Ruthenium(II)-Catalyzed C-H Allenylation-Based Approach to Allenoic Acids

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Supporting Information

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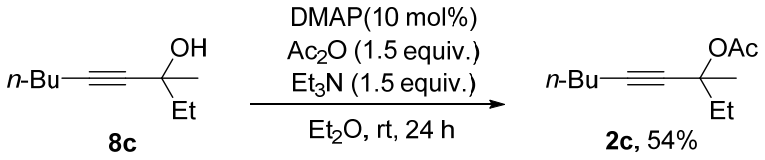
General Information

^{1}H NMR, ^{13}C NMR, and ^{19}F NMR spectra were recorded in $CDCl_3$ using a Bruker AM 300 MHz NMR spectrometer (^{1}H at 300 MHz, ^{13}C at 75 MHz, ^{19}F at 282 MHz) or a Bruker AM 400 MHz NMR spectrometer (^{1}H at 400 MHz, ^{13}C at 100 MHz, ^{19}F at 376 MHz) using TMS (^{1}H, $\delta = 0$), residual $CHCl_3$ (7.26 ppm) in $CDCl_3$, and $CFCl_3$ (^{19}F $CFCl_3$, $\delta = 0$) as the internal standards, respectively. IR spectra were recorded with a Perkin–Elmer 983G instrument. Elemental analyses were measured with a Carlo-Erba EA1110 elementary analysis instrument. Mass spectrometry was performed with an HP 5989A system. High-resolution mass spectrometry was determined with a Finnigan MAT 8430 or Bruker APEXIII instrument. [Ru(*p*-cymene)Cl_2]$_2$ was purchased from *J&K Scientific*. The boiling range of the petroleum ether was 60-90 ^{o}C unless noted otherwise. Other commercially available chemicals including benzoic acids were purchased and used without additional purification unless noted otherwise. Propargylic acetates were prepared according to the literature procedures.[1] The apparatus used in this study is shown as follows:

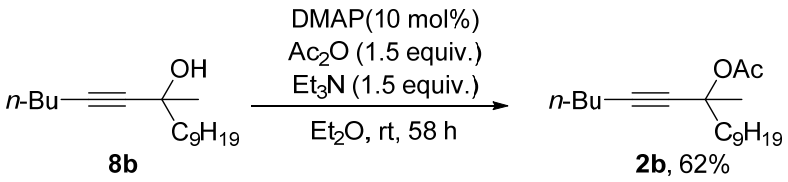
Synthesis of new starting materials

1. Synthesis of 3-methylnon-4-yn-3-yl acetate **2c**. [1] (wxy-2-155)



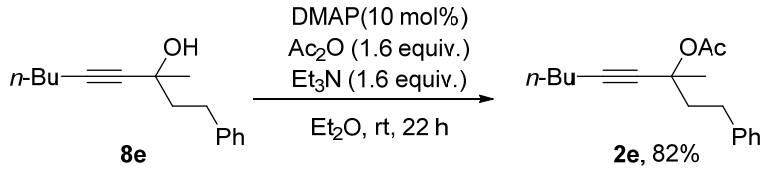
Typical Procedure I: To a dried round flask were added DMAP (0.3065 g, 3.0 mmol), Et_3N (6.4 mL, *d* = 0.73 g/mL, 4.6720 g, 46.1 mmol), **8c** (5.8880 g, 30 mmol)/Et_2O (50 mL), and Ac_2O (4.3 mL, *d* = 1.08 g/mL, 4.6440 g, 45.5 mmol) sequentially. The reaction was complete after 24 h as monitored by TLC (eluent: petroleum ether/ethyl acetate = 25/1). To the resulting mixture was added an aqueous solution of saturated NH_4Cl. The organic phase was separated and the aqueous phase was extracted with 30 mL of ethyl acetate. The combined organic phase then washed with brine and dried over anhydrous Na_2SO_4. After filtration, evaporation of the solvent and chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 25/1, 1500 mL) afforded **2c** (3.1611 g, 54%) as a liquid: ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.22 (t, *J* = 6.9 Hz, 2 H, CH_2), 2.01 (s, 3 H, OAc), 2.00-1.88 (m, 1 H, one proton of CH_2), 1.86-1.73 (m, 1 H, one proton of CH_2), 1.62 (s, 3 H, CH_3), 1.54-1.33 (m, 4 H, $CH_2 \times 2$), 1.00 (t, *J* = 7.5 Hz, 3 H, CH_3), 0.91 (t, *J* = 7.2 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.3, 85.6, 80.2, 76.3, 34.6, 30.7, 26.2, 22.0, 21.8, 18.3, 13.5, 8.5; IR (neat) *v* (cm^{-1}) 2961, 2937, 2875, 2241, 1746, 1464, 1368, 1329, 1304, 1242, 1164, 1138, 1117, 1038, 1015; MS (EI): *m/z* (%) 196 (M^+, 3.47), 154.2 (M^+ - Ac, 99.63), 43 (100); HRMS Calcd for $C_{12}H_{20}O_2$ (M^+): 196.1463; Found: 196.1462.

2. Synthesis of 7-methylhexadec-5-yn-7-yl acetate **2b**. [1] (wxy-2-158)



Following **Typical Procedure I**, the reaction of **8b** (5.8912 g, 20 mmol), DMAP (204.5 mg, 2 mmol), Et_3N (4.2 mL, *d* = 0.73 g/mL, 3.066 g, 30.3 mmol,), and Ac_2O (2.9 mL, *d* = 1.08 g/mL, 3.132 g, 30.7 mmol) in 35 mL Et_2O afforded **2b** (3.6521 g, 62%) (eluent: petroleum ether/ethyl acetate = 25/1, 1500 mL) as a liquid: ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.21 (t, *J* = 6.9 Hz, 2 H, CH_2), 2.00 (s, 3 H, OAc), 1.98-1.85 (m, 1 H, one proton of CH_2), 1.81-1.65 (m, 1 H, one proton of CH_2), 1.63 (s, 3 H, CH_3), 1.52-1.34 (m, 6 H, CH_2 × 3), 1.34-1.19 (m, 12 H, CH_2 × 6), 0.94-0.82 (m, 6 H, CH_3 × 2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.3, 85.5, 80.6, 76.0, 41.7, 31.9, 30.7, 29.52, 29.50, 29.3, 26.7, 24.2, 22.6, 22.0, 21.8, 18.4, 14.0, 13.5; IR (neat) *v* (cm^{-1}) 2955, 2927, 2856, 2245, 1747, 1467, 1367, 1328, 1237, 1166, 1015; MS (EI): *m/z* (%) 294 (M^+, 42.04), 252 (100); HRMS Calcd for $C_{19}H_{34}O_2$ (M^+): 294.2559; Found: 294.2557.

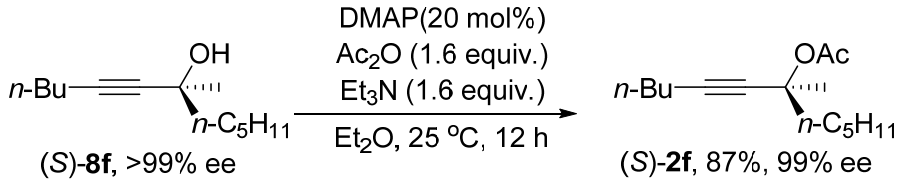
3. Synthesis of 3-methyl-1-phenylnon-4-yn-3-yl acetate **2e**. [1] (wxy-2-189)



Following **Typical Procedure I**, the reaction of **8e** (0.9217 g, 4 mmol), DMAP (41.0 mg, 0.4 mmol), Et_3N (0.9 mL, *d* = 0.73 g/mL, 0.657 g, 6.5 mmol,), and Ac_2O (0.6 mL, *d* = 1.08 g/mL, 0.648 g, 6.4 mmol) in 10 mL Et_2O afforded **2e** (0.8510 g, 82%) (eluent: petroleum ether/ethyl acetate = 30/1, 800 mL) as a liquid: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.33-7.14 (m,

5 H, ArH), 2.81 (t, J = 8.4 Hz, 2 H, CH_2), 2.32-2.19 (m, 3 H, CH_2 and one proton of CH_2), 2.11-2.00 (m, 1 H, one proton of CH_2), 1.99 (s, 3 H, OAc), 1.70 (s, 3 H, CH_3), 1.58-1.36 (m, 4 H, CH_2 × 2), 0.92 (t, J = 7.2 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.3, 141.7, 128.4, 128.3, 125.8, 86.0, 80.1, 75.5, 43.6, 30.8, 30.6, 26.8, 21.9, 21.8, 18.3, 13.5; IR (neat) v (cm^{-1}) 3092, 3063, 3027, 2953, 2934, 2873, 2244, 1747, 1742, 1739, 1733, 1604, 1498, 1455, 1369, 1236, 1169, 1088, 1064, 1015; MS (EI): m/z (%) 272 (M^+, 1.98), 181 (100); HRMS Calcd for $C_{18}H_{24}O_2$ (M^+): 272.1776; Found: 272.1776.

4. Synthesis of (S)-6-methyldodec-7-yn-6-yl acetate (*S*)-**2f**. [1,2] (wxy-3-155)

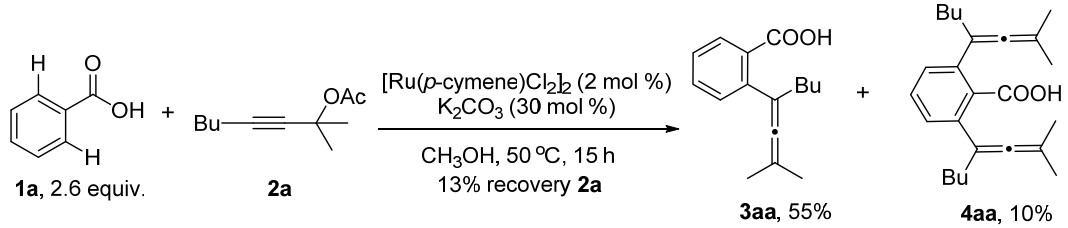


Compound (*S*)-**8f**[2] was prepared by preparative HPLC separation of racemic 6-methyldodec-7-yn-6-ol **8f**: >99% ee (HPLC conditions: Chiralcel AD-H column, hexane/i-PrOH = 99/1, 1.0 mL/min, λ = 214 nm, t_R(major) = 9.8 min, t_R(minor) = 10.7 min); (*S*)-**2f** was prepared following **Typical Procedure I**: the reaction of (*S*)-**8f** (393.7 mg, 2.0 mmol), DMAP (41.0 mg, 0.4 mmol), Et_3N (0.42 mL, d = 0.73 g/mL, 306.6 mg, 3.1 mmol), and Ac_2O (0.3 mL, d = 1.08 g/mL, 324.0 mg, 3.2 mmol) in 2.0 mL Et_2O afforded (*S*)-**8f** (416.1 mg, 87%) (eluent: petroleum ether/ethyl acetate = 50/1, 500 mL) as an oil: 99% ee (HPLC conditions: Chiralcel OZ-H column, *n*-hexane/*i*-PrOH = 100/1, 1.0 mL/min, λ = 214 nm, t_R(major) = 11.4 min, t_R(minor) = 15.9 min); $[\alpha]_D^{20}$ = -31.1 (c = 0.92, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.21 (t, J = 7.1 Hz, 2 H, CH_2), 2.01 (s, 3 H, CH_3), 1.98-1.83 (m, 1 H, one proton of CH_2), 1.81-1.68 (m, 1 H, one proton of CH_2), 1.63 (s, 3 H, CH_3), 1.55-1.21 (m,

10 H, CH_2 × 5), 0.90 (t, J = 7.2 Hz, 6 H, CH_3 × 2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.3, 85.5, 80.5, 76.0, 41.6, 31.7, 30.7, 26.7, 23.9, 22.5, 22.1, 21.8, 18.4, 13.9, 13.5; IR (neat) ν (cm^{-1}) 2958, 2934, 2873, 2249, 1747, 1467, 1367, 1240, 1160, 1122, 1049, 1013; MS (EI): *m/z* (%) 238 (M^+, 0.93), 43 (100); HRMS Calcd for $C_{15}H_{26}O_2$ $(M+Na)^+$: 261.1830; Found: 261.1827.

Ru(II)-Catalyzed C-H Allenylation of Benzoic Acids

1. Synthesis of 2-(2-methylocta-2,3-dien-4-yl)benzoic acid **3aa** and 2,6-bis(2-methylocta-2,3-dien-4-yl)benzoic acid **4aa**. (wxy-2-083, wxy-1-160)



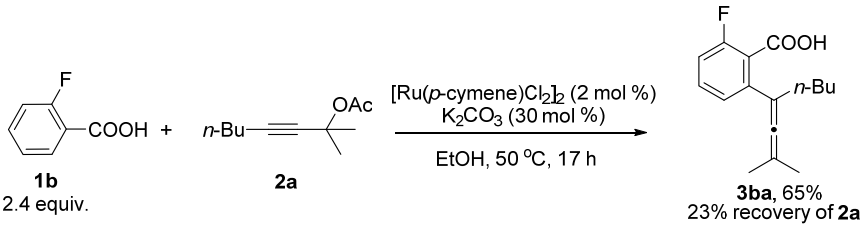
Typical Procedure II: To a dried Schlenk tube were sequentially added benzoic acid **1a** (317.4 mg, 2.6 mmol), K_2CO_3 (41.9 mg, 0.3 mmol), [Ru(*p*-cymene)Cl_2]$_2$ (12.4 mg, 0.02 mmol), 2-methyloct-3-yn-2-yl acetate **2a** (182.5 mg, 1 mmol), and CH_3OH (2.5 mL) in open air atmosphere. The reaction tube was put into an oil bath preheated to 50 ^oC. The reaction was complete after being stirred for 15 h as monitored by TLC. After filteration through a short column of silica gel eluted with ethyl acetate (20 mL × 3) and concentration in vacuo, the crude residual was purified by chromatography on silica gel [eluent: petroleum ether/ethyl acetate = 10/1 (500 mL) to petroleum ether/ethyl acetate = 20/1 (1000 mL)] to afford **3aa** (134.3 mg, 55%) and **4aa** (18.9 mg, 10%). 13% recovery of **2a** was determined by ^1H NMR analysis of the crude product using 35 μL of CH_2Br_2 as the internal standard.

3aa: oil; ^1H NMR (300 MHz, $CDCl_3$) δ 11.80 (bs, 1 H, COOH), 7.81 (dd, J_1 = 7.8 Hz, J_2 =

1.1 Hz, 1 H, ArH), 7.45 (td, J_1 = 7.5 Hz, J_2 = 1.5 Hz, 1 H, ArH), 7.36-7.24 (m, 2 H, ArH), 2.34 (t, J = 7.1 Hz, 2 H, CH_2), 1.71 (s, 6 H, 2 × CH_3), 1.52-1.33 (m, 4 H, 2 × CH_2), 0.91 (t, J = 7.1 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 200.8, 174.8, 141.6, 131.8, 130.1, 129.6, 129.5, 126.3, 103.3, 96.9, 33.3, 30.1, 22.2, 20.1, 14.0; IR (neat) v (cm^{-1}) 3527-2082 (COOH), 1957, 1695, 1598, 1570, 1487, 1451, 1407, 1377, 1362, 1299, 1264, 1138, 1085; MS (EI): m/z (%) 244 (M^+, 6.53), 187 (100); HRMS Cacld. for $C_{16}H_{20}O_2$ (M^+): 244.1463; Found: 244.1465.

4aa: oil; ^{1}H NMR (300 MHz, $CDCl_3$) δ 11.05 (bs, 1 H, COOH), 7.31 (dd, J_1 = 8.3 Hz, J_2 = 7.1 Hz, 1 H, ArH), 7.14 (d, J = 7.2 Hz, 2 H, ArH) , 2.28 (t, J = 7.2 Hz, 4 H, 2 × CH_2), 1.71 (s, 12 H, 4 × CH_3), 1.50-1.30 (m, 8 H, 4 × CH_2), 0.90 (t, J = 7.1 Hz, 6 H, 2 × CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 200.7, 173.6, 138.4, 131.6, 129.0, 126.7, 102.1, 96.9, 33.9, 30.0, 22.3, 20.5, 14.0; IR (neat) v (cm^{-1}) 3402-2211 (COOH), 1959, 1699, 1576, 1456, 1377, 1362, 1286, 1188, 1131; MS (EI): m/z (%) 366 (M^+, 100.00); HRMS Cacld. for $C_{25}H_{34}O_2$ (M^+): 366.2559; Found: 366.2558.

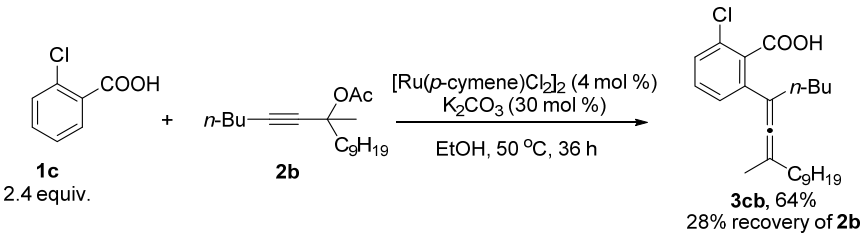
2. Synthesis of 2-fluoro-6-(2-methylocta-2,3-dien-4-yl)benzoic acid **3ba**. (wxy-2-132)



Following **Typical Procedure II**, the reaction of **1b** (336.2 mg, 2.4 mmol), **2a** (182.5 mg, 1 mmol), **2b** (336.2 mg, 1.0 mmol), K_2CO_3 (41.5 mg, 0.3 mmol), and [Ru(p-cymene)Cl_2]$_2$ (12.2 mg, 0.02 mmol) in 2.5 mL of EtOH afforded **3ba** (170.4 mg, 65%) as a solid (eluent:

petroleum ether/ethyl acetate = 20/1, 1500 mL): m.p. 85.9-86.0 °C (petroleum ether/ethyl acetate); 23% recovery of **2b** was determined by ^{1}H NMR analysis of the crude product using 35 μL CH_2Br_2 as the internal standard. ^{1}H NMR (300 MHz, $CDCl_3$) δ 12.20 (bs, 1 H, COOH), 7.35 (td, J_1 = 8.1 Hz, J_2 = 6.0 Hz, 1 H, ArH), 7.14 (d, J = 7.2 Hz, 1 H, ArH), 6.97 (dt, J_1 = 8.7 Hz, J_2 = 0.6 Hz, 1 H, ArH), 2.38 (t, J = 7.2 Hz, 2 H, CH_2), 1.75 (s, 6 H, 2 × CH_3), 1.53-1.32 (m, 4 H, 2 × CH_2), 0.92 (t, J = 7.1 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 202.2, 172.9, 159.6 (d, J = 248.2 Hz), 140.1 (d, J = 2.0 Hz), 131.0 (d, J = 9.0 Hz), 122.6 (d, J = 2.8 Hz), 120.5 (d, J = 15.9 Hz), 113.4 (d, J = 21.4 Hz), 100.9 (d, J = 2.0 Hz), 99.3, 32.2, 30.0, 22.2, 19.7, 14.0; ^{19}F NMR (282 MHz, $CDCl_3$) δ -116.21; IR (neat) ν (cm^{-1}) 3300-2200 (COOH), 1955, 1704, 1699, 1609, 1575, 1456, 1404, 1362, 1296, 1262, 1236, 1125, 1057; Raman ν (cm^{-1}) 1950, 1609; MS (EI): *m/z* (%) 262 (M^+, 14.38), 205 (100); Anal. Calcd. for $C_{16}H_{19}FO_2$ (%): C, 73.26; H, 7.30; Found: C, 72.89; H, 7.19.

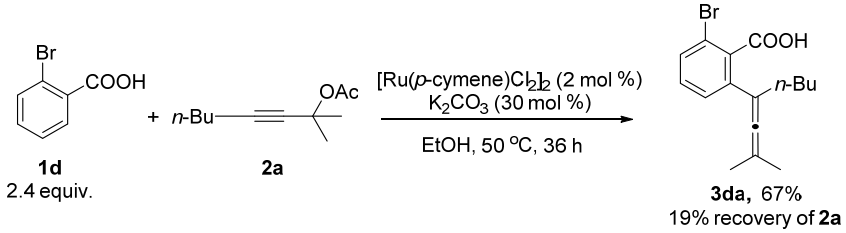
3. Synthesis of 2-chloro-6-(7-methylhexadeca-5,6-dien-5-yl)benzoic acid **3cb**. (wxy-2-195)



Following **Typical Procedure II**, the reaction of **1c** (375.4 mg, 2.4 mmol), **2b** (295.0 mg, 1.0 mmol), K_2CO_3 (41.5 mg, 0.3 mmol), and [Ru(*p*-cymene)Cl_2]$_2$ (24.4 mg, 0.04 mmol) in 2.5 mL of EtOH afforded **3cb** (249.2 mg, 64%) as an oil (eluent: petroleum ether/ethyl acetate/HOAc = 500/30/4, 1500 mL). 28% recovery of **2b** was determined by ^{1}H NMR analysis of the crude product using 35 μL CH_2Br_2 as the internal standard. ^{1}H NMR (300

MHz, $CDCl_3$) δ 11.35 (bs, 1 H, COOH), 7.33-7.20 (m, 3 H, ArH), 2.44-2.28 (m, 2 H, CH_2), 1.99 (m, 2 H, CH_2), 1.77 (s, 3 H, CH_3), 1.51-1.31 (m, 6 H, CH_2 × 3), 1.31-1.11 (m, 12 H, CH_2 × 6), 0.91 (t, J = 6.9 Hz, 3 H, CH_3), 0.87 (t, J = 6.6 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 201.3, 173.7, 139.9, 131.9, 130.9, 130.2, 127.3, 125.8, 103.2, 102.3, 34.0, 33.0, 31.9, 30.1, 29.6, 29.5, 29.29, 29.27, 27.3, 22.6, 22.3, 18.3, 14.1, 13.9; IR (neat) ν (cm^{-1}) 3535-2138 (COOH), 1952, 1704, 1700, 1588, 1564, 1464, 1398, 1287, 1189, 1154, 1129; MS (EI): *m/z* (%) 392 [M^+(^{37}Cl), 2.62], 390 [M^+(^{35}Cl), 7.42], 333 (100); HRMS Calcd for $C_{24}H_{35}O_2{}^{35}Cl$ (M^+): 390.2326; Found: 390.2327.

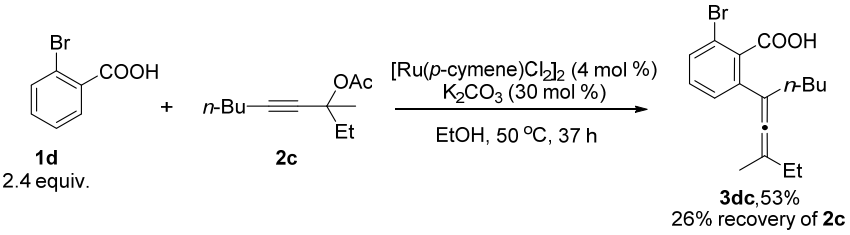
4. Synthesis of 2-bromo-6-(2-methylocta-2,3-dien-4-yl)benzoic acid **3da**. (wxy-2-190)



Following **Typical Procedure II**, the reaction of **1d** (482.4 mg, 2.4 mmol), **2a** (182.5 mg, 1.0 mmol), K_2CO_3 (41.5 mg, 0.3 mmol), and [Ru(*p*-cymene)Cl_2]$_2$ (12.3 mg, 0.02 mmol) in 2.5 mL of EtOH afforded **3da** (216.0 mg, 67%) as a solid (first round eluent: petroleum ether/ethyl acetate/AcOH = 500/30/4, 1000 mL, the impure part was further purified in second round, eluent: petroleum ether/ethyl acetate/AcOH = 500/30/4, 1000 mL): m.p. 85.2-85.3 °C (petroleum ether/DCM). 19% recovery of **2a** was determined by ^{1}H NMR analysis of the crude product using 35 μL CH_2Br_2 as the internal standard. ^{1}H NMR (300 MHz, $CDCl_3$) δ 11.15 (bs, 1 H, COOH), 7.45 (dd, J_1 = 7.8 Hz, J_2 = 1.2 Hz, 1 H, ArH), 7.28 (dd, J_1 = 8.0 Hz, J_2 = 1.4 Hz, 1 H, ArH), 7.22 (t, J = 7.5 Hz, 1 H, ArH), 2.34 (t, J = 7.2 Hz, 2 H,

CH_2), 1.75 (s, 6 H, CH_3 × 2), 1.51-1.30 (m, 4 H, CH_2 × 2), 0.91 (t, J = 7.2 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 201.4, 174.1, 140.1, 134.0, 130.6, 130.5, 126.5, 119.3, 101.2, 98.7, 33.0, 29.9, 22.2, 20.3, 14.0; IR (neat) ν (cm^{-1}) 3471-2168 (COOH), 1955, 1705, 1588, 1557, 1443, 1288, 1186, 1150, 1124; MS (EI): *m/z* (%) 324 [M^+(^{81}Br), 1.85], 322 [M^+(^{79}Br), 2.45], 265 (100); Anal. Calcd. for $C_{16}H_{19}BrO_2$ (%): C, 59.45; H, 5.93; Found: C, 59.42; H, 6.00.

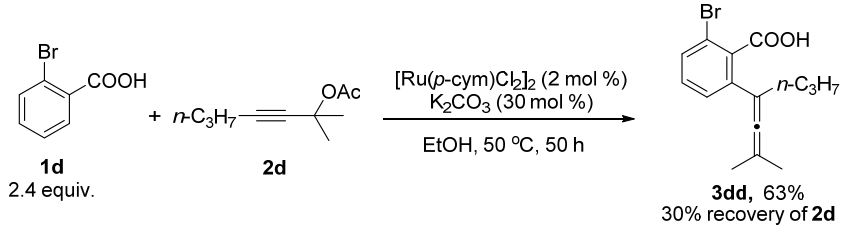
5. Synthesis of 2-bromo-6-(3-methylnona-3,4-dien-5-yl)benzoic acid **3dc**. (wxy-2-186, wxy-3-016)



Following **Typical Procedure II**, the reaction of **1d** (478.9 mg, 2.4 mmol), **2c** (196.3 mg, 1.0 mmol), K_2CO_3 (41.5 mg, 0.3 mmol), and [Ru(*p*-cymene)Cl_2]$_2$ (24.5 mg, 0.04 mmol) in 2.5 mL of EtOH afforded **3dc** (179.9 mg, 53%) as an oil (eluent: petroleum ether/ethyl acetate = 10/1, 1000 mL). 26% recovery of **2c** was determined by ^1H NMR analysis of the crude product using 35 μL CH_2Br_2 as the internal standard. ^1H NMR (300 MHz, $CDCl_3$) δ 11.02 (bs, 1 H, COOH), 7.45 (dd, J_1 = 7.5 Hz, J_2 = 0.9 Hz, 1 H, ArH), 7.32-7.17 (m, 2 H, ArH), 2.35 (t, J = 7.1 Hz, 2 H, CH_2), 2.12-1.86 (m, 2 H, CH_2), 1.78 (s, 3 H, CH_3), 1.54-1.30 (m, 4 H, CH_2 × 2), 0.99 (t, J = 7.4 Hz, 3 H, CH_3), 0.91 (t, J = 6.9 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 200.6, 174.1, 140.1, 134.0, 130.52, 130.46, 126.6, 119.2, 104.7, 103.1, 33.2, 30.0, 27.2, 22.2, 18.5, 13.9, 12.1; IR (neat) ν (cm^{-1}) 3561-2142 (COOH), 1953, 1700, 1587, 1558, 1455, 1446, 1398, 1376, 1287, 1185, 1152, 1124, 1057; MS (EI): *m/z* (%) 338

[M^+(^{81}Br), 7.88], 336 [M^+(^{79}Br), 8.92], 279 (100); HRMS Calcd for $C_{17}H_{21}O_2{}^{79}Br$ (M^+): 336.0725; Found: 336.0726.

6. Synthesis of 2-bromo-6-(2-methylhepta-2,3-dien-4-yl)benzoic acid **3dd**. (wxy-3-022)

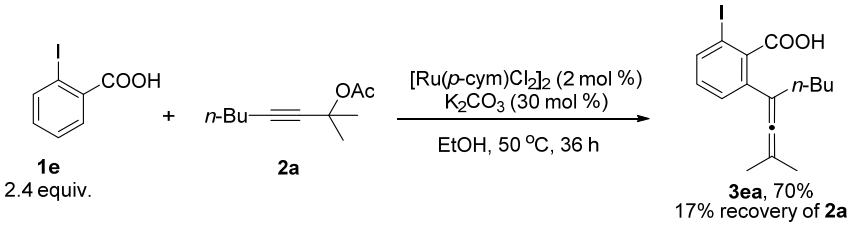


1d 2.4 equiv. **2d**

3dd, 63%
30% recovery of **2d**

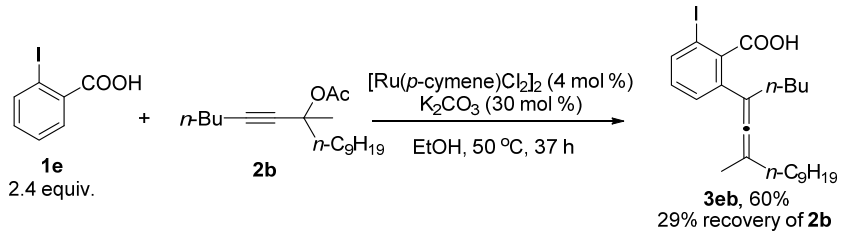
Following **Typical Procedure II**, the reaction of **1d** (481.4 mg, 2.4 mmol), **2d** (168.2 mg, 1.0 mmol), K_2CO_3 (41.4 mg, 0.3 mmol), and [Ru(*p*-cymene)Cl_2]$_2$ (12.5 mg, 0.02 mmol) in 2.5 mL of EtOH afforded **3dd** (195.1 mg, 63%) as a solid (eluent: petroleum ether /ethyl acetate = 15/1, 1000 mL): m.p. 95.5-99.1 ^{o}C (determined without recrystallization. Recrystallization is not possible). 30% recovery of **2d** was determined by ^{1}H NMR analysis of the crude product using 35 μL CH_2Br_2 as the internal standard. ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.64 (bs, 1 H, COOH), 7.45 (dd, J_1 = 7.8 Hz, J_2 = 1.5 Hz, 1 H, ArH), 7.29 (dd, J_1 = 7.8 Hz, J_2 = 1.5 Hz, 1 H, ArH), 7.23 (t, J = 7.8 Hz, 1 H, ArH), 2.32 (t, J = 7.5 Hz, 2 H, CH_2), 1.75 (s, 6 H, CH_3 × 2), 1.55-1.40 (m, 2 H, CH_2), 0.96 (t, J = 7.4 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 201.5, 173.9, 140.1, 134.0, 130.6, 130.5, 126.5, 119.3, 101.1, 98.7, 35.4, 21.0, 20.3, 13.7; IR (neat) v (cm^{-1}) 3587-2125 (COOH), 1957, 1699, 1588, 1558, 1447, 1294, 1182, 1152, 1124; MS (EI): *m/z* (%) 310 [M^+(^{81}Br), 1.70], 308 [M^+(^{79}Br), 2.03], 263 (100); Anal. Calcd. for $C_{15}H_{17}BrO_2$ (%): C, 58.27; H, 5.54; Found: C, 58.38; H, 5.71.

7. Synthesis of 2-iodo-6-(2-methylocta-2,3-dien-4-yl)benzoic acid **3ea**. (wxy-2-174)



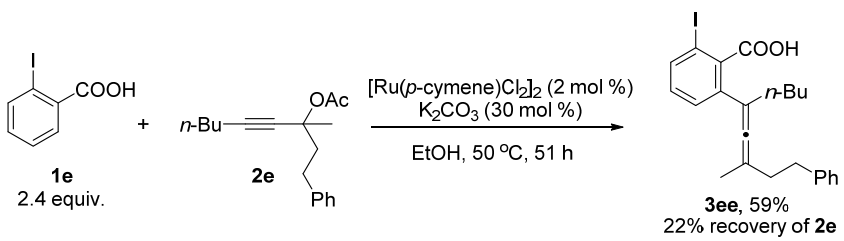
Following **Typical Procedure II**, the reaction of **1e** (595.2 mg, 2.4 mmol), **2a** (182.4 mg, 1.0 mmol), K_2CO_3 (41.5 mg, 0.3 mmol), and [Ru(*p*-cymene)Cl_2]$_2$ (12.3 mg, 0.02 mmol) in 2.5 mL of EtOH afforded **3ea** (258.7 mg, 70%) as a solid (first round eluent: petroleum ether/ethyl acetate/HOAc = 50/30/4, 1500 mL, the impure part was further purified in second round, eluent: petroleum ether/ethyl acetate/HOAc = 50/30/4, 1500 mL): m. p. 106.1-106.8 ^{o}C (petroleum ether/DCM). 17% recovery of **2a** was determined by ^{1}H NMR analysis of the crude product using 35 μL CH_2Br_2 as the internal standard. ^{1}H NMR (300 MHz, $CDCl_3$) δ 11.62 (bs, 1 H, COOH), 7.70 (d, *J* = 8.1 Hz, 1 H, ArH), 7.29 (d, *J* = 7.8 Hz, 1 H, ArH), 7.04 (t, *J* = 7.8 Hz, 1 H, ArH), 2.32 (t, *J* = 7.2 Hz, 2 H, CH_2), 1.75 (s, 6 H, CH_3 × 2), 1.51-1.30 (m, 4 H, CH_2 × 2), 0.90 (t, *J* = 7.1 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 201.1, 175.3, 139.8, 138.0, 137.2, 130.6, 127.5, 101.5, 98.3, 92.1, 33.1, 29.8, 22.1, 20.5, 14.0; IR (neat) *v* (cm^{-1}) 3500-2000 (COOH), 1699, 1584, 1551, 1443, 1395, 1299, 1184, 1146, 1395, 1299, 1184, 1146, 1122; MS (EI): *m/z* (%) 370 (M^+, 1.66), 313 (100); Anal. Calcd. for $C_{16}H_{19}IO_2$ (%): C, 51.91; H, 5.17; Found: C, 51.89; H, 5.17.

8. Synthesis of 2-iodo-6-(7-methylhexadeca-5,6-dien-5-yl)benzoic acid **3eb**. (wxy-2-179, wxy-3-017)



Following **Typical Procedure II**, the reaction of **1e** (595.3 mg, 2.4 mmol), **2b** (294.6 mg, 1.0 mmol), K_2CO_3 (41.5 mg, 0.3 mmol), and [Ru(*p*-cymene)Cl_2]$_2$ (24.5 mg, 0.04 mmol) in 2.5 mL of EtOH afforded **3eb** (287.5 mg, 60%) as an oil (eluent: petroleum ether/ethyl acetate = 10/1, 1000 mL). 29% recovery of **2b** was determined by ^{1}H NMR analysis of the crude product using 35 μL CH_2Br_2 as the internal standard. ^{1}H NMR (300 MHz, $CDCl_3$) δ 11.87 (bs, 1 H, COOH), 7.68 (d, *J* = 7.8 Hz, 1 H, ArH), 7.27 (d, *J* = 7.5 Hz, 1 H, ArH), 7.02 (t, *J* = 8.0 Hz, 1 H, ArH), 2.34 (t, *J* = 7.1 Hz, 2 H, CH_2), 1.98 (t, *J* = 7.1 Hz, 2 H, CH_2), 1.79 (s, 3 H, CH_3), 1.60-1.10 (m, 18 H, CH_2 × 9), 0.99-0.75 (m, 6 H, CH_3 × 2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 200.8, 175.3, 139.9, 138.0, 137.1, 130.5, 127.5, 102.8, 102.6, 92.2, 34.0, 33.2, 31.9, 30.0, 29.6, 29.5, 29.2, 27.3, 22.6, 22.2, 18.8, 14.1, 14.0; IR (neat) *v* (cm^{-1}) 3550-2100 (COOH), 1953, 1704, 1581, 1551, 1456, 1378, 1286; MS (EI): *m/z* (%) 482 (M^+, 14.26), 425 (100); HRMS Calcd for $C_{24}H_{35}O_2I$ (M^+): 482.1682; Found: 482.1679.

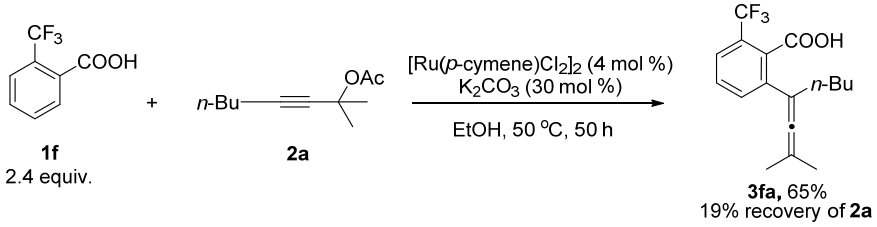
9. Synthesis of 2-iodo-6-(3-methyl-1-phenylnona-3,4-dien-5-yl)benzoic acid **3ee**. (wxy-2-196)



Following **Typical Procedure II**, the reaction of **1e** (593.0 mg, 2.4 mmol), **2e** (268.9 mg,

1.0 mmol), K_2CO_3 (41.8 mg, 0.3 mmol), and [Ru(*p*-cymene)Cl_2]$_2$ (12.3 mg, 0.02 mmol) in 2.5 mL of EtOH afforded **3ee** (270.1 mg, 59%) as an oil (eluent: petroleum ether/ethyl acetate /HOAc = 500/30/4，1500 mL). 22% recovery of **2e** was determined by ^{1}H NMR analysis of the crude product using 35 μL CH_2Br_2 as the internal standard. ^{1}H NMR (300 MHz, $CDCl_3$) δ 11.09 (bs, 1 H, COOH), 7.70 (dd, J_1 = 8.1 Hz, J_2 = 1.1 Hz, 1 H, ArH), 7.26-7.06 (m, 6 H, ArH), 7.02 (t, J = 7.8 Hz, 1 H, ArH), 2.80-2.61 (m, 2 H, CH_2), 2.39-2.18 (m, 4 H, CH_2 × 2), 1.82 (s, 3 H, CH_3), 1.41-1.24 (m, 4 H, CH_2 × 2), 0.88 (t, J = 7.1 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 200.9, 175.1, 141.8, 139.7, 137.9, 137.3, 130.7, 128.3, 128.1, 127.6, 125.7, 103.6, 102.1, 92.2, 35.6, 33.6, 33.2, 29.9, 22.2, 19.0, 14.0; IR (neat) v (cm^{-1}) 3578-2142 (COOH), 1946, 1700, 1581, 1552, 1495, 1454, 1286, 1188, 1145, 1122; MS (EI): *m/z* (%) 461 (M^++1, 1.04), 460 (M^+, 3.92), 91.2 (100); HRMS Calcd for $C_{23}H_{25}O_2I$ (M^+): 460.0899; Found: 460.0902.

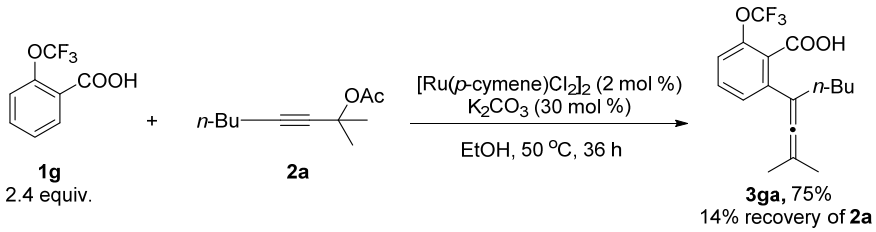
10. Synthesis of 2-(2-methylocta-2,3-dien-4-yl)-6-(trifluoromethyl)benzoic acid **3fa**. (wxy-3-037)



Following **Typical Procedure II**, the reaction of **1f** (456.0 mg, 2.4 mmol), **2a** (182.7 mg, 1.0 mmol), K_2CO_3 (41.7 mg, 0.3 mmol), and [Ru(*p*-cymene)Cl_2]$_2$ (24.5 mg, 0.04 mmol) in 2.5 mL of EtOH afforded **3fa** (203.9 mg, 65%) as a solid (eluent: petroleum ether/ethyl acetate = 10/1, 1300 mL): m.p. 92.0-93.0 °C (petroleum ether/DCM); 19% recovery of **2a**

was determined by ^{1}H NMR analysis of the crude product using 35 μL CH_2Br_2 as the internal standard. ^{1}H NMR (300 MHz, $CDCl_3$) δ 11.49 (bs, 1 H, COOH), 7.62-7.46 (m, 3 H, ArH), 2.36 (t, *J* = 7.2 Hz, 2 H, CH_2), 1.76 (s, 6 H, CH_3 × 2), 1.55-1.35 (m, 4 H, CH_2 × 2), 0.94 (t, *J* = 7.1 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 201.1, 174.2, 140.0, 131.9, 130.4 (q, *J* = 2.1 Hz), 129.5, 127.7 (q, *J* = 31.7 Hz), 124.1 (q, *J* = 4.8 Hz), 123.5 (q, *J* = 287.7 Hz), 101.0, 98.4, 33.7, 29.8, 22.2, 20.1, 13.9; ^{19}F NMR (282 MHz, $CDCl_3$) δ -59.7; IR (neat) *v* (cm^{-1}) 3557-2155 (COOH), 1961, 1714, 1700, 1597, 1583, 1464, 1398, 1363, 1319, 1291, 1190, 1169, 1138, 1066; MS (EI): *m/z* (%) 312 (M^+, 3.00), 251 (100); Anal. Calcd. for $C_{17}H_{19}F_3O_2$ (%): C, 65.37; H, 6.13; Found: C, 65.34; H, 6.15.

11. Synthesis of 2-(2-methylocta-2,3-dien-4-yl)-6-(trifluoromethoxy)benzoic acid **3ga**. (wxy-3-036)

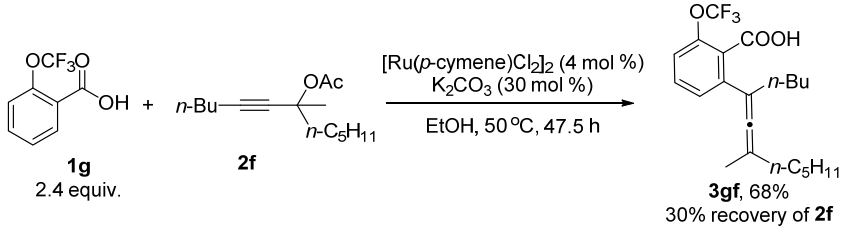


1g 2.4 equiv. **2a** **3ga,** 75% 14% recovery of **2a**

Following **Typical Procedure II**, the reaction of **1g** (494.5 mg, 2.4 mmol), **2a** (182.3 mg, 1.0 mmol), K_2CO_3 (41.9 mg, 0.3 mmol), and [Ru(*p*-cymene)Cl_2]$_2$ (12.4 mg, 0.02 mmol) in 2.5 mL of EtOH afforded **3ga** (247.5 mg, 75%) as a solid (eluent: petroleum ether/ethyl acetate = 10/1, 1200 mL): m.p. 83.9-84.3 °C (petroleum ether/DCM); 14% recovery of **2a** was determined by ^{1}H NMR analysis of the crude product using 35 μL CH_2Br_2 as the internal standard. ^{1}H NMR (300 MHz, $CDCl_3$) δ 11.93 (bs, 1 H, COOH), 7.39 (t, *J* = 8.1 Hz, 1 H, ArH), 7.28 (d, *J* = 8.1 Hz, 1 H, ArH), 7.17 (d, *J* = 8.1 Hz, 1 H, ArH), 2.38 (t, *J* = 7.1 Hz, 2 H,

CH_2), 1.75 (s, 6 H, CH_3 × 2), 1.53-1.31 (m, 4 H, CH_2 × 2), 0.92 (t, J = 7.1 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 201.9, 172.9, 146.1 (q, J = 1.4 Hz), 140.5, 130.5, 125.8, 125.5, 120.5 (q, J = 257.4 Hz), 117.6 (q, J = 1.4 Hz), 100.8, 99.2, 32.5, 29.9, 22.2, 19.8, 13.9; ^{19}F NMR (282 MHz, $CDCl_3$) δ -57.3 (s, 3 F); IR (neat) ν (cm^{-1}) 3566-2146 (COOH), 1957, 1714, 1699, 1604, 1575, 1464, 1456, 1404, 1363, 1259, 1214, 1168, 1133, 1065, 1029; MS (EI): *m/z* (%) 328 (M^+, 8.21), 271 (100); Anal. Calcd. for $C_{17}H_{19}F_3O_3$ (%): C, 62.19; H, 5.83; Found: C, 62.07; H, 5.89.

12. Synthesis of 2-(7-methyldodeca-5,6-dien-5-yl)-6-(trifluoromethoxy)benzoic acid **3gf**. (wxy-3-048)

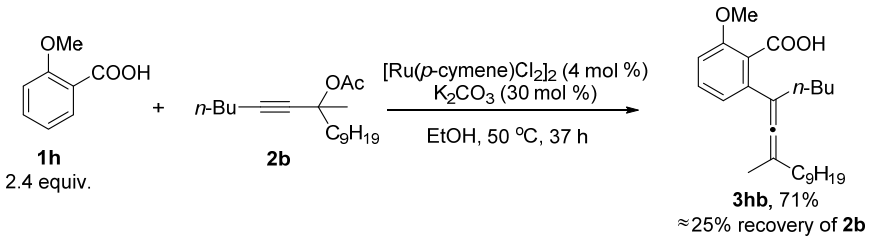


1g 2.4 equiv. **2f** **3gf**, 68% 30% recovery of **2f**

Following **Typical Procedure II**, the reaction of **1g** (494.8 mg, 2.4 mmol), **2f** (238.1 mg, 1.0 mmol), K_2CO_3 (41.7 mg, 0.3 mmol), and [Ru(*p*-cymene)Cl_2]$_2$ (24.6 mg, 0.04 mmol) in 2.5 mL of EtOH afforded **3gf** (262.1 mg, 68%) as an oil (eluent: petroleum ether/ethyl acetate = 9/1, 1000 mL); 30% recovery of **2f** was determined by ^1H NMR analysis of the crude product using 35 μL CH_2Br_2 as the internal standard. ^1H NMR (300 MHz, $CDCl_3$) δ 12.16 (bs, 1 H, COOH), 7.39 (t, J = 8.0 Hz, 1 H, ArH), 7.27 (d, J = 7.8 Hz, 1 H, ArH), 7.17 (d, J = 8.1 Hz, 1 H, ArH), 2.46-2.25 (m, 2 H, CH_2), 1.98 (t, J = 7.4 Hz, 2 H, CH_2), 1.76 (s, 3 H, CH_3), 1.55-1.30 (m, 6 H, CH_2 × 3), 1.30-1.11 (m, 4 H, CH_2 × 2), 0.91 (t, J = 7.2 Hz, 3 H, CH_3), 0.81 (t, J = 6.8 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 201.6, 172.8, 146.1 (q, J = 1.4 Hz),

140.8, 130.5, 125.8, 125.7, 120.5 (q, J = 257.2 Hz), 117.7 (q, J = 1.4 Hz), 103.3, 102.2, 33.9, 32.7, 31.4, 30.1, 27.0, 22.5, 22.2, 17.9, 13.92, 13.89; ^{19}F NMR (282 MHz, $CDCl_3$) δ -57.4; IR (neat) ν (cm^{-1}) 3550-2100 (COOH), 1953, 1714, 1700, 1604, 1575, 1467, 1404, 1259, 1216, 1171, 1133, 1064; MS (EI): *m/z* (%) 384 (M^+, 33.82), 385 (M^+ + 1, 7.98), 327 (100); HRMS Calcd for $C_{21}H_{27}O_3F_3$ (M^+): 384.1912; Found: 384.1914.

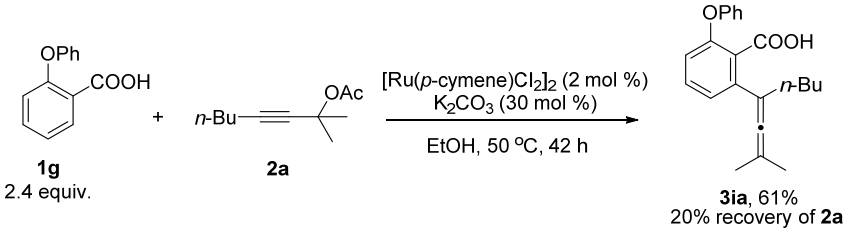
13. Synthesis of 2-methoxy-6-(7-methylhexadeca-5,6-dien-5-yl)benzoic acid **3hb**. (wxy-2-181, wxy-3-015)



Following **Typical Procedure II**, the reaction of **1h** (365.5 mg, 2.4 mmol), **2b** (294.4 mg, 1.0 mmol), K_2CO_3 (41.5 mg, 0.3 mmol), and [Ru(*p*-cymene)Cl_2]$_2$ (24.5 mg, 0.04 mmol) in 2.5 mL of EtOH afforded **3hb** (274.5 mg, 71%) as an oil (eluent: petroleum ether/ethyl acetate /HOAc = 500/50/2, 1000 mL). About 25% recovery of **2b** was determined by ^{1}H NMR analysis of the crude product using 35 μL CH_2Br_2 as the internal standard. ^{1}H NMR (300 MHz, $CDCl_3$) δ 11.98 (bs, 1 H, COOH), 7.30 (t, J = 8.1 Hz, 1 H, ArH), 6.95 (d, J = 7.5 Hz, 1 H, ArH), 6.79 (d, J = 8.4 Hz, 1 H, ArH), 3.83 (s, 3 H, OMe), 2.37 (t, J = 7.2 Hz, 2 H, CH_2), 2.08-1.89 (m, 2 H, CH_2), 1.79 (s, 3 H, CH_3), 1.51-1.31 (m, 6 H, CH_2 × 3), 1.31-1.14 (m, 12 H, CH_2 × 6), 0.95-0.80 (m, 6 H, CH_3 × 2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 201.2, 174.7, 156.4, 138.7, 130.2, 122.1, 119.5, 108.9, 102.8, 102.4, 55.8, 34.0, 32.7, 31.9, 30.2, 29.6, 29.5, 29.32, 29.27, 27.4, 22.6, 22.3, 18.1, 14.05, 13.95; IR (neat) ν (cm^{-1}) 3523-2138 (COOH),

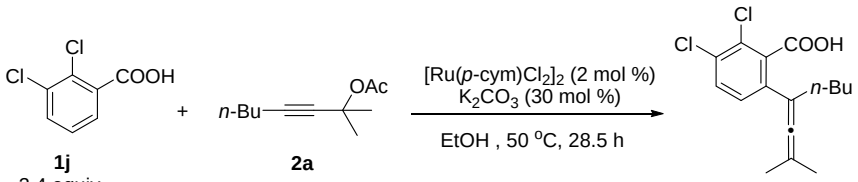
1949, 1699, 1595, 1580, 1471, 1296, 1267, 1126, 1089, 1066; MS (EI): *m/z* (%) 386 (M^+, 25.55), 329 (100); HRMS Calcd for $C_{25}H_{38}O_3$ (M^+): 386.2821; Found: 386.2823.

14. Synthesis of 2-(2-methylocta-2,3-dien-4-yl)-6-phenoxybenzoic acid **3ia**. (wxy-3-011)



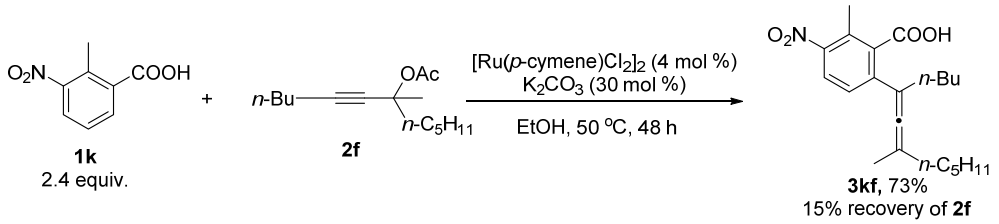
Following **Typical Procedure II**, the reaction of **1i** (514.2 mg, 2.4 mmol), **2a** (182.2 mg, 1.0 mmol), K_2CO_3 (42.0 mg, 0.3 mmol), and [Ru(*p*-cymene)Cl_2]$_2$ (12.5 mg, 0.02 mmol) in 2.5 mL of EtOH afforded **3ia** (203.8 mg, 61%) as a solid (eluent: petroleum ether/ethyl acetate = 10/1, 1000 mL): m.p. 126.8-127.1 ^oC (petroleum ether/DCM). 20% recovery of **2a** was determined by ^1H NMR analysis of the crude product using 35 μL CH_2Br_2 as the internal standard. ^1H NMR (300 MHz, $CDCl_3$) δ 12.29 (bs, 1 H, COOH), 7.35-7.25 (m, 2 H, ArH), 7.21 (t, J = 8.0 Hz, 1 H, ArH), 7.13-6.95 (m, 4 H, ArH), 6.66 (d, J = 8.1 Hz, 1 H, ArH), 2.36 (t, J = 7.1 Hz, 2 H, CH_2), 1.60 (s, 6 H, CH_3 × 2), 1.50-1.25 (m, 4 H, CH_2 × 2), 0.88 (t, J = 7.1 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 202.1, 174.5, 156.9, 154.7, 138.6, 130.2, 129.7, 124.6, 123.7, 121.3, 119.6, 115.8, 100.6, 99.5, 32.1, 30.0, 22.2, 19.7, 14.0; IR (neat) ν (cm^{-1}) 3540-2258 (COOH), 1953, 1703, 1595, 1575, 1490, 1456, 1294, 1257, 1211, 1162, 1128, 1066; MS (EI): *m/z* (%) 336 (M^+, 1.34); 279 (100); Anal. Calcd. for $C_{22}H_{24}O_3$ (%): C, 78.54; H, 7.19; Found: C, 78.55; H, 7.10.

15. Synthesis of 2,3-dichloro-6-(2-methylocta-2,3-dien-4-yl)benzoic acid **3ja**. (wxy-2-182)



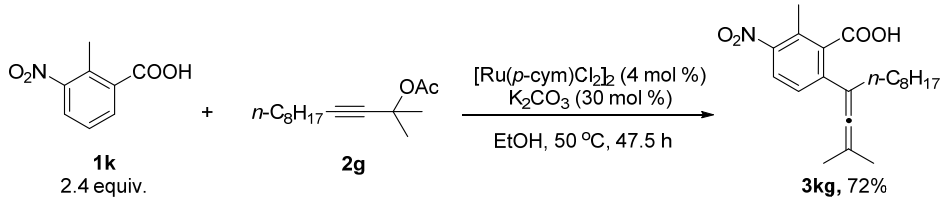
Following **Typical Procedure II**, the reaction of **1j** (458.4 mg, 2.4 mmol), **2a** (182.5 mg, 1.0 mmol), K_2CO_3 (41.7 mg, 0.3 mmol), and [Ru(*p*-cymene)Cl_2]$_2$ (12.2 mg, 0.02 mmol) in 2.5 mL of EtOH afforded **3ja** (203.5 mg, 54%) as a solid (first round eluent: petroleum ether/ethyl acetate/HOAc = 500/30/4, 1500 mL; The impure part was further purified in second round, eluent: petroleum ether/ethyl acetate/HOAc = 500/40/4, 1500 mL): m.p. 103.1-103.9 ^{o}C, (petroleum ether/DCM). 29% recovery of **2a** was determined by ^{1}H NMR analysis of the crude product using 35 μL CH_2Br_2 as the internal standard. ^{1}H NMR (300 MHz, $CDCl_3$) δ 11.58 (bs, 1 H, COOH), 7.46 (d, J = 8.4 Hz, 1 H, ArH), 7.20 (d, J = 8.7 Hz, 1 H, ArH), 2.33 (t, J = 7.2 Hz, 2 H, CH_2), 1.75 (s, 6 H, CH_3 × 2), 1.50-1.30 (m, 4 H, CH_2 × 2), 0.91 (t, J = 7.1 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 201.6, 172.9, 137.9, 133.6, 131.1, 130.9, 129.2, 126.7, 100.4, 99.3, 32.7, 29.8, 22.1, 20.1, 13.9; IR (KBr) ν (cm^{-1}) 3351-2129 (COOH), 1955, 1704, 1549, 1458, 1412, 1375, 1362, 1276, 1251, 1180, 1059, 1025; MS (EI): *m/z* (%) 316 [M^+(^{37}Cl^{37}Cl), 0.43], 314 [M^+(^{37}Cl^{35}Cl), 1.61], 312 [M^+(^{35}Cl^{35}Cl), 1.94], 255 (100); Anal. Calcd. for $C_{16}H_{18}Cl_2O_2$ (%): C, 61.36; H, 5.79; Found: C, 61.26; H, 5.81.

