. Method     : E:\DATA\WRQ\WRQ-05-F-S\WRQ-05-F-S 2020-06-16 19-46-40\WRQ-4-IE-95-5-DAD-1ML
                      -30MIN.M
    Last changed    : 6/16/2020 7:46:40 PM by SYSTEM
    Analysis Method : E:\DATA\WRQ\WRQ-05-F-S\WRQ-05-F-S 2020-06-16 19-46-40\WRQ-4-IE-95-5-DAD-1ML
                      -30MIN.M (Sequence Method)
    Last changed    : 7/20/2020 8:39:30 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

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

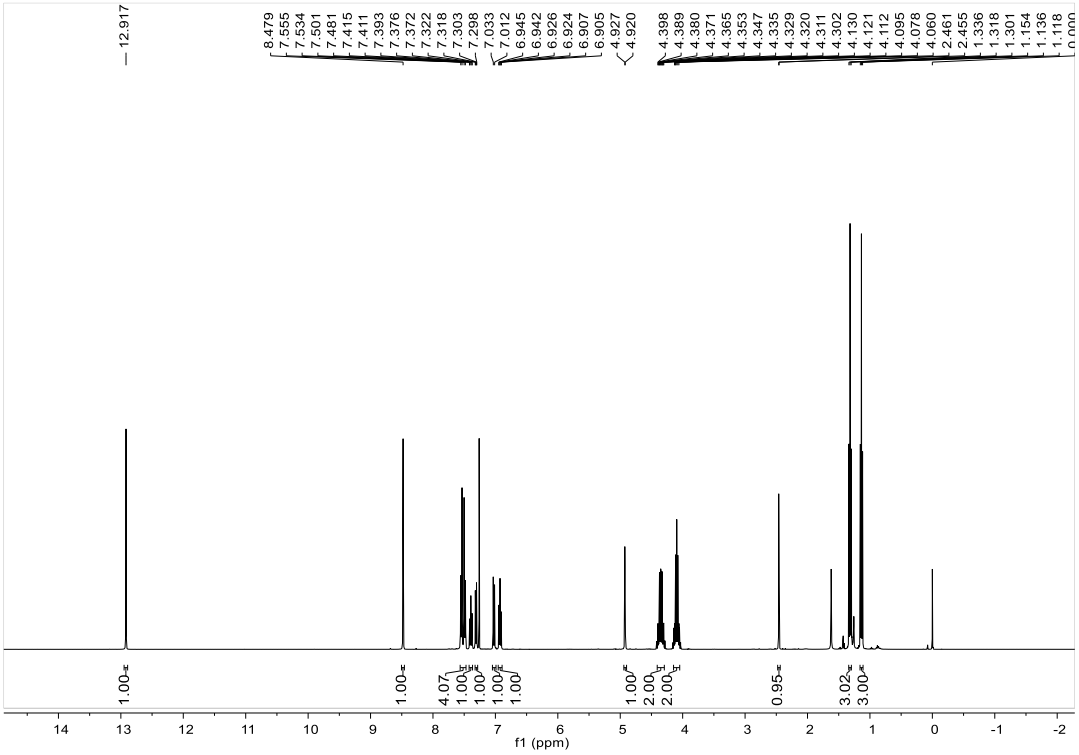

    Signal 1: DAD1 A, Sig=254,4 Ref=360,100

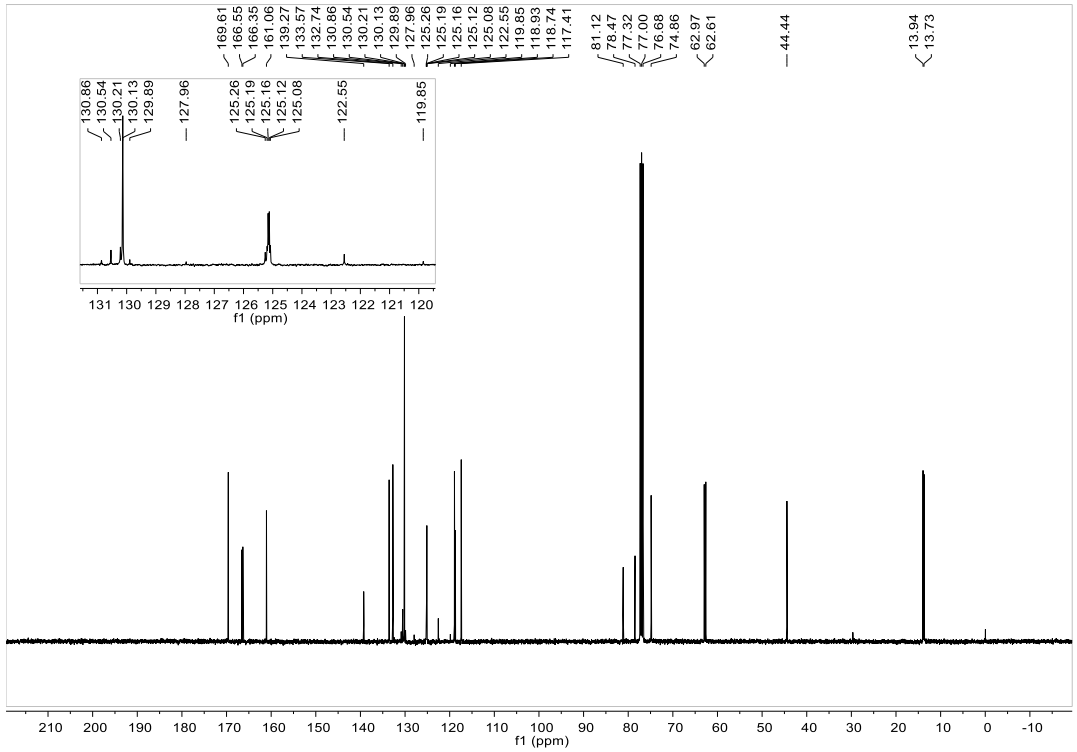
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  11.284 BB    0.3092  338.43790   14.56021   5.0712
       2  12.722 MM R  0.4516 6335.30176  233.79028  94.9288

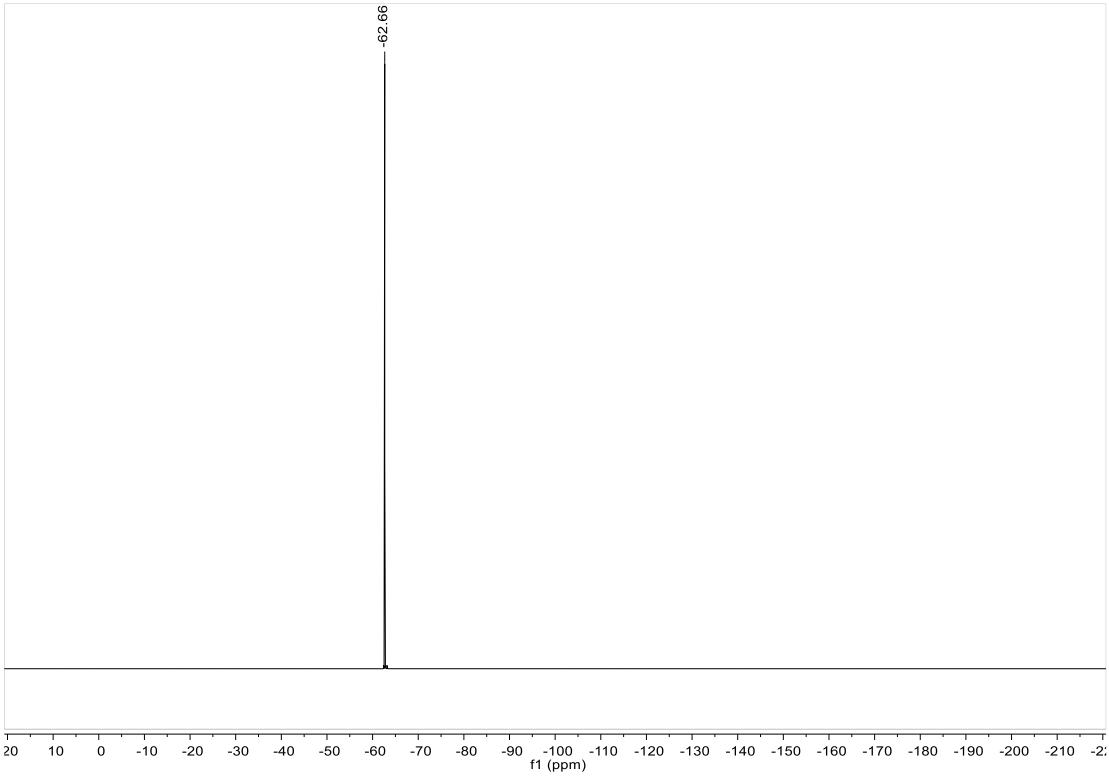
    Totals :                  6673.73965  248.35049
```


F3C
CO2Et
CO2Et
N
OH

3g

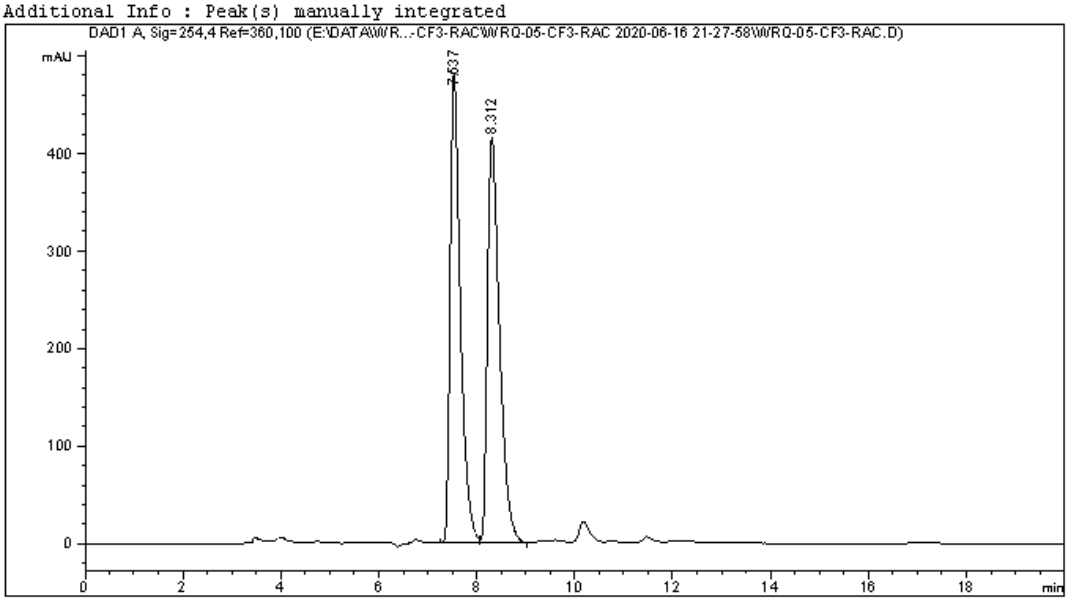

12.917
8.479
7.555
7.534
7.501
7.481
7.415
7.411
7.393
7.376
7.372
7.322
7.318
7.303
7.298
7.033
7.012
6.945
6.942
6.926
6.924
6.907
6.905
4.927
4.920
4.398
4.389
4.380
4.371
4.365
4.353
4.347
4.335
4.329
4.320
4.311
4.302
4.130
4.121
4.112
4.095
4.078
4.060
2.461
2.455
1.336
1.318
1.301
1.154
1.136
1.118
1.00
1.00
4.07
1.00
1.00
1.00
1.00
2.00
2.00
0.95
3.02
3.00
f1 (ppm)


169.61
166.55
166.35
161.06
139.27
133.57
132.74
130.86
130.54
130.21
130.13
129.89
127.96
125.26
125.19
125.16
125.12
125.08
122.55
119.85
118.93
118.74
117.41
81.12
78.47
77.32
77.00
76.68
74.86
62.97
62.61
44.44
13.94
13.73
f1 (ppm)


-62.66
20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

Data File E:\DATA\WRQ\WRQ-05-CF3-RAC\WRQ-05-CF3-RAC 2020-06-16 21-27-58\WRQ-05-CF3-RAC.D
Sample Name: WRQ-05-CF3-RAC

```
    ======================================================================
    Acq. Operator   : SYSTEM                          Seq. Line :   1
    Acq. Instrument : 1260                             Location :   63
    Injection Date  : 6/16/2020 9:29:32 PM                 Inj :   1
                                                   Inj Volume : 7.000 µl
    Acq. Method     : E:\DATA\WRQ\WRQ-05-CF3-RAC\WRQ-05-CF3-RAC 2020-06-16 21-27-58\WRQ-4-IE-95-5
                      -DAD-1ML-30MIN.M
    Last changed    : 6/16/2020 9:27:58 PM by SYSTEM
    Analysis Method : E:\DATA\WRQ\WRQ-05-CF3-RAC\WRQ-05-CF3-RAC 2020-06-16 21-27-58\WRQ-4-IE-95-5
                      -DAD-1ML-30MIN.M (Sequence Method)
    Last changed    : 7/20/2020 9:55:02 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

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

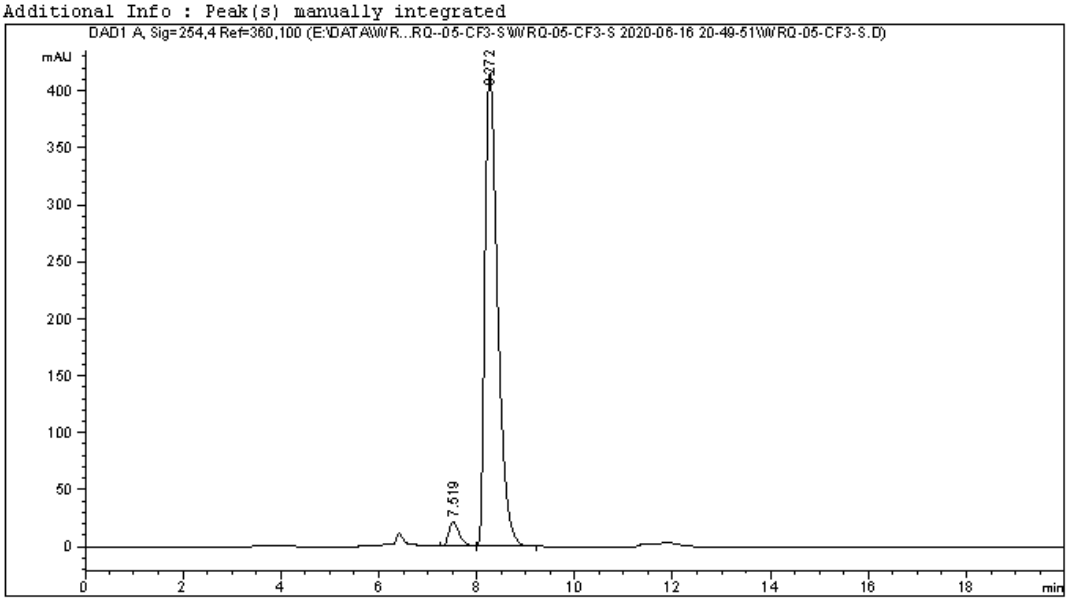

    Signal 1: DAD1 A, Sig=254,4 Ref=360,100

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   7.537 BV    0.2208 7093.07568  481.72916  50.0025
       2   8.312 VB    0.2573 7092.35742  415.08438  49.9975

    Totals :                  1.41854e4   896.81354
```

Data File E:\DATA\WRQ\WRQ--05-CF3-S\WRQ-05-CF3-S 2020-06-16 20-49-51\WRQ-05-CF3-S.D
Sample Name: WRQ-05-CF3-S

```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   1
    Acq. Instrument : 1260                           Location :   64
    Injection Date  : 6/16/2020 8:51:23 PM                Inj :   1
                                                  Inj Volume : 10.000 µl
    Acq. Method     : E:\DATA\WRQ\WRQ--05-CF3-S\WRQ-05-CF3-S 2020-06-16 20-49-51\WRQ-4-IE-95-5-
                      DAD-1ML-30MIN.M
    Last changed    : 6/16/2020 8:49:51 PM by SYSTEM
    Analysis Method : E:\DATA\WRQ\WRQ--05-CF3-S\WRQ-05-CF3-S 2020-06-16 20-49-51\WRQ-4-IE-95-5-
                      DAD-1ML-30MIN.M (Sequence Method)
    Last changed    : 7/20/2020 9:54:07 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```



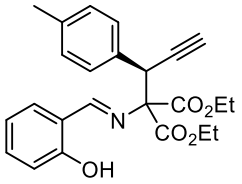

```
    ======================================================================
                           Area Percent Report
    ======================================================================

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

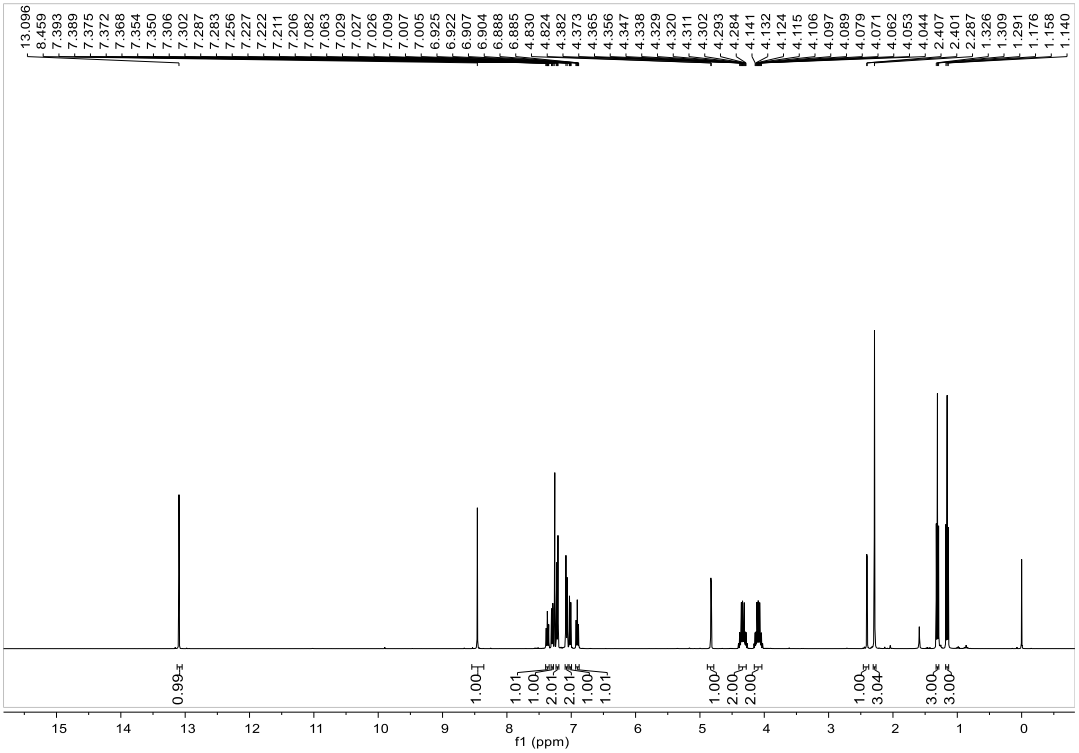

    Signal 1: DAD1 A, Sig=254,4 Ref=360,100

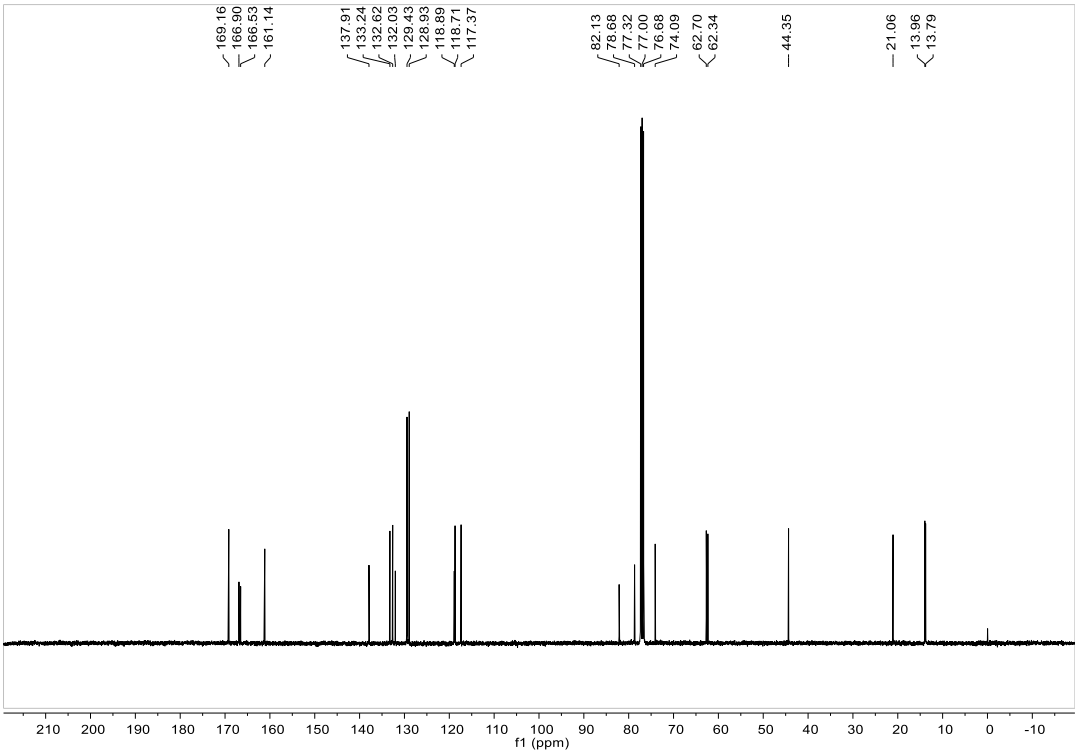
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   7.519 BB    0.2238  320.80942   21.65504   4.1342
       2   8.272 BB    0.2762 7439.04053  414.89069  95.8658

    Totals :                   7759.84995  436.54573
```


