Electronic Supplementary Material (ESI) for ChemComm.

Copper-Catalyzed Radical Approach to Allenyl Halides

Yulong Song,[a] Xin Huang,[a] Shihua Song,[a] Xinyu Duan,[a] Xiaoyan Wu,[a] Feng Jiang,[a] Yuchen Zhang,[a] Junjie Fan,[a] Chunling Fu[a] and Shengming Ma*[a]

[a] Laboratory of Molecular Recognition and Synthesis, Department of Chemistry, Zhejiang University, Hangzhou 310027, Zhejiang, People's Republic of China.

Fax: (+86)21-64167510

E-mail: masm@sioc.ac.cn

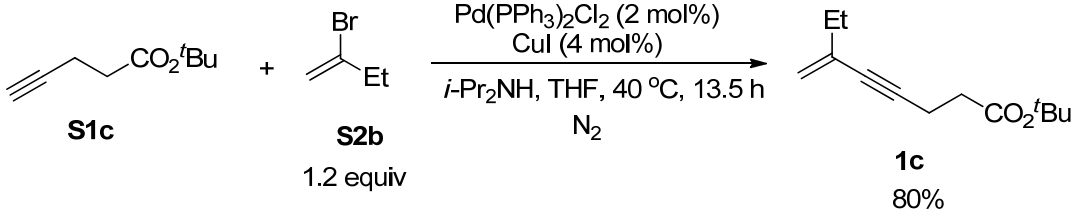
Supporting Information

General Information

^{1}H NMR and ^{13}C NMR spectra were recorded in $CDCl_3$ with a Bruker AM 300 MHz NMR spectrometer (^{1}H at 300 MHz, ^{13}C at 75 MHz, ^{19}F at 282 MHz) using TMS (^{1}H, $\delta = 0$), residual $CHCl_3$ (^{13}C, $\delta = 77.0$), and $CFCl_3$ (^{19}F, $\delta = 0$) as the internal standards. 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. $Pd(PPh_3)_4$ was prepared according to the known literature.[1] $Pd(PPh_3)_2Cl_2$, *i*-Pr_2NH and CuI were purchased from *Adamas*. 1,10-Phen was purchased from *Accela*. $CHCl_3$ was distilled with CaH_2 under N_2 before use. THF was distilled with Na wire using benzophenone as the indicator under N_2 before use. All the temperatures are referred to the oil baths used. Compounds **4a**[2], **4b**[2], **4c**[2], **4d**[2], **4e**[2], **4f**[2], **2**[3], **1a**[4], **1b**[5], **1f**[6], **1h**[7], **1j**[8], **1k**[9], **1m**[9], **1n**[9], **1q**[9], **1u**[10], **1v**[11], **1w**[11], **1x**[11], **1y**[11], **1z**[11], **1z-1**,[11] and **7**[12] were prepared as reported in the literature. The methods of synthesizing this materials **1c**, **1d**, **1e**, **1g**, **1l**, **1o**, **1p**, **1r**, **1s**, **1t** and **1t-1** were based on the literature.[4] The materials **1x**, **1y**, **1z**, **1z-1**, and the product **6ka** are unstable under air atmosphere and should be used right after preparation.

Synthesis of starting materials.

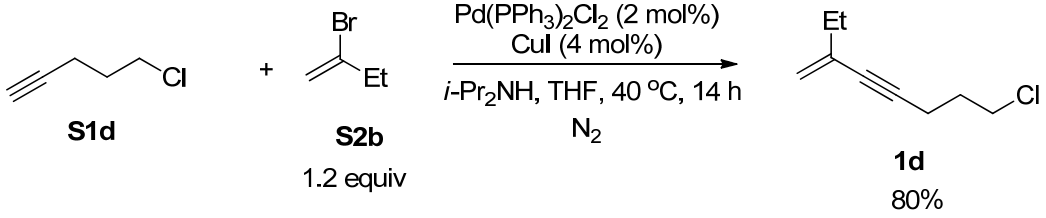
1. Preparation of *tert*-butyl 6-ethylhept-6-en-4-ynoate **1c** (syl-3-102).



S1c + **S2b** (1.2 equiv) → **1c** (80%)

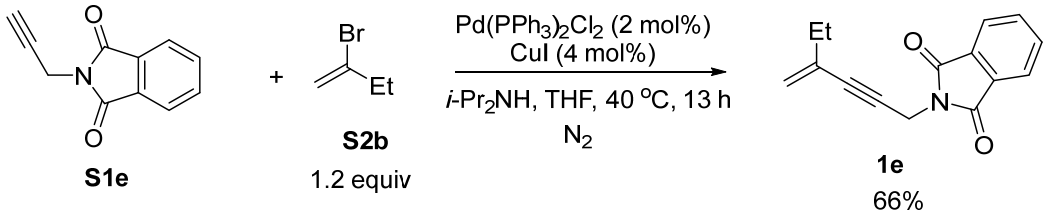
Typical Procedure I: To a three-neck flask were added $Pd(PPh_3)_2Cl_2$ (0.0702 g, 0.1 mmol), CuI (0.0384 g, 0.2 mmol), *i*-Pr_2NH (2.1 mL, d = 0.719 g/mL, 1.510 g, 15.0 mmol), THF (10 mL), **S2b** (0.6 mL, d = 1.333 g/mL, 0.800 g, 5.9 mmol), and **S1c** (0.7701 g, 5.0 mmol) sequentially under N_2 atmosphere. The reaction mixture was then stirred at 40 °C for 13.5 hours as monitored by TLC. The resulting mixture was filtrated through a pad of silica gel and eluted with ethyl acetate (10 mL × 3). After evaporation of the solvent, the residue was purified by chromatography on silica gel to afford **1c** (0.8355 g, 80%) (eluent: petroleum ether (60~90 °C)/ethyl acetate (20/1) (400 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.20 (s, 1 H, one proton of $=CH_2$), 5.15 (s, 1 H, one proton of $=CH_2$), 2.58 (t, *J* = 6.9 Hz, 2 H, CH_2), 2.46 (t, *J* = 7.7 Hz, 2 H, CH_2), 2.13 (q, *J* = 7.4 Hz, 2 H, CH_2), 1.46 (s, 9 H, CH_3 × 3), 1.07 (t, *J* = 7.5 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.2, 133.3, 118.8, 87.9, 81.3, 80.5, 34.8, 30.5, 28.0, 15.3, 12.7; IR (neat) ν (cm^{-1}) 3094, 2974, 2935, 2227, 1732, 1612, 1480, 1457, 1393, 1368, 1247, 1208, 1151, 1089, 1068, 1023; MS (EI): m/z (%) 208 (M^+, 0.25), 193 (M^+-CH_3, 4.32), 57 (100); HRMS calcd. for $C_{13}H_{20}O_2$ (M^+): 208.1463; Found: 208.1458.

2. Preparation of 7-chloro-2-ethylhepten-3-yne **1d** (syl-3-98).



Following **Typical Procedure I**, the reaction of $Pd(PPh_3)_2Cl_2$ (0.0695 g, 0.1 mmol), CuI (0.0378 g, 0.2 mmol), *i*-Pr_2NH (2.1 mL, d = 0.719 g/mL, 1.510 g, 15.0 mmol), THF (10 mL), **S2b** (0.6 mL, d = 1.333 g/mL, 0.800 g, 5.9 mmol), and **S1d** (0.53 mL, d = 0.968 g/mL, 0.513 g, 5.0 mmol) afforded **1d** (0.6215 g, 80%) after chromatography on silica gel (eluent: petroleum ether (60~90 °C) (300 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.22 (s, 1 H, one proton of $=CH_2$), 5.17 (s, 1 H, one proton of $=CH_2$), 3.67 (t, *J* = 6.3 Hz, 2 H, CH_2), 2.51 (t, *J* = 6.8 Hz, 2 H, CH_2), 2.15 (q, *J* = 7.3 Hz, 2 H, CH_2), 1.99 (quint, *J* = 6.6 Hz, 2 H, CH_2), 1.08 (t, *J* = 7.4 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 133.3, 119.0, 87.7, 81.9, 43.7, 31.4, 30.5, 16.7, 12.8; IR (neat) ν (cm^{-1}) 3097, 2968, 2935, 2877, 2847, 2219, 1612, 1454, 1441, 1373, 1354, 1330, 1293; MS (EI): m/z (%) 158 (M^+(^{37}Cl), 16.02), 156 (M^+(^{35}Cl), 47.45), 79 (100); HRMS calcd. for $C_9H_{13}{}^{35}Cl$ (M^+): 156.0700; Found: 156.0701.

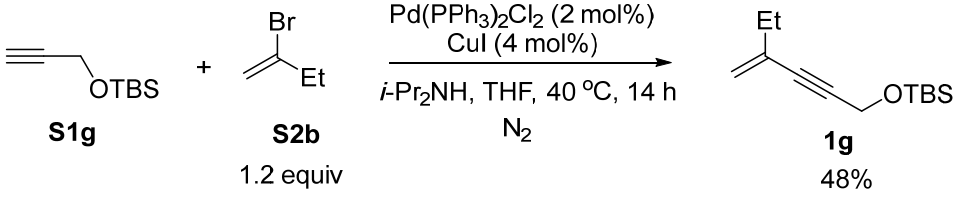
3. Preparation of 2-(4-ethyl-4-penten-2-ynyl)-1,3-dioxoisoindolin **1e** (syl-3-101).



Following **Typical Procedure I**, the reaction of $Pd(PPh_3)_2Cl_2$ (0.0706 g, 0.1 mmol), CuI (0.0379 g, 0.2 mmol), *i*-Pr_2NH (2.1 mL, d = 0.719 g/mL, 1.510 g, 15.0 mmol), THF

(10 mL), **S2b** (0.6 mL, d = 1.333 g/mL, 0.800 g, 5.9 mmol), and **S1e** (0.9251 g, 5.0 mmol) afforded **1e** (0.7840 g, 66%) after chromatography on silica gel (eluent: petroleum ether (60~90 °C)/ethyl acetate (5/1) (300 mL)): solid; m.p. 72.8-73.7 °C (DCM/*n*-hexane); ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.05-7.82 (m, 2 H, ArH), 7.82-7.65 (m, 2 H, ArH), 5.29 (s, 1 H, one proton of $=CH_2$), 5.22 (s, 1 H, one proton of $=CH_2$), 4.59 (s, 2 H, NCH_2), 2.13 (q, *J* = 7.3 Hz, 2 H, CH_2), 1.05 (t, *J* = 7.5 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.0, 134.1, 132.3, 132.0, 123.4, 120.8, 83.4, 82.2, 30.0, 27.7, 12.6; IR (KBr) ν (cm^{-1}) 3088, 3044, 2967, 2939, 2895, 1770, 1724, 1610, 1466, 1421, 1392, 1349, 1325, 1247, 1189, 1120, 1088; MS (EI): m/z (%) 239 (M^+, 100); Anal. Calcd. for $C_{15}H_{13}NO_2$ (%): C 75.30, H 5.48, N 5.85; Found: C 75.16, H 5.71, N 5.52.

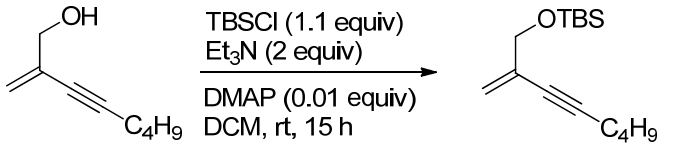
4. Preparation of 2-ethyl-5-(*tert*-butyldimethylsiloxy)penten-3-yne **1g** (syl-3-99).



Following **Typical Procedure I**, the reaction of $Pd(PPh_3)_2Cl_2$ (0.0703 g, 0.1 mmol), CuI (0.0379 g, 0.2 mmol), *i*-Pr_2NH (2.1 mL, d = 0.719 g/mL, 1.510 g, 15.0 mmol), THF (10 mL), **S2b** (0.6 mL, d = 1.333 g/mL, 0.800 g, 5.9 mmol), and **S1g** (0.8521 g, 5.0 mmol) afforded **1g** (0.5346 g, 48%) after chromatography on silica gel (eluent: petroleum ether (60~90 °C)/ethyl acetate (20/1) (420 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.28 (s, 1 H, one proton of $=CH_2$), 5.21 (s, 1 H, one proton of $=CH_2$), 4.44 (s, 2 H, OCH_2), 2.17 (q, *J* = 7.4 Hz, 2 H, CH_2), 1.09 (t, *J* = 7.5 Hz, 3 H, CH_3), 0.92 (s, 9 H,

$CH_3 \times 3$), 0.14 (s, 6 H, $CH_3 \times 2$); ^{13}C NMR (75 MHz, $CDCl_3$) δ 132.9, 119.9, 87.5, 85.3, 52.1, 30.2, 25.8, 18.3, 12.7, -5.1; IR (neat) ν (cm^{-1}) 3096, 2958, 2930, 2885, 2858, 2224, 1613, 1472, 1463, 1390, 1364, 1291, 1255, 1101, 1078, 1037, 1006; MS (EI): m/z (%) 224 (M^+, 0.10), 167 ((M- *t*-Bu)$^+$, 22.09), 137 (100); HRMS calcd. for $C_9H_{15}OSi$ (M-*t*-Bu) $^+$: 167.0887; Found: 167.0889.

5. Preparation of 2-(((*tert*-butyldimethylsilyl)oxy)methyl)octen-3-yne **1i** (syl-3-167).[13]

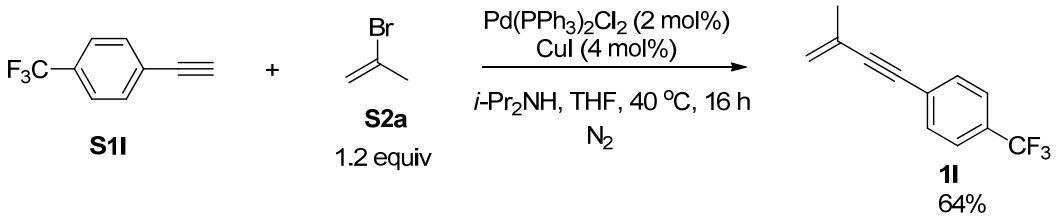


A **1i**
83%, purity = 98%

To a Schlenk tube with an ice-water bath were added TBSCl (0.6650 g, 4.4 mmol), Et_3N (1.1 mL, d = 0.73 g/mL, 0.803 g, 8.0 mmol), DMAP (0.0053 g, 0.04 mmol), and **A** (0.5418 g, 3.9 mmol) in DCM (2 mL). The ice-water bath was removed and the resulting mixture was warmed up to room temperature with stirring for 15 hours as monitored by TLC. The resulting mixture was diluted with 20 mL of DCM and was washed with a saturated sodium chloride solution (20 mL × 3), and the combined organic phase was dried over anhydrous Na_2SO_4. After filtration and evaporation of the solvent, the residue was purified by chromatography on silica gel to afford **1i** (0.8344 g, 83%, purity = 98%) (eluent: petroleum ether (60~90 °C)/ethyl acetate (10/1) (110 mL)): liquid; ^1H NMR (300 MHz, $CDCl_3$) δ 5.48 (s, 1 H, one proton of $=CH_2$), 5.34 (s, 1 H, one proton of $=CH_2$), 4.11 (s, 2 H, OCH_2), 2.30 (t, *J* = 6.6 Hz, 2 H, CH_2), 1.58-

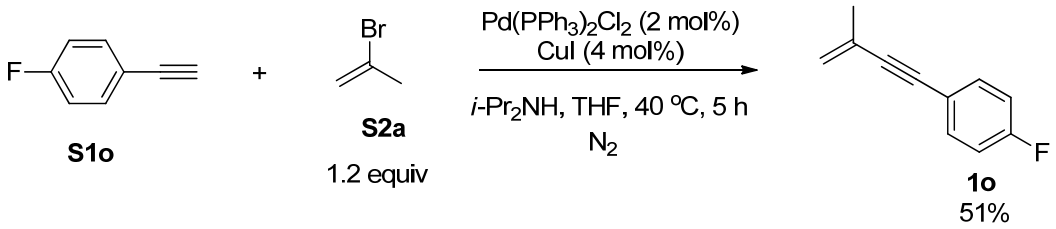
1.32 (m, 4 H, CH_2 × 2), 0.92 (s, 12 H, CH_3 × 4), 0.08 (s, 6 H, CH_3 × 2) ; ^{13}C NMR (75 MHz, $CDCl_3$) δ 131.3, 117.7, 91.3, 78.5, 65.3, 30.8, 25.8, 22.0, 19.0, 18.4, 13.6, -5.4; IR (neat) ν (cm^{-1}) 2963, 2929, 2852, 2228, 1618, 1472, 1459, 1375, 1358, 1257, 1117, 1006; MS (EI): m/z (%) 224 (M^+, 0.01), 195 ((M- *t*-Bu)$^+$, 22.74), 75 (100); HRMS calcd. for $C_{11}H_{19}OSi$ (M- *t*-Bu) $^+$: 195.1200; Found: 195.1199.

6. Preparation of 2-methyl-4-(4-(trifluoromethyl)phenyl)buten-3-yne (syl-3-70, syl-3-162).



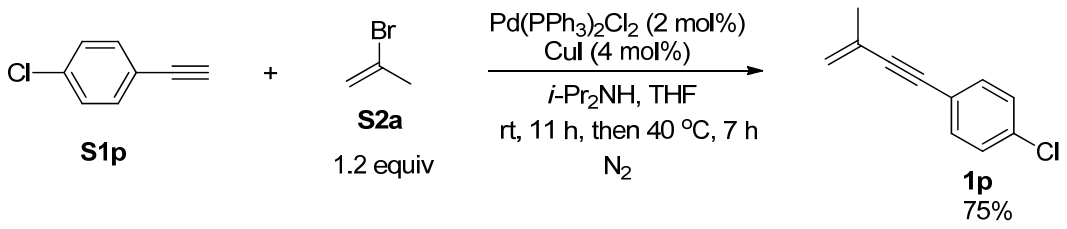
Following **Typical Procedure I**, the reaction of $Pd(PPh_3)_2Cl_2$ (0.0701 g, 0.1 mmol), CuI (0.0385 g, 0.2 mmol), *i*-Pr_2NH (2.1 mL, d = 0.719 g/mL, 1.510 g, 15.0 mmol), THF (10 mL), **S2a** (0.53 mL, d = 1.362 g/mL, 0.722 g, 6.0 mmol), and **S1l** (0.81 mL, d = 1.043 g/mL, 0.845 g, 5.0 mmol) afforded **1l** (0.6681 g, 64%) (eluent: petroleum ether (60~90 °C) (300 mL)): solid; m.p. 37.2-38.5 °C (DCM/*n*-hexane); ^1H NMR (300 MHz, $CDCl_3$) δ 7.57 (d, *J* = 6.6 Hz, 2 H, ArH), 7.53 (d, *J* = 6.6 Hz, 2 H, ArH), 5.45 (d, *J* = 0.6 Hz, 1 H, one proton of $=CH_2$), 5.36 (quint, *J* = 1.2 Hz, 1 H, one proton of $=CH_2$), 2.00 (s, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 131.8, 129.8 (q, *J* = 32.4 Hz), 127.1, 126.4, 125.2 (q, *J* = 3.7 Hz), 123.9 (q, *J* = 270.5 Hz), 123.1, 92.9, 86.9, 23.3; ^{19}F NMR (282 MHz, $CDCl_3$) δ -63.3 (s, 3 F, CF_3); IR (KBr) ν (cm^{-1}) 2926, 2847, 1618, 1325, 1169, 1132, 1106, 1067, 1019; MS (EI): m/z (%) 210 (M^+, 2.83), 181 (100); HRMS calcd. for $C_{12}H_9F_3$ (M^+): 210.0651; Found: 210.0652.

7. Preparation of 2-methyl-4-(4-fluorophenyl)buten-3-yne **1o** (syl-3-84).



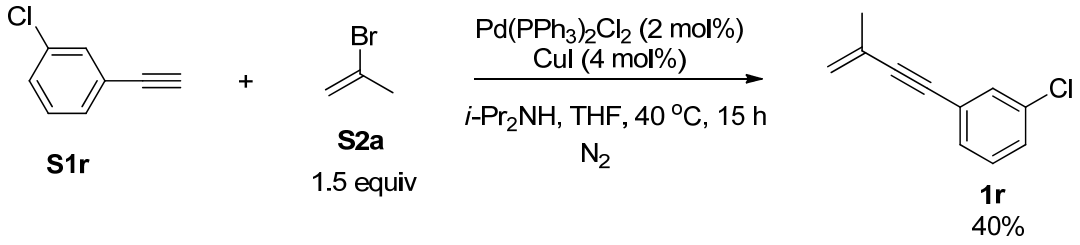
Following **Typical Procedure I**, the reaction of $Pd(PPh_3)_2Cl_2$ (0.1402 g, 0.2 mmol), CuI (0.0765 g, 0.4 mmol), *i*-Pr_2NH (4.2 mL, d = 0.719 g/mL, 3.020 g, 29.9 mmol), THF (20 mL), **S2a** (1.1 mL, d = 1.362 g/mL, 1.498 g, 12.4 mmol), and **S1o** (1.2001 g, 10 mmol) afforded **1o** (0.7915 g, 49%) after chromatography on silica gel (eluent: petroleum ether (60~90 °C) (300 mL)): liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47-7.36 (m, 2 H, ArH), 7.00 (t, *J* = 8.8 Hz, 2 H, ArH), 5.39 (d, *J* = 0.8 Hz, 1 H, one proton of $=CH_2$), 5.30 (quint, *J* = 1.6 Hz, 1 H, one proton of $=CH_2$), 1.98 (s, 3 H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.4 (d, *J* = 247.4 Hz), 133.45, 133.37, 126.7, 122.0, 119.3, 115.6, 115.4, 90.2, 87.3, 23.4; ^{19}F NMR (282 MHz, $CDCl_3$) δ -111.7; IR (neat) ν (cm^{-1}) 3097, 3049, 2955, 2924, 1600, 1507, 1456, 1432, 1373, 1315, 1236, 1218, 1156, 1092, 1014; MS (EI): m/z (%) 160 (M^+, 100); HRMS calcd. for $C_{11}H_9F$ (M^+): 160.0682; Found: 160.0675.

8. Preparation of 2-methyl-4-(4-chlorophenyl)buten-3-yne **1p** (syl-3-72).



To a flame-dried three-neck flask were added $Pd(PPh_3)_2Cl_2$ (0.2102 g, 0.3 mmol), CuI (0.1144 g, 0.6 mmol), *i*-Pr_2NH (6.3 mL, d = 0.719 g/mL, 4.530 g, 44.9 mmol), THF (30 mL), **S2a** (1.6 mL, d = 1.362 g/mL, 2.179 g, 18.0 mmol), and **S1p** (2.0482 g, 15 mmol) sequentially under N_2 atmosphere. The reaction mixture was stirred at room temperature for 11 hours. Then the resulting mixture was warmed up to 40 ºC, stirred for 7 hours as monitored by TLC, filtrated through a pad of silica gel (2 cm), eluted with ethyl acetate (10 mL × 3). After evaporation of the solvent, the residue was purified by chromatography on silica gel to afford **1p** (1.9786 g, 75%) (eluent: petroleum ether (60~90 ºC) (300 mL)): solid; m.p. 34.7-36.3 ºC (DCM/*n*-hexane); ^1H NMR (400 MHz, $CDCl_3$) δ 7.36 (d, *J* = 8.8 Hz, 2 H, ArH), 7.28 (d, *J* = 8.4 Hz, 2 H, ArH), 5.40 (s, 1 H, one proton of $=CH_2$), 5.31 (quint, *J* = 1.4 Hz, 1 H, one proton of $=CH_2$), 1.98 (s, 3 H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 134.1, 132.8, 128.6, 126.6, 122.4, 121.7, 91.5, 87.2, 23.4; IR (KBr) ν (cm^{-1}) 3096, 2975, 2954, 2922, 1614, 1591, 1490, 1454, 1398, 1373, 1315, 1264, 1093, 1014; MS (EI): m/z (%) 178 ($M^+(^{37}Cl)$, 24.55), 176 ($M^+(^{35}Cl)$, 88.75), 141 (100); Anal. Calcd. for $C_{11}H_9Cl$ (%): C 74.79, H 5.14; Found: C 74.81, H 5.21.

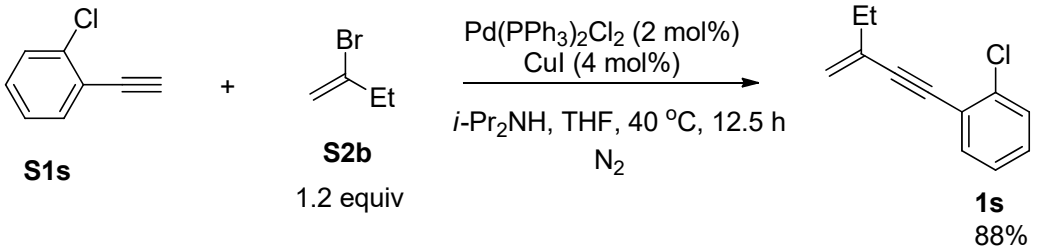
9. Preparation of 2-methyl-4-(3-chlorophenyl)buten-3-yne **1r** (syl-3-94).



Following **Typical Procedure I**, the reaction of $Pd(PPh_3)_2Cl_2$ (0.1402 g, 0.2 mmol),

CuI (0.0762 g, 0.4 mmol), *i*-Pr_2NH (4.2 mL, d = 0.719 g/mL, 3.020 g, 29.9 mmol), THF (20 mL), **S2a** (1.3 mL, d = 1.362 g/mL, 1.771 g, 14.6 mmol), and **S1r** (1.2 mL, d = 1.109 g/mL, 1.331 g, 9.8 mmol) afforded **1r** (0.6957 g, 40%) after chromatography on silica gel (eluent: petroleum ether (60~90 °C) (300 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.43 (s, 1 H, ArH), 7.39-7.14 (m, 3 H, ArH), 5.41 (s, 1 H, one proton of $=CH_2$), 5.33 (s, 1 H, one proton of $=CH_2$), 1.98 (s, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 134.1, 131.4, 129.7, 129.5, 128.4, 126.5, 125.0, 122.7, 91.7, 86.9, 23.3; IR (neat) ν (cm^{-1}) 3297, 3088, 3067, 2959, 2924, 2847, 2219, 1612, 1591, 1561, 1474, 1450, 1407, 1373, 1315, 1199, 1160, 1092, 1078; MS (EI): m/z (%) 178 (M^+(^{37}Cl), 24.31), 176 (M^+(^{35}Cl), 79.03), 141 (100); HRMS calcd. for $C_{11}H_9{}^{35}Cl$ (M^+): 176.0387; Found: 176.0385.

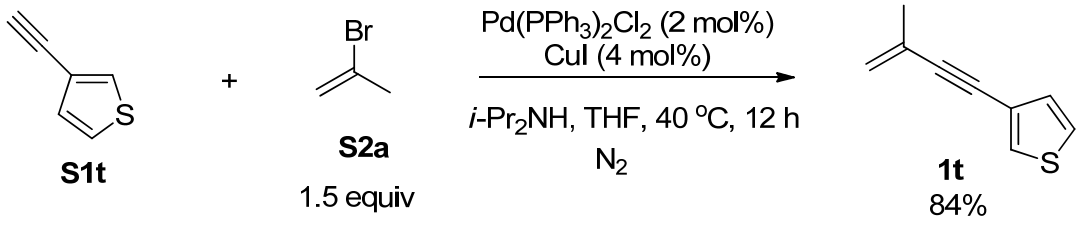
10. Preparation of 2-ethyl-4-(2-chlorophenyl)buten-3-yne **1s** (syl-3-103).



Following **Typical Procedure I**, the reaction of $Pd(PPh_3)_2Cl_2$ (0.0703 g, 0.1 mmol), CuI (0.0382 g, 0.2 mmol), *i*-Pr_2NH (2.1 mL, d = 0.719 g/mL, 1.510 g, 15.0 mmol), THF (10 mL), **S2b** (0.6 mL, d = 1.333 g/mL, 0.800 g, 5.9 mmol), and **S1s** (0.6 mL, d = 1.125 g/mL, 0.675 g, 5.0 mmol) afforded **1s** (0.7708 g, 88%) (eluent: petroleum ether (60~90 °C) (300 mL)): liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50-7.44 (m, 1 H, ArH), 7.43-7.35 (m, 1 H, ArH), 7.28-7.15 (m, 2 H, ArH), 5.46 (t, *J* = 0.8 Hz, 1 H, one proton of

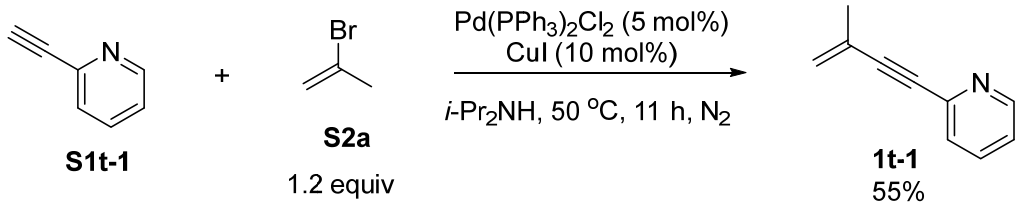
=CH_2), 5.35 (q, J = 1.6 Hz, 1 H, one proton of =CH_2), 2.30 (q, J = 7.5 Hz, 2 H, CH_2), 1.19 (t, J = 7.4 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 135.8, 133.2, 133.0, 129.2, 129.1, 126.4, 123.2, 120.9, 95.0, 85.9, 30.3, 12.9; IR (neat) ν (cm^{-1}) 3088, 3067, 2970, 2936, 2873, 2206, 1609, 1583, 1561, 1474, 1438, 1394, 1371, 1328, 1305, 1250, 1129, 1057, 1032; MS (EI): m/z (%) 192 (M^+(^{37}Cl), 33.34), 190 (M^+(^{35}Cl), 100); HRMS calcd. for $C_{12}H_{11}{}^{35}Cl$ (M^+): 190.0549; Found: 190.0549.

11. Preparation of 2-methyl-4-(thien-3-yl)buten-3-yne **1t** (syl-3-88).



Following **Typical Procedure I**, the reaction of $Pd(PPh_3)_2Cl_2$ (0.0843 g, 0.12 mmol), CuI (0.0452 g, 0.24 mmol), *i*-Pr_2NH (2.5 mL, d = 0.719 g/mL, 1.798 g, 17.8 mmol), THF (12 mL), **S2a** (0.8 mL, d = 1.362 g/mL, 1.090 g, 9.0 mmol), and **S1t** (0.6 mL, d = 1.098 g/mL, 0.659 g, 6.1 mmol) afforded **1t** (0.7550 g, 84%) (eluent: petroleum ether (60~90 °C) (300 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.47-7.36 (m, 1 H, ArH), 7.31-7.17 (m, 1 H, ArH), 7.16-7.03 (m, 1 H, ArH), 5.37 (s, 1 H, one proton of =CH_2), 5.27 (s, 1 H, one proton of =CH_2), 1.96 (s, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 129.7, 128.4, 126.7, 125.2, 122.2, 121.8, 90.0, 83.5, 23.4; IR (neat) ν (cm^{-1}) 3107, 2973, 2953, 2920, 2852, 2207, 1800, 1671, 1612, 1520, 1451, 1433, 1372, 1356, 1294, 1215, 1158, 1078, 1011; MS (EI): m/z (%) 148 (M^+, 100); HRMS calcd. for C_9H_8S (M^+): 148.0341; Found: 148.0337.

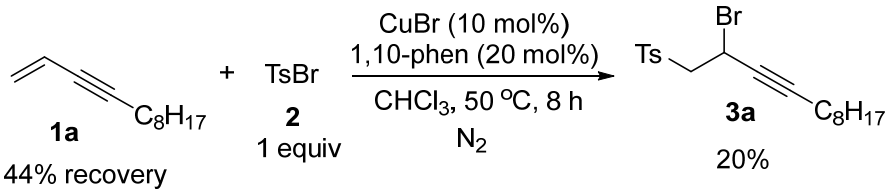
12. Preparation of 2-methyl-4-(pyridine -2-yl)buten-3-yne **1t-1** (syl-5-78).



Following **Typical Procedure I**, the reaction of $Pd(PPh_3)_2Cl_2$ (0.1751 g, 0.25 mmol), CuI (0.0947 g, 0.5 mmol), **S2a** (0.55 mL, d = 1.362 g/mL, 0.749 g, 6.2 mmol), *i*-Pr_2NH (3 mL), and **S1t-1** (0.5 mL, d = 1.02 g/mL, 0.510 g, 5.0 mmol) afforded **1t-1** (0.3920 g, 55%) (eluent: petroleum ether (60~90 °C)/ethyl acetate (10/1) (330 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.57 (d, J = 4.8 Hz, 1 H, ArH), 7.63 (dt, J_1 = 7.4 Hz, J_2 = 1.5 Hz, 1 H, ArH), 7.42 (d, J = 7.8 Hz, 1 H, ArH), 7.26-7.15 (m, 1 H, ArH), 5.51 (s, 1 H, one proton of $=CH_2$), 5.38 (t, J = 1.5 Hz, 1 H, one proton of $=CH_2$), 2.00 (s, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 149.8, 143.3, 135.9, 126.9, 126.0, 123.6, 122.5, 90.2, 87.5, 22.9; IR (neat) ν (cm^{-1}) 3051, 2962, 2919, 2214, 1611, 1562, 1427, 1373, 1316, 1260, 1149, 1090, 1014; MS (EI): m/z (%) 143 (M^+, 99.2), 142 (100); HRMS calcd. for $C_{10}H_9N$ (M^+): 143.0730; Found: 143.0725.

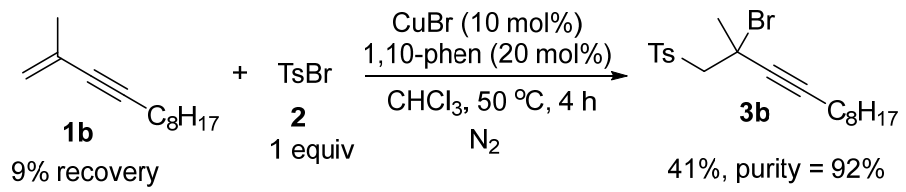
Synthesis of products

1. Preparation of 1-tosyldodec-3-yn-2-yl bromide **3a** (syl-4-135).

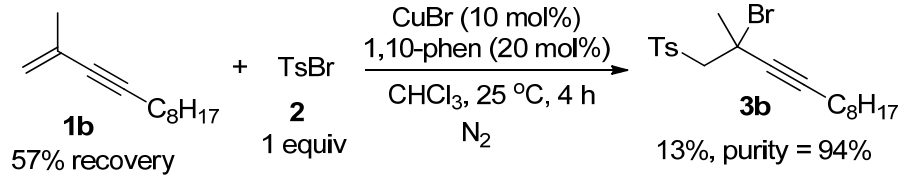


Typical Procedure II: To a flame-dried Schlenk tube were added 1,10-phen (0.0185 g, 0.06 mmol), CuBr (0.0074 g, 0.03 mmol), **1a** (0.0824, 0.5 mmol)/$CHCl_3$ (2 mL), **2** (0.1172 g, 0.5 mmol), and $CHCl_3$ (3 mL) sequentially under N_2 atmosphere. The resulting mixture was stirred at 50 °C for 8 hours as monitored by TLC. The resulting mixture was filtrated through a pad of silica gel eluted with ethyl acetate (10 mL × 3). After evaporation of the solvent under reduced pressure, the yield of the crude product was determined by ^{1}H NMR analysis using mesitylene (23 μL) as the internal standard (22% by NMR, 44% recovery of **1a**). After evaporation of the solvent, the residue was purified by chromatography on silica gel to afford **3a** (0.0406 g, 20%) (eluent: petroleum ether (60~90 °C)/ethyl acetate = 20/1 (420 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.81 (d, *J* = 8.1 Hz, 2 H, ArH), 7.36 (d, *J* = 7.8 Hz, 2 H, ArH), 4.95-4.80 (m, 1 H, CH), 3.90-3.68 (m, 2 H, SO_2CH_2), 2.45 (s, 3 H, CH_3), 2.12-1.92 (m, 2 H, CH_2), 1.45-1.15 (m, 12 H, CH_2 × 6), 0.89 (t, *J* = 6.6 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 145.1, 136.0, 129.8, 128.6, 91.3, 76.5, 63.9, 31.7, 29.1, 28.9, 28.8, 27.9, 27.3, 22.6, 21.6, 18.8, 14.0; IR (neat) ν (cm^{-1}) 2927, 2855, 2237, 1597, 1494, 1465, 1401, 1375, 1319, 1304, 1257, 1143, 1087, 1018; MS (EI) *m/z*: 163 ((M-Ts-HBr)$^+$, 15.60), 139 (100); HRMS calcd. for $C_{19}H_{27}O_2S^{79}Br\ Na^+$($M^+$ + Na): 421.0807; Found: 421.0810.

2. Preparation of 2-methyl-1-tosyldodec-3-yn-2-yl bromide **3b** (syl-4-81, syl-4-104).



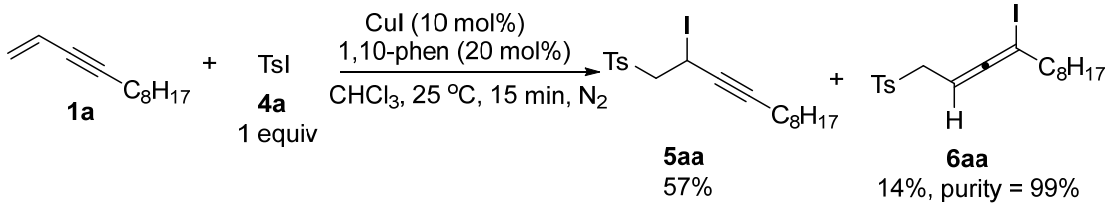
Following **Typical Procedure II**, the reaction of 1,10-phen (0.0109 g, 0.06 mmol), CuBr (0.0045 g, 0.03 mmol), **1b** (0.0535, 0.3 mmol)/$CHCl_3$ (2 mL), **2** (0.0704 g, 0.3 mmol), and $CHCl_3$ (1 mL) afforded **3b** (0.0550 g, 41%, purity = 92%) (eluent: petroleum ether (60~90 °C)/ethyl acetate = 20/1 (420 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.81 (d, *J* = 8.1 Hz, 2 H, ArH), 7.36 (d, *J* = 8.1 Hz, 2 H, ArH), 4.07 (d, *J* = 14.1 Hz, 1 H, one proton of SO_2CH_2), 3.85 (d, *J* = 14.1 Hz, 1 H, one proton of SO_2CH_2), 2.45 (s, 3 H, CH_3), 2.29 (s, 3 H, CH_3), 1.96 (t, *J* = 6.8 Hz, 2 H, CH_2), 1.45-1.15 (m, 12 H, CH_2 × 6), 0.89 (t, *J* = 6.3 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 144.9, 137.1, 129.7, 128.6, 90.7, 80.7, 68.9, 46.7, 34.2, 31.8, 29.1, 29.0, 28.8, 27.9, 22.6, 21.6, 18.9, 14.1; IR (neat) ν (cm^{-1}) 2927, 2856, 2243, 1716, 1597, 1456, 1399, 1375, 1321, 1304, 1285, 1255, 1186, 1151, 1117, 1087, 1053; MS (EI) *m/z*: 163 ($(M\text{-Ts-HBr})^+$, 16.50), 91 (100); HRMS calcd. for $C_{20}H_{29}O_2S^{79}Br$ (M^+): 412.1072; Found: 412.1071.



Following **Typical Procedure II**, the reaction of 1,10-phen (0.0179 g, 0.1 mmol), CuBr (0.0070 g, 0.05 mmol), **1b** (0.0893, 0.5 mmol)/$CHCl_3$ (2 mL), **2** (0.1173 g, 0.5 mmol, and $CHCl_3$ (3 mL) afforded **3b** (0.0276 g, 13%, purity = 94%) after chromatography on silica gel (eluent: petroleum ether (60~90 °C)/ethyl ether = 10/1 (330 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.81 (d, *J* = 8.1 Hz, 2 H, ArH), 7.36

(d, J = 8.1 Hz, 2 H, ArH), 4.06 (d, J = 14.4 Hz, 1 H, one proton of SO_2CH_2), 3.85 (d, J = 14.4 Hz, 1 H, one proton of SO_2CH_2), 2.45 (s, 3 H, CH_3), 2.29 (s, 3 H, CH_3), 1.97 (t, J = 6.6 Hz, 2 H, CH_2), 1.45-1.18 (m, 12 H, CH_2 × 6), 0.89 (t, J = 6.8 Hz, 3 H, CH_3).

3. Preparation of 1-tosyldodec-3-yn-2-yl iodide **5aa** and 1-tosyldodeca-2,3-dien-4-yl iodide **6aa** (syl-4-93).



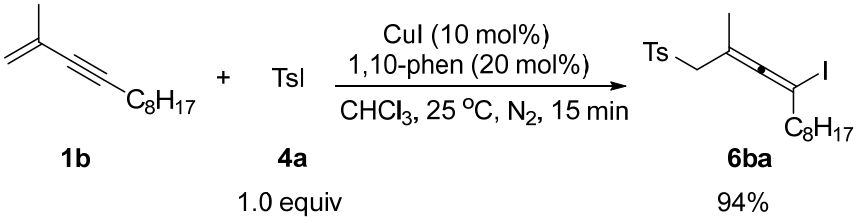
To a flame-dried Schlenk tube were added 1,10-phen (0.0179 g, 0.1 mmol) in glove box. Then CuI (0.0097 g, 0.05 mmol), **1a** (0.0821, 0.5 mmol)/$CHCl_3$ (2 mL), **4a** (0.1412 g, 0.5 mmol), and $CHCl_3$ (3 mL) were added sequentially under N_2 atmosphere. The resulting mixture was stirred at 25 °C for 15 min as monitored by TLC and filtrated through a pad of silica gel eluted with ethyl ether (10 mL × 3). After evaporation of the solvent under reduced pressure at 20 °C, the yield of the crude product was determined by ^{1}H NMR analysis using mesitylene (23 μL) as the internal standard (56% yield of **5aa**, 17% yield of **6aa**). Then the crude residue was transferred to the column with the help of eluent (petroleum ether/ DCM = 5/1) and purified by a flash chromatography on silica gel at -50 °C to afford **5aa** (0.1274 g, 57%) and **6aa** (0.0306 g, 14%, purity = 99%) (eluent: petroleum ether (60~90 °C)/ethyl ether/DCM = 30/1/1 (640 mL)).

5aa: liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.80 (d, J = 8.4 Hz, 2 H, ArH), 7.35 (d, J

= 8.1 Hz, 2 H, ArH), 4.93-4.82 (m, 1 H, CH), 3.90 (dd, J_1 = 14.1 Hz, J_2 = 10.8 Hz, 1 H, one proton of SO_2CH_2), 3.76 (dd, J_1 = 14.1 Hz, J_2 = 3.9 Hz, 1 H, one proton of SO_2CH_2), 2.44 (s, 3 H, CH_3), 1.92 (t, J = 6.0 Hz, 2 H, CH_2), 1.39-1.15 (m, 12 H, CH_2 × 6), 0.89 (t, J = 6.6 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 145.0, 135.9, 129.7, 128.5, 90.5, 79.1, 65.6, 31.7, 29.0, 28.9, 28.7, 27.8, 22.5, 21.6, 18.9, 14.0, -3.3; IR (neat) ν (cm^{-1}) 2926, 2854, 2232, 1597, 1495, 1464, 1401, 1317, 1303, 1253, 1160, 1139, 1086, 1016; MS (EI) *m/z*: 446 (M^+, 4.60), 91 (100); HRMS calcd. for $C_{19}H_{27}O_2SI$ (M^+): 446.0777; Found: 446.0776.

6aa: liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.77 (d, J = 8.1 Hz, 2 H, ArH), 7.36 (d, J = 8.1 Hz, 2 H, ArH), 5.03-4.93 (m, 1 H, CH), 3.77 (d, J = 7.8 Hz, 2 H, SCH_2), 2.45 (s, 3 H, CH_3), 2.15-2.03 (m, 2 H, CH_2), 1.41-1.09 (m, 12 H, CH_2 × 6), 0.89 (t, J = 6.8 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 205.2, 144.9, 135.3, 129.9, 128.5, 81.6, 64.3, 56.4, 39.7, 31.8, 29.2, 29.1, 28.8, 28.2, 22.6, 21.6, 14.1; IR (neat) ν (cm^{-1}) 2925, 2854, 1597, 1491, 1457, 1405, 1320, 1303, 1234, 1153, 1136, 1087, 1042; MS (EI) *m/z*: 446 (M^+, 6.87), 91 (100); HRMS calcd. for $C_{19}H_{27}O_2SI$ (M^+): 446.0777; Found: 446.0779.

4. Preparation of 2-methyl-1-tosyldodeca-2,3-dien-4-yl iodide **6ba** (syl-4-133, syl-3-49).

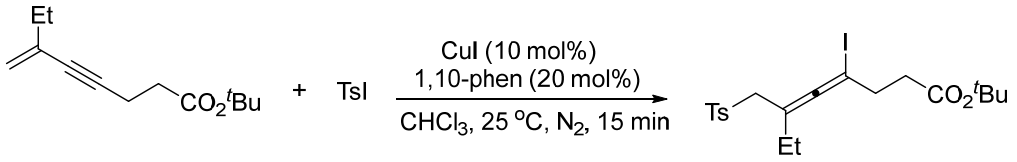


1b **4a** 1.0 equiv **6ba** 94%

Typical Procedure III: To a flame-dried Schlenk tube were added 1,10-phen (0.0361

g, 0.2 mmol), CuI (0.0191 g, 0.05 mmol), **1b** (0.1781 g, 1 mmol)/$CHCl_3$ (4 mL), **4a** (0.2821 g, 1 mmol), and $CHCl_3$ (6 mL) sequentially under N_2 atmosphere. The resulting mixture was stirred at 25 ºC for 15 min as monitored by TLC and filtrated through a short column of silica gel eluted with ethyl ether (10 mL × 3). After evaporation of the solvent under reduced pressure at 20 ºC, the yield of the crude product was determined by ^{1}H NMR analysis using mesitylene (46 μL) as the internal standard (96% by NMR). Then the crude residue was transferred to the column with the help of solvent (petroleum ether/ DCM = 5/1) and purified by a flash chromatography on silica gel to afford **6ba** (0.4303 g, 94%) (eluent: petroleum ether (60~90 ºC)/ethyl acetate = 10/1 (220 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.78 (d, *J* = 8.1 Hz, 2 H, ArH), 7.36 (d, *J* = 8.1 Hz, 2 H, ArH), 3.79 (d, *J* = 13.8 Hz, 1 H, one proton of SO_2CH_2), 3.72 (d, *J* = 13.8 Hz, 1 H, one proton of SO_2CH_2), 2.44 (s, 3 H, CH_3), 2.01 (t, *J* = 6.5 Hz, 2 H, CH_2), 1.84 (s, 3 H, CH_3), 1.46-1.05 (m, 12 H, CH_2 × 6), 0.89 (t, *J* = 6.5 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 203.7, 144.6, 135.5, 129.8, 128.1, 91.8, 63.2, 60.7, 40.0, 31.6, 29.0, 28.9, 28.7, 28.0, 22.4, 21.4, 18.4, 13.9; IR (neat) ν (cm^{-1}) 2955, 2925, 2854, 1955, 1597, 1493, 1456, 1401, 1373, 1320, 1304, 1238, 1153, 1126, 1087, 1038, 1021; MS (EI): m/z (%) 460 (M^+, 16.40), 91 (100); HRMS calcd for $C_{20}H_{29}IO_2SNa^+$ (M^+ + Na): 483.0825; Found: 483.0825.

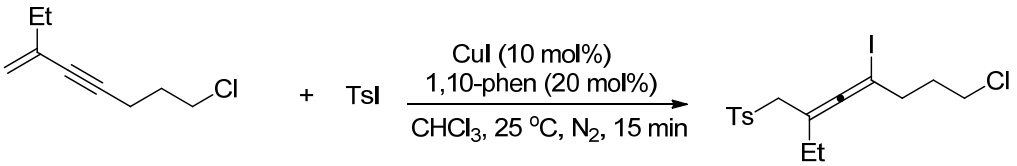
5. Preparation of *tert*-butyl 4-iodo-6-(tosylmethyl)octa-4,5-dienoate (syl-3-176, syl-3-145).



1c **4a** 1.0 equiv **6ca** 93%

Following **Typical Procedure III**, the reaction of 1,10-phen (0.0358 g, 0.2 mmol), CuI (0.0189 g, 0.1 mmol), **1c** (0.2076 g, 1 mmol)/$CHCl_3$ (4 mL), **4a** (0.2821 g, 1 mmol), and $CHCl_3$ (6 mL) afforded **6ca** (0.4545 g, 93%) (eluent: petroleum ether (60~90 °C)/ethyl ether = 5/1 (480 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.78 (d, *J* = 8.1 Hz, 2 H, ArH), 7.37 (d, *J* = 7.5 Hz, 2 H, ArH), 3.79 (d, *J* = 14.4 Hz, 1 H, one proton of SO_2CH_2), 3.73 (d, *J* = 14.7 Hz, 1 H, one proton of SO_2CH_2), 2.55-2.37 (m, 5 H, CH_3 + CH_2), 2.37-2.18 (m, 3 H, one proton of CH_2 + CH_2), 2.18-2.00 (m, 1 H, one proton of CH_2), 1.45 (s, 9 H, CH_3 × 3), 0.98 (t, *J* = 7.4 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 203.4, 170.8, 145.0, 135.9, 130.0, 128.3, 99.8, 80.6, 62.9, 59.7, 36.0, 35.1, 28.1, 25.2, 21.6, 11.6; IR (neat) ν (cm^{-1}) 2975, 2931, 1955, 1726, 1597, 1457, 1393, 1366, 1318, 1306, 1242, 1150, 1087, 1036; MS (EI): m/z (%) 432 ((M-^{t}Bu-H)$^+$, 0.73), 213 (100); HRMS calcd. for $C_{20}H_{27}IO_4SNa^+$ (M^+ + Na): 513.0567; Found: 513.0568.

6. Preparation of 1-chloro-6-(tosylmethyl)octa-4,5-dien-4-yl iodide **6da** (syl-3-159).

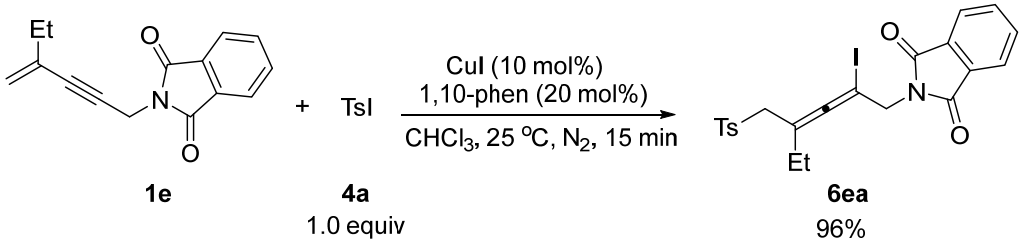


1d **4a** 1.0 equiv **6da** 89%

Following **Typical Procedure III**, the reaction of 1,10-phen (0.0361 g, 0.2 mmol), CuI (0.0191 g, 0.1 mmol), **1d** (0.1563 g, 1 mmol)/$CHCl_3$ (4 mL), **4a** (0.2822 g, 1 mmol),

and $CHCl_3$ (6 mL) afforded pure **6da** (0.2206 g) and impure product (0.1997 g) (eluent: petroleum ether (60~90 °C)/ethyl ether/DCM = 5/1/1 (280 mL)). The impure product was further purified by chromatography on silica gel (eluent: petroleum ether (60~90 °C)/ethyl ether = 5/1 (600 mL)) to afford pure **6da** (0.1709 g). The two parts of the product were combined to produce pure **6da** (0.3915 g, 89%): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.78 (d, *J* = 6.6 Hz, 2 H, ArH), 7.37 (d, *J* = 7.5 Hz, 2 H, ArH), 3.81 (d, *J* = 13.8 Hz, 1 H, one proton of SCH_2), 3.73 (d, *J* = 13.8 Hz, 1 H, one proton of SCH_2), 3.49 (t, *J* = 5.7 Hz, 2 H, CH_2), 2.45 (s, 3 H, CH_3), 2.37-2.17 (m, 3 H, one proton of CH_2 + CH_2), 2.15-1.98 (m, 1 H, one proton of CH_2), 1.92-1.73 (m, 2 H, CH_2), 0.99 (t, *J* = 6.6 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 203.7, 144.9, 135.8, 130.0, 128.3, 99.2, 63.3, 59.8, 43.3, 37.5, 31.5, 25.2, 21.6, 11.6; IR (neat) ν (cm^{-1}) 2965, 2933, 2869, 1953, 1720, 1691, 1597, 1493, 1455, 1435, 1402, 1317, 1300, 1149, 1086; MS (EI): m/z (%) 440 (M^+(^{37}Cl), 4.51), 438 (M^+(^{35}Cl), 12.04), 91 (100); HRMS calcd. for $C_{16}H_{20}{}^{35}ClIO_2SNa^+$ (M^+ + Na): 460.9809; Found: 460.9807.

7. Preparation of 2-(2-iodo-4-(tosylmethyl)hexa-2,3-dien-1-yl) 1,3-dioxoisoindolin **6ea** (syl-3-161).

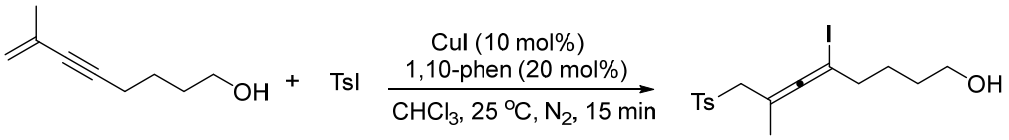


1e **4a** 1.0 equiv **6ea** 96%

Following **Typical Procedure III**, the reaction of 1,10-phen (0.0361 g, 0.2 mmol), CuI (0.0191 g, 0.1 mmol), **1e** (0.2389 g, 1 mmol)/$CHCl_3$ (4 mL), **4a** (0.2823 g, 1 mmol),

and $CHCl_3$ (6 mL) afforded **6ea** (0.5026 g, 96%) eluent: petroleum ether (60~90 °C)/ethyl ether/DCM (4/1/1 (300 mL) to 2/1/1 (400 mL))): solid; m.p. 147.1-148.5 °C (DCM/*n*-hexane); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.95-7.82 (m, 2 H, ArH), 7.82-7.65 (m, 4 H, ArH), 7.35 (d, *J* = 7.8 Hz, 2 H, ArH), 4.36 (d, *J* = 15.6 Hz, 1 H, one proton of NCH_2), 4.27 (d, *J* = 15.6 Hz, 1 H, one proton of NCH_2), 3.75 (d, *J* = 13.5 Hz, 1 H, one proton of SO_2CH_2), 3.65 (d, *J* = 13.8 Hz, 1 H, one proton of SO_2CH_2), 2.43 (s, 3 H, CH_3), 2.12 (q, *J* = 7.2 Hz, 2 H, CH_2), 0.91 (t, *J* = 7.4 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 204.0, 167.1, 144.9, 135.9, 134.2, 131.7, 130.1, 128.2, 123.5, 103.3, 59.1, 57.4, 44.7, 25.0, 21.6, 11.3; IR (KBr) ν (cm^{-1}) 3061, 2969, 2930, 1958, 1772, 1720, 1597, 1494, 1468, 1422, 1387, 1317, 1263, 1237, 1190, 1150, 1111, 1087, 1019; MS (EI): m/z (%) 239 ((M-Ts-I)$^+$, 100); Anal. Calcd. for $C_{22}H_{20}INO_4S$ (%): C 50.68, H 3.87, N 2.69; Found: C 50.62, H 4.04, N 2.54.

8. Preparation of 5-iodo-7-methyl-8-tosylocta-5,6-dien-1-ol **6fa** (syl-3-165).

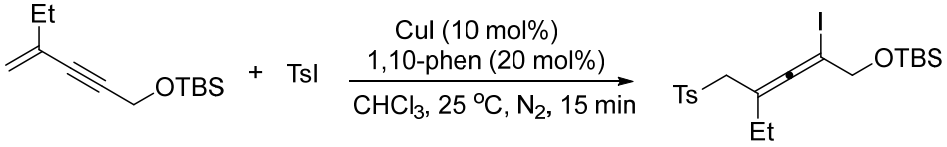


1f **4a** (1.0 equiv) **6fa** (97%)

Following **Typical Procedure III**, the reaction of 1,10-phen (0.0361 g, 0.2 mmol), CuI (0.0189 g, 0.1 mmol), **1f** (0.1375 g, 1 mmol)/$CHCl_3$ (4 mL), **4a** (0.2823 g, 1 mmol), and $CHCl_3$ (6 mL) afforded **6fa** (0.4069 g, 97%) (eluent: petroleum ether (60~90 °C)/ethyl ether/DCM (20/20/1 (820 mL) to 1/1/1 (300 mL))): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.79 (d, *J* = 8.1 Hz, 2 H, ArH), 7.37 (d, *J* = 8.1 Hz, 2 H, ArH), 3.79 (d,

J = 13.5 Hz, 1 H, one proton of SO_2CH_2), 3.71 (d, J = 13.8 Hz, 1 H, one proton of SO_2CH_2), 3.62 (t, J = 5.7 Hz, 2 H, OCH_2), 2.45 (s, 3 H, CH_3), 2.25-2.04 (m, 2 H, CH_2), 1.84 (s, 3 H, CH_3), 1.80 (s, 1 H, OH), 1.58-1.35 (m, 4 H, $CH_2 \times 2$); ^{13}C NMR (75 MHz, $CDCl_3$) δ 204.0, 144.9, 135.8, 130.0, 128.2, 92.2, 62.8, 62.3, 60.9, 40.1, 31.1, 25.2, 21.6, 18.8; IR (neat) ν (cm^{-1}) 3520, 3411, 3062, 2933, 2869, 1955, 1716, 1671, 1597, 1494, 1452, 1401, 1315, 1303, 1289, 1243, 1184, 1149, 1123, 1086, 1018; MS (EI): m/z (%) 138 ($(M\text{-Ts-I})^+$, 4.81), 109 (100); HRMS calcd. for $C_{16}H_{21}IO_3SNa^+$ (M^+ + Na): 443.0148; Found: 443.0148.

9. Preparation of 4-(tosylmethyl)-1-((*tert*-butyldimethylsilyl)oxy)hexa-2,3-dien-2-yl iodide **6ga** (syl-3-184, syl-3-169).

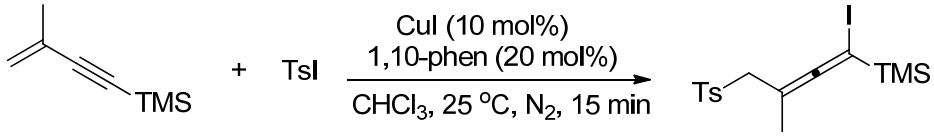


1g 2 mmol | **4a** 1.0 equiv | **6ga** 97%

Following **Typical Procedure III**, the reaction of 1,10-phen (0.0721 g, 0.4 mmol), CuI (0.0384 g, 0.2 mmol), **1g** (0.4478 g, 2 mmol)/$CHCl_3$ (8 mL), **4a** (0.5650 g, 2 mmol), and $CHCl_3$ (12 mL) afforded **6ga** (0.9857 g, 97%) (eluent: petroleum ether (60~90 °C)/ethyl ether/DCM = 30/6/1 (370 mL)): liquid; ^1H NMR (300 MHz, $CDCl_3$) δ 7.78 (d, J = 8.1 Hz, 2 H, ArH), 7.37 (d, J = 8.1 Hz, 2 H, ArH), 3.91 (d, J = 13.2 Hz, 1 H, one proton of SO_2CH_2), 3.85 (d, J = 12.9 Hz, 1 H, one proton of SO_2CH_2), 3.79 (s, 2 H, CH_2), 2.45 (s, 3 H, CH_3), 2.38-2.10 (m, 2 H, CH_2), 1.00 (t, J = 7.4 Hz, 3 H, CH_3), 0.88 (s, 9 H, $CH_3 \times 3$), 0.05 (s, 6 H, $CH_3 \times 2$); ^{13}C NMR (75 MHz, $CDCl_3$) δ 202.9, 144.8,

135.8, 130.0, 128.3, 101.0, 67.1, 66.5, 59.4, 25.7, 25.1, 21.7, 18.2, 11.6, -5.2; IR (neat) ν (cm^{-1}) 2955, 2929, 2877, 2852, 1953, 1596, 1495, 1470, 1457, 1397, 1362, 1317, 1257, 1150, 1104, 1092, 1029, 1006; MS (EI): m/z (%) 449 ((M-^{t}Bu)$^+$, 41.67), 321 (100); Anal. Calcd. for $C_{20}H_{31}IO_3SSi$ (%): C 47.43, H 6.17; Found: C 47.37, H 6.07.

10. Preparation of 3-methyl-1-(trimethylsilyl)-4-tosylbuta-1,2-dienyl iodide **6ha** (syl-3-170).



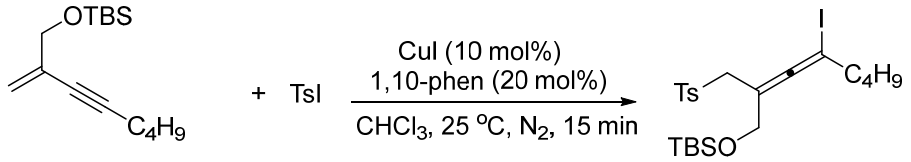
1h 3% recovery

4a 1.0 equiv

6ha 75%

Following **Typical Procedure III**, the reaction of 1,10-phen (0.0358 g, 0.2 mmol), CuI (0.0192 g, 0.1 mmol), **1h** (0.1375 g, 1 mmol)/$CHCl_3$ (4 mL), **4a** (0.2820 g, 1 mmol), and $CHCl_3$ (6 mL) afforded **6ha** (0.3130 g, 75%) (eluent: petroleum ether (60~90 °C)/ethyl ether/DCM = 40/8/1 (245 mL)): solid; m.p. 91.6-93.0 °C (DCM/*n*-hexane); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.77 (d, *J* = 8.1 Hz, 2 H, ArH), 7.34 (d, *J* = 7.8 Hz, 2 H, ArH), 3.75 (d, *J* = 13.8 Hz, 1 H, one proton of SO_2CH_2), 3.68 (d, *J* = 13.8 Hz, 1 H, one proton of SO_2CH_2), 2.41 (s, 3 H, CH_3), 1.77 (s, 3 H, CH_3), 0.11 (s, 9 H, CH_3 × 3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 207.6, 144.8, 136.2, 130.1, 128.1, 89.0, 60.4, 57.7, 21.6, 17.8, -1.5; IR (KBr) ν (cm^{-1}) 3048, 2956, 2917, 2896, 1929, 1596, 1494, 1434, 1402, 1373, 1357, 1313, 1302, 1248, 1203, 1186, 1150, 1119, 1084, 1019, 1006; MS (EI): m/z (%) 138 ((M-I-CH_3)$^+$, 2.63), 73 (100); Anal. Calcd. for $C_{15}H_{21}IO_2SSi$ (%): C 42.86, H 5.04; Found: C 42.83, H 5.05.

11. Preparation of 2-(((*tert*-butyldimethylsilyl)oxy)methyl)-1-tosylocta-2,3-dien-4-yl iodide **6ia** (syl-3-177, syl-3-172).

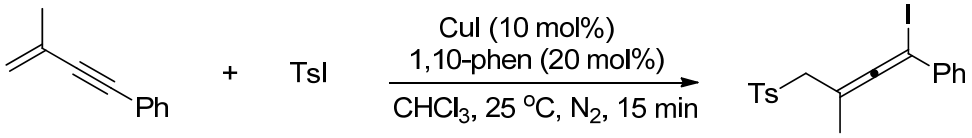


1i **4a** **6ia**

1.0 equiv 94%

Following **Typical Procedure III**, the reaction of 1,10-phen (0.0359 g, 0.2 mmol), CuI (0.0188 g, 0.1 mmol), **1i** (0.2566 g, purity = 98%, 1 mmol)/$CHCl_3$ (4 mL), **4a** (0.2827 g, 1 mmol), and $CHCl_3$ (6 mL) afforded **6ia** (0.5000 g, 94%) (eluent: petroleum ether (60~90 °C)/ethyl ether = 5/1 (240 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.77 (d, *J* = 8.1 Hz, 2 H, ArH), 7.35 (d, *J* = 7.8 Hz, 2 H, ArH), 4.15 (s, 2 H, OCH_2), 3.82 (s, 2 H, SO_2CH_2), 2.43 (s, 3 H, CH_3), 2.10 (t, *J* = 6.9 Hz, 2 H, CH_2), 1.40-1.15 (m, 4 H, CH_2 × 2), 0.87 (s, 12 H, CH_3 × 4), 0.05 (s, 6 H, CH_3 × 2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 202.3, 144.8, 136.0, 130.0, 128.3, 96.2, 65.8, 61.9, 55.8, 39.9, 31.1, 25.8, 21.6, 21.4, 18.2, 13.7, -5.4; IR (neat) ν (cm^{-1}) 2956, 2929, 2857, 1959, 1598, 1494, 1463, 1404, 1361, 1321, 1304, 1257, 1184, 1152, 1128, 1086, 1019, 1006; MS (EI): m/z (%) 195 ((M-Ts-I-Bu)$^+$, 19.62), 75 (100); HRMS calcd. for $C_{22}H_{35}IO_3SSiNa^+$ (M^+ + Na): 557.1013; Found: 557.1014.

12. Preparation of 3-methyl-1-phenyl-4-tosylbuta-1,2-dien-1-yl iodide **6ja** (syl-3-155, syl-3-61).

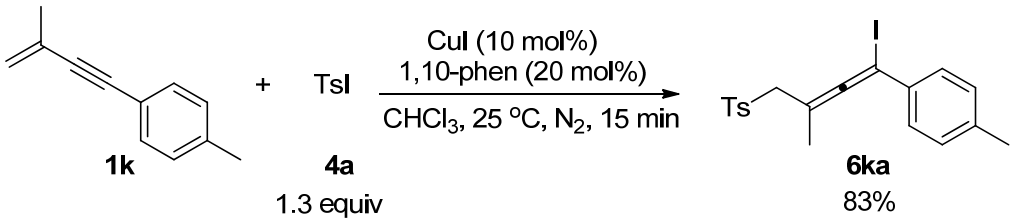


1j **4a** **6ja**

1.3 equiv 96%

Typical Procedure IV: To a flame-dried Schlenk tube were added 1,10-phen (0.0362 g, 0.2 mmol), CuI (0.0187 g, 0.1 mmol), **1j** (0.1423 g, 1 mmol)/$CHCl_3$ (4 mL), **4a** (0.3664 g, 1.3 mmol), and $CHCl_3$ (6 mL) sequentially under N_2 atmosphere. The resulting mixture was stirred at 25 °C for 15 min as monitored by TLC and filtrated through a pad of silica gel eluted with ethyl ether (10 mL × 3). After evaporation of the solvent under reduced pressure at 20 °C, the yield of the crude product was determined by ^1H NMR analysis using CH_2Br_2 (35 μL) as the internal standard (96% by NMR). Then the crude residue was transferred to the column with the help of solvent (petroleum ether/DCM = 5/1) and purified by a flash chromatography on silica gel to afford **6ja** (0.4076 g, 96%) (eluent: petroleum ether (60~90 °C)/ethyl ether/DCM = 4/1/1 (300 mL)): liquid; ^1H NMR (300 MHz, $CDCl_3$) δ 7.76 (d, *J* = 8.1 Hz, 2 H, ArH), 7.35-7.13 (m, 7 H, ArH), 3.94 (d, *J* = 13.8 Hz, 1 H, one proton of SO_2CH_2), 3.82 (d, *J* = 13.8 Hz, 1 H, one proton of SO_2CH_2), 2.38 (s, 3 H, CH_3), 2.00 (s, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 206.4, 144.8, 135.5, 134.8, 130.0, 128.7, 128.2, 128.14, 128.07, 93.8, 62.8, 60.4, 21.6, 18.5; IR (neat) ν (cm^{-1}) 3059, 2981, 2920, 1939, 1667, 1596, 1577, 1490, 1445, 1401, 1376, 1317, 1303, 1242, 1204, 1179, 1148, 1117, 1086, 1018, 1000; MS (EI): m/z (%) 424 (M^+, 1.93), 115 (100); HRMS calcd for $C_{18}H_{17}IO_2SNa^+$ (M^+ + Na): 446.9886; Found: 446.9885.

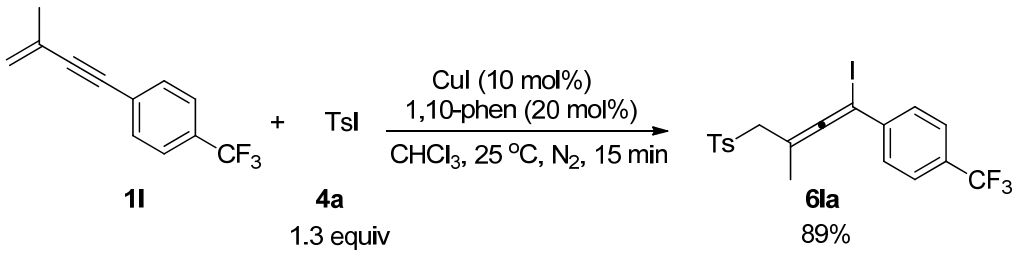
13. Preparation of 3-methyl-1-(4-methylphenyl)-4-tosylbuta-1,2-dienyl iodide **6ka** (zyc-3-95, zyc-3-107)



1k + **4a** 1.3 equiv → **6ka** 83%

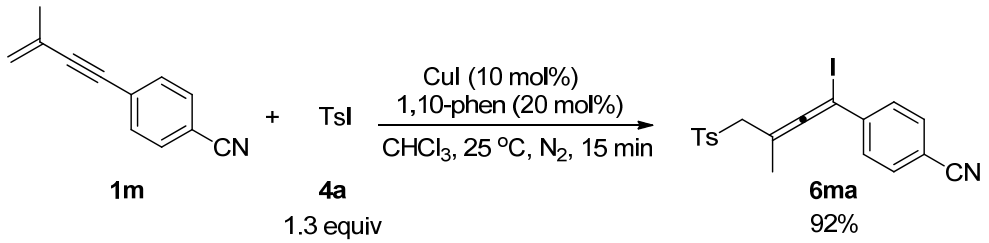
Following **Typical Procedure III**, the reaction of 1,10-phen (0.0361 g, 0.2 mmol), CuI (0.0191 g, 0.1 mmol), **1k** (0.1562 g, 1.0 mmol)/$CHCl_3$ (4 mL), **4a** (0.3665 g, 1.3 mmol), and $CHCl_3$ (6 mL) afforded **6ka** (0.3633 g, 83%) (eluent: petroleum ether (60~90 °C)/ethyl ether/DCM = 210/35/1 (900 mL)): solid; m.p. 78.8-79.6 °C (ethyl ether/*n*-hexane); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.76 (d, *J* = 8.1 Hz, 2 H, ArH), 7.25 (d, *J* = 8.1 Hz, 2 H, ArH), 7.14 (d, *J* = 8.1 Hz, 2 H, ArH), 7.03 (d, *J* = 8.1 Hz, 2 H, ArH), 3.93 (d, *J* = 13.8 Hz, 1 H, one proton of SO_2CH_2), 3.82 (d, *J* = 13.8 Hz, 1 H, one proton of SO_2CH_2), 2.39 (s, 3 H, CH_3), 2.34 (s, 3 H, CH_3), 1.99 (s, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 206.2, 144.8, 138.3, 135.6, 132.0, 130.0, 128.8, 128.6, 128.1, 93.6, 63.0, 60.6, 21.6, 21.0, 18.6; IR (KBr) ν (cm^{-1}) 3042, 2989, 2954, 2917, 2898, 1931, 1595, 1507, 1493, 1450, 1401, 1374, 1310, 1182, 1153, 1111, 1087; MS (EI): *m/z* (%) 311 ($(M-I)^+$, 5.92), 231 (100); Anal. Calcd. for $C_{19}H_{19}IO_2S$ (%): C 52.06, H 4.37, Found: C 51.80, H 4.49.

14. Preparation of 1-(4-(trifluoromethyl)phenyl)-3-methyl-4-tosylbuta-1,2-dienyl iodide **6la** (syl-3-164, syl-3-109).



Following **Typical Procedure IV**, the reaction of 1,10-phen (0.0362 g, 0.2 mmol), CuI (0.0189 g, 0.1 mmol), **1l** (0.2098 g, 1 mmol)/$CHCl_3$ (4 mL), **4a** (0.3665 g, 1.3 mmol), and $CHCl_3$ (6 mL) afforded **6la** (0.4384 g, 89%) (eluent: petroleum ether (60~90 °C)/ ethyl ether /DCM = 60/12/1 (365 mL)): solid; m.p. 109.4-111.2 °C (DCM/*n*-hexane); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.77 (d, *J* = 8.1 Hz, 2 H, ArH), 7.48 (d, *J* = 8.1 Hz, 2 H, ArH), 7.41 (d, *J* = 8.4 Hz, 2 H, ArH), 7.26 (d, *J* = 8.1 Hz, 2 H, ArH), 3.97 (d, *J* = 13.5 Hz, 1 H, one proton of SCH_2), 3.83 (d, *J* = 13.8 Hz, 1 H, one proton of SCH_2), 2.38 (s, 3 H, CH_3), 2.02 (s, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 207.3, 145.0, 138.5, 135.6, 130.0, 129.9 (q, *J* = 32.6 Hz), 129.0, 128.0, 125.0 (q, *J* = 3.7 Hz), 123.7 (q, *J* = 270.2 Hz), 94.6, 60.6, 60.1, 21.4, 18.4; ^{19}F NMR (282 MHz, $CDCl_3$) δ -63.0 (s, 3 F, CF_3); IR (KBr) ν (cm^{-1}) 3058, 2985, 2923, 1938, 1614, 1597, 1494, 1450, 1408, 1377, 1324, 1304, 1241, 1166, 1149, 1127, 1086, 1068, 1015; MS (EI): m/z (%) 210 ((M-Ts-I)$^+$, 98.87), 141 (100); Anal. Calcd. for $C_{19}H_{16}F_3IO_2S$ (%): C 46.36, H 3.28; Found: C 45.91, H 3.39.

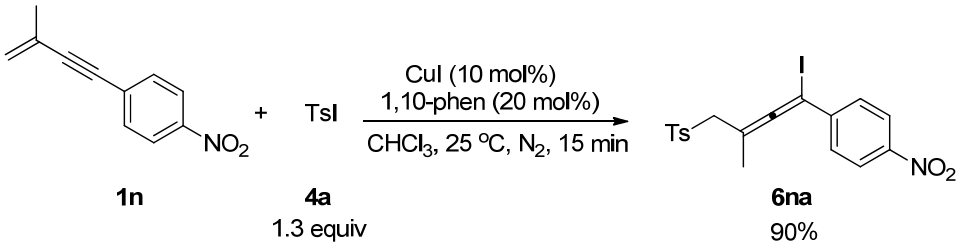
15. Preparation of 3-methyl-1-(4-cyanophenyl)-4-tosylbuta-1,2-dienyl iodide **6ma** (dxy-3-193).



1m **4a** (1.3 equiv) **6ma** 92%

Following **Typical Procedure IV**, the reaction of 1,10-phen (0.0358 g, 0.2 mmol), CuI (0.0191 g, 0.1 mmol), **1m** (0.1671 g, 1 mmol)/$CHCl_3$ (4 mL), **4a** (0.3663 g, 1.3 mmol), and $CHCl_3$ (6 mL) afforded **6ma** (0.4134 g, 92%) (eluent: petroleum ether (60~90 °C)/ether/DCM = 30/15/1 (460 mL) to 20/10/3 (330 mL) to 1/1/1 (450 mL)): solid; m. p. 158.9-160.4 °C (*n*-hexane/DCM); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.79 (d, *J* = 8.1 Hz, 2 H, ArH), 7.55 (d, *J* = 8.4 Hz, 2 H, ArH), 7.49 (d, *J* = 8.4 Hz, 2 H, ArH), 7.32 (d, *J* = 8.1 Hz, 2 H, ArH), 3.95 (d, *J* = 13.5 Hz, 1 H, one proton of SO_2CH_2), 3.81 (d, *J* = 13.5 Hz, 1 H, one proton of SO_2CH_2), 2.43 (s, 3 H, CH_3), 2.03 (s, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 208.0, 145.1, 139.6, 135.9, 132.0, 130.1, 129.4, 128.1, 118.3, 111.7, 95.0, 60.4, 60.1, 21.7, 18.5; IR (KBr) ν (cm^{-1}) 3055, 3040, 3020, 2975, 2922, 2896, 2225, 1933, 1602, 1594, 1558, 1499, 1408, 1401, 1376, 1318, 1304, 1290, 1246, 1181, 1149, 1118, 1086, 1018, 1002; MS (EI): m/z (%) 210 ($(M-I)^+$, 4.09), 91(100); HRMS calcd for $C_{19}H_{16}NIO_2SNa^+$ (M^+ + Na): 471.9839, Found: 471.9838.

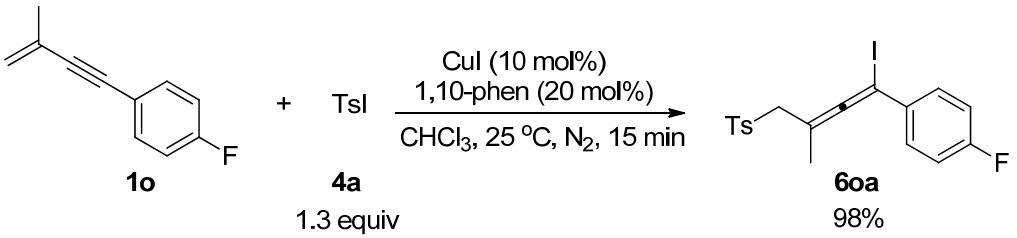
16. Preparation of 3-methyl-1-(4-nitrophenyl)-4-tosylbuta-1,2-dienyl iodide **6na** (syl-3-175, syl-3-126).



