Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Pd/Gorlos-Phos-Catalyzed Cross-Coupling Between Two Different Aryl Chlorides in the Presence of B_2Pin_2 and Cytotoxicity Studies of the Products

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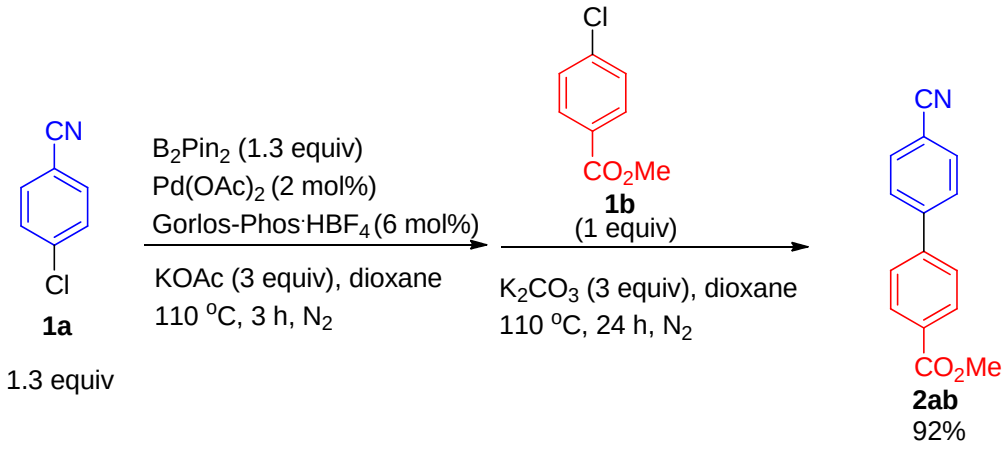
Supporting Information

General information

^{1}H NMR, ^{13}C NMR, and ^{19}F NMR spectra were recorded in $CDCl_3$ using a Bruker AM 300 MHz NMR spectrometer (^{1}H at 300 MHz, ^{13}C at 75 MHz, ^{19}F at 282 MHz). NMR spectra were taken using TMS (^{1}H, $\delta = 0$), residual $CHCl_3$ (7.26 ppm) in $CDCl_3$, and $CFCl_3$ (^{19}F $CFCl_3$, $\delta = 0$) as the internal standards, respectively. IR spectra were recorded with a Perkin–Elmer 983G instrument. Elemental analyses were measured with a Carlo-Erba EA1110 elementary analysis instrument. Mass spectrometry was performed with an HP 5989A system. High-resolution mass spectrometry was determined with a Finnigan MAT 8430 or Bruker APEXIII instrument. $Pd(OAc)_2$ was purchased from Adamas. Dioxane was distilled from Na wire using benzophenone as the indicator under N_2 before use. Gorlos-Phos·HBF_4 was prepared according to our previous work.[1]

Pd/Gorlos-Phos-Catalyzed Cross-Coupling Between Two Different Aryl Chlorides in the Presence of B_2Pin_2

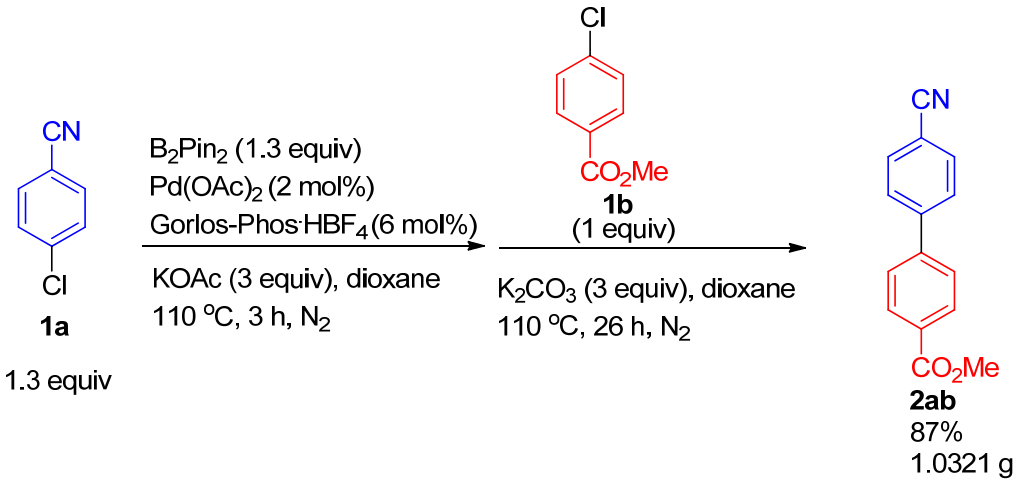
1. Synthesis of methyl 4-(methoxycarbonyl)-4'-cyano-1,1'-biphenyl 2ab (dxy-2-059)



Typical Procedure I: To a flame-dried Schlenk tube were added $Pd(OAc)_2$ (0.0046 g, 0.02 mmol), Gorlos-Phos·HBF_4 (0.0285 g, 0.06 mmol), B_2Pin_2 (0.3296 g, 1.3 mmol), KOAc (0.2952 g, 3 mmol), and **1a** (0.1786 g, 1.3 mmol)/dioxane (2 mL) sequentially under N_2 atmosphere. The reaction mixture was stirred at 110 °C for 3 h until **1a** was comleptely comsumed as monitored by TLC. The Schlenk tube was then lifted from the oil bath and followed by the quick addition of K_2CO_3 (0.4170 g, 3 mmol) and **1b** (0.1702 g, 1 mmol)/dioxane (2 mL) under N_2 atmosphere. The resulting mixture was heated in an oil bath preheated at 110 °C with stirring. After 24 h, the reaction was complete as monitored by TLC. The resulting mixture was cooled to room temperature, diluted with ethyl acetate (10 mL), and filtered through a short column of silica gel (eluent: ethyl acetate (3 × 10 mL)). After evaporation of the solvent, the crude residual was purified by chromatography (eluent: petroleum ether (60 °C ~ 90 °C)/ethyl acetate/DCM = 20/1/3 (720 mL)) on silica gel to afford **2ab**[2]

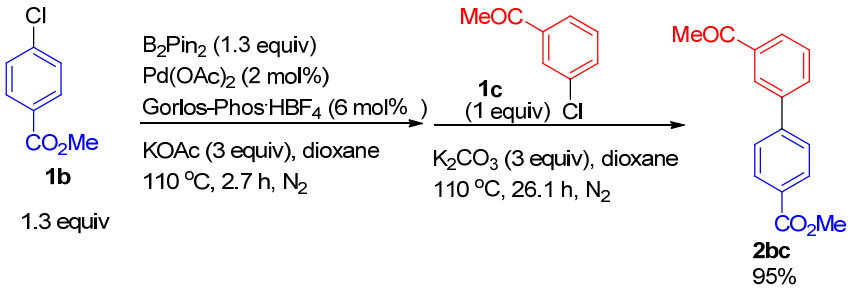
(0.2174 g, 92%): solid; m.p. 143.5-143.7 °C (*n*-hexane/ethyl acetate) (lit.[2] 142-144 °C (diisopropyl ether)); ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.15 (d, *J* = 8.4 Hz, 2H, ArH), 7.84-7.63 (m, 6H, ArH), 3.96 (s, 3H, OCH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.6, 144.4, 143.4, 132.7, 130.3, 130.2, 127.9, 127.2, 118.7, 111.8, 52.3; IR (KBr) ν (cm^{-1}) 3045, 2959, 2926, 2849, 2226, 1727, 1718, 1606, 1432, 1391, 1279, 1203, 1182, 1104, 1004; MS (70 ev, EI) m/z (%) 238 (M^+ + 1, 8.61), 237 (M^+, 50.86), 206 (100).

Gram scale:



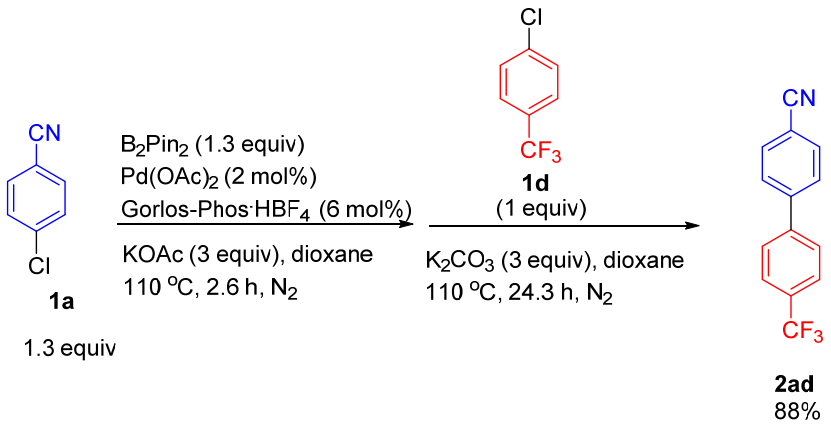
Following **Typical Procedure I**, the reaction of $Pd(OAc)_2$ (0.0225 g, 0.1 mmol), Gorlos-Phos·HBF_4 (0.1438 g, 0.3 mmol), B_2Pin_2 (1.6507 g, 6.5 mmol), KOAc (1.4727 g, 15 mmol), **1a** (0.8940 g, 6.5 mmol)/dioxane (10 mL) for the first step, K_2CO_3 (2.0734 g, 15 mmol) and **1b** (0.8527 g, 5mmol)/dioxane (10 mL) for the second step, afforded **2ab** (1.0321 g, 87%) (eluent: petroleum ether (60 °C ~ 90 °C)/ethyl acetate/DCM = 20/1/3 (1440 mL): solid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.15 (d, *J* = 8.4 Hz, 2H, ArH), 7.90-7.60 (m, 6H, ArH), 3.96 (s, 3H, OCH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.6, 144.4, 143.4, 132.7, 130.3, 130.1, 127.9, 127.2, 118.6, 111.8. 52.3.

2. Synthesis of 4-(methoxycarbonyl)-3'-acetyl-1,1'-biphenyl 2bc. (dxy-1-116)



Following **Typical Procedure I**, the reaction of $Pd(OAc)_2$ (0.0043 g, 0.02 mmol), Gorlos-Phos·HBF_4 (0.0285 g, 0.06 mmol), B_2Pin_2 (0.3305 g, 1.3 mmol), KOAc (0.2952 g, 3 mmol), **1b** (0.2267 g, 1.3 mmol)/dioxane (2 mL) for the first step, K_2CO_3 (0.4153 g, 3 mmol) and **1c** (0.1600 g, 1mmol)/dioxane (2 mL) for the second step, afforded **2bc**[3] (0.2431 g, 95%) (eluent: petroleum ether (60 °C ~ 90 °C)/ethyl acetate (30/1) (310 mL) to petroleum ether (60 °C ~ 90 °C)/ethyl acetate/DCM (300/10/1) (310 mL) to petroleum ether (60 °C ~ 90 °C)/ethyl acetate/DCM (75/5/1)) (320 mL): solid; m.p. 110.3-111.2 °C (*n*-hexane/ethyl acetate) (lit.[3] 109-110 °C); ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.21 (t, *J* = 1.7 Hz, 1H, ArH), 8.13 (d, *J* = 8.7 Hz, 2H, ArH), 8.02-7.94 (m, 1H, ArH), 7.86-7.78 (m, 1H, ArH), 7.69 (d, *J* = 8.4 Hz, 2H, ArH), 7.57 (t, *J* = 7.8 Hz, 1H, ArH), 3.95 (s, 3H, OCH_3), 2.67 (s, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 197.8, 166.8, 144.5, 140.5, 137.7, 131.8, 130.2, 129.4, 129.2, 128.0, 127.1, 126.9, 52.2, 26.7; IR (KBr) ν (cm^{-1}) 3039, 3000, 2959, 1722, 1681, 1608, 1581, 1429, 1403, 1367, 1294, 1247, 1189, 1113, 1019; MS (70 ev, EI) m/z (%) 255 (M^+ + 1 , 10.79), 254 (M^+, 60.28), 239 (100).

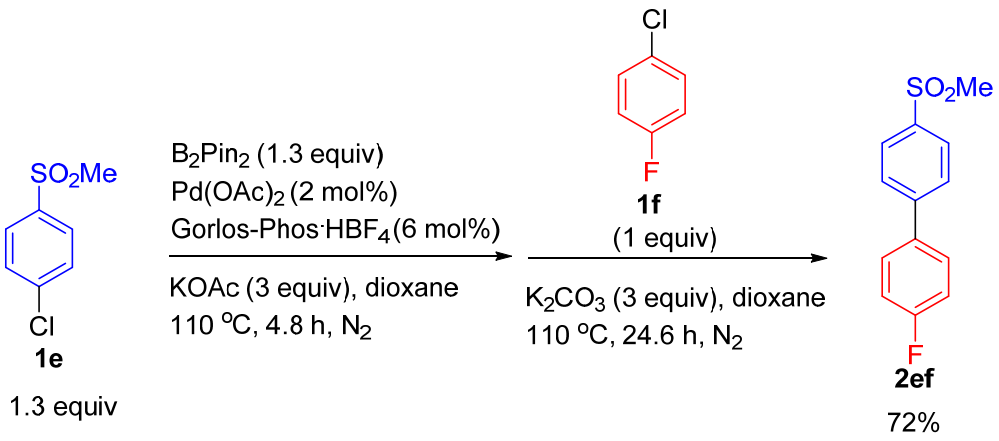
3. Synthesis of 4-(trifluoromethyl)-4’-cyano-1,1’-biphenyl 2ad (dxy-1-132)



Following **Typical Procedure I**, the reaction of $Pd(OAc)_2$ (0.0044 g, 0.02 mmol), Gorlos-Phos·HBF_4 (0.0289 g, 0.06 mmol), B_2Pin_2 (0.3303 g, 1.3 mmol), KOAc (0.2939 g, 3 mmol), **1a** (0.1800 g, 1.3 mmol)/dioxane (2 mL) for the first step, K_2CO_3 (0.4153 g, 3 mmol) and **1d** (0.1840 g, 1mmol)/dioxane (2 mL) for the second step, afforded **2ad**[4] (0.2168 g, 88%) (eluent: petroleum ether (60 °C ~ 90 °C)/ethyl acetate = 15/1): solid; m. p. 130.6-130.9 °C (*n*-hexane); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.81-7.66 (m, 8H, ArH); ^{13}C NMR (75 MHz, $CDCl_3$) δ 144.1, 142.7 (d, J = 1.4 Hz), 132.8, 130.7 (q, J = 32.4 Hz), 128.0, 127.6, 126.1 (q, J = 3.7 Hz), 124.0 (q, J = 270.9 Hz), 118.6, 112.0; ^{19}F NMR (282 MHz, $CDCl_3$) δ -63.1; IR (KBr) ν (cm^{-1}) 2230, 1617, 1606, 1498, 1394, 1328, 1163, 1125, 1071, 1022, 1007; MS (70 ev, EI) m/z (%) 248 (M^+ + 1, 15.43), 247 (M^+, 100).

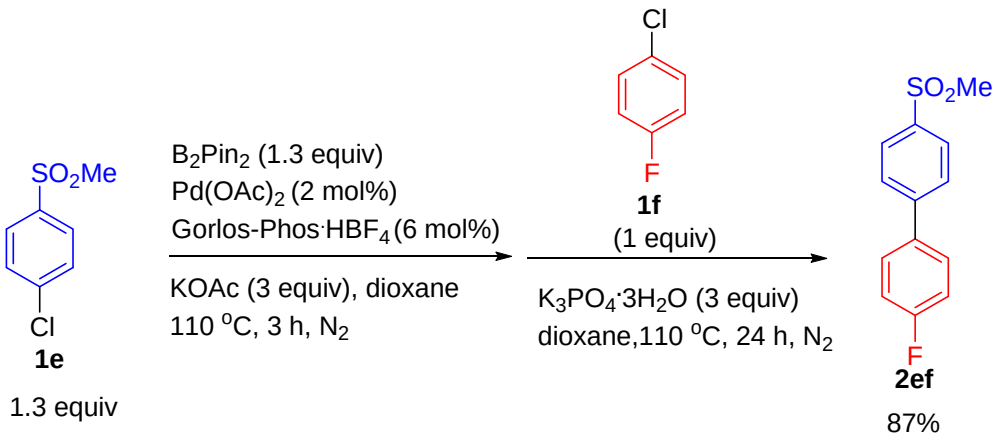
4. Synthesis of 4'-fluoro-4-(methylsulfonyl)-1,1'-biphenyl 2ef.

(1) The reaction with K_2CO_3 being used in the second step (Dxy-1-143)



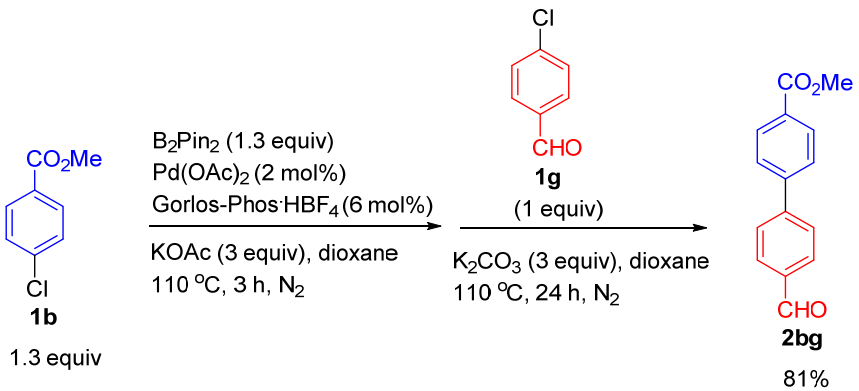
Following **Typical Procedure I**, the reaction of $Pd(OAc)_2$ (0.0046 g, 0.02 mmol), Gorlos-Phos·HBF_4 (0.0284 g, 0.06 mmol), B_2Pin_2 (0.3310 g, 1.3 mmol), KOAc (0.2943 g, 3 mmol), **1e** (0.2531 g, 1.3 mmol)/dioxane (2 mL) for the first step, K_2CO_3 (0.4152 g, 3 mmol), and **1f** (0.1352 g, 1 mmol)/dioxane (2 mL) for the second step, afforded impure **2ef** (0.2367 g) (eluent: petroleum ether (60 °C ~ 90 °C)/ethyl acetate/DCM = 5:1:2 (300 mL)), which was further purified by recrystallization to afford **2ef**[5] (0.1804 g, 72%): solid; m. p. 137.4-139.3 °C (*n*-hexane/DCM) (lit.[5] 144-146 °C); ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.01 (d, *J* = 8.1 Hz, 2H, ArH), 7.73 (d, *J* = 8.4 Hz, 2H, ArH), 7.64-7.53 (m, 2H, ArH), 7.18 (t, *J* = 8.6 Hz, 2H, ArH), 3.10 (s, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 163.2 (d, *J* = 246.8 Hz), 145.6, 139.1, 135.2 (d, *J* = 2.8 Hz), 129.1 (d, *J* = 8.3 Hz), 127.9, 127.8, 116.2 (d, *J* = 21.4 Hz), 44.6; ^{19}F NMR (282 MHz, $CDCl_3$) δ –113.7; IR (KBr) ν (cm^{-1}) 3059, 3010, 2927, 1595, 1519, 1488, 1419, 1389, 1375, 1299, 1265, 1246, 1196, 1180, 1143, 1094, 1076; MS (70 ev, EI) m/z (%) 251 (M^+ + 1, 16.21), 250 (M^+, 100).

(2) The reaction with $K_3PO_4·3H_2O$ being used in the second step (Dxy-2-062**)**



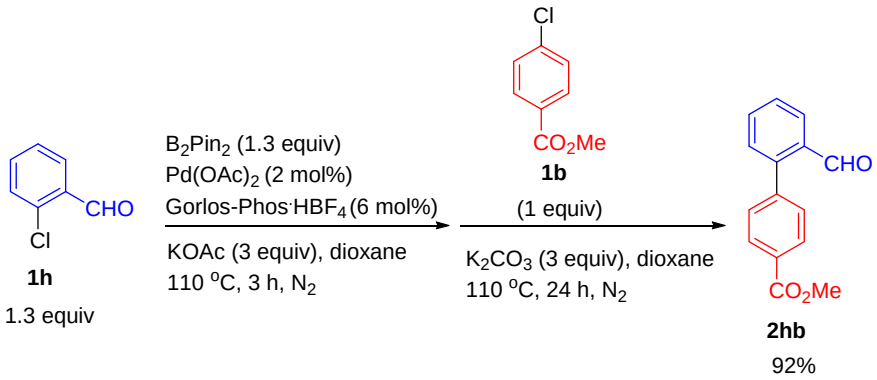
Following **Typical Procedure I**, the reaction of $Pd(OAc)_2$ (0.0047 g, 0.02 mmol), Gorlos-Phos·HBF_4 (0.0285 g, 0.06 mmol), B_2Pin_2 (0.3305 g, 1.3 mmol), KOAc (0.2950 g, 3 mmol), **1e** (0.2480 g, 1.3 mmol)/dioxane (2 mL) for the first step, $K_3PO_4 \cdot 3H_2O$ (0.7981 g, 3 mmol), and **1f** (0.1300 g, 1 mmol)/dioxane (2 mL) for the second step, afforded **2ef**[5] (0.2179 g, 87%) (eluent: petroleum ether (60 °C ~ 90 °C)/ethyl acetate/DCM = 20:1:3 (480 mL) to 12:1:3 (640 mL)): ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.01 (d, J = 8.1 Hz, 2H, ArH), 7.73 (d, J = 8.4 Hz, 2H, ArH), 7.63-7.53 (m, 2H, ArH), 7.19 (t, J = 8.6 Hz, 2H, ArH), 3.11 (s, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 163.1 (d, J = 246.8 Hz), 145.5, 139.0, 135.1 (d, J = 2.8 Hz), 129.1 (d, J = 8.3 Hz), 127.9, 127.7, 116.0 (d, J = 21.4 Hz), 44. 5; ^{19}F NMR (282 MHz, $CDCl_3$) δ –113.6.

5. Synthesis of 4'-formyl-4-(methoxycarbonyl)-1,1'-biphenyl 2bg (dxy-2-007)



Following **Typical Procedure I**, the reaction of $Pd(OAc)_2$ (0.0046 g, 0.02 mmol), Gorlos-Phos·HBF_4 (0.0290 g, 0.06 mmol), B_2Pin_2 (0.3304 g, 1.3 mmol), KOAc (0.2945 g, 3 mmol), **1b** (0.2268 g, 1.3 mmol)/dioxane (2 mL) for the first step, K_2CO_3 (0.4143 g, 3 mmol), and **1g** (0.1406 g, 1 mmol)/dioxane (2 mL) for the second step, afforded **2bg**[6] (0.1944 g, 81%) (eluent: petroleum ether (60 °C ~ 90 °C)/ ethyl acetate /DCM = 20:1:1 (330 mL) to 16:1:1 (380 mL) to 10:1:1 (240 mL)): solid; m. p. 116.1-119.0°C (*n*-hexane/DCM); ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.08 (s, 1H, CHO), 8.14 (d, *J* = 8.1 Hz, 2H, ArH), 7.98 (d, *J* = 8.1 Hz, 2H, ArH), 7.78 (d, *J* = 8.1 Hz, 2H, ArH), 7.70 (d, *J* = 8.1 Hz, 2H, ArH), 3.95 (s, 3H, OCH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 191.7, 166.7, 145.8, 144.0, 135.7, 130.3, 130.2, 129.9, 127.9, 127.3, 52.2; IR (KBr) ν (cm^{-1}) 2959, 2839, 2740, 1735, 1724, 1685, 1605, 1577, 1558, 1431, 1392, 1282, 1219, 1171, 1102, 1006; MS (70 ev, EI) m/z (%) 241 (M^+ + 1, 13.31), 240 (M^+, 84.46), 209 (100).

6. Synthesis of 2'-formyl-4-(methoxycarbonyl)-1,1'-biphenyl 2hb (dxy-2-028)

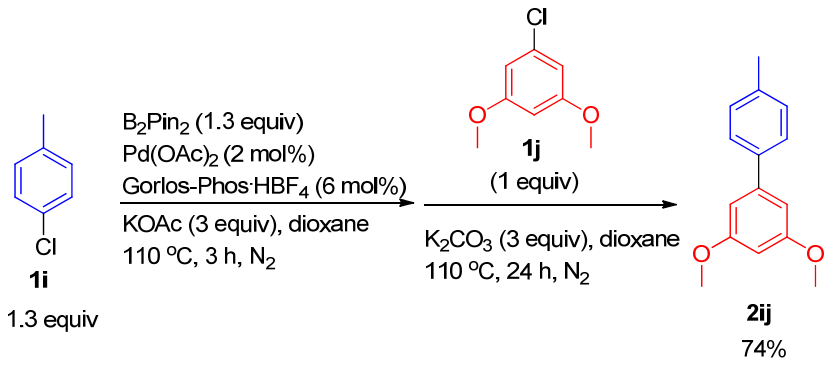


Following **Typical Procedure I**, the reaction of $Pd(OAc)_2$ (0.0043 g, 0.02 mmol), Gorlos-Phos·HBF_4 (0.0286 g, 0.06 mmol), B_2Pin_2 (0.3299 g, 1.3 mmol), KOAc (0.2947 g, 3 mmol), **1h** (0.1880 g, 1.3 mmol)/dioxane (2 mL) for the first step, K_2CO_3

(0.4147 g, 3 mmol), and **1b** (0.1736 g, 1 mmol)/dioxane (2 mL) for the second step, afforded **2hb**[7] (0.2196 g, 92%) (eluent: petroleum ether (60 °C ~ 90 °C)/ethyl acetate/DCM = 35:1:1 (750 mL)): solid; m. p. 66.5-68.9 °C (*n*-hexane/DCM); ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.96 (s, 1H, CHO), 8.15 (d, J = 8.1 Hz, 2H, ArH), 8.05 (dd, J_1 = 8.0 Hz, J_2 = 1.4 Hz, 1H, ArH), 7.67 (td, J_1 = 7.5 Hz, J_2 = 1.5 Hz, 1H, ArH), 7.46 (t, J = 6.5 Hz, 4H, ArH), 3.97 (s, 3H, OCH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 191.7, 166.6, 144.6, 142.4, 133.65, 133.62, 130.6, 130.0, 129.8, 129.6, 128.4, 127.9, 52.2; IR (KBr) ν (cm^{-1}) 2956, 2859, 2757, 1715, 1694, 1654, 1605, 1593, 1560, 1474, 1431, 1401, 1320, 1283, 1187, 1106, 1021, 1004; MS (70 ev, EI) m/z (%) 241 (M^+ + 1, 5.06), 240 (M^+, 38.73), 152 (100).

7. Synthesis of 3,5-dimethoxy-4'-methyl-1,1'-biphenyl 2ij. (dxy-1-182, dxy-2-035)

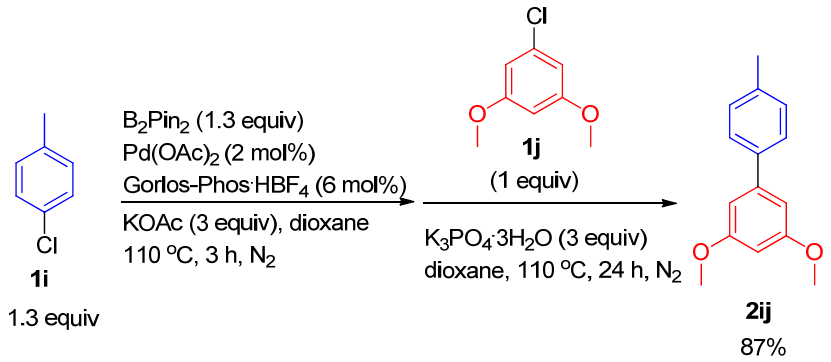
(1) The reaction with K_2CO_3 being used in the second step



Following **Typical Procedure I**, the reaction of $Pd(OAc)_2$ (0.0045 g, 0.02 mmol), Gorlos-Phos·HBF_4 (0.0285 g, 0.06 mmol), B_2Pin_2 (0.3301 g, 1.3 mmol), KOAc (0.2944 g, 3 mmol), **1i** (0.1650 g, 1.3 mmol)/dioxane (2 mL) for the first step, K_2CO_3 (0.4144 g, 3 mmol), and **1j** (0.1757 g, 1 mmol)/dioxane (2 mL) for the second step, afforded **2ij**[8] (0.1682 g, 74%) (petroleum ether (60 °C ~ 90 °C)/ethyl ether = 80:1 (240

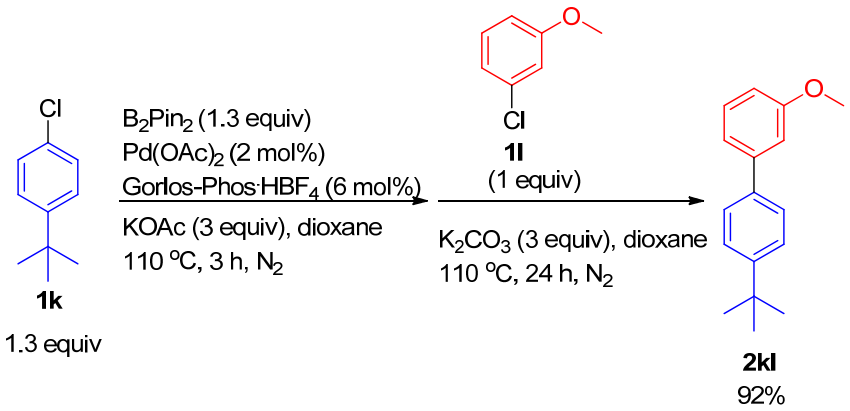
mL) to 60:1 (240 mL)): solid; m. p. 53.7-55.7 (*n*-hexane/DCM) (lit.[8] 55-56 °C (hexane)); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.48 (d, *J* = 8.1 Hz, 2H, ArH), 7.23 (d, *J* = 6.6 Hz, 2H, ArH), 6.72 (d, *J* = 2.4 Hz, 2H, ArH), 6.45 (t, *J* = 2.3 Hz, 1H, ArH), 3.84 (s, 6H, 2×OCH_3), 2.39 (s, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 161.0, 143.4, 138.3, 137.3, 129.4, 127.0, 105.2, 99.0, 55.3, 21.0; IR (KBr) ν (cm^{-1}) 3015, 2981, 2955, 2934, 2835, 1592, 1568, 1516, 1453, 1424, 1399, 1352, 1323, 1313, 1220, 1205, 1151, 1114, 1070, 1060, 1034; MS (70 ev, EI) m/z (%) 229 (M^+ + 1, 18.50), 228 (M^+, 100).

(2) The reaction with $K_3PO_4 \cdot 3H_2O$ being used in the second step



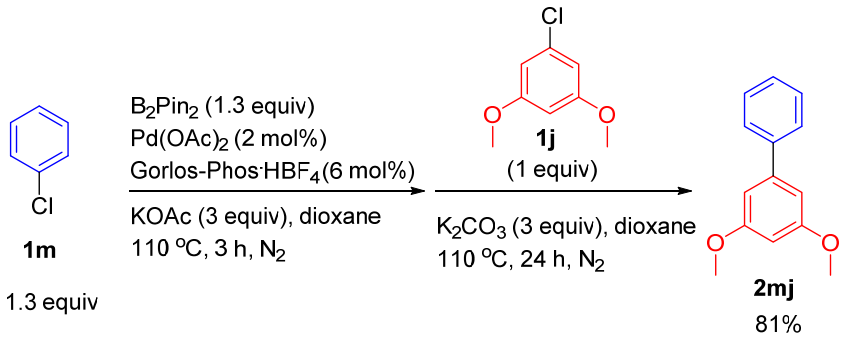
Following **Typical Procedure I**, the reaction of $Pd(OAc)_2$ (0.0045 g, 0.02 mmol), Gorlos-Phos·HBF_4 (0.0283 g, 0.06 mmol), B_2Pin_2 (0.3312 g, 1.3 mmol), KOAc (0.2949 g, 3 mmol), **1i** (0.1654 g, 1.3 mmol)/dioxane (2 mL) for the first step, $K_3PO_4 \cdot 3H_2O$ (0.7984 g, 3 mmol), and **1j** (0.1761 g, 1 mmol)/dioxane (2 mL) for the second step, afforded **2ij**[8] (0.1981 g, 87%) (petroleum ether (60 °C ~ 90 °C)/ ethyl acetate = 45:1 (300 mL)): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.47 (d, *J* = 8.1 Hz, 2H, ArH), 7.23 (d, *J* = 7.8 Hz, 2H, ArH), 6.72 (d, *J* = 2.4 Hz, 2H, ArH), 6.45 (t, *J* = 2.3 Hz, 1H, ArH), 3.84 (s, 6H, 2 × OCH_3), 2.39 (s, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 161.0, 143.3, 138.2, 137.3, 129.4, 127.0, 105.2, 99.0, 55.3, 21.0.

8. Synthesis of 4'-*tert*-Butyl-3-methoxy-1,1'-biphenyl 2kl. (dxy-2-016)



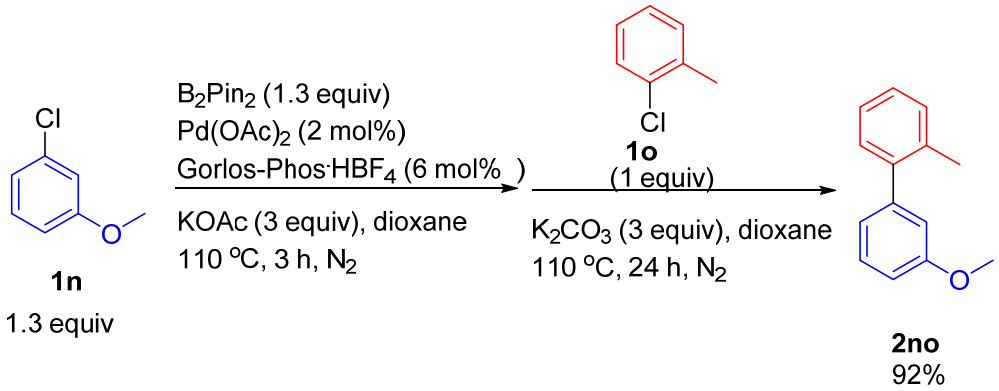
Following **Typical Procedure I**, the reaction of $Pd(OAc)_2$ (0.0045 g, 0.02 mmol), Gorlos-Phos·HBF_4 (0.0288 g, 0.06 mmol), B_2Pin_2 (0.3305 g, 1.3 mmol), KOAc (0.2943 g, 3 mmol), **1k** (0.2253 g, 1.3 mmol)/dioxane (2 mL) for the first step, K_2CO_3 (0.4143 g, 3 mmol), and **1l** (0.1451 g, 1 mmol)/dioxane (2 mL) for the second step, afforded **2kl**[9] (0.2253 g, 92%, purity = 98%) (petroleum ether (60 °C ~ 90 °C)/ ethyl acetate = 200:1 (800 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.54 (d, J = 8.7 Hz, 2H, ArH), 7.47 (d, J = 8.7 Hz, 2H, ArH), 7.35 (t, J = 7.8 Hz, 1H, ArH), 7.21-7.11 (m, 2H, ArH), 6.88 (ddd, J_1 = 8.1 Hz, J_2 = 2.6 Hz, J_3 = 0.9 Hz, 1H, ArH), 3.86 (s, 3H, OCH_3), 1.36 (s, 9H, 3×CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.8, 150.4, 142.6, 138.1, 129.7, 126.8, 125.7, 119.5, 112.7, 112.3, 55.2, 34.5, 31.3; IR (neat) ν (cm^{-1}) 3028, 2962, 2904, 2867, 2830, 1600, 1584, 1560, 1517, 1481, 1463, 1397, 1362, 1296, 1269, 1222, 1213, 1178, 1111, 1054, 1033, 1010; MS (70 ev, EI) m/z (%) 241 (M^+ + 1, 7.05), 240 (M^+, 37.78), 225 (100).

9. Synthesis of 3,5-dimethoxy-1,1'-biphenyl 2mj (dxy-1-183)



Following **Typical Procedure I**, the reaction of $Pd(OAc)_2$ (0.0045 g, 0.02 mmol), Gorlos-Phos·HBF_4 (0.0285 g, 0.06 mmol), B_2Pin_2 (0.3305 g, 1.3 mmol), KOAc (0.2947 g, 3 mmol), **1m** (0.1465 g, 1.3 mmol)/dioxane (2 mL) for the first step, K_2CO_3 (0.4145 g, 3 mmol), and **1j** (0.1766 g, 1 mmol)/dioxane (2 mL) for the second step, afforded **2mj**[10] (0.1747 g, 81%) (eluent: petroleum ether (60 °C ~ 90 °C)/ether = 80:1 (400 mL): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.61-7.55 (m, 2H, ArH), 7.47-7.32 (m, 3H, ArH), 6.74 (d, J = 2.4 Hz, 2H, ArH), 6.47 (t, J = 2.3 Hz, 1H, ArH), 3.85 (s, 6H, 2×OCH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 161.0, 143.5, 141.2, 128.7, 127.5, 127.2, 105.4, 99.2, 55.4; IR (neat) ν (cm^{-1}) 3000, 2955, 2937, 2837, 1596, 1575, 1500, 1463, 1417, 1352, 1335, 1218, 1205, 1155, 1066, 1025; MS (70 ev, EI) m/z (%) 215 (M^++1, 16.48), 214 (M^+, 100).

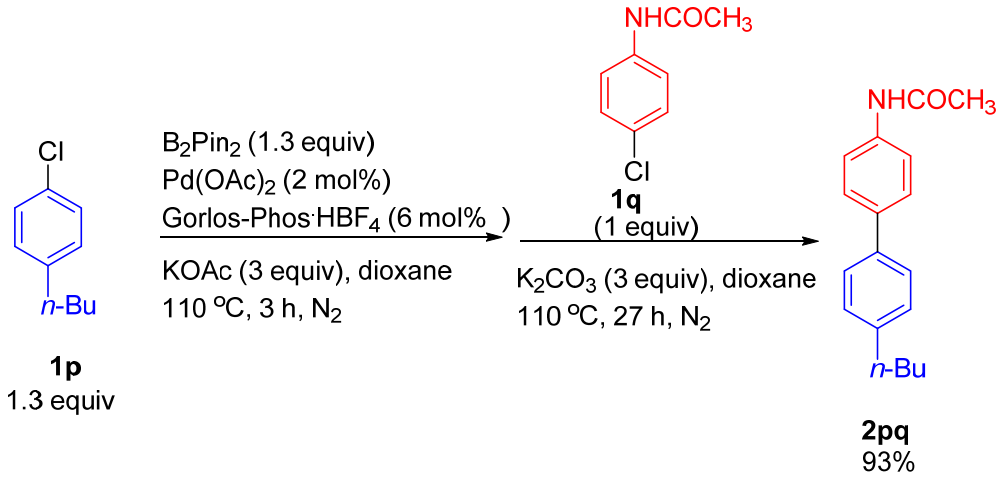
10. Synthesis of 3'-methoxy-2-methyl-1,1'-biphenyl 2no (dxy-3-037)



Following **Typical Procedure I**, the reaction of $Pd(OAc)_2$ (0.0046 g, 0.02 mmol),

Gorlos-Phos·HBF_4 (0.0285 g, 0.06 mmol), B_2Pin_2 (0.3303 g, 1.3 mmol), KOAc (0.2946 g, 3 mmol), **1n** (0.1892 g, 1.3 mmol)/dioxane (2 mL) for the first step, K_2CO_3 (0.4146 g, 3 mmol) and **1o** (0.1278 g, 1 mmol)/dioxane (2 mL) for the second step, afforded **2no**[11] (0.1844 g, 92%) (eluent: petroleum ether (60 °C ~ 90 °C) (500 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.31-7.14 (m, 5H, ArH), 6.94-6.80 (m, 3H, ArH), 3.76 (s, 3H, OCH_3), 2.26 (s, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.3, 143.3, 141.8, 135.2, 130.2, 129.6, 129.0, 127.2, 125.7, 121.6, 114.8, 112.2, 55.0, 20.3; IR (neat) ν (cm^{-1}) 3061, 3014, 2954, 2834, 1599, 1581, 1476, 1464, 1423, 1317, 1297, 1277, 1221, 1212, 1178, 1046, 1023; MS (70 ev, EI) m/z (%) 199 (M^+ + 1 , 29.96), 198 (M^+, 100).

