Benzene Construction via Pd-catalyzed Cyclization of 2,7-Alkadiynylic Carbonates in the Presence of Alkynes

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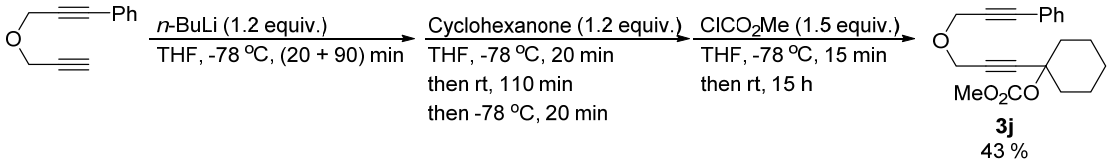
Supporting Information

General information	S2
Synthesis of new starting materials	S3-S6
Synthesis of products **5aa/6aa**~**5la/6la**.	S7-S29
Control experiments	S30-S38
Deuterium labelling experiments	S39-S43
Synthetic applications	S44-S48
References	S49
Copies of the ^{1}H NMR and ^{13}C NMR spectra	S50-S140

General Information

^{1}H NMR and ^{13}C 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). 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*. TFP was purchased from *J&K*. Na_2CO_3 was bought from *Hongguang Chemical Reagent Co., Ltd*. CH_3CN was refluxed over CaH_2 and distilled right before use. Unless otherwise indicated, chemicals and solvents were purchased from commercial suppliers. All the temperatures are referred to the oil baths used. 2,7-Alkadiynylic carbonates were prepared according to our previous literatures.[1,2]

Synthesis of starting material 3j.[1] (wwt-2-35)

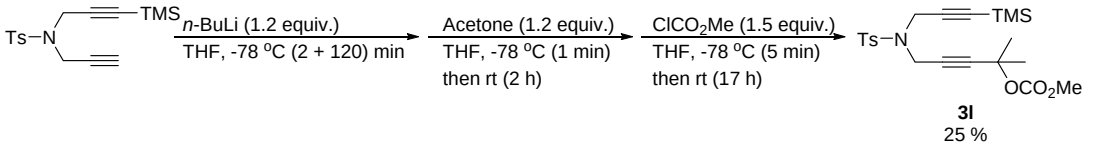


3j
43 %

To a solution of 3-(propargyloxy)phenylpropyne (5086.4 mg, 30 mmol) in THF (120 mL) was added dropwise *n*-BuLi (2.5 M in hexane, 14.4 mL, 36 mmol) at -78 °C within 20 minutes. After lithiation for 90 minutes at -78 °C, cyclohexanone (3.7 mL, d = 0.95 g/mL, 3515.0 mg, 36 mmol) was added dropwise with an addition funnel at -78 °C within 20 minutes. The cooling bath was removed and the reaction mixture was warmed up to room temperature and stirred for 110 minutes. Then the mixture was cooled down to -78 °C again for 20 minutes. Methyl chloroformate (3.5 mL, d = 1.22 g/mL, 4270.0 mg, 45 mmol) was added dropwise at -78 °C within 15 minutes. The cooling bath was removed and the reaction mixture was warmed up to room temperature and stirred for 15 h. After the reaction was complete as monitored by TLC (eluent: petroleum ether/ethyl acetate = 20/1), it was quenched with a saturated aqueous solution of NH_4Cl (50 mL). The resulting mixture was extracted with ethyl acetate (30 mL × 3) and the combined organic phase was dried over anhydrous Na_2SO_4. After filtration and evaporation of the solvent, the crude residue was purified by chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 50/1 (3570 mL) ot 10/1 (1100 mL)) to afford **3j** (4165.2 mg, 43%) as a liquid: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.50-7.39 (m, 2 H, ArH), 7.35-7.27 (m, 3 H, ArH), 4.49 (s, 2 H, OCH_2), 4.40 (s, 2 H, OCH_2), 3.76 (s, 3 H, OCH_3), 2.24-2.10 (m, 2 H, CH_2), 1.95-1.80 (m, 2 H, CH_2), 1.75-1.46 (m, 5 H, CH_2 × 2 and one proton of CH_2), 1.41-1.23 (m, 1 H,

one proton of CH_2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 153.3, 131.8, 128.5, 128.3, 122.5, 86.6, 86.3, 84.3, 81.9, 77.6, 57.1, 56.8, 54.3, 36.8, 24.9, 22.6; IR (neat) ν (cm^{-1}) 2938, 2859, 2236, 2197, 1752, 1490, 1441, 1349, 1274, 1247, 1217, 1181, 1130, 1082, 1016; MS (EI): *m/z* (%) 326 (M^+, 1.79), 115 (100); HRMS calcd. for $C_{20}H_{22}O_4$ [M^+]: 326.1518; Found: 326.1521.

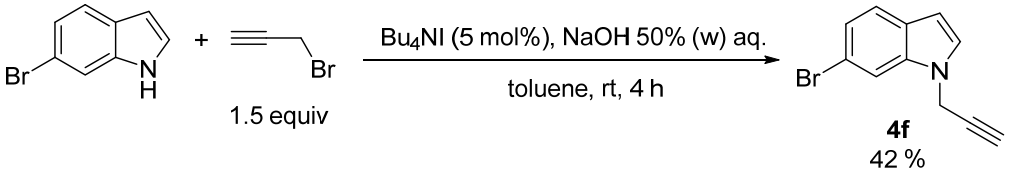
Synthesis of starting material 3l.[1] (zyc-3-26)



To a solution of *N*-(prop-2-ynyl)-*N*-(3-(trimethylsilyl)prop-2-ynyl)-*p*-tolylsulfonamide (1596.2 mg, 5 mmol) in THF (12 mL) was added dropwise *n*-BuLi (2.5 M in hexane, 2.4 mL, 6 mmol) at -78 °C within 2 minutes. After lithiation for 120 minutes at -78 °C, acetone (0.45 mL, d = 0.788 g/mL, 354.6 mg, 6.1 mmol) was added dropwise with an addition funnel at -78 °C within 1 minutes. The cooling bath was removed and the reaction mixture was warmed up to room temperature and stirred for 2 hours. Then methyl chloroformate (0.58 mL, d = 1.22 g/mL, 707.6 mg, 7.5 mmol) was added dropwise at -78 °C within 5 minutes. The cooling bath was removed and the reaction mixture was warmed up to room temperature and stirred for 17 h. After the reaction was complete as monitored by TLC (eluent: petroleum ether/ethyl acetate = 20/1), it was quenched with a saturated aqueous solution of NH_4Cl (10 mL). The resulting mixture was extracted with ethyl acetate (10 mL × 3) and the combined organic phase was dried over anhydrous Na_2SO_4. After filtration and evaporation of

the solvent, the crude residue was purified by chromatography on silica gel (eluent: petroleum ether/ethyl acetate/diethyl ether = 20/1/1) to afford **3l** (560.2 mg, purity = 96%, 25%) as a liquid: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.70 (d, *J* = 8.4 Hz, 2 H, ArH), 7.30 (d, *J* = 8.1 Hz, 2 H, ArH), 4.18 (s, 4 H, OCH_2 × 2), 3.73 (s, 3 H, OCH_3), 2.42 (s, 3 H, CH_3), 1.53 (s, 6 H, CH_3 × 2), 0.05 (s, 9 H, CH_3 × 3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 153.2, 143.6, 135.0, 129.4, 127.7, 97.4, 90.7, 86.0, 77.1, 73.5, 54.1, 37.0, 36.3, 28.3, 21.3, -0.6; IR (neat) ν (cm^{-1}) 2990, 2958, 2900, 2180, 1755, 1598, 1495, 1441, 1354, 1278, 1194, 1165, 1138, 1096, 1045, 1003; MS (EI): *m/z* (%) 435 (M^+, 3.89), 73 (100); HRMS calcd. for $C_{21}H_{29}NO_5SSi$ [M^+]: 435.1536; Found: 435.1539.

Synthesis of starting material 4f.[3] (zyc-1-124)

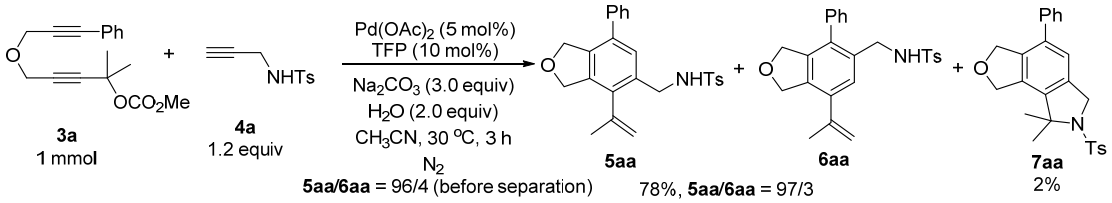


To a dry flask were added 6-bromoindole (1000.8 mg, 5 mmol)/toluene (4 mL), propargyl bromide (0.6 mL, d = 1.52 g/mL, 912.0 mg, 7.7 mmol)/toluene (6 mL), Bu_4NI (92.9 mg, 0.25 mmol), and an aqueous solution of NaOH (2492.4 mg in 2.5 mL of H_2O) sequentially. The reaction was complete after being stirred at room temperature for 4 hours as monitored by TLC. The resulting mixture was transferred to a separation funnel. Water (5 mL) and ethyl acetate (10 mL) were then added. After the separation of the organic phase, the aqueous phase was extracted with ethyl acetate (10 mL × 2). The combined organic phase was washed with brine (10 mL) and dried over anhydrous Na_2SO_4. After filtration and evaporation of the solvent, the

residue was purified by column chromatography on silica gel (eluent: petroleum ether (60-90 ^{o}C)/ethyl acetate = 150/1) to afford impure **4f** (985.6 mg), which was further purified by recrystallization (*n*-hexane/DCM) to afford pure **4f** (493.9 mg, 42%) as a solid: m.p. 51.8-52.7 ^{o}C (*n*-hexane/DCM); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.54 (t, J = 0.9 Hz, 1 H, ArH), 7.46 (dd, J_1 = 8.3 Hz, J_2 = 0.5 Hz, 1 H, ArH), 7.22 (dd, J_1 = 8.4 Hz, J_2 = 1.5 Hz, 1 H, ArH), 7.14 (d, J = 3.3 Hz, 1 H, ArH), 6.48 (dd, J_1 = 3.3 Hz, J_2 = 0.9 Hz, 1 H, ArH), 4.77 (d, J = 2.4 Hz, 2 H, NCH_2), 2.40 (t, J = 2.6 Hz, 1 H, ≡CH); ^{13}C NMR (75 MHz, $CDCl_3$) δ 136.5, 127.9, 127.7, 123.1, 122.3, 115.5, 112.4, 102.3, 77.1, 73.9, 35.8; IR (KBr) ν (cm^{-1}) 3270, 3123, 3102, 2932, 2117, 1869, 1724, 1700, 1695, 1685, 1601, 1560, 1504, 1462, 1425, 1418, 1391, 1343, 1334, 1317, 1308, 1247, 1191, 1100, 1057, 1042; MS (EI): *m/z* (%) 235 ($M(^{81}Br)^{+}$, 32.16), 233 ($M(^{79}Br)^{+}$, 31.09), 154 (100); Anal. Calcd. for $C_{11}H_8BrN$ (%): C 56.44, H 3.44, N 5.98, Found: C 56.28, H 3.47, N 5.98.

Synthesis of products 5aa/6aa~5ia/6ia.

1. Synthesis of *N*-((7-phenyl-4-(propen-2-yl)-1,3-dihydroisobenzofuran-5-yl)methyl)-4-methylbenzenesulfonamide (**5aa**). (wwt-1-193)



Typical Procedure I: To a flame-dried Schlenk tube containing Na_2CO_3 (318.6 mg, 3.0 mmol) were added $Pd(OAc)_2$ (11.1 mg, 0.05 mmol), TFP (23.3 mg, 0.1 mmol), **4a** (250.9 mg, 1.2 mmol)/CH_3CN (2.0 mL), **3a** (286.7 mg, 1.0 mmol)/CH_3CN (6.0 mL), and H_2O (35.6 mg, 2.0 mmol)/CH_3CN (2.0 mL) sequentially under nitrogen atmosphere. The reaction was complete after being stirred at 30 ^oC for 3 hours as monitored by TLC. The resulting mixture was filtrated through a short column of silica gel and eluted with ethyl acetate (30 mL × 3). After evaporation, the crude residue (**5aa**/**6aa** = 96/4 as determined by ^1H NMR analysis of the crude product using mesitylene (46 μL) as the internal standard) was purified by chromatography on silica gel (eluent: petroleum ether (60-90 ^oC)/ethyl acetate = 5/1 (360 mL) to petroleum ether (60-90 ^oC)/ethyl acetate/DCM = 4/1/1 (600 mL)) to afford **7aa** (6.7 mg, 2%) and **5aa**/**6aa** (326.7 mg, 78%, **5aa**/**6aa** = 97/3 as determined by ^1H NMR analysis of the isolated product).

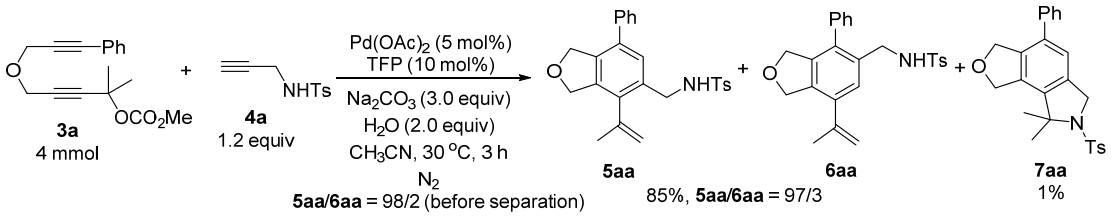
5aa/**6aa** (97/3): solid; ^1H NMR (300 MHz, $CDCl_3$) δ 7.73 (d, *J* = 8.1 Hz, 2 H, ArH), 7.48-7.30 (m, 3 H, ArH), 7.30-7.20 (m, 4 H, ArH), 7.15 (s, 1 H, ArH), 5.26-5.09 (m, 3 H, one proton of $=CH_2$ and OCH_2), 5.08-4.87 (m, 3 H, OCH_2 and NH), 4.85-4.77 (m, 1 H, one proton of $=CH_2$), 4.15 (d, *J* = 6.0 Hz, 2 H, NCH_2), 2.39

(s, 3 H, CH_3), 1.89 (s, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.4, 141.9, 139.3, 138.3, 136.8, 136.7, 136.0, 134.7, 132.9, 129.6, 128.6, 128.5, 127.6, 127.5, 127.1, 116.6, 73.7, 72.9, 44.3, 23.4, 21.4; IR (KBr) ν (cm^{-1}) 3277, 3061, 2916, 2856, 1598, 1477, 1446, 1327, 1290, 1159, 1093, 1058; MS (EI): *m/z* (%) 419 (M^+, 0.09), 264 ($(M\text{-}Ts)^+$, 100); Anal. Calcd. for $C_{25}H_{25}NO_3S$ (%): C 71.57, H 6.01, N 3.34; Found: C 71.53, H 6.07, N 3.17. The following signals are discernible for **6aa**: ^1H NMR (300 MHz, $CDCl_3$) δ 4.77-4.72 (m, 1 H, OCH_2), 3.98 (d, J = 6.3 Hz, 2 H, NCH_2), 2.06 (s, 3 H, CH_3).

This mixture may further be purified by recrystallization to afford the pure product **5aa**: m.p. 152.4-154.5 °C (*n*-hexane/DCM).

7aa: liquid; ^1H NMR (300 MHz, $CDCl_3$) δ 7.83 (d, J = 8.4 Hz, 2 H, ArH), 7.49-7.27 (m, 7 H, ArH), 7.10 (s, 1 H, ArH), 5.19 (s, 2 H, OCH_2), 5.11 (s, 2 H, OCH_2), 4.68 (s, 2 H, NCH_2), 2.41 (s, 3 H, CH_3), 1.78 (s, 6 H, CH_3 × 2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.2, 139.6, 139.2, 138.1, 137.9, 135.8, 133.8, 133.0, 129.5, 128.7, 127.8, 127.7, 127.4, 121.8, 72.8, 71.2, 70.6, 53.1, 27.1, 21.5; IR (neat) ν (cm^{-1}) 2971, 2923, 2854, 1763, 1598, 1495, 1467, 1409, 1339, 1273, 1162, 1149, 1135, 1093, 1058, 1005; MS (EI): *m/z* (%) 419 (M^+, 0.1), 404 ($(M\text{-}CH_3)^+$, 100); HRMS calcd. for $C_{24}H_{22}NO_3S$ $[(M\text{-}CH_3)^+]$: 404.1320; Found: 404.1321.

Gram-scale synthesis of 5aa. (zyc-1-166)



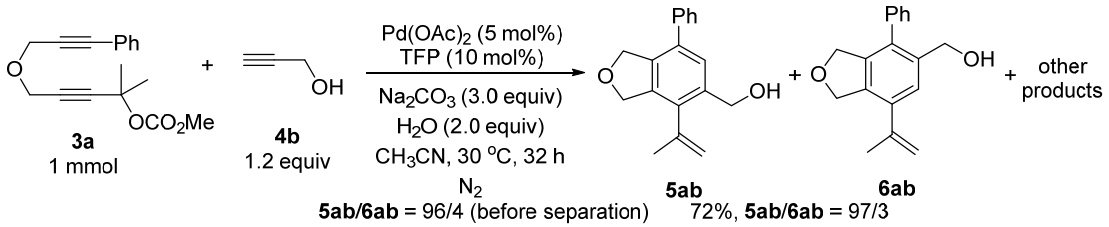
Following **Typical Procedure I**, the reaction of **3a** (1144.6 mg, 4.0 mmol), **4a** (1004.7 mg, 4.8 mmol), $Pd(OAc)_2$ (44.5 mg, 0.2 mmol), TFP (93.4 mg, 0.4 mmol), Na_2CO_3 (1272.2 mg, 12.0 mmol), and H_2O (144.4 mg, 8.0 mmol) in CH_3CN (40 mL) afforded **7aa** (20.5 mg, 1%) and **5aa**/**6aa** (1417.3 mg, 85%, **5aa**/**6aa** = 97/3 as determined by ^1H NMR analysis of the isolated product) (eluent: petroleum ether (60-90 ^oC)/ethyl acetate/DCM = 10/1/1 (2040 mL) to 5/1/1 (210 mL)) (**5aa**/**6aa** = 98/2 as determined by ^1H NMR analysis of the crude product using mesitylene (184 μL) as the internal standard).

5aa/**6aa** (97/3): ^1H NMR (300 MHz, $CDCl_3$) δ 7.72 (d, *J* = 8.1 Hz, 2 H, ArH), 7.44-7.18 (m, 7 H, ArH), 7.15 (s, 1 H, ArH), 5.25-5.16 (m, 1 H, one proton of $=CH_2$), 5.26-5.06 (m, 3 H, one proton of NH and OCH_2), 5.05-4.95 (m, 2 H, OCH_2), 4.84-4.76 (m, 1 H, one proton of $=CH_2$), 4.15 (d, *J* = 6.0 Hz, 2 H, NCH_2), 2.37 (s, 3 H, CH_3), 1.89 (s, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.4, 141.9, 139.3, 138.2, 136.9, 136.6, 136.0, 134.7, 132.9, 129.6, 128.6, 128.4, 127.6, 127.5, 127.1, 116.5, 73.6, 72.9, 44.2, 23.4, 21.4. The following signals are discernible for **6aa**: ^1H NMR (300 MHz, $CDCl_3$) δ 4.75-4.72 (m, 1 H, one proton of $=CH_2$), 3.97 (d, *J* = 6.0 Hz, 2 H, NCH_2), 2.04 (s, 3 H, CH_3).

7aa: liquid; ^1H NMR (300 MHz, $CDCl_3$) δ 7.83 (d, *J* = 8.1 Hz, 2 H, ArH), 7.47-7.25 (m, 7 H, ArH), 7.10 (s, 1 H, ArH), 5.19 (s, 2 H, OCH_2), 5.11 (s, 2 H, OCH_2), 4.68 (s, 2 H, NCH_2), 2.41 (s, 3 H, CH_3), 1.78 (s, 6 H, $CH_3 \times 2$); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.2, 139.6, 139.2, 138.0, 137.8, 135.8, 133.8, 132.9, 129.5, 128.7, 127.8, 127.7, 127.3, 121.8, 72.8, 71.1, 70.6, 53.1, 27.1, 21.5.

The following compounds **5ab**/**6ab** ~ **5ia**/**6ia** were prepared according to **Typical Procedure I**.

2. Synthesis of (7-phenyl-4-(propen-2-yl)-1,3-dihydroisobenzofuran-5-yl)methanol (**5ab**). (wwt-1-189, wwt-2-90)



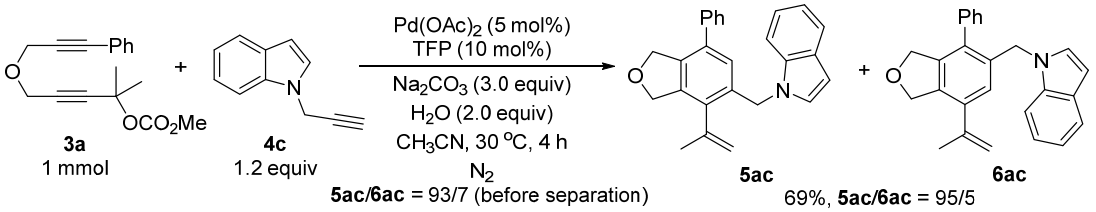
5ab/6ab = 96/4 (before separation) 72%, **5ab/6ab** = 97/3

The reaction of Na_2CO_3 (316.4 mg, 3.0 mmol), $Pd(OAc)_2$ (11.3 mg, 0.05 mmol), TFP (23.1 mg, 0.1 mmol), **4b** (67.3 mg, 1.2 mmol), **3a** (285.4 mg, 1.0 mmol), and H_2O (36.2 mg, 2.0 mmol) in CH_3CN (10 mL) afforded **5ab**/**6ab** (190.1 mg, 72%, **5ab**/**6ab** = 97/3 as determined by ^1H NMR analysis of the isolated product) (eluent: petroleum ether (60-90 ^oC)/ethyl acetate = 10/1 (330 mL) to 5/1 (480 mL)) (**5ab**/**6ab** = 96/4 as determined by ^1H NMR analysis of the crude product using mesitylene (46 μL) as the internal standard).

5ab/**6ab** (97/3): liquid; ^1H NMR (300 MHz, $CDCl_3$) δ 7.48-7.28 (m, 6 H, ArH), 5.29-5.23 (m, 1 H, one proton of $=CH_2$), 5.20-5.14 (m, 2 H, OCH_2), 5.09-5.02 (m, 2 H, OCH_2), 4.91-4.85 (m, 1 H, one proton of $=CH_2$), 4.68 (s, 2 H, OCH_2), 2.34 (br s, 1 H, OH), 2.00 (t, J = 1.2 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 142.4, 139.7, 137.9, 137.6, 136.2, 135.5, 134.6, 128.6, 127.7, 127.6, 127.4, 116.0, 73.7, 72.9, 62.4, 23.6; IR (neat) ν (cm^{-1}) 3416, 3076, 3029, 2938, 2911, 2855, 1640, 1601, 1474, 1446, 1370, 1329, 1287, 1195, 1136, 1057; GC-MS (GC condition: injector: 280 ^oC; column:

DB-5, temperature: 60 °C (2 min), 20 °C/min to 280 °C, 280 °C (30 min)) (EI) *m/z* (%) for **5ab**: T_R 5.3 min: 266 (M^+, 100); for **6ab**: T_R 5.2 min: 266 (M^+, 100); HRMS calcd. for $C_{18}H_{18}O_2$ [M^+]: 266.1307; Found: 266.1306. The following signals are discernible for **6ab**: ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.23-5.20 (m, 1 H, one proton of $=CH_2$), 5.00-4.97 (m, 2 H, OCH_2), 4.84-4.80 (m, 2 H, OCH_2), 4.48 (s, 2 H, CH_2), 2.14-2.10 (m, 3 H, CH_3).

3. Synthesis of 1-((7-phenyl-4-(propen-2-yl)-1,3-dihydroisobenzofuran-5-yl)methyl)-1*H*-indole (**5ac**). (wwt-2-123, zyc-1-68)



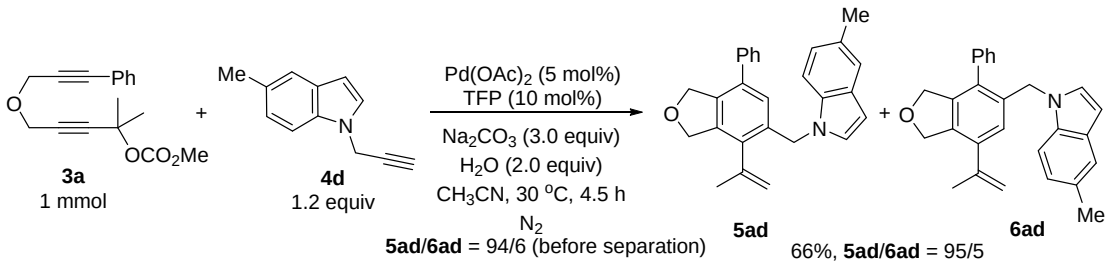
The reaction of Na_2CO_3 (317.6 mg, 3 mmol), $Pd(OAc)_2$ (11.3 mg, 0.05 mmol), TFP (23.1 mg, 0.1 mmol), **4c** (185.4 mg, 1.2 mmol), **3a** (285.4 mg, 1.0 mmol), and H_2O (36.2 mg, 2 mmol) in CH_3CN (10 mL) afforded **5ac**/**6ac** (252.9 mg, 69%, **5ac**/**6ac** = 95/5 as determined by ^{1}H NMR analysis of the isolated product) (eluent: petroleum ether (60-90 °C)/ethyl acetate = 40/1) (**5ac**/**6ac** = 93/7 as determined by ^{1}H NMR analysis of the crude product using mesitylene (46 μL) as the internal standard).

5ac/**6ac** (95/5): solid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.66-7.57 (m, 1 H, ArH), 7.47-7.00 (m, 9 H, ArH), 6.93 (s, 1 H, ArH), 6.52-6.46 (m, 1 H, ArH), 5.34-5.26 (m, 3 H, one proton of $=CH_2$ and NCH_2), 5.21-5.15 (m, 2 H, OCH_2), 5.11-5.05 (m, 2 H, OCH_2), 4.99-4.93 (m, 1 H, one proton of $=CH_2$), 1.88 (s, 3 H, CH_3); ^{13}C NMR (75

MHz, $CDCl_3$) δ 142.1, 139.4, 138.4, 136.6, 136.3, 135.8, 134.7, 133.9, 128.6, 128.0, 127.6, 127.4, 127.3, 121.5, 120.9, 119.4, 116.3, 109.5, 101.7, 73.7, 72.9, 47.2, 23.1; GC-MS (GC condition: injector: 280 ^oC; column: DB-5, temperature: 60 ^oC (2 min), 20 ^oC/min to 280 ^oC, 280 ^oC (30 min)) (EI) *m/z* (%) for **5ac**: T_R 6.8 min: 365 (M^+, 71.0), 350 (100); for **6ac**: T_R 7.5 min: 365 (M^+, 31.1), 350 (100); HRMS calcd. for $C_{26}H_{23}NO$ [M^+]: 365.1780, Found: 365.1784. The following signals are discernible for **6ac**: ^1H NMR (300 MHz, $CDCl_3$) δ 6.46-6.42 (m, 1 H, ArH), 5.46-5.38 (m, 3 H, one proton of $=CH_2$ and NCH_2), 5.13 (s, 2 H, OCH_2).

This mixture may further be purified by recrystallization to afford the pure product **5ac**: m.p. 121.5-123.3 ^oC (*n*-hexane/DCM).

4. Synthesis of 5-methyl-1-((7-phenyl-4-(propen-2-yl)-1,3-dihydroisobenzofuran-5-yl)methyl)-1*H*-indole (**5ad**). (zyc-1-102)

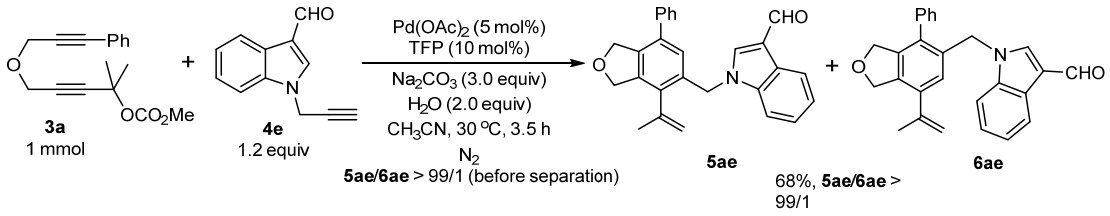


The reaction of Na_2CO_3 (317.9 mg, 3.0 mmol), $Pd(OAc)_2$ (11.8 mg, 0.05 mmol), TFP (24.1 mg, 0.1 mmol), **4d** (203.5 mg, 1.2 mmol), **3a** (285.7 mg, 1.0 mmol), and H_2O (37.5 mg, 2.1 mmol) in CH_3CN (10 mL) afforded **5ad**/**6ad** (249.9 mg, 66%, **5ad**/**6ad** = 95/5 as determined by ^1H NMR analysis of the isolated product) (eluent: petroleum ether (60-90 ^oC)/ethyl acetate = 30/1) (**5ad**/**6ad** = 94/6 as determined by ^1H NMR analysis of the crude product using mesitylene (46 μL) as the internal standard).

5ad/**6ad** (95/5): solid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.46-7.10 (m, 7 H, ArH), 7.07-6.88 (m, 3 H, ArH), 6.42 (d, *J* = 3.3 Hz, 1 H, ArH), 5.37-5.24 (m, 3 H, one proton of $=CH_2$ and NCH_2), 5.22-5.16 (m, 2 H, OCH_2), 5.12-5.05 (m, 2 H, OCH_2), 5.01-4.93 (m, 1 H, one proton of $=CH_2$), 2.42 (s, 3 H, CH_3), 1.89 (s, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 142.2, 139.5, 138.4, 136.6, 135.8, 134.8, 134.1, 128.9, 128.6, 128.1, 127.7, 127.45, 127.37, 123.2, 120.6, 116.3, 109.2, 101.1, 73.8, 73.0, 47.3, 23.2, 21.3; IR (KBr) ν (cm^{-1}) 3073, 3028, 2965, 2909, 2832, 1508, 1485, 1474, 1437, 1390, 1371, 1345, 1333, 1301, 1256, 1227, 1180, 1132, 1059, 1029; MS (EI): *m/z* (%) 379 (M^+, 77.0), 364 ($(M-CH_3)^+$, 100.0); HRMS calcd. for $C_{27}H_{25}NO$ [M^+]: 379.1936, Found: 379.1937. The following signals are discernible for **6ad**: ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.36 (s, 1 H, ArH), 5.44-5.41 (m, 3 H, NCH_2 and one proton of $=CH_2$), 2.45 (s, 3 H, CH_3).

This mixture may further be purified by recrystallization to afford the pure product **5ad**: m.p. 119.6-122.8 ^{o}C (DCM).

5. Synthesis of 1-((7-phenyl-4-(propen-2-yl)-1,3-dihydroisobenzofuran-5-yl)methyl)-1*H*-indole-3-carbaldehyde (**5ae**). (zyc-1-79)

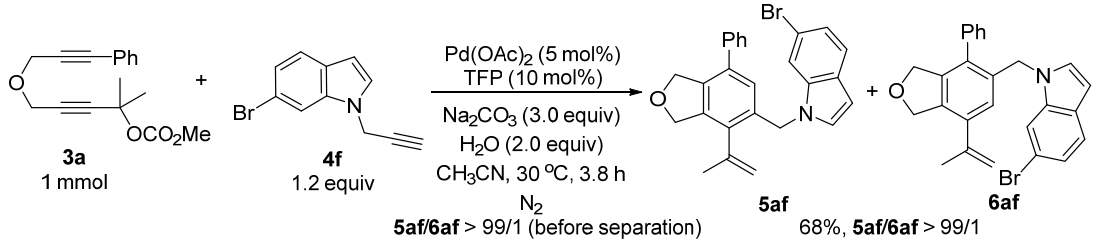


The reaction of Na_2CO_3 (318.8 mg, 3.0 mmol), $Pd(OAc)_2$ (11.5 mg, 0.05 mmol), TFP (24.2 mg, 0.1 mmol), **4e** (220.2 mg, 1.2 mmol), **3a** (285.9 mg, 1.0 mmol), and

H_2O (37.0 mg, 2.0 mmol) in CH_3CN (10 mL) afforded **5ae** (268.9 mg, 68%, **5ae**/**6ae** > 99/1 as determined by ^1H NMR analysis of the isolated product) (eluent: petroleum ether (60-90 ^oC)/ethyl acetate = 10/1 (880 mL) to 5/1 (1000 mL)) (**5ae**/**6ae** > 99/1 as determined by ^1H NMR analysis of the crude product using mesitylene (46 μL) as the internal standard).

5ae: solid; m.p. 158.8-160.4 ^oC (*n*-hexane/DCM); ^1H NMR (300 MHz, $CDCl_3$) δ 9.97 (s, 1 H, CHO), 8.35-8.26 (m, 1 H, ArH), 7.64 (s, 1 H, ArH), 7.45-7.22 (m, 8 H, ArH), 7.07 (s, 1 H, ArH), 5.38 (s, 2 H, NCH_2), 5.34-5.28 (m, 1 H, one proton of $=CH_2$), 5.25-5.19 (m, 2 H, OCH_2), 5.13-5.06 (m, 2 H, OCH_2), 4.98-4.91 (m, 1 H, one proton of $=CH_2$), 1.87 (s, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 184.5, 141.7, 139.00, 138.96, 138.3, 137.7, 137.4, 136.2, 135.1, 131.6, 128.7, 127.9, 127.7, 127.6, 125.3, 124.0, 123.0, 122.0, 118.4, 116.7, 110.2, 73.7, 72.9, 48.0, 23.1; IR (KBr) ν (cm^{-1}) 3111, 3080, 3059, 3019, 2962, 2938, 2911, 2836, 1659, 1615, 1577, 1531, 1466, 1435, 1406, 1388, 1352, 1259, 1157, 1136, 1057, 1046, 1013; MS (EI): *m/z* (%) 393 (M^+, 100.0); HRMS calcd. for $C_{27}H_{23}NO_2$ [M^+]: 393.1729, Found: 393.1728.

6. Synthesis of 6-bromo-1-((7-phenyl-4-(propen-2-yl)-1,3-dihydroisobenzofuran-5-yl) methyl)-1*H*-indole (**5af**). (zyc-1-130)



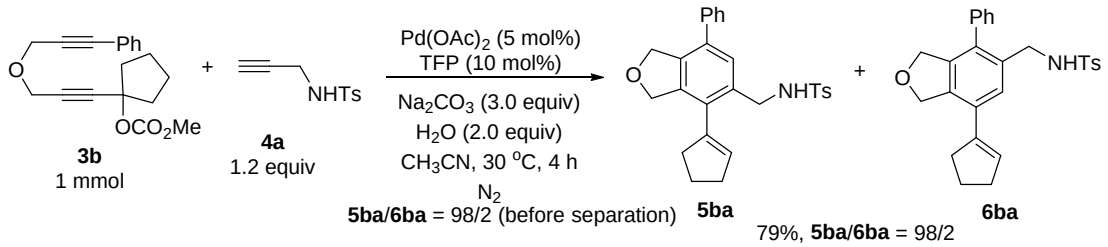
The reaction of Na_2CO_3 (317.5 mg, 3.0 mmol), $Pd(OAc)_2$ (11.6 mg, 0.05 mmol),

TFP (23.9 mg, 0.1 mmol), **4f** (281.7 mg, 1.2 mmol), **3a** (286.0 mg, 1.0 mmol), and H_2O (37.2 mg, 2.0 mmol) in CH_3CN (10 mL) afforded **5af** (304.3 mg, 68%, **5af**/**6af** > 99/1 as determined by ^1H NMR analysis of the isolated product) (The crude product was purified by column chromatography on silica gel (petroleum ether (60-90 ^oC)/ethyl acetate = 40/1) to afford impure **5af** (365.9 mg), which was further purified with chromatography (petroleum ether (60-90 ^oC)/DCM = 1/1)) (**5af**/**6af** > 99/1 as determined by ^1H NMR analysis of the crude product using mesitylene (46 μL) as the internal standard).

5af: solid; m.p. 120.6-122.7 ^oC (DCM); ^1H NMR (300 MHz, $CDCl_3$) δ 7.55-7.43 (m, 2 H, ArH), 7.42-7.22 (m, 5 H, ArH), 7.19 (dd, J_1 = 8.4 Hz, J_2 = 1.8 Hz, 1 H, ArH), 7.03 (d, J = 3.3 Hz, 1 H, ArH), 6.91 (s, 1 H, ArH), 6.48 (d, J = 3.3 Hz, 1 H, ArH), 5.36-5.31 (m, 1 H, one proton of $=CH_2$), 5.28 (s, 2 H, NCH_2), 5.24-5.17 (m, 2 H, OCH_2), 5.14-5.06 (m, 2 H, OCH_2), 5.02-4.95 (m, 1 H, one proton of $=CH_2$), 1.88 (s, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 142.0, 139.4, 138.6, 137.1, 136.9, 135.8, 134.9, 133.4, 128.7, 128.6, 127.7, 127.5, 127.4, 127.3, 122.8, 122.1, 116.5, 115.2, 112.6, 102.0, 73.7, 73.0, 47.3, 23.2; IR (KBr) ν (cm^{-1}) 3072, 3025, 2964, 2938, 2898, 2854, 1637, 1602, 1561, 1504, 1461, 1439, 1391, 1366, 1332, 1319, 1241, 1205, 1187, 1130, 1094, 1057, 1043, 1031; MS (EI): *m/z* (%) 443 ($M(^{81}Br)^+$, 79.0), 445 ($M(^{79}Br)^+$, 80.5), 428 (100); Anal. Calcd. for $C_{26}H_{22}BrNO$ (%): C 70.28, H 4.99, N 3.15; Found: C 69.98, H 5.12, N 2.95.

7. Synthesis of N-((4-(cyclopentenyl)-7-phenyl-1,3-dihydroisobenzofuran-5-yl)meth-

yl)-4-methylbenzenesulfonamide (**5ba**). (wwt-2-130)



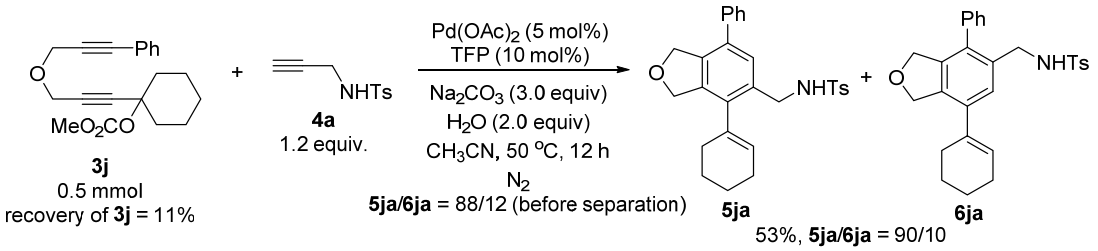
The reaction of Na_2CO_3 (318.2 mg, 3.0 mmol), $Pd(OAc)_2$ (11.3 mg, 0.05 mmol), TFP (23.4 mg, 0.1 mmol), **4a** (251.1 mg, 1.2 mmol), **3b** (311.7 mg, 1.0 mmol), and H_2O (36.0 mg, 2.0 mmol) in CH_3CN (10 mL) afforded **5ba**/**6ba** (351.0 mg, 79%, **5ba**/**6ba** = 98/2 as determined by ^1H NMR analysis of the isolated product) (The crude product was purified by column chromatography on silica gel to afford a part of pure **5ba**/**6ba** (petroleum ether (60-90 °C)/ethyl acetate = 8/1 (450 mL) to 6/1 (490 mL)) and impure **5ba**/**6ba** (determined by TLC). The impure part was further purified by column chromatography on silica gel (petroleum ether (60-90 °C)/ethyl acetate = 8/1) to afford another part of pure **5ba**/**6ba**) (**5ba**/**6ba** = 98/2 as determined by ^1H NMR analysis of the crude product using mesitylene (46 μL) as the internal standard).

5ba/**6ba** (98/2): solid; ^1H NMR (300 MHz, $CDCl_3$) δ 7.71 (d, *J* = 8.4 Hz, 2 H, ArH), 7.42-7.27 (m, 3 H, ArH), 7.27-7.17 (m, 4 H, ArH), 7.15 (s, 1 H, ArH), 5.60-5.50 (m, 1 H, =CH), 5.29 (br s, 1 H, NH), 5.15-5.02 (m, 2 H, OCH_2), 5.02-4.85 (m, 2 H, OCH_2), 4.10 (d, *J* = 6.0 Hz, 2 H, NCH_2), 2.50-2.28 (m, 7 H, CH_2 × 2 and CH_3), 2.00-1.70 (m, 2 H, CH_2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.1, 139.5, 139.1, 138.7, 136.6, 136.0, 134.2, 133.6, 131.5, 131.0, 129.3, 128.4, 127.4, 127.2, 126.8, 73.3, 73.0, 44.4, 36.2, 32.9, 23.6, 21.2; IR (neat) ν (cm^{-1}) 3274, 3059, 3027, 2949, 2920, 2847, 1599, 1495, 1474, 1446, 1328, 1266, 1161, 1094, 1055; MS (EI): *m/z* (%)

290 ((M-Ts)$^+$, 100); Anal. Calcd. for $C_{27}H_{27}NO_3S$ (%): C 72.78, H 6.11, N 3.14; Found: C 72.58, H 6.31, N 3.06. The following signals are discernible for **6ba**: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.52 (d, *J* = 8.4 Hz, 2 H, ArH), 4.75-4.70 (m, 2 H, OCH_2), 3.95 (d, *J* = 6.3 Hz, 2 H, CH_2).

This mixture may further be purified by recystallization to afford the pure product **5ba**: m.p. 123.4-125.3 ^{o}C (*n*-hexane/DCM).

8. Synthesis of *N*-((4-(cyclohexenyl)-7-phenyl-1,3-dihydroisobenzofuran-5-yl)methyl)-4-methylbenzenesulfonamide (**5ja**). (zyc-3-38)

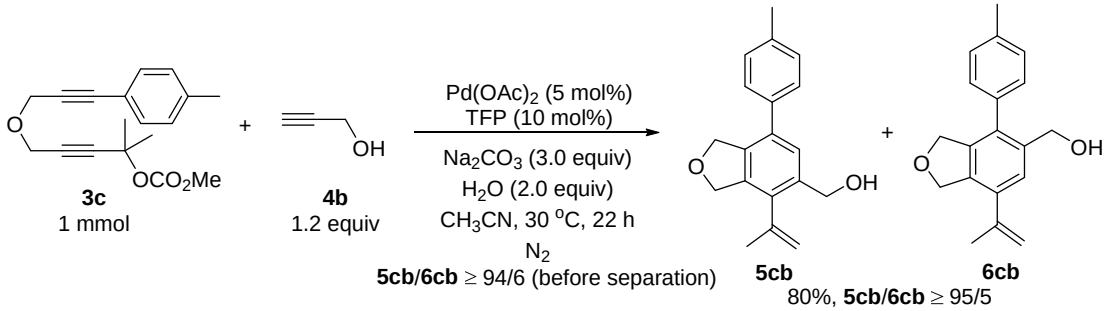


The reaction of Na_2CO_3 (159.0 mg, 1.5 mmol), $Pd(OAc)_2$ (5.6 mg, 0.025 mmol), TFP (11.6 mg, 0.05 mmol), **4a** (125.7 mg, 0.6 mmol), **3j** (163.4 mg, 0.5 mmol), and H_2O (18.1 mg, 1.0 mmol) in CH_3CN (5 mL) afforded **5ja**/**6ja** (121.1 mg, 53%, **5ja**/**6ja** = 90/10 as determined by ^{1}H NMR analysis of the isolated product) (eluent: petroleum ether (60-90 ^{o}C)/ethyl acetate = 10/1 (1100 mL) to 5/1 (480 mL)) (**5ja**/**6ja** = 88/12 as determined by ^{1}H NMR analysis of the crude product using mesitylene (23 μL) as the internal standard, recovery of **3j** = 11%).

5ja/**6ja** (90/10): solid; m.p. 152.2-153.8 ^{o}C (*n*-hexane/DCM); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.74 (d, *J* = 8.1 Hz, 2 H, ArH), 7.44-7.29 (m, 3 H, ArH), 7.26 (d, *J* = 8.1 Hz, 4 H, ArH), 7.14 (s, 1 H, ArH), 5.57-5.44 (m, 1 H, =CH), 5.19-5.16 (m, 2 H,

OCH_2), 5.03-4.93 (m, 2 H, OCH_2), 4.88 (t, J = 6.0 Hz, 1 H, NH), 4.22-4.01 (m, 2 H, NCH_2), 2.39 (s, 3 H, CH_3), 2.13-1.91 (m, 4 H, CH_2 × 2), 1.70-1.50 (m, 4 H, CH_2 × 2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.4, 139.4, 138.9, 136.8, 136.7, 136.6, 135.1, 134.4, 133.3, 129.6, 128.6, 128.5, 127.6, 127.5, 127.4, 127.1, 73.7, 72.9, 44.4, 29.3, 25.1, 22.6, 21.7, 21.4; IR (neat) ν (cm^{-1}) 3277, 2927, 2856, 1599, 1475, 1447, 1436, 1328, 1162, 1094, 1054; MS (EI): *m/z* (%) 459 (M^+, 0.69), 304 ($(M-Ts)^+$, 100); HRMS calcd. for $C_{28}H_{29}NO_3S$ [M^+]: 459.1868; Found: 459.1862. The following signals are discernible for **6ja**: ^1H NMR (300 MHz, $CDCl_3$) δ 7.53 (d, J = 8.4 Hz, 2 H, ArH), 7.17 (d, J = 8.4 Hz, 2 H, ArH), 5.74-5.66 (m, 1 H, =CH), 4.75-4.70 (m, 2 H, OCH_2), 4.66 (t, J = 6.0 Hz, 1 H, NH), 4.97 (d, J = 6.3 Hz, 2 H, NCH_2), 2.15-2.25 (m, 4 H, CH_2 × 2), 1.82-1.77 (m, 4 H, CH_2 × 2).

9. Synthesis of (4-(propen-2-yl)-7-(p-tolyl)-1,3-dihydroisobenzofuran-5-yl)methanol (**5cb**). (cfsy-zy-138, wwt-2-23)

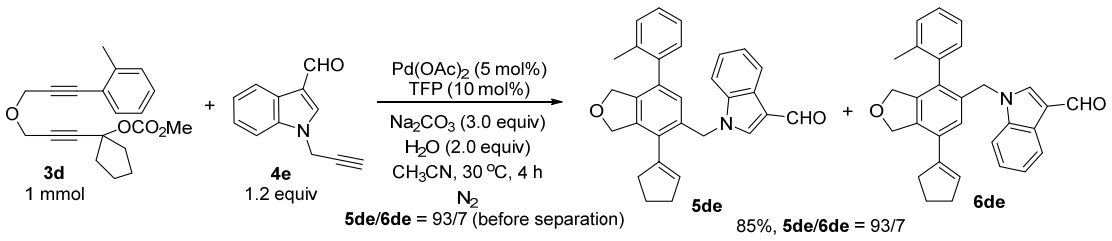


The reaction of Na_2CO_3 (317.5 mg, 3.0 mmol), $Pd(OAc)_2$ (11.3 mg, 0.05 mmol), TFP (23.4 mg, 0.1 mmol), **4b** (66.8 mg, 1.2 mmol), **3c** (300.7 mg, 1.0 mmol), and H_2O (36.0 μL, d = 1.0 g/mL, 2.0 mmol) in CH_3CN (10 mL) afforded **5cb** (224.5 mg, 80%, **5cb**/**6cb** ≥ 95/5 as determined by ^1H NMR analysis of the isolated product)

(eluent: petroleum ether (60-90 ^{o}C)/ethyl acetate = 20/1 (525 mL) to 5/1 (480 mL)) (**5cb**/**6cb** ≥ 94/6 as determined by ^{1}H NMR analysis of the crude product using mesitylene (46 μL) as the internal standard).

5cb: liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 (s, 1 H, ArH), 7.27 (d, *J* = 8.0 Hz, 2 H, ArH), 7.21 (d, *J* = 8.0 Hz, 2 H, ArH), 5.29-5.23 (m, 1 H, one proton of =CH_2), 5.21-5.14 (m, 2 H, OCH_2), 5.08-5.01 (m, 2 H, OCH_2), 4.91-4.85 (m, 1 H, one proton of =CH_2), 4.68 (s, 2 H, OCH_2), 2.38 (s, 3 H, CH_3), 2.23 (br s, 1 H, OH), 2.01 (s, 3 H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.5, 137.9, 137.5, 137.2, 136.8, 136.2, 135.3, 134.6, 129.3, 127.6, 115.9, 73.8, 72.9, 62.5, 23.7, 21.1; IR (neat) ν (cm^{-1}) 3412, 3076, 3024, 2915, 2858, 1640, 1518, 1477, 1444, 1370, 1330, 1286, 1136, 1057; MS (EI): *m/z* (%) 280 (M^+, 100); HRMS calcd. for $C_{19}H_{20}O_2$ [M^+]: 280.1463; Found: 280.1465. The following signals are discernible for **6cb**: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.10 (d, *J* = 8.0 Hz, 2 H, ArH), 5.01-4.98 (m, 1 H, one proton of =CH_2), 4.85-4.82 (m, 2 H, OCH_2), 4.49 (s, 2 H, OCH_2), 2.13 (s, 3 H, CH_3).

10. Synthesis of 1-((4-(cyclopentenyl)-7-(o-tolyl)-1,3-dihydroisobenzofuran-5-yl) methyl)-1*H*-indole-3-carbaldehyde (**5de**). (zyc-1-94)



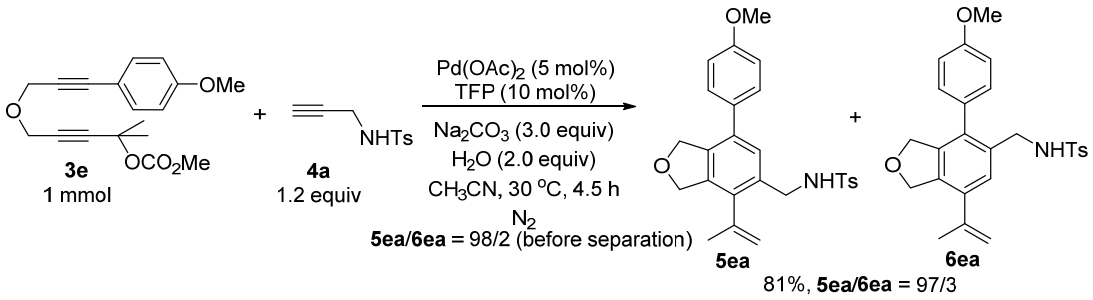
The reaction of **3d** (324.4 mg, 1.0 mmol), **4e** (219.8 mg, 1.2 mmol), $Pd(OAc)_2$ (11.1 mg, 0.05 mmol), TFP (23.7 mg, 0.1 mmol), Na_2CO_3 (317.8 mg, 3.0 mmol), and

H_2O (37.1 mg, 2.1 mmol) in CH_3CN (10 mL) afforded **5de**/**6de** (366.7 mg, 85%, **5de**/**6de** = 93/7 as determined by ^1H NMR analysis of the isolated product) (eluent: petroleum ether (60-90 ^oC)/ethyl acetate = 10/1 (550 mL) to 5/1 (720 mL)) (**5de**/**6de** = 93/7 as determined by ^1H NMR analysis of the crude product using mesitylene (46 μL) as the internal standard).

5de/**6de** (93/7): solid; ^1H NMR (300 MHz, $CDCl_3$) δ 9.93 (s, 1 H, CHO), 8.36-8.15 (m, 1 H, ArH), 7.59 (s, 1 H, ArH), 7.40-6.95 (m, 7 H, ArH), 6.84 (s, 1 H, ArH), 5.72-5.58 (m, 1 H, =CH), 5.33 (s, 2 H, NCH_2), 5.14-5.01 (m, 2 H, OCH_2), 4.97-4.85 (m, 2 H, OCH_2), 2.52-2.25 (m, 4 H, CH_2 × 2), 2.00 (s, 3 H, CH_3), 1.96-1.79 (m, 2 H, CH_2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 184.5, 139.6, 139.1, 138.6, 138.3, 137.3, 135.2, 134.7, 131.9, 131.7, 131.2, 130.3, 128.8, 128.6, 127.8, 125.7, 125.3, 123.9, 122.9, 122.0, 118.3, 110.2, 73.5, 73.4, 48.5, 36.1, 33.2, 23.6, 19.8; IR (KBr) ν (cm^{-1}) 3045, 2951, 2925, 2846, 1655, 1617, 1577, 1560, 1530, 1466, 1401, 1387, 1362, 1332, 1164, 1134, 1081, 1051, 1040; MS (EI): *m/z* (%) 433 (M^+, 100.0); Anal. Calcd. for $C_{30}H_{27}NO_2$ (%): C 83.11, H 6.28, N 3.23; Found: C 82.74, H 6.43, N 2.99. The following signals are discernible for **6de**: ^1H NMR (300 MHz, $CDCl_3$) δ 10.37 (s, 1 H, CHO), 8.64-8.55 (m, 1 H, ArH), 2.72-2.51 (m, 4 H, CH_2 × 2).

This mixture may further be purified by recrystallization to afford the pure product **5de**: m.p. 84.0-87.0 ^oC (DCM).

11. Synthesis of *N*-((7-(4-methoxyphenyl)-4-(propen-2-yl)-1,3-dihydroisobenzo-furan-5-yl)methyl)-4-methylbenzenesulfonamide (**5ea**). (zyc-1-101)



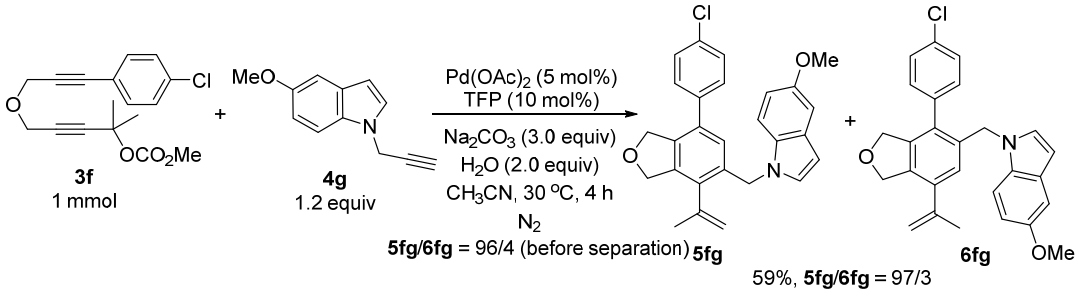
The reaction of **3e** (315.7 mg, 1.0 mmol), **4a** (252.2 mg, 1.2 mmol), $Pd(OAc)_2$ (11.4 mg, 0.05 mmol), TFP (23.9 mg, 0.1 mmol), Na_2CO_3 (318.4 mg, 3.0 mmol), and H_2O (37.0 mg, 2.0 mmol) in CH_3CN (10 mL) afforded **5ea**/**6ea** (364.0 mg, 81%, **5ea**/**6ea** = 97/3 as determined by ^1H NMR analysis of the isolated product) (eluent: petroleum ether (60-90 ^oC)/ethyl acetate = 5/1 (360 mL) to 3/1 (600 mL)) (**5ea**/**6ea** = 98/2 as determined by ^1H NMR analysis of the crude product using mesitylene (46 μL) as the internal standard).