16. Synthesis of 2-methyl-6-(7-methyldodeca-5,6-dien-5-yl)-3-nitrobenzoic acid **3kf**. (wxy-3-152)



Following **Typical Procedure II**, the reaction of **1k** (434.9 mg, 2.4 mmol), **2a** (238.0 mg, 1.0 mmol), K_2CO_3 (41.9 mg, 0.3 mmol), and [Ru(*p*-cymene)Cl_2]$_2$ (24.5 mg, 0.04 mmol) in 2.5 mL of EtOH afforded **3kf** (260.1 mg, 73%) as an oil (eluent: petroleum ether/ethyl acetate = 9/1, 1500 mL); 15% recovery of **2a** was determined by ^{1}H NMR analysis of the crude product using 35 μL CH_2Br_2 as the internal standard. ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.89 (bs, 1 H, COOH), 7.91 (d, *J* = 8.4 Hz, 1 H, ArH), 7.34 (d, *J* = 8.8 Hz, 1 H, ArH), 2.57 (s, 3 H, CH_3), 2.46-2.31 (m, 2 H, CH_2), 2.00 (t, *J* = 8.0 Hz, 2 H, CH_2), 1.77 (s, 3 H, CH_3), 1.53-1.33 (m, 6 H, CH_2 × 3), 1.33-1.18 (m, 4 H, CH_2 × 2), 0.92 (t, *J* = 7.2 Hz, 3 H, CH_3), 0.83 (t, *J* = 6.8 Hz, 3 H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.3, 174.4, 148.3, 143.0, 134.8, 130.0, 126.2, 125.3, 103.2, 102.5, 33.8, 32.9, 31.3, 30.0, 26.9, 22.4, 22.1, 18.2, 16.5, 13.9, 13.8; IR (neat) *v* (cm^{-1}) 3578-2138 (COOH), 1951, 1704, 1592, 1580, 1525, 1463, 1347, 1281, 1131; MS (EI): *m/z* (%) 360 (M^+ + 1, 57.36), 359 (M^+, 82.16), 41 (100); HRMS Calcd for $C_{21}H_{30}NO_4$ $(M+H)^+$: 360.2175; Found: 360.2169.

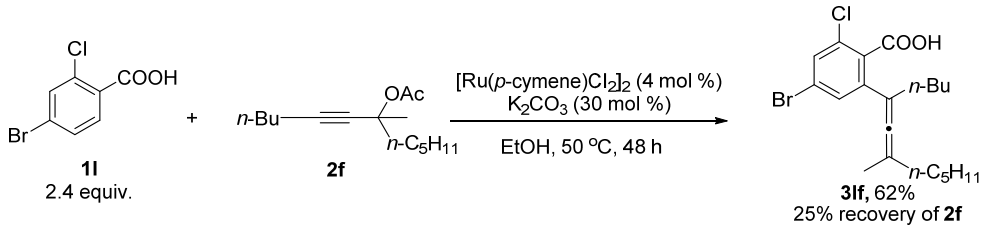
17. Synthesis of 2-methyl-6-(2-methyldodeca-2,3-dien-4-yl)-3-nitrobenzoic acid **3kg**. (wxy-3-023)



Following **Typical Procedure II**, the reaction of **1k** (434.7 mg, 2.4 mmol), **2g** (238.9 mg,

1.0 mmol), K_2CO_3 (41.5 mg, 0.3 mmol), and [Ru(*p*-cymene)Cl_2]$_2$ (24.5 mg, 0.04 mmol) in 2.5 mL of EtOH afforded **3kg** (258.8 mg, 72%) as an oil (eluent: petroleum ether/ethyl acetate/AcOH = 450/50/2, 1300 mL); ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.78 (bs, 1 H, COOH), 7.91 (d, *J* = 8.7 Hz, 1 H, ArH), 7.34 (d, *J* = 8.7 Hz, 1 H, ArH), 2.55 (s, 3 H, CH_3), 2.37 (t, *J* = 7.1 Hz, 2 H, CH_2), 1.75 (s, 6 H, CH_3 × 2), 1.52-1.18 (m, 12 H, CH_2 × 6), 0.87 (t, *J* = 6.6 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 201.7, 174.2, 148.3, 142.9, 134.9, 130.1, 126.1, 125.3, 101.4, 98.9, 33.1, 31.8, 29.4, 29.3, 29.1, 27.7, 22.6, 20.0, 16.5, 14.0; IR (neat) *v* (cm^{-1}) 3712-2116 (COOH), 1955, 1704, 1700, 1593, 1581, 1525, 1520, 1348, 1279, 1130; MS (EI): *m/z* (%) 360 (M^+ + 1, 18.98), 359 (M^+, 73.54), 43 (100); HRMS Calcd for $C_{21}H_{29}NO_4$ (M^+): 359.2097; Found: 359.2098.

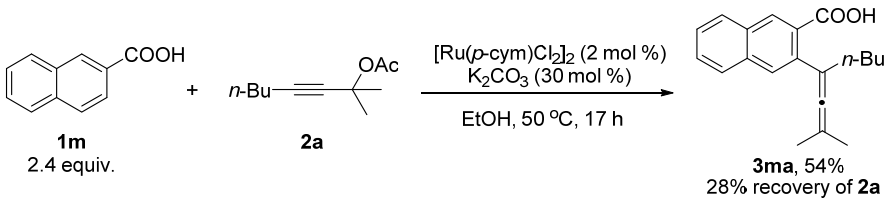
18. Synthesis of 4-bromo-2-chloro-6-(7-methyldodeca-5,6-dien-5-yl)benzoic acid **3lf**. (wxy-3-153)



Following **Typical Procedure II**, the reaction of **1l** (564.0 mg, 2.4 mmol), **2f** (240.0 mg, 1.0 mmol), K_2CO_3 (41.8 mg, 0.3 mmol), and [Ru(*p*-cymene)Cl_2]$_2$ (24.6 mg, 0.04 mmol) in 5.0 mL of EtOH afforded **3lf** (256.9 mg, 62%) as an oil (eluent: petroleum ether/ethyl acetate = 10/1, 1500 mL); 25% recovery of **2f** was determined by ^{1}H NMR analysis of the crude product using 35 μL CH_2Br_2 as the internal standard. ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.98 (bs, 1 H, COOH), 7.45 (d, *J* = 1.6 Hz, 1 H, ArH), 7.39 (d, *J* = 1.6 Hz, 1 H, ArH), 2.40-2.25 (m, 2

H, CH_2), 2.06-1.90 (m, 2 H, CH_2), 1.75 (s, 3 H, CH_3), 1.50-1.30 (m, 6 H, CH_2 × 3), 1.30-1.17 (m, 4 H, CH_2 × 2), 0.91 (t, J = 7.0 Hz, 3 H, CH_3), 0.83 (t, J = 7.0 Hz, 3 H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.6, 173.2, 141.5, 131.8, 130.7, 129.8, 128.9, 123.7, 103.9, 101.6, 33.9, 32.6, 31.4, 30.0, 26.9, 22.5, 22.2, 18.2, 14.0, 13.9; IR (neat) ν (cm^{-1}) 3561-2194 (COOH), 1952, 1708, 1573, 1548, 1456, 1397, 1367, 1285, 1186, 1133; MS (EI): *m/z* (%) 416 [M^+($^{81}Br^{37}Cl$), 6.24], 414 [M^+($^{81}Br^{35}Cl$) and/or M^+($^{79}Br^{37}Cl$), 24.07], 412 [M^+($^{79}Br^{35}Cl$), 20.38], 41 (100); HRMS Calcd for $C_{20}H_{27}{}^{79}Br^{35}ClO_2$ $(M+H)^+$: 413.0883; Found: 413.0877.

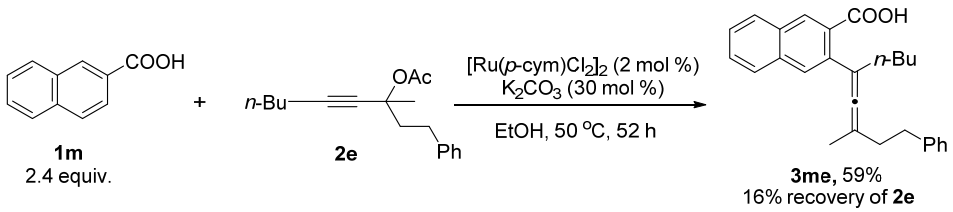
19. Synthesis of 3-(2-methylocta-2,3-dien-4-yl)-2-naphthoic acid **3ma**. (wxy-2-134, wxy-2-139)



Following **Typical Procedure II**, the reaction of **1m** (413.2 mg, 2.4 mmol), K_2CO_3 (41.5 mg, 0.3 mmol), **2a** (182.3 mg, 1.0 mmol), and [Ru(*p*-cymene)Cl_2]$_2$ (12.5 mg, 0.02 mmol) in 5.0 mL of EtOH afforded **3ma** (157.8 mg, 54%) (eluent: petroleum ether/ethyl acetate/HOAc = 500/40/4, 1500 mL): oil; 28% recovery of **2a** was determined by ^{1}H NMR analysis of the crude product using 35 μL CH_2Br_2 as the internal standard. ^{1}H NMR (300 MHz, $CDCl_3$) δ 11.33 (bs, 1 H, COOH), 8.39 (s, 1 H, ArH), 7.88 (d, J = 8.1 Hz, 1 H, ArH), 7.82 (d, J = 8.1 Hz, 1 H, ArH), 7.76 (s, 1 H, ArH), 7.55 (td, J_1 = 7.4 Hz, J_2 = 1.3 Hz, 1 H, ArH), 7.48 (td, J_1 = 7.5 Hz, J_2 = 1.3 Hz, 1 H, ArH), 2.45 (t, J = 7.2 Hz, 2 H, CH_2), 1.76 (s, 6 H, 2 × CH_3), 1.60-1.39 (m, 4 H, 2 × CH_2), 0.94 (t, J = 7.2 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 201.5, 174.7, 137.2, 134.8, 131.5, 131.1, 128.6, 128.4, 128.2, 127.9, 127.5, 126.3, 103.4, 97.1, 33.4, 30.2,

22.3, 20.2, 14.1; IR (neat) *v* (cm^{-1}) 3617-2090 (COOH), 1953, 1699, 1695, 1682, 1629, 1590, 1464, 1447, 1404, 1361, 1286, 1214, 1138, 1082; MS (EI): m/z (%) = 294 (M^+, 1.41), 237 (100); HRMS Calcd for $C_{20}H_{22}O_2$ (M^+): 294.1620; Found: 294.1618.

20. Synthesis of 3-(6-methyl-8-phenylocta-4,5-dien-4-yl)-2-naphthoic acid **3me**. (wxy-1-197)



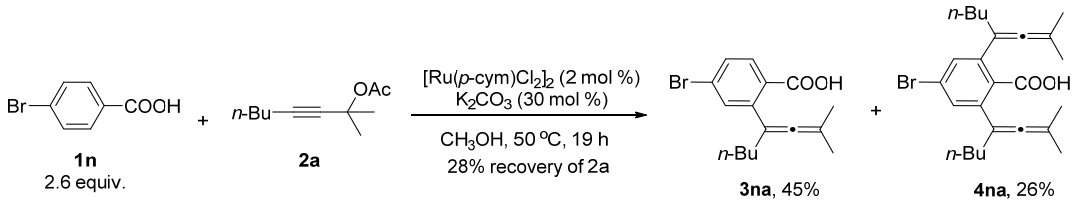
1m
2.4 equiv.

2e

3me, 59%
16% recovery of **2e**

Following **Typical Procedure II**, the reaction of **1m** (413.6 mg, 2.4 mmol), **2e** (272.0 mg, 1.0 mmol), K_2CO_3 (41.6 mg, 0.3 mmol), and [Ru(*p*-cymene)Cl_2]$_2$ (12.5 mg, 0.02 mmol) in 5.0 mL of EtOH afforded **3me** (228.3 mg, 59%) as an oil (eluent: petroleum ether/ethyl acetate/AcOH = 500/40/4, 1200 mL); 16% recovery of **2e** was determined by ^{1}H NMR analysis of the crude product using 35 μL CH_2Br_2 as the internal standard. ^{1}H NMR (300 MHz, $CDCl_3$) δ 11.54 (bs, 1 H, COOH), 8.39 (s, 1 H, ArH), 7.84 (d, *J* = 8.1 Hz, 1 H, ArH), 7.79 (d, *J* = 8.1 Hz, 1 H, ArH), 7.71 (s, 1 H, ArH), 7.53 (t, *J* = 7.4 Hz, 1 H, ArH), 7.45 (t, *J* = 7.4 Hz, 1 H, ArH), 7.24-7.09 (m, 4 H, ArH), 7.09-6.97 (m, 1 H, ArH), 2.88-2.68 (m, 2 H, CH_2), 2.48-2.25 (m, 4 H, CH_2× 2), 1.82 (s, 3 H, CH_3), 1.55-1.31 (m, 4 H, CH_2 × 2), 0.92 (t, *J* = 6.9 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 200.8, 174.4, 142.2, 137.3, 134.9, 131.8, 131.2, 128.7, 128.28, 128.25, 128.1, 127.9, 127.5, 126.3, 125.6, 105.8, 100.9, 35.9, 34.0, 33.8, 30.3, 22.4, 18.8, 14.1; IR (neat) *v* (cm^{-1}) 3300-2100 (COOH), 1951, 1703, 1699, 1695, 1683, 1629, 1496, 1454, 1404, 1287, 1214, 1138; MS (EI): *m/z* (%) 384 (M^+, 2.34), 131 (100); HRMS Calcd for $C_{27}H_{28}O_2$ (M^+): 384.2089; Found: 384.2087.

21. Synthesis of 4-bromo-2-(2-methylocta-2,3-dien-4-yl)benzoic acid **3na** 4-bromo-2,6-bis(2-methylocta-2,3-dien-4-yl)benzoic acid **4na**. (wxy-2-135)



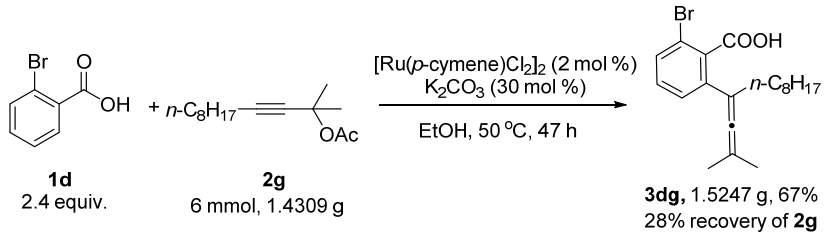
Following **Typical Procedure II**, the reaction of **1n** (522.5 mg, 2.6 mmol), **2a** (182.3 mg, 1.0 mmol), K_2CO_3 (41.9 mg, 0.3 mmol), and [Ru(*p*-cymene)Cl_2]$_2$ (12.4 mg, 0.02 mmol) in 2.5 mL of MeOH afforded **3na** (144.7 mg, 45%) and **4na** (57.0 mg, 26%) (eluent: petroleum ether/ethyl acetate/HOAc = 500/40/4, 2000 mL). 28% recovery of **2a** was determined by ^{1}H NMR analysis of the crude product using 35 μL CH_2Br_2 as the internal standard.

3na: solid; m.p. 69.9-71.9 ^{o}C (petroleum ether/DCM); ^{1}H NMR (300 MHz, $CDCl_3$) δ 11.21 (bs, 1 H, COOH), 7.69 (d, J = 8.1 Hz, 1 H, ArH), 7.47 (s, 1 H, ArH), 7.42 (dd, J_1 = 8.1 Hz, J_2 = 1.8 Hz, 1 H, ArH), 2.29 (t, J = 7.1 Hz, 2 H, CH_2), 1.71 (s, 6 H, CH_3 × 2), 1.50-1.30 (m, 4 H, CH_2 × 2), 0.91 (t, J = 7.1 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 200.9, 173.6, 143.8, 132.6, 131.8, 129.5, 128.2, 126.6, 102.7, 97.7, 33.2, 30.1, 22.2, 20.0, 14.0; IR (neat) ν (cm^{-1}) 3583-2181 (COOH), 1957, 1699, 1583, 1557, 1416, 1362, 1296, 1141, 1100; MS (EI): *m/z* (%) 324 [M^+(^{81}Br), 0.75], 322 [M^+(^{79}Br), 0.83], 265 (100); Anal. Calcd. for $C_{16}H_{19}BrO_2$ (%): C, 59.45; H, 5.93; Found: C, 59.41; H, 5.91.

4na: solid; m.p. 107.9-109.7 ^{o}C (petroleum ether/DCM); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.27 (s, 2 H, ArH), 2.25 (t, J = 7.1 Hz, 4 H, CH_2 × 2), 1.70 (s, 12 H, CH_3 × 4), 1.50-1.28 (m, 8 H, CH_2 × 4), 0.90 (t, J = 7.1 Hz, 6 H, CH_3 × 2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 200.8, 173.8, 140.5, 130.5, 129.6, 123.1, 101.4, 97.6, 33.7, 29.9, 22.2, 20.4, 14.0; IR (neat) ν (cm^{-1})

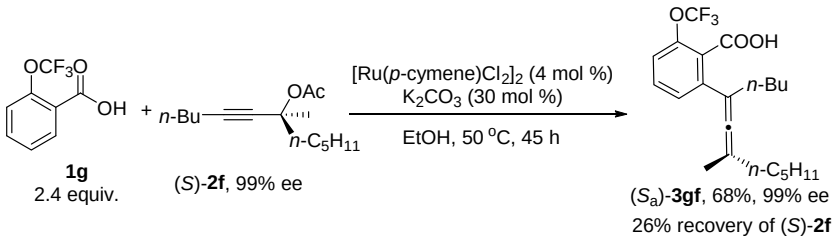
3484-2198 (COOH), 1963, 1704, 1564, 1444, 1362, 1282, 1130; MS (EI): *m/z* (%) 446 [M^+(^{81}Br), 55.06], 444 [M^+(^{79}Br), 49.23], 41 (100); Anal. Calcd. for $C_{25}H_{33}BrO_2$ (%): C, 67.41; H, 7.47; Found: C, 67.06; H, 7.39.

22. Synthesis of 2-bromo-6-(2-methyldodeca-2,3-dien-4-yl)benzoic acid **3dg**. (wxy-3-061)



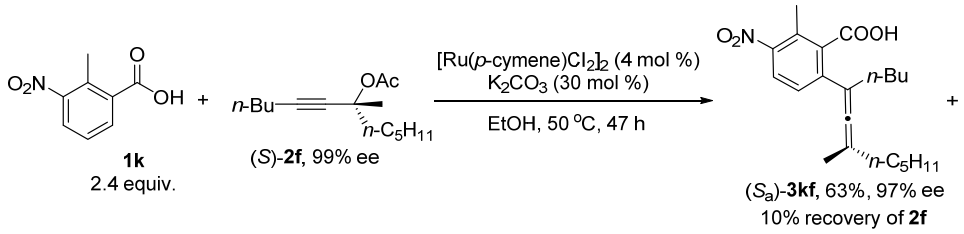
Following **Typical Procedure II**, the reaction of **1d** (2.8936 g, 14.4 mmol), **2g** (1.4309 g, 6.0 mmol), K_2CO_3 (248.9 mg, 1.8 mmol), and [Ru(*p*-cymene)Cl_2]$_2$ (73.5 mg, 0.12 mmol) in 15 mL of EtOH afforded **3dg** (1.5247 g, 67%) as an oil (first round eluent: petroleum ether/ethyl acetate/HOAc = 500/50/4, 2000 mL; The impure part was further purified in second round, eluent: petroleum ether/ethyl acetate/HOAc = 500/50/4, 1500 mL). 28% recovery of **2g** was determined by ^{1}H NMR analysis of the crude product using 210 μL CH_2Br_2 as the internal standard. ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.57 (bs, 1 H, COOH), 7.46 (dd, J_1 = 7.7 Hz, J_2 = 1.4 Hz, 1 H, ArH), 7.29 (dd, J_1 = 7.8 Hz, J_2 = 1.5 Hz, 1 H, ArH), 7.23 (t, J = 7.8 Hz, 1 H, ArH), 2.33 (t, J = 7.1 Hz, 2 H, CH_2), 1.75 (s, 6 H, CH_3 × 2), 1.51-1.15 (m, 12 H, CH_2 × 6), 0.87 (t, J = 6.6 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 201.4, 173.7, 140.1, 133.9, 130.6, 130.5, 126.5, 119.3, 101.3, 98.7, 33.3, 31.9, 29.5, 29.3, 29.2, 27.8, 22.6, 20.3, 14.1; IR (neat) *v* (cm^{-1}) 3557-2159 (COOH), 1958, 1706, 1587, 1557, 1443, 1385, 1361, 1287, 1188, 1152, 1126, 1056; MS (EI): *m/z* (%) 380 [M(^{81}Br)$^+$, 46.19], 378 [M(^{79}Br)$^+$, 49.29], 43 (100); HRMS Calcd for $C_{20}H_{27}O_2{}^{79}Br$ (M^+): 378.1194; Found: 378.1193.

23. Synthesis of (S_a)-2-(7-methyldodeca-5,6-dien-5-yl)-6-(trifluoromethoxy)benzoic acid (S_a)-**3gf**. (wxy-3-156)



Following **Typical Procedure II**, the reaction of **1g** (148.7 mg, 0.72 mmol), (*S*)-**2f** (71.1 mg, 0.3 mmol, 99% ee), K_2CO_3 (12.3 mg, 0.09 mmol), and [Ru(*p*-cymene)Cl_2]$_2$ (7.5 mg, 0.012 mmol) in 0.8 mL of EtOH afforded (S_a)-**3gf** (78.1 mg, 68%) as an oil (eluent: petroleum ether/ethyl acetate = 10/1, 1000 mL): 99% ee (HPLC conditions: Chiralcel OJ-3 column, CO_2/*i*-PrOH = 98/2, 1.0 mL/min, λ = 254 nm, t_R(major) = 1.34 min, t_R(minor) = 1.55 min); $[\alpha]_D^{20}$ = + 126.3 (*c* = 1.225, $CHCl_3$); 26% recovery of (*S*)-**2f** was determined by ^{1}H NMR analysis of the crude product using 10.5 μL CH_2Br_2 as the internal standard. ^{1}H NMR (400 MHz, $CDCl_3$) δ 11.33 (bs, 1 H, COOH), 7.40 (t, *J* = 8.0 Hz, 1 H, ArH), 7.27 (d, *J* = 7.6 Hz, 1 H, ArH), 7.17 (d, *J* = 8.4 Hz, 1 H, ArH), 2.45-2.28 (m, 2 H, CH_2), 2.05-1.88 (m, 2 H, CH_2), 1.75 (s, 3 H, CH_3), 1.52-1.30 (m, 6 H, CH_2 × 3), 1.30-1.15 (m, 4 H, CH_2 × 2), 0.91 (t, *J* = 7.2 Hz, 3 H, CH_3), 0.81 (t, *J* = 6.8 Hz, 3 H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.5, 172.6, 146.1, 140.8, 130.5, 125.7, 120.4 (q, *J* = 257.3 Hz), 117.69, 117.65, 103.3, 102.2, 33.9, 32.7, 31.4, 30.1, 26.9, 22.5, 22.2, 17.9, 13.95, 13.92; ^{19}F NMR (376 MHz, $CDCl_3$) δ -57.4; IR (neat) *v* (cm^{-1}) 3467-2151 (COOH), 1952, 1709, 1604, 1575, 1467, 1403, 1258, 1215, 1171, 1064; MS (EI): *m/z* (%) 384 (M^+, 8.24), 323 (100); HRMS Calcd for $C_{21}H_{28}F_3O_3$ $(M+H)^+$: 385.1991; Found: 385.1985.

24. Synthesis of (S_a)-2-methyl-6-(7-methyldodeca-5,6-dien-5-yl)-3-nitrobenzoic acid (S_a)-**3kf**. (wxy-3-157)



1k
2.4 equiv.

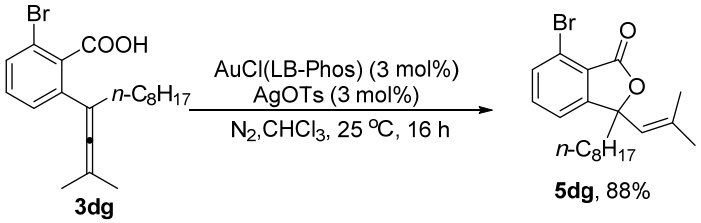
(*S*)-**2f**, 99% ee

(S_a)-**3kf**, 63%, 97% ee
10% recovery of **2f**

Following **Typical Procedure II**, the reaction of **1k** (130.5 mg, 0.72 mmol), (*S*)-**2f** (71.0 mg, 0.3 mmol, 99% ee), K_2CO_3 (12.5 mg, 0.09 mmol), and [Ru(*p*-cymene)Cl_2]$_2$ (7.3 mg, 0.012 mmol) in 0.8 mL of EtOH afforded (S_a)-**3kf** (67.4 mg, 63%) as an oil (eluent: petroleum ether/ethyl acetate = 9/1, 1500 mL); 97% ee (HPLC conditions: Chiralcel OZ-H column, *n*-hexane/*i*-PrOH = 100/1, 1.0 mL/min, λ = 214 nm, t_R(major) = 38.5 min, t_R(minor) = 27.7 min); $[\alpha]_D^{20}$ = + 69.5 (*c* = 0.85, $CHCl_3$); 10% recovery of (*S*)-**2f** was determined by ^{1}H NMR analysis of the crude product using 10.5 μL CH_2Br_2 as the internal standard. ^{1}H NMR (300 MHz, $CDCl_3$) δ 11.53 (bs, 1 H, COOH), 7.91 (t, *J* = 8.4 Hz, 1 H, ArH), 7.33 (d, *J* = 8.7 Hz, 1 H, ArH), 2.56 (s, 3 H, CH_3), 2.46-2.28 (m, 2 H, CH_2), 1.99 (t, *J* = 7.2 Hz, 2 H, CH_2), 1.76 (s, 3 H, CH_3), 1.53-1.32 (m, 6 H, CH_2 × 3), 1.32-1.12 (m, 4 H, CH_2 × 2), 0.92 (t, *J* = 7.1 Hz, 3 H, CH_3), 0.83 (t, *J* = 6.9 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 201.3, 174.3, 148.4, 143.1, 134.8, 130.1, 126.3, 125.3, 103.2, 102.5, 33.9, 33.0, 31.4, 30.0, 26.9, 22.4, 22.2, 18.2, 16.5, 13.94, 13.88; IR (neat) *v* (cm^{-1}) 3561-2129 (COOH), 1953, 1742, 1704, 1592, 1581, 1525, 1465, 1347, 1280, 1131; MS (EI): *m/z* (%) 359 (M^+, 4.60), 302 (100); HRMS Calcd for $C_{21}H_{30}NO_4$ $(M + H)^+$: 360.2175; Found: 360.2171.

Synthetic applications

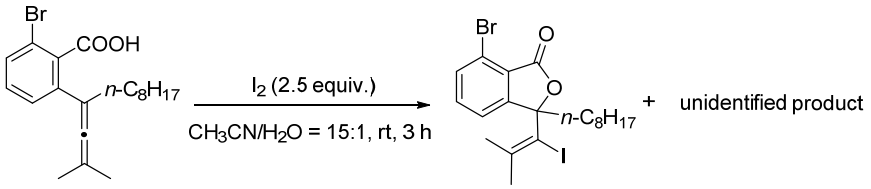
25. Synthesis of 7-bromo-3-(2-methylprop-1-en-1-yl)-3-octylisobenzofuran-1(3*H*)-one **5dg**. (fjj-1-015)



To a dry Schlenk tube were added AgOTs (4.3 mg, 0.015 mmol, weighed in a glove box, 98%), AuCl(LB-Phos) (9.0 mg, 0.015 mmol), and $CHCl_3$ (1.5 mL) under nitrogen atmosphere sequentially. After stirring for 15 min at 25 ^oC, **3dg** (190.0 mg, 0.5 mmol) and $CHCl_3$ (1 mL) were added. After being continuously stirred at 25 ^oC for 16 h, the reaction was complete as monitored by TLC. After filtration through a short column of silica gel (eluent: DCM, 10 mL × 3) and evaporation, the crude mixture was purified by column chromatography on silica gel afforded **5dg** (167.3 mg, 88%) (eluent: petroleum ether /ethyl acetate = 200/1, 1500 mL) as an oil: ^1H NMR (300MHz, $CDCl_3$) δ 7.64 (d, *J* = 7.8 Hz, 1 H, ArH), 7.50 (t, *J* = 7.7 Hz, 1 H, ArH), 7.32 (d, *J* = 7.8 Hz, 1 H, ArH), 5.42 (s, 1 H, =CH), 2.18-2.03 (m, 1 H, one proton from CH_2), 1.92-1.78 (m, 1 H, one proton from CH_2), 1.74 (s, 3 H, CH_3), 1.60 (s, 3 H, CH_3), 1.40-1.07 (m, 11 H, CH_2 × 5 and one proton from CH_2), 1.04-0.89 (m, 1 H, one proton from CH_2), 0.85 (t, *J* = 6.6 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.7, 156.4, 139.7, 135.1, 133.4, 124.2, 122.5, 120.6, 120.5, 87.0, 41.2, 31.8, 29.4, 29.3, 29.1, 27.4, 23.1, 22.6, 19.1, 14.1; IR (neat) ν (cm^{-1}) 3075, 2926, 2855, 1770, 1668, 1597, 1583, 1462, 1376, 1322, 1235, 1129, 1091, 1045; MS (EI): *m/z* (%) 380 [$M(^{81}Br)^+$, 0.73], 378 [$M(^{79}Br)^+$, 0.62], 265 (100). Anal. Calcd. for $C_{20}H_{27}BrO_2$ (%): C, 63.33; H, 7.17;

Found: C, 63.41; H, 7.22.

26. Iodolactonization reaction of **3dg** with iodine to afford **6dg.** (wxy-3-066)



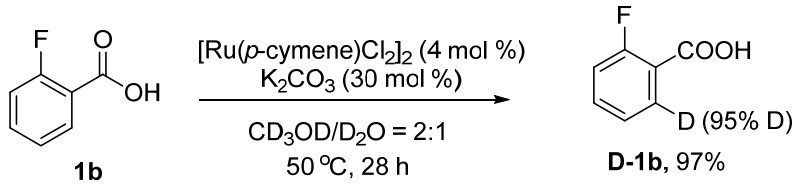
3dg **6dg**, 71%

To a dried Schlenk tube were added **3dg** (189.7 mg, 0.5 mmol), CH_3CN (2.5 mL), I_2 (317.0 mg, 1.25 mmol), and H_2O (165 μL) sequentially at rt. After being stirred for 3 h at rt, the reaction was complete as monitored by TLC. A saturated aqueous solution of $Na_2S_2O_3$ (3 mL) and 3 mL of ethyl acetate were added. The organic phase was separated and the aqueous phase was extracted with ethyl acetate (2×5 mL). The combined organic layer was evaporated and purification by flash column chromatography on silica gel [(eluent: petroleum/ethyl acetate = 60/1 (500 mL) to petrpleum/ethyl acetate = 50/1 (200 mL), then petrpleum/ethyl acetate = 10/1 (300 mL)] afforded **6dg** (179.2 mg, 71%, 98% purity) and an unidentified product (18.3 mg).

6dg: oil; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.92 (d, *J* = 7.8 Hz, 1 H, ArH), 7.68 (d, *J* = 7.8 Hz, 1 H, ArH), 7.51 (t, *J* = 7.8 Hz, 1 H, ArH), 2.70-2.54 (m, 1 H, one proton from CH_2), 2.10 (s, 3 H, CH_3), 2.05 (s, 3 H, CH_3), 2.15-1.93 (m, 1 H, one proton from CH_2), 1.49-1.00 (m, 12 H, CH_2 × 6), 0.86 (t, *J* = 6.6 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.7, 154.8, 143.2, 134.4, 133.9, 123.7, 123.4, 120.2, 97.9, 89.5, 42.4, 37.0, 31.6, 29.2, 29.1, 29.0, 23.5, 22.8, 22.5, 14.0; IR (neat) ν (cm^{-1}) 3080, 2955, 2926, 2854, 1777, 1770, 1595, 1581, 1460, 1430, 1377, 1367, 1321, 1234, 1181, 1130, 1096, 1046; MS (EI): *m/z* (%) 506 [M(^{81}Br)$^+$, 1.91], 504 [M(^{79}Br)$^+$, 1.50], 345 (100); HRMS Calcd for $C_{20}H_{26}O_2BrI$ (M^+): 504.0161; Found: 504.0163.

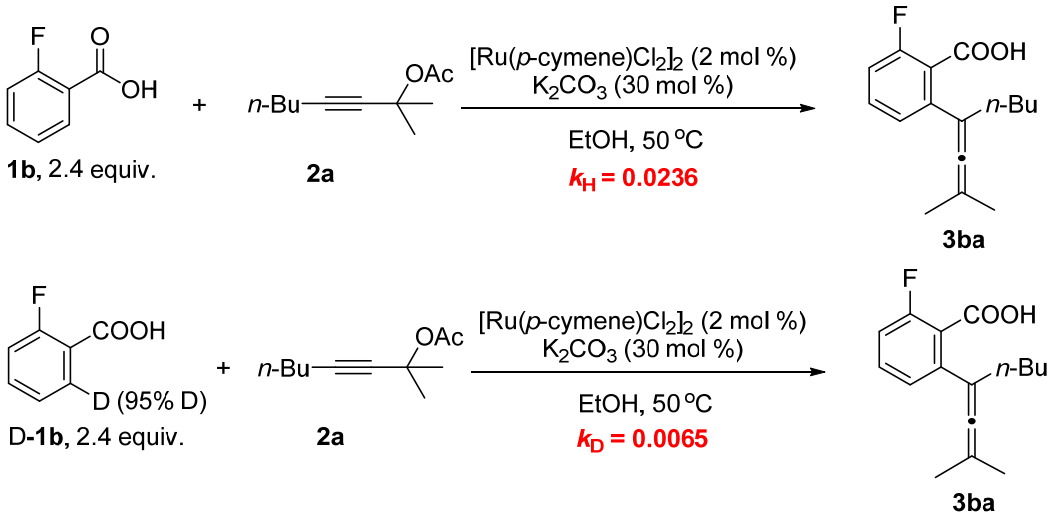
Mechanism studies

(a) H/D exchange experiment. (wxy-4-090)

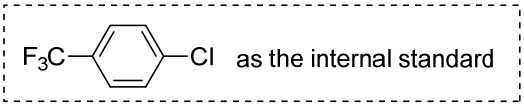


To a dried Schlenk tube were sequentially added 2-fluorobenzoic acid **1b** (420.5 mg, 3.0 mmol), [Ru(*p*-cymene)Cl_2]$_2$ (73.7 mg, 0.12 mmol), and K_2CO_3 (124.6 mg, 0.9 mmol) in open air atmosphere. After being evacuated and backfilled with nitrogen three times, 1.5 mL of CD_3OD and 0.75 mL of D_2O was added. Then, the reaction tube was put into an oil bath preheated to 50 ^oC. After 28 h, 10 mL of HCl (2 M) was added, and extracted with ethyl acetate (20 mL × 2). After concentration in vacuo, the crude residual was directly purified by chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 3/1, 300 mL) to afford **D-1b** as a white solid (410.6 mg, 97%, 95% deuterium): m.p. 123.5-123.7 ^oC (petroleum ether/diethyl ether); ^1H NMR (300 MHz, $CDCl_3$) δ 11.78 (bs, 1 H, ArH), 7.60 (td, J_1 = 8.0 Hz, J_2 = 5.0 Hz, 1 H, ArH), 7.34-7.14 (m, 2 H, ArH), the following signal is disscernible for **1b**: δ 8.06 (td, J_1 = 7.7 Hz, J_2 = 1.8 Hz, 0.05 H, ArH); ^{13}C NMR (75 MHz, $CDCl_3$) δ 170.1, 162.6 (d, J = 260.6 Hz), 135.6 (d, J = 9.7 Hz), 132.5 (t, J = 25.2 Hz), 124.96 (d, J = 3.5 Hz), 117.3, 117.0, the following signals is disscernible for **1b**: δ 132.7, 124.03 (d, J = 4.1 Hz); ^{19}F NMR (282 MHz, $CDCl_3$) δ 108.8, the following signal is disscernible for **1b**: δ 108.7; IR (neat) ν (cm^{-1}) 3600-2047 (COOH), 1695, 1609, 1461, 1412, 1300; MS (EI): *m/z* (%) 141 (M^+, 80.37), 124 (100); HRMS Calcd for $C_7H_4DFO_2$ (M^+): 141.0336; Found: 141.0337.

(b) Kinetic isotope effect studies: (wxy-4-088A, wxy-4-091)



k_H/k_D = 3.6 is determined by NMR monitoring throughout from 2.5 h to 8 h



To a dried Schlenk tube were added **1b** (168.2 mg, 1.2 mmol), [Ru(*p*-cymene)Cl_2]$_2$ (6.1 mg, 0.01 mmol), K_2CO_3 (20.7 mg, 0.15 mmol), **2a** (92.0 mg, 0.5 mmol)/EtOH (1.25 mL), 1-chloro-4-(trifluoromethyl)benzene (22.0 μL, *d* = 1.353 g/mL, 29.8mg, 0.165 mmol) sequentially at rt. The reaction tube was put into an oil bath preheated to 50 °C. An aliquot of the resulting mixture was taken for ^{19}F NMR analysis every 30 mins.

In another dried Schlenk tube, the reaction of **D-1b** (169.3 mg, 1.2 mmol), [Ru(*p*-cymene)Cl_2]$_2$ (6.1 mg, 0.01 mmol), K_2CO_3 (20.7 mg, 0.15 mmol), **2a** (91.2 mg, 0.5 mmol), EtOH (1.25 mL) and 1-chloro-4-(trifluoromethyl)benzene (22.0 μL) was conducted at the same scale. The reaction mixture was treated with the same procedure above, an aliquot of the resulting mixture was taken for ^{19}F NMR analysis every 30 mins. After being stirred for 11 h, the reaction residual was concentrated in vacuo and directly purified by chromatography to recover **D-1b** on silica gel (eluent: petroleum ether/ethyl acetate = 20/1, 1000 mL). 14.8 mg of purified **D-1b** was obtained, the deuterium content of D-**1b** was

decreased slightly (93% deuterium). ^{1}H NMR (300 MHz, $CDCl_3$) δ 11.27 (bs, 1 H, ArH),), 7.60 (td, J_1 = 7.8 Hz, J_2 = 4.6 Hz, 1 H, ArH), 7.32-7.12 (m, 2 H, ArH), the following signal is disscernible for **1b**: δ 8.05 (t, J = 7.7 Hz, 0.07 H, ArH); ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.8, 162.7 (d, J = 260.6 Hz), 135.7 (d, J = 9.7 Hz), 132.5 (t, J = 24.8 Hz), 124.0 (d, J = 4.1 Hz), 117.3, 117.1, the following signals is disscernible for **1b**: δ 132.8, 124.1 (d, J = 4.1 Hz); ^{19}F NMR (282 MHz, $CDCl_3$) δ 108.73, the following signal is disscernible for **1b**: δ 108.67.

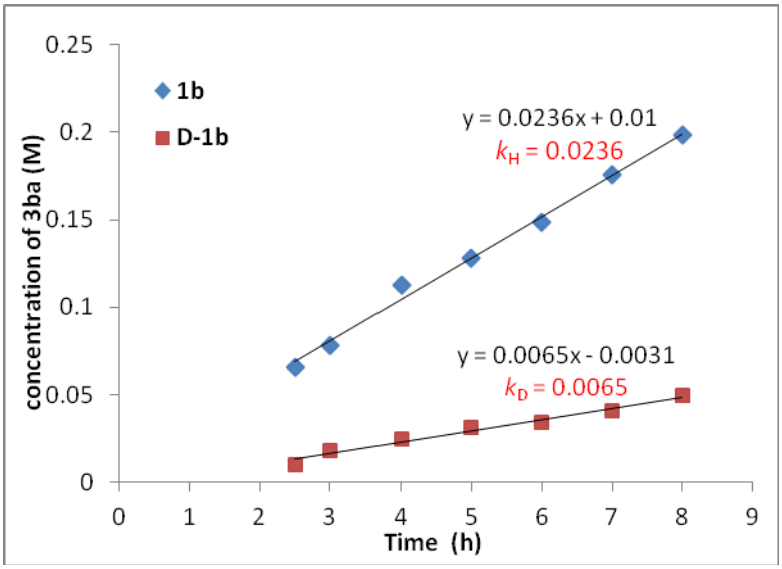
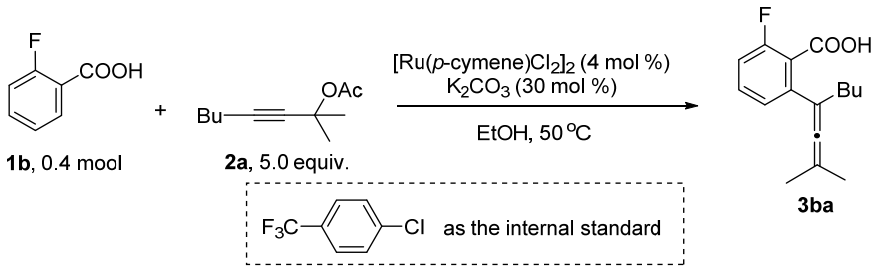


Figure S1. Plot of the concentrations of **3ba** over time.

The NMR yield and concentration of **3ba** over time are listed blew:

time (h)	**1b was** used as substrate		[D]-**1b was** used as substrate	
	NMR yield of **3ba** (%)	[**3ba**] (M)	NMR yield of **3ba** (%)	[**3ba**] (M)
2.5	16.5	0.066	2.6	0.0104
3	19.6	0.0784	4.5	0.018
4	28.2	0.1128	6.1	0.0244
5	32.1	0.1284	7.8	0.0312
6	37.2	0.1488	8.5	0.034
7	43.9	0.1756	10.3	0.0412
8	49.6	0.1984	12.4	0.0496

(c) Determination of the order for 2-fluorobenzoic acid **1b.** (wxy-3-182)



To a dried Schlenk tube were added 2-fluorobenzoic acid **1b** (56.1 mg, 0.4 mmol), [Ru(*p*-cymene)Cl_2]$_2$ (9.9 mg, 0.016 mmol), K_2CO_3 (16.7 mg, 0.12 mmol), **2a** (365.0 mg, 2.0 mmol)/EtOH (1 mL), and 1-chloro-4-(trifluoromethyl)benzene (18 μL, *d* = 1.353 g/mL, 24.4 mg, 0.135 mmol) sequentially at rt. Then, the reaction tube was put into an oil bath preheated to 50 °C. An aliquot of the resulting mixture was taken for ^{19}F NMR analysis every 40 mins.

Time (min)	Recovery of **1b** (%)	[**1b**] (M)	ln([**1b**])
40	69.1	0.2764	-1.28591
80	60.5	0.242	-1.41882
120	52.5	0.21	-1.56065
160	46.8	0.1872	-1.67558
200	41.4	0.1656	-1.79818
240	37.2	0.1488	-1.90515
280	34.0	0.136	-1.9951
320	30.8	0.1232	-2.09395

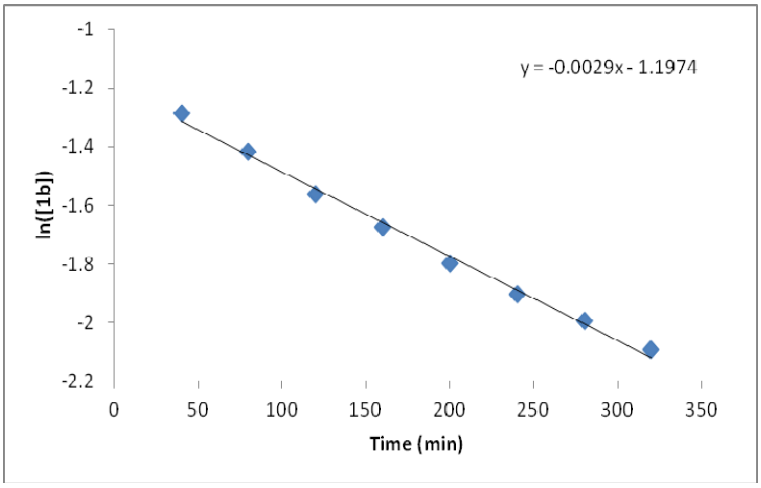
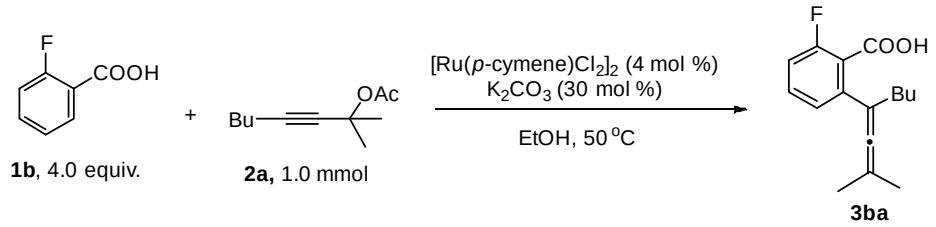


Figure S2. A first-order dependence of initial rate on **1b**.

(d) Determination of the order for propargylic acetate **2a.** (wxy-3-185)



Seven parallel experiments were carried out following the procedure below:

To a dried Schlenk tube were added 2-fluorobenzoic acid **1b** (112.1 mg, 0.8 mmol), [Ru(*p*-cymene)Cl_2]$_2$ (4.9 mg, 0.008 mmol), K_2CO_3 (8.3 mg, 0.06 mmol), and **2a** (36.5 mg, 0.2 mmol)/EtOH (0.5 mL) sequentially at rt. The reaction tube was put into an oil bath preheated to 50 °C. After being stirred for corresponding reaction time, the reaction mixture was filtrated through a short column of silica gel eluted with ethyl acetate (20 mL × 2) and concentration in vacuo. To the reaction residue was added 7 μL of CH_2Br_2 and analyzed with ^{1}H NMR measurement.

	Time (min)	Recovery of **2a** (%)	[**2a**] (M)	ln([**2a**])
wxy-3-185-G	30	85.9	0.3434	-1.06886
wxy-3-185-F	60	80.6	0.3224	-1.13196
wxy-3-185-E	90	76.4	0.3056	-1.18548
wxy-3-185-D	120	69.8	0.2794	-1.27511
wxy-3-185-C	150	67.8	0.271	-1.30564
wxy-3-185-B	180	64.1	0.2564	-1.36102
wxy-3-185-A	210	58.5	0.234	-1.45243

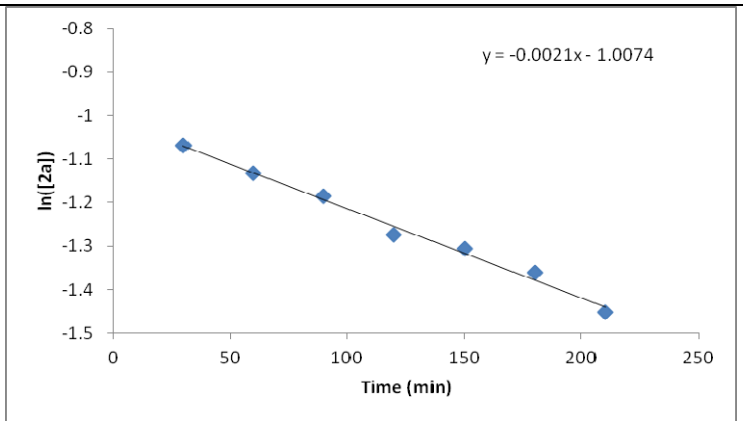
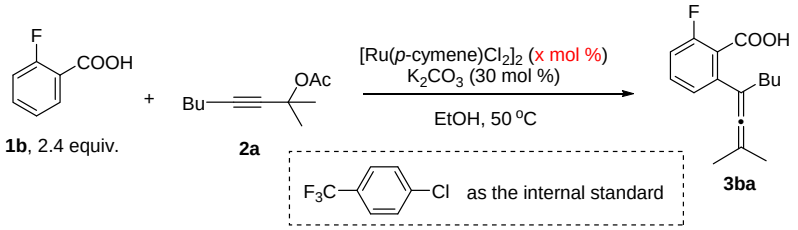


Figure S3. A first-order dependence of initial rate on **2a**.

(e) the dependency of the reaction rate on concentration of the ruthenium catalyst. (wxy-4-088A, wxy-4-088B, wxy-4-089A, wxy-4-089B)

Following **Typical Procedure II**, Four experiments with different concentration of $[Ru(p\text{-cymene})Cl_2]_2$ were carried out. The usage amount of starting material are listed blew:



	$[Ru(p\text{-cymene})Cl_2]_2$	**2a** (1.0 equiv.)	**1b** (2.4 equiv.)	K_2CO_3 (30 mol%)	F_3C—Cl	EtOH
2 mol% cat. (WXY-4-088A)	6.1 mg, 0.01 mmol	92.0 mg, 0.5 mmol	168.2 mg, 1.2 mmol	20.7 mg, 0.15 mmol	22.0 μL, 0.165 mmol	1.25 mL
3 mol% cat. (WXY-4-088B)	9.2 mg, 0.015 mmol	92.0 mg, 0.5 mmol	168.2 mg, 1.2 mmol	20.7 mg, 0.15 mmol	22.0 μL, 0.165 mmol	1.25 mL
4 mol% cat. (WXY-4-089A)	12.2 mg, 0.02 mmol	91.3 mg, 0.5 mmol	168.1 mg, 1.2 mmol	20.7 mg, 0.15 mmol	22.0 μL, 0.165 mmol	1.25 mL
6 mol% cat. (WXY-4-089B)	18.4 mg, 0.03 mmol	91.2 mg, 0.5 mmol	168.1 mg, 1.2 mmol	20.8 mg, 0.15 mmol	22.0 μL, 0.165 mmol	1.25 mL

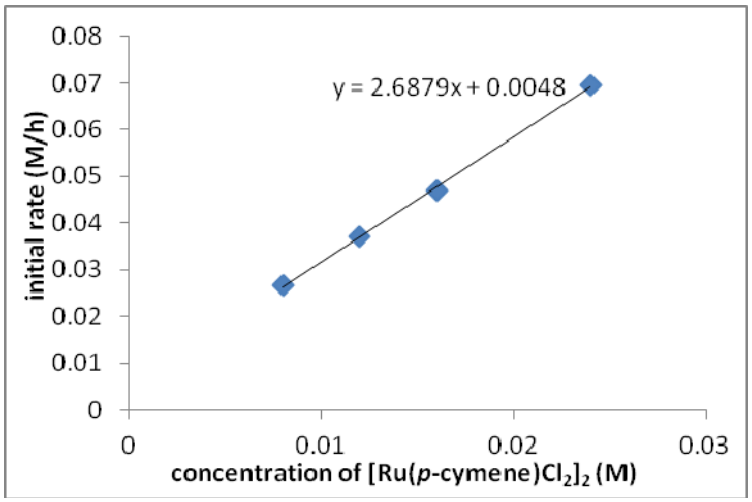


Figure S4. A first-order dependence of initial rate on the amount of $[Ru(p\text{-cymene})Cl_2]_2$.

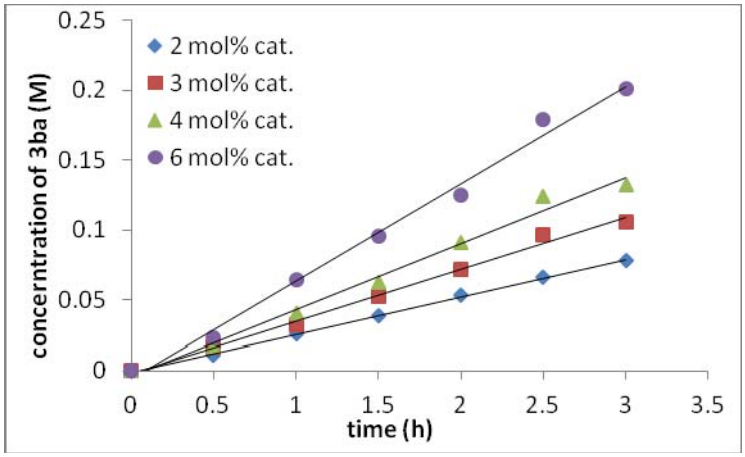


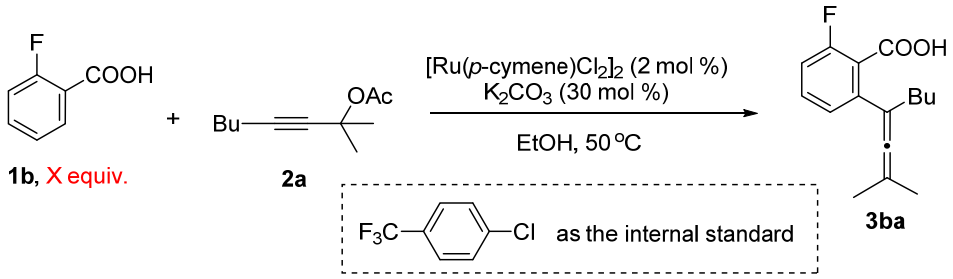
Figure S5. Plot of the concentrations of **3ba** over time with four different initial concentrations of $[Ru(p\text{-cymene})Cl_2]_2$.

The relevant data are listed below:

time (h)	NMR yield of **3ba** (%)			
	(2 mol% cat.) Wxy-4-088A	(3 mol% cat.) Wxy-4-088B	(4 mol% cat.) WXY-4-089A	(6 mol% cat.) WXY-4-089B
0.5	2.7	4.1	4.4	5.8
1	6.5	8.1	10.1	16.1
1.5	9.6	13	15.6	23.9
2	13.3	17.9	22.8	31.2
2.5	16.5	24.2	31	44.7
3	19.6	26.5	33	50.2

(f) the dependency of the reaction rate with different molar ratio of benzoic acid **1b** and propargylic acetate **2a**. (wxy-4-088A, wxy-4-093, wxy-4-113,wxy-4-094)

Following **Typical Procedure II**, Four experiments with different molar ratio of benzoic acid **1b** and propargylic acetate **2a** were carried out. The usage amount of starting material are listed blew:



	$[Ru(p\text{-cymene})Cl_2]_2$	**2a** (1.0 equiv.)	**1b** (2.4 equiv.)	K_2CO_3 (30 mol%)	F_3C—Cl	EtOH
1b:**2a** = 2.4:1 (WXY-4-088A)	6.1 mg, 0.01 mmol	92.0 mg, 0.5 mmol	168.2 mg, 1.2 mmol	20.7 mg, 0.15 mmol	22.0 μL, 0.165 mmol	1.25 mL
1b:**2a** = 1.5:1 (WXY-4-093)	6.2 mg, 0.01 mmol	91.1 mg, 0.5 mmol	105.1 mg, 0.75 mmol	20.9 mg, 0.15 mmol	22.0 μL, 0.165 mmol	1.25 mL
1b:**2a** = 1:1 (WXY-4-113)	6.1 mg, 0.01 mmol	91.4 mg, 0.5 mmol	70.1 mg, 0.5 mmol	20.7 mg, 0.15 mmol	22.0 μL, 0.165 mmol	1.25 mL
1b:**2a** = 1:1.5 (WXY-4-094)	6.1 mg, 0.01 mmol	136.8 mg, 0.75 mmol	70.1 mg, 0.5 mmol	20.5 mg, 0.15 mmol	22.0 μL, 0.165 mmol	1.25 mL

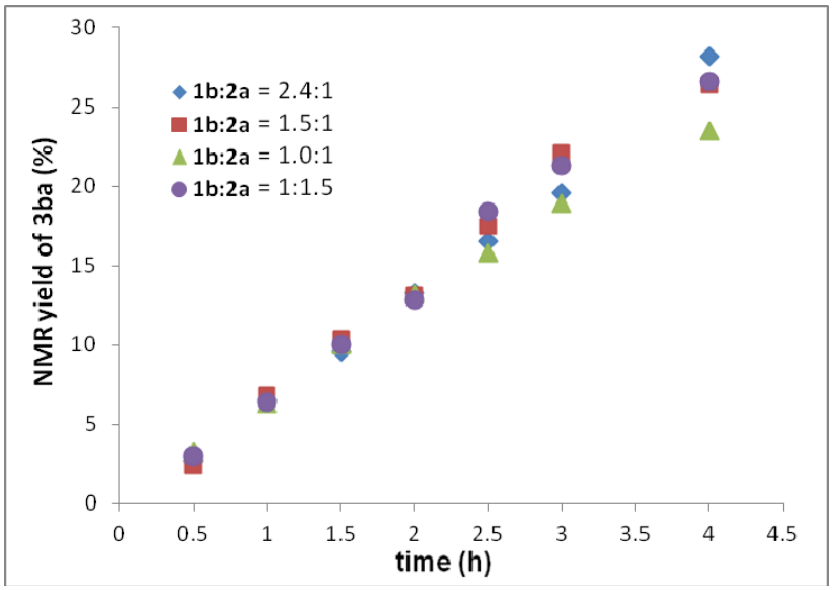


Figure S6. NMR yield of **3ba** vs. time depending on the molar ratio of **1b** and **2a**.