CO2Et
CO2Et
N
OH

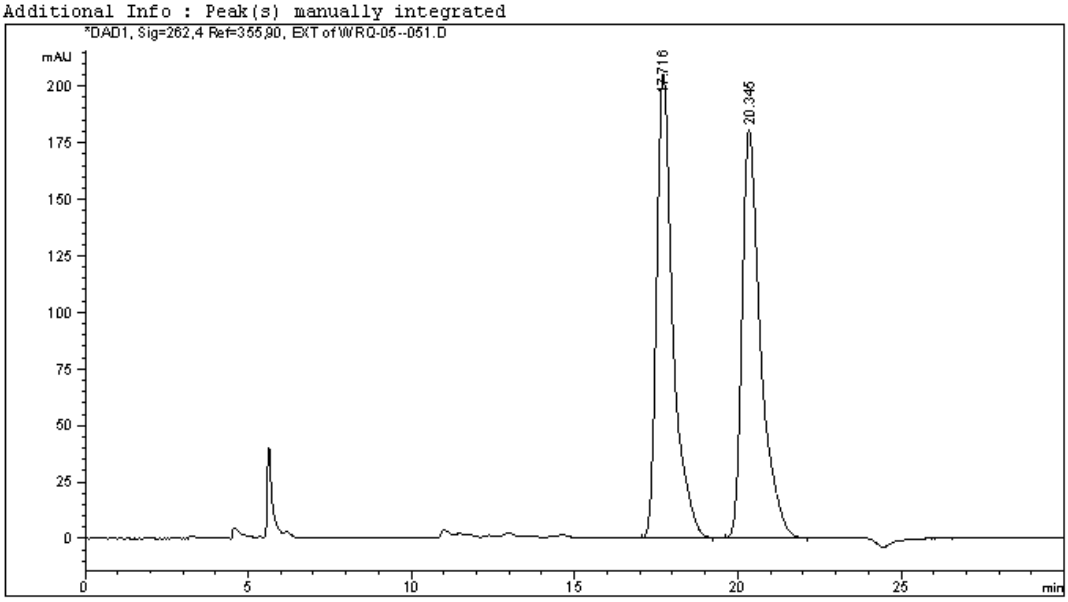
3h


13.096
8.459
7.393
7.389
7.375
7.372
7.368
7.354
7.350
7.306
7.302
7.287
7.283
7.256
7.227
7.222
7.211
7.206
7.082
7.063
7.029
7.027
7.026
7.009
7.007
7.005
6.925
6.922
6.907
6.904
6.888
6.885
4.830
4.824
4.382
4.373
4.365
4.356
4.347
4.338
4.329
4.320
4.311
4.302
4.293
4.284
4.141
4.132
4.124
4.115
4.106
4.097
4.089
4.079
4.071
4.062
4.053
4.044
2.407
2.401
2.287
1.326
1.309
1.291
1.176
1.158
1.140
0.99
1.00
1.01
1.00
2.01
2.01
1.00
1.01
1.00
2.00
2.00
1.00
3.04
3.00
3.00
f1 (ppm)


169.16
166.90
166.53
161.14
137.91
133.24
132.62
132.03
129.43
128.93
118.89
118.71
117.37
82.13
78.68
77.32
77.00
76.68
74.09
62.70
62.34
44.35
21.06
13.96
13.79
f1 (ppm)

Data File E:\DATA\WRQ\WRQ-05--05\WRQ-05-05-06-95-5 2019-12-18 01-01-11\WRQ-05--051.D
Sample Name: WRQ-05--06-Me

```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   2
    Acq. Instrument : 1260                          Location :   52
    Injection Date  : 12/18/2019 2:04:08 AM              Inj :   1
                                                  Inj Volume : 10.000 µl
    Acq. Method     : E:\DATA\WRQ\WRQ-05--05\WRQ-05-05-06-95-5 2019-12-18 01-01-11\WRQ-2-95-5-DAD
                      -1ML-60MIN.M
    Last changed    : 12/18/2019 1:01:11 AM by SYSTEM
    Analysis Method : E:\DATA\WRQ\WRQ-05--05\WRQ-05-05-06-95-5 2019-12-18 01-01-11\WRQ-2-95-5-DAD
                      -1ML-60MIN.M (Sequence Method)
    Last changed    : 7/21/2020 8:15:01 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

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

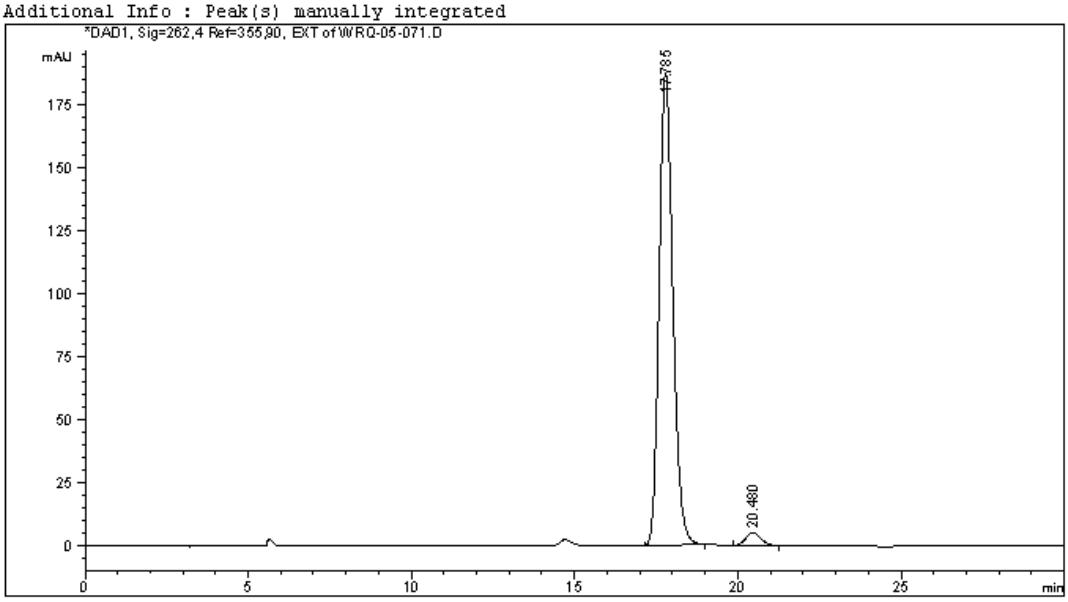

    Signal 1: DAD1, Sig=262,4 Ref=355,90, EXT
     Signal has been modified after loading from rawdata file!

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  17.716 BB    0.5042 7095.61768  205.33069  49.9542
       2  20.345 BB    0.5712 7108.61523  180.61755  50.0458

    Totals :                  1.42042e4   385.94824
```

Data File E:\DATA\WRQ\WRQ-05--07\WRQ-05-07-8-9-10 2019-12-19 23-03-29\WRQ-05-071.D
Sample Name: WRQ-05-08

```
    ======================================================================
    Acq. Operator   : SYSTEM                          Seq. Line :   2
    Acq. Instrument : 1260                              Location :   54
    Injection Date  : 12/19/2019 11:36:27 PM                 Inj :   1
                                                    Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\WRQ\WRQ-05--07\WRQ-05-07-8-9-10 2019-12-19 23-03-29\WRQ-2-95-5-DAD-
                      1ML-30MIN.M
    Last changed    : 12/19/2019 11:03:29 PM by SYSTEM
    Analysis Method : E:\DATA\WRQ\WRQ-05--07\WRQ-05-07-8-9-10 2019-12-19 23-03-29\WRQ-2-95-5-DAD-
                      1ML-30MIN.M (Sequence Method)
    Last changed    : 7/21/2020 8:16:33 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```



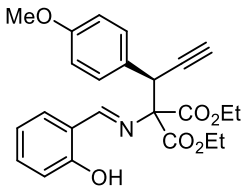

```
    ======================================================================
                           Area Percent Report
    ======================================================================

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

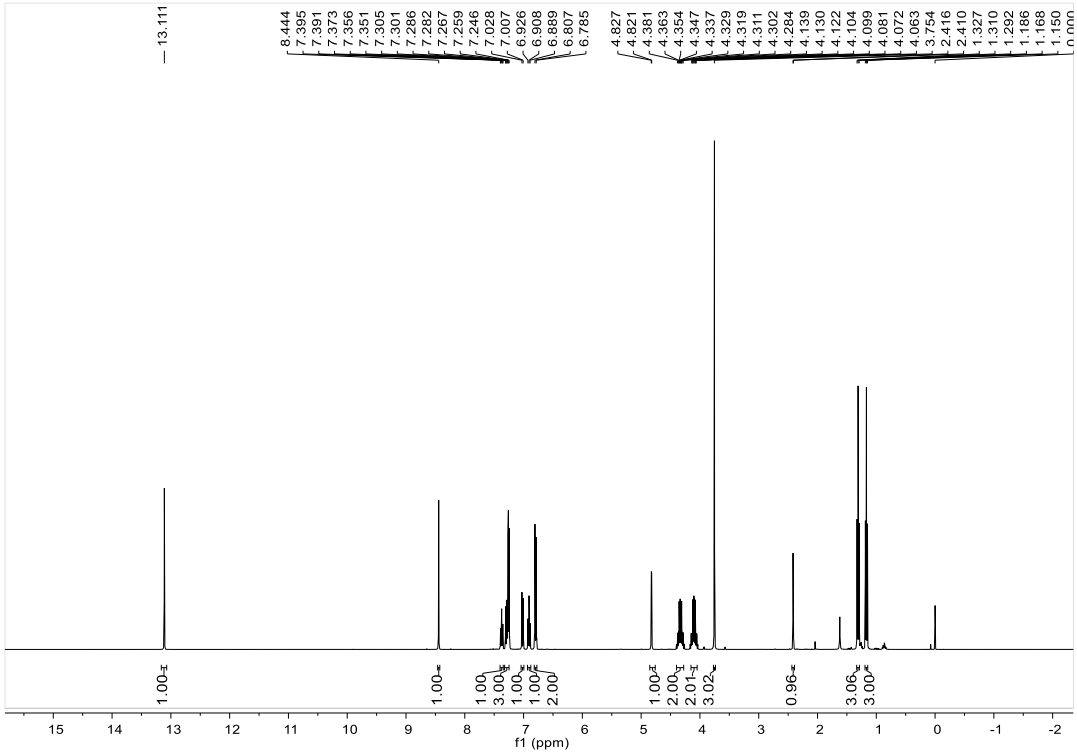

    Signal 1: DAD1, Sig=262,4 Ref=355,90, EXT
     Signal has been modified after loading from rawdata file!

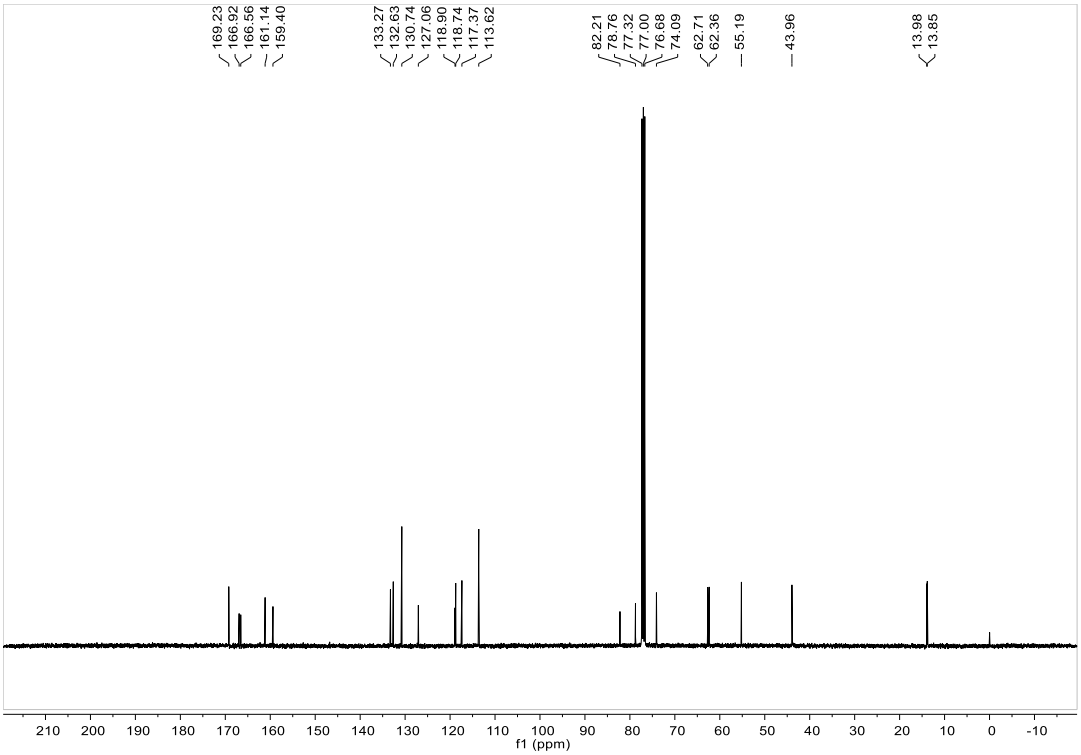
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  17.785 BB    0.4526 5495.39795  187.11365  97.0338
       2  20.480 BB    0.3829  167.98982    5.18680   2.9662

    Totals :                  5663.38777  192.30045
```


MeO
CO2Et
CO2Et
N
OH

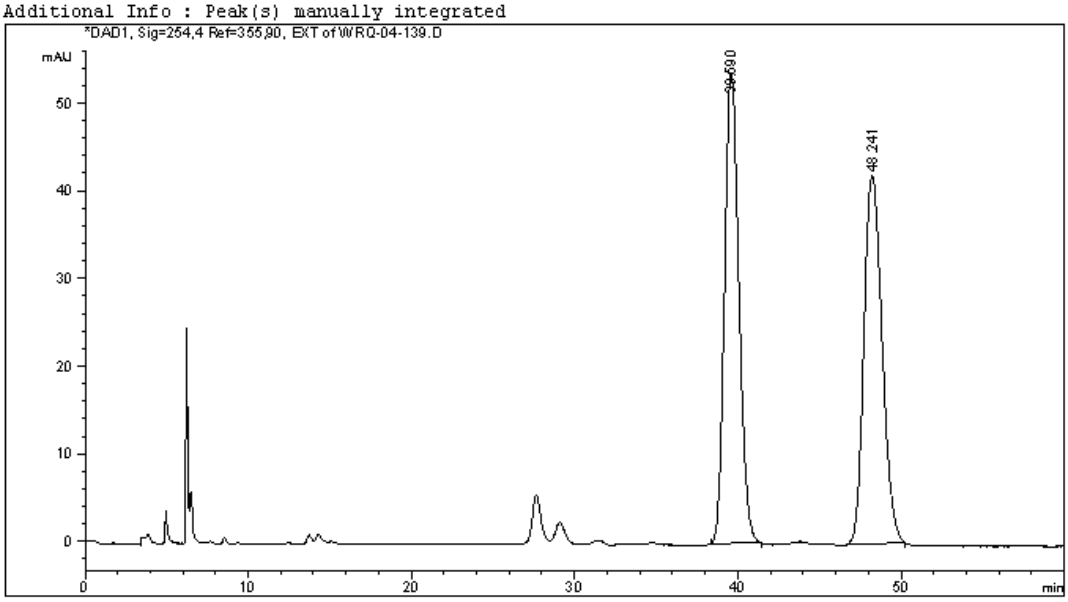
3i


13.111
8.444
7.395
7.391
7.373
7.356
7.351
7.305
7.301
7.286
7.282
7.267
7.259
7.246
7.028
7.007
6.926
6.908
6.889
6.807
6.785
4.827
4.821
4.381
4.363
4.354
4.347
4.337
4.329
4.319
4.311
4.302
4.284
4.139
4.130
4.122
4.104
4.099
4.081
4.072
4.063
3.754
2.416
2.410
1.327
1.310
1.292
1.186
1.168
1.150
1.00
1.00
1.00
3.00
1.00
1.00
2.00
1.00
2.00
2.01
3.02
0.96
3.06
3.00
f1 (ppm)


169.23
166.92
166.56
161.14
159.40
133.27
132.63
130.74
127.06
118.90
118.74
117.37
113.62
82.21
78.76
77.32
77.00
76.68
74.09
62.71
62.36
55.19
43.96
13.98
13.85
f1 (ppm)

Data File E:\DATA\WRQ\WRQ-04-139\WRQ-04-139-AD-98 2019-12-01 00-46-53\WRQ-04-139.D
Sample Name: WRQ-04-139

```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   1
    Acq. Instrument : 1260                          Location :   51
    Injection Date  : 12/1/2019 12:48:18 AM              Inj :   1
                                                  Inj Volume : 10.000 µl
    Acq. Method     : E:\DATA\WRQ\WRQ-04-139\WRQ-04-139-AD-98 2019-12-01 00-46-53\WRQ-2-98-2-DAD-
                      1ML.M
    Last changed    : 12/1/2019 12:46:53 AM by SYSTEM
    Analysis Method : E:\DATA\WRQ\WRQ-04-139\WRQ-04-139-AD-98 2019-12-01 00-46-53\WRQ-2-98-2-DAD-
                      1ML.M (Sequence Method)
    Last changed    : 7/20/2020 10:47:02 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

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

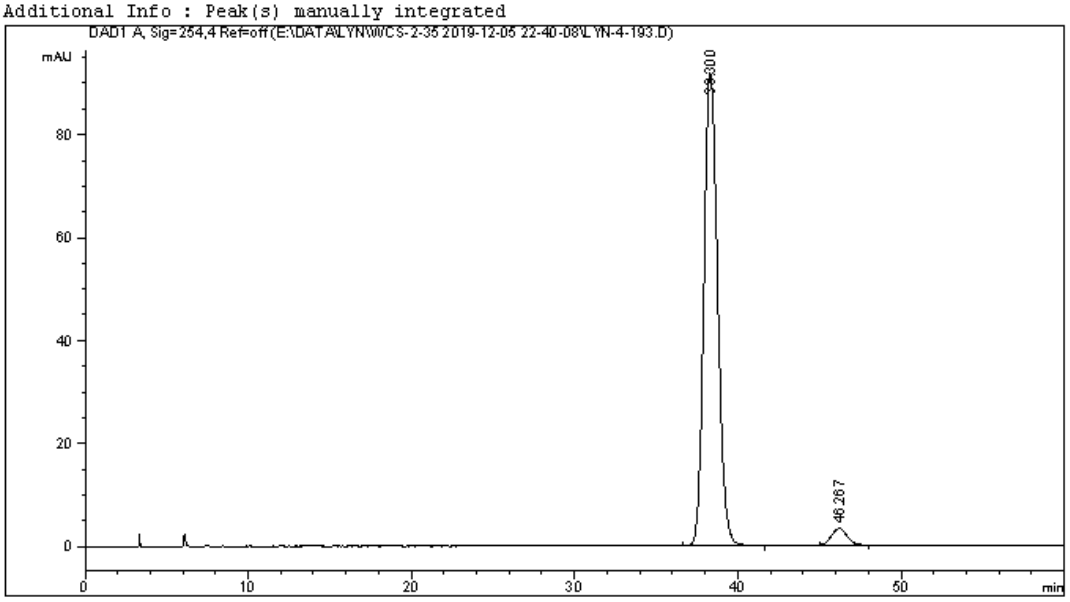

    Signal 1: DAD1, Sig=254,4 Ref=355,90, EXT
     Signal has been modified after loading from rawdata file!