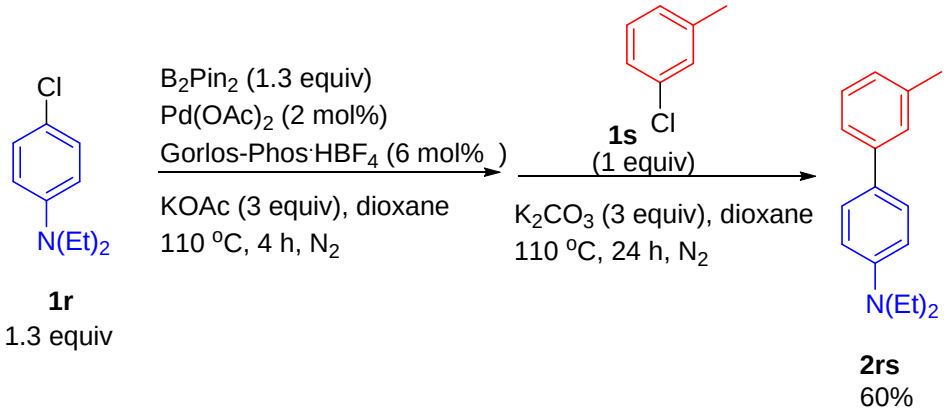
11. Synthesis of N-(4'-butyl-[1,1'-biphenyl]-4-yl)acetamide 2pq (dxy-3-046)



Following **Typical Procedure I**, the reaction of $Pd(OAc)_2$ (0.0043 g, 0.02 mmol), Gorlos-Phos·HBF_4 (0.0286 g, 0.06 mmol), B_2Pin_2 (0.3304 g, 1.3 mmol), KOAc (0.2943 g, 3 mmol), **1p** (0.2195 g, 1.3 mmol)/dioxane (2 mL) for the first step, K_2CO_3 (0.4148 g, 3 mmol) and **1q** (0.1693 g, 1 mmol)/dioxane (2 mL) for the second step, afforded **2pq** (0.2491 g, 93%) (eluent: petroleum ether (60 °C ~ 90 °C)/ethyl

acetate/DCM = 2/1/1 (400 mL)): solid; m.p. 172.9-173.4 °C (*n*-hexane/DCM); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.71 (br, 1H, NH); 7.60-7.42 (m, 6H, ArH), 7.22 (d, *J* = 8.1 Hz, 2H, ArH), 2.64 (t, *J* = 7.8 Hz, 2H, CH_2), 2.18 (s, 3H, $COCH_3$), 1.70-1.54 (m, 2H, CH_2), 1.46-1.30 (m, 2H, CH_2), 0.94 (t, *J* = 7.4 Hz, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.0, 141.8, 137.6, 137.0, 128.8, 127.2, 126.5, 120.5, 35.2, 33.5, 24.3, 22.3, 13.9; IR (KBr) ν (cm^{-1}) 3302, 2959, 2925, 2855, 1661, 1603, 1570, 1543, 1499, 1421, 1398, 1384, 1317; MS (70 ev, EI) m/z (%) 268 (M^+ + 1 , 15.88), 267 (M^+, 85.24), 182 (100); Anal. Calcd for $C_{18}H_{21}NO$: C 80.86, H 7.92, N 5.24; Found: C 80.83, H 7.67, N 5.14.

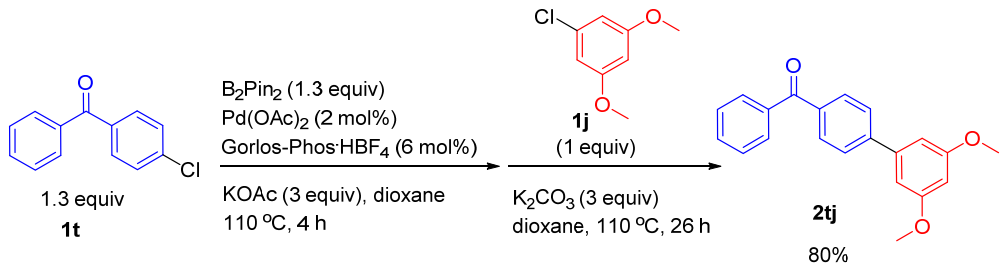
12. Synthesis of *N*,*N*-diethyl-3'-methyl-[1,1'-biphenyl]-4-amine 2rs (dxy-3-044)



Following **Typical Procedure I**, the reaction of $Pd(OAc)_2$ (0.0047 g, 0.02 mmol), Gorlos-Phos·HBF_4 (0.0289 g, 0.06 mmol), B_2Pin_2 (0.3305 g, 1.3 mmol), KOAc (0.2948 g, 3 mmol), **1r** (0.2390 g, 1.3 mmol)/dioxane (2 mL) for the first step, K_2CO_3 (0.4148 g, 3 mmol) and **1s** (0.1267 g, 1 mmol)/dioxane (2 mL) for the second step. The crude residue was first purified by chromatography on silica gel (eluent: petroleum ether/ DCM = 8/1 (720 mL)) to afford impure **2rs** (0.1758 g), which was

further purified by chromatography on silica gel (eluent: petroleum ether (60 °C ~ 90 °C) / ethyl acetate = 400/1 (800 mL)) to afford **2rs** (0.1515 g, purity = 95%, 60%): solid; m.p. 53.7-54.5 °C (*n*-hexane/DCM); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.46 (d, *J* = 9.0 Hz, 2H, ArH), 7.39-7.31 (m, 2H, ArH), 7.30-7.22 (m, 1H, ArH), 7.10-7.00 (m, 1H, ArH), 6.72 (d, *J* = 9.0 Hz, 2H, ArH), 3.36 (q, *J* = 7.1 Hz, 4H, CH_2 × 2), 2.39 (s, 3H, CH_3), 1.17 (t, *J* = 7.1 Hz, 6H, CH_3 × 2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 147.1, 141.3, 138.0, 128.5, 128.1, 127.9, 126.9, 126.5, 123.2, 111.9, 44.3, 21.6, 12.6; IR (neat) ν (cm^{-1}) 2970, 2928, 1612, 1524, 1485, 1398, 1374, 1356, 1267, 1200, 1155, 1092, 1077, 1010; MS (70 ev, EI) m/z (%) 240 (M^+ + 1 , 8.10), 239 (M^+, 47.95), 224 (100); HRMS calcd for $C_{17}H_{21}N$ (M^+): 239.1674, found: 239.1678.

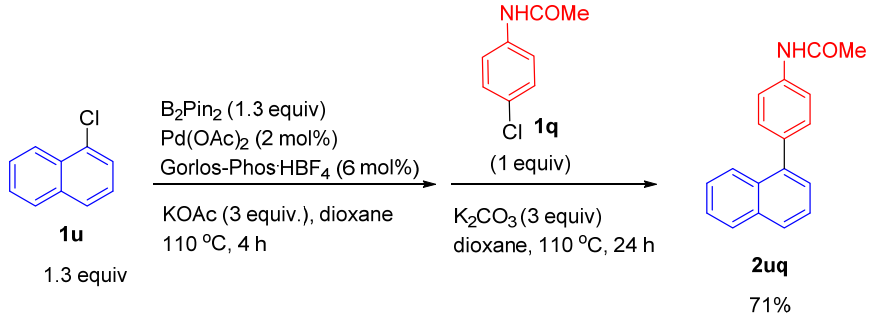
13. Synthesis of 3,5-dimethoxy-4'-benzoyl-1,1'-biphenyl 2tj. (dxy-1-175)



Following **Typical Procedure I**, the reaction of $Pd(OAc)_2$ (0.0047 g, 0.02 mmol), Gorlos-Phos·HBF_4 (0.0286 g, 0.06 mmol), B_2Pin_2 (0.3301 g, 1.3 mmol), KOAc (0.2948 g, 3 mmol), **1t** (0.2822 g, 1.3 mmol)/dioxane (2 mL) for the first step, K_2CO_3 (0.4147 g, 3 mmol), and **1j** (0.1761 g, 1 mmol)/dioxane (2 mL) for the second step afforded **2tj** (0.2536 g, 80%) (eluent: petroleum ether (60 °C ~ 90 °C)/ethyl acetate/DCM = 20:1:2 (550 mL)): solid; m. p. 107-108 °C (*n*-hexane/ethyl acetate); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.91-7.80 (m, 4H, ArH), 7.71-7.65 (m, 2H, ArH), 7.63-7.56 (m, 1H, ArH), 7.54-7.46 (m, 2H, ArH), 6.77 (d, *J* = 2.4 Hz, 2H,

ArH), 6.51 (t, J = 2.4 Hz, 1H, ArH), 3.85 (s, 6H, OCH_3 × 2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 196.2, 161.1, 145.1, 142.1, 137.6, 136.4, 132.3, 130.6, 129.9, 128.2, 127.0, 105.5, 99.9, 55.4; IR (KBr) ν (cm^{-1}) 3054, 3013, 2958, 2936, 2836, 1657, 1597, 1455, 1428, 1399, 1353, 1318, 1274, 1206, 1158, 1083, 1068, 1038; MS (70 eV, EI) m/z (%): 319 (M^+ + 1, 23.93), 318 (M^+, 100); Anal. Calcd for $C_{21}H_{18}O_3$: C 79.22, H 5.70. Found: C 79.21, H 5.74.

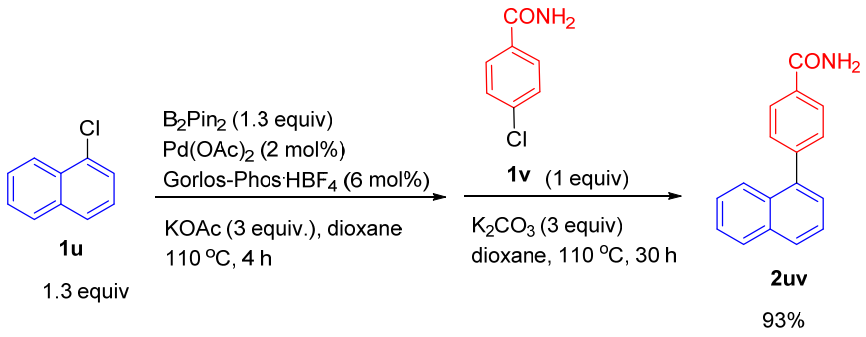
14. Synthesis of *N*-(4-(1-naphthalenyl)phenyl)acetamide 2uq (dxy-1-169)



Following **Typical Procedure I**, the reaction of $Pd(OAc)_2$ (0.0046 g, 0.02 mmol), Gorlos-Phos·HBF_4 (0.0285 g, 0.06 mmol), B_2Pin_2 (0.3299 g, 1.3 mmol), KOAc (0.2948 g, 3 mmol), **1u** (0.2111 g, 1.3 mmol)/dioxane (2 mL) for the first step, K_2CO_3 (0.4150 g, 3 mmol), and **1q** (0.1733 g, 1 mmol)/dioxane (2 mL) for the second step, afforded impure **2uq** (0.2619 g) (eluent: petroleum ether (60 °C ~ 90 °C)/ethyl acetate/DCM = 10:1:1 (240 mL) to 3:1:1 (300 mL) to 1:1:1 (300 mL)), which was further purified by recrystallization to afford pure **2uq**[12] (0.1861 g, 71%): solid; m. p. 205.4-206.4 °C (DCM/ethyl acetate) (lit.[11] 198-199 °C (petroleum/benzene)); ^1H NMR (300 MHz, $CDCl_3$) δ 7.90-7.80 (m, 3H, ArH), 7.63 (d, J = 8.4 Hz, 2H, ArH), 7.55-7.35 (m, 7H, ArH + NH), 2.24 (s, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 168.5, 139.5, 137.0, 136.7, 133.8, 131.6, 130.6, 128.3, 127.6, 126.9, 126.0, 125.9, 125.8,

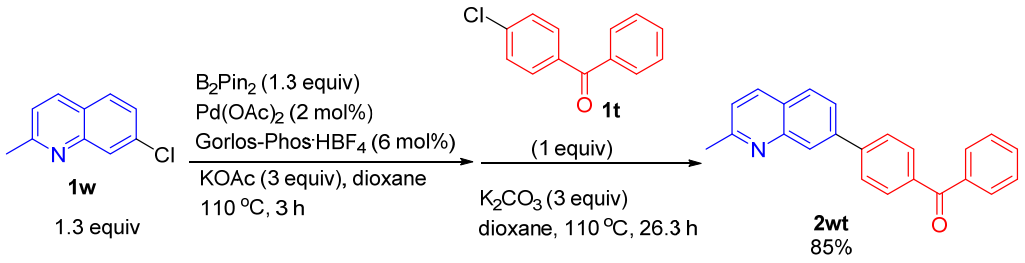
125.3, 119.8, 24.6; IR (KBr) ν (cm^{-1}) 3228, 3096, 3049, 1655, 1589, 1541, 1519, 1504. 1395, 1366, 1311, 1295, 1263, 1180. 1105, 1025; MS (70 ev, EI) m/z (%) 262 (M^+ + 1, 17.03), 261 (M^+, 82.98), 219 (100);

15. Synthesis of 4-(naphthalen-1-yl)benzamide 2uv (dxy-1-171)



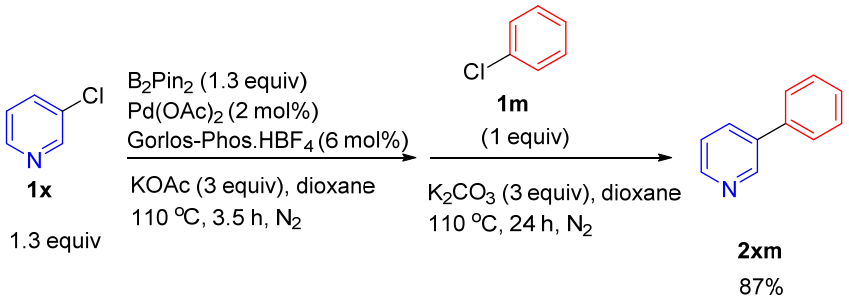
Following **Typical Procedure I**, the reaction of $Pd(OAc)_2$ (0.0048 g, 0.02 mmol), Gorlos-Phos·HBF_4 (0.0288 g, 0.06 mmol), B_2Pin_2 (0.3305 g, 1.3 mmol), KOAc (0.2947 g, 3 mmol), **1u** (0.2117 g, 1.3 mmol)/dioxane (2 mL) for the first step, K_2CO_3 (0.4143 g, 3 mmol), and **1v** (0.1589 g, 1 mmol)/dioxane (2 mL) for the second step, afforded **2uv**[13] (0.2293 g, 93%) (eluent: petroleum ether (60 °C ~ 90 °C) (300 mL) to ethyl acetate/DCM = 1:2 (300 mL)): solid; m. p. 181.7-182.2 °C (ethyl acetate); ^1H NMR (300 MHz, $CDCl_3$) δ 8.03-7.79 (m, 5H, ArH), 7.65-7.36 (m, 6H, ArH), 6.28 (brs, 2H, NH_2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.3, 144.7, 139.0, 133.8, 132.2, 131.3, 130.3, 128.4, 128.2, 127.4, 126.9, 126.3, 126.0, 125.6, 125.3; IR (KBr) ν (cm^{-1}) 3338, 3179, 1642, 1614, 1554, 1414, 1397, 1019; MS (70 ev, EI) m/z (%) 248 (M^+ + 1, 18.64), 247 (M^+, 100).

16. Synthesis of (4-(2-methylquinolin-7-yl)phenyl) phenyl ketone 2wt (dxy-1-150)



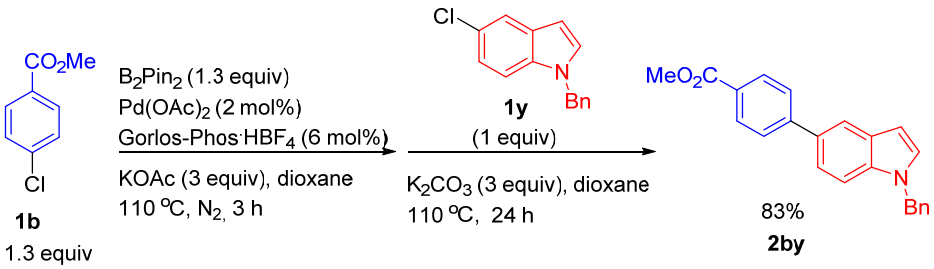
Following **Typical Procedure I**, the reaction of $Pd(OAc)_2$ (0.0046 g, 0.02 mmol), Gorlos-Phos·HBF_4 (0.0286 g, 0.06 mmol), B_2Pin_2 (0.3306 g, 1.3 mmol), KOAc (0.2951 g, 3 mmol), **1w** (0.2387 g, 1.3 mmol)/dioxane (2 mL) for the first step, K_2CO_3 (0.4154 g, 3 mmol), and **1t** (0.2182 g, 1 mmol)/dioxane (2 mL) for the second step, afforded **2wt** (0.2749 g, 85%) (eluent: petroleum ether (60 °C ~ 90 °C)/ethyl acetate/DCM = 10:1:1 (360 mL) to 6:1:1 (400 mL) to 5:1:1 (560 mL)): solid; m. p. 123.4-124.5 °C (hexane/DCM); ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.32 (s, 1H, ArH), 8.07 (d, *J* = 8.1 Hz, 1H, ArH), 8.00-7.73 (m, 8H, ArH), 7.61 (t, *J* = 7.4 Hz, 1H, ArH), 7.51 (t, *J* = 7.4 Hz, 2H, ArH), 7.31 (d, *J* = 8.7 Hz, 1H, ArH), 2.77 (s, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 196.2, 159.8, 148.0, 144.4, 140.7, 137.7, 136.6, 135.8, 132.4, 130.8, 129.9, 128.3, 128.2, 127.2, 126.9, 126.0, 124.9, 122.3, 25.4; IR (KBr) ν (cm^{-1}) 3065, 2919, 1658, 1620, 1602, 1573, 1521, 1493, 1444, 1401, 1362, 1315, 1286, 1277, 1216, 1201, 1154, 1124, 1070, 1025; MS (70 ev, EI) m/z (%) 324 (M^+ + 1, 32.97), 323 (M^+, 100); Anal. Calcd for $C_{23}H_{17}NO$: C 85.42, H 5.30, N 4.33. Found: C 85.40, H 5.44, N 4.12.

17. Synthesis of 3-phenylpyridine 2xm (dxy-2-009)



Following **Typical Procedure I**, the reaction of $Pd(OAc)_2$ (0.0044 g, 0.02 mmol), Gorlos-Phos·HBF_4 (0.0289 g, 0.06 mmol), B_2Pin_2 (0.3306 g, 1.3 mmol), KOAc (0.2944 g, 3 mmol), **1x** (0.1425 g, 1.3 mmol)/dioxane (2 mL) for the first step, K_2CO_3 (0.4148 g, 3 mmol), and **1m** (0.1130 g, 1 mmol)/dioxane (2 mL) for the second step, afforded **2xm**[14] (0.1356 g, 87%) (eluent: petroleum ether (60 °C ~ 90 °C)/ethyl acetate /DCM = 10:1:1 (240 mL) to 8:1:1 (300 mL) to 5:1:1 (420 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.85 (d, J = 2.1 Hz, 1H, ArH), 8.59 (dd, J_1 = 5.0 Hz, J_2 = 1.7 Hz, 1H, ArH), 7.90-7.80 (m, 1H, ArH), 7.62-7.52 (m, 2H, ArH), 7.51-7.29 (m, 4H, ArH); ^{13}C NMR (75 MHz, $CDCl_3$) δ 148.4, 148.3, 137.7, 136.5, 134.2, 129.0, 128.0, 127.0, 123.4; IR (neat) ν (cm^{-1}) 3406, 3055, 3031, 1582, 1566, 1473, 1450, 1408, 1336. 1274, 1235, 1188, 1126, 1107, 1074, 1023, 1006; MS (70 ev, EI) m/z (%) 156 (M^+ + 1, 11.64), 155 (M^+, 100).

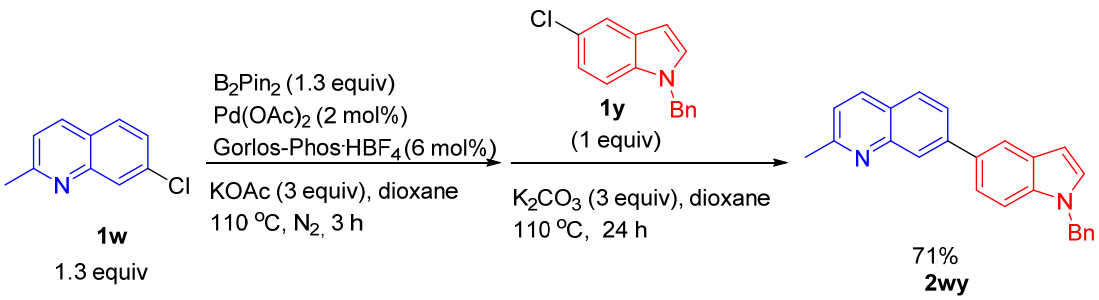
18. Synthesis of methyl 4-(1-benzyl-1*H*-indol-5-yl)benzoate 2by. (dxy-2-010)



Following **Typical Procedure I**, the reaction of $Pd(OAc)_2$ (0.0047 g, 0.02 mmol),

Gorlos-Phos·HBF_4 (0.0285 g, 0.06 mmol), B_2Pin_2 (0.3301 g, 1.3 mmol), KOAc (0.2950 g, 3 mmol), **1b** (0.2268 g, 1.3 mmol)/dioxane (2 mL) for the first step, K_2CO_3 (0.4150 g, 3 mmol), and **1y** (0.2418 g, 1 mmol)/dioxane (2 mL) for the second step, afforded **2by** (0.2832 g, 83%) (eluent: petroleum ether (60 °C ~ 90 °C)/diethyl ether/DCM = 40:1:12 (580 mL)): solid; m. p. 123.8-126.3 °C (hexane/DCM); ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.08 (d, *J* = 8.7 Hz, 2H, ArH), 7.90 (d, *J* = 1.2 Hz, 1H, ArH), 7.79 (d, *J* = 6.9 Hz, 2H, ArH), 7.43 (dd, J_1 = 8.7 Hz, J_2 = 1.7 Hz, 1H, ArH), 7.37-7.24 (m, 4H, ArH), 7.16 (d, *J* = 3.0 Hz, 1H, ArH), 7.13-7.04 (m, 2H, ArH), 6.61 (d, *J* = 2.4 Hz, 1H, ArH), 5.32 (s, 2H, CH_2), 3.92 (s, 3H, OCH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.2, 147.0, 137.2, 136.2, 131.7, 130.0, 129.2, 128.8, 127.8, 127.7, 127.0, 126.7, 121.4, 119.8, 110.1, 102.3, 52.0, 50.2; IR (KBr) ν (cm^{-1}) 3127, 3088, 3026, 2946, 2856, 2839, 1717, 1605, 1506, 1491, 1478, 1450, 1434, 1411, 1384, 1353, 1336, 1317, 1280, 1263, 1193, 1152, 1111, 1077, 1019; MS (70 ev, EI) m/z (%) 342 (M^+ + 1, 24.98), 341 (M^+, 100); Anal. Calcd for $C_{23}H_{19}NO_2$: C 80.92, H 5.61, N 4.10. Found: C 80.71, H 5.66, N 3.87.

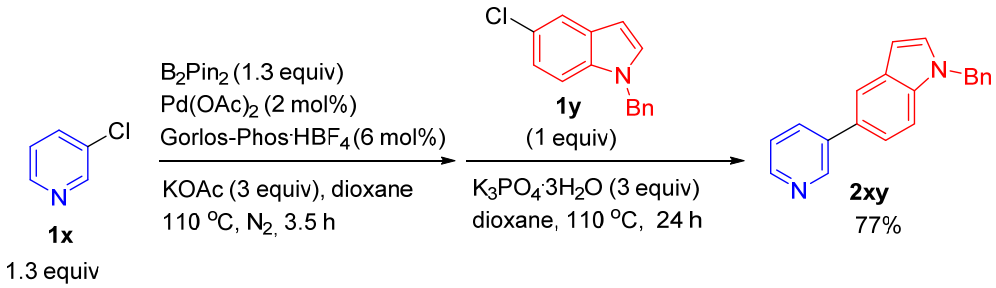
19. Synthesis of 7-(1-benzyl-1*H*-indol-5-yl)-2-methylquinoline 2wy (dxy-2-018)



Following **Typical Procedure I**, the reaction of $Pd(OAc)_2$ (0.0046 g, 0.02 mmol), Gorlos-Phos·HBF_4 (0.0283 g, 0.06 mmol), B_2Pin_2 (0.3304 g, 1.3 mmol), KOAc

(0.2950 g, 3 mmol), **1w** (0.2386 g, 1.3 mmol)/dioxane (2 mL) for the first step, K_2CO_3 (0.4152 g, 3 mmol), and **1y** (0.2422 g, 1 mmol)/dioxane (2 mL) for the second step, afforded **2wy** (0.2490 g, 71%) (eluent: petroleum ether (60 °C ~ 90 °C)/ethyl acetate/DCM = 10:1:2 (520 mL) to 9:1:2 (360 mL) to 8:1:2 (440 mL)): solid; m. p. 154.4-155.4 °C (hexane/DCM); ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.30 (s, 1H, ArH), 8.06-7.98 (m, 2H, ArH), 7.86-7.76 (m, 2H, ArH), 7.60 (dd, J_1 = 8.6 Hz, J_2 = 1.7 Hz, 1H, ArH), 7.42-7.08 (m, 8H, ArH), 6.63 (d, J = 3.3 Hz, 1H, ArH), 5.33 (s, 2H, CH_2), 2.75 (s, 3H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.2, 148.2, 143.4, 137.3, 136.0, 135.8, 132.1, 129.2, 129.1, 128.8, 127.62, 127.60, 126.7, 125.85, 125.80, 125.0, 121.6, 121.4, 119.9, 110.1, 102.2, 50.1, 25.4; IR (KBr) ν (cm^{-1}) 1616, 1600, 1512, 1495, 1482, 1452, 1439, 1413, 1390, 1371, 1355, 1345, 1305, 1287, 1184, 1169, 1126, 1079, 1055, 1027; MS (70 ev, EI) m/z (%) 349 (M^+ + 1, 15.47), 348 (M^+, 55.81), 91 (100); Anal. Calcd for $C_{25}H_{20}N_2$: C 86.17, H 5.79, N 8.04. Found: C 86.17, H 5.87, N 7.99.

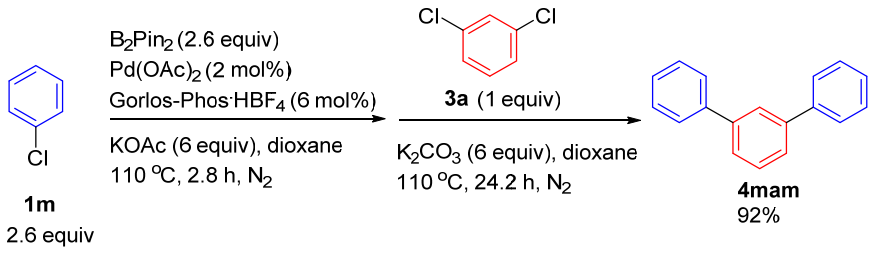
20. Synthesis of 1-benzyl-5-(3-pyridinyl)-1*H*-indole 2xy (dxy-2-056)



Following **Typical Procedure I**, the reaction of $Pd(OAc)_2$ (0.0043 g, 0.02 mmol), Gorlos-Phos·HBF_4 (0.0283 g, 0.06 mmol), B_2Pin_2 (0.3294 g, 1.3 mmol), KOAc (0.2955 g, 3 mmol), **1x** (0.1480 g, 1.3 mmol)/dioxane (2 mL) for the first step, $K_3PO_4 \cdot 3H_2O$ (0.7979 g, 3 mmol), and **1y** (0.2419 g, 1 mmol)/dioxane (2 mL) for the

second step, afforded **2xy** (0.2180 g, 77%) (eluent: petroleum ether (60 °C ~ 90 °C)/ethyl acetate/DCM = 6:1:1 (400 mL) to 4:1:1 (450 mL)): solid; m. p. 132.7-135.5 °C (hexane/DCM); ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.90 (s, 1H, ArH), 8.54 (d, J = 4.5 Hz, 1H, ArH), 7.90 (dt, J_1 = 8.1 Hz, J_2 = 2.0 Hz, 1H, ArH), 7.86 (t, J = 1.2 Hz, 1H, ArH), 7.43-7.23 (m, 6H, ArH), 7.19 (d, J = 3.0 Hz, 1H, ArH), 7.17-7.10 (m, 2H, ArH), 6.63 (d, J = 3.0 Hz, 1H, ArH), 5.35 (s, 2H, CH_2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 148.5, 147.4, 137.9, 137.2, 136.1, 134.4, 129.5, 129.33, 129.27, 128.8, 127.7, 126.7, 123.4, 121.2, 119.7, 110.3, 102.2, 50.2; IR (KBr) ν (cm^{-1}) 1620, 1584, 1575, 1508, 1498, 1467, 1454, 1439, 1413, 1391, 1350, 1332, 1310, 1290, 1270, 1261, 1201, 1184, 1177, 1075, 1042, 1025, 1014; MS (70 ev, EI) m/z (%) 284 (M^+, 37.41), 91(100); Anal. Calcd for $C_{20}H_{16}N_2$: C 84.48, H 5.67, N 9.85; Found: C 84.26, H 5.74, N 9.79.

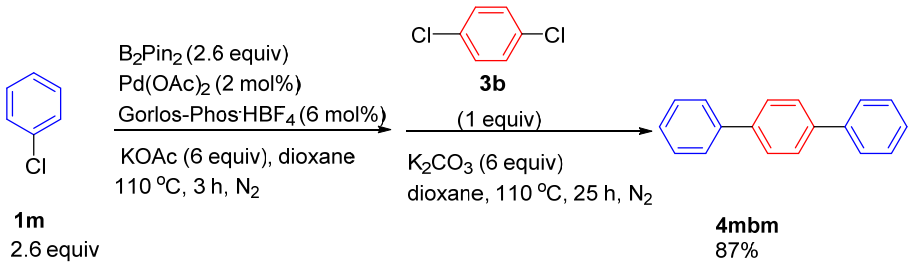
21. Synthesis of 1,3-diphenylbenzene 4mam (dxy-1-134)



Following **Typical Procedure I**, the reaction of $Pd(OAc)_2$ (0.0048 g, 0.02 mmol), Gorlos-Phos·HBF_4 (0.0286 g, 0.06 mmol), B_2Pin_2 (0.6596 g, 2.6 mmol), KOAc (0.5883 g, 6 mmol), **1m** (0.2934 g, 2.6 mmol)/dioxane (4 mL) for the first step, K_2CO_3 (0.8297 g, 6 mmol), and **3a** (0.1466 g, 1 mmol)/dioxane (4 mL) for the second step, afforded **4mam**[15] (0.2113 g, 92%) (eluent: petroleum ether (60 °C ~ 90 °C) (400 mL)): solid; m. p. 86.9-87.1 °C (*n*-hexane/diethyl ether) (lit[14]. 86-88 °C

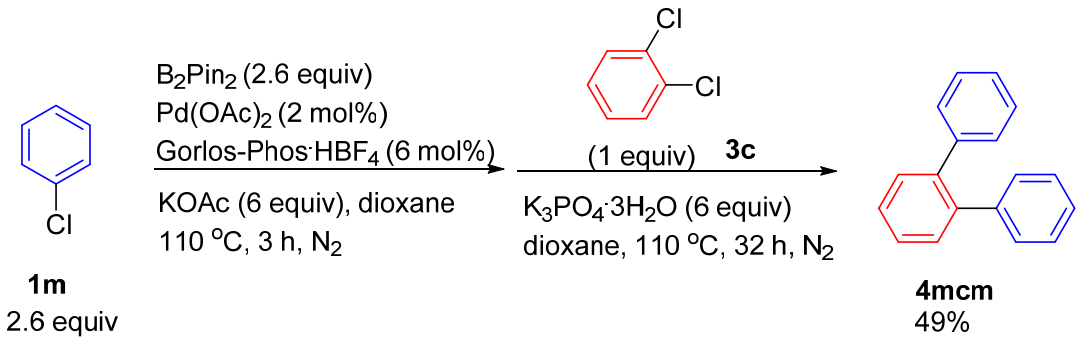
(*n*-hexane/ethyl acetate)); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.82-7.77 (m, 1H, ArH), 7.68-7.60 (m, 4H, ArH), 7.60-7.54 (m, 2H, ArH), 7.53-7.40 (m, 5H, ArH), 7.40-7.30 (m, 2H, ArH); ^{13}C NMR (75 MHz, $CDCl_3$) δ 141.8, 141.2, 129.2, 128.8, 127.4, 127.3, 126.2, 126.1; IR (KBr) ν (cm^{-1}) 3057, 3028, 1596, 1569, 1495, 1474, 1438, 1403; MS (70 ev, EI) m/z (%) 231 (M^+ + 1, 19.77), 230 (M^+, 100).

22. Synthesis of 1,4-diphenylbenzene 4mbm (dxy-2-065)



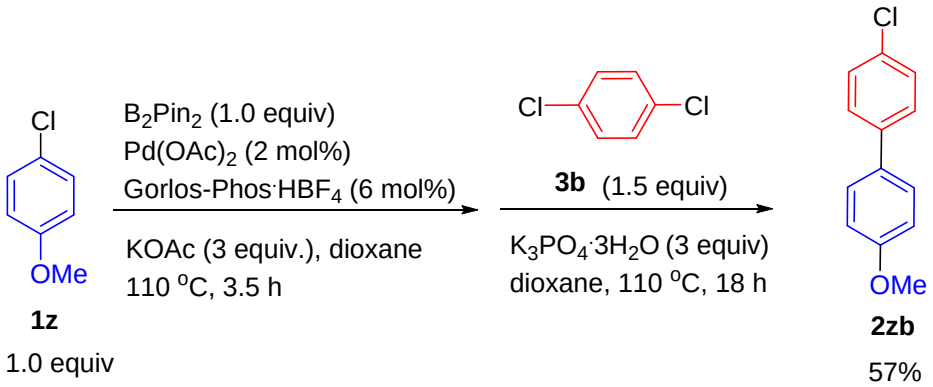
Following **Typical Procedure I**, the reaction of $Pd(OAc)_2$ (0.0046 g, 0.02 mmol), Gorlos-Phos·HBF_4 (0.0283 g, 0.06 mmol), B_2Pin_2 (0.6604 g, 2.6 mmol), KOAc (0.5890 g, 6 mmol), **1m** (0.2928 g, 2.6 mmol)/dioxane (4 mL) for the first step, K_2CO_3 (0.8399 g, 6 mmol), and **3b** (0.1474 g, 1 mmol)/dioxane (4 mL) for the second step, afforded **4mbm**[16] (0.2010 g, 87%) (eluent: petroleum ether (60 °C ~ 90 °C) to petroleum ether (60 °C ~ 90 °C)/DCM = 40:1): solid; m. p. 211.0-212.7 °C (*n*-hexane/DCM) (lit[15]. 210-212 °C (hexane)); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.70-7.61 (m, 8H, ArH), 7.50-7.42 (m, 4H, ArH), 7.40-7.32 (m, 2H, ArH); ^{13}C NMR (75 MHz, $CDCl_3$) δ 140.7, 140.1, 128.8, 127.5, 127.3, 127.0; IR (KBr) ν (cm^{-1}) 3060, 3034, 1595, 1576, 1481, 1455, 1404, 1340, 1259, 1192, 1169, 1133, 1075, 1028, 1004; MS (70 ev, EI) m/z (%) 231 ((M^+ + 1), 19.27), 230 (M^+, 100).

23. Synthesis of 1,2-diphenylbenzene 4mcm (dxy-2-057)



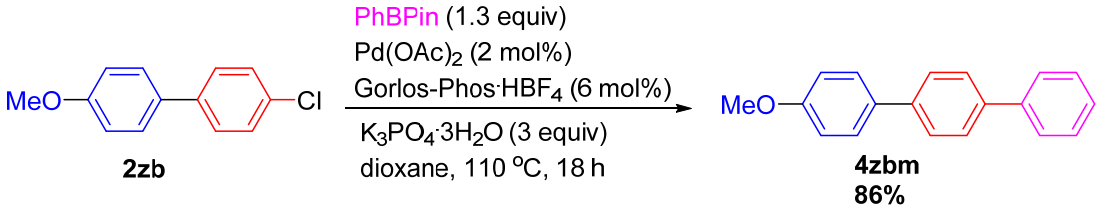
Following **Typical Procedure I**, the reaction of $Pd(OAc)_2$ (0.0047 g, 0.02 mmol), Gorlos-Phos·HBF_4 (0.0290 g, 0.06 mmol), B_2Pin_2 (0.6598 g, 2.6 mmol), KOAc (0.5875 g, 6 mmol), **1m** (0.2925 g, 2.6 mmol)/dioxane (4 mL) for the first step, $K_3PO_4·3H_2O$ (1.5975 g, 6 mmol), and **3c** (0.1478 g, 1 mmol)/dioxane (4 mL) for the second step, afforded **4mcm**[17] (0.1139 g, 49%) (petroleum ether (60 °C ~ 90 °C)(450 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.47-7.35 (m, 4H, ArH), 7.30-7.05 (m, 10H, ArH); ^{13}C NMR (75 MHz, $CDCl_3$) δ 141.5, 140.5, 130.6, 129.9, 127.8, 127.5, 126.4; IR (neat) ν (cm^{-1}) 3058, 3020, 1599, 1575, 1495, 1472, 1452, 1443, 1429, 1180, 1156, 1115, 1073, 1009; MS (70 ev, EI) m/z (%) 231 (M^+ + 1, 18.66), 230 (M^+, 100).

24. Synthesis of 4-methoxy-1,1':4',1''-terphenyl 4zbm (dxy-2-096, dxy-2-104)



Following Typical Procedure I, the reaction of $Pd(OAc)_2$ (0.0043 g, 0.02 mmol), Gorlos-Phos·HBF_4 (0.0290 g, 0.06 mmol), B_2Pin_2 (0.2540 g, 1.0 mmol), KOAc (0.2945 g, 3 mmol), **1z** (0.1430 g, 1.0 mmol)/dioxane (2 mL) for the first step,

$K_3PO_4 \cdot 3H_2O$ (0.7989 g, 3 mmol), and **3b** (0.2206 g, 1.5 mmol)/dioxane (2 mL) for the second step, afforded **2zb**[18] (0.1260 g, 57%) (eluent: petroleum ether (60 °C ~ 90 °C)): solid; 114.6-115.2 °C (*n*-hexane/DCM) (lit.[17] 113-114 °C (EtOH)); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.53-7.42 (m, 4H, ArH), 7.40-7.33 (m, 2H, ArH), 6.97 (d, J = 8.7 Hz, 2H, ArH), 3.84 (s, 3H, OCH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.3, 139.2, 132.6, 132.4, 128.8, 128.0, 127.9, 114.3, 55.3; IR (KBr) ν (cm^{-1}) 2963, 2933, 2840, 1607, 1524, 1486, 1397, 1308, 1291, 1264, 1201, 1180, 1133, 1102, 1038, 1014; MS (70 ev, EI) m/z (%) 220 (M^+(^{37}Cl), 31.70), 218 (M^+(^{35}Cl), 100).



To a flame-dried Schlenk tube were added $Pd(OAc)_2$ (0.0026 g, 0.011 mmol), Gorlos-Phos·HBF_4 (0.0153 g, 0.032 mmol), Ph-BPin (0.1402 g, 0.69 mmol)/dioxane (1 mL), $K_3PO_4 \cdot 3H_2O$ (0.4236 g, 1.59 mmol), and **2zb** (0.1151 g, 0.53 mmol)/dioxane (1 mL) sequentially under N_2 atmosphere. The resulting mixture was stirred at 110 °C in a preheated oil bath. After 18 h, the reaction was complete as monitored by TLC. The reaction mixture was cooled to room temperature and quenched with an aqueous solution of hydrochloric acid (3 M, 15 mL). The resulting mixture was extracted with DCM (20 mL × 3) and washed with an aqueous solution of $NaHCO_3$ (15 mL). The combined organic layer was dried over anhydrous Na_2SO_4. After filtration and evaporation, a white solid was obtained, which was then washed with petroleum ether (5 mL × 3) and dried under vacuum to afford **4zbm**[19] (0.1183 g, 86%): solid; m. p.