1n **4a** (1.3 equiv) **6na** 90%

Following **Typical Procedure IV**, the reaction of 1,10-phen (0.0359 g, 0.2 mmol), CuI (0.0189 g, 0.1 mmol), **1n** (0.1869 g, 1 mmol)/$CHCl_3$ (4 mL), **4a** (0.3668 g, 1.3 mmol), and $CHCl_3$ (6 mL) afforded **6na** (0.4216 g, 90%) (eluent: petroleum ether (60~90 ºC)/ ethyl ether /DCM (5/1/0 (360 mL)) to (5/1/1 (280 mL))): solid; m.p. 131.1-131.5 ºC (ethyl ether/*n*-hexane); ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.10 (d, *J* = 8.7 Hz, 2 H, ArH), 7.80 (d, *J* = 8.1 Hz, 2 H, ArH), 7.51 (d, *J* = 8.7 Hz, 2 H, ArH), 7.33 (d, *J* = 8.1 Hz, 2 H, ArH), 3.97 (d, *J* = 13.5 Hz, 1 H, one proton of SO_2CH_2), 3.84 (d, *J* = 13.8 Hz, 1 H, one proton of SO_2CH_2), 2.43 (s, 3 H, CH_3), 2.04 (s, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 208.4, 147.2, 145.2, 141.4, 135.8, 130.1, 129.6, 128.1, 123.4, 95.1, 60.0, 59.7, 21.6, 18.5; IR (neat) ν (cm^{-1}) 1933, 1598, 1517, 1491, 1449, 1407, 1377, 1343, 1317, 1242, 1184, 1149, 1110, 1086; MS (EI): m/z (%) 342 ($(M-I)^+$, 0.21), 246 (100); Anal. Calcd. for $C_{18}H_{16}INO_4S$ (%): C 46.07, H 3.44, N 2.98; Found: C 45.95, H 3.57, N 2.94 .

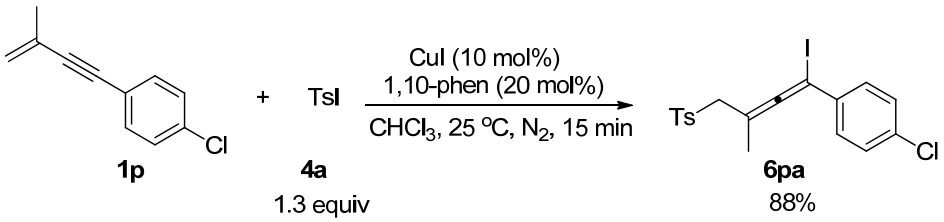
17. Preparation of 3-methyl-1-(4-fluorophenyl)-4-tosylbuta-1,2-dienyl iodide **6oa** (syl-3-128, syl-3-182).



Following **Typical Procedure IV**, the reaction of 1,10-phen (0.0361 g, 0.2 mmol), CuI (0.0189 g, 0.1 mmol), **1o** (0.1601 g, 1 mmol)/$CHCl_3$ (4 mL), **4a** (0.3668 g, 1.3 mmol), and $CHCl_3$ (6 mL) afforded **6oa** (0.4343 g, 98%) (eluent: petroleum ether (60~90 ºC)/ethyl ether/DCM = 30/6/1 (370 mL) to petroleum ether (60~90 ºC)/etnyl

ether = 2/1 (90 mL)): solid; m. p. 82.3-84.4 °C (*n*-hexane/DCM); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.76 (d, *J* = 8.1 Hz, 2 H, ArH), 7.37-7.18 (m, 4 H, ArH), 6.93 (t, *J* = 8.7 Hz, 2 H, ArH), 3.94 (d, *J* = 13.5 Hz, 1 H, one proton of SO_2CH_2), 3.81 (d, *J* = 13.8 Hz, 1 H, one proton of SO_2CH_2), 2.40 (s, 3 H, CH_3), 2.00 (s, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 206.5, 162.4 (d, *J* = 247.4 Hz), 144.9, 135.7, 131.0, 130.4, 130.3, 130.0, 128.1, 115.1, 114.9, 94.0, 61.2, 60.4, 21.6, 18.6; ^{19}F NMR (282 MHz, $CDCl_3$) δ -113.7; IR (KBr) ν (cm^{-1}) 3067, 2989, 2921, 1940, 1596, 1505, 1451, 1407, 1379, 1317, 1303, 1231, 1184, 1117, 1086, 1017; MS (EI): m/z (%) 160 ($(M-Ts-I)^+$, 100); Anal. Calcd. for $C_{18}H_{16}FIO_2S$ (%): C 48.88, H 3.65; Found: C 48.75, H 3.79.

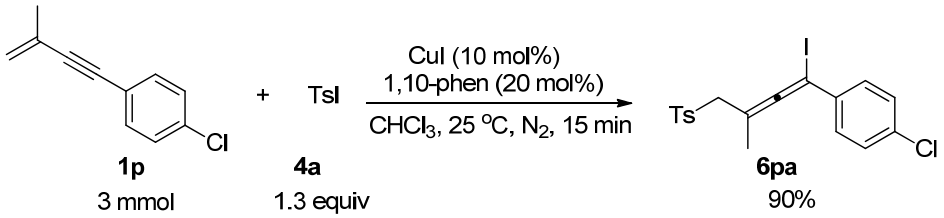
18. Preparation of 3-methyl-1-(4-chlorophenyl)-4-tosylbuta-1,2-dienyl iodide **6pa** (jf-2-189).



Following **Typical Procedure IV**, the reaction of 1,10-phen (0.0358 g, 0.2 mmol), CuI (0.0195 g, 0.1 mmol), **1p** (0.1758 g, 1.0 mmol)/$CHCl_3$ (4 mL), **4a** (0.3661 g, 1.3 mmol), and $CHCl_3$ (6 mL) afforded **6pa** (0.4034 g, 88%) (eluent: petroleum ether (60~90 °C)/ethyl ether = 5/1): solid; m.p. 91.9-93.3 °C (petroleum ether/ ethyl ether); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.76 (d, *J* = 8.1 Hz, 2 H, ArH), 7.34-7.16 (m, 6 H, ArH), 3.95 (d, *J* = 13.5 Hz, 1 H, one proton of SO_2CH_2), 3.80 (d, *J* = 13.8 Hz, 1 H, one proton of SO_2CH_2), 2.41 (s, 3 H, CH_3), 2.01 (s, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 206.7, 144.9, 135.7, 134.1, 133.5, 130.0, 129.9, 128.2, 128.1, 94.2, 61.1, 60.4, 21.6, 18.5; IR

(KBr) ν (cm^{-1}) 2969, 2901, 1938, 1593, 1565, 1488, 1448, 1402, 1379, 1312, 1302, 1291, 1243, 1206, 1184, 1146, 1113, 1085, 1031, 1011; MS (EI): *m/z* (%) 178 ((M-I-Ts)$^+$ (^{37}Cl), 32.38), 176 ((M-I-Ts)$^+$ (^{35}Cl), 100); Anal. Calcd. for $C_{18}H_{16}ClIO_2S$ (%): C 47.13, H 3.52, Found: C 47.13, H 3.61.

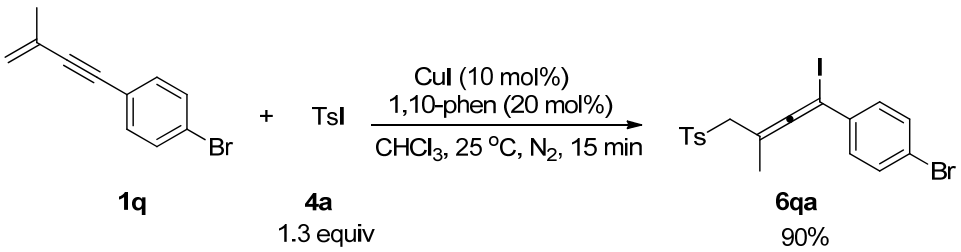
Gram-scale (syl-4-57)**:**



1p 3 mmol; **4a** 1.3 equiv; **6pa** 90%

Following **Typical Procedure IV**, the reaction of 1,10-phen (0.1080 g, 0.6 mmol), CuI (0.0578 g, 0.3 mmol), **1p** (0.5279 g, 3.0 mmol)/$CHCl_3$ (12 mL), **4a** (1.0995 g, 3.9 mmol), and $CHCl_3$ (18 mL) afforded **6pa** (1.2381 g, 90%) (eluent: petroleum ether/ethyl ether/DCM = 10/1/1 (360 mL) to petroleum ether/ethyl ether/DCM = 4/1/1 (150 mL)); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.76 (d, *J* = 8.1 Hz, 2 H, ArH), 7.33-7.14 (m, 6 H, ArH), 3.95 (d, *J* = 13.8 Hz, 1 H, one proton of SO_2CH_2), 3.81 (d, *J* = 13.8 Hz, 1 H, one proton of SO_2CH_2), 2.40 (s, 3 H, CH_3), 2.01 (s, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 206.7, 144.9, 135.6, 134.1, 133.5, 130.0, 129.9, 128.2, 128.1, 94.2, 61.1, 60.3, 21.6, 18.5.

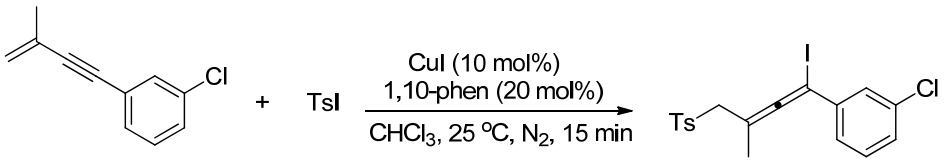
19. Preparation of 3-methyl-1-(4-bromophenyl)-4-tosylbuta-1,2-dienyl iodide **6qa** (syl-3-157, fjj-1-058).



1q **4a** 1.3 equiv **6qa** 90%

Following **Typical Procedure IV**, the reaction of 1,10-phen (0.0361 g, 0.2 mmol), CuI (0.0191 g, 0.1 mmol), **1q** (0.2203 g, 1 mmol)/$CHCl_3$ (4 mL), **4a** (0.3669 g, 1.3 mmol), and $CHCl_3$ (6 mL) afforded **6qa** (0.4521 g, 90%) (eluent: petroleum ether (60~90 ºC)/ ethyl ether /DCM = 10/1/1 (600 mL)): solid; m.p. 96.5-96.9 ºC (ethyl ether/ petroleum ether); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.75 (d, *J* = 8.1 Hz, 2 H, ArH), 7.35 (d, *J* = 8.4 Hz, 2 H, ArH), 7.26 (d, *J* = 7.8 Hz, 2 H, ArH), 7.15 (d, *J* = 8.4 Hz, 2 H, ArH), 3.94 (d, *J* = 13.8 Hz, 1 H, one proton of SO_2CH_2), 3.80 (d, *J* = 13.8 Hz, 1 H, one proton of SO_2CH_2), 2.40 (s, 3 H, CH_3), 2.00 (s, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 206.7, 145.0, 135.6, 134.0, 131.2, 130.2, 130.0, 128.1, 122.4, 94.3, 61.2, 60.3, 21.6, 18.5; IR (KBr) ν (cm^{-1}) 2852, 1938, 1593, 1561, 1485, 1438, 1400, 1379, 1344, 1313, 1302, 1242, 1147, 1108, 1085, 1074, 1008; MS (EI): m/z (%) 222 ((M-Ts-I)$^+$(^{81}Br), 90.91), 220 ((M-Ts-I)$^+$(^{79}Br), 95.61), 115 (100); Anal. Calcd. for $C_{18}H_{16}BrIO_2S$ (%): C 42.96, H 3.20; Found: C 43.02, H 3.31.

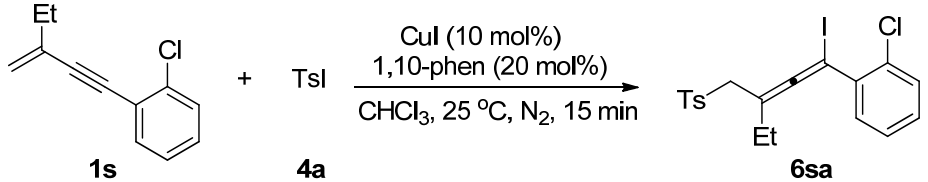
20. Preparation of 3-methyl-1-(3-chlorophenyl)-4-tosylbuta-1,2-dienyl iodide **6ra** (syl-3-158, wxy-3-169)



1r **4a** 1.3 equiv **6ra** 95%

Following Typical Procedure IV, the reaction of 1,10-phen (0.0358 g, 0.2 mmol), CuI (0.0191 g, 0.1 mmol), **1r** (0.1760 g, 1 mmol)/$CHCl_3$ (4 mL), **4a** (0.3670 g, 1.3 mmol), and $CHCl_3$ (6 mL) afforded **6ra** (0.4356 g, 95%) (eluent: petroleum ether (60~90 °C)/ethyl ether /DCM = 10/1/1 (600 mL)): solid; m.p. 95.0-95.3 °C (petroleum ether/ethyl ether); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.76 (d, *J* = 8.4 Hz, 2 H, ArH), 7.35-7.10 (m, 6 H, ArH), 3.94 (d, *J* = 13.8 Hz, 1 H, one proton of SO_2CH_2), 3.81 (d, *J* = 13.8 Hz, 1 H, one proton of SO_2CH_2), 2.38 (s, 3 H, CH_3), 2.01 (s, 3 H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 206.9, 144.9, 137.0, 135.6, 134.1, 130.0, 129.4, 128.8, 128.3, 128.1, 127.0, 94.5, 60.5, 60.3, 21.6, 18.6; IR (KBr) ν (cm^{-1}) 3061, 2984, 2921, 1941, 1589, 1564, 1474, 1407, 1374, 1317, 1147, 1086; MS (EI): m/z (%) 331 ((M-HI)$^+$(^{37}Cl), 6.52), 332 ((M-HI)$^+$(^{35}Cl), 2.07), 191 (100); Anal. Calcd. for $C_{18}H_{16}ClIO_2S$ (%): C 47.13, H 3.52; Found: C 46.89, H 3.61.

21. Preparation of 1-(2-chlorophenyl)-3-(tosylmethyl)penta-1,2-dienyl iodide **6sa** (syl-4-16).

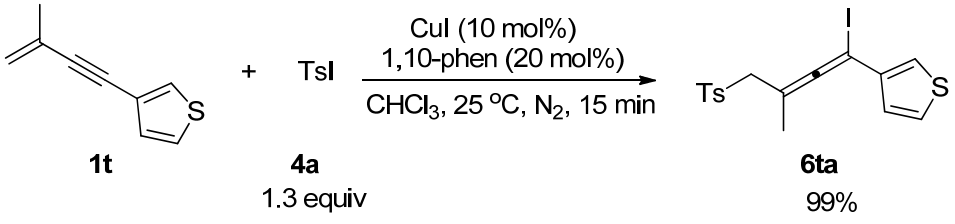


1s **4a** 1.3 equiv **6sa** 90%

Following **Typical Procedure IV**, the reaction of 1,10-phen (0.0365 g, 0.2 mmol),

CuI (0.0192 g, 0.1 mmol), **1s** (0.1907 g, 1 mmol)/$CHCl_3$ (4 mL), **4a** (0.3668 g, 1.3 mmol), and $CHCl_3$ (6 mL) afforded **6sa** (0.4236 g, 90%) (eluent: petroleum ether (60~90 °C)/ethyl ether/DCM = 30/6/1 (370 mL)): solid; m.p. 76.8-78.3 °C (DCM/*n*-hexane); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.65 (d, *J* = 8.1 Hz, 2 H, ArH), 7.35-7.14 (m, 4 H, ArH), 7.11 (d, *J* = 8.1 Hz, 2 H, ArH), 3.86 (s, 2 H, SO_2CH_2), 2.55-2.35 (m, 1 H, one proton of CH_2), 2.30 (s, 3 H, CH_3), 2.27-2.13 (m, 1 H, one proton of CH_2), 1.12 (t, *J* = 7.4 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 205.5, 144.6, 135.6, 135.0, 132.2, 131.0, 129.8, 129.7, 129.5, 128.1, 126.7, 99.4, 59.3, 54.7, 24.9, 21.6, 11.6; IR (KBr) ν (cm^{-1}) 3065, 2976, 2964, 2925, 2871, 1945, 1596, 1472, 1432, 1410, 1376, 1309, 1297, 1265, 1240, 1206, 1184, 1151, 1111, 1086, 1056, 1039; MS (ESI) *m/z*: 497 (M^+(^{37}Cl) + Na), 495 (M^+(^{35}Cl) + Na); Anal. Calcd. for $C_{19}H_{18}ClIO_2S$ (%): C 48.27, H 3.84; Found: C 48.23, H 3.95.

22. Preparation of 3-methyl-1-(thien-3-yl)-4-tosylbuta-1,2-dienyl iodide **6ta** (syl-3-156, ssh-06-076).

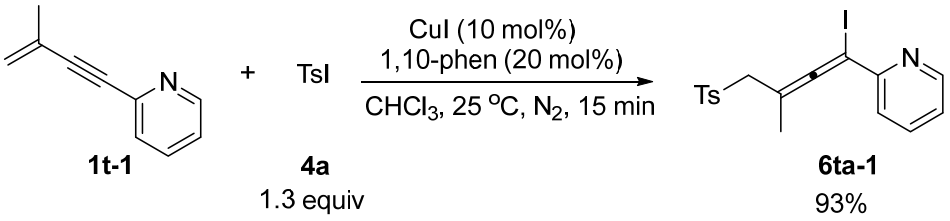


1t **4a** 1.3 equiv **6ta** 99%

Following **Typical Procedure IV**, the reaction of 1,10-phen (0.0362 g, 0.2 mmol), CuI (0.0191g, 0.1 mmol), **1t** (0.1483 g, 1 mmol)/$CHCl_3$ (4 mL), **4a** (0.3667 g, 1.3 mmol), and $CHCl_3$ (6 mL) afforded **6ta** (0.4242 g, 99%) (eluent: petroleum ether (60~90 °C)/ ethyl ether /DCM = 4/1/1 (300 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$)

δ 7.79 (d, J = 8.4 Hz, 2 H, ArH), 7.40-7.25 (m, 3 H, ArH), 7.25-7.15 (m, 1 H, ArH), 6.91 (dd, J_1 = 5.1 Hz, J_2 = 1.4 Hz, 1 H, ArH), 3.92 (d, J = 13.8 Hz, 1 H, one proton of SO_2CH_2), 3.81 (d, J = 13.8 Hz, 1 H, one proton of SO_2CH_2), 2.43 (s, 3 H, CH_3), 1.98 (s, 3 H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 206.4, 144.9, 136.8, 135.9, 130.1, 128.1, 126.7, 126.3, 126.1, 93.8, 60.6, 55.0, 21.6, 18.8; IR (neat) ν (cm^{-1}) 3102, 2985, 2920, 1940, 1596, 1493, 1449, 1401, 1316, 1303, 1220, 1184, 1144, 1086, 1018; MS (EI): m/z (%) 303 ($(M-I)^+$, 3.18), 302 ($(M-HI)^+$, 16.66), 147 (100); HRMS calcd. for $C_{16}H_{15}IO_2S_2Na^+$ (M^+ + Na): 452.9450, found: 452.9450.

23. Preparation of 3-methyl-1-(pyridine -2-yl)-4-tosylbuta-1,2-dien-1-yl iodide **6ta-1** (syl-5-83).

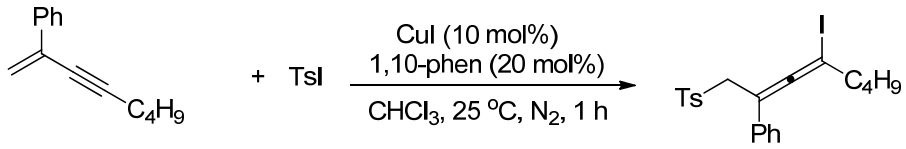


1t-1 **4a** 1.3 equiv **6ta-1** 93%

Following **Typical Procedure IV**, the reaction of CuI (0.0185 g, 0.1 mmol), 1,10-phen (0.0361 g, 0.2 mmol), **1t-1** (0.1430 g, 1 mmol)/$CHCl_3$ (4 mL), **4a** (0.3667 g, 1.3 mmol), and $CHCl_3$ (6 mL) afforded **6ta-1** (0.3963 g, 93%) (eluent: petroleum ether (60~90 °C)/ethyl ether/DCM = 4/1/1 (300 mL) to petroleum ether (60~90 °C)/ethyl ether = 1/1 (300 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.57 (d, J = 4.5 Hz, 1 H, ArH), 7.80 (d, J = 8.4 Hz, 2 H, ArH), 7.57-7.40 (m, 2 H, ArH), 7.30 (d, J = 8.1 Hz, 2 H, ArH), 7.15-7.06 (m, 1 H, ArH), 3.97 (d, J = 13.8 Hz, 1 H, one proton of SO_2CH_2), 3.85 (d, J = 13.8 Hz, 1 H, one proton of SO_2CH_2), 2.41 (s, 3 H, CH_3), 2.04 (s, 3 H, CH_3); ^{13}C NMR (100

MHz, $CDCl_3$) δ 208.5, 151.6, 149.3, 144.9, 136.04, 135.98, 130.0, 128.0, 122.5, 121.3, 95.1, 66.1, 60.2, 21.5, 18.4; IR (neat) ν (cm^{-1}) 3048, 2962, 2922, 2857, 1940, 1596, 1581, 1563, 1467, 1425, 1401, 1376, 1316, 1260, 1147, 1085, 1018; MS (EI): m/z (%) 298 ($(M-I)^+$, 0.68), 270 ($(M-Ts)^+$, 0.54), 142 (100); HRMS calcd. for $C_{17}H_{16}INO_2S$ (M^+): 424.9941, found: 424.9942.

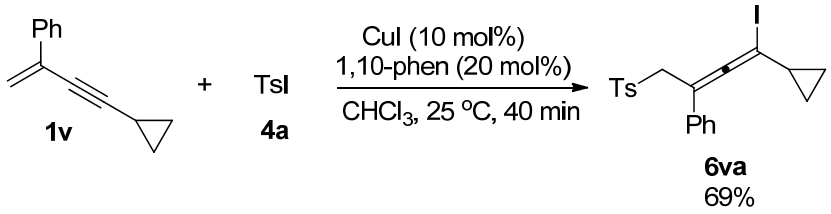
24. Preparation of 2-phenyl-1-tosylocta-2,3-dien-4-yl iodide **6ua** (syl-4-3).



1u **4a** 1.0 equiv **6ua** 84%

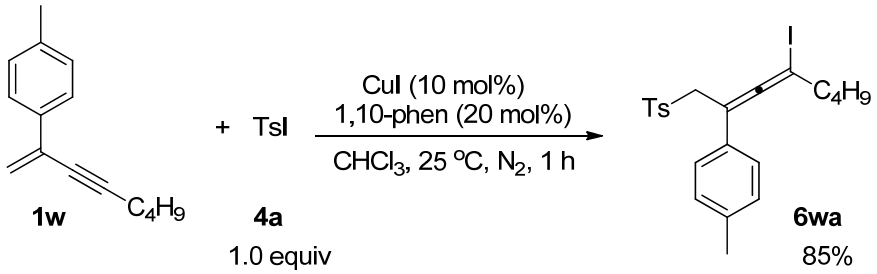
Following **Typical Procedure III**, the reaction of 1,10-phen (0.0361 g, 0.2 mmol), CuI (0.0191 g, 0.1 mmol), **1u** (0.1938 g, purity = 95%, 1 mmol)/$CHCl_3$ (4 mL), **4a** (0.2821 g, 1 mmol), and $CHCl_3$ (6 mL) afforded **6ua** (0.3901 g, 84%) after chromatography on silica gel (eluent: petroleum ether (60~90 °C)/ethyl ether/DCM = 35/7/1 (430 mL)): liquid; ^1H NMR (300 MHz, $CDCl_3$) δ 7.71 (d, *J* = 8.1 Hz, 2 H, ArH), 7.35-7.15 (m, 7 H, ArH), 4.23 (s, 2 H, SCH_2), 2.37 (s, 3 H, CH_3), 2.22 (t, *J* = 7.4 Hz, 2 H, CH_2), 1.52-1.18 (m, 4 H, CH_2 × 2), 0.88 (t, *J* = 7.2 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 204.6, 144.8, 135.7, 132.8, 129.8, 128.5, 127.8, 126.8, 97.1, 66.9, 57.3, 39.8, 31.2, 21.5, 13.7; IR (neat) ν (cm^{-1}) 3059, 3028, 2956, 2929, 2870, 1936, 1596, 1493, 1451, 1403, 1377, 1320, 1303, 1233, 1154, 1133, 1086; MS (ESI) *m/z*: 489 (M^+ + Na); HRMS calcd. for $C_{21}H_{23}IO_2SNa^+$ (M^+ + Na): 489.0356; Found: 489.0359.

25. Preparation of 1-cyclopropyl-3-phenyl-4-tosylbuta-1,2-dienyl iodide **6va** (syl-4-45).



Following **Typical Procedure III**, the reaction of 1,10-phen (0.0182 g, 0.1 mmol), CuI (0.0095 g, 0.05 mmol), **1v** (0.0889, 0.5 mmol)/$CHCl_3$ (2 mL), **4a** (0.1415 g, 0.5 mmol), and $CHCl_3$ (3 mL) afforded **6va** (0.1642 g, 69%) (eluent: petroleum ether (60~90 °C)/ethyl ether/DCM = 40/8/1 (490 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.72 (d, *J* = 8.4 Hz, 2 H, ArH), 7.32-7.15 (m, 7 H, ArH), 4.24 (d, *J* = 15.6 Hz, 1 H, one proton of SO_2CH_2), 4.19 (d, *J* = 14.7 Hz, 1 H, one proton of SO_2CH_2), 2.37 (s, 3 H, CH_3), 1.37-1.23 (m, 1 H, CH), 0.81-0.70 (m, 2 H, CH_2), 0.69-0.51 (m, 2 H, CH_2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 204.5, 144.7, 135.7, 132.7, 129.8, 128.5, 128.4, 127.9, 126.7, 97.9, 71.4, 57.2, 21.5, 19.3, 9.7; IR (neat) ν (cm^{-1}) 1933, 1596, 1494, 1451, 1404, 1319, 1303, 1229, 1153, 1134, 1086, 1025; MS (EI) *m/z*: 450 (M^+, 0.32), 246 ($(M\text{-Ph-I})^+$, 24.22), 91 (100); Anal. Calcd. for $C_{20}H_{19}IO_2S$ (%): C 53.34, H 4.25; Found: C 53.22, H 4.43.

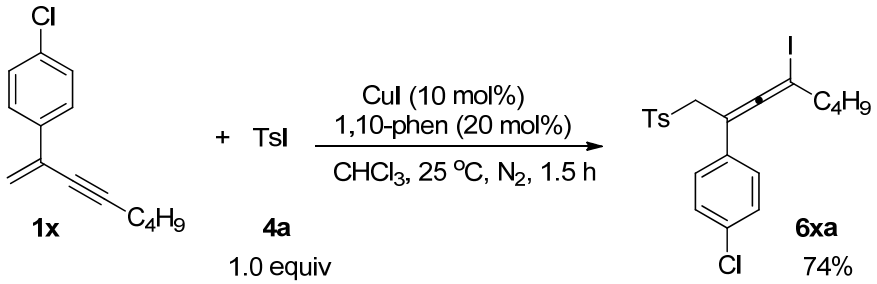
26. Preparation of 2-(4-methylphenyl)-1-tosylocta-2,3-dien-4-yl iodide **6wa** (syl-4-6).



Following **Typical Procedure III**, the reaction of 1,10-phen (0.0362 g, 0.2 mmol), CuI (0.0195 g, 0.1 mmol), **1w** (0.1984 g, 1 mmol)/$CHCl_3$ (4 mL), **4a** (0.2820 g, 1 mmol),

and $CHCl_3$ (6 mL) afforded **6wa** (0.4080 g, 85%) eluent: petroleum ether (60~90 °C)/ethyl ether/DCM = 30/6/1 (370 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.72 (d, *J* = 8.1 Hz, 2 H, ArH), 7.27 (d, *J* = 8.1 Hz, 2 H, ArH), 7.20 (d, *J* = 8.1 Hz, 2 H, ArH), 7.11 (d, *J* = 8.4 Hz, 2 H, ArH), 4.22 (s, 2 H, SO_2CH_2), 2.40 (s, 3 H, CH_3), 2.32 (s, 3 H, CH_3), 2.19 (t, *J* = 7.5 Hz, 2 H, CH_2), 1.49-1.18 (m, 4 H, CH_2 × 2), 0.87 (t, *J* = 7.2 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 204.4, 144.8, 138.0, 135.8, 129.84, 129.79, 129.3, 128.6, 126.8, 97.2, 66.9, 57.4, 39.9, 31.2, 21.6, 21.1, 13.7; IR (neat) ν (cm^{-1}) 3027, 2956, 2925, 2857, 1935, 1597, 1511, 1494, 1456, 1422, 1401, 1378, 1321, 1303, 1235, 1188, 1156, 1134, 1108, 1087; MS (ESI) *m/z*: 503 (M^+ + Na); HRMS calcd. for $C_{22}H_{25}IO_2SNa^+$ (M^+ +Na): 503.0512; Found: 503.0513.

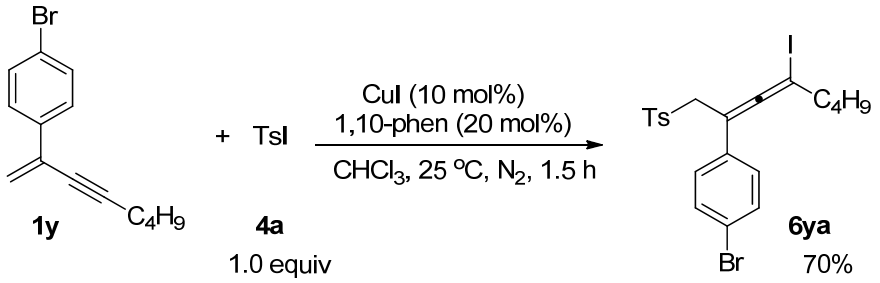
27. Preparation of 2-(4-chlorophenyl)-1-tosylocta-2,3-dien-4-yl iodide **6xa** (syl-4-13).



Following **Typical Procedure III**, the reaction of 1,10-phen (0.0363 g, 0.2 mmol), CuI (0.0192 g, 0.1 mmol), **1x** (0.2274 g, purity = 96%, 1 mmol)/$CHCl_3$ (4 mL), **4a** (0.2824 g, 1 mmol), and $CHCl_3$ (6 mL) afforded **6xa** (0.3710 g, 74%) (eluent: petroleum ether (60~90 °C)/ethyl ether/DCM = 30/6/1 (370 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.71 (d, *J* = 8.4 Hz, 2 H, ArH), 7.30-7.22 (m, 6 H, ArH), 4.20 (s, 2 H, SCH_2), 2.41 (s, 3 H, CH_3), 2.22 (t, *J* = 7.4 Hz, 2 H, CH_2), 1.49-1.18 (m, 4 H, CH_2 × 2), 0.88 (t, *J* = 7.1 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 204.6, 145.0, 135.7, 133.8, 131.5,

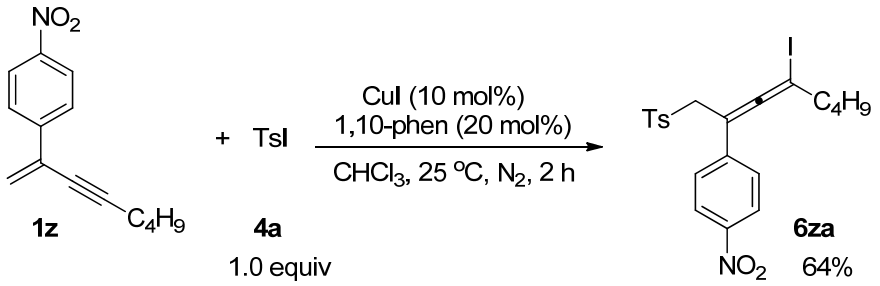
129.9, 128.8, 128.5, 128.1, 96.2, 67.1, 57.4, 39.7, 31.2, 21.6, 21.5, 13.7; IR (neat) ν (cm^{-1}) 2956, 2929, 2871, 1937, 1596, 1492, 1464, 1418, 1401, 1377, 1321, 1303, 1235, 1184, 1154, 1134, 1087, 1012; MS (ESI) *m/z*: 525 (M^+(^{37}Cl) + Na), 523 (M^+(^{35}Cl) + Na); HRMS calcd. for $C_{21}H_{22}{}^{35}ClIO_2SNa^+$ (M^+ + Na): 522.9966; Found: 522.9965.

28. Preparation of 2-(4-bromophenyl)-1-tosylocta-2,3-dien-4-yl iodide **6ya** (syl-4-18).



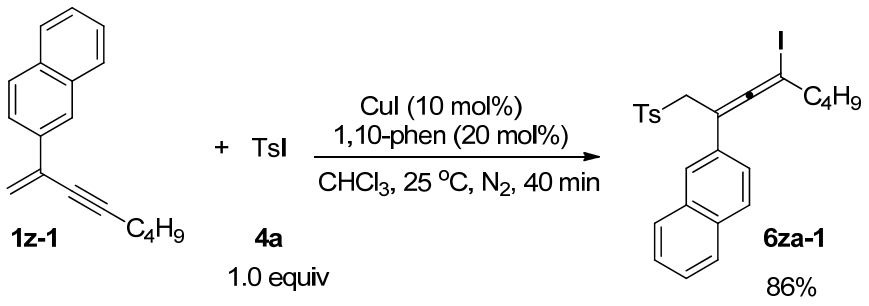
Following **Typical Procedure III**, the reaction of 1,10-phen (0.0360 g, 0.2 mmol), CuI (0.0193 g, 0.1 mmol), **1y** (0.2622 g, 1 mmol)/$CHCl_3$ (4 mL), **4a** (0.2821 g, 1 mmol), and $CHCl_3$ (6 mL) afforded **6ya** (0.3812 g, 70%) (eluent: petroleum ether (60~90 °C)/ethyl ether/DCM = 30/6/1 (370 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.71 (d, *J* = 8.1 Hz, 2 H, ArH), 7.41 (d, *J* = 8.7 Hz, 2 H, ArH), 7.28 (d, *J* = 7.5 Hz, 2 H, ArH), 7.16 (d, *J* = 8.7 Hz, 2 H, ArH), 4.19 (s, 2 H, SO_2CH_2), 2.41 (s, 3 H, CH_3), 2.23 (t, *J* = 7.4 Hz, 2 H, CH_2), 1.52-1.15 (m, 4 H, CH_2 × 2), 0.88 (t, *J* = 7.2 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 204.6, 145.0, 135.7, 132.0, 131.7, 129.9, 128.5, 128.4, 122.0, 96.3, 67.1, 57.3, 39.7, 31.2, 21.6, 21.5, 13.6; IR (neat) ν (cm^{-1}) 2955, 2928, 2860, 1933, 1596, 1488, 1463, 1416, 1371, 1320, 1300, 1233, 1155, 1133, 1086, 1072, 1003; MS (ESI) *m/z*: 569 (M^+(^{81}Br) + Na), 567 (M^+(^{79}Br) + Na); HRMS calcd. for $C_{21}H_{22}{}^{79}BrIO_2SNa^+$ (M^+ + Na): 566.9461; Found: 566.9461.

29. Preparation of 2-(4-nitrophenyl)-1-tosylocta-2,3-dien-4-yl iodide **6za** (syl-4-37).



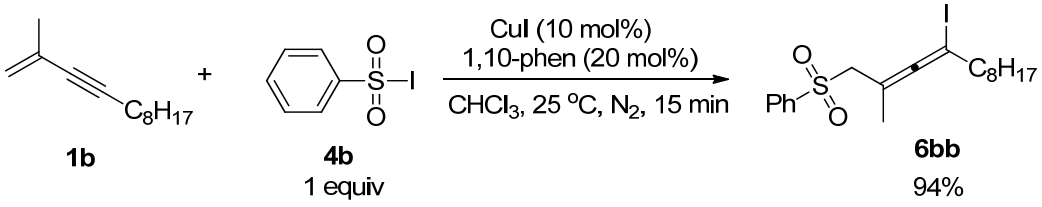
Following **Typical Procedure III**, the reaction of 1,10-phen (0.0361 g, 0.2 mmol), CuI (0.0192 g, 0.1 mmol), **1z** (0.2415 g, purity = 95%, 1 mmol)/$CHCl_3$ (4 mL), **4a** (0.2823 g, 1 mmol), and $CHCl_3$ (6 mL) afforded **6za** (0.3269 g, 64%) (eluent: petroleum ether (60~90 °C)/ethyl ether/DCM = 30/6/1 (370 mL) to 15/5/1 (210 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.15 (d, *J* = 8.7 Hz, 2 H, ArH), 7.73 (d, *J* = 8.1 Hz, 2 H, ArH), 7.48 (d, *J* = 9.0 Hz, 2 H, ArH), 7.31 (d, *J* = 8.1 Hz, 2 H, ArH), 4.26 (s, 2 H, SO_2CH_2), 2.41 (s, 3 H, CH_3), 2.27 (t, *J* = 7.4 Hz, 2 H, CH_2), 1.54-1.17 (m, 4 H, CH_2 × 2), 0.89 (t, *J* = 7.2 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 205.7, 146.7, 145.3, 140.0, 135.5, 130.0, 128.4, 127.5, 123.8, 95.4, 67.3, 57.0, 39.2, 31.1, 21.6, 21.5, 13.6; IR (neat) ν (cm^{-1}) 2956, 2930, 2870, 1929, 1594, 1518, 1494, 1464, 1423, 1402, 1342, 1322, 1303, 1235, 1186, 1154, 1134, 1109, 1086, 1034, 1014; MS (ESI) *m/z*: 534 (M^+ + Na); HRMS calcd. for $C_{21}H_{22}INO_4SNa^+$ (M^+ + Na): 534.0206; Found: 534.0209.

30. Preparation of 2-(naphthalen-2-yl)-1-tosylocta-2,3-dien-4-yl iodide **6za-1** (syl-4-28).



Following **Typical Procedure III**, the reaction of 1,10-phen (0.0355 g, 0.2 mmol), CuI (0.0187 g, 0.1 mmol), **1z-1** (0.2342 g, 1 mmol)/$CHCl_3$ (4 mL), **4a** (0.2825 g, 1 mmol), and $CHCl_3$ (6 mL) afforded **6za-1** (0.4457 g, 86%) (eluent: petroleum ether (60~90 °C)/ethyl ether/DCM = 30/6/1 (370 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.83-7.64 (m, 5 H, ArH), 7.56 (s, 1 H, ArH), 7.48-7.33 (m, 3 H, ArH), 7.12 (d, *J* = 8.1 Hz, 2 H, ArH), 4.36 (d, *J* = 15.6 Hz, 1 H, one proton of SO_2CH_2), 4.30 (d, *J* = 14.7 Hz, 1 H, one proton of SO_2CH_2), 2.29 (t, *J* = 7.4 Hz, 2 H, CH_2), 2.20 (s, 3 H, CH_3), 1.57-1.20 (m, 4 H, CH_2 × 2), 0.88 (t, *J* = 7.2 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 205.1, 144.7, 135.6, 133.0, 132.5, 130.0, 129.6, 128.5, 128.14, 128.11, 127.4, 126.4, 126.3, 125.5, 124.6, 97.4, 67.1, 57.4, 39.8, 31.1, 21.5, 21.3, 13.6; IR (neat) ν (cm^{-1}) 3055, 2956, 2929, 2870, 1934, 1596, 1515, 1504, 1464, 1405, 1377, 1320, 1303, 1233, 1154, 1135, 1107, 1086, 1014; MS (ESI) *m/z*: 539 (M^+ + Na); HRMS calcd. for $C_{25}H_{25}IO_2SNa^+$ (M^+ + Na): 539.0512; Found: 539.0512.

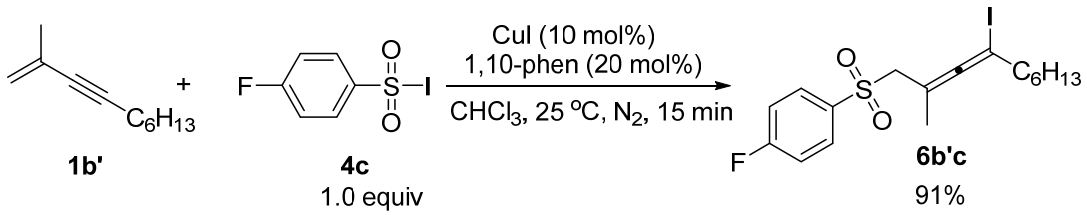
31. Preparation of 2-methyl-1-(phenylsulfonyl)dodeca-2,3-dien-4-yl iodide **6bb** (syl-3-194).



Following **Typical Procedure III**, the reaction of 1,10-phen (0.0360 g, 0.2 mmol),

CuI (0.0191 g, 0.1 mmol), **1b** (0.1782 g, 1 mmol)/$CHCl_3$ (4 mL), **4b** (0.2682 g, 1 mmol), and $CHCl_3$ (6 mL) afforded **6bb** (0.4219 g, 94%) (eluent: petroleum ether (60~90 ^oC)/ethyl ether/DCM = 30/6/1 (370 mL)): liquid; ^1H NMR (300 MHz, $CDCl_3$) δ 7.91 (d, J = 7.8 Hz, 2 H, ArH), 7.75-7.50 (m, 3 H, ArH), 3.82 (d, J = 13.5 Hz, 1 H, one proton of SO_2CH_2), 3.74 (d, J = 13.8 Hz, 1 H, one proton of SO_2CH_2), 2.00 (t, J = 6.9 Hz, 2 H, CH_2), 1.86 (s, 3 H, CH_3), 1.40-1.10 (m, 12 H, CH_2 × 6), 0.89 (t, J = 6.8 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 203.9, 138.8, 133.8, 129.3, 128.3, 91.7, 63.5, 60.8, 40.2, 31.8, 29.2, 29.1, 28.8, 28.1, 22.6, 18.6, 14.1; IR (neat) ν (cm^{-1}) 2959, 2925, 2854, 1953, 1447, 1320, 1308, 1242, 1153, 1126, 1085; MS (ESI) *m/z*: 469 (M^+ + Na); HRMS calcd. for $C_{19}H_{27}IO_2SNa^+$ (M^+ + Na): 469.0669; Found: 469.0670.

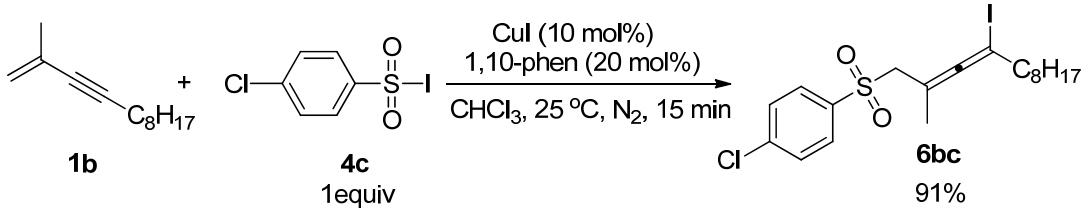
32. Preparation of 2-methyl-1-((4-fluorophenyl)sulfonyl)deca-2,3-dien-4-yl iodide **6b'c** (syl-5-92).



Following **Typical Procedure III**, the reaction of CuI (0.0192 g, 0.1 mmol), 1,10-phen (0.0365 g, 0.2 mmol), **1b'** (0.1501 g, 1 mmol)/$CHCl_3$ (4 mL), **4c** (0.2865 g, 1 mmol), and $CHCl_3$ (6 mL) afforded **6b'c** (0.3969 g, 91%) (eluent: petroleum ether (60~90 ^oC)/ethyl ether = 5/1 (240 mL)): liquid; ^1H NMR (400 MHz, $CDCl_3$) δ 7.93 (dd, J_1 = 8.4 Hz, J_2 = 5.2 Hz, 2 H, ArH), 7.26 (t, J = 8.6 Hz, 2 H, ArH), 3.80 (d, J = 14.0 Hz, 1 H, one proton of SO_2CH_2), 3.75 (d, J = 13.6 Hz, 1 H, one proton of SO_2CH_2), 2.08 (t,

J = 7.0 Hz, 2 H, CH_2), 1.87 (s, 3 H, CH_3), 1.42-1.10 (m, 8 H, CH_2 × 4), 0.89 (t, J = 7.0 Hz, 3 H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.9, 165.9 (d, J = 254.4 Hz), 134.8 (d, J = 3.2 Hz), 131.2 (d, J = 9.5 Hz), 116.7 (d, J = 22.1 Hz), 91.7, 63.4, 61.0, 40.3, 31.3, 28.8, 27.8, 22.5, 18.5, 13.9; IR (neat) ν (cm^{-1}) 2956, 2929, 2856, 1955, 1592, 1494, 1457, 1404, 1323, 1291, 1236, 1086; MS (EI) *m/z*: 436 (M^+, 0.26), 309 ($(M-I)^+$, 5.03), 79 (100); HRMS calcd. for $C_{17}H_{22}FO_2S$ $(M-I)^+$: 309.1319; Found: 309.1319.

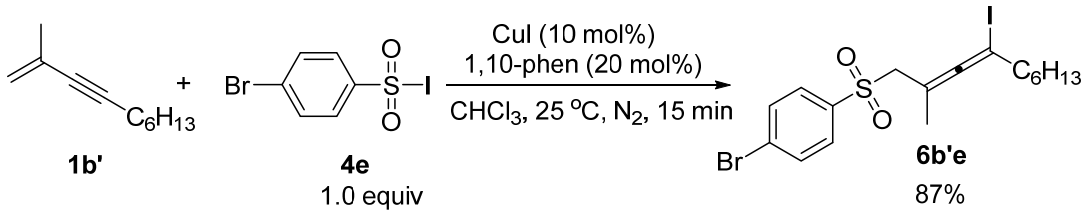
33. Preparation of 2-methyl-1-((4-chlorophenyl)sulfonyl)dodeca-2,3-dien-4-yl iodide **6bc** (syl-4-25).



Following **Typical Procedure III**, the reaction of 1,10-phen (0.0360 g, 0.2 mmol), CuI (0.0195 g, 0.1 mmol), **1b** (0.1779 g, 1 mmol)/$CHCl_3$ (4 mL), **4c** (0.3020 g, 1 mmol), and $CHCl_3$ (6 mL) afforded **6bc** (0.4364 g, 91%) (eluent: petroleum ether (60~90 °C)/ethyl ether/DCM = 25/5/1 (310 mL)): liquid; ^1H NMR (300 MHz, $CDCl_3$) δ 7.85 (d, J = 8.7 Hz, 2 H, ArH), 7.55 (d, J = 8.7 Hz, 2 H, ArH), 3.82 (d, J = 13.8 Hz, 1 H, one proton of SO_2CH_2), 3.76 (d, J = 13.8 Hz, 1 H, one proton of SO_2CH_2), 2.05 (t, J = 6.9 Hz, 2 H, CH_2), 1.87 (s, 3 H, CH_3), 1.38-1.10 (m, 12 H, CH_2 × 6), 0.88 (t, J = 6.6 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 203.8, 140.5, 137.0, 129.7, 129.6, 91.5, 63.3, 60.8, 40.2, 31.6, 29.05, 28.99, 28.8, 28.1, 22.5, 18.5, 14.0; IR (neat) ν (cm^{-1}) 3088, 2925, 2854, 1955, 1582, 1476, 1457, 1395, 1323, 1280, 1242, 1156, 1125, 1088, 1013; MS

(ESI) *m/z*: 521 ($M^+(^{37}Cl)$ + K), 519 ($M^+(^{35}Cl)$ + K); HRMS calcd. for $C_{19}H_{26}{}^{35}ClIO_2SNa^+$ (M^+ + Na): 503.0279; Found: 503.0284.

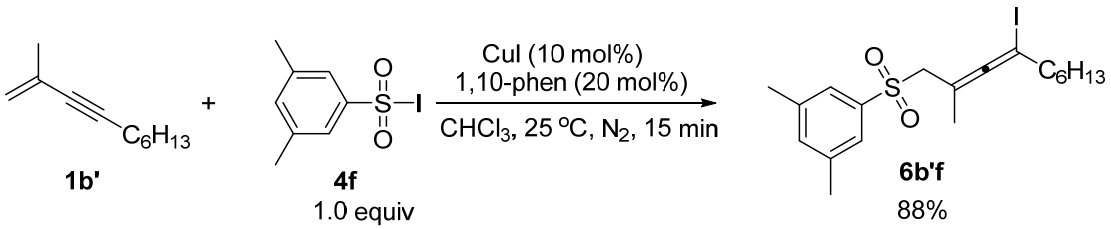
34. Preparation of 2-methyl-1-((4-bromophenyl)sulfonyl)deca-2,3-dien-4-yl iodide **6b'e** (syl-5-89).



Following **Typical Procedure III**, the reaction of CuI (0.0196 g, 0.1 mmol), 1,10-phen (0.0357 g, 0.2 mmol), **1b'** (0.1505 g, 1 mmol)/$CHCl_3$ (4 mL), **4e** (0.3481 g, 1 mmol), and $CHCl_3$ (6 mL) afforded **6b'e** (0.4337 g, 87%) (eluent: petroleum ether (60~90 °C)/ethyl ether = 5/1 (240 mL)): liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77 (d, *J* = 8.4 Hz, 2 H, ArH), 7.72 (d, *J* = 8.8 Hz, 2 H, ArH), 3.80 (d, *J* = 13.6 Hz, 1 H, one proton of SO_2CH_2), 3.74 (d, *J* = 14.0 Hz, 1 H, one proton of SO_2CH_2), 2.05 (dt, J_1 = 7.0 Hz, J_2 = 2.4 Hz, 2 H, CH_2), 1.87 (s, 3 H, CH_3), 1.40-1.10 (m, 8 H, CH_2 × 4), 0.89 (t, *J* = 7.2 Hz, 3 H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.9, 137.6, 132.8, 129.9, 129.3, 91.6, 63.4, 60.9, 40.3, 31.3, 28.9, 27.9, 22.5, 18.5, 14.0; IR (neat) ν (cm^{-1}) 2956, 2929, 2856, 1592, 1494, 1466, 1404, 1323, 1291, 1236, 1150, 1086, 1013; MS (EI) *m/z*: 496 (M^+, 0.08), 371 (($M(^{81}Br)$-I)$^+$, 0.74), 369 (($M(^{79}Br)$-I)$^+$, 0.82), 79 (100); HRMS calcd. for $C_{17}H_{22}{}^{79}BrO_2S$ (M-I)$^+$: 369.0518; Found: 369.0519.

35. Preparation of 2-methyl-1-((3,5-dimethylphenyl)sulfonyl)deca-2,3-dien-4-yl iodide

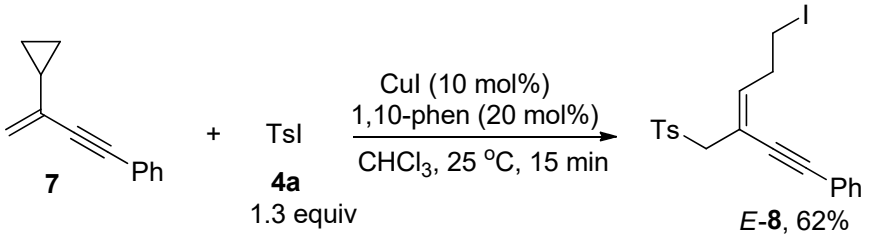
6b'f (syl-5-94).



Following **Typical Procedure III**, the reaction of CuI (0.0191 g, 0.1 mmol), 1,10-phen (0.0357 g, 0.2 mmol), **1b'** (0.1501 g, 1 mmol)/$CHCl_3$ (4 mL), **4f** (0.2962 g, 1 mmol), and $CHCl_3$ (6 mL) afforded **6b'f** (0.3945 g, 88%) (eluent: petroleum ether (60~90 °C)/ethyl ether = 5/1 (240 mL)): liquid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 (s, 2 H, ArH), 7.25 (s, 1 H, ArH), 3.78 (d, *J* = 13.6 Hz, 1 H, one proton of SO_2CH_2), 3.71 (d, *J* = 13.6 Hz, 1 H, one proton of SO_2CH_2), 2.40 (s, 6 H, CH_3 × 2), 2.05 (t, *J* = 7.0 Hz, 2 H, CH_2), 1.86 (s, 3 H, CH_3), 1.38-1.15 (m, 8 H, CH_2 × 4), 0.89 (t, *J* = 7.0 Hz, 3 H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.8, 139.3, 138.6, 135.5, 125.7, 91.9, 63.2, 60.8, 40.3, 31.3, 28.8, 27.8, 22.5, 21.2, 18.6, 14.0; IR (neat) ν (cm^{-1}) 2954, 2925, 2856, 1955, 1607, 1456, 1378, 1320, 1302, 1270, 1146, 1104, 1025; MS (EI) *m/z*: 446 (M^+, 0.34), 319 ($(M-I)^+$, 6.59), 79 (100); HRMS calcd. for $C_{19}H_{27}IO_2S$ (M^+): 446.0771; Found: 446.0770.

Mechanistic studies

Preparation of (*E*)-6-phenyl-4-(tosylmethyl) hex-3-en-5-yn-1-yl iodide *E*-**8** (syl-4-48).

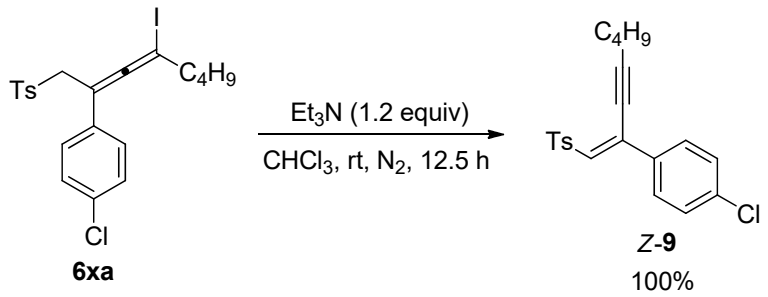


Following **Typical Procedure IV**, the reaction of 1,10-phen (0.0179 g, 0.1 mmol),

CuI (0.0094 g, 0.05 mmol), **7** (0.0828 g, 0.5 mmol)/$CHCl_3$ (2 mL), **4a** (0.1834 g, 0.65 mmol), and $CHCl_3$ (3 mL) afforded *E*-**8** (0.1379 g, 62%) (eluent: petroleum ether (60~90 °C)/ethyl ether/DCM = 10/2/1 (520 mL)): solid; m.p. 156.0-156.7 °C (DCM/hexane); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.81 (d, *J* = 7.8 Hz, 2 H, ArH), 7.40-7.10 (m, 7 H, ArH), 5.92 (t, *J* = 7.2 Hz, 1 H, =CH), 3.94 (s, 2 H, SO_2CH_2), 3.15 (t, *J* = 6.9 Hz, 2 H, CH_2), 2.93 (q, *J* = 7.1 Hz, 2 H, CH_2), 2.32 (s, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 144.9, 144.3, 135.3, 131.4, 129.6, 128.9, 128.6, 128.1, 122.2, 113.8, 95.7, 85.3, 63.0, 34.6, 21.4, 2.4; IR (KBr) ν (cm^{-1}) 2979, 2926, 2202, 1596, 1491, 1441, 1426, 1407, 1381, 1299, 1262, 1248, 1196, 1185, 1177, 1155, 1133, 1084, 1055, 1019; MS (EI) *m/z*: 450 (M^+, 0.34), 167 (100); Anal. Calcd. for $C_{20}H_{19}IO_2S$ (%): C 53.34, H 4.25; Found: C 53.19, H 4.34.

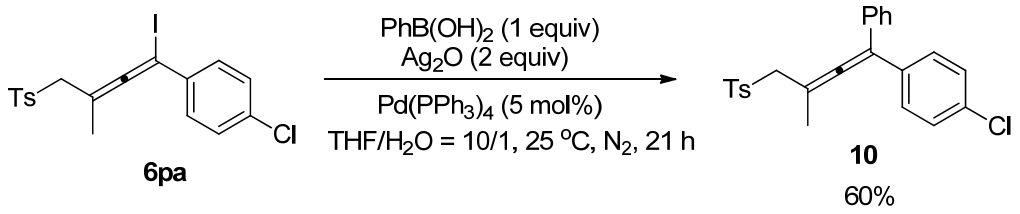
Synthetic applications

1. Preparation of (*Z*)- 2-(4-chlorophenyl)-1-tosyl-1-octen-3-yne **9** (syl-4-43).



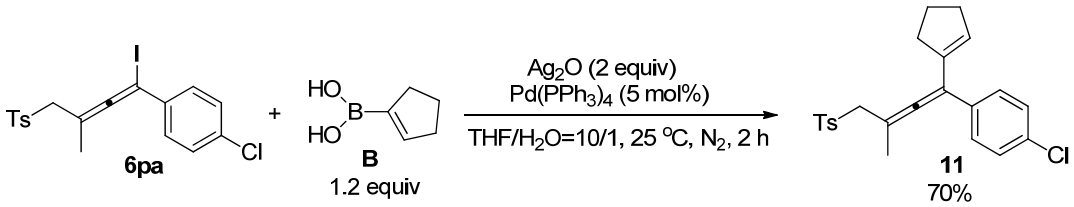
To a flame-dried Schlenk tube were added **6xa** (0.0505 g, 0.1 mmol)/$CHCl_3$ (1 mL). Then Et_3N (0.0125 g, 0.12 mmol) was added under N_2 atmosphere. The reaction mixture was stirred at room temperature for 12.5 hours as monitored by TLC. The resulting mixture was eluted with DCM (10 mL). After evaporation of the solvent under reduced pressure at room temperature, the yield of the crude product was determined by ^1H NMR analysis using mesitylene (9.2 μL) as the internal standard (100% by NMR). Then the crude residue was transferred to the column with the help of eluent (petroleum ether (60~90 ºC)/ethyl ether/DCM = 4/1/1) and purified by a flash chromatography on silica gel to afford *Z*-**9** (0.0374 g, 100%) (eluent: petroleum ether/ethyl ether/DCM = 20/4/1 (250 mL)): solid; m. p. 104.0-104.9 ºC (*n*-hexane/DCM); ^1H NMR (300 MHz, $CDCl_3$) δ 7.91 (d, *J* = 8.1 Hz, 2 H, ArH), 7.55 (d, *J* = 8.4 Hz, 2 H, ArH), 7.38-7.27 (m, 4 H, ArH), 7.03 (s, 1 H, =CH), 2.53 (t, *J* = 7.2 Hz, 2 H, CH_2), 2.44 (s, 3 H, CH_3), 1.73-1.55 (m, 2 H, CH_2), 1.55-1.47 (m, 2 H, CH_2), 0.96 (t, *J* = 7.1 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 144.3, 138.3, 136.6, 135.2, 134.3, 132.7, 129.5, 128.8, 128.3, 127.9, 109.4, 75.2, 30.0, 22.1, 21.6, 19.8, 13.5; IR (KBr) ν (cm^{-1}) 3052, 2957, 2931, 2871, 2232, 1593, 1579, 1551, 1490, 1465, 1422, 1401, 1375, 1318, 1302, 1223, 1182, 1148, 1085, 1013; MS (EI): m/z (%) 374 (M^+(^{37}Cl), 0.82), 372 (M^+(^{35}Cl), 1.62), 139 (100); Anal. Calcd. for $C_{21}H_{21}ClO_2S$ (%): C 67.64, H 5.68; Found: C 67.68, H 5.72.

2. Preparation of 1-(4-chlorophenyl)-1-phenyl-3-methyl-4-tosylbuta-1,2-diene **10** (syl-4-35).[14]



Typical Procedure V: To a Schlenk tube were added $PhB(OH)_2$ (0.0241 g, 0.2 mmol), Ag_2O (0.0931 g, 0.4 mmol), **6pa** (0.0920 g, 0.2 mmol), THF (1 mL), H_2O (0.1 mL), and $Pd(PPh_3)_4$ (0.0117 g, 0.01 mmol) under N_2 atmosphere. The resulting mixture was stirred at 25 ºC for 21 hours as monitored by TLC and filtrated through a pad of silica gel eluted with ethyl ether (10 mL × 3). After evaporation of the solvent under reduced pressure at room temperature, the yield of the crude product was determined by ^1H NMR analysis using mesitylene (18.4 μL) as the internal standard (65% by NMR). Then the crude residue was transferred to the column with the help of solvent (petroleum ether /DCM = 5/1) and purified by a flash chromatography on silica gel to afford **10** (0.0494 g, 60%) (eluent: petroleum ether (60~90 ºC)/ethyl ether/DCM = 30/6/1 (300 mL)): liquid; ^1H NMR (300 MHz, $CDCl_3$) δ 7.67 (d, *J* = 8.1 Hz, 2 H, ArH), 7.33-7.18 (m, 5 H, ArH), 7.15-7.06 (m, 4 H, ArH), 7.04 (d, *J* = 8.4 Hz, 2 H, ArH), 3.96 (d, *J* = 14.1 Hz, 1 H, one proton of SO_2CH_2), 3.90 (d, *J* = 13.8 Hz, 1 H, one proton of SO_2CH_2), 2.35 (s, 3 H, CH_3), 2.05 (s, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 207.4, 144.5, 135.7, 135.2, 134.6, 133.1, 129.8, 129.6, 128.44, 128.38, 128.35, 128.0, 127.6, 109.2, 93.2, 61.3, 21.6, 19.1; IR (neat) ν (cm^{-1}) 3059, 3024, 2985, 2922, 1948, 1597, 1489, 1447, 1403, 1374, 1317, 1303, 1242, 1166, 1146, 1087, 1014, 1002; MS (ESI) *m/z*: 433 (M^+(^{37}Cl) + Na), 431 (M^+(^{35}Cl) + Na); HRMS calcd. for $C_{24}H_{21}{}^{35}ClO_2SNa^+$ (M^+ + Na): 431.0843; Found: 431.0841.

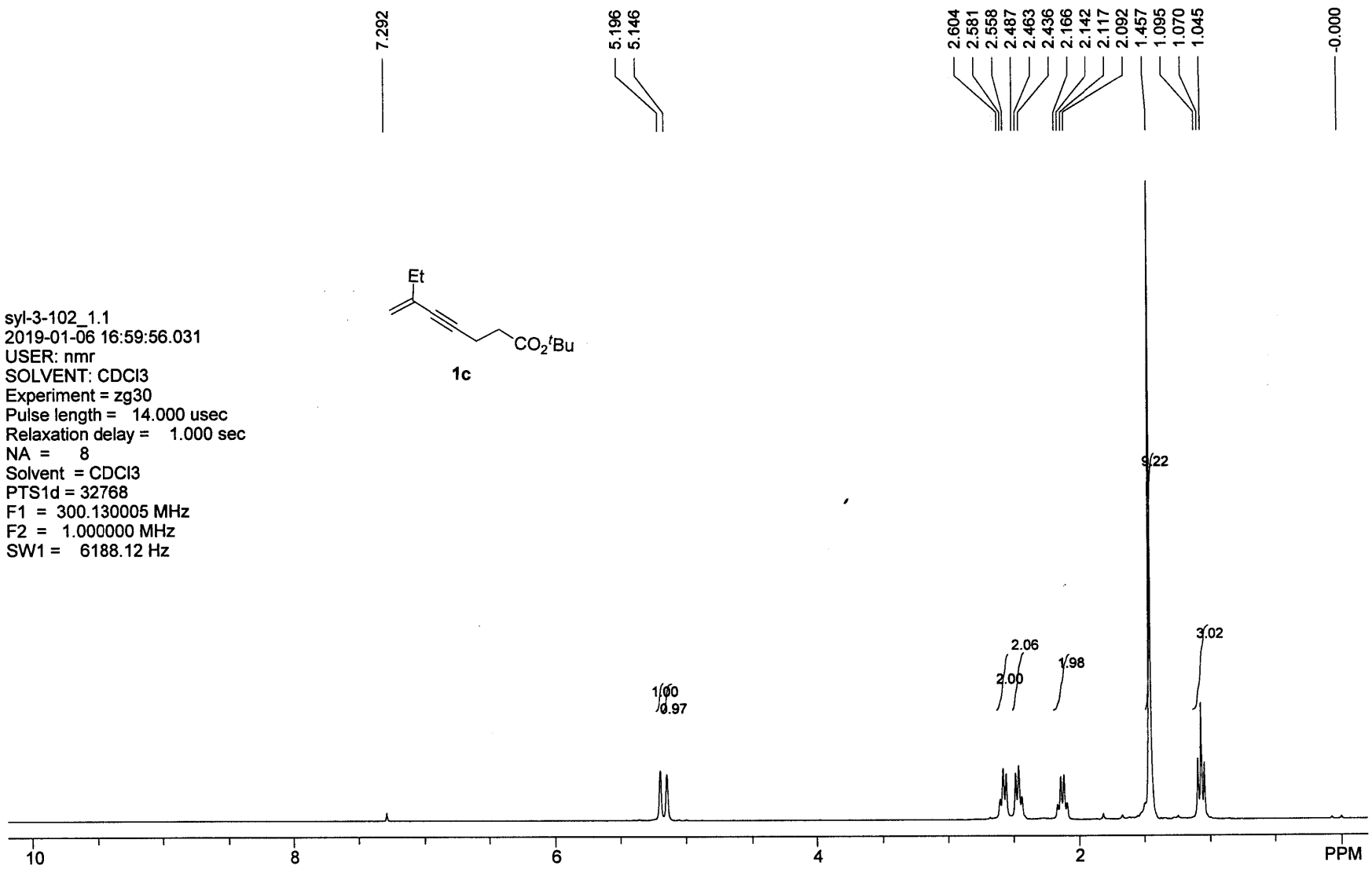
3. Preparation of 1-(4-chlorophenyl)-1-(cyclopentenyl)-3-methyl-4-tosylbuta-1,2-diene **11** (syl-4-60).

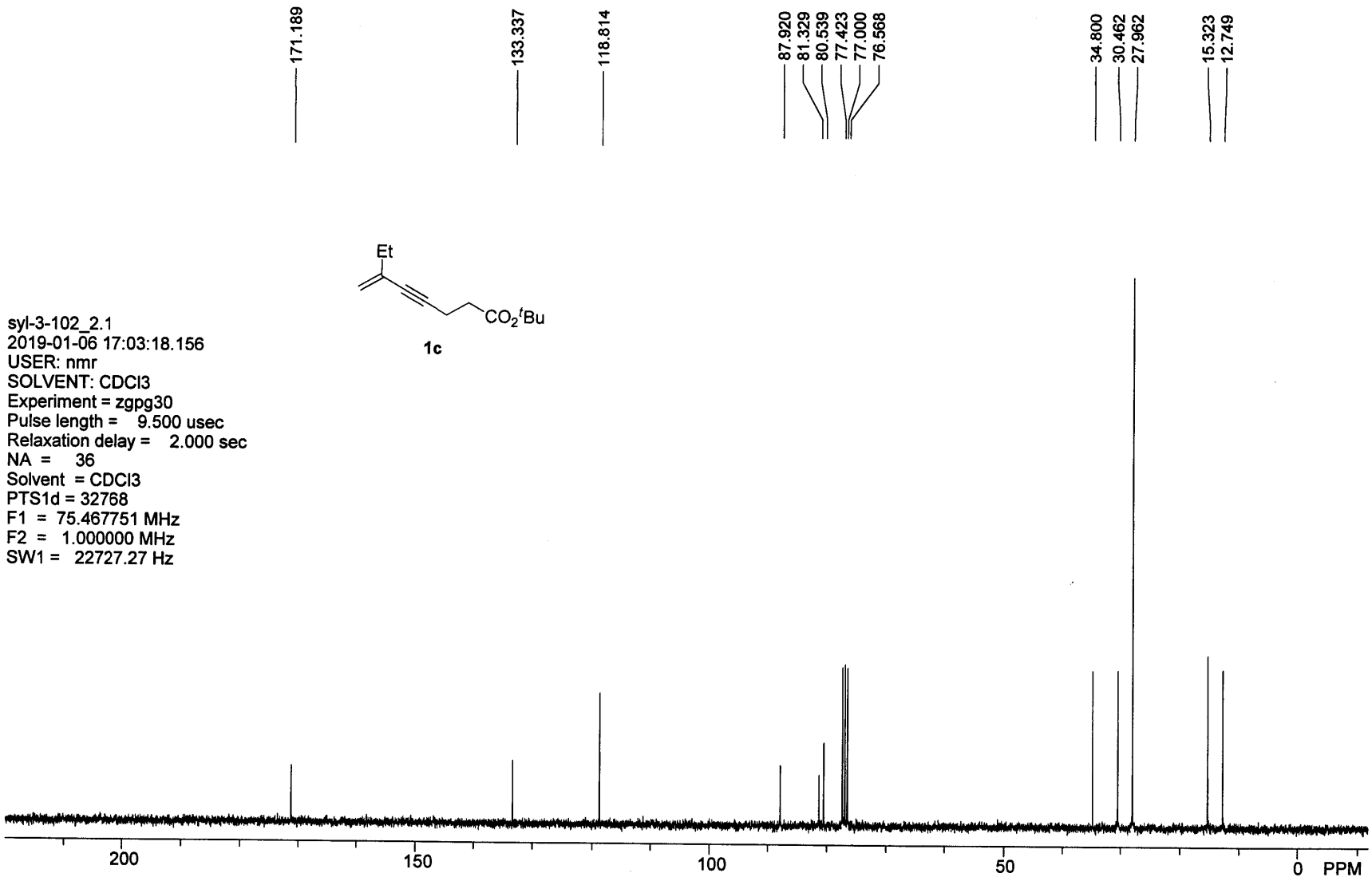


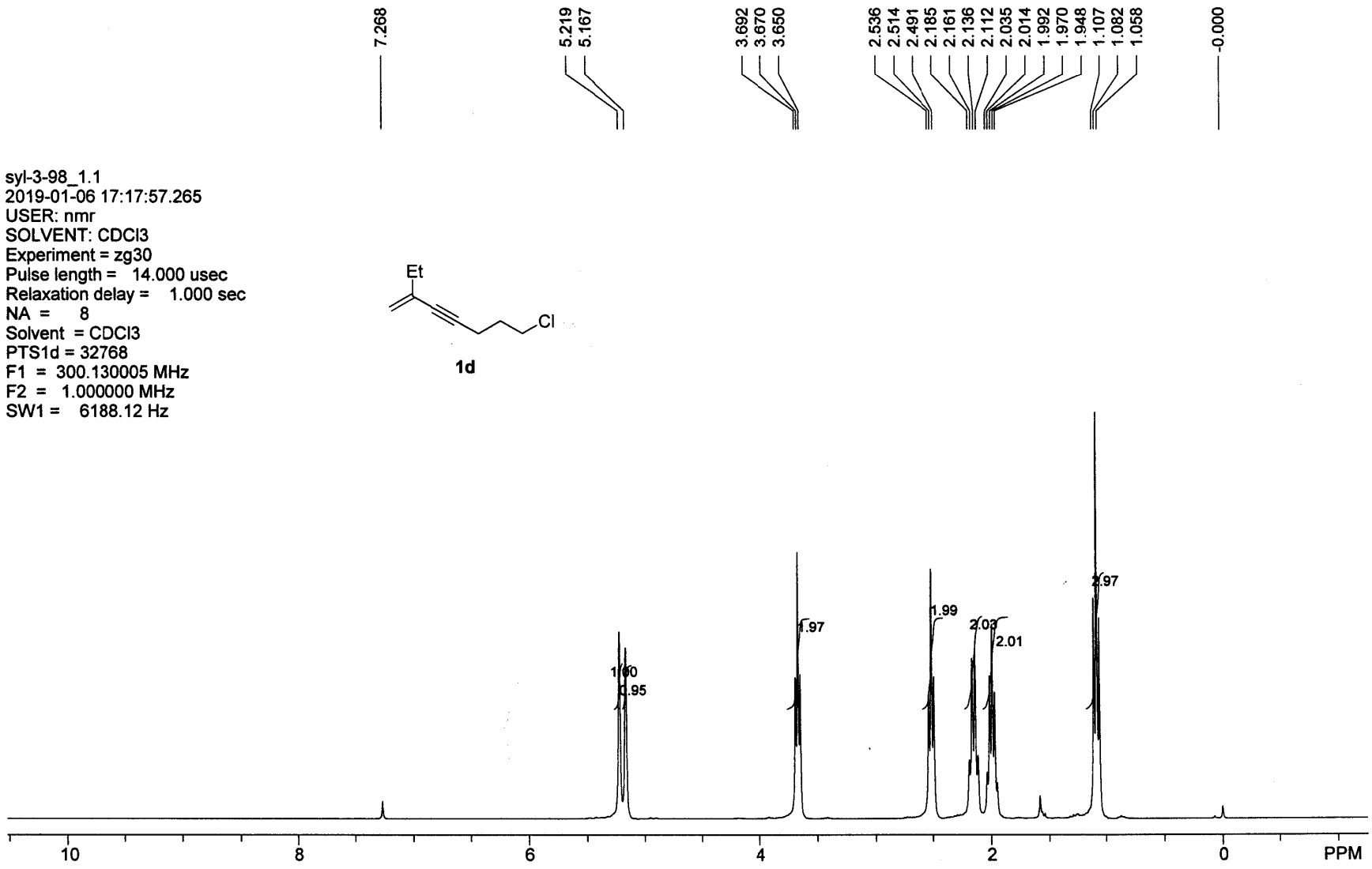
Following **Typical Procedure V**, the reaction of $Pd(PPh_3)_4$ (0.0116 g, 0.01 mmol), Ag_2O (0.0930 g, 0.4 mmol), **6pa** (0.0920 g, 0.2 mmol), **B** (0.0270 g, 0.24 mmol), THF (1 mL), and H_2O (0.1 mL) afforded **11** (0.0562 g, 70%) (eluent: petroleum ether (60~90 °C)/ethyl ether/DCM = 20/4/1 (250 mL)): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.65 (d, *J* = 8.1 Hz, 2 H, ArH), 7.22 (d, *J* = 8.7 Hz, 2 H, ArH), 7.12 (d, *J* = 8.1 Hz, 2 H, ArH), 7.03 (d, *J* = 8.4 Hz, 2 H, ArH), 5.49 (s, 1 H, =CH), 3.87 (d, *J* = 13.8 Hz, 1 H, one proton of SO_2CH_2), 3.80 (d, *J* = 13.8 Hz, 1 H, one proton of SO_2CH_2), 2.45-2.35 (m, 5 H, CH_3 + CH_2), 2.35-2.10 (m, 2 H, CH_2), 1.97 (s, 3 H, CH_3), 1.94-1.80 (m, 2 H, CH_2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 208.1, 144.5, 138.4, 135.3, 134.8, 132.8, 130.3, 130.0, 129.5, 128.1, 106.4, 91.7, 61.6, 34.1, 33.5, 23.0, 21.6, 19.2; IR (neat) ν (cm^{-1}) 3061, 2955, 2922, 2844, 1936, 1597, 1489, 1440, 1402, 1374, 1317, 1303, 1243, 1152, 1137, 1087, 1039, 1015, 1001; MS (EI) *m/z*: 400 (M^+(^{37}Cl), 0.36), 398 (M^+ (^{35}Cl), 0.50), 334 (100); Anal. Calcd for $C_{23}H_{23}ClO_2S$ (%): C 69.24, H 5.81; Found: C 69.23, H 5.84.

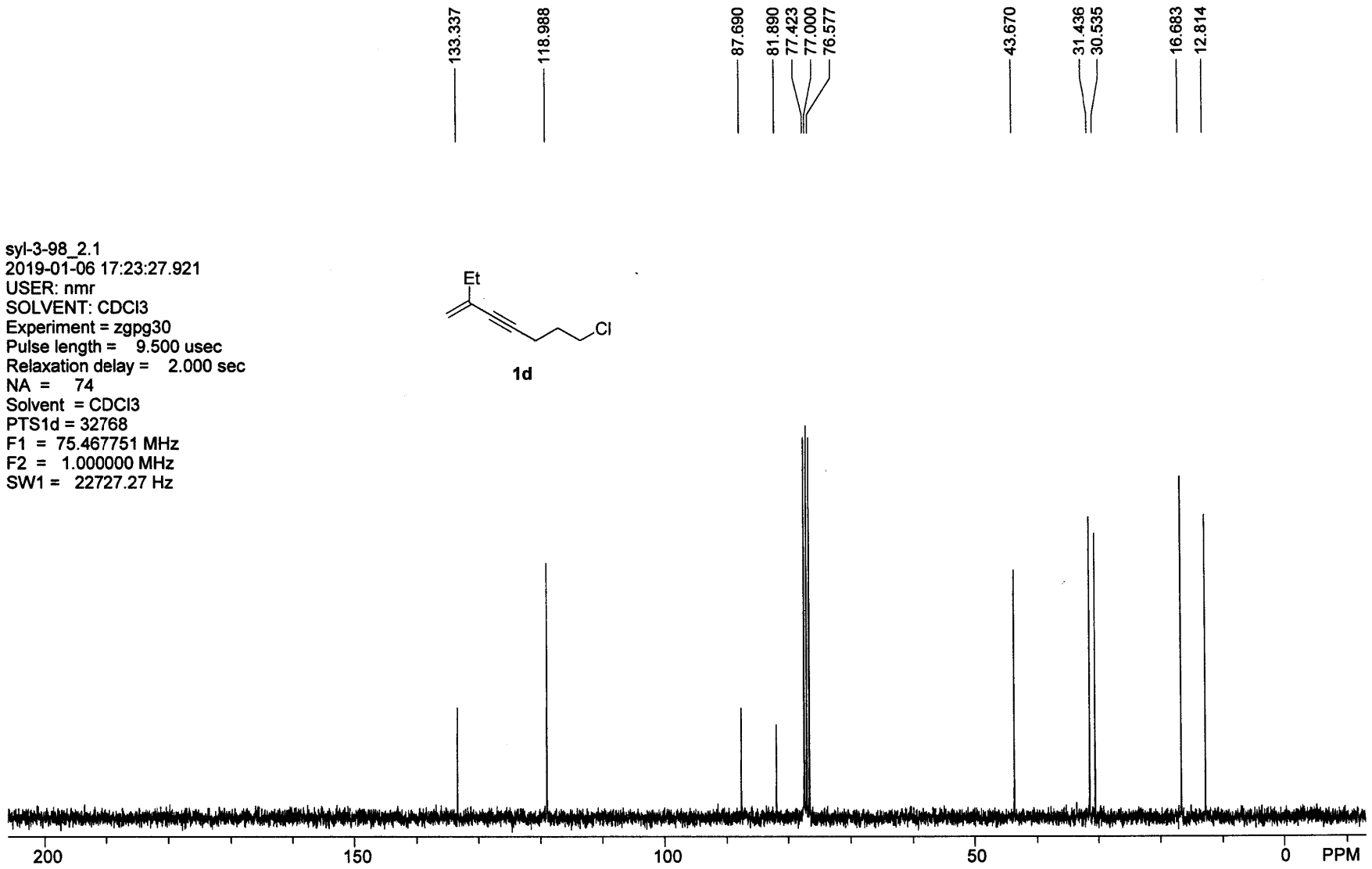
References

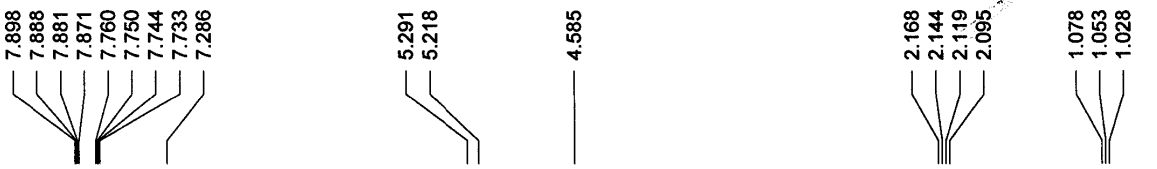
1. S. O. Mihigo, W. Mammo, M. Bezabih, K. Andrae-Marobela, B. M. Abegaz, *Bioorg. Med. Chem.* **2010**, *18*, 2464.
2. N. Lu, Z. Zhang, N. Ma, C. Wu, G. Zhang, Q. Liu, T. Liu, *Org. Lett*. **2018**, *20*, 4318.
3. L. Fu, X. Bao, S. Li, L. Wang, Z. Liu, W. Chen, Q. Xia, G. Liang, *Tetrahedron* **2017**, *73*, 2504.
4. C. Luken, C. Moberg, *Org. Lett*. **2008**, *10*, 2505.
5. M. Feuerstein, L. Chahen, H. Doucet, M. Santelli, *Tetrahedron* **2016**, *62*, 112.
6. A. S. K. Hashmi, P. Sinha, *Adv. Synth. Catal.* **2004**, *346*, 432.
7. H. Kinoshita, T. Ishikawa, K. Miura, *Org. Lett*. **2011**, *13*, 6192.
8. Y. Zhang, B. Yu, B. Gao, T. Zhang, H. Huang, *Org. Lett.* **2019**, *21*, 535.
9. A. Pourjavadi, G. Bagheri Marandi, *J. Chem. Research (S)*. **2002**, *552*.
10. M. Rubina, M. Conley, V. Gevorgyan, *J. Am. Chem. Soc.* **2006**, *128*, 5818.
11. H. Chen, J. Zhang, D. Zhigang Wang, *Org. Lett.* **2015**, *17*, 2098.
12. X. Zhu, W. Deng, M. Chiou, C. Ye, W. Jian, Y. Zeng, Y. Jiao, L. Ge, Y. Li, X. Zhang, H. Bao, *J. Am. Chem. Soc.* **2019**, *141*, 548.
13. P. A. Peixoto, R. Severin, C. Tseng, D. Y.-K. Chen, *Angew. Chem. Int. Ed.* **2011**, *50*, 3013.
14. Vayer, M.; Guillot, R.; Bour, C.; Gandon, V. *J. Org. Chem.* **2018**, *83*, 11309.


