Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Pd-catalyzed coupling reaction of cyclobutanols with propargylic carbonates

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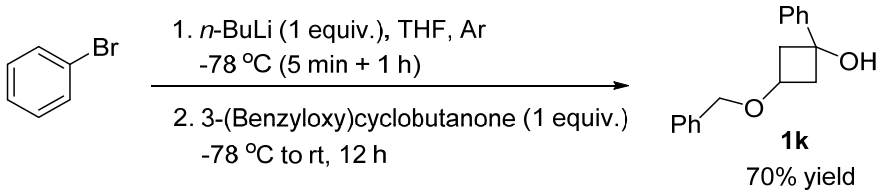
Supporting information

General Information. NMR spectra were taken with an Agilent-400 spectrometer (400 MHz for ^{1}H NMR, 100 MHz for ^{13}C NMR, and 376 MHz for ^{19}F NMR). Chemical shifts were recorded in ppm in relative to the TMS in $CDCl_3$ and $CFCl_3$ (^{19}F CPD, $\delta = 0$) as the internal standards, respectively, and coupling constants were reported in Hz. All reactions were carried out in flame-dried Schlenk tubes under an argon atmosphere. $Pd(dba)_2$ was purchased from TCI Chemicals; XPhos was purchased from Energy Chemical; $K_3PO_4 \cdot 3H_2O$ was purchased from Sinopharm Reagent Co., Ltd.; Toluene, THF, and dioxane were dried over sodium wire with benzophenone as the indicator and distilled freshly before use. DCM and CH_3CN were dried over CaH_2 and distilled freshly before use. DMF was dried over $MgSO_4$ and distilled before use. Petroleum ether (60 ^{o}C - 90 ^{o}C) was used for chromatography. All the temperatures are referred to the oil baths, acetone/dry ice bath, or ice/water bath used. NMR yields and recovery of starting material were determined by ^{1}H NMR analysis of the related reaction mixtures using dibromomethane as the internal standard. Cyclobutanols **1a-1g**,[1a] **1h**,[1b] **1i**,[1c] **1j**,[1d] **1k**,[1a] were synthesized according to the reported procedures. **1l** was purchased from Bide Pharmatech Ltd. Propargylic carbonates **2a**,[2a] **2b**,[2b] **2c**,[2b] **2e**,[2b] **2f**,[2c] **2h**,[2d] **2k**,[2d] were synthesized from propargylic alcohols according to the reported procedures.

Experimental details and analytical data

1. Synthesis of cyclobutanol 1k.

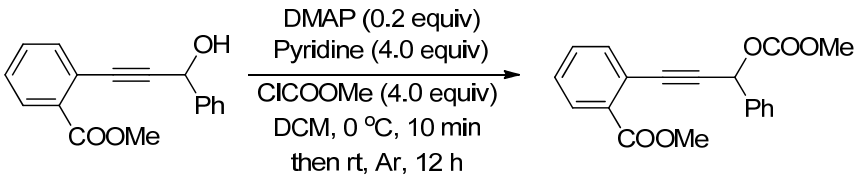
Synthesis of 3-(benzyloxy)-1-phenylcyclobutanol (**1k**)[1a] (wpl-4-190)



To a flame-dried three-necked flask charged with bromobenzene (0.42 mL, d = 1.491 g/mL, 0.6262 g, 4 mmol) and 10 mL of THF was cooled to -78 °C. Then *n*-BuLi (1.6 mL, 2.5 M in hexane, 4.0 mmol, 1.0 equiv.) was added dropwise via a syringe within 5 min under Ar. The resulting mixture was stirred for 1.0 h at -78 °C followed by the addition of 3-(benzyloxy)cyclobutanone (0.7037 g, 4.0 mmol, 1.0 equiv.). The resulting mixture was naturally warmed up to room temperature and stirred for 12 h. Then the mixture was cooled to 0 °C with an ice/water bath and quenched with a saturated solution of NH_4Cl (aq) (10 mL), extracted with ethyl acetate (10 mL × 3), washed with a saturated solution of NaCl (aq) (20 mL), dried over anhydrous Na_2SO_4, filtered, and concentrated. The residue was purified by column chromatography on silica gel [eluent: petroleum ether/ethyl acetate = 20/1 (0.4 L) to 5/1 (0.4 L)] to afford **1k** (0.7121 g, 70%) as an oil: ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.47-7.41 (m, 2 H, Ar-H), 7.38-7.21 (m, 8 H, Ar-H), 4.43 (s, 2 H, OCH_2), 3.82 (quint, *J* = 6.7 Hz, 1 H, CH), 2.95-2.86 (m, 2 H, CH_2), 2.58 (s, 1 H, OH), 2.48-2.39 (m, 2 H, CH_2); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 145.4, 138.0, 128.5, 128.4, 127.8, 127.7, 127.3, 125.1, 70.4, 70.2, 65.3, 45.2; IR (neat, cm^{-1}): ν = 3405, 3061, 3029, 2984, 2937, 2864, 1602, 1495, 1447, 1348, 1237, 1154, 1094, 1049, 1026; MS (70 eV, EI) *m/z* (%): 163 [(M^+-CH_2Ph), 22.0], 91 (100); HRMS Calcd for $C_{10}H_{11}O_2$ (M^+-CH_2Ph): 163.0759; Found: 163.0751.

2. Synthesis of propargylic carbonates 2d, 2g, 2i and 2j.

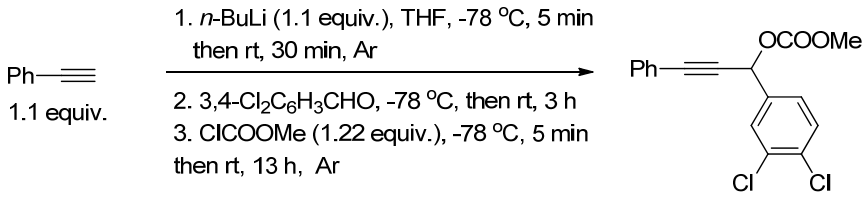
(1) Synthesis of 3-(2-(methoxycarbonyl)phenyl)-1-phenylprop-2-ynyl methyl carbonate (**2d**)[2a] (wpl-2-98)



2d
79% yield

To a three-necked flask were added 3-(2-(methoxycarbonyl)phenyl)-1-phenylprop-2-ynol[2f] (1.3401 g, 5.0 mmol), CH_2Cl_2 (10 mL), and DMAP (0.1220 g, 1.0 mmol) under Ar. After being cooled to 0 °C with stirring, pyridine (1.6 mL, d = 0.983 g/mL, 1.5728 g, 19.9 mmol) was added. Methyl chloroformate (1.5 mL, d = 1.223 g/mL, 1.8345 g, 19.4 mmol) was then added dropwise slowly within 10 minutes. The reaction was naturally warmed up to room temperature and stirred overnight (12 h). The resulting mixture was then diluted with CH_2Cl_2 (20 mL), and this CH_2Cl_2 solution was washed sequentially with water (20 mL × 3), 1 mol/L HCl (aq) (20 mL × 3), a saturated solution of NaCl (aq) (30 mL), dried over anhydrous Na_2SO_4, filtered, and concentrated. The residue was purified by column chromatography on silica gel [eluent: petroleum ether/ethyl acetate = 20/1 (0.8 L) to 10/1 (0.8 L)] to afford **2d** (1.2960 g, 79%) as an oil: ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.94 (dd, *J* = 8.0 Hz, 1.2 Hz, 1 H, Ar-H), 7.71-7.63 (m, 2 H, Ar-H), 7.59 (dd, *J* = 7.6 Hz, 1.0 Hz, 1 H, Ar-H), 7.51-7.34 (m, 5 H, Ar-H), 6.58 (s, 1 H, OCH), 3.829 (s, 3 H, CH_3), 3.826 (s, 3 H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 166.5, 154.9, 136.4, 134.2, 132.3, 131.6, 130.4, 129.2, 128.7, 128.6, 127.9, 122.2, 89.5, 86.6, 70.3, 55.0, 52.0; IR (neat, cm^{-1}): ν = 2953, 1747, 1728, 1595, 1568, 1486, 1439, 1250, 1191, 1129, 1083, 1044, 1009; MS (70 eV, EI) *m/z* (%): 324 [M^+, 3.19], 233 (100); HRMS Calcd for $C_{19}H_{16}O_5$ (M^+): 324.0998; Found: 324.1001.

(2) Synthesis of 1-(3,4-dichlorophenyl)-3-phenylprop-2-ynyl methyl carbonate (**2g**) (wpl-2-92)

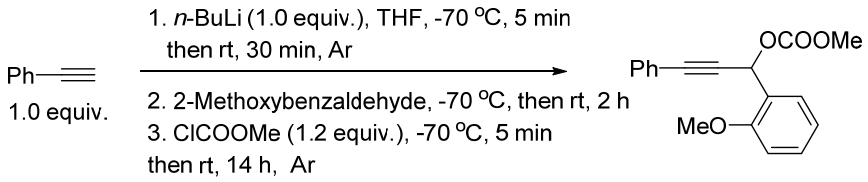


2g
50% yield

To a three-necked bottle were added THF (40 mL) and phenylacetylene (4.4 mL, d = 0.93 g/mL, 4.092 g, 40.1 mmol). After being cooled with an acetone/dry ice bath to -78 °C with stirring, *n*-BuLi (16.0 mL, 2.5 M in hexane, 40.0 mmol) was added dropwise in 5 minutes. Then the acetone/dry ice bath was removed, and reaction was stirred at room temperature for 30 minutes. The resulting mixture was cooled to -78 °C with an acetone/dry ice bath again, followed by addition of 3,4-dichlorobenzaldehyde (6.3 g, 36.0 mmol). The acetone/dry ice bath was removed, and the reaction was stirred for 3 hours at room temperture. Then the reaction was cooled to -78 °C again with acetone/dry ice bath, and methyl chloroformate (3.4 mL, d = 1.223 g/mL, 4.1582 g, 44.0 mmol) was added slowly in 5 minutes. Then the acetone/dry ice bath was removed, the resulting mixture was stirred at room temperature for 13 hours, then cooled to 0 °C with an ice/water bath and quenched with H_2O (40 mL). The reaction was extracted with ethyl acetate (30 mL × 3), washed with a saturated solution of NaCl (aq) (40 mL), dried over anhydrous Na_2SO_4, filtered, and concentrated. The residue was purified by column chromatography on silica gel [eluent: petroleum ether/ethyl acetate = 80/1 (1.6 L)] to afford **2g** (6.0213 g, 50%) as an oil: ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.71 (d, *J* = 2.0 Hz, 1 H, Ar-H), 7.52-7.41 (m, 4 H, Ar-H), 7.40-7.28 (m, 3 H, Ar-H), 6.47 (s, 1 H, OCH), 3.83 (s, 3 H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 154.7, 136.7, 133.4, 132.9, 131.9, 130.7, 129.7, 129.2, 128.3, 127.0, 121.4, 88.7, 83.7, 68.7, 55.3; IR (neat, cm^{-1}): ν = 2956, 2232, 1747, 1597, 1566, 1491, 1470, 1440, 1401, 1329, 1307, 1253, 1194, 1131, 1098, 1070, 1032, 1013; MS (70 eV, EI) *m/z* (%): 338 [M^+($^{37,37}Cl_2$), 3.16], 336 [M^+($^{37,35}Cl_2$), 18.58], 334 [M^+($^{35,35}Cl_2$), 27.63], 259 (100); HRMS Calcd for $C_{17}H_{12}{}^{35,35}Cl_2O_3$ (M^+): 334.0163; Found: 334.0166.

(3) Synthesis of 1-(2-methoxy)-3-phenylprop-2-ynyl methyl carbonate (**2i**)

(wpl-4-180)

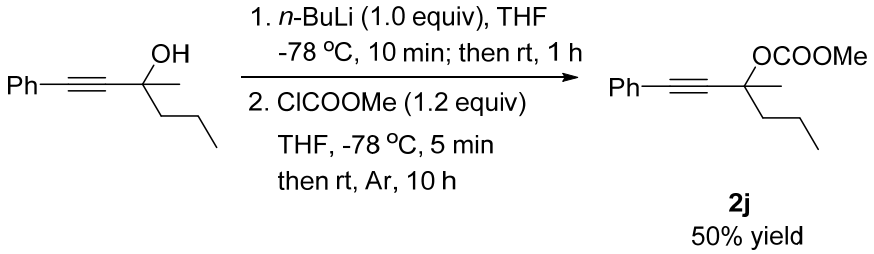


2i
22% yield

To a three-necked flask were added THF (60 mL) and phenylacetylene (4.4 mL, d = 0.93 g/mL, 4.092 g, 40.1 mmol) under Ar. After being cooled to -70 °C with an ethanol/dry ice bath with stirring, *n*-BuLi (16.0 mL, 2.5 M in hexane, 40.0 mmol) was added dropwise within 5 minutes. Then the ethanol/dry ice bath was removed, and reaction was stirred at room temperature for 30 minutes. The resulting mixture was cooled to -70 °C with an ethanol/dry ice bath again, followed by addition of 2-methoxybenzaldehyde (5.46 g, 40.0 mmol). The ethanol/dry ice bath was removed, and the reaction was stirred for 2 hours at room temperture. Then the reaction was cooled to -70 °C again with an ethanol/dry ice bath, and methyl chloroformate (3.7 mL, d = 1.223 g/mL, 4.5251 g, 48.0 mmol) was added slowly within 5 minutes. Then the ethanol/dry ice bath was removed, and the resulting mixture was stirred at room temperature for 14 hours, cooled to 0 °C with an ice/water bath, and quenched with a saturated solution of NH_4Cl (aq) (50 mL). The reaction was extracted with ethyl acetate (30 mL × 3), washed with a saturated solution of NaCl (aq) (30 mL), dried over anhydrous Na_2SO_4, filtered, and concentrated. The residue was purified by recrystallize (petroleum ether/dichloromethane) to afford **2i** (2.5716 g, 22%) as a white soild: m.p. = 70.1-71.0 °C (petroleum ether/dichloromethane); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.78-7.73 (m, 1 H, Ar-H), 7.51-7.44 (m, 2 H, Ar-H), 7.38-7.27 (m, 4 H, Ar-H), 7.01 (t, *J* = 7.4 Hz, 1 H, Ar-H), 6.92 (s, 1 H, CH), 6.91 (d, *J* = 7.6 Hz, 1 H, Ar-H), 3.87 (s, 3 H, OCH_3), 3.82 (s, 3 H, OCH_3); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 156.8, 154.9, 131.9, 130.6, 129.1, 128.7, 128.2, 124.6, 122.2, 120.6, 110.8, 87.3, 85.0, 65.0, 55.6, 54.9; IR (neat, cm^{-1}): ν = 2961, 2939, 2841, 2232, 1746, 1602, 1587, 1491, 1464, 1440, 1342, 1318, 1286, 1267, 1248, 1239, 1173, 1162, 1114, 1094, 1073, 1050,

1022, 1007; MS (70 eV, EI) *m/z* (%): 296 (M^+, 22.87), 115 (100); Anal. Calcd for $C_{18}H_{16}O_4$: C 72.96, H 5.44; Found: C 72.89, H 5.45.

(4) Synthesis of 3-methyl-1-phenyl-1-hexyn-3-yl methyl carbonate (**2j**) (wpl-2-124)



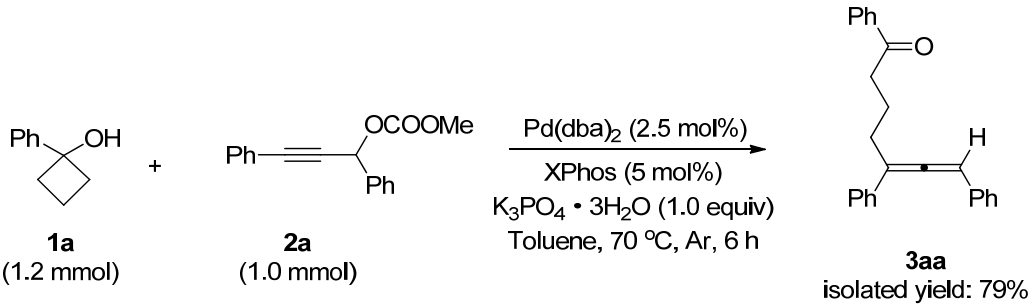
2j
50% yield

To a three-necked bottle were added THF (30 mL) and 3-methyl-1-phenyl-1-hexyn-3-ol[2g] (1.8824 g, 10.0 mmol). The reaction was cooled to -78 °C with an acetone/dry ice bath with stirring, *n*-BuLi (4.0 mL, 2.5 M in hexane, 10.0 mmol) was added dropwise in 10 minutes. The acetone/dry ice bath was removed and stirred at room temperature for one hour. The reaction was cooled to -78 °C with acetone/dry ice bath again, and methyl chloroformate (0.95 mL, d = 1.223 g/mL, 1.1618 g, 12.3 mmol) was added dropwise slowly in 5 minutes. The acetone/dry ice bath was removed and stirred at room temperature for 10 hours. The reaction mixture was cooled to 0 °C with an ice/water bath and quenched with H_2O (20 mL). The reaction was extracted with ethyl acetate (20 mL × 3), washed with a solution of saturated NaCl (aq) (30 mL), dried over anhydrous Na_2SO_4, filtered, and concentrated. The residue was purified by column chromatography on silica gel [eluent: petroleum ether/ethyl acetate = 60/1 (1.2 L)] to afford **2j** (1.2301 g, 50%) as an oil: ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.48-7.39 (m, 2 H, Ar-H), 7.35-7.24 (m, 3 H, Ar-H), 3.77 (s, 3 H, OCH_3), 2.10-1.97 (m, 1 H, one proton of CH_2), 1.96-1.82 (m, 1 H, one proton of CH_2), 1.79 (s, 3 H, CH_3), 1.70-1.50 (m, 2 H, CH_2), 0.98 (t, *J* = 7.2 Hz, 3 H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 153.5, 131.8, 128.4, 128.1, 122.4, 88.6, 85.4, 77.9, 54.2, 43.7, 26.4, 17.6, 14.0; IR (neat, cm^{-1}): ν = 2960, 2874, 1750, 1599, 1490, 1440, 1375, 1320, 1257, 1188, 1149, 1133, 1115, 1072, 1046, 1027, 1014; MS (70 eV, EI) *m/z* (%): 246 [M^+, 5.08], 142 (100); HRMS Calcd for $C_{15}H_{18}O_3$ (M^+):

246.1256; Found: 246.1253 .

3. Palladium-catalyzed synthesis of δ-allenyl ketones

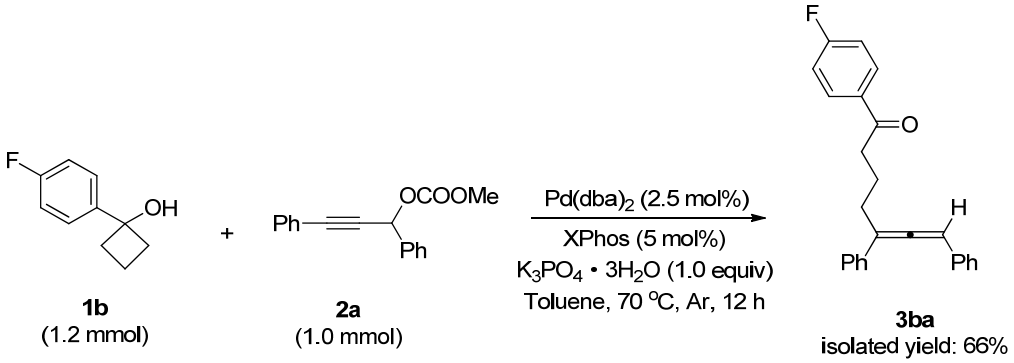
(1) Preparation of 1,5,7-triphenylhepta-5,6-dien-1-one (**3aa**) (wpl-2-79)



1a (1.2 mmol) **2a** (1.0 mmol) **3aa** isolated yield: 79%

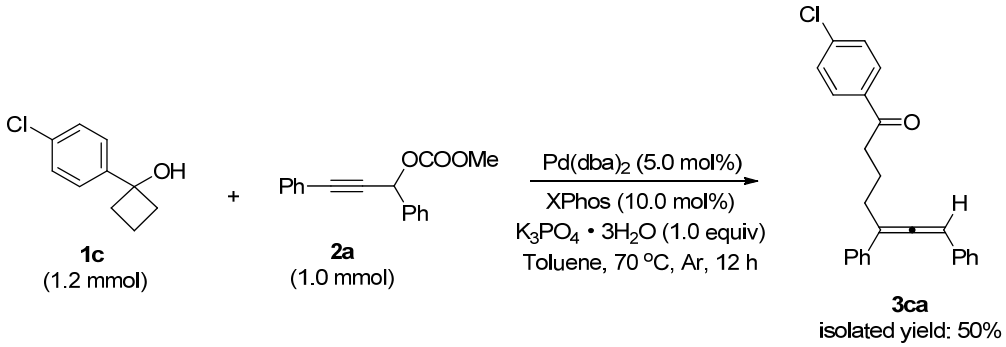
Typical Procedure A: To a flame-dried Schlenk tube were added $Pd(dba)_2$ (14.4 mg, 0.025 mmol), XPhos (23.7 mg, 0.05 mmol), and $K_3PO_4 \cdot 3H_2O$ (266.3 mg, 1.0 mmol) under Ar atmosphere. Then **1a** (soild, 177.6 mg, 1.2 mmol), **2a** (266.2 mg, 1.0 mmol)/toluene (1.0 mL), and toluene (4.0 mL) were then added sequentially under Ar atmosphere at room temperature. The Schlenk tube was then stirred at 70 ^{o}C until completion of the reaction as monitored by TLC and ^{1}H NMR (6 h). The crude reaction mixture was filtrated through a short column of silica gel (height: 3 cm, *Φ*: 3.5 cm) eluted with ethyl acetate (60 mL). After evaporation, the residue was purified by chromatography on silica gel to afford **3aa** (267.3 mg, 79%) [eluent: petroleum ether/ethyl acetate = 60/1 (2.0 L)] as a white solid: m.p. = 60.6-66.1 ^{o}C (petroleum ether/dichloromethane); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.93-7.80 (m, 2 H, Ar-H), 7.57-7.43 (m, 3 H, Ar-H), 7.42-7.16 (m, 10 H, Ar-H), 6.56 (t, *J* = 3.0 Hz, 1 H, =CH), 3.06 (t, *J* = 7.2 Hz, 2 H, CH_2CO), 2.76-2.60 (m, 2 H, CH_2), 2.18-1.98 (m, 2 H, CH_2); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 206.4, 199.9, 136.9, 135.7, 134.4, 132.9, 128.7, 128.53, 128.48, 128.0, 127.2, 127.1, 126.8, 126.1, 109.3, 98.3, 38.0, 29.6, 22.2; IR (neat, cm^{-1}): ν = 3057, 2957, 2891, 2843, 1934, 1680, 1594, 1578, 1490, 1460, 1445, 1404, 1370, 1339, 1285, 1268, 1229, 1196, 1174, 1156, 1072, 1028; MS (70 eV, EI) *m/z* (%): 338 (M^+, 1.61), 220 (100); Anal. Calcd for $C_{25}H_{22}O$: C 88.72, H 6.55; Found: C 88.57, H 6.62.

(2) Preparation of 1-(4-fluorophenyl)-5,7-diphenylhepta-5,6-dien-1-one (**3ba**) (wpl-2-73)



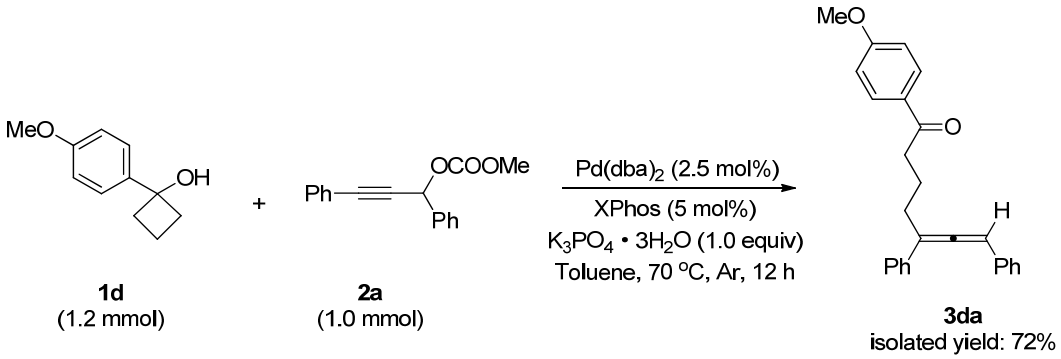
Typical Procedure B: To a flame-dried Schlenk tube were added $Pd(dba)_2$ (14.5 mg, 0.025 mmol), XPhos (23.6 mg, 0.05 mmol), and $K_3PO_4 \cdot 3H_2O$ (266.7 mg, 1.0 mmol) under Ar atmosphere. Then **1b** (liquid, 199.1 mg, 1.2 mmol) in toluene (1.0 mL), **2a** (266.0 mg, 1.0 mmol) in toluene (1.0 mL), and toluene (3.0 mL) were then added sequentially under Ar atmosphere at room temperature. The Schlenk tube was stirred at 70 °C until completion of the reaction as monitored by TLC and ^{1}H NMR (12 h). The crude reaction mixture was filtrated through a short column of silica gel (height: 3 cm, *Φ*: 3.5 cm) eluted with ethyl acetate (60 mL). After evaporation, the residue was purified by chromatography on silica gel to afford **3ba** (235.6 mg, 66%) [eluent: petroleum ether/ethyl acetate = 80/1 (1.2 L)] as a white solid: m.p. = 80.6-81.3 °C (petroleum ether/dichloromethane); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.92-7.80 (m, 2 H, Ar-H), 7.47 (d, *J* = 7.6 Hz, 2 H, Ar-H), 7.38-7.15 (m, 8 H, Ar-H), 7.04 (t, *J* = 8.6 Hz, 2 H, Ar-H), 6.56 (t, *J* = 3.0 Hz, 1 H, =CH), 3.10-2.94 (m, 2 H, CH_2CO) 2.76-2.60 (m, 2 H, CH_2), 2.17-1.97 (m, 2 H, CH_2); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 206.4, 198.3, 165.5 (d, *J* = 252.8 Hz), 135.7, 134.3, 133.3 (d, *J* = 2.3 Hz), 130.6 (d, *J* = 9.1 Hz), 128.8, 128.5, 127.19, 127.14, 126.8, 126.0, 115.5 (d, *J* = 21.3 Hz), 109.2, 98.3, 37.8, 29.5, 22.1; ^{19}F NMR (376 MHz, $CFCl_3$): δ = -106.1; IR (neat, cm^{-1}): ν = 3030, 2957, 2902, 2865, 2843, 2825, 1932, 1677, 1592, 1504, 1491, 1461, 1445, 1406, 1369, 1328, 1298, 1264, 1229, 1194, 1160, 1151, 1098, 1072, 1029; MS (70 eV, EI) *m/z* (%): 356 (M^+, 3.68), 238 (100); Anal. Calcd for $C_{25}H_{21}FO$: C 84.24, H 5.94; Found: C 83.94, H 5.93.

(3) Preparation of 1-(4-chlorophenyl)-5,7-diphenylhepta-5,6-dien-1-one (**3ca**) (wpl-2-27)



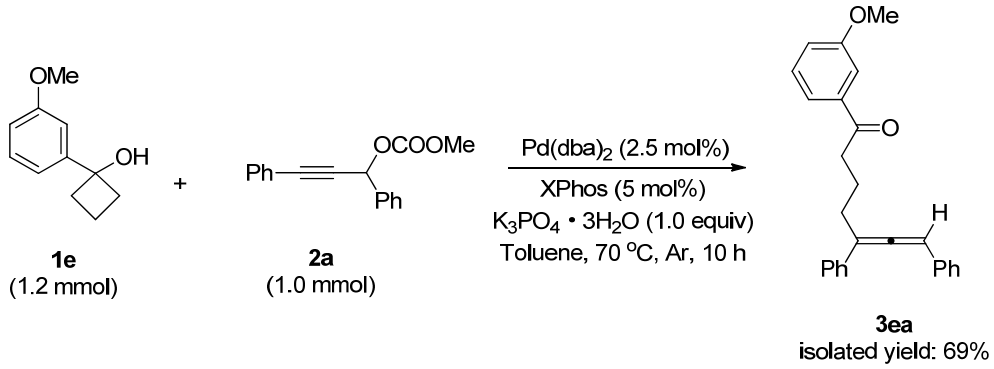
Following **Typical Procedure A**, the reaction of $Pd(dba)_2$ (28.9 mg, 0.05 mmol), XPhos (47.8 mg, 0.10 mmol), $K_3PO_4 \cdot 3H_2O$ (266.9 mg, 1.0 mmol), **1c** (soild, 219.1 mg, 1.2 mmol), **2a** (266.5 mg, 1.0 mmol)/toluene (1.0 mL), and toluene (4.0 mL) for 12 h afforded **3ca** (187.9 mg, 50%) [eluent: petroleum ether/ethyl acetate = 120:1 (1.0 L) for the first run, then all the products (245.5 mg) was collected and treated with the second run of chromatography, eluent: petroleum ether/ethyl acetate = 100/1 (0.8 L)] as an oil: ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.82-7.72 (m, 2 H, Ar-H), 7.47 (d, *J* = 7.6 Hz, 2 H, Ar-H), 7.40-7.18 (m, 10 H, Ar-H), 6.57 (t, *J* = 3.0 Hz, 1 H, =CH), 3.10-2.94 (m, 2 H, CH_2CO), 2.76-2.60 (m, 2 H, CH_2), 2.17-1.97 (m, 2 H, CH_2); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 206.4, 198.6, 139.2, 135.6, 135.2, 134.3, 129.3, 128.8, 128.7, 128.5, 127.2, 127.1, 126.7, 126.0, 109.2, 98.3, 37.9, 29.5, 22.0; IR (neat, cm^{-1}): ν = 3082, 3060, 3027, 2931, 2894, 1932, 1682, 1588, 1491, 1446, 1399, 1365, 1314, 1259, 1223, 1198, 1175, 1091, 1028, 1012; MS (70 eV, EI) *m/z* (%): 374 [M^+(^{37}Cl), 3.58], 372 [M^+(^{35}Cl), 10.17], 139 (100); HRMS Calcd for $C_{25}H_{21}{}^{35}ClO$ (M^+): 372.1281; Found: 372.1284.

(4) Preparation of 1-(4-methoxyphenyl)-5,7-diphenylhepta-5,6-dien-1-one (**3da**) (wpl-2-7)



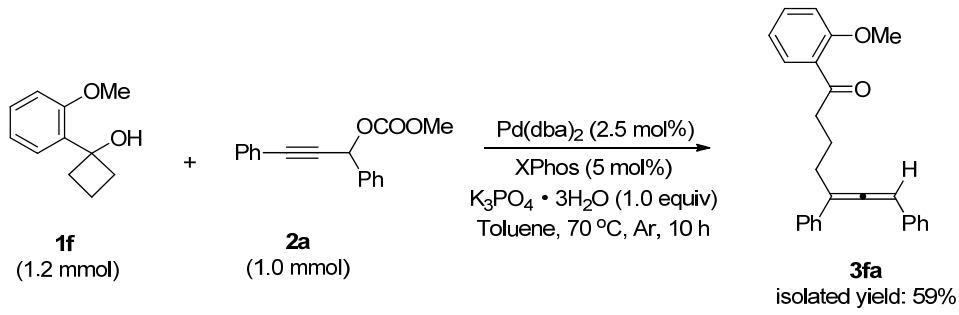
Following **Typical Procedure B**, the reaction of $Pd(dba)_2$ (14.5 mg, 0.025 mmol), XPhos (23.6 mg, 0.05 mmol), $K_3PO_4 \cdot 3H_2O$ (267.0 mg, 1.0 mmol), **1d** (liquid, 214.2 mg, 1.2 mmol)/toluene (1.0 mL), **2a** (266.4 mg, 1.0 mmol)/toluene (1.0 mL), and toluene (3.0 mL) for 12 h afforded **3da** (263.7 mg, 72%) [eluent: petroleum ether/ethyl acetate = 40/1 (0.4 L) to 30/1 (0.6 L)] as a yellow solid: m.p. = 88.9-89.4 ^oC (petroleum ether/ethyl acetate); ^1H NMR (400 MHz, $CDCl_3$) δ = 7.85 (d, *J* = 8.8 Hz, 2 H, Ar-H), 7.47 (d, *J* = 7.6 Hz, 2 H, Ar-H), 7.38-7.28 (m, 6 H, Ar-H), 7.26-7.18 (m, 2 H, Ar-H), 6.86 (d, *J* = 8.4 Hz, 2 H, Ar-H), 6.56 (t, *J* = 3.0 Hz, 1 H, =CH), 3.85 (s, 3 H, OMe), 3.02 (t, *J* = 7.2 Hz, 2 H, CH_2CO), 2.75-2.60 (m, 2 H, CH_2), 2.12-1.97 (m, 2 H, CH_2); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 206.4, 198.6, 163.3, 135.8, 134.4, 130.2, 130.0, 128.7, 128.5, 127.13, 127.08, 126.8, 126.1, 113.6, 109.3, 98.2, 55.4, 37.7, 29.7, 22.5; IR (neat, cm^{-1}): ν = 3025, 2953, 2894, 2842, 1933, 1672, 1599, 1574, 1507, 1493, 1459, 1447, 1417, 1370, 1346, 1311, 1259, 1201, 1182, 1105, 1073, 1024; MS (70 eV, EI) *m/z* (%): 368 (M^+, 2.23), 250 (100); Anal. Calcd for $C_{26}H_{24}O_2$: C 84.75, H 6.57; Found: C 84.45, H 6.61.

(5) Preparation of 1-(3-methoxyphenyl)-5,7-diphenylhepta-5,6-dien-1-one (**3ea**) (wpl-2-71)



Following **Typical Procedure A**, the reaction of $Pd(dba)_2$ (14.5 mg, 0.025 mmol), XPhos (23.6 mg, 0.05 mmol), $K_3PO_4 \cdot 3H_2O$ (266.2 mg, 1.0 mmol), **1e** (soild, 213.6 mg, 1.2 mmol), **2a** (265.9 mg, 1.0 mmol)/toluene (1.0 mL), and toluene (4.0 mL) for 10 h afforded **3ea** (251.2 mg, 69%) [eluent: petroleum ether/ethyl acetate = 80/1 (0.4 L) to 40/1 (0.8 L)] as an oil: ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.51-7.40 (m, 4 H, Ar-H), 7.38-7.18 (m, 9 H, Ar-H), 7.10-7.05 (m, 1 H, Ar-H), , 7.11-7.04 (m, 1 H, Ar-H), 6.56 (t, *J* = 3.0 Hz, 1 H, =CH), 3.82 (s, 3 H, OCH_3), 3.06 (t, *J* = 7.2 Hz, 2 H, CH_2CO), 2.75-2.60 (m, 2 H, CH_2), 2.18-1.98 (m, 2 H, CH_2); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 206.3, 199.6, 160.0, 138.2, 135.7, 134.3, 129.4, 128.7, 128.5, 127.1, 126.7, 126.0, 120.6, 119.3, 112.1, 109.2, 98.24, 98.16, 55.3, 38.0, 29.5, 22.3; IR (neat, cm^{-1}): ν = 2953, 2893, 1932, 1684, 1596, 1580, 1491, 1450, 1437, 1406, 1369, 1327, 1286, 1267, 1252, 1192, 1156, 1073, 1042; MS (70 eV, EI) *m/z* (%): 368 (M^+, 3.30), 135 (100); HRMS Calcd for $C_{26}H_{24}O_2$ (M^+): 368.1776; Found: 368.1772.

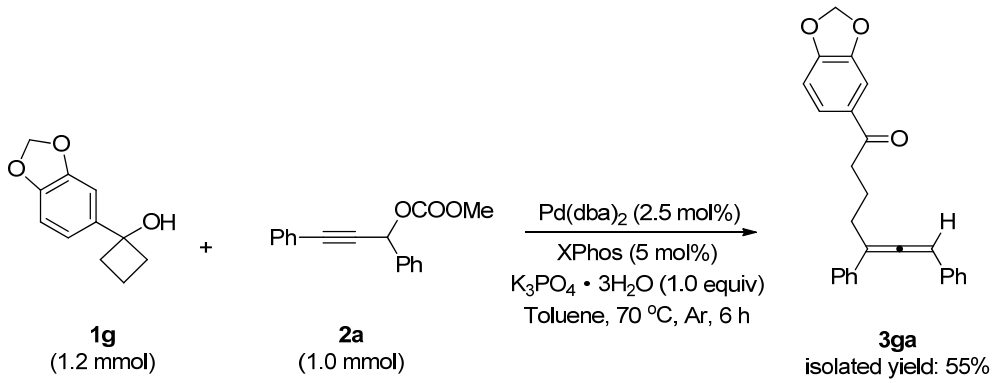
(6) Preparation of 1-(2-methoxyphenyl)-5,7-diphenylhepta-5,6-dien-1-one (**3fa**) (wpl-2-70)



Following **Typical Procedure B**, the reaction of $Pd(dba)_2$ (14.4 mg, 0.025 mmol), XPhos (23.9 mg, 0.05 mmol), $K_3PO_4 \cdot 3H_2O$ (266.4 mg, 1.0 mmol), **1f** (liquid, 213.5

mg, 1.2 mmol)/toluene (1.0 mL), **2a** (266.1 mg, 1.0 mmol)/toluene (1.0 mL), and toluene (3.0 mL) for 10 h afforded **3fa** (216.2 mg, 59%) [eluent: petroleum ether/ethyl acetate = 80/1 (0.4 L) to 40/1 (0.8 L)] as a white solid: m.p. = 76.0-76.7 ^{o}C (petroleum ether/dichloromethane); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.62 (dd, J_1 = 8.0 Hz, J_2 = 1.6 Hz, 1 H, Ar-H), 7.52-7.38 (m, 3 H, Ar-H), 7.36-7.12 (m, 8 H, Ar-H), 6.96 (t, J = 7.2 Hz, 1 H, Ar-H), 6.90 (d, J = 8.4 Hz, 1 H, Ar-H), 6.52 (t, J = 2.8 Hz, 1 H, =CH), 3.78 (s, 3 H, CH_3), 3.08 (t, J = 7.2 Hz, 2 H, CH_2CO), 2.67-2.53 (m, 2 H, CH_2), 2.11-1.95 (m, 2 H, CH_2); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 206.4, 202.4, 158.3, 135.8, 134.4, 133.1, 130.1, 128.6, 128.4, 127.0, 126.9, 126.7, 126.1, 120.5, 111.4, 109.5, 98.0, 55.3, 43.3, 29.7, 22.7; IR (neat, cm^{-1}): ν = 2969, 2933, 2901, 1927, 1659, 1593, 1493, 1481, 1462, 1447, 1434, 1392, 1373, 1315, 1277, 1236, 1188, 1158, 1112, 1077, 1021, 1002; MS (70 eV, EI) *m/z* (%): 368 (M^+, 4.45), 135 (100); Anal. Calcd for $C_{26}H_{24}O_2$: C 84.75, H 6.57; Found: C 84.37, H 6.59.

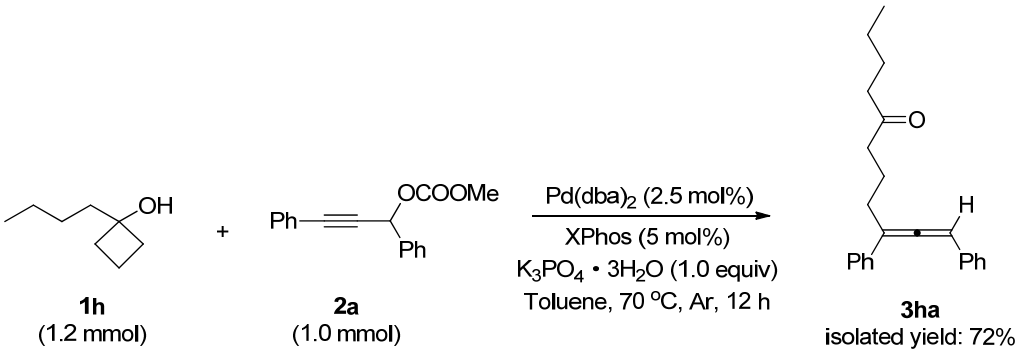
(7) Preparation of 1-(3,4-methylenedioxyphenyl)-5,7-diphenylhepta-5,6-dien-1-one (**3ga**) (wpl-2-82)



Following **Typical Procedure B**, the reaction of $Pd(dba)_2$ (14.4 mg, 0.025 mmol), XPhos (23.7 mg, 0.05 mmol), $K_3PO_4 \cdot 3H_2O$ (266.5 mg, 1.0 mmol), **1g** (liquid, 230.7 mg, 1.2 mmol)/toluene (1.0 mL), **2a** (266.0 mg, 1.0 mmol)/toluene (1.0 mL), and toluene (3.0 mL) for 6 h afforded **3ga** (210.3 mg, 55%) [eluent: petroleum ether/ethyl acetate = 40:1 (1.2 L) for the first run, then all the product (270.2 mg) was collected and treated with the second run of chromatography, eluent: dichloromethane/ether/petroleum ether = 1/1/15 (0.6 L), finally crystalized (petroleum

ether/dichloromethane) to provide the desired **3ga** as a yellow solid]: m.p. = 79.1-80.1 ^{o}C (petroleum ether/dichloromethane); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.53-7.18 (m, 12 H, Ar-H), 6.76 (d, *J* = 8.0 Hz, 1 H, Ar-H), 6.57 (t, *J* = 3.0 Hz, 1 H, =CH), 6.02 (s, 2 H, OCH_2O), 2.99 (t, *J* = 7.2 Hz, 2 H, CH_2CO), 2.77-2.58 (m, 2 H, CH_2), 2.17-1.95 (m, 2 H, CH_2); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 206.4, 198.0, 151.5, 148.1, 135.7, 134.4, 131.8, 128.7, 128.5, 127.14, 127.10, 126.8, 126.1, 124.2, 109.3, 107.8, 107.7, 101.7, 98.2, 37.7, 29.6, 22.4; IR (neat, cm^{-1}): ν = 2893, 2838, 1933, 1798, 1681, 1620, 1597, 1501, 1484, 1440, 1409, 1363, 1338, 1326, 1284, 1260, 1246, 1193, 1108, 1069, 1036; MS (70 eV, EI) *m/z* (%): 382 (M^+, 7.87), 264 (100); Anal. Calcd for $C_{26}H_{22}O_3$: C 81.65, H 5.80; Found: C 81.33, H 5.84.

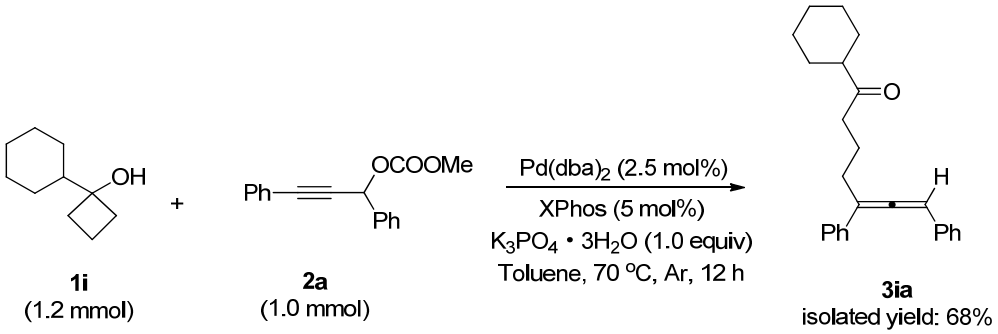
(8) Preparation of 9,11-diphenylundecan-9,10-dien-5-one **(3ha)** (wpl-2-39)



Following **Typical Procedure B**, the reaction of $Pd(dba)_2$ (14.4 mg, 0.025 mmol), XPhos (23.9 mg, 0.05 mmol), $K_3PO_4 \cdot 3H_2O$ (266.7 mg, 1.0 mmol), **1h** (liquid, 153.5 mg, 1.2 mmol)/toluene (1.0 mL), **2a** (266.4 mg, 1.0 mmol)/toluene (1.0 mL), and toluene (3.0 mL) for 12 h afforded **3ha** (229.2 mg, 72%) [eluent: petroleum ether/ethyl acetate = 80/1 (1.2 L)] as an oil: ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.45 (d, *J* = 8.0 Hz, 2 H, Ar-H), 7.37-7.15 (m, 8 H, Ar-H), 6.55 (bs, 1 H, =CH), 2.58 (t, *J* = 6.6 Hz, 2 H, CH_2), 2.50 (t, *J* = 7.2 Hz, 2 H, CH_2), 2.30 (t, *J* = 7.6 Hz, 2 H, CH_2), 1.98-1.80 (m, 2 H, CH_2), 1.56-1.42 (quint, *J* = 7.5 Hz, 2 H, CH_2), 1.33-1.18 (m, 2 H, CH_2), 0.86 (t, *J* = 7.4 Hz, 3 H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 211.0, 206.3, 135.7, 134.3, 128.7, 128.5, 127.1, 127.0, 126.7, 126.0, 109.2, 98.2, 42.4, 42.0, 29.4, 25.8, 22.2, 21.9, 13.8; IR (neat, cm^{-1}): ν = 2955, 2930, 2871, 1932, 1708, 1596, 1493,

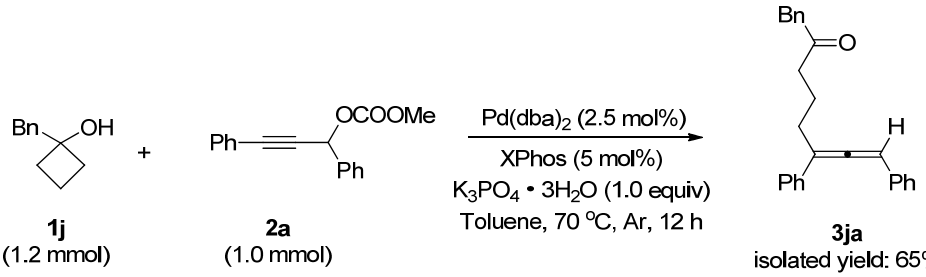
1447, 1408, 1370, 1257, 1126, 1073, 1027; MS (70 eV, EI) *m/z* (%): 318 (M^+, 6.43), 200 (100); HRMS Calcd for $C_{23}H_{26}O$ (M^+): 318.1984; Found: 318.1986.

(9) Preparation of 1-cyclohexyl-5,7-diphenylhepta-5,6-dien-1-one (**3ia**) (wpl-2-48)



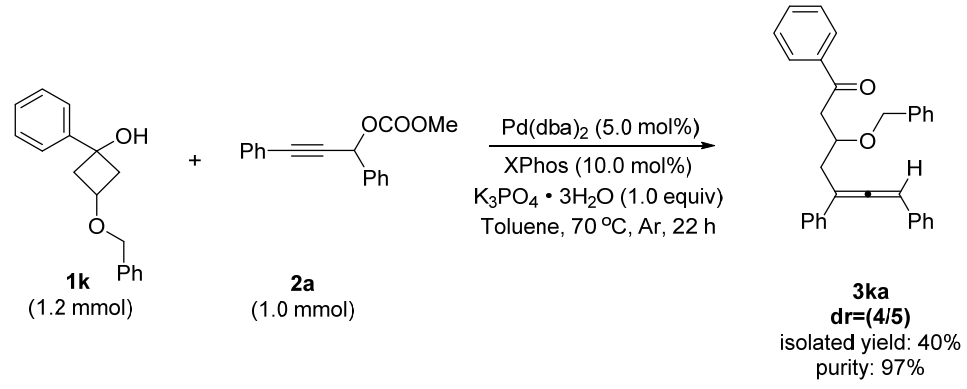
Following **Typical procedure B**, the reaction of $Pd(dba)_2$ (14.5 mg, 0.025 mmol), XPhos (23.7 mg, 0.05 mmol), $K_3PO_4 \cdot 3H_2O$ (265.9 mg, 1.0 mmol), **1i** (liquid, 184.7 mg, 1.2 mmol)/toluene (1.0 mL), **2a** (265.9 mg, 1.0 mmol)/toluene (1.0 mL), and toluene (3.0 mL) for 12 h afforded **3ia** (233.5 mg, 68%) [eluent: petroleum ether/ethyl acetate = 80/1 (0.8 L)] as an oil: ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.45 (d, *J* = 7.6 Hz, 2 H, Ar-H), 7.38-7.27 (m, 6 H, Ar-H), 7.27-7.18 (m, 2 H, Ar-H), 6.55 (t, *J* = 3.0 Hz, 1 H, =CH), 2.65-2.45 (m, 4 H, 2×CH_2), 2.28-2.18 (m, 1 H, CH), 1.95-1.81 (m, 2 H, CH_2), 1.80-1.66 (m, 4 H, 2×CH_2), 1.65-1.60 (m, 1 H, one proton of CH_2), 1.32-1.08 (m, 5 H, 2×CH_2 + one proton of CH_2); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 213.8, 206.4, 135.7, 134.3, 128.7, 128.5, 127.09, 127.06, 126.7, 126.0, 109.3, 98.2, 50.7, 39.8, 29.4, 28.4, 28.3, 25.7, 25.58, 25.56, 21.7; IR (neat, cm^{-1}): ν = 2927, 2852, 1932, 1703, 1596, 1492, 1446, 1406, 1372, 1312, 1239, 1192, 1143, 1073, 1027; MS (70 eV, EI) *m/z* (%): 344 (M^+, 2.61), 84 (100); HRMS Calcd for $C_{25}H_{28}O$ (M^+): 344.2140; Found: 344.2137.