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]        %
    ----|-------|----|-------|----------|----------|--------|
       1  39.590 BB    0.7156 3262.94092   53.64431  50.2936
       2  48.241 BB    0.9024 3224.84229   41.97158  49.7064

    Totals :                  6487.78320   95.61590
```

Data File E:\DATA\LYN\WCS-2-35 2019-12-05 22-40-08\LYN-4-193.D
Sample Name: WRQ-04-145

```
    ======================================================================
    Acq. Operator   : SYSTEM                          Seq. Line :   4
    Acq. Instrument : 1260                             Location :   53
    Injection Date  : 12/6/2019 12:15:38 AM                 Inj :   1
                                                    Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\LYN\WCS-2-35 2019-12-05 22-40-08\WRQ-2-98-2--70min-DAD.M
    Last changed    : 12/5/2019 10:40:08 PM by SYSTEM
    Analysis Method : E:\DATA\LYN\WCS-2-35 2019-12-05 22-40-08\WRQ-2-98-2--70min-DAD.M (Sequence
                      Method)
    Last changed    : 7/20/2020 10:21:54 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```



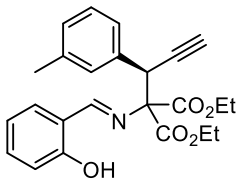

```
    ======================================================================
                            Area Percent Report
    ======================================================================

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

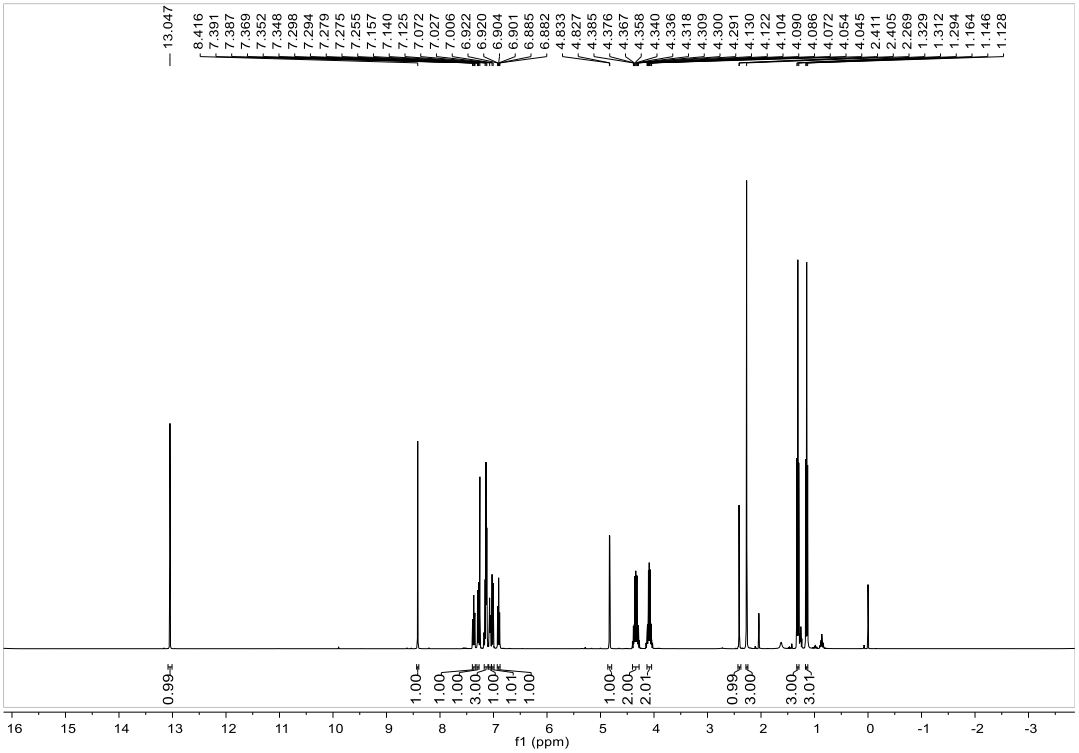

    Signal 1: DAD1 A, Sig=254,4 Ref=off

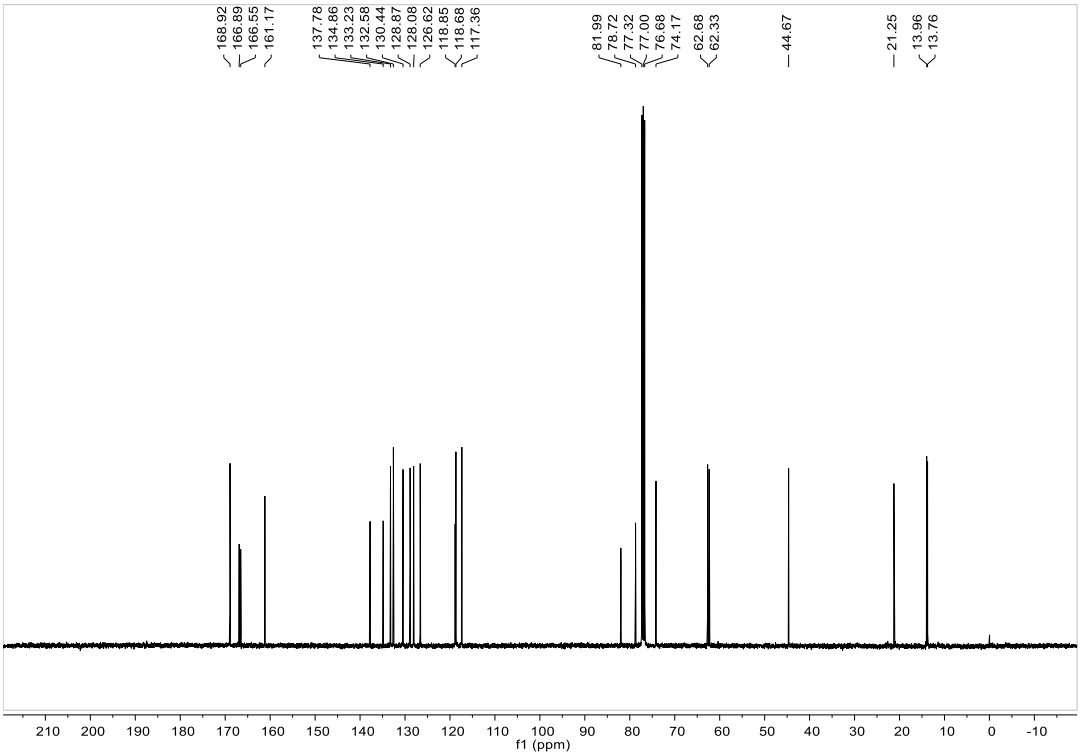
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]        %
    ----|-------|----|-------|----------|----------|--------|
       1  38.300 MM R  0.9701 5340.50244   91.74818  96.0108
       2  46.267 MM R  1.0928  221.89731    3.38426   3.9892

    Totals :                  5562.39975   95.13244
```


CO2Et
CO2Et
N
OH

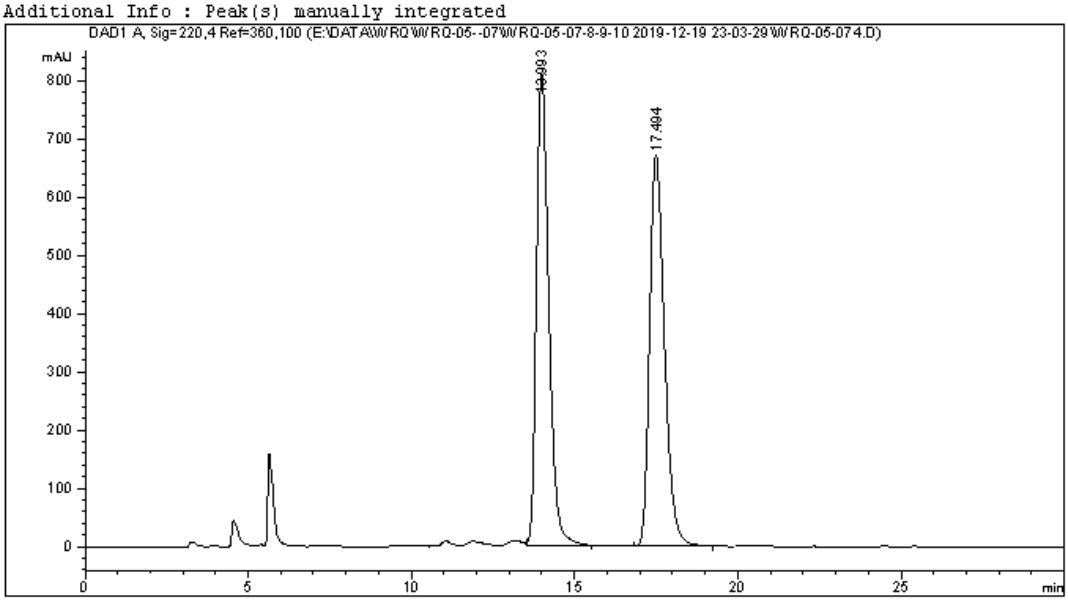
3j


13.047
8.416
7.391
7.387
7.369
7.352
7.348
7.298
7.294
7.279
7.275
7.255
7.157
7.140
7.125
7.072
7.027
7.006
6.922
6.920
6.904
6.901
6.885
6.882
4.833
4.827
4.385
4.376
4.367
4.358
4.340
4.336
4.318
4.309
4.300
4.291
4.130
4.122
4.104
4.090
4.086
4.072
4.054
4.045
2.411
2.405
2.269
1.329
1.312
1.294
1.164
1.146
1.128
0.99
1.00
1.00
3.00
1.00
1.01
1.00
1.00
2.00
2.01
0.99
3.00
3.00
3.01
f1 (ppm)


168.92
166.89
166.55
161.17
137.78
134.86
133.23
132.58
130.44
128.87
128.08
126.62
118.85
118.68
117.36
81.99
78.72
77.32
77.00
76.68
74.17
62.68
62.33
44.67
21.25
13.96
13.76
f1 (ppm)

Data File E:\DATA\WRQ\WRQ-05--07\WRQ-05-07-8-9-10 2019-12-19 23-03-29\WRQ-05-074.D
Sample Name: WRQ-05-10

```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   5
    Acq. Instrument : 1260                          Location :   56
    Injection Date  : 12/20/2019 1:41:06 AM              Inj :   1
                                                  Inj Volume : 10.000 µl
    Acq. Method     : E:\DATA\WRQ\WRQ-05--07\WRQ-05-07-8-9-10 2019-12-19 23-03-29\WRQ-2-95-5-DAD-
                      1ML-60MIN.M
    Last changed    : 12/19/2019 11:03:29 PM by SYSTEM
    Analysis Method : E:\DATA\WRQ\WRQ-05--07\WRQ-05-07-8-9-10 2019-12-19 23-03-29\WRQ-2-95-5-DAD-
                      1ML-60MIN.M (Sequence Method)
    Last changed    : 7/21/2020 8:21:55 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

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

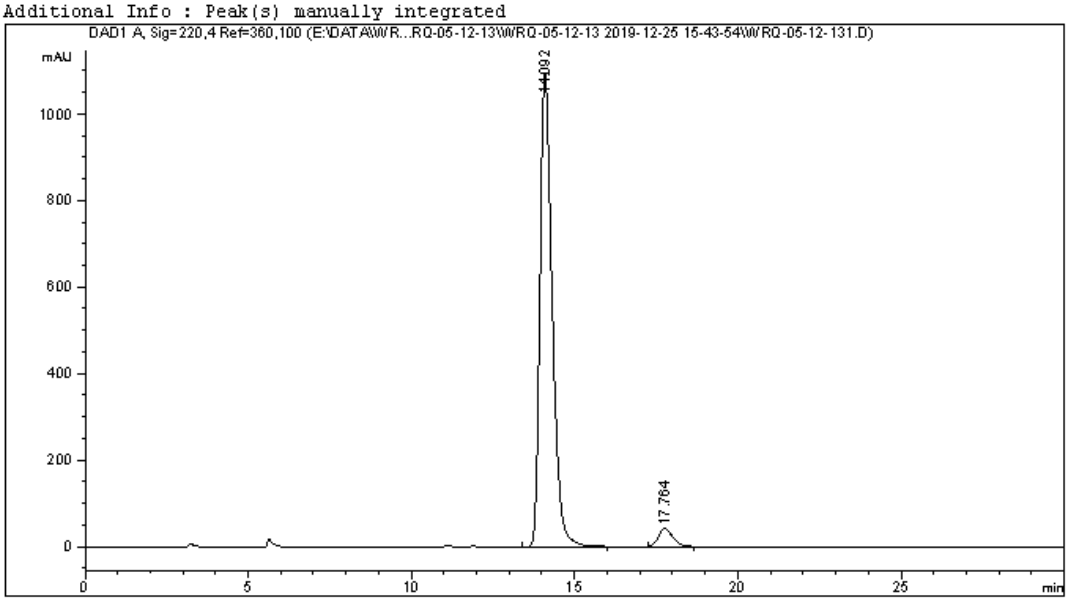

    Signal 1: DAD1 A, Sig=220,4 Ref=360,100

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  13.993 FM R  0.4369 2.12496e4   810.65375  50.1537
       2  17.494 BB    0.4772 2.11193e4   671.04120  49.8463

    Totals :                  4.23688e4  1481.69495
```

Data File E:\DATA\WRQ\WRQ-05-12-13\WRQ-05-12-13 2019-12-25 15-43-54\WRQ-05-12-131.D
Sample Name: WRQ-05-13-Me

```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   2
    Acq. Instrument : 1260                           Location :   52
    Injection Date  : 12/25/2019 4:16:51 PM              Inj :   1
                                                   Inj Volume : 7.000 µl
    Acq. Method     : E:\DATA\WRQ\WRQ-05-12-13\WRQ-05-12-13 2019-12-25 15-43-54\WRQ-2-95-5-DAD-
                      1ML-30MIN.M
    Last changed    : 12/25/2019 3:43:54 PM by SYSTEM
    Analysis Method : E:\DATA\WRQ\WRQ-05-12-13\WRQ-05-12-13 2019-12-25 15-43-54\WRQ-2-95-5-DAD-
                      1ML-30MIN.M (Sequence Method)
    Last changed    : 7/21/2020 8:23:13 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```



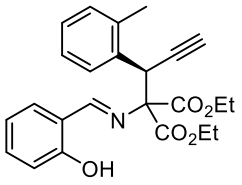

```
    ======================================================================
                           Area Percent Report
    ======================================================================

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

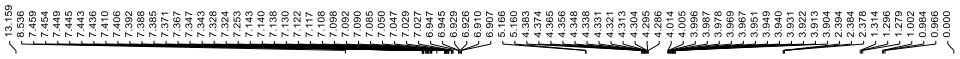

    Signal 1: DAD1 A, Sig=220,4 Ref=360,100

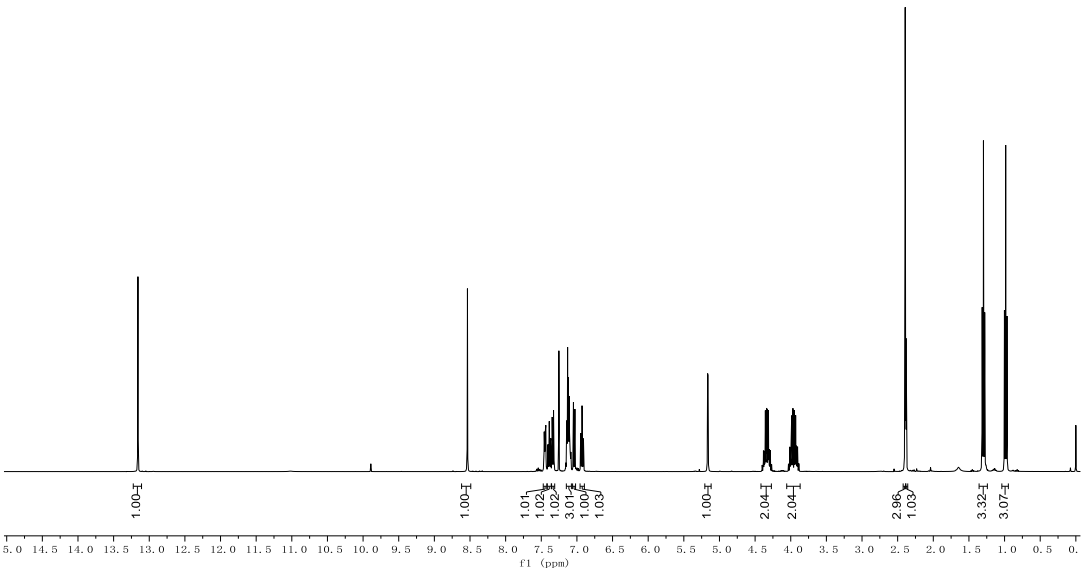
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  14.092 MM R  0.4321 2.83623e4  1093.86584  95.7648
       2  17.764 MM R  0.5092 1254.31787   41.05161   4.2352

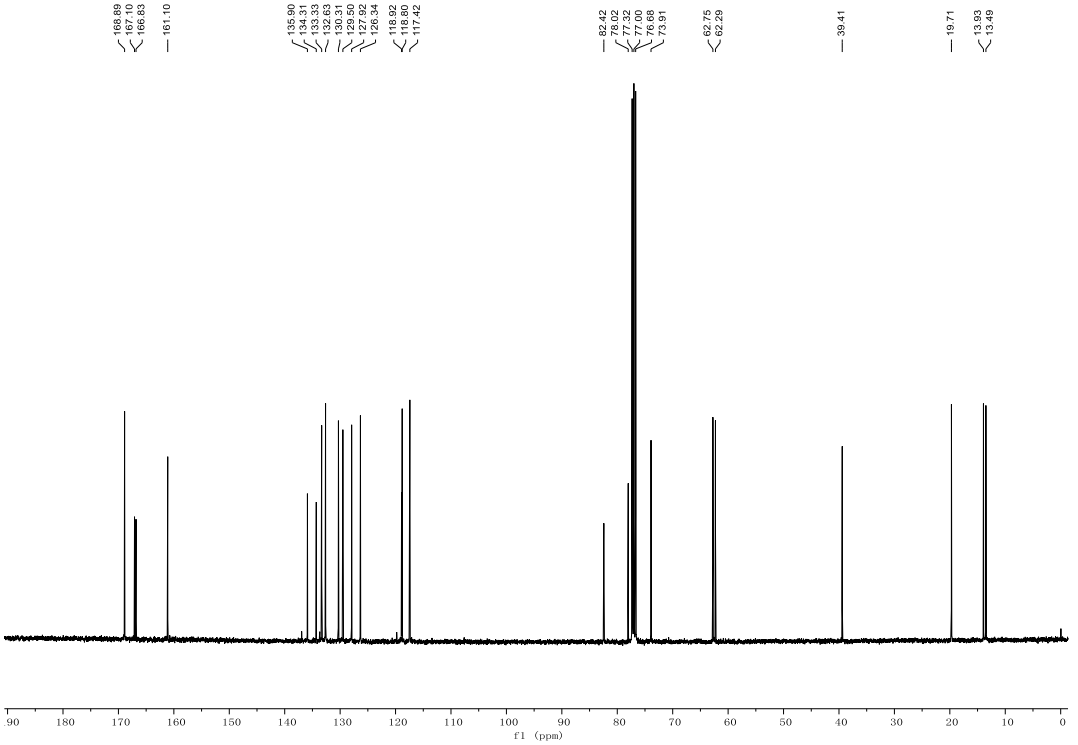
    Totals :                  2.96166e4  1134.91745
```



3k

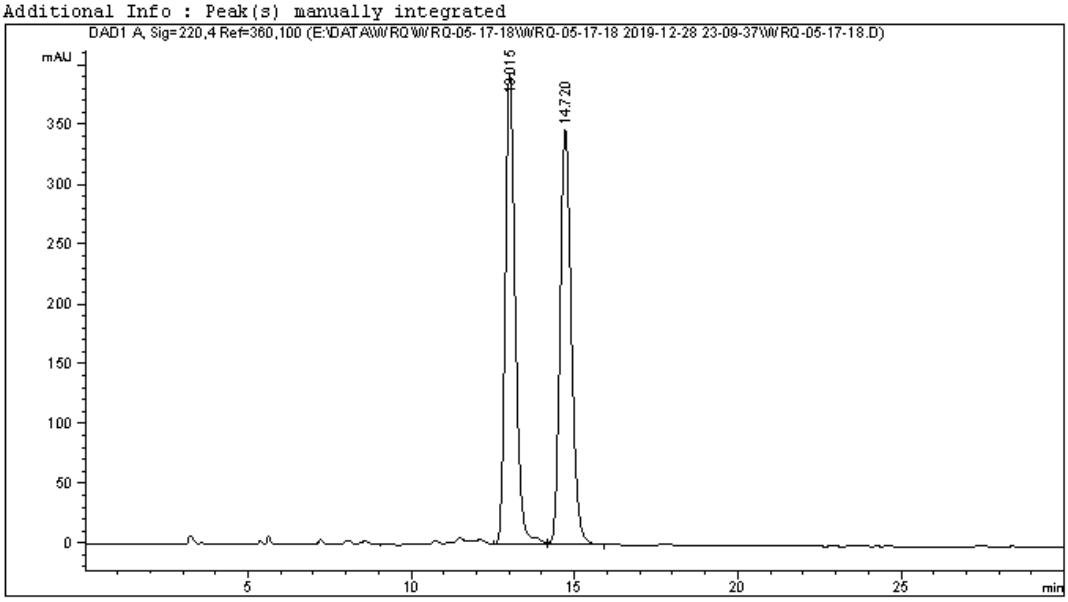






Data File E:\DATA\WRQ\WRQ-05-17-18\WRQ-05-17-18 2019-12-28 23-09-37\WRQ-05-17-18.D
Sample Name: WRQ-05-17