7.292
5.196
5.146
2.604
2.581
2.558
2.487
2.463
2.436
2.166
2.142
2.117
2.092
1.457
1.095
1.070
1.045
-0.000
Et
CO2tBu
1c
syl-3-102_1.1
2019-01-06 16:59:56.031
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
9.22
3.02
2.06
1.98
2.00
1.00
0.97
10
8
6
4
2
PPM

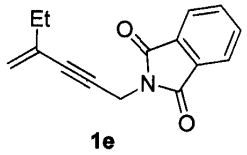

171.189
133.337
118.814
87.920
81.329
80.539
77.423
77.000
76.568
34.800
30.462
27.962
15.323
12.749
Et
CO2tBu
1c
syl-3-102_2.1
2019-01-06 17:03:18.156
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 36
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
200
150
100
50
0
PPM


7.268
5.219
5.167
3.692
3.670
3.650
2.536
2.514
2.491
2.185
2.161
2.136
2.112
2.035
2.014
1.992
1.970
1.948
1.107
1.082
1.058
-0.000
syl-3-98_1.1
2019-01-06 17:17:57.265
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
Et
Cl
1d
1.00
0.95
1.97
1.99
2.03
2.01
2.97
10
8
6
4
2
0
PPM

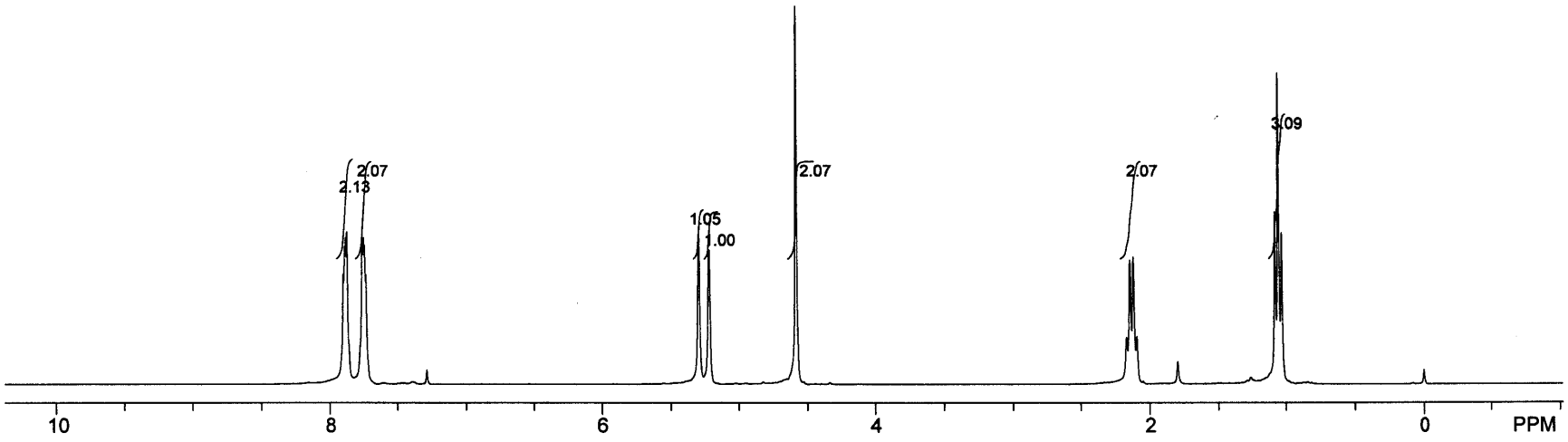

133.337
118.988
87.690
81.890
77.423
77.000
76.577
43.670
31.436
30.535
16.683
12.814
syl-3-98_2.1
2019-01-06 17:23:27.921
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 74
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
Et
Cl
1d
200
150
100
50
0
PPM

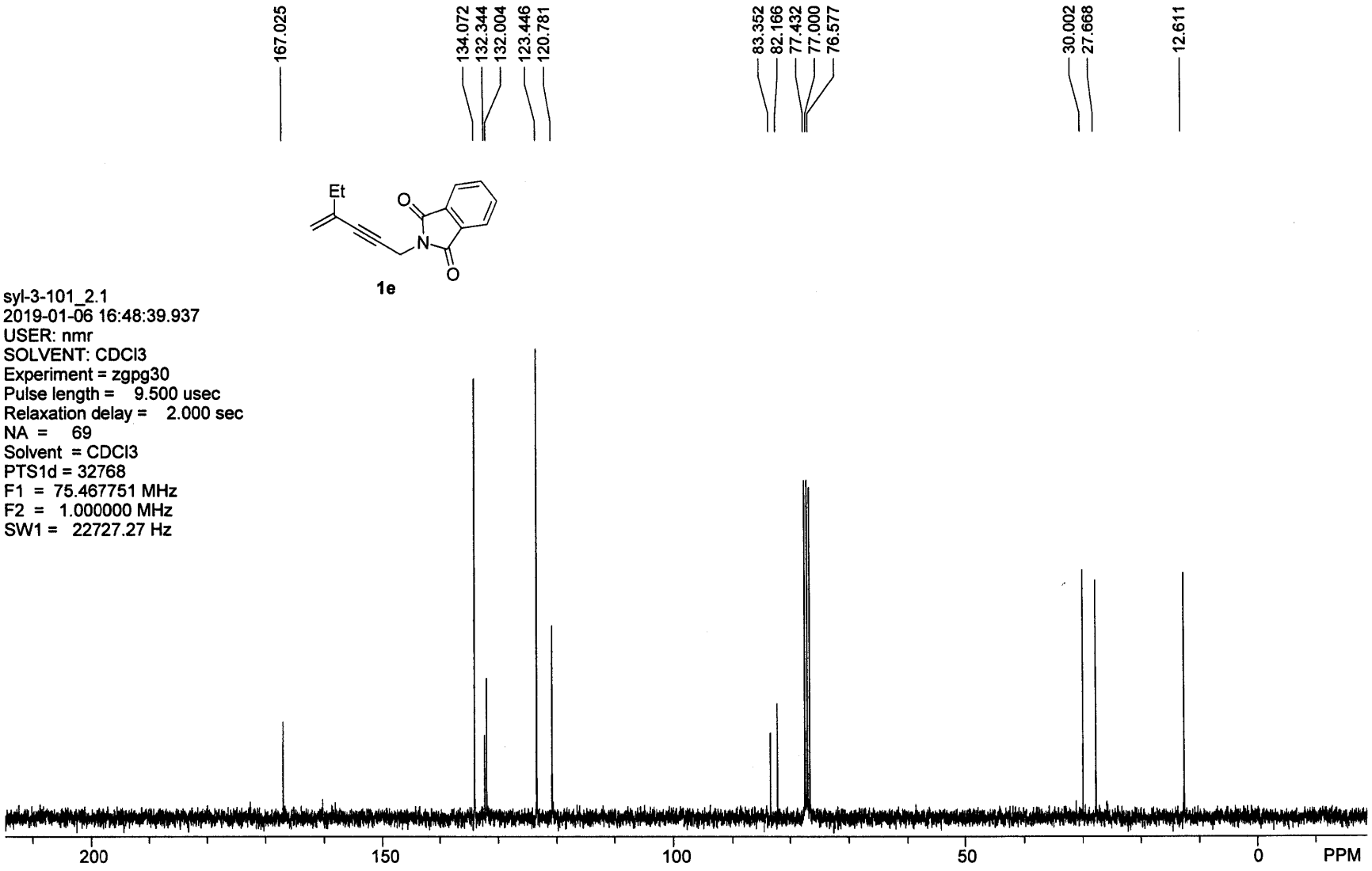


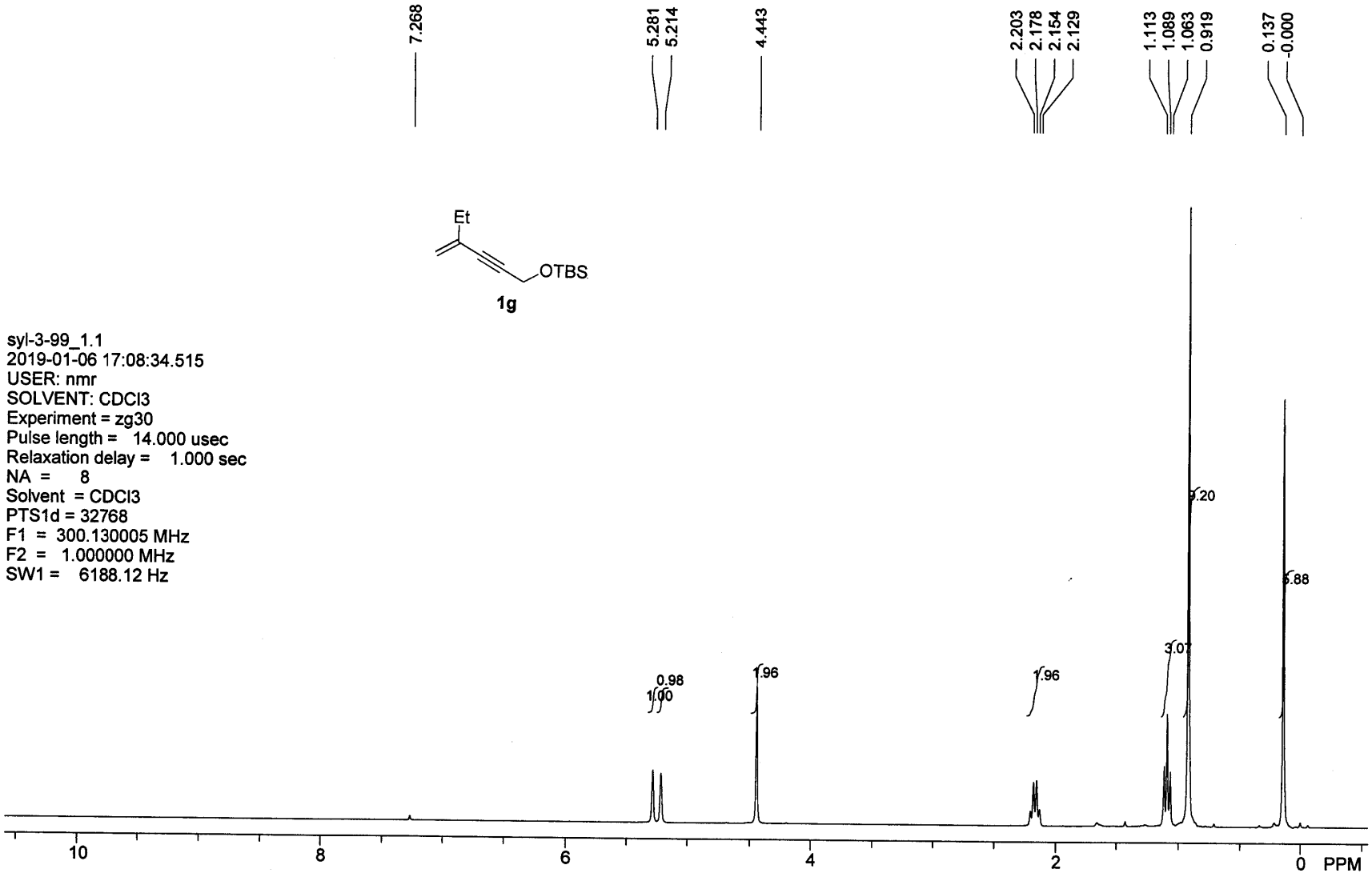
syl-3-101_1.1
2019-01-06 16:43:36.640
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz

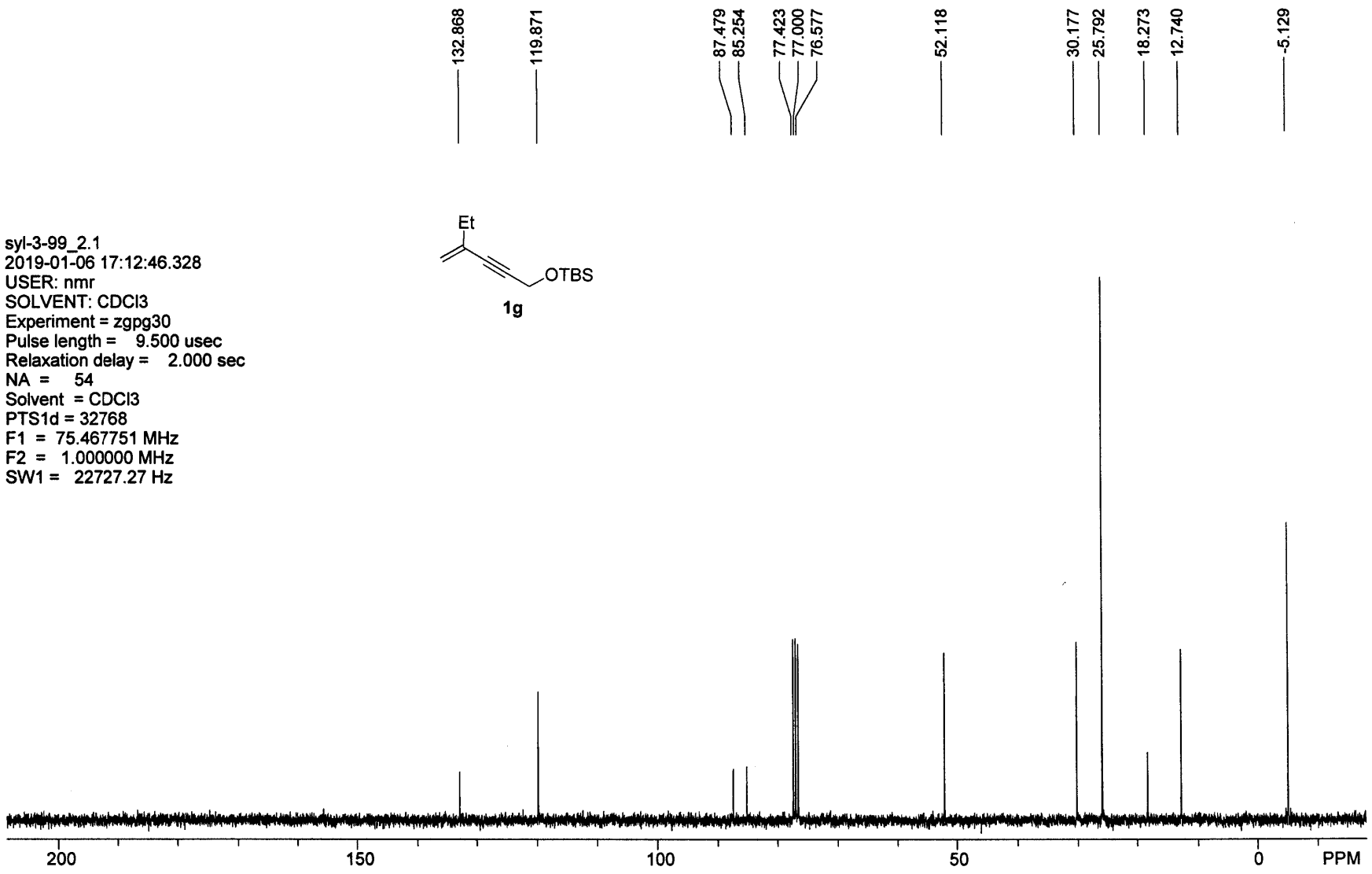


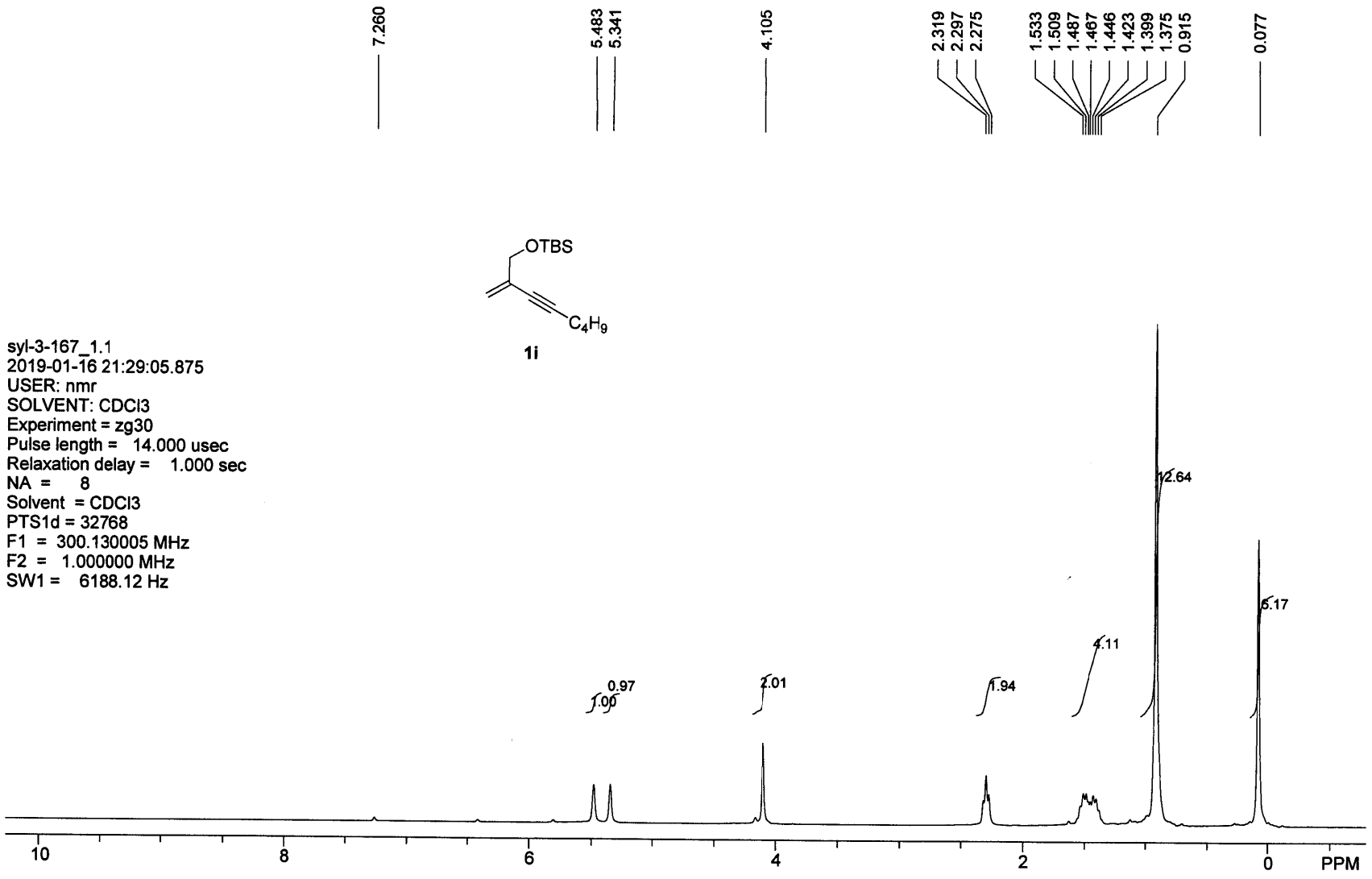
1e

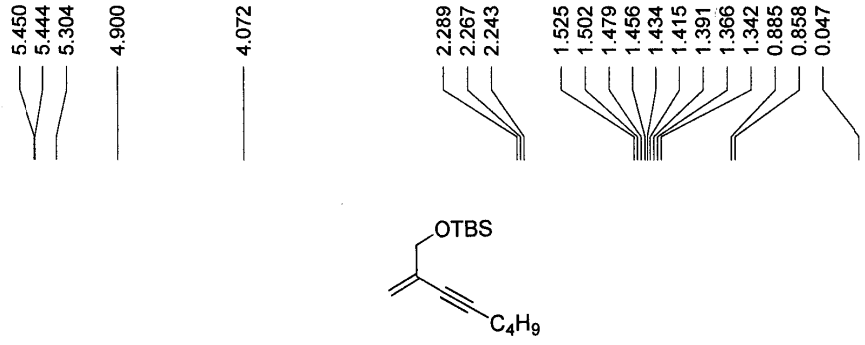



167.025
134.072
132.344
132.004
123.446
120.781
83.352
82.166
77.432
77.000
76.577
30.002
27.668
12.611
Et
O
N
O
1e
syl-3-101_2.1
2019-01-06 16:48:39.937
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 69
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
200
150
100
50
0
PPM


7.268
5.281
5.214
4.443
2.203
2.178
2.154
2.129
1.113
1.089
1.063
0.919
0.137
-0.000
Et
OTBS
1g
syl-3-99_1.1
2019-01-06 17:08:34.515
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
0.98
1.00
1.96
1.96
3.07
9.20
5.88
10
8
6
4
2
0
PPM


132.868
119.871
87.479
85.254
77.423
77.000
76.577
52.118
30.177
25.792
18.273
12.740
-5.129
Et
OTBS
1g
syl-3-99_2.1
2019-01-06 17:12:46.328
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 54
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
200
150
100
50
0
PPM

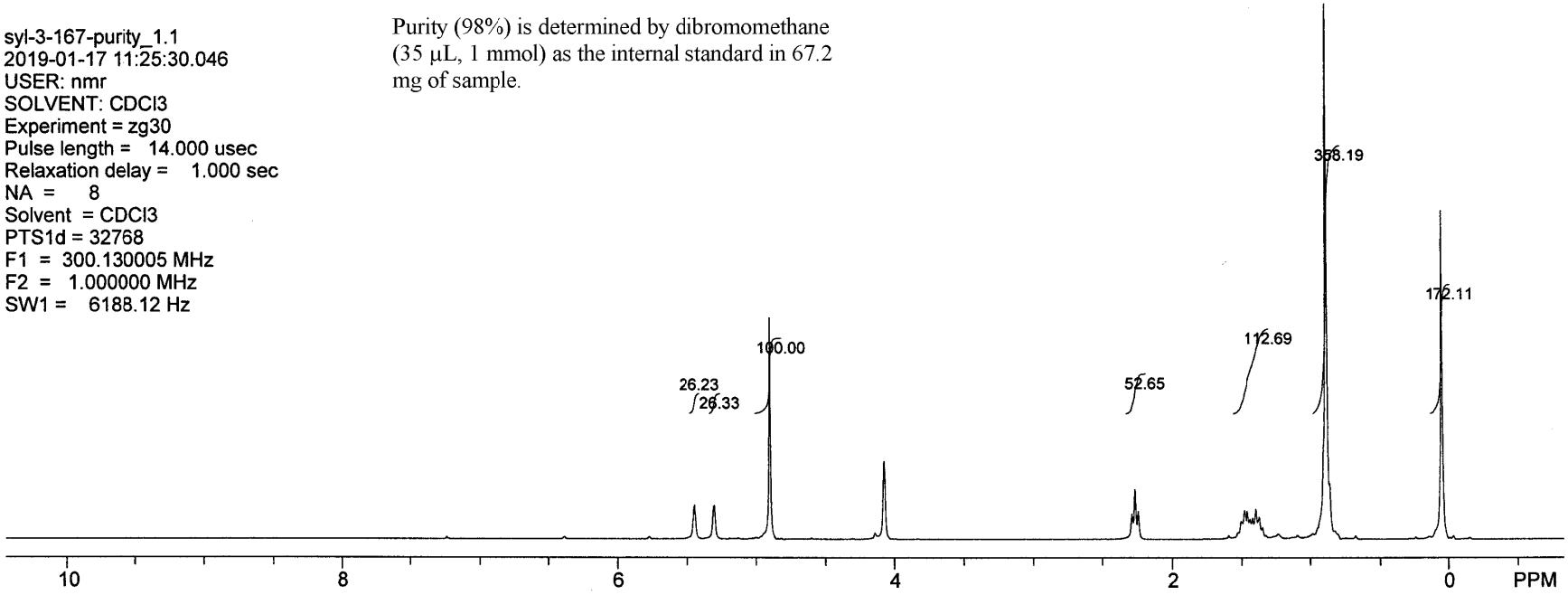

7.260
5.483
5.341
4.105
2.319
2.297
2.275
1.533
1.509
1.487
1.467
1.446
1.423
1.399
1.375
0.915
0.077
OTBS
C_4H_9
1i
syl-3-167_1.1
2019-01-16 21:29:05.875
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
1.00
0.97
2.01
1.94
4.11
12.64
6.17
10
8
6
4
2
0
PPM

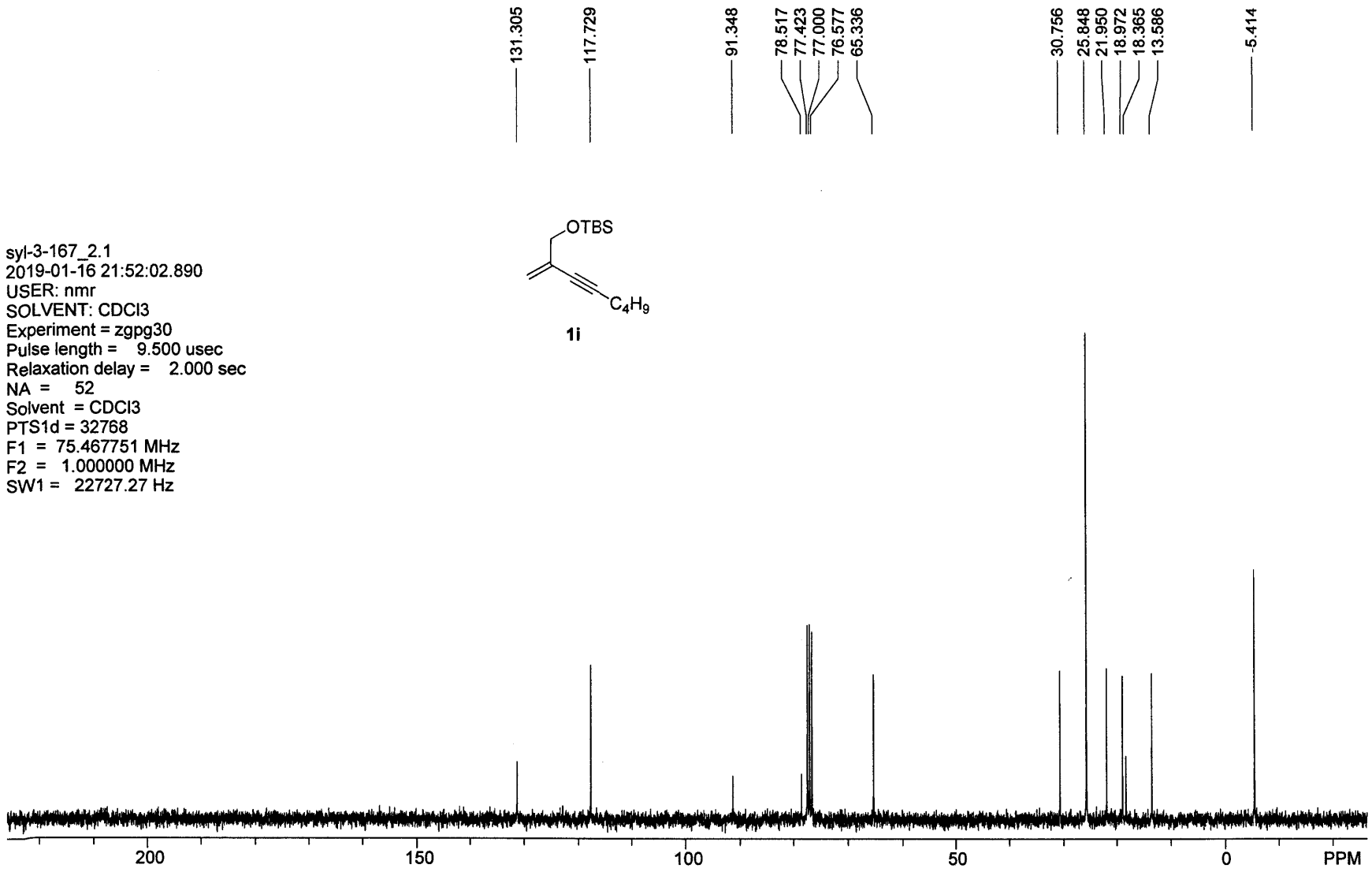


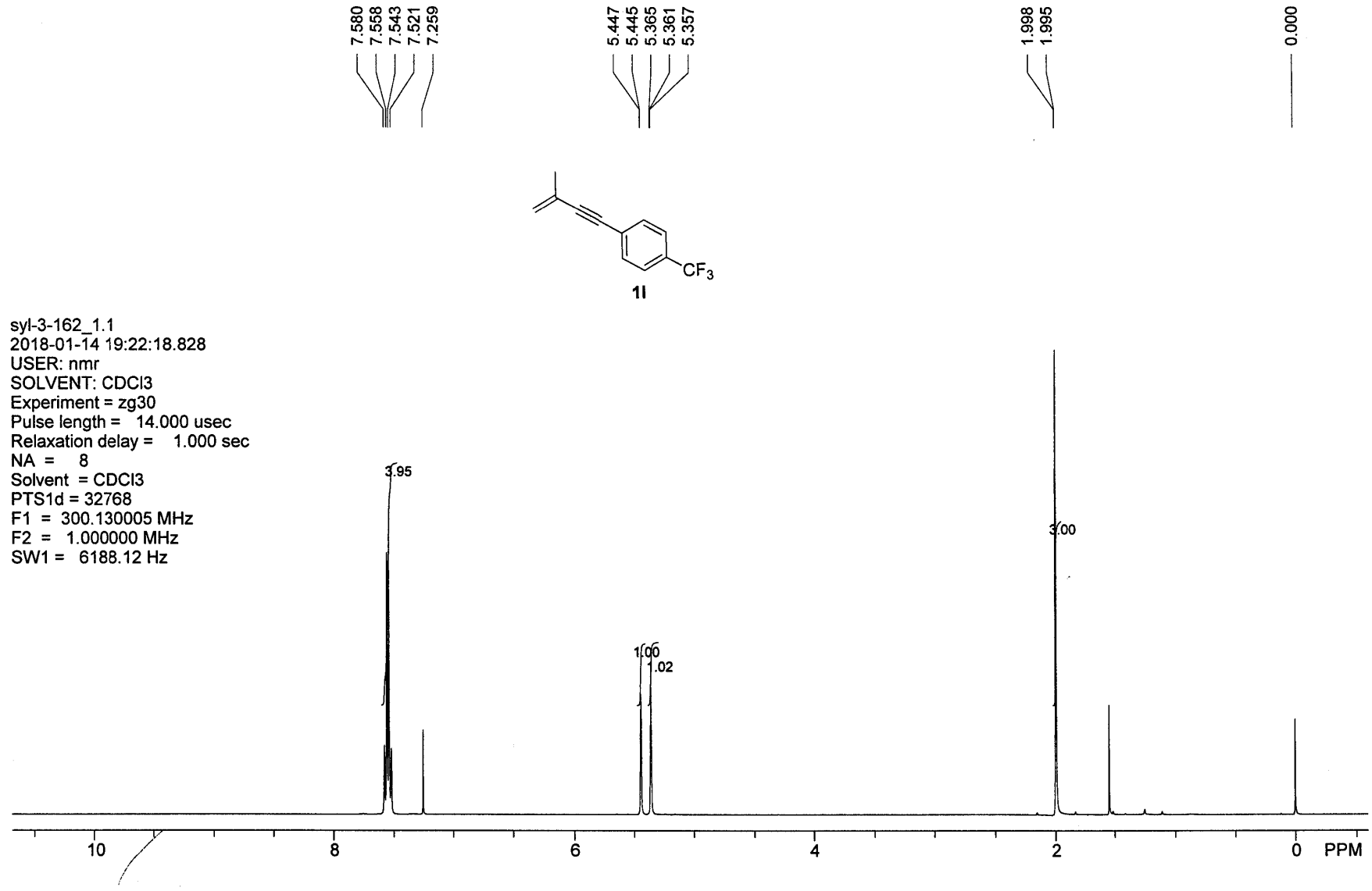
1i

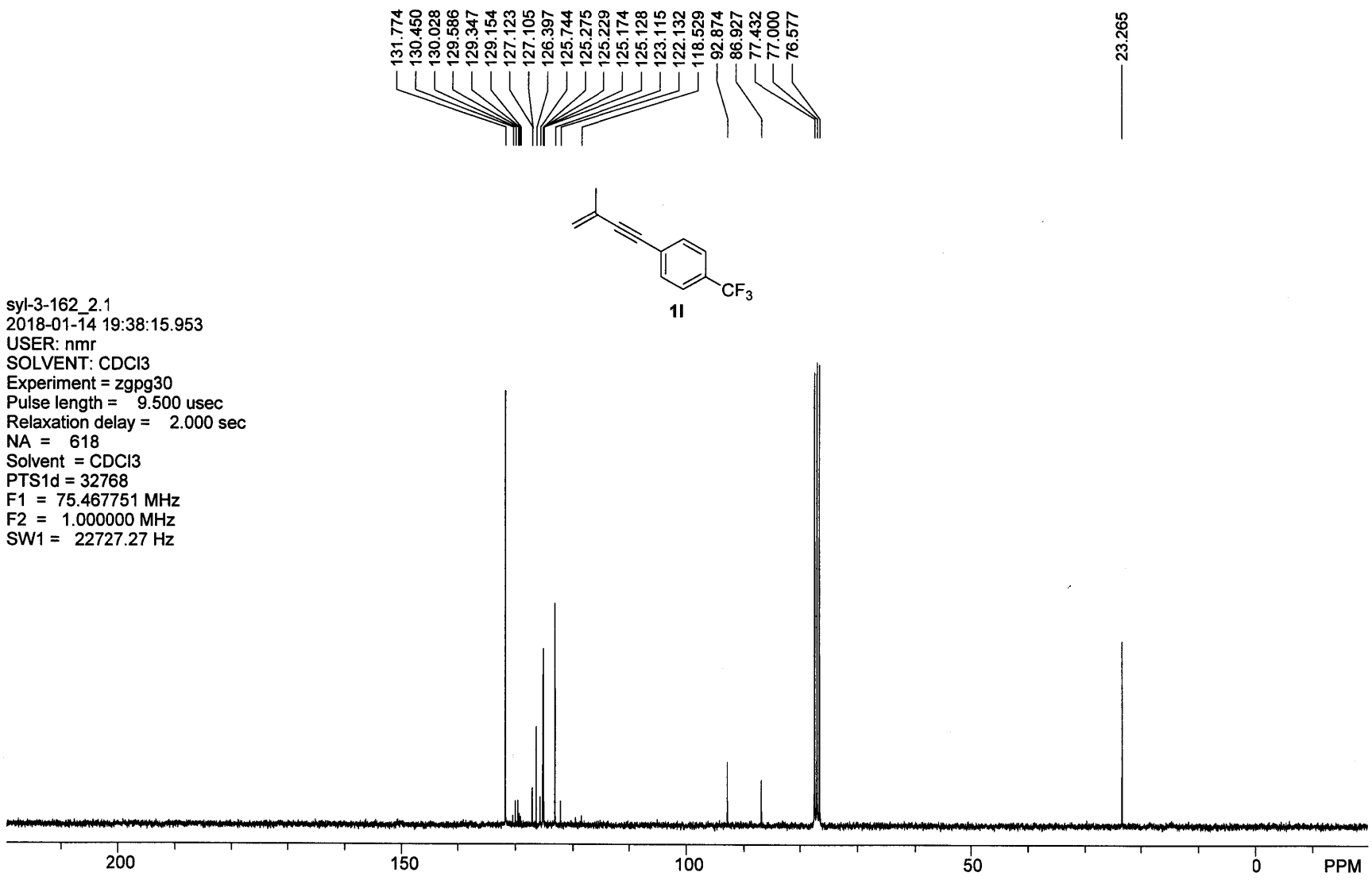
Purity (98%) is determined by dibromomethane (35 μL, 1 mmol) as the internal standard in 67.2 mg of sample.

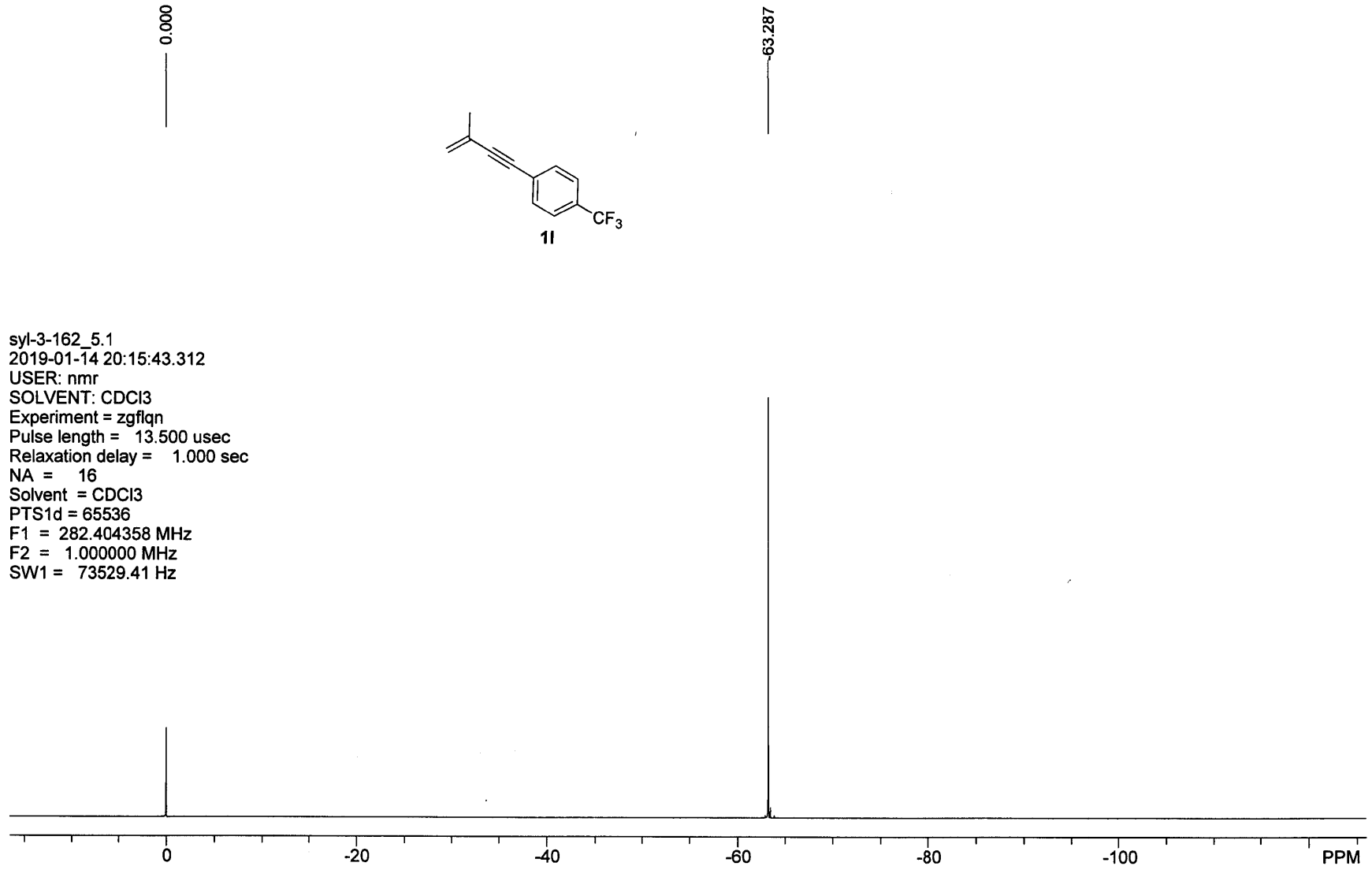
syl-3-167-purity_1.1
2019-01-17 11:25:30.046
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz

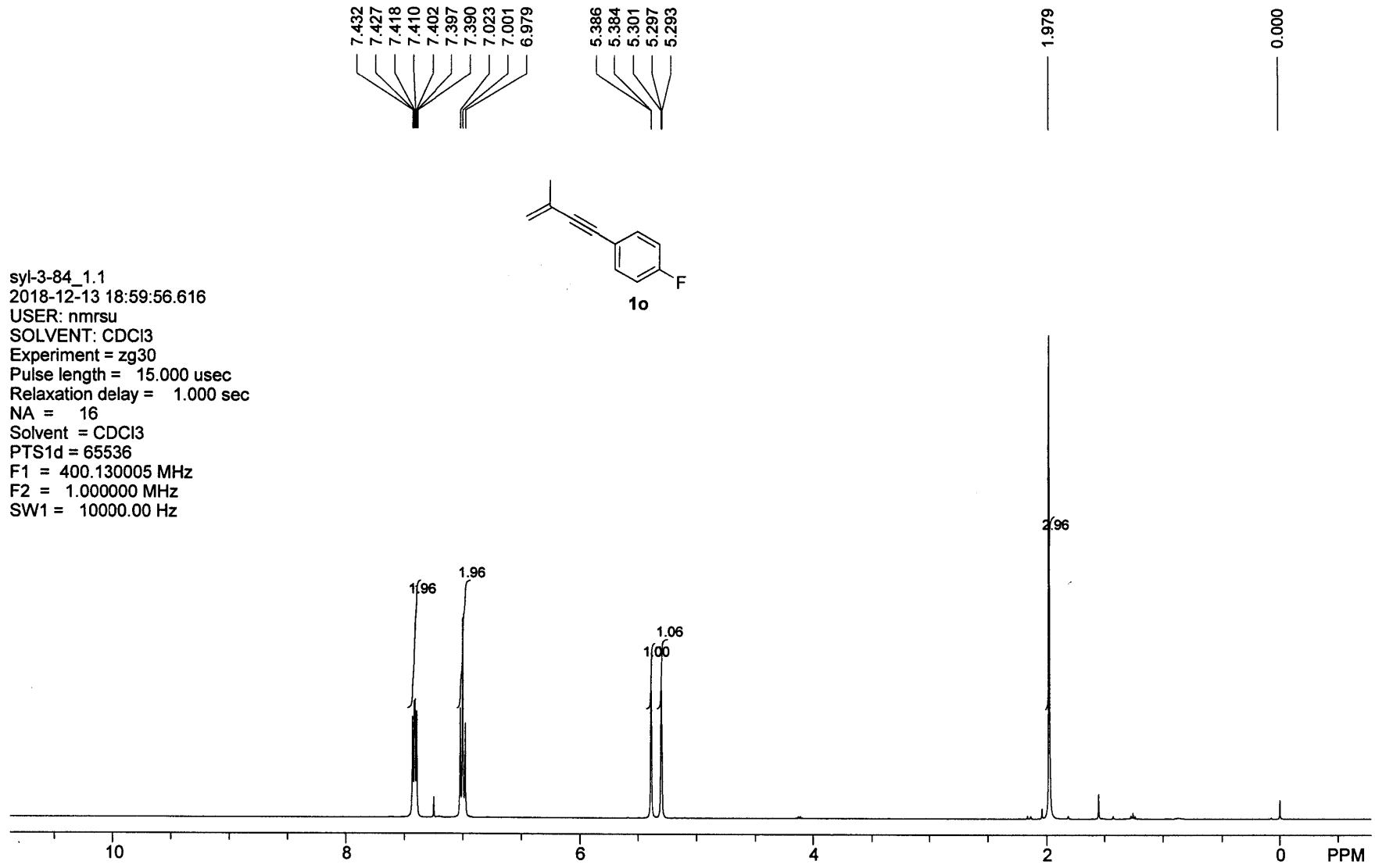


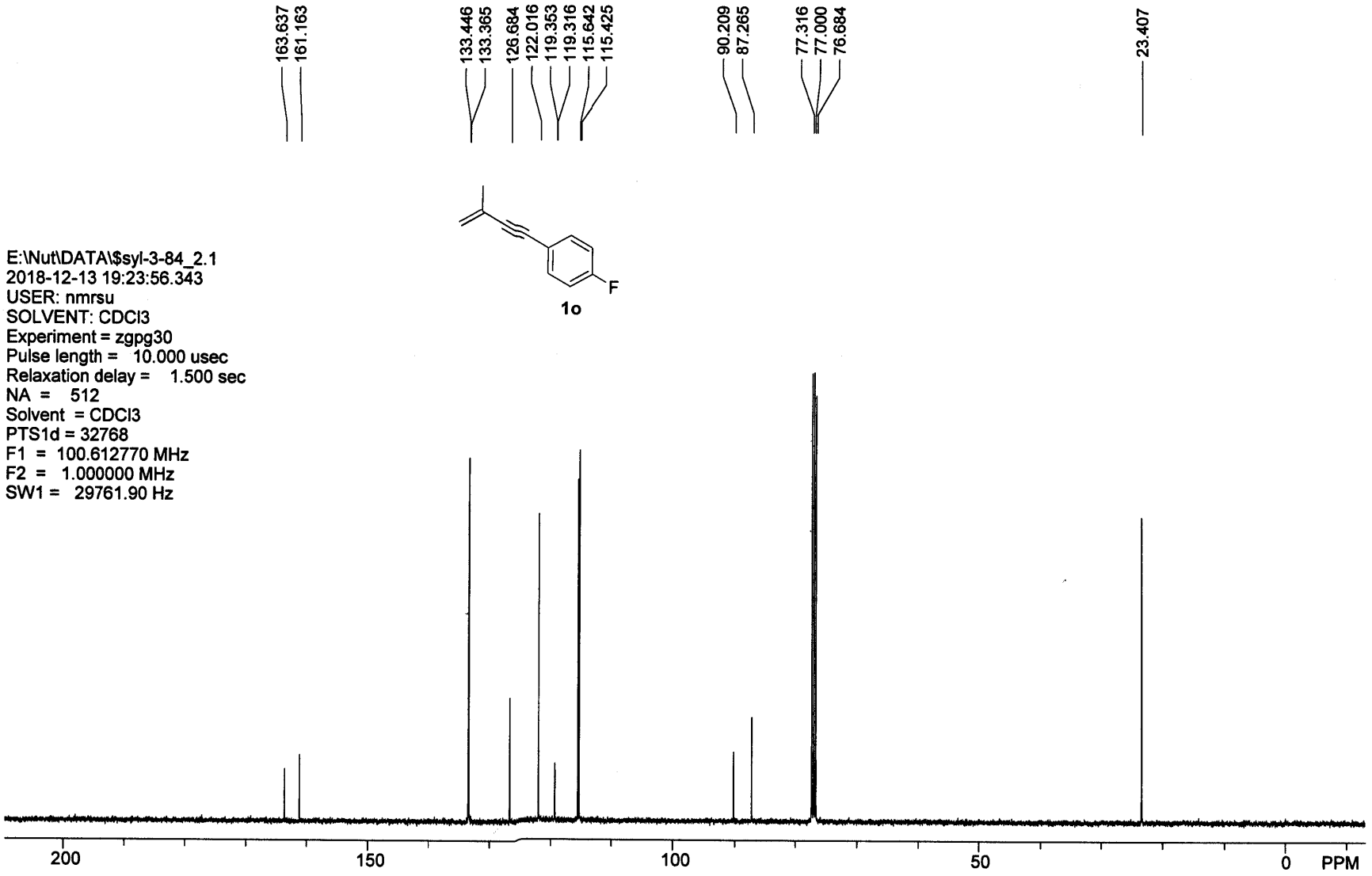

131.305
117.729
91.348
78.517
77.423
77.000
76.577
65.336
30.756
25.848
21.950
18.972
18.365
13.586
5.414
OTBS
C_4H_9
1i
syl-3-167_2.1
2019-01-16 21:52:02.890
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 52
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
200
150
100
50
0
PPM

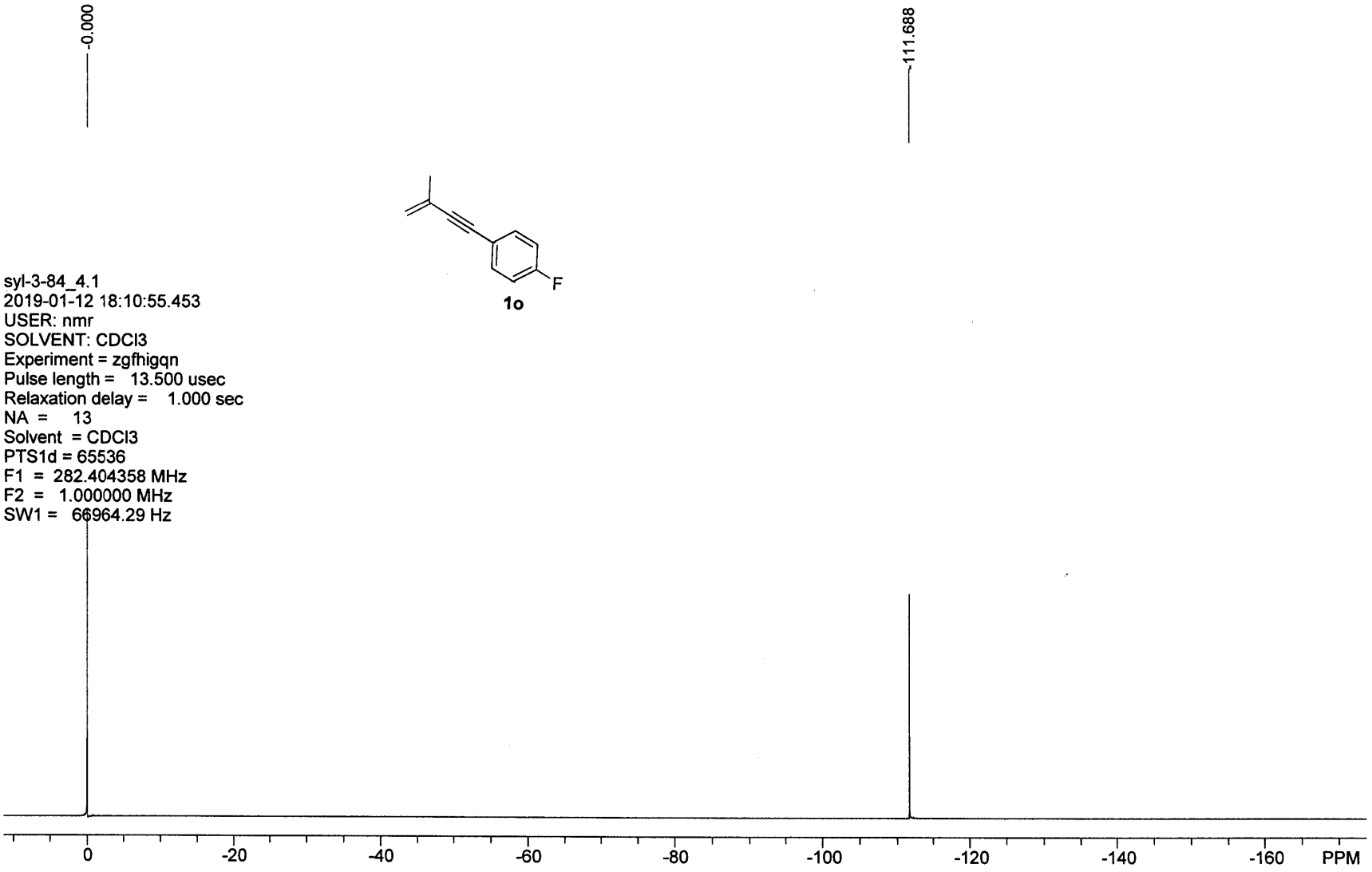

7.580
7.558
7.543
7.521
7.259
5.447
5.445
5.365
5.361
5.357
1.998
1.995
0.000
CF3
1l
syl-3-162_1.1
2018-01-14 19:22:18.828
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
3.95
1.00
1.02
3.00
10
8
6
4
2
0
PPM

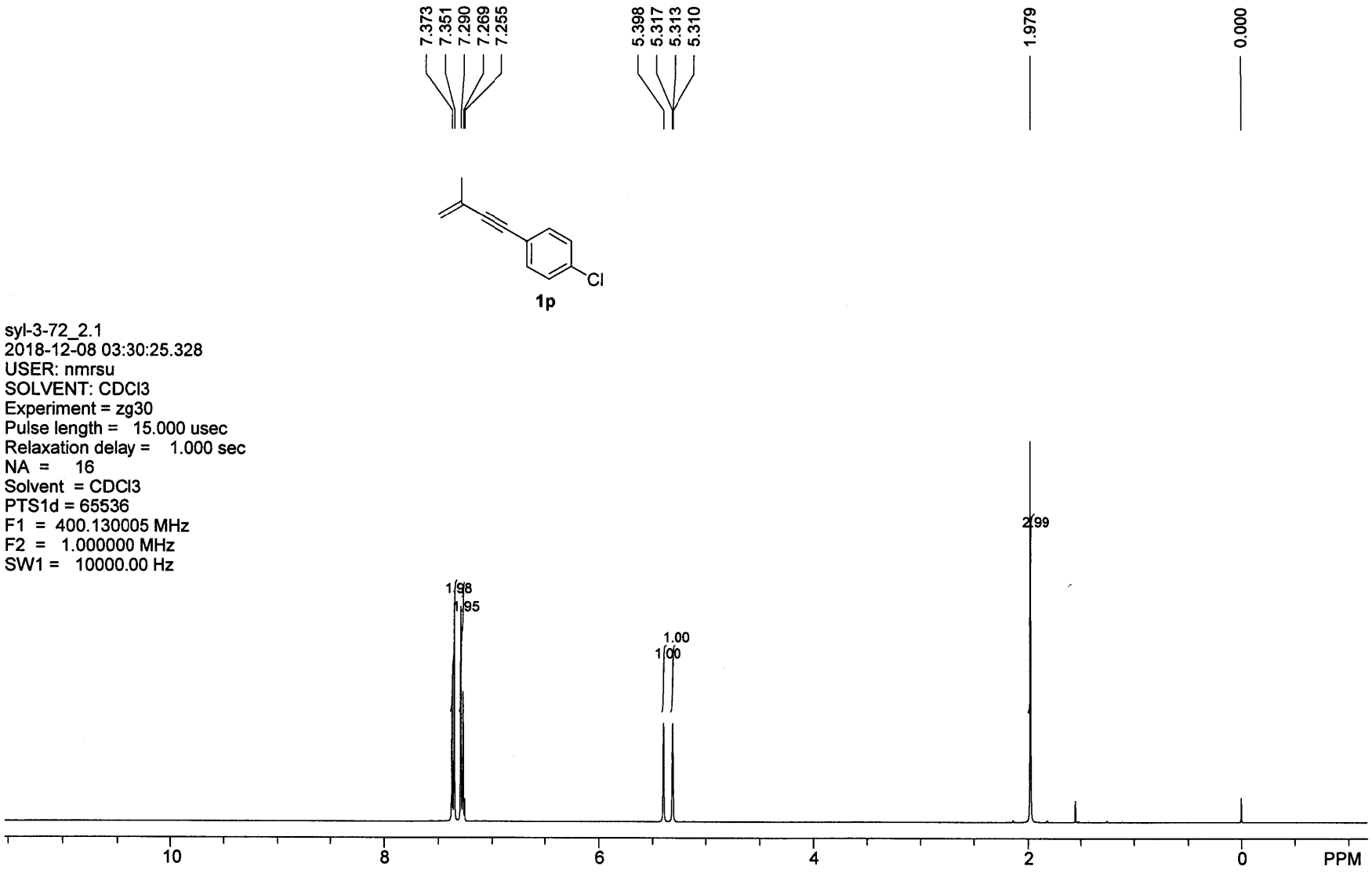

131.774
130.450
130.028
129.586
129.347
129.154
127.123
127.105
126.397
125.744
125.275
125.229
125.174
125.128
123.115
122.132
118.529
92.874
86.927
77.432
77.000
76.577
23.265
CF3
1l
syl-3-162_2.1
2018-01-14 19:38:15.953
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 618
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
200
150
100
50
0
PPM

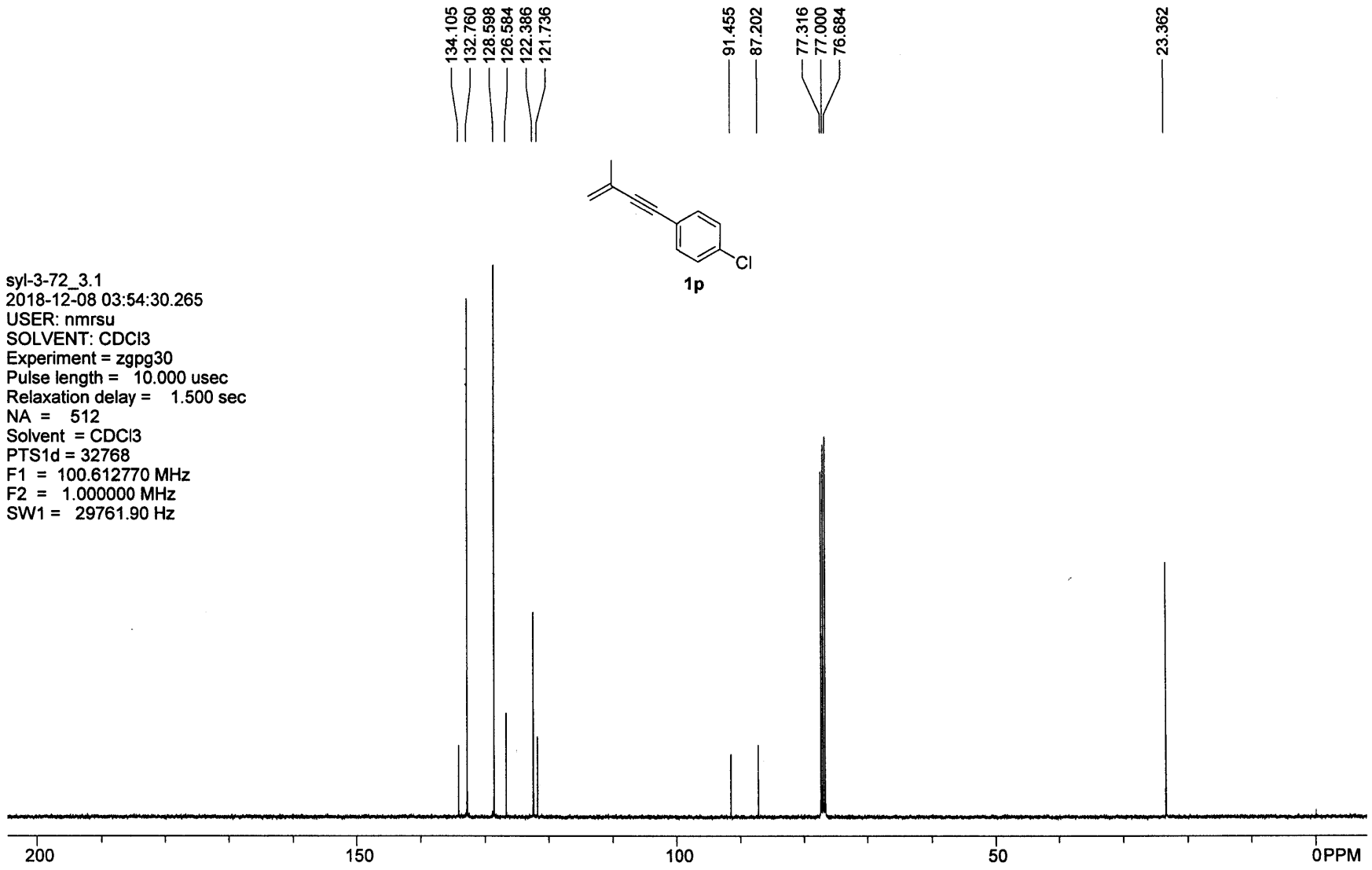

0.000
-63.287
CF3
1l
syl-3-162_5.1
2019-01-14 20:15:43.312
USER: nmr
SOLVENT: CDCl3
Experiment = zgflqn
Pulse length = 13.500 usec
Relaxation delay = 1.000 sec
NA = 16
Solvent = CDCl3
PTS1d = 65536
F1 = 282.404358 MHz
F2 = 1.000000 MHz
SW1 = 73529.41 Hz
0
-20
-40
-60
-80
-100
PPM

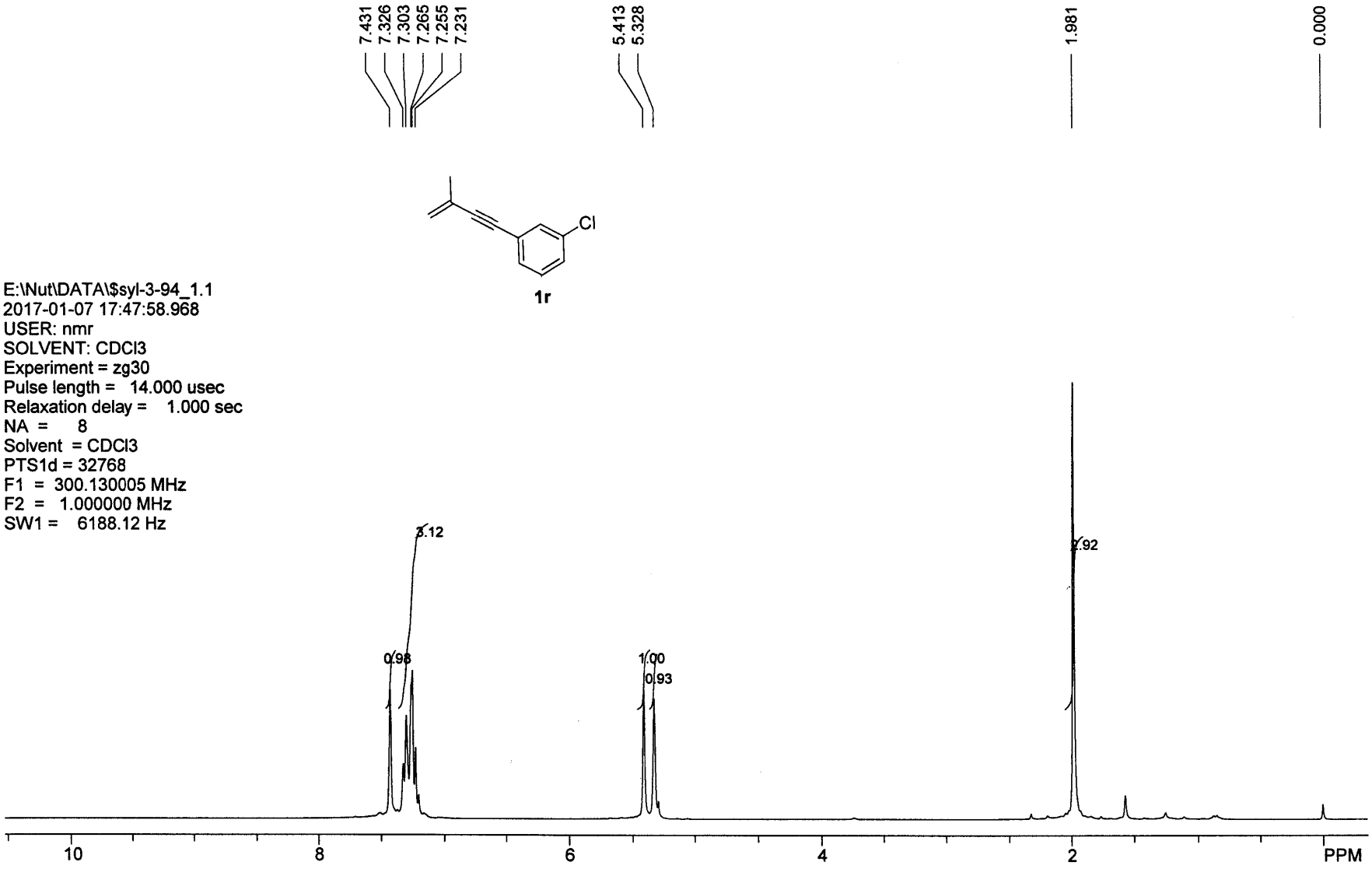

7.432
7.427
7.418
7.410
7.402
7.397
7.390
7.023
7.001
6.979
5.386
5.384
5.301
5.297
5.293
1.979
0.000
F
1o
syl-3-84_1.1
2018-12-13 18:59:56.616
USER: nmrsu
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 15.000 usec
Relaxation delay = 1.000 sec
NA = 16
Solvent = CDCl3
PTS1d = 65536
F1 = 400.130005 MHz
F2 = 1.000000 MHz
SW1 = 10000.00 Hz
1.96
1.96
1.06
1.00
2.96
10
8
6
4
2
0
PPM

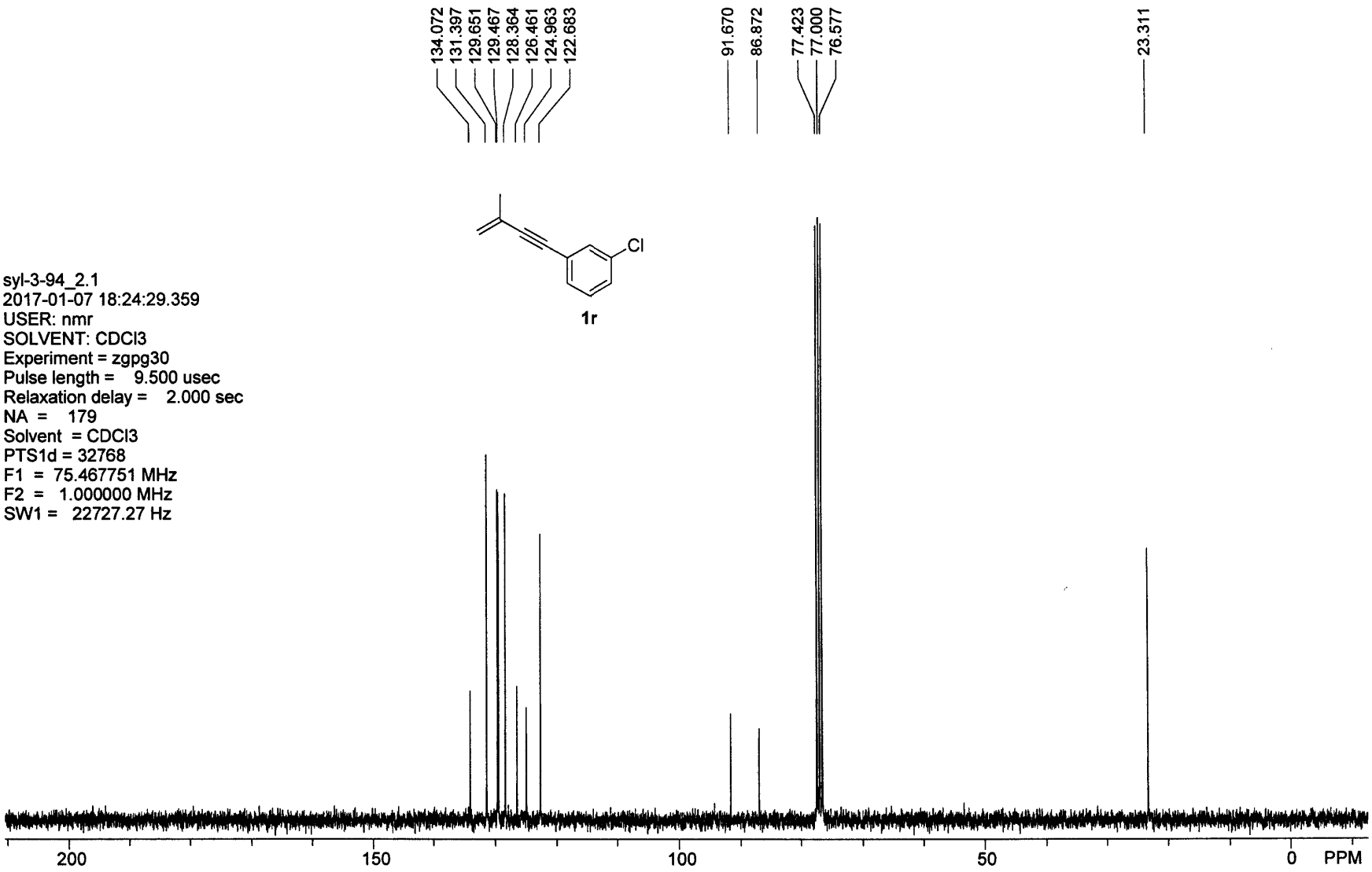

163.637
161.163
133.446
133.365
126.684
122.016
119.353
119.316
115.642
115.425
90.209
87.265
77.316
77.000
76.684
23.407
F
1o
E:\Nut\DATA\$syl-3-84_2.1
2018-12-13 19:23:56.343
USER: nmrsu
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 10.000 usec
Relaxation delay = 1.500 sec
NA = 512
Solvent = CDCl3
PTS1d = 32768
F1 = 100.612770 MHz
F2 = 1.000000 MHz
SW1 = 29761.90 Hz
200
150
100
50
0
PPM

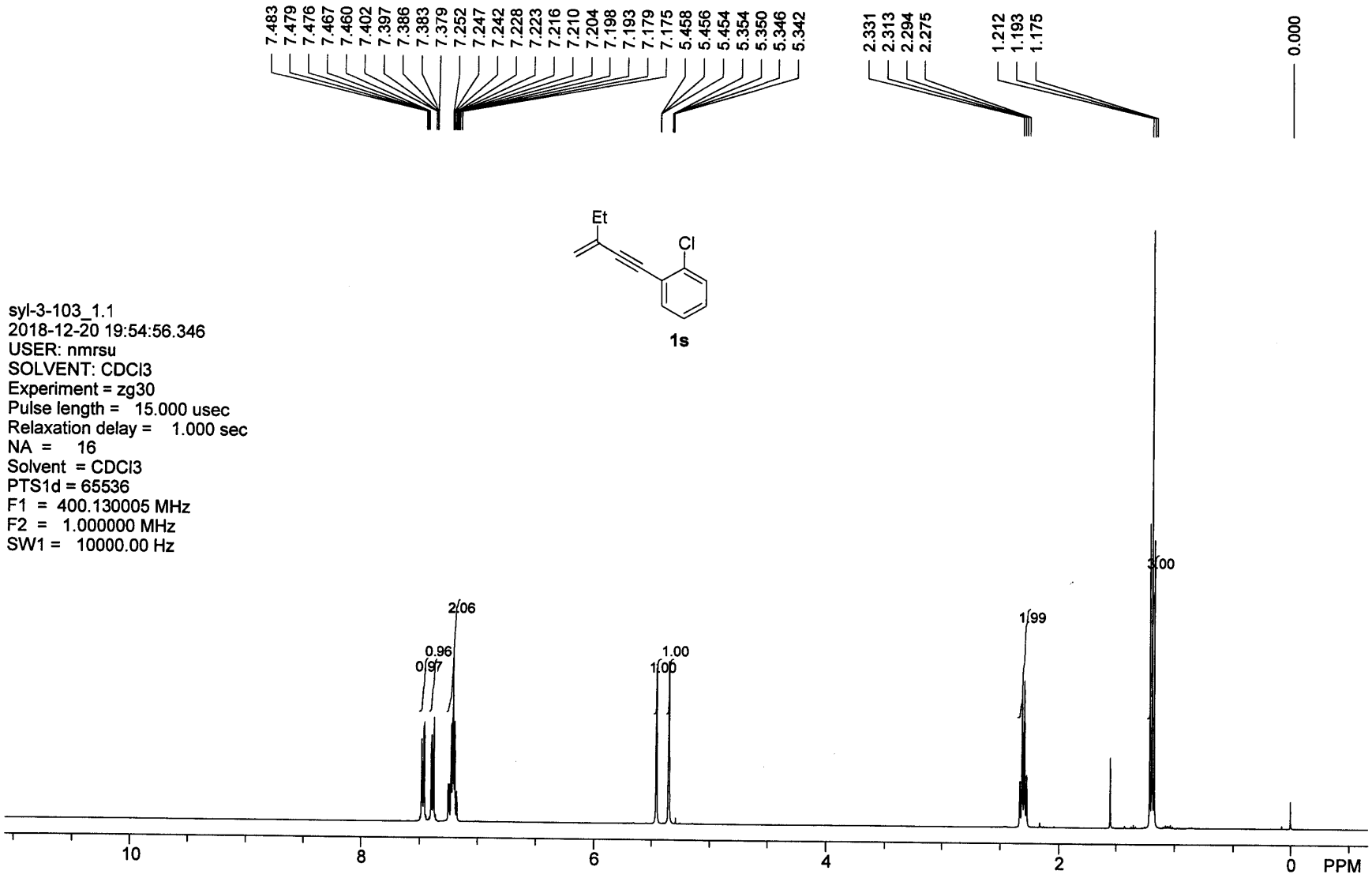

-0.000
-111.688
F
1o
syl-3-84_4.1
2019-01-12 18:10:55.453
USER: nmr
SOLVENT: CDCl3
Experiment = zgfhigqn
Pulse length = 13.500 usec
Relaxation delay = 1.000 sec
NA = 13
Solvent = CDCl3
PTS1d = 65536
F1 = 282.404358 MHz
F2 = 1.000000 MHz
SW1 = 66964.29 Hz
0
-20
-40
-60
-80
-100
-120
-140
-160
PPM

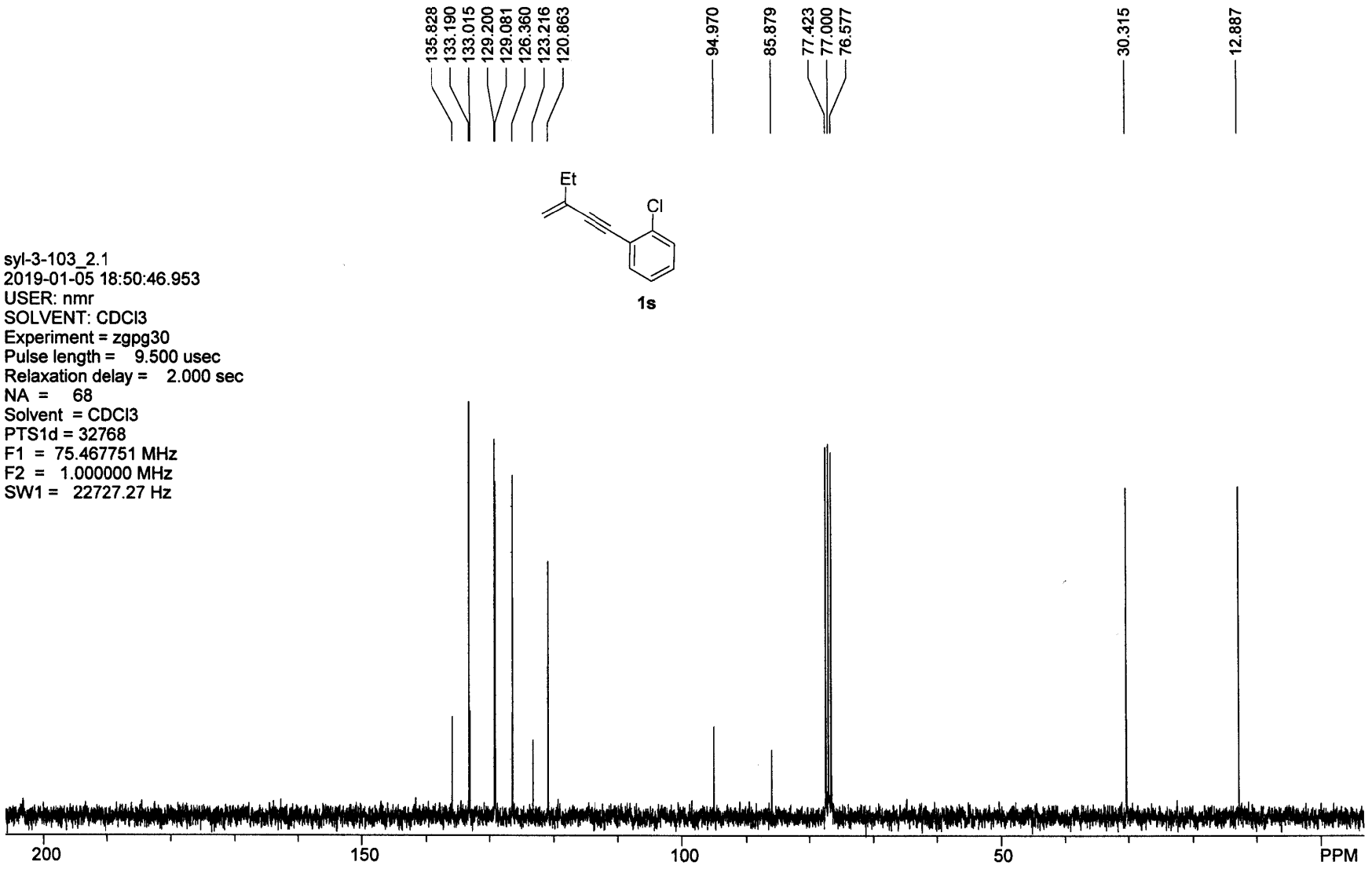

7.373
7.351
7.290
7.269
7.255
5.398
5.317
5.313
5.310
1.979
0.000
Cl
1p
syl-3-72_2.1
2018-12-08 03:30:25.328
USER: nmrsu
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 15.000 usec
Relaxation delay = 1.000 sec
NA = 16
Solvent = CDCl3
PTS1d = 65536
F1 = 400.130005 MHz
F2 = 1.000000 MHz
SW1 = 10000.00 Hz
1.98
1.95
1.00
1.00
2.99
10
8
6
4
2
0
PPM