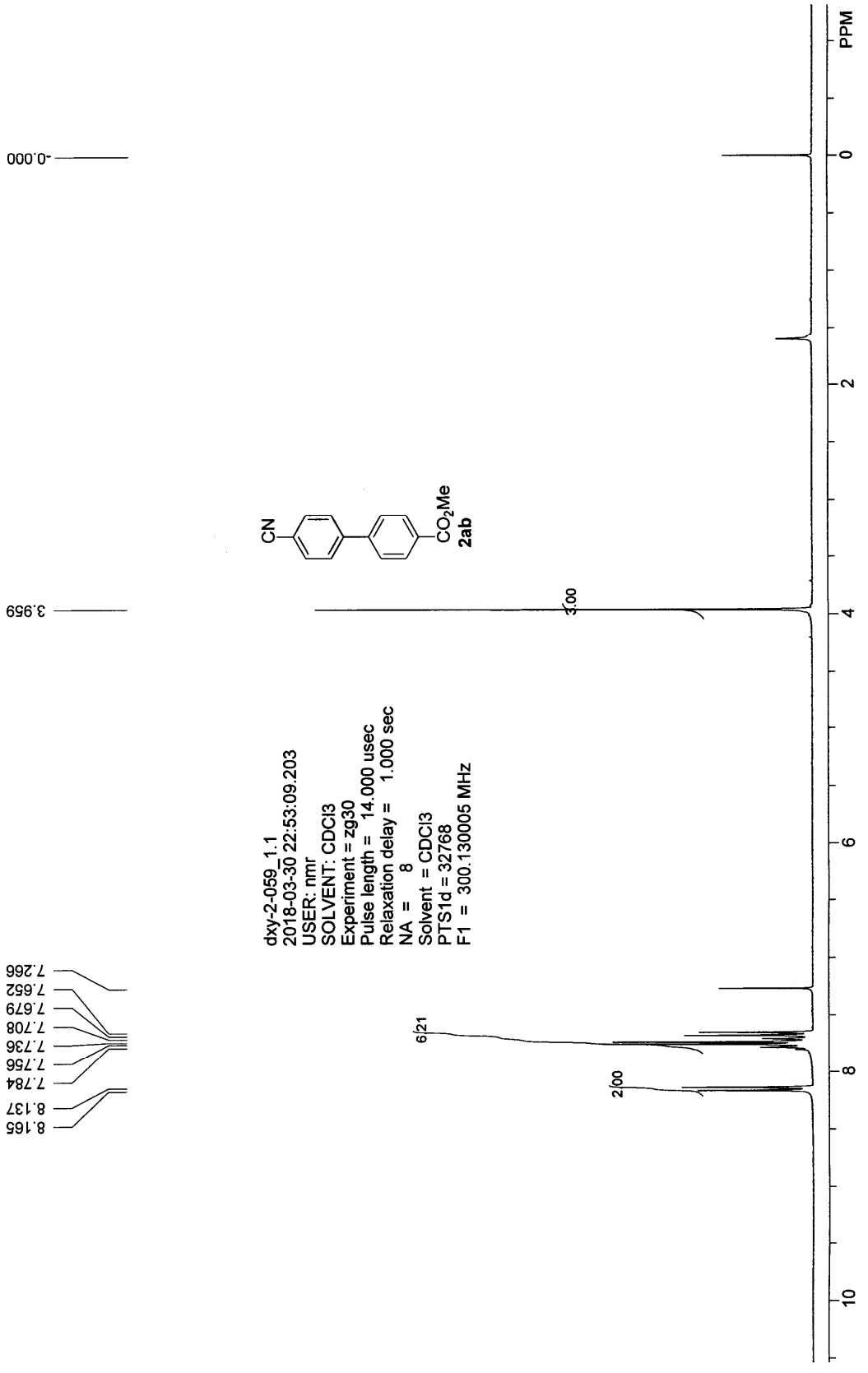
225.3-225.8 ^{o}C (DCM) (lit.[18] 224-225 ^{o}C (MeOH)); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.72-7.53 (m, 8H, ArH), 7.51-7.41 (m, 2H, ArH), 7.40-7.32 (m, 1H, ArH), 7.00 (d, *J* = 8.7 Hz, 2H, ArH), 3.87 (s, 3H, OCH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.2, 140.8, 139.7, 139.5, 133.2, 128.8, 128.1, 127.5, 127.2, 127.03, 126.99, 114.3, 55.4; IR (KBr) ν (cm^{-1}) 3056, 3034, 3002, 2961, 2937, 2836, 1607, 1582, 1534, 1508, 1485, 1466, 1449, 1440, 1402, 1287, 1255, 1219, 1179, 1141, 1117, 1043, 1030; MS (70 ev, EI) m/z (%) 261 (M^+ + 1, 21.36), 260 (M^+, 100).

Cytotoxicity Study

Human non-small cell lung cancer A549 cells were from ATCC (Manassas, VA) and cultured in RPMI 1640 supplemented with 10% FBS, 100 U/mL penicillin and 100 mg/mL streptomycin at 37 °C in humidified air containing 5% CO_2. The cells were seeded in 96-well plates at a density of 2000 cells/well and cultured overnight. Then the cells were treated with tested compounds at 10 μM for 72 hr and the sulforhodamine B (SRB) assay was used to measure the cell mass. Briefly, the cells were fixed with 10% trichloroacetic acid for 1 hr at 4°C. After washed with deionized water and air-dried, the cells were stained with 0.1% SRB dissolved in 1% acetic acid for 30 minutes and subsequently washed four times with 1% acetic acid to remove unbound dye. The plates were left to dry at room temperature and 100 μl of 10 mM TRIS base was added to solubilize the protein-bound SRB. The absorbance at 540 nm was measured with a microplate reader (SpectraMax M2, Molecular Devices). Cytotoxicity was determined by inhibition of cellular growth with the following formula: inhibitory rate (%) = (A_{540} of vehicle control − A_{540} of treated cells) / (A_{540} of vehicle control − A_{540} of blank control)*100.

Entry	References	Known compounds
1	B. Lü, P. Li, C. Fu, L. Xue, Z. Lin, and S. Ma, *Adv. Synth. Catal.*, 2011, **353**, 100.	**-**
2	D. I. Fletcher, C. R. Ganellin, A. Piergentili, P. M. Dunnb and D. H. Jenkinsonb, *Bioorg. Med. Chem.*, 2007, **15**, 5457.	**2ab**
3	J. P. Wolfe, R. A. Singer, B. H. Yang and S. L. Buchwald, *J. Am. Chem. Soc*., 1999, **121**, 9550.	**2bc**
4	N. Kataoka, Q. Shelby, J. P. Stambuli and J. F. Hartwig, *J. Org. Chem*., 2002, **67**, 5553.	**2ad**
5	B. Steiniger and F. R. Wuest, *J Label CompdRadiopharm*., 2006, **49**, 817.	**2ef**
6	T. Cornilleau, P. Hermange and E. Fouquet, *Chem. Commun.*, 2016, **52**, 10040.	**2bg**
7	J. Zhao, D. Yue, M. A. Campo and R. C. Larock, *J. Am. Chem. Soc.*, 2007, **129**, 5288.	**2hb**
8	V. Percec, G. M. Golding, J. Smidrkal and O. Weichold, *J. Org. Chem.*, 2004, **69**, 3447	**2ij**
9	S. E. Denmark, R. C. Smith, W. T. Chang and J. M. Muhuhi, *J. Am. Chem. Soc*., 2009, **131**, 3104.	**2kl**
10	G. A. Molander and L. Iannazzo, *J. Org. Chem.*, 2011, **76**, 9182.	**2mj**
11	H. Min, H. Miyamura and S. Kobayashi, *Chem. Lett*. 2016, **45**, 837.	**2no**
12	B. A. Marshall and W. A. Waters, *J. Chem. Soc.*, 1959, **0**, 381.	**2uq**
13	M. Larhed, G. Lindeberg and A. Hallberg, *Tetrahedron Lett.*, 1996, **37**, 8219.	**2uv**
14	L. Ackermann, C. J. Gschrei, A. Althammer and M. Riederer, *Chem. Commun.*, 2006, 1419.	**2xm**

15	M. Chang, C. Chan, S. Lin and M. Wu, *Tetrahedron*, 2013, **69**, 9616.	**4mam**
16	O. V. Petrova, A, I. Mikhaleva, L. N. Sobenina and B. A. Trofimov, *Russ. J. Org. Chem*., 2010, **46**, 452.	**4mbm**
17	C. Diebold, J. Becht, J. Lu, P. Toy and C. Drian, *Eur. J. Org. Chem.*, 2012, 893.	**4mcm**
18	S. E. Denmark, R. C. Smith and S. A. Tymonko, *Tetrahedron*, 2007, **63**, 5730.	**2zb**
19	C. Cho, I. Kim and K. Park, *Tetrahedron,* 2004, **60**, 4589.	**4zbm**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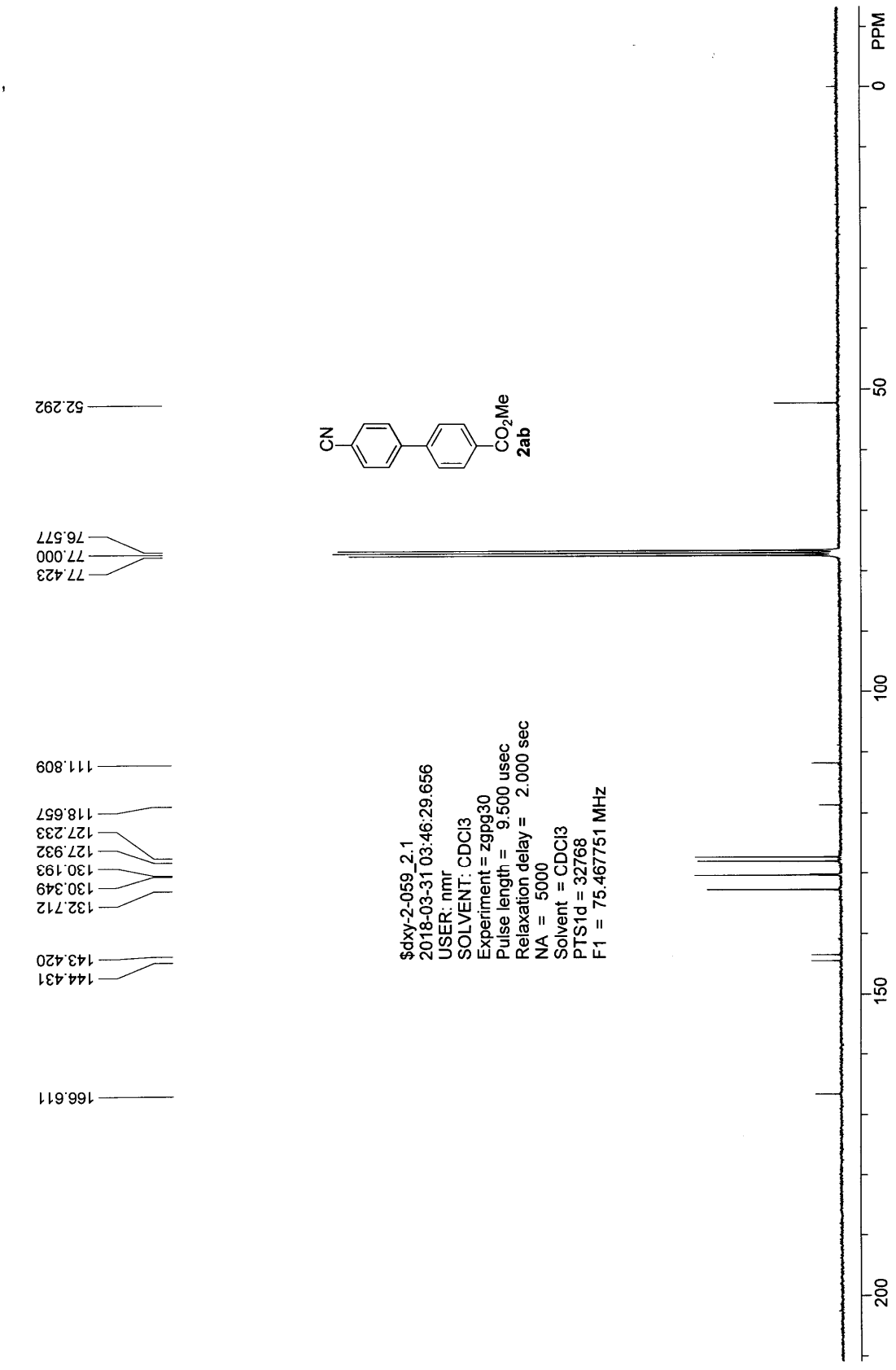
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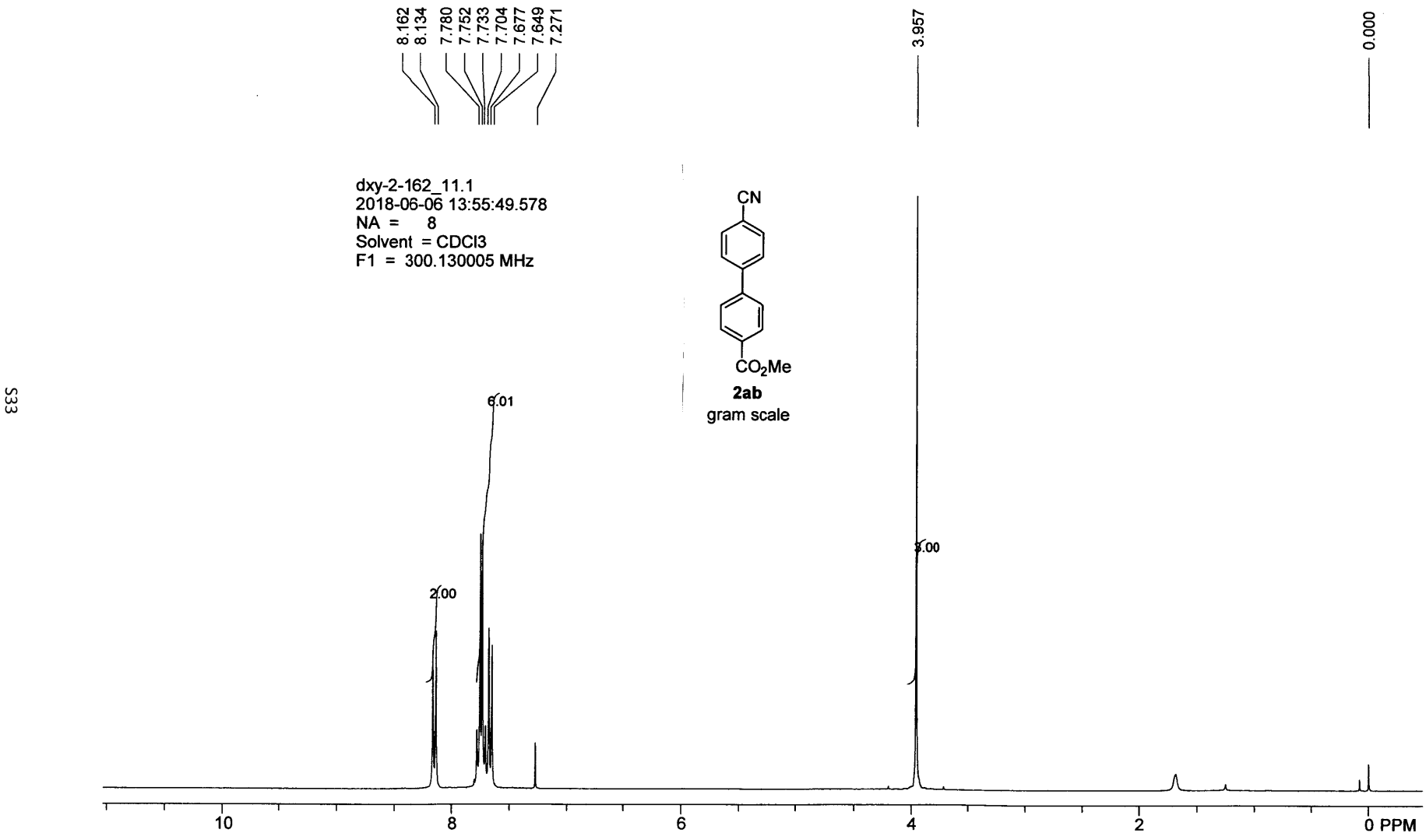
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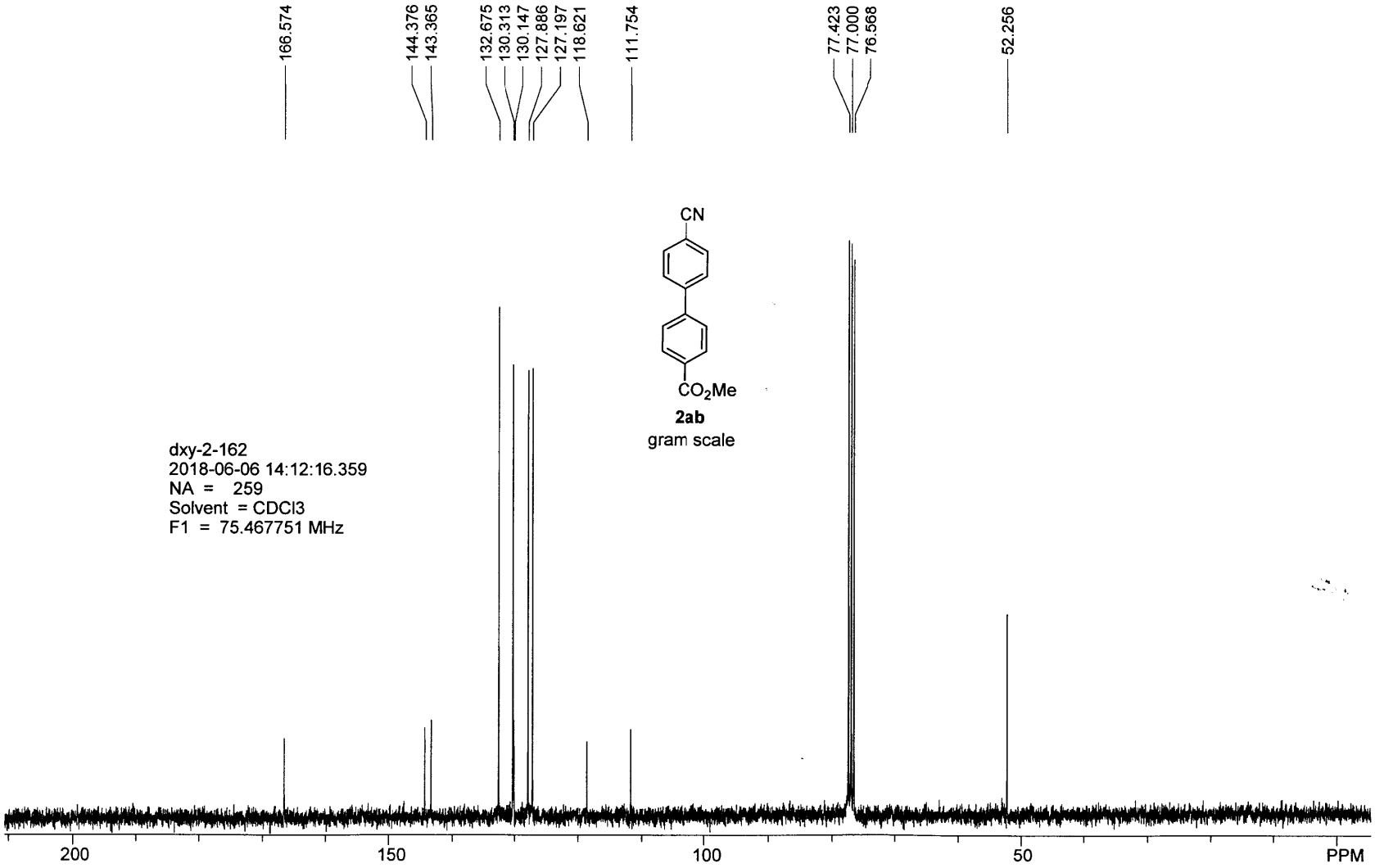
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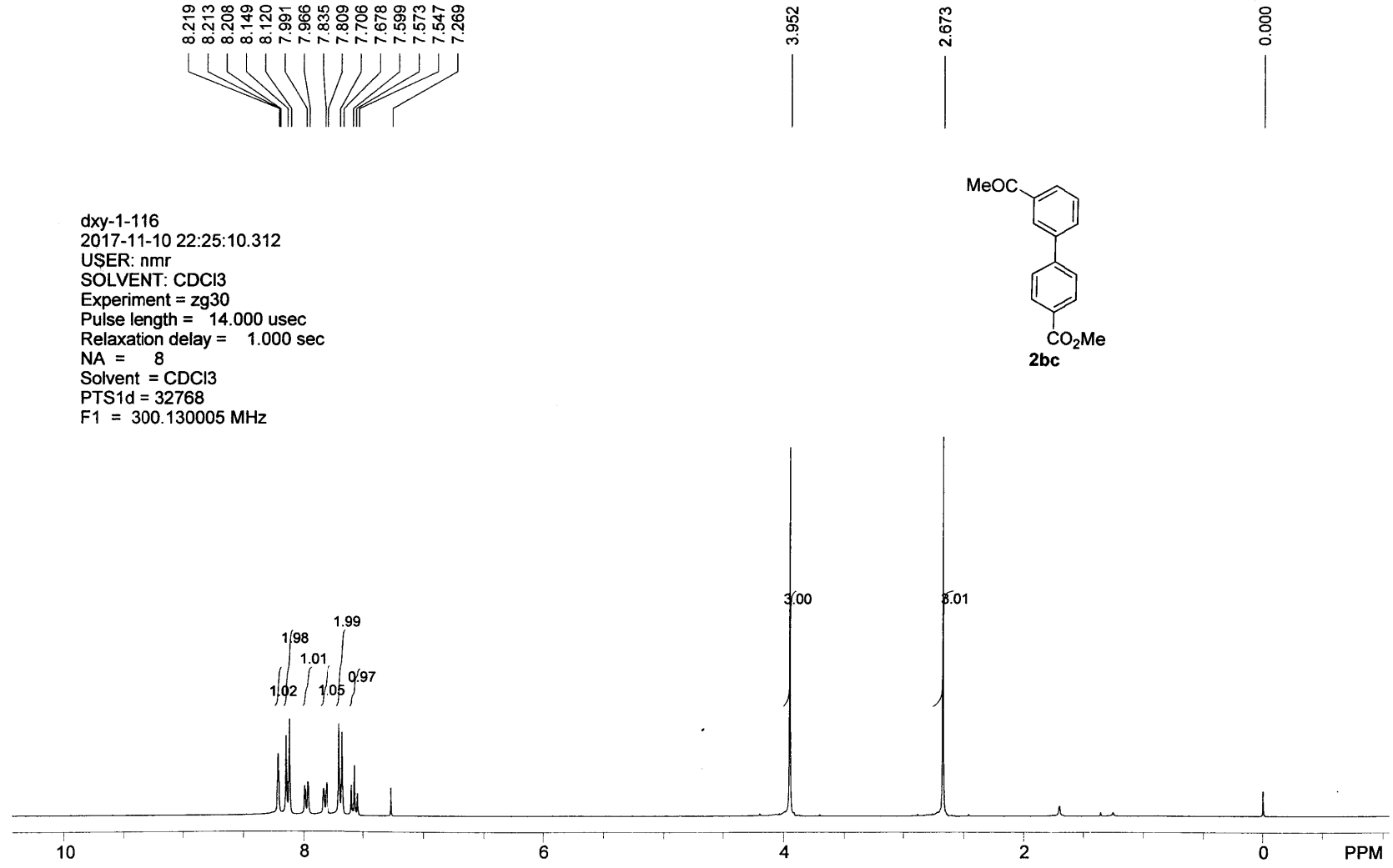
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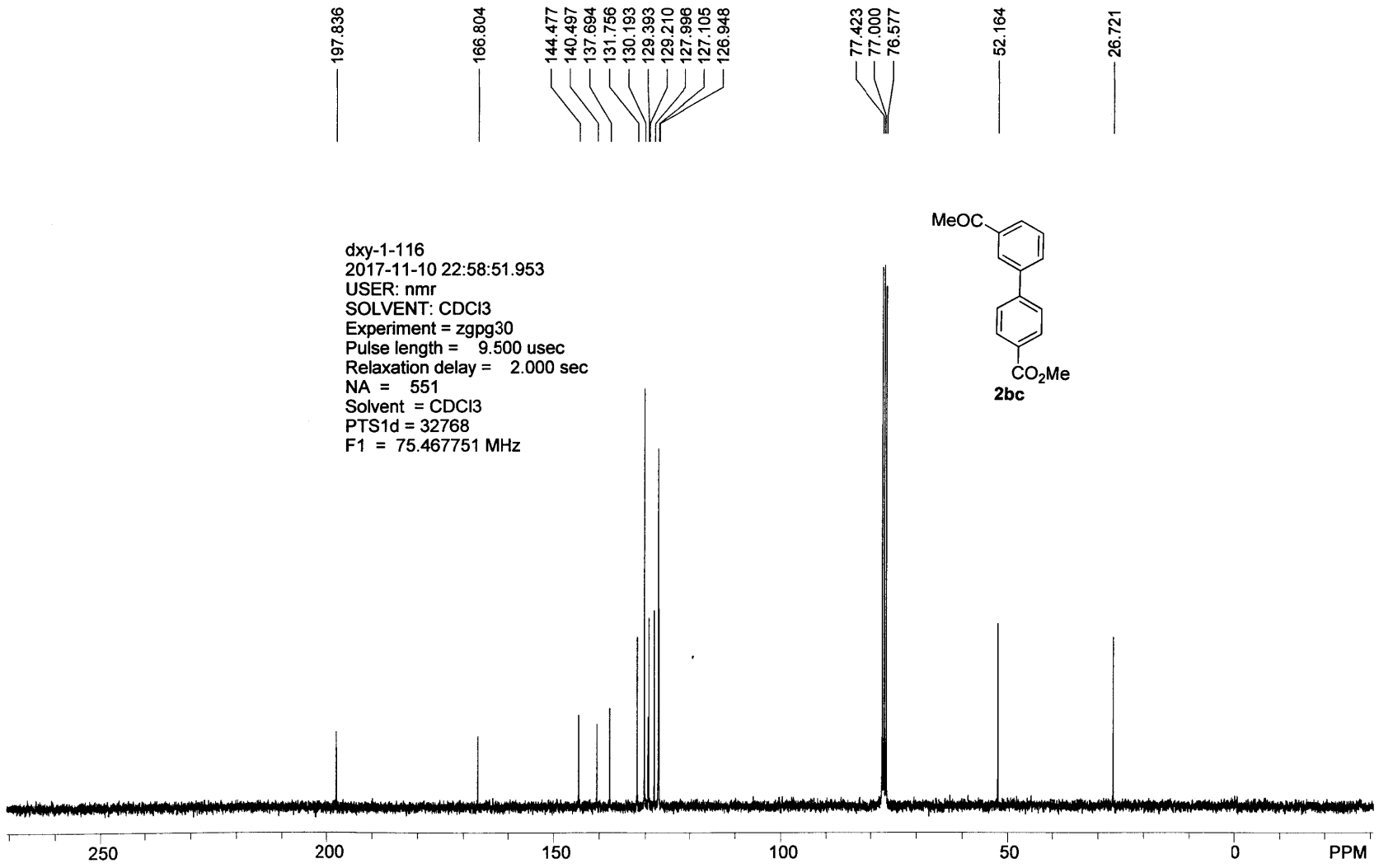
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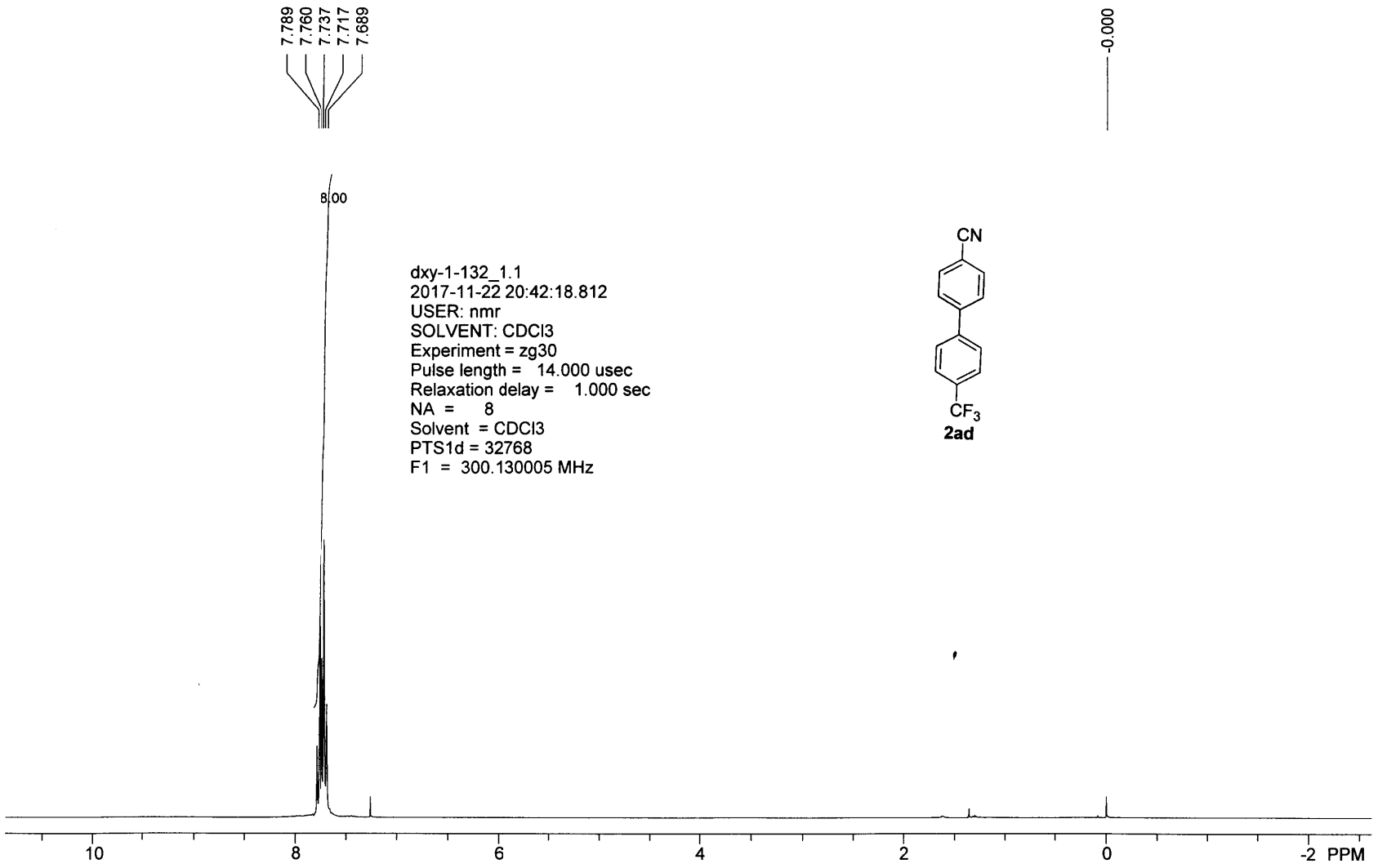
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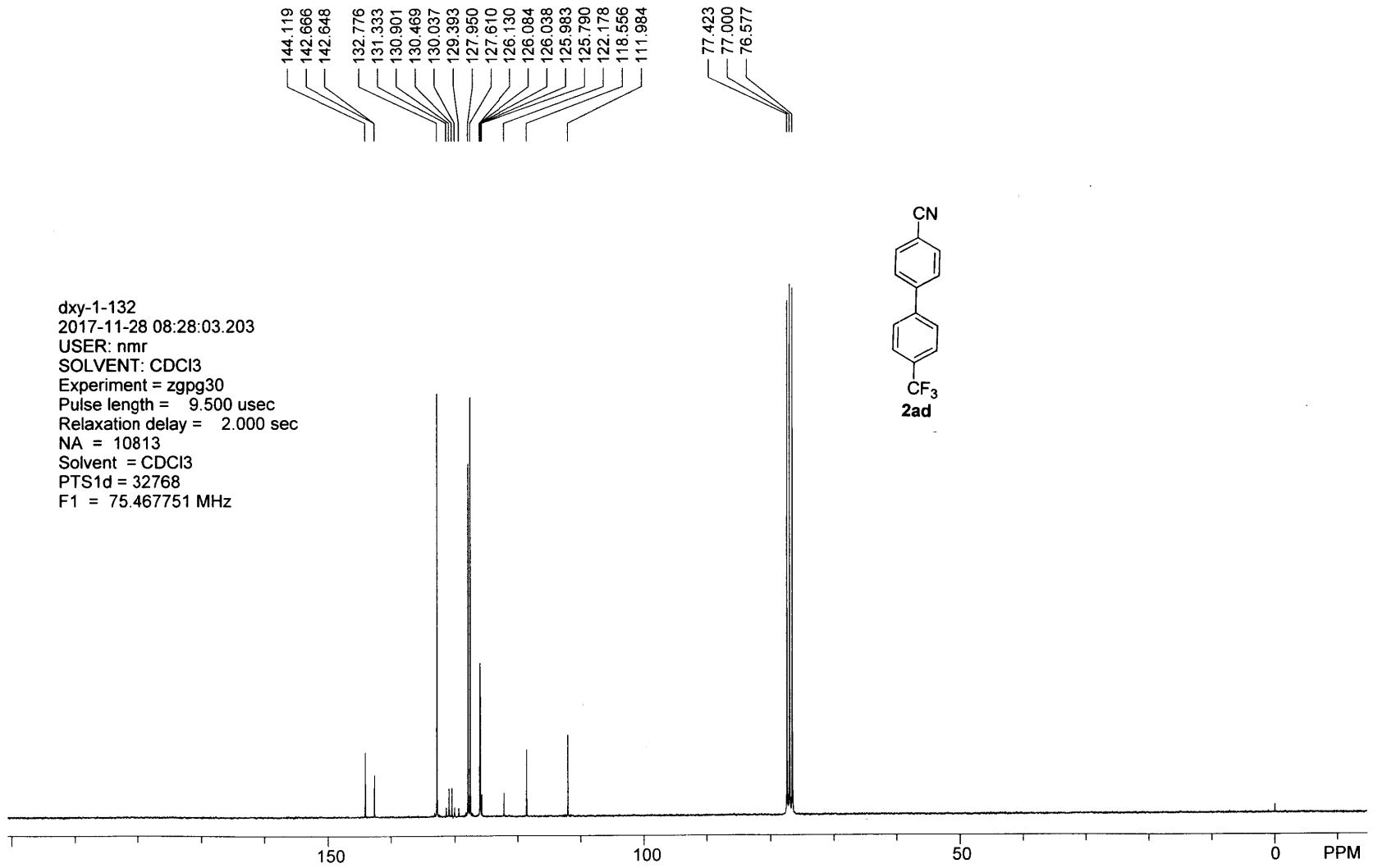
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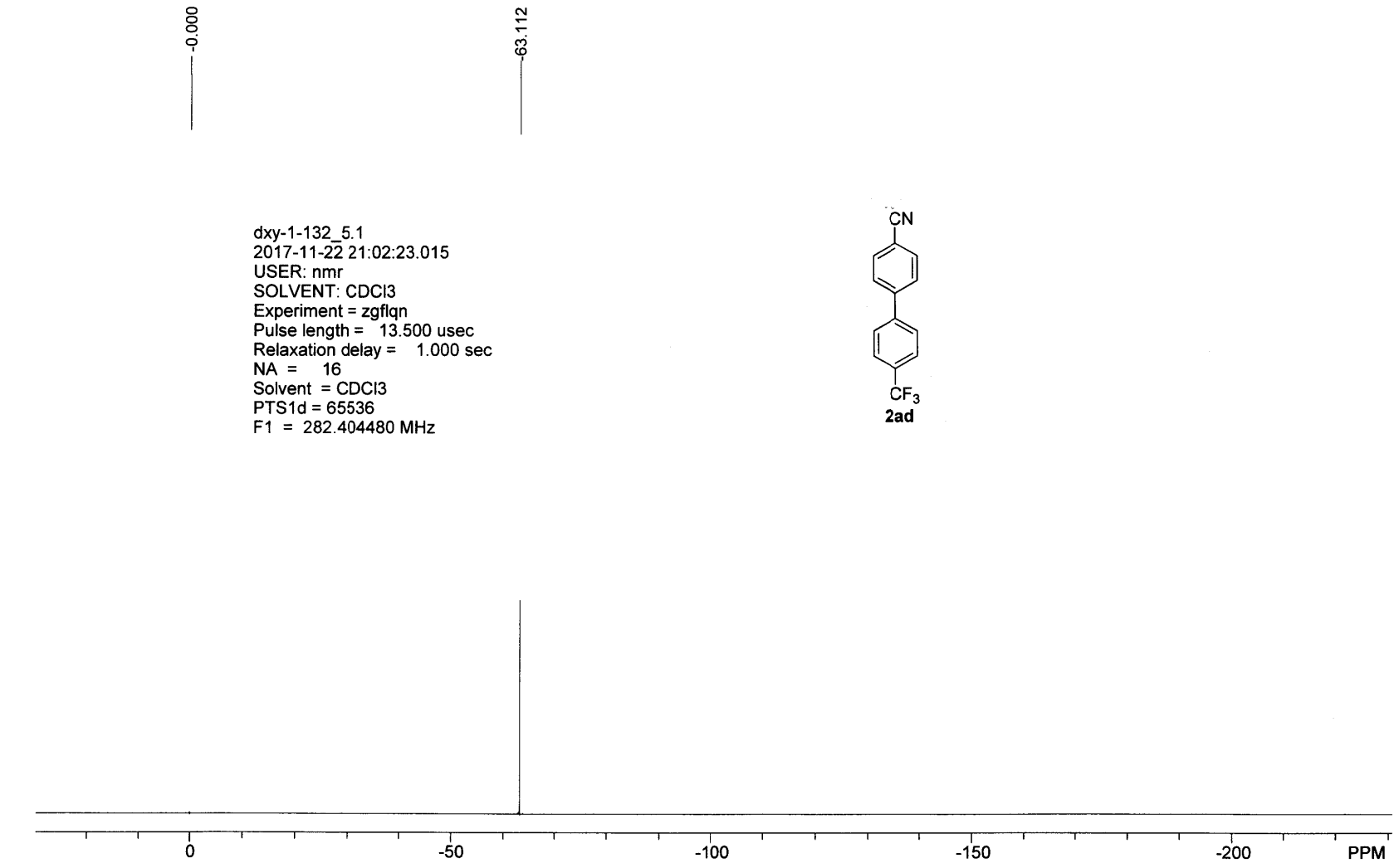
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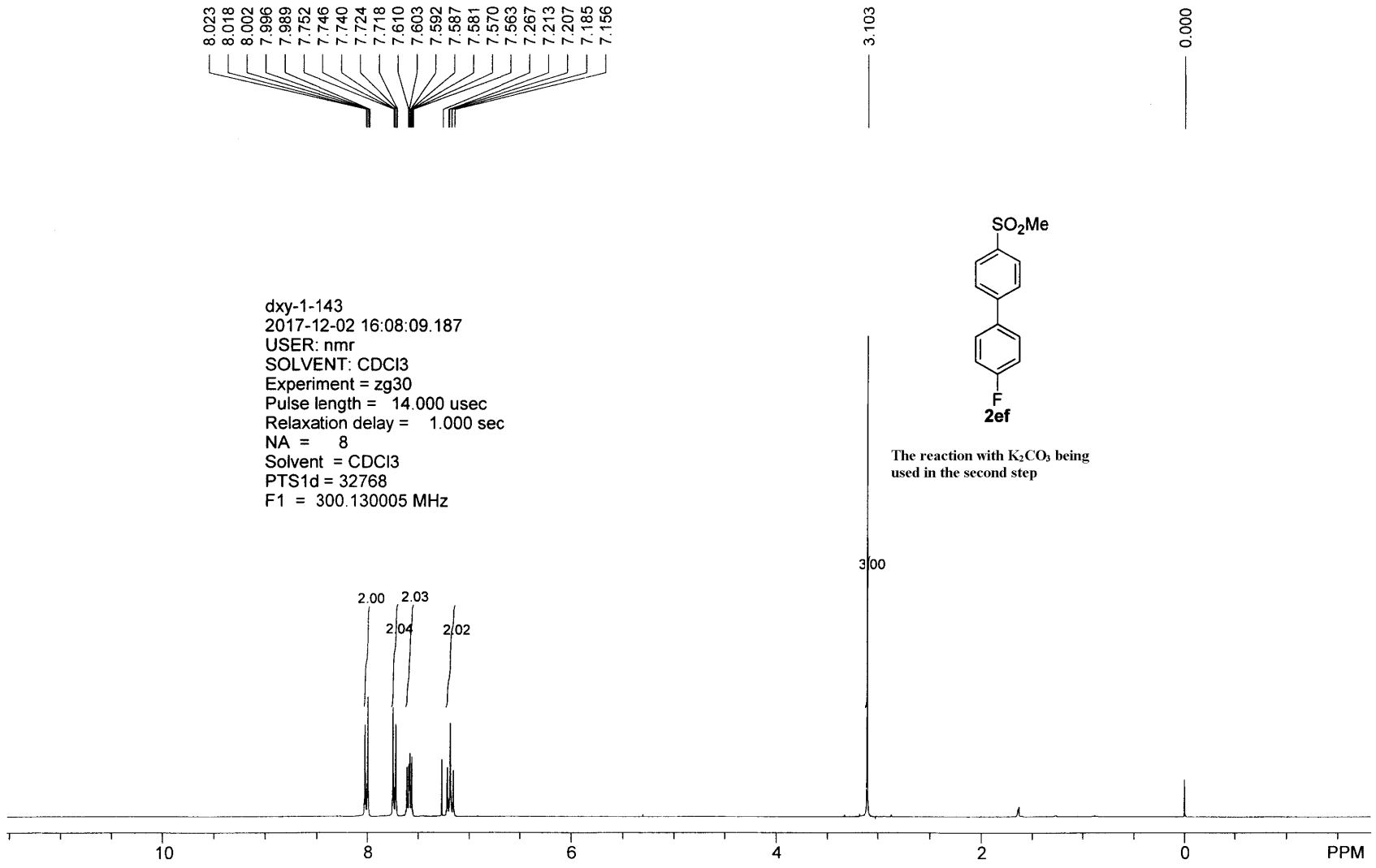
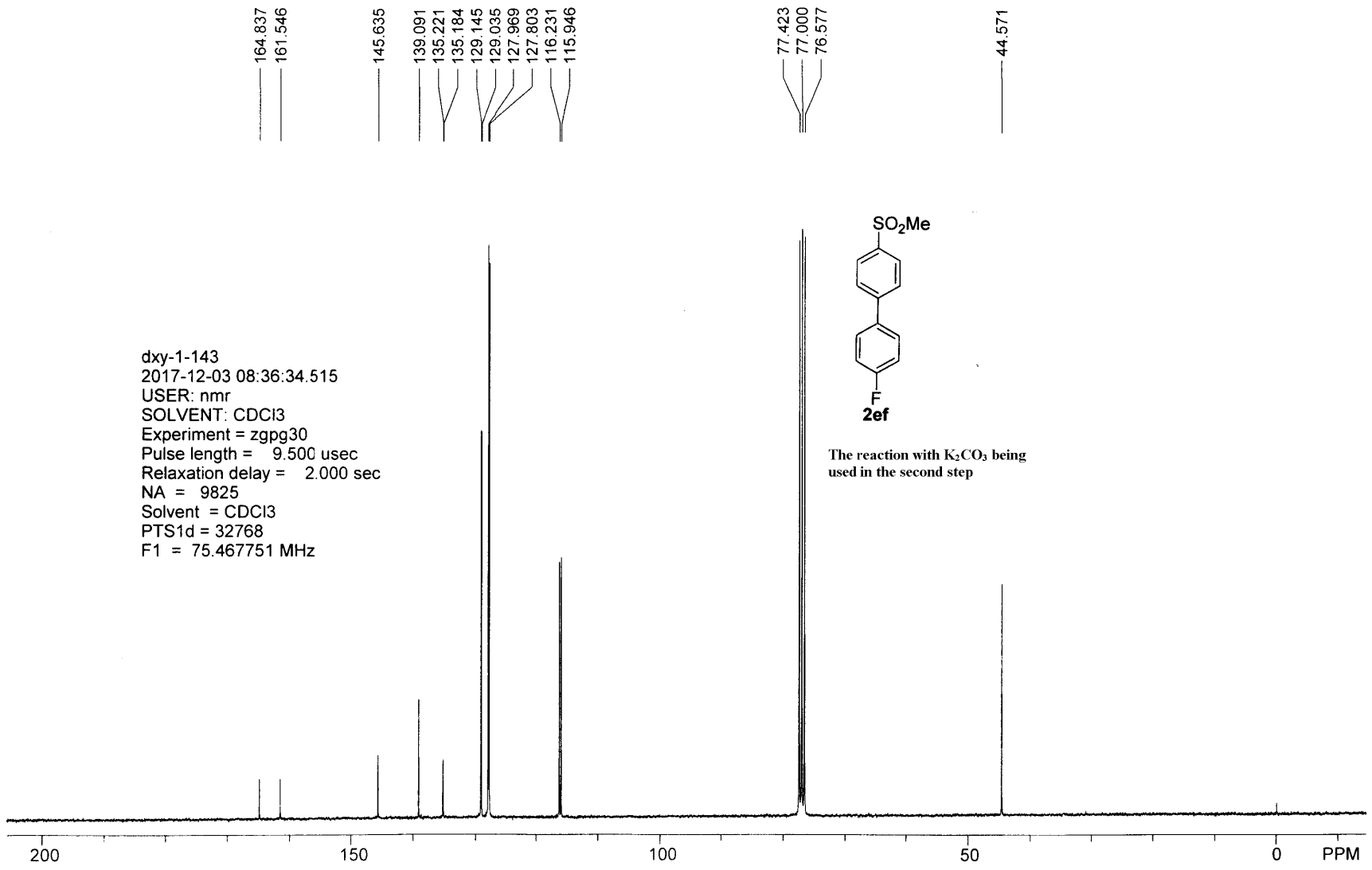
PPM

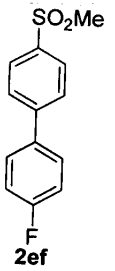

8.023
8.018
8.002
7.996
7.989
7.752
7.746
7.740
7.724
7.718
7.610
7.603
7.592
7.587
7.581
7.570
7.563
7.267
7.213
7.207
7.185
7.156
3.103
0.000
dxy-1-143
2017-12-02 16:08:09.187
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
SO2Me
F
2ef
The reaction with K2CO3 being used in the second step
3.00
2.00
2.03
2.04
2.02
10
8
6
4
2
0
PPM


164.837
161.546
145.635
139.091
135.221
135.184
129.145
129.035
127.969
127.803
116.231
115.946
77.423
77.000
76.577
44.571
dxy-1-143
2017-12-03 08:36:34.515
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 9825
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
SO2Me
F
2ef
The reaction with K2CO3 being used in the second step
200
150
100
50
0
PPM

-0.000

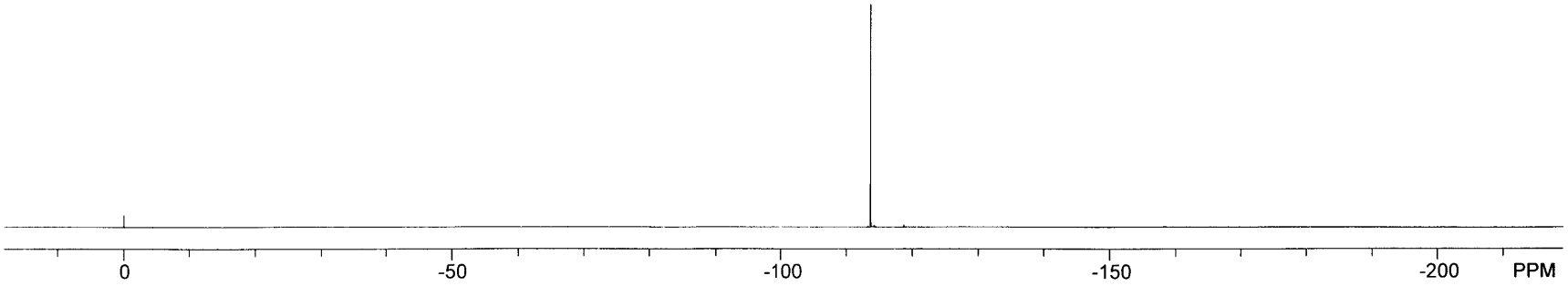
-113.659

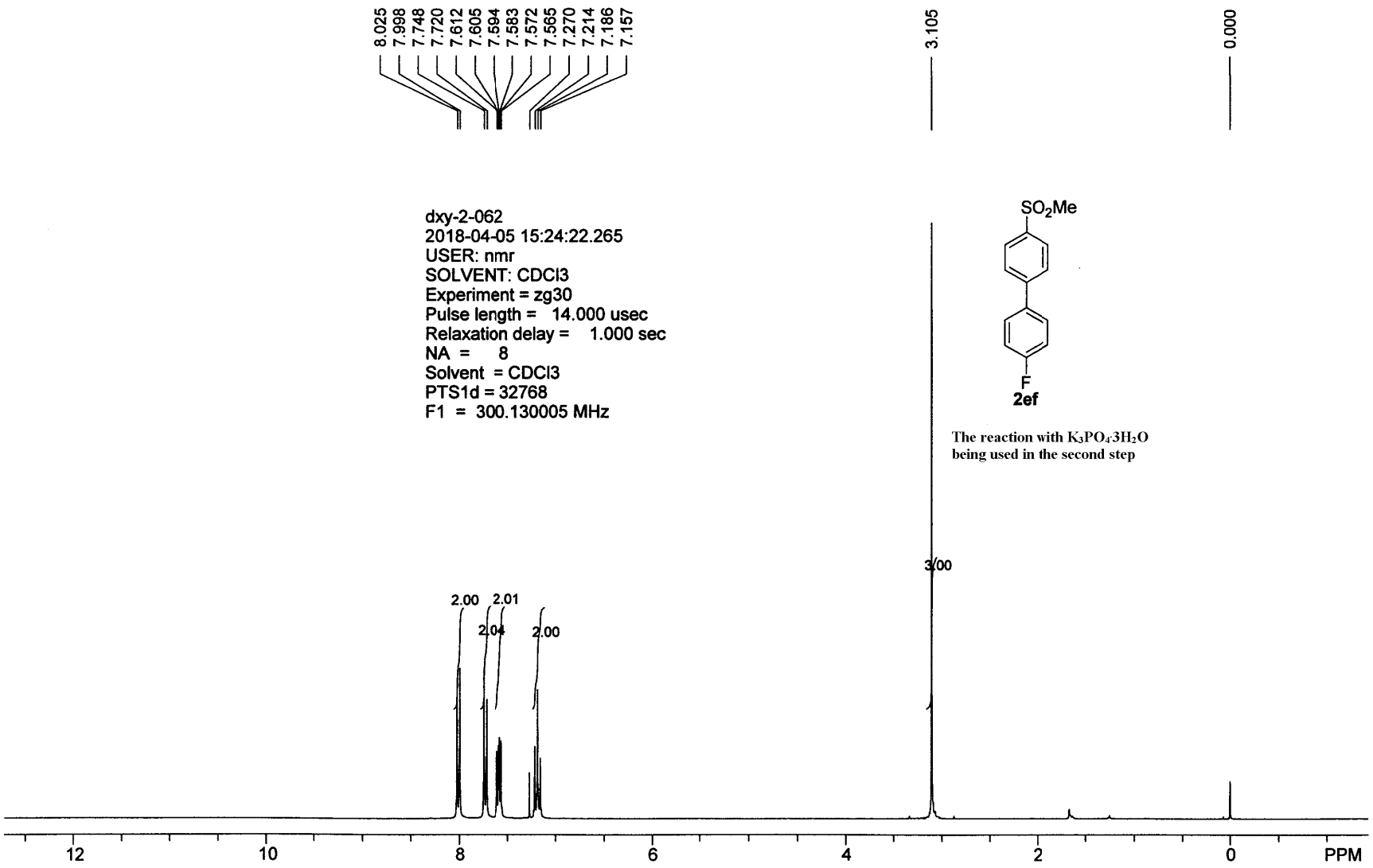
dxy-1-143
2017-12-08 20:45:52.703
USER: nmr
SOLVENT: CDCl3
Experiment = zgfhigqn
Pulse length = 13.500 usec
Relaxation delay = 1.000 sec
NA = 16
Solvent = CDCl3
PTS1d = 65536
F1 = 282.404480 MHz

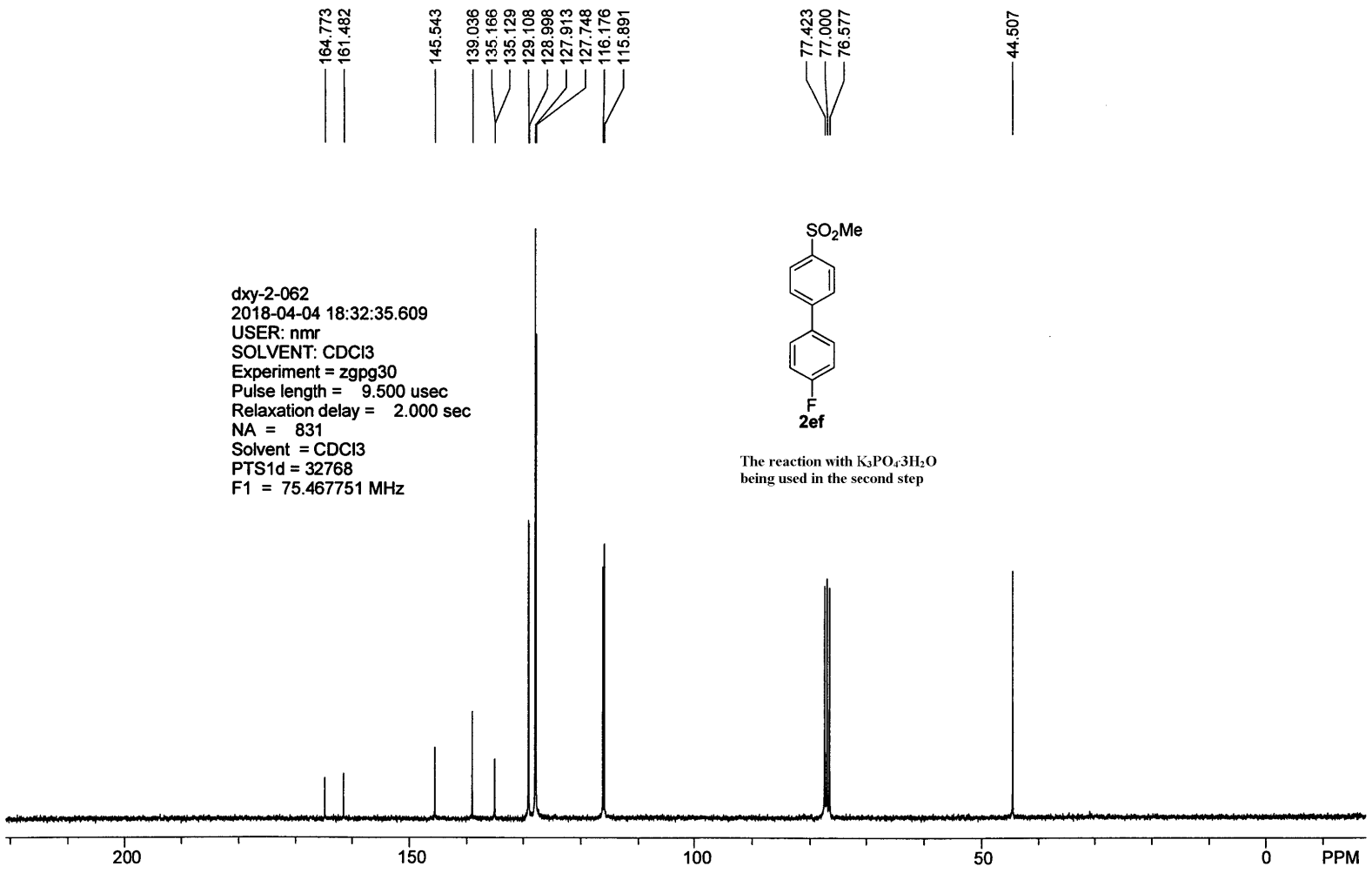


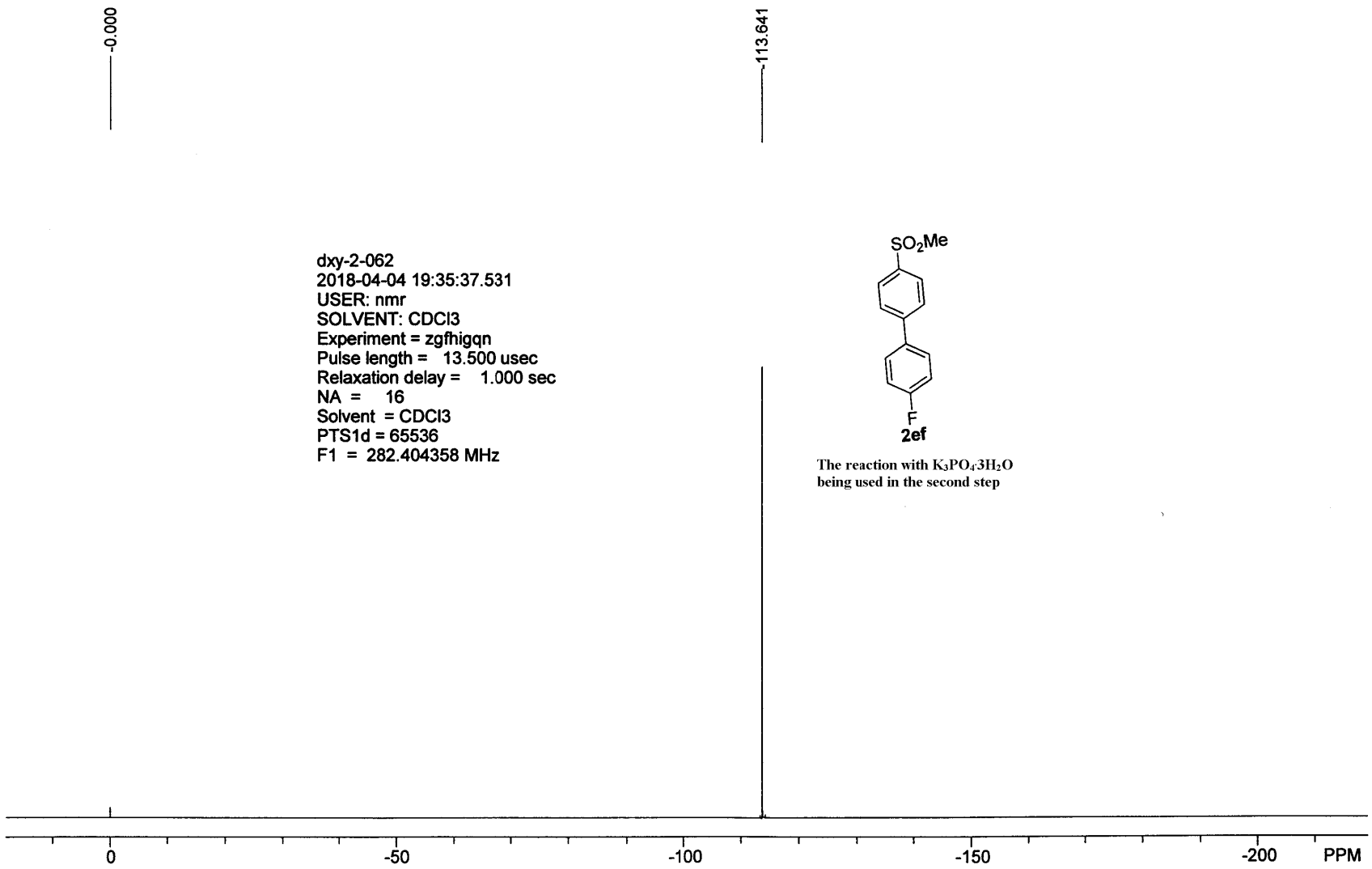
2ef

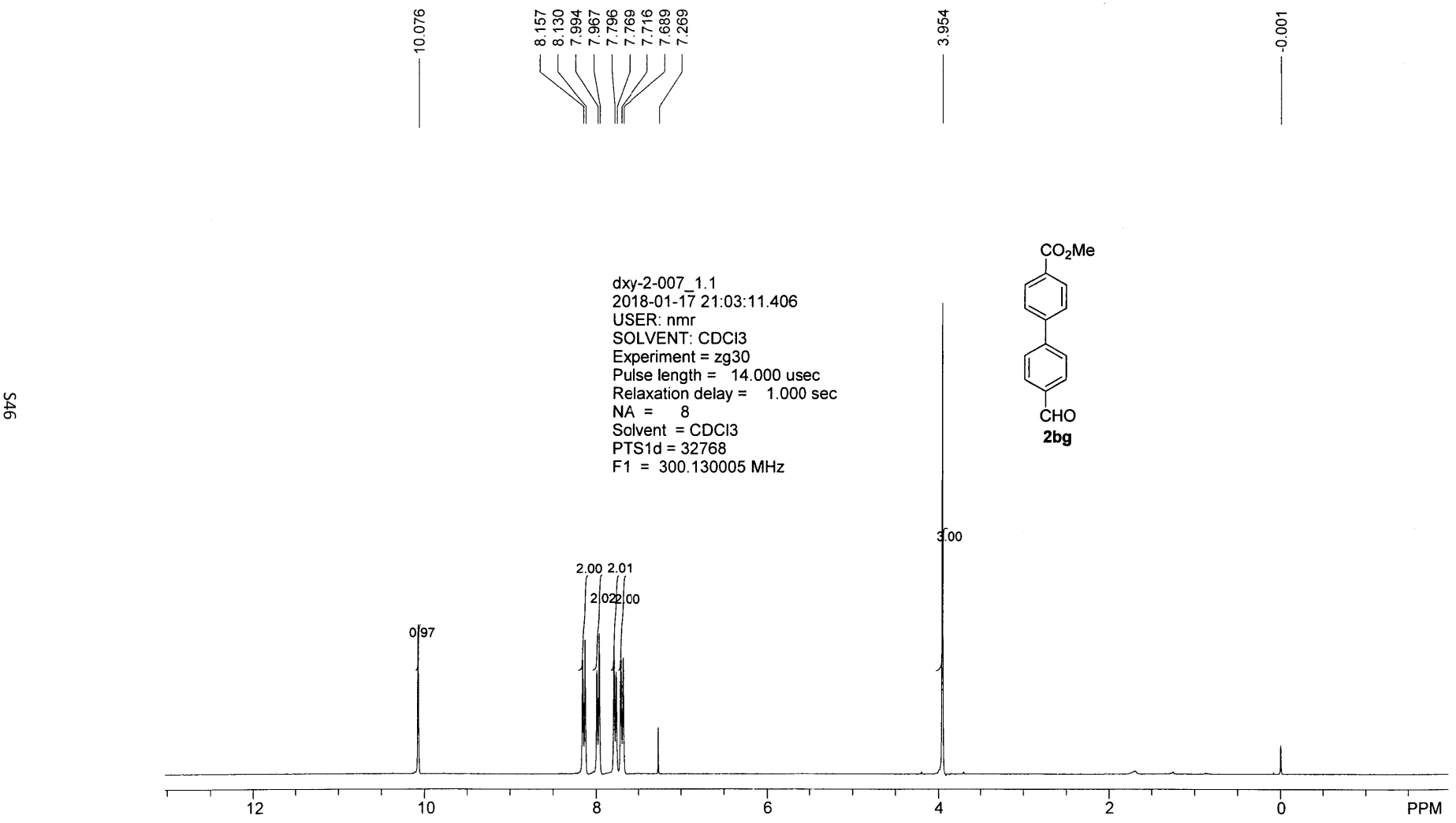
The reaction with K_2CO_3 being used in the second step

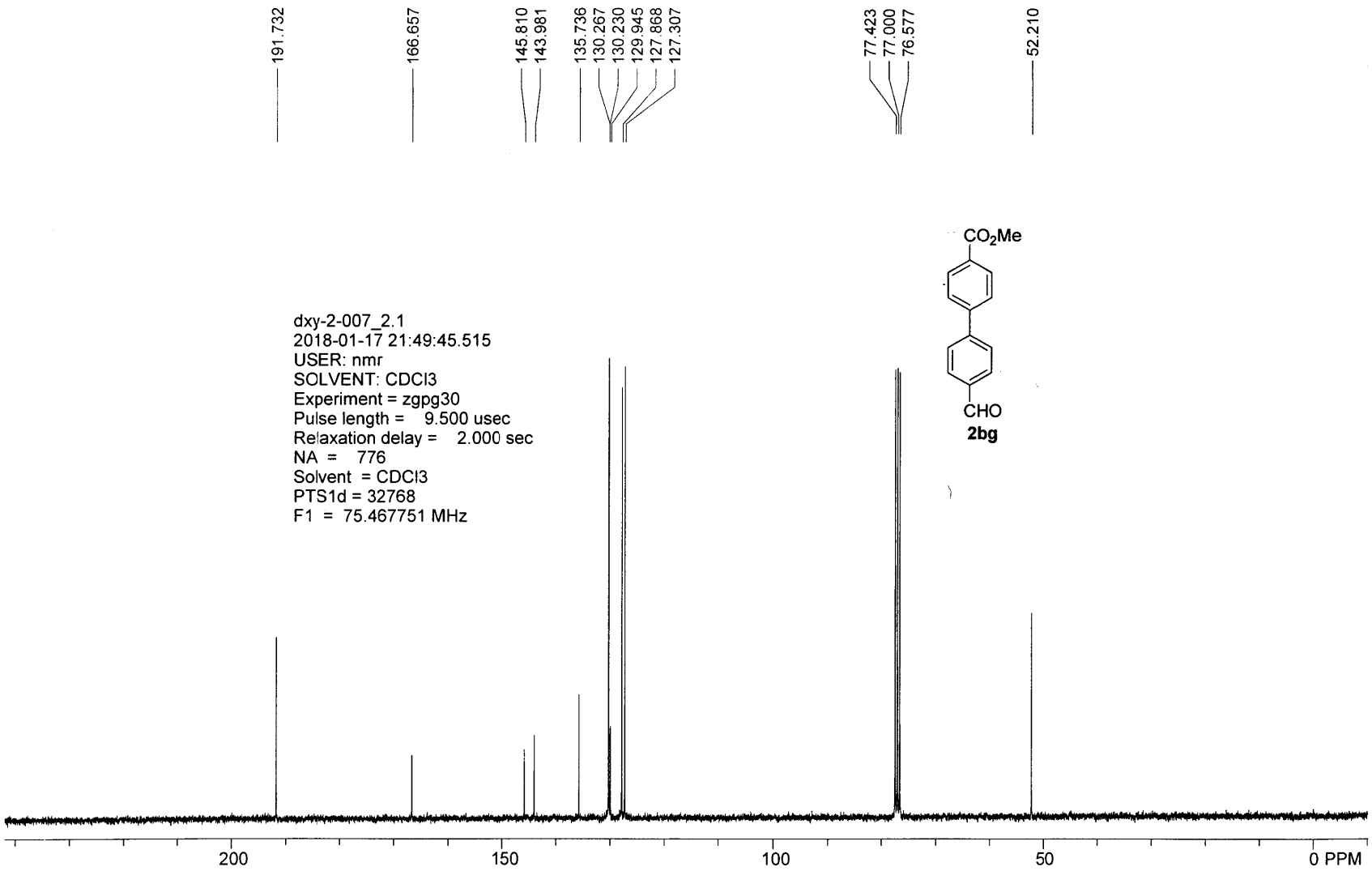


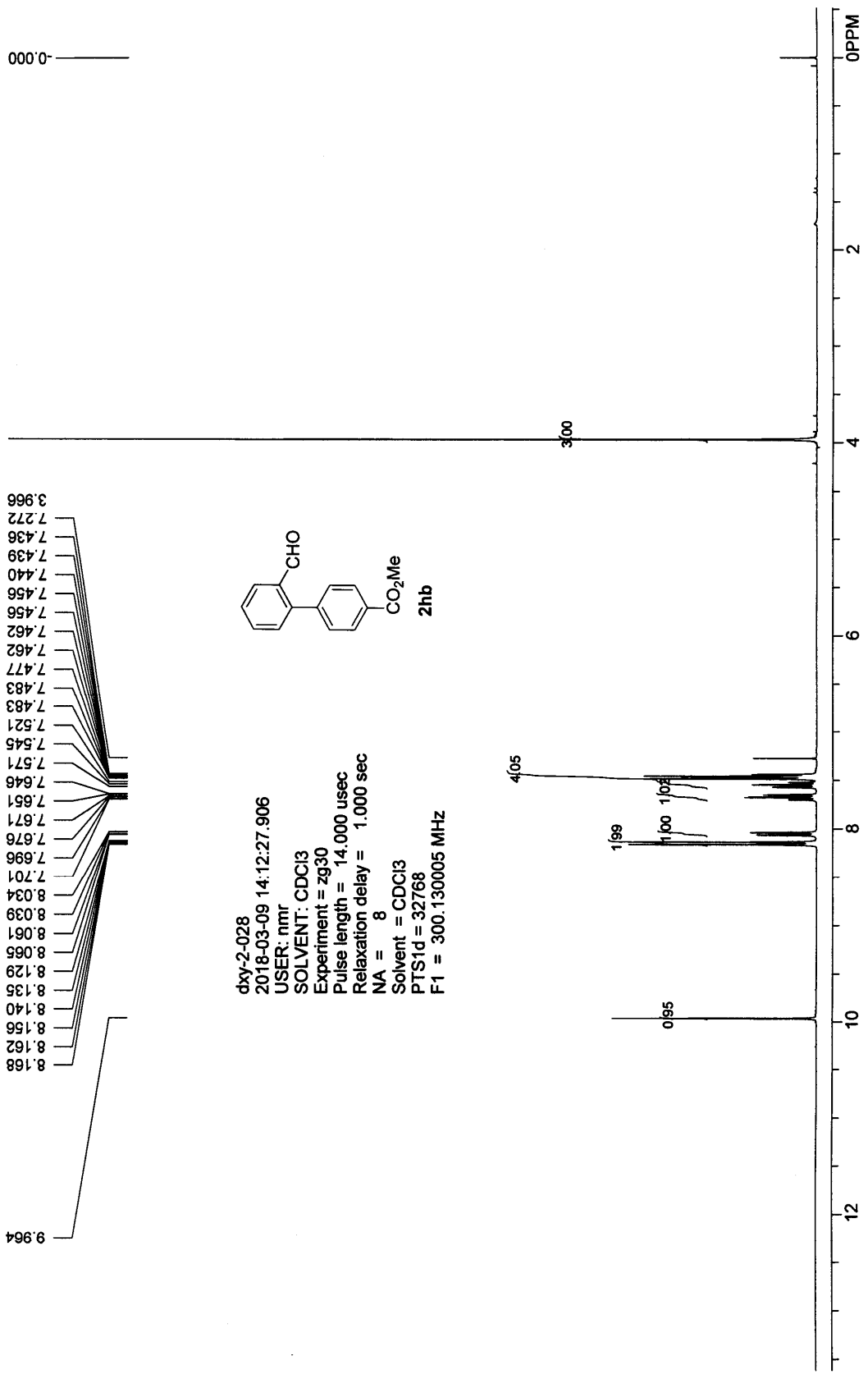

8.025
7.998
7.748
7.720
7.612
7.605
7.594
7.583
7.572
7.565
7.270
7.214
7.186
7.157
3.105
0.000
dxy-2-062
2018-04-05 15:24:22.265
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
SO2Me
F
2ef
The reaction with K3PO43H2O
being used in the second step
3.00
2.00
2.01
2.04
2.00
12
10
8
6
4
2
0
PPM

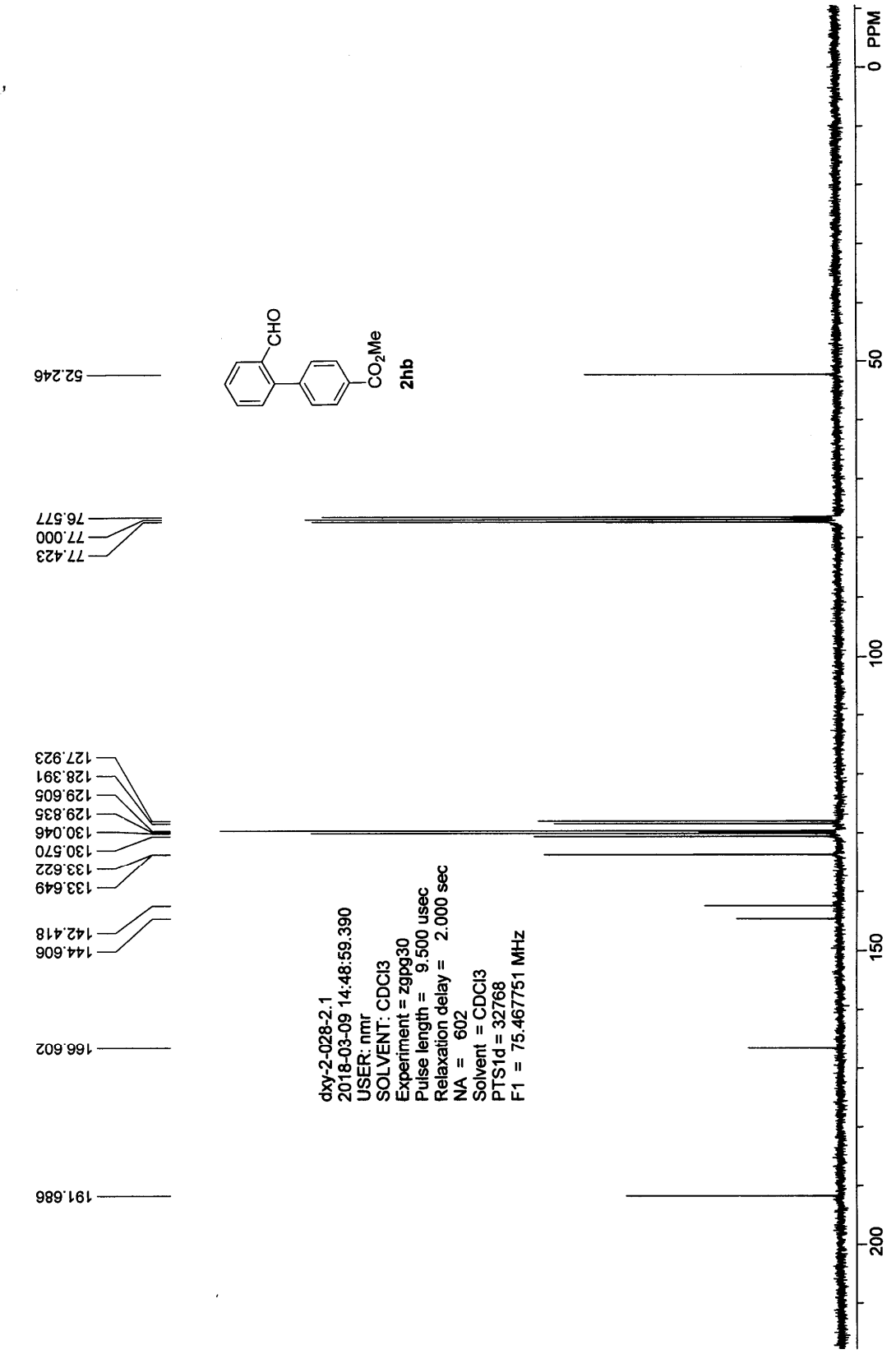

164.773
161.482
145.543
139.036
135.166
135.129
129.108
128.998
127.913
127.748
116.176
115.891
77.423
77.000
76.577
44.507
dxy-2-062
2018-04-04 18:32:35.609
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 831
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
SO2Me
F
2ef
The reaction with K3PO4 3H2O
being used in the second step
200
150
100
50
0
PPM

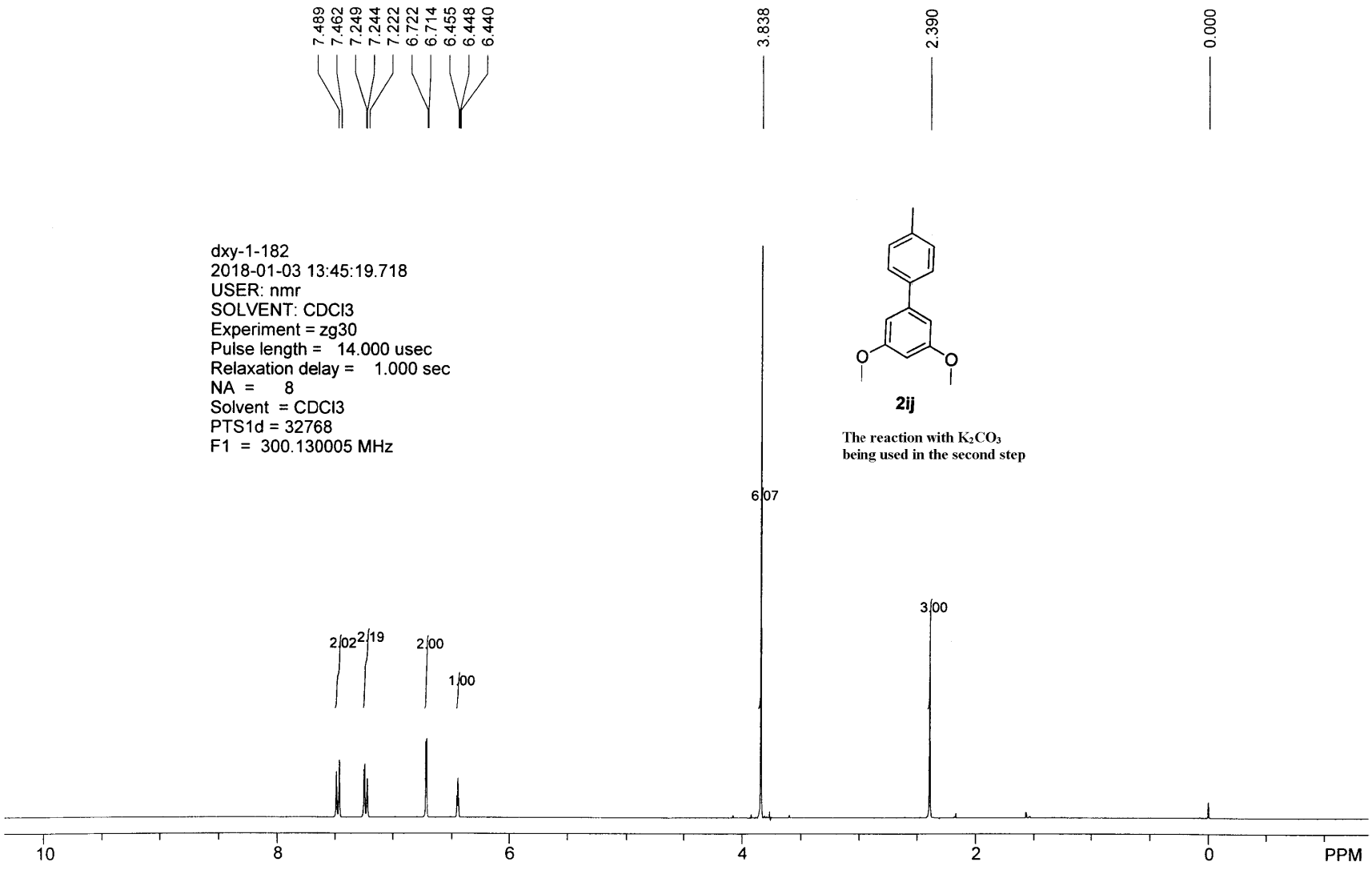

-0.000
-113.641
dxy-2-062
2018-04-04 19:35:37.531
USER: nmr
SOLVENT: CDCl3
Experiment = zgfhigqn
Pulse length = 13.500 usec
Relaxation delay = 1.000 sec
NA = 16
Solvent = CDCl3
PTS1d = 65536
F1 = 282.404358 MHz
SO2Me
F
2ef
The reaction with K3PO4·3H2O being used in the second step
0
-50
-100
-150
-200
PPM

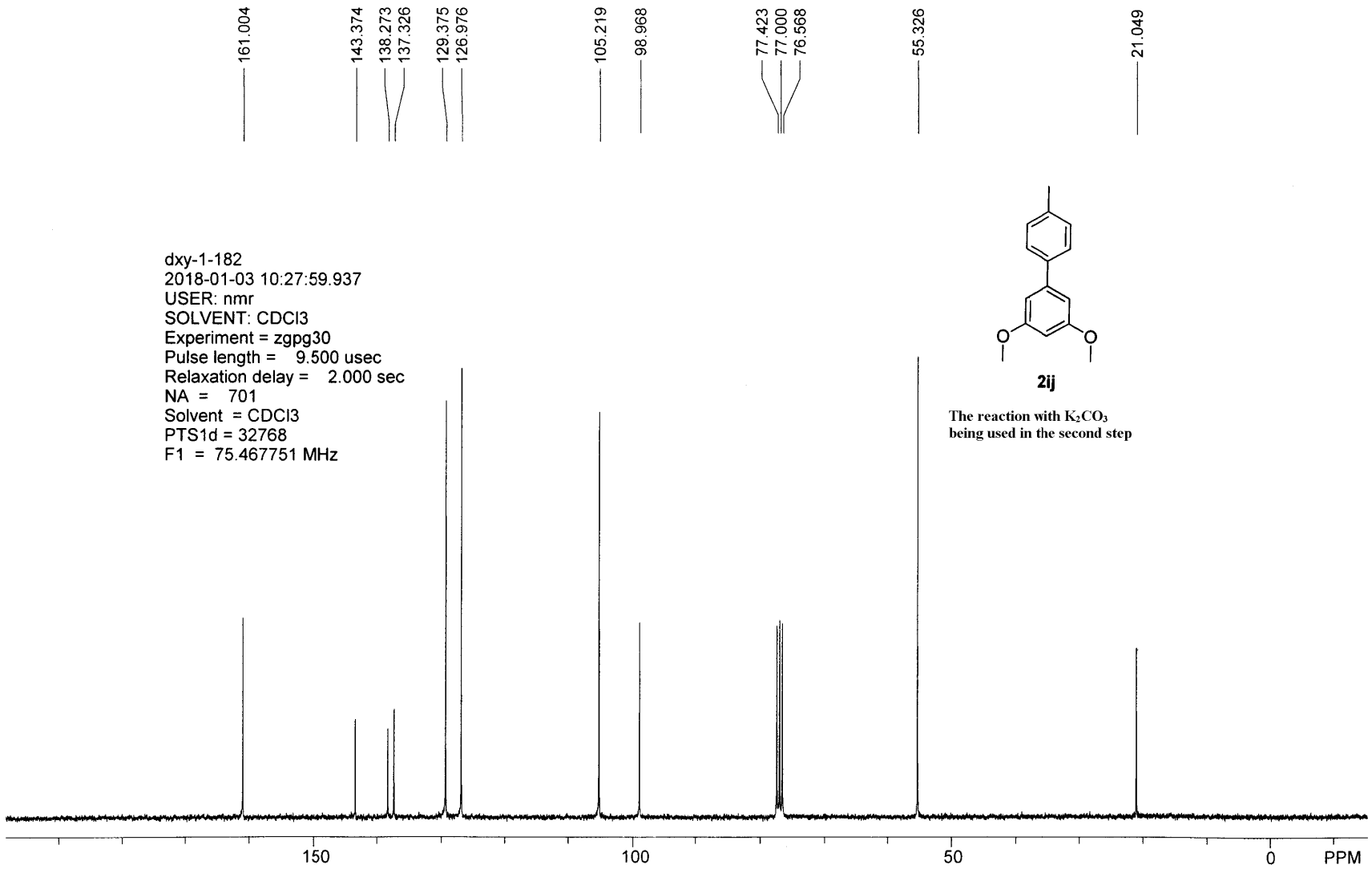

10.076
8.157
8.130
7.994
7.967
7.796
7.769
7.716
7.689
7.269
3.954
-0.001
dxy-2-007_1.1
2018-01-17 21:03:11.406
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
CO2Me
CHO
2bg
0.97
2.00
2.01
2.02
2.00
3.00
12
10
8
6
4
2
0
PPM