```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   1
    Acq. Instrument : 1260                          Location :   51
    Injection Date  : 12/28/2019 11:11:03 PM             Inj :   1
                                                  Inj Volume : 7.000 µl
    Acq. Method     : E:\DATA\WRQ\WRQ-05-17-18\WRQ-05-17-18 2019-12-28 23-09-37\WRQ-2-95-5-DAD-
                      1ML-30MIN.M
    Last changed    : 12/28/2019 11:09:37 PM by SYSTEM
    Analysis Method : E:\DATA\WRQ\WRQ-05-17-18\WRQ-05-17-18 2019-12-28 23-09-37\WRQ-2-95-5-DAD-
                      1ML-30MIN.M (Sequence Method)
    Last changed    : 6/27/2021 10:28:38 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

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

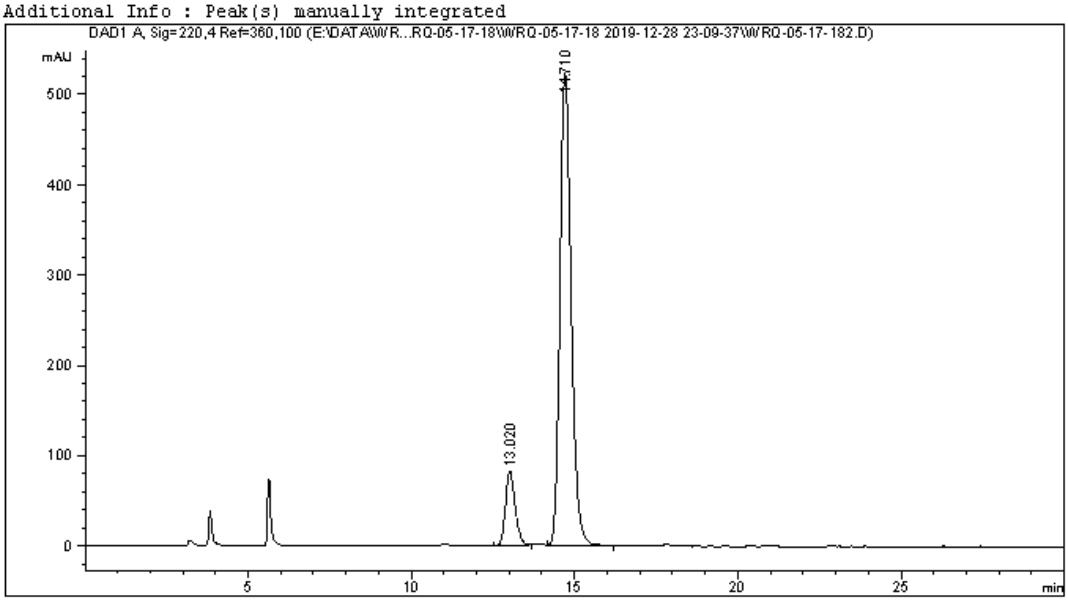

    Signal 1: DAD1 A, Sig=220,4 Ref=360,100

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  13.015 BV    0.3285 8453.36914  393.68243  50.3530
       2  14.720 VB    0.3734 8334.84082  346.79016  49.6470

    Totals :                  1.67882e4   740.47260
```

Data File E:\DATA\WRQ\WRQ-05-17-18\WRQ-05-17-18 2019-12-28 23-09-37\WRQ-05-17-182.D
Sample Name: WRQ-05-19

```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   3
    Acq. Instrument : 1260                           Location :   53
    Injection Date  : 12/29/2019 12:14:03 AM              Inj :   1
                                                  Inj Volume : 7.000 µl
    Acq. Method     : E:\DATA\WRQ\WRQ-05-17-18\WRQ-05-17-18 2019-12-28 23-09-37\WRQ-2-95-5-DAD-
                      1ML-30MIN.M
    Last changed    : 12/28/2019 11:09:37 PM by SYSTEM
    Analysis Method : E:\DATA\WRQ\WRQ-05-17-18\WRQ-05-17-18 2019-12-28 23-09-37\WRQ-2-95-5-DAD-
                      1ML-30MIN.M (Sequence Method)
    Last changed    : 6/27/2021 10:28:38 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```



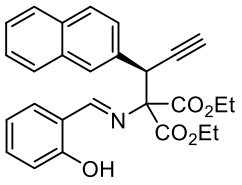

```
    ======================================================================
                           Area Percent Report
    ======================================================================

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

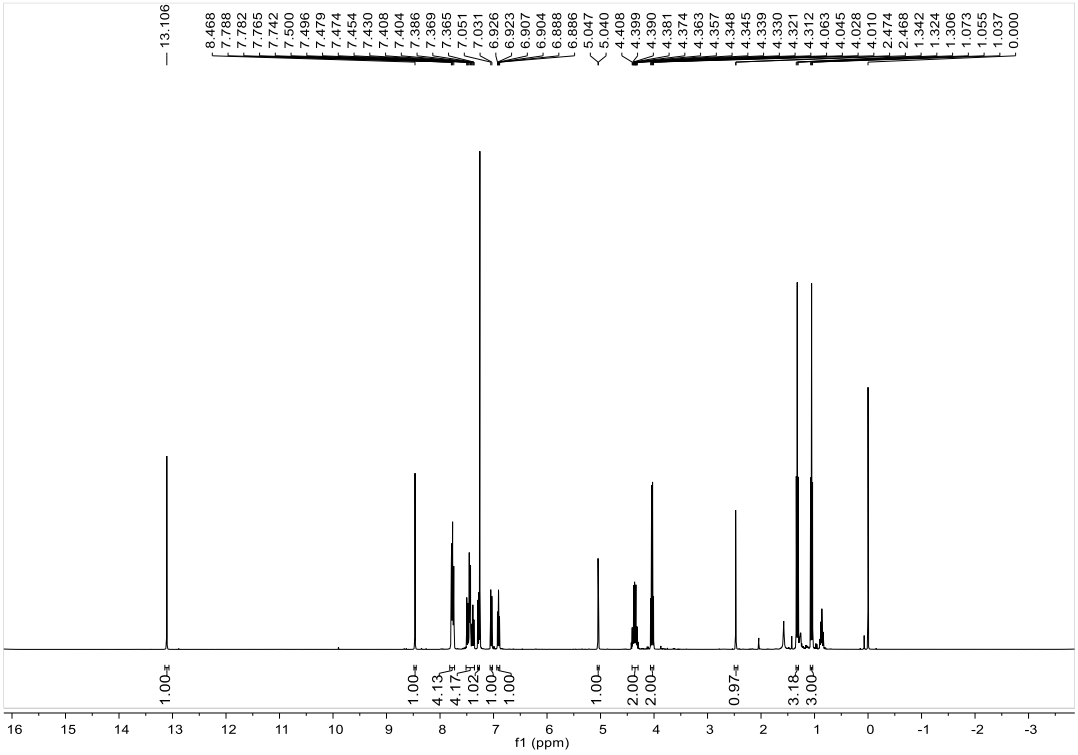

    Signal 1: DAD1 A, Sig=220,4 Ref=360,100

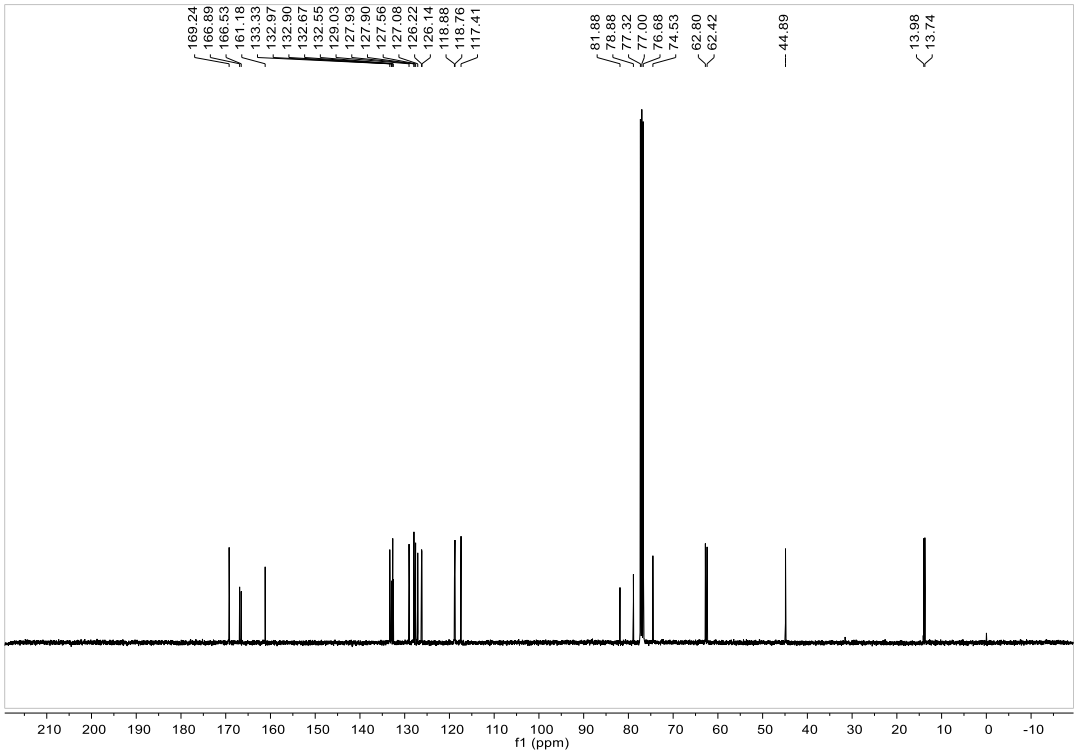
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  13.020 BB    0.3092 1688.35095   82.01949  11.8889
       2  14.710 BB    0.3702 1.25127e4   522.84393  88.1111

    Totals :                  1.42011e4   604.86343
```


CO2Et
CO2Et
N
OH

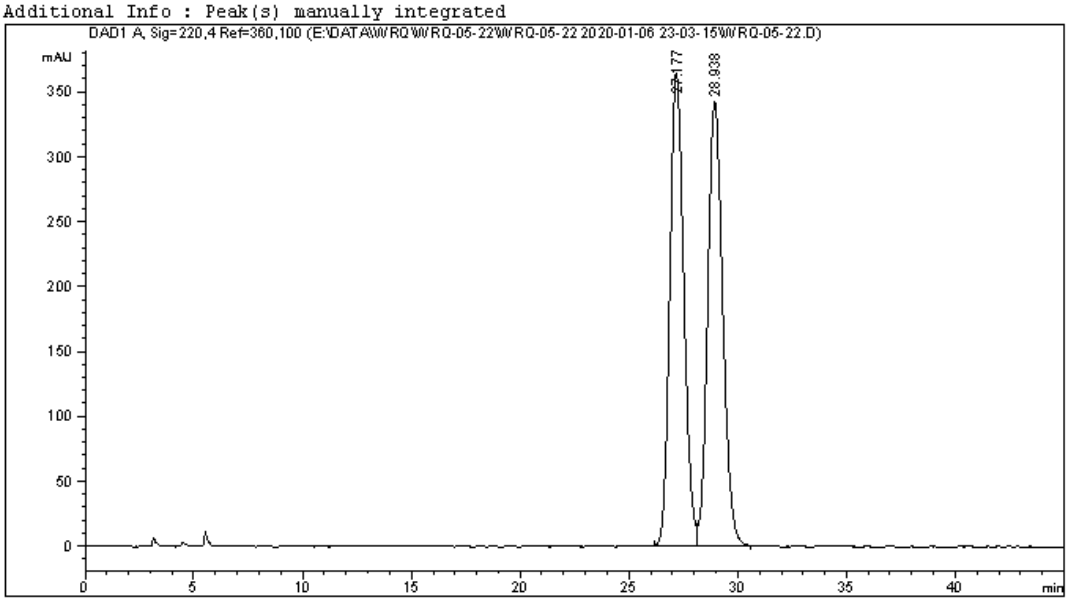
3l


13.106
8.468
7.788
7.782
7.765
7.742
7.500
7.496
7.479
7.474
7.454
7.430
7.408
7.404
7.386
7.369
7.365
7.051
7.031
6.926
6.923
6.907
6.904
6.888
6.886
5.047
5.040
4.408
4.399
4.390
4.381
4.374
4.363
4.357
4.348
4.345
4.339
4.330
4.321
4.312
4.063
4.045
4.028
4.010
2.474
2.468
1.342
1.324
1.306
1.073
1.055
1.037
0.000
1.00
1.00
4.13
4.17
1.02
1.00
1.00
1.00
2.00
2.00
0.97
3.18
3.00
f1 (ppm)


169.24
166.89
166.53
161.18
133.33
132.97
132.90
132.67
132.55
129.03
127.93
127.90
127.56
127.08
126.22
126.14
118.88
118.76
117.41
81.88
78.88
77.32
77.00
76.68
74.53
62.80
62.42
44.89
13.98
13.74
f1 (ppm)

Data File E:\DATA\WRQ\WRQ-05-22\WRQ-05-22 2020-01-06 23-03-15\WRQ-05-22.D
Sample Name: WRQ-05-22

```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   1
    Acq. Instrument : 1260                           Location :   51
    Injection Date  : 1/6/2020 11:04:48 PM               Inj :   1
                                                  Inj Volume : 7.000 µl
    Acq. Method     : E:\DATA\WRQ\WRQ-05-22\WRQ-05-22 2020-01-06 23-03-15\WRQ-2-95-5-DAD-1ML-
                      30MIN.M
    Last changed    : 1/6/2020 11:52:23 PM by SYSTEM
                      (modified after loading)
    Analysis Method : E:\DATA\WRQ\WRQ-05-22\WRQ-05-22 2020-01-06 23-03-15\WRQ-2-95-5-DAD-1ML-
                      30MIN.M (Sequence Method)
    Last changed    : 7/21/2020 8:59:28 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

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

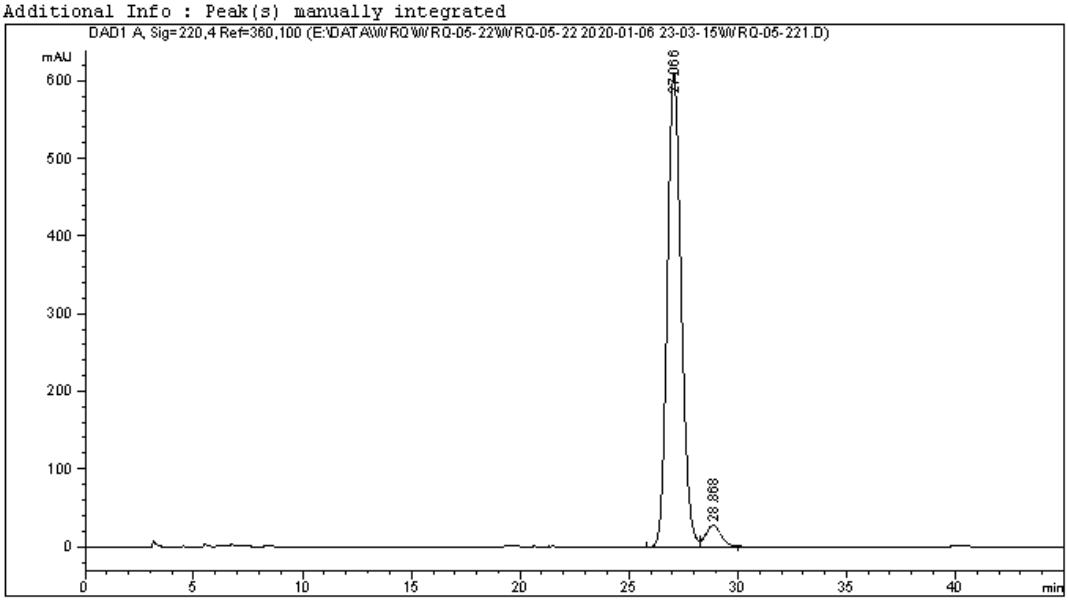

    Signal 1: DAD1 A, Sig=220,4 Ref=360,100

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  27.177 BV    0.6639 1.62153e4   363.14661  49.7110
       2  28.938 VB    0.6908 1.64038e4   341.86246  50.2890

    Totals :                  3.26191e4   705.00906
```

Data File E:\DATA\WRQ\WRQ-05-22\WRQ-05-22 2020-01-06 23-03-15\WRQ-05-221.D
Sample Name: WRQ-05-23

```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   2
    Acq. Instrument : 1260                           Location :   52
    Injection Date  : 1/6/2020 11:54:17 PM               Inj :   1
                                                  Inj Volume : 7.000 µl
    Acq. Method     : E:\DATA\WRQ\WRQ-05-22\WRQ-05-22 2020-01-06 23-03-15\WRQ-2-95-5-DAD-1ML-
                      30MIN.M
    Last changed    : 1/6/2020 11:52:23 PM by SYSTEM
    Analysis Method : E:\DATA\WRQ\WRQ-05-22\WRQ-05-22 2020-01-06 23-03-15\WRQ-2-95-5-DAD-1ML-
                      30MIN.M (Sequence Method)
    Last changed    : 7/21/2020 8:58:57 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```



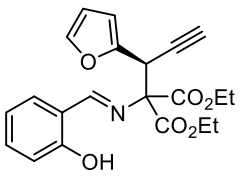

```
    ======================================================================
                           Area Percent Report
    ======================================================================