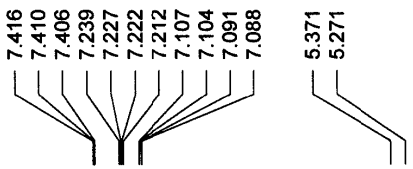

134.105
132.760
128.598
126.584
122.386
121.736
91.455
87.202
77.316
77.000
76.684
23.362
Cl
1p
syl-3-72_3.1
2018-12-08 03:54:30.265
USER: nmrsu
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 10.000 usec
Relaxation delay = 1.500 sec
NA = 512
Solvent = CDCl3
PTS1d = 32768
F1 = 100.612770 MHz
F2 = 1.000000 MHz
SW1 = 29761.90 Hz
200
150
100
50
0PPM

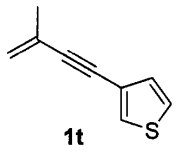

7.431
7.326
7.303
7.265
7.255
7.231
5.413
5.328
1.981
0.000
Cl
1r
E:\Nut\DATA\$syl-3-94_1.1
2017-01-07 17:47:58.968
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
3.12
0.98
1.00
0.93
2.92
10
8
6
4
2
PPM


134.072
131.397
129.651
129.467
128.364
126.461
124.963
122.683
91.670
86.872
77.423
77.000
76.577
23.311
Cl
1r
syl-3-94_2.1
2017-01-07 18:24:29.359
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 179
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
200
150
100
50
0
PPM


7.483
7.479
7.476
7.467
7.460
7.402
7.397
7.386
7.383
7.379
7.252
7.247
7.242
7.228
7.223
7.216
7.210
7.204
7.198
7.193
7.179
7.175
5.458
5.456
5.454
5.354
5.350
5.346
5.342
2.331
2.313
2.294
2.275
1.212
1.193
1.175
0.000
Et
Cl
1s
syl-3-103_1.1
2018-12-20 19:54:56.346
USER: nmrsu
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 15.000 usec
Relaxation delay = 1.000 sec
NA = 16
Solvent = CDCl3
PTS1d = 65536
F1 = 400.130005 MHz
F2 = 1.000000 MHz
SW1 = 10000.00 Hz
0.97
0.96
2.06
1.00
1.00
1.99
3.00
10
8
6
4
2
0
PPM

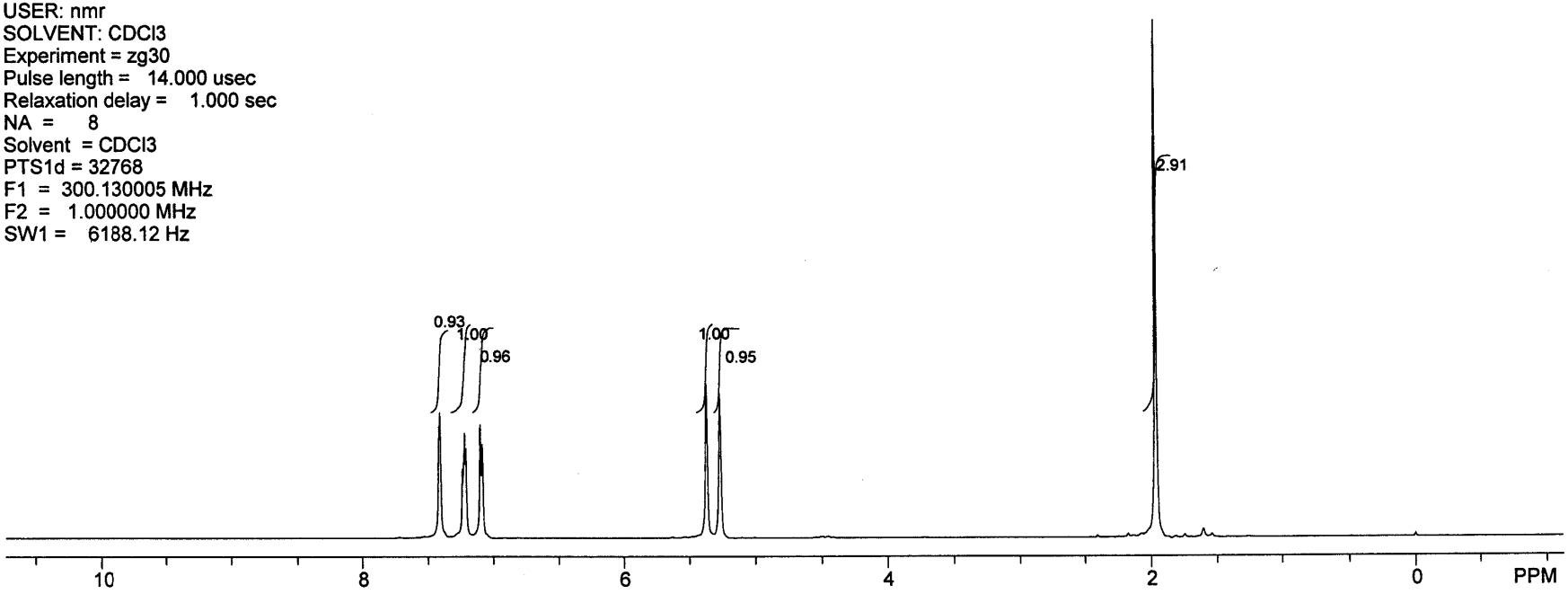

135.828
133.190
133.015
129.200
129.081
126.360
123.216
120.863
94.970
85.879
77.423
77.000
76.577
30.315
12.887
Et
Cl
1s
syl-3-103_2.1
2019-01-05 18:50:46.953
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 68
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
200
150
100
50
PPM

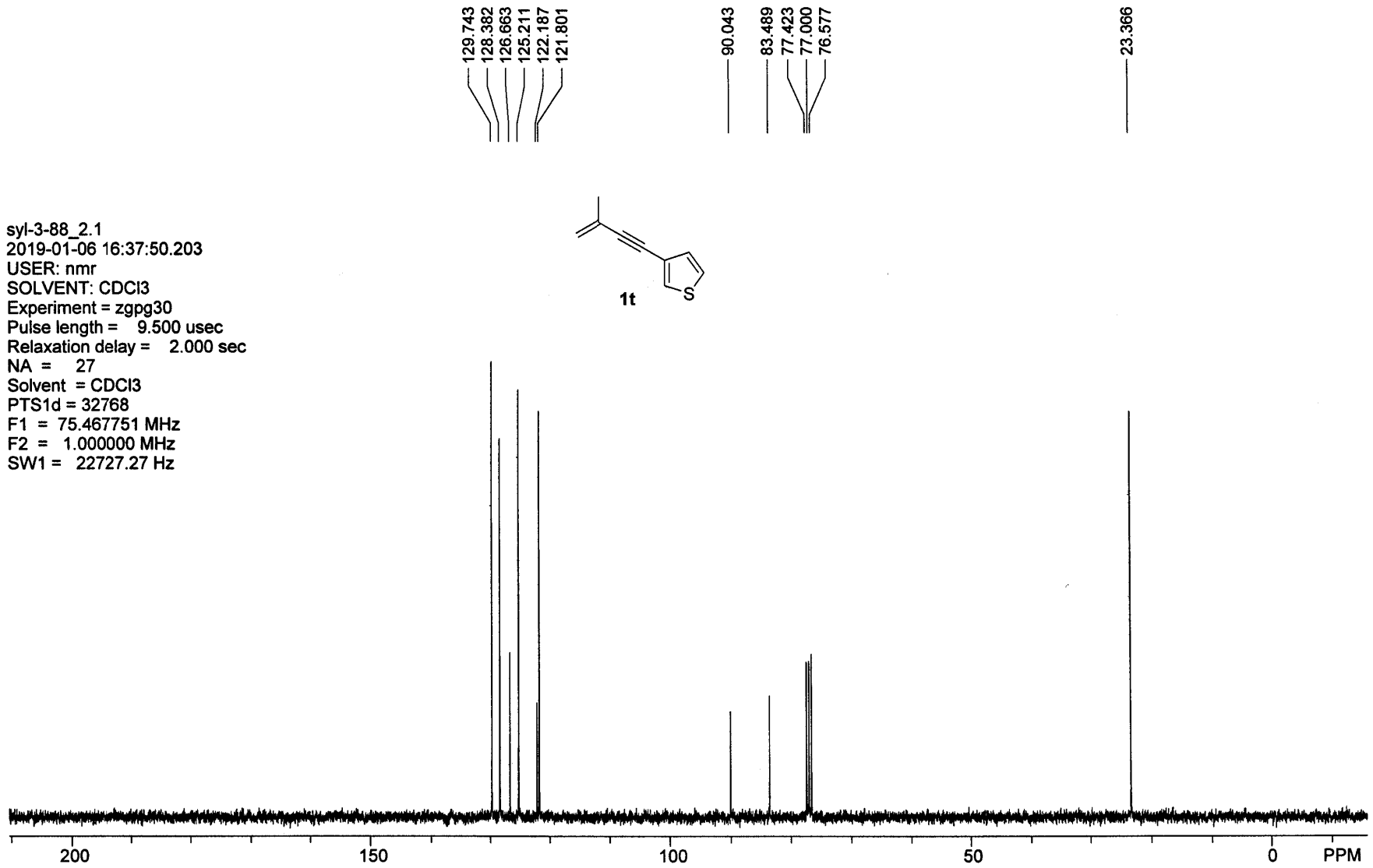


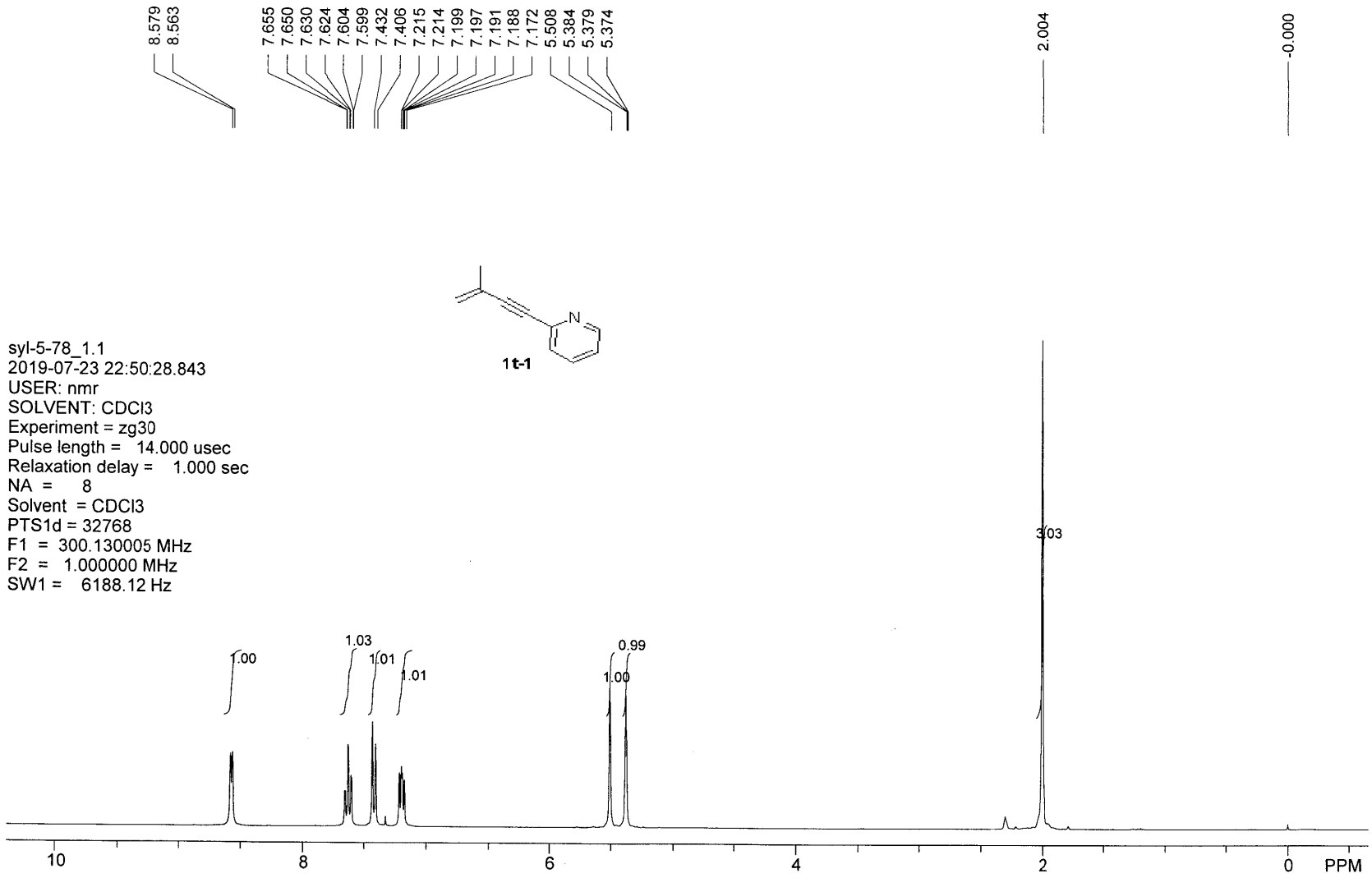


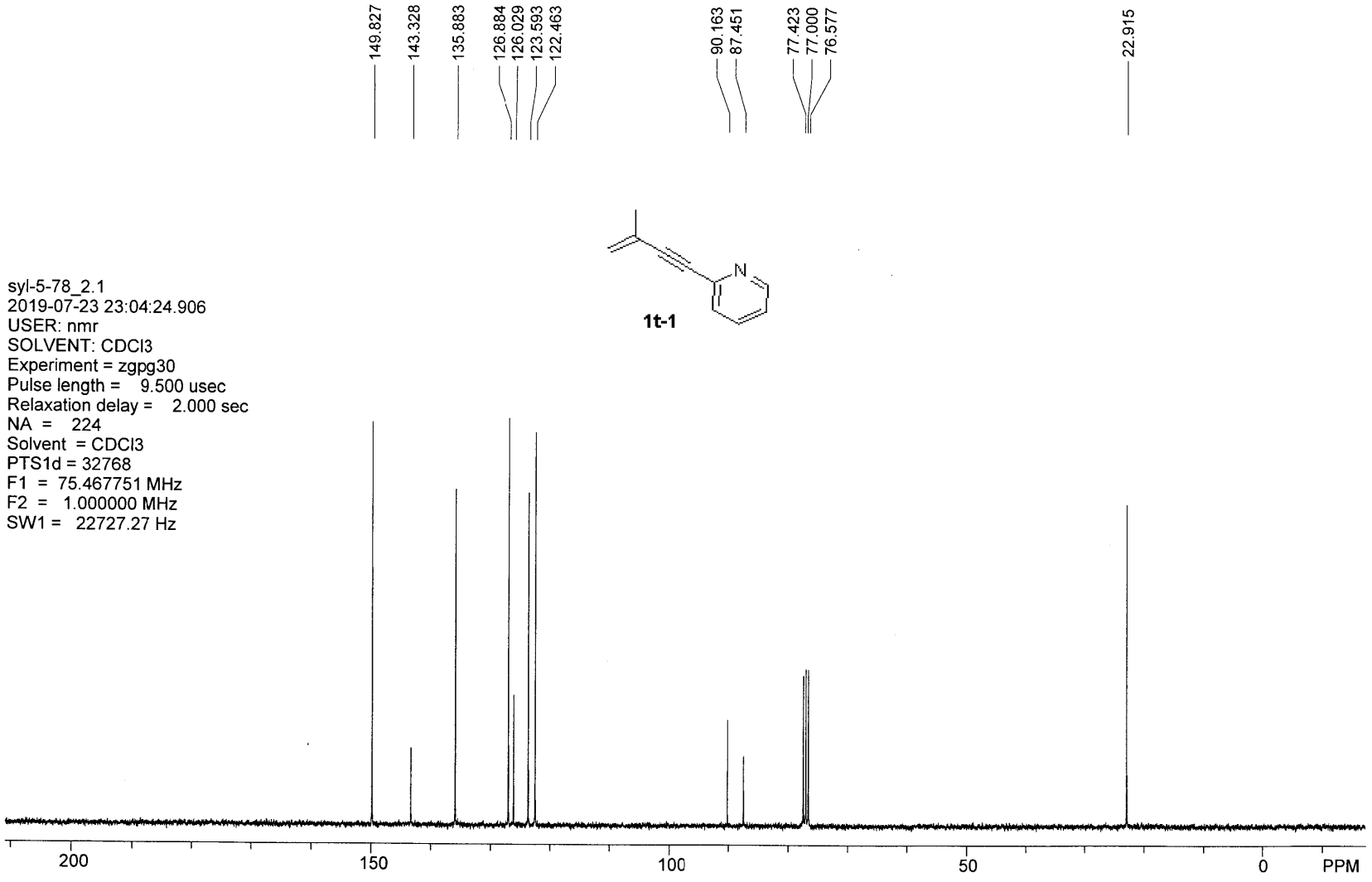
1t

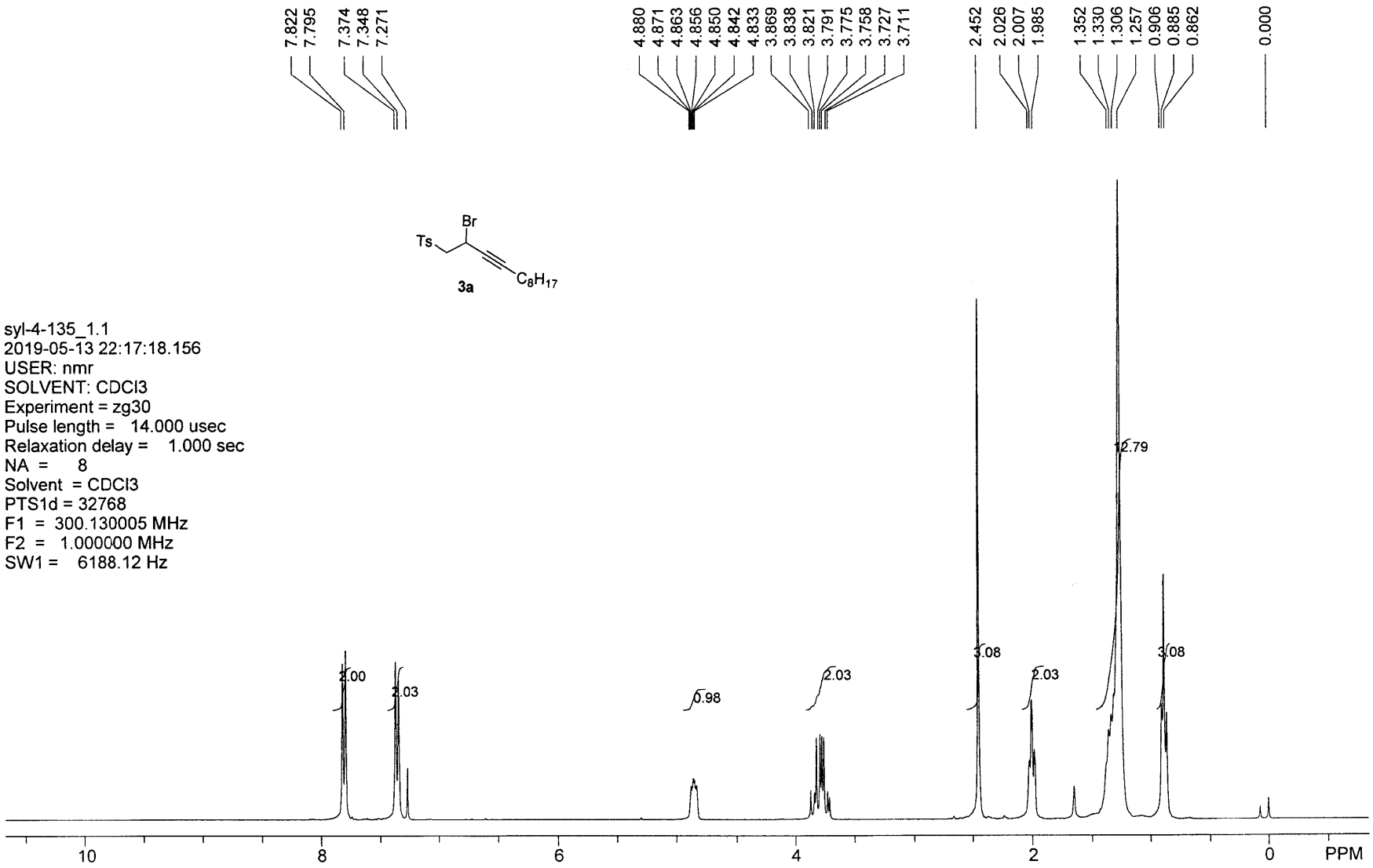
syl-3-88_1.1
2019-01-06 16:35:14.203
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz

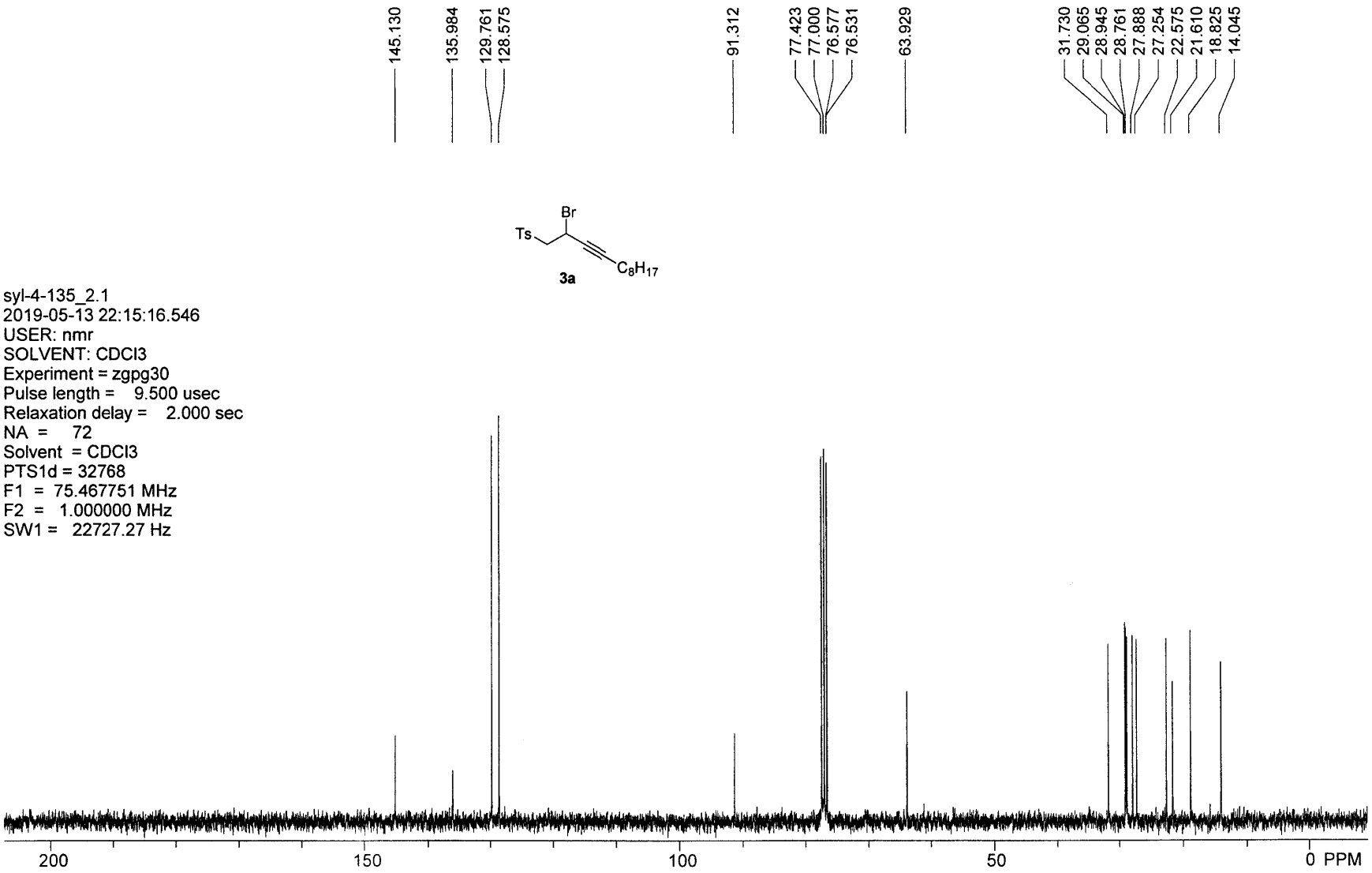


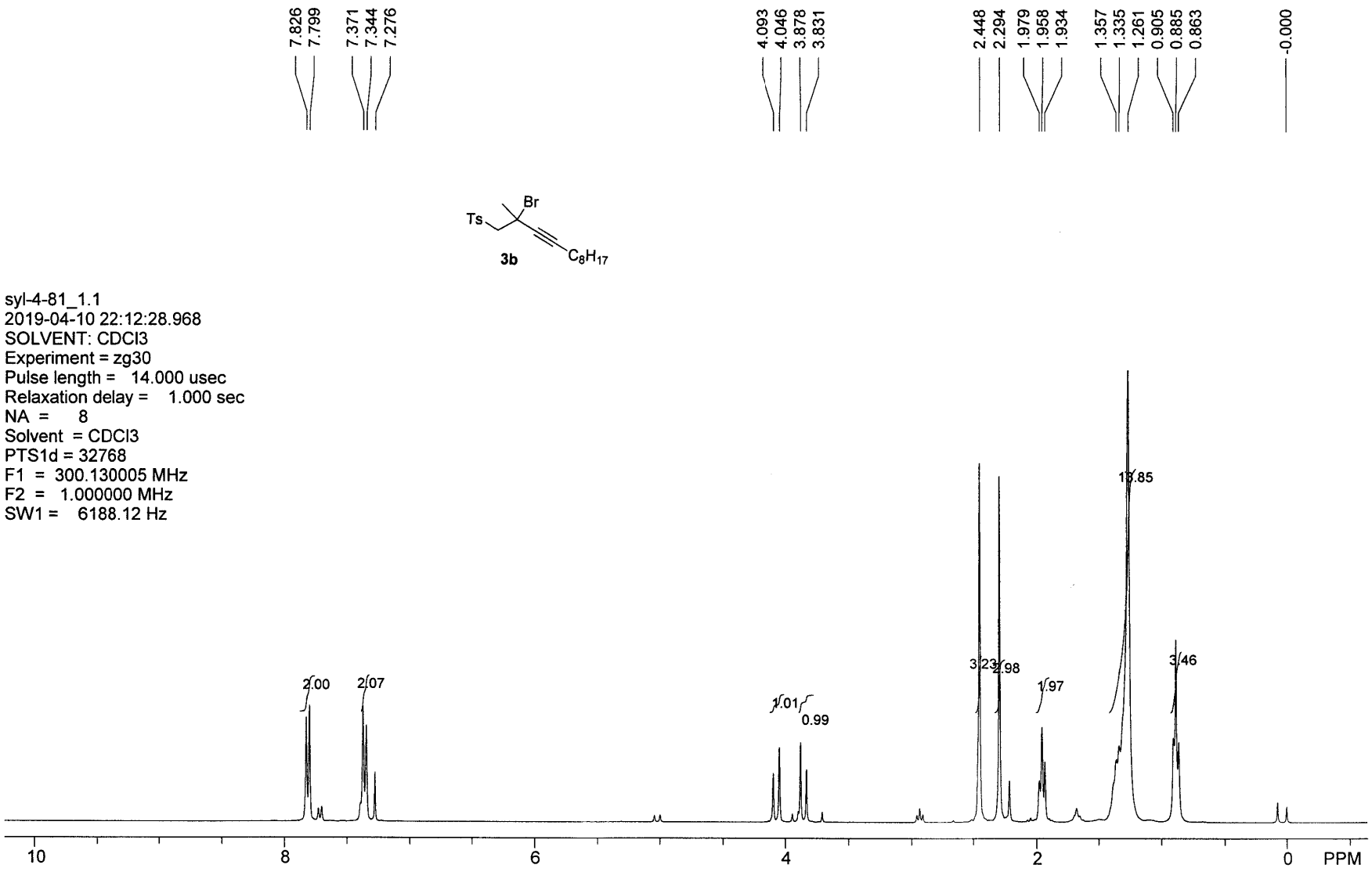

129.743
128.382
126.663
125.211
122.187
121.801
90.043
83.489
77.423
77.000
76.577
23.366
S
1t
syl-3-88_2.1
2019-01-06 16:37:50.203
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 27
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
200
150
100
50
0
PPM

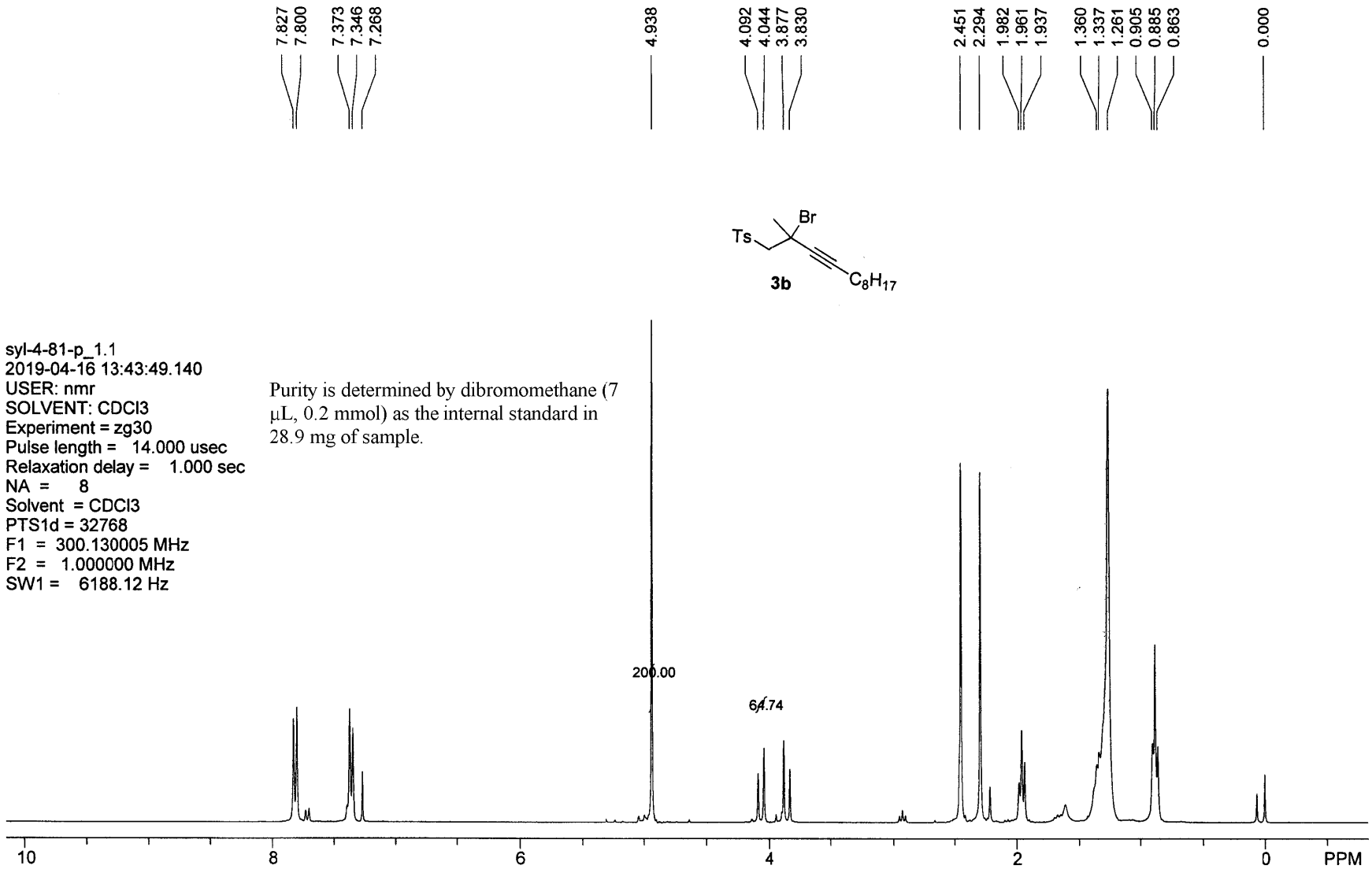

8.579
8.563
7.655
7.650
7.630
7.624
7.604
7.599
7.432
7.406
7.215
7.214
7.199
7.197
7.191
7.188
7.172
5.508
5.384
5.379
5.374
2.004
-0.000
N
1t-1
syl-5-78_1.1
2019-07-23 22:50:28.843
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
1.00
1.03
1.01
1.01
1.00
0.99
3.03
10
8
6
4
2
0
PPM

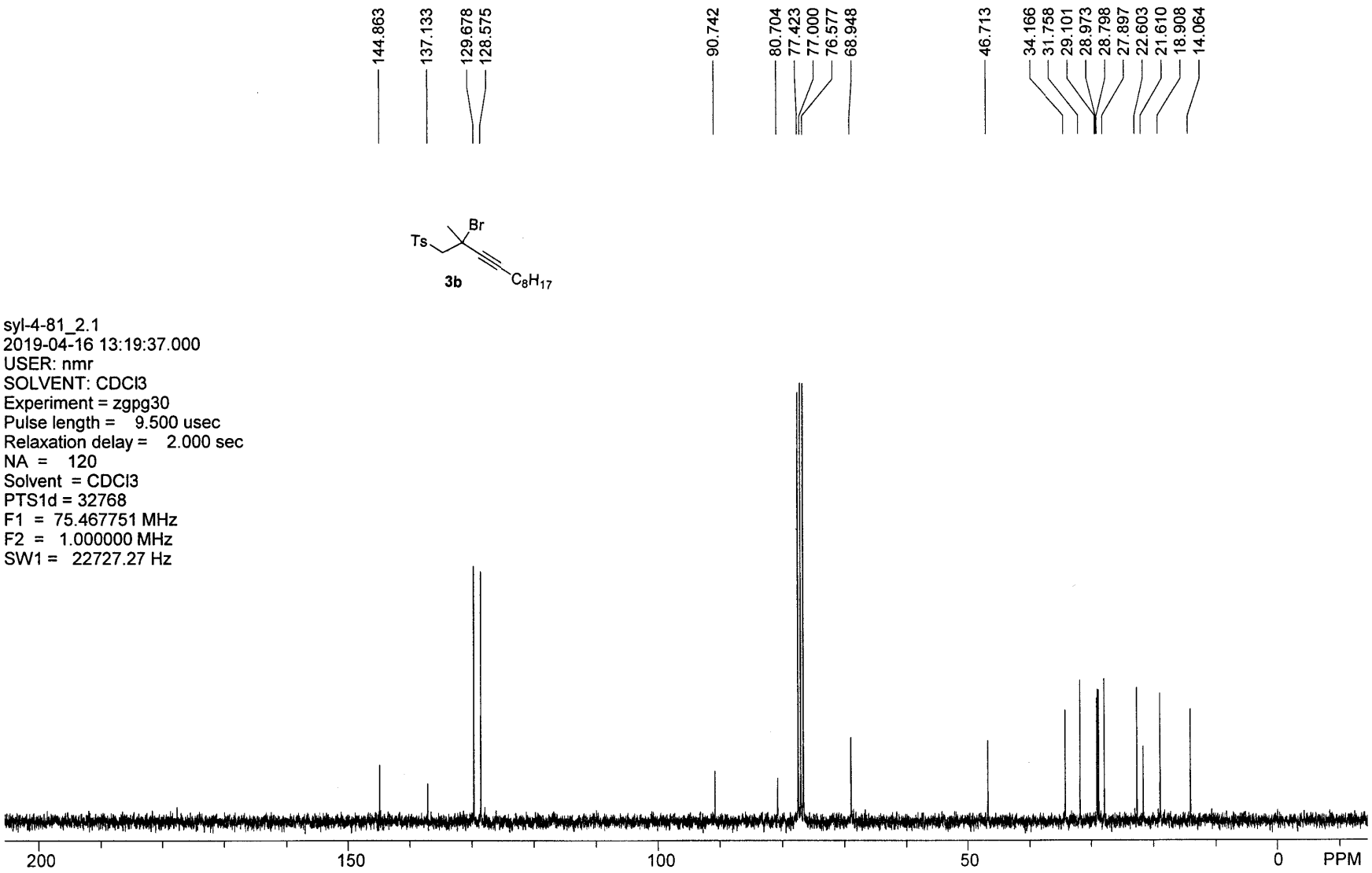

149.827
143.328
135.883
126.884
126.029
123.593
122.463
90.163
87.451
77.423
77.000
76.577
22.915
N
1t-1
syl-5-78_2.1
2019-07-23 23:04:24.906
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 224
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
200
150
100
50
0
PPM

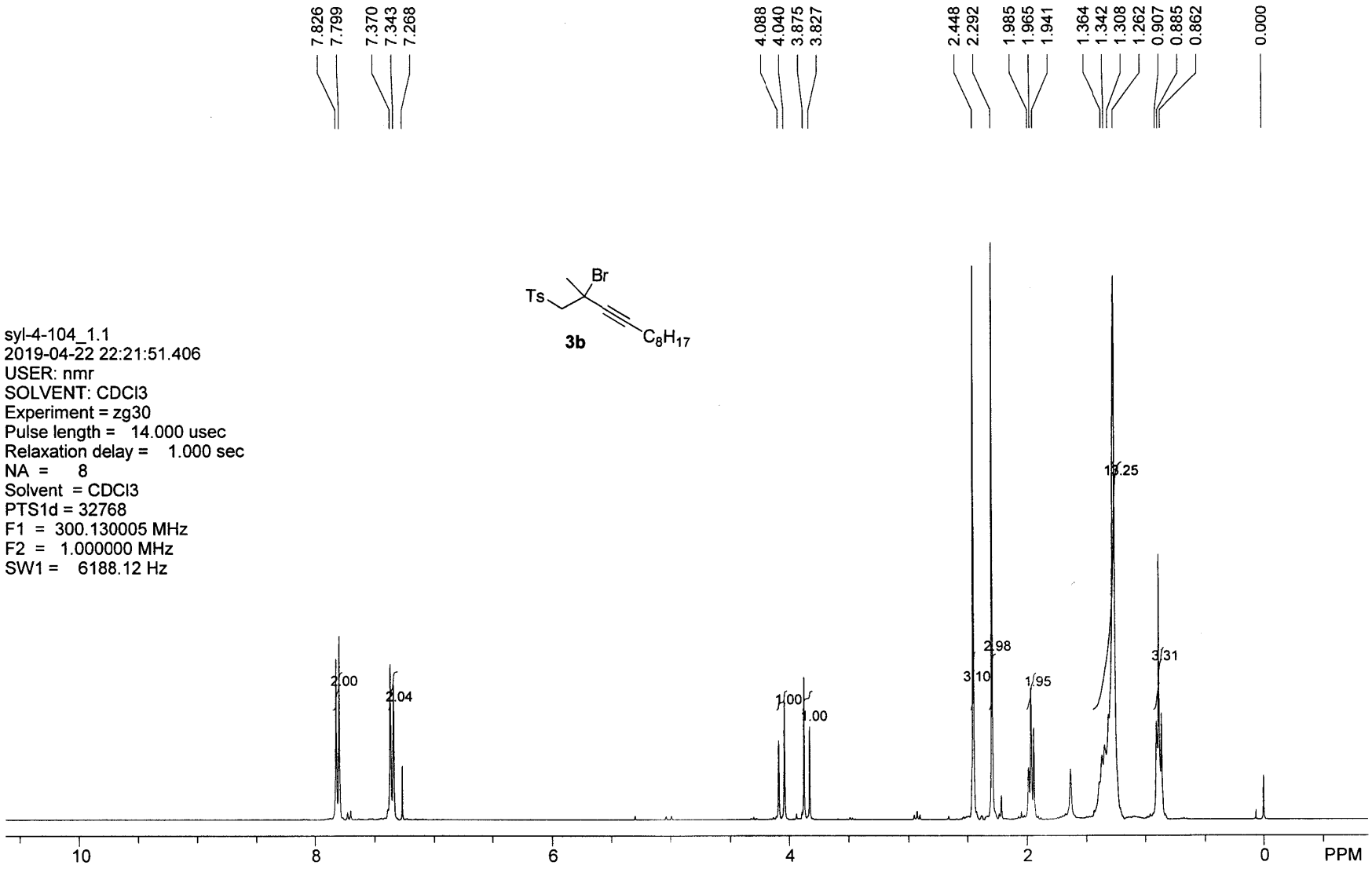

7.822
7.795
7.374
7.348
7.271
4.880
4.871
4.863
4.856
4.850
4.842
4.833
3.869
3.838
3.821
3.791
3.775
3.758
3.727
3.711
2.452
2.026
2.007
1.985
1.352
1.330
1.306
1.257
0.906
0.885
0.862
0.000
Br
Ts
C8H17
3a
syl-4-135_1.1
2019-05-13 22:17:18.156
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
2.00
2.03
0.98
2.03
3.08
2.03
12.79
3.08
10
8
6
4
2
0
PPM

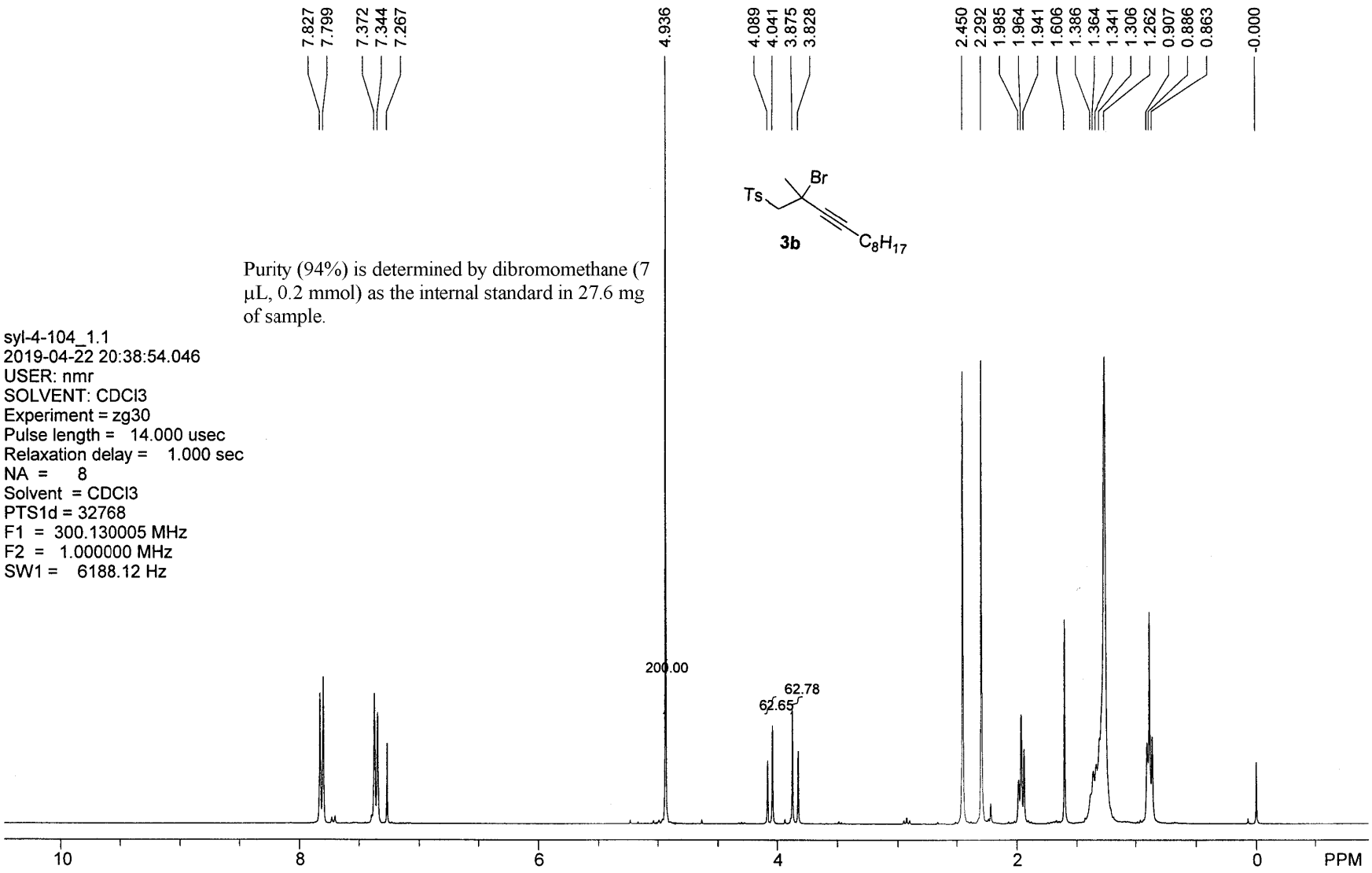

145.130
135.984
129.761
128.575
91.312
77.423
77.000
76.577
76.531
63.929
31.730
29.065
28.945
28.761
27.888
27.254
22.575
21.610
18.825
14.045
Br
Ts
C_8H_{17}
3a
syl-4-135_2.1
2019-05-13 22:15:16.546
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 72
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
200
150
100
50
0 PPM

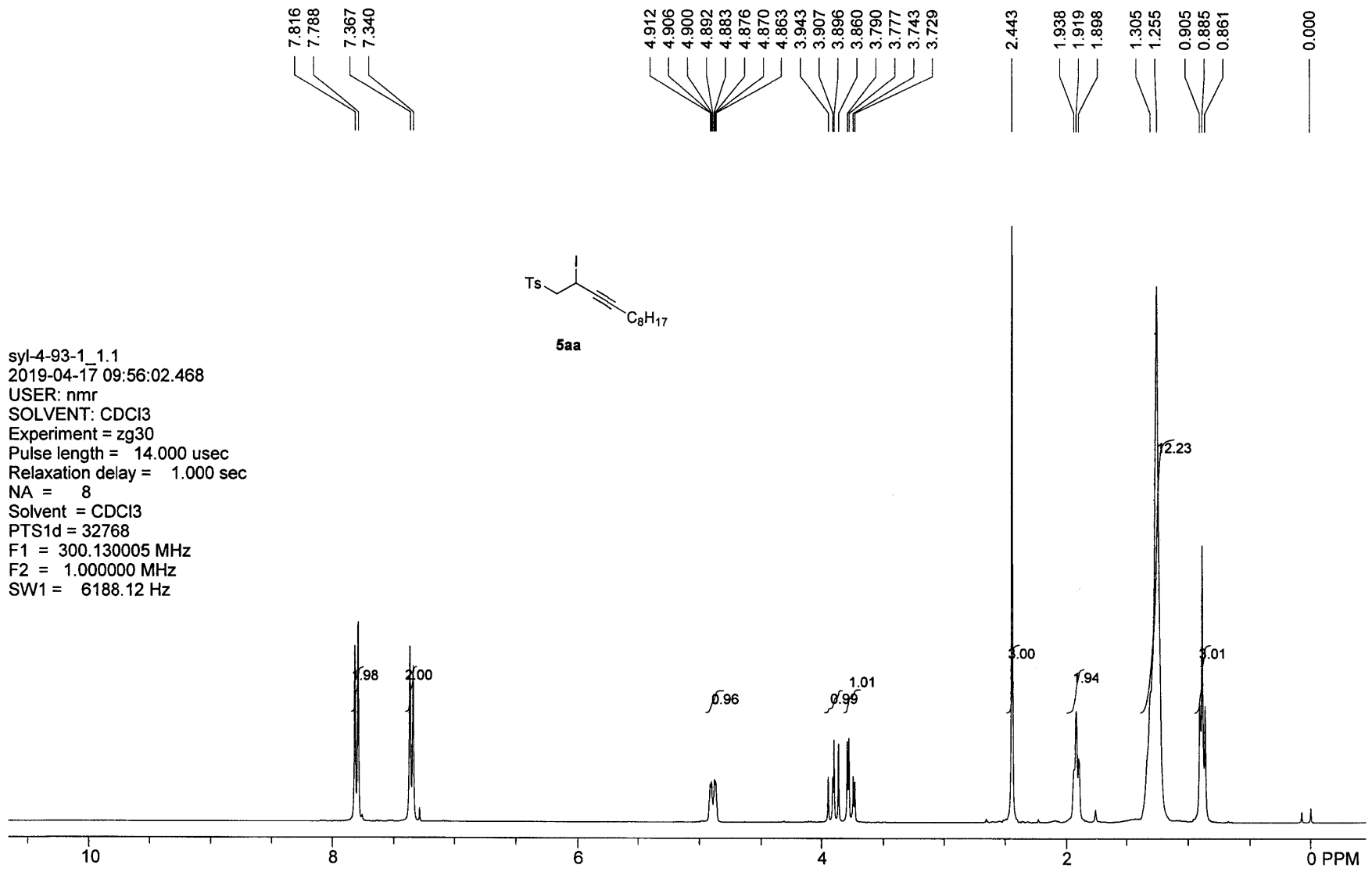

7.826
7.799
7.371
7.344
7.276
4.093
4.046
3.878
3.831
2.448
2.294
1.979
1.958
1.934
1.357
1.335
1.261
0.905
0.885
0.863
-0.000
Br
Ts
C8H17
3b
syl-4-81_1.1
2019-04-10 22:12:28.968
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
2.00
2.07
1.01
0.99
3.23
2.98
1.97
13.85
3.46
10
8
6
4
2
0
PPM

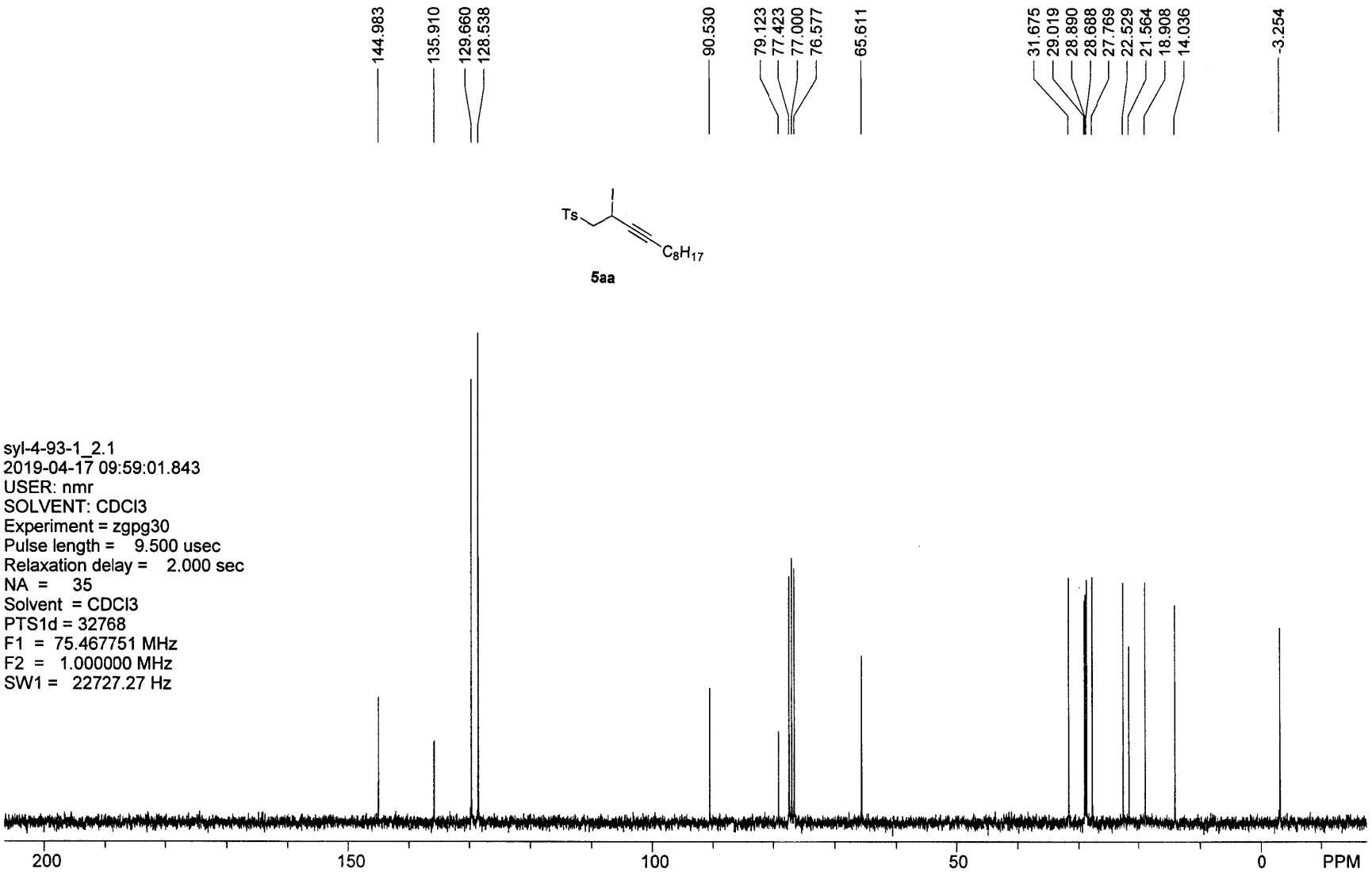

7.827
7.800
7.373
7.346
7.268
4.938
4.092
4.044
3.877
3.830
2.451
2.294
1.982
1.961
1.937
1.360
1.337
1.261
0.905
0.885
0.863
0.000
Br
Ts
C8H17
3b
syl-4-81-p_1.1
2019-04-16 13:43:49.140
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
Purity is determined by dibromomethane (7 μL, 0.2 mmol) as the internal standard in 28.9 mg of sample.
200.00
64.74
10
8
6
4
2
0
PPM

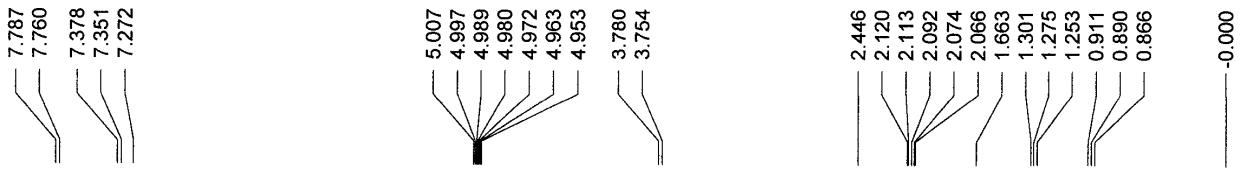

144.863
137.133
129.678
128.575
90.742
80.704
77.423
77.000
76.577
68.948
46.713
34.166
31.758
29.101
28.973
28.798
27.897
22.603
21.610
18.908
14.064
Ts
Br
3b
C_8H_{17}
syl-4-81_2.1
2019-04-16 13:19:37.000
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 120
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
200
150
100
50
0
PPM

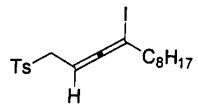

7.826
7.799
7.370
7.343
7.268
4.088
4.040
3.875
3.827
2.448
2.292
1.985
1.965
1.941
1.364
1.342
1.308
1.262
0.907
0.885
0.862
0.000
Br
Ts
C8H17
3b
syl-4-104_1.1
2019-04-22 22:21:51.406
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
2.00
2.04
1.00
1.00
3.10
2.98
1.95
13.25
3.31
10
8
6
4
2
0
PPM


7.827
7.799
7.372
7.344
7.267
4.936
4.089
4.041
3.875
3.828
2.450
2.292
1.985
1.964
1.941
1.606
1.386
1.364
1.341
1.306
1.262
0.907
0.886
0.863
-0.000
Br
Ts
3b
C_8H_{17}
Purity (94%) is determined by dibromomethane (7 μL, 0.2 mmol) as the internal standard in 27.6 mg of sample.
syl-4-104_1.1
2019-04-22 20:38:54.046
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
200.00
62.78
62.65
10
8
6
4
2
0
PPM


7.816
7.788
7.367
7.340
4.912
4.906
4.900
4.892
4.883
4.876
4.870
4.863
3.943
3.907
3.896
3.860
3.790
3.777
3.743
3.729
2.443
1.938
1.919
1.898
1.305
1.255
0.905
0.885
0.861
0.000
Ts
I
C8H17
5aa
syl-4-93-1_1.1
2019-04-17 09:56:02.468
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
1.98
2.00
0.96
0.99
1.01
3.00
1.94
12.23
3.01
10
8
6
4
2
0 PPM

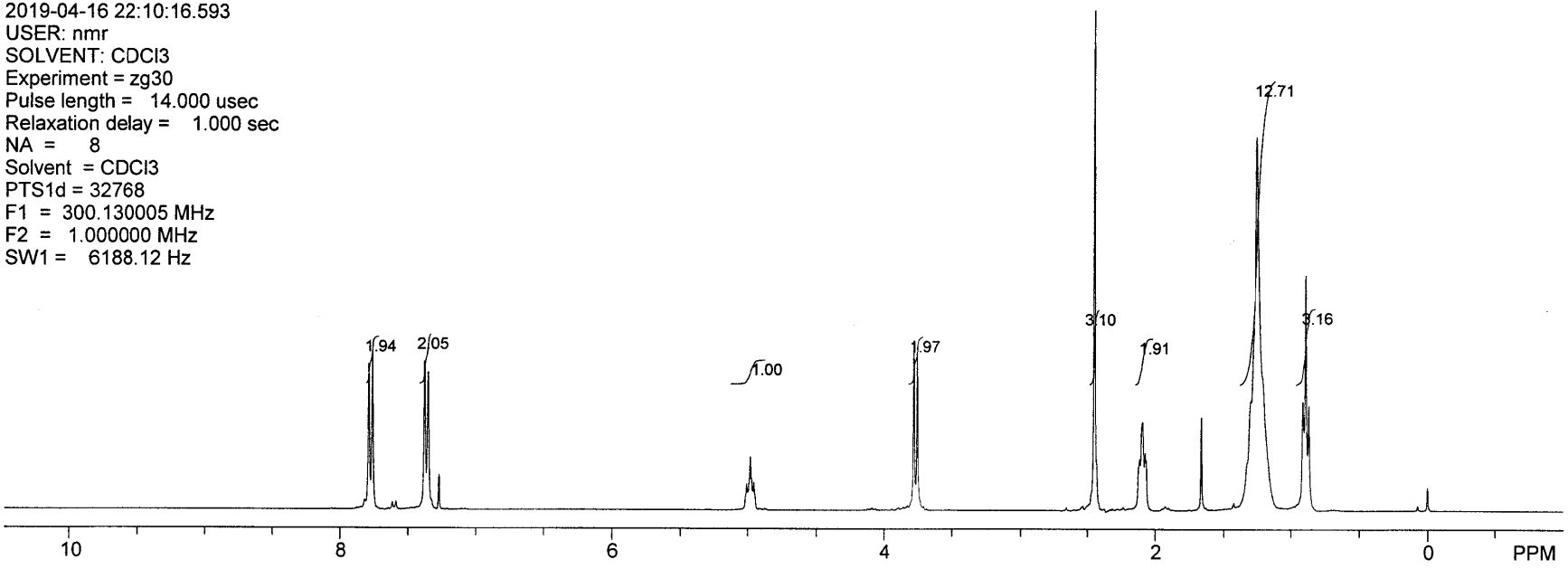

144.983
135.910
129.660
128.538
90.530
79.123
77.423
77.000
76.577
65.611
31.675
29.019
28.890
28.688
27.769
22.529
21.564
18.908
14.036
-3.254
Ts
I
C_8H_{17}
5aa
syl-4-93-1_2.1
2019-04-17 09:59:01.843
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 35
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
200
150
100
50
0
PPM

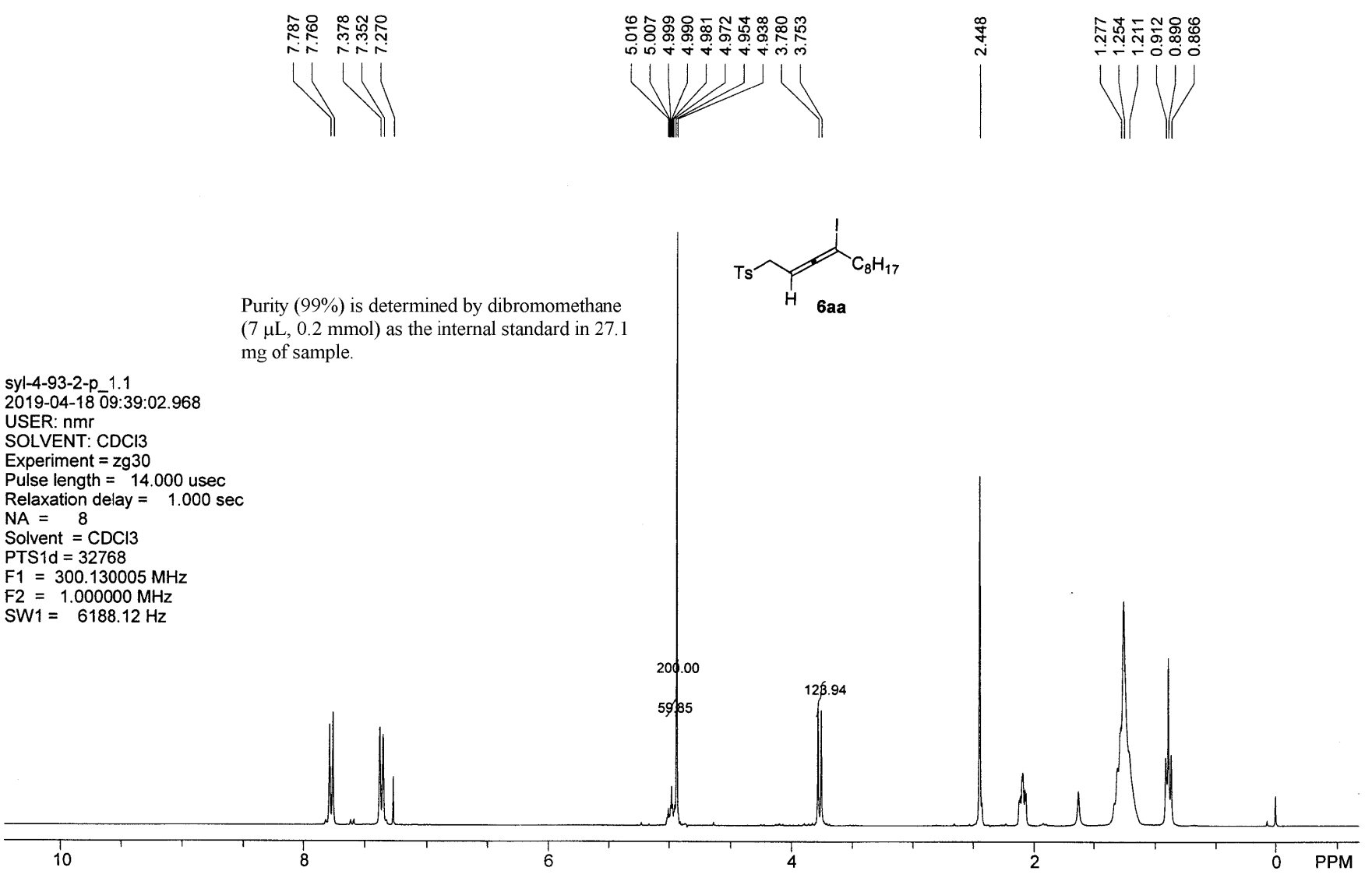


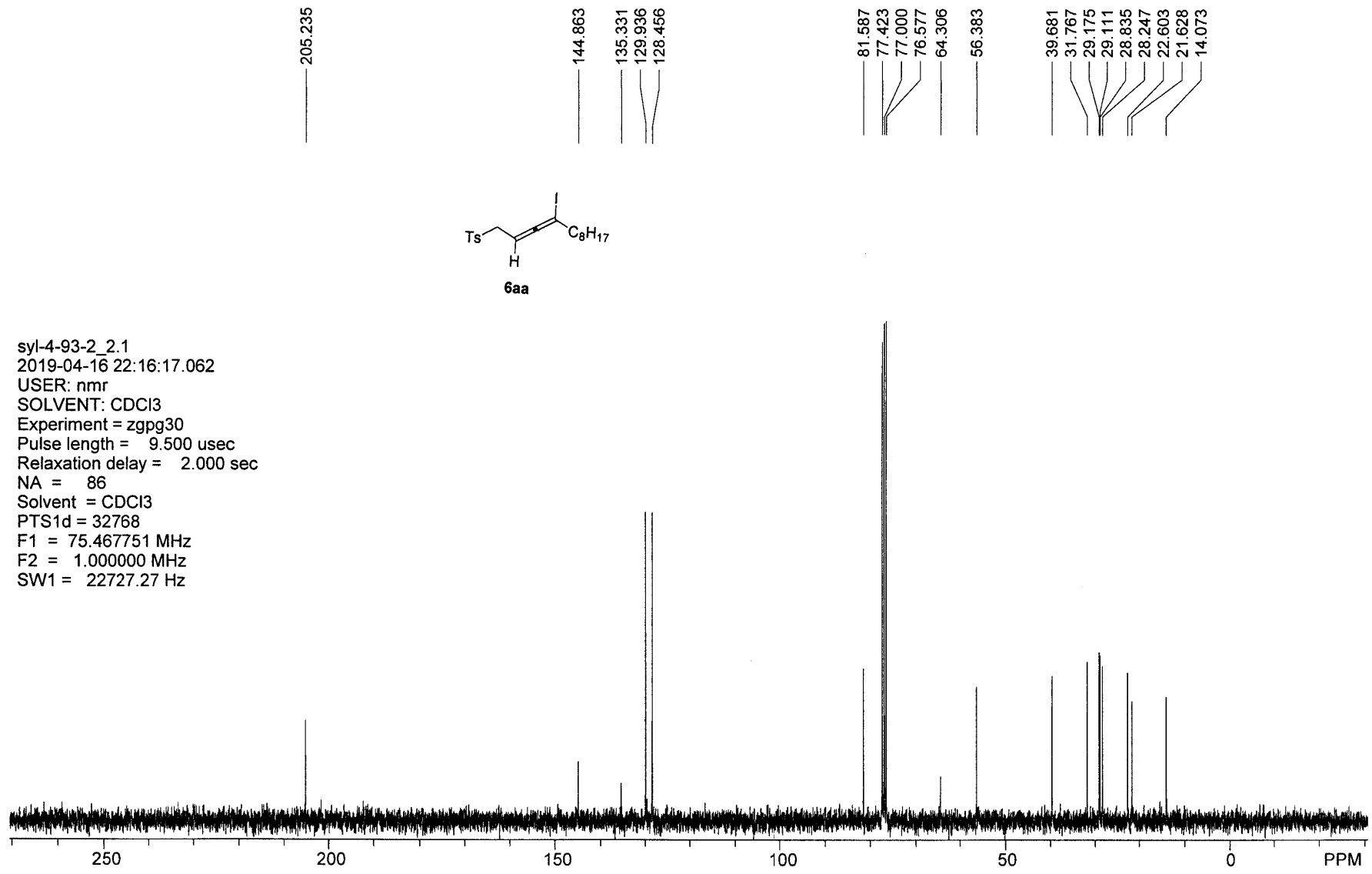


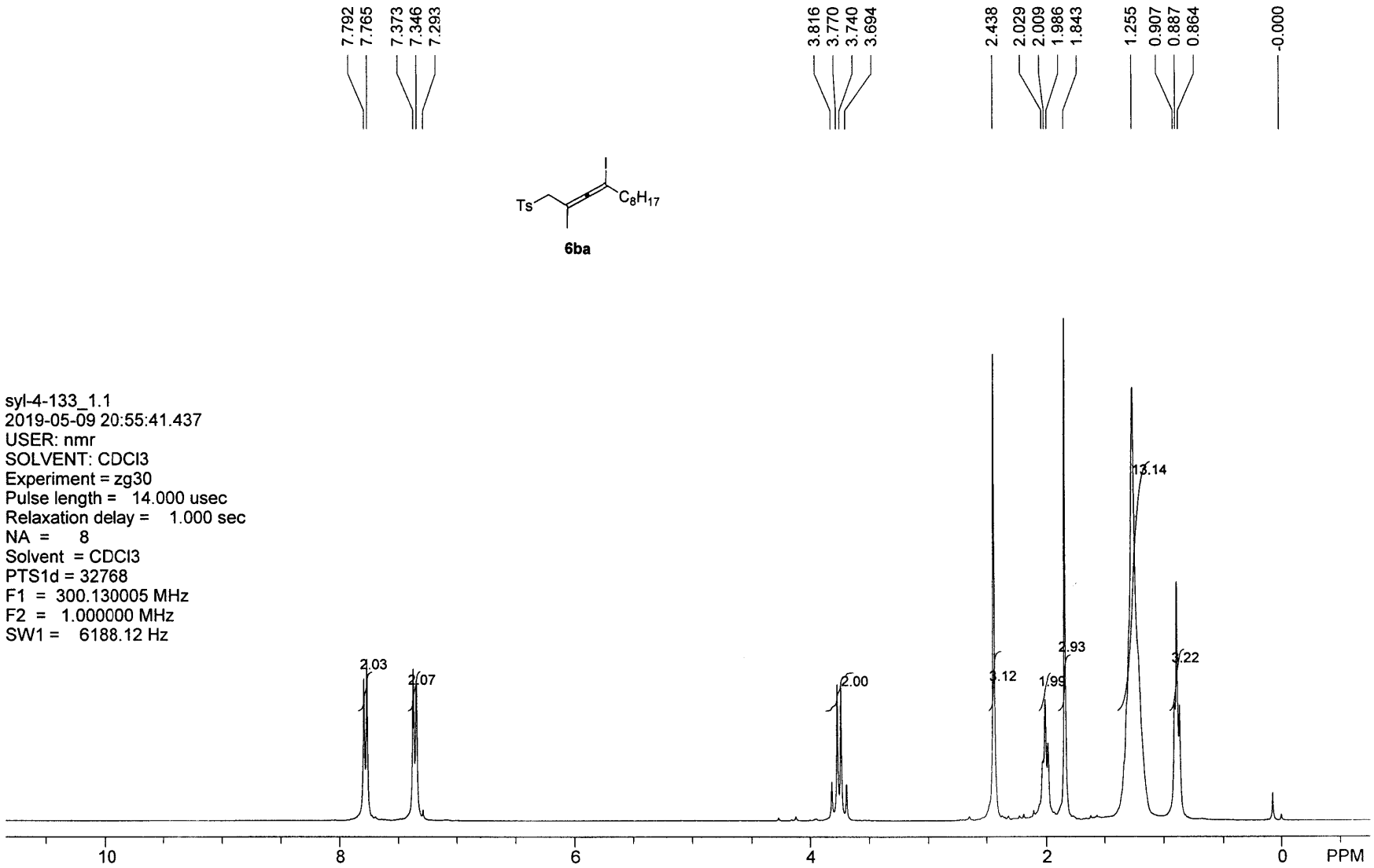
6aa

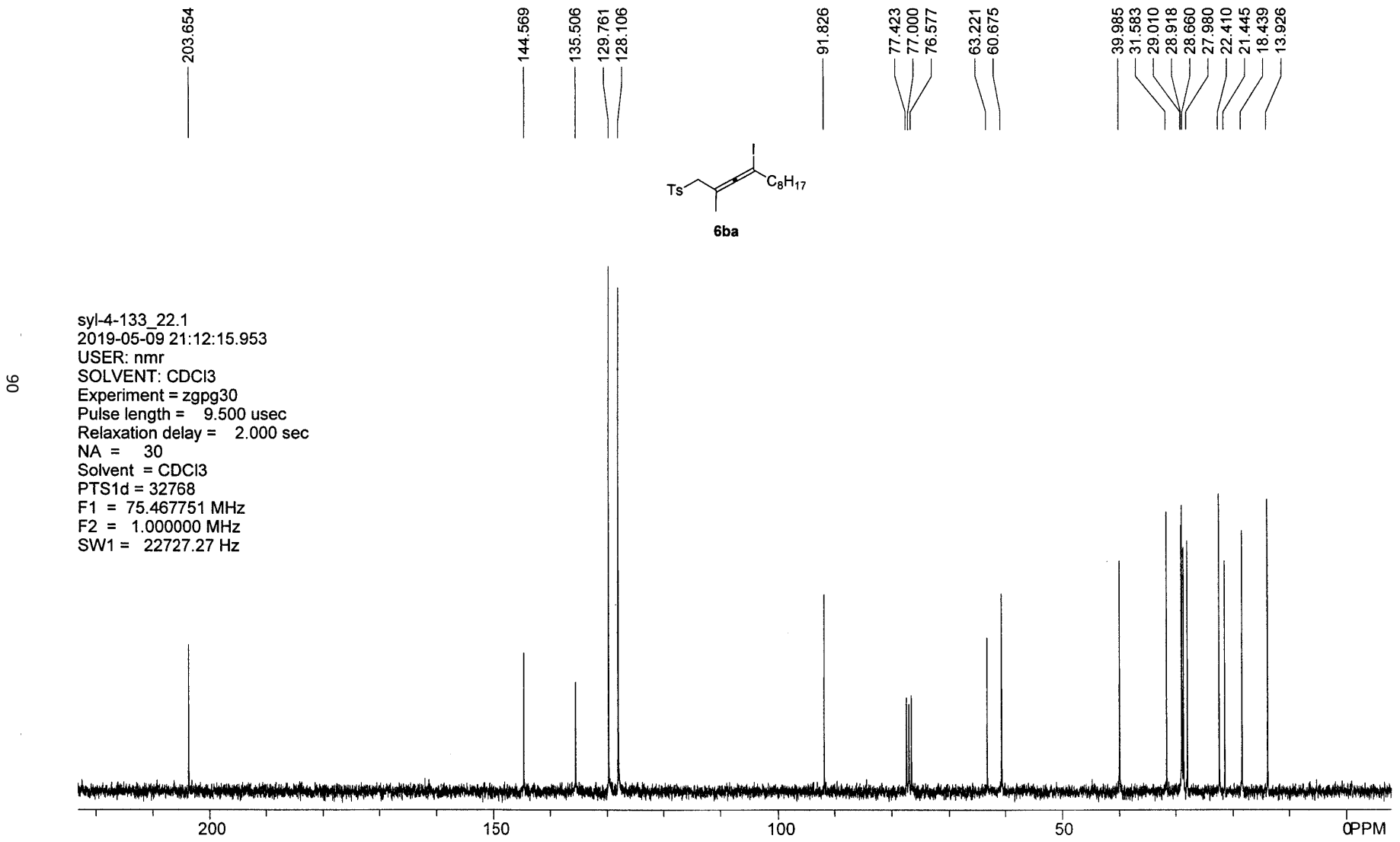
syl-4-93-2_1.1
2019-04-16 22:10:16.593
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz

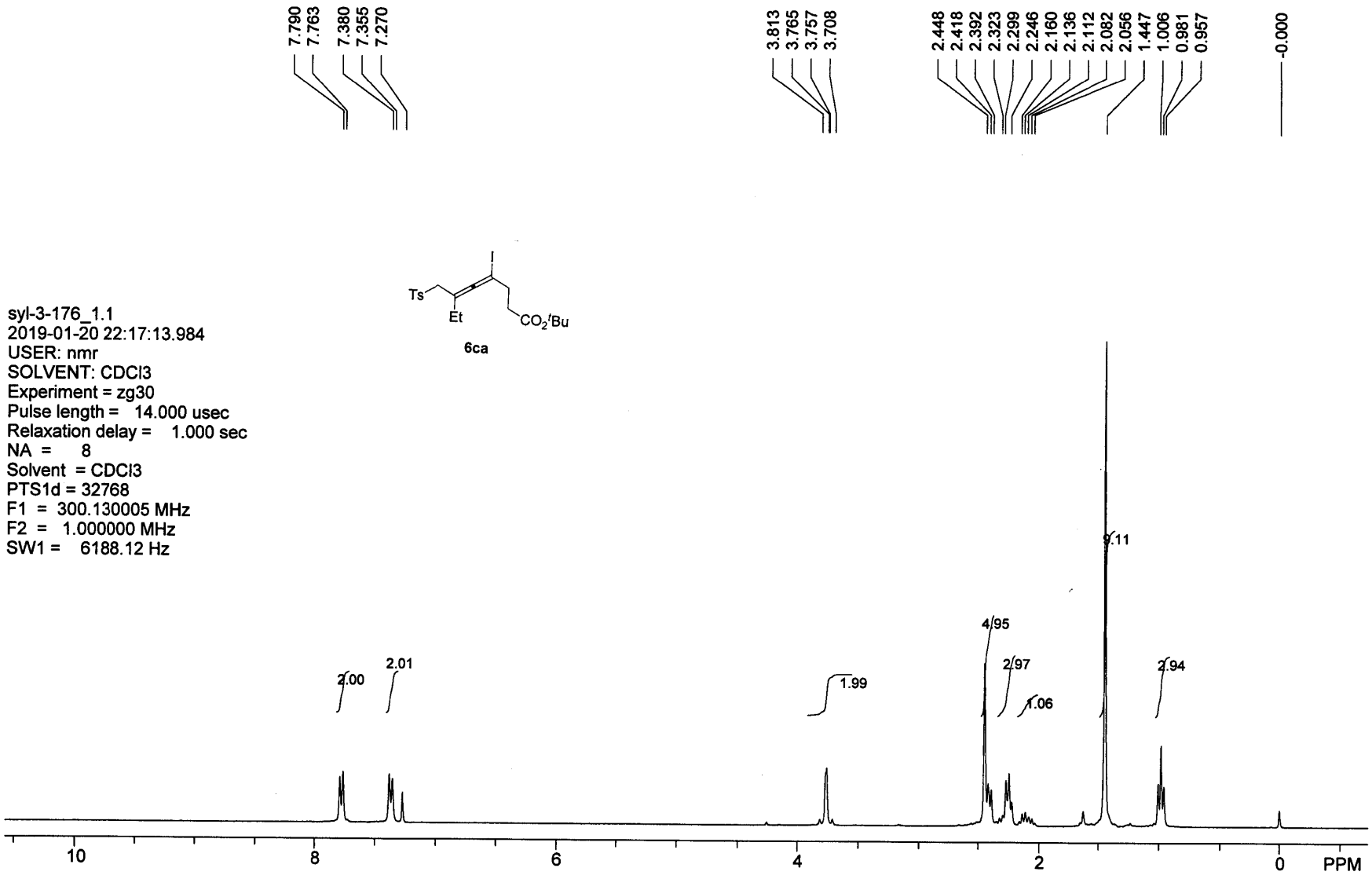


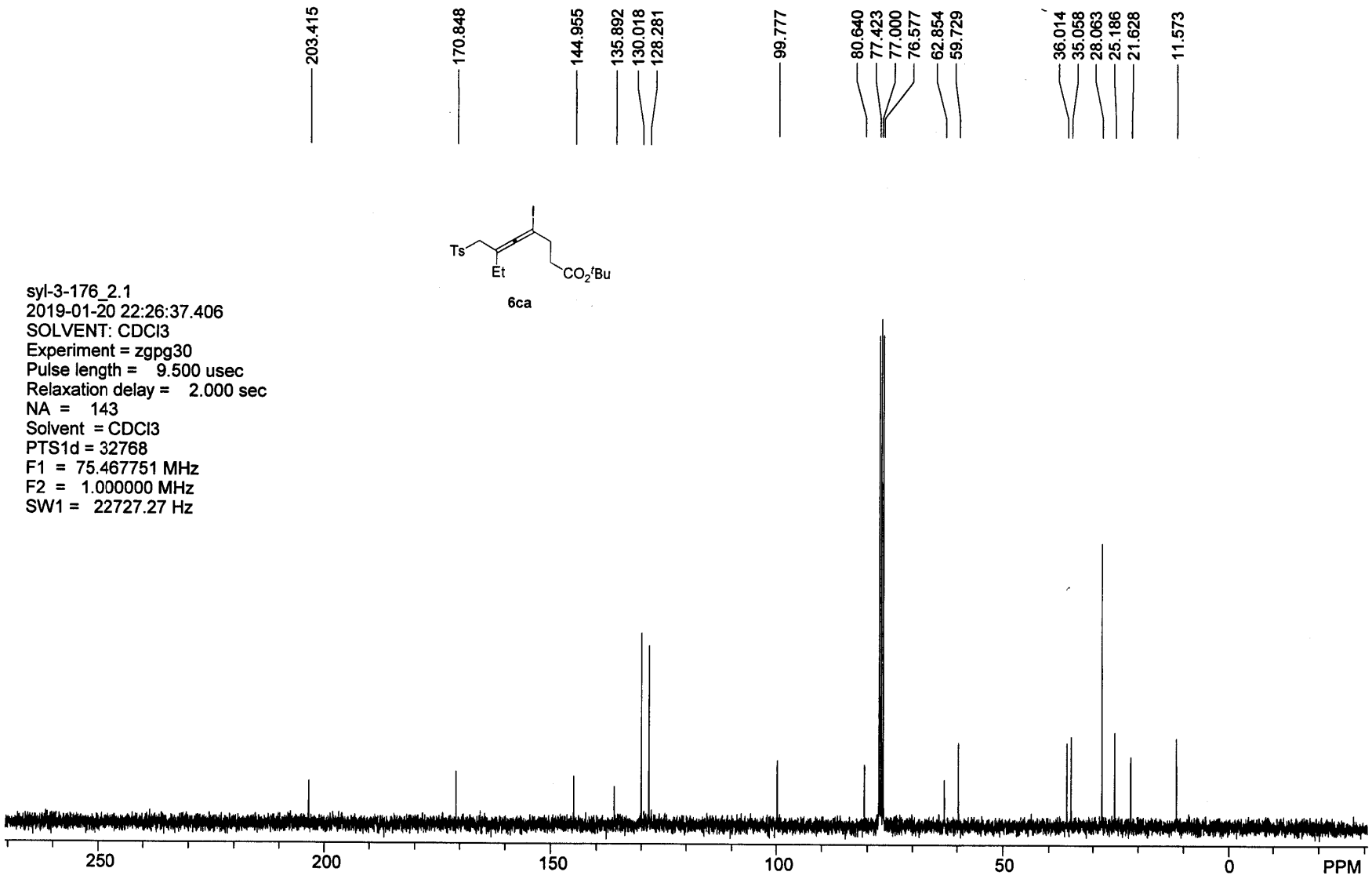

7.787
7.760
7.378
7.352
7.270
5.016
5.007
4.999
4.990
4.981
4.972
4.954
4.938
3.780
3.753
2.448
1.277
1.254
1.211
0.912
0.890
0.866
Ts
H
C_8H_{17}
6aa
Purity (99%) is determined by dibromomethane (7 μL, 0.2 mmol) as the internal standard in 27.1 mg of sample.
syl-4-93-2-p_1.1
2019-04-18 09:39:02.968
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
200.00
59.85
128.94
10
8
6
4
2
0
PPM

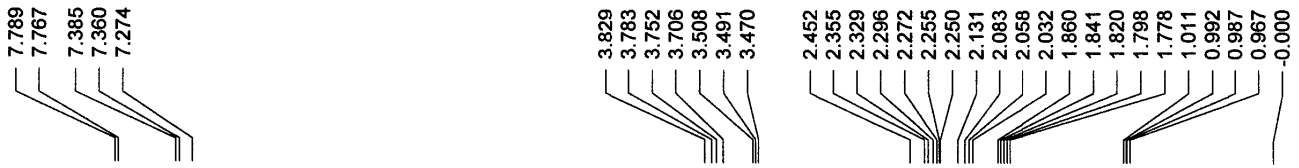

205.235
144.863
135.331
129.936
128.456
81.587
77.423
77.000
76.577
64.306
56.383
39.681
31.767
29.175
29.111
28.835
28.247
22.603
21.628
14.073
Ts
I
C_8H_{17}
H
6aa
syl-4-93-2_2.1
2019-04-16 22:16:17.062
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 86
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
250
200
150
100
50
0
PPM

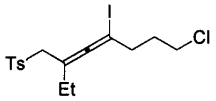

7.792
7.765
7.373
7.346
7.293
3.816
3.770
3.740
3.694
2.438
2.029
2.009
1.986
1.843
1.255
0.907
0.887
0.864
-0.000
Ts
C8H17
6ba
syl-4-133_1.1
2019-05-09 20:55:41.437
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
2.03
2.07
2.00
3.12
1.99
2.93
13.14
3.22
10
8
6
4
2
0
PPM


203.654
144.569
135.506
129.761
128.106
91.826
77.423
77.000
76.577
63.221
60.675
39.985
31.583
29.010
28.918
28.660
27.980
22.410
21.445
18.439
13.926
Ts
I
C8H17
6ba
syl-4-133_22.1
2019-05-09 21:12:15.953
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 30
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
200
150
100
50
0
PPM


7.790
7.763
7.380
7.355
7.270
3.813
3.765
3.757
3.708
2.448
2.418
2.392
2.323
2.299
2.246
2.160
2.136
2.112
2.082
2.056
1.447
1.006
0.981
0.957
-0.000
Ts
Et
I
CO2tBu
6ca
syl-3-176_1.1
2019-01-20 22:17:13.984
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
2.00
2.01
1.99
4.95
2.97
1.06
9.11
2.94
10
8
6
4
2
0
PPM

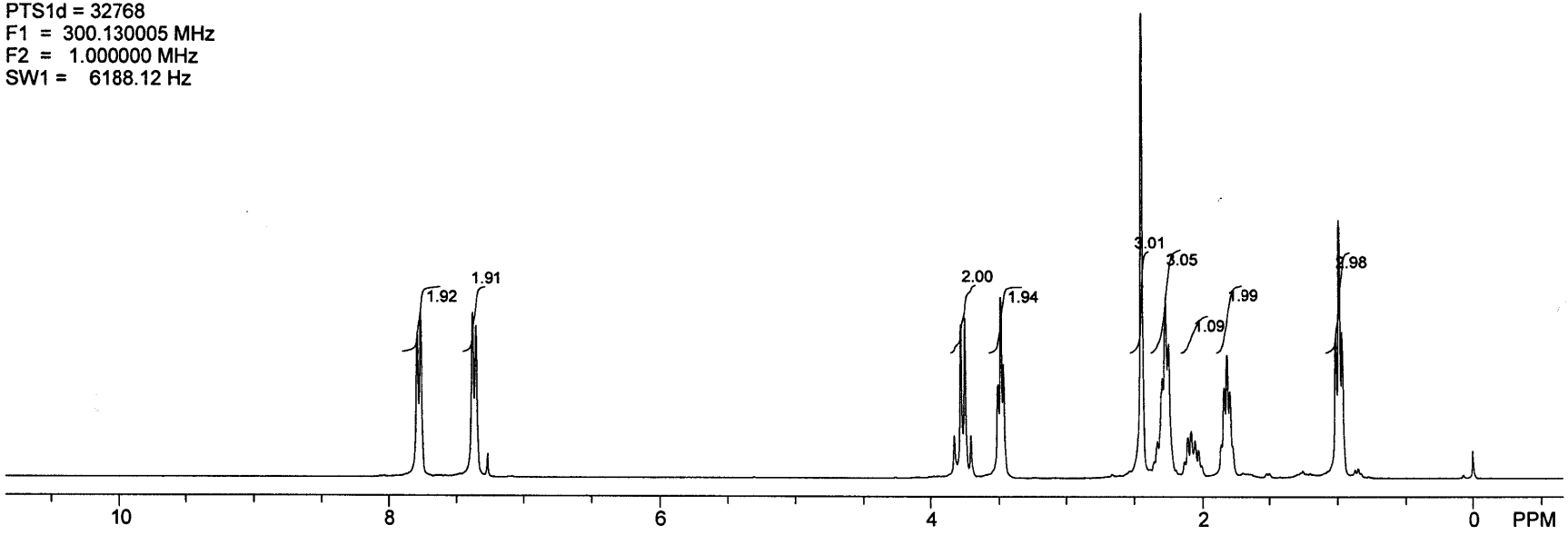

203.415
170.848
144.955
135.892
130.018
128.281
99.777
80.640
77.423
77.000
76.577
62.854
59.729
36.014
35.058
28.063
25.186
21.628
11.573
Ts
Et
CO2tBu
6ca
syl-3-176_2.1
2019-01-20 22:26:37.406
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 143
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
250
200
150
100
50
0
PPM

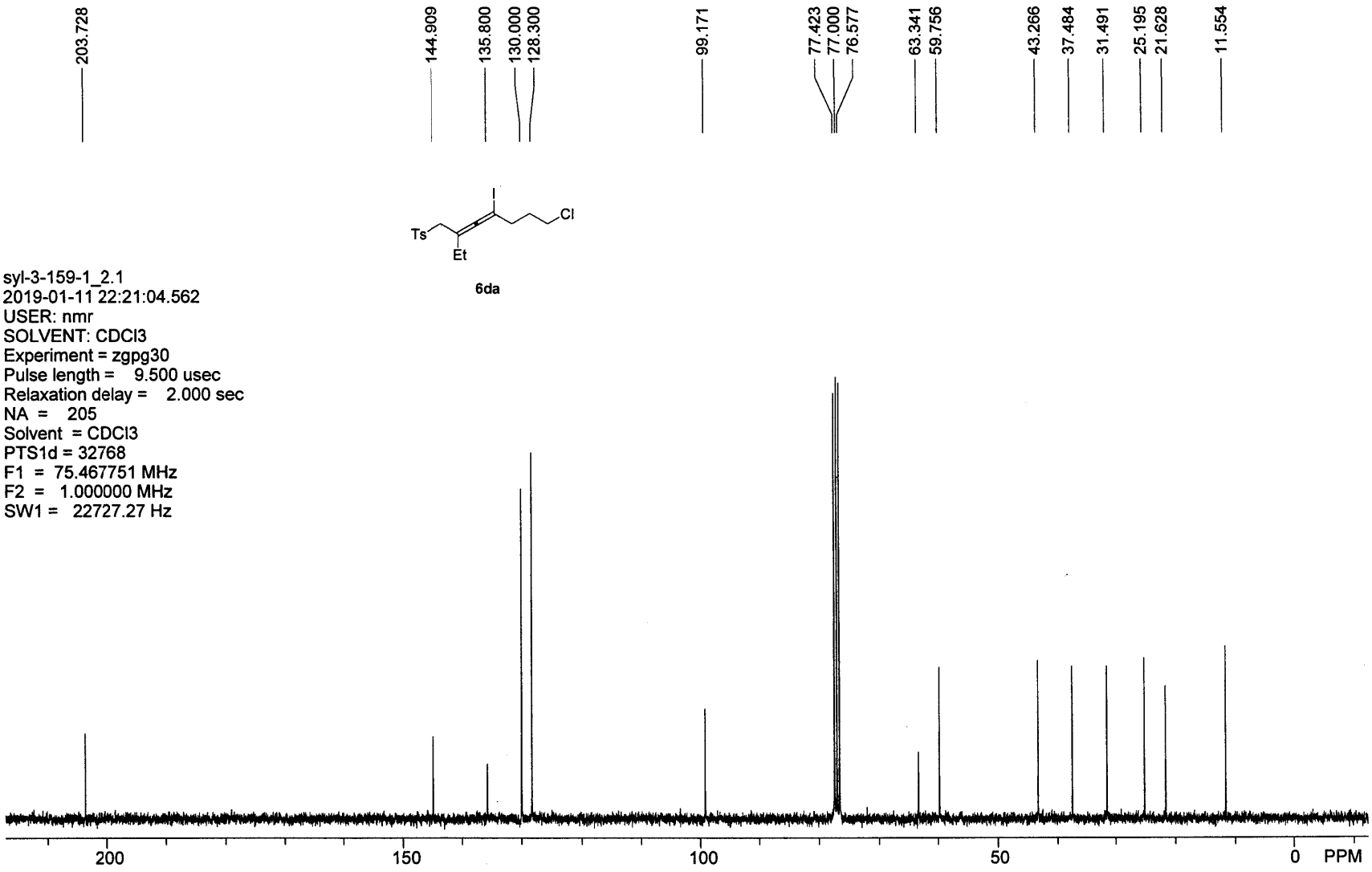


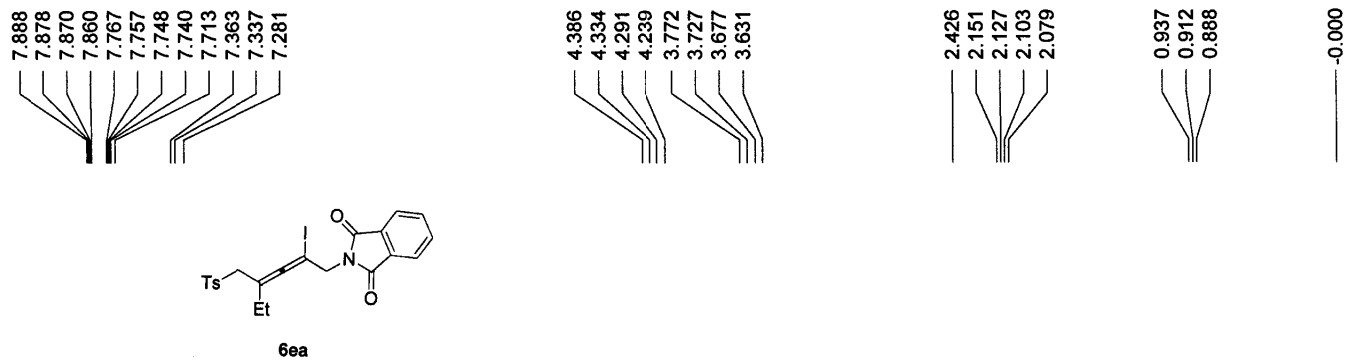


6da

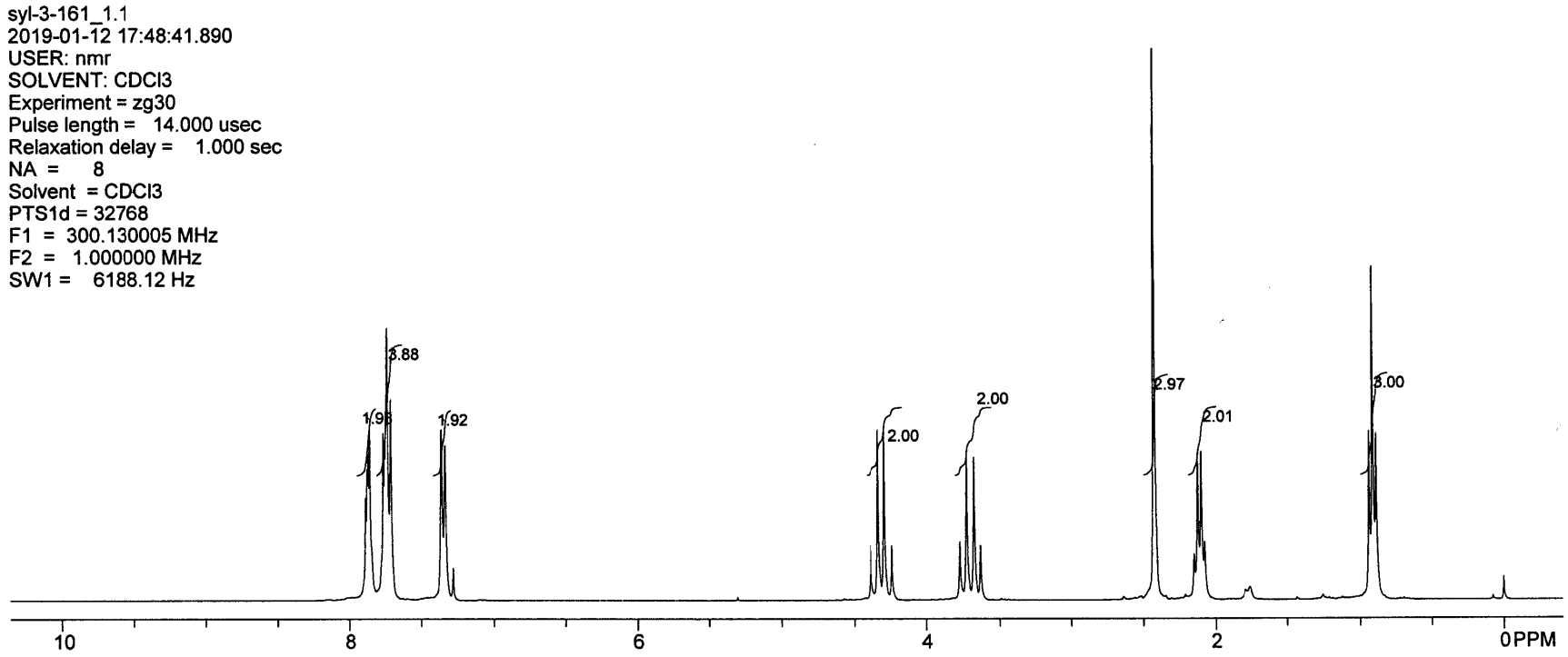
syl-3-159-1_1.1
2019-01-11 22:07:55.562
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz

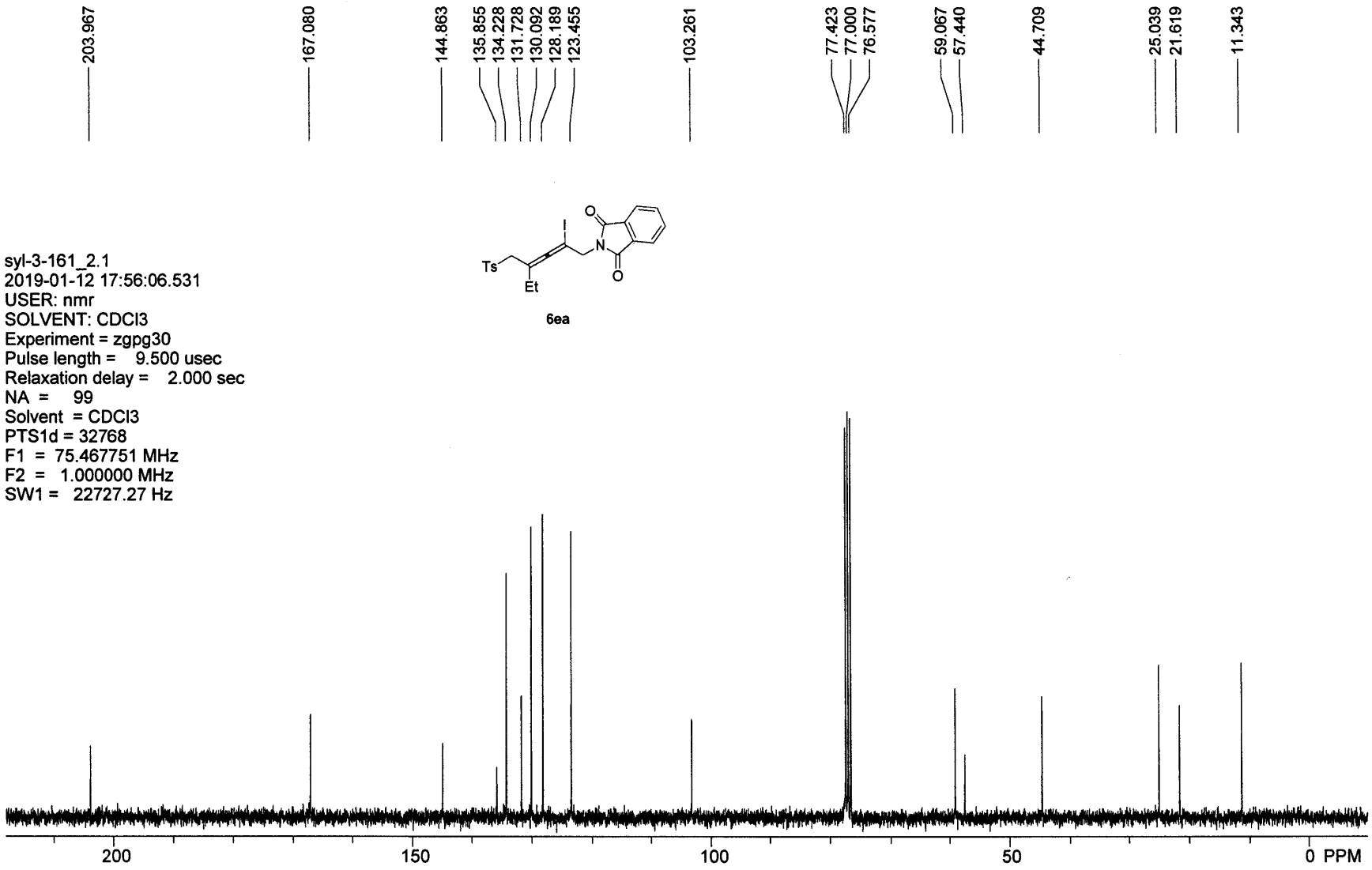


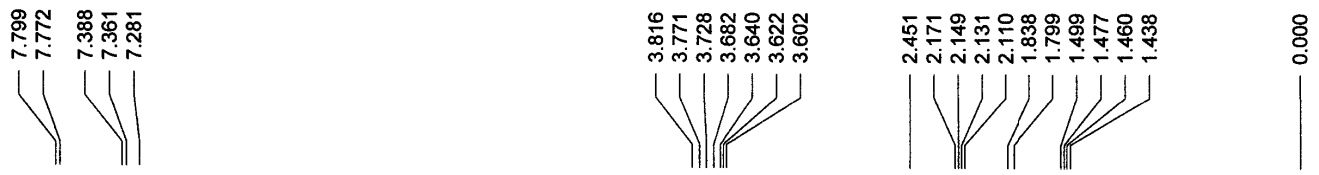

203.728
144.909
135.800
130.000
128.300
99.171
77.423
77.000
76.577
63.341
59.756
43.266
37.484
31.491
25.195
21.628
11.554
I
Cl
Ts
Et
6da
syl-3-159-1_2.1
2019-01-11 22:21:04.562
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 205
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
200
150
100
50
0
PPM

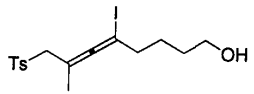

7.888
7.878
7.870
7.860
7.767
7.757
7.748
7.740
7.713
7.363
7.337
7.281
4.386
4.334
4.291
4.239
3.772
3.727
3.677
3.631
2.426
2.151
2.127
2.103
2.079
0.937
0.912
0.888
-0.000
O
I
N
O
Ts
Et
O

6ea


syl-3-161_1.1
2019-01-12 17:48:41.890
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
3.88
1.98
1.92
2.00
2.00
2.97
2.01
3.00
10
8
6
4
2
0 PPM

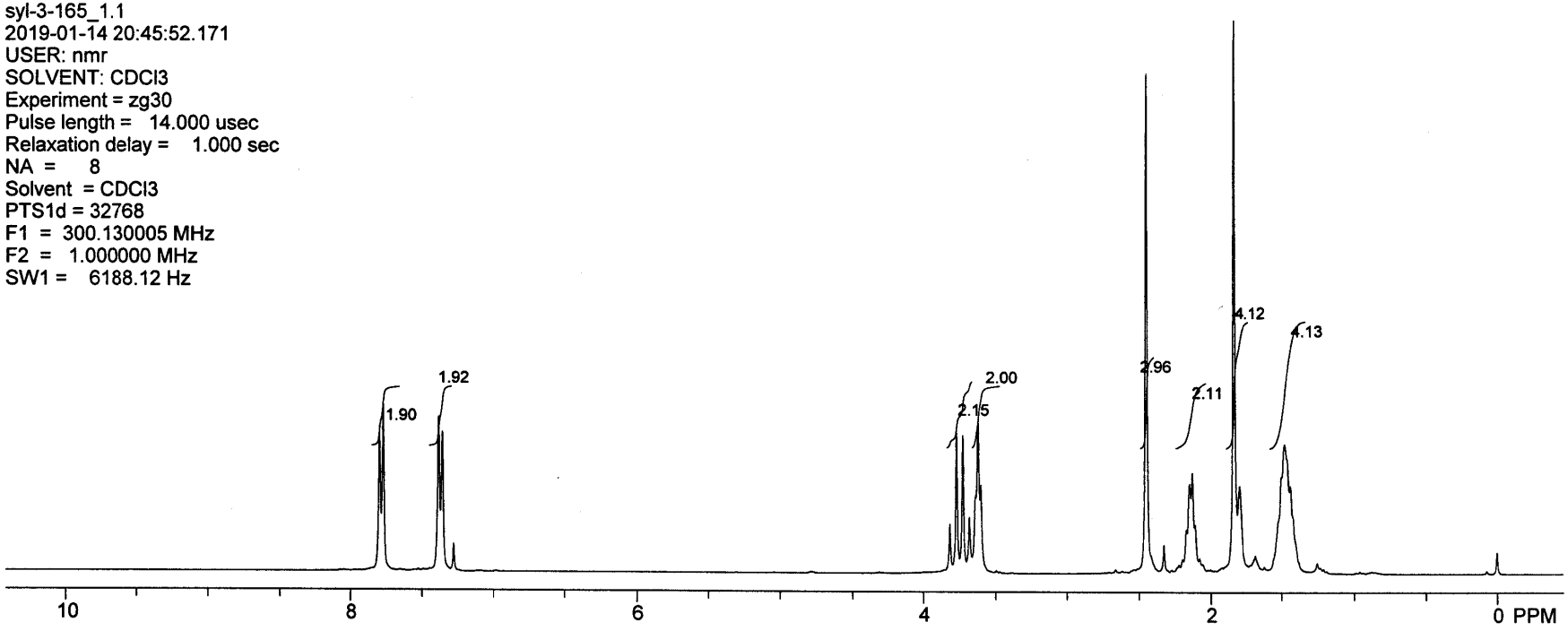

203.967
167.080
144.863
135.855
134.228
131.728
130.092
128.189
123.455
103.261
77.423
77.000
76.577
59.067
57.440
44.709
25.039
21.619
11.343
O
I
N
O
Ts
Et
6ea
syl-3-161_2.1
2019-01-12 17:56:06.531
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 99
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
200
150
100
50
0 PPM

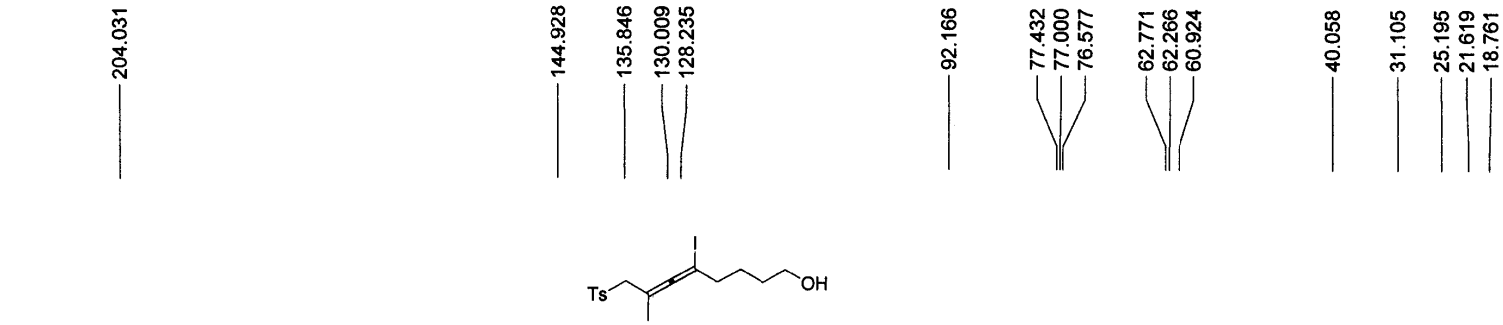




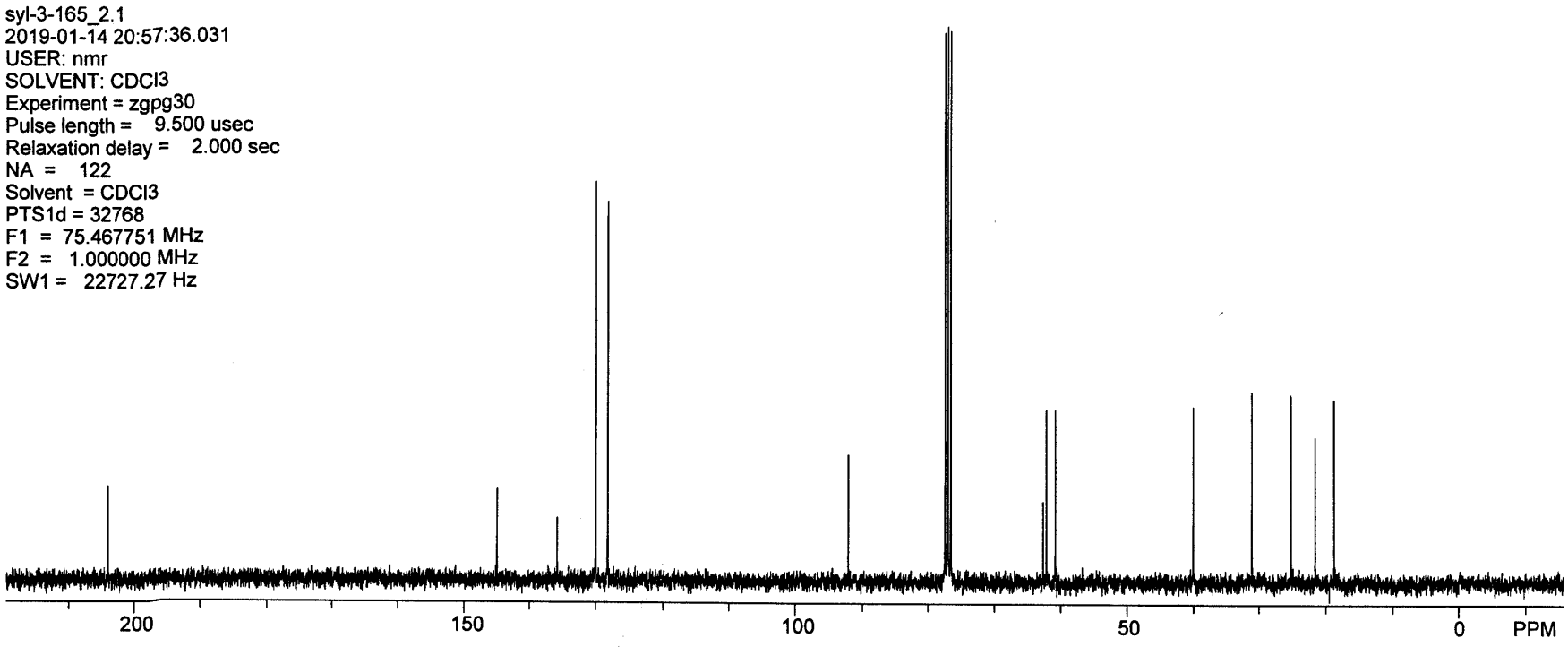
6fa

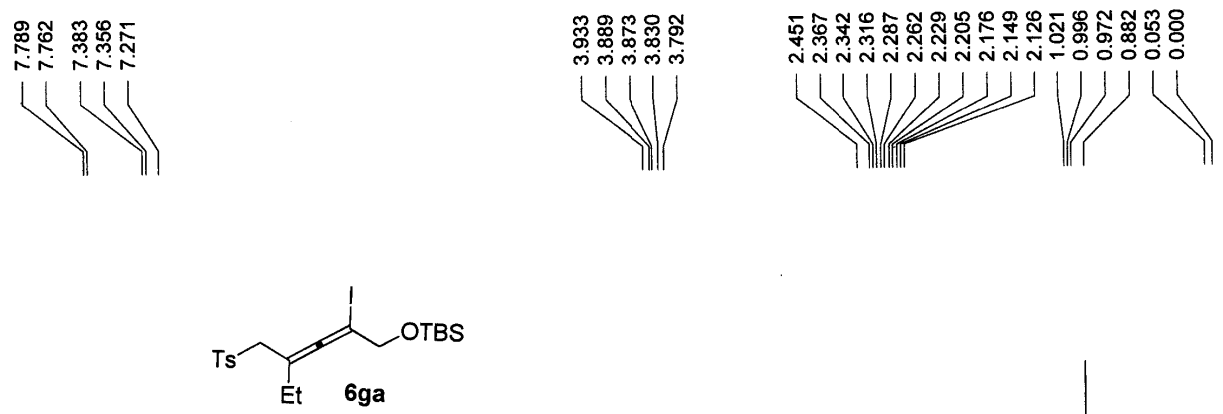
syl-3-165_1.1
2019-01-14 20:45:52.171
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz




204.031
144.928
135.846
130.009
128.235
92.166
77.432
77.000
76.577
62.771
62.266
60.924
40.058
31.105
25.195
21.619
18.761
Ts
I
OH

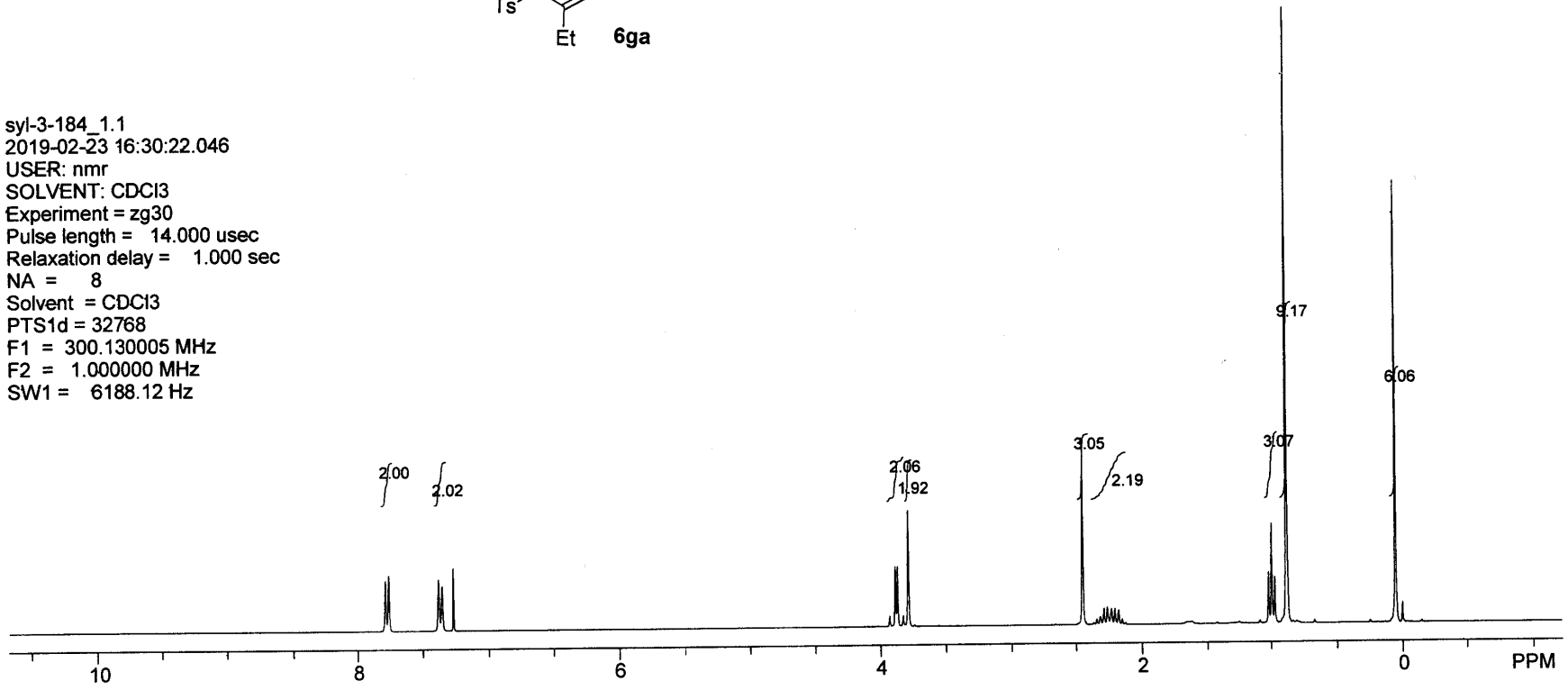
6fa

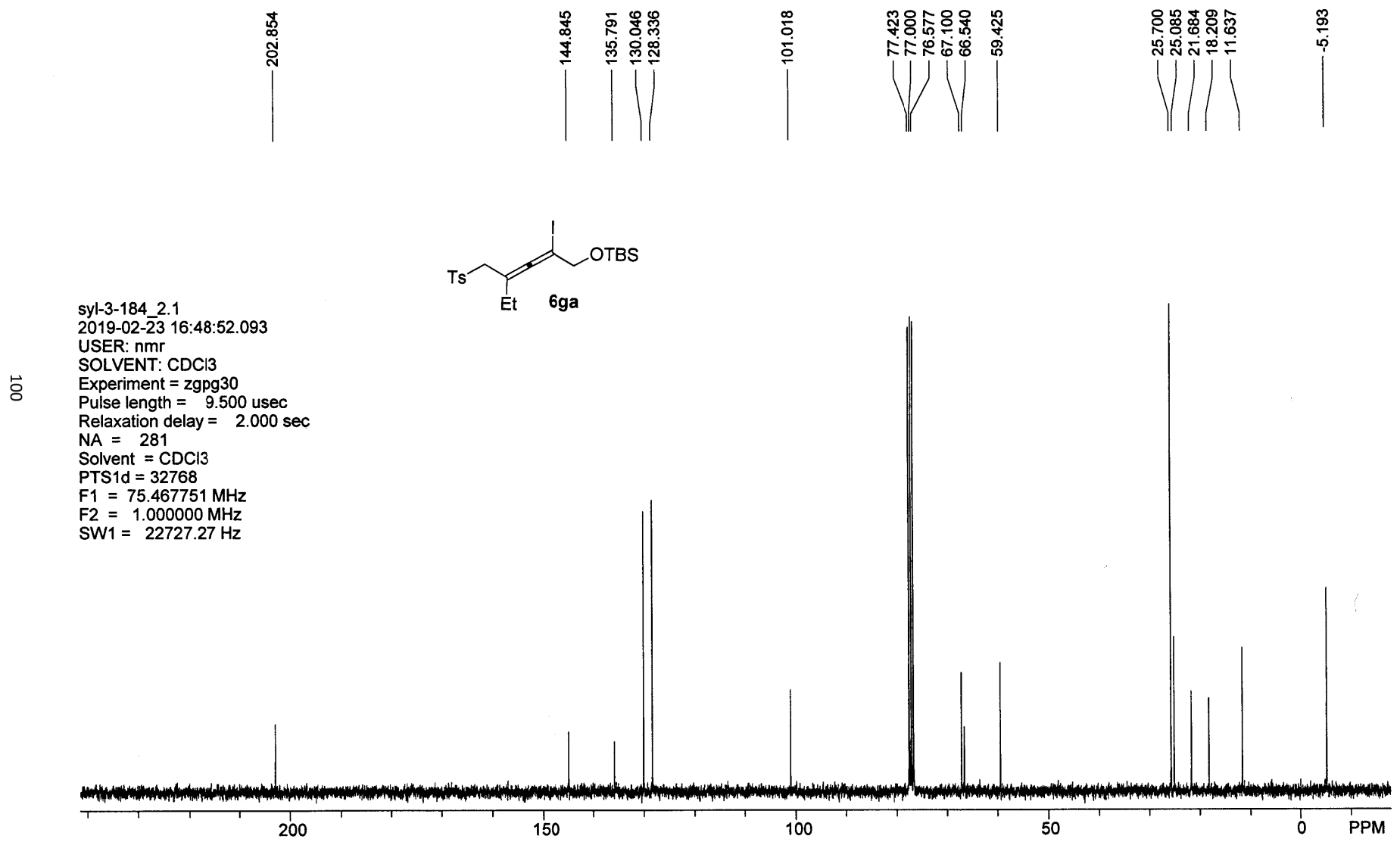

syl-3-165_2.1
2019-01-14 20:57:36.031
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 122
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
200
150
100
50
0
PPM

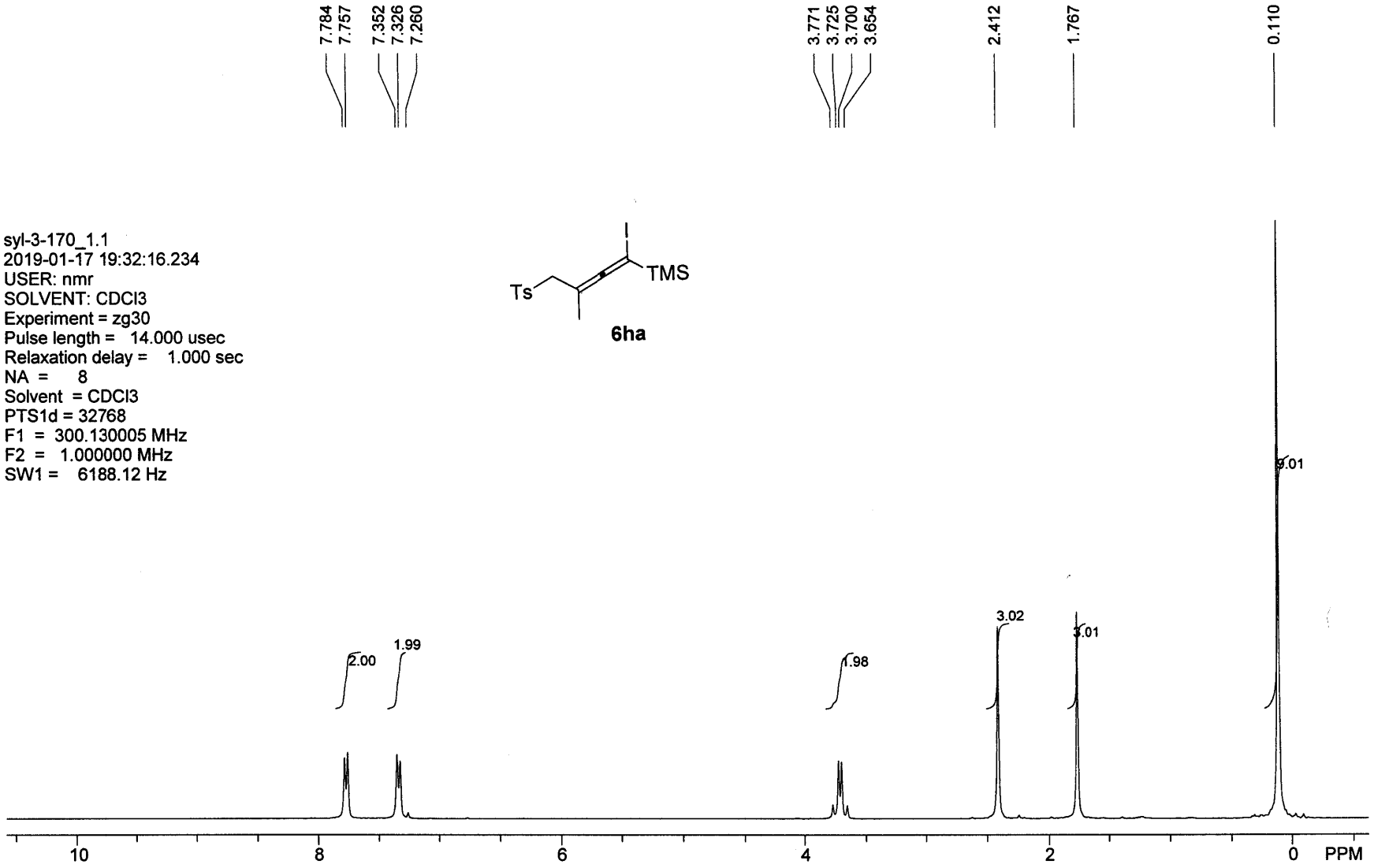

7.789
7.762
7.383
7.356
7.271
3.933
3.889
3.873
3.830
3.792
2.451
2.367
2.342
2.316
2.287
2.262
2.229
2.205
2.176
2.149
2.126
1.021
0.996
0.972
0.882
0.053
0.000

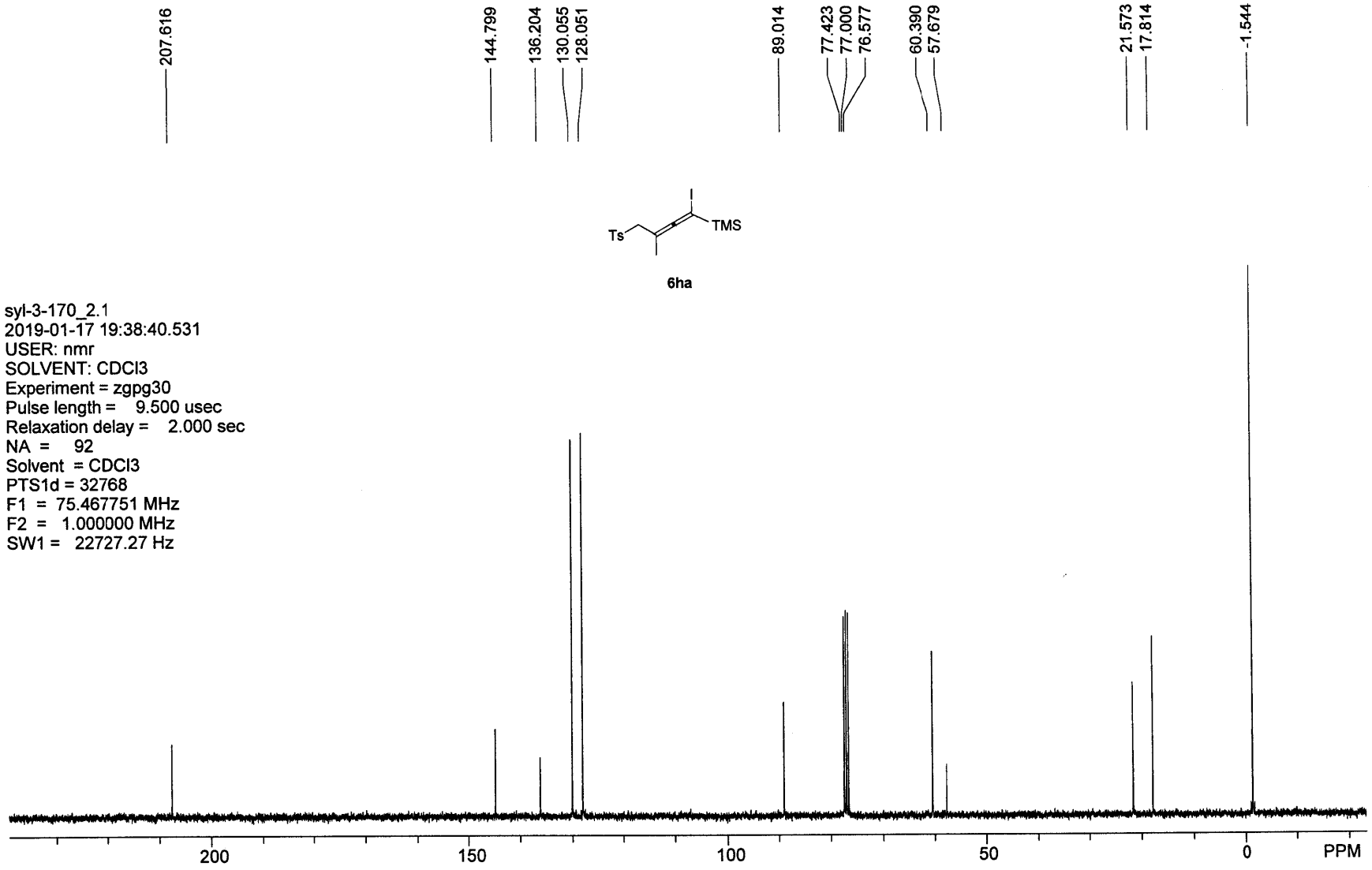
I
OTBS
Ts
Et

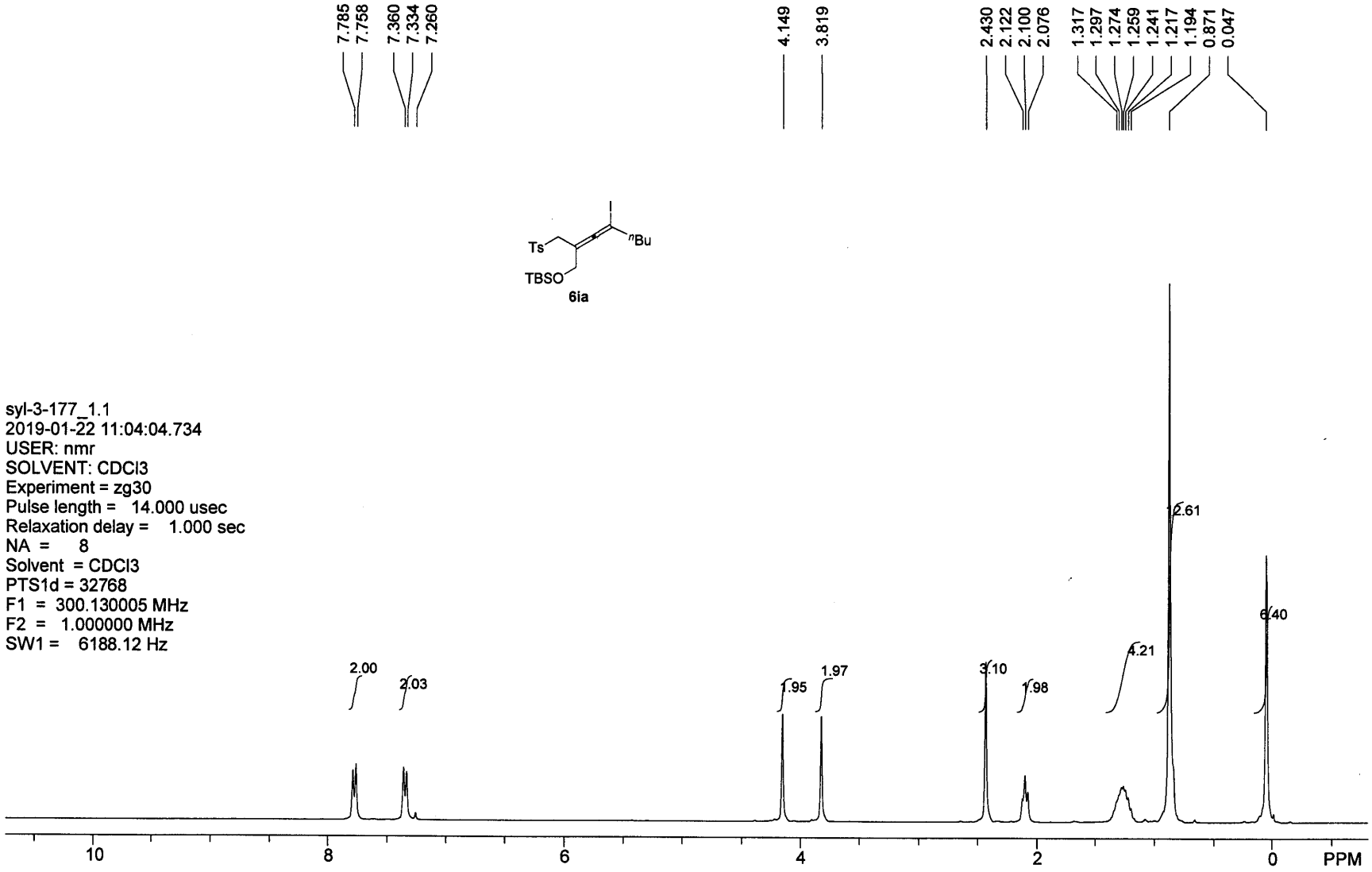
6ga

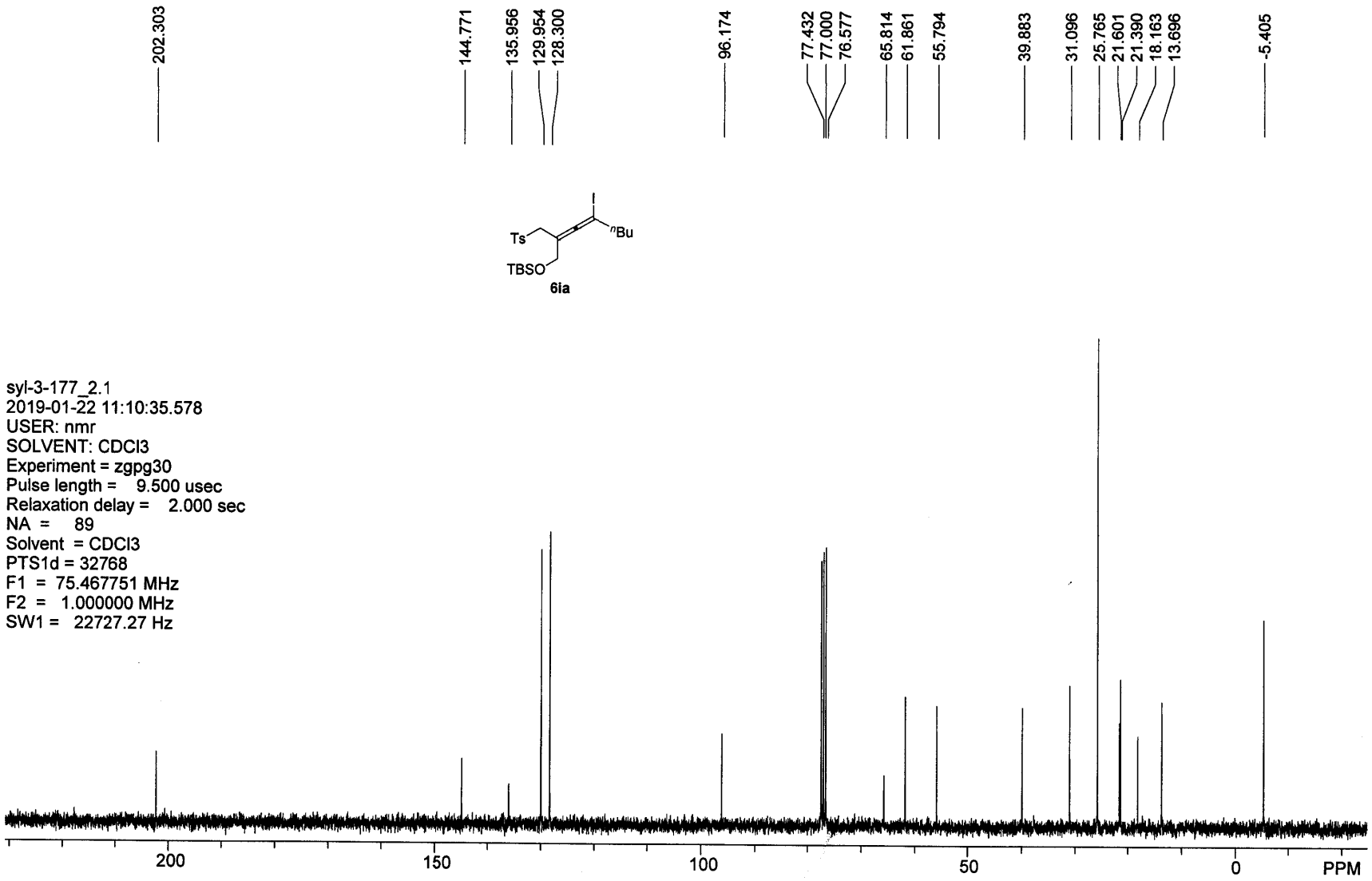

syl-3-184_1.1
2019-02-23 16:30:22.046
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
2.00
2.02
2.06
1.92
3.05
2.19
9.17
3.07
6.06
10
8
6
4
2
0
PPM

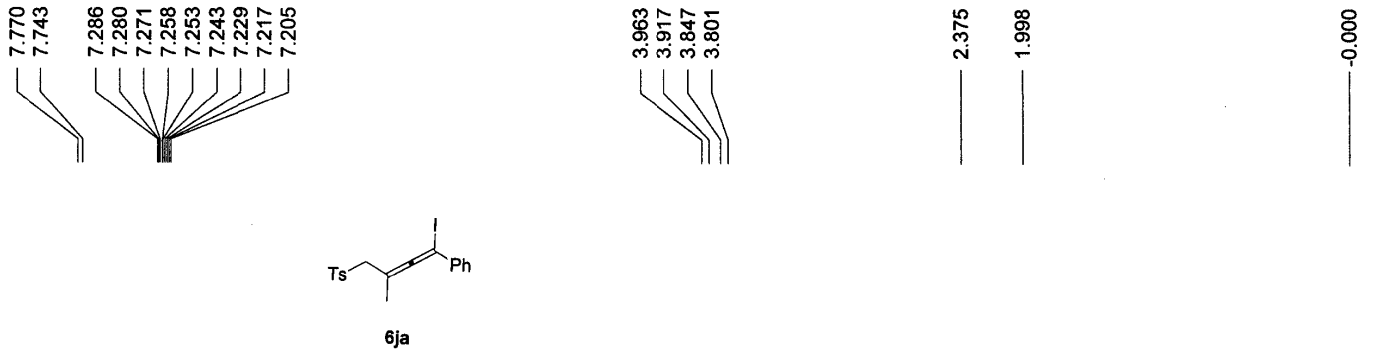

202.854
144.845
135.791
130.046
128.336
101.018
77.423
77.000
76.577
67.100
66.540
59.425
25.700
25.085
21.684
18.209
11.637
-5.193
OTBS
Ts
Et
6ga
syl-3-184_2.1
2019-02-23 16:48:52.093
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 281
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
200
150
100
50
0
PPM

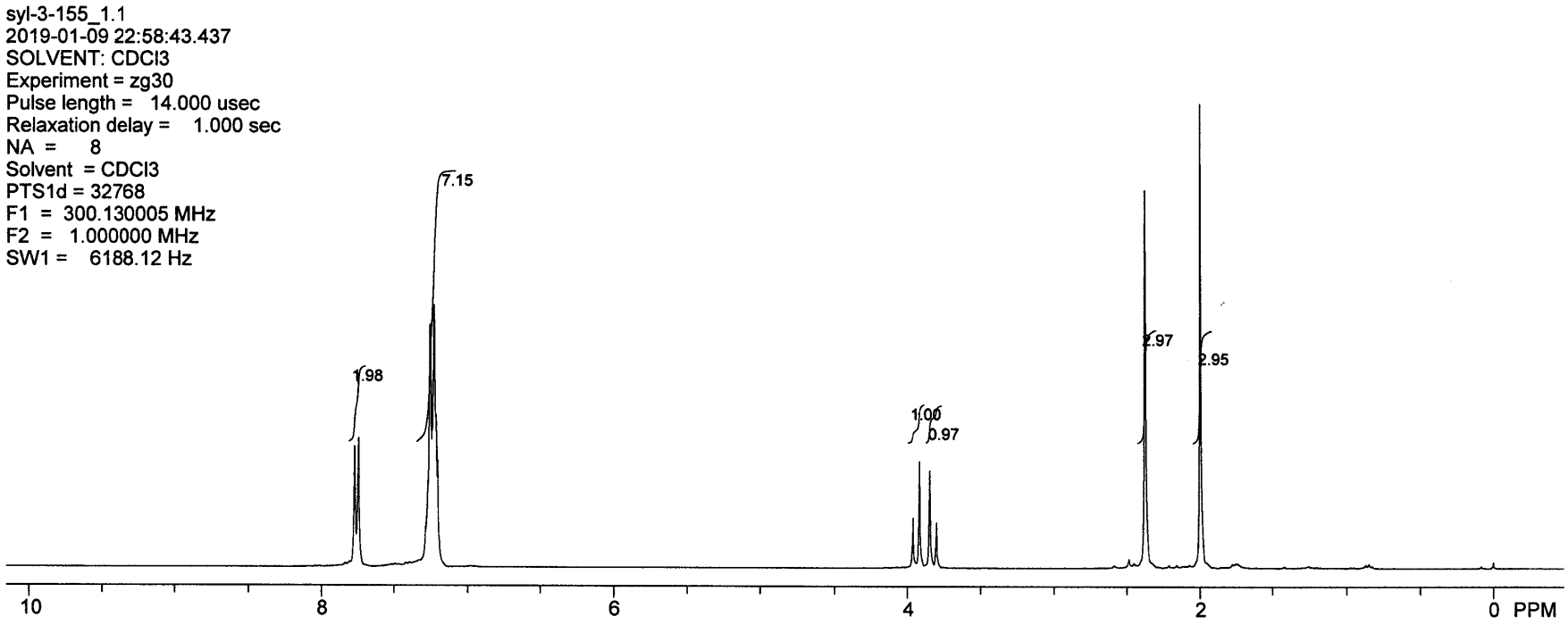

7.784
7.757
7.352
7.326
7.260
3.771
3.725
3.700
3.654
2.412
1.767
0.110
syl-3-170_1.1
2019-01-17 19:32:16.234
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
Ts
TMS
6ha
2.00
1.99
1.98
3.02
3.01
9.01
10
8
6
4
2
0
PPM

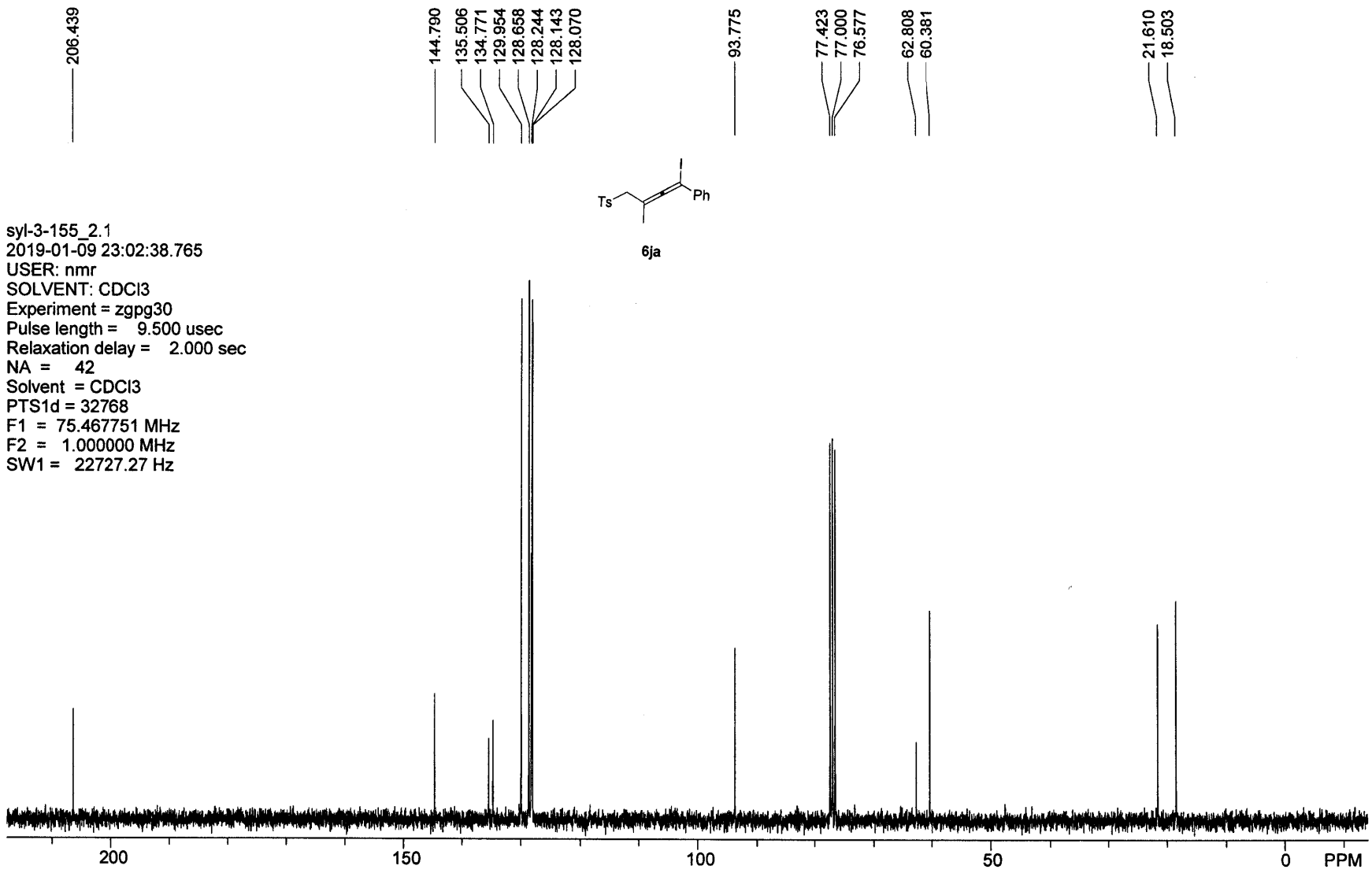

207.616
144.799
136.204
130.055
128.051
89.014
77.423
77.000
76.577
60.390
57.679
21.573
17.814
-1.544
I
TMS
Ts
6ha
syl-3-170_2.1
2019-01-17 19:38:40.531
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 92
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
200
150
100
50
0
PPM

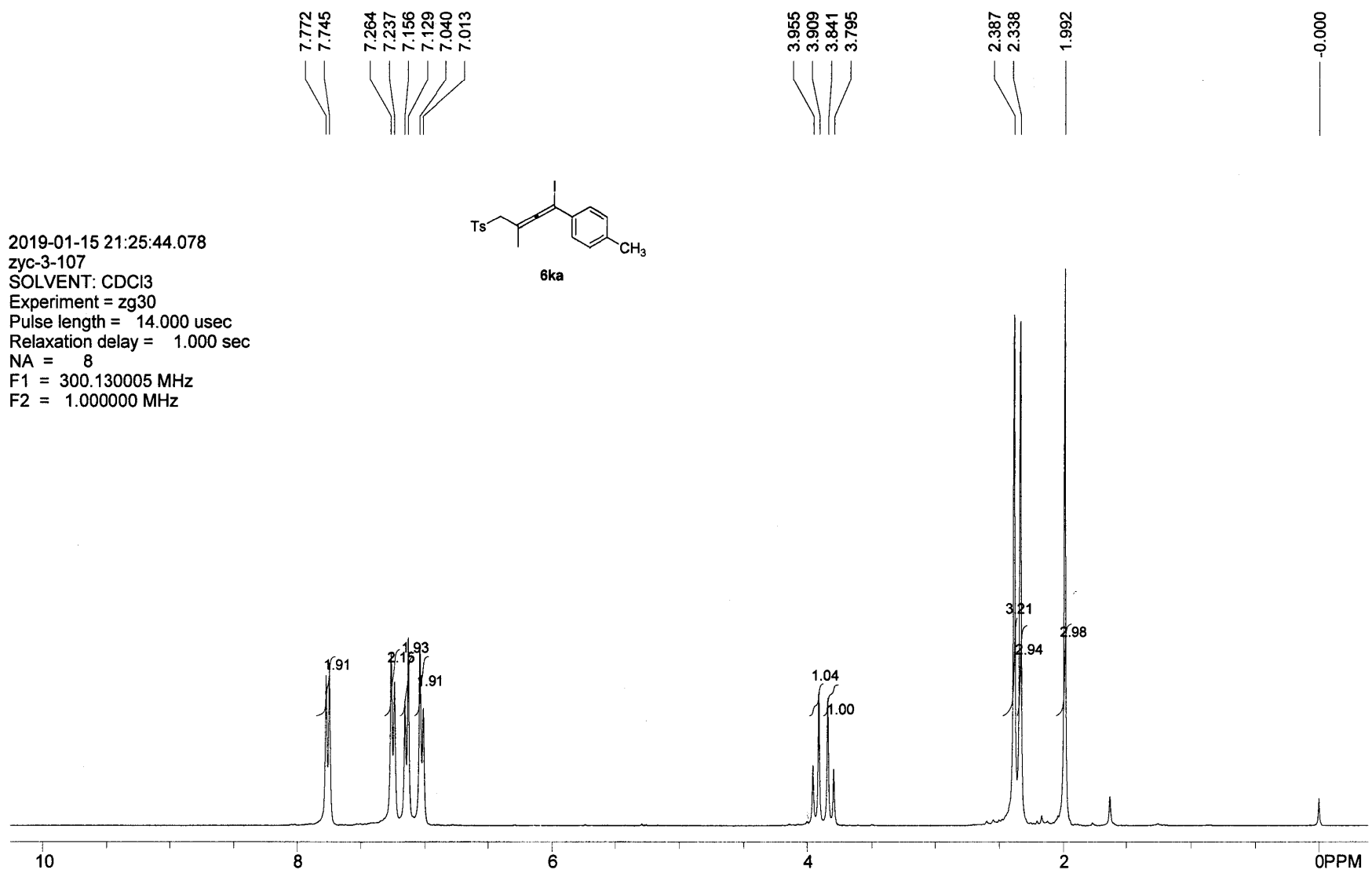

7.785
7.758
7.360
7.334
7.260
4.149
3.819
2.430
2.122
2.100
2.076
1.317
1.297
1.274
1.259
1.241
1.217
1.194
0.871
0.047
Ts
TBSO
nBu
6ia
syl-3-177_1.1
2019-01-22 11:04:04.734
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
2.00
2.03
1.95
1.97
3.10
1.98
4.21
12.61
6.40
10
8
6
4
2
0
PPM

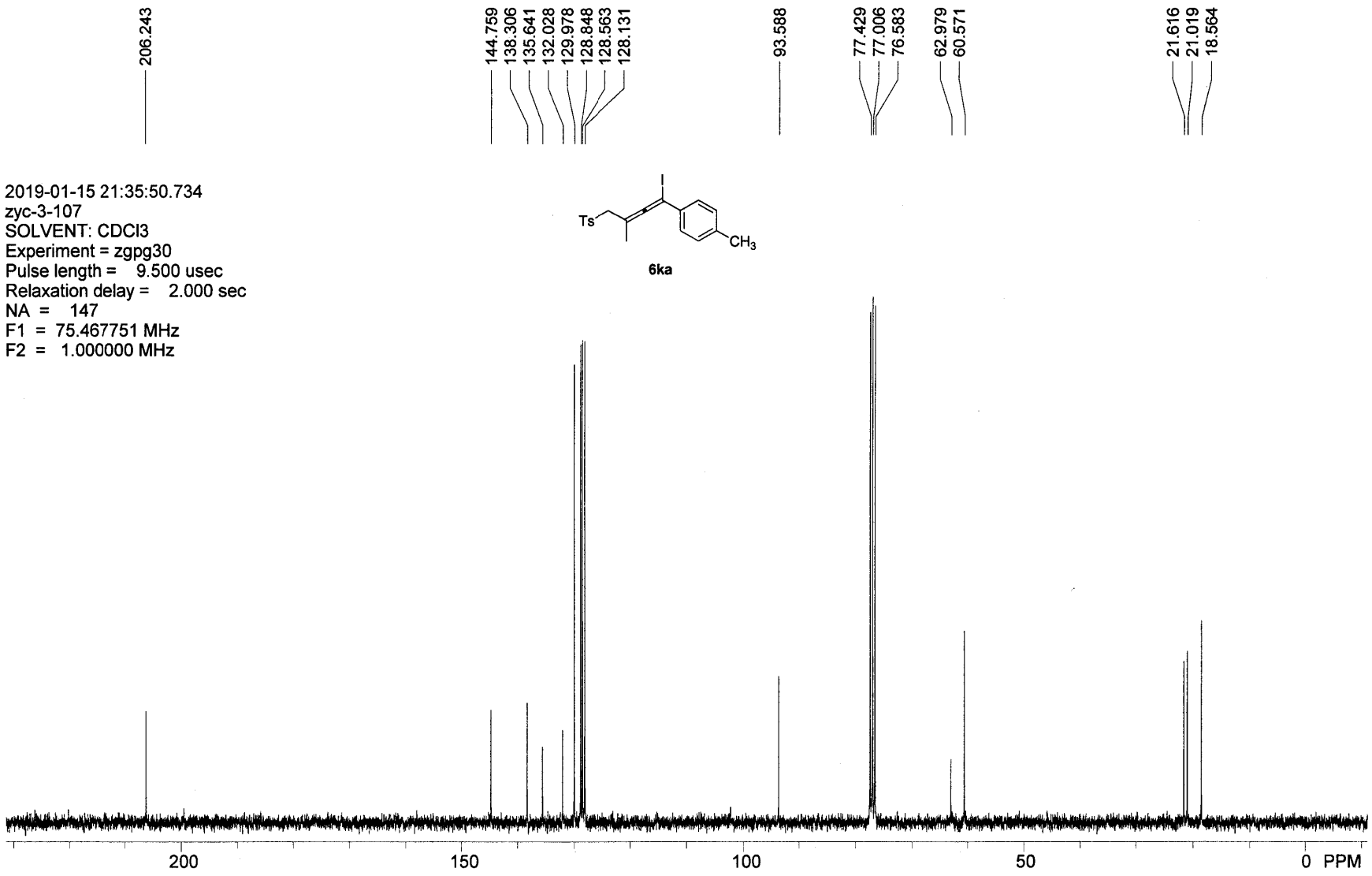

202.303
144.771
135.956
129.954
128.300
96.174
77.432
77.000
76.577
65.814
61.861
55.794
39.883
31.096
25.765
21.601
21.390
18.163
13.696
-5.405
I
Ts
nBu
TBSO
6ia
syl-3-177_2.1
2019-01-22 11:10:35.578
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 89
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
200
150
100
50
0
PPM

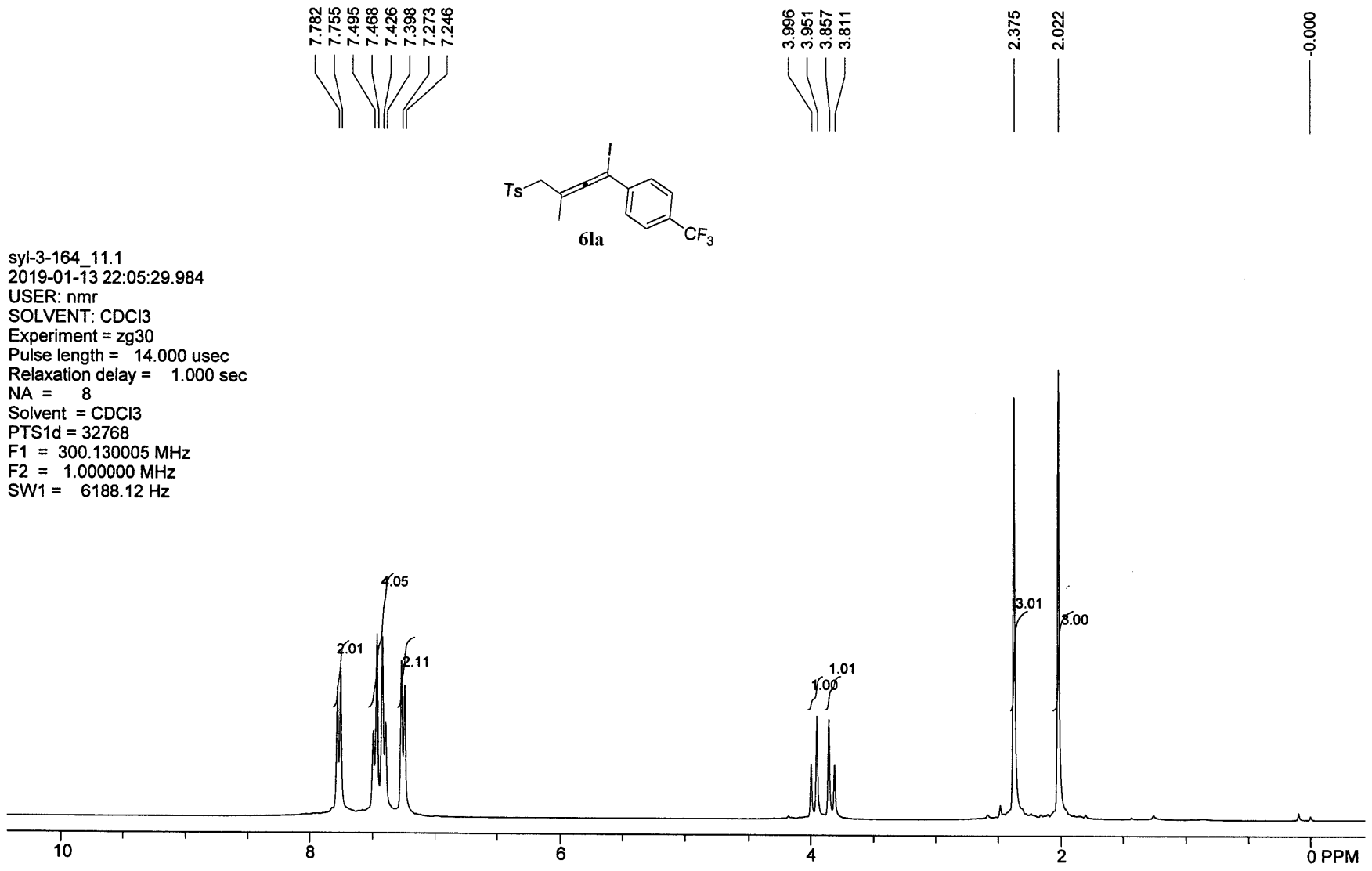

7.770
7.743
7.286
7.280
7.271
7.258
7.253
7.243
7.229
7.217
7.205
3.963
3.917
3.847
3.801
2.375
1.998
-0.000
Ts
Ph
6ja