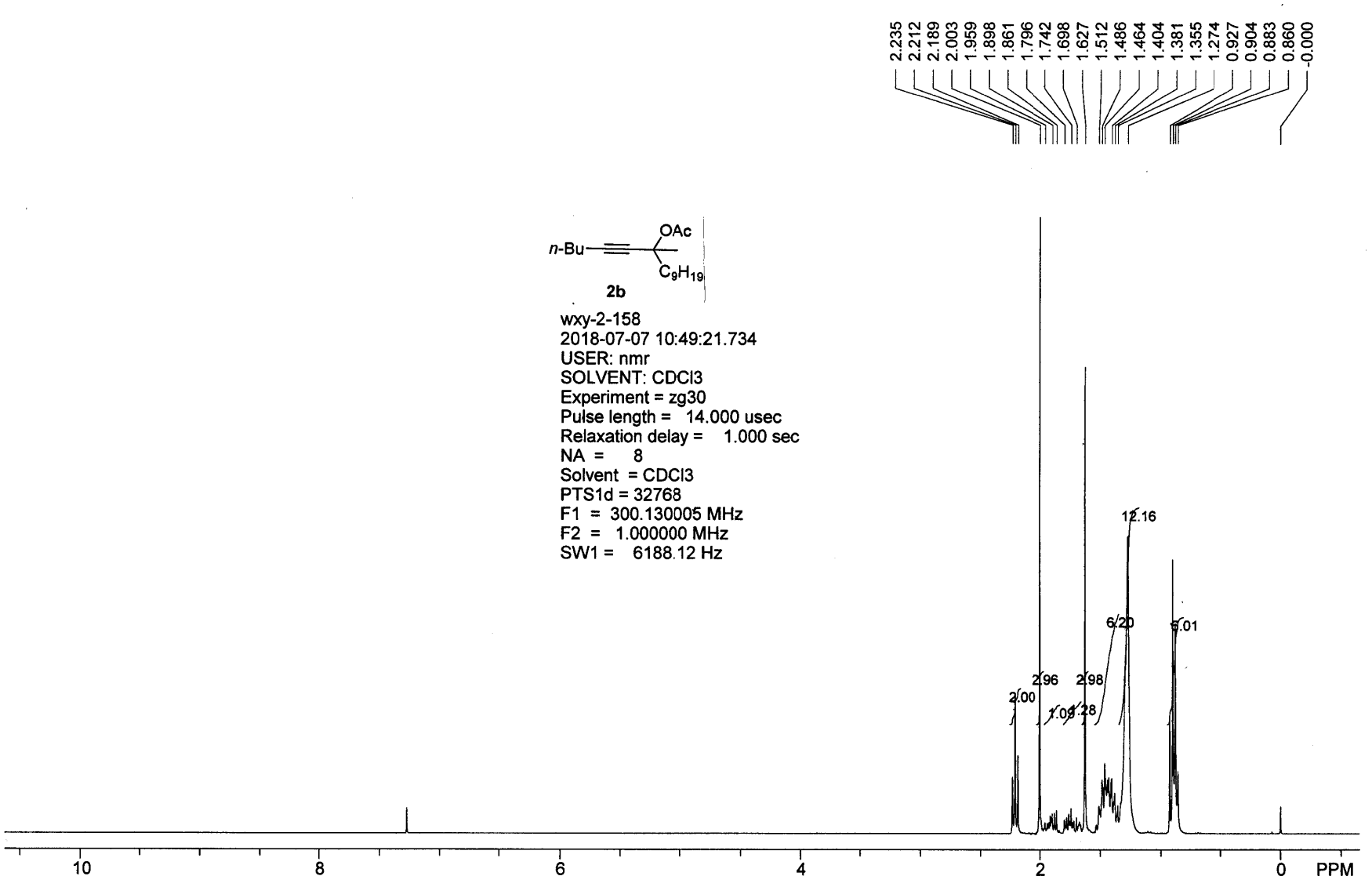
The experimental data are as follows:

time (h)	NMR yield of **3ba (%)**			
	1b:**2a** = 2.4:1	**1b**:**2a** = 1.5:1	**1b**:**2a** = 1:1	**1b**:**2a** = 1:1.5
0.5	2.7	2.4	3.3	3
1	6.5	6.8	6.3	6.4
1.5	9.6	10.4	10	10
2	13.3	13.1	13.2	12.8
2.5	16.5	17.5	15.8	18.4
3	19.6	22.1	18.9	21.3
4	28.2	26.4	23.5	26.6

References：

[1] page 186-190, Ph. D. dissertation, S, Wu. Zhejiang University, **2010**.

[2] W, Zhang.; S. Ma. *Chem. Commun.,* 2018, **54**, 6064.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2.189
2.003
1.959
1.898
1.861
1.796
1.742
1.698
1.627
1.512
1.486
1.464
1.404
1.381
1.355
1.274
0.927
0.904
0.883
0.860
-0.000
OAc
n-Bu
C9H19
2b
wxy-2-158
2018-07-07 10:49:21.734
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
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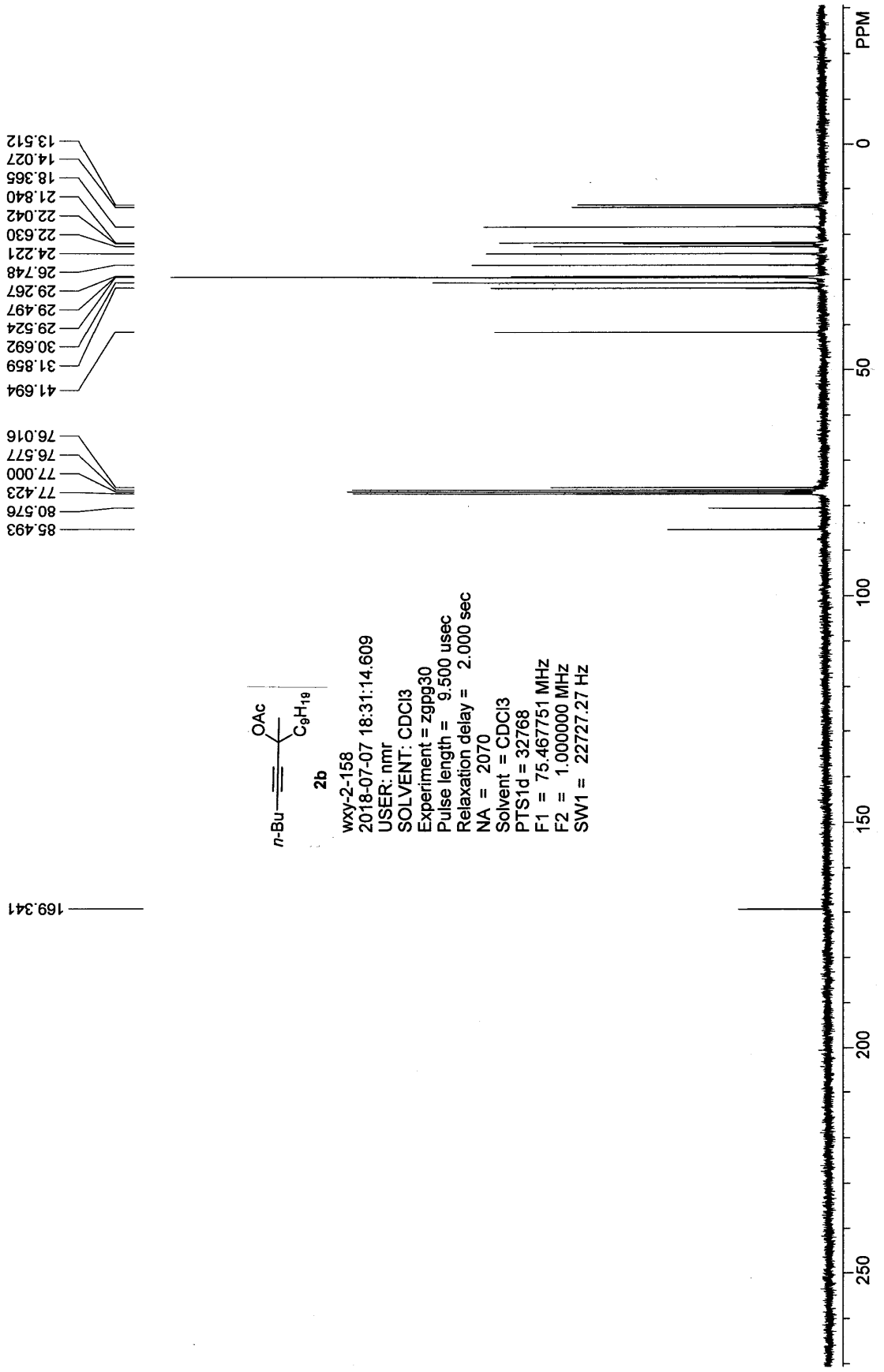
6.20
6.01
2.96
2.98
2.00
1.09
1.28
10
8
6
4
2
0
PPM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18.365
21.840
22.042
22.630
24.221
26.748
29.267
29.497
29.524
30.692
31.859
41.694
76.016
76.577
77.000
77.423
80.576
85.493
169.341
OAc
n-Bu
C9H19
2b
wxy-2-158
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SOLVENT: CDCl3
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Relaxation delay = 2.000 sec
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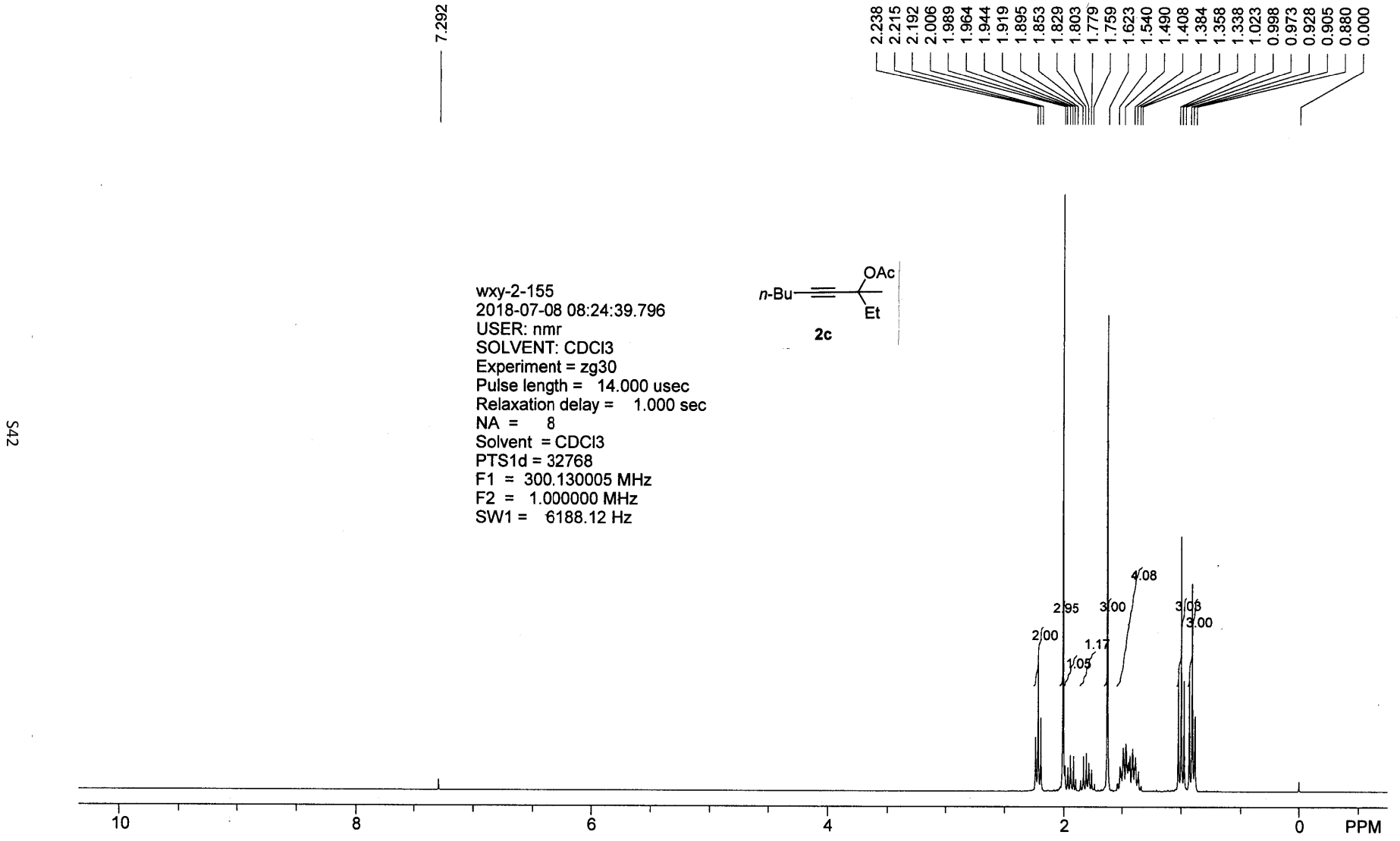
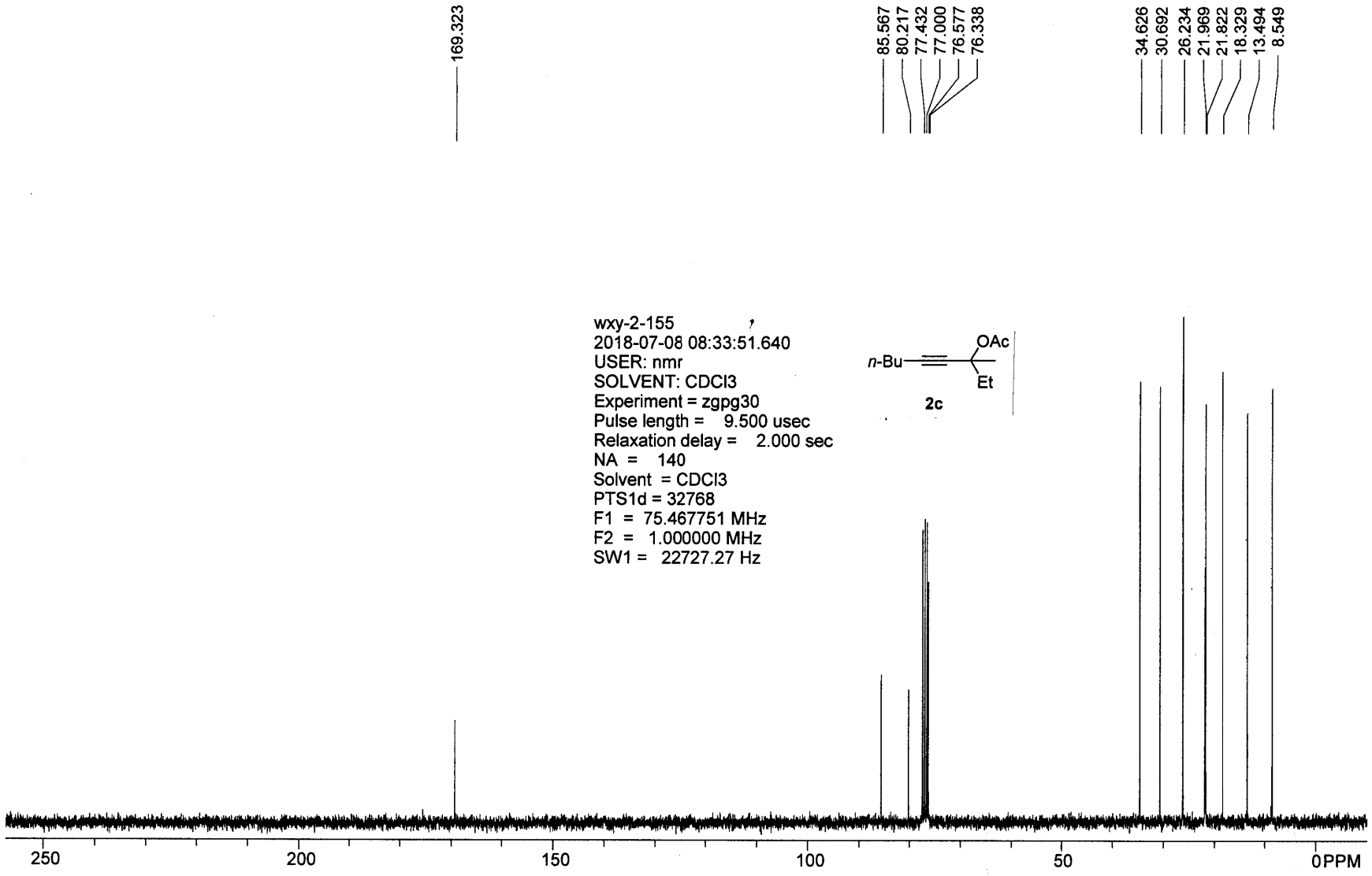
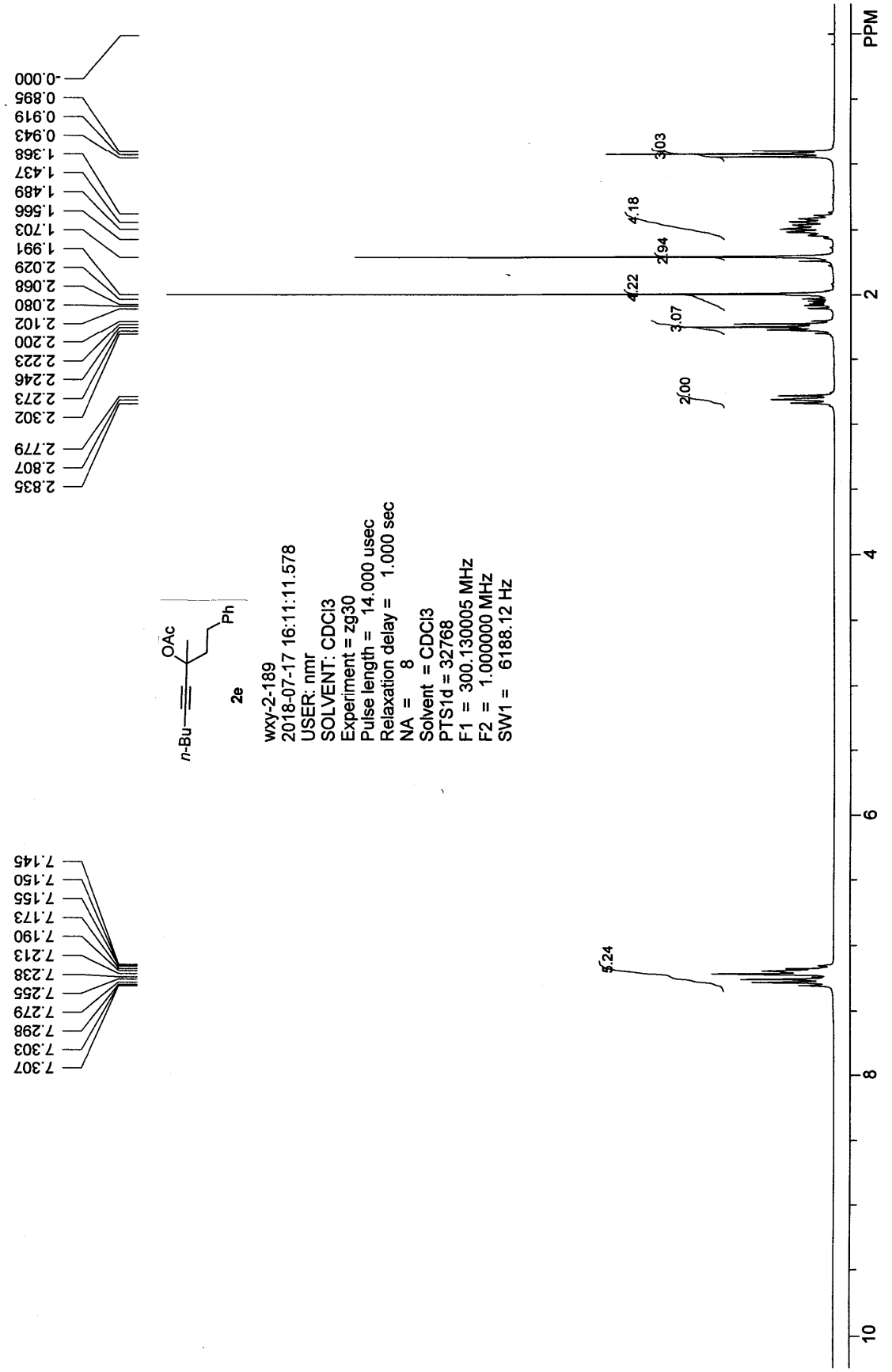
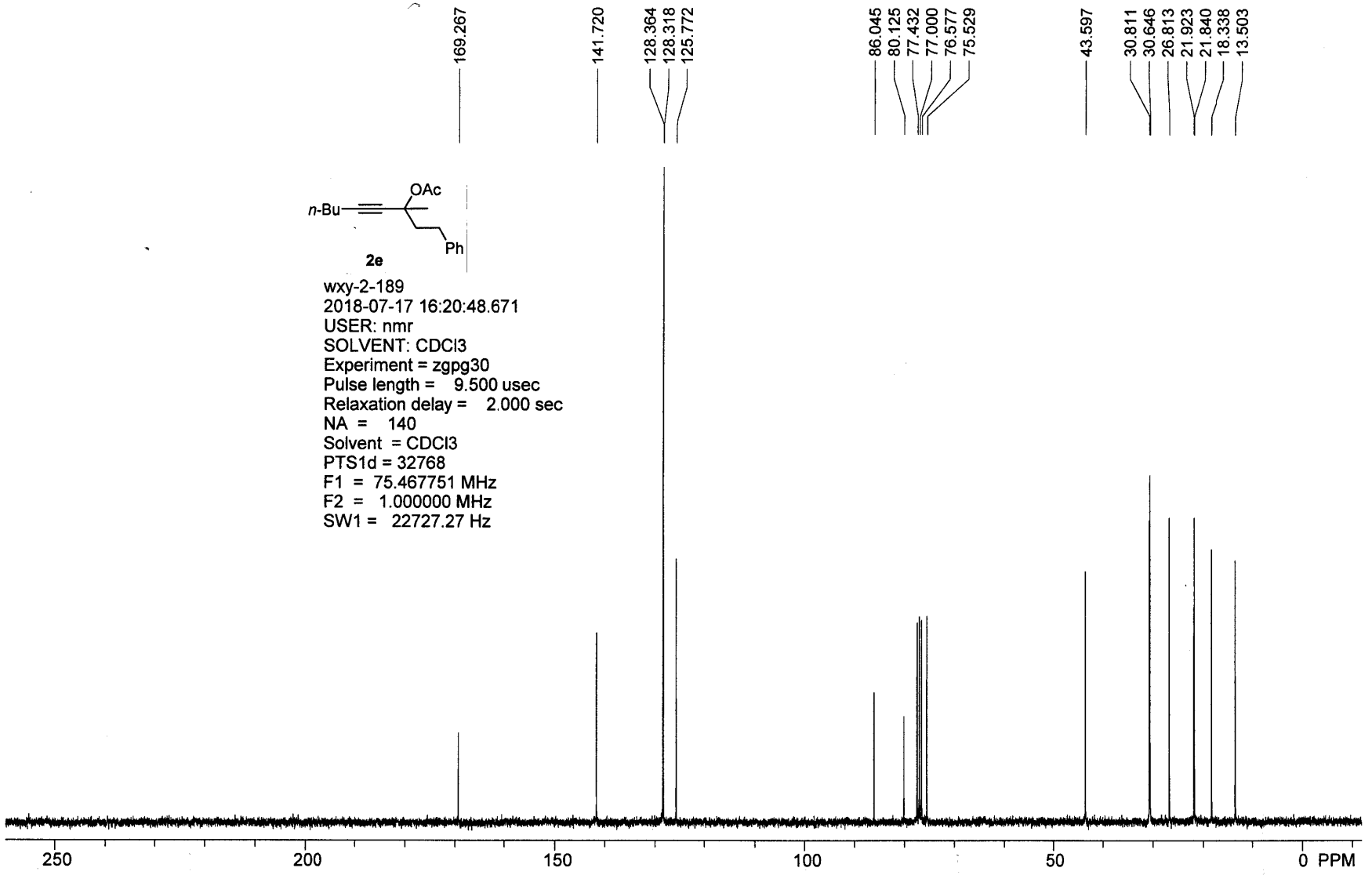
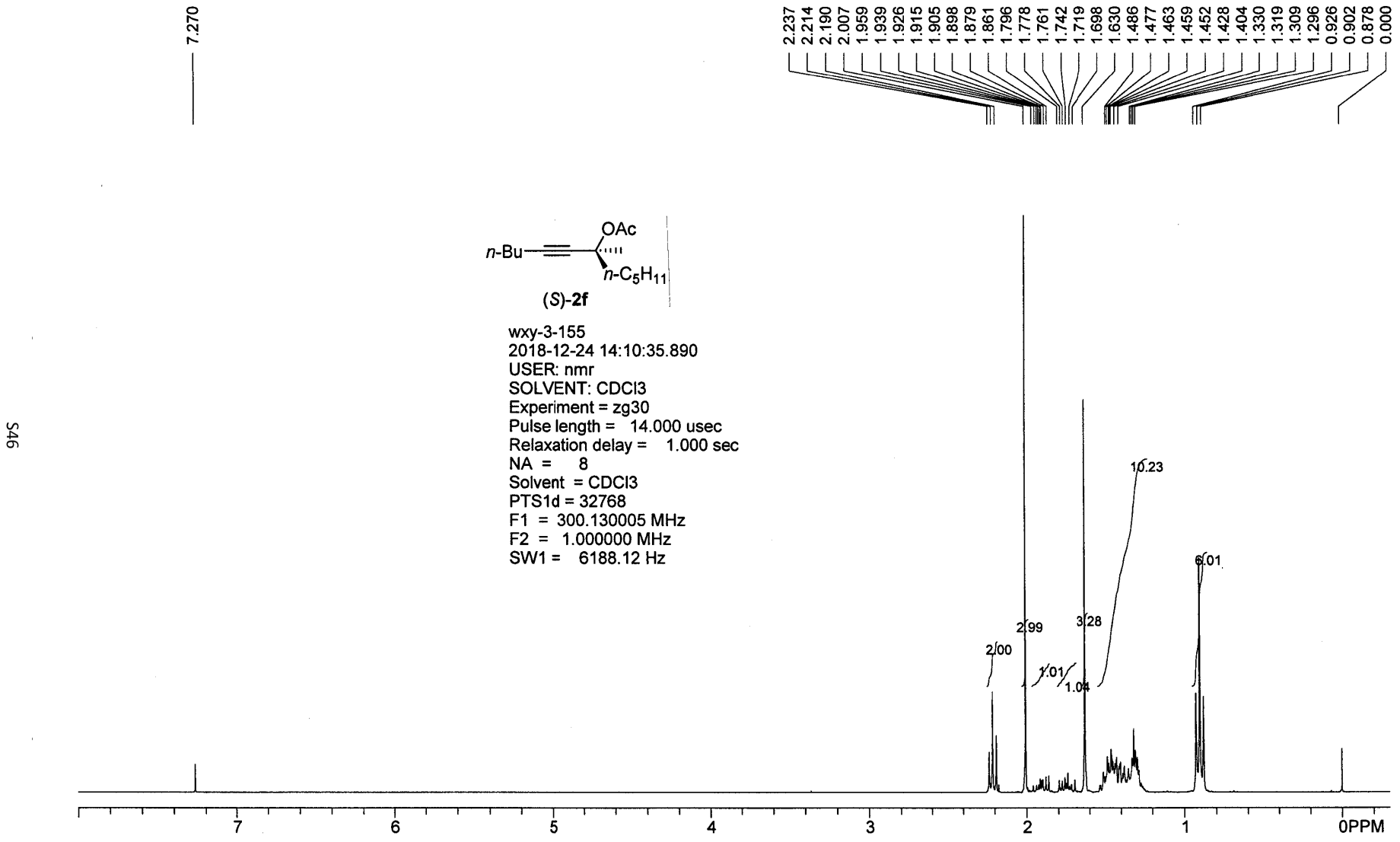
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
PPM
0
50
100
150
200
250

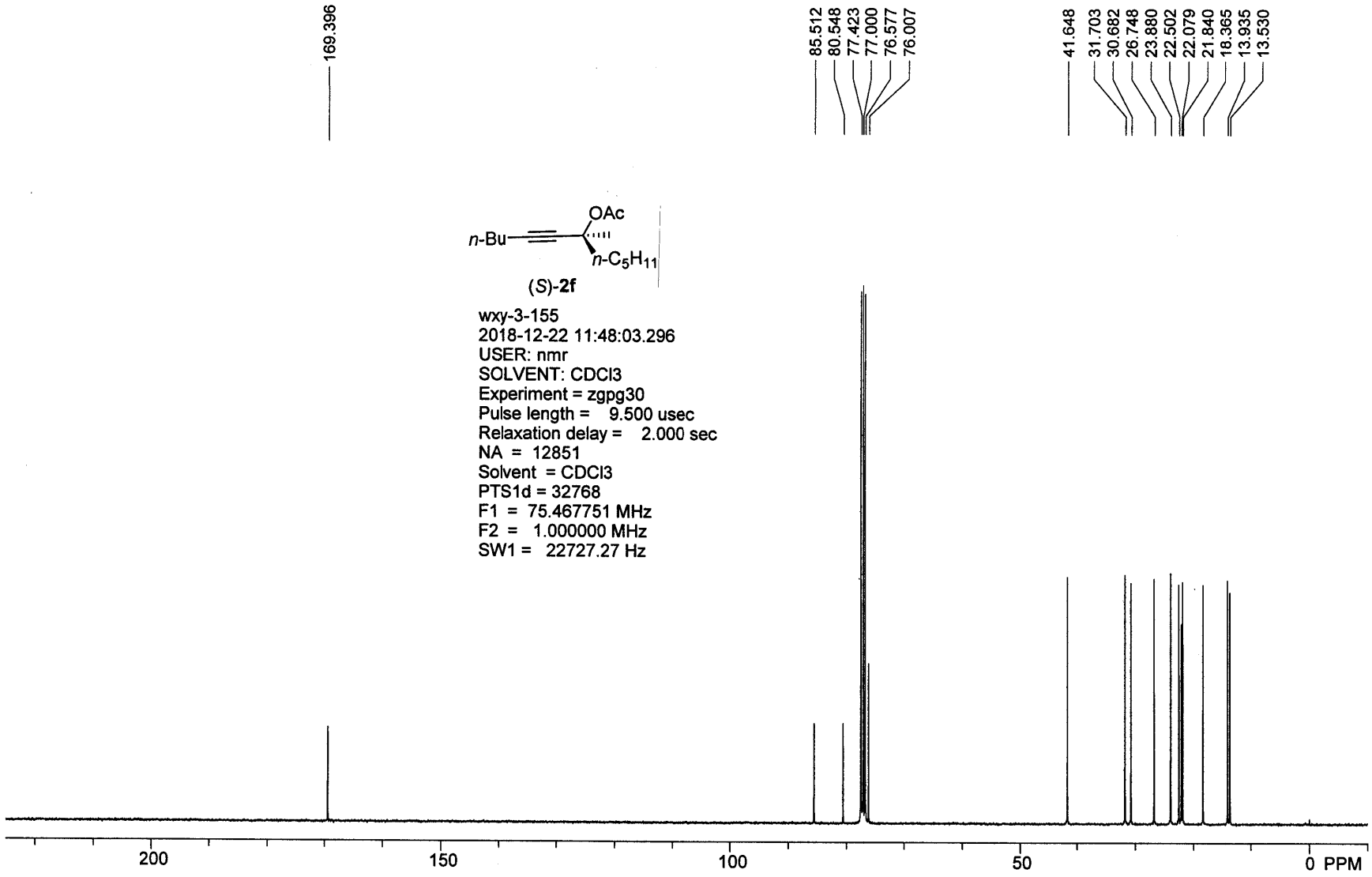

7.292
2.238
2.215
2.192
2.006
1.989
1.964
1.944
1.919
1.895
1.853
1.829
1.803
1.779
1.759
1.623
1.540
1.490
1.408
1.384
1.358
1.338
1.023
0.998
0.973
0.928
0.905
0.880
0.000
wxy-2-155
2018-07-08 08:24:39.796
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
n-Bu
OAc
Et
2c
2.00
2.95
1.05
1.17
3.00
4.08
3.03
3.00
10
8
6
4
2
0
PPM


169.323
85.567
80.217
77.432
77.000
76.577
76.338
34.626
30.692
26.234
21.969
21.822
18.329
13.494
8.549
wxy-2-155
2018-07-08 08:33:51.640
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 140
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
n-Bu
OAc
Et
2c
250
200
150
100
50
0PPM


wxy-2-189
2018-07-17 16:11:11.578
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
2e
n-Bu
OAc
Ph
PPM
5.24
2.00
3.07
2.94
4.18
3.03
4.22
7.307
7.303
7.298
7.279
7.255
7.238
7.213
7.190
7.173
7.155
7.150
7.145
2.835
2.807
2.779
2.302
2.273
2.246
2.223
2.200
2.102
2.080
2.068
2.029
1.991
1.703
1.566
1.489
1.437
1.368
0.943
0.919
0.895
0.000


169.267
141.720
128.364
128.318
125.772
86.045
80.125
77.432
77.000
76.577
75.529
43.597
30.811
30.646
26.813
21.923
21.840
18.338
13.503
OAc
n-Bu
Ph
2e
wxy-2-189
2018-07-17 16:20:48.671
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 140
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
250
200
150
100
50
0 PPM


7.270
2.237
2.214
2.190
2.007
1.959
1.939
1.926
1.915
1.905
1.898
1.879
1.861
1.796
1.778
1.761
1.742
1.719
1.698
1.630
1.486
1.477
1.463
1.459
1.452
1.428
1.404
1.330
1.319
1.309
1.296
0.926
0.902
0.878
0.000
n-Bu
OAc
n-C5H11
(S)-2f
wxy-3-155
2018-12-24 14:10:35.890
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
2.00
2.99
1.01
1.04
3.28
10.23
6.01
7
6
5
4
3
2
1
0PPM


169.396
85.512
80.548
77.423
77.000
76.577
76.007
41.648
31.703
30.682
26.748
23.880
22.502
22.079
21.840
18.365
13.935
13.530
OAc
n-Bu
n-C5H11
(S)-2f
wxy-3-155
2018-12-22 11:48:03.296
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 12851
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
200
150
100
50
0 PPM

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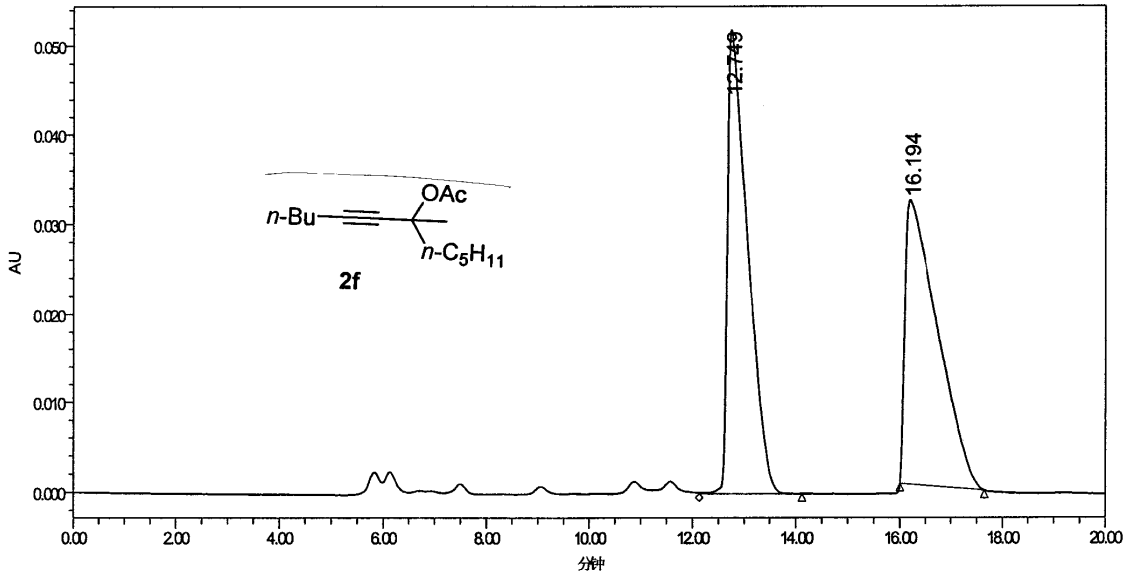
Project Name: defaults for copy

Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	wxy-3-155-rac-oz-h-100-0-1-214	Acquired By:	Breeze
Sample Type:	未知	Date Acquired:	2018/12/28 18:01:32 CST
Vial:	999	Acq. Method:	zgj100
Injection #:	5	Date Processed:	2018/12/28 18:37:06 CST
Injection Volume:	10.00 ul	Channel Name:	W2489 ChA
Run Time:	20.00 Minutes	Channel Desc.:	W2489 ChA 214nm
Column Type:		Sample Set Name:	



	RT (min)	Area (碘sec)	%Area	Height (碘)	% Height
1	12.749	1410827	50.01	51966	62.00
2	16.194	1410533	49.99	31849	38.00

Report Method: Individual Report ASC

Page: 1 (共计 1)

Printed: 2018/12/28 21:15:25 PRC

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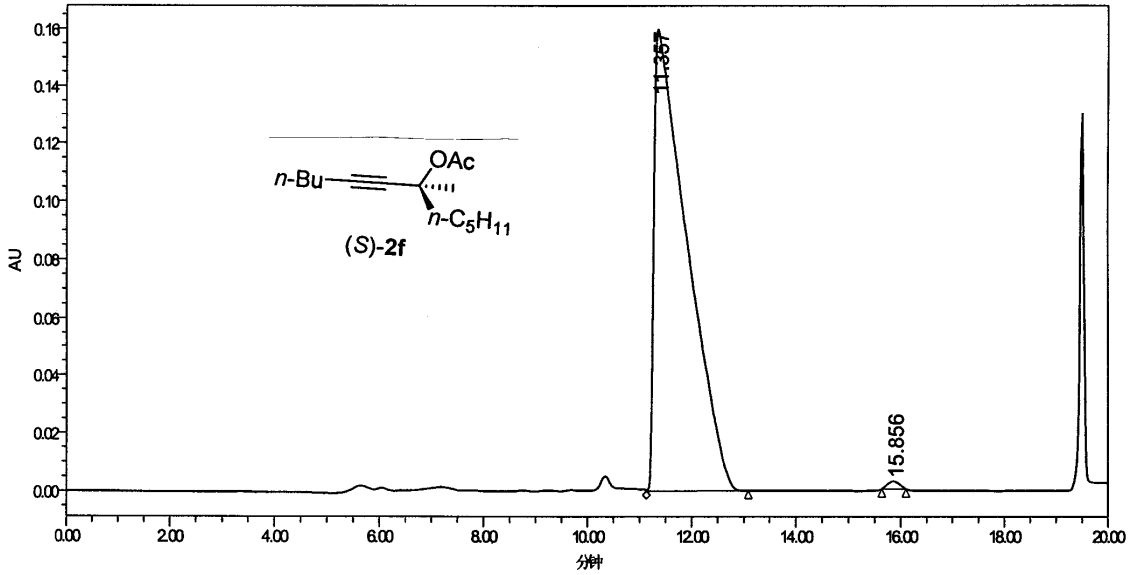
Project Name: defaults for copy

Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	wxy-3-155-oz-h-100-0-1-214	Acquired By:	Breeze
Sample Type:	未知	Date Acquired:	2018/12/28 17:25:08 CST
Vial:	999	Acq. Method:	zgj100
Injection #:	4	Date Processed:	2019/1/8 17:26:46 CST
Injection Volume:	10.00 ul	Channel Name:	W2489 ChA
Run Time:	20.00 Minutes	Channel Desc.:	W2489 ChA 214nm
Column Type:		Sample Set Name:	



	RT (min)	Area (微sec)	%Area	Height (微)	% Height
1	11.357	6972839	99.40	160112	98.36
2	15.856	41964	0.60	2662	1.64

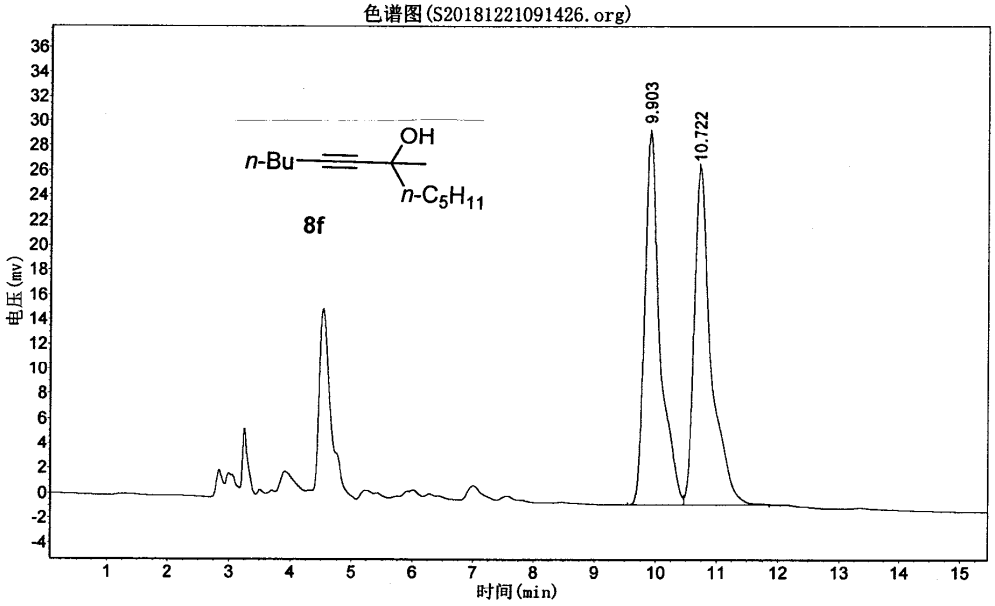
qya-7-024-racemic

实验时间：2018-12-21,9:14:26
谱图文件:D:\浙大智达\N2000\样品\S20181221091426.org
方法文件:D:\浙大智达\N2000\djx.mtd

实验者：wxy
报告时间：2018-12-21,9:57:48
积分方法:面积归一法

实验内容简介：
AD-H,n-hexane/i-PrOH = 99/1,1.0,214

色谱图(S20181221091426.org)



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		9.903	29890.783	523274.000	49.8870
2		10.722	27129.158	525644.063	50.1130
总计			57019.941	1048918.063	100.0000

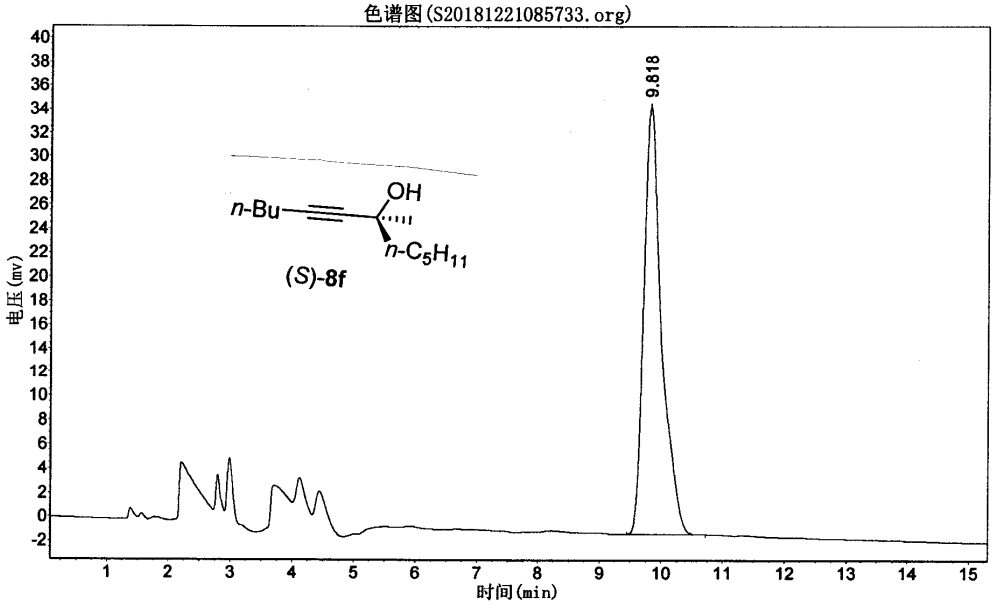
qya-7-024-S

实验时间：2018-12-21,8:57:33
谱图文件:D:\浙大智达\N2000\样品\S20181221085733.org
方法文件:D:\浙大智达\N2000\djx.mtd

实验者：wxy
报告时间：2018-12-21,10:04:52
积分方法:面积归一法

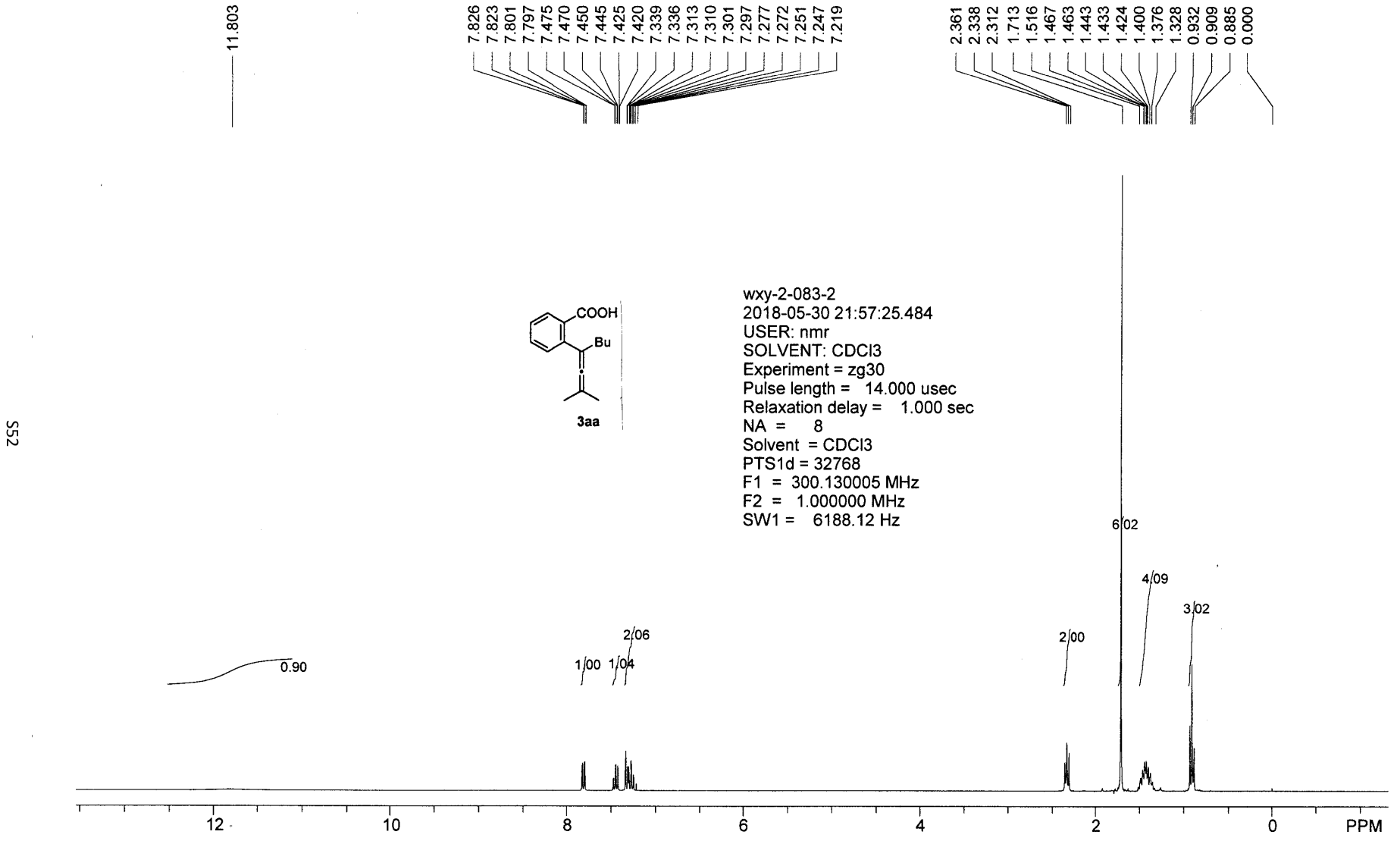
实验内容简介：
AD=-H,n-hexane/i-PrOH = 99/1,1.0,214

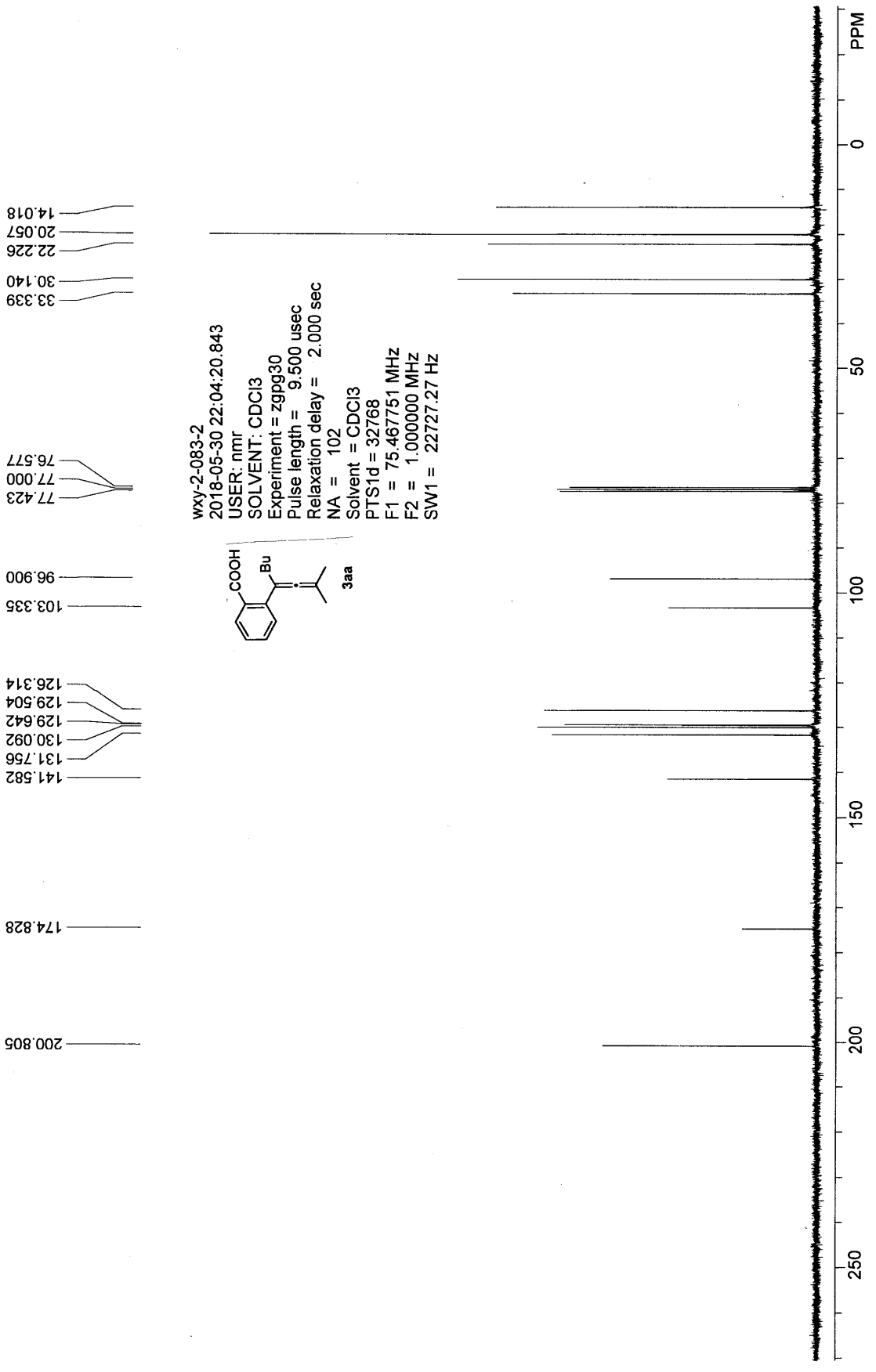
色谱图(S20181221085733.org)