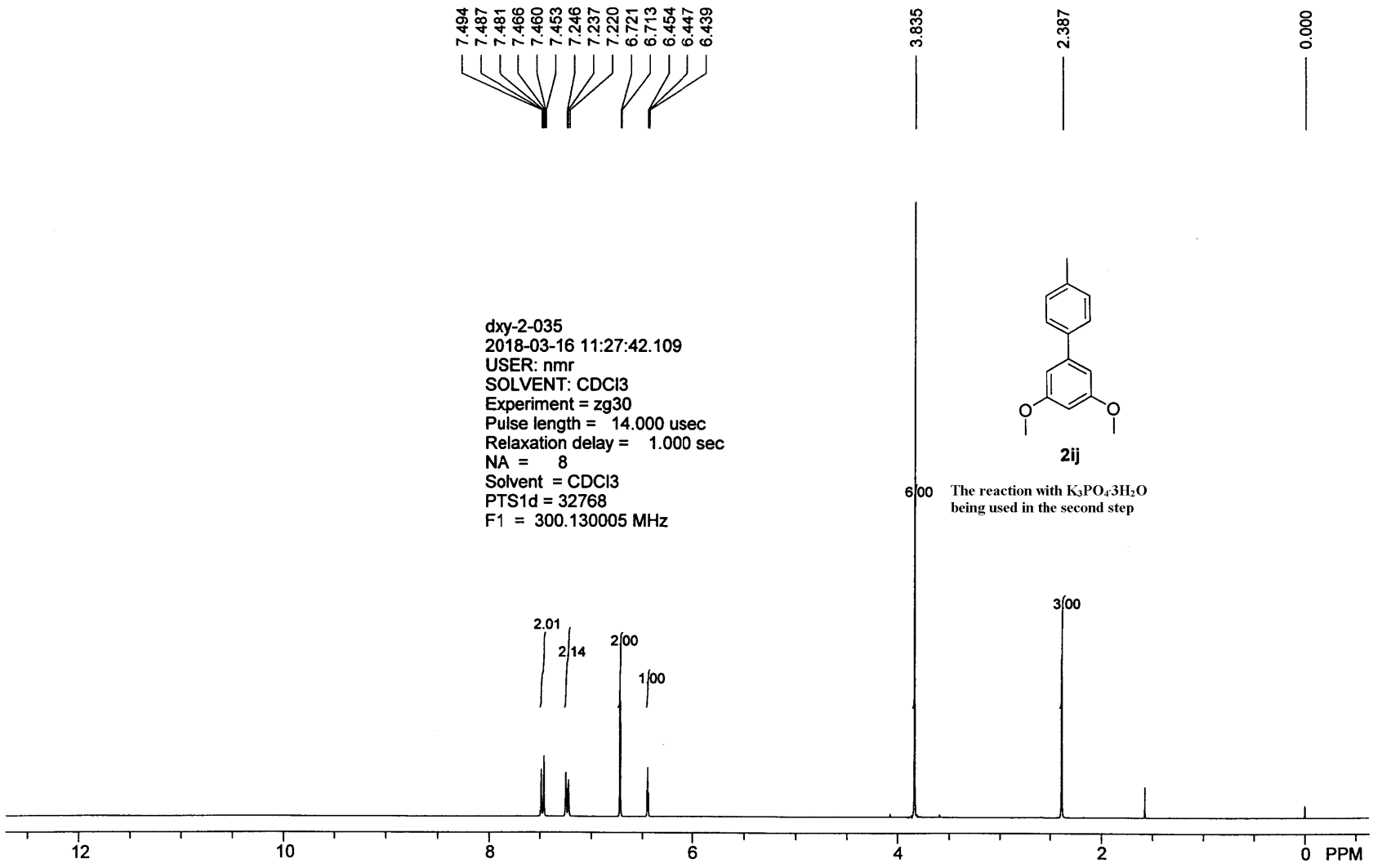

191.732
166.657
145.810
143.981
135.736
130.267
130.230
129.945
127.868
127.307
77.423
77.000
76.577
52.210
dxy-2-007_2.1
2018-01-17 21:49:45.515
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 776
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
CO2Me
CHO
2bg
200
150
100
50
0 PPM

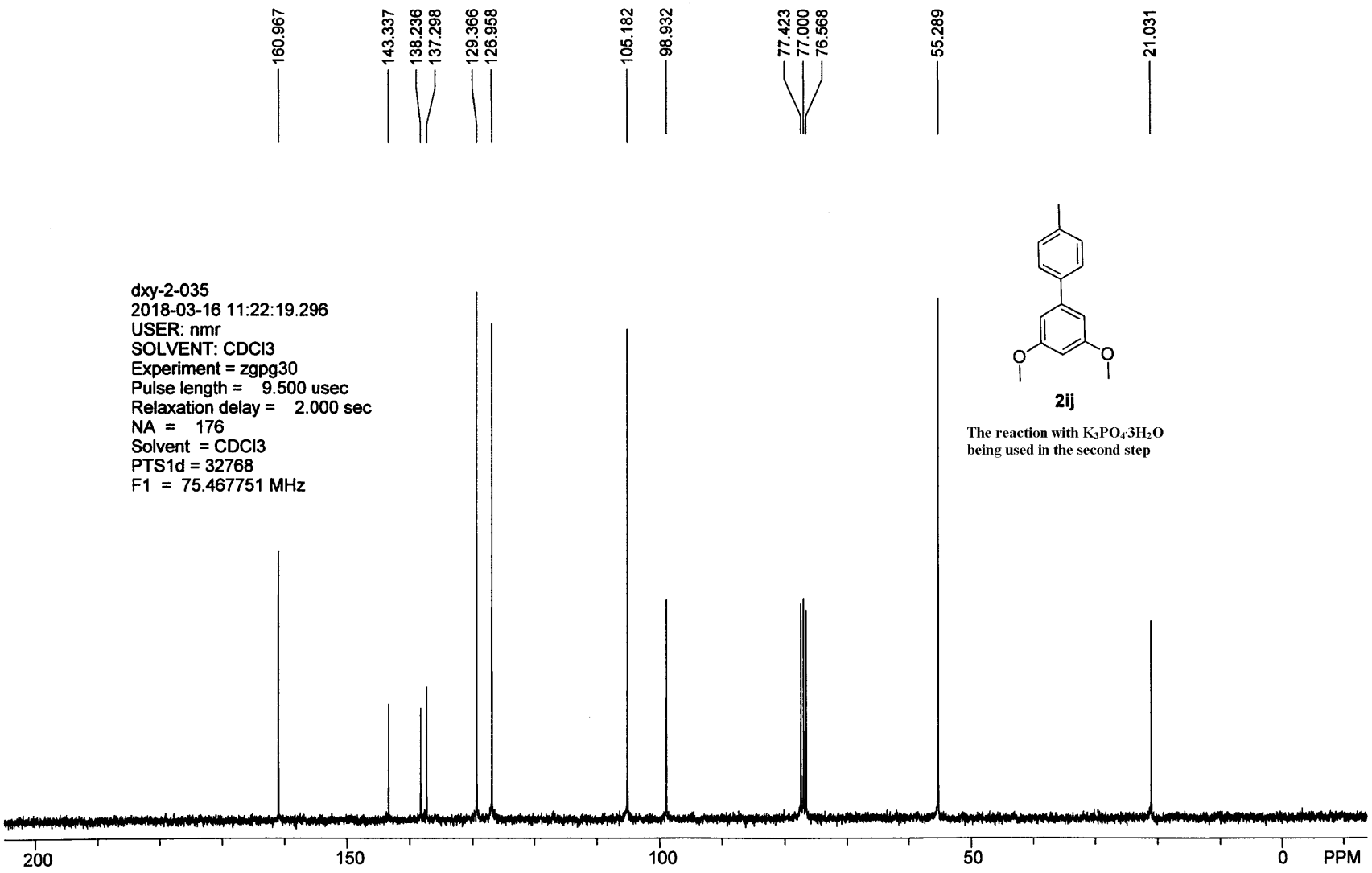

dxy-2-028
2018-03-09 14:12:27.906
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
CHO
CO2Me
2hb
3.00
4.05
1.99
1.00
1.03
0.95
0PPM
2
4
6
8
10
12
-0.000
9.964
8.168
8.162
8.156
8.140
8.135
8.129
8.065
8.061
8.039
8.034
7.701
7.696
7.676
7.671
7.661
7.646
7.571
7.545
7.521
7.483
7.483
7.477
7.462
7.462
7.456
7.440
7.439
7.436
7.272
3.966

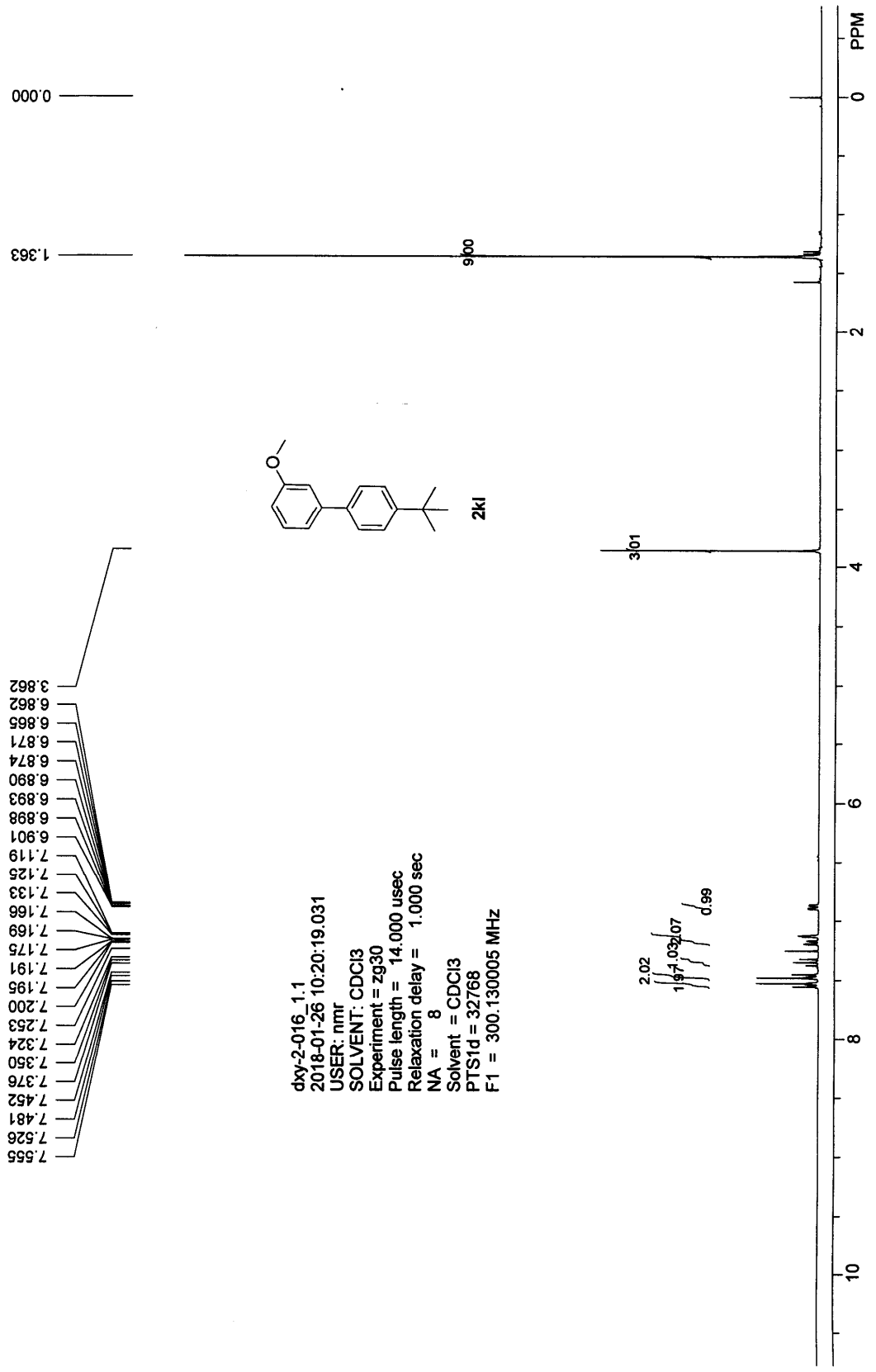

dxy-2-028-2.1
2018-03-09 14:48:59.390
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 602
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
CHO
CO2Me
2hb
52.246
76.577
77.000
77.423
127.923
128.391
129.605
129.835
130.046
130.570
133.622
133.649
142.418
144.606
166.602
191.686
0 PPM
50
100
150
200

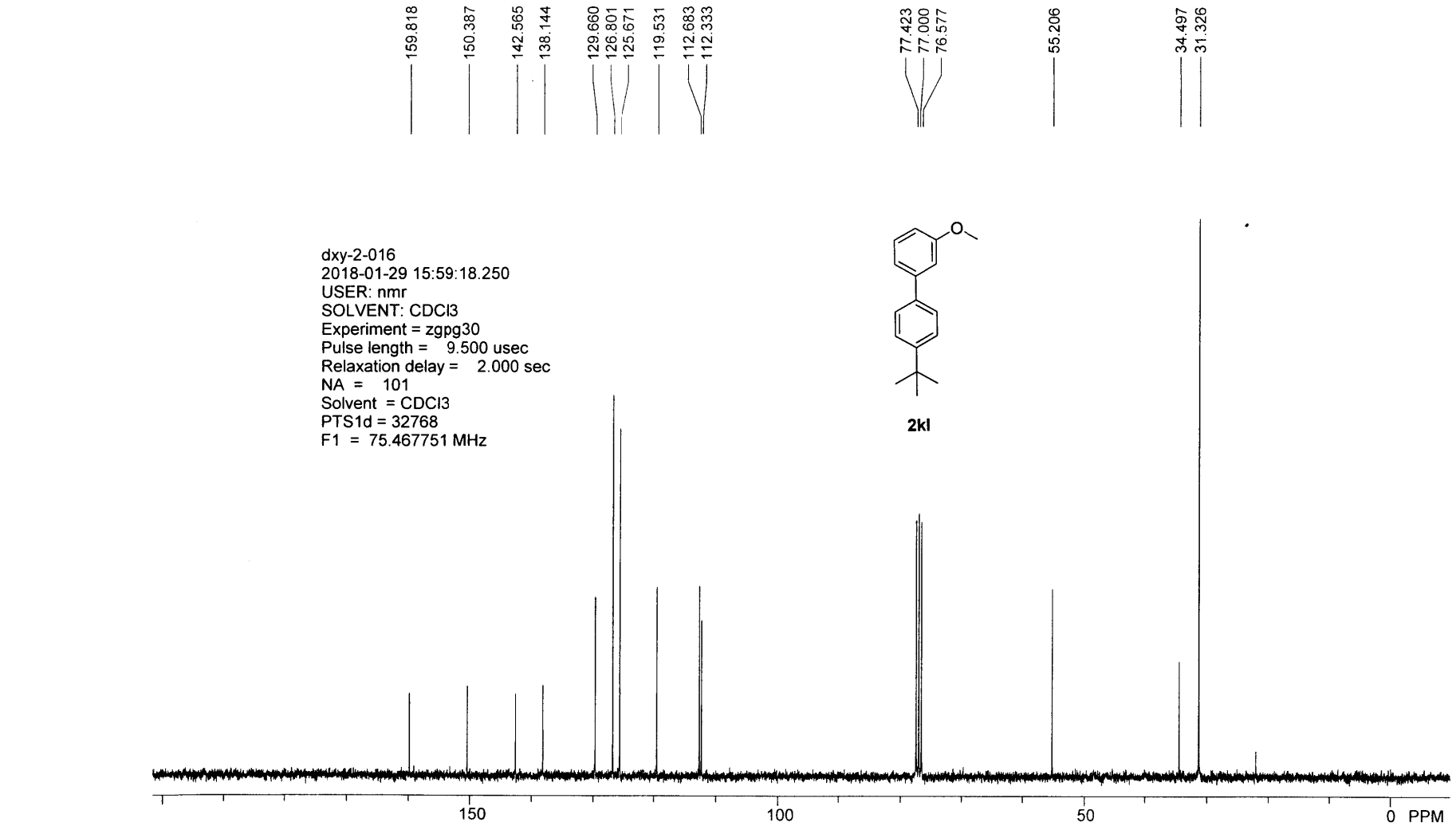

7.489
7.462
7.249
7.244
7.222
6.722
6.714
6.455
6.448
6.440
3.838
2.390
0.000
dxy-1-182
2018-01-03 13:45:19.718
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
2ij
The reaction with K2CO3
being used in the second step
6.07
3.00
2.02
2.19
2.00
1.00
10
8
6
4
2
0
PPM

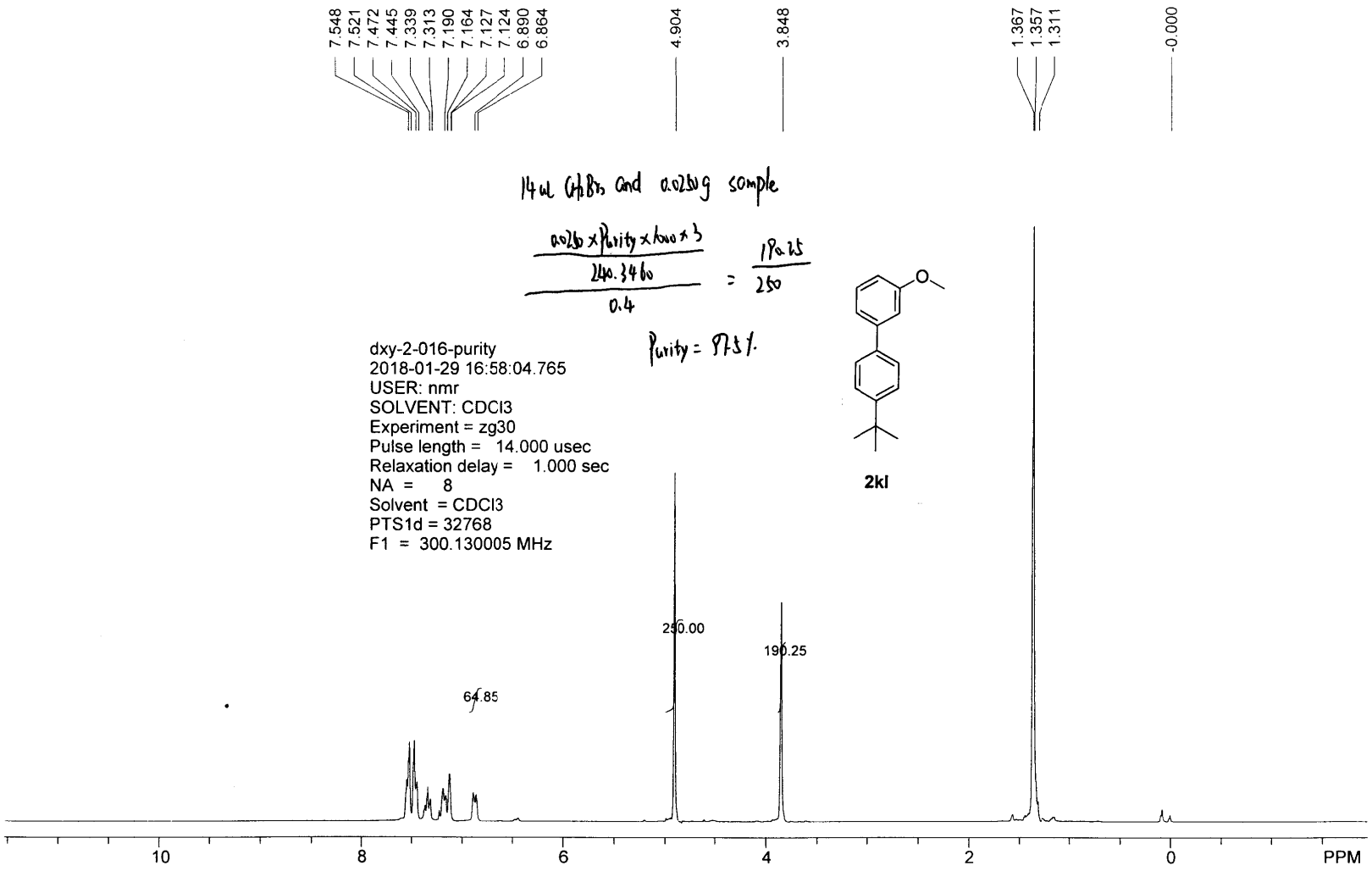

161.004
143.374
138.273
137.326
129.375
126.976
105.219
98.968
77.423
77.000
76.568
55.326
21.049
dxy-1-182
2018-01-03 10:27:59.937
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 701
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
O
O
2ij
The reaction with K_2CO_3 being used in the second step
150
100
50
0
PPM

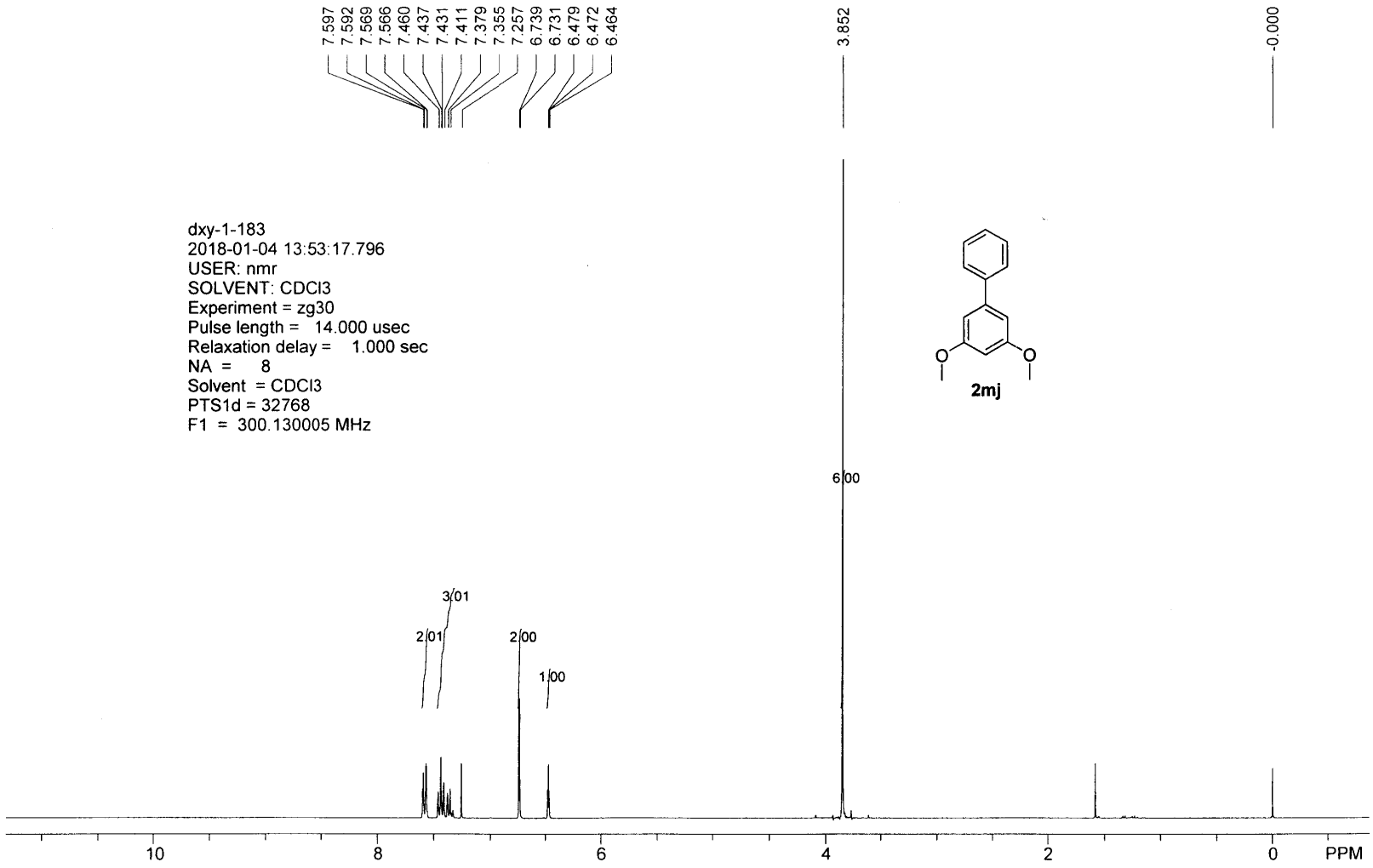

7.494
7.487
7.481
7.466
7.460
7.453
7.246
7.237
7.220
6.721
6.713
6.454
6.447
6.439
3.835
2.387
0.000
dxy-2-035
2018-03-16 11:27:42.109
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
2ij
6.00
The reaction with K3PO4·3H2O being used in the second step
3.00
2.01
2.14
2.00
1.00
12
10
8
6
4
2
0 PPM

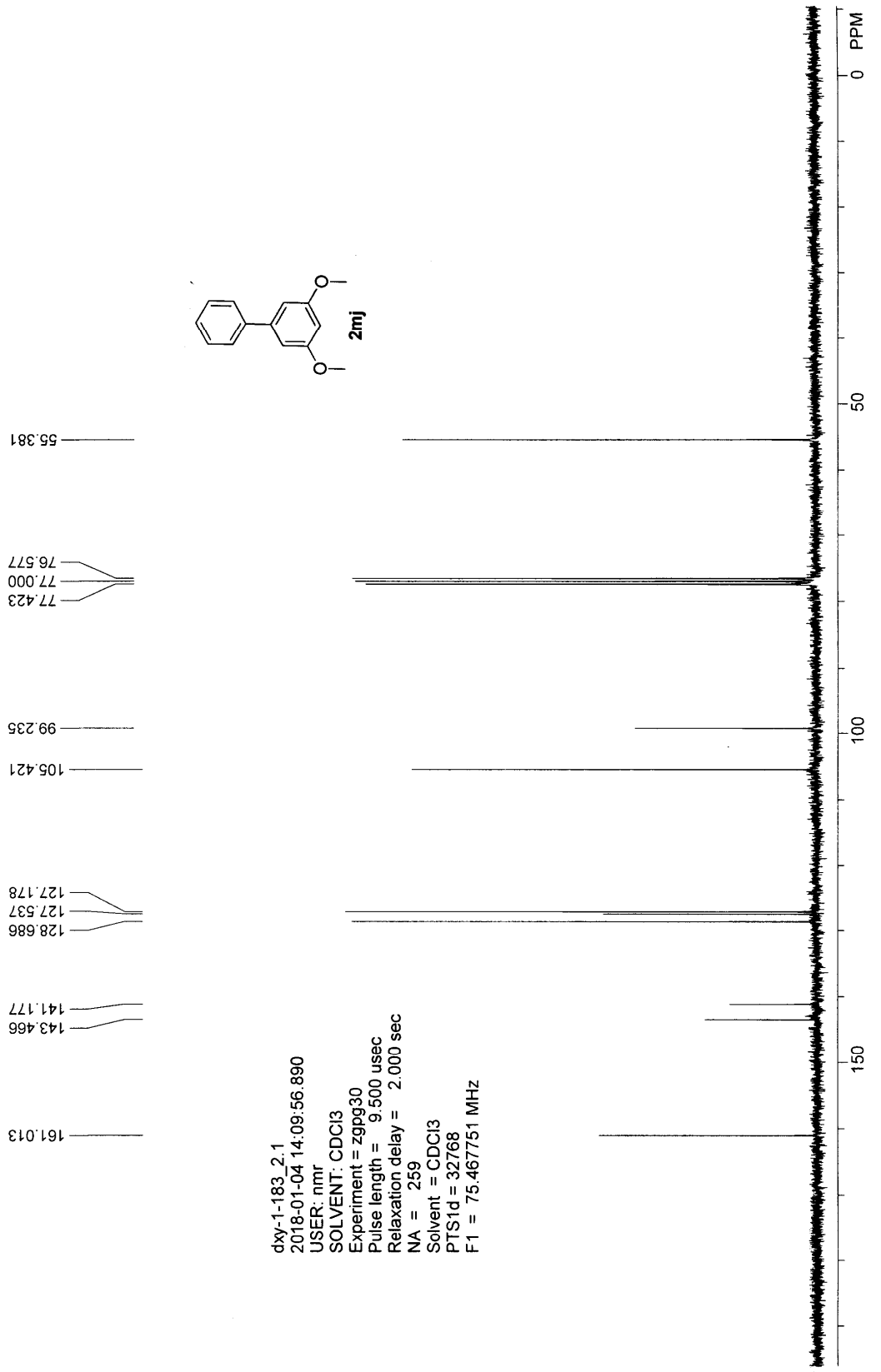

160.967
143.337
138.236
137.298
129.366
126.958
105.182
98.932
77.423
77.000
76.568
55.289
21.031
dxy-2-035
2018-03-16 11:22:19.296
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 176
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
O
O
2ij
The reaction with $K_3PO_4·3H_2O$ being used in the second step
200
150
100
50
0
PPM

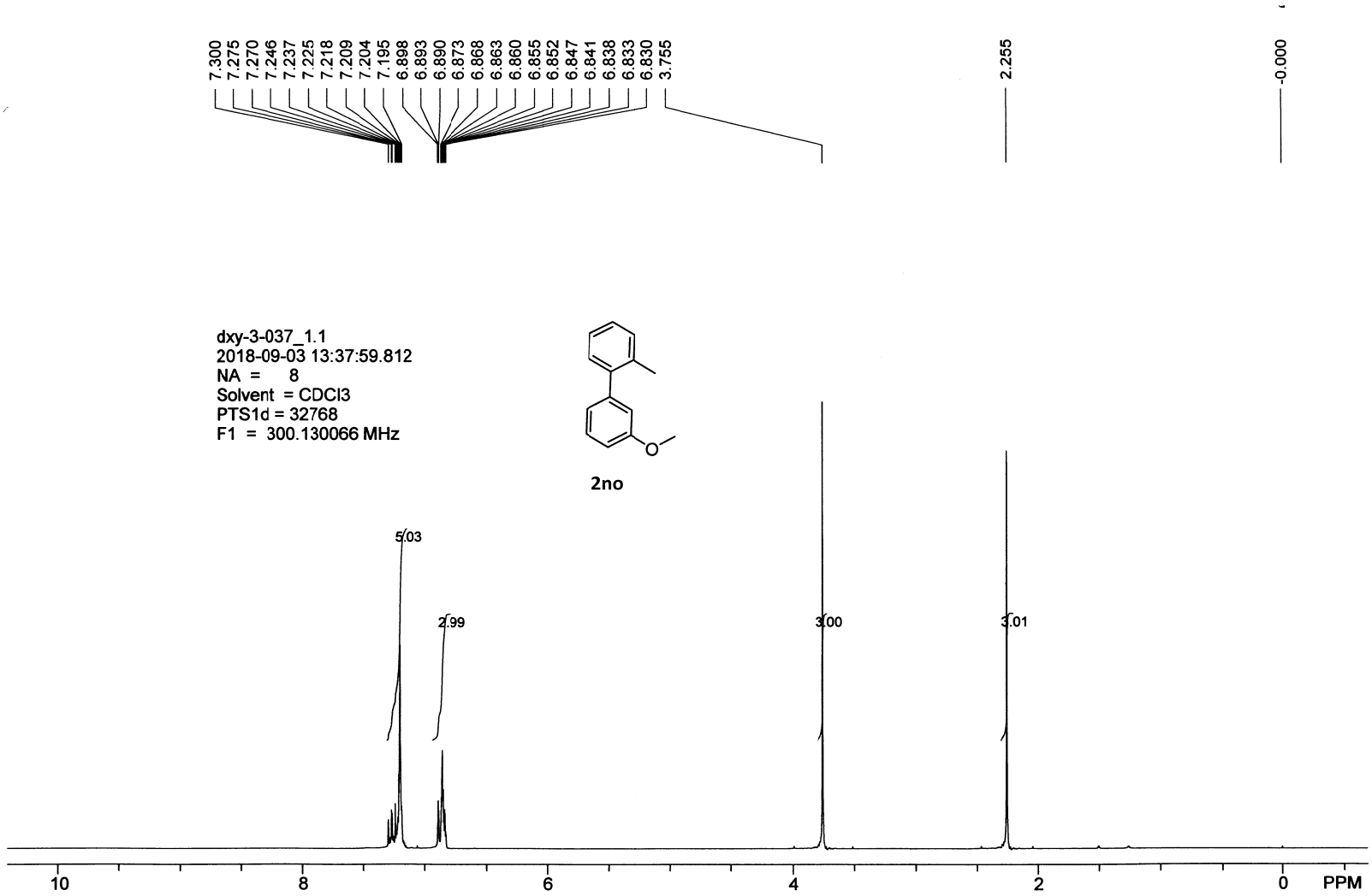

dxy-2-016_1.1
2018-01-26 10:20:19.031
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
2kl
9.00
3.01
2.02
1.97
1.03
2.07
0.99
1.363
0.000
3.862
6.862
6.865
6.871
6.874
6.890
6.893
6.898
6.901
7.119
7.125
7.133
7.166
7.169
7.175
7.191
7.195
7.200
7.253
7.324
7.350
7.376
7.452
7.481
7.526
7.555
PPM

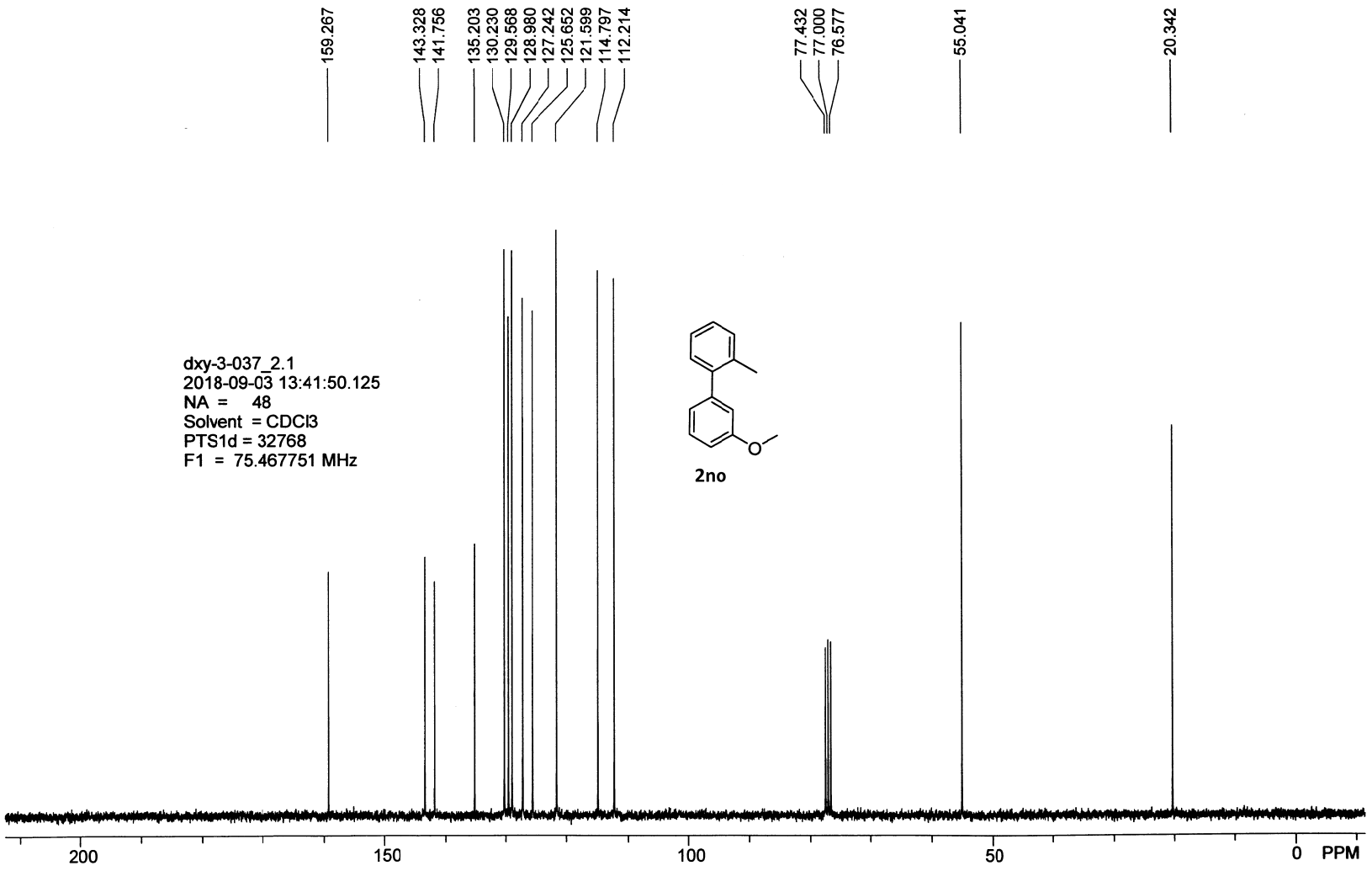

159.818
150.387
142.565
138.144
129.660
126.801
125.671
119.531
112.683
112.333
77.423
77.000
76.577
55.206
34.497
31.326
dxy-2-016
2018-01-29 15:59:18.250
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 101
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
O
2kl
150
100
50
0 PPM

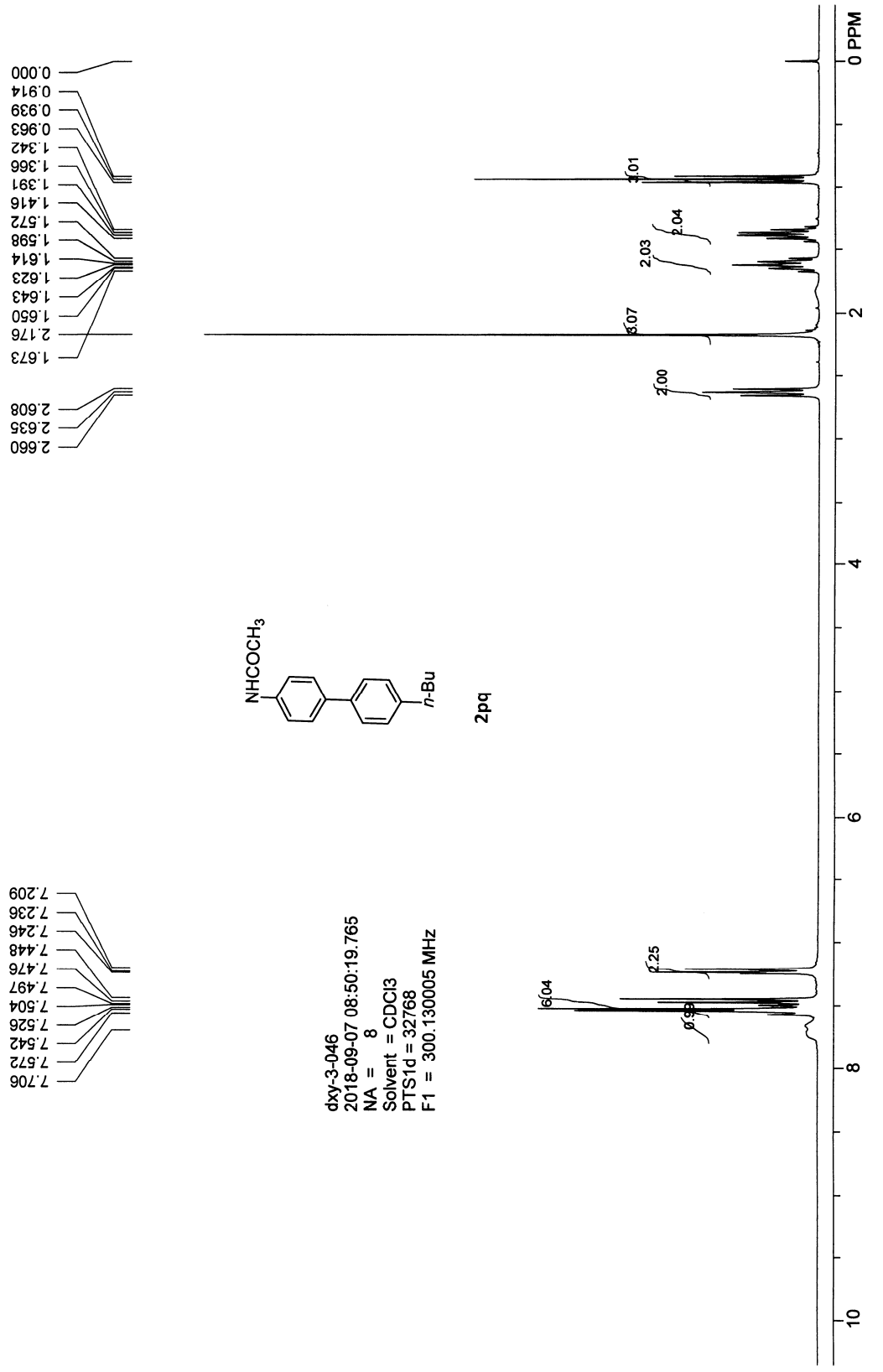

7.548
7.521
7.472
7.445
7.339
7.313
7.190
7.164
7.127
7.124
6.890
6.864
4.904
3.848
1.367
1.357
1.311
-0.000
14 uL CH2Br2 and 0.025g sample
0.025 x Purity x 1000 x 3 / 249.3460 / 0.4 = 19.25 / 250
Purity = 97.3%
dxy-2-016-purity
2018-01-29 16:58:04.765
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
2kl
250.00
190.25
64.85
10
8
6
4
2
0
PPM