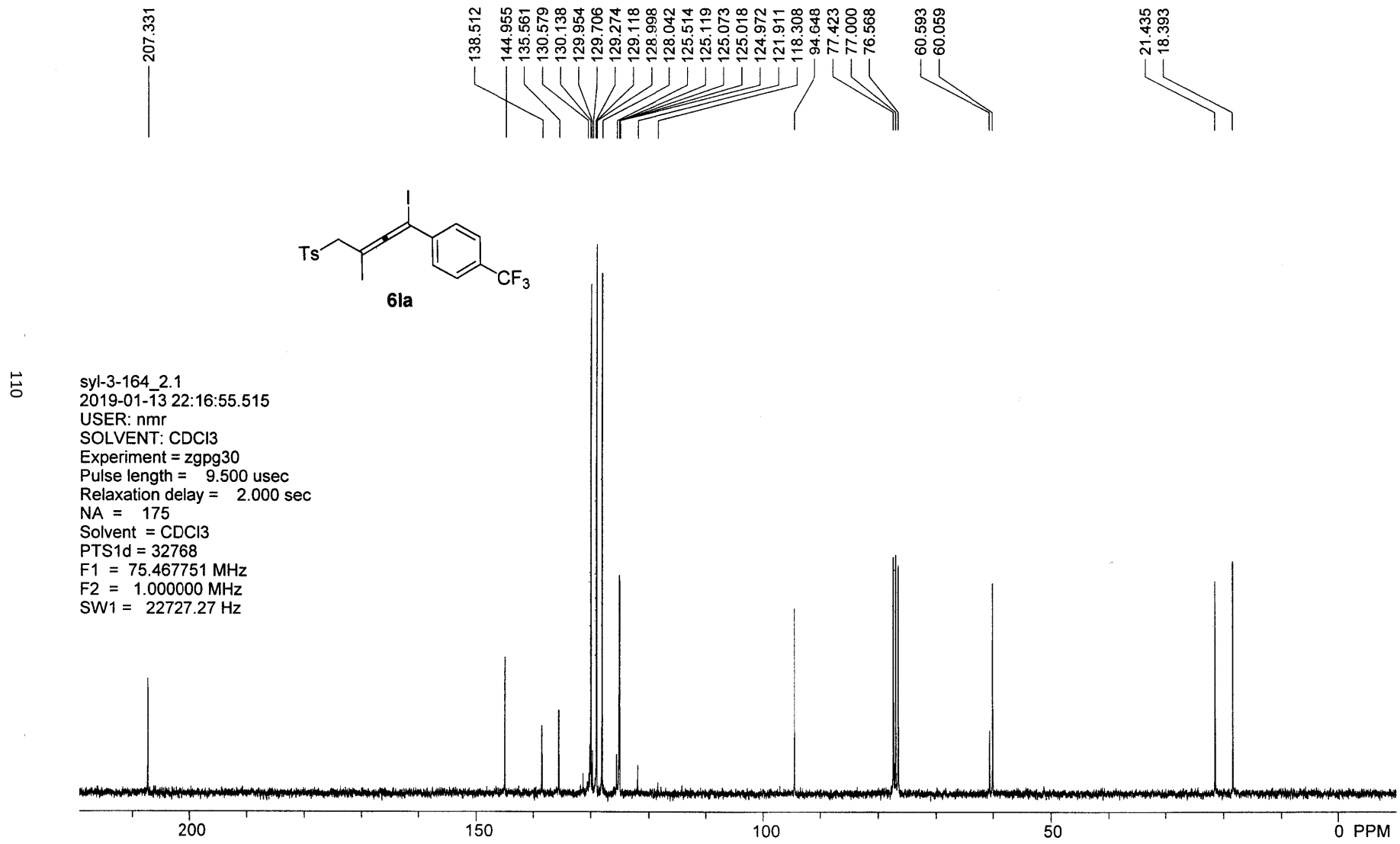

syl-3-155_1.1
2019-01-09 22:58:43.437
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
1.98
7.15
1.00
0.97
2.97
2.95
10
8
6
4
2
0 PPM

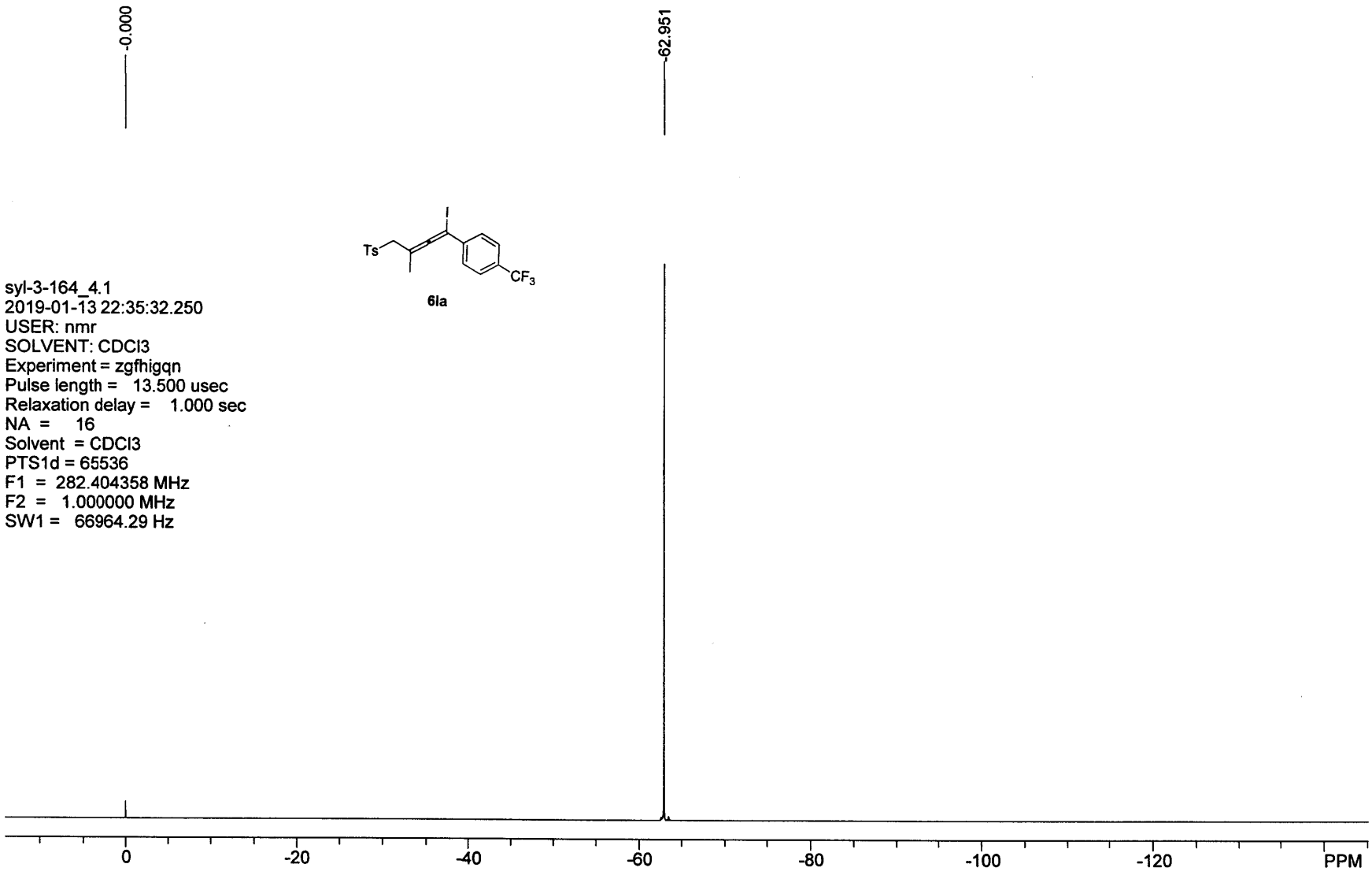

206.439
144.790
135.506
134.771
129.954
128.658
128.244
128.143
128.070
93.775
77.423
77.000
76.577
62.808
60.381
21.610
18.503
Ts
Ph
6ja
syl-3-155_2.1
2019-01-09 23:02:38.765
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 42
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
200
150
100
50
0
PPM

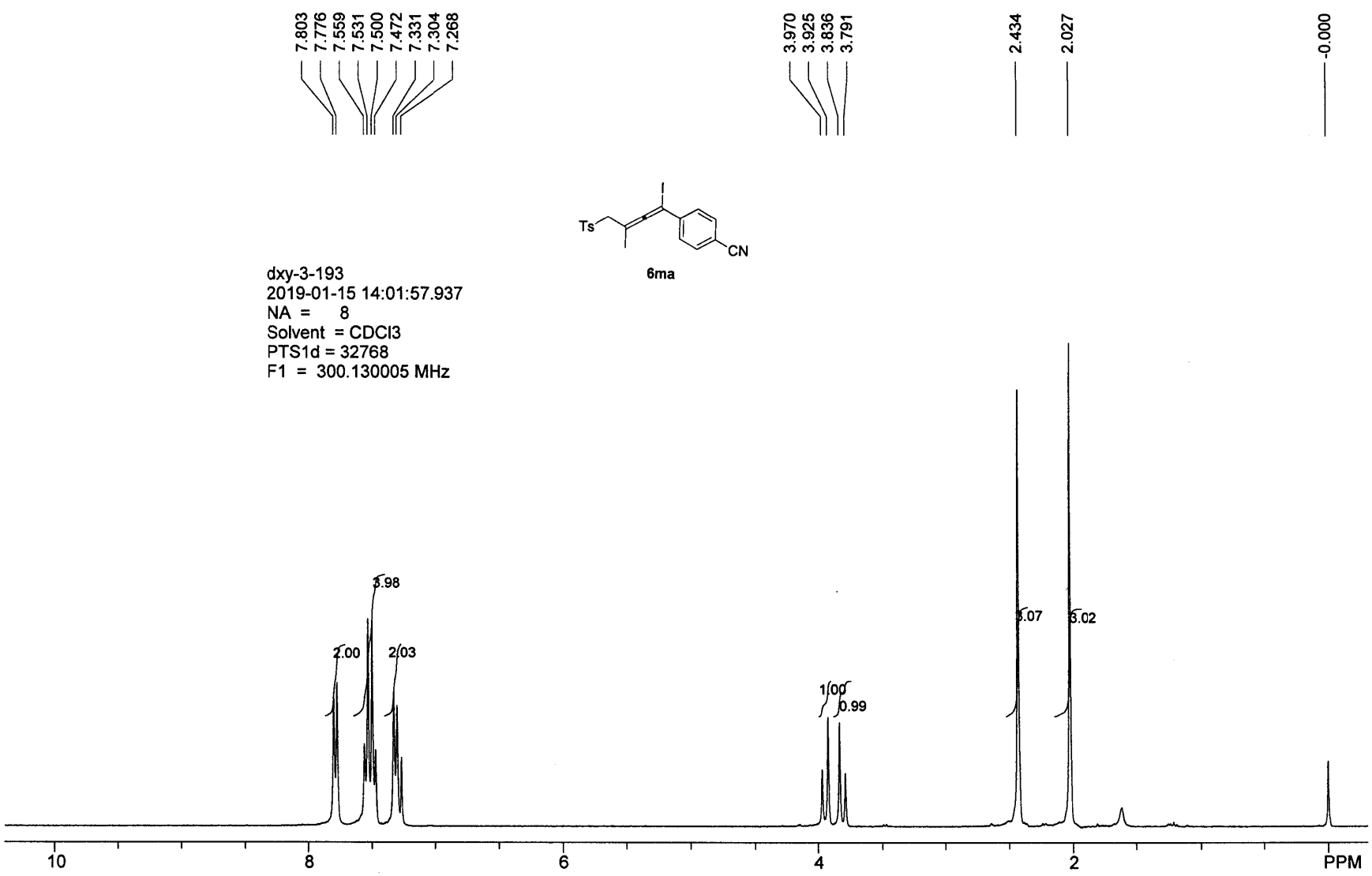

7.772
7.745
7.264
7.237
7.156
7.129
7.040
7.013
3.955
3.909
3.841
3.795
2.387
2.338
1.992
-0.000
Ts
CH3
6ka
2019-01-15 21:25:44.078
zyc-3-107
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
F1 = 300.130005 MHz
F2 = 1.000000 MHz
1.91
2.15
1.93
1.91
1.04
1.00
3.21
2.94
2.98
10
8
6
4
2
0PPM

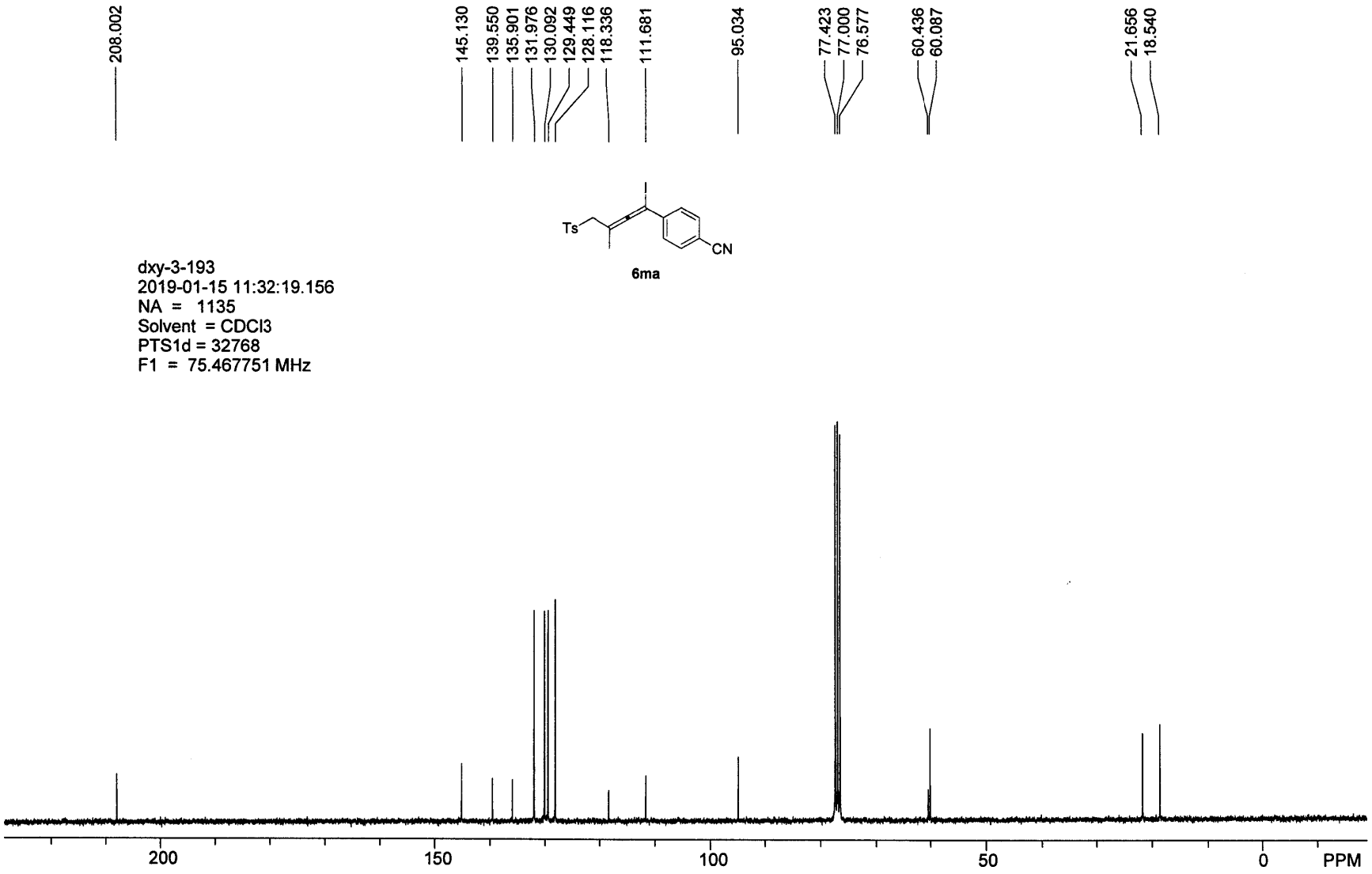

206.243
144.759
138.306
135.641
132.028
129.978
128.848
128.563
128.131
93.588
77.429
77.006
76.583
62.979
60.571
21.616
21.019
18.564
2019-01-15 21:35:50.734
zyc-3-107
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 147
F1 = 75.467751 MHz
F2 = 1.000000 MHz
Ts
CH3
6ka
200
150
100
50
0 PPM

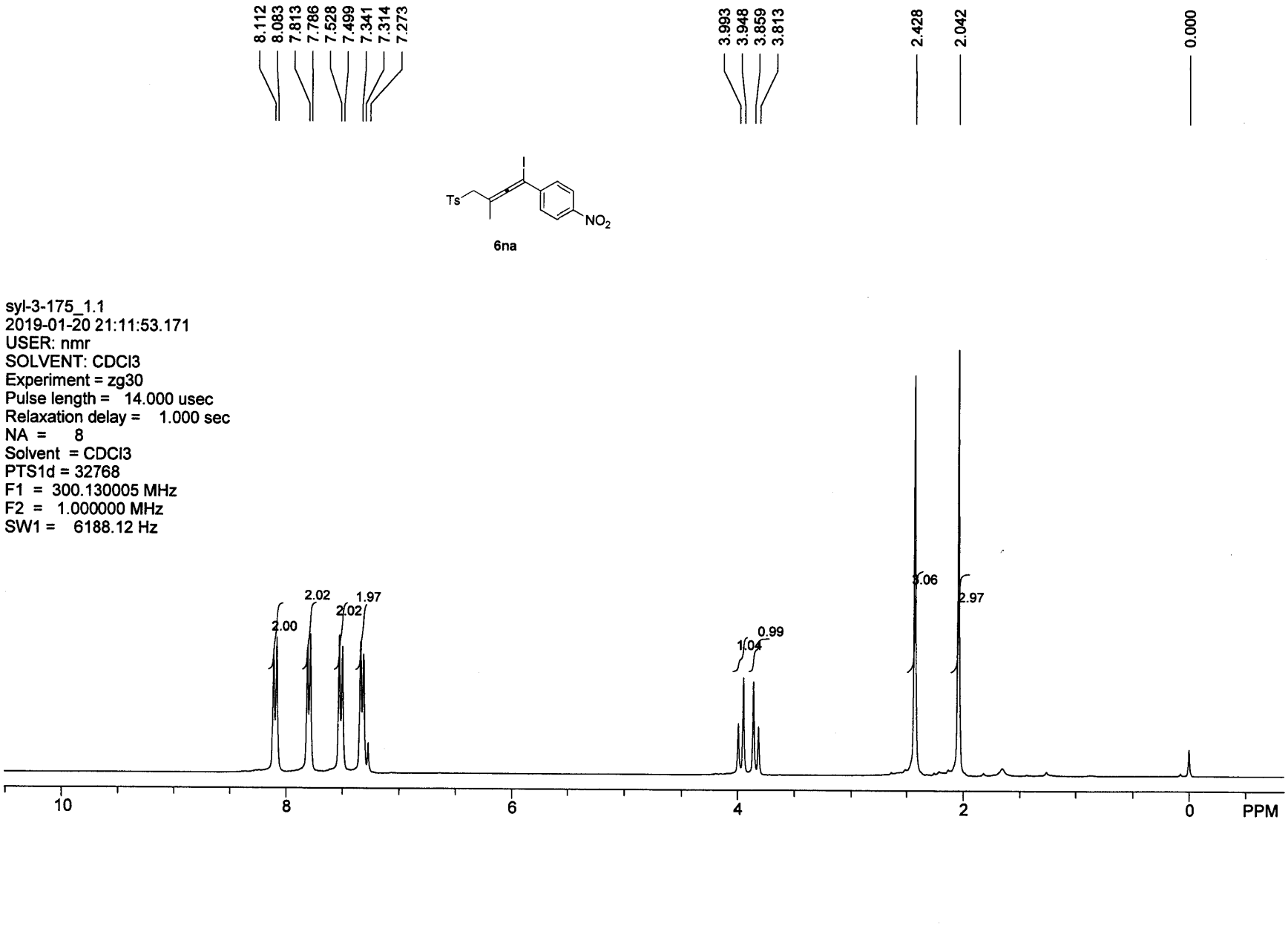

7.782
7.755
7.495
7.468
7.426
7.398
7.273
7.246
3.996
3.951
3.857
3.811
2.375
2.022
-0.000
Ts
CF3
6la
syl-3-164_11.1
2019-01-13 22:05:29.984
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
2.01
4.05
2.11
1.00
1.01
3.01
3.00
10
8
6
4
2
0 PPM


207.331
138.512
144.955
135.561
130.579
130.138
129.954
129.706
129.274
129.118
128.998
128.042
125.514
125.119
125.073
125.018
124.972
121.911
118.308
94.648
77.423
77.000
76.568
60.593
60.059
21.435
18.393
Ts
I
CF3
6la
syl-3-164_2.1
2019-01-13 22:16:55.515
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 175
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
200
150
100
50
0 PPM

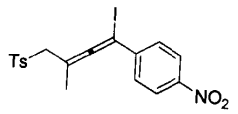

-0.000
-62.951
Ts
I
CF3
6la
syl-3-164_4.1
2019-01-13 22:35:32.250
USER: nmr
SOLVENT: CDCl3
Experiment = zgfhigqn
Pulse length = 13.500 usec
Relaxation delay = 1.000 sec
NA = 16
Solvent = CDCl3
PTS1d = 65536
F1 = 282.404358 MHz
F2 = 1.000000 MHz
SW1 = 66964.29 Hz
0
-20
-40
-60
-80
-100
-120
PPM


7.803
7.776
7.559
7.531
7.500
7.472
7.331
7.304
7.268
3.970
3.925
3.836
3.791
2.434
2.027
-0.000
Ts
CN
6ma
dxy-3-193
2019-01-15 14:01:57.937
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
3.98
2.00
2.03
1.00
0.99
3.07
3.02
10
8
6
4
2
PPM

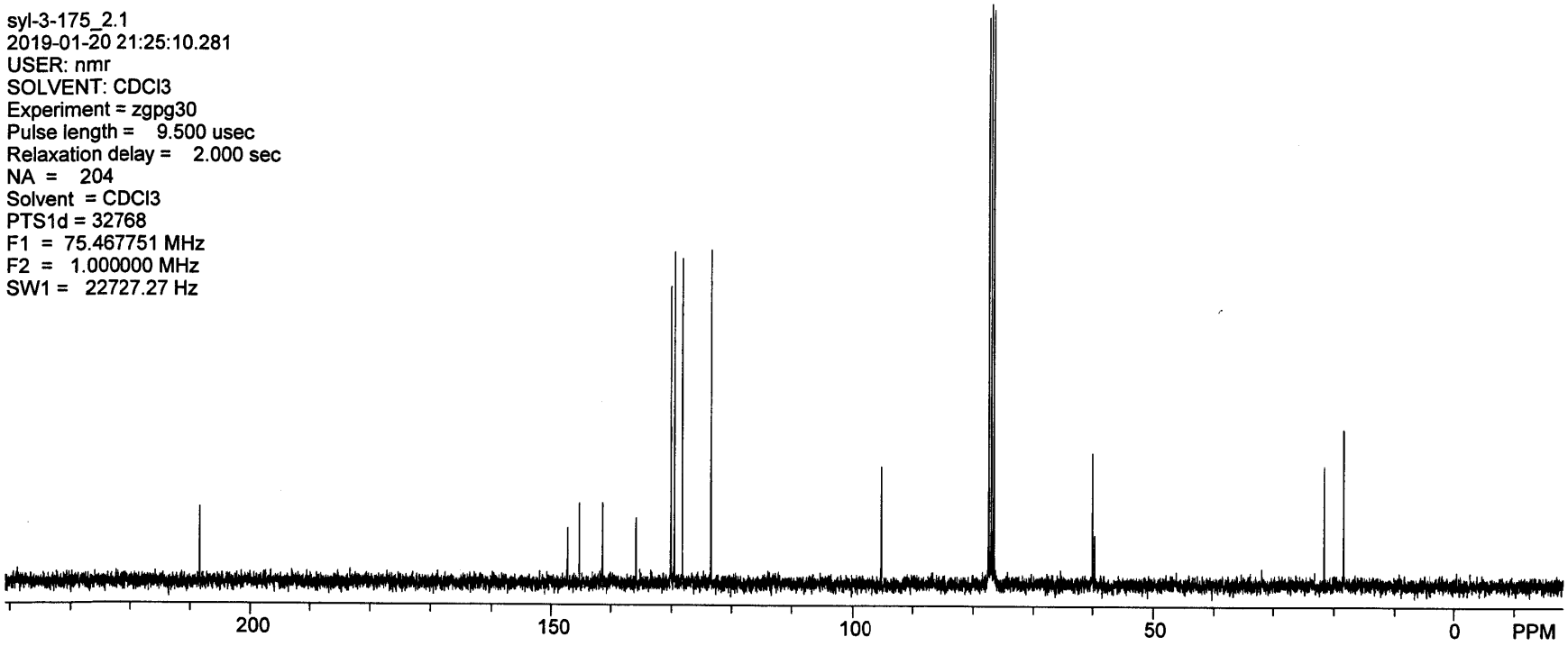

208.002
145.130
139.550
135.901
131.976
130.092
129.449
128.116
118.336
111.681
95.034
77.423
77.000
76.577
60.436
60.087
21.656
18.540
Ts
CN
6ma
dxy-3-193
2019-01-15 11:32:19.156
NA = 1135
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
200
150
100
50
0
PPM

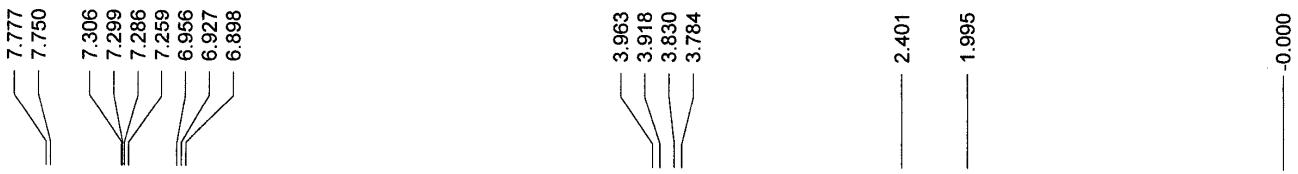

8.112
8.083
7.813
7.786
7.528
7.499
7.341
7.314
7.273
3.993
3.948
3.859
3.813
2.428
2.042
0.000
Ts
I
NO2
6na
syl-3-175_1.1
2019-01-20 21:11:53.171
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
2.00
2.02
2.02
1.97
1.04
0.99
3.06
2.97
10
8
6
4
2
0
PPM

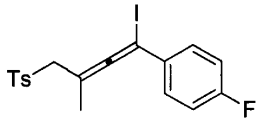
208.397
147.198
145.222
141.379
135.828
130.129
129.550
128.143
123.382
95.135
77.423
77.000
76.577
60.032
59.692
21.628
18.457



6na

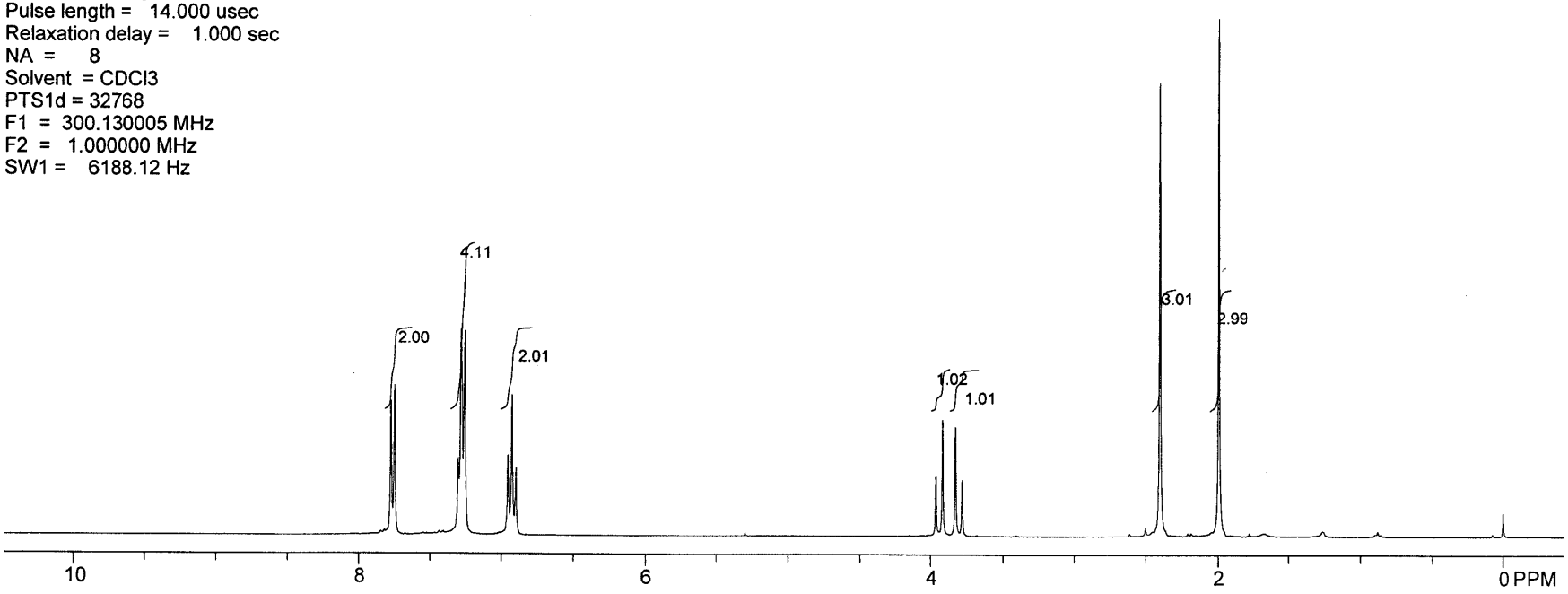


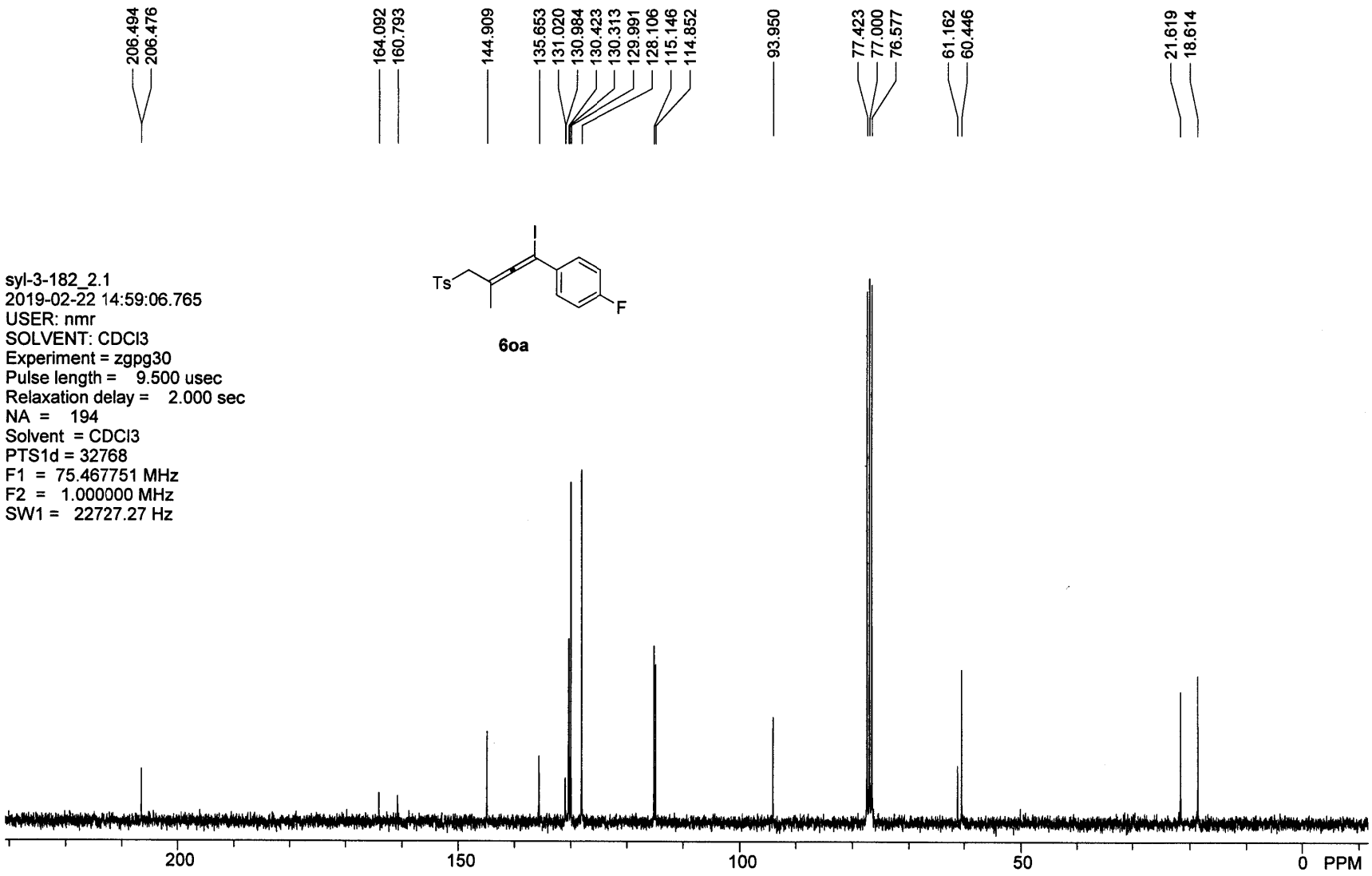


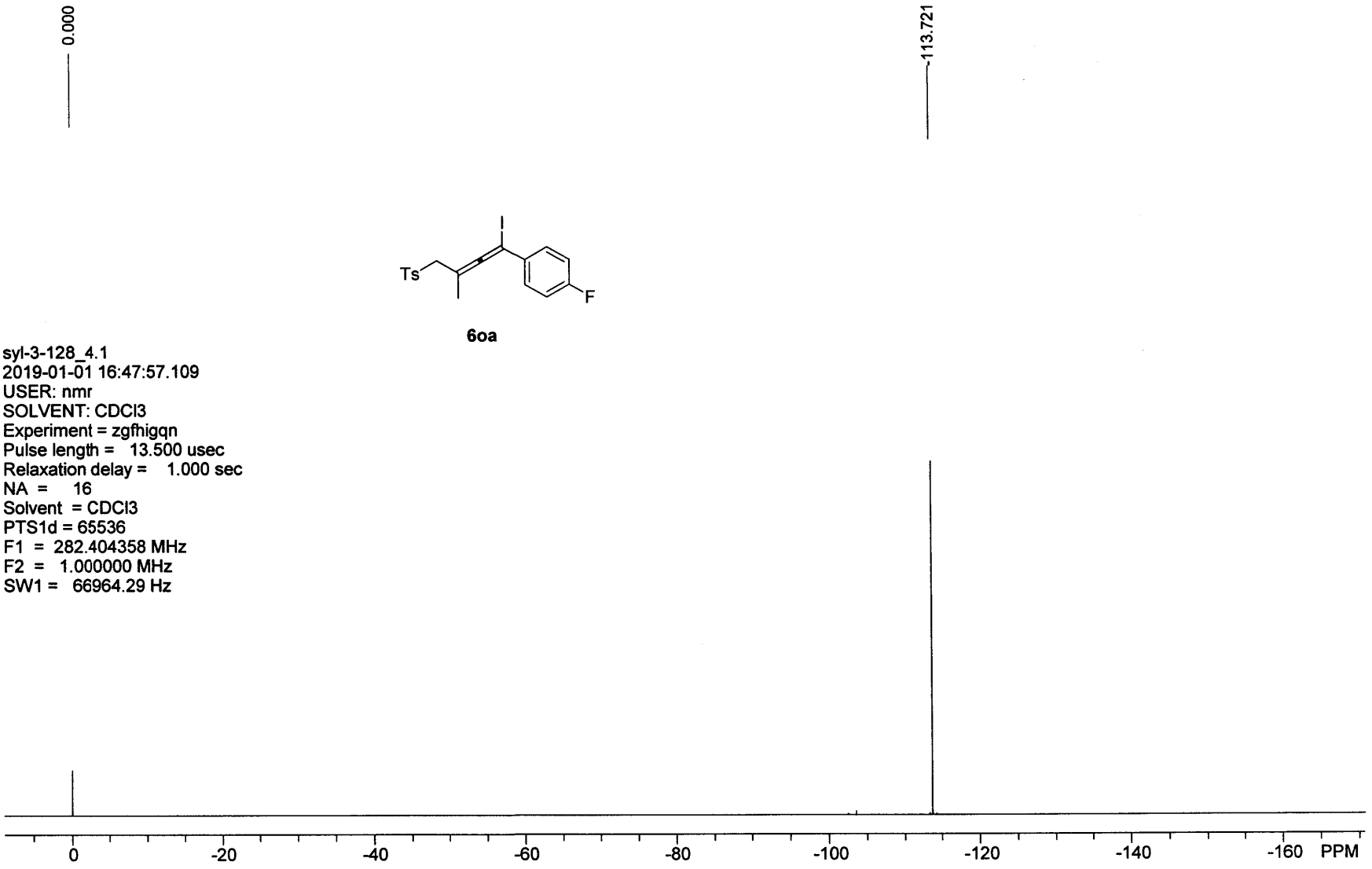


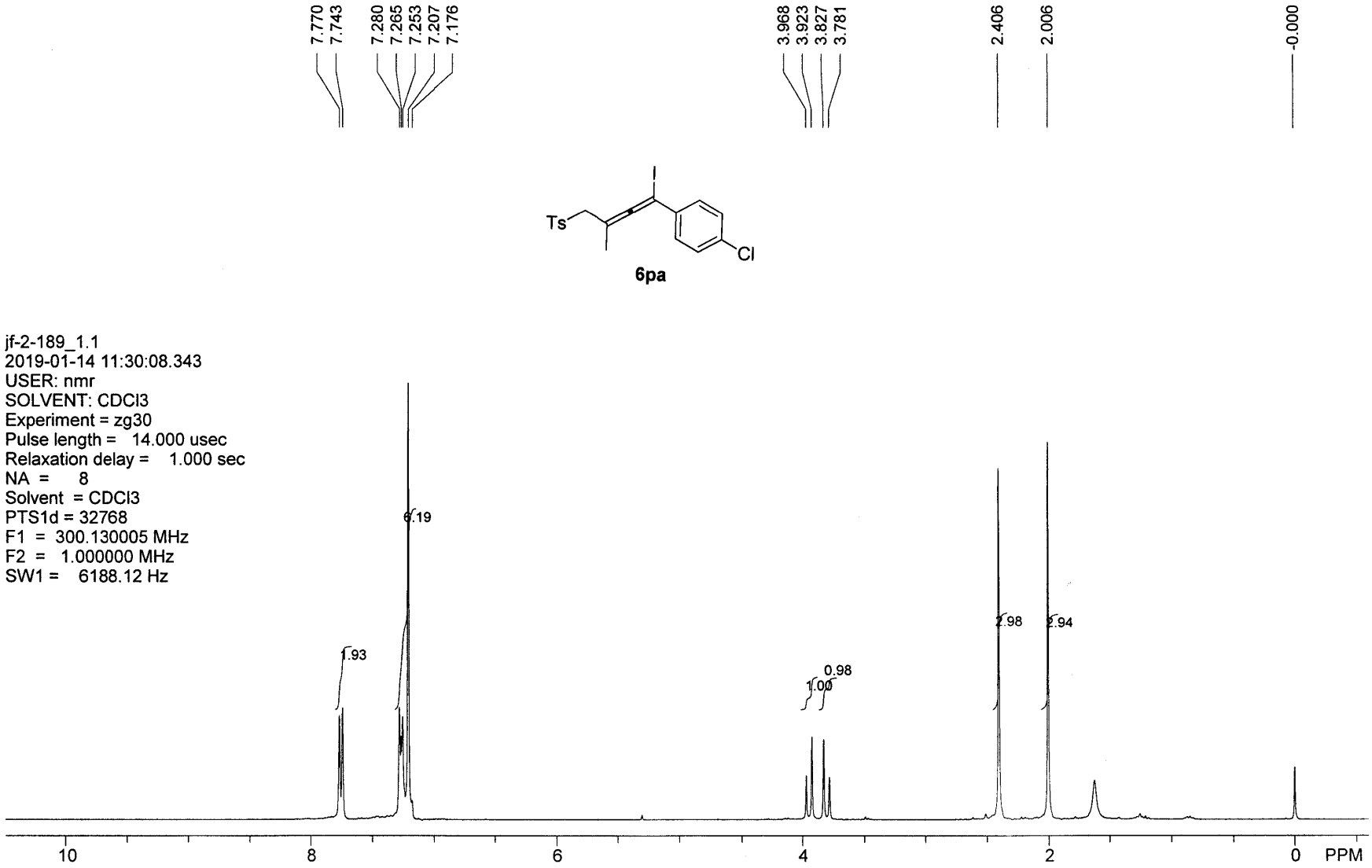
6oa

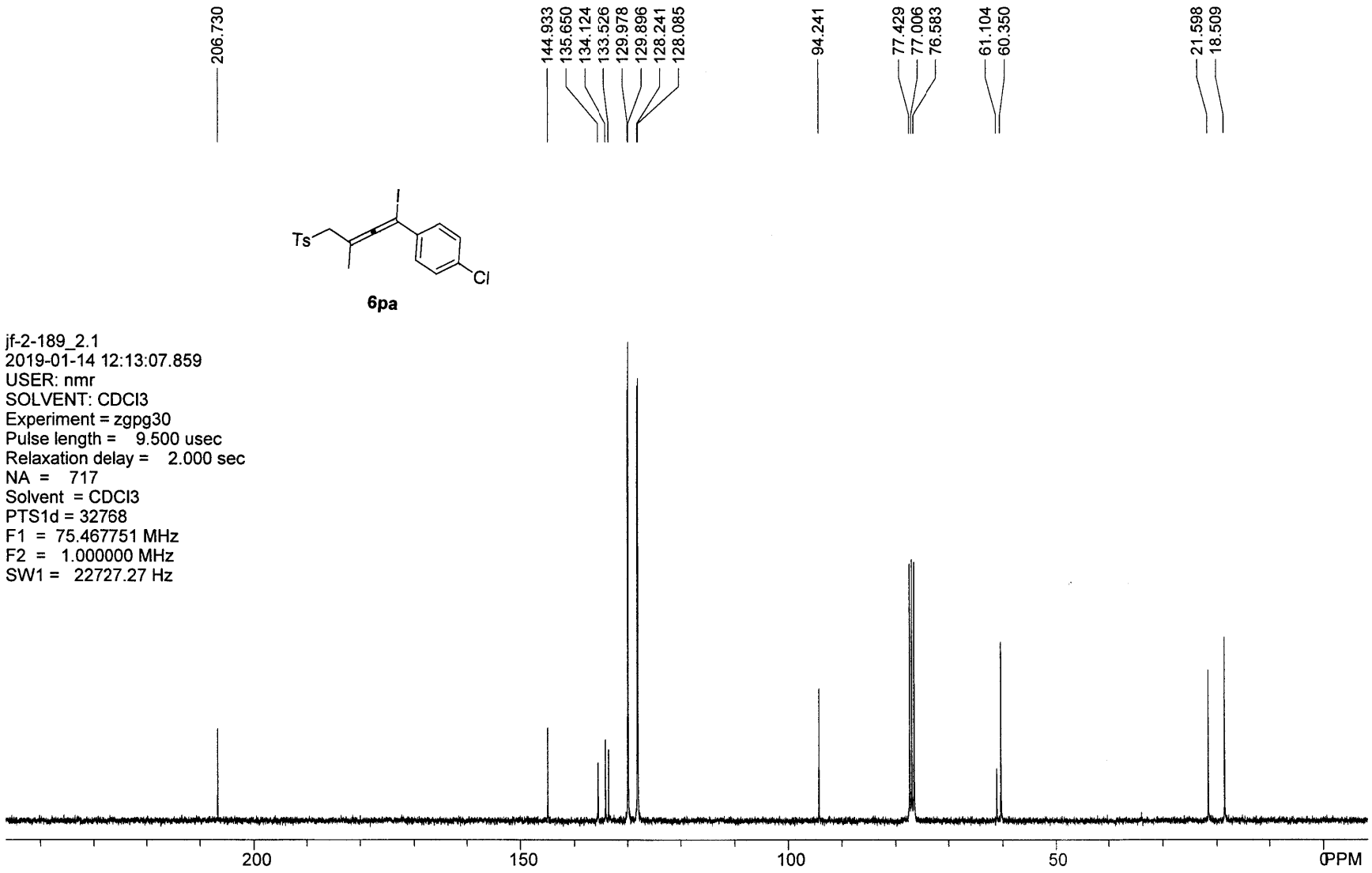
syl-3-182_1.1
2019-02-22 14:46:05.375
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz

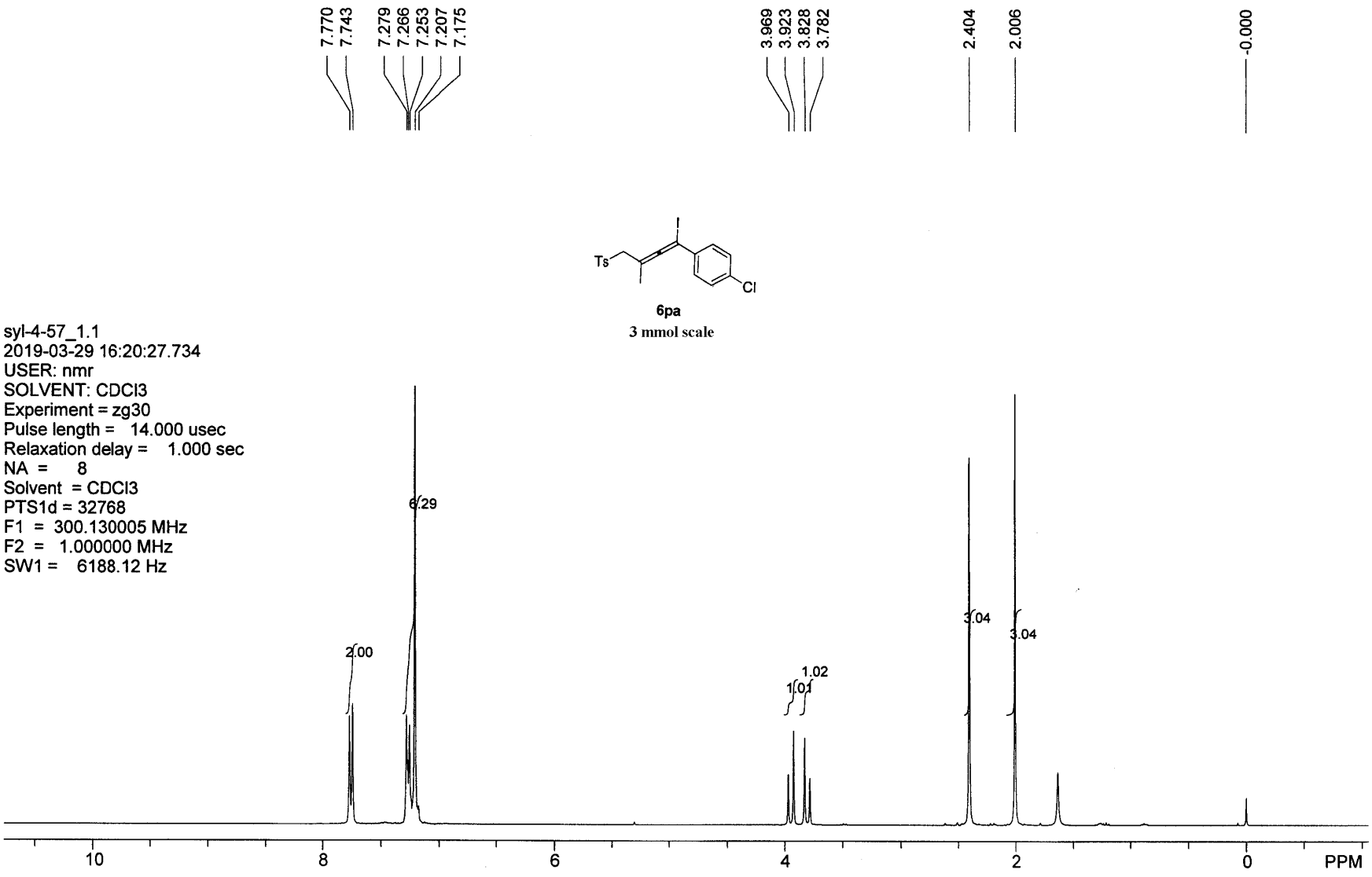


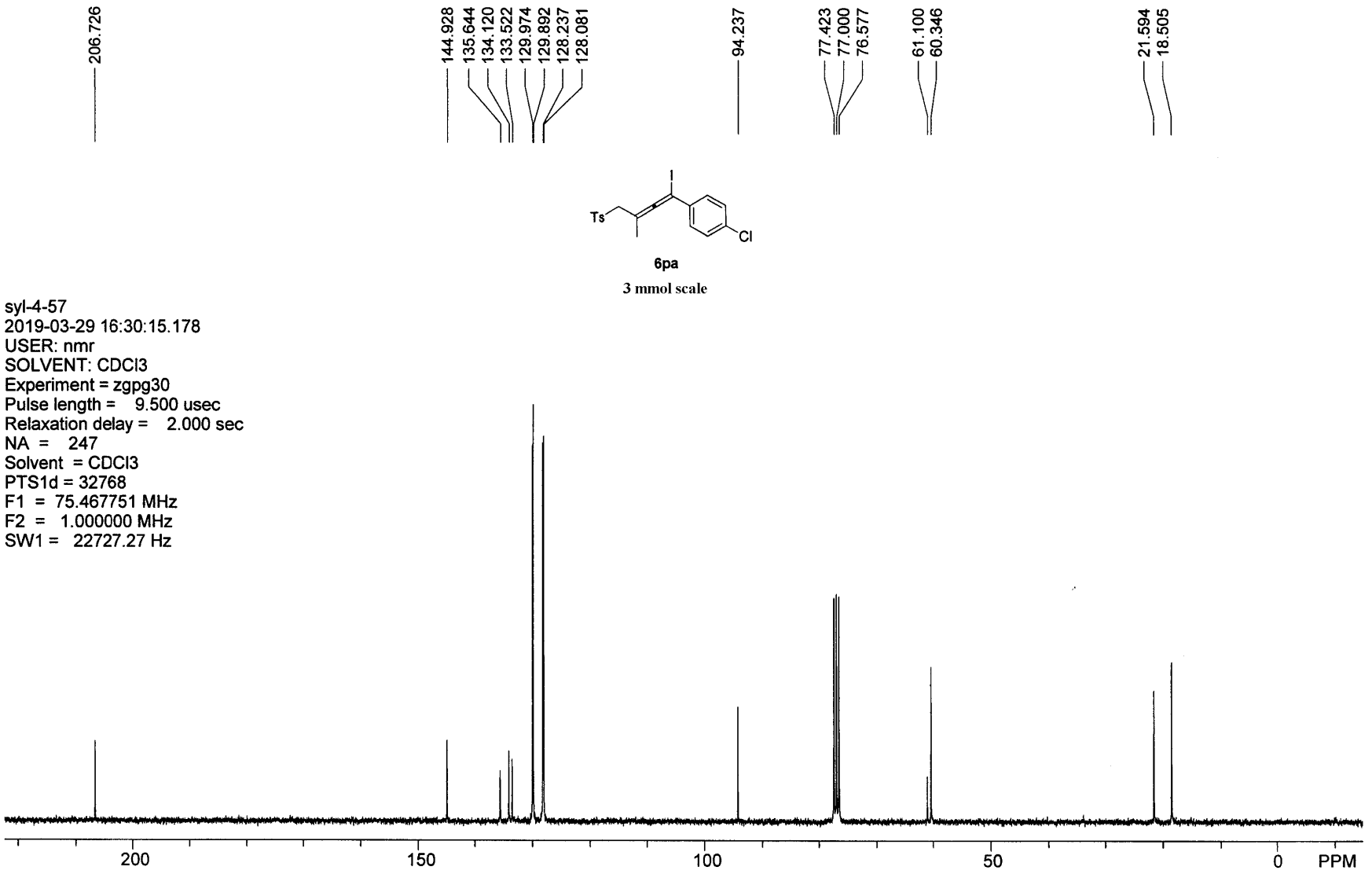

206.494
206.476
164.092
160.793
144.909
135.653
131.020
130.984
130.423
130.313
129.991
128.106
115.146
114.852
93.950
77.423
77.000
76.577
61.162
60.446
21.619
18.614
I
Ts
F
6oa
syl-3-182_2.1
2019-02-22 14:59:06.765
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 194
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
200
150
100
50
0 PPM

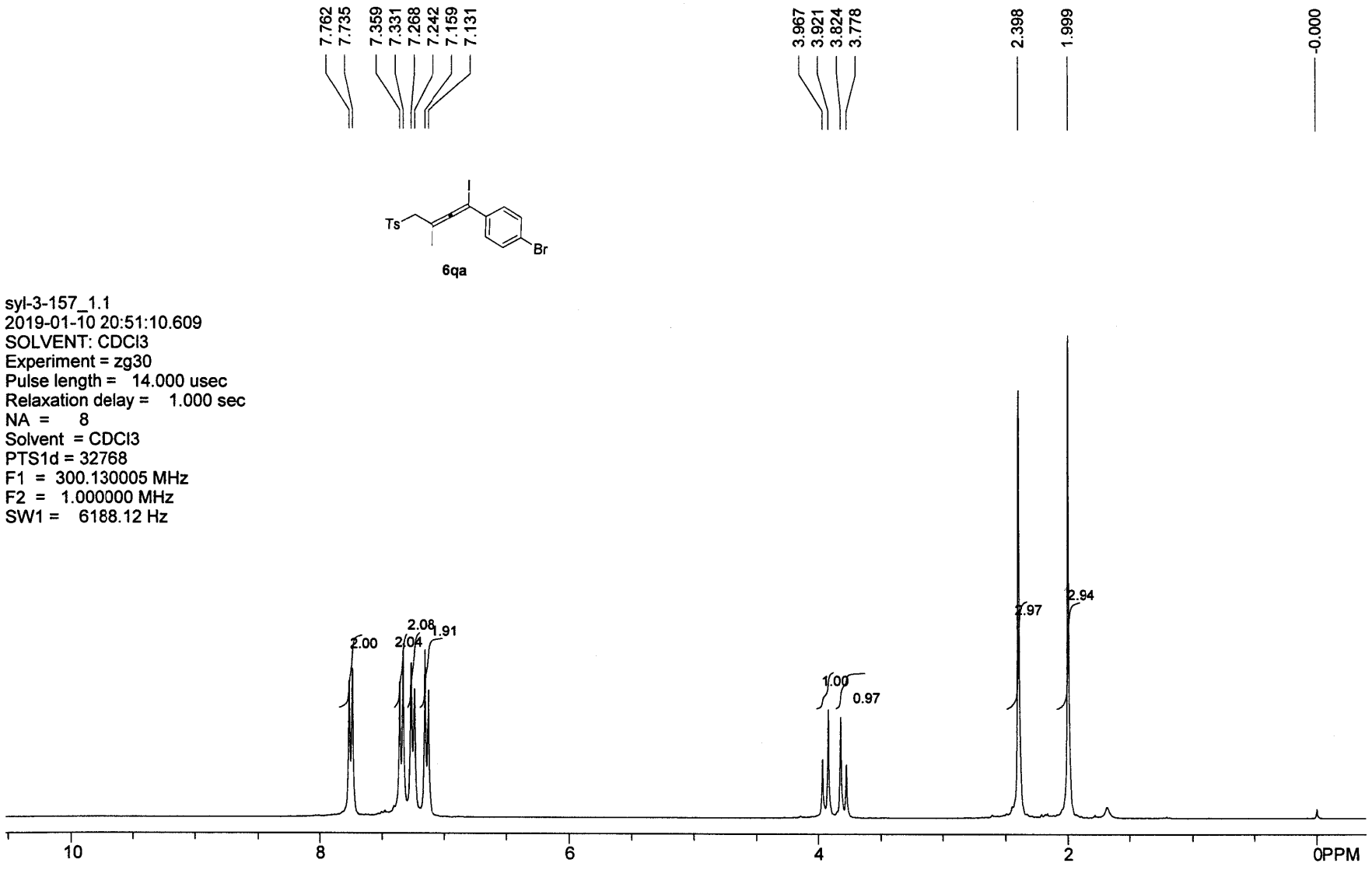

0.000
113.721
I
Ts
F
6oa
syl-3-128_4.1
2019-01-01 16:47:57.109
USER: nmr
SOLVENT: CDCl3
Experiment = zgfhigqn
Pulse length = 13.500 usec
Relaxation delay = 1.000 sec
NA = 16
Solvent = CDCl3
PTS1d = 65536
F1 = 282.404358 MHz
F2 = 1.000000 MHz
SW1 = 66964.29 Hz
0
-20
-40
-60
-80
-100
-120
-140
-160
PPM

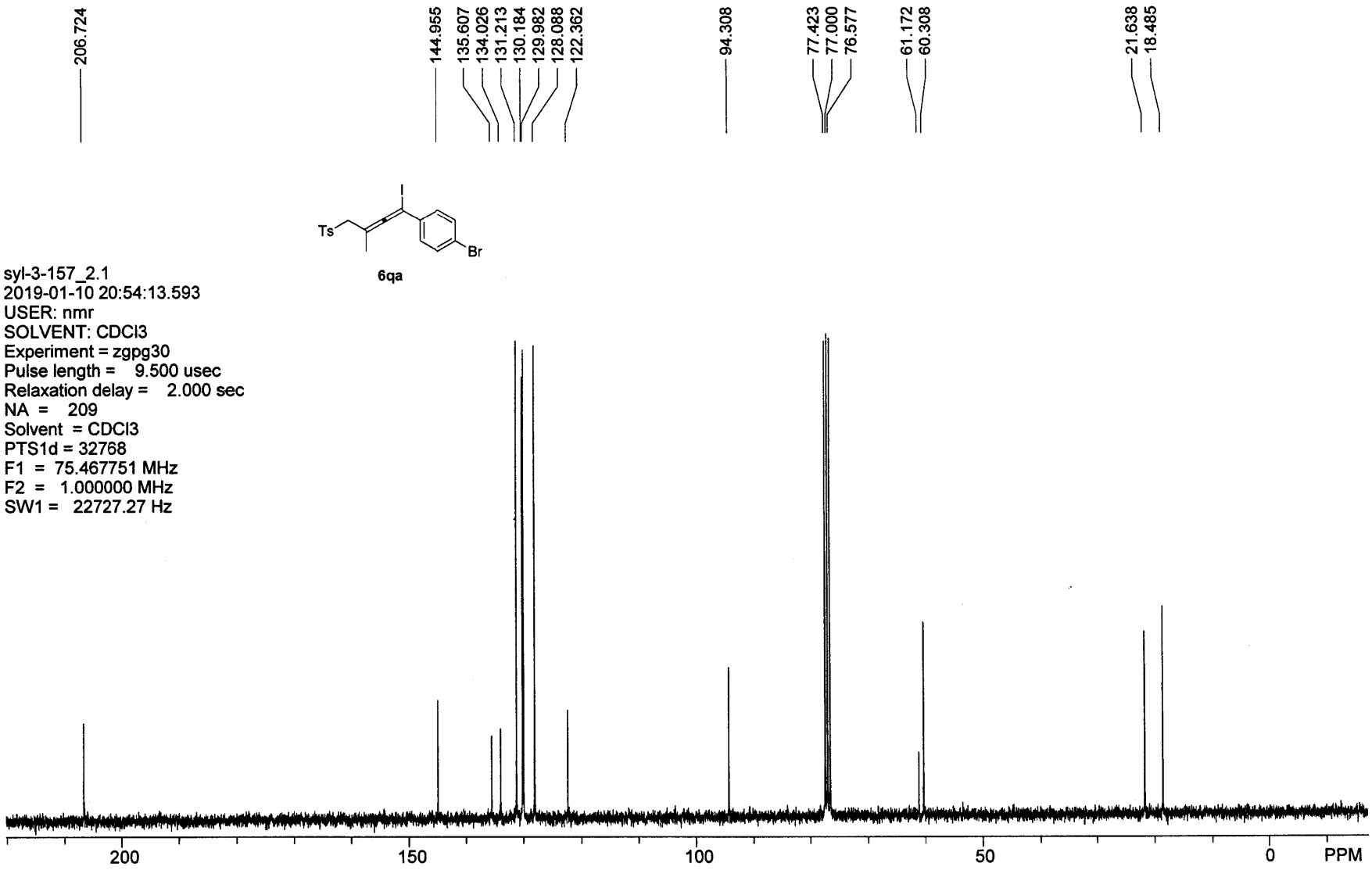

7.770
7.743
7.280
7.265
7.253
7.207
7.176
3.968
3.923
3.827
3.781
2.406
2.006
-0.000
Ts
Cl
6pa
jf-2-189_1.1
2019-01-14 11:30:08.343
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
6.19
1.93
0.98
1.00
2.98
2.94
10
8
6
4
2
0
PPM

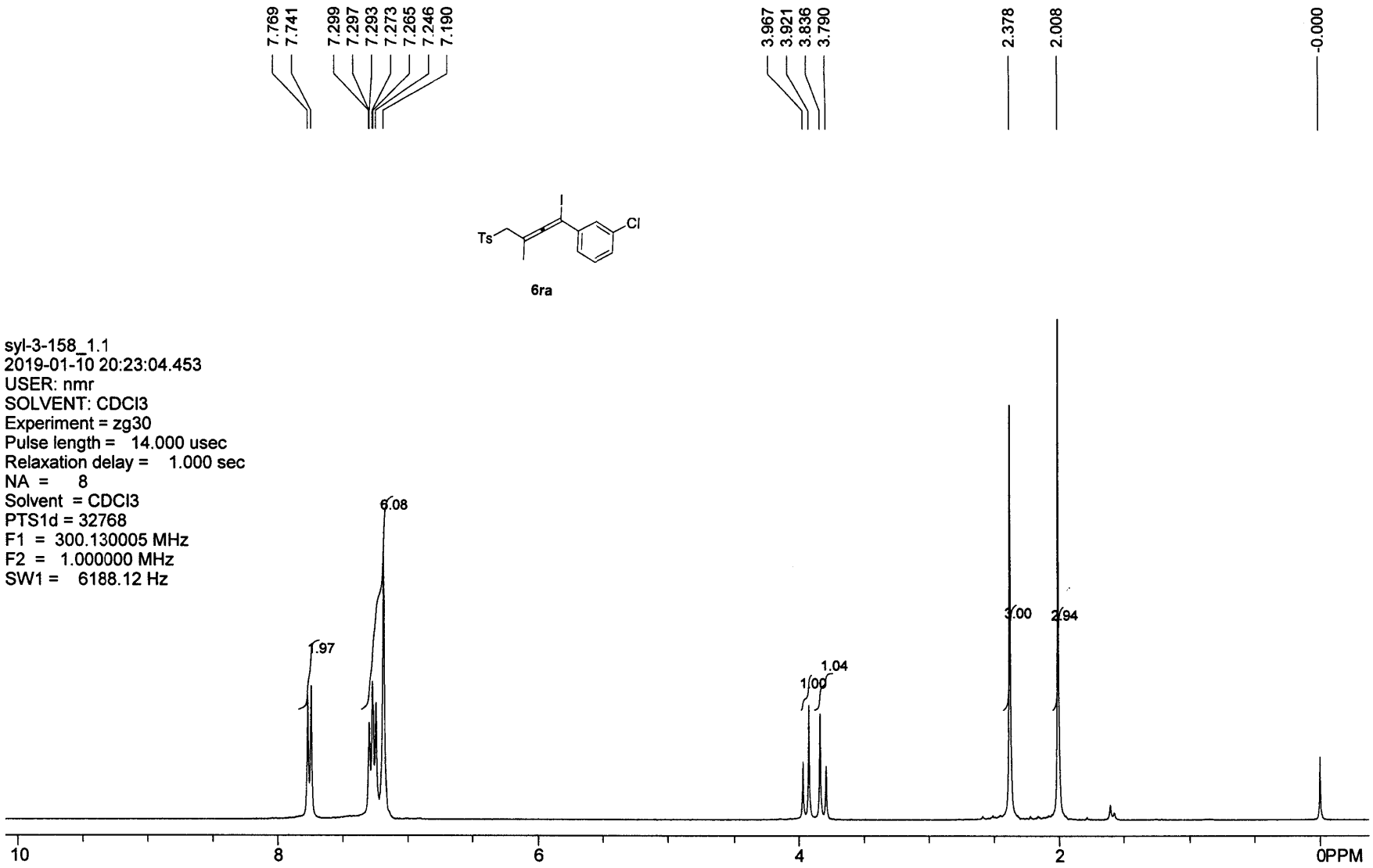

206.730
144.933
135.650
134.124
133.526
129.978
129.896
128.241
128.085
94.241
77.429
77.006
76.583
61.104
60.350
21.598
18.509
Ts
Cl
6pa
jf-2-189_2.1
2019-01-14 12:13:07.859
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 717
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
200
150
100
50
0
PPM

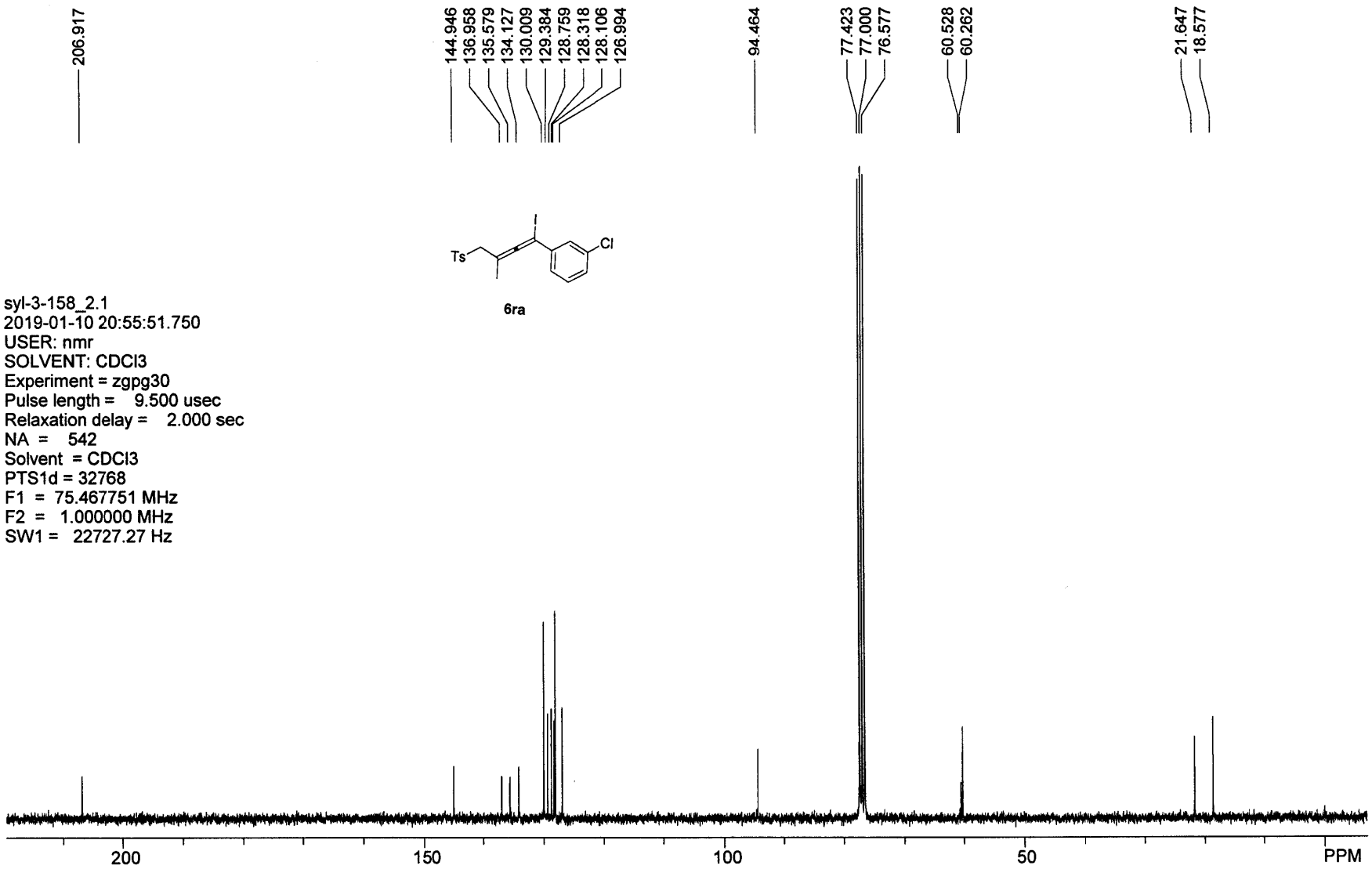

7.770
7.743
7.279
7.266
7.253
7.207
7.175
3.969
3.923
3.828
3.782
2.404
2.006
-0.000
Ts
Cl
6pa
3 mmol scale
syl-4-57_1.1
2019-03-29 16:20:27.734
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
6.29
2.00
1.02
1.01
3.04
3.04
10
8
6
4
2
0
PPM

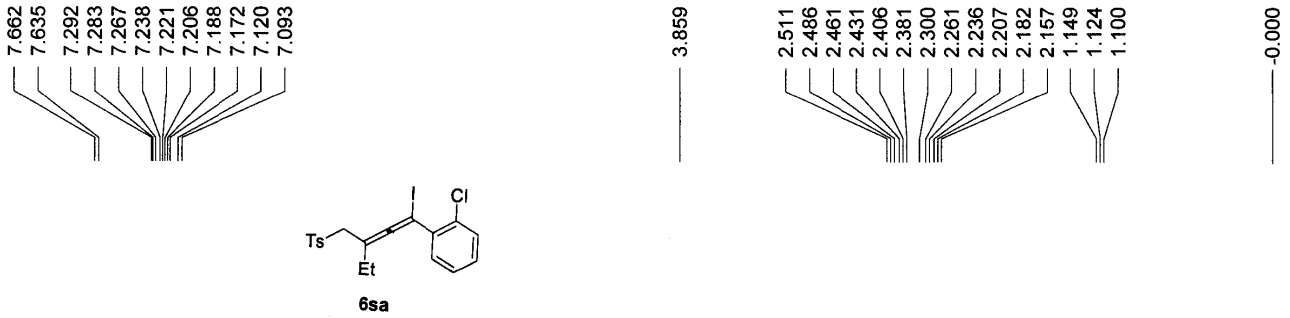

206.726
144.928
135.644
134.120
133.522
129.974
129.892
128.237
128.081
94.237
77.423
77.000
76.577
61.100
60.346
21.594
18.505
Ts
I
Cl
6pa
3 mmol scale
syl-4-57
2019-03-29 16:30:15.178
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 247
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
200
150
100
50
0
PPM

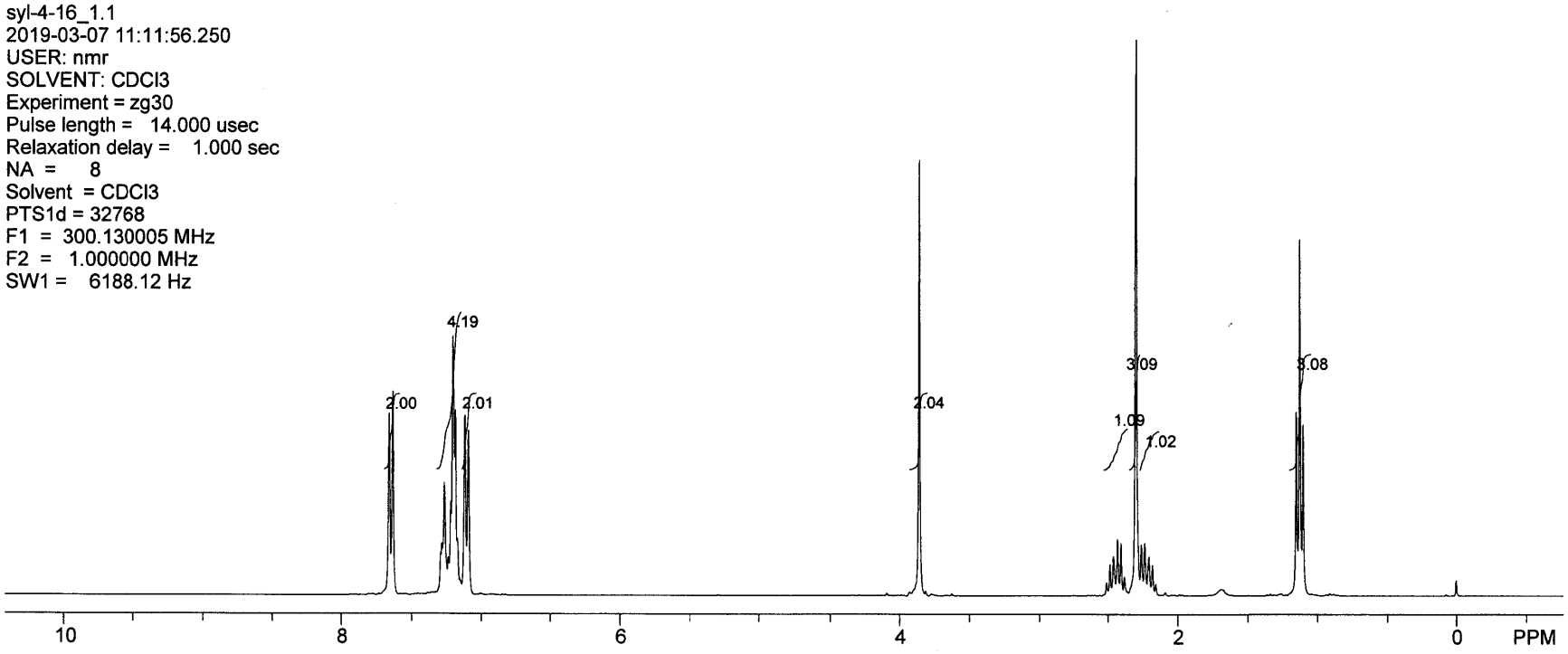

7.762
7.735
7.359
7.331
7.268
7.242
7.159
7.131
3.967
3.921
3.824
3.778
2.398
1.999
-0.000
I
Ts
Br
6qa
syl-3-157_1.1
2019-01-10 20:51:10.609
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
2.00
2.04
2.08
1.91
1.00
0.97
2.97
2.94
10
8
6
4
2
0PPM

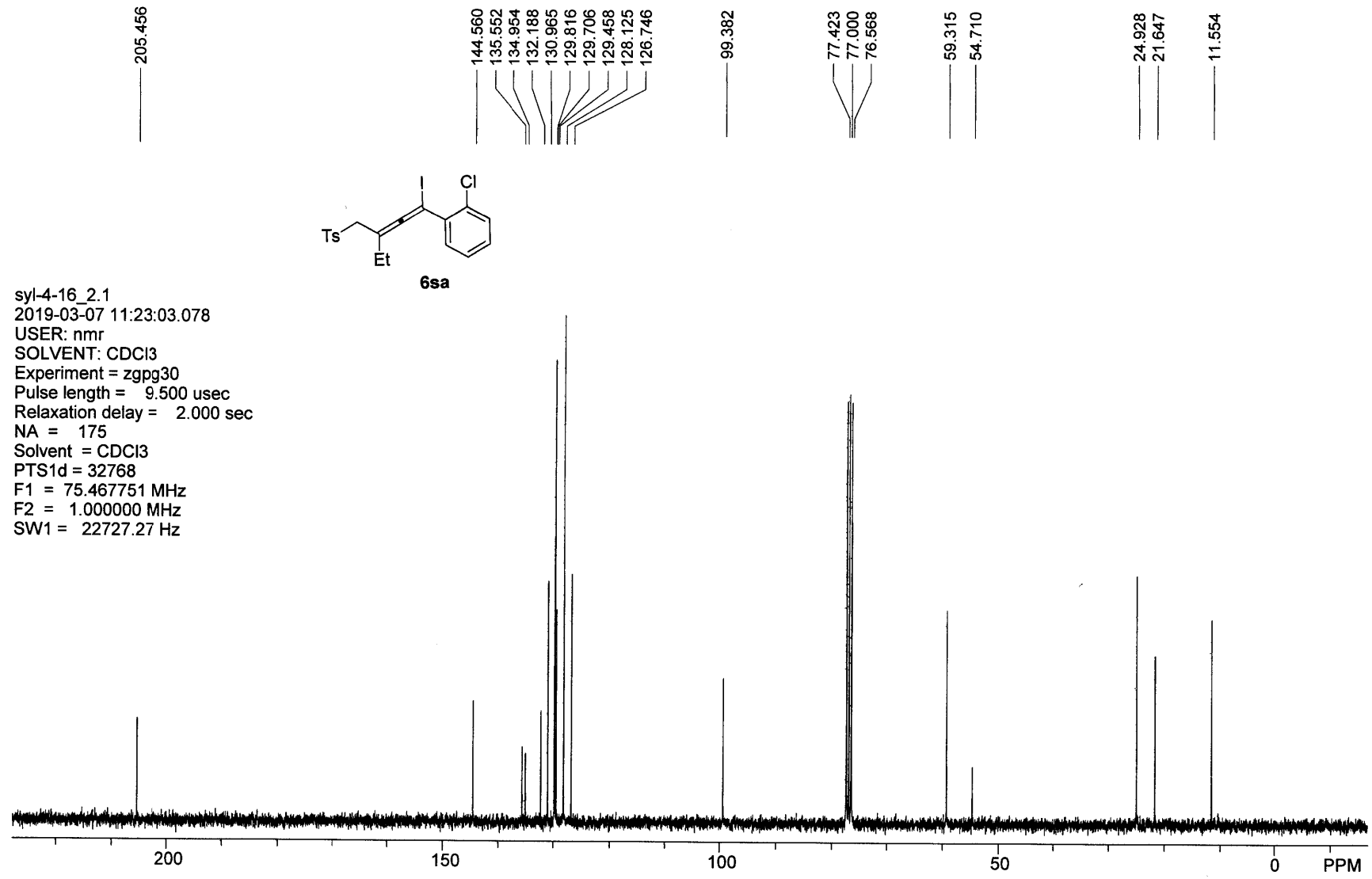

206.724
144.955
135.607
134.026
131.213
130.184
129.982
128.088
122.362
94.308
77.423
77.000
76.577
61.172
60.308
21.638
18.485
I
Ts
Br
6qa
syl-3-157_2.1
2019-01-10 20:54:13.593
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 209
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
200
150
100
50
0
PPM