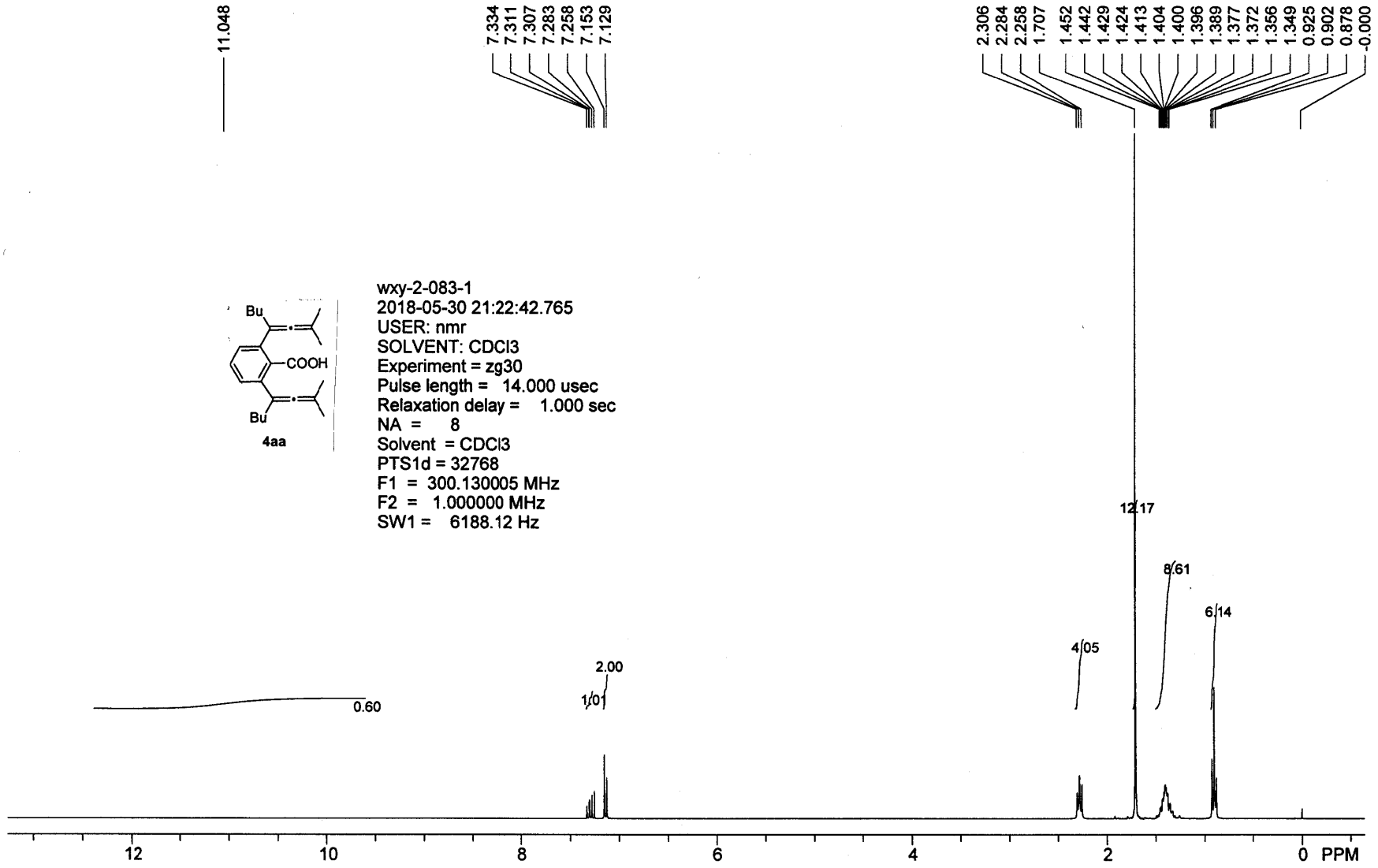


分析结果表

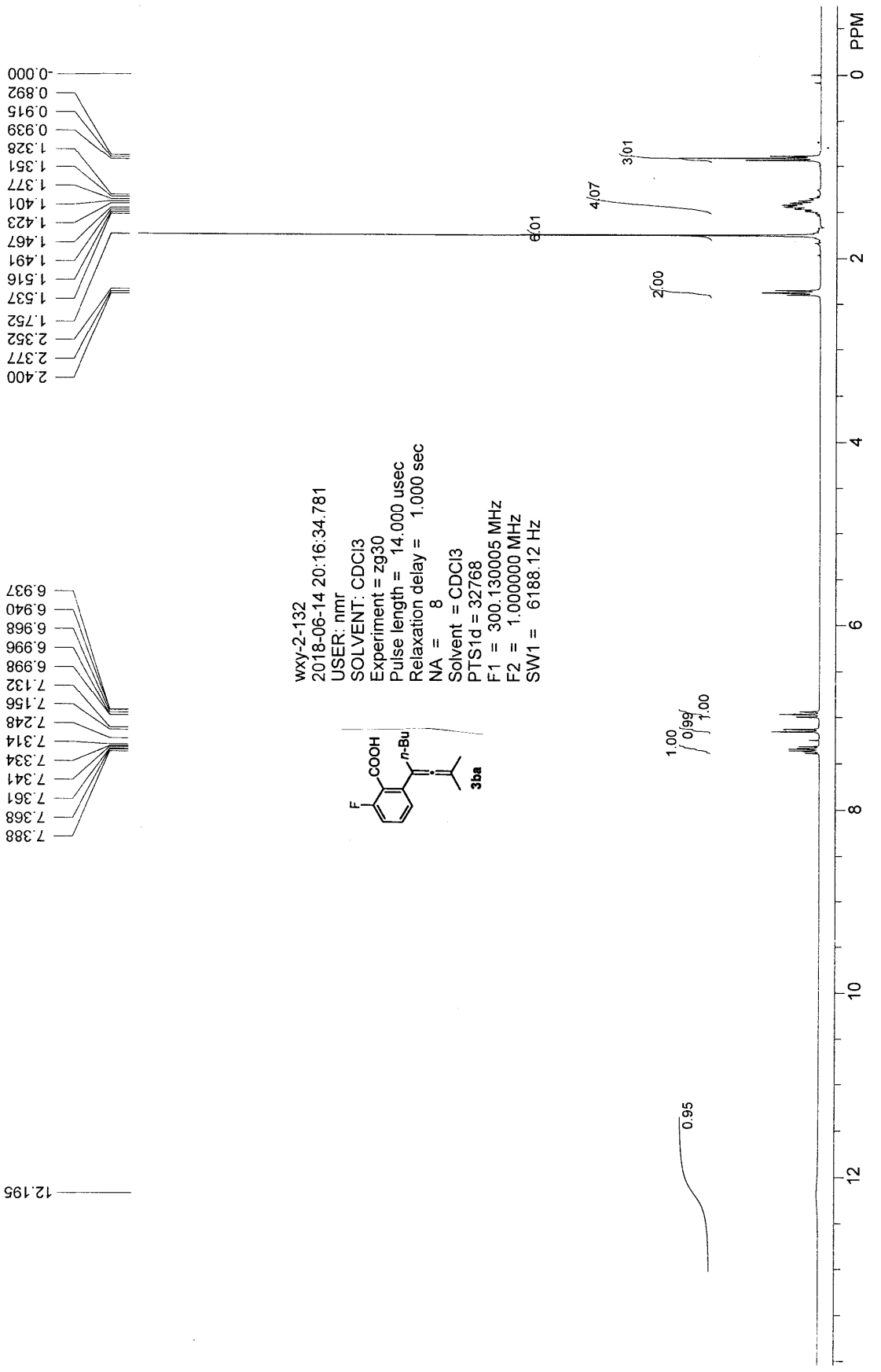
峰号	峰名	保留时间	峰高	峰面积	含量
1		9.818	35601.500	713972.188	100.0000
总计			35601.500	713972.188	100.0000

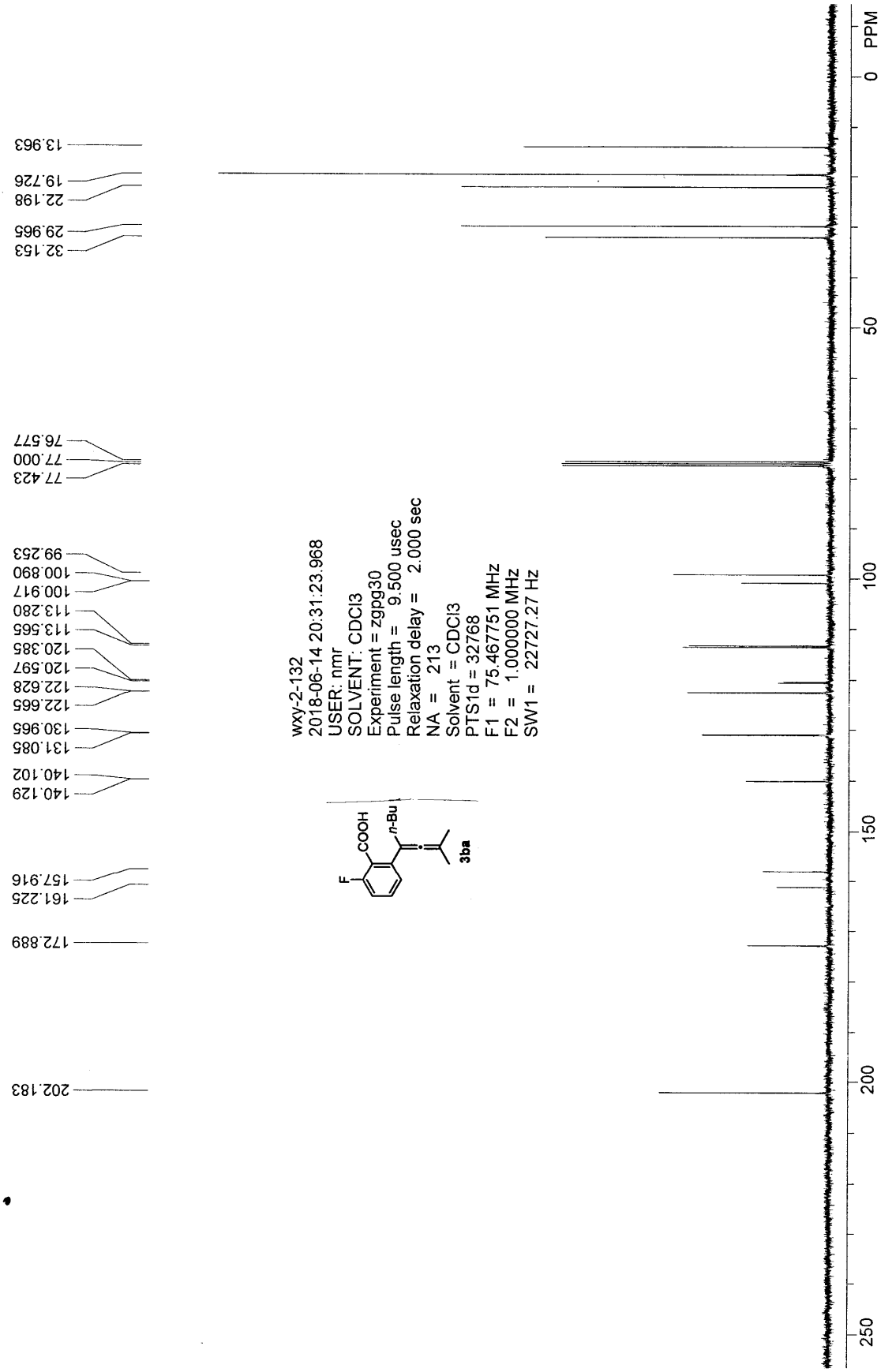

11.803
7.826
7.823
7.801
7.797
7.475
7.470
7.450
7.445
7.425
7.420
7.339
7.336
7.313
7.310
7.301
7.297
7.277
7.272
7.251
7.247
7.219
2.361
2.338
2.312
1.713
1.516
1.467
1.463
1.443
1.433
1.424
1.400
1.376
1.328
0.932
0.909
0.885
0.000
COOH
Bu
3aa
wxy-2-083-2
2018-05-30 21:57:25.484
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
0.90
1.00
1.04
2.06
2.00
6.02
4.09
3.02
12
10
8
6
4
2
0
PPM

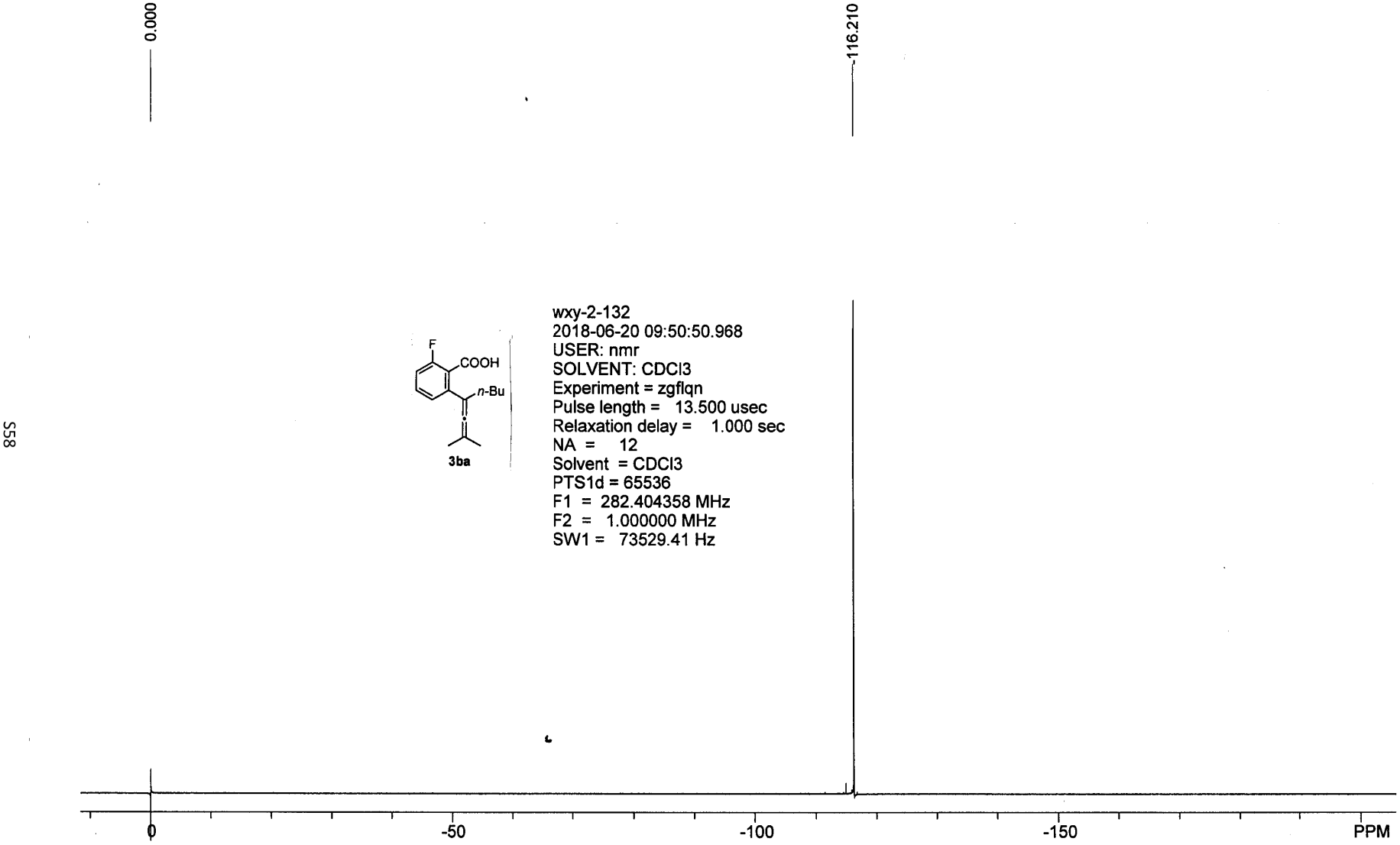

wxy-2-083-2
2018-05-30 22:04:20.843
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 102
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
COOH
Bu
3aa
14.018
20.057
22.226
30.140
33.339
76.577
77.000
77.423
96.900
103.335
126.314
129.504
129.642
130.092
131.756
141.582
174.828
200.805
PPM
0
50
100
150
200
250

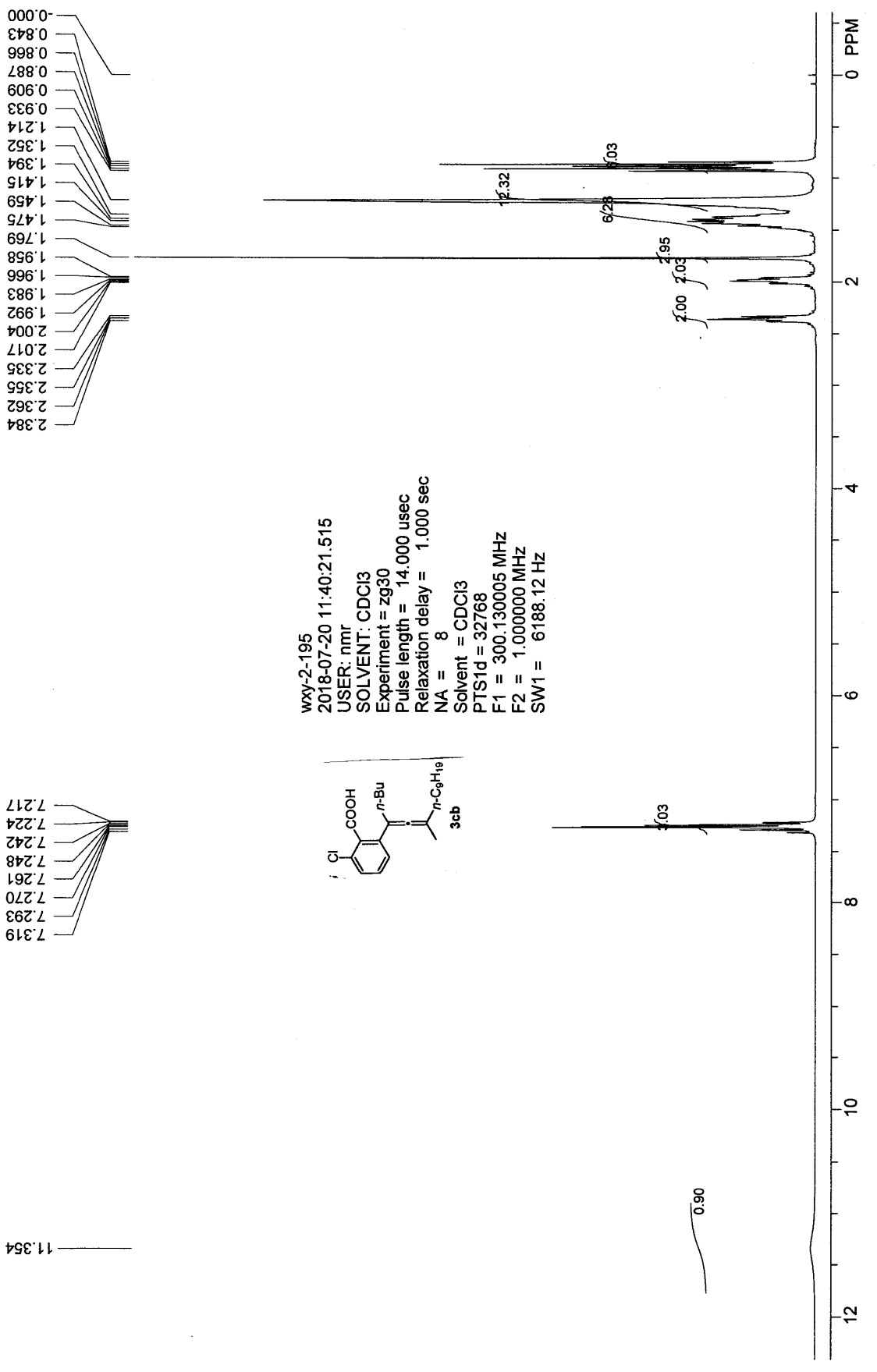

11.048
7.334
7.311
7.307
7.283
7.258
7.153
7.129
2.306
2.284
2.258
1.707
1.452
1.442
1.429
1.424
1.413
1.404
1.400
1.396
1.389
1.377
1.372
1.356
1.349
0.925
0.902
0.878
-0.000
Bu
COOH
Bu
4aa
wxy-2-083-1
2018-05-30 21:22:42.765
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
12.17
8.61
6.14
4.05
2.00
1.01
0.60
12
10
8
6
4
2
0 PPM

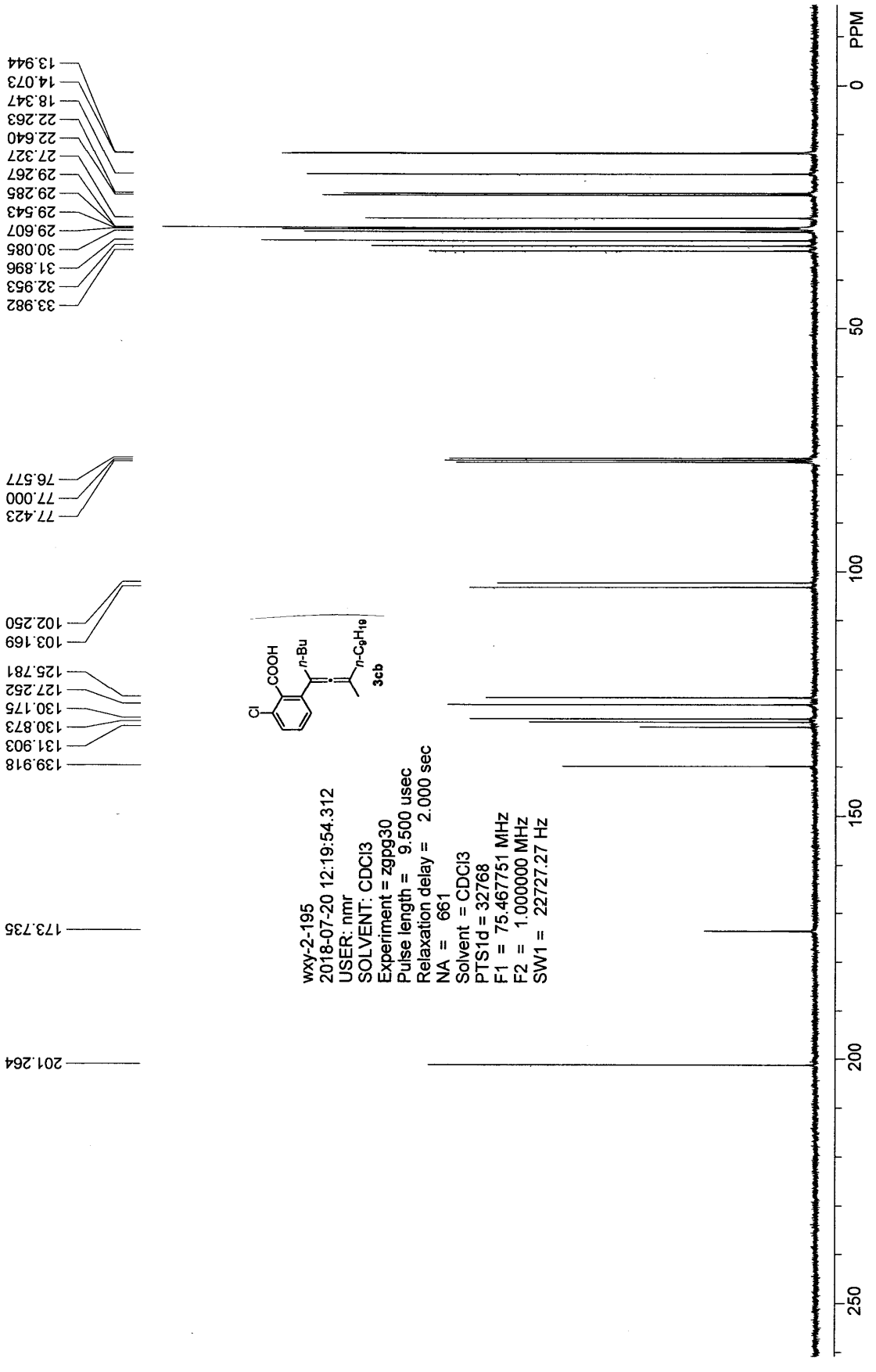

wxy-2-083-1
2018-05-30 21:51:12.343
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 472
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
COOH
Bu
Bu
4aa
14.036
20.489
22.281
29.993
33.854
76.577
77.000
77.423
96.919
102.103
126.691
128.952
131.581
138.355
173.634
200.676
0
50
100
150
200
250
PPM

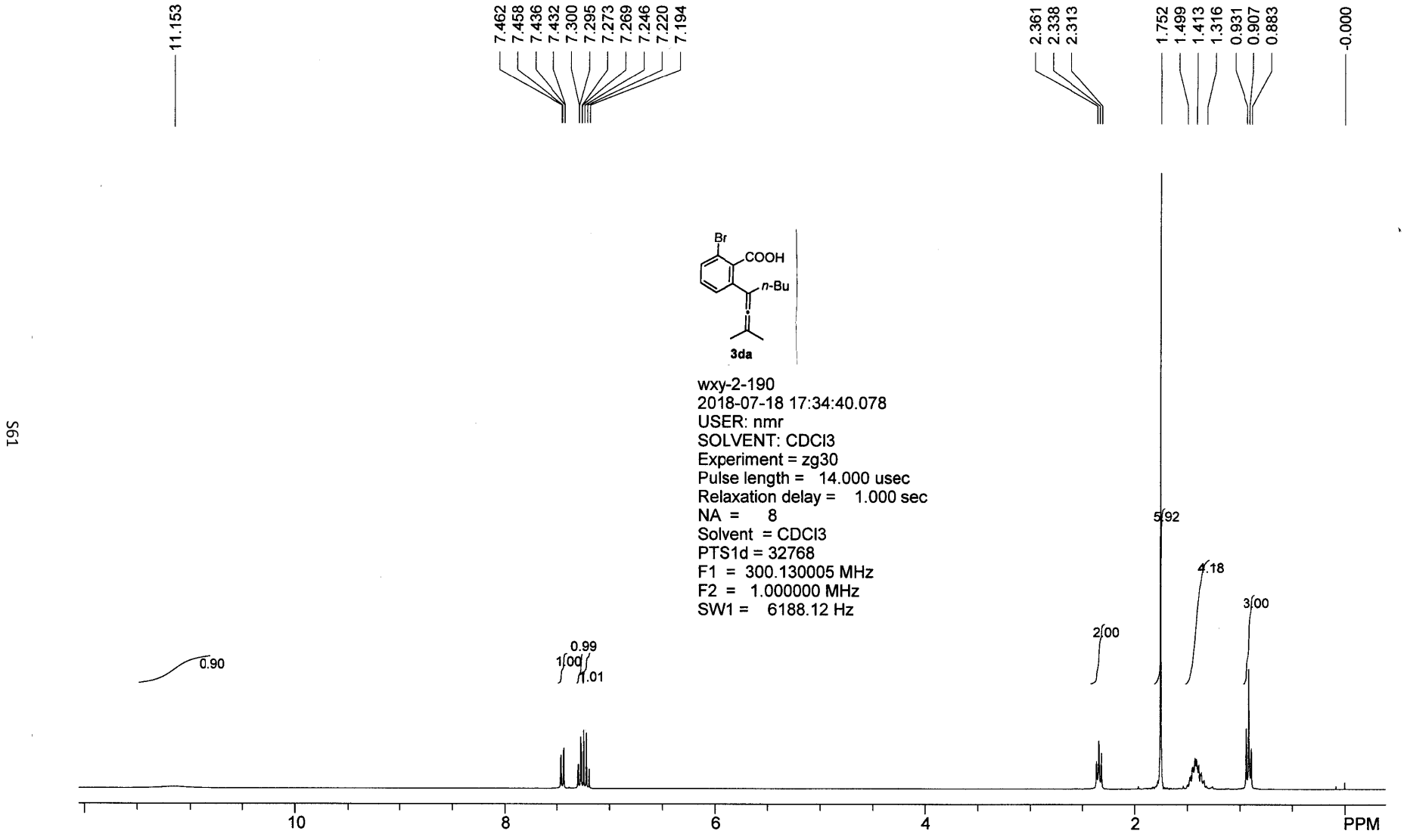

wxy-2-132
2018-06-14 20:16:34.781
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
COOH
n-Bu
F
3ba
6.01
4.07
3.01
2.00
1.00
0.95
PPM

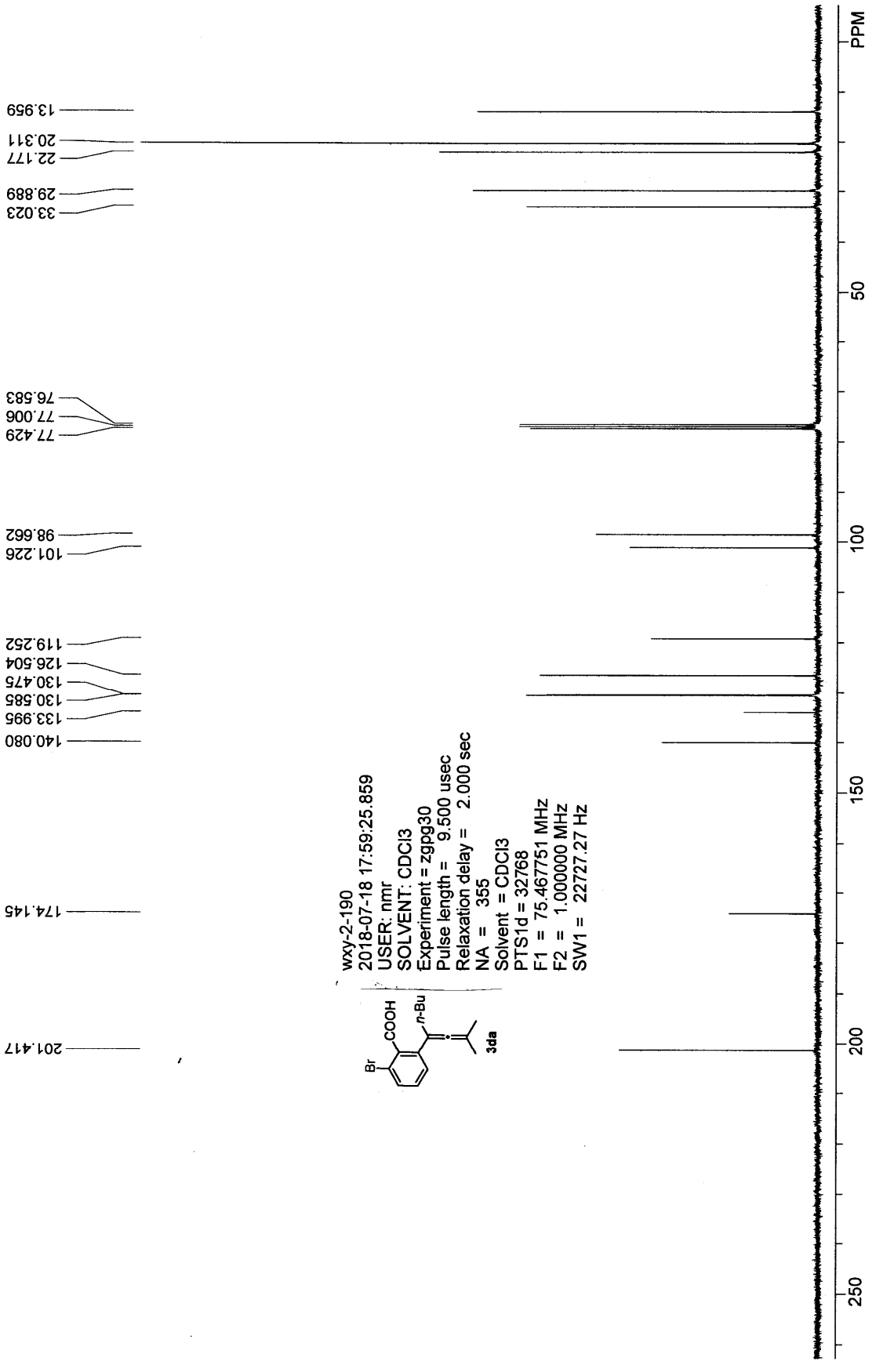

13.963
19.726
22.198
29.965
32.153
76.577
77.000
77.423
99.253
100.890
100.917
113.280
113.565
120.385
120.597
122.628
122.665
130.965
131.085
140.102
140.129
157.916
161.225
172.889
202.183
wxy-2-132
2018-06-14 20:31:23.968
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 213
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
F
COOH
n-Bu
3ba
0
50
100
150
200
250
PPM

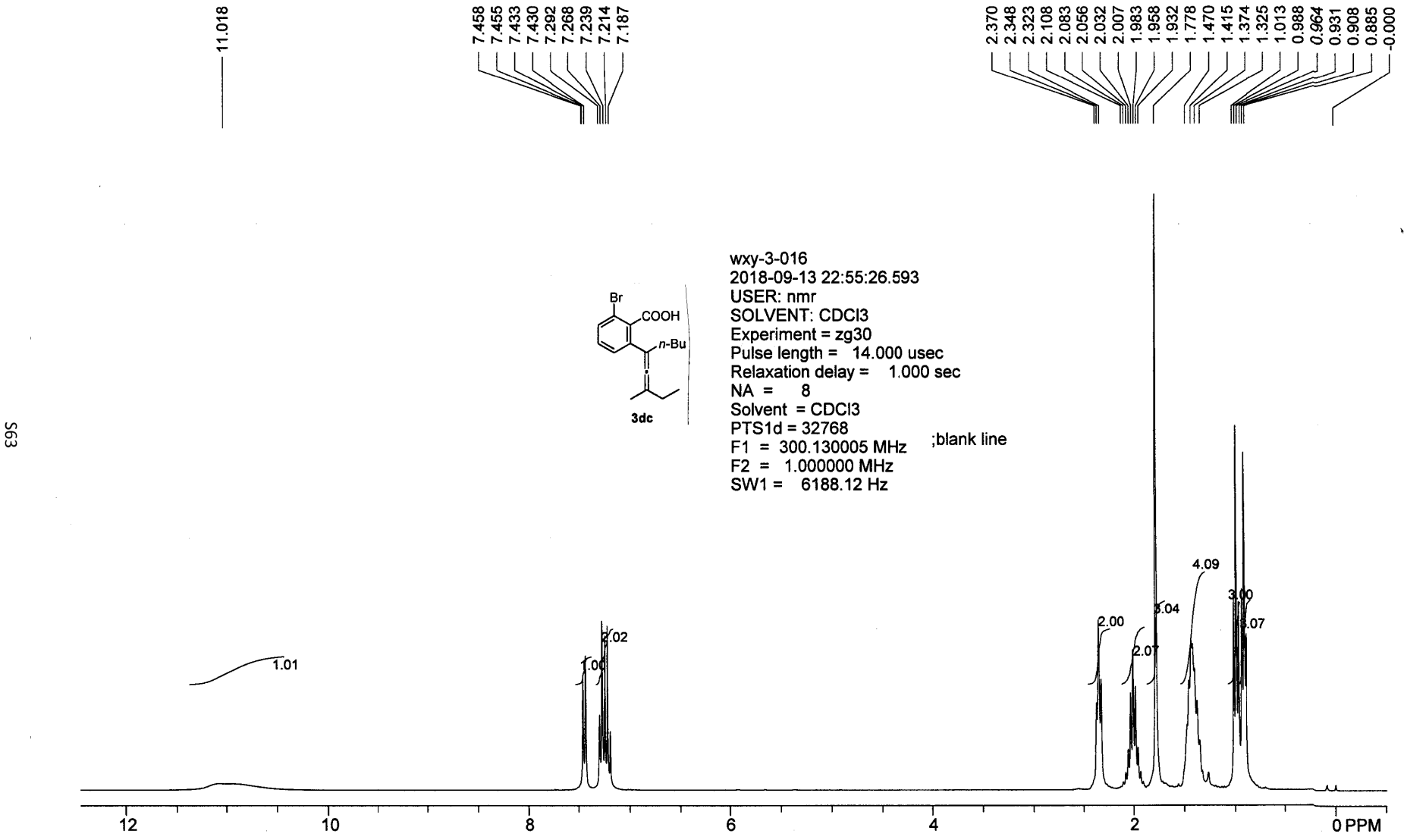

0.000
116.210
F
COOH
n-Bu
3ba
wxy-2-132
2018-06-20 09:50:50.968
USER: nmr
SOLVENT: CDCl3
Experiment = zgflqn
Pulse length = 13.500 usec
Relaxation delay = 1.000 sec
NA = 12
Solvent = CDCl3
PTS1d = 65536
F1 = 282.404358 MHz
F2 = 1.000000 MHz
SW1 = 73529.41 Hz
0
-50
-100
-150
PPM

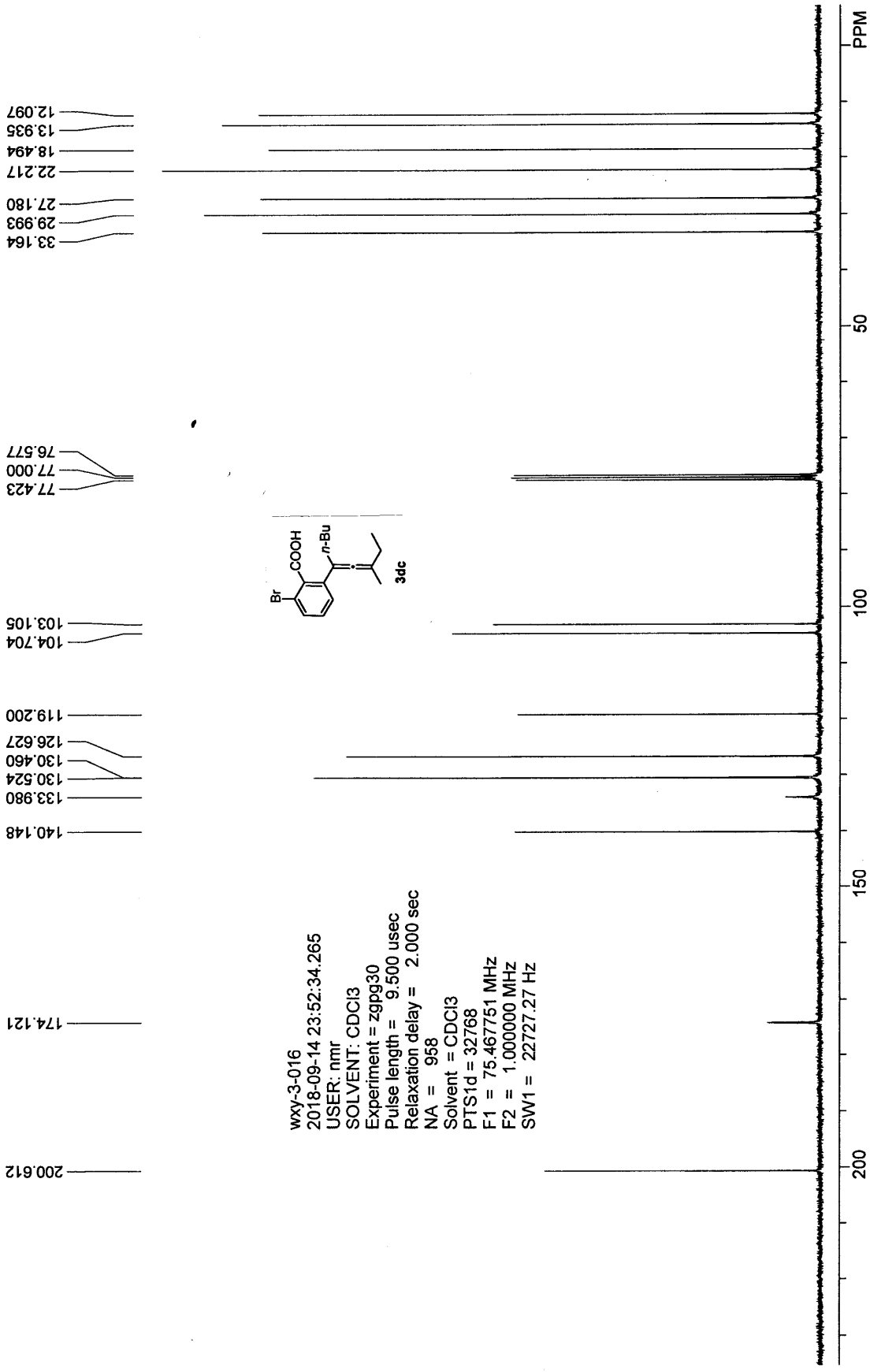

wxy-2-195
2018-07-20 11:40:21.515
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
3cb
PPM

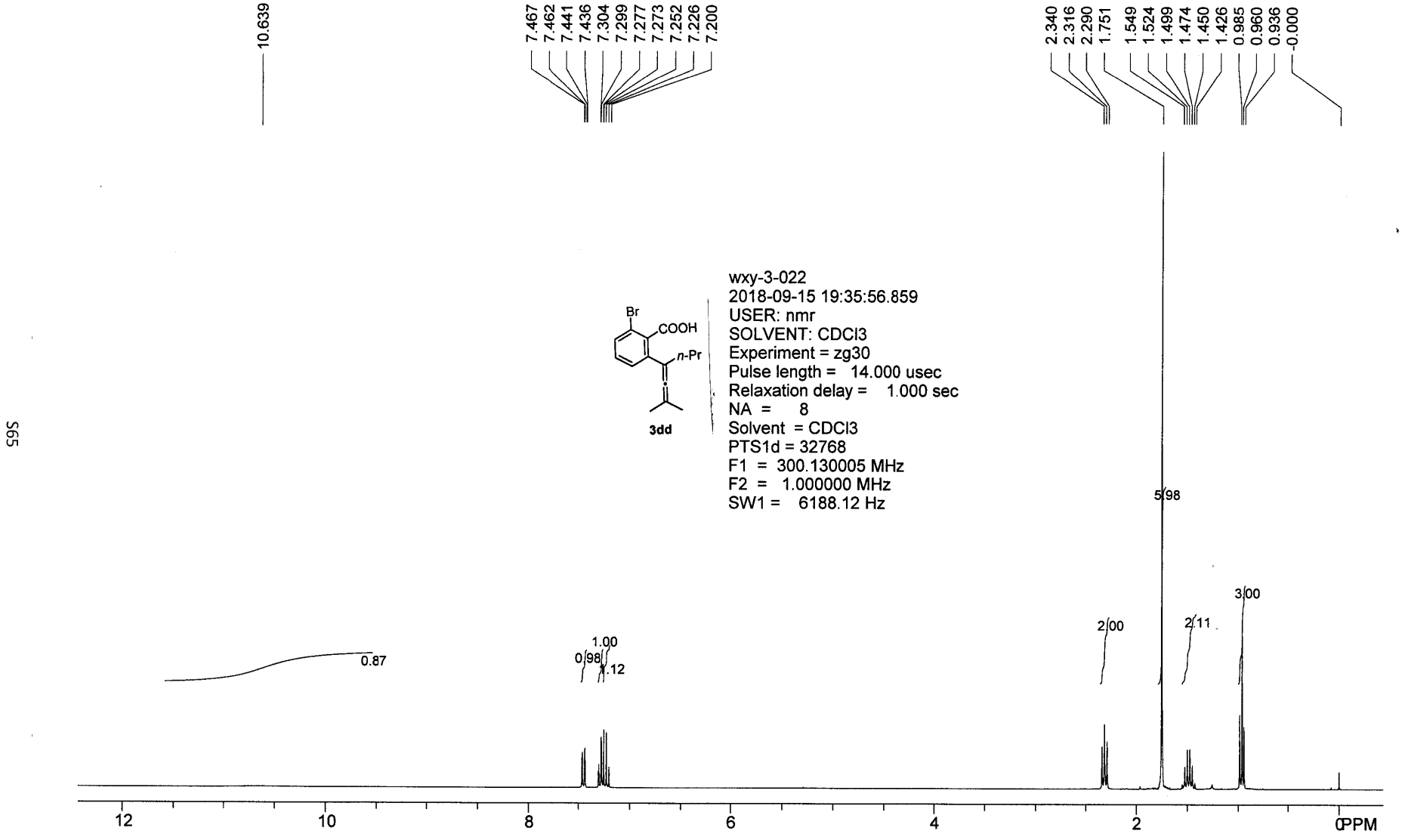

wxy-2-195
2018-07-20 12:19:54.312
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 661
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
3cb
PPM
0
50
100
150
200
250
13.944
14.073
18.347
22.263
22.640
27.327
29.267
29.285
29.543
29.607
30.085
31.896
32.953
33.982
76.577
77.000
77.423
102.250
103.169
125.781
127.252
130.175
130.873
131.903
139.918
173.735
201.264

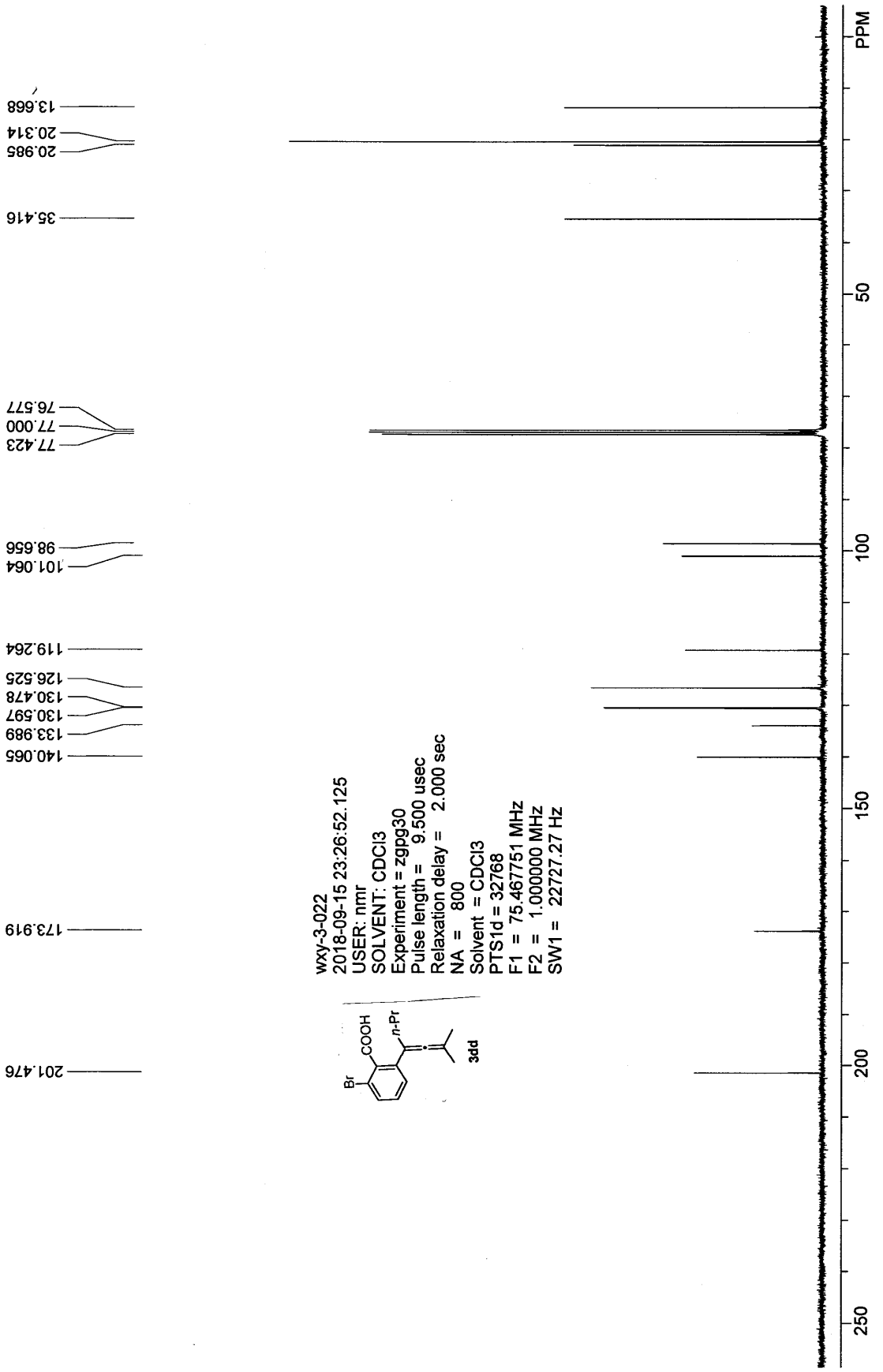

11.153
7.462
7.458
7.436
7.432
7.300
7.295
7.273
7.269
7.246
7.220
7.194
2.361
2.338
2.313
1.752
1.499
1.413
1.316
0.931
0.907
0.883
-0.000
Br
COOH
n-Bu
3da
wxy-2-190
2018-07-18 17:34:40.078
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
0.90
0.99
1.00
1.01
2.00
5.92
4.18
3.00
10
8
6
4
2
PPM

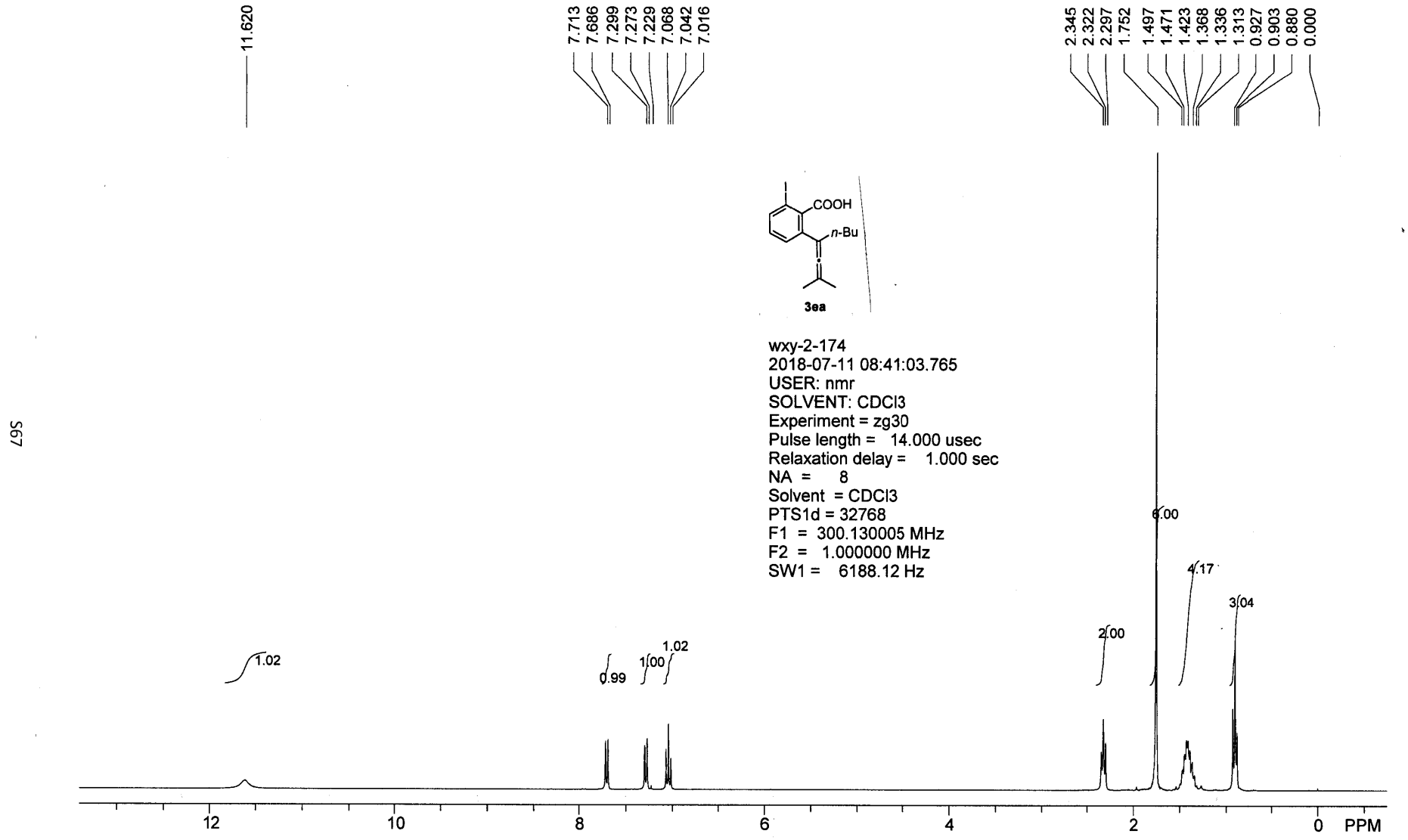

13.959
20.311
22.177
29.889
33.023
76.583
77.006
77.429
98.662
101.226
119.252
126.504
130.475
130.585
133.995
140.080
174.145
201.417
wxy-2-190
2018-07-18 17:59:25.859
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 355
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
COOH
n-Bu
Br
3da
PPM
250
200
150
100
50

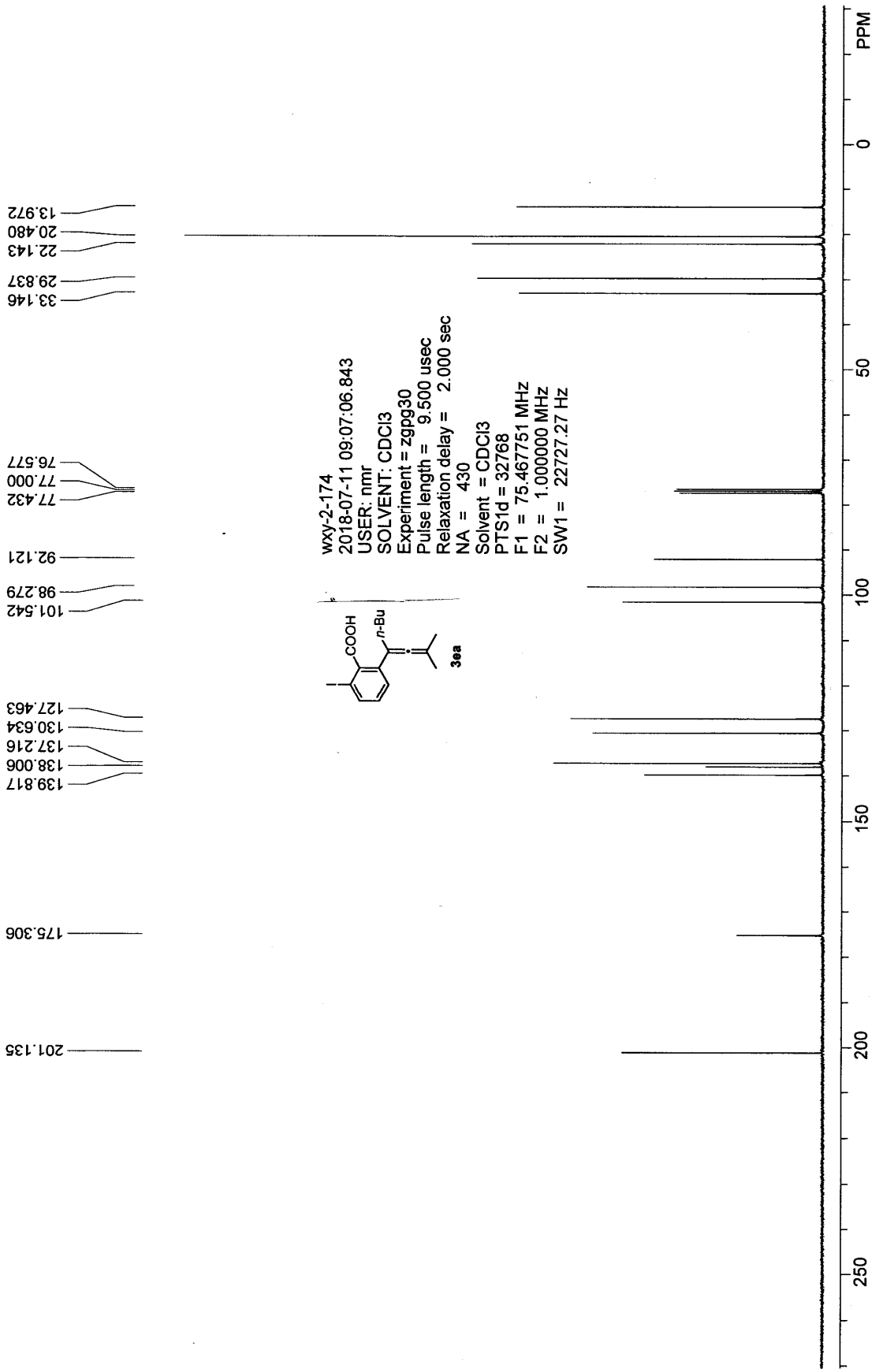

11.018
7.458
7.455
7.433
7.430
7.292
7.268
7.239
7.214
7.187
2.370
2.348
2.323
2.108
2.083
2.056
2.032
2.007
1.983
1.958
1.932
1.778
1.470
1.415
1.374
1.325
1.013
0.988
0.964
0.931
0.908
0.885
-0.000
Br
COOH
n-Bu
3dc
wxy-3-016
2018-09-13 22:55:26.593
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
;blank line
1.01
1.00
2.02
2.00
2.07
3.04
4.09
3.00
3.07
12
10
8
6
4
2
0 PPM