5ea/**6ea** (97/3): solid; ^1H NMR (300 MHz, $CDCl_3$) δ 7.73 (d, *J* = 8.4 Hz, 2 H, ArH), 7.24 (d, *J* = 7.8 Hz, 2 H, ArH), 7.19 (d, *J* = 9.0 Hz, 2 H, ArH), 7.14 (s, 1 H, ArH), 6.91 (d, *J* = 9.0 Hz, 2 H, ArH), 5.24-5.14 (m, 2 H, one proton of $=CH_2$ and NH), 5.14-5.09 (m, 2 H, OCH_2), 5.01-4.94 (m, 2 H, OCH_2), 4.83-4.75 (m, 1 H, one proton of $=CH_2$), 4.13 (d, *J* = 5.7 Hz, 2 H, NCH_2), 3.83 (s, 3 H, CH_3), 2.38 (s, 3 H, CH_3), 1.87 (s, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.1, 143.3, 142.0, 138.1, 136.9, 136.3, 135.5, 134.3, 132.8, 131.7, 129.5, 128.7, 128.1, 127.1, 116.5, 114.0, 73.7, 72.8, 55.2, 44.2, 23.4, 21.4; IR (KBr) ν (cm^{-1}) 3265, 3005, 2963, 2927, 2895, 2834, 1654, 1643, 1610, 1519, 1482, 1450, 1438, 1412, 1348, 1325, 1290, 1251, 1211, 1179, 1159, 1117, 1093, 1054, 1036; MS (EI): *m/z* (%) 449 (M^+, 0.2), 294 ($(M\text{-}Ts)^+$, 100.0); Anal. Calcd. for $C_{26}H_{27}NO_4S$ (%): C 69.46, H 6.05, N 3.12; Found: C 69.46, H 6.14, N 2.86.

The following signals are discernible for **6ea**: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.54 (d, *J* = 8.1 Hz, 2 H, ArH), 4.93-4.91 (m, 2 H, OCH_2), 3.98 (d, *J* = 6.0 Hz, 2 H, NCH_2), 2.04 (s, 3 H, CH_3).

This mixture may further be purified by recrystallization to afford the pure product **5ea**: m.p. 152.4-154.1 ^{o}C (*n*-hexane/DCM).

12. Synthesis of 1-((7-(4-chlorophenyl)-4-(propen-2-yl)-1,3-dihydroisobenzofuran -5-yl)methyl)-5-methoxy-1*H*-indole (**5fg**). (zyc-1-87)



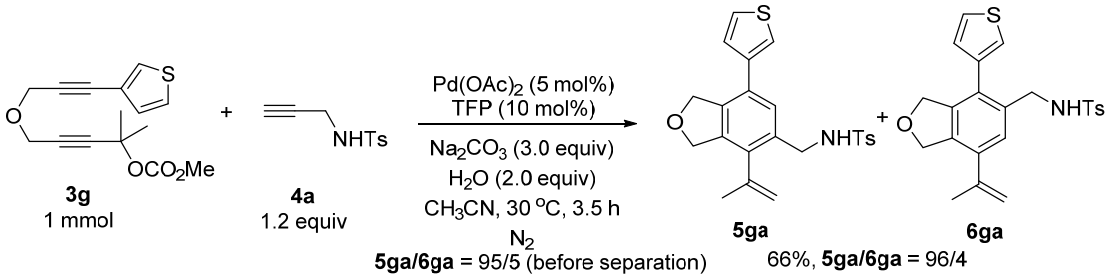
The reaction of Na_2CO_3 (318.2 mg, 3.0 mmol), $Pd(OAc)_2$ (11.5 mg, 0.05 mmol), TFP (24.8 mg, 0.1 mmol), **4g** (223.7 mg, 1.2 mmol), **3f** (320.8 mg, 1.0 mmol), and H_2O (36.8 mg, 2.0 mmol) in CH_3CN (10 mL) afforded **5fg**/**6fg** (252.3 mg, 59%, **5fg**/**6fg** = 97/3 as determined by ^{1}H NMR analysis of the isolated product) (The crude product was purified by column chromatography on silica gel (petroleum ether (60-90 ^{o}C)/ethyl acetate = 20/1) to afford impure **5fg**/**6fg** (281.1 mg), which was further purified with chromatography (petroleum ether (60-90 ^{o}C)/ethyl acetate = 40/1 (1000 mL) to 20/1 (700 mL)) (**5fg**/**6fg** = 96/4 as determined by ^{1}H NMR analysis of the crude product using mesitylene (46 μL) as the internal standard).

5fg/6fg (97/3): solid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.31 (d, *J* = 8.4 Hz, 2 H,

ArH), 7.18-7.10 (m, 3 H, ArH), 7.08 (d, J = 2.4 Hz, 1 H, ArH), 7.04 (d, J = 3.0 Hz, 1 H, ArH), 6.87-6.77 (m, 2 H, ArH), 6.44 (dd, J_1 = 3.3 Hz, J_2 = 0.6 Hz, 1 H, ArH), 5.36-5.31 (m, 1 H, one proton of $=CH_2$), 5.29 (s, 2 H, NCH_2), 5.18-5.12 (m, 2 H, OCH_2), 5.12-5.06 (m, 2 H, OCH_2), 5.01-4.93 (m, 1 H, one proton of $=CH_2$), 3.83 (s, 3 H, OCH_3), 1.91 (s, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 154.0, 142.0, 138.6, 137.9, 136.6, 136.0, 134.3, 133.58, 133.56, 131.6, 129.0, 128.9, 128.8, 128.6, 127.0, 116.4, 112.0, 110.3, 102.5, 101.3, 73.6, 73.0, 55.7, 47.4, 23.1; IR (KBr) ν (cm^{-1}) 3101, 2986, 2964, 2903, 2830, 1619, 1577, 1487, 1449, 1385, 1366, 1342, 1295, 1255, 1239, 1187, 1150, 1131, 1092, 1054, 1026, 1014; MS (EI): *m/z* (%) 431 ($M(^{37}Cl)^+$, 25.2), 429 ($M(^{35}Cl)^+$, 69.1), 414 (100.0); HRMS calcd. for $C_{27}H_{24}{}^{35}ClNO_2$ [M^+]: 429.1496, Found: 429.1493. The following signals are discernible for **6fg**: ^1H NMR (300 MHz, $CDCl_3$) δ 6.38 (dd, J_1 = 3.2 Hz, J_2 = 0.8 Hz, 1 H, ArH), 5.22-5.18 (m, 2 H, OCH_2), 5.06-5.04 (m, 2 H, OCH_2), 4.93-4.91 (m, 1 H, one proton of $=CH_2$), 3.86 (s, 3 H, OCH_3), 1.97 (s, 3 H, CH_3).

This mixture may further be purified by recrystallization to afford the pure product **5fg**: m.p. 146.2-147.7 ^oC (*n*-hexane/DCM).

13. Synthesis of *N*-((4-(propen-2-yl)-7-(thiophen-3-yl)-1,3-dihydroisobenzofuran-5-yl)methyl)-4-methylbenzenesulfonamide (**5ga**). (zyc-1-131)

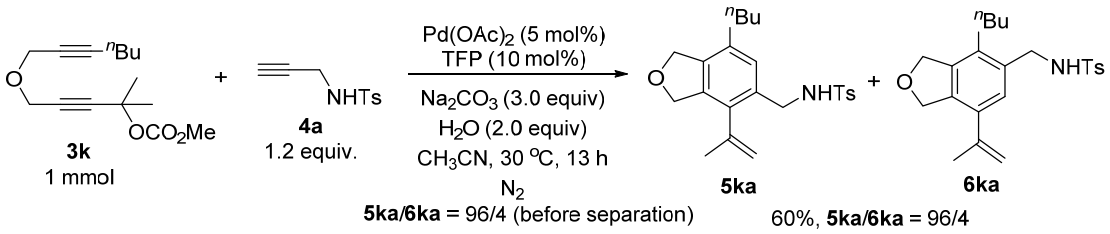


The reaction of Na_2CO_3 (318.2 mg, 3.0 mmol), $Pd(OAc)_2$ (11.4 mg, 0.05 mmol), TFP (24.1 mg, 0.1 mmol), **4a** (251.5 mg, 1.2 mmol), **3g** (292.1 mg, 1.0 mmol), and H_2O (37.0 mg, 2.0 mmol) in CH_3CN (10 mL) afforded **5ga**/**6ga** (281.8 mg, 66%, **5ga**/**6ga** = 96/4 as determined by ^1H NMR analysis of the isolated product) (The crude product was purified by column chromatography on silica gel (petroleum ether (60-90 ^oC)/ethyl acetate = 5/1 (500 mL) to 3/1 (300 mL)) to afford impure **5ga**/**6ga** (356.6 mg), which was further purified by recrystallization (*n*-hexane/DCM)) (**5ga**/**6ga** = 95/5 as determined by ^1H NMR analysis of the crude product using mesitylene (46 μL) as the internal standard).

5ga/6ga (96/4): solid; m.p. 146.2-148.3 ^oC (*n*-hexane/DCM); ^1H NMR (300 MHz, $CDCl_3$) δ 7.73 (d, *J* = 8.4 Hz, 2 H, ArH), 7.35 (t, *J* = 3.9 Hz, 1 H, ArH), 7.26 (d, *J* = 3.6 Hz, 2 H, ArH), 7.23 (s, 1 H, ArH), 7.13 (d, *J* = 4.2 Hz, 2 H, ArH), 5.29 (t, *J* = 5.9 Hz, 1 H, NH), 5.22-5.10 (m, 3 H, one proton of $=CH_2$ and OCH_2), 5.03-4.93 (m, 2 H, OCH_2), 4.82-4.73 (m, 1 H, one proton of $=CH_2$), 4.12 (d, *J* = 5.7 Hz, 2 H, CH_2), 2.39 (s, 3 H, CH_3), 1.86 (s, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.4, 141.8, 139.7, 138.2, 136.7, 135.9, 135.7, 132.8, 129.6, 129.0, 127.5, 127.0, 126.7, 126.1, 121.9, 116.5, 74.1, 72.9, 44.1, 23.4, 21.4; IR (KBr) ν (cm^{-1}) 3315, 3193, 2963, 2942, 2918, 2854, 1642, 1598, 1495, 1438, 1370, 1325, 1213, 1187, 1162, 1124, 1096, 1057, 1027; MS (EI): *m/z* (%) 425 (M^+, 0.07), 270 ($(M\text{-}Ts)^+$, 100.0); Anal. Calcd. for

$C_{23}H_{23}NO_3S_2$ (%): C 64.92, H 5.45, N 3.29, Found: C 64.57, H 5.49, N 3.21. The following signals are discernible for **6ga**: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.59 (d, J = 8.1 Hz, 2 H, ArH), 7.05-7.00 (m, 1 H, ArH), 6.88-6.83 (m, 1 H, ArH), 4.93-4.90 (m, 2 H, OCH_2), 4.02 (d, J = 5.7 Hz, 2 H, NCH_2), 2.03 (s, 3 H, CH_3).

14. Synthesis of *N*-((7-butyl-4-(propen-2-yl)-1,3-dihydroisobenzofuran-5-yl)methyl)-4-methylbenzenesulfonamide (**5ka**). (zyc-3-32)

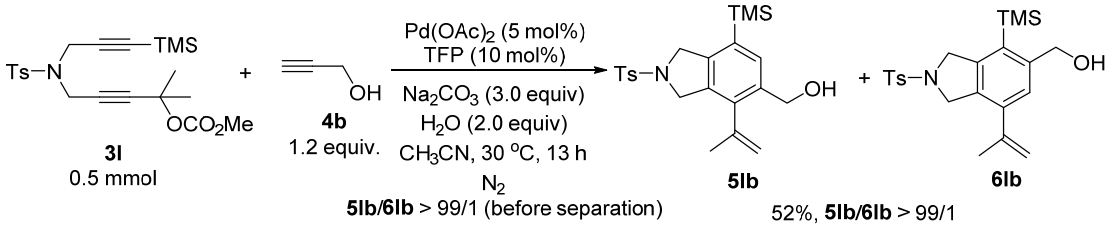


The reaction of Na_2CO_3 (318.0 mg, 3.0 mmol), $Pd(OAc)_2$ (11.2 mg, 0.05 mmol), TFP (23.3 mg, 0.1 mmol), **4a** (251.3 mg, 1.2 mmol), **3k** (266.2 mg, 1.0 mmol), and H_2O (36.1 mg, 2.0 mmol) in CH_3CN (10 mL) afforded **5ka**/**6ka** (238.8 mg, 60%, **5ka**/**6ka** = 96/4 as determined by ^{1}H NMR analysis of the isolated product) (eluent: petroleum ether (60-90 °C)/ethyl acetate = 10/1) (**5ka**/**6ka** = 96/4 as determined by ^{1}H NMR analysis of the crude product using mesitylene (46 μL) as the internal standard).

5ka/**6ka** (96/4): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.73 (d, J = 8.4 Hz, 2 H, ArH), 7.27 (d, J = 7.8 Hz, 2 H, ArH), 6.92 (s, 1 H, ArH), 5.37 (t, J = 5.3 Hz, 1 H, NH), 5.19-5.08 (m, 1 H, one proton of $=CH_2$), 5.08-4.96 (m, 2 H, OCH_2), 4.96-4.86 (m, 2 H, OCH_2), 4.75-4.65 (m, 1 H, one proton of $=CH_2$), 4.06 (d, J = 6.0 Hz, 2 H, NCH_2), 2.42 (s, 3 H, CH_3), 2.37 (t, J = 7.8 Hz, 2 H, CH_2), 1.82 (s, 3 H, CH_3), 1.55-1.38 (m, 2 H, CH_2), 1.38-1.21 (m, 2 H, CH_2), 0.90 (t, J = 7.2 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz,

$CDCl_3$) δ 143.1, 142.0, 137.0, 136.81, 136.76, 134.9, 134.3, 132.2, 129.4, 128.2, 127.0, 116.1, 73.0, 72.8, 44.0, 32.6, 31.9, 23.4, 22.4, 21.3, 13.7; IR (neat) ν (cm^{-1}) 3277, 2956, 2930, 2859, 1639, 1598, 1444, 1329, 1161, 1094, 1056; MS (EI): *m/z* (%) 399 (M^+, 0.04), 244 ($(M\text{-}Ts)^+$, 100); HRMS calcd. for $C_{23}H_{29}NO_3S$ [M^+]: 399.1868; Found: 399.1864. The following signals are discernible for **6ka**: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.00 (s, 1 H, ArH), 4.85-4.80 (m, 1 H, one proton of $=CH_2$), 1.98 (s, 3 H, CH_3).

15. Synthesis of (4-(propen-2-yl)-2-tosyl-7-(trimethylsilyl)isoindolin-5-yl)methanol (**5lb**). (zyc-3-33)

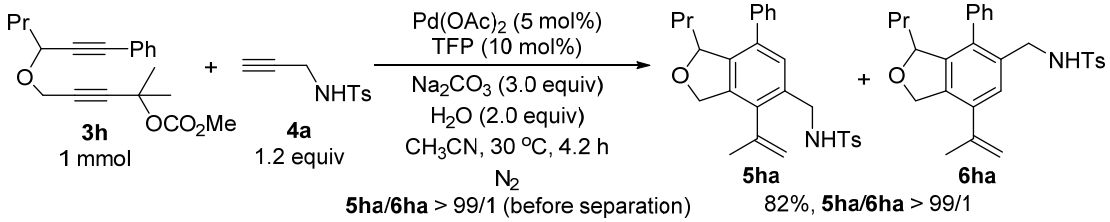


The reaction of Na_2CO_3 (158.9 mg, 1.5 mmol), $Pd(OAc)_2$ (5.5 mg, 0.025 mmol), TFP (11.6 mg, 0.05 mmol), **4b** (33.7 mg, 0.6 mmol), **3l** (227.1 mg, 0.5 mmol), and H_2O (18.1 mg, 1.0 mmol) in CH_3CN (5 mL) afforded **5lb**/**6lb** (108.7 mg, 52%, **5lb**/**6lb** > 99/1 as determined by ^{1}H NMR analysis of the isolated product) (eluent: petroleum ether (60-90 °C)/ethyl acetate = 10/1 (1000 mL) to 5/1 (600 mL)) (**5lb**/**6lb** > 99/1 as determined by ^{1}H NMR analysis of the crude product using mesitylene (23 μL) as the internal standard).

5lb: solid; m.p. 112.9-114.2 °C (*n*-hexane/DCM); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.75 (d, *J* = 8.1 Hz, 2 H, ArH), 7.44 (s, 1 H, ArH), 7.32 (d, *J* = 7.8 Hz, 2 H, ArH),

5.31-5.21 (m, 1 H, one proton of $=CH_2$), 4.83-4.76 (m, 1 H, one proton of $=CH_2$), 4.71-4.63 (m, 2 H, NCH_2), 4.63-4.54 (m, 2 H, NCH_2), 4.49 (s, 2 H, OCH_2), 2.40 (s, 3 H, CH_3), 1.96 (s, 4 H, OH and CH_3), 0.27 (s, 9 H, $CH_3 \times 3$); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.7, 142.1, 140.6, 138.7, 136.0, 133.7, 133.3, 133.1, 129.8, 127.4, 116.1, 62.5, 54.4, 52.4, 23.6, 21.4, -1.0; IR (neat) ν (cm^{-1}) 3527, 2954, 2908, 2856, 1637, 1597, 1383, 1347, 1251, 1163, 1098, 1066; MS (EI): *m/z* (%) 415 (M^+, 18.08), 91 (100); HRMS calcd. for $C_{22}H_{29}NO_3SSi$ [M^+]: 415.1637; Found: 415.1635.

16. Synthesis of *N*-((7-phenyl-4-(propen-2-yl)-1-propyl-1,3-dihydroisobenzofuran-5-yl)methyl)-4-methylbenzenesulfonamide (**5ha**). (zyc-1-71, wwt-2-132)

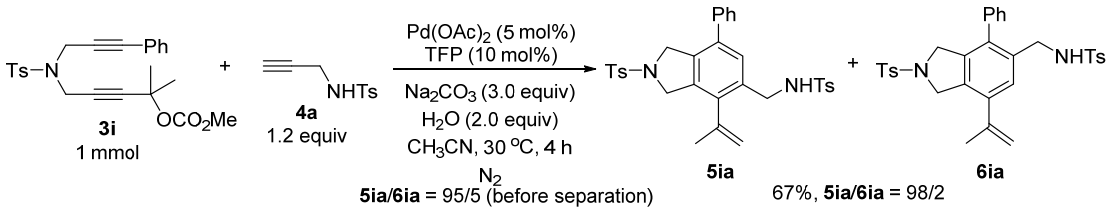


The reaction of **3h** (329.1 mg, 1.0 mmol), **4a** (251.5 mg, 1.2 mmol), $Pd(OAc)_2$ (11.2 mg, 0.05 mmol), TFP (23.8 mg, 0.1 mmol), Na_2CO_3 (318.7 mg, 3.0 mmol), and H_2O (35.8 mg, 2.0 mmol) in CH_3CN (10 mL) afforded **5ha** (378.7 mg, 82%, **5ha**/**6ha** > 99/1 as determined by ^1H NMR analysis of the isolated product) (eluent: petroleum ether (60-90 °C)/ethyl acetate = 10/1 (500 mL) to 5/1 (500 mL)) (**5ha**/**6ha** > 99/1 as determined by ^1H NMR analysis of the crude product using mesitylene (46 μL) as the internal standard).

5ha: solid; m.p. 127.4-130.2 °C (*n*-hexane/DCM); ^1H NMR (300 MHz, $CDCl_3$) δ 7.73 (d, *J* = 8.1 Hz, 2 H, ArH), 7.44-7.33 (m, 3 H, ArH), 7.28-7.20 (m, 4 H, ArH),

7.02 (s, 1 H, ArH), 5.66-5.56 (m, 1 H, OCH), 5.23-5.16 (m, 1 H, one proton of $=CH_2$), 5.04-4.92 (m, 2 H, OCH_2), 4.86-4.78 (m, 1 H, one proton of $=CH_2$), 4.75 (t, J = 6.0 Hz, 1 H, NH), 4.15 (d, J = 6.3 Hz, 2 H, NCH_2), 2.39 (s, 3 H, CH_3), 1.91 (s, 3 H, CH_3), 1.33-1.02 (m, 4 H, $CH_2 \times 2$), 0.67 (t, J = 7.1 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.3, 141.9, 139.4, 139.2, 138.2, 136.8, 135.9, 135.4, 132.6, 129.5, 129.3, 128.4, 128.0, 127.4, 127.1, 116.5, 84.0, 71.6, 44.2, 35.9, 23.4, 21.4, 18.1, 13.6; IR (neat) ν (cm^{-1}) 3280, 3061, 3030, 2957, 2929, 2871, 1641, 1599, 1495, 1446, 1329, 1289, 1160, 1094, 1072; MS (EI): *m/z* (%): 461 (M^+, 0.07), 418 ($(M-C_3H_7)^+$, 20.6), 306 (100); Anal. Calcd. for $C_{28}H_{31}NO_3S$ (%): C 72.85, H 6.77, N 3.03; Found: C 72.49, H 6.64, N 2.62.

17. Synthesis of *N*-((7-phenyl-4-(propen-2-yl)-2-(p-tosyl)isoindolin-5-yl)methyl)-4-methylbenzenesulfonamide (**5ia**). (wwt-2-31, zyc-1-143)



The reaction of Na_2CO_3 (318.6 mg, 3.0 mmol), $Pd(OAc)_2$ (11.4 mg, 0.05 mmol), TFP (23.1 mg, 0.1 mmol), **4a** (251.2 mg, 1.2 mmol), **3i** (440.8 mg, 1.0 mmol), and H_2O (36.2 mg, 2.0 mmol) in CH_3CN (10 mL) afforded **5ia/6ia** (382.8 mg, 67%, **5ia**/**6ia** = 98/2 as determined by ^1H NMR analysis of the isolated product) (eluent: petroleum ether (60-90 °C)/ethyl acetate = 7/1 to 5/1 to petroleum ether (60-90 °C)/ethyl acetate/DCM = 6/1/1) (**5ia**/**6ia** = 95/5 as determined by ^1H NMR analysis of

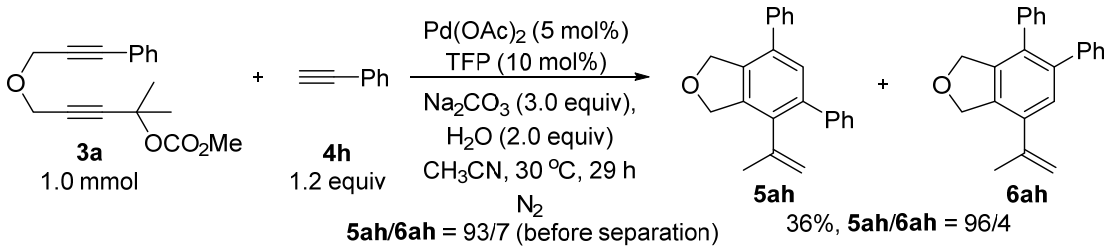
the crude product using mesitylene (46 μL) as the internal standard).

5ia/6ia (98/2): solid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.77-7.64 (m, 4 H, ArH), 7.46-7.16 (m, 9 H, ArH), 7.08 (s, 1 H, ArH), 5.26-5.18 (m, 1 H, one proton of $=CH_2$), 4.79-4.74 (m, 1 H, one proton of $=CH_2$), 4.70 (t, *J* = 5.7 Hz, 1 H, NH), 4.64-4.57 (m, 2 H, NCH_2), 4.52-4.45 (m, 2 H, NCH_2), 4.08 (d, *J* = 6.0 Hz, 2 H, NCH_2), 2.40 (s, 3 H, CH_3), 2.38 (s, 3 H, CH_3), 1.87 (s, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.7, 143.5, 141.4, 138.8, 137.2, 136.7, 136.1, 135.0, 133.6, 133.41, 133.37, 129.8, 129.6, 129.0, 128.6, 127.8, 127.7, 127.5, 127.0, 117.0, 53.6, 52.9, 44.1, 23.4, 21.4; IR (neat) ν (cm^{-1}) 3301, 2968, 2923, 2847, 1597, 1493, 1477, 1426, 1344, 1329, 1157, 1095, 1067; MS (EI): *m/z* (%) 572 (M^+, 2.87), 417 (100); Anal. Calcd. for $C_{32}H_{32}N_2O_4S_2$ (%): C 67.11, H 5.63, N 4.89; Found: C 66.97, H 5.86, N 4.81. The following signals are discernible for **6ia**: ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.24-4.21 (m, 2 H, NCH_2), 3.89 (d, *J* = 6.3 Hz, 2 H, CH_2), 1.75 (s, 3 H, CH_3).

This mixture may further be purified by recrystallization to afford the pure product **5ia**: m.p. 160.7-163.0 ^{o}C (*n*-hexane/DCM).

Control experiments

1. Synthesis of 5,7-diphenyl-4-(propen-2-yl)-1,3-dihydroisobenzofuran (**5ah**). (zyc-1-113)



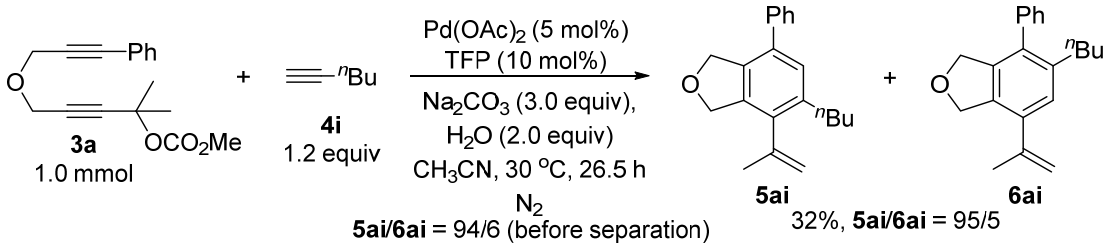
Following **Typical Procedure I**, the reaction of **3a** (286.1 mg, 1.0 mmol), **4h** (122.8 mg, 1.2 mmol), $Pd(OAc)_2$ (11.1 mg, 0.05 mmol), TFP (24.0 mg, 0.1 mmol), Na_2CO_3 (317.4 mg, 3.0 mmol), and H_2O (36.1 mg, 2.0 mmol) in CH_3CN (10 mL) afforded **5ah**/**6ah** (119.7 mg, purity = 93%, 36%, **5ah**/**6ah** = 96/4 as determined by ^1H NMR analysis of the isolated product) (The crude product was purified by column chromatography on silica gel (petroleum ether (60-90 ^oC)/ethyl acetate = 60/1) to afford impure **5ah**/**6ah** (determined by TLC). The impure **5ah**/**6ah** were purified by column chromatography on silica gel (petroleum ether (60-90 ^oC)/ethyl acetate = 100/1) to afford pure **5ah**/**6ah** and impure **5ah**/**6ah** (determined by TLC). The impure part was further purified by chromatography on silica gel (petroleum ether/ethyl acetate = 100/1) to afford another part of pure **5ah**/**6ah**.) (**5ah**/**6ah** = 93/7 as determined by ^1H NMR analysis of the crude product using mesitylene (46 μL) as the internal standard).

5ah/**6ah** (96/4): solid; ^1H NMR (300 MHz, $CDCl_3$) δ 7.55-7.27 (m, 11 H, ArH), 5.34-5.26 (m, 2 H, OCH_2), 5.25-5.19 (m, 1 H, one proton of $=CH_2$), 5.19-5.14 (m, 2 H, OCH_2), 5.07-5.02 (m, 1 H, one proton of $=CH_2$), 1.61 (s, 3 H, CH_3); ^{13}C NMR (75

MHz, $CDCl_3$) δ 143.6, 141.1, 139.8, 138.7, 136.2, 135.3, 134.5, 129.8, 129.1, 128.7, 128.0, 127.8, 127.5, 127.0, 116.9, 74.0, 73.8, 23.1; IR (KBr) ν (cm^{-1}) 3075, 3054, 3022, 2966, 2942, 2899, 2826, 1654, 1633, 1598, 1561, 1497, 1464, 1442, 1388, 1367, 1356, 1259, 1144, 1061, 1024; MS (EI): *m/z* (%) 312 (M^+, 65.83), 267 (100). The following signals are discernible for **6ah**: ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.40-5.36 (m, 2 H, OCH_2), 4.93-4.91 (m, 1 H, one proton of OCH_2).

This mixture may further be purified by recrystallization to afford the pure product **5ah**: m.p. 87.2-89.2 ^{o}C ($CHCl_3$); Anal. Calcd. for $C_{23}H_{20}O$ (%): C 88.43, H 6.45; Found: C 88.20, H 6.51.

2. Synthesis of 5-butyl-7-phenyl-4-(propen-2-yl)-1,3-dihydroisobenzofuran (**5ai**). (zyc-2-28)

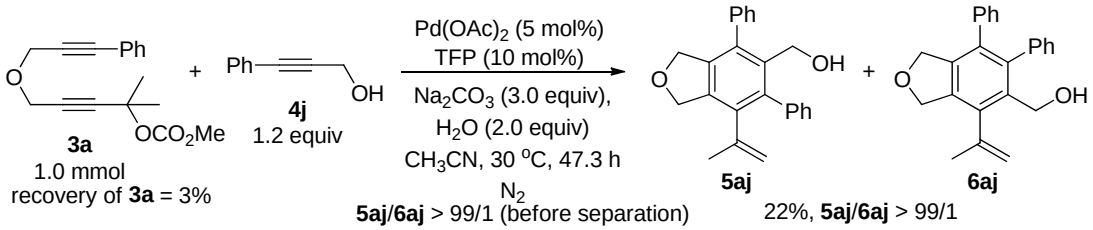


Following **Typical Procedure I**, the reaction of **3a** (285.9 mg, 1.0 mmol), **4i** (99.1 mg, 1.2 mmol), $Pd(OAc)_2$ (11.7 mg, 0.05 mmol), TFP (24.3 mg, 0.1 mmol), Na_2CO_3 (319.0 mg, 3.0 mmol), and H_2O (35.7 mg, 2.0 mmol) in CH_3CN (10 mL) afforded **5ai**/**6ai** (98.4 mg, purity = 95%, 32%, **5ai**/**6ai** = 95/5 as determined by ^{1}H NMR of the isolated product) (The crude product was purified by column chromatography on silica gel (petroleum ether (60-90 ^{o}C)/ethyl acetate = 100/1) to afford impure **5ai**/**6ai** (163.4 mg), which was further purified by column

chromatography on silica gel (petroleum ether (60-90 ^{o}C)/ethyl acetate = 100/1)) (**5ai**/**6ai** = 94/6 as determined by ^{1}H NMR analysis of the crude product using mesitylene (46 μL) as the internal standard).

5ai/**6ai** (95/5): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.48-7.27 (m, 5 H, ArH), 7.20 (s, 1 H, ArH), 5.28-5.23 (m, 1 H, one proton of $=CH_2$), 5.23-5.18 (m, 2 H, OCH_2), 5.10-5.03 (m, 2 H, OCH_2), 4.92-4.85 (m, 1 H, one proton of $=CH_2$), 2.63 (t, *J* = 8.0 Hz, 2 H, CH_2), 2.02 (t, *J* = 1.2 Hz, 3 H, CH_3), 1.64-1.52 (m, 2 H, CH_2), 1.47-1.31 (m, 2 H, CH_2), 0.93 (t, *J* = 7.4 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.1, 140.2, 139.6, 137.8, 136.1, 134.20, 134.18, 128.6, 128.5, 127.7, 127.2, 115.5, 73.9, 73.3, 34.3, 32.0, 23.8, 22.8, 14.0; IR (neat) ν (cm^{-1}) 3076, 3028, 2956, 2925, 2858, 1640, 1601, 1569, 1500, 1475, 1446, 1373, 1363, 1338, 1287, 1136, 1103, 1089, 1058, 1032; MS (EI): *m/z* (%) 292 (M^+, 100); HRMS calcd. for $C_{21}H_{24}O$ [M^+]: 292.1827, Found: 292.1826. The following signals are discernible for **6ai**: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.12 (s, 1 H, ArH), 4.84-4.79 (m, 1 H, one proton of $=CH_2$), 2.47 (t, *J* = 7.0 Hz, 2 H, CH_2), 2.16-2.13 (m, 3 H, CH_3), 0.77 (t, *J* = 7.2 Hz, 3 H, CH_3).

3. Synthesis of (4,6-diphenyl-7-(propen-2-yl)-1,3-dihydroisobenzofuran-5-yl)meth -anol (**5aj**). (zyc-2-35)

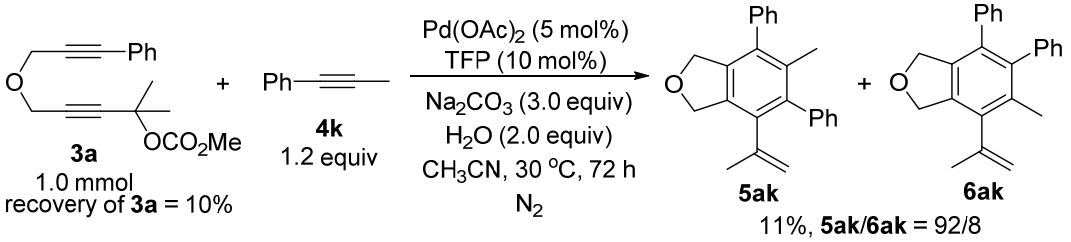


Following **Typical Procedure I**, the reaction of **3a** (286.5 mg, 1.0 mmol), **4j**

(159.4 mg, 1.2 mmol), $Pd(OAc)_2$ (11.9 mg, 0.05 mmol), TFP (23.4 mg, 0.1 mmol), Na_2CO_3 (318.7 mg, 3.0 mmol), and H_2O (36.1 mg, 2.0 mmol) in CH_3CN (10 mL) afforded **5aj** (75.4 mg, 22%, **5aj**/**6aj** > 99/1 as determined by ^1H NMR of the isolated product) (eluent: petroleum ether (60-90 ^oC)/ethyl acetate = 10/1) (**5aj**/**6aj** > 99/1 as determined by ^1H NMR of the crude product using mesitylene (46 μL) as the internal standard, recovery of **3a** = 3%).

5aj: solid; m.p. 146.6-148.5 ^oC (DCM/hexane); ^1H NMR (300 MHz, $CDCl_3$) δ 7.53-7.28 (m, 10 H, ArH), 5.18-5.12 (m, 2 H, OCH_2), 5.08-5.02 (m, 1 H, one proton of $=CH_2$), 4.97-4.91 (m, 2 H, OCH_2), 4.88-4.81 (m, 1 H, one proton of $=CH_2$), 4.22 (d, J = 6.0 Hz, 2 H, OCH_2), 1.58 (t, J = 1.1 Hz, 3 H, CH_3), 1.32 (t, J = 6.0 Hz, 1 H, OH); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.4, 140.3, 138.9, 138.5, 137.8, 136.8, 136.5, 135.8, 135.2, 130.2, 128.8, 128.4, 127.7, 127.5, 127.1, 116.5, 74.1, 74.0, 59.5, 23.2; IR (KBr) ν (cm^{-1}) 3447, 3074, 3060, 3028, 2951, 2911, 2860, 1639, 1598, 1497, 1469, 1441, 1364, 1310, 1272, 1205, 1177, 1049, 1024, 1001; MS (EI): *m/z* (%) 342 (M^+, 19.42), 279 (100); HRMS calcd. for $C_{24}H_{22}O_2$ [M^+]: 342.1620, Found: 342.1620.

4. Synthesis of 5-methyl-4,6-diphenyl-7-(propen-2-yl)-1,3-dihydroisobenzofuran (**5ak**). (zyc-2-51, zyc-2-60)



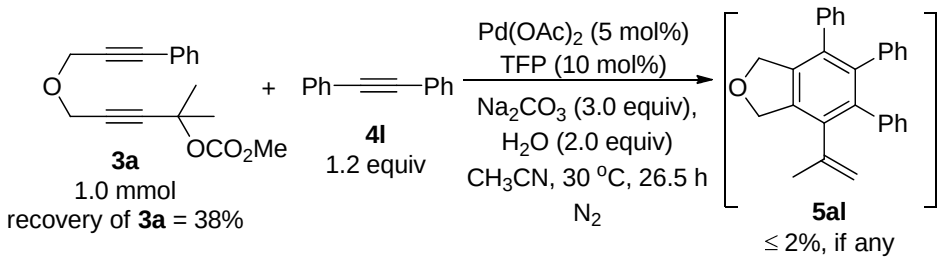
Following **Typical Procedure I**, the reaction of **3a** (286.2 mg, 1.0 mmol), **4k**

(143.4 mg, 1.2 mmol), $Pd(OAc)_2$ (11.3 mg, 0.05 mmol), TFP (23.8 mg, 0.1 mmol), Na_2CO_3 (318.3 mg, 3.0 mmol), and H_2O (36.1 mg, 2.0 mmol) in CH_3CN (10 mL) afforded **5ak/6ak** (36.0 mg, 11%, **5ak**/**6ak** = 92/8 as determined by ^1H NMR analysis of the isolated product) (eluent: petroleum ether (60-90 ^oC)/ethyl acetate = 100/1) (Recovery of **3a** = 10% as determined by ^1H NMR of the crude product using mesitylene (46 μL) as the internal standard).

5ak/**6ak** (92/8): solid; ^1H NMR (300 MHz, $CDCl_3$) δ 7.48-7.18 (m, 10 H, ArH), 5.17-5.09 (m, 2 H, OCH_2), 5.05-4.99 (m, 1 H, one proton of $=CH_2$), 4.95-4.89 (m, 2 H, OCH_2), 4.87-4.81 (m, 1 H, one proton of $=CH_2$), 1.85 (s, 3 H, CH_3), 1.57 (s, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.9, 140.5, 139.8, 139.7, 137.3, 136.2, 134.7, 134.2, 133.3, 130.1, 128.7, 128.5, 127.8, 127.2, 126.7, 116.2, 74.3, 74.1, 23.3, 18.4; IR (KBr) ν (cm^{-1}) 3075, 3045, 3019, 2959, 2899, 2850, 1493, 1439, 1371, 1358, 1330, 1289, 1261, 1220, 1177, 1056, 1027; GC-MS (GC condition: injector: 280 ^oC; column: DB-5, temperature: 60 ^oC (2 min), 20 ^oC/min to 280 ^oC, 280 ^oC (30 min)) (EI) *m/z* (%) for **5ak**: T_R 5.6 min: 326 (M^+, 100), 311 ($(M-CH_3)^+$, 29.97); for **6ak**: T_R 5.4 min: 326 (M^+, 100), 311 ($(M-CH_3)^+$, 18.14); HRMS calcd. for $C_{24}H_{22}O$ [M^+]: 326.1671, Found: 326.1672. The following signals are discernible for **6ak**: ^1H NMR (300 MHz, $CDCl_3$) δ 7.19-7.06 (m, 6 H, ArH), 7.03-6.96 (m, 4 H, ArH), 5.30-5.25 (m, 1 H, one proton of $=CH_2$), 4.98-4.95 (m, 2 H, OCH_2), 2.05 (s, 6 H, $CH_3 \times 2$).

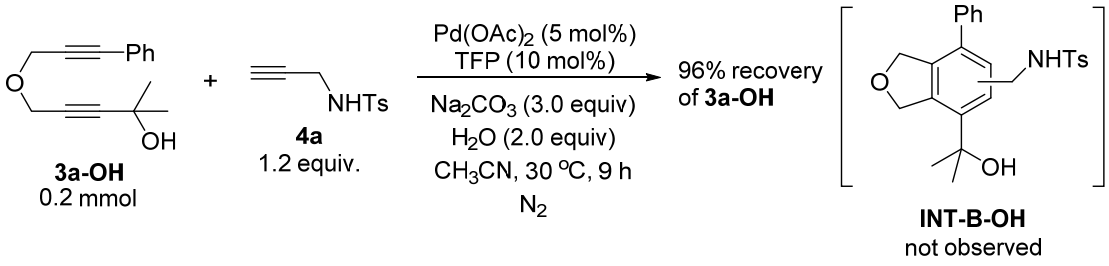
This mixture may further be purified by recrystallization to afford the pure product **5ak**: m.p. 146.8-148.3 ^oC (DCM/*n*-hexane).

5. The reaction of **3a** and **4l** under the standard conditions. (zyc-2-138)



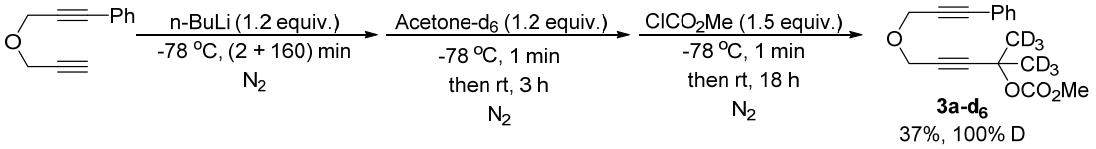
Following **Typical Procedure I**, the reaction of **3a** (286.1 mg, 1.0 mmol), **4l** (213.8 mg, 1.2 mmol), $Pd(OAc)_2$ (11.1 mg, 0.05 mmol), TFP (23.8 mg, 0.1 mmol), Na_2CO_3 (317.9 mg, 3.0 mmol), and H_2O (35.9 mg, 2.0 mmol) in CH_3CN (10 mL) afforded 7.3 mg of a complicated mixture (**5al,** ≤ 2%, if any) with 38% recovery of **3a** as determined by ^1H NMR analysis of the crude residue using mesitylene (46 μL) as the internal standard.

6. The reaction of **3a-OH** and **4a** under the standard conditions. (zyc-3-31)



Following **Typical Procedure I**, the reaction of **3a-OH** (45.5 mg, 0.2 mmol), **4a** (50.3 mg, 0.24 mmol), $Pd(OAc)_2$ (2.2 mg, 0.01 mmol), TFP (4.7 mg, 0.02 mmol), Na_2CO_3 (63.7 mg, 0.6 mmol), and H_2O (7.2 μL, d = 1.0 g/mL, 7.2 mg, 0.4 mmol) in CH_3CN (2 mL) afforded 96% recovery of **3a-OH** as determined by ^1H NMR analysis of the crude residue using mesitylene (9.2 μL) as the internal standard.

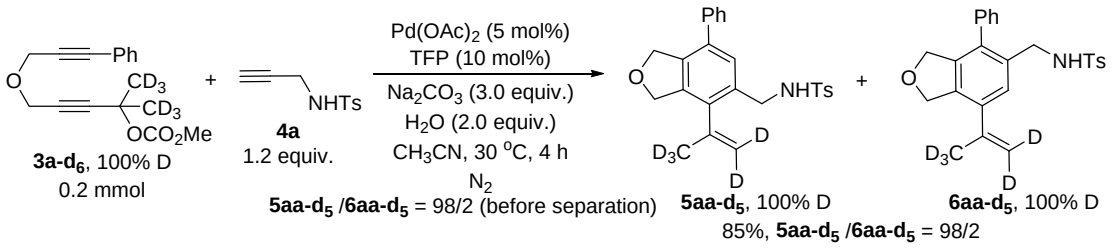
7. Synthesis of starting material **3a-d_6**. (zyc-2-74)



To a solution of 3-(propargyloxy)-phenyl-1-propyne (942.7 mg, 5.54 mmol) in THF (13 mL) were added dropwise *n*-BuLi (2.5 M in hexane, 2.64 mL, 6.6 mmol) at -78 °C within 2 minutes under N_2 atmosphere. After lithiation for 160 minutes at -78 °C, acetone-d_6 (0.5 mL, d = 0.852 g/mL, 426.0 mg, 6.64 mmol, 99.8% D) was added dropwise with an addition funnel at -78 °C within 1 minute. After the cooling bath was removed, the reaction mixture was warmed up to room temperature and stirred for 3 h. Then methyl chloroformate (0.64 mL, d = 1.22 g/mL, 780.8 mg, 8.26 mmol) was added dropwise at -78 °C within 1 minute. After the cooling bath was removed, the reaction mixture was warmed up to room temperature and stirred for 18 h. After the reaction was complete as monitored by TLC (eluent: petroleum ether/ ethyl acetate = 10/1), it was quenched with an aqueous solution of saturated NH_4Cl (10 mL). The resulting mixture was extracted with ethyl acetate (10 mL × 3) and the combined organic phase was dried over anhydrous Na_2SO_4. After filtration and evaporation of the solvent, the crude residue was purified by chromatography on silica gel (eluent: petroleum ether (60-90 °C)/ ethyl acetate = 100/1 (500 mL), and then petroleum ether/ ethyl acetate/diethyl ether = 100/1/1 (1500 mL) to 100/4/1 (800 mL)) to afford **3a-d_6** (594.3 mg, 37%, 100% D) as a liquid: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.49-7.41 (m, 2 H, ArH), 7.36-7.27 (m, 3 H, ArH), 4.47 (s, 2 H, OCH_2), 4.36 (s, 2 H, OCH_2), 3.76 (s, 3 H, OCH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 153.5, 131.7, 128.5, 128.2, 122.5, 87.3,

86.7, 84.3, 80.0, 73.8, 57.2, 56.8, 54.3, 27.8 (septet); IR (neat) ν (cm^{-1}) 3054, 3022, 2955, 2898, 2852, 1755, 1599, 1490, 1442, 1383, 1351, 1271, 1233, 1194, 1119, 1083, 1013; MS (EI): *m/z* (%) 292 (M^+, 1.39), 115 (100); HRMS calcd. for $C_{17}H_{12}D_6O_4$[M^+]: 292.1582, Found: 292.1582.

8. Synthesis of *N*-((7-phenyl-4-(propen-2-yl-d_5)-1,3-dihydroisobenzofuran-5-yl)methyl)-4-methylbenzenesulfonamide (**5aa-d$_5$**). (zyc-2-76)



Following **Typical Procedure I**, the reaction of **3a-d$_6$** (58.6 mg, 0.2 mmol), **4a** (50.4 mg, 0.24 mmol), $Pd(OAc)_2$ (2.3 mg, 0.01 mmol), TFP (4.8 mg, 0.02 mmol), Na_2CO_3 (63.6 mg, 0.4 mmol), and H_2O (7.2 μL, d = 1.00 g/mL, 7.2 mg, 0.4 mmol) in CH_3CN (2 mL) afforded **5aa-d$_5$**/**6aa-d$_5$** (72.4 mg, 85%, 100% D, **5aa-d$_5$**/**6aa-d$_5$** = 98/2 as determined by ^{1}H NMR analysis of the isolated product) (eluent: petroleum ether (60-90 ^{o}C)/ethyl acetate = 5/1) (**5aa-d$_5$**/**6aa-d$_5$** = 98/2 as determined by ^{1}H NMR analysis of the crude product using mesitylene (9.2 μL) as the internal standard).

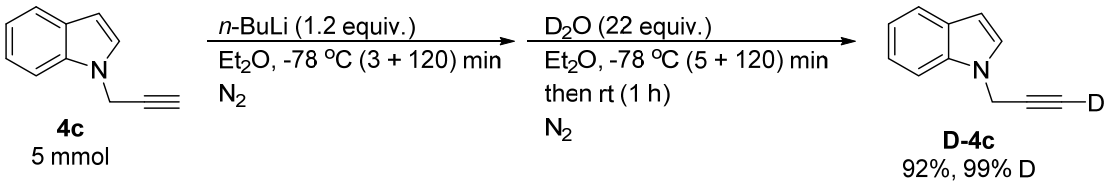
5aa-d$_5$/**6aa-d$_5$** (98/2): solid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.72 (d, *J* = 8.1 Hz, 2 H, ArH), 7.42-7.10 (m, 8 H, ArH), 5.36-5.20 (t, *J* = 6.0 Hz, 1 H, NH), 5.15-5.04 (m, 2 H, OCH_2), 5.03-4.90 (m, 2 H, OCH_2), 4.14 (d, *J* = 5.7 Hz, 2 H, NCH_2), 2.36 (s, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.3, 141.6, 139.3, 138.1, 136.9, 136.5, 135.9, 134.6, 133.0, 129.5, 128.5, 128.4, 127.6, 127.4, 127.0, 116.0 (pentet), 73.6, 72.8, 44.1,

22.3 (septet); IR (KBr) ν (cm^{-1}) 3259, 3054, 2901, 2830, 1598, 1495, 1470, 1453, 1330, 1306, 1153, 1114, 1096, 1073, 1050; MS (EI): *m/z* (%) 424 (M^+, 0.14), 269 ($(M-Ts)^+$, 100); HRMS calcd. for $C_{25}H_{20}D_5NO_3S$ [M^+]: 424.1869, Found: 424.1870. The following signals are discernible for **6aa**: ^1H NMR (300 MHz, $CDCl_3$) δ 7.53 (d, *J* = 8.4 Hz, 2 H, ArH), 4.74-4.70 (m, 2 H, CH_2), 3.96 (d, *J* = 5.7 Hz, 2 H, NCH_2).

This mixture may further be purified by recrystallization to afford the pure product **5aa-d$_5$**: m.p. 150.4-152.0 ^oC (*n*-hexane/DCM).

Deuterium labelling experiments

1. Synthesis of starting material **D-4c**. (zyc-3-35)

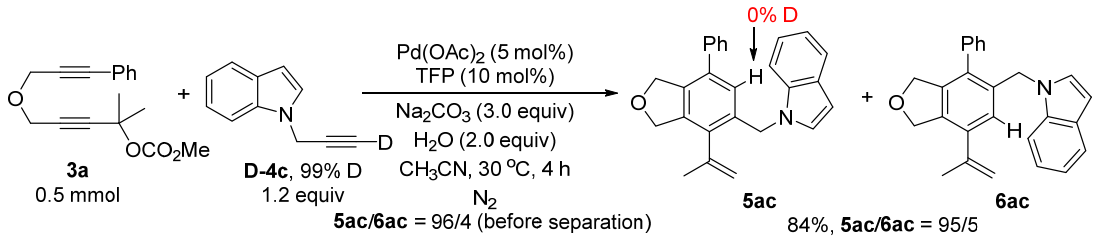


4c
5 mmol

D-4c
92%, 99% D

To a solution of **4c** (776.1 mg, 5.0 mmol) in Et_2O (30 mL) were added dropwise *n*-BuLi (2.5 M in hexane, 2.4 mL, 6.0 mmol) at -78 °C within 3 minutes under N_2 atmosphere. After lithiation for 120 minutes at -78 °C, D_2O (2.2 mL, d = 1.0 g/mL, 2200 mg, 110 mmol, 99.9% D) was added dropwise at -78 °C within 5 minutes. The resulting mixture was stirred for 2 hours. After the cooling bath was removed, the reaction mixture was warmed up to room temperature and stirred for 1 h. The resulting mixture was dried over anhydrous Na_2SO_4. After filtration and evaporation of the solvent, the crude residue was purified by chromatography on silica gel (eluent: petroleum ether (60-90 °C)/ ethyl acetate = 10/1) to afford **D-4c** (719.6 mg, 92%, 99% D) as a solid: m.p. 41.9-42.5 °C (*n*-hexane/Et_2O, -20 °C); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.64 (d, *J* = 7.8 Hz, 1 H, ArH), 7.40 (d, *J* = 8.1 Hz, 1 H, ArH), 7.30-7.21 (m, 1 H, ArH), 7.20 (d, *J* = 3.3 Hz, 1 H, ArH), 7.18-7.09 (m, 1 H, ArH), 6.57-6.51 (m, 1 H, ArH), 4.86 (s, 2 H, NCH_2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 135.7, 128.8, 127.2, 121.8, 121.1, 119.8, 109.3, 102.0, 77.2 (t, *J* = 7.6 Hz), 73.2 (t, *J* = 38.5 Hz), 35.7; IR (neat) ν (cm^{-1}) 3055, 2589, 1986, 1612, 1514, 1484, 1463, 1397, 1363, 1335, 1315, 1259, 1189, 1012; MS (EI): *m/z* (%) 156 (M^+, 61.71), 155 (100); HRMS calcd. for $C_{11}H_8DN$ [M^+]: 156.0798, Found: 156.0795. The following signal is discernible for **4c**: 2.32 (s, 1 H, ≡CH).

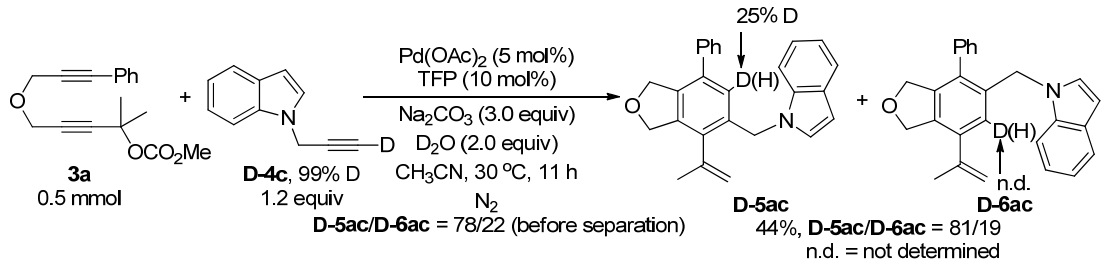
2. Synthesis of **5ac**. (zyc-3-36)



Following **Typical Procedure I**, the reaction of Na_2CO_3 (159.0 mg, 1.5 mmol), $Pd(OAc)_2$ (5.6 mg, 0.025 mmol), TFP (11.7 mg, 0.05 mmol), **D-4c** (93.8 mg, 0.6 mmol, 100% D), **3a** (143.0 mg, 0.5 mmol), and H_2O (18.1 mg, 1.0 mmol) in CH_3CN (5 mL) afforded **5ac**/**6ac** (153.4 mg, 84%, 0% D, **5ac**/**6ac** = 95/5 as determined by ^1H NMR analysis of the isolated product) (eluent: petroleum ether (60-90 ^oC)/ethyl acetate = 30/1) (**5ac**/**6ac** = 96/4 as determined by ^1H NMR analysis of the crude product using mesitylene (23 μL) as the internal standard).