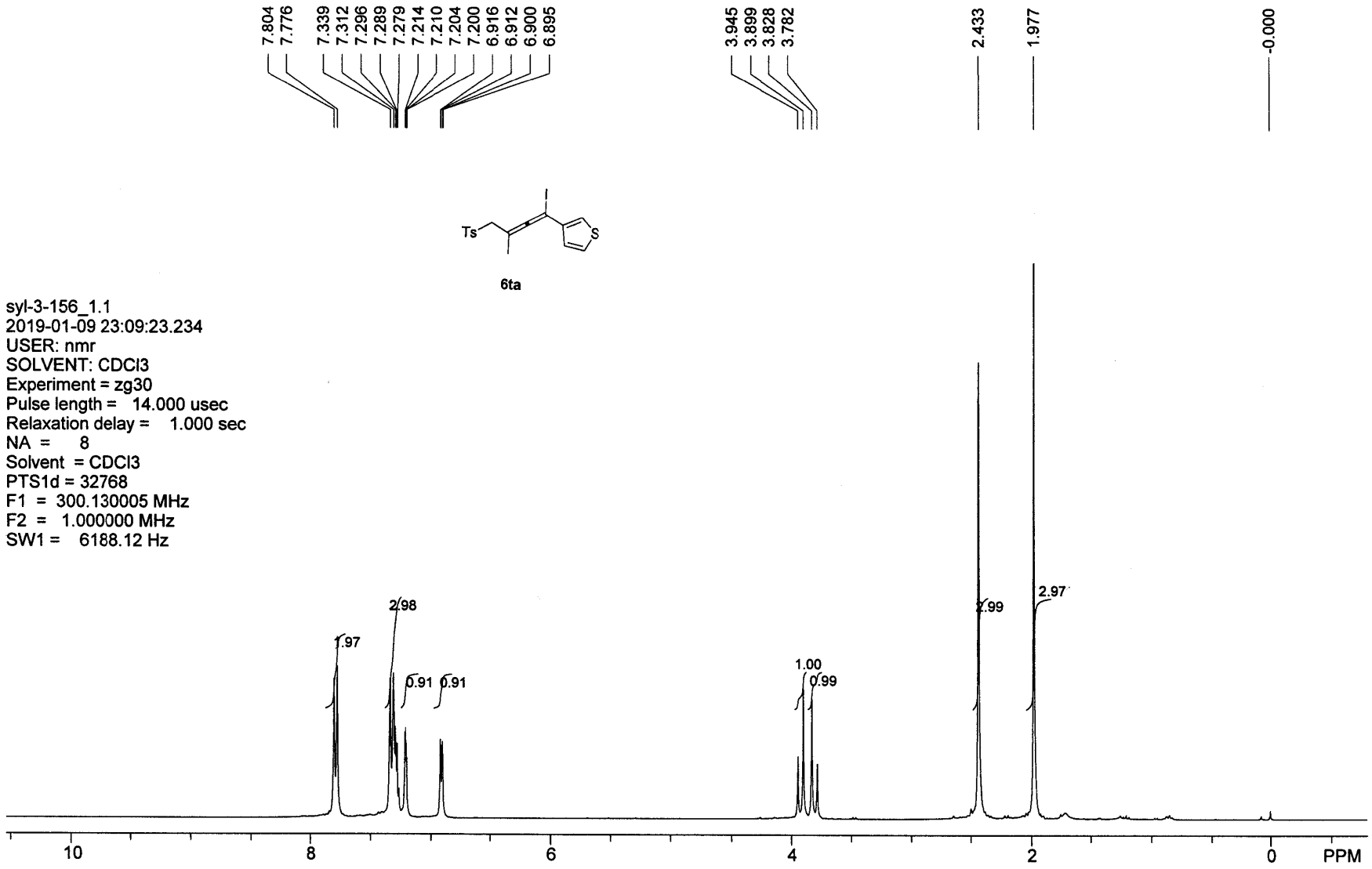

7.769
7.741
7.299
7.297
7.293
7.273
7.265
7.246
7.190
3.967
3.921
3.836
3.790
2.378
2.008
-0.000
I
Ts
Cl
6ra
syl-3-158_1.1
2019-01-10 20:23:04.453
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
6.08
1.97
1.04
1.00
3.00
2.94
10
8
6
4
2
0PPM

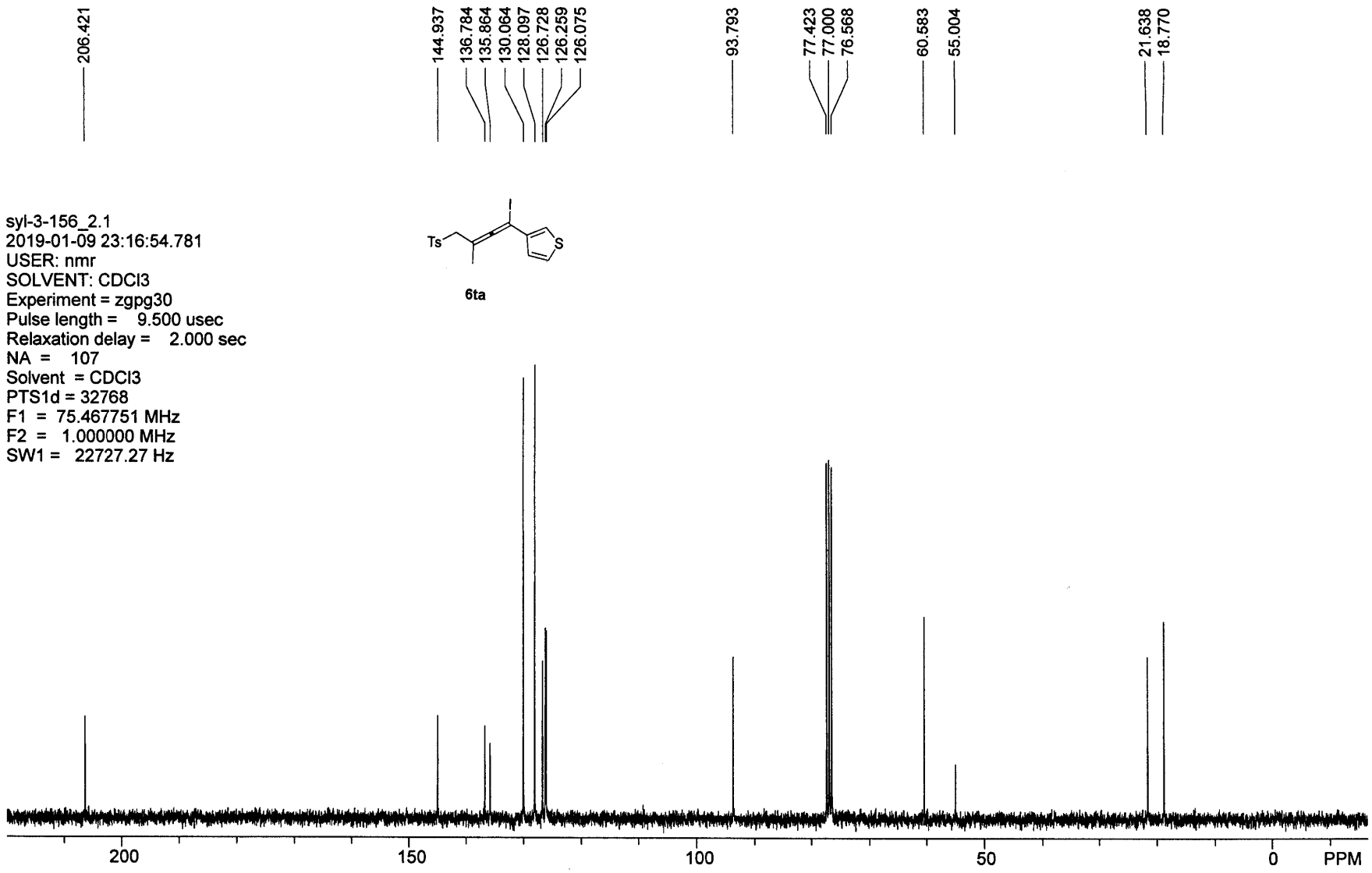

206.917
144.946
136.958
135.579
134.127
130.009
129.384
128.759
128.318
128.106
126.994
94.464
77.423
77.000
76.577
60.528
60.262
21.647
18.577
Ts
Cl
6ra
syl-3-158_2.1
2019-01-10 20:55:51.750
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 542
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
200
150
100
50
PPM

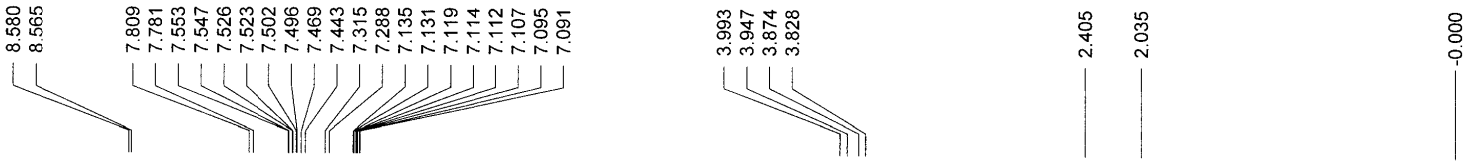

7.662
7.635
7.292
7.283
7.267
7.238
7.221
7.206
7.188
7.172
7.120
7.093
3.859
2.511
2.486
2.461
2.431
2.406
2.381
2.300
2.261
2.236
2.207
2.182
2.157
1.149
1.124
1.100
-0.000
Cl
Ts
Et
6sa

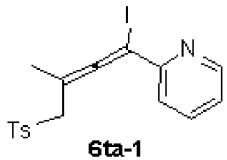

syl-4-16_1.1
2019-03-07 11:11:56.250
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
4.19
2.00
2.01
2.04
3.09
1.09
1.02
3.08
10
8
6
4
2
0
PPM


205.456
144.560
135.552
134.954
132.188
130.965
129.816
129.706
129.458
128.125
126.746
99.382
77.423
77.000
76.568
59.315
54.710
24.928
21.647
11.554
I
Cl
Ts
Et
6sa
syl-4-16_2.1
2019-03-07 11:23:03.078
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 175
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
200
150
100
50
0
PPM

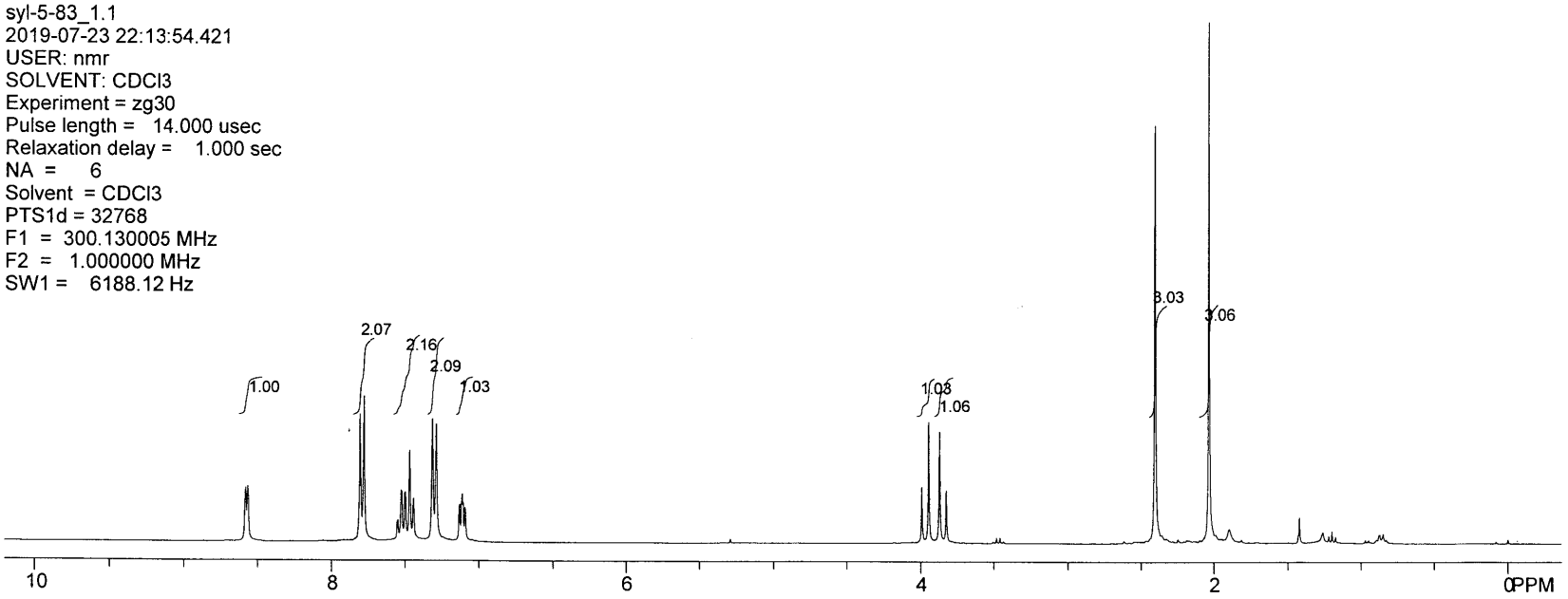

7.804
7.776
7.339
7.312
7.296
7.289
7.279
7.214
7.210
7.204
7.200
6.916
6.912
6.900
6.895
3.945
3.899
3.828
3.782
2.433
1.977
-0.000
Ts
I
S
6ta
syl-3-156_1.1
2019-01-09 23:09:23.234
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
1.97
2.98
0.91
0.91
1.00
0.99
2.99
2.97
10
8
6
4
2
0
PPM

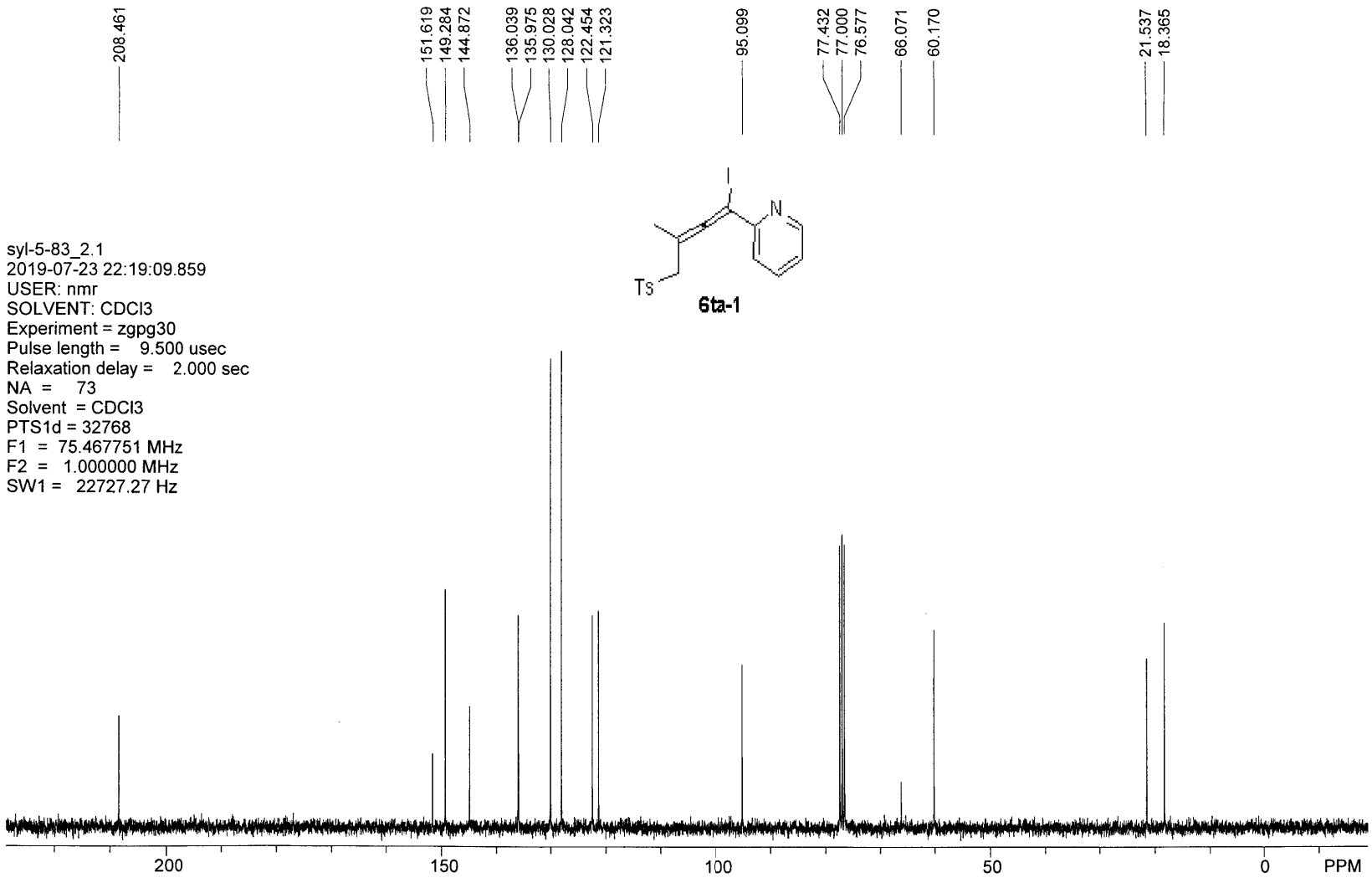

206.421
144.937
136.784
135.864
130.064
128.097
126.728
126.259
126.075
93.793
77.423
77.000
76.568
60.583
55.004
21.638
18.770
syl-3-156_2.1
2019-01-09 23:16:54.781
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 107
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
Ts
I
S
6ta
200
150
100
50
0
PPM

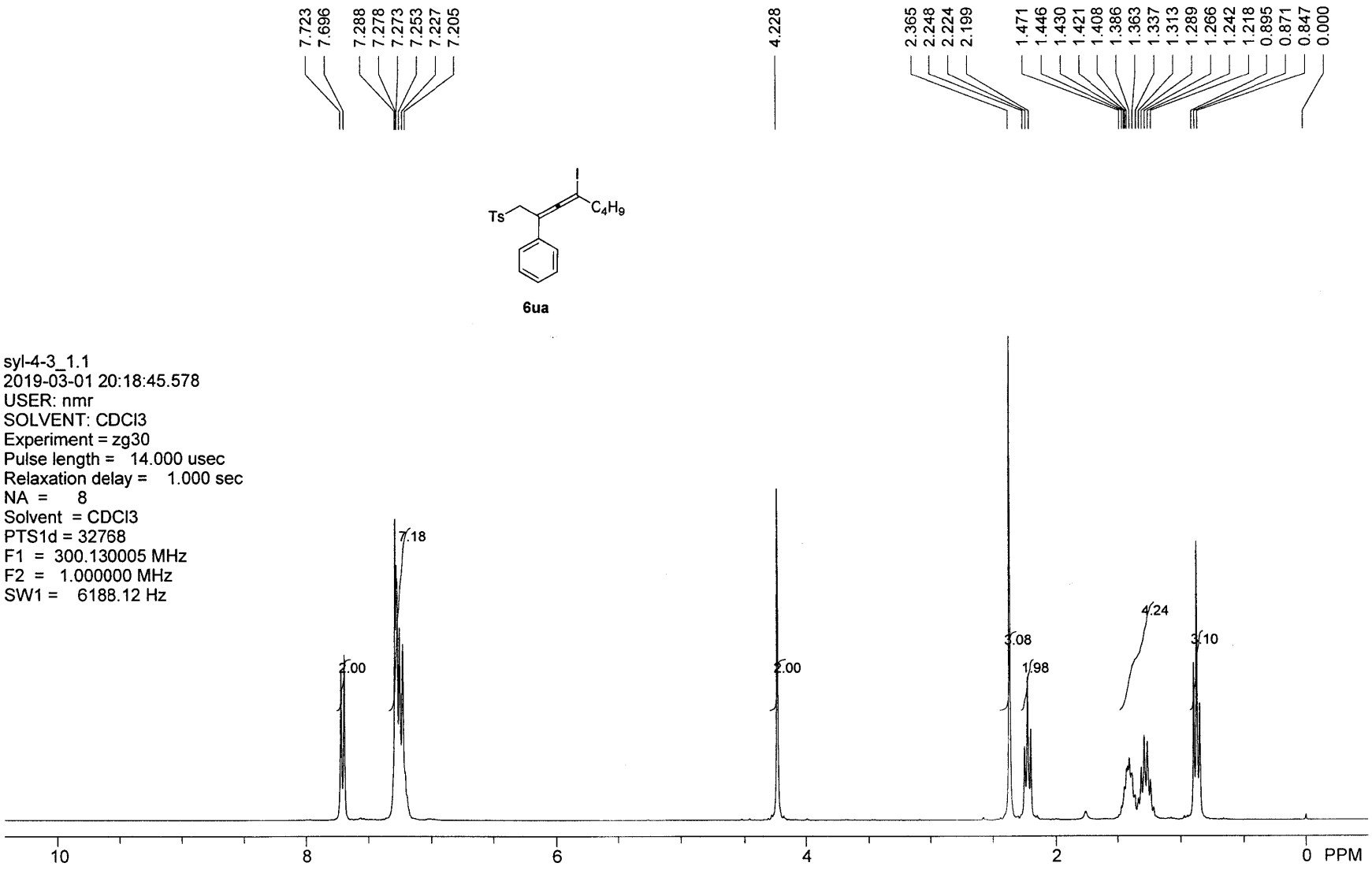

8.580
8.565
7.809
7.781
7.553
7.547
7.526
7.523
7.502
7.496
7.469
7.443
7.315
7.288
7.135
7.131
7.119
7.114
7.112
7.107
7.095
7.091
3.993
3.947
3.874
3.828
2.405
2.035
-0.000

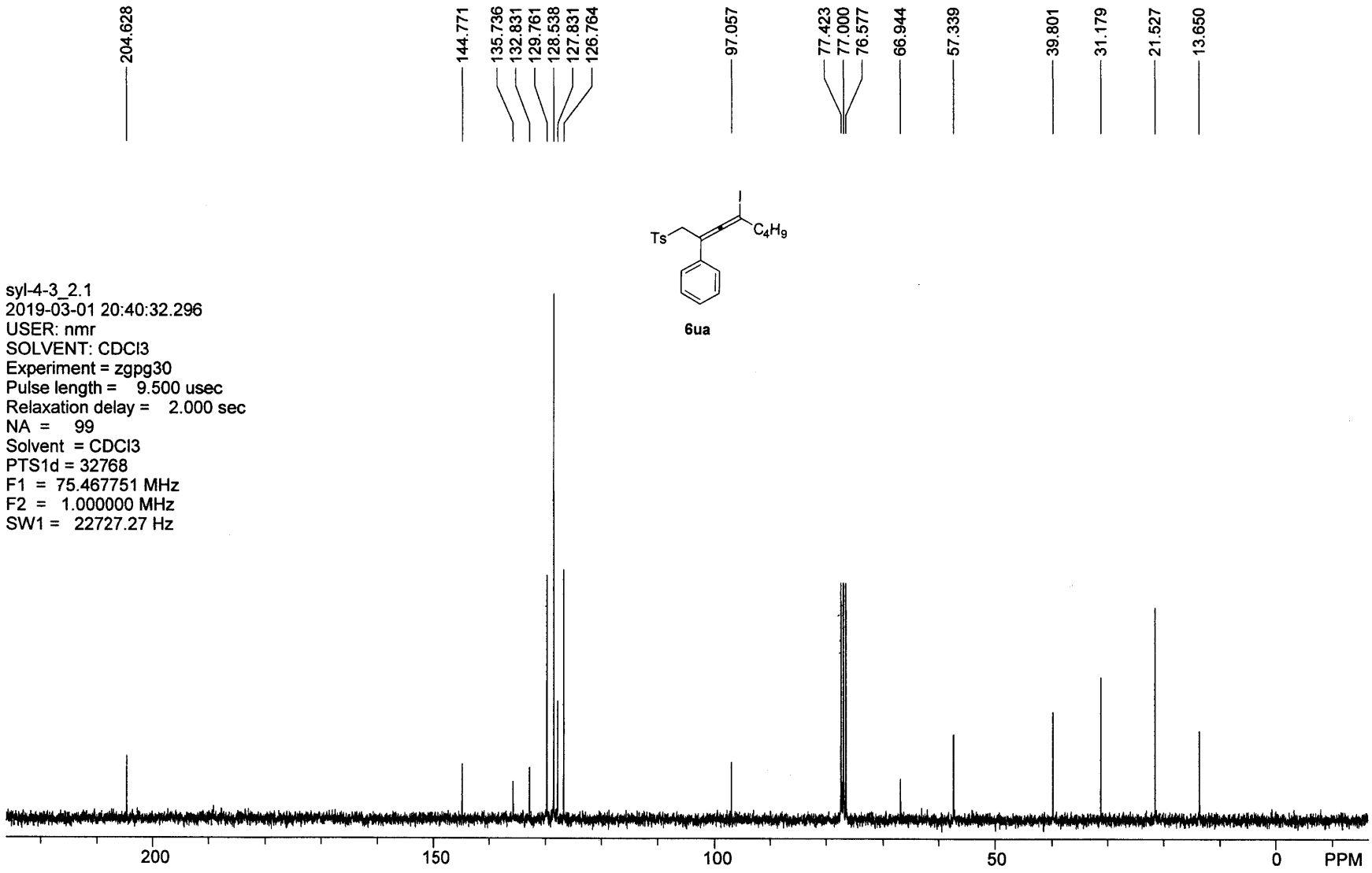

I
N
Ts

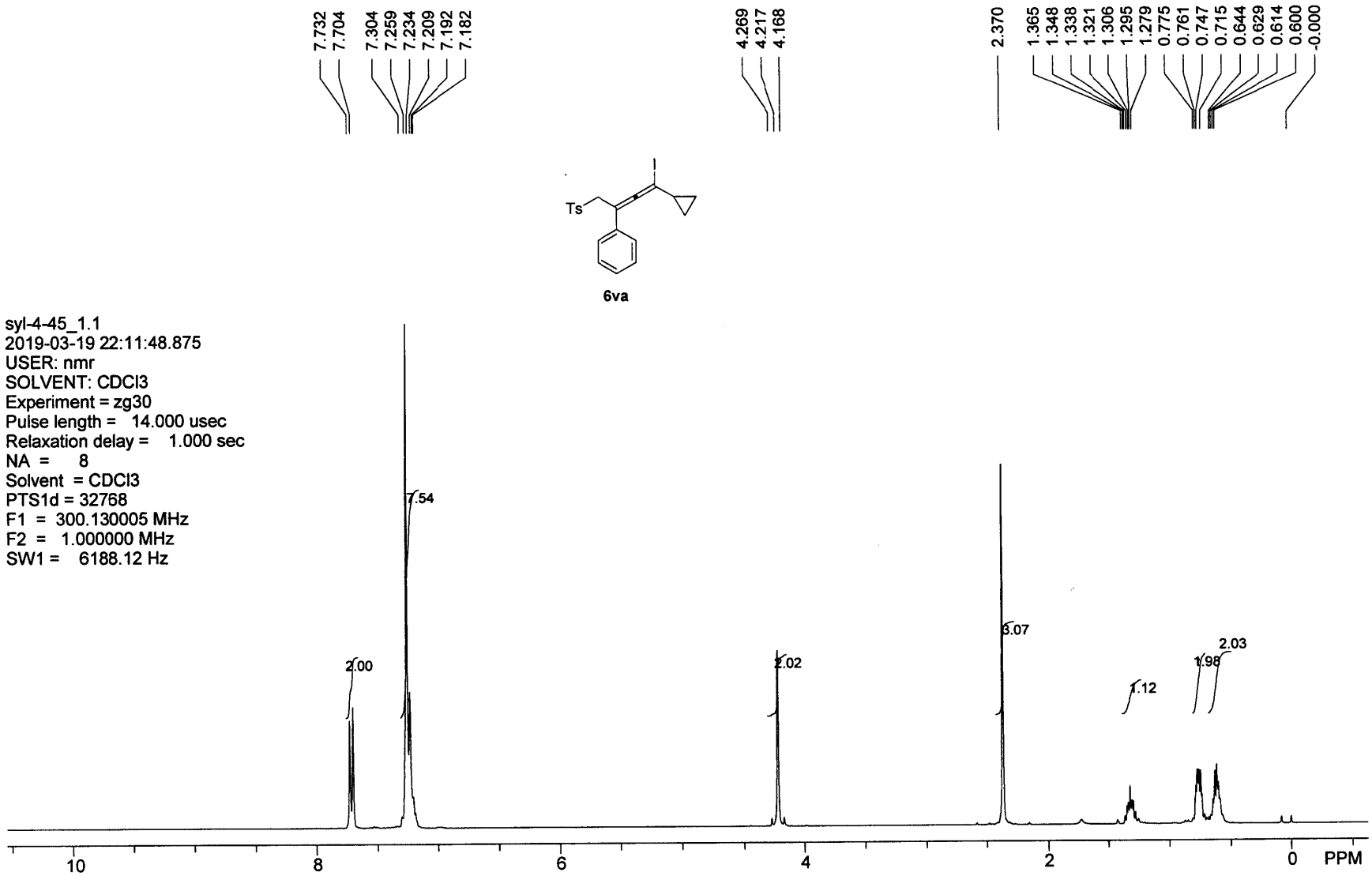
6ta-1

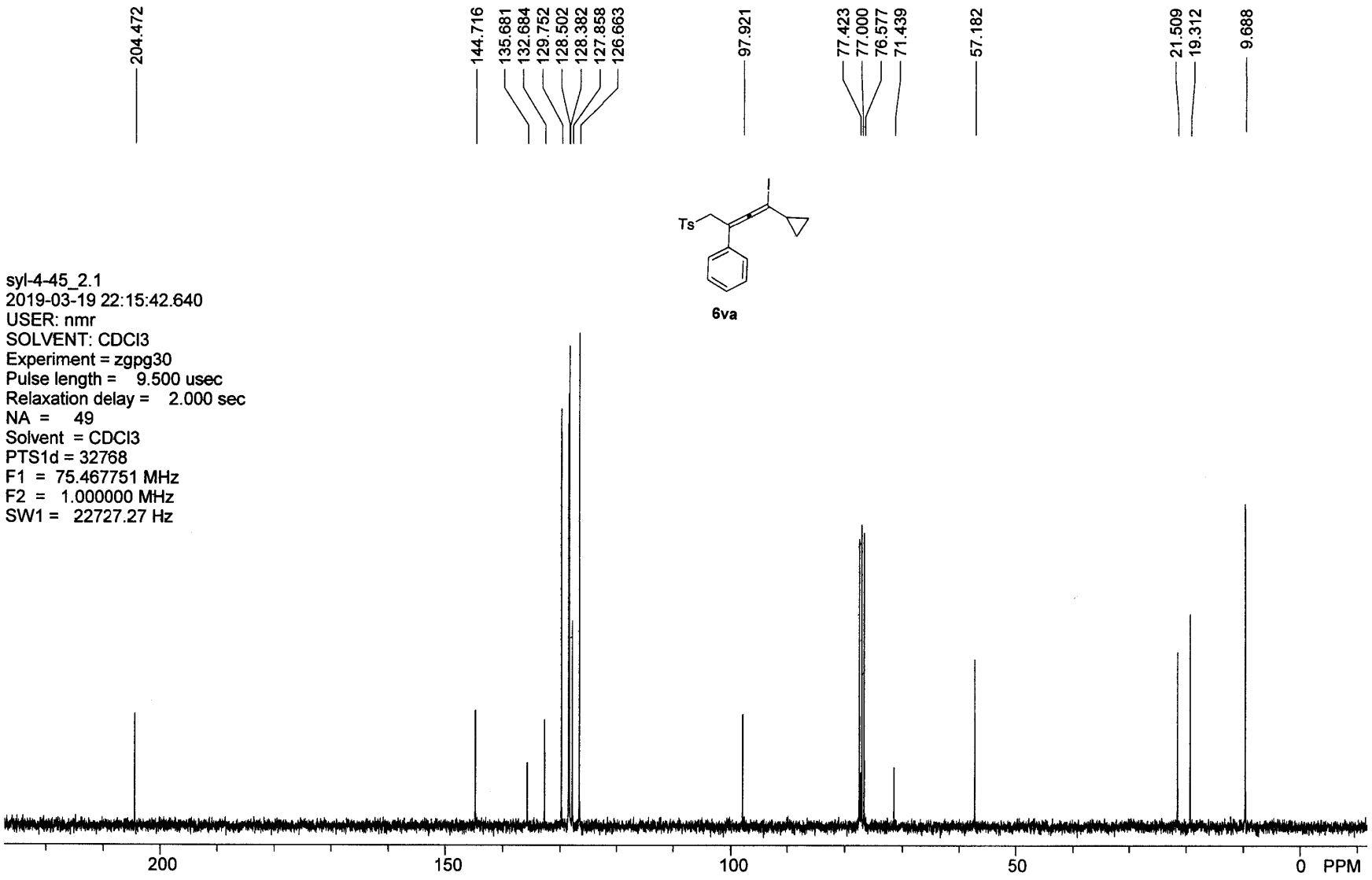

syl-5-83_1.1
2019-07-23 22:13:54.421
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 6
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
1.00
2.07
2.16
2.09
1.03
1.08
1.06
3.03
3.06
10
8
6
4
2
0PPM

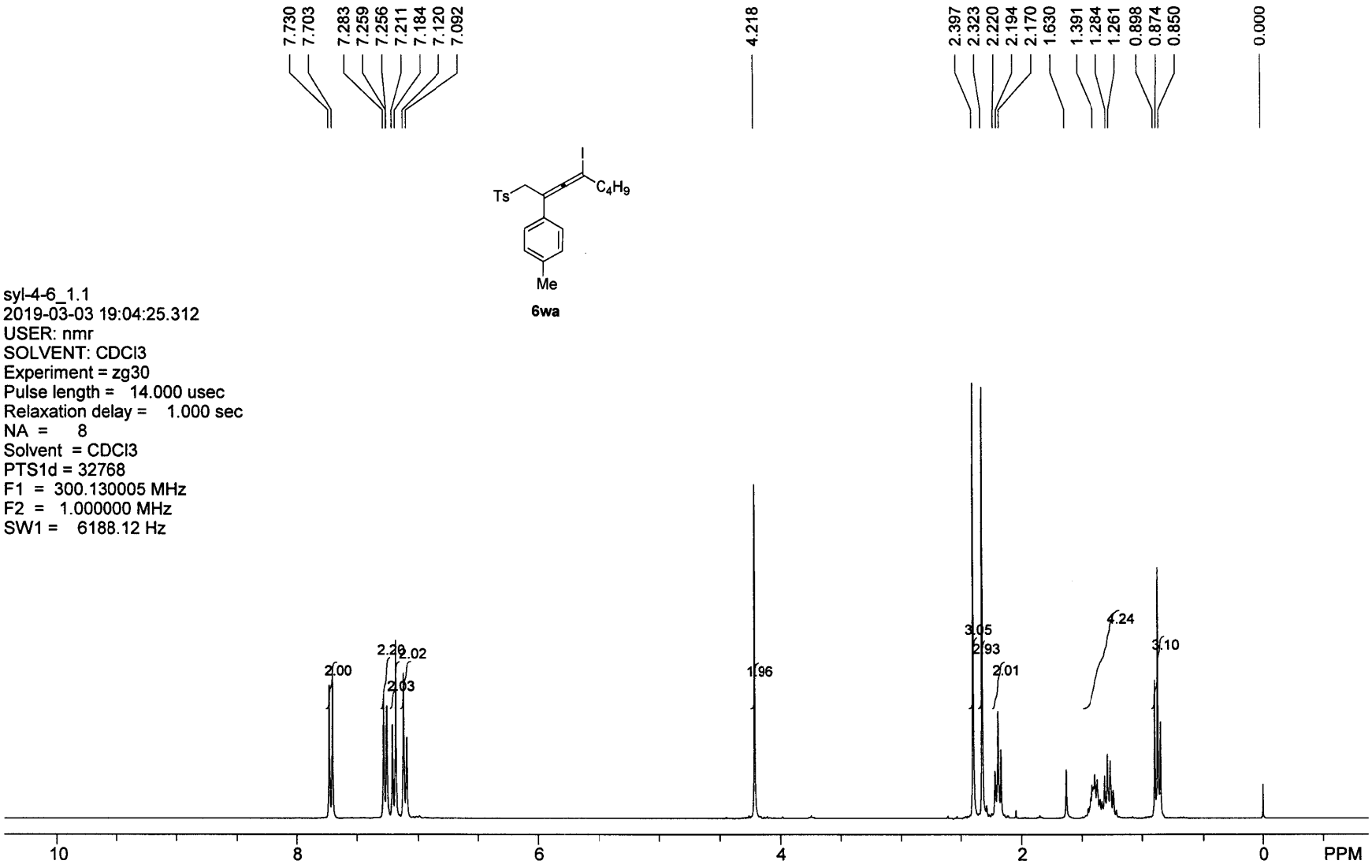

208.461
151.619
149.284
144.872
136.039
135.975
130.028
128.042
122.454
121.323
95.099
77.432
77.000
76.577
66.071
60.170
21.537
18.365
I
N
Ts
6ta-1
syl-5-83_2.1
2019-07-23 22:19:09.859
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 73
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
200
150
100
50
0
PPM

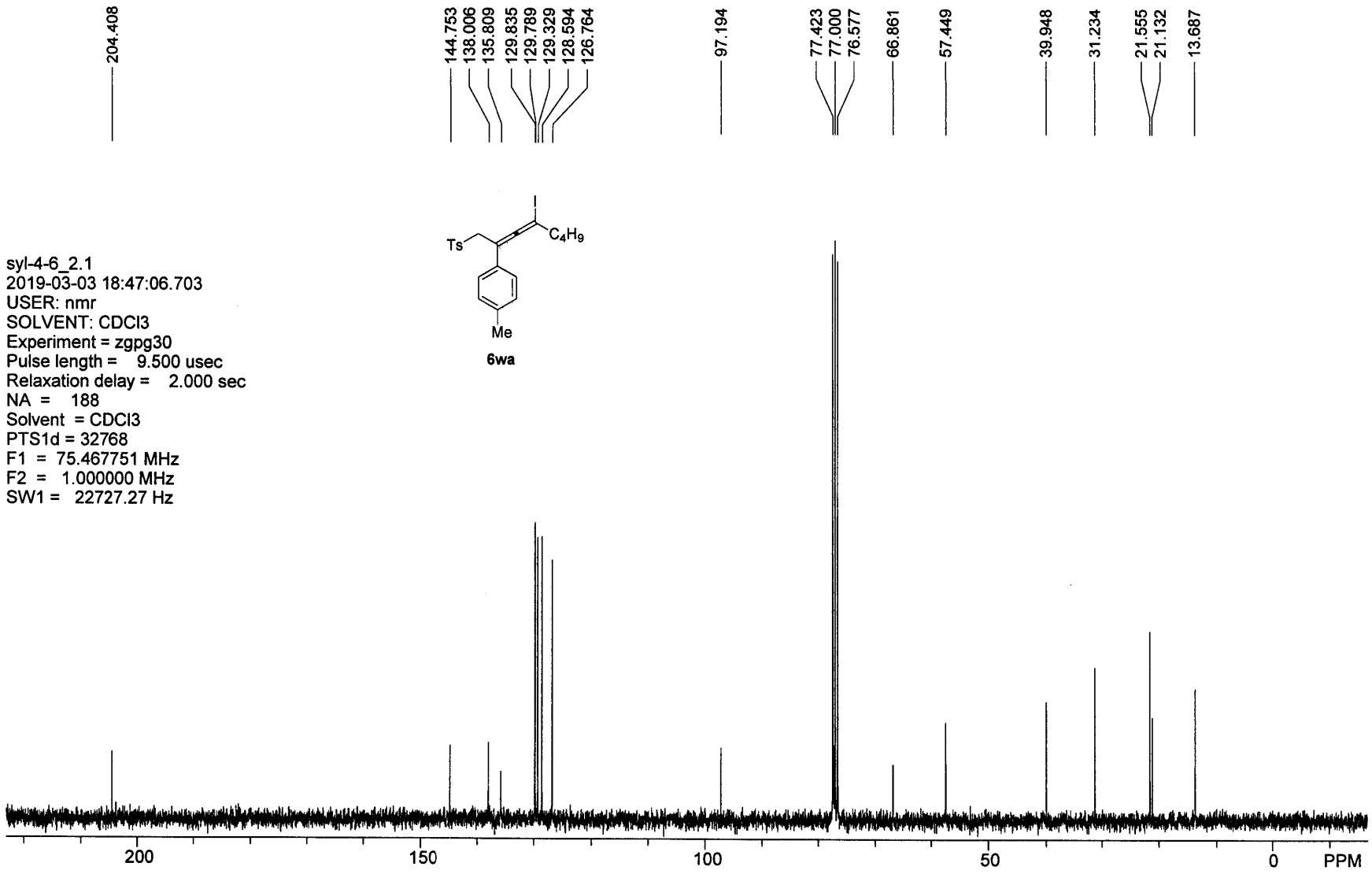

7.723
7.696
7.288
7.278
7.273
7.253
7.227
7.205
4.228
2.365
2.248
2.224
2.199
1.471
1.446
1.430
1.421
1.408
1.386
1.363
1.337
1.313
1.289
1.266
1.242
1.218
0.895
0.871
0.847
0.000
Ts
C4H9
6ua
syl-4-3_1.1
2019-03-01 20:18:45.578
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
2.00
7.18
2.00
3.08
1.98
4.24
3.10
10
8
6
4
2
0 PPM

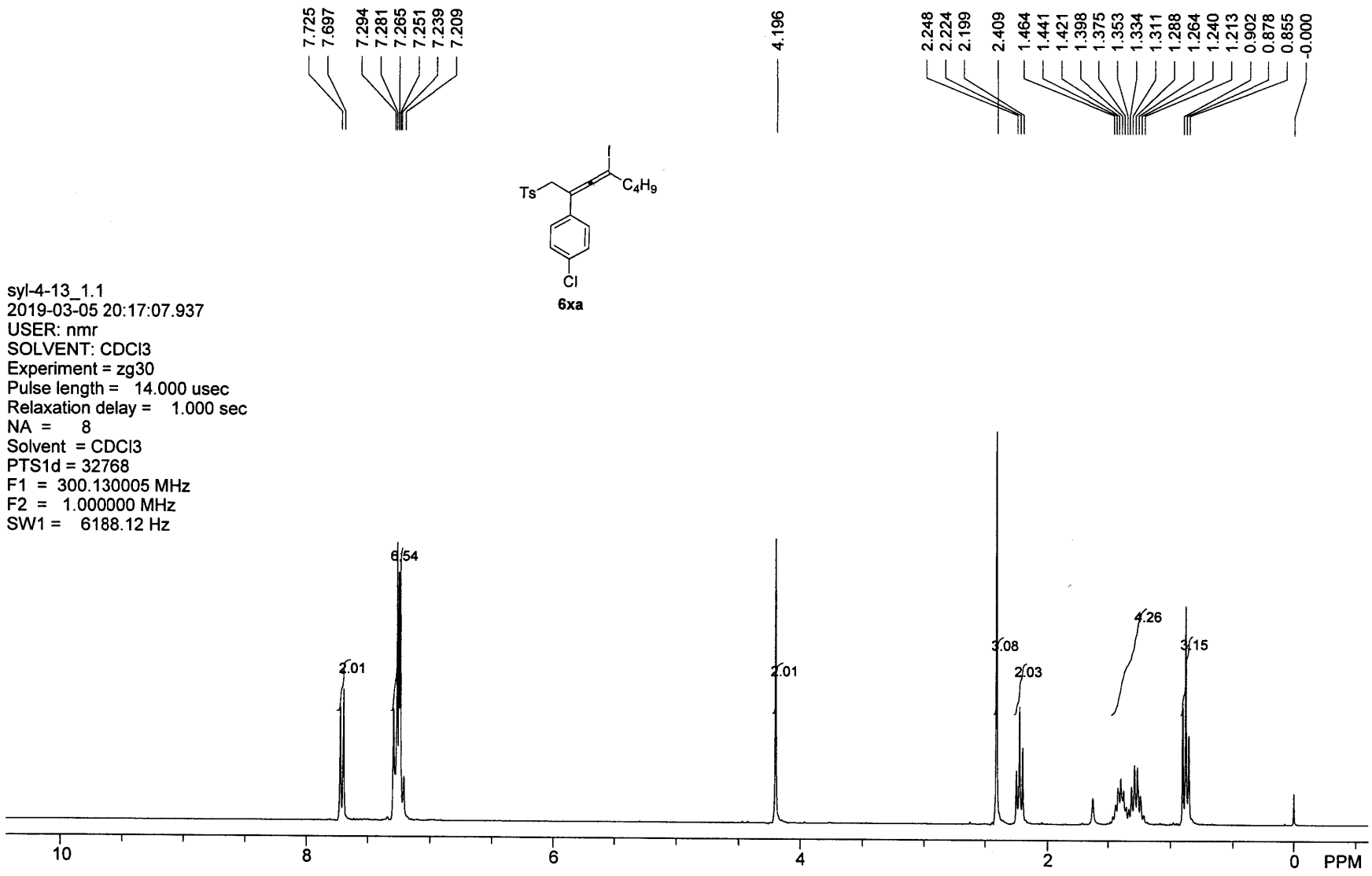

204.628
144.771
135.736
132.831
129.761
128.538
127.831
126.764
97.057
77.423
77.000
76.577
66.944
57.339
39.801
31.179
21.527
13.650
Ts
C_4H_9
6ua
syl-4-3_2.1
2019-03-01 20:40:32.296
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 99
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
200
150
100
50
0
PPM

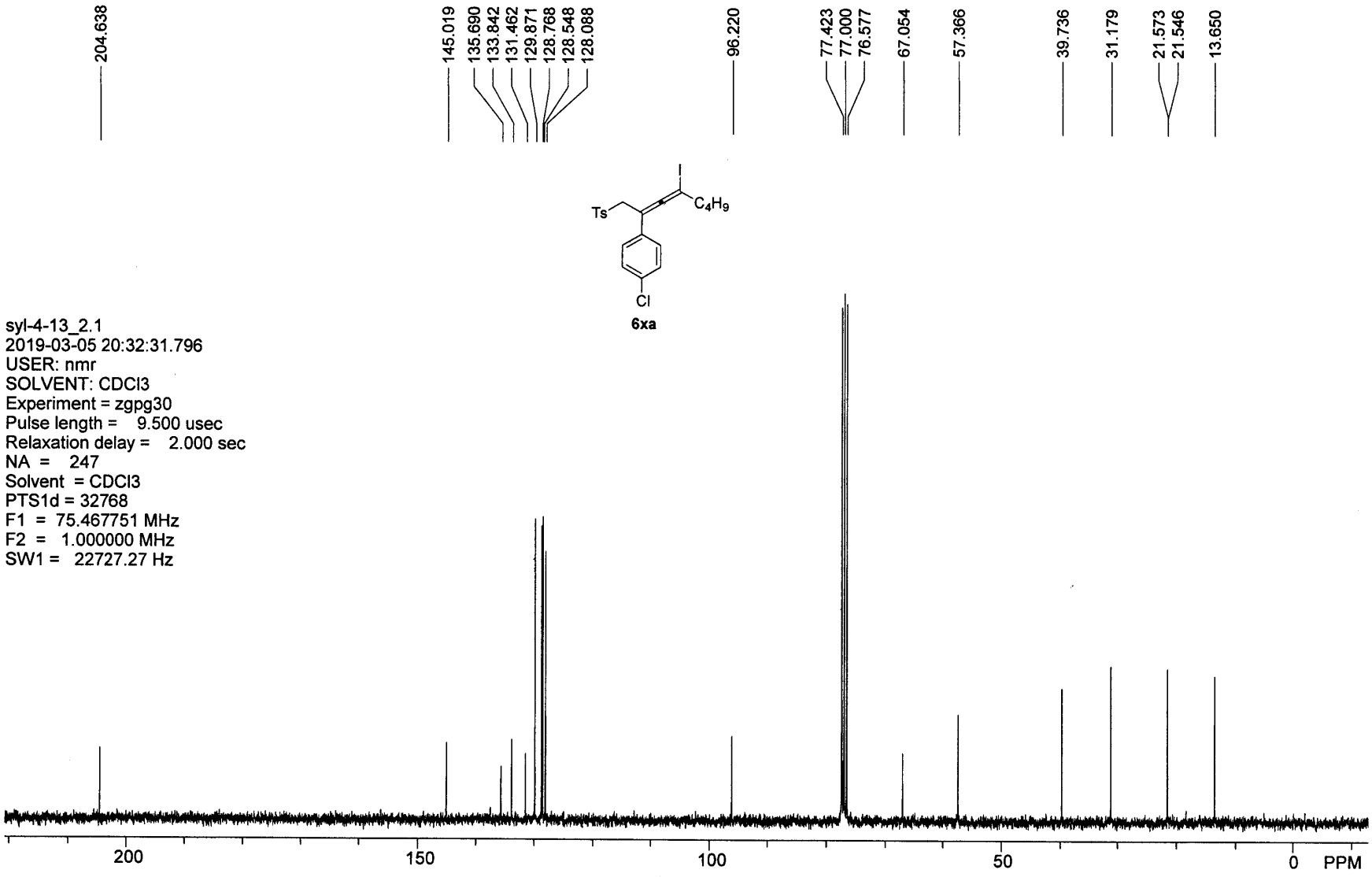

7.732
7.704
7.304
7.259
7.234
7.209
7.192
7.182
4.269
4.217
4.168
2.370
1.365
1.348
1.338
1.321
1.306
1.295
1.279
0.775
0.761
0.747
0.715
0.644
0.629
0.614
0.600
-0.000
Ts
I
6va
syl-4-45_1.1
2019-03-19 22:11:48.875
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
2.00
7.54
2.02
6.07
1.12
1.98
2.03
10
8
6
4
2
0
PPM

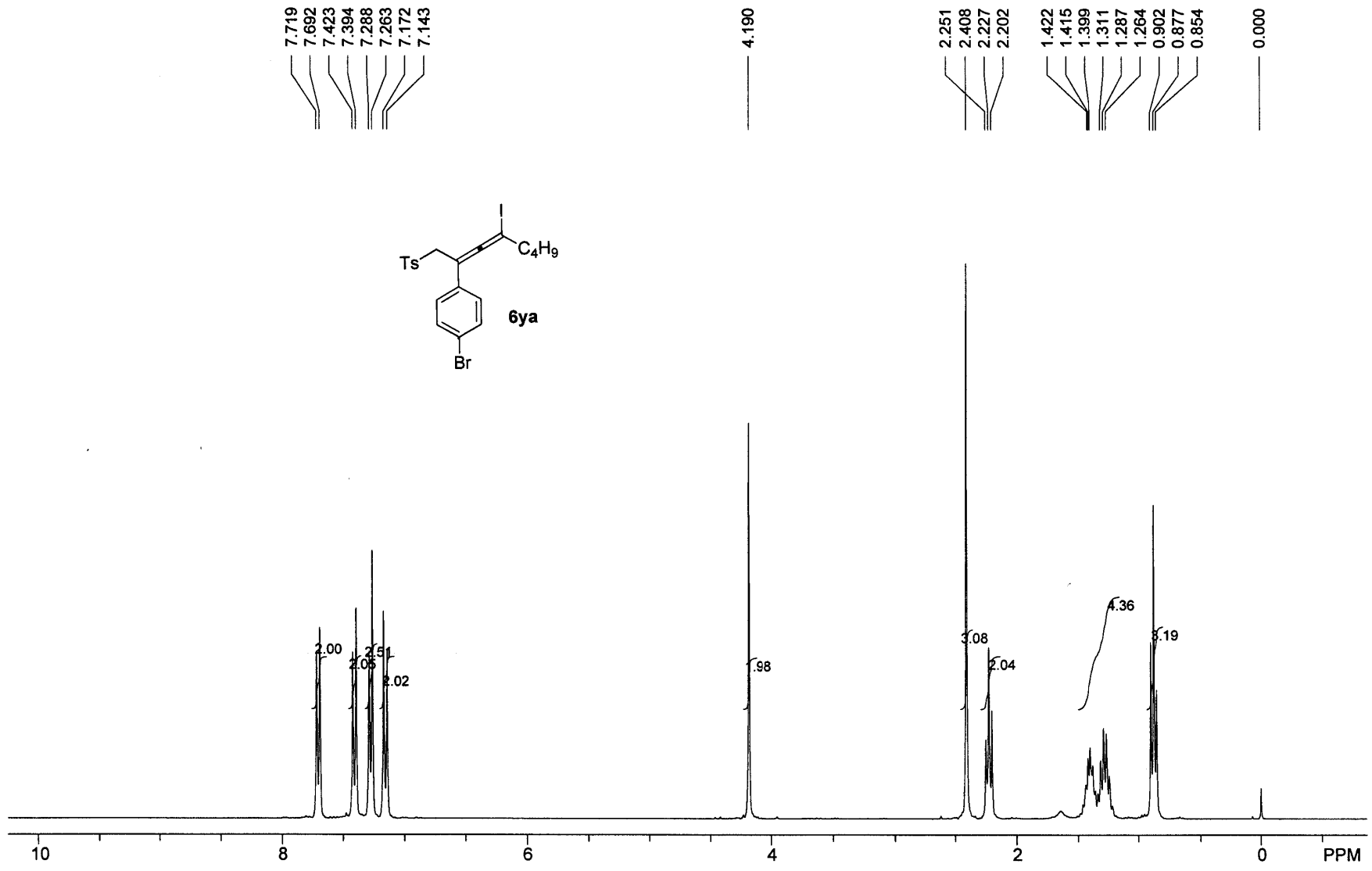

204.472
144.716
135.681
132.684
129.752
128.502
128.382
127.858
126.663
97.921
77.423
77.000
76.577
71.439
57.182
21.509
19.312
9.688
Ts
6va
syl-4-45_2.1
2019-03-19 22:15:42.640
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 49
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
200
150
100
50
0 PPM

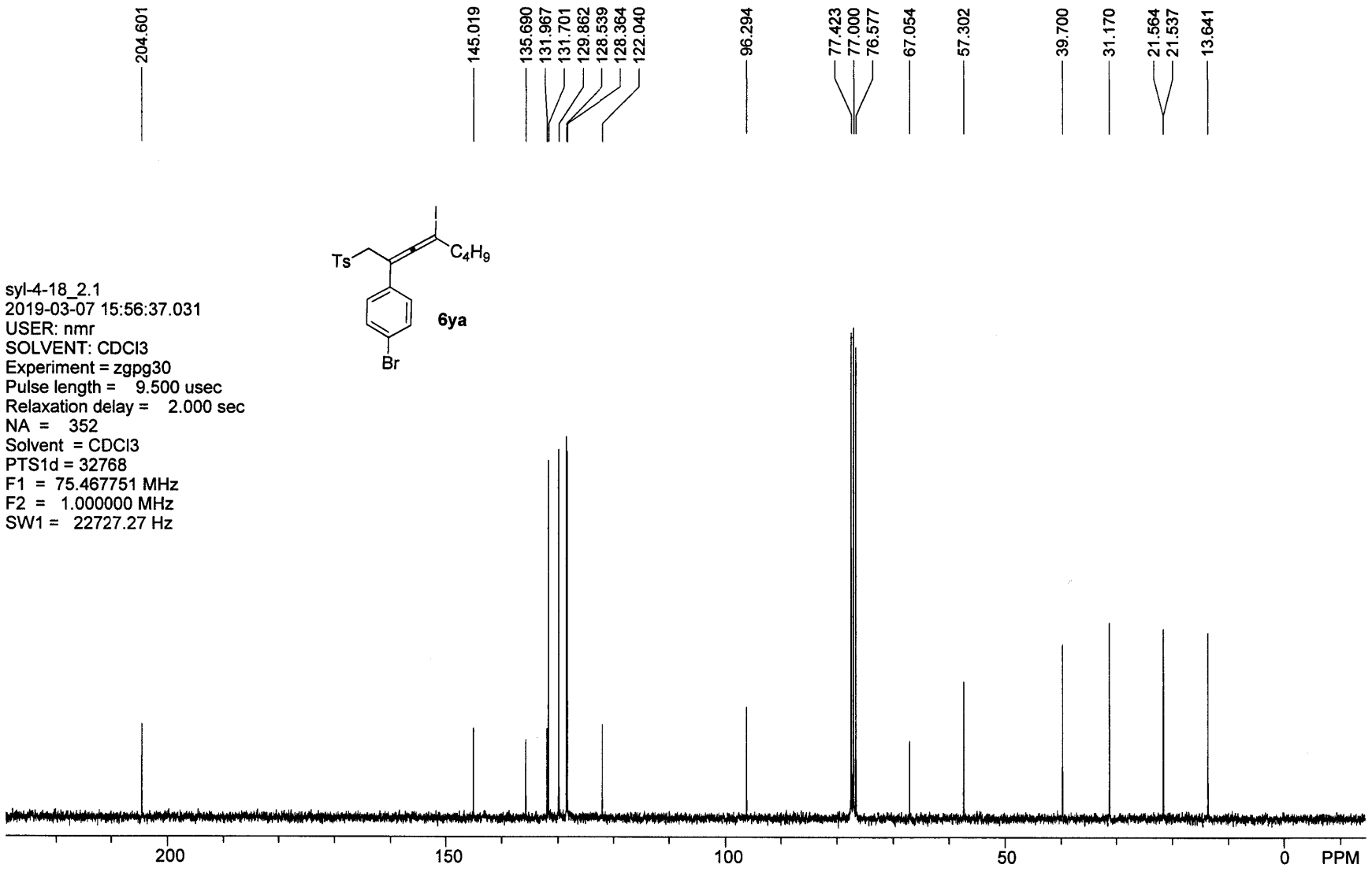

7.730
7.703
7.283
7.259
7.256
7.211
7.184
7.120
7.092
4.218
2.397
2.323
2.220
2.194
2.170
1.630
1.391
1.284
1.261
0.898
0.874
0.850
0.000
Ts
I
C4H9
Me
6wa
syl-4-6_1.1
2019-03-03 19:04:25.312
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
2.00
2.20
2.02
2.03
1.96
3.05
2.93
2.01
4.24
3.10
10
8
6
4
2
0
PPM

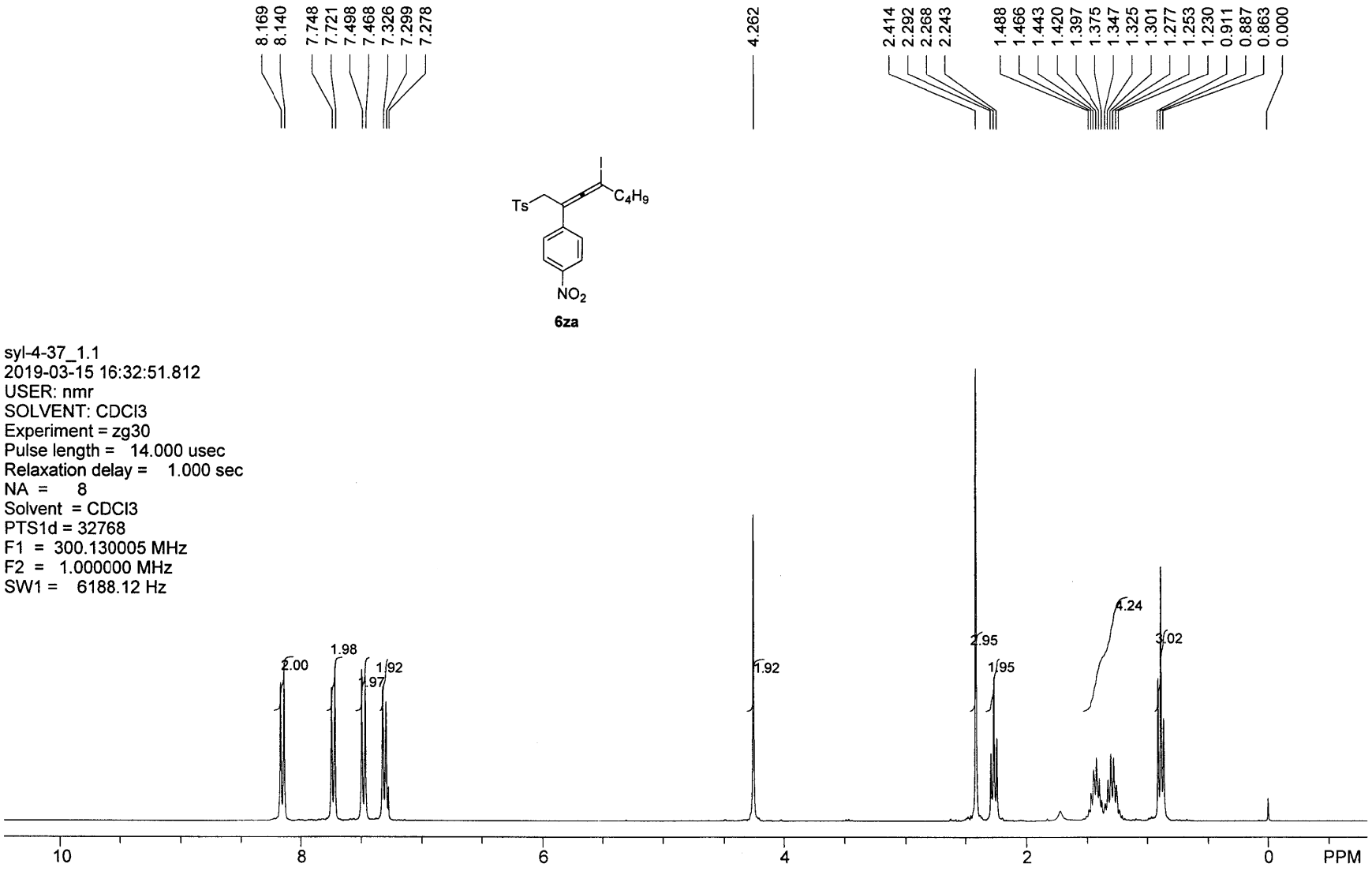

204.408
144.753
138.006
135.809
129.835
129.789
129.329
128.594
126.764
97.194
77.423
77.000
76.577
66.861
57.449
39.948
31.234
21.555
21.132
13.687
I
Ts
C4H9
Me
6wa
syl-4-6_2.1
2019-03-03 18:47:06.703
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 188
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
200
150
100
50
0
PPM

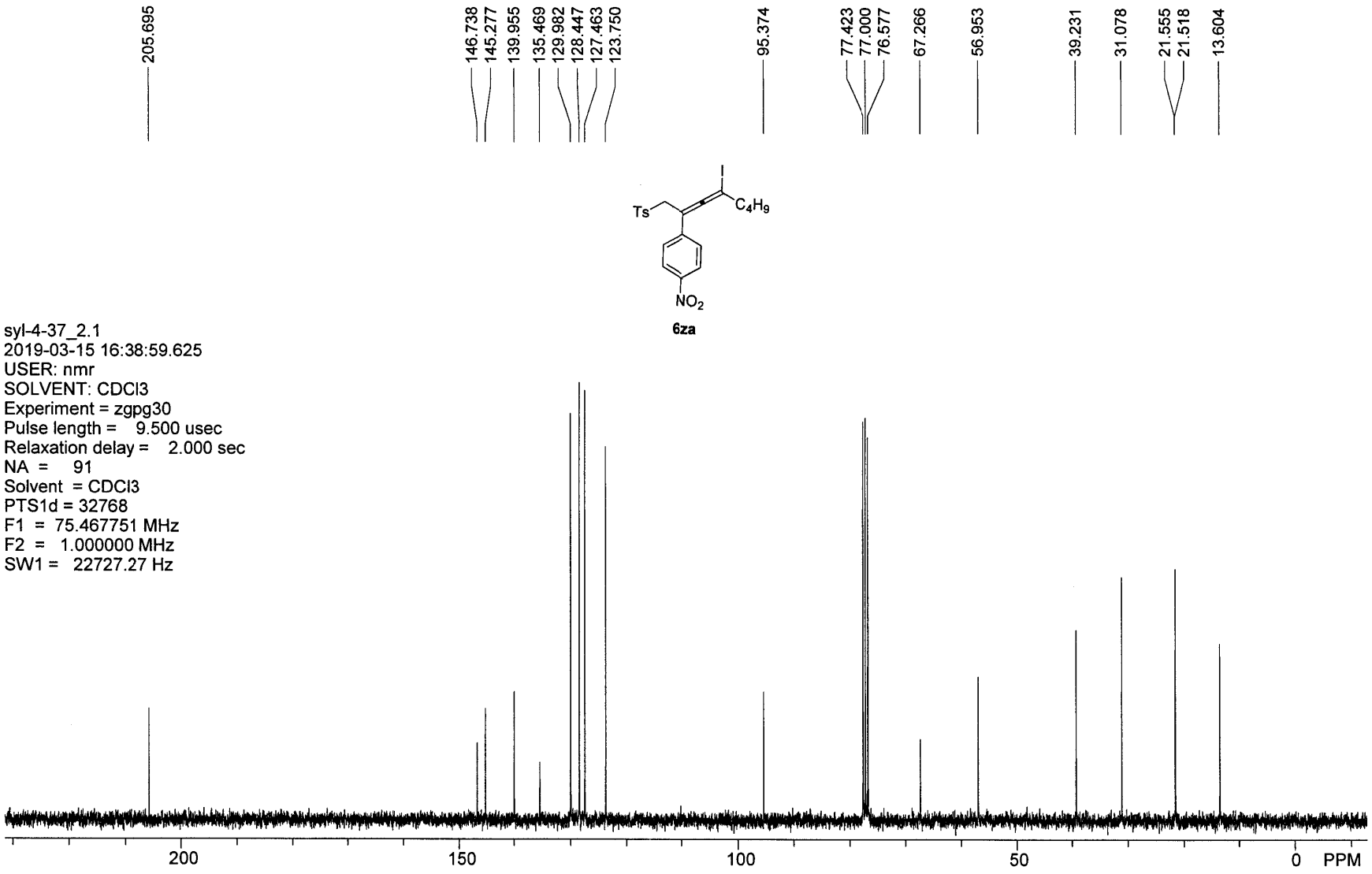

7.725
7.697
7.294
7.281
7.265
7.251
7.239
7.209
4.196
2.248
2.224
2.199
2.409
1.464
1.441
1.421
1.398
1.375
1.353
1.334
1.311
1.288
1.264
1.240
1.213
0.902
0.878
0.855
-0.000
Ts
C4H9
Cl
6xa
syl-4-13_1.1
2019-03-05 20:17:07.937
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
2.01
6.54
2.01
3.08
2.03
4.26
3.15
10
8
6
4
2
0
PPM

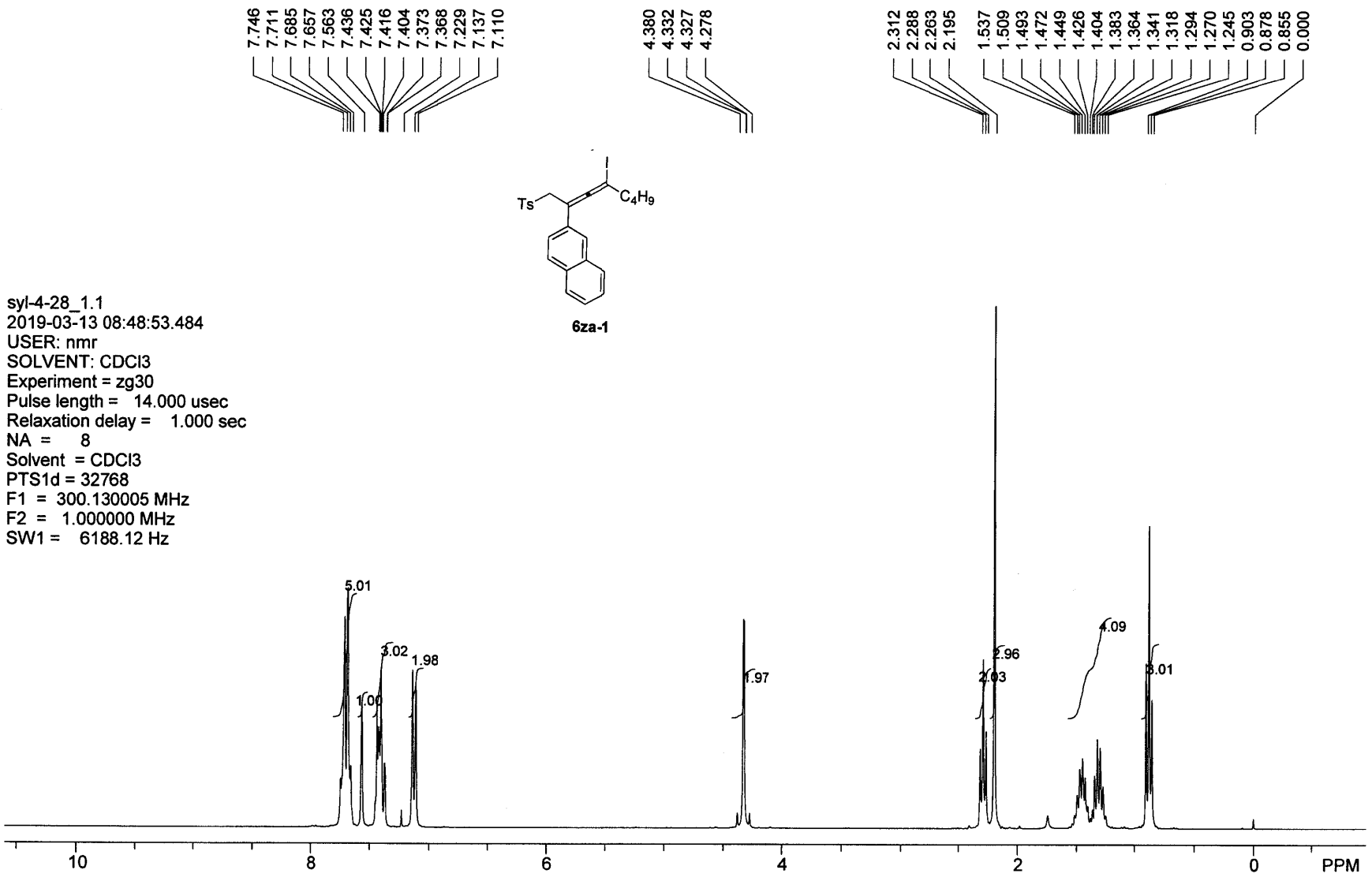

204.638
145.019
135.690
133.842
131.462
129.871
128.768
128.548
128.088
96.220
77.423
77.000
76.577
67.054
57.366
39.736
31.179
21.573
21.546
13.650
I
Ts
C4H9
Cl
6xa
syl-4-13_2.1
2019-03-05 20:32:31.796
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 247
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
200
150
100
50
0
PPM

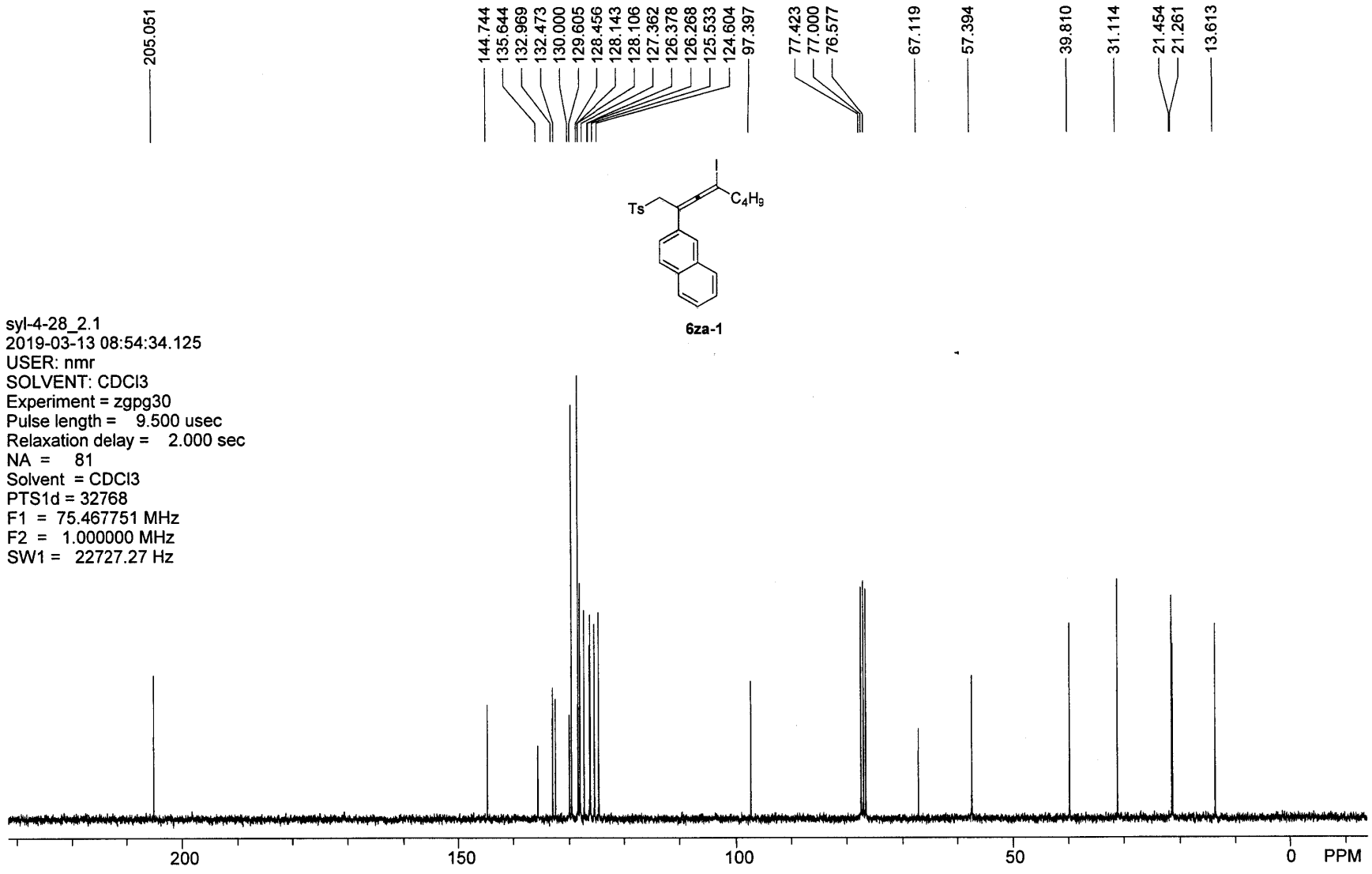

7.719
7.692
7.423
7.394
7.288
7.263
7.172
7.143
4.190
2.251
2.408
2.227
2.202
1.422
1.415
1.399
1.311
1.287
1.264
0.902
0.877
0.854
0.000
I
C4H9
Ts
6ya
Br
2.00
2.05
2.51
2.02
1.98
3.08
2.04
4.36
3.19
10
8
6
4
2
0
PPM

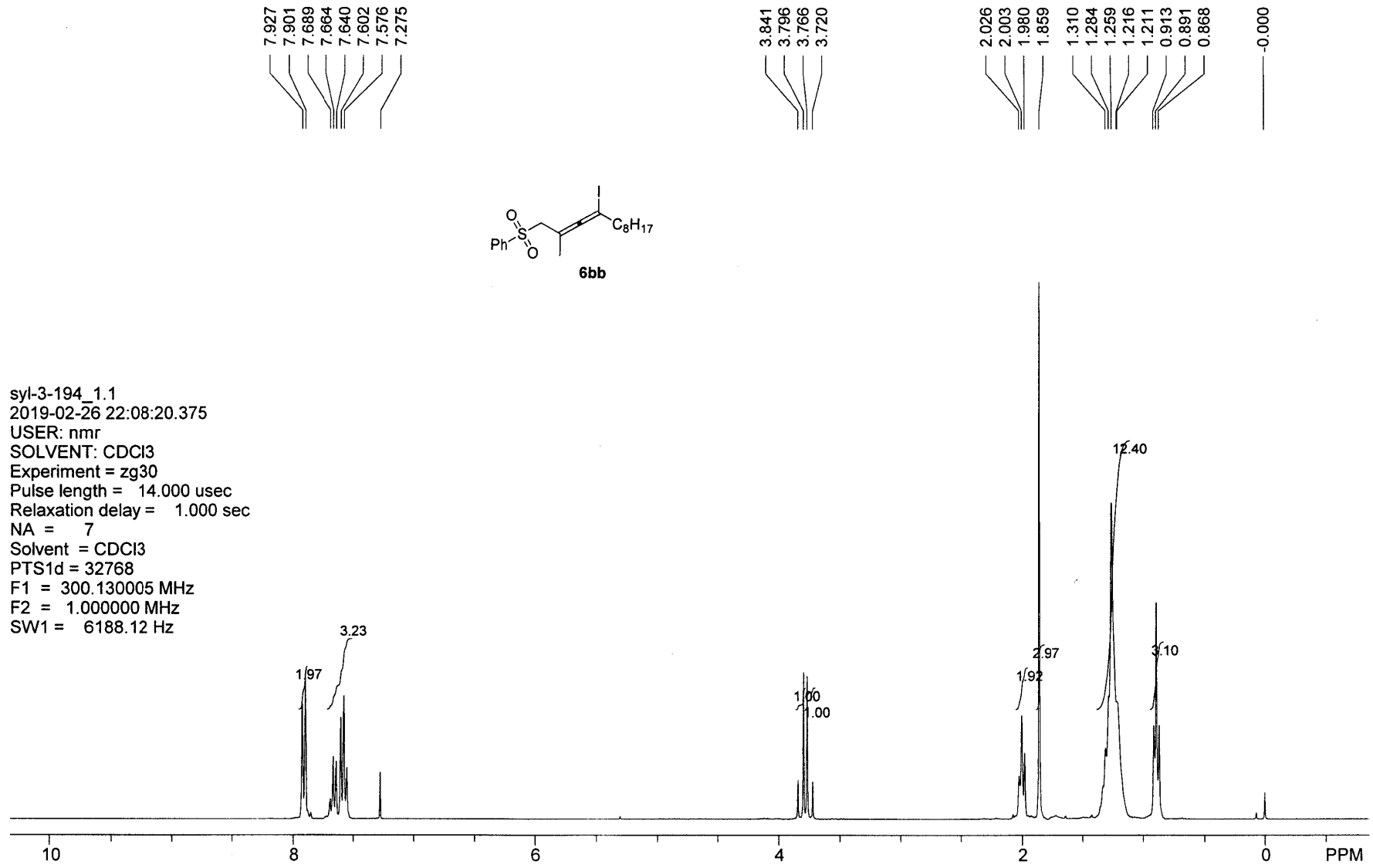

204.601
145.019
135.690
131.967
131.701
129.862
128.539
128.364
122.040
96.294
77.423
77.000
76.577
67.054
57.302
39.700
31.170
21.564
21.537
13.641
Ts
C4H9
Br
6ya
syl-4-18_2.1
2019-03-07 15:56:37.031
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 352
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
200
150
100
50
0
PPM