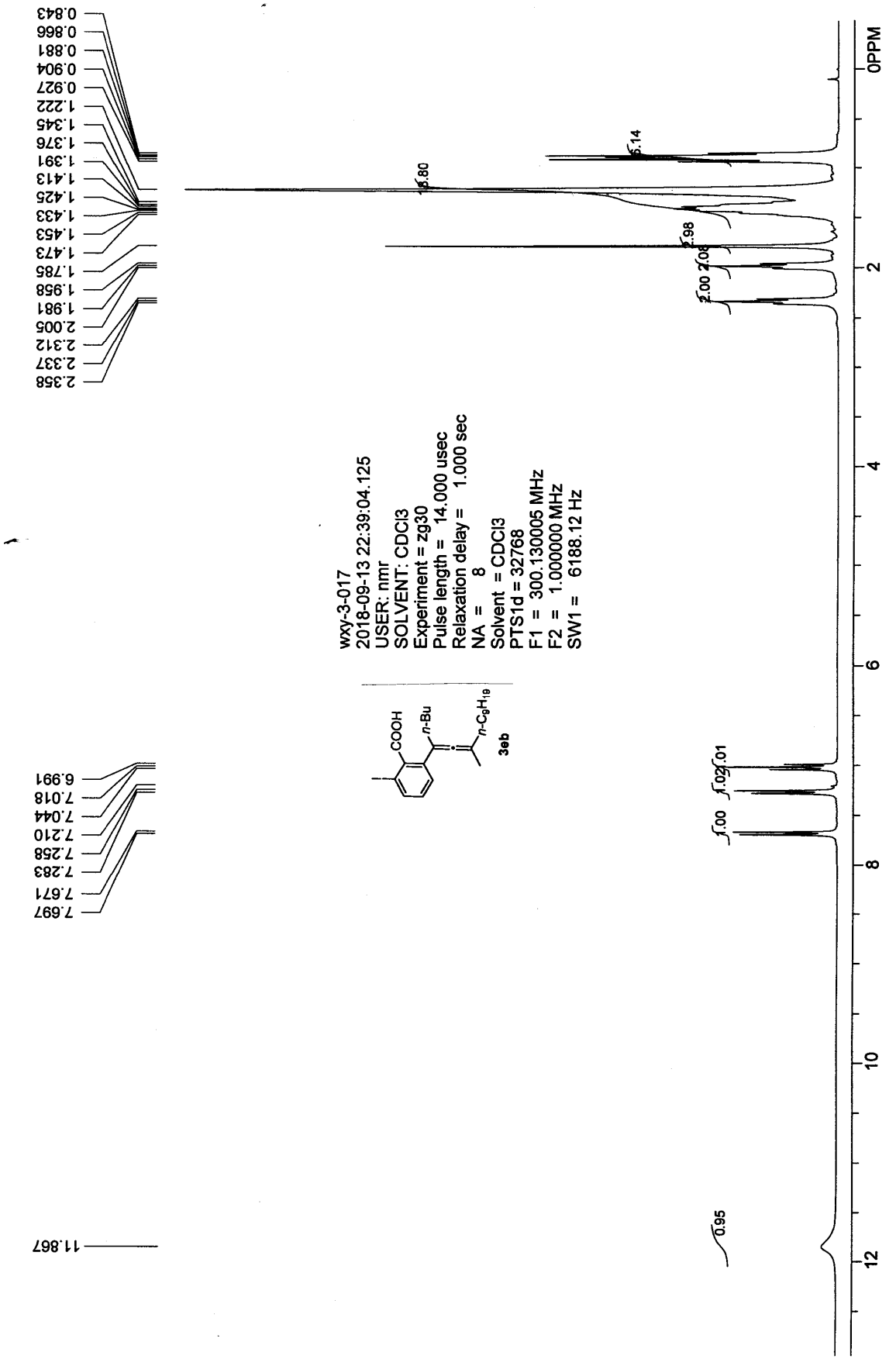

wxy-3-016
2018-09-14 23:52:34.265
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 958
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
Br
COOH
n-Bu
3dc
12.097
13.935
18.494
22.217
27.180
29.993
33.164
76.577
77.000
77.423
103.105
104.704
119.200
126.627
130.460
130.524
133.980
140.148
174.121
200.612
PPM
50
100
150
200

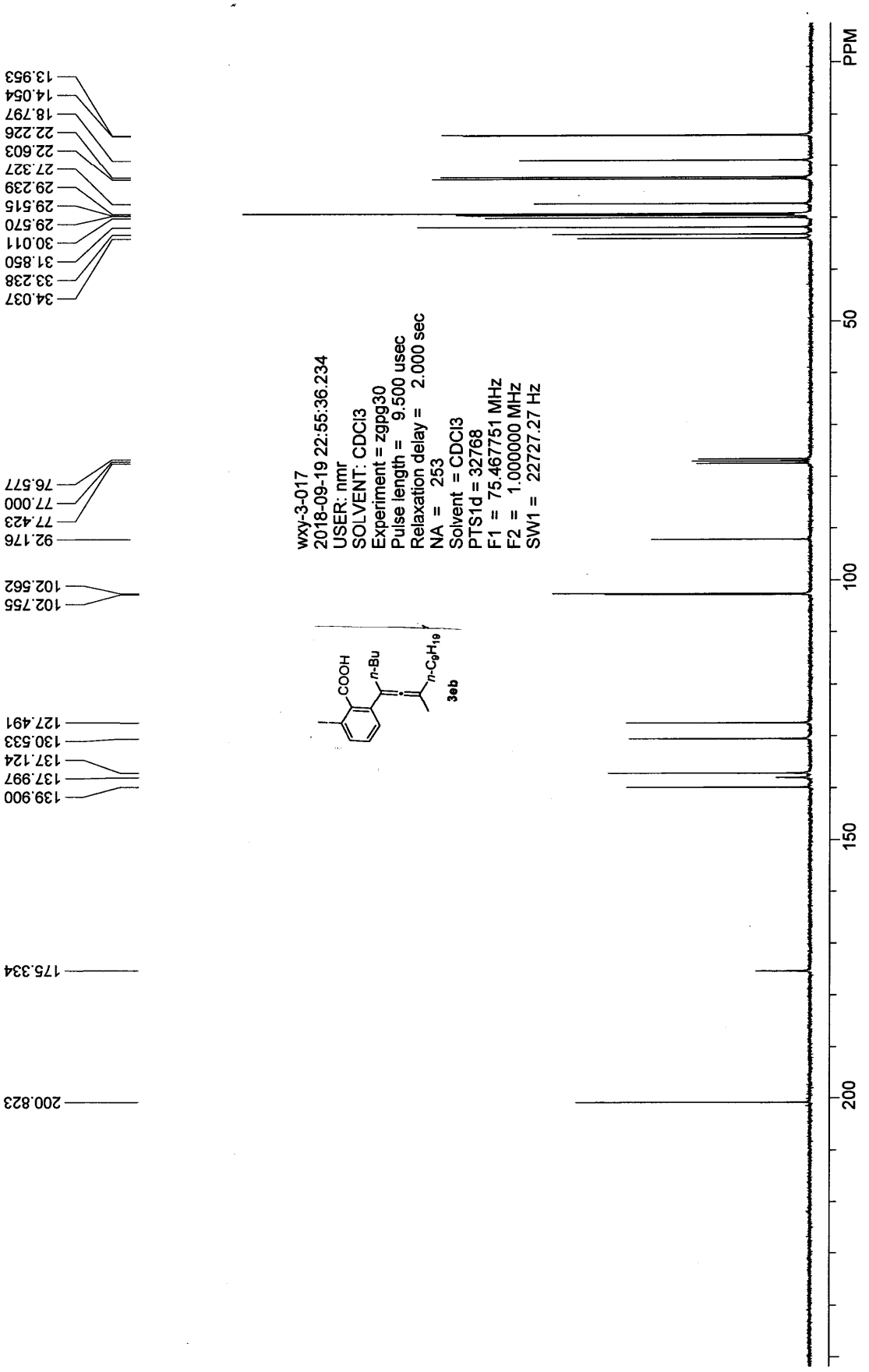

10.639
7.467
7.462
7.441
7.436
7.304
7.299
7.277
7.273
7.252
7.226
7.200
2.340
2.316
2.290
1.751
1.549
1.524
1.499
1.474
1.450
1.426
0.985
0.960
0.936
-0.000
Br
COOH
n-Pr
3dd
wxy-3-022
2018-09-15 19:35:56.859
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
0.87
1.00
0.98
1.12
2.00
5.98
2.11
3.00
12
10
8
6
4
2
0
PPM

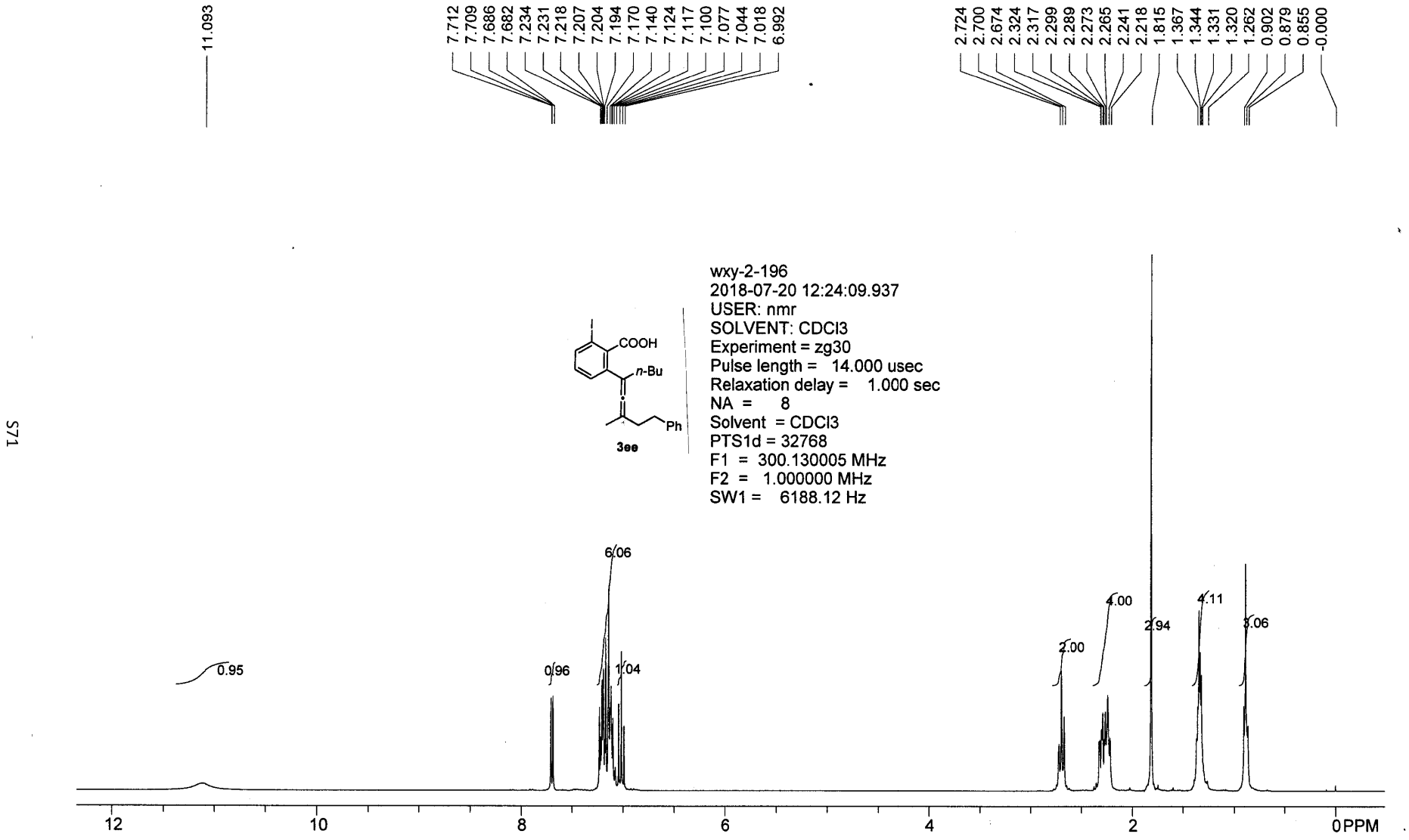

wxy-3-022
2018-09-15 23:26:52.125
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 800
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
3dd
Br
COOH
n-Pr
201.476
173.919
140.065
133.989
130.597
130.478
126.525
119.264
101.064
98.656
77.423
77.000
76.577
35.416
20.985
20.314
13.668
PPM
250
200
150
100
50

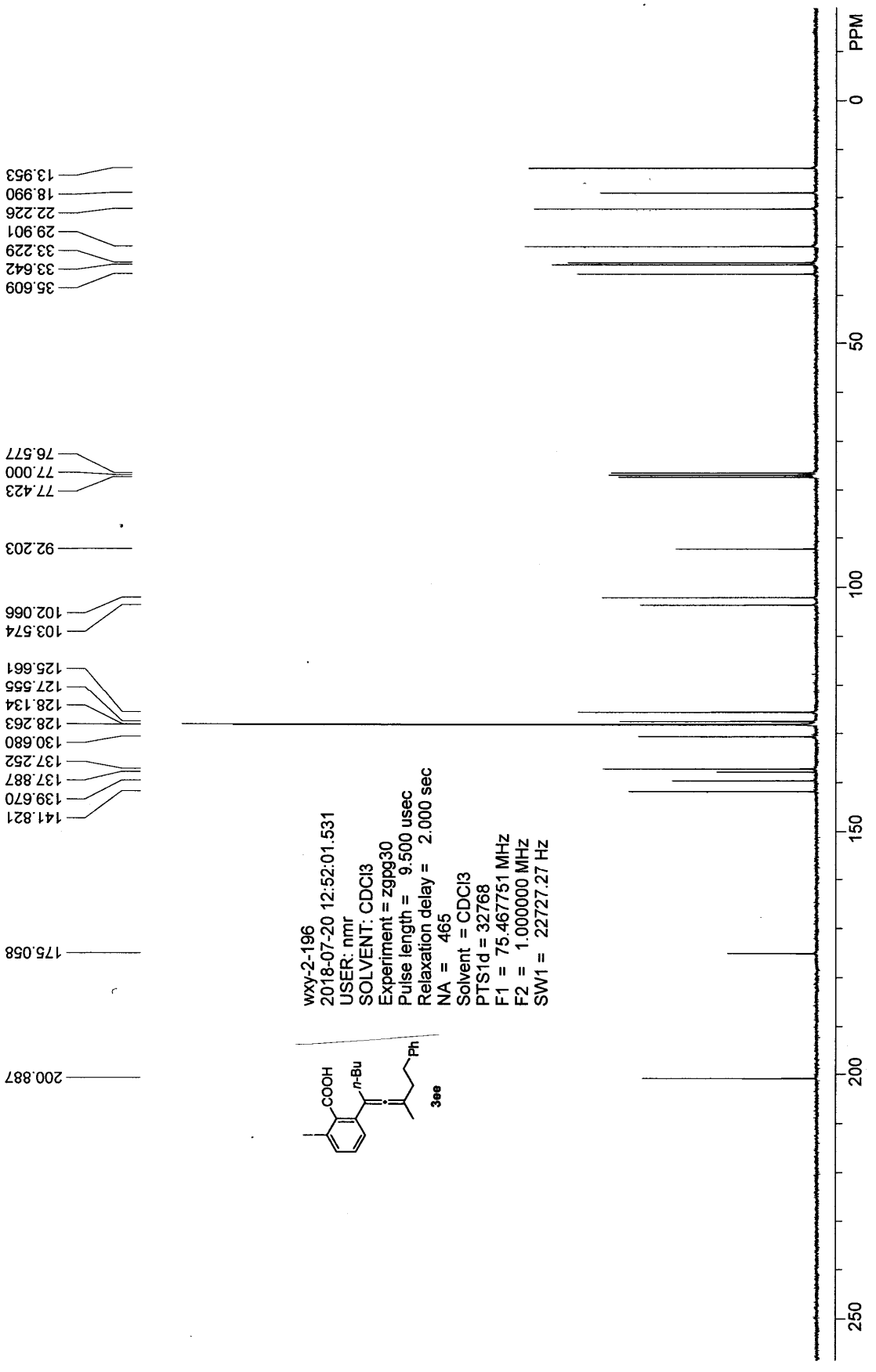

11.620
7.713
7.686
7.299
7.273
7.229
7.068
7.042
7.016
2.345
2.322
2.297
1.752
1.497
1.471
1.423
1.368
1.336
1.313
0.927
0.903
0.880
0.000
COOH
n-Bu
3ea
wxy-2-174
2018-07-11 08:41:03.765
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
1.02
0.99
1.00
1.02
2.00
6.00
4.17
3.04
12
10
8
6
4
2
0
PPM

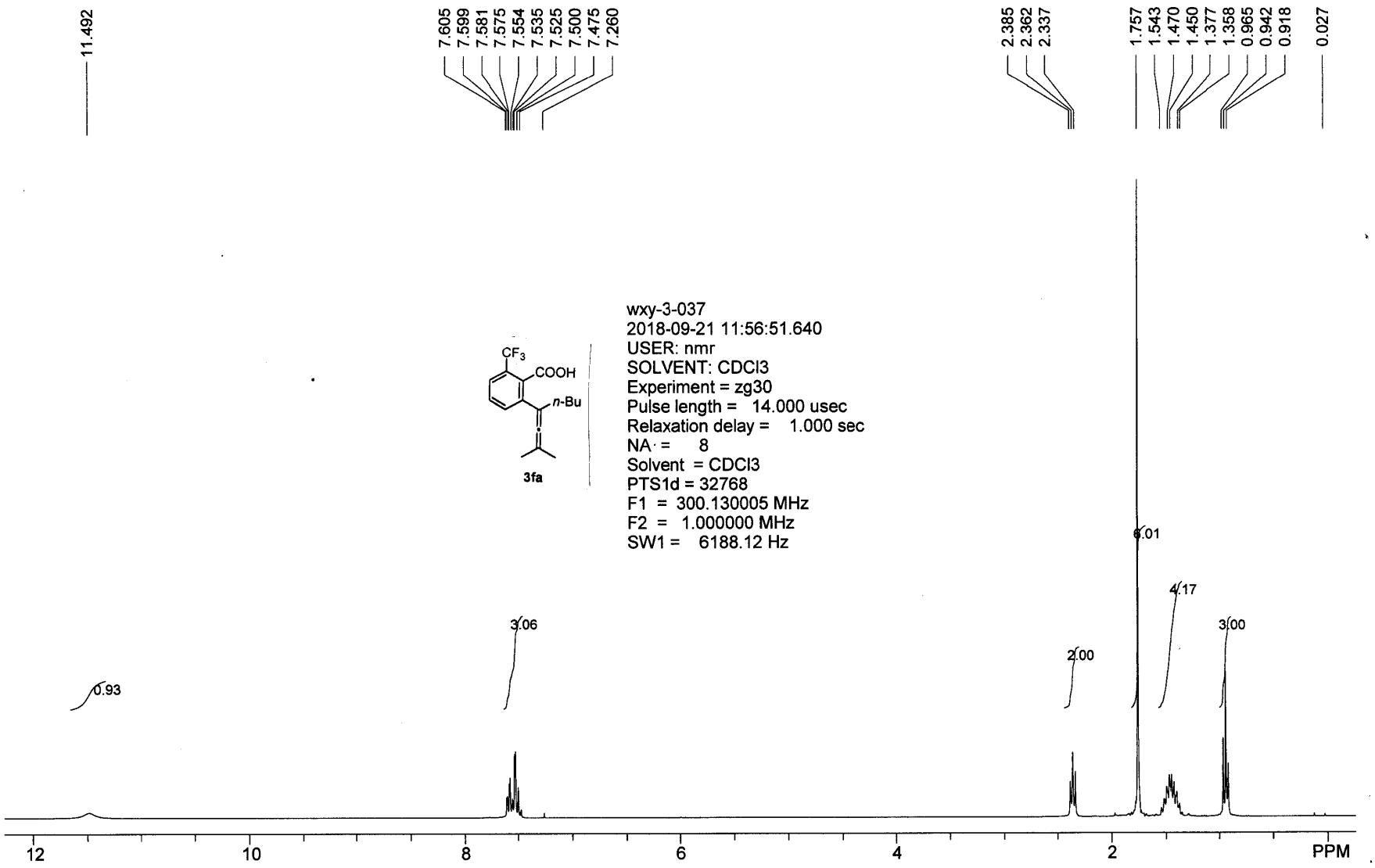

wxy-2-174
2018-07-11 09:07:06.843
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 430
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
COOH
n-Bu
3ea
13.972
20.480
22.143
29.837
33.146
76.577
77.000
77.432
92.121
98.279
101.542
127.463
130.634
137.216
138.006
139.817
175.306
201.135
PPM
250
200
150
100
50
0

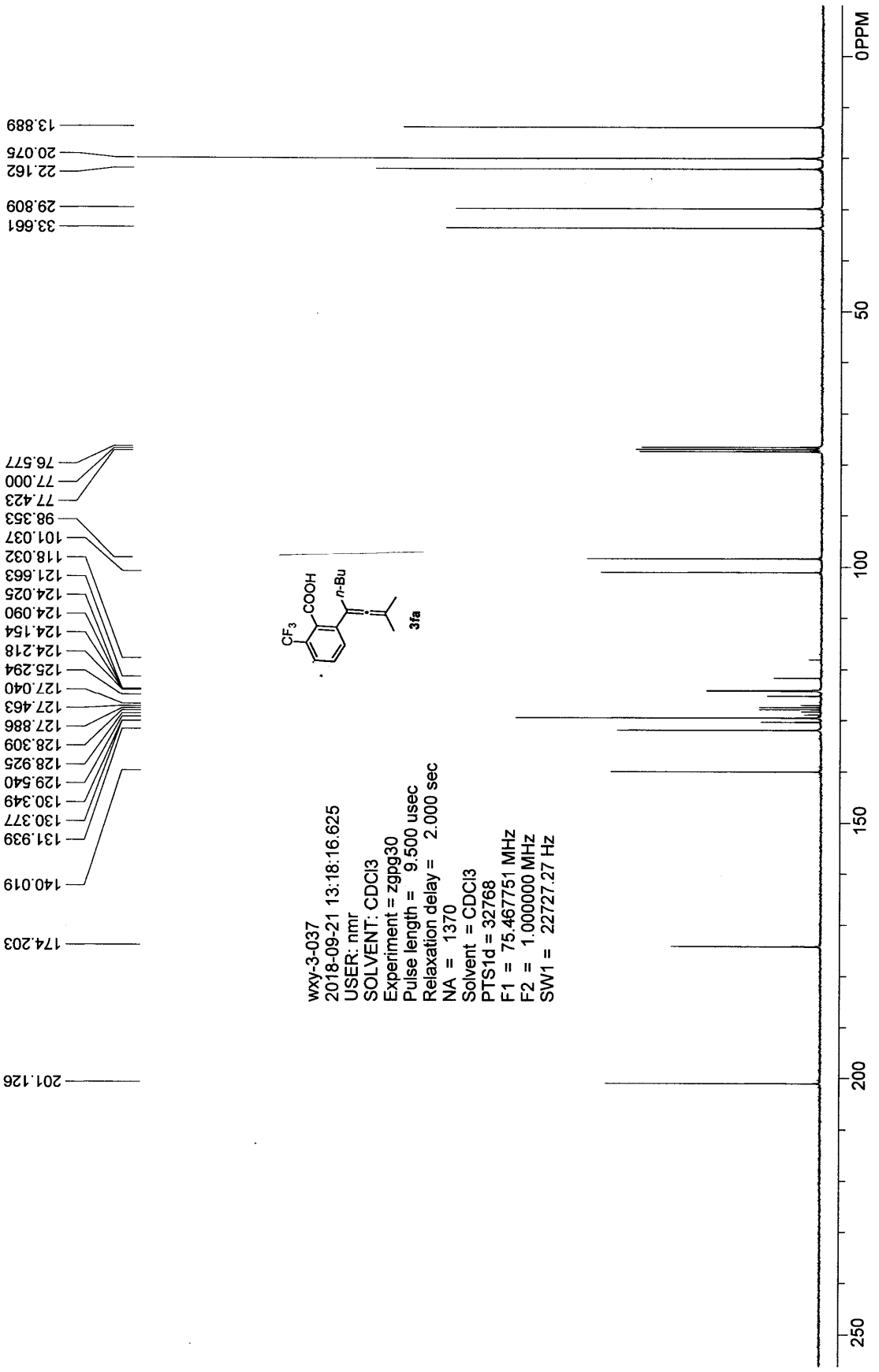

wxy-3-017
2018-09-13 22:39:04.125
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
COOH
n-Bu
n-C9H19
3eb
0PPM
2
4
6
8
10
12
0.95
1.00
1.02
1.01
2.00
2.08
2.98
5.80
6.14
11.867
6.991
7.018
7.044
7.210
7.258
7.283
7.671
7.697
0.843
0.866
0.881
0.904
0.927
1.222
1.345
1.376
1.391
1.413
1.425
1.433
1.453
1.473
1.785
1.958
1.981
2.005
2.312
2.337
2.358

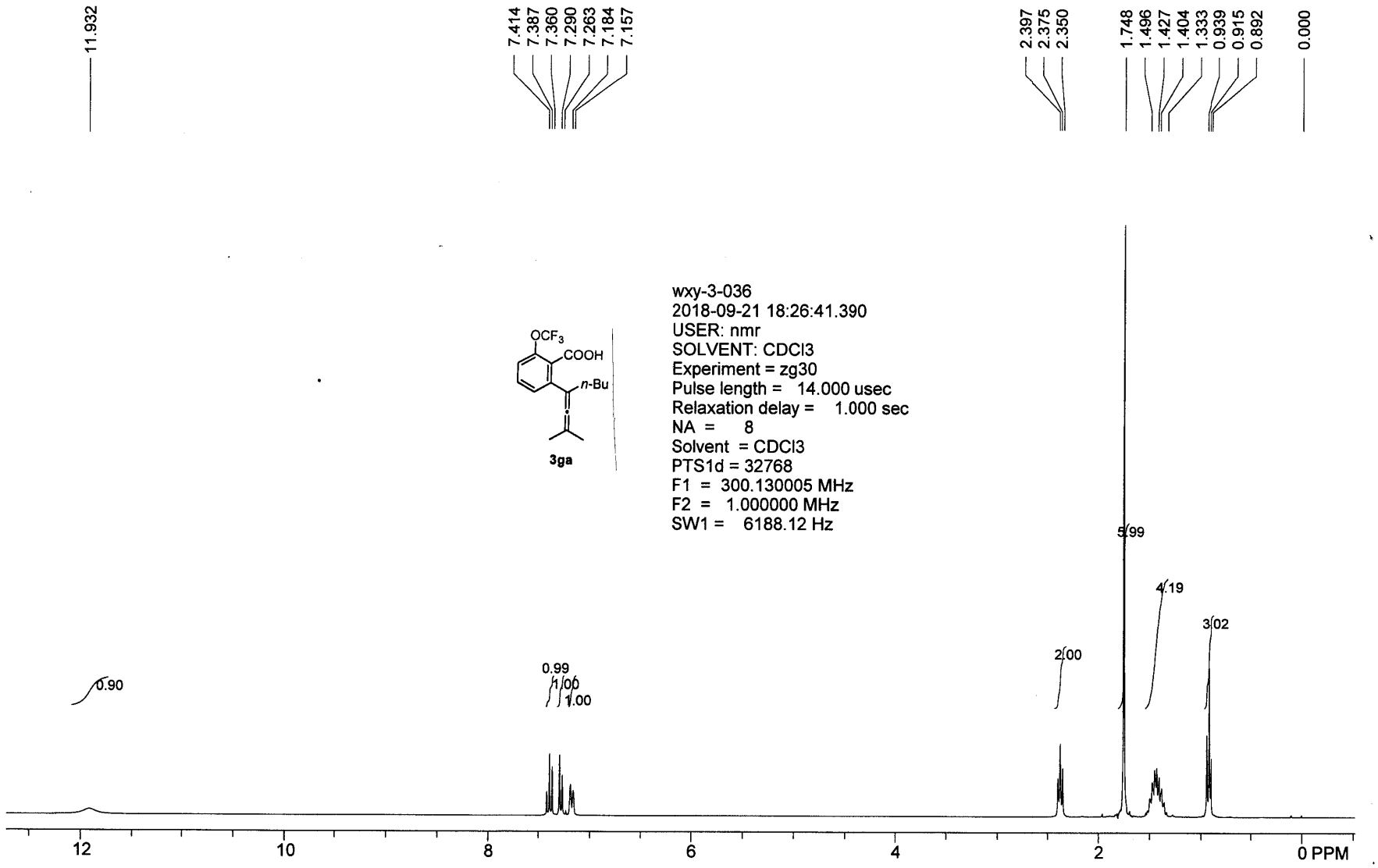

wxy-3-017
2018-09-19 22:55:36.234
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 253
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
COOH
n-Bu
n-C9H19
3eb
PPM
50
100
150
200
13.953
14.054
18.797
22.226
22.603
27.327
29.239
29.515
29.570
30.011
31.850
33.238
34.037
76.577
77.000
77.423
92.176
102.562
102.755
127.491
130.533
137.124
137.997
139.900
175.334
200.823

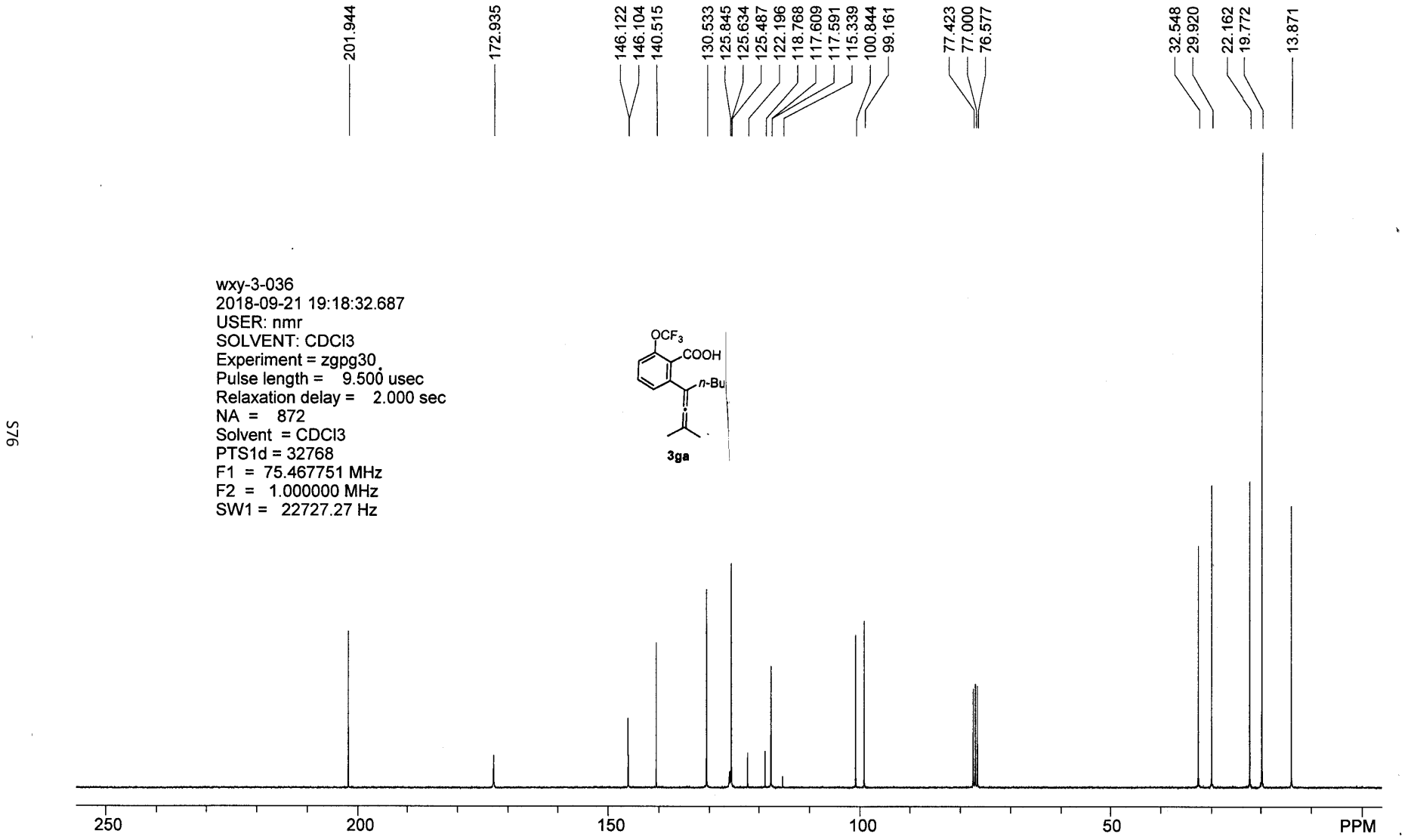

11.093
7.712
7.709
7.686
7.682
7.234
7.231
7.218
7.207
7.204
7.194
7.170
7.140
7.124
7.117
7.100
7.077
7.044
7.018
6.992
2.724
2.700
2.674
2.324
2.317
2.299
2.289
2.273
2.265
2.241
2.218
1.815
1.367
1.344
1.331
1.320
1.262
0.902
0.879
0.855
-0.000
COOH
n-Bu
Ph
3ee
wxy-2-196
2018-07-20 12:24:09.937
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
0.95
0.96
6.06
1.04
2.00
4.00
2.94
4.11
3.06
12
10
8
6
4
2
0PPM

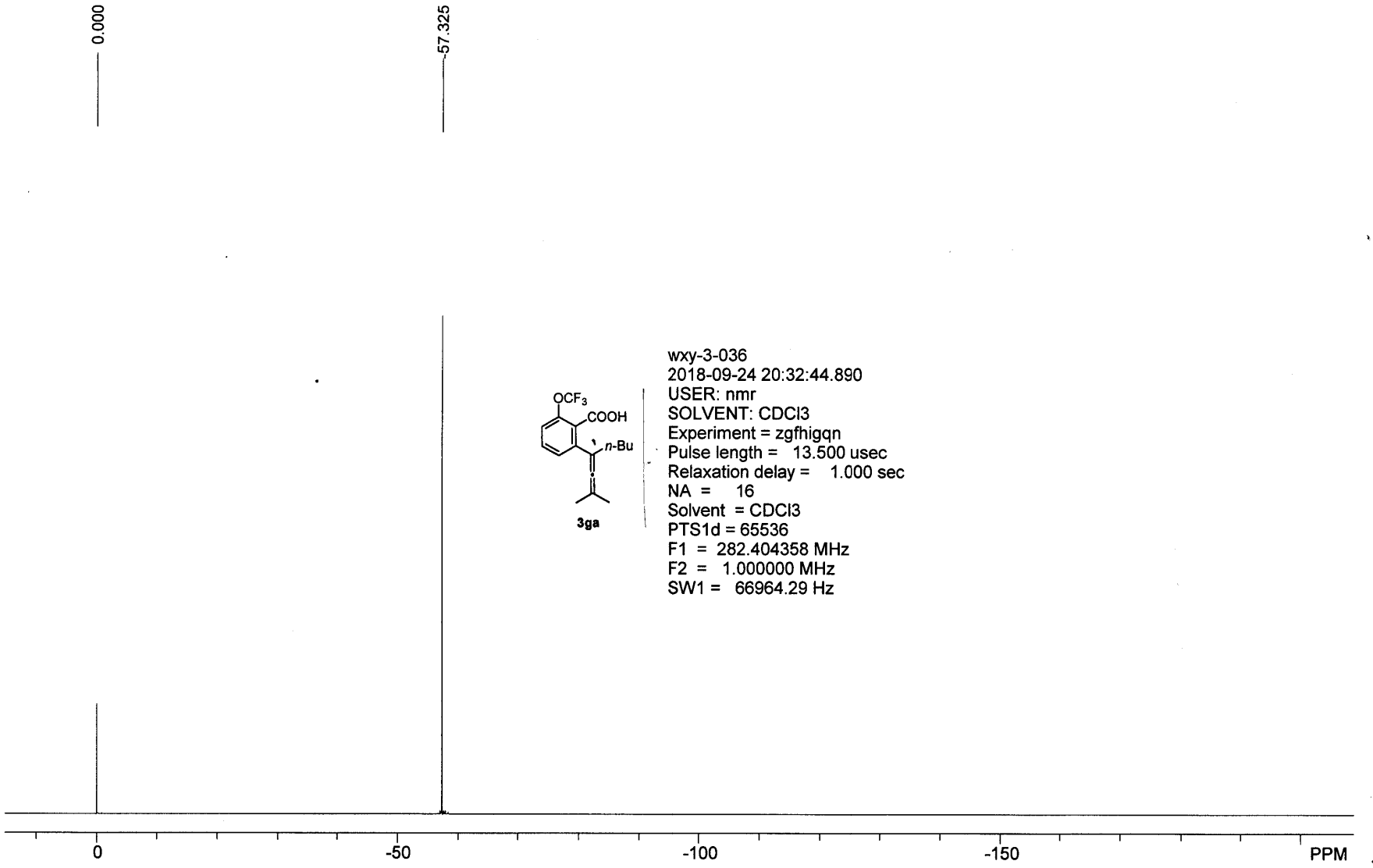

wxy-2-196
2018-07-20 12:52:01.531
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 465
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
COOH
n-Bu
Ph
3ee
200.887
175.058
141.821
139.670
137.887
137.252
130.680
128.263
128.134
127.555
125.661
103.574
102.066
92.203
77.423
77.000
76.577
35.609
33.642
33.229
29.901
22.226
18.990
13.953
250
200
150
100
50
0
PPM

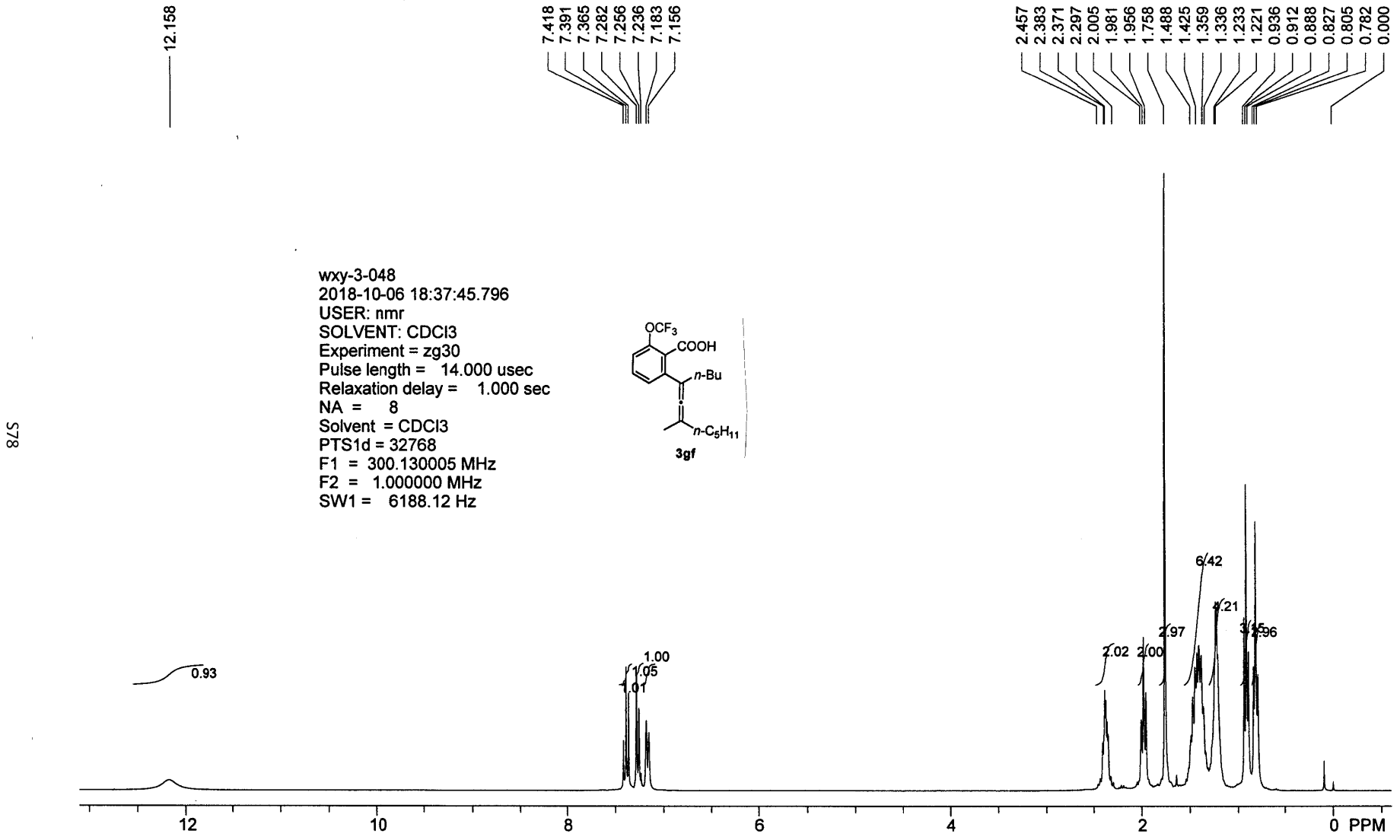

11.492
7.605
7.599
7.581
7.575
7.554
7.535
7.525
7.500
7.475
7.260
2.385
2.362
2.337
1.757
1.543
1.470
1.450
1.377
1.358
0.965
0.942
0.918
0.027
CF3
COOH
n-Bu
3fa
wxy-3-037
2018-09-21 11:56:51.640
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
0.93
3.06
2.00
6.01
4.17
3.00
12
10
8
6
4
2
PPM

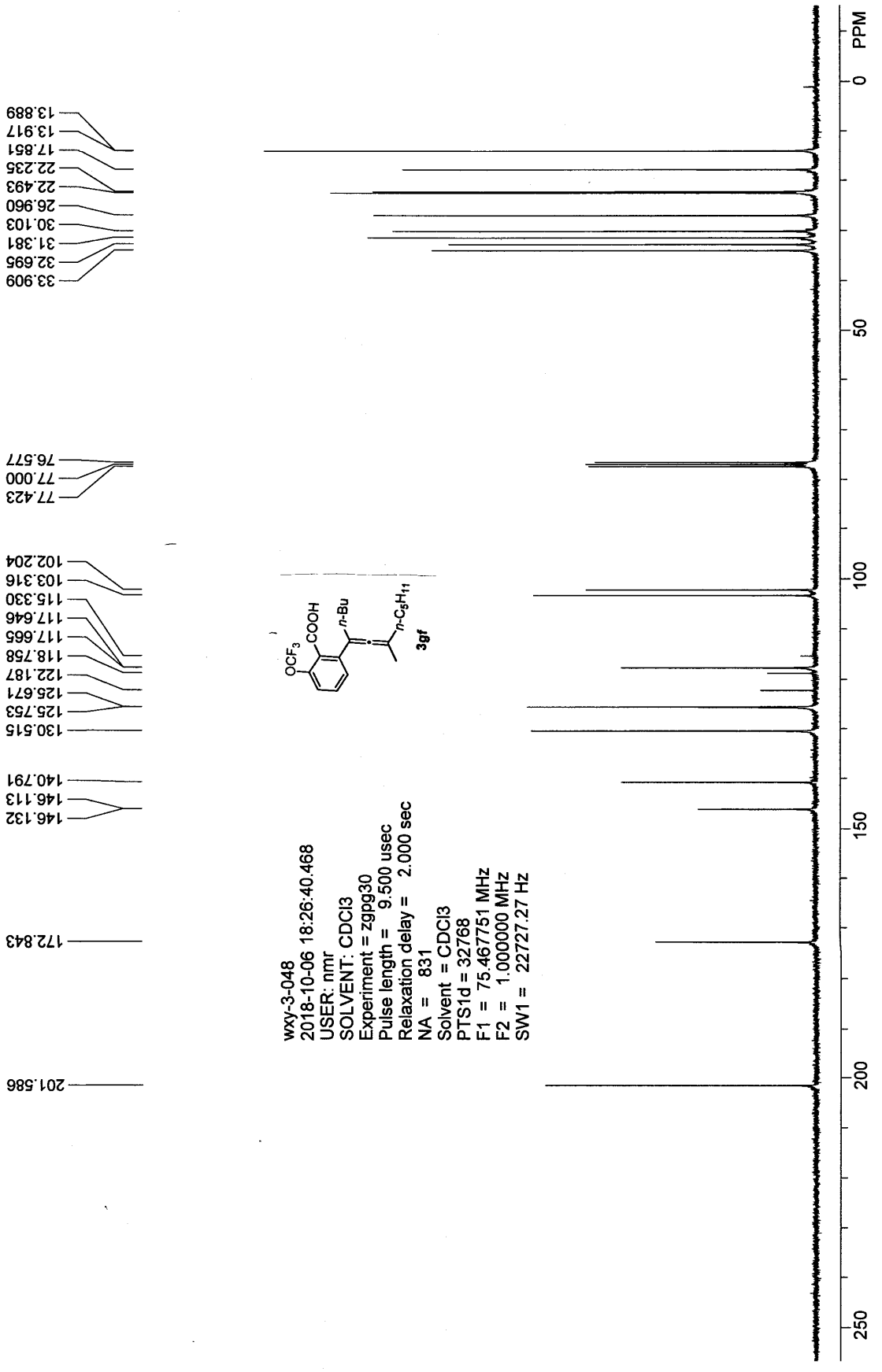

13.889
20.075
22.162
29.809
33.661
76.577
77.000
77.423
98.353
101.037
118.032
121.663
124.025
124.090
124.154
124.218
125.294
127.040
127.463
127.886
128.309
128.925
129.540
130.349
130.377
131.939
140.019
174.203
201.126
wxy-3-037
2018-09-21 13:18:16.625
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 1370
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
CF3
COOH
n-Bu
3fa
0PPM
50
100
150
200
250

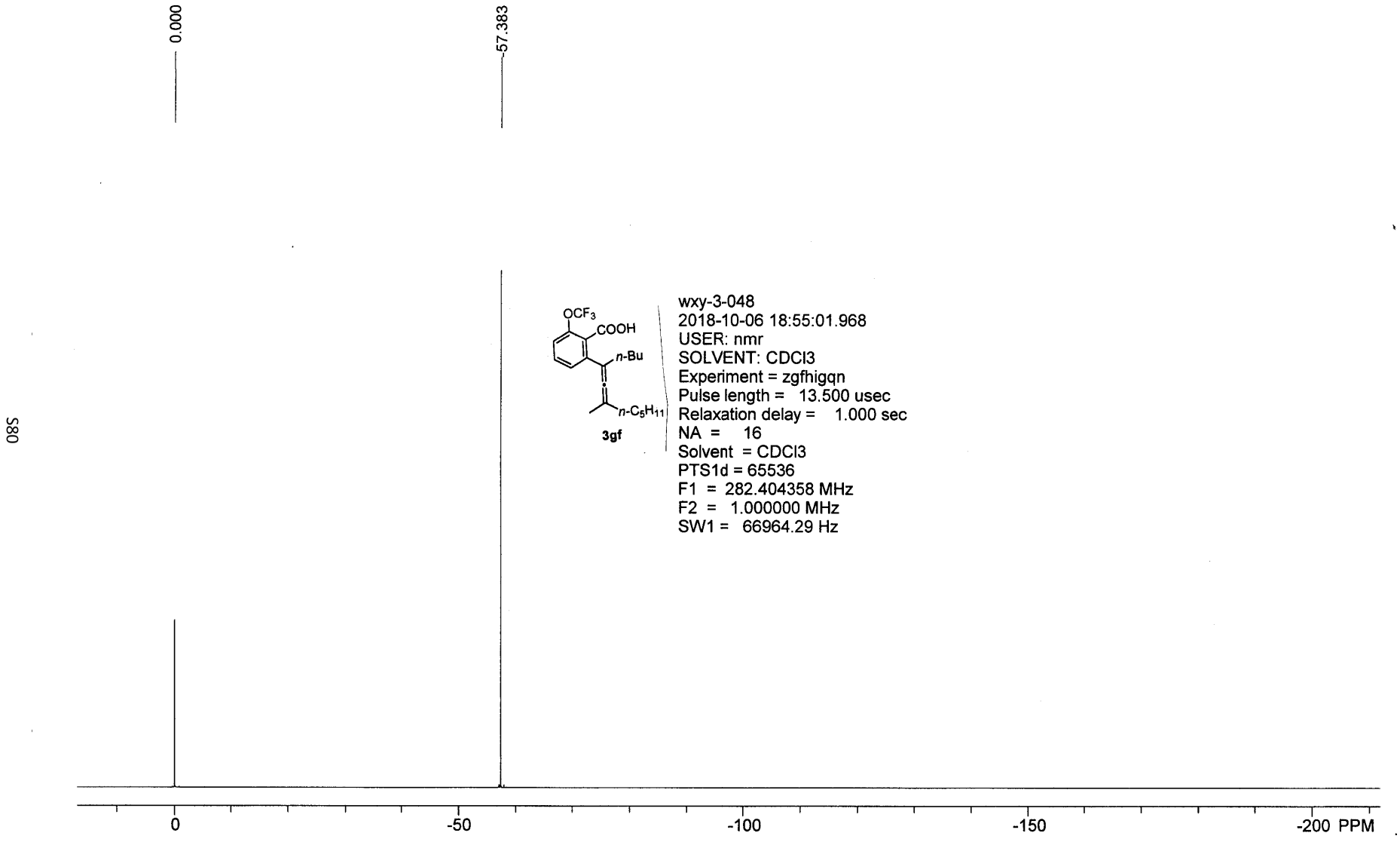

11.932
7.414
7.387
7.360
7.290
7.263
7.184
7.157
2.397
2.375
2.350
1.748
1.496
1.427
1.404
1.333
0.939
0.915
0.892
0.000
OCF3
COOH
n-Bu
3ga
wxy-3-036
2018-09-21 18:26:41.390
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
0.90
0.99
1.00
1.00
2.00
5.99
4.19
3.02
12
10
8
6
4
2
0 PPM

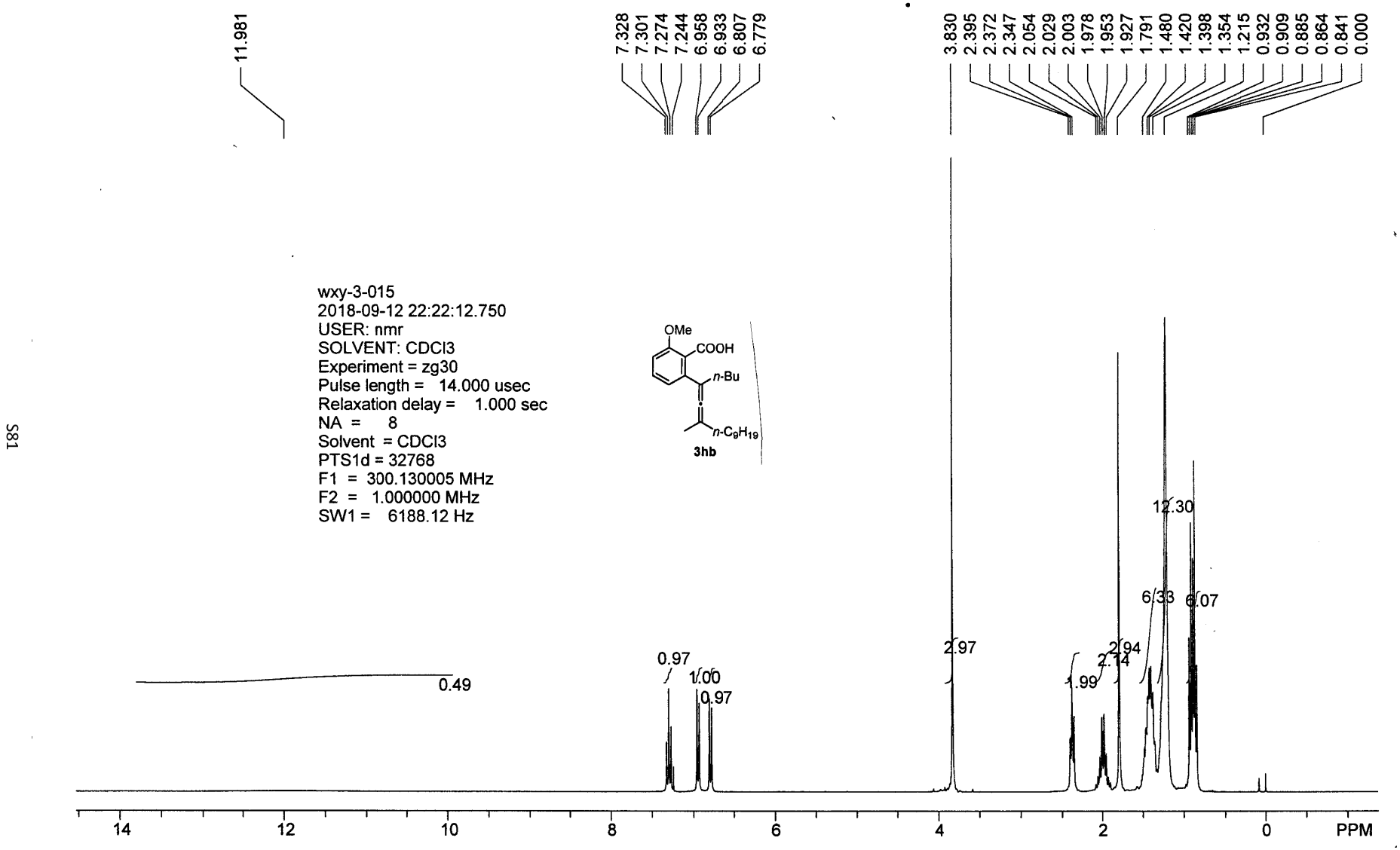

201.944
172.935
146.122
146.104
140.515
130.533
125.845
125.634
125.487
122.196
118.768
117.609
117.591
115.339
100.844
99.161
77.423
77.000
76.577
32.548
29.920
22.162
19.772
13.871
wxy-3-036
2018-09-21 19:18:32.687
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 872
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
OCF3
COOH
n-Bu
3ga
250
200
150
100
50
PPM

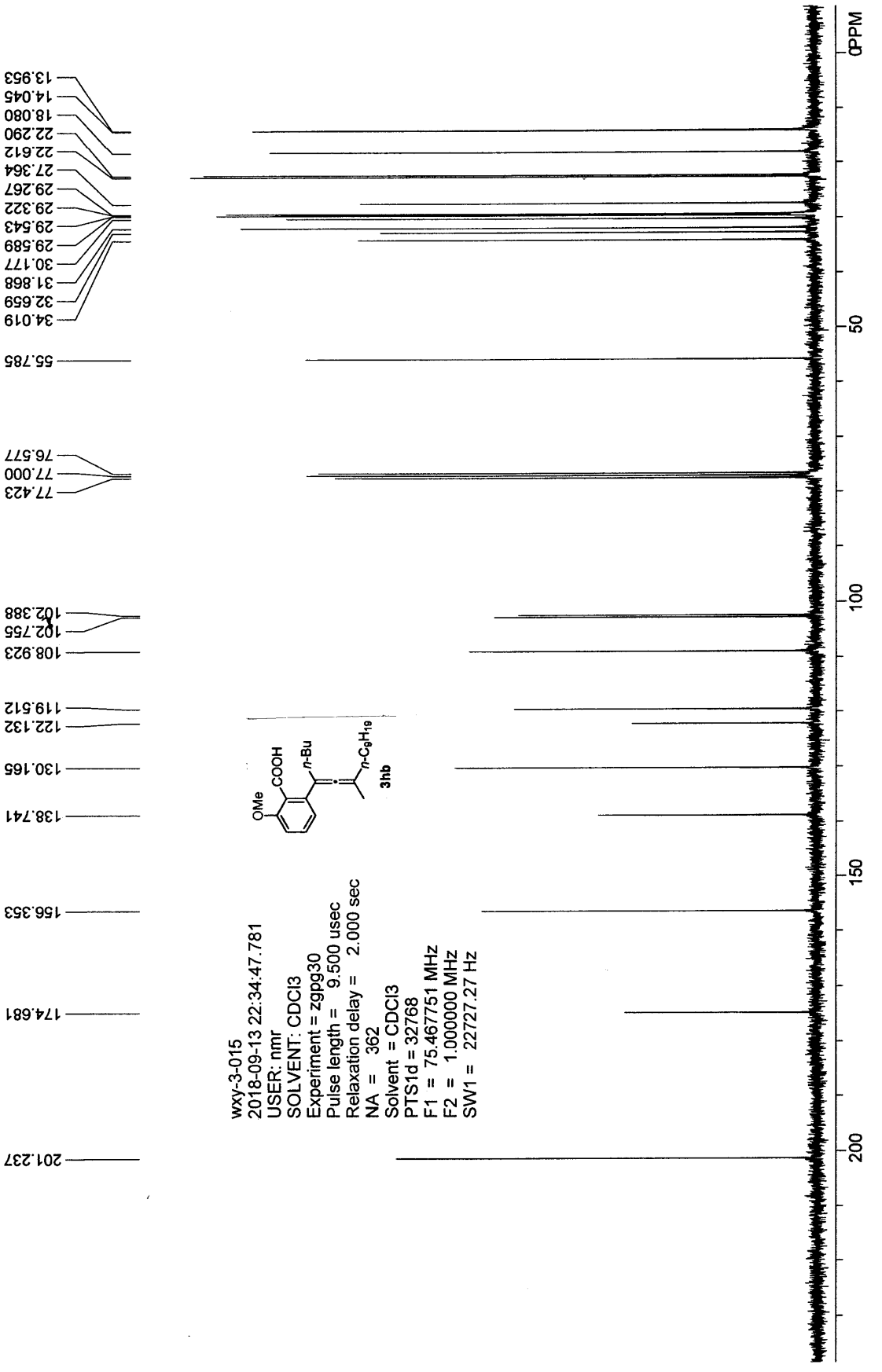

0.000
57.325
OCF3
COOH
n-Bu
3ga
wxy-3-036
2018-09-24 20:32:44.890
USER: nmr
SOLVENT: CDCl3
Experiment = zgfhigqn
Pulse length = 13.500 usec
Relaxation delay = 1.000 sec
NA = 16
Solvent = CDCl3
PTS1d = 65536
F1 = 282.404358 MHz
F2 = 1.000000 MHz
SW1 = 66964.29 Hz
0
-50
-100
-150
PPM