5ac/**6ac** (95/5): solid; ^1H NMR (300 MHz, $CDCl_3$) δ 7.62 (d, *J* = 7.8 Hz, 1 H, ArH), 7.45-7.03 (m, 9 H, ArH), 6.93 (s, 1 H, ArH), 6.56-6.47 (m, 1 H, ArH), 5.37-5.28 (m, 3 H, one proton of $=CH_2$ and NCH_2), 5.23-5.17 (m, 2 H, OCH_2), 5.13-5.06 (m, 2 H, OCH_2), 5.02-4.95 (m, 1 H, one proton of $=CH_2$), 1.89 (s, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 142.1, 139.4, 138.4, 136.6, 136.3, 135.8, 134.8, 134.0, 128.6, 128.0, 127.6, 127.5, 127.3, 121.5, 120.9, 119.4, 116.3, 109.5, 101.7, 73.7, 73.0, 47.2, 23.1. The following signals are discernible for **6ac**: ^1H NMR (300 MHz, $CDCl_3$) δ 6.48-6.44 (m, 1 H, ArH), 5.46-5.38 (m, 3 H, one proton of $=CH_2$ and NCH_2), 5.15 (s, 2 H, OCH_2).

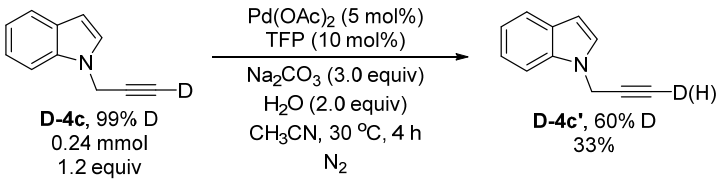
3. Synthesis of **D-5ac**. (zyc-3-46)



Following **Typical Procedure I**, the reaction of Na_2CO_3 (158.9 mg, 1.5 mmol), $Pd(OAc)_2$ (5.5 mg, 0.025 mmol), TFP (11.5 mg, 0.05 mmol), **D-4c** (93.7 mg, 0.6 mmol, 100% D), **3a** (143.0 mg, 0.5 mmol), and D_2O (20.1 mg, 1.0 mmol) in CH_3CN (5 mL) afforded **D-5ac**/**D-6ac** (81.1 mg, 44%, 25% D, **D-5ac**/**D-6ac** = 81/19 as determined by ^1H NMR analysis of the isolated product) (eluent: petroleum ether (60-90 ^oC)/ethyl acetate = 30/1) (**D-5ac**/**D-6ac** = 78/22 as determined by ^1H NMR analysis of the crude product using mesitylene (23 μL) as the internal standard).

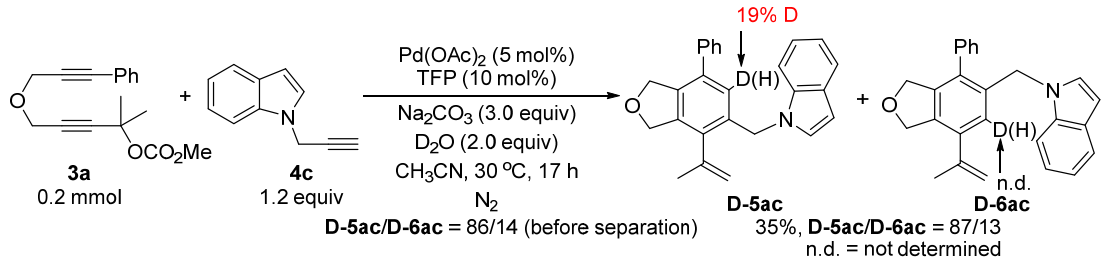
D-5ac/**D-6ac** (81/19): solid; ^1H NMR (300 MHz, $CDCl_3$) δ 7.61 (d, *J* = 7.8 Hz, 1 H, ArH), 7.45-7.01 (m, 9 H, ArH), 6.51 (d, *J* = 3.0 Hz, 1 H, ArH), 5.34-5.25 (m, 3 H, one proton of $=CH_2$ and NCH_2), 5.23-5.15 (m, 2 H, OCH_2), 5.12-5.04 (m, 2 H, OCH_2), 5.01-4.93 (m, 1 H, one proton of $=CH_2$), 1.88 (s, 3 H, CH_3); IR (neat) ν (cm^{-1}) 3056, 2912, 2852, 1640, 1611, 1512, 1475, 1462, 1362, 1317, 1191, 1060; MS (EI): *m/z* (%) 366 ($(M(D))^+$, 34.62), 365 ($(M(H))^+$, 67.29), 350 (100); HRMS calcd. for $C_{26}H_{22}DNO$ [M^+]: 366.1842, Found: 366.1836. The following signals are discernible for **D-6ac**: ^1H NMR (300 MHz, $CDCl_3$) δ 6.47-6.43 (m, 1 H, ArH), 5.45-5.39 (m, 2 H, NCH_2), 5.14 (s, 2 H, OCH_2). The following signal is discernible for **5ac**: 6.93 (s, 1 H, ArH).

4. Synthesis of **D-4c'**. (zyc-3-48)



Following **Typical Procedure I**, the reaction of Na_2CO_3 (63.6 mg, 0.6 mmol), $Pd(OAc)_2$ (2.2 mg, 0.01 mmol), TFP (4.6 mg, 0.02 mmol), **D-4c** (37.6 mg, 0.24 mmol), and H_2O (7.2 μL, d = 1.0 g/mL, 7.2 mg, 0.4 mmol) in CH_3CN (2 mL) afforded **D-4c'** (12.5 mg, 33%, 60% D) (eluent: petroleum ether (60-90 °C)/ethyl acetate = 20/1) as a solid: ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 (d, *J* = 7.6 Hz, 1 H, ArH), 7.40 (d, *J* = 8.4 Hz, 1 H, ArH), 7.28-7.21 (m, 1 H, ArH), 7.19 (d, *J* = 3.2 Hz, 1 H, ArH), 7.17-7.09 (m, 1 H, ArH), 6.53 (d, *J* = 3.2 Hz, 1 H, ArH), 4.88-4.82 (m, 2 H, NCH_2). The following signal is discernible for **4c**: 2.38 (t, *J* = 2.0 Hz, 1 H, ≡CH).

5. Synthesis of **D-5ac**. (zyc-3-41)



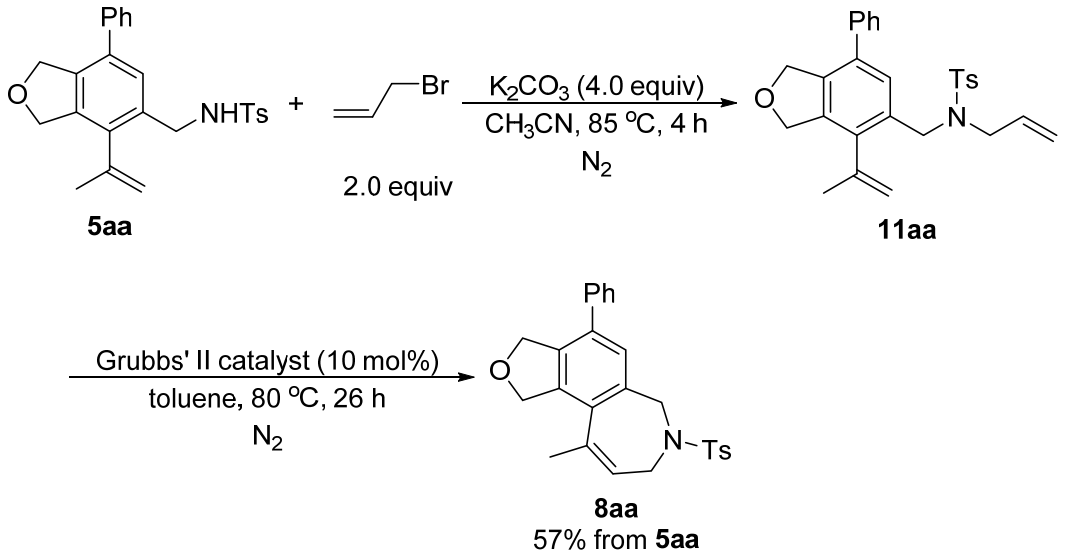
Following **Typical Procedure I**, the reaction of Na_2CO_3 (63.6 mg, 0.6 mmol), $Pd(OAc)_2$ (2.2 mg, 0.01 mmol), TFP (4.6 mg, 0.02 mmol), **4c** (37.2 mg, 0.24 mmol), **3a** (57.4 mg, 0.2 mmol), and D_2O (8.0 μL, d = 1.0 g/mL, 8.0 mg, 0.4 mmol) in CH_3CN (2 mL) afforded **D-5ac**/**D-6ac** (25.9 mg, 35%, 19% D, **D-5ac**/**D-6ac** = 87/13 as determined by ^{1}H NMR analysis of the isolated product) (eluent: petroleum ether (60-90 °C)/ethyl acetate = 30/1) (**D-5ac**/**D-6ac** = 86/14 as determined by ^{1}H NMR

analysis of the crude product using mesitylene (9.2 μL) as the internal standard).

D-5ac/**D-6ac** (87/13): solid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.63 (d, *J* = 7.8 Hz, 1 H, ArH), 7.48-7.04 (m, 9 H, ArH), 6.52 (d, *J* = 3.0 Hz, 1 H, ArH), 5.38-5.28 (m, 3 H, one proton of $=CH_2$ and NCH_2), 5.24-5.17 (m, 2 H, OCH_2), 5.13-5.07 (m, 2 H, OCH_2), 5.02-4.95 (m, 1 H, one proton of $=CH_2$), 1.90 (s, 3 H, CH_3). The following signals are discernible for **D-6ac**: ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.48-6.44 (m, 1 H, ArH), 5.46-5.42 (m, 2 H, NCH_2), 5.15 (s, 2 H, OCH_2). The following signal is discernible for **5ac**: 6.93 (s, 1 H, ArH).

Synthetic applications

1. Synthesis of 10-methyl-4-phenyl-7-(*p*-tosyl)-1,3,6,8-tetrahydro-1*H*-isobenzofuro-[5,4-*c*]azepine (**8aa**).[5,6] (zyc-1-148, zyc-1-164)



To a flame-dried Schlenk tube were added K_2CO_3 (429.6 mg, 3.12 mmol), **5aa** (327.1 mg, 0.78 mmol), and allyl bromide (188.6 mg, 1.56 mmol)/CH_3CN (2 mL) sequentially under N_2 atmosphere. Then the Schlenk tube was equipped with an internal condenser pipe and the resulting mixture was stirred with an oil bath pre-heated at 85 ^oC. The reaction was complete after being stirred for 4 hours as monitored by TLC. The reaction mixture was filtrated through a short column of silica gel eluted with ethyl acetate (5 mL × 3). After evaporation, the residue was purified by chromatography on silica gel to afford **11aa** (296.1 mg) (eluent: petroleum ether (60-90 ^oC)/ethyl acetate/DCM = 20/1/1)**:** solid; ^1H NMR (300 MHz, $CDCl_3$) δ 7.75 (d, *J* = 8.4 Hz, 2 H, ArH), 7.45-7.25 (m, 8 H, ArH), 5.63-5.45 (m, 1 H, =CH), 5.30-5.10 (m, 3 H, one proton of $=CH_2$ and OCH_2), 5.07-4.90 (m, 4 H, OCH_2 and $=CH_2$), 4.83 (s, 1 H, one proton of $=CH_2$), 4.44 (s, 2 H, NCH_2), 3.82 (d, *J* = 6.6 Hz, 2

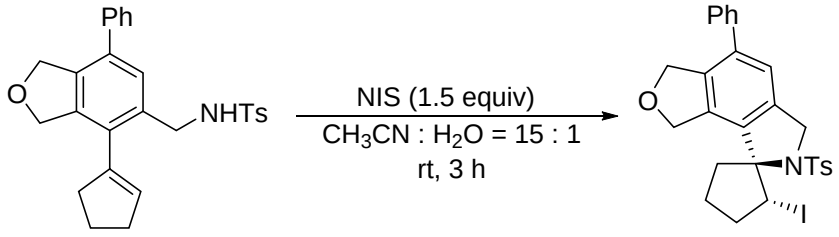
H, NCH_2), 2.39 (s, 3 H, CH_3), 1.95 (s, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.2, 141.8, 139.5, 138.1, 137.3, 136.1, 135.7, 134.5, 132.7, 132.4, 129.7, 128.5, 127.6, 127.4, 127.2, 127.1, 119.1, 116.3, 73.7, 72.9, 49.9, 47.0, 23.3, 21.4. which was used directly in the next step without further characterization.

To a flame-dried Schlenk tube were added Grubbs' II catalyst (17.1 mg, 0.02 mmol), **11aa** (94.9 mg, 0.21 mmol), and toluene (10 mL) sequentially under N_2 atmosphere. Then the Schlenk tube was equipped with an internal condenser pipe and the resulting mixture was stirred in an oil bath pre-heated at 80 ^oC. The reaction was complete after being stirred for 26 hours as monitored by TLC (eluent: petroleum ether (60-90 ^oC)/ethyl acetate/DCM = 20/1/1). The resulting mixture was filtrated through a short column of silica gel eluted with ethyl acetate (10 mL × 3). After evaporation, the residue was purified by chromatography on silica gel to afford **8aa** (61.1 mg, 57% from **5aa**) (eluent: petroleum ether (60-90 ^oC)/ethyl acetate/DCM = 20/1/1 to 10/1/1).

8aa: solid; m.p. 162.6-164.6 ^oC (DCM/hexane); ^1H NMR (300 MHz, $CDCl_3$) δ 7.73 (d, J = 8.4 Hz, 2 H, ArH), 7.49-7.34 (m, 3 H, ArH), 7.33-7.18 (m, 4 H, ArH), 6.95 (s, 1 H, ArH), 5.86 (tq, J_1 = 7.4 Hz, J_2 = 1.4 Hz, 1 H, =CH), 5.11 (s, 4 H, OCH_2 × 2), 4.22 (s, 2 H, NCH_2), 3.52 (brs, 2 H, NCH_2), 2.37 (s, 3 H, CH_3), 2.00 (d, J = 1.2 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.3, 141.0, 139.1, 137.9, 137.4, 136.2, 135.2, 133.5, 132.7, 129.6, 129.3, 128.6, 127.65, 127.56, 127.4, 122.3, 73.3, 72.8, 49.1, 42.9, 21.3, 21.2; IR (neat) ν (cm^{-1}) 3054, 3028, 2963, 2917, 2857, 1631, 1597, 1493, 1457, 1446, 1400, 1378, 1362, 1325, 1307, 1292, 1164, 1112, 1089, 1072, 1059;

MS (EI): *m/z* (%) 431 (M^+, 11.94), 276 ($(M\text{-}Ts)^+$, 100); HRMS calcd. for $C_{26}H_{25}NO_3S$ [M^+]: 431.1555, Found: 431.1558.

2. Synthesis of *trans*-2-iodo-4'-phenyl-7'-(p-tosyl)-1',3',6'-trihydrospiro[cyclopentane-1,8'-[7*H*]-furo[3,4-e]isoindole] (**9ba**).[7] (wwt-3-51)



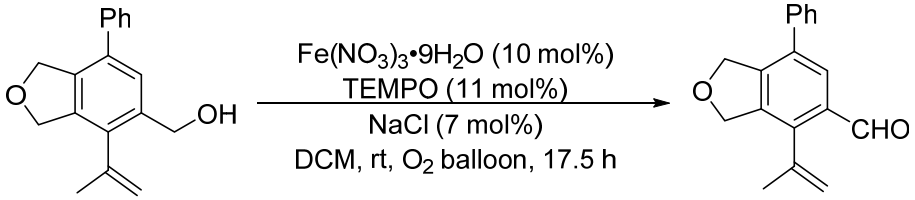
5ba **9ba** 55%

To a dry Schlenk tube were added **5ba** (89.1 mg, 0.2 mmol), CH_3CN (2.0 mL), water (0.14 mL), and NIS (67.8 mg, 0.3 mmol) sequentially. The reaction was complete after being stirred at room temperature for 3 hours as monitored by TLC. It was then quenched with an aqueous solution of saturated $Na_2S_2O_3$ (5 mL). The resulting mixture was extracted with ethyl ether (10 mL × 3), and the combined organic phase was dried over anhydrous Na_2SO_4. After filtration and evaporation of the solvent, the crude residue was purified by chromatography on silica gel to afford **9ba** (62.3 mg, 55%) (eluent: petroleum ether (60-90 ^oC)/ethyl acetate = 15/1).

9ba: solid; m.p. 135.0-137.2 ^oC (DCM/hexane); ^1H NMR (300 MHz, $CDCl_3$) δ 7.82 (d, *J* = 8.1 Hz, 2 H, ArH), 7.50-7.28 (m, 7 H, ArH), 7.12 (s, 1 H, ArH), 5.33 (dd, J_1 = 12.9 Hz, J_2 = 7.2 Hz, 1 H, CH), 5.27-5.14 (m, 3 H, OCH_2 and one proton of OCH_2), 5.07 (d, *J* = 12.3 Hz, 1 H, one proton of CH_2), 4.64 (d, *J*= 12.9 Hz, 1 H, one proton of NCH_2), 4.59 (d, *J* = 12.9 Hz, 1 H, one proton of NCH_2), 2.95-2.75 (m, 1 H,

one proton of CH_2), 2.60-2.45 (m, 1 H, one proton of CH_2), 2.43 (s, 3 H, CH_3), 2.41-2.24 (m, 2 H, CH_2), 2.24-2.05 (m, 1 H, one proton of CH_2), 1.98-1.80 (m, 1 H, one proton of CH_2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.7, 140.3, 139.4, 138.3, 137.3, 136.1, 134.9, 133.9, 129.8, 128.7, 127.8, 127.7, 127.2, 121.5, 83.4, 73.0, 72.5, 53.3, 39.1, 36.8, 35.3, 26.3, 21.5; IR (KBr) ν (cm^{-1}) 2952, 2926, 2852, 1654, 1317, 1598, 1560, 1466, 1450, 1345, 1161, 1130, 1096, 1065; MS (EI): *m/z* (%) 289 ($(M\text{-}Ts\text{-}I)^+$, 1.79), 258 (100); Anal. Calcd. for $C_{27}H_{26}INO_3S$ (%): C 56.75, H 4.59, N 2.45, Found: C 56.99, H 4.89, N 2.12.

3. Synthesis of 7-phenyl-4-(propen-2-yl)-1,3-dihydroisobenzofuran-5-carbaldehyde (**10ab**).[8] (zyc-2-34)



5ab **10ab** 49%

To a dry Schlenk tube were added $Fe(NO_3)_3$•$9H_2O$ (16.8 mg, 0.04 mmol), TEMPO (6.9 mg, 0.044 mmol), NaCl (1.6 mg, 0.028 mmol), DCM (2.0 mL), and **5ab** (106.3 mg, 0.4 mmol)/DCM (0.5 mL) sequentially. After being equipped with an O_2 balloon, the reaction was stirred at room temperature for 17.5 hours as monitored by TLC. The resulting reaction mixture was then filtrated through a short column of silica gel eluted with ethyl acetate (10 mL × 3). After evaporation, the residue was purified by chromatography on silica gel to afford **10ab** (51.2 mg, 49%) (eluent: petroleum ether (60-90 ^oC)/ethyl acetate= 20/1).

10ab: solid; m.p. 83.0-83.7 ^{o}C (*n*-hexane/DCM); ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.22 (s, 1 H, CHO), 7.93 (s, 1 H, ArH), 7.52-7.36 (m, 5 H, ArH), 5.52-5.44 (m, 1 H, one proton of $=CH_2$), 5.29-5.23 (m, 2 H, OCH_2), 5.19-5.13 (m, 2 H, OCH_2), 5.07-4.98 (m, 1 H, one proton of $=CH_2$), 2.13 (t, J = 1.2 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 191.3, 143.3, 140.1, 140.0, 139.0, 138.5, 135.2, 133.2, 128.8, 127.9, 127.8, 127.7, 118.3, 73.8, 72.6, 24.4; IR (KBr) ν (cm^{-1}) 3079, 2965, 2915, 2869, 2837, 2754, 1685, 1630, 1596, 1560, 1502, 1468, 1450, 1393, 1384, 1332, 1313, 1294, 1211, 1178, 1135, 1083, 1057, 1030, 1009; MS (EI): *m/z* (%) 264 (M^+, 100); Anal. Calcd. for $C_{18}H_{16}O_2$ (%): C 81.79, H 6.10, Found: C 81.82, H 6.10.

References

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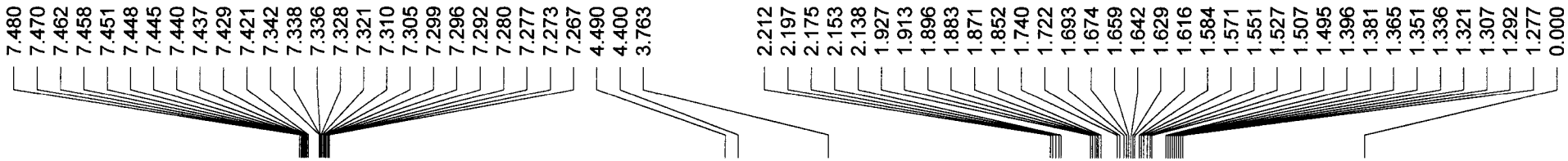

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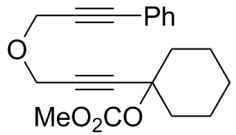

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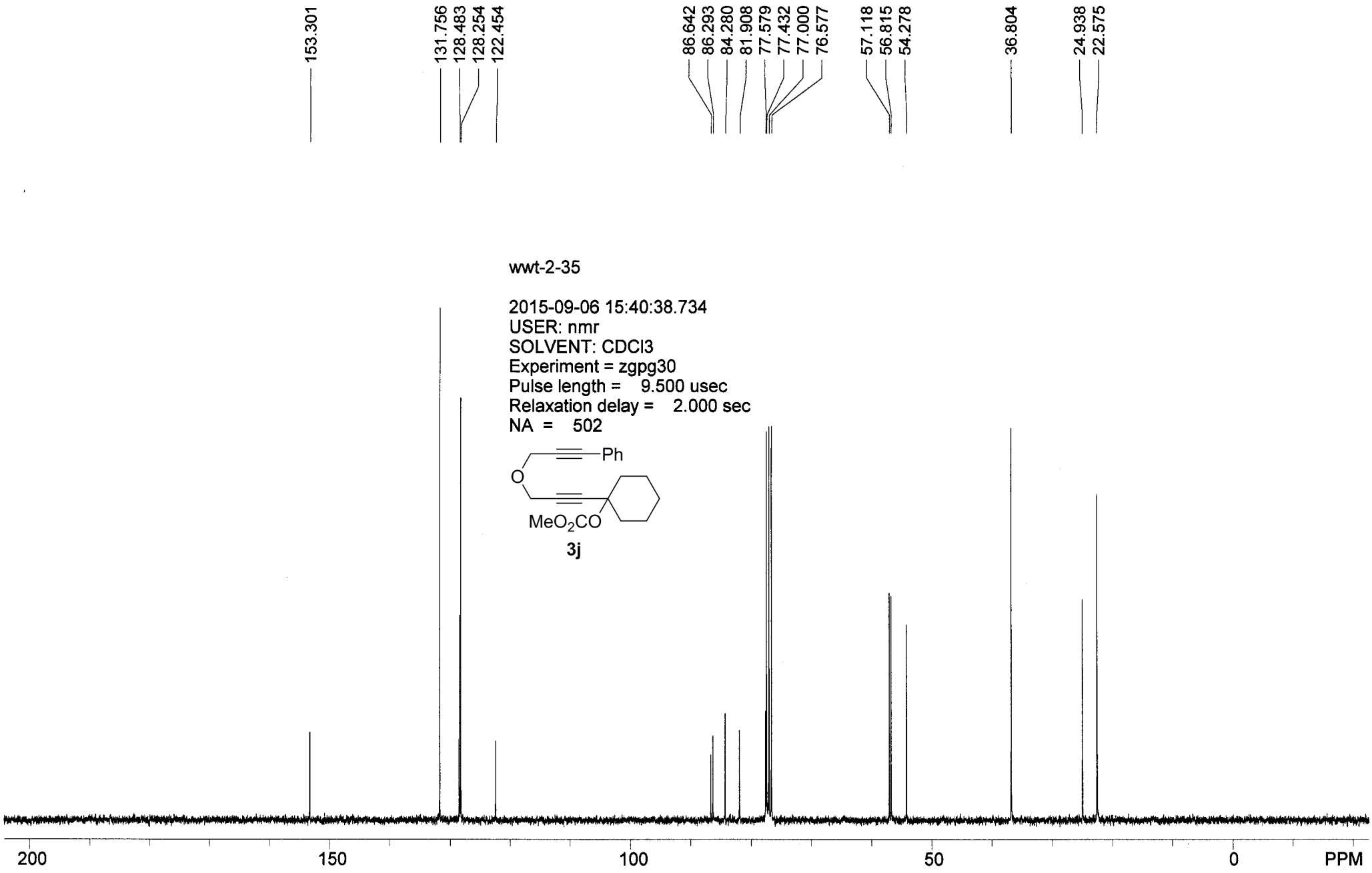
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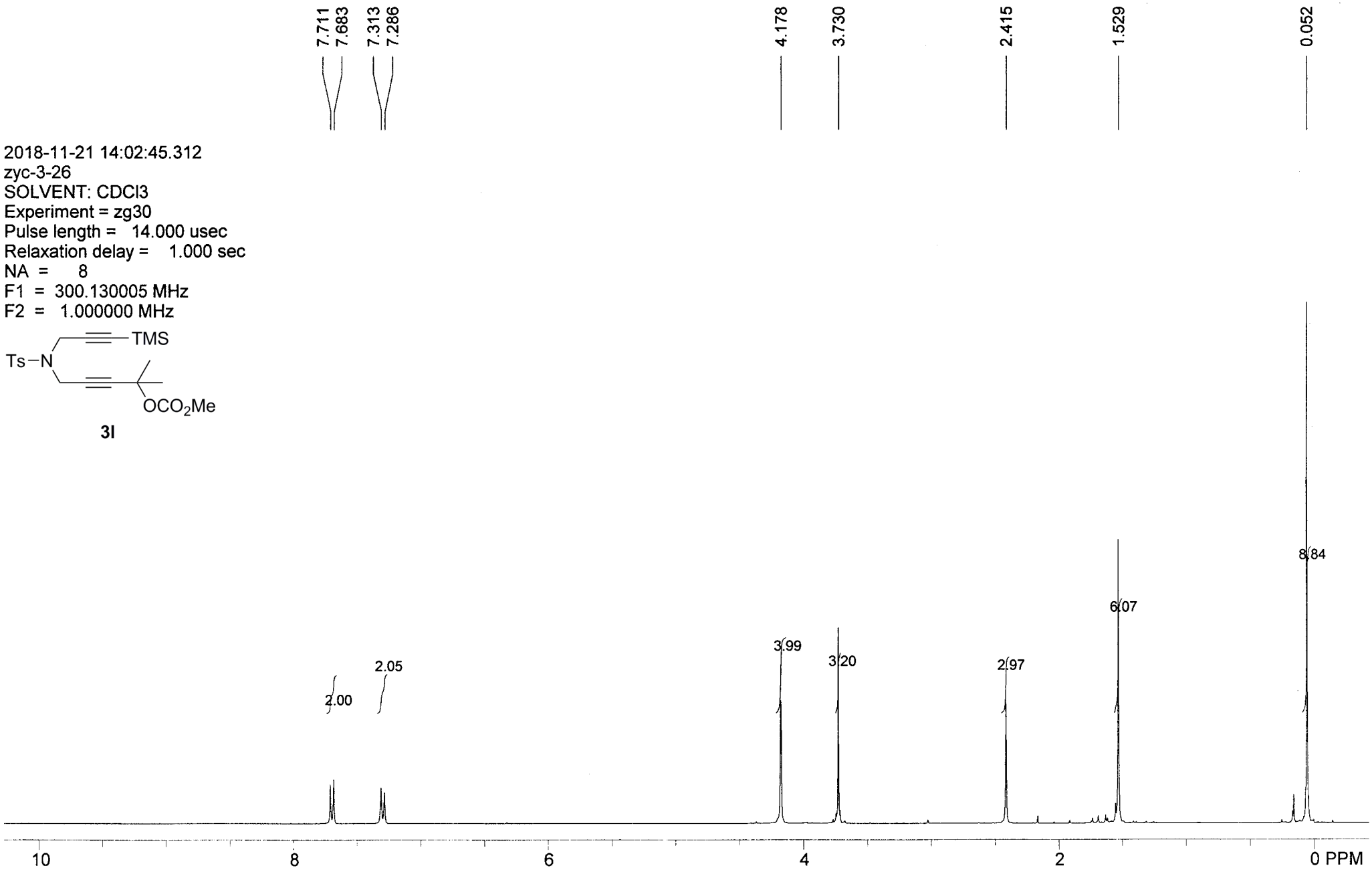
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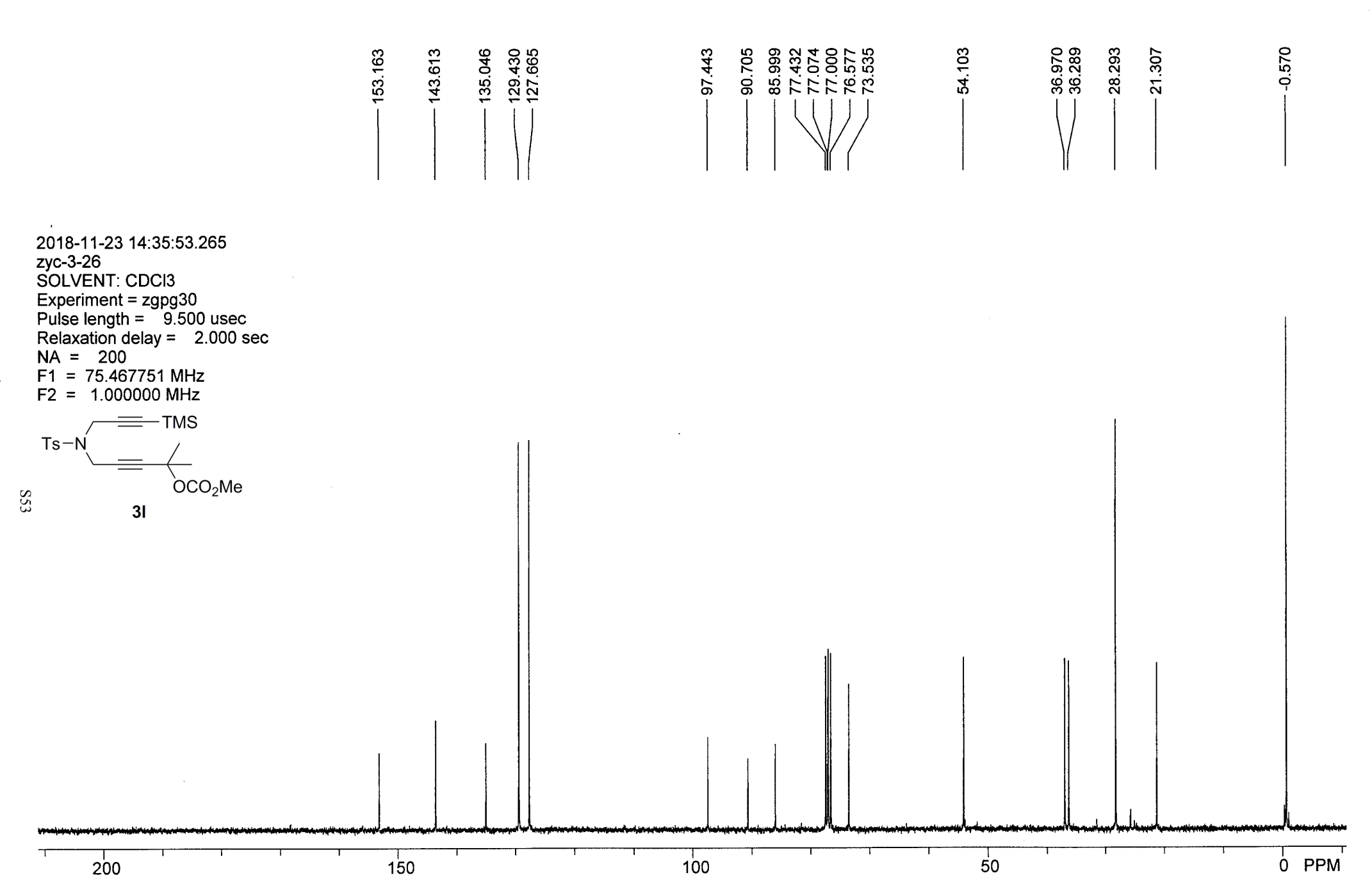
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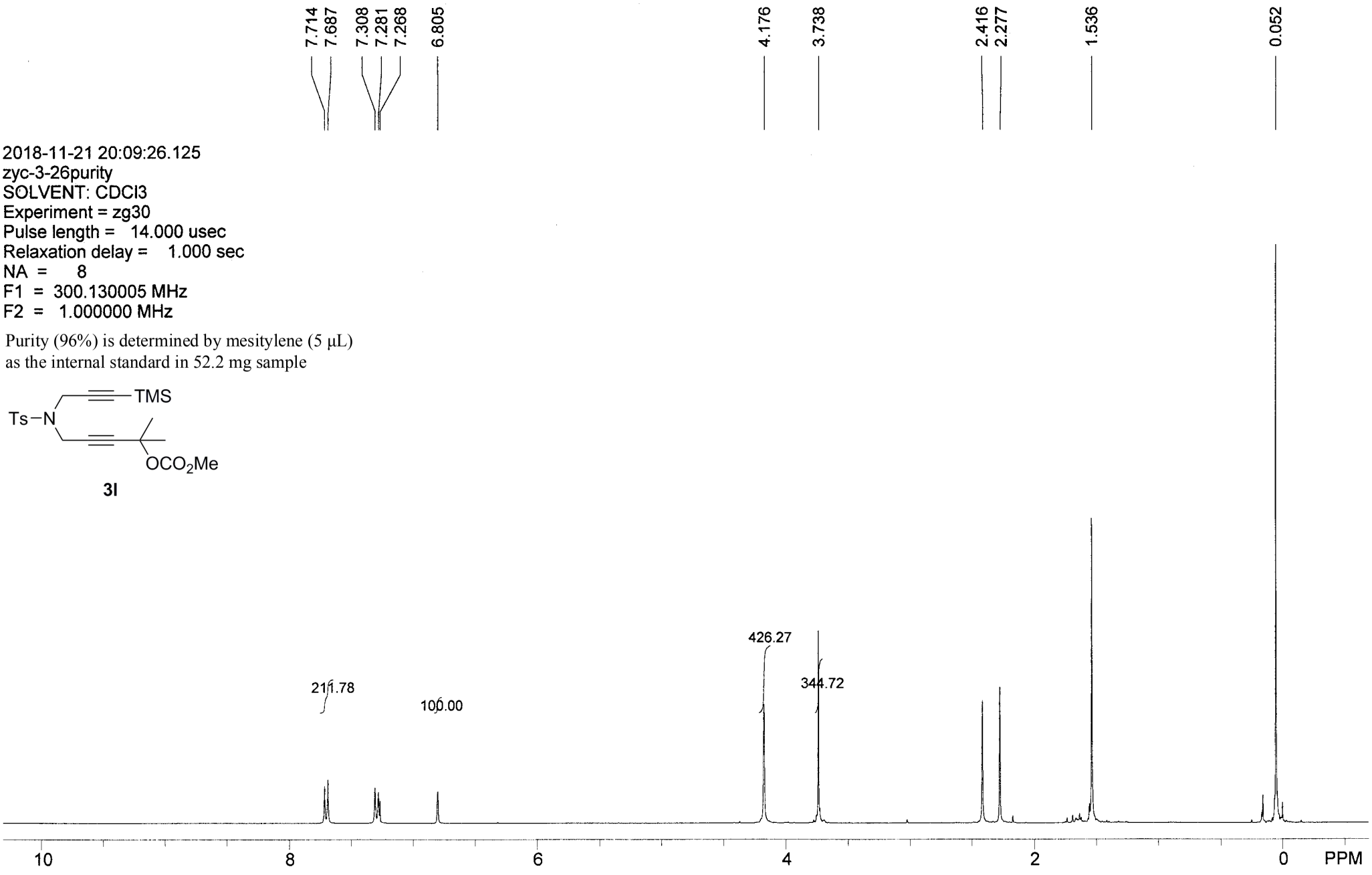
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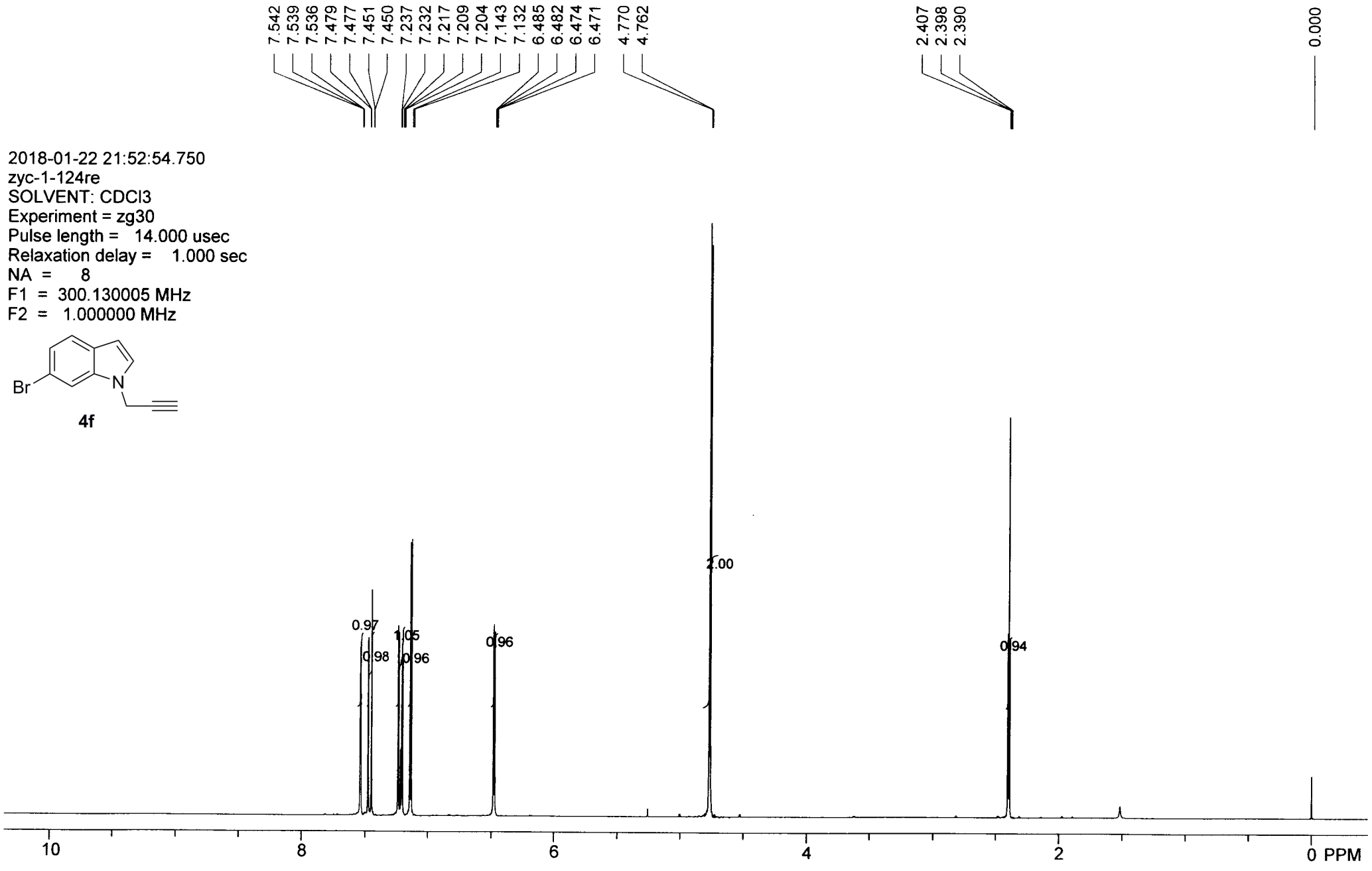
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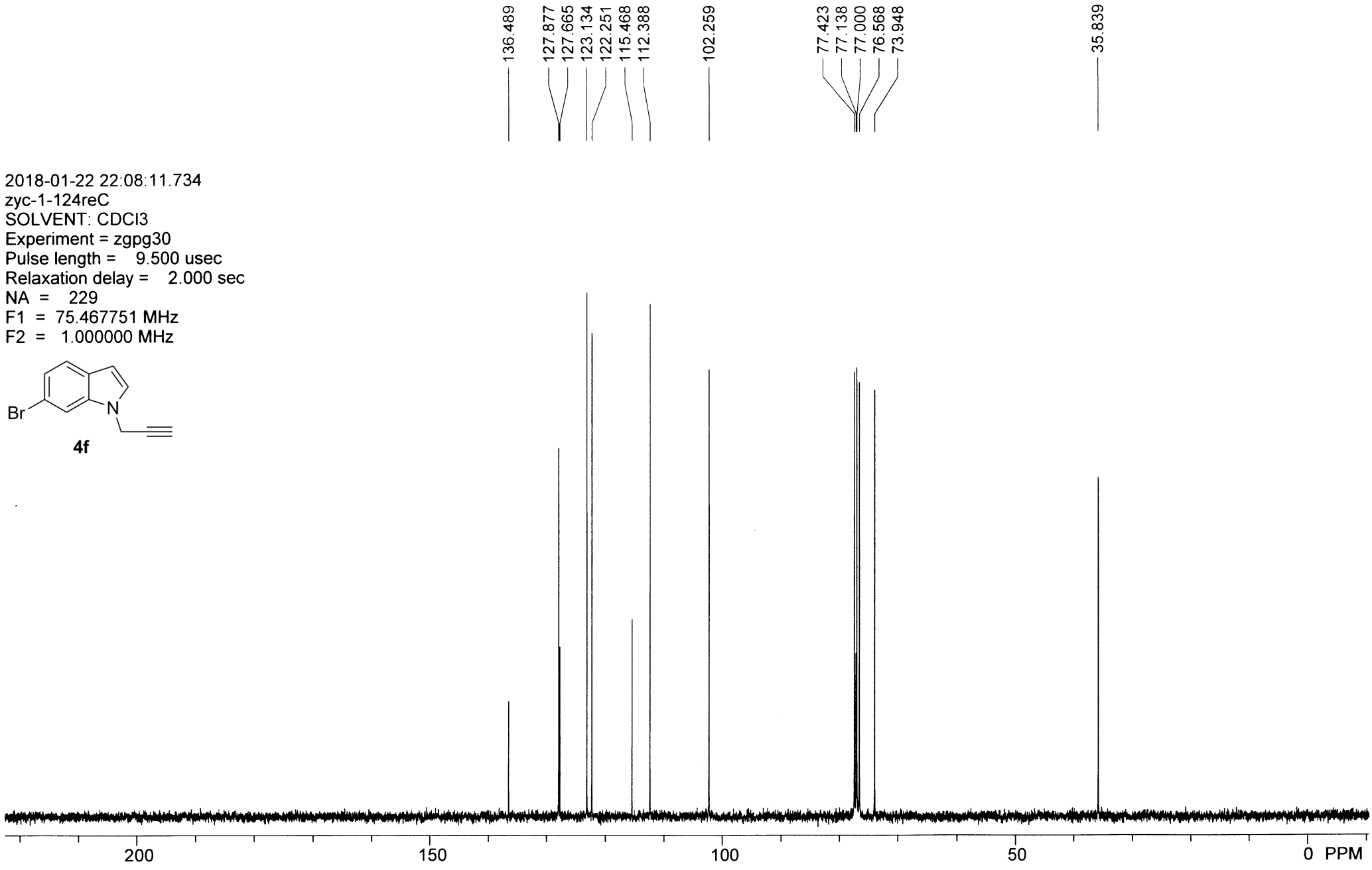
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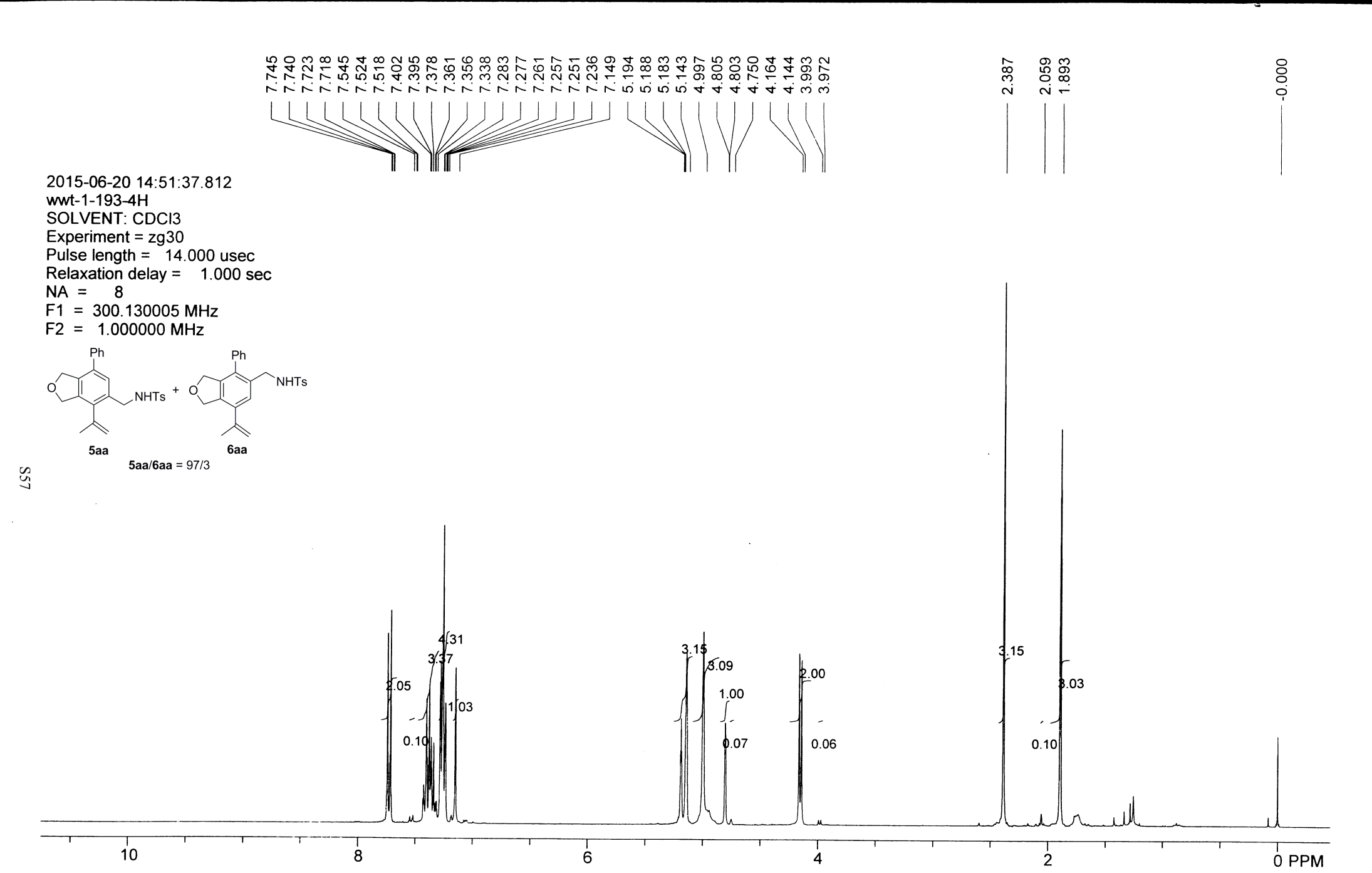
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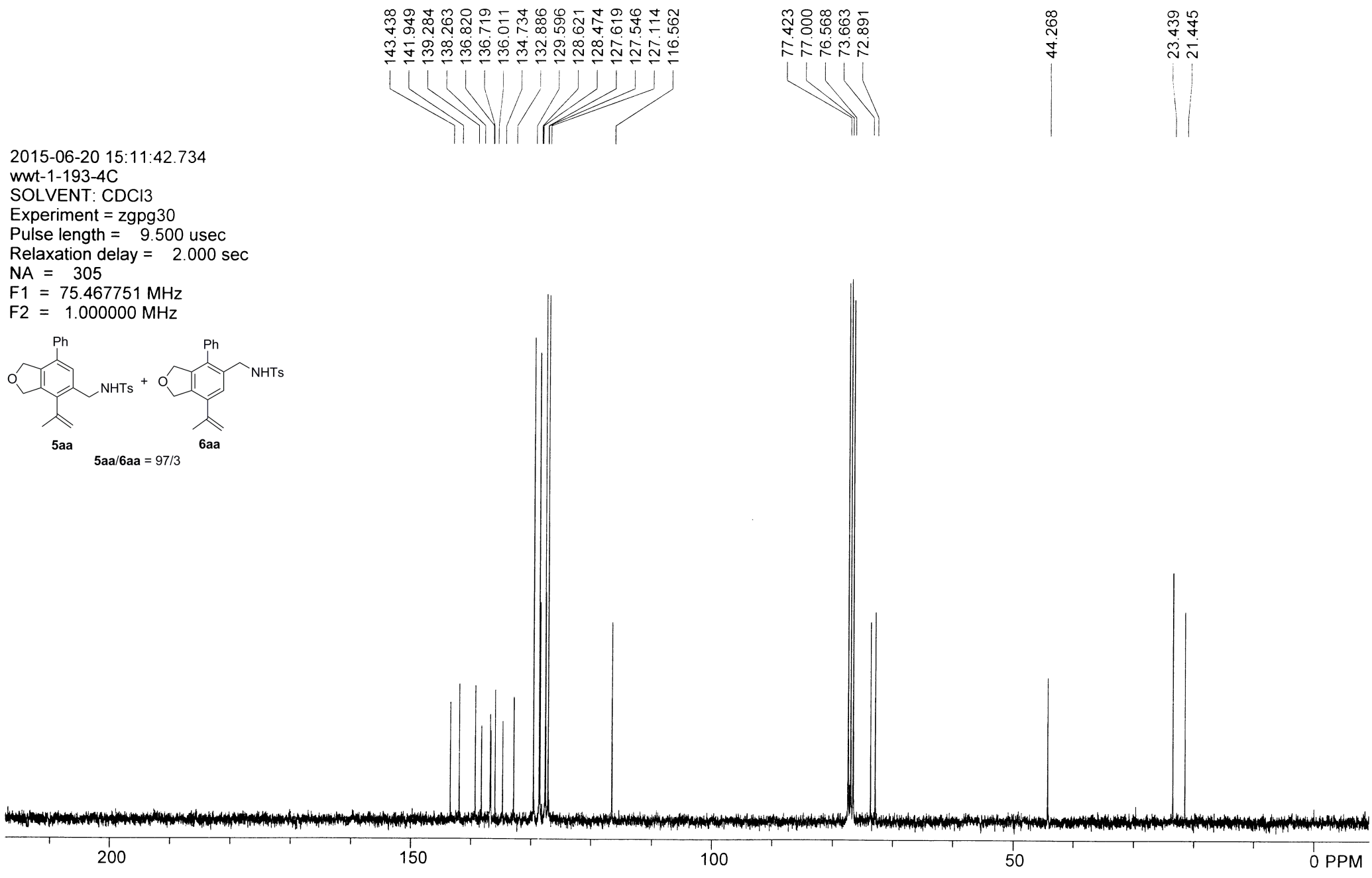
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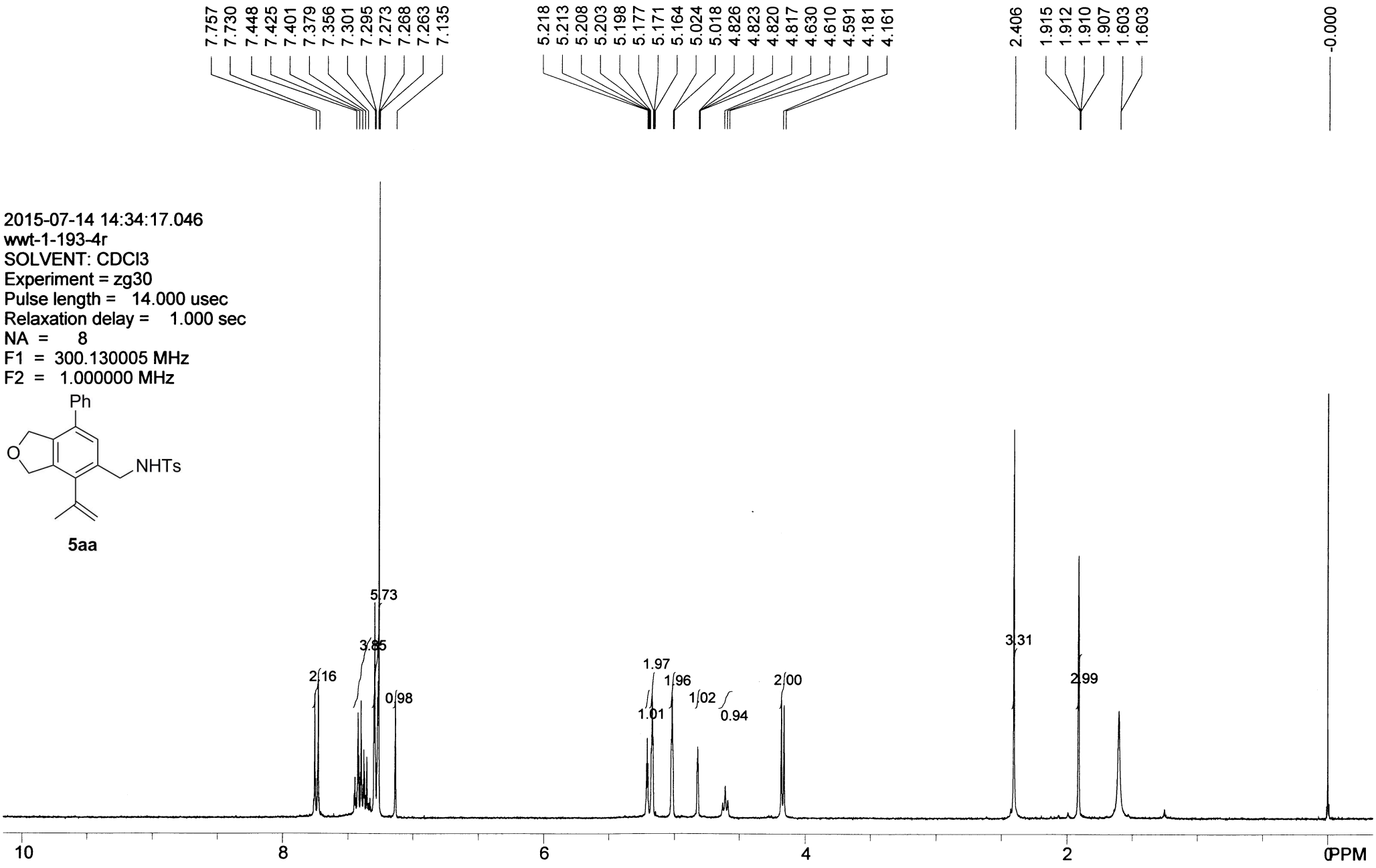
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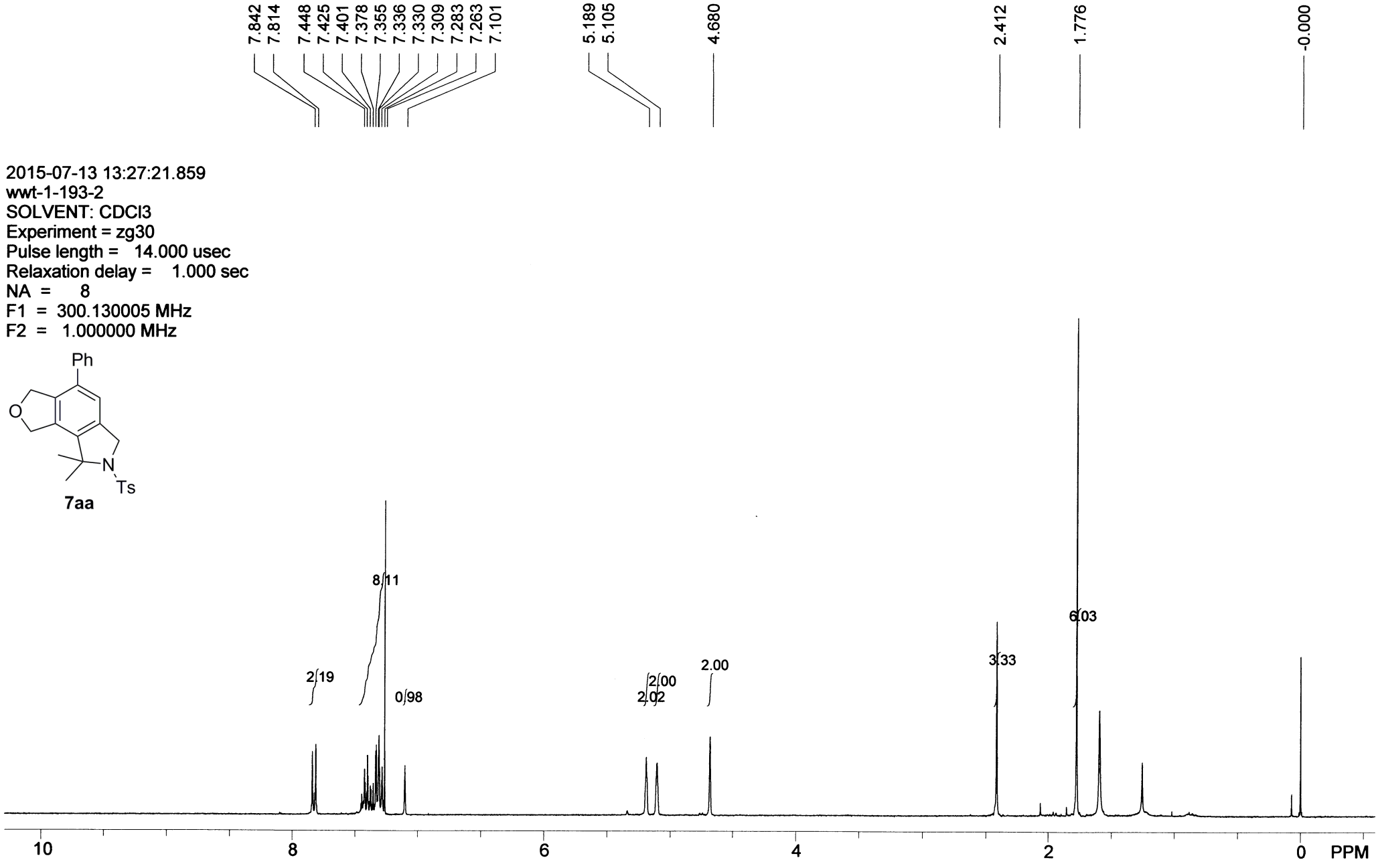
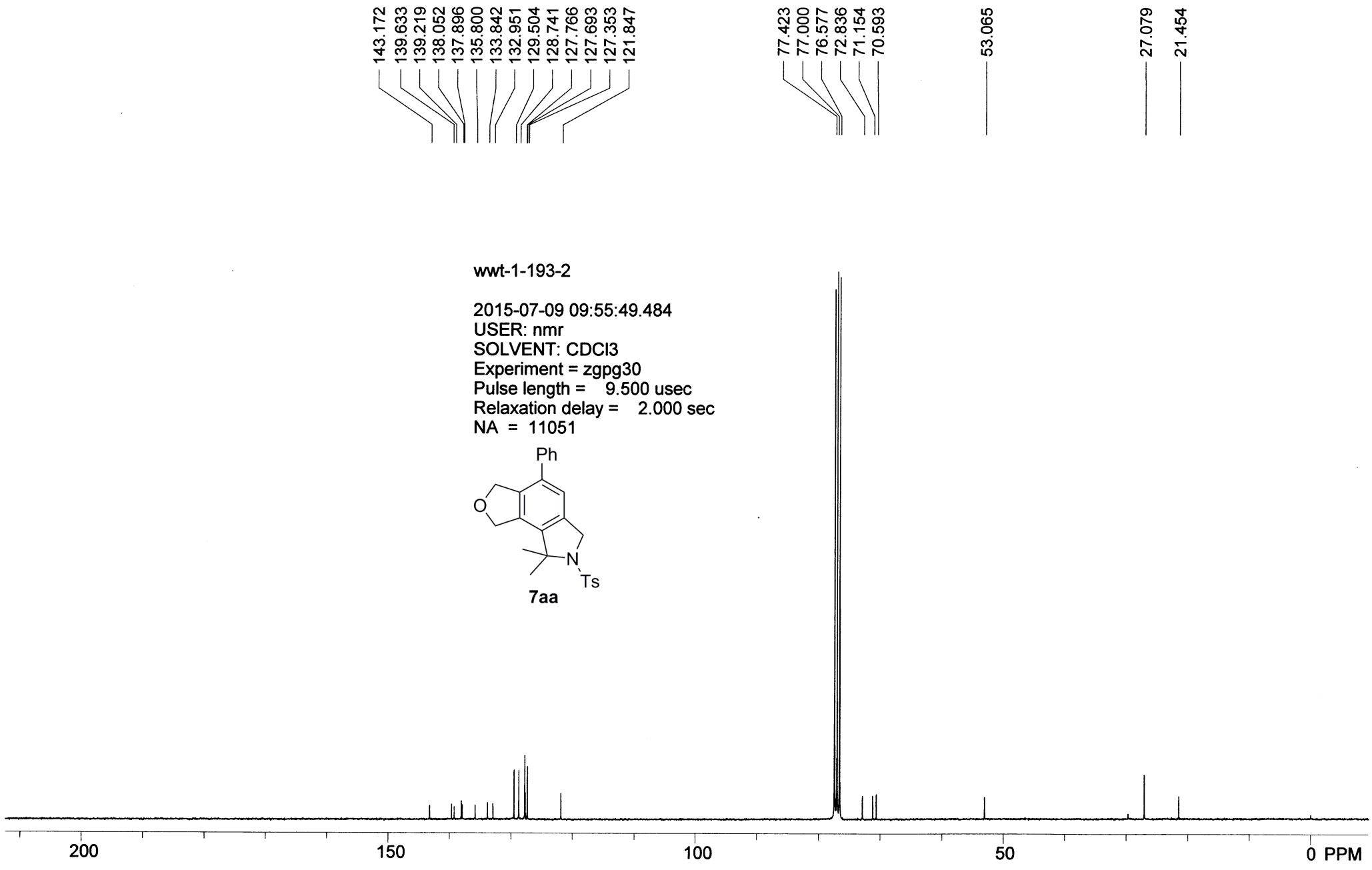
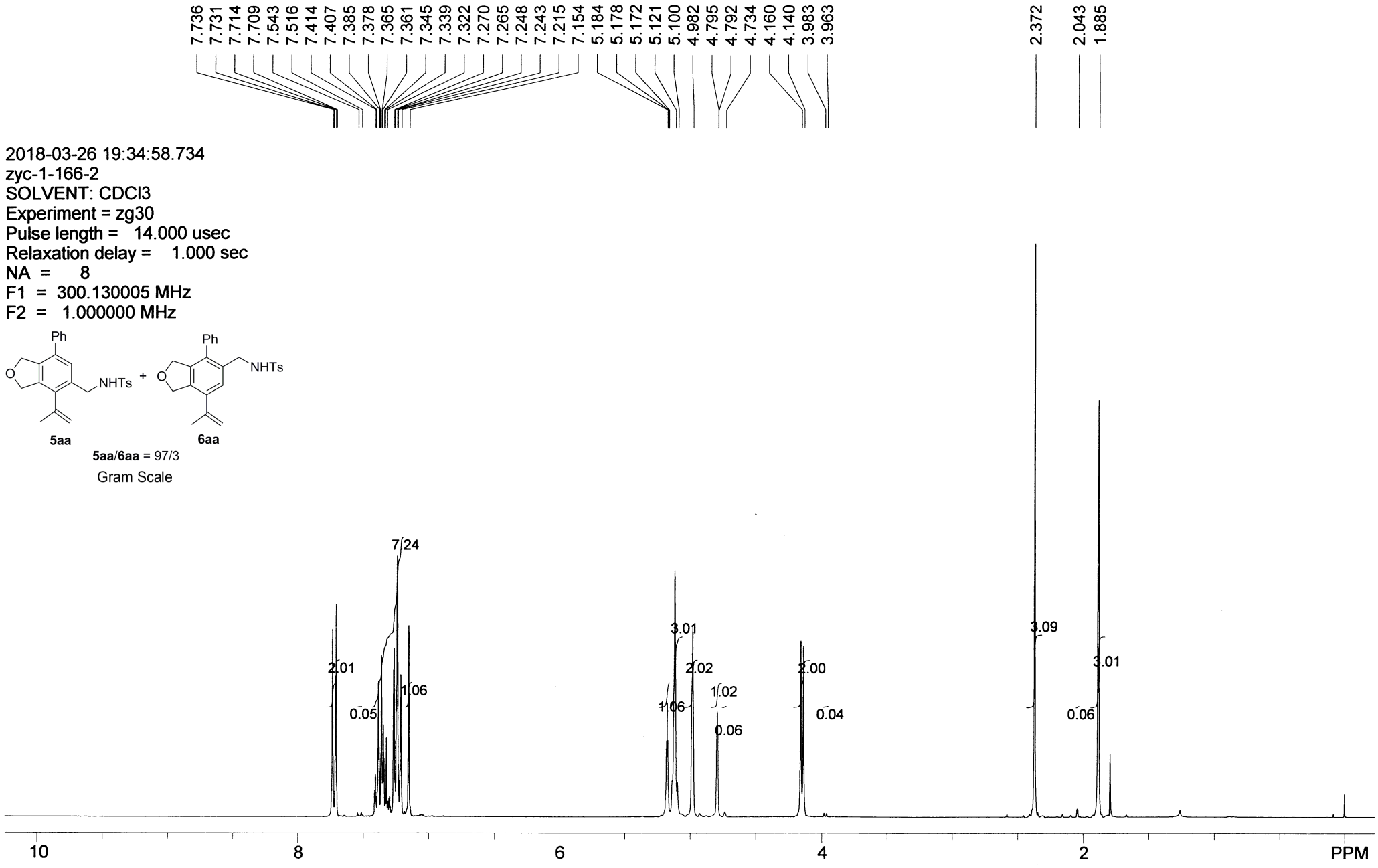
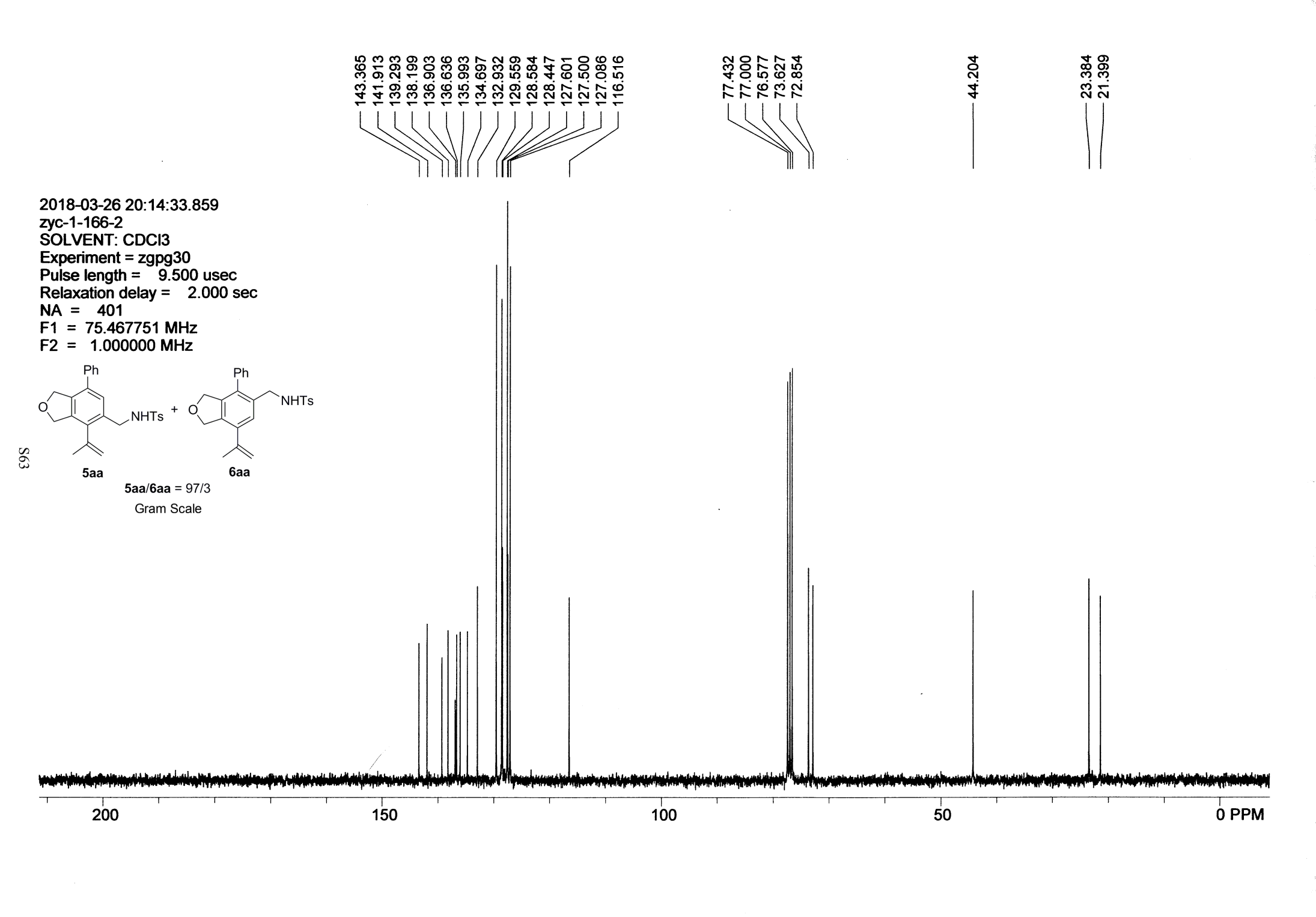
7.448
7.425
7.401
7.379
7.356
7.301
7.295
7.273
7.268
7.263
7.135
5.218
5.213
5.208
5.203
5.198
5.177
5.171
5.164
5.024
5.018
4.826
4.823
4.820
4.817
4.630
4.610
4.591
4.181
4.161
2.406
1.915
1.912
1.910
1.907
1.603
1.603
-0.000
2015-07-14 14:34:17.046
wwt-1-193-4r
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
Ph
O
NHTs
5aa
5.73
3.85
2.16
0.98
1.97
1.96
1.02
1.01
0.94
2.00
3.31
2.99
10
8
6
4
2
0
PPM

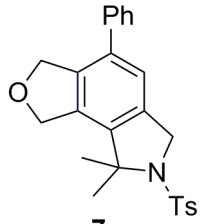

7.842
7.814
7.448
7.425
7.401
7.378
7.355
7.336
7.330
7.309
7.283
7.263
7.101
5.189
5.105
4.680
2.412
1.776
-0.000
2015-07-13 13:27:21.859
wwt-1-193-2
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
Ph
O
N
Ts
7aa
2.19
8.11
0.98
2.00
2.02
2.00
3.33
6.03
10
8
6
4
2
0
PPM


143.172
139.633
139.219
138.052
137.896
135.800
133.842
132.951
129.504
128.741
127.766
127.693
127.353
121.847
77.423
77.000
76.577
72.836
71.154
70.593
53.065
27.079
21.454
wwt-1-193-2
2015-07-09 09:55:49.484
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 11051
Ph
O
N
Ts
7aa
200
150
100
50
0 PPM

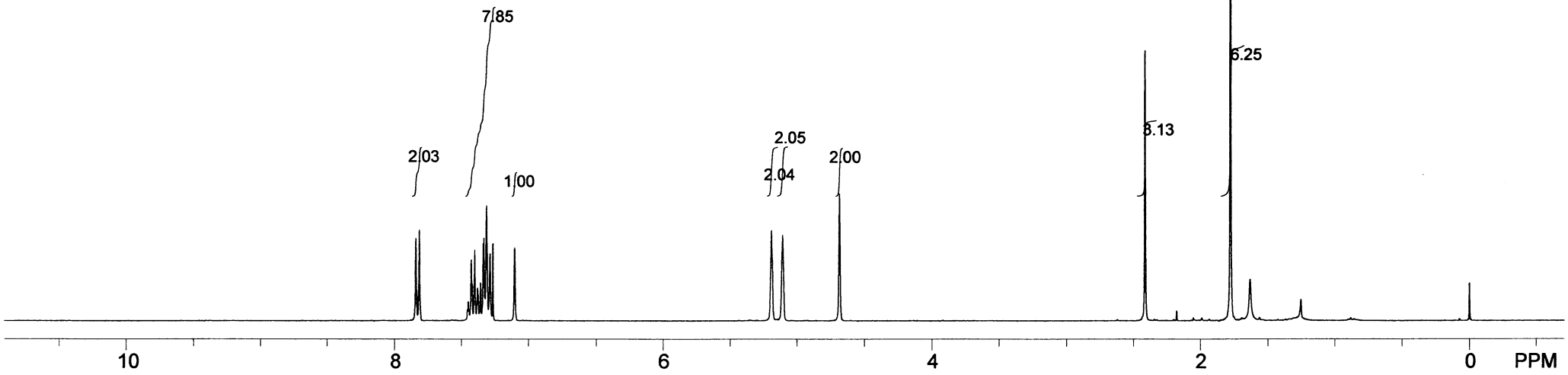

7.736
7.731
7.714
7.709
7.543
7.516
7.414
7.407
7.385
7.378
7.365
7.361
7.345
7.339
7.322
7.270
7.265
7.248
7.243
7.215
7.154
5.184
5.178
5.172
5.121
5.100
4.982
4.795
4.792
4.734
4.160
4.140
3.983
3.963
2.372
2.043
1.885
2018-03-26 19:34:58.734
zyc-1-166-2
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
Ph
NHTs
+
Ph
NHTs
5aa
6aa
5aa/6aa = 97/3
Gram Scale
7.24
2.01
0.05
1.06
3.01
1.06
2.02
1.02
0.06
2.00
0.04
3.09
0.06
3.01
10
8
6
4
2
PPM

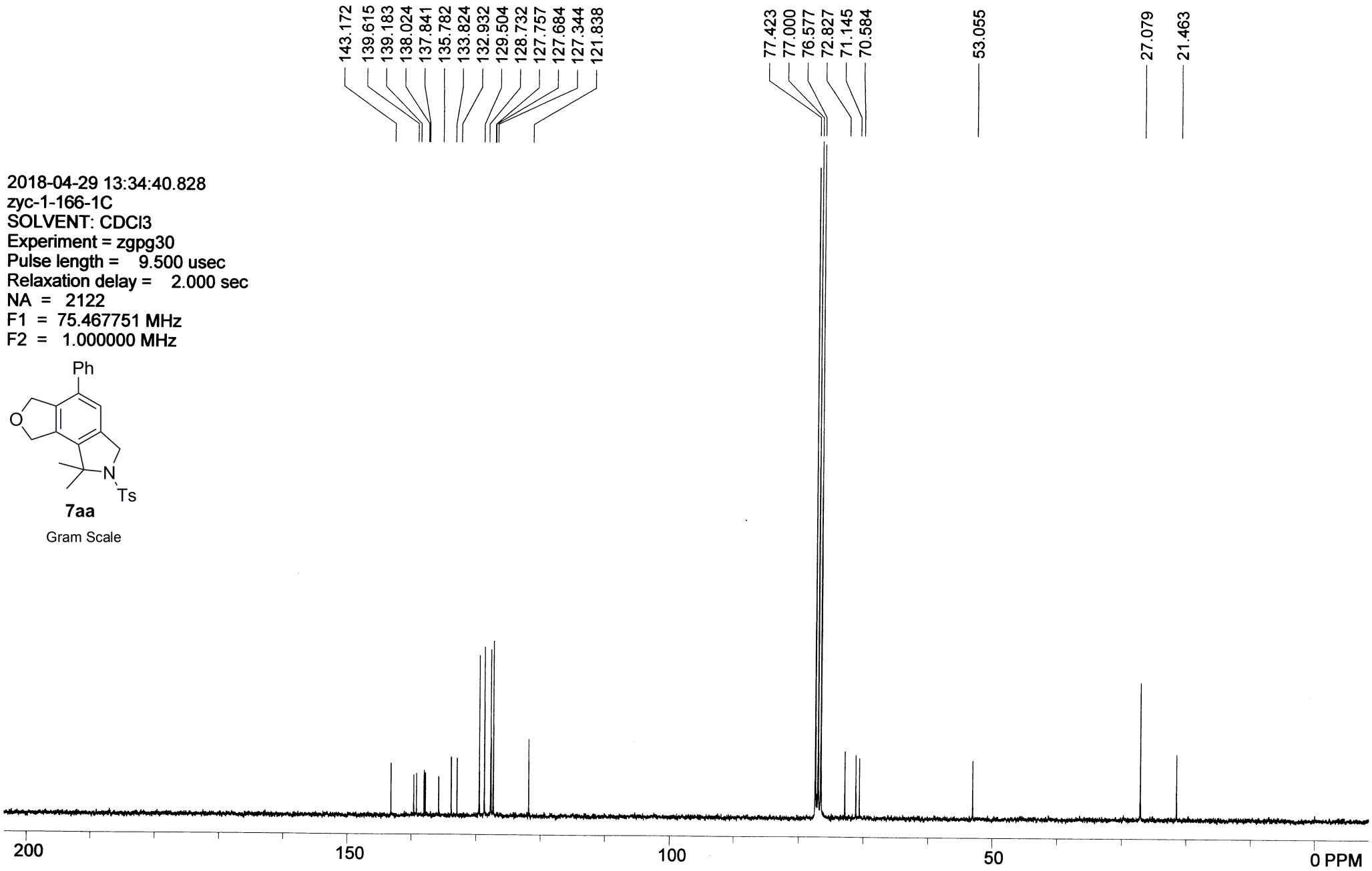

143.365
141.913
139.293
138.199
136.903
136.636
135.993
134.697
132.932
129.559
128.584
128.447
127.601
127.500
127.086
116.516
77.432
77.000
76.577
73.627
72.854
44.204
23.384
21.399
2018-03-26 20:14:33.859
zyc-1-166-2
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 401
F1 = 75.467751 MHz
F2 = 1.000000 MHz
Ph
NHTs
O
+
Ph
NHTs
O
5aa
6aa
5aa/6aa = 97/3
Gram Scale
200
150
100
50
0 PPM

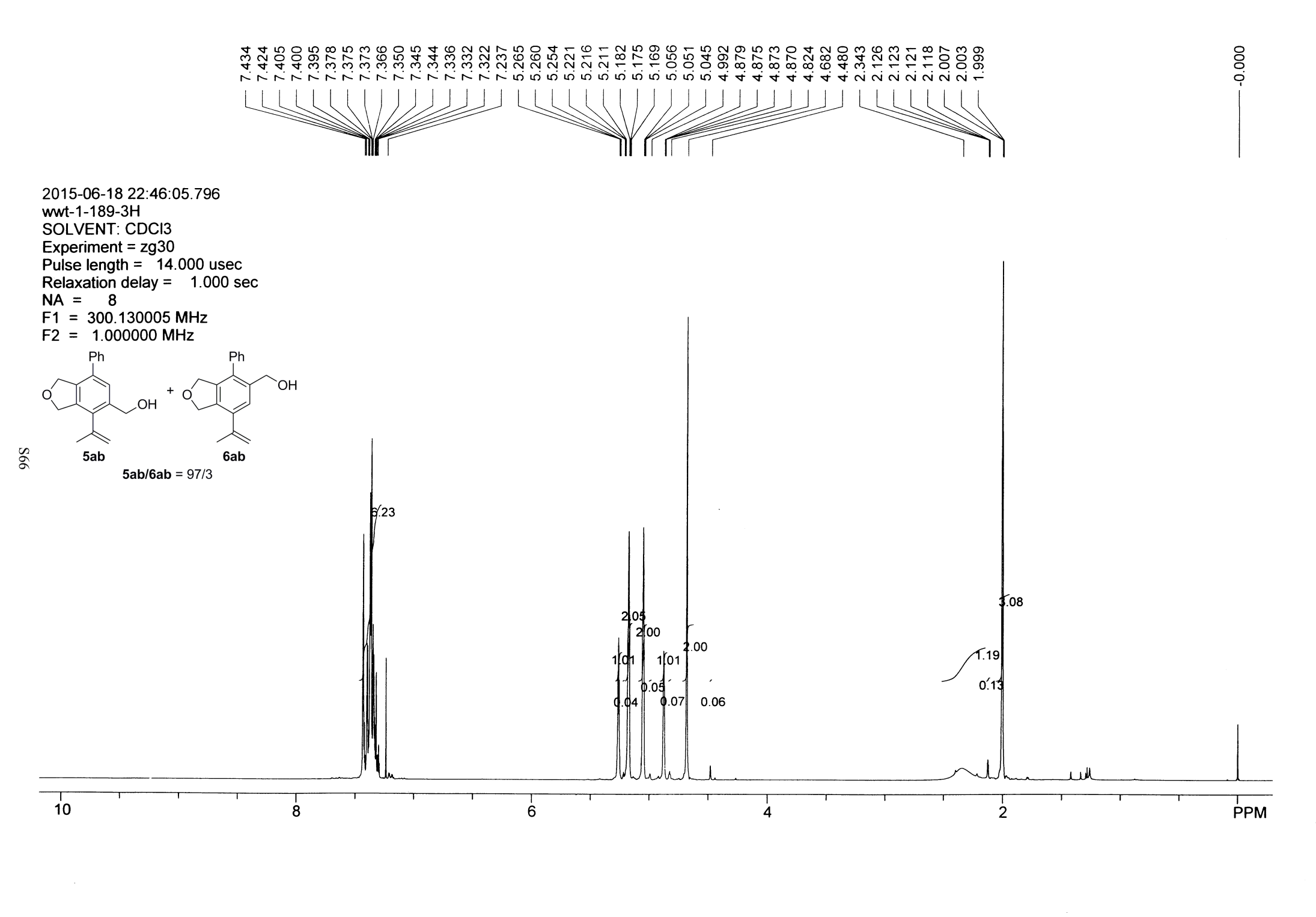
2018-03-29 21:26:19.250
zyc-1-166-1
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz

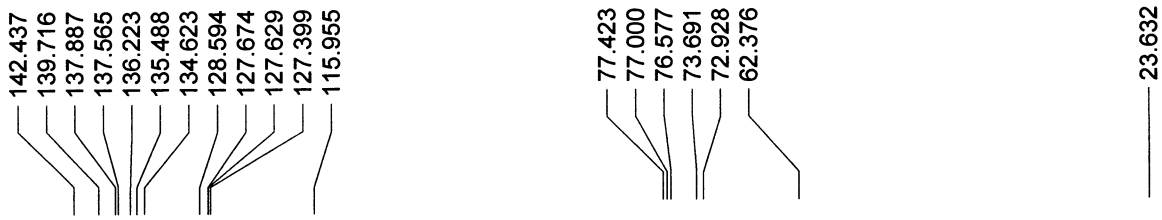


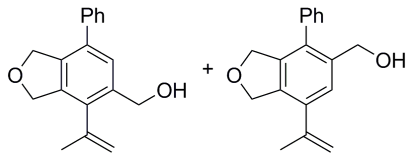
7aa
Gram Scale




143.172
139.615
139.183
138.024
137.841
135.782
133.824
132.932
129.504
128.732
127.757
127.684
127.344
121.838
77.423
77.000
76.577
72.827
71.145
70.584
53.055
27.079
21.463
2018-04-29 13:34:40.828
zyc-1-166-1C
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 2122
F1 = 75.467751 MHz
F2 = 1.000000 MHz
Ph
O
N
Ts
7aa
Gram Scale
200
150
100
50
0 PPM


7.434
7.424
7.405
7.400
7.395
7.378
7.375
7.373
7.366
7.350
7.345
7.344
7.336
7.332
7.322
7.237
5.265
5.260
5.254
5.221
5.216
5.211
5.182
5.175
5.169
5.056
5.051
5.045
4.992
4.879
4.875
4.873
4.870
4.824
4.682
4.480
2.343
2.126
2.123
2.121
2.118
2.007
2.003
1.999
-0.000
2015-06-18 22:46:05.796
wwt-1-189-3H
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
Ph
OH
O
+
5ab
6ab
5ab/6ab = 97/3
6.23
2.05
2.00
1.01
1.01
2.00
0.05
0.04
0.07
0.06
3.08
1.19
0.13
10
8
6
4
2
PPM



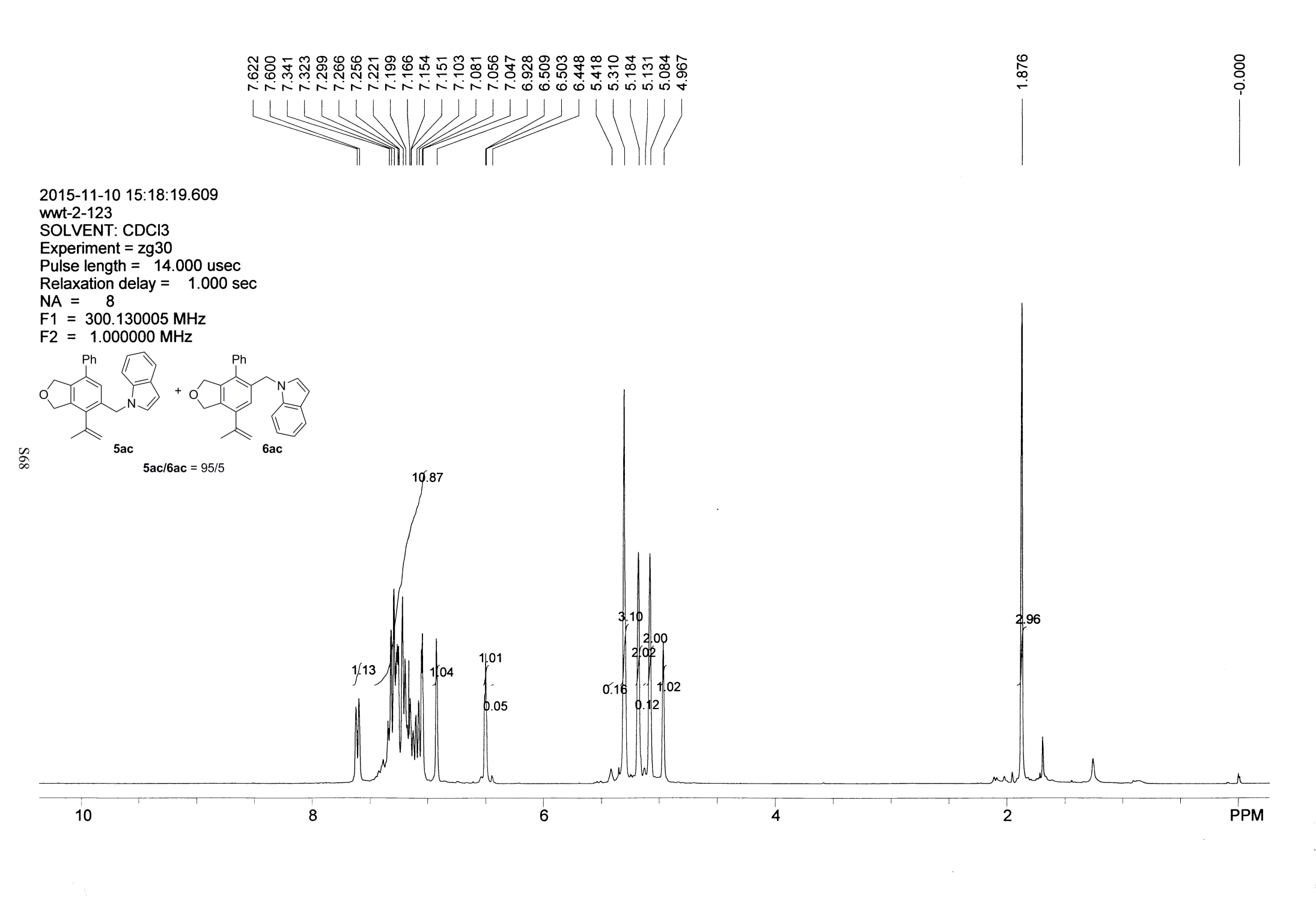


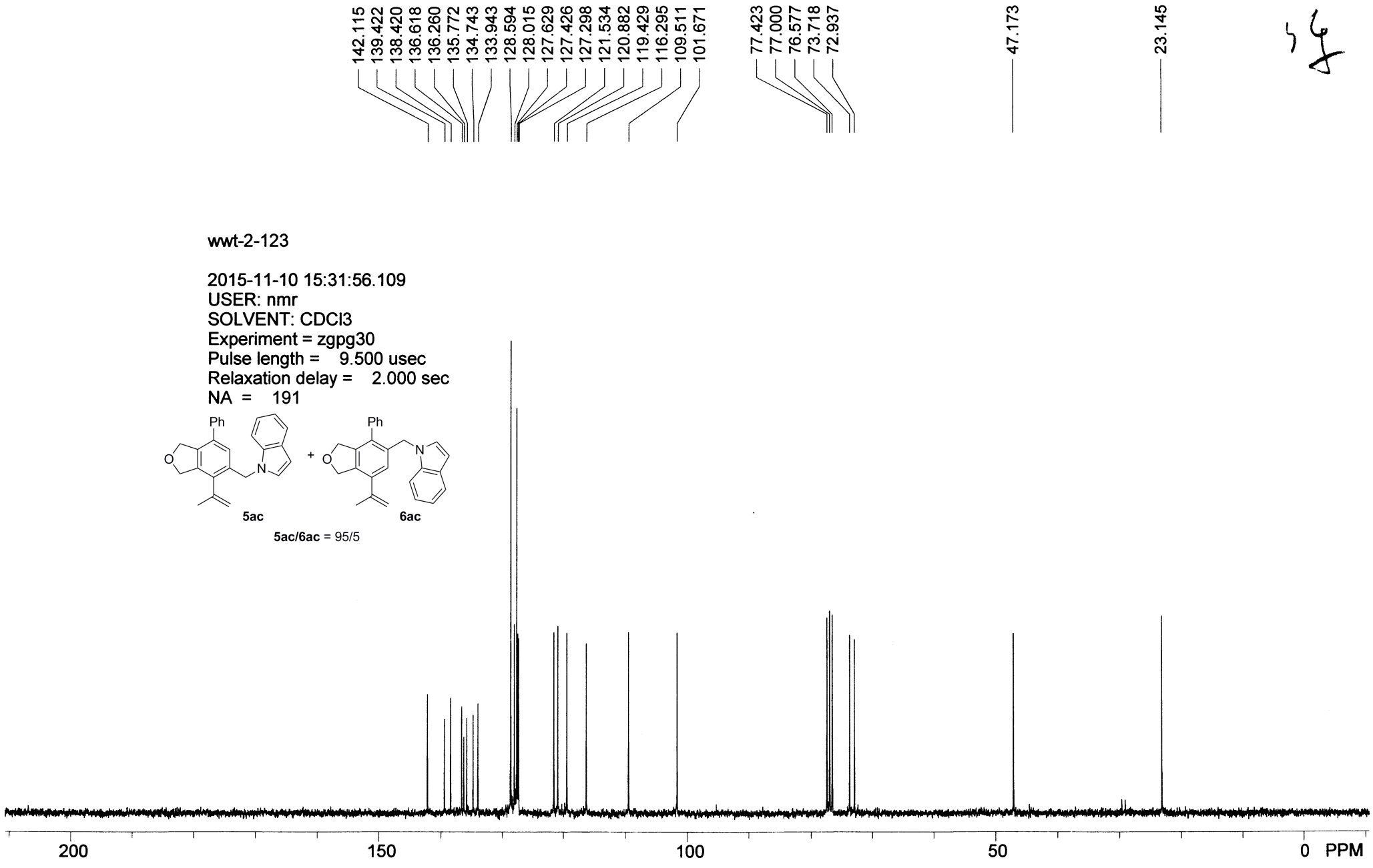
5ab **6ab**

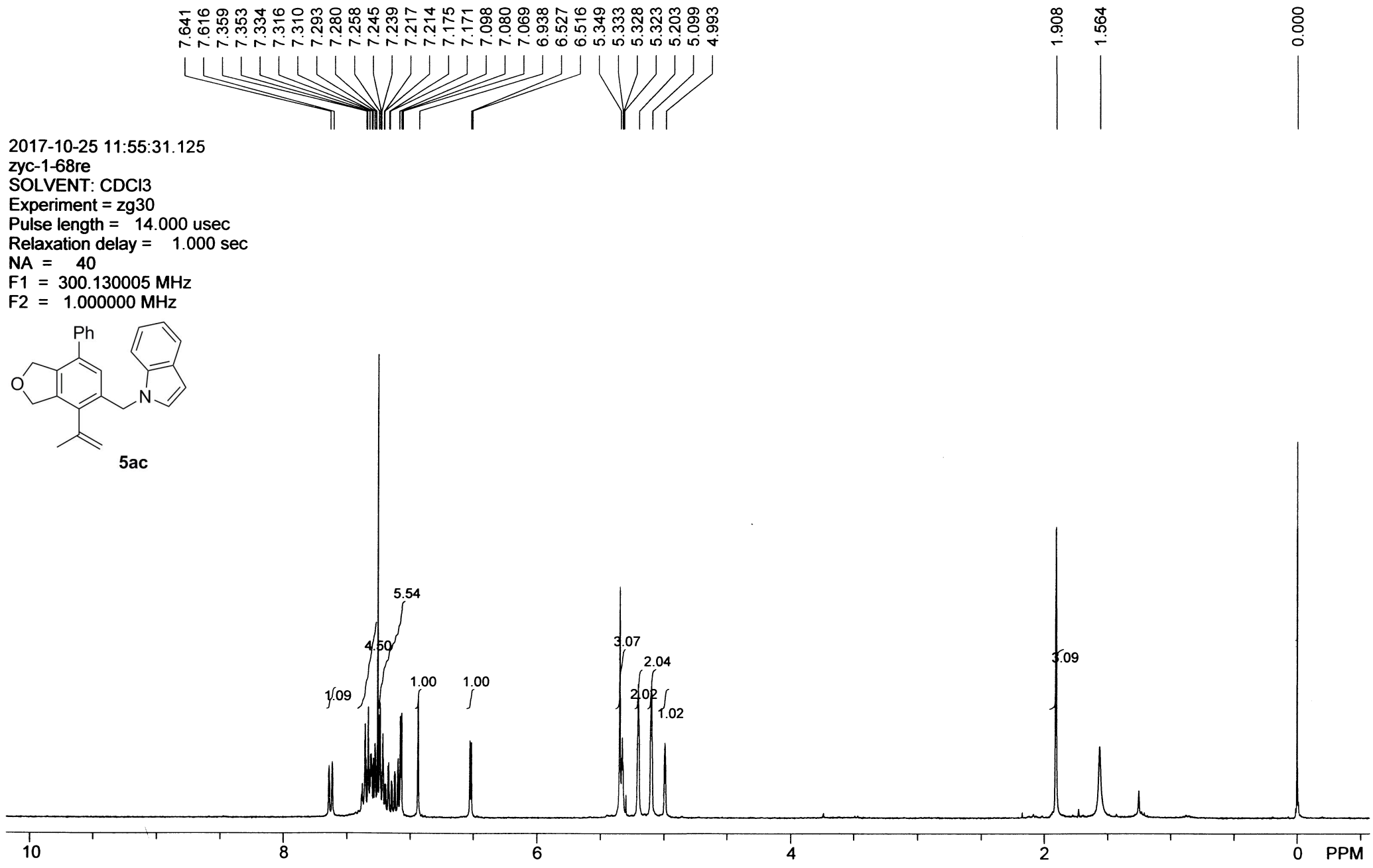
5ab/6ab = 97/3

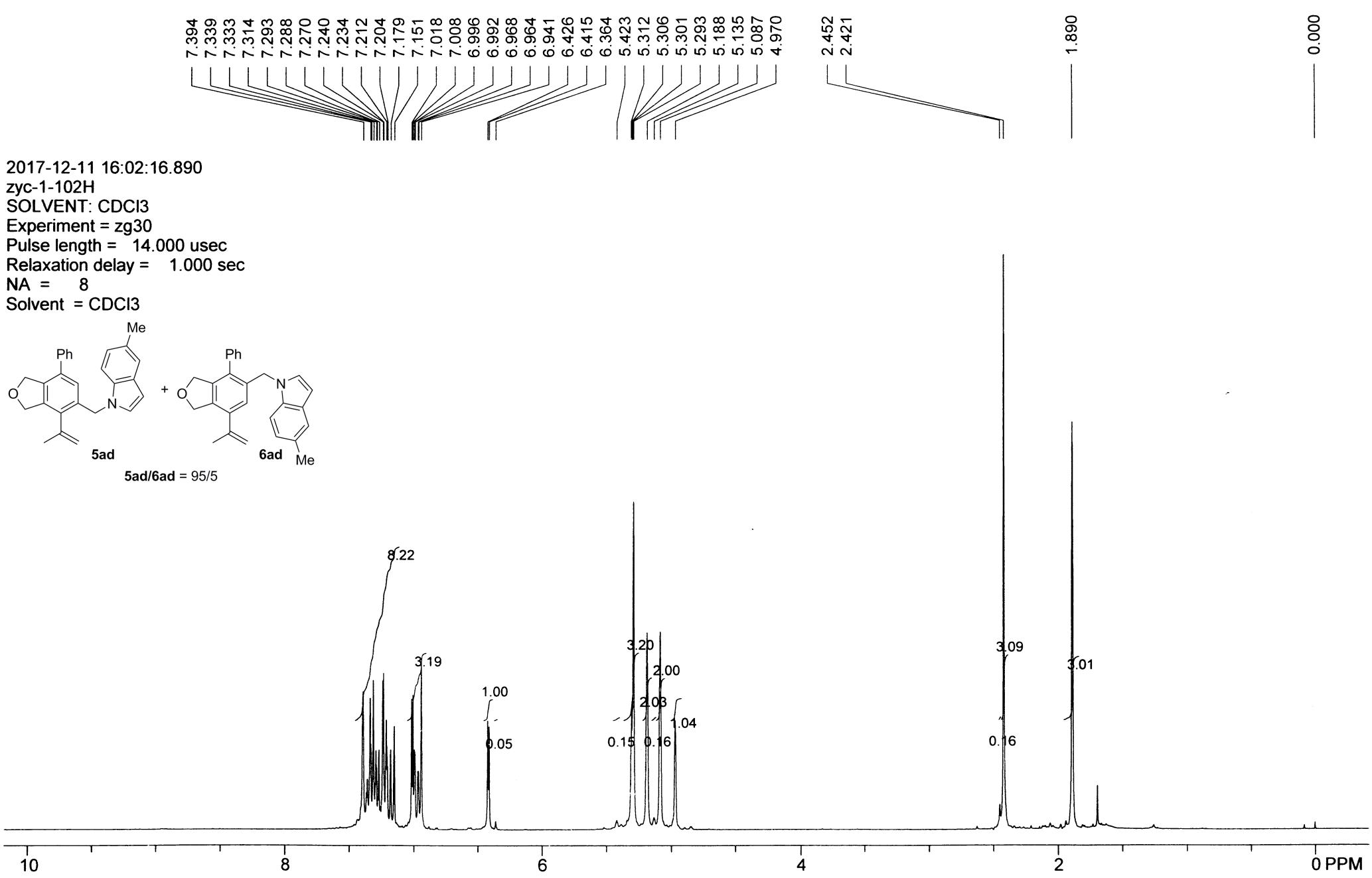
wwt-1-189-3

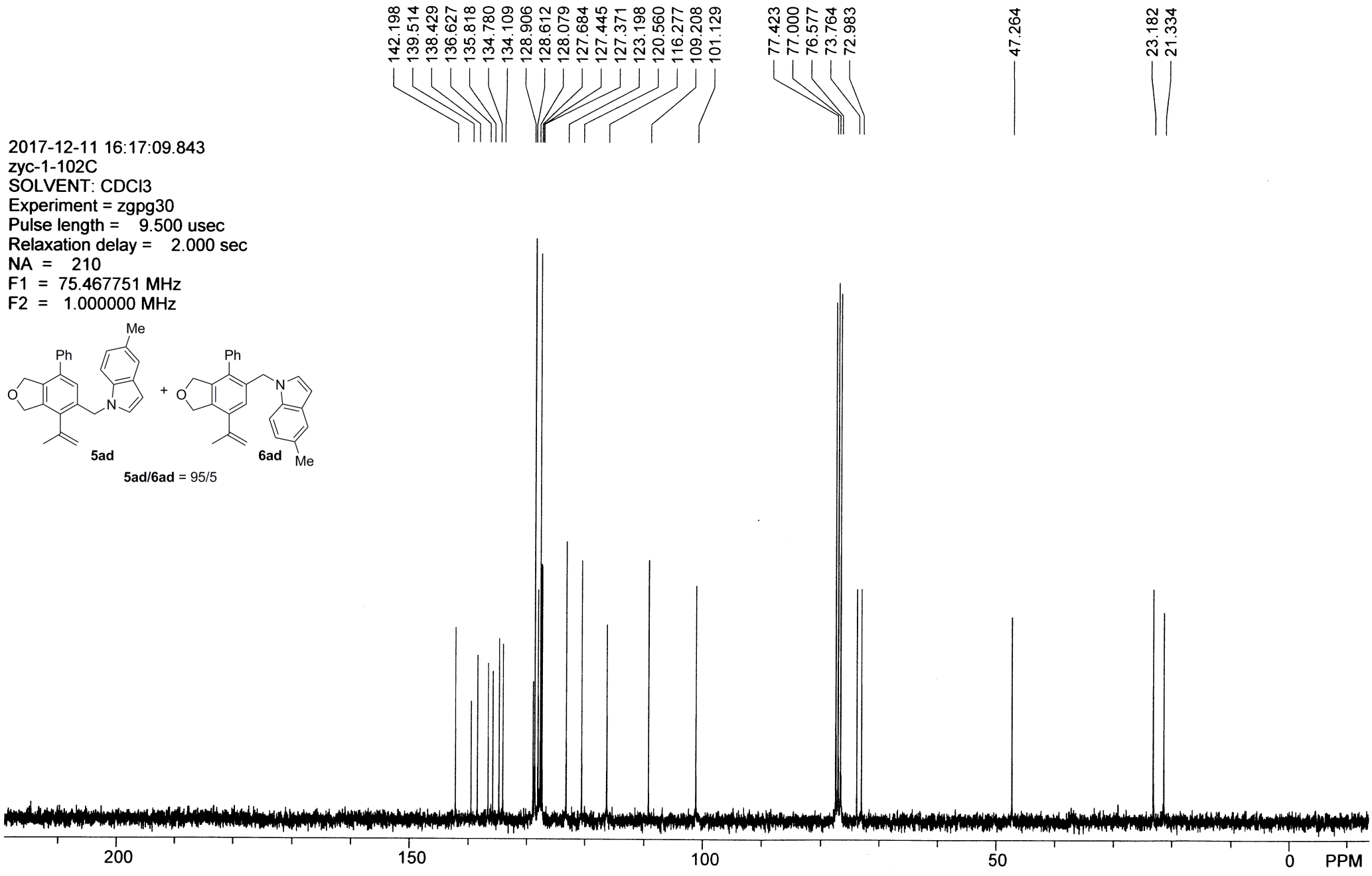
2015-06-19 13:33:30.031
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 126

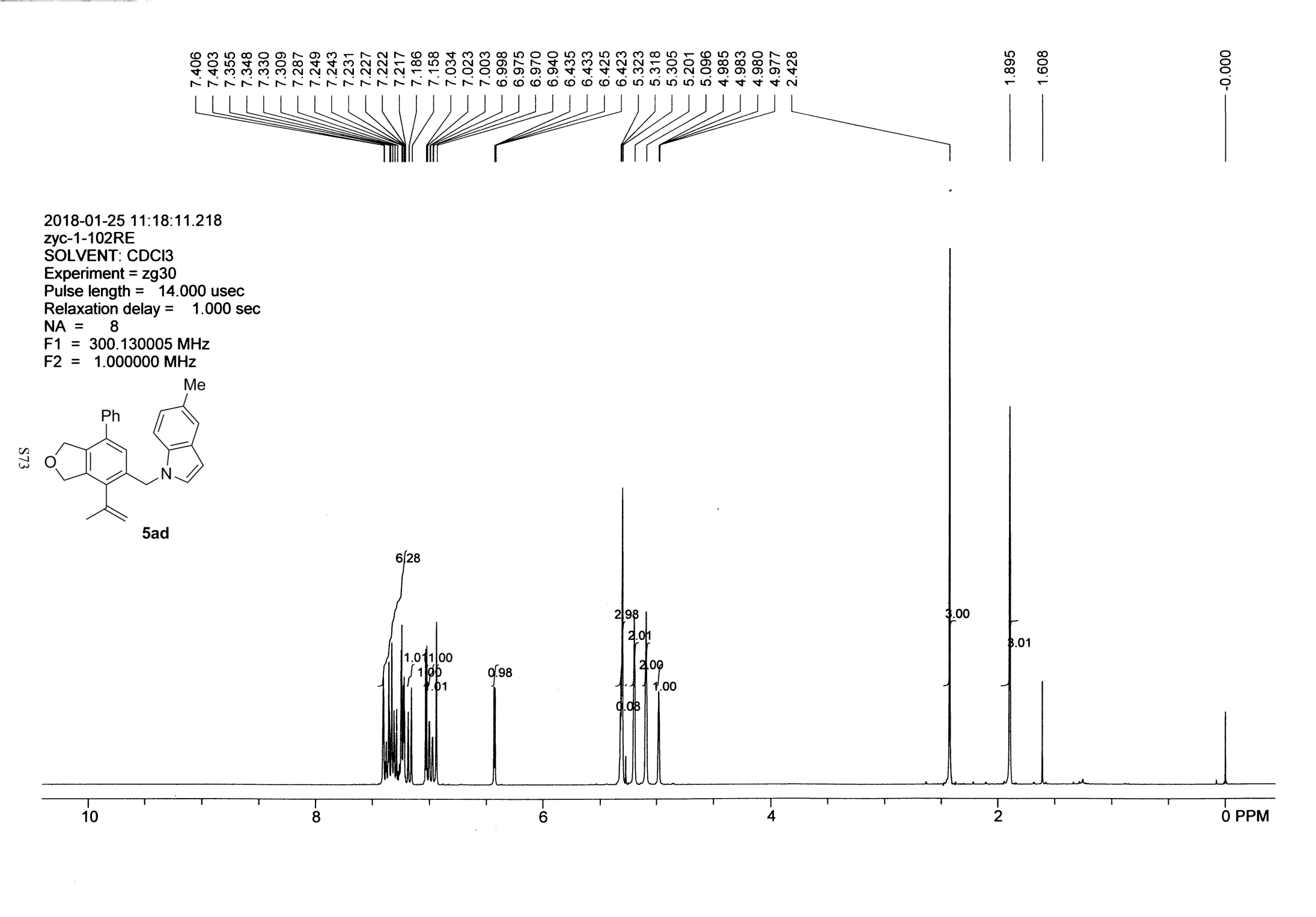

7.622
7.600
7.341
7.323
7.299
7.266
7.256
7.221
7.199
7.166
7.154
7.151
7.103
7.081
7.056
7.047
6.928
6.509
6.503
6.448
5.418
5.310
5.184
5.131
5.084
4.967
1.876
-0.000
2015-11-10 15:18:19.609
wwt-2-123
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
Ph
O
N
5ac
+
6ac
5ac/6ac = 95/5
10.87
1.13
1.04
1.01
0.05
3.10
2.00
2.02
0.16
0.12
1.02
2.96
10
8
6
4
2
PPM

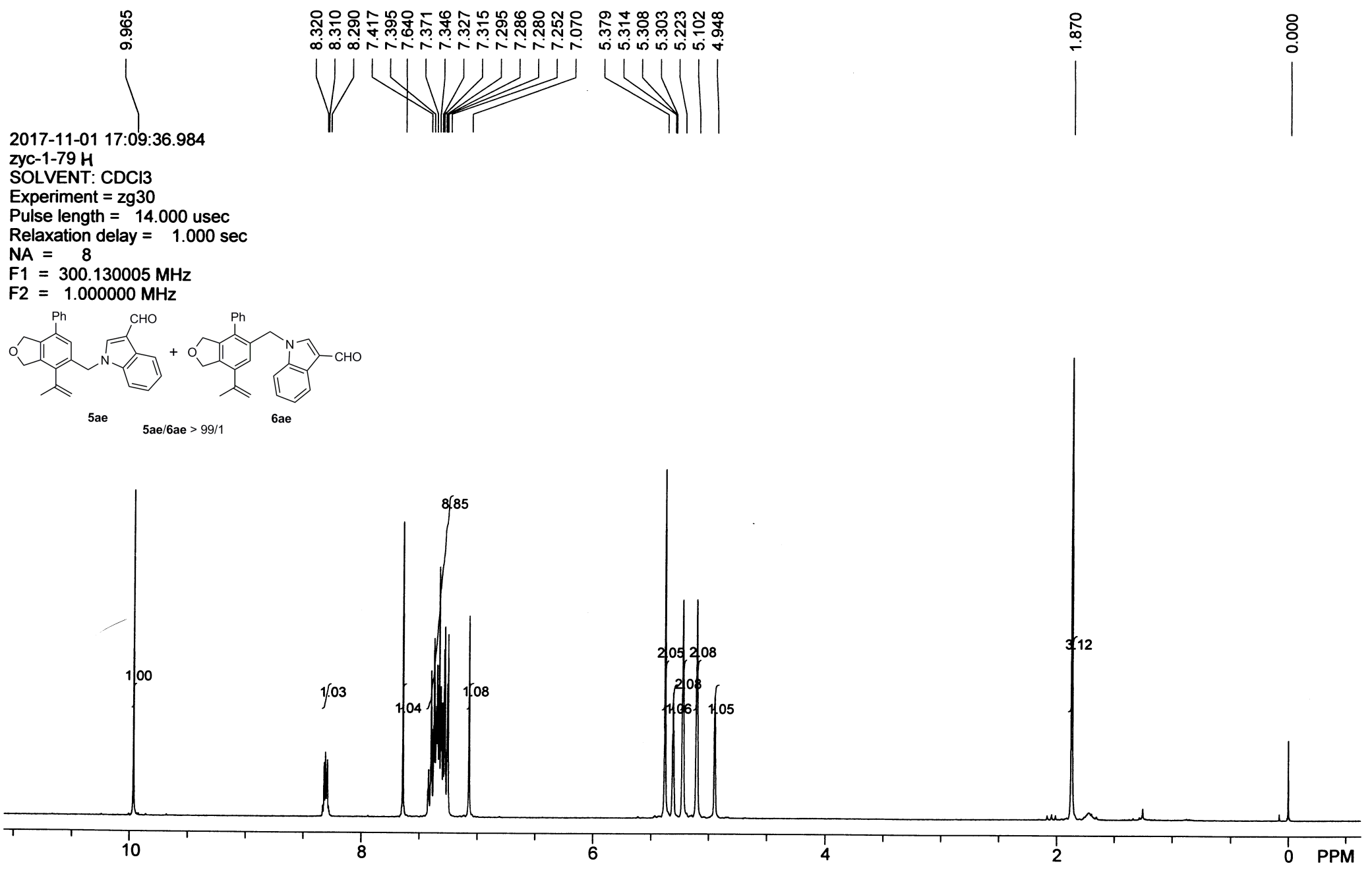

142.115
139.422
138.420
136.618
136.260
135.772
134.743
133.943
128.594
128.015
127.629
127.426
127.298
121.534
120.882
119.429
116.295
109.511
101.671
77.423
77.000
76.577
73.718
72.937
47.173
23.145
wwt-2-123
2015-11-10 15:31:56.109
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 191
Ph
O
N
5ac
+
Ph
O
N
6ac
5ac/6ac = 95/5
200
150
100
50
0 PPM

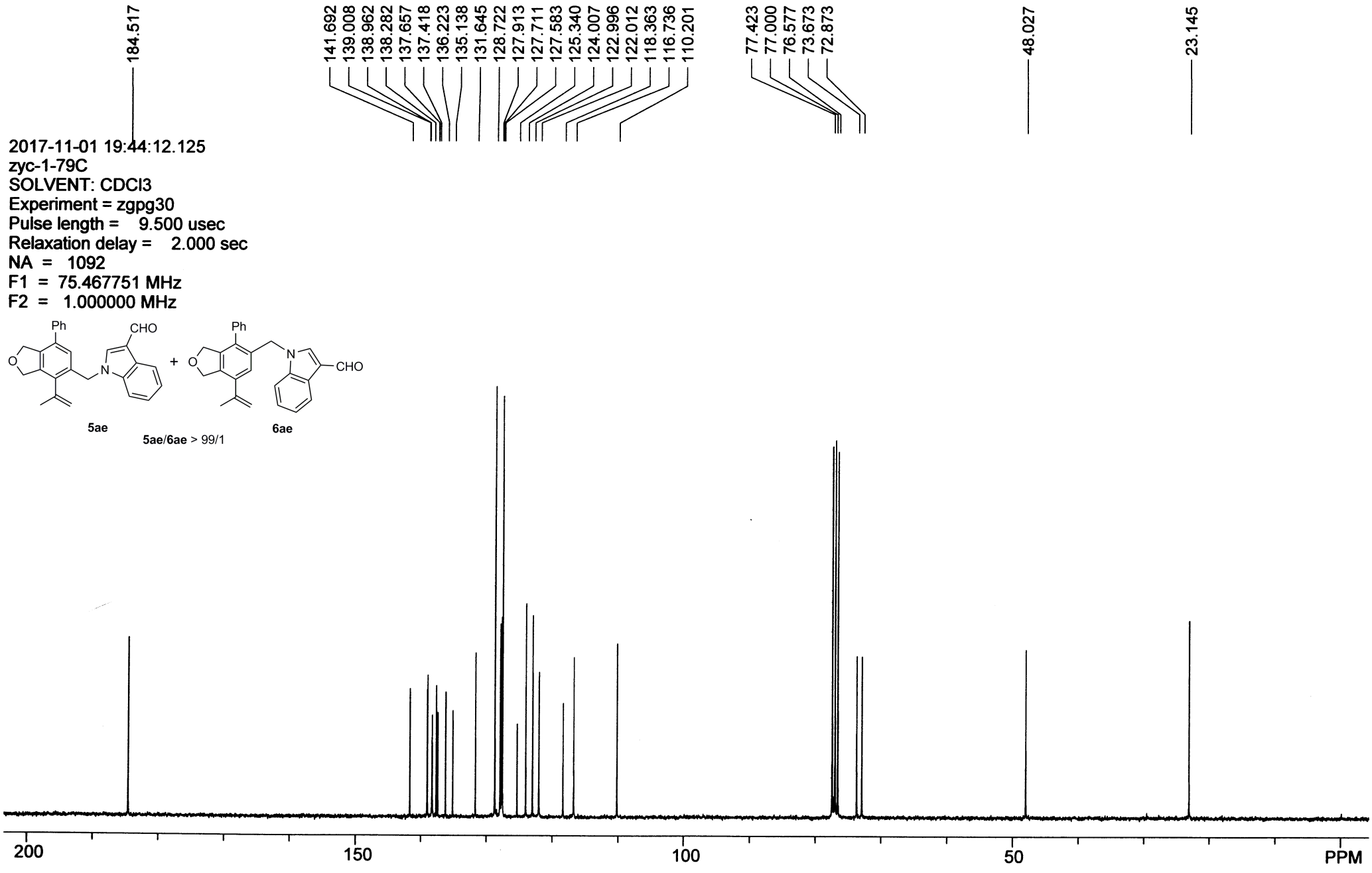

7.641
7.616
7.359
7.353
7.334
7.316
7.310
7.293
7.280
7.258
7.245
7.239
7.217
7.214
7.175
7.171
7.098
7.080
7.069
6.938
6.527
6.516
5.349
5.333
5.328
5.323
5.203
5.099
4.993
1.908
1.564
0.000
2017-10-25 11:55:31.125
zyc-1-68re
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 40
F1 = 300.130005 MHz
F2 = 1.000000 MHz
Ph
O
N
5ac
1.09
4.50
5.54
1.00
1.00
3.07
2.04
2.02
1.02
3.09
10
8
6
4
2
0
PPM

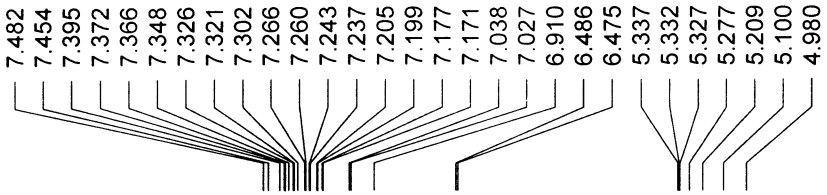

7.394
7.339
7.333
7.314
7.293
7.288
7.270
7.240
7.234
7.212
7.204
7.179
7.151
7.018
7.008
6.996
6.992
6.968
6.964
6.941
6.426
6.415
6.364
5.423
5.312
5.306
5.301
5.293
5.188
5.135
5.087
4.970
2.452
2.421
1.890
0.000
2017-12-11 16:02:16.890
zyc-1-102H
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
Me
Ph
O
N
5ad
+
Ph
O
N
6ad
Me
5ad/6ad = 95/5
8.22
3.19
1.00
0.05
3.20
2.00
2.03
1.04
0.15
0.16
3.09
0.16
3.01
10
8
6
4
2
0 PPM


142.198
139.514
138.429
136.627
135.818
134.780
134.109
128.906
128.612
128.079
127.684
127.445
127.371
123.198
120.560
116.277
109.208
101.129
77.423
77.000
76.577
73.764
72.983
47.264
23.182
21.334
2017-12-11 16:17:09.843
zyc-1-102C
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 210
F1 = 75.467751 MHz
F2 = 1.000000 MHz
Me
Ph
O
N
5ad
+
Ph
O
N
6ad
Me
5ad/6ad = 95/5
200
150
100
50
0
PPM

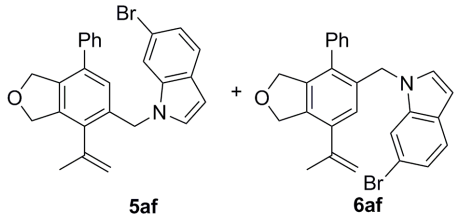

7.406
7.403
7.355
7.348
7.330
7.309
7.287
7.249
7.243
7.231
7.227
7.222
7.217
7.186
7.158
7.034
7.023
7.003
6.998
6.975
6.970
6.940
6.435
6.433
6.425
6.423
5.323
5.318
5.305
5.201
5.096
4.985
4.983
4.980
4.977
2.428
1.895
1.608
-0.000
2018-01-25 11:18:11.218
zyc-1-102RE
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
Me
Ph
O
N
5ad
6.28
1.01
1.00
1.00
1.01
0.98
2.98
2.01
2.00
1.00
0.08
3.00
3.01
10
8
6
4
2
0 PPM


9.965
8.320
8.310
8.290
7.417
7.395
7.640
7.371
7.346
7.327
7.315
7.295
7.286
7.280
7.252
7.070
5.379
5.314
5.308
5.303
5.223
5.102
4.948
1.870
0.000
2017-11-01 17:09:36.984
zyc-1-79 H
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
Ph
CHO
O
N
+
5ae
6ae
5ae/6ae > 99/1
1.00
1.03
1.04
8.85
1.08
2.05
2.08
2.08
1.06
1.05
3.12
10
8
6
4
2
0
PPM


184.517
141.692
139.008
138.962
138.282
137.657
137.418
136.223
135.138
131.645
128.722
127.913
127.711
127.583
125.340
124.007
122.996
122.012
118.363
116.736
110.201
77.423
77.000
76.577
73.673
72.873
48.027
23.145
2017-11-01 19:44:12.125
zyc-1-79C
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 1092
F1 = 75.467751 MHz
F2 = 1.000000 MHz
Ph
CHO
O
N
+
Ph
N
CHO
O
5ae
6ae
5ae/6ae > 99/1
200
150
100
50
PPM



2018-01-29 18:16:36.515
zyc-1-130H
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz

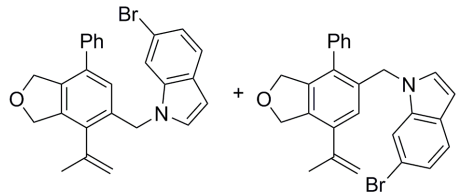


5af **6af**

5af/6af > 99/1

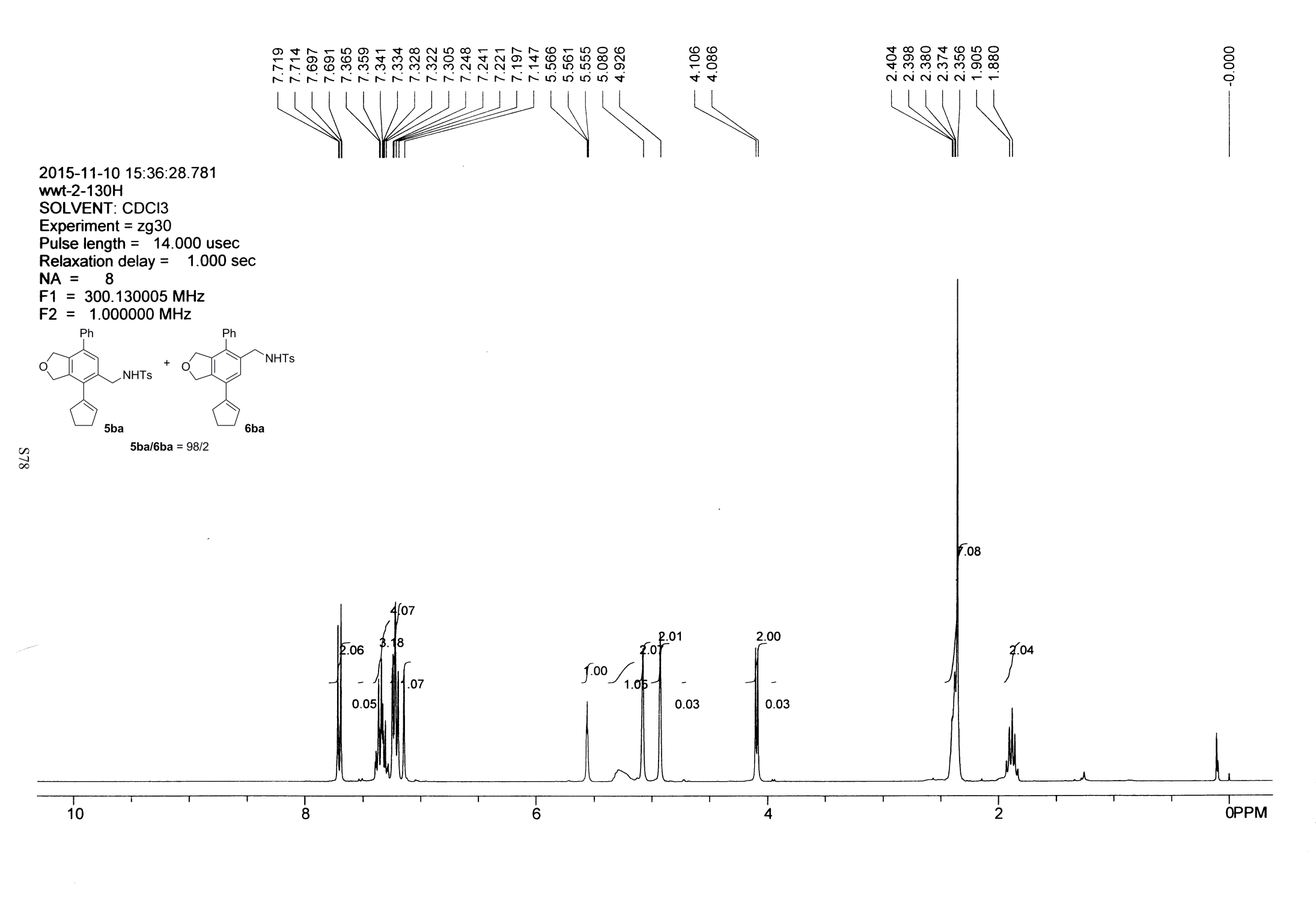
141.995
139.357
138.604
137.078
136.940
135.837
134.872
133.355
128.676
128.630
127.665
127.546
127.380
127.298
122.757
122.141
116.525
115.192
112.554
101.983
77.423
77.000
76.577
73.737
72.965
47.255
23.209

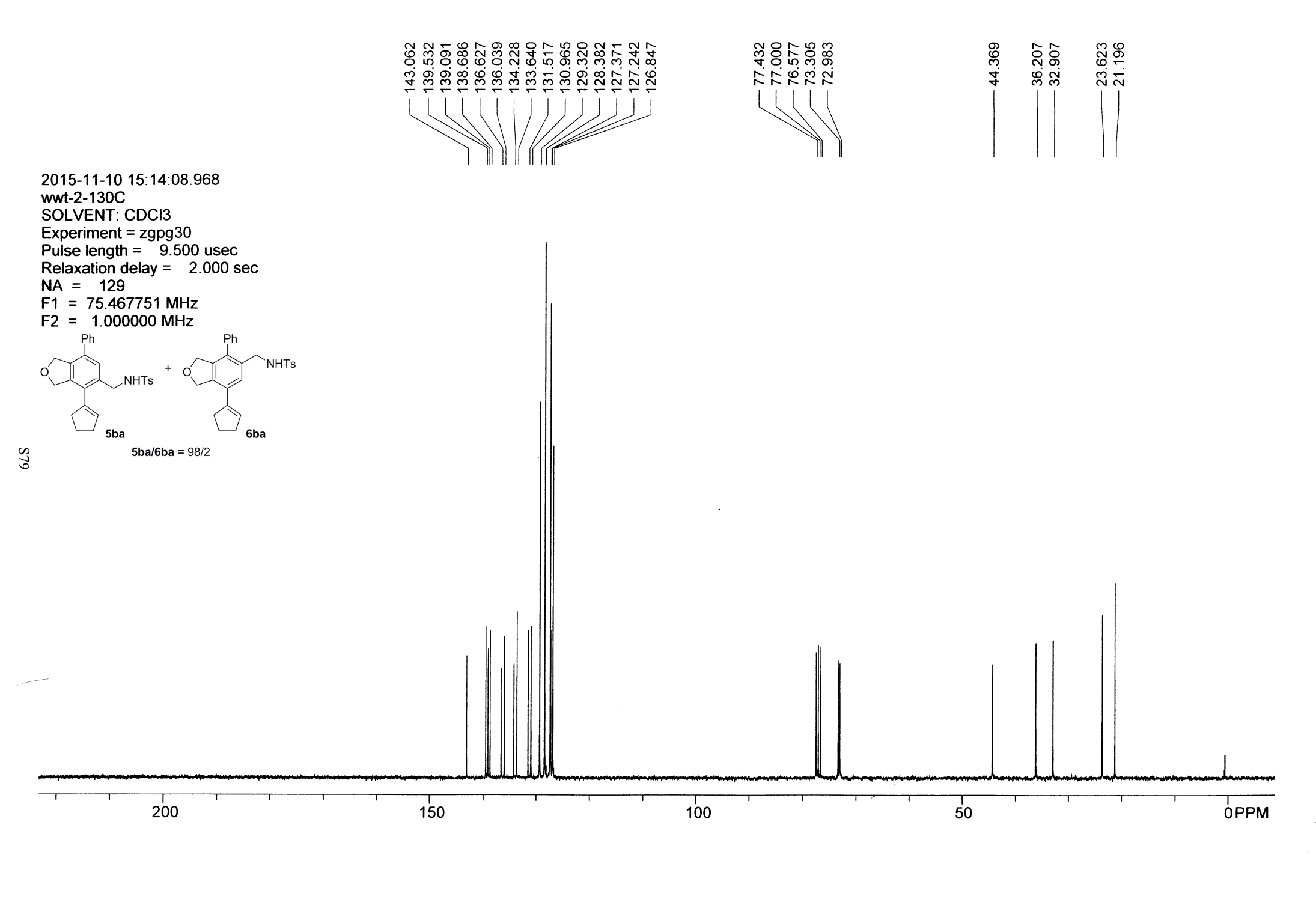
2018-01-29 18:41:36.671
zyc-1-130C
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 383
F1 = 75.467751 MHz
F2 = 1.000000 MHz

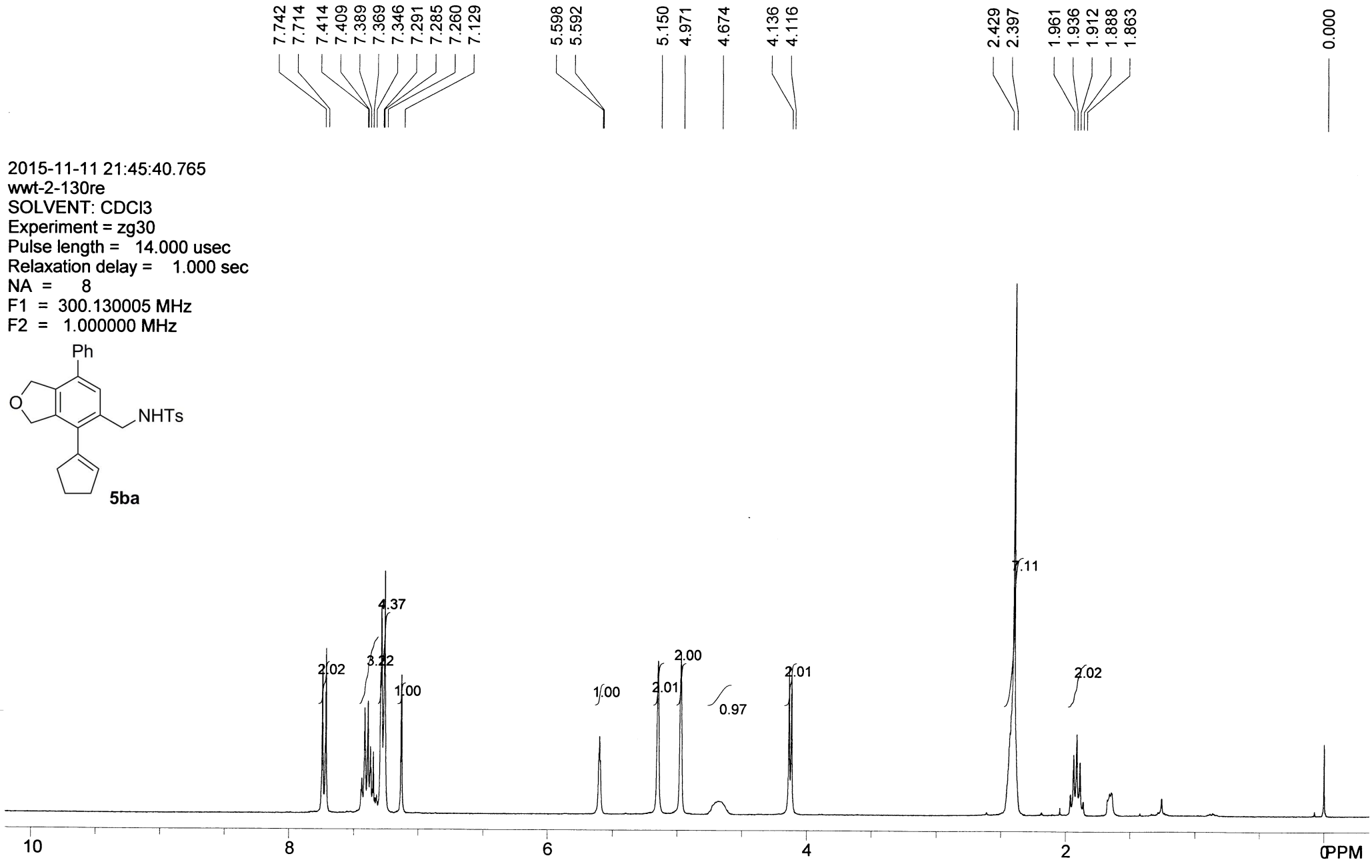


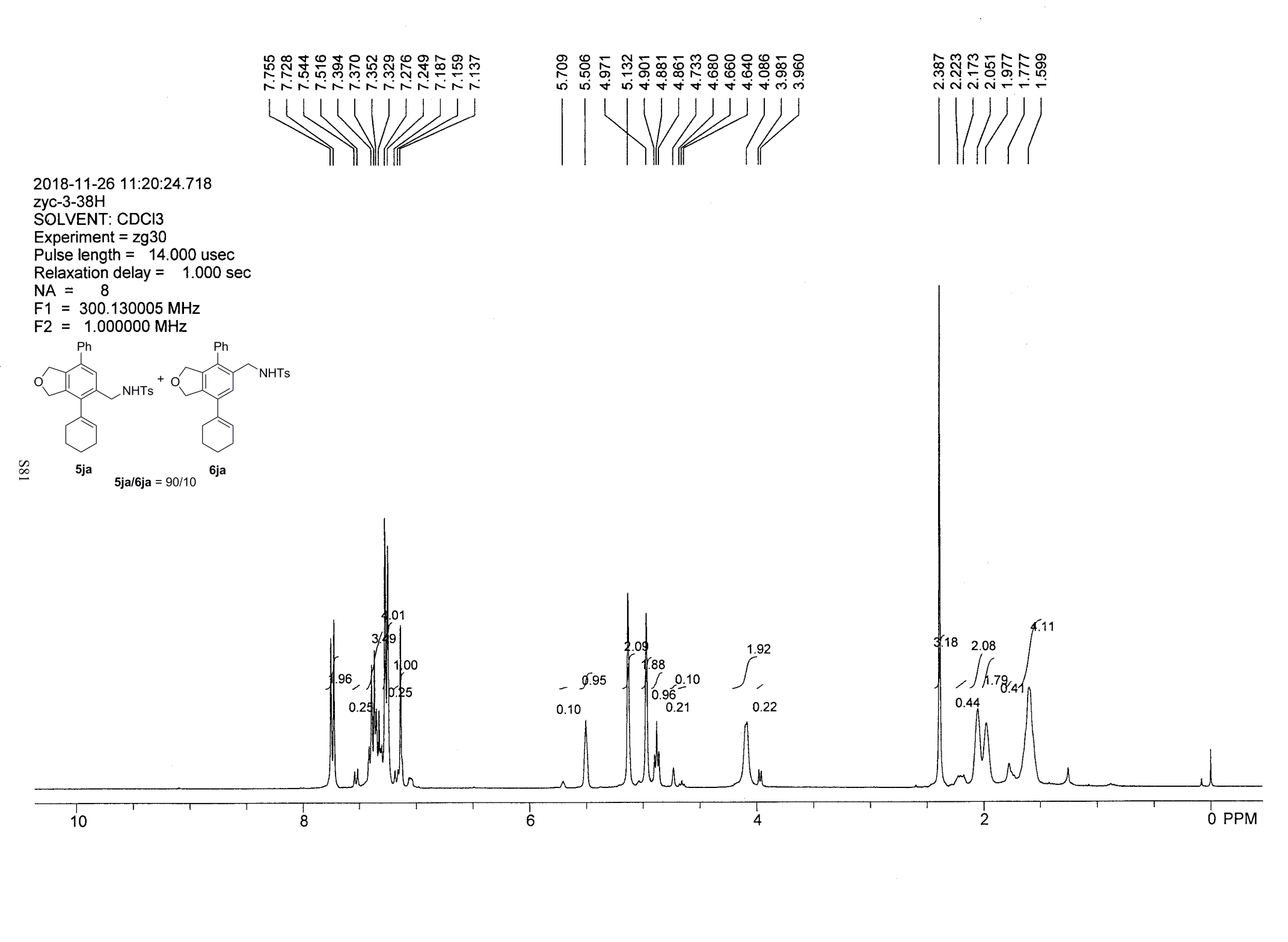
5af **6af**

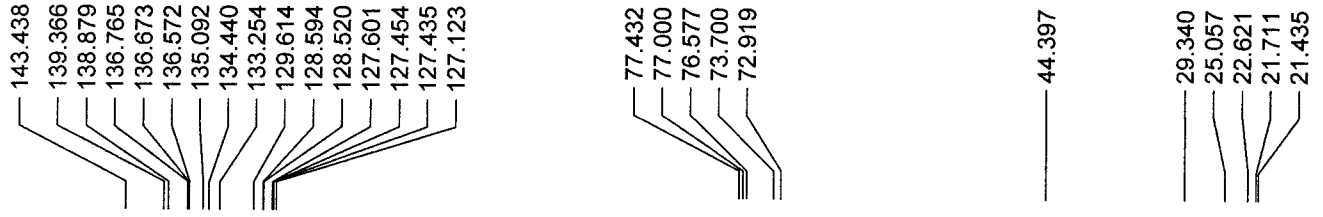
5af/6af > 99/1


7.719
7.714
7.697
7.691
7.365
7.359
7.341
7.334
7.328
7.322
7.305
7.248
7.241
7.221
7.197
7.147
5.566
5.561
5.555
5.080
4.926
4.106
4.086
2.404
2.398
2.380
2.374
2.356
1.905
1.880
-0.000
2015-11-10 15:36:28.781
wwt-2-130H
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
Ph
NHTs
5ba
Ph
NHTs
6ba
5ba/6ba = 98/2
2.06
4.07
3.18
1.07
0.05
1.00
1.05
2.07
2.01
0.03
2.00
0.03
7.08
2.04
10
8
6
4
2
0PPM

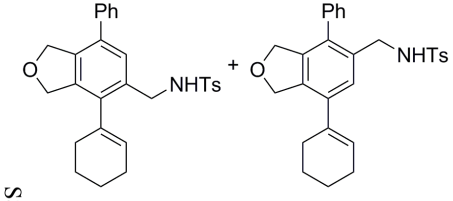

143.062
139.532
139.091
138.686
136.627
136.039
134.228
133.640
131.517
130.965
129.320
128.382
127.371
127.242
126.847
77.432
77.000
76.577
73.305
72.983
44.369
36.207
32.907
23.623
21.196
2015-11-10 15:14:08.968
wwt-2-130C
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 129
F1 = 75.467751 MHz
F2 = 1.000000 MHz
Ph
NHTs
O
+
5ba
6ba
5ba/6ba = 98/2
200
150
100
50
0PPM


7.742
7.714
7.414
7.409
7.389
7.369
7.346
7.291
7.285
7.260
7.129
5.598
5.592
5.150
4.971
4.674
4.136
4.116
2.429
2.397
1.961
1.936
1.912
1.888
1.863
0.000
2015-11-11 21:45:40.765
wwt-2-130re
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
Ph
O
NHTs
5ba
2.02
4.37
3.22
1.00
1.00
2.01
2.00
0.97
2.01
7.11
2.02
10
8
6
4
2
0
PPM


7.755
7.728
7.544
7.516
7.394
7.370
7.352
7.329
7.276
7.249
7.187
7.159
7.137
5.709
5.506
4.971
5.132
4.901
4.881
4.861
4.733
4.680
4.660
4.640
4.086
3.981
3.960
2.387
2.223
2.173
2.051
1.977
1.777
1.599
2018-11-26 11:20:24.718
zyc-3-38H
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
Ph
NHTs
O
+
5ja
6ja
5ja/6ja = 90/10
S81
1.96
0.25
3.49
4.01
1.00
0.25
0.10
0.95
2.09
1.88
0.96
0.10
0.21
1.92
0.22
3.18
0.44
2.08
1.79
0.41
4.11
10
8
6
4
2
0 PPM

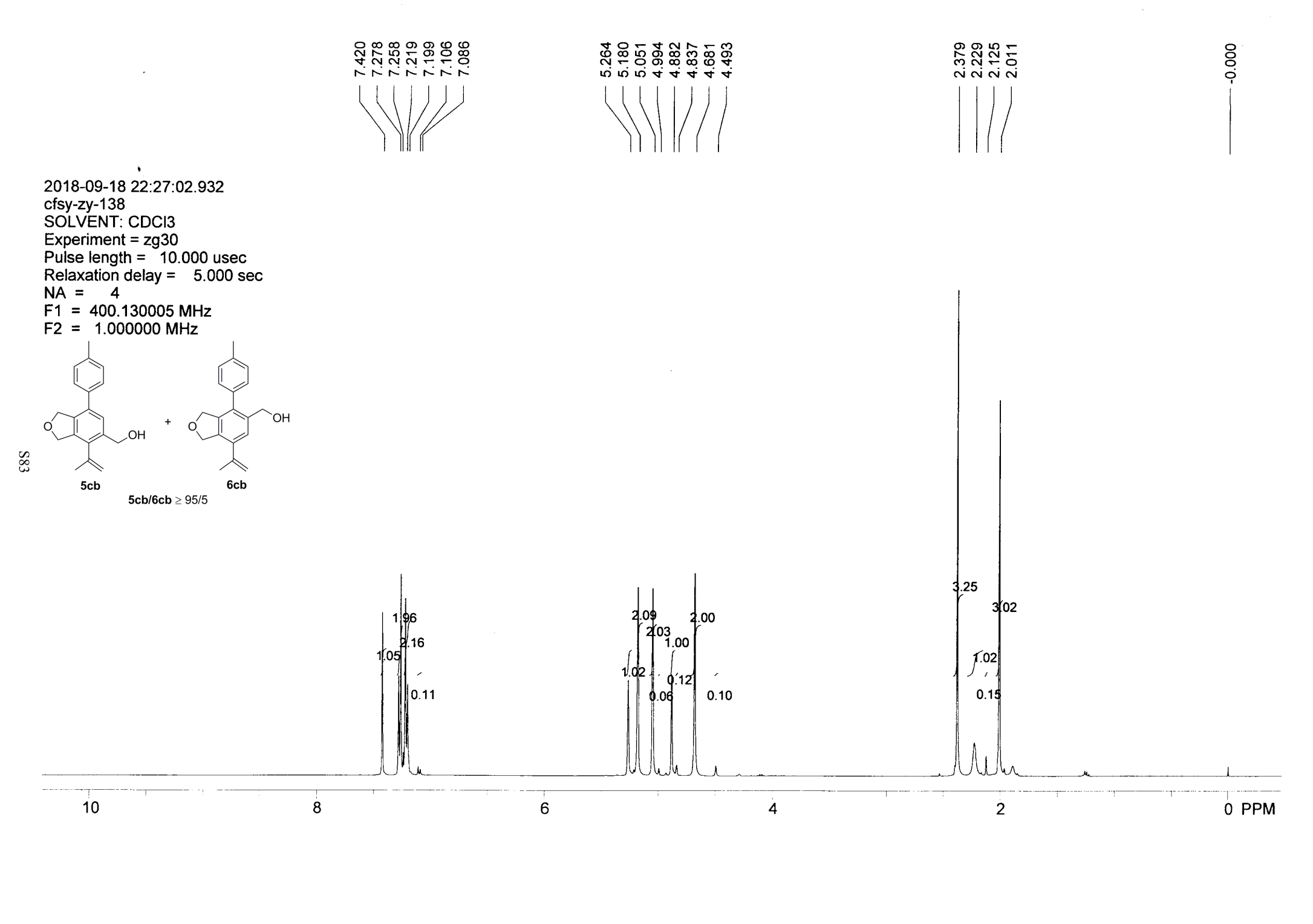


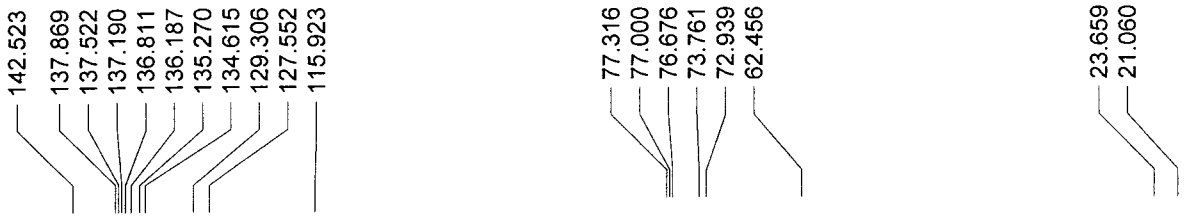
2018-11-26 12:50:56.453
zyc-3-38C
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 1520
F1 = 75.467751 MHz
F2 = 1.000000 MHz



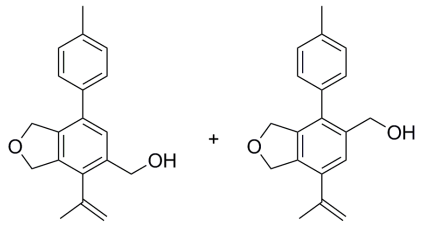
5ja **6ja**

5ja/6ja = 90/10


7.420
7.278
7.258
7.219
7.199
7.106
7.086
5.264
5.180
5.051
4.994
4.882
4.837
4.681
4.493
2.379
2.229
2.125
2.011
-0.000
2018-09-18 22:27:02.932
cfsy-zy-138
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 10.000 usec
Relaxation delay = 5.000 sec
NA = 4
F1 = 400.130005 MHz
F2 = 1.000000 MHz
O
OH
+
O
OH
5cb
6cb
5cb/6cb ≥ 95/5
1.05
1.96
2.16
0.11
1.02
2.09
2.03
1.00
0.12
0.06
2.00
0.10
3.25
1.02
0.15
3.02
10
8
6
4
2
0 PPM

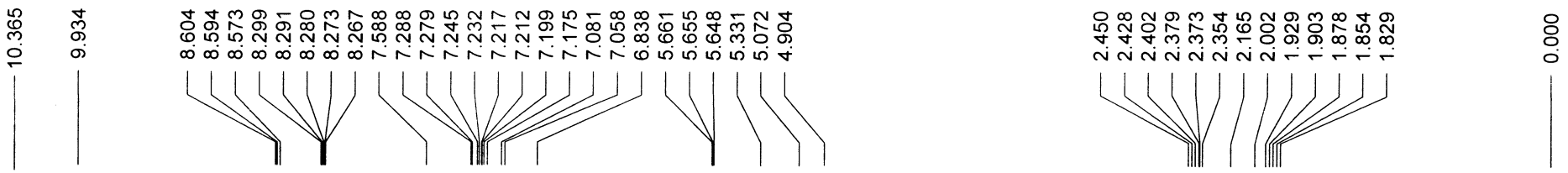


2018-09-18 22:34:58.797
cfsy-zy-138
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 10.000 usec
Relaxation delay = 2.000 sec
NA = 128
F1 = 100.612770 MHz
F2 = 1.000000 MHz

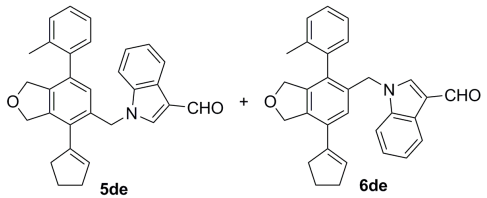


5cb **6cb**

5cb/6cb ≥ 95/5

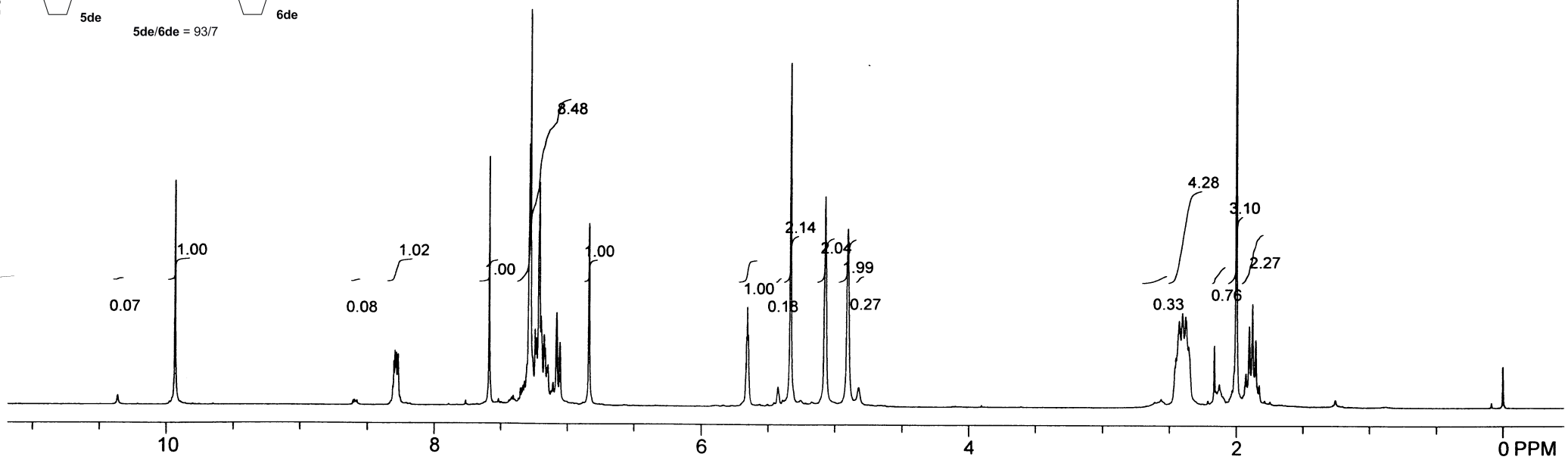


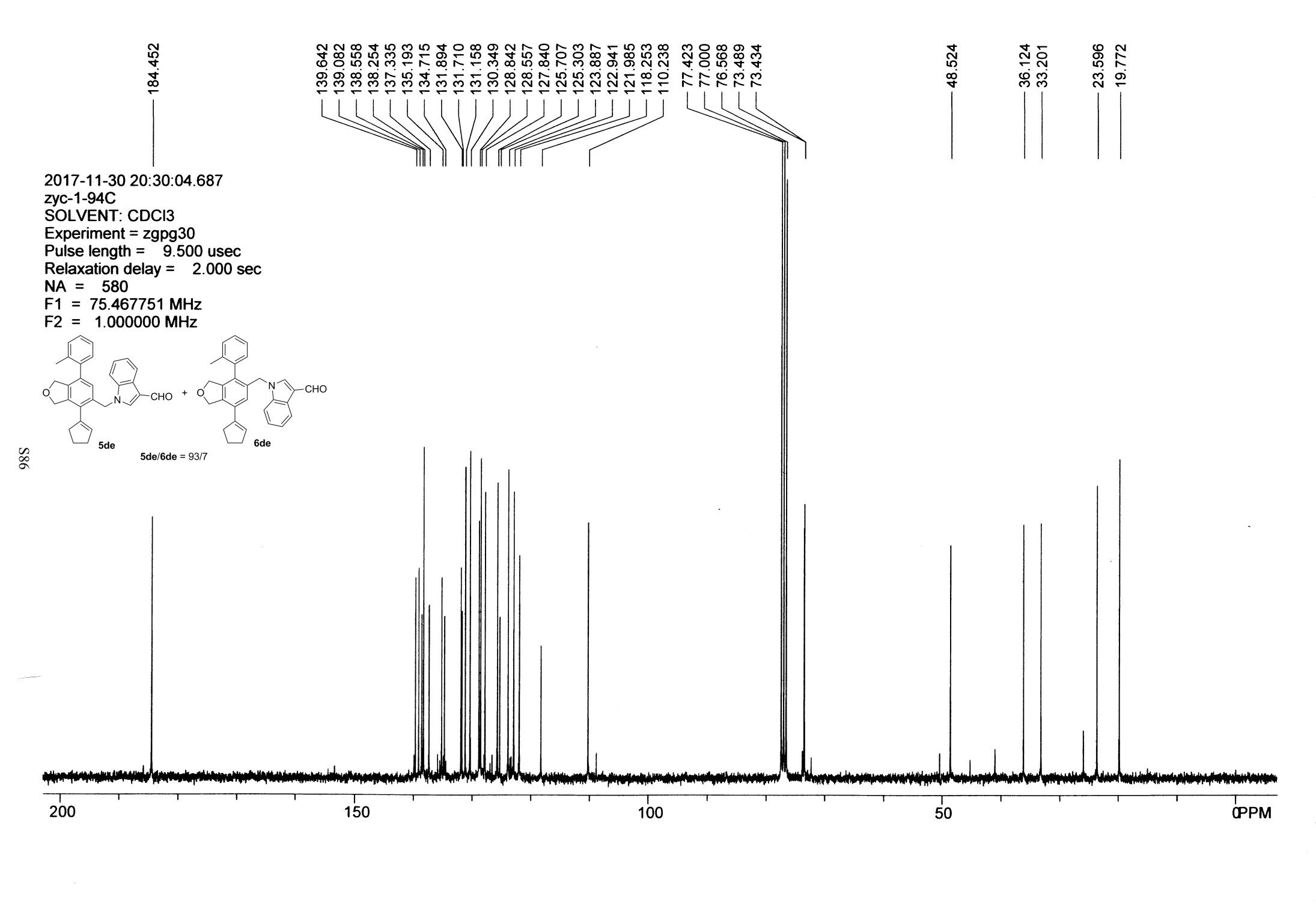
2017-11-30 16:36:24.093
zyc-1-94H
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz

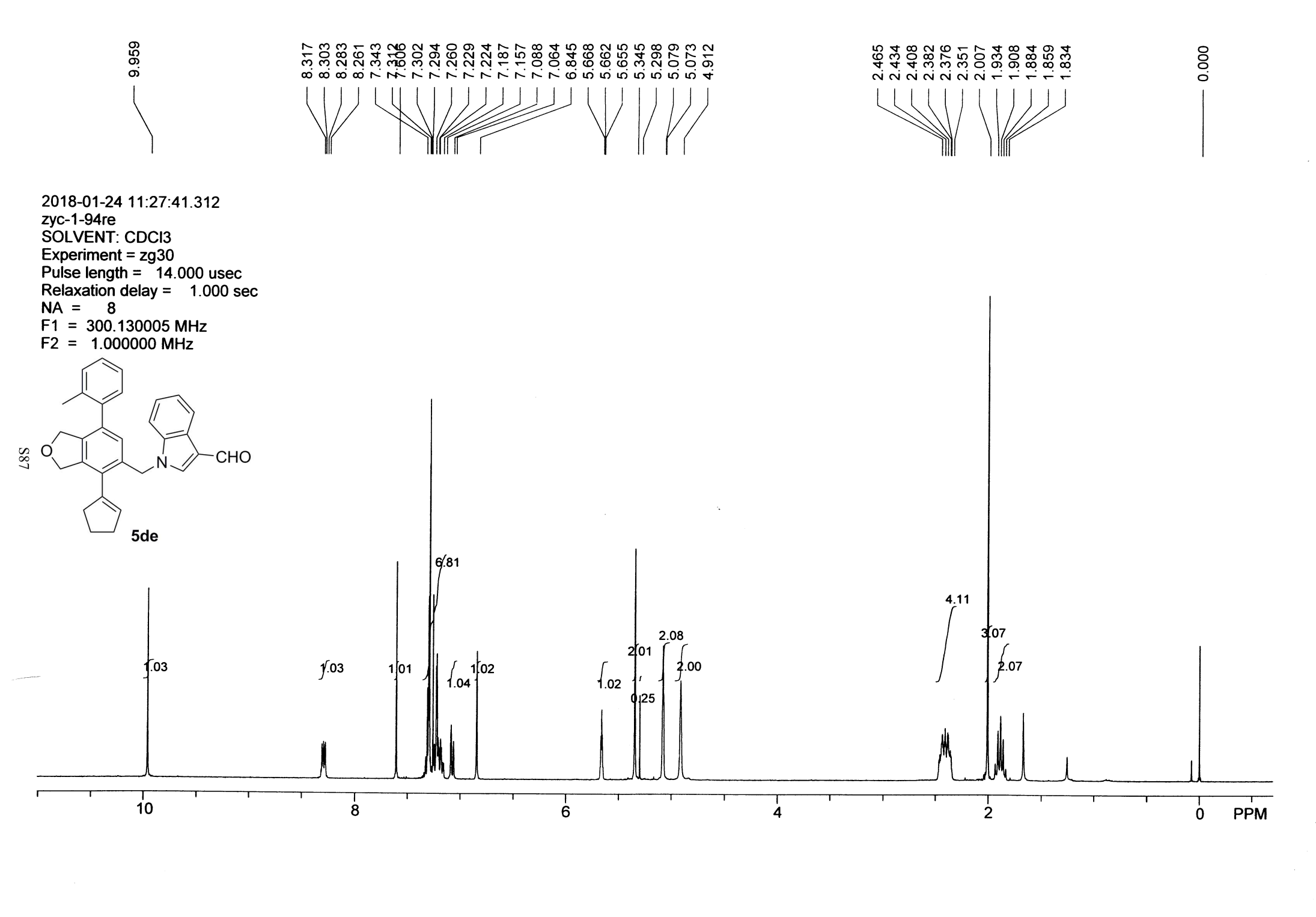


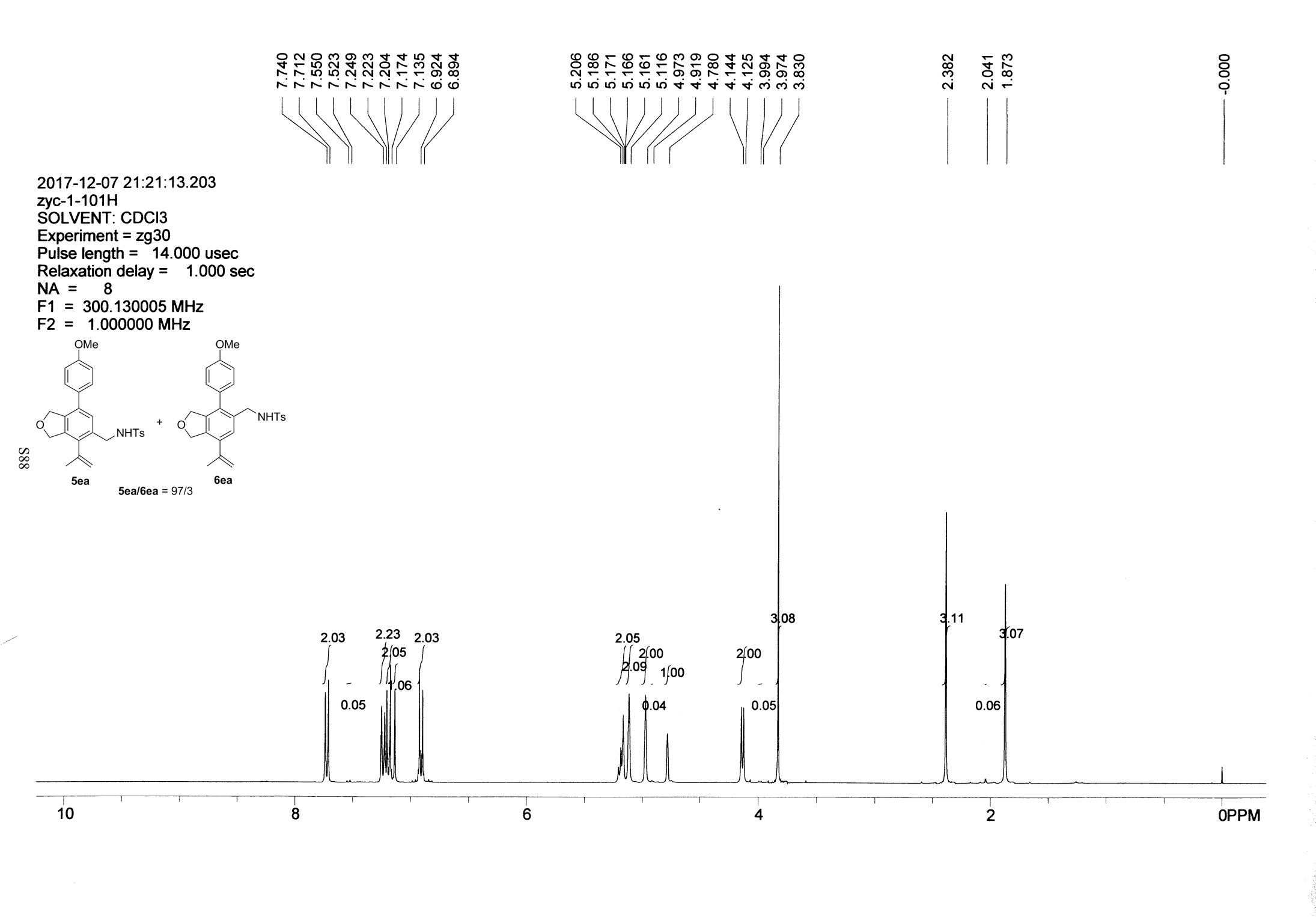
5de **6de**

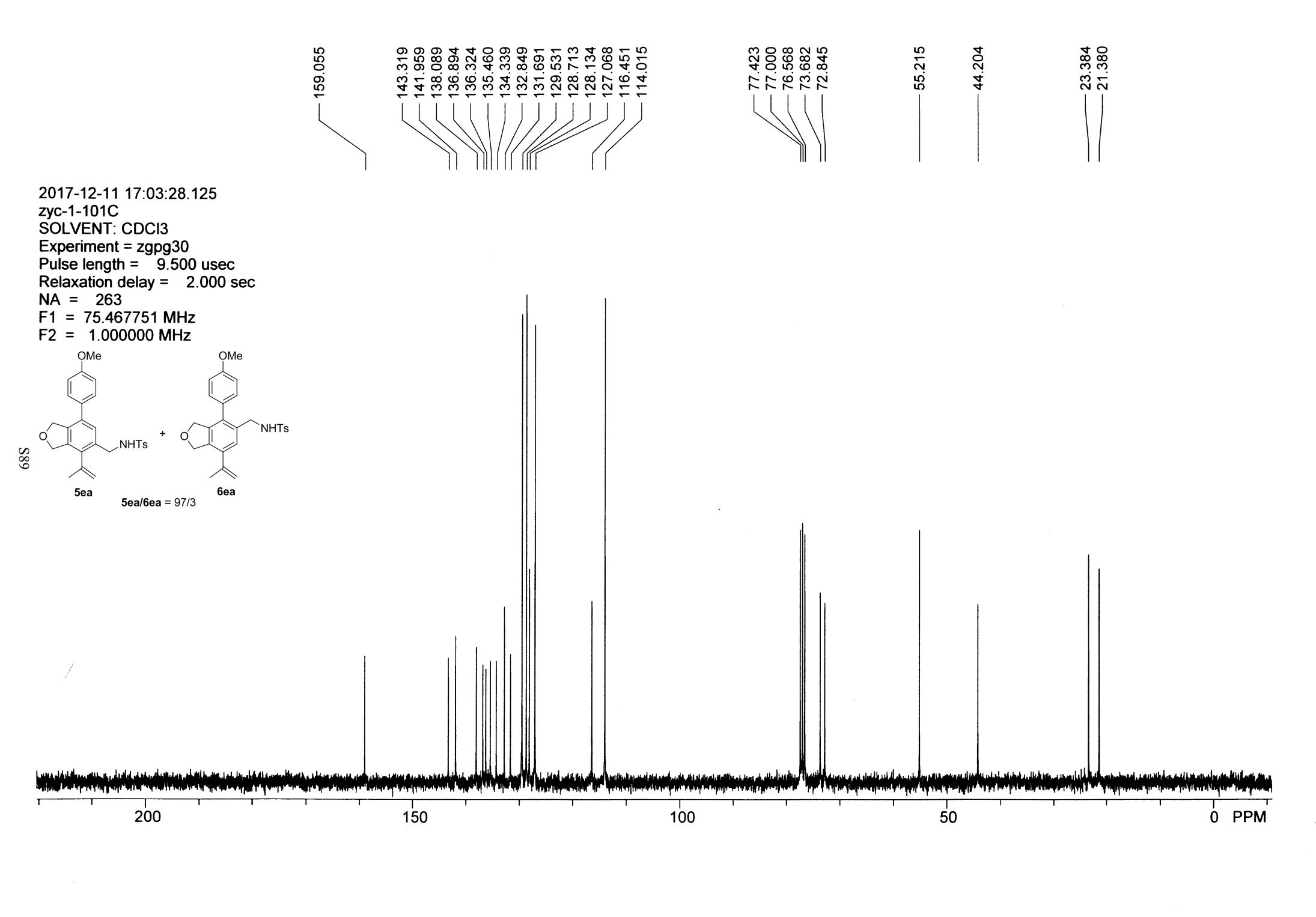
5de/6de = 93/7

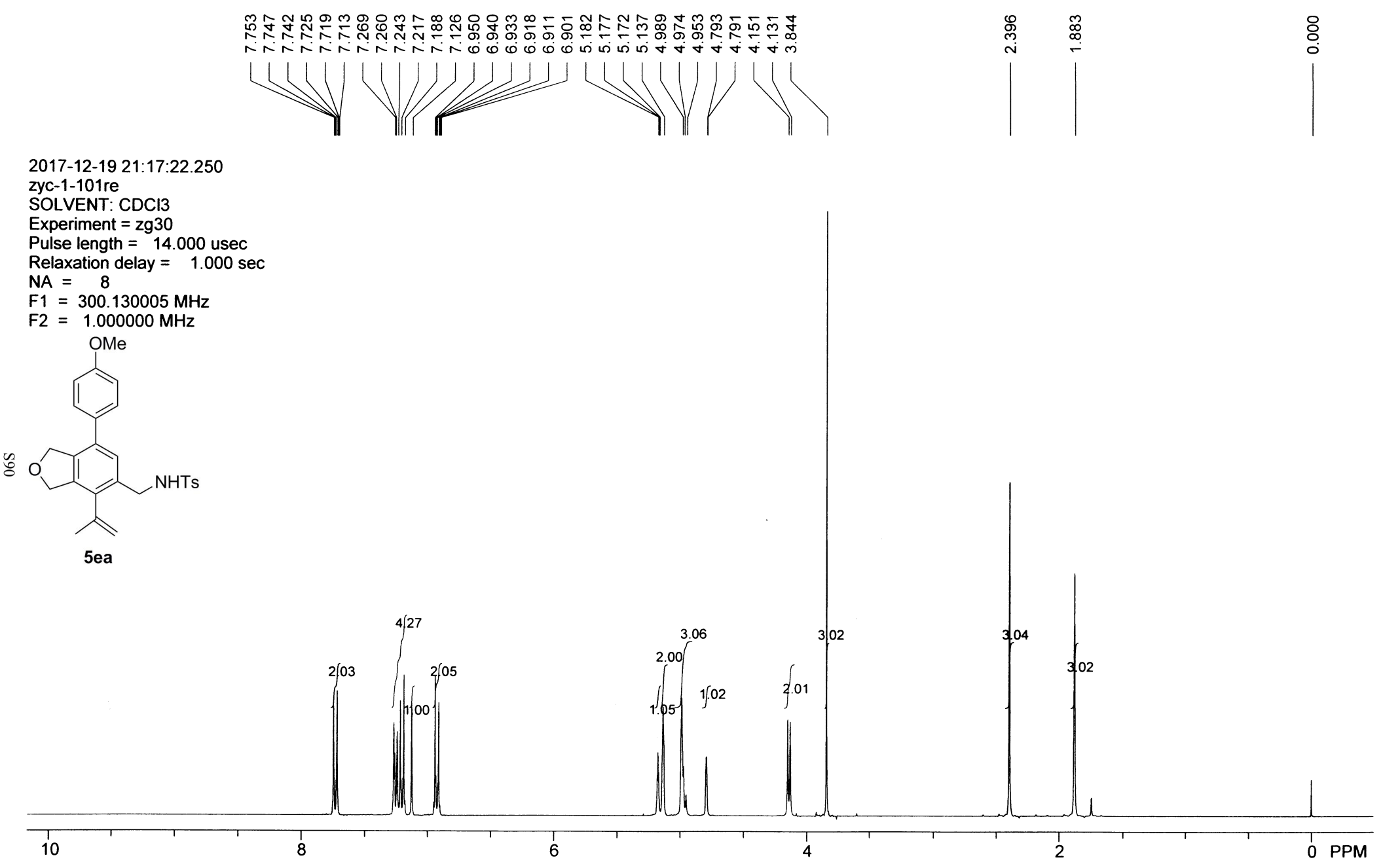


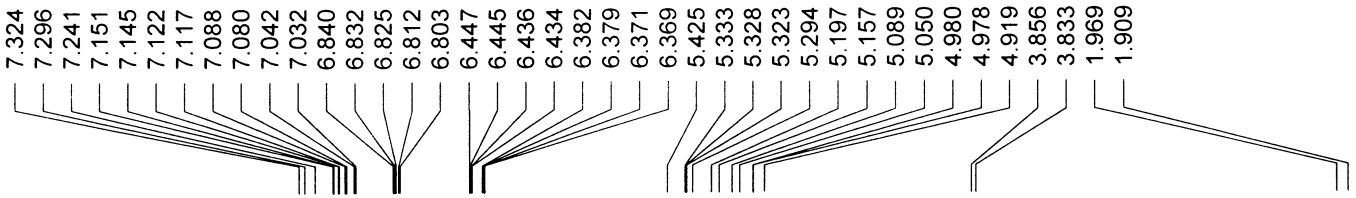

184.452
139.642
139.082
138.558
138.254
137.335
135.193
134.715
131.894
131.710
131.158
130.349
128.842
128.557
127.840
125.707
125.303
123.887
122.941
121.985
118.253
110.238
77.423
77.000
76.568
73.489
73.434
48.524
36.124
33.201
23.596
19.772
2017-11-30 20:30:04.687
zyc-1-94C
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 580
F1 = 75.467751 MHz
F2 = 1.000000 MHz
CHO
5de
6de
5de/6de = 93/7
98S
200
150
100
50
0PPM


9.959
8.317
8.303
8.283
8.261
7.343
7.312
7.306
7.302
7.294
7.260
7.229
7.224
7.187
7.157
7.088
7.064
6.845
5.668
5.662
5.655
5.345
5.298
5.079
5.073
4.912
2.465
2.434
2.408
2.382
2.376
2.351
2.007
1.934
1.908
1.884
1.859
1.834
0.000
2018-01-24 11:27:41.312
zyc-1-94re
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
O
N
CHO
5de
S87
1.03
1.03
1.01
6.81
1.04
1.02
1.02
2.01
0.25
2.08
2.00
4.11
3.07
2.07
10
8
6
4
2
0
PPM

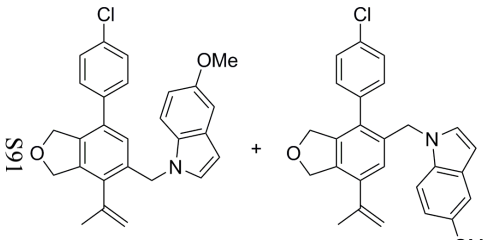

7.740
7.712
7.550
7.523
7.249
7.223
7.204
7.174
7.135
6.924
6.894
5.206
5.186
5.171
5.166
5.161
5.116
4.973
4.919
4.780
4.144
4.125
3.994
3.974
3.830
2.382
2.041
1.873
-0.000
2017-12-07 21:21:13.203
zyc-1-101H
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
OMe
NHTs
O
+
5ea
6ea
5ea/6ea = 97/3
S88
2.03
2.23
2.05
2.03
0.05
1.06
2.05
2.00
2.09
1.00
0.04
2.00
0.05
3.08
3.11
3.07
0.06
10
8
6
4
2
0PPM


159.055
143.319
141.959
138.089
136.894
136.324
135.460
134.339
132.849
131.691
129.531
128.713
128.134
127.068
116.451
114.015
77.423
77.000
76.568
73.682
72.845
55.215
44.204
23.384
21.380
2017-12-11 17:03:28.125
zyc-1-101C
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 263
F1 = 75.467751 MHz
F2 = 1.000000 MHz
OMe
NHTs
5ea
6ea
5ea/6ea = 97/3
200
150
100
50
0 PPM

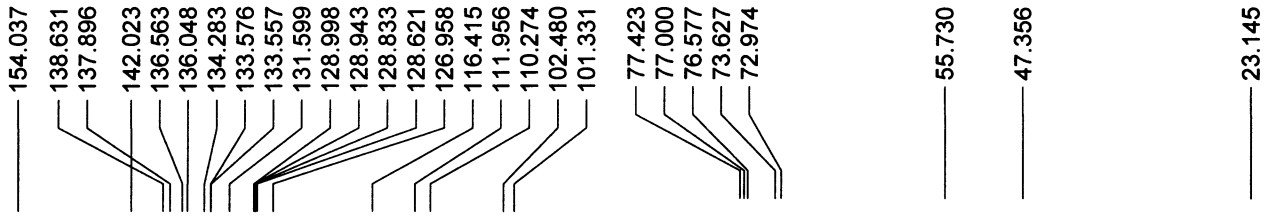

7.753
7.747
7.742
7.725
7.719
7.713
7.269
7.260
7.243
7.217
7.188
7.126
6.950
6.940
6.933
6.918
6.911
6.901
5.182
5.177
5.172
5.137
4.989
4.974
4.953
4.793
4.791
4.151
4.131
3.844
2.396
1.883
0.000
2017-12-19 21:17:22.250
zyc-1-101re
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
OMe
O
NHTs
5ea
2.03
4.27
1.00
2.05
1.05
2.00
3.06
1.02
2.01
3.02
3.04
3.02
10
8
6
4
2
0 PPM



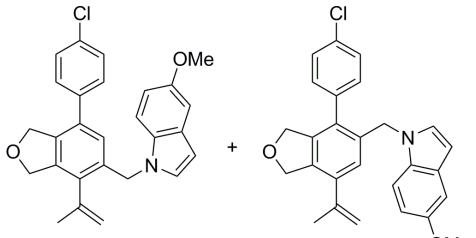
2017-11-19 21:25:23.234
zyc-1-87H
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz



5fg/6fg = 97/3

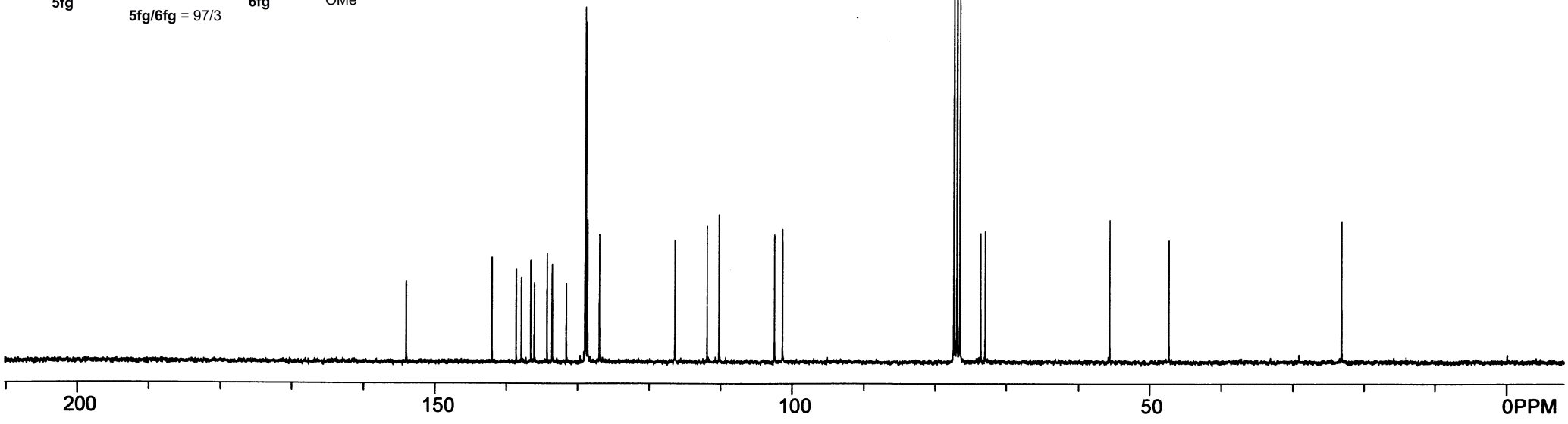


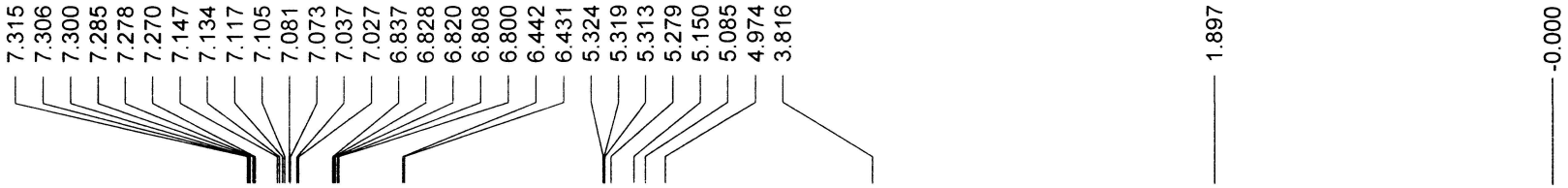
2017-11-19 22:46:27.531
zyc-1-87'C
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 1329
F1 = 75.467751 MHz
F2 = 1.000000 MHz



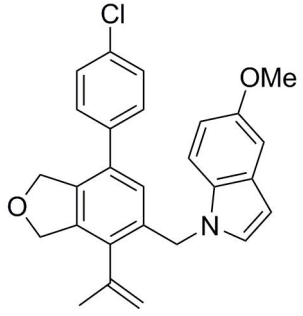
5fg **6fg**

5fg/6fg = 97/3

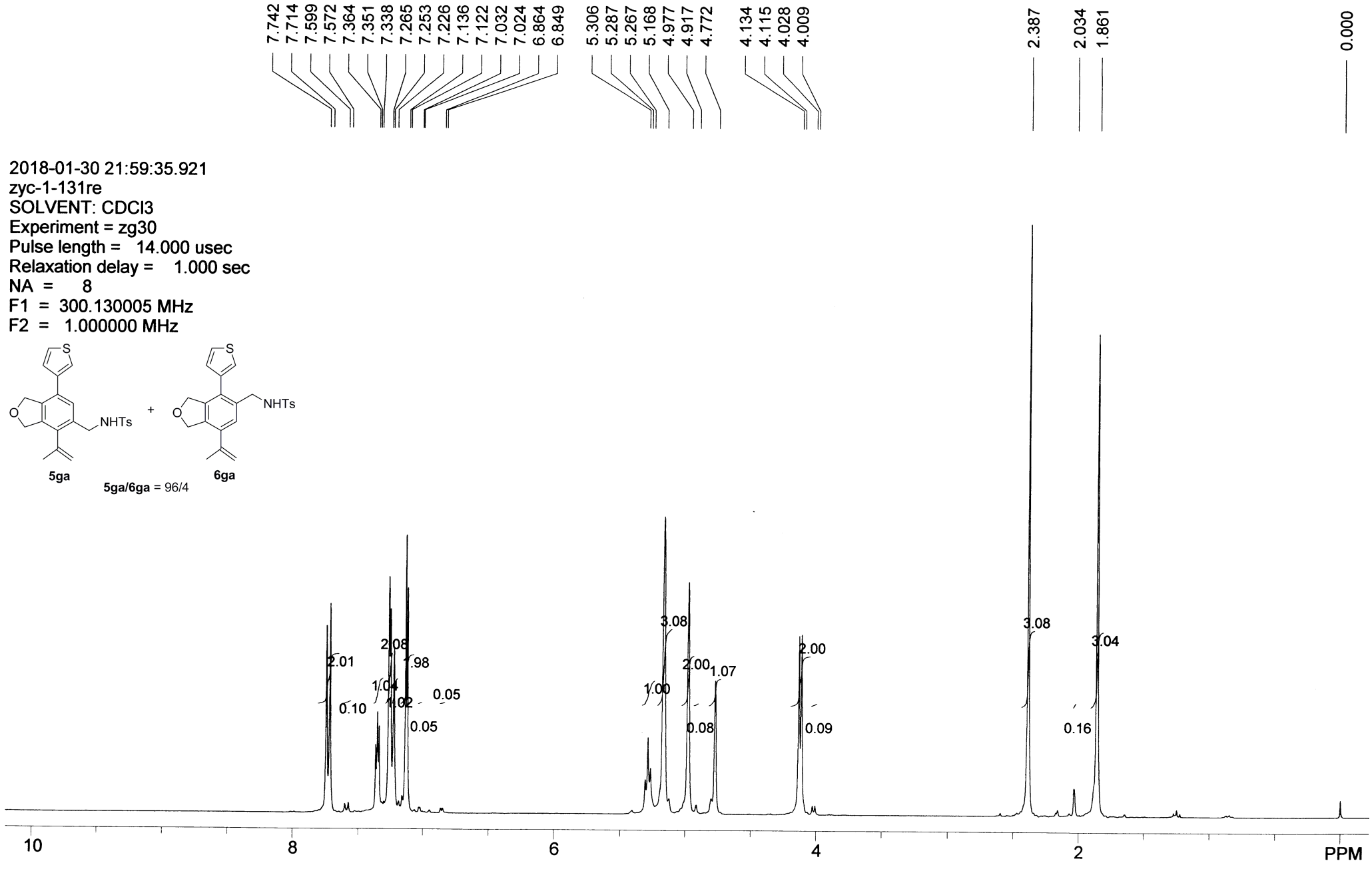


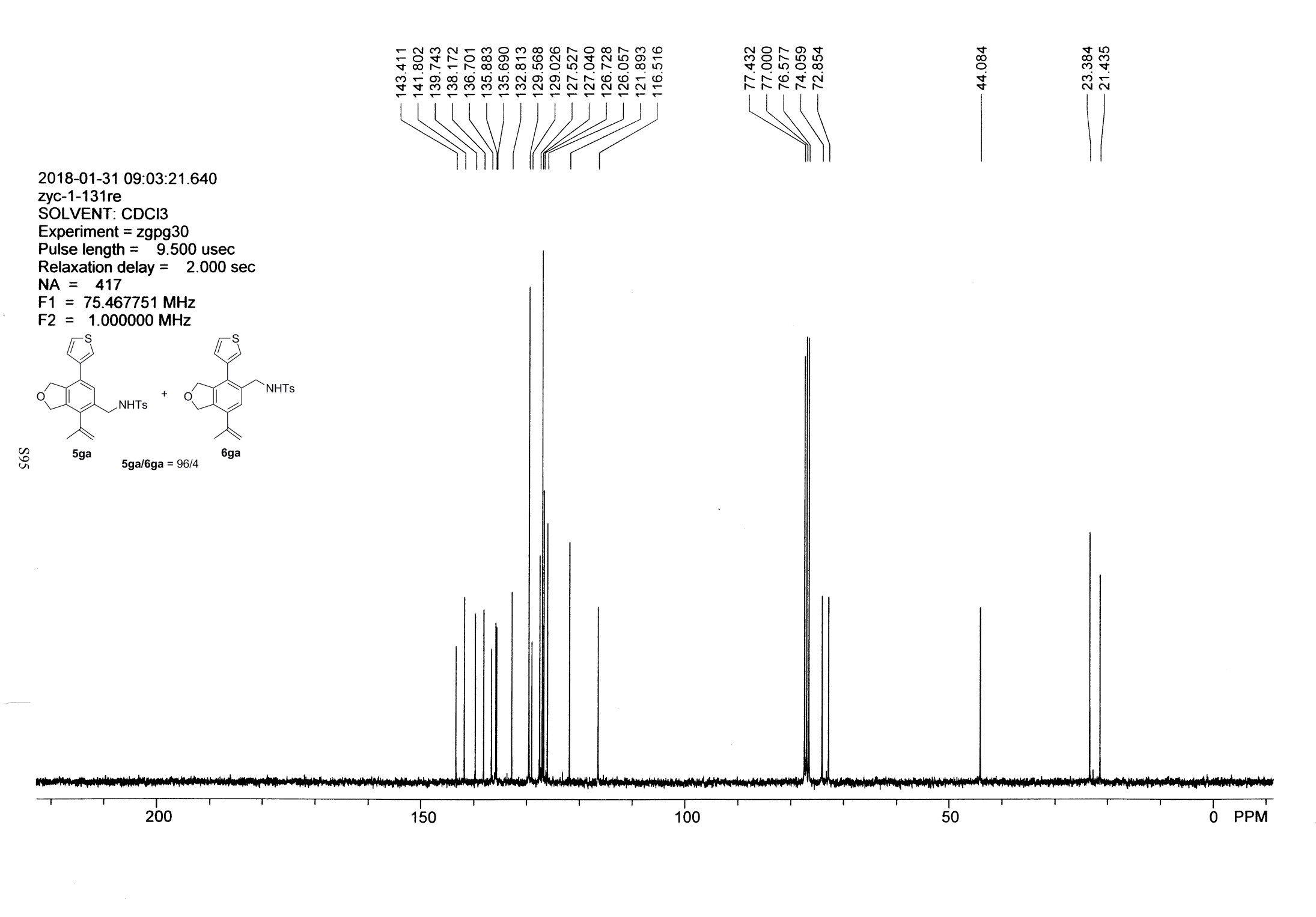


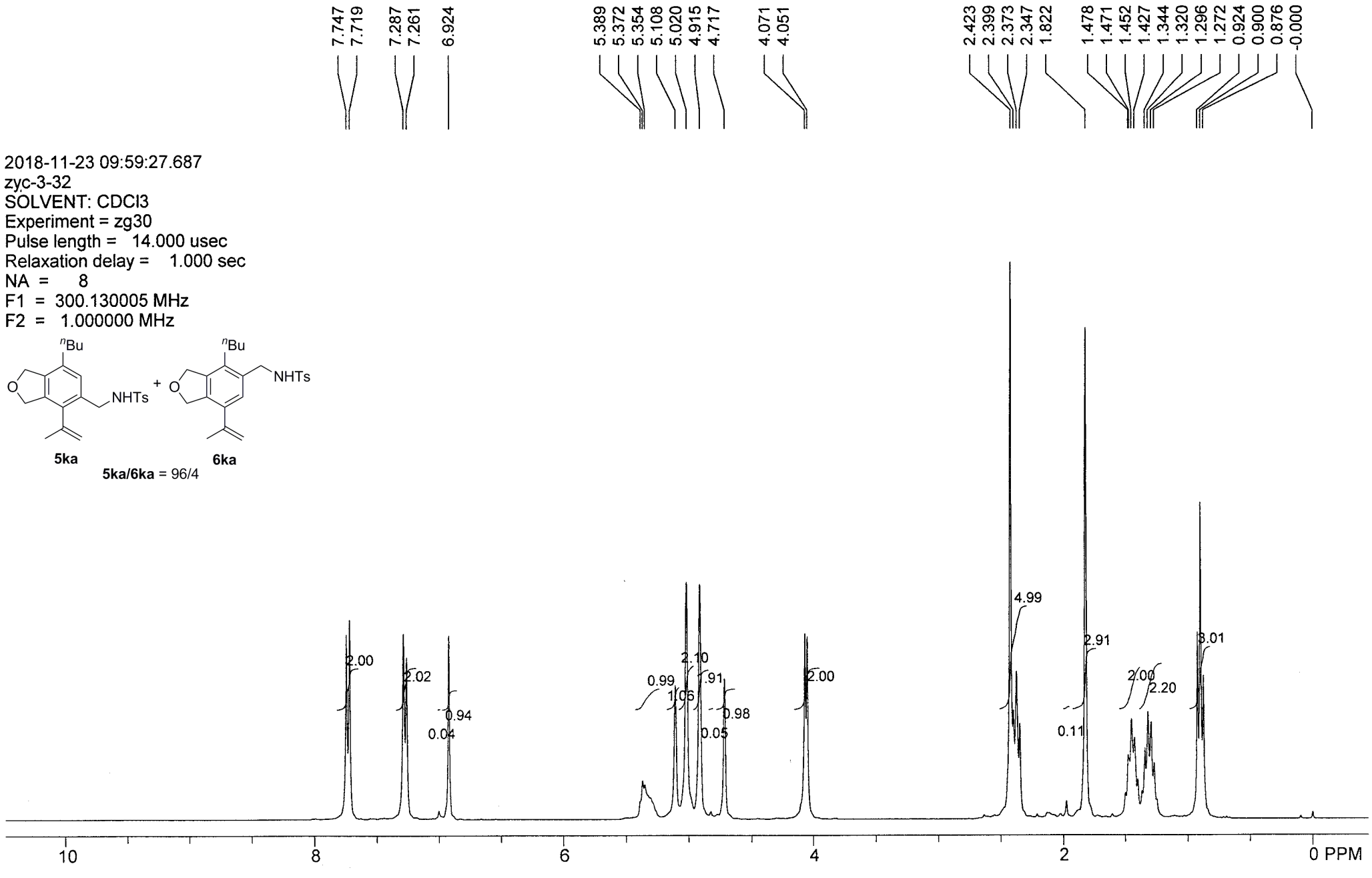
2017-12-17 09:08:09.625
zyc-1-87reH
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz

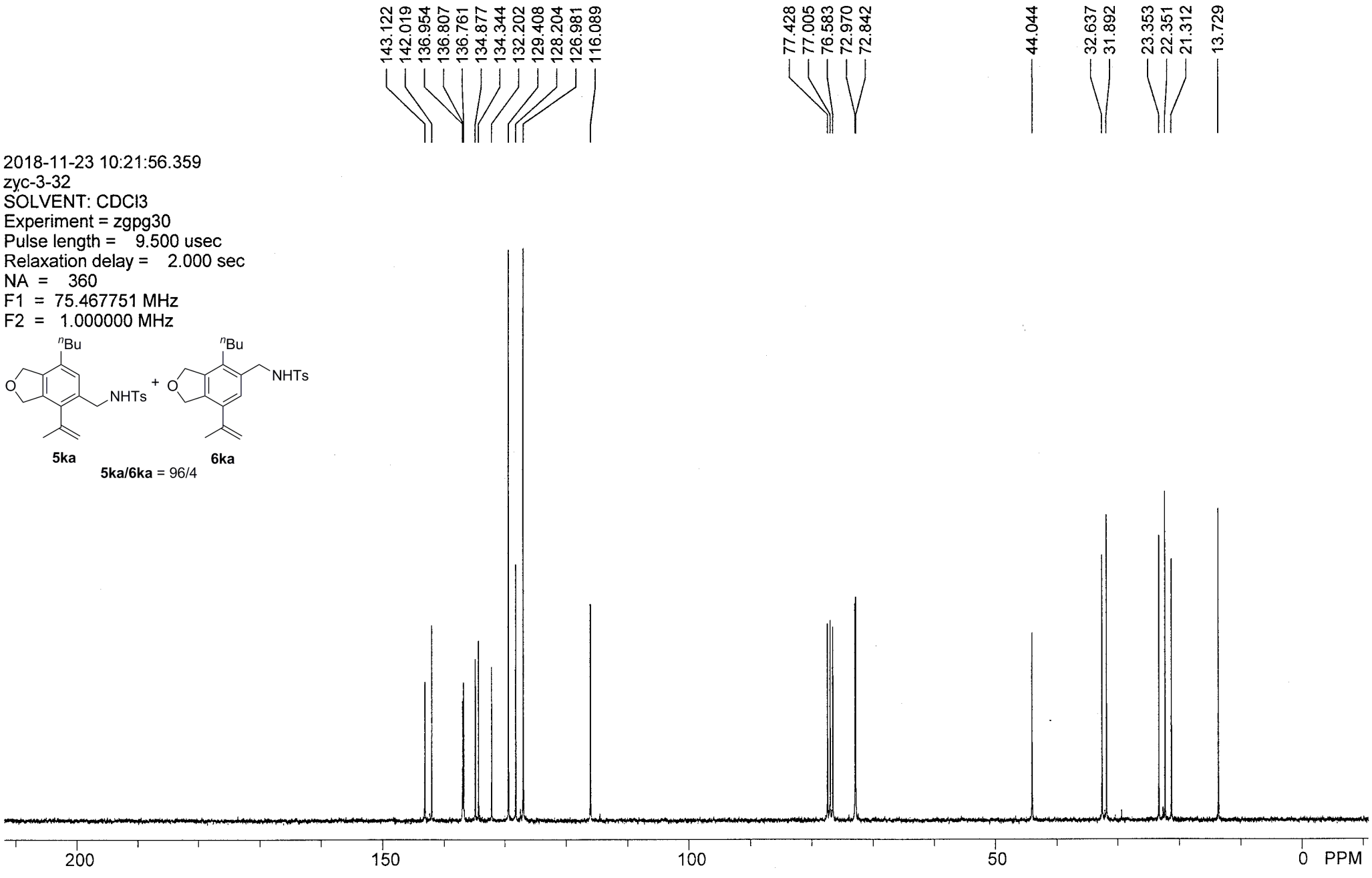


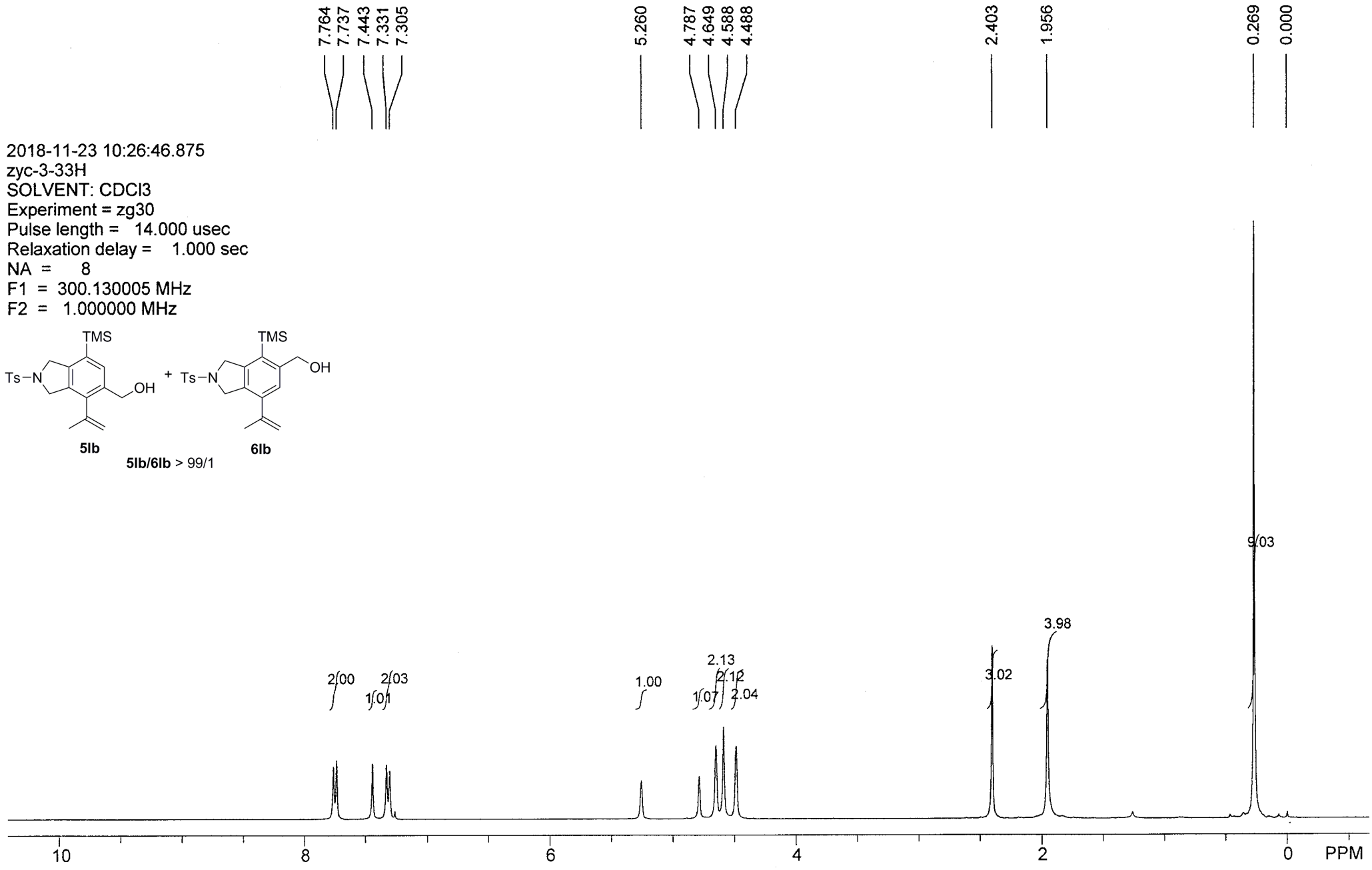
5fg

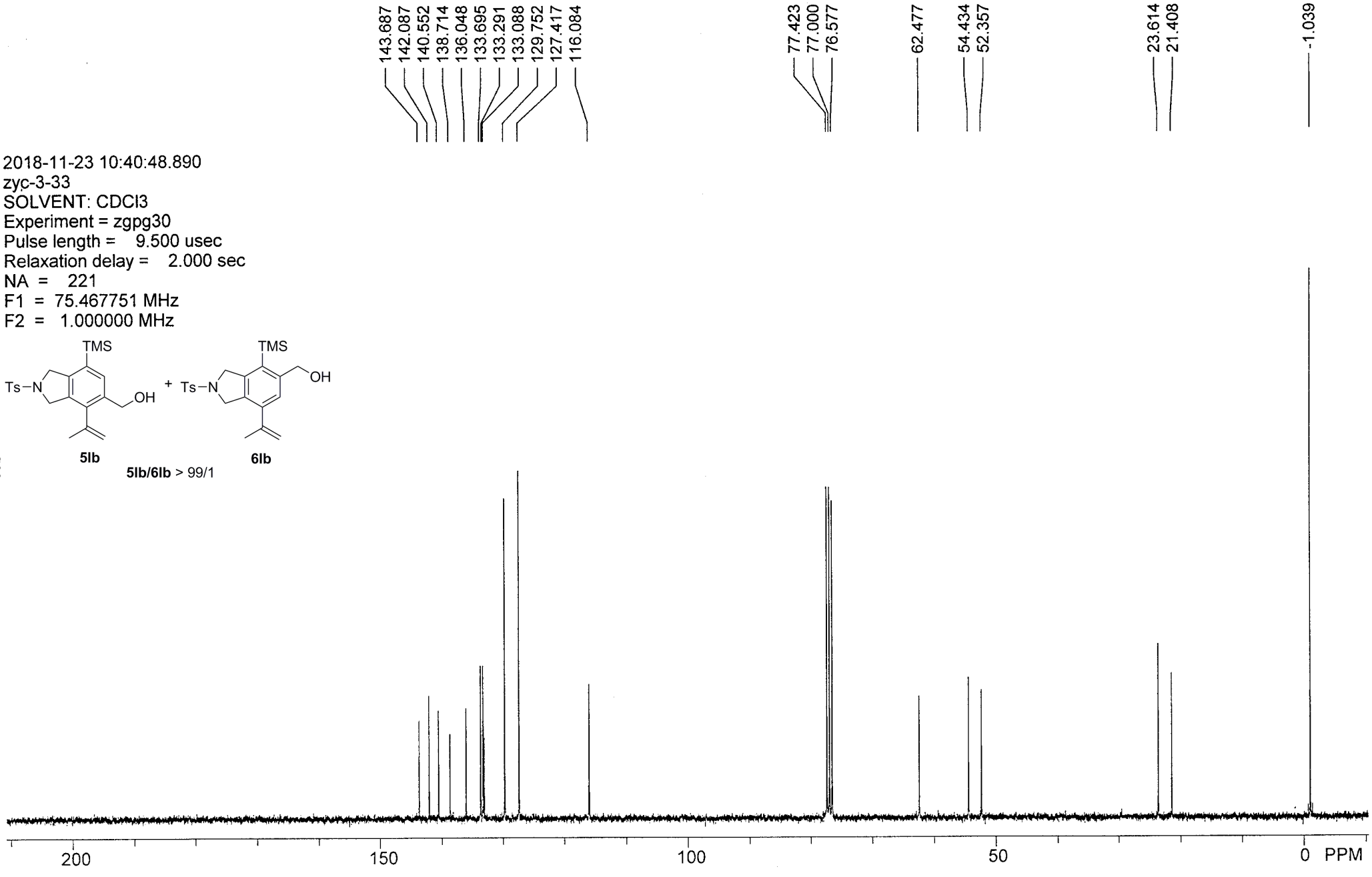

7.742
7.714
7.599
7.572
7.364
7.351
7.338
7.265
7.253
7.226
7.136
7.122
7.032
7.024
6.864
6.849
5.306
5.287
5.267
5.168
4.977
4.917
4.772
4.134
4.115
4.028
4.009
2.387
2.034
1.861
0.000
2018-01-30 21:59:35.921
zyc-1-131re
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
NHTs
NHTs
5ga
6ga
5ga/6ga = 96/4
2.01
0.10
2.08
1.04
1.02
.98
0.05
0.05
3.08
1.00
2.00
1.07
0.08
2.00
0.09
3.08
0.16
3.04
10
8
6
4
2
PPM

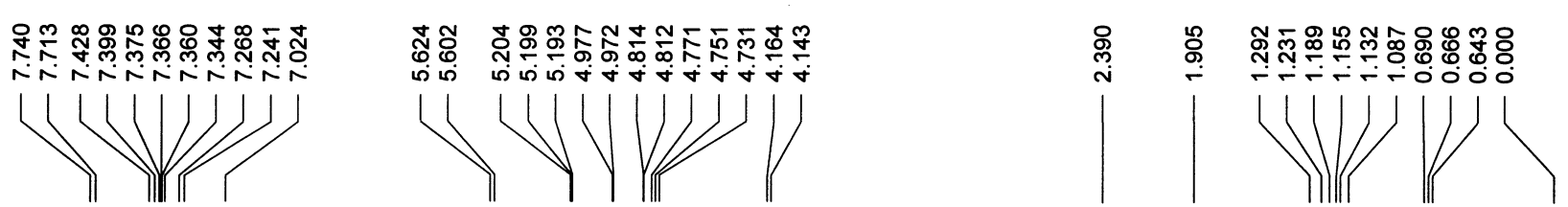

143.411
141.802
139.743
138.172
136.701
135.883
135.690
132.813
129.568
129.026
127.527
127.040
126.728
126.057
121.893
116.516
77.432
77.000
76.577
74.059
72.854
44.084
23.384
21.435
2018-01-31 09:03:21.640
zyc-1-131re
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 417
F1 = 75.467751 MHz
F2 = 1.000000 MHz
S
O
NHTs
+
5ga
6ga
5ga/6ga = 96/4
200
150
100
50
0
PPM


7.747
7.719
7.287
7.261
6.924
5.389
5.372
5.354
5.108
5.020
4.915
4.717
4.071
4.051
2.423
2.399
2.373
2.347
1.822
1.478
1.471
1.452
1.427
1.344
1.320
1.296
1.272
0.924
0.900
0.876
-0.000
2018-11-23 09:59:27.687
zyc-3-32
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
nBu
NHTs
5ka
6ka
5ka/6ka = 96/4
S96
2.00
2.02
0.94
0.04
0.99
1.06
2.10
1.91
0.98
0.05
2.00
4.99
0.11
2.91
2.00
2.20
3.01
10
8
6
4
2
0 PPM

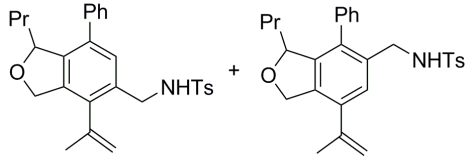

143.122
142.019
136.954
136.807
136.761
134.877
134.344
132.202
129.408
128.204
126.981
116.089
77.428
77.005
76.583
72.970
72.842
44.044
32.637
31.892
23.353
22.351
21.312
13.729
2018-11-23 10:21:56.359
zyc-3-32
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 360
F1 = 75.467751 MHz
F2 = 1.000000 MHz
nBu
NHTs
O
+
5ka
6ka
5ka/6ka = 96/4
200
150
100
50
0
PPM


7.764
7.737
7.443
7.331
7.305
5.260
4.787
4.649
4.588
4.488
2.403
1.956
0.269
0.000
2018-11-23 10:26:46.875
zyc-3-33H
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
TMS
Ts–N
OH
+
Ts–N
TMS
OH
5Ib
6Ib
5Ib/6Ib > 99/1
2.00
2.03
1.01
1.00
2.13
2.12
1.07
2.04
3.02
3.98
9.03
10
8
6
4
2
0
PPM


143.687
142.087
140.552
138.714
136.048
133.695
133.291
133.088
129.752
127.417
116.084
77.423
77.000
76.577
62.477
54.434
52.357
23.614
21.408
-1.039
2018-11-23 10:40:48.890
zyc-3-33
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 221
F1 = 75.467751 MHz
F2 = 1.000000 MHz
TMS
Ts–N
OH
+
Ts–N
TMS
OH
5Ib
6Ib
5Ib/6Ib > 99/1
S99
200
150
100
50
0 PPM

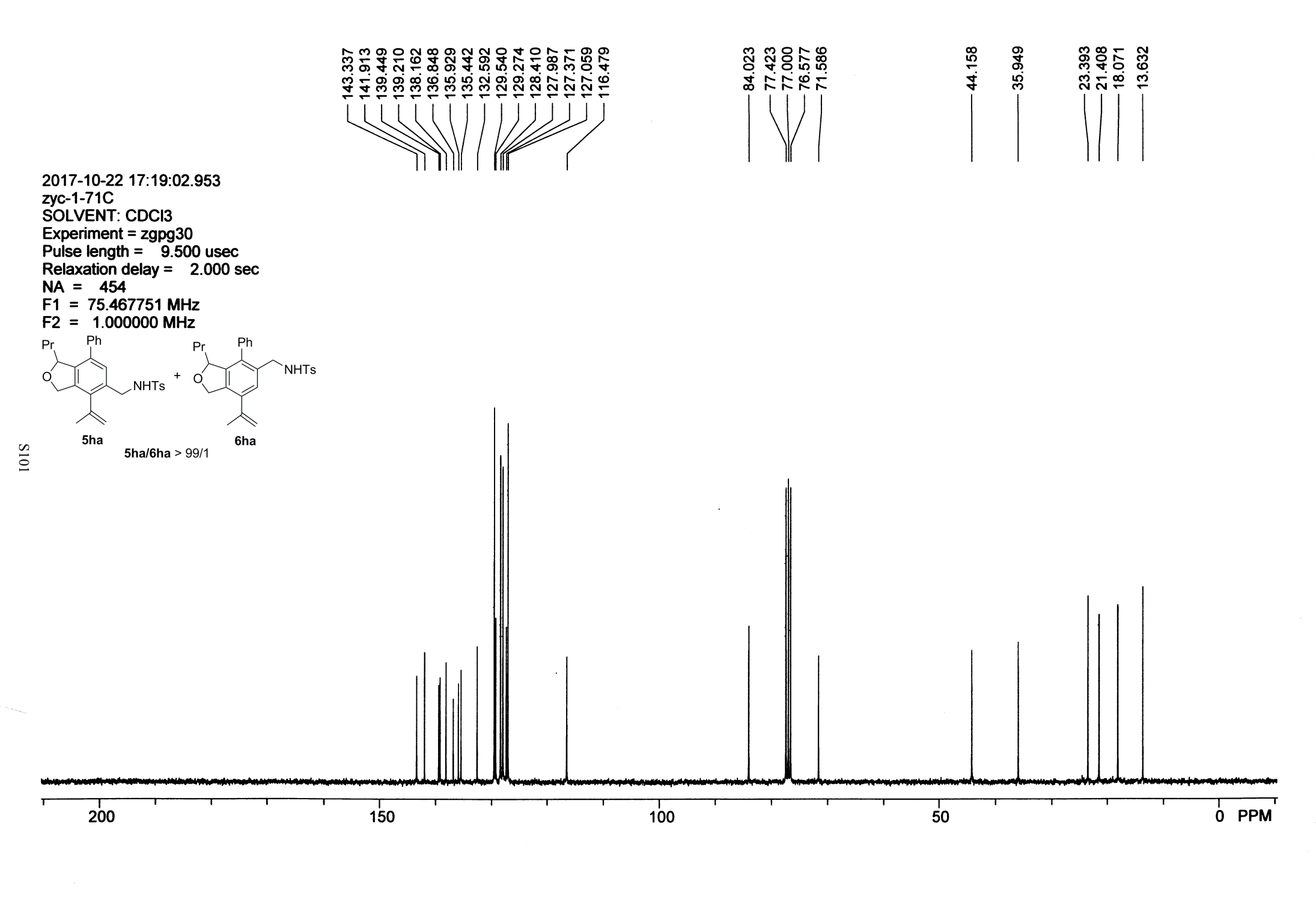


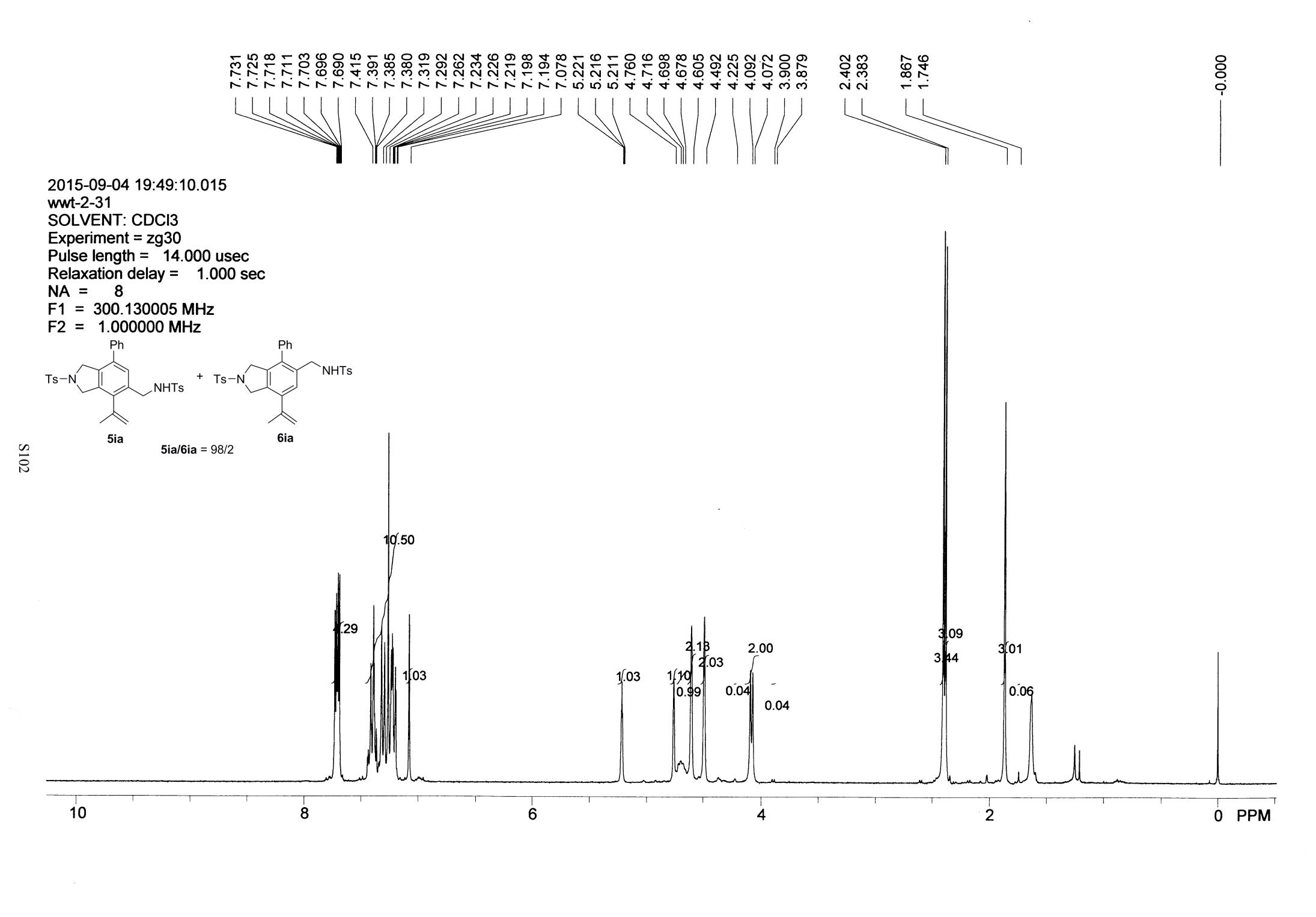
2017-10-22 13:44:42.125
zyc-1-71H
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz

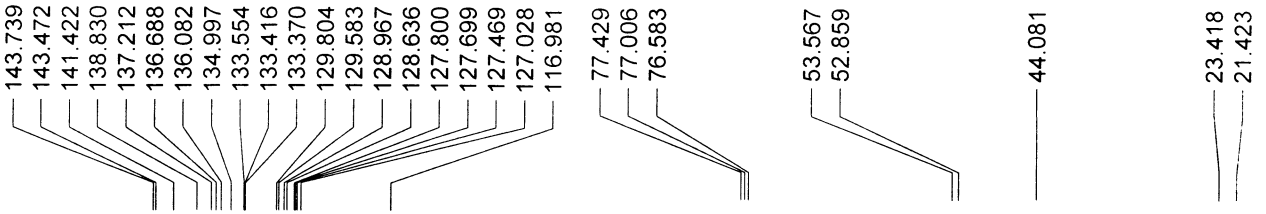


5ha **6ha**

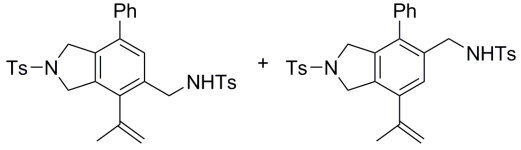
5ha/6ha > 99/1


143.337
141.913
139.449
139.210
138.162
136.848
135.929
135.442
132.592
129.540
129.274
128.410
127.987
127.371
127.059
116.479
84.023
77.423
77.000
76.577
71.586
44.158
35.949
23.393
21.408
18.071
13.632
2017-10-22 17:19:02.953
zyc-1-71C
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 454
F1 = 75.467751 MHz
F2 = 1.000000 MHz
Pr
Ph
O
NHTs
+
5ha
6ha
5ha/6ha > 99/1
200
150
100
50
0 PPM


7.731
7.725
7.718
7.711
7.703
7.696
7.690
7.415
7.391
7.385
7.380
7.319
7.292
7.262
7.234
7.226
7.219
7.198
7.194
7.078
5.221
5.216
5.211
4.760
4.716
4.698
4.678
4.605
4.492
4.225
4.092
4.072
3.900
3.879
2.402
2.383
1.867
1.746
-0.000
2015-09-04 19:49:10.015
wwt-2-31
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
Ph
Ts–N
NHTs
5ia
+
6ia
5ia/6ia = 98/2
10.50
4.29
1.03
1.03
1.10
0.99
2.13
2.03
2.00
0.04
0.04
3.09
3.44
3.01
0.06
10
8
6
4
2
0 PPM
S102



2015-09-03 15:00:16.937
wwt-2-31C
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 196
F1 = 75.467751 MHz
F2 = 1.000000 MHz

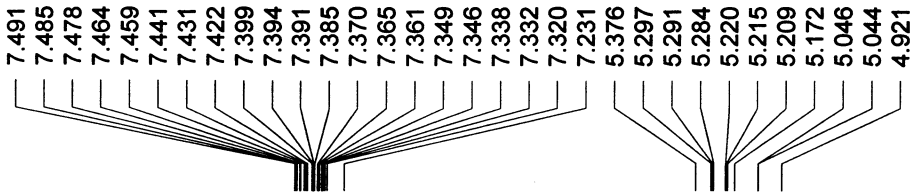


5ia

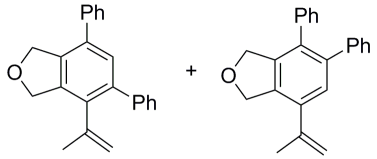
+

6ia

5ia/6ia = 98/2

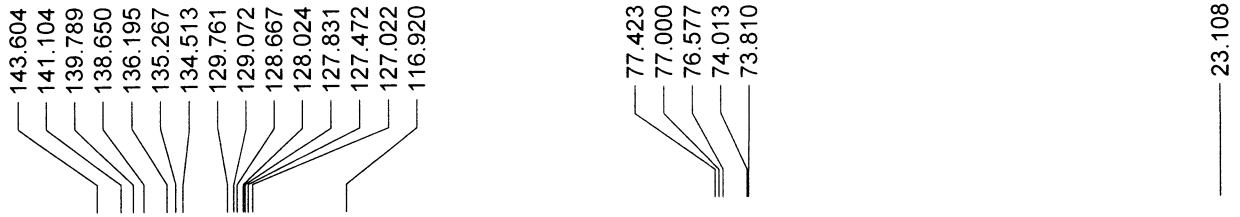


2017-12-24 09:39:48.484
zyc-1-113
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz

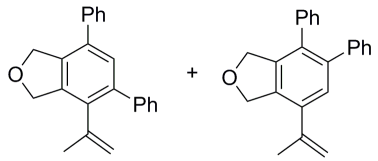


5ah **6ah**

5ah/6ah = 96/4



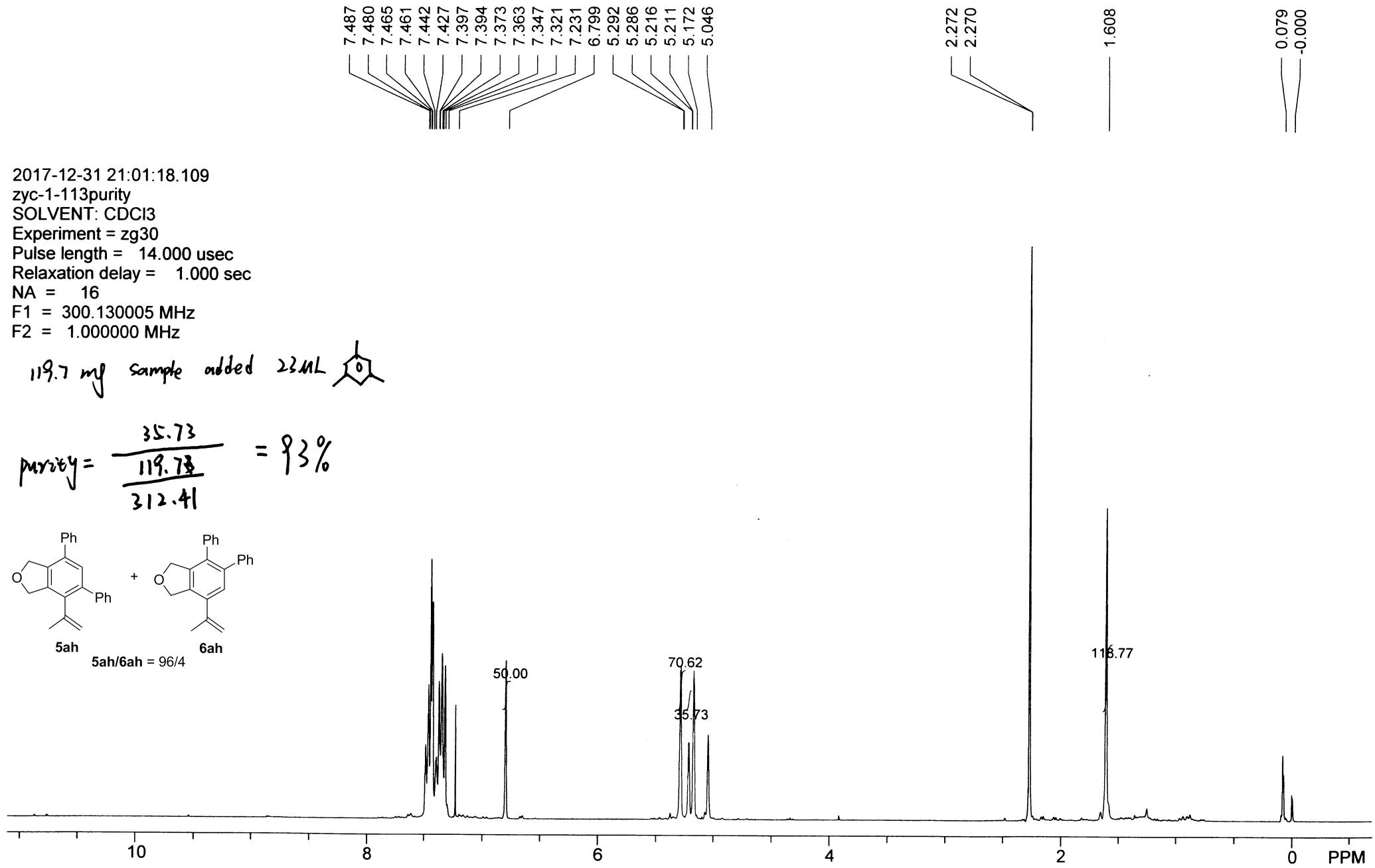
2017-12-24 12:17:19.265
zyc-1-113c
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 789
F1 = 75.467751 MHz
F2 = 1.000000 MHz

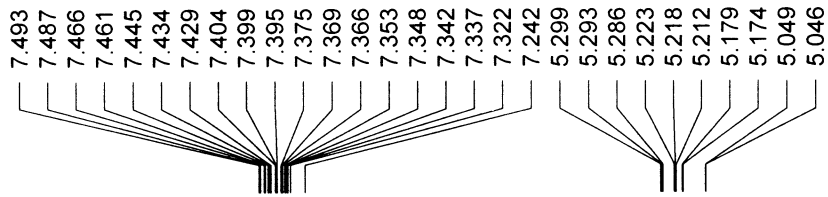


5ah **6ah**

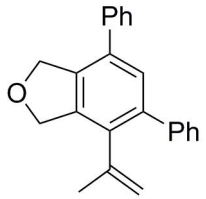
5ah/6ah = 96/4

S105

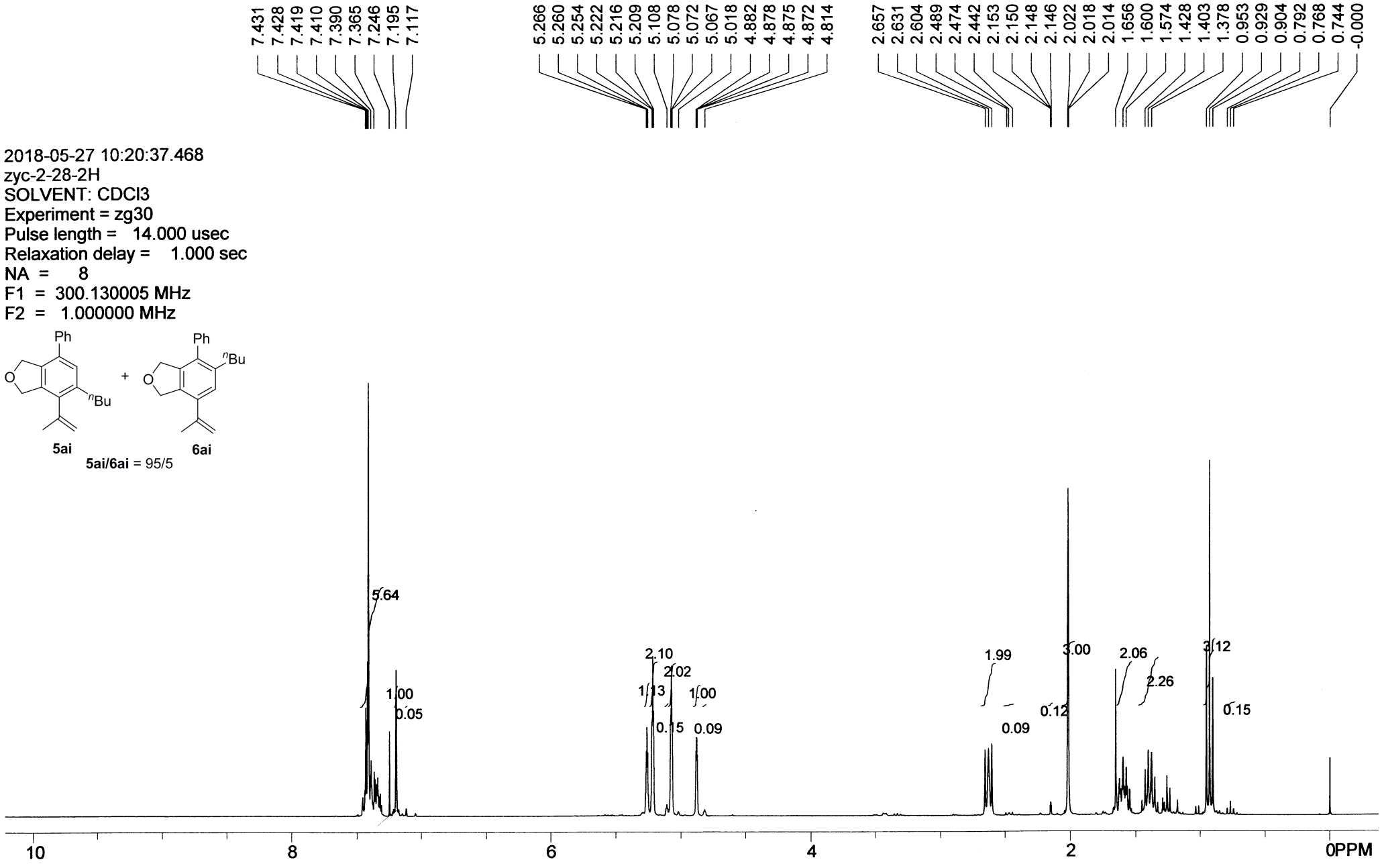

7.487
7.480
7.465
7.461
7.442
7.427
7.397
7.394
7.373
7.363
7.347
7.321
7.231
6.799
5.292
5.286
5.216
5.211
5.172
5.046
2.272
2.270
1.608
0.079
-0.000
2017-12-31 21:01:18.109
zyc-1-113purity
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 16
F1 = 300.130005 MHz
F2 = 1.000000 MHz
119.7 mg sample added 23mL
purity = 35.73 / 119.78 / 312.41 = 93%
Ph
Ph
O
5ah
Ph
Ph
O
6ah
5ah/6ah = 96/4
50.00
70.62
35.73
116.77
10
8
6
4
2
0
PPM

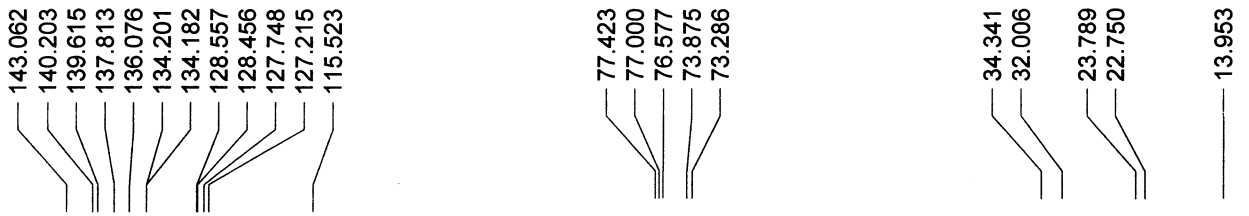


2018-01-02 21:39:50.656
zyc-1-113re
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz

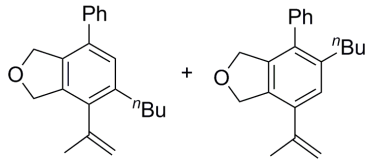


5ah


7.431
7.428
7.419
7.410
7.390
7.365
7.246
7.195
7.117
5.266
5.260
5.254
5.222
5.216
5.209
5.108
5.078
5.072
5.067
5.018
4.882
4.878
4.875
4.872
4.814
2.657
2.631
2.604
2.489
2.474
2.442
2.153
2.150
2.148
2.146
2.022
2.018
2.014
1.656
1.600
1.574
1.428
1.403
1.378
0.953
0.929
0.904
0.792
0.768
0.744
-0.000
2018-05-27 10:20:37.468
zyc-2-28-2H
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
Ph
nBu
5ai
6ai
5ai/6ai = 95/5
5.64
1.00
0.05
2.10
2.02
1.13
1.00
0.15
0.09
1.99
0.09
0.12
3.00
2.06
2.26
3.12
0.15
10
8
6
4
2
0PPM

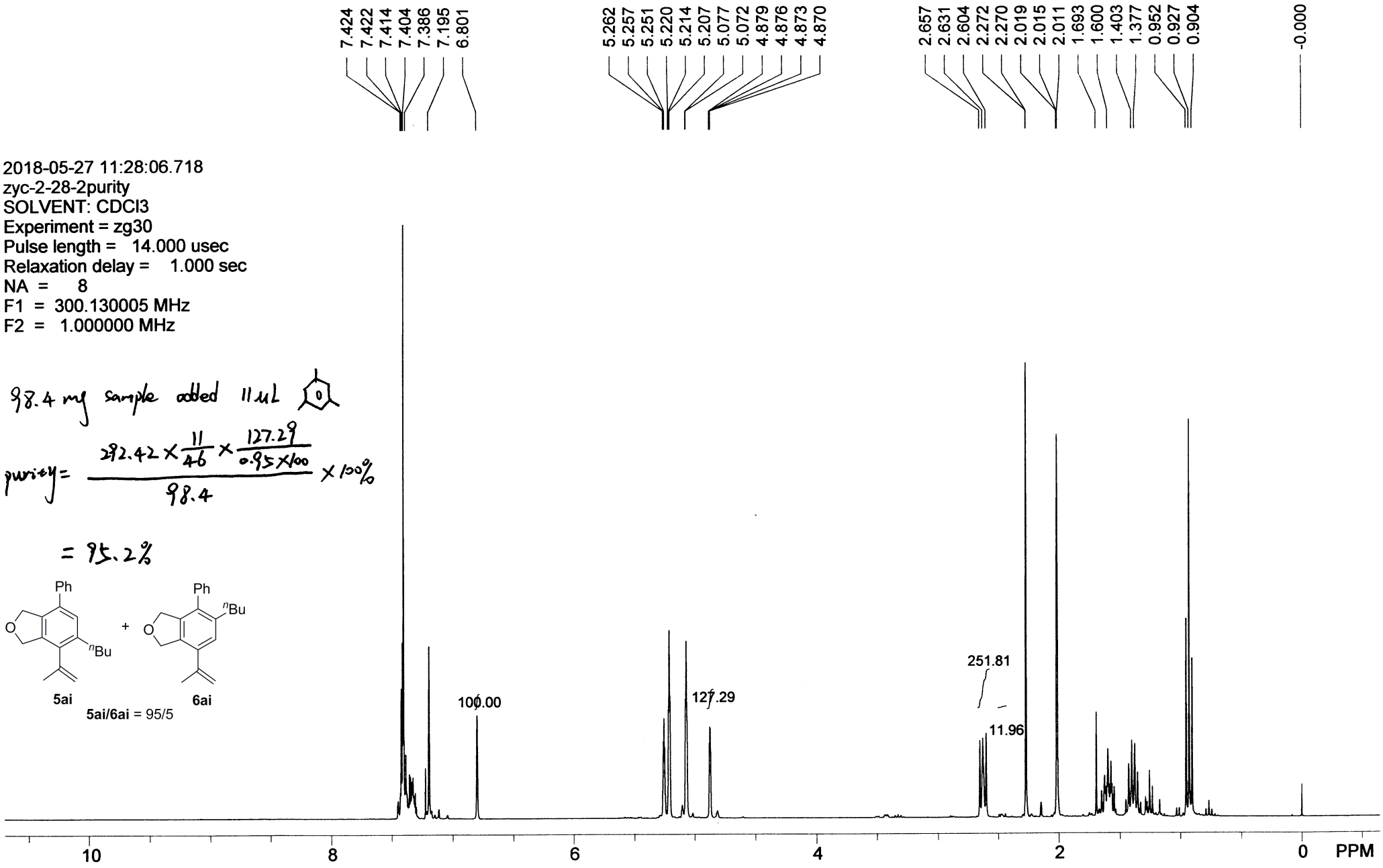


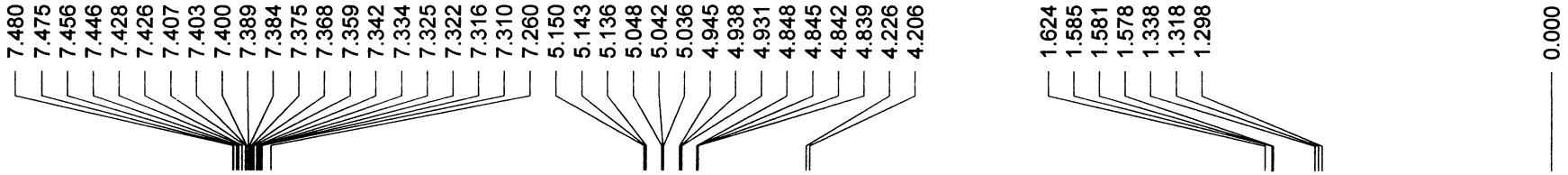
2018-05-25 21:59:06.421
zyc-2-28-2
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 102
F1 = 75.467751 MHz
F2 = 1.000000 MHz



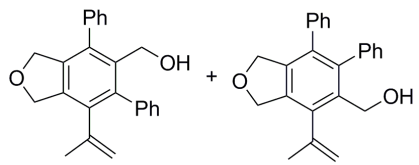
5ai **6ai**

5ai/6ai = 95/5


7.424
7.422
7.414
7.404
7.386
7.195
6.801
5.262
5.257
5.251
5.220
5.214
5.207
5.077
5.072
4.879
4.876
4.873
4.870
2.657
2.631
2.604
2.272
2.270
2.019
2.015
2.011
1.693
1.600
1.403
1.377
0.952
0.927
0.904
-0.000
2018-05-27 11:28:06.718
zyc-2-28-2purity
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
98.4 mg sample added 11 uL
purity = 292.42 × 11/46 × 127.29/(0.95×100) × 100% / 98.4
= 95.2%
Ph
nBu
5ai
+
Ph
nBu
6ai
5ai/6ai = 95/5
100.00
127.29
251.81
11.96
10
8
6
4
2
0
PPM

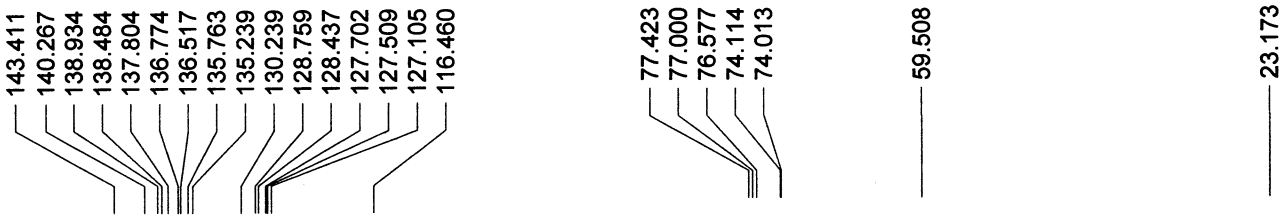


2018-06-01 09:34:08.187
zyc-2-35
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz

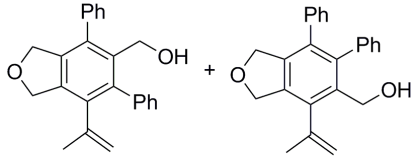


5aj **6aj**

5aj/6aj > 99/1

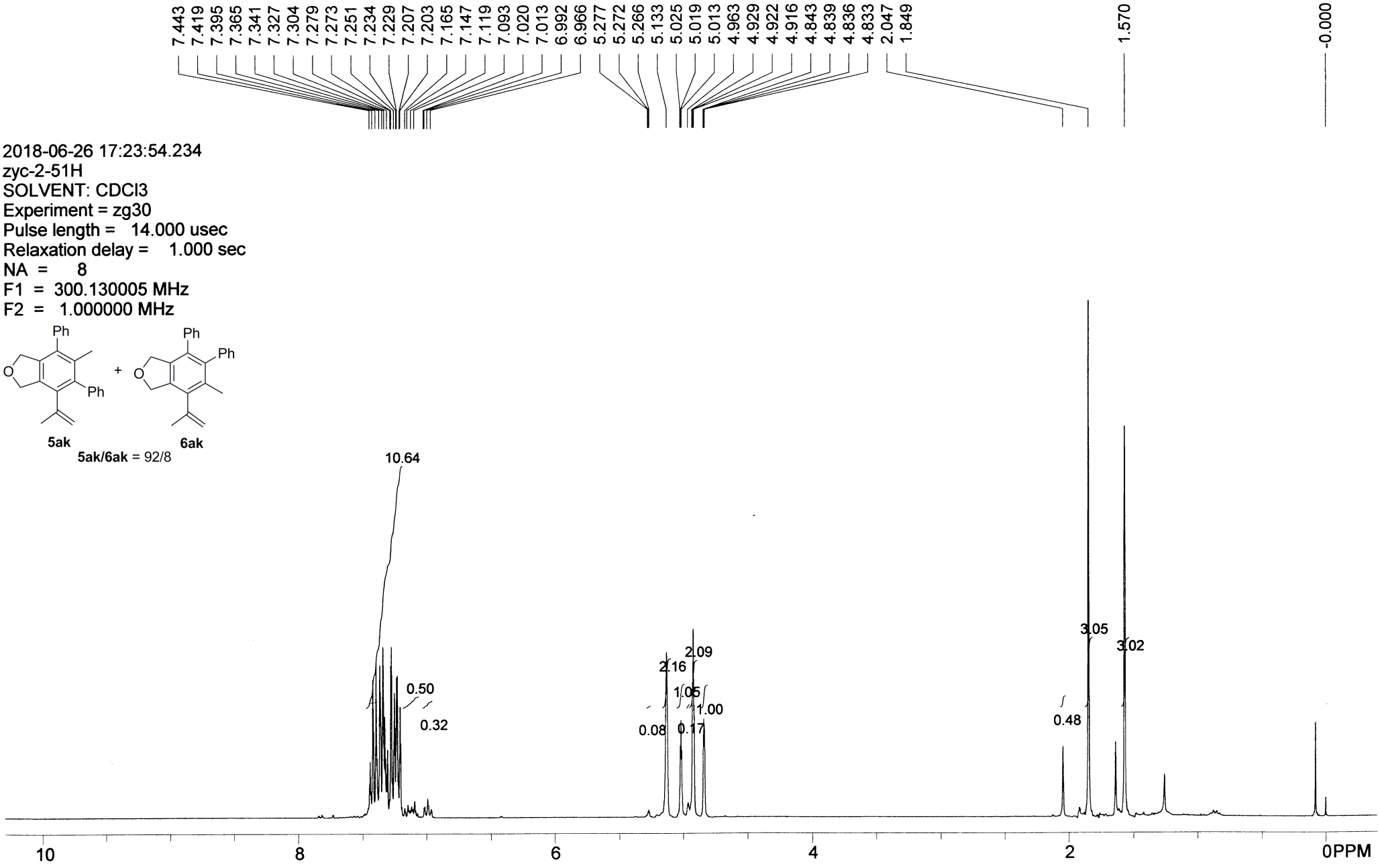


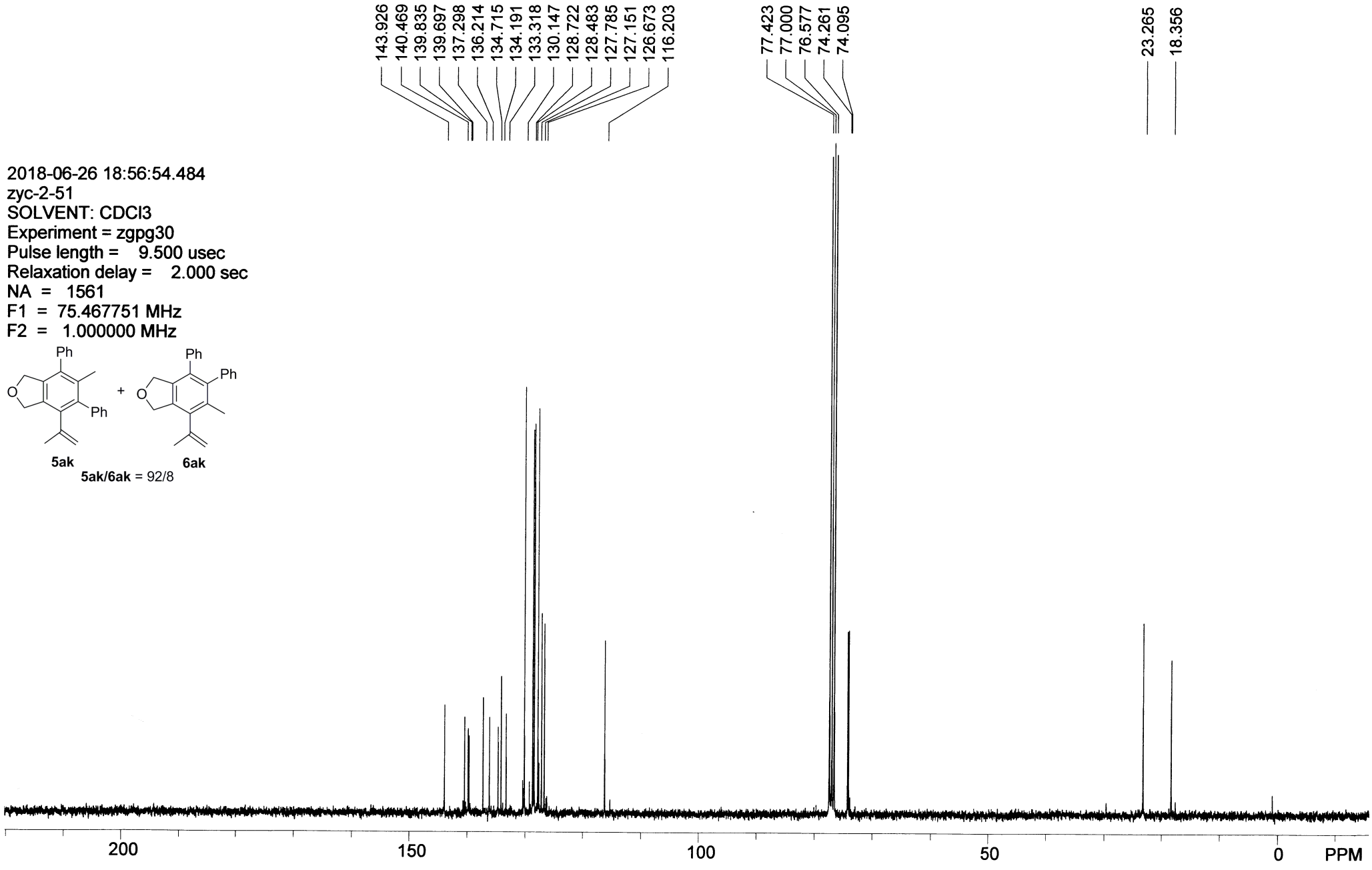
2018-05-29 21:41:41.187
zyc-2-35
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 43
F1 = 75.467751 MHz
F2 = 1.000000 MHz

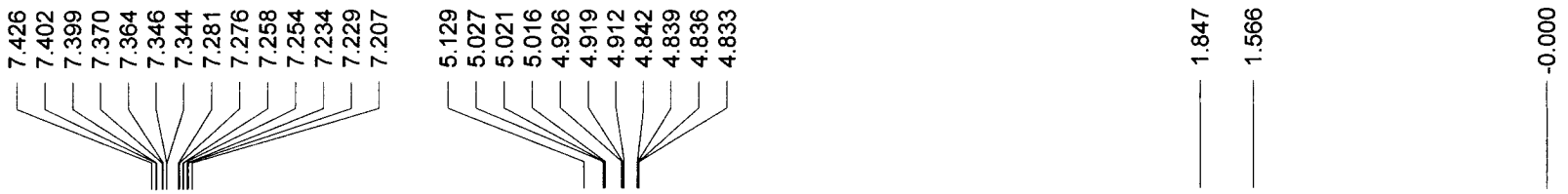


5aj **6aj**

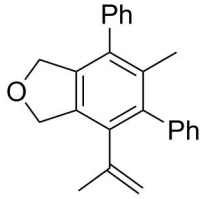
5aj/6aj > 99/1


7.443
7.419
7.395
7.365
7.341
7.327
7.304
7.279
7.273
7.251
7.234
7.229
7.207
7.203
7.165
7.147
7.119
7.093
7.020
7.013
6.992
6.966
5.277
5.272
5.266
5.133
5.025
5.019
5.013
4.963
4.929
4.922
4.916
4.843
4.839
4.836
4.833
2.047
1.849
1.570
-0.000
2018-06-26 17:23:54.234
zyc-2-51H
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
Ph
Ph
O
+
Ph
Ph
O
5ak
6ak
5ak/6ak = 92/8
10.64
0.50
0.32
0.08
2.16
2.09
1.05
1.00
0.17
0.48
3.05
3.02
10
8
6
4
2
0PPM

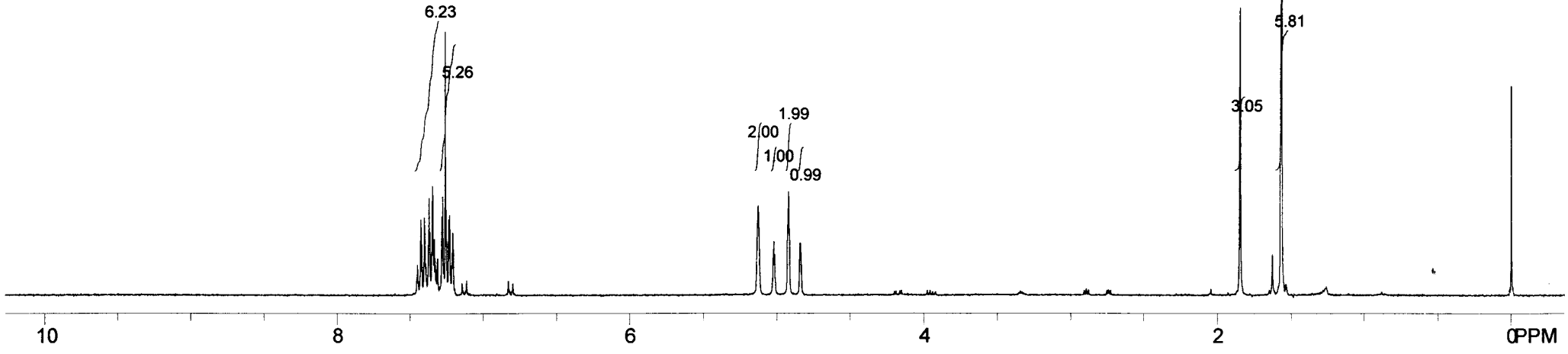

143.926
140.469
139.835
139.697
137.298
136.214
134.715
134.191
133.318
130.147
128.722
128.483
127.785
127.151
126.673
116.203
77.423
77.000
76.577
74.261
74.095
23.265
18.356
2018-06-26 18:56:54.484
zyc-2-51
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 1561
F1 = 75.467751 MHz
F2 = 1.000000 MHz
Ph
Ph
O
+
Ph
Ph
O
5ak
6ak
5ak/6ak = 92/8
200
150
100
50
0
PPM

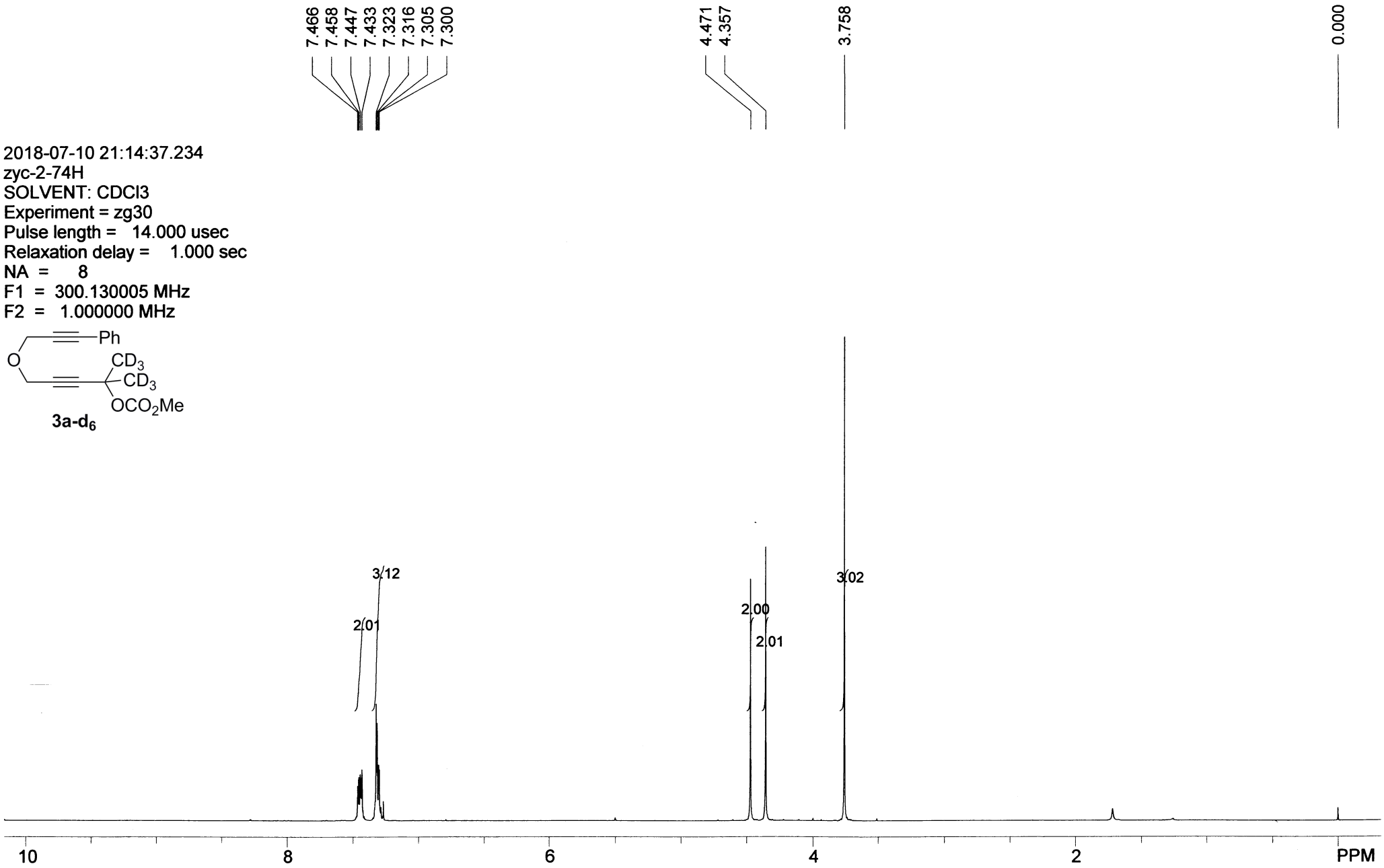


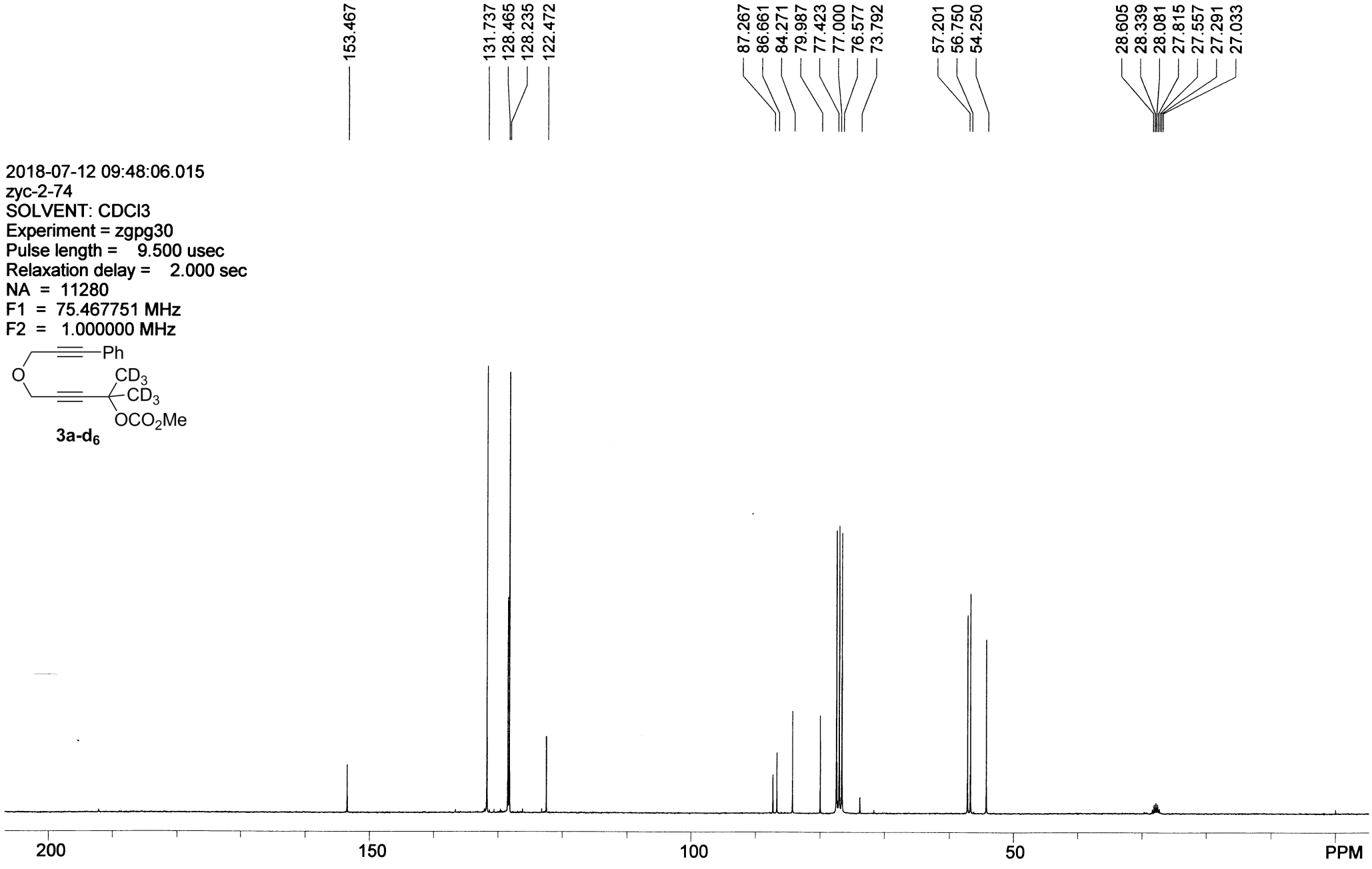
2018-07-10 16:39:25.046
zyc-2-60crystal
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz

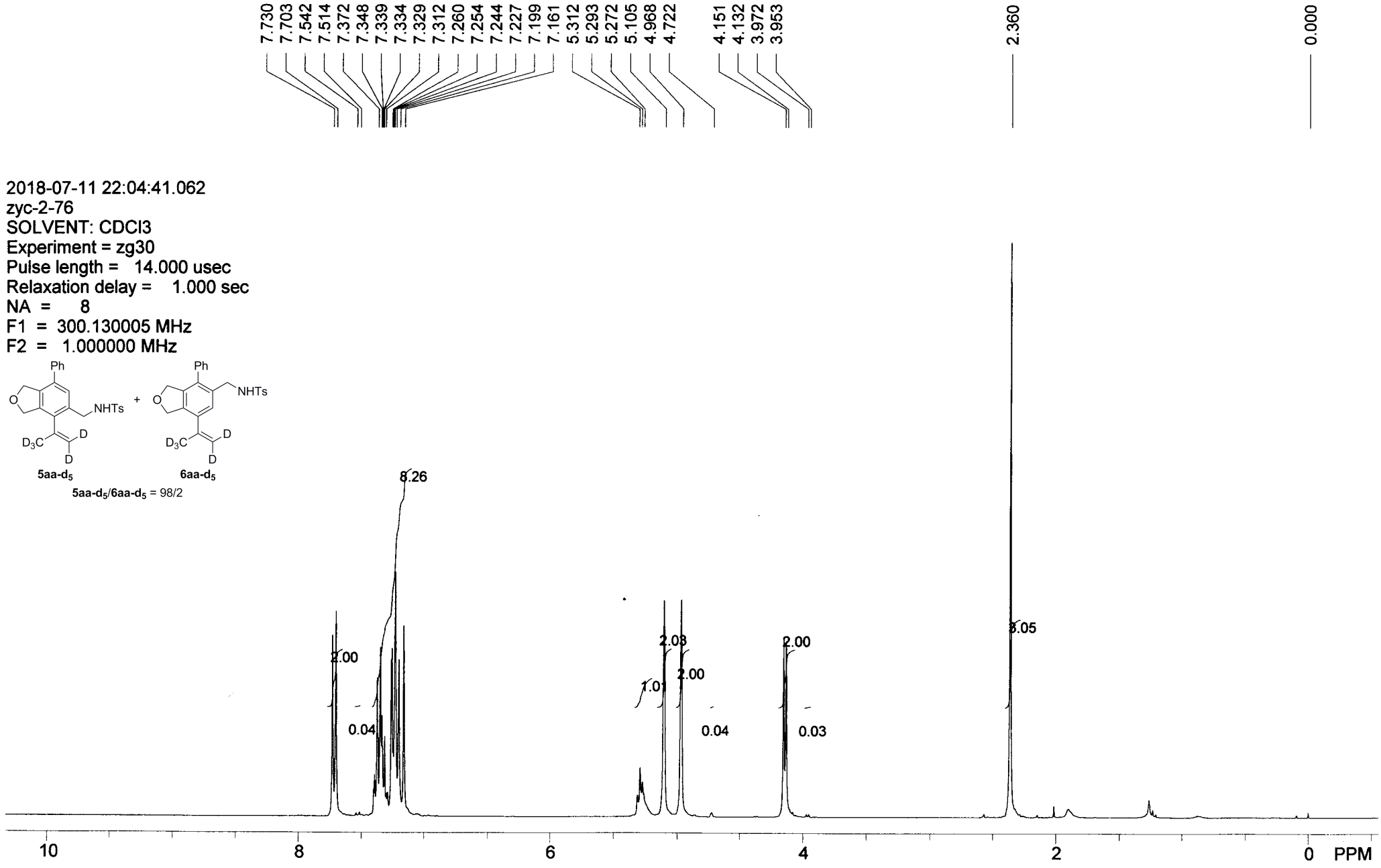


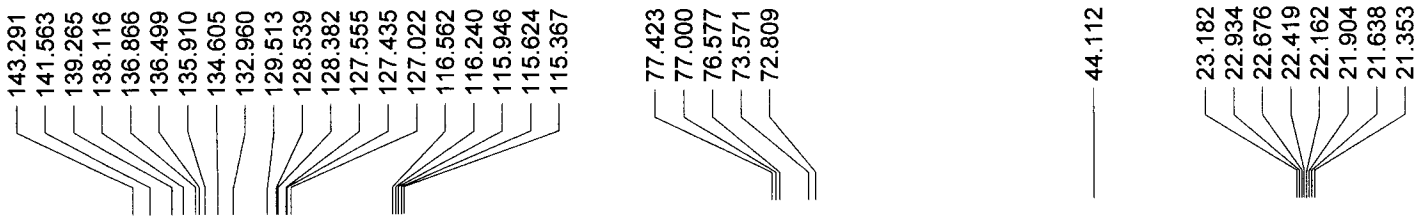
5ak



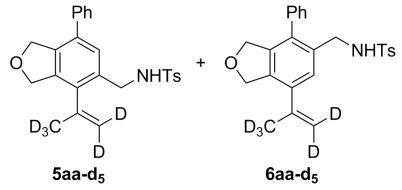

7.466
7.458
7.447
7.433
7.323
7.316
7.305
7.300
4.471
4.357
3.758
0.000
2018-07-10 21:14:37.234
zyc-2-74H
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
Ph
O
CD3
CD3
OCO2Me
3a-d6
3.12
2.01
2.00
2.01
3.02
10
8
6
4
2
PPM


153.467
131.737
128.465
128.235
122.472
87.267
86.661
84.271
79.987
77.423
77.000
76.577
73.792
57.201
56.750
54.250
28.605
28.339
28.081
27.815
27.557
27.291
27.033
2018-07-12 09:48:06.015
zyc-2-74
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 11280
F1 = 75.467751 MHz
F2 = 1.000000 MHz
Ph
O
CD3
CD3
OCO2Me
3a-d6
200
150
100
50
PPM

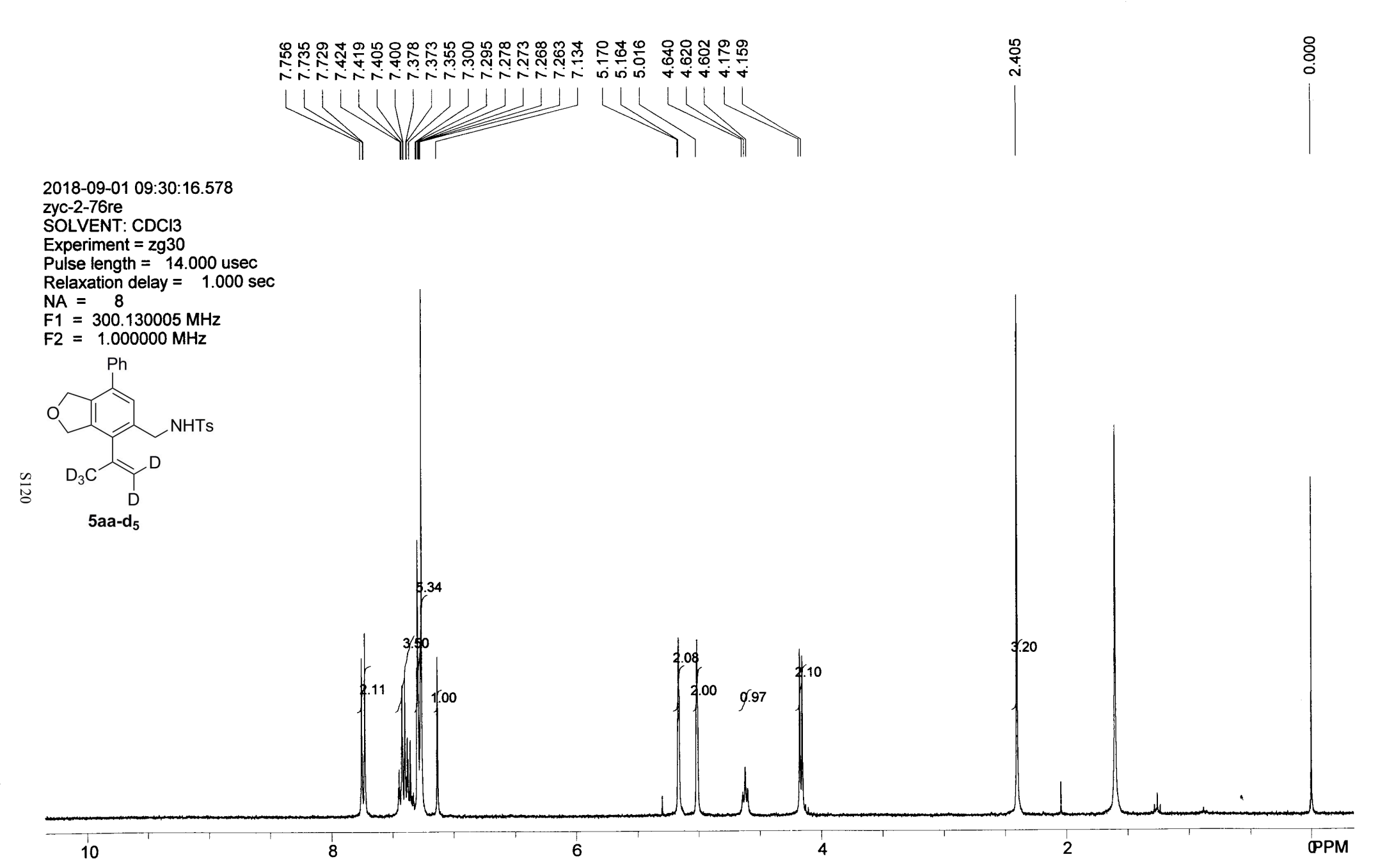

7.730
7.703
7.542
7.514
7.372
7.348
7.339
7.334
7.329
7.312
7.260
7.254
7.244
7.227
7.199
7.161
5.312
5.293
5.272
5.105
4.968
4.722
4.151
4.132
3.972
3.953
2.360
0.000
2018-07-11 22:04:41.062
zyc-2-76
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
Ph
NHTs
D3C
D
D
5aa-d5
6aa-d5
5aa-d5/6aa-d5 = 98/2
8.26
2.00
0.04
2.03
1.01
2.00
0.04
2.00
0.03
3.05
10
8
6
4
2
0
PPM

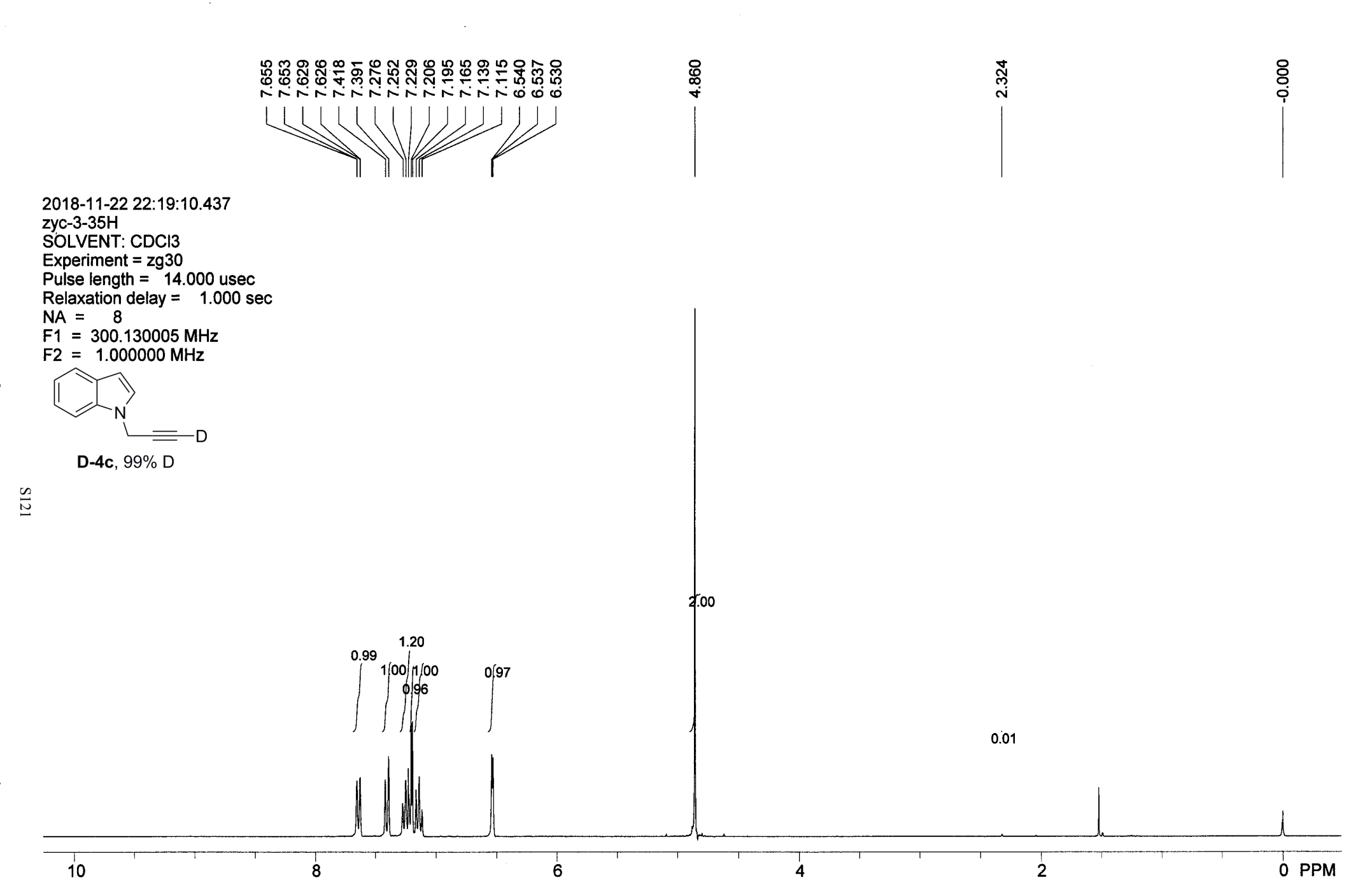


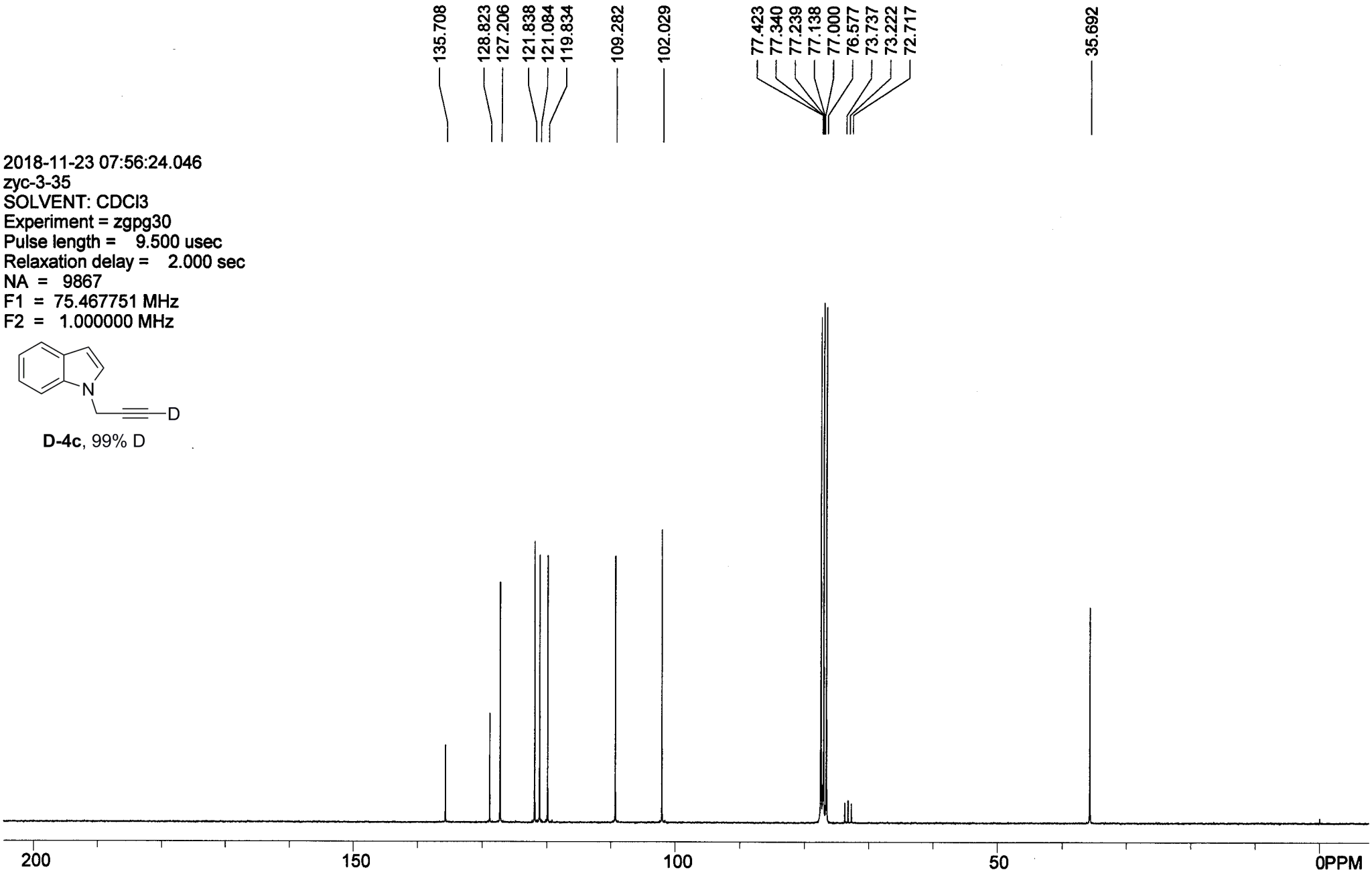
2018-07-13 09:14:55.328
zyc-2-76C
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 11005
F1 = 75.467751 MHz
F2 = 1.000000 MHz



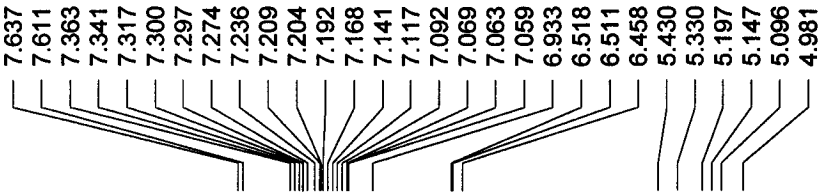
5aa-d$_5$/6aa-d$_5$ = 98/2


7.756
7.735
7.729
7.424
7.419
7.405
7.400
7.378
7.373
7.355
7.300
7.295
7.278
7.273
7.268
7.263
7.134
5.170
5.164
5.016
4.640
4.620
4.602
4.179
4.159
2.405
0.000
2018-09-01 09:30:16.578
zyc-2-76re
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
Ph
O
NHTs
D
D3C
D
5aa-d5
5.34
3.50
2.11
1.00
2.08
2.00
0.97
2.10
3.20
10
8
6
4
2
0
PPM

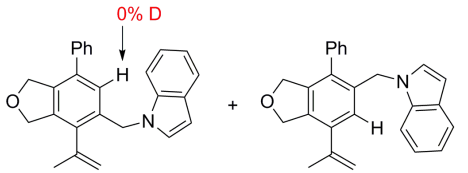

7.655
7.653
7.629
7.626
7.418
7.391
7.276
7.252
7.229
7.206
7.195
7.165
7.139
7.115
6.540
6.537
6.530
4.860
2.324
-0.000
2018-11-22 22:19:10.437
zyc-3-35H
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
N
D
D-4c, 99% D
0.99
1.00
1.20
1.00
0.96
0.97
2.00
0.01
10
8
6
4
2
0 PPM


135.708
128.823
127.206
121.838
121.084
119.834
109.282
102.029
77.423
77.340
77.239
77.138
77.000
76.577
73.737
73.222
72.717
35.692
2018-11-23 07:56:24.046
zyc-3-35
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 9867
F1 = 75.467751 MHz
F2 = 1.000000 MHz
N
D
200
150
100
50
0PPM

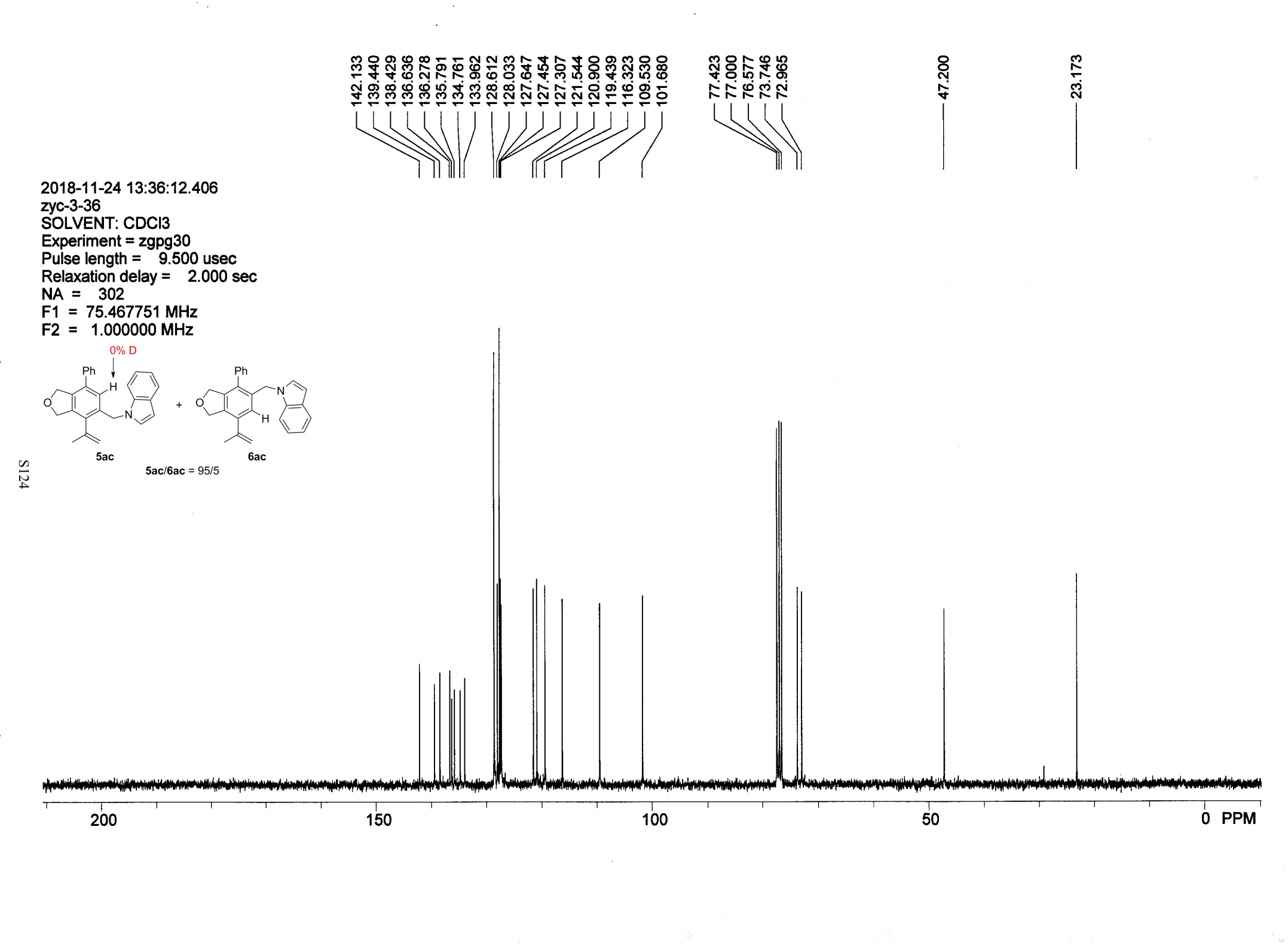
D-4c, 99% D

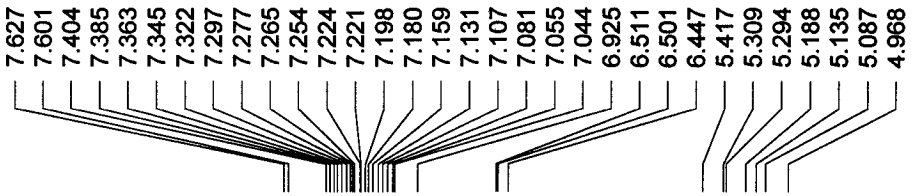


2018-11-24 13:15:56.671
zyc-3-36H
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz

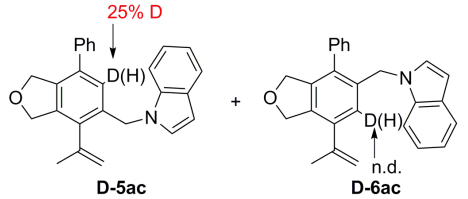


5ac/6ac = 95/5

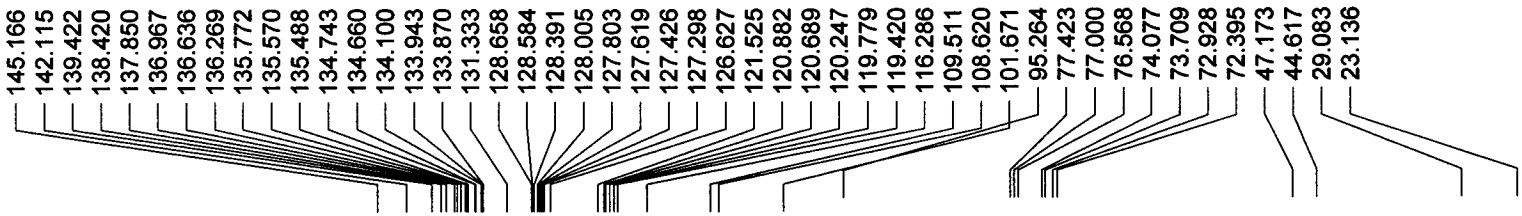

142.133
139.440
138.429
136.636
136.278
135.791
134.761
133.962
128.612
128.033
127.647
127.454
127.307
121.544
120.900
119.439
116.323
109.530
101.680
77.423
77.000
76.577
73.746
72.965
47.200
23.173
2018-11-24 13:36:12.406
zyc-3-36
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 302
F1 = 75.467751 MHz
F2 = 1.000000 MHz
0% D
Ph
H
O
N
5ac
+
Ph
N
O
H
6ac
5ac/6ac = 95/5
200
150
100
50
0 PPM



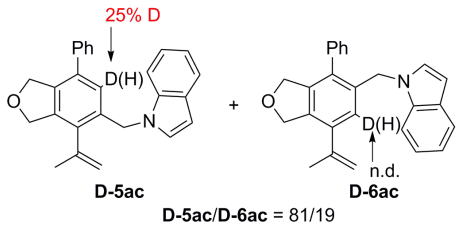
2018-11-27 22:22:54.078
zyc-3-46
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz



D-5ac/D-6ac = 81/19

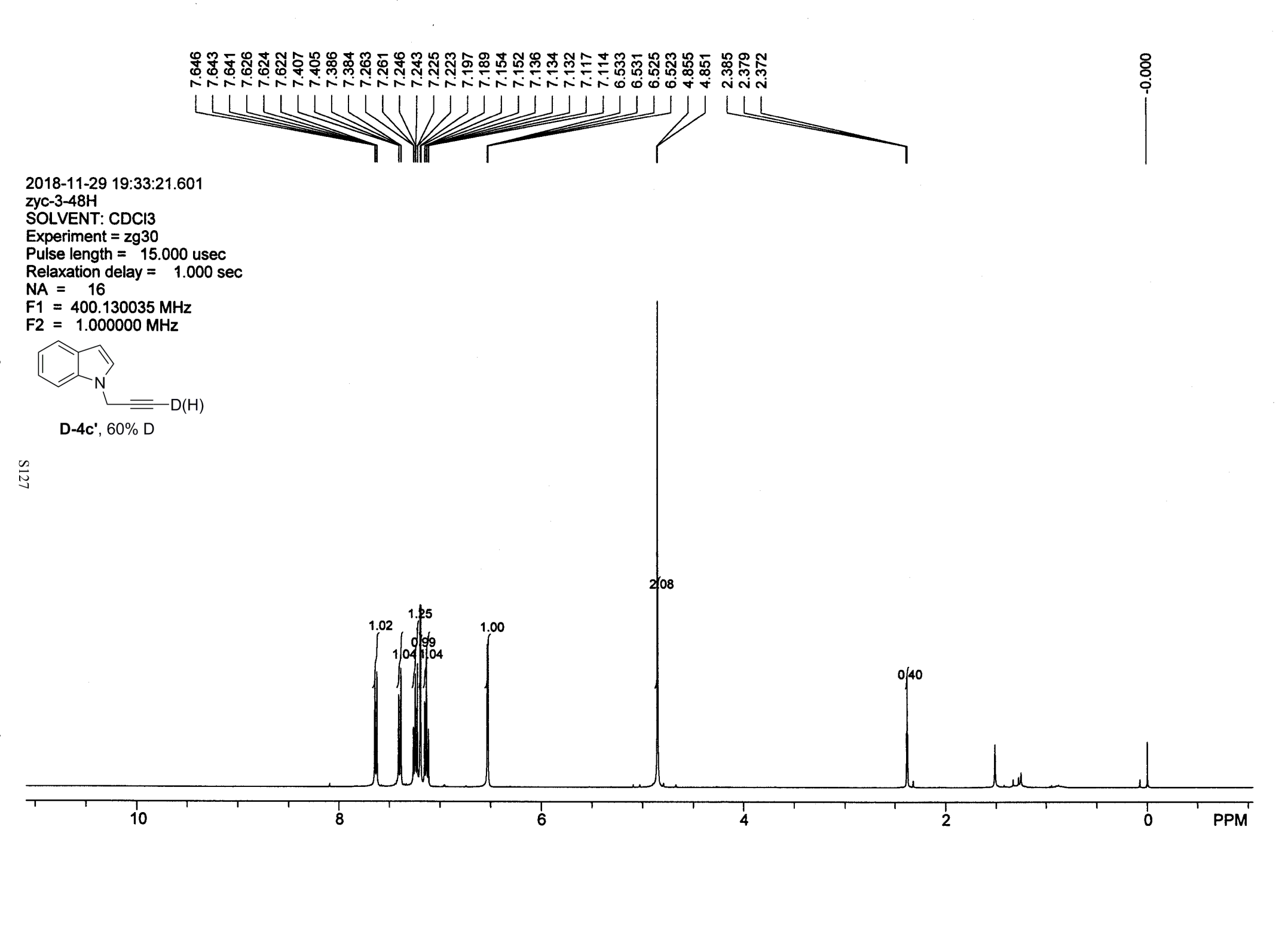


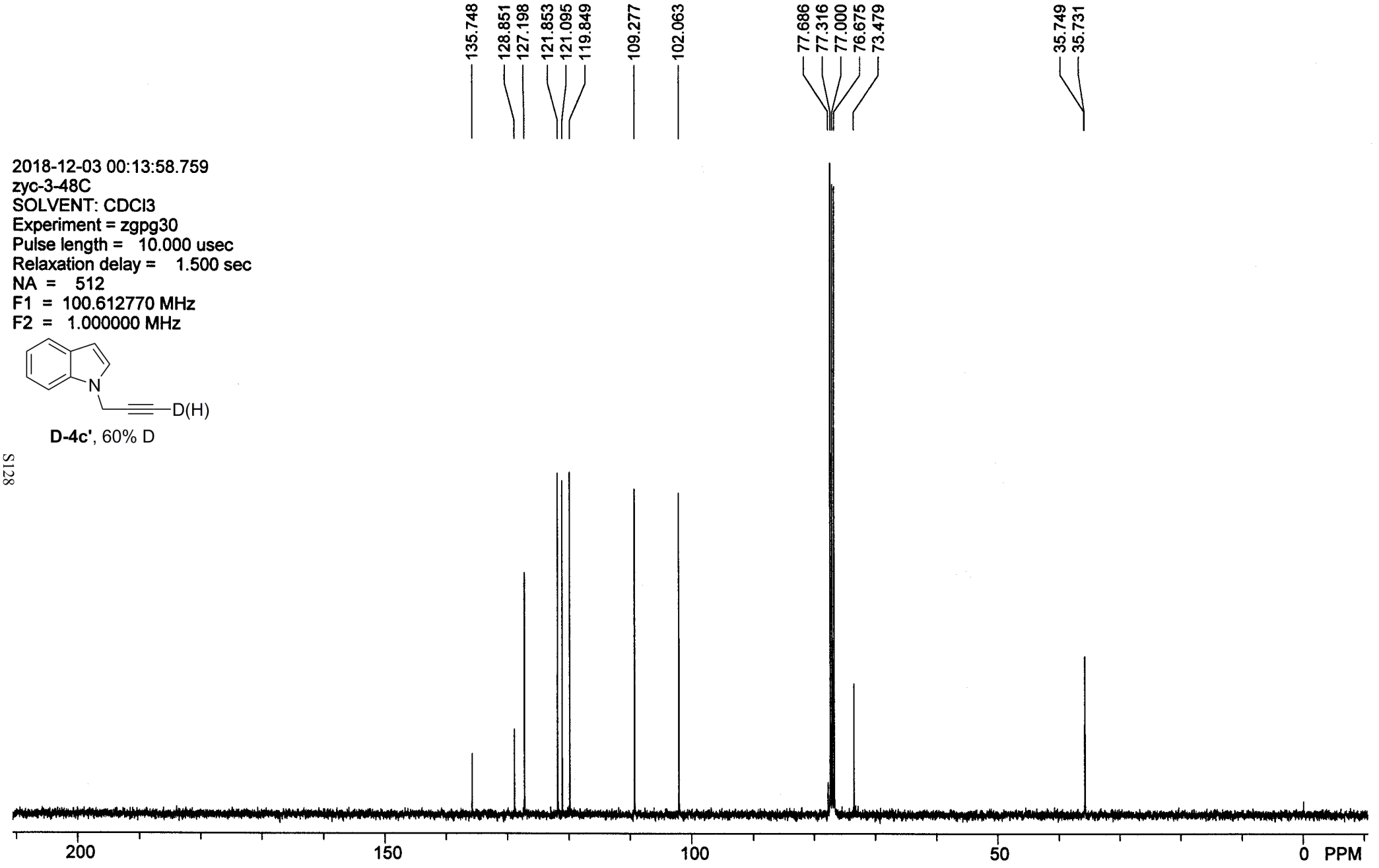
2018-11-28 08:44:56.375
zyc-3-46
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 10627
F1 = 75.467751 MHz
F2 = 1.000000 MHz



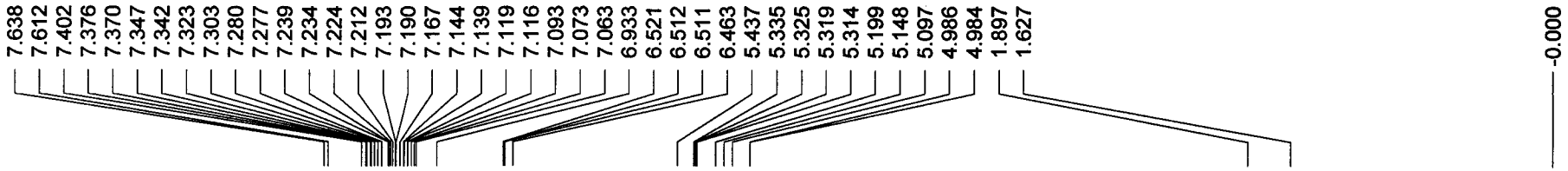
D-5ac **D-6ac**

D-5ac/D-6ac = 81/19

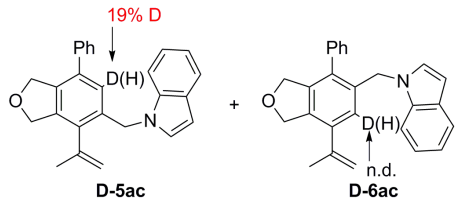

7.646
7.643
7.641
7.626
7.624
7.622
7.407
7.405
7.386
7.384
7.263
7.261
7.246
7.243
7.225
7.223
7.197
7.189
7.154
7.152
7.136
7.134
7.132
7.117
7.114
6.533
6.531
6.525
6.523
4.855
4.851
2.385
2.379
2.372
-0.000
2018-11-29 19:33:21.601
zyc-3-48H
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 15.000 usec
Relaxation delay = 1.000 sec
NA = 16
F1 = 400.130035 MHz
F2 = 1.000000 MHz
N
D(H)
D-4c', 60% D
1.02
1.04
1.25
0.99
1.04
1.00
2.08
0.40
10
8
6
4
2
0
PPM


135.748
128.851
127.198
121.853
121.095
119.849
109.277
102.063
77.686
77.316
77.000
76.675
73.479
35.749
35.731
2018-12-03 00:13:58.759
zyc-3-48C
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 10.000 usec
Relaxation delay = 1.500 sec
NA = 512
F1 = 100.612770 MHz
F2 = 1.000000 MHz
N
D(H)
200
150
100
50
0 PPM

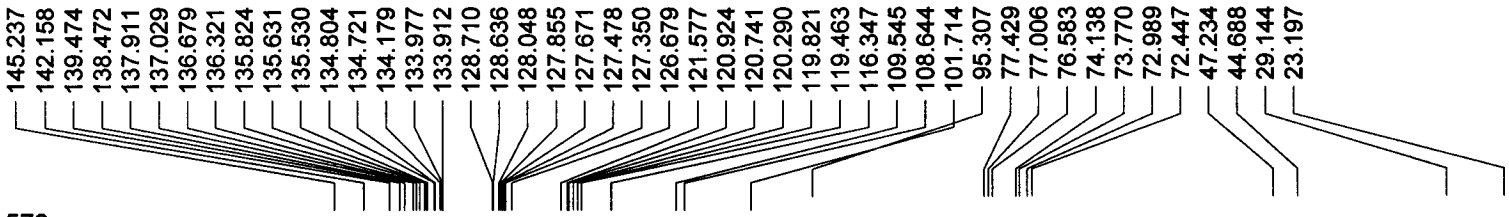
D-4c', 60% D



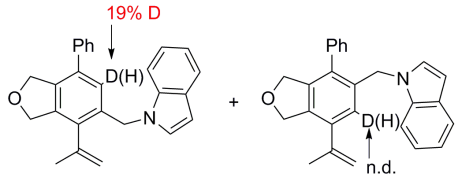
2018-11-26 22:28:43.109
zyc-3-41H
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz



D-5ac/D-6ac = 87/13

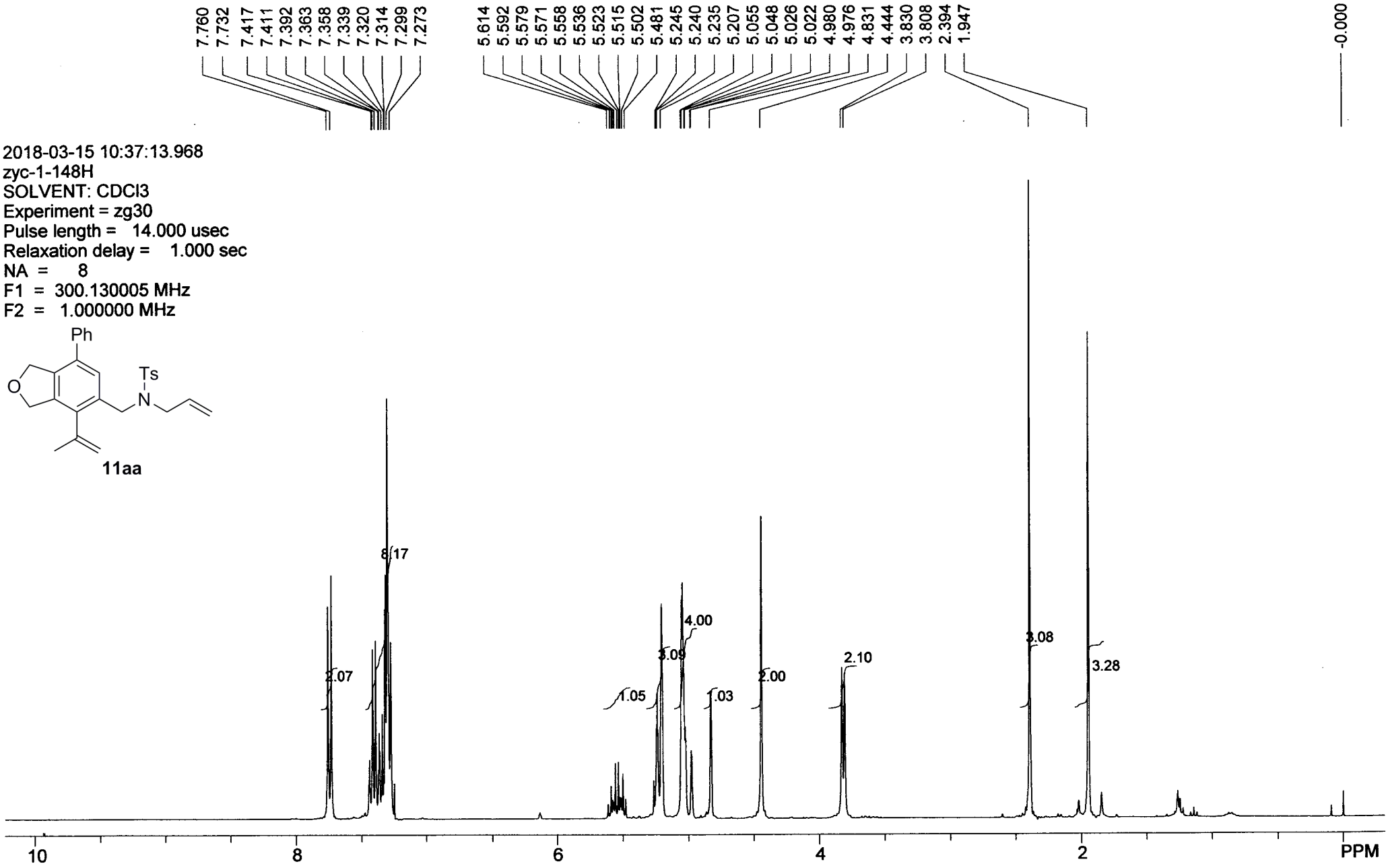


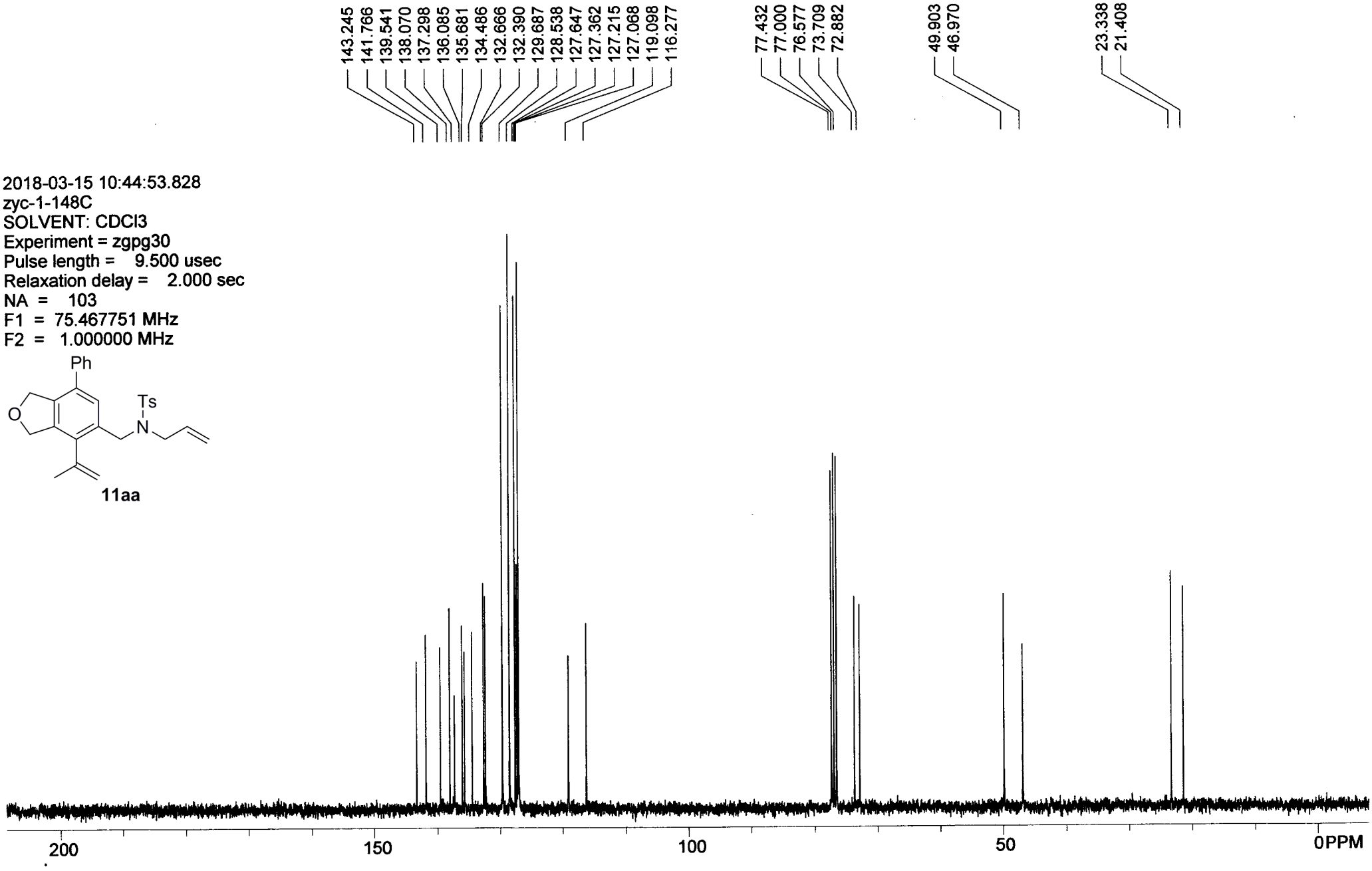
2018-11-27 08:51:22.578
zyc-3-41
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 10000
F1 = 75.467751 MHz
F2 = 1.000000 MHz

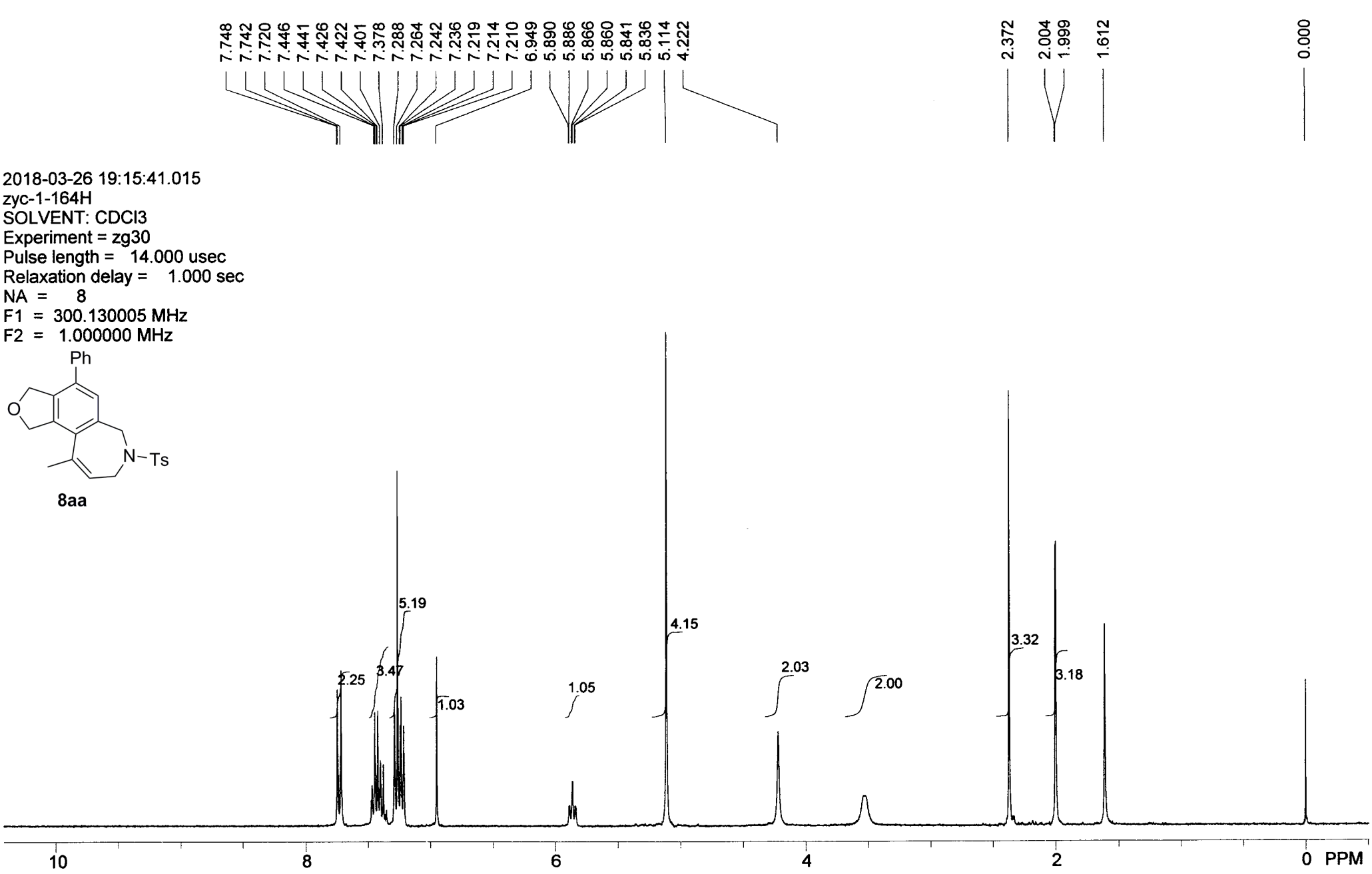


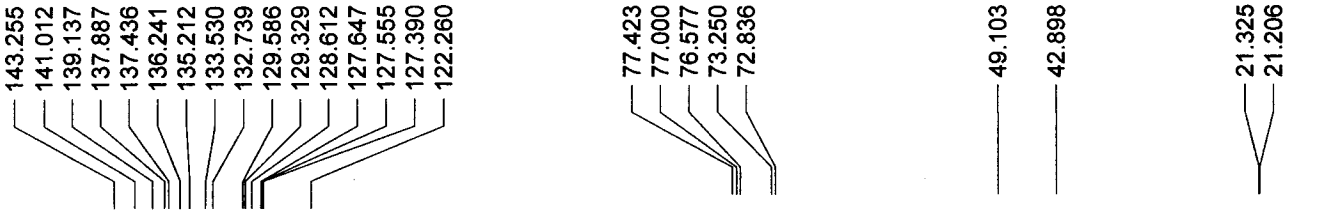
D-5ac **D-6ac**

D-5ac/D-6ac = 87/13

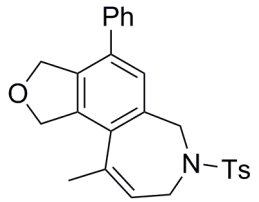

7.760
7.732
7.417
7.411
7.392
7.363
7.358
7.339
7.320
7.314
7.299
7.273
5.614
5.592
5.579
5.571
5.558
5.536
5.523
5.515
5.502
5.481
5.245
5.240
5.235
5.207
5.055
5.048
5.026
5.022
4.980
4.976
4.831
4.444
3.830
3.808
2.394
1.947
-0.000
2018-03-15 10:37:13.968
zyc-1-148H
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
Ph
Ts
N
O
11aa
2.07
8.17
1.05
3.09
4.00
1.03
2.00
2.10
3.08
3.28
10
8
6
4
2
PPM


143.245
141.766
139.541
138.070
137.298
136.085
135.681
134.486
132.666
132.390
129.687
128.538
127.647
127.362
127.215
127.068
119.098
116.277
77.432
77.000
76.577
73.709
72.882
49.903
46.970
23.338
21.408
2018-03-15 10:44:53.828
zyc-1-148C
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 103
F1 = 75.467751 MHz
F2 = 1.000000 MHz
Ph
Ts
O
N
11aa
200
150
100
50
0PPM

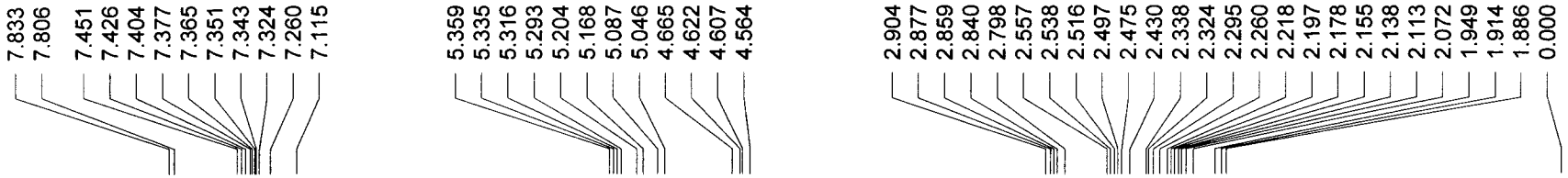

7.748
7.742
7.720
7.446
7.441
7.426
7.422
7.401
7.378
7.288
7.264
7.242
7.236
7.219
7.214
7.210
6.949
5.890
5.886
5.866
5.860
5.841
5.836
5.114
4.222
2.372
2.004
1.999
1.612
0.000
2018-03-26 19:15:41.015
zyc-1-164H
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
Ph
O
N–Ts
8aa
2.25
3.47
5.19
1.03
1.05
4.15
2.03
2.00
3.32
3.18
10
8
6
4
2
0 PPM



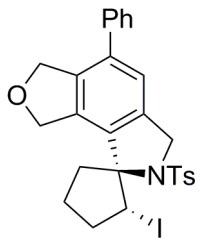
2018-03-26 19:29:01.671
zyc-1-164C
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 203
F1 = 75.467751 MHz
F2 = 1.000000 MHz



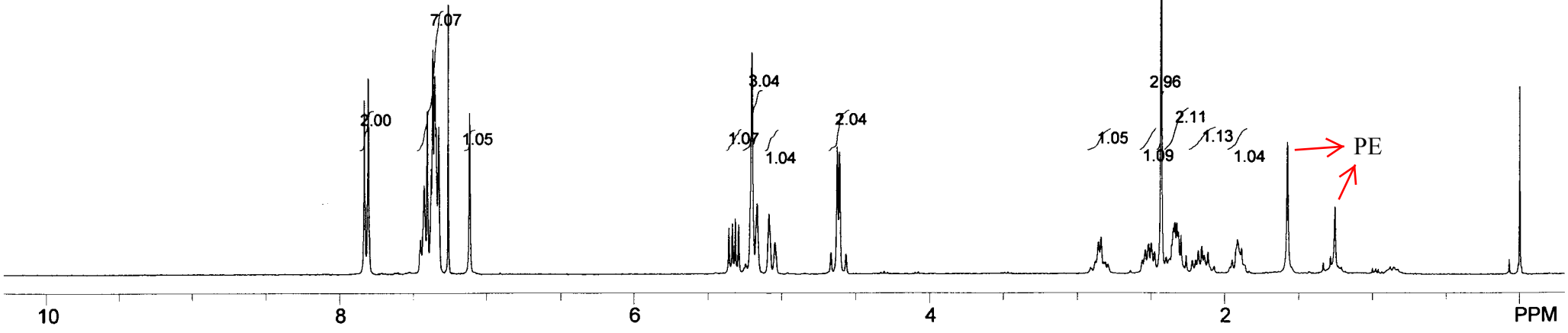
8aa

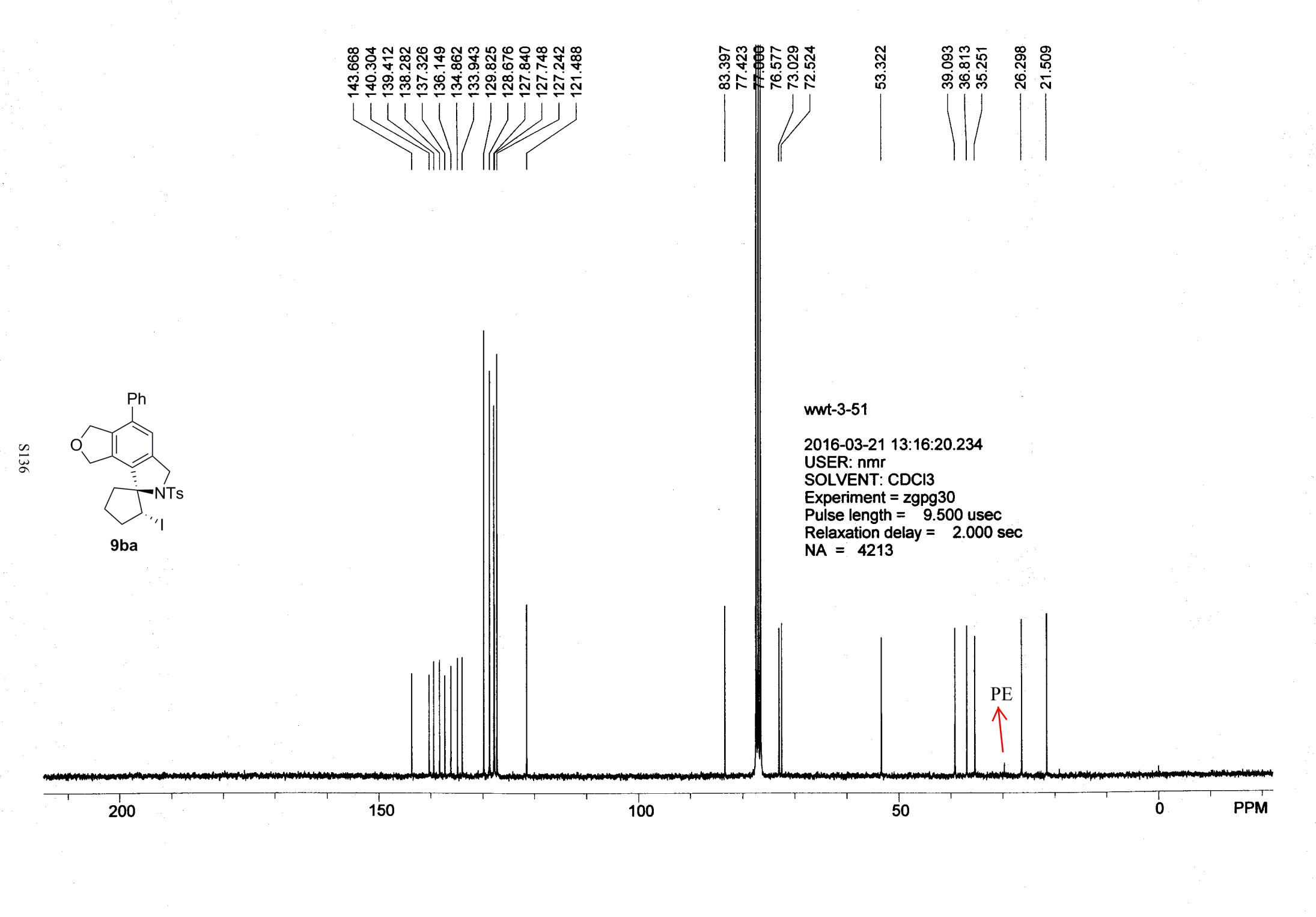


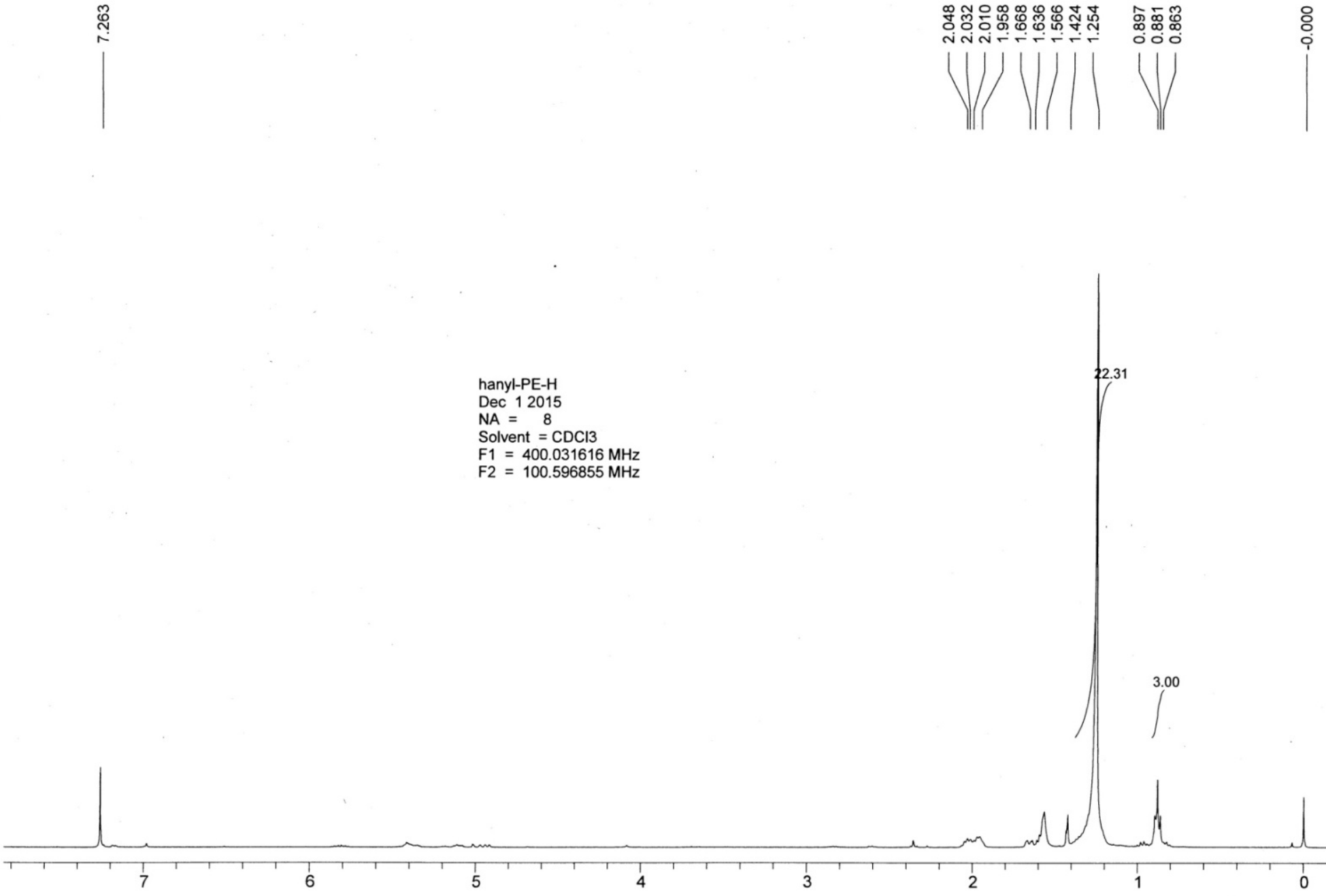
2016-03-26 16:39:23.843
wwt-3-51
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz

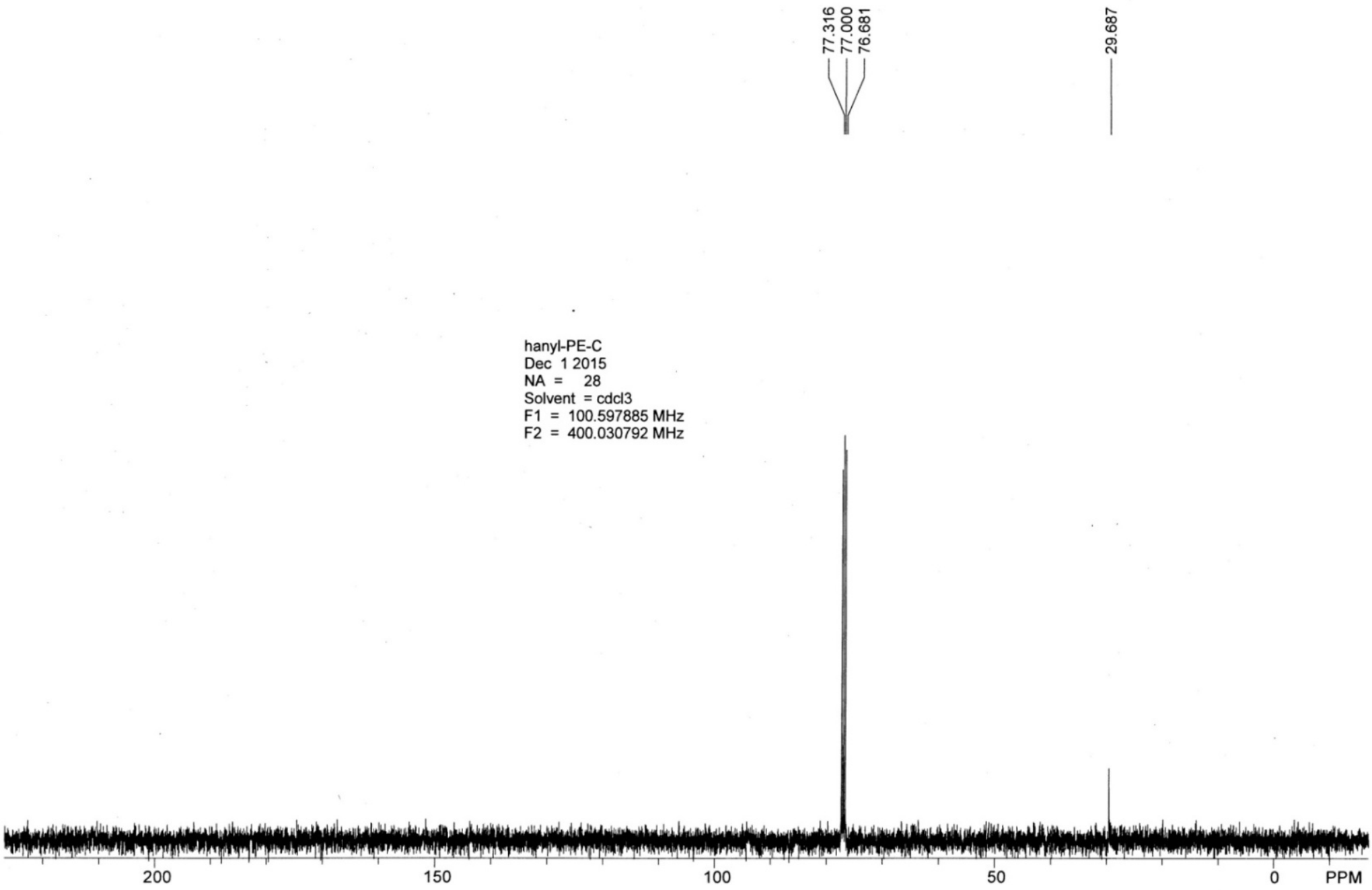


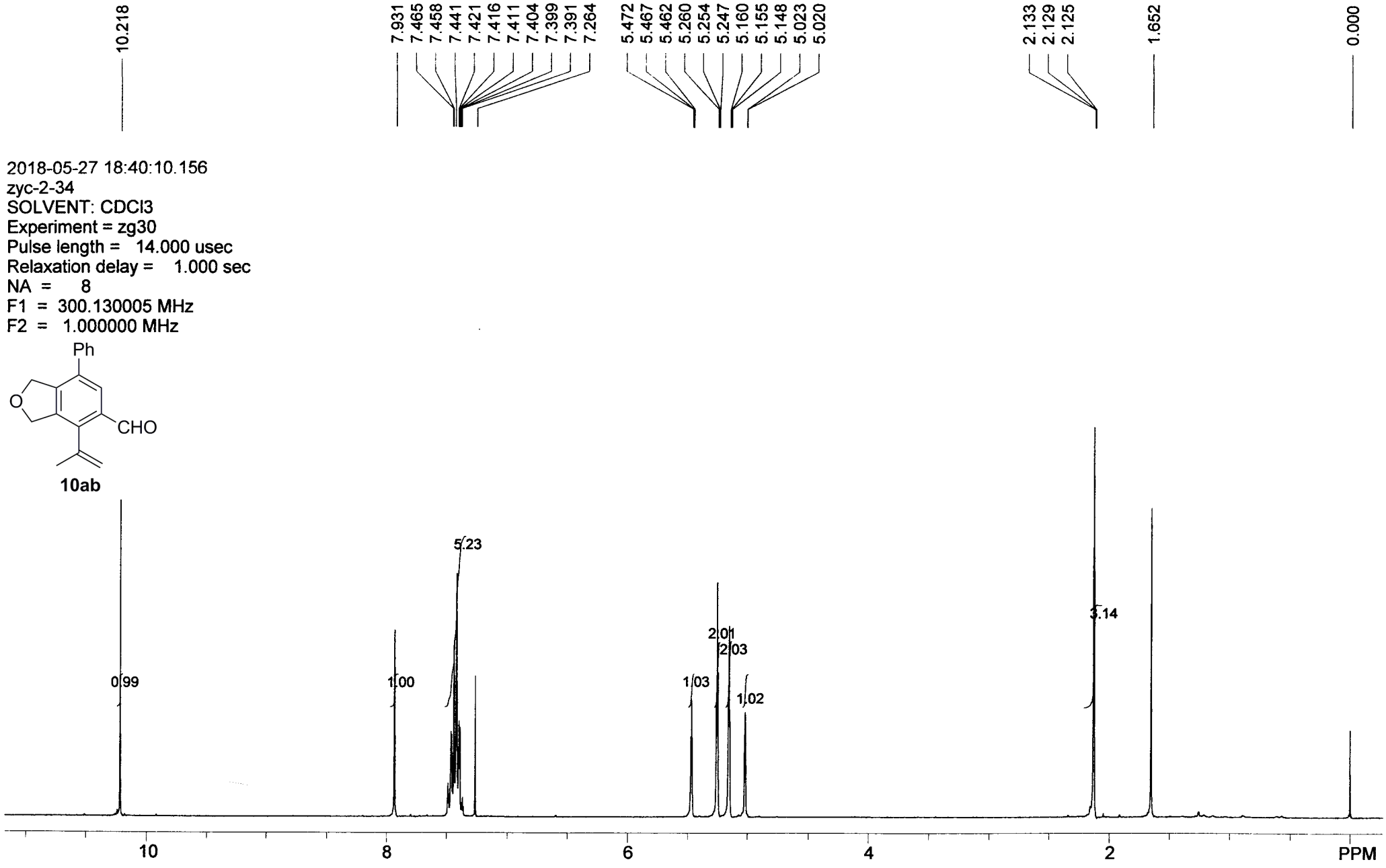
9ba

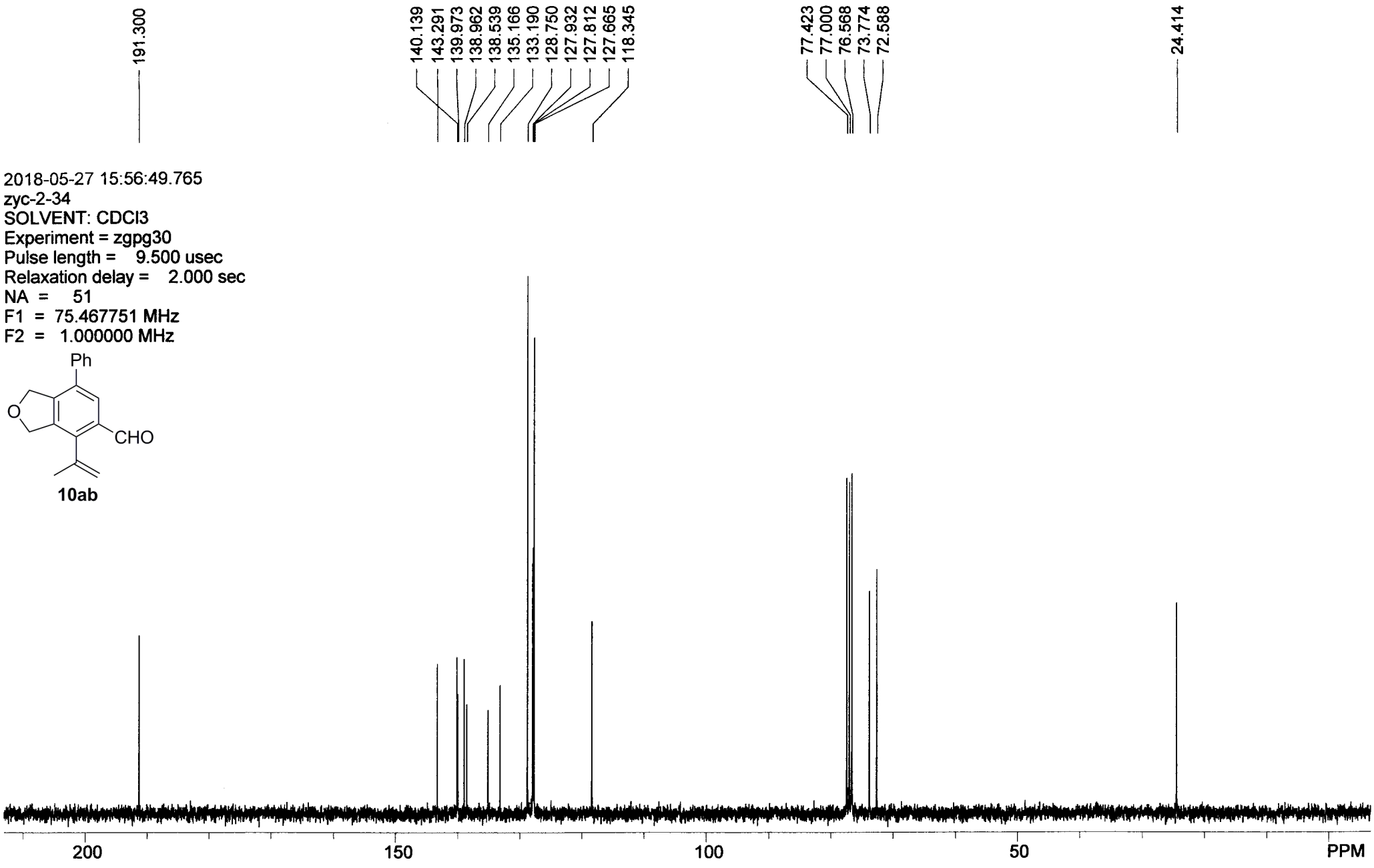



143.668
140.304
139.412
138.282
137.326
136.149
134.862
133.943
129.825
128.676
127.840
127.748
127.242
121.488
83.397
77.423
77.000
76.577
73.029
72.524
53.322
39.093
36.813
35.251
26.298
21.509
Ph
O
NTs
I
9ba
wwt-3-51
2016-03-21 13:16:20.234
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 4213
PE
200
150
100
50
0
PPM


7.263
2.048
2.032
2.010
1.958
1.668
1.636
1.566
1.424
1.254
0.897
0.881
0.863
-0.000
hanyl-PE-H
Dec 1 2015
NA = 8
Solvent = CDCl3
F1 = 400.031616 MHz
F2 = 100.596855 MHz
22.31
3.00
7
6
5
4
3
2
1
0
PPM


77.316
77.000
76.681
29.687
hanyl-PE-C
Dec 1 2015
NA = 28
Solvent = cdcl3
F1 = 100.597885 MHz
F2 = 400.030792 MHz
200
150
100
50
0
PPM


10.218
7.931
7.465
7.458
7.441
7.421
7.416
7.411
7.404
7.399
7.391
7.264
5.472
5.467
5.462
5.260
5.254
5.247
5.160
5.155
5.148
5.023
5.020
2.133
2.129
2.125
1.652
0.000
2018-05-27 18:40:10.156
zyc-2-34
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
Ph
O
CHO
10ab
0.99
1.00
5.23
1.03
2.01
2.03
1.02
3.14
10
8
6
4
2
PPM


191.300
140.139
143.291
139.973
138.962
138.539
135.166
133.190
128.750
127.932
127.812
127.665
118.345
77.423
77.000
76.568
73.774
72.588
24.414
2018-05-27 15:56:49.765
zyc-2-34
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 51
F1 = 75.467751 MHz
F2 = 1.000000 MHz
Ph
O
CHO
10ab
200
150
100
50
PPM