(10) Preparation of 1,6,8-triphenylocta-6,7-dien-2-one (**3ja**) (wpl-2-72)



Following **Typical Procedure B:** the reaction of $Pd(dba)_2$ (14.2 mg, 0.025 mmol), XPhos (23.6 mg, 0.05 mmol), $K_3PO_4 \cdot 3H_2O$ (266.9 mg, 1.0 mmol), **1j** (liquid, 194.7 mg, 1.2 mmol)/toluene (1.0 mL), **2a** (266.5 mg, 1.0 mmol)/toluene (1.0 mL), and toluene (3.0 mL) for 12 h afforded **3ja** (230.4 mg, 65%) [eluent: petroleum ether/ethyl acetate = 80/1 (1.2 L)] as an oil: ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.41 (d, *J* = 7.2 Hz, 2 H, Ar-H), 7.36-7.16 (m, 11 H, Ar-H), 7.12 (d, *J* = 6.8 Hz, 2 H, Ar-H), 6.50 (t, *J* = 3.0 Hz, 1 H, =CH), 3.59 (s, 2 H, CH_2 from Bn), 2.60-2.48 (m, 4 H, 2×CH_2), 1.95-1.80 (m, 2 H, CH_2); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 208.0, 206.3, 135.7, 134.3, 134.2, 129.3, 128.7, 128.6, 128.5, 127.1, 126.9, 126.7, 126.0, 109.2, 98.3, 98.2, 50.1, 41.3, 29.3, 21.8; IR (neat, cm^{-1}): ν = 3083, 3060, 3027, 2931, 2893, 1932, 1709, 1596, 1493, 1447, 1405, 1364, 1311, 1185, 1093, 1074, 1029; MS (70 eV, EI) *m/z* (%): 352 (M^+, 1.12), 149 (100); HRMS Calcd for $C_{26}H_{24}O$ (M^+): 352.1827; Found: 352.1829.

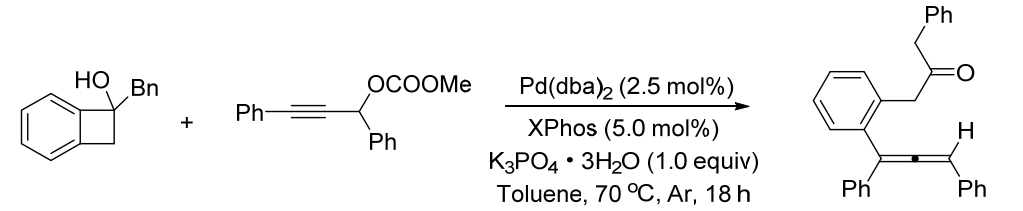
(11) Preparation of 1,5,7-triphenyl-3-benzyloxyhepta-5,6-dien-1-one (**3ka**) (wpl-4-194)



Following **Typical Procedure B**, the reaction of $Pd(dba)_2$ (29.0 mg, 0.05 mmol), XPhos (47.4 mg, 0.10 mmol), $K_3PO_4 \cdot 3H_2O$ (267.0 mg, 1.0 mmol), **1k** (liquid, 305.7 mg, 1.2 mmol)/toluene (2.5 mL), and **2a** (266.4 mg, 1.0 mmol)/toluene (2.5 mL) for

22 h afforded **3ka** (177.7 mg, 40%, 97%purity, dr = 4/5: determined by crude ^{1}H NMR analysis) [eluent: petroleum ether/ethyl acetate = 150/1 (0.4 L) to 100/1 (0.8 L)] as an yellow oil: ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.89-7.79 (m, 2 H, Ar-H), 7.57-7.09 (m, 19 H, Ar-H), [6.53 (t, *J* = 2.8 Hz, 0.45 H), 6.49 (t, *J* = 2.6 Hz, 0.55 H), 1 H, =CH], [4.57 (s, 1.06 H), 4.53 (s, 0.85 H), 2 H, OCH_2], 4.50-4.38 (m, 1 H, CH), 3.41-3.33 (m, 1 H, one proton of CH_2), 3.27-3.17 (m, 1 H, one proton of CH_2), 3.03-2.94 (m, 1 H, one proton of CH_2), 2.94-2.82 (m, 1 H, one proton of CH_2); IR (neat, cm^{-1}): ν = 3084, 3060, 3028, 2903, 2863, 1934, 1682, 1596, 1580, 1493, 1447, 1355, 1211, 1181, 1093, 1071, 1027, 1001; MS (70 eV, EI) *m/z* (%): 353 [(M^+-CH_2Ph), 2.02], 233 (100); HRMS Calcd for $C_{25}H_{21}O_2$ (M^+-CH_2Ph): 353.1542; Found: 353.1532; Anal. Calcd for $C_{32}H_{28}O_2$: C 86.45, H 6.35; Found: C 86.01, H 6.35.

(12) Preparation of 1-(2-(1,3-diphenylpropa-1,2-dienyl)phenyl)-3-phenylpropan-2 -one (**3la**) (wpl-2-159)

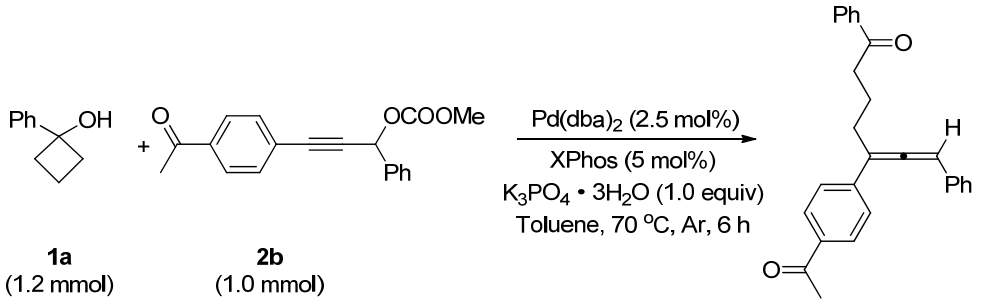


1l (1.2 mmol) **2a** (1.0 mmol) **3la** isolated yield: 51% Purity: 97%

Following **Typical Procedure B:** the reaction of $Pd(dba)_2$ (14.4 mg, 0.025 mmol), XPhos (23.7 mg, 0.05 mmol), $K_3PO_4 \cdot 3H_2O$ (267.0 mg, 1.0 mmol), **1l** (liquid, 253.0 mg, 1.2 mmol)/toluene (1.0 mL), **2a** (266.1 mg, 1.0 mmol)/toluene (1.0 mL) and toluene (3.0 mL) for 18 h afforded **3la** (210.5 mg, 51%, 97% purity) [eluent: petroleum ether/ethyl acetate = 100:1 (0.4 L) to 80:1 (1.6 L) for the first run, then all the products (220.3 mg) was collected and treated with the second run of chromatography, eluent: petroleum ether/ethyl acetate = 100/1 (3.0 L)] as an oil: ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.54-7.13 (m, 17 H, Ar-H), 6.97 (d, *J* = 6.0 Hz, 2 H, Ar-H), 6.49 (s, 1 H, =CH), 3.73 (s, 2 H, CH_2), 3.43 (s, 2 H, CH_2); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 206.6, 205.1, 135.9, 135.8, 134.0, 133.6, 133.1, 131.3, 130.7,

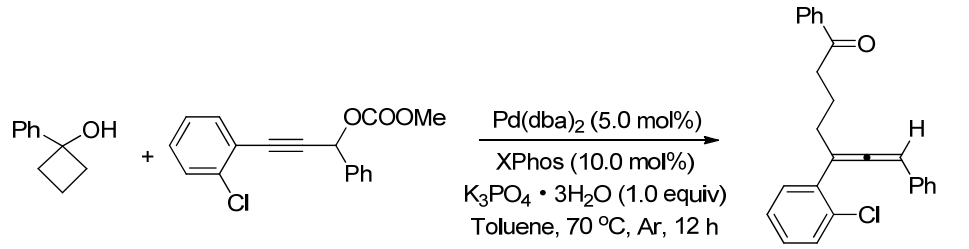
129.4, 128.8, 128.7, 128.5, 128.1, 127.52, 127.47, 127.4, 127.1, 127.0, 126.8, 111.2, 97.6, 49.2, 46.9; IR (neat, cm^{-1}): ν = 3058, 3026, 1931, 1716, 1596, 1489, 1446, 1408, 1328, 1181, 1055, 1028, 1000; MS (70 eV, EI) *m/z* (%): 400 [M^+, 8.15], 91 (100); HRMS Calcd for $C_{30}H_{24}O$ (M^+): 400.1827; Found: 400.1832.

(13) Preparation of 5-(4-acetylphenyl)-1,7-diphenylhepta-5,6-dien-1-one (**3ab**) (wpl-2-93)



Following **Typical Procedure A**, the reaction of $Pd(dba)_2$ (14.4 mg, 0.025 mmol), XPhos (23.7 mg, 0.05 mmol), $K_3PO_4 \cdot 3H_2O$ (266.4 mg, 1.0 mmol), **1a** (soild, 177.5 mg, 1.2 mmol), **2a** (308.1 mg, 1.0 mmol)/toluene (1.0 mL), and toluene (4.0 mL) for 6 h afforded **3ab** (272.0 mg, 72%) [eluent: petroleum ether/ethyl acetate = 50:1 (1.2 L) to 30:1 (0.4 L), then dichloromethane/ethyl acetate/petroleum ether = 1/2/40 (0.8 L)] as a yellow solid: m.p. = 85.3-86.1 °C (petroleum ether/dichloromethane); ^1H NMR (400 MHz, $CDCl_3$) δ = 7.98-7.80 (m, 4 H, Ar-H), 7.63-7.47 (m, 3 H, Ar-H), 7.46-7.17 (m, 7 H, Ar-H), 6.62 (t, *J* = 2.8 Hz, 1 H, =CH), 3.08 (t, *J* = 7.0 Hz, 2 H, CH_2CO), 2.78-2.62 (m, 2 H, CH_2), 2.58 (s, 3 H, $COCH_3$), 2.18-2.00 (m, 2 H, CH_2); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 207.5, 199.7, 197.5, 140.9, 136.9, 135.7, 133.6, 132.9, 128.8, 128.6, 128.5, 127.9, 127.4, 126.9, 126.1, 109.0, 98.8, 37.8, 29.5, 26.5, 22.2; IR (neat, cm^{-1}): ν = 3059, 2957, 2894, 1925, 1677, 1596, 1578, 1495, 1448, 1428, 1408, 1372, 1359, 1343, 1309, 1286, 1263, 1197, 1157, 1114, 1074, 1014, 1001; MS (70 eV, EI) *m/z* (%): 380 (M^+, 8.79), 262 (100); Anal. Calcd for $C_{27}H_{24}O_2$: C 85.23, H 6.36; Found: C 84.87, H 6.35.

(14) Preparation of 5-(2-chlorophenyl)-1,7-diphenylhepta-5,6-dien-1-one (**3ac**) (wpl-2-90)



1a (1.0 mmol)

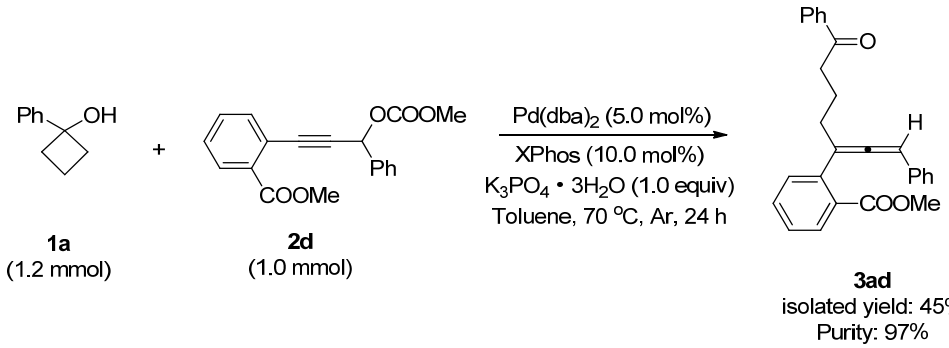
2c (1.2 mmol)

3ac isolated yield: 64%

To a flame-dried Schlenk tube were added $Pd(dba)_2$ (28.7 mg, 0.05 mmol), XPhos (47.4 mg, 0.10 mmol), and $K_3PO_4 \cdot 3H_2O$ (266.4 mg, 1.0 mmol) under Ar atmosphere. Then **1a** (soild, 148.0 mg, 1.0 mmol), **2c** (355.0 mg, 1.2 mmol)/toluene (1.0 mL), and toluene (4.0 mL) were then added sequentially under Ar atmosphere at room temperature. The Schlenk tube was then stirred at 70 °C until completion of the reaction monitored by TLC and ^{1}H NMR (12 h). The crude reaction mixture was filtrated through a short column of silica gel (height: 3 cm, *Φ*: 3.5 cm) eluted with ethyl acetate (60 mL). After evaporation, the residue was purified by chromatography on silica gel to afford **3ac** (240.3 mg, 64%) [eluent: petroleum ether/ethyl acetate = 100/1 (1.2 L)] as an oil: ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.95-7.82 (m, 2 H, Ar-H), 7.51 (t, *J* = 7.4 Hz, 1 H, Ar-H), 7.46-7.10 (m, 11 H, Ar-H), 6.34 (t, *J* = 3.0 Hz, 1 H, =CH), 3.06 (t, *J* = 7.2 Hz, 2 H, CH_2CO), 2.72-2.53 (m, 2 H, CH_2), 2.08-1.90 (m, 2 H, CH_2); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 204.5, 199.9, 136.9, 136.5, 134.3, 132.89, 132.85, 130.1, 129.9, 128.6, 128.5, 128.4, 128.0, 127.04, 127.01, 126.8, 107.6, 96.2, 37.8, 32.8, 22.2; IR (neat, cm^{-1}): ν = 3060, 2931, 1946, 1682, 1597, 1580, 1495, 1472, 1448, 1434, 1364, 1319, 1260, 1225, 1199, 1179, 1123, 1072, 1035, 1001; MS (70 eV, EI) *m/z* (%): 374 [M^+(^{37}Cl), 0.50], 372 [M^+(^{35}Cl), 1.58], 105 (100); HRMS Calcd for $C_{25}H_{21}{}^{35}ClO$ (M^+): 372.1281; Found: 372.1277.

(15) Preparation of 5-(2-(methoxycarbonyl)phenyl)-1,7-diphenylhepta-5,6-dien-1-one

(**3ad**) (wpl-2-112)



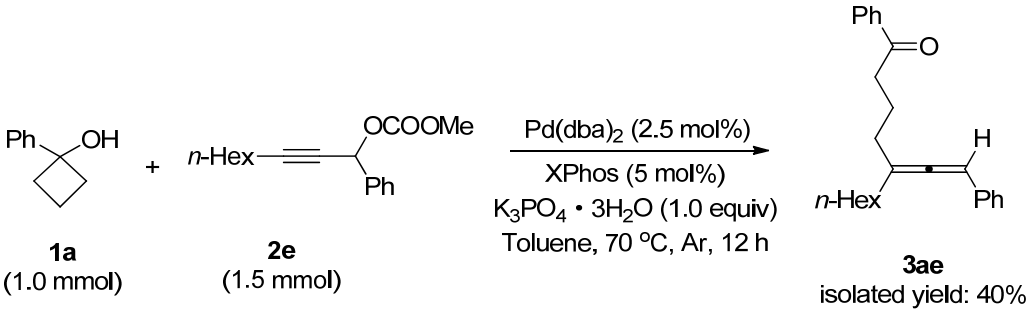
1a
(1.2 mmol)

2d
(1.0 mmol)

3ad
isolated yield: 45%
Purity: 97%

Following **Typical Procedure A**, the reaction of $Pd(dba)_2$ (28.7 mg, 0.05 mmol), XPhos (47.5 mg, 0.10 mmol), $K_3PO_4 \cdot 3H_2O$ (268.0 mg, 1.0 mmol), **1a** (soild, 177.1 mg, 1.2 mmol), **2d** (324.0 mg, 1.0 mmol)/toluene (1.0 mL) and toluene (4.0 mL) for 24 h afforded **3ad** (183.4 mg, 45%, 97% purity) [eluent: petroleum ether/ethyl acetate = 40:1 (0.6 L) to 20:1 (0.4 L) for the first run, then all the products (204.5 mg) was collected and treated with the second run of chromatography, eluent: petroleum ether/ethyl acetate = 100/1 (3.0 L)] as an oil: ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.89 (d, *J* = 8.4 Hz, 2 H, Ar-H), 7.79 (dd, *J* = 7.6, 1.2 Hz, 1 H, Ar-H), 7.57-7.23 (m, 10 H, Ar-H), 7.22-7.14 (m, 1 H, Ar-H), 6.32 (t, *J* = 3.2 Hz, 1 H, =CH), 3.84 (s, 3 H, OMe), 3.09 (t, *J* = 7.2 Hz, 2 H, CH_2CO), 2.67-2.48 (m, 2 H, CH_2), 2.12-1.94 (m, 2 H, CH_2); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 203.7, 200.1, 168.0, 138.9, 137.0, 134.7, 132.8, 131.7, 130.1, 129.91, 129.86, 128.5, 128.4, 128.0, 127.2, 127.0, 126.9, 109.9, 96.3, 52.1, 37.8, 33.4, 22.3; IR (neat, cm^{-1}): ν = 3061, 3027, 2948, 1947, 1721, 1682, 1596, 1579, 1484, 1447, 1433, 1365, 1289, 1254, 1226, 1196, 1124, 1085, 1027, 1001; MS (70 eV, EI) *m/z* (%): 396 (M^+, 34.14), 105 (100); HRMS Calcd for $C_{27}H_{24}O_3$ (M^+): 396.1725; Found: 396.1729.

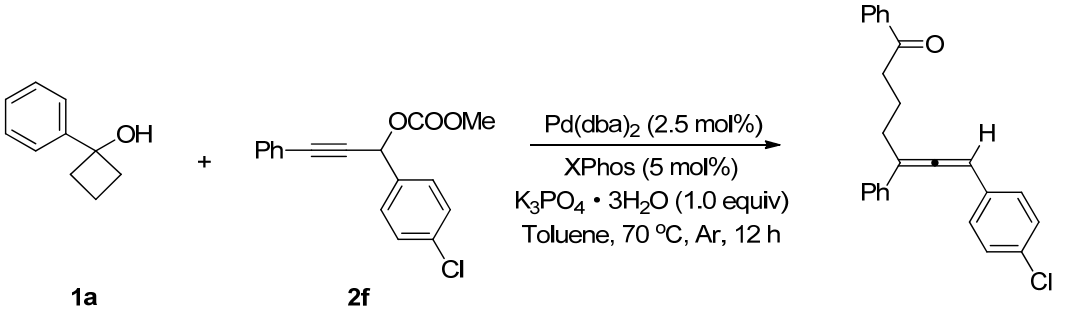
(16) Preparation of 5-hexyl-1,7-diphenylhepta-5,6-dien-1-one (**3ae**) (wpl-2-44)



Typical Procedure C: To a flame-dried Schlenk tube were added $Pd(dba)_2$ (14.5 mg, 0.025 mmol) , XPhos (23.9 mg, 0.05 mmol), and $K_3PO_4 \cdot 3H_2O$ (266.7 mg, 1.0 mmol) under Ar atmosphere. **1a** (soild, 148.1 mg, 1.0 mmol), **2e** (410.2 mg, 1.5 mmol) in toluene (1.0 mL), and toluene (4.0 mL) were then added sequentially under Ar atmosphere at room temperature. The Schlenk tube was then stirred at 70 ^{o}C until completion of the reaction as monitored by TLC and ^{1}H NMR (12 h). The crude reaction mixture was filtrated through a short column of silica gel (height: 3 cm, Φ: 3.5 cm) eluted with ethyl acetate (60 mL). After evaporation, the residue was purified by chromatography on silica gel to afford **3ae** (138.5 mg, 40%) [eluent: petroleum ether/ethyl acetate = 80:1 (0.8 L) for the first run, then all the product was collected and treated with the second run of chromatography, eluent: petroleum ether/diethyl ether = 250:1 (2.0 L)] as an oil: ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.89-7.83 (m, 2 H, Ar-H), 7.51 (t, *J* = 7.4 Hz, 1 H, Ar-H), 7.39 (t, *J* = 7.8 Hz, 2 H, Ar-H), 7.32-7.23 (m, 4 H, Ar-H), 7.21-7.12 (m, 1 H, Ar-H), 6.15 (quint, *J* = 3.0 Hz, 1 H, =CH), 3.05-2.92 (m, 2 H, CH_2), 2.25-2.16 (m, 2 H, CH_2), 2.15-2.04 (m, 2 H, CH_2), 2.02-1.85 (m, 2 H, CH_2), 1.54-1.40 (m, 2 H, CH_2), 1.38-1.18 (m, 6 H, 3×CH_2), 0.85 (t, *J* = 7.0 Hz, 3 H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 202.2, 200.2, 137.0, 135.9, 132.8, 128.51, 128.46, 128.0, 126.4, 108.1, 95.7, 38.0, 32.7, 32.2, 31.6, 29.1, 27.6, 22.6, 22.0, 14.0; IR (neat, cm^{-1}): ν = 3030, 2953, 2925, 2854, 1946, 1685, 1597, 1580, 1495, 1448, 1406, 1364, 1260, 1230, 1209, 1179, 1072, 1001; MS (70 eV, EI) *m/z* (%): 346 (M^+, 4.35), 105 (100); HRMS Calcd for $C_{25}H_{30}O$ (M^+): 346.2297; Found: 346.2293.

(17) Preparation of 7-(4-chlorophenyl)-1,5-diphenylhepta-5,6-dien-1-one (**3af**)

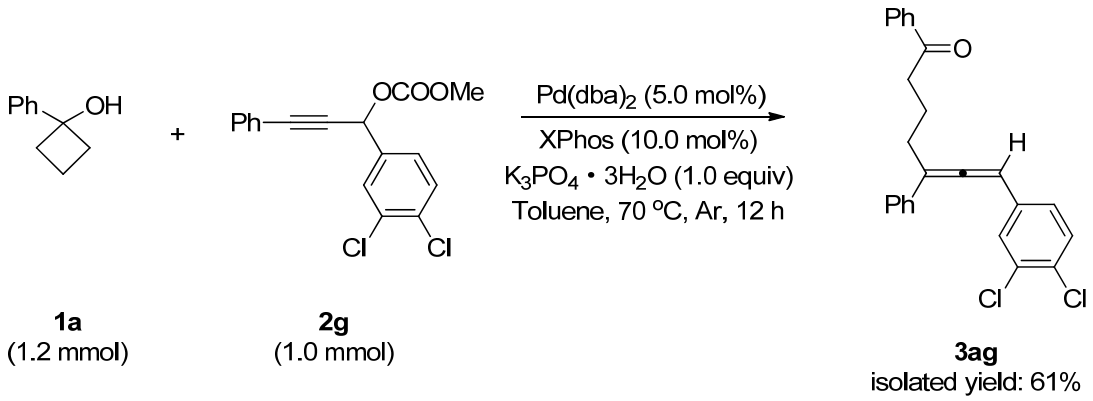
(wpl-2-26)



1a (1.2 mmol) + **2f** (1.0 mmol) → **3af** isolated yield: 66%

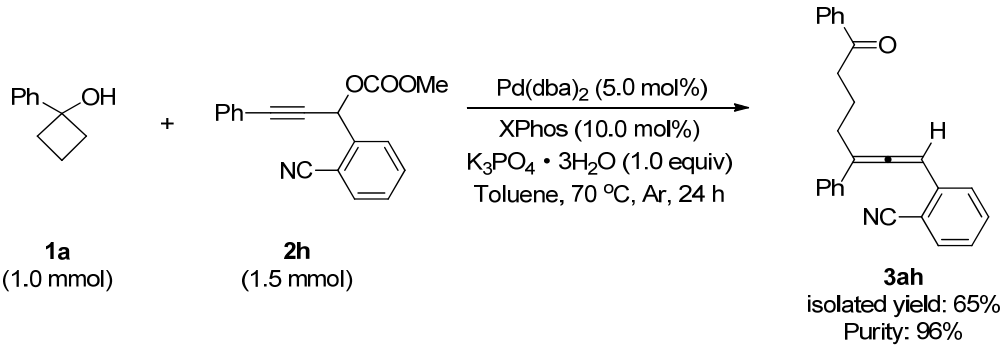
Following **Typical Procedure A**, the reaction of $Pd(dba)_2$ (14.5 mg, 0.025 mmol), XPhos (23.9 mg, 0.05 mmol), $K_3PO_4 \cdot 3H_2O$ (266.4 mg, 1.0 mmol), **1a** (soild, 177.9 mg, 1.2 mmol), **2f** (300.7 mg, 1.0 mmol)/toluene (1.0 mL), and toluene (4.0 mL) for 12 h afforded **3af** (246.0 mg, 66%) [eluent: petroleum ether/ethyl acetate = 80/1 (0.8 L)] as a white solid: m.p. = 96.5-97.0 °C (petroleum ether/dichloromethane); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.89-7.82 (m, 2 H, Ar-H), 7.58-7.49 (m, 1 H, Ar-H), 7.48-7.37 (m, 4 H, Ar-H), 7.36-7.28 (m, 2 H, Ar-H), 7.27-7.20 (m, 5 H, Ar-H), 6.52 (t, 1 H, *J* = 3.0 Hz, =CH), 3.06 (t, 2 H, *J* = 7.0 Hz, CH_2CO), 2.75-2.60 (m, 2 H, CH_2), 2.16-1.98 (m, 2 H, CH_2); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 206.6, 199.8, 136.9, 135.4, 133.0, 132.7, 128.9, 128.6, 128.5, 127.9, 127.3, 126.1, 109.8, 97.4, 37.9, 29.6, 22.1; IR (neat, cm^{-1}): ν = 3057, 2954, 2894, 1934, 1716, 1685, 1595, 1578, 1489, 1448, 1433, 1406, 1371, 1345, 1329, 1311, 1265, 1232, 1197, 1176, 1087, 1072, 1030, 1010, 1002; MS (70 eV, EI) *m/z* (%): 374 [M^+(^{37}Cl), 1.10], 372 [M^+(^{35}Cl), 3.29], 220 (100); Anal. Calcd for $C_{25}H_{21}OCl$: C 80.53, H 5.68; Found: C 80.54, H 5.77.

(18) Preparation of 7-(3,4-dichlorophenyl)-1,5-diphenylhepta-5,6-dien-1-one (**3ag**) (wpl-2-104)



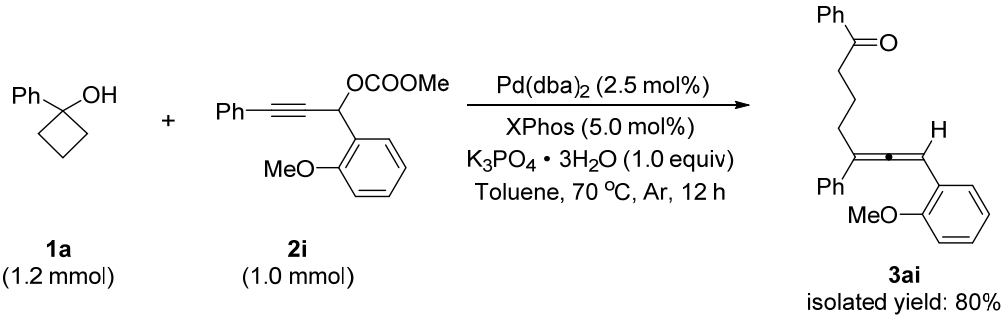
Following **Typical Procedure A**, the reaction of $Pd(dba)_2$ (28.6 mg, 0.05 mmol), XPhos (47.7 mg, 0.10 mmol), $K_3PO_4 \cdot 3H_2O$ (266.3 mg, 1.0 mmol), **1a** (soild, 178.0 mg, 1.2 mmol), **2g** (335.0 mg, 1.0 mmol)/toluene (1.0 mL), and toluene (4.0 mL) for 12 h afforded **3ag** (247.8 mg, 61%) [eluent: petroleum ether/ethyl acetate = 60:1 (0.8 L) to 40:1 (0.8 L), finally re-crystalized (petroleum ether/dichloromethane) to provide the desired **3ag** as a white solid]: m.p. = 113.4-114.3 ^{o}C (petroleum ether/dichloromethane); ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.87 (d, J = 6.8 Hz, 2 H, Ar-H), 7.70-7.20 (m, 10 H, Ar-H), 7.13 (dd, J_1 = 8.4 Hz, J_2 = 2.0 Hz, 1 H, Ar-H), 6.46 (t, J = 2.8 Hz, 1 H, =CH), 3.06 (t, J = 7.0 Hz, 2 H, CH_2CO), 2.81-2.55 (m, 2 H, CH_2), 2.20-1.94 (m, 2 H, CH_2); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 206.8, 199.6, 136.8, 135.0, 134.8, 133.0, 132.8, 130.7, 130.6, 128.7, 128.5, 128.2, 127.9, 127.5, 126.1, 125.9, 110.3, 96.6, 37.8, 29.5, 22.0; IR (neat, cm^{-1}): ν = 3064, 2951, 2895, 1938, 1686, 1596, 1555, 1492, 1474, 1448, 1433, 1418, 1406, 1373, 1359, 1343, 1329, 1312, 1275, 1265, 1231, 1199, 1179, 1158, 1128, 1073, 1057, 1026, 1002; MS (70 eV, EI) *m/z* (%): 408 [M^+($^{37,35}Cl_2$), 1.09], 406 [M^+($^{35,35}Cl_2$), 1.76], 220 (100); Anal. Calcd for $C_{25}H_{20}Cl_2O$: C 73.72, H 4.95; Found: C 73.64, H 4.95.

(19) Preparation of 7-(2-cyanophenyl)-1,5-diphenylhepta-5,6-dien-1-one (**3ah**) (wpl-2-111)



Following **Typical Procedure C**, the reaction of $Pd(dba)_2$ (28.6 mg, 0.05 mmol), XPhos (47.5 mg, 0.10 mmol), $K_3PO_4 \cdot 3H_2O$ (266.2 mg, 1.0 mmol), **1a** (soild, 148.0 mg, 1.0 mmol), **2h** (436.0 mg, 1.5 mmol)/toluene (1.0 mL), and toluene (4.0 mL) for 24 h afforded **3ah** (244.0 mg, 65%, 96% purity) [eluent: petroleum ether/ethyl acetate = 40:1 (0.6 L) to 30:1 (0.4 L) to 20:1 (0.8 L) for the first run, then all the products (276.0 mg) was collected and treated with the second run of chromatography, eluent: petroleum ether/ethyl acetate = 80:1 (1.2 L) to 40:1 (1.2 L)] as an oil: ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.91 (d, J = 7.2 Hz, 2 H, Ar-H), 7.61 (dd, J_1 = 7.6 Hz, J_2 = 0.8 Hz, 1 H, Ar-H), 7.58-7.49 (m, 2 H, Ar-H), 7.48-7.38 (m, 5 H, Ar-H), 7.37-7.30 (m, 2 H, Ar-H), 7.28-7.20 (m, 2 H, Ar-H), 6.93 (t, J = 3.0 Hz, 1 H, =CH), 3.08 (t, J = 7.0 Hz, 2 H, CH_2CO), 2.82-2.63 (m, 2 H, CH_2), 2.14-2.00 (m, 2 H, CH_2); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 208.0, 199.6, 138.1, 136.8, 134.6, 133.04, 132.96, 132.7, 128.6, 128.5, 127.9, 127.6, 127.2, 127.0, 126.2, 117.6, 110.9, 110.2, 95.1, 37.8, 29.6, 22.2; IR (neat, cm^{-1}): ν = 3059, 2931, 2221, 1932, 1682, 1596, 1579, 1485, 1446, 1407, 1364, 1296, 1260, 1226, 1201, 1180, 1160, 1089, 1074, 1029, 1001; MS (70 eV, EI) *m/z* (%): 363 [M^+, 10.98], 105 (100); HRMS Calcd for $C_{26}H_{21}NO$ (M^+): 363.1623; Found: 363.1625.

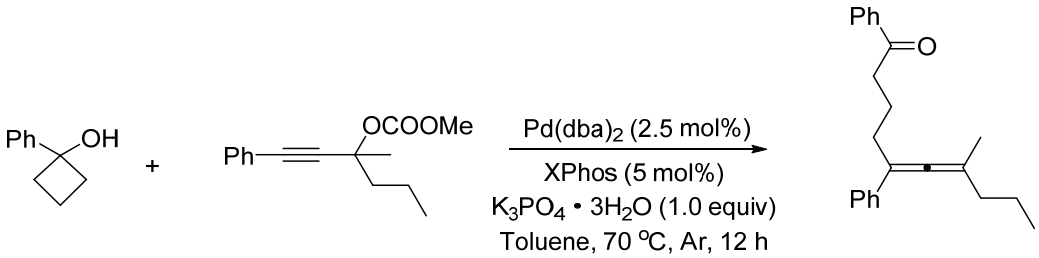
(20) Preparation of 7-(2-methoxyphenyl)-1,5-diphenylhepta-5,6-dien-1-one (**3ai**) (wpl-4-184)



1a (1.2 mmol) **2i** (1.0 mmol) **3ai** isolated yield: 80%

Following **Typical Procedure A**, the reaction of $Pd(dba)_2$ (14.3 mg, 0.025 mmol), XPhos (23.6 mg, 0.05 mmol), $K_3PO_4 \cdot 3H_2O$ (266.7 mg, 1.0 mmol), **1a** (soild, 177.6 mg, 1.2 mmol), **2i** (soild, 297.0 mg, 1.0 mmol), and toluene (5.0 mL) for 12 h afforded **3ai** (294.8 mg, 80%) [eluent: petroleum ether/ethyl acetate = 80:1 (0.4 L) to 40:1 (0.8 L)] as an oil: ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.88-7.83 (m, 2 H, Ar-H), 7.54-7.44 (m, 3 H, Ar-H), 7.42-7.35 (m, 3 H, Ar-H), 7.34-7.28 (m, 2 H, Ar-H), 7.24-7.15 (m, 2 H, Ar-H), 6.96 (t, *J* = 3.0 Hz, 1 H, =CH), 6.91-6.84 (m, 2 H, Ar-H), 3.83 (s, 3 H, OCH_3), 3.06 (t, *J* = 7.2 Hz, 2 H, CH_2CO), 2.76-2.59 (m, 2 H, CH_2), 2.17-2.00 (m, 2 H, CH_2); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 206.9, 200.1, 156.2, 137.0, 136.1, 132.8, 128.5, 128.2, 128.0, 127.6, 126.9, 126.0, 122.7, 120.8, 111.1, 108.3, 92.2, 55.5, 38.0, 29.6, 22.3; IR (neat, cm^{-1}): ν = 3059, 3025, 3003, 2935, 2900, 2837, 1932, 1683, 1596, 1581, 1492, 1463, 1448, 1391, 1365, 1287, 1245, 1200, 1171, 1159, 1103, 1074, 1048, 1027, 1001; MS (70 eV, EI) *m/z* (%): 353 [(M^+-CH_3), 2.88], 220 (100); HRMS Calcd for $C_{26}H_{24}O_2$ (M^+): 368.1771; Found: 368.1769.

(21) Preparation of 7-methyl-1,5-diphenyldeca-5,6-dien-1-one **(3aj)** (wpl-2-128)

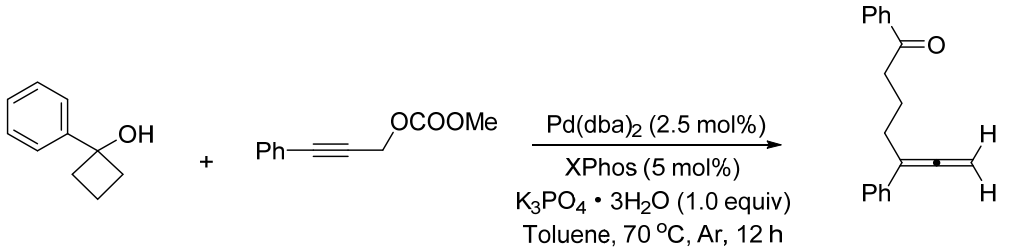


1a (1.2 mmol) **2j** (1.0 mmol) **3aj** isolated yield: 53%

Following **Typical Procedure A**, the reaction of $Pd(dba)_2$ (14.4 mg, 0.025 mmol), XPhos (23.7 mg, 0.05 mmol), $K_3PO_4 \cdot 3H_2O$ (266.9 mg, 1.0 mmol), **1a** (soild, 177.6 mg, 1.2 mmol), **2j** (246.0 mg, 1.0 mmol)/toluene (1.0 mL), and toluene (4.0 mL) for

12 h afforded **3aj** (169.2 mg, 53%) [eluent: petroleum ether/ethyl acetate = 100:1 (0.8 L)] as an oil: ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.98-7.88 (m, 2 H, Ar-H), 7.53 (t, *J* = 7.2 Hz, 1 H, Ar-H), 7.47-7.34 (m, 4 H, Ar-H), 7.29 (t, *J* = 7.8 Hz, 2 H, Ar-H), 7.16 (t, *J* = 7.6 Hz, 1 H, Ar-H), 3.10-3.02 (m, 2 H, CH_2CO), 2.52 (t, *J* = 7.2 Hz, 2 H, CH_2C=), 2.10-1.92 (m, 4 H, 2×CH_2), 1.78 (s, 3 H, Me), 1.55-1.40 (m, 2 H, CH_2), 0.91 (t, *J* = 7.4 Hz, 3 H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 201.4, 200.2, 138.1, 137.1, 132.8, 128.5, 128.2, 128.0, 126.1, 125.9, 103.9, 103.2, 38.1, 36.5, 29.7, 22.5, 21.0, 18.9, 14.0; IR (neat, cm^{-1}): ν = 3059, 2956, 2930, 2870, 1947, 1684, 1597, 1580, 1492, 1447, 1407, 1366, 1319, 1225, 1198, 1179, 1156, 1073, 1028, 1001; MS (70 eV, EI) *m/z* (%): 318 (M^+, 3.58), 220 (100); HRMS Calcd for $C_{23}H_{26}O$ (M^+): 318.1984; Found: 318.1982.

(22) Preparation of 1,5-diphenylhepta-5,6-dien-1-one (**3ak**) (wpl-4-182)

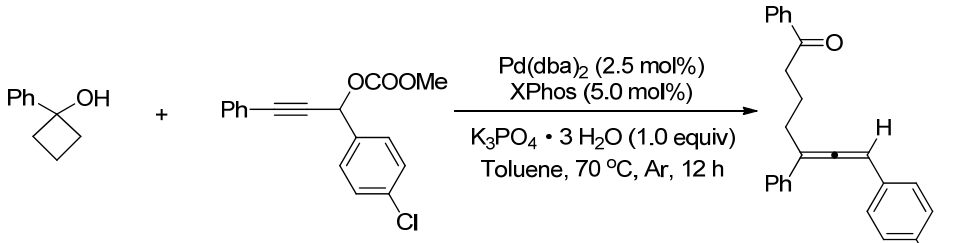


1a (1.2 mmol) **2k** (1.0 mmol) **3ak** isolated yield: 50%

Following **Typical Procedure A**, the reaction of $Pd(dba)_2$ (14.6 mg, 0.025 mmol), XPhos (24.0 mg, 0.05 mmol), $K_3PO_4 \cdot 3H_2O$ (266.4 mg, 1.0 mmol), **1a** (soild, 177.9 mg, 1.2 mmol), **2k** (190.4 mg, 1.0 mmol)/toluene (1.0 mL), and toluene (4.0 mL) for 12 h afforded **3ak** (131.5 mg, 50%) [eluent: petroleum ether/ethyl acetate = 80/1 (0.8 L)] as an oil: ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.95 (d, *J* = 8.0 Hz, 2 H, Ar-H), 7.58-7.50 (m, 1 H, Ar-H), 7.48-7.36 (m, 4 H, Ar-H), 7.32 (t, *J* = 7.6 Hz, 2 H, Ar-H), 7.20 (t, *J* = 7.4 Hz, 1 H, Ar-H), 5.08 (t, *J* = 3.2 Hz, 2 H, =CH), 3.07 (t, *J* = 7.2 Hz, 2 H, CH_2CO), 2.58-2.49 (m, 2 H, CH_2), 2.04 (quint, *J* = 7.3 Hz, 2 H, CH_2); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 208.5, 200.1, 137.0, 136.1, 132.9, 128.5, 128.4, 128.0, 126.7, 126.0, 104.4, 78.5, 37.9, 29.0, 22.3; IR (neat, cm^{-1}): ν = 3084, 3058, 3030, 1939, 1682,

1597, 1580, 1494, 1448, 1407, 1365, 1227, 1199, 1180, 1075, 1028, 1001; MS (70 eV, EI) *m/z* (%): 262 (M^+, 4.03), 105 (100); HRMS Calcd for $C_{19}H_{18}O$ (M^+): 262.1352; Found: 262.1348.

4. Gram scale synthesis of 7-(4-chlorophenyl)-1,5-diphenylhepta-5,6-dien-1-one (3af) (wpl-2-117)



1a (6.0 mmol)

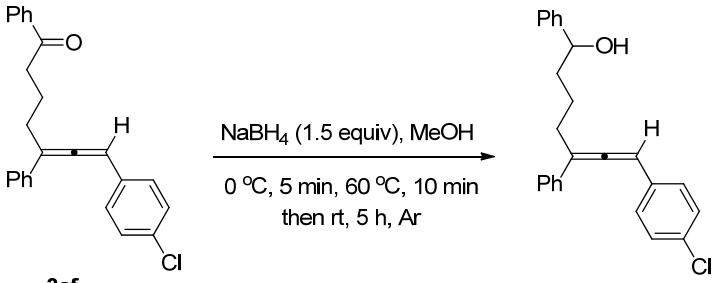
2f (5.0 mmol)

3af, 1.2305 g, 66% yield

Following **Typical Procedure A**, the reaction of $Pd(dba)_2$ (71.9 mg, 0.125 mmol), XPhos (119.2 mg, 0.250 mmol), $K_3PO_4 \cdot 3H_2O$ (1332.0 mg, 5.0 mmol), **1a** (soild, 888.0 mg, 6.0 mmol), **2f** (1502.7 mg, 5.0 mmol)/toluene (5.0 mL), and toluene (20.0 mL) for 12 h afforded **3af** (1.2305 g, 66%) [eluent: petroleum ether/ethyl acetate = 40/1 (0.8 L) to 30/1 (1.2L)] as a white solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.85 (d, *J* = 7.6 Hz, 2 H, Ar-H), 7.52 (t, *J* = 7.2 Hz, 1 H, Ar-H), 7.48-7.36 (m, 4 H, Ar-H), 7.32 (t, *J* = 7.6 Hz, 2 H, Ar-H), 7.27-7.17 (m, 5 H, Ar-H), 6.50 (t, *J* = 2.8 Hz, 1 H, =CH), 3.04 (t, *J* = 7.0 Hz, 2 H, CH_2CO), 2.75-2.60 (m, 2 H, CH_2), 2.16-1.97 (m, 2 H, CH_2); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 206.5, 199.7, 136.8, 135.4, 132.9, 132.6, 128.9, 128.6, 128.5, 127.9, 127.3, 126.0, 109.7, 97.3, 37.8, 29.5, 22.1.

5. Transformations of product 3af.

(1) Reduction with $NaBH_4$ for the synthesis of 7-(4-chlorophenyl)-1,5-diphenyl hepta-5,6-dien-1-ol (**4**) (wpl-2-133, wpl-3-157)

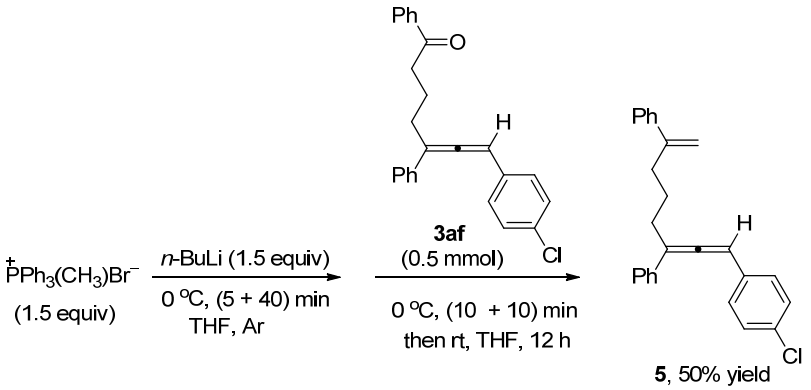


3af
(0.5 mmol)

4 (dr = 1/0.9)
93% yield

To a flame-dried schlenk tube were added **3af** (186.4 mg, 0.5 mmol) and MeOH (5.0 mL). The reaction was cooled to 0 ^{o}C and $NaBH_4$ (28.7 mg, 0.75 mmol, 1.5 equiv) was added in portion. Then, the reaction was stirred at 0 ^{o}C for 5 min and 60 ^{o}C for 10 min. The resulting mixture was allowed to cool to room temperature and stirred at room temperature for 5 h. The reaction was then cooled to 0 ^{o}C and quenched with water (10 mL). The aqueous layer was extracted by EtOAc (10 mL × 3). The combined organic phases was washed with brine (20 mL), dried over Na_2SO_4, filtered, and concentrated under reduced pressure. After evaporation, the residue was purified by chromatography on silica gel [eluent: petroleum ether/ethyl acetate = 40/1 (0.8 L) to 20/1 (1.0 L)] to afford **4** (173.9 mg, 93%, dr = 1/0.9: determined by quantitative ^{13}C NMR analysis) as a colorless oil: ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.39 (d, *J* = 7.6 Hz, 2 H, ArH), 7.33-7.02 (m, 12 H, ArH), 6.50-6.42 (m, 1 H, =CH), 4.67-4.54 (m, 1 H, OCH), 2.65-2.49 (m, 2 H, CH_2), 2.01-1.52 (m, 5 H, 2×CH_2 + OH); IR (neat, cm^{-1}): ν = 3577, 3372, 3028, 2930, 1932, 1674, 1596, 1489, 1451, 1381, 1308, 1215, 1089, 1028, 1012; MS (70 eV, EI) *m/z* (%): 376 [M^+(^{37}Cl), 11.31], 374 [M^+(^{35}Cl), 31.87], 105 (100); Anal. Calcd for $C_{25}H_{23}ClO$: C, 80.09; H, 6.18; Found: C, 79.88; H, 6.50.

(2) Reaction with a Wittig reagent for the synthesis of 1-(4-chlorophenyl)-3,7-diphenyl-octa-1,2,7-triene (**5**) [3] (wpl-2-174)



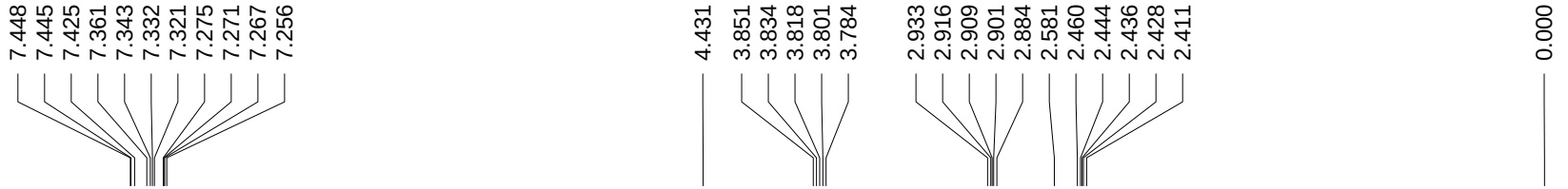
To a mixture of methyltriphenylphosphonium bromide (267.0 mg, 0.75 mmol, 1.5 equiv) in dry THF (5 mL) at 0 °C (ice/water bath) was slowly added *n*-BuLi (2.5 M in hexane, 0.3 mL, 0.75 mmol, 1.5 equiv) within 5 minutes under Ar atmosphere with stirring and the solution was stirred at 0 °C vigorously for 40 minutes. A solution of **3af** (186.5 mg, 0.5 mmol, 1.0 equiv) in dry THF (2.5 mL) was then added dropwise at 0 °C to the resulting mixture over a period of 10 minutes. After being stirred at 0 °C for another 10 minutes, the ice/water bath was removed. After 12 hours at room temperature, the reaction was quenched by addition of a saturated solution of NH_4Cl (aq, 15 mL) and extracted with EtOAc (15 mL × 3), and the combined organic phase was dried by Na_2SO_4. The solvent was then removed in vacuo to give a crude mixture, which was purified by silica gel column chromatography [petroleum ether/ethyl acetate = 200/1 (0.6 L)] to afford **5** (92.7 mg, 50%) as an oil: ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.50-7.10 (m, 14 H, Ar-H), 6.45 (t, *J* = 2.6 Hz, 1 H, =CH), 5.27 (s, 1 H, one proton of $=CH_2$), 5.03 (s, 1 H, one proton of $=CH_2$), 2.70-2.48 (m, 4 H, 2×CH_2), 1.85-1.66 (m, 2 H, CH_2); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 206.5, 147.9, 141.0, 135.6, 133.0, 132.5, 128.8, 128.5, 128.2, 127.9, 127.3, 127.2, 126.1, 126.0, 112.6, 110.0, 97.1, 34.9, 29.5, 26.2; IR (neat, cm^{-1}): ν = 3104, 3083, 3052, 2932, 2898, 2828, 1935, 1624, 1595, 1573, 1489, 1450, 1434, 1382, 1349, 1328, 1292, 1276, 1216, 1195, 1161, 1088, 1072, 1027, 1012; MS (70 eV, EI) *m/z* (%): 372 [M^+(^{37}Cl), 11.41], 370 [M^+(^{35}Cl), 31.85], 205 (100); HRMS Calcd for $C_{26}H_{23}{}^{35}Cl$ (M^+): 370.1488; Found: 370.1490.

References:

[1] a) K. Jia, F. Zhang, H. Huang, Y. Chen, *J. Am. Chem*. *Soc.* 2016, **138**, 1514; b) H. Zeng, P. Pan, J. Chen, H. Gong, C. Jun, *Eur. J. Org. Chem*. 2017, 1070; c) B. M. Casey, C. A. Eakin, R. A. Flowers II, *Tetrahedron Lett.* 2009, **50**, 1264; d) C. S. A. Antunes, M. Bietti, O. Lanzalunga, M. Salamone, *J. Org. Chem*. 2004, **69**, 5281.

[2] a) R. Shen, J. Yang, S. Zhu, C. Chen, L. Wu, *Adv. Synth. Catal.* 2015, **357**, 1259; b) P. Wu, M. Jia, W. Lin, S. Ma, *Org. Lett*. 2018, **20**, 554; c) M. Xiong, H. Hu, X. Hu, Y. Liu, *Org. Lett*. 2018, **20**, 3661; d) H. Luo, Y. Yu, S. Ma, *Org. Chem. Front.* 2016, **3**, 1705; e) A. Zhang, S. Ma, *J. Org. Chem.* 2002, **67**, 2287; f) G. Gangadhararao, R. Kotikalapudi, M. N. Reddy, K. C. K. Swamy, *Beilstein J. Org. Chem.* 2014, **10**, 996. g) J. Ying, C. Zhou, X. Wu, *Org. Biomol. Chem.* 2018, **16**, 1065.

[3] K.-P. Wang, S. Y. Yun, D. Lee, D. J. Wink, *J. Am. Chem*. *Soc.* 2009, **131**, 15114.



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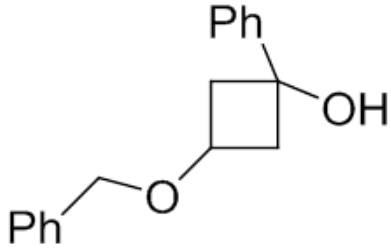

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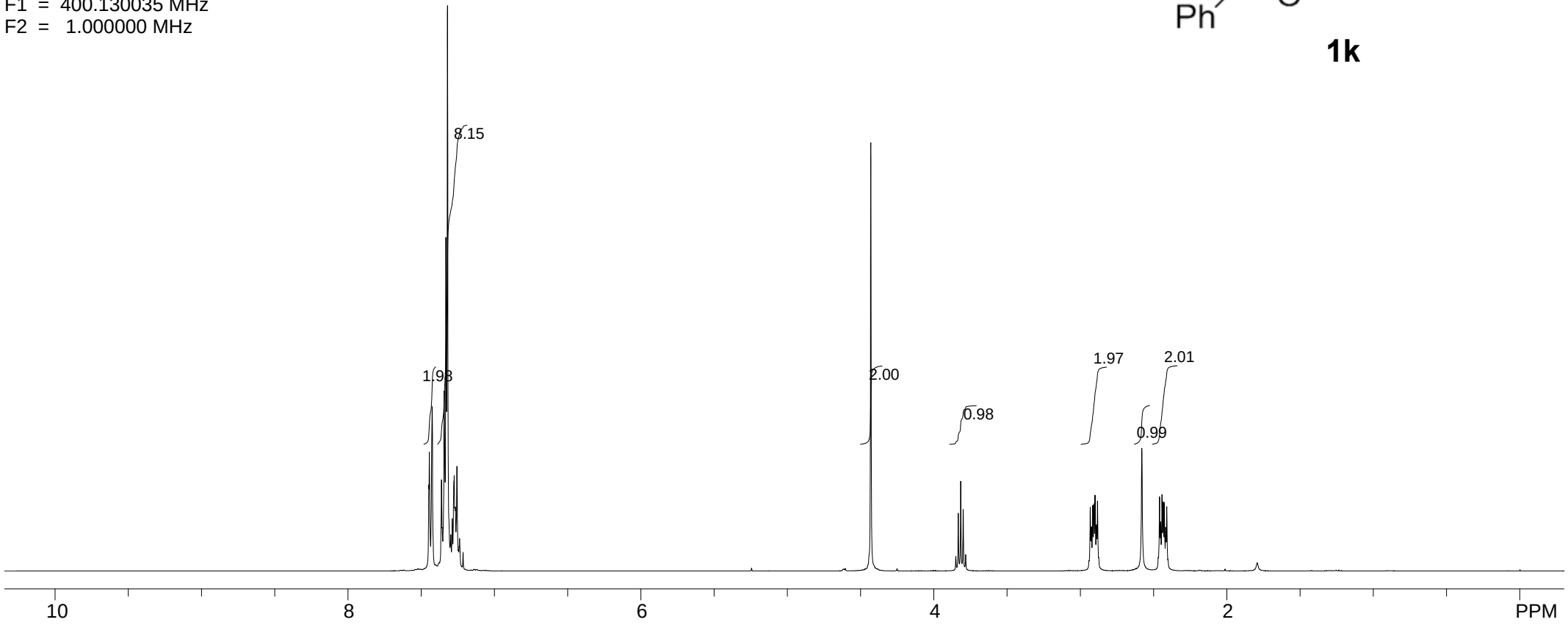

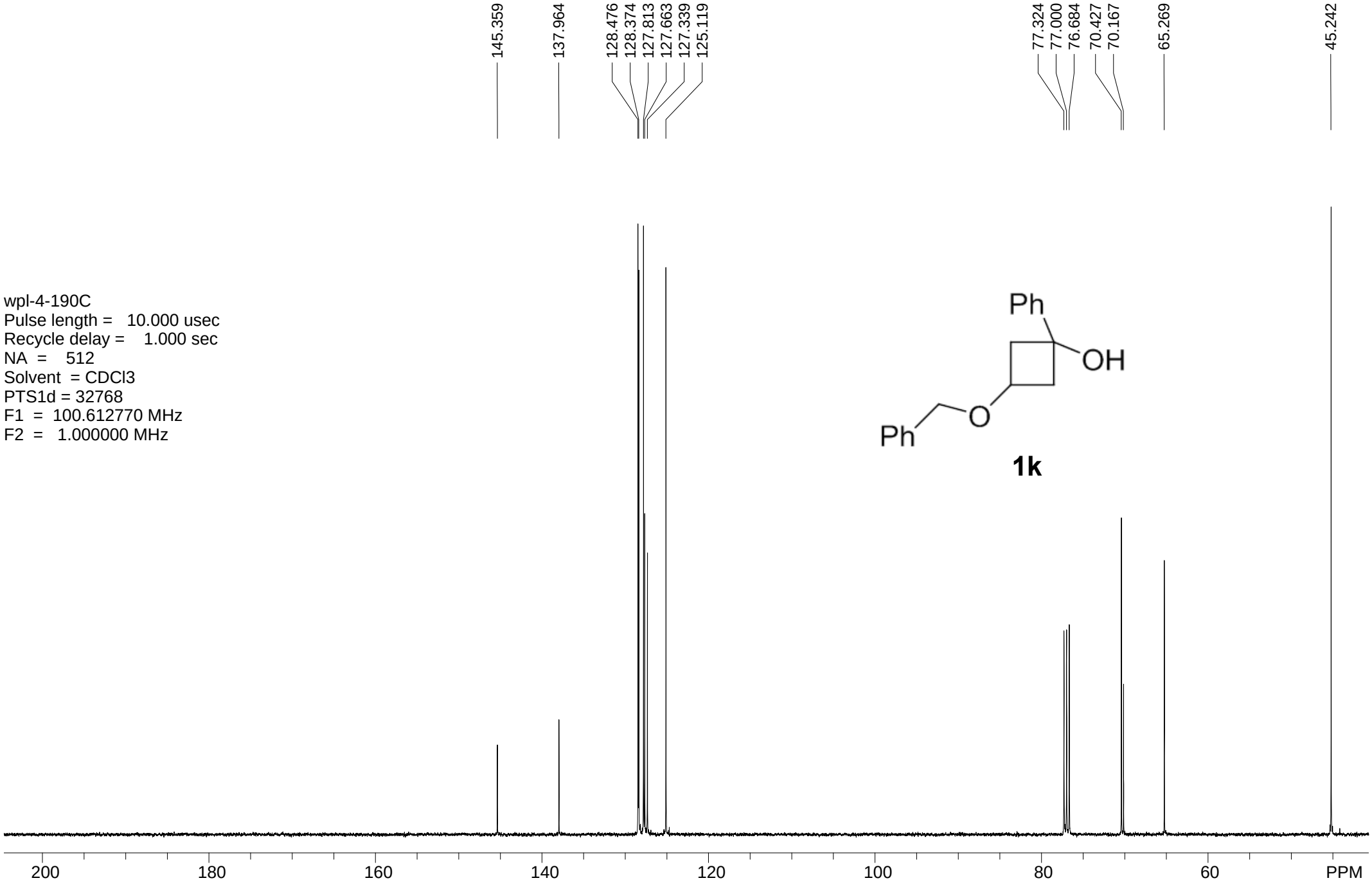
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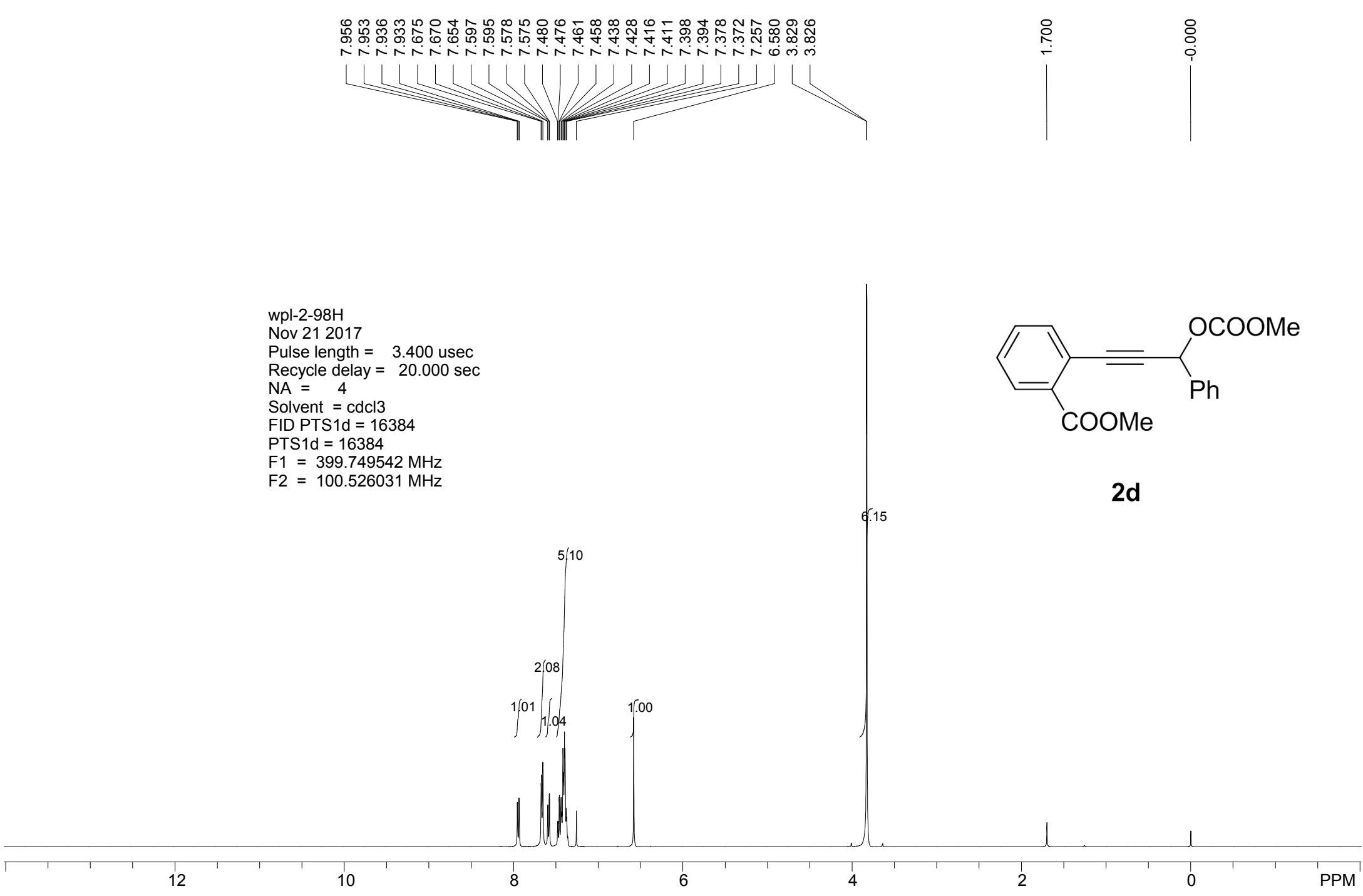
80
60
PPM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7.416
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10
8
6
4
2
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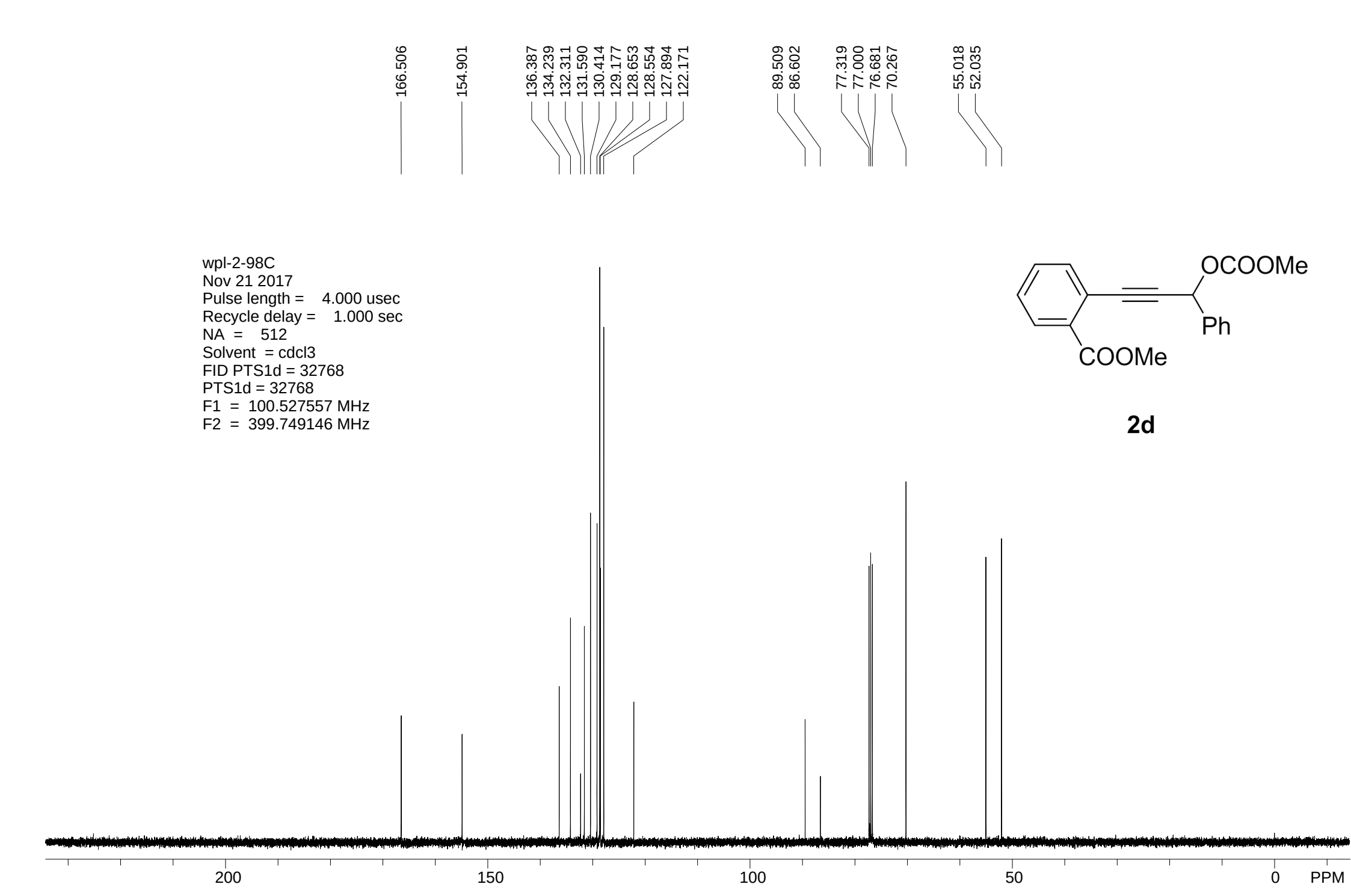
PPM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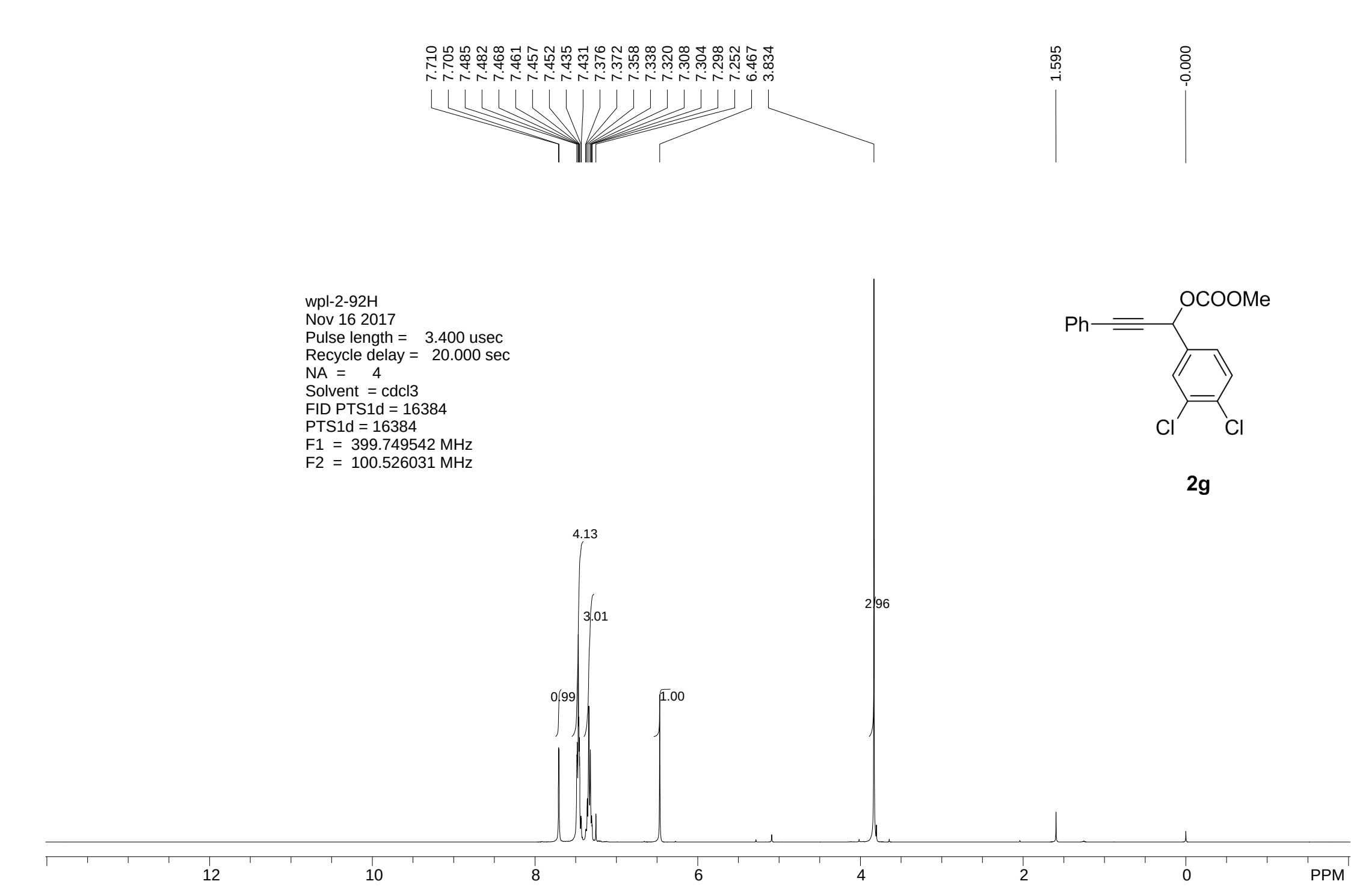
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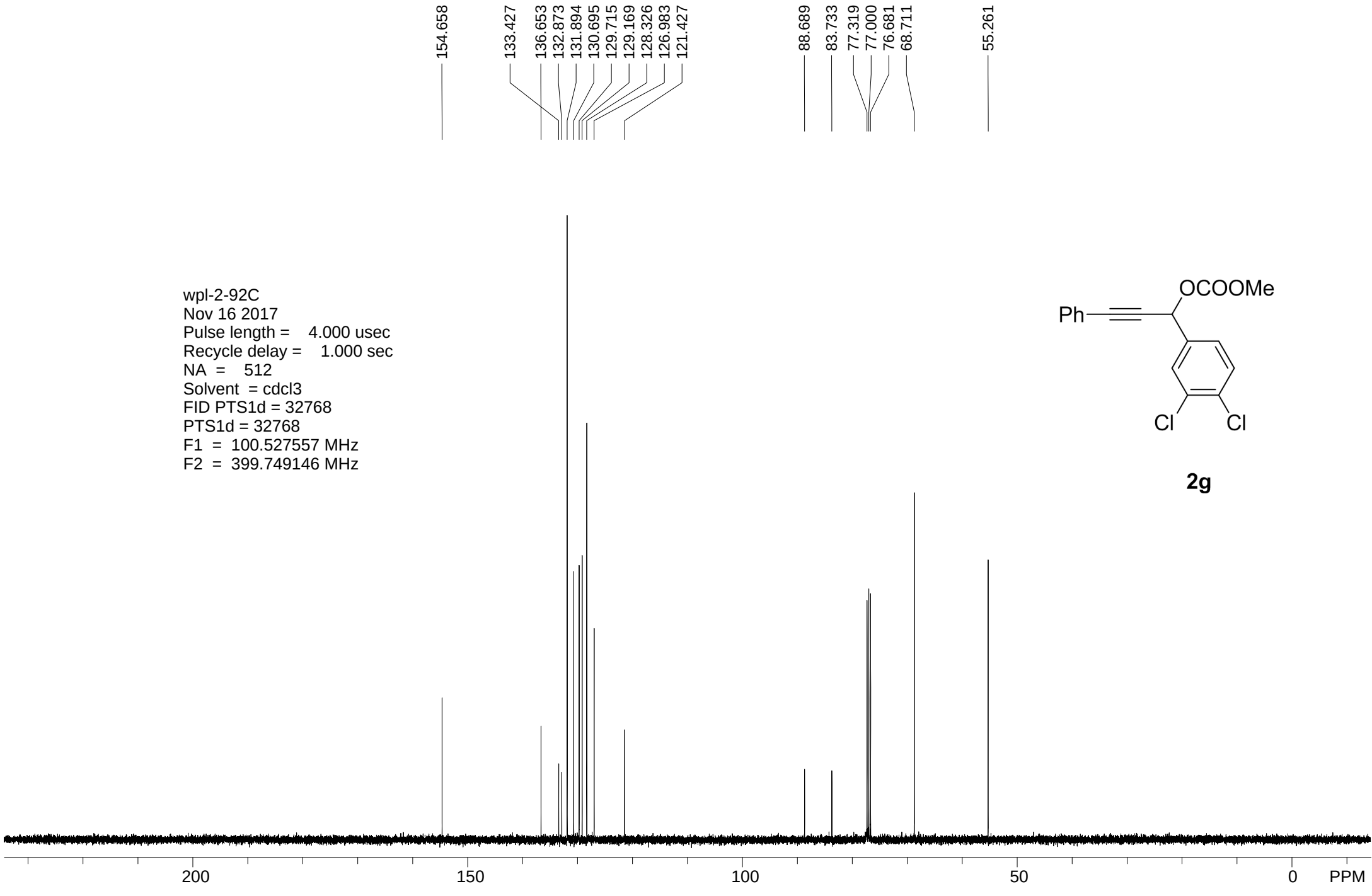
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Recycle delay = 1.000 sec
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F2 = 399.749146 MHz
OCOOMe
Ph
Cl
Cl
2g
200
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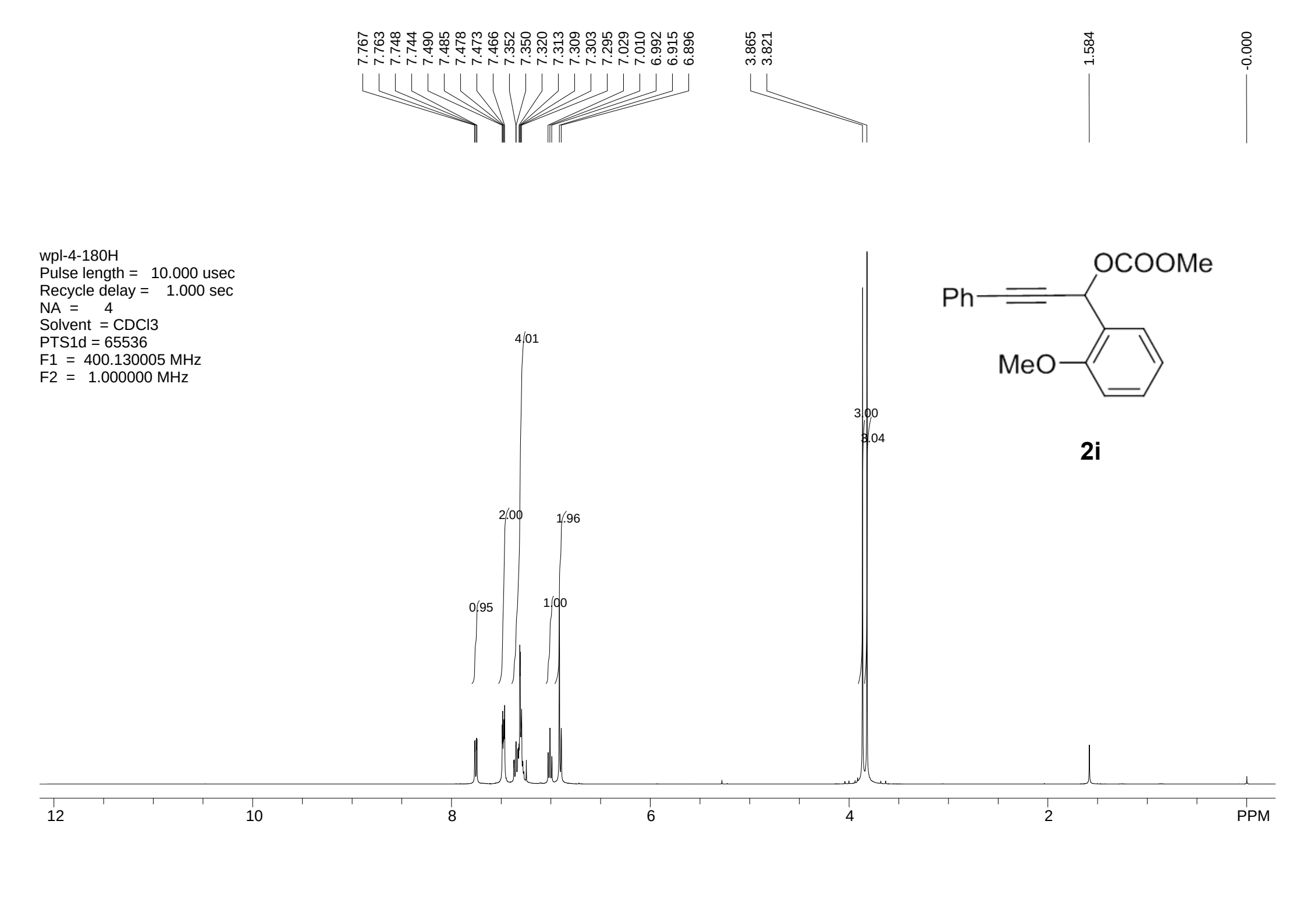
100
50
0
PPM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Ph
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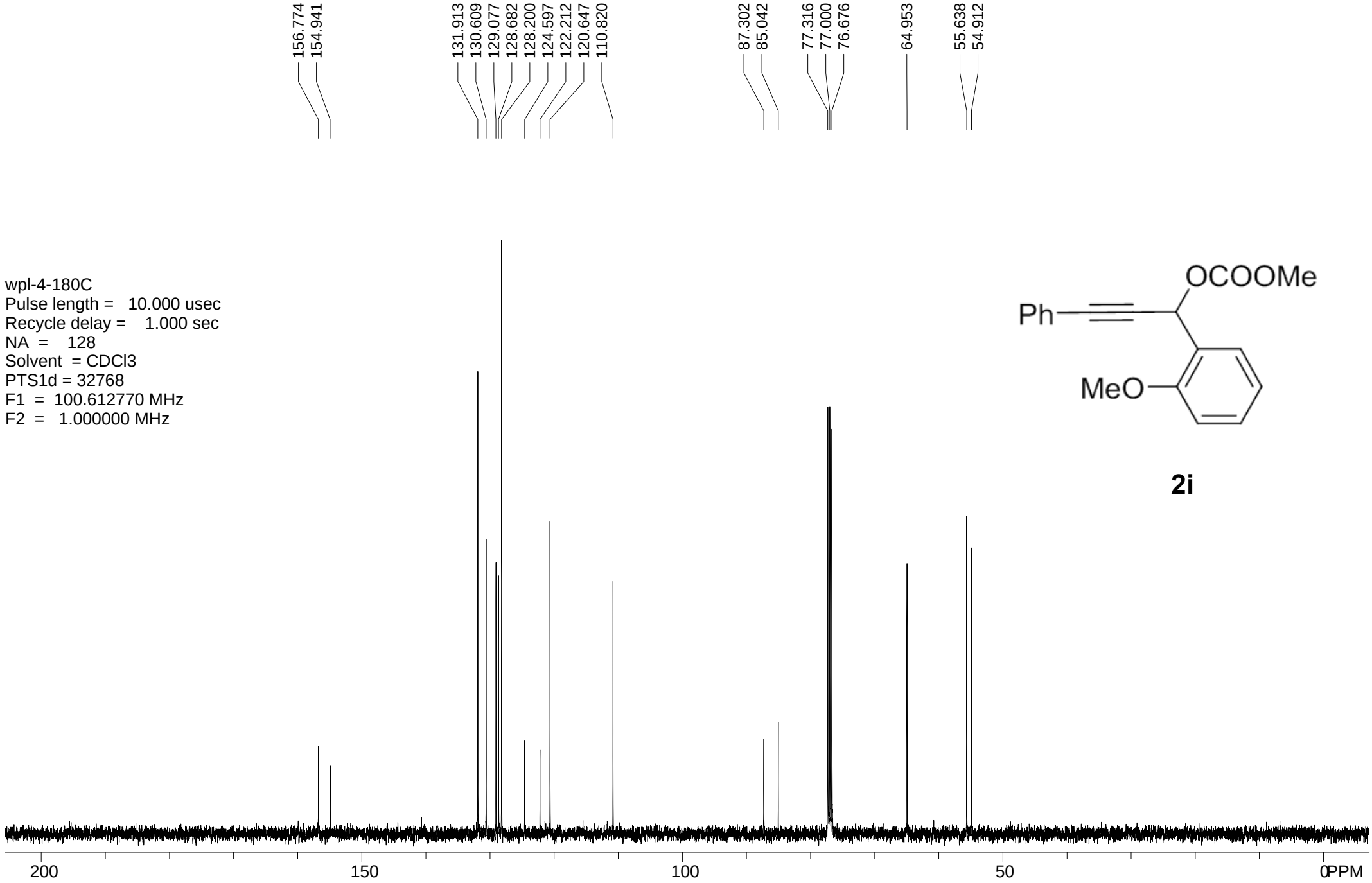
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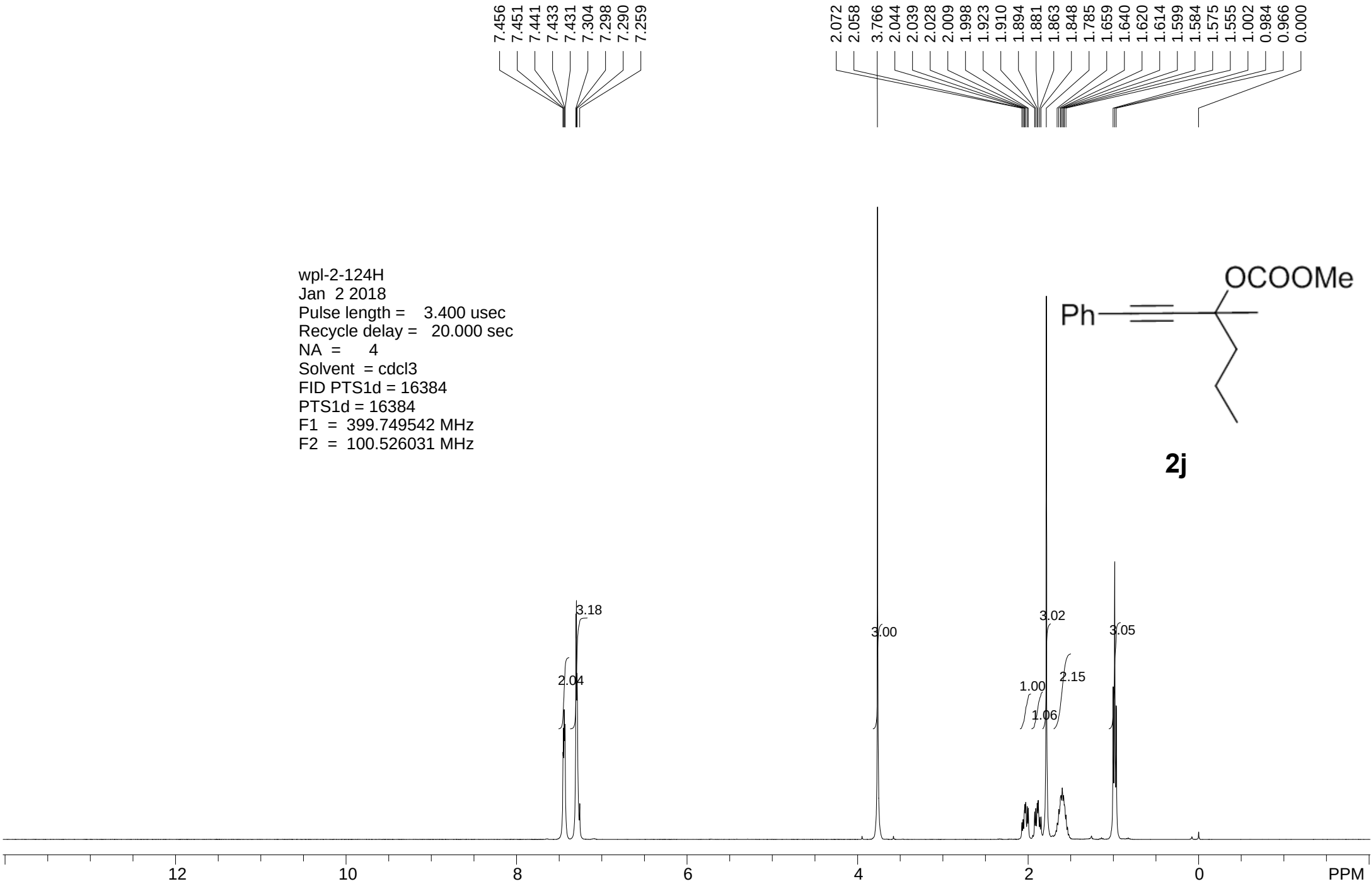
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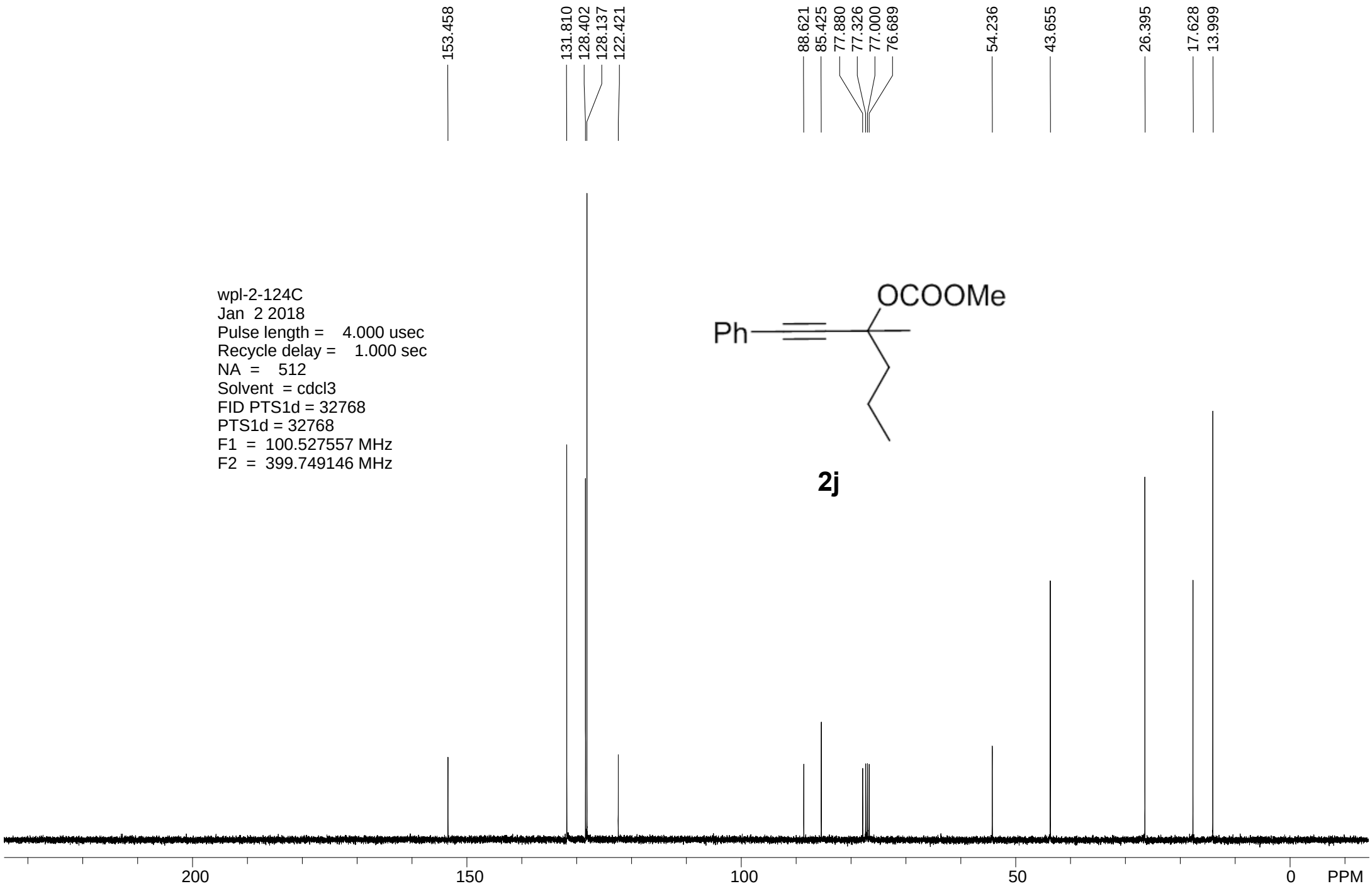
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Jan 2 2018
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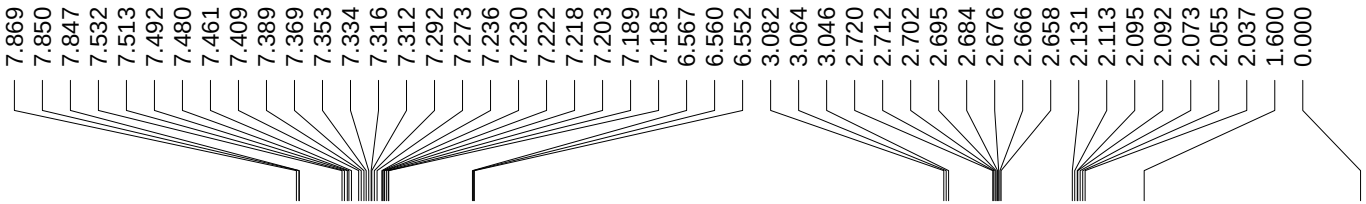
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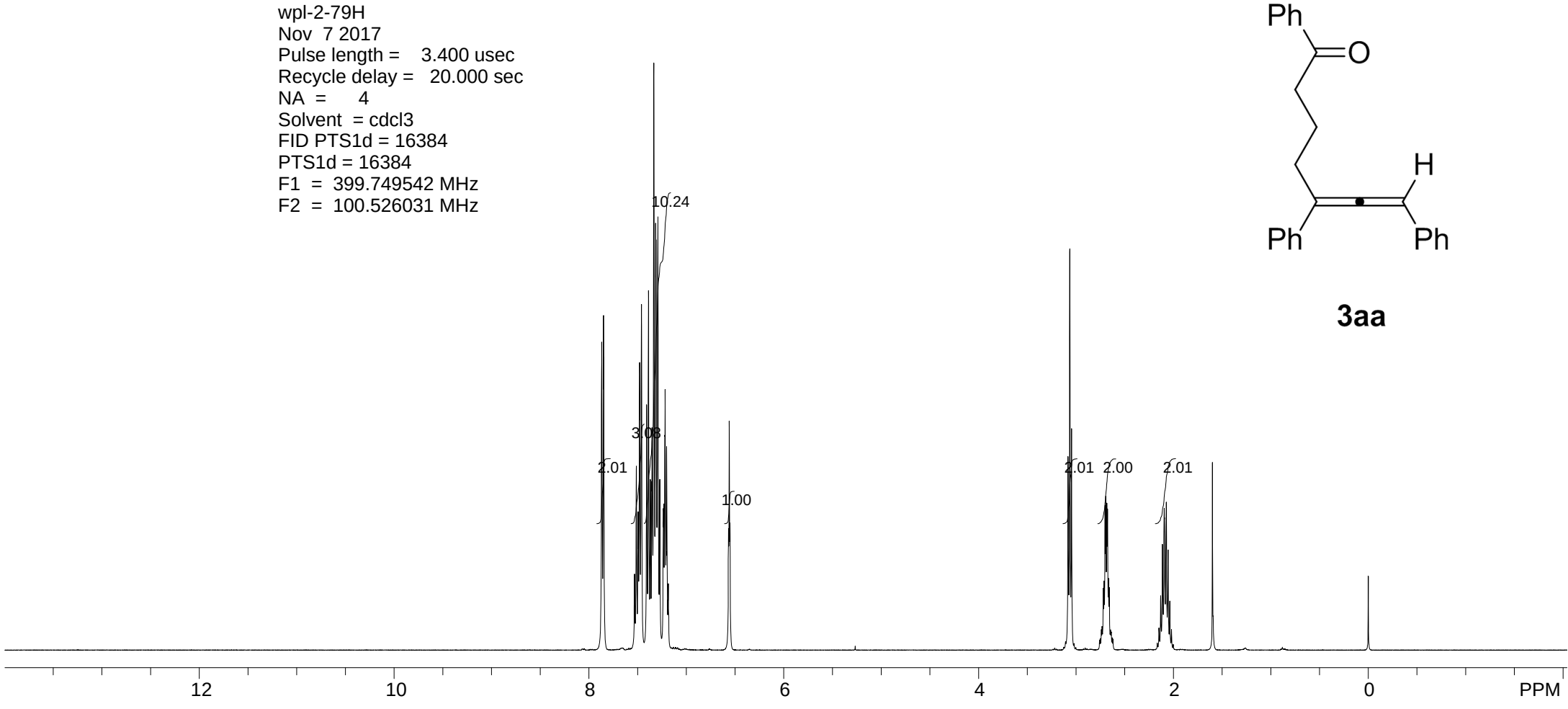
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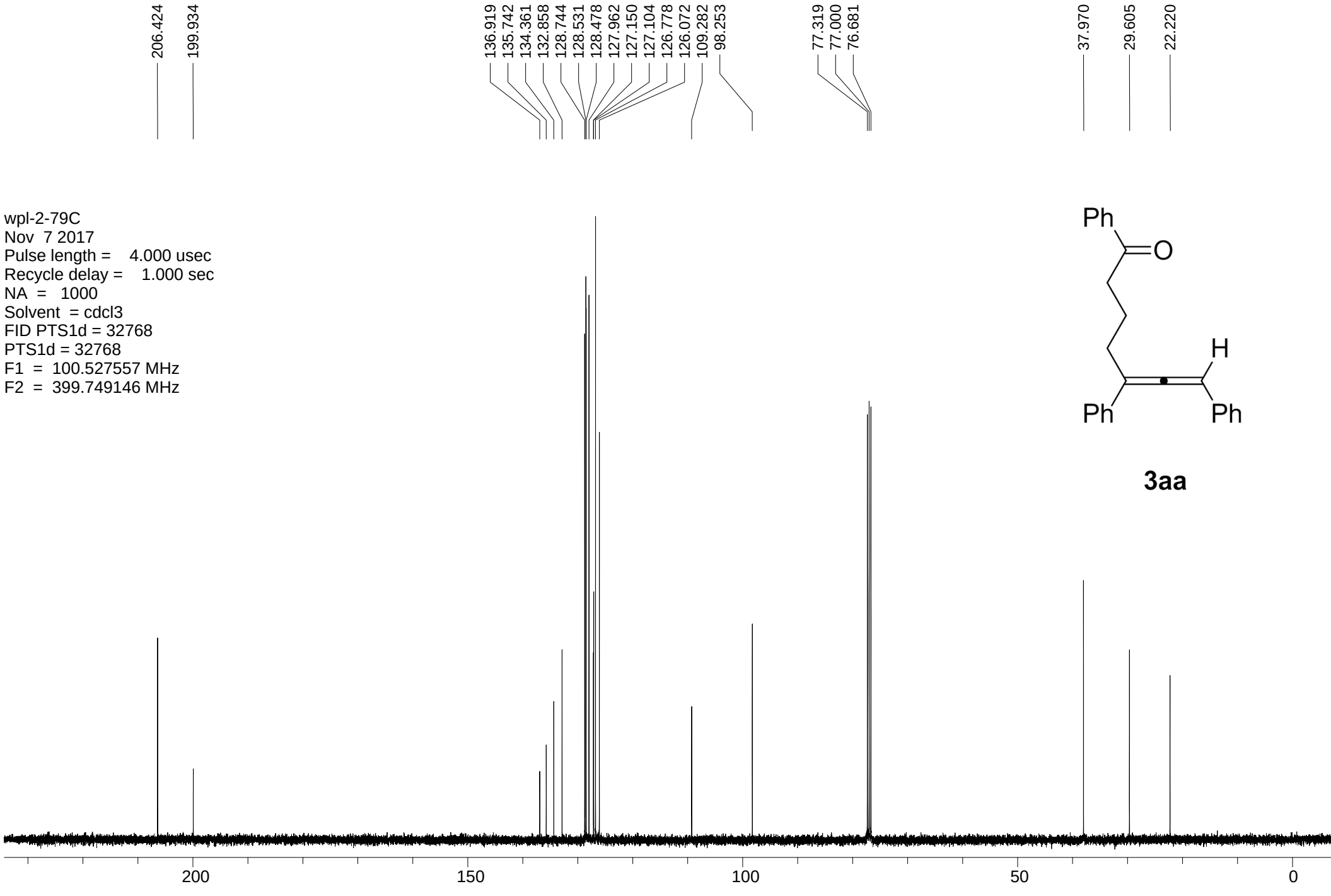
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wpl-2-79C
Nov 7 2017
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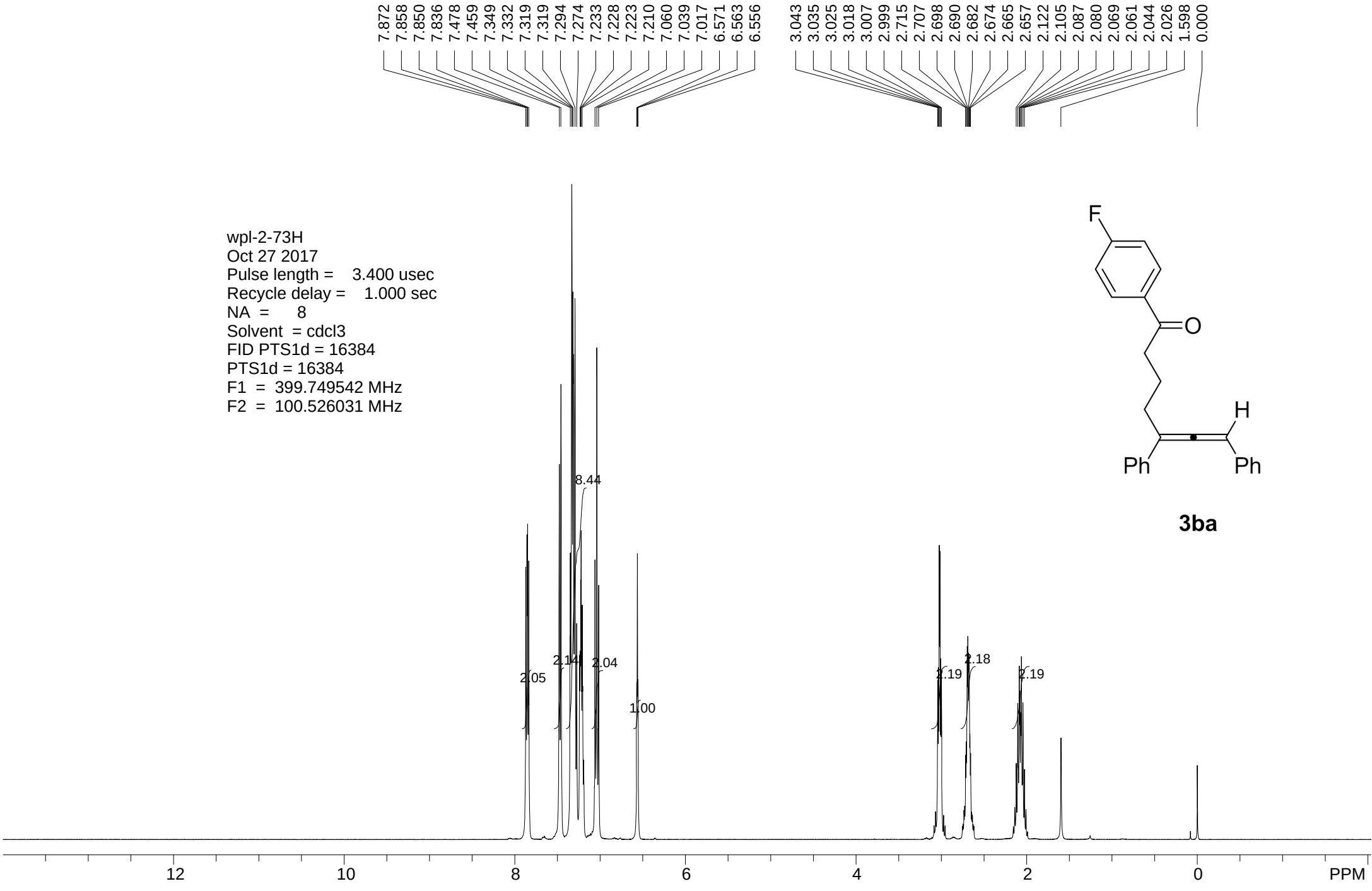
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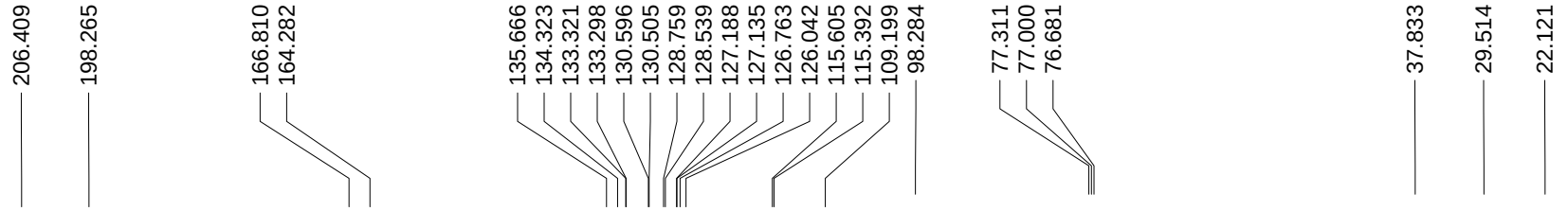
PPM



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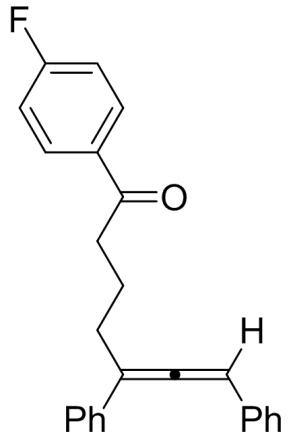
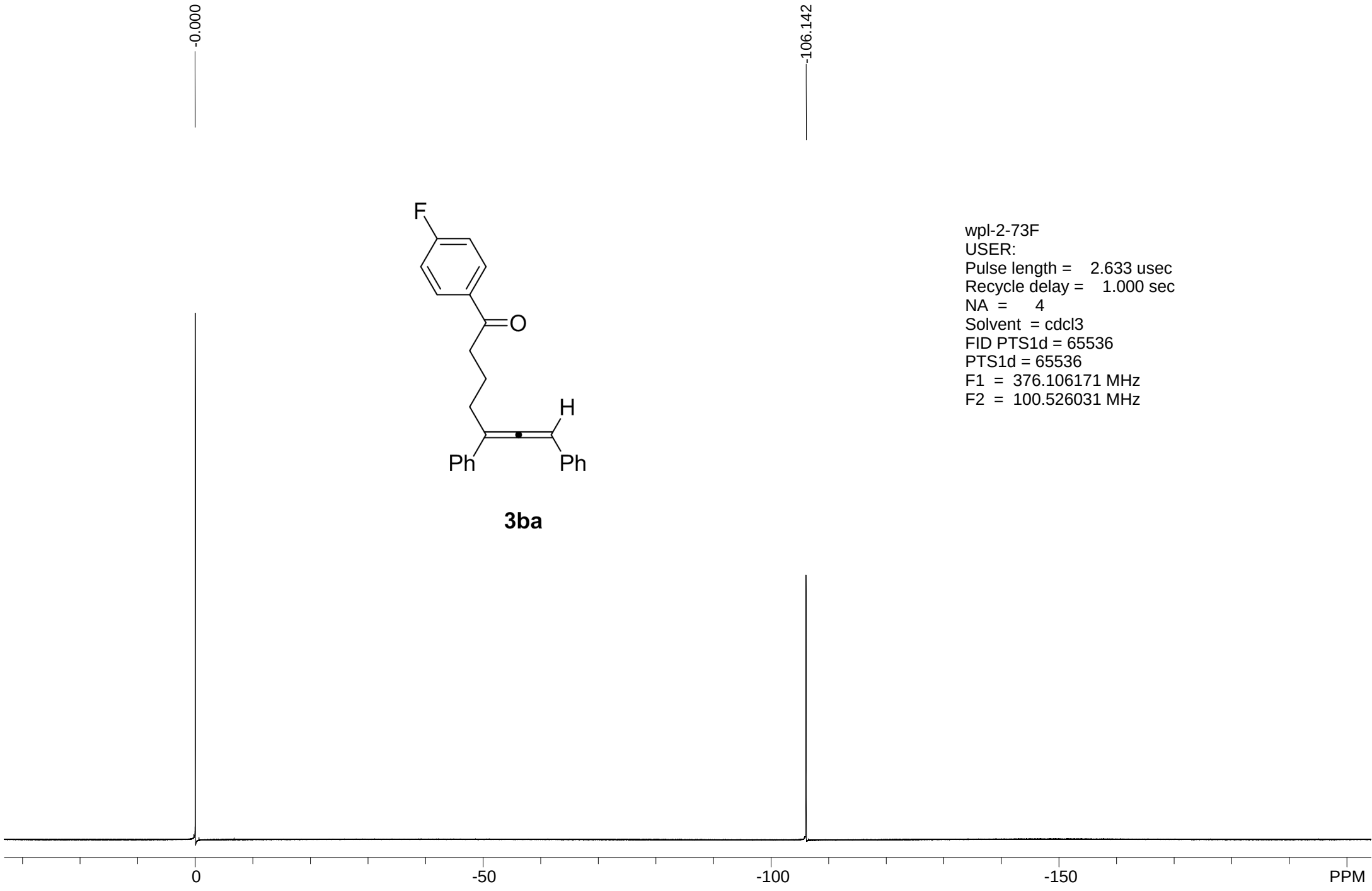
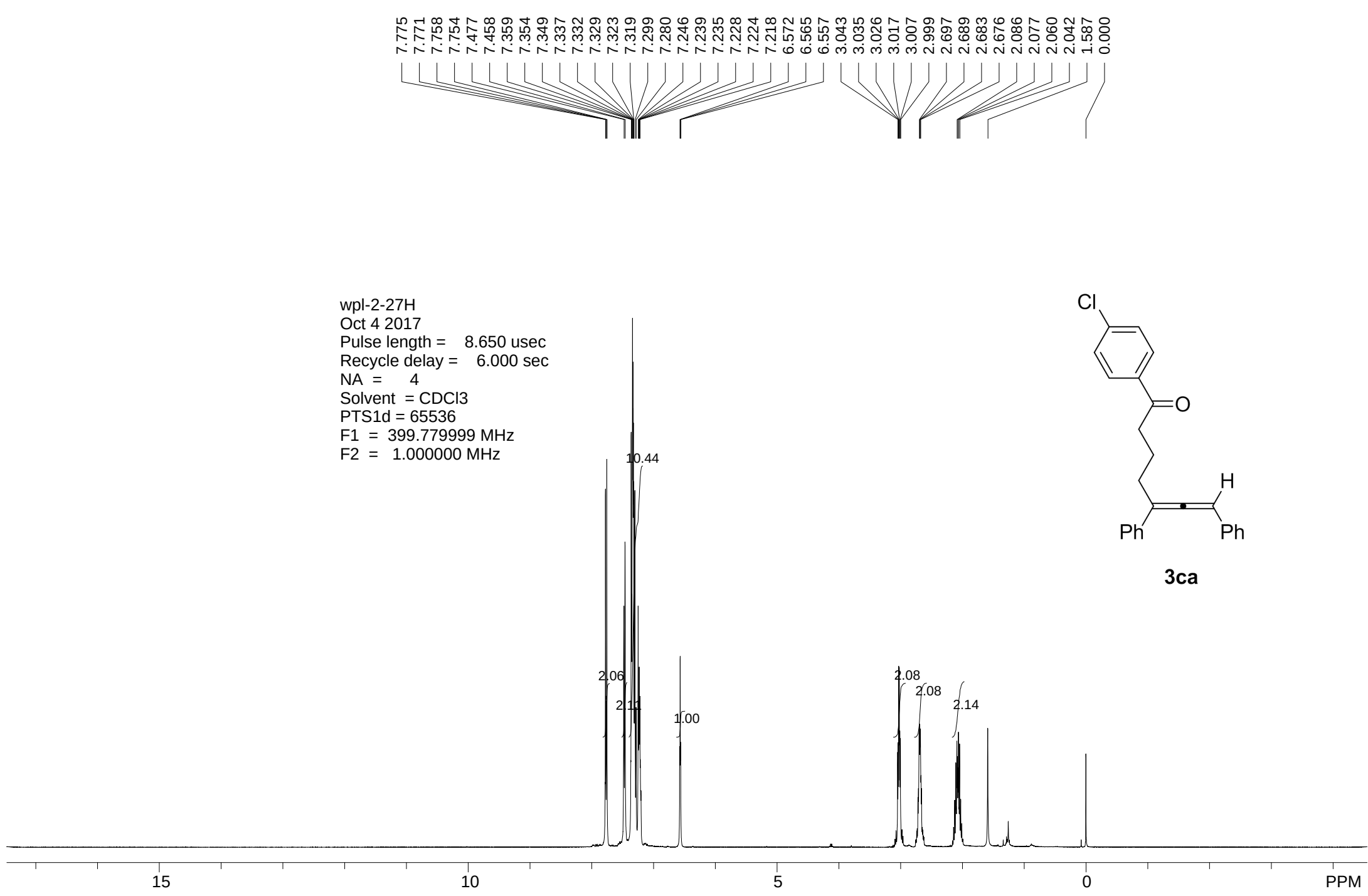
FID PTS1d = 32768
PTS1d = 32768
F1 = 100.527557 MHz
F2 = 399.749146 MHz

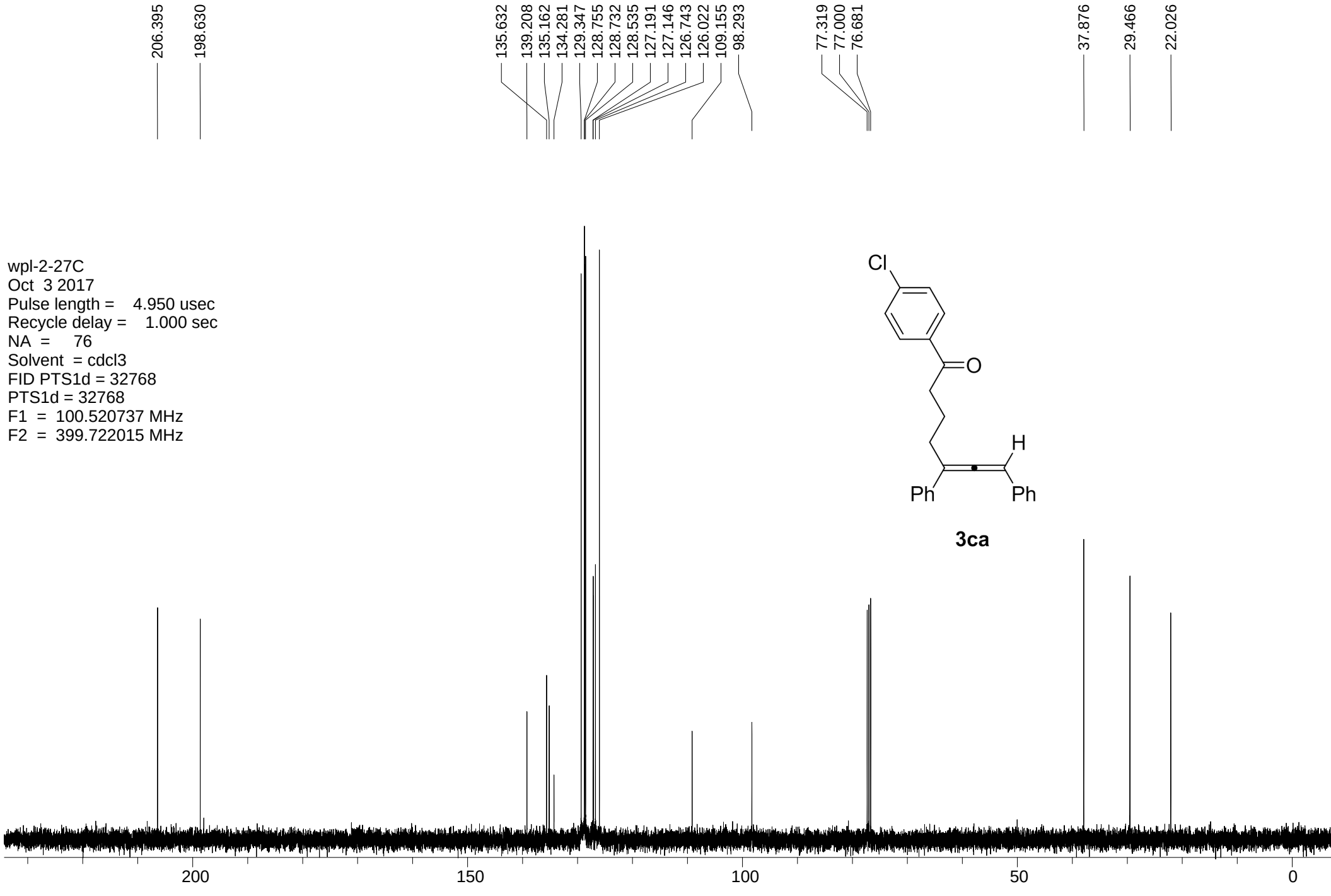


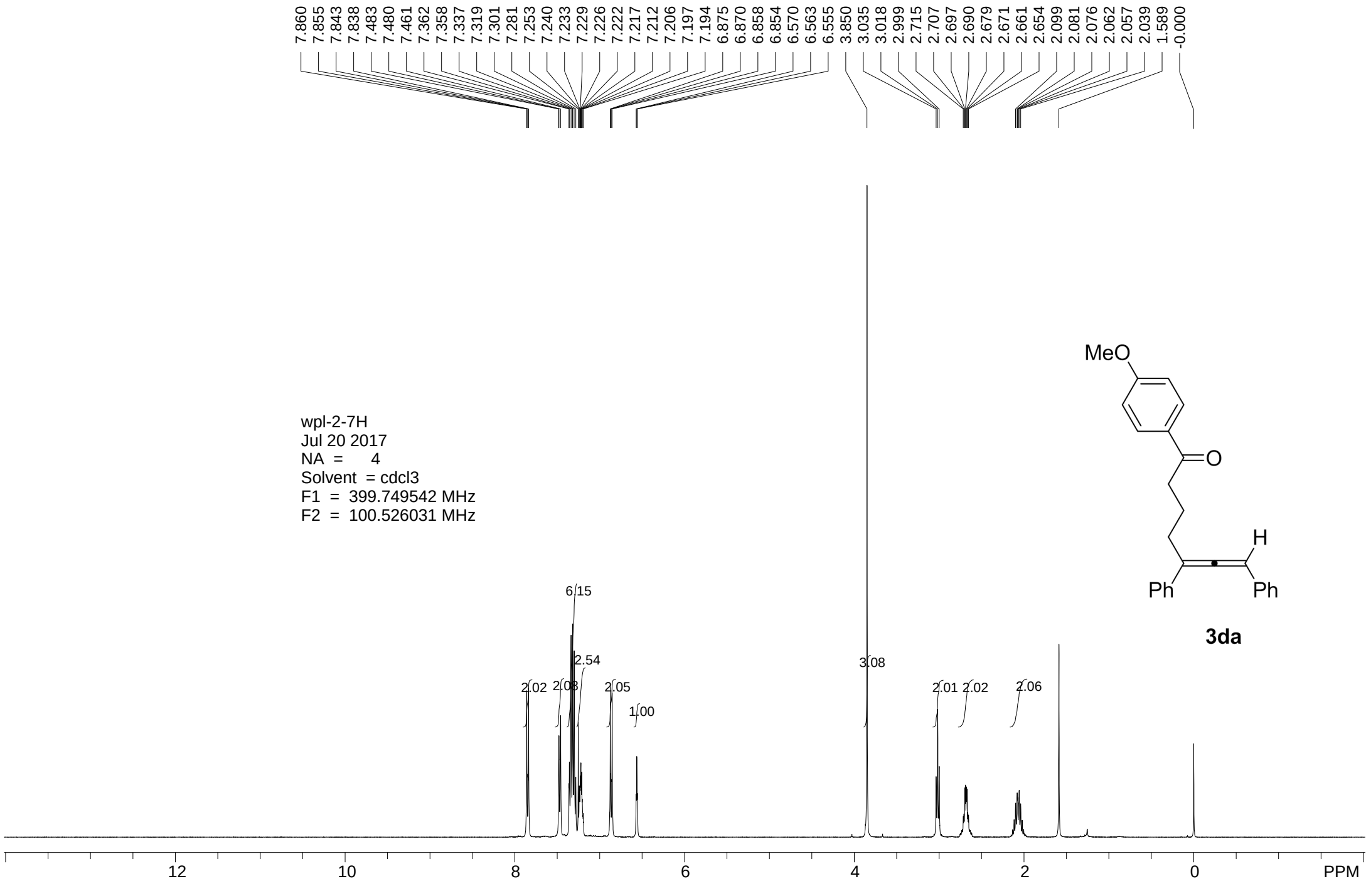
3ba

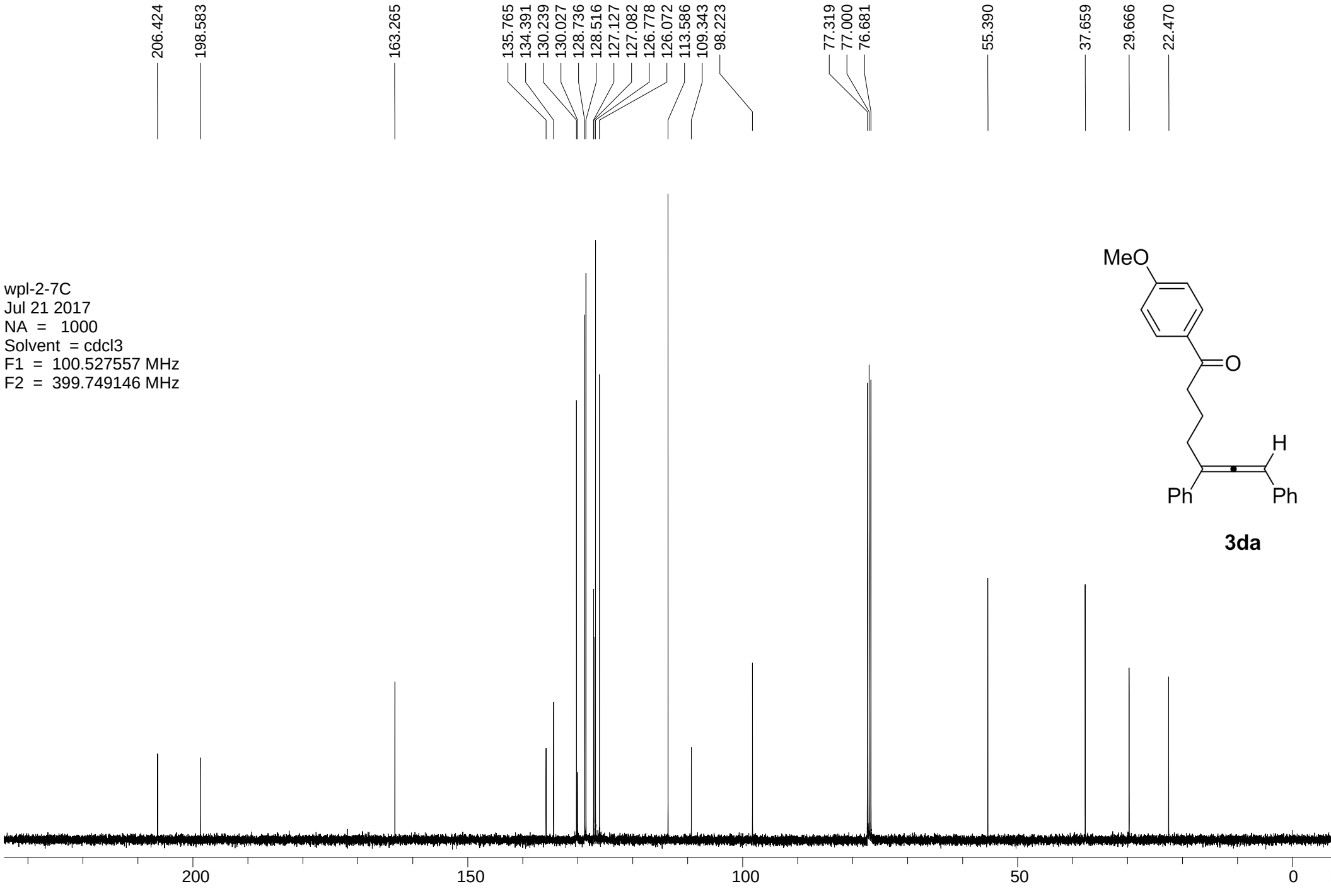

-0.000
-106.142
F
O
H
Ph
Ph
wpl-2-73F
USER:
Pulse length = 2.633 usec
Recycle delay = 1.000 sec
NA = 4
Solvent = cdcl3
FID PTS1d = 65536
PTS1d = 65536
F1 = 376.106171 MHz
F2 = 100.526031 MHz
0
-50
-100
-150
PPM

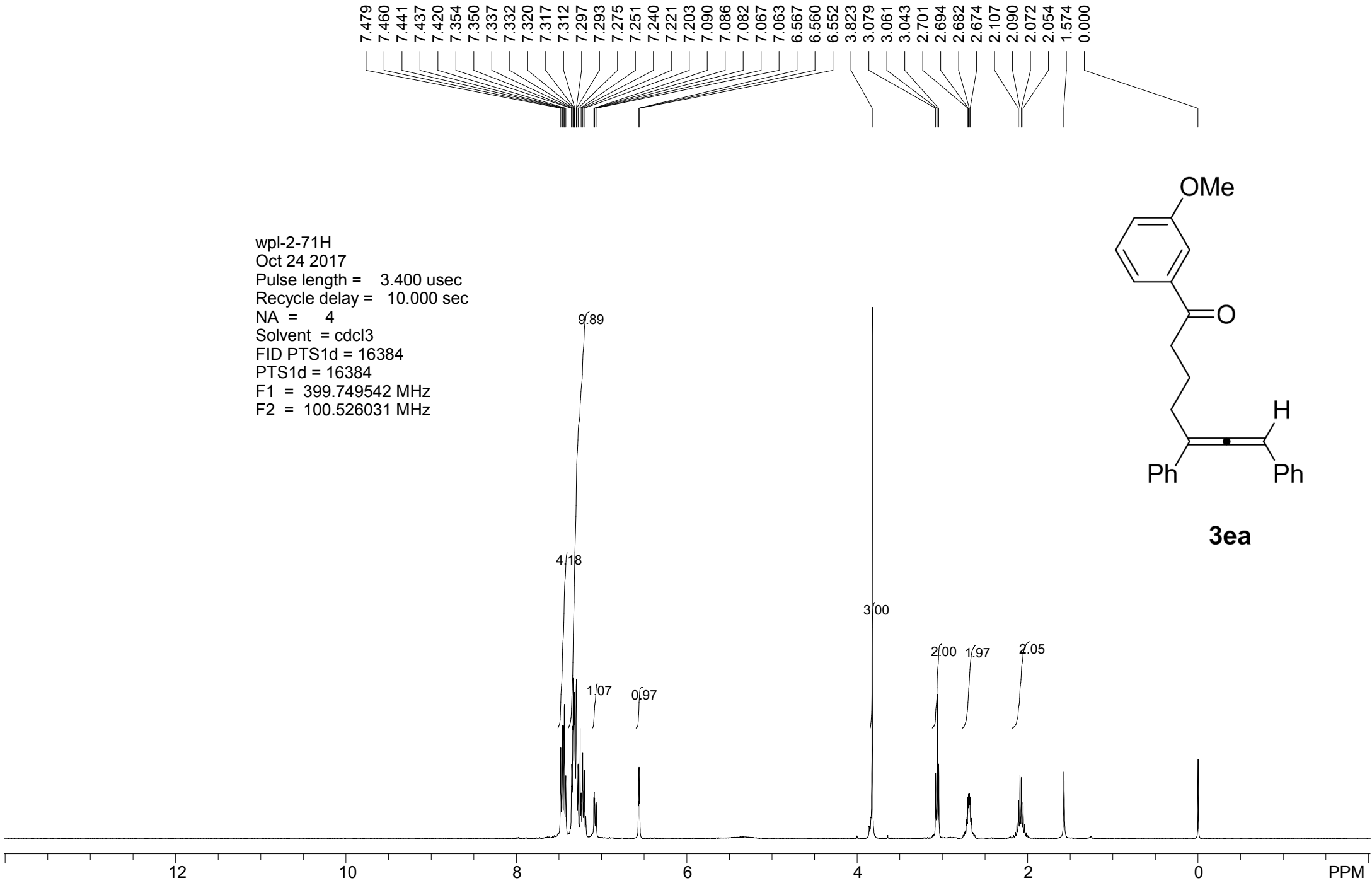
3ba

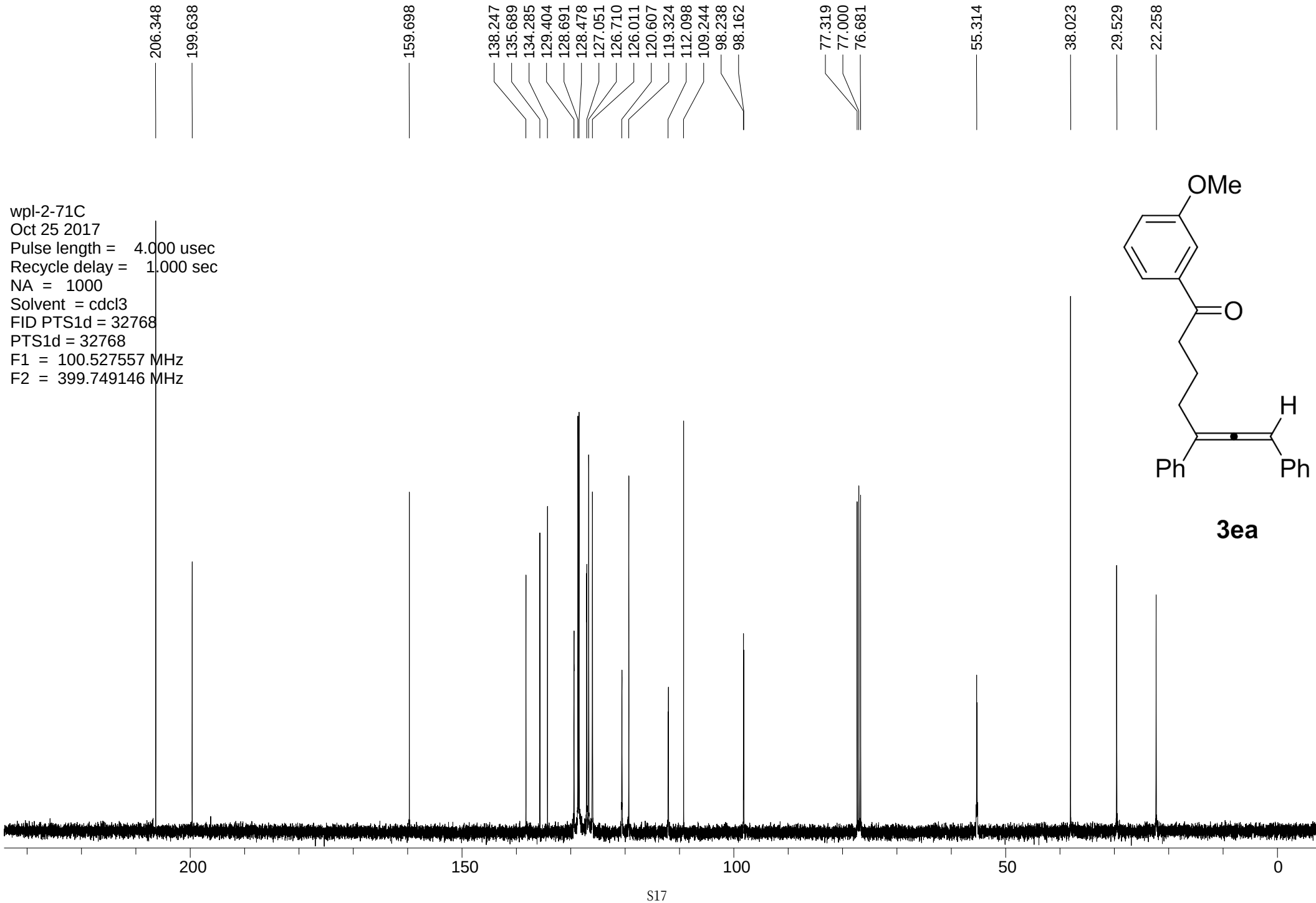

7.775
7.771
7.758
7.754
7.477
7.458
7.359
7.354
7.349
7.337
7.332
7.329
7.323
7.319
7.299
7.280
7.246
7.239
7.235
7.228
7.224
7.218
6.572
6.565
6.557
3.043
3.035
3.026
3.017
3.007
2.999
2.697
2.689
2.683
2.676
2.086
2.077
2.060
2.042
1.587
0.000
wpl-2-27H
Oct 4 2017
Pulse length = 8.650 usec
Recycle delay = 6.000 sec
NA = 4
Solvent = CDCl3
PTS1d = 65536
F1 = 399.779999 MHz
F2 = 1.000000 MHz
10.44
2.06
2.11
1.00
2.08
2.08
2.14
Cl
O
H
Ph
Ph
3ca
15
10
5
0
PPM

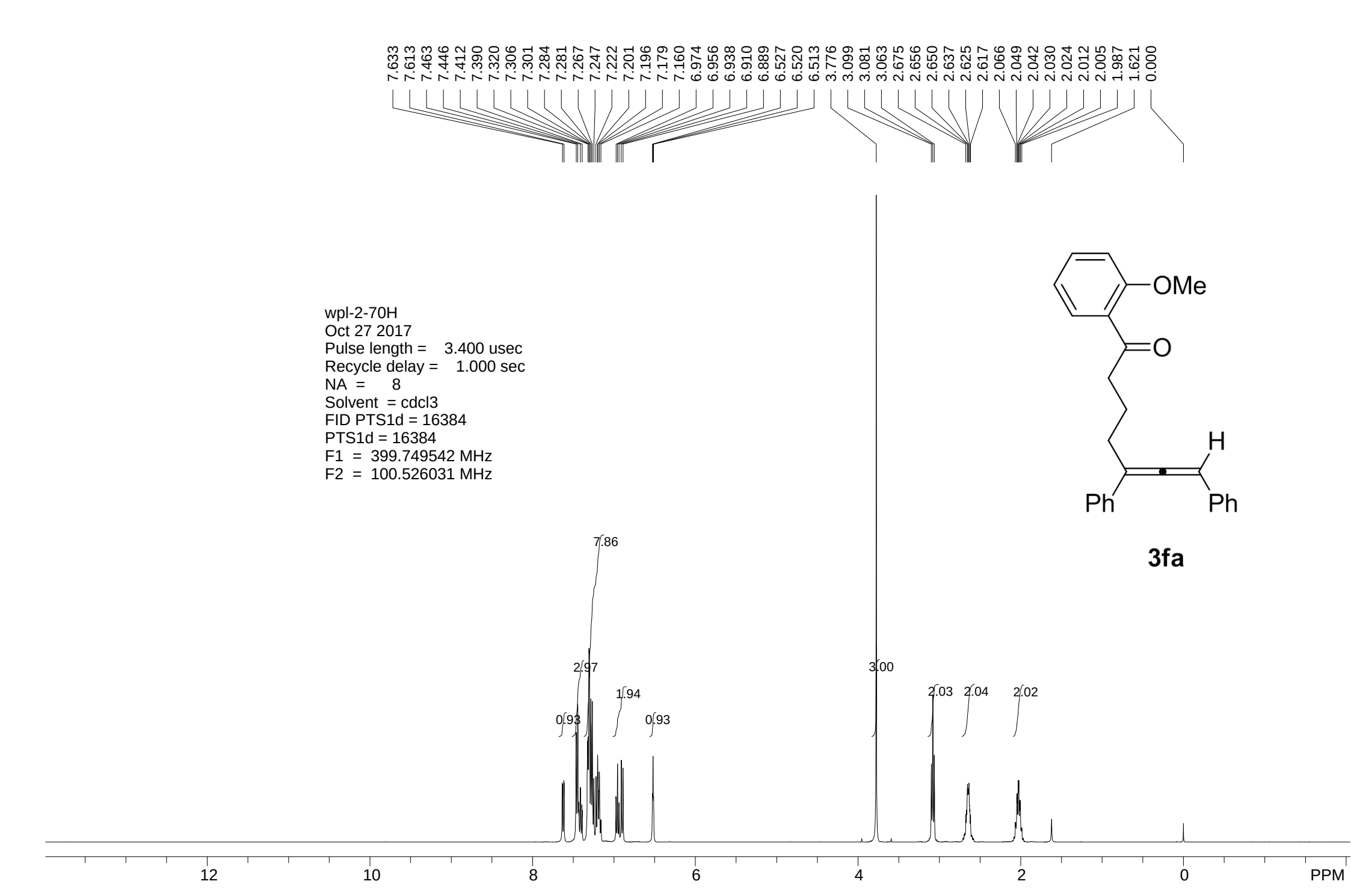

206.395
198.630
135.632
139.208
135.162
134.281
129.347
128.755
128.732
128.535
127.191
127.146
126.743
126.022
109.155
98.293
77.319
77.000
76.681
37.876
29.466
22.026
wpl-2-27C
Oct 3 2017
Pulse length = 4.950 usec
Recycle delay = 1.000 sec
NA = 76
Solvent = cdcl3
FID PTS1d = 32768
PTS1d = 32768
F1 = 100.520737 MHz
F2 = 399.722015 MHz
Cl
O
H
Ph
Ph
3ca
200
150
100
50
0
PPM

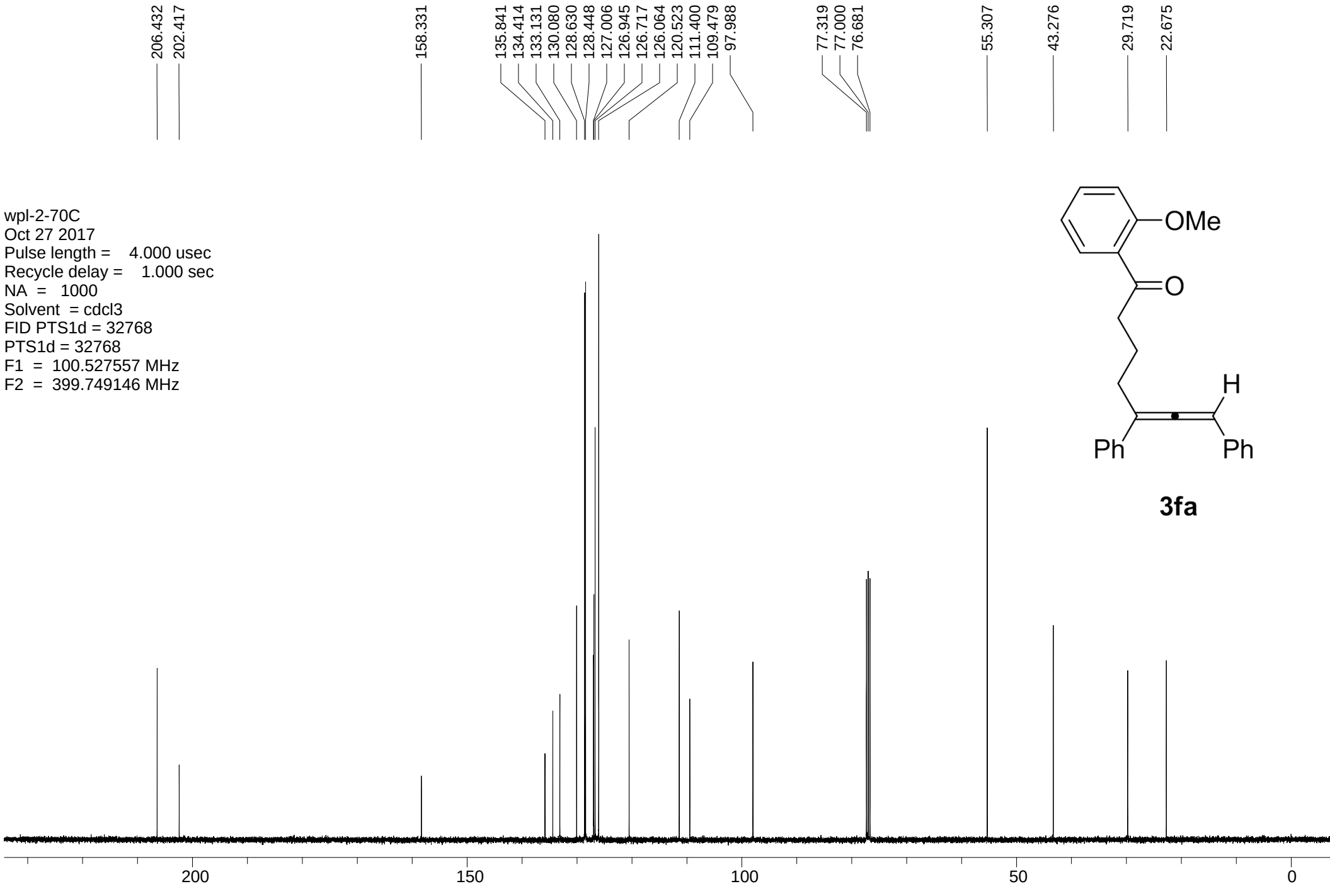

7.860
7.855
7.843
7.838
7.483
7.480
7.461
7.362
7.358
7.337
7.319
7.301
7.281
7.253
7.240
7.233
7.229
7.226
7.222
7.217
7.212
7.206
7.197
7.194
6.875
6.870
6.858
6.854
6.570
6.563
6.555
3.850
3.035
3.018
2.999
2.715
2.707
2.697
2.690
2.679
2.671
2.661
2.654
2.099
2.081
2.076
2.062
2.057
2.039
1.589
-0.000
wpl-2-7H
Jul 20 2017
NA = 4
Solvent = cdcl3
F1 = 399.749542 MHz
F2 = 100.526031 MHz
MeO
O
H
Ph
Ph
3da
6.15
2.54
2.02
2.08
2.05
1.00
3.08
2.01
2.02
2.06
12
10
8
6
4
2
0
PPM

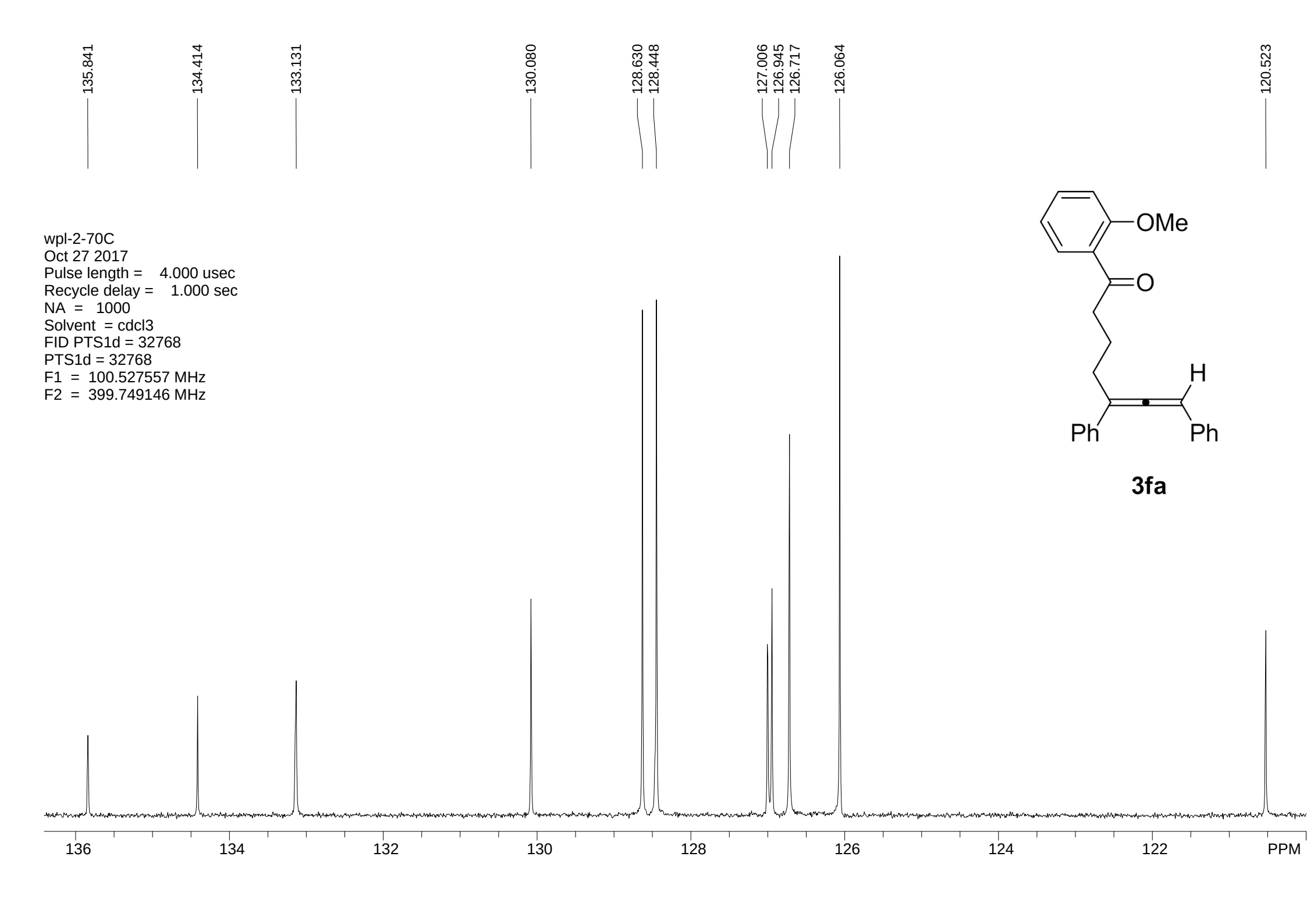

206.424
198.583
163.265
135.765
134.391
130.239
130.027
128.736
128.516
127.127
127.082
126.778
126.072
113.586
109.343
98.223
77.319
77.000
76.681
55.390
37.659
29.666
22.470
wpl-2-7C
Jul 21 2017
NA = 1000
Solvent = cdcl3
F1 = 100.527557 MHz
F2 = 399.749146 MHz
MeO
O
H
Ph
Ph
3da
200
150
100
50
0
PPM

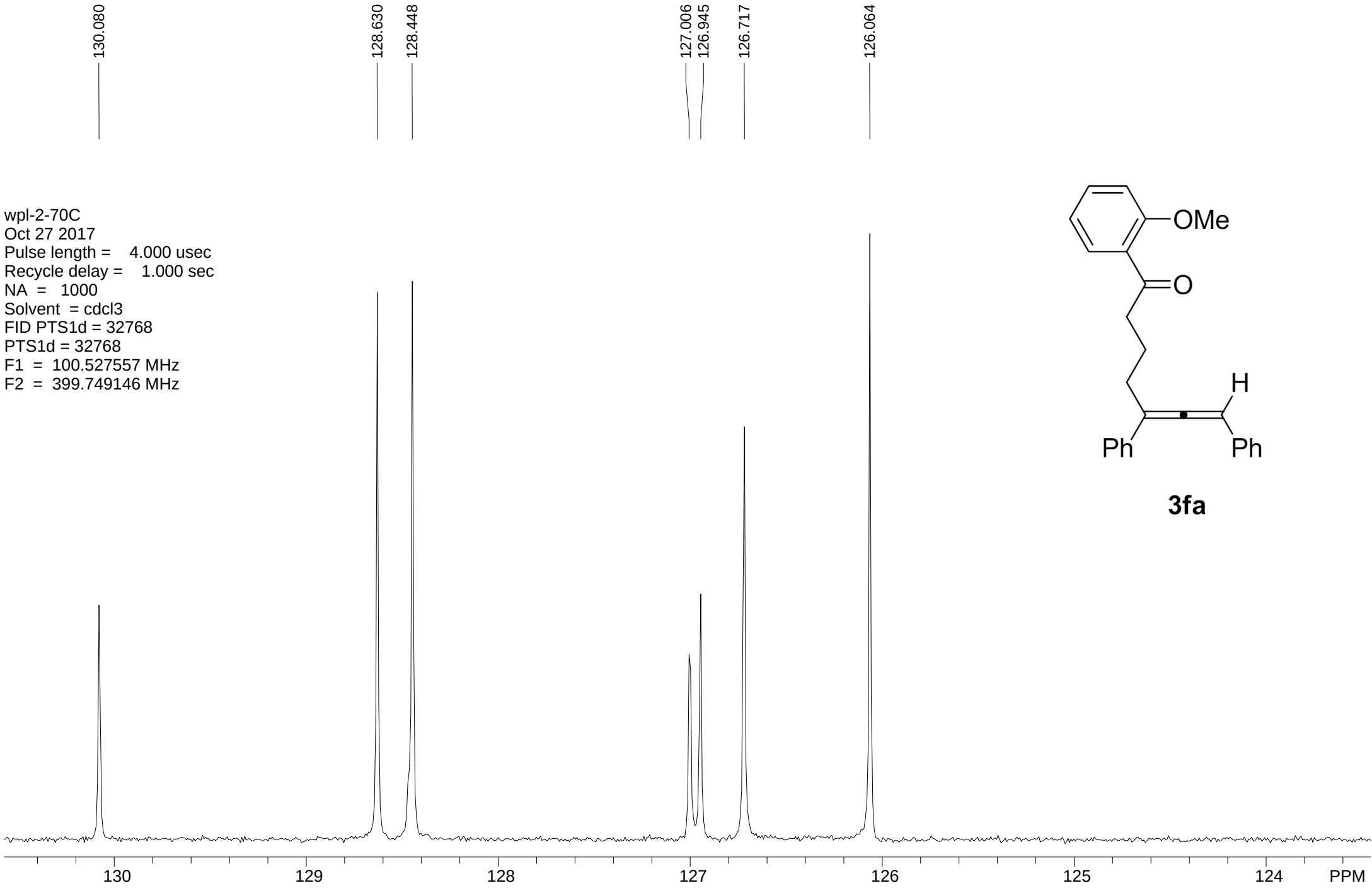

7.479
7.460
7.441
7.437
7.420
7.354
7.350
7.337
7.332
7.320
7.317
7.312
7.297
7.293
7.275
7.251
7.240
7.221
7.203
7.090
7.086
7.082
7.067
7.063
6.567
6.560
6.552
3.823
3.079
3.061
3.043
2.701
2.694
2.682
2.674
2.107
2.090
2.072
2.054
1.574
0.000
wpl-2-71H
Oct 24 2017
Pulse length = 3.400 usec
Recycle delay = 10.000 sec
NA = 4
Solvent = cdcl3
FID PTS1d = 16384
PTS1d = 16384
F1 = 399.749542 MHz
F2 = 100.526031 MHz
OMe
O
H
Ph
Ph
3ea
9.89
4.18
1.07
0.97
3.00
2.00
1.97
2.05
12
10
8
6
4
2
0
PPM

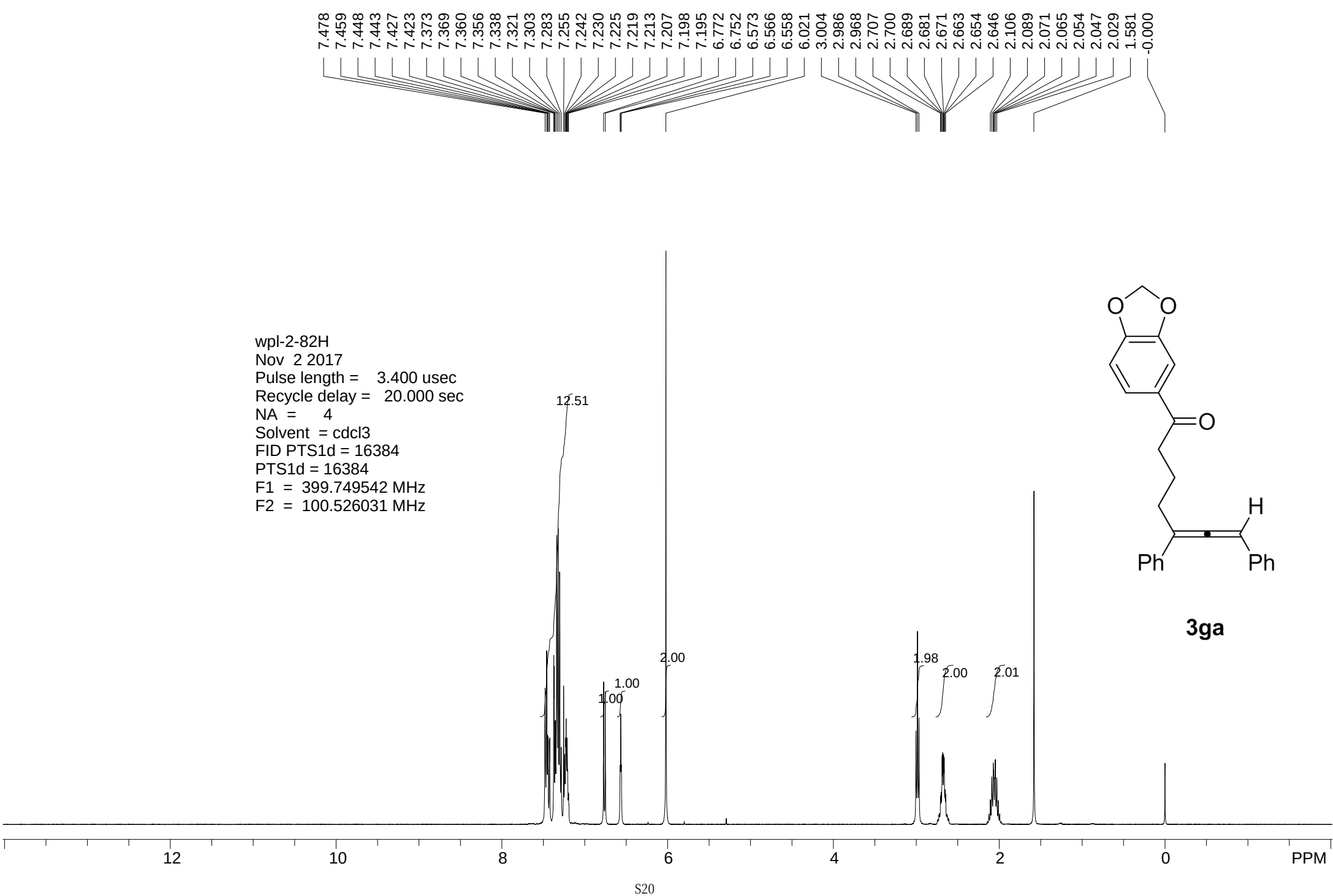

206.348
199.638
159.698
138.247
135.689
134.285
129.404
128.691
128.478
127.051
126.710
126.011
120.607
119.324
112.098
109.244
98.238
98.162
77.319
77.000
76.681
55.314
38.023
29.529
22.258
wpl-2-71C
Oct 25 2017
Pulse length = 4.000 usec
Recycle delay = 1.000 sec
NA = 1000
Solvent = cdcl3
FID PTS1d = 32768
PTS1d = 32768
F1 = 100.527557 MHz
F2 = 399.749146 MHz
OMe
O
H
Ph
Ph
3ea
200
150
100
50
0
PPM

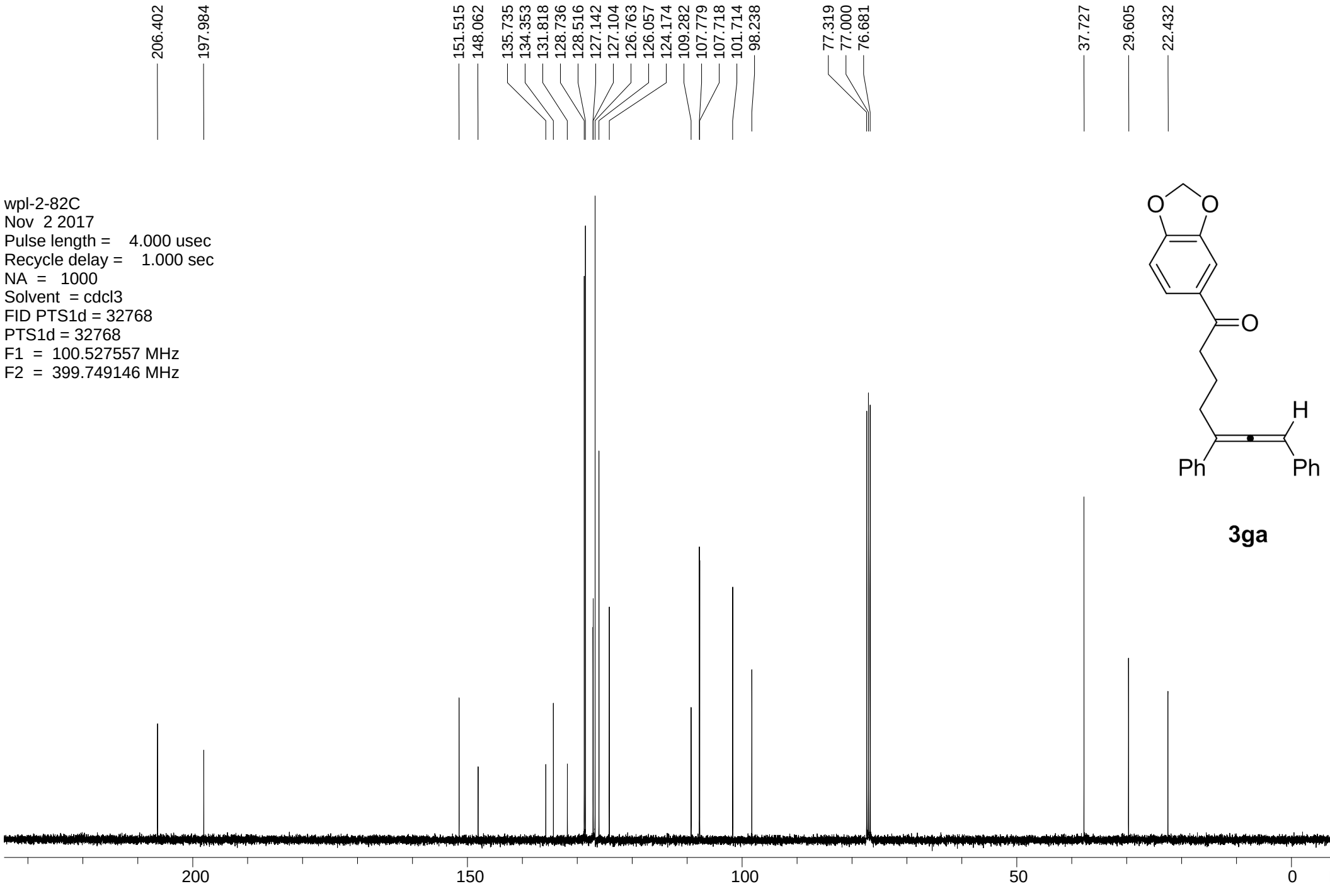

7.633
7.613
7.463
7.446
7.412
7.390
7.320
7.306
7.301
7.284
7.281
7.267
7.247
7.222
7.201
7.196
7.179
7.160
6.974
6.956
6.938
6.910
6.889
6.527
6.520
6.513
3.776
3.099
3.081
3.063
2.675
2.656
2.650
2.637
2.625
2.617
2.066
2.049
2.042
2.030
2.024
2.012
2.005
1.987
1.621
0.000
wpl-2-70H
Oct 27 2017
Pulse length = 3.400 usec
Recycle delay = 1.000 sec
NA = 8
Solvent = cdcl3
FID PTS1d = 16384
PTS1d = 16384
F1 = 399.749542 MHz
F2 = 100.526031 MHz
OMe
O
H
Ph
Ph
3fa
7.86
2.97
1.94
0.93
0.93
3.00
2.03
2.04
2.02
12
10
8
6
4
2
0
PPM

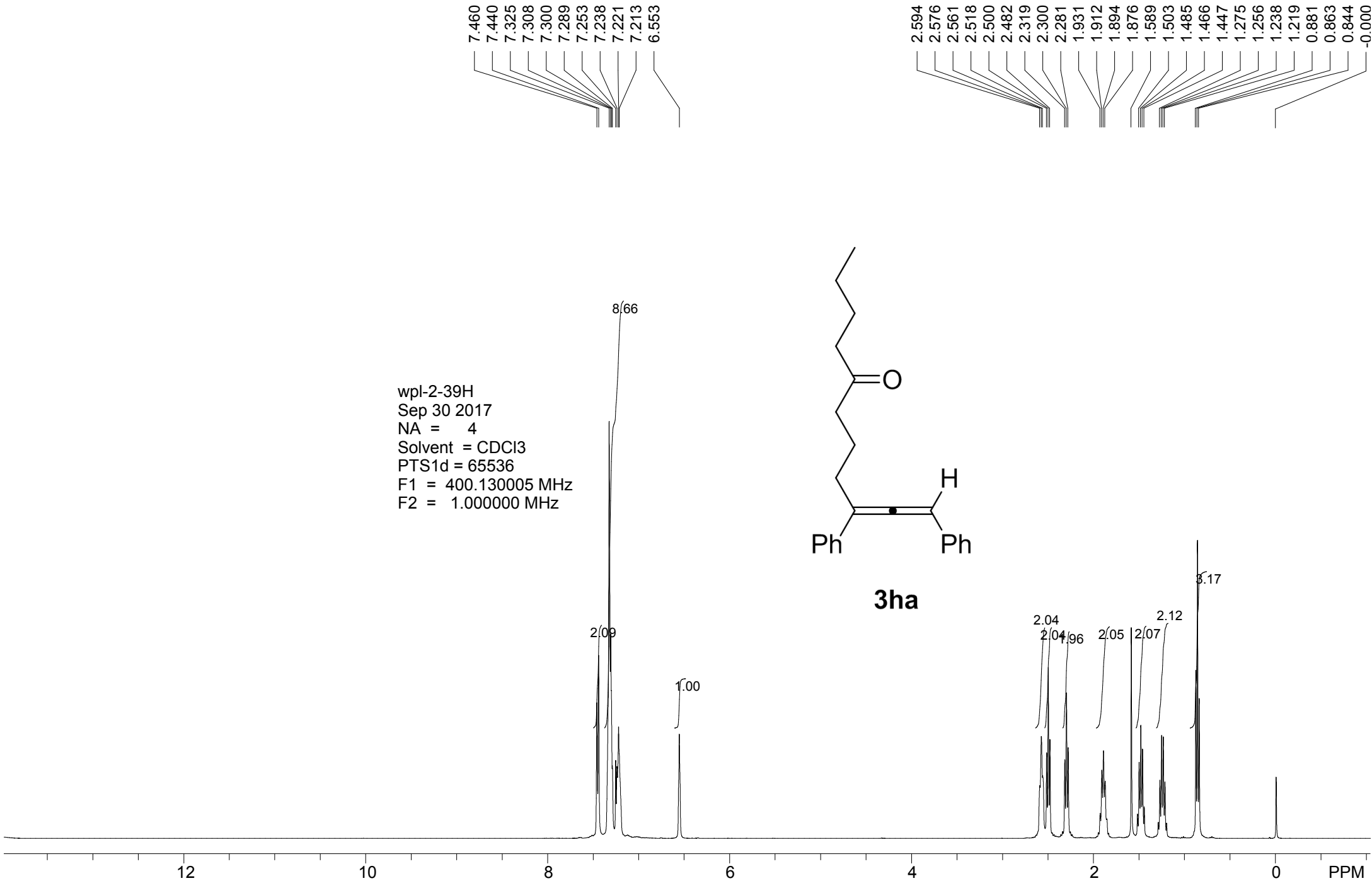

206.432
202.417
158.331
135.841
134.414
133.131
130.080
128.630
128.448
127.006
126.945
126.717
126.064
120.523
111.400
109.479
97.988
77.319
77.000
76.681
55.307
43.276
29.719
22.675
wpl-2-70C
Oct 27 2017
Pulse length = 4.000 usec
Recycle delay = 1.000 sec
NA = 1000
Solvent = cdcl3
FID PTS1d = 32768
PTS1d = 32768
F1 = 100.527557 MHz
F2 = 399.749146 MHz
OMe
O
H
Ph
Ph
3fa
200
150
100
50
0
PPM

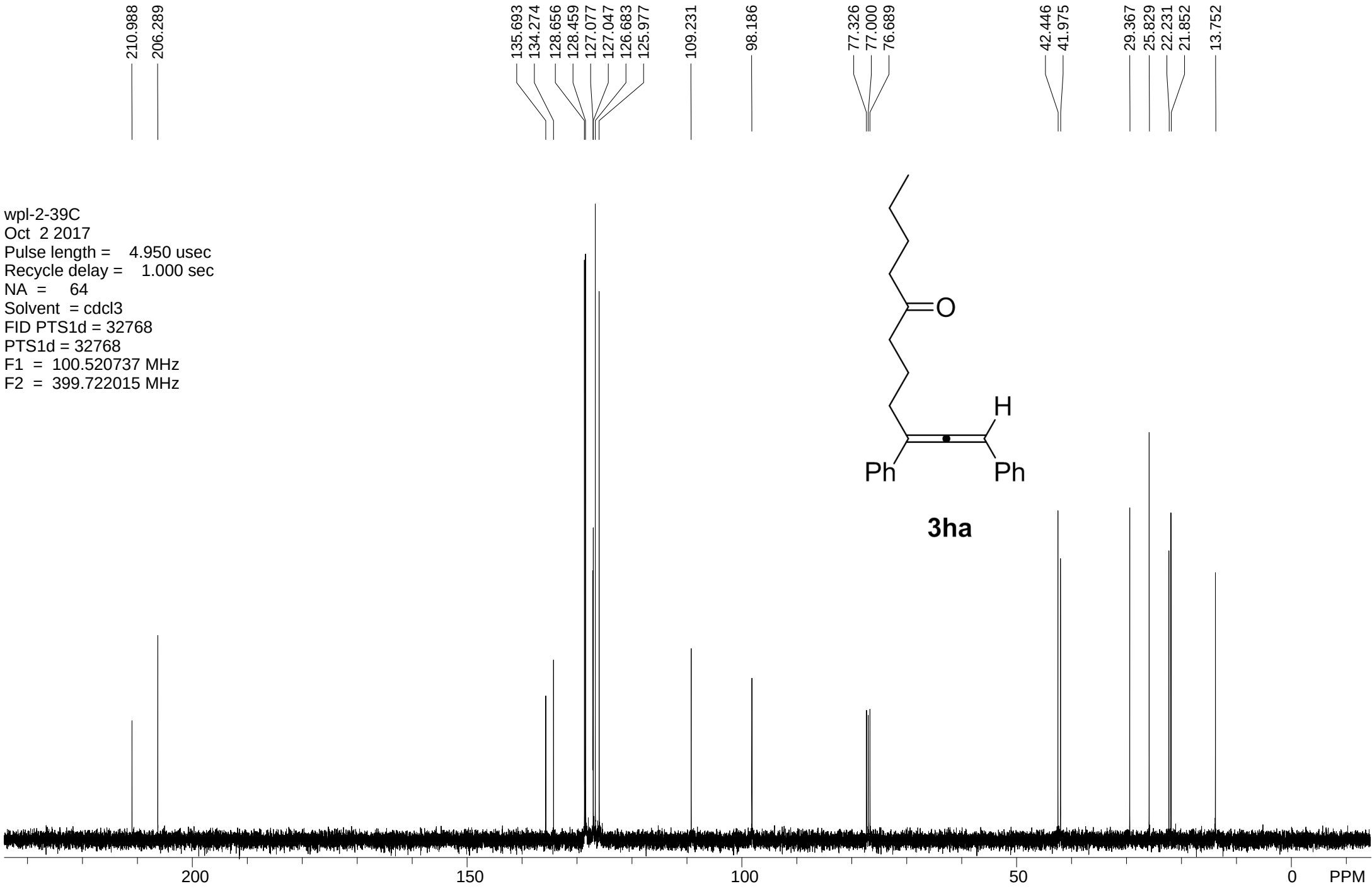

135.841
134.414
133.131
130.080
128.630
128.448
127.006
126.945
126.717
126.064
120.523
wpl-2-70C
Oct 27 2017
Pulse length = 4.000 usec
Recycle delay = 1.000 sec
NA = 1000
Solvent = cdcl3
FID PTS1d = 32768
PTS1d = 32768
F1 = 100.527557 MHz
F2 = 399.749146 MHz
OMe
O
H
Ph
Ph
3fa
136
134
132
130
128
126
124
122
PPM

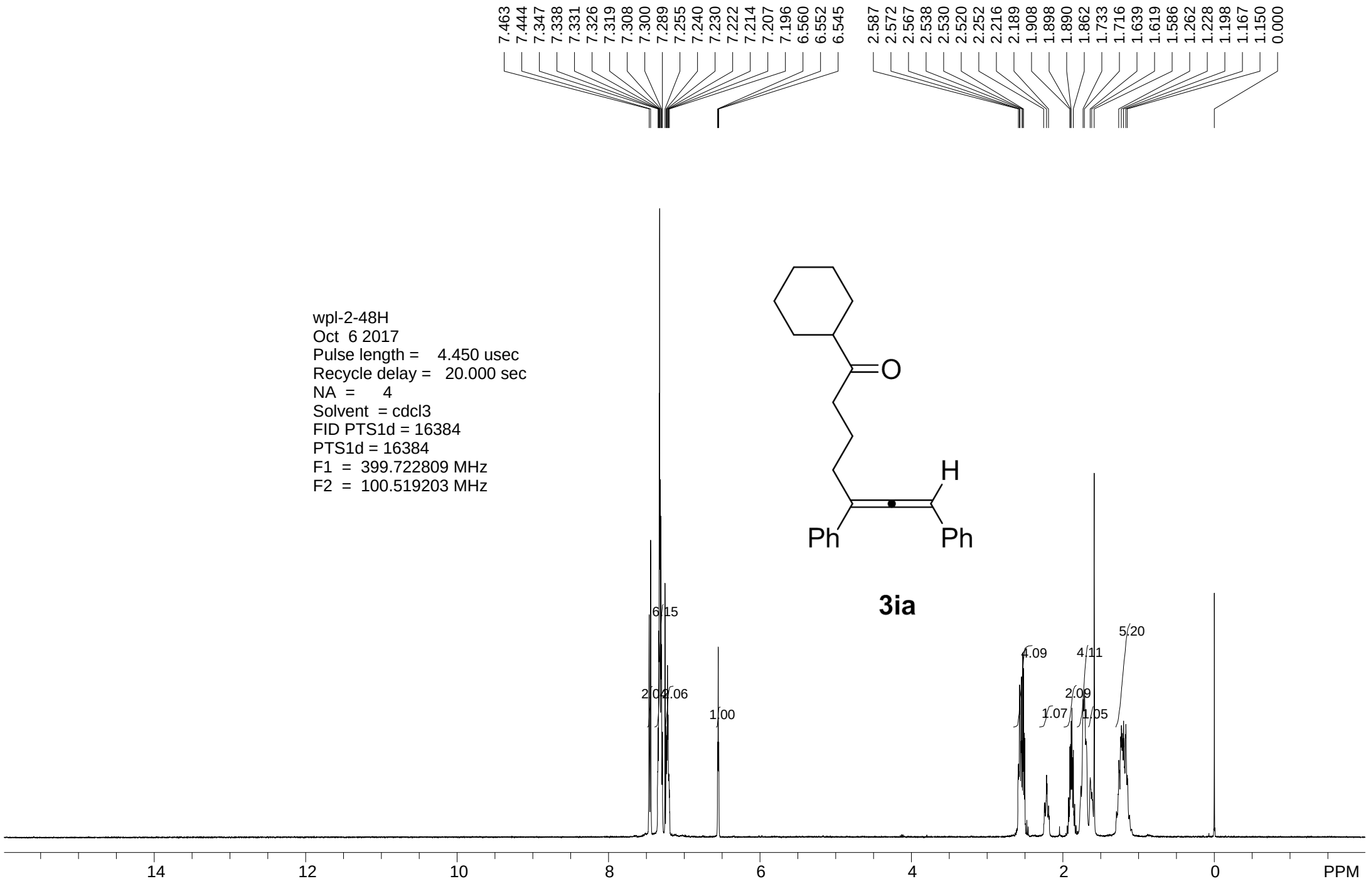

130.080
128.630
128.448
127.006
126.945
126.717
126.064
wpl-2-70C
Oct 27 2017
Pulse length = 4.000 usec
Recycle delay = 1.000 sec
NA = 1000
Solvent = cdcl3
FID PTS1d = 32768
PTS1d = 32768
F1 = 100.527557 MHz
F2 = 399.749146 MHz
OMe
O
H
Ph
Ph
3fa
130
129
128
127
126
125
124
PPM

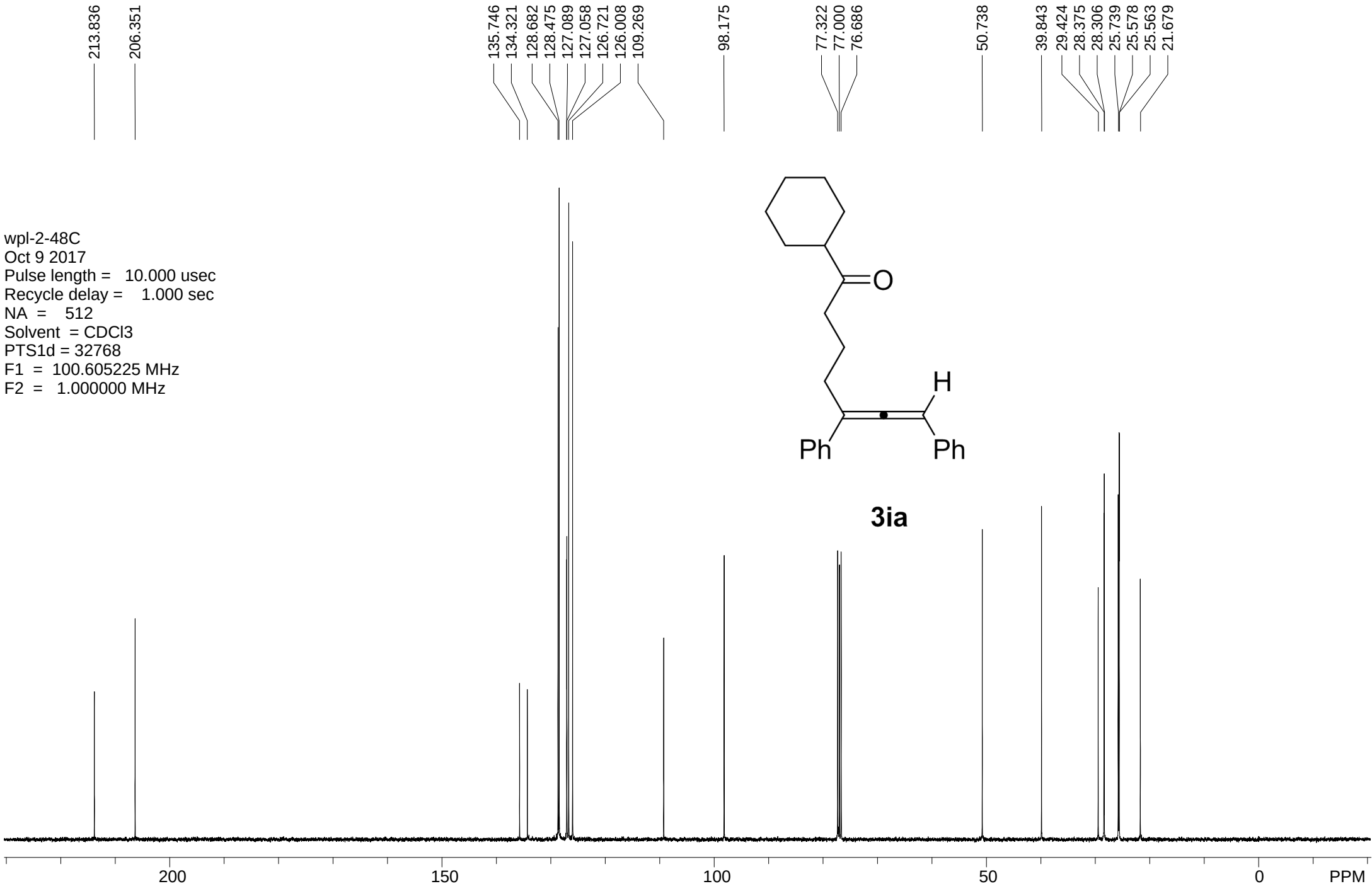

7.478
7.459
7.448
7.443
7.427
7.423
7.373
7.369
7.360
7.356
7.338
7.321
7.303
7.283
7.255
7.242
7.230
7.225
7.219
7.213
7.207
7.198
7.195
6.772
6.752
6.573
6.566
6.558
6.021
3.004
2.986
2.968
2.707
2.700
2.689
2.681
2.671
2.663
2.654
2.646
2.106
2.089
2.071
2.065
2.054
2.047
2.029
1.581
-0.000
wpl-2-82H
Nov 2 2017
Pulse length = 3.400 usec
Recycle delay = 20.000 sec
NA = 4
Solvent = cdcl3
FID PTS1d = 16384
PTS1d = 16384
F1 = 399.749542 MHz
F2 = 100.526031 MHz
12.51
1.00
1.00
2.00
1.98
2.00
2.01
O
O
O
H
Ph
Ph
3ga
12
10
8
6
4
2
0
PPM

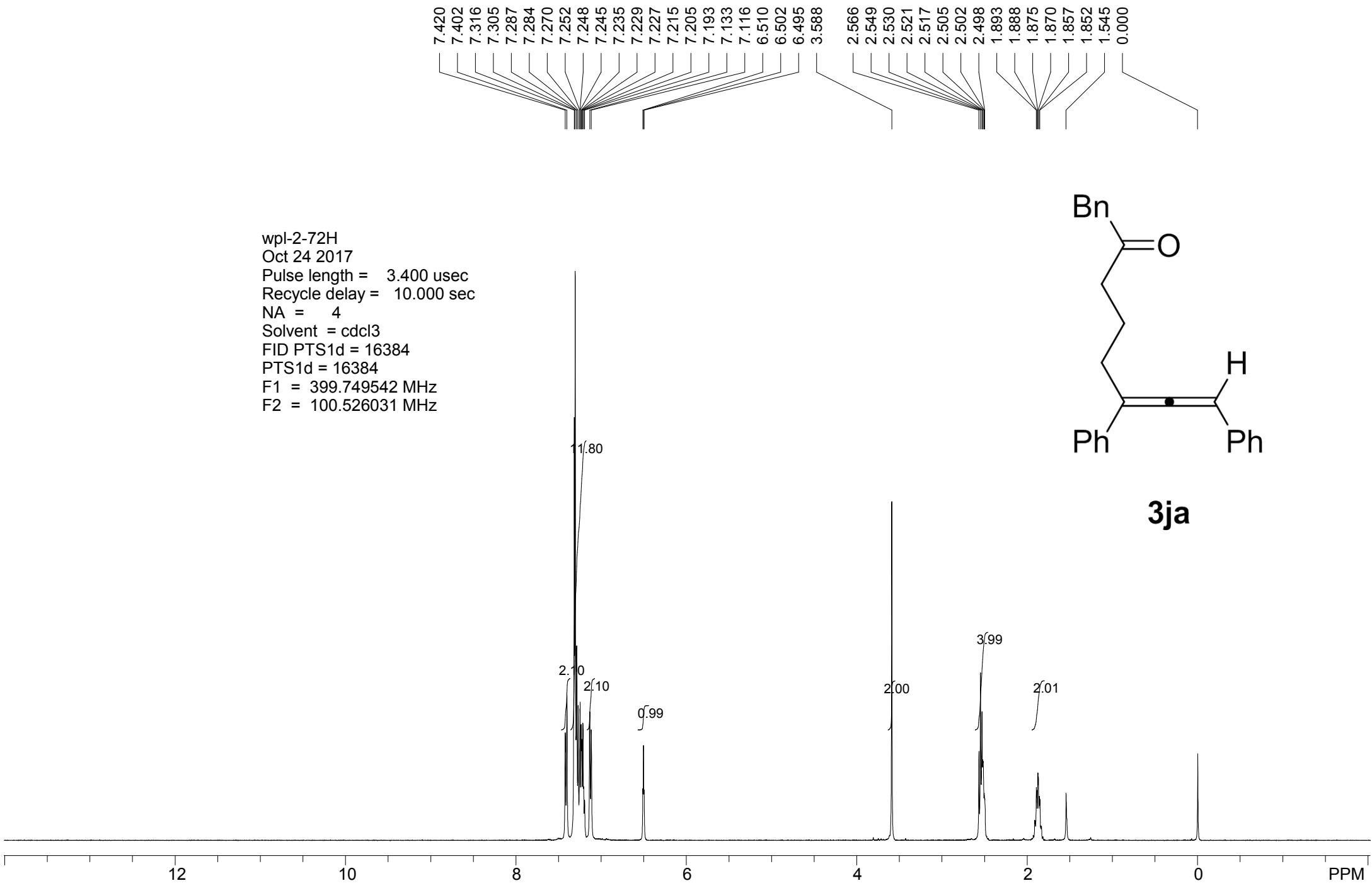

206.402
197.984
151.515
148.062
135.735
134.353
131.818
128.736
128.516
127.142
127.104
126.763
126.057
124.174
109.282
107.779
107.718
101.714
98.238
77.319
77.000
76.681
37.727
29.605
22.432
wpl-2-82C
Nov 2 2017
Pulse length = 4.000 usec
Recycle delay = 1.000 sec
NA = 1000
Solvent = cdcl3
FID PTS1d = 32768
PTS1d = 32768
F1 = 100.527557 MHz
F2 = 399.749146 MHz
O
O
O
H
Ph
Ph
3ga
200
150
100
50
0
PPM

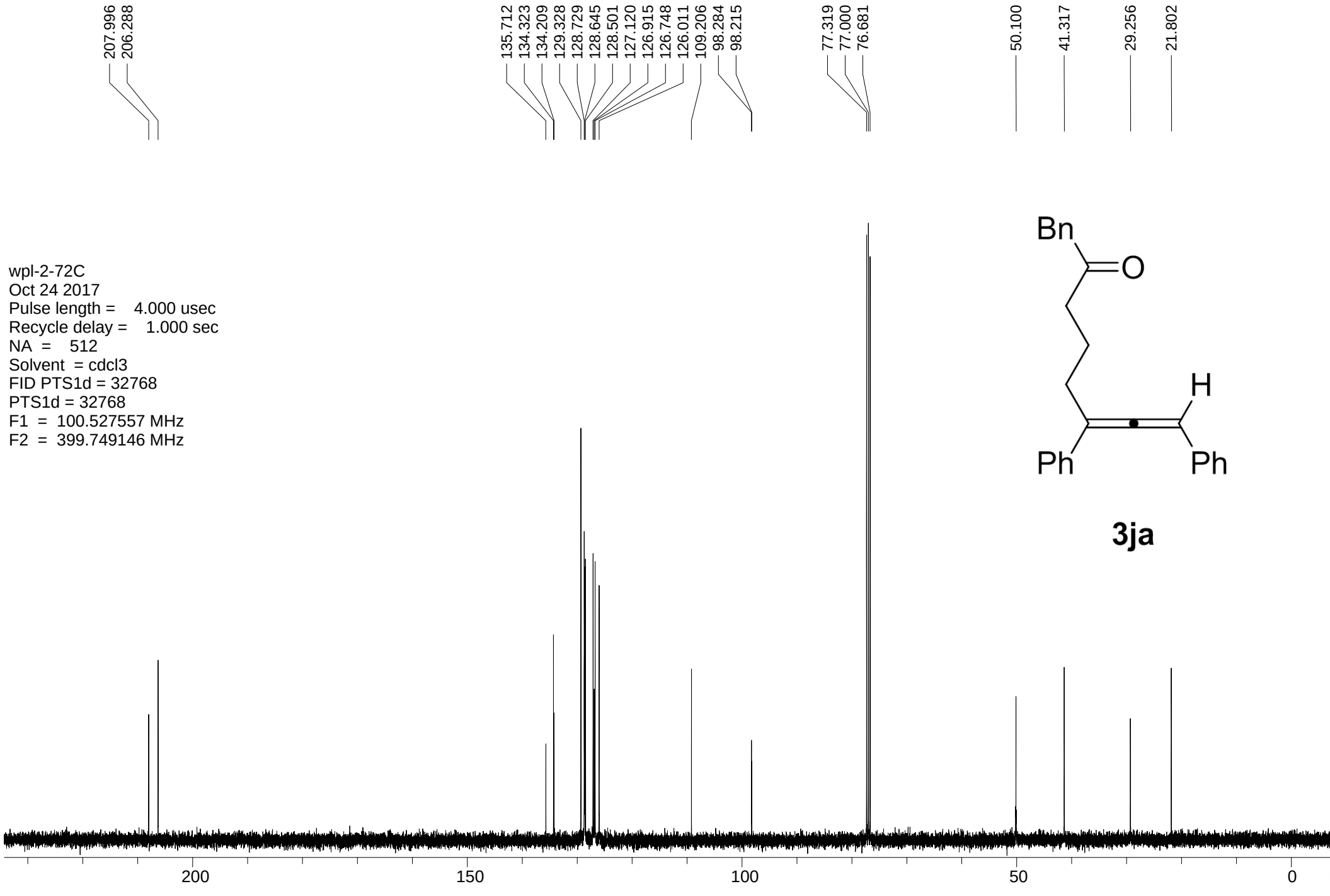

7.460
7.440
7.325
7.308
7.300
7.289
7.253
7.238
7.221
7.213
6.553
2.594
2.576
2.561
2.518
2.500
2.482
2.319
2.300
2.281
1.931
1.912
1.894
1.876
1.589
1.503
1.485
1.466
1.447
1.275
1.256
1.238
1.219
0.881
0.863
0.844
-0.000
wpl-2-39H
Sep 30 2017
NA = 4
Solvent = CDCl3
PTS1d = 65536
F1 = 400.130005 MHz
F2 = 1.000000 MHz
O
H
Ph
Ph
3ha
8.66
2.09
1.00
2.04
2.04
1.96
2.05
2.07
2.12
3.17
12
10
8
6
4
2
0
PPM

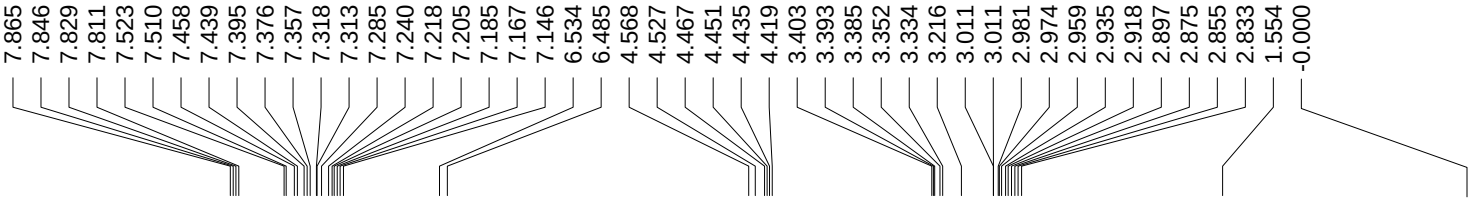

210.988
206.289
135.693
134.274
128.656
128.459
127.077
127.047
126.683
125.977
109.231
98.186
77.326
77.000
76.689
42.446
41.975
29.367
25.829
22.231
21.852
13.752
wpl-2-39C
Oct 2 2017
Pulse length = 4.950 usec
Recycle delay = 1.000 sec
NA = 64
Solvent = cdcl3
FID PTS1d = 32768
PTS1d = 32768
F1 = 100.520737 MHz
F2 = 399.722015 MHz
O
H
Ph
Ph
3ha
200
150
100
50
0
PPM


7.463
7.444
7.347
7.338
7.331
7.326
7.319
7.308
7.300
7.289
7.255
7.240
7.230
7.222
7.214
7.207
7.196
6.560
6.552
6.545
2.587
2.572
2.567
2.538
2.530
2.520
2.252
2.216
2.189
1.908
1.898
1.890
1.862
1.733
1.716
1.639
1.619
1.586
1.262
1.228
1.198
1.167
1.150
0.000
wpl-2-48H
Oct 6 2017
Pulse length = 4.450 usec
Recycle delay = 20.000 sec
NA = 4
Solvent = cdcl3
FID PTS1d = 16384
PTS1d = 16384
F1 = 399.722809 MHz
F2 = 100.519203 MHz
O
H
Ph
Ph
3ia
6.15
2.00
2.06
1.00
4.09
4.11
5.20
2.09
1.07
1.05
14
12
10
8
6
4
2
0
PPM

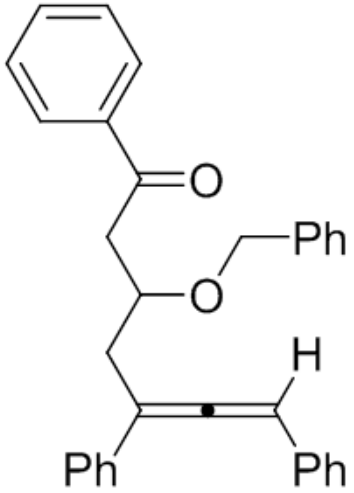

213.836
206.351
135.746
134.321
128.682
128.475
127.089
127.058
126.721
126.008
109.269
98.175
77.322
77.000
76.686
50.738
39.843
29.424
28.375
28.306
25.739
25.578
25.563
21.679
wpl-2-48C
Oct 9 2017
Pulse length = 10.000 usec
Recycle delay = 1.000 sec
NA = 512
Solvent = CDCl3
PTS1d = 32768
F1 = 100.605225 MHz
F2 = 1.000000 MHz
O
H
Ph
Ph
3ia
200
150
100
50
0
PPM


7.420
7.402
7.316
7.305
7.287
7.284
7.270
7.252
7.248
7.245
7.235
7.229
7.227
7.215
7.205
7.193
7.133
7.116
6.510
6.502
6.495
3.588
2.566
2.549
2.530
2.521
2.517
2.505
2.502
2.498
1.893
1.888
1.875
1.870
1.857
1.852
1.545
0.000
wpl-2-72H
Oct 24 2017
Pulse length = 3.400 usec
Recycle delay = 10.000 sec
NA = 4
Solvent = cdcl3
FID PTS1d = 16384
PTS1d = 16384
F1 = 399.749542 MHz
F2 = 100.526031 MHz
Bn
O
H
Ph
Ph
3ja
11.80
2.10
2.10
0.99
2.00
3.99
2.01
12
10
8
6
4
2
0
PPM

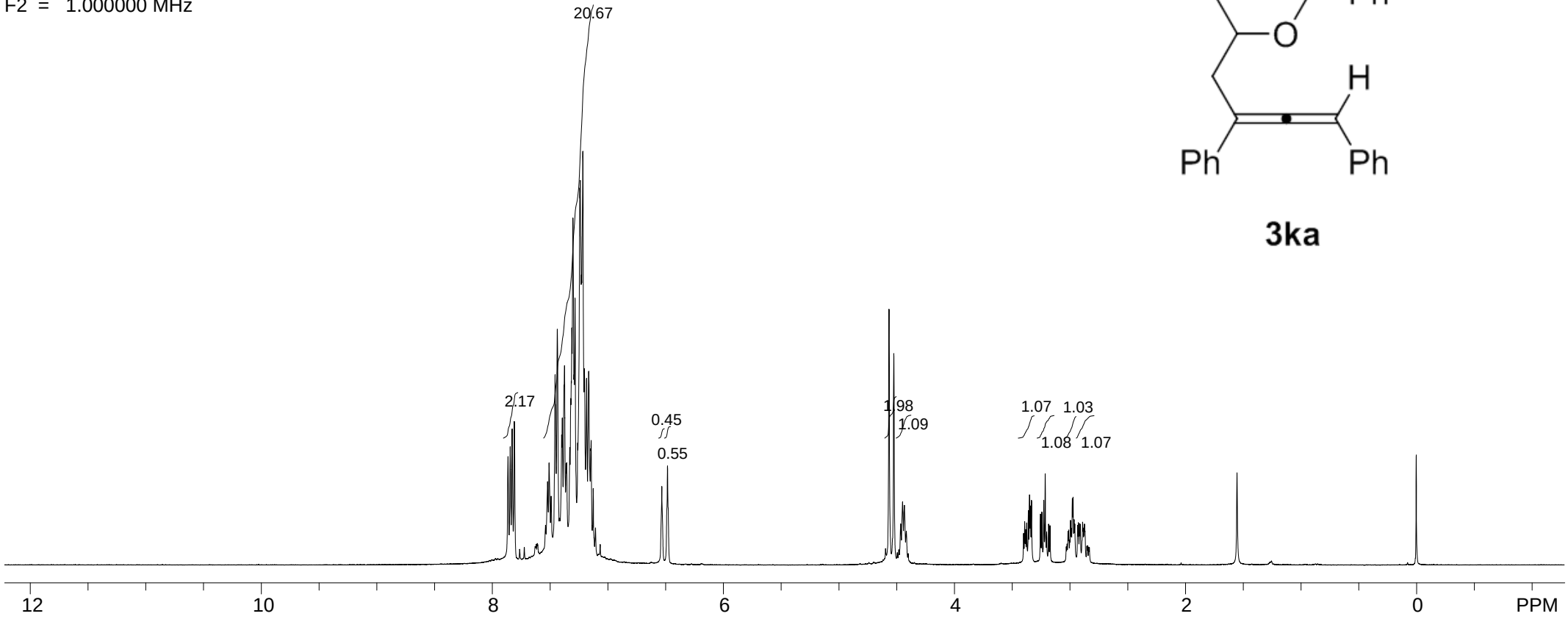

207.996
206.288
135.712
134.323
134.209
129.328
128.729
128.645
128.501
127.120
126.915
126.748
126.011
109.206
98.284
98.215
77.319
77.000
76.681
50.100
41.317
29.256
21.802
wpl-2-72C
Oct 24 2017
Pulse length = 4.000 usec
Recycle delay = 1.000 sec
NA = 512
Solvent = cdcl3
FID PTS1d = 32768
PTS1d = 32768
F1 = 100.527557 MHz
F2 = 399.749146 MHz
Bn
O
H
Ph
Ph
3ja
200
150
100
50
0
PPM



wpl-4-194H
Pulse length = 10.000 usec
Recycle delay = 20.000 sec
NA = 4
Solvent = CDCl3
PTS1d = 65536
F1 = 400.130005 MHz
F2 = 1.000000 MHz



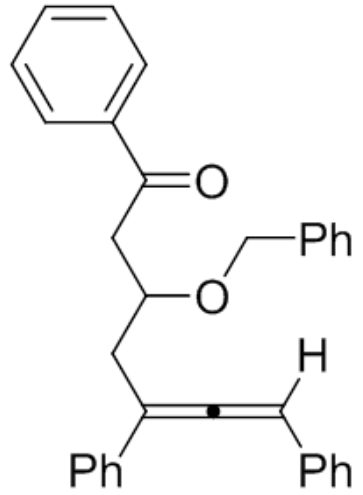
3ka



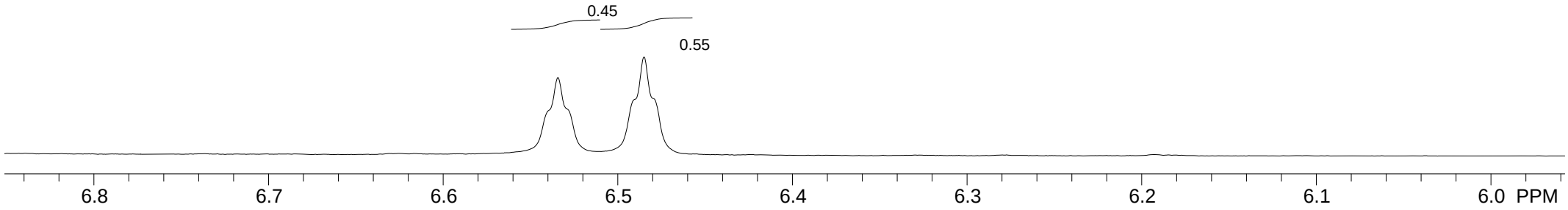
6.534

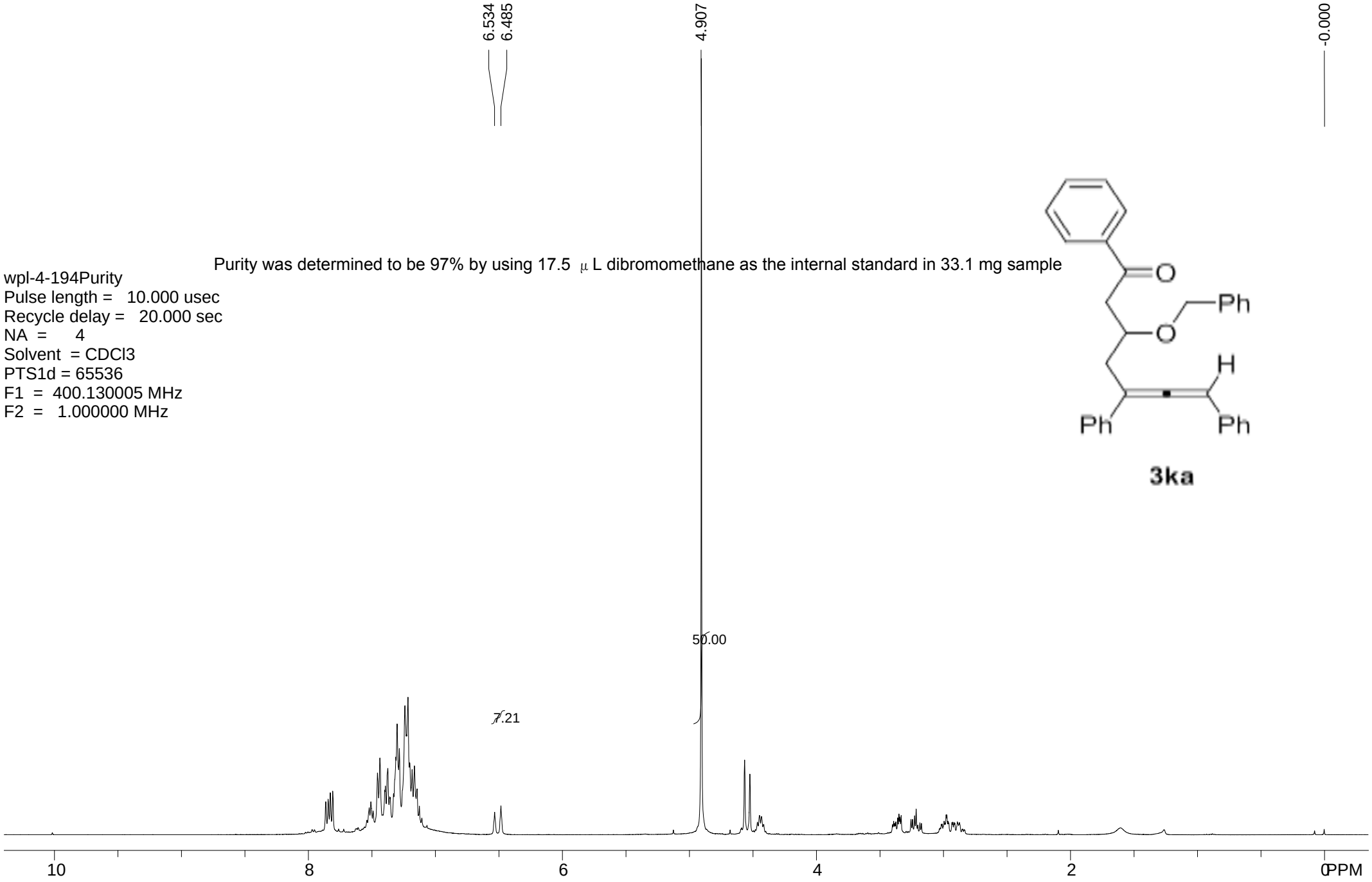
6.485

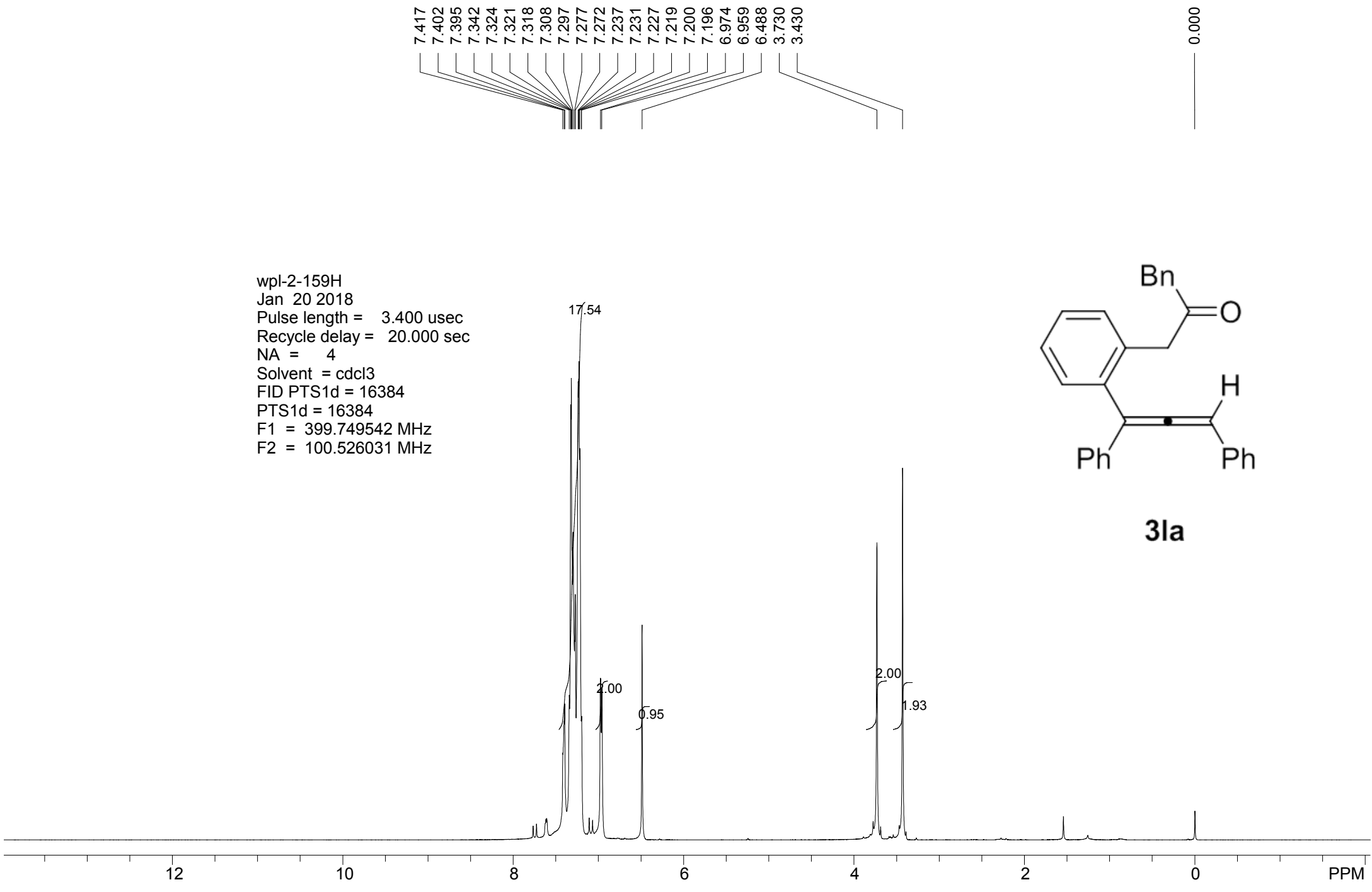
wpl-4-194H
Pulse length = 10.000 usec
Recycle delay = 20.000 sec
NA = 4
Solvent = CDCl3
PTS1d = 65536
F1 = 400.130005 MHz
F2 = 1.000000 MHz

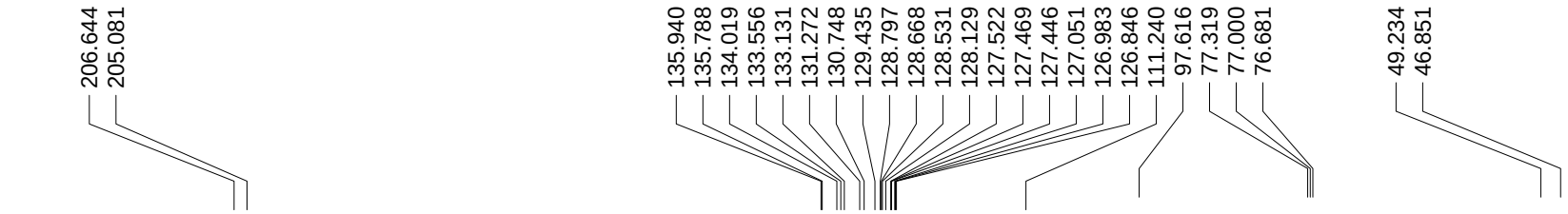


3ka

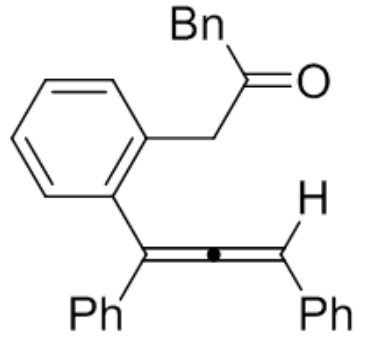



6.534
6.485
4.907
-0.000
Purity was determined to be 97% by using 17.5 μL dibromomethane as the internal standard in 33.1 mg sample
wpl-4-194Purity
Pulse length = 10.000 usec
Recycle delay = 20.000 sec
NA = 4
Solvent = CDCl3
PTS1d = 65536
F1 = 400.130005 MHz
F2 = 1.000000 MHz
O
Ph
O
H
Ph
Ph
3ka
50.00
7.21
10
8
6
4
2
0
PPM

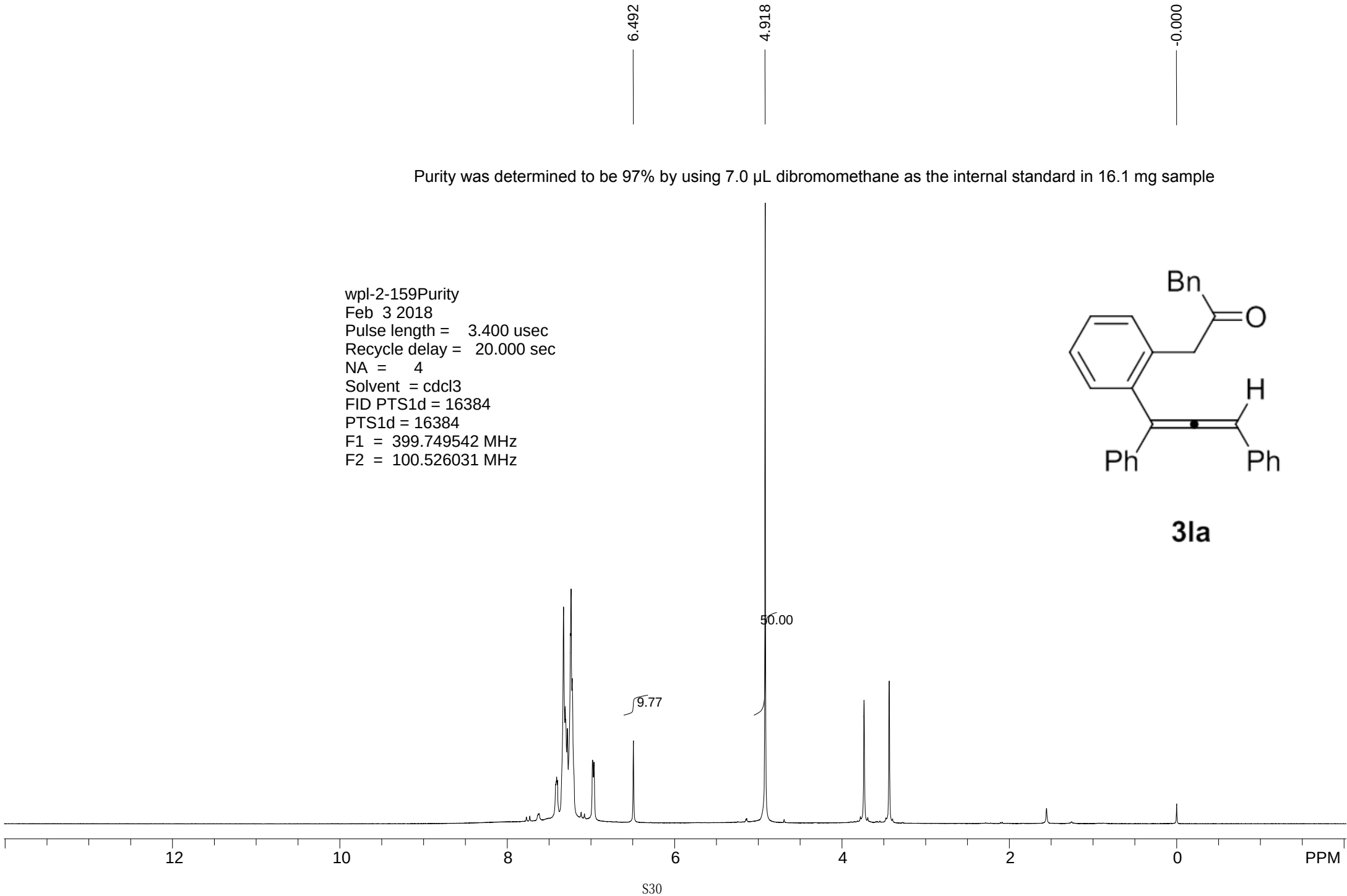

7.417
7.402
7.395
7.342
7.324
7.321
7.318
7.308
7.297
7.277
7.272
7.237
7.231
7.227
7.219
7.200
7.196
6.974
6.959
6.488
3.730
3.430
0.000
wpl-2-159H
Jan 20 2018
Pulse length = 3.400 usec
Recycle delay = 20.000 sec
NA = 4
Solvent = cdcl3
FID PTS1d = 16384
PTS1d = 16384
F1 = 399.749542 MHz
F2 = 100.526031 MHz
17.54
2.00
0.95
2.00
1.93
Bn
O
H
Ph
Ph
3la
12
10
8
6
4
2
0
PPM

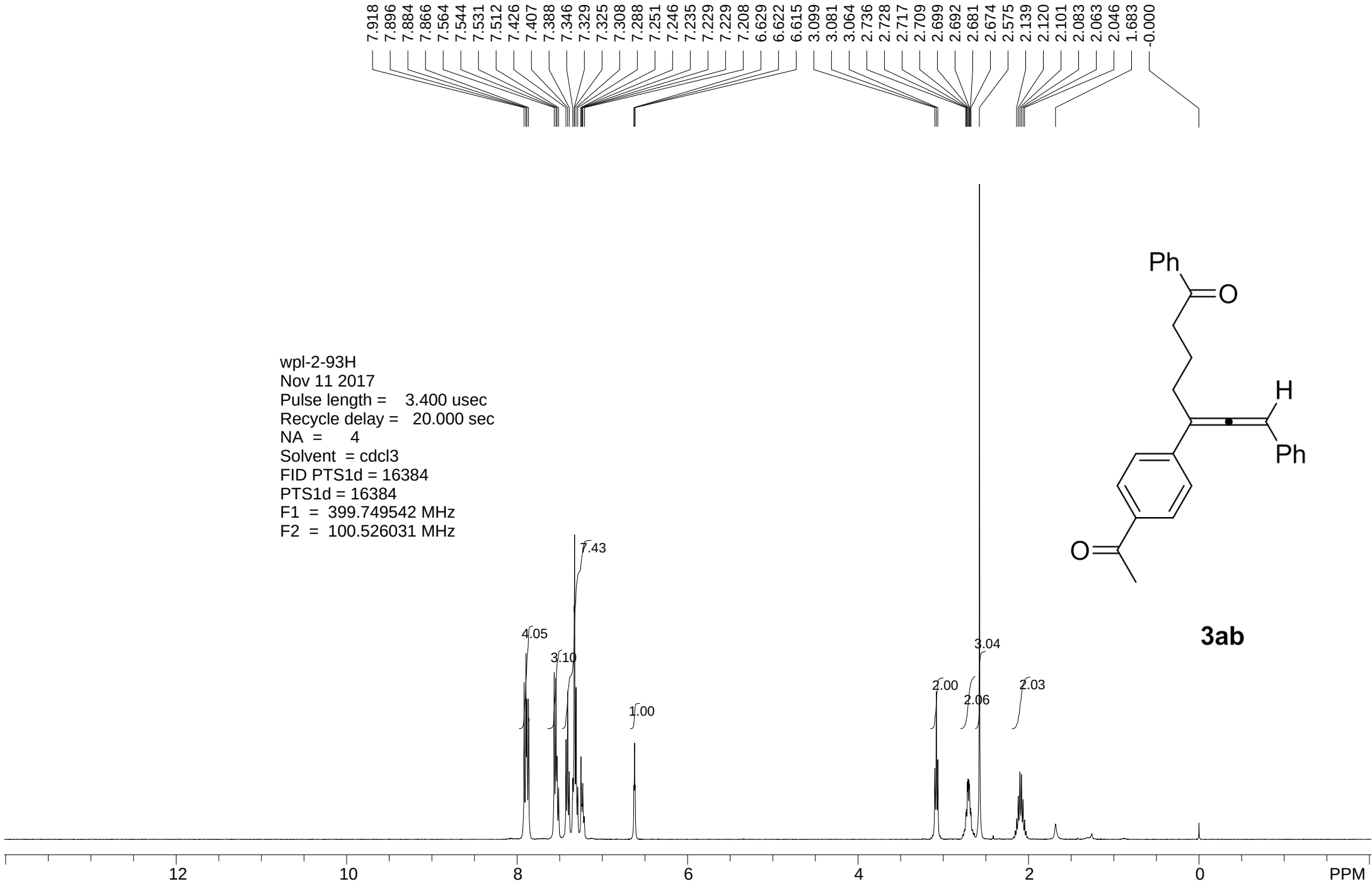


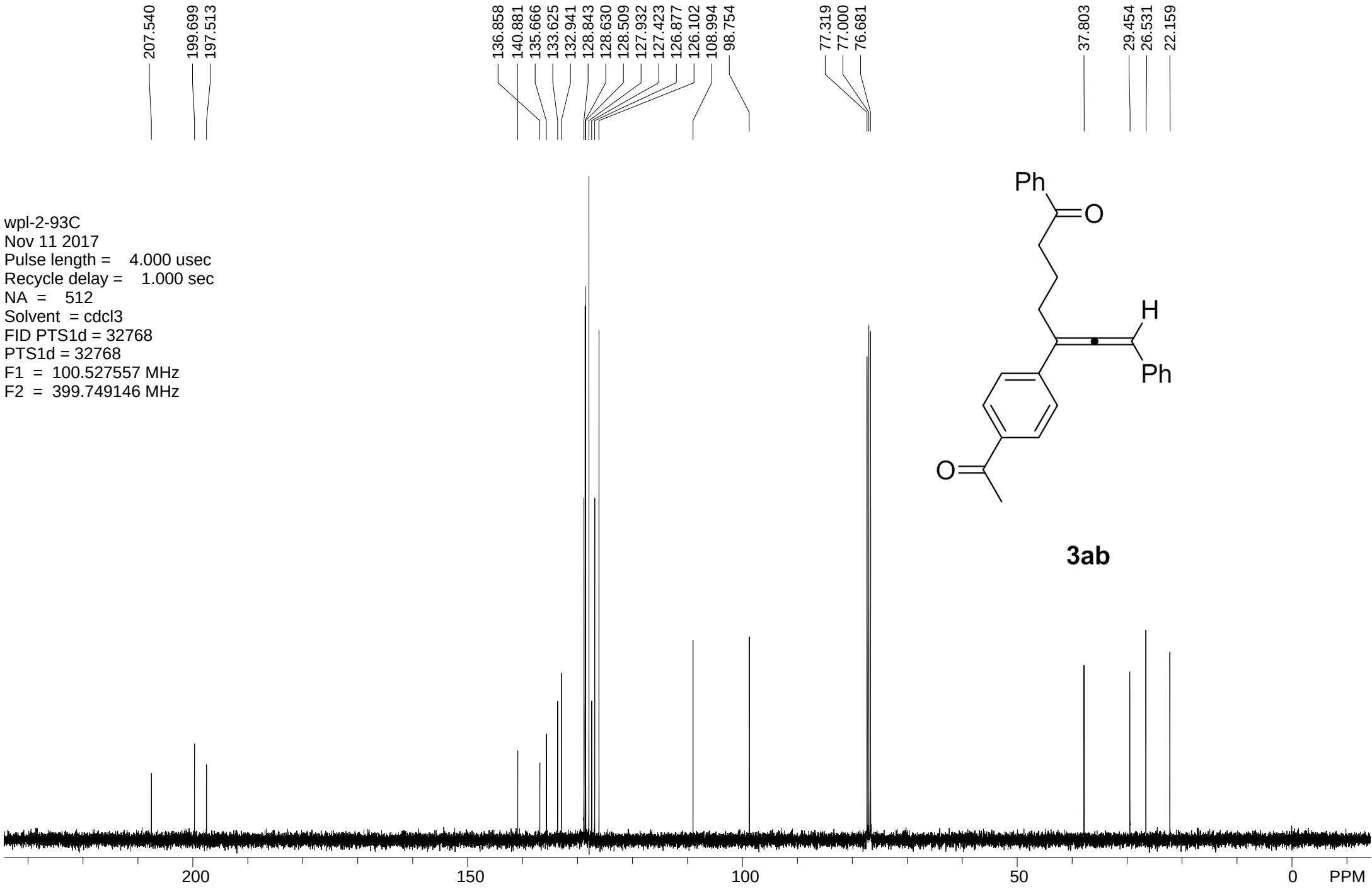
wpl-2-159C
Jan 20 2018
Pulse length = 4.000 usec
Recycle delay = 1.000 sec
NA = 512
Solvent = cdcl3
FID PTS1d = 32768
PTS1d = 32768
F1 = 100.527557 MHz
F2 = 399.749146 MHz

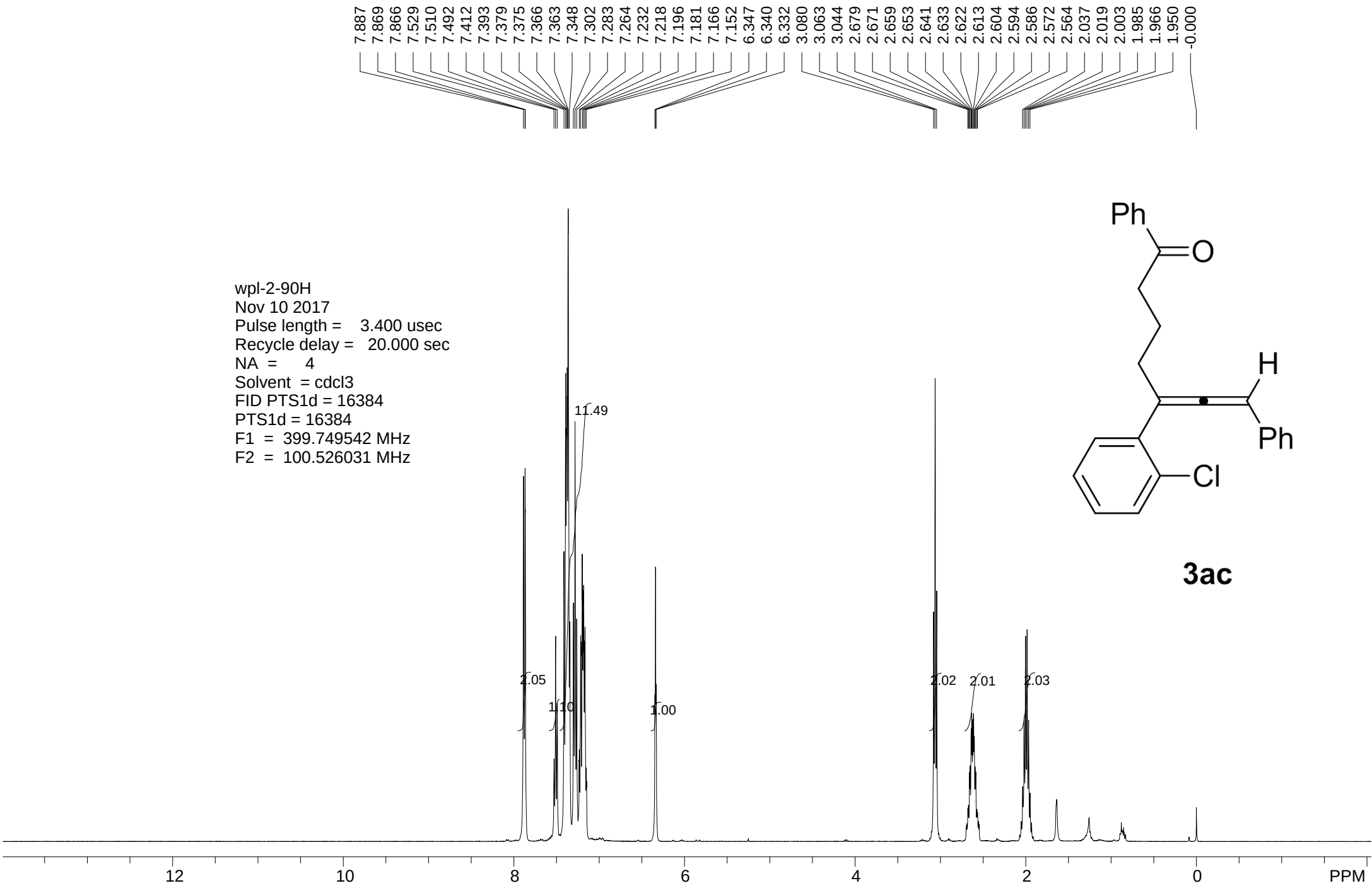


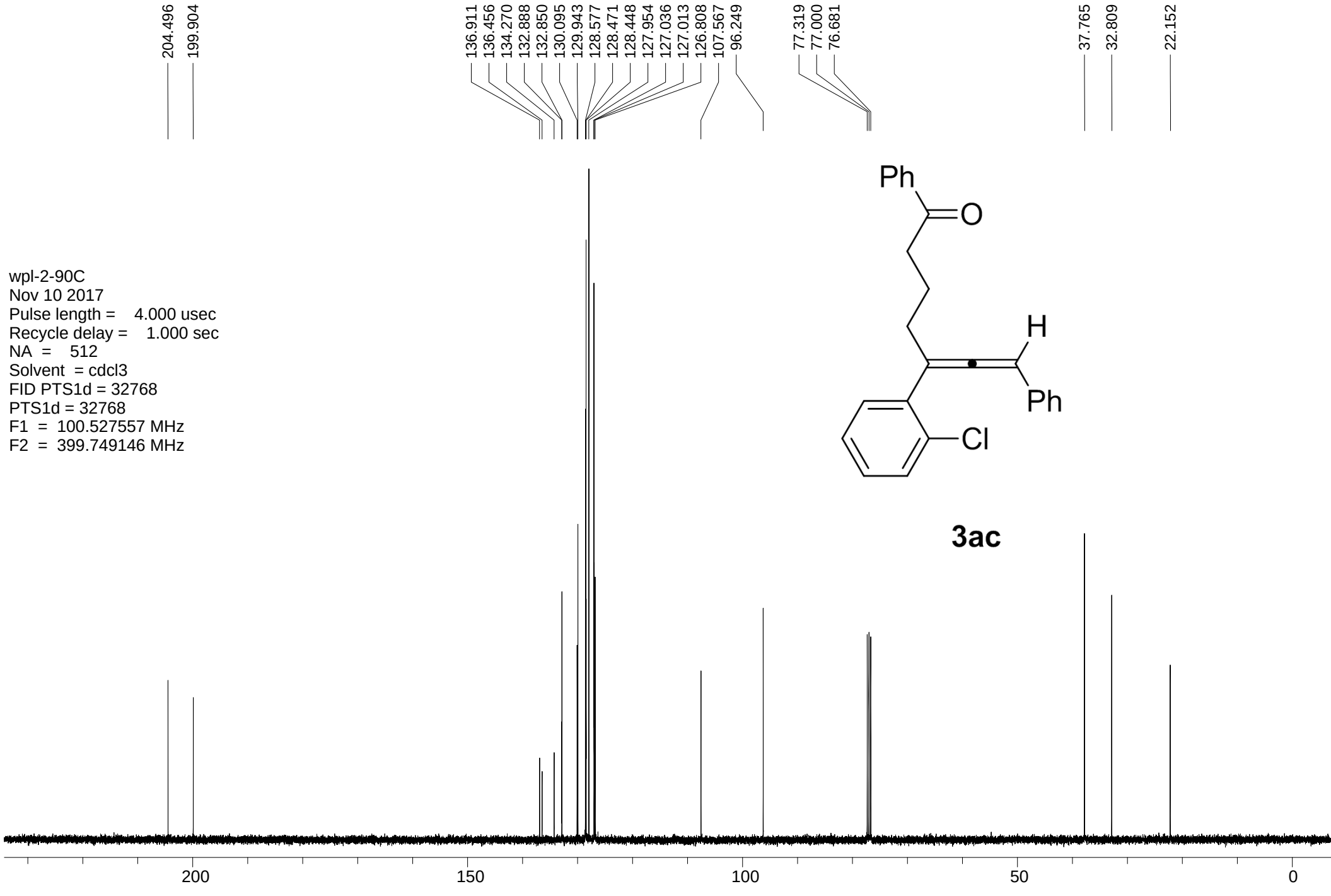
3la

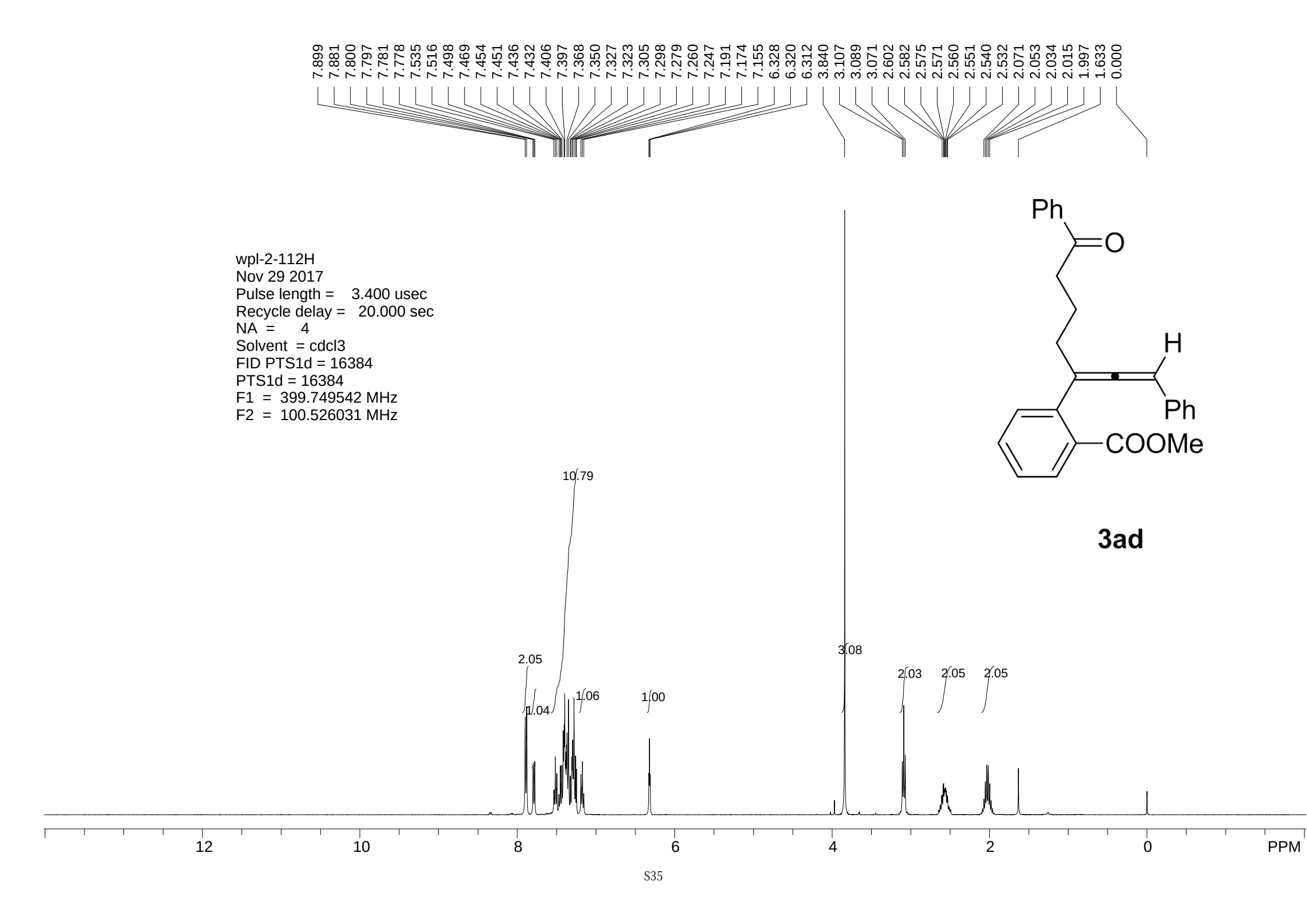

6.492
4.918
-0.000
Purity was determined to be 97% by using 7.0 µL dibromomethane as the internal standard in 16.1 mg sample
wpl-2-159Purity
Feb 3 2018
Pulse length = 3.400 usec
Recycle delay = 20.000 sec
NA = 4
Solvent = cdcl3
FID PTS1d = 16384
PTS1d = 16384
F1 = 399.749542 MHz
F2 = 100.526031 MHz
Bn
O
H
Ph
Ph
3la
50.00
9.77
12
10
8
6
4
2
0
PPM

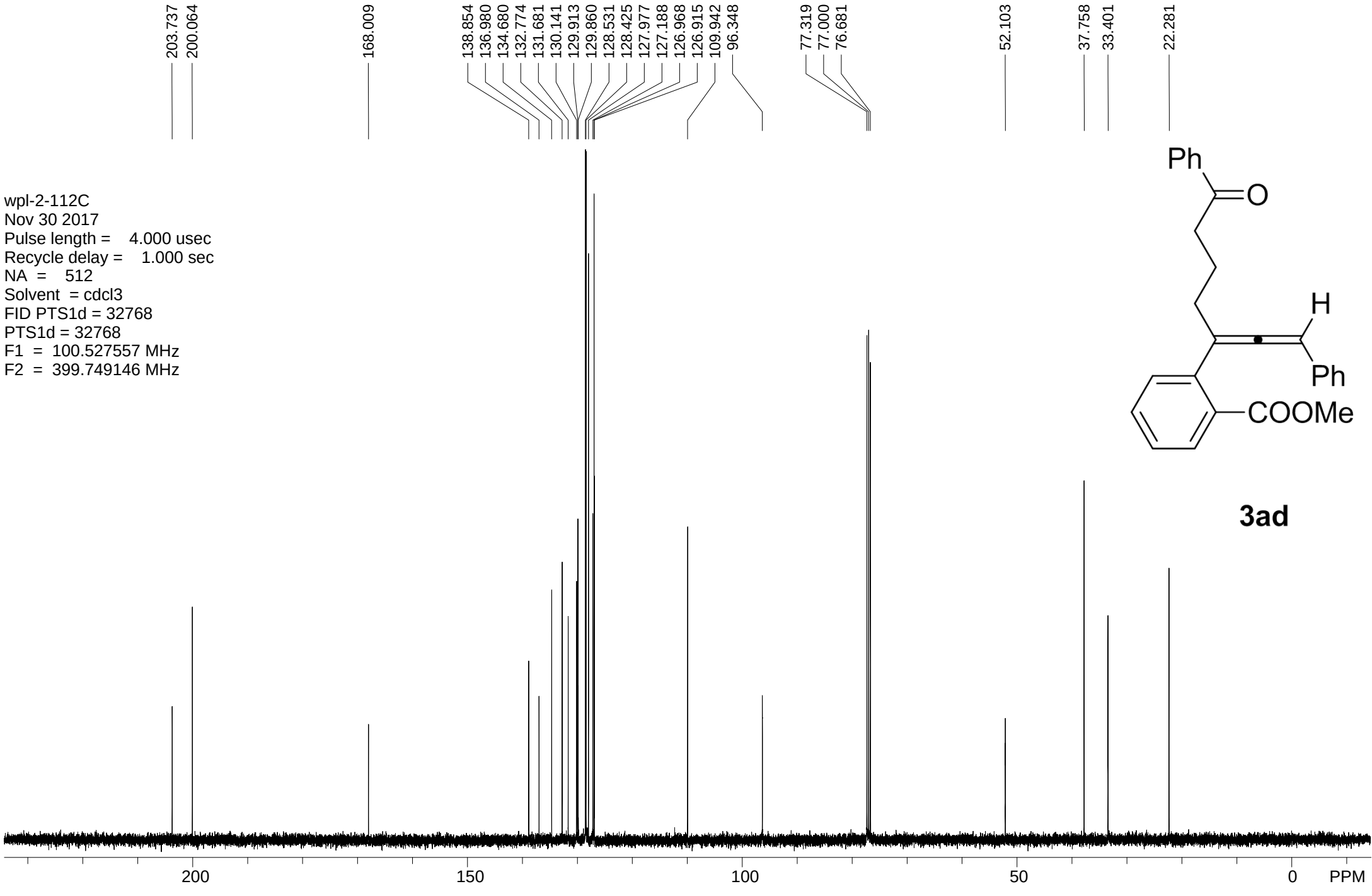

7.918
7.896
7.884
7.866
7.564
7.544
7.531
7.512
7.426
7.407
7.388
7.346
7.329
7.325
7.308
7.288
7.251
7.246
7.235
7.229
7.229
7.208
6.629
6.622
6.615
3.099
3.081
3.064
2.736
2.728
2.717
2.709
2.699
2.692
2.681
2.674
2.575
2.139
2.120
2.101
2.083
2.063
2.046
1.683
-0.000
wpl-2-93H
Nov 11 2017
Pulse length = 3.400 usec
Recycle delay = 20.000 sec
NA = 4
Solvent = cdcl3
FID PTS1d = 16384
PTS1d = 16384
F1 = 399.749542 MHz
F2 = 100.526031 MHz
Ph
O
H
Ph
O
3ab
7.43
4.05
3.10
1.00
2.00
2.06
3.04
2.03
12
10
8
6
4
2
0
PPM

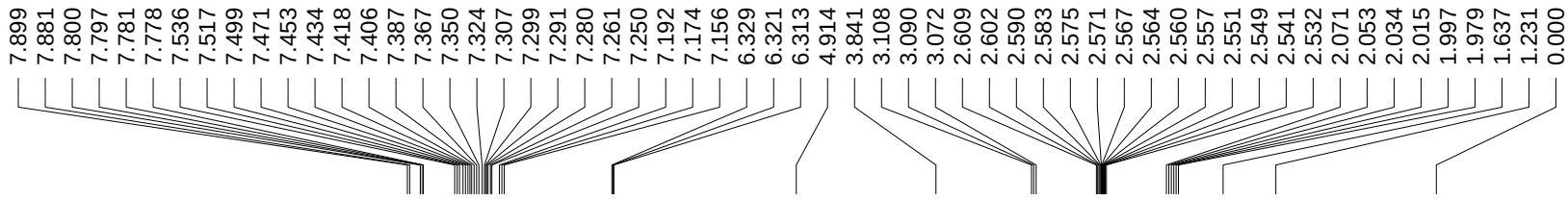

207.540
199.699
197.513
136.858
140.881
135.666
133.625
132.941
128.843
128.630
128.509
127.932
127.423
126.877
126.102
108.994
98.754
77.319
77.000
76.681
37.803
29.454
26.531
22.159
wpl-2-93C
Nov 11 2017
Pulse length = 4.000 usec
Recycle delay = 1.000 sec
NA = 512
Solvent = cdcl3
FID PTS1d = 32768
PTS1d = 32768
F1 = 100.527557 MHz
F2 = 399.749146 MHz
Ph
O
H
Ph
O
3ab
200
150
100
50
0
PPM


7.887
7.869
7.866
7.529
7.510
7.492
7.412
7.393
7.379
7.375
7.366
7.363
7.348
7.302
7.283
7.264
7.232
7.218
7.196
7.181
7.166
7.152
6.347
6.340
6.332
3.080
3.063
3.044
2.679
2.671
2.659
2.653
2.641
2.633
2.622
2.613
2.604
2.594
2.586
2.572
2.564
2.037
2.019
2.003
1.985
1.966
1.950
-0.000
wpl-2-90H
Nov 10 2017
Pulse length = 3.400 usec
Recycle delay = 20.000 sec
NA = 4
Solvent = cdcl3
FID PTS1d = 16384
PTS1d = 16384
F1 = 399.749542 MHz
F2 = 100.526031 MHz
11.49
2.05
1.10
1.00
2.02
2.01
2.03
Ph
O
H
Ph
Cl
3ac
12
10
8
6
4
2
0
PPM


204.496
199.904
136.911
136.456
134.270
132.888
132.850
130.095
129.943
128.577
128.471
128.448
127.954
127.036
127.013
126.808
107.567
96.249
77.319
77.000
76.681
37.765
32.809
22.152
wpl-2-90C
Nov 10 2017
Pulse length = 4.000 usec
Recycle delay = 1.000 sec
NA = 512
Solvent = cdcl3
FID PTS1d = 32768
PTS1d = 32768
F1 = 100.527557 MHz
F2 = 399.749146 MHz
Ph
O
H
Ph
Cl
3ac
200
150
100
50
0
PPM

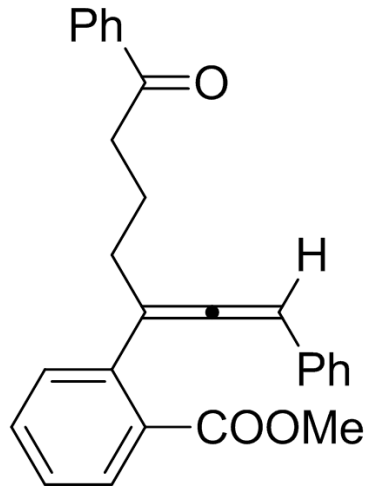

7.899
7.881
7.800
7.797
7.781
7.778
7.535
7.516
7.498
7.469
7.454
7.451
7.436
7.432
7.406
7.397
7.368
7.350
7.327
7.323
7.305
7.298
7.279
7.260
7.247
7.191
7.174
7.155
6.328
6.320
6.312
3.840
3.107
3.089
3.071
2.602
2.582
2.575
2.571
2.560
2.551
2.540
2.532
2.071
2.053
2.034
2.015
1.997
1.633
0.000
wpl-2-112H
Nov 29 2017
Pulse length = 3.400 usec
Recycle delay = 20.000 sec
NA = 4
Solvent = cdcl3
FID PTS1d = 16384
PTS1d = 16384
F1 = 399.749542 MHz
F2 = 100.526031 MHz
Ph
O
H
Ph
COOMe
3ad
10.79
2.05
1.04
1.06
1.00
3.08
2.03
2.05
2.05
12
10
8
6
4
2
0
PPM


203.737
200.064
168.009
138.854
136.980
134.680
132.774
131.681
130.141
129.913
129.860
128.531
128.425
127.977
127.188
126.968
126.915
109.942
96.348
77.319
77.000
76.681
52.103
37.758
33.401
22.281
wpl-2-112C
Nov 30 2017
Pulse length = 4.000 usec
Recycle delay = 1.000 sec
NA = 512
Solvent = cdcl3
FID PTS1d = 32768
PTS1d = 32768
F1 = 100.527557 MHz
F2 = 399.749146 MHz
Ph
O
H
Ph
COOMe
3ad
200
150
100
50
0
PPM

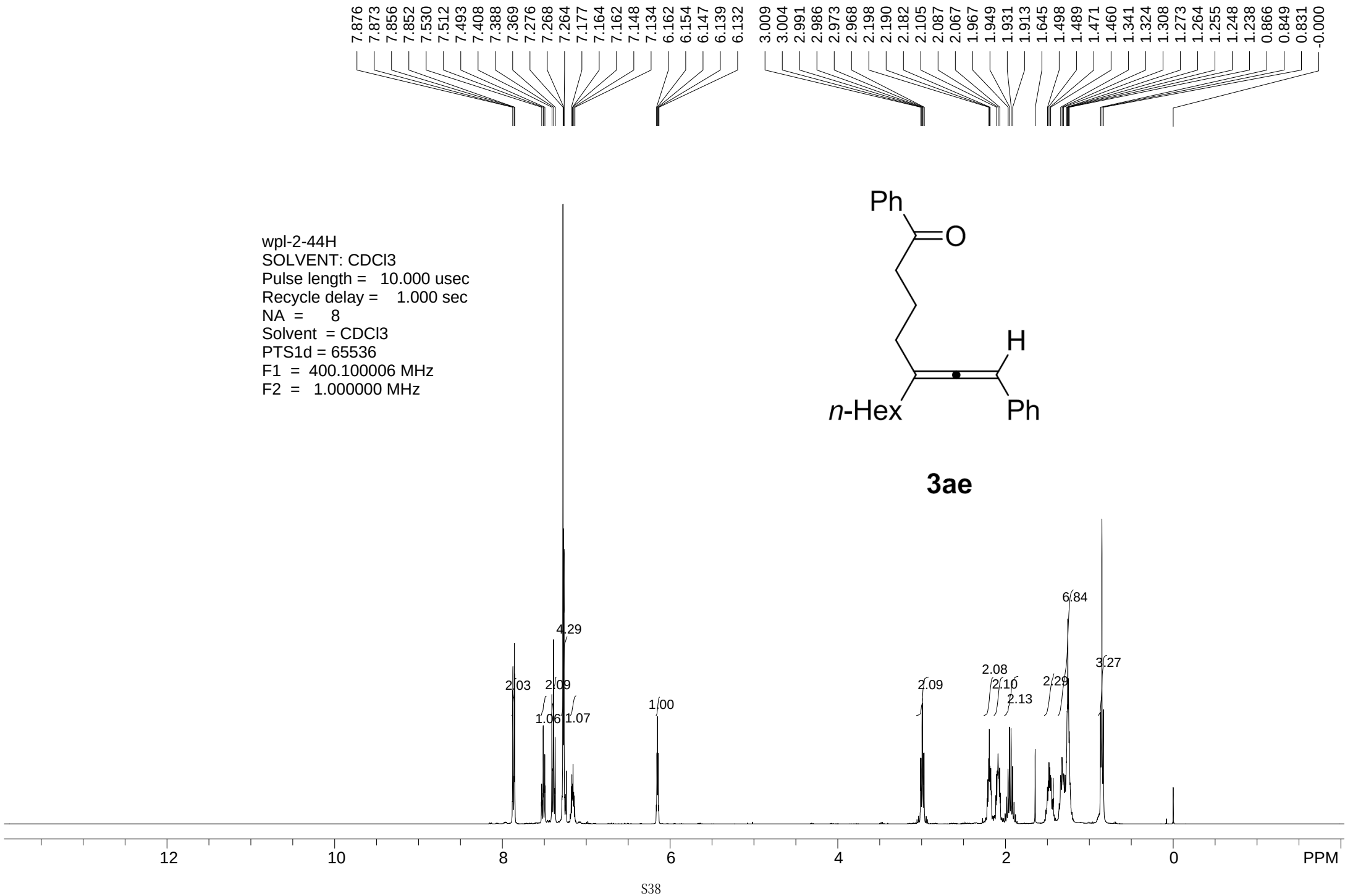


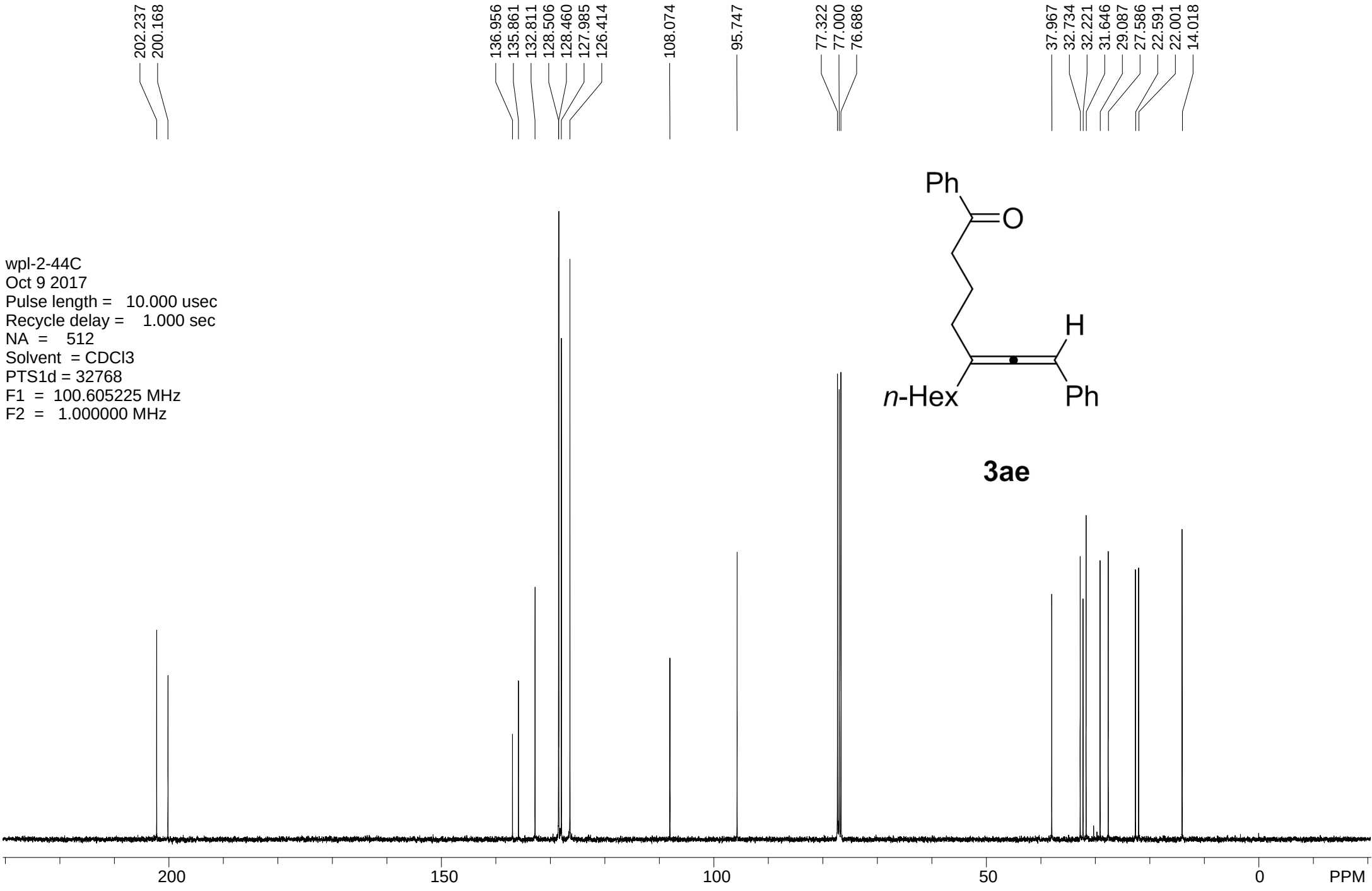
Purity was determined to be 97% by using 7.5 μL dibromomethane as the internal standard in 24.8 mg sample

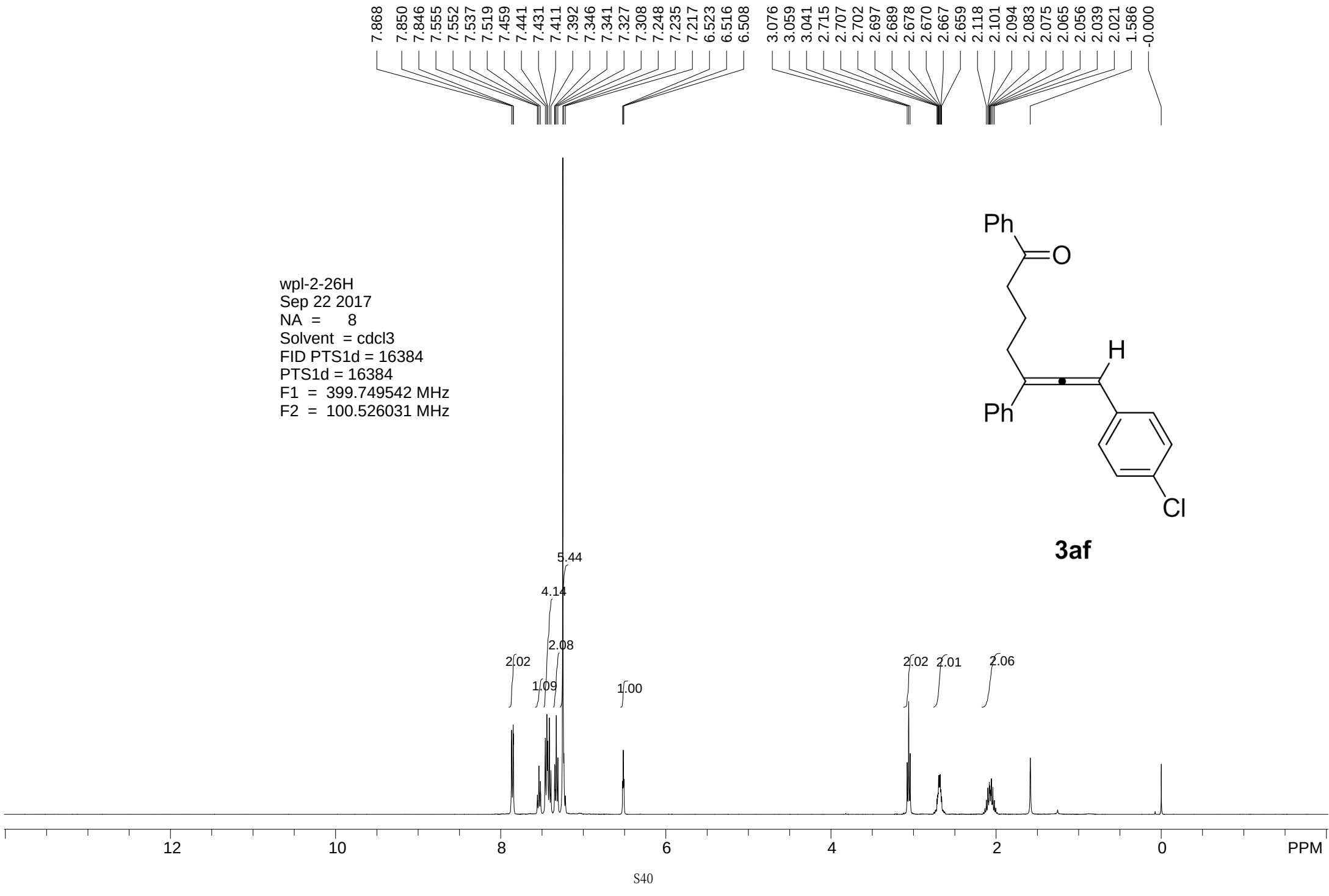
wpl-2-112P
Nov 30 2017
Pulse length = 3.400 usec
Recycle delay = 20.000 sec
NA = 4
Solvent = cdcl3
FID PTS1d = 16384
PTS1d = 16384
F1 = 399.749542 MHz
F2 = 100.526031 MHz

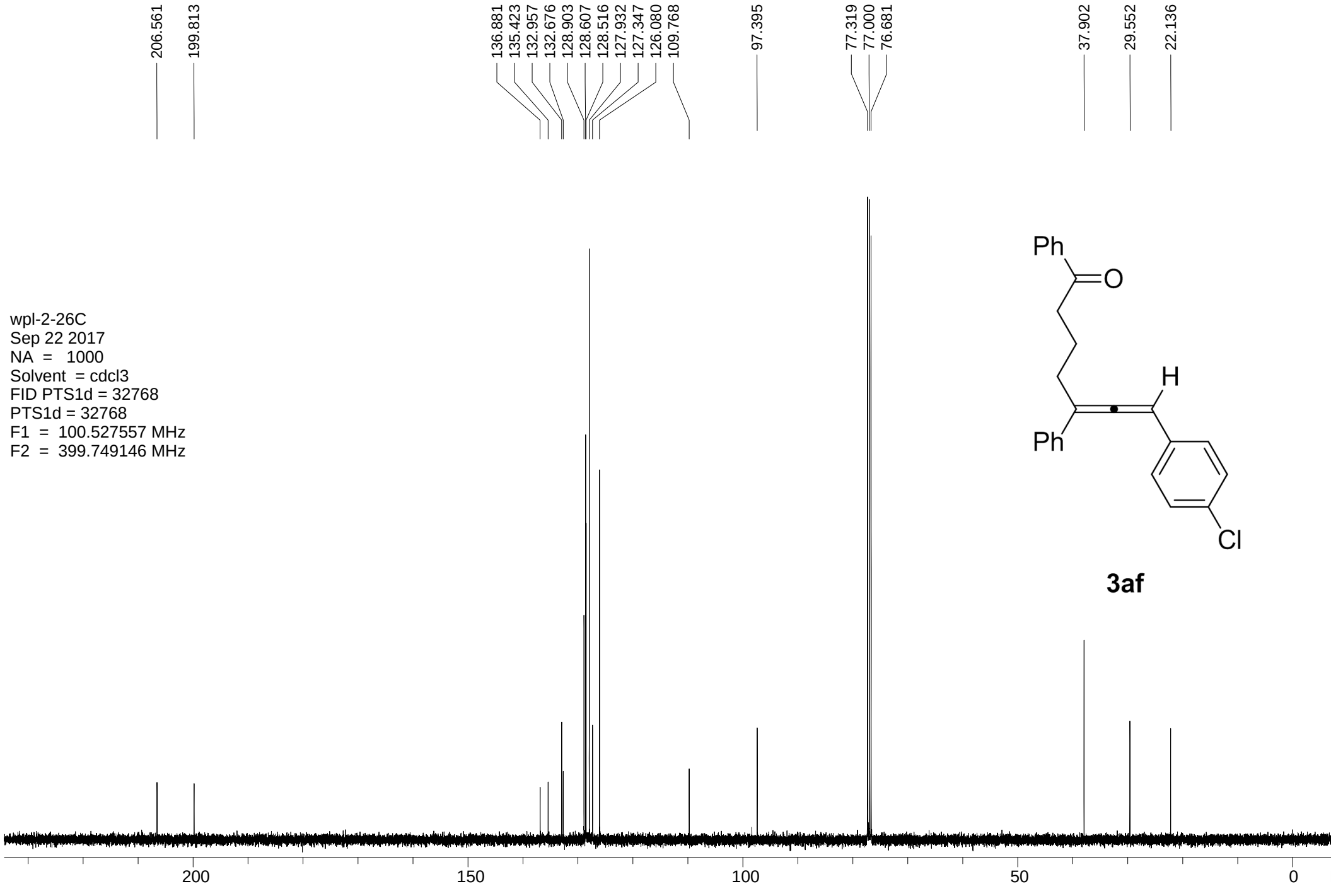


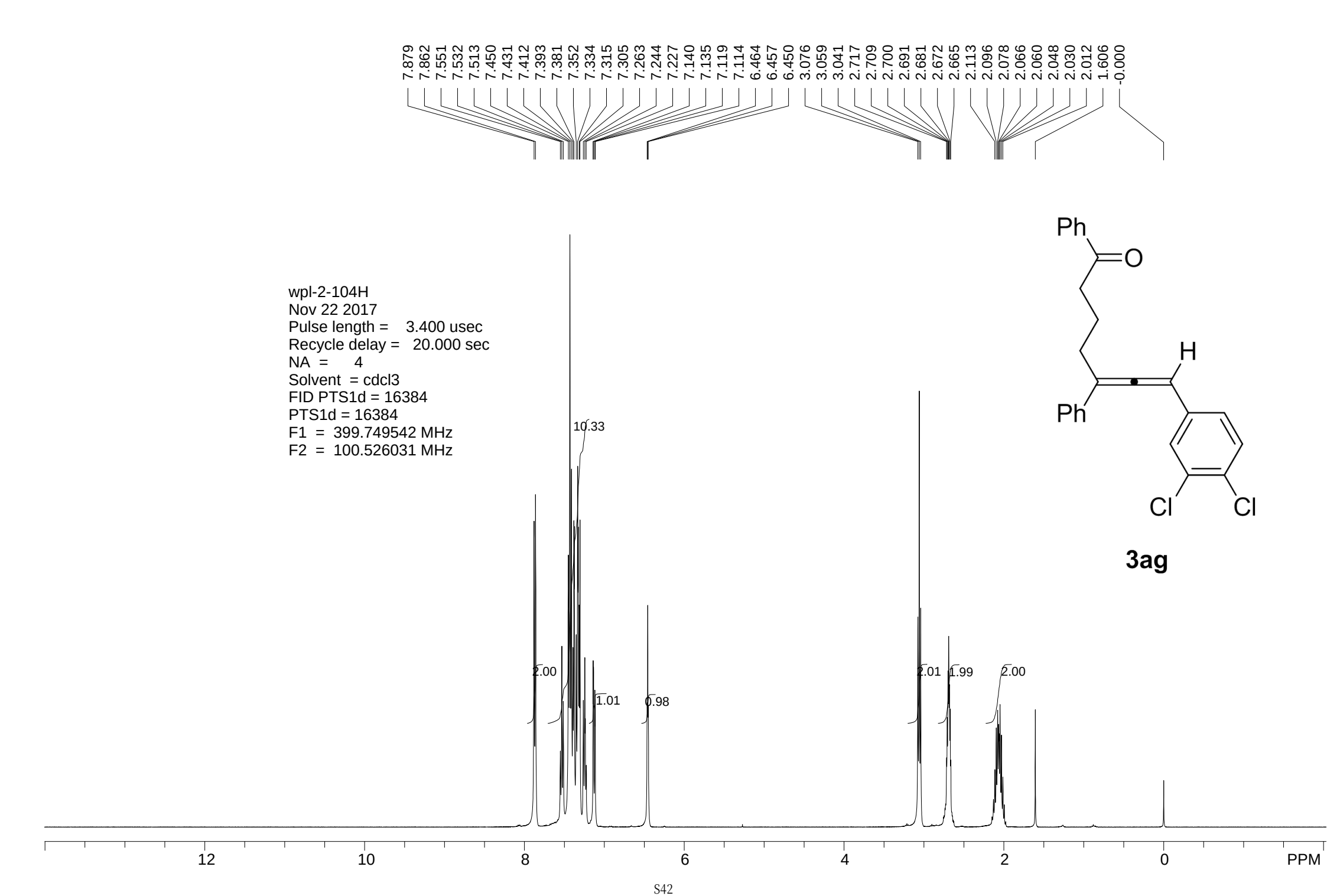
3ad

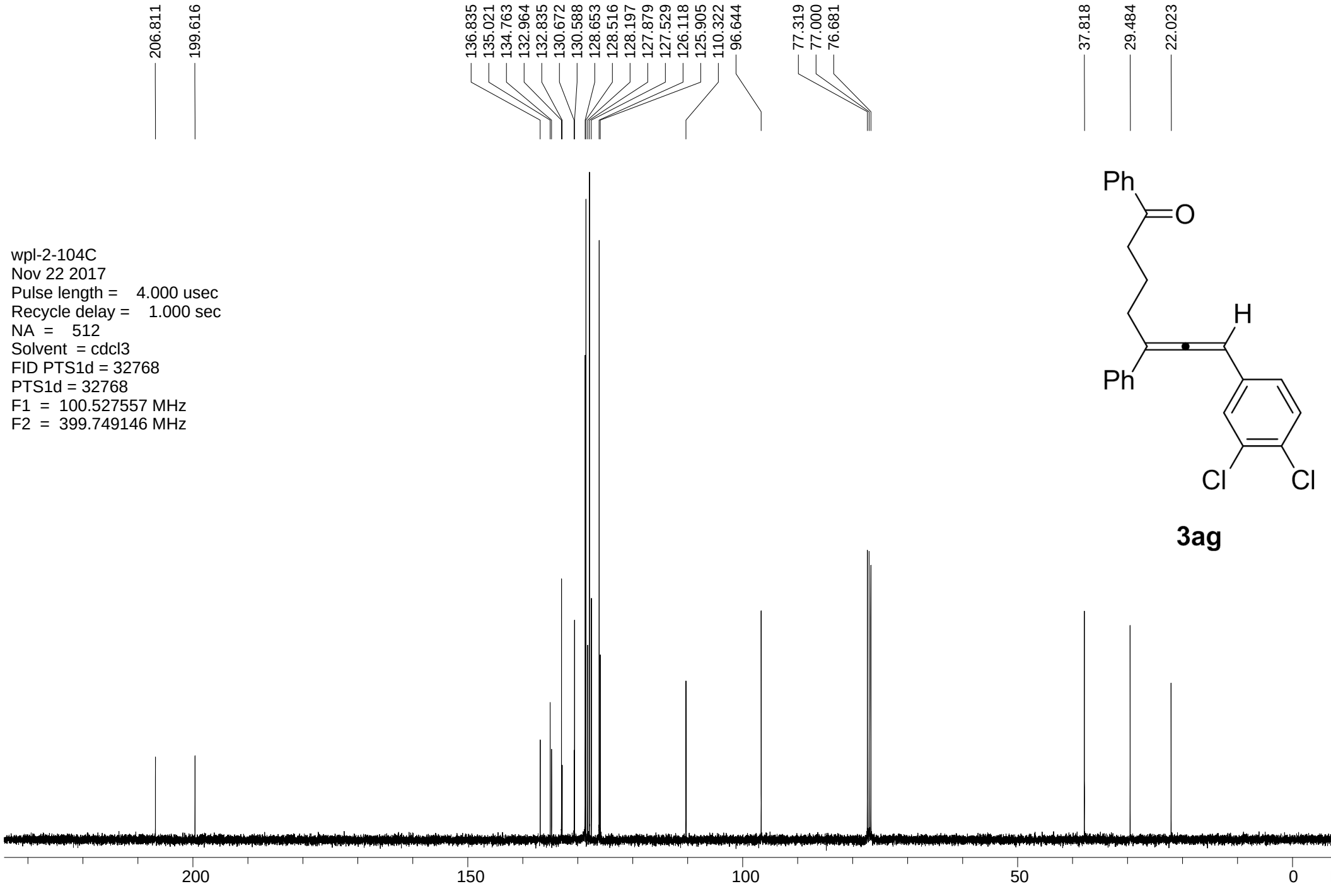

7.876
7.873
7.856
7.852
7.530
7.512
7.493
7.408
7.388
7.369
7.276
7.268
7.264
7.177
7.164
7.162
7.148
7.134
6.162
6.154
6.147
6.139
6.132
3.009
3.004
2.991
2.986
2.973
2.968
2.198
2.190
2.182
2.105
2.087
2.067
1.967
1.949
1.931
1.913
1.645
1.498
1.489
1.471
1.460
1.341
1.324
1.308
1.273
1.264
1.255
1.248
1.238
0.866
0.849
0.831
-0.000
wpl-2-44H
SOLVENT: CDCl3
Pulse length = 10.000 usec
Recycle delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 65536
F1 = 400.100006 MHz
F2 = 1.000000 MHz
Ph
O
H
n-Hex
Ph
3ae
2.03
2.09
4.29
1.06
1.07
1.00
2.09
2.08
2.10
2.13
2.29
6.84
3.27
12
10
8
6
4
2
0
PPM

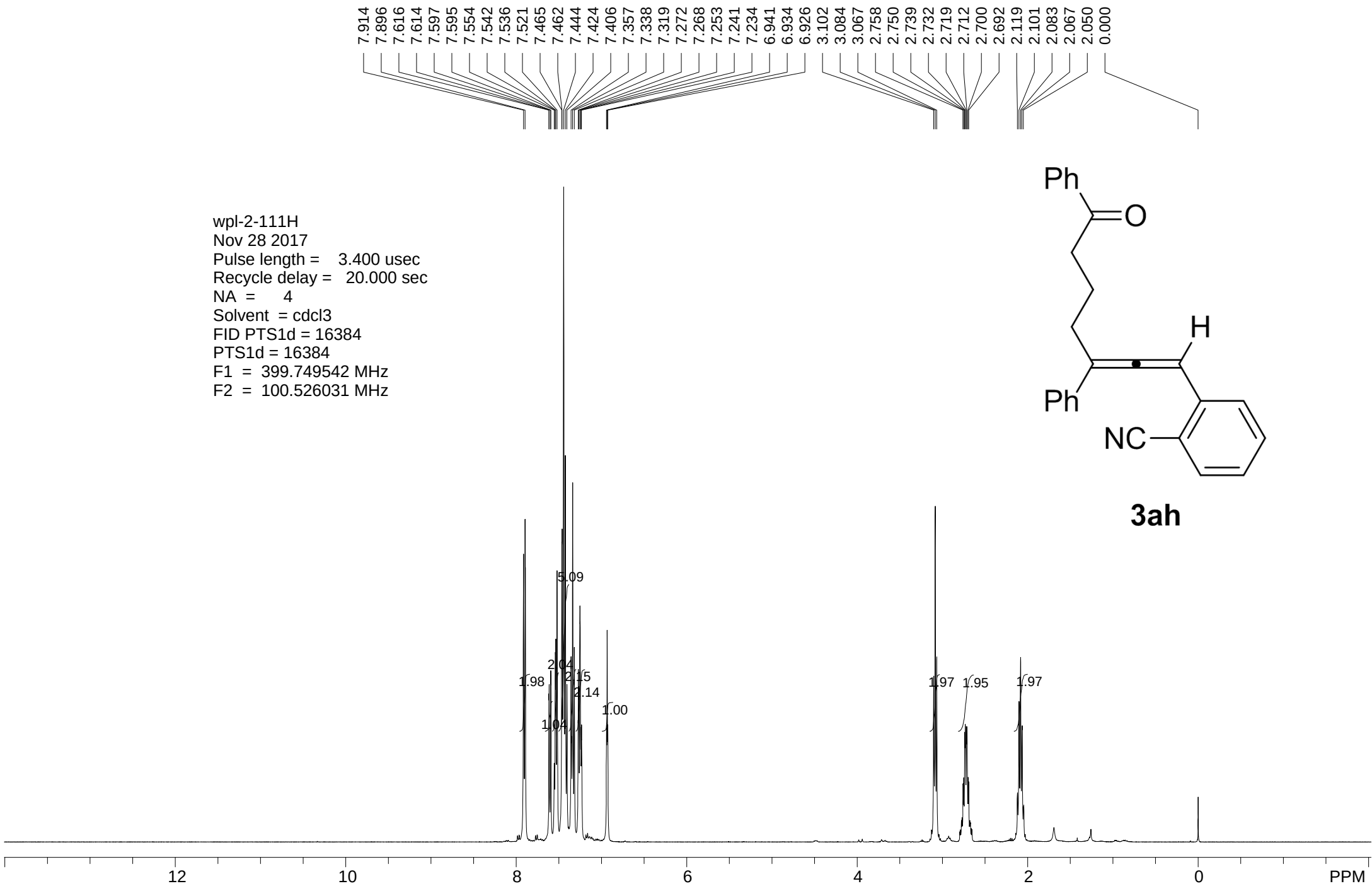

202.237
200.168
136.956
135.861
132.811
128.506
128.460
127.985
126.414
108.074
95.747
77.322
77.000
76.686
37.967
32.734
32.221
31.646
29.087
27.586
22.591
22.001
14.018
wpl-2-44C
Oct 9 2017
Pulse length = 10.000 usec
Recycle delay = 1.000 sec
NA = 512
Solvent = CDCl3
PTS1d = 32768
F1 = 100.605225 MHz
F2 = 1.000000 MHz
Ph
O
H
n-Hex
Ph
3ae
200
150
100
50
0
PPM

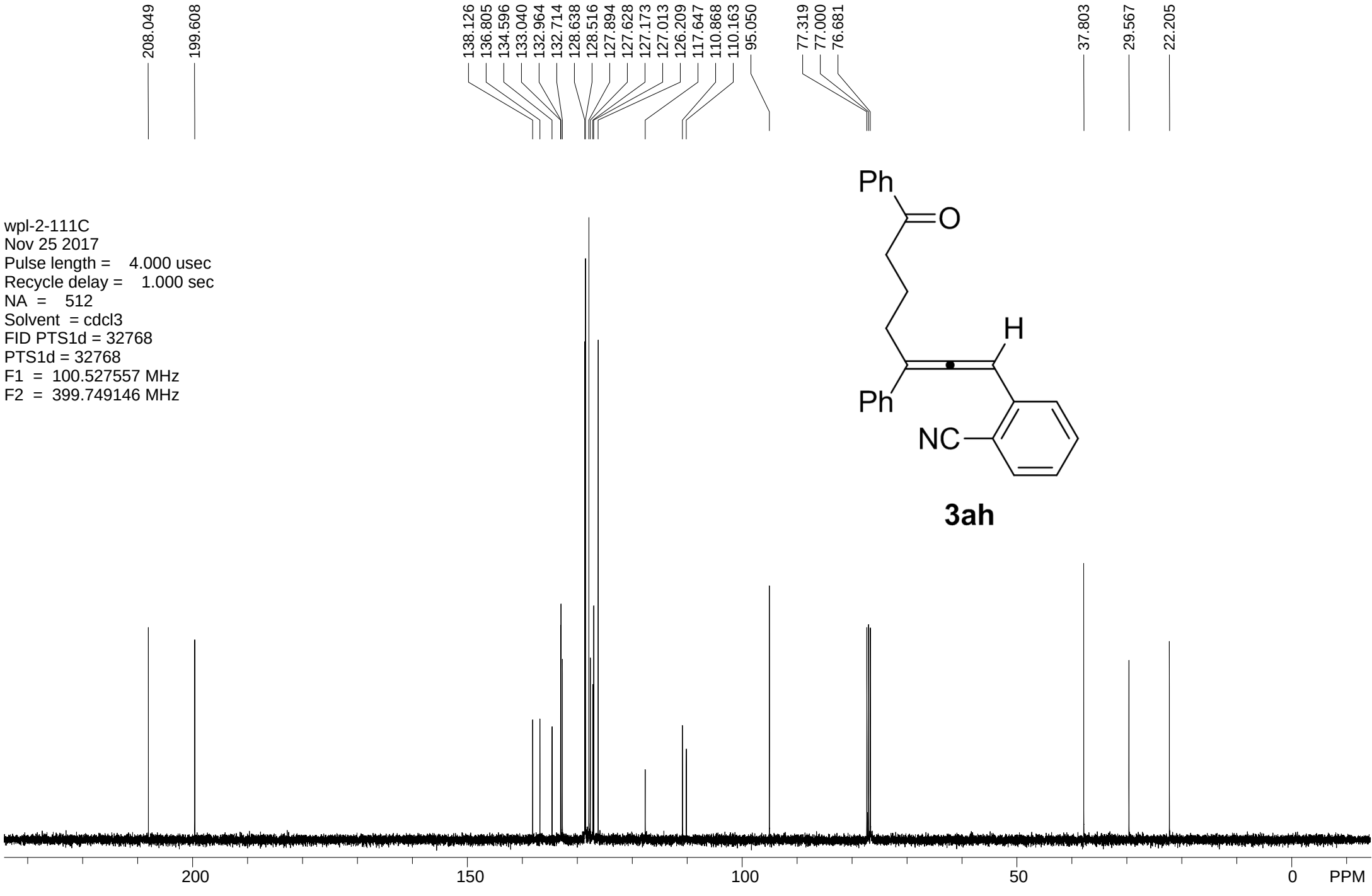

7.868
7.850
7.846
7.555
7.552
7.537
7.519
7.459
7.441
7.431
7.411
7.392
7.346
7.341
7.327
7.308
7.248
7.235
7.217
6.523
6.516
6.508
3.076
3.059
3.041
2.715
2.707
2.702
2.697
2.689
2.678
2.670
2.667
2.659
2.118
2.101
2.094
2.083
2.075
2.065
2.056
2.039
2.021
1.586
-0.000
wpl-2-26H
Sep 22 2017
NA = 8
Solvent = cdcl3
FID PTS1d = 16384
PTS1d = 16384
F1 = 399.749542 MHz
F2 = 100.526031 MHz
Ph
O
H
Ph
Cl
3af
5.44
4.14
2.08
2.02
1.09
1.00
2.02
2.01
2.06
12
10
8
6
4
2
0
PPM

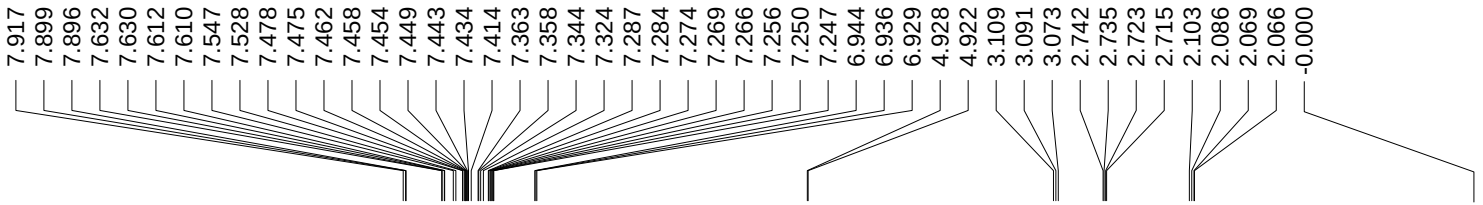

206.561
199.813
136.881
135.423
132.957
132.676
128.903
128.607
128.516
127.932
127.347
126.080
109.768
97.395
77.319
77.000
76.681
37.902
29.552
22.136
wpl-2-26C
Sep 22 2017
NA = 1000
Solvent = cdcl3
FID PTS1d = 32768
PTS1d = 32768
F1 = 100.527557 MHz
F2 = 399.749146 MHz
Ph
O
H
Ph
Cl
3af
200
150
100
50
0
PPM


7.879
7.862
7.551
7.532
7.513
7.450
7.431
7.412
7.393
7.381
7.352
7.334
7.315
7.305
7.263
7.244
7.227
7.140
7.135
7.119
7.114
6.464
6.457
6.450
3.076
3.059
3.041
2.717
2.709
2.700
2.691
2.681
2.672
2.665
2.113
2.096
2.078
2.066
2.060
2.048
2.030
2.012
1.606
-0.000
wpl-2-104H
Nov 22 2017
Pulse length = 3.400 usec
Recycle delay = 20.000 sec
NA = 4
Solvent = cdcl3
FID PTS1d = 16384
PTS1d = 16384
F1 = 399.749542 MHz
F2 = 100.526031 MHz
10.33
2.00
1.01
0.98
2.01
1.99
2.00
Ph
O
H
Ph
Cl
Cl
3ag
12
10
8
6
4
2
0
PPM


206.811
199.616
136.835
135.021
134.763
132.964
132.835
130.672
130.588
128.653
128.516
128.197
127.879
127.529
126.118
125.905
110.322
96.644
77.319
77.000
76.681
37.818
29.484
22.023
wpl-2-104C
Nov 22 2017
Pulse length = 4.000 usec
Recycle delay = 1.000 sec
NA = 512
Solvent = cdcl3
FID PTS1d = 32768
PTS1d = 32768
F1 = 100.527557 MHz
F2 = 399.749146 MHz
Ph
O
H
Ph
Cl
Cl
3ag
200
150
100
50
0
PPM

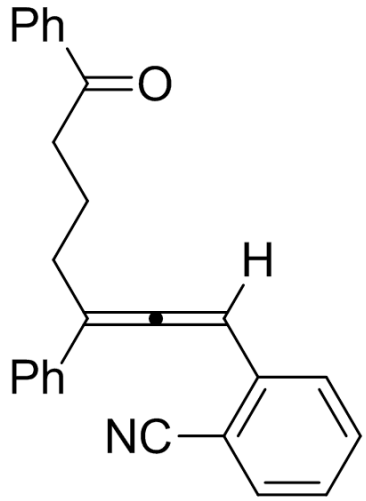

7.914
7.896
7.616
7.614
7.597
7.595
7.554
7.542
7.536
7.521
7.465
7.462
7.444
7.424
7.406
7.357
7.338
7.319
7.272
7.268
7.253
7.241
7.234
6.941
6.934
6.926
3.102
3.084
3.067
2.758
2.750
2.739
2.732
2.719
2.712
2.700
2.692
2.119
2.101
2.083
2.067
2.050
0.000
wpl-2-111H
Nov 28 2017
Pulse length = 3.400 usec
Recycle delay = 20.000 sec
NA = 4
Solvent = cdcl3
FID PTS1d = 16384
PTS1d = 16384
F1 = 399.749542 MHz
F2 = 100.526031 MHz
Ph
O
H
Ph
NC
3ah
5.09
2.04
2.15
2.14
1.98
1.04
1.00
1.97
1.95
1.97
12
10
8
6
4
2
0
PPM


208.049
199.608
138.126
136.805
134.596
133.040
132.964
132.714
128.638
128.516
127.894
127.628
127.173
127.013
126.209
117.647
110.868
110.163
95.050
77.319
77.000
76.681
37.803
29.567
22.205
wpl-2-111C
Nov 25 2017
Pulse length = 4.000 usec
Recycle delay = 1.000 sec
NA = 512
Solvent = cdcl3
FID PTS1d = 32768
PTS1d = 32768
F1 = 100.527557 MHz
F2 = 399.749146 MHz
Ph
O
H
Ph
NC
3ah
200
150
100
50
0
PPM

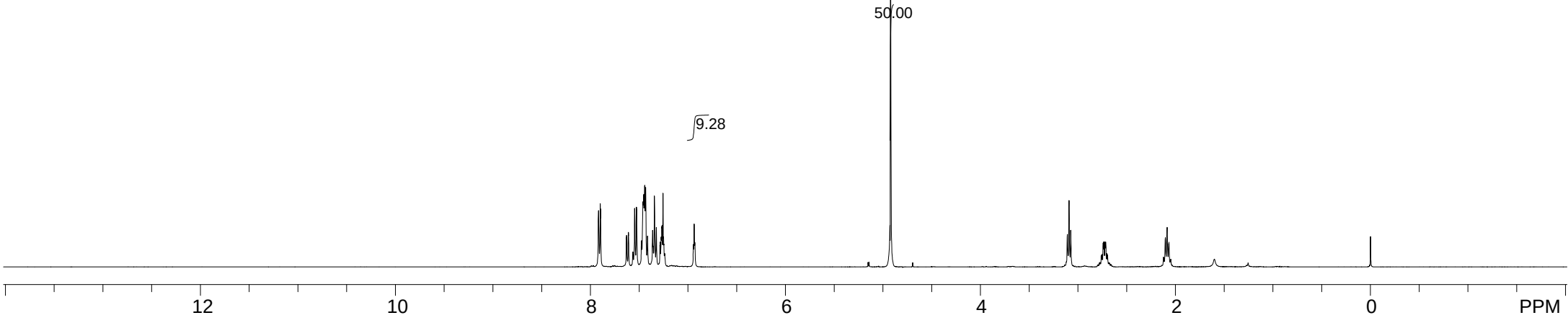


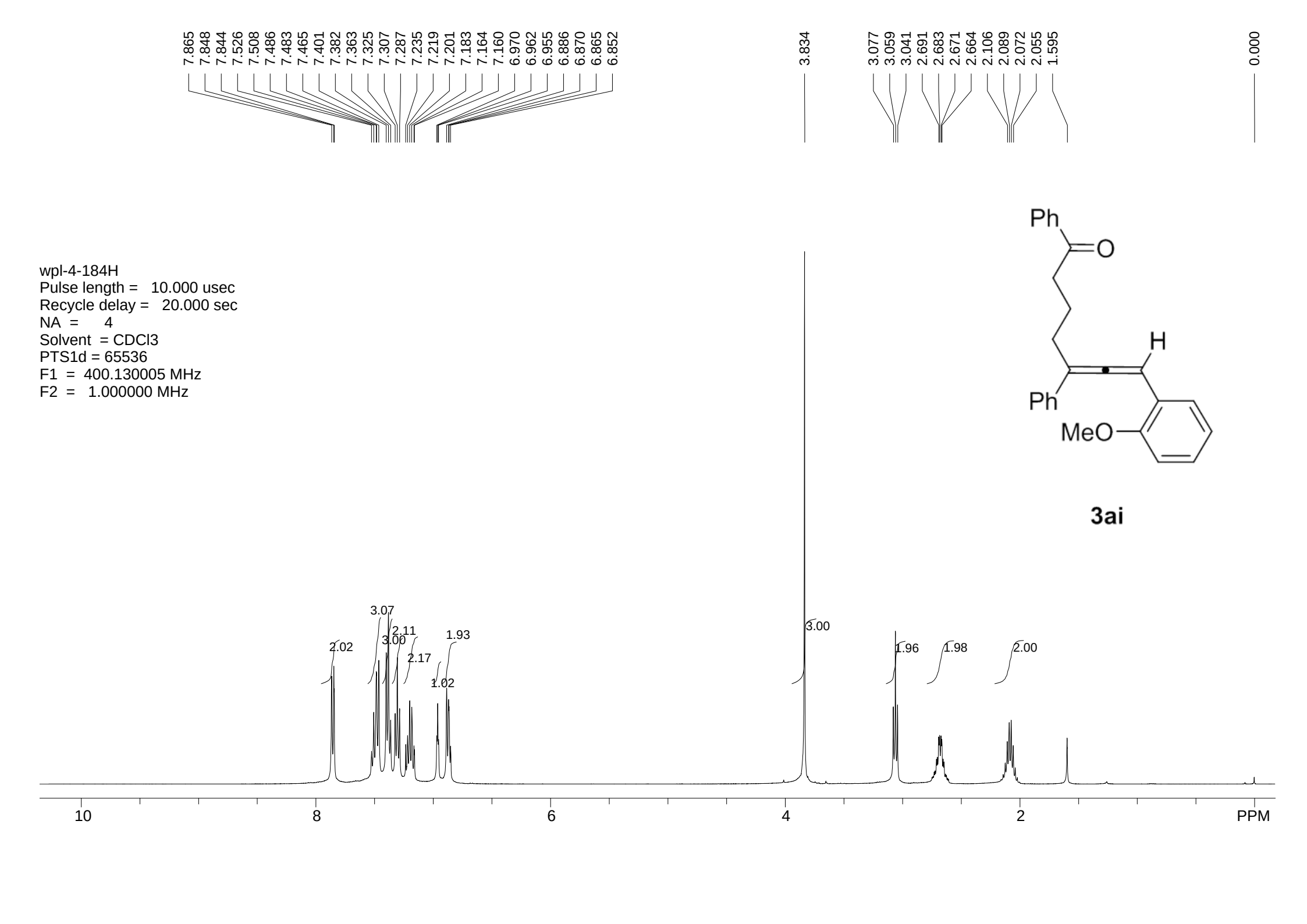
Purity was determined to be 96% by using 17.5 μL dibromomethane as the internal standard in 35.2 mg sample

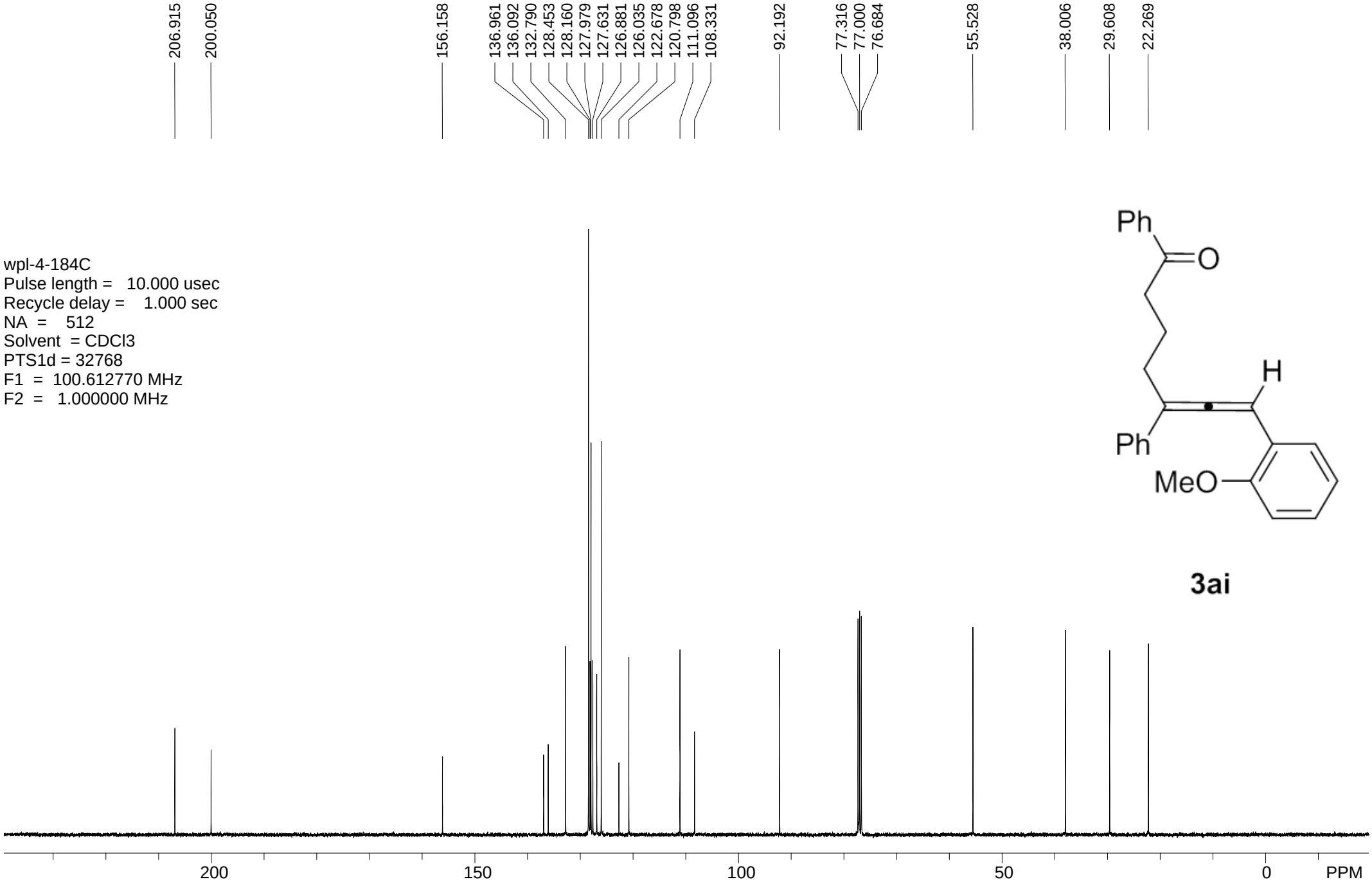
wpl-2-111P
Nov 28 2017
Pulse length = 3.400 usec
Recycle delay = 20.000 sec
NA = 4
Solvent = cdcl3
FID PTS1d = 16384
PTS1d = 16384
F1 = 399.749542 MHz
F2 = 100.526031 MHz

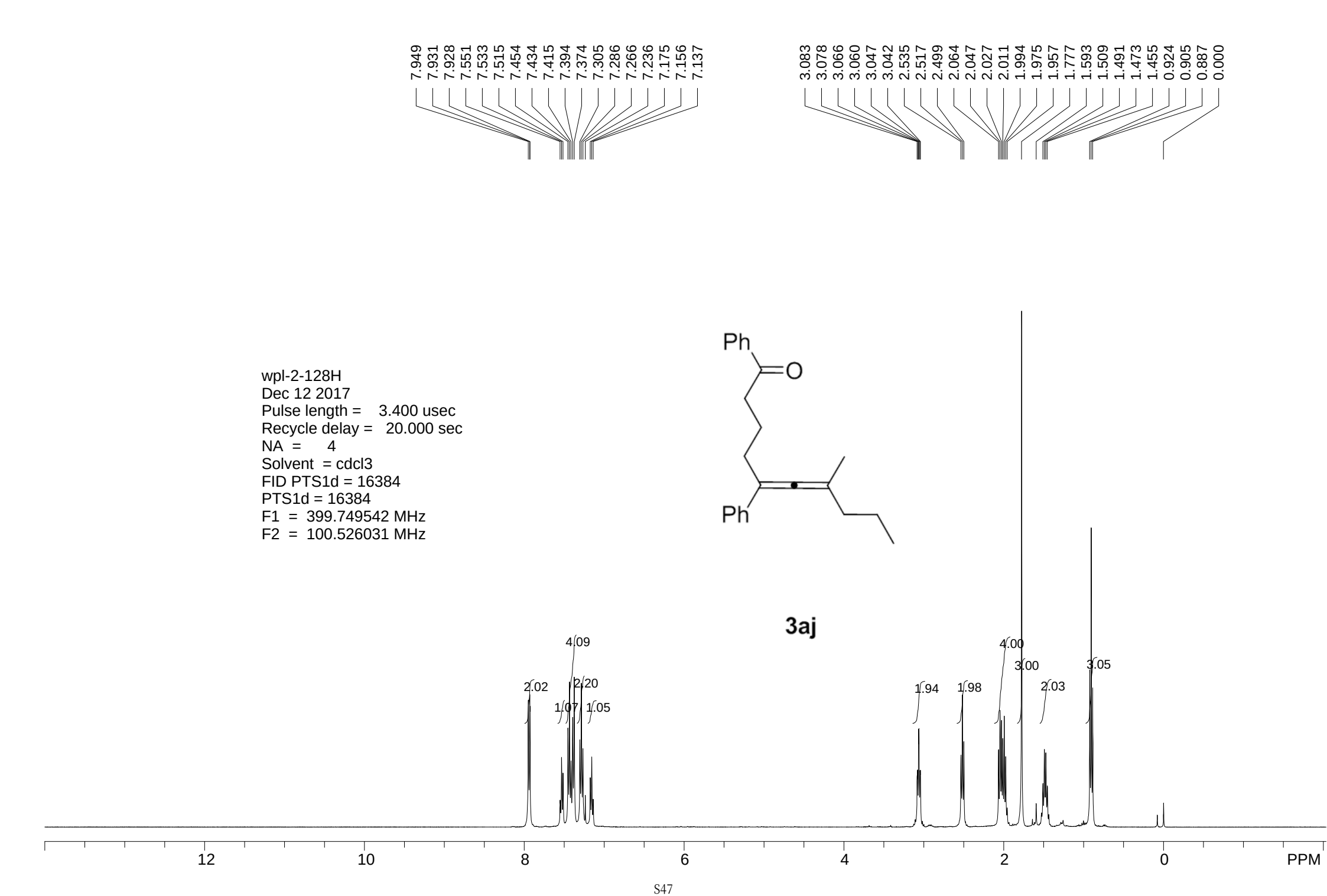


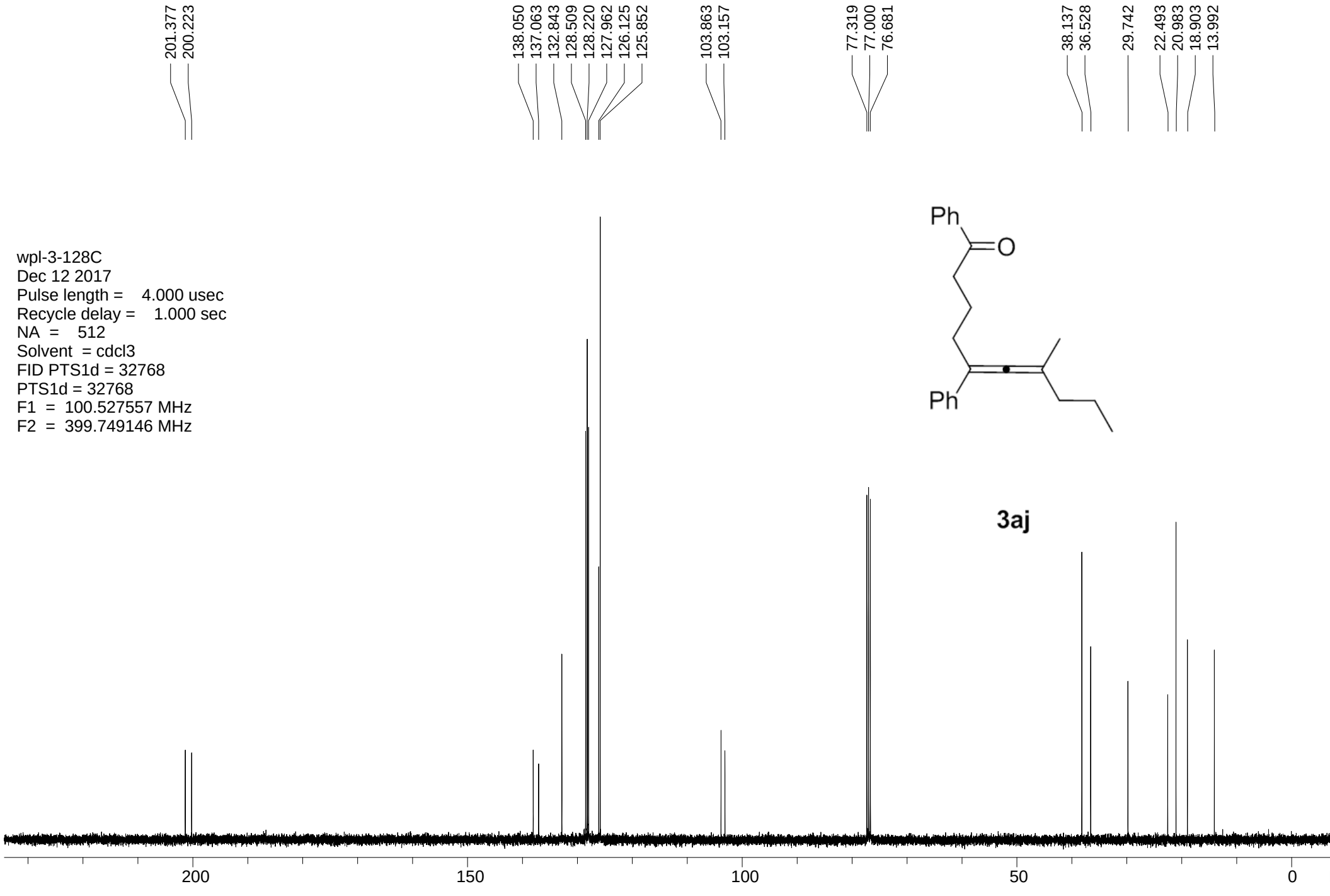
3ah

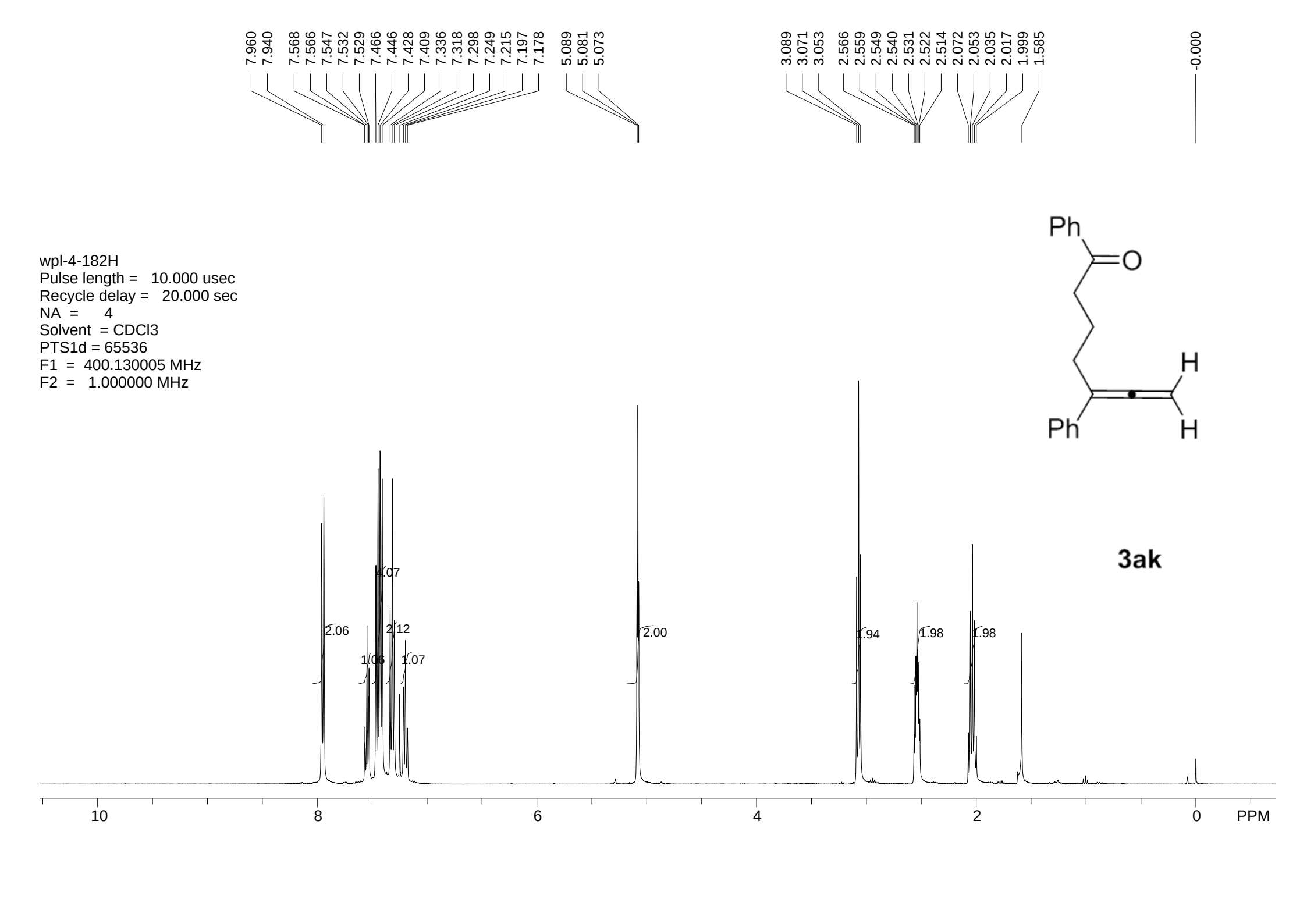


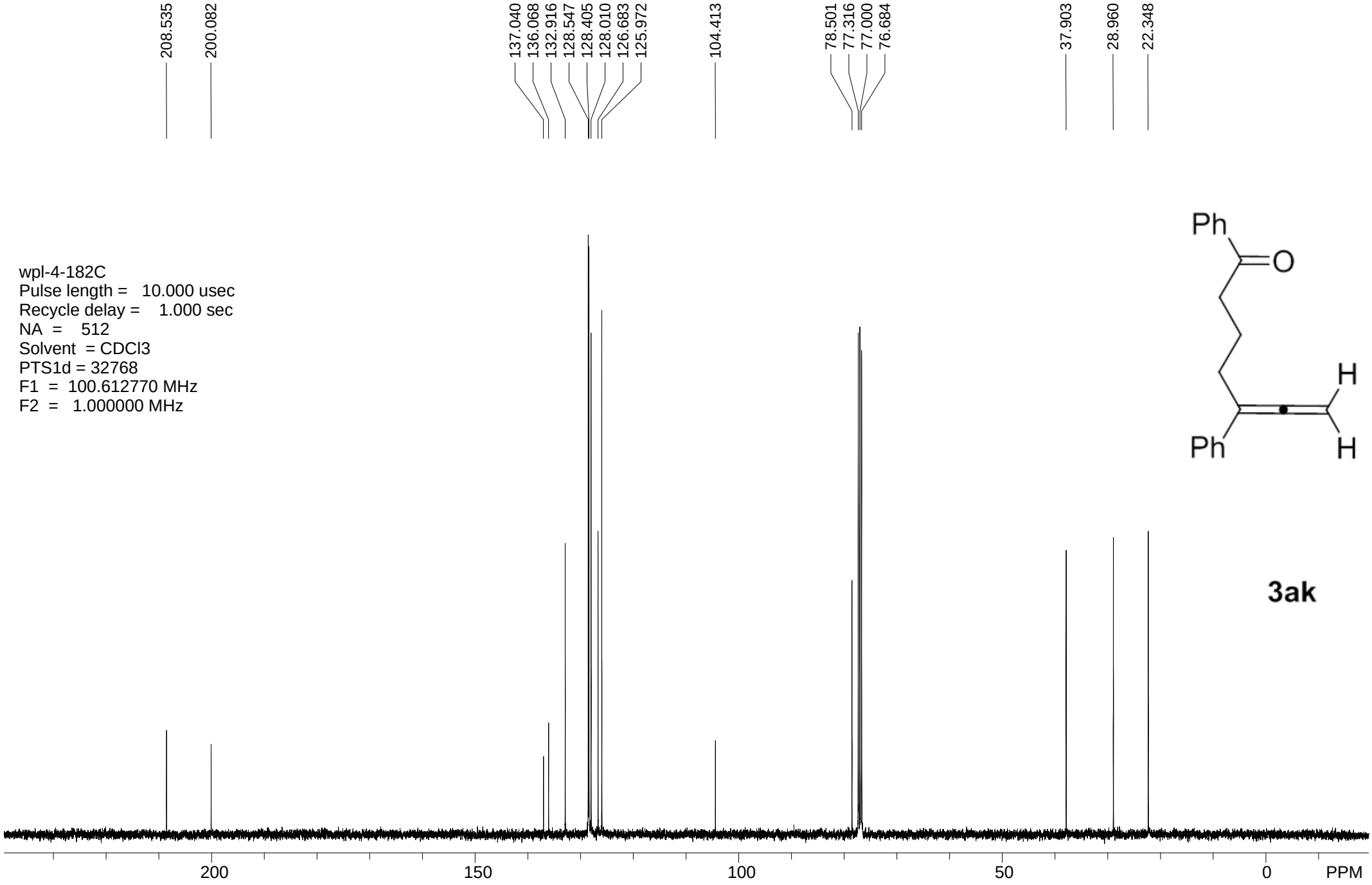

7.865
7.848
7.844
7.526
7.508
7.486
7.483
7.465
7.401
7.382
7.363
7.325
7.307
7.287
7.235
7.219
7.201
7.183
7.164
7.160
6.970
6.962
6.955
6.886
6.870
6.865
6.852
3.834
3.077
3.059
3.041
2.691
2.683
2.671
2.664
2.106
2.089
2.072
2.055
1.595
0.000
wpl-4-184H
Pulse length = 10.000 usec
Recycle delay = 20.000 sec
NA = 4
Solvent = CDCl3
PTS1d = 65536
F1 = 400.130005 MHz
F2 = 1.000000 MHz
Ph
O
H
Ph
MeO
3ai
2.02
3.07
3.00
2.11
2.17
1.02
1.93
3.00
1.96
1.98
2.00
10
8
6
4
2
PPM

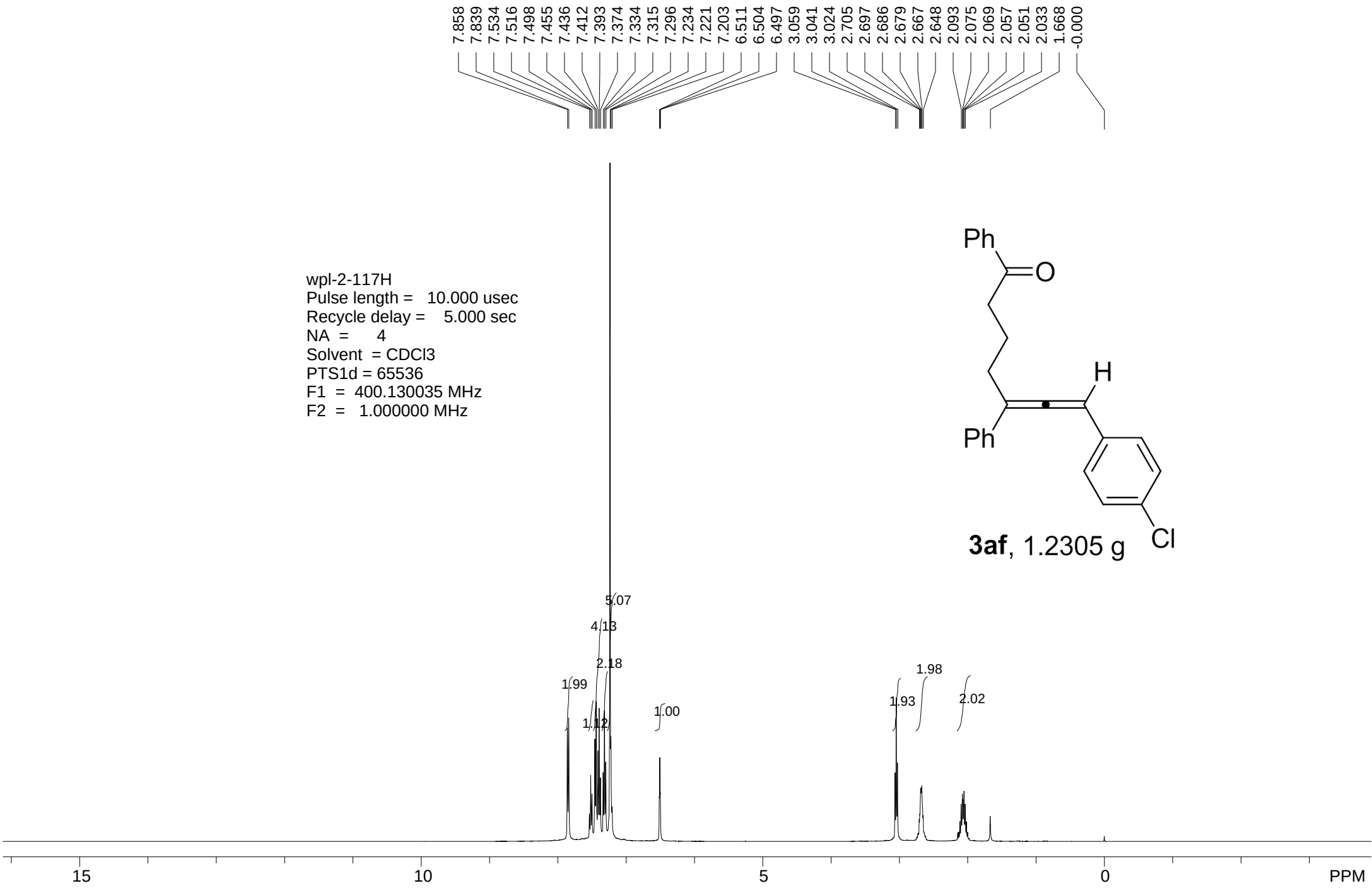

206.915
200.050
156.158
136.961
136.092
132.790
128.453
128.160
127.979
127.631
126.881
126.035
122.678
120.798
111.096
108.331
92.192
77.316
77.000
76.684
55.528
38.006
29.608
22.269
wpl-4-184C
Pulse length = 10.000 usec
Recycle delay = 1.000 sec
NA = 512
Solvent = CDCl3
PTS1d = 32768
F1 = 100.612770 MHz
F2 = 1.000000 MHz
Ph
O
H
Ph
MeO
3ai
200
150
100
50
0
PPM

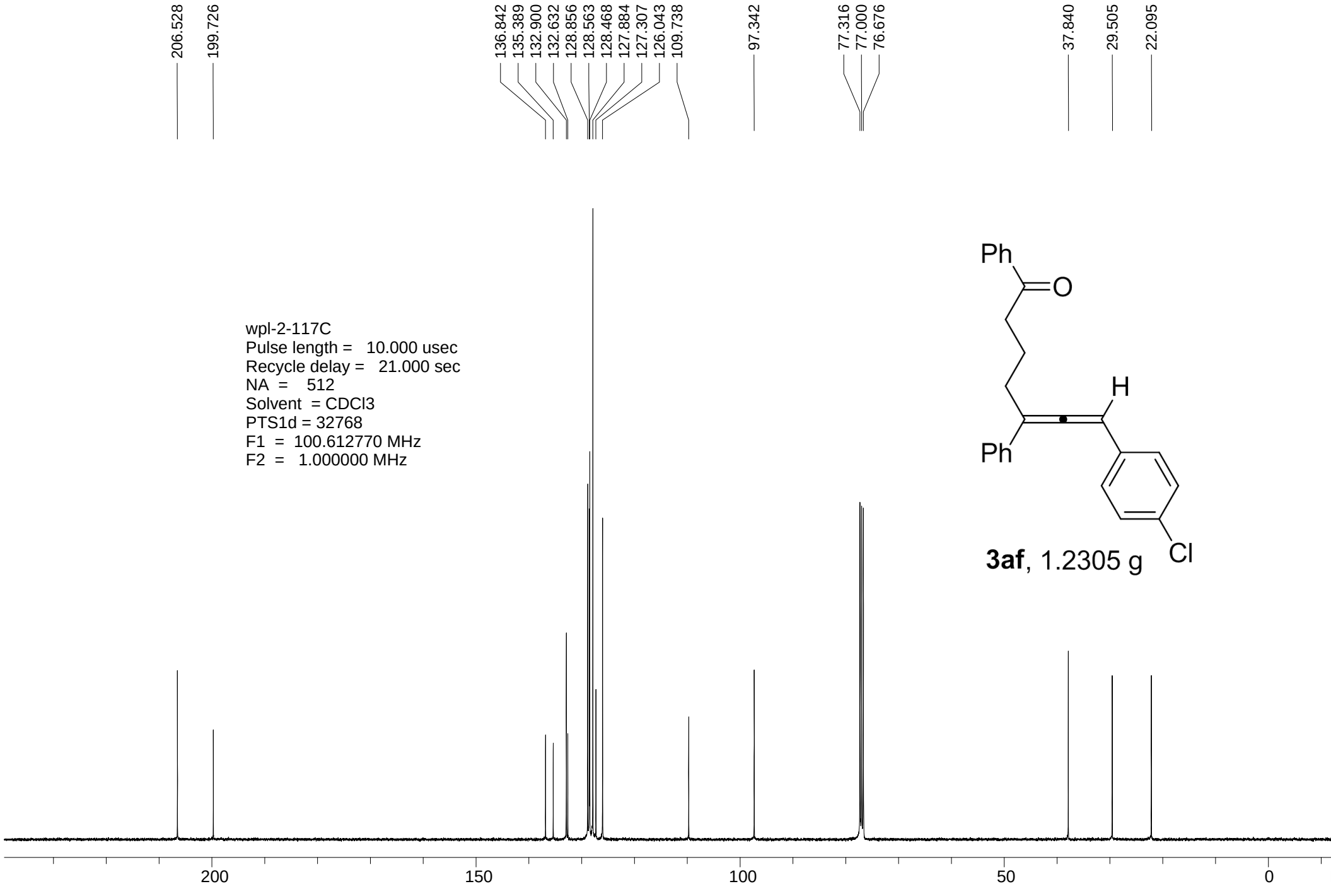

7.949
7.931
7.928
7.551
7.533
7.515
7.454
7.434
7.415
7.394
7.374
7.305
7.286
7.266
7.236
7.175
7.156
7.137
3.083
3.078
3.066
3.060
3.047
3.042
2.535
2.517
2.499
2.064
2.047
2.027
2.011
1.994
1.975
1.957
1.777
1.593
1.509
1.491
1.473
1.455
0.924
0.905
0.887
0.000
wpl-2-128H
Dec 12 2017
Pulse length = 3.400 usec
Recycle delay = 20.000 sec
NA = 4
Solvent = cdcl3
FID PTS1d = 16384
PTS1d = 16384
F1 = 399.749542 MHz
F2 = 100.526031 MHz
Ph
O
Ph
3aj
4.09
2.02
2.20
1.07
1.05
1.94
1.98
4.00
3.00
2.03
3.05
12
10
8
6
4
2
0
PPM

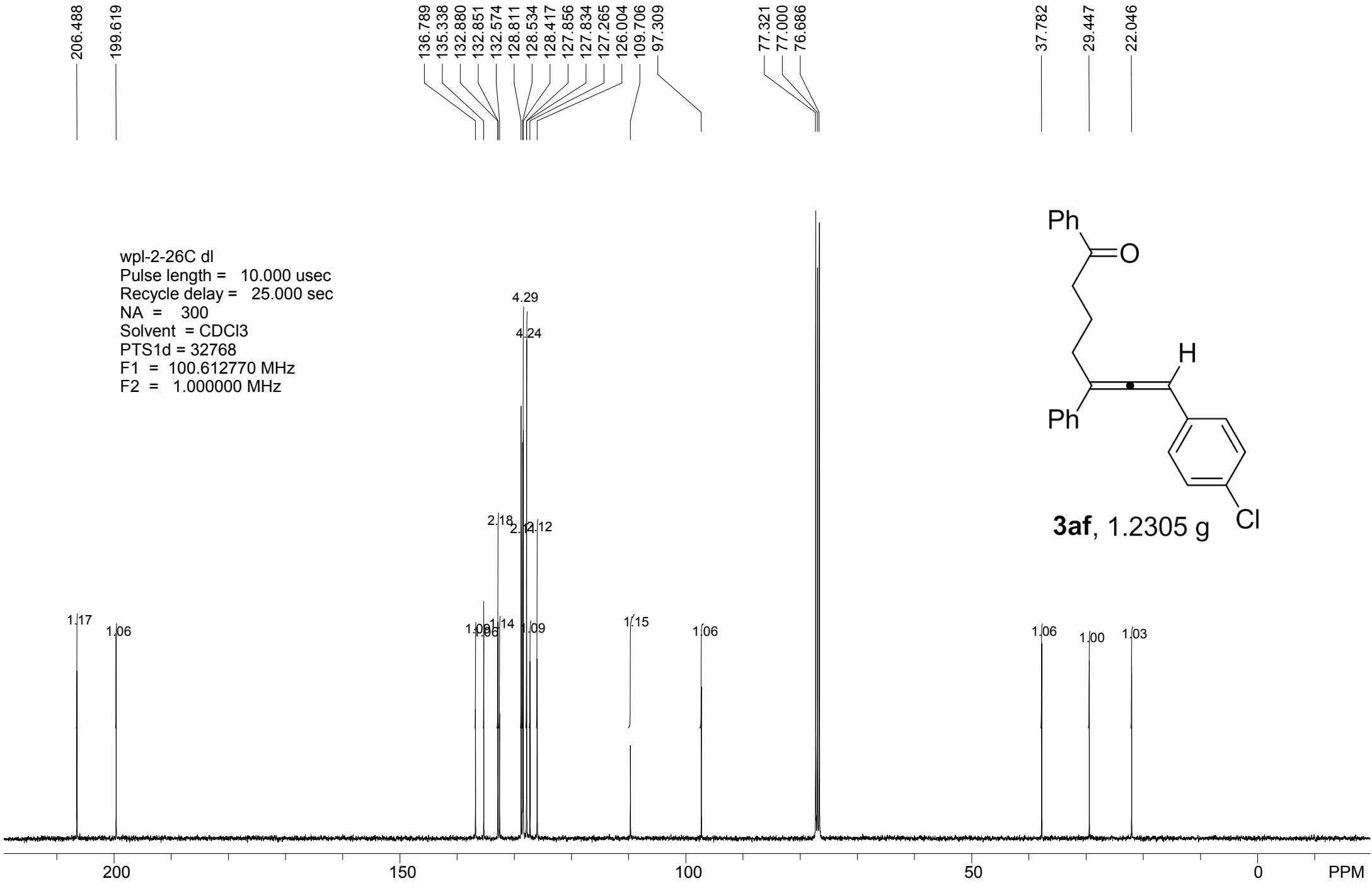

201.377
200.223
138.050
137.063
132.843
128.509
128.220
127.962
126.125
125.852
103.863
103.157
77.319
77.000
76.681
38.137
36.528
29.742
22.493
20.983
18.903
13.992
wpl-3-128C
Dec 12 2017
Pulse length = 4.000 usec
Recycle delay = 1.000 sec
NA = 512
Solvent = cdcl3
FID PTS1d = 32768
PTS1d = 32768
F1 = 100.527557 MHz
F2 = 399.749146 MHz
Ph
O
Ph
3aj
200
150
100
50
0
PPM

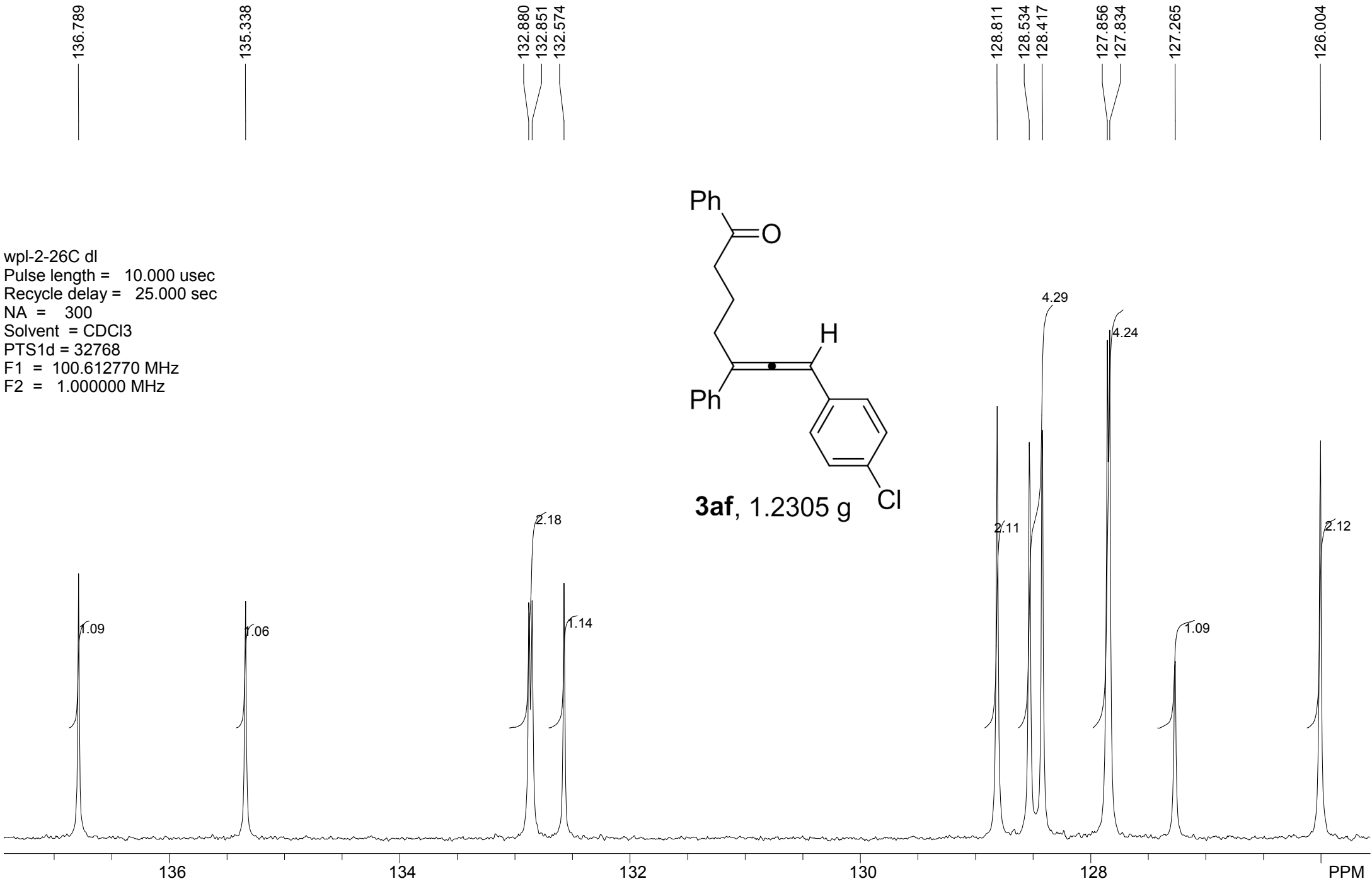

7.960
7.940
7.568
7.566
7.547
7.532
7.529
7.466
7.446
7.428
7.409
7.336
7.318
7.298
7.249
7.215
7.197
7.178
5.089
5.081
5.073
3.089
3.071
3.053
2.566
2.559
2.549
2.540
2.531
2.522
2.514
2.072
2.053
2.035
2.017
1.999
1.585
-0.000
wpl-4-182H
Pulse length = 10.000 usec
Recycle delay = 20.000 sec
NA = 4
Solvent = CDCl3
PTS1d = 65536
F1 = 400.130005 MHz
F2 = 1.000000 MHz
Ph
O
H
Ph
H
3ak
2.06
4.07
2.12
1.06
1.07
2.00
1.94
1.98
1.98
10
8
6
4
2
0
PPM

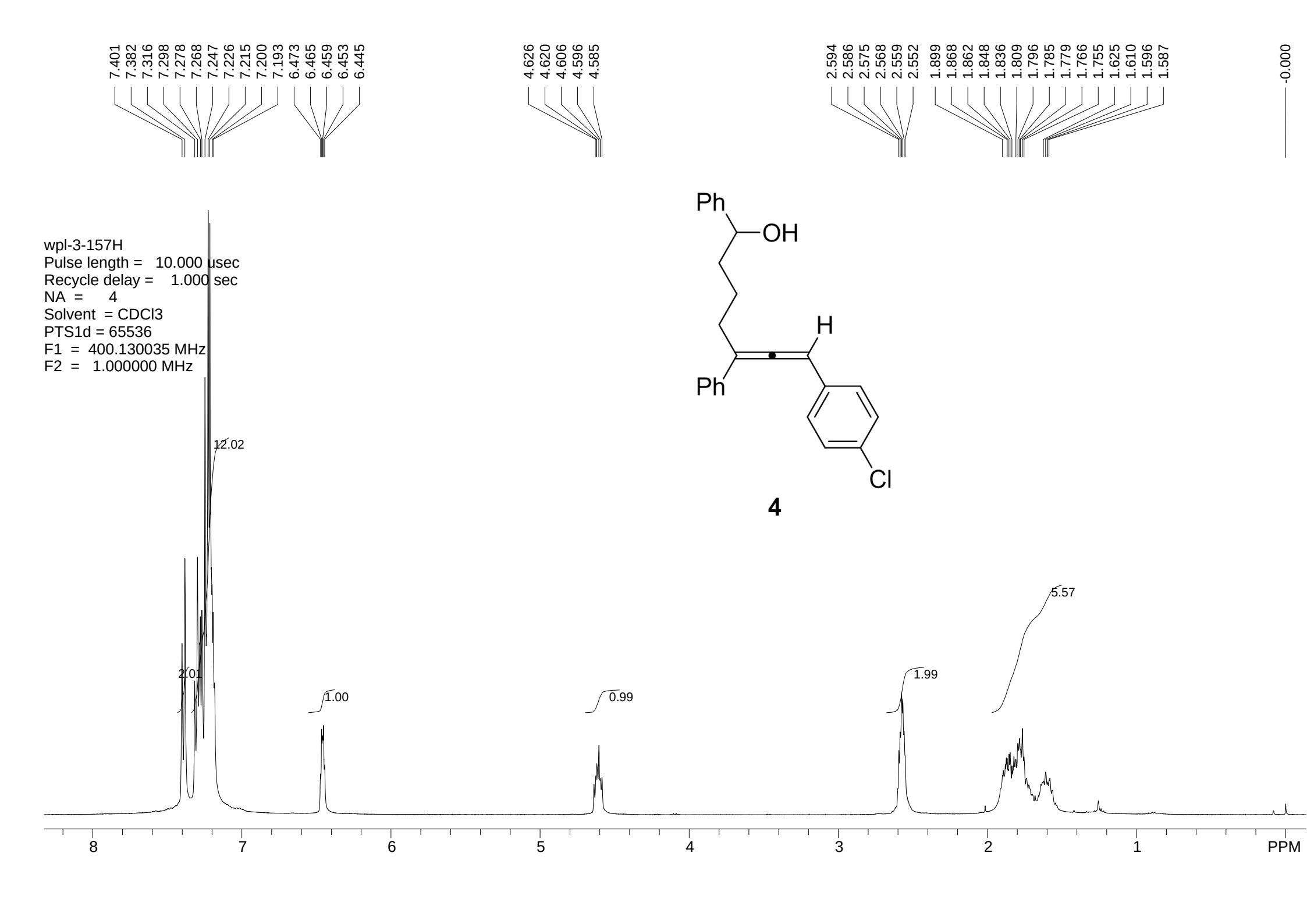

208.535
200.082
137.040
136.068
132.916
128.547
128.405
128.010
126.683
125.972
104.413
78.501
77.316
77.000
76.684
37.903
28.960
22.348
wpl-4-182C
Pulse length = 10.000 usec
Recycle delay = 1.000 sec
NA = 512
Solvent = CDCl3
PTS1d = 32768
F1 = 100.612770 MHz
F2 = 1.000000 MHz
Ph
O
H
Ph
H
3ak
200
150
100
50
0
PPM

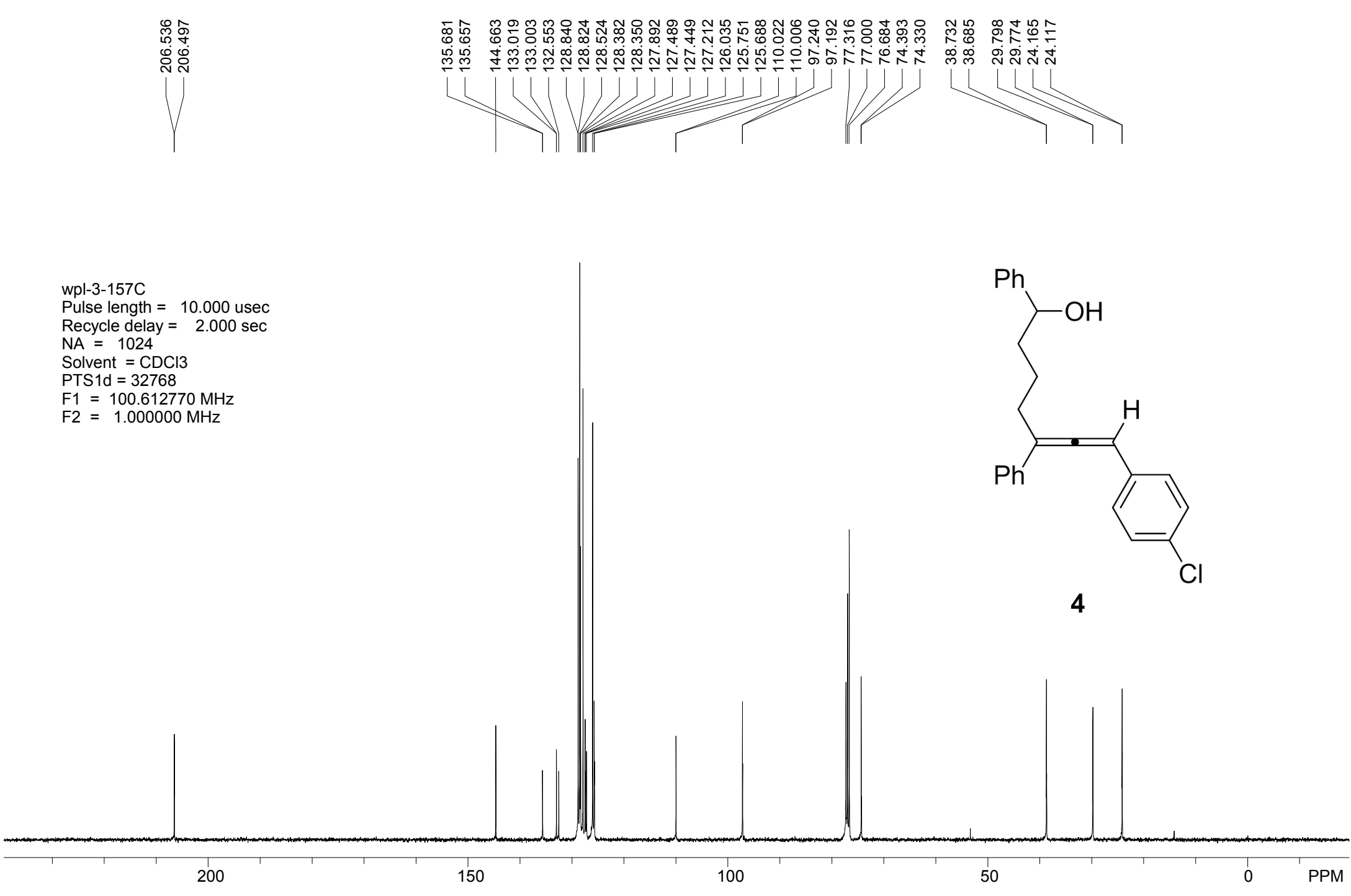

7.858
7.839
7.534
7.516
7.498
7.455
7.436
7.412
7.393
7.374
7.334
7.315
7.296
7.234
7.221
7.203
6.511
6.504
6.497
3.059
3.041
3.024
2.705
2.697
2.686
2.679
2.667
2.648
2.093
2.075
2.069
2.057
2.051
2.033
1.668
-0.000
wpl-2-117H
Pulse length = 10.000 usec
Recycle delay = 5.000 sec
NA = 4
Solvent = CDCl3
PTS1d = 65536
F1 = 400.130035 MHz
F2 = 1.000000 MHz
Ph
O
H
Ph
Cl
3af, 1.2305 g
5.07
4.13
2.18
1.99
1.12
1.00
1.98
1.93
2.02
15
10
5
0
PPM

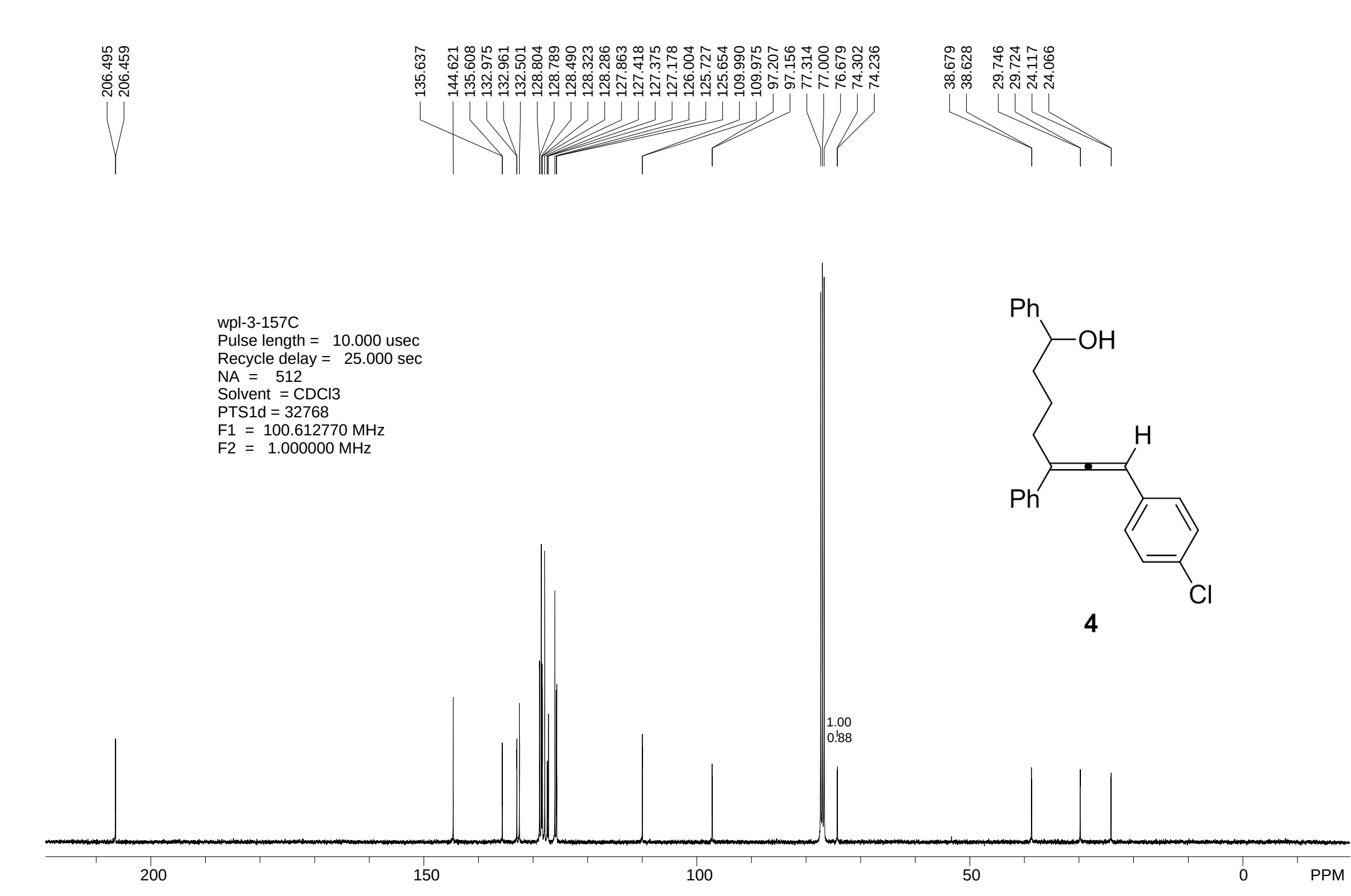

206.528
199.726
136.842
135.389
132.900
132.632
128.856
128.563
128.468
127.884
127.307
126.043
109.738
97.342
77.316
77.000
76.676
37.840
29.505
22.095
wpl-2-117C
Pulse length = 10.000 usec
Recycle delay = 21.000 sec
NA = 512
Solvent = CDCl3
PTS1d = 32768
F1 = 100.612770 MHz
F2 = 1.000000 MHz
Ph
O
H
Ph
Cl
3af, 1.2305 g
200
150
100
50
0
PPM

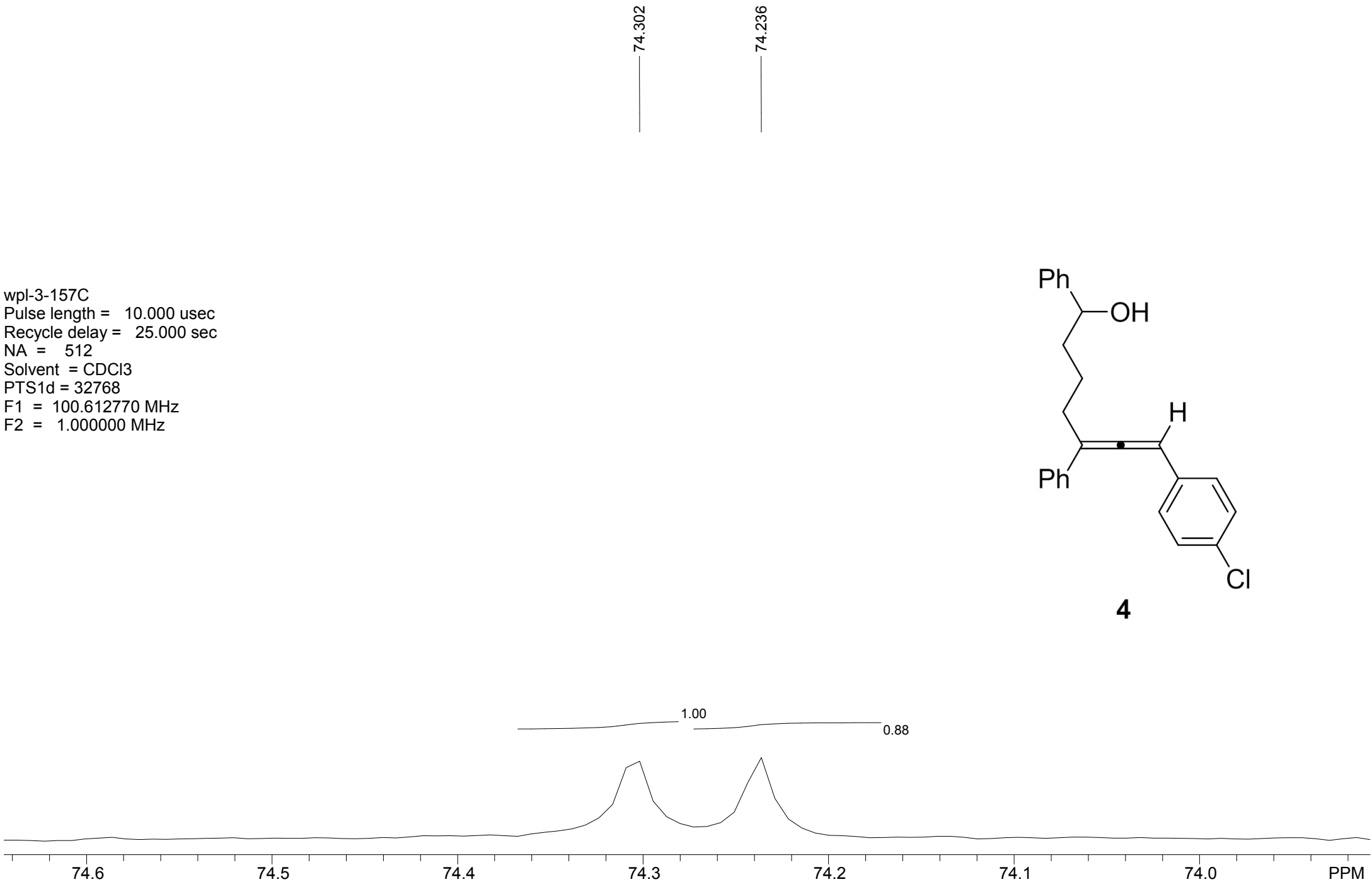

206.488
199.619
136.789
135.338
132.880
132.851
132.574
128.811
128.534
128.417
127.856
127.834
127.265
126.004
109.706
97.309
77.321
77.000
76.686
37.782
29.447
22.046
wpl-2-26C dl
Pulse length = 10.000 usec
Recycle delay = 25.000 sec
NA = 300
Solvent = CDCl3
PTS1d = 32768
F1 = 100.612770 MHz
F2 = 1.000000 MHz
4.29
4.24
2.18
2.12
1.17
1.06
1.14
1.09
1.15
1.06
1.06
1.00
1.03
Ph
O
H
Ph
Cl
3af, 1.2305 g
200
150
100
50
0
PPM

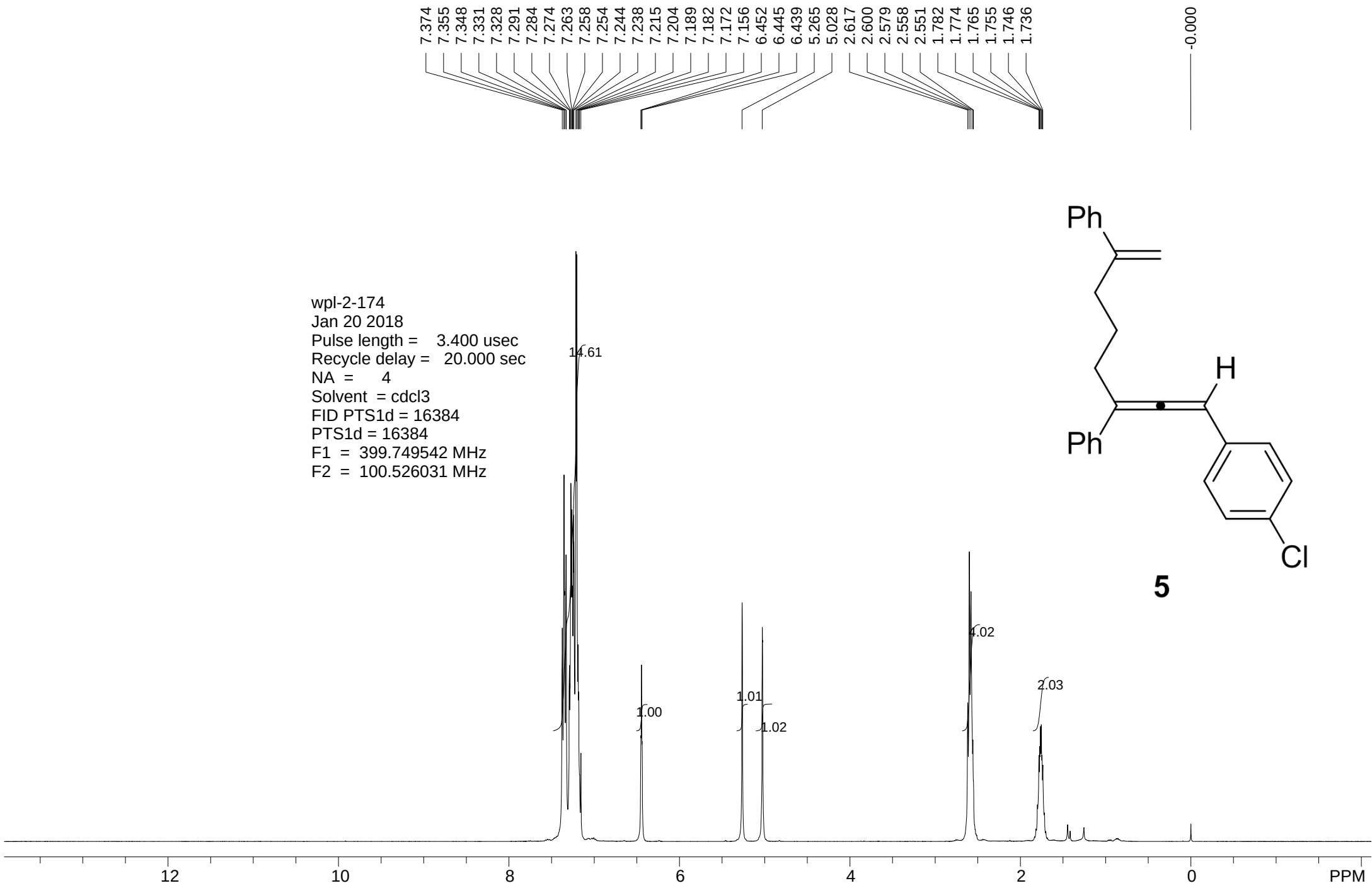

136.789
135.338
132.880
132.851
132.574
128.811
128.534
128.417
127.856
127.834
127.265
126.004
wpl-2-26C dl
Pulse length = 10.000 usec
Recycle delay = 25.000 sec
NA = 300
Solvent = CDCl3
PTS1d = 32768
F1 = 100.612770 MHz
F2 = 1.000000 MHz
Ph
O
H
Ph
Cl
3af, 1.2305 g
1.09
1.06
2.18
1.14
2.11
4.29
4.24
1.09
2.12
136
134
132
130
128
PPM

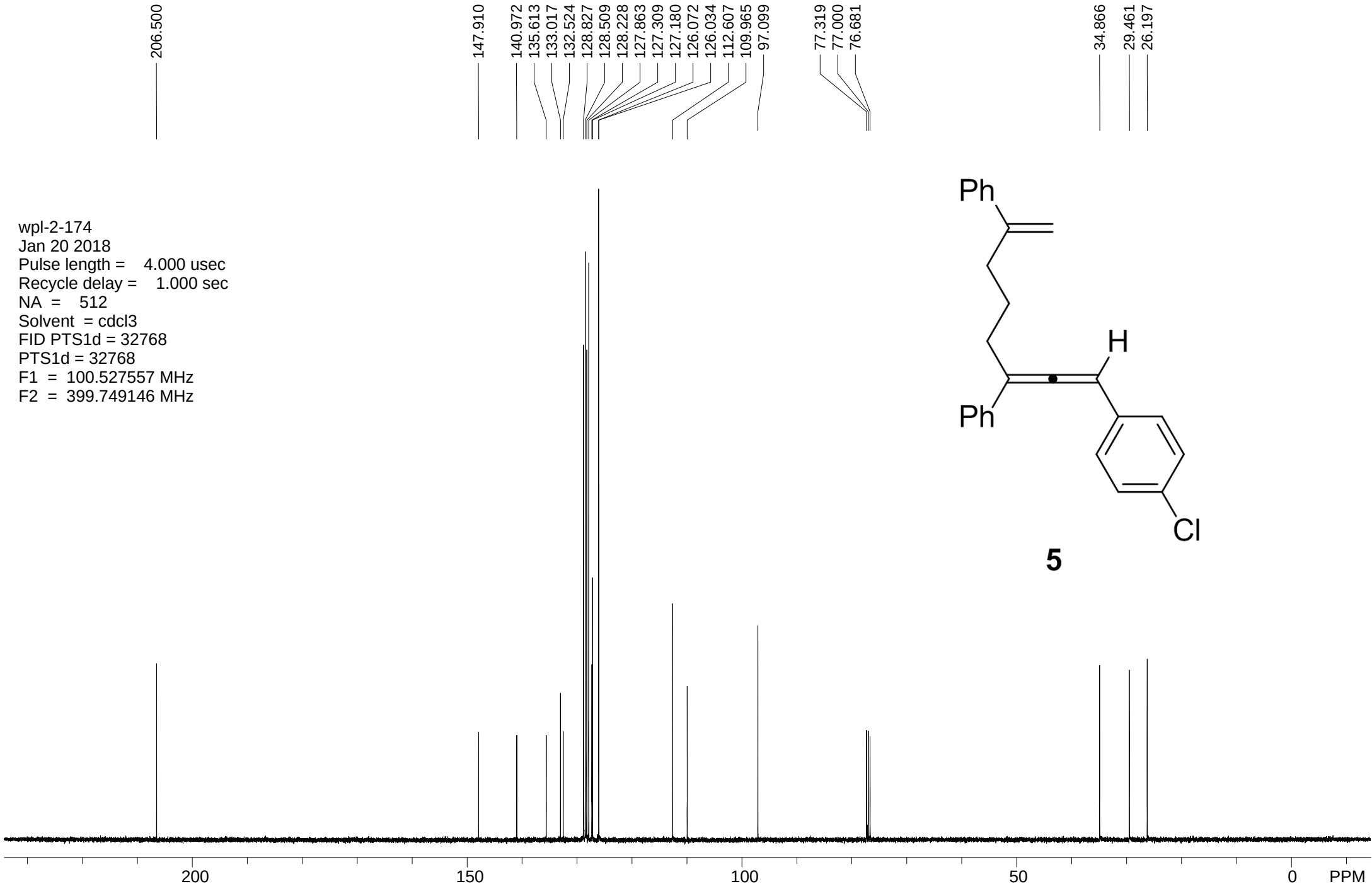

7.401
7.382
7.316
7.298
7.278
7.268
7.247
7.226
7.215
7.200
7.193
6.473
6.465
6.459
6.453
6.445
4.626
4.620
4.606
4.596
4.585
2.594
2.586
2.575
2.568
2.559
2.552
1.899
1.868
1.862
1.848
1.836
1.809
1.796
1.785
1.779
1.766
1.755
1.625
1.610
1.596
1.587
-0.000
wpl-3-157H
Pulse length = 10.000 usec
Recycle delay = 1.000 sec
NA = 4
Solvent = CDCl3
PTS1d = 65536
F1 = 400.130035 MHz
F2 = 1.000000 MHz
Ph
OH
H
Ph
Cl
4
12.02
2.01
1.00
0.99
1.99
5.57
8
7
6
5
4
3
2
1
PPM


206.536
206.497
135.681
135.657
144.663
133.019
133.003
132.553
128.840
128.824
128.524
128.382
128.350
127.892
127.489
127.449
127.212
126.035
125.751
125.688
110.022
110.006
97.240
97.192
77.316
77.000
76.684
74.393
74.330
38.732
38.685
29.798
29.774
24.165
24.117
wpl-3-157C
Pulse length = 10.000 usec
Recycle delay = 2.000 sec
NA = 1024
Solvent = CDCl3
PTS1d = 32768
F1 = 100.612770 MHz
F2 = 1.000000 MHz
Ph
OH
H
Ph
Cl
4
200
150
100
50
0
PPM


206.495
206.459
135.637
144.621
135.608
132.975
132.961
132.501
128.804
128.789
128.490
128.323
128.286
127.863
127.418
127.375
127.178
126.004
125.727
125.654
109.990
109.975
97.207
97.156
77.314
77.000
76.679
74.302
74.236
38.679
38.628
29.746
29.724
24.117
24.066
wpl-3-157C
Pulse length = 10.000 usec
Recycle delay = 25.000 sec
NA = 512
Solvent = CDCl3
PTS1d = 32768
F1 = 100.612770 MHz
F2 = 1.000000 MHz
Ph
OH
H
Ph
Cl
4
1.00
0.88
200
150
100
50
0
PPM


74.302
74.236
wpl-3-157C
Pulse length = 10.000 usec
Recycle delay = 25.000 sec
NA = 512
Solvent = CDCl3
PTS1d = 32768
F1 = 100.612770 MHz
F2 = 1.000000 MHz
Ph
OH
H
Ph
Cl
4
1.00
0.88
74.6
74.5
74.4
74.3
74.2
74.1
74.0
PPM


7.374
7.355
7.348
7.331
7.328
7.291
7.284
7.274
7.263
7.258
7.254
7.244
7.238
7.215
7.204
7.189
7.182
7.172
7.156
6.452
6.445
6.439
5.265
5.028
2.617
2.600
2.579
2.558
2.551
1.782
1.774
1.765
1.755
1.746
1.736
-0.000
wpl-2-174
Jan 20 2018
Pulse length = 3.400 usec
Recycle delay = 20.000 sec
NA = 4
Solvent = cdcl3
FID PTS1d = 16384
PTS1d = 16384
F1 = 399.749542 MHz
F2 = 100.526031 MHz
14.61
1.00
1.01
1.02
4.02
2.03
Ph
H
Ph
Cl
5
12
10
8
6
4
2
0
PPM


206.500
147.910
140.972
135.613
133.017
132.524
128.827
128.509
128.228
127.863
127.309
127.180
126.072
126.034
112.607
109.965
97.099
77.319
77.000
76.681
34.866
29.461
26.197
wpl-2-174
Jan 20 2018
Pulse length = 4.000 usec
Recycle delay = 1.000 sec
NA = 512
Solvent = cdcl3
FID PTS1d = 32768
PTS1d = 32768
F1 = 100.527557 MHz
F2 = 399.749146 MHz
Ph
H
Ph
Cl
5
200
150
100
50
0
PPM