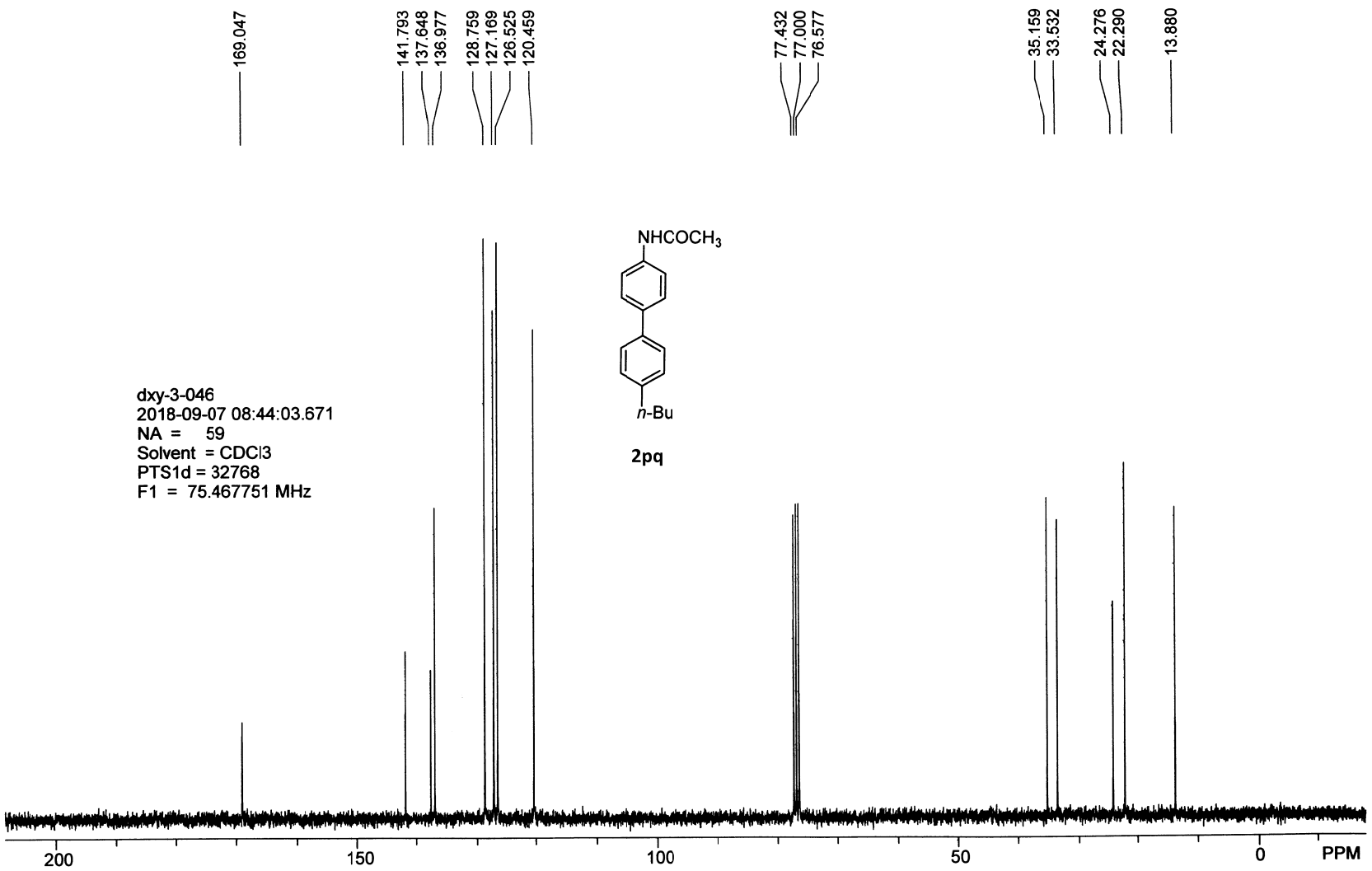

7.597
7.592
7.569
7.566
7.460
7.437
7.431
7.411
7.379
7.355
7.257
6.739
6.731
6.479
6.472
6.464
3.852
-0.000
dxy-1-183
2018-01-04 13:53:17.796
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
2mj
6.00
3.01
2.01
2.00
1.00
10
8
6
4
2
0
PPM

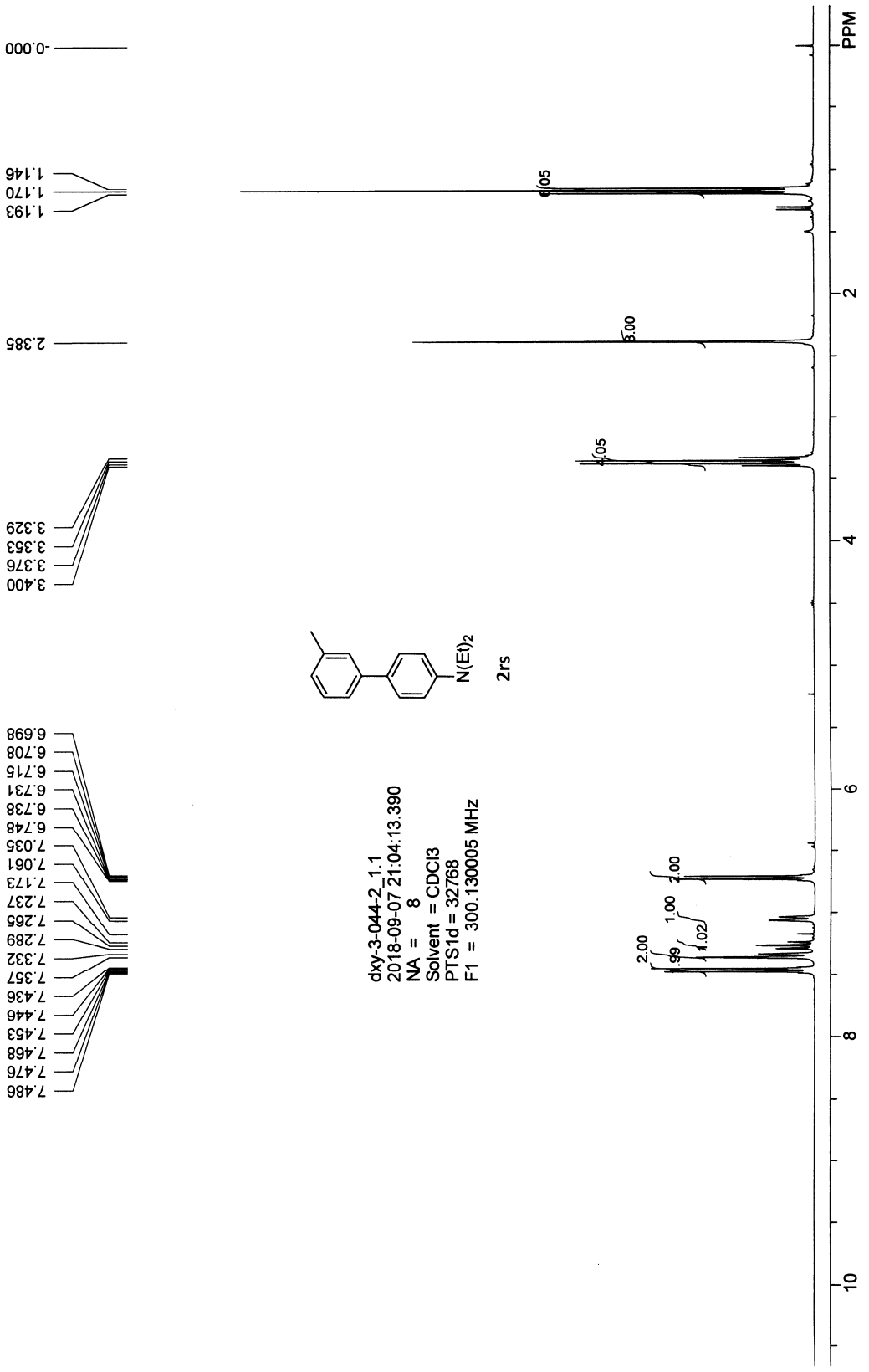

dxy-1-183_2.1
2018-01-04 14:09:56.890
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 259
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
2mj
161.013
143.466
141.177
128.686
127.537
127.178
105.421
99.235
77.423
77.000
76.577
55.381
PPM
0
50
100
150

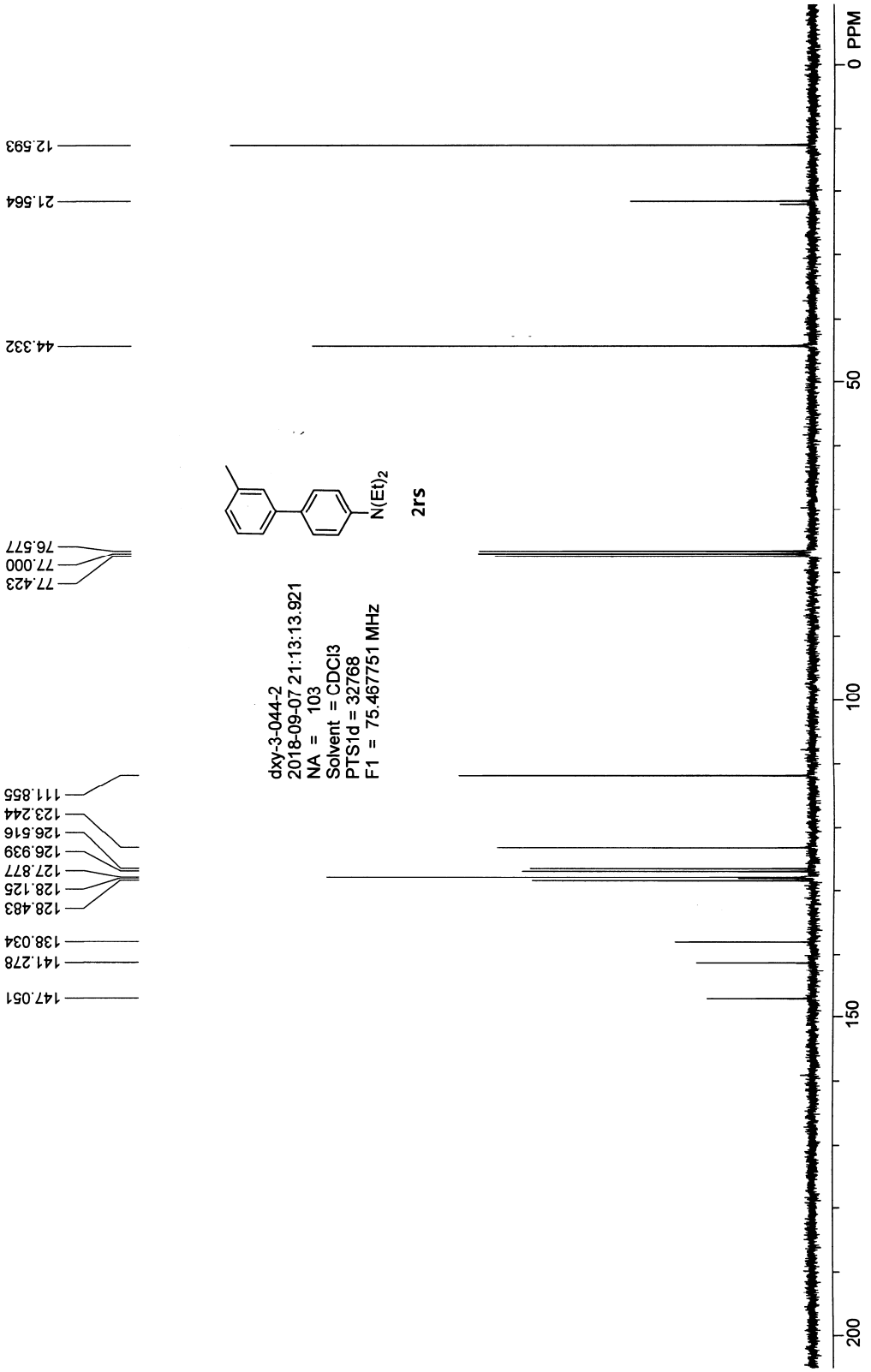

7.300
7.275
7.270
7.246
7.237
7.225
7.218
7.209
7.204
7.195
6.898
6.893
6.890
6.873
6.868
6.863
6.860
6.855
6.852
6.847
6.841
6.838
6.833
6.830
3.755
2.255
-0.000
dxy-3-037_1.1
2018-09-03 13:37:59.812
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130066 MHz
O
2no
5.03
2.99
3.00
3.01
10
8
6
4
2
0
PPM

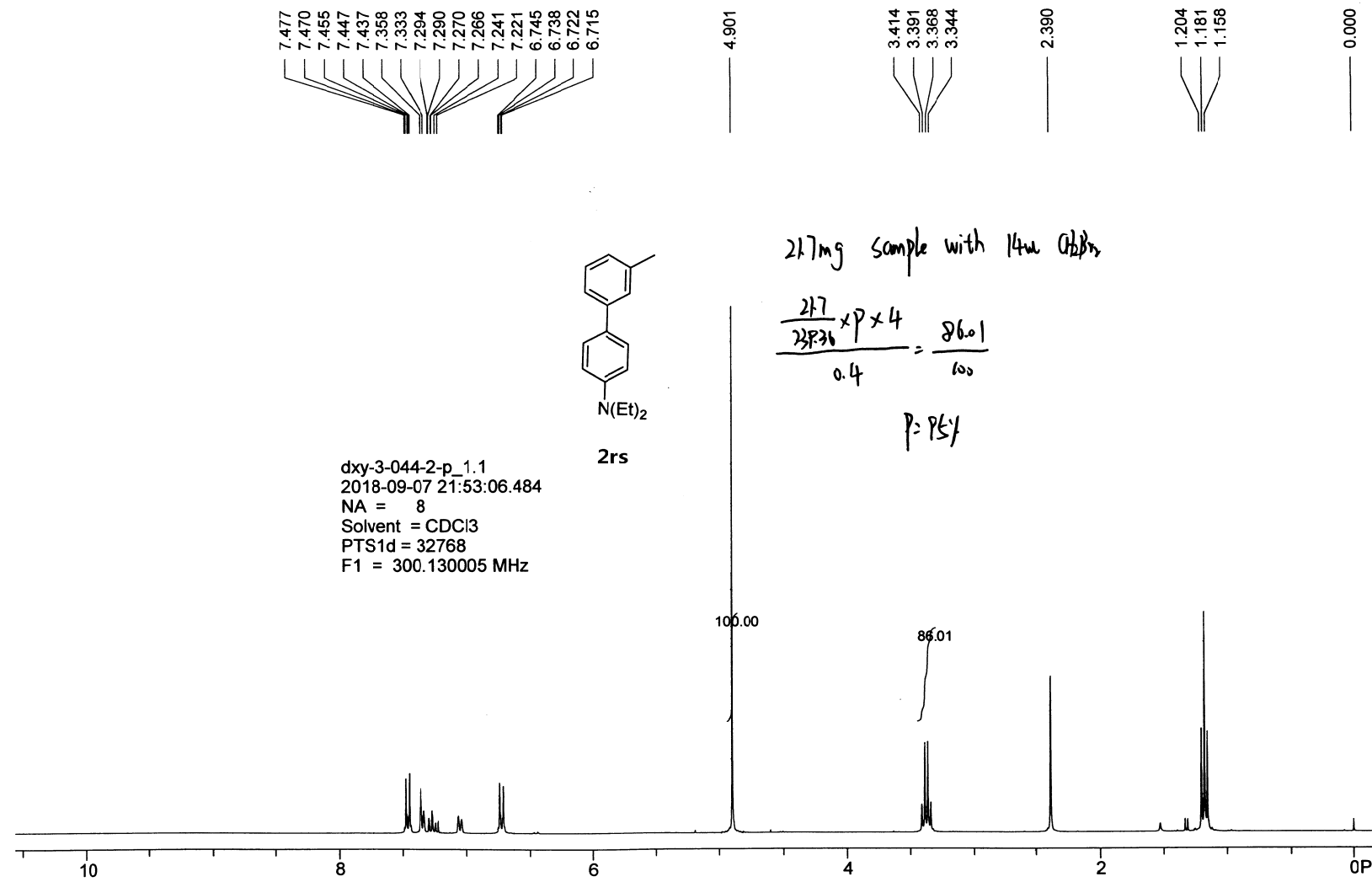

159.267
143.328
141.756
135.203
130.230
129.568
128.980
127.242
125.652
121.599
114.797
112.214
77.432
77.000
76.577
55.041
20.342
dxy-3-037_2.1
2018-09-03 13:41:50.125
NA = 48
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
O
2no
200
150
100
50
0 PPM

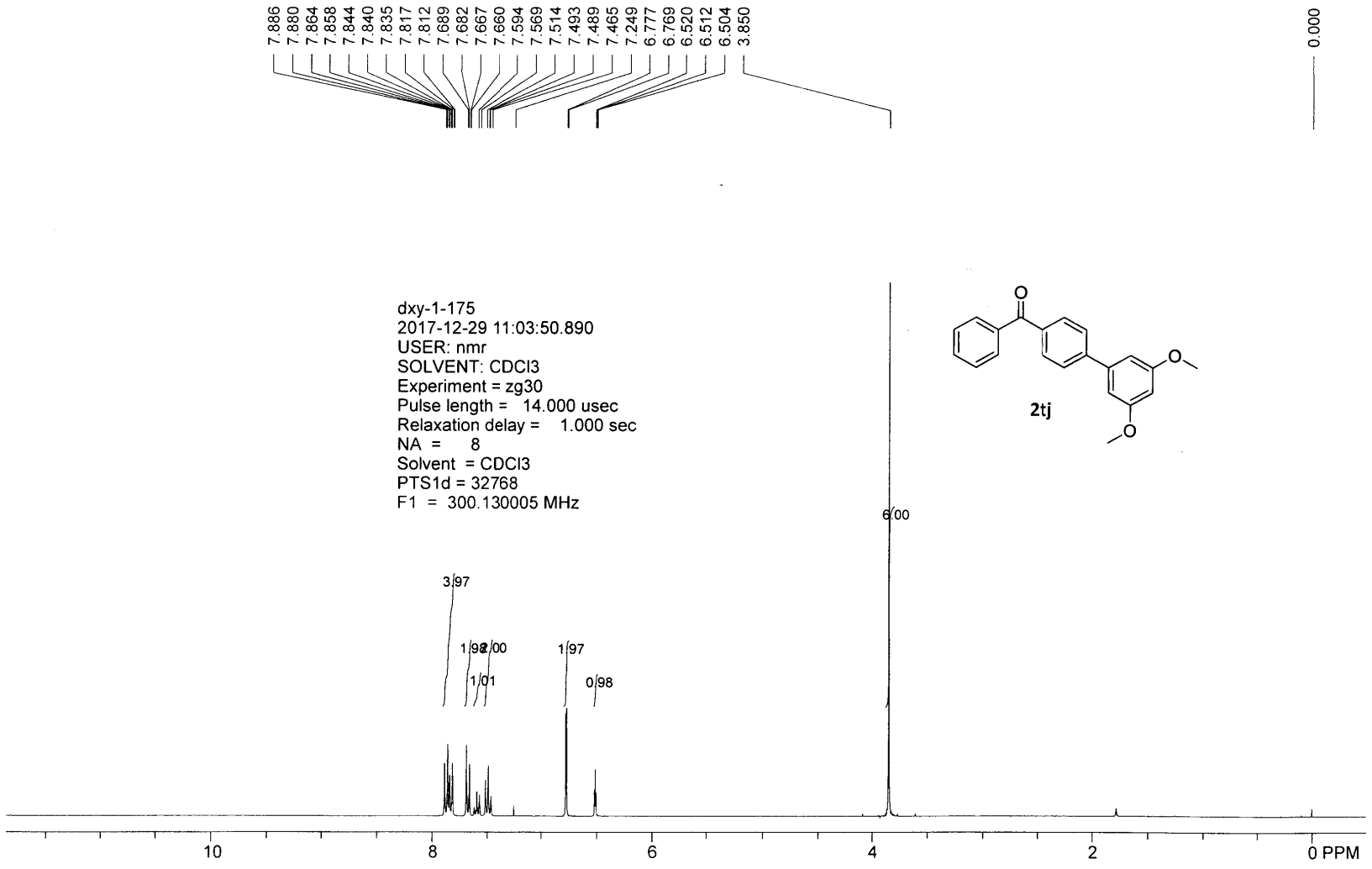

0.000
0.914
0.939
0.963
1.342
1.366
1.391
1.416
1.572
1.598
1.614
1.623
1.643
1.650
2.176
1.673
2.608
2.635
2.660
7.209
7.236
7.246
7.448
7.476
7.497
7.504
7.526
7.542
7.572
7.706
NHCOCH3
n-Bu
2pq
dxy-3-046
2018-09-07 08:50:19.765
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
6.04
2.25
3.01
2.03
2.04
3.07
2.00
0 PPM

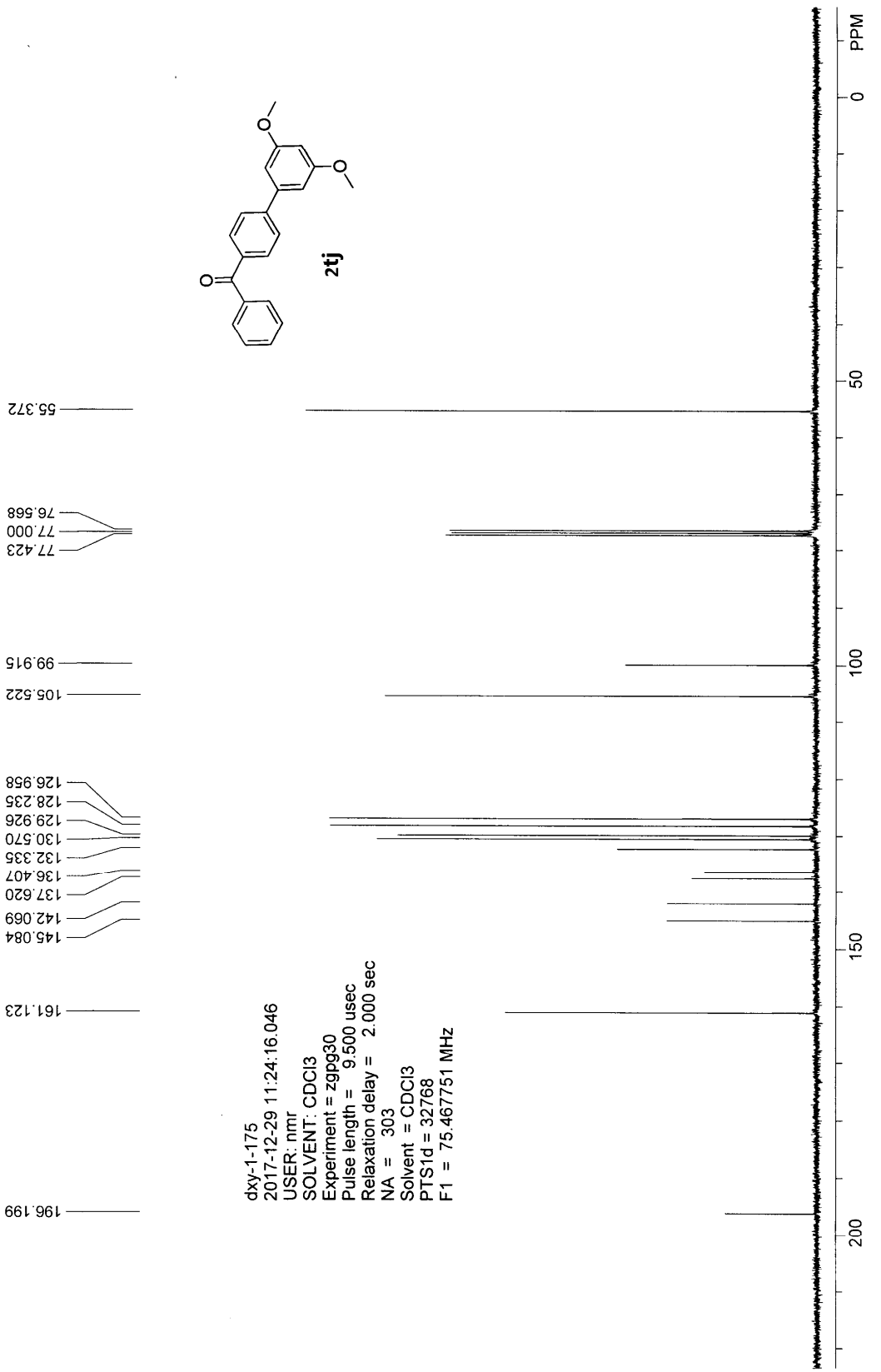

169.047
141.793
137.648
136.977
128.759
127.169
126.525
120.459
77.432
77.000
76.577
35.159
33.532
24.276
22.290
13.880
NHCOCH3
n-Bu
2pq
dxy-3-046
2018-09-07 08:44:03.671
NA = 59
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
200
150
100
50
0
PPM

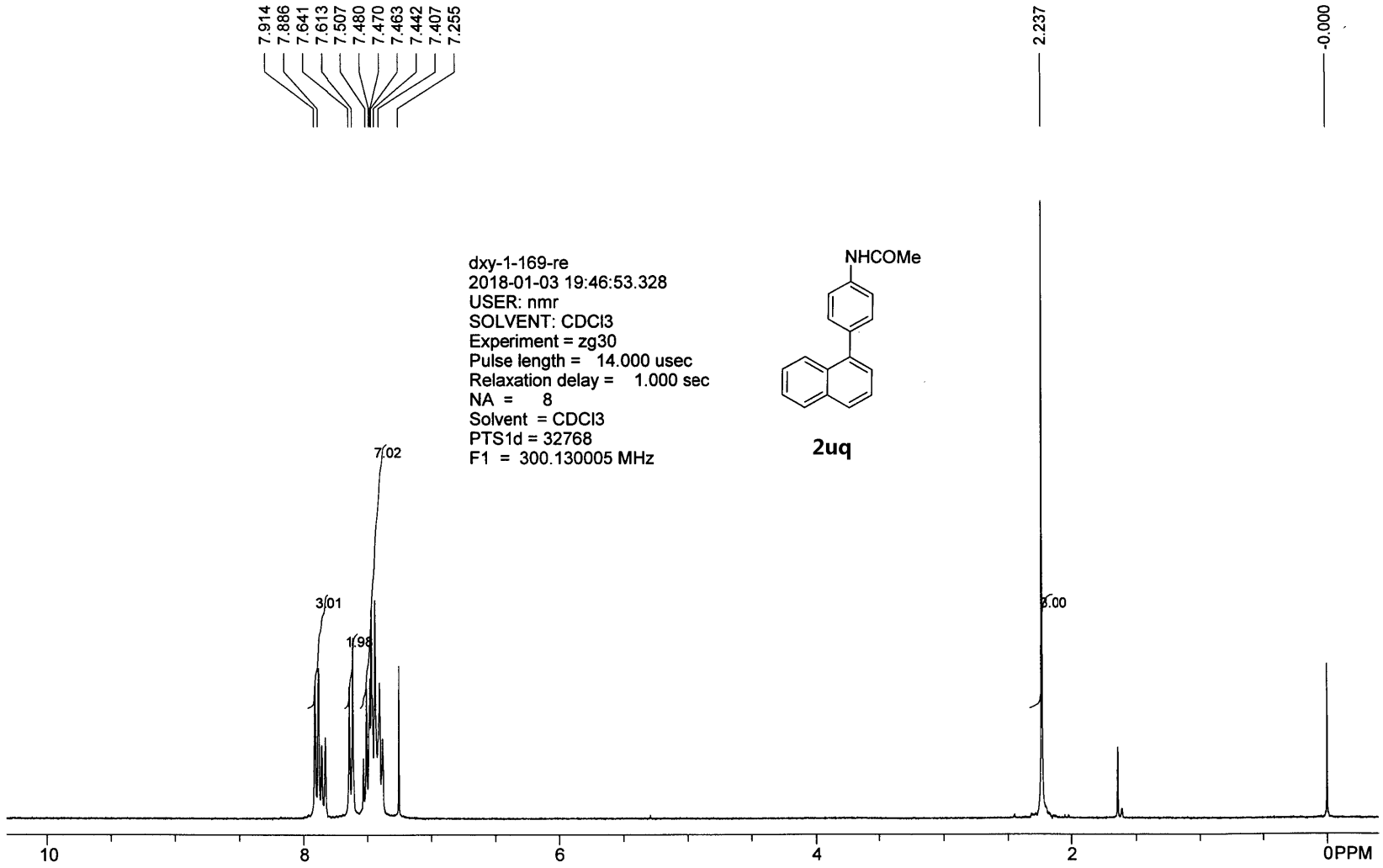

dxy-3-044-2_1.1
2018-09-07 21:04:13.390
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
N(Et)2
2rs
PPM
6.05
3.00
4.05
2.00
1.00
1.02
.99
2.00
-0.000
1.146
1.170
1.193
2.385
3.329
3.353
3.376
3.400
6.698
6.708
6.715
6.731
6.738
6.748
7.035
7.061
7.173
7.237
7.265
7.289
7.332
7.357
7.436
7.446
7.453
7.468
7.476
7.486

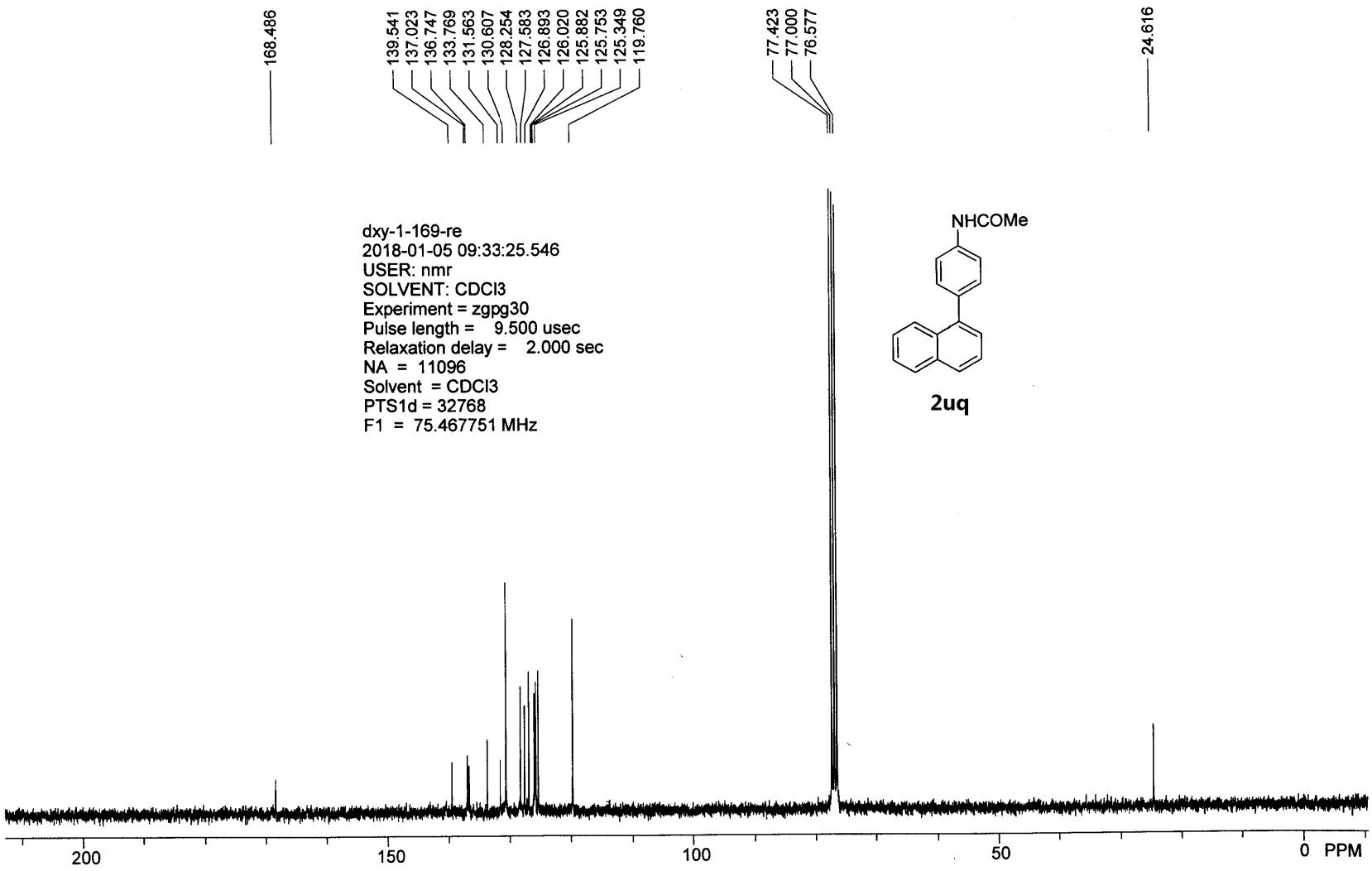

12.593
21.564
44.332
76.577
77.000
77.423
111.855
123.244
126.516
126.939
127.877
128.125
128.483
138.034
141.278
147.051
dxy-3-044-2
2018-09-07 21:13:13.921
NA = 103
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
N(Et)2
2rs
0 PPM
50
100
150
200

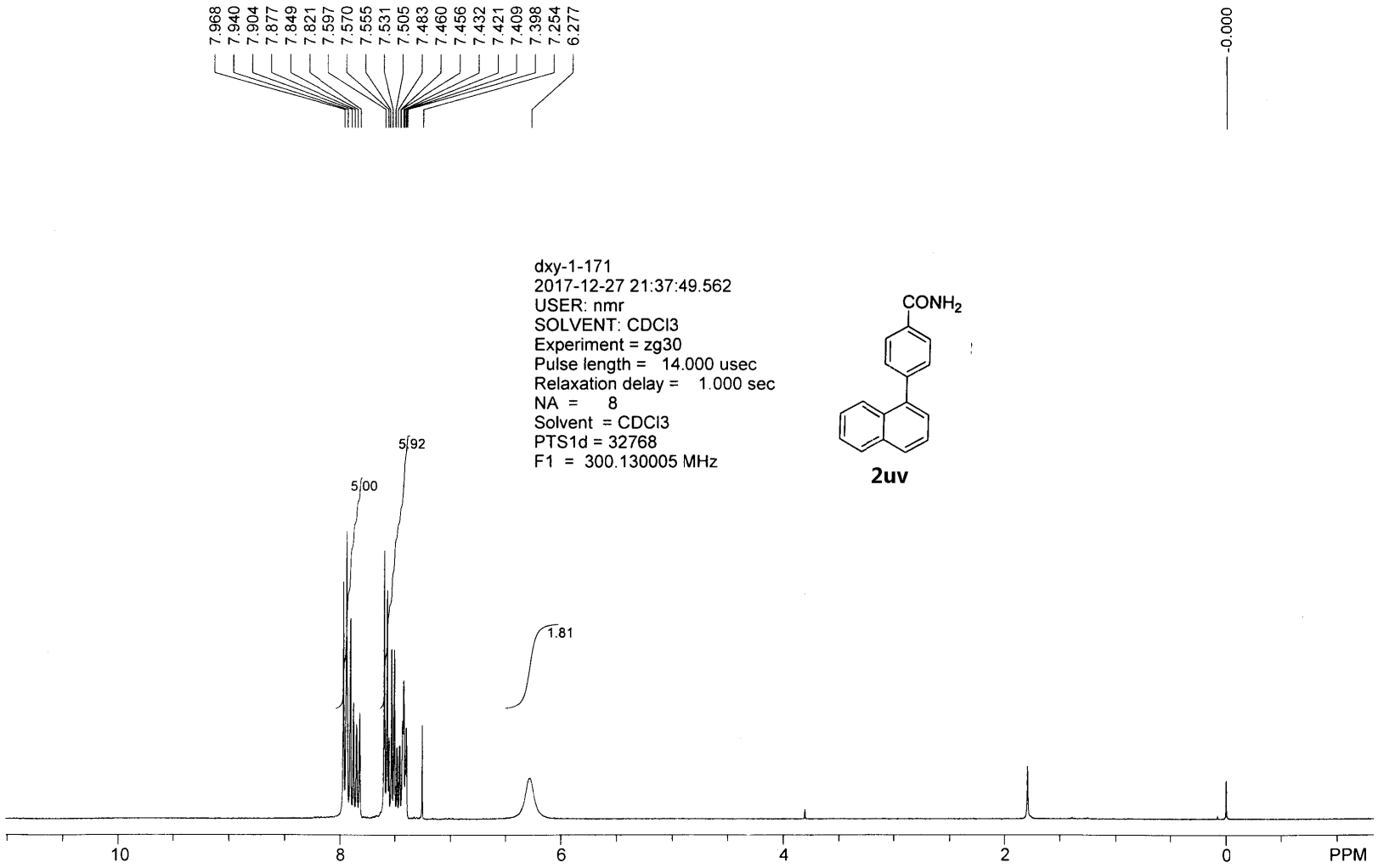

7.477
7.470
7.455
7.447
7.437
7.358
7.333
7.294
7.290
7.270
7.266
7.241
7.221
6.745
6.738
6.722
6.715
4.901
3.414
3.391
3.368
3.344
2.390
1.204
1.181
1.158
0.000
N(Et)2
2rs
217mg sample with 14mm CH2Br2
217/238.36 x P x 4 / 0.4 = 86.01 / 100
P: 95%
dxy-3-044-2-p_1.1
2018-09-07 21:53:06.484
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
100.00
86.01
10
8
6
4
2
0PPM

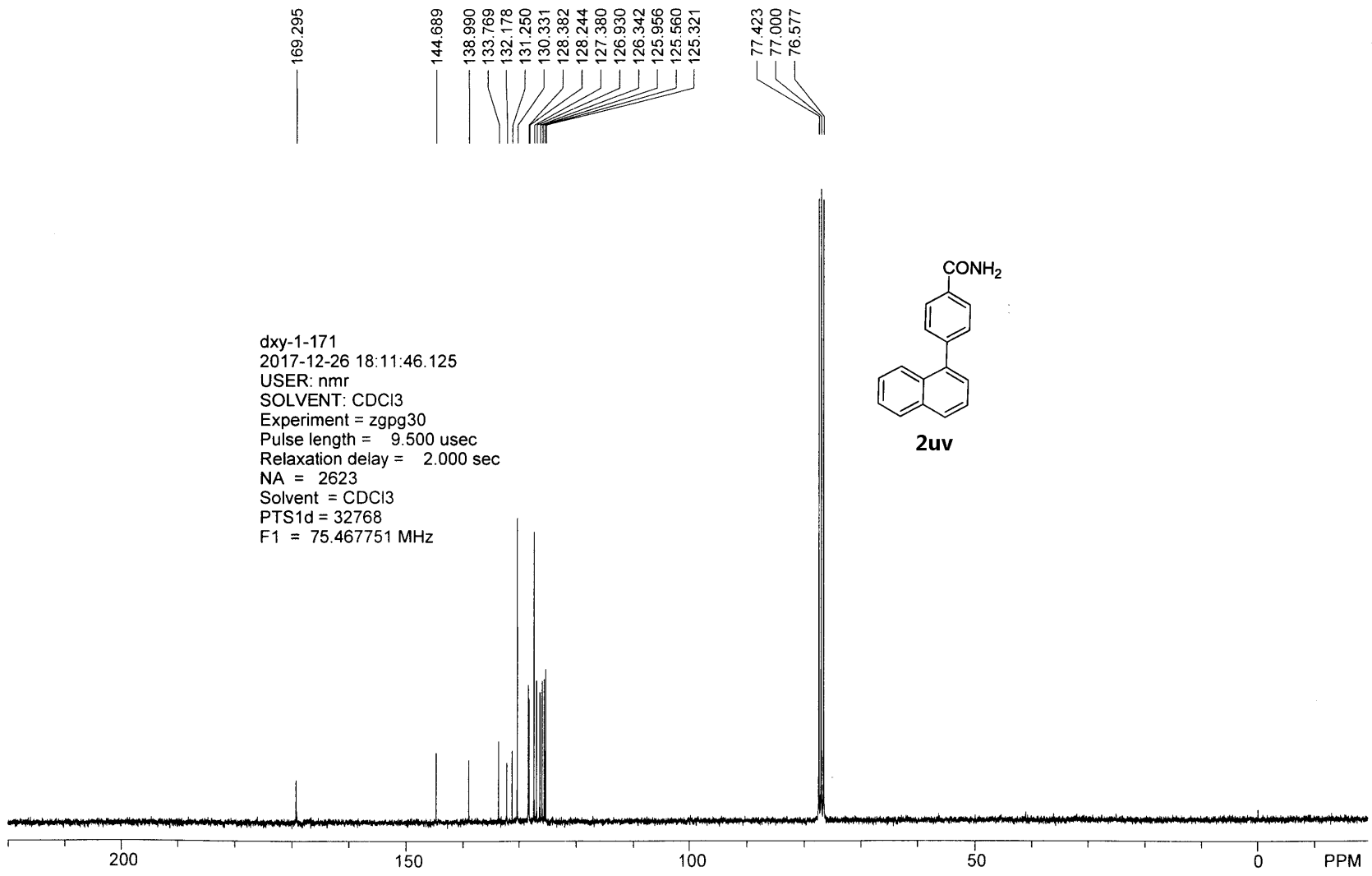

7.886
7.880
7.864
7.858
7.844
7.840
7.835
7.817
7.812
7.689
7.682
7.667
7.660
7.594
7.569
7.514
7.493
7.489
7.465
7.249
6.777
6.769
6.520
6.512
6.504
3.850
0.000
dxy-1-175
2017-12-29 11:03:50.890
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
2tj
6.00
3.97
1.98
2.00
1.01
1.97
0.98
10
8
6
4
2
0 PPM

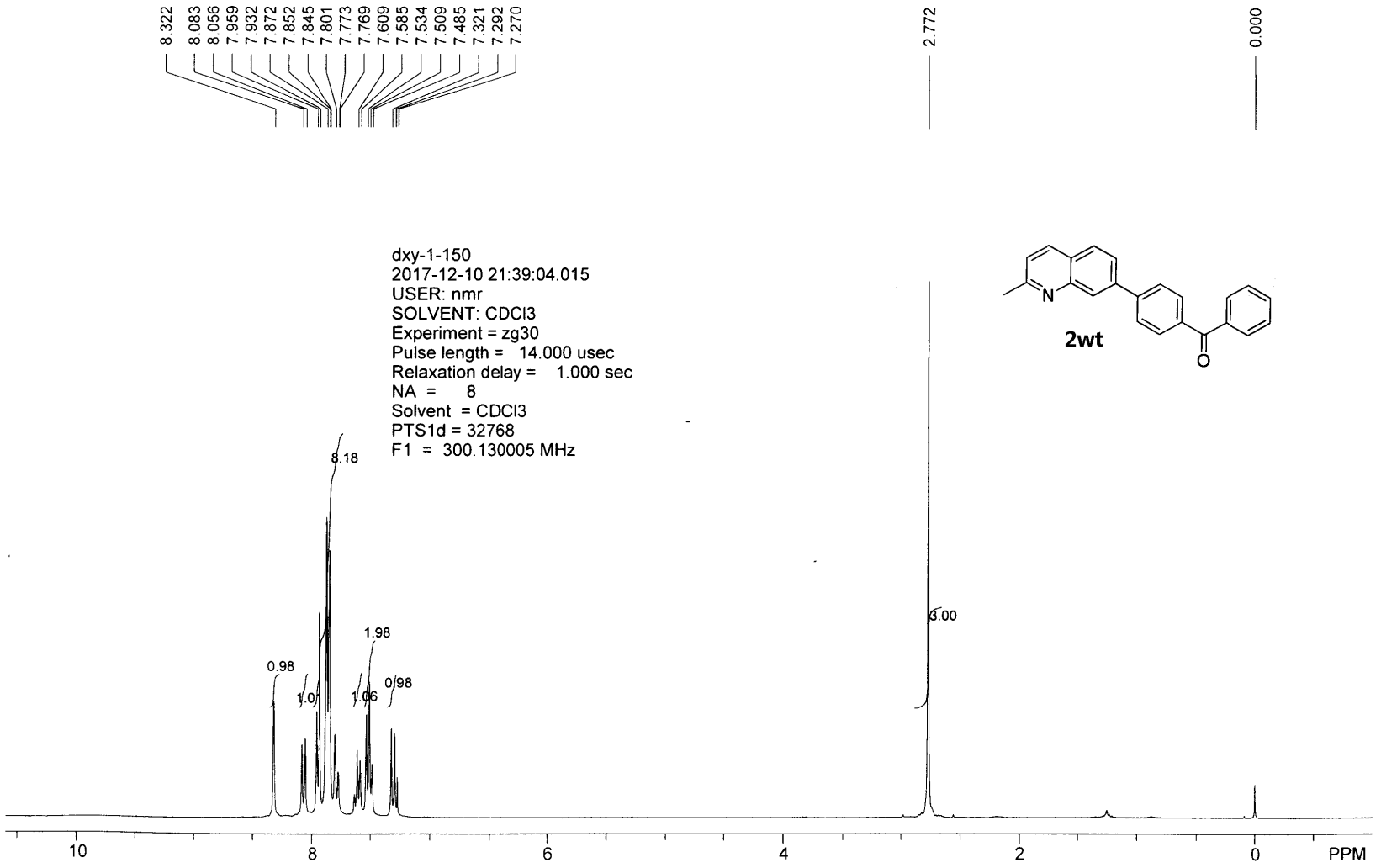

2tj
55.372
76.568
77.000
77.423
99.915
105.522
126.958
128.235
129.926
130.570
132.335
136.407
137.620
142.069
145.084
161.123
196.199
dxy-1-175
2017-12-29 11:24:16.046
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 303
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
PPM
0
50
100
150
200

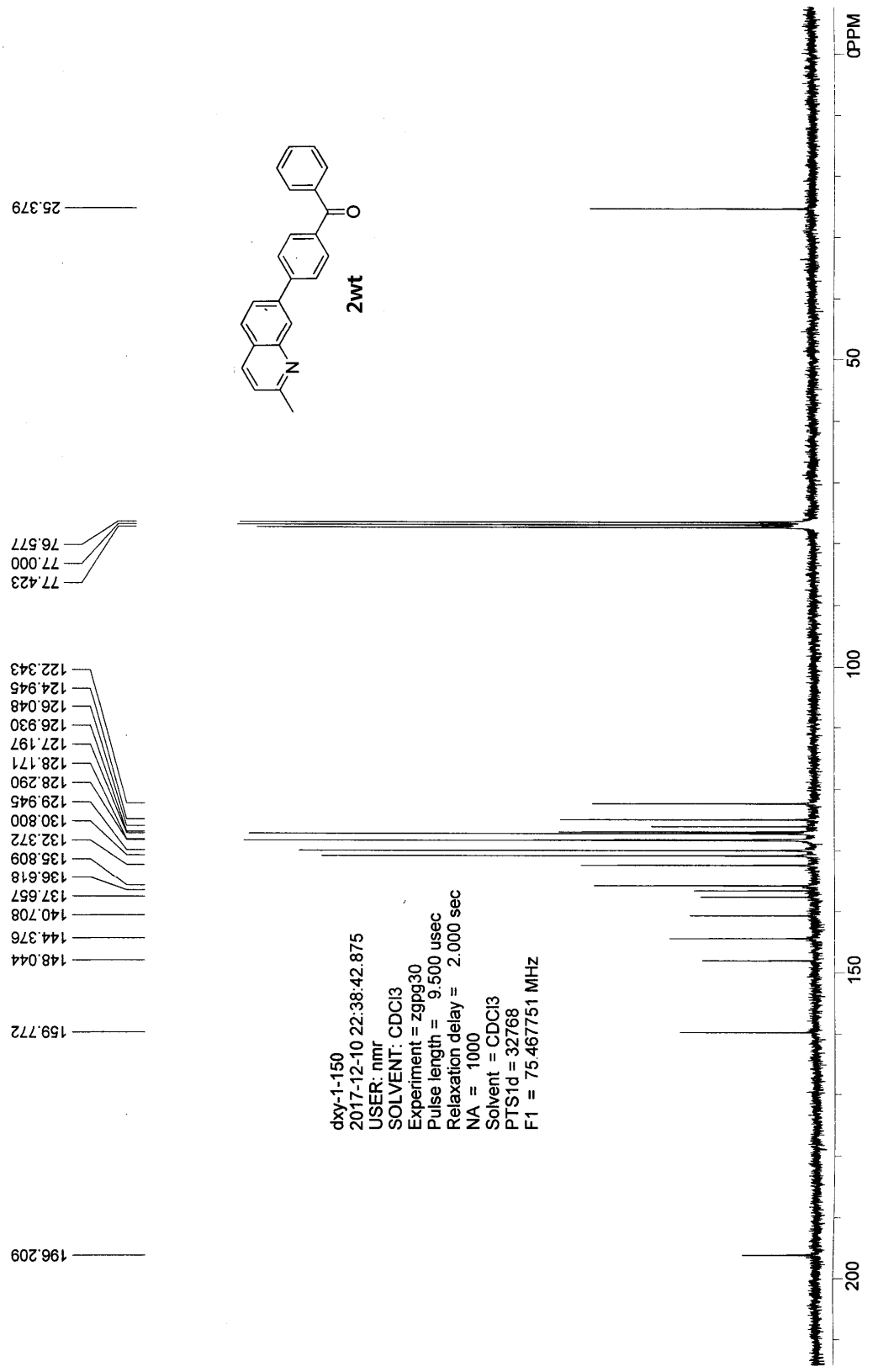

7.914
7.886
7.641
7.613
7.507
7.480
7.470
7.463
7.442
7.407
7.255
2.237
-0.000
dxy-1-169-re
2018-01-03 19:46:53.328
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
NHCOMe
2uq
7.02
3.01
1.98
3.00
10
8
6
4
2
0PPM

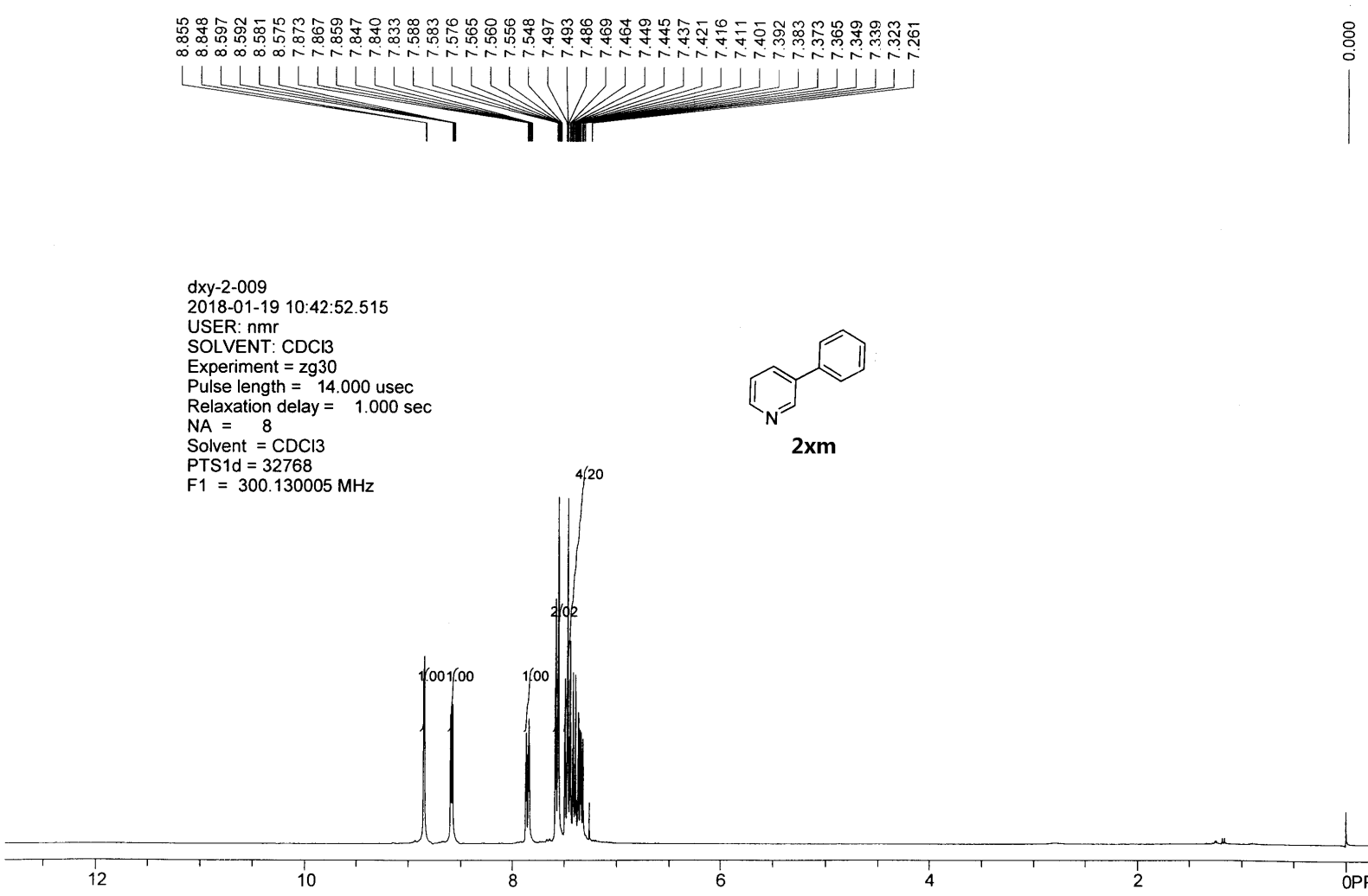

168.486
139.541
137.023
136.747
133.769
131.563
130.607
128.254
127.583
126.893
126.020
125.882
125.753
125.349
119.760
77.423
77.000
76.577
24.616
dxy-1-169-re
2018-01-05 09:33:25.546
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 11096
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
NHCOMe
2uq
200
150
100
50
0 PPM

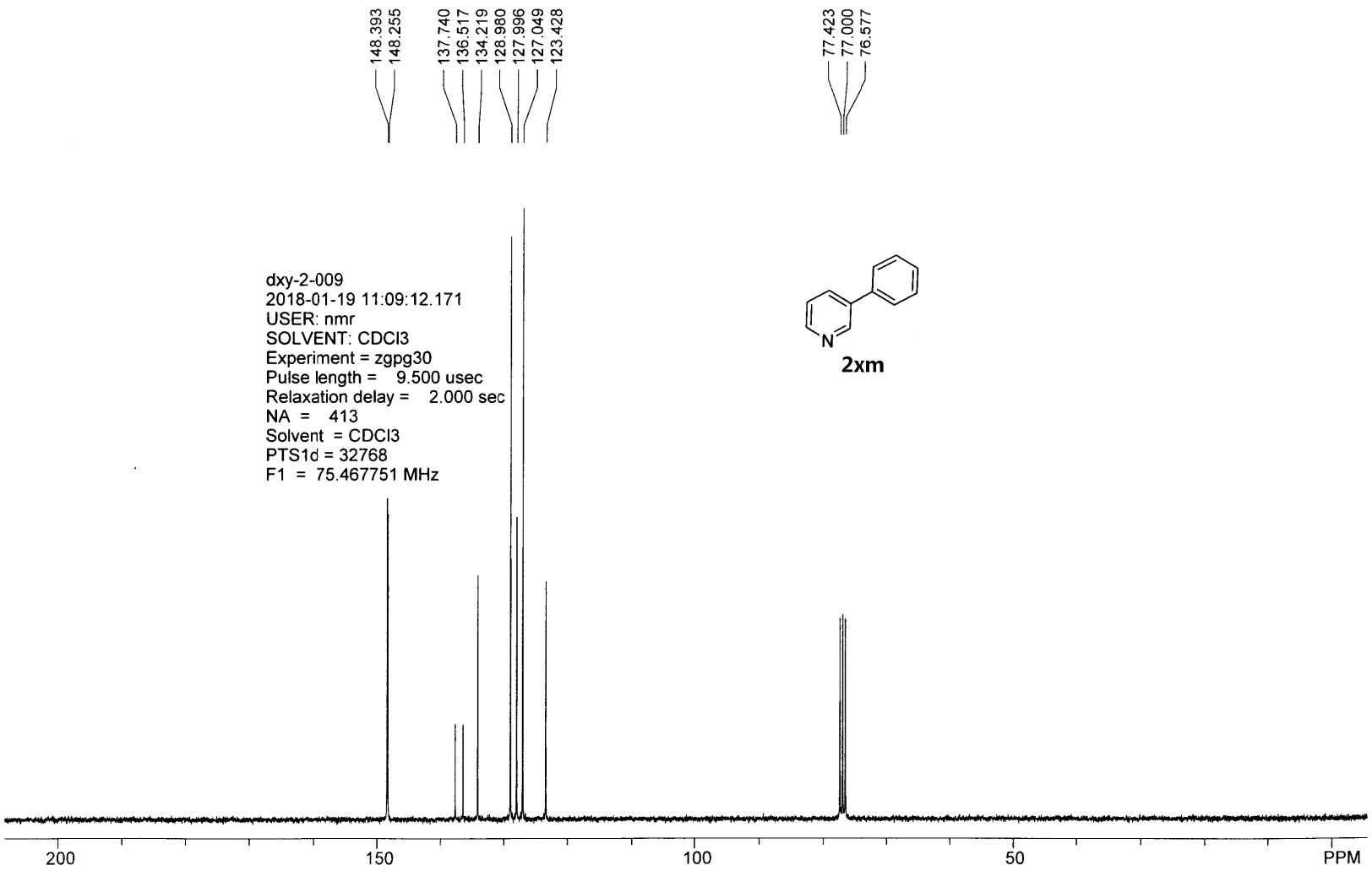

7.968
7.940
7.904
7.877
7.849
7.821
7.597
7.570
7.555
7.531
7.505
7.483
7.460
7.456
7.432
7.421
7.409
7.398
7.254
6.277
-0.000
dxy-1-171
2017-12-27 21:37:49.562
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
CONH2
2uv
5.00
5.92
1.81
10
8
6
4
2
0
PPM

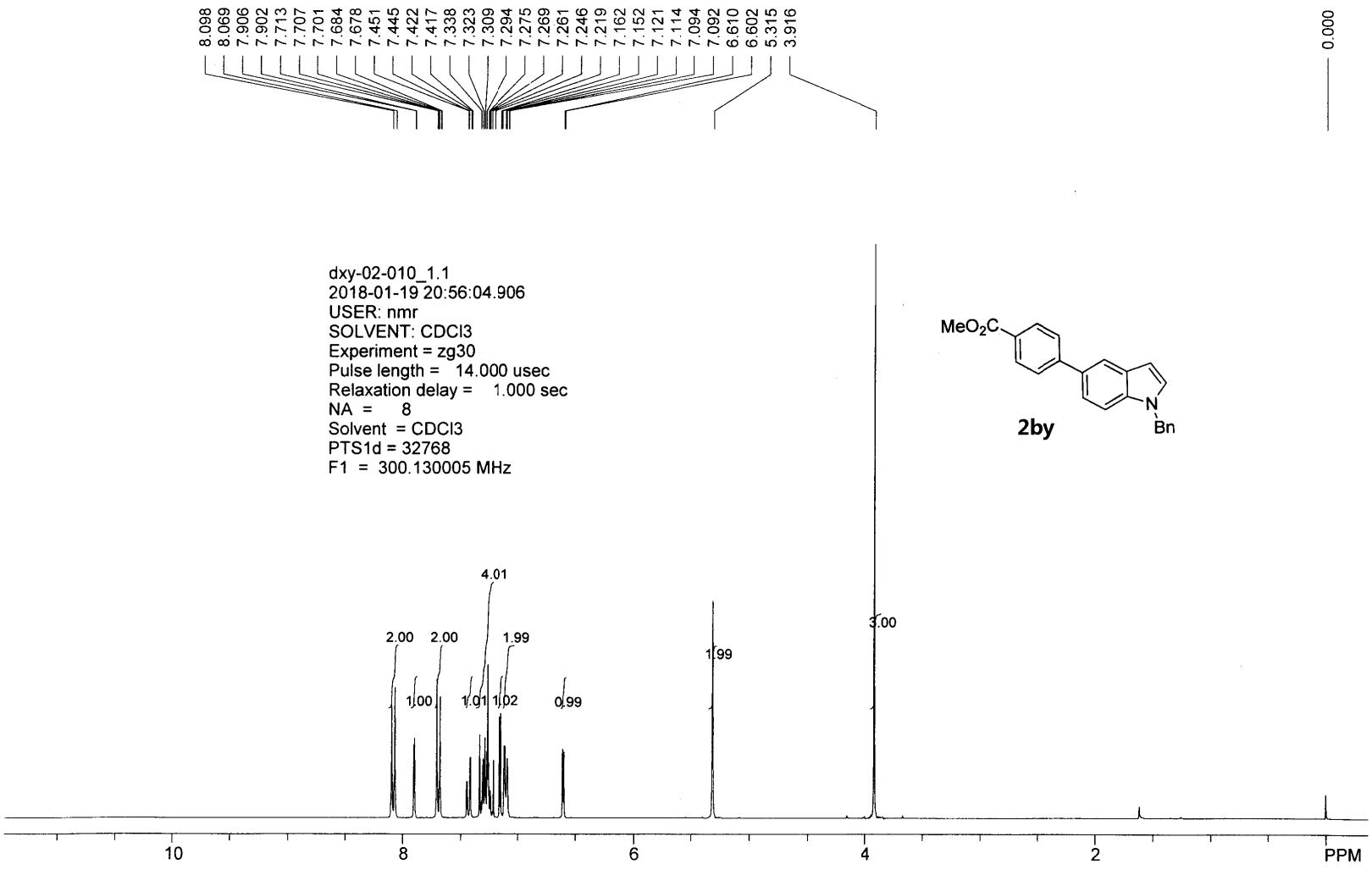

169.295
144.689
138.990
133.769
132.178
131.250
130.331
128.382
128.244
127.380
126.930
126.342
125.956
125.560
125.321
77.423
77.000
76.577
dxy-1-171
2017-12-26 18:11:46.125
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 2623
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
CONH2
2uv
200
150
100
50
0
PPM

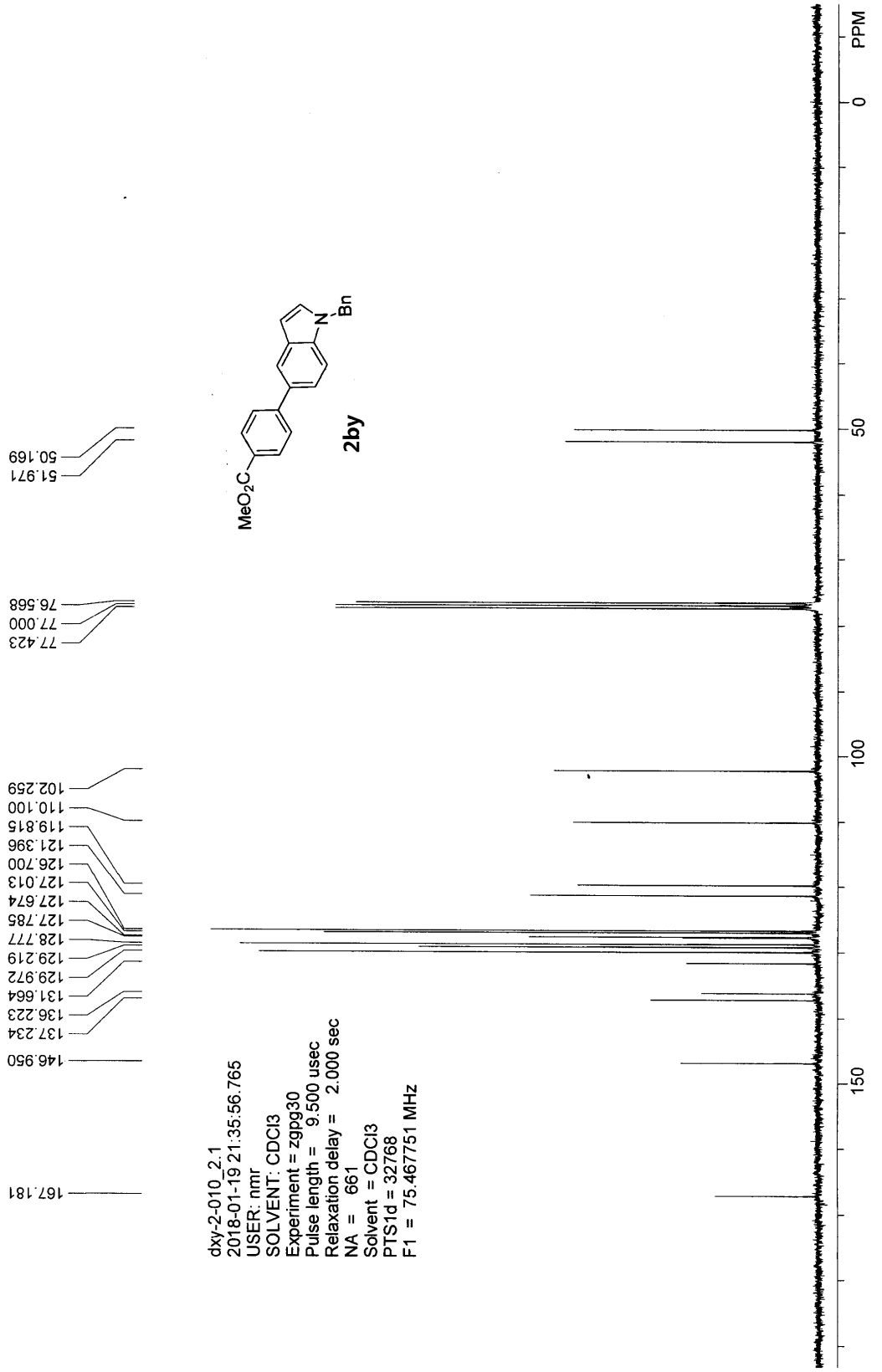

8.322
8.083
8.056
7.959
7.932
7.872
7.852
7.845
7.801
7.773
7.769
7.609
7.585
7.534
7.509
7.485
7.321
7.292
7.270
2.772
0.000
dxy-1-150
2017-12-10 21:39:04.015
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
2wt
N
O
8.18
0.98
1.0
1.98
1.06
0.98
6.00
10
8
6
4
2
0
PPM

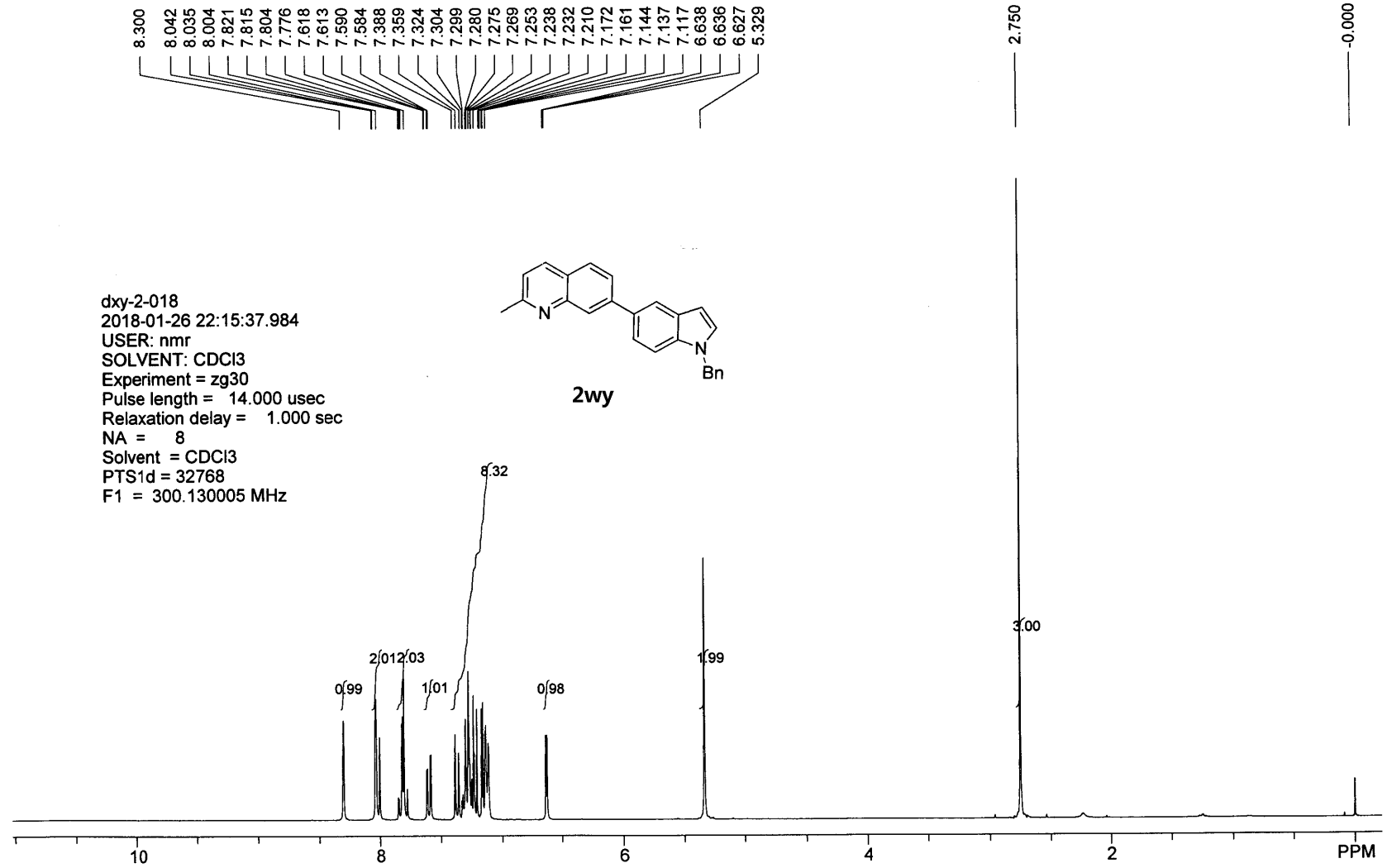

25.379
76.577
77.000
77.423
122.343
124.945
126.048
126.930
127.197
128.171
128.290
129.945
130.800
132.372
135.809
136.618
137.657
140.708
144.376
148.044
159.772
196.209
dxy-1-150
2017-12-10 22:38:42.875
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 1000
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
2wt
O
N
200
150
100
50
PPM

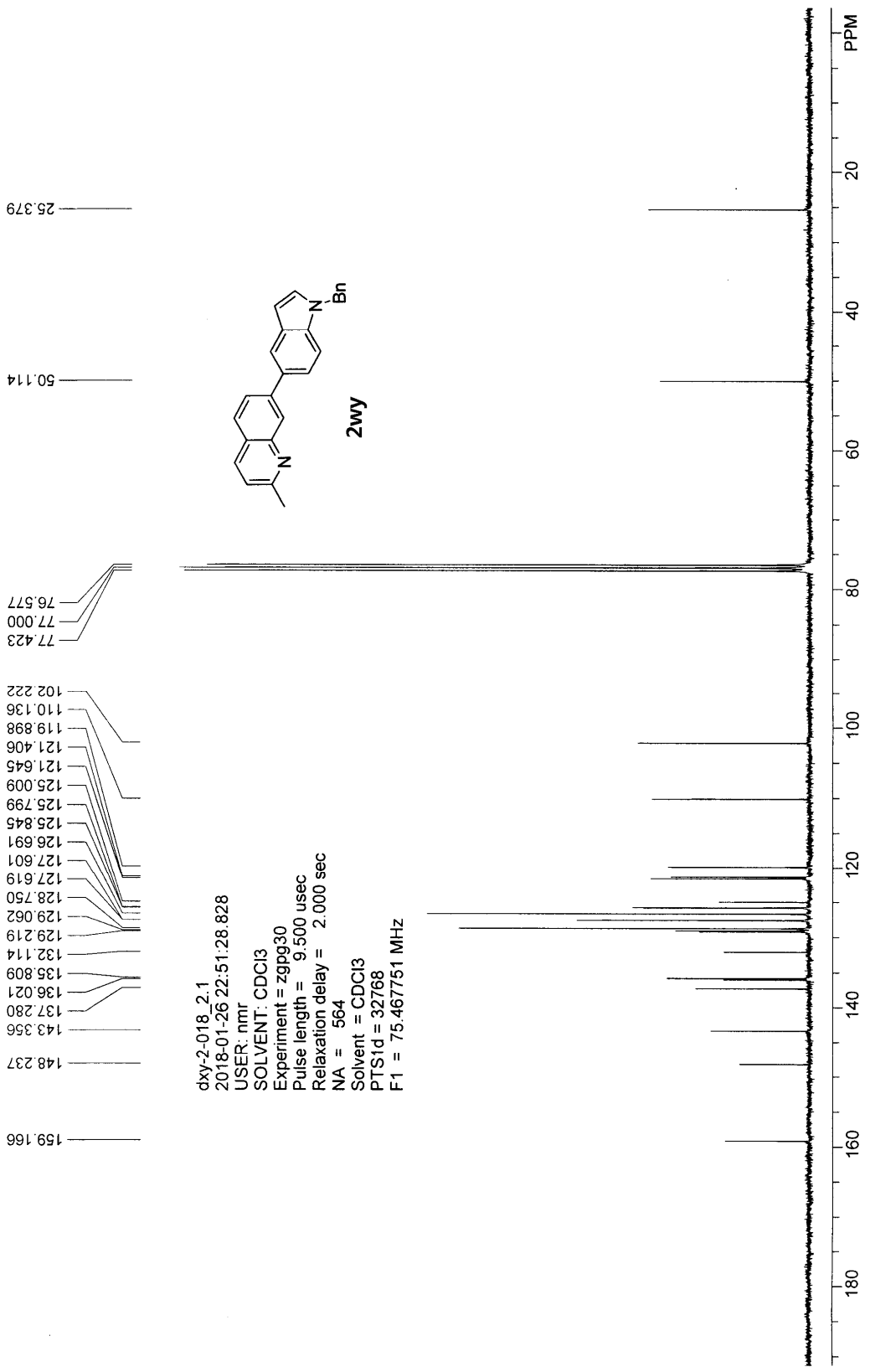

8.855
8.848
8.597
8.592
8.581
8.575
7.873
7.867
7.859
7.847
7.840
7.833
7.588
7.583
7.576
7.565
7.560
7.556
7.548
7.497
7.493
7.486
7.469
7.464
7.449
7.445
7.437
7.421
7.416
7.411
7.401
7.392
7.383
7.373
7.365
7.349
7.339
7.323
7.261
0.000
dxy-2-009
2018-01-19 10:42:52.515
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
N
2xm
4.20
2.02
1.00
1.00
1.00
12
10
8
6
4
2
0PPM

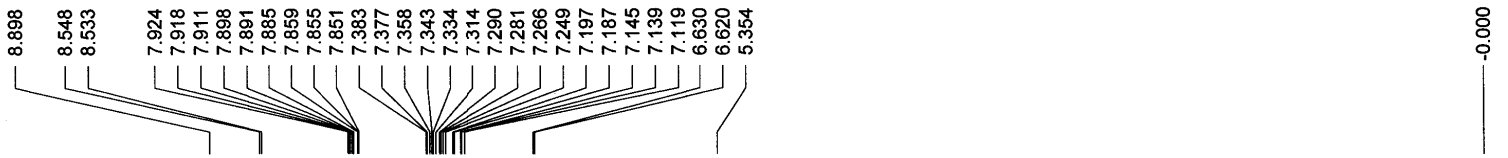

148.393
148.255
137.740
136.517
134.219
128.980
127.996
127.049
123.428
77.423
77.000
76.577
dxy-2-009
2018-01-19 11:09:12.171
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 413
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
N
2xm
200
150
100
50
PPM


8.098
8.069
7.906
7.902
7.713
7.707
7.701
7.684
7.678
7.451
7.445
7.422
7.417
7.338
7.323
7.309
7.294
7.275
7.269
7.261
7.246
7.219
7.162
7.152
7.121
7.114
7.094
7.092
6.610
6.602
5.315
3.916
0.000
dxy-02-010_1.1
2018-01-19 20:56:04.906
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
MeO2C
N
Bn
2by
4.01
2.00
2.00
1.99
3.00
1.99
1.00
1.01
1.02
0.99
10
8
6
4
2
PPM

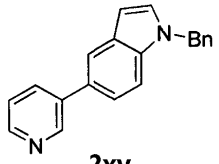

dxy-2-010_2.1
2018-01-19 21:35:56.765
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 661
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
167.181
146.950
137.234
136.223
131.664
129.972
129.219
128.777
127.785
127.674
127.013
126.700
121.396
119.815
110.100
102.259
77.423
77.000
76.568
51.971
50.169
MeO2C
N
Bn
2by
PPM
0
50
100
150


8.300
8.042
8.035
8.004
7.821
7.815
7.804
7.776
7.618
7.613
7.590
7.584
7.388
7.359
7.324
7.304
7.299
7.280
7.275
7.269
7.253
7.238
7.232
7.210
7.172
7.161
7.144
7.137
7.117
6.638
6.636
6.627
5.329
2.750
-0.000
dxy-2-018
2018-01-26 22:15:37.984
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
N
N
Bn
2wy
8.32
0.99
2.01
2.03
1.01
0.98
1.99
3.00
10
8
6
4
2
PPM

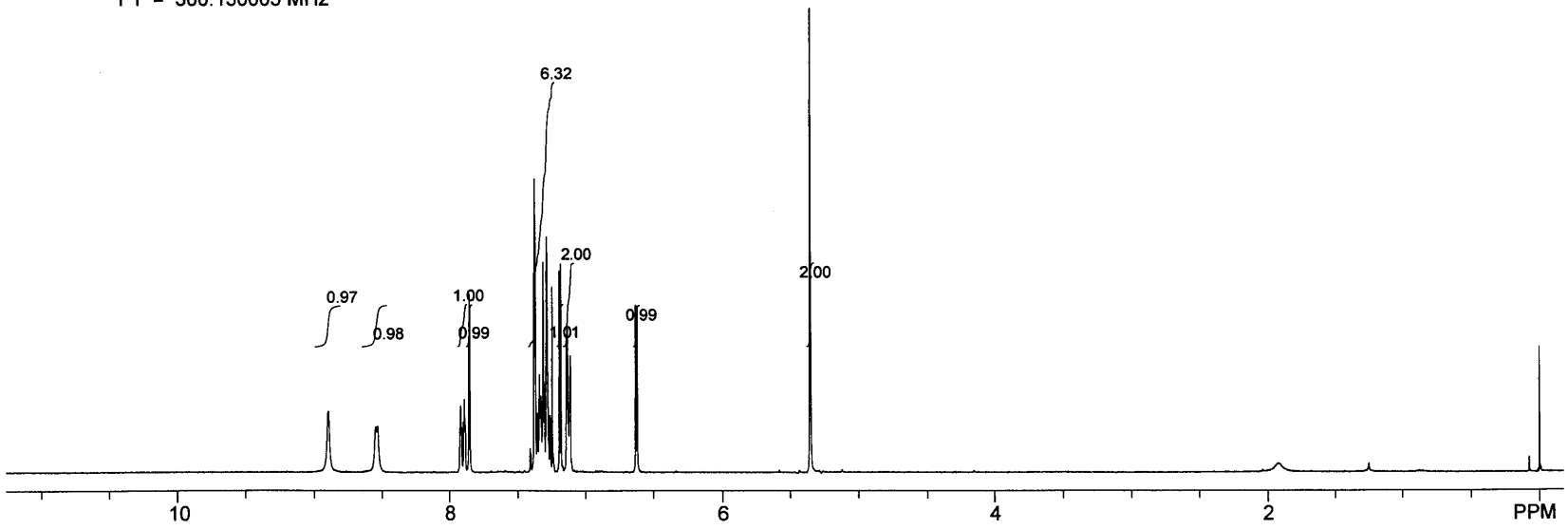

dxy-2-018_2.1
2018-01-26 22:51:28.828
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 564
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
2wy
Bn
N
N
159.166
148.237
143.356
137.280
136.021
135.809
132.114
129.219
129.062
128.750
127.619
127.601
126.691
125.845
125.799
125.009
121.645
121.406
119.898
110.136
102.222
77.423
77.000
76.577
50.114
25.379
PPM
20
40
60
80
100
120
140
160
180

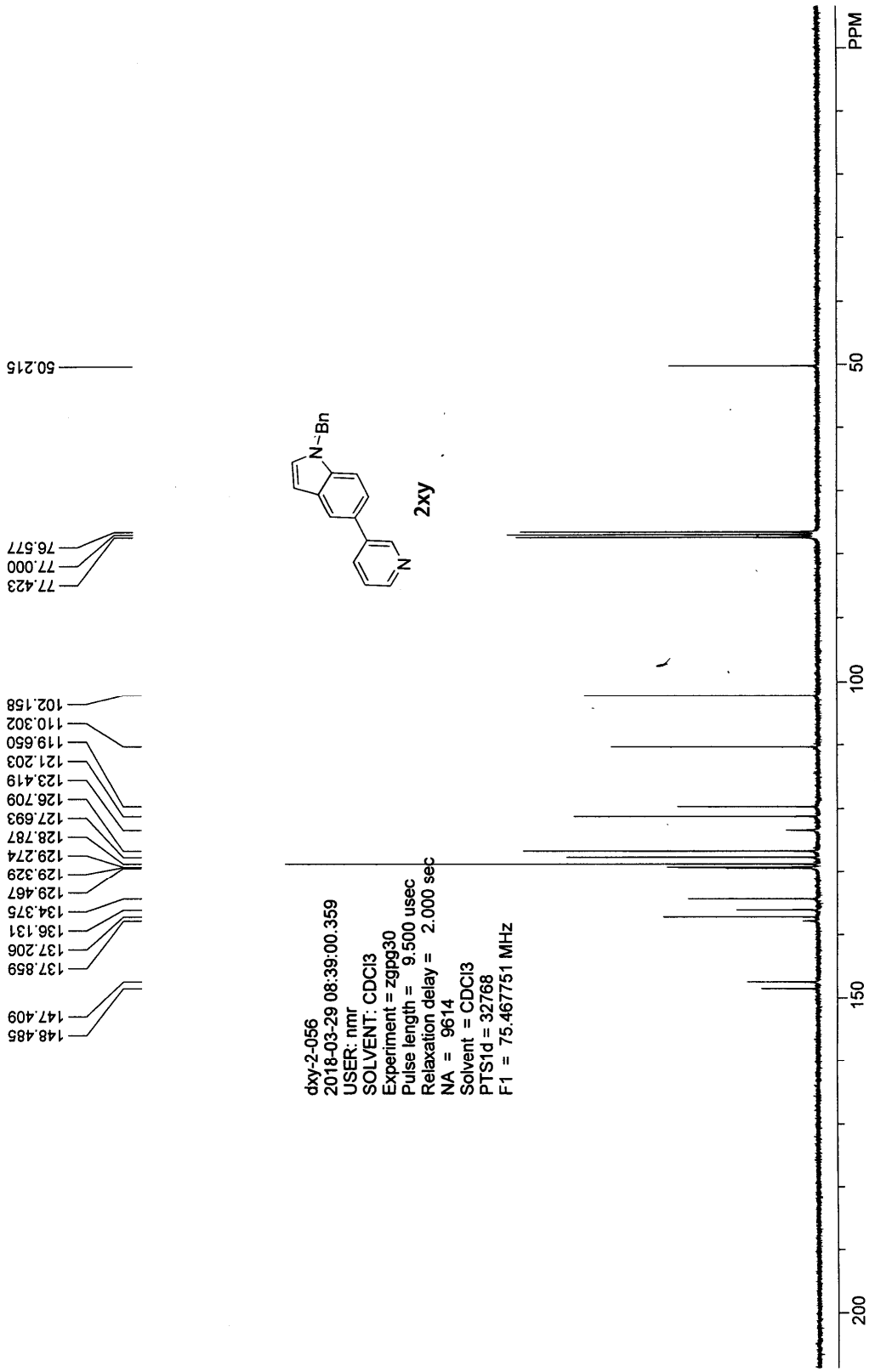


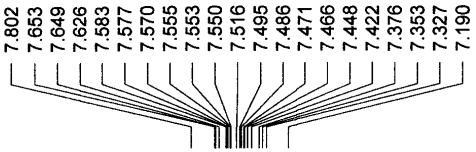
dxy-2-056
2018-03-28 19:07:06.343
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz



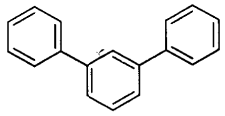
2xy



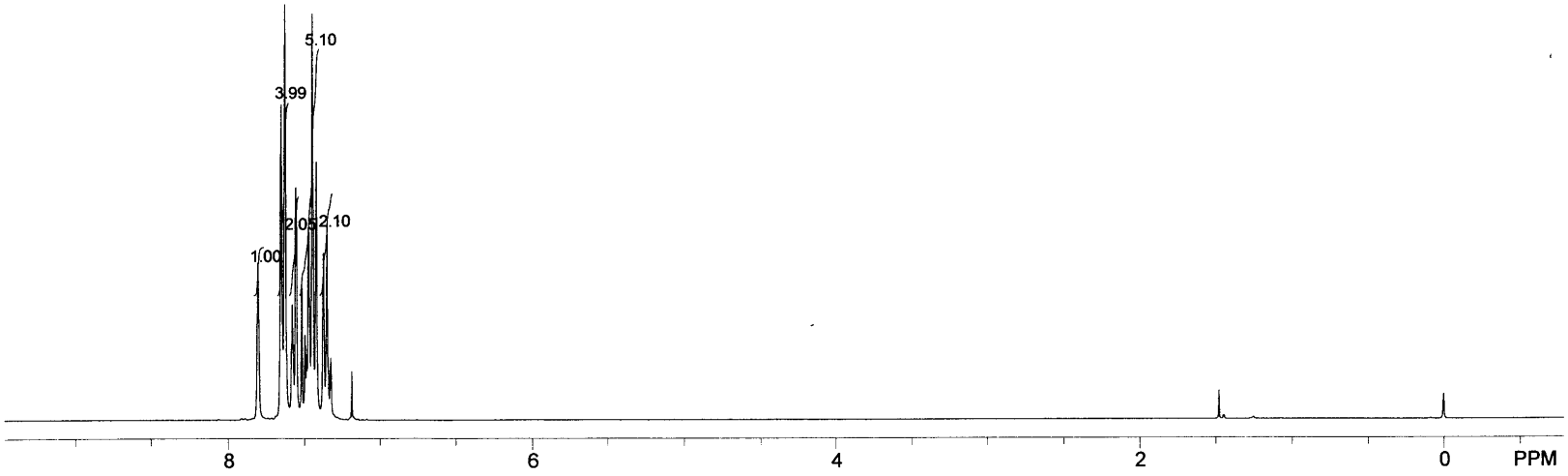

dxy-2-056
2018-03-29 08:39:00.359
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 9614
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
N-Bn
N
2xy
PPM
200
150
100
50
148.485
147.409
137.859
137.206
136.131
134.375
129.467
129.329
129.274
128.787
127.693
126.709
123.419
121.203
119.650
110.302
102.158
77.423
77.000
76.577
50.215

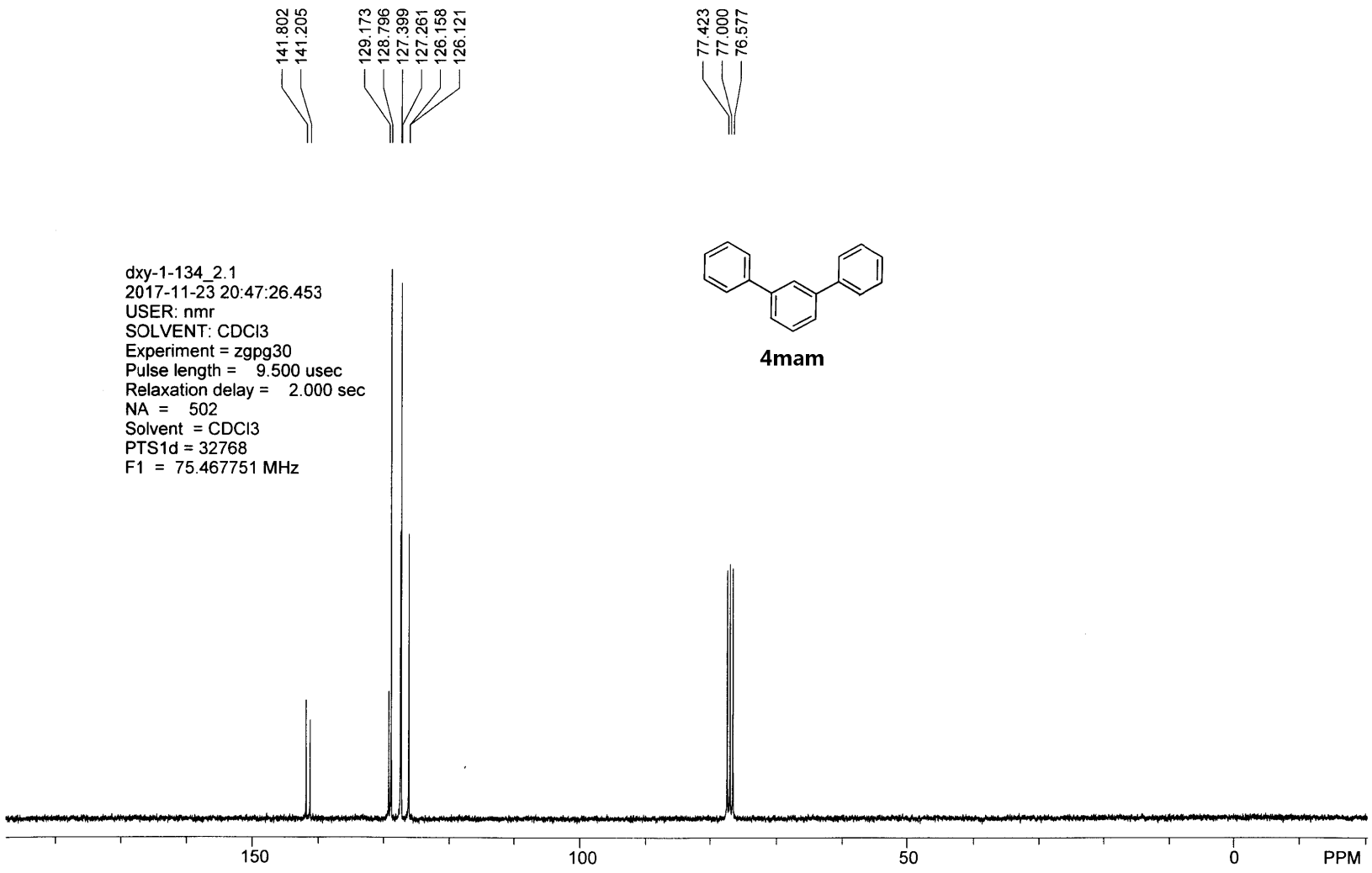


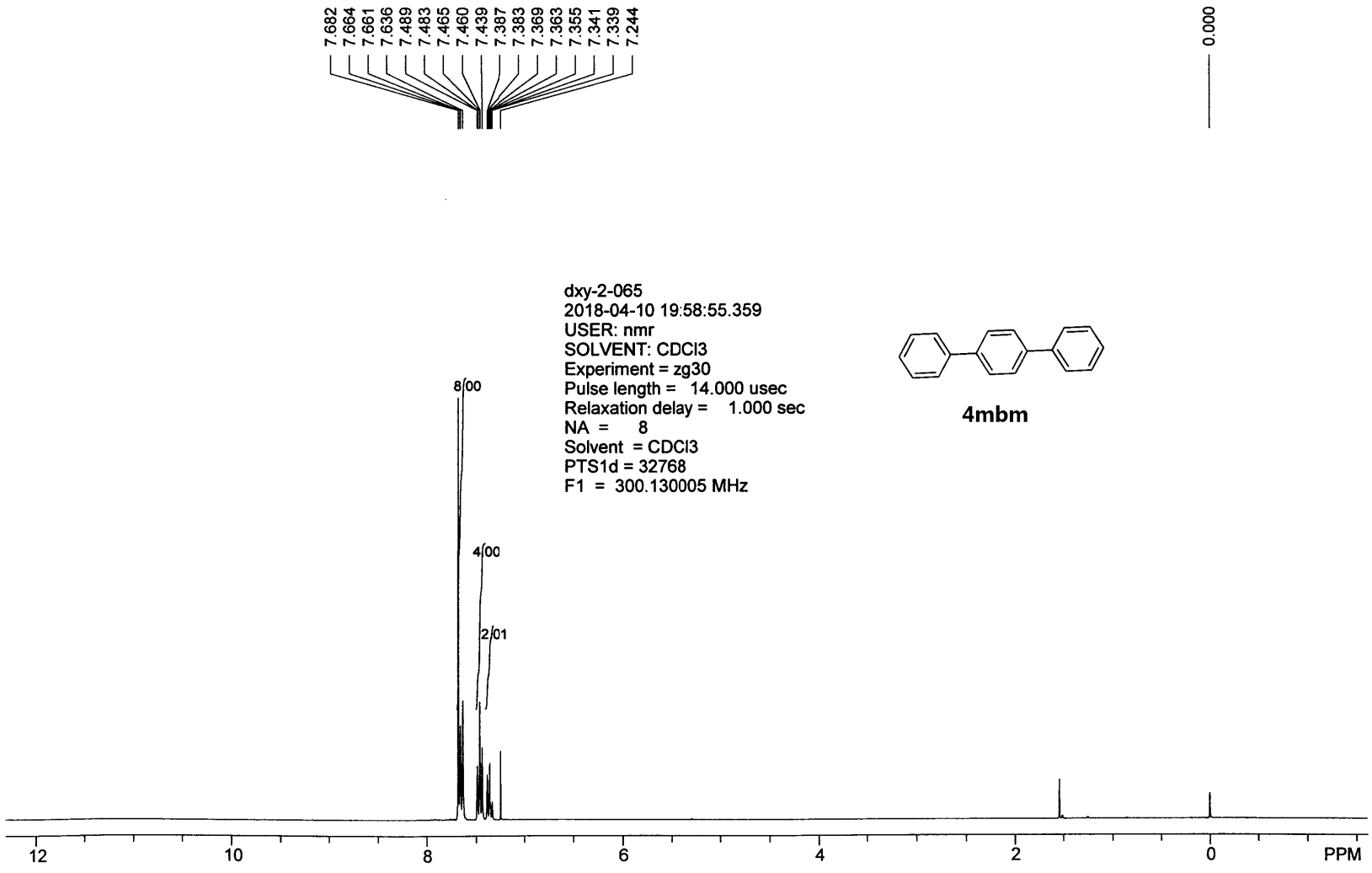
dxy-1-134-1126_1.1
2017-11-26 22:22:41.468
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz

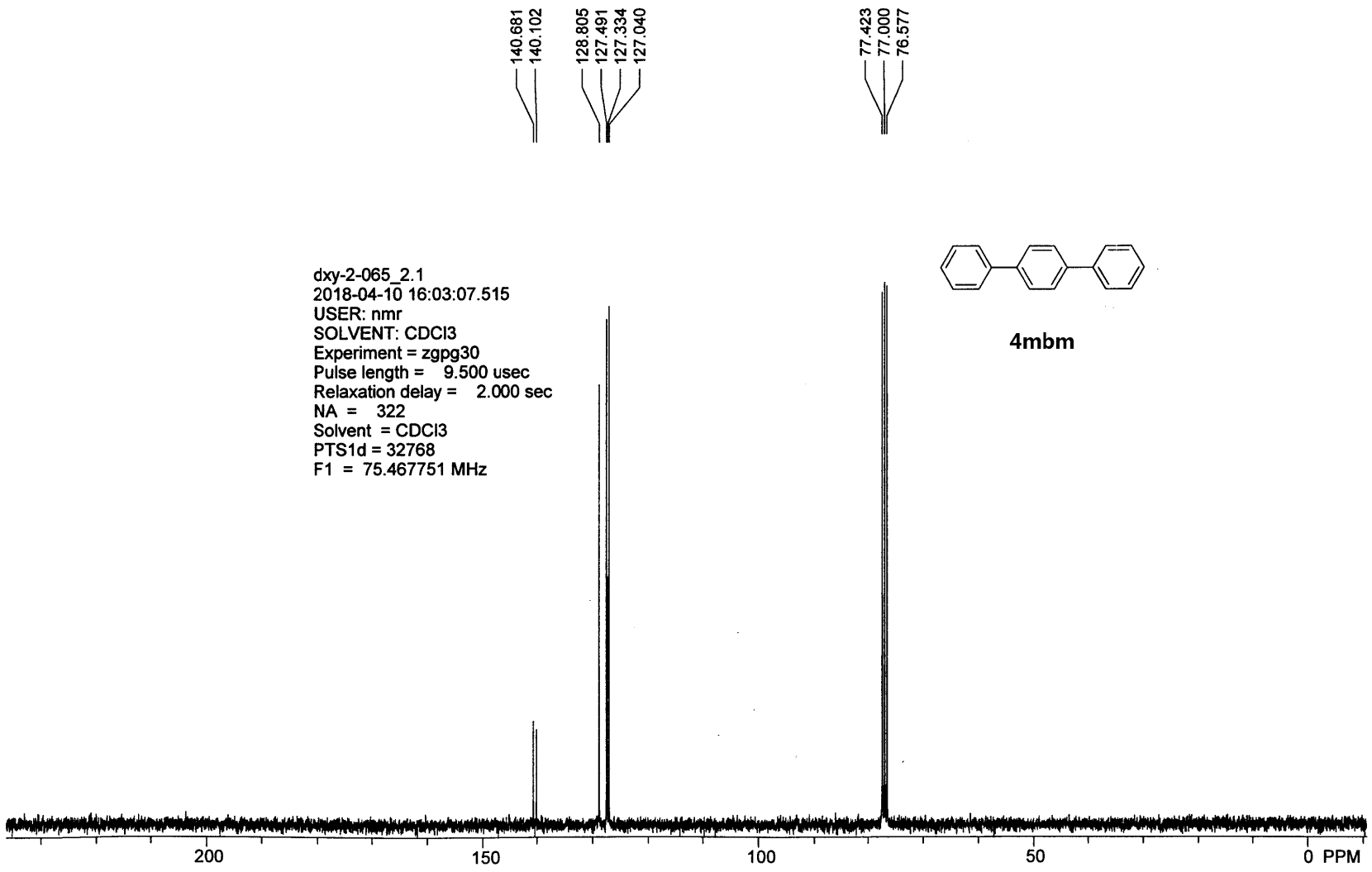


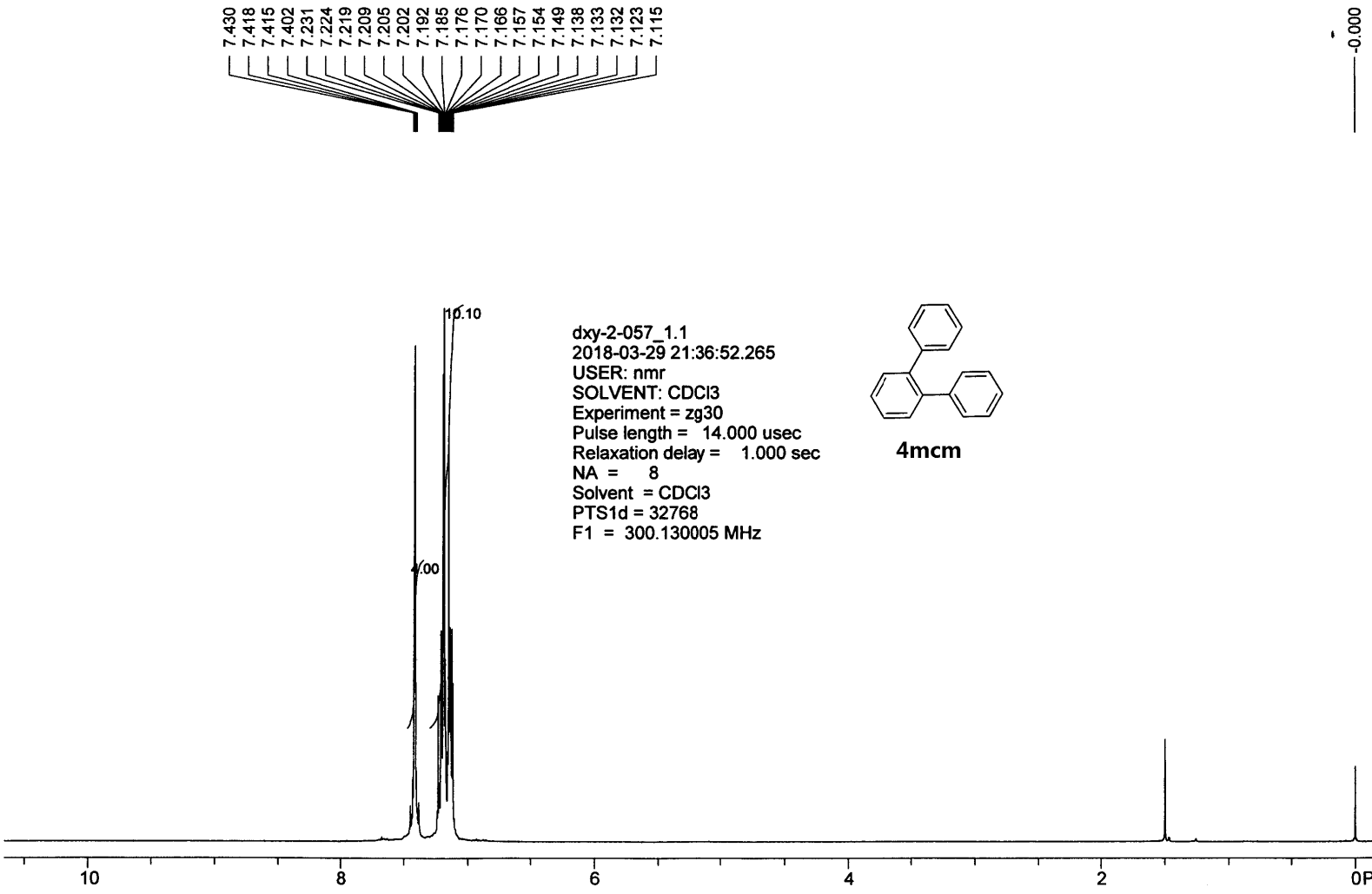
4mam

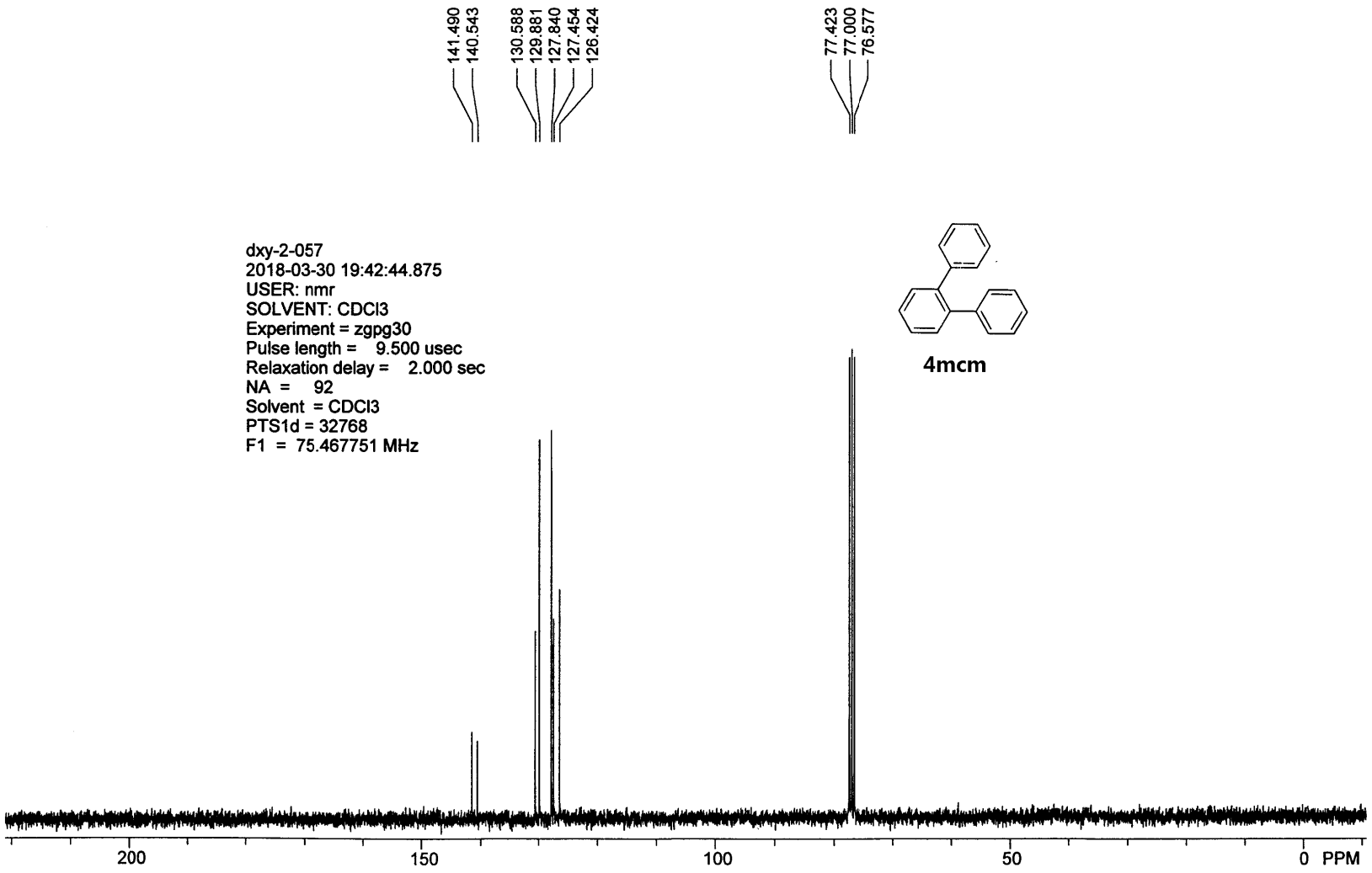


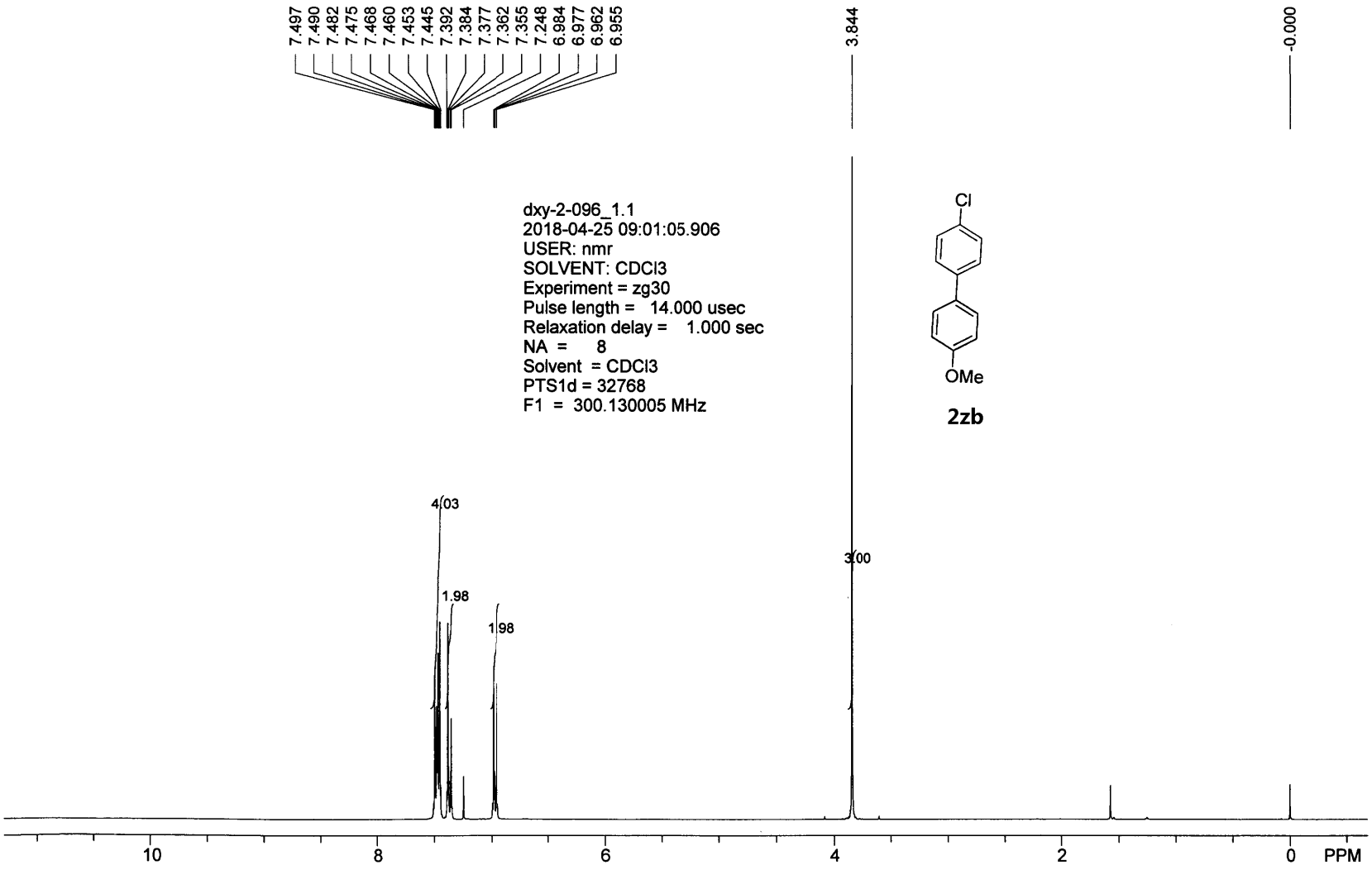

141.802
141.205
129.173
128.796
127.399
127.261
126.158
126.121
77.423
77.000
76.577
dxy-1-134_2.1
2017-11-23 20:47:26.453
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 502
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
4mam
150
100
50
0
PPM

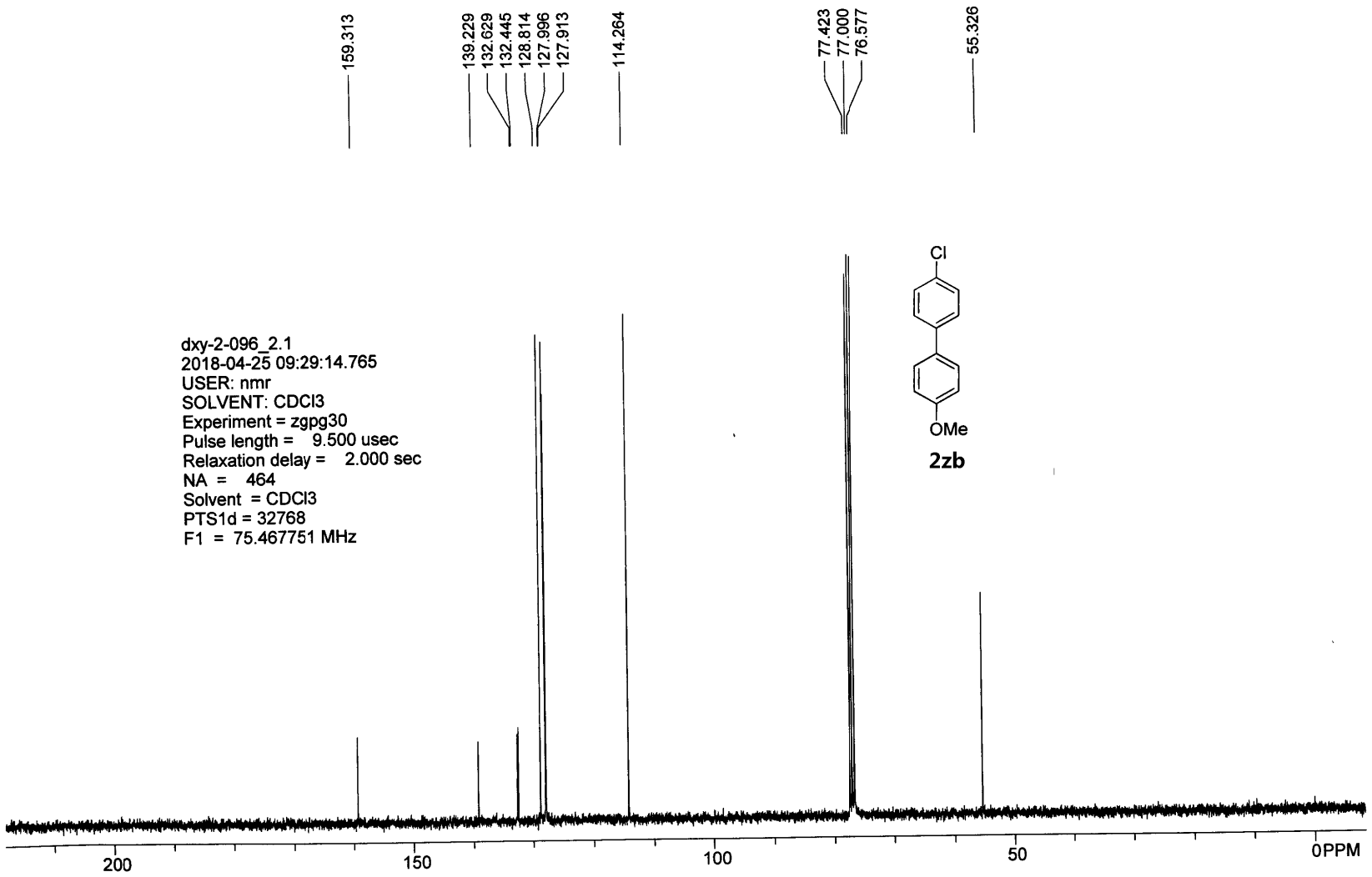

7.682
7.664
7.661
7.636
7.489
7.483
7.465
7.460
7.439
7.387
7.383
7.369
7.363
7.355
7.341
7.339
7.244
0.000
dxy-2-065
2018-04-10 19:58:55.359
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
4mbm
8.00
4.00
2.01
12
10
8
6
4
2
0
PPM

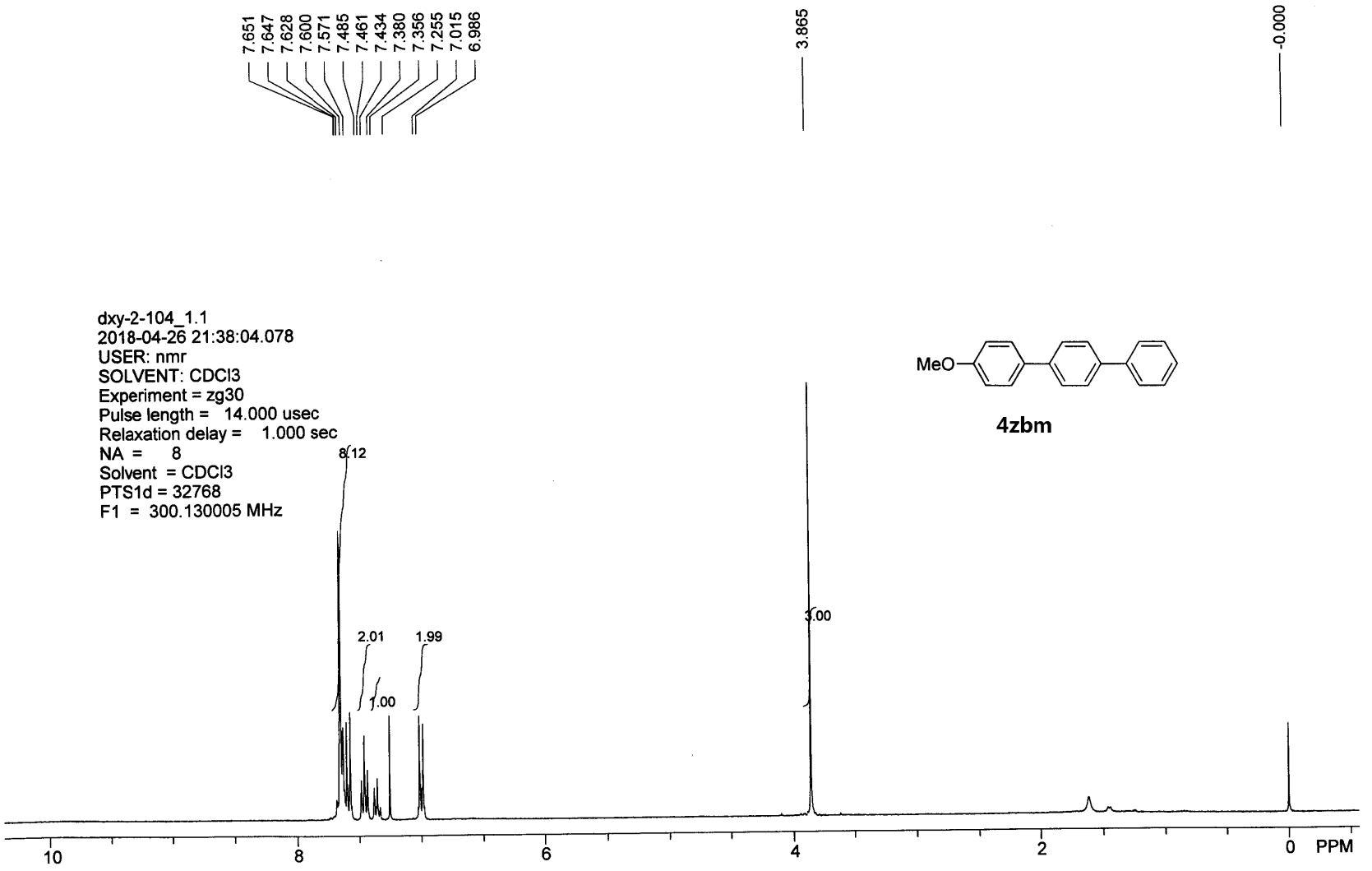

140.681
140.102
128.805
127.491
127.334
127.040
77.423
77.000
76.577
dxy-2-065_2.1
2018-04-10 16:03:07.515
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 322
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
4mbm
200
150
100
50
0 PPM

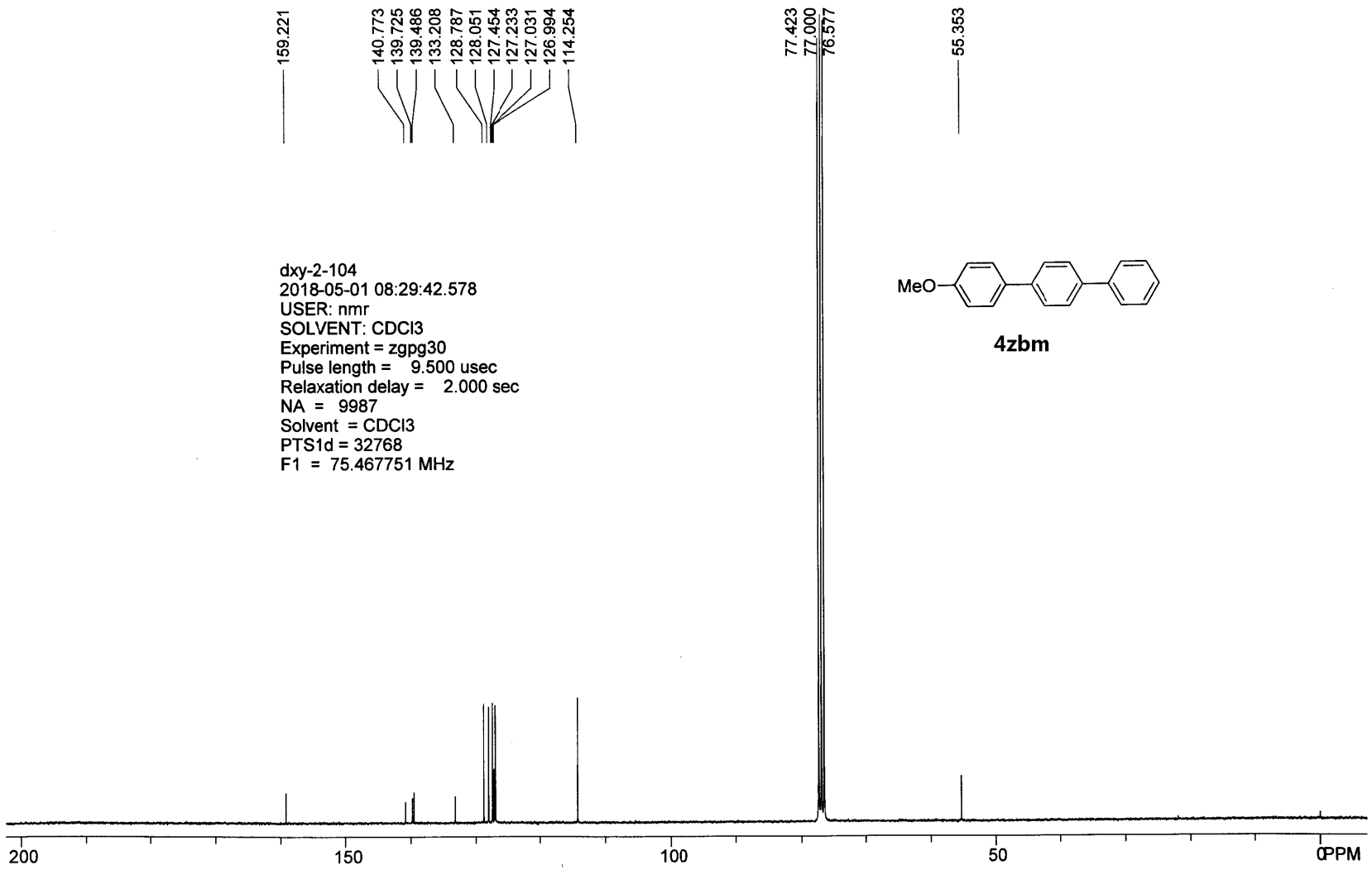

7.430
7.418
7.415
7.402
7.231
7.224
7.219
7.209
7.205
7.202
7.192
7.185
7.176
7.170
7.166
7.157
7.154
7.149
7.138
7.133
7.132
7.123
7.115
-0.000
10.10
4.00
dxy-2-057_1.1
2018-03-29 21:36:52.265
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
4mcm
10
8
6
4
2
0PPM


141.490
140.543
130.588
129.881
127.840
127.454
126.424
77.423
77.000
76.577
dxy-2-057
2018-03-30 19:42:44.875
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 92
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
4mcm
200
150
100
50
0 PPM


7.497
7.490
7.482
7.475
7.468
7.460
7.453
7.445
7.392
7.384
7.377
7.362
7.355
7.248
6.984
6.977
6.962
6.955
3.844
-0.000
dxy-2-096_1.1
2018-04-25 09:01:05.906
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
Cl
OMe
2zb
4.03
1.98
1.98
3.00
10
8
6
4
2
0
PPM


159.313
139.229
132.629
132.445
128.814
127.996
127.913
114.264
77.423
77.000
76.577
55.326
dxy-2-096_2.1
2018-04-25 09:29:14.765
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 464
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
Cl
OMe
2zb
200
150
100
50
0PPM


7.651
7.647
7.628
7.600
7.571
7.485
7.461
7.434
7.380
7.356
7.255
7.015
6.986
3.865
-0.000
dxy-2-104_1.1
2018-04-26 21:38:04.078
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
8.12
2.01
1.99
1.00
3.00
MeO
4zbm
10
8
6
4
2
0
PPM


159.221
140.773
139.725
139.486
133.208
128.787
128.051
127.454
127.233
127.031
126.994
114.254
77.423
77.000
76.577
55.353
dxy-2-104
2018-05-01 08:29:42.578
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 9987
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
MeO
4zbm
200
150
100
50
0PPM