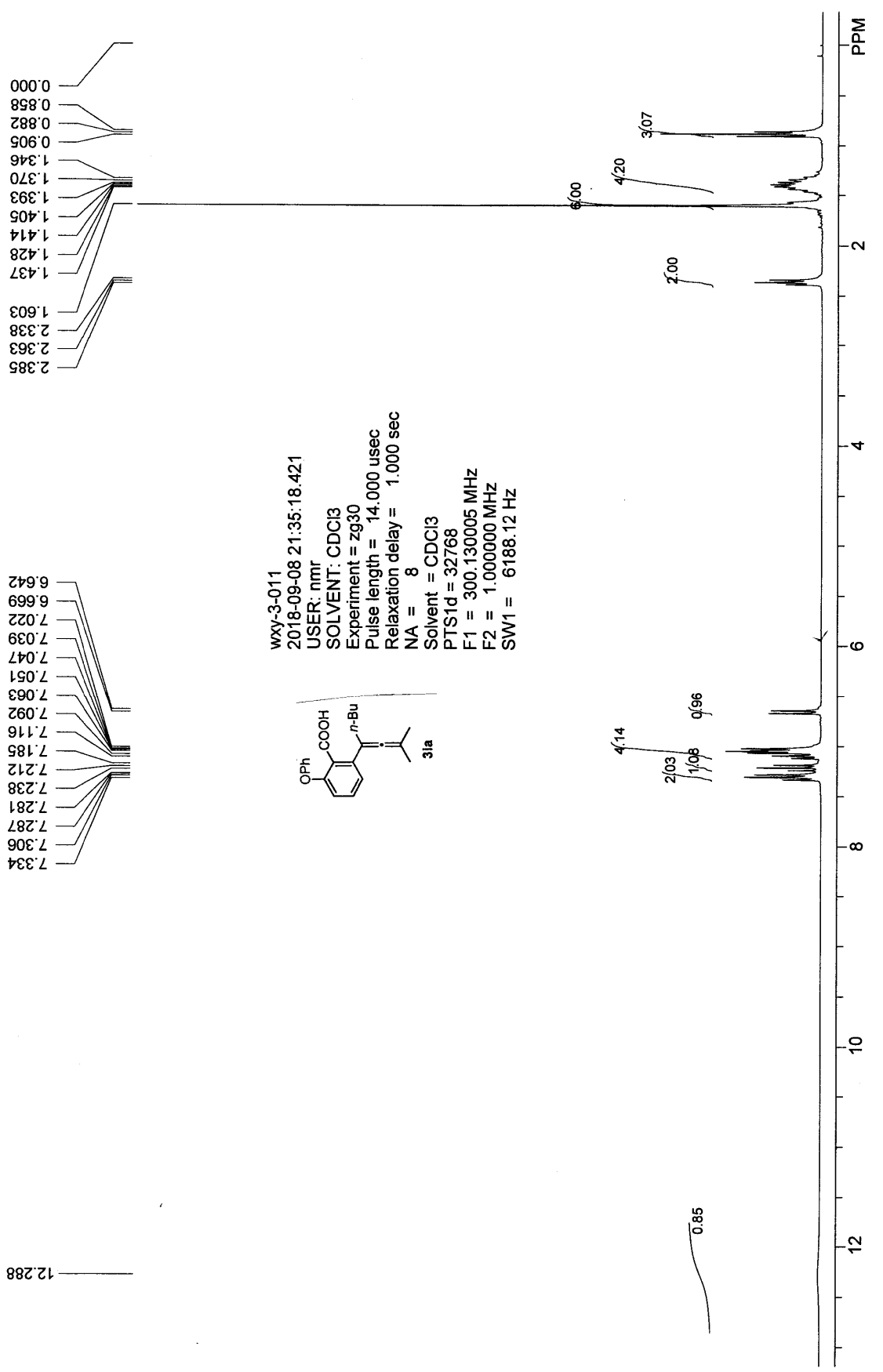

12.158
7.418
7.391
7.365
7.282
7.256
7.236
7.183
7.156
2.457
2.383
2.371
2.297
2.005
1.981
1.956
1.758
1.488
1.425
1.359
1.336
1.233
1.221
0.936
0.912
0.888
0.827
0.805
0.782
0.000
wxy-3-048
2018-10-06 18:37:45.796
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
OCF3
COOH
n-Bu
n-C5H11
3gf
0.93
1.00
1.05
1.01
2.02
2.00
2.97
6.42
4.21
3.15
2.96
12
10
8
6
4
2
0 PPM

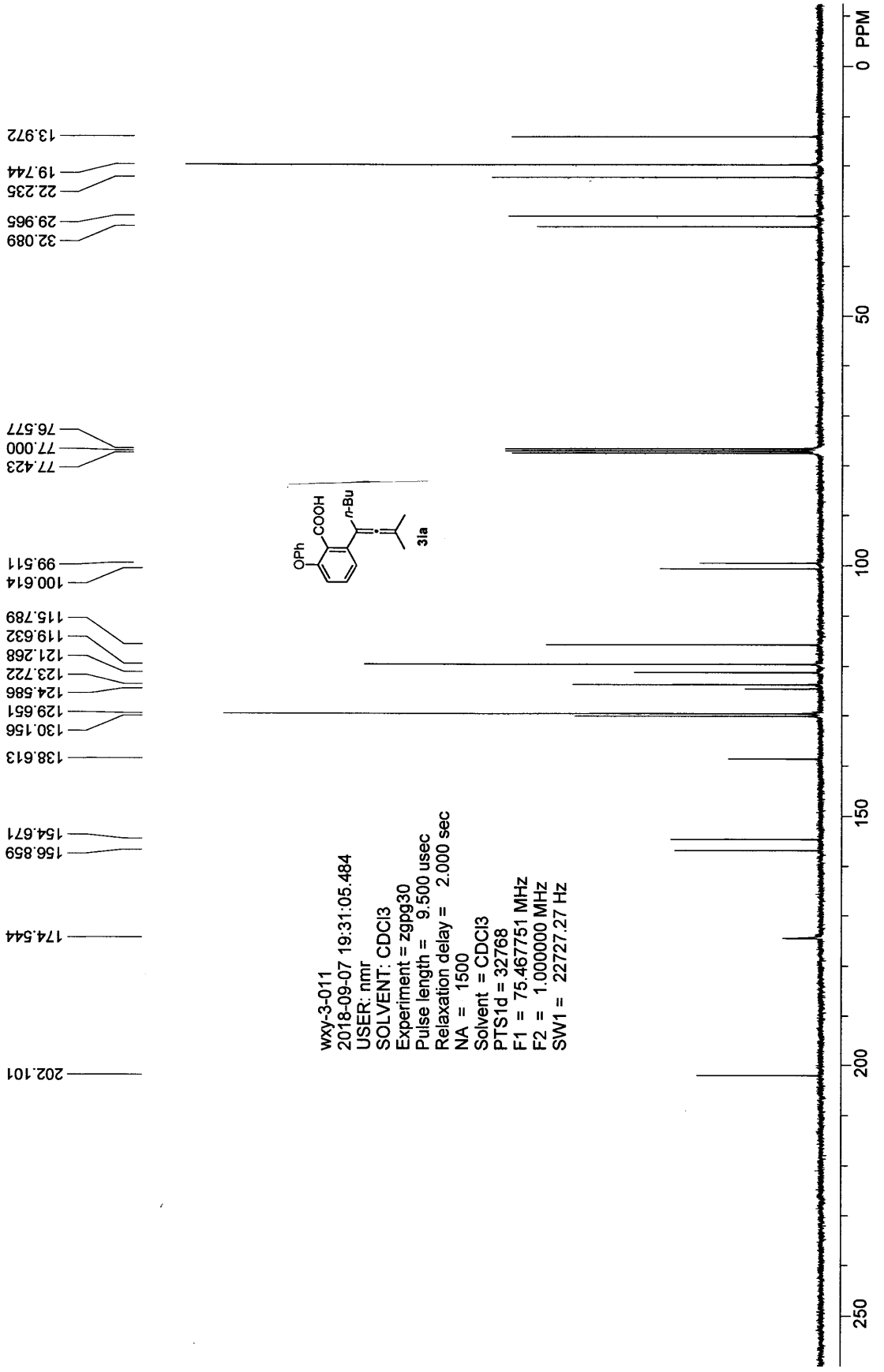

13.889
13.917
17.851
22.235
22.493
26.960
30.103
31.381
32.695
33.909
76.577
77.000
77.423
102.204
103.316
115.330
117.646
117.665
118.758
122.187
125.671
125.753
130.515
140.791
146.113
146.132
172.843
201.586
wxy-3-048
2018-10-06 18:26:40.468
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 831
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
OCF3
COOH
n-Bu
n-C5H11
3gf
250
200
150
100
50
0
PPM

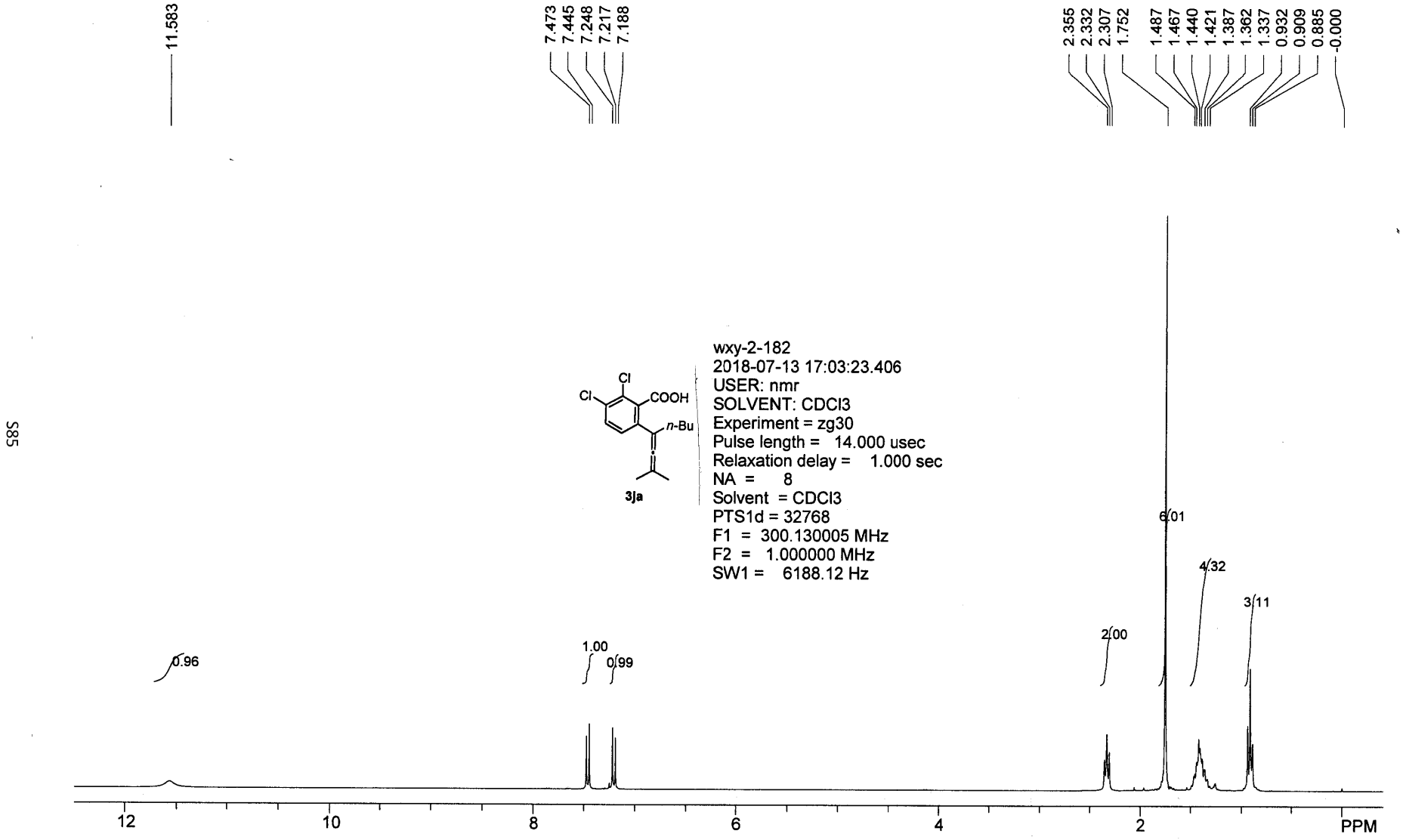

0.000
57.383
OCF3
COOH
n-Bu
n-C5H11
3gf
wxy-3-048
2018-10-06 18:55:01.968
USER: nmr
SOLVENT: CDCl3
Experiment = zgfhigqn
Pulse length = 13.500 usec
Relaxation delay = 1.000 sec
NA = 16
Solvent = CDCl3
PTS1d = 65536
F1 = 282.404358 MHz
F2 = 1.000000 MHz
SW1 = 66964.29 Hz
0
-50
-100
-150
-200 PPM

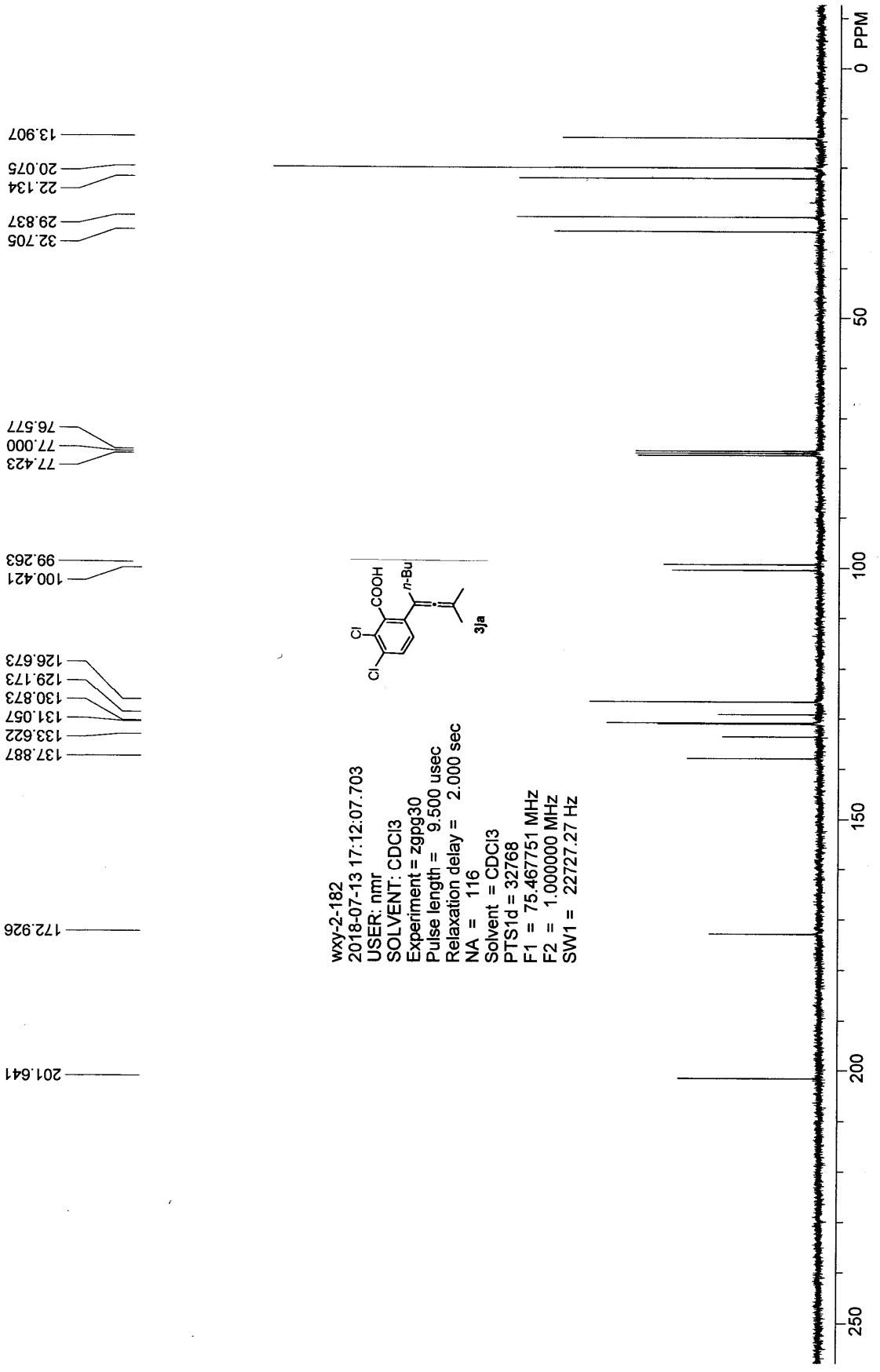

11.981
7.328
7.301
7.274
7.244
6.958
6.933
6.807
6.779
3.830
2.395
2.372
2.347
2.054
2.029
2.003
1.978
1.953
1.927
1.791
1.480
1.420
1.398
1.354
1.215
0.932
0.909
0.885
0.864
0.841
0.000
wxy-3-015
2018-09-12 22:22:12.750
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
OMe
COOH
n-Bu
n-C6H19
3hb
0.49
0.97
1.00
0.97
2.97
1.99
2.14
2.94
6.33
12.30
6.07
14
12
10
8
6
4
2
0
PPM

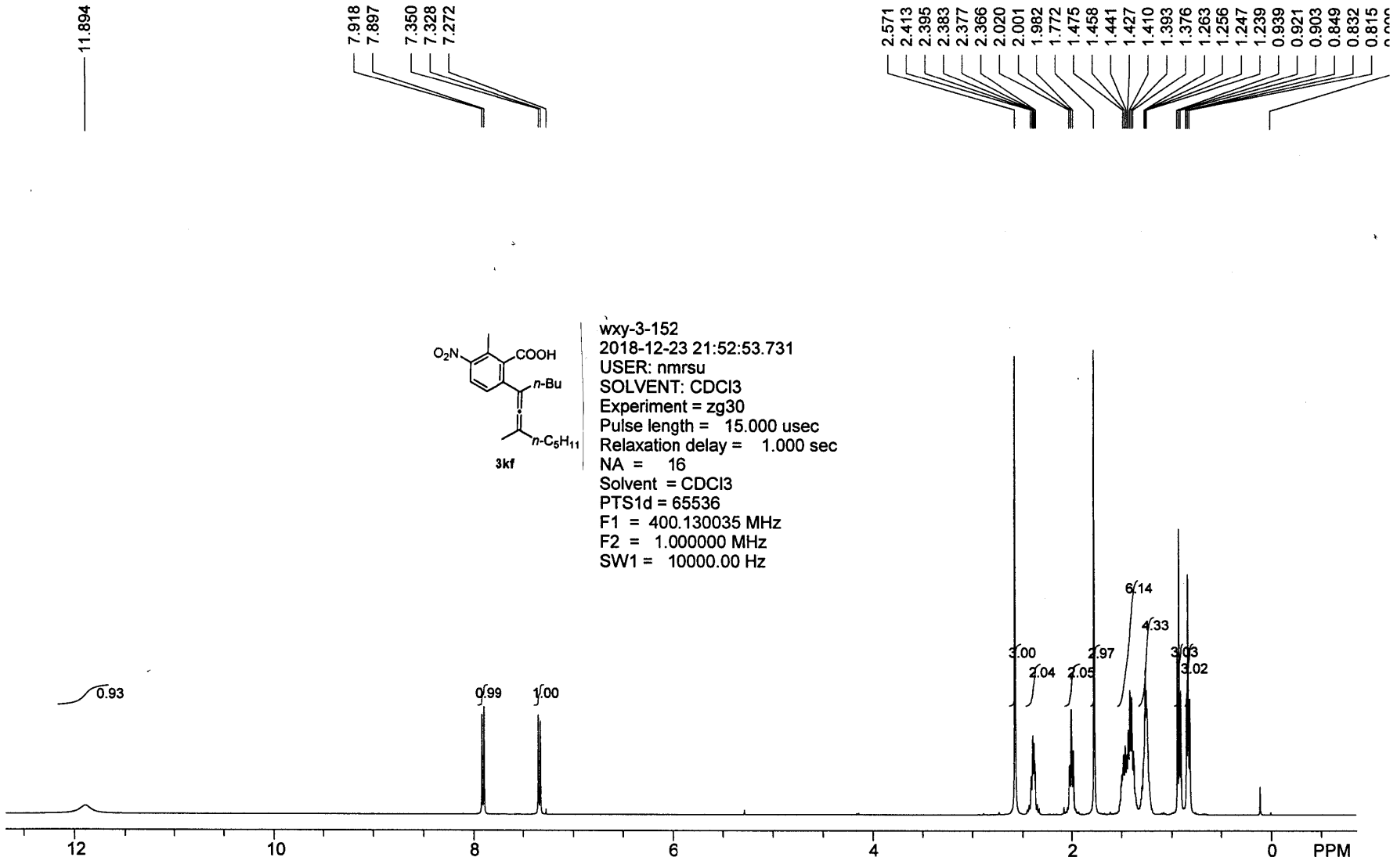

wxy-3-015
2018-09-13 22:34:47.781
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 362
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
OMe
COOH
n-Bu
n-C6H13
3hb
13.953
14.045
18.080
22.290
22.612
27.364
29.267
29.322
29.543
29.589
30.177
31.868
32.659
34.019
55.785
76.577
77.000
77.423
102.388
102.755
108.923
119.512
122.132
130.165
138.741
156.353
174.681
201.237
PPM
50
100
150
200

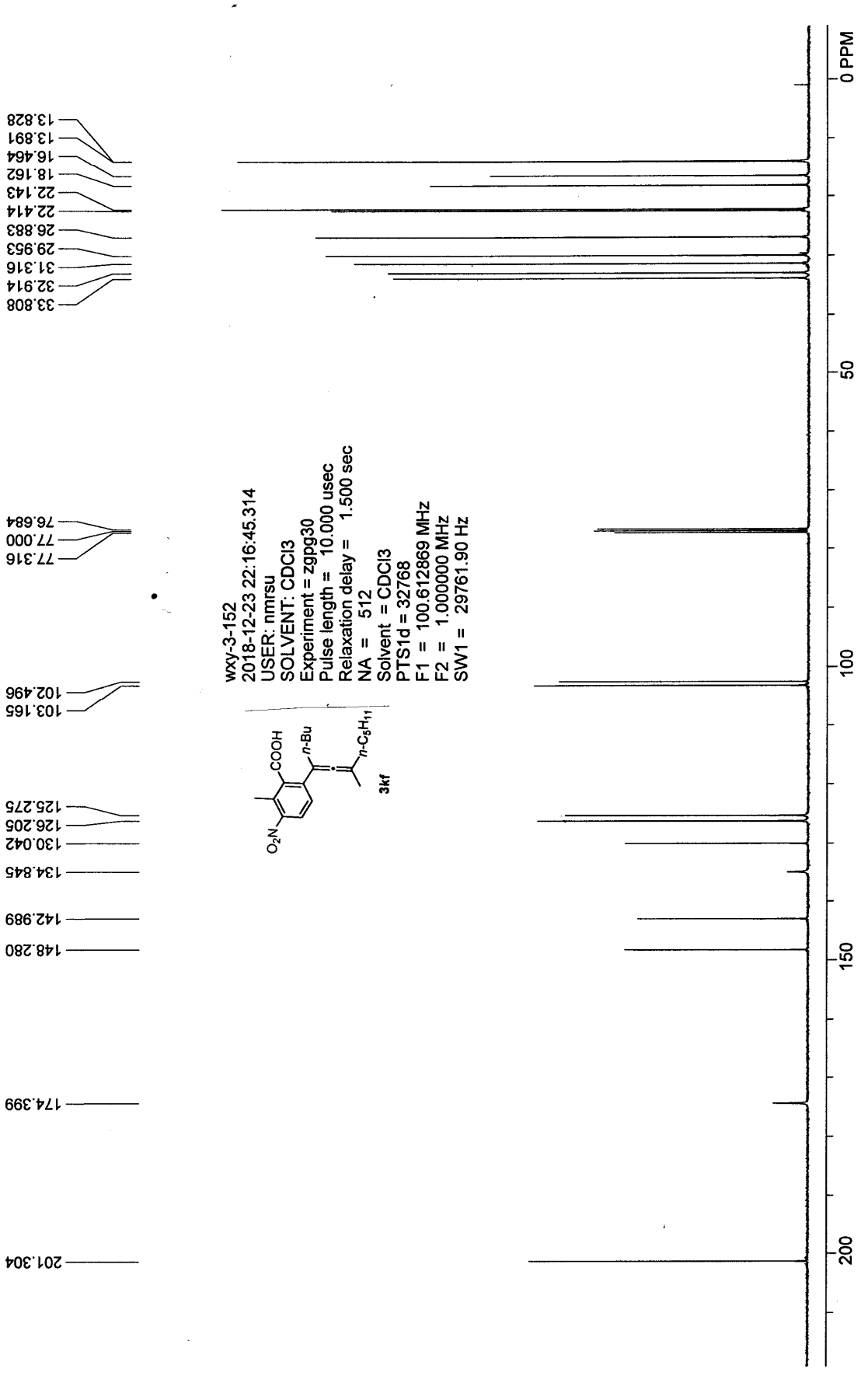

wxy-3-011
2018-09-08 21:35:18.421
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
OPh
COOH
n-Bu
3la
PPM

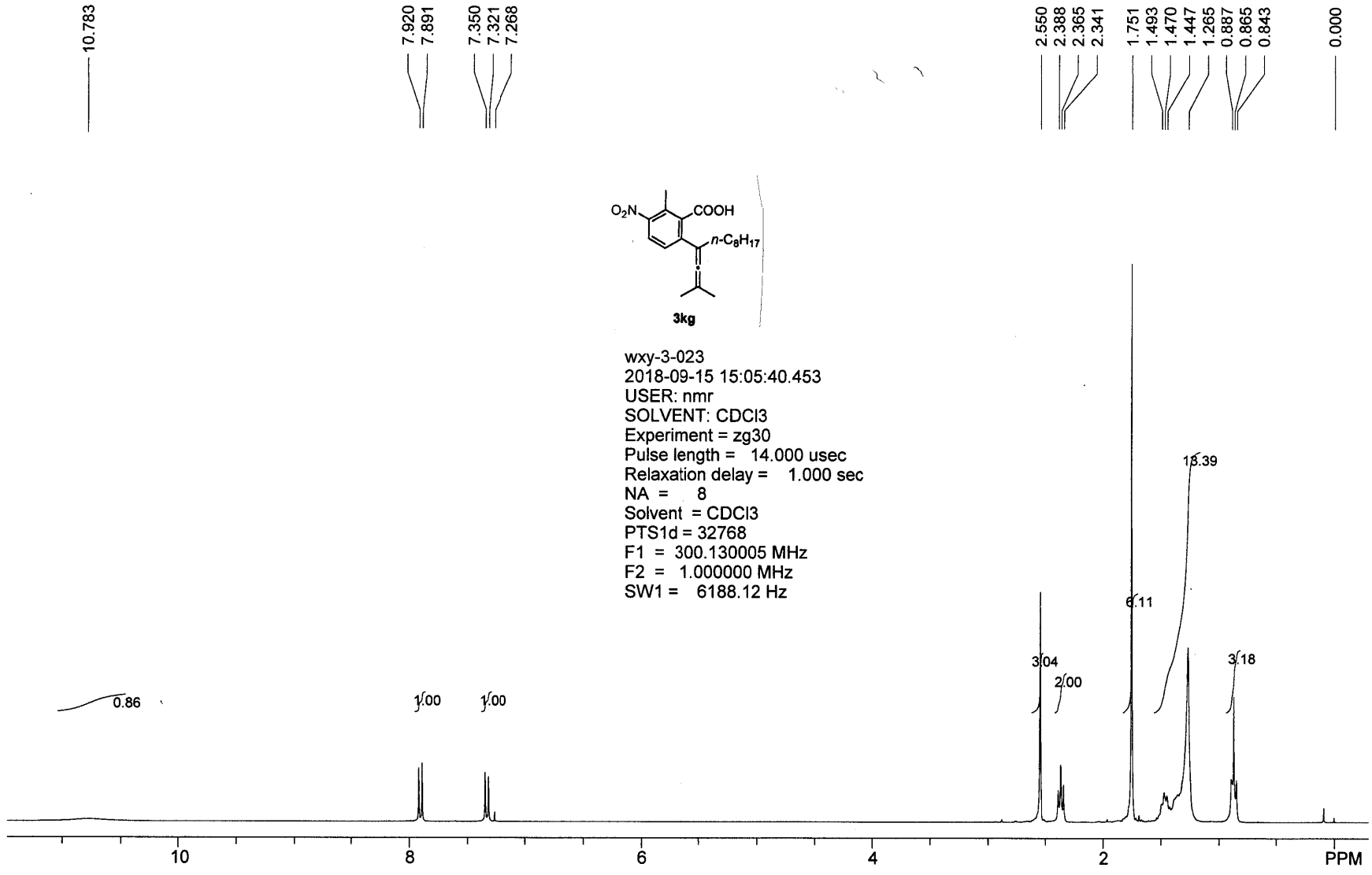

13.972
19.744
22.235
29.965
32.089
76.577
77.000
77.423
99.511
100.614
115.789
119.632
121.268
123.722
124.586
129.651
130.156
138.613
154.671
156.859
174.544
202.101
wxy-3-011
2018-09-07 19:31:05.484
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 1500
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
OPh
COOH
n-Bu
3la
0 PPM
50
100
150
200
250

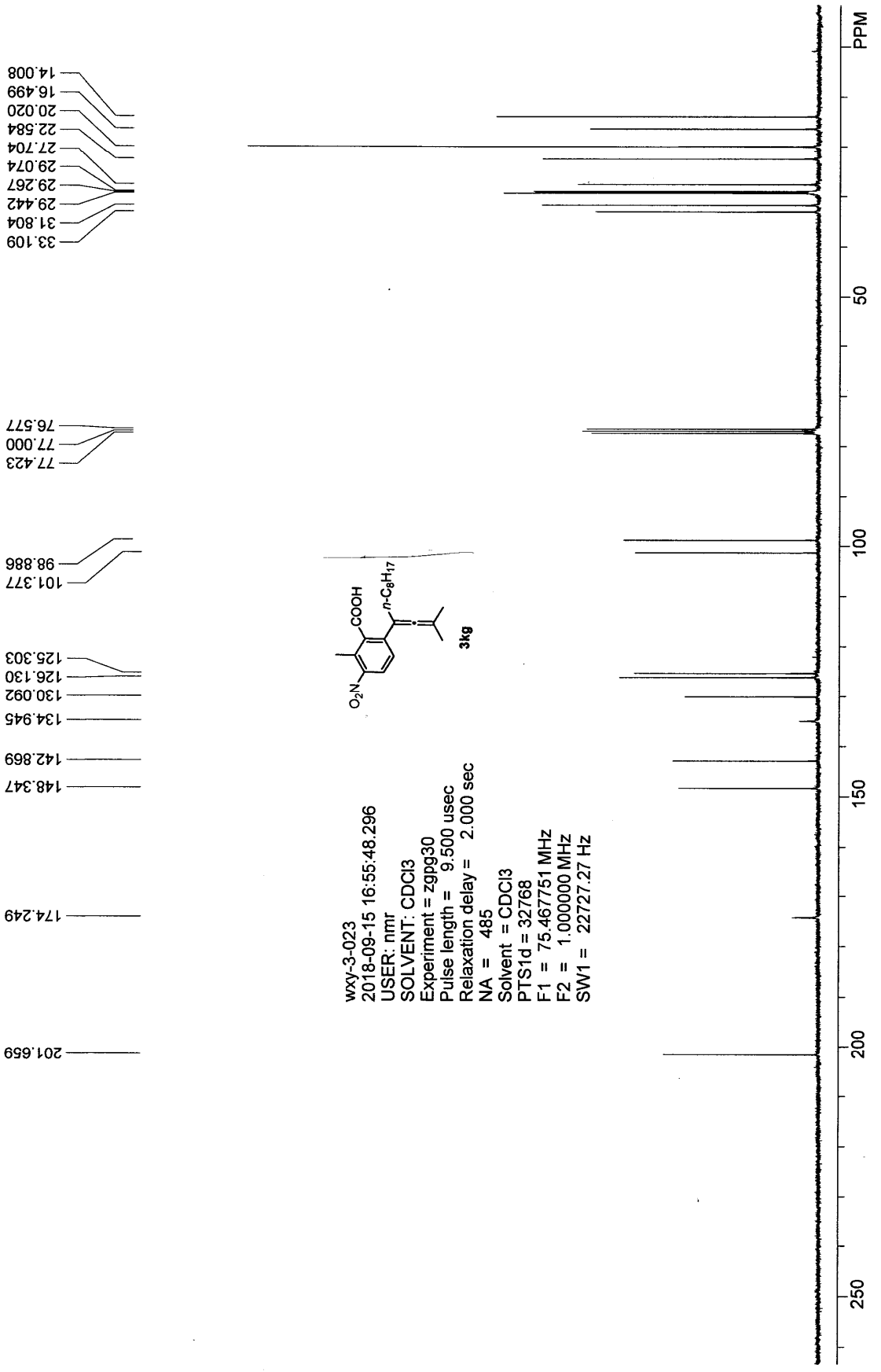

11.583
7.473
7.445
7.248
7.217
7.188
2.355
2.332
2.307
1.752
1.487
1.467
1.440
1.421
1.387
1.362
1.337
0.932
0.909
0.885
-0.000
Cl
Cl
COOH
n-Bu
3ja
wxy-2-182
2018-07-13 17:03:23.406
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
0.96
1.00
0.99
2.00
6.01
4.32
3.11
12
10
8
6
4
2
PPM

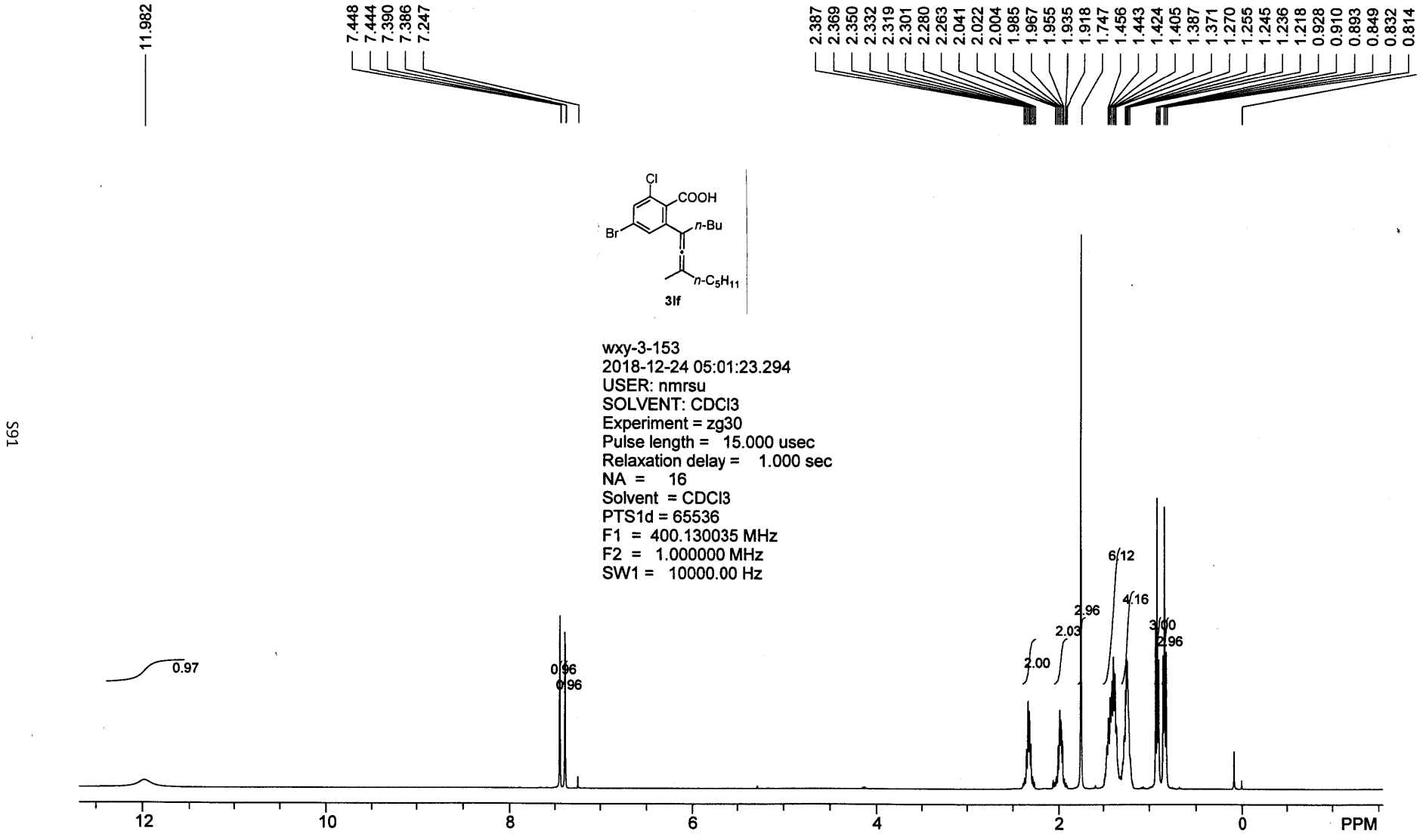

wxy-2-182
2018-07-13 17:12:07.703
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 116
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
Cl
COOH
n-Bu
Cl
3ja
13.907
20.075
22.134
29.837
32.705
76.577
77.000
77.423
99.263
100.421
126.673
129.173
130.873
131.057
133.622
137.887
172.926
201.641
0 PPM
50
100
150
200
250

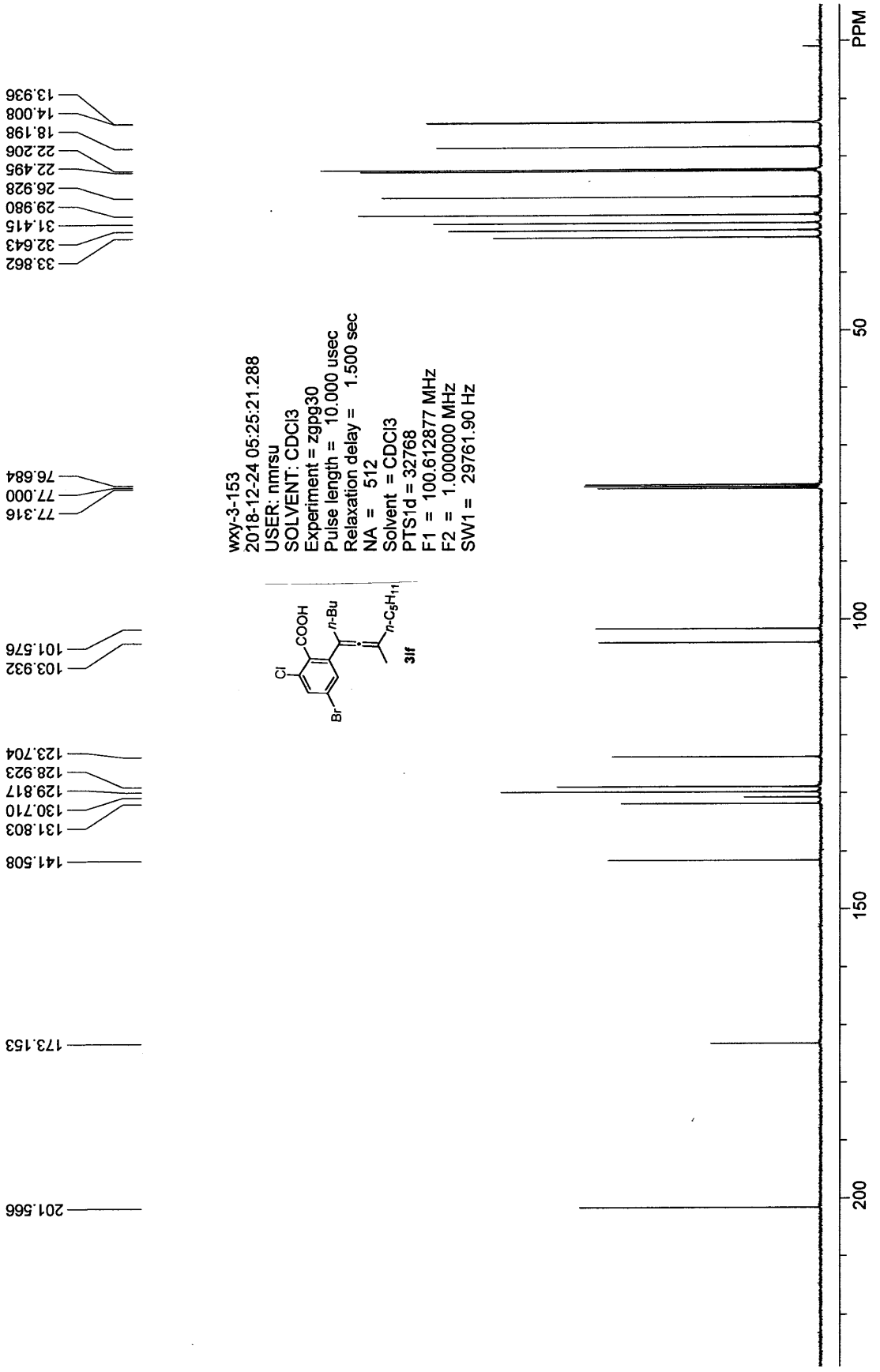

11.894
7.918
7.897
7.350
7.328
7.272
2.571
2.413
2.395
2.383
2.377
2.366
2.020
2.001
1.982
1.772
1.475
1.458
1.441
1.427
1.410
1.393
1.376
1.263
1.256
1.247
1.239
0.939
0.921
0.903
0.849
0.832
0.815
O2N
COOH
n-Bu
n-C5H11
3kf
wxy-3-152
2018-12-23 21:52:53.731
USER: nmrsu
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 15.000 usec
Relaxation delay = 1.000 sec
NA = 16
Solvent = CDCl3
PTS1d = 65536
F1 = 400.130035 MHz
F2 = 1.000000 MHz
SW1 = 10000.00 Hz
0.93
0.99
1.00
3.00
2.04
2.05
2.97
6.14
4.33
3.03
3.02
12
10
8
6
4
2
0
PPM

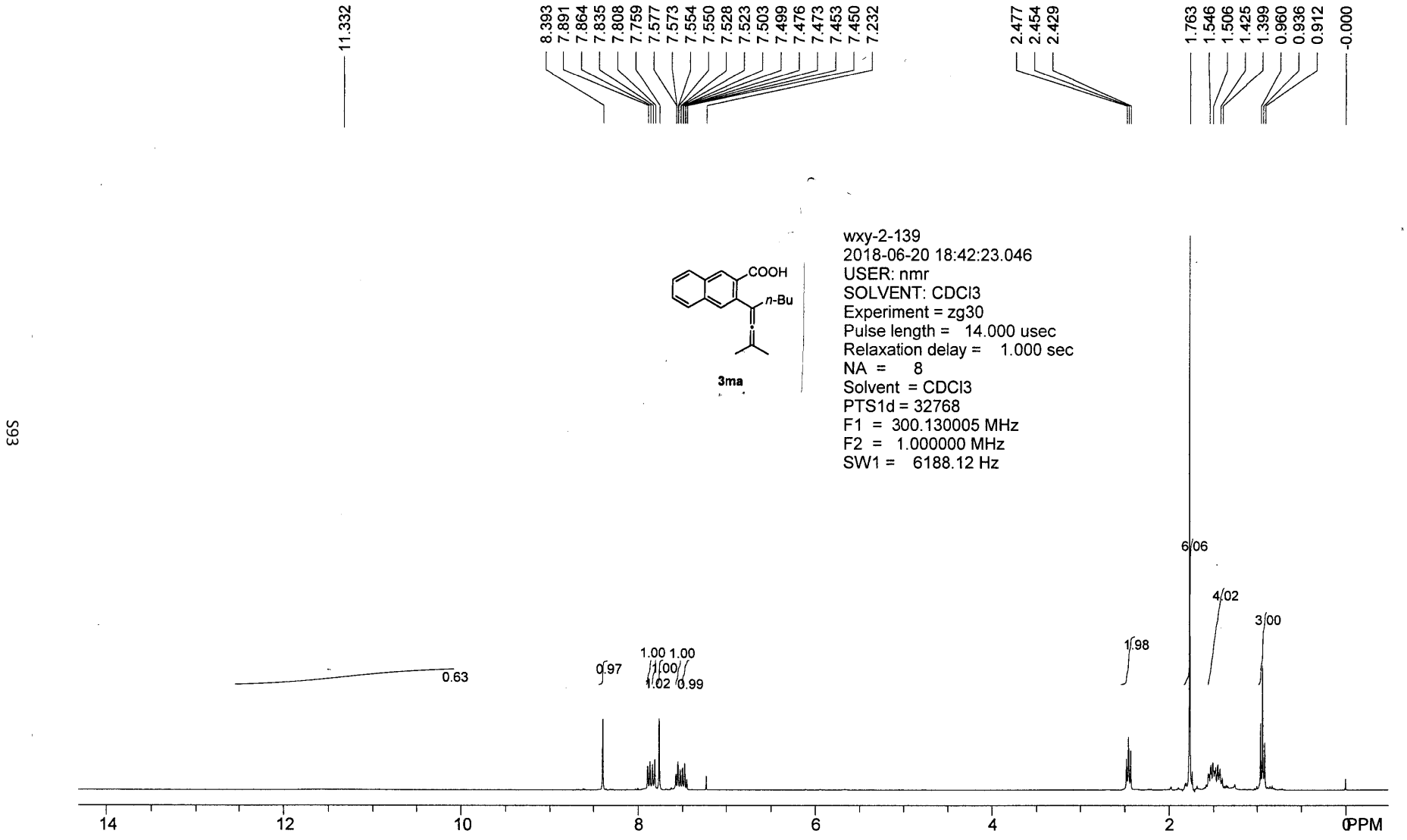

wxy-3-152
2018-12-23 22:16:45.314
USER: nmrsu
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 10.000 usec
Relaxation delay = 1.500 sec
NA = 512
Solvent = CDCl3
PTS1d = 32768
F1 = 100.612869 MHz
F2 = 1.000000 MHz
SW1 = 29761.90 Hz
COOH
n-Bu
n-C5H11
O2N
3kf
201.304
174.399
148.280
142.989
134.845
130.042
126.205
125.275
103.165
102.496
77.316
77.000
76.684
33.808
32.914
31.316
29.953
26.883
22.414
22.143
18.162
16.464
13.891
13.828
200
150
100
50
0 PPM

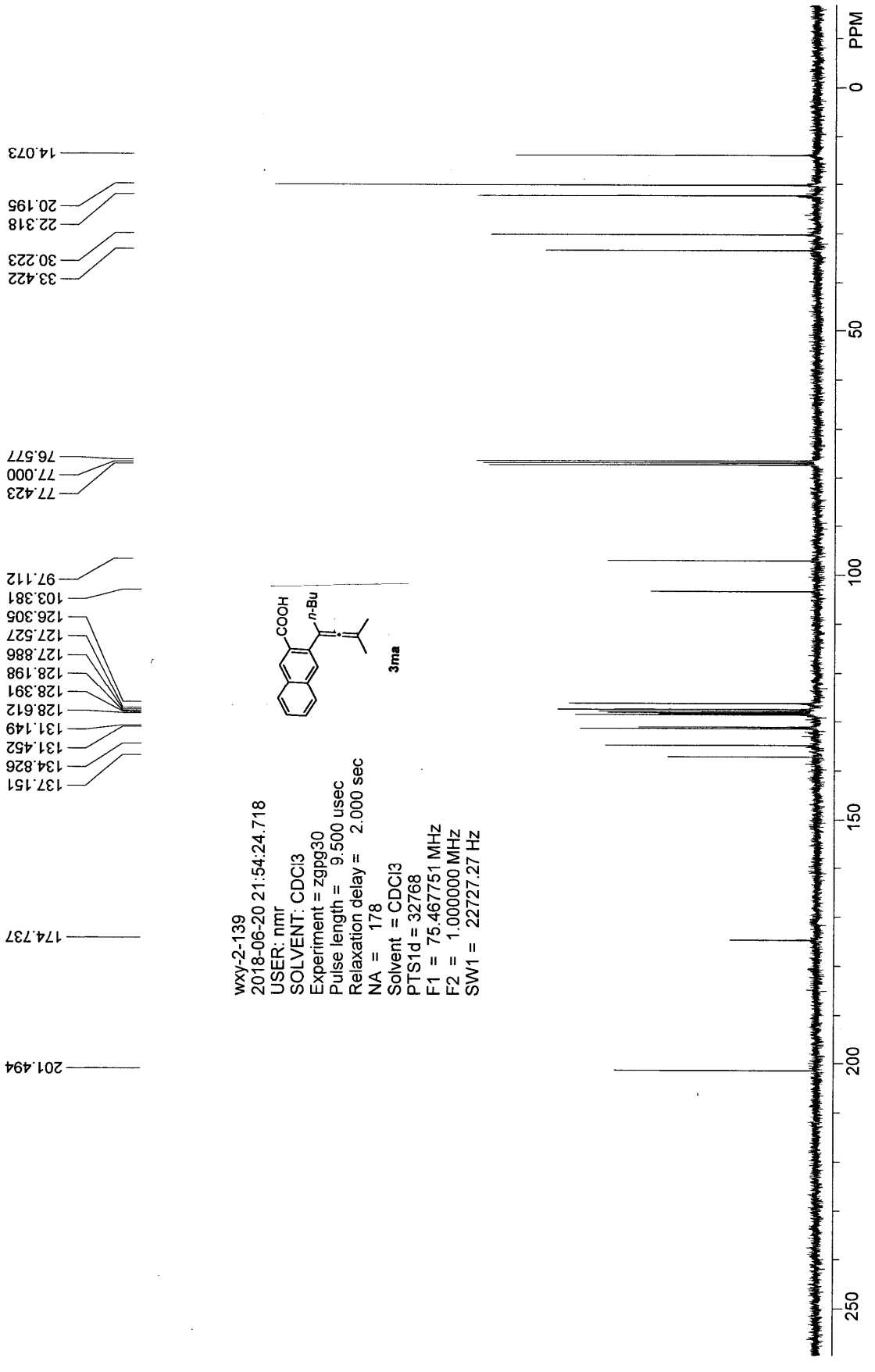

10.783
7.920
7.891
7.350
7.321
7.268
2.550
2.388
2.365
2.341
1.751
1.493
1.470
1.447
1.265
0.887
0.865
0.843
0.000
O2N
COOH
n-C8H17
3kg
wxy-3-023
2018-09-15 15:05:40.453
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
0.86
1.00
1.00
3.04
2.00
6.11
18.39
3.18
10
8
6
4
2
PPM

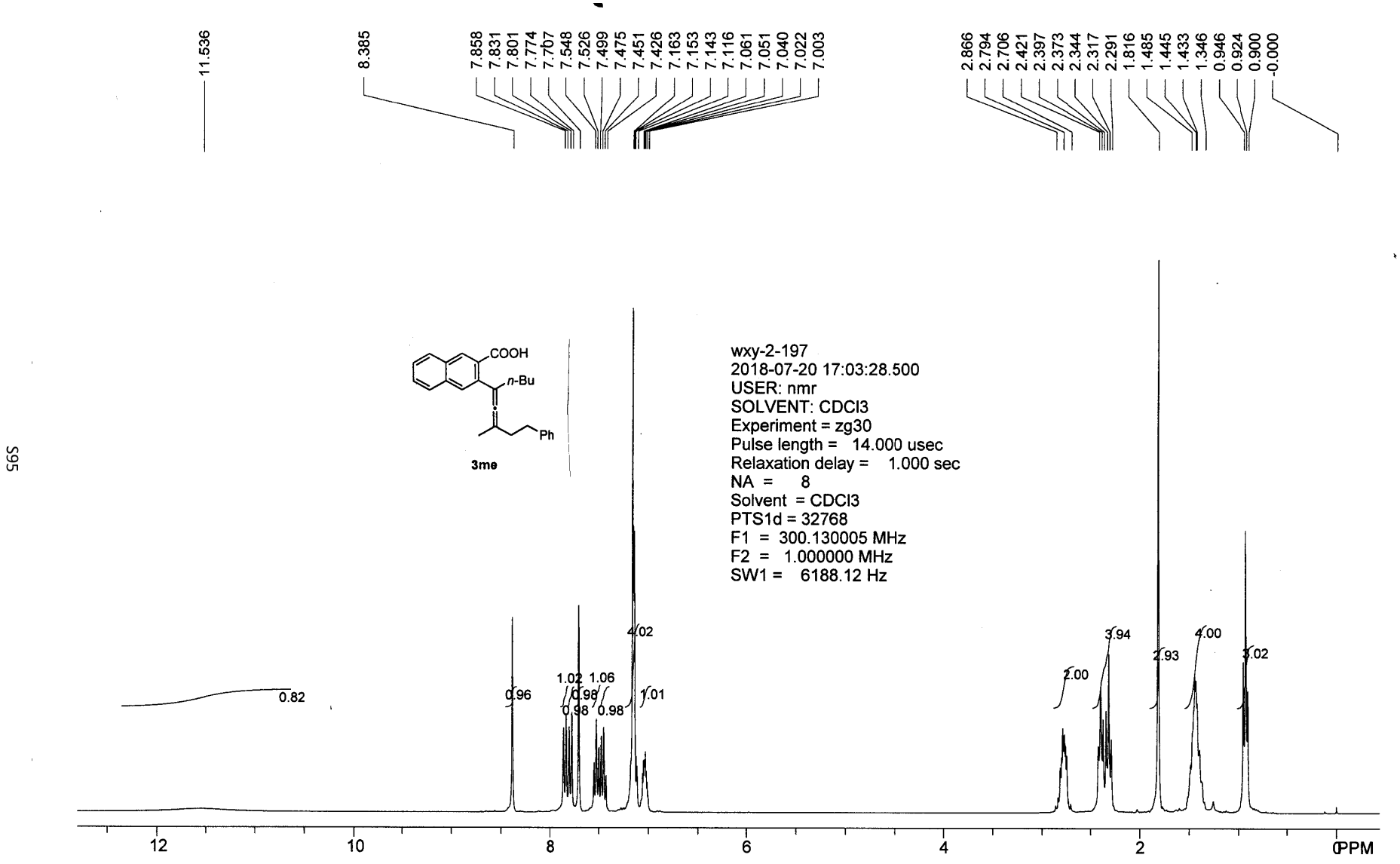

14.008
16.499
20.020
22.584
27.704
29.074
29.267
29.442
31.804
33.109
76.577
77.000
77.423
98.886
101.377
125.303
126.130
130.092
134.945
142.869
148.347
174.249
201.659
PPM
50
100
150
200
250
COOH
n-C8H17
O2N
3kg
wxy-3-023
2018-09-15 16:55:48.296
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 485
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz

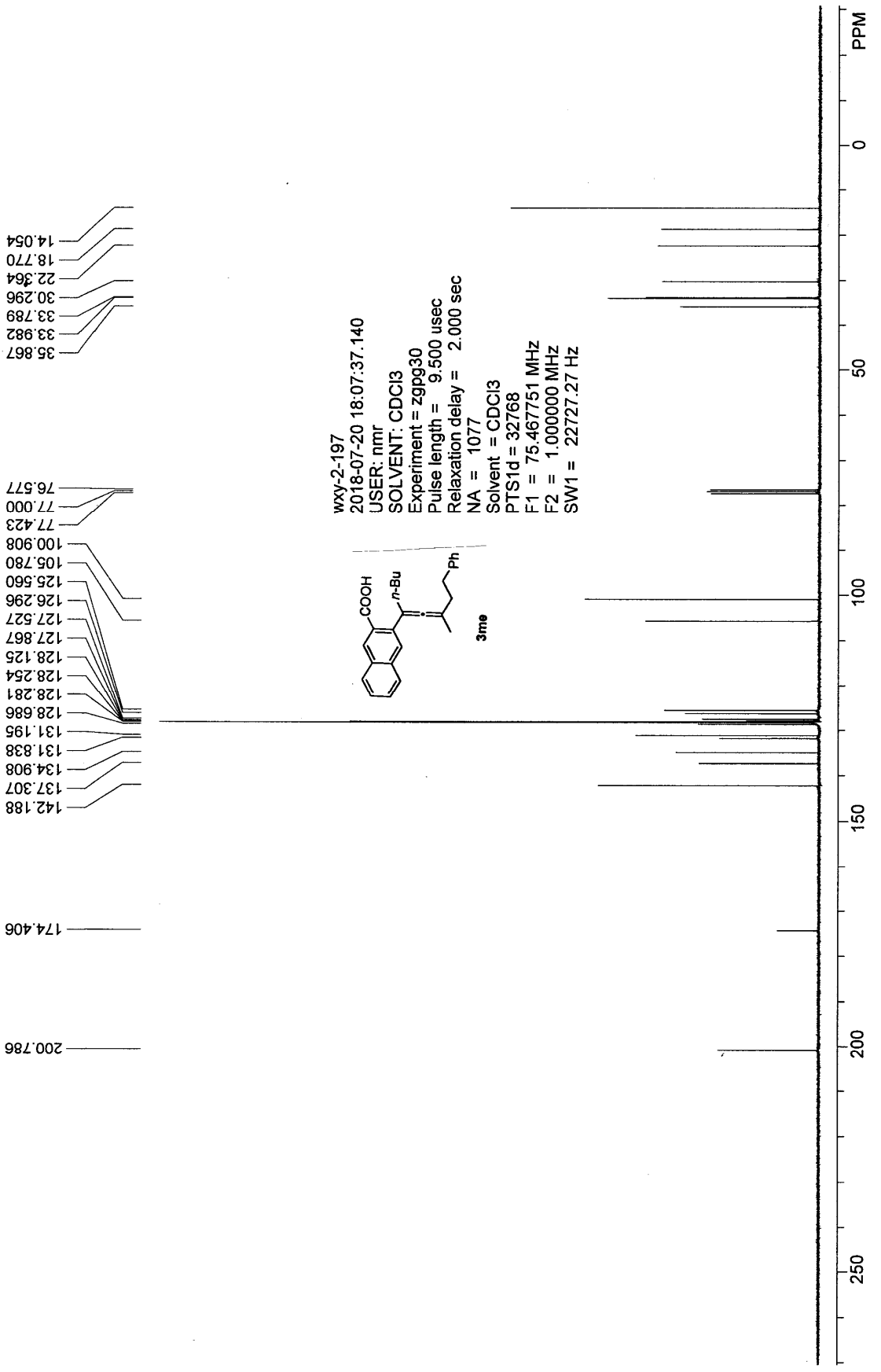

11.982
7.448
7.444
7.390
7.386
7.247
2.387
2.369
2.350
2.332
2.319
2.301
2.280
2.263
2.041
2.022
2.004
1.985
1.967
1.955
1.935
1.918
1.747
1.456
1.443
1.424
1.405
1.387
1.371
1.270
1.255
1.245
1.236
1.218
0.928
0.910
0.893
0.849
0.832
0.814
Cl
COOH
Br
n-Bu
n-C5H11
3lf
wxy-3-153
2018-12-24 05:01:23.294
USER: nmrsu
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 15.000 usec
Relaxation delay = 1.000 sec
NA = 16
Solvent = CDCl3
PTS1d = 65536
F1 = 400.130035 MHz
F2 = 1.000000 MHz
SW1 = 10000.00 Hz
0.97
0.96
0.96
2.00
2.03
2.96
6.12
4.16
3.00
2.96
12
10
8
6
4
2
0
PPM

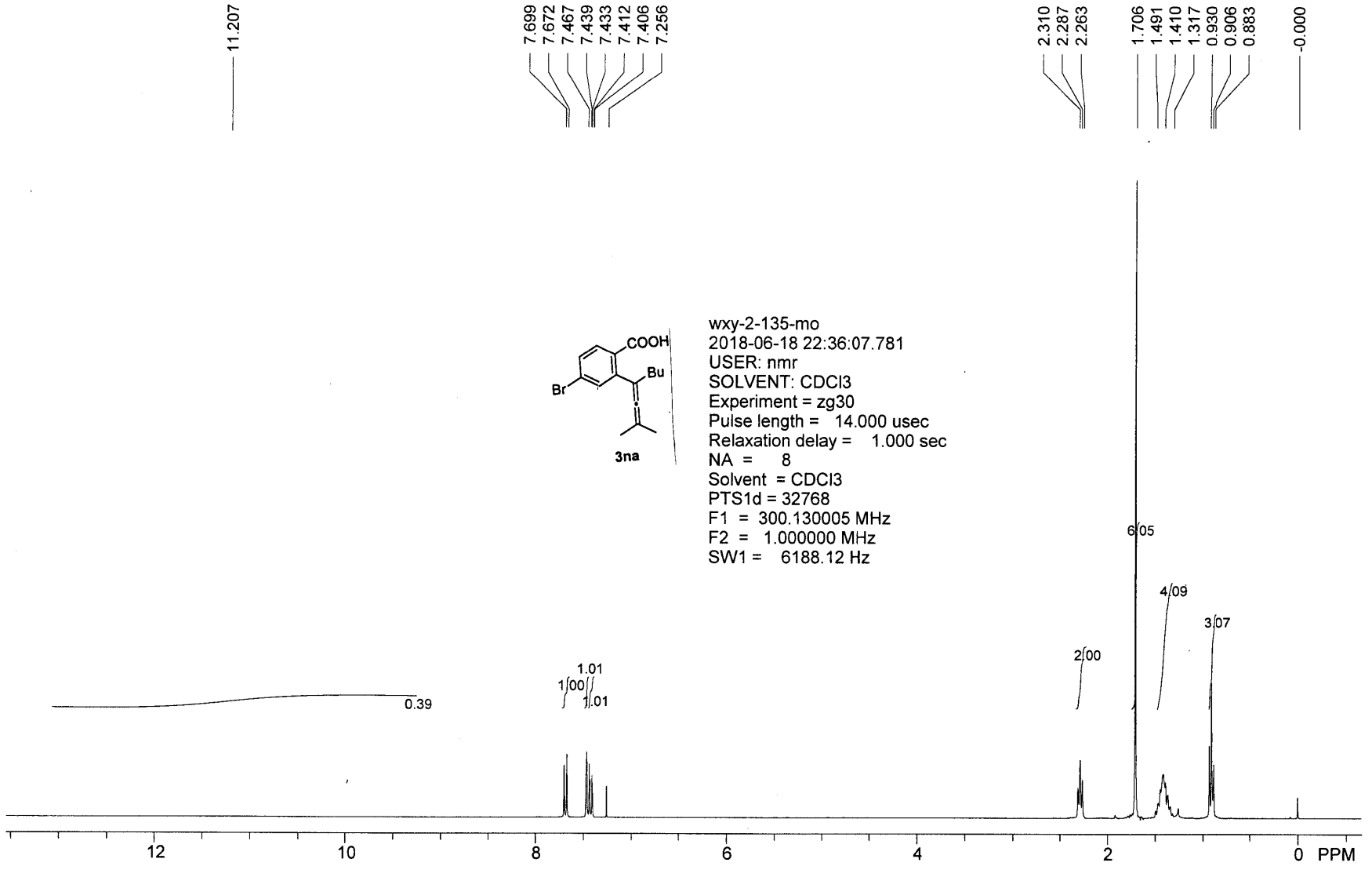

wxy-3-153
2018-12-24 05:25:21.288
USER: nmrsu
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 10.000 usec
Relaxation delay = 1.500 sec
NA = 512
Solvent = CDCl3
PTS1d = 32768
F1 = 100.612877 MHz
F2 = 1.000000 MHz
SW1 = 29761.90 Hz
COOH
Cl
n-Bu
n-C5H11
Br
3lf
PPM
13.936
14.008
18.198
22.206
22.495
26.928
29.980
31.415
32.643
33.862
76.684
77.000
77.316
101.576
103.932
123.704
128.923
129.817
130.710
131.803
141.508
173.153
201.566

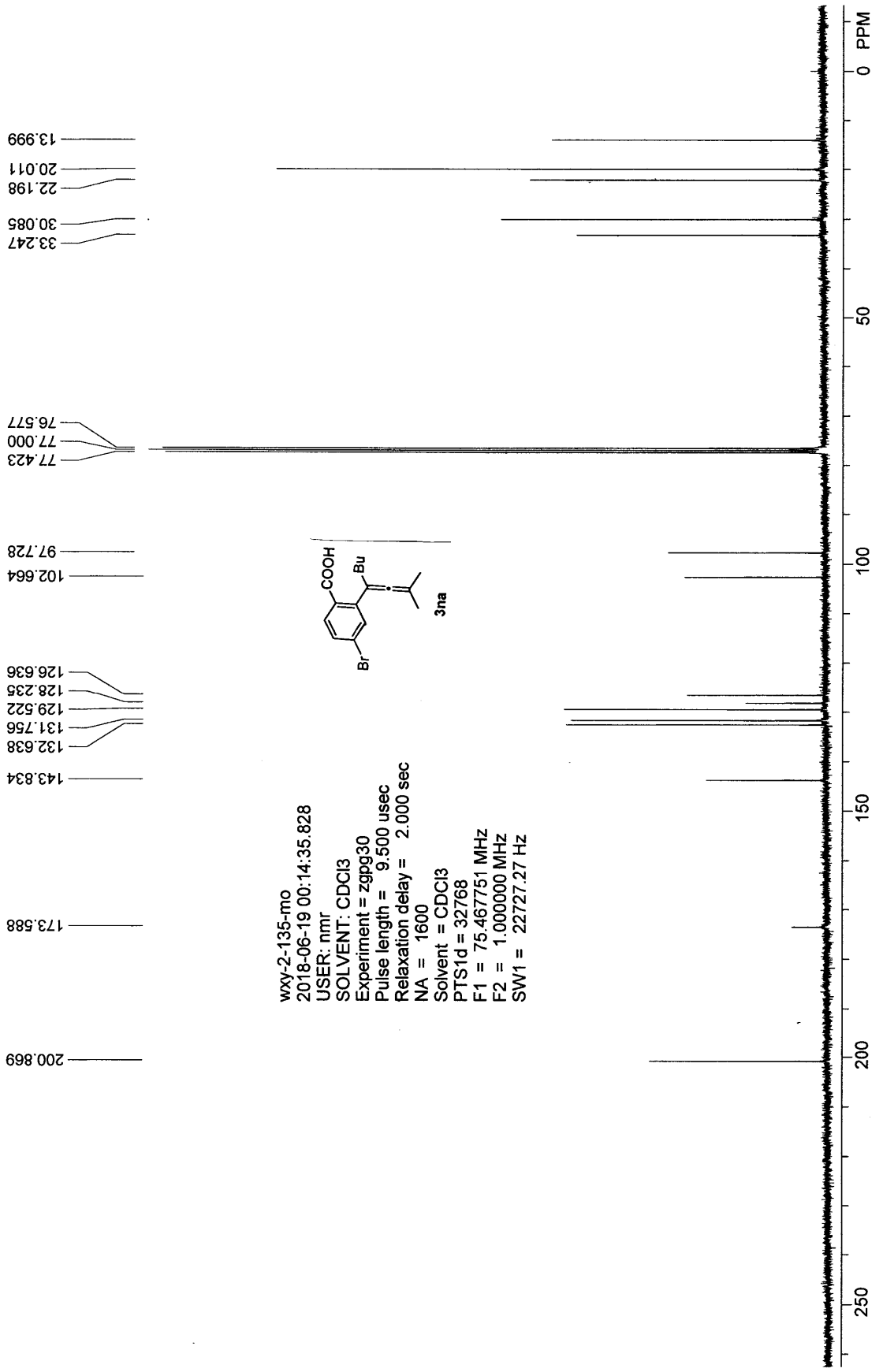

11.332
8.393
7.891
7.864
7.835
7.808
7.759
7.577
7.573
7.554
7.550
7.528
7.523
7.503
7.499
7.476
7.473
7.453
7.450
7.232
2.477
2.454
2.429
1.763
1.546
1.506
1.425
1.399
0.960
0.936
0.912
-0.000
COOH
n-Bu
3ma
wxy-2-139
2018-06-20 18:42:23.046
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
0.63
0.97
1.00
1.00
1.00
1.02
0.99
1.98
6.06
4.02
3.00
14
12
10
8
6
4
2
0
PPM

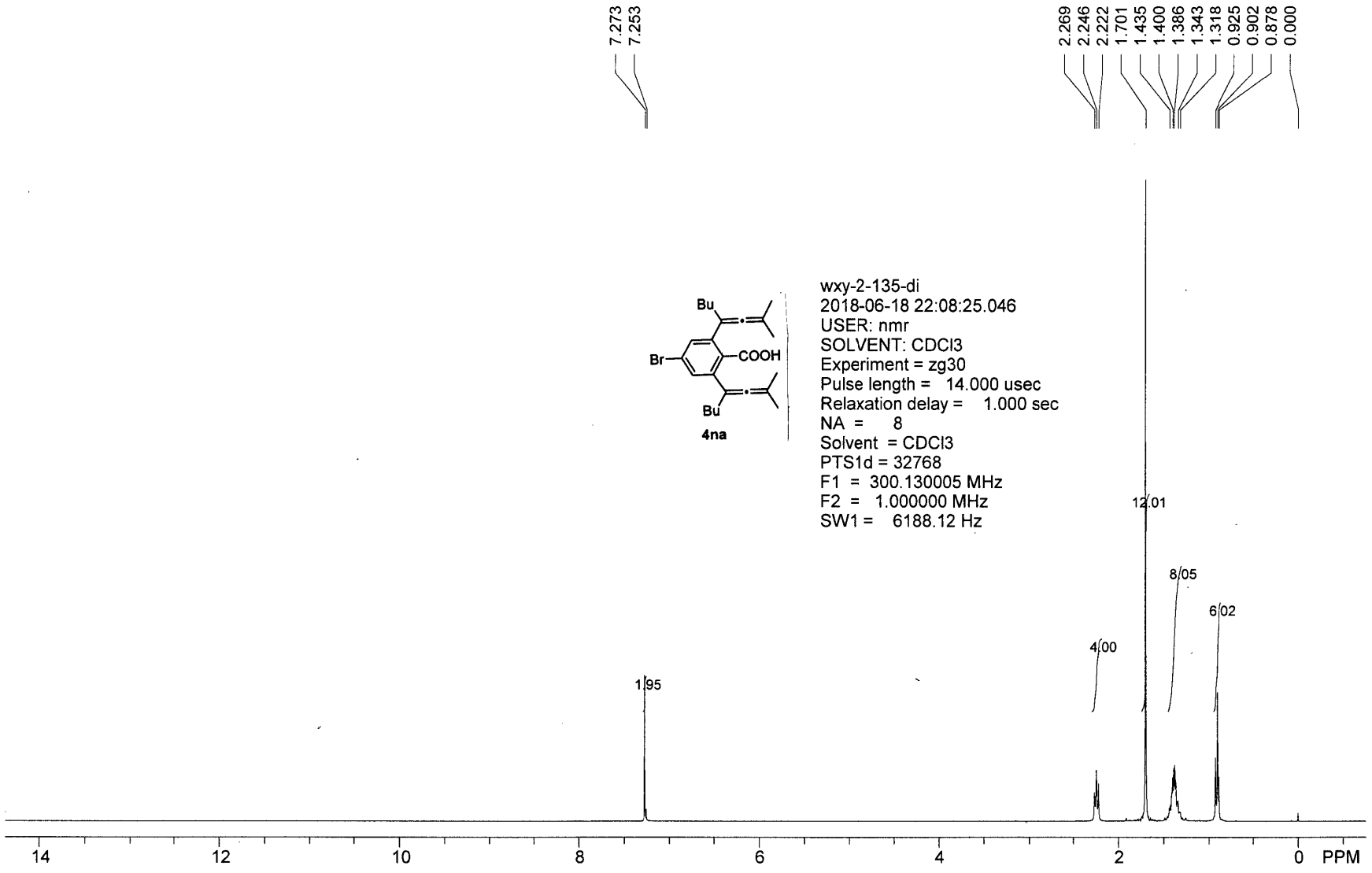

14.073
20.195
22.318
30.223
33.422
76.577
77.000
77.423
97.112
103.381
126.305
127.527
127.886
128.198
128.391
128.612
131.149
131.452
134.826
137.151
174.737
201.494
COOH
n-Bu
3ma
wxy-2-139
2018-06-20 21:54:24.718
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 178
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
250
200
150
100
50
0
PPM

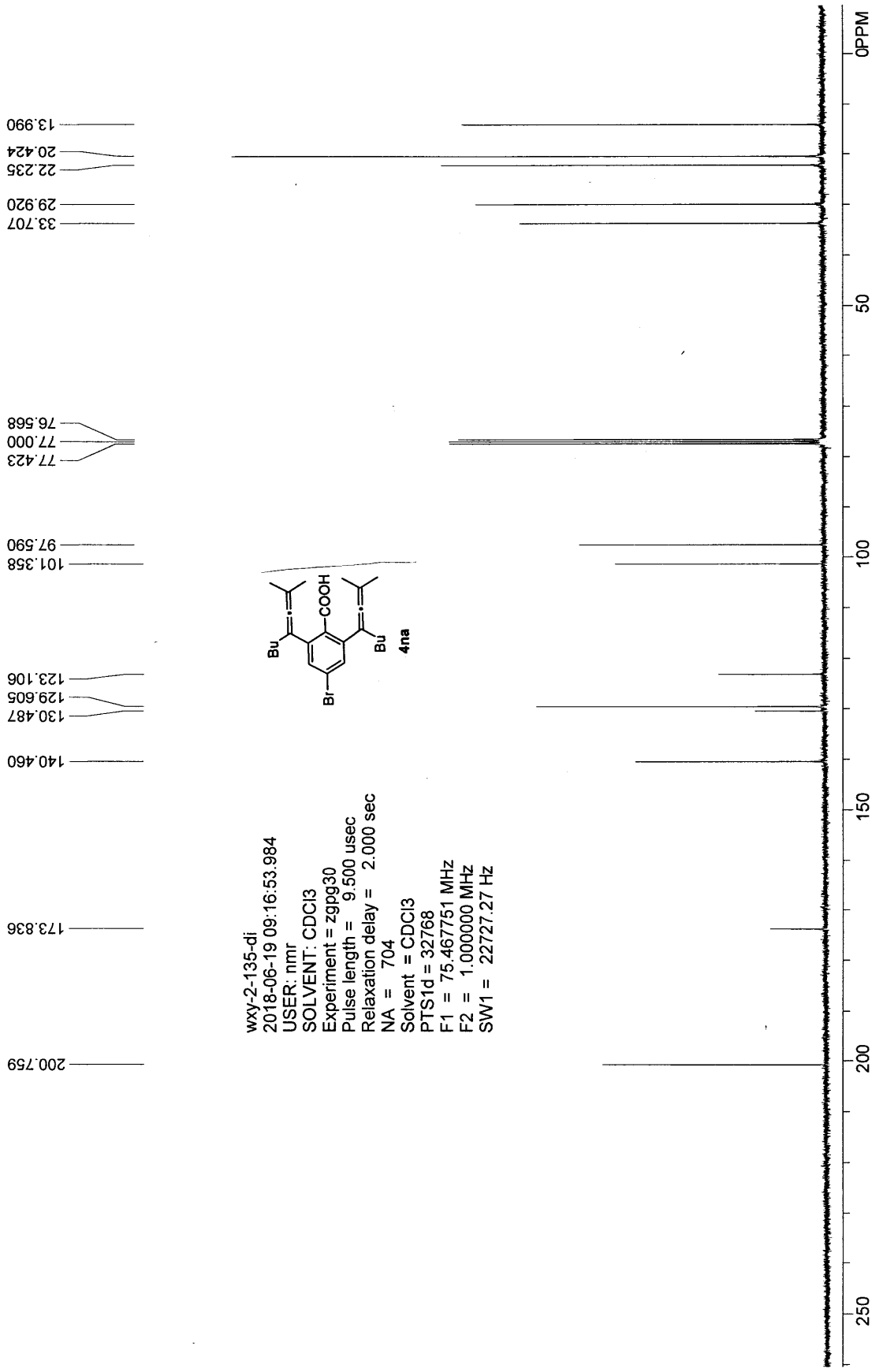

11.536
8.385
7.858
7.831
7.801
7.774
7.707
7.548
7.526
7.499
7.475
7.451
7.426
7.163
7.153
7.143
7.116
7.061
7.051
7.040
7.022
7.003
2.866
2.794
2.706
2.421
2.397
2.373
2.344
2.317
2.291
1.816
1.485
1.445
1.433
1.346
0.946
0.924
0.900
-0.000
COOH
n-Bu
Ph
3me
wxy-2-197
2018-07-20 17:03:28.500
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
0.82
0.96
1.02
0.98
1.06
0.98
0.98
4.02
1.01
2.00
3.94
2.93
4.00
3.02
12
10
8
6
4
2
0
PPM

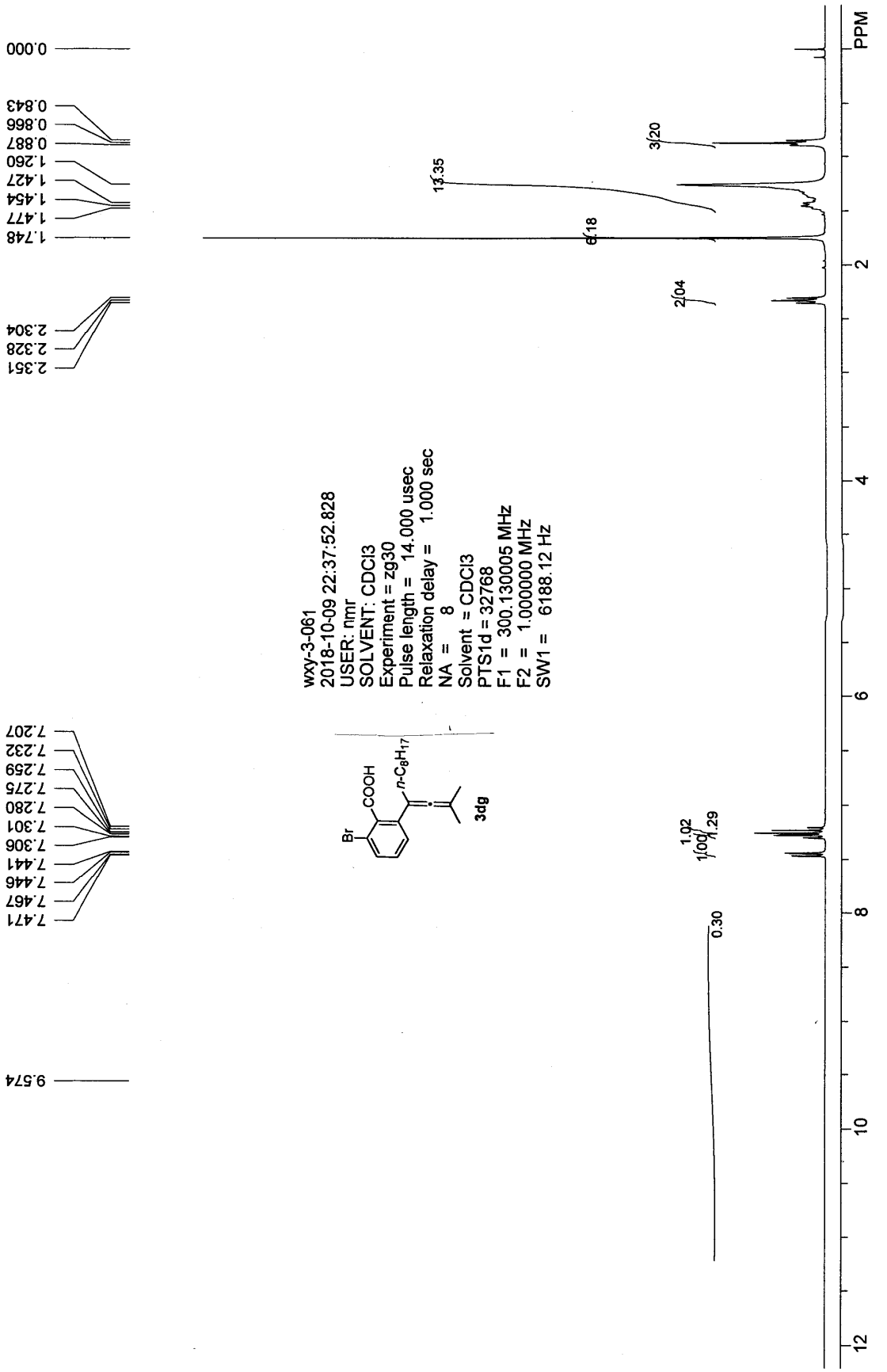

wxy-2-197
2018-07-20 18:07:37.140
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 1077
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
COOH
n-Bu
Ph
3me
14.054
18.770
22.364
30.296
33.789
33.982
35.867
76.577
77.000
77.423
100.908
105.780
125.560
126.296
127.527
127.867
128.125
128.254
128.281
128.686
131.195
131.838
134.908
137.307
142.188
174.406
200.786
PPM
0
50
100
150
200
250

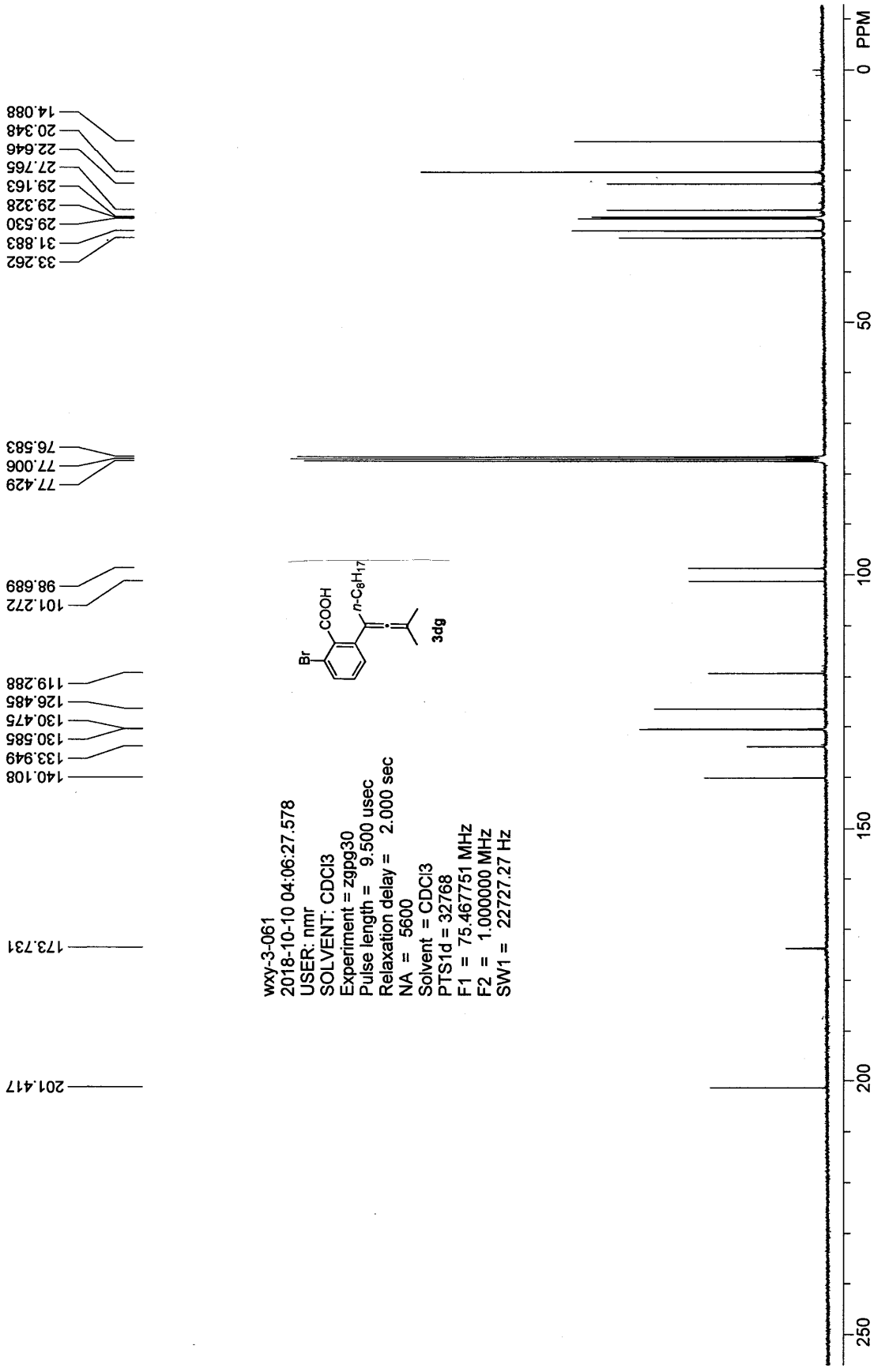

11.207
7.699
7.672
7.467
7.439
7.433
7.412
7.406
7.256
2.310
2.287
2.263
1.706
1.491
1.410
1.317
0.930
0.906
0.883
-0.000
COOH
Bu
Br
3na
wxy-2-135-mo
2018-06-18 22:36:07.781
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
0.39
1.00
1.01
1.01
2.00
6.05
4.09
3.07
12
10
8
6
4
2
0
PPM

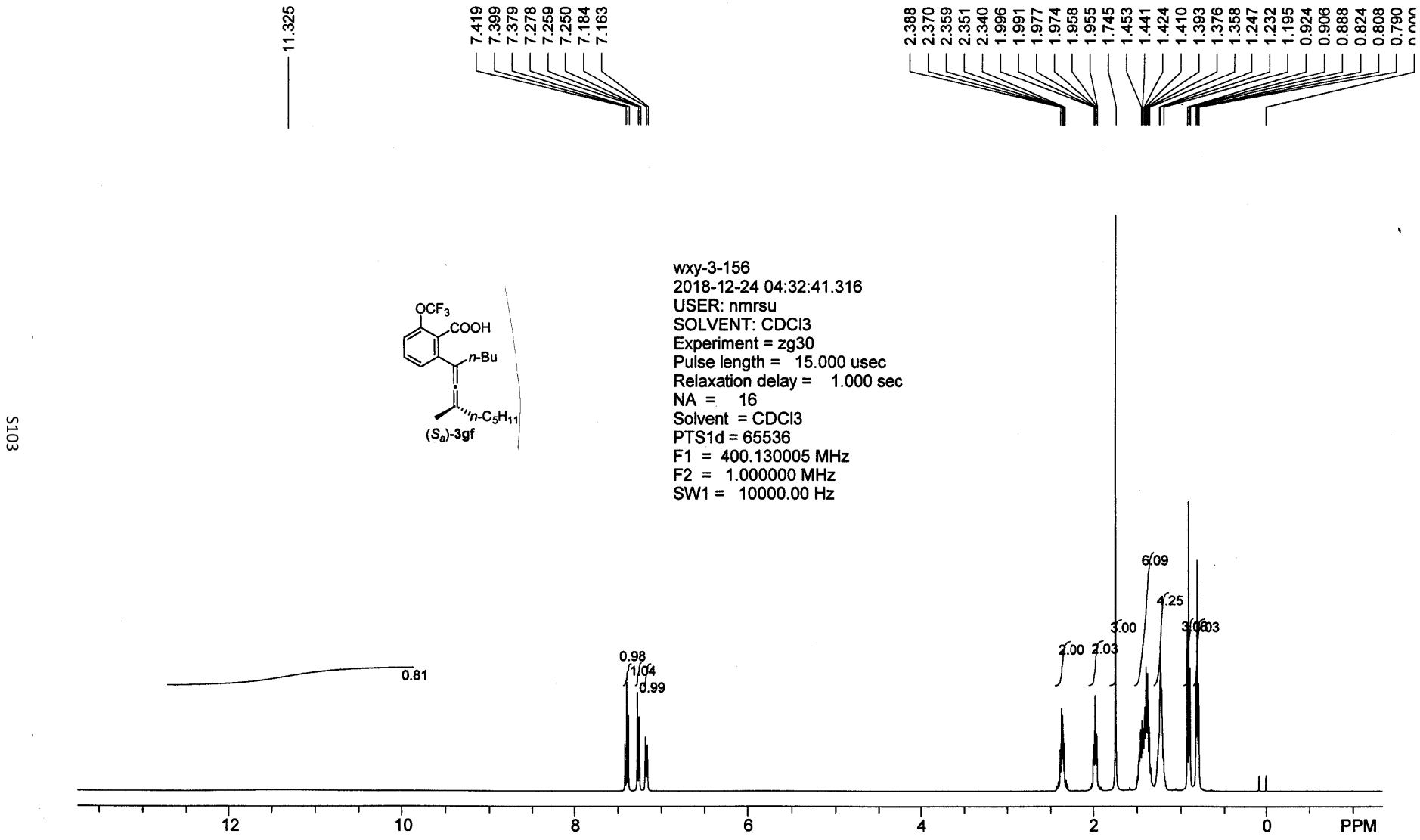

13.999
20.011
22.198
30.085
33.247
76.577
77.000
77.423
97.728
102.664
126.636
128.235
129.522
131.756
132.638
143.834
173.588
200.869
COOH
Bu
Br
3na
wxy-2-135-mo
2018-06-19 00:14:35.828
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 1600
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
250
200
150
100
50
0 PPM

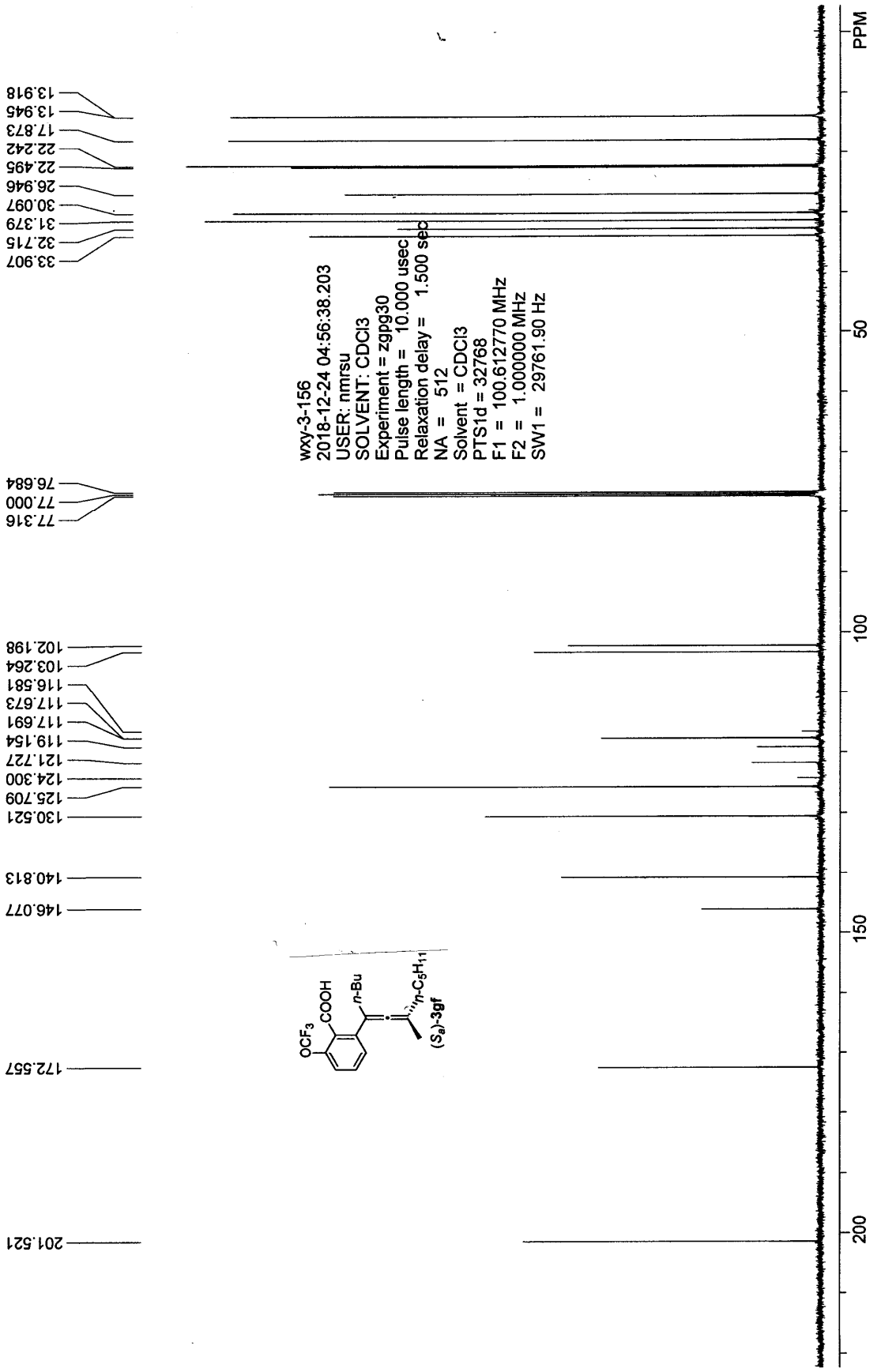

7.273
7.253
2.269
2.246
2.222
1.701
1.435
1.400
1.386
1.343
1.318
0.925
0.902
0.878
0.000
Bu
Br
COOH
Bu
4na
wxy-2-135-di
2018-06-18 22:08:25.046
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
1.95
4.00
12.01
8.05
6.02
14
12
10
8
6
4
2
0
PPM

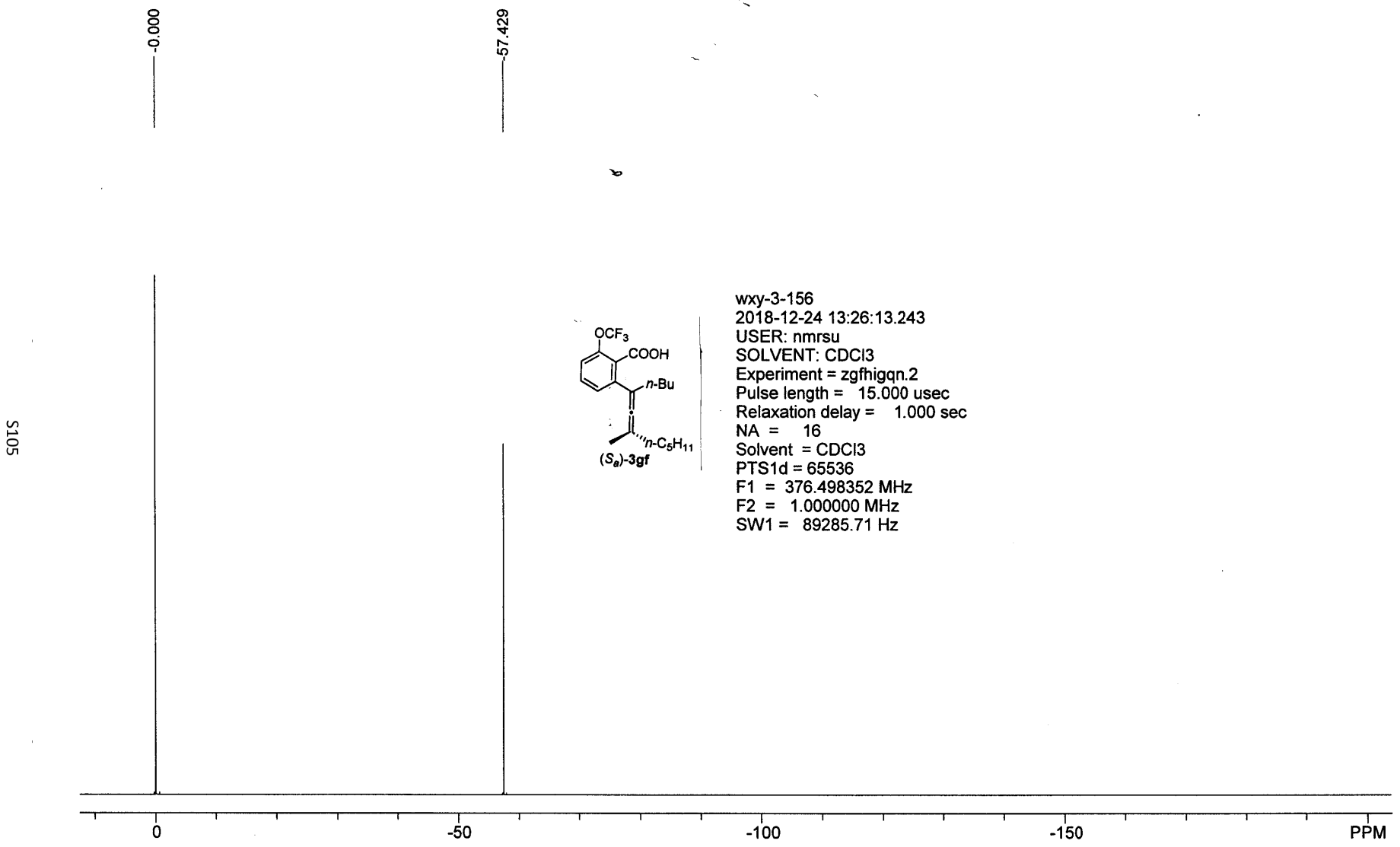

13.990
20.424
22.235
29.920
33.707
76.568
77.000
77.423
97.590
101.358
123.106
129.605
130.487
140.460
173.836
200.759
wxy-2-135-di
2018-06-19 09:16:53.984
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 704
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
Bu
COOH
Bu
Br
4na
0PPM
50
100
150
200
250


wxy-3-061
2018-10-09 22:37:52.828
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
Br
COOH
n-C8H17
3dg
PPM
0.000
0.843
0.866
0.887
1.260
1.427
1.454
1.477
1.748
2.304
2.328
2.351
7.207
7.232
7.259
7.275
7.280
7.301
7.306
7.441
7.446
7.467
7.471
9.574
1.35
6.18
3.20
2.04
1.02
1.00
1.29
0.30


14.088
20.348
22.646
27.765
29.163
29.328
29.530
31.883
33.262
76.583
77.006
77.429
98.689
101.272
119.288
126.485
130.475
130.585
133.949
140.108
173.731
201.417
wxy-3-061
2018-10-10 04:06:27.578
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 5600
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
Br
COOH
n-C8H17
3dg
0
50
100
150
200
250
PPM


11.325
7.419
7.399
7.379
7.278
7.259
7.250
7.184
7.163
2.388
2.370
2.359
2.351
2.340
1.996
1.991
1.977
1.974
1.958
1.955
1.745
1.453
1.441
1.424
1.410
1.393
1.376
1.358
1.247
1.232
1.195
0.924
0.906
0.888
0.824
0.808
0.790
OCF3
COOH
n-Bu
n-C5H11
(Sa)-3gf
wxy-3-156
2018-12-24 04:32:41.316
USER: nmrsu
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 15.000 usec
Relaxation delay = 1.000 sec
NA = 16
Solvent = CDCl3
PTS1d = 65536
F1 = 400.130005 MHz
F2 = 1.000000 MHz
SW1 = 10000.00 Hz
0.81
0.98
1.04
0.99
2.00
2.03
3.00
6.09
4.25
3.06
3.03
12
10
8
6
4
2
0
PPM


wxy-3-156
2018-12-24 04:56:38.203
USER: nmrsu
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 10.000 usec
Relaxation delay = 1.500 sec
NA = 512
Solvent = CDCl3
PTS1d = 32768
F1 = 100.612770 MHz
F2 = 1.000000 MHz
SW1 = 29761.90 Hz
PPM
13.918
13.945
17.873
22.242
22.495
26.946
30.097
31.379
32.715
33.907
76.684
77.000
77.316
102.198
103.264
116.581
117.673
117.691
119.154
121.727
124.300
125.709
130.521
140.813
146.077
172.557
201.521
OCF3
COOH
n-Bu
n-C5H11
(Sa)-3gf

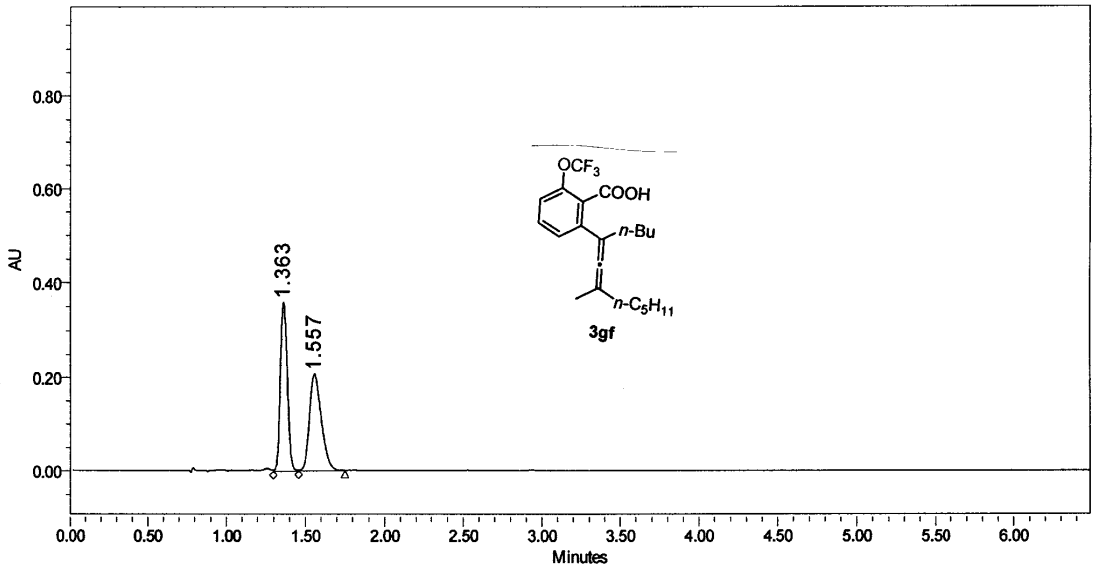

-0.000
-57.429
OCF3
COOH
n-Bu
n-C5H11
(Sa)-3gf
wxy-3-156
2018-12-24 13:26:13.243
USER: nmrsu
SOLVENT: CDCl3
Experiment = zgfhigqn.2
Pulse length = 15.000 usec
Relaxation delay = 1.000 sec
NA = 16
Solvent = CDCl3
PTS1d = 65536
F1 = 376.498352 MHz
F2 = 1.000000 MHz
SW1 = 89285.71 Hz
0
-50
-100
-150
PPM

Empower 3

Default Individual Report

SAMPLE INFORMATION

Sample Name:	wxy-3-048-rac-oj-3-98-2	Acquired By:	System
Sample Type:	Unknown	Sample Set Name	
Vial:	1:D,2	Acq. Method Set:	test1
Injection #:	2	Processing Method	TEST
Injection Volume:	0.50 ul	Channel Name:	PDA Ch2 254nm@4.8nm
Run Time:	50.0 Minutes	Proc. Chnl. Descr.:	PDA Ch2 254nm@4.8nm
Date Acquired:	1/21/2019 10:11:06 AM CST		
Date Processed:	1/21/2019 4:20:48 PM CST		



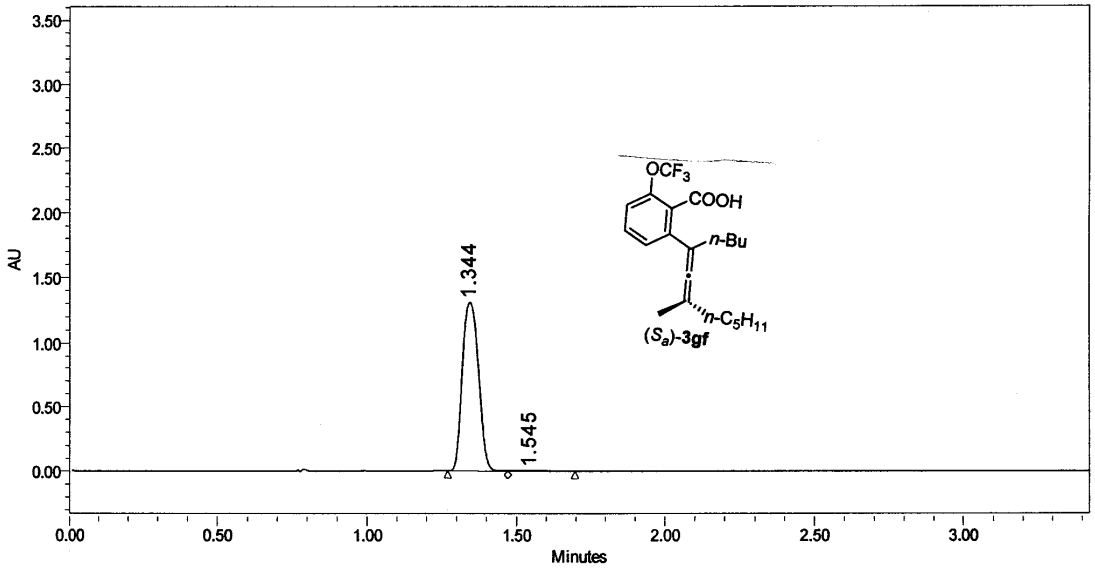
	RT	Peak Type	Height	Width (sec)	Area	% Area
1	1.363	Unknown	359850	9.600	1093604	50.27
2	1.557	Unknown	207147	17.500	1081703	49.73

Empower 3

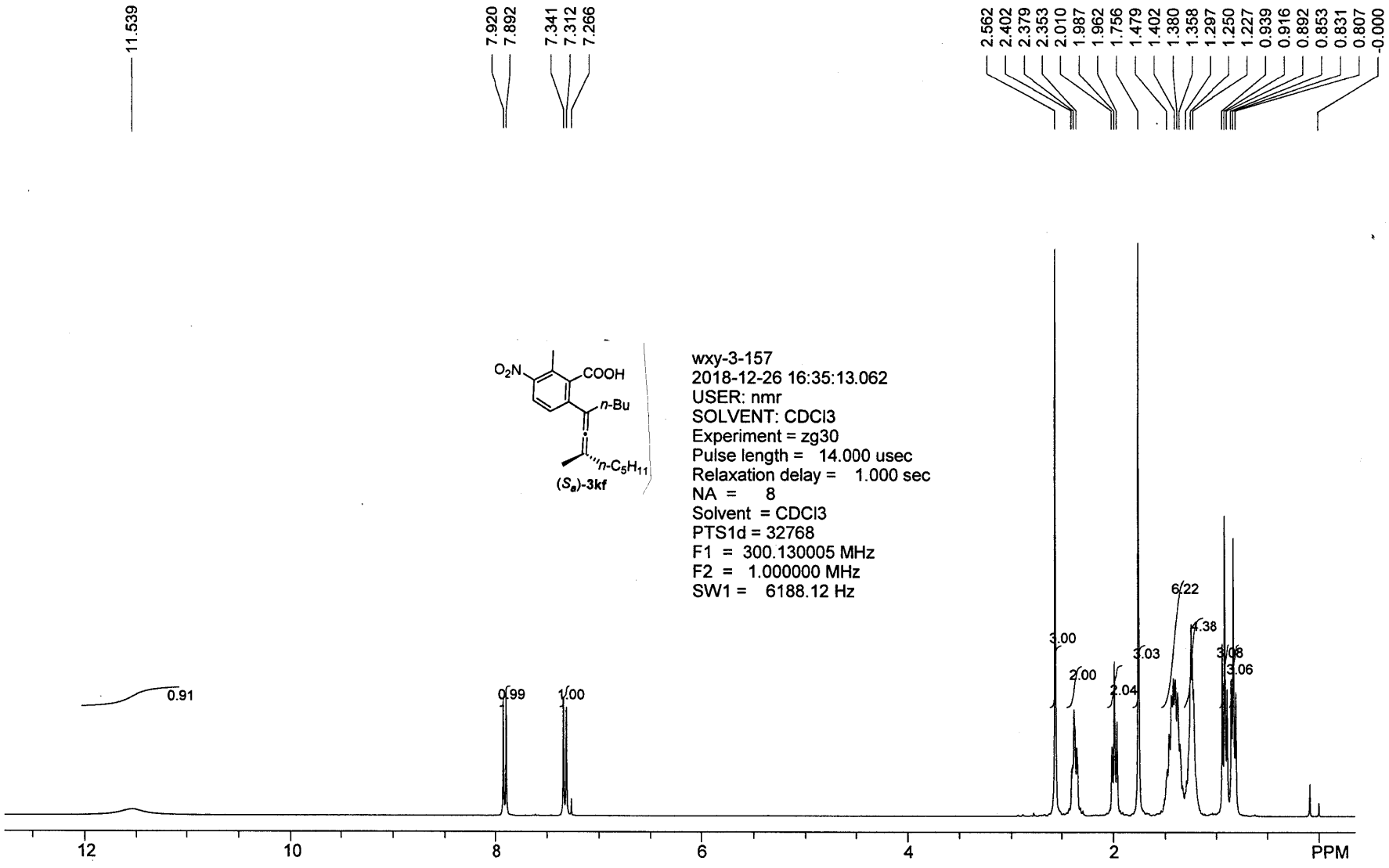
Default Individual Report

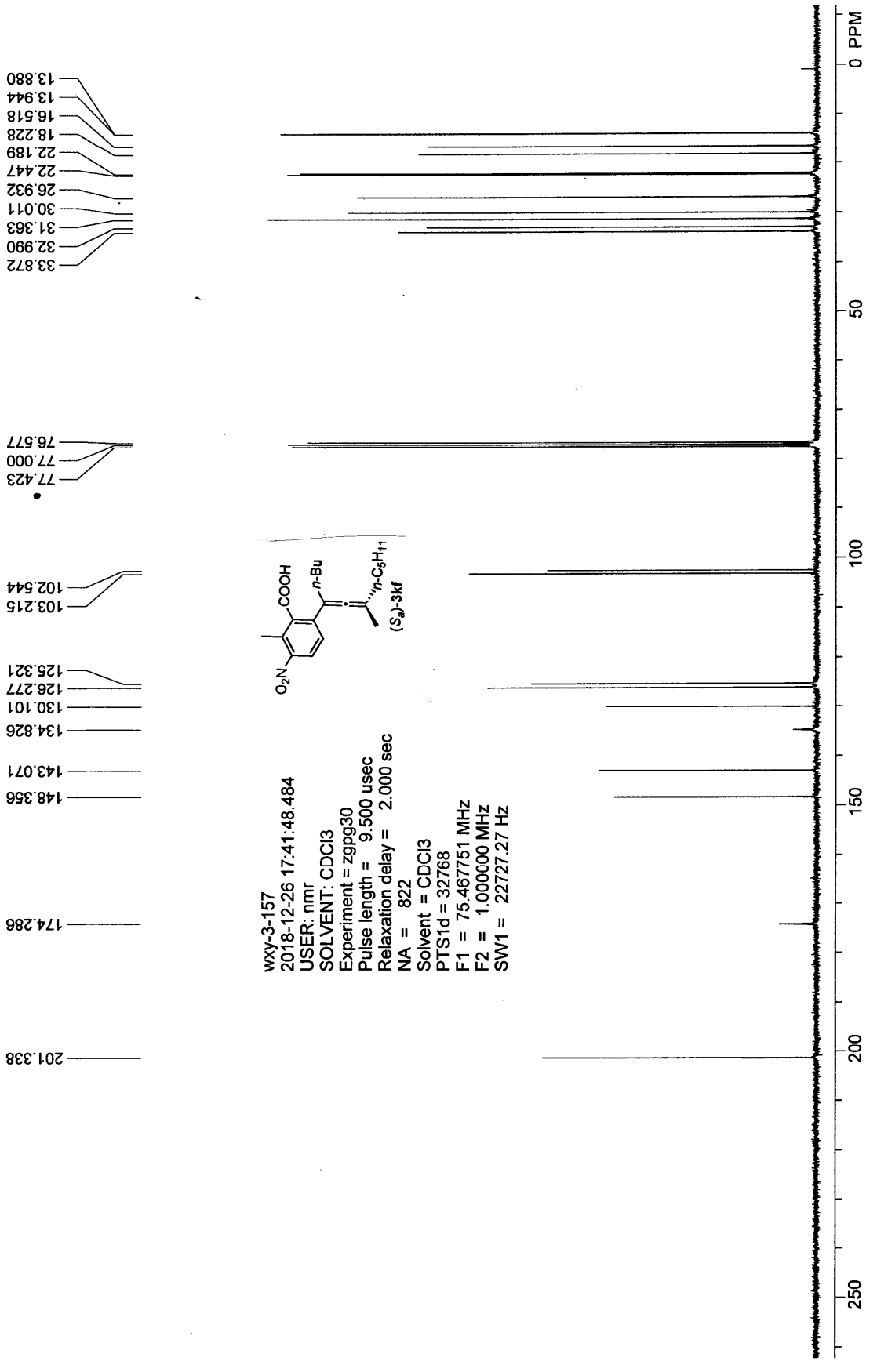
SAMPLE INFORMATION

Sample Name:	wxy-3-156	Acquired By:	System
Sample Type:	Unknown	Sample Set Name	
Vial:	1:D,5	Acq. Method Set:	test1
Injection #:	1	Processing Method	TEST
Injection Volume:	1.00 ul	Channel Name:	PDA Ch2 254nm@4.8nm
Run Time:	50.0 Minutes	Proc. Chnl. Descr.:	PDA Ch2 254nm@4.8nm
Date Acquired:	1/21/2019 10:41:55 AM CST		
Date Processed:	1/21/2019 4:22:18 PM CST		