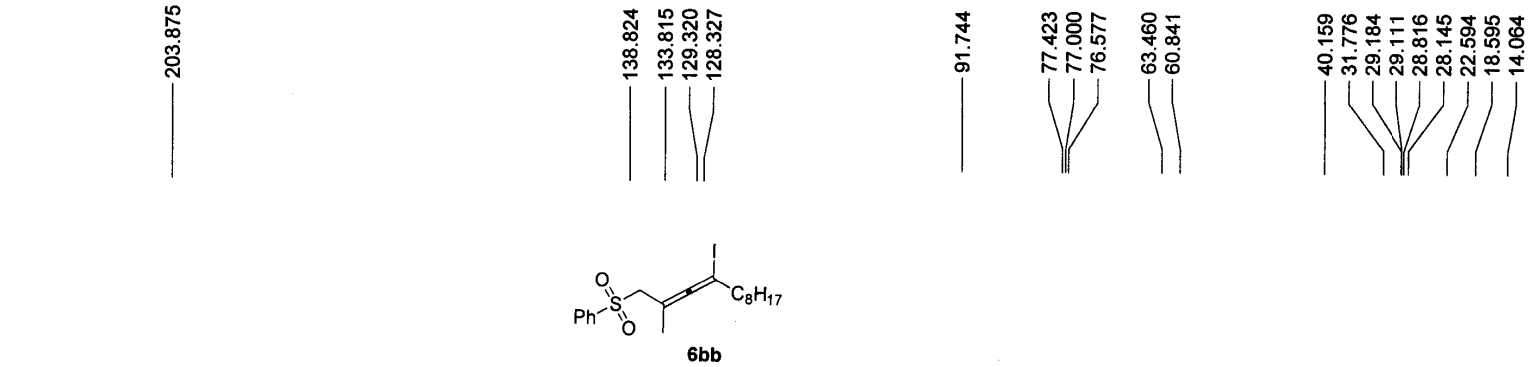

8.169
8.140
7.748
7.721
7.498
7.468
7.326
7.299
7.278
4.262
2.414
2.292
2.268
2.243
1.488
1.466
1.443
1.420
1.397
1.375
1.347
1.325
1.301
1.277
1.253
1.230
0.911
0.887
0.863
0.000
Ts
C4H9
NO2
6za
syl-4-37_1.1
2019-03-15 16:32:51.812
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
2.00
1.98
1.92
1.97
1.92
2.95
1.95
4.24
3.02
10
8
6
4
2
0
PPM


205.695
146.738
145.277
139.955
135.469
129.982
128.447
127.463
123.750
95.374
77.423
77.000
76.577
67.266
56.953
39.231
31.078
21.555
21.518
13.604
I
Ts
C4H9
NO2
6za
syl-4-37_2.1
2019-03-15 16:38:59.625
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 91
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
200
150
100
50
0
PPM


7.746
7.711
7.685
7.657
7.563
7.436
7.425
7.416
7.404
7.373
7.368
7.229
7.137
7.110
4.380
4.332
4.327
4.278
2.312
2.288
2.263
2.195
1.537
1.509
1.493
1.472
1.449
1.426
1.404
1.383
1.364
1.341
1.318
1.294
1.270
1.245
0.903
0.878
0.855
0.000
Ts
C4H9
6za-1
syl-4-28_1.1
2019-03-13 08:48:53.484
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
5.01
1.00
3.02
1.98
1.97
2.03
2.96
4.09
3.01
10
8
6
4
2
0
PPM

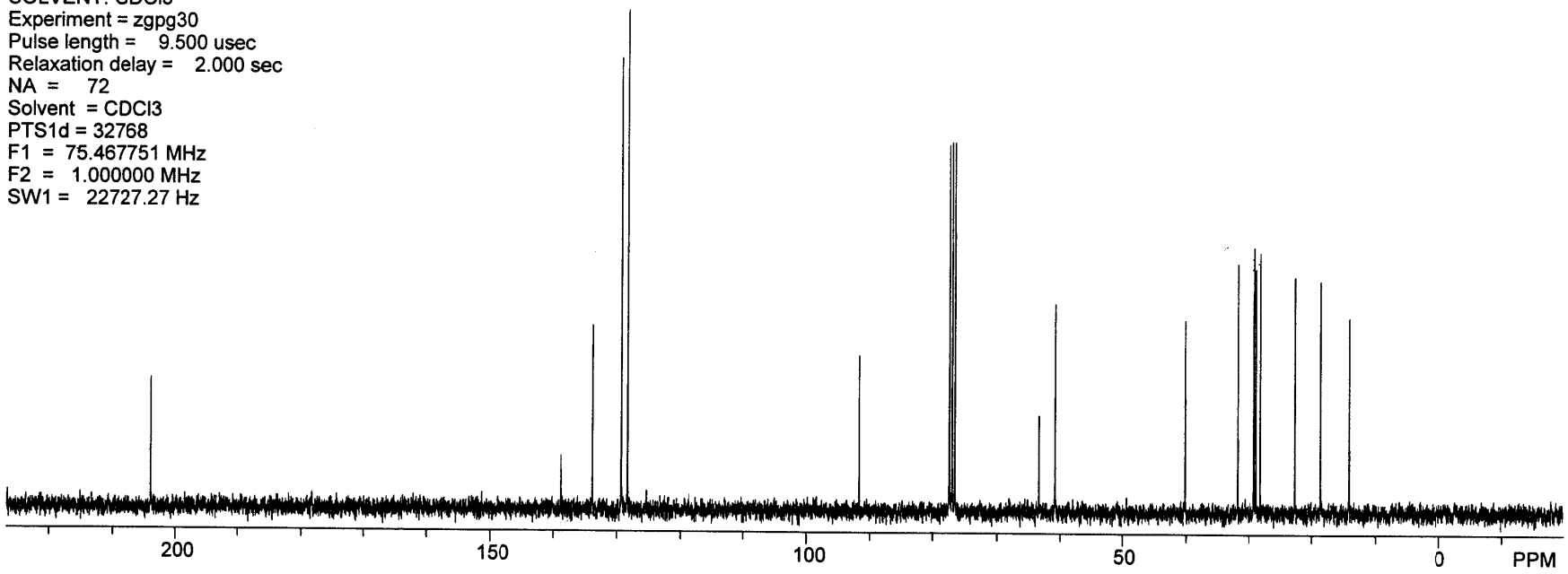

205.051
144.744
135.644
132.969
132.473
130.000
129.605
128.456
128.143
128.106
127.362
126.378
126.268
125.533
124.604
97.397
77.423
77.000
76.577
67.119
57.394
39.810
31.114
21.454
21.261
13.613
Ts
I
C4H9
6za-1
syl-4-28_2.1
2019-03-13 08:54:34.125
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 81
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
200
150
100
50
0
PPM

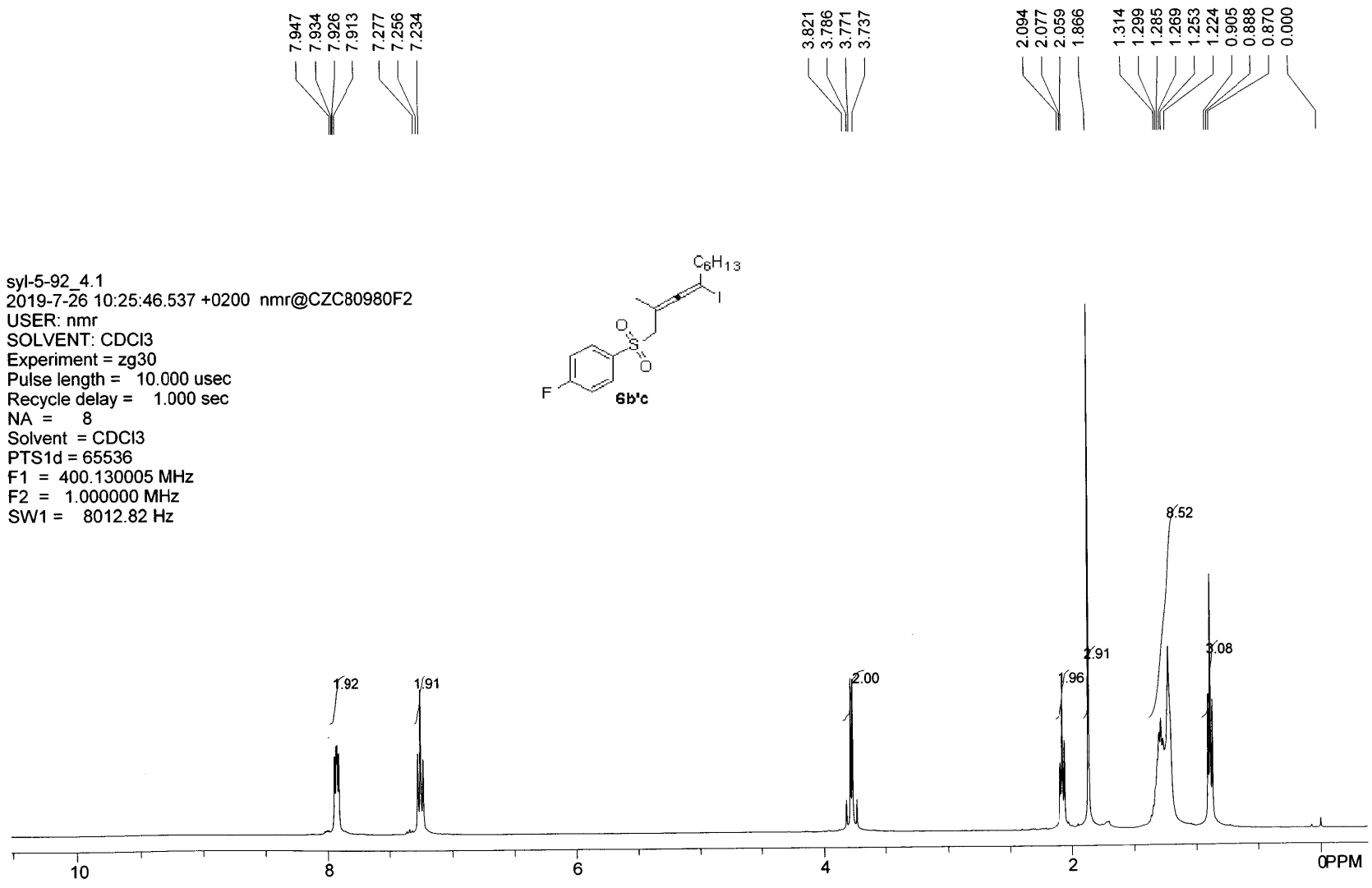

7.927
7.901
7.689
7.664
7.640
7.602
7.576
7.275
3.841
3.796
3.766
3.720
2.026
2.003
1.980
1.859
1.310
1.284
1.259
1.216
1.211
0.913
0.891
0.868
-0.000
I
O
S
O
Ph
C8H17
6bb
syl-3-194_1.1
2019-02-26 22:08:20.375
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 7
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
1.97
3.23
1.00
1.00
1.92
2.97
12.40
3.10
10
8
6
4
2
0
PPM

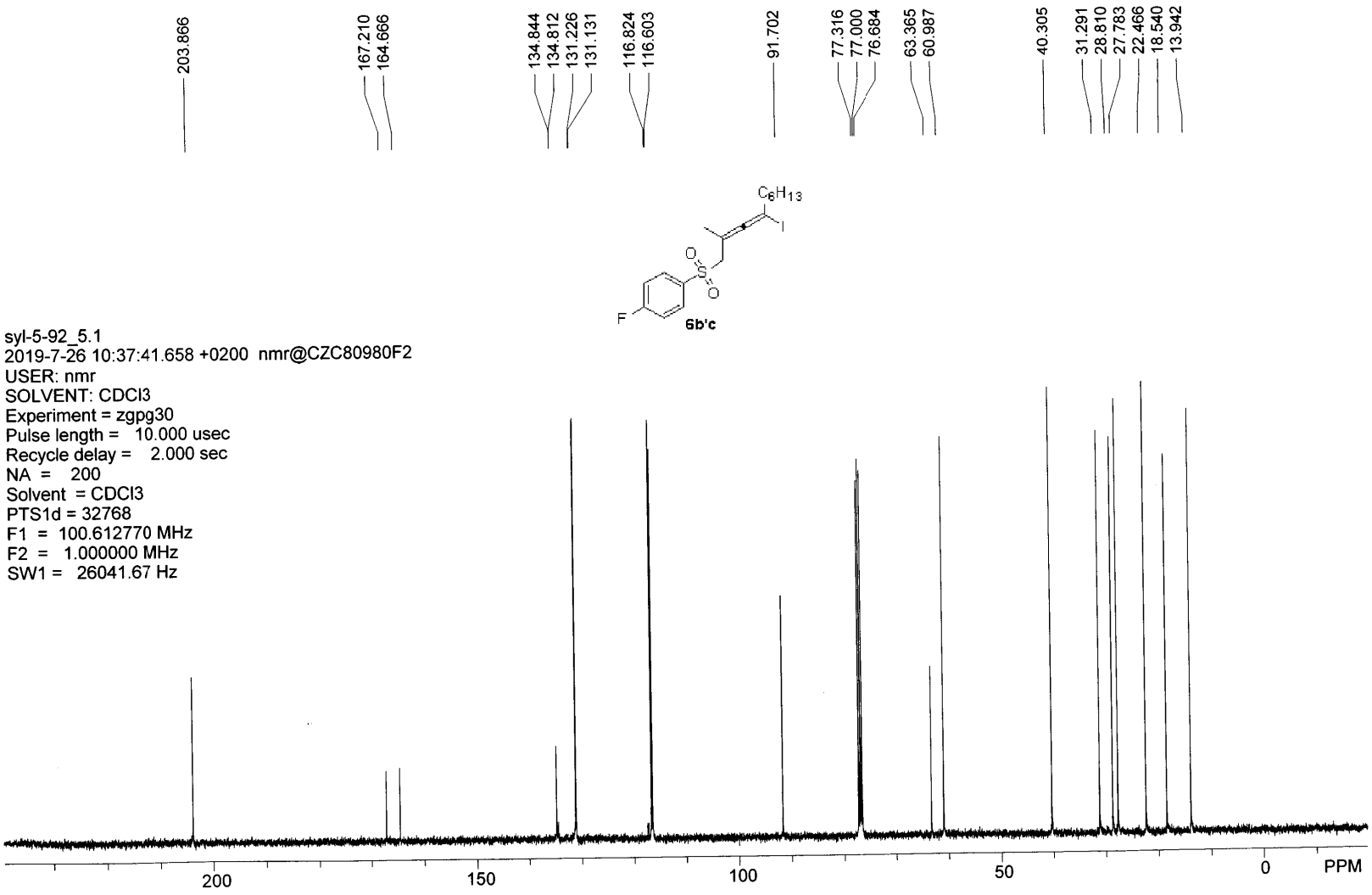


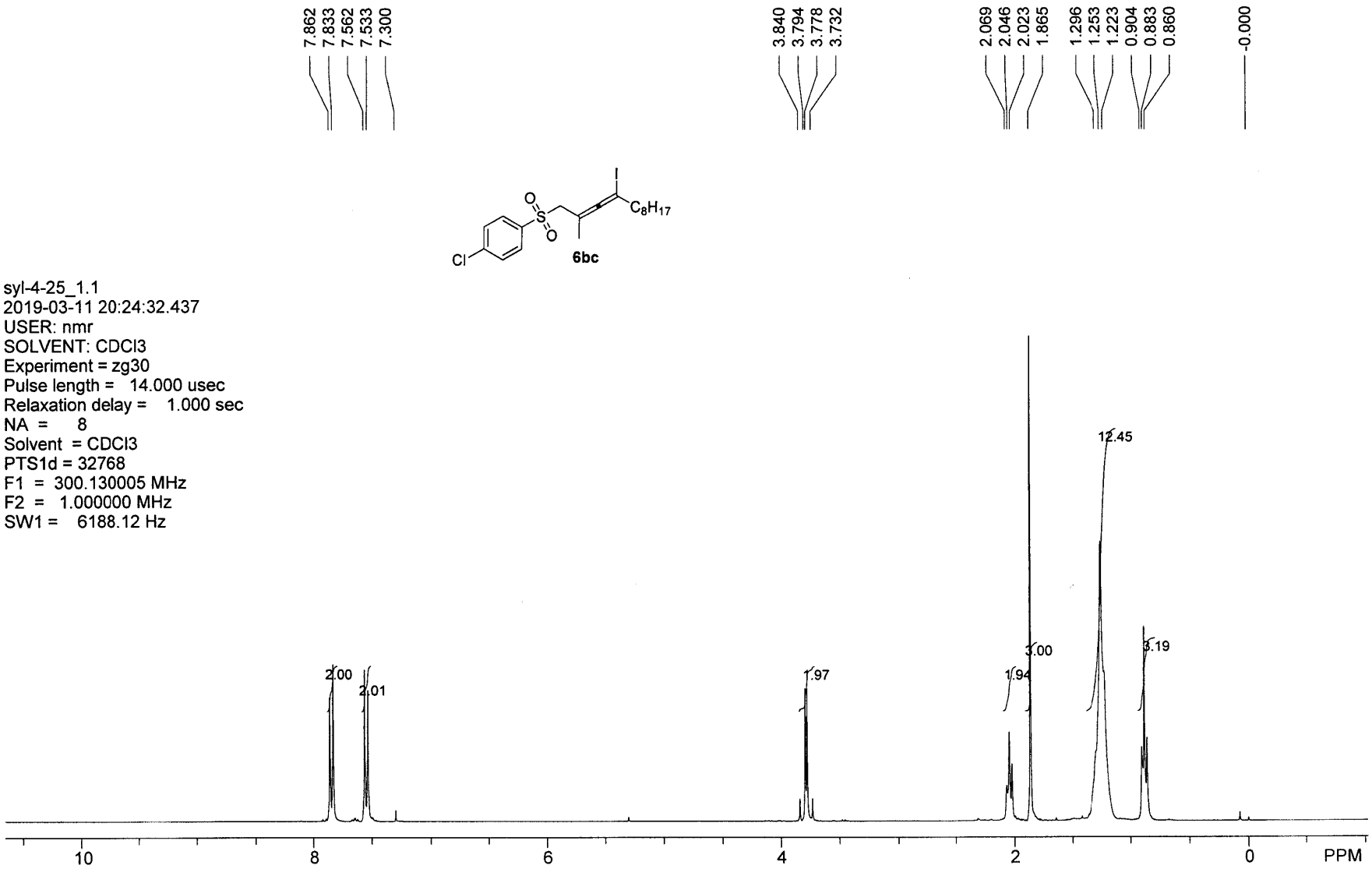
6bb

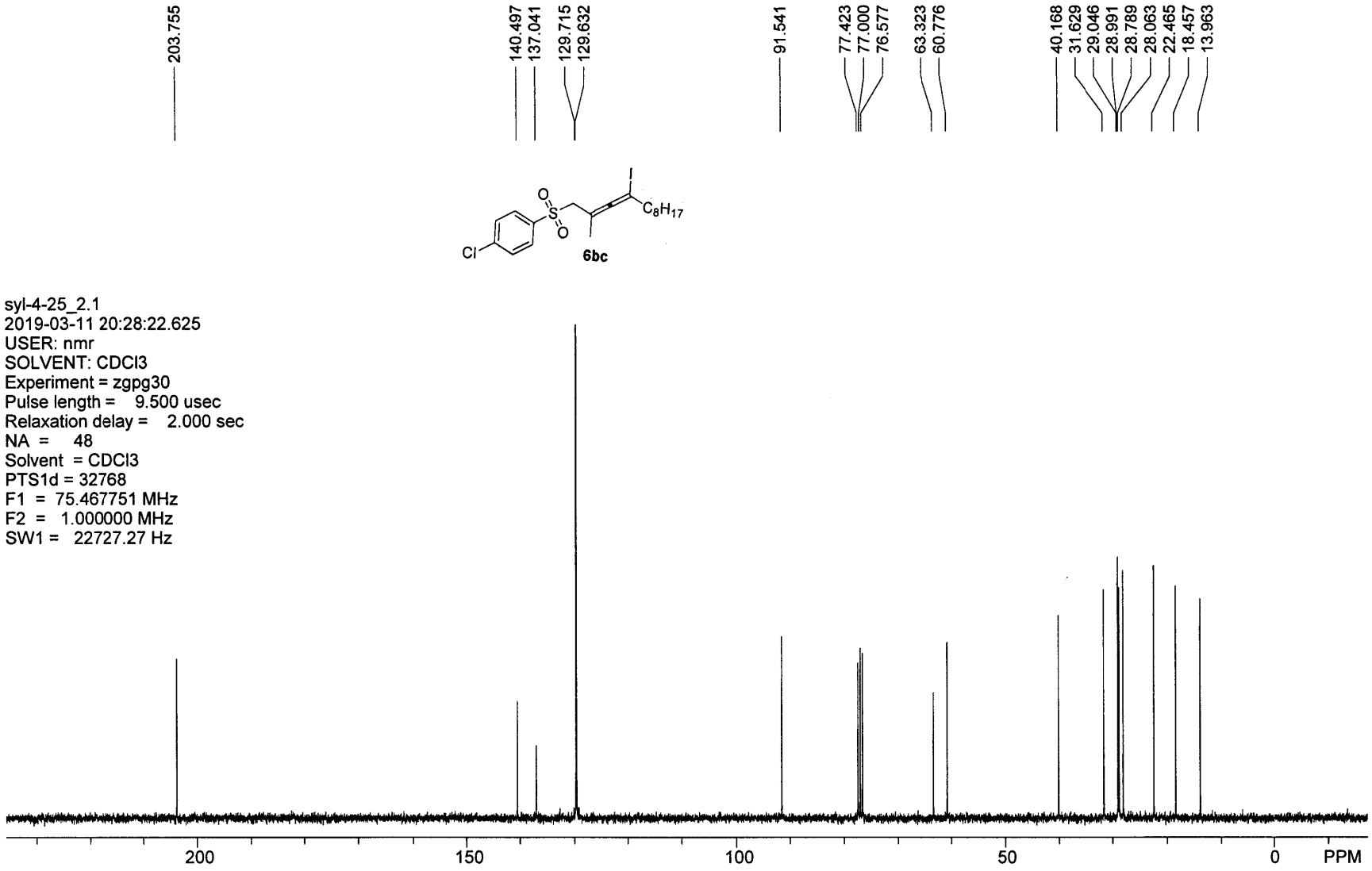
syl-3-194_2.1
2019-02-26 22:13:27.562
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 72
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz

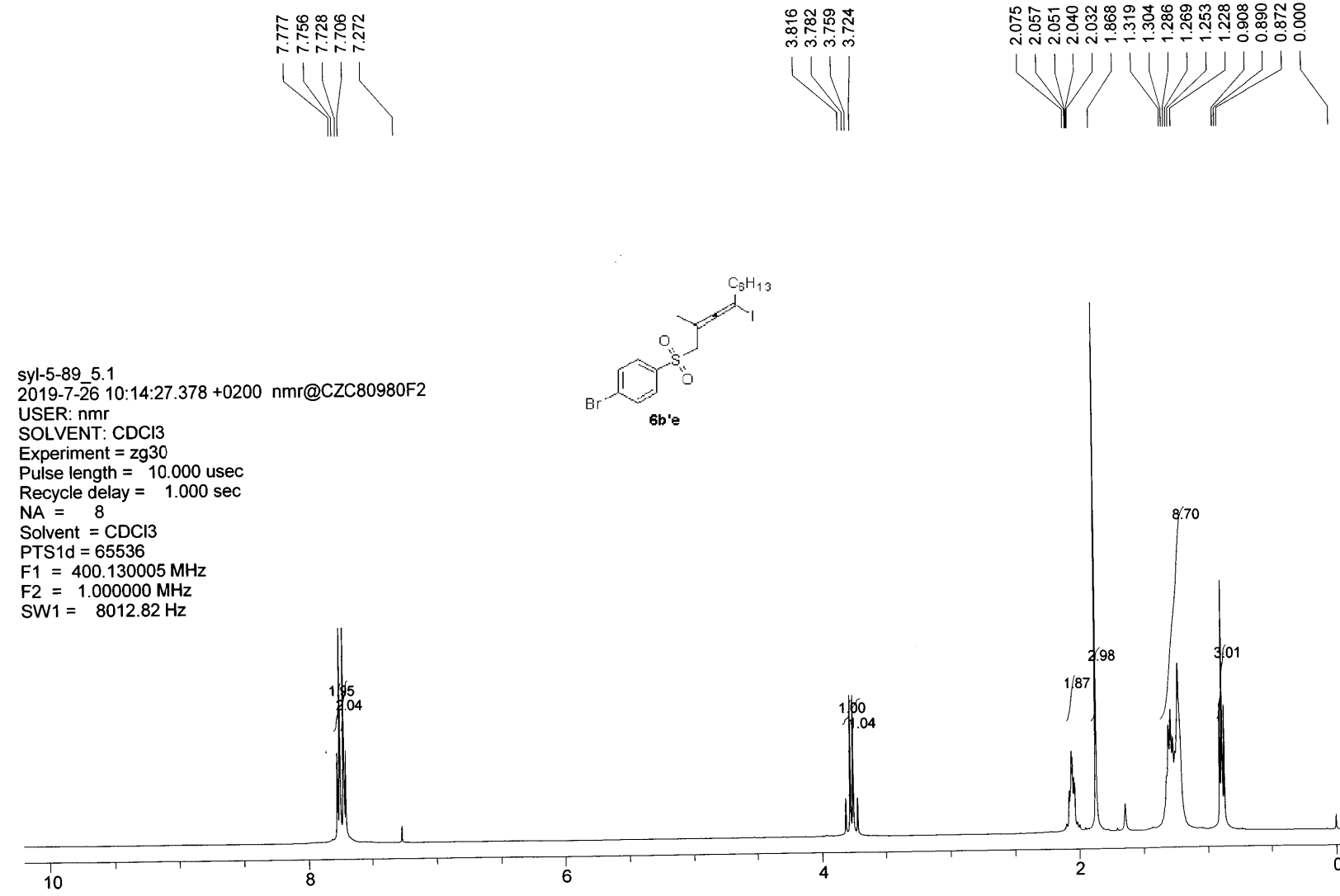


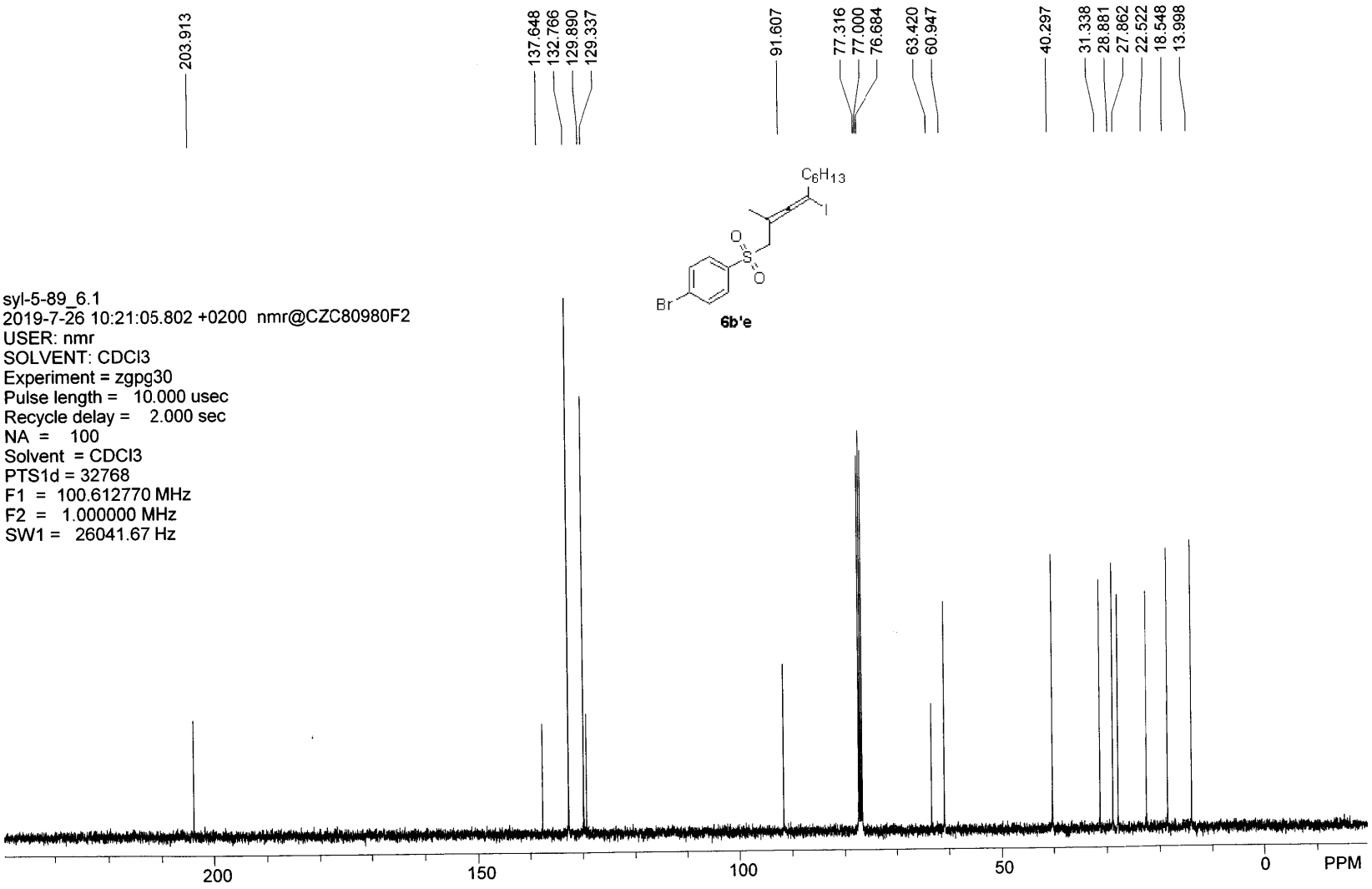

7.947
7.934
7.926
7.913
7.277
7.256
7.234
3.821
3.786
3.771
3.737
2.094
2.077
2.059
1.866
1.314
1.299
1.285
1.269
1.253
1.224
0.905
0.888
0.870
0.000
syl-5-92_4.1
2019-7-26 10:25:46.537 +0200 nmr@CZC80980F2
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 10.000 usec
Recycle delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 65536
F1 = 400.130005 MHz
F2 = 1.000000 MHz
SW1 = 8012.82 Hz
C6H13
I
O
S
O
F
6b'c
1.92
1.91
2.00
1.96
2.91
8.52
3.08
10
8
6
4
2
0PPM

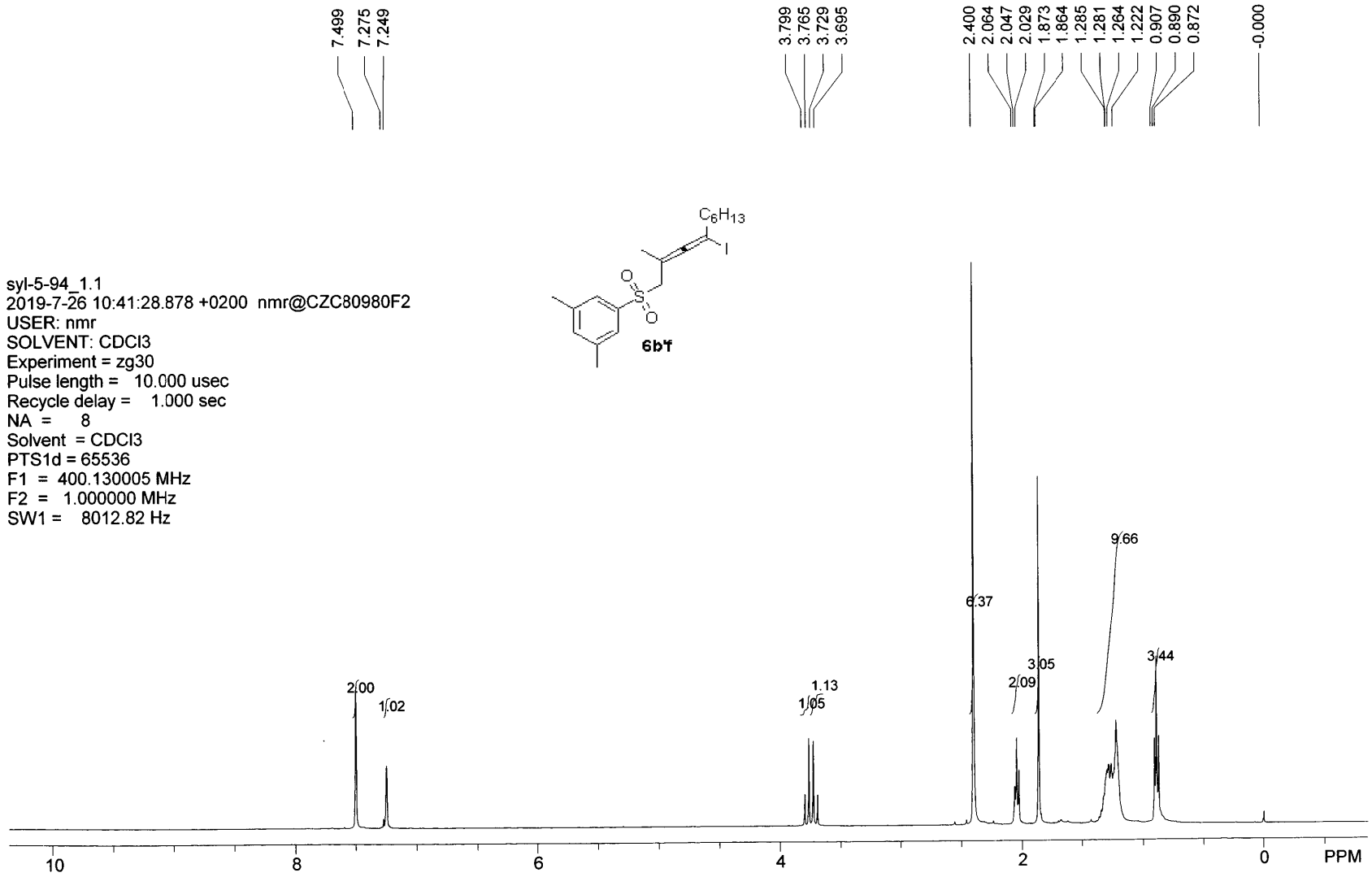

203.866
167.210
164.666
134.844
134.812
131.226
131.131
116.824
116.603
91.702
77.316
77.000
76.684
63.365
60.987
40.305
31.291
28.810
27.783
22.466
18.540
13.942
C6H13
I
O
S
O
F
6b'c
syl-5-92_5.1
2019-7-26 10:37:41.658 +0200 nmr@CZC80980F2
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 10.000 usec
Recycle delay = 2.000 sec
NA = 200
Solvent = CDCl3
PTS1d = 32768
F1 = 100.612770 MHz
F2 = 1.000000 MHz
SW1 = 26041.67 Hz
200
150
100
50
0
PPM

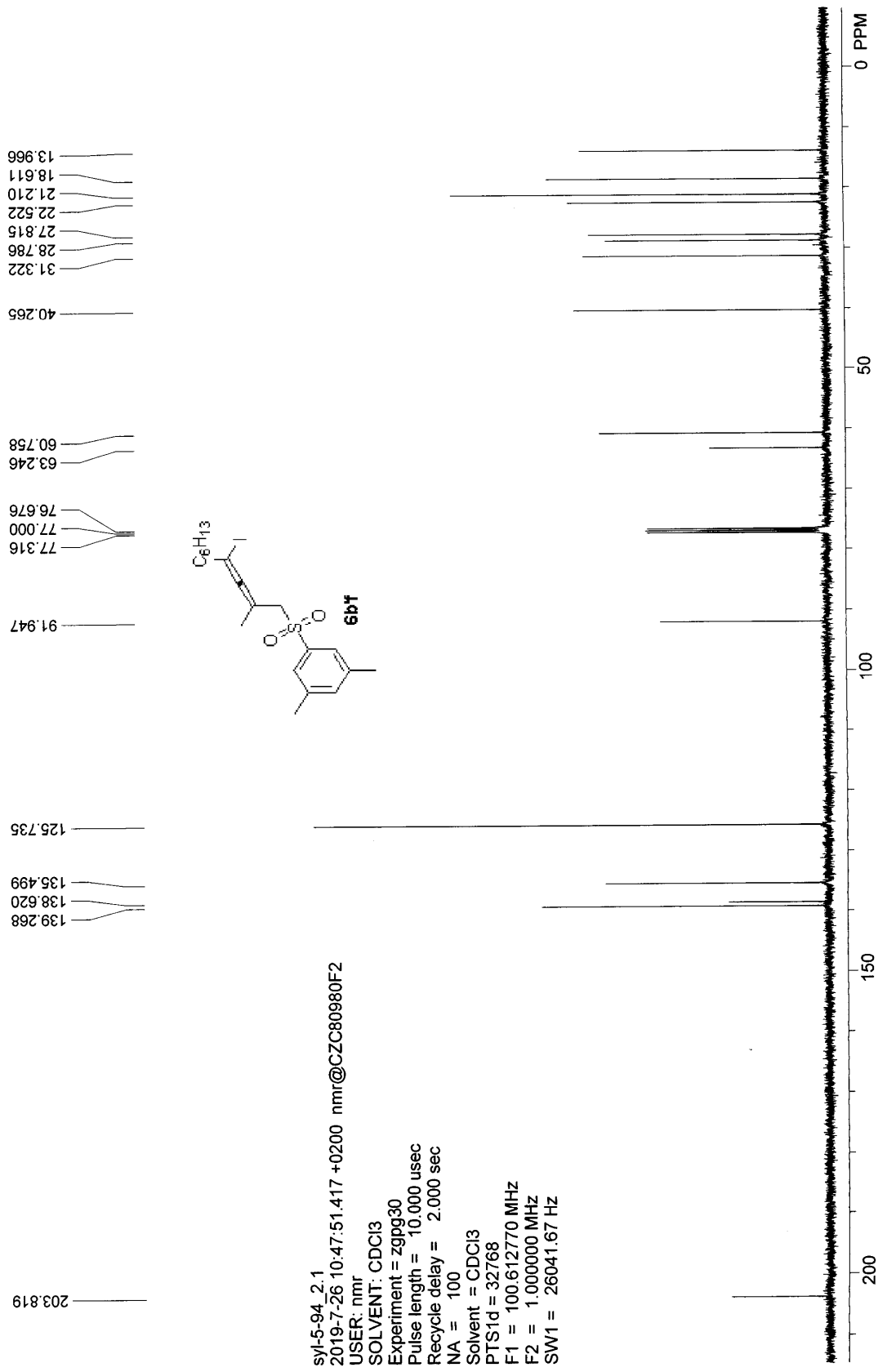

7.862
7.833
7.562
7.533
7.300
3.840
3.794
3.778
3.732
2.069
2.046
2.023
1.865
1.296
1.253
1.223
0.904
0.883
0.860
-0.000
O
S
O
Cl
I
C8H17
6bc
syl-4-25_1.1
2019-03-11 20:24:32.437
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
2.00
2.01
1.97
1.94
3.00
12.45
3.19
10
8
6
4
2
0
PPM

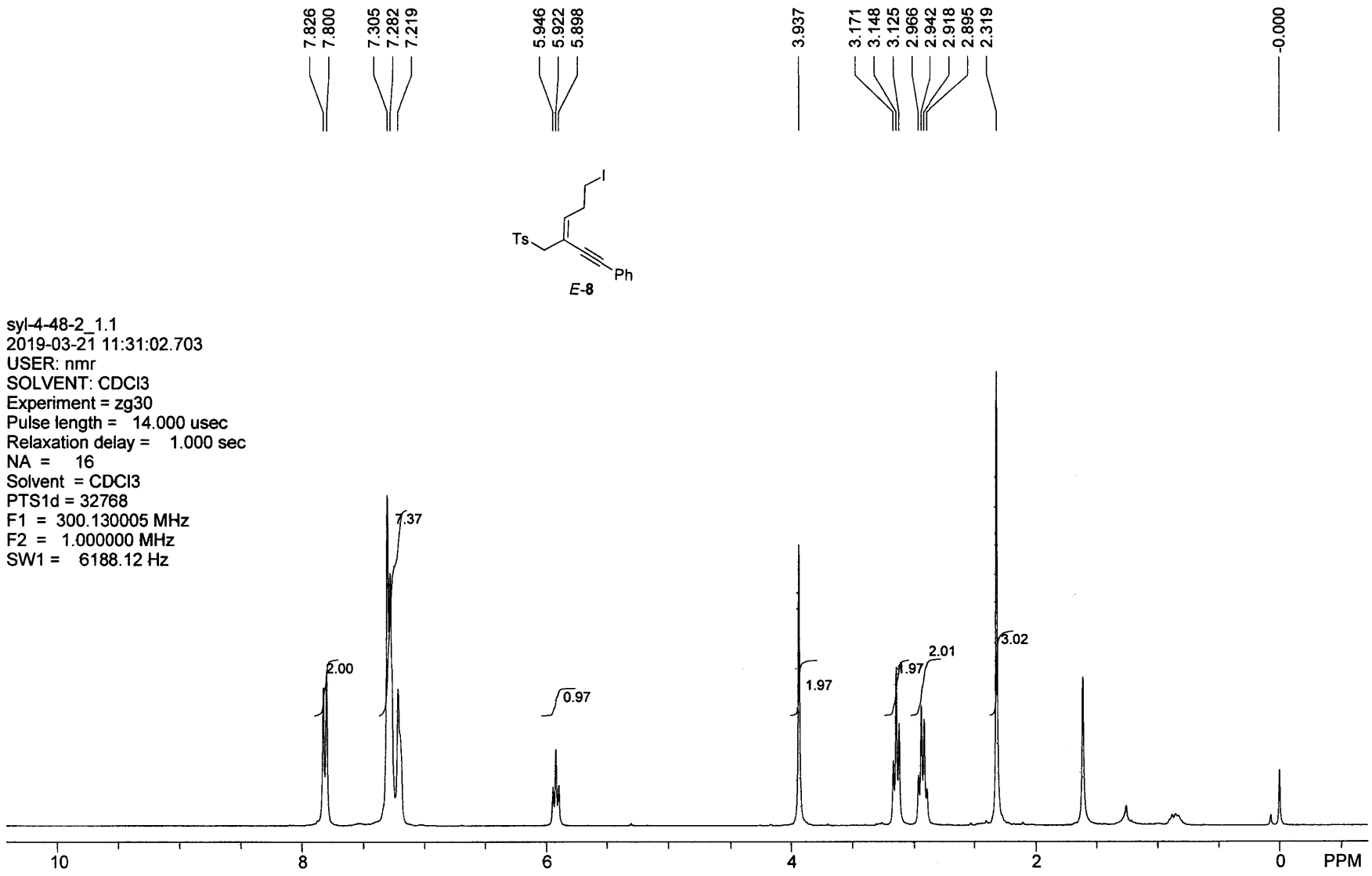

203.755
140.497
137.041
129.715
129.632
91.541
77.423
77.000
76.577
63.323
60.776
40.168
31.629
29.046
28.991
28.789
28.063
22.465
18.457
13.963
6bc
syl-4-25_2.1
2019-03-11 20:28:22.625
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 48
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
200
150
100
50
0
PPM

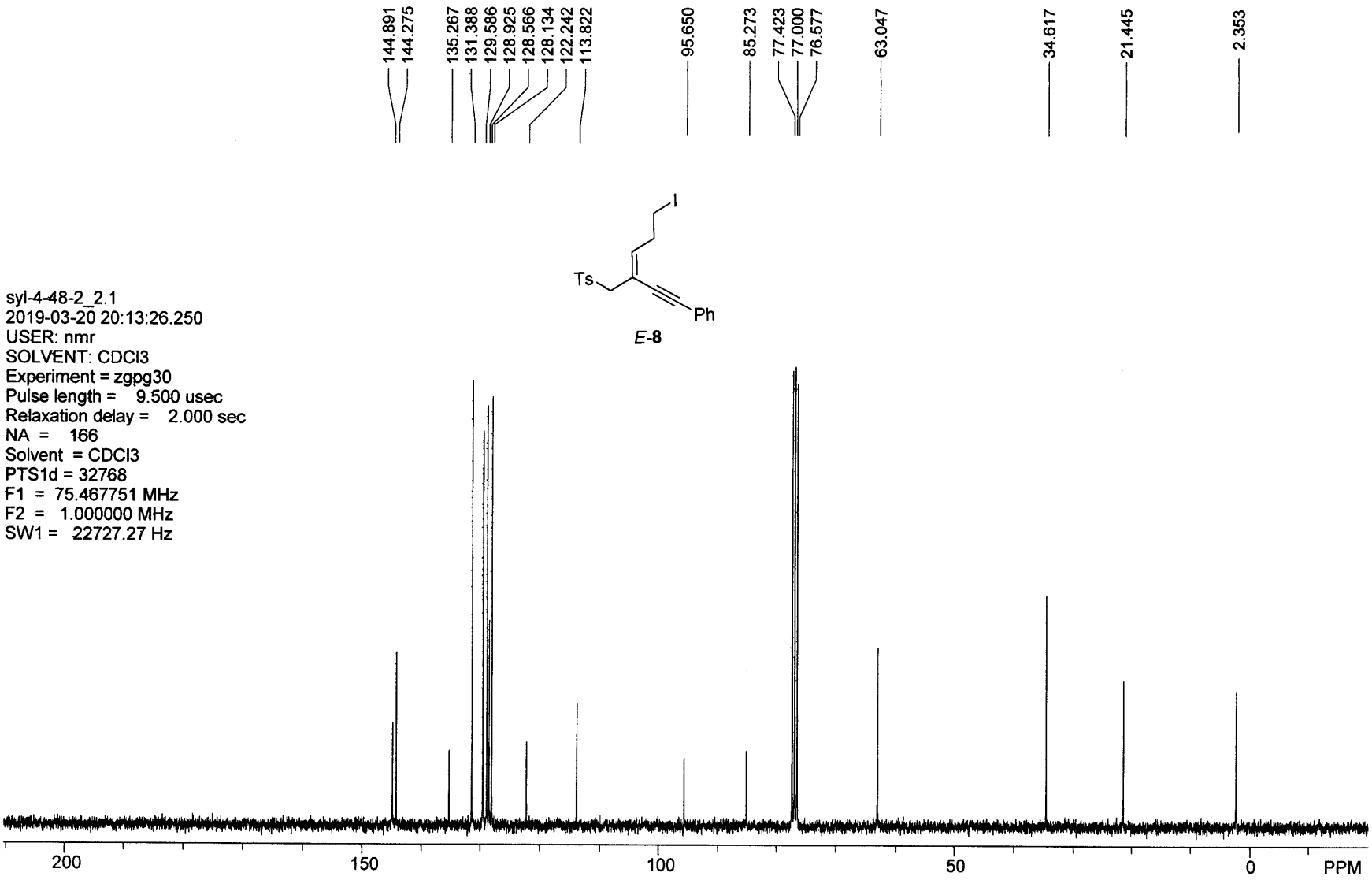

7.777
7.756
7.728
7.706
7.272
3.816
3.782
3.759
3.724
2.075
2.057
2.051
2.040
2.032
1.868
1.319
1.304
1.286
1.269
1.253
1.228
0.908
0.890
0.872
0.000
C8H13
I
O
S
O
Br
6b'e
syl-5-89_5.1
2019-7-26 10:14:27.378 +0200 nmr@CZC80980F2
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 10.000 usec
Recycle delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 65536
F1 = 400.130005 MHz
F2 = 1.000000 MHz
SW1 = 8012.82 Hz
1.95
2.04
1.00
1.04
1.87
2.98
8.70
3.01
10
8
6
4
2
0 PPM

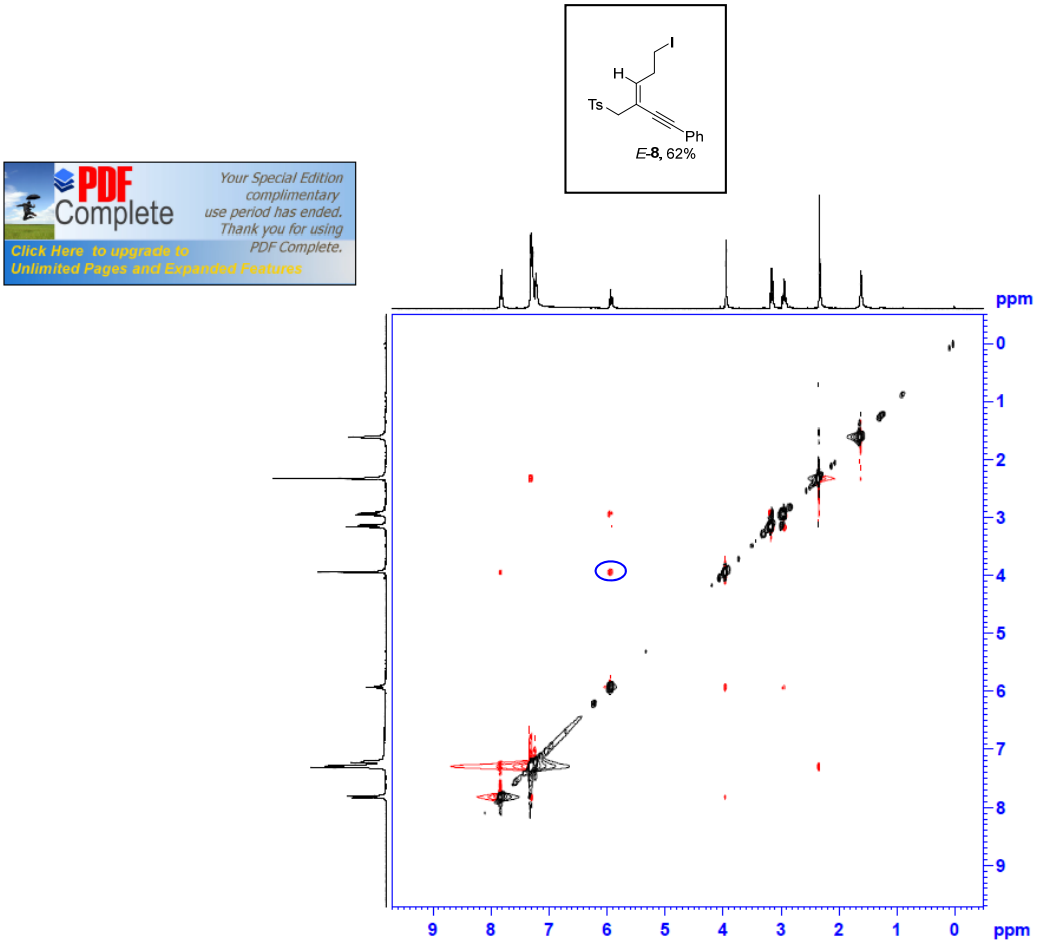

203.913
137.648
132.766
129.890
129.337
91.607
77.316
77.000
76.684
63.420
60.947
40.297
31.338
28.881
27.862
22.522
18.548
13.998
C6H13
I
O
S
O
Br
6b'e
syl-5-89_6.1
2019-7-26 10:21:05.802 +0200 nmr@CZC80980F2
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 10.000 usec
Recycle delay = 2.000 sec
NA = 100
Solvent = CDCl3
PTS1d = 32768
F1 = 100.612770 MHz
F2 = 1.000000 MHz
SW1 = 26041.67 Hz
200
150
100
50
0
PPM

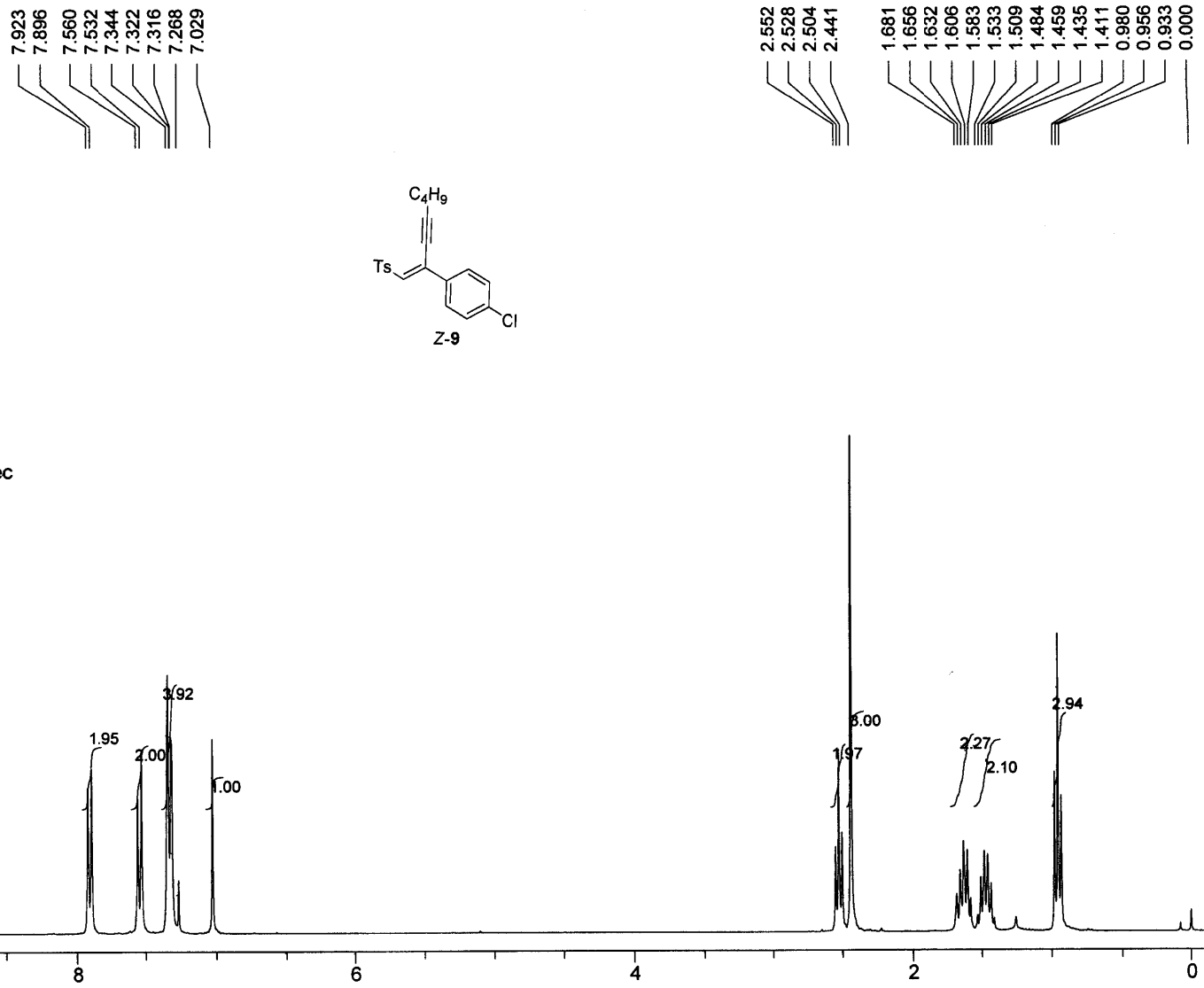

7.499
7.275
7.249
3.799
3.765
3.729
3.695
2.400
2.064
2.047
2.029
1.873
1.864
1.285
1.281
1.264
1.222
0.907
0.890
0.872
-0.000
C6H13
I
O
S
O
6b'f
syl-5-94_1.1
2019-7-26 10:41:28.878 +0200 nmr@CZC80980F2
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 10.000 usec
Recycle delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 65536
F1 = 400.130005 MHz
F2 = 1.000000 MHz
SW1 = 8012.82 Hz
2.00
1.02
1.13
1.05
6.37
2.09
3.05
9.66
3.44
10
8
6
4
2
0
PPM

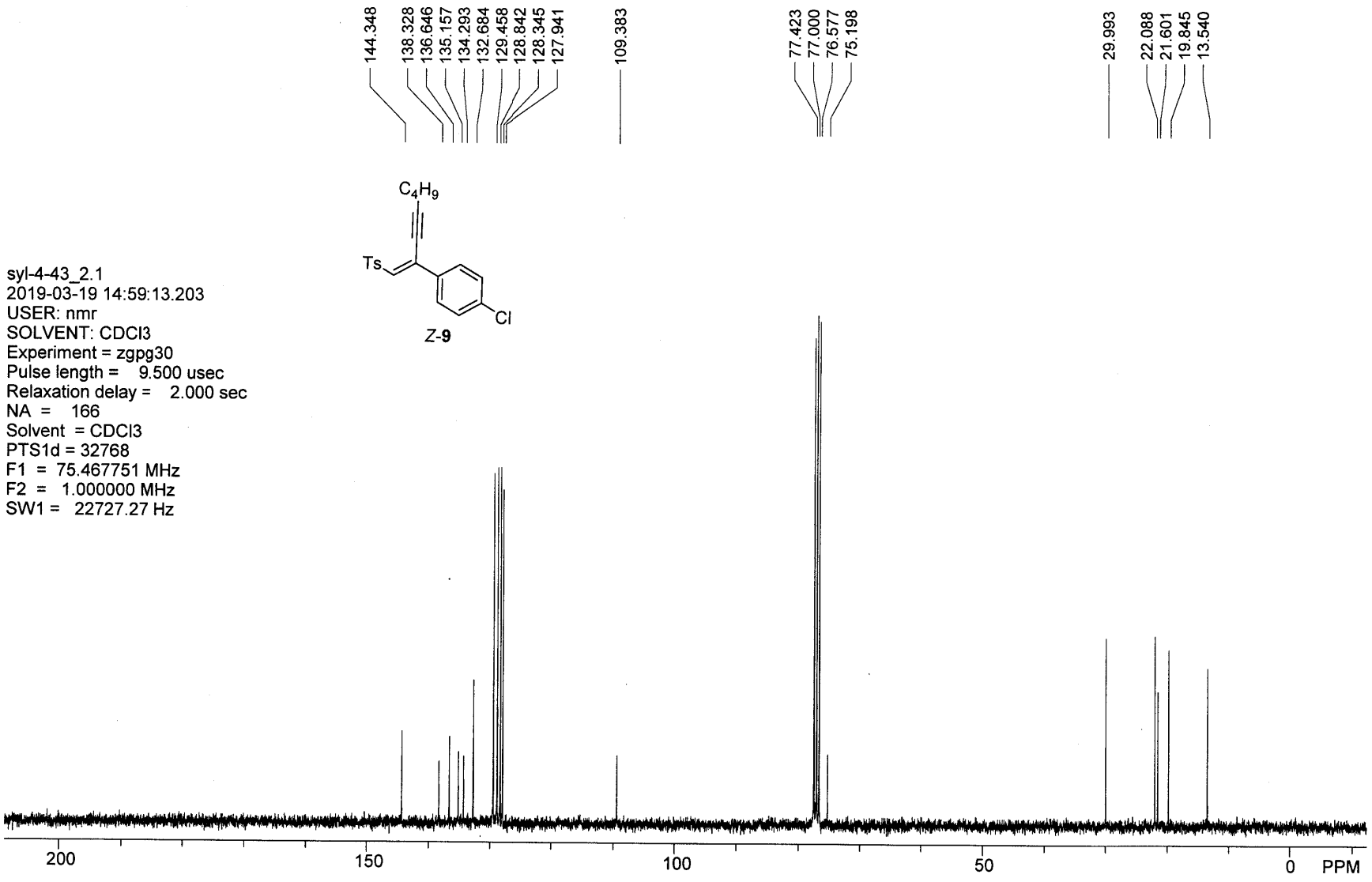

syl-5-94_2.1
2019-7-26 10:47:51.417 +0200 nmr@CZC80980F2
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 10.000 usec
Recycle delay = 2.000 sec
NA = 100
Solvent = CDCl3
PTS1d = 32768
F1 = 100.6122770 MHz
F2 = 1.000000 MHz
SW1 = 26041.67 Hz
1.49
203.819
139.268
138.620
135.499
125.735
91.947
77.316
77.000
76.676
63.246
60.758
40.265
31.322
28.786
27.815
22.522
21.210
18.611
13.966
200
150
100
50
0 PPM

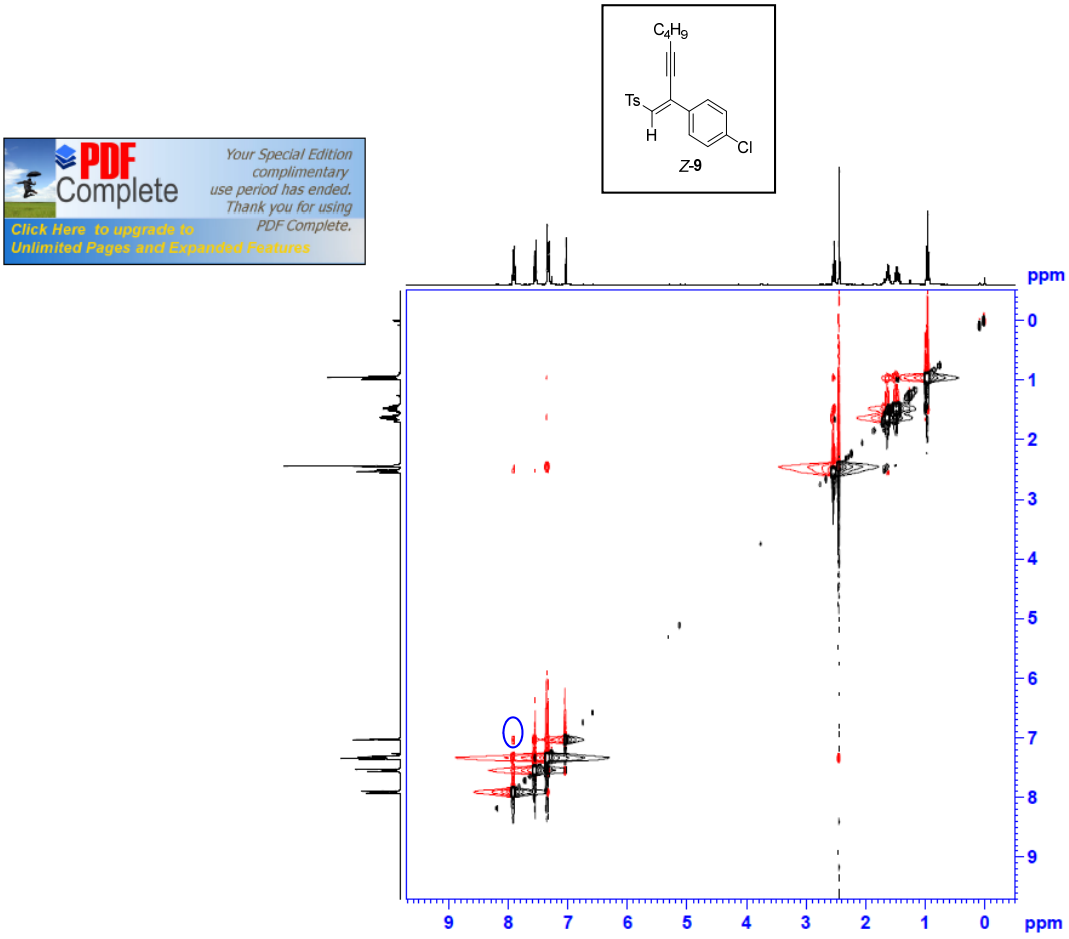

7.826
7.800
7.305
7.282
7.219
5.946
5.922
5.898
3.937
3.171
3.148
3.125
2.966
2.942
2.918
2.895
2.319
-0.000
I
Ts
Ph
E-8
syl-4-48-2_1.1
2019-03-21 11:31:02.703
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 16
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
2.00
7.37
0.97
1.97
1.97
2.01
3.02
10
8
6
4
2
0
PPM

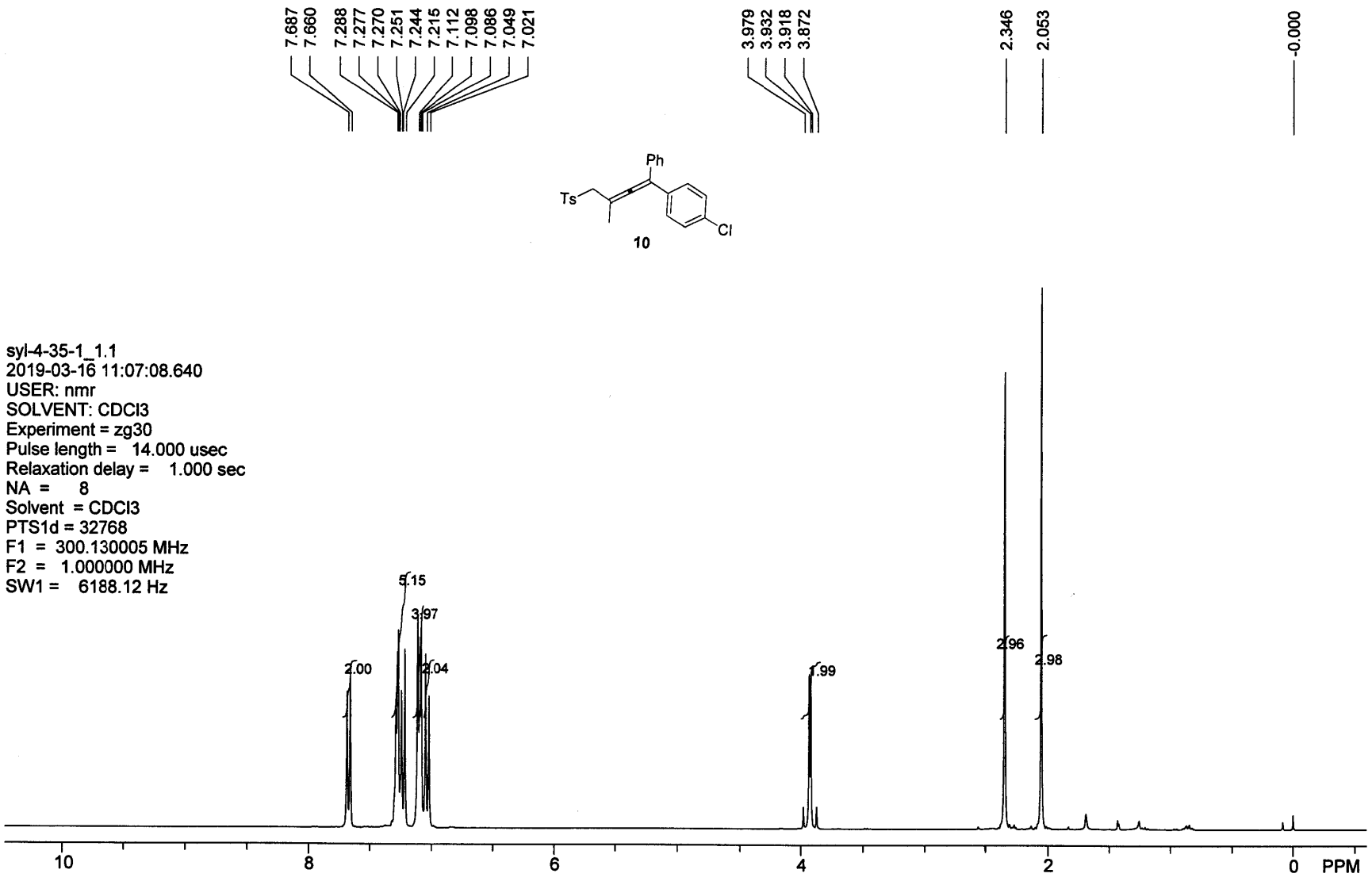

144.891
144.275
135.267
131.388
129.586
128.925
128.566
128.134
122.242
113.822
95.650
85.273
77.423
77.000
76.577
63.047
34.617
21.445
2.353
I
Ts
Ph
E-8
syl-4-48-2_2.1
2019-03-20 20:13:26.250
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 166
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
200
150
100
50
0
PPM

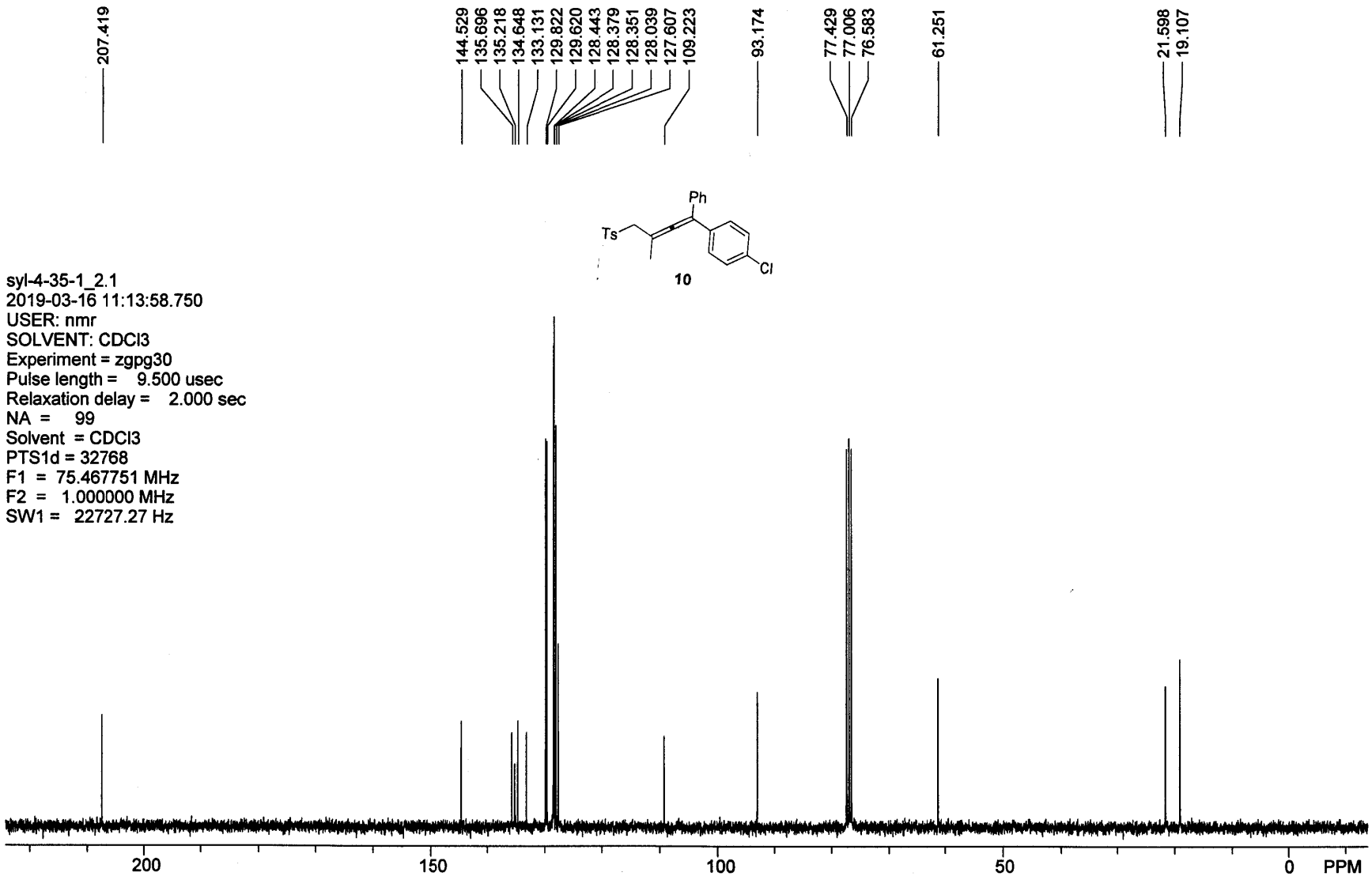


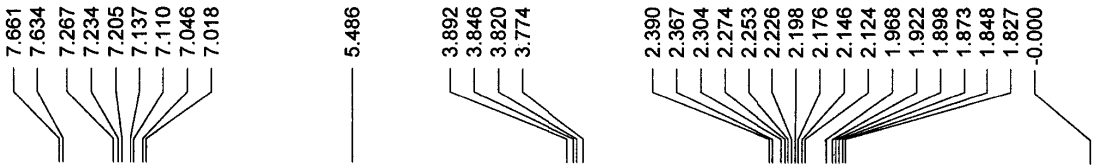

7.923
7.896
7.560
7.532
7.344
7.322
7.316
7.268
7.029
2.552
2.528
2.504
2.441
1.681
1.656
1.632
1.606
1.583
1.533
1.509
1.484
1.459
1.435
1.411
0.980
0.956
0.933
0.000
C4H9
Ts
Cl
Z-9
syl-4-43_1.1
2019-03-19 14:48:27.796
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
1.95
2.00
2.92
1.00
1.97
6.00
2.27
2.10
2.94
10
8
6
4
2
0
PPM

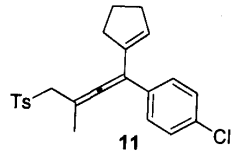

144.348
138.328
136.646
135.157
134.293
132.684
129.458
128.842
128.345
127.941
109.383
77.423
77.000
76.577
75.198
29.993
22.088
21.601
19.845
13.540
C4H9
Ts
Cl
Z-9
syl-4-43_2.1
2019-03-19 14:59:13.203
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 166
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
200
150
100
50
0
PPM



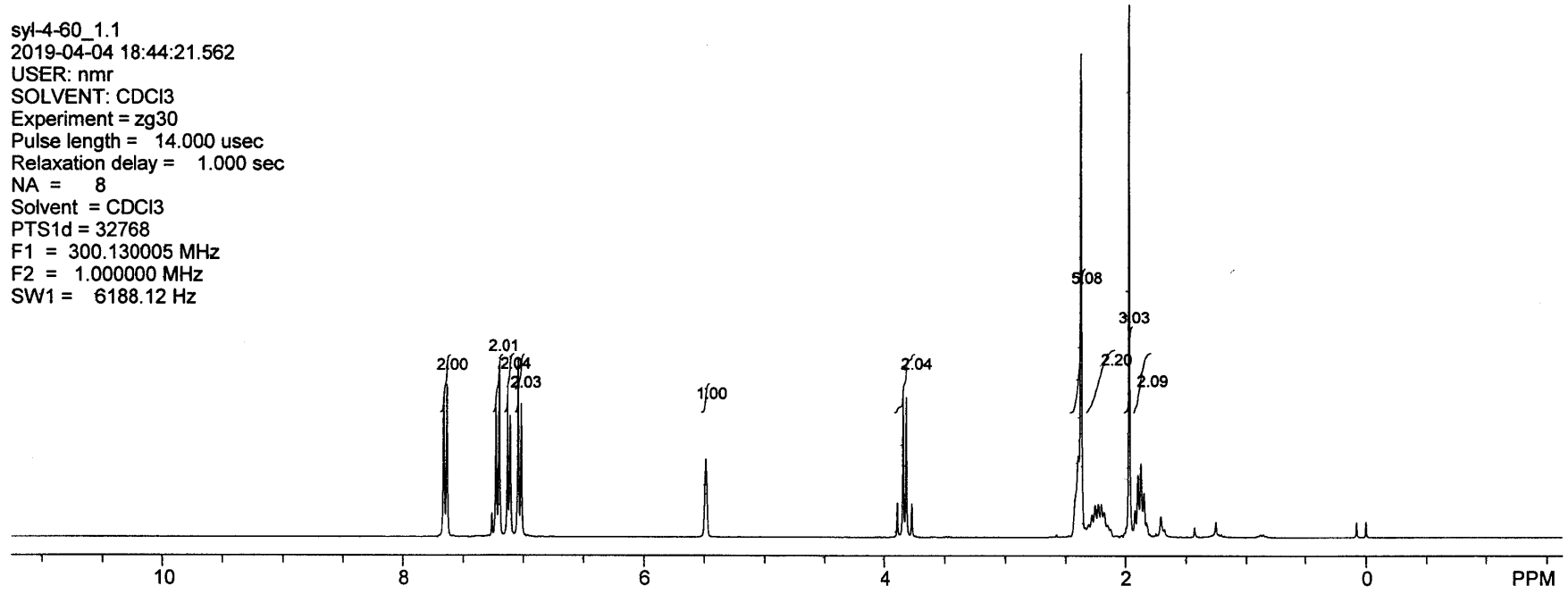

7.687
7.660
7.288
7.277
7.270
7.251
7.244
7.215
7.112
7.098
7.086
7.049
7.021
3.979
3.932
3.918
3.872
2.346
2.053
-0.000
Ph
Ts
Cl
10
syl-4-35-1_1.1
2019-03-16 11:07:08.640
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
5.15
3.97
2.00
2.04
1.99
2.96
2.98
10
8
6
4
2
0
PPM


207.419
144.529
135.696
135.218
134.648
133.131
129.822
129.620
128.443
128.379
128.351
128.039
127.607
109.223
93.174
77.429
77.006
76.583
61.251
21.598
19.107
Ph
Ts
Cl
10
syl-4-35-1_2.1
2019-03-16 11:13:58.750
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 99
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
200
150
100
50
0
PPM

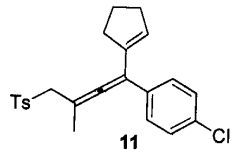

7.661
7.634
7.267
7.234
7.205
7.137
7.110
7.046
7.018
5.486
3.892
3.846
3.820
3.774
2.390
2.367
2.304
2.274
2.253
2.226
2.198
2.176
2.146
2.124
1.968
1.922
1.898
1.873
1.848
1.827
-0.000


Ts
Cl

11


syl-4-60_1.1
2019-04-04 18:44:21.562
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
2.00
2.01
2.04
2.03
1.00
2.04
5.08
3.03
2.20
2.09
10
8
6
4
2
0
PPM

208.063
144.538
138.389
135.273
134.777
132.800
130.291
129.997
129.537
128.112
106.365
91.658
77.429
77.006
76.583
61.628
34.053
33.519
22.986
21.570
19.171



11

syl-4-60_2.1
2019-04-04 18:23:51.593
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 94
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz