Supporting Information

Palladium Catalyzed Regioselective Elimination-Hydrocarbonylation of Propargylic Alcohols

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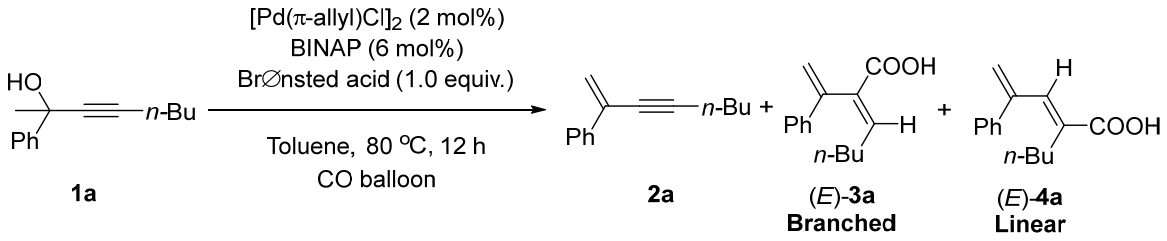
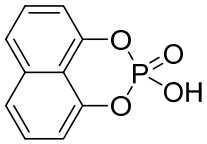
General Information. NMR spectra were taken with an Agilent-400 spectrometer (400 MHz for ^{1}H NMR, 100 MHz for ^{13}C NMR) in $CDCl_3$, DMSO-*d*$_6$, C_6D_6 or $(CD_3)_2CO$. All ^{1}H NMR experiments were measured with tetramethylsilane (0 ppm) in $CDCl_3$ or the signal of residual C_6H_6 (7.16 ppm) in C_6D_6 or the signal of residual DMSO (2.50 ppm) in DMSO-*d*$_6$ as the internal reference; ^{13}C NMR experiments were measured in relative to the signal of $CDCl_3$ (77.0 ppm) or the signal of DMSO-*d*$_6$ (39.52 ppm) or the signal of $(CD_3)_2CO$ (29.84 ppm). All reactions were carried out in flame-dried Schlenk tubes. $[(\pi\text{-allyl})PdCl]_2$ was purchased from J&K Chemicals; BINAP was purchased from Shanghai 9dingchem Co., Ltd.; $(PhO)_2POOH$ was purchased from Energy Chemical and purified through stirring with 1 M HCl, extracting with dichloromethane, and removing solvents under vacuum or purchased from Shanghai aladdin Biochemical Technology Co., Ltd. without purification; ^{n}BuLi was purchased from Energy Chemical and Infinity Scientific (Beijing) Co. Ltd.. Toluene was dried over sodium wire with benzophenone as the indicator and distilled freshly before use. The reaction should be conducted in a hood working efficiently due to the toxicity of CO gas. All the temperatures are referred to the oil baths used. Recoveries of substrates were determined by ^{1}H NMR analysis of the crude product using dibromomethane or 1,3,5-trimethylbenzene as the internal standard. Column chromatography was conducted on 300-400 mesh silica gel purchased from Huanghai chemicals or Biotage Isolera One flash chromatography purification system using flash silica gel column (12 g) purchased from Santai Tech. Inc.. Petroleum ether (b.p. 60-90 ^{o}C) purchased from Shanghai Titan Scientific Co., Ltd. was used for chromatography. The starting propargylic alcohols were synthesized according to the reported procedures. [1]

Table S1 Screening of Brønsted acids [a]



Entry	Brønsted acid	NMR yield[b] (%)			Recovery[b] of **1a** (%)
		2a	(*E*)-**3a**	(*E*)-**4a**	
1	$(PhO)_2POOH$	0	79	1	1
2	$(n\text{-}BuO)_2POOH$	3	0	9	13
3	Ph_2POOH	1	2	5	0
4	Acid 1	21	26	7	5
5	H_3PO_3	55	0	0	0
6	HPO_3	1	0	0	99
7	CH_3COOH	0	0	0	37
8	CF_3COOH	24	0	0	0



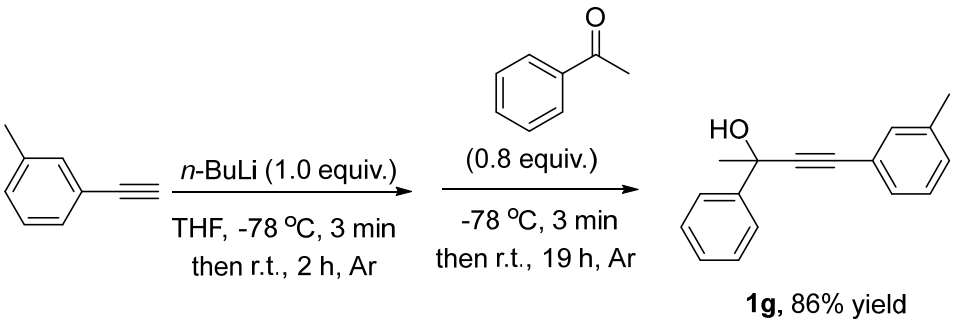
Acid 1

[a] **Reaction conditions**: **1a** (1.0 mmol), $[Pd(\pi\text{-allyl})Cl]_2$ (2 mol%), BINAP (6 mol%), and Brønsted acid (1.0 equiv.) in toluene (5 mL) at 80 °C under 1 atm. of CO unless otherwise noted. [b] Determined by ^1H NMR analysis of the crude product using dibromomethane as the internal standard.

Experimental details and analytical data

1. Synthesis of propargylic alcohols

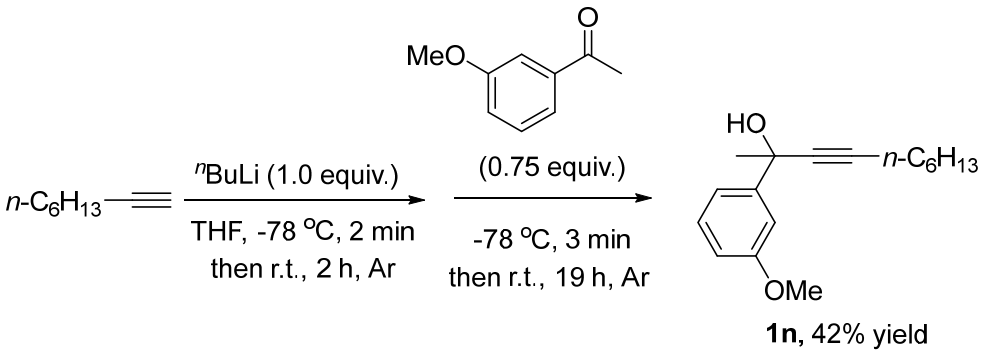
(1) Preparation of 4-(3-Methylphenyl)-2-phenylbut-3-yn-2-ol (1g) (Yy-2-078)



Typical Procedure I: [1] To an oven-dried flask (100 mL) were added (3-methylphenyl)acetylene (2.65 mL, d = 0.900 g/mL, 2.3850 g, 20 mmol) and THF (50 mL) under argon. Then a solution of *n*BuLi (2.5 M in hexane, 8 mL, 20 mmol) was added dropwise at -78 °C within 3 min under argon. Then the cooling bath was removed and the mixture was stirred at room temperature for 2 h. Acetophenone (1.90 mL, d =1.03 g/mL, 1.9570 g, 16 mmol) was then added dropwise at -78 °C within 3 min. The resulting mixture was warmed up to room temperature, stirred for 19 h, and quenched with a saturated aqueous solution of NH_4Cl (20 mL). After extraction with ethyl acetate (20 mL x 3), the organic layer was dried over anhydrous Na_2SO_4. After filtration and concentration under reduced pressure, the crude product was purified by column chromatography on silica gel to afford **1g** (3.2544 g, 86%) (eluent: petroleum ether /dichloromethane/ethyl ether = 60/1/1) as a yellow solid: m.p. 47.5 - 48.0 °C (petroleum ether/ethyl acetate); **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.78-7.67 (m, 2 H, Ar-H), 7.43-7.34 (m, 2 H, Ar-H), 7.34-7.25 (m, 3 H, Ar-H), 7.20 (t, *J* = 7.6 Hz, 1 H, Ar-H), 7.13 (d, *J* = 7.2 Hz, 1 H, Ar-H), 2.53 (s, 1 H, OH), 2.32 (s, 3 H, CH_3), 1.86 (s, 3 H, CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 145.7, 138.0, 132.3, 129.3, 128.8, 128.3, 128.2, 127.7, 125.0, 122.3, 92.1, 85.1, 70.4, 33.3, 21.2; **IR** (neat): *v* = 3292 (br), 2986, 2926, 1599, 1580, 1484, 1365, 1221, 1087, 1051 cm^{-1}; **MS** (70 eV, EI) *m/z* (%): 237 (M^++1, 2.28) 236 (M^+, 14.21), 221 (100); **Anal.** calcd for $C_{17}H_{16}O$: C 86.40, H 6.82; Found: C 86.44, H 6.73.

The following compounds were prepared according to **Typical Procedure I**.

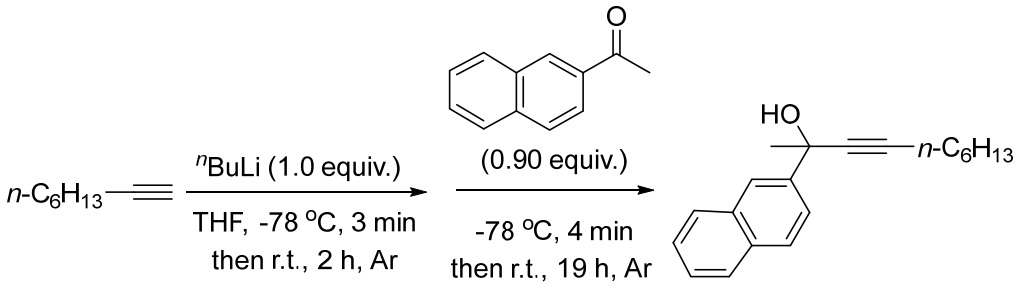
(2) Preparation of 2-(3-methoxyphenyl)dec-3-yn-2-ol (1n) (Yy-2-070)



1n, 42% yield

The reaction of 1-octyne (3.0 mL, d = 0.746 g/ mL, 2.2380 g, 20 mmol)/THF (50 mL), ^{n}BuLi (2.5 M in hexane, 8 mL, 20 mmol), and 3-methoxyacetophenone (2.1 mL, d = 1.094 g/mL, 2.2974 g, 15 mmol) afforded **1n** (1.6462 g, 42%) (eluent: petroleum ether/ethyl ether/CH_2Cl_2 = 20/1/1) as a colorless oil: **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.31-7.17 (m, 3 H, Ar-H), 6.82 (dt, J_1 = 7.2 Hz, J_2 = 2.1 Hz, 1 H, Ar-H), 3.82 (s, 3 H, OCH_3), 2.40-2.30 (m, 1 H, OH), 2.27 (t, J = 7.2 Hz, 2 H, CH_2), 1.73 (s, 3 H, CH_3), 1.60-1.48 (m, 2 H, CH_2), 1.46-1.36 (m, 2 H, CH_2), 1.35-1.24 (m, 4 H, CH_2 x 2), 0.89 (t, J = 6.8 Hz, 3 H, CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 159.4, 148.0, 129.2, 117.4, 112.9, 110.8, 85.7, 83.6, 70.0, 55.2, 33.5, 31.3, 28.57, 28.55, 22.5, 18.7, 14.0; **IR** (neat): ν = 3425 (br), 2929, 2858, 2242, 1600, 1485, 1432, 1257, 1159, 1043 cm^{-1}; **MS** (70 eV, EI) *m/z* (%): 261 (M^++1, 3.89), 260 (M^+, 21.47), 245 (100); **HRMS** calcd for $C_{17}H_{24}O_2$ [M^+]: 260.1776; Found: 260.1778.

(3) Preparation of 2-(2-naphthyl)dec-3-yn-2-ol (1o) (Yy-2-050)

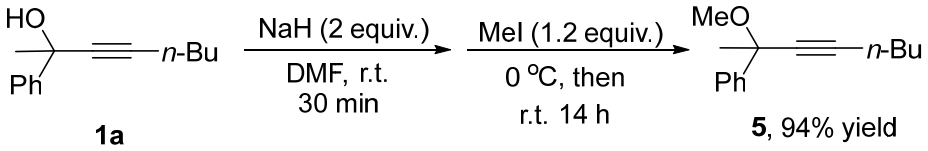


1o, 19% yield

The reaction of 1-octyne (3.0 mL, d = 0.746 g/mL, 2.2380 g, 20.0 mmol)/THF (50 mL), ^{n}BuLi (2.5 M in hexane, 8 mL, 20.0 mmol), and 2-acetonaphthone (3.1263 g, 18 mmol)/THF (20 mL) afforded **1o** (0.9534 g, 19%) (eluent: petroleum ether/ethyl

acetate = 100/1) as a yellow oil: **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.11 (s, 1 H, Ar-H), 7.90-7.77 (m, 3 H, Ar-H), 7.74 (dd, J_1 = 8.4 Hz, J_2 = 1.6 Hz, 1 H, Ar-H), 7.53-7.38 (m, 2 H, Ar-H), 2.48 (s, 1 H, OH), 2.29 (t, J = 7.0 Hz, 2 H, CH_2), 1.82 (s, 3 H, CH_3), 1.65-1.50 (m, 2 H, CH_2), 1.50-1.38 (m, 2 H, CH_2), 1.38-1.24 (m, 4 H, CH_2 x 2), 0.89 (t, J = 6.8 Hz, 3 H, CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 143.5, 133.0, 132.8, 128.3, 128.0, 127.5, 126.1, 125.9, 123.7, 123.3, 85.9, 83.8, 70.1, 33.4, 31.3, 28.59, 28.56, 22.5, 18.8, 14.0; **IR** (neat): v = 3378 (br), 3055, 2926, 2858, 2240, 1457, 1359, 1196, 1084 cm^{-1}; **MS** (70 eV, EI) *m/z* (%): 280 (M^+, 1.46), 105 (100); **HRMS** calcd for $C_{20}H_{24}O$ [M^+]: 280.1827; Found: 280.1830.

2. Preparation of 2-phenyloct-3-yn-2-yl methyl ether (5) (Yy-2-121)

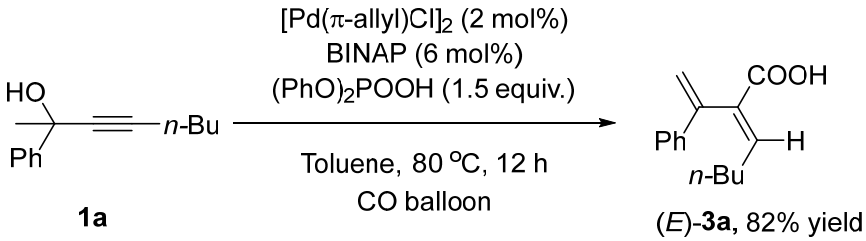


To a flame-dried Schlenk tube were added NaH (400 mg, 10 mmol) in a glove box, followed by the addition of 2 mL of DMF. Then **1a** (1.0074 g, 5 mmol) in DMF (3 mL) was added slowly within 5 min. After being stirred at room temperature for 30 min, the Schlenk tube was cooled to 0 °C with a water/ice bath for 5 min before MeI (0.37 mL, d = 2.28 g/mL, 843.6 mg, 6 mmol) was added. The resulting mixture was stirred at room temperature for 14 h and then the reaction was cooled to 0 °C with a water/ice bath for 5 min before quenched with a saturated aqueous solution of NaCl (10 mL). After extraction with Et_2O (10 mL x 3), the organic layer was combined and washed with H_2O (20 mL x 6) to remove DMF. The organic layer was dried over anhydrous $MgSO_4$. After filtration and concentration under reduced pressure, the crude product was purified by column chromatography on silica gel to afford **5** (1.0165 g, 94%) (eluent: petroleum ether/ethyl ether = 50/1) as a colorless oil: **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.64-7.55 (m, 2 H, Ar-H), 7.38-7.30 (m, 2 H, Ar-H), 7.30-7.22 (m, 1 H, Ar-H), 3.19 (s, 3 H, OCH_3), 2.34 (t, J = 7.0 Hz, 2 H, CH_2), 1.69 (s, 3 H, CH_3), 1.64-1.42 (m, 4 H, CH_2 x 2), 0.94 (t, J = 7.4 Hz, 3 H, CH_3); **^{13}C NMR**

(100 MHz, $CDCl_3$) δ = 143.2, 128.1, 127.5, 126.0, 88.1, 79.9, 76.5, 52.1, 32.9, 30.8, 21.9, 18.4, 13.5; **IR** (neat): *v* = 3455, 2924, 2854, 2232, 1489, 1449, 1329, 1140, 1002 cm^{-1}; **MS** (ESI) *m/z* (%): 217 $(M+H)^+$, 185 $(M-OMe)^+$; **HRMS** calcd for $C_{15}H_{21}O^+$ $(M+H)^+$: 217.1587; Found: 217.1586.

3. Synthesis of 1,3-dien-3-yl carboxylic acids

(1) Preparation of (*E*)-2-(1-phenylvinyl)hept-2-enoic acid [(*E*)-3a] (Yy-2-009, Yy-1-183)



Typical Procedure Ⅱ: To a flame-dried Schlenk tube were added $[Pd(\pi\text{-allyl})Cl]_2$ (7.5 mg, 0.02 mmol), BINAP (39.3 mg, 0.06 mmol), and $(PhO)_2POOH$ (387.1 mg, 1.5 mmol) sequentially under argon. After addition of each chemical, the flask was degassed and refilled with Ar. Then **1a** (202.1 mg, 1 mmol)/toluene (5 mL) was added under argon. The resulting mixture was subsequently frozen with a liquid nitrogen bath, degassed to remove the argon inside completely, and refilled with CO by a balloon of CO for three times. Then the resulting mixture was stirred at 80 °C with a balloon of CO for 12 h. After that, the resulting mixture was diluted with 5 mL of ethyl acetate, filtered through a short column of silica gel (2 cm) eluted with ethyl acetate (10 mL x 3), and concentrated. The residue was purified by column chromatography on silica gel to afford impure (*E*)-**3a** (200.5 mg) [eluent: petroleum ether/dichloromethane/ethyl ether = 20/1/1], which was recrystallized (petroleum ether/dichloromethane) to afford pure (*E*)-**3a** (189.5 mg, 82%) as a yellow solid: m.p. 72.6-73.2 °C (petroleum ether/dichloromethane); **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.40-7.22 (m, 5 H, Ar-H), 7.19 (t, *J* = 7.8 Hz, 1 H, =CH), 5.78 (s, 1 H, one proton of $=CH_2$), 5.11 (s, 1 H, one proton of $=CH_2$), 2.22 (q, *J* = 7.5 Hz, 2 H), 1.48-1.36 (m, 2 H, CH_2), 1.36-1.22 (m, 2 H, CH_2), 0.85 (t, *J* = 7.2 Hz, 3 H, CH_3); **^{13}C NMR** (100

MHz, $CDCl_3$) δ = 172.3, 148.9, 142.1, 139.0, 132.9, 128.4, 127.8, 125.7, 116.5, 30.8, 29.5, 22.4, 13.8; **IR** (neat): ν = 2954, 2926, 2854, 2651, 2525, 1679, 1621, 1493, 1418, 1274 cm^{-1}; **MS** (70 eV, EI) *m/z* (%): 231 (M^++1, 4.17), 230 (M^+, 25.77), 143 (100); **Anal.** Calcd for $C_{15}H_{18}O_2$: C 78.23, H 7.88; Found: C 78.26, H 8.11.

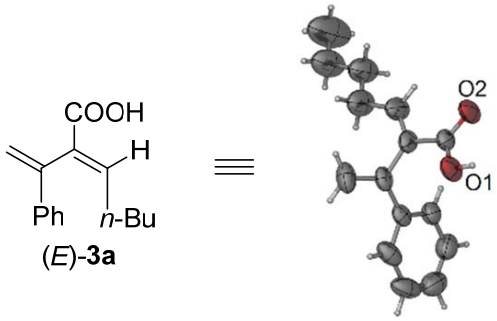
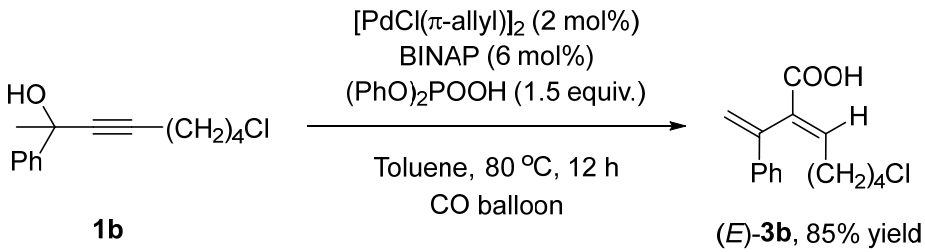


Figure S1

(*E*)-**3a**: $C_{15}H_{18}O_2$, MW = 230.29, monoclinic, space group *I*2/*a*, final *R* indexes [$I > 2\sigma(I)$], R_1 = 0.0719, wR_2 = 0.2203; *R* indexes (all data), R_1 = 0.0759, wR_2 = 0.2259, *a* = 8.7690(2) Å, *b* = 17.0752(4) Å, *c* = 18.1342(3) Å, α = 90°, β = 89.897(2)°, γ = 90°, *V* = 2715.27(10) $Å^3$, *T* = 293(2) K, *Z* = 8, reflections collected/unique 29293/2375 [*R*int = 0.0773], no. of observations [> $2\sigma(I)$] 2120, parameters: 156. Supplementary crystallographic data have been deposited at the Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif. CCDC-1582245.

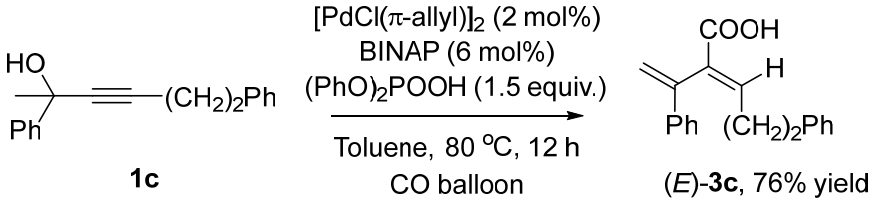
(2) Preparation of (*E*)-7-chloro-2-(1-phenylvinyl)hept-2-enoic acid [(*E*)-3b] (Yy-2-086)



Following **Typical Procedure II**, the reaction of $[PdCl(\pi\text{-allyl})]_2$ (7.5 mg, 0.02 mmol), BINAP (39.3 mg, 0.06 mmol), $(PhO)_2POOH$ (386.2 mg, 1.5 mmol), and **1b** (236.7 mg, 1 mmol)/toluene (5 mL) afforded (*E*)-**3b** (224.9 mg, 85%) [using Biotage

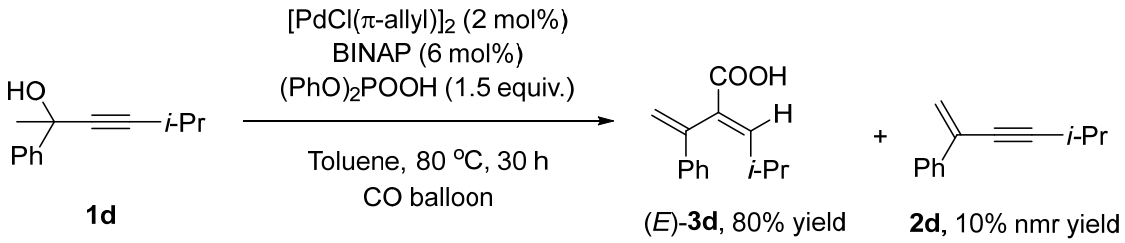
Isorela One purification system on flash silica gel column (Santai Tech. Inc., 12 g), eluent: petroleum ether/ethyl acetate = 2% (2 CV), 2%-17% (14 CV), 17% (6 CV)] as a yellow oil: **^{1}H NMR** (400 MHz, DMSO-*d*$_6$) δ = 12.3 (brs, 1 H, COOH), 7.45-7.24 (m, 5 H, Ar-H), 6.96 (t, *J* = 7.6 Hz, 1 H, =CH), 5.83 (s, 1 H, one proton of =CH_2), 5.06 (s, 1 H, one proton of =CH_2), 3.58 (t, *J* = 6.4 Hz, 2 H, CH_2Cl), 2.17 (q, *J* = 7.3 Hz, 2 H, CH_2), 1.78-1.61 (m, 2 H, CH_2), 1.58-1.46 (m, 2 H, CH_2); **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 171.9, 147.6, 141.9, 138.8, 133.5, 128.5, 127.9, 125.7, 116.7, 44.5, 32.1, 28.9, 25.9; **IR** (neat): *v* = 2952, 2934, 2861, 2640, 2518, 1679, 1618, 1493, 1411, 1267 cm^{-1}; **MS** (70 eV, EI) *m/z* (%): 267 [M^+(^{37}Cl)+1, 1.43)], 266 [M^+(^{37}Cl), 9.00)], 265 [M^+(^{35}Cl)+1, 5.11], 264 [M^+(^{35}Cl), 26.17], 143 (100); **HRMS** Calcd for $C_{15}H_{17}O_2{}^{35}Cl$ (M^+): 264.0917; Found: 264.0921.

(3) Preparation of (*E*)-5-phenyl-2-(1-phenylvinyl)pent-2-enoic acid [(*E*)-3c] (Yy-2-075)



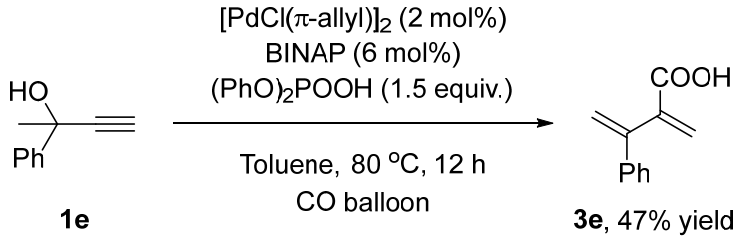
Following **Typical Procedure II**, the reaction of [PdCl(π-allyl)]$_2$ (7.5 mg, 0.02 mmol), BINAP (39.3 mg, 0.06 mmol), $(PhO)_2POOH$ (386.9 mg, 1.5 mmol), and **1c** (250.3 mg, 1 mmol)/toluene (5 mL) afforded (*E*)-**3c** (210.8 mg, 76%) (eluent: petroleum ether/ethyl acetate = 20/1) as a yellow solid: m.p. 98.1-98.5 °C (petroleum ether/dichloromethane); **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 11.7 (brs, 1 H, COOH), 7.31-7.13 (m, 9 H, 8 protons of Ar-H and one proton of =CH), 7.12-7.04 (m, 2 H, Ar-H), 5.72 (s, 1 H, one proton of =CH_2), 4.95 (s, 1 H, one proton of =CH_2), 2.74 (t, *J* = 7.4 Hz, 2 H, CH_2), 2.54 (q, *J* = 7.5 Hz, 2 H, CH_2); **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 171.9, 147.3, 142.0, 140.7, 138.9, 133.6, 128.5, 128.4, 127.8, 126.2, 125.7, 116.7, 34.9, 31.7; IR (neat): *v* = 3025, 2929, 2862, 2639, 2534, 1677, 1635, 1616, 1493, 1424, 1277 cm^{-1}; **MS** (70 eV, EI) *m/z* (%): 279 (M^++1, 1.36), 278 (M^+, 6.03), 91 (100); **Anal.** Calcd for $C_{19}H_{18}O_2$: C 81.99, H 6.52; Found: C 81.68, H 6.51.

(4) Preparation of (*E*)-4-methyl-2-(1-phenylvinyl)pent-2-enoic acid [(*E*)-3d] (cfsy-1-200, Yy-2-109)



Following **Typical Procedure II**, the reaction of $[PdCl(\pi\text{-allyl})]_2$ (7.7 mg, 0.02 mmol), BINAP (39.3 mg, 0.06 mmol), $(PhO)_2POOH$ (379.8 mg, 1.5 mmol), and **1d** (188.5 mg, 1 mmol)/toluene (5 mL) afforded (*E*)-**3d** (174.0 mg, 80%) (10% of **2d** was also detected through ^1H NMR analysis of the crude product using dibromomethane as the internal standard) [eluent: petroleum ether/ethyl acetate = 400/20 to 800/160 to 100/25 (v/v)] as a yellow solid: m.p. 100.3-100.6 °C (petroleum ether/dichloromethane); **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.40-7.20 (m, 5 H, Ar-H), 6.96 (d, *J* = 10.4 Hz, 1 H, =CH), 5.77 (s, 1 H, one proton of $=CH_2$), 5.11 (s, 1 H, one proton of $=CH_2$), 2.72-2.58 (m, 1 H, CH), 1.01 (d, *J* = 6.4 Hz, 6 H, CH_3 x 2); **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 172.5, 154.6, 142.2, 138.9, 130.6, 128.4, 127.8, 125.7, 116.2, 29.0, 22.1; **IR** (neat): *v* = 2962, 2869, 2644, 2514, 1682, 1633, 1612, 1493, 1412, 1267 cm^{-1}; **MS** (70 eV, EI) *m/z* (%): 217 (M^++1, 4.18), 216 (M^+, 26.88), 157 (100); **Anal.** Calcd for $C_{14}H_{16}O_2$: C 77.75, H 7.46; Found: C 77.62, H 7.40.

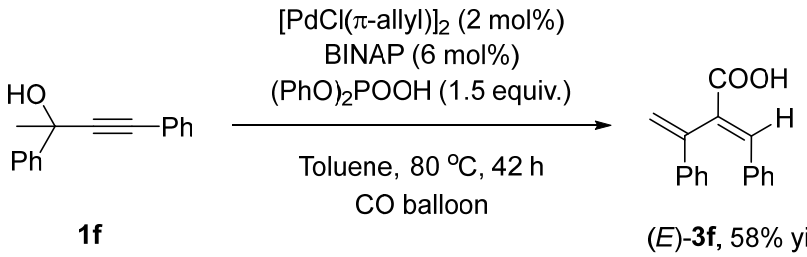
(5) Preparation of 2-(1-phenylvinyl)prop-2-enoic acid (3e) (Yy-2-182)



Following **Typical Procedure II**, the reaction of $[PdCl(\pi\text{-allyl})]_2$ (7.5 mg, 0.02 mmol), BINAP (39.4 mg, 0.06 mmol), $(PhO)_2POOH$ (379.1 mg, 1.5 mmol), and **1e** (146.2 mg, 1 mmol)/toluene (5 mL) afforded **3e**[2] (81.3 mg, 47%) via double chromatography on silica gel (first round: using Biotage Isorela One purification

system on flash silica gel column (Santai Tech. Inc., 12 g), eluent: petroleum ether/ethyl acetate = 2% (2 CV), 2%-17% (14 CV), 17% (6 CV); Then all the product was collected for the second round chromatopraphy on silica gel, eluent: petroleum ether/ethyl acetate = 200/10 to 250/50 to 200/50 to 200/100) (v/v) as a yellow solid, m.p. 101.6-102.3 °C (petroleum ether/dichloromethane) (reported:[2] 106-107 °C): **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 10.85 (brs, 1 H, COOH), 7.40-7.18 (m, 5 H, Ar-H), 6.45 (s, 1 H, one proton of $=CH_2$), 5.89 (s, 1 H, one proton of $=CH_2$), 5.53 (s, 1 H, one proton of $=CH_2$), 5.37 (s, 1 H, one proton of $=CH_2$); **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 172.0, 145.3, 141.2, 139.2, 130.4, 128.3, 127.9, 126.4, 116.8; **IR** (neat): ν = 3023, 2921, 2854, 2637, 2563, 1679, 1612, 1490, 1426, 1260, 1154 cm^{-1}; **MS** (70 eV, EI) *m/z* (%): 175 (M^++1, 4.17), 174 (M^+, 33.00), 129 (100).

(6) Preparation of (*E*)-3-phenyl-2-(1-phenylvinyl)prop-2-enoic acid [(*E*)-3f] (Yy-2-029)

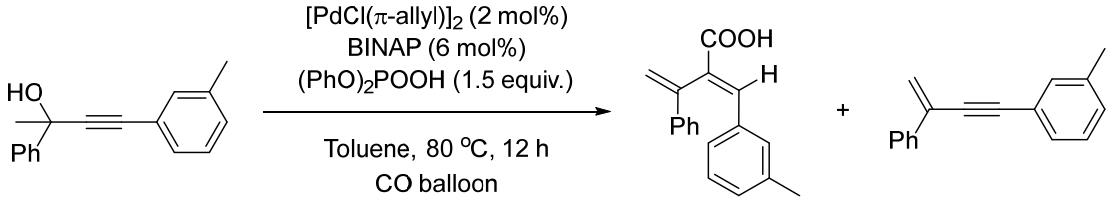


1f (*E*)-**3f**, 58% yield

Following **Typical Procedure II**, the reaction of $[PdCl(\pi\text{-allyl})]_2$ (7.5 mg, 0.02 mmol), BINAP (39.3 mg, 0.06 mmol), $(PhO)_2POOH$ (386.9 mg, 1.5 mmol), and **1f** (222.1 mg, 1 mmol)/toluene (5 mL) afforded (*E*)-**3f** (145.2 mg, 58%) (eluent: petroleum ether/ethyl acetate = 10/1) as a yellow solid: m.p. 141.3-141.8 °C (petroleum ether/dichloromethane); **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.96 (s, 1 H, =CH), 7.64-7.54 (m, 2 H, Ar-H), 7.54-7.45 (m, 2 H, Ar-H), 7.37-7.21 (m, 6 H, Ar-H), 5.86 (s, 1 H, one proton of $=CH_2$), 5.28 (s, 1 H, one proton of $=CH_2$); **^{13}C NMR** (100 MHz, $(CD_3)_2CO$) δ = 168.5, 143.6, 141.4, 138.4, 135.4, 134.0, 133.5, 131.0, 130.2, 129.4, 129.3, 128.3, 117.3; **IR** (neat): ν = 3051, 2922, 2852, 2622, 2509, 1676, 1602, 1494, 1420, 1270, 1204 cm^{-1}; **MS** (70 eV, EI) *m/z* (%): 251 (M^++1, 3.57), 250 (M^+, 19.39), 205 (100); **Anal.** Calcd for $C_{17}H_{14}O_2$: C 81.58, H 5.64; Found: C 81.42, H

5.91.

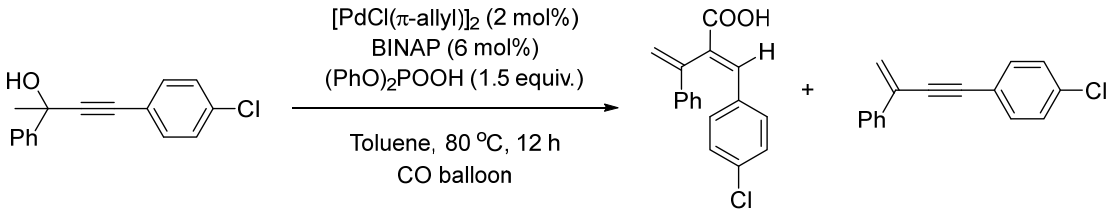
(7) Preparation of (*E*)-3-(3-methylphenyl)-2-(1-phenylvinyl)prop-2-enoic acid [(*E*)-3g] (Yy-2-085)



1g (*E*)-**3g**, 40% yield **2g**, 29% nmr yield

Following **Typical Procedure II**, the reaction of $[PdCl(\pi\text{-allyl})]_2$ (7.5 mg, 0.02 mmol), BINAP (39.3 mg, 0.06 mmol), $(PhO)_2POOH$ (386.9 mg, 1.5 mmol), and **1g** (236.4 mg, 1 mmol)/toluene (5 mL) afforded (*E*)-**3g** (106.4 mg, 40%) (29% **2g** was also detected through ^1H NMR analysis of the crude product using dibromomethane as the internal standard) [using Biotage Isorela One purification system on flash silica gel column (Santai Tech. Inc., 12 g), eluent: petroleum ether/ethyl acetate = 2% (2 CV), 2%-17% (14 CV), 17% (6 CV)] as a yellow solid: m.p. 118.3-118.8 °C (petroleum ether/dichloromethane); **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.93 (s, 1 H, =CH), 7.53-7.45 (m, 2 H, Ar-H), 7.42 (d, *J* = 7.2 Hz, 1 H, Ar-H), 7.36 (s, 1 H, Ar-H), 7.35-7.26 (m, 3 H, Ar-H), 7.16-7.06 (m, 2 H, Ar-H), 5.85 (s, 1 H, one proton of $=CH_2$), 5.27 (s, 1 H, one proton of $=CH_2$), 2.26 (s, 3 H, CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 172.9, 143.3, 142.9, 138.2, 138.0, 134.0, 131.7, 131.1, 130.6, 128.6, 128.3, 128.1, 127.4, 125.8, 116.8, 21.3; **IR** (neat): *v* = 3049, 2919, 2852, 2629, 2514, 1678, 1603, 1492, 1421, 1279, 1242 cm^{-1}; **MS** (70 eV, EI) *m/z* (%): 265 (M^++1, 4.94), 264 (M^+, 24.31), 219 (100); **Anal.** Calcd for $C_{18}H_{16}O_2$: C 81.79, H 6.10; Found: C 81.68, H 6.18.

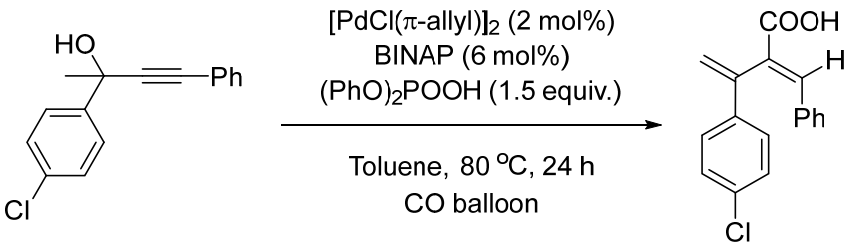
(8) Preparation of (*E*)-3-(4-chlorophenyl)-2-(1-phenylvinyl)prop-2-enoic acid [(*E*)-3h] (Yy-2-102)



1h (*E*)-**3h**, 48% yield **2h**, 14% nmr yield

Following **Typical Procedure II**, the reaction of [PdCl(π-allyl)]$_2$ (7.5 mg, 0.02 mmol), BINAP (39.3 mg, 0.06 mmol), $(PhO)_2POOH$ (379.3 mg, 1.5 mmol), and **1h** (256.1 mg, 1 mmol)/toluene (5 mL) afforded (*E*)-**3h** (138.0 mg, 48%) [eluent: pure dichloromethane (400 mL) to dichloromethane/MeOH = 150/1] as a yellow solid: m.p. 145.3-146.0 °C (petroleum ether/dichloromethane); **^{1}H NMR** (400 MHz, DMSO-d_6) δ = 12.67 (s, 1 H, COOH), 7.79 (s, 1 H, =CH), 7.58 (d, J = 8.8 Hz, 2 H, Ar-H), 7.47 (d, J = 7.2 Hz, 2 H, Ar-H), 7.41-7.28 (m, 5 H, Ar-H), 5.91 (s, 1 H, one proton of $=CH_2$), 5.17 (s, 1 H, one proton of $=CH_2$); **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 172.8, 142.5, 141.8, 137.7, 135.8, 132.4, 131.80, 131.77, 128.75, 128.74, 128.3, 125.7, 116.9; **IR** (neat): v = 2795, 2622, 2510, 1676, 1601, 1589, 1490, 1418, 1281 cm^{-1}; **MS** (70 eV, EI) *m/z* (%): 287 [M^+(^{37}Cl)+1, 1.55], 286 [M^+(^{37}Cl), 8.69], 285 [M^+(^{35}Cl)+1, 5.43], 284 [M^+(^{35}Cl), 27.39], 203 (100); **Anal.** Calcd for $C_{17}H_{13}ClO_2$: C 71.71, H 4.60; Found: C 71.60, H 4.80.

(9) Preparation of (*E*)-3-phenyl-2-(1-(4-chlorophenyl)vinyl)prop-2-enoic acid [(*E*)-3i] (Yy-2-033)

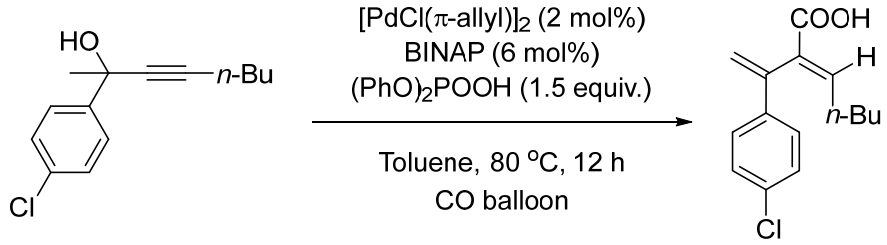


1i (*E*)-**3i**, 51% yield

Following **Typical Procedure II**, the reaction of [PdCl(π-allyl)]$_2$ (7.5 mg, 0.02 mmol), BINAP (39.3 mg, 0.06 mmol), $(PhO)_2POOH$ (386.6 mg, 1.5 mmol), and **1i** (256.1 mg, 1 mmol)/toluene (5 mL) afforded impure (*E*)-**3i** (eluent: petroleum

ether/ethyl acetate = 10/1) as yellow solid, which was recrystallized from petroleum ether/dichloromethane to give (*E*)-**3i** as a white solid (146.1 mg, 51%): m.p. 139.2-140.5 °C (petroleum ether/dichloromethane); **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.96 (s, 1 H, =CH), 7.62-7.50 (m, 2 H, Ar-H), 7.49-7.38 (m, 2 H, Ar-H), 7.36-7.22 (m, 5 H, Ar-H), 5.85 (s, 1 H, one proton of $=CH_2$), 5.30 (s, 1 H, one proton of $=CH_2$); **^{13}C NMR** (100 MHz, DMSO-d_6) δ = 167.9, 142.0, 140.0, 137.0, 134.2, 132.62, 132.56, 129.98, 129.50, 128.7, 128.6, 127.4, 116.8; **IR** (neat): ν = 3029, 2805, 2635, 2513, 1670, 1600, 1488, 1416, 1270, 1206 cm^{-1}; **MS** (70 eV, EI) *m/z* (%): 287 [M^+(^{37}Cl)+1, 1.65], 286 [M^+(^{37}Cl), 9.22], 285 [M^+(^{35}Cl)+1, 5.73], 284 [M^+(^{35}Cl), 27.61], 204 (100); **HRMS** Calcd for $C_{17}H_{13}{}^{35}ClO_2$: 284.0604; Found: 284.0601.

(10) Preparation of (*E*)-2-(1-(4-chlorophenyl)vinyl)hept-2-enoic acid [(*E*)-3j] (Yy-2-103)

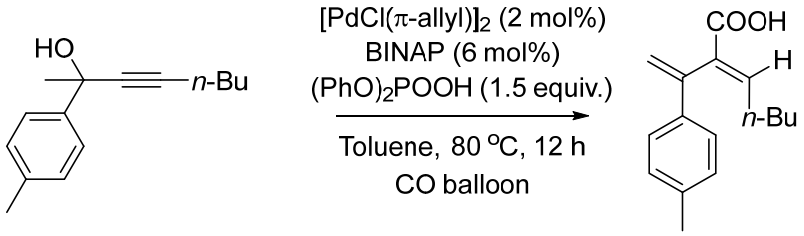


1j (*E*)-**3j**, 69% yield

Following **Typical Procedure II**, the reaction of $[PdCl(\pi\text{-allyl})]_2$ (7.5 mg, 0.02 mmol), BINAP (39.4 mg, 0.06 mmol), $(PhO)_2POOH$ (379.2 mg, 1.5 mmol), **1h** (236.2 mg, 1 mmol)/toluene (5 mL) afforded (*E*)-**3j** (182.0 mg, 69%) [using Biotage Isorela One purification system on flash silica gel column (Santai Tech. Inc., 12 g), eluent: petroleum ether/ethyl acetate = 2% (2 CV), 2%-17% (14 CV), 17% (6 CV)] as a white solid: m.p. 68.8-69.8 °C (petroleum ether/dichloromethane); **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.32-7.23 (m, 4 H, Ar-H), 7.20 (t, *J* = 7.6 Hz, 1 H, =CH), 5.77 (s, 1 H, one proton of $=CH_2$), 5.13 (s, 1 H, one proton of $=CH_2$), 2.21 (q, *J* = 7.6 Hz, 2 H, CH_2), 1.48-1.24 (m, 4 H, CH_2 x 2), 0.86 (t, *J* = 7.2 Hz, 3 H, CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 172.0, 149.3, 141.1, 137.6, 133.7, 132.5, 128.6, 127.1, 117.1, 30.8, 29.5, 22.4, 13.8; **IR** (neat): ν = 2966, 2931, 2872, 2638, 2538, 1673, 1620, 1488, 1432,

1287 cm^{-1}; **MS** (70 eV, EI) *m/z* (%): 267 [M^+(^{37}Cl)+1, 2.82)], 266 [M^+(^{37}Cl), 13.47)], 265 [M^+(^{35}Cl)+1, 6.86], 264 [M^+(^{35}Cl), 40.59], 177 (100); **Anal.** Calcd for $C_{15}H_{17}ClO_2$: C 68.05, H 6.47; Found: C 68.04, H 6.41.

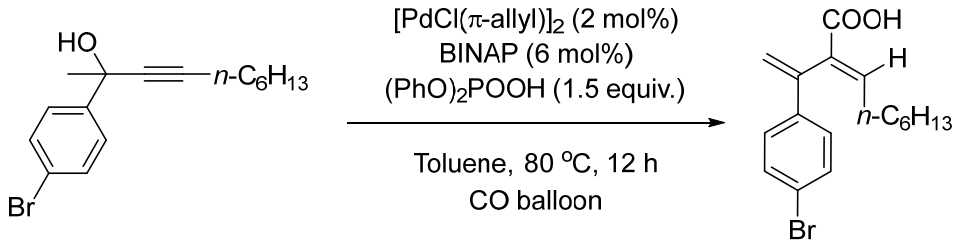
(11) Preparation of (*E*)-2-(1-(p-tolyl)vinyl)hept-2-enoic acid [(*E*)-3k] (Yy-2-105)



1k (*E*)-**3k**, 40% yield

Following **Typical Procedure II**, the reaction of [PdCl(π-allyl)]$_2$ (7.5 mg, 0.02 mmol), BINAP (39.3 mg, 0.06 mmol), $(PhO)_2POOH$ (379.1 mg, 1.5 mmol), and **1k** (216.9 mg, 1 mmol)/toluene (5 mL) afforded (*E*)-**3k** (105.2 mg, 94% purity, 40%) [using Biotage Isorela One purification system on flash silica gel column (Santai Tech. Inc., 12 g), eluent: petroleum ether/ethyl acetate = 2% (2 CV), 2%-17% (14 CV), 17% (6 CV)] as a yellow oil: **^{1}H NMR** (400 MHz, DMSO-*d*$_6$) δ = 12.21 (s, 1 H, COOH), 7.23 (d, *J* = 7.6 Hz, 2 H, Ar-H), 7.13 (d, *J* = 7.6 Hz, 2 H, Ar-H), 6.93 (t, *J* = 7.8 Hz, 1 H, =CH), 5.76 (s, 1 H, one proton of $=CH_2$), 4.98 (s, 1 H, one proton of $=CH_2$), 2.28 (s, 3 H, CH_3), 2.11 (q, *J* = 7.5 Hz, 2 H, CH_2), 1.43-1.15 (m, 4 H, CH_2 x 2), 0.81 (t, *J* = 7.2 Hz, 3 H, CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 172.0, 148.8, 141.8, 137.7, 136.1, 132.9, 129.1, 125.6, 115.6, 30.9, 29.5, 22.5, 21.1, 13.8; **IR** (neat): *v* = 2956, 2925, 2859, 2644, 2522, 1683, 1632, 1512, 1412, 1274 cm^{-1}; **MS** (70 eV, EI) *m/z* (%): 245 (M^++1, 5.60), 244 (M^+, 30.89), 157 (100); **HRMS** Calcd for $C_{16}H_{20}O_2$ (M^+): 244.1463; Found: 244.1467.

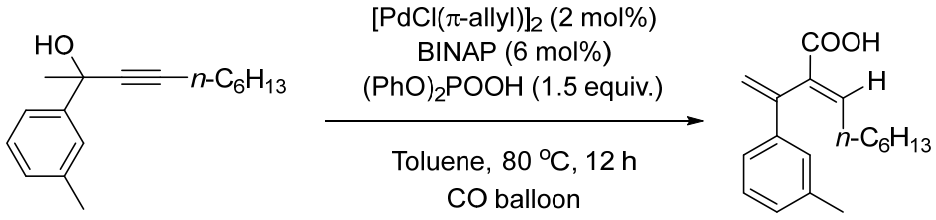
(12) Preparation of (*E*)-2-(1-(4-bromophenyl)vinyl)non-2-enoic acid [(*E*)-3l] (Yy-2-022)



1l (*E*)-**3l**, 73% yield

Following **Typical Procedure II**, the reaction of [PdCl(π-allyl)]$_2$ (7.5 mg, 0.02 mmol), BINAP (39.3 mg, 0.06 mmol), (PhO)$_2$POOH (386.9 mg, 1.5 mmol), and **1l** (309.2 mg, 1 mmol)/toluene (5 mL) afforded (*E*)-**3l** (247.6 mg, 73%) (eluent: petroleum ether/ethyl acetate = 20/1) as a yellow solid: m.p. 83.0-83.4 °C (dichloromethane); **^{1}H NMR** (400 MHz, DMSO-*d*$_6$) δ = 12.32 (s, 1 H, COOH), 7.52 (d, *J* = 8.0 Hz, 2 H, Ar-H), 7.28 (d, *J* = 8.4 Hz, 2 H, Ar-H), 6.96 (t, *J* = 7.6 Hz, 1 H, =CH), 5.85 (s, 1 H, =CH$_2$), 5.09 (s, 1 H, =CH$_2$), 2.11 (q, *J* = 7.6 Hz, 2 H, CH$_2$), 1.44-1.30 (m, 2 H, CH$_2$), 1.28-1.10 (m, 6 H, CH$_2$ x 3), 0.81 (t, *J* = 7.0 Hz, 3 H, CH$_3$); **^{13}C NMR** (100 MHz, CDCl$_3$): δ = 172.0, 149.4, 141.1, 138.0, 132.4, 131.5, 127.4, 121.9, 117.2, 31.5, 29.8, 29.0, 28.6, 22.5, 14.0; **IR** (neat): *v* = 2923, 2850, 2634, 2528, 1679, 1603, 1482, 1419, 1283, 1204 cm^{-1}; **MS** (70 eV, EI) *m/z* (%): 339 [M$^+$(^{81}Br)+1, 3.04], 338 [M$^+$(^{81}Br), 16.30], 337 [M$^+$(^{79}Br)+1, 3.75], 336 [M$^+$(^{79}Br), 16.49], 142 (100); **Anal.** Calcd for C$_{17}$H$_{21}$BrO$_2$: C 60.54, H 6.28; Found: C 60.36, H 6.36.

(13) Preparation of (*E*)**-2-(1-(m-tolyl)vinyl)non-2-enoic acid [**(*E*)**-3m] (Yy-2-037)**

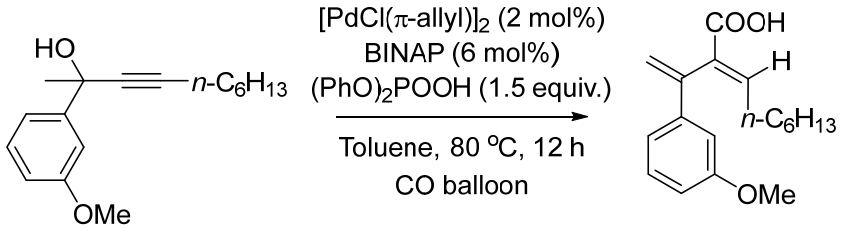


1m (*E*)-**3m**, 74% yield

Following **Typical Procedure II**, the reaction of [PdCl(π-allyl)]$_2$ (7.5 mg, 0.02 mmol), BINAP (39.3 mg, 0.06 mmol), (PhO)$_2$POOH (386.9 mg, 1.5 mmol), and **1m** (244.6 mg, 1 mmol)/toluene (5 mL) afforded (*E*)-**3m** (202.3 mg, 74%) via double chromatography on silica gel (first round eluent: petroleum ether/ethyl acetate = 20/1;

second round eluent: dichloromethane/MeOH = 150/1) as a yellow oil: **^{1}H NMR** (400 MHz, DMSO-*d*$_6$) δ = 12.23 (s, 1 H, COOH), 7.26-7.06 (m, 4 H, Ar-H), 6.94 (t, *J* = 7.6 Hz, 1 H, =CH), 5.79 (s, 1 H, one proton of =CH_2), 5.02 (s, 1 H, one proton of =CH_2), 2.29 (s, 3 H, CH_3), 2.13 (q, *J* = 7.3 Hz, 2 H, CH_2), 1.46-1.32 (m, 2 H, CH_2), 1.30-1.11 (m, 6 H, CH_2 x 3), 0.81 (t, *J* = 6.6 Hz, 3 H, CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 172.0, 148.9, 142.1, 138.9, 138.0, 132.9, 128.7, 128.3, 126.4, 122.9, 116.4, 31.5, 29.8, 29.1, 28.7, 22.5, 21.5, 14.0; **IR** (neat): *v* = 2955, 2924, 2855, 2644, 2523, 1683, 1633, 1456, 1414, 1277 cm^{-1}; **MS** (70 eV, EI) *m/z* (%): 273 (M^++1, 4.33), 272 (M^+, 20.18), 157 (100); **HRMS** Calcd for $C_{18}H_{24}O_2$ (M^+): 272.1776; Found: 272.1778.

(14) Preparation of (*E*)-2-(1-(3-methoxyphenyl)vinyl)non-2-enoic acid [(*E*)-3n] (Yy-2-080)

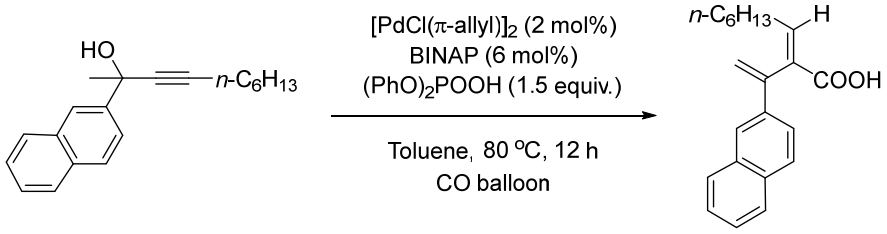


1n (*E*)-**3n**, 76% yield

Following **Typical Procedure II**, the reaction of [PdCl(π-allyl)]$_2$ (7.5 mg, 0.02 mmol), BINAP (39.3 mg, 0.06 mmol), $(PhO)_2POOH$ (387.0 mg, 1.5 mmol), and **1n** (260.4 mg, 1 mmol)/toluene (5 mL) afforded (*E*)-**3n** (219.2 mg, 76%) [using Biotage Isorela One purification system on flash silica gel column (Santai Tech. Inc., 12 g), eluent: petroleum ether/ethyl acetate = 2% (2 CV), 2%-17% (14 CV), 17% (6 CV)] as a yellow oil: **^{1}H NMR** (400 MHz, DMSO-*d*$_6$) δ = 12.25 (s, 1 H, COOH), 7.25 (t, *J* = 8.4 Hz, 1 H, =CH), 7.00-6.81 (m, 4 H, Ar-H), 5.84 (s, 1 H, one proton of =CH_2), 5.05 (s, 1 H, one proton of =CH_2), 3.75 (s, 3 H, CH_3), 2.13 (q, *J* = 7.5 Hz, 2 H, CH_2), 1.45-1.30 (m, 2 H, CH_2), 1.30-1.10 (m, 6 H, CH_2 x 3), 0.81 (t, *J* = 6.8 Hz, 3 H, CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 172.1, 159.7, 148.9, 142.0, 140.5, 132.8, 129.4, 118.4, 116.8, 113.2, 111.5, 55.2, 31.5, 29.8, 29.1, 28.7, 22.5, 14.0; **IR** (neat): *v* = 2954, 2925, 2855, 2649, 2522, 1683, 1576, 1487, 1463, 1417, 1277, 1224 cm^{-1}; **MS** (70 eV,

EI) *m/z* (%): 289 (M^++1, 10.12), 288 (M^+, 46.37), 173 (100); **HRMS** Calcd for $C_{18}H_{24}O_3$ (M^+): 288.1725; Found: 288.1724.

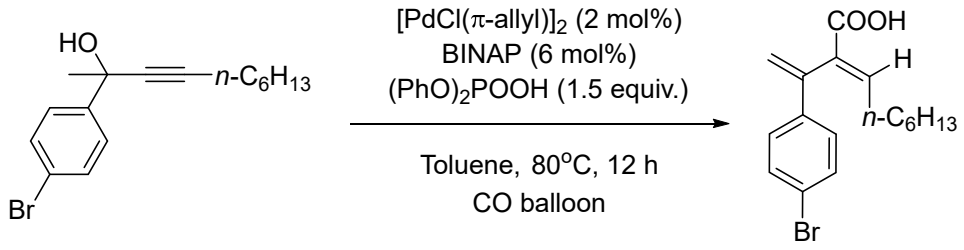
(15) Preparation of (*E*)-2-(1-(2-naphthyl)vinyl)non-2-enoic acid [(*E*)-3o] (cfsy-2-001, Yy-2-188)



1o (*E*)-**3o**, 75% yield

Following **Typical Procedure II**, the reaction of [PdCl(π-allyl)]$_2$ (7.5 mg, 0.02 mmol), BINAP (39.6 mg, 0.06 mmol), $(PhO)_2POOH$ (379.3 mg, 1.5 mmol), and **1o** (280.8 mg, 1 mmol)/toluene (5 mL) afforded (*E*)-**3o** (231.7 mg, 75%) (eluent: petroleum ether/ethyl acetate = 400/20 to 800/160) as a yellow oil: **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 10.74 (brs, 1 H, COOH), 7.83-7.70 (m, 3 H, Ar-H), 7.66 (s, 1 H, Ar-H), 7.62-7.55 (m, 1 H, Ar-H), 7.48-7.38 (m, 2 H, Ar-H), 7.24 (t, *J* = 7.6 Hz, 1 H, =CH), 5.92 (s, 1 H, one proton of $=CH_2$), 5.19 (s, 1 H, one proton of $=CH_2$), 2.22 (q, *J* = 7.5 Hz, 2 H, CH_2), 1.49-1.36 (m, 2 H, CH_2), 1.32-1.12 (m, 6 H, CH_2 x 3), 0.81 (t, *J* = 6.8 Hz, 3 H, CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 172.3, 149.2, 142.0, 136.2, 133.3, 133.0, 132.8, 128.3, 128.1, 127.5, 126.1, 125.9, 124.9, 123.7, 117.0, 31.5, 29.8, 29.1, 28.6, 22.5, 14.0; **IR** (neat): *v* = 3054, 2923, 2855, 2645, 2521, 1681, 1625, 1456, 1414, 1273 cm^{-1}; **MS** (70 eV, EI) *m/z* (%): 309 (M^++1, 11.08), 308 (M^+, 47.97), 179 (100); **HRMS** Calcd for $C_{21}H_{24}O_2$: 308.1776; Found: 308.1778.

(16) Preparation of (*E*)-2-(1-(4-bromophenyl)vinyl)non-2-enoic acid [(*E*)-3l] in Gram scale (Yy-2-111)



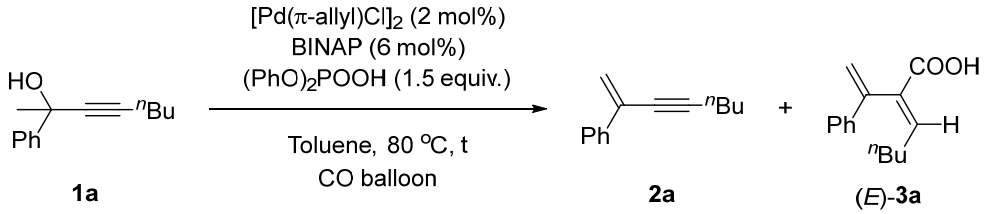
1l
5 mmol

(*E*)-**3l**, 77%
1.3016 g

Following **Typical Procedure II**, the reaction of $[PdCl(\pi\text{-allyl})]_2$ (37.3 mg, 0.10 mmol), BINAP (196.6 mg, 0.30 mmol), $(PhO)_2POOH$ (1.8954 g, 7.5 mmol), and **1l** (1.5464 g, 5 mmol)/toluene (25 mL) afforded (*E*)-**3l** (1.3016 g, 77%) via double chromatography on silica gel (first round eluent: dichloromethane/MeOH = 150/1; Then all the crude product was collected for the second round chromatography, eluent: dichloromethane/MeOH = 150/1) as a light yellow solid: **^{1}H NMR** (400 MHz, DMSO-d_6) δ = 12.36 (s, 1 H, COOH), 7.53 (d, J = 8.8 Hz, 2 H, Ar-H), 7.29 (d, J = 8.8 Hz, 2 H, Ar-H), 6.98 (t, J = 7.8 Hz, 1 H, =CH), 5.87 (s, 1 H, $=CH_2$), 5.10 (s, 1 H, $=CH_2$), 2.12 (q, J = 7.3 Hz, 2 H, CH_2), 1.50-1.30 (m, 2 H, CH_2), 1.30-1.11 (m, 6 H, CH_2 x 3), 0.81 (t, J = 7.0 Hz, 3 H, CH_3).

.

4. Reaction monitoring using substrate 1a [a] (Yy-2-194)



t/h	NMR yield of enyne **2a** [b]/%	NMR yield of product **3a** [b]/%
0.17	83	8
0.33	78	25
0.5	69	35
1	51	48
2	30	57
3	24	75
4	16	76
6	8	86
8	2	92
10	0	94

[a] **Following Typical Procedure II,** the reaction of **1a** (201.9 mg, 1 mmol), $[Pd(\pi\text{-allyl})Cl]_2$ (7.5 mg, 0.02 mmol), BINAP (39.3 mg, 0.06 mmol), and $(PhO)_2POOH$ (379.1 mg, 1.5 equiv.) in toluene (5 mL) was conducted at 80 °C under 1 atm. of CO. [b] Determined by taking 0.3 mL of the reaction solution followed by ^1H NMR analysis using 3.5 μL of dibromomethane as the internal standard.

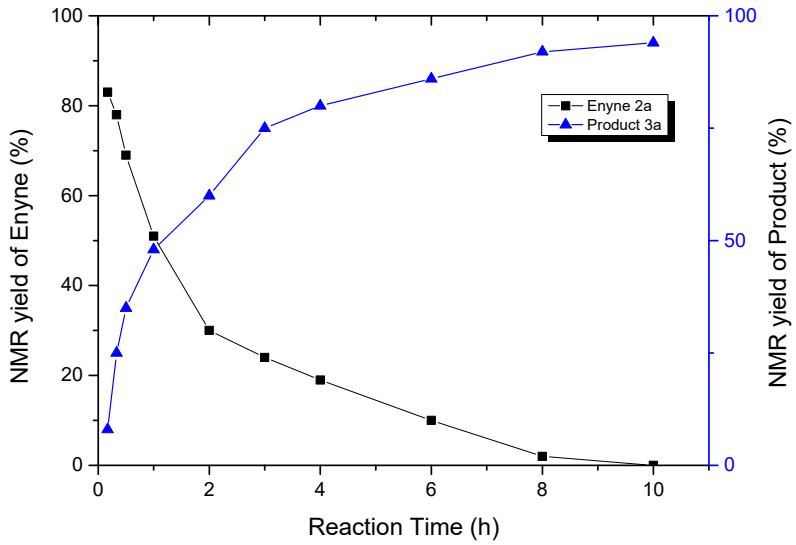
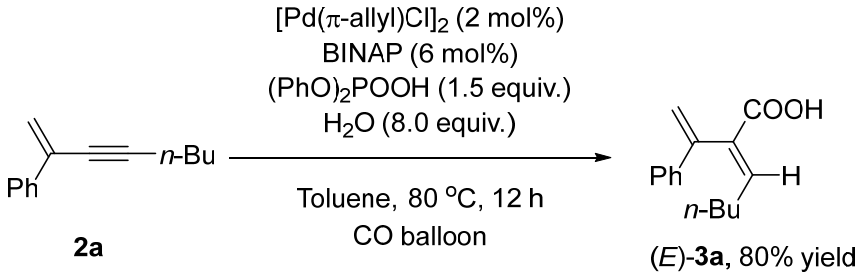


Figure S2. Reaction monitoring of **2a** and (E)-**3a**

5. Control experiments

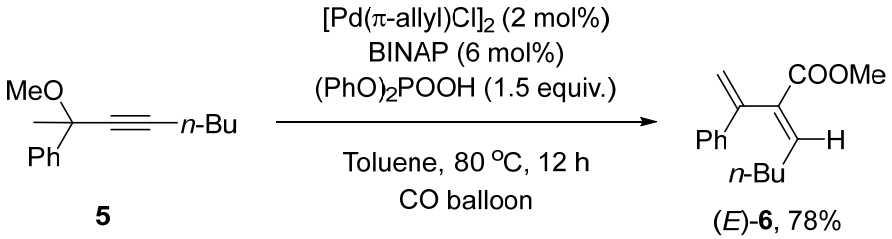
(1) Preparation of (*E*)-2-(1-phenylvinyl)hept-2-enoic acid [(*E*)-3a] via enyne (2a) (Yy-2-119)



2a (*E*)-**3a**, 80% yield

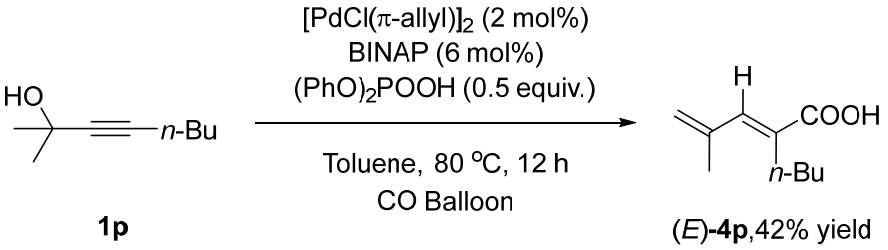
To a flame-dried Schlenk tube were added [Pd(π-allyl)Cl]$_2$ (7.5 mg, 0.02 mmol), BINAP (39.3 mg, 0.06 mmol), and (PhO)$_2$POOH (379.6 mg, 1.5 mmol) sequentially under argon. After addition of each chemical, the flask was degassed and refilled with Ar. Then **2a**[1] (182.2 mg, 1 mmol)/toluene (2 mL) and H_2O (144.2 mg, 8 mmol)/toluene (3 mL) were added under argon. The resulting mixture was then frozen with a liquid nitrogen bath, degassed to remove the argon inside completely, and refilled with CO by a balloon of CO for three times. Then the resulting mixture was stirred at 80 °C with a balloon of CO for 12 h. After that, the resulting mixture was diluted with 5 mL of ethyl acetate, filtered through a short column of silica gel (2 cm) eluted with ethyl acetate (10 mL x 3), and concentrated. The residue was purified by column chromatography on silica gel to afford (*E*)-**3a** (182.9 mg, 80%) (eluent: petroleum ether/ethyl acetate = 20/1) as a yellow solid: **^{1}H NMR** (400 MHz, $CDCl_3$): δ = 7.35 (d, *J* = 6.8 Hz, 2 H, Ar-H), 7.32-7.24 (m, 3 H, Ar-H), 7.19 (t, *J* = 7.6 Hz, 1 H, =CH), 5.79 (s, 1 H, one proton of =CH_2), 5.11 (s, 1 H, one proton of =CH_2), 2.22 (q, *J* = 7.5 Hz, 2 H, CH_2), 1.46-1.36 (m, 2 H, CH_2), 1.36-1.23 (m, 2 H, CH_2), 0.85 (t, *J* = 7.4 Hz, 3 H, CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 172.2, 148.9, 142.1, 139.0, 132.9, 128.4, 127.8, 125.7, 116.6, 30.8, 29.5, 22.5, 13.8.

(2) Preparation of methyl (*E*)-2-(1-phenylvinyl)hept-2-enoate [(*E*)-6] via (2-methoxyoct-3-yn-2-yl)benzene (5) (Yy-2-122)



Following **Typical Procedure II**, the reaction of $[PdCl(\pi\text{-allyl})]_2$ (7.5 mg, 0.02 mmol), BINAP (39.4 mg, 0.06 mmol), $(PhO)_2POOH$ (379.4 mg, 1.5 mmol), and **5** (215.6 mg, 1 mmol)/toluene (5 mL) afforded (*E*)-**6** (196.6 mg, 96% purity, 78%) (eluent: petroleum ether/ethyl acetate = 20/1) as a colorless oil: **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.40-7.20 (m, 5 H, Ar-H), 7.10 (t, *J* = 7.6 Hz, 1 H, =CH), 5.79 (s, 1 H, one proton of $=CH_2$), 5.12 (s, 1 H, one proton of $=CH_2$), 3.63 (s, 3 H, CH_3), 2.22 (q, *J* = 7.3 Hz, 2 H, CH_2), 1.48-1.38 (m, 2 H, CH_2), 1.38-1.26 (m, 2 H, CH_2), 0.86 (t, *J* = 7.2 Hz, 3 H, CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 167.4, 146.4, 142.6, 139.2, 133.3, 128.4, 127.7, 125.7, 116.2, 51.9, 31.0, 29.3, 22.5, 13.8; **IR** (neat): *v* = 2948, 2862, 1714, 1620, 1491, 1439, 1243, 1142, 1048 cm^{-1}; **MS** (70 eV, EI) *m/z* (%): 245 (M^++1, 8.58), 244 (M^+, 48.44), 143 (100); **HRMS** Calcd for $C_{16}H_{20}O_2$ (M^+): 244.1463; Found: 244.1464.

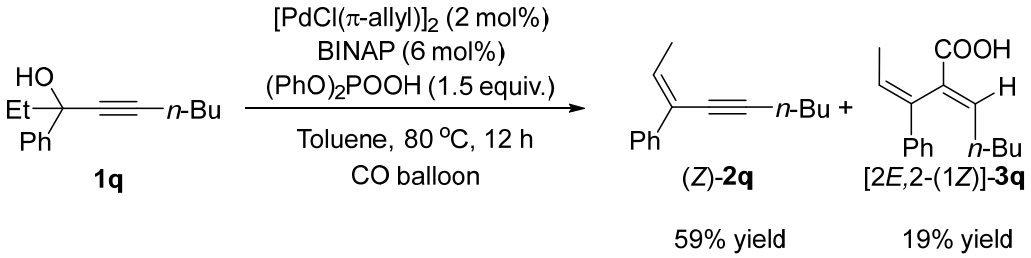
(3) Preparation of (*E*)-4-methyl-2-butylpent-2,4-dienioic acid [(*E*)-4p] via 2-methyloct-3-yn-2-ol (1p) (Yy-2-100)



To a flame-dried Schlenk tube were added $[Pd(\pi\text{-allyl})Cl]_2$ (7.5 mg, 0.02 mmol), BINAP (39.3 mg, 0.06 mmol), and $(PhO)_2POOH$ (126.3 mg, 0.5 mmol) sequentially under argon. After addition of each chemical, the flask was degassed and refilled with Ar. Then **1p** (140.4 mg, 1 mmol)/toluene (5mL) was added under argon. The resulting mixture was subsequently frozen with a liquid nitrogen bath, degassed to remove the argon inside completely, and refilled with CO by a balloon of CO for three times. The

resulting mixture was stirred at 80 ºC with a balloon of CO for 12 h. After that, the resulting mixture was diluted with 5 mL of ethyl acetate, filtered through a short column of silica gel (2 cm) eluted with ethyl acetate (10 mL x 3), and concentrated. The residue was purified by column chromatography on silica gel for double chromatography on silica gel to afford (*E*)-**4p** (79.3 mg, 90% purity, 42%) (first round eluent: petroleum ether/ethyl acetate = 20/1; Then all the crude product was collected for the second round chromatography, eluent: dichloromethane/MeOH = 150/1) as a yellow oil: **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.23 (s, 1 H, =CH), 5.22 (s, 1 H, one proton of $=CH_2$), 5.13 (s, 1 H, one proton of $=CH_2$), 2.46 (t, *J* = 7.8 Hz, 2 H, CH_2), 1.96 (s, 3 H, CH_3), 1.50-1.30 (m, 4 H, CH_2 x 2), 0.91 (t, *J* = 7.2 Hz, 3 H, CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 174.4, 142.7, 140.7, 131.8, 120.5, 32.2, 26.9, 22.8, 22.4, 13.9; **IR** (neat): *v* = 2958, 2928, 2861, 1678, 1629, 1454, 1415, 1269, 1210, 1144, 1066 cm^{-1}; **MS** (70 eV, EI) *m/z* (%): 169 (M^++1, 8.06), 168 (M^+, 71.03), 79 (100); **HRMS** Calcd for $C_{10}H_{16}O_2$ (M^+): 168.1150; Found: 168.1153.

(4) Preparation of (*Z*)-3-phenyl-2-nonen-4-yne [(*Z*)-2q] and (*E*)-2-((*Z*)-1-phenylprop-1-en-1-yl)hept-2-enoic acid {[2*E*,2-(1*Z*)]-3q} via 3-phenylnon-4-yn-3-ol (1q) (Yy-3-090)



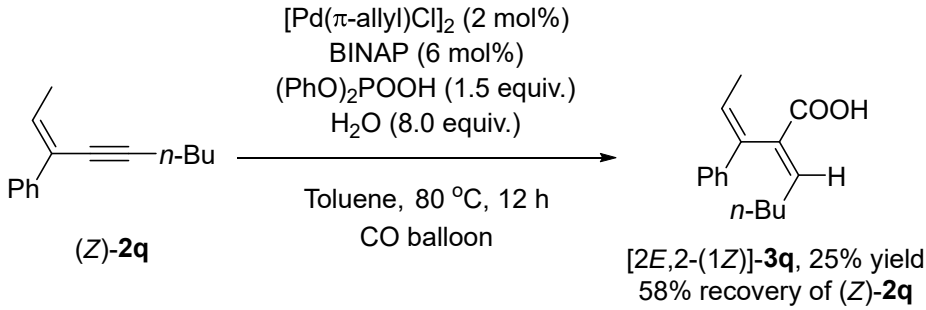
Following **Typical Procedure II**, the reaction of $[PdCl(\pi\text{-allyl})]_2$ (7.5 mg, 0.02 mmol), BINAP (39.3 mg, 0.06 mmol), $(PhO)_2POOH$ (379.3 mg, 1.5 mmol), and **1q** (216.0 mg, 1 mmol)/toluene (5 mL) afforded (*Z*)-**2q**[3] [(117.3 mg, 59% yield, stabilized with 3.8 mg 2,6-di-tert-butyl-4-methylphenol (BHT)] and [2*E*,2-(1*Z*)]-**3q** (49.6 mg, 93% purity, 19% yield) (eluent: petroleum ether/ethyl acetate = 400/20 to 800/160) (v/v).

(*Z*)-**2q**[3]: yellow oil; **^{1}H NMR** (*Z*)-**2q** (400 MHz, $CDCl_3$) δ = 7.68-7.50 (m, 2 H,

Ar-H), 7.38-7.27 (m, 2 H, Ar-H), 7.27-7.18 (m, 1 H, Ar-H), 6.38 (q, J = 6.9 Hz, 1 H, =CH), 2.47 (t, J = 7.0 Hz, 2 H, CH_2), 2.04 (d, J = 7.2 Hz, 3 H, CH_3), 1.72-1.45 (m, 4 H, CH_2 x 2), 0.95 (t, J = 7.4 Hz, 3 H, CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 138.9, 131.5, 128.2, 127.1, 125.8, 124.7, 96.7, 77.6, 31.0, 22.0, 19.3, 16.7, 13.6; **IR** (neat): v = 2958, 2931, 2872, 2860, 1494, 1466, 1446, 1433, 1348 cm^{-1}; **MS** (70 eV, EI) *m/z* (%): 199 (M^++1, 16.69), 198 (M^+, 100).

[2*E*,2-(1*Z*)]-**3q**: yellow oil; **^{1}H NMR** (400 MHz, C_6D_6) δ = 7.40-7.32 (m, 2 H, Ar-H), 7.27 (t, J = 7.4 Hz, 1 H, =CH), 7.14-7.09 (m, 2 H, Ar-H), 7.08-7.00 (m, 1 H, Ar-H), 6.06 (q, J = 6.8 Hz, 1 H, =CH), 1.86 (q, J = 7.3 Hz, 2 H, CH_2), 1.55 (d, J = 6.8 Hz, 3 H, CH_3), 1.16-0.97 (m, 4 H, CH_2 x 2), 0.68 (t, J = 7.0 Hz, 3 H, CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 172.3, 149.2, 140.2, 135.1, 130.1, 128.3, 126.9, 126.4, 125.6, 30.3, 29.5, 22.5, 15.5, 13.8; **IR** (neat): v = 2958, 2929, 2872, 2858, 2645, 2537, 1683, 1623, 1494, 1441, 1416, 1274, 1210, 1152, 1032 cm^{-1}; **MS** (70 eV, EI) *m/z* (%): 245 (M^++1, 10.39), 244 (M^+, 55.42), 129 (100); **HRMS** Calcd for $C_{16}H_{20}O_2$ (M^+): 244.1463; Found: 244.1465.

(5) Preparation of (*E*)-2-((*Z*)-1-phenylprop-1-en-1-yl)hept-2-enoic acid {[2*E*,2-(1*Z*)]-3q} via (*Z*)-3-phenyl-2-nonen-4-yne [(*Z*)-2q] (Yy-3-094)



To a flame-dried Schlenk tube were added [Pd(π-allyl)Cl]$_2$ (7.5 mg, 0.02 mmol), BINAP (39.3 mg, 0.06 mmol), and (PhO)$_2$POOH (379.1 mg, 1.5 mmol) sequentially under argon. After addition of each chemical, the flask was degassed and refilled with Ar. Then (*Z*)-**2q** (198.3 mg, 1 mmol)/toluene (2 mL) and H_2O (144.4 mg, 8 mmol)/toluene (3 mL) were added under argon. The resulting mixture was then frozen with a liquid nitrogen bath, degassed to remove the argon inside completely, and

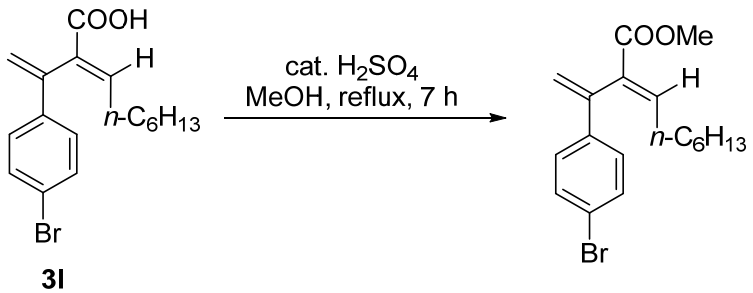
refilled with CO by a balloon of CO for three times. Then the resulting mixture was stirred at 80 ºC with a balloon of CO for 12 h. After that, the resulting mixture was diluted with 5 mL of ethyl acetate, filtered through a short column of silica gel (1 cm) eluted with ethyl acetate (10 mL x 3), and concentrated. The residue was purified by column chromatography on silica gel to afford [2*E*,2-(1*Z*)]-**3q** (66.3 mg, 93% purity, 25%) and (*Z*)-**2q** [115.0 mg, 58% yield, stabilized with 3.8 mg 2,6-di-tert-butyl-4-methylphenol (BHT)] (eluent: petroleum ether/ethyl acetate = 500/0 to 400/20 to 800/160) (v/v).

(*Z*)-**2q**[3]: **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.65-7.50 (m, 2 H, Ar-H), 7.38-7.26 (m, 2 H, Ar-H), 7.26-7.16 (m, 1 H, Ar-H), 6.38 (q, *J* = 6.9 Hz, 1 H, =CH), 2.47 (t, *J* = 7.0 Hz, 2 H, CH_2), 2.04 (d, *J* = 7.2 Hz, 3 H, CH_3), 1.69-1.45 (m, 4 H, CH_2 x 2), 0.95 (t, *J* = 7.2 Hz, 3 H, CH_3).

[2*E*,2-(1*Z*)]-**3q**: **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.40-7.15 (m, 6 H, Ar-H and =CH), 6.24 (q, *J* = 6.8 Hz, 1 H, =CH), 2.09 (q, *J* = 7.3 Hz, 2 H, CH_2), 1.69 (d, *J* = 6.8 Hz, 3 H, CH_3), 1.48-1.20 (m, 4 H, CH_2 x 2), 0.84 (t, *J* = 7.2 Hz, 3 H, CH_3).

6. Synthetic applications:

(1) Preparation of methyl (*E*)-2-(1-(4-bromophenyl)vinyl)non-2-enoate (Yy-2-161)

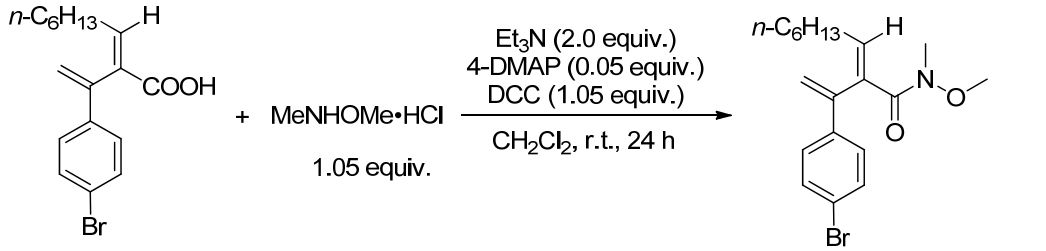


3l (*E*)-**3l-ester**, 81% yield

To a flame-dried flask were added (*E*)-**3l** (790.3 mg, 2.34 mmol) and 20 mL MeOH, then 5 drop of conc. H_2SO_4 in 10 mL MeOH was added. The resulting mixture was refluxed for 7 h. After cooling to room temperature, the mixture was quenched with a saturated aqueous solution of $NaHCO_3$ (20 mL) and extracted with

ethyl acetate (30 mL x 3). The organic layer was washed with a saturated aqueous solution of NaCl (50 mL) and dried over anhydrous Na_2SO_4. After filtration and concentration under reduced pressure, the crude product was purified by column chromatography on silica gel to afford (*E*)-**3l-ester** (668.6 mg, 81%) (eluent: petroleum ether/ethyl acetate = 20/1) as a colorless oil: **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.43 (d, *J* = 8.8 Hz, 2 H, Ar-H), 7.25-7.18 (m, 2 H, Ar-H), 7.10 (t, *J* = 7.8 Hz, 1 H, =CH), 5.77 (s, 1 H, one proton of $=CH_2$), 5.13 (s, 1 H, one proton of $=CH_2$), 3.64 (s, 3 H, OCH_3), 2.19 (q, *J* = 7.5 Hz, 2 H, CH_2), 1.50-1.36 (m, 2 H, CH_2), 1.34-1.16 (m, 6 H, CH_2 x 3), 0.86 (t, *J* = 6.8 Hz, 3 H); **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 167.2, 146.9, 141.6, 138.3, 132.8, 131.5, 127.4, 121.8, 116.8, 51.9, 31.5, 29.6, 29.0, 28.8, 22.5, 14.0; **IR** (neat): *v* = 2924, 2856, 1715, 1637, 1483, 1435, 1386, 1240, 1191, 1057, 1005 cm^{-1}; **MS** (70 eV, EI) *m/z* (%): 353 [M^+(^{81}Br)+1, 8.82], 352 [M^+(^{81}Br), 45.4], 351 [M^+(^{79}Br)+1, 11.39], 350 [M^+(^{79}Br), 46.2], 141 (100); **HRMS** Calcd for $C_{18}H_{23}{}^{79}BrO_2$ (M^+): 350.0876; Found: 350.0878.

(2) Preparation of (*E*)**-2-(1-(4-bromophenyl)vinyl)-*N*-methoxy-*N*-methylnon-2-enamide [**(*E*)**-3l-Weinreb amide]** [4] **(Yy-2-180)**

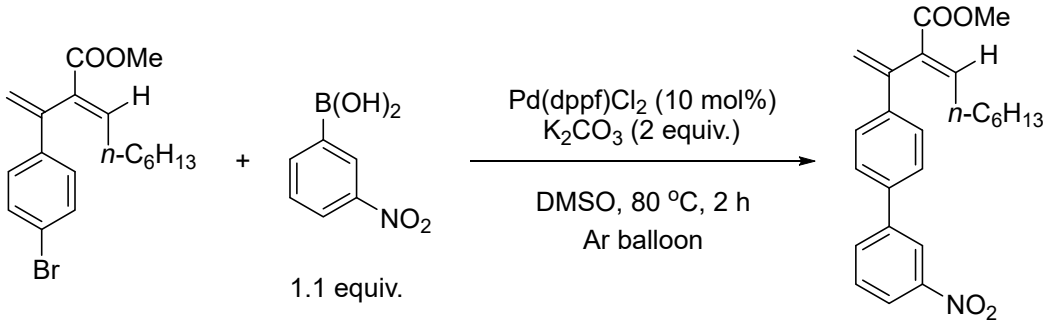


(*E*)-**3l** (*E*)-**3l-Weireb amide**, 47% yield

To a Schlenk tube equipped with an empty balloon were added (*E*)-**3l** (674.3 mg, 2 mmol), MeNHOMe•HCl (206.8 mg, 2.1 mmol), and CH_2Cl_2 (4 mL). The reaction was then stirred at 0 ºC for 5 min, Et_3N (0.56 mL, d = 0.728 g/mol, 407.7mg, 4 mmol), 4-DMAP (12.2 mg, 0.10 mmol), DCC (442.3 mg, 2.1 mmol), and CH_2Cl_2 (2 mL) were then added sequentially. The resulting mixture was stirred at room temperature for 24 h before diluted with CH_2Cl_2 (15 mL) and quenched with H_2O (25 mL). The organic layer was separated and the aqueous phase was extracted with CH_2Cl_2 (20

mL), the organic phase was combined and washed with a saturated solution of $NaHCO_3$, H_2O, a saturated solution of NaCl (aq.) sequentially, and dried over anhydrous $MgSO_4$. After filtration and concentration under reduced pressure, the crude product was purified by column chromatography on silica gel to afford (*E*)-**3l-Weinreb amide** (368.4 mg, 98% purity, 47%) (eluent: petroleum ether/ethyl acetate = 200/10 to 600/60) as a colorless oil: **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.44 (d, *J* = 8.8 Hz, 2 H, Ar-H), 7.34 (d, *J* = 8.4 Hz, 2 H, Ar-H), 6.42 (t, *J* = 7.6 Hz, 1 H, =CH), 5.68 (s, 1 H, one proton of $=CH_2$), 5.20 (s, 1 H, one proton of $=CH_2$), 3.46 (s, 3 H, OCH_3), 3.14 (s, 3 H, NCH_3), 2.04 (q, *J* = 7.5 Hz, 2 H, CH_2), 1.44-1.32 (m, 2 H, CH_2), 1.32-1.10 (m, 6 H, CH_2 x 3), 0.85 (t, *J* = 6.8 Hz, 3 H, CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 170.7, 142.6, 140.7, 138.3, 135.3, 131.3, 128.1, 121.7, 115.6, 60.4, 33.4, 31.4, 29.1, 28.9, 28.8, 22.4, 14.0; **IR** (neat): *v* = 2924, 2856, 1648, 1455, 1363, 1182, 1110, 1071 cm^{-1}; **MS** (70 eV, EI) *m/z* (%): 382 [$M^+(Br^{81})$+1, 1.84], 381 [$M^+(Br^{81})$, 8.21], 380 [$M^+(^{79}Br)$+1, 1.92], 379 [$M^+(^{79}Br)$, 8.09], 142 (100); **HRMS** Calcd for $C_{19}H_{26}{}^{79}BrNO_2$ (M^+): 379.1147; Found: 379.1151.

(3) Coupling [5] of (*E*)-3l-ester with (3-nitrophenyl)boronic acid (Yy-2-141)

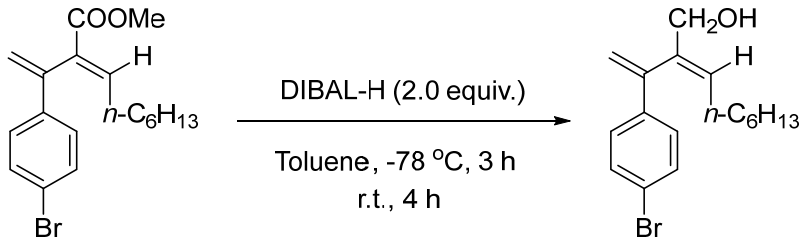


(*E*)-**3l-ester** (*E*)-**7**, 80% yield

To a flame-dried Schlenk tube were added $Pd(dppf)Cl_2$ (14.6 mg, 0.02 mmol), (3-nitrophenyl)boronic acid (37.5 mg, 0.22 mmol), and K_2CO_3 (55.4 mg, 0.4 mmol) sequentially. After addition of each chemical, the flask was degassed and refilled with argon. Then (*E*)-**3l-ester** (70.2 mg, 0.2 mmol)/DMSO (2 mL) was added under argon. The resulting mixture was stirred at 80 °C with a balloon of argon for 2 h, diluted with 2 mL of ethyl acetate, cooled to room temperature, and quenched with 10 mL of H_2O.

After extraction with ethyl acetate (20 mL x 3) and washed with a saturated solution of NaCl (10 mL x 3), the organic layer was dried over anhydrous Na_2SO_4. After filtration through a layer of silica gel and concentration under reduced pressure, the residue was purified by column chromatography on silica gel using a pipette column to afford (*E*)-**7** (66.8 mg, 94% purity, 80%) (eluent: petroleum ether/ethyl acetate = 100/1 to 100/5) (v/v) as a colorless oil: **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.45 (t, *J* = 1.8 Hz, 1 H, Ar-H), 8.19 (dd, J_1 = 7.8 Hz, J_2 = 1.4 Hz, 1 H, Ar-H), 7.91 (d, *J* = 7.6 Hz, 1 H, Ar-H), 7.66-7.55 (m, 3 H, Ar-H), 7.49 (d, *J* = 8.4 Hz, 2 H, Ar-H), 7.15 (t, *J* = 7.6 Hz, 1 H, =CH), 5.88 (s, 1 H, one proton of $=CH_2$), 5.20 (s, 1 H, one proton of $=CH_2$), 3.67 (s, 3 H, OCH_3), 2.24 (q, *J* = 7.5 Hz, 2 H, CH_2), 1.54-1.40 (m, 2 H, CH_2), 1.38-1.18 (m, 6 H, CH_2 x 3), 0.86 (t, *J* = 6.6 Hz, 3 H, CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 167.3, 148.7, 146.8, 142.4, 141.9, 139.6, 137.9, 133.0, 132.8, 129.7, 127.2, 126.5, 122.0, 121.7, 117.0, 52.0, 31.5, 29.6, 29.1, 28.8, 22.5, 14.0; **IR** (neat): 2925, 2855, 1713, 1529, 1435, 1347, 1242, 1054 cm^{-1}; **MS** (70 eV, EI) *m/z* (%): 394 (M^++1, 17.36), 393 (M^+, 66.39), 264 (100); **HRMS** Calcd for $C_{24}H_{27}NO_4$: 393.1940; Found: 393.1936.

(4) Reduction [6] **of** (*E*)**-3l-ester with DIBAL-H (Yy-2-146)**

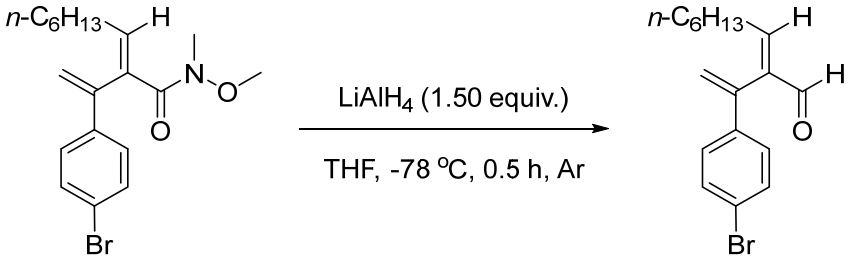


(*E*)-**3l-ester** (*E*)-**8**, 60% yield

To a flame-dried Schlenk tube was added (*E*)-**3l-ester** (175.4 mg, 0.5 mmol)/toluene (5 mL). After the tube was stirred at -78°C for 10 min, DIBAL-H (1 mL, 1.0 M in toluene, 1 mmol) was added dropwise within 5 min. The resulting mixture was stirred at -78°C for 3 hours and stirred at room temperature for 4 h, quenched with a saturated solution of potassium sodium tartrate (Rochelle's salt) (5 mL), extracted with Et_2O (5 mL x 3), dried over anhydrous $MgSO_4$, filtered, and

concentrated *in vacuo*. The residue was purified by column chromatography on silica gel to afford (*E*)-**8** (96.4 mg, 60%) as a colorless oil (eluent: petroleum ether/ethyl acetate = 200/10 to 200/20) (v/v): **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 7.44 (d, *J* = 8.8 Hz, 2 H, Ar-H), 7.27 (d, *J* = 8.4 Hz, 2 H, Ar-H), 5.76 (t, *J* = 7.4 Hz, 1 H, =CH), 5.65 (d, *J* = 1.6 Hz, 1 H, one proton of $=CH_2$), 5.14 (d, *J* = 1.2 Hz, 1 H, one proton of $=CH_2$), 4.07 (s, 2 H, CH_2O), 2.01 (q, *J* = 7.3 Hz, 2 H, CH_2), 1.53 (br, 1 H, OH), 1.40-1.15 (m, 8 H, CH_2 x 4), 0.86 (t, *J* = 6.8 Hz, 3 H, CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 144.3, 139.4, 138.1, 131.5, 130.9, 128.0, 121.8, 115.8, 66.6, 31.6, 29.6, 29.0, 28.7, 22.6, 14.0; **IR** (neat): 3315 (br), 2954, 2922, 2853, 1484, 1458, 1387, 1071, 1006 cm^{-1}; **MS** (70 eV, EI) *m/z* (%): 325 [M^+(^{81}Br)+1, 2.53], 324 [M^+(^{81}Br), 10.12], 323 [M^+(^{79}Br)+1, 2.02], 322 [M^+(^{79}Br), 10.56], 212 (100); **HRMS** Calcd for $C_{17}H_{23}{}^{79}BrO$ (M^+): 322.0932; Found: 322.0936.

(5) Reduction of (*E*)-3l-Weinreb amide with $LiAH_4$ [7] (Yy-2-183)



(*E*)-**3l-Weinreb amide** (*E*)-**9**, 56% yield

To a flame-dried Schlenk tube fulfilled with argon were added (*E*)-**3l-Weinreb amide** (150.7 mg, 0.4 mmol) and THF (5 mL). The reaction was stirred at -78 °C for 10 min, $LiAlH_4$ (0.6 mL, 1 M in THF, 0.6 mmol) was then added dropwise within 2 min. The resulting mixture was stirred at -78 °C for 0.5 h, quenched with ethyl acetate (5 mL), and poured into 1 M HCl (20 mL). After extraction with ethyl acetate (15 mL x 3), the organic layer was washed with a saturated solution of NaCl (aq.) and dried over anhydrous Na_2SO_4. After filtration and concentration under reduced pressure, the crude product was purified by column chromatography on silica gel to afford (*E*)-**9** (78.9 mg, 90% purity, 56%) (eluent: petroleum ether/ethyl acetate = 50/1) as a colorless oil: **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 9.54 (s, 1 H, CHO), 7.42 (d, *J* = 8.0 Hz,

2 H, Ar-H), 7.17 (d, J = 8.4 Hz, 2 H, Ar-H), 6.81 (t, J = 7.6 Hz, 1 H, =CH), 5.84 (s, 1 H, one proton of $=CH_2$), 5.12 (s, 1 H, one proton of $=CH_2$), 2.30 (q, J = 7.5 Hz, 2 H, CH_2), 1.52-1.38 (m, 2 H, CH_2), 1.36-1.08 (m, 6 H, CH_2 x 3), 0.86 (t, J = 6.8 Hz, 3 H, CH_3); **^{13}C NMR** (100 MHz, $CDCl_3$) δ = 193.2, 157.4, 144.0, 139.7, 137.7, 131.6, 127.4, 121.9, 117.6, 31.4, 29.9, 29.0, 28.5, 22.5, 14.0; **IR** (neat): v = 3442 (br), 2923, 2856, 1685, 1590, 1483, 1392, 1272, 1217, 1071, 1006 cm^{-1}; **MS** (70 eV, EI) *m/z* (%): 323 [M^+(^{81}Br)+1, 3.02], 322 [M^+(^{81}Br), 15.02], 321 [M^+(^{79}Br)+1, 5.44], 320 [M^+(^{79}Br), 14.94], 149 (100); **HRMS** Calcd for $C_{17}H_{21}{}^{79}BrO$ (M^+): 320.0776; Found: 320.0772.

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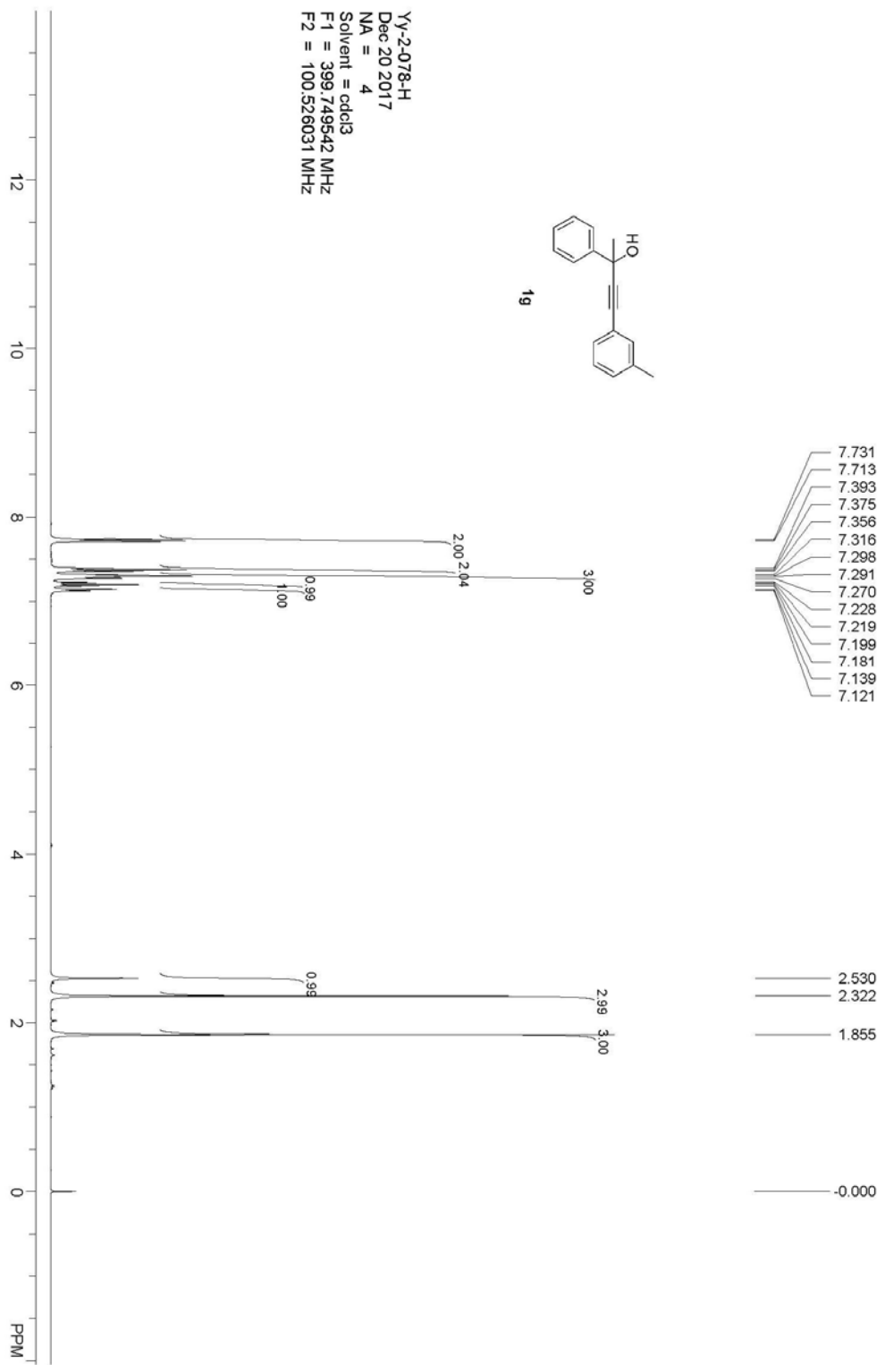
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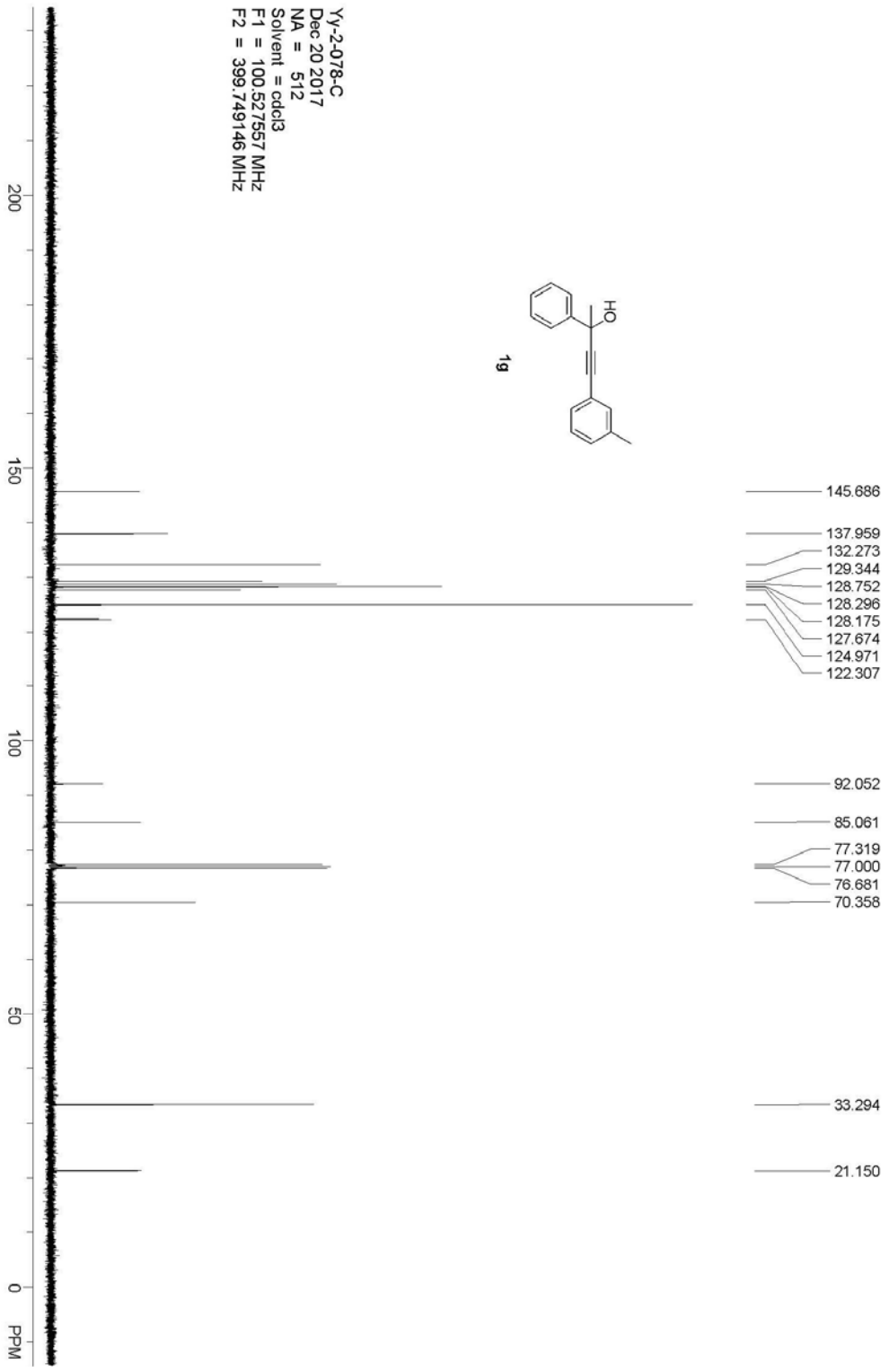
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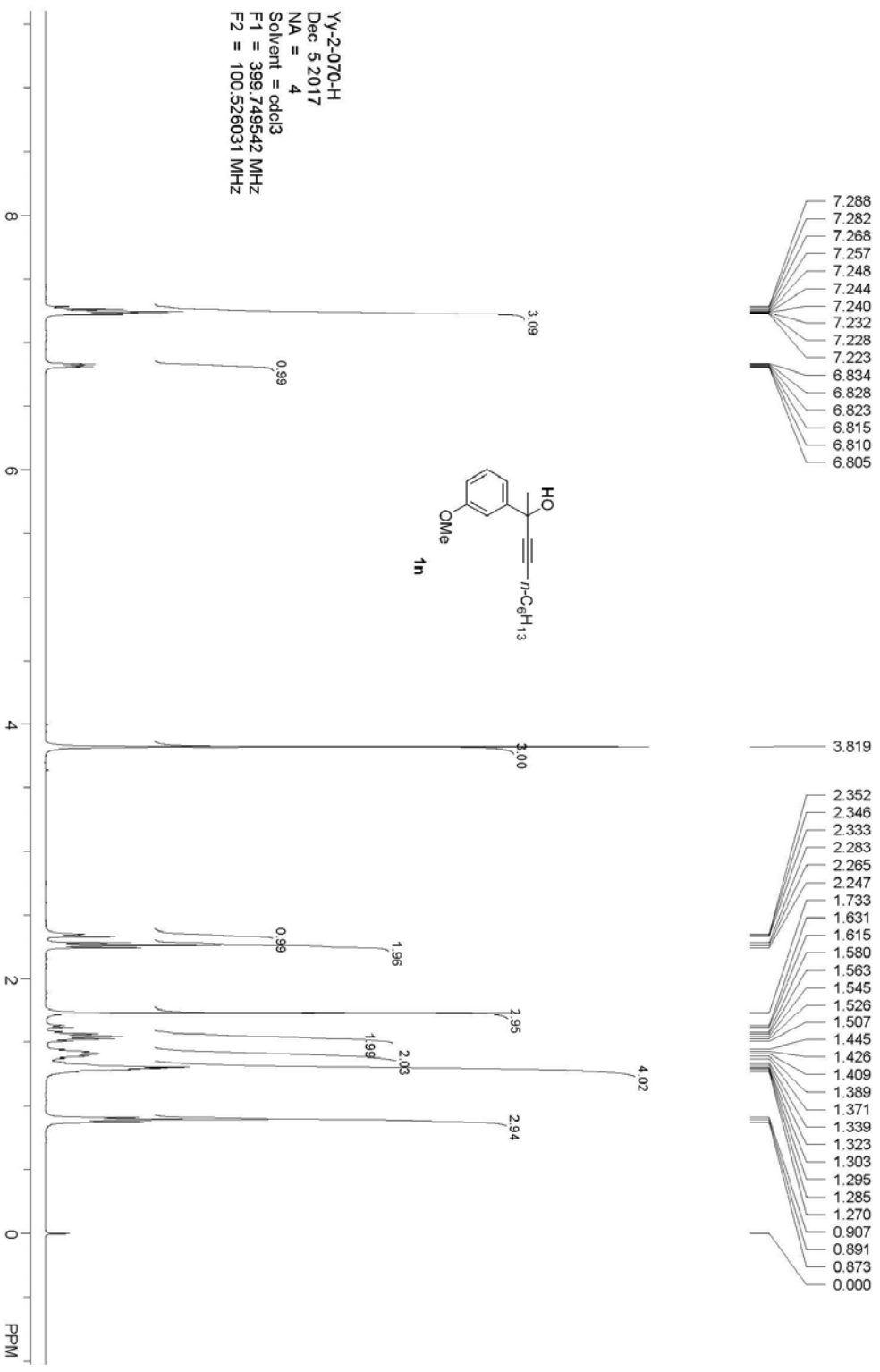
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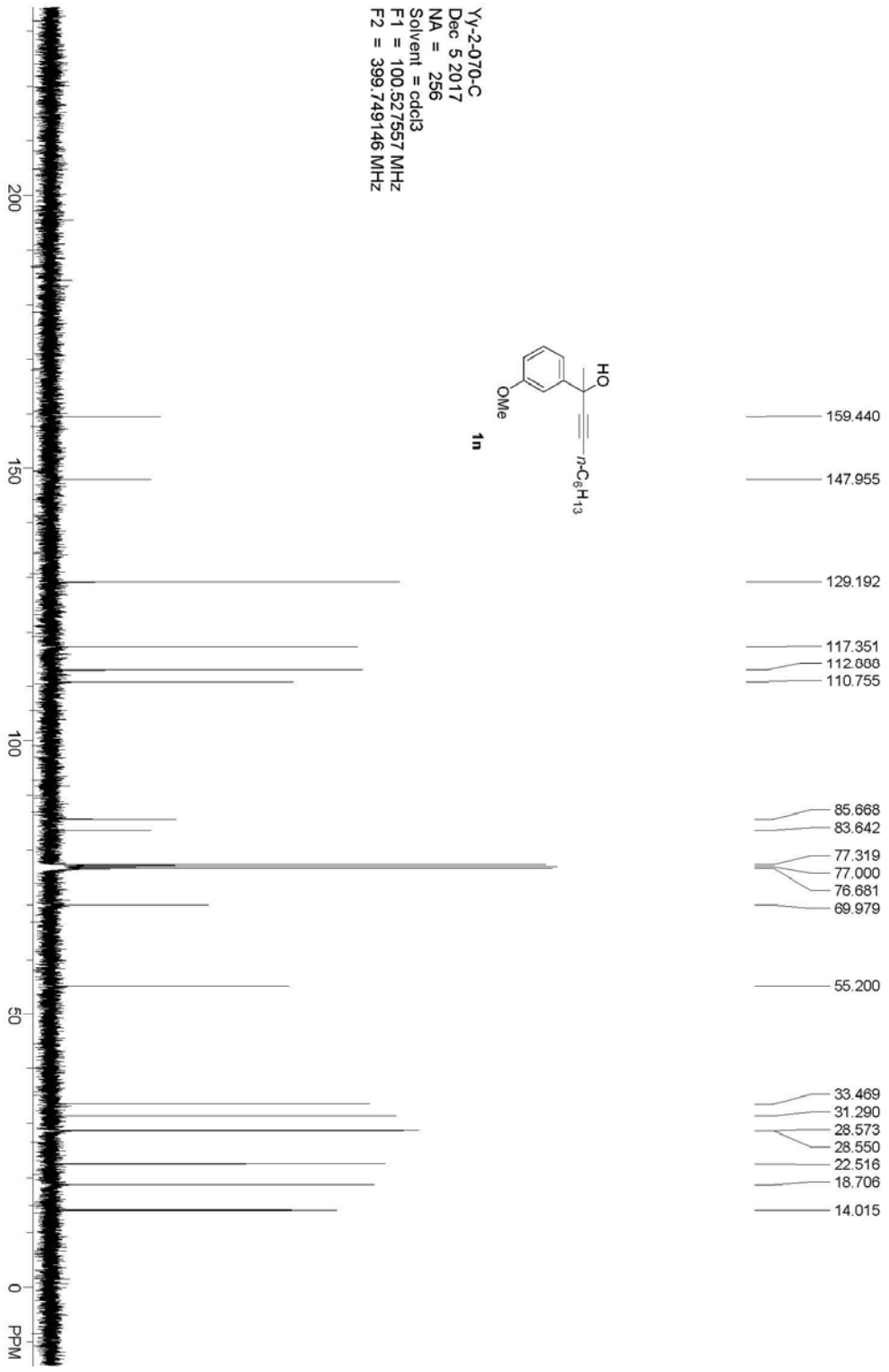
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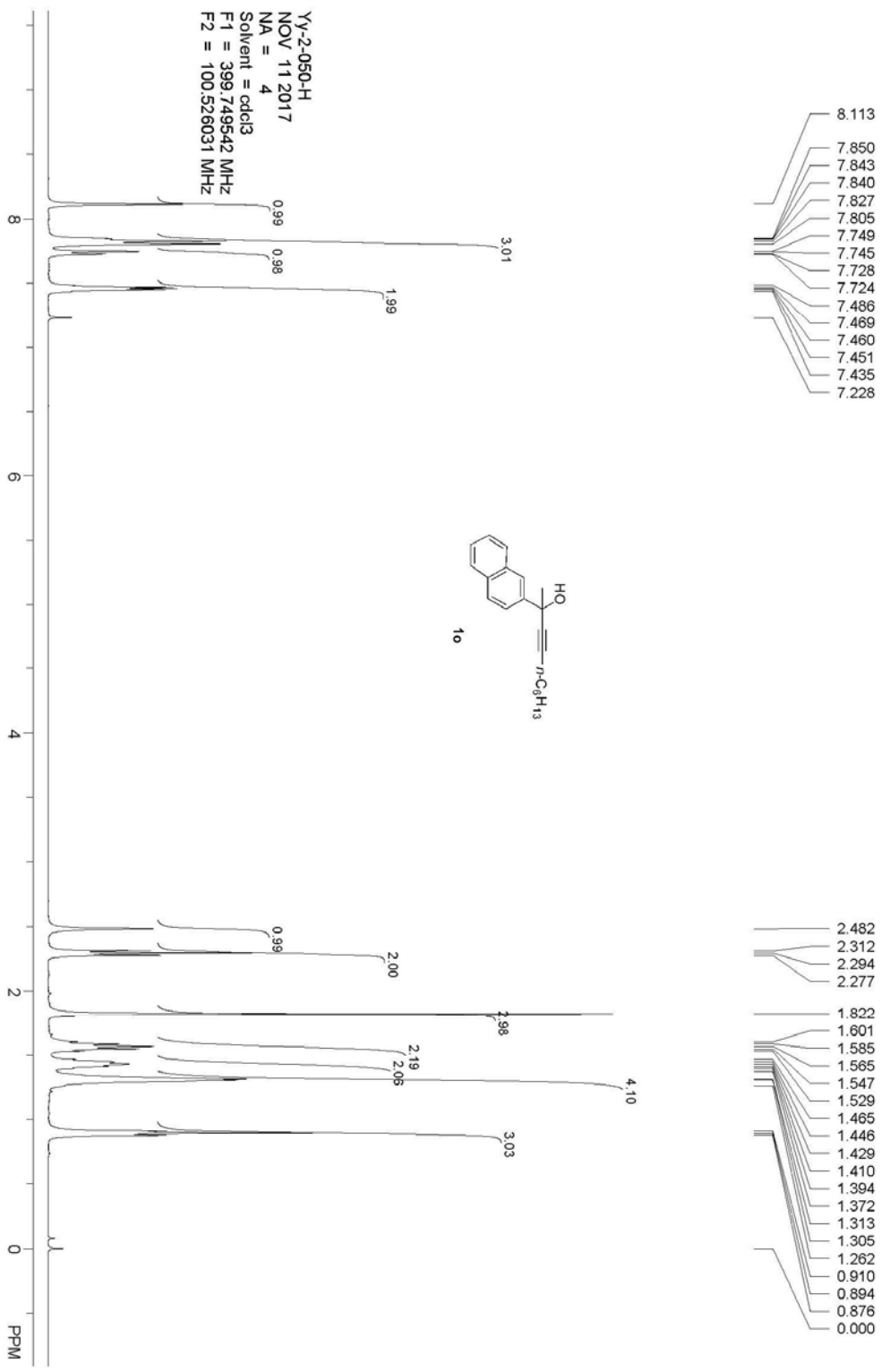
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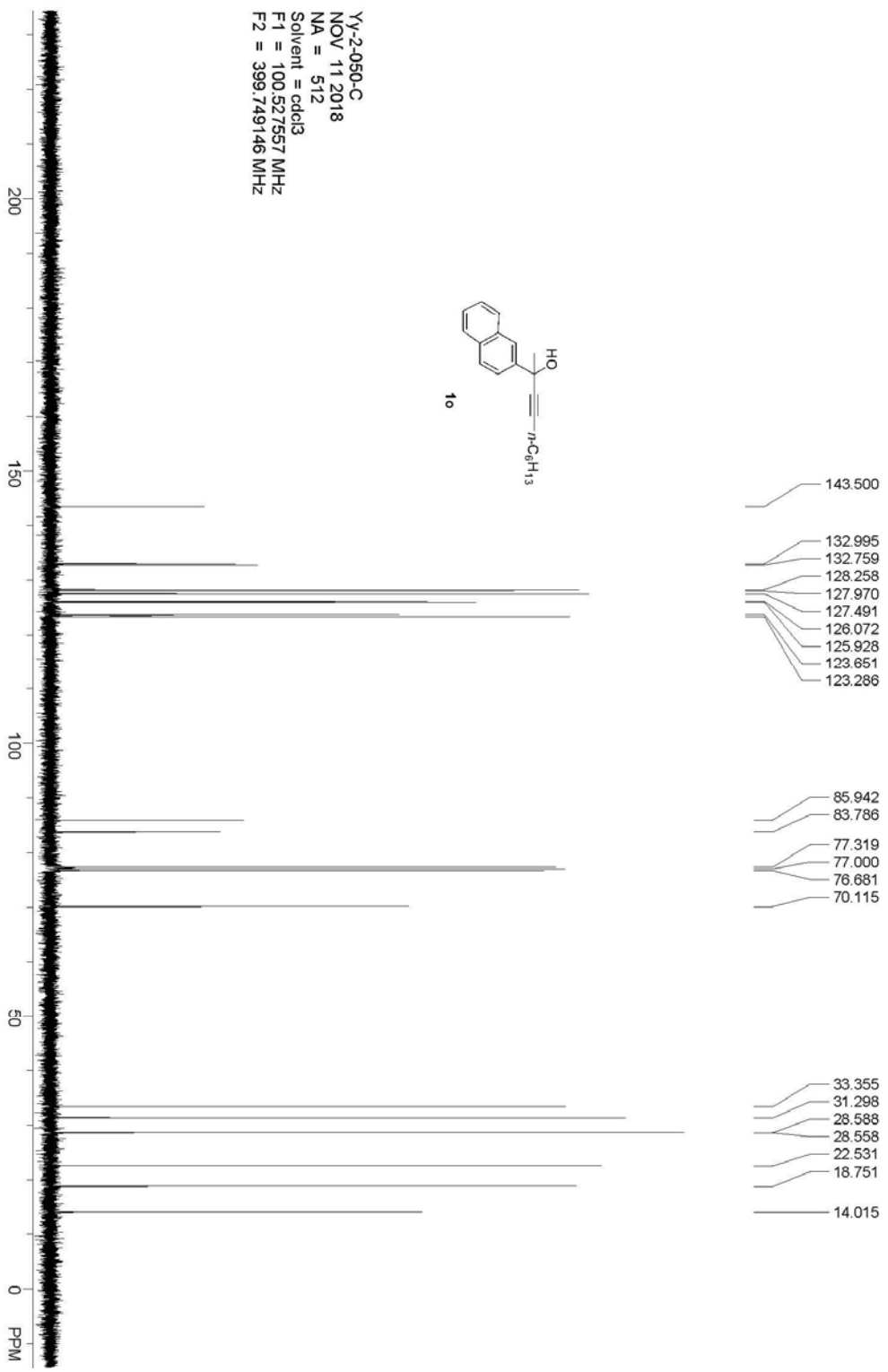
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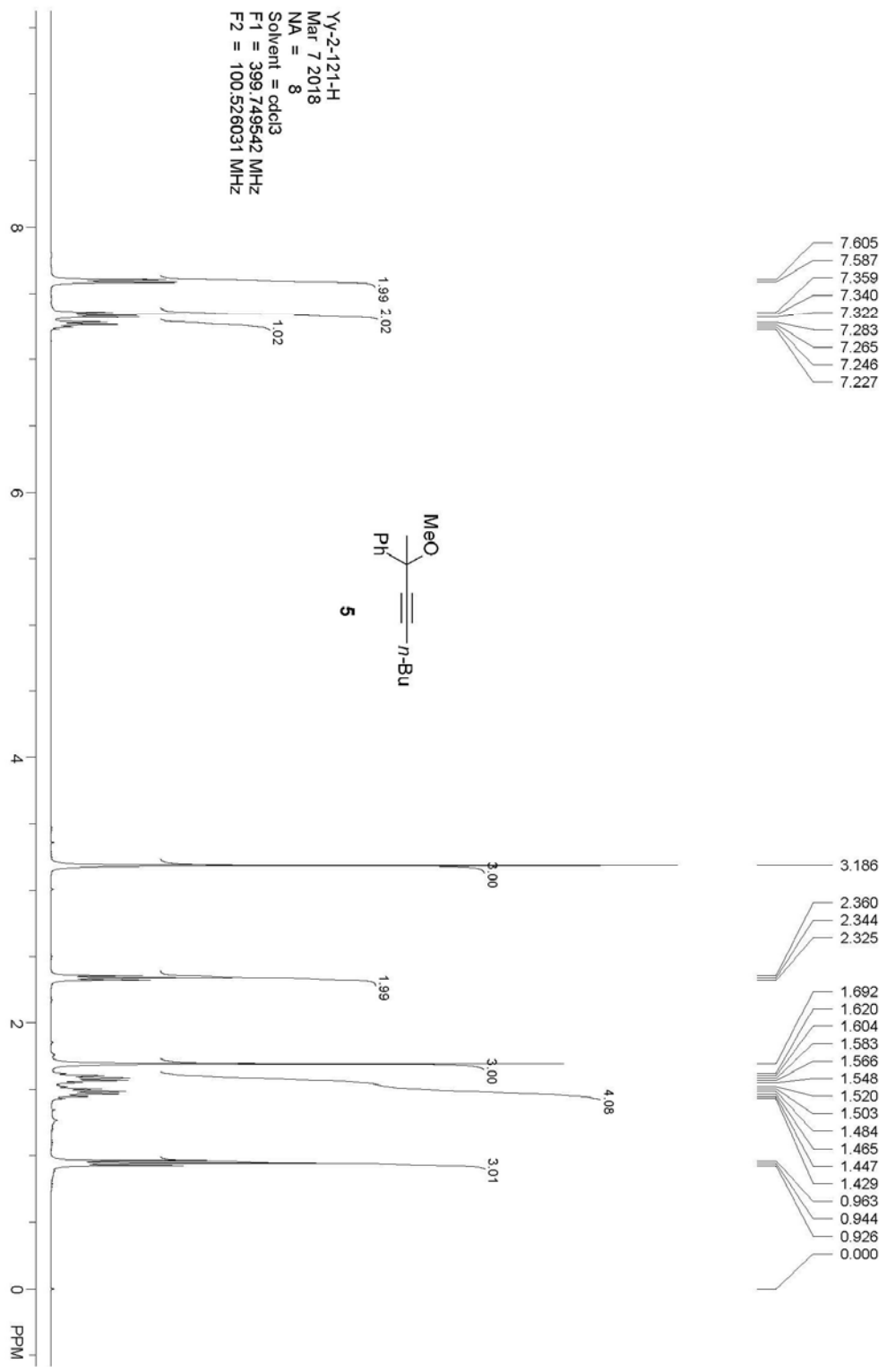
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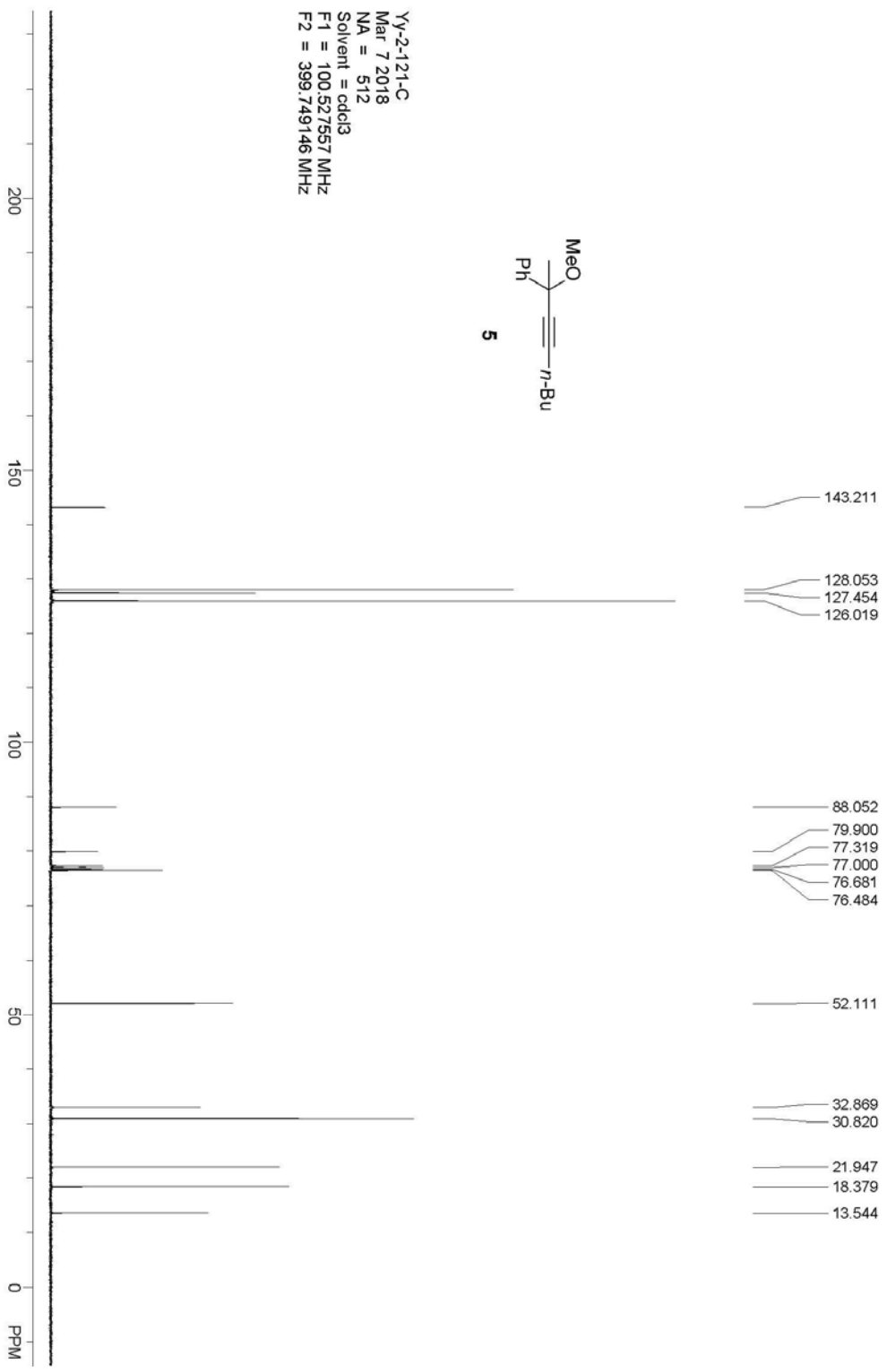
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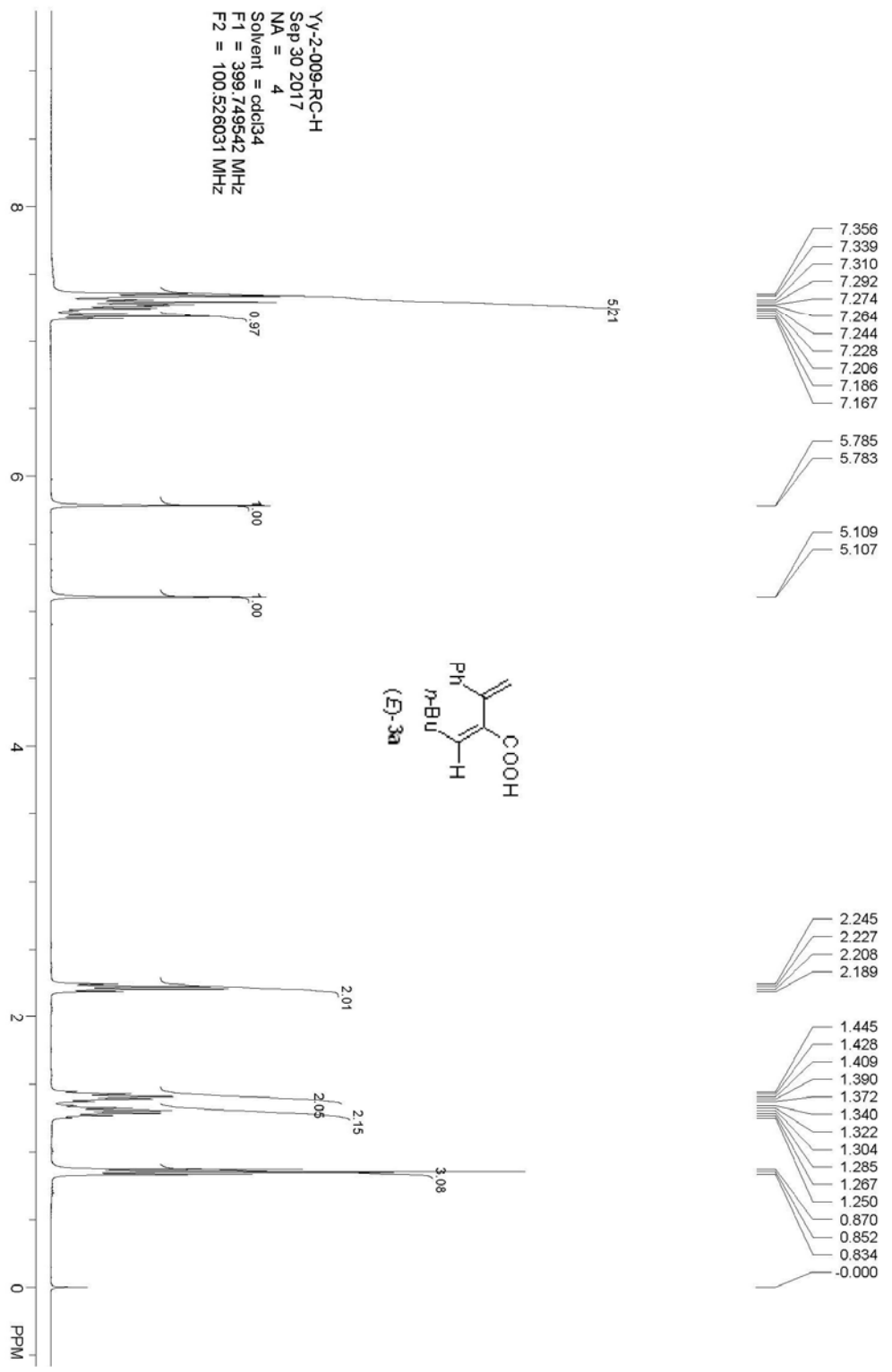
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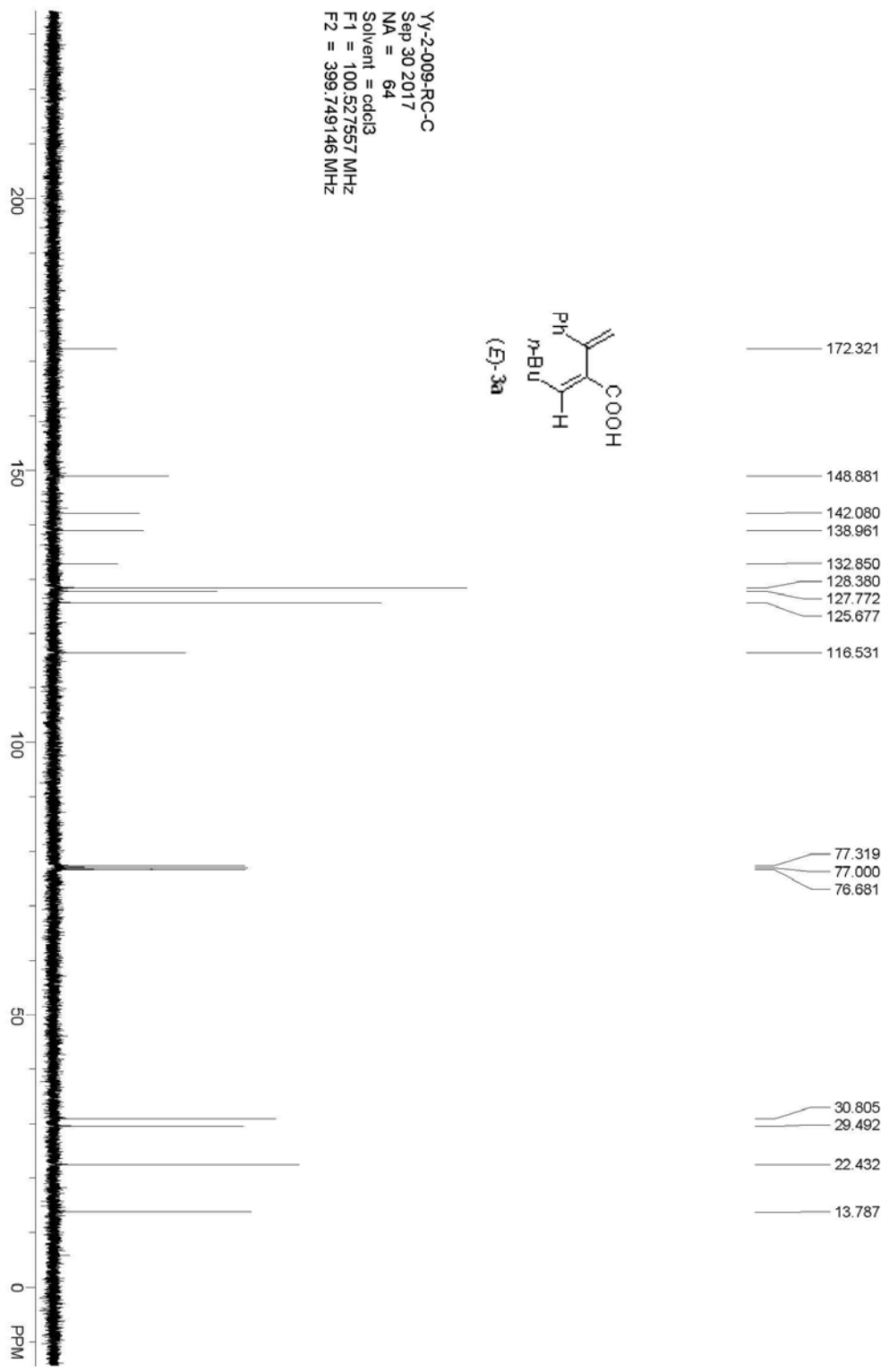
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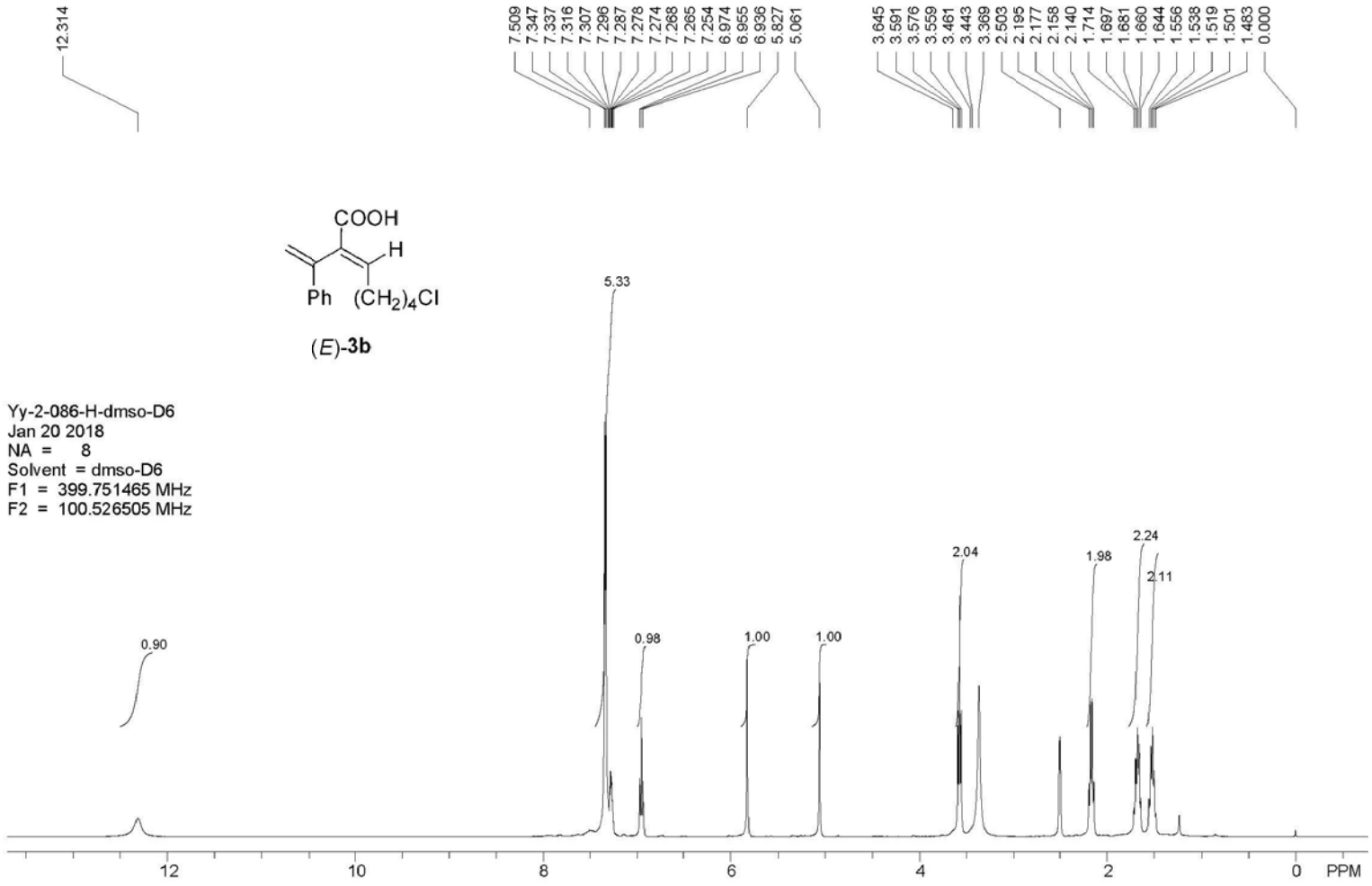
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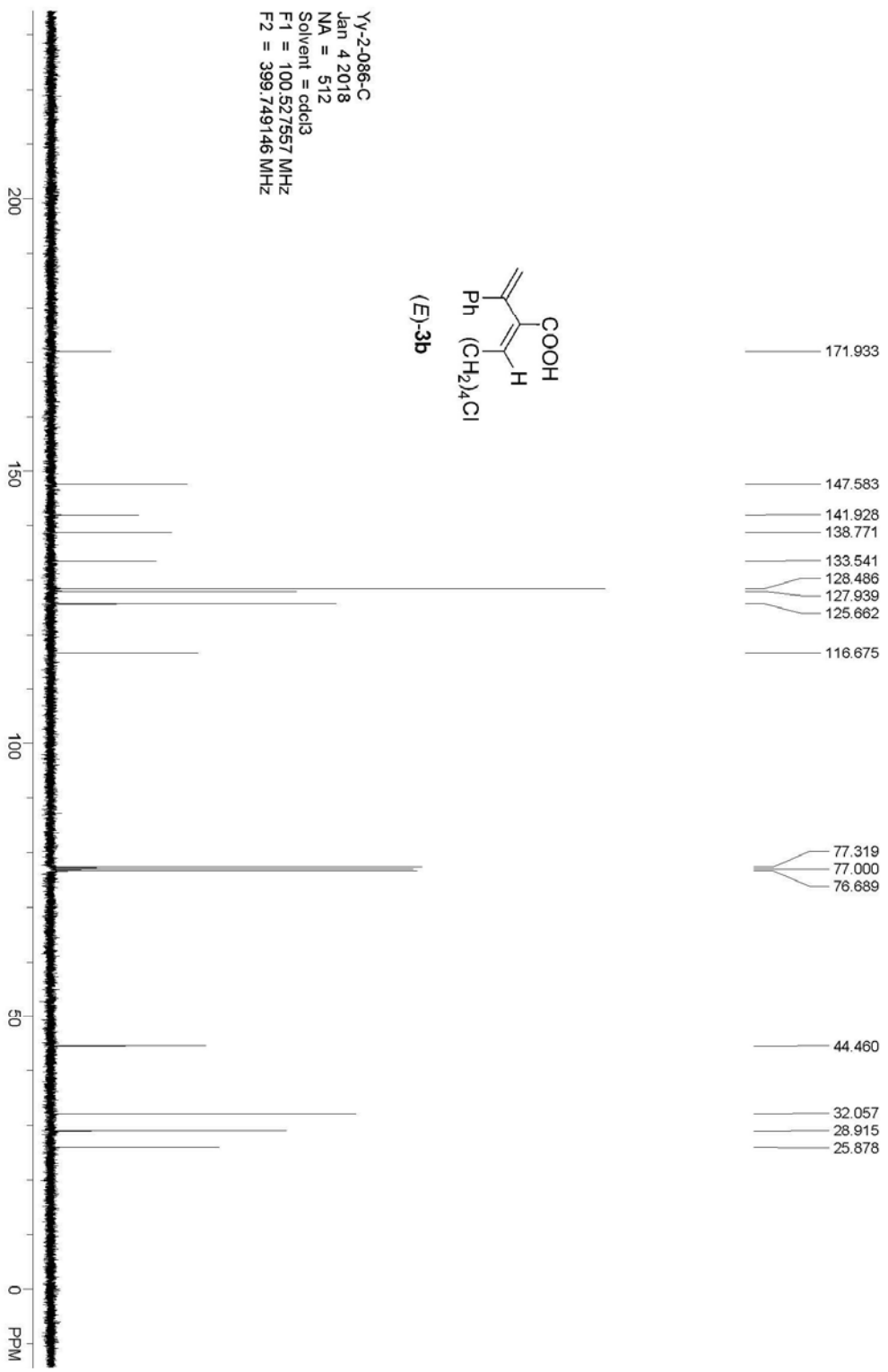
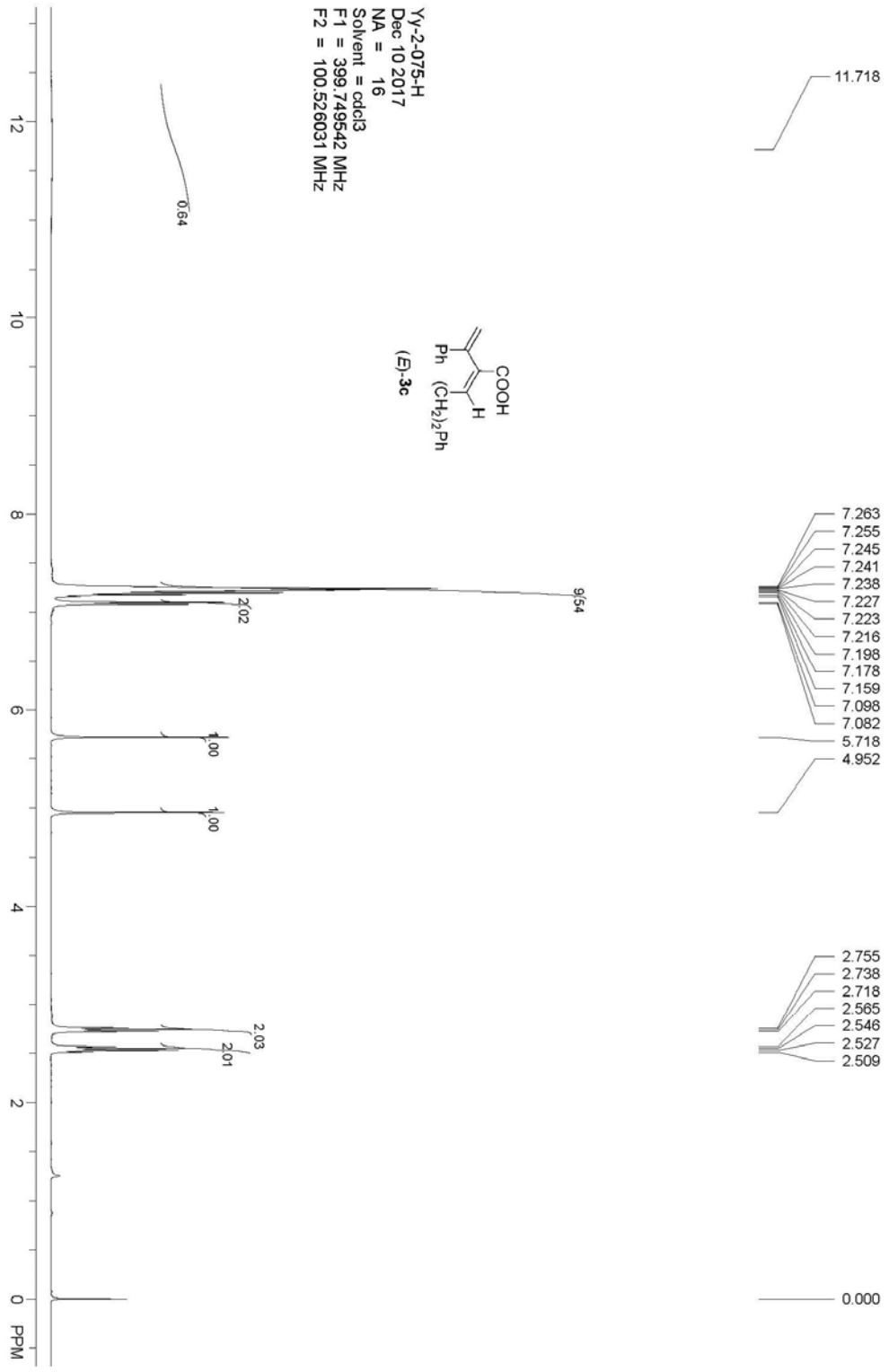
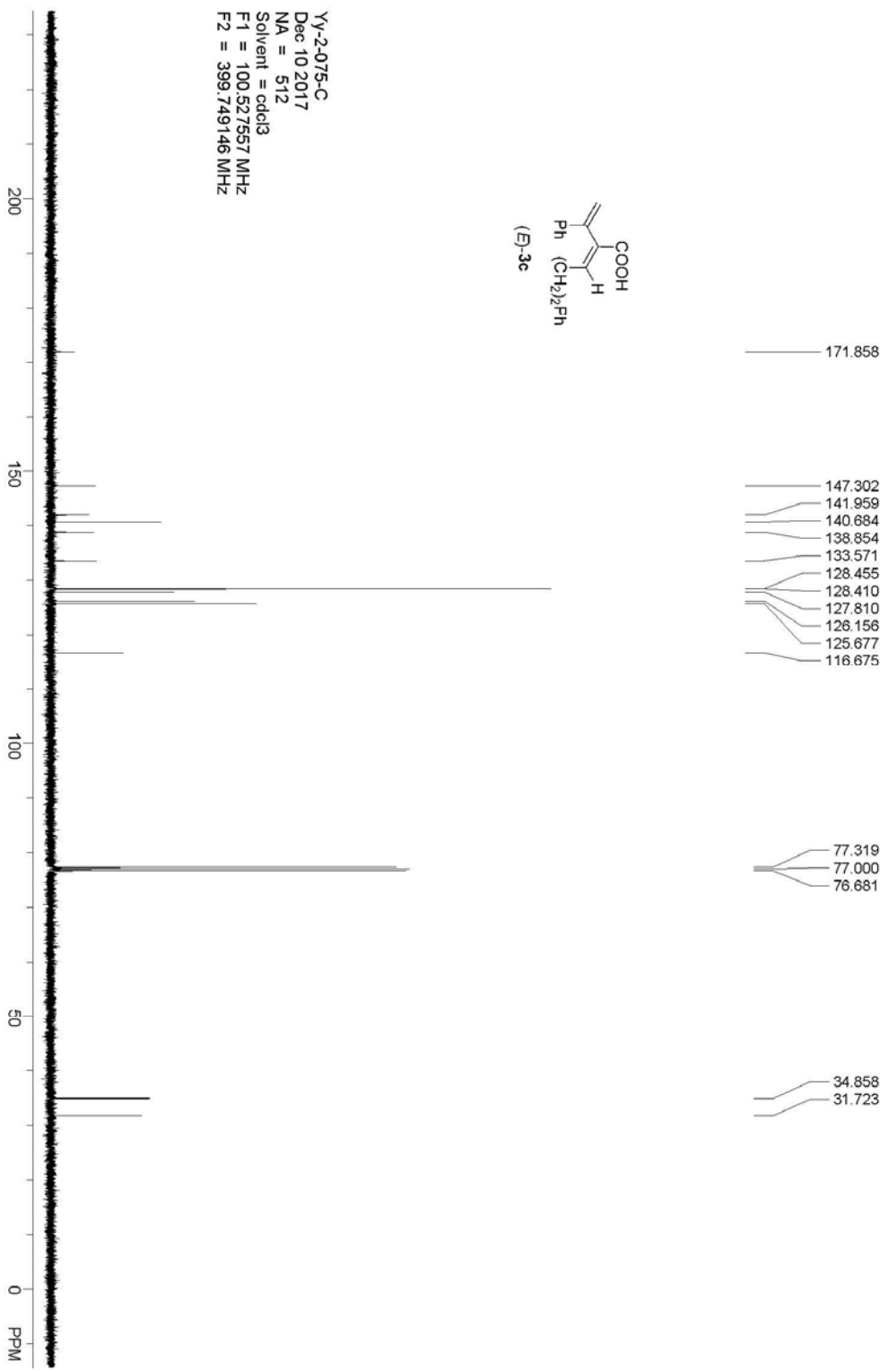
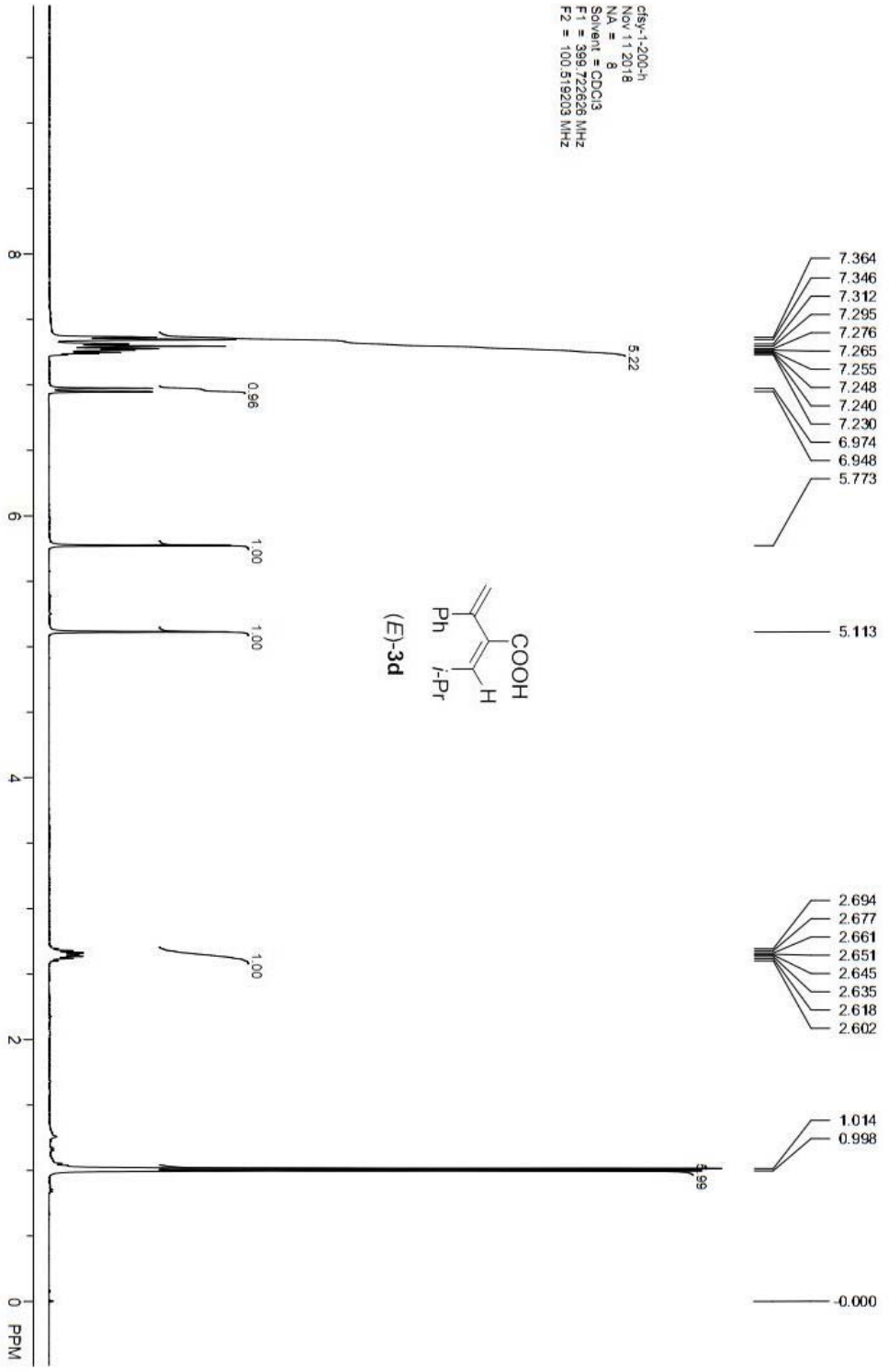
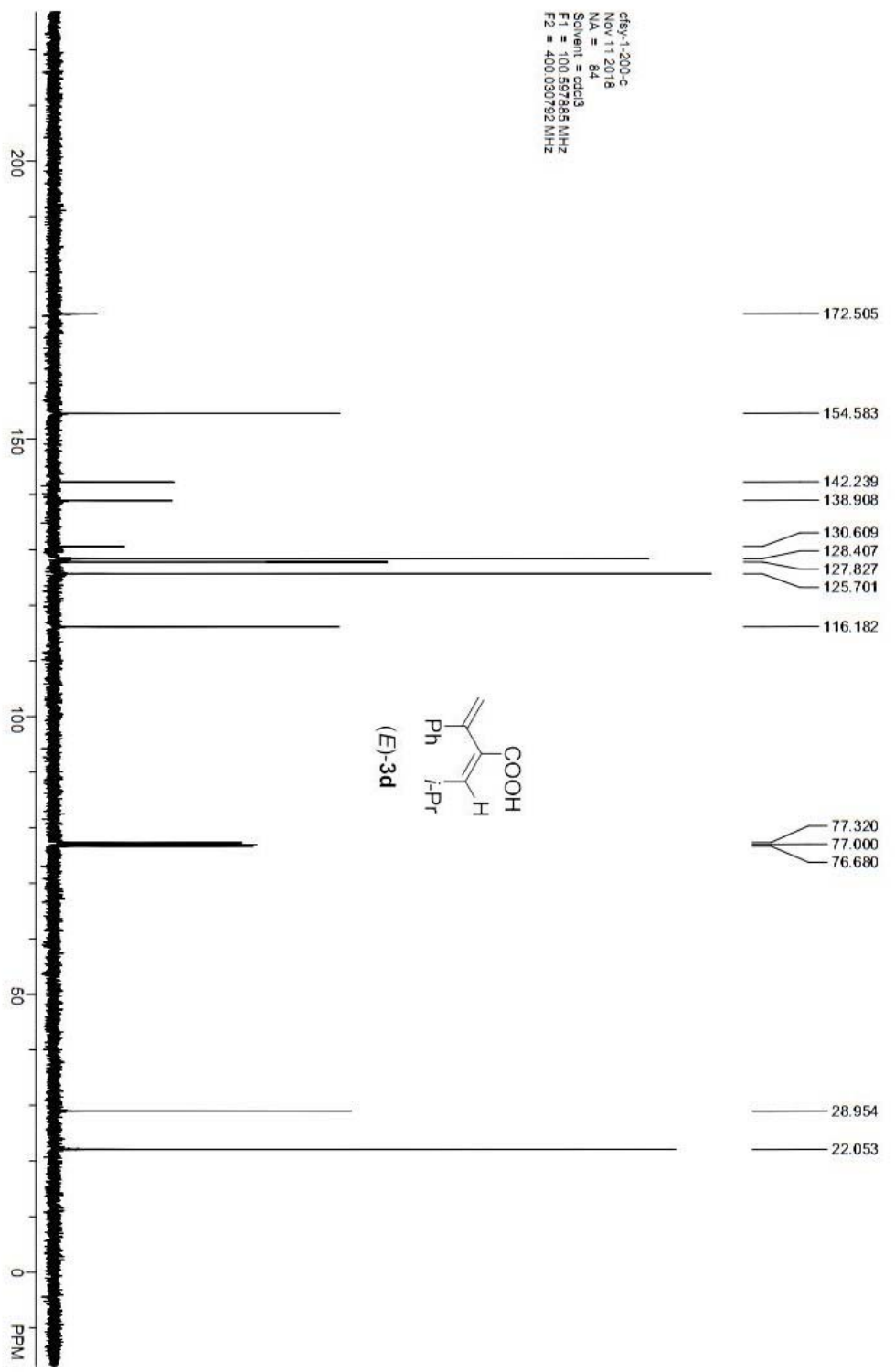
0
PPM

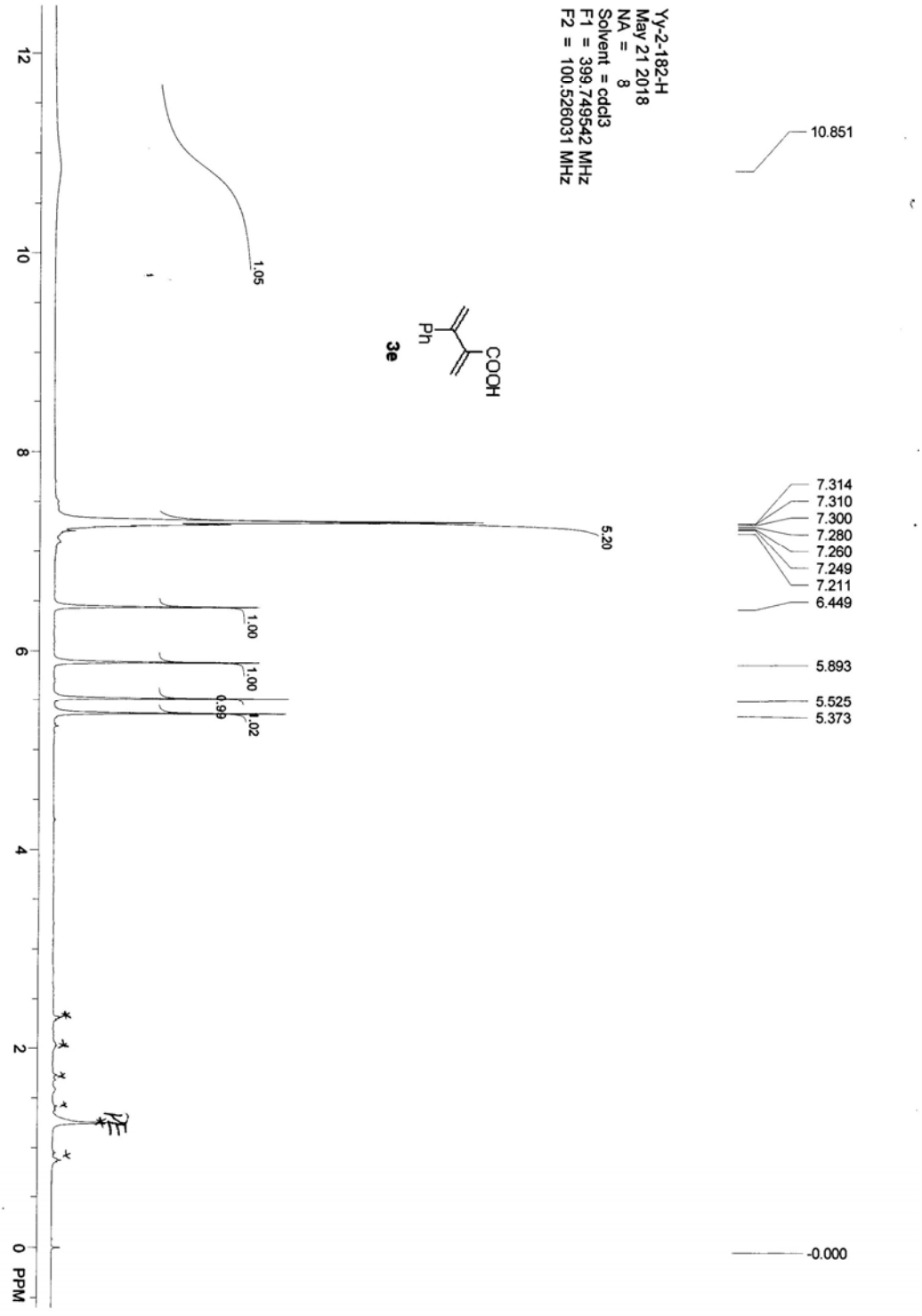

Yy-2-086-C
Jan 4 2018
NA = 512
Solvent = cdcl3
F1 = 100.527557 MHz
F2 = 399.749146 MHz
(E)-3b
COOH
Ph
H
(CH2)4Cl
171.933
147.583
141.928
138.771
133.541
128.486
127.939
125.662
116.675
77.319
77.000
76.689
44.460
32.057
28.915
25.878
200
150
100
50
0
PPM

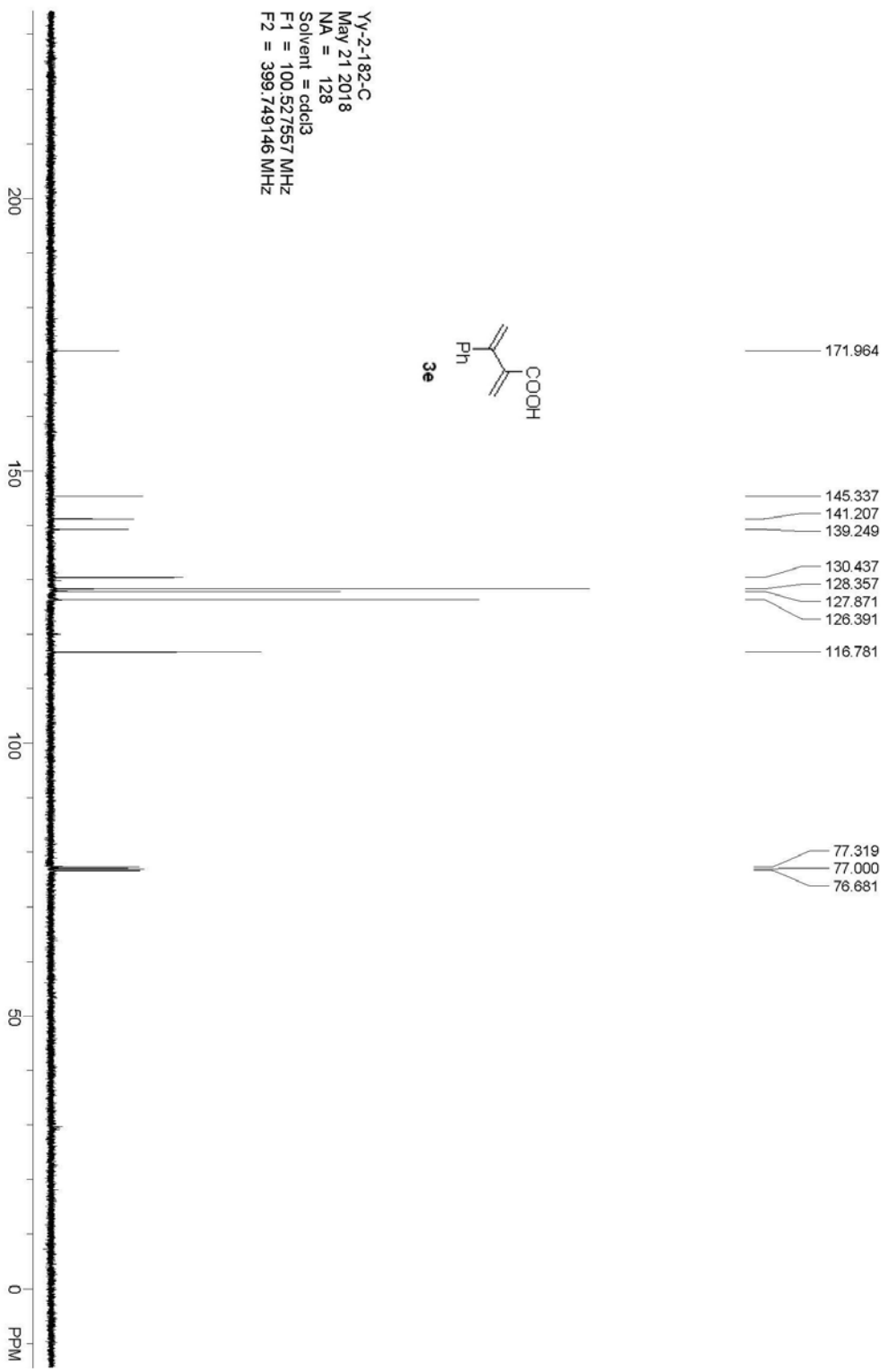

Yy-2-075-H
Dec 10 2017
NA = 16
Solvent = cdcl3
F1 = 399.749542 MHz
F2 = 100.526031 MHz
(E)-3c
COOH
H
Ph
(CH2)2Ph
11.718
7.263
7.255
7.245
7.241
7.238
7.227
7.223
7.216
7.198
7.178
7.159
7.098
7.082
5.718
4.952
2.755
2.738
2.718
2.565
2.546
2.527
2.509
0.000
0.64
9.54
2.02
1.00
1.00
2.03
2.01
12
10
8
6
4
2
0
PPM

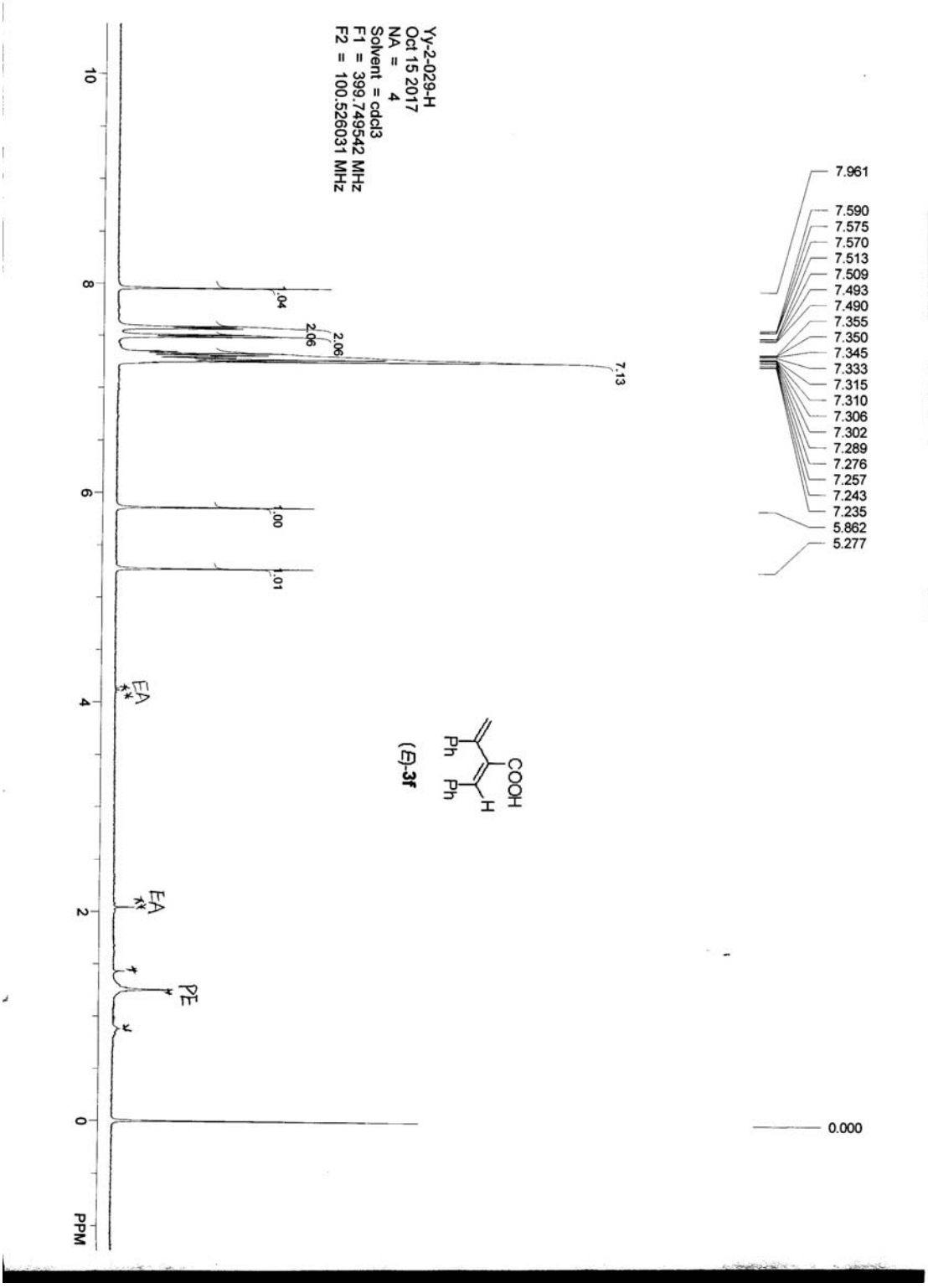

Yy-2-075-C
Dec 10 2017
NA = 512
Solvent = cdcl3
F1 = 100.527557 MHz
F2 = 399.749146 MHz
(E)-3c
COOH
H
Ph
(CH2)2Ph
171.858
147.302
141.959
140.684
138.854
133.571
128.455
128.410
127.810
126.156
125.677
116.675
77.319
77.000
76.681
34.858
31.723
200
150
100
50
0
PPM

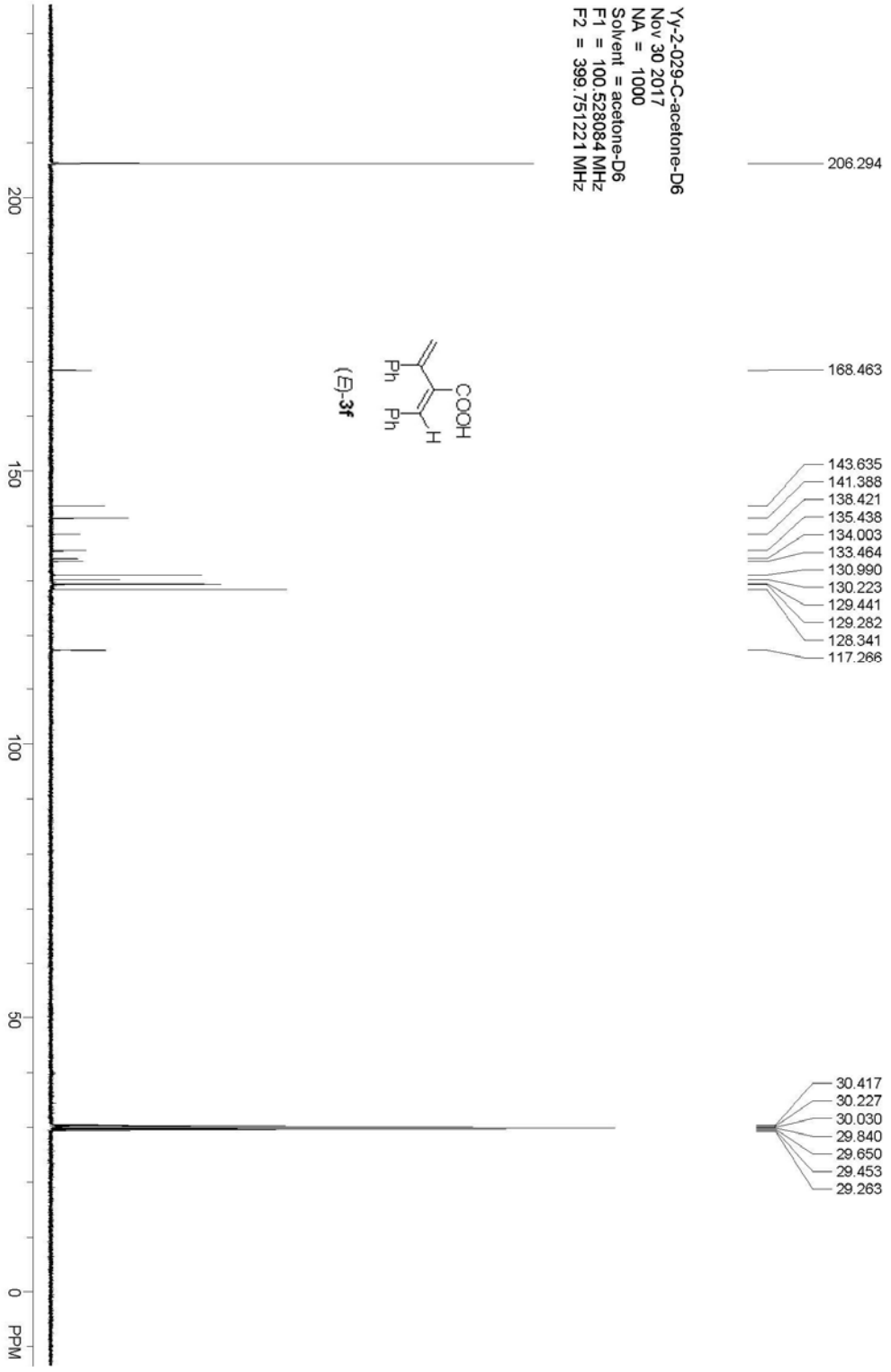

cfsy-1-200-h
Nov 11 2018
NA = 8
Solvent = CDCl3
F1 = 399.722626 MHz
F2 = 100.519203 MHz
7.364
7.346
7.312
7.295
7.276
7.265
7.255
7.248
7.240
7.230
6.974
6.948
5.773
5.113
2.694
2.677
2.661
2.651
2.645
2.635
2.618
2.602
1.014
0.998
0.000
5.22
0.96
1.00
1.00
1.00
5.99
COOH
H
Ph
i-Pr
(E)-3d
8
6
4
2
0
PPM

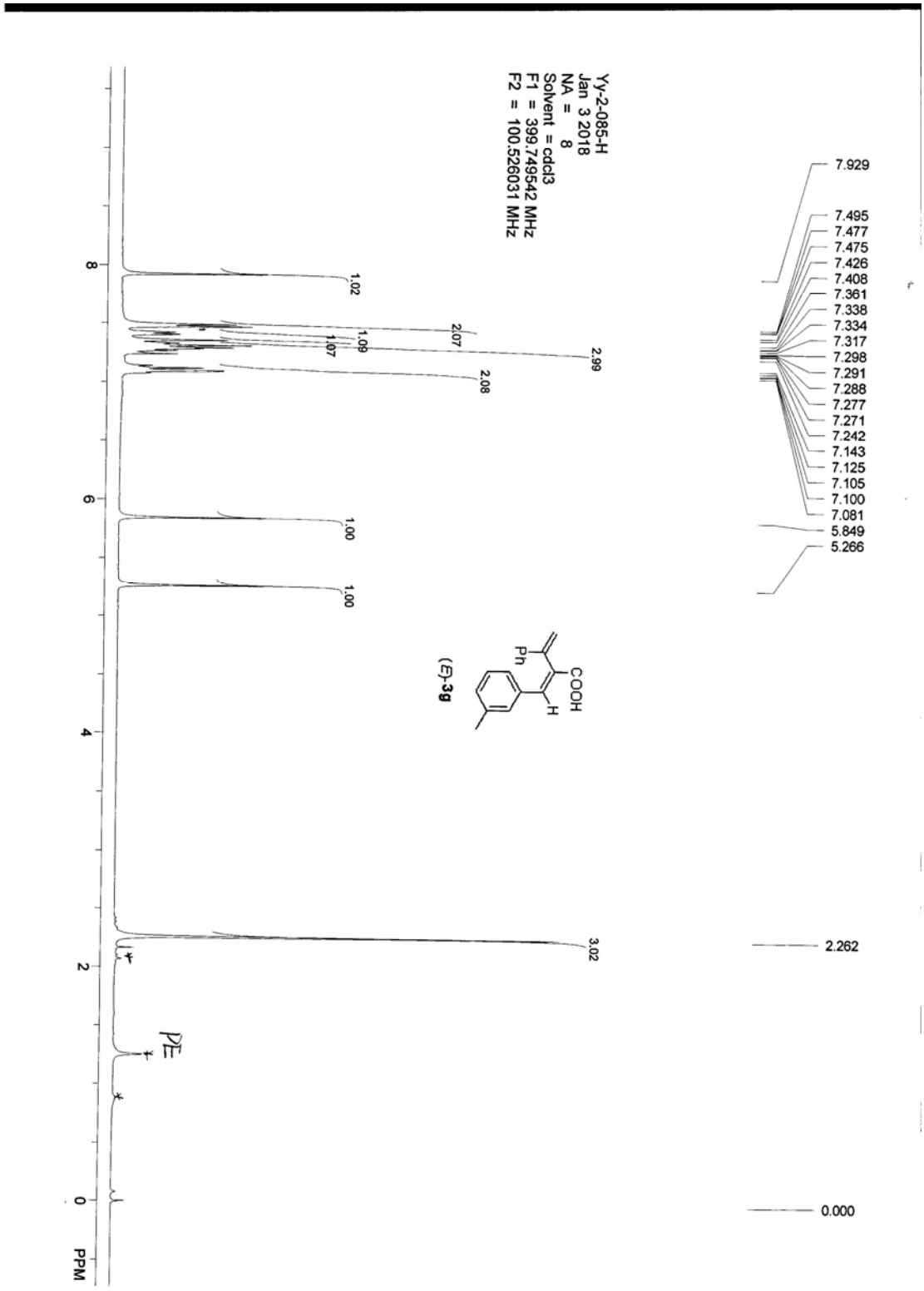

cfsy-1-200-c
Nov 11 2018
NA = 84
Solvent = cdcl3
F1 = 100.597685 MHz
F2 = 400.030792 MHz
172.505
154.583
142.239
138.908
130.609
128.407
127.827
125.701
116.182
77.320
77.000
76.680
28.954
22.053
200
150
100
50
0
PPM
COOH
Ph
i-Pr
H
(E)-3d

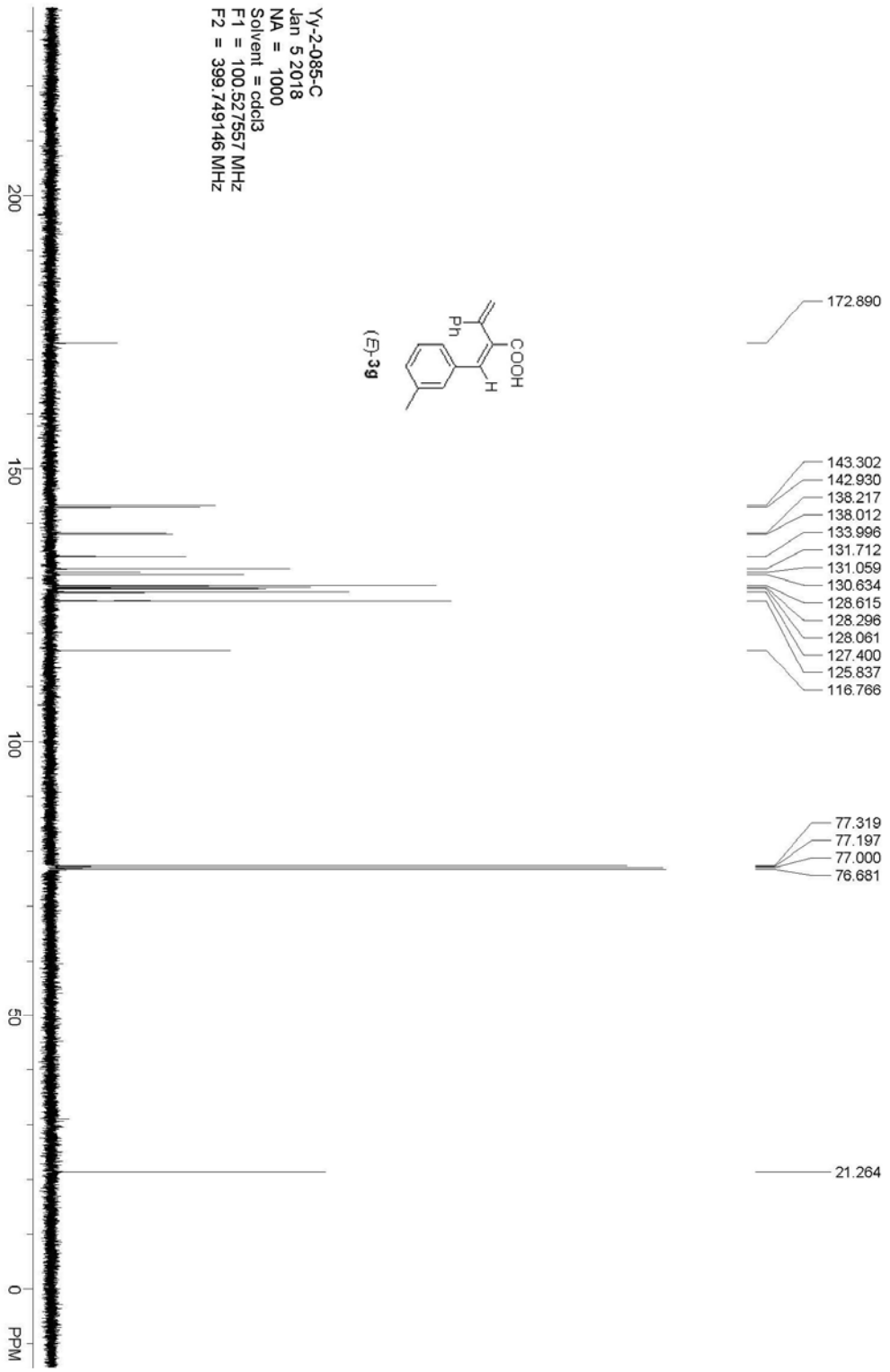

Yy-2-182-H
May 21 2018
NA = 8
Solvent = cdcl3
F1 = 399.749542 MHz
F2 = 100.526031 MHz
3e
Ph
COOH
10.851
7.314
7.310
7.300
7.280
7.260
7.249
7.211
6.449
5.893
5.525
5.373
-0.000
1.05
5.20
1.00
1.00
0.99
1.02
12
10
8
6
4
2
0 PPM

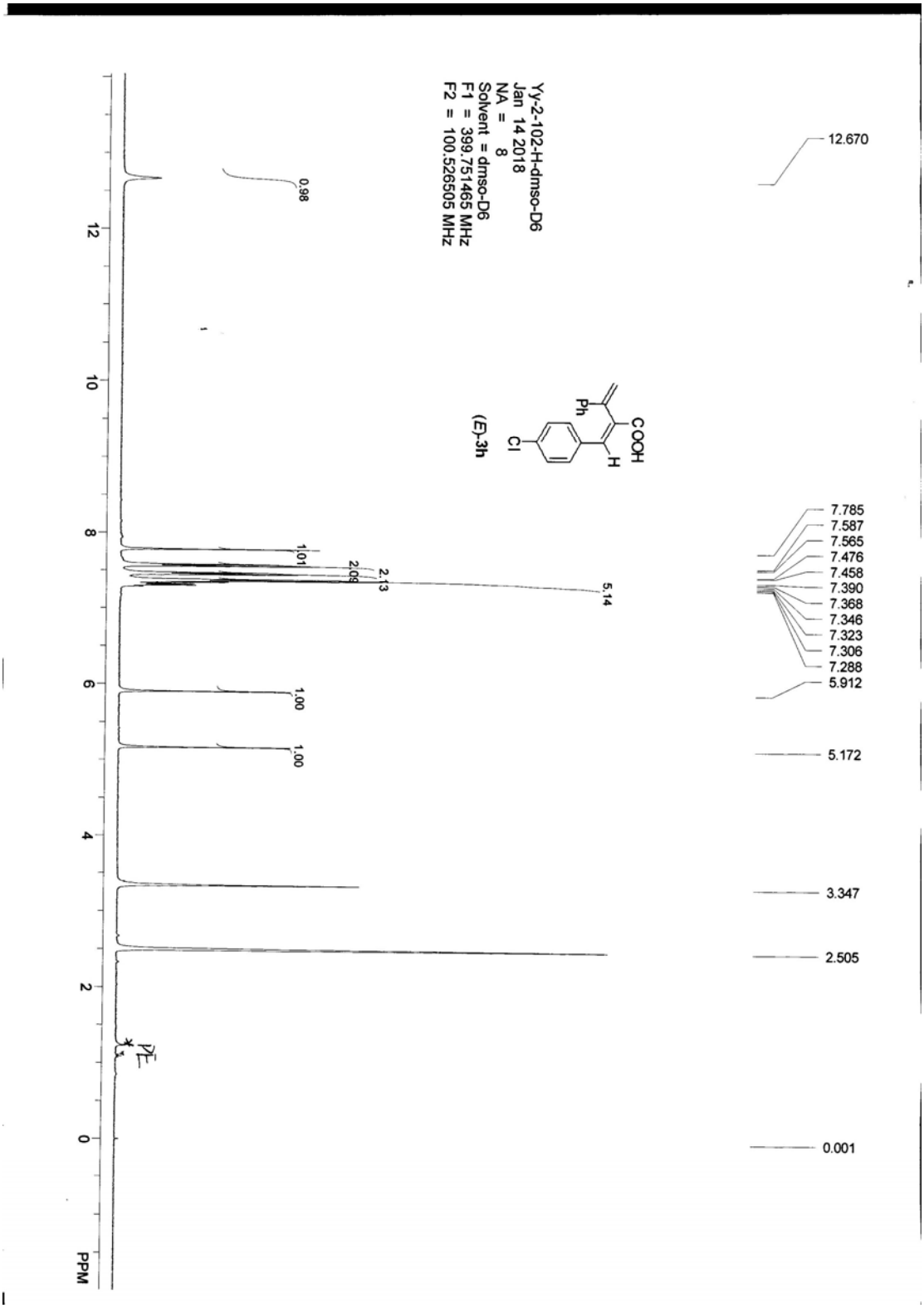

Yy-2-182-C
May 21 2018
NA = 128
Solvent = cdcl3
F1 = 100.527557 MHz
F2 = 399.749146 MHz
Ph
COOH
3e
171.964
145.337
141.207
139.249
130.437
128.357
127.871
126.391
116.781
77.319
77.000
76.681
200
150
100
50
0
PPM

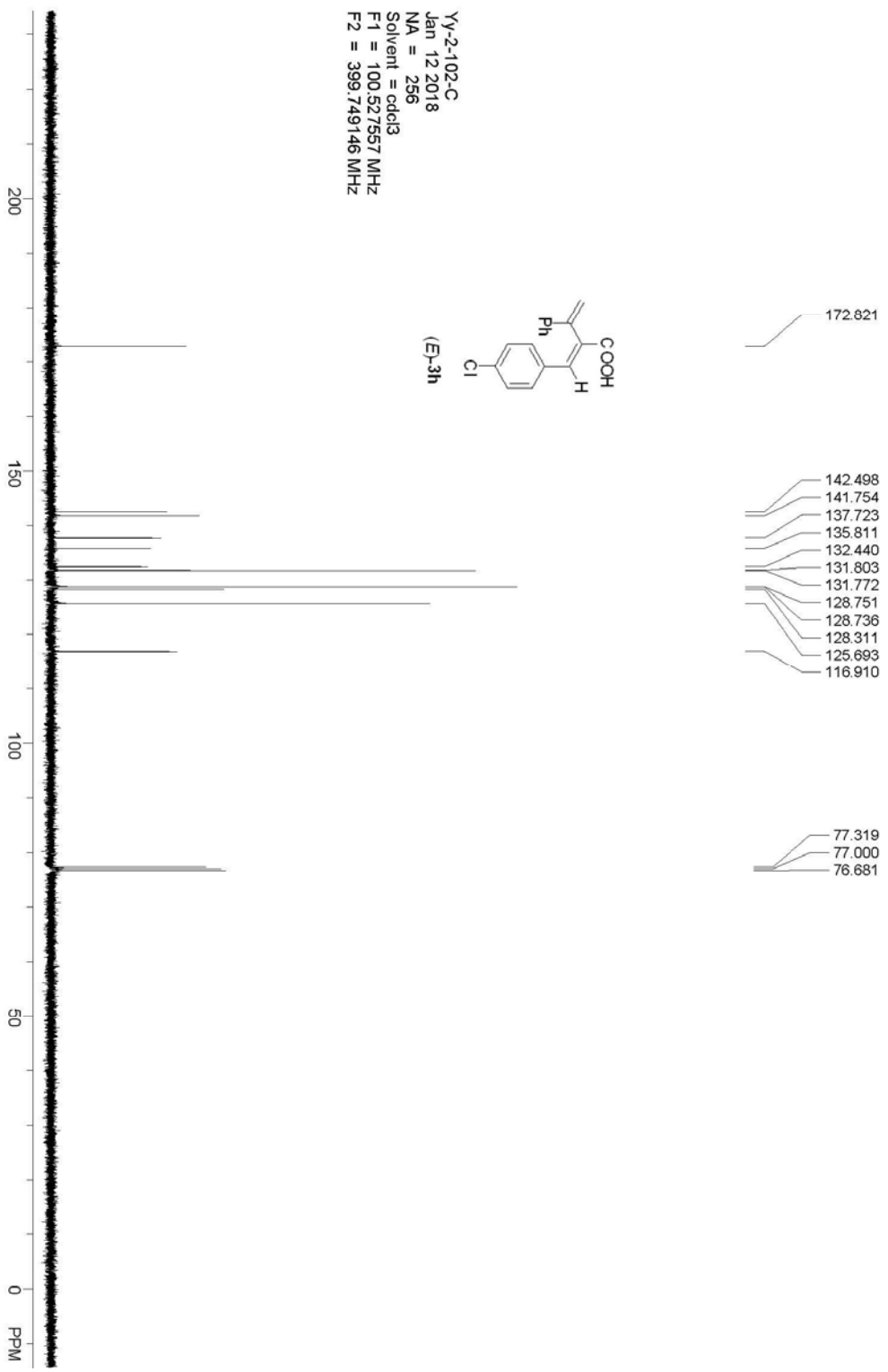

Yy-2-029-H
Oct 15 2017
NA = 4
Solvent = cdcl3
F1 = 399.749542 MHz
F2 = 100.526031 MHz
1.04
2.06
2.06
7.13
1.00
1.01
EA
EA
PE
PPM
(E)-3f
Ph
Ph
COOH
H
7.961
7.590
7.575
7.570
7.513
7.509
7.493
7.490
7.355
7.350
7.345
7.333
7.315
7.310
7.306
7.302
7.289
7.276
7.257
7.243
7.235
5.862
5.277
0.000

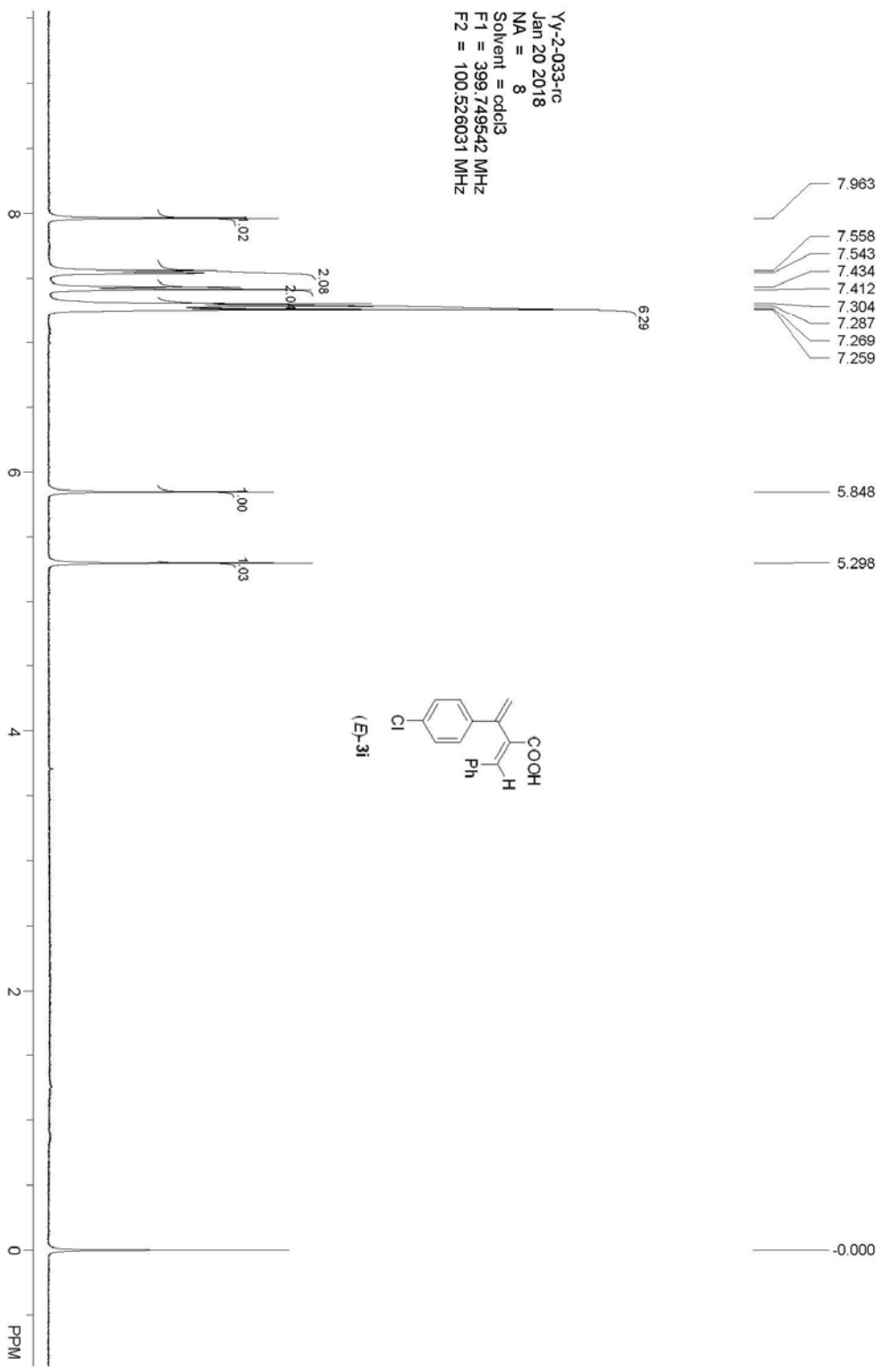

Yy-2-029-C-acetone-D6
Nov 30 2017
NA = 1000
Solvent = acetone-D6
F1 = 100.528084 MHz
F2 = 399.751221 MHz
(E)-3f
Ph
Ph
COOH
H
206.294
168.463
143.635
141.388
138.421
135.438
134.003
133.464
130.990
130.223
129.441
129.282
128.341
117.266
30.417
30.227
30.030
29.840
29.650
29.453
29.263
200
150
100
50
0
PPM

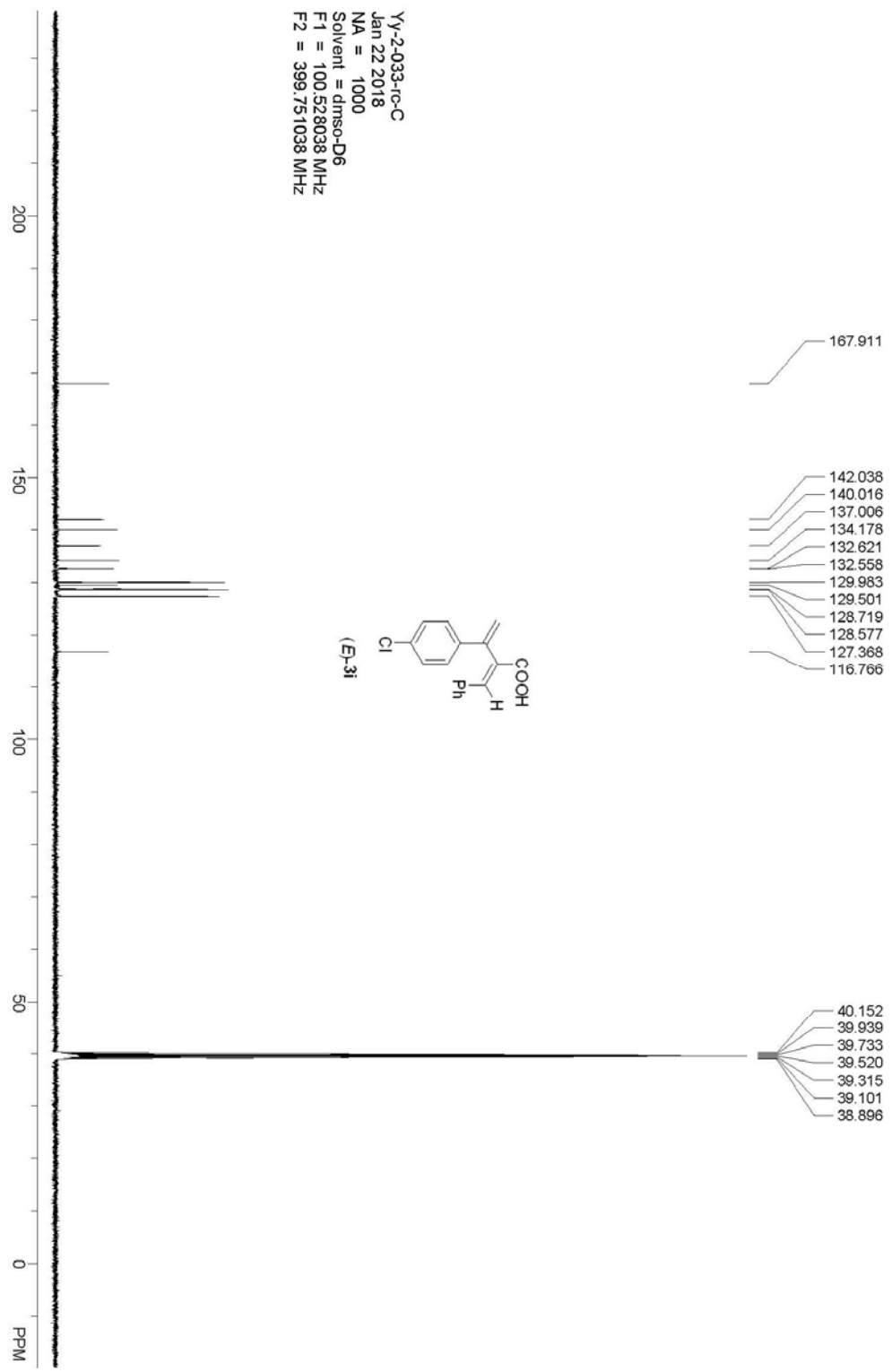

Yy-2-085-H
Jan 3 2018
NA = 8
Solvent = cdcl3
F1 = 399.749542 MHz
F2 = 100.526031 MHz
7.929
7.495
7.477
7.475
7.426
7.408
7.361
7.338
7.334
7.317
7.298
7.291
7.288
7.277
7.271
7.242
7.143
7.125
7.105
7.100
7.081
5.849
5.266
2.262
0.000
1.02
2.07
1.09
1.07
2.99
2.08
1.00
1.00
3.02
Ph
COOH
H
(E)-3g
PE
PPM

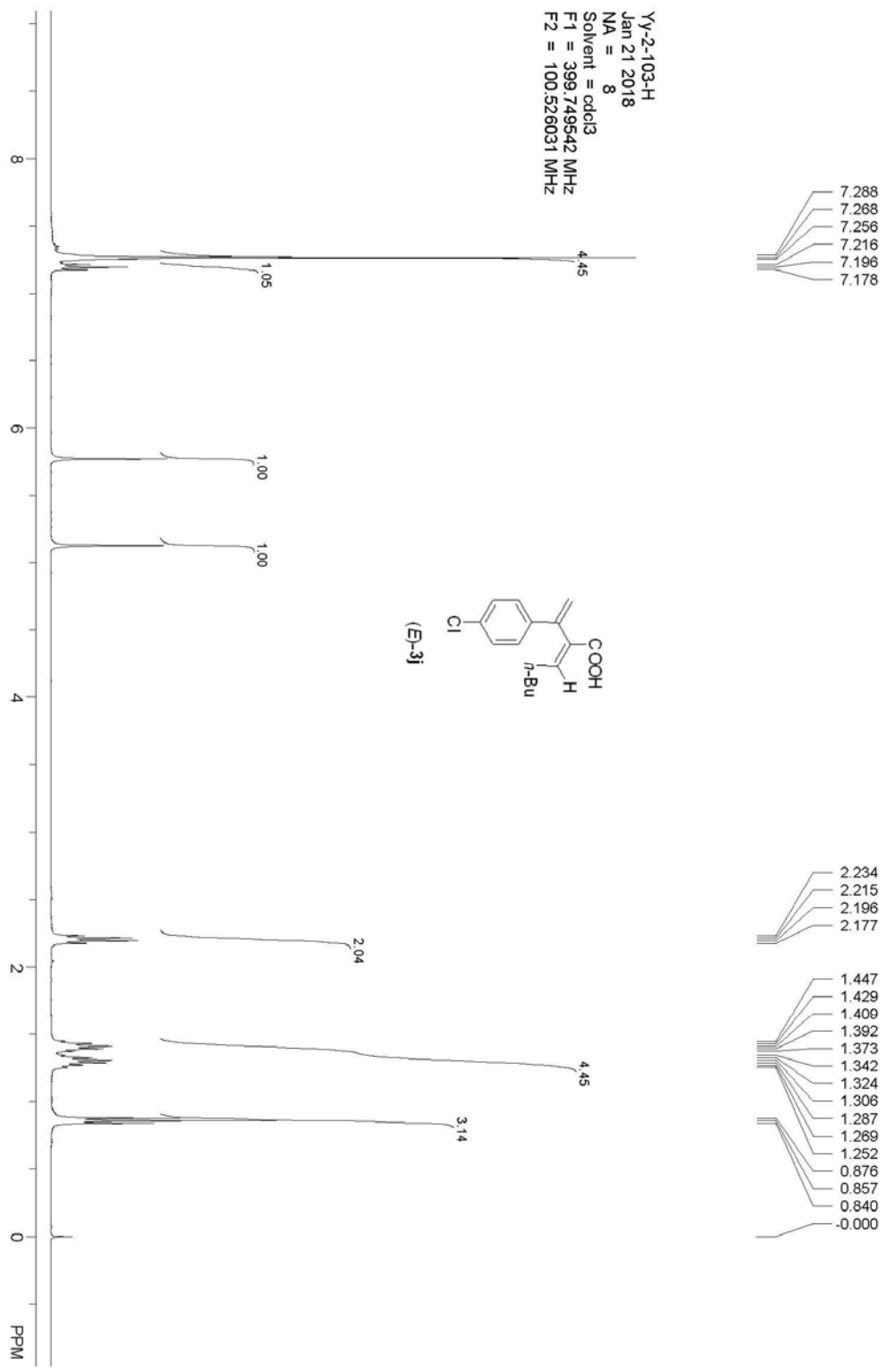

Yy-2-085-C
Jan 5 2018
NA = 1000
Solvent = cdcl3
F1 = 100.527557 MHz
F2 = 399.749146 MHz
(E)-3g
Ph
COOH
H
172.890
143.302
142.930
138.217
138.012
133.996
131.712
131.059
130.634
128.615
128.296
128.061
127.400
125.837
116.766
77.319
77.197
77.000
76.681
21.264
200
150
100
50
0
PPM

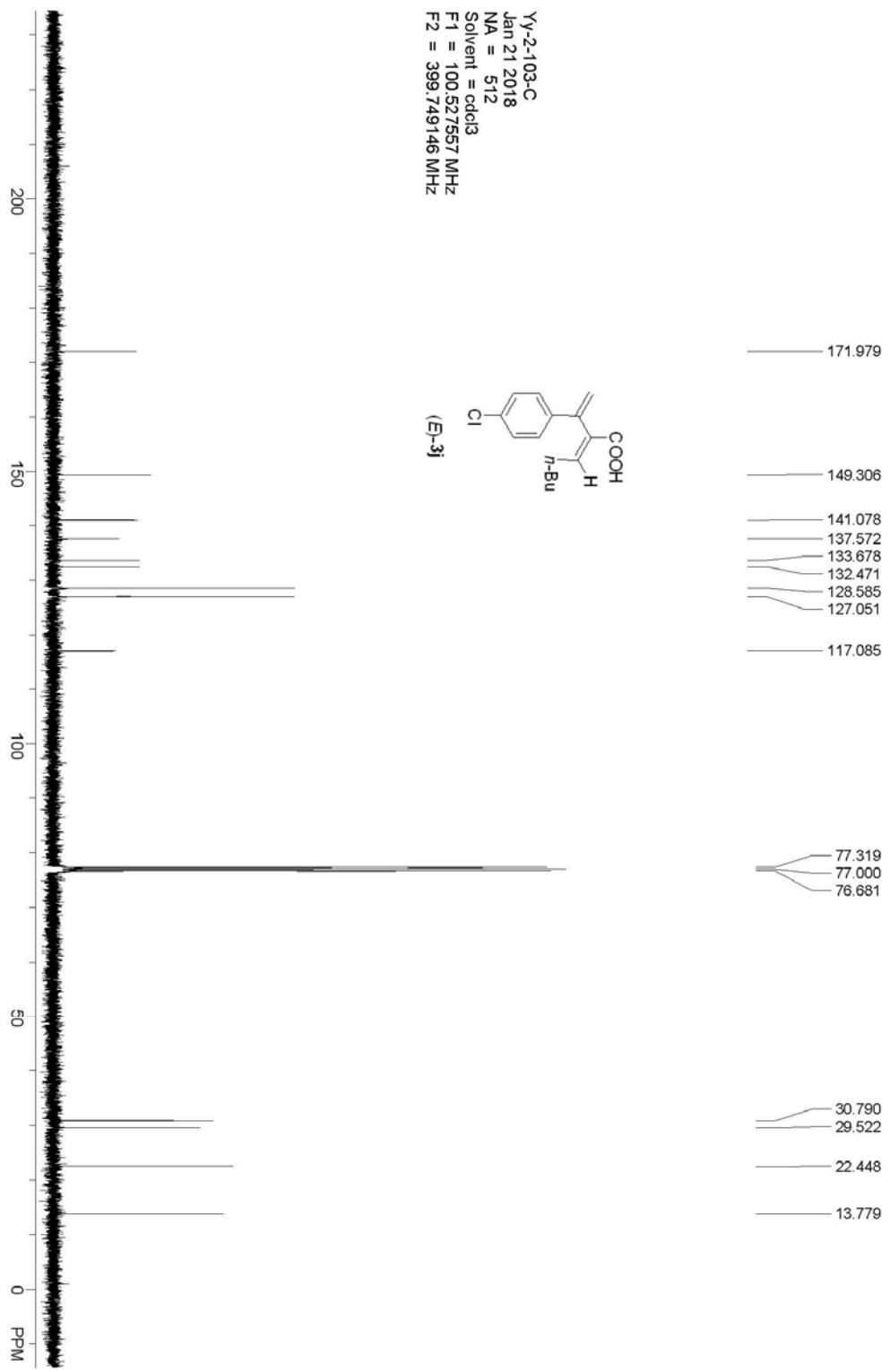

Yy-2-102-H-dmso-D6
Jan 14 2018
NA = 8
Solvent = dmso-D6
F1 = 399.751465 MHz
F2 = 100.526505 MHz
(E)-3h
Ph
COOH
Cl
H
12.670
7.785
7.587
7.565
7.476
7.458
7.390
7.368
7.346
7.323
7.306
7.288
5.912
5.172
3.347
2.505
0.001
0.98
1.01
2.09
2.13
5.14
1.00
1.00
PE
PPM

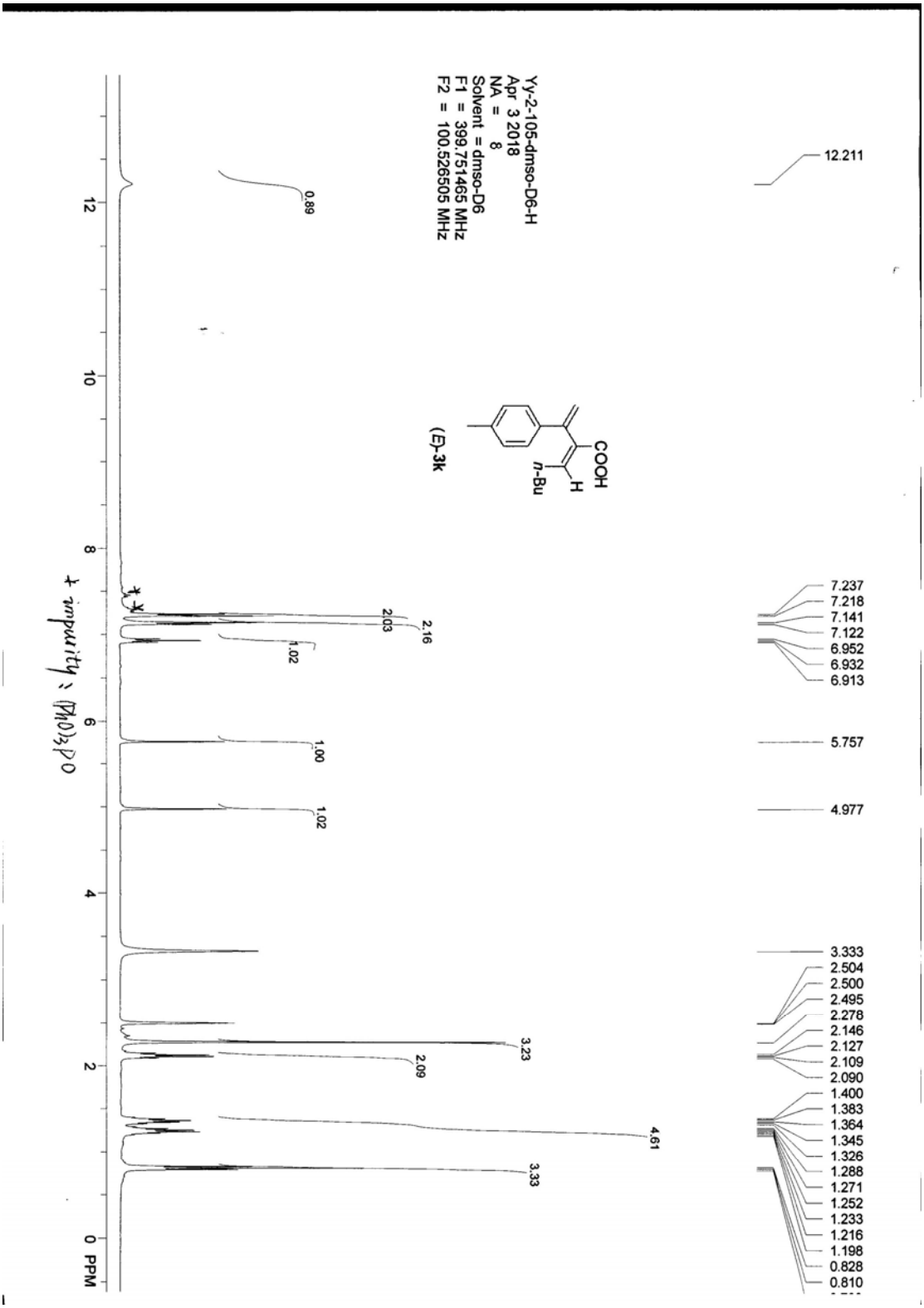

Yy-2-102-C
Jan 12 2018
NA = 256
Solvent = cdcl3
F1 = 100.527557 MHz
F2 = 399.749146 MHz
(E)-3h
Ph
COOH
Cl
H
172.821
142.498
141.754
137.723
135.811
132.440
131.803
131.772
128.751
128.736
128.311
125.693
116.910
77.319
77.000
76.681
200
150
100
50
0
PPM

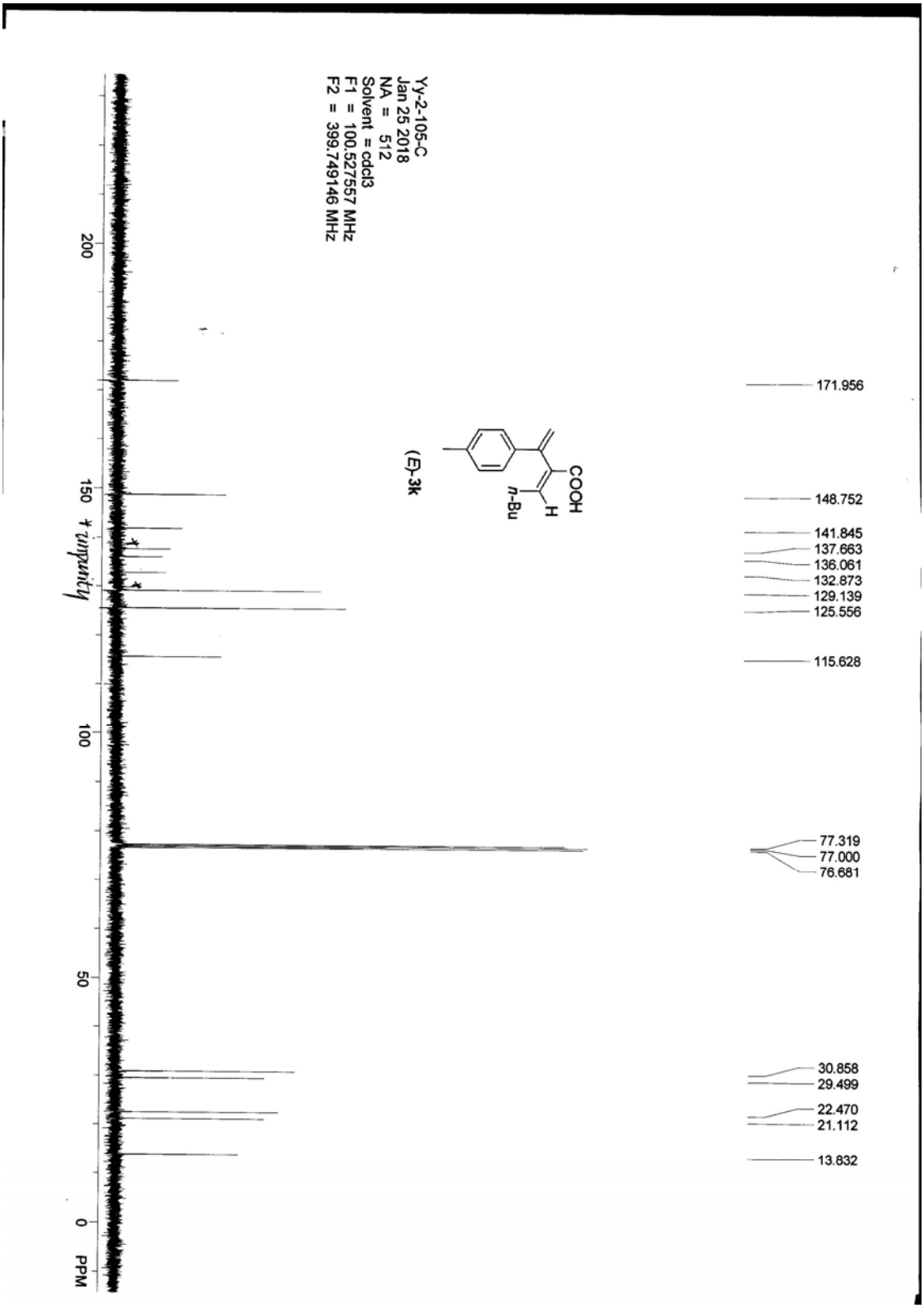

Yy-2-033-rc
Jan 20 2018
NA = 8
Solvent = cdcl3
F1 = 399.749542 MHz
F2 = 100.526031 MHz
7.963
7.558
7.543
7.434
7.412
7.304
7.287
7.269
7.259
5.848
5.298
-0.000
1.02
2.08
2.04
6.29
1.00
1.03
8
6
4
2
0
PPM
Cl
Ph
COOH
H
(E)-3i

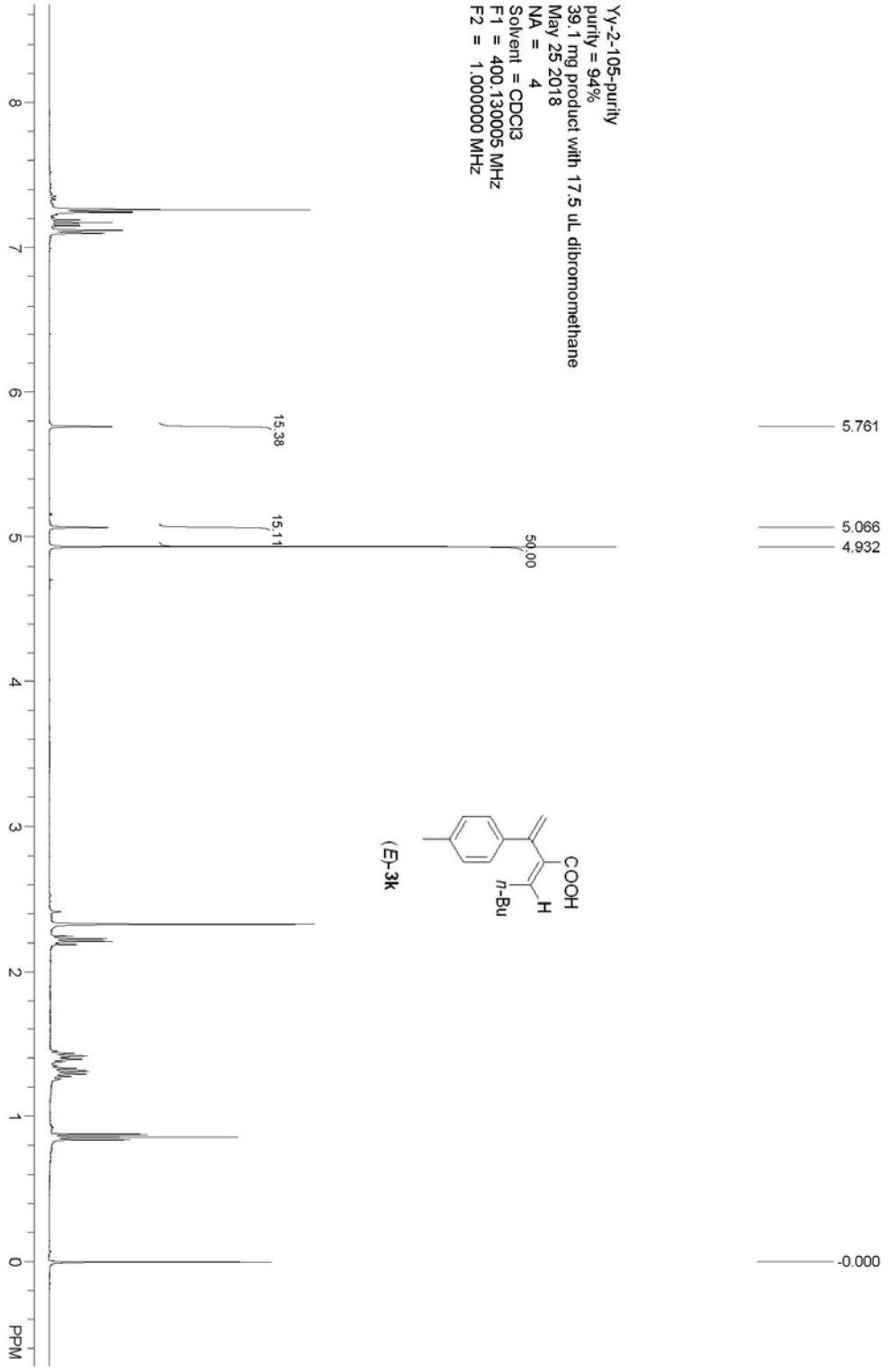

Yy-2-033-rc-C
Jan 22 2018
NA = 1000
Solvent = dmso-D6
F1 = 100.528038 MHz
F2 = 399.751038 MHz
(E)-3i
Cl
Ph
H
COOH
167.911
142.038
140.016
137.006
134.178
132.621
132.558
129.983
129.501
128.719
128.577
127.368
116.766
40.152
39.939
39.733
39.520
39.315
39.101
38.896
200
150
100
50
0
PPM

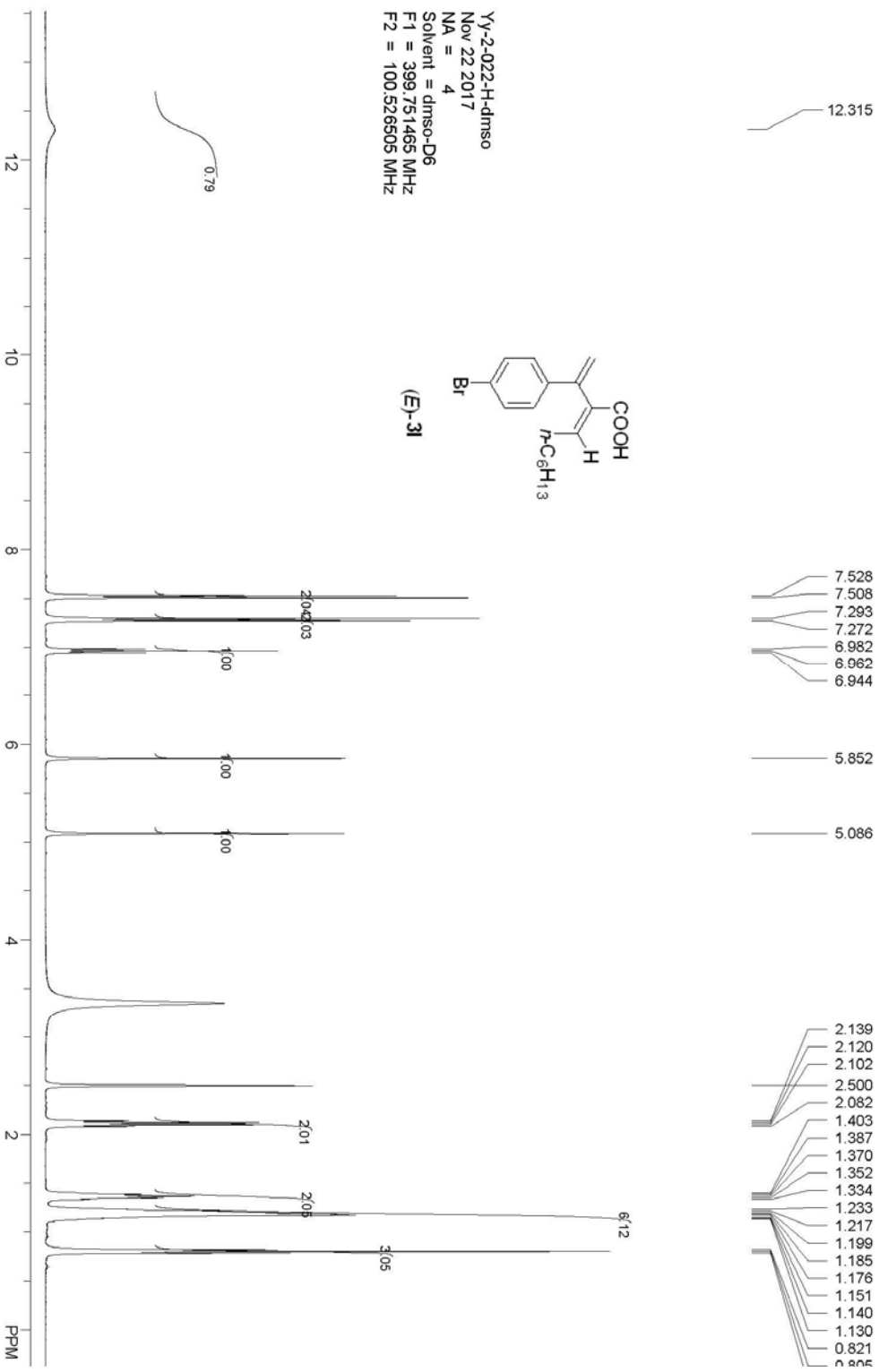

Yy-2-103-H
Jan 21 2018
NA = 8
Solvent = cdcl3
F1 = 399.749542 MHz
F2 = 100.526031 MHz
4.45
1.05
1.00
1.00
2.04
4.45
3.14
PPM
0
2
4
6
8
COOH
H
n-Bu
Cl
(E)-3j
7.288
7.268
7.256
7.216
7.196
7.178
2.234
2.215
2.196
2.177
1.447
1.429
1.409
1.392
1.373
1.342
1.324
1.306
1.287
1.269
1.252
0.876
0.857
0.840
-0.000

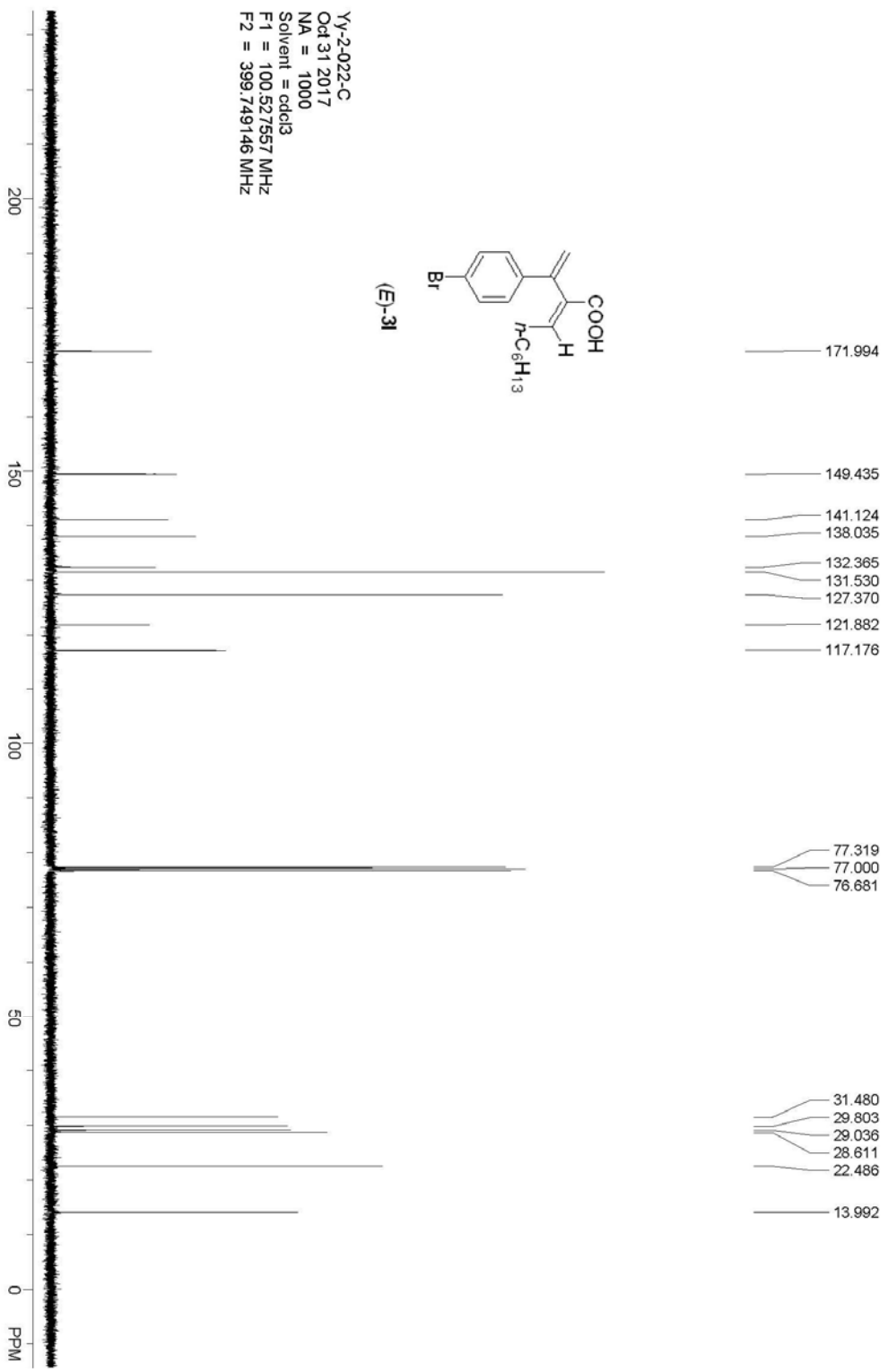

Yy-2-103-C
Jan 21 2018
NA = 512
Solvent = cdcl3
F1 = 100.527557 MHz
F2 = 399.749146 MHz
(E)-3j
Cl
COOH
n-Bu
H
171.979
149.306
141.078
137.572
133.678
132.471
128.585
127.051
117.085
77.319
77.000
76.681
30.790
29.522
22.448
13.779
200
150
100
50
0
PPM

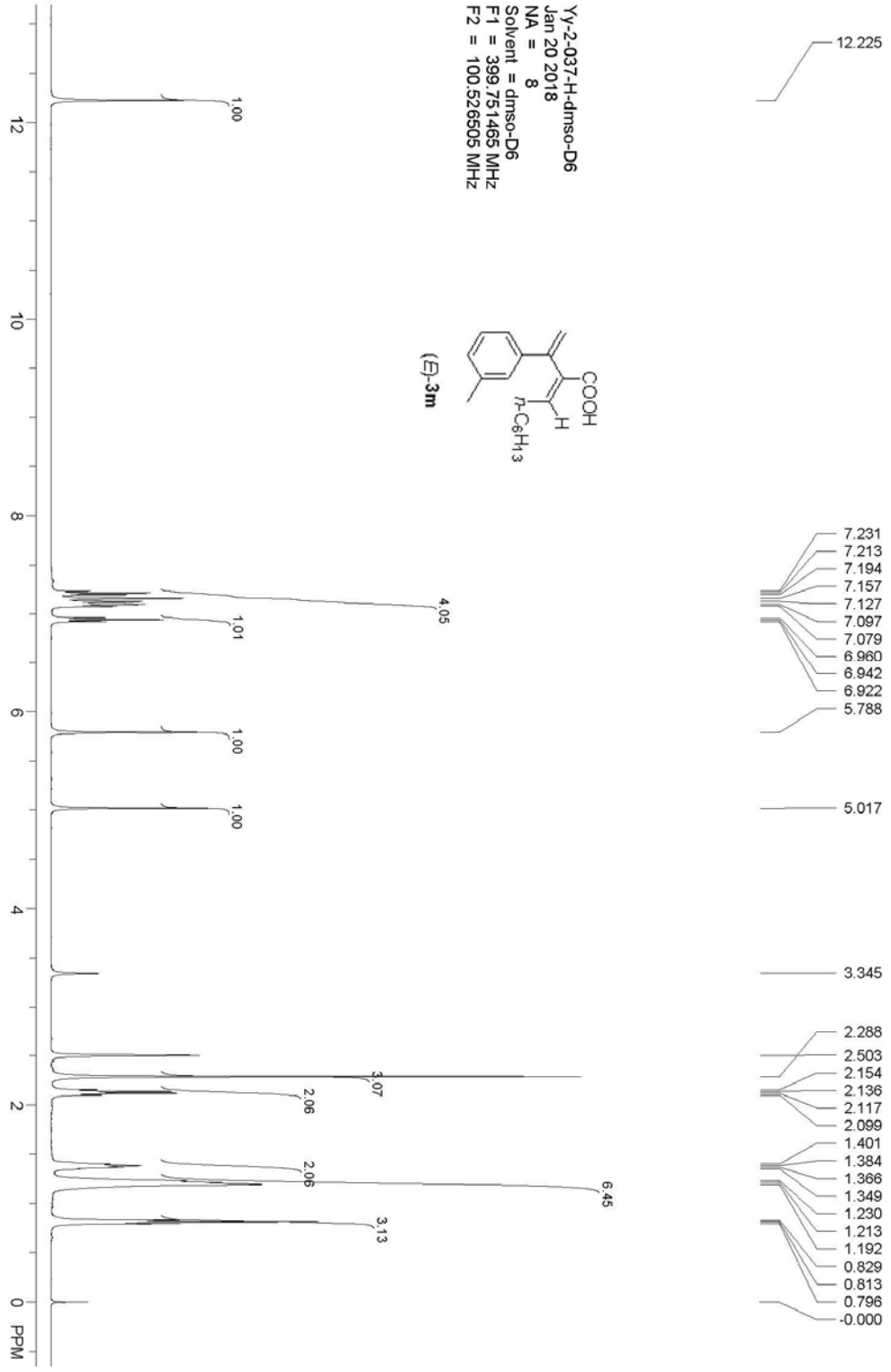

Yy-2-105-dmso-D6-H
Apr 3 2018
NA = 8
Solvent = dmso-D6
F1 = 399.751465 MHz
F2 = 100.526505 MHz
(E)-3k
COOH
H
n-Bu
12.211
7.237
7.218
7.141
7.122
6.952
6.932
6.913
5.757
4.977
3.333
2.504
2.500
2.495
2.278
2.146
2.127
2.109
2.090
1.400
1.383
1.364
1.345
1.326
1.288
1.271
1.252
1.233
1.216
1.198
0.828
0.810
0.89
2.03
2.16
1.02
1.00
1.02
3.23
2.09
4.61
3.33
* impurity ~ (PhO)2PO
12
10
8
6
4
2
0 PPM

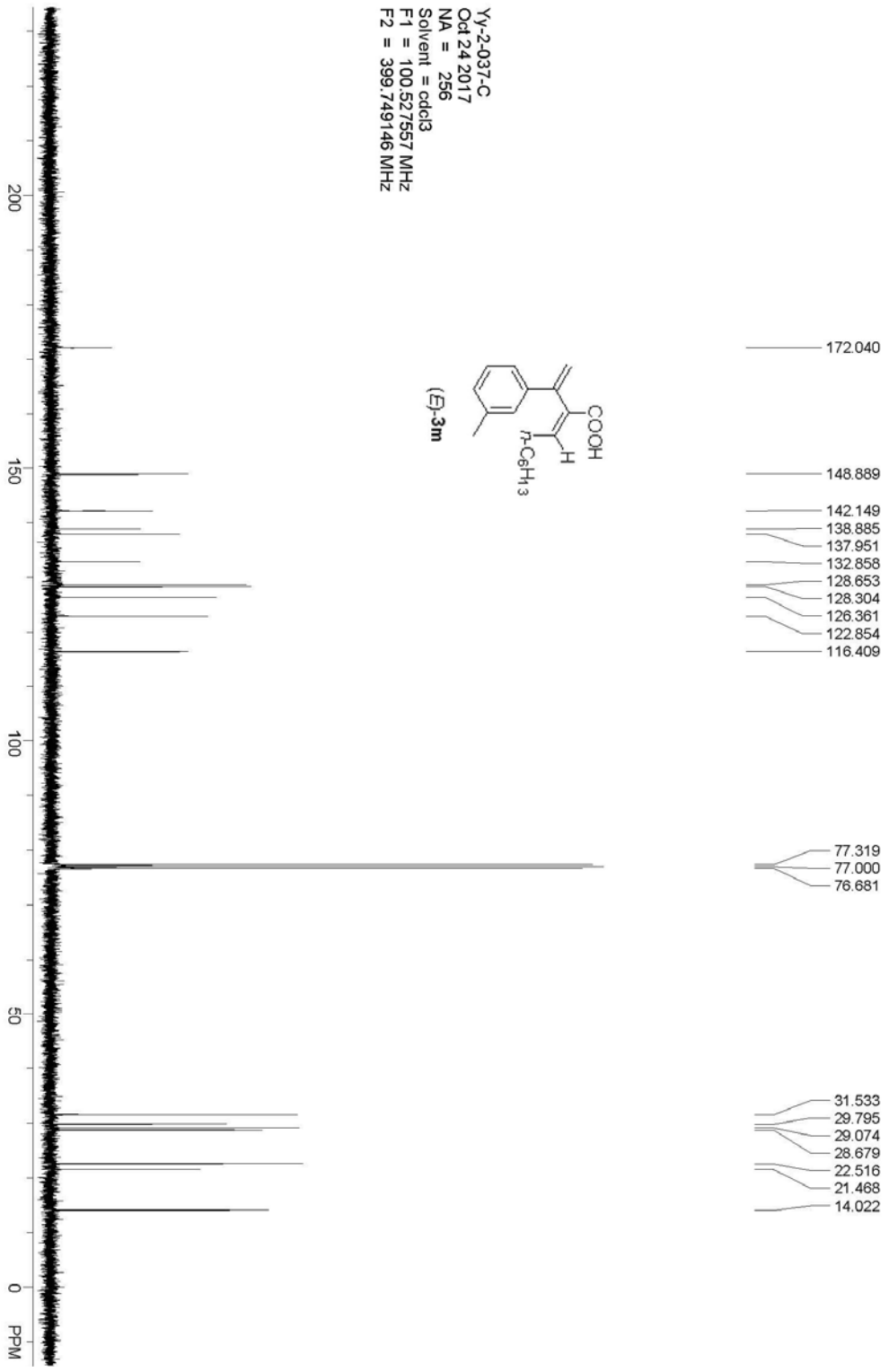

Yy-2-105-C
Jan 25 2018
NA = 512
Solvent = cdcl3
F1 = 100.527557 MHz
F2 = 399.749146 MHz
(E)-3k
COOH
H
n-Bu
171.956
148.752
141.845
137.663
136.061
132.873
129.139
125.556
115.628
77.319
77.000
76.681
30.858
29.499
22.470
21.112
13.832
* impurity
200
150
100
50
0
PPM

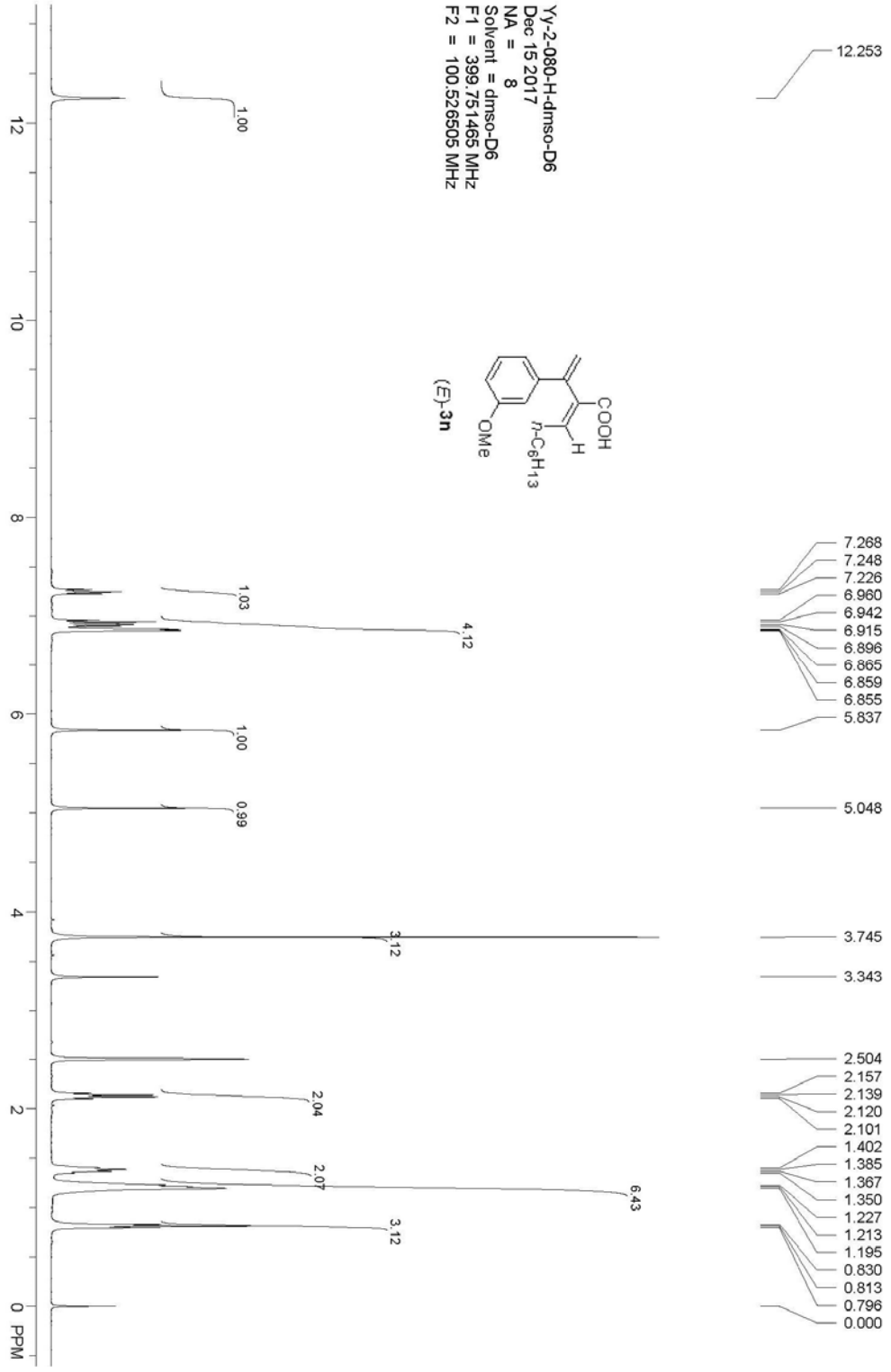

Yy-2-105-purity
purity = 94%
39.1 mg product with 17.5 uL dibromomethane
May 25 2018
NA = 4
Solvent = CDCl3
F1 = 400.130005 MHz
F2 = 1.000000 MHz
15.38
15.11
50.00
5.761
5.066
4.932
-0.000
COOH
H
n-Bu
(E)-3k
PPM

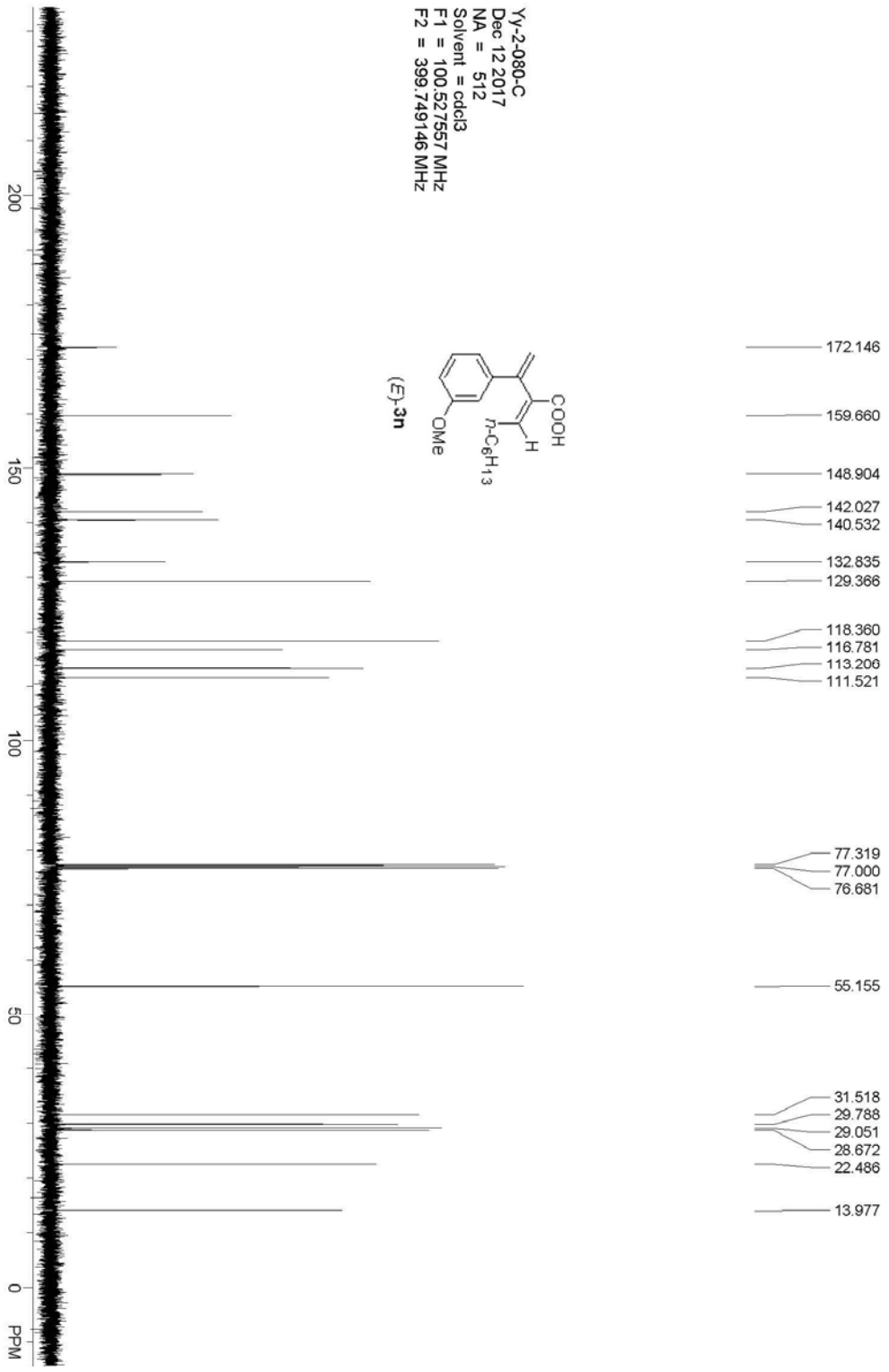

Yy-2-022-H-dmso
Nov 22 2017
NA = 4
Solvent = dmso-D6
F1 = 399.751465 MHz
F2 = 100.526505 MHz
(E)-3l
Br
COOH
H
n-C6H13
12.315
7.528
7.508
7.293
7.272
6.982
6.962
6.944
5.852
5.086
2.139
2.120
2.102
2.500
2.082
1.403
1.387
1.370
1.352
1.334
1.233
1.217
1.199
1.185
1.176
1.151
1.140
1.130
0.821
0.79
2.04
2.03
1.00
1.00
1.00
2.01
2.05
6.12
3.05
12
10
8
6
4
2
PPM

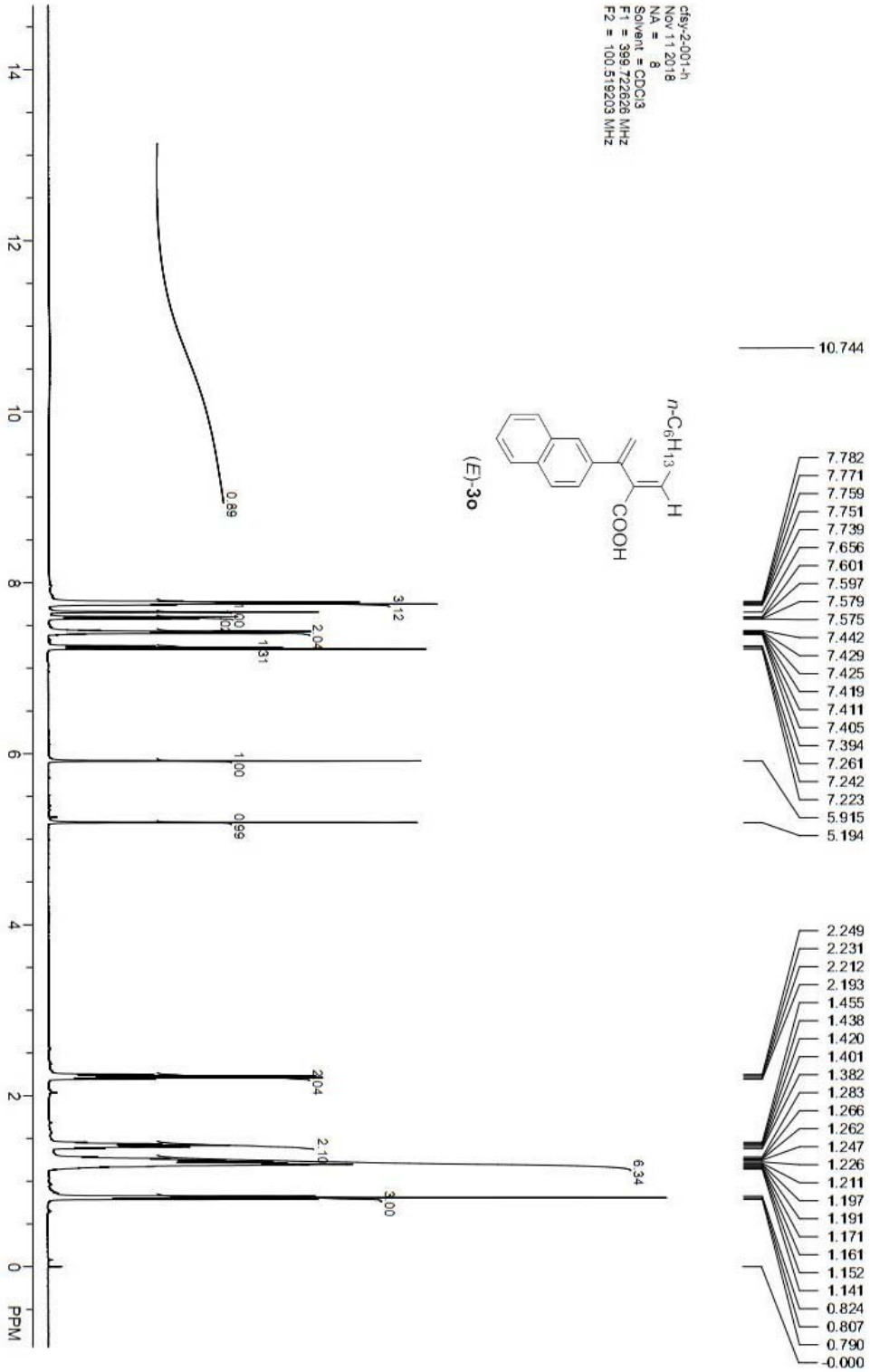

Yy-2-022-C
Oct 31 2017
NA = 1000
Solvent = cdcl3
F1 = 100.527557 MHz
F2 = 399.749146 MHz
(E)-3l
Br
COOH
H
n-C6H13
171.994
149.435
141.124
138.035
132.365
131.530
127.370
121.882
117.176
77.319
77.000
76.681
31.480
29.803
29.036
28.611
22.486
13.992
200
150
100
50
0
PPM

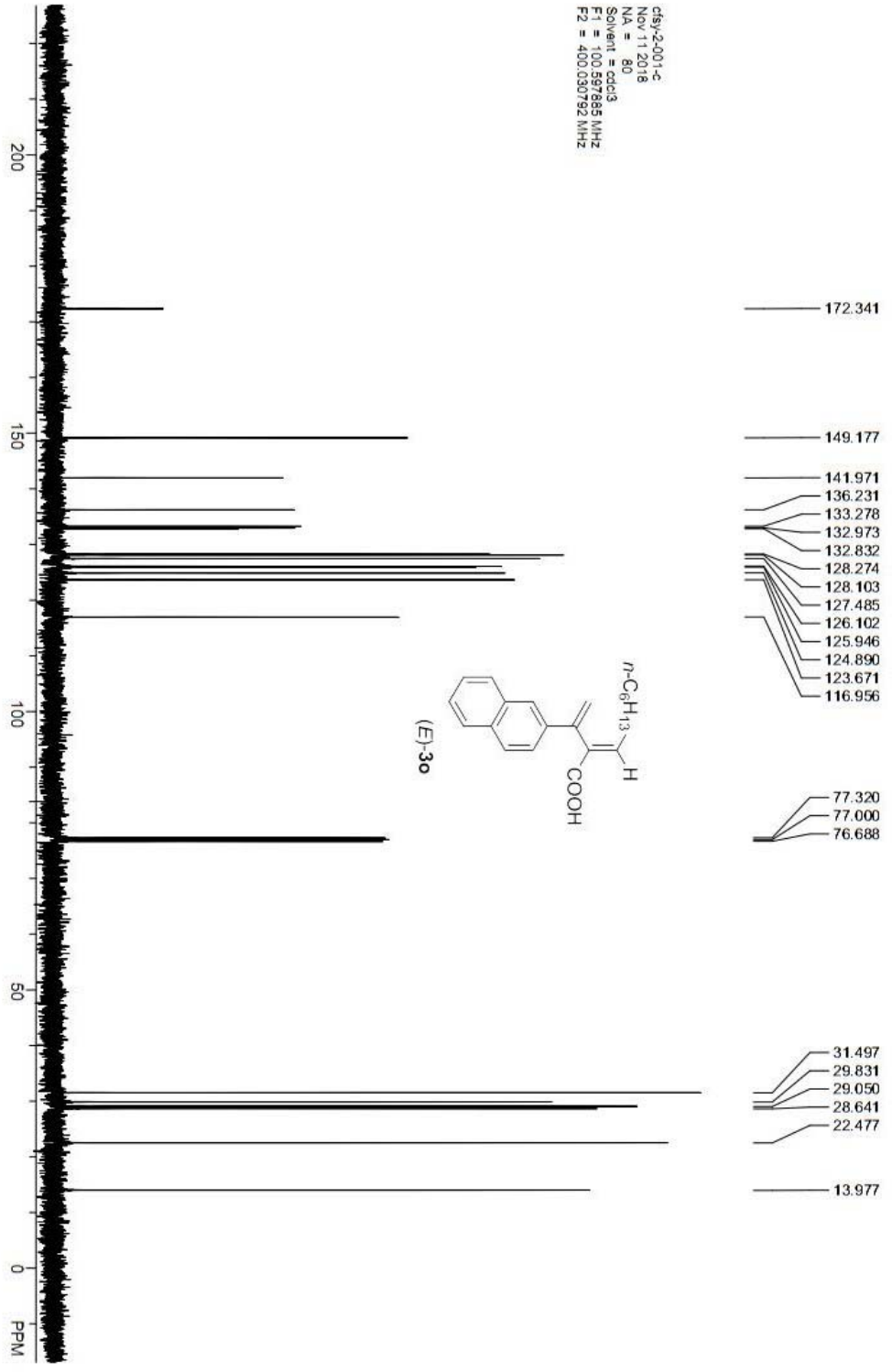

Yy-2-037-H-dmso-D6
Jan 20 2018
NA = 8
Solvent = dmso-D6
F1 = 399.751465 MHz
F2 = 100.526505 MHz
(E)-3m
COOH
H
n-C6H13
12.225
7.231
7.213
7.194
7.157
7.127
7.097
7.079
6.960
6.942
6.922
5.788
5.017
3.345
2.288
2.503
2.154
2.136
2.117
2.099
1.401
1.384
1.366
1.349
1.230
1.213
1.192
0.829
0.813
0.796
-0.000
1.00
4.05
1.01
1.00
1.00
3.07
2.06
2.06
6.45
3.13
12
10
8
6
4
2
0
PPM

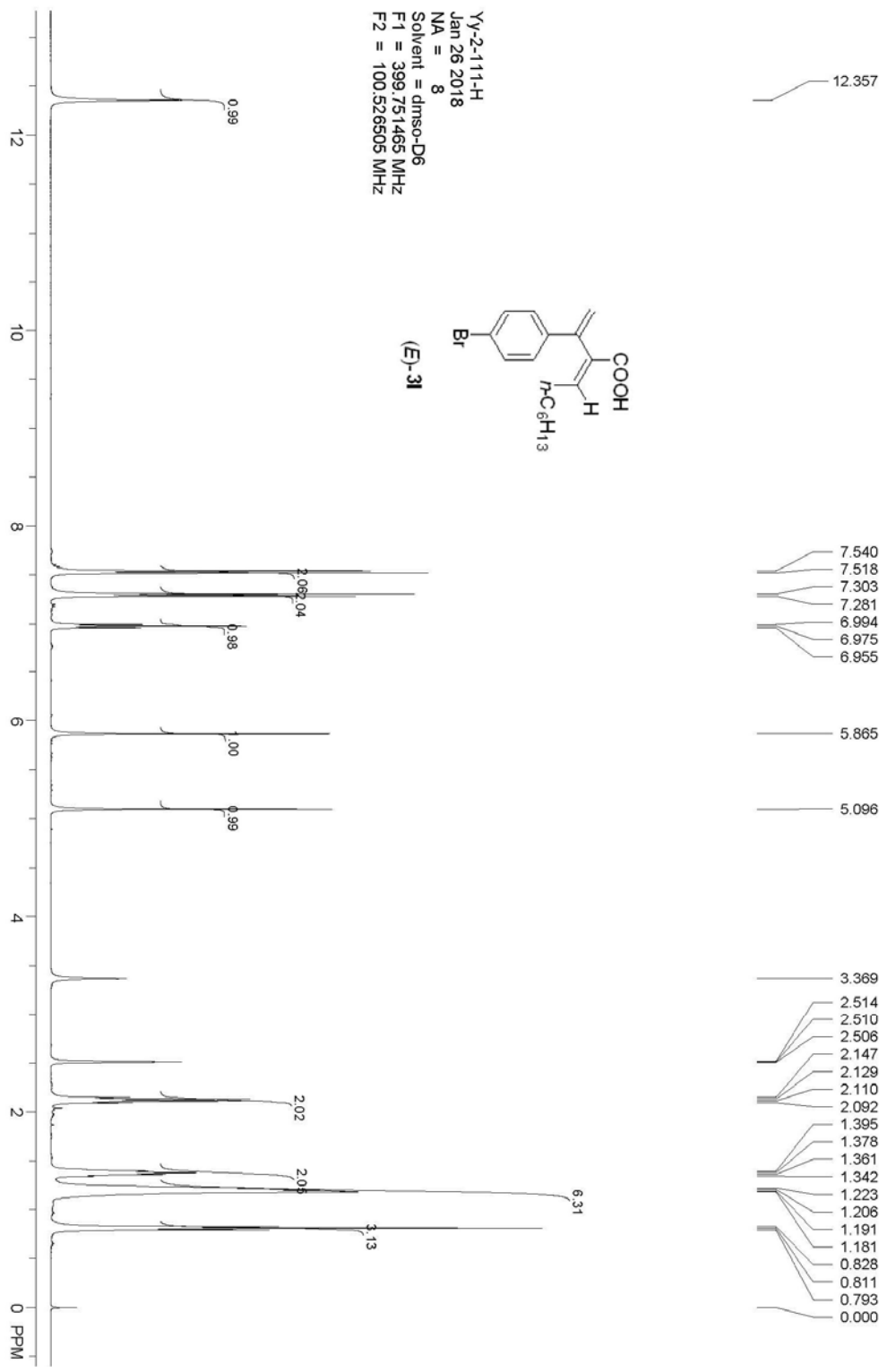

Yy-2-037-C
Oct 24 2017
NA = 256
Solvent = cdcl3
F1 = 100.527557 MHz
F2 = 399.749146 MHz
(E)-3m
COOH
H
n-C6H13
172.040
148.889
142.149
138.885
137.951
132.858
128.653
128.304
126.361
122.854
116.409
77.319
77.000
76.681
31.533
29.795
29.074
28.679
22.516
21.468
14.022
200
150
100
50
0
PPM

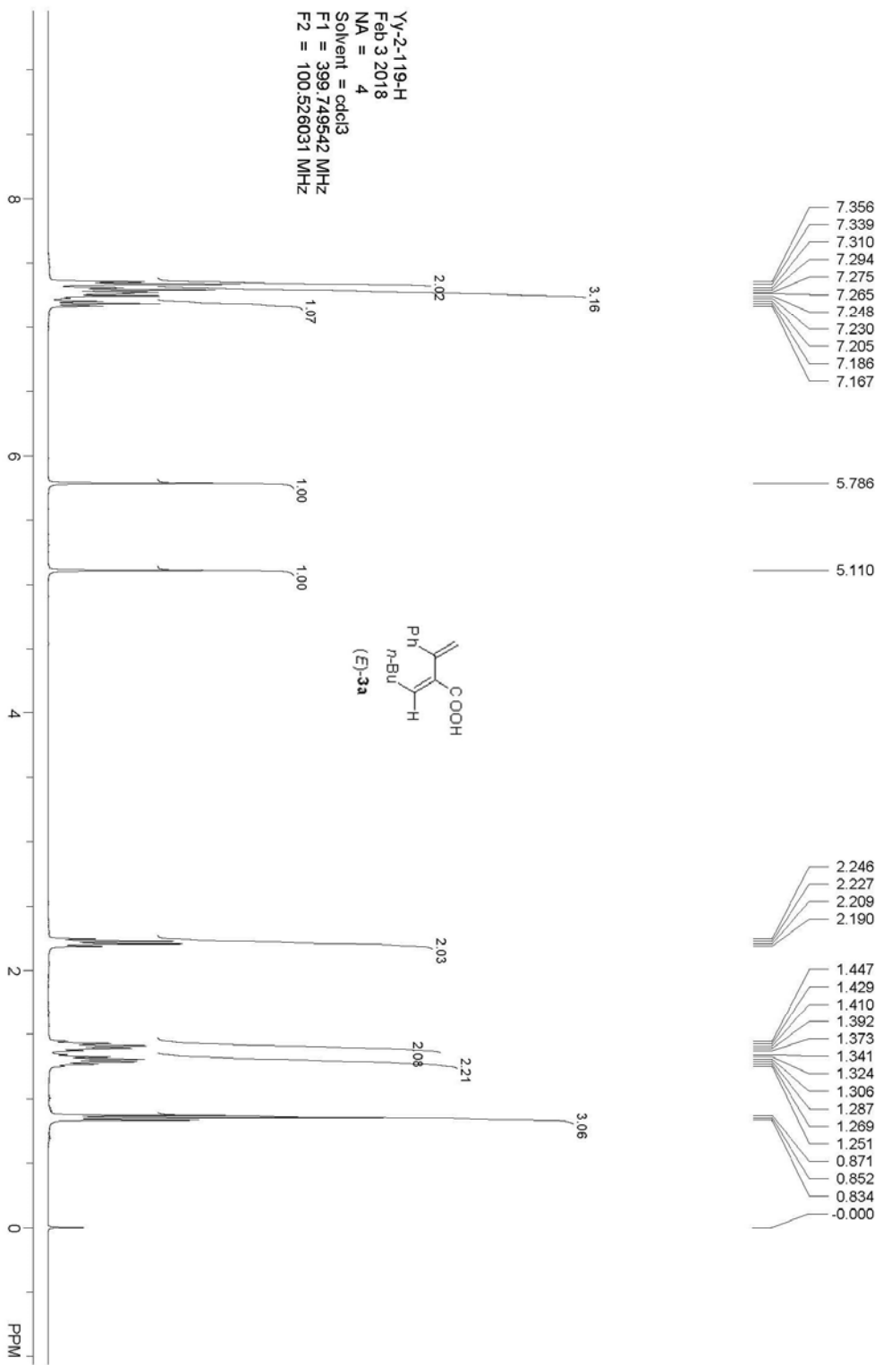

Yy-2-080-H-dmso-D6
Dec 15 2017
NA = 8
Solvent = dmso-D6
F1 = 399.751465 MHz
F2 = 100.526505 MHz
(E)-3n
COOH
H
n-C6H13
OMe
12.253
7.268
7.248
7.226
6.960
6.942
6.915
6.896
6.865
6.859
6.855
5.837
5.048
3.745
3.343
2.504
2.157
2.139
2.120
2.101
1.402
1.385
1.367
1.350
1.227
1.213
1.195
0.830
0.813
0.796
0.000
1.00
1.03
4.12
1.00
0.99
3.12
2.04
2.07
6.43
3.12
12
10
8
6
4
2
0 PPM

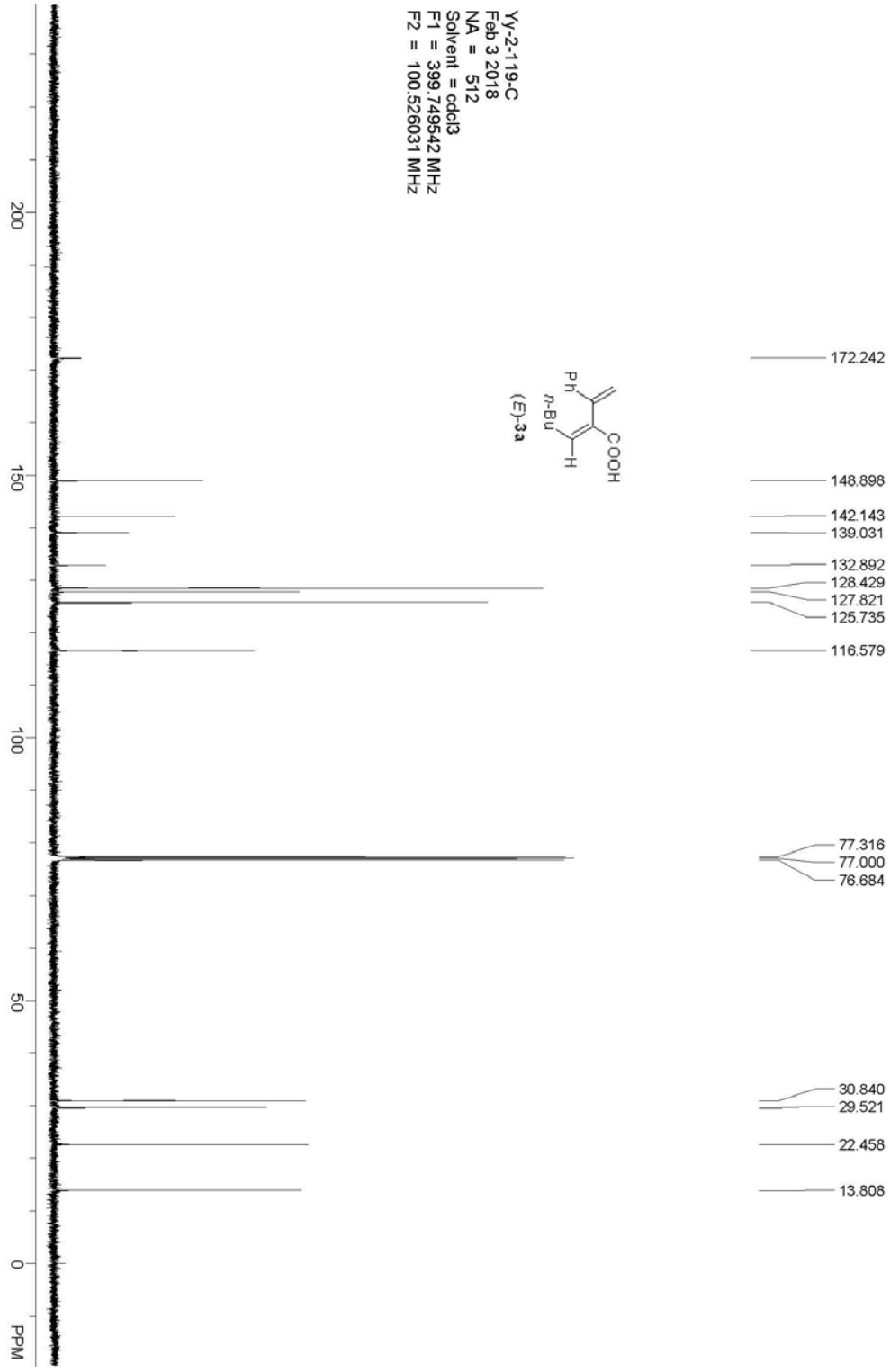

Yy-2-080-C
Dec 12 2017
NA = 512
Solvent = cdcl3
F1 = 100.527557 MHz
F2 = 399.749146 MHz
(E)-3n
COOH
H
n-C6H13
OMe
172.146
159.660
148.904
142.027
140.532
132.835
129.366
118.360
116.781
113.206
111.521
77.319
77.000
76.681
55.155
31.518
29.788
29.051
28.672
22.486
13.977
200
150
100
50
0
PPM

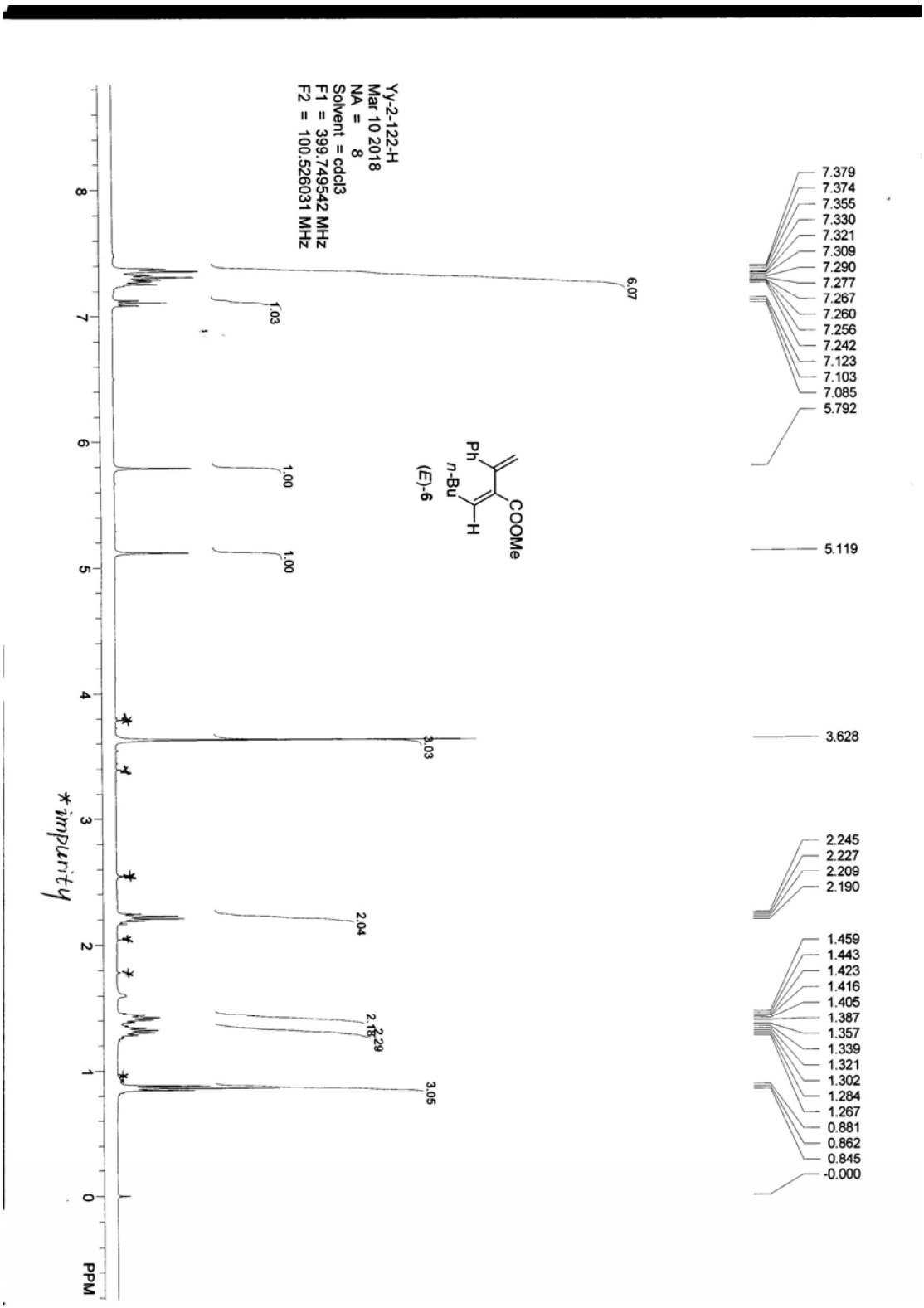

cfsy-2-001-h
Nov 11 2018
NA = 8
Solvent = CDCl3
F1 = 399.722626 MHz
F2 = 100.519203 MHz
(E)-3o
n-C6H13
H
COOH
10.744
7.782
7.771
7.759
7.751
7.739
7.656
7.601
7.597
7.579
7.575
7.442
7.429
7.425
7.419
7.411
7.405
7.394
7.261
7.242
7.223
5.915
5.194
2.249
2.231
2.212
2.193
1.455
1.438
1.420
1.401
1.382
1.283
1.266
1.262
1.247
1.226
1.211
1.197
1.191
1.171
1.161
1.152
1.141
0.824
0.807
0.790
-0.000
0.89
3.12
2.04
1.31
1.00
0.99
2.04
2.10
6.34
3.00
PPM

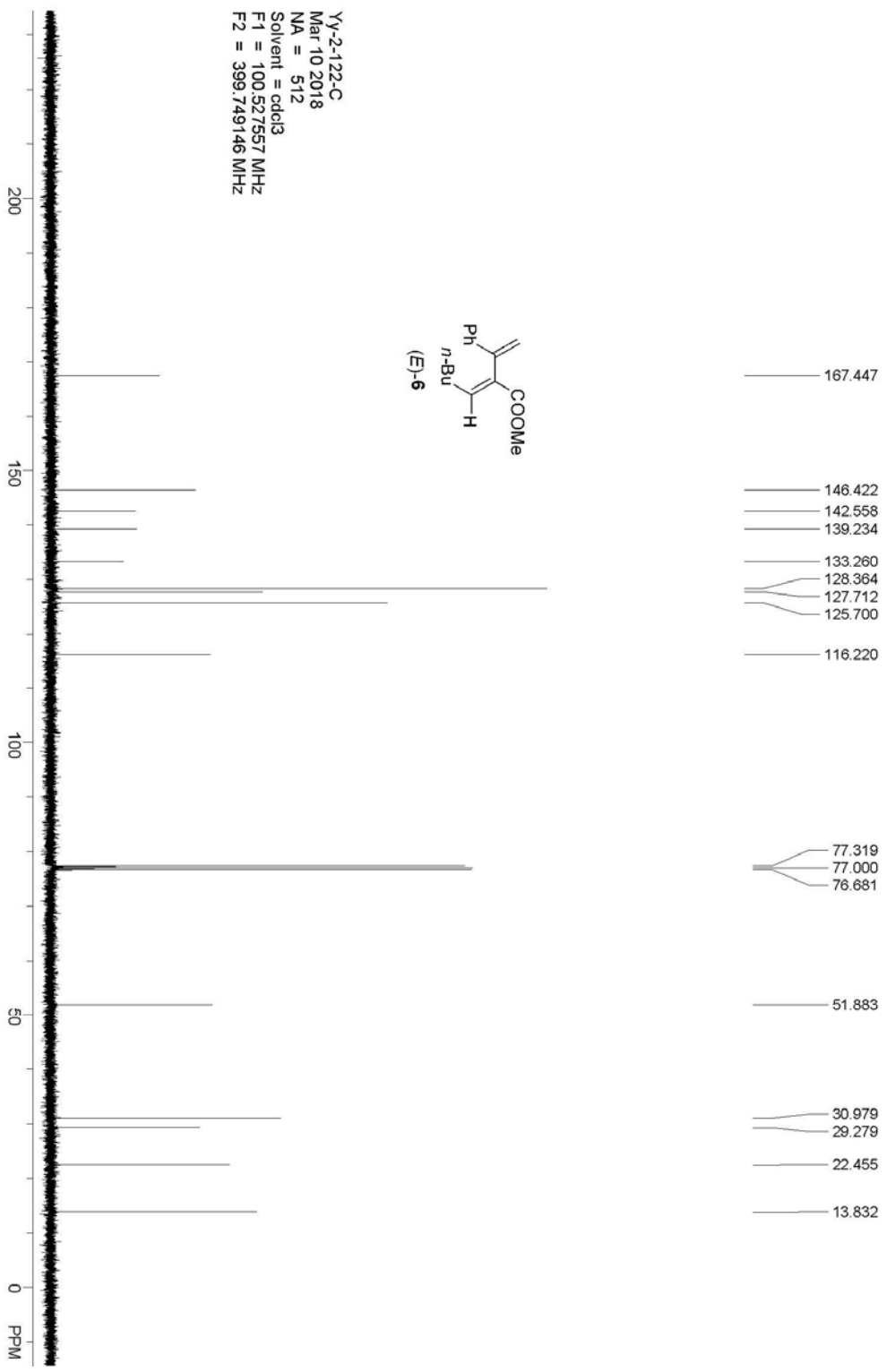

cfsy-2-001-c
Nov 11 2018
NA = 80
Solvent = cdcl3
F1 = 100.597885 MHz
F2 = 400.030792 MHz
(E)-3o
n-C6H13
H
COOH
172.341
149.177
141.971
136.231
133.278
132.973
132.832
128.274
128.103
127.485
126.102
125.946
124.890
123.671
116.956
77.320
77.000
76.688
31.497
29.831
29.050
28.641
22.477
13.977
200
150
100
50
0
PPM

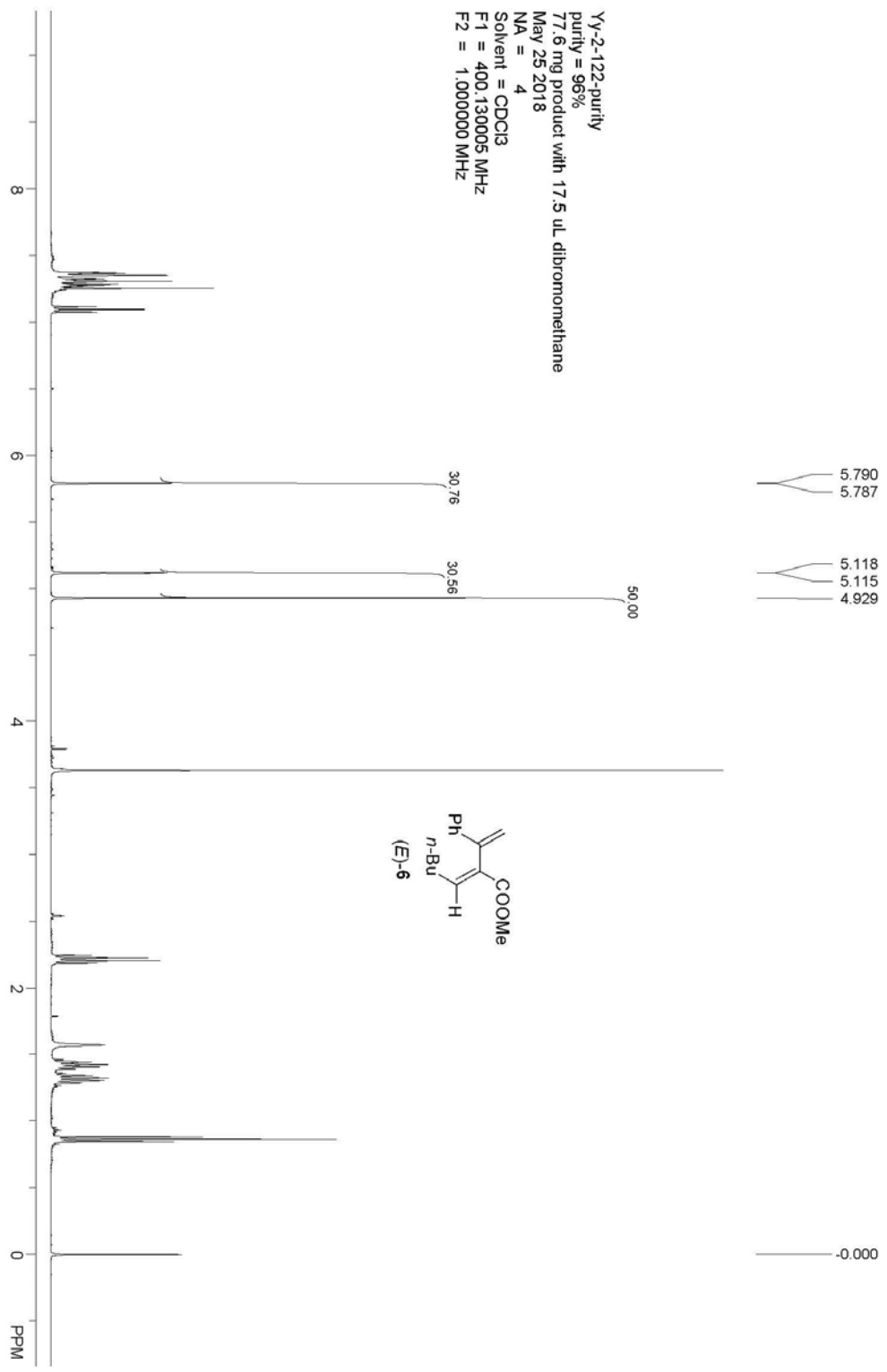

Yy-2-111-H
Jan 26 2018
NA = 8
Solvent = dmso-D6
F1 = 399.751465 MHz
F2 = 100.526505 MHz
(E)-3l
Br
COOH
H
n-C6H13
12.357
7.540
7.518
7.303
7.281
6.994
6.975
6.955
5.865
5.096
3.369
2.514
2.510
2.506
2.147
2.129
2.110
2.092
1.395
1.378
1.361
1.342
1.223
1.206
1.191
1.181
0.828
0.811
0.793
0.000
0.99
2.06
2.04
0.98
1.00
0.99
2.02
2.05
6.31
3.13
12
10
8
6
4
2
0 PPM

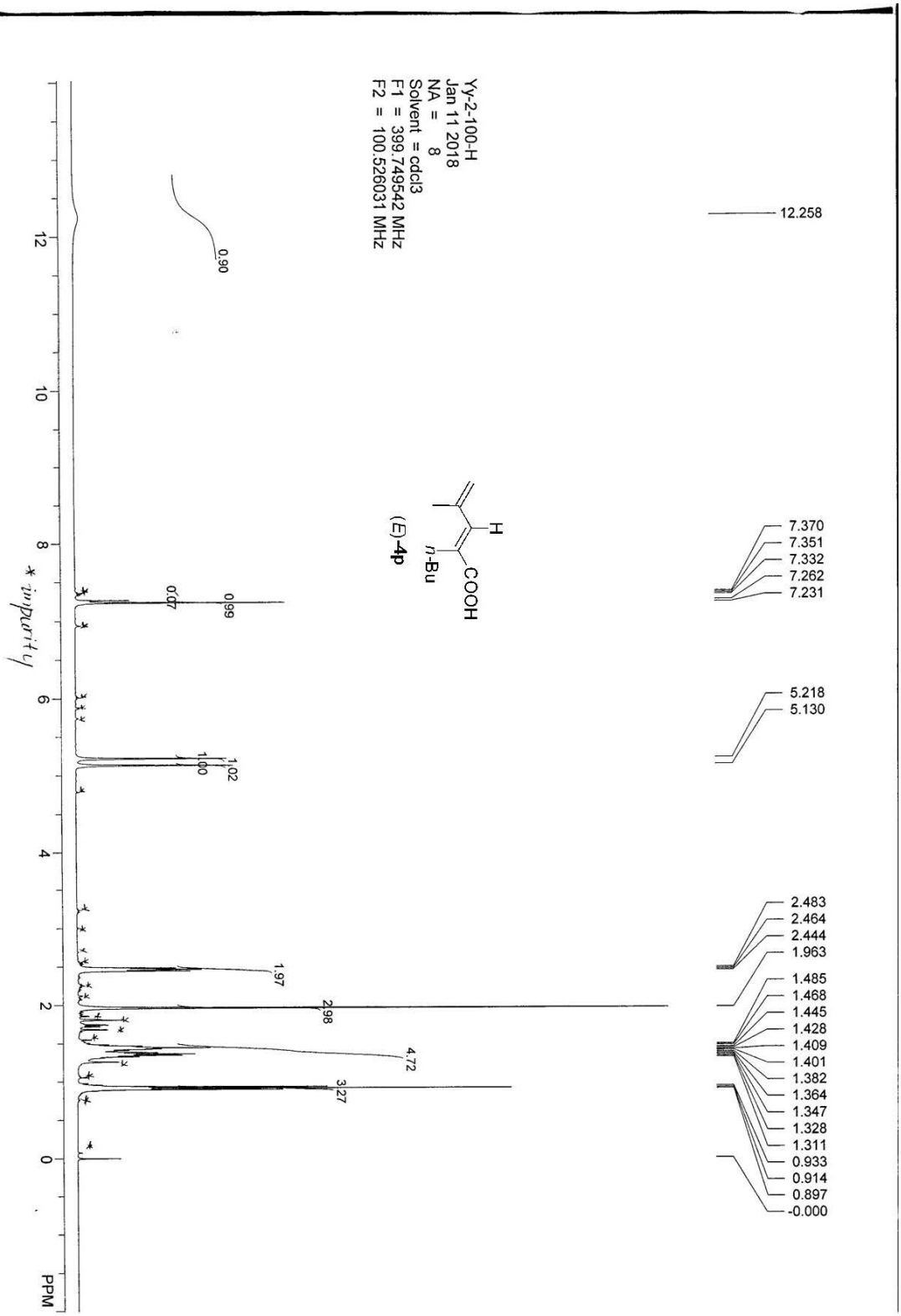

Yy-2-119-H
Feb 3 2018
NA = 4
Solvent = cdcl3
F1 = 399.749542 MHz
F2 = 100.526031 MHz
Ph
n-Bu
H
COOH
(E)-3a
7.356
7.339
7.310
7.294
7.275
7.265
7.248
7.230
7.205
7.186
7.167
5.786
5.110
2.246
2.227
2.209
2.190
1.447
1.429
1.410
1.392
1.373
1.341
1.324
1.306
1.287
1.269
1.251
0.871
0.852
0.834
-0.000
3.16
2.02
1.07
1.00
1.00
2.03
2.08
2.21
3.06
8
6
4
2
0
PPM

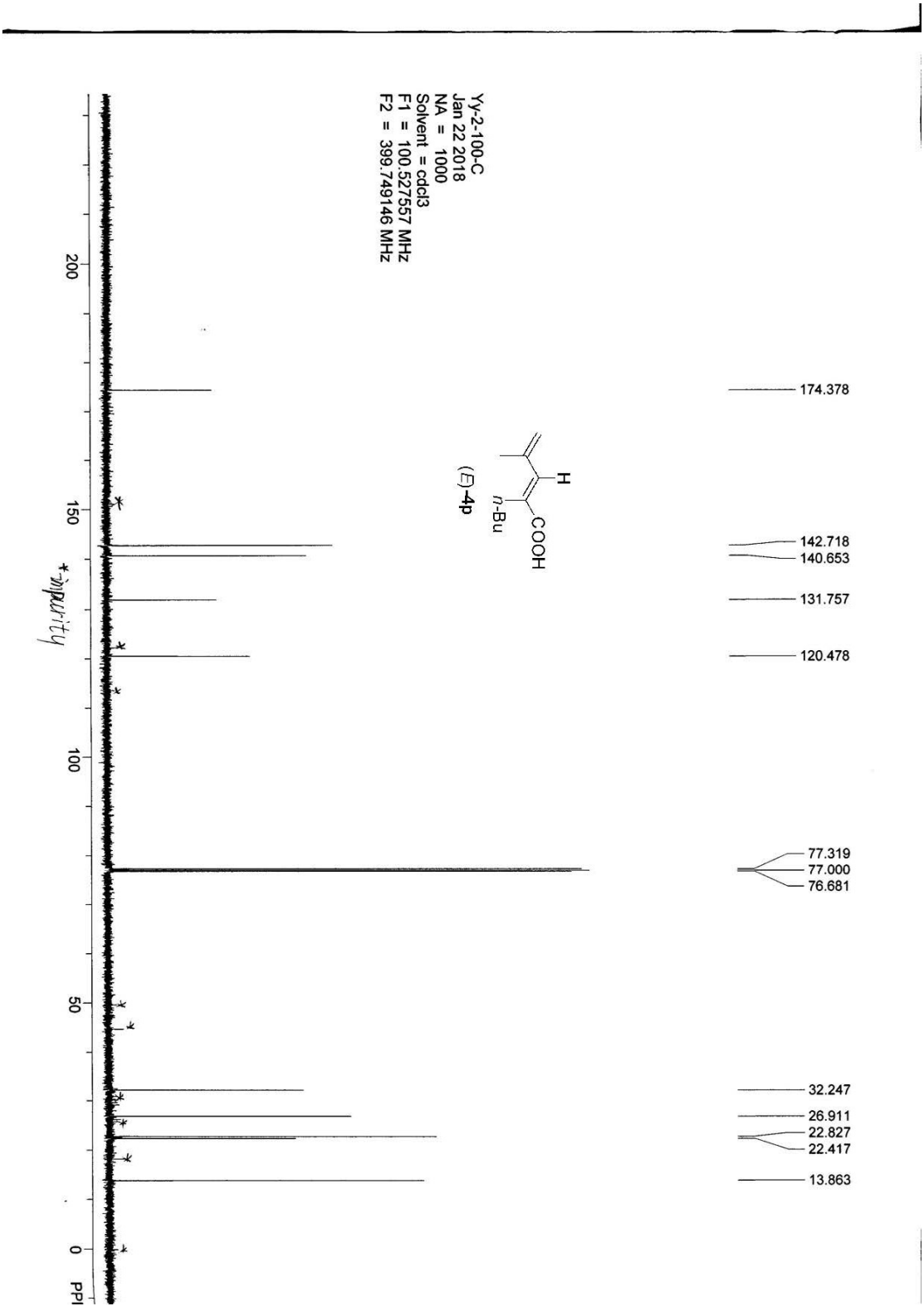

Yy-2-119-C
Feb 3 2018
NA = 512
Solvent = cdcl3
F1 = 399.749542 MHz
F2 = 100.526031 MHz
(E)-3a
Ph
n-Bu
H
COOH
172.242
148.898
142.143
139.031
132.892
128.429
127.821
125.735
116.579
77.316
77.000
76.684
30.840
29.521
22.458
13.808
200
150
100
50
0
PPM

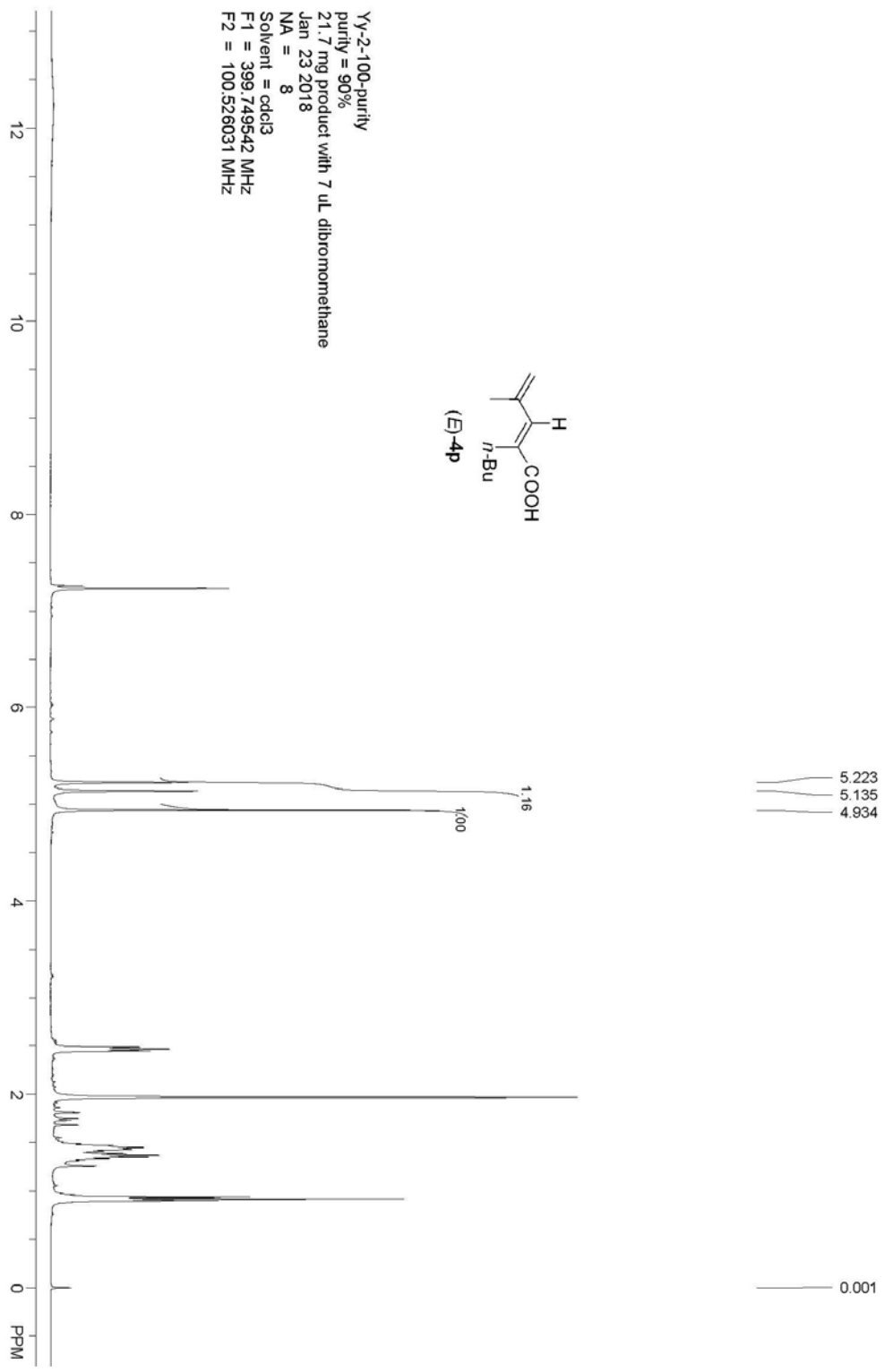

Yy-2-122-H
Mar 10 2018
NA = 8
Solvent = cdcl3
F1 = 399.749542 MHz
F2 = 100.526031 MHz
Ph
n-Bu
H
COOMe
(E)-6
*impurity
PPM
6.07
1.03
1.00
1.00
3.03
2.04
2.18
2.29
3.05
7.379
7.374
7.355
7.330
7.321
7.309
7.290
7.277
7.267
7.260
7.256
7.242
7.123
7.103
7.085
5.792
5.119
3.628
2.245
2.227
2.209
2.190
1.459
1.443
1.423
1.416
1.405
1.387
1.357
1.339
1.321
1.302
1.284
1.267
0.881
0.862
0.845
-0.000

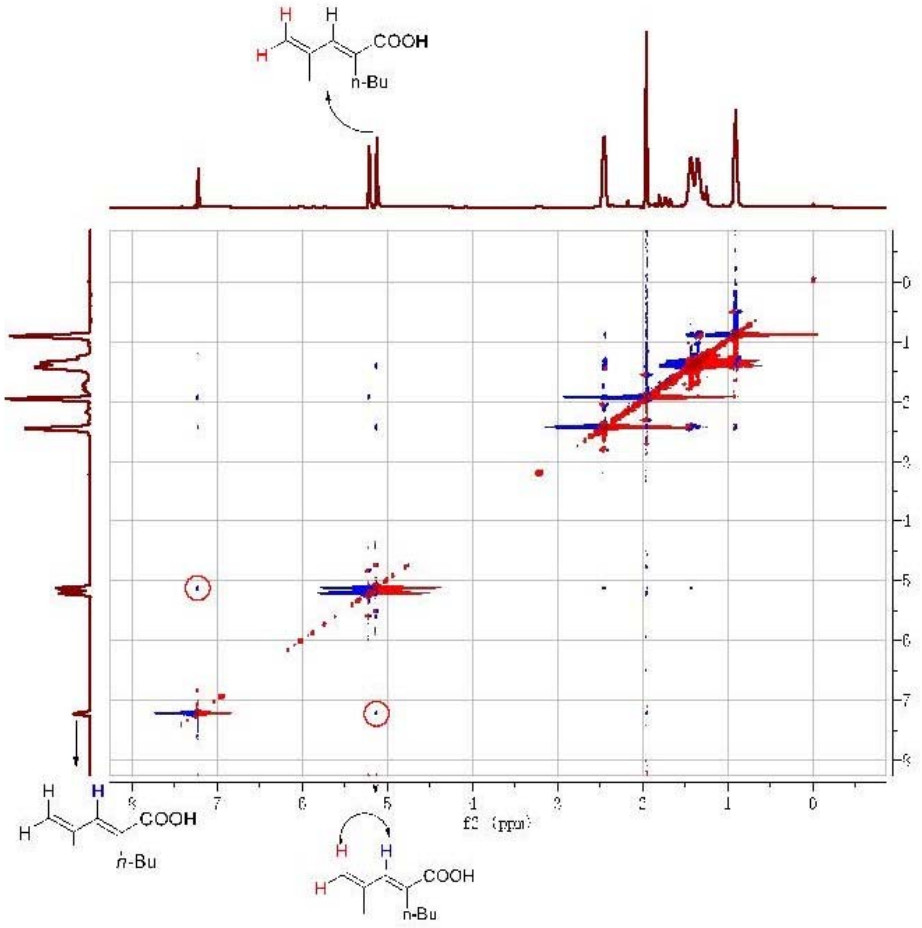

Yy-2-122-C
Mar 10 2018
NA = 512
Solvent = cdcl3
F1 = 100.527557 MHz
F2 = 399.749146 MHz
Ph
n-Bu
H
COOMe
(E)-6
167.447
146.422
142.558
139.234
133.260
128.364
127.712
125.700
116.220
77.319
77.000
76.681
51.883
30.979
29.279
22.455
13.832
200
150
100
50
0
PPM


Yy-2-122-purity
purity = 96%
77.6 mg product with 17.5 uL dibromomethane
May 25 2018
NA = 4
Solvent = CDCl3
F1 = 400.130005 MHz
F2 = 1.000000 MHz
30.76
30.56
50.00
5.790
5.787
5.118
5.115
4.929
-0.000
Ph
n-Bu
H
COOMe
(E)-6
PPM

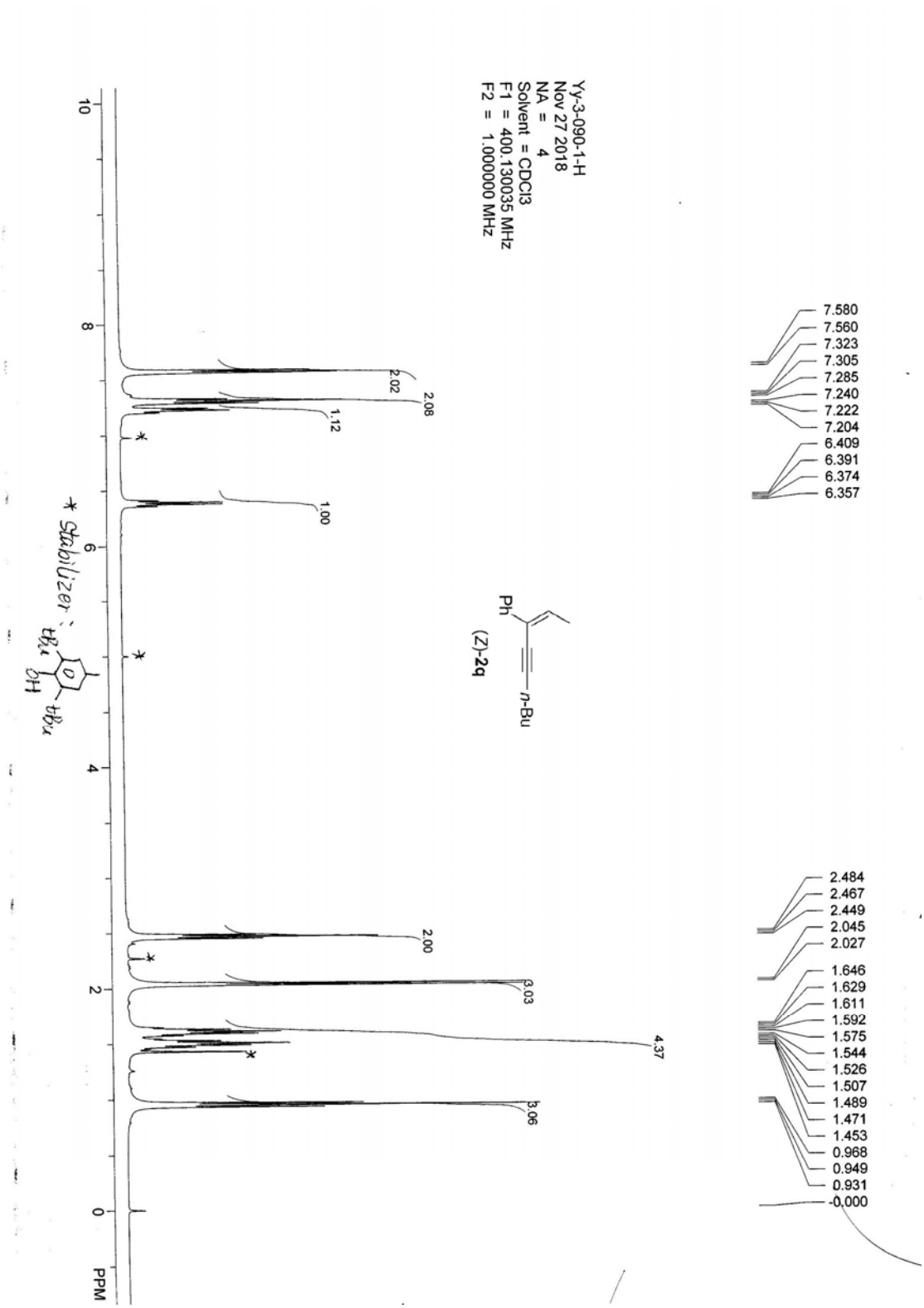

Yy-2-100-H
Jan 11 2018
NA = 8
Solvent = cdcl3
F1 = 399.749542 MHz
F2 = 100.526031 MHz
H
n-Bu
COOH
(E)-4p
12.258
7.370
7.351
7.332
7.262
7.231
5.218
5.130
2.483
2.464
2.444
1.963
1.485
1.468
1.445
1.428
1.409
1.401
1.382
1.364
1.347
1.328
1.311
0.933
0.914
0.897
-0.000
0.90
0.07
0.99
1.02
1.00
1.97
2.98
4.72
3.27
impurity
PPM

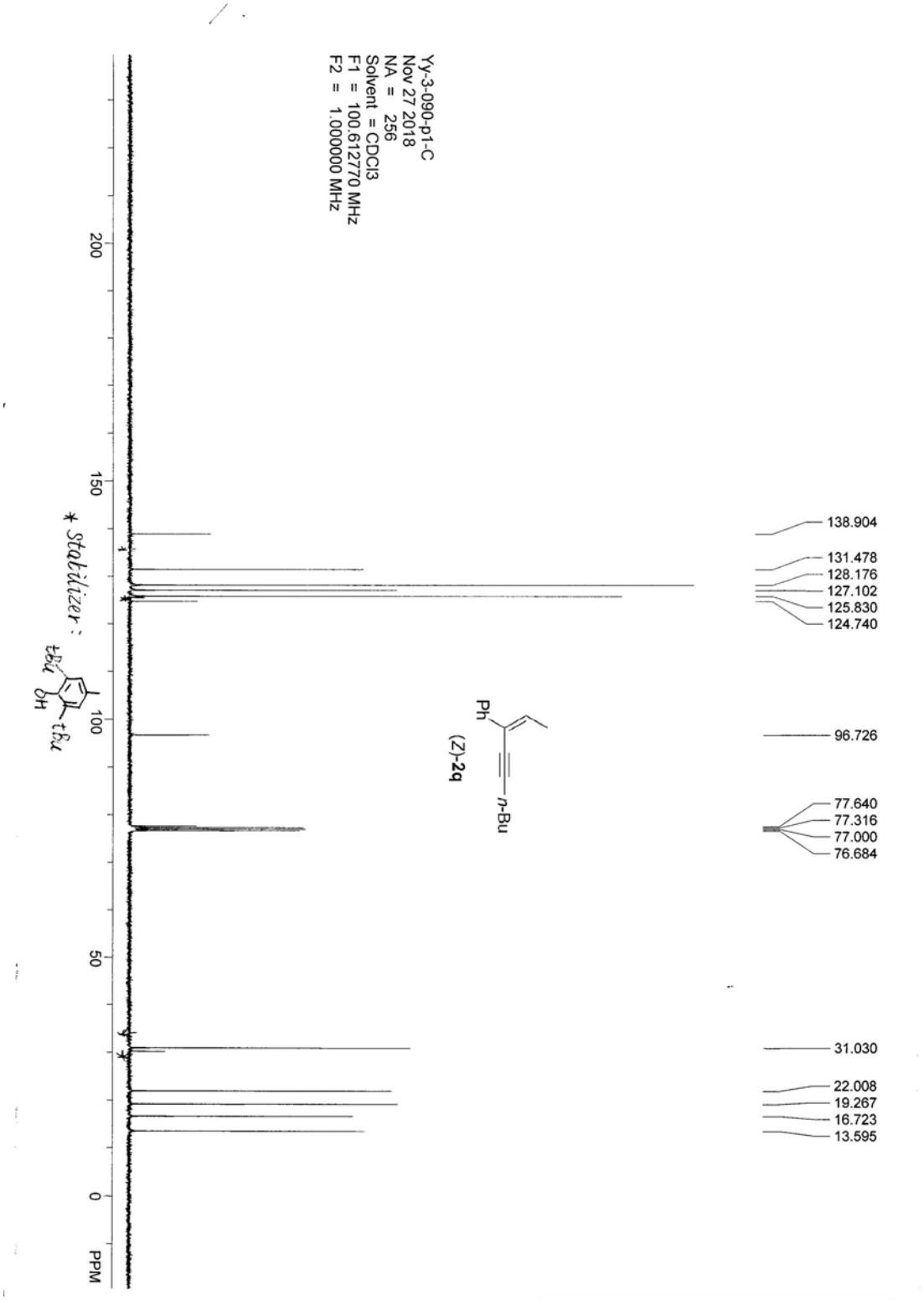

Yy-2-100-C
Jan 22 2018
NA = 1000
Solvent = cdcl3
F1 = 100.527557 MHz
F2 = 399.749146 MHz
(E)-4p
n-Bu
H
COOH
174.378
142.718
140.653
131.757
120.478
77.319
77.000
76.681
32.247
26.911
22.827
22.417
13.863
200
150
100
50
0
PPM

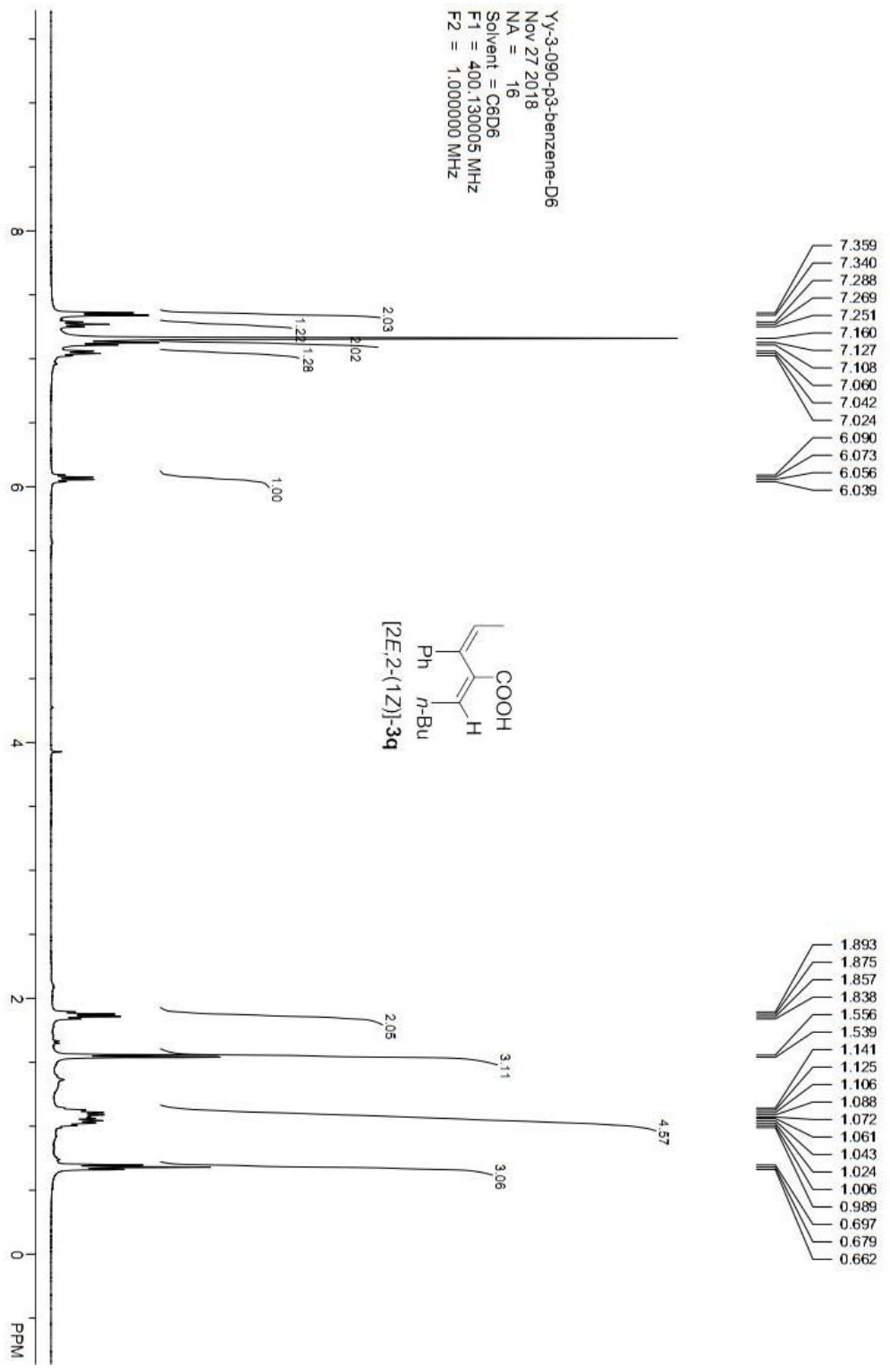

Yy-2-100-purity
purity = 90%
21.7 mg product with 7 uL dibromomethane
Jan 23 2018
NA = 8
Solvent = cdcl3
F1 = 399.749542 MHz
F2 = 100.526031 MHz
(E)-4p
H
n-Bu
COOH
1.16
1.00
5.223
5.135
4.934
0.001
12
10
8
6
4
2
0
PPM

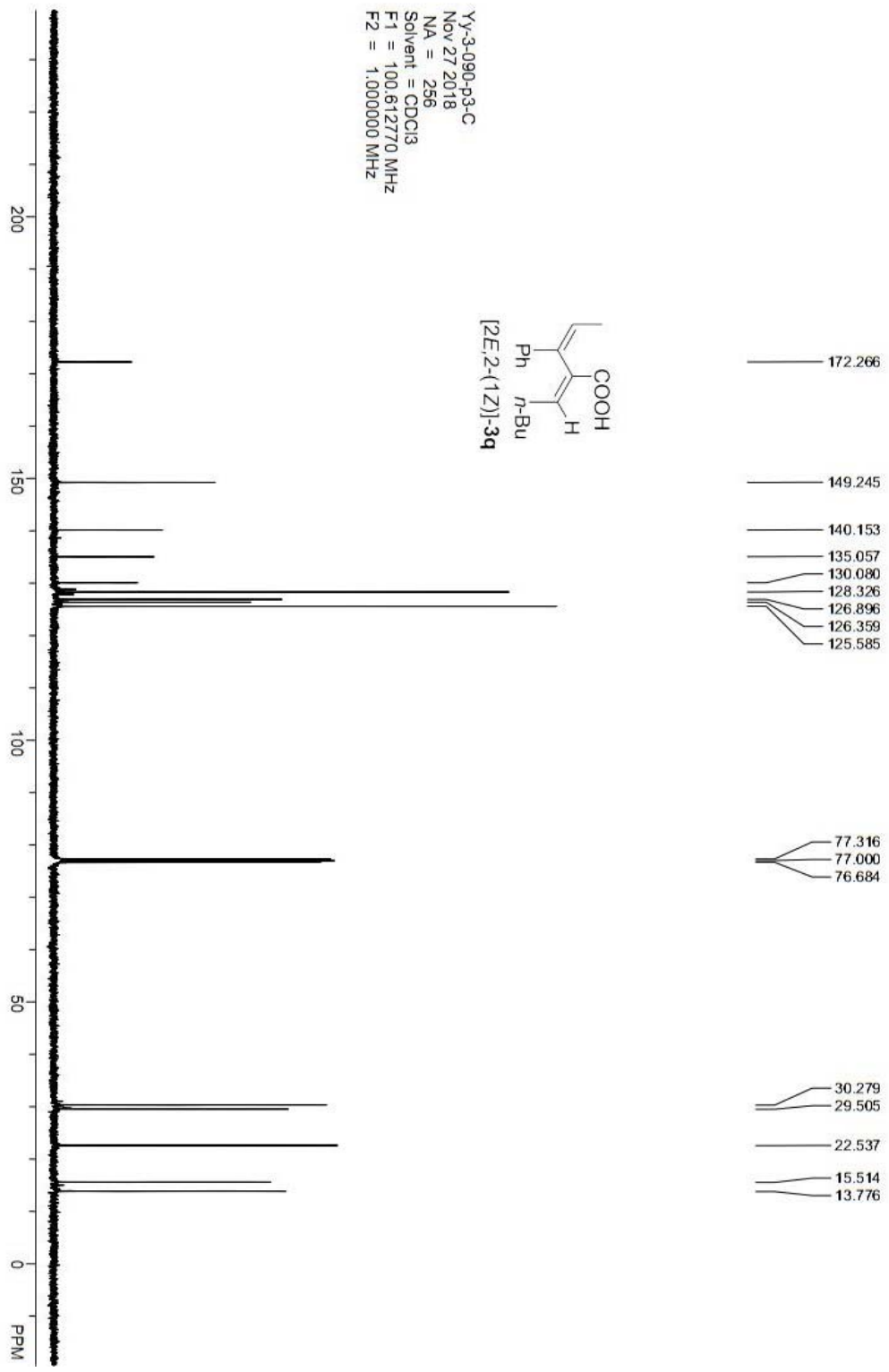


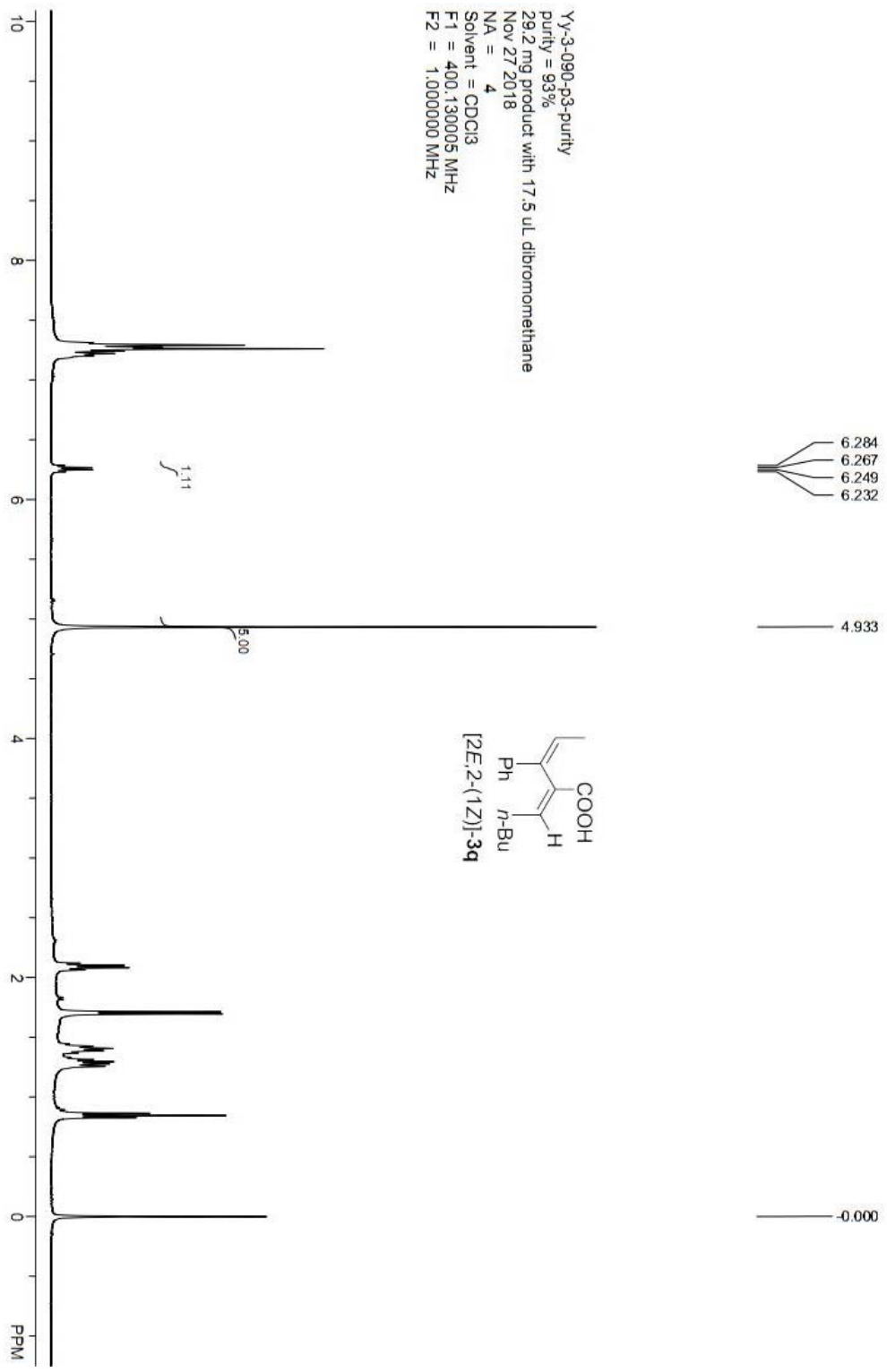
	Parameter	Value
1	Title	Yy-2-100-NOE
2	Origin	Varian
3	Solvent	cdcl3
4	Temperature	26.0
5	Pulse Sequence	NOESY
6	Experiment	2D-NOESY
7	Number of Scans	16
8	Receiver Gain	36
9	Relaxation Delay	1.0000
10	Pulse Width	0.0000
11	Acquisition Time	0.1499
12	Spectrometer Frequency	(399.75, 399.75)
13	Spectral Width	(3655.0, 3655.0)
14	Lowest Frequency	(-350.8, -350.8)
15	Nucleus	(1H, 1H)
16	Acquired Size	(548, 200)
17	Spectral Size	(1024, 1024)

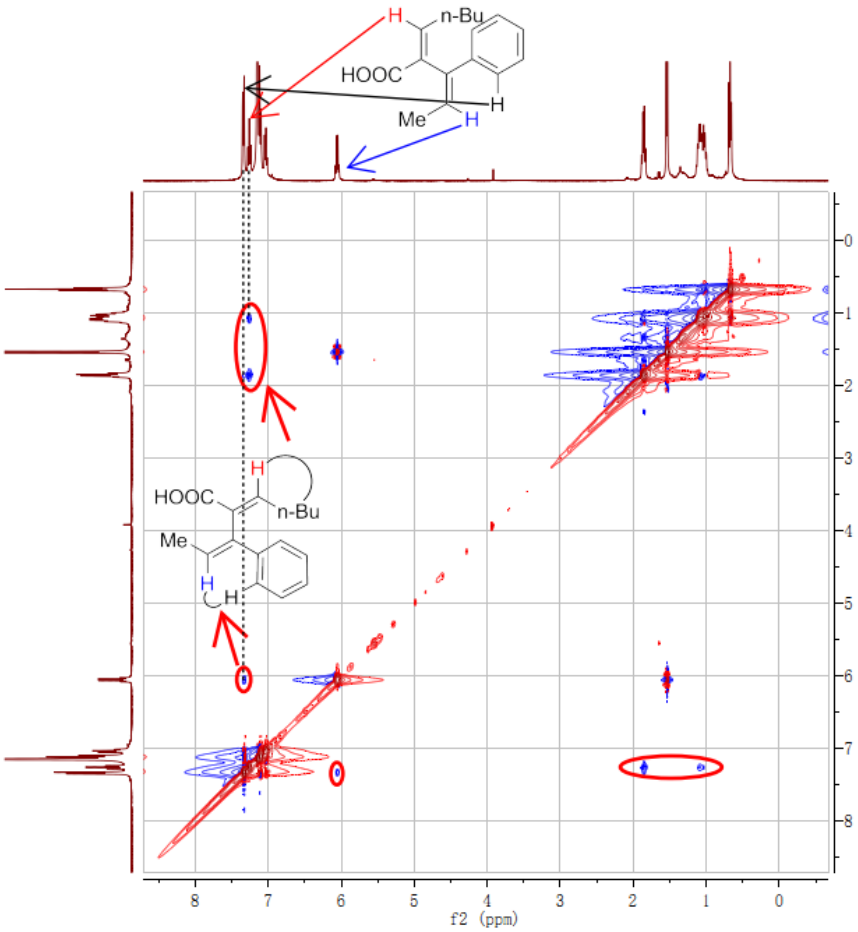

Yy-3-090-1-H
Nov 27 2018
NA = 4
Solvent = CDCl3
F1 = 400.130035 MHz
F2 = 1.000000 MHz
(Z)-2q
Ph
n-Bu
* Stabilizer
7.580
7.560
7.323
7.305
7.285
7.240
7.222
7.204
6.409
6.391
6.374
6.357
2.484
2.467
2.449
2.045
2.027
1.646
1.629
1.611
1.592
1.575
1.544
1.526
1.507
1.489
1.471
1.453
0.968
0.949
0.931
-0.000
2.02
2.08
1.12
1.00
2.00
3.03
4.37
3.06
PPM


Yy-3-090-p1-C
Nov 27 2018
NA = 256
Solvent = CDCl3
F1 = 100.612770 MHz
F2 = 1.000000 MHz
* Stabilizer :
tBu
OH
tBu
Ph
n-Bu
(Z)-2q
138.904
131.478
128.176
127.102
125.830
124.740
96.726
77.640
77.316
77.000
76.684
31.030
22.008
19.267
16.723
13.595
200
150
100
50
0
PPM

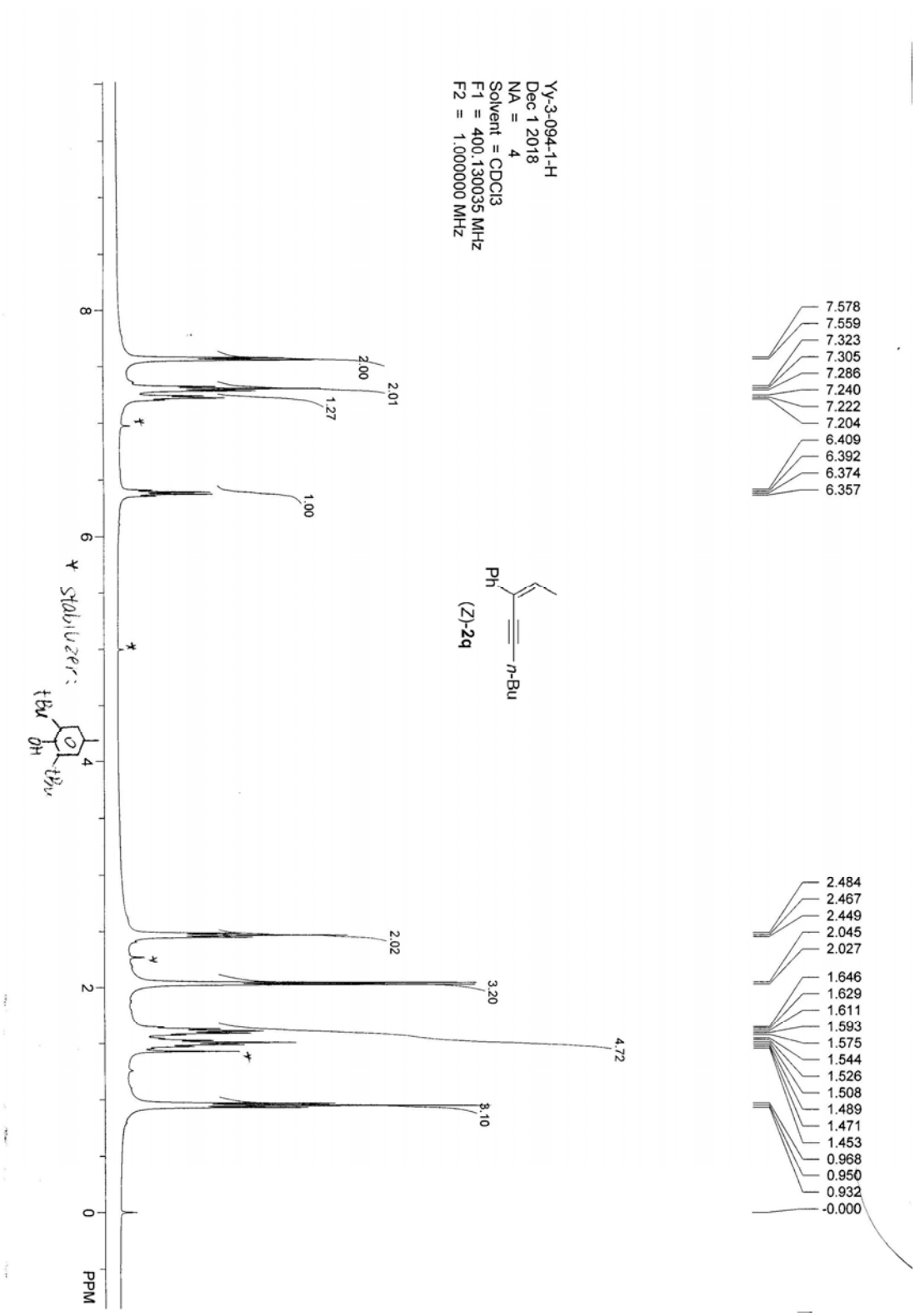

Yy-3-090-p3-benzene-D6
Nov 27 2018
NA = 16
Solvent = C6D6
F1 = 400.130005 MHz
F2 = 1.000000 MHz
7.359
7.340
7.288
7.269
7.251
7.160
7.127
7.108
7.060
7.042
7.024
6.090
6.073
6.056
6.039
1.893
1.875
1.857
1.838
1.556
1.539
1.141
1.125
1.106
1.088
1.072
1.061
1.043
1.024
1.006
0.989
0.697
0.679
0.662
2.03
1.22
2.02
1.28
1.00
2.05
3.11
4.57
3.06
COOH
H
Ph
n-Bu
[2E,2-(1Z)]-3q
8
6
4
2
0
PPM

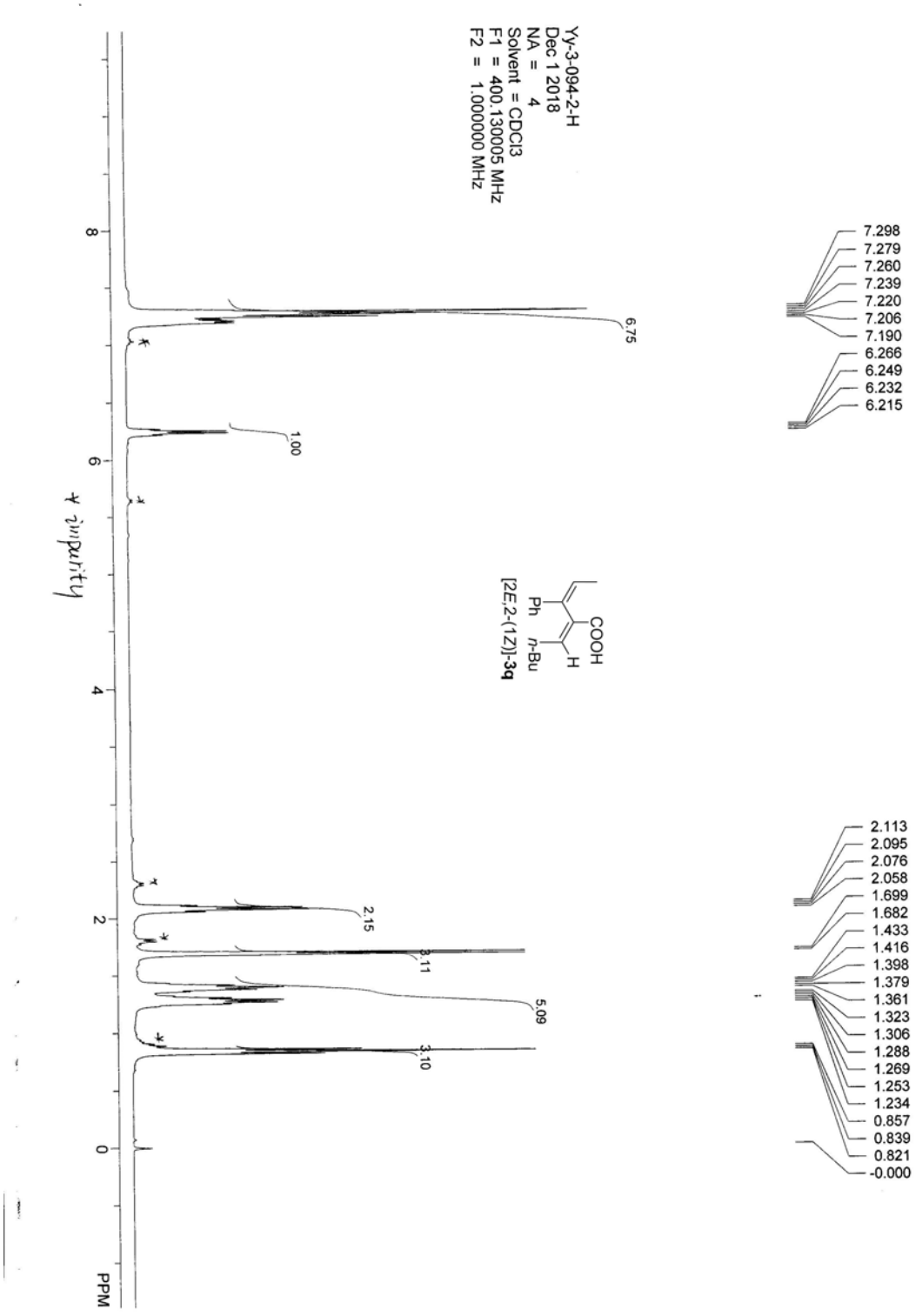

Yy-3-090-p3-C
Nov 27 2018
NA = 256
Solvent = CDCl3
F1 = 100.612770 MHz
F2 = 1.000000 MHz
[2E,2-(1Z)]-3q
Ph
n-Bu
COOH
H
172.266
149.245
140.153
135.057
130.080
128.326
126.896
126.359
125.585
77.316
77.000
76.684
30.279
29.505
22.537
15.514
13.776
200
150
100
50
0
PPM

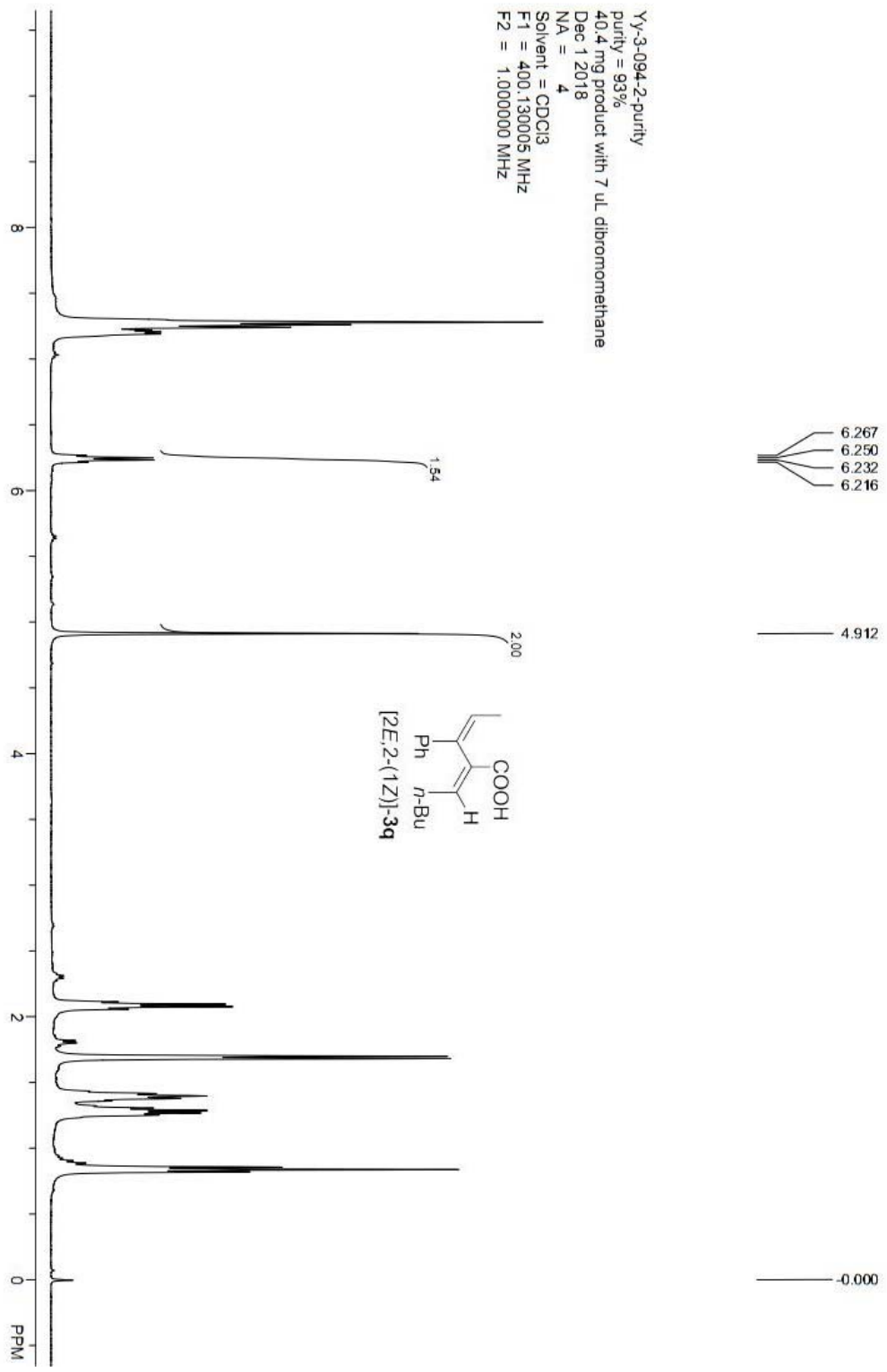

Yy-3-090-p3-purity
purity = 93%
29.2 mg product with 17.5 uL dibromomethane
Nov 27 2018
NA = 4
Solvent = CDCl3
F1 = 400.130005 MHz
F2 = 1.000000 MHz
1.11
5.00
6.284
6.267
6.249
6.232
4.933
-0.000
COOH
H
Ph
n-Bu
[2E,2-(1Z)]-3q
10
8
6
4
2
0
PPM

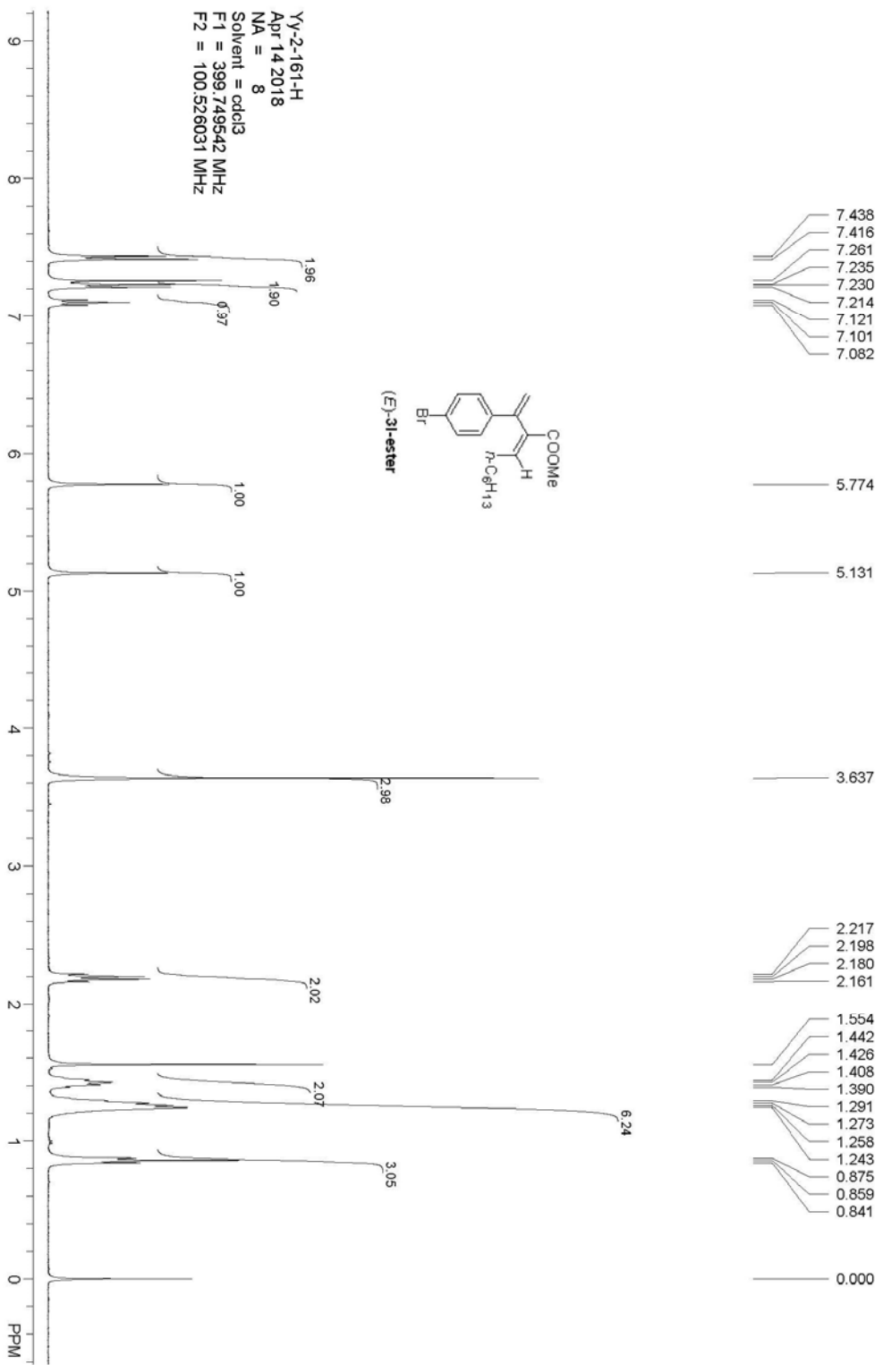


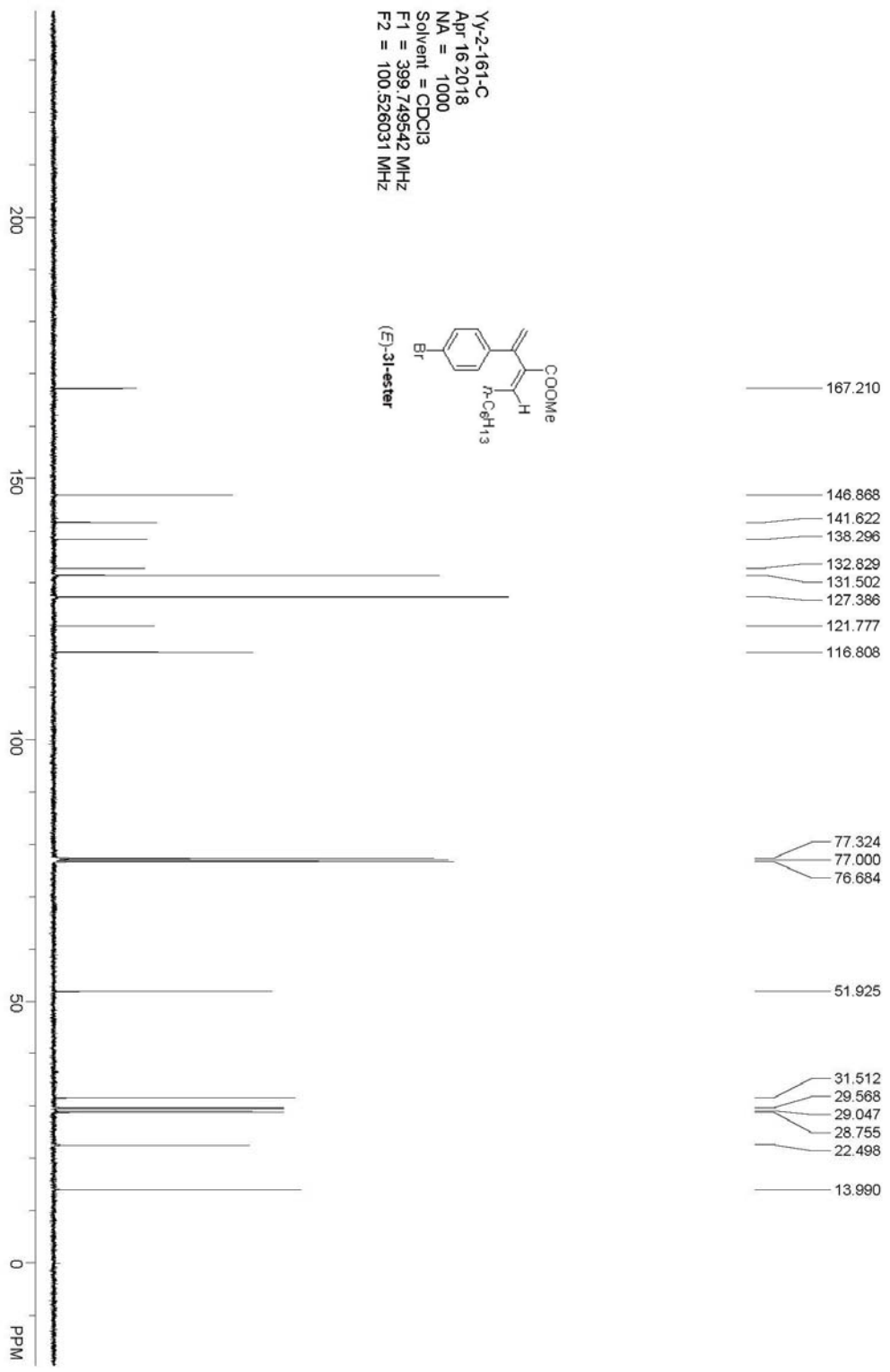
	Parameter	Value
1	Title	Yy-3-090-p3-NOE
2	Origin	Bruker BioSpin GmbH
3	Solvent	C6D6
4	Temperature	297.7
5	Pulse Sequence	noesygpphpp
6	Experiment	NOESY
7	Number of Scans	4
8	Receiver Gain	20
9	Relaxation Delay	1.9836
10	Pulse Width	10.0000
11	Presaturation Frequency	
12	Spectrometer Frequency	(400.13, 400.13)
13	Spectral Width	(3759.4, 3759.4)
14	Lowest Frequency	(-273.1, -273.1)
15	Nucleus	(1H, 1H)
16	Acquired Size	(1024, 256)
17	Spectral Size	(1024, 1024)

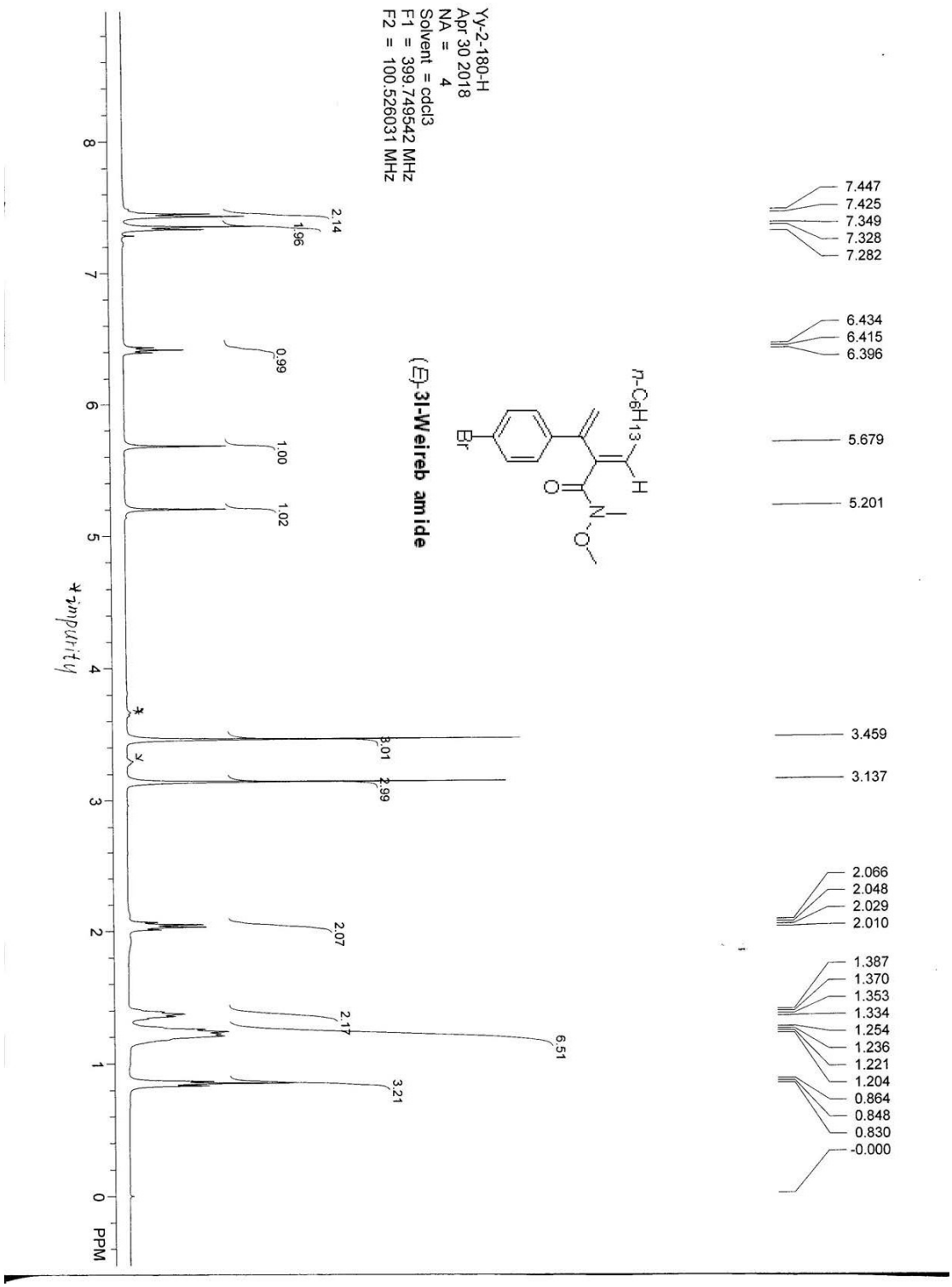

Yy-3-094-1-H
Dec 1 2018
NA = 4
Solvent = CDCl3
F1 = 400.130035 MHz
F2 = 1.000000 MHz
Ph
n-Bu
(Z)-2q
Stabilizer:
7.578
7.559
7.323
7.305
7.286
7.240
7.222
7.204
6.409
6.392
6.374
6.357
2.484
2.467
2.449
2.045
2.027
1.646
1.629
1.611
1.593
1.575
1.544
1.526
1.508
1.489
1.471
1.453
0.968
0.950
0.932
-0.000
2.00
2.01
1.27
1.00
2.02
3.20
4.72
3.10
8
6
4
2
0
PPM

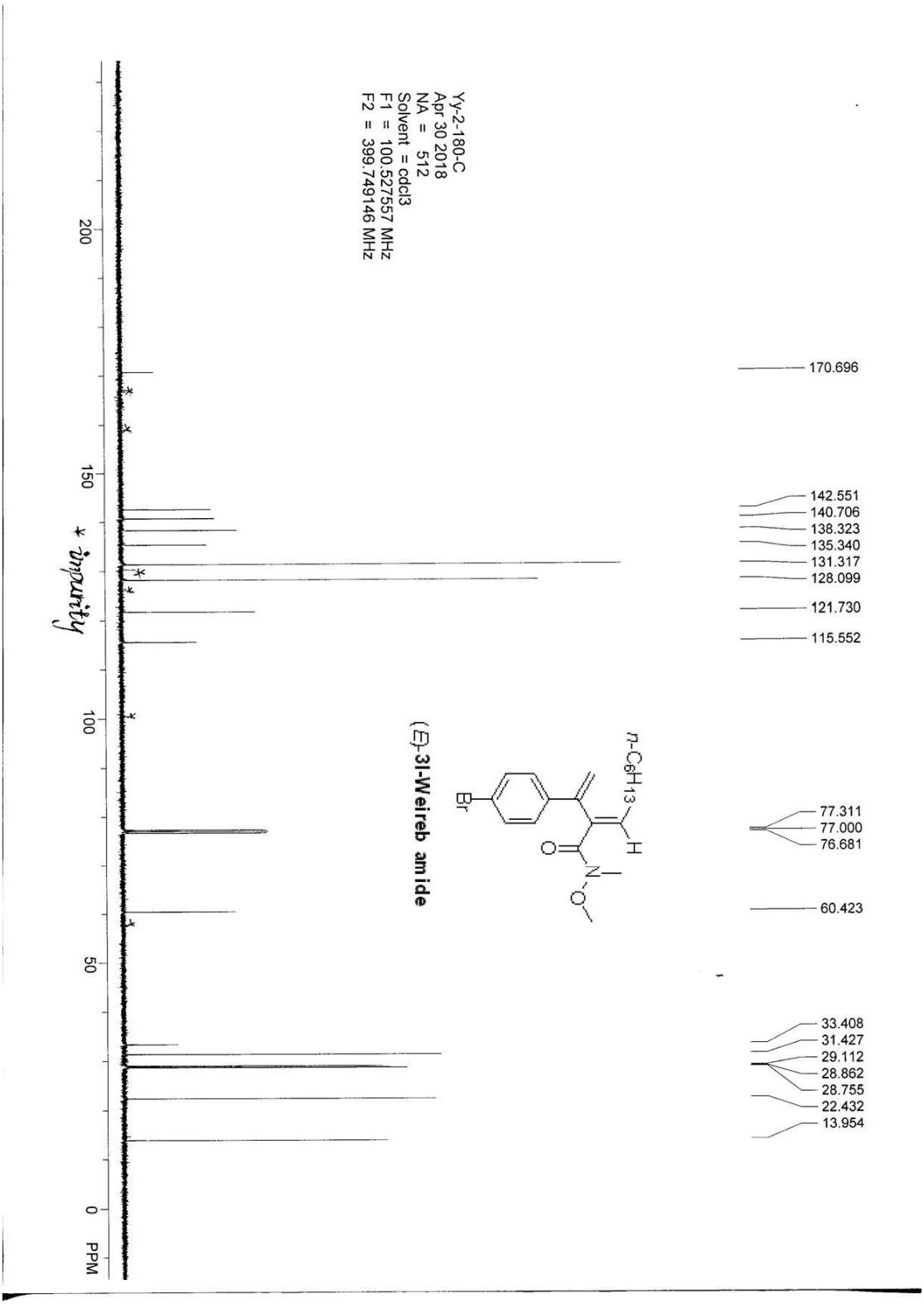

Yy-3-094-2-H
Dec 1 2018
NA = 4
Solvent = CDCl3
F1 = 400.130005 MHz
F2 = 1.000000 MHz
COOH
Ph
n-Bu
H
[2E,2-(1Z)]-3q
7.298
7.279
7.260
7.239
7.220
7.206
7.190
6.266
6.249
6.232
6.215
2.113
2.095
2.076
2.058
1.699
1.682
1.433
1.416
1.398
1.379
1.361
1.323
1.306
1.288
1.269
1.253
1.234
0.857
0.839
0.821
-0.000
6.75
1.00
2.15
3.11
5.09
3.10
PPM

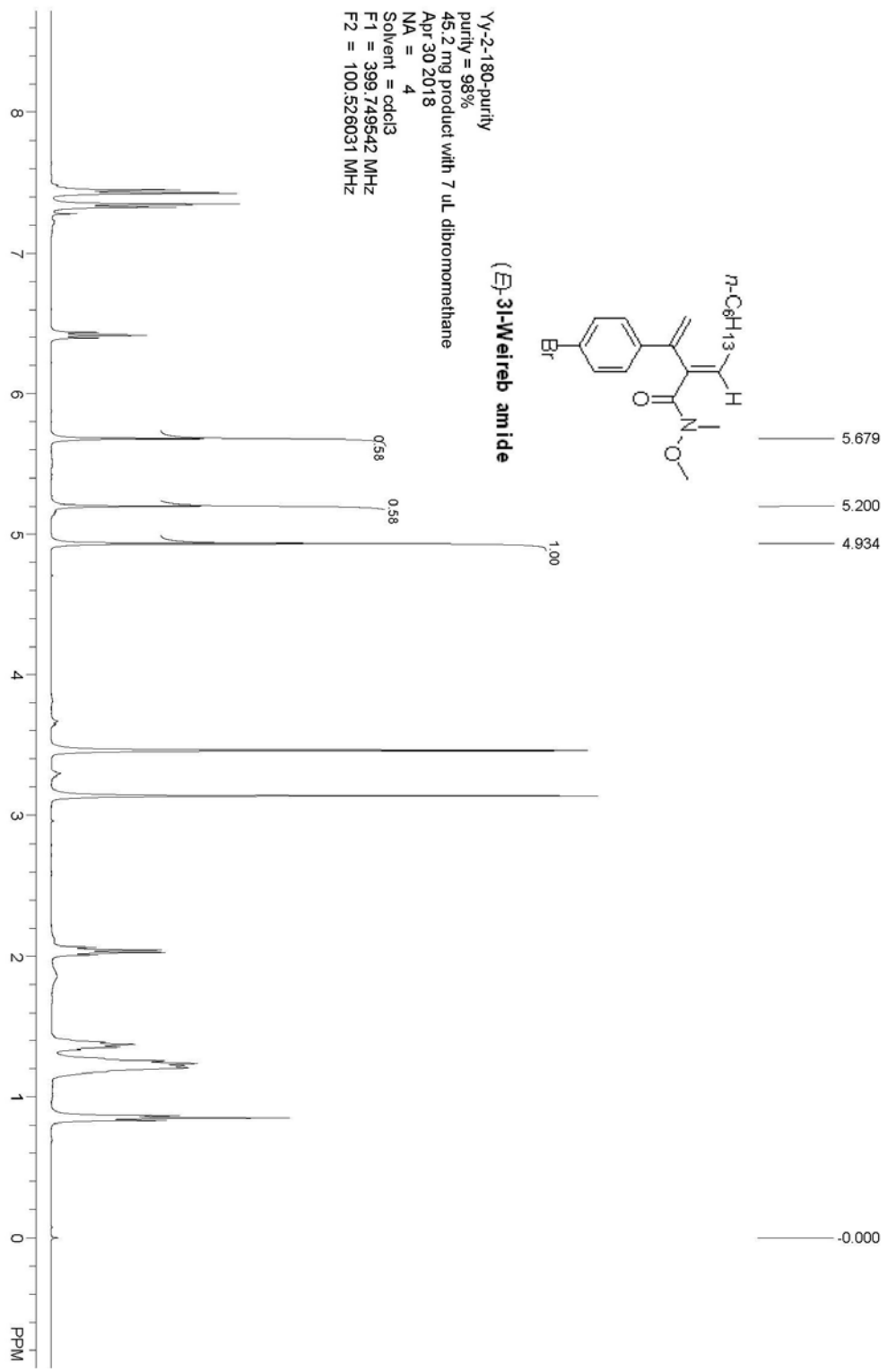

Yy-3-094-2-purity
purity = 93%
40.4 mg product with 7 uL dibromomethane
Dec 1 2018
NA = 4
Solvent = CDCl3
F1 = 400.130005 MHz
F2 = 1.000000 MHz
1.54
2.00
COOH
H
Ph
n-Bu
[2E,2-(1Z)]-3q
6.267
6.250
6.232
6.216
4.912
-0.000
8
6
4
2
0
PPM

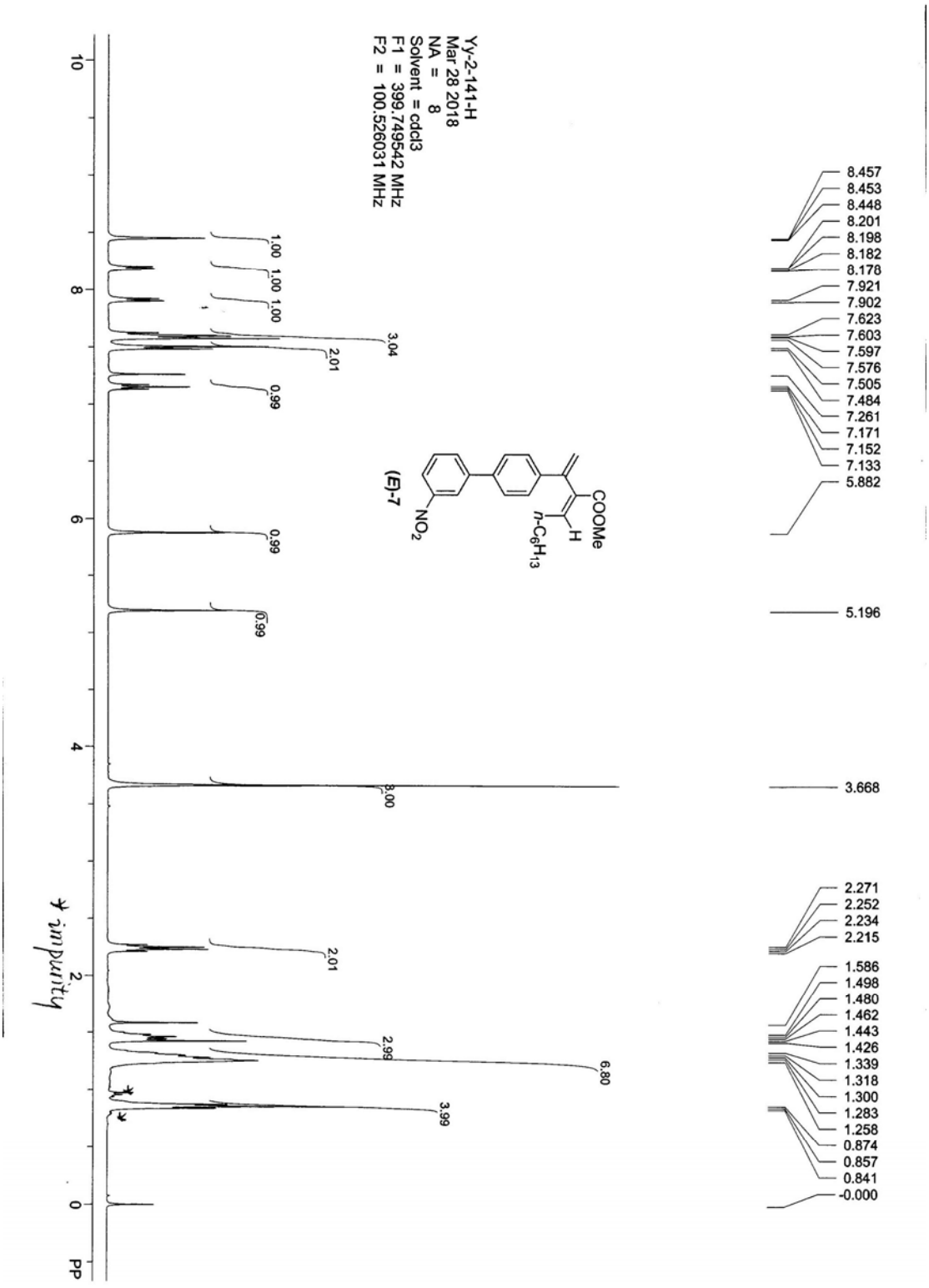

Yy-2-161-H
Apr 14 2018
NA = 8
Solvent = cdcl3
F1 = 399.749542 MHz
F2 = 100.526031 MHz
(E)-3l-ester
Br
COOMe
H
n-C6H13
7.438
7.416
7.261
7.235
7.230
7.214
7.121
7.101
7.082
5.774
5.131
3.637
2.217
2.198
2.180
2.161
1.554
1.442
1.426
1.408
1.390
1.291
1.273
1.258
1.243
0.875
0.859
0.841
0.000
1.96
1.90
0.37
1.00
1.00
2.98
2.02
2.07
6.24
3.05
PPM

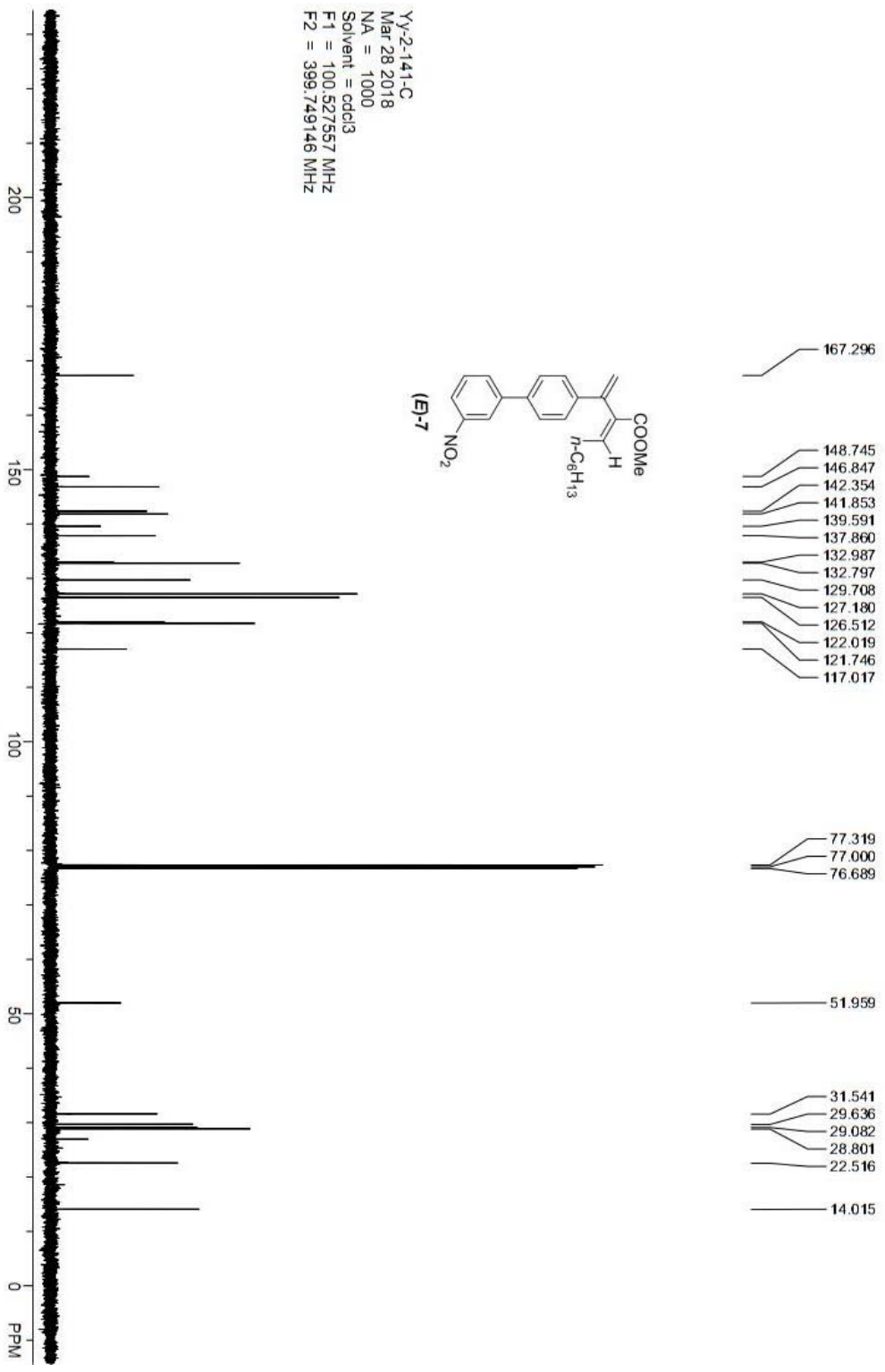

Yy-2-161-C
Apr 16 2018
NA = 1000
Solvent = CDCl3
F1 = 399.749542 MHz
F2 = 100.526031 MHz
(E)-3l-ester
COOMe
H
n-C6H13
Br
167.210
146.868
141.622
138.296
132.829
131.502
127.386
121.777
116.808
77.324
77.000
76.684
51.925
31.512
29.568
29.047
28.755
22.498
13.990
200
150
100
50
0
PPM

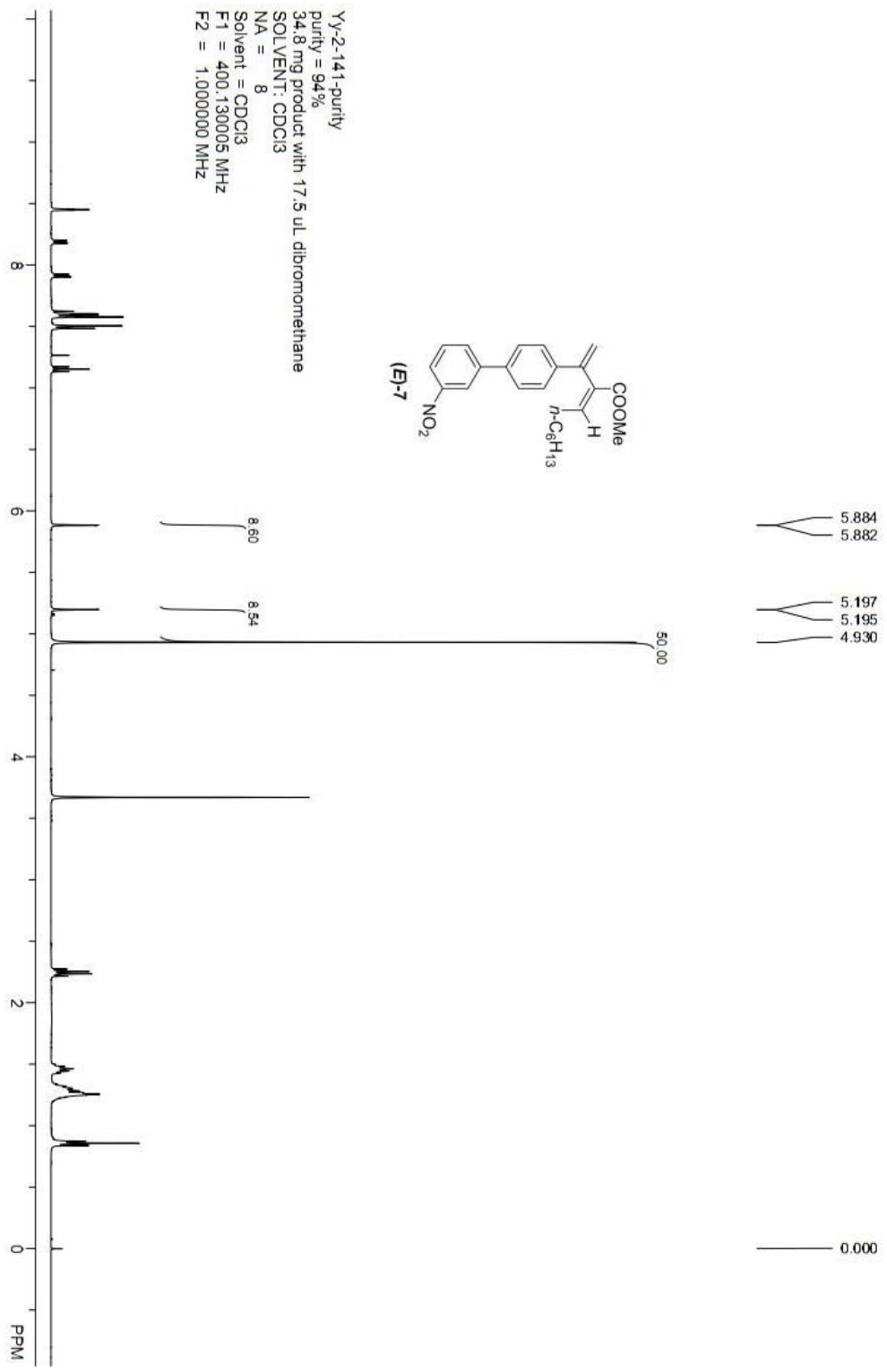

Yy-2-180-H
Apr 30 2018
NA = 4
Solvent = cdcl3
F1 = 399.749542 MHz
F2 = 100.526031 MHz
(E)-3l-Weireb amide
n-C6H13
Br
O
N
O
H
impurity
PPM
7.447
7.425
7.349
7.328
7.282
6.434
6.415
6.396
5.679
5.201
3.459
3.137
2.066
2.048
2.029
2.010
1.387
1.370
1.353
1.334
1.254
1.236
1.221
1.204
0.864
0.848
0.830
-0.000
2.14
1.96
0.99
1.00
1.02
3.01
2.99
2.07
2.17
6.51
3.21

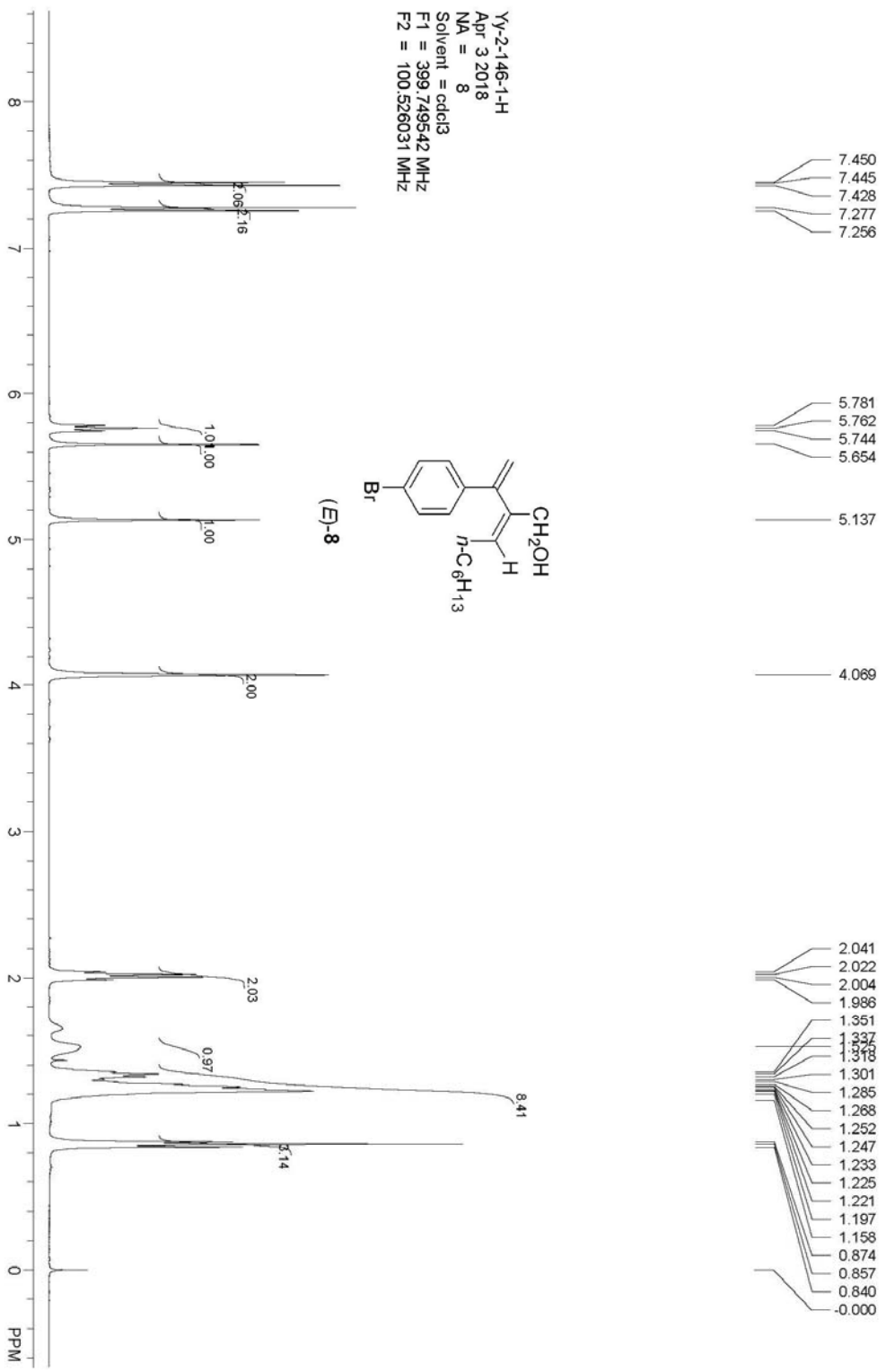

Yy-2-180-C
Apr 30 2018
NA = 512
Solvent = cdcl3
F1 = 100.527557 MHz
F2 = 399.749146 MHz
(E)-3l-Weireb amide
n-C6H13
Br
H
O
N
O
170.696
142.551
140.706
138.323
135.340
131.317
128.099
121.730
115.552
77.311
77.000
76.681
60.423
33.408
31.427
29.112
28.862
28.755
22.432
13.954
* impurity
200
150
100
50
0
PPM

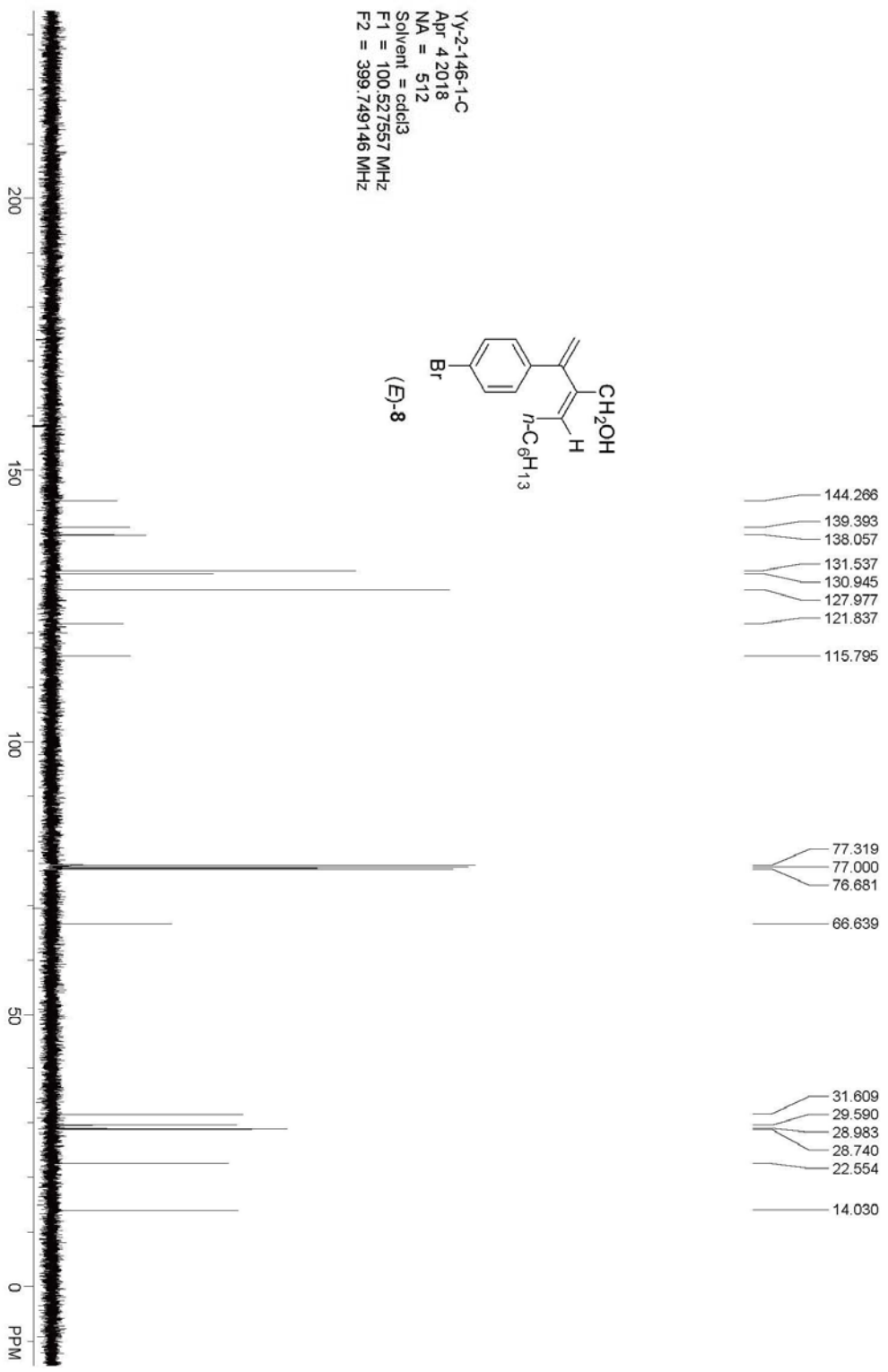

Yy-2-180-purity
purity = 98%
45.2 mg product with 7 uL dibromomethane
Apr 30 2018
NA = 4
Solvent = cdcl3
F1 = 399.749542 MHz
F2 = 100.526031 MHz
(E)-3l-Weireb amide
n-C6H13
H
Br
O
N
O
0.58
0.58
1.00
5.679
5.200
4.934
-0.000
PPM

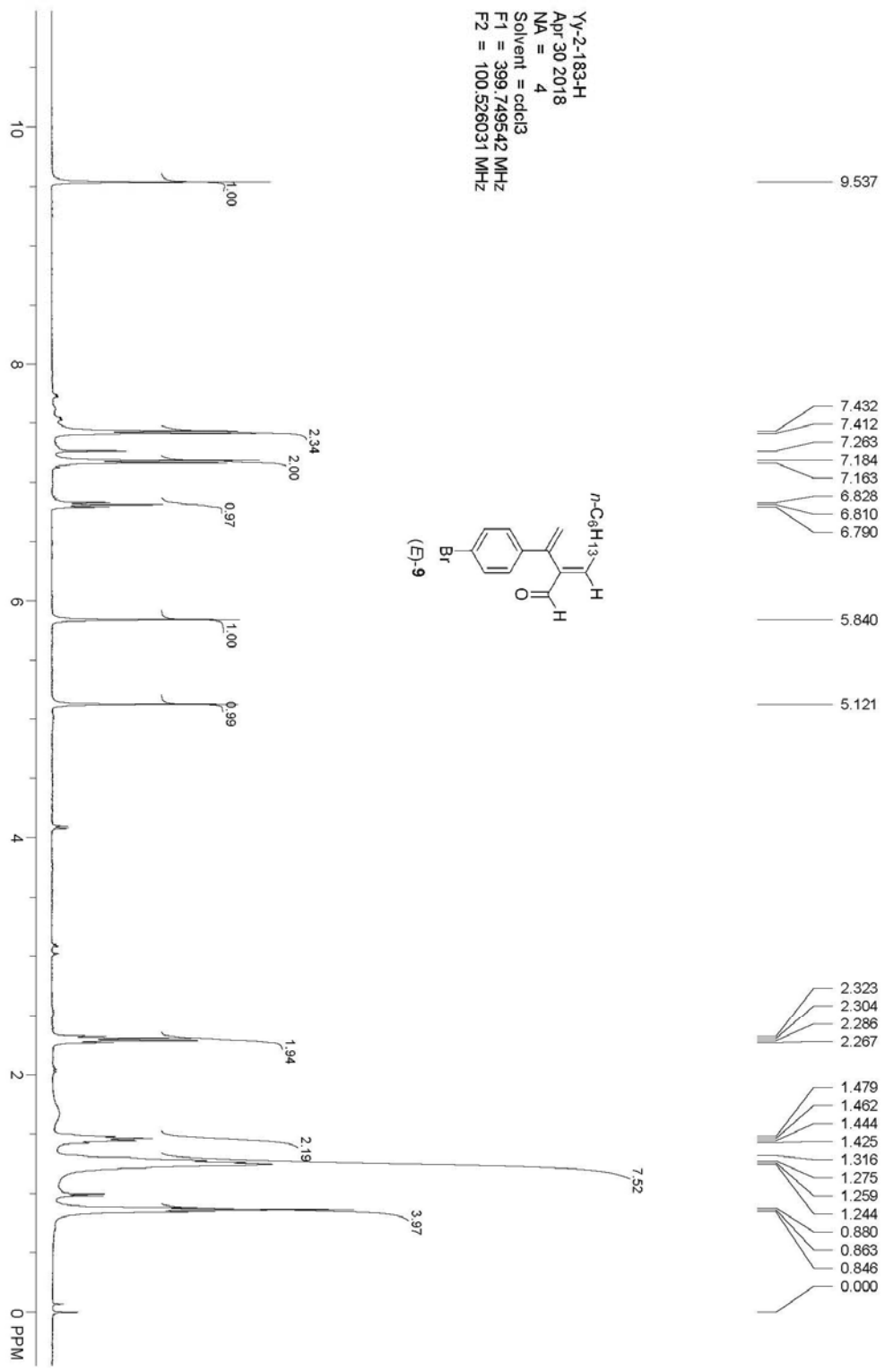

Yy-2-141-H
Mar 28 2018
NA = 8
Solvent = cdcl3
F1 = 399.749542 MHz
F2 = 100.526031 MHz
(E)-7
COOMe
H
n-C6H13
NO2
8.457
8.453
8.448
8.201
8.198
8.182
8.176
7.921
7.902
7.623
7.603
7.597
7.576
7.505
7.484
7.261
7.171
7.152
7.133
5.882
5.196
3.668
2.271
2.252
2.234
2.215
1.586
1.498
1.480
1.462
1.443
1.426
1.339
1.318
1.300
1.283
1.258
0.874
0.857
0.841
-0.000
1.00
1.00
1.00
3.04
2.01
0.99
0.99
0.99
8.00
2.01
2.99
6.80
3.99
* impurity
10
8
6
4
2
0
PP

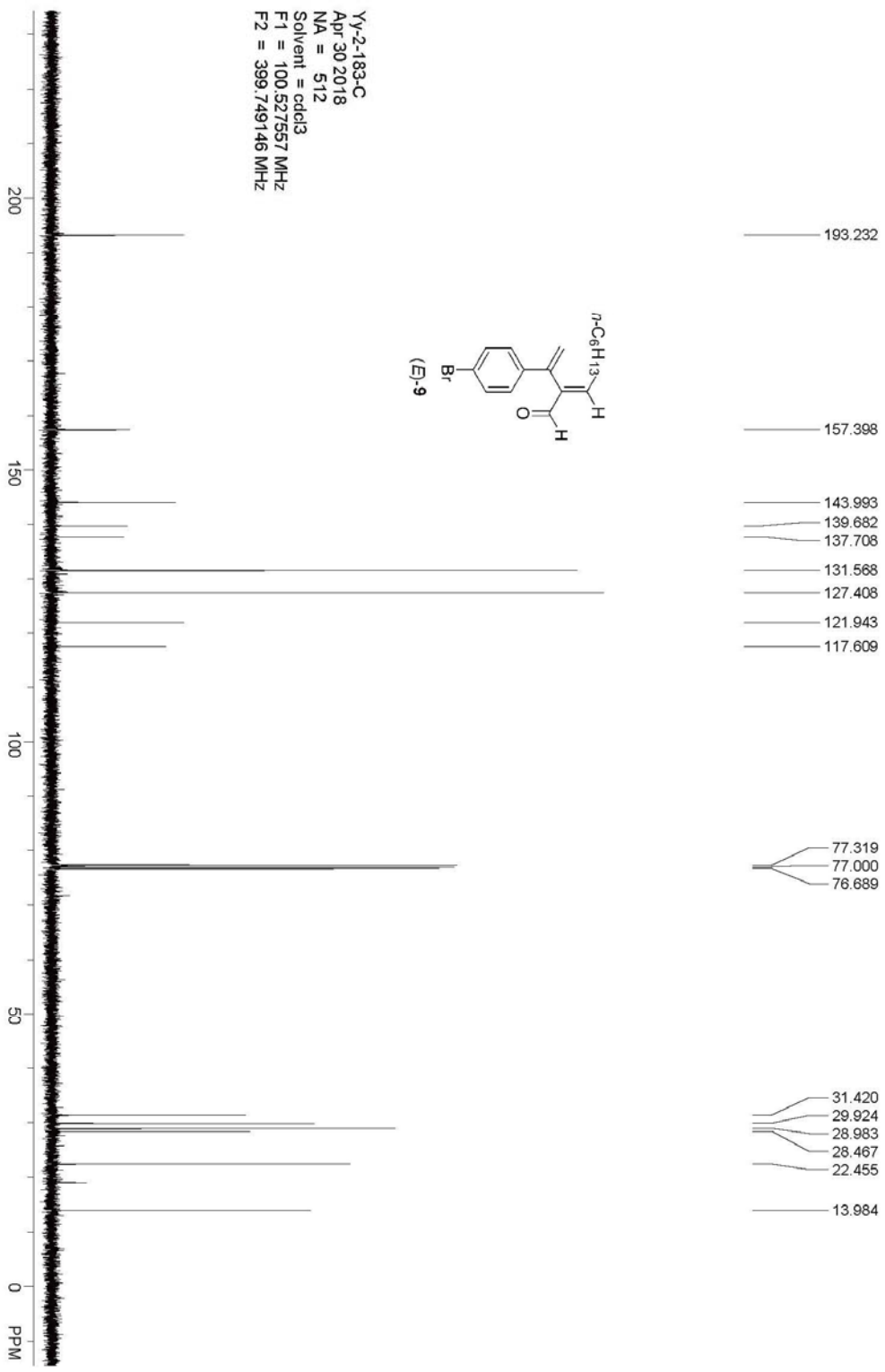

Yy-2-141-C
Mar 28 2018
NA = 1000
Solvent = cdcl3
F1 = 100.527557 MHz
F2 = 399.749146 MHz
(E)-7
NO2
n-C6H13
H
COOMe
167.296
148.745
146.847
142.354
141.853
139.591
137.860
132.987
132.797
129.708
127.180
126.512
122.019
121.746
117.017
77.319
77.000
76.689
51.959
31.541
29.636
29.082
28.801
22.516
14.015
200
150
100
50
0
PPM

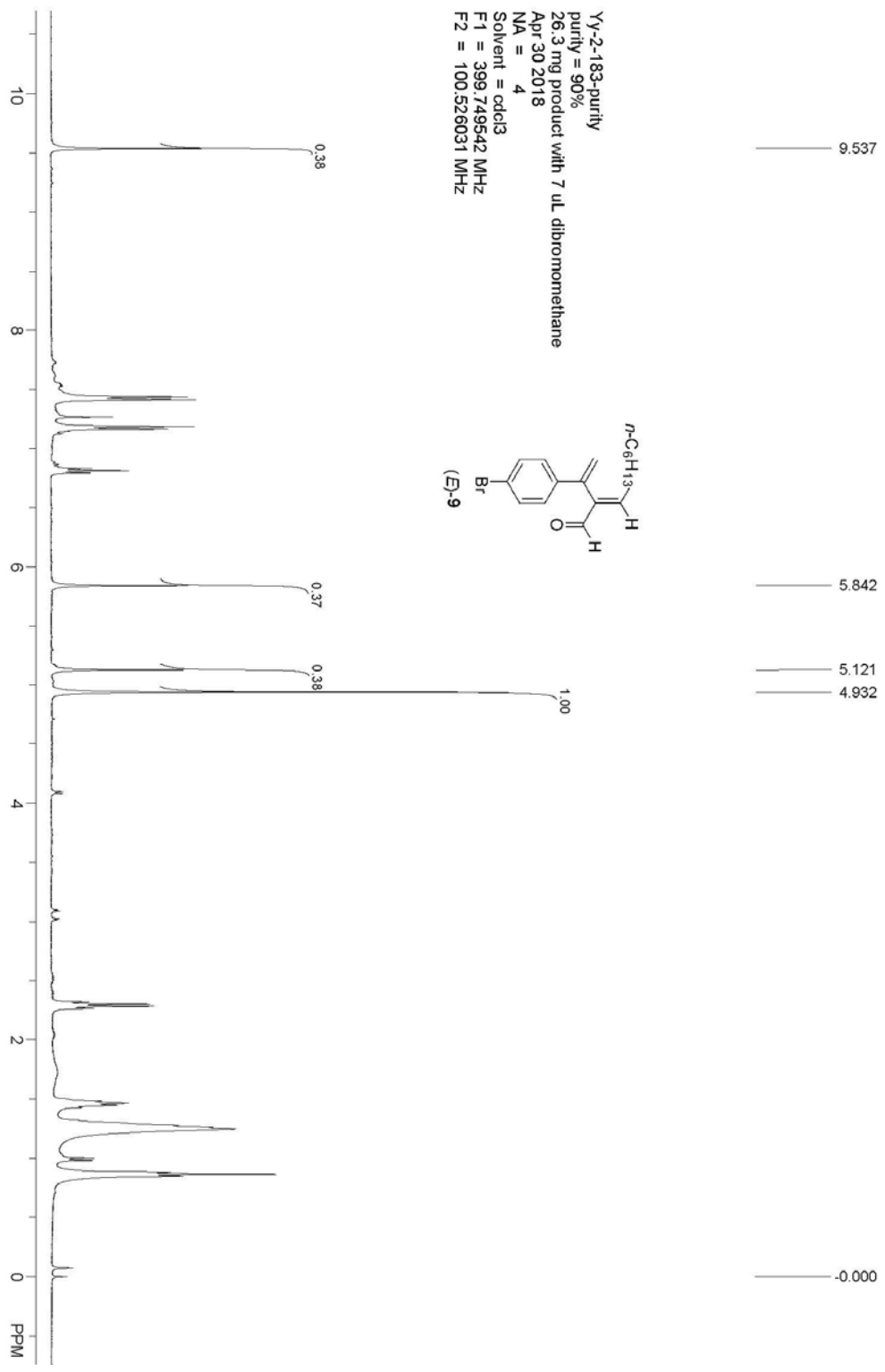

Yy-2-141-purity
purity = 94%
34.8 mg product with 17.5 uL dibromomethane
SOLVENT: CDCl3
NA = 8
Solvent = CDCl3
F1 = 400.130005 MHz
F2 = 1.000000 MHz
(E)-7
COOMe
H
n-C6H13
NO2
8.60
8.54
50.00
5.884
5.882
5.197
5.195
4.930
0.000
8
6
4
2
0
PPM


Yy-2-146-1-H
Apr 3 2018
NA = 8
Solvent = cdcl3
F1 = 399.749542 MHz
F2 = 100.526031 MHz
(E)-8
Br
CH2OH
H
n-C6H13
7.450
7.445
7.428
7.277
7.256
5.781
5.762
5.744
5.654
5.137
4.069
2.041
2.022
2.004
1.986
1.351
1.337
1.318
1.301
1.285
1.268
1.252
1.247
1.233
1.225
1.221
1.197
1.158
0.874
0.857
0.840
-0.000
2.06
2.16
1.01
1.00
1.00
2.00
2.03
0.97
8.41
3.14
PPM


Yy-2-146-1-C
Apr 4 2018
NA = 512
Solvent = cdcl3
F1 = 100.527557 MHz
F2 = 399.749146 MHz
(E)-8
Br
CH2OH
H
n-C6H13
144.266
139.393
138.057
131.537
130.945
127.977
121.837
115.795
77.319
77.000
76.681
66.639
31.609
29.590
28.983
28.740
22.554
14.030
200
150
100
50
0
PPM


Yy-2-183-H
Apr 30 2018
NA = 4
Solvent = cdcl3
F1 = 399.749542 MHz
F2 = 100.526031 MHz
(E)-9
9.537
7.432
7.412
7.263
7.184
7.163
6.828
6.810
6.790
5.840
5.121
2.323
2.304
2.286
2.267
1.479
1.462
1.444
1.425
1.316
1.275
1.259
1.244
0.880
0.863
0.846
0.000
1.00
2.34
2.00
0.97
1.00
0.99
1.94
2.19
7.52
3.97
10
8
6
4
2
0 PPM


Yy-2-183-C
Apr 30 2018
NA = 512
Solvent = cdcl3
F1 = 100.527557 MHz
F2 = 399.749146 MHz
(E)-9
Br
O
H
n-C6H13
H
193.232
157.398
143.993
139.682
137.708
131.568
127.408
121.943
117.609
77.319
77.000
76.689
31.420
29.924
28.983
28.467
22.455
13.984
200
150
100
50
0
PPM


Yy-2-183-purity
purity = 90%
26.3 mg product with 7 uL dibromomethane
Apr 30 2018
NA = 4
Solvent = cdcl3
F1 = 399.749542 MHz
F2 = 100.526031 MHz
(E)-9
9.537
5.842
5.121
4.932
-0.000
0.38
0.37
0.38
1.00
PPM