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

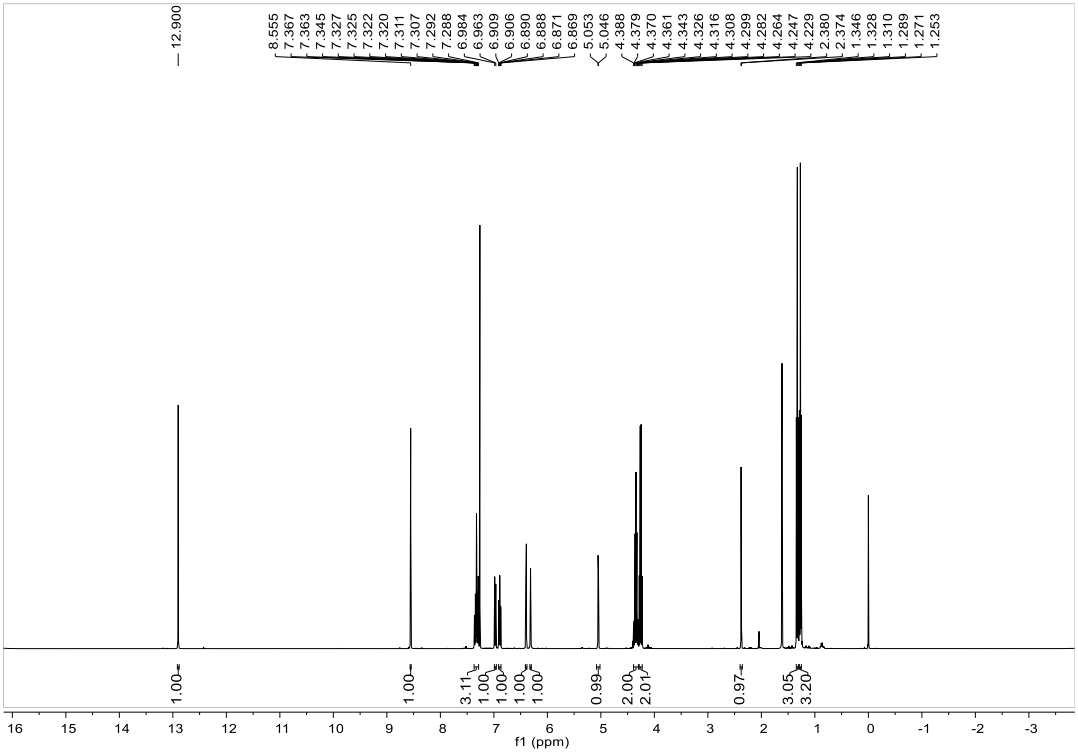

    Signal 1: DAD1 A, Sig=220,4 Ref=360,100

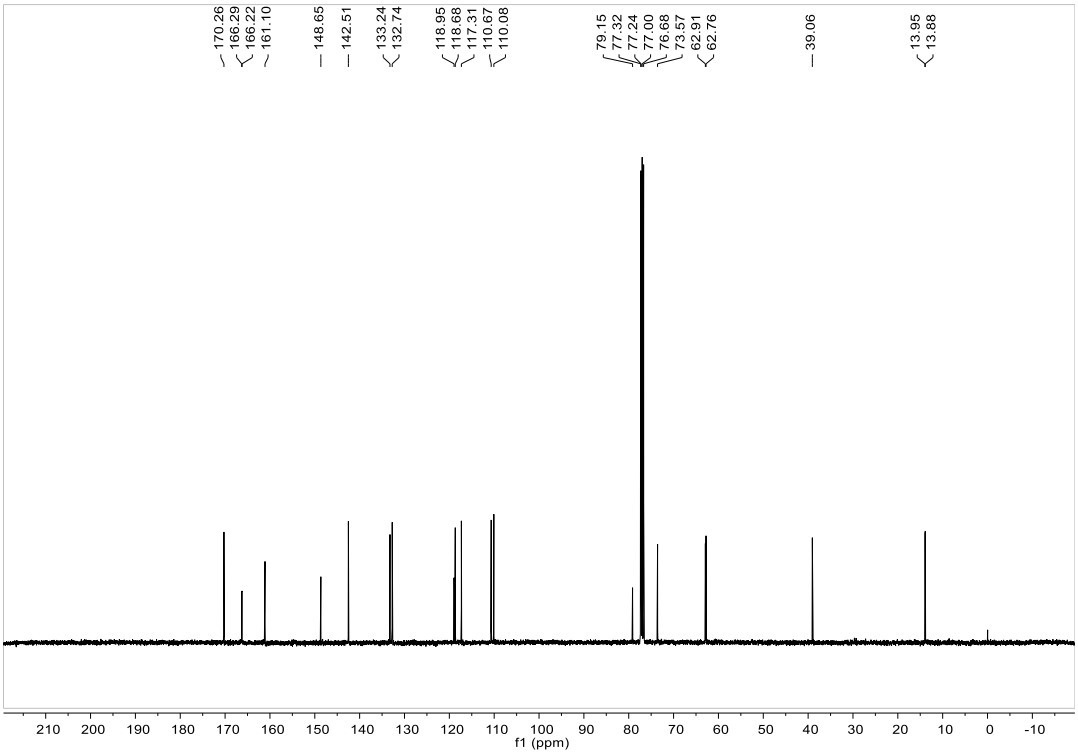
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  27.066 MF R  0.7405 2.70678e4   609.22577  95.4301
       2  28.868 FM R  0.8004 1296.21387   26.99238   4.5699

    Totals :                  2.83640e4   636.21815
```


O
N
CO2Et
CO2Et
OH

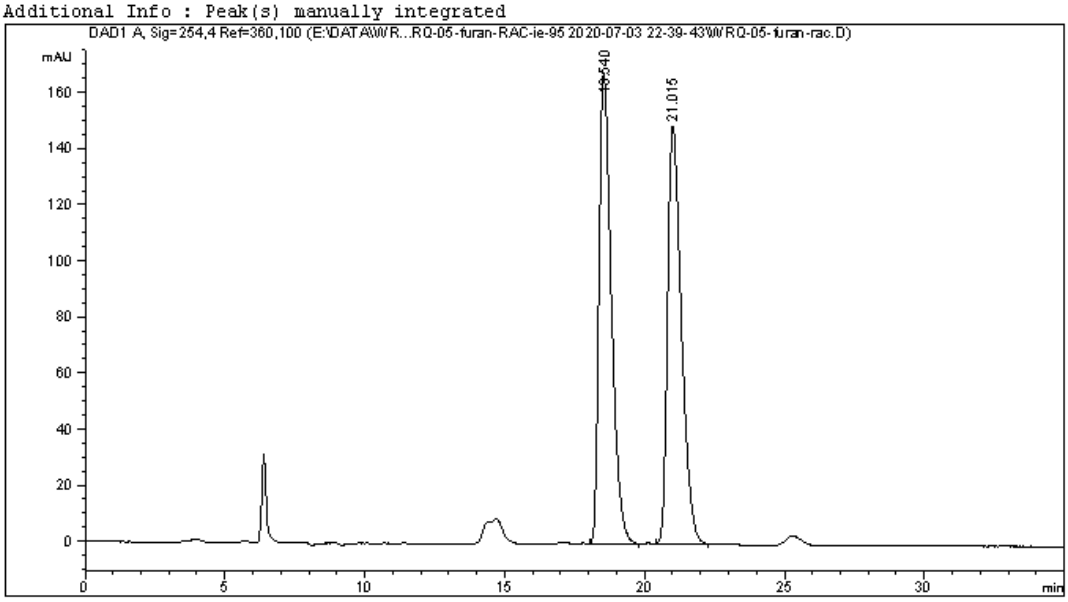
3m


12.900
8.555
7.367
7.363
7.345
7.327
7.325
7.322
7.320
7.311
7.307
7.292
7.288
6.984
6.963
6.909
6.906
6.890
6.888
6.871
6.869
5.053
5.046
4.388
4.379
4.370
4.361
4.343
4.326
4.316
4.308
4.299
4.282
4.264
4.247
4.229
2.380
2.374
1.346
1.328
1.310
1.289
1.271
1.253
1.00
1.00
3.11
1.00
1.00
1.00
1.00
0.99
2.00
2.01
0.97
3.05
3.20
f1 (ppm)


170.26
166.29
166.22
161.10
148.65
142.51
133.24
132.74
118.95
118.68
117.31
110.67
110.08
79.15
77.32
77.24
77.00
76.68
73.57
62.91
62.76
39.06
13.95
13.88
f1 (ppm)

Data File E:\DATA\WR...n-rac\WRQ-05-furan-RAC-ie-95 2020-07-03 22-39-43\WRQ-05-furan-rac.D
Sample Name: WRQ-05-furan-rac

```
    ======================================================================
    Acq. Operator   : SYSTEM                          Seq. Line :   1
    Acq. Instrument : 1260                             Location :   63
    Injection Date  : 7/3/2020 10:41:20 PM                  Inj :   1
                                                    Inj Volume : 15.000 µl
    Acq. Method     : E:\DATA\WRQ\WRQ-05-furan-rac\WRQ-05-furan-RAC-ie-95 2020-07-03 22-39-43\WRQ
                      -4-IE-95-5-DAD-1ML-30MIN.M
    Last changed    : 7/3/2020 10:39:43 PM by SYSTEM
    Analysis Method : E:\DATA\WRQ\WRQ-05-furan-rac\WRQ-05-furan-RAC-ie-95 2020-07-03 22-39-43\WRQ
                      -4-IE-95-5-DAD-1ML-30MIN.M (Sequence Method)
    Last changed    : 7/20/2020 8:19:08 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

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

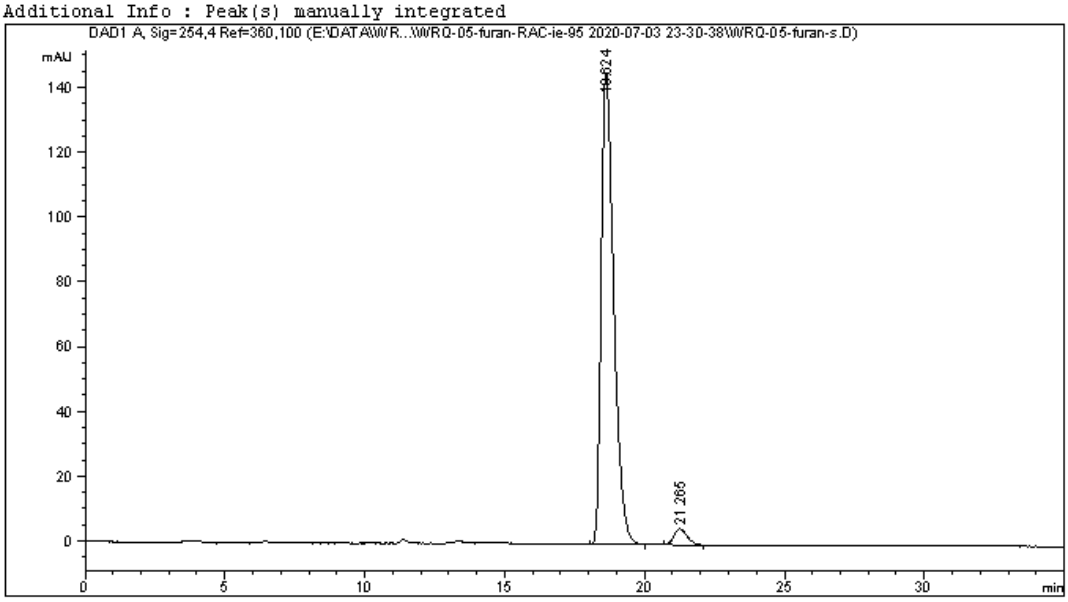

    Signal 1: DAD1 A, Sig=254,4 Ref=360,100

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  18.540 BB    0.4409 5114.61133  167.67819  50.1239
       2  21.015 BB    0.5158 5089.32129  148.88193  49.8761

    Totals :                  1.02039e4   316.56012
```

Data File E:\DATA\WR...furan-s\WRQ-05-furan-RAC-ie-95 2020-07-03 23-30-38\WRQ-05-furan-s.D
Sample Name: WRQ-05-furan-s

```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   1
    Acq. Instrument : 1260                           Location :   64
    Injection Date  : 7/3/2020 11:32:10 PM               Inj :   1
                                                  Inj Volume : 15.000 µl
    Acq. Method     : E:\DATA\WRQ\WRQ-05-furan-s\WRQ-05-furan-RAC-ie-95 2020-07-03 23-30-38\WRQ-4
                      -IE-95-5-DAD-1ML-30MIN.M
    Last changed    : 7/3/2020 11:30:38 PM by SYSTEM
    Analysis Method : E:\DATA\WRQ\WRQ-05-furan-s\WRQ-05-furan-RAC-ie-95 2020-07-03 23-30-38\WRQ-4
                      -IE-95-5-DAD-1ML-30MIN.M (Sequence Method)
    Last changed    : 7/20/2020 8:31:18 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```



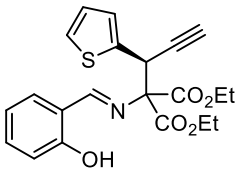

```
    ======================================================================
                           Area Percent Report
    ======================================================================

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

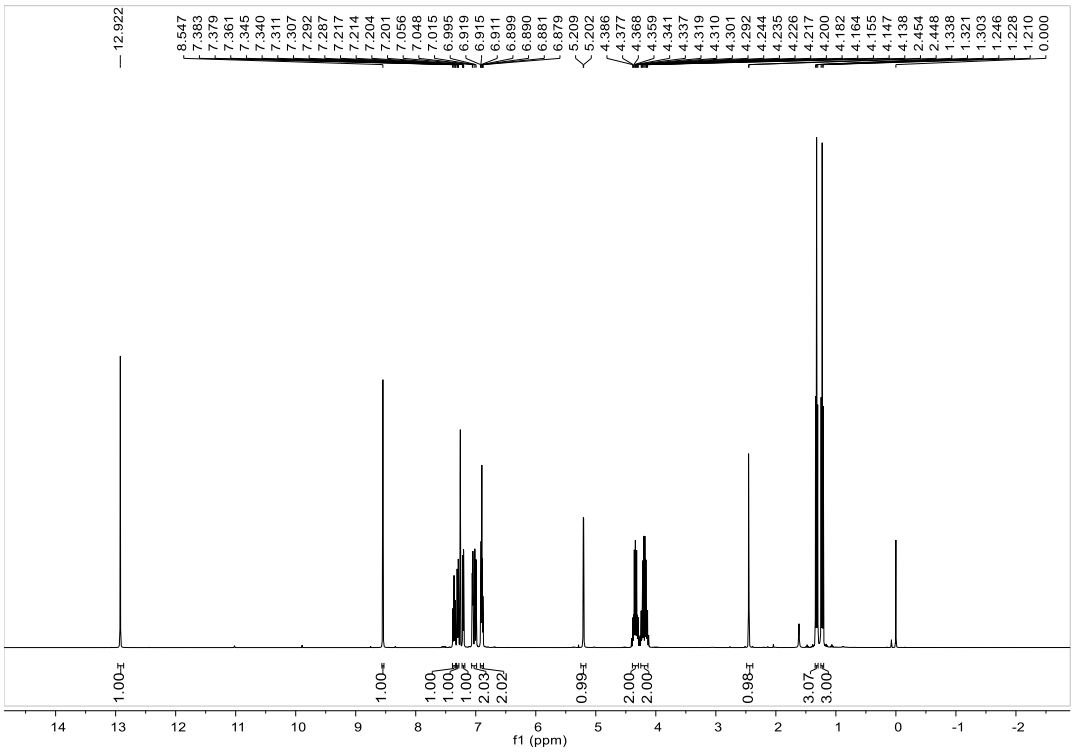

    Signal 1: DAD1 A, Sig=254,4 Ref=360,100

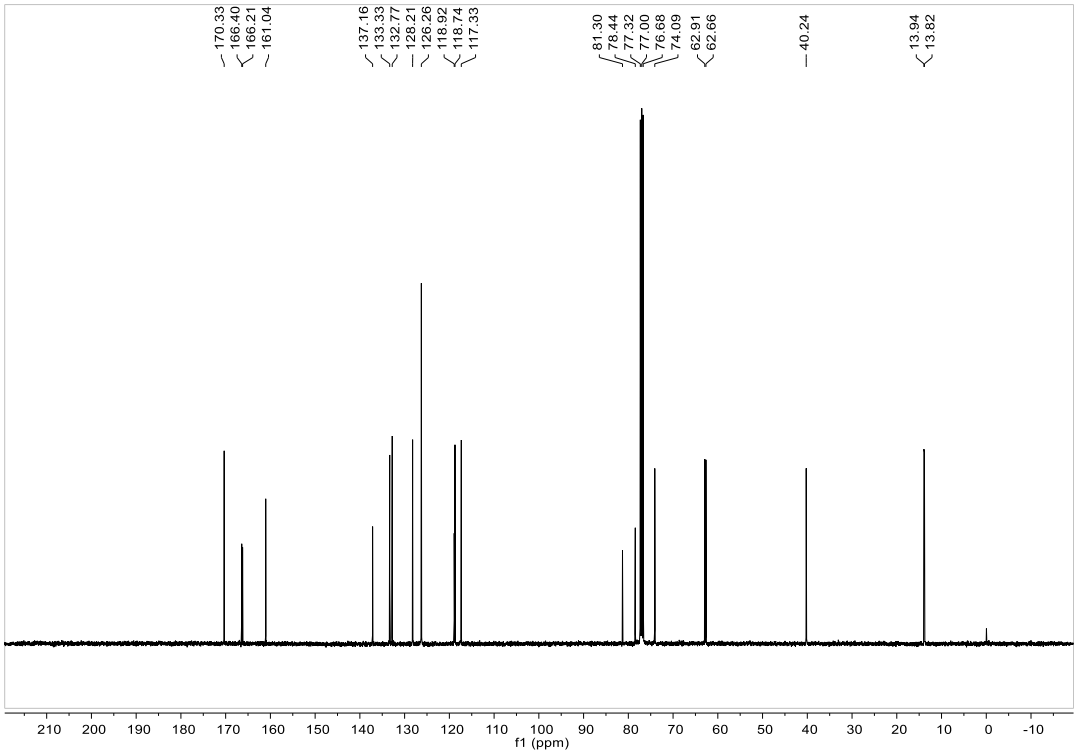
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  18.624 BB    0.4687 4531.56299  145.35938  96.4745
       2  21.265 BB    0.3869  165.59886    5.03218   3.5255

    Totals :                  4697.16185  150.39156
```


S
N
CO2Et
CO2Et
OH

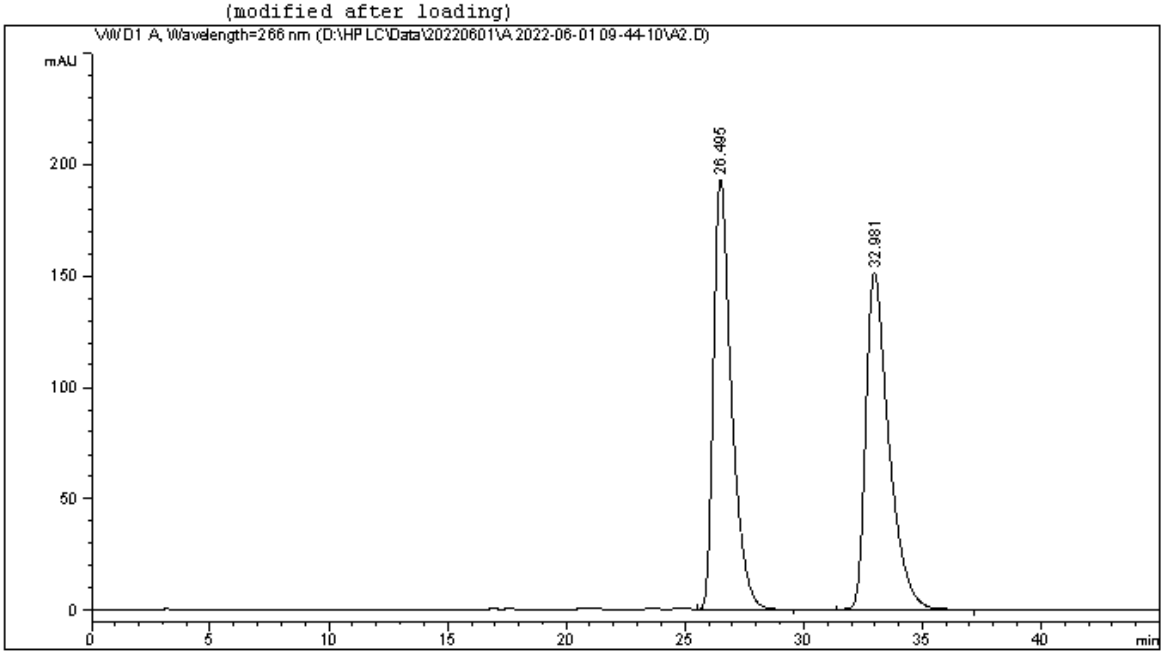
3n


12.922
8.547
7.383
7.379
7.361
7.345
7.340
7.311
7.307
7.292
7.287
7.217
7.214
7.204
7.201
7.056
7.048
7.015
6.995
6.919
6.915
6.911
6.899
6.890
6.881
6.879
5.209
5.202
4.386
4.377
4.368
4.359
4.341
4.337
4.319
4.310
4.301
4.292
4.244
4.235
4.226
4.217
4.200
4.182
4.164
4.155
4.147
4.138
2.454
2.448
1.338
1.321
1.303
1.246
1.228
1.210
0.000
1.00
1.00
1.00
1.00
2.03
2.02
0.99
2.00
2.00
0.98
3.07
3.00
f1 (ppm)


170.33
166.40
166.21
161.04
137.16
133.33
132.77
128.21
126.26
118.92
118.74
117.33
81.30
78.44
77.32
77.00
76.68
74.09
62.91
62.66
40.24
13.94
13.82
f1 (ppm)

Data File D:\HPLC\Data\20220601\A 2022-06-01 09-44-10\A2.D
Sample Name: CGXG-SAIFEN-RAC

```
    ======================================================================
    Acq. Operator   : SYSTEM                          Seq. Line :   3
    Sample Operator : SYSTEM
    Acq. Instrument : 1260                             Location :   81
    Injection Date  : 6/1/2022 11:22:07 AM                 Inj :   1
                                                     Inj Volume : 5.000 µl
    Acq. Method     : D:\HPLC\Data\20220601\A 2022-06-01 09-44-10\ADH(1-6)-95-5-5UL-1ml-30min-
                      220nm.M
    Last changed    : 6/1/2022 12:09:45 PM by SYSTEM
                      (modified after loading)
    Analysis Method : D:\HPLC\Data\20220601\A 2022-06-01 09-44-10\ADH(1-6)-95-5-5UL-1ml-30min-
                      220nm.M (Sequence Method)
    Last changed    : 6/1/2022 12:58:12 PM by SYSTEM
                      (modified after loading)
```




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

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

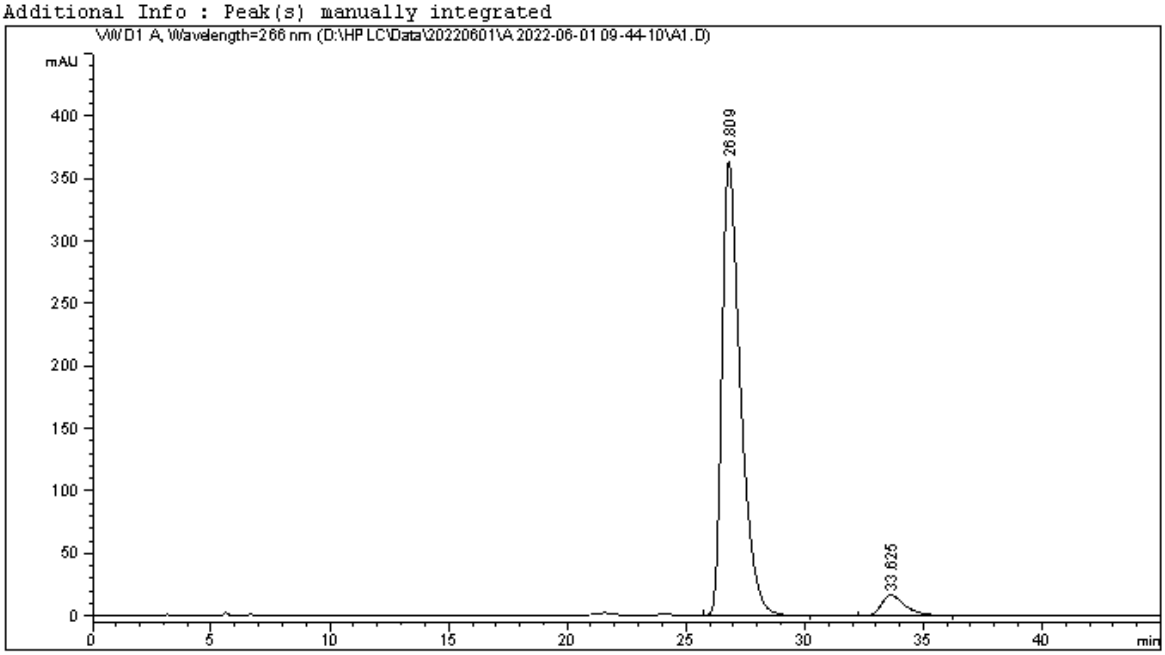

    Signal 1: VWD1 A, Wavelength=266 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  26.495 BB    0.7861 1.01842e4   193.16212  49.8009
       2  32.981 BB    1.0089 1.02656e4   151.42755  50.1991

    Totals :                  2.04498e4   344.58968
```

Data File D:\HPLC\Data\20220601\A 2022-06-01 09-44-10\A1.D
Sample Name: CGXG-SAIFEN-S

```
    ======================================================================
    Acq. Operator   : SYSTEM                          Seq. Line :   2
    Sample Operator : SYSTEM
    Acq. Instrument : 1260                             Location :   82
    Injection Date  : 6/1/2022 10:30:51 AM                 Inj :   1
                                                     Inj Volume : 5.000 µl
    Acq. Method     : D:\HPLC\Data\20220601\A 2022-06-01 09-44-10\ADH(1-6)-95-5-5UL-1ml-30min-
                      220nm.M
    Last changed    : 6/1/2022 11:21:10 AM by SYSTEM
                      (modified after loading)
    Analysis Method : D:\HPLC\Data\20220601\A 2022-06-01 09-44-10\ADH(1-6)-95-5-5UL-1ml-30min-
                      220nm.M (Sequence Method)
    Last changed    : 6/1/2022 12:58:42 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```

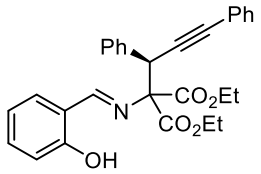



```
    ======================================================================
                            Area Percent Report
    ======================================================================

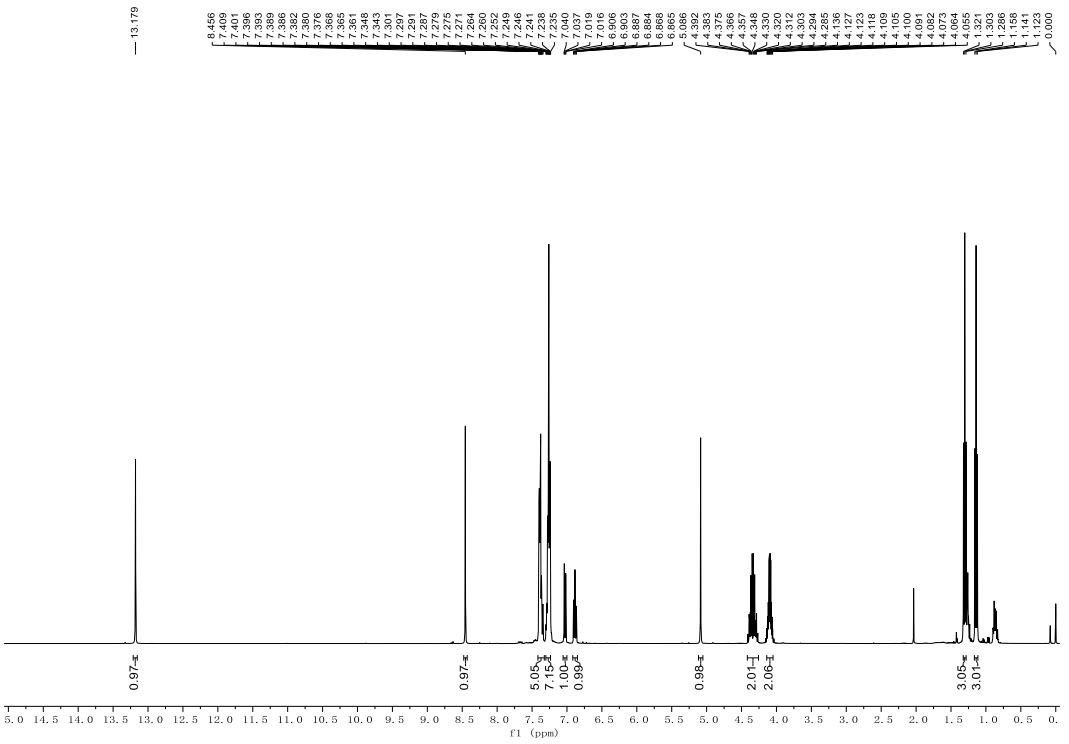
    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Do not use Multiplier & Dilution Factor with ISTDs

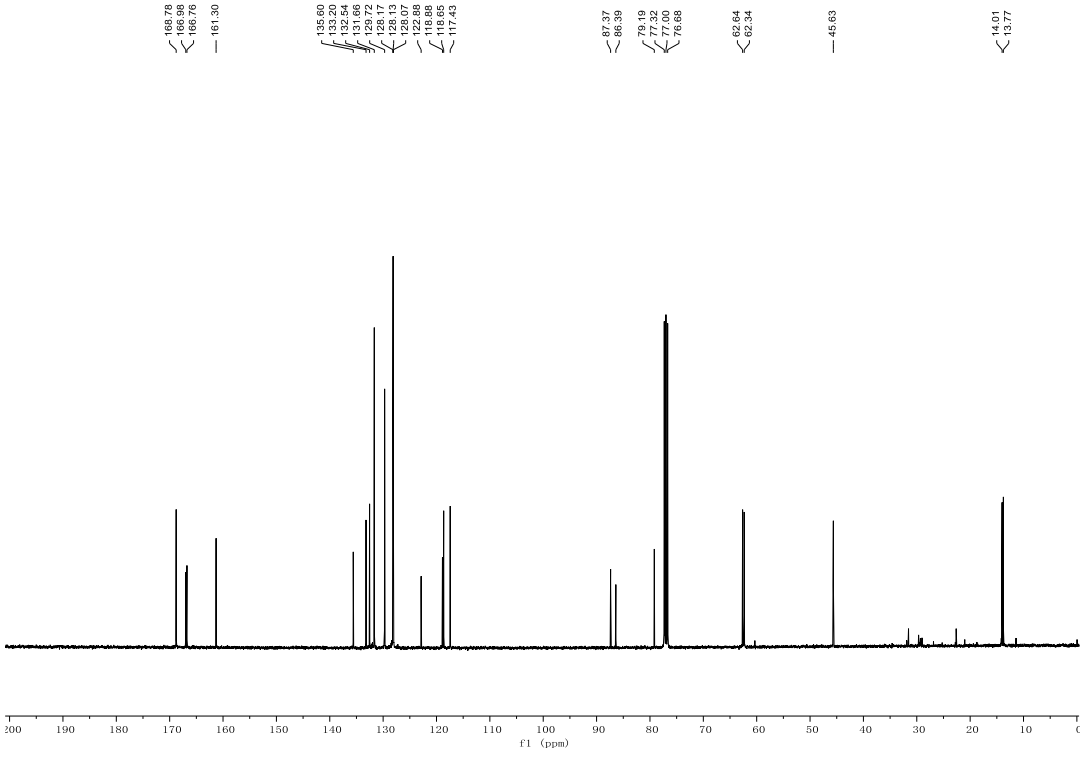

    Signal 1: VWD1 A, Wavelength=266 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  26.809 BB    0.8130 1.96538e4   362.83182  94.7681
       2  33.625 BB    0.9829 1085.04285   16.14867   5.2319
```


Ph
Ph
N
CO2Et
CO2Et
OH

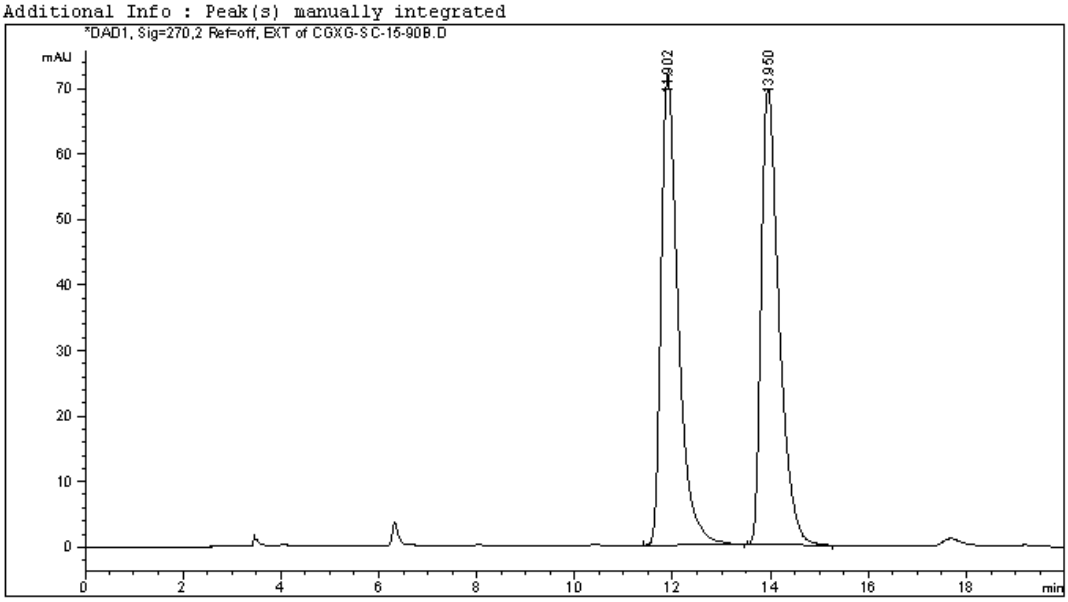
4


13.179
8.456
7.409
7.401
7.396
7.393
7.389
7.386
7.382
7.380
7.376
7.368
7.365
7.361
7.348
7.343
7.301
7.297
7.291
7.287
7.279
7.275
7.271
7.264
7.260
7.252
7.249
7.246
7.241
7.238
7.235
7.040
7.037
7.019
7.016
6.906
6.903
6.887
6.884
6.868
6.865
5.086
4.392
4.383
4.375
4.366
4.357
4.348
4.330
4.320
4.312
4.303
4.294
4.285
4.136
4.127
4.123
4.118
4.109
4.105
4.100
4.091
4.082
4.073
4.064
4.055
1.321
1.303
1.286
1.136
1.141
1.123
0.000
0.97
0.97
5.05
7.15
1.00
0.99
0.98
2.01
2.06
3.05
3.01
f1 (ppm)


168.78
166.98
166.76
161.30
135.60
133.20
132.54
131.66
129.72
128.17
128.13
128.07
122.88
118.88
118.65
117.43
87.37
86.39
79.19
77.32
77.00
76.68
62.64
62.34
45.63
14.01
13.77
f1 (ppm)

Data File E:\DATA\CGXG\CGXG-SC-15-90B 2021-07-04 09-30-36\CGXG-SC-15-90B.D
Sample Name: CGXG-SC-15-90B

```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   1
    Acq. Instrument : 1260                           Location :   31
    Injection Date  : 7/4/2021 9:32:09 AM                 Inj :   1
                                                  Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\CGXG\CGXG-SC-15-90B 2021-07-04 09-30-36\CGXG-2-IE-95-5-254DAD-1ML-
                      30MIN-5UL.M
    Last changed    : 7/4/2021 9:52:00 AM by SYSTEM
                      (modified after loading)
    Analysis Method : E:\DATA\CGXG\CGXG-SC-15-90B 2021-07-04 09-30-36\CGXG-2-IE-95-5-254DAD-1ML-
                      30MIN-5UL.M (Sequence Method)
    Last changed    : 7/17/2021 5:47:38 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

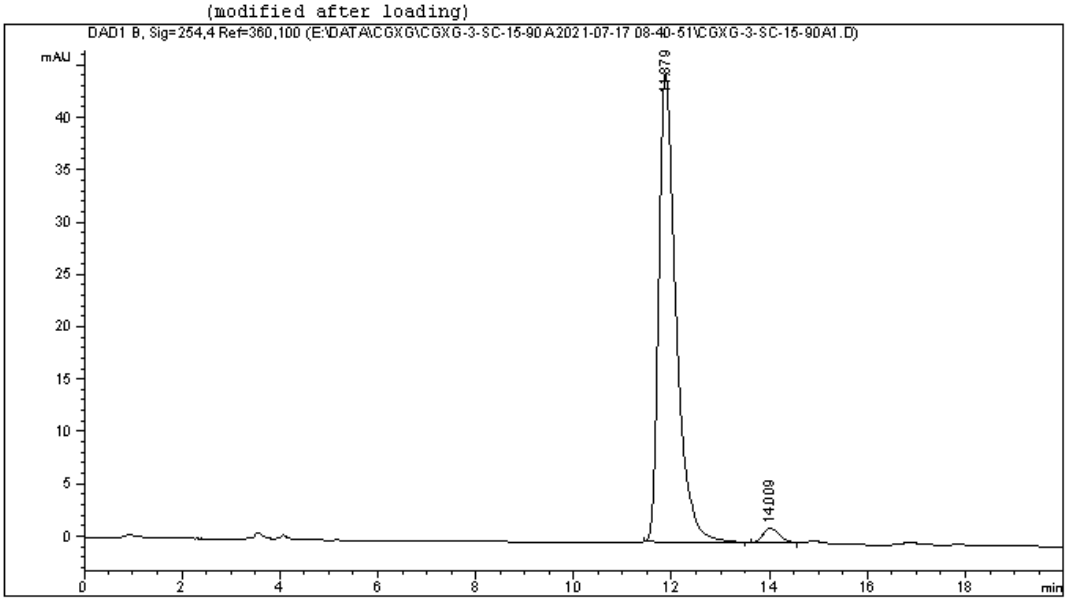
    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Do not use Multiplier & Dilution Factor with ISTDs


    Signal 1: DAD1, Sig=270,2 Ref=off, EXT
     Signal has been modified after loading from rawdata file!

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]        %
    ----|-------|----|-------|----------|----------|--------|
       1  11.902 BB    0.3600 1738.35571   71.93020  49.7479
       2  13.950 BB    0.3757 1755.97742   69.52423  50.2521
```

Data File E:\DATA\CGXG\CGXG-3-SC-15-90A 2021-07-17 08-40-51\CGXG-3-SC-15-90A1.D
Sample Name: CGXG-3-SC-15-90A

```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   2
    Acq. Instrument : 1260                           Location :   71
    Injection Date  : 7/17/2021 8:53:31 AM               Inj :   1
                                                   Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\CGXG\CGXG-3-SC-15-90A 2021-07-17 08-40-51\CGXG-2-IE-95-5-254DAD-1ML
                      -30MIN-5UL.M
    Last changed    : 7/17/2021 9:14:40 AM by SYSTEM
                      (modified after loading)
    Analysis Method : E:\DATA\CGXG\CGXG-3-SC-15-90A 2021-07-17 08-40-51\CGXG-2-IE-95-5-254DAD-1ML
                      -30MIN-5UL.M (Sequence Method)
    Last changed    : 7/17/2021 5:48:26 PM by SYSTEM
                      (modified after loading)
```



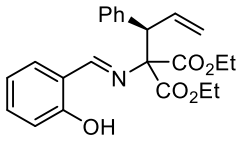

```
    ======================================================================
                           Area Percent Report
    ======================================================================

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

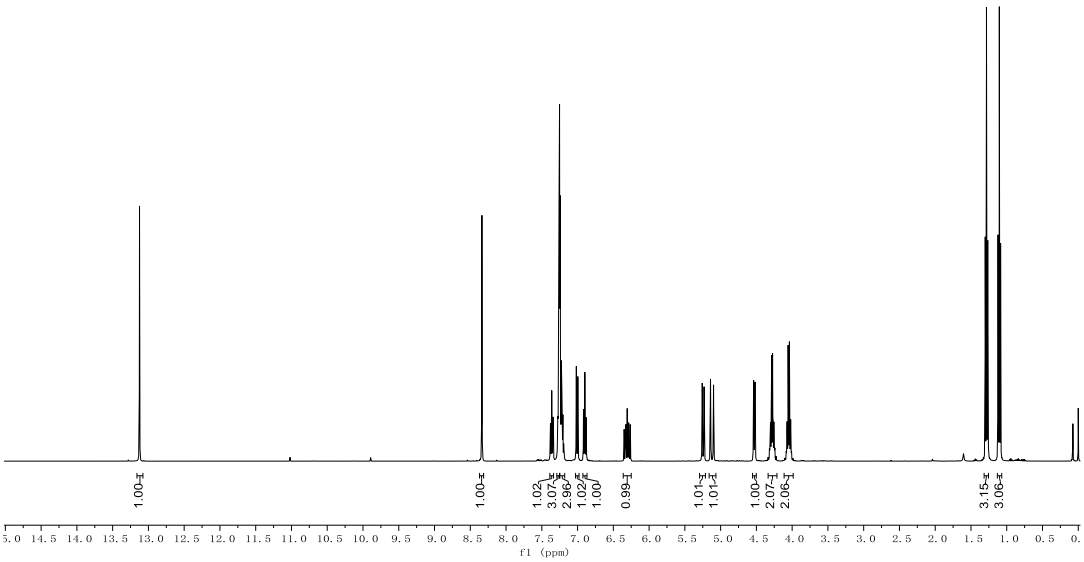

    Signal 1: DAD1 B, Sig=254,4 Ref=360,100

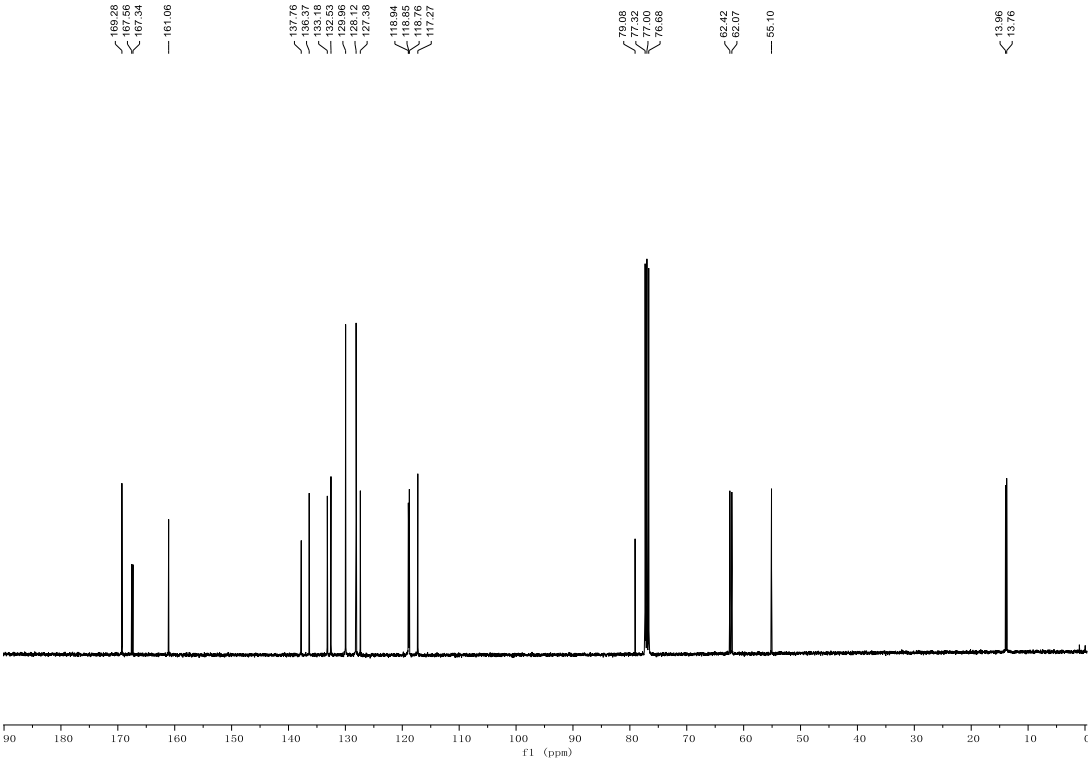
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  11.879 BB    0.3616 1095.14478   44.72906  97.1312
       2  14.009 BB    0.2731   32.34587    1.39722   2.8688

    Totals :                  1127.49064   46.12627
```


Ph
N
CO2Et
CO2Et
OH

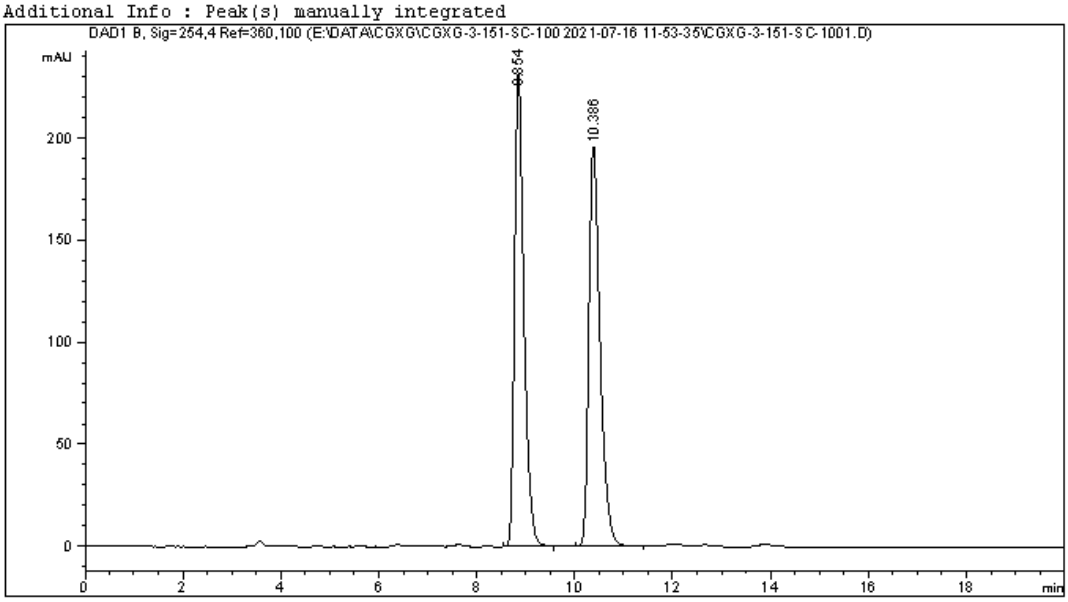
5


1.00
1.00
1.02
3.07
2.96
1.02
1.00
0.99
1.01
1.01
1.00
2.07
2.06
3.15
3.06
f1 (ppm)


169.28
167.56
167.34
161.06
137.76
136.37
133.18
132.53
129.96
128.12
127.38
118.94
118.85
118.76
117.27
79.08
77.32
77.00
76.68
62.42
62.07
55.10
13.96
13.76
f1 (ppm)

Data File E:\DATA\CGXG\CGXG-3-151-SC-100 2021-07-16 11-53-35\CGXG-3-151-SC-1001.D
Sample Name: CGXG-3-151-RAC

```
    ======================================================================
    Acq. Operator   : SYSTEM                          Seq. Line :   2
    Acq. Instrument : 1260                             Location :   71
    Injection Date  : 7/16/2021 12:06:12 PM                 Inj :   1
                                                    Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\CGXG\CGXG-3-151-SC-100 2021-07-16 11-53-35\CGXG-2-IE-95-5-254DAD-
                      1ML-30MIN-5UL.M
    Last changed    : 7/16/2021 12:36:31 PM by SYSTEM
                      (modified after loading)
    Analysis Method : E:\DATA\CGXG\CGXG-3-151-SC-100 2021-07-16 11-53-35\CGXG-2-IE-95-5-254DAD-
                      1ML-30MIN-5UL.M (Sequence Method)
    Last changed    : 7/17/2021 5:39:15 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

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

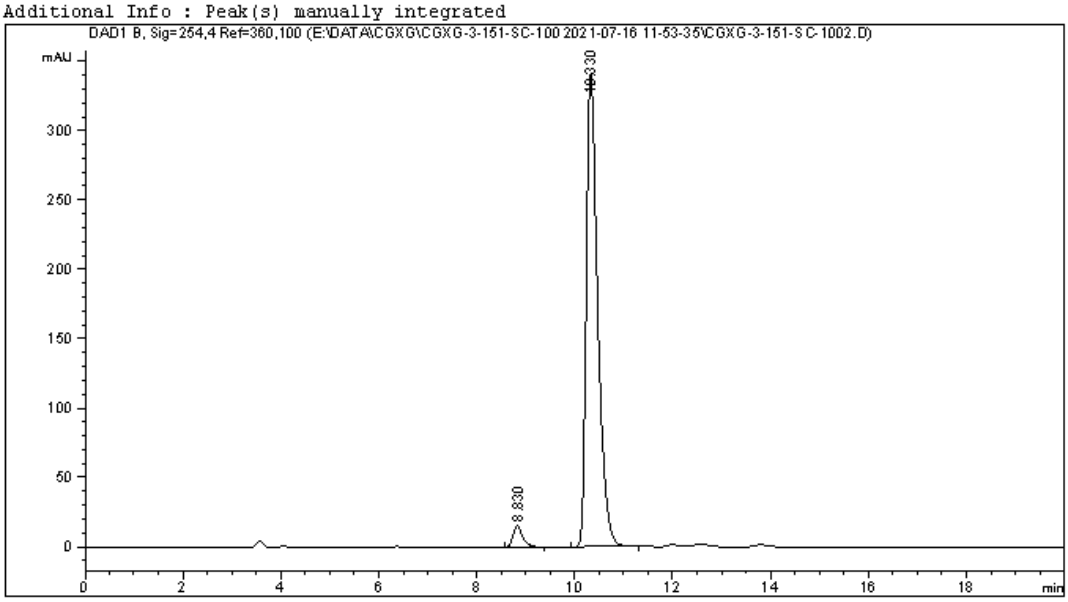

    Signal 1: DAD1 B, Sig=254,4 Ref=360,100

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   8.854 BB    0.1989 3055.35693  231.66972  49.6508
       2  10.386 BB    0.2365 3098.33350  195.87973  50.3492

    Totals :                  6153.69043  427.54945
```

Data File E:\DATA\CGXG\CGXG-3-151-SC-100 2021-07-16 11-53-35\CGXG-3-151-SC-1002.D
Sample Name: CGXG-3-151-S

```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   3
    Acq. Instrument : 1260                           Location :   72
    Injection Date  : 7/16/2021 12:38:30 PM              Inj :   1
                                                   Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\CGXG\CGXG-3-151-SC-100 2021-07-16 11-53-35\CGXG-2-IE-95-5-254DAD-
                      1ML-30MIN-5UL.M
    Last changed    : 7/16/2021 1:01:45 PM by SYSTEM
                      (modified after loading)
    Analysis Method : E:\DATA\CGXG\CGXG-3-151-SC-100 2021-07-16 11-53-35\CGXG-2-IE-95-5-254DAD-
                      1ML-30MIN-5UL.M (Sequence Method)
    Last changed    : 7/17/2021 5:39:40 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```



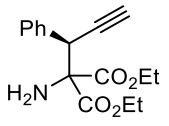

```
    ======================================================================
                            Area Percent Report
    ======================================================================

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


    Signal 1: DAD1 B, Sig=254,4 Ref=360,100

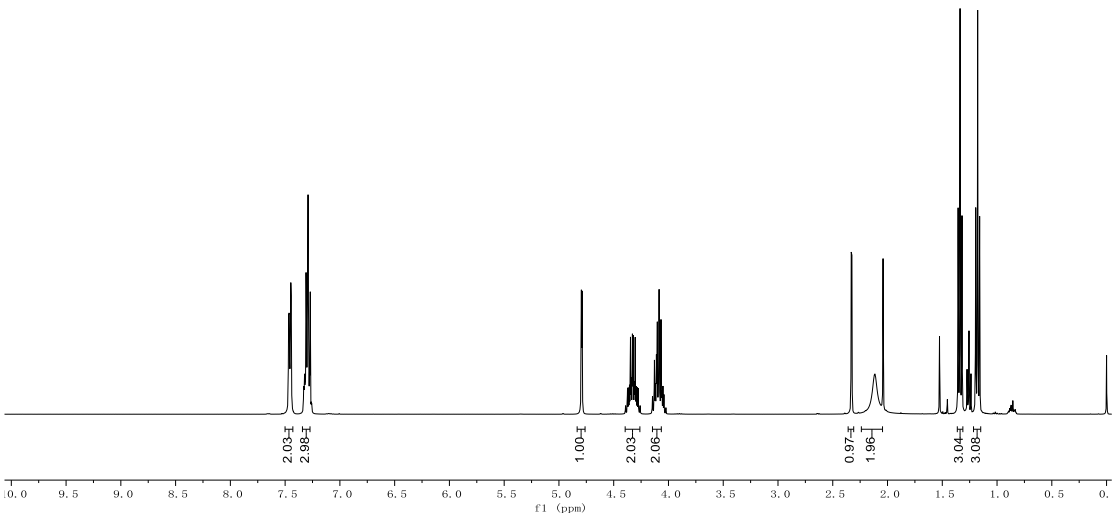
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1   8.830 BB    0.1970  198.56895   15.14498   3.4739
       2  10.330 BB    0.2438 5517.49658  341.02316  96.5261

    Totals :                  5716.06554  356.16814
```

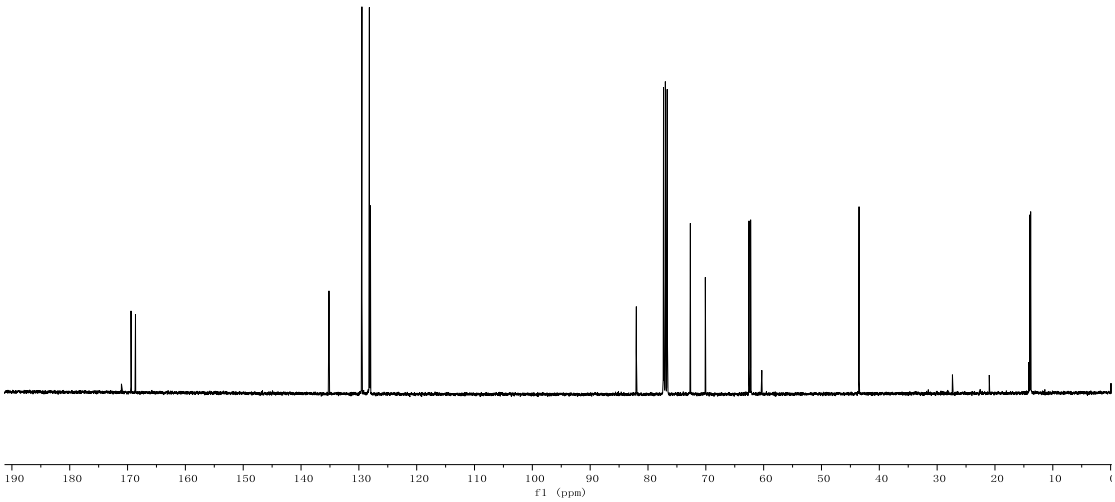

Ph
H_2N
CO_2Et
CO_2Et

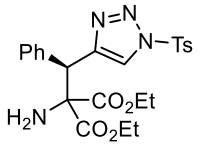
6


7.466
7.463
7.459
7.453
7.448
7.444
7.436
7.331
7.326
7.323
7.318
7.309
7.305
7.296
7.291
7.286
7.283
7.277
7.271
7.262
4.795
4.789
4.392
4.374
4.365
4.356
4.347
4.339
4.329
4.322
4.312
4.304
4.295
4.286
4.277
4.269
4.146
4.126
4.120
4.110
4.104
4.102
4.092
4.088
4.083
4.077
4.068
4.059
4.051
4.042
4.024
2.331
2.295
2.116
1.356
1.338
1.320
1.195
1.177
1.159
0.000

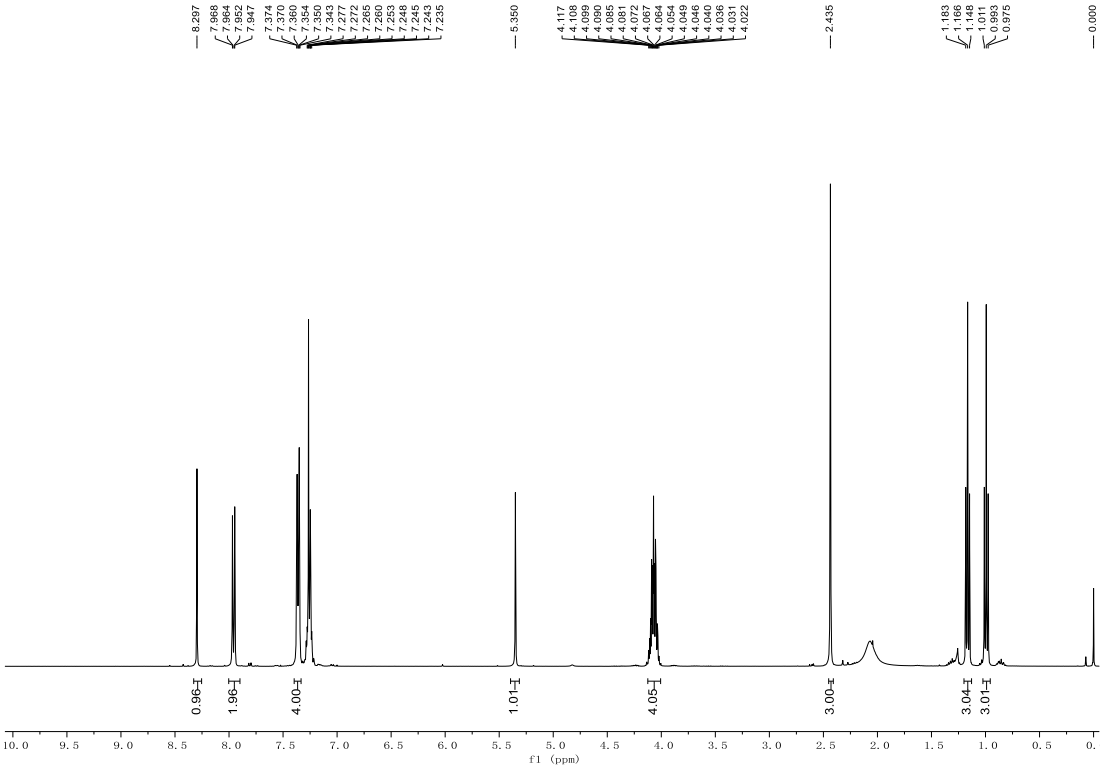

2.03
2.98
1.00
2.03
2.06
0.97
1.96
3.04
3.08
f1 (ppm)

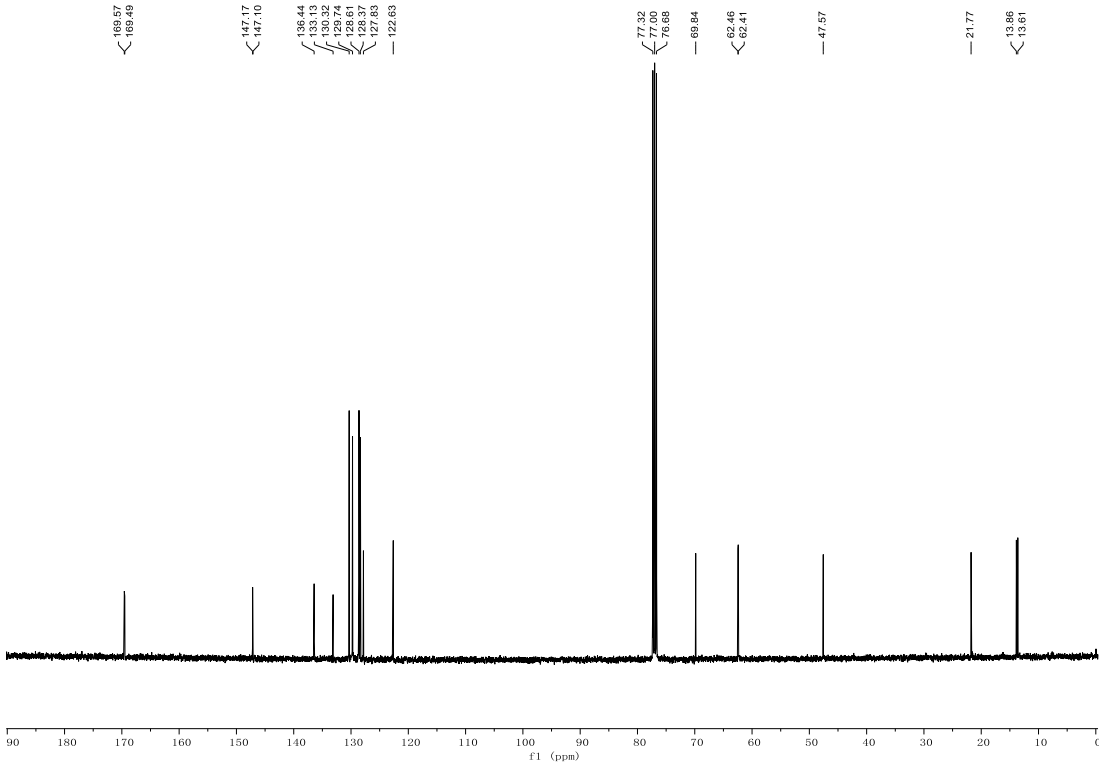

169.40
168.63
135.17
129.47
128.16
128.00
82.02
77.32
77.00
76.68
72.68
70.09
62.53
62.25
43.50
13.96
13.83


f1 (ppm)


N=N
N–Ts
Ph
CO2Et
H2N
CO2Et

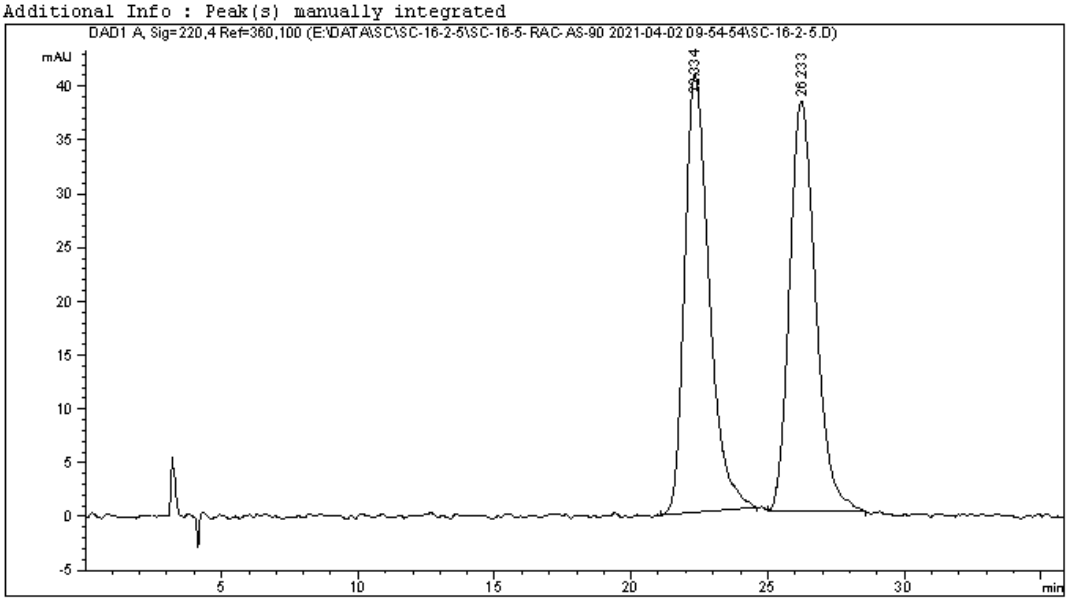
7


8.297
7.968
7.964
7.952
7.947
7.374
7.370
7.360
7.354
7.353
7.343
7.277
7.272
7.265
7.260
7.253
7.246
7.245
7.243
7.235
5.350
4.117
4.108
4.099
4.090
4.085
4.081
4.072
4.067
4.064
4.054
4.049
4.046
4.040
4.036
4.031
4.022
2.435
1.183
1.166
1.148
1.011
0.993
0.975
0.000
0.96
1.96
4.00
1.01
4.05
3.00
3.04
3.01
f1 (ppm)


169.57
169.49
147.17
147.10
136.44
133.13
130.32
129.74
128.61
128.37
127.83
122.63
77.32
77.00
76.68
69.84
62.46
62.41
47.57
21.77
13.86
13.61
f1 (ppm)

Data File E:\DATA\SC\SC-16-2-5\SC-16-5-RAC-AS-90 2021-04-02 09-54-54\SC-16-2-5.D
Sample Name: SC-16-5-RAC-AS-90

```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   1
    Acq. Instrument : 1260                          Location :   81
    Injection Date  : 4/2/2021 9:56:17 AM               Inj :   1
                                                  Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\SC\SC-16-2-5\SC-16-5-RAC-AS-90 2021-04-02 09-54-54\SC-1-ASH-90-10-
                      DAD-1ML.M
    Last changed    : 4/2/2021 9:54:54 AM by SYSTEM
    Analysis Method : E:\DATA\SC\SC-16-2-5\SC-16-5-RAC-AS-90 2021-04-02 09-54-54\SC-1-ASH-90-10-
                      DAD-1ML.M (Sequence Method)
    Last changed    : 6/27/2021 10:34:22 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

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

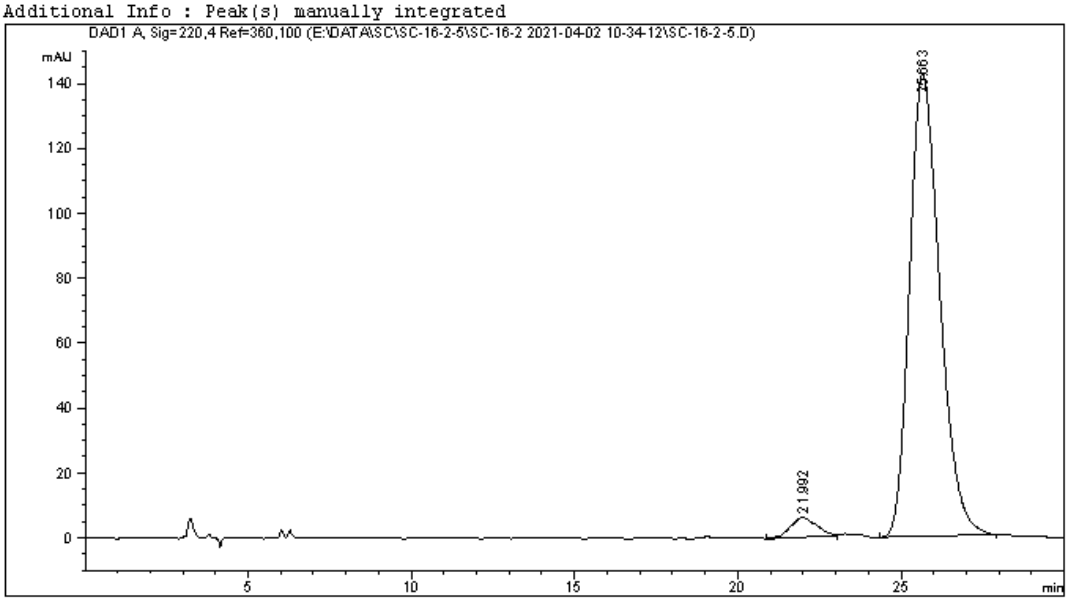

    Signal 1: DAD1 A, Sig=220,4 Ref=360,100

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  22.334 BB    0.8360 2548.25391   40.83508  50.1935
       2  26.233 BB    0.8144 2528.60669   38.09251  49.8065

    Totals :                  5076.86060   78.92759
```

Data File E:\DATA\SC\SC-16-2-5\SC-16-2 2021-04-02 10-34-12\SC-16-2-5.D
Sample Name: SC-16-2

```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   1
    Acq. Instrument : 1260                           Location :   82
    Injection Date  : 4/2/2021 10:35:35 AM               Inj :   1
                                                   Inj Volume : 5.000 µl
    Acq. Method     : E:\DATA\SC\SC-16-2-5\SC-16-2 2021-04-02 10-34-12\SC-1-ASH-90-10-DAD-1ML.M
    Last changed    : 4/2/2021 10:34:39 AM by SYSTEM
                      (modified after loading)
    Analysis Method : E:\DATA\SC\SC-16-2-5\SC-16-2 2021-04-02 10-34-12\SC-1-ASH-90-10-DAD-1ML.M (
                      Sequence Method)
    Last changed    : 6/27/2021 10:33:49 PM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```



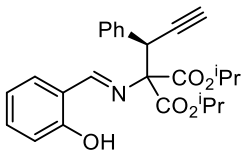

```
    ======================================================================
                          Area Percent Report
    ======================================================================

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

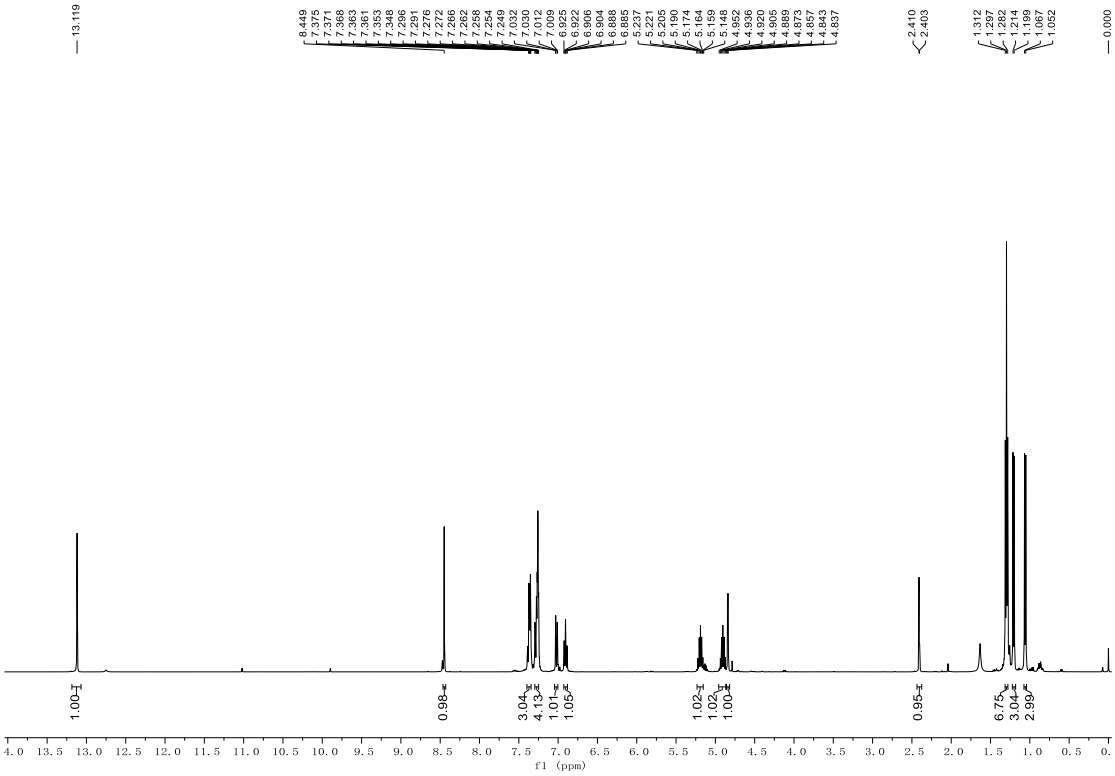

    Signal 1: DAD1 A, Sig=220,4 Ref=360,100

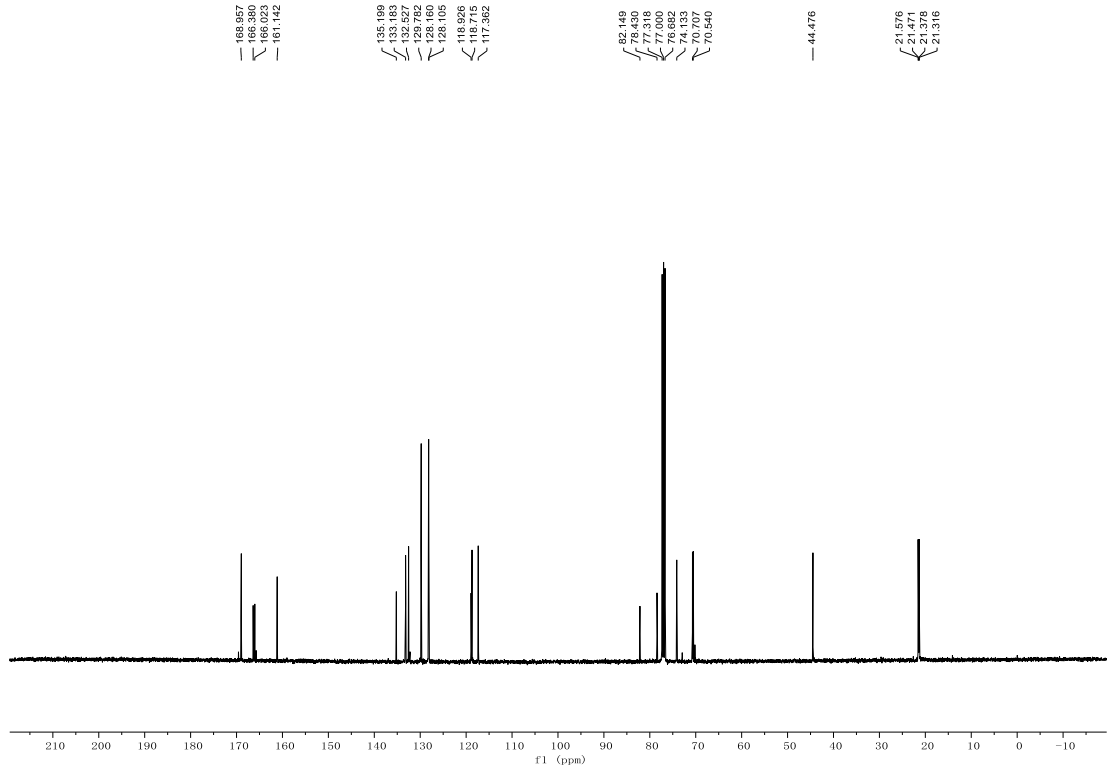
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  21.992 BB    0.6461  331.77695    6.02809   3.4881
       2  25.663 BB    0.9405 9179.81152  142.70569  96.5119

    Totals :                  9511.58847  148.73378
```


Ph
N
$CO_2{}^iPr$
$CO_2{}^iPr$
OH

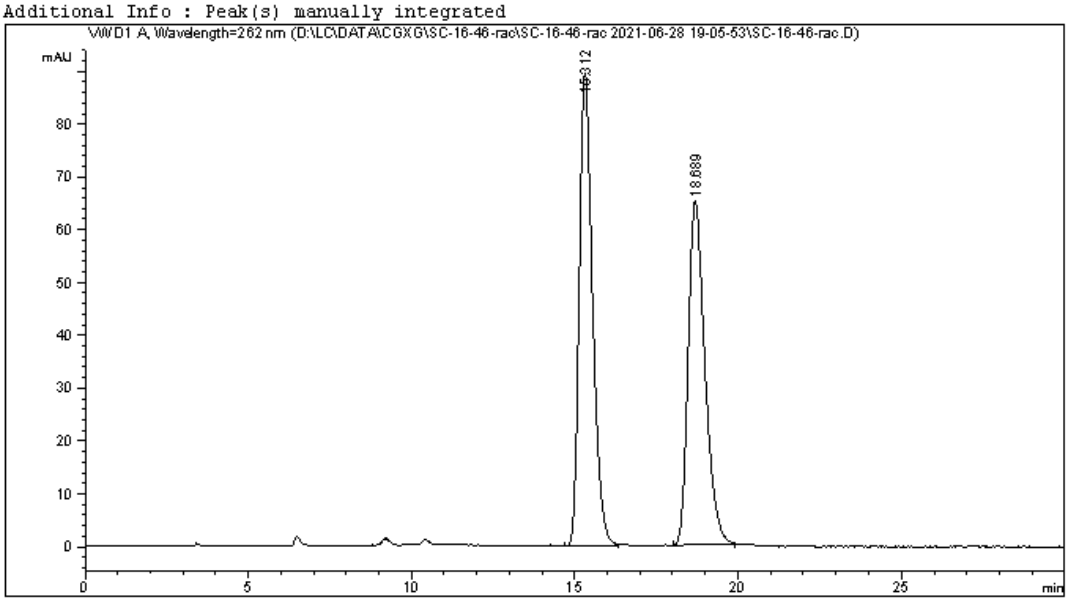
3a'


13.119
8.449
7.375
7.371
7.368
7.363
7.361
7.353
7.348
7.296
7.291
7.276
7.272
7.266
7.262
7.258
7.254
7.249
7.032
7.030
7.012
7.009
6.925
6.922
6.906
6.904
6.888
6.885
5.237
5.221
5.205
5.190
5.174
5.164
5.159
5.148
4.952
4.936
4.920
4.905
4.889
4.873
4.857
4.843
4.837
2.410
2.403
1.312
1.297
1.282
1.214
1.199
1.067
1.052
0.000
1.00
0.98
3.04
4.13
1.01
1.05
1.02
1.02
1.00
0.95
6.75
3.04
2.99
f1 (ppm)


168.957
168.380
166.023
161.142
135.199
133.183
132.527
129.782
128.160
128.105
118.926
118.715
117.362
82.149
78.430
77.318
77.000
76.682
74.133
70.707
70.540
44.476
21.576
21.471
21.378
21.316
f1 (ppm)

Data File D:\LC\DATA\CGXG\SC-16-46-rac\SC-16-46-rac 2021-06-28 19-05-53\SC-16-46-rac.D
Sample Name: SC-16-46-rac

```
    ======================================================================
    Acq. Operator   : 系统                          Seq. Line :   1
    Sample Operator : 系统
    Acq. Instrument : 1200                           Location :   61
    Injection Date  : 6/28/2021 7:06:37 PM              Inj :   1
                                                    Inj Volume : 5.000 µl
    Acq. Method     : D:\LC\DATA\CGXG\SC-16-46-rac\SC-16-46-rac 2021-06-28 19-05-53\CGXG-ADH-95-5
                      -262NM-1ML-30MIKN-5UL.M
    Last changed    : 6/28/2021 5:23:03 PM by 系统
    Analysis Method : D:\LC\DATA\CGXG\SC-16-46-rac\SC-16-46-rac 2021-06-28 19-05-53\CGXG-ADH-95-5
                      -262NM-1ML-30MIKN-5UL.M (Sequence Method)
    Last changed    : 7/17/2021 5:35:14 PM by 系统
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

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

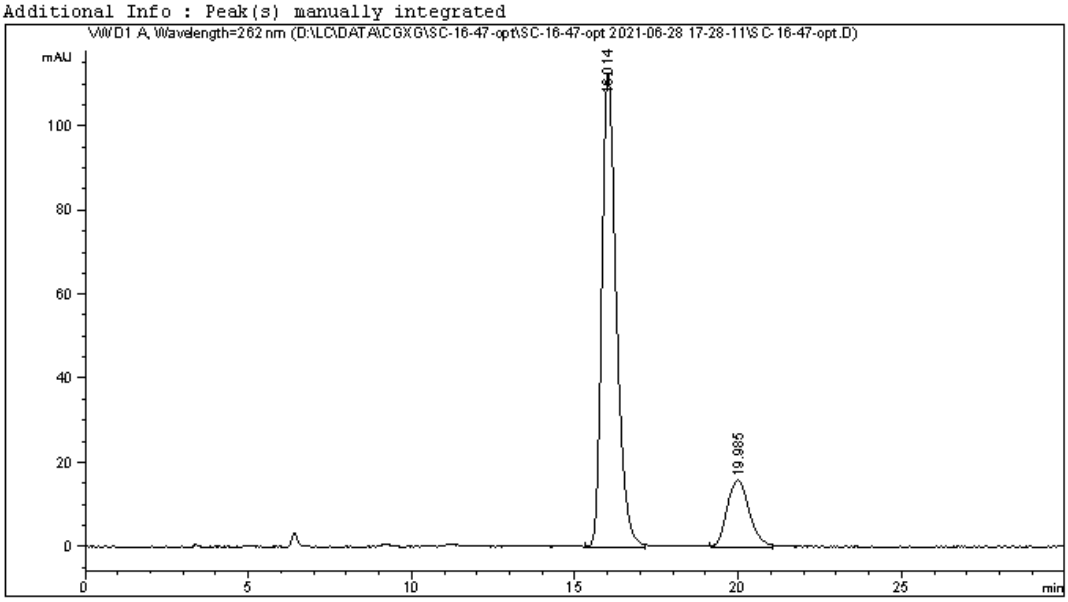

    Signal 1: VWD1 A, Wavelength=262 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  15.312 VB    0.4287 2539.73535   89.47033  52.2218
       2  18.689 BB    0.5356 2323.62671   65.12861  47.7782

    Totals :                  4863.36206  154.59894
```

Data File D:\LC\DATA\CGXG\SC-16-47-opt\SC-16-47-opt 2021-06-28 17-28-11\SC-16-47-opt.D
Sample Name: SC-16-47-opt

```
    ======================================================================
    Acq. Operator   : 系统                          Seq. Line :   1
    Sample Operator : 系统
    Acq. Instrument : 1200                            Location :   61
    Injection Date  : 6/28/2021 5:28:54 PM                 Inj :   1
                                                    Inj Volume : 5.000 µl
    Acq. Method     : D:\LC\DATA\CGXG\SC-16-47-opt\SC-16-47-opt 2021-06-28 17-28-11\CGXG-ADH-95-5
                      -262NM-1ML-30MIKN-5UL.M
    Last changed    : 6/28/2021 5:23:03 PM by 系统
    Analysis Method : D:\LC\DATA\CGXG\SC-16-47-opt\SC-16-47-opt 2021-06-28 17-28-11\CGXG-ADH-95-5
                      -262NM-1ML-30MIKN-5UL.M (Sequence Method)
    Last changed    : 7/17/2021 5:34:54 PM by 系统
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

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


    Signal 1: VWD1 A, Wavelength=262 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  16.014 BV    0.4669 3480.48633  112.61360  82.3388
       2  19.985 VV    0.5955  746.54651   15.82673  17.6612

    Totals :                  4227.03284  128.44033
```