	RT	Peak Type	Height	Width (sec)	Area	% Area
1	1.344	Unknown	1308604	12.100	4947258	99.74
2	1.545	Unknown	2011	13.550	13023	0.26

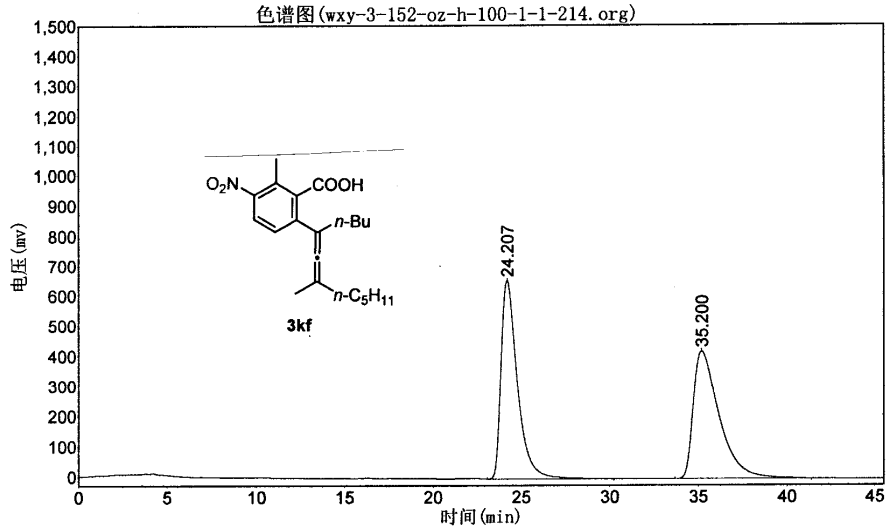

11.539
7.920
7.892
7.341
7.312
7.266
2.562
2.402
2.379
2.353
2.010
1.987
1.962
1.756
1.479
1.402
1.380
1.358
1.297
1.250
1.227
0.939
0.916
0.892
0.853
0.831
0.807
-0.000
O2N
COOH
n-Bu
n-C5H11
(Sa)-3kf
wxy-3-157
2018-12-26 16:35:13.062
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
0.91
0.99
1.00
3.00
2.00
2.04
3.03
6.22
4.38
3.08
3.06
12
10
8
6
4
2
PPM


13.880
13.944
16.518
18.228
22.189
22.447
26.932
30.011
31.363
32.990
33.872
76.577
77.000
77.423
102.544
103.215
125.321
126.277
130.101
134.826
143.071
148.356
174.286
201.338
0 PPM
50
100
150
200
250
wxy-3-157
2018-12-26 17:41:48.484
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 822
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
COOH
n-Bu
n-C5H11
(Sa)-3kf

wxy-3-152-oz-h-100-1-1-214

实验时间：2018-12-29,11:10:52　　报告时间：2018-12-29,18:52:14
谱图文件:D:\zhuguangjiong\wxy\20181228\wxy-3-152-oz-h-100-1-1-214.org

实验内容简介：



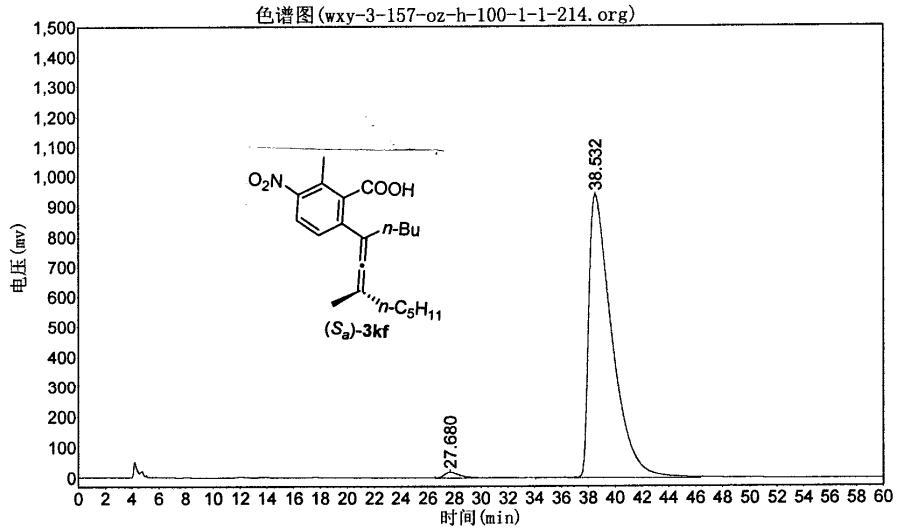
分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		24.207	657798.938	42242948.000	49.6882
2		35.200	422149.750	42773028.000	50.3118
总计			1079948.688	85015976.000	100.0000

wxy-3-157-oz-h-100-1-1-214

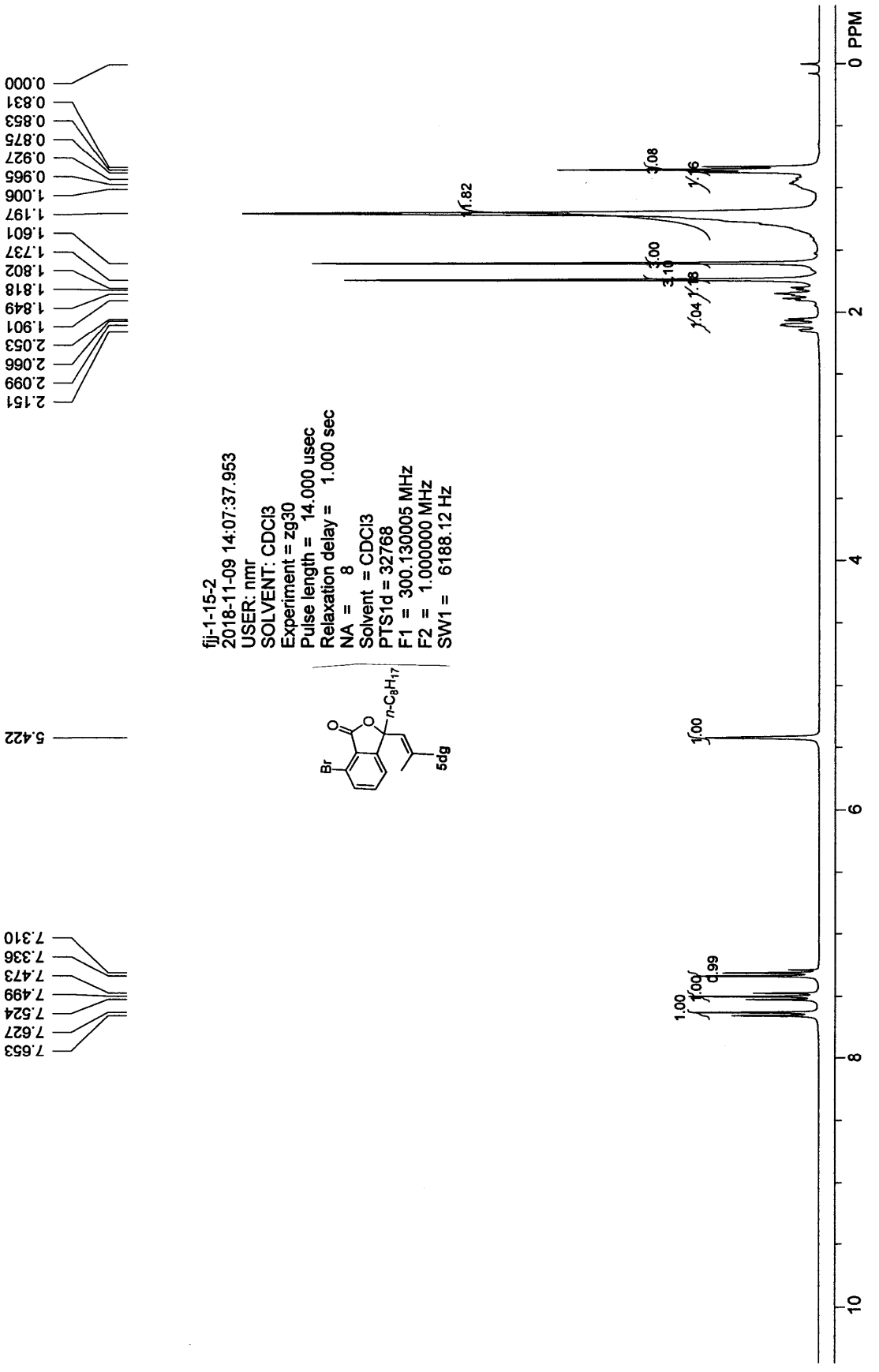
实验时间：2018-12-29,11:57:06　　报告时间：2018-12-29,18:53:21
谱图文件:D:\zhuguangjiong\wxy\20181228\wxy-3-157-oz-h-100-1-1-214.org

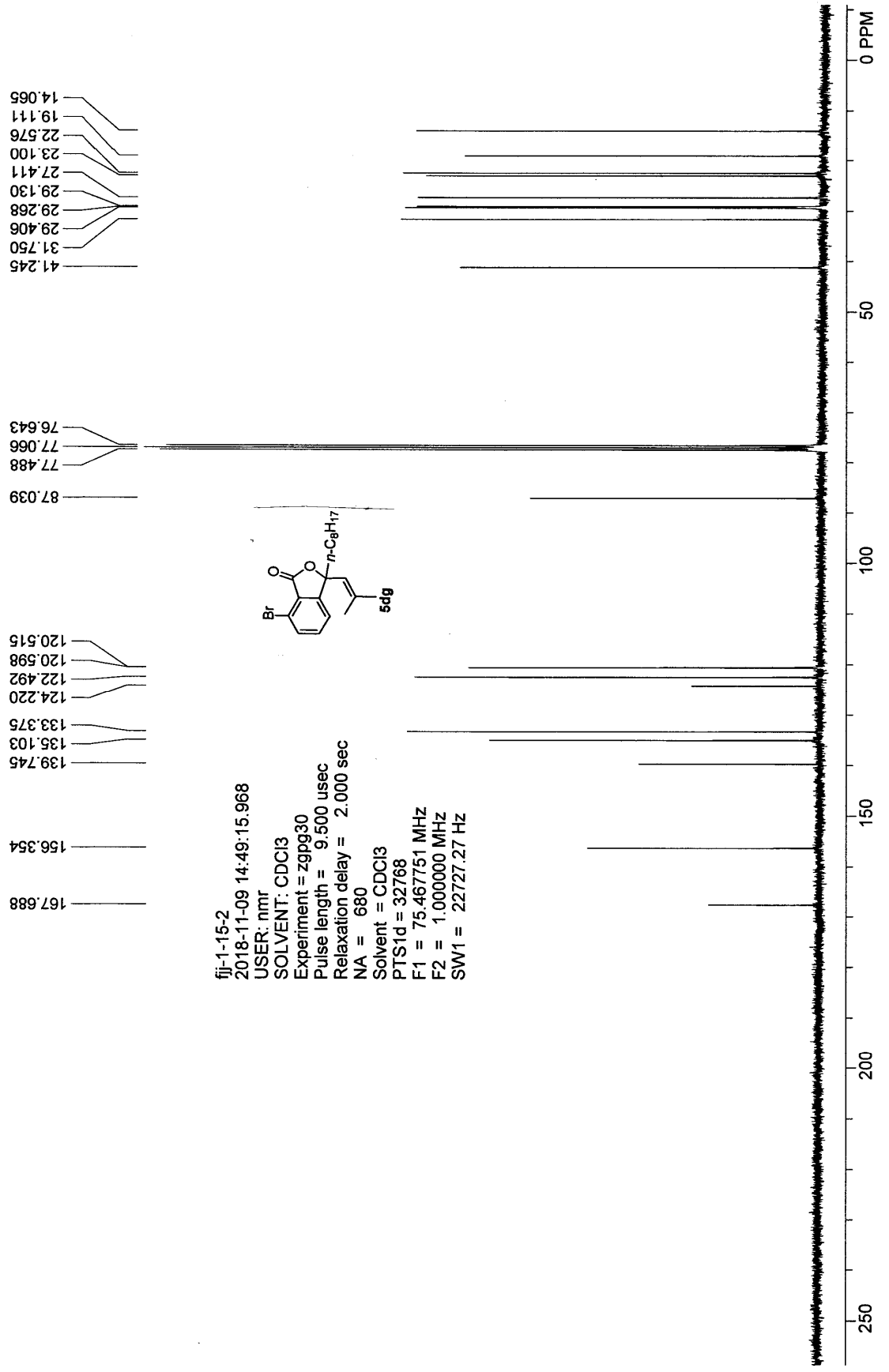
实验内容简介：

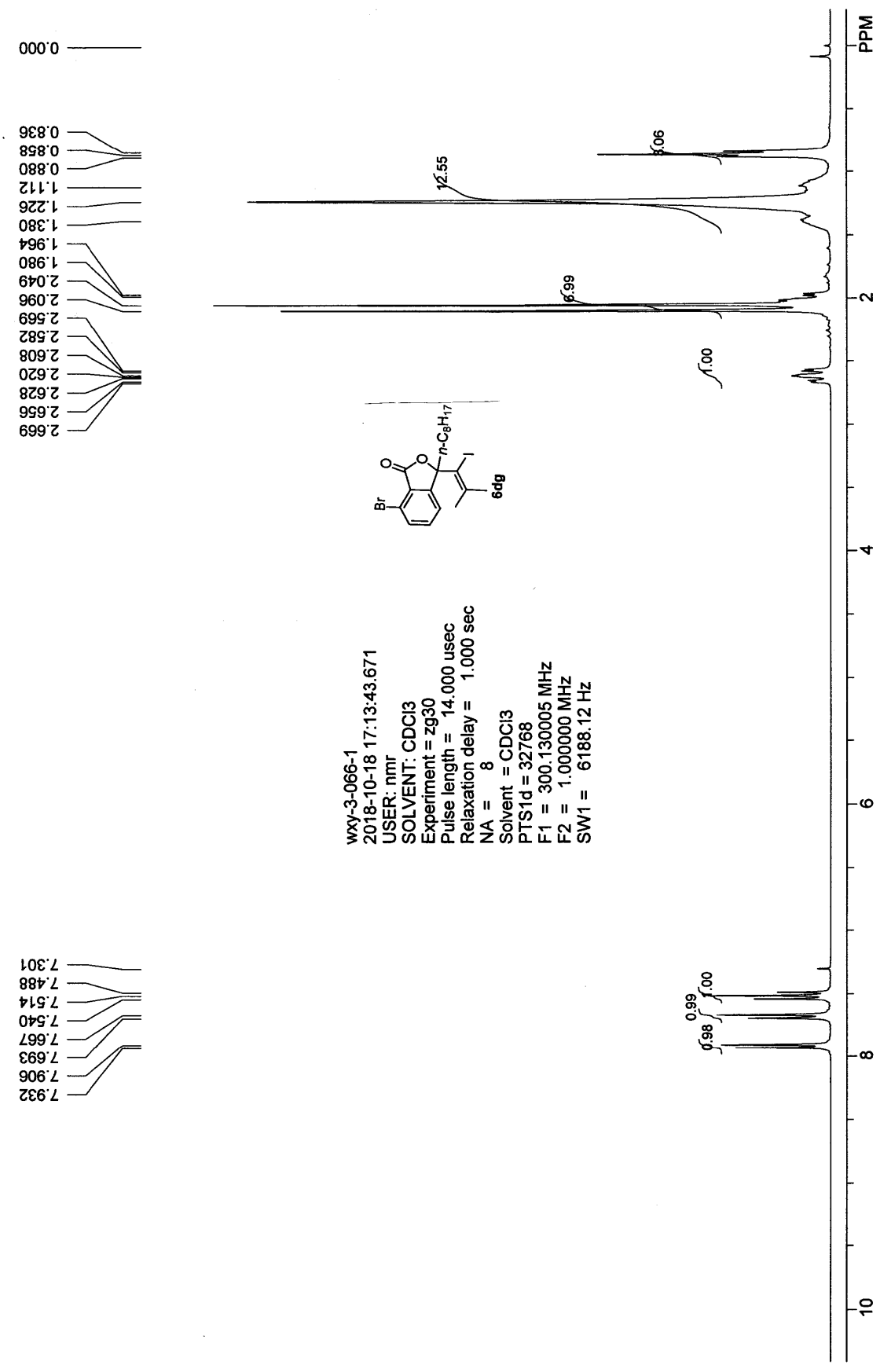


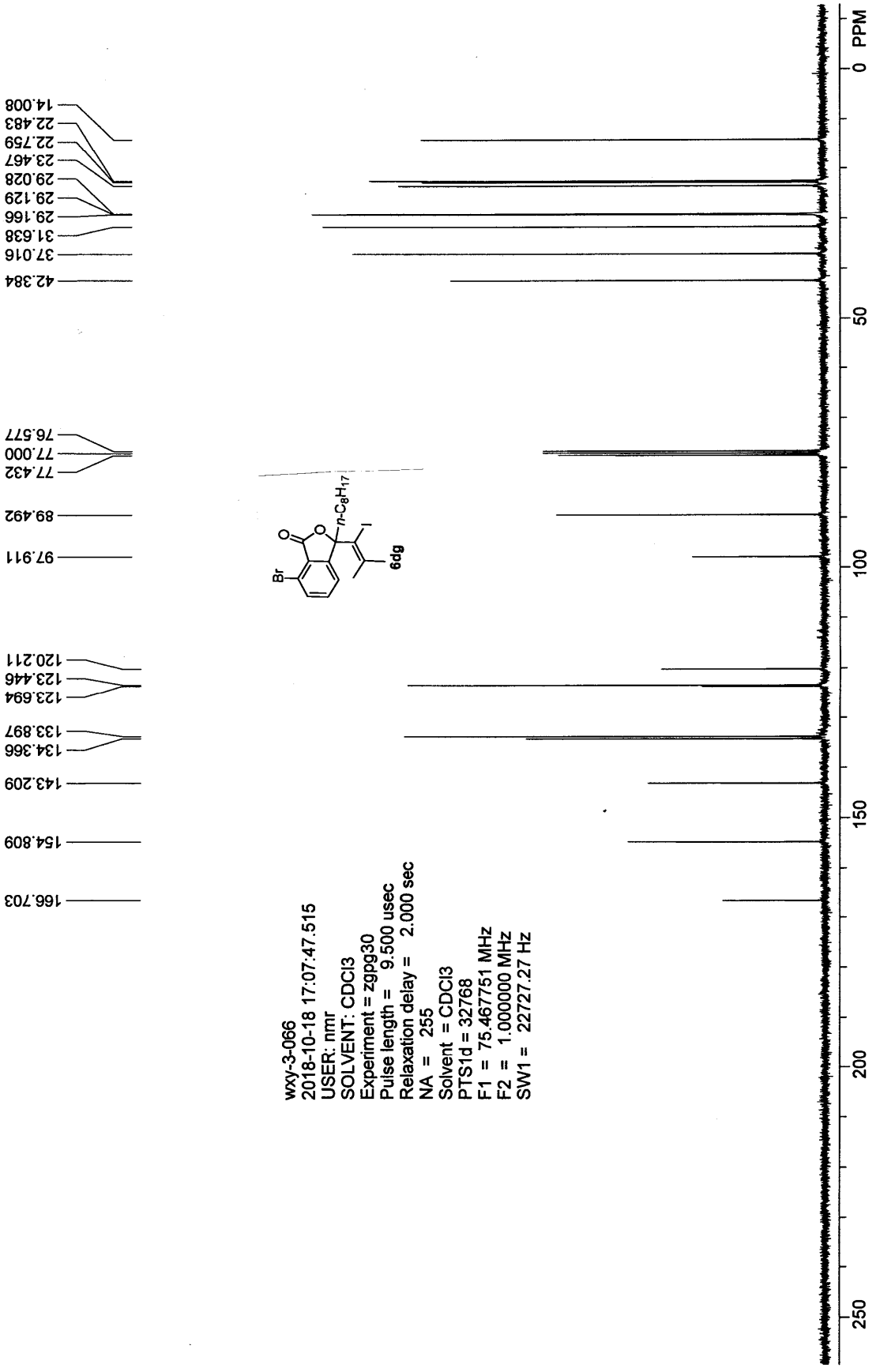
分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		27.680	19245.367	1573623.125	1.3856
2		38.532	940428.125	111992480.000	98.6144
总计			959673.492	113566103.125	100.0000

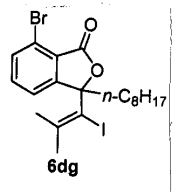

fjj-1-15-2
2018-11-09 14:07:37.953
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
5dg
0 PPM


14.065
19.111
22.576
23.100
27.411
29.130
29.268
29.406
31.750
41.245
76.643
77.066
77.488
87.039
120.515
120.598
122.492
124.220
133.375
135.103
139.745
156.354
167.688
0 PPM
50
100
150
200
250
fjj-1-15-2
2018-11-09 14:49:15.968
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 680
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
Br
n-C6H17
5dg

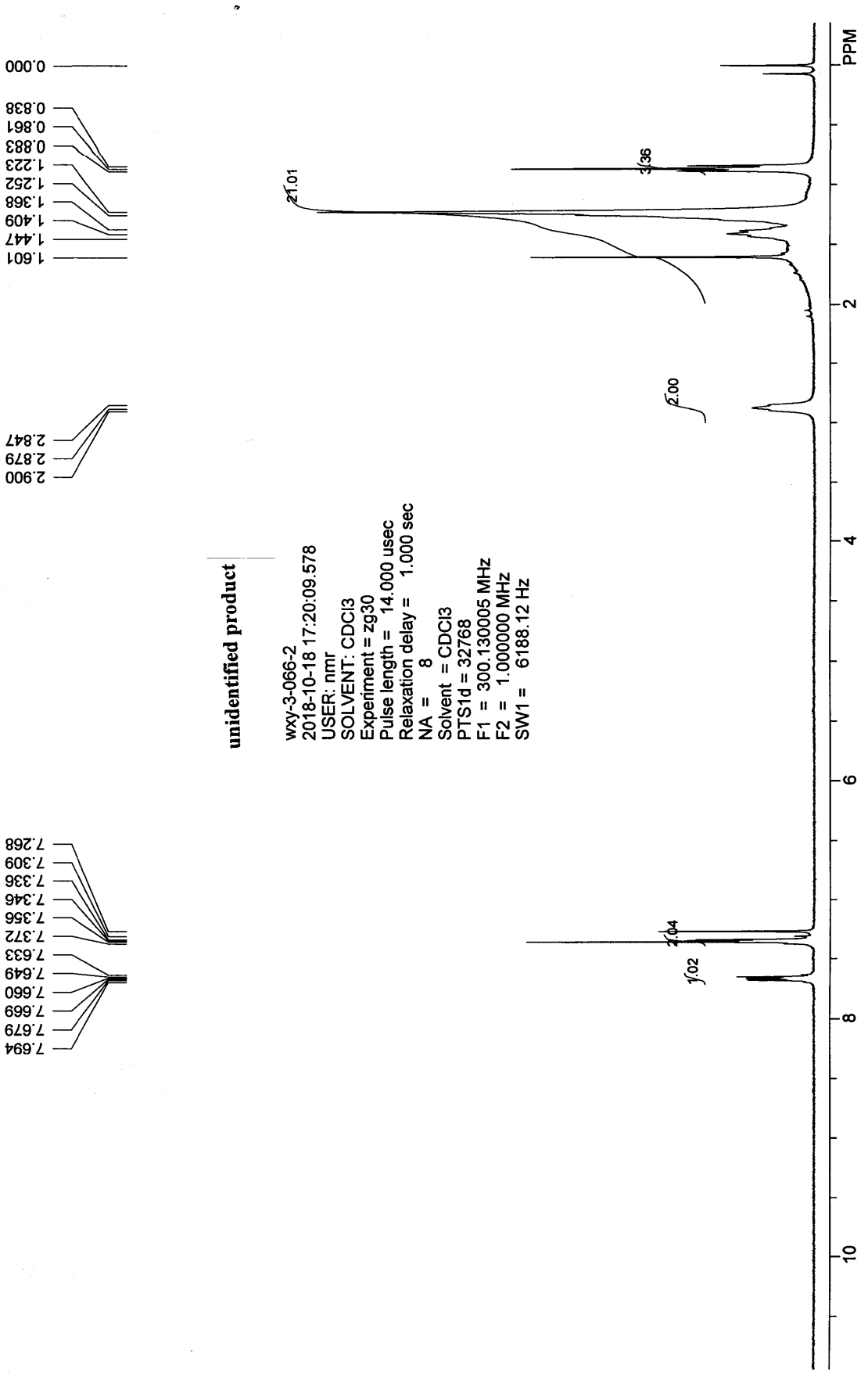

0.000
0.836
0.858
0.880
1.112
1.226
1.380
1.964
1.980
2.049
2.096
2.569
2.582
2.608
2.620
2.628
2.656
2.669
7.301
7.488
7.514
7.540
7.667
7.693
7.906
7.932
12.55
6.99
3.06
1.00
0.99
1.00
0.98
wxy-3-066-1
2018-10-18 17:13:43.671
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
Br
O
O
n-C6H17
I
6dg
PPM
10
8
6
4
2

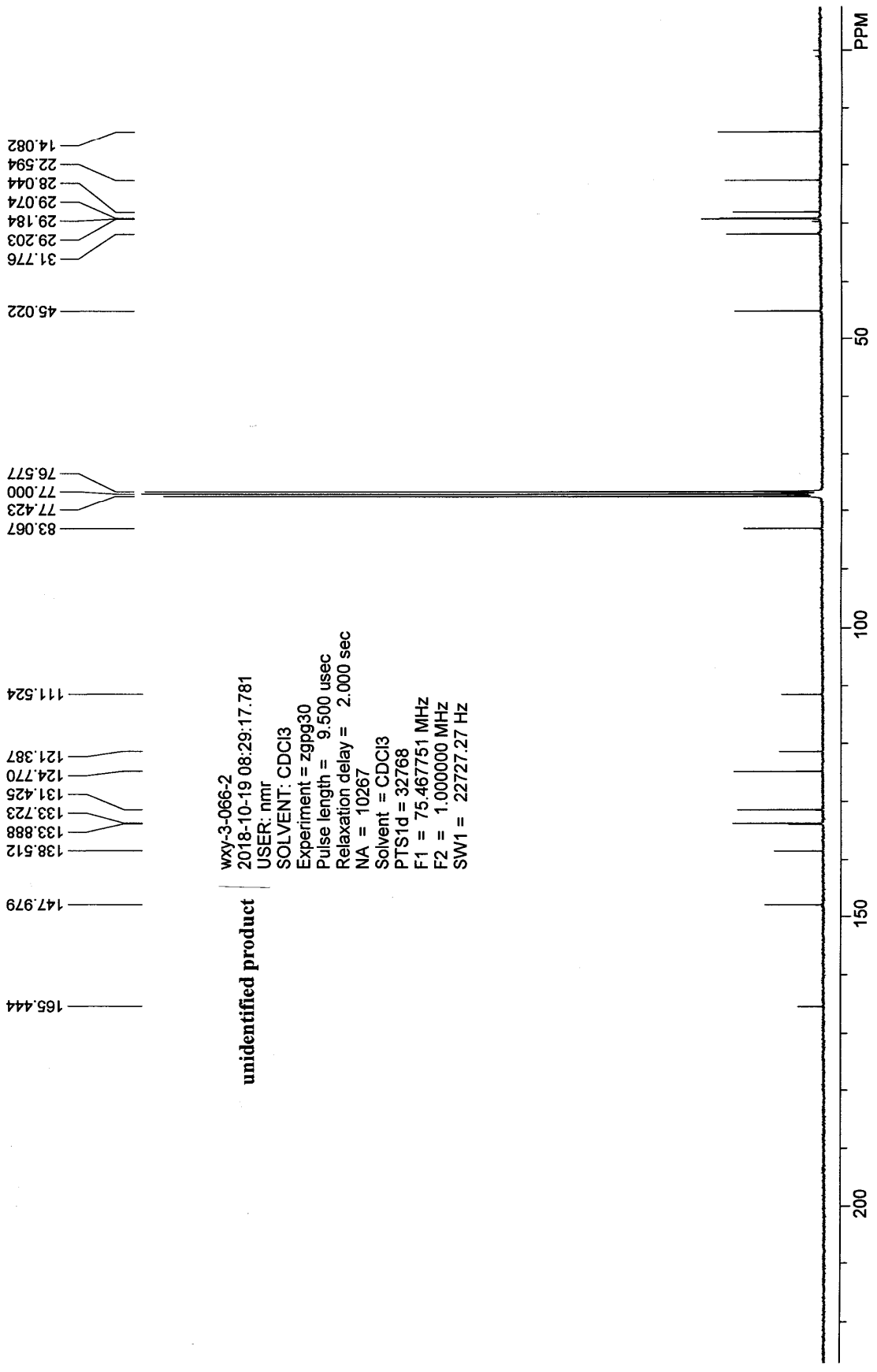

14.008
22.483
22.759
23.467
29.028
29.129
29.166
31.638
37.016
42.384
76.577
77.000
77.432
89.492
97.911
120.211
123.446
123.694
133.897
134.366
143.209
154.809
166.703
wxy-3-066
2018-10-18 17:07:47.515
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 255
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
Br
I
n-C8H17
6dg
250
200
150
100
50
0 PPM

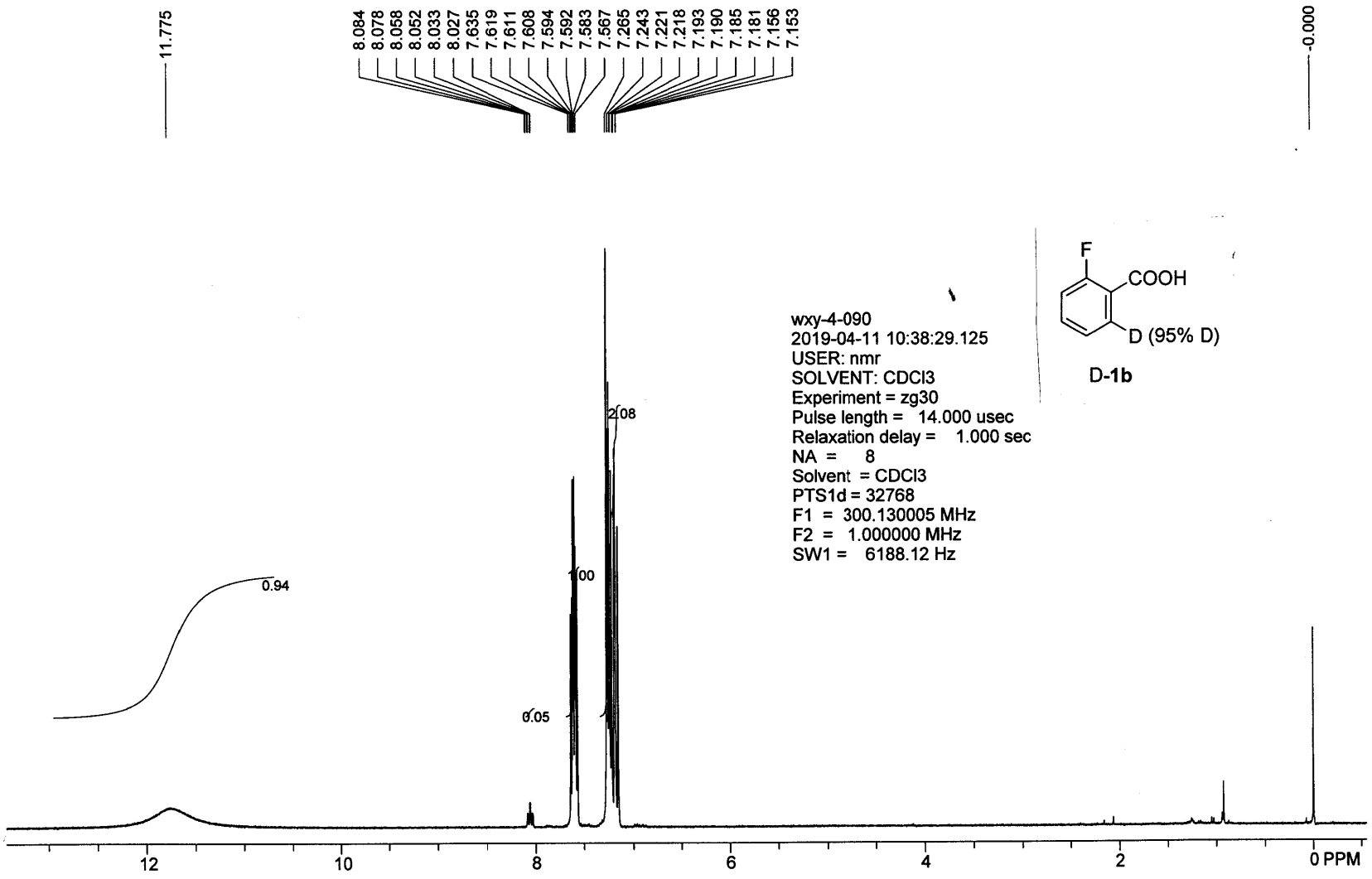
Purity (98%) is determined by mesitylene (11.0 μ L, 0.239 mmol) as the intrenal standard in 120.9 mg sample.

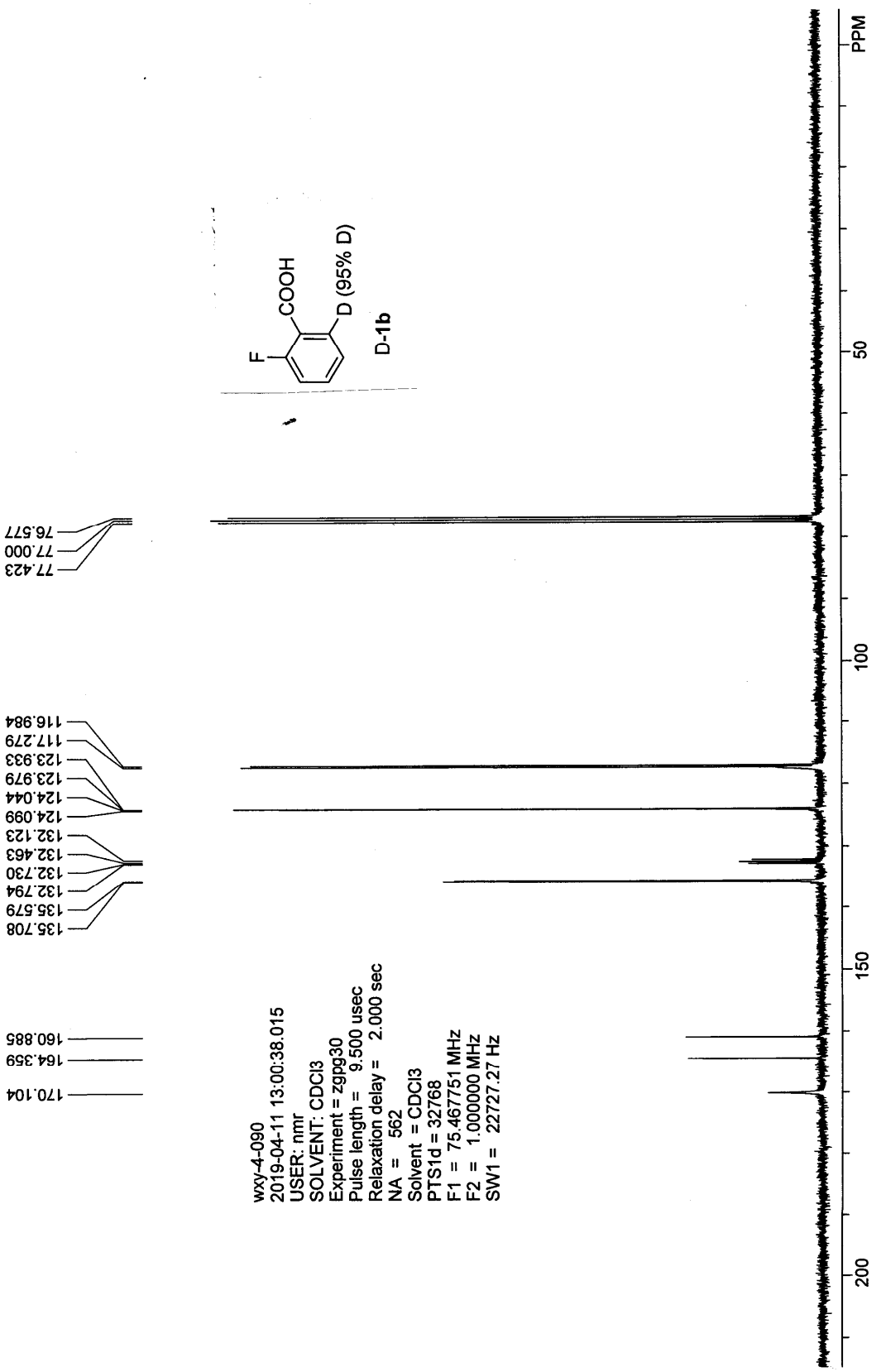


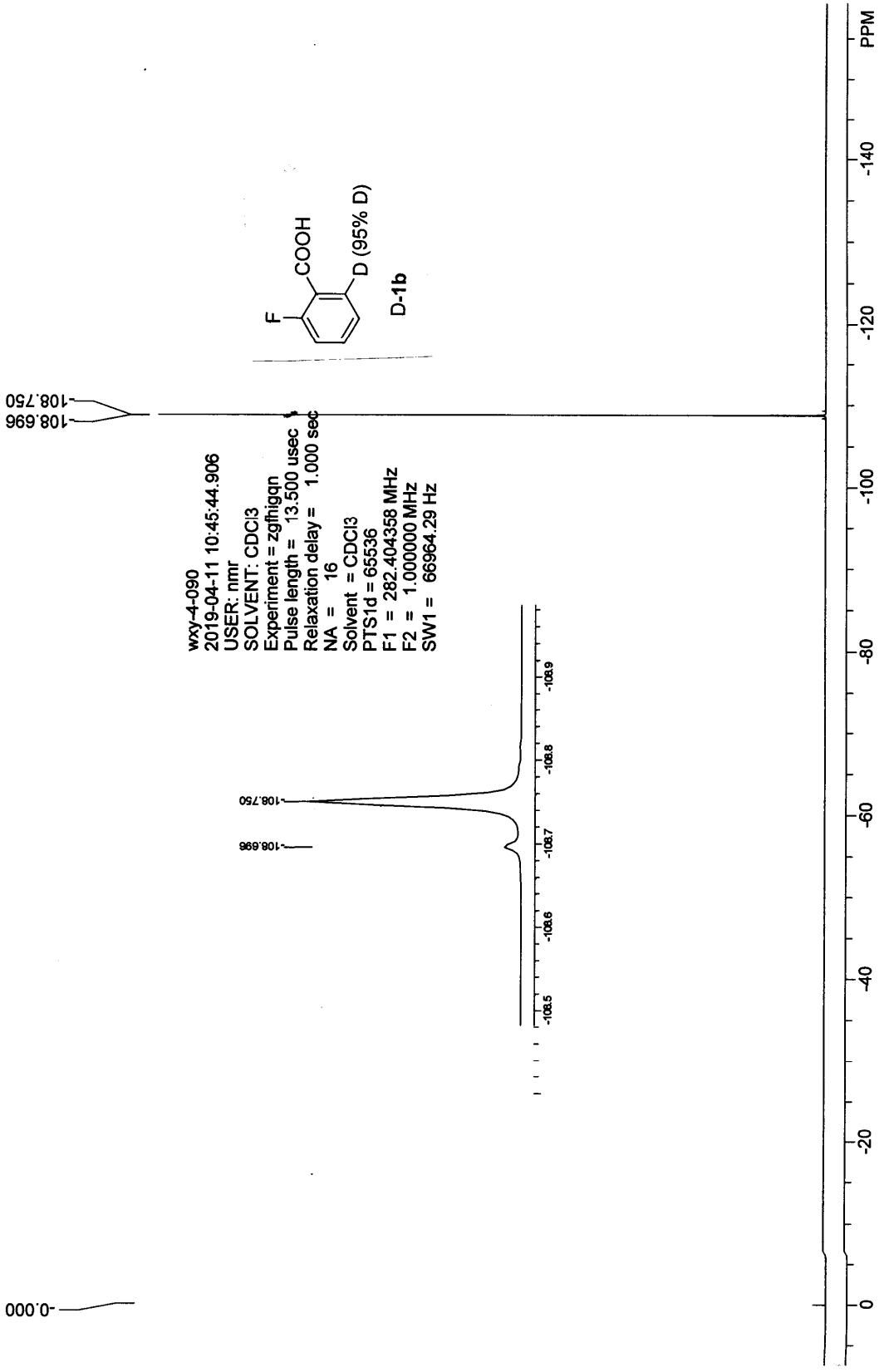
wxy-3-066-1-purity
2018-11-15 19:25:16.406
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz

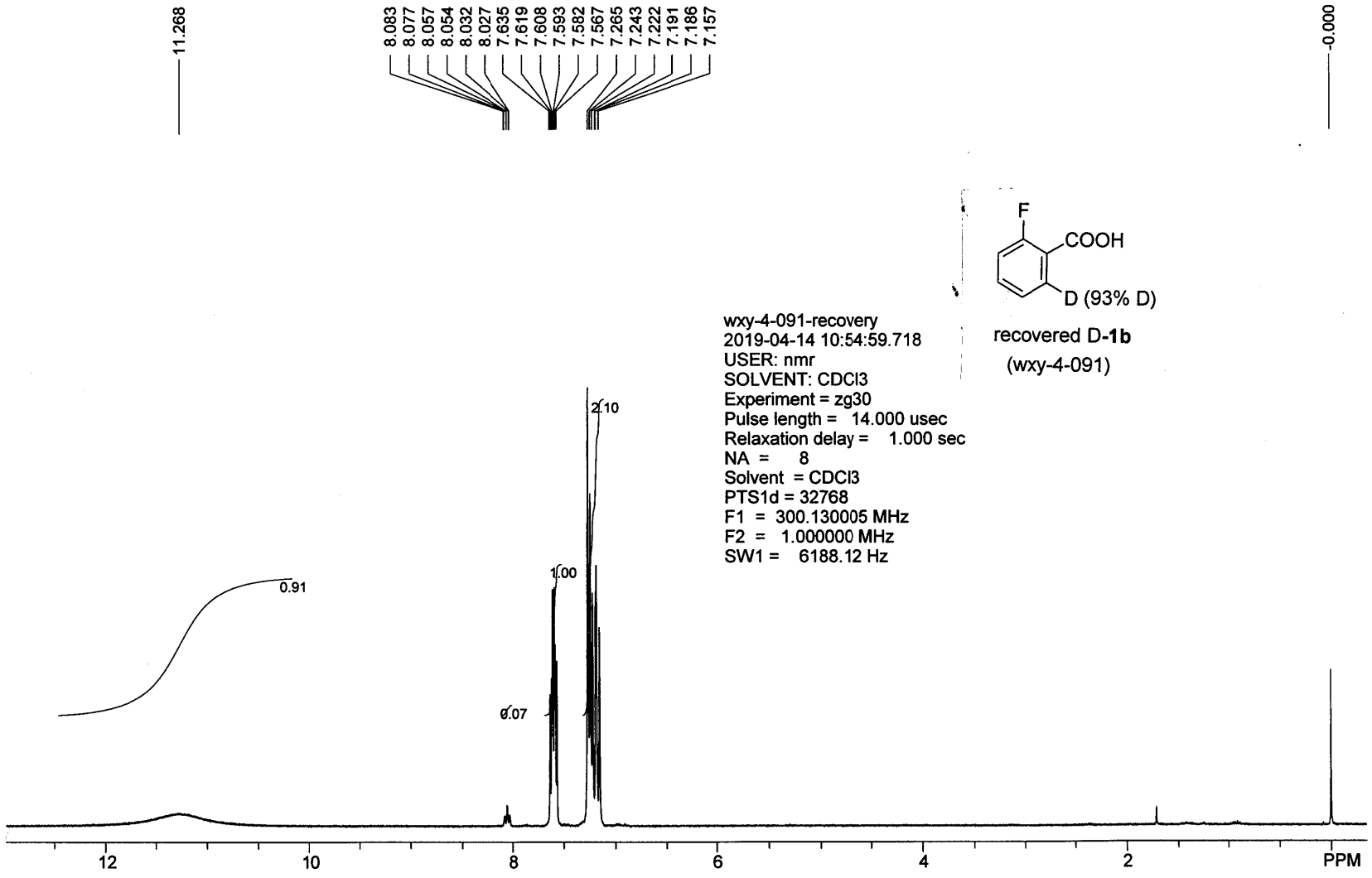

unidentified product
wxy-3-066-2
2018-10-18 17:20:09.578
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
21.01
3.36
2.00
2.04
1.02
PPM
0.000
0.838
0.861
0.883
1.223
1.252
1.368
1.409
1.447
1.601
2.847
2.879
2.900
7.268
7.309
7.336
7.346
7.356
7.372
7.633
7.649
7.660
7.669
7.679
7.694

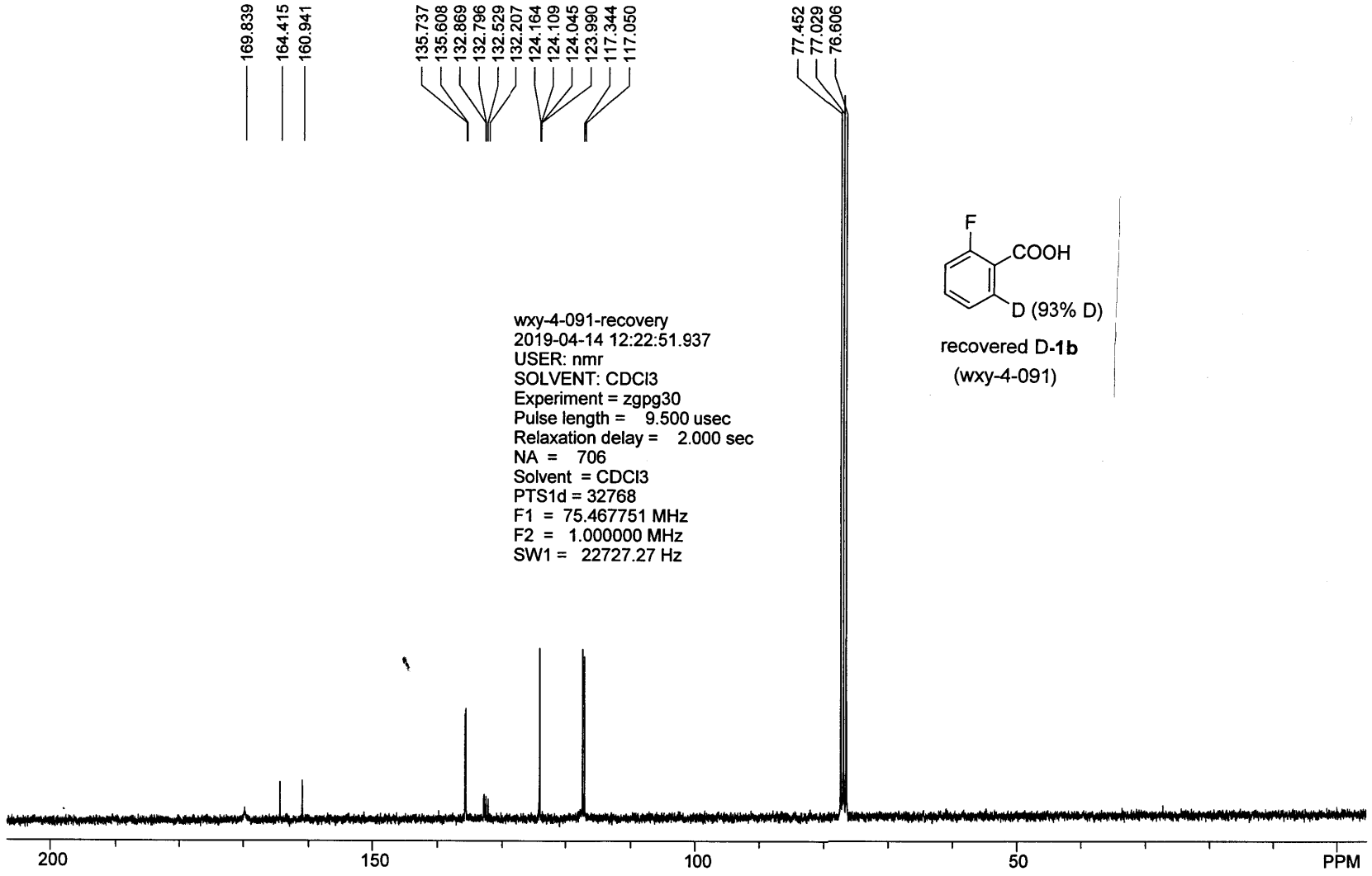

14.082
22.594
28.044
29.074
29.184
29.203
31.776
45.022
76.577
77.000
77.423
83.067
111.524
121.387
124.770
131.425
133.723
133.888
138.512
147.979
165.444
unidentified product
wxy-3-066-2
2018-10-19 08:29:17.781
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 10267
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
PPM
200
150
100
50

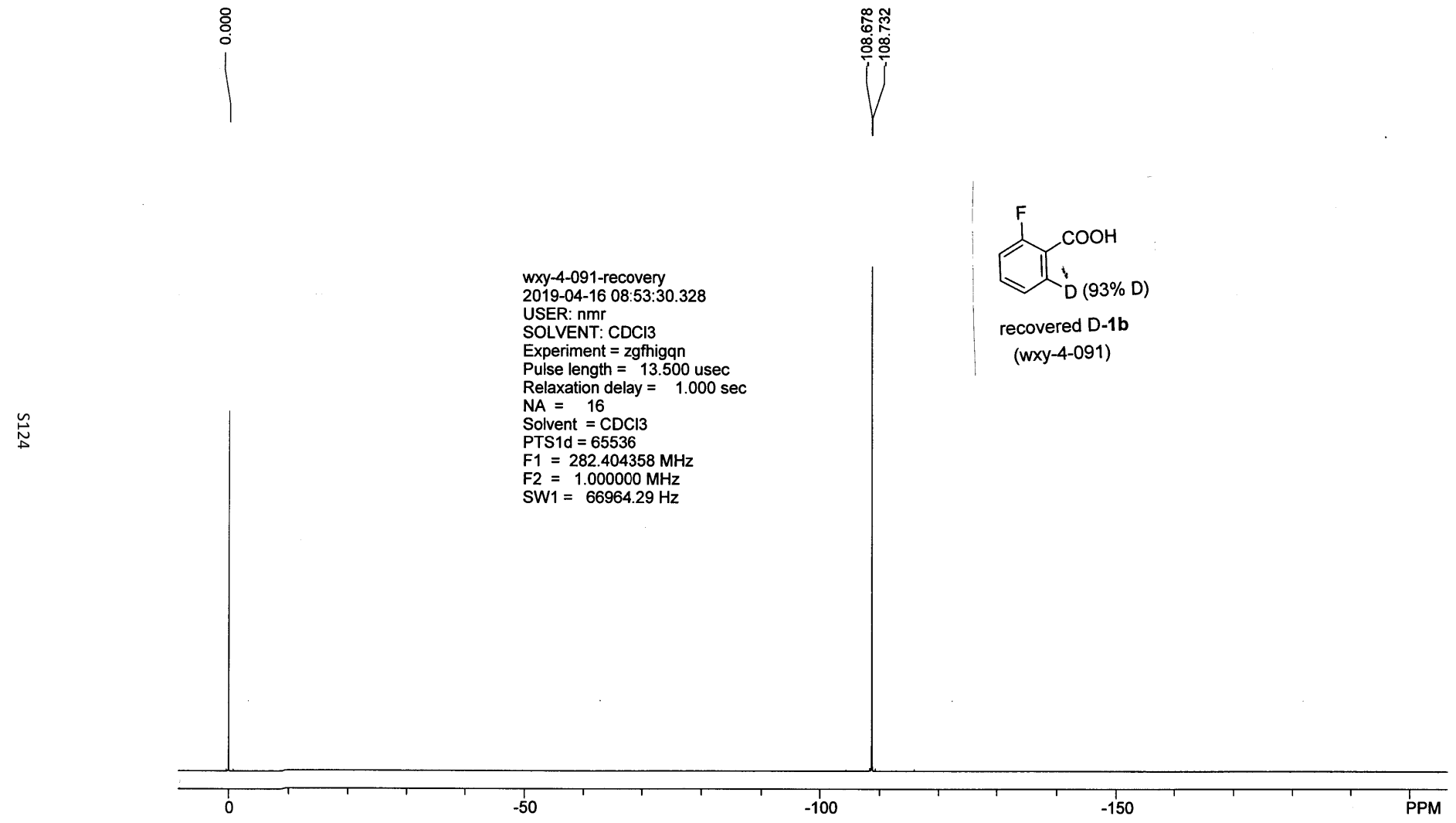

11.775
8.084
8.078
8.058
8.052
8.033
8.027
7.635
7.619
7.611
7.608
7.594
7.592
7.583
7.567
7.265
7.243
7.221
7.218
7.193
7.190
7.185
7.181
7.156
7.153
-0.000
wxy-4-090
2019-04-11 10:38:29.125
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
F
COOH
D (95% D)
D-1b
0.94
1.00
2.08
0.05
12
10
8
6
4
2
0 PPM


F
COOH
D (95% D)
D-1b
wxy-4-090
2019-04-11 13:00:38.015
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 562
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
170.104
164.359
160.885
135.708
135.579
132.794
132.730
132.463
132.123
124.099
124.044
123.979
123.933
117.279
116.984
77.423
77.000
76.577
PPM
200
150
100
50


wxy-4-090
2019-04-11 10:45:44.906
USER: nmr
SOLVENT: CDCl3
Experiment = zgfhigqn
Pulse length = 13.500 usec
Relaxation delay = 1.000 sec
NA = 16
Solvent = CDCl3
PTS1d = 65536
F1 = 282.404358 MHz
F2 = 1.000000 MHz
SW1 = 66964.29 Hz
F
COOH
D (95% D)
D-1b
-108.750
-108.696
-0.000
PPM


11.268
8.083
8.077
8.057
8.054
8.032
8.027
7.635
7.619
7.608
7.593
7.582
7.567
7.265
7.243
7.222
7.191
7.186
7.157
-0.000
F
COOH
D (93% D)
recovered D-1b
(wxy-4-091)
wxy-4-091-recovery
2019-04-14 10:54:59.718
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
0.91
0.07
1.00
2.10
12
10
8
6
4
2
PPM


169.839
164.415
160.941
135.737
135.608
132.869
132.796
132.529
132.207
124.164
124.109
124.045
123.990
117.344
117.050
77.452
77.029
76.606
wxy-4-091-recovery
2019-04-14 12:22:51.937
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 706
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
F
COOH
D (93% D)
recovered D-1b
(wxy-4-091)
200
150
100
50
PPM


0.000
108.678
108.732
wxy-4-091-recovery
2019-04-16 08:53:30.328
USER: nmr
SOLVENT: CDCl3
Experiment = zgfhigqn
Pulse length = 13.500 usec
Relaxation delay = 1.000 sec
NA = 16
Solvent = CDCl3
PTS1d = 65536
F1 = 282.404358 MHz
F2 = 1.000000 MHz
SW1 = 66964.29 Hz
F
COOH
D (93% D)
recovered D-1b
(wxy-4-091)
0
-50
-100
-150
PPM