Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Rh-Catalyzed Oxidative Homo-coupling Cyclization of 2,3-Allenols to Conjugated Furylenones

Xiaoyan Wu , Junjie Fan, Xin Huang and Shengming Ma*

Laboratory of Molecular Recognition and Synthesis, Department of Chemistry, Zhejiang University, Hangzhou 310027, Zhejiang, P. R. China.

E-mail: masm@sioc.ac.cn

Supporting Information

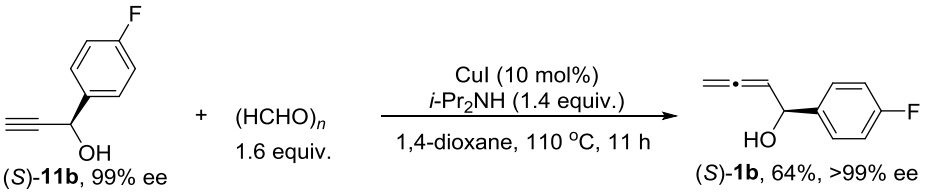
Table of Contents

General Information

^{1}H NMR, ^{13}C NMR, and ^{19}F NMR spectra were recorded in $CDCl_3$ using a Bruker AM 300 MHz NMR spectrometer (^{1}H at 300 MHz, ^{13}C at 75 MHz, ^{19}F at 282 MHz). All ^{1}H NMR spectra were measured with TMS (0 ppm) in $CDCl_3$. All ^{19}F NMR spectra were measured with $CFCl_3$ (0 ppm) as the internal standard, respectively. All ^{13}C NMR spectra were recorded in relative to the signal of $CDCl_3$ (77.0 ppm). IR spectra were recorded with a Perkin–Elmer 983G instrument. Elemental analyses were conducted 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. $[Cp^*RhCl_2]_2$ was purchased from *Strem* and *HWRK CHEM*. The range of boiling point of the petroleum ether used for chromatography was 60-90 ^{o}C unless noted otherwise. Other commercially available chemicals were purchased and used without additional purification unless noted otherwise. Optically active propargylic alcohols (*R*)-**3** or (*S*)-**3** were prepared via Novozym-435-catalyzed enzymatic kinetic resolution.[1] Optically active or racemic 2,3-allenols were prepared according to the literature procedures.[2]

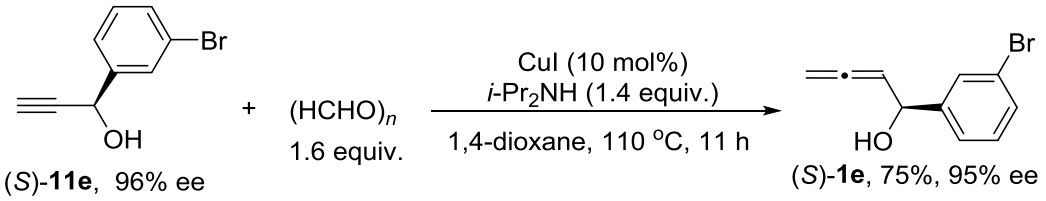
Synthesis of New Starting Materials

1. Synthesis of (*S*)-1-(4-fluorophenyl)buta-2,3-dien-1-ol (*S*)-**1b.**[2] (wxy-3-040)



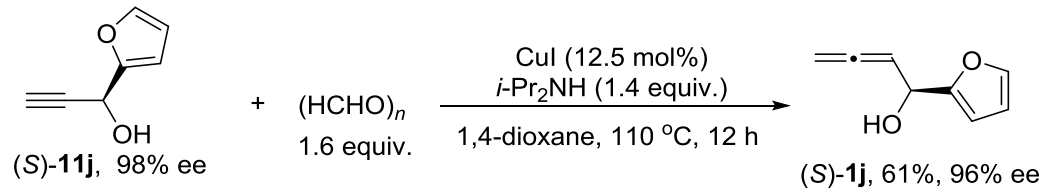
Typical Procedure I： To a dried three-necked flask were added CuI (158.6 mg, 0.8 mmol), paraformaldehyde (1.1535 g, 12.8 mmol), dioxane (8.0 mL), *i*-Pr_2NH (1.6 mL, d = 0.716 g/mL, 1.1456 g, 11.3 mmol), (*S*)-**11b** (1.2014 g, 8.0 mmol, 99% ee) and dioxane (4.0 mL) sequentially. The reaction tube was then equipped with a condenser and put into an oil bath preheated 110 ^{o}C. The reaction was complete after stirring for 11 hours as monitored by TLC. The resulting mixture was cooled to room temperature and filtered through a short column of silica gel (eluent: Et_2O (50 mL × 3). After evaporation of the solvent, the crude residue was purified via chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 20/1, 1000 mL) to afford (*S*)-**1b** (842.1 mg, 64%) as a liquid: >99% ee (HPLC conditions: Chiralcel OD-H column, *n*-hexane/*i*-PrOH = 90/10, 0.5 mL/min, λ = 254 nm, t_R(minor) = 10.7 min, t_R(major) = 11.4 min); $[\alpha]_D^{20}$ = +36.9 (*c* = 1.43, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.41-7.30 (m, 2 H, ArH), 7.03 (t, *J* = 8.9 Hz, 2 H, ArH), 5.40 (q, *J* = 6.5 Hz, 1 H, =CH), 5.29-5.20 (m, 1 H, OCH), 4.99-4.86 (m, 2 H, $=CH_2$), 2.37 (s, 1 H, OH); ^{13}C NMR (75 MHz, $CDCl_3$) δ 207.1, 162.3 (d, *J* = 244.7 Hz), 138.5 (d, *J* = 3.5 Hz), 127.8 (d, *J* = 8.3 Hz), 115.3 (d, *J* = 21.4 Hz), 95.1, 78.3, 71.3; ^{19}F NMR (282 MHz, $CDCl_3$) δ -115.2; IR (neat) *v* (cm^{-1}) 3383, 3067, 2994, 2890, 1955, 1604, 1511, 1416, 1224, 1157, 1035, 1014; MS (EI): *m/z* (%) 164 (M^+, 1.23), 125 (100); HRMS Calcd for $C_{10}H_9FO$ (M^+): 164.0637; Found: 164.0635.

2. Synthesis of (*S*)-1-(3-bromophenyl)buta-2,3-dien-1-ol (*S*)-**1e.**[2] (wxy-3-104)



Following **Typical Procedure I**, the reaction of CuI (80.1 mg, 0.4 mmol), (*S*)-**11e** (723.6 mg, 3.4 mmol, 96% ee), paraformaldehyde (490.1 mg, 5.4 mmol), and *i*-Pr_2NH (0.68 mL, d = 0.716 g/mL, 486.9 mg, 4.8 mmol) in dioxane (1.7 mL) afforded (*S*)-**1e** (576.4 mg, 75%) as an oil: 95% ee (HPLC conditions: Chiralcel OD-H column, *n*-hexane/*i*-PrOH = 95/5, 0.5 mL/min, λ = 254 nm, t_R(minor) = 10.3 min, t_R(major) = 11.4 min); $[\alpha]_D^{20}$ = +38.5 (*c* = 1.40, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.52 (t, *J* = 1.7 Hz, 1 H, ArH), 7.39 (dt, J_1 = 7.8 Hz, J_2 = 1.7 Hz, 1 H, ArH), 7.32-7.24 (m, 1 H, ArH), 7.19 (t, *J* = 7.7 Hz, 1 H, ArH), 5.36 (q, *J* = 6.5 Hz, 1 H, OCH), 5.23-5.13 (m, 1 H, =CH), 4.98-4.85 (m, 2 H, $=CH_2$), 2.76 (d, *J* = 4.2 Hz, 1 H, OH); ^{13}C NMR (75 MHz, $CDCl_3$) δ 207.1, 144.9, 130.7, 130.0, 129.1, 124.6, 122.4, 94.6, 78.4, 71.2; IR (neat) *v* (cm^{-1}) 3389, 3062, 2876, 1954, 1594, 1570, 1474, 1427, 1186, 1093, 1071, 1034; MS (EI): *m/z* (%) 225 [($M(^{81}Br)^+$, 2.39], 223 [($M(^{79}Br)^+$, 2.66], 77 (100); HRMS Calcd for $C_{10}H_8{}^{79}BrO$ $(M-H)^-$: 222.9764; Found: 222.9764.

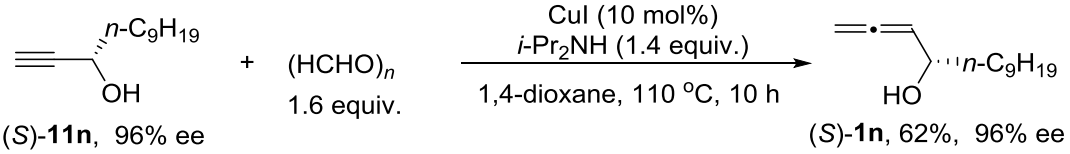
3. Synthesis of (*S*)-1-(furan-2-yl)buta-2,3-dien-1-ol (*S*)-**1j.**[2] (wxy-3-114)



Following **Typical Procedure I**, the reaction of CuI (119.2 mg, 0.6 mmol), (*S*)-**11j** (611.0

mg, 5.0 mmol, 96% ee), paraformaldehyde (723.8 mg, 8.0 mmol), and *i*-Pr_2NH (1.0 mL, d = 0.716 g/mL, 716.0 mg, 7.1 mmol) in dioxane (7.5 mL) afforded (*S*)-**1j** (432.6 mg, 61%, 96% purity) (eluent: petroleum ether/ethyl acetate = 25/1 (800 mL) to petroleum ether/ethyl acetate = 10/1 (500 mL)] as a liquid: 96% ee (HPLC conditions: Chiralcel OD-H column, *n*-hexane/*i*-PrOH = 90/10, 1.0 mL/min, λ = 220 nm, t_R(minor) = 7.3 min, t_R(major) = 8.8 min); $[\alpha]_D^{20}$ = + 28.1 (*c* = 1.05, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.39 (dd, J_1 = 1.8 Hz, J_2 = 0.9 Hz, 1 H, ArH), 6.34 (dd, J_1 = 3.0 Hz, J_2 = 1.8 Hz, 1 H, ArH), 6.29 (d, *J* = 3.3 Hz, 1 H, ArH), 5.52 (q, *J* = 6.6 Hz, 1 H, OCH), 5.32-5.20 (m, 1 H, =CH), 5.03-4.90 (m, 2 H, $=CH_2$), 2.49 (s, 1 H, OH); ^{13}C NMR (75 MHz, $CDCl_3$) δ 207.5, 154.9, 142.1, 110.0, 106.4, 91.9, 78.0, 65.5; IR (neat) *v* (cm^{-1}) 3390, 3120, 2991, 2900, 1957, 1713, 1601, 1504, 1374, 1290, 1223, 1181, 1146, 1116, 1074, 1010; MS (EI): *m/z* (%) 136 (M^+, 8.50), 97 (100); HRMS Calcd for $C_8H_8NaO_2$ (M^++Na): 159.0422; Found: 159.0415.

4. Synthesis of (*S*)-trideca-1,2-dien-4-ol (*S*)-**1n.**[2] (wxy-3-042)



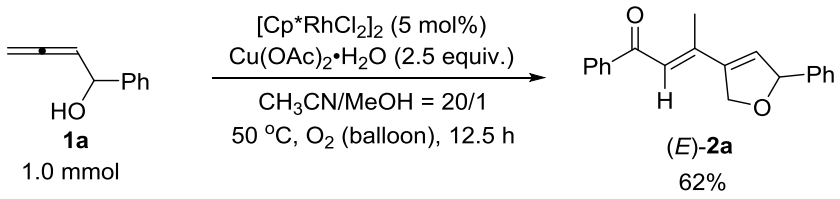
Following **Typical Procedure I**, the reaction of CuI (114.5 mg, 0.6 mmol), (*S*)-**11n** (1.0831 g, 6 mmol, 96% ee), paraformaldehyde (864.9 mg, 9.6 mmol), and *i*-Pr_2NH (1.2 mL, d = 0.716 g/mL, 859.2 mg, 8.5 mmol) in dioxane (9 mL) afforded (*S*)-**1n** (718.4 mg, 62%) (eluent: petroleum ether/ethyl acetate = 40/1, 800 mL) as a liquid; 96% ee (GC conditions: restek Rt-bdex (30*0.25*0.25), carrier N_2; 10.0 psi; injector 300 °C; detector (FID, H_2, 0.4 MPa), 250 °C; t_R(major) = 108.5 min, t_R(minor) = 109.2 min,) $[\alpha]_D^{20}$ = +3.7 (*c* = 1.80, $CHCl_3$); ^{1}H

NMR (300 MHz, $CDCl_3$) δ 5.22 (q, *J* = 6.2 Hz, 1 H, =CH), 4.87-4.75 (m, 2 H, $=CH_2$), 4.20-4.09 (m, 1 H, CH), 2.58-2.38 (m, 1 H, OH), 1.66-1.49 (m, 2 H, CH_2), 1.49-1.18 (m, 14 H, CH_2 × 7), 0.88 (t, *J* = 6.8 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 207.0, 94.7, 76.9, 69.7, 37.3, 31.8, 29.52, 29.47, 29.4, 29.2, 25.3, 22.6, 14.0; IR (neat) *v* (cm^{-1}) 3361, 2954, 2925, 2855, 1956, 1467, 1061, 1011; MS (EI): *m/z* (%) 157 [$(M-(CH_2=C=CH))^+$, 9.82], 69 (100); Anal. Calcd. for $C_{13}H_{24}O$ (%): C, 79.53; H, 12.32; Found: C, 79.09; H, 12.28.

Rh(III)-Catalyzed Oxidative Homo-coupling Cyclization of 2,3-Allenols

1. Synthesis of (*E*)-1-phenyl-3-(2-phenyl-2,5-dihydrofuran-4-yl)but-2-en-1-one (*E*)-**2a**.

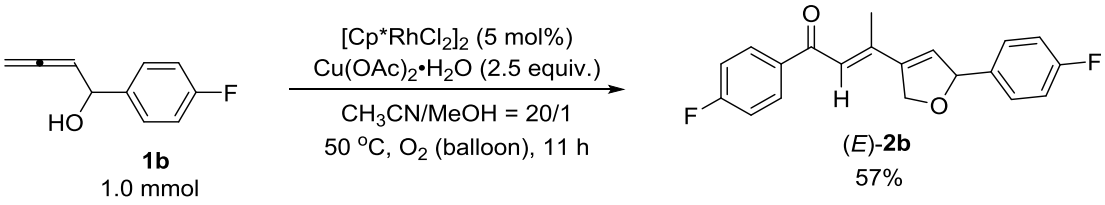
(hx-15-188)



Typical Procedure II: To a dried Schlenk tube were added $[Cp^*RhCl_2]_2$ (31.1 mg, 0.05 mmol) and $Cu(OAc)_2•H_2O$ (499.5 mg, 2.5 mmol) under air atmosphere. The Schlenk tube was then degassed to remove the air inside completely and refilled with O_2 by a balloon of O_2 for three times. After **1a** (146.4 mg, 1 mmol)/CH_3CN (1.0 mL) and MeOH (50 μL) were added sequentially, the reaction tube was put into an oil bath pre-heated at 50 ^oC. The reaction was complete after stirring for 12.5 h as monitored by TLC. After removing the O_2 balloon, the resulting mixture was diluted with ethyl acetate (15 mL) and filtered through a short column of silica gel eluted with ethyl acetate (20 mL × 2). The combined filtrate was then concentrated in vacuo and the crude residue was purified via chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 15/1, 600 mL) to afford (*E*)-**2a** (90.6 mg, 62%): liquid; ^1H NMR (300 MHz, $CDCl_3$) δ 7.97-7.87 (m, 2 H, ArH), 7.58-7.51 (m, 1 H, ArH), 7.50-7.42 (m, 2 H, ArH), 7.40-7.25 (m, 5 H, ArH), 6.56 (s, 1 H, =CH), 6.32 (q, J = 1.8 Hz, 1 H, =CH), 5.99-5.90 (m, 1 H, OCH), 5.15 (ddd, J_1 = 11.4 Hz, J_2 = 5.4 Hz, J_3 = 1.8 Hz, 1 H, one proton of OCH_2), 5.02 (ddd, J_1 = 11.4 Hz, J_2 = 5.4 Hz, J_3 = 2.1 Hz, 1 H, one proton of OCH_2), 2.36 (d, J = 1.2 Hz, 3 H, Me); ^{13}C NMR (75 MHz, $CDCl_3$) δ 191.8, 144.9, 141.4, 141.0, 138.8, 132.8, 132.6, 128.6, 128.5, 128.1, 128.0, 126.3, 122.1, 88.9, 74.8, 16.6; IR (neat) ν (cm^{-1}) 3080, 3061, 3029, 2951, 2916, 2849, 1657, 1594, 1585, 1492, 1448, 1403, 1364,

1345, 1295, 1243, 1217, 1178, 1096, 1073, 1048, 1026, 1006; MS (EI): *m/z* (%) 290 (M^+, 22.8), 185 (100); HRMS Cacld. for $C_{20}H_{18}O_2$ (M^+): 290.1307; Found: 290.1304.

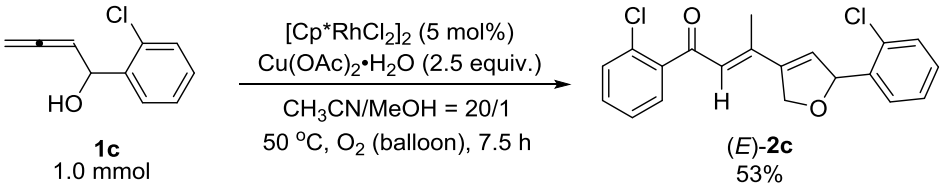
2. Synthesis of (*E*)-1-(4-fluorophenyl)-3-(2-(4-fluorophenyl)-2,5-dihydrofuran-4-yl)-but-2-en-1-one (*E*)-**2b**. (wxy-1-155)



Following **Typical Procedure II,** the reaction of **1b** (166.9 mg, 1.0 mmol), $[Cp^*RhCl_2]_2$ (31.3 mg, 0.05 mmol), and $Cu(OAc)_2•H_2O$ (500.6 mg, 2.5 mmol) in CH_3CN (1.0 mL)/MeOH (50 μL) afforded (*E*)-**2b** (95.2 mg, 57%) (eluent: petroleum ether/ethyl acetate = 15/1, 600 mL); solid; m.p. 99.8-100.5 °C (petroleum ether/CH_2Cl_2); ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.04-7.88 (m, 2 H, ArH), 7.38-7.23 (m, 2 H, ArH), 7.22-7.11 (m, 2 H, ArH), 7.11-7.00 (m, 2 H, ArH), 6.53 (s, 1 H, =CH), 6.32 (d, *J* = 1.8 Hz, 1 H, =CH), 6.00-5.90 (m, 1 H, OCH), 5.15 (ddd, J_1 = 11.4 Hz, J_2 = 5.4 Hz, J_3 = 1.8 Hz, 1 H, one proton of OCH_2), 5.01 (ddd, J_1 = 11.4 Hz, J_2 = 3.6 Hz, J_3 = 2.1 Hz, 1 H, one proton of OCH_2), 2.36 (d, *J* = 0.9 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 190.3, 165.5 (d, *J* = 253.0 Hz), 162.6 (d, *J* = 244.8 Hz), 145.0, 141.6, 136.8 (d, *J* = 2.8 Hz), 135.1 (d, *J* = 2.8 Hz), 132.7, 130.8 (d, *J* = 9.7 Hz), 128.2 (d, *J* = 8.3 Hz), 122.0, 115.7 (d, *J* = 21.4 Hz), 115.5 (d, *J* = 21.4 Hz), 88.4, 74.8, 16.7; ^{19}F NMR (282 MHz, $CDCl_3$) δ -105.9 (s, 1 F), -114.4 (s, 1 F); IR (KBr) *v* (cm^{-1}) 3108, 3080, 3054, 3033, 2890, 2853, 1655, 1622, 1601, 1505, 1448, 1410, 1370, 1343, 1298, 1293, 1247, 1216, 1190, 1160, 1160, 1154, 1105, 1090, 1063, 1044, 1014, 1007, 1006; MS (EI): *m/z* (%) 326 (M^+,

6.66), 123 (100); Anal. Calcd. for $C_{20}H_{16}F_2O_2$ (%): C, 73.61; H, 4.94; Found: C, 73.41; H, 5.03.

3. Preparation of (*E*)-1-(2-chlorophenyl)-3-(2-(2-chlorophenyl)-2,5-dihydrofuran-4-yl)but-2-en-1-one (*E*)-**2c**. (wxy-1-125)



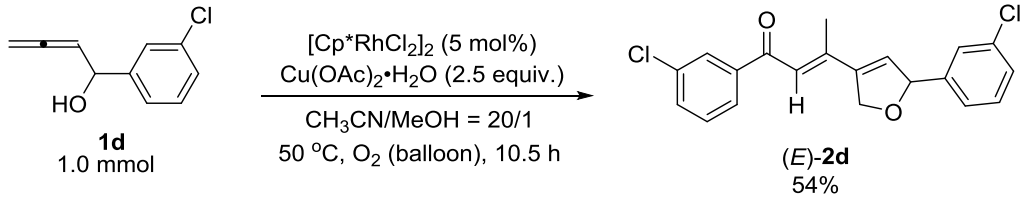
1c
1.0 mmol

(*E*)-**2c**
53%

Following **Typical Procedure II**, the reaction of **1c** (180.7 mg, 1.0 mmol), $[Cp^*RhCl_2]_2$ (31.0 mg, 0.05 mmol), and $Cu(OAc)_2•H_2O$ (500.2 mg, 2.5 mmol) in CH_3CN (1.0 mL)/MeOH (50 μL) afforded (*E*)-**2c** (95.9 mg, 53%) (eluent: petroleum ether/ethyl acetate = 20/1, 600 mL): solid; m.p. 91.1-93.0 ^oC (petroleum ether/$CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 7.55-7.45 (m, 2 H, ArH), 7.44-7.18 (m, 6 H, ArH), 6.52 (q, J = 1.8 Hz, 1 H, =CH), 6.35-6.27 (m, 2 H, OCH and =CH), 5.12 (ddd, J_1 = 11.5 Hz, J_2 = 5.5 Hz, J_3 = 1.9 Hz, 1 H, one proton of OCH_2), 5.01 (ddd, J_1 = 11.4 Hz, J_2 = 5.7 Hz, J_3 = 2.1 Hz, 1 H, one proton of OCH_2), 2.41 (s, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$): δ 192.9, 145.6, 141.5, 140.4, 138.6, 132.4, 131.7, 131.3, 131.1, 130.3, 129.5, 129.4, 128.9, 127.3, 127.0, 124.8, 86.0, 75.0, 16.6; IR (neat) ν (cm^{-1}) 3067, 1665, 1609, 1591, 1579, 1469, 1435, 1368, 1299, 1264, 1239, 1212, 1160, 1127, 1098, 1078, 1050, 1034, 1004; MS (EI): *m/z* (%) 362 [($M(^{37}Cl^{37}Cl)^+$, 0.53], 360 [($M(^{37}Cl^{35}Cl)^+$, 1.67], 358 [($M(^{35}Cl^{35}Cl)^+$, 2.13], 139 (100); Anal. Calcd. for $C_{20}H_{16}Cl_2O_2$ (%): C, 66.87; H, 4.49; Found: C, 66.74; H, 4.59.

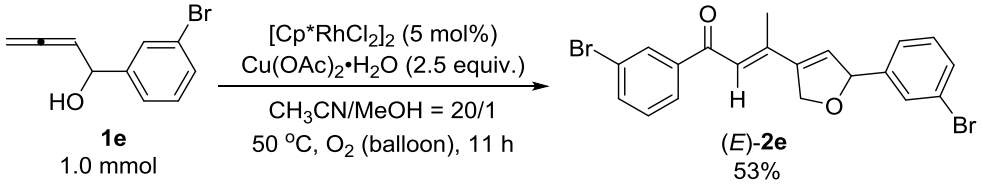
4. Preparation of (*E*)-1-(3-chlorophenyl)-3-(2-(3-chlorophenyl)-2,5-dihydrofuran-4-yl)but-

2-en-1-one (*E*)-**2d**. (wxy-1-134)



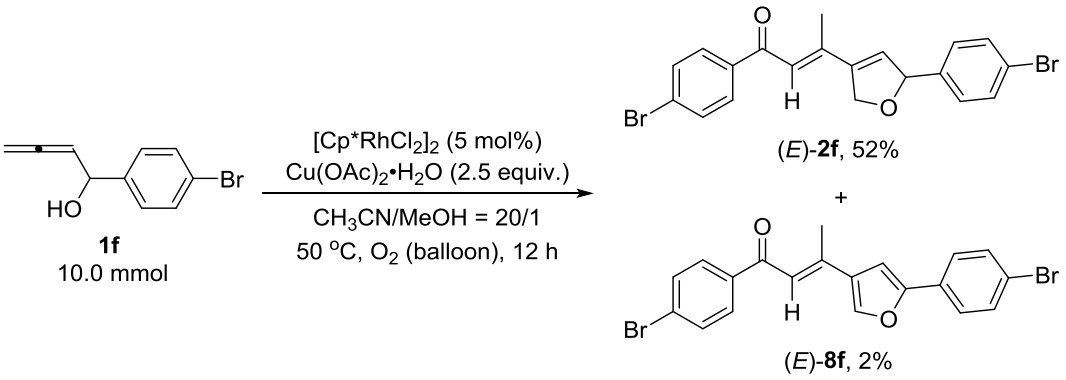
Following **Typical Procedure II**, the reaction of **1d** (180.7 mg, 1.0 mmol), $[Cp^*RhCl_2]_2$ (31.1 mg, 0.05 mmol), and $Cu(OAc)_2•H_2O$ (500.5 mg, 2.5 mmol) in CH_3CN (1.0 mL)/MeOH (50 μL) afforded (*E*)-**2d** (97.1 mg, 54%) (eluent: petroleum ether/ethyl acetate = 25/1, 600 mL): oil; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.87 (t, *J* = 1.7 Hz, 1 H, ArH), 7.78 (d, *J* = 7.5 Hz, 1 H, ArH), 7.55-7.47 (m, 1 H, ArH), 7.41 (t, *J* = 7.8 Hz, 1 H, ArH), 7.35-7.24 (m, 3 H, ArH), 7.24-7.18 (m, 1 H, ArH), 6.50 (s, 1 H, CH=), 6.33 (q, *J* = 1.8 Hz, 1 H, =CH), 5.95-5.89 (m, 1 H, OCH), 5.16 (ddd, J_1 = 11.7 Hz, J_2 = 5.4 Hz, J_3 = 1.8 Hz, 1 H, one proton of OCH_2), 5.02 (ddd, J_1 = 11.7 Hz, J_2 = 5.4 Hz, J_3 = 1.8 Hz, 1 H, one proton of OCH_2), 2.36 (s, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 190.2, 145.8, 143.1, 141.6, 140.3, 134.8, 134.5, 132.7, 132.6, 129.9, 128.2, 128.1, 126.4, 126.2, 124.3, 121.7, 88.2, 74.9, 16.7; IR (neat) *ν* (cm^{-1}) 3066, 2851, 1659, 1614, 1593, 1575, 1432, 1367, 1346, 1298, 1242, 1213, 1163, 1101, 1078, 1055; MS (EI): *m/z* (%) 362 [(M($^{37}Cl^{37}Cl$)$^+$, 0.85], 360 [(M($^{37}Cl^{35}Cl$)$^+$, 4.19], 358 [(M($^{35}Cl^{35}Cl$)$^+$, 7.15], 139 (100); HRMS Calcd for $C_{20}H_{16}O_2{}^{35}Cl^{35}Cl$ (M^+): 358.0527; Found: 358.0524.

5. Synthesis of (*E*)-1-(3-bromophenyl)-3-(2-(3-bromophenyl)-2,5-dihydrofuran-4-yl)-but-2-en-1-one (*E*)-**2e**. (wxy-1-192)



Following **Typical Procedure II**, the reaction of **1e** (227.3 mg, 1.0 mmol), $[Cp^*RhCl_2]_2$ (31.5 mg, 0.05 mmol), and $Cu(OAc)_2•H_2O$ (499.9 mg, 2.5 mmol) in CH_3CN (1.0 mL)/MeOH (50 μL) afforded impure (*E*)-**2e** (131.4 mg) after chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 20/1, 600 mL). The impure (*E*)-**2e** was further purified via chromatography on silica gel to afford pure (*E*)-**2e** (eluent: petroleum ether (30-60 ^oC)/Et_2O (6/1 (600 mL)) to afford pure (*E*)-**2e** (119.7 mg, 53%): oil; ^1H NMR (300 MHz, $CDCl_3$) δ 8.02 (s, 1 H, ArH), 7.82 (d, *J* = 7.8 Hz, 1 H, ArH), 7.71-7.64 (m, 1 H, ArH), 7.48 (s, 1 H, ArH), 7.46-7.38 (m, 1 H, ArH), 7.38-7.30 (m, 1 H, ArH), 7.28-7.19 (m, 2 H, ArH), 6.49 (s, 1 H, =CH), 6.33 (q, *J* = 1.8 Hz, 1 H, =CH), 5.95-5.88 (m, 1 H, OCH), 5.16 (ddd, J_1 = 11.7 Hz, J_2 = 5.4 Hz, J_3 = 1.8 Hz, 1 H, one proton of OCH_2), 5.02 (ddd, J_1 = 11.4 Hz, J_2 = 3.3 Hz, J_3 = 2.1 Hz, 1 H, one proton of OCH_2), 2.36 (s, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 190.1, 145.8, 143.3, 141.7, 140.5, 135.5, 132.7, 131.15, 131.09, 130.2, 130.1, 129.3, 126.7, 124.8, 122.9, 122.8, 121.7, 88.2, 74.9, 16.7; IR (KBr) *v* (cm^{-1}) 3063, 2850, 1661, 1593, 1575, 1569, 1472, 1429, 1367, 1296, 1241, 1211, 1102, 1084, 1070, 1054; MS (EI): *m/z* (%) 450 [($M(^{81}Br^{81}Br)^+$, 9.07], 448 [($M(^{81}Br^{79}Br)^+$, 18.29], 446 [($M(^{79}Br^{79}Br)^+$, 11.09], 265 (100); HRMS Cacld. for $C_{20}H_{16}O_2{}^{79}Br^{79}Br$ (M^+): 445.9517; Found: 445.9521.

6. Synthesis of (*E*)-1-(3-bromophenyl)-3-(2-(3-bromophenyl)-2,5-dihydrofuran-4-yl)-but-2-en-1-one (*E*)-**2f** and (*E*)-**8f**. (wxy-5-012)



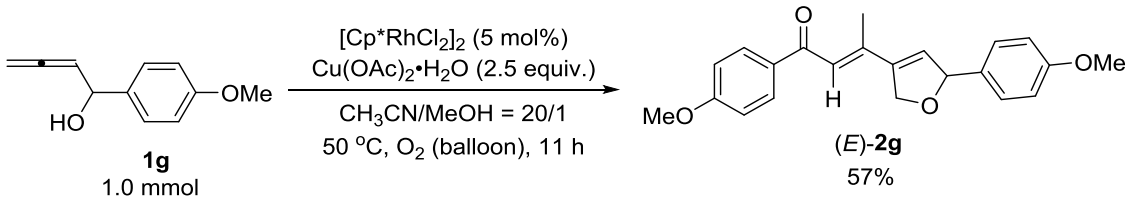
Following **Typical Procedure II**, the reaction of **1f** (2.2519 g, 10.0 mmol), $[Cp^*RhCl_2]_2$ (312.5 mg, 0.5 mmol), and $Cu(OAc)_2•H_2O$ (5.0019 g, 25 mmol) in CH_3CN (10 mL)/MeOH (0.5 mL) afforded (*E*)-**2f** (1.1697 g, 52%) and (*E*)-**8f** (37.0 mg, 2%) [eluent: petroleum ether/ethyl acetate = 25/1 (500 mL) to petroleum ether/ethyl acetate = 10/1 (500 mL) to petroleum ether/ethyl acetate = 5/1 (300 mL)].

(*E*)-**2f:** solid; m.p. 121.5-122.5 °C (petroleum ether/ethyl acetate); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.78 (d, *J* = 8.4 Hz, 2 H, ArH), 7.61 (d, *J* = 8.7 Hz, 2 H, ArH), 7.50 (d, *J* = 8.4 Hz, 2 H, ArH), 7.21 (d, *J* = 8.4 Hz, 2 H, ArH), 6.50 (s, 1 H, =CH), 6.31 (q, *J* = 1.8 Hz, 1 H, =CH), 6.00-5.85 (m, 1 H, OCH), 5.14 (ddd, J_1 = 11.4 Hz, J_2 = 5.4 Hz, J_3 = 1.8 Hz, 1 H, one proton of OCH_2), 5.01 (ddd, J_1 = 11.4 Hz, J_2 = 3.6 Hz, J_3 = 2.0 Hz, 1 H, one proton of OCH_2), 2.35 (d, *J* = 0.9 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 190.7, 145.5, 141.7, 140.0, 137.6, 132.7, 131.9, 131.8, 129.7, 128.0, 127.9, 122.0, 121.8, 88.4, 74.9, 16.8; IR (KBr) *v* (cm^{-1}) 3080, 2849, 1657, 1588, 1485, 1404, 1243, 1215, 1176, 1090, 1070, 1049, 1009; MS (EI): *m/z* (%) 450 [($M(^{81}Br^{81}Br)^+$, 5.25], 448 [($M(^{81}Br^{79}Br)^+$, 10.07], 446 [($M(^{79}Br^{79}Br)^+$, 5.19], 265 (100); Anal. Calcd. for $C_{20}H_{16}Br_2O_2$ (%): C, 53.60; H, 3.60; Found: C, 53.56; H, 3.61.

(*E*)-**8f:** solid; m.p. 164.1-164.8 °C (petroleum ether/CH_2Cl_2); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.85 (d, *J* = 8.4 Hz, 2 H, ArH), 7.78 (s, 1 H, ArH), 7.63-7.30 (m, 6 H, ArH and =CH), 7.10

(s, 1 H, =CH), 6.92 (s, 1 H, =CH), 2.52 (s, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 190.4, 154.5, 146.6, 142.4, 138.3, 132.0, 131.8, 130.6, 129.7, 128.9, 127.5, 125.6, 122.1, 118.6, 102.8, 17.6; IR (KBr) *v* (cm^{-1}) 2922, 2847, 1654, 1612, 1600, 1582, 1479, 1397, 1315, 1219, 1198, 1168, 1151, 1070, 1043, 1006; MS (EI): m/z (%) 448 [($M(^{81}Br^{81}Br)^+$, 4.60], 446 [($M(^{81}Br^{79}Br)^+$, 12.76], 444 [($M(^{79}Br^{79}Br)^+$, 7.39], 183 (100); Anal. Calcd. for $C_{20}H_{14}Br_2O_2$ (%): C, 53.84; H, 3.16; Found: C, 53.58; H, 3.10.

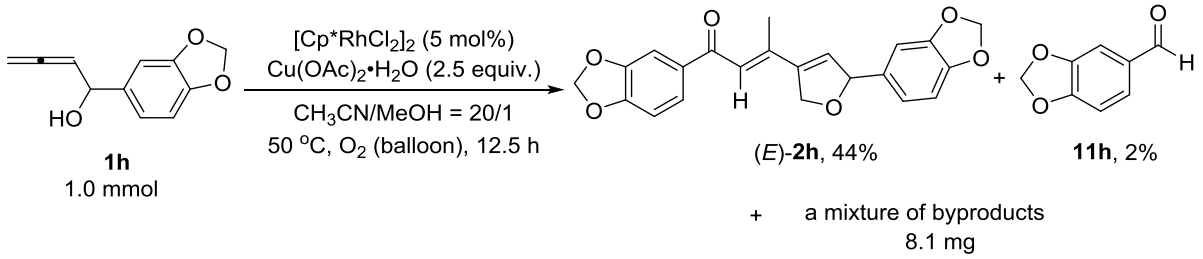
7. Synthesis of (*E*)-1-(4-methoxyphenyl)-3-(2-(4-methoxyphenyl)-2,5-dihydrofuran-4-yl)-but-2-en-1-one (*E*)-**2g**. (wxy-1-151)



Following **Typical Procedure II**, the reaction of **1g** (175.3 mg, 1.0 mmol), $[Cp^*RhCl_2]_2$ (30.7 mg, 0.05 mmol), and $Cu(OAc)_2•H_2O$ (500.3 mg, 2.5 mmol) in CH_3CN (1.0 mL)/MeOH (50 μL) afforded (*E*)-**2g** (99.9 mg, 57%) (eluent: petroleum ether/ethyl acetate = 10/1, 600 mL): solid; m.p. 97.9-99.3 °C (petroleum ether/CH_2Cl_2); ^1H NMR (300 MHz, $CDCl_3$) δ 7.92 (d, *J* = 9.0 Hz, 2 H, ArH), 7.25 (d, *J* = 8.7 Hz, 2 H, ArH), 6.95 (d, *J* = 8.7 Hz, 2 H, ArH), 6.90 (d, *J* = 8.7 Hz, 2 H, ArH), 6.54 (s, 1 H, =CH), 6.28 (q, *J* = 1.8 Hz, 1 H, =CH), 5.94-5.87 (m, 1 H, OCH), 5.13 (ddd, J_1 = 11.4 Hz, J_2 = 5.4 Hz, J_3 = 1.8 Hz, 1 H, one proton of OCH_2), 4.99 (ddd, J_1 = 11.4 Hz, J_2 = 3.8 Hz, J_3 = 1.8 Hz, 1 H, one proton of OCH_2), 3.87 (s, 3 H, OCH_3), 3.80 (s, 3 H, OCH_3), 2.34 (d, *J* = 0.9 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 190.6, 163.3, 159.5, 143.8, 141.5, 133.2, 132.3, 131.7, 130.5, 127.9, 122.4, 114.0, 113.7, 88.6, 74.6, 55.4, 55.2, 16.6; IR (KBr) *v* (cm^{-1}) 3082, 3004, 2963, 2895, 2845, 1655, 1605, 1585, 1510,

1470, 1437, 1420, 1403, 1375, 1354, 1313, 1300, 1291, 1261, 1244, 1224, 1188, 1176, 1169, 1123, 1112, 1091, 1048, 1025, 1001; MS (EI): *m/z* (%) 350 (M^+, 8.23), 135 (100); Anal. Calcd. for $C_{22}H_{22}O_4$ (%): C, 75.41; H, 6.33; Found: C, 75.25; H, 6.40.

8. Synthesis of (*E*)-1-(benzodioxol-5-yl)-3-(2-(benzodioxol-5-yl)-2,5-dihydrofuran-4-yl)but-2-en-1-one (*E*)-**2h**. (wxy-1-147)



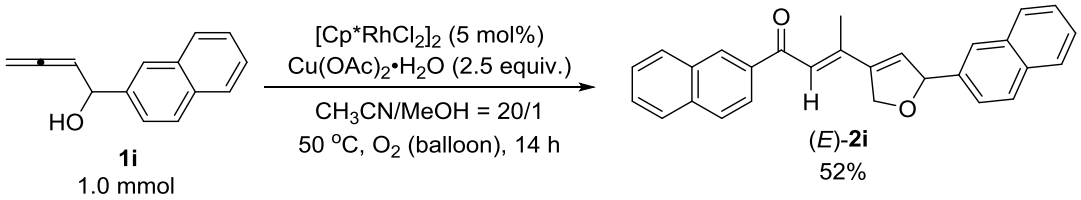
Following **Typical Procedure II**, the reaction of **1h** (191.4 mg, 1.0 mmol), $[Cp^*RhCl_2]_2$ (31.2 mg, 0.05 mmol), and $Cu(OAc)_2•H_2O$ (499.5 mg, 2.5 mmol) in CH_3CN (1.0 mL)/MeOH (50 μL) afforded (*E*)-**2h** (82.6 mg, 44%), **11h** (3.1 mg, 2%) and a mixture of other byproducts (8.1 mg) (eluent: petroleum ether/ethyl acetate = 10/1, 600 mL).

(*E*)-**2h**: solid; m.p. 137.7-138.4 °C (petroleum ether/CH_2Cl_2); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.51 (dd, J_1 = 8.1 Hz, J_2 = 1.8 Hz, 1 H, ArH), 7.42 (d, J = 1.8 Hz, 1 H, ArH), 6.86 (d, J = 8.1 Hz, 1 H, ArH), 6.79 (m, 3 H, ArH), 6.47 (s, 1 H, =CH), 6.25 (q, J = 1.8 Hz, 1 H, =CH), 6.05 (s, 2 H, CH_2), 5.95 (s, 2 H, CH_2), 5.88-5.83 (m, 1 H, OCH), 5.11 (ddd, J_1 = 11.7 Hz, J_2 = 5.4 Hz, J_3 = 1.8 Hz, 1 H, one proton of OCH_2), 4.97 (ddd, J_1 = 11.4 Hz, J_2 = 3.5 Hz, J_3 = 2.0 Hz, 1 H, one proton of OCH_2), 2.32 (d, J = 0.9 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 190.1, 151.6, 148.2, 147.9, 147.5, 143.9, 141.6, 135.0, 133.6, 132.2, 124.5, 122.4, 120.0, 108.2, 108.0, 107.8, 107.0, 101.8, 101.1, 88.8, 74.6, 16.6; IR (KBr) *v* (cm^{-1}) 3047, 2900, 2855,

2781, 1651, 1596, 1499, 1485, 1441, 1295, 1273, 1248, 1199, 1179, 1139, 1110, 1098, 1038, 1002; MS (EI): *m/z* (%) 378 (M^+, 34.14), 229 (100); Anal. Calcd. for $C_{22}H_{18}O_6$ (%): C, 69.84; H, 4.80; Found: C, 69.63; H, 4.87.

11h: ^{1}H NMR (300 MHz, $CDCl_3$) δ 9.82 (s, 1 H, CHO), 7.42 (dd, J_1 = 7.8 Hz, J_2 = 1.5 Hz, 1 H, ArH), 7.34 (d, J = 1.5 Hz, 1 H, ArH), 6.94 (d, J = 7.8 Hz, 1 H, ArH), 6.08 (s, 2 H, CH_2).

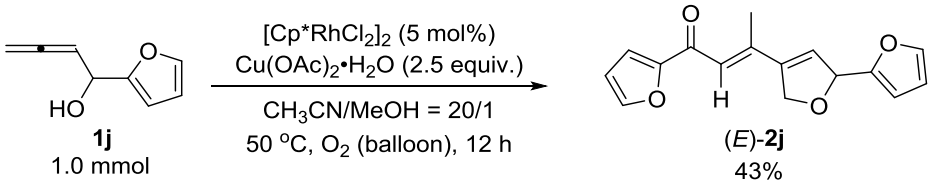
9. Synthesis of (*E*)-1-(naphthalen-2-yl)-3-(2-(naphthalen-2-yl)-2,5-dihydrofuran-4-yl)-but-2-en-1-one (*E*)-**2i**. (wxy-1-189, wxy-2-073)



Following **Typical Procedure II**, the reaction of **1i** (196.7 mg, 1.0 mmol), $[Cp^*RhCl_2]_2$ (31.0 mg, 0.05 mmol), and $Cu(OAc)_2•H_2O$ (499.9 mg, 2.5 mmol) in CH_3CN (1.0 mL)/MeOH (50 μL) afforded (*E*)-**2i** (105.8 mg, 52%, 97% purity) (eluent: petroleum ether/ethyl acetate = 20/1, 600 mL): solid; m.p. 127.8-128.6 °C (petroleum ether/Et_2O); ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.49 (dd, J_1 = 8.4 Hz, J_2 = 0.9 Hz, 1 H, ArH), 8.08 (d, J = 8.4 Hz, 1 H, ArH), 7.96 (d, J = 8.1 Hz, 1 H, ArH), 7.91-7.84 (m, 2 H, ArH), 7.83-7.75 (m, 2 H, ArH), 7.61-7.43 (m, 7 H, ArH), 6.73-6.68 (m, 1 H, OCH), 6.59 (q, J = 1.8 Hz, 1 H, =CH), 6.44 (s, 1 H, =CH), 5.15 (ddd, J_1 = 11.4 Hz, J_2 = 5.4 Hz, J_3 = 1.8 Hz, 1 H, one proton of OCH_2), 5.05 (ddd, J_1 = 11.4 Hz, J_2 = 3.6 Hz, J_3 = 2.1 Hz, 1 H, one proton of OCH_2), 2.41 (d, J = 0.9 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 195.5, 144.8, 141.8, 138.0, 136.5, 133.83, 133.75, 132.6, 132.1, 130.5, 130.1, 128.9, 128.4, 127.6, 126.4, 126.3, 125.8, 125.7, 125.6, 125.5, 124.5, 123.4,

122.9, 85.9, 74.7, 16.7; IR (neat) ν (cm^{-1}) 3048, 2849, 1652, 1579, 1508, 1436, 1395, 1367, 1294, 1230, 1178, 1110, 1077, 1055; MS (EI): *m/z* (%) 390 (M^+, 39.82), 235 (100); Anal. Calcd. for $C_{28}H_{22}O_2$ (%): C, 86.13; H, 5.68; Found: C, 85.75; H, 5.75.

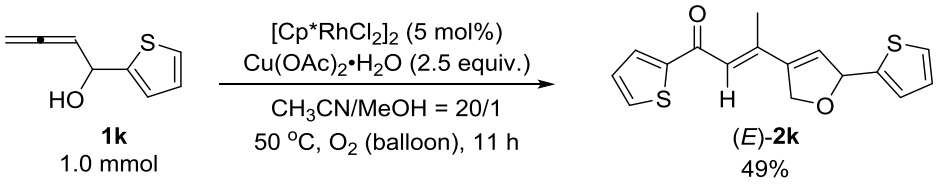
10. Synthesis of (*E*)-1-(furan-2-yl)-3-(2-(furan-2-yl)-2,5-dihydrofuran-4-yl)-but-2-en-1-one (*E*)-**2j**. (wxy-2-020)



Following **Typical Procedure II**, the reaction of **1j** (136.2 mg, 1.0 mmol), $[Cp^*RhCl_2]_2$ (31.0 mg, 0.05 mmol), and $Cu(OAc)_2•H_2O$ (500.0 mg, 2.5 mmol) in CH_3CN (1.0 mL)/MeOH (50 μL) afforded (*E*)-**2j** (57.6 mg, 43%) (eluent: petroleum ether/ethyl acetate = 20/1, 600 mL): oil; ^1H NMR (300 MHz, $CDCl_3$) δ 7.59 (d, J = 1.2 Hz, 1 H, furyl), 7.41 (d, J = 0.9 Hz, 1 H, furyl), 7.20 (d, J = 3.6 Hz, 1 H, furyl), 6.58-6.52 (m, 2 H, furyl and =CH), 6.39-6.29 (m, 3 H, furyl and =CH), 5.99-5.93 (m, 1 H, OCH), 5.08 (ddd, J_1 = 11.4 Hz, J_2 = 5.4 Hz, J_3 = 2.1 Hz, 1 H, one proton of OCH_2), 4.94 (ddd, J_1 = 11.4 Hz, J_2 = 3.3 Hz, J_3 = 2.1 Hz, 1 H, one proton of OCH_2), 2.49 (d, J = 0.9 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 179.3, 154.3, 152.9, 146.04, 146.01, 143.4, 142.9, 129.5, 120.5, 116.7, 112.4, 110.3, 107.8, 81.6, 74.2, 16.5; IR (neat) ν (cm^{-1}) 3121, 2853, 1652, 1591, 1501, 1467, 1387, 1305, 1256, 1154, 1092, 1052, 1013; MS (EI): m/z (%) 270 (M^+, 4.34), 95 (100); HRMS Cacld. for $C_{16}H_{14}O_4$ (M^+): 270.0892; Found: 270.0893.

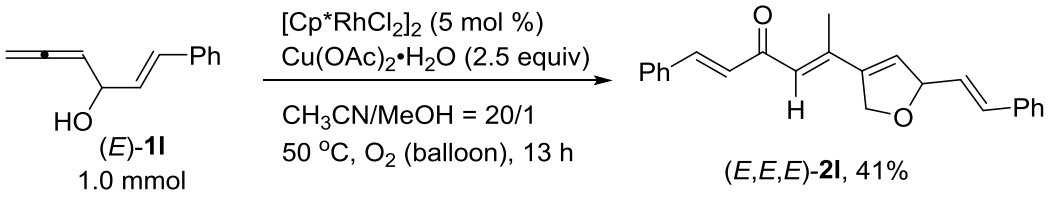
11. Synthesis of

(*E*)-1-(thiophen-2-yl)-3-(2-(thiophen-2-yl)-2,5-dihydrofuran-4-yl)-but-2-en-1-one (*E*)-**2k**. (wxy-1-191)



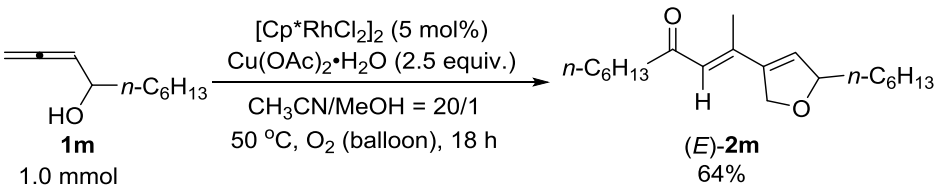
Following **Typical Procedure II**, the reaction of **1k** (153.9 mg, 1.0 mmol), $[Cp^*RhCl_2]_2$ (31.5 mg, 0.05 mmol), and $Cu(OAc)_2•H_2O$ (499.0 mg, 2.5 mmol) in CH_3CN (1.0 mL)/MeOH (50 μL) afforded (*E*)-**2k** (75.0 mg, 49%) (eluent: petroleum ether/ethyl acetate = 15/1, 600 mL): solid; m.p. 77.9-78.6 °C (petroleum ether/CH_2Cl_2); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.70 (dd, J_1 = 3.9 Hz, J_2 = 0.9 Hz, 1 H, thienyl), 7.64 (dd, J_1 = 4.8 Hz, J_2 = 0.9 Hz, 1 H, thienyl), 7.31 (dd, J_1 = 5.1 Hz, J_2 = 1.2 Hz, 1 H, thienyl), 7.14 (dd, J_1 = 5.1 Hz, J_2 = 3.9 Hz, 1 H, thienyl), 7.08-6.97 (m, 2 H, thienyl), 6.51 (s, 1 H, =CH), 6.38 (q, J = 1.8 Hz, 1 H, =CH), 6.25-6.18 (m, 1 H, OCH), 5.11 (ddd, J_1 = 11.4 Hz, J_2 = 5.1 Hz, J_3 = 1.8 Hz, 1 H, one proton of OCH_2), 4.96 (ddd, J_1 = 11.4 Hz, J_2 = 3.3 Hz, J_3 = 1.8 Hz, 1 H, one proton of OCH_2), 2.45 (d, J = 1.2 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 183.5, 146.5, 145.4, 144.6, 142.2, 133.7, 132.0, 131.3, 128.2, 126.9, 125.9, 124.9, 121.5, 84.0, 74.1, 16.6; IR (KBr) ν (cm^{-1}) 3084, 3071, 2953, 2899, 2855, 1637, 1612, 1581, 1518, 1473, 1436, 1415, 1354, 1297, 1272, 1251, 1238, 1221, 1163, 1090, 1066, 1042; MS (EI): *m/z* (%) 302 (M^+, 12.23), 273 (100); Anal. Calcd. for $C_{16}H_{14}O_2S_2$ (%): C, 63.55; H, 4.67; Found: C, 63.48; H, 4.72.

12. Synthesis of (*E*,*E*,*E*)-**2l**. (wxy-3-024)



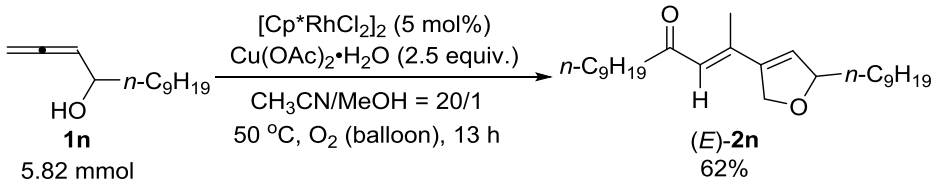
Following **Typical Procedure II**, the reaction of (*E*)-**1l** (172.8 mg, 1.0 mmol), $[Cp^*RhCl_2]_2$ (31.0 mg, 0.05 mmol), and $Cu(OAc)_2•H_2O$ (499.2 mg, 2.5 mmol) in CH_3CN (1.0 mL)/MeOH (50 μL) afforded impure (*E*,*E*,*E*)-**2l** (85.5 mg) after chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 20/1, 700 mL). The impure (*E*,*E*,*E*)-**2l** was further purified via chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 15/1, 500 mL) to afford pure (*E*,*E*,*E*)-**2l** (70.4 mg, 41%): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.63-7.51 (m, 3 H, ArH and =CH), 7.43-7.34 (m, 5 H, ArH), 7.34-7.20 (m, 3 H, ArH), 6.85 (d, J = 16.2 Hz, 1 H, =CH), 6.64 (d, J = 15.9 Hz, 1 H, =CH), 6.25 (q, J = 1.8 Hz, 1 H, =CH), 6.18 (dd, J_1 = 15.9 Hz, J_2 = 7.2 Hz, 1 H, =CH), 6.11 (s, 1 H, =CH), 5.59-5.49 (m, 1 H, OCH), 5.01 (ddd, J_1 = 11.4 Hz, J_2 = 5.1 Hz, J_3 = 1.8 Hz, 1 H, one proton of OCH_2), 4.90 (ddd, J_1 = 11.4 Hz, J_2 = 3.6 Hz, J_3 = 2.1 Hz, 1 H, one proton of OCH_2), 2.41 (d, J = 0.9 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 190.2, 144.9, 142.7, 141.9, 136.3, 134.6, 131.9, 131.5, 130.3, 128.8, 128.5, 128.3, 128.0, 127.9, 127.8, 126.6, 124.3, 87.8, 74.2, 16.4; IR (neat) ν (cm^{-1}) 3081, 3059, 3026, 2848, 1668, 1653, 1641, 1622, 1616, 1575, 1495, 1447, 1367, 1327, 1302, 1189, 1116, 1048; MS (EI): *m/z* (%) 342 (M^+, 24.75), 312 (100); HRMS Cacld. for $C_{24}H_{22}O_2$ (M^+): 342.1620; Found: 342.1621.

13. Synthesis of (*E*)-2-(2-hexyl-2,5-dihydrofuran-4-yl)dec-2-en-4-one (*E*)-**2m**. (hx-16-008, hx-15-181)



Following **Typical Procedure II**, the reaction of **1m** (154.5 mg, 1.0 mmol), $[Cp^*RhCl_2]_2$ (31.0 mg, 0.05 mmol), and $Cu(OAc)_2•H_2O$ (499.2 mg, 2.5 mmol) in CH_3CN (1 mL)/MeOH (50 μL) afforded (*E*)-**2m** (98.0 mg, 64%) [eluent: petroleum ether/ethyl acetate = 50/1 (500 mL) to 20/1 (400 mL)]: solid; m.p. 38.2-42.4 °C (determined without recrystallization): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.27-6.20 (m, 1 H, =CH), 5.80 (s, 1 H, =CH), 5.01-4.89 (m, 1 H, OCH), 4.82 (ddd, J_1 = 11.4 Hz, J_2 = 5.1 Hz, J_3 = 1.8 Hz, 1 H, one proton of OCH_2), 4.74 (ddd, J_1 = 11.4 Hz, J_2 = 3.6 Hz, J_3 = 2.1 Hz, 1 H, one proton of OCH_2), 2.47 (t, J = 7.5 Hz, 2 H, CH_2), 2.32 (d, J = 0.9 Hz, 3 H, Me), 1.68-1.48 (m, 4 H, CH_2 × 2), 1.47-1.18 (m, 14 H, CH_2 × 7), 0.88 (t, J = 6.8 Hz, 6 H, Me × 2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 201.8, 143.9, 141.2, 133.6, 123.5, 87.3, 73.9, 44.7, 35.7, 31.7, 31.6, 29.3, 28.8, 25.2, 24.1, 22.5, 22.4, 16.0, 14.00, 13.96; IR (neat) ν (cm^{-1}) 2955, 2928, 2856, 1685, 1616, 1589, 1466, 1406, 1367, 1201, 1132, 1101, 1054; MS (EI): *m/z* (%) 307 (M^+ + 1, 6.5), 306 (M^+, 23.2), 221 (100); HRMS Cacld. for $C_{20}H_{34}O_2$ (M^+): 306.2559; Found: 306.2563.

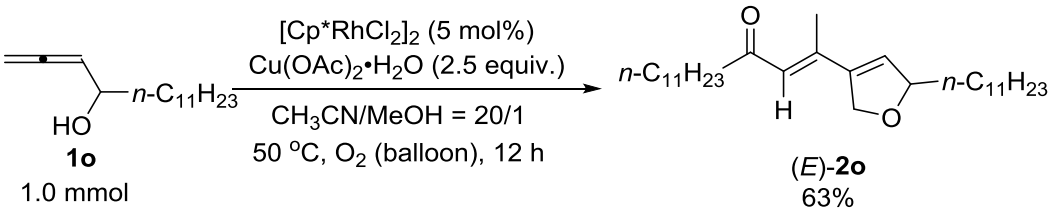
14. Synthesis of (*E*)-2-(2-nonyl-2,5-dihydrofuran-4-yl)tridec-2-en-4-one (*E*)-**2n**. (wxy-3-041)



Following **Typical Procedure II**, the reaction of **1n** (1.1990 g, 5.82 mmol, 97% purity), $[Cp^*RhCl_2]_2$ (185.4 mg, 0.3 mmol), and $Cu(OAc)_2•H_2O$ (2.9945 g, 15 mmol) in CH_3CN (6

mL)/MeOH (0.3 mL) afforded (*E*)-**2n** (714.3 mg, 62%) (eluent: petroleum ether/ethyl acetate = 35/1, 1000 mL): solid; m.p. 38.2-42.4 ^{o}C (determined without recrystallization); ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.24 (q, J = 1.8 Hz, 1 H, =CH), 5.80 (s, 1 H, =CH), 5.00-4.88 (m, 1 H, OCH), 4.82 (ddd, J_1 = 11.4 Hz, J_2 = 5.1 Hz, J_3 = 1.8 Hz, 1 H, one proton of OCH_2), 4.74 (ddd, J_1 = 11.4 Hz, J_2 = 3.6 Hz, J_3 = 1.8 Hz, 1 H, one proton of OCH_2), 2.46 (t, J = 7.5 Hz, 2 H, CH_2), 2.32 (d, J = 0.9 Hz, 3 H, CH_3), 1.68-1.50 (m, 4 H, CH_2 × 2), 1.48-1.15 (m, 26 H, CH_2 × 13), 0.88 (t, J = 6.8 Hz, 6 H, CH_3 × 2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 201.8, 143.9, 141.3, 133.6, 123.6, 87.3, 74.0, 44.8, 35.7, 31.8, 29.6, 29.53, 29.50, 29.4, 29.3, 29.22, 29.19, 25.3, 24.2, 22.6, 16.1, 14.0; IR (neat) ν (cm^{-1}) 2953, 2925, 2854, 1687, 1683, 1616, 1589, 1464, 1405, 1367, 1131, 1103, 1046; MS (EI): *m/z* (%) 390 (M^+, 9.17), 263 (100); Anal. Calcd. for $C_{26}H_{46}O_2$ (%): C, 79.94; H, 11.87; Found: C, 79.72; H, 11.81.

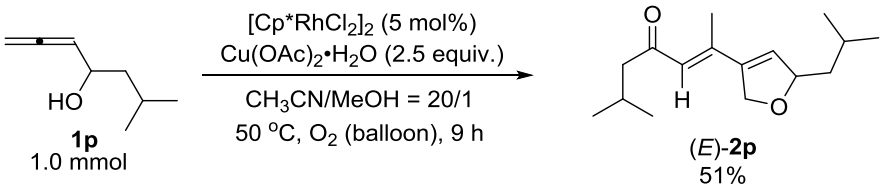
15. Synthesis of (*E*)-2-(2-undecyl-2,5-dihydrofuran-4-yl)pentadec-2-en-4-one (*E*)-**2o**. (fjj-1-118)



Following **Typical Procedure II**, the reaction of **1o** (223.9 mg, 1 mmol), $[Cp^*RhCl_2]_2$ (30.8 mg, 0.05 mmol), and $Cu(OAc)_2•H_2O$ (499.0 mg, 2.5 mmol) in CH_3CN (1.0 mL)/MeOH (50 μL) afforded (*E*)-**2o** (141.2 mg, 63%) [eluent: petroleum ether/ethyl acetate = 80/1 (300 mL) to 50/1 (300 mL)]: solid; m.p. 44.7-47.2 ^{o}C (determined without recrystallization); ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.24 (s, 1 H, =CH), 5.80 (s, 1 H, =CH), 5.00-4.89 (m, 1 H, OCH), 4.88-4.68 (m, 2 H, OCH_2), 2.47 (t, J = 7.5 Hz, 2 H, CH_2), 2.32 (s, 3 H, CH_3), 1.68-1.50 (m, 4

H, CH_2 × 2), 1.48-1.15 (m, 34 H, CH_2 × 17), 0.88 (t, *J* = 6.6 Hz, 6 H, CH_3 × 2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 201.8, 143.9, 141.3, 133.6, 123.6, 87.3, 74.0, 44.8, 35.8, 31.9, 29.7, 29.62, 29.58, 29.55, 29.5, 29.4, 29.3, 29.2, 25.3, 24.2, 22.6, 16.1, 14.1; IR (neat) *v* (cm^{-1}) 2954, 2918, 2849, 1683, 1591, 1470, 1133, 1096; MS (EI): *m/z* (%) 446 (M^+, 6.92), 263 (100); Anal. Calcd. for $C_{30}H_{54}O_2$ (%): C, 80.65; H, 12.18; Found: C, 80.22; H, 12.07.

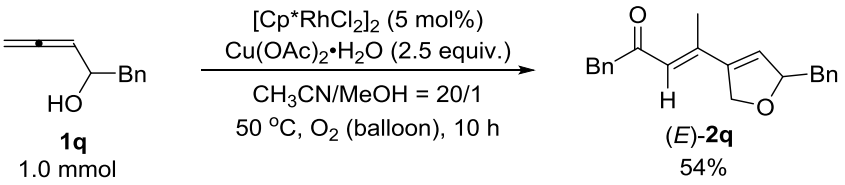
16. Preparation of (*E*)-2-(2-isobutyl-2,5-dihydrofuran-4-yl)-6-methylhept-2-en-4-one (*E*)-**2p**. (wxy-1-135)



Following **Typical Procedure II**, the reaction of **1p** (128.2 mg, 1.0 mmol), $[Cp^*RhCl_2]_2$ (31.2 mg, 0.05 mmol), and $Cu(OAc)_2•H_2O$ (500.7 mg, 2.5 mmol) in CH_3CN (1.0 mL)/MeOH (50 μL) afforded (*E*)-**2p** (64.8 mg, 51%) (eluent: petroleum ether/ethyl acetate = 30/1, 600 mL): liquid; ^1H NMR (300 MHz, $CDCl_3$) δ 6.25 (d, *J* = 1.8 Hz, 1 H, =CH), 5.79 (s, 1 H, CH=), 5.03-4.95 (m, 1 H, OCH), 4.81 (ddd, J_1 = 11.4 Hz, J_2 = 5.4 Hz, J_3 = 1.8 Hz, 1 H, one proton of OCH_2), 4.73 (ddd, J_1 = 11.4 Hz, J_2 = 3.5 Hz, J_3 = 2.0 Hz, 1 H, one proton of OCH_2), 2.35 (d, *J* = 6.9 Hz, 2 H, $COCH_2$), 2.32 (d, *J* = 6.9 Hz, 3 H, CH_3), 2.21-2.07 (m, 1 H, CH), 1.88-1.72 (m, 1 H, CH), 1.58-1.47 (m, 1 H, one proton of CH_2), 1.45-1.34 (m, 1 H, one proton of CH_2), 0.94 (t, *J* = 6.6 Hz, 12 H, CH_3 × 4); ^{13}C NMR (75 MHz, $CDCl_3$) δ 201.4, 143.8, 141.0, 134.1, 123.8, 85.7, 73.6, 53.7, 44.7, 25.1, 25.0, 23.2, 22.5, 22.4, 16.0; IR (neat) *v* (cm^{-1}) 2956, 2929, 2870, 1683, 1616, 1587, 1467, 1404, 1386, 1367, 1287, 1171, 1148, 1123, 1099, 1052 ; MS (EI): *m/z* (%) 250 (M^+, 7.99), 109 (100); HRMS Calcd for $C_{16}H_{26}O_2$

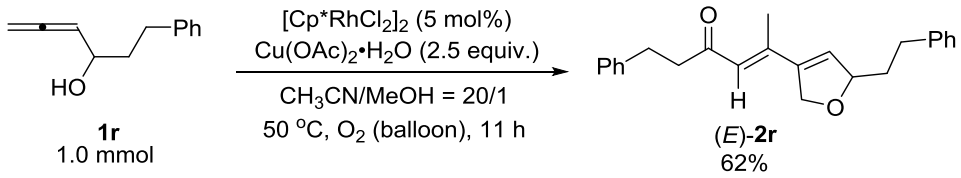
(M^+): 250.1933; Found: 250.1934.

17. Synthesis of (*E*)-1-phenyl-4-(2-benzyl-2,5-dihydrofuran-4-yl)-pent-3-en-2-one (*E*)-**2q**. (wxy-1-145)



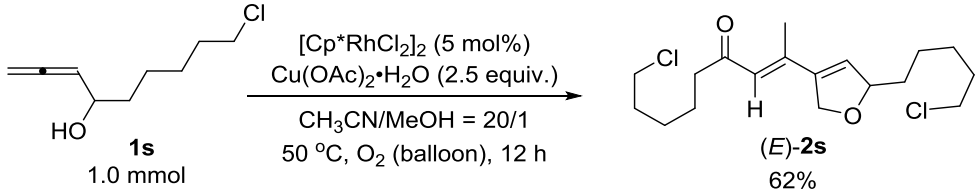
Following **Typical Procedure II**, the reaction of **1q** (160.3 mg, 1.0 mmol), $[Cp^*RhCl_2]_2$ (31.0 mg, 0.05 mmol), and $Cu(OAc)_2•H_2O$ (500.3 mg, 2.5 mmol) in CH_3CN (1.0 mL)/MeOH (50 μL) afforded (*E*)-**2q** (85.9 mg, 54%) (eluent: petroleum ether/ethyl acetate = 20/1, 800 mL): oil; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.35-7.15 (m, 10 H, ArH), 6.18 (q, *J* = 1.8 Hz, 1 H, =CH), 5.80 (s, 1 H, =CH), 5.18-5.08 (m, 1 H, OCH), 4.72-4.60 (m, 2 H, OCH_2), 3.72 (s, 2 H, CH_2CO), 2.94 (dd, J_1 = 13.8 Hz, J_2 = 6.6 Hz, 1 H, one proton of CH_2), 2.82 (dd, J_1 = 13.5 Hz, J_2 = 6.6 Hz, 1 H, one proton of CH_2), 2.27 (d, *J* = 1.2 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 198.3, 145.2, 141.7, 137.5, 134.5, 133.1, 129.4, 129.2, 128.6, 128.3, 126.9, 126.4, 122.9, 88.0, 74.0, 51.6, 42.3, 16.1; IR (neat) *v* (cm^{-1}) 3087, 3062, 3028, 2917, 2848, 1687, 1683, 1616, 1588, 1495, 1454, 1404, 1366, 1326, 1207, 1189, 1104, 1031, 1003; MS (EI): *m/z* (%) 318 (M^+, 7.72), 319 (M^+ + 1, 7.56), 227 (100), 91 (100); HRMS Cacld. for $C_{22}H_{22}O_2$ (M^+): 318.1620; Found: 318.1623.

18. Preparation of (*E*)-1-phenyl-5-(2-phenethyl-2,5-dihydrofuran-4-yl)-hex-4-en-3-one (*E*)-**2r**. (wxy-1-131)



Following **Typical Procedure II**, the reaction of **1r** (174.5 mg, 1.0 mmol), $[Cp^*RhCl_2]_2$ (31.0 mg, 0.05 mmol), and $Cu(OAc)_2•H_2O$ (500.1 mg, 2.5 mmol) in CH_3CN (1.0 mL)/MeOH (50 μL) afforded (*E*)-**2r** (106.6 mg, 62%) (eluent: petroleum ether/ethyl acetate = 20/1, 600 mL): liquid; ^1H NMR (300 MHz, $CDCl_3$) δ 7.34-7.24 (m, 4 H, ArH), 7.23-7.14 (m, 6 H, ArH), 6.18 (q, J = 1.8 H, 1 H, =CH), 5.76 (s, 1 H, =CH), 5.02-4.92 (m, 1 H, OCH), 4.80 (ddd, J_1 = 11.3 Hz, J_2 = 5.1 Hz, J_3 = 1.4 Hz, 1 H, one proton of OCH_2), 4.71 (ddd, J_1 = 11.4 Hz, J_2 = 3.3 Hz, J_3 = 1.8 Hz, 1 H, one proton of OCH_2), 2.92 (t, J = 7.7 Hz, 2 H, CH_2), 2.85-2.59 (m, 4 H, CH_2 × 2), 2.30 (s, 3 H, CH_3), 1.98-1.82 (m, 2 H, CH_2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 200.3, 144.2, 141.7, 141.4, 141.0, 133.5, 128.38, 128.34, 128.32, 128.26, 126.0, 125.8, 123.5, 86.5, 74.0, 46.1, 37.2, 31.4, 30.0, 16.1; IR (neat) v (cm^{-1}) 3080, 3061, 3026, 2925, 2852, 1683, 1616, 1589, 1496, 1454, 1111, 1072, 1031; MS (EI): *m/z* (%) 346 (M^+, 10.90), 91 (100); HRMS Calcd for $C_{24}H_{26}O_2$ (M^+): 346.1933; Found: 346.1937.

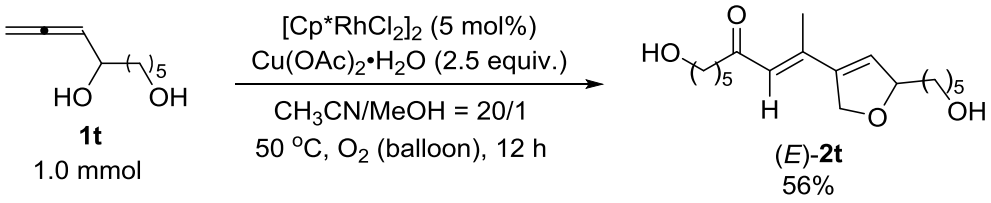
19. Synthesis of (*E*)-2-(2-(5-chloropentyl)-2,5-dihydrofuran-4-yl)-9-chloro-non-2-en-4-one (*E*)-**2s**. (wxy-2-018)



Following **Typical Procedure II**, the reaction of **1s** (174.5 mg, 1.0 mmol), $[Cp^*RhCl_2]_2$ (30.9 mg, 0.05 mmol), and $Cu(OAc)_2•H_2O$ (500.0 mg, 2.5 mmol) in CH_3CN (1.0 mL)/MeOH

(50 μL) afforded (*E*)-**2s** (107.8 mg, 62%) (eluent: petroleum ether/ethyl acetate = 20/1, 600 mL): liquid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.24 (q, *J* = 1.5 Hz, 1 H, =CH), 5.80 (s, 1 H, =CH), 5.03-4.91 (m, 1 H, OCH), 4.86-4.70 (m, 2 H, OCH_2), 3.54 (t, *J* = 6.5 Hz, 4 H, CH_2 × 2), 2.51 (t, *J* = 7.2 Hz, 2 H, $COCH_2$), 2.33 (s, 3 H, CH_3), 1.85-1.72 (m, 4 H, CH_2 × 2), 1.70-1.55 (m, 4 H, CH_2 × 2), 1.55-1.38 (m, 6 H, CH_2 × 3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 201.0, 143.9, 141.2, 133.4, 123.3, 86.9, 73.8, 44.8, 44.7, 44.2, 35.3, 32.3, 32.2, 26.7, 26.2, 24.4, 23.1, 15.9; IR (neat) *v* (cm^{-1}) 2928, 2852, 1683, 1588, 1368, 1111, 1046; MS (EI): *m/z* (%) 350 [M($^{37}Cl^{37}Cl$)$^+$, 1.28], 348 [M($^{37}Cl^{35}Cl$)$^+$, 5.84], 346 [M($^{35}Cl^{35}Cl$)$^+$, 7.74], 241 (100); HRMS Cacld. for $C_{18}H_{28}O_2{}^{35}Cl^{35}Cl$ (M^+): 346.1466; Found: 346.1465.

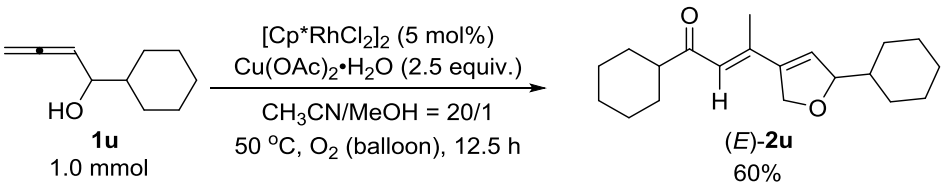
20. Synthesis of (*E*)-**2t**. (fjj-1-121)



Following **Typical Procedure II**, the reaction of **1t** (156.4 mg, 1 mmol), $[Cp^*RhCl_2]_2$ (30.9 mg, 0.05 mmol), and $Cu(OAc)_2•H_2O$ (499.2 mg, 2.5 mmol) in CH_3CN (1.0 mL)/MeOH (50 μL) afforded (*E*)-**2t** (87.1 mg, 56%) [eluent: petroleum ether/ethyl acetate = 1/1 (500 mL) to petroleum ether/ethyl acetate 1/2 (500 mL) to ethyl acetate (500 mL)]: oil; ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.25 (s, 1 H, =CH), 5.81 (s, 1 H, =CH), 5.02-4.90 (m, 1 H, OCH), 4.86-4.68 (m, 2 H, OCH_2), 3.63 (t, *J* = 6.2 Hz, 4 H, OCH_2 × 2), 2.58-2.40 (4 H, CH_2 and OH × 2), 2.32 (s, 3 H, CH_3), 1.72-1.50 (m, 8 H, CH_2 × 4), 1.48-1.30 (m, 6 H, CH_2 × 3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 201.7, 144.1, 141.1, 133.5, 123.4, 87.1, 73.9, 62.4, 62.3, 44.5, 35.5, 32.4, 32.3, 25.6,

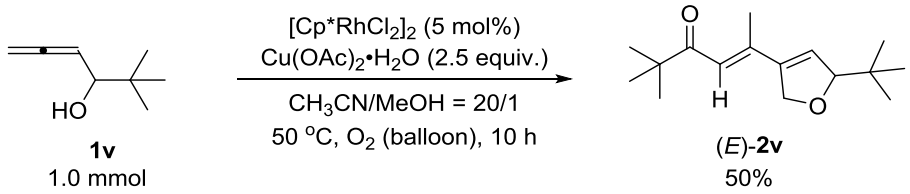
25.2, 24.9, 23.6, 16.0; IR (neat) *v* (cm^{-1}) 3390, 2932, 2858, 1682, 1587, 1119, 1051; MS (EI): *m/z* (%) 310 (M^+, 7.20), 69 (100); HRMS Cacld. for $C_{18}H_{30}O_4$ (M^+): 310.2144; Found: 310.2143.

21. Synthesis of (*E*)-1-cyclohexyl-3-(2-cyclohexyl-2,5-dihydrofuran-4-yl)but-2-en-1-one (*E*)-**2u**. (wxy-2-145)



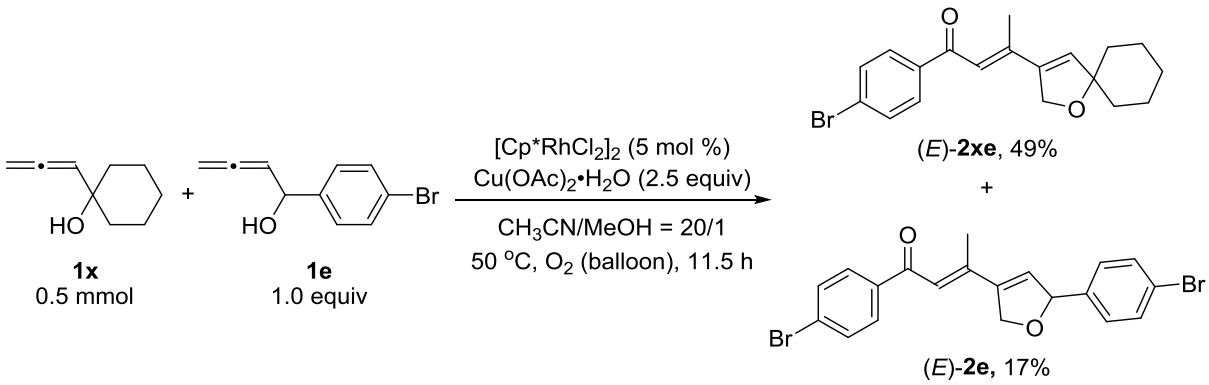
Following **Typical Procedure II**, the reaction of **1u** (152.2 mg, 1.0 mmol), $[Cp^*RhCl_2]_2$ (31.1 mg, 0.05 mmol), and $Cu(OAc)_2•H_2O$ (499.9 mg, 2.5 mmol) in CH_3CN (1.0 mL)/MeOH (50 μL) afforded (*E*)-**2u** (91.1 mg, 60%) (eluent: petroleum ether/ethyl acetate = 40/1, 800 mL): liquid; ^1H NMR (300 MHz, $CDCl_3$) δ 6.24 (s, 1 H, =CH), 5.84 (s, 1 H, =CH), 4.85-4.70 (m, 3 H, OCH + OCH_2), 2.45-2.25 (m, 4 H, CH + CH_3), 1.90-1.60 (m, 10 H, 5 × CH_2), 1.60-1.45 (m, 1 H, CH), 1.41-0.94 (m, 10 H, 5 × CH_2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 204.7, 144.3, 141.8, 131.9, 122.8, 91.8, 74.3, 51.9, 43.5, 28.7, 28.5, 28.4, 26.4, 26.1, 26.0, 25.8, 25.7, 16.1; IR (neat) *v* (cm^{-1}) 2926, 2852, 1682, 1615, 1587, 1450, 1368, 1314, 1289, 1233, 1185, 1146, 1098, 1051; MS (EI): *m/z* (%) 302 (M^+, 7.47), 83 (100); HRMS Cacld. for $C_{20}H_{30}O_2$ (M^+): 302.2246; Found: 302.2247.

22. Synthesis of (*E*)-2,2-dimethyl-5-(2-(tert-butyl)-2,5-dihydrofuran-4-yl)-hex-4-en-3-one (*E*)-**2v**. (wxy-1-159)



Following **Typical Procedure II**, the reaction of **1v** (128.1 mg, 1.0 mmol), $[Cp^*RhCl_2]_2$ (31.0 mg, 0.05 mmol), and $Cu(OAc)_2•H_2O$ (499.9 mg, 2.5 mmol) in CH_3CN (1.0 mL)/MeOH (50 μL) afforded (*E*)-**2v** (63.4 mg, 50%) [eluent: petroleum ether (30-60 ^oC)/ethyl acetate = 50/1, 800 mL]: solid; low melting point; ^1H NMR (300 MHz, $CDCl_3$) δ 6.23 (d, *J* = 1.5 Hz, 1 H, =CH), 6.06 (s, 1 H, =CH), 4.86-4.74 (m, 2 H, OCH_2), 4.64-4.57 (m, 1 H, OCH), 2.29 (d, *J* = 0.9 Hz, 3 H, CH_3), 1.16 (s, 9 H, *t*-Bu), 0.92 (s, 9 H, *t*-Bu); ^{13}C NMR (75 MHz, $CDCl_3$) δ 206.8, 144.2, 142.5, 130.6, 119.9, 95.8, 74.6, 44.0, 36.1, 26.5, 25.6, 16.2; IR (neat) *v* (cm^{-1}) 2958, 2902, 2868, 1679, 1616, 1587, 1478, 1393, 1365, 1312, 1192, 1102, 1066, 1036, 1016; MS (EI): *m/z* (%) 250 (M^+, 2.87), 57 (100); HRMS Cacld. for $C_{16}H_{26}O_2$ (M^+): 250.1933; Found: 250.1932.

23. The cross-coupling reaction between **1x** and **1e.** (wxy-7-163)

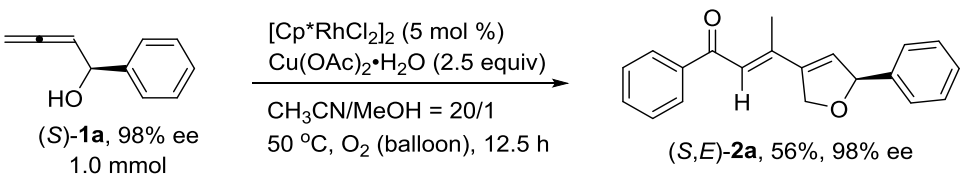


To a dried Schlenk tube were added $[Cp^*RhCl_2]_2$ (15.5 mg, 0.025 mmol) and $Cu(OAc)_2•H_2O$ (250.0 mg, 1.25 mmol) under air atmosphere. The Schlenk tube was then degassed to remove the air inside completely and refilled with O_2 by a balloon of O_2 for three

times. **1x** (70.5 mg, 0.5 mmol)/CH_3CN (0.5 mL), **1e** (112.8 mg, 0.5 mmol)/CH_3CN (0.5 mL) and MeOH (50 μL) were added sequentially, the reaction tube was put into an oil bath pre-heated at 50 ^oC. The reaction was complete after stirring for 11.5 h as monitored by TLC. After removing the O_2 balloon, the resulting mixture was filtered through a short column of silica gel eluted with ethyl acetate (15 mL × 3). The combined filtrate was then concentrated in vacuo and the crude residue was purified via chromatography on silica gel [eluent: petroleum ether/ethyl acetate = 50/1 (250 mL) to petroleum ether/ethyl acetate = 20/1 (250 mL)] to afford (*E*)-**2xe** (88.9 mg, 49%) and impure (*E*)-**2e** (20.2 mg). The impure (*E*)-**2e** was further purified via column chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 25/1, 500 mL) to afford pure (*E*)-**2e** (18.6 mg, 17%).

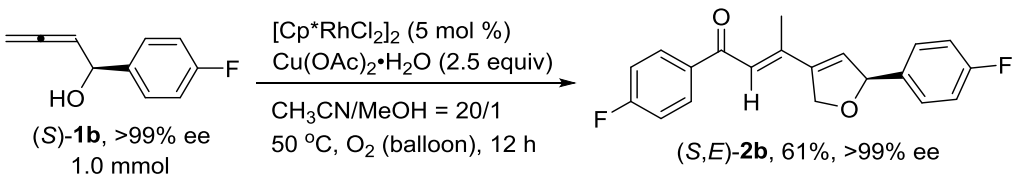
(*E*)-**2xe**, solid; m.p. 105.5-107.6 ^oC (petroleum ether/DCM); ^1H NMR (300 MHz, $CDCl_3$) δ 7.76 (d, *J* = 6.6 Hz, 2 H, ArH), 7.60 (d, *J* = 6.6 Hz, 2 H, ArH), 6.41 (s, 1 H, =CH), 6.38 (s, 1 H, =CH), 4.88 (d, *J* = 1.2 Hz, 2 H, OCH_2), 2.36 (d, *J* = 0.9 Hz, 3 H, Me), 1.80-1.35 (m, 10 H, CH_2 × 5); ^{13}C NMR (75 MHz, $CDCl_3$) δ 190.8, 146.7, 140.0, 137.8, 137.7, 131.8, 129.7, 127.7, 120.7, 90.7, 72.6, 36.6, 25.2, 23.2, 16.7; IR (neat) *v* (cm^{-1}) 2931, 2854, 1656, 1588, 1212, 1071, 1042, 1009; MS (EI): *m/z* (%) 362 [($M(^{81}Br)^+$, 42.42], 360 [($M(^{79}Br)^+$, 41.69], 183 (100); HRMS Calcd for $C_{19}H_{21}{}^{79}BrO_2$ (M^+): 360.0725; Found: 360.0723.

24. Synthesis of (*S*,*E*)-1-phenyl-3-(2-phenyl-2,5-dihydrofuran-4-yl)but-2-en-1-one (*S*,*E*)-**2a**. (wxy-3-112)



Following **Typical Procedure II**, the reaction of (*S*)-**1a**[2,3] (146.2 mg, 1.0 mmol), $[Cp^*RhCl_2]_2$ (31.0 mg, 0.05 mmol), and $Cu(OAc)_2•H_2O$ (499.8 mg, 2.5 mmol) in CH_3CN (1.0 mL)/MeOH (50 μL) afforded (*S,E*)-**2a** (81.1 mg, 56%) (eluent: petroleum ether/ethyl acetate = 25/1, 800 mL): solid; m.p. 82.4-82.7 ^oC (petroleum ether/ethyl acetate); 98% ee (HPLC conditions: Chiralcel IC column, *n*-hexane/*i*-PrOH = 90/10, 1 mL/min, λ = 214 nm, t_R(major) = 21.9 min, t_R(minor) = 24.2 min); $[\alpha]_D^{20}$ = -345.8 (*c* = 1.16, $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 7.96-7.88 (m, 2 H, ArH), 7.57 (m, 1 H, ArH), 7.48 (m, 2 H, ArH), 7.42-7.27 (m, 5 H, ArH), 6.57 (s, 1 H, =CH), 6.34 (q, J_1 = 3.6 Hz, J_2 = 1.5 Hz, 1 H, =CH), 6.00-5.96 (m, 1 H, OCH), 5.17 (ddd, J_1 = 11.4 Hz, J_2 = 5.4 Hz, J_3 = 2.1 Hz, 1 H, one proton of OCH_2), 5.03 (ddd, J_1 = 11.4 Hz, J_2 = 5.4 Hz, J_3 = 2.1 Hz, 1 H, one proton of OCH_2), 2.37 (d, *J* = 1.2 Hz, 3 H, Me); ^{13}C NMR (100 MHz, $CDCl_3$) δ 192.0 145.0, 141.5, 141.1, 138.9, 132.9, 132.8, 128.7, 128.6, 128.2, 128.1, 126.4, 122.3, 89.1, 74.9, 16.7; IR (KBr) *v* (cm^{-1}) 3089, 3065, 3035, 2854, 1651, 1612, 1595, 1581, 1492, 1479, 1455, 1446, 1366, 1350, 1274, 1243, 1218, 1196, 1182, 1160, 1088, 1075, 1049, 1009; MS (EI): *m/z* (%) 290 (M^+, 8.48), 185 (100); Anal. Calcd. for $C_{20}H_{18}O_2$ (%): C, 82.73; H, 6.25; Found: C, 82.01; H, 6.31. HRMS Cacld. for $C_{20}H_{18}O_2$ (M^+): 290.1307; Found: 290.1307.

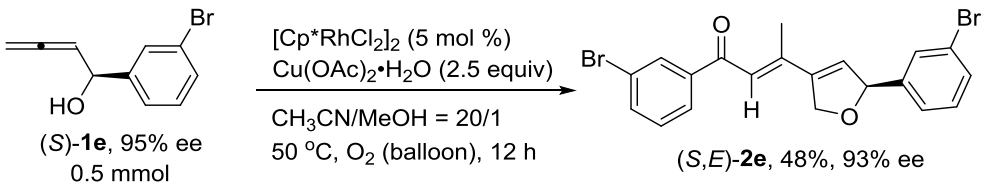
25. Synthesis of (*S,E*)-1-(4-fluorophenyl)-3-(2-(4-fluorophenyl)-2,5-dihydrofuran-4-yl)-but-2-en-1-one (*S,E*)-**2b**. (wxy-3-044)



Following **Typical Procedure II**, the reaction of (*S*)-**1b** (164.6 mg, 1.0 mmol, >99% ee),

$[Cp^*RhCl_2]_2$ (31.0 mg, 0.05 mmol), and $Cu(OAc)_2•H_2O$ (498.9 mg, 2.5 mmol) in CH_3CN (1.0 mL)/MeOH (50 μL) afforded (*S*,*E*)-**2b** (99.8 mg, 61%) (eluent: petroleum ether/ethyl acetate = 15/1, 500 mL): solid; m.p. 90.5-91.1 °C (petroleum ether/CH_2Cl_2); >99% ee (HPLC conditions: Chiralcel OZ-H column, *n*-hexane/*i*-PrOH = 90/10, 1 mL/min, λ = 214 nm, t_R(major) = 9.8 min, t_R(minor) = 13.6 min); $[\alpha]_D^{20}$ = -323.7 (*c* = 1.01, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.01-7.90 (m, 2 H, ArH), 7.36-7.25 (m, 2 H, ArH), 7.20-7.09 (m, 2 H, ArH), 7.09-7.00 (m, 2 H, ArH), 6.53 (s, 1 H, =CH), 6.31 (q, *J* = 1.8 Hz, 1 H, =CH), 5.98-5.87 (m, 1 H, OCH), 5.14 (ddd, J_1 = 11.7 Hz, J_2 = 5.7 Hz, J_3 = 2.1 Hz, 1 H, one proton of OCH_2), 5.01 (ddd, J_1 = 11.7 Hz, J_2 = 3.6 Hz, J_3 = 2.1 Hz, 1 H, one proton of OCH_2), 2.36 (d, *J* = 0.9 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 190.2, 165.5 (d, *J* = 253.1 Hz), 162.5 (d, *J* = 245.4 Hz), 145.0, 141.6, 136.8 (d, *J* = 3.5 Hz), 135.2 (d, *J* = 2.7 Hz), 132.6, 130.8 (d, *J* = 9.7 Hz), 128.2 (d, *J* = 8.3 Hz), 122.0, 115.6 (d, *J* = 21.4 Hz), 115.5 (d, *J* = 21.4 Hz), 88.3, 74.7, 16.6; ^{19}F NMR (282 MHz, $CDCl_3$) δ -105.9 (s, 1 F), -114.4 (s, 1 F); IR (neat) *v* (cm^{-1}) 3073, 2851, 1622, 1652, 1600, 1506, 1411, 1368, 1305, 1295, 1221, 1155, 1088, 1049, 1013; MS (EI): *m/z* (%) 326 (M^+, 5.52), 123 (100); Anal. Calcd. for $C_{20}H_{16}F_2O_2$ (%): C, 73.61; H, 4.94; Found: C, 73.64; H, 5.11.

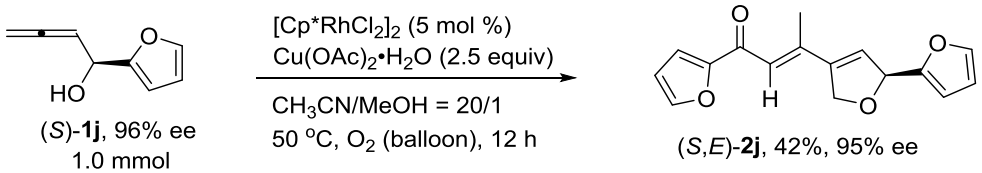
26. Synthesis of (*S*,*E*)-1-(3-bromophenyl)-3-(2-(3-bromophenyl)-2,5-dihydrofuran-4-yl)but-2-en-1-one (*S*,*E*)-**2e**. (wxy-3-109)



Following **Typical Procedure II**, the reaction of (*S*)-**1e** (112.1 mg, 0.5 mmol, 95% ee),

$[Cp^*RhCl_2]_2$ (15.7 mg, 0.025 mmol), and $Cu(OAc)_2•H_2O$ (250.0 mg, 1.25 mmol) in CH_3CN (0.5 mL)/MeOH (25 μL) afforded (*S,E*)-**2e** (54.0 mg, 48%) (eluent: petroleum ether/ethyl acetate = 25/1, 800 mL): oil; 93% ee (HPLC conditions: Chiralcel IC column, *n*-hexane/*i*-PrOH = 90/10, 1 mL/min, λ = 214 nm, t_R(minor) = 17.7 min, t_R(major) = 18.7 min); $[\alpha]_D^{20}$ = -258.2 (*c* = 0.91, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.03 (t, *J* = 1.7 Hz, 1 H, ArH), 7.83 (d, *J* = 7.8 Hz, 1 H, ArH), 7.72-7.64 (m, 1 H, ArH), 7.52-7.38 (m, 2 H, ArH), 7.36 (t, *J* = 7.8 Hz, 1 H, ArH), 7.30-7.19 (m, 2 H, ArH), 6.49 (s, 1 H, =CH), 6.33 (q, *J* = 1.5 Hz, 1 H, =CH), 5.97-5.88 (m, 1 H, OCH), 5.16 (ddd, J_1 = 11.4 Hz, J_2 = 5.4 Hz, J_3 = 1.8 Hz, 1 H, one proton of OCH_2), 5.03 (ddd, J_1 = 11.1 Hz, J_2 = 3.3 Hz, J_3 = 1.8 Hz, 1 H, one proton of OCH_2), 2.36 (s, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 190.2, 145.8, 143.3, 141.7, 140.6, 135.6, 132.7, 131.2, 131.1, 130.22, 130.18, 129.3, 126.7, 124.9, 122.9, 122.8, 121.8, 88.2, 75.0, 16.8; IR (KBr) *v* (cm^{-1}) 3062, 2955, 2850, 1660, 1592, 1571, 1472, 1428, 1367, 1296, 1240, 1211, 1101, 1084, 1070, 1054; MS (EI): *m/z* (%) 450 [($M(^{81}Br^{81}Br)^+$, 4.30], 448 [($M(^{81}Br^{79}Br)^+$, 9.42], 446 [($M(^{79}Br^{79}Br)^+$, 7.12], 265 (100); HRMS Cacld. for $C_{20}H_{16}O_2{}^{79}Br^{79}Br$ (M^+): 445.9512; Found: 445.9517.

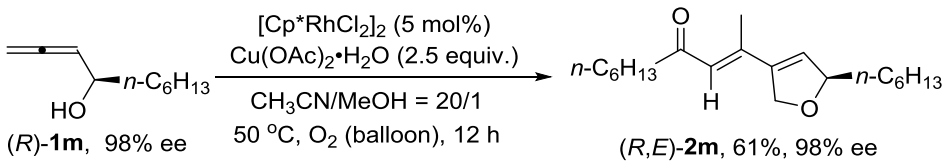
27. Synthesis of (*S,E*)-1-(furan-2-yl)-3-(2-(furan-2-yl)-2,5-dihydrofuran-4-yl)-but-2-en-1-one (*S,E*)-**2j**. (wxy-3-116)



Following **Typical Procedure II**, the reaction of (*S*)-**1j** (136.5 mg, 1.0 mmol, 96% purity, 96% ee), $[Cp^*RhCl_2]_2$ (31.0 mg, 0.05 mmol), and $Cu(OAc)_2•H_2O$ (500.0 mg, 2.5 mmol) in

CH_3CN (1.0 mL)/MeOH (50 μL) afforded (*S*,*E*)-**2j** (55.2 mg, 42%) (eluent: petroleum ether/ethyl acetate = 20/1, 1600 mL): solid; m.p. 87.5-88.1 ^oC (determined without recrystallization); 95% ee (HPLC conditions: Chiralcel OD-H column, *n*-hexane/*i*-PrOH = 85/15, 1 mL/min, λ = 220 nm, t_R(major) = 23.4 min, t_R(minor) = 19.0 min); $[\alpha]_D^{20}$ = -295.6 (*c* = 1.01, $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 7.60 (d, *J* = 0.9 Hz, 1 H, furyl), 7.46-7.36 (m, 1 H, furyl), 7.20 (dd, J_1 = 3.6 Hz, J_2 = 0.6 Hz, 1 H, furyl), 6.58-6.50 (m, 2 H, furyl and =CH), 6.39-6.27 (m, 3 H, furyl and =CH), 5.99-5.93 (m, 1 H, OCH), 5.08 (ddd, J_1 = 11.7 Hz, J_2 = 5.4 Hz, J_3 = 2.1 Hz, 1 H, one proton of OCH_2), 4.94 (ddd, J_1 = 11.4 Hz, J_2 = 3.3 Hz, J_3 = 2.1 Hz, 1 H, one proton of OCH_2), 2.49 (d, *J* = 0.9 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 179.3, 154.3, 152.9, 146.04, 146.01, 143.4, 142.9, 129.5, 120.5, 116.7, 112.4, 110.3, 107.8, 81.6, 74.2, 16.5; IR (neat) *v* (cm^{-1}) 3123, 2963, 2853, 1651, 1591, 1465, 1387, 1305, 1257, 1153, 1092, 1052, 1013; MS (EI): *m/z* (%) 270 (M^+, 4.56), 95 (100); HRMS Cacld. for $C_{16}H_{14}O_4$ (M^+): 270.0887; Found: 270.0886.

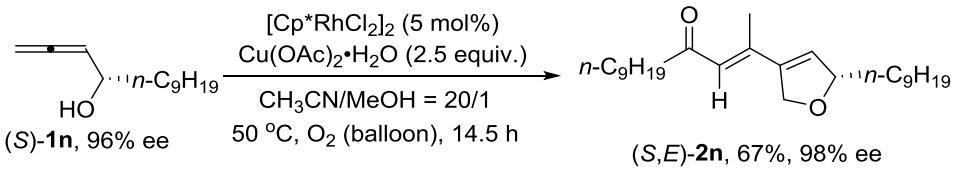
28. Synthesis of (*R*,*E*)-2-(2-hexyl-2,5-dihydrofuran-4-yl)dec-2-en-4-one (*R*,*E*)-**2m**. (wxy-3-113)



Following **Typical Procedure II**, the reaction of (*S*)-**1m**[2,4] (154.9 mg, 1.0 mmol, , 98% ee), $[Cp^*RhCl_2]_2$ (31.0 mg, 0.05 mmol), and $Cu(OAc)_2•H_2O$ (499.9 mg, 2.5 mmol) in CH_3CN (1 mL)/MeOH (50 μL) afforded (*R*,*E*)-**2m** (93.3 mg, 61%) (eluent: petroleum ether/ethyl acetate = 50/1, 700 mL): liquid; 98% ee (HPLC conditions: Chiralcel IC

column, *n*-hexane/*i*-PrOH = 99/1, 1.0 mL/min, λ = 220 nm, t_R(major) = 9.4 min, t_R(minor) = 11.2 min; $[\alpha]_D^{20}$ = -106.0 (*c* = 1.37, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.24 (q, *J* = 1.8 Hz, 1 H, =CH), 5.80 (s, 1 H, =CH), 5.01-4.90 (m, 1 H, OCH), 4.82 (ddd, J_1 = 11.4 Hz, J_2 = 5.4 Hz, J_2 = 1.8 Hz, 1 H, one proton of OCH_2), 4.74 (ddd, J_1 = 11.1 Hz, J_2 = 3.6 Hz, J_3 = 1.8 Hz, 1 H, one proton of OCH_2), 2.47 (t, *J* = 7.5 Hz, 2 H, CH_2), 2.32 (s, 3 H, Me), 1.68-1.50 (m, 4 H, CH_2 × 2), 1.45-1.19 (m, 14 H, CH_2 × 7), 0.88 (t, *J* = 6.6 Hz, 6 H, Me × 2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 201.8, 143.9, 141.3, 133.6, 123.5, 87.3, 73.9, 44.8, 35.7, 31.7, 31.6, 29.3, 28.8, 25.2, 24.1, 22.5, 22.4, 16.0, 14.01, 13.97; IR (neat) *v* (cm^{-1}) 2955, 2928, 2856, 1683, 1616, 1589, 1466, 1454, 1405, 1367, 1263, 1132, 1100, 1053; MS (EI): *m/z* (%) 306 (M^+, 9.28), 221 (100); HRMS Cacld. for $C_{20}H_{34}NaO_2$ (M^++ Na): 329.2457; Found: 329.2453.

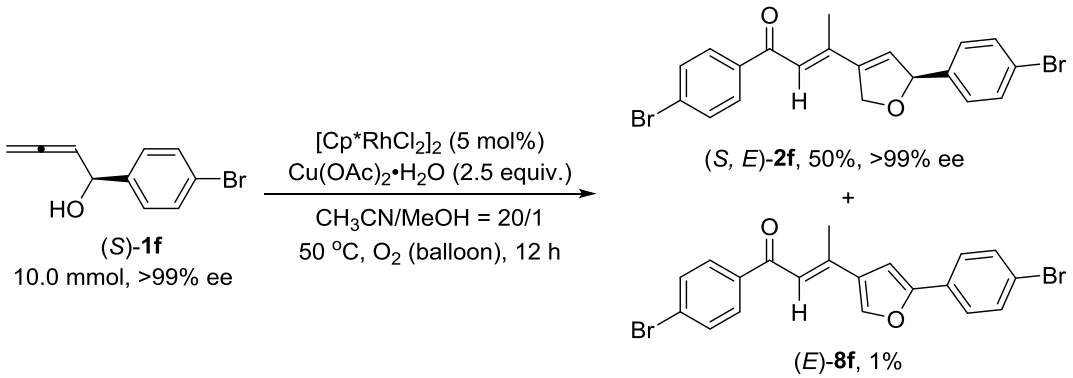
29. Synthesis of (*S*,*E*)-2-(2-nonyl-2,5-dihydrofuran-4-yl)tridec-2-en-4-one (*S*,*E*)-**2n**. (wxy-3-043)



Following **Typical Procedure II**, the reaction of (*S*)-**1n** (196.3 mg, 1.0 mmol, 96% ee), $[Cp^*RhCl_2]_2$ (30.9 mg, 0.05 mmol), and $Cu(OAc)_2•H_2O$ (499.3 mg, 2.5 mmol) in CH_3CN (1 mL)/MeOH (50 μL) afforded (*S*,*E*)-**2n** (130.5 mg, 67%) (eluent: petroleum ether /ethyl acetate = 50/1, 800 mL): solid; m.p. 38.5-42.7 °C (determined without recrystallization); 98% ee (HPLC conditions: Chiralcel IC column, *n*-hexane/*i*-PrOH = 99/1, 0.7 mL/min, λ = 254 nm, t_R(minor) = 13.0 min, t_R(major) = 15.1 min); $[\alpha]_D^{20}$ =

+89.3 (*c* = 1.00, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.23 (q, *J* = 1.8 Hz, 1 H, =CH), 5.80 (s, 1 H, =CH), 5.00-4.89 (m, 1 H, OCH), 4.81 (ddd, J_1 = 11.4 Hz, J_2 = 5.1 Hz, J_2 = 2.1 Hz, 1 H, one proton of OCH_2), 4.74 (ddd, J_1 = 11.4 Hz, J_2 = 3.6 Hz, J_2 = 1.8 Hz, 1 H, one proton of OCH_2), 2.46 (t, *J* = 7.4 Hz, 2 H, CH_2), 2.32 (s, 3 H, CH_3), 1.67-1.50 (m, 4 H, CH_2 × 2), 1.48-1.18 (m, 26 H, CH_2 × 13), 0.88 (t, *J* = 6.6 Hz, 6 H, CH_3 × 2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 201.8, 143.9, 141.3, 133.6, 123.5, 87.3, 74.0, 44.8, 35.7, 31.8, 29.6, 29.53, 29.50, 29.4, 29.3, 29.22, 29.19, 25.3, 24.2, 22.6, 16.1, 14.1; IR (neat) *v* (cm^{-1}) 2956, 2925, 2854, 1687, 1684, 1616, 1589, 1466, 1405, 1367, 1131, 1103, 1047; MS (EI): *m/z* (%) 390 (M^+, 8.57), 263 (100); Anal. Calcd. for $C_{26}H_{46}O_2$ (%): C, 79.94; H, 11.87; Found: C, 79.99; H, 11.52.

30. Synthesis of (*S*, *E*)-1-(4-bromophenyl)-3-(2-(4-bromophenyl)-2,5-dihydrofuran-4-yl)but-2-en-1-one (*S*, *E*)-**2f** and (*E*)-**8f**. (wxy-5-019)



Following **Typical Procedure II**, the reaction of (*S*)-**1f**[2,5] (2.2596 g, 10.0 mmol, >99% ee), $[Cp^*RhCl_2]_2$ (310.5 mg, 0.5 mmol), and $Cu(OAc)_2•H_2O$ (5.0081 g, 25 mmol) in CH_3CN (10 mL)/MeOH (0.5 mL) afforded (*S*, *E*)-**2f** (1.1240 g, 52%) and (*E*)-**8f** (31.6 mg, 1%) [eluent:

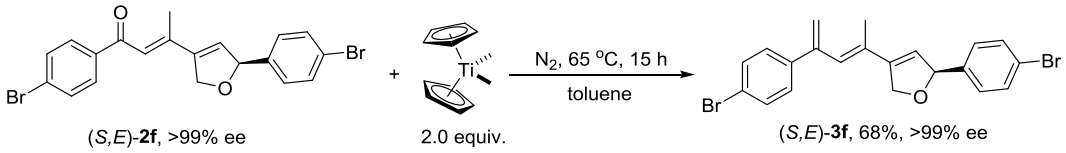
petroleum ether/ethyl acetate = 50/1 (500 mL) to petroleum ether/ethyl acetate = 20/1 (800 mL)].

(*S*, *E*)-**2f:** solid; m.p. 123.3-123.6 ^{o}C (petroleum ether/CH_2Cl_2); >99% ee (HPLC conditions: Chiralcel IC column, *n*-hexane/*i*-PrOH = 90/10, 0.8 mL/min, λ = 254 nm, t_R(major) = 18.9 min, t_R(minor) = 15.7 min); $[\alpha]_D^{20}$ = -344.3 (*c* = 0.535, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.78 (d, *J* = 8.4 Hz, 2 H, ArH), 7.61 (d, *J* = 8.7 Hz, 2 H, ArH), 7.50 (d, *J* = 8.4 Hz, 2 H, ArH), 7.21 (d, *J* = 8.4 Hz, 2 H, ArH), 6.50 (s, 1 H, =CH), 6.31 (q, *J* = 1.8 Hz, 1 H, =CH), 5.98-5.82 (m, 1 H, OCH), 5.14 (ddd, J_1 = 11.4 Hz, J_2 = 5.4 Hz, J_3 = 1.8 Hz, 1 H, one proton of OCH_2), 5.01 (ddd, J_1 = 11.4 Hz, J_2 = 3.6 Hz, J_3 = 2.0 Hz, 1 H, one proton of OCH_2), 2.35 (d, *J* = 0.9 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 190.7, 145.4, 141.7, 140.0, 137.6, 132.7, 131.9, 131.8, 129.7, 128.0, 127.9, 122.0, 121.8, 88.4, 74.9, 16.7; IR (KBr) *v* (cm^{-1}) 3089, 2855, 1654, 1614, 1591, 1246, 1216, 1111, 1093, 1070, 1050, 1006; MS (EI): *m/z* (%) 450 [($M(^{81}Br^{81}Br)^+$, 3.46], 448 [($M(^{81}Br^{79}Br)^+$, 2.46], 446 [($M(^{79}Br^{79}Br)^+$, 0.77], 183 (100); Anal. Calcd. for $C_{20}H_{16}Br_2O_2$ (%): C, 53.60; H, 3.60; Found: C, 53.36; H, 3.52.

(*E*)-**8f:** solid; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.84 (d, *J* = 8.7 Hz, 2 H, ArH), 7.77 (s, 1 H, ArH), 7.63-7.28 (m, 6 H, ArH and =CH), 7.10 (s, 1 H, =CH), 6.92 (s, 1 H, =CH), 2.52 (s, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 190.3, 154.4, 146.6, 142.4, 138.2, 132.0, 131.8, 130.6, 129.7, 128.9, 127.5, 125.5, 122.0, 118.6, 102.7, 17.6.

Synthetic Applications

1. Preparation of (*S*,*E*)-**3f.** (wxy-5-038)

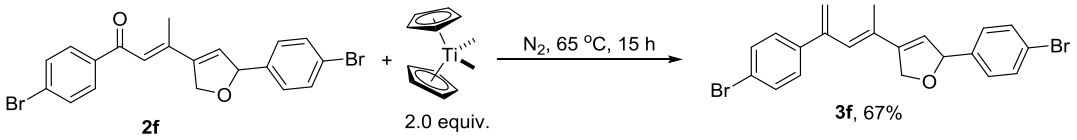


(*S*,*E*)-**2f**, >99% ee 2.0 equiv. (*S*,*E*)-**3f**, 68%, >99% ee

Typical Procedure III: To a Schlenk tube were added (*S*,*E*)-**2f** (89.5 mg, 0.2 mmol), evacuated and backfilled with nitrogen three times. Then, $CpTiMe_2$ (0.4 mmol, 4 mL, 0.1 M in toluene, which was prepared according to literature[7]) was added via a syringe. The reaction tube was put into a pre-heated 65 ^oC oil bath. The reaction was complete after 15 h as monitored by TLC (eluent: petroleum ether/ethyl acetate = 10/1). Ethyl acetate (5 mL) was added to dilute the reaction mixture, which was filtered through a celite pad and eluted with ethyl acetate (20 mL × 3). The combined filtrate was then concentrated in vacuo and the crude residue was purified via chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 50/1, 300 mL) to afford (*S*,*E*)-**3f** (60.7 mg, 68%): solid; 62.6-64.3 ^oC (determined without recrystallization); >99% ee (HPLC conditions: Chiralcel IA column, *n*-hexane/*i*-PrOH = 90/10, 0.7 mL/min, λ = 254 nm, t_R(major) = 9.6 min, t_R(minor) = 10.7 min); $[\alpha]_D^{20}$ = -196.5 (c = 0.46, $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 7.56-7.36 (m, 4 H, ArH), 7.30-7.12 (m, 4 H, ArH), 5.97 (s, 1 H, =CH), 5.92-5.76 (m, 2 H, =CH and OCH), 5.63 (s, 1 H, one proton of $=CH_2$), 5.21 (s, 1 H, one proton of $=CH_2$), 5.16-5.04 (m, 1 H, one proton of OCH_2), 5.02-4.90 (m, 1 H, one proton of OCH_2), 1.89 (d, *J* = 0.9 Hz, 3 H, Me); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.5, 141.9, 141.1, 139.5, 131.9, 131.6, 131.5, 128.4, 128.2, 128.0, 126.0, 121.8, 121.6, 117.0, 88.1, 75.3, 16.0; IR (neat) *v* (cm^{-1}) 3088, 2950, 2920, 2850, 1484, 1403, 1390, 1108, 1100, 1087, 1070, 1052, 1008; MS (EI): *m/z* (%) 448 [(M($^{81}Br^{81}Br$)$^+$,

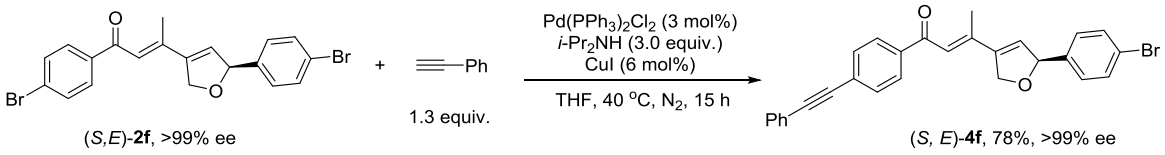
45.87], 446 [(M(^{81}Br^{79}Br)$^+$, 94.71], 444 [(M(^{79}Br^{79}Br)$^+$, 58.76], 183 (100); HRMS Cacld. for $C_{21}H_{18}O^{79}Br^{79}Br$ (M^+): 443.9724; Found: 443.9724.

2. Preparation of (±)-**3f.** (wxy-5-037)



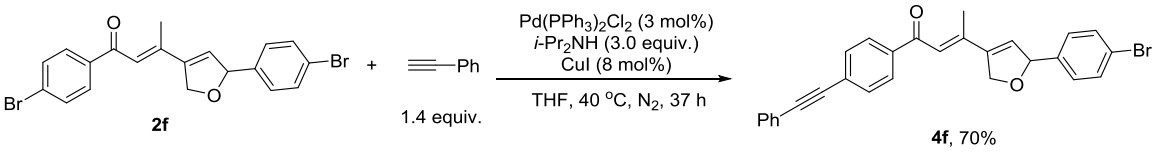
Following **Typical Procedure III**, The reaction of **2f** (89.7 mg, 0.2 mmol) and $CpTiMe_2$[7] (0.4 mmol, 4 mL, 0.1 M in toluene) afforded **3f** (60.0 mg, 67%) (eluent: petroleum ether/ethyl acetate = 50/1, 300 mL): solid; 63.7-65.9 °C (determined without recrystallization); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.52-7.43 (m, 4 H, ArH), 7.28-7.17 (m, 4 H, ArH), 5.97 (s, 1 H, =CH), 5.92-5.80 (m, 2 H, =CH and OCH), 5.64 (s, 1 H, one proton of $=CH_2$), 5.22 (s, 1 H, one proton of $=CH_2$), 5.15-5.04 (m, 1 H, one proton of OCH_2), 5.02-4.92 (m, 1 H, one proton of OCH_2), 1.90 (s, 3 H, Me); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.6, 141.9, 141.1, 139.5, 131.9, 131.6, 131.5, 128.4, 128.3, 128.1, 126.0, 121.8, 121.7, 117.0, 88.1, 75.3, 16.1; IR (neat) *v* (cm^{-1}) 3087, 2951, 2849, 1484, 1404, 1390, 1108, 1101, 1086, 1070, 1051, 1008; MS (EI): *m/z* (%) 448 [(M(^{81}Br^{81}Br)$^+$, 43.27], 446 [(M(^{81}Br^{79}Br)$^+$, 86.73], 444 [(M(^{79}Br^{79}Br)$^+$, 52.13], 183 (100); HRMS Cacld. for $C_{21}H_{18}O^{79}Br^{79}Br$ (M^+): 443.9724; Found: 443.9723.

3. Preparation of (*S,E*)-**4f.** (wxy-5-032)



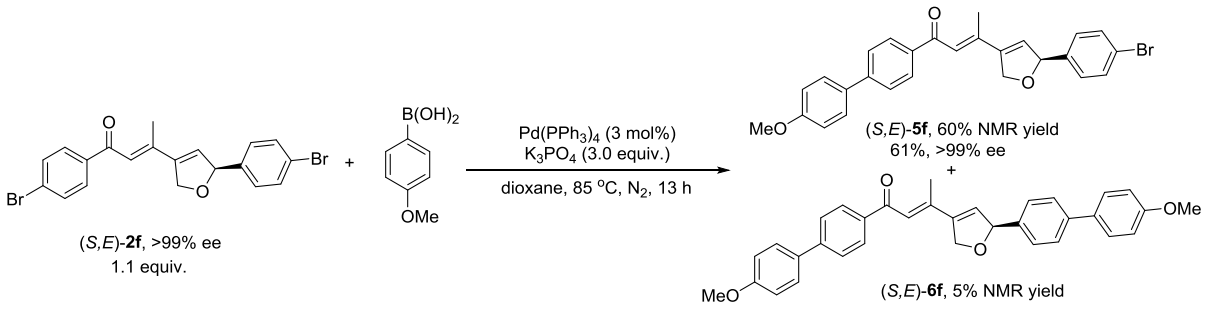
Typical Procedure IV: To a Schlenk tube were added $Pd(PPh_3)_2Cl_2$ (4.2 mg, 0.006 mmol) and CuI (2.4 mg, 0.013 mmol), evacuated and backfilled with nitrogen three times, freshly distilled THF (1.6 mL), *i*-Pr_2NH (85 μL, d = 0.717 g/mL, 60.9 mg, 0.6 mmol), (*S*)-**2f** (90.0 mg, 0.2 mmol), and phenylacetylene (28.5 μL, d = 0.93 g/mL, 26.5 mg, 0.26 mmol) were added sequentially. Then, the reaction tube was put into a pre-heated 40 ^oC oil bath. The reaction was complete after 15 h as monitored by TLC (eluent: petroleum ether/acetone = 10/1). The resulting mixture was filtered through a celite pad and eluted with ethyl acetate (20 mL × 3). The combined filtrate was then concentrated in vacuo and the crude residue was purified via chromatography on silica gel (eluent: petroleum ether/acetone = 20/1, 500 mL) to afford (*S*,*E*)-**4f** (73.2 mg, 78%): solid; m.p. 152.4-153.6 ^oC (petroleum ether/DCM); >99% ee (HPLC conditions: Chiralcel IA column, *n*-hexane/*i*-PrOH = 90/10, 1.3 mL/min, λ = 254 nm, t_R(major) = 12.8 min, t_R(minor) = 14.2 min); $[\alpha]_D^{20}$ = -426.0 (c = 0.67, $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 7.90 (d, *J* = 8.4 Hz, 2 H, ArH), 7.61 (d, *J* = 8.1 Hz, 2 H, ArH), 7.58-7.52 (m, 2 H, ArH), 7.49 (d, *J* = 8.4 Hz, 2 H, ArH), 7.42-7.31 (m, 3 H, ArH), 7.20 (d, *J* = 8.4 Hz, 2 H, ArH), 6.54 (s, 1 H, =CH), 6.28 (q, *J* = 1.5 Hz, 1 H, =CH), 5.98-5.83 (m, 1 H, =CH), 5.16 (ddd, J_1 = 11.4 Hz, J_2 = 5.4 Hz, J_3 = 1.8 Hz, 1 H, one proton of OCH_2), 5.02 (ddd, J_1 = 11.4 Hz, J_2 = 3.6 Hz, J_3 = 1.8 Hz, 1 H, one proton of OCH_2), 2.35 (s, 3 H, Me); ^{13}C NMR (75 MHz, $CDCl_3$) δ 190.8, 145.0, 141.8, 140.1, 137.9, 132.4, 131.72, 131.69, 128.8, 128.4, 128.2, 128.0, 127.9, 122.6, 122.2, 121.9, 92.7, 88.7, 88.3, 74.9, 16.7; IR (neat) *v* (cm^{-1}) 2916, 2857, 1652, 1602, 1485, 1442, 1406, 1240, 1216, 1049, 1010; MS (EI): *m/z* (%) 470 [$M(^{81}Br)^+$, 91.53], 468 [$M(^{79}Br)^+$, 75.55], 263 (100); Anal. Calcd. for $C_{28}H_{21}BrO_2$ (%): C, 71.65; H, 4.51; Found: C, 71.40; H, 4.50.

4. Preparation of (±)-**4f.** (wxy-5-026)



Following **Typical Procedure IV**, The reaction of $Pd(PPh_3)_2Cl_2$ (2.3 mg, 0.003 mmol), CuI (1.6 mg, 0.008 mmol), THF (0.8 mL), *i*-Pr_2NH (43 μL, d = 0.717 g/mL, 30.8 mg, 0.3 mmol), **2f** (45.0 mg, 0.1 mmol), and phenylacetylene (15 μL, d = 0.93 g/mL, 14.0 mg, 0.14 mmol) afforded **4f** (32.9 mg, 70%) (eluent: petroleum ether/acetone = 20/1, 500 mL): solid; m.p. 141.9-143.8 ^{o}C (petroleum ether/DCM); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.90 (d, *J* = 8.4 Hz, 2 H, ArH), 7.62 (d, *J* = 8.7 Hz, 2 H, ArH), 7.59-7.52 (m, 2 H, ArH), 7.50 (d, *J* = 8.4 Hz, 2 H, ArH), 7.42-7.30 (m, 3 H, ArH), 7.21 (d, *J* = 8.4 Hz, 2 H, ArH), 6.55 (s, 1 H, =CH), 6.30 (q, *J* = 1.5 Hz, 1 H, =CH), 5.99-5.82 (m, 1 H, OCH), 5.15 (ddd, J_1 = 11.4 Hz, J_2 = 5.4 Hz, J_3 = 1.8 Hz, 1 H, one proton of OCH_2), 5.03 (ddd, J_1 = 11.4 Hz, J_2 = 3.6 Hz, J_3 = 1.8 Hz, 1 H, one proton of OCH_2), 2.36 (s, 3 H, Me); ^{13}C NMR (75 MHz, $CDCl_3$) δ 190.9, 145.0, 141.8, 140.1, 138.0, 132.4, 131.8, 131.7, 128.8, 128.4, 128.2, 128.1, 128.0, 122.7, 122.2, 122.0, 92.8, 88.7, 88.4, 75.0, 16.7; IR (neat) *v* (cm^{-1}) 2923, 2857, 1652, 1603, 1239, 1216, 1089, 1049, 1010; MS (EI): *m/z* (%) 470 [M(^{81}Br)$^{+}$, 17.29], 468 [M(^{79}Br)$^{+}$, 13.82], 205 (100); Anal. Calcd. for $C_{28}H_{21}BrO_2$ (%): C, 71.65; H, 4.51; Found: C, 71.38; H, 4.55.

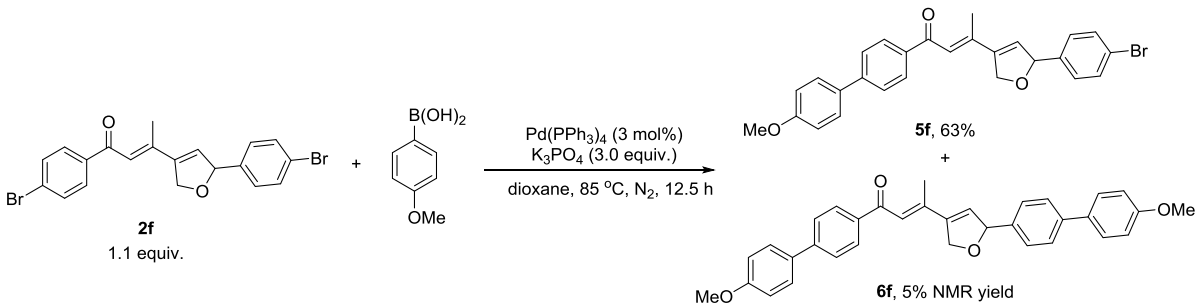
5. Preparation of (*S*,*E*)-**5f.** (wxy-5-036)



Typical Procedure V: To a Schlenk tube were added (*S*,*E*)-**2f** (98.5 mg, 0.22 mmol), (4-methoxyphenyl)boronic acid (30.5 mg, 0.2 mmol), $Pd(PPh_3)_4$ (6.9 mg, 0.006 mmol), and K_3PO_4 (126.8 mg, 0.6 mmol). After being evacuated and backfilled with nitrogen three times, dioxane (1.6 mL) was added. Then, the reaction tube was put into a pre-heated 85 ^oC oil bath. The reaction was complete after 13 h as monitored by TLC (eluent: petroleum ether/ethyl acetate = 9/1). The resulting mixture was filtered through a pad of celite and eluted with ethyl acetate (20 mL) and DCM (20 mL × 2). The combined filtrate was then concentrated in vacuo. The reaction afforded (*S*,*E*)-**5f** in 60% NMR yield together with 5% NMR of (*S*,*E*)-**6f**, which was analyzed by ^1H NMR using 7 μL of CH_2Br_2 as the internal standard. The crude residue was then purified via chromatography on silica gel [eluent: petroleum ether/ ethyl acetate = 9/1 (500 mL) to petroleum ether/ ethyl acetate = 6/1 (350 mL)] to afford (*S*,*E*)-**5f** (57.8 mg, 61%): solid; m.p. 143.2-145.0 ^oC (petroleum ether/DCM); >99% ee (HPLC conditions: Chiralcel IA column, *n*-hexane/*i*-PrOH = 80/20, 1.3 mL/min, λ = 254 nm, t_R(major) = 12.9 min, t_R(minor) = 16.4 min); $[\alpha]_D^{20}$ = -288.1 (c = 0.695, $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 7.97 (d, *J* = 8.7 Hz, 2 H, ArH), 7.66 (d, *J* = 8.1 Hz, 2 H, ArH), 7.59 (d, *J* = 8.7 Hz, 2 H, ArH), 7.50 (d, *J* = 8.4 Hz, 2 H, ArH), 7.23 (t, *J* = 8.0 Hz, 2 H, ArH), 7.01 (d, *J* = 8.7 Hz, 2 H, ArH), 6.60 (s, 1 H, =CH), 6.29 (d, *J* = 1.8 Hz, 1 H, =CH), 6.00-5.80 (m, 1 H, OCH), 5.17 (ddd, J_1 = 11.4 Hz, J_2 = 5.4 Hz, J_3 = 2.4 Hz, 1 H, one proton of OCH_2), 5.04 (ddd, J_1 = 11.4

Hz, J_2 = 3.3 Hz, J_3 = 1.8 Hz, 1 H, one proton of OCH_2), 3.87 (s, 3 H, OMe), 2.37 (s, 3 H, Me); ^{13}C NMR (75 MHz, $CDCl_3$) δ 191.4, 160.0, 145.2, 144.3, 141.9, 140.2, 136.9, 132.3, 132.0, 131.8, 128.9, 128.4, 128.1, 126.7, 122.7, 122.0, 114.4, 88.4, 75.0, 55.4, 16.7; IR (neat) ν (cm^{-1}) 3002, 2951, 2865, 2836, 1642, 1617, 1599, 1579, 1524, 1496, 1301, 1277, 1256, 1221, 1193, 1180, 1052, 1037, 1011, 1001; MS (EI): *m/z* (%) 476 [$M(^{81}Br)^+$, 16.52], 474 [$M(^{79}Br)^+$, 14.50], 211 (100); Anal. Calcd. for $C_{27}H_{23}BrO_3$ (%): C, 68.22; H, 4.88; Found: C, 68.40; H, 4.74.

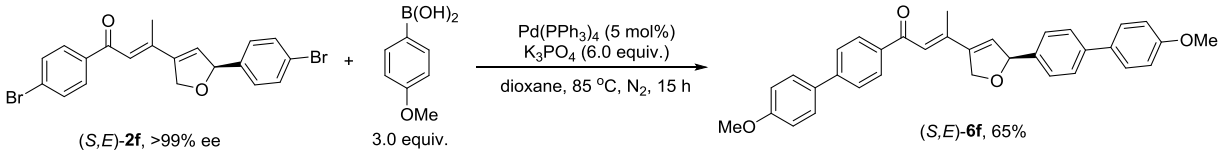
6. Preparation of (±)-**5f.** (wxy-5-007, wxy-5-029)



Following **Typical Procedure V:** The reaction of **2f** (49.4 mg, 0.11 mmol), (4-methoxyphenyl)boronic acid (15.2 mg, 0.1 mmol), $Pd(PPh_3)_4$ (3.6 mg, 0.003 mmol), K_3PO_4 (63.7 mg, 0.3 mmol), dioxane (0.8 mL) afforded (*S,E*)-**5f** in 60% NMR yield and (*S,E*)-**6f** in 5% NMR yield. The crude residue was then purified via chromatography on silica gel (eluent: petroleum ether/ ethyl acetate = 10/1, 1000 mL) to afford **5f** (29.9 mg, 63%): solid; m.p. 171.7-172.7 °C (petroleum ether/DCM); ^1H NMR (300 MHz, $CDCl_3$) δ 7.97 (d, *J* = 8.7 Hz, 2 H, ArH), 7.66 (d, *J* = 8.1 Hz, 2 H, ArH), 7.59 (d, *J* = 8.7 Hz, 2 H, ArH), 7.50 (d, *J* = 8.4 Hz, 2 H, ArH), 7.23 (t, *J* = 8.1 Hz, 2 H, ArH), 7.01 (d, *J* = 9.0 Hz, 2 H, ArH), 6.60 (s, 1 H, =CH), 6.28 (q, *J* = 1.5 Hz, 1 H, =CH), 5.95-5.87 (m, 1 H, OCH), 5.17 (ddd, J_1 = 11.4 Hz,

J_2 = 5.4 Hz, J_3 = 2.4 Hz, 1 H, one proton of OCH_2), 5.04 (ddd, J_1 = 11.4 Hz, J_2 = 3.3 Hz, J_3 = 1.8 Hz, 1 H, one proton of OCH_2), 3.87 (s, 3 H, OMe), 2.37 (s, 3 H, Me); ^{13}C NMR (75 MHz, $CDCl_3$) δ 191.4, 160.0, 145.2, 144.3, 141.9, 140.2, 136.9, 132.3, 132.0, 131.8, 128.9, 128.4, 128.1, 126.7, 122.7, 122.0, 114.4, 88.4, 75.0, 55.4, 16.7; IR (neat) *v* (cm^{-1}) 3002, 2955, 2856, 2836, 1642, 1616, 1599, 1577, 1525, 1496, 1301, 1277, 1256, 1221, 1193, 1181, 1052, 1037, 1011, 1001; MS (EI): *m/z* (%) 476 [M(^{81}Br)$^+$, 22.56], 474 [M(^{79}Br)$^+$, 21.12], 263 (100); Anal. Calcd. for $C_{27}H_{23}BrO_3$ (%): C, 68.22; H, 4.88; Found: C, 68.58; H, 4.88.

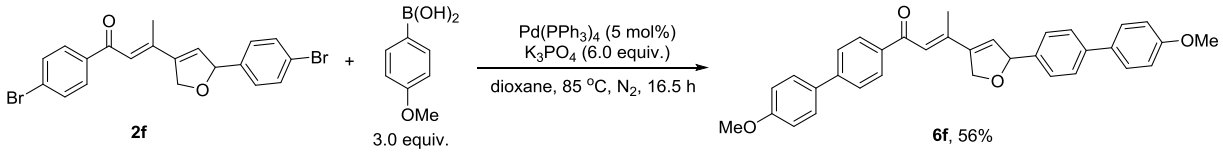
7. Preparation of (*S*,*E*)-**6f.** (wxy-5-031)



Following**Typical Procedure V:** The reaction of (*S*,*E*)-**2f** (89.9 mg, 0.2 mmol), (4-methoxyphenyl)boronic acid (90.8 mg, 0.6 mmol), $Pd(PPh_3)_4$ (11.5 mg, 0.01 mmol), K_3PO_4 (253.9 mg, 1.2 mmol), dioxane (2.4 mL) afforded (*S*,*E*)-**6f** (65.1 mg, 65%) [eluent: petroleum ether/ acetone = 9/1 (500 mL) to petroleum ether/ acetone = 4/1 (500 mL) to DCM (300 mL)]: solid; 185 °C (decompose); The ee of (*S*,*E*)-**6f** could not be determinded by the HPLC conditions with the commercially available chiral columns. $[\alpha]_D^{20}$ = -387.5 (c = 0.32, $CHCl_3$); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.98 (d, *J* = 8.7 Hz, 2 H, ArH), 7.66 (d, *J* = 8.4 Hz, 2 H, ArH), 7.61-7.42 (m, 6 H, ArH), 7.39 (d, *J* = 8.1 Hz, 2 H, ArH), 6.99 (t, *J* = 8.9 Hz, 4 H, ArH), 6.62 (s, 1 H, =CH), 6.37 (q, *J* = 1.5 Hz, 1 H, =CH), 6.05-5.94 (m, 1 H, OCH), 5.17 (m, 1 H, one proton of OCH_2), 5.06 (m, 1 H, one proton of OCH_2), 3.86 (s, 3 H, OMe), 3.85 (s, 3 H, Me), 2.39 (s, 3 H, Me); ^{13}C NMR (75 MHz, $CDCl_3$) δ 191.4, 160.0, 159.3, 145.1, 144.7,

141.7, 140.8, 139.5, 137.1, 133.3, 132.6, 132.3, 128.9, 128.4, 128.1, 127.0, 126.9, 126.7, 122.4, 114.4, 114.2, 88.9, 74.9, 55.4, 55.3, 16.7; IR (neat) ν (cm^{-1}) 2955, 2834, 1651, 1601, 1525, 1497, 1294, 1274, 1250, 1190, 1038; MS (EI): *m/z* (%) 502 (M^+, 37.65), 211 (100); HRMS Cacld. for $C_{34}H_{30}O_4$ (M^+): 502.2144; Found: 502.2143.

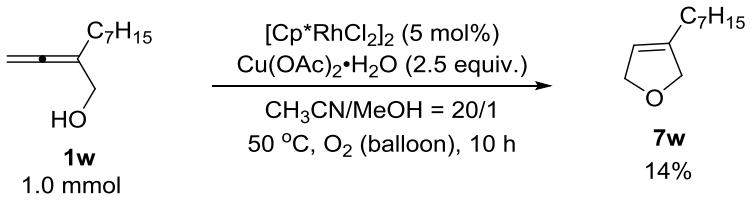
8. Preparation of (±)-**6f.** (wxy-5-024)



Following**Typical Procedure V:** The reaction of **2f** (179.5 mg, 0.4 mmol), (4-methoxyphenyl)boronic acid (182.1 mg, 1.2 mmol), $Pd(PPh_3)_4$ (23.1 mg, 0.02 mmol), K_3PO_4 (509.0 mg, 2.4 mmol), and dioxane (4.8 mL) afforded **6f** (120.1 mg, 56%, 93% purity) [eluent: petroleum ether/acetone = 9/1 (500 mL) to petroleum ether/acetone = 4/1 (500 mL) to DCM (200 mL)]: solid; 190 °C (decompose); ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.98 (d, *J* = 8.1 Hz, 2 H, ArH), 7.66 (d, *J* = 8.1 Hz, 2 H, ArH), 7.61-7.47 (m, 6 H, ArH), 7.39 (d, *J* = 8.1 Hz, 2 H, ArH), 6.99 (t, *J* = 8.9 Hz, 4 H, ArH), 6.62 (s, 1 H, =CH), 6.37 (q, *J* = 1.2 Hz, 1 H, =CH), 6.04-5.95 (m, 1 H, OCH), 5.26-5.15 (m, 1 H, one proton of OCH_2), 5.12-5.00 (m, 1 H, one proton of OCH_2), 3.87 (s, 3 H, OMe), 3.85 (s, 3 H, Me), 2.40 (s, 3 H, Me); ^{13}C NMR (75 MHz, $CDCl_3$) δ 191.5, 160.0, 159.3, 145.1, 144.7, 141.7, 140.8, 139.5, 137.1, 133.3, 132.6, 132.3, 128.9, 128.4, 128.1, 127.0, 126.9, 126.7, 122.4, 114.4, 114.3, 88.9, 74.9, 55.4, 55.3, 16.7; IR (neat) ν (cm^{-1}) 2958, 2929, 2837, 1651, 1600, 1525, 1497, 1465, 1441, 1294, 1274, 1249, 1221, 1190, 1038; MS (EI): *m/z* (%) 502 (M^+, 35.03), 211 (100); HRMS Cacld. for $C_{34}H_{30}O_4$ (M^+): 502.2144; Found: 502.2146.

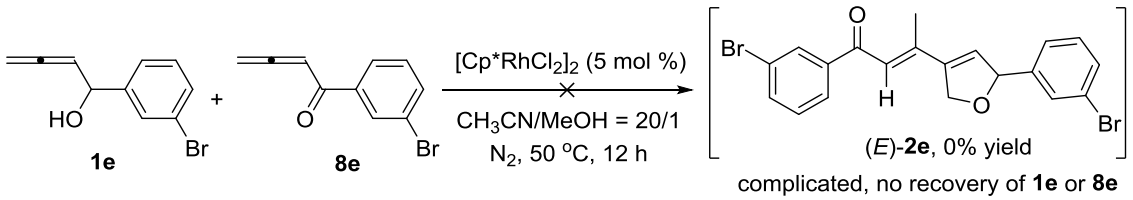
Mechanistic Studies

1. Synthesis of 3-heptyl-2,5-dihydrofuran **7w**. (wxy-1-146)



Following **Typical Procedure II**, the reaction of **1w**[8] (168.3 mg, 1.0 mmol), $[Cp^*RhCl_2]_2$ (30.9 mg, 0.05 mmol), and $Cu(OAc)_2•H_2O$ (500.3 mg, 2.5 mmol) in CH_3CN (1.0 mL)/MeOH (50 μL) afforded **7w** (23.1 mg, 14%, 98% purity) (eluent: petroleum ether/ethyl acetate = 50/1, 600 mL): liquid; ^1H NMR (300 MHz, $CDCl_3$) δ 5.48-5.43 (m, 1 H =CH), 4.66-4.58 (m, 2 H, OCH_2), 4.56-4.48 (m, 2 H, OCH_2), 2.08 (t, *J* = 7.1 Hz, 2 H, CH_2), 1.55-1.39 (m, 2 H, CH_2), 1.35-1.20 (m, 8 H, CH_2 × 4), 0.89 (t, *J* = 6.8 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 140.7, 118.6, 77.03, 75.9, 31.7, 29.4, 29.1, 27.6, 27.1, 22.6, 14.0; IR (neat) *v* (cm^{-1}) 2956, 2927, 2855, 1662, 1467, 1378, 1163, 1073; MS (EI): *m/z* (%) 168 (M^+, 5.57), 69 (100); HRMS Cacld. for $C_{11}H_{20}O$ (M^+): 168.1514; Found: 168.1512.

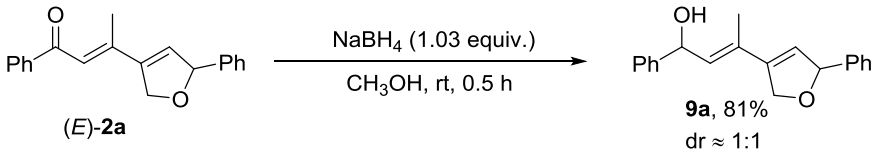
2. The reaction of **1e** with **8e** without $Cu(OAc)_2•H_2O$ and O_2. (wxy-3-027)



To a dried Schlenk tube were added $[Cp^*RhCl_2]_2$ (5.1 mg, 0.008 mmol), **8e** (111.8 mg, 0.5 mmol), and allenol **1e** (112.8 mg, 0.5 mmol) under N_2 atmosphere subsequently. After being stirred for 12 h at 50 °C, the resulting mixture was filtrated through a short column of silica gel (eluent with ethyl acetate 20 mL × 3). After evaporation of the solvent, the crude product

was analyzed by ^{1}H NMR spectrum with mesitylene as an internal standard. No signal of the corresponding products was found.

3. Preparation of **9a**. (wxy-3-158)

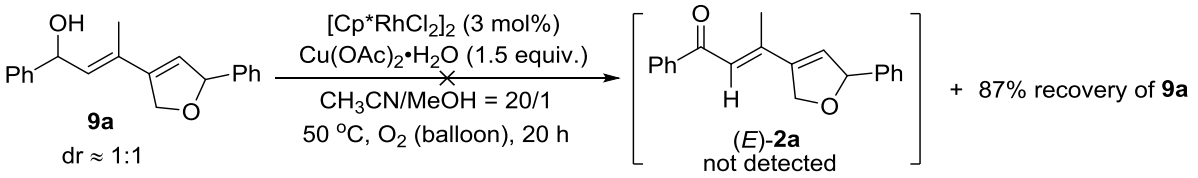


To a Schlenk tube were added (*E*)-**2a** (58.0 mg, 0.2 mmol), MeOH (3.0 mL), $NaBH_4$ (7.8 mg, 0.206 mmol) sequentially. The reaction was complete after 0.5 h as monitored by TLC (eluent: petroleum ether/ethyl acetate = 5/1). The resulting mixture was added an aqueous of saturated NH_4Cl and extracted with ethyl acetate (5 mL × 3). After evaporation, 15 μL mesitylene was added. 86% NMR yield of **9a** (dr ≈ 1:1) was was determined based on ^{1}H NMR analysis of the crude product with mesitylene as the internal standard. Then, the residue was purified via chromatography on silica gel [eluent: petroleum ether/ethyl acetate = 10/1 (1000 mL) to petroleum ether/ethyl acetate = 6/1 (420 mL)] afforded the diastereoisomers **9a**.

The less polar compound (24.5 mg, 92% purity, 39% yield): oil; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.42-7.22 (m, 10 H, ArH), 5.88-5.78 (m, 2 H, CH and =CH), 5.57 (d, J = 8.4 Hz, 1 H, =CH), 5.47 (d, J = 8.4 Hz, 1 H, OCH), 5.02 (ddd, J_1 = 11.4 Hz, J_2 = 5.4 Hz, J_3 = 1.8 Hz, 1 H, one proton of OCH_2), 4.82 (ddd, J_1 = 11.4 Hz, J_2 = 3.2 Hz, J_3 = 2.0 Hz, 1 H, one proton of OCH_2), 2.22 (s, 1 H, OH), 1.98 (d, J = 0.9 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.2, 141.9, 141.1, 131.5, 129.6, 128.7, 128.5, 127.9, 127.7, 126.4, 126.3, 125.9, 88.8, 75.2, 70.6, 14.8; IR (neat) ν (cm^{-1}) 3396, 3029, 2853, 1615, 1601, 1492, 1454, 1071, 1051, 1008; MS (EI): *m/z* (%) 292.4 (M^+, 2.98), 105 (100); HRMS Calcd for $C_{20}H_{20}NaO_2$ $(M+Na)^+$: 315.1361; Found: 315.1357.

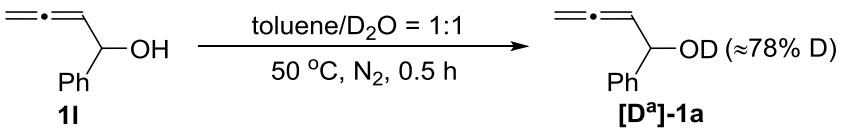
The more polar compound (25.7 mg, 95% purity, 42% yield): oil; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.45-7.20 (m, 10 H, ArH), 5.94-5.80 (m, 2 H, OCH and =CH), 5.58 (d, J = 8.4 Hz, 1 H, =CH), 5.48 (d, J = 8.4 Hz, 1 H, OCH), 4.97 (ddd, J_1 = 11.4 Hz, J_2 = 5.4 Hz, J_3 = 2.1 Hz, 1 H, one proton of OCH_2), 4.89 (ddd, J_1 = 11.4 Hz, J_2 = 3.5 Hz, J_3 = 2.3 Hz, 1 H, one proton of OCH_2), 2.20 (s, 1 H, OH), 1.98 (d, J = 0.6 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.2, 141.9, 141.0, 131.5, 129.6, 128.7, 128.5, 127.8, 127.7, 126.3, 125.9, 88.7, 75.1, 70.6, 14.8; IR (neat) v (cm^{-1}) 3406, 3029, 2852, 1611, 1599, 1492, 1454, 1265, 1195, 1071, 1051, 1010; MS (EI): *m/z* (%) 292.4 (M^+, 2.92), 105 (100); HRMS Calcd for $C_{20}H_{20}NaO_2$ $(M+Na)^+$: 315.1361; Found: 315.1356.

4. The reaction of **9a** under standard conditions. (wxy-3-168)



To a dried Schlenk tube were added $[Cp^*RhCl_2]_2$ (3.2 mg, 0.005 mmol) and $Cu(OAc)_2•H_2O$ (49.9 mg, 0.25 mmol) under air atmosphere. The Schlenk tube was then degassed to remove the air inside completely, and refilled with O_2 by a balloon of O_2 for three times. After **9a** (dr ≈ 1:1, 49.3 mg, 0.17 mmol)/CH_3CN (0.4 mL), and MeOH (20 μL) were added sequentially, the reaction tube was put into a pre-heated 50 °C oil bath. The reaction was stirred for 20 h. Then, the reaction was filtered through a short column of silica gel and eluted with ethyl acetate (20 mL × 3). The combined filtrate was then concentrated in vacuo and the crude residue was taken for NMR analysis with mesitylene (15.5 μL) as an internal standard. No signal of the (*E*)-**2a** was found, and 87% recovery of **9a** was determined.

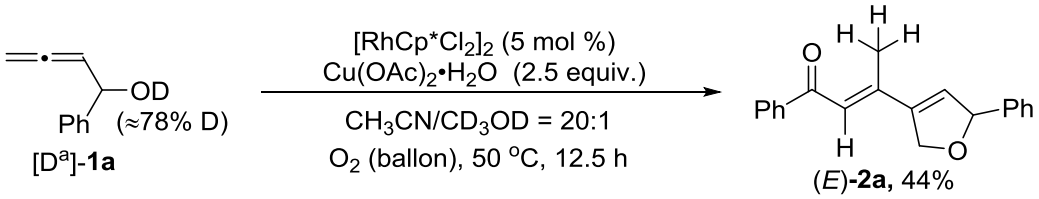
5. Preparation of [D^a]-**1a** (wxy-4-029).



1l **[D^a]-1a**

To a dried flask was added **1a** (220.3 mg, 1.5 mmol), freshly distilled toluene (0.5 mL), and D_2O (0.5 mL) sequentially under nitrogen atmosphere. Then, the flask was put into an oil bath preheated to 50 ^oC. After being stired for 30 min, the solvent was evaporated by vacuum carefully. Freshly distilled toluene (0.5 mL) and D_2O (0.5 mL) was added to the flask for the second time, and the solvent was evaporated again. The same operation was repeated for three times. After being dried under vacuum line completely, 220.0 mg [D^a]-**1a** was obtained with about 78% deuterated ratio: oil; ^1H NMR (300 MHz, $CDCl_3$) δ 7.45-7.25 (m, 5 H, ArH), 5.44 (q, *J* = 6.6 Hz, 1 H, =CH), 5.32-5.22 (m, 1 H, OCH), 4.92 (m, 2 H, =CH_2), the following signals is disscernible for **1a**: δ 2.27 (s, 0.22 H, OH); ^{13}C NMR (75 MHz, $CDCl_3$) δ 207.1, 142.7, 128.4, 127.7, 126.0, 95.0, 78.0. 71.8; IR (neat) *v* (cm^{-1}) 3364, 3063, 3030, 2886, 1955, 1494, 1452, 1024; MS (EI): *m/z* (%) 147 (M^+, 1.65), 107 (100); HRMS Cacld. for $C_{10}H_9DO$ (M^+): 147.0794; Found: 147.0792.

6. Deuterium-labeling experiments with [D^a]-**1a** (wxy-4-029).

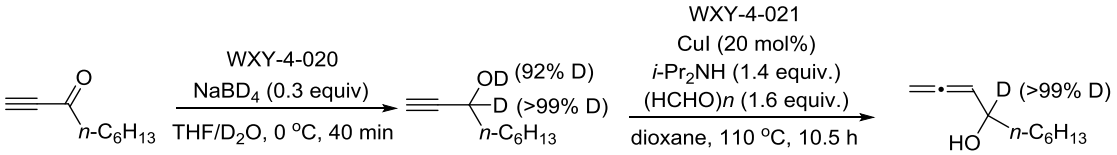


[D^a]-**1a** (*E*)-**2a**, 44%

Following **Typical Procedure II**, the reaction of [D^a]-**1a** (146.9 mg, 1.0 mmol), $[Cp^*RhCl_2]_2$ (31.0 mg, 0.05 mmol), and $Cu(OAc)_2•H_2O$ (499.9 mg, 2.5 mmol) in CH_3CN (1.0 mL)/CD_3OD (50 μL) afforded (*E*)-**2a** (63.9 mg, 44%) (eluent: petroleum ether/ethyl acetate 20/1, 700 mL): oil; ^1H NMR (300 MHz, $CDCl_3$) δ 7.96-7.88 (m, 2 H, ArH), 7.60-7.52

(m, 1 H, ArH), 7.52-7.42 (m, 2 H, ArH), 7.42-7.26 (m, 5 H, ArH), 6.57 (s, 1 H, =CH), 6.34 (q, J = 1.8 Hz, 1 H, =CH), 6.00-5.93 (m, 1 H, OCH), 5.17 (ddd, J_1 = 11.4 Hz, J_2 = 5.3 Hz, J_3 = 1.5 Hz, 1 H, one proton of OCH_2), 5.03 (ddd, J_1 = 11.4 Hz, J_2 = 3.6 Hz, J_3 = 2.1 Hz, 1 H, one proton of OCH_2), 2.37 (s, 3 H, Me); ^{13}C NMR (75 MHz, $CDCl_3$) δ 191.9, 144.9, 141.4, 141.0, 138.8, 132.9, 132.7, 128.6, 128.5, 128.2, 128.1, 126.3, 122.2, 89.0, 74.9, 16.6.

7. Preparation of [D^b]-**1m**. (wxy-4-020, wxy-4-021)



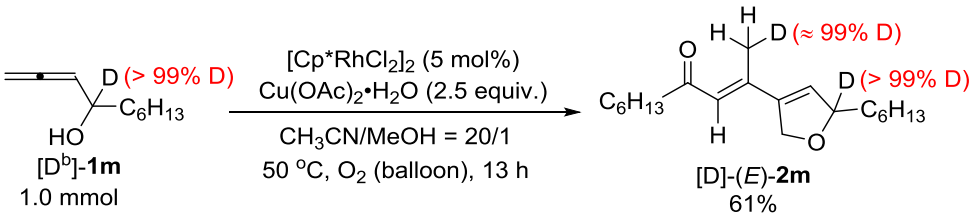
10m, 89% [D]-**11m**, 76% [D^b]-**1m**, 61%

Preparation of [D]-**11m** (wxy-4-020): To a dried three-necked flask were added **10m**[6a] (3.3012 g, 23.9 mmol), freshly distilled THF (28 mL), and D_2O (3 mL) under nitrogen atmosphere. Then, $NaBD_4$ (302.5 mg, 7.2 mmol) was added in small portions under 0 °C. After being stirred for 40 min at 0 °C, the reaction mixture was added 20 mL of saturated NH_4Cl aqueous solution, extracted with Et_2O (20 mL ×3). Organic phase was combined and dried with Na_2SO_4. After filtration and concentration, the residue was purified via column chromatography on silica gel afforded [D]-**11m** (2.2596 g, 76%) [The crude product was purified via column chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 20/1, 1000 mL) to afford a part of pure [D]-**11m** and impure [D]-**11m** (determined by TLC). The impure part was further purified via column chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 20/1, 500 mL) to afford another part of pure [D]-**11m**]: oil; ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.49 (s, 1 H, ≡CH), 1.80-1.60 (m, 2 H, CH_2), 1.55-1.20 (m, 8 H, CH_2 × 4), 0.89 (t, J = 6.6 Hz, 3 H, CH_3), the following signal is disscernible for **11m**: δ 2.13 (s, 0.08 H, OH); ^{13}C NMR (75 MHz, $CDCl_3$) δ 84.6 (t, J = 7.2 Hz), 72.7 (t, J = 19.3 Hz), 61.8

(t, J = 22.4 Hz), 37.4, 31.6, 28.8, 24.9, 22.5, 14.0, the following signals are disscernible for **11m**: δ 85.0, 71.9; IR (neat) ν (cm^{-1}) 3368, 2954, 2927, 2858, 2597, 1466, 1393, 1063; MS (EI): *m/z* (%) 142 (M^+, 0.03), 140 [$(M-D)^+$, 0.14], 57 [$(M-C_6H_{13})^+$, 59.4], 43 (100); HRMS Cacld. for $C_9H_{14}D_2O$ (M^+): 142.1327; Found: 142.1330.

Preparation of [D^b]-**1m** (wxy-4-021): Following **Typical Procedure I**, The reaction of CuI (388.0 mg, 2.0 mmol), [D]-**11m** (1.4230 g, 10.0 mmol), paraformaldehyde (1.4420 g, 16.0 mmol), and *i*-Pr_2NH (2.0 mL, d = 0.716 g/mL, 1.4320 g, 14.2 mmol) in dioxane (15 mL) afforded [D^b]-**1m** (0.9519 g, 61%, >99% D) (eluent: petroleum ether/ethyl acetate = 25/1, 1000 mL) as an oil: ^{1}H NMR (300 MHz, $CDCl_3$) δ 5.24 (t, J = 6.6 Hz, 1 H, =CH), 4.85 (d, J = 6.6 Hz, 2 H, $=CH_2$), 1.70 (s, 1 H, OH), 1.64-1.15 (m, 10 H, CH_2 × 5), 0.89 (t, J = 6.6 Hz, 3 H, CH_3); ^{13}C NMR (75 MHz, $CDCl_3$) δ 207.0, 94.8, 77.3, 69.3 (t, J = 22.1 Hz), 37.4, 31.8, 29.1, 25.3, 22.6, 14.0; IR (neat) ν (cm^{-1}) 3347, 2956, 2928, 2857, 1958, 1463, 1049; MS (EI): *m/z* (%) 116 [$(M-CH_2=C=CH)^+$, 19.6], 70 (100); HRMS Cacld. for $C_{10}H_{17}DO$ (M^+): 155.1420; Found: 155.1418.

8. Deuterium-labeling experiments with [D^b]-**1m** (wxy-4-024).



Following **Typical Procedure II**, the reaction of [D^b]-**1m** (155.2 mg, 1.0 mmol), $[Cp^*RhCl_2]_2$ (30.9 mg, 0.05 mmol), and $Cu(OAc)_2•H_2O$ (499.9 mg, 2.5 mmol) in CH_3CN (1.0 mL)/MeOH (50 μL) afforded [D]-(*E*)-**2m** (93.3 mg, 61%) (eluent: petroleum ether/ethyl

acetate 25/1, 500 mL): oil; ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.23 (s, 1 H, =CH), 5.81 (s, 1 H, =CH), 4.78 (qd, J_1 = 11.4 Hz, J_2 = 1.7 Hz, 2 H, OCH_2), 2.47 (t, J = 7.4 Hz, 2 H, CH_2), 2.29 (s, 2 H, CH_2D), 1.66-1.50 (m, 4 H, CH_2 × 2), 1.45-1.20 (m, 14 H, CH_2 × 7), 0.88 (t, J = 6.9 Hz, 6 H, Me × 2); ^{13}C NMR (75 MHz, $CDCl_3$) δ 201.7, 143.8, 141.3, 133.5, 123.5, 86.8 (t, J = 22.1 Hz), 73.9, 44.7, 35.6, 31.7, 31.5, 29.3, 28.8, 25.2, 24.1, 22.5, 22.4, 15.8 (t, J = 19.7 Hz), 14.0, 13.9; IR (neat) v (cm^{-1}) 2955, 2928, 2856, 1682, 1615, 1586, 1466, 1133, 1076; MS (EI): *m/z* (%) 308.5 (M^+, 6.3), 223 (100); HRMS Cacld. for $C_{20}H_{32}D_2O_2$ (M^+): 308.2684; Found: 308.2686.

References:

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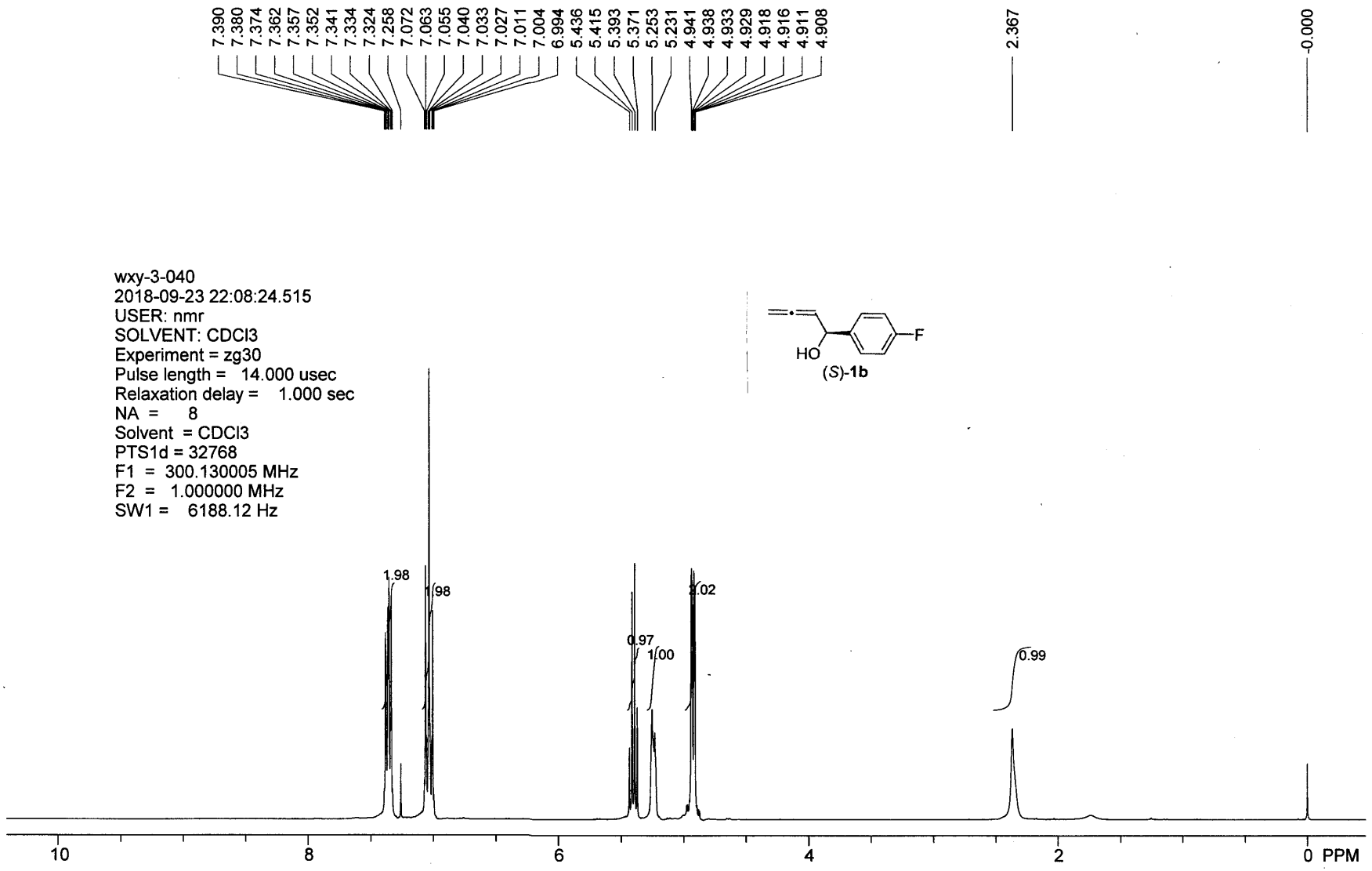
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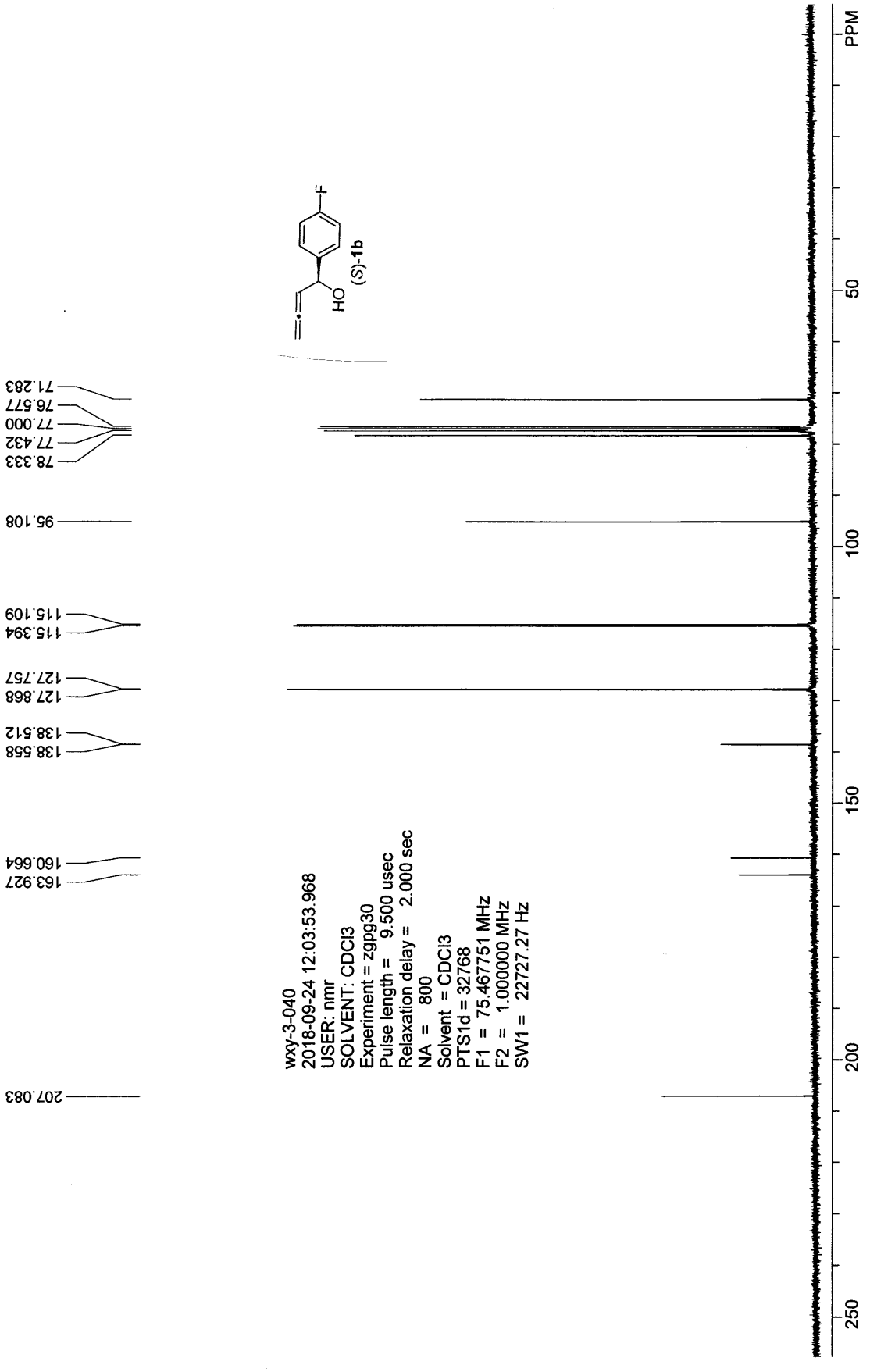
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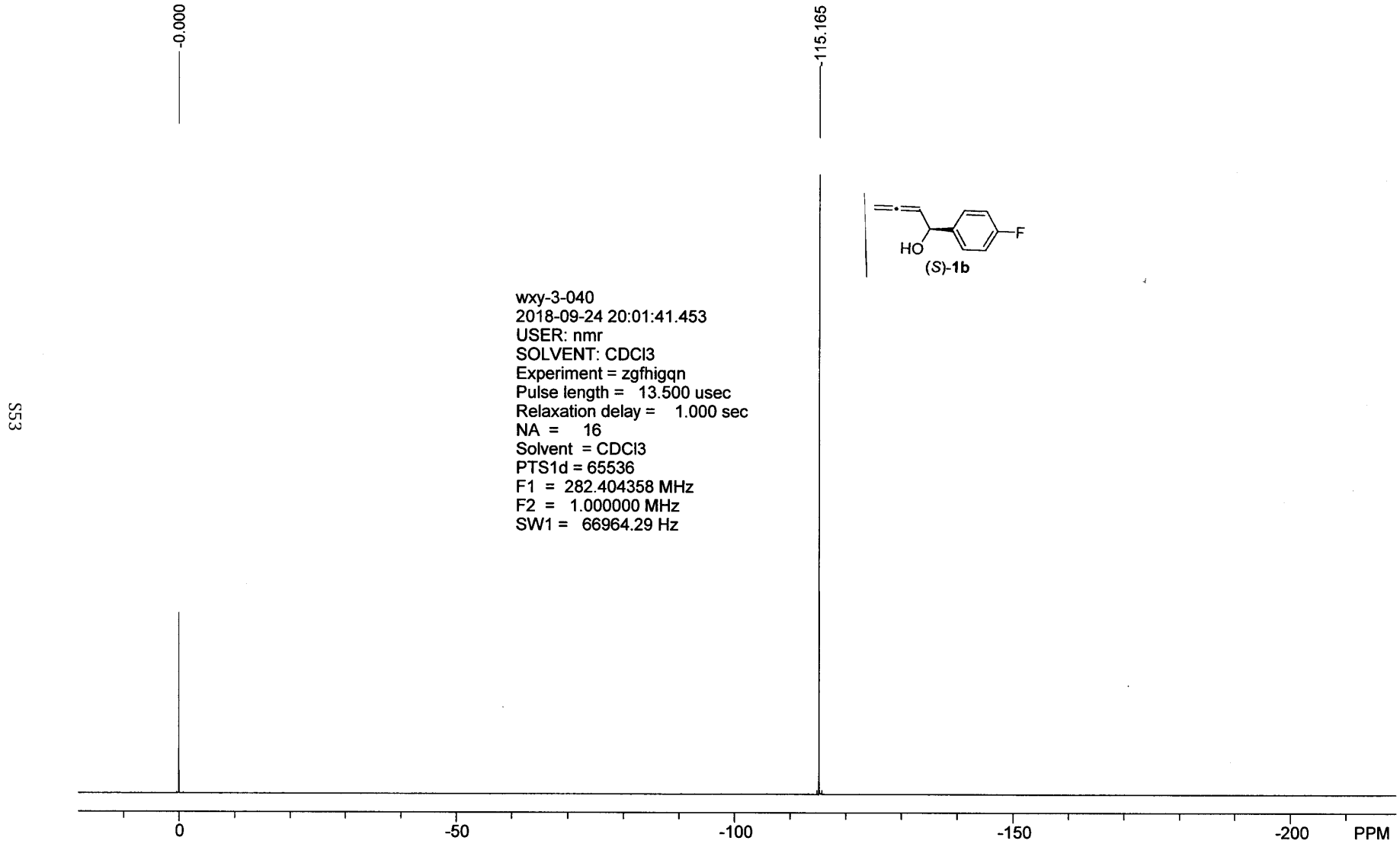
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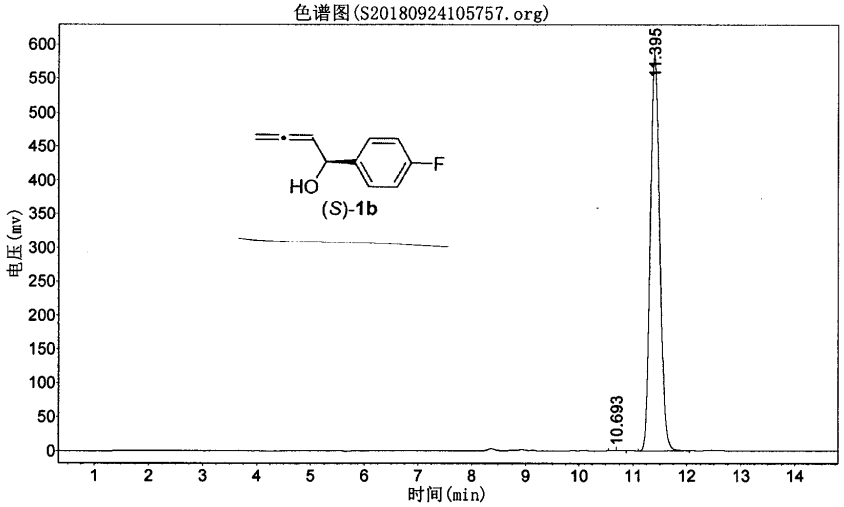

分析结果表

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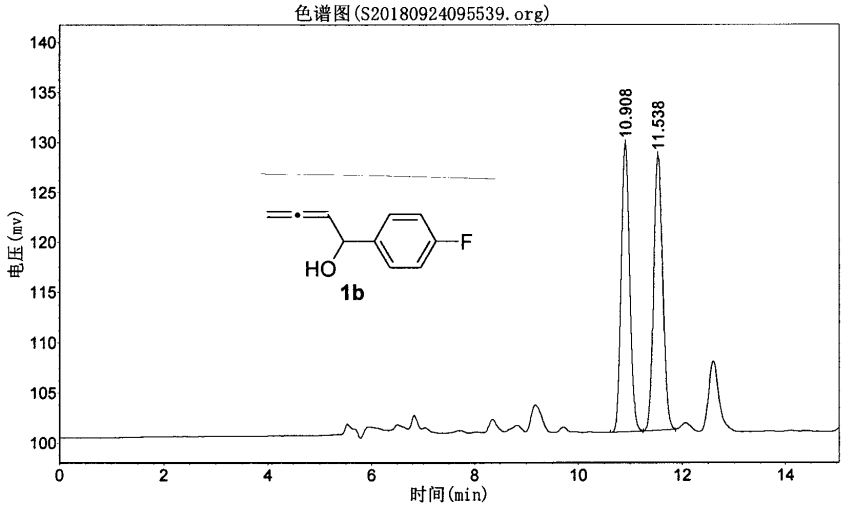

分析结果表

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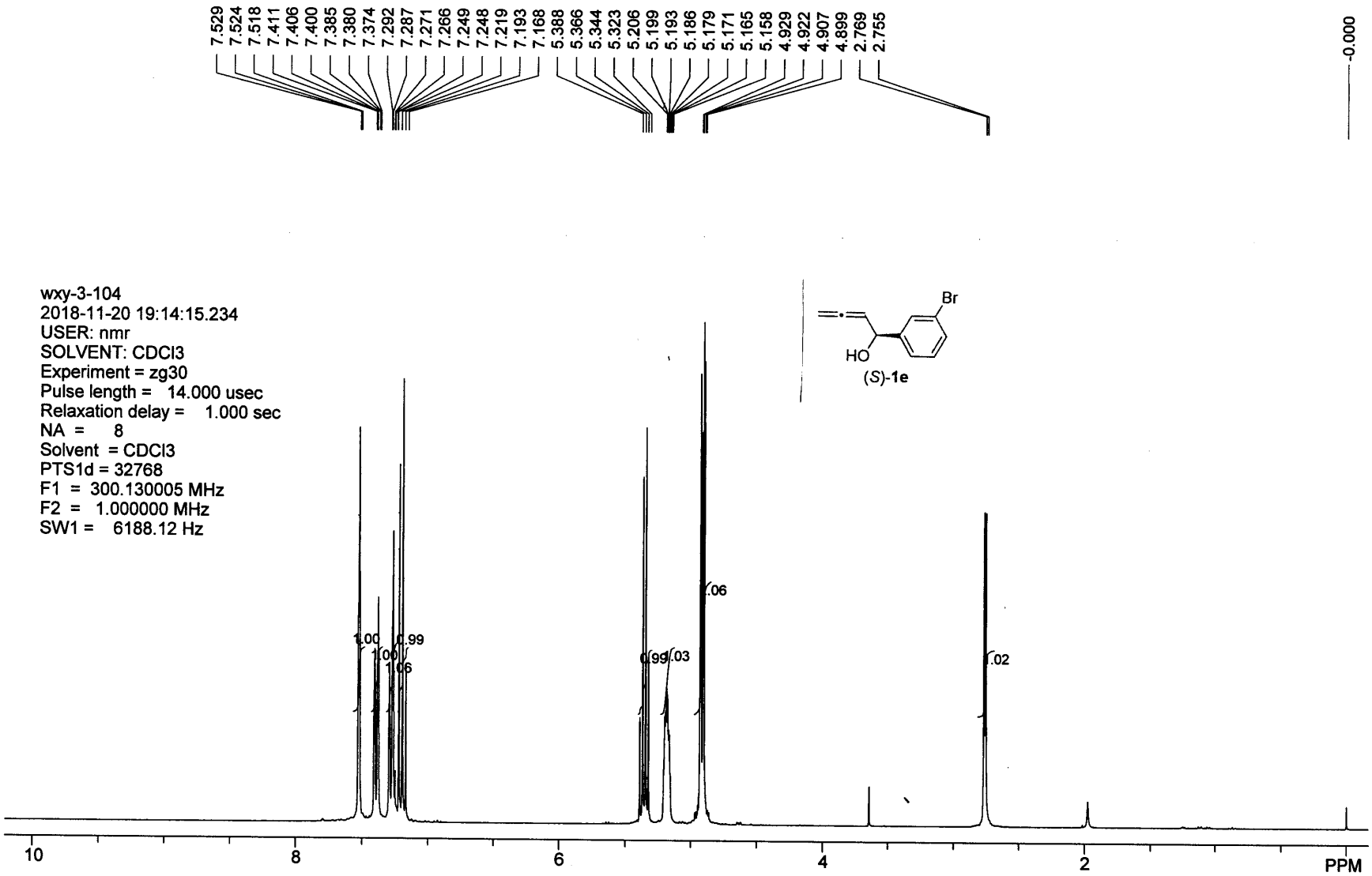
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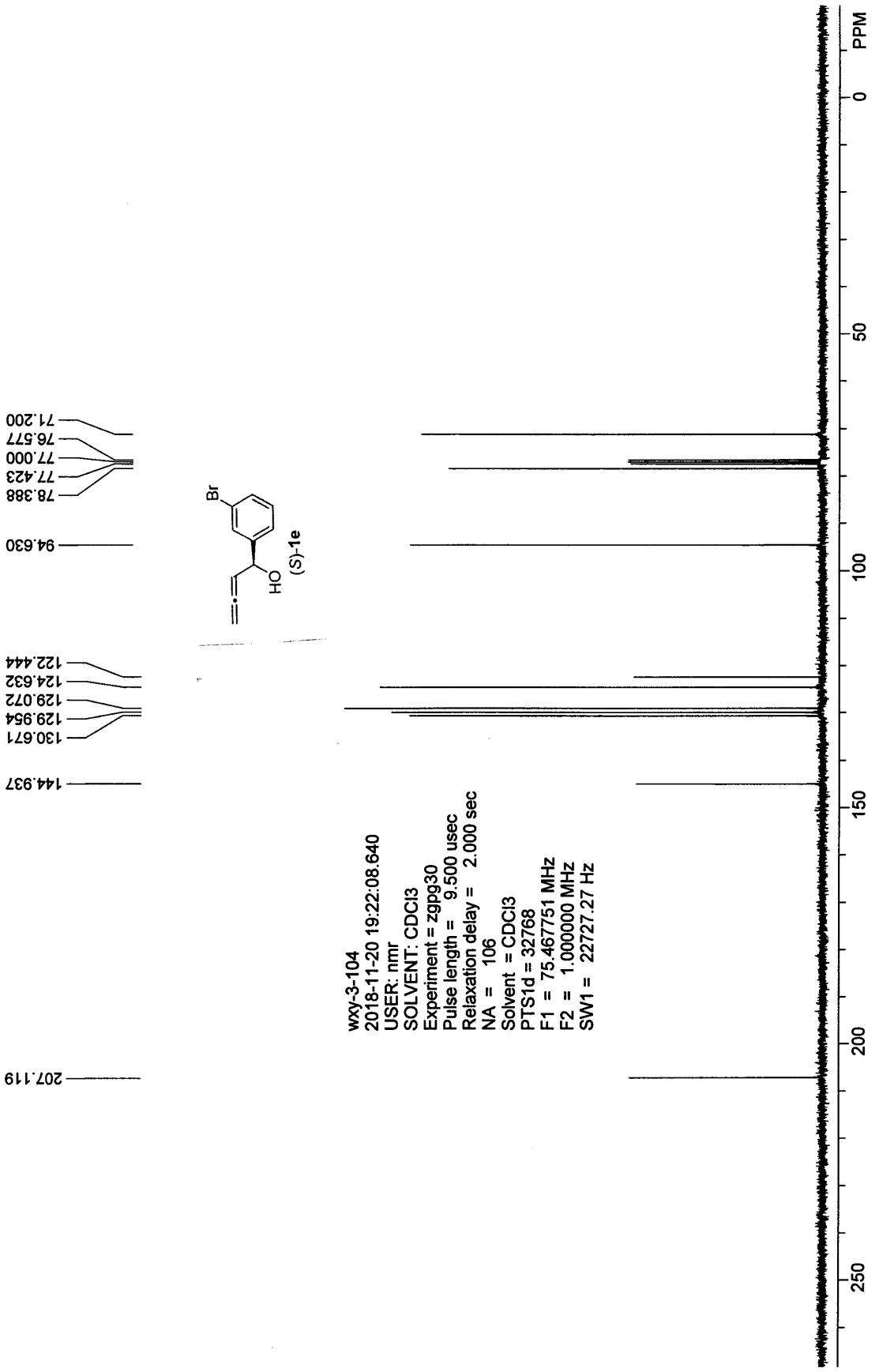
PPM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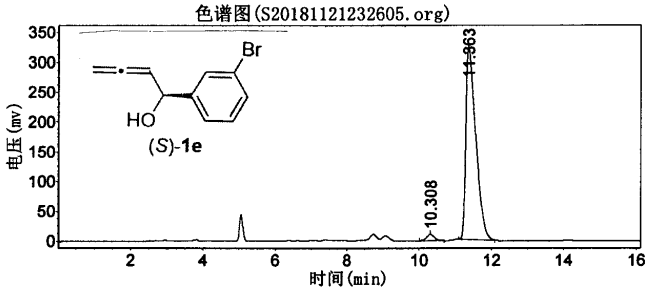
wxy-3-104-Chiral

实验时间：2018-11-21,23:26:05
谱图文件:D:\浙大智达\N2000\样品\S20181121232605.org
方法文件:D:\浙大智达\N2000\djx.mtd

实验者：wxy
报告时间：2018-12-11,12:46:20
积分方法:面积归一法

实验内容简介：
OD-H,n-hexane/i-PrOH = 95/5,1.0,254

色谱图(S20181121232605.org)



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		10.308	10769.917	147315.500	2.3293
2		11.363	321465.625	6177091.000	97.6707
总计			332235.542	6324406.500	100.0000

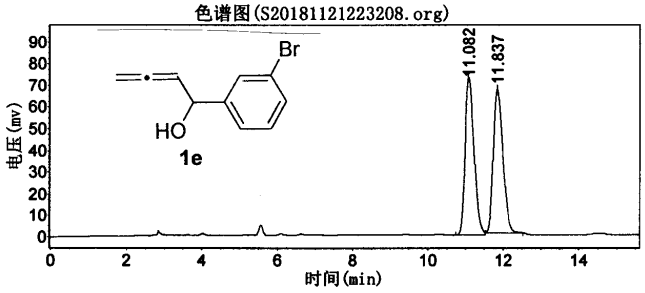
wxy-3-104-racemic

实验时间：2018-11-21,22:32:08
谱图文件:D:\浙大智达\N2000\样品\S20181121223208.org
方法文件:D:\浙大智达\N2000\djx.mtd

实验者：wxy
报告时间：2018-11-21,23:49:35
积分方法:面积归一法

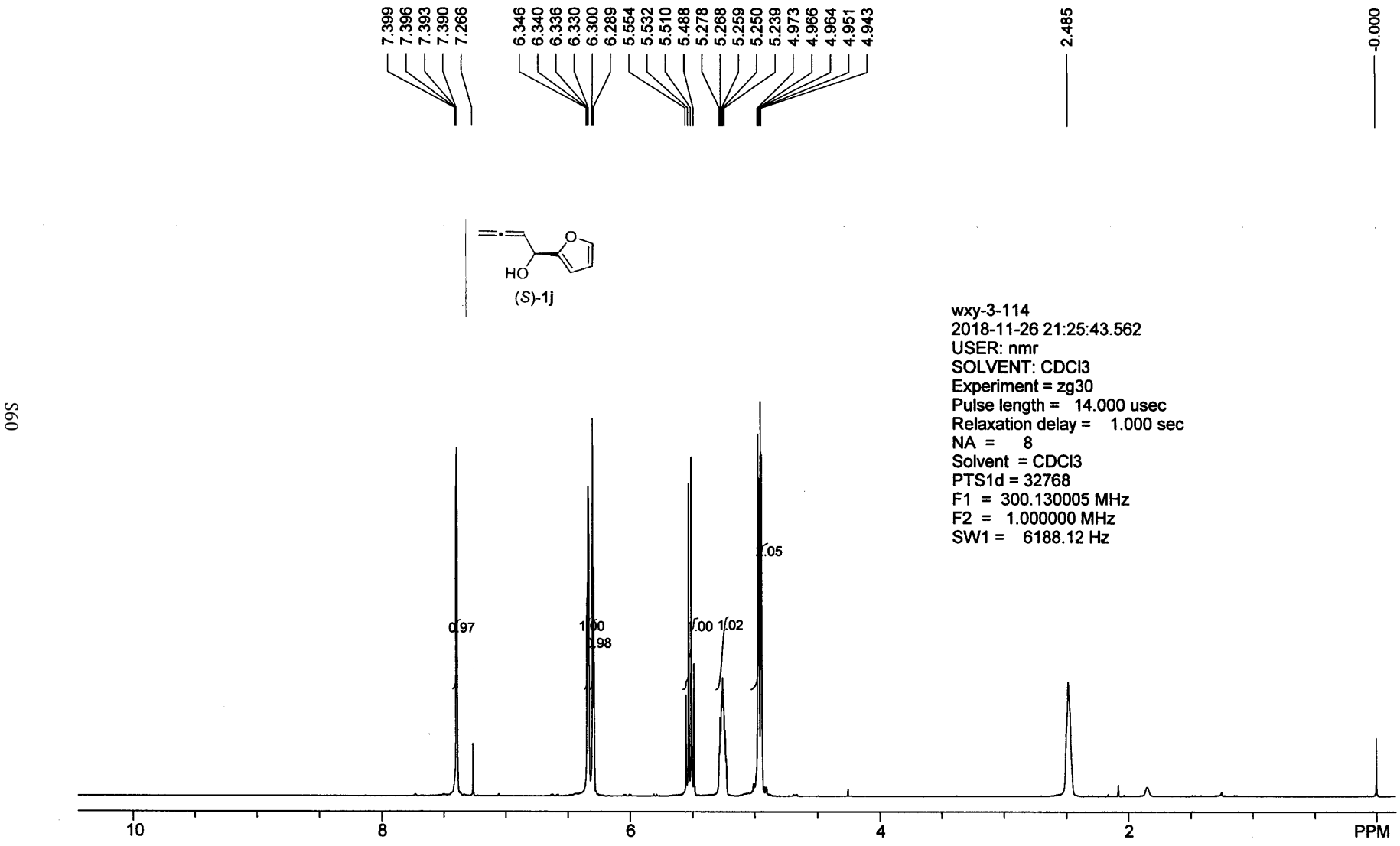
实验内容简介：
OD-H,n-hexane/i-PrOH = 95/5,1.0,254

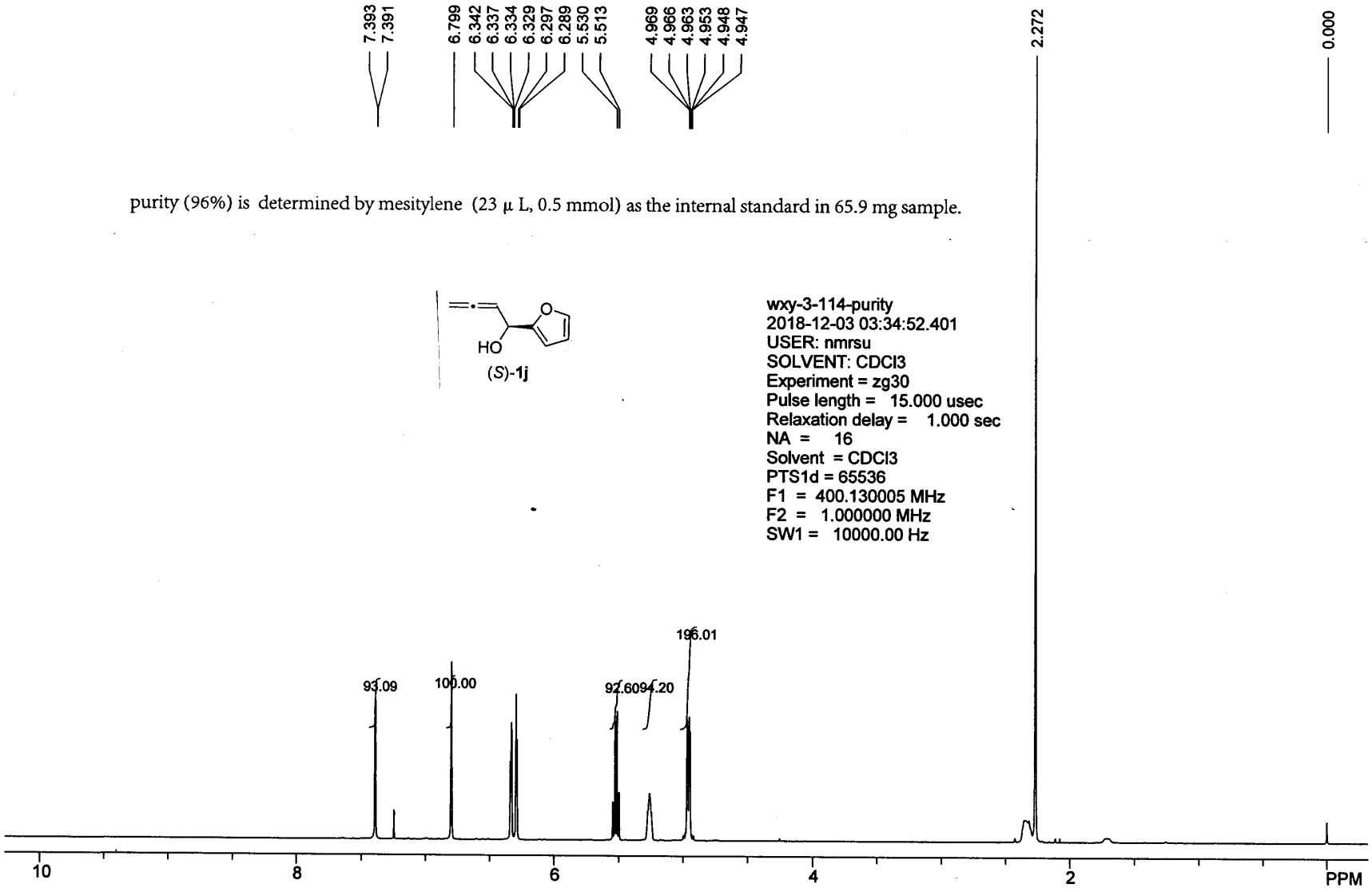
色谱图(S20181121223208.org)

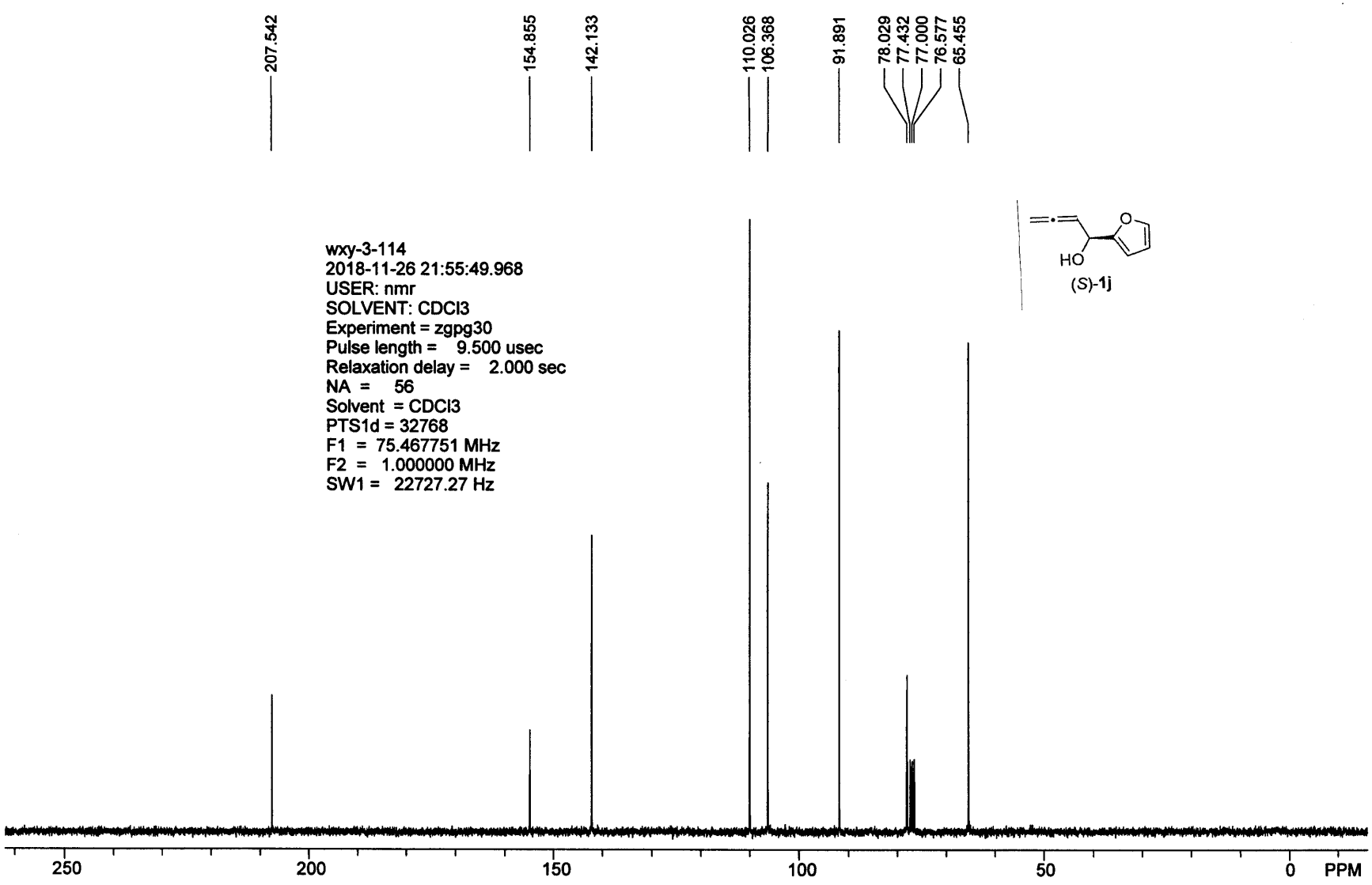


分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		11.082	72686.227	1196967.875	50.4561
2		11.837	66179.430	1175327.625	49.5439
总计			138865.656	2372295.500	100.0000


7.399
7.396
7.393
7.390
7.266
6.346
6.340
6.336
6.330
6.300
6.289
5.554
5.532
5.510
5.488
5.278
5.268
5.259
5.250
5.239
4.973
4.966
4.964
4.951
4.943
2.485
-0.000
HO
O
(S)-1j
wxy-3-114
2018-11-26 21:25:43.562
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
0.97
1.00
0.98
1.00
1.02
1.05
10
8
6
4
2
PPM


7.393
7.391
6.799
6.342
6.337
6.334
6.329
6.297
6.289
5.530
5.513
4.969
4.966
4.963
4.953
4.948
4.947
2.272
0.000
purity (96%) is determined by mesitylene (23 μ L, 0.5 mmol) as the internal standard in 65.9 mg sample.
HO
(S)-1j
wxy-3-114-purity
2018-12-03 03:34:52.401
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
93.09
100.00
92.60
94.20
196.01
10
8
6
4
2
PPM


207.542
154.855
142.133
110.026
106.368
91.891
78.029
77.432
77.000
76.577
65.455
wxy-3-114
2018-11-26 21:55:49.968
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 56
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
HO
O
(S)-1j
250
200
150
100
50
0
PPM

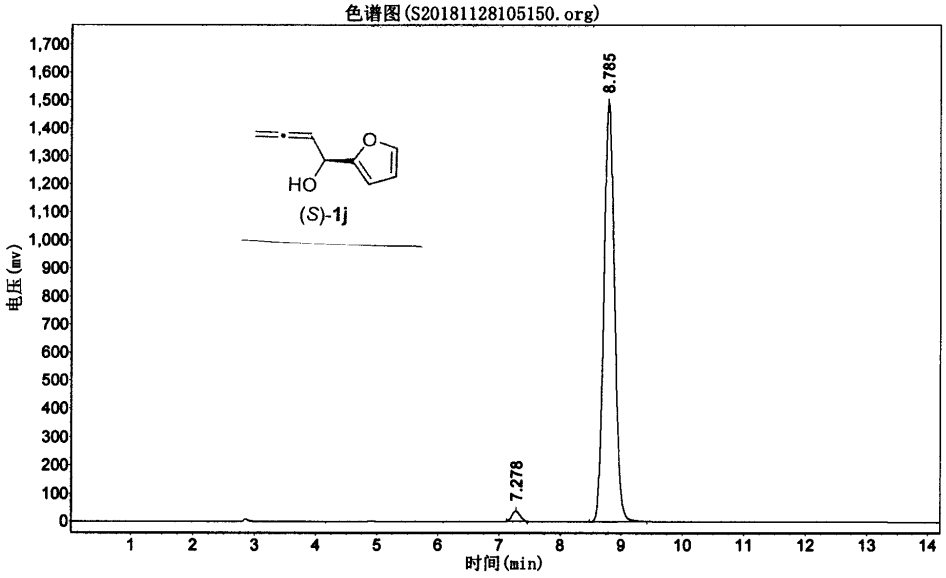
wxy-3-114-Chiral

实验时间：2018-11-28,10:51:50
谱图文件:D:\浙大智达\N2000\样品\S20181128105150.org
方法文件:D:\浙大智达\N2000\djx.mtd

实验者：wxy
报告时间：2018-11-28,12:05:51
积分方法:面积归一法

实验内容简介：
od-H,n-hexane/i-PrOH = 90/10,1.0,220

色谱图(S20181128105150.org)



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		7.278	35930.438	331924.594	1.7927
2		8.785	1491204.125	18183154.000	98.2073
总计			1527134.563	18515078.594	100.0000

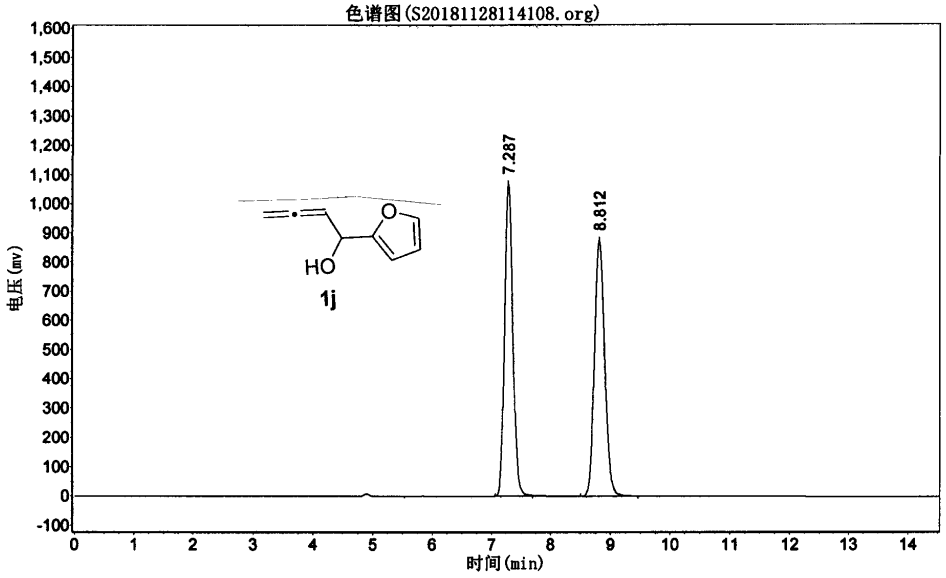
wxy-3-114-racemic

实验时间：2018-11-28,11:41:08
谱图文件:D:\浙大智达\N2000\样品\S20181128114108.org
方法文件:D:\浙大智达\N2000\djx.mtd

实验者：wxy
报告时间：2018-11-28,12:03:45
积分方法:面积归一法

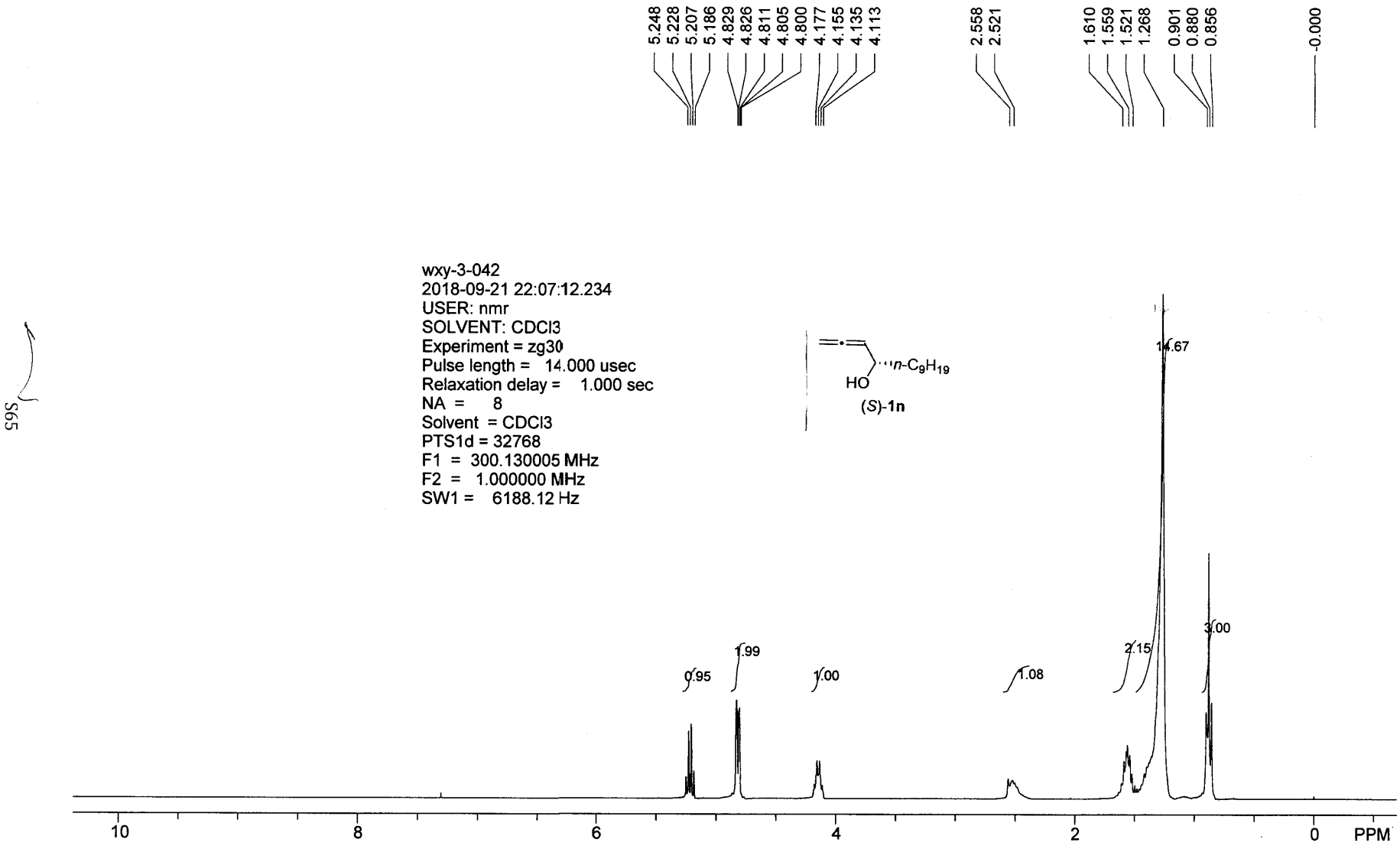
实验内容简介：
od-H,n-hexane/i-PrOH = 90/10,1.0,220

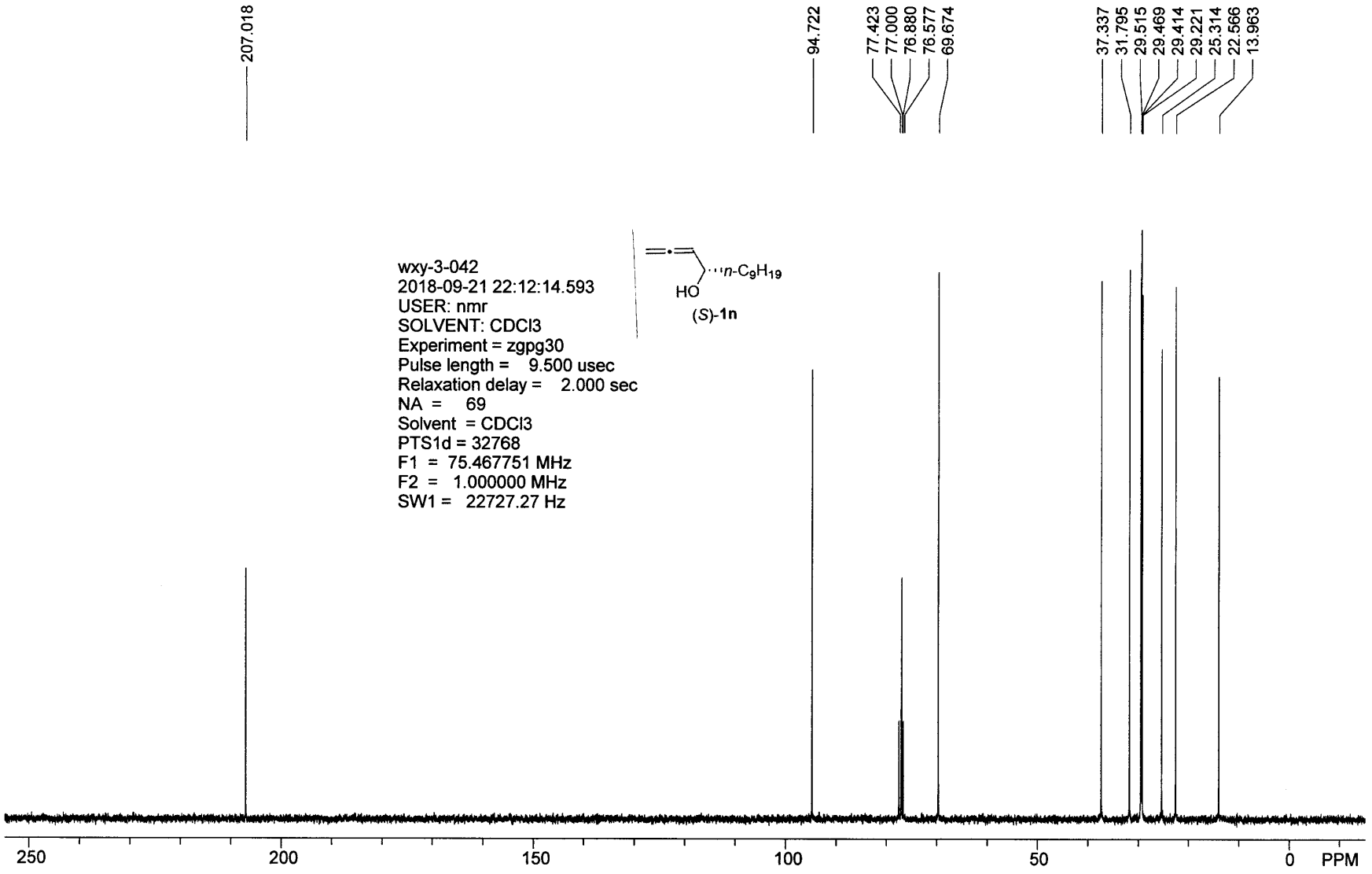
色谱图(S20181128114108.org)



分析结果表

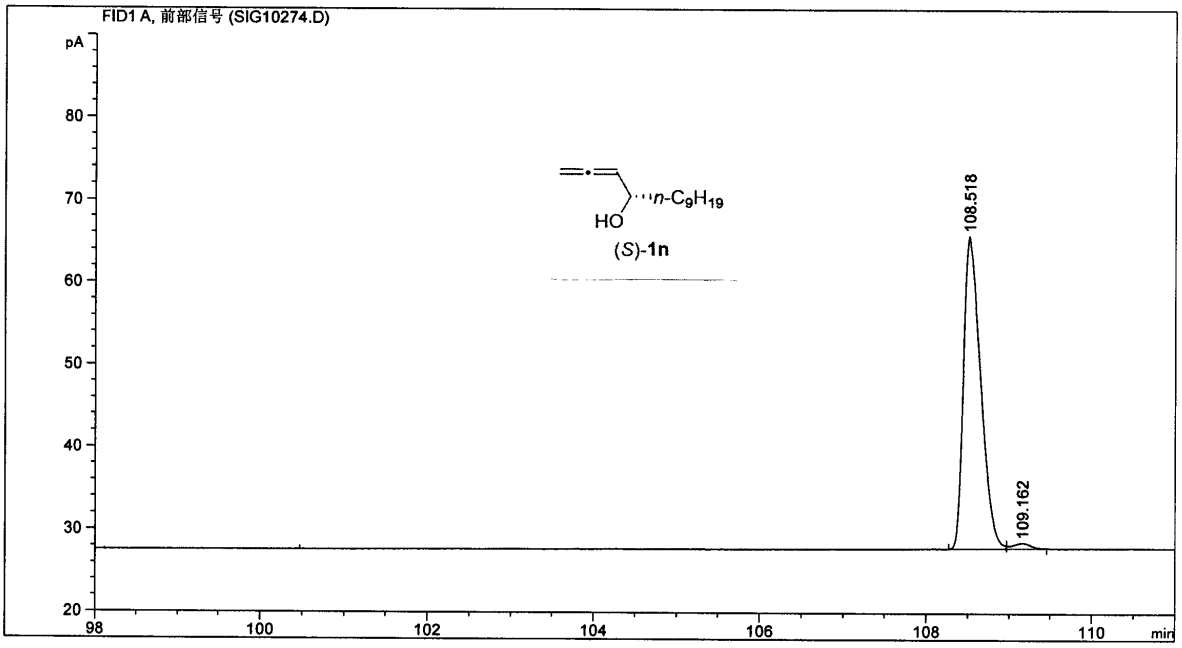
峰号	峰名	保留时间	峰高	峰面积	含量
1		7.287	1064891.875	10312150.000	49.7654
2		8.812	871514.125	10409357.000	50.2346
总计			1936406.000	20721507.000	100.0000


5.248
5.228
5.207
5.186
4.829
4.826
4.811
4.805
4.800
4.177
4.155
4.135
4.113
2.558
2.521
1.610
1.559
1.521
1.268
0.901
0.880
0.856
-0.000
wxy-3-042
2018-09-21 22:07:12.234
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
HO
n-C9H19
(S)-1n
0.95
1.99
1.00
1.08
2.15
14.67
3.00
10
8
6
4
2
0
PPM


207.018
94.722
77.423
77.000
76.880
76.577
69.674
37.337
31.795
29.515
29.469
29.414
29.221
25.314
22.566
13.963
wxy-3-042
2018-09-21 22:12:14.593
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
HO
n-C9H19
(S)-1n
250
200
150
100
50
0
PPM

数据文件: C:\CHEM32\1\DATA\SIG10274.D
样品名称: gbj-10-159-chiral

```
==========================================================================
操作者            : wang
仪器              : 仪器 1                                  位置 : 样品瓶 1
进样日期          : 2018/10/16 10:57:21
                                                        进样量 : 手动
采集方法          : C:\CHEM32\1\METHODS\DEF_GC7890A.M
最后修改          : 2018/10/16 10:57:18 :  wang
                        (调用后修改)
分析方法          : C:\CHEM32\1\METHODS\DEF_GC-OFF.M
最后修改          : 2018/10/17 15:43:04 :  wang
                        (调用后修改)
样品信息          : rtbdex
```

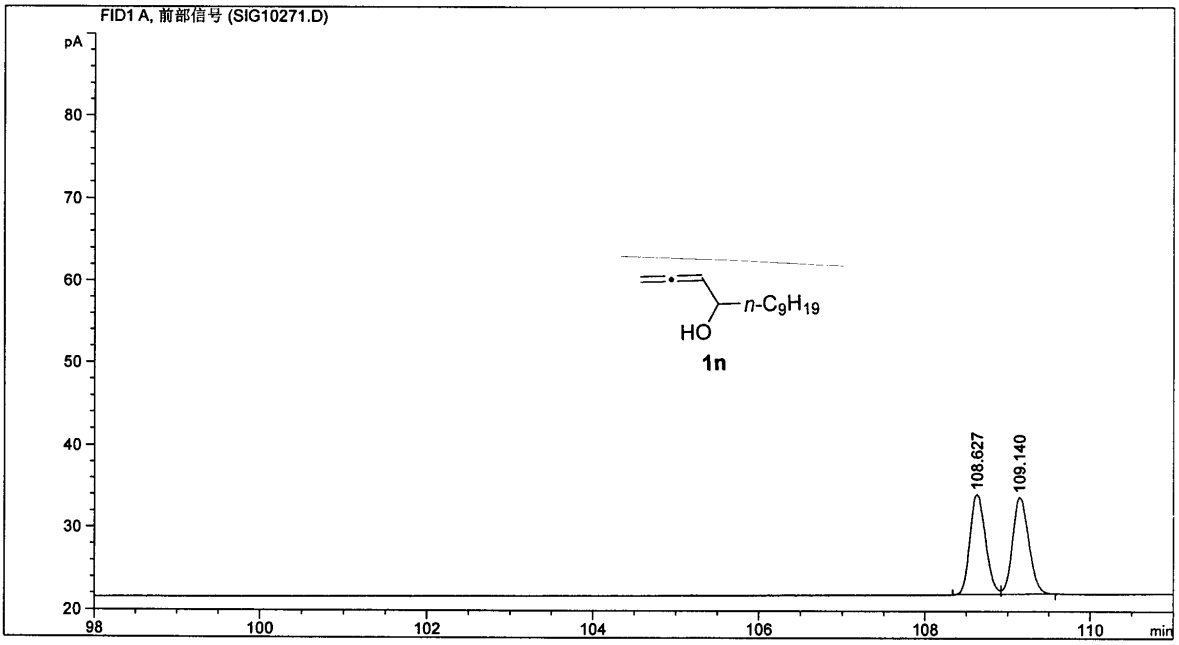



```
==========================================================================
                          面积百分比报告
==========================================================================

排序                    :      信号
乘积因子:               :      1.0000
稀释因子:               :      1.0000
内标使用乘积因子和稀释因子


信号 1: FID1 A, 前部信号

 峰 保留时间 类型  峰宽      峰面积       峰高       峰面积
  #   [min]        [min]   [pA*s]       [pA]         %
----|-------|----|-------|----------|----------|--------|
   1 108.518 MF R  0.2375  545.44348   37.88842 98.13579
   2 109.162 FM R  0.2529   10.36135 6.82758e-1  1.86421

总量 :                    555.80483   38.57117

==========================================================================
                        *** 报告结束 ***
```

仪器 1 2018/10/17 15:43:13 wang　　页 1/1

数据文件: C:\CHEM32\1\DATA\SIG10271.D
样品名称: gbj-10-159-rac

操作者 : wang
仪器 : 仪器 1 位置 : 样品瓶 1
进样日期 : 2018/10/15 12:46:07
进样量 : 手动
采集方法 : C:\CHEM32\1\METHODS\DEF_GC7890A.M
最后修改 : 2018/10/15 12:46:06 : wang
(调用后修改)
分析方法 : C:\CHEM32\1\METHODS\DEF_GC-OFF.M
最后修改 : 2018/10/17 15:43:04 : wang
(调用后修改)
样品信息 : rtbdex



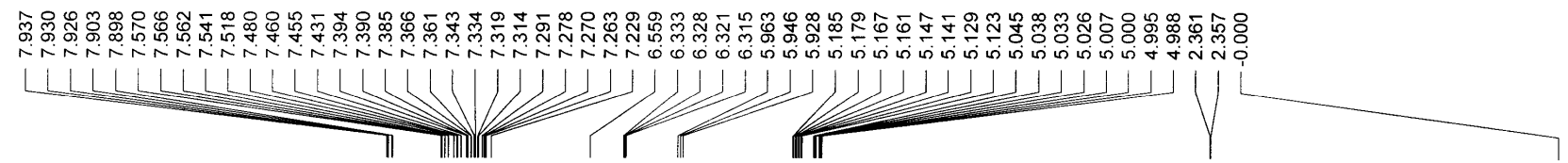
面积百分比报告

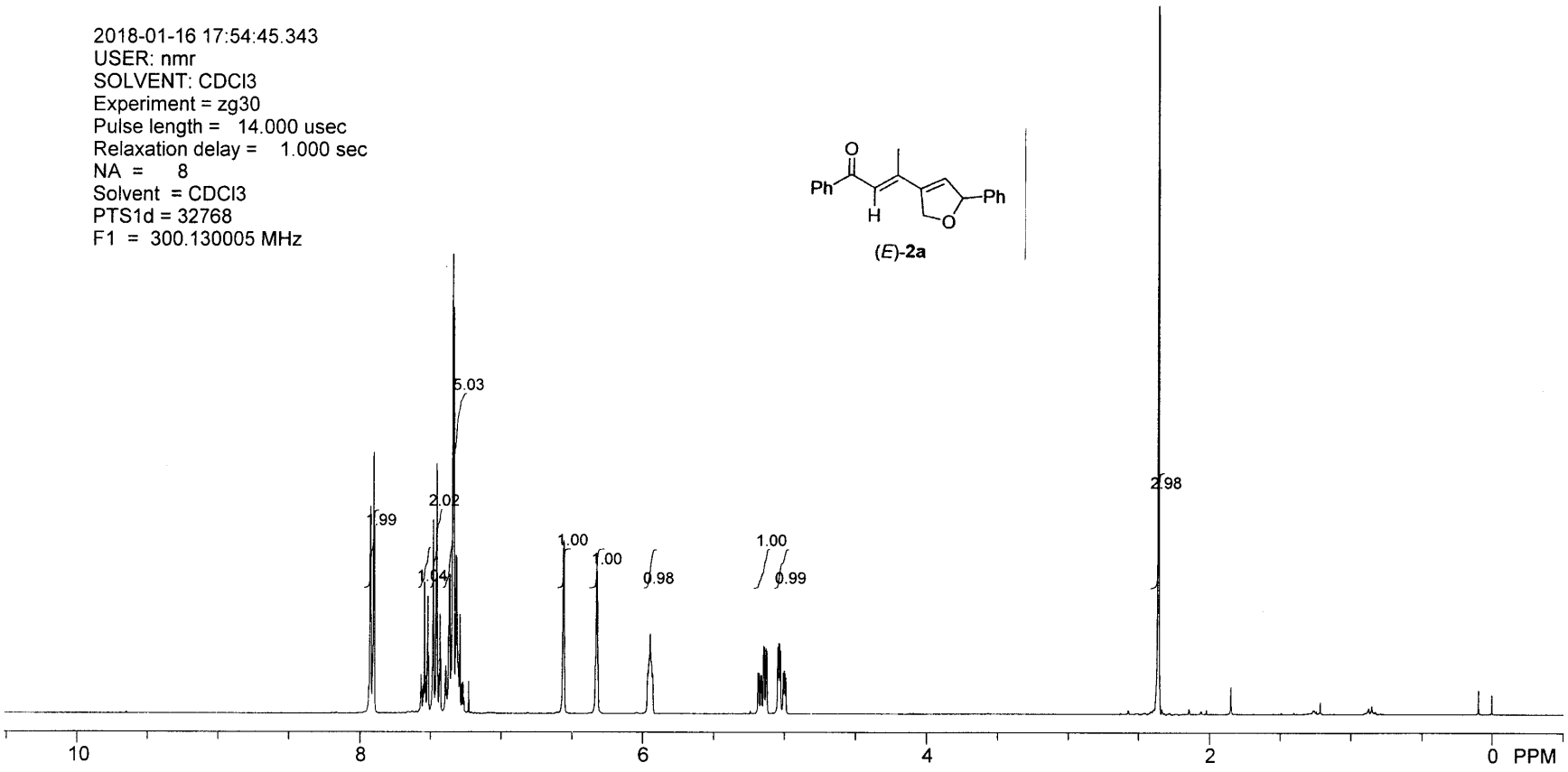
排序 : 信号
乘积因子: : 1.0000
稀释因子: : 1.0000
内标使用乘积因子和稀释因子

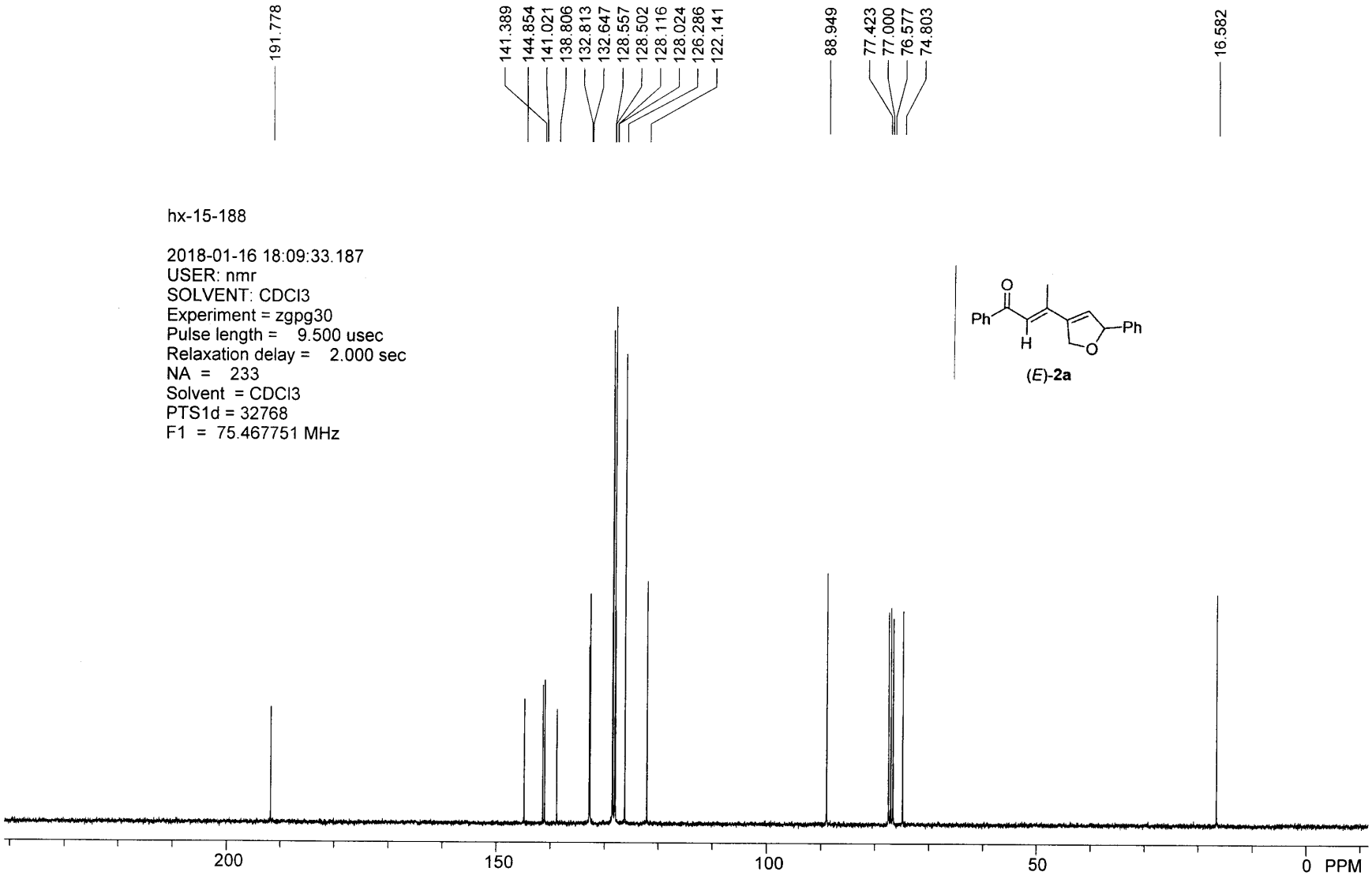
信号 1: FID1 A, 前部信号

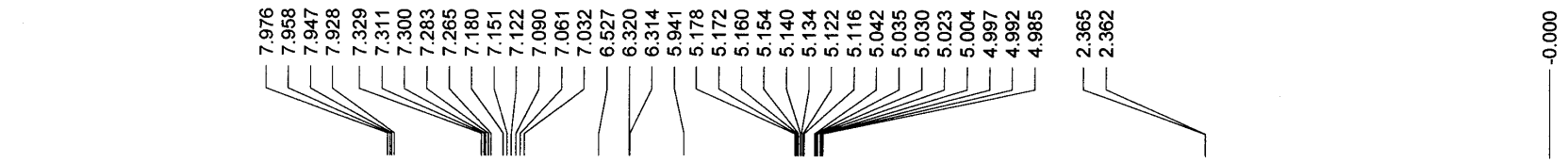
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [pA*s]	峰高 [pA]	峰面积 %
1	108.627	MF R	0.2082	159.14456	12.14610	49.78487
2	109.140	FM R	0.2143	160.51993	11.79749	50.21513
总量 :				319.66449	23.94359	

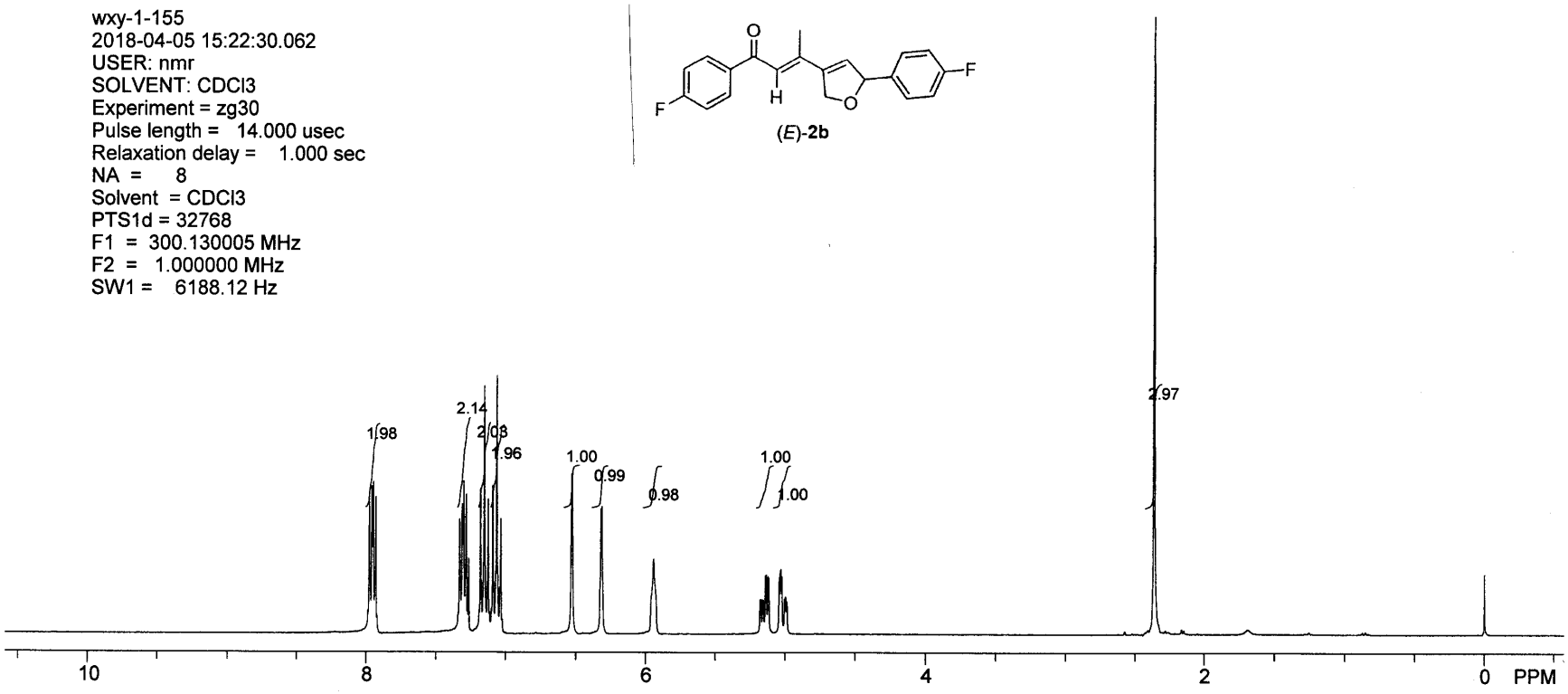
*** 报告结束 ***

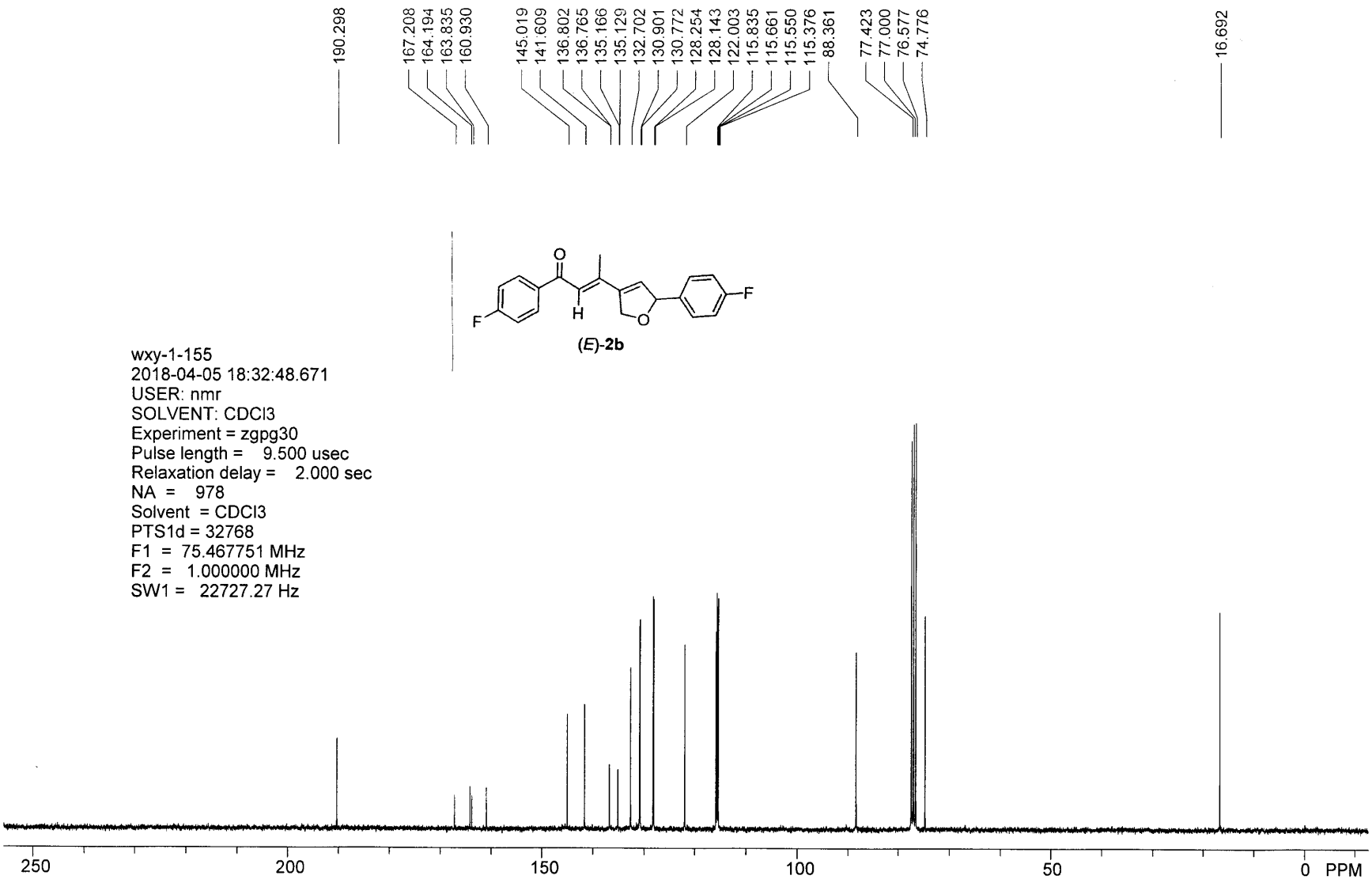

7.937
7.930
7.926
7.903
7.898
7.570
7.566
7.562
7.541
7.518
7.480
7.460
7.455
7.431
7.394
7.390
7.385
7.366
7.361
7.343
7.334
7.319
7.314
7.291
7.278
7.270
7.263
7.229
6.559
6.333
6.328
6.321
6.315
5.963
5.946
5.928
5.185
5.179
5.167
5.161
5.147
5.141
5.129
5.123
5.045
5.038
5.033
5.026
5.007
5.000
4.995
4.988
2.361
2.357
-0.000

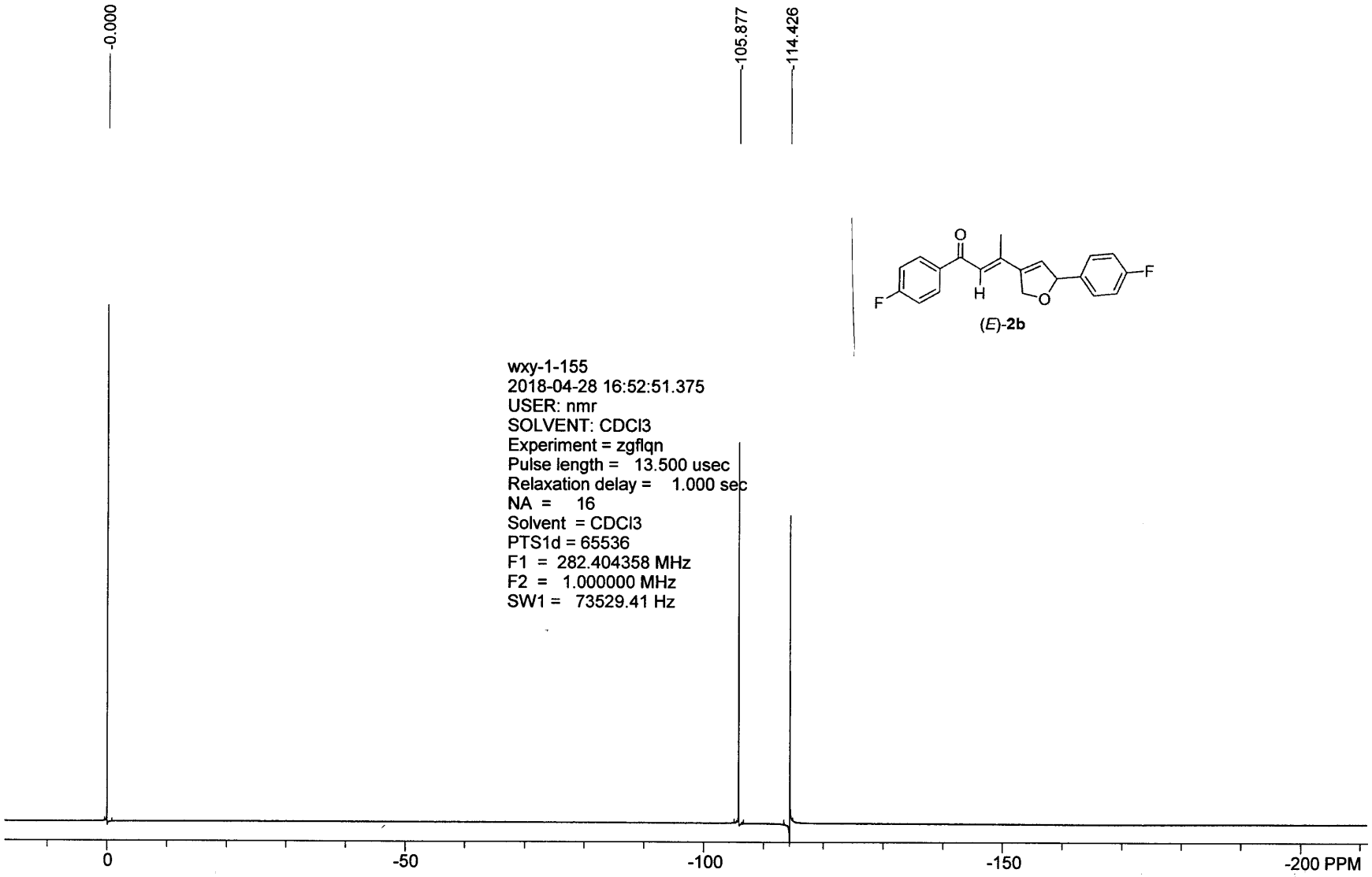

hx-15-188
2018-01-16 17:54:45.343
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
O
Ph
H
O
Ph
(E)-2a
5.03
2.02
1.99
1.04
1.00
1.00
0.98
1.00
0.99
2.98
10
8
6
4
2
0 PPM

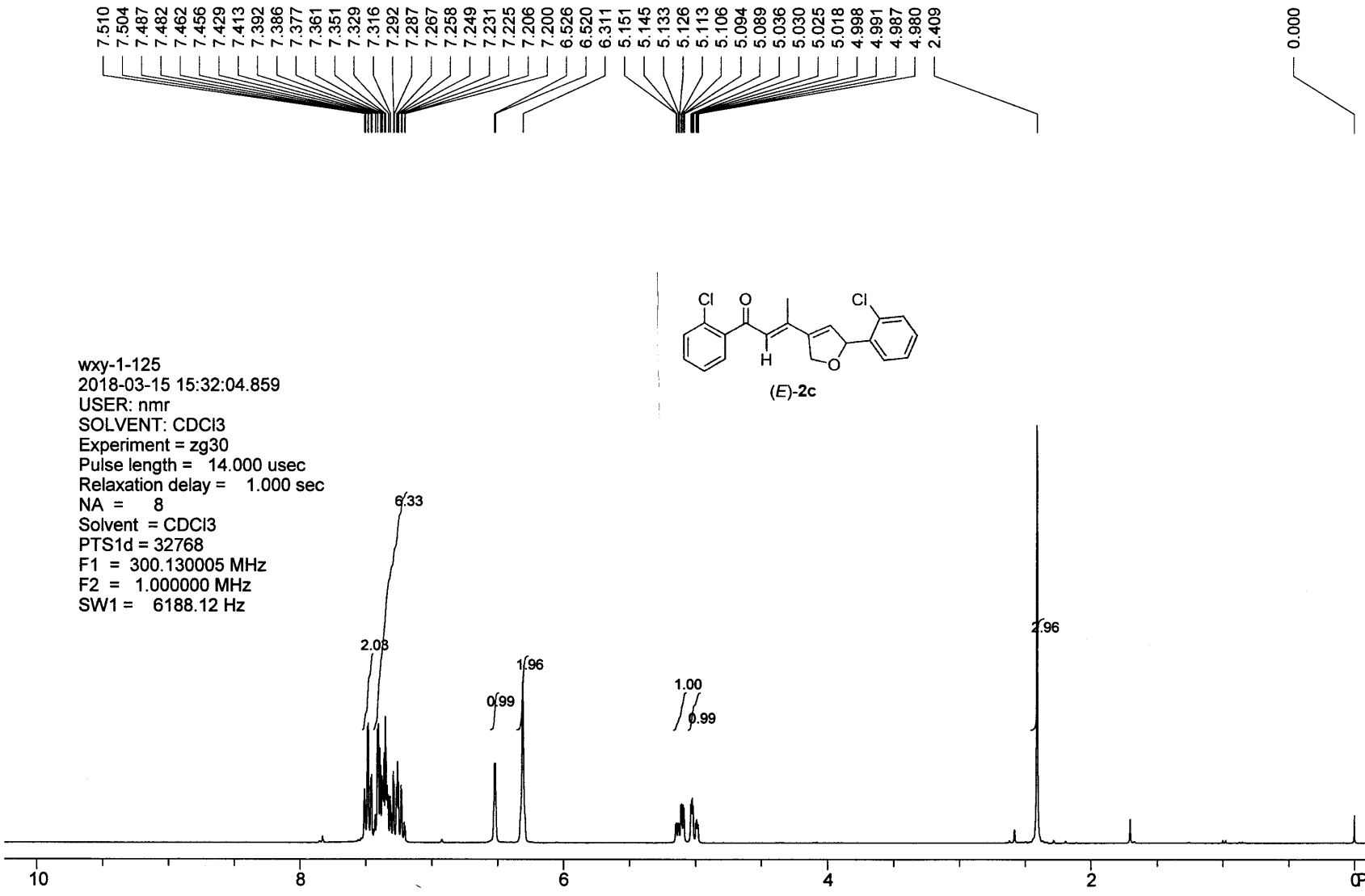

191.778
141.389
144.854
141.021
138.806
132.813
132.647
128.557
128.502
128.116
128.024
126.286
122.141
88.949
77.423
77.000
76.577
74.803
16.582
hx-15-188
2018-01-16 18:09:33.187
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 233
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
O
Ph
H
Ph
O
(E)-2a
200
150
100
50
0 PPM

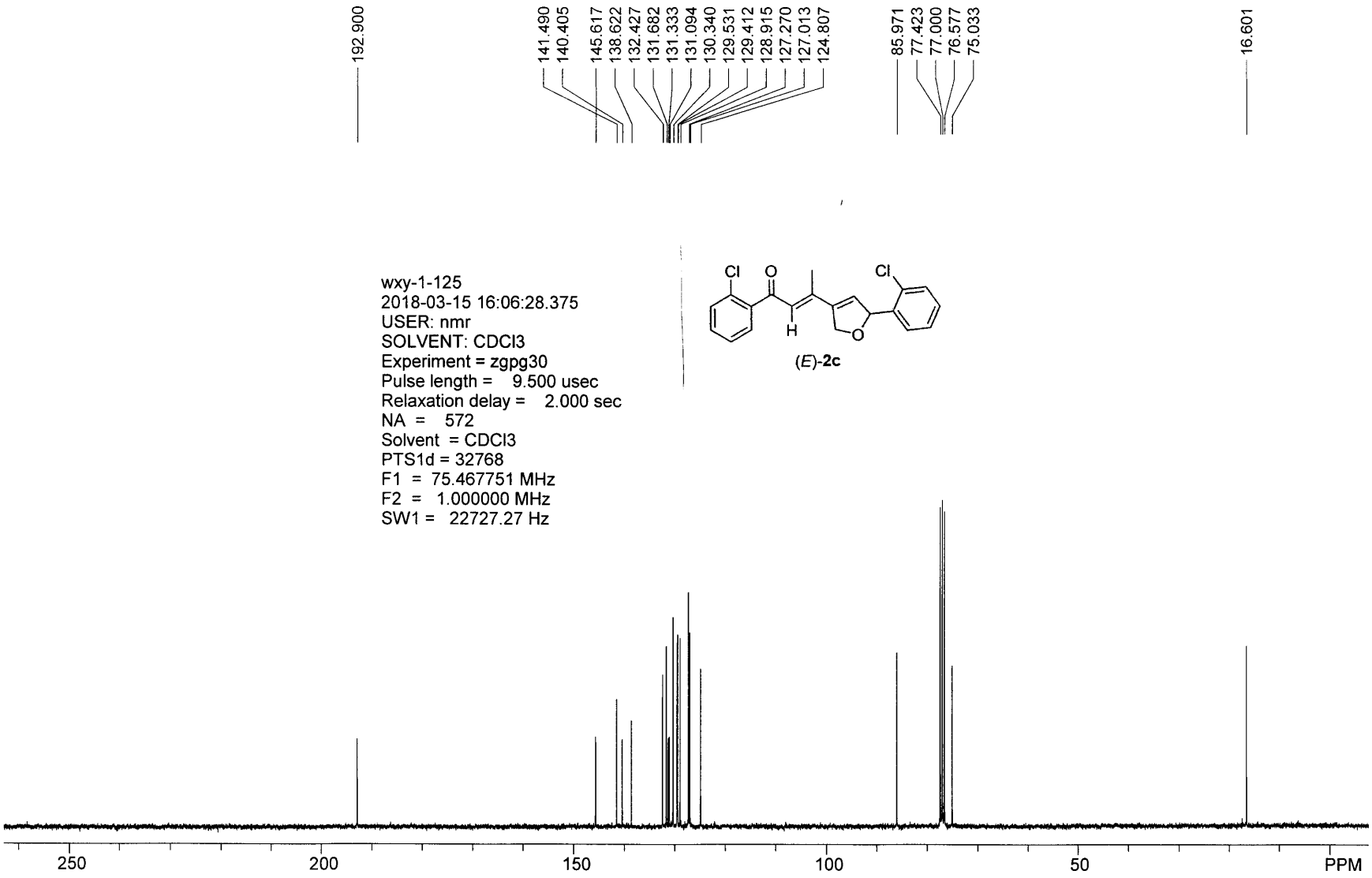

7.976
7.958
7.947
7.928
7.329
7.311
7.300
7.283
7.265
7.180
7.151
7.122
7.090
7.061
7.032
6.527
6.320
6.314
5.941
5.178
5.172
5.160
5.154
5.140
5.134
5.122
5.116
5.042
5.035
5.030
5.023
5.004
4.997
4.992
4.985
2.365
2.362
-0.000

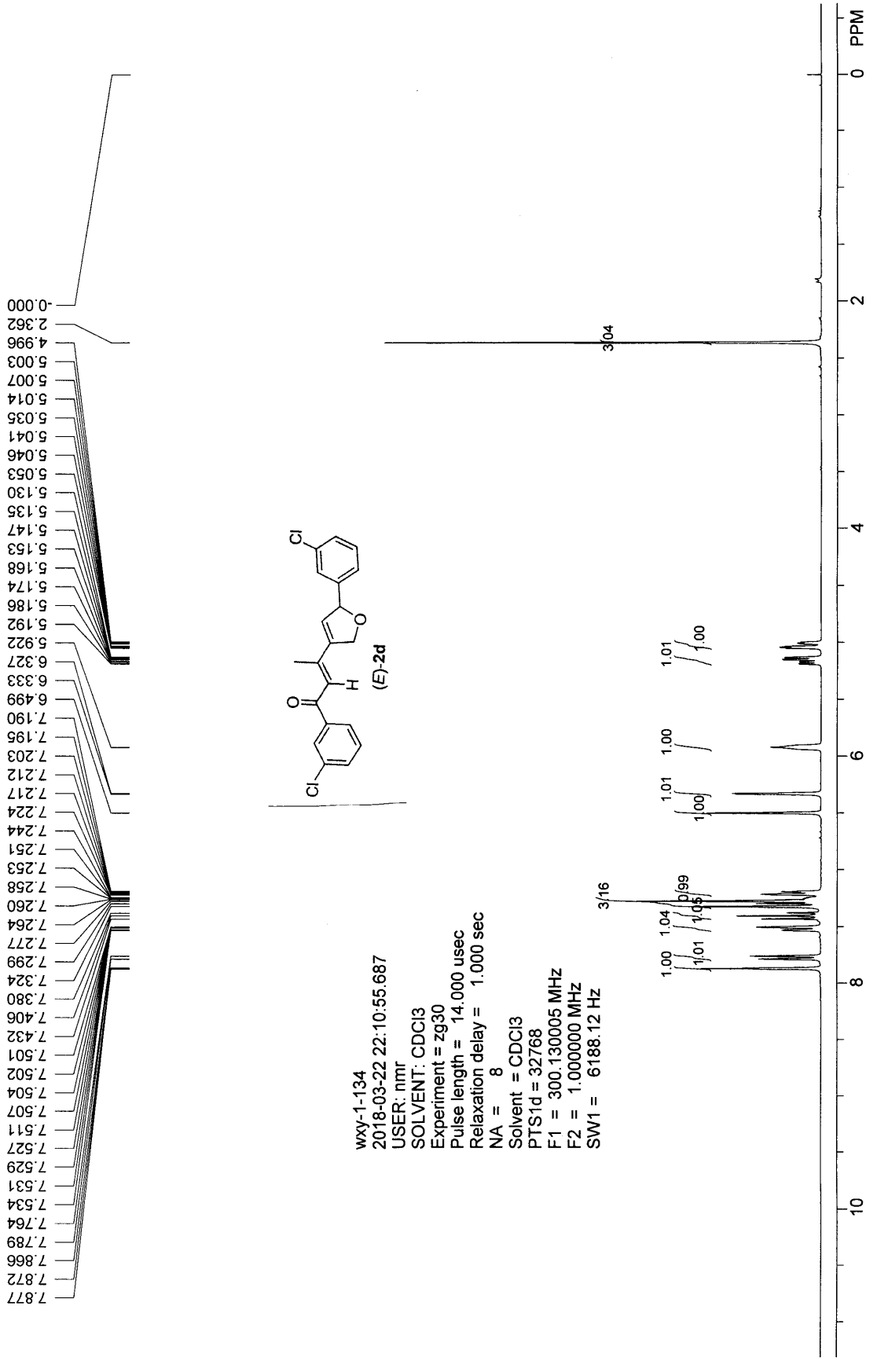

wxy-1-155
2018-04-05 15:22:30.062
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
O
H
O
F
F
(E)-2b
1.98
2.14
2.08
1.96
1.00
0.99
0.98
1.00
1.00
2.97
10
8
6
4
2
0
PPM

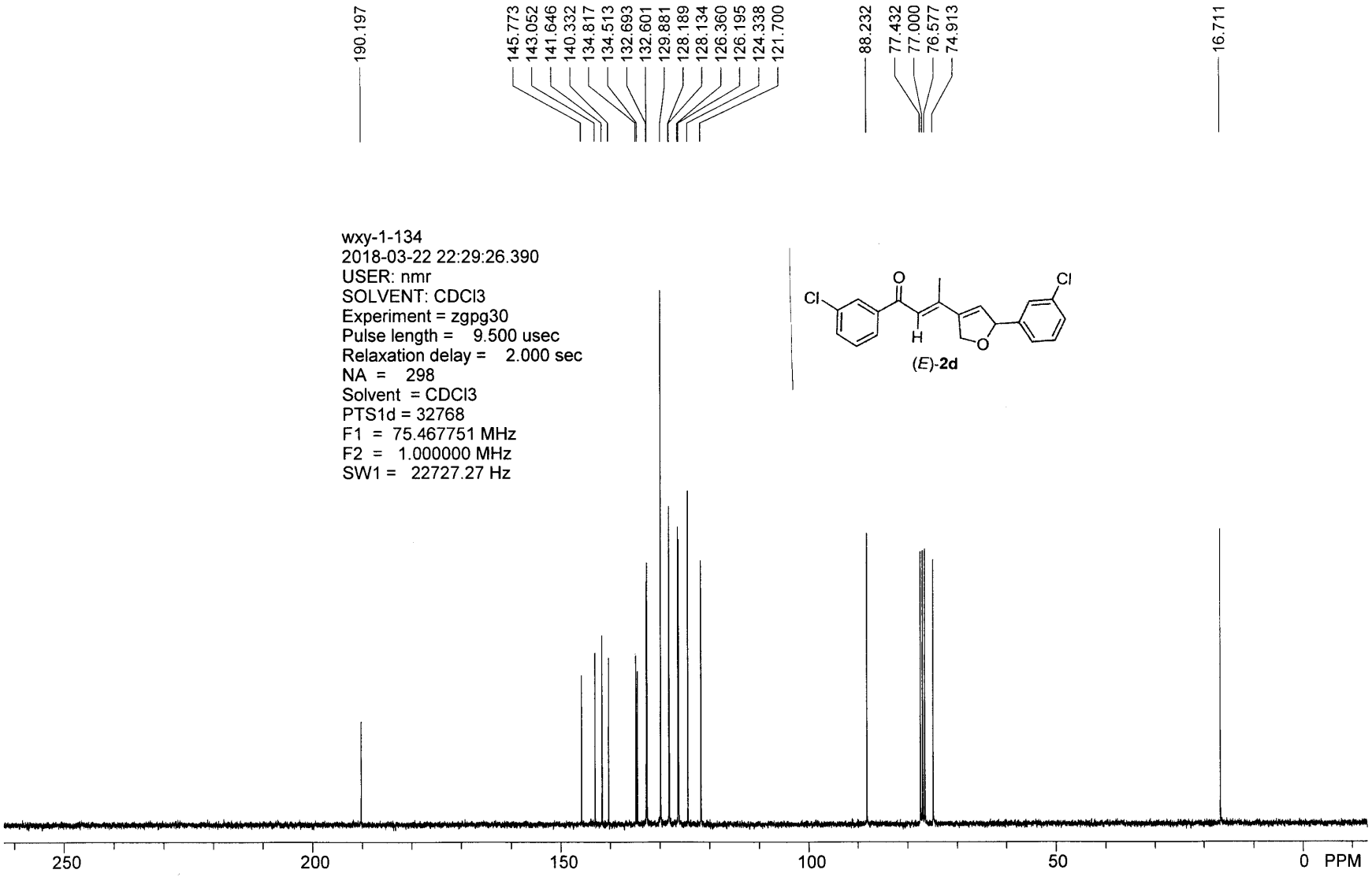

190.298
167.208
164.194
163.835
160.930
145.019
141.609
136.802
136.765
135.166
135.129
132.702
130.901
130.772
128.254
128.143
122.003
115.835
115.661
115.550
115.376
88.361
77.423
77.000
76.577
74.776
16.692
O
H
O
F
F
(E)-2b
wxy-1-155
2018-04-05 18:32:48.671
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 978
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
250
200
150
100
50
0 PPM

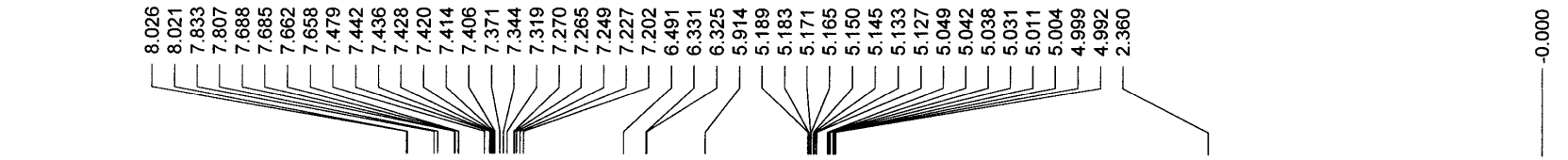

-0.000
105.877
114.426
O
H
O
F
F
(E)-2b
wxy-1-155
2018-04-28 16:52:51.375
USER: nmr
SOLVENT: CDCl3
Experiment = zgflqn
Pulse length = 13.500 usec
Relaxation delay = 1.000 sec
NA = 16
Solvent = CDCl3
PTS1d = 65536
F1 = 282.404358 MHz
F2 = 1.000000 MHz
SW1 = 73529.41 Hz
0
-50
-100
-150
-200 PPM

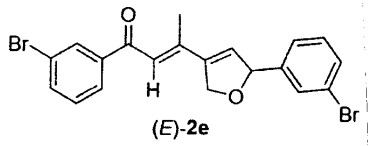

7.510
7.504
7.487
7.482
7.462
7.456
7.429
7.413
7.392
7.386
7.377
7.361
7.351
7.329
7.316
7.292
7.287
7.267
7.258
7.249
7.231
7.225
7.206
7.200
6.526
6.520
6.311
5.151
5.145
5.133
5.126
5.113
5.106
5.094
5.089
5.036
5.030
5.025
5.018
4.998
4.991
4.987
4.980
2.409
0.000
Cl
O
Cl
H
O
(E)-2c
wxy-1-125
2018-03-15 15:32:04.859
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
6.33
2.08
1.96
0.99
1.00
0.99
2.96
10
8
6
4
2
0
PPM


192.900
141.490
140.405
145.617
138.622
132.427
131.682
131.333
131.094
130.340
129.531
129.412
128.915
127.270
127.013
124.807
85.971
77.423
77.000
76.577
75.033
16.601
wxy-1-125
2018-03-15 16:06:28.375
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 572
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
Cl
O
Cl
H
O
(E)-2c
250
200
150
100
50
PPM


wxy-1-134
2018-03-22 22:10:55.687
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
(E)-2d
PPM

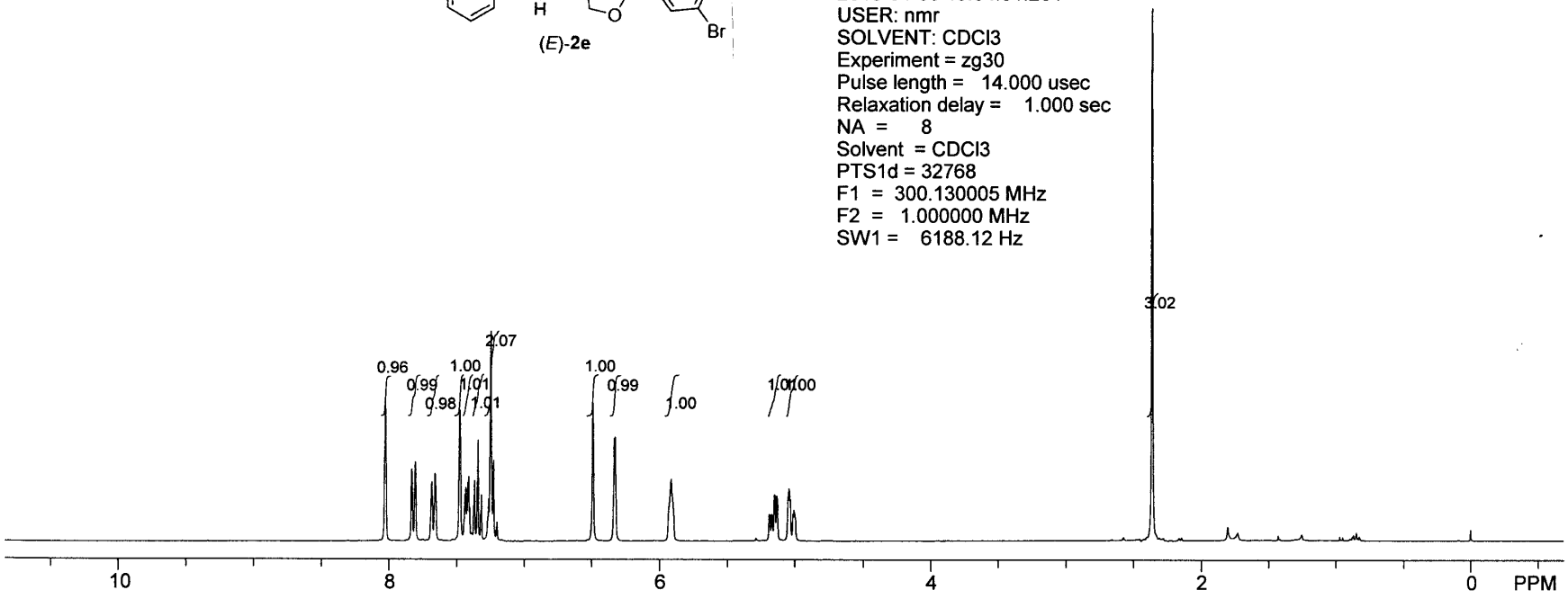

190.197
145.773
143.052
141.646
140.332
134.817
134.513
132.693
132.601
129.881
128.189
128.134
126.360
126.195
124.338
121.700
88.232
77.432
77.000
76.577
74.913
16.711
wxy-1-134
2018-03-22 22:29:26.390
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 298
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
Cl
O
Cl
H
O
(E)-2d
250
200
150
100
50
0 PPM

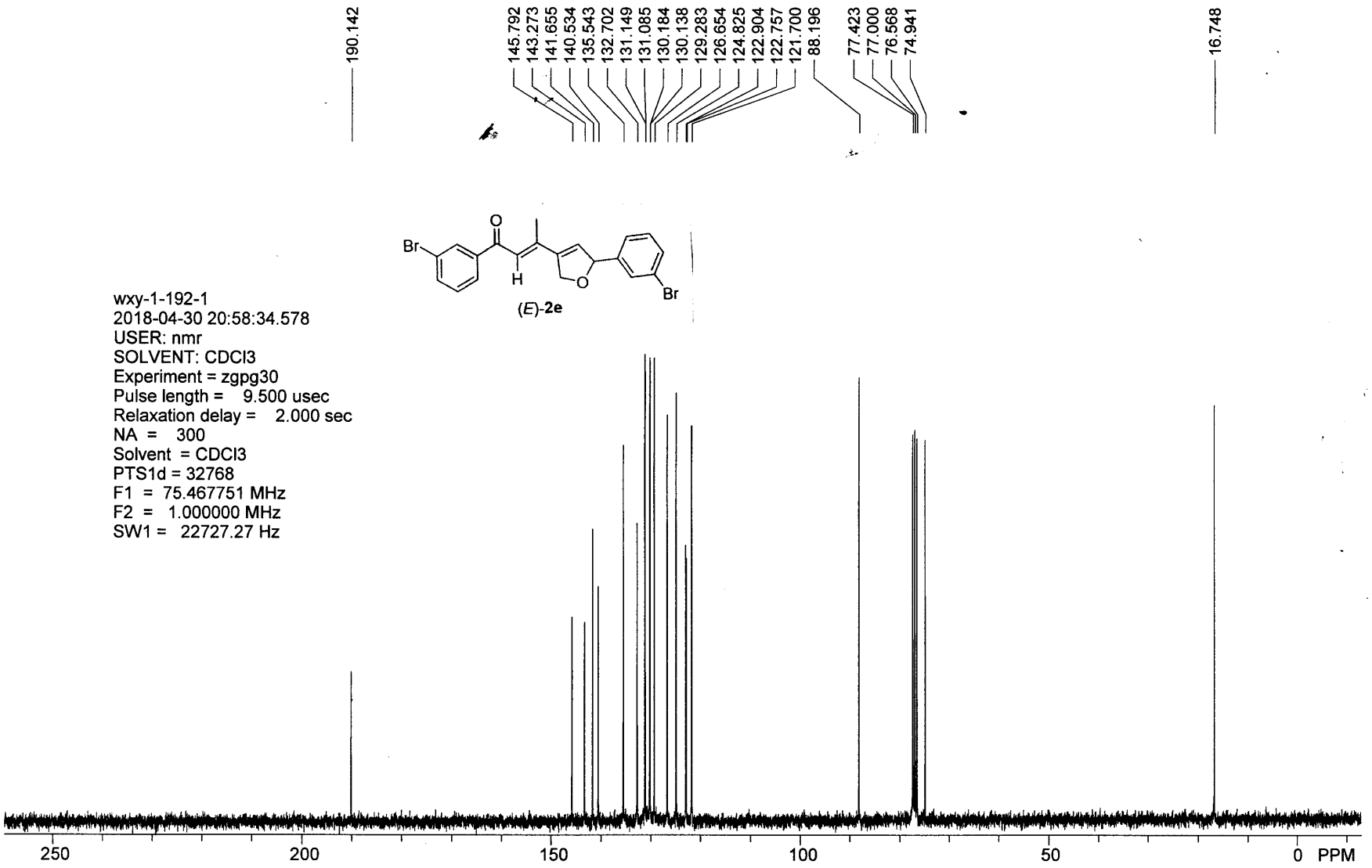


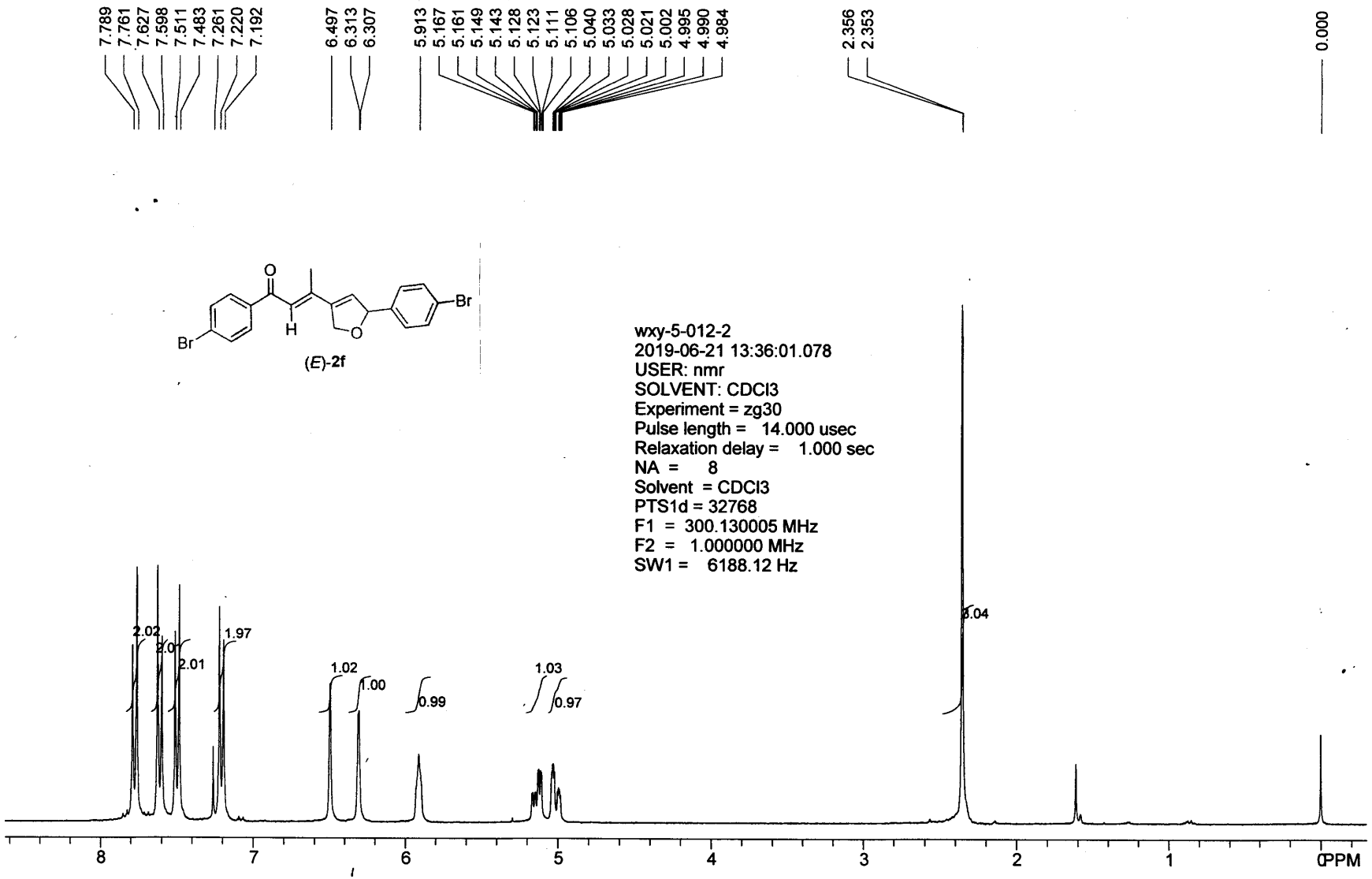


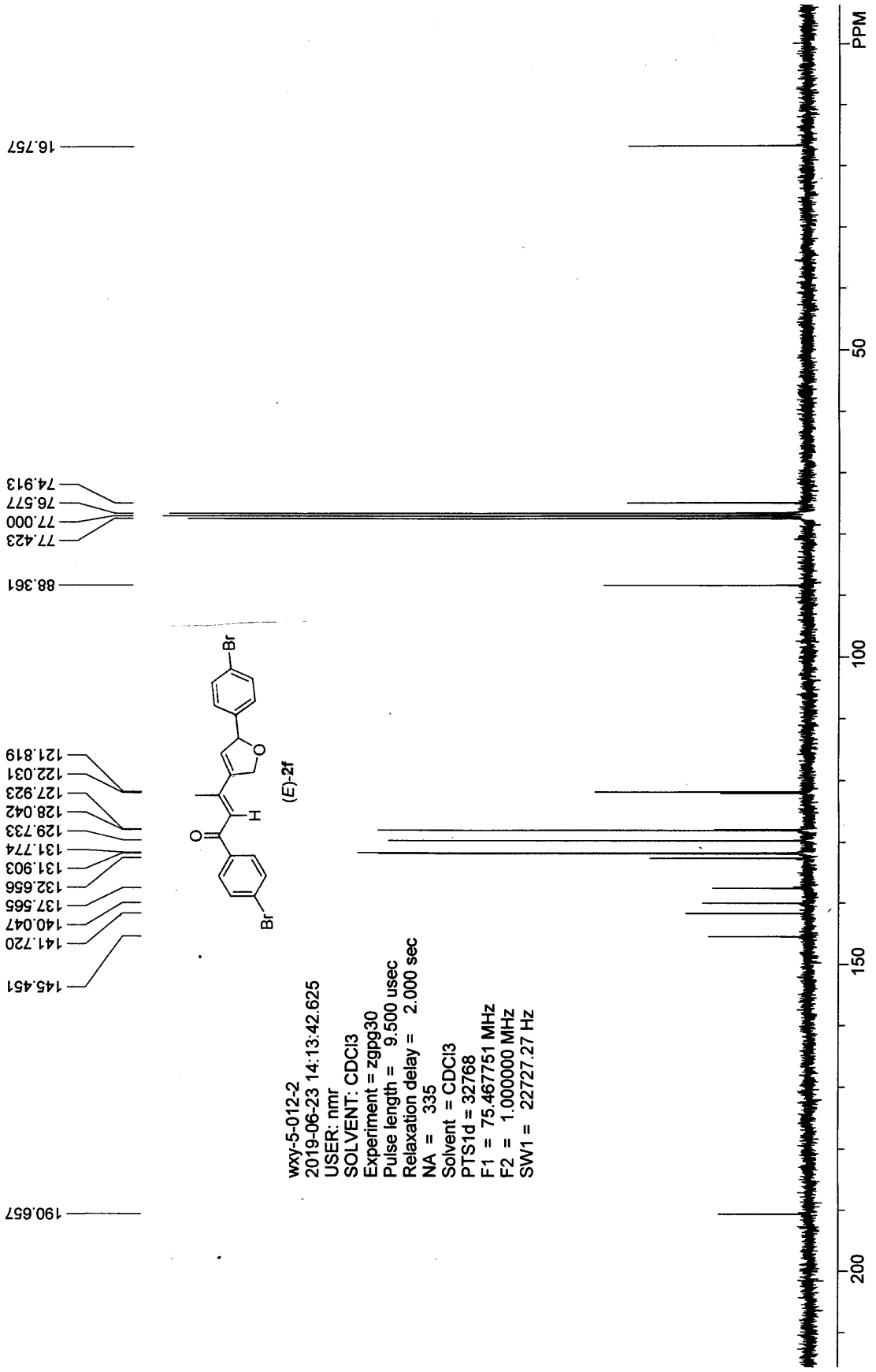
(*E*)-**2e**

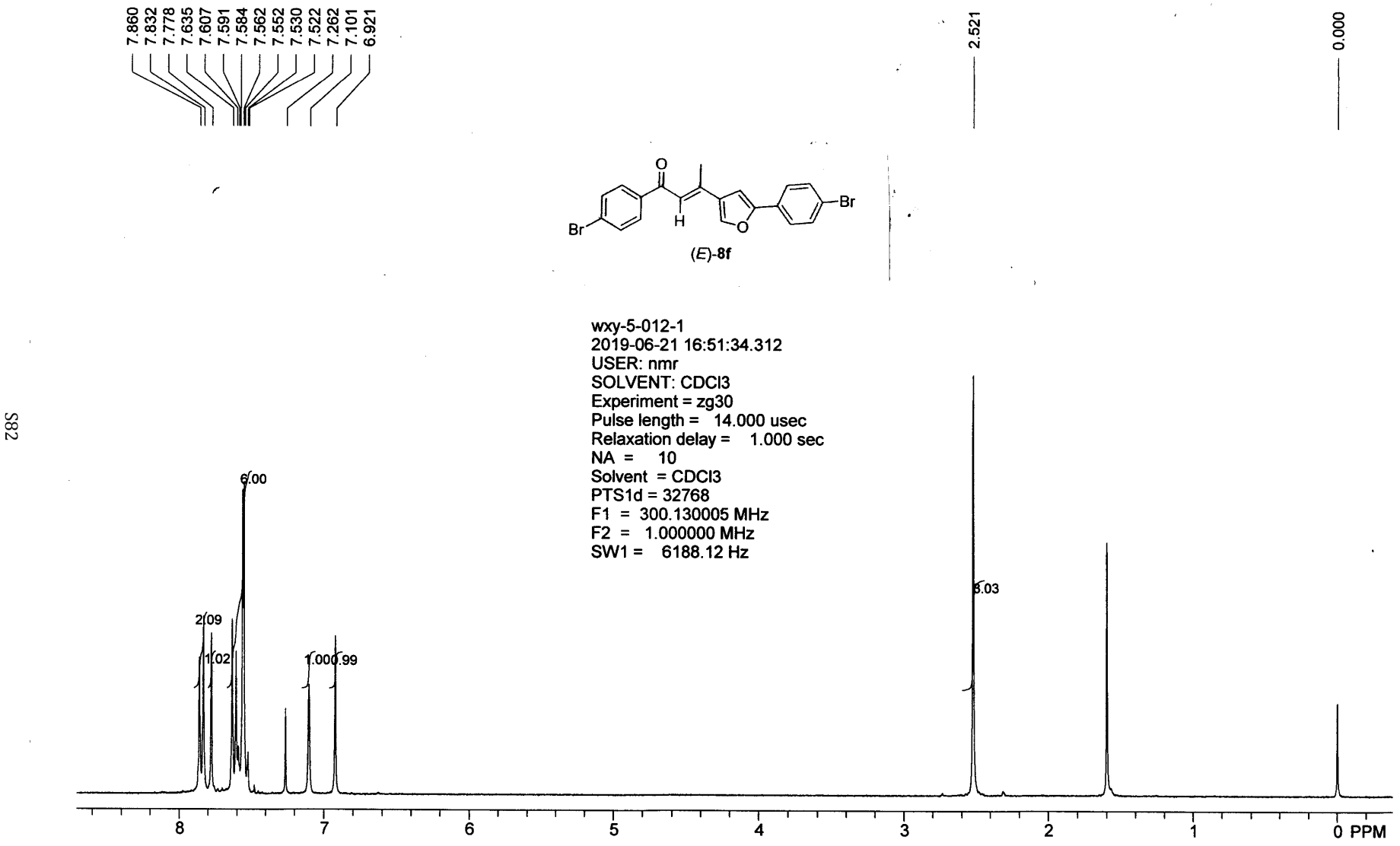
wxy-1-192-1
2018-04-30 19:04:31.281
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz

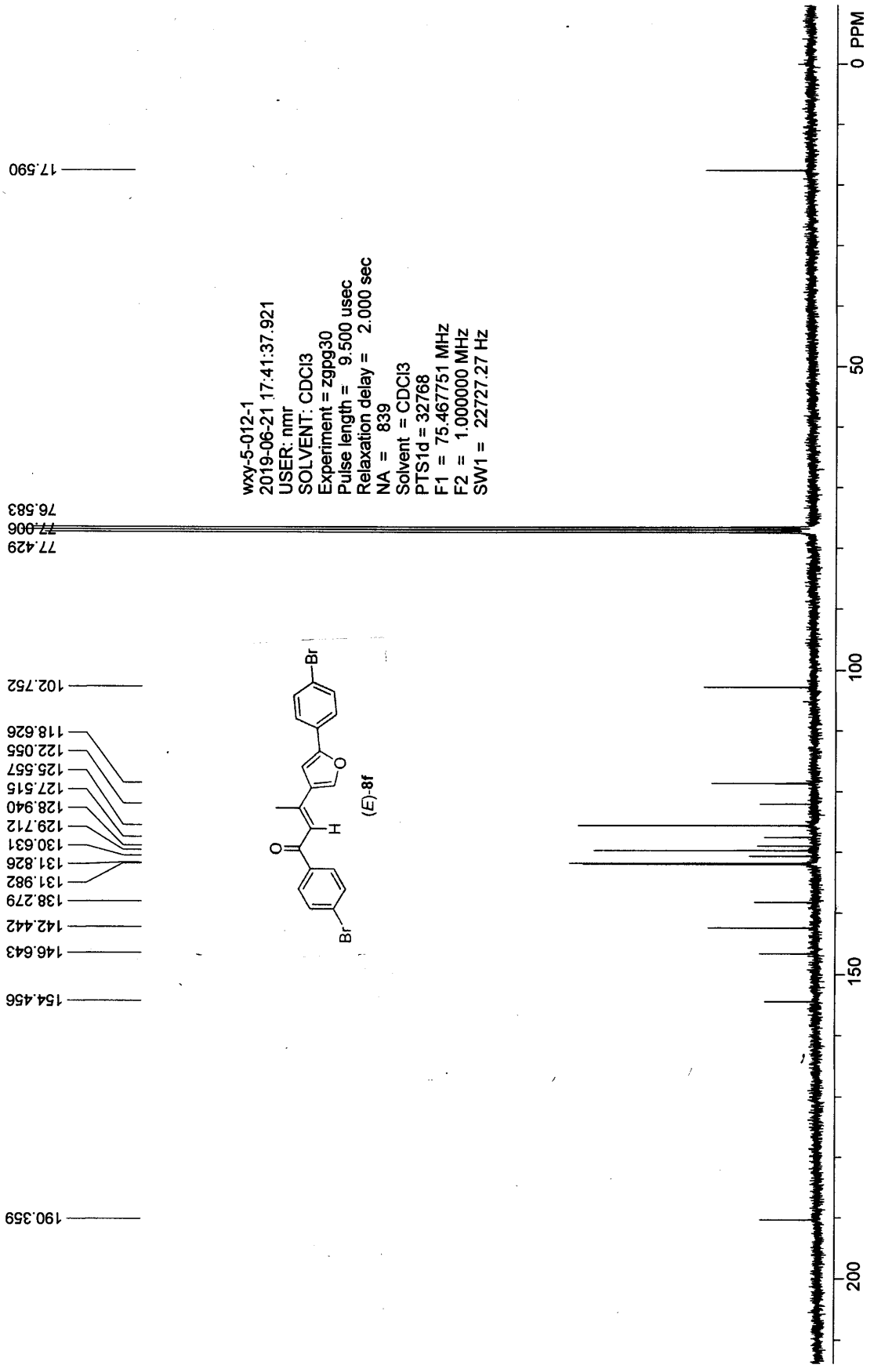


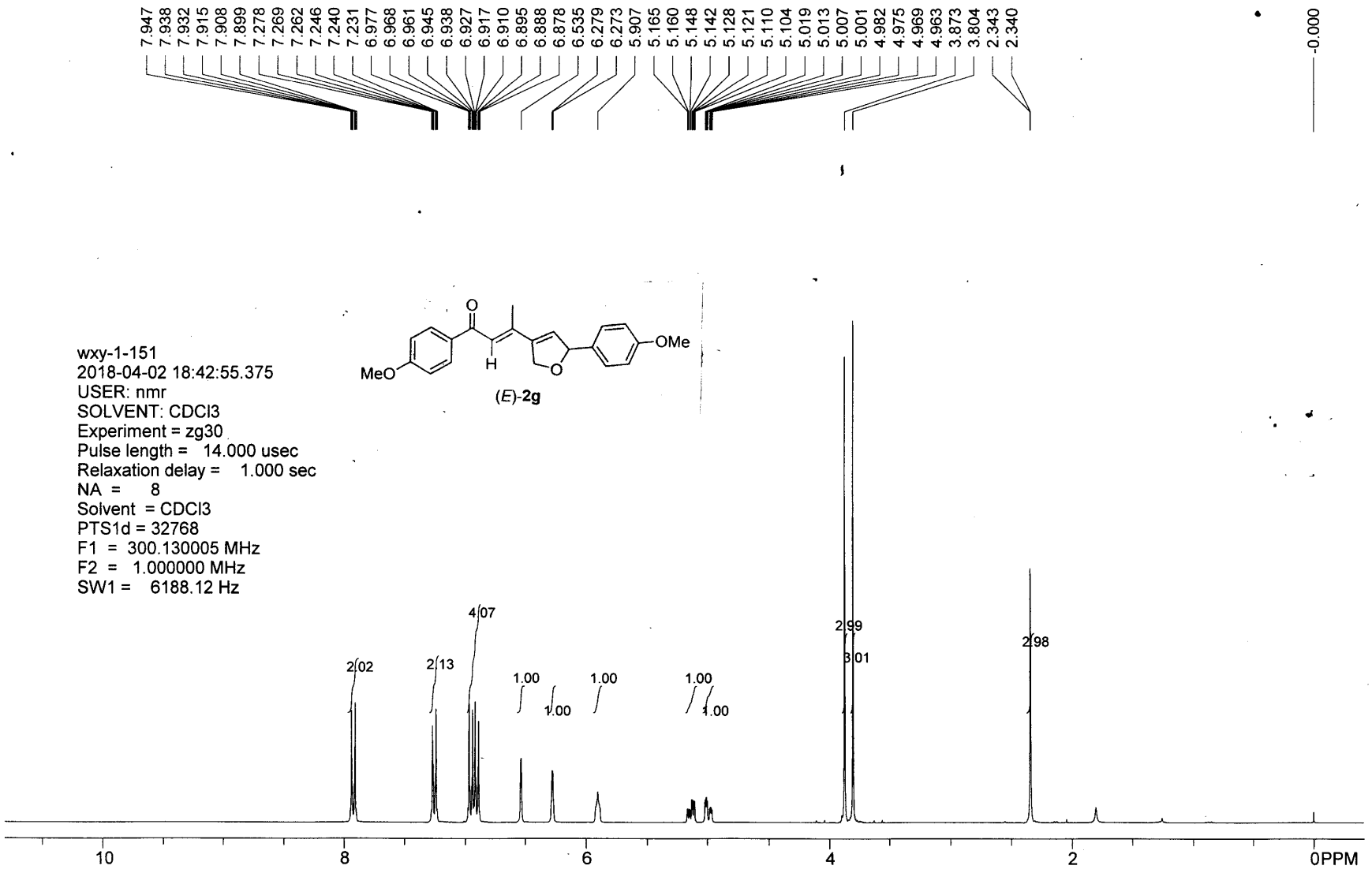

190.142
145.792
143.273
141.655
140.534
135.543
132.702
131.149
131.085
130.184
130.138
129.283
126.654
124.825
122.904
122.757
121.700
88.196
77.423
77.000
76.568
74.941
16.748
Br
O
H
O
Br
(E)-2e
wxy-1-192-1
2018-04-30 20:58:34.578
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 300
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
250
200
150
100
50
0 PPM

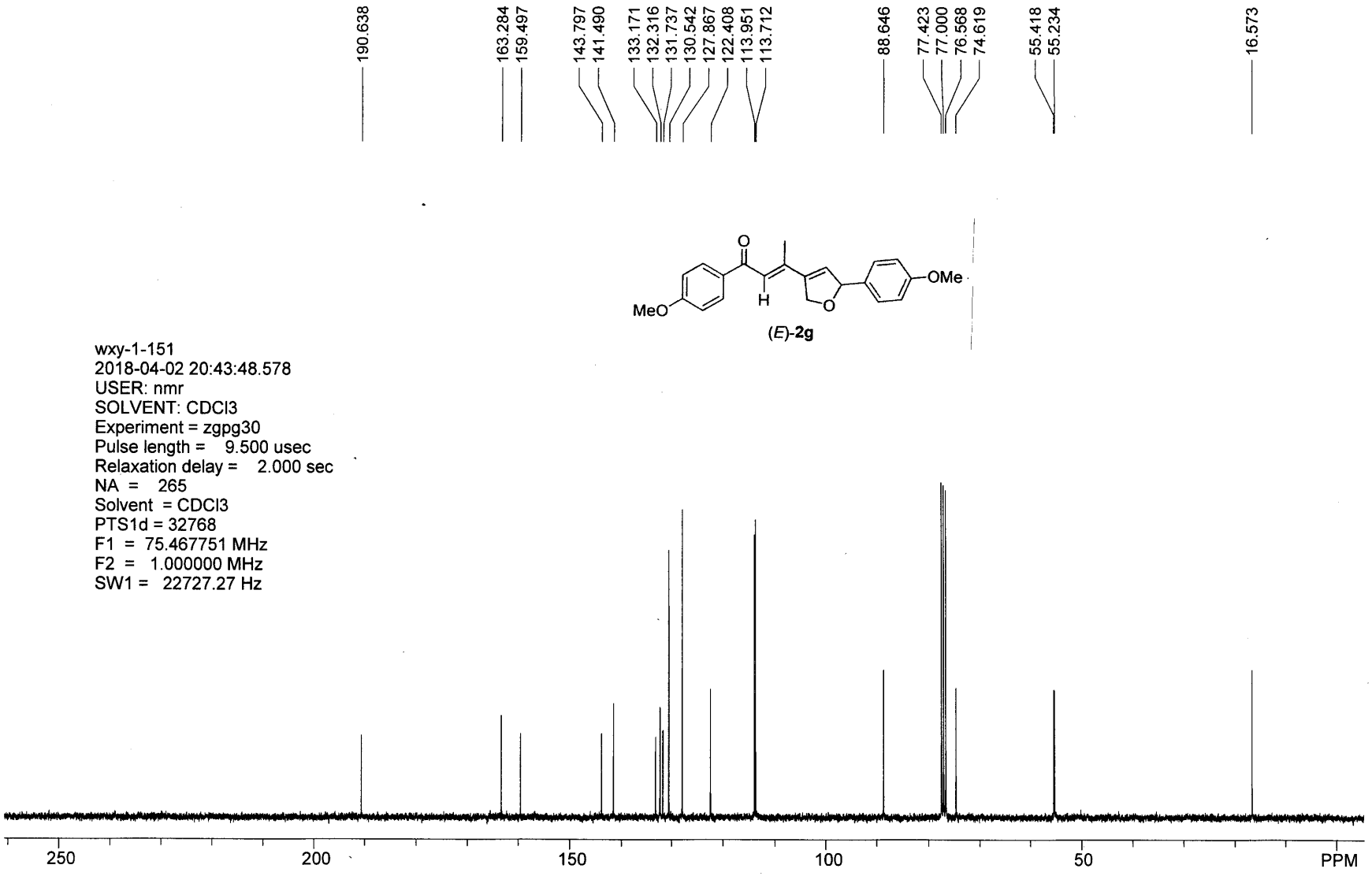

7.789
7.761
7.627
7.598
7.511
7.483
7.261
7.220
7.192
6.497
6.313
6.307
5.913
5.167
5.161
5.149
5.143
5.128
5.123
5.111
5.106
5.040
5.033
5.028
5.021
5.002
4.995
4.990
4.984
2.356
2.353
0.000
O
H
Br
O
Br
(E)-2f
wxy-5-012-2
2019-06-21 13:36:01.078
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
2.02
2.01
2.01
1.97
1.02
1.00
0.99
1.03
0.97
3.04
8
7
6
5
4
3
2
1
0
PPM

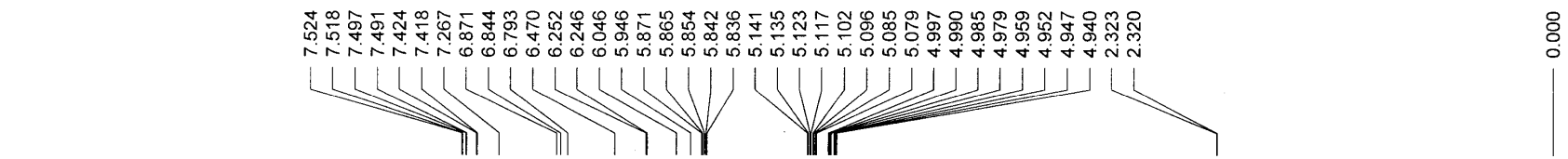

190.657
145.451
141.720
140.047
137.565
132.656
131.903
131.774
129.733
128.042
127.923
122.031
121.819
88.361
77.423
77.000
76.577
74.913
16.757
PPM
50
100
150
200
wxy-5-012-2
2019-06-23 14:13:42.625
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 335
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
(E)-2f

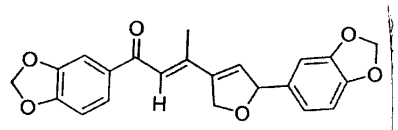

7.860
7.832
7.778
7.635
7.607
7.591
7.584
7.562
7.552
7.530
7.522
7.262
7.101
6.921
2.521
0.000
O
H
O
Br
Br
(E)-8f
wxy-5-012-1
2019-06-21 16:51:34.312
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 10
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
6.00
2.09
1.02
1.00
0.99
6.03
8
7
6
5
4
3
2
1
0 PPM


wxy-5-012-1
2019-06-21 17:41:37.921
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 839
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
(E)-8f
17.590
76.583
77.006
77.429
102.752
118.626
122.055
125.557
127.515
128.940
129.712
130.631
131.826
131.982
138.279
142.442
146.643
154.456
190.359
0 PPM
50
100
150
200

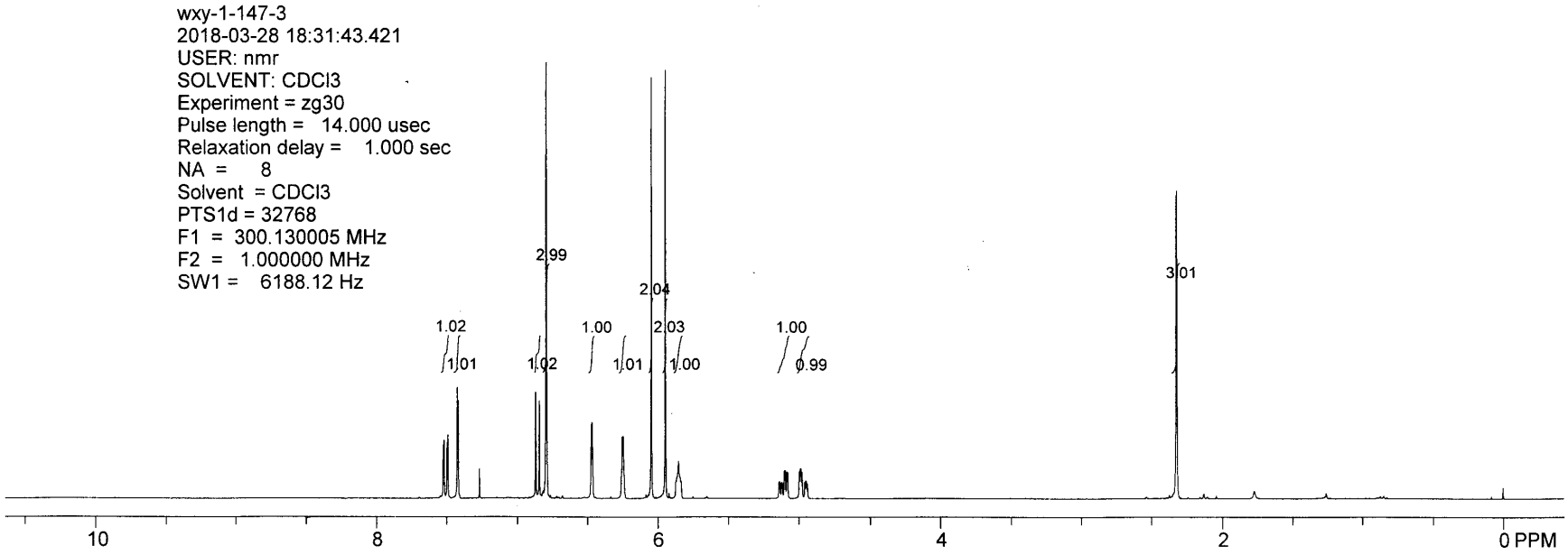

7.947
7.938
7.932
7.915
7.908
7.899
7.278
7.269
7.262
7.246
7.240
7.231
6.977
6.968
6.961
6.945
6.938
6.927
6.917
6.910
6.895
6.888
6.878
6.535
6.279
6.273
5.907
5.165
5.160
5.148
5.142
5.128
5.121
5.110
5.104
5.019
5.013
5.007
5.001
4.982
4.975
4.969
4.963
3.873
3.804
2.343
2.340
-0.000
wxy-1-151
2018-04-02 18:42:55.375
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
O
H
MeO
OMe
O
(E)-2g
2.02
2.13
4.07
1.00
1.00
1.00
1.00
1.00
2.99
3.01
2.98
10
8
6
4
2
0PPM

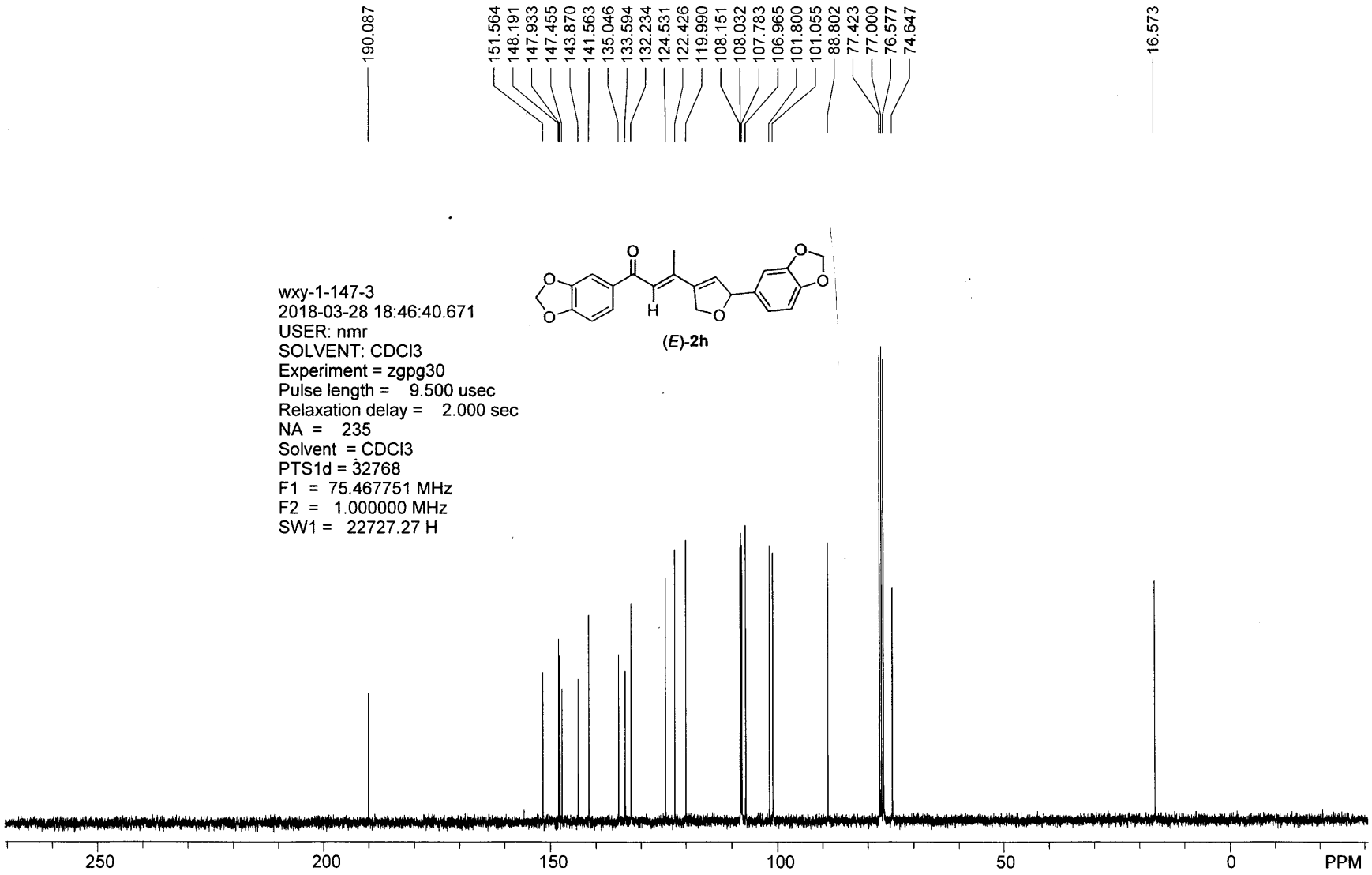

190.638
163.284
159.497
143.797
141.490
133.171
132.316
131.737
130.542
127.867
122.408
113.951
113.712
88.646
77.423
77.000
76.568
74.619
55.418
55.234
16.573
O
H
O
MeO
OMe
(E)-2g
wxy-1-151
2018-04-02 20:43:48.578
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 265
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
250
200
150
100
50
PPM

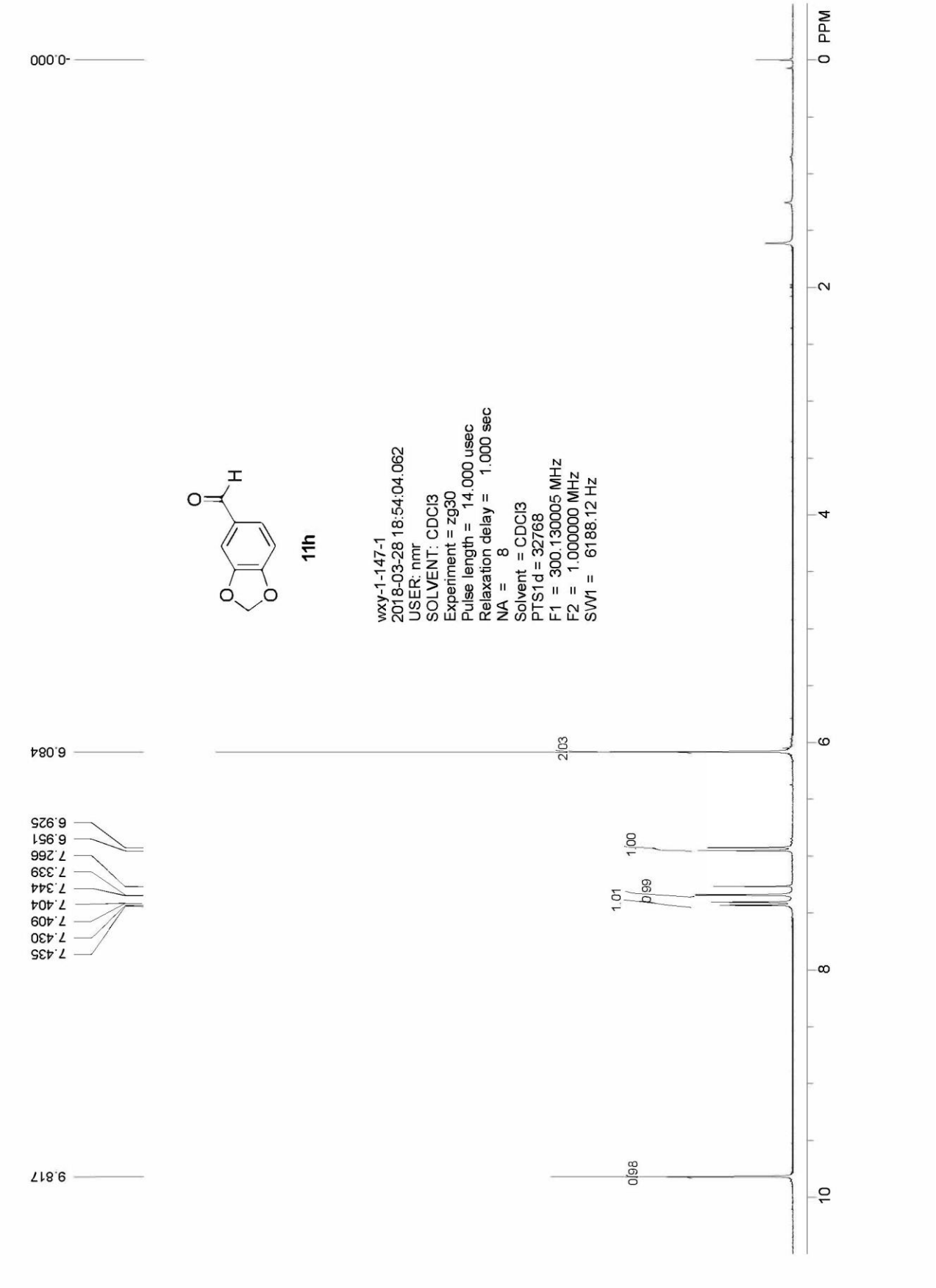

7.524
7.518
7.497
7.491
7.424
7.418
7.267
6.871
6.844
6.793
6.470
6.252
6.246
6.046
5.946
5.871
5.865
5.854
5.842
5.836
5.141
5.135
5.123
5.117
5.102
5.096
5.085
5.079
4.997
4.990
4.985
4.979
4.959
4.952
4.947
4.940
2.323
2.320
0.000

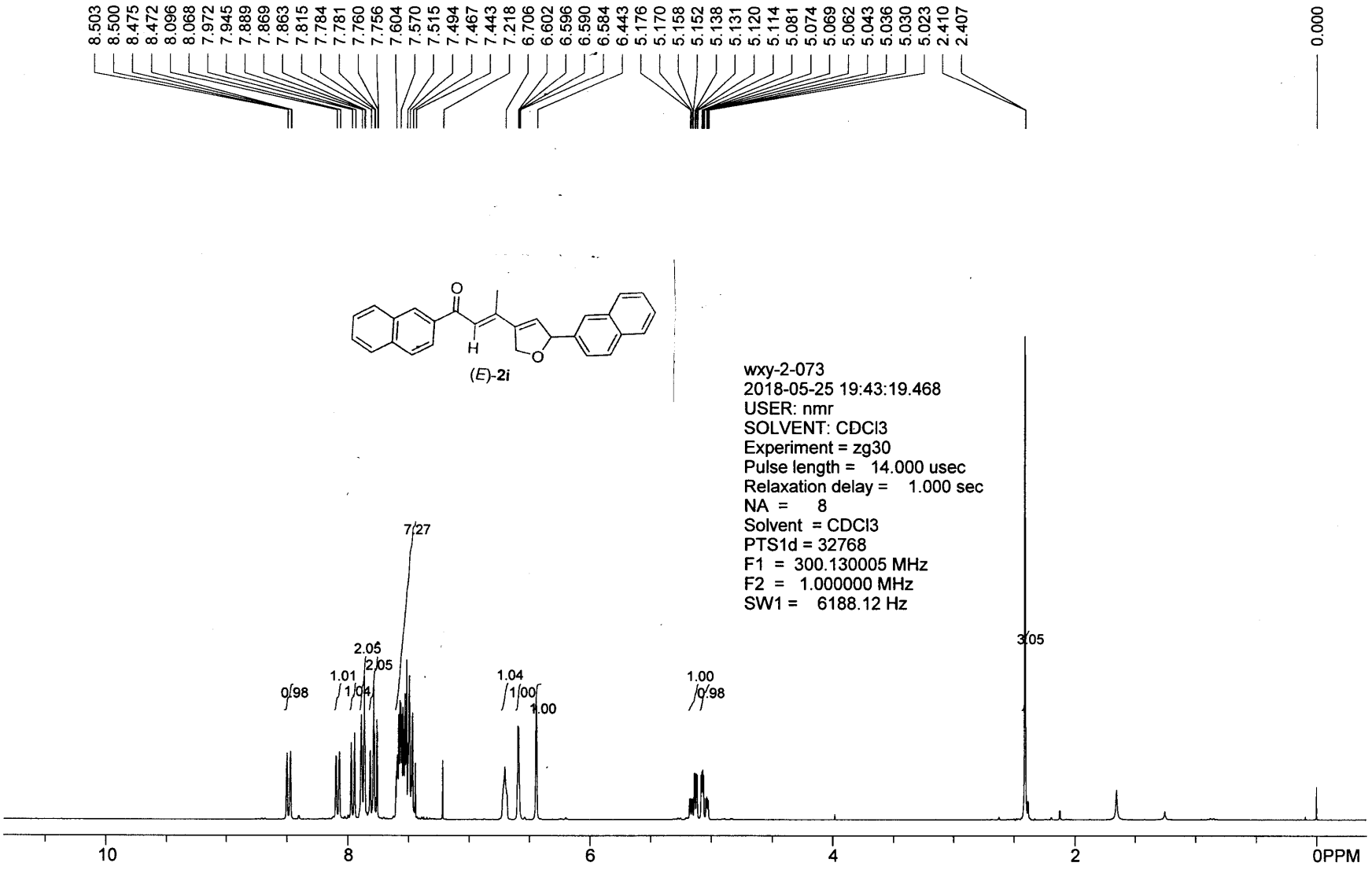


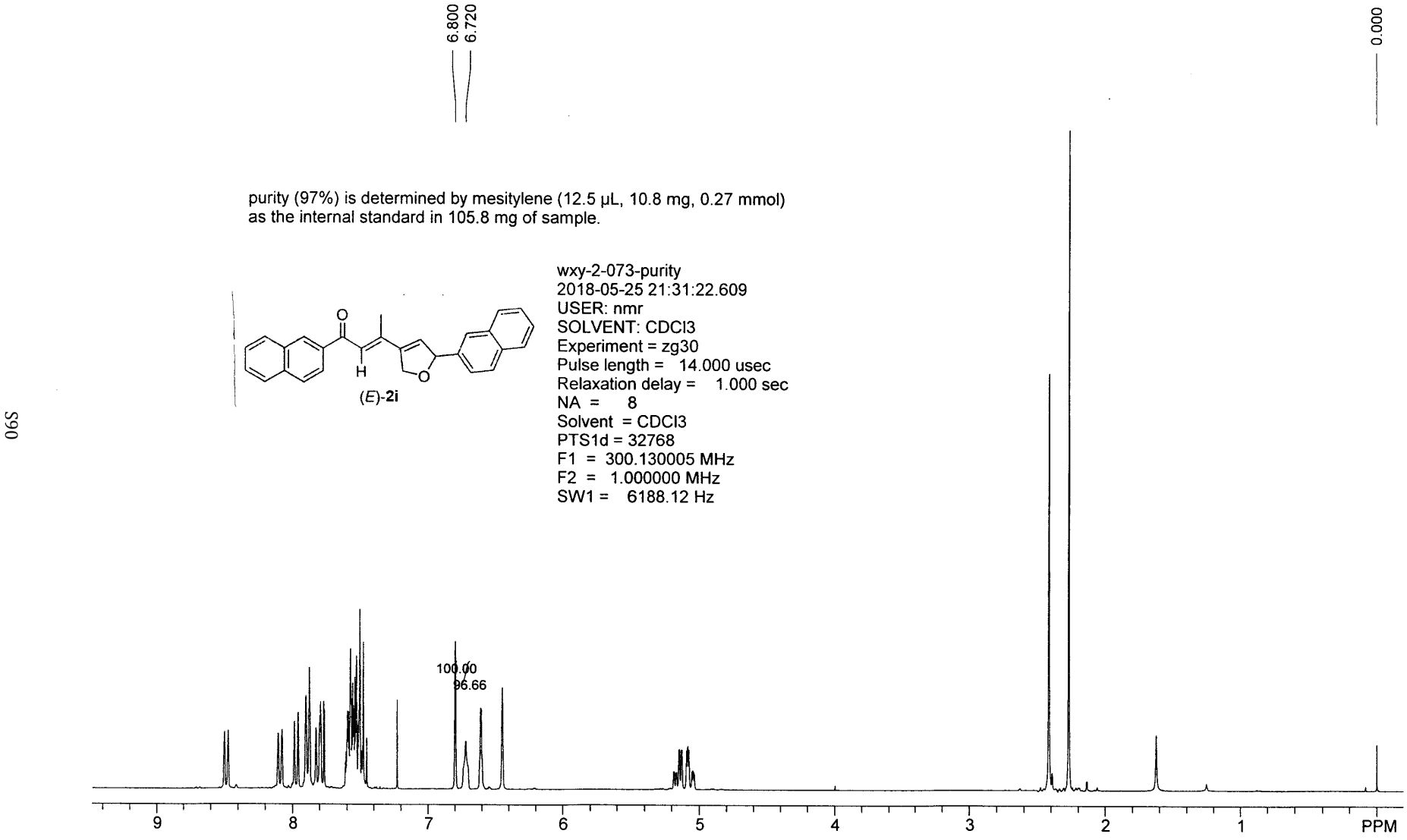
(*E*)-**2h**

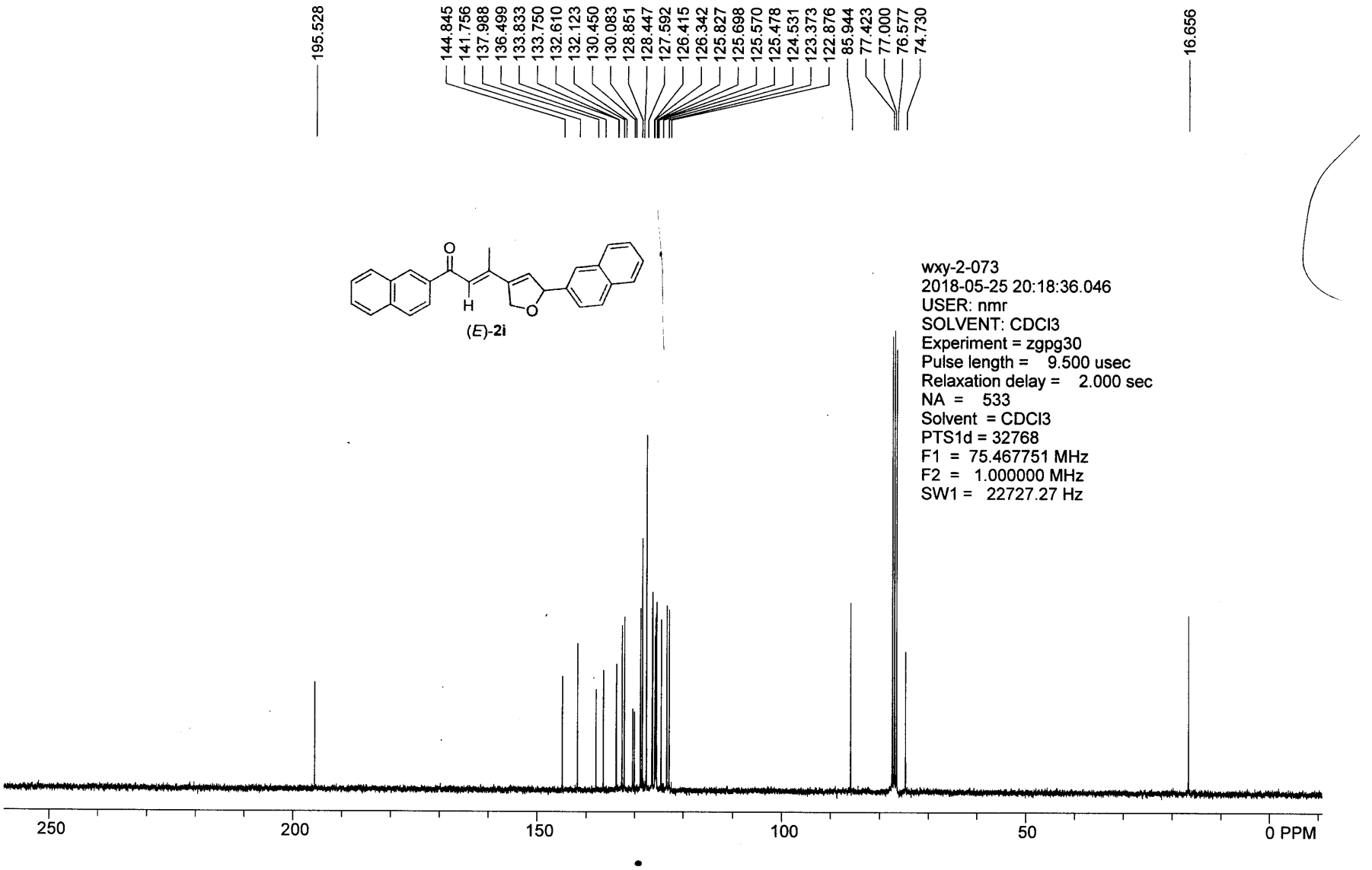

wxy-1-147-3
2018-03-28 18:31:43.421
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
1.02
1.01
2.99
1.02
1.00
1.01
2.04
2.03
1.00
1.00
0.99
3.01
10
8
6
4
2
0 PPM

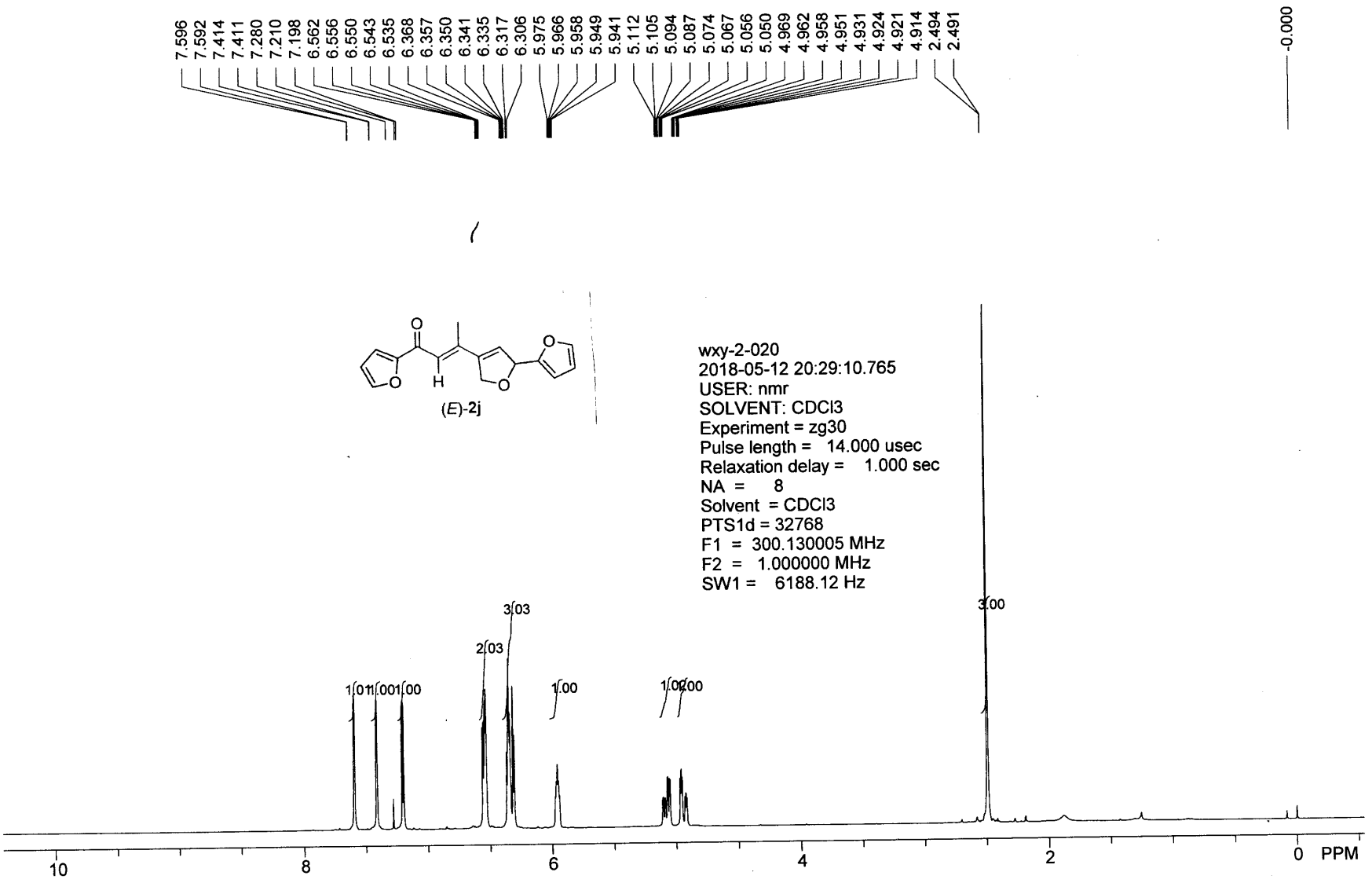

190.087
151.564
148.191
147.933
147.455
143.870
141.563
135.046
133.594
132.234
124.531
122.426
119.990
108.151
108.032
107.783
106.965
101.800
101.055
88.802
77.423
77.000
76.577
74.647
16.573
wxy-1-147-3
2018-03-28 18:46:40.671
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 235
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 H
(E)-2h
250
200
150
100
50
0
PPM

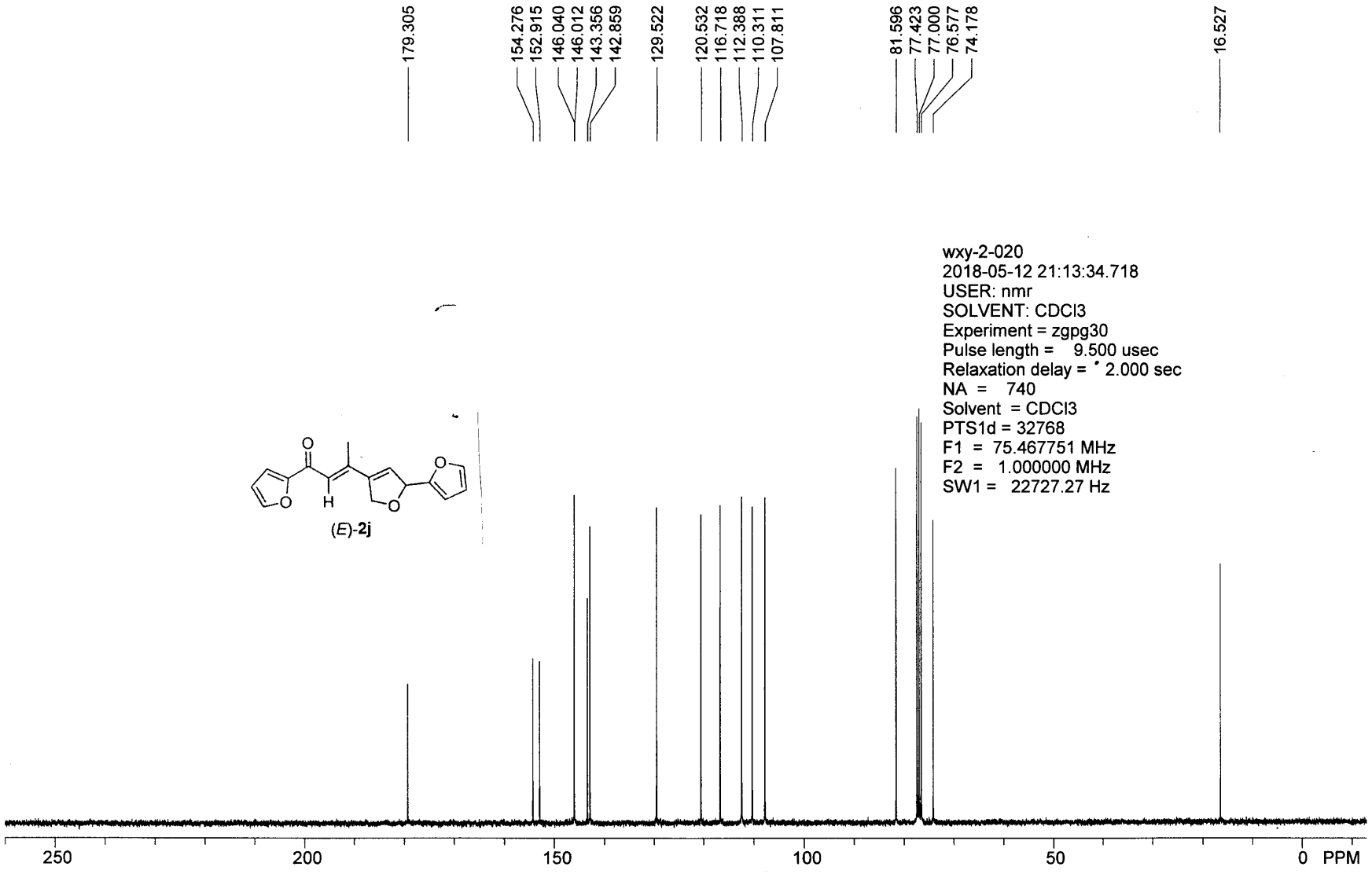

11h
wxy-1-147-1
2018-03-28 18:54:04.062
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
9.817
6.084
7.435
7.430
7.409
7.404
7.344
7.339
7.266
6.951
6.925
0.000
2.03
1.00
0.99
1.01
0.98
0 PPM
2
4
6
8
10

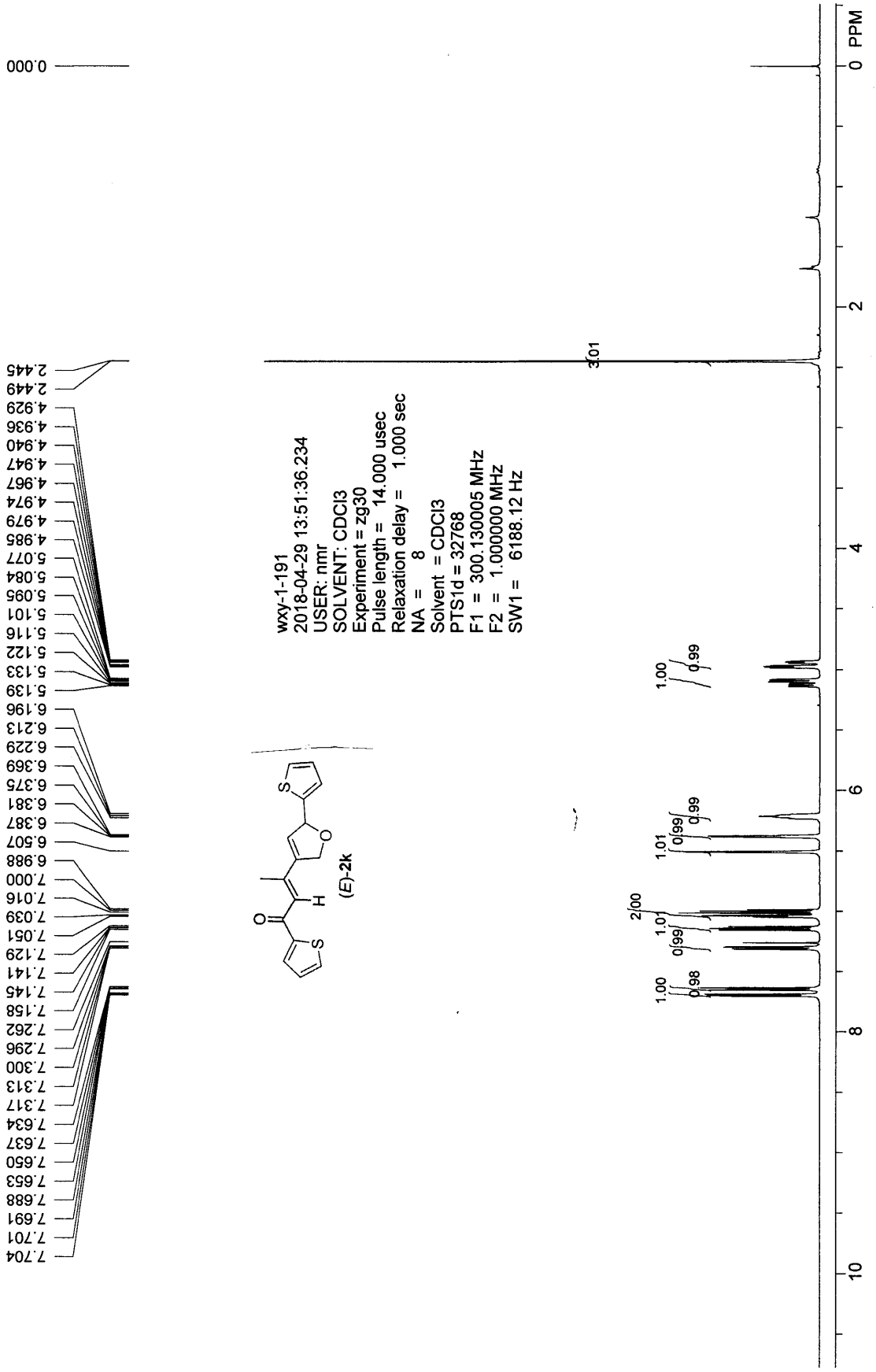

8.503
8.500
8.475
8.472
8.096
8.068
7.972
7.945
7.889
7.869
7.863
7.815
7.784
7.781
7.760
7.756
7.604
7.570
7.515
7.494
7.467
7.443
7.218
6.706
6.602
6.596
6.590
6.584
6.443
5.176
5.170
5.158
5.152
5.138
5.131
5.120
5.114
5.081
5.074
5.069
5.062
5.043
5.036
5.030
5.023
2.410
2.407
0.000
O
H
O
(E)-2i
wxy-2-073
2018-05-25 19:43:19.468
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
0.98
1.01
1.04
2.05
2.05
7.27
1.04
1.00
1.00
1.00
0.98
3.05
10
8
6
4
2
0PPM

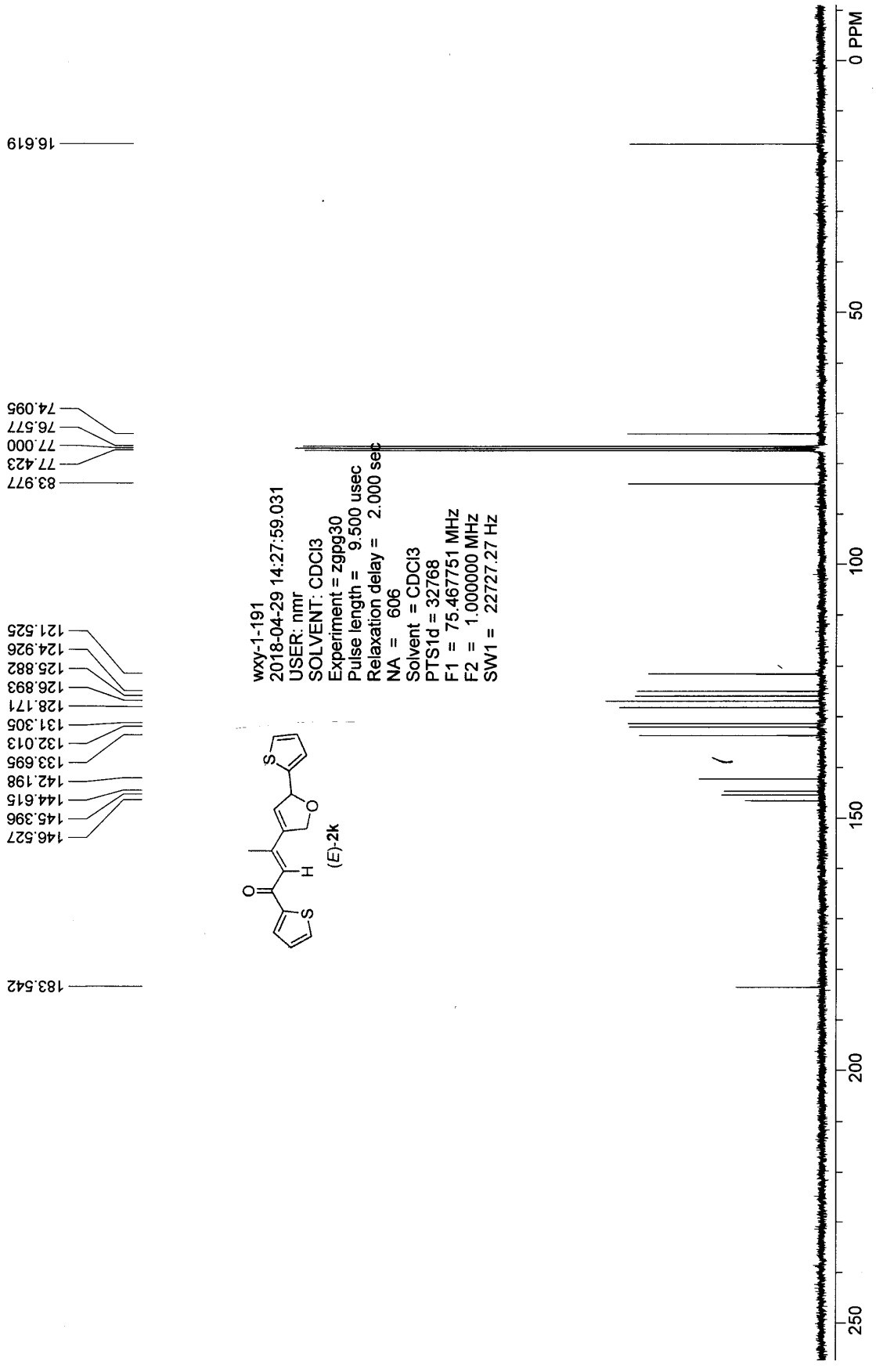

6.800
6.720
0.000
purity (97%) is determined by mesitylene (12.5 µL, 10.8 mg, 0.27 mmol)
as the internal standard in 105.8 mg of sample.
O
H
O
(E)-2i
wxy-2-073-purity
2018-05-25 21:31:22.609
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
100.00
96.66
9
8
7
6
5
4
3
2
1
PPM


195.528
144.845
141.756
137.988
136.499
133.833
133.750
132.610
132.123
130.450
130.083
128.851
128.447
127.592
126.415
126.342
125.827
125.698
125.570
125.478
124.531
123.373
122.876
85.944
77.423
77.000
76.577
74.730
16.656
O
H
O
(E)-2i
wxy-2-073
2018-05-25 20:18:36.046
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 533
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
250
200
150
100
50
0 PPM


7.596
7.592
7.414
7.411
7.280
7.210
7.198
6.562
6.556
6.550
6.543
6.535
6.368
6.357
6.350
6.341
6.335
6.317
6.306
5.975
5.966
5.958
5.949
5.941
5.112
5.105
5.094
5.087
5.074
5.067
5.056
5.050
4.969
4.962
4.958
4.951
4.931
4.924
4.921
4.914
2.494
2.491
-0.000
(E)-2j
wxy-2-020
2018-05-12 20:29:10.765
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
1.01
1.00
1.00
2.03
3.03
1.00
1.00
3.00
10
8
6
4
2
0
PPM


179.305
154.276
152.915
146.040
146.012
143.356
142.859
129.522
120.532
116.718
112.388
110.311
107.811
81.596
77.423
77.000
76.577
74.178
16.527
wxy-2-020
2018-05-12 21:13:34.718
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 740
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
(E)-2j
250
200
150
100
50
0 PPM


wxy-1-191
2018-04-29 13:51:36.234
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
(E)-2k
0.000
2.445
2.449
4.929
4.936
4.940
4.947
4.967
4.974
4.979
4.985
5.077
5.084
5.095
5.101
5.116
5.122
5.133
5.139
6.196
6.213
6.229
6.369
6.375
6.381
6.387
6.507
6.988
7.000
7.016
7.039
7.051
7.129
7.141
7.145
7.158
7.262
7.296
7.300
7.313
7.317
7.634
7.637
7.650
7.653
7.688
7.691
7.701
7.704
3.01
1.00
0.99
1.01
0.99
0.99
2.00
1.01
0.99
1.00
0.98
0 PPM


wxy-1-191
2018-04-29 14:27:59.031
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 606
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
(E)-2k
183.542
146.527
145.396
144.615
142.198
133.695
132.013
131.305
128.171
126.893
125.882
124.926
121.525
83.977
77.423
77.000
76.577
74.095
16.619
250
200
150
100
50
0 PPM

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) 180508_wxy_1_191

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: 180508_wxy_1_191

Bond precision: C-C = 0.0043 A Wavelength=0.71073

Cell: a=8.1839(7) b=17.0403(12) c=21.7793(14)
alpha=90 beta=90 gamma=90
Temperature: 293 K

	Calculated	Reported
Volume	3037.3(4)	3037.2(4)
Space group	P b c a	P b c a
Hall group	-P 2ac 2ab	-P 2ac 2ab
Moiety formula	C16 H14 O2 S2	C16 H14 O2 S2
Sum formula	C16 H14 O2 S2	C16 H14 O2 S2
Mr	302.39	302.39
Dx,g cm-3	1.323	1.323
Z	8	8
Mu (mm-1)	0.348	0.348
F000	1264.0	1264.0
F000'	1266.47	
h,k,lmax	9,20,26	9,20,26
Nref	2781	2777
Tmin,Tmax	0.843,0.870	0.865,1.000
Tmin'	0.843	

Correction method= # Reported T Limits: Tmin=0.865 Tmax=1.000
AbsCorr = MULTI-SCAN

Data completeness= 0.999 Theta(max)= 25.349

R(reflections)= 0.0502(1994) wR2(reflections)= 0.1420(2777)

S = 1.035 Npar= 181

The following ALERTS were generated. Each ALERT has the format
test-name_ALERT_alert-type_alert-level.
Click on the hyperlinks for more details of the test.

Alert level C

```
PLAT340_ALERT_3_C Low Bond Precision on  C-C Bonds ..............    0.00427 Ang.
PLAT978_ALERT_2_C Number C-C Bonds with Positive Residual Density.         0 Info
```

Alert level G

```
PLAT199_ALERT_1_G Reported _cell_measurement_temperature ..... (K)       293 Check
PLAT200_ALERT_1_G Reported   _diffrn_ambient_temperature ..... (K)       293 Check
PLAT380_ALERT_4_G Incorrectly? Oriented X(sp2)-Methyl Moiety .....       C16 Check
PLAT398_ALERT_2_G Deviating  C-O-C    Angle From 120 for O2             109.5 Degree
PLAT793_ALERT_4_G Model has Chirality at C10         (Centro SPGR)         S Verify
PLAT910_ALERT_3_G Missing # of FCF Reflection(s) Below Theta(Min).         4 Note
```

```
   0 ALERT level A = Most likely a serious problem - resolve or explain
   0 ALERT level B = A potentially serious problem, consider carefully
   2 ALERT level C = Check. Ensure it is not caused by an omission or oversight
   6 ALERT level G = General information/check it is not something unexpected

   2 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
   2 ALERT type 2 Indicator that the structure model may be wrong or deficient
   2 ALERT type 3 Indicator that the structure quality may be low
   2 ALERT type 4 Improvement, methodology, query or suggestion
   0 ALERT type 5 Informative message, check
```

It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

Publication of your CIF in IUCr journals

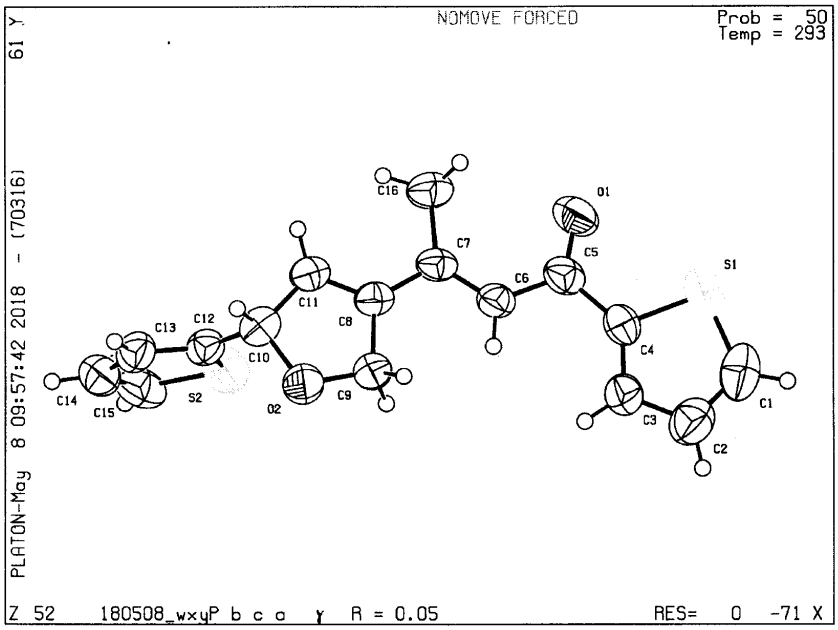
A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica, Journal of Applied Crystallography, Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E* or *IUCrData*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

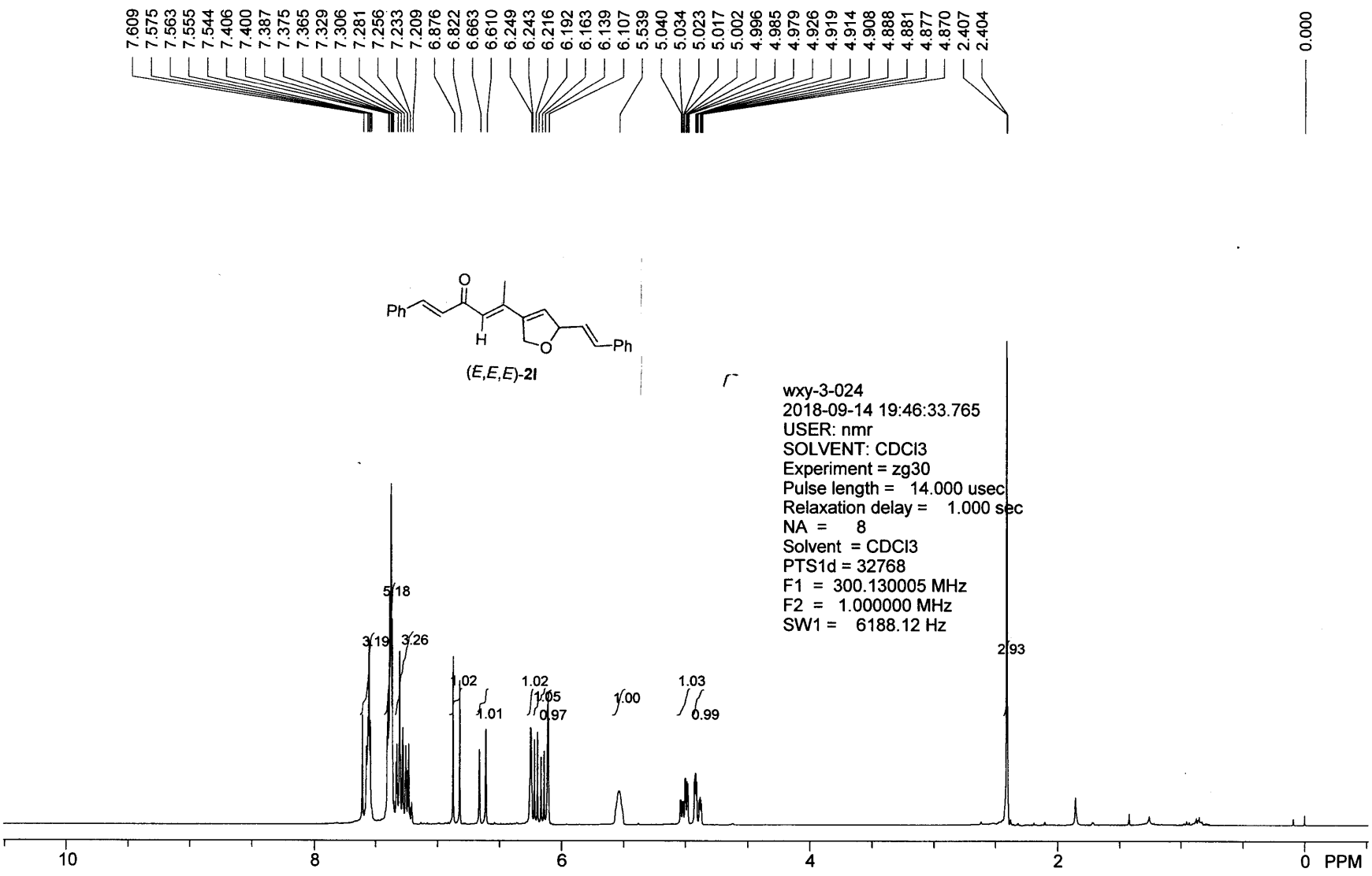
Publication of your CIF in other journals

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

PLATON version of 23/04/2018; check.def file version of 23/04/2018

Datablock 180508_wxy_1_191 - ellipsoid plot

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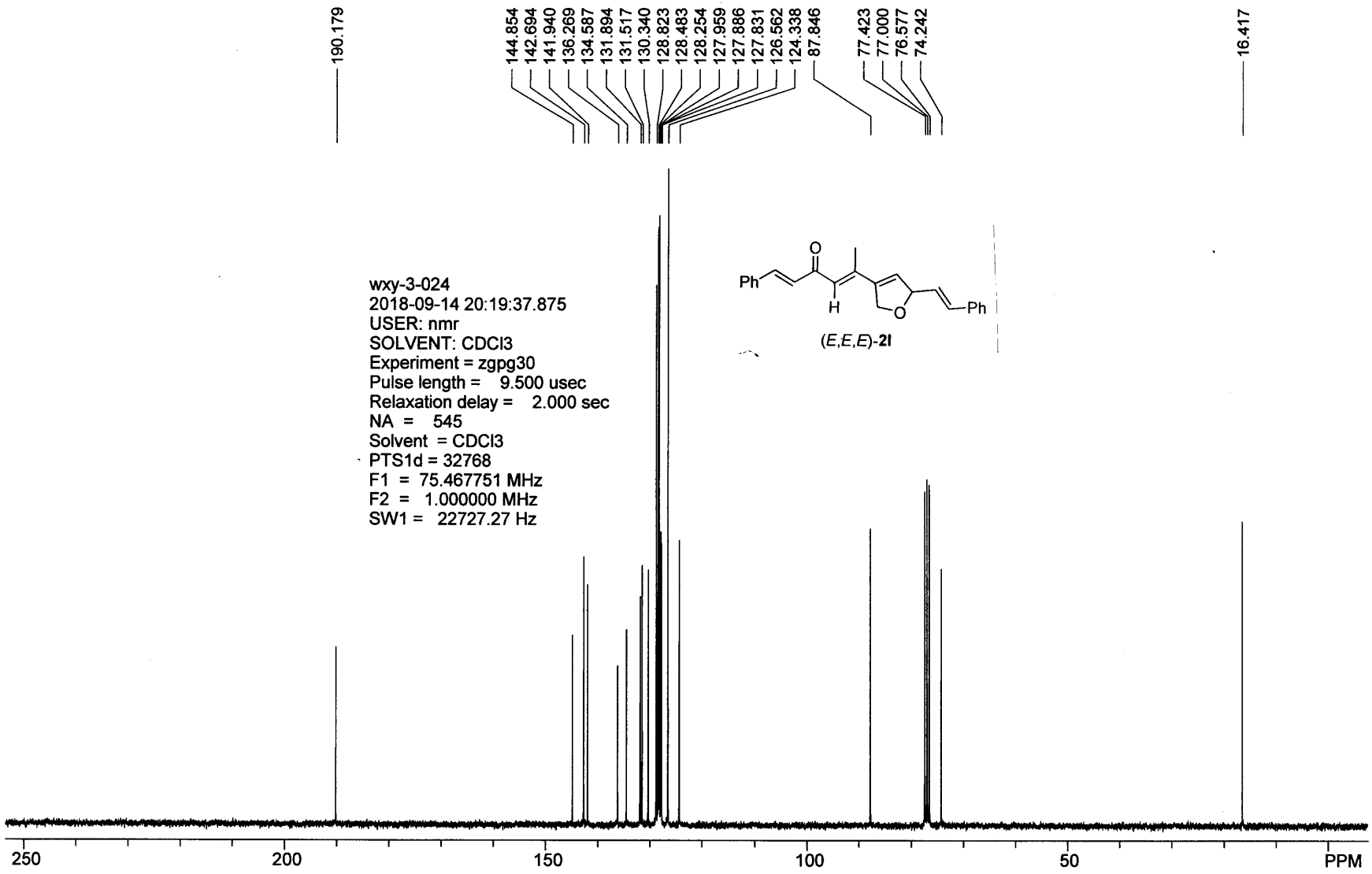
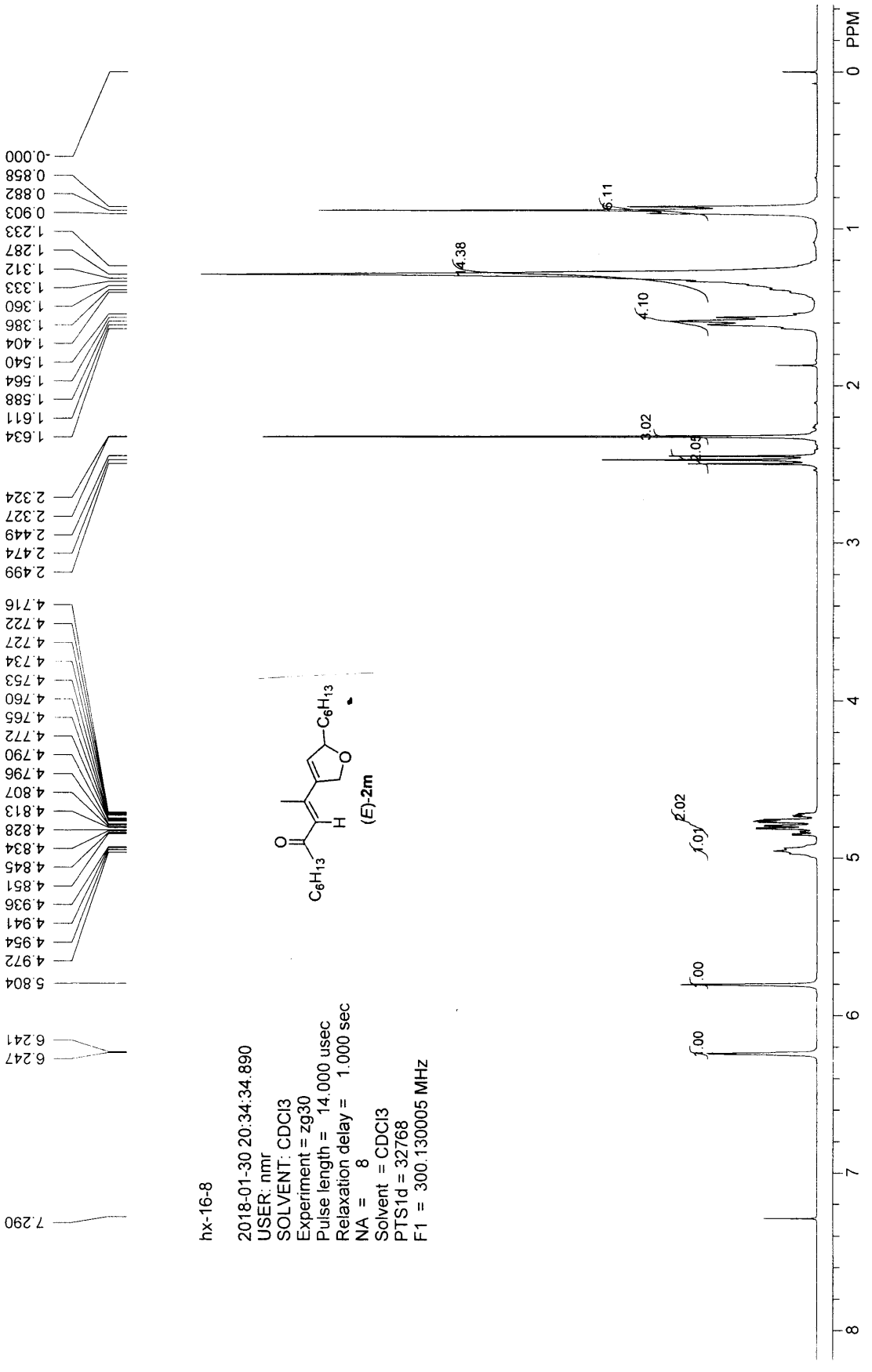
7.406
7.400
7.387
7.375
7.365
7.329
7.306
7.281
7.256
7.233
7.209
6.876
6.822
6.663
6.610
6.249
6.243
6.216
6.192
6.163
6.139
6.107
5.539
5.040
5.034
5.023
5.017
5.002
4.996
4.985
4.979
4.926
4.919
4.914
4.908
4.888
4.881
4.877
4.870
2.407
2.404
0.000
Ph
O
H
O
Ph
(E,E,E)-2l
wxy-3-024
2018-09-14 19:46:33.765
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
5.18
3.19
3.26
1.02
1.01
1.02
1.05
0.97
1.00
1.03
0.99
2.93
10
8
6
4
2
0 PPM


190.179
144.854
142.694
141.940
136.269
134.587
131.894
131.517
130.340
128.823
128.483
128.254
127.959
127.886
127.831
126.562
124.338
87.846
77.423
77.000
76.577
74.242
16.417
wxy-3-024
2018-09-14 20:19:37.875
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 545
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
Ph
O
H
O
Ph
(E,E,E)-2l
250
200
150
100
50
PPM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2018-01-30 20:34:34.890
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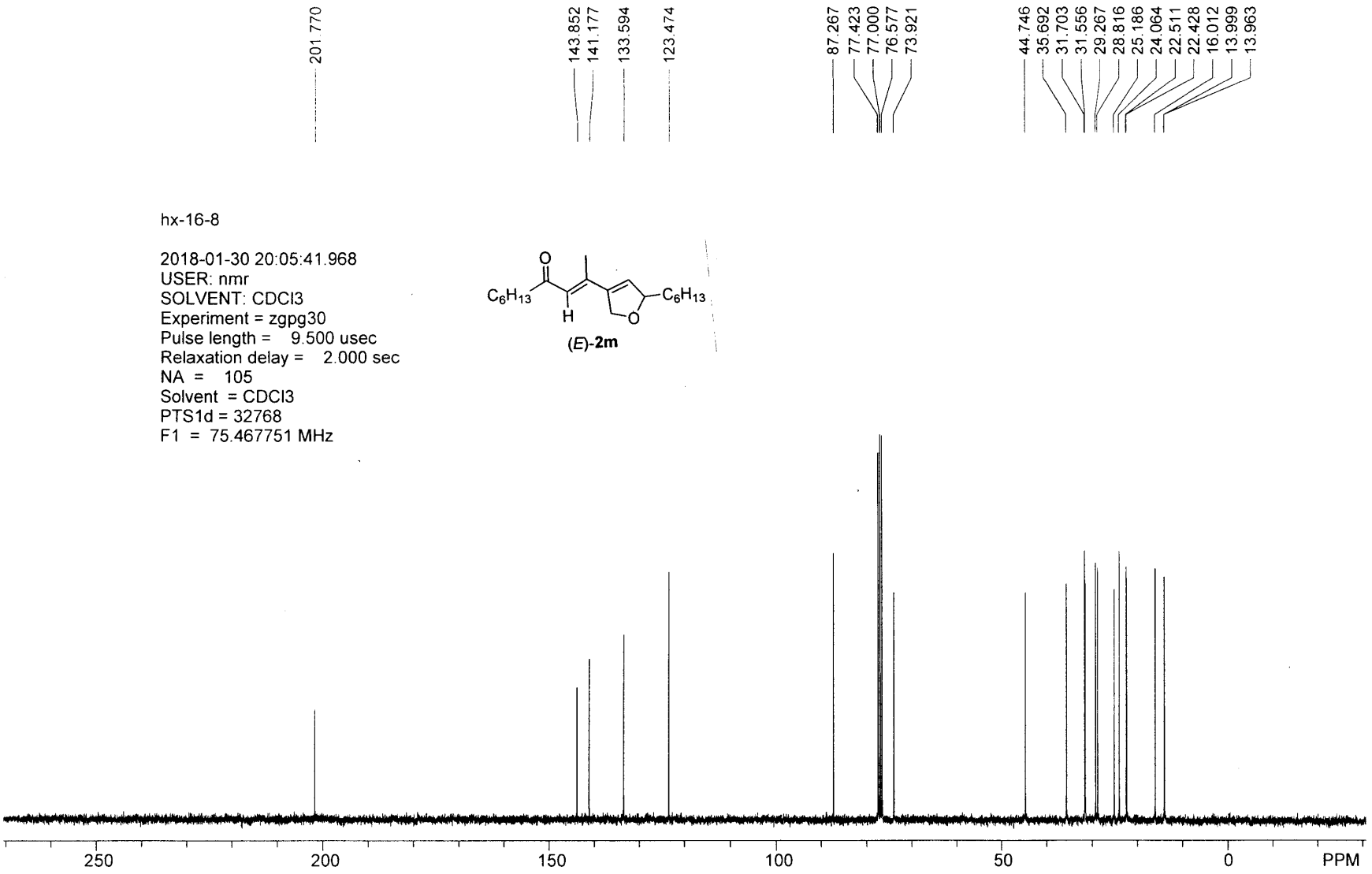
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
(E)-2m
7.290
6.247
6.241
5.804
4.972
4.954
4.941
4.936
4.851
4.845
4.834
4.828
4.813
4.807
4.796
4.790
4.772
4.765
4.760
4.753
4.734
4.727
4.722
4.716
2.499
2.474
2.449
2.327
2.324
1.634
1.611
1.588
1.564
1.540
1.404
1.386
1.360
1.333
1.312
1.287
1.233
0.903
0.882
0.858
-0.000
1.00
1.00
1.01
2.02
3.02
2.09
4.38
4.10
6.11
0
1
2
3
4
5
6
7
8
PPM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123.474
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77.423
77.000
76.577
73.921
44.746
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31.556
29.267
28.816
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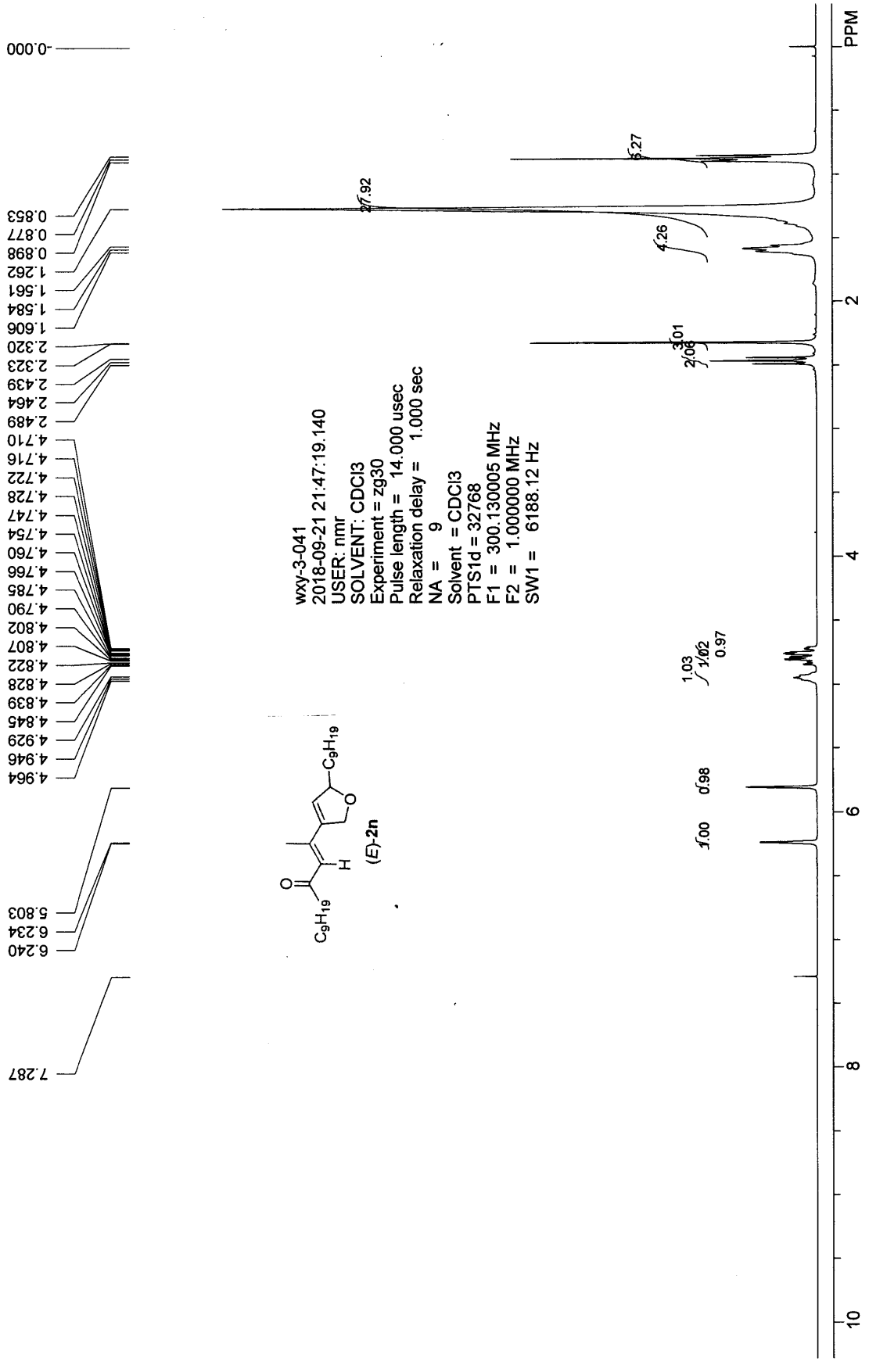
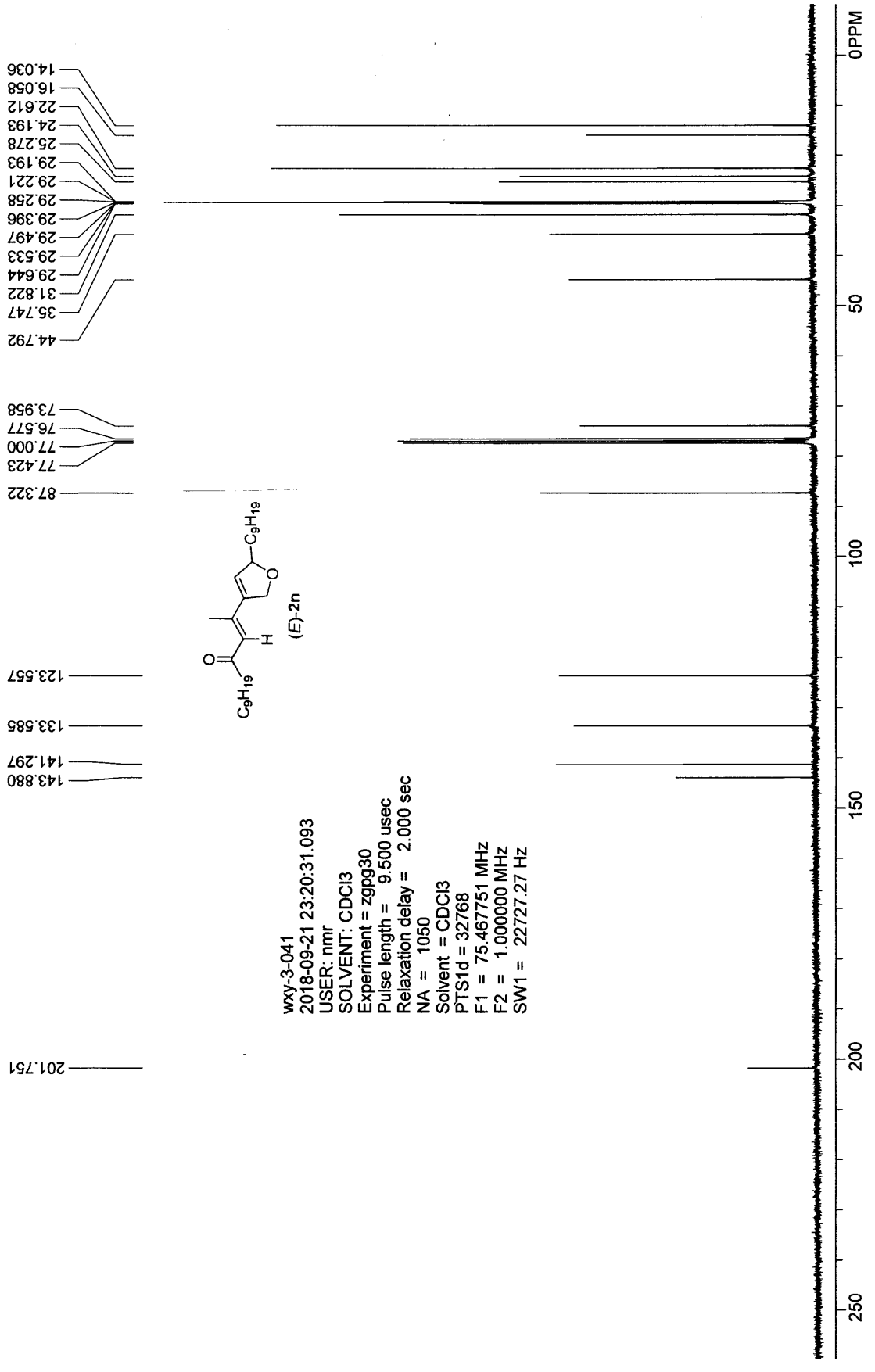
22.511
22.428
16.012
13.999
13.963
hx-16-8
2018-01-30 20:05:41.968
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 105
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
(E)-2m
250
200
150
100
50
0
PPM


wxy-3-041
2018-09-21 21:47:19.140
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 9
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
(E)-2n
PPM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25.278
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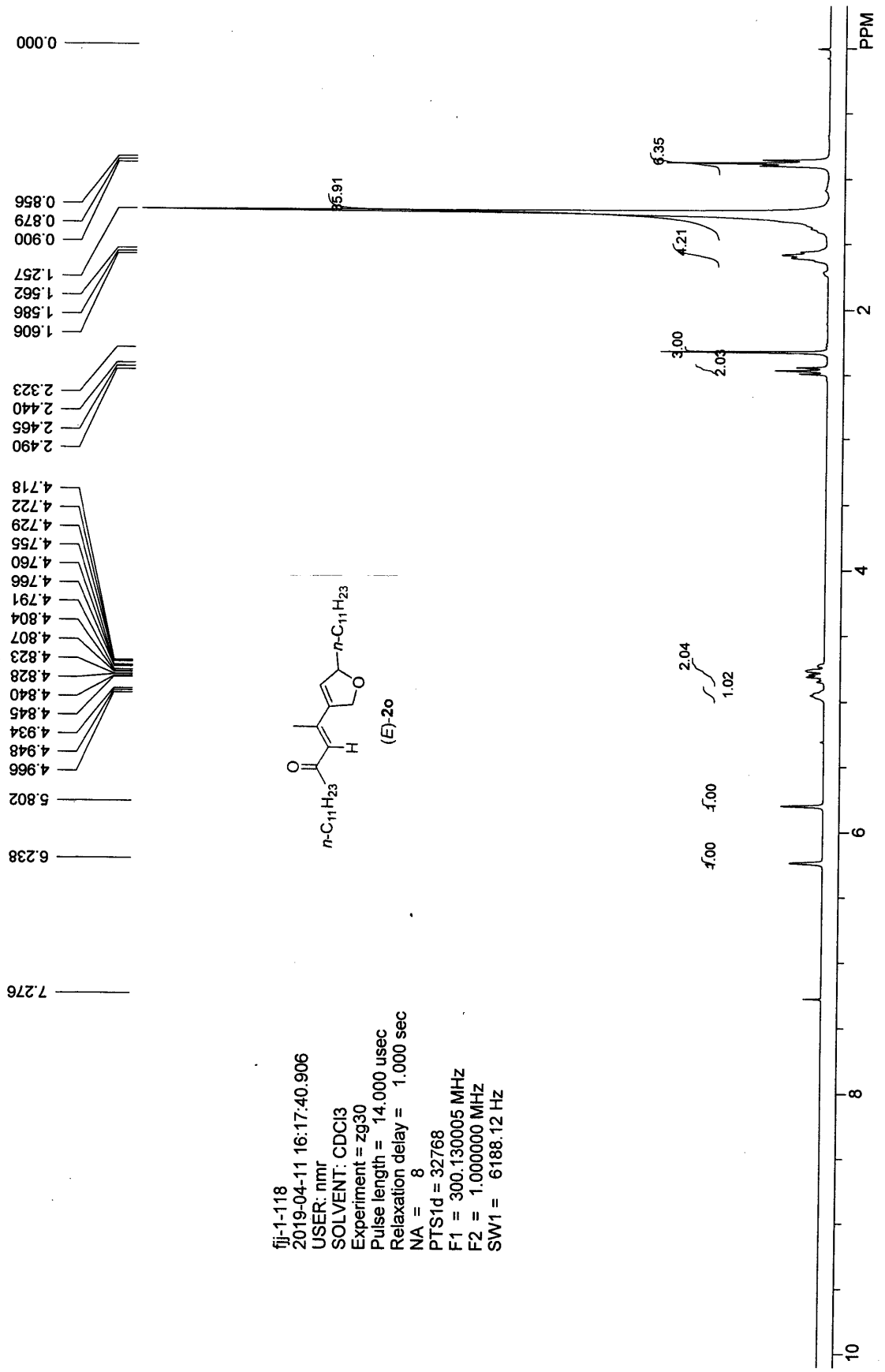
29.497
29.533
29.644
31.822
35.747
44.792
73.958
76.577
77.000
77.423
87.322
123.557
133.585
141.297
143.880
201.751
0PPM
50
100
150
200
250
wxy-3-041
2018-09-21 23:20:31.093
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 1050
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
(E)-2n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2019-04-11 16:17:40.906
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SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
(E)-2o
PPM
0.000
0.856
0.879
0.900
1.257
1.562
1.586
1.606
2.323
2.440
2.465
2.490
4.718
4.722
4.729
4.755
4.760
4.766
4.791
4.804
4.807
4.823
4.828
4.840
4.845
4.934
4.948
4.966
5.802
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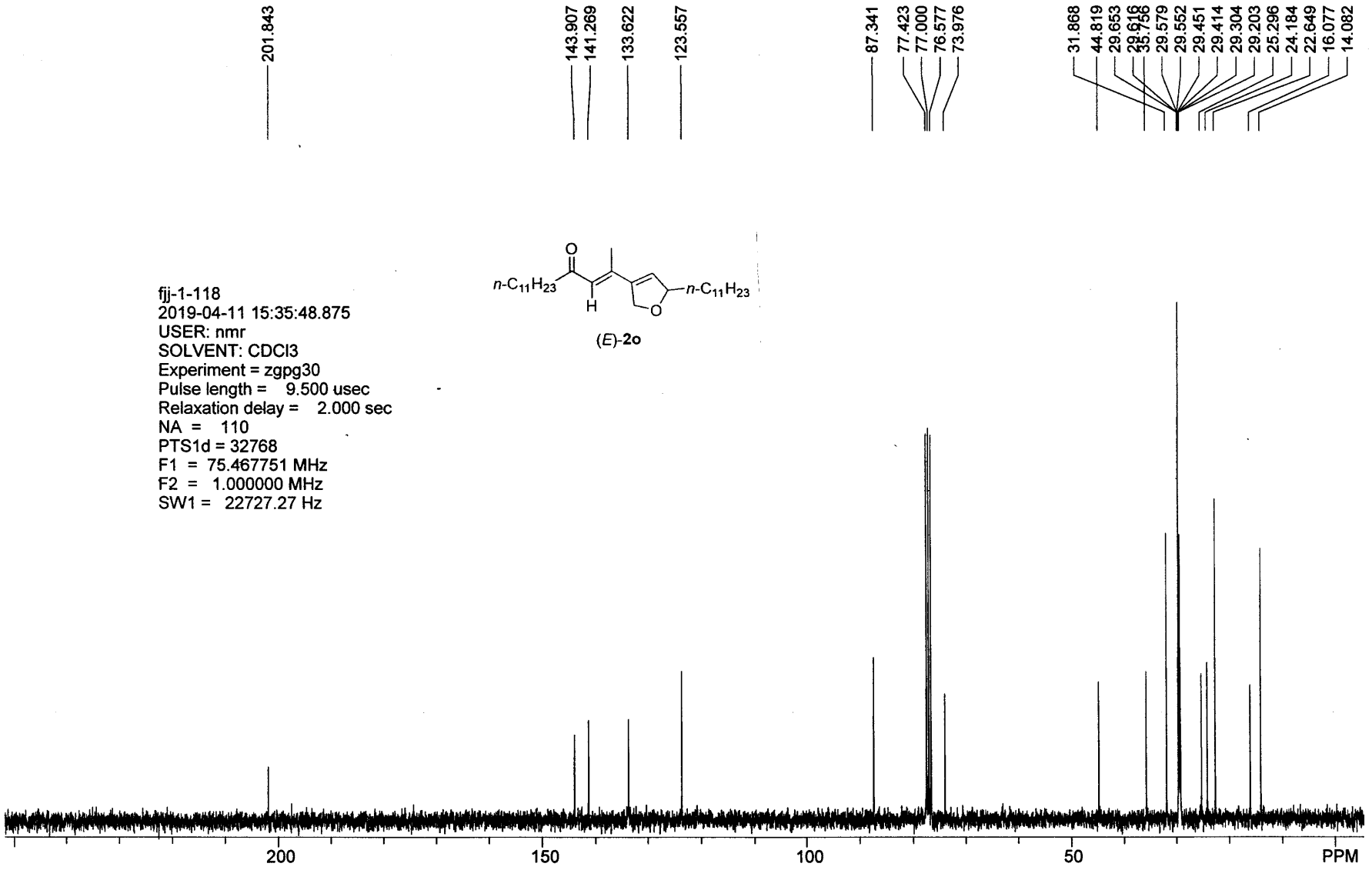
7.276
5.91
6.35
4.21
3.00
2.03
2.04
1.02
1.00
1.00
1.00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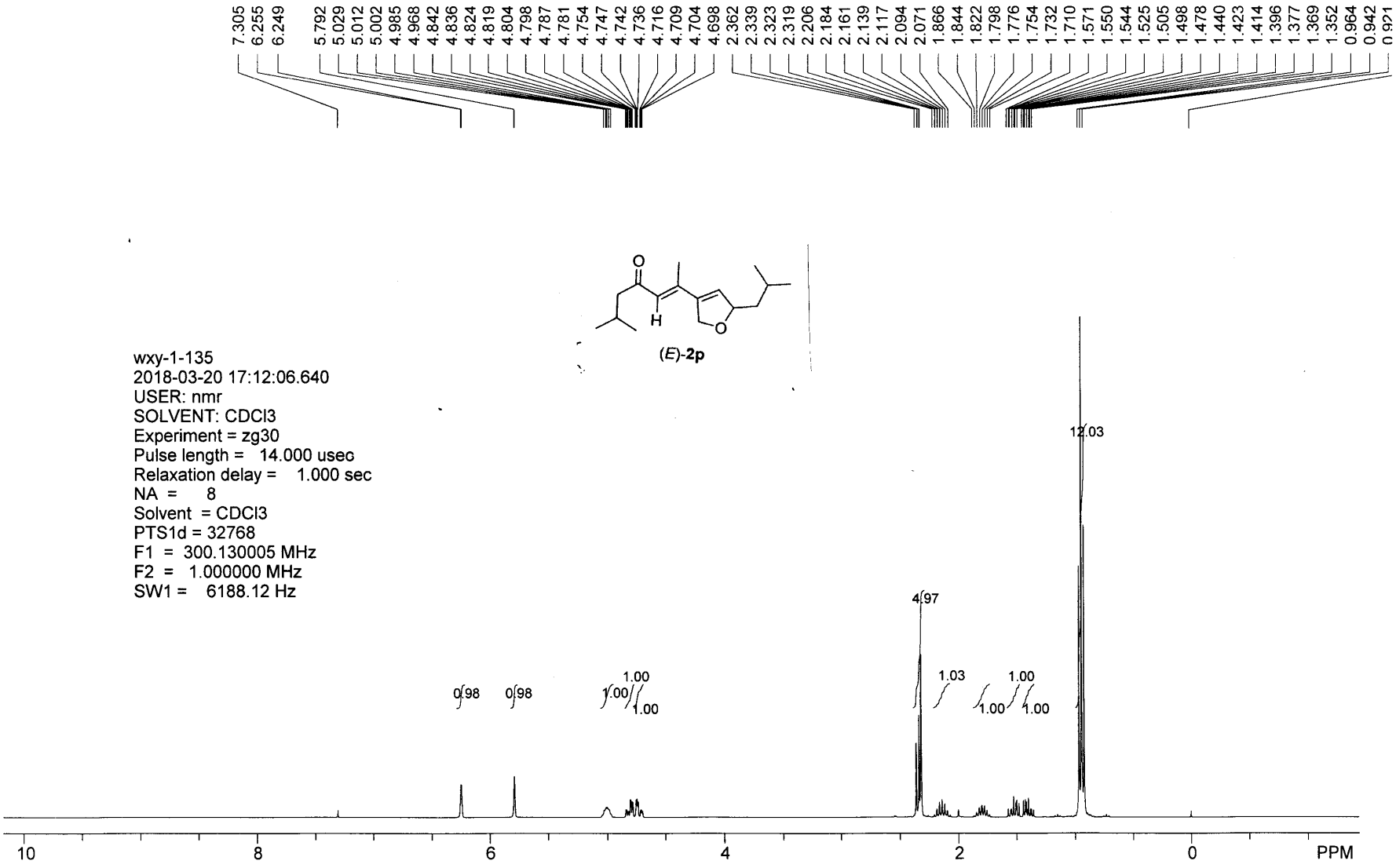
123.557
87.341
77.423
77.000
76.577
73.976
31.868
44.819
29.653
29.616
29.579
29.552
29.451
29.414
29.304
29.203
25.296
24.184
22.649
16.077
14.082
fjj-1-118
2019-04-11 15:35:48.875
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 110
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
n-C11H23
n-C11H23
(E)-2o
200
150
100
50
PPM


7.305
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4.709
4.704
4.698
2.362
2.339
2.323
2.319
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2.184
2.161
2.139
2.117
2.094
2.071
1.866
1.844
1.822
1.798
1.776
1.754
1.732
1.710
1.571
1.550
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1.525
1.505
1.498
1.478
1.440
1.423
1.414
1.396
1.377
1.369
1.352
0.964
0.942
0.921
O
H
O
(E)-2p
wxy-1-135
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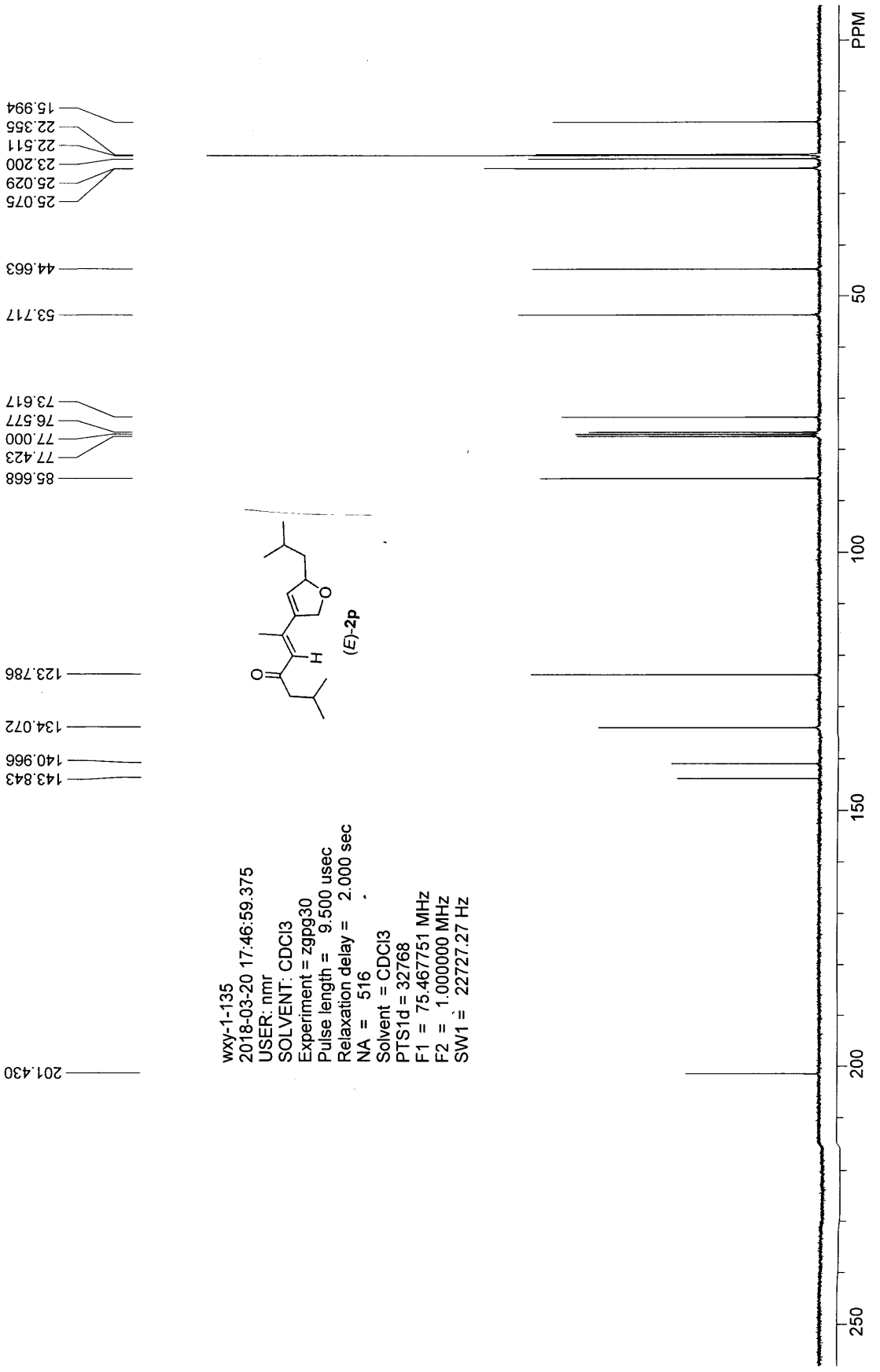
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
0.98
0.98
1.00
1.00
1.00
4.97
1.03
1.00
1.00
1.00
12.03
10
8
6
4
2
0
PPM


wxy-1-135
2018-03-20 17:46:59.375
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 516
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
(E)-2p
15.994
22.355
22.511
23.200
25.029
25.075
44.663
53.717
73.617
76.577
77.000
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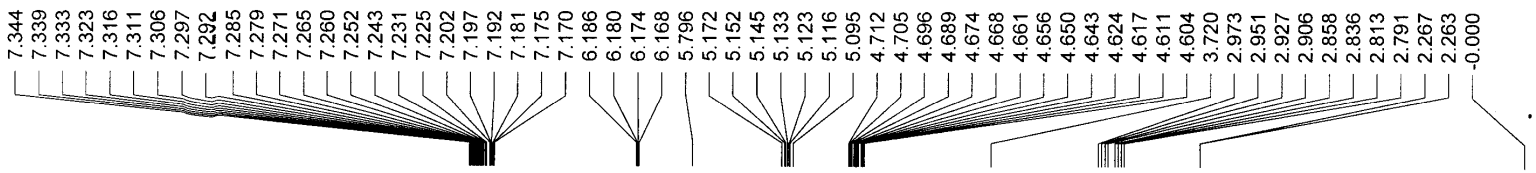
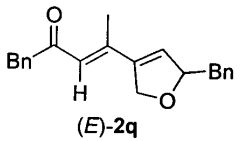
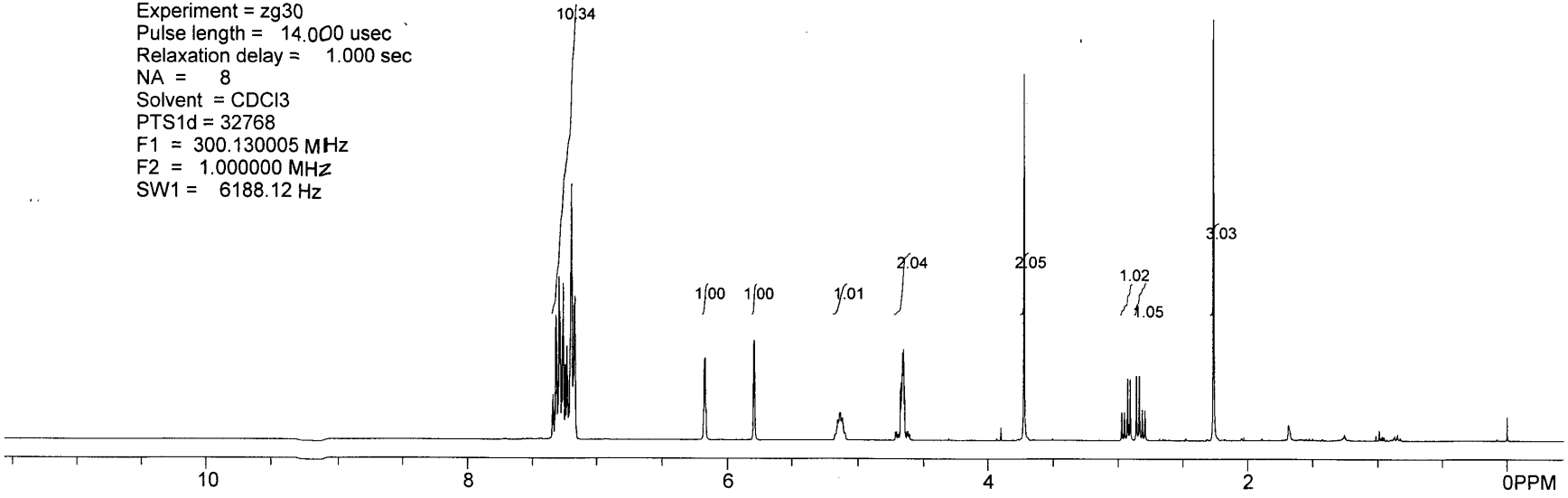
143.843
201.430
PPM
250
200
150
100
50

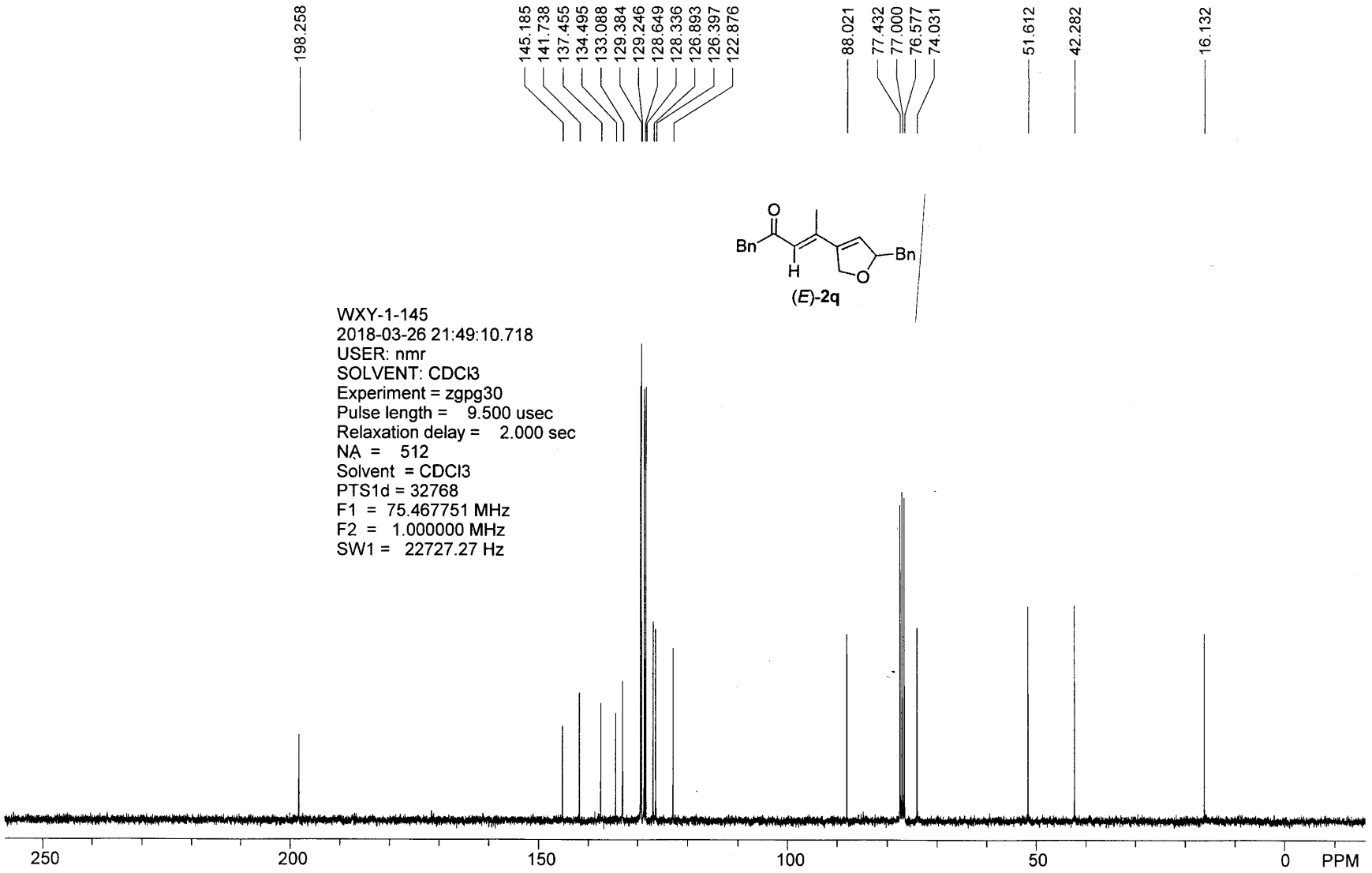




(*E*)-**2q**

wxy-1-145
2018-03-26 21:17:42.109
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz




198.258
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137.455
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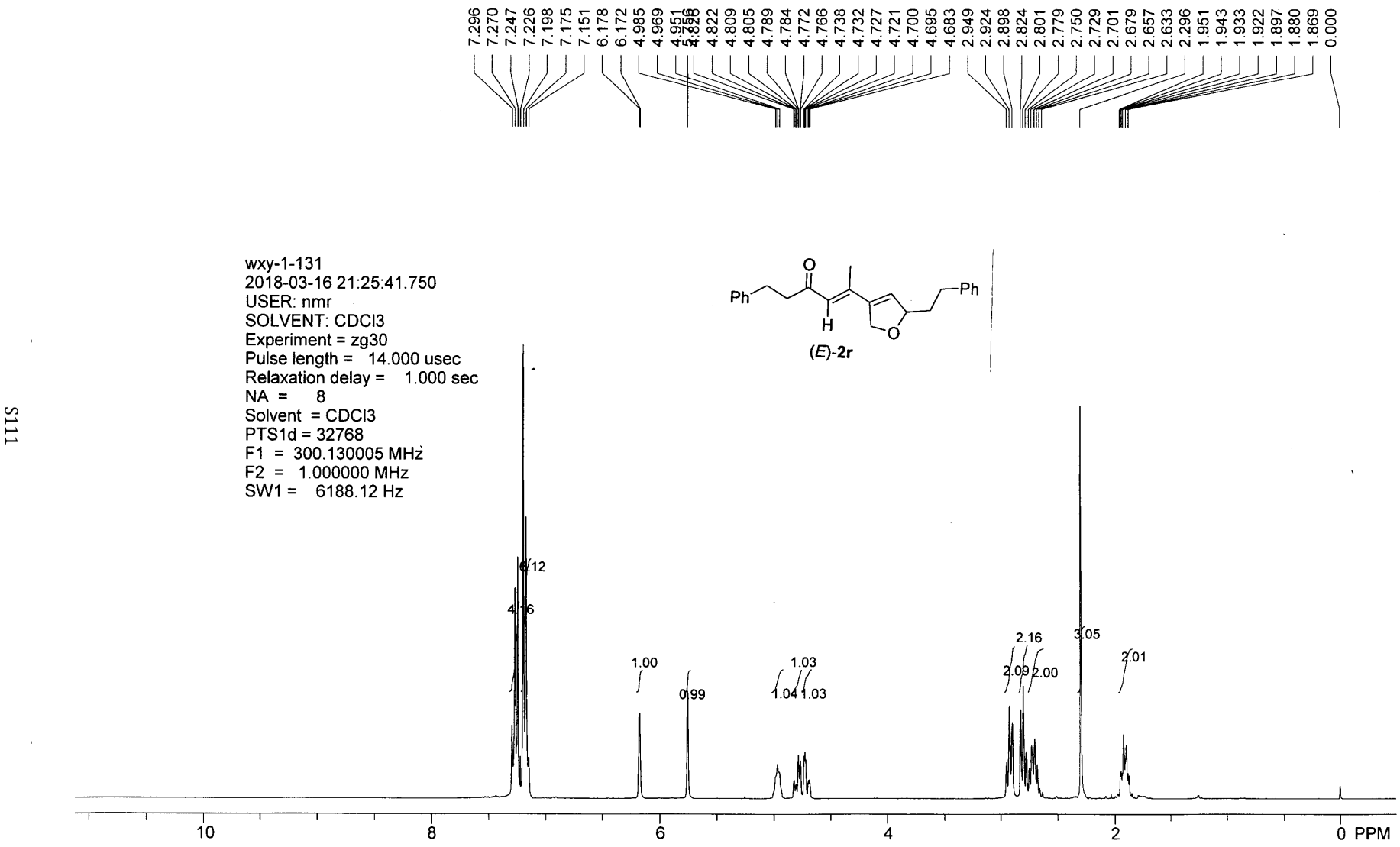
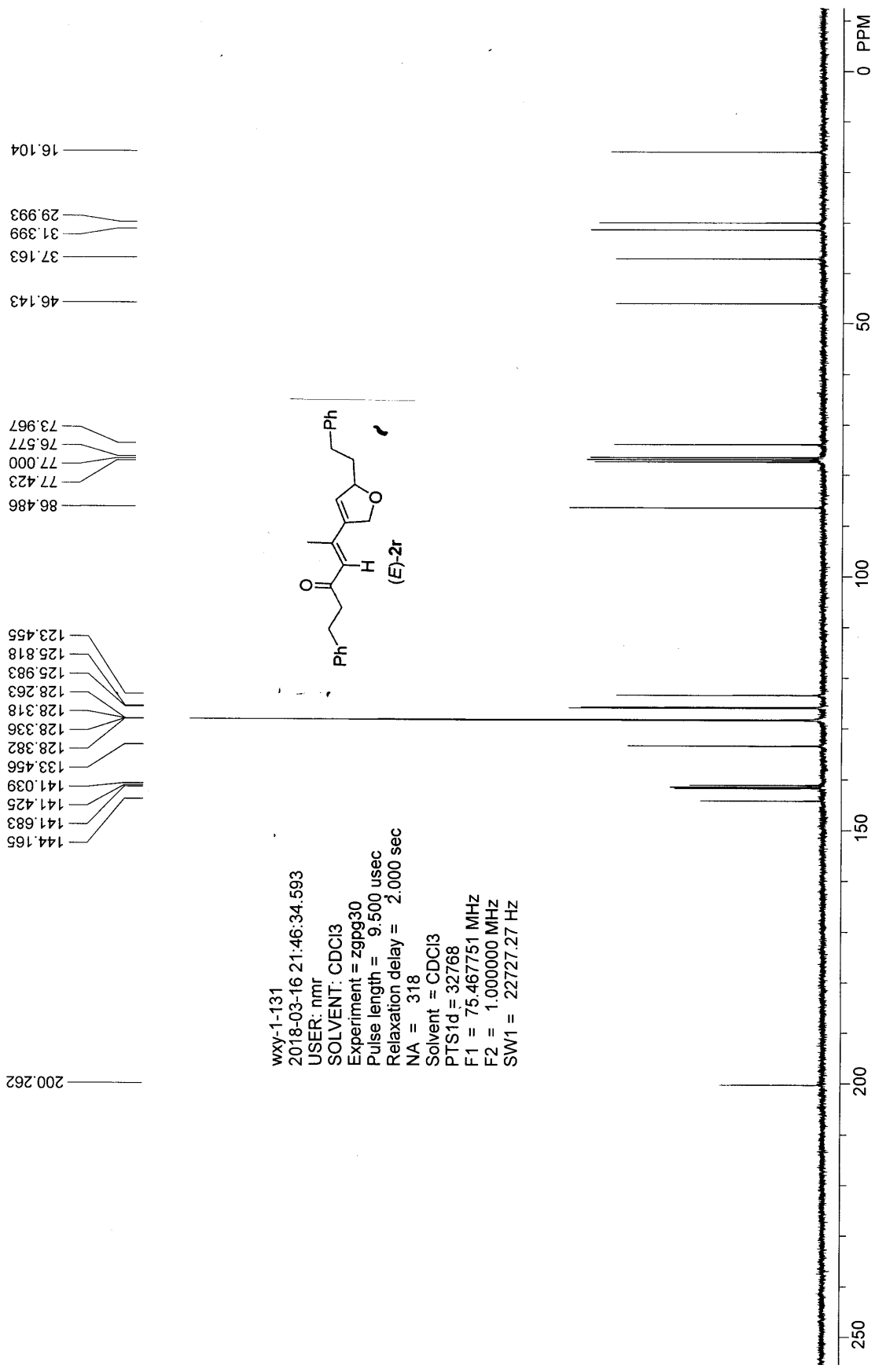
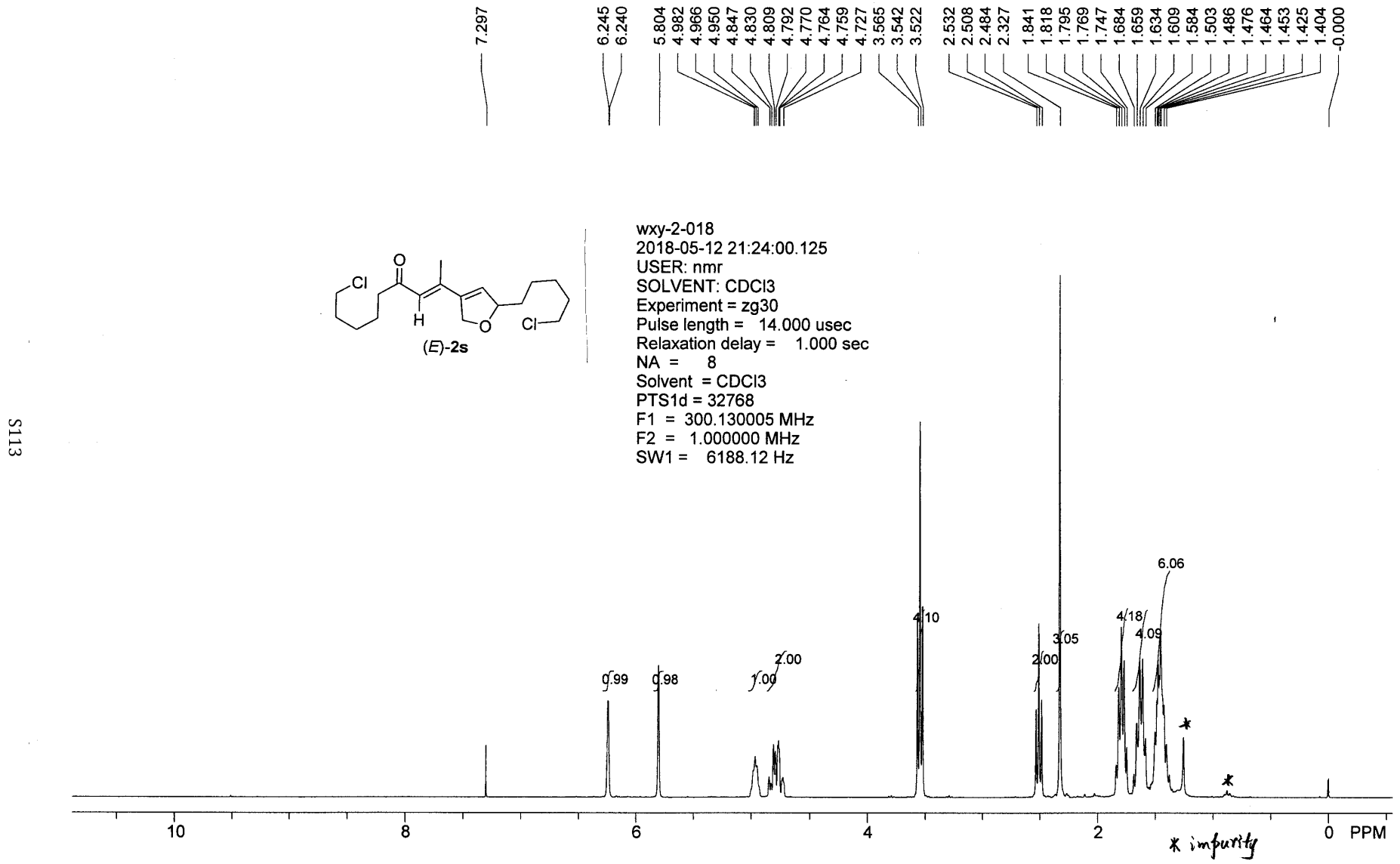
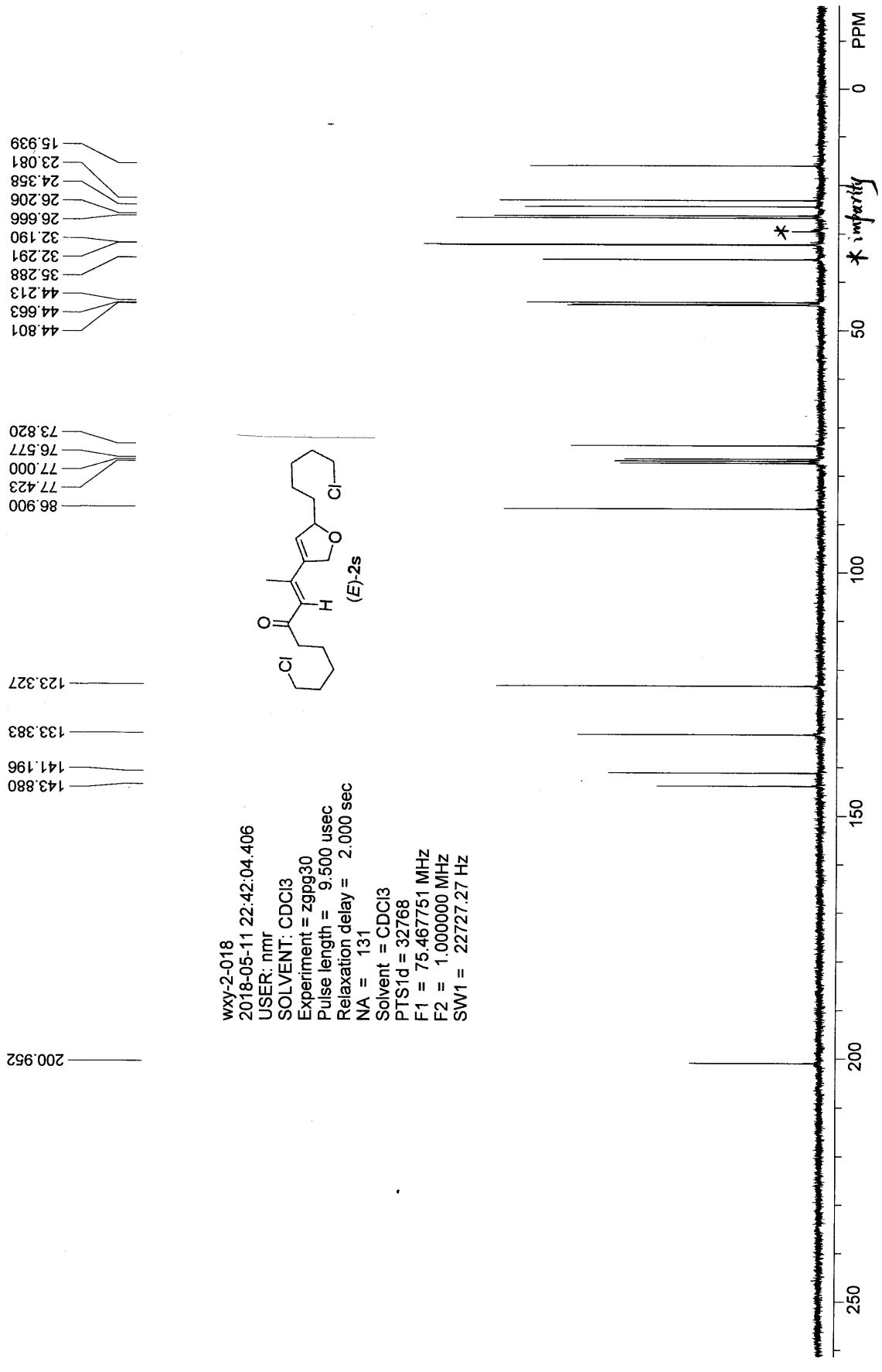
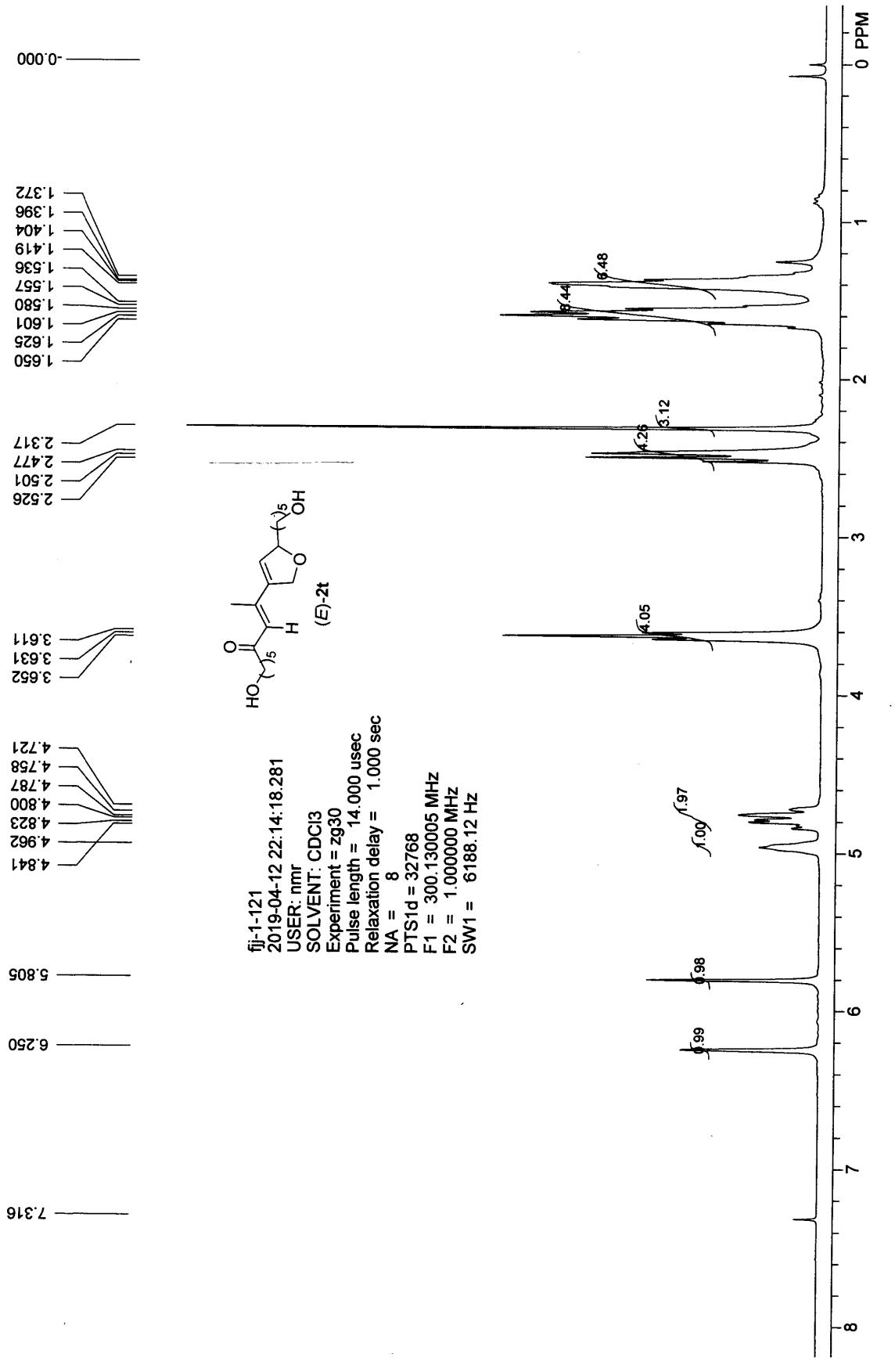
129.246
128.649
128.336
126.893
126.397
122.876
88.021
77.432
77.000
76.577
74.031
51.612
42.282
16.132
O
Bn
H
Bn
O
(E)-2q
WXY-1-145
2018-03-26 21:49:10.718
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 512
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
250
200
150
100
50
0
PPM

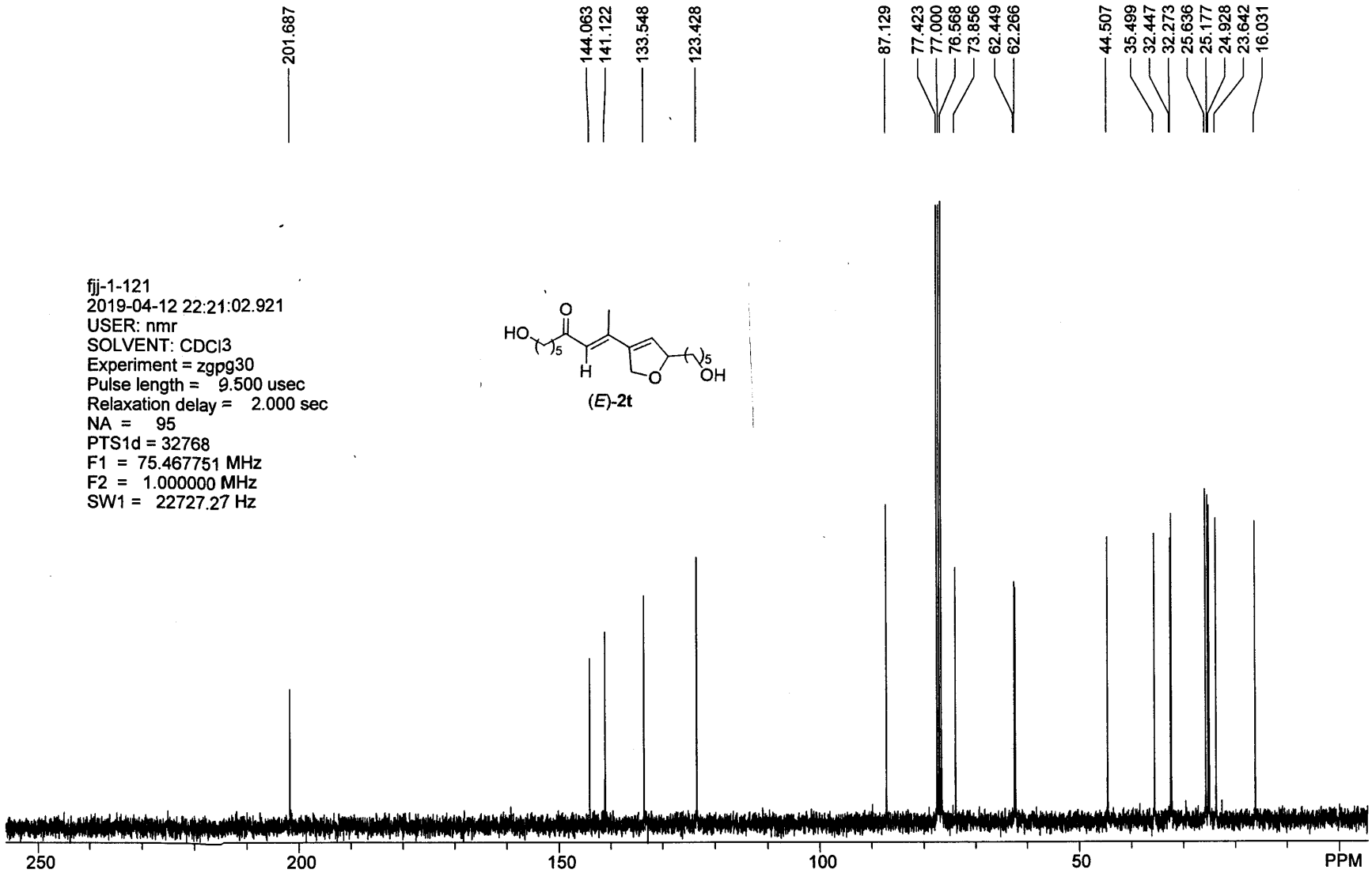

7.296
7.270
7.247
7.226
7.198
7.175
7.151
6.178
6.172
4.985
4.969
4.951
4.822
4.809
4.805
4.789
4.784
4.772
4.766
4.738
4.732
4.727
4.721
4.700
4.695
4.683
2.949
2.924
2.898
2.824
2.801
2.779
2.750
2.729
2.701
2.679
2.657
2.633
2.296
1.951
1.943
1.933
1.922
1.897
1.880
1.869
0.000
wxy-1-131
2018-03-16 21:25:41.750
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
O
Ph
Ph
H
O
(E)-2r
1.00
0.99
1.03
1.04
1.03
2.16
2.09
2.00
3.05
2.01
10
8
6
4
2
0 PPM

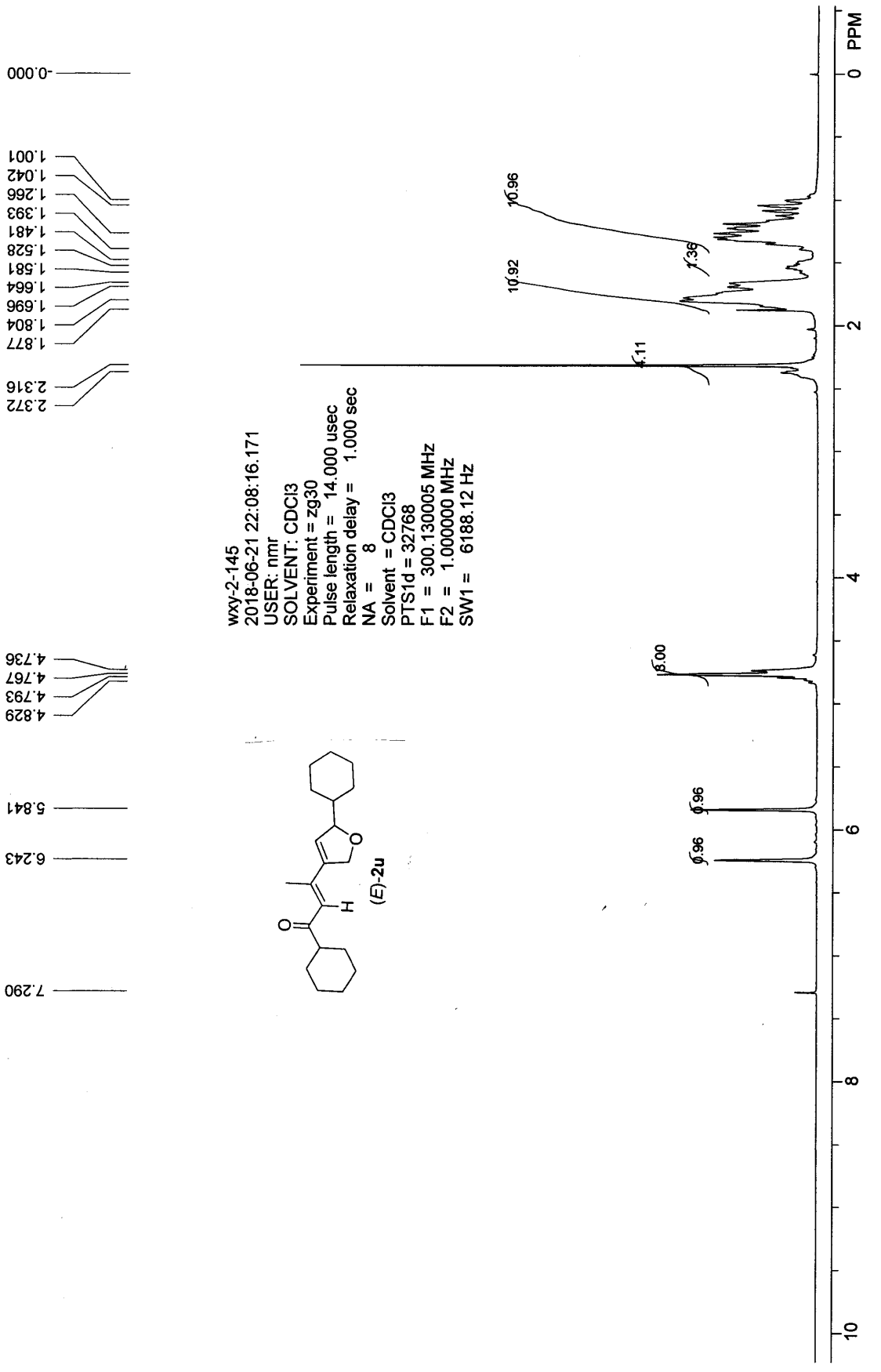

wxy-1-131
2018-03-16 21:46:34.593
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 318
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
(E)-2r
Ph
Ph
O
O
H
200.262
144.165
141.683
141.425
141.039
133.456
128.382
128.336
128.318
128.263
125.983
125.818
123.455
86.486
77.423
77.000
76.577
73.967
46.143
37.163
31.399
29.993
16.104
250
200
150
100
50
0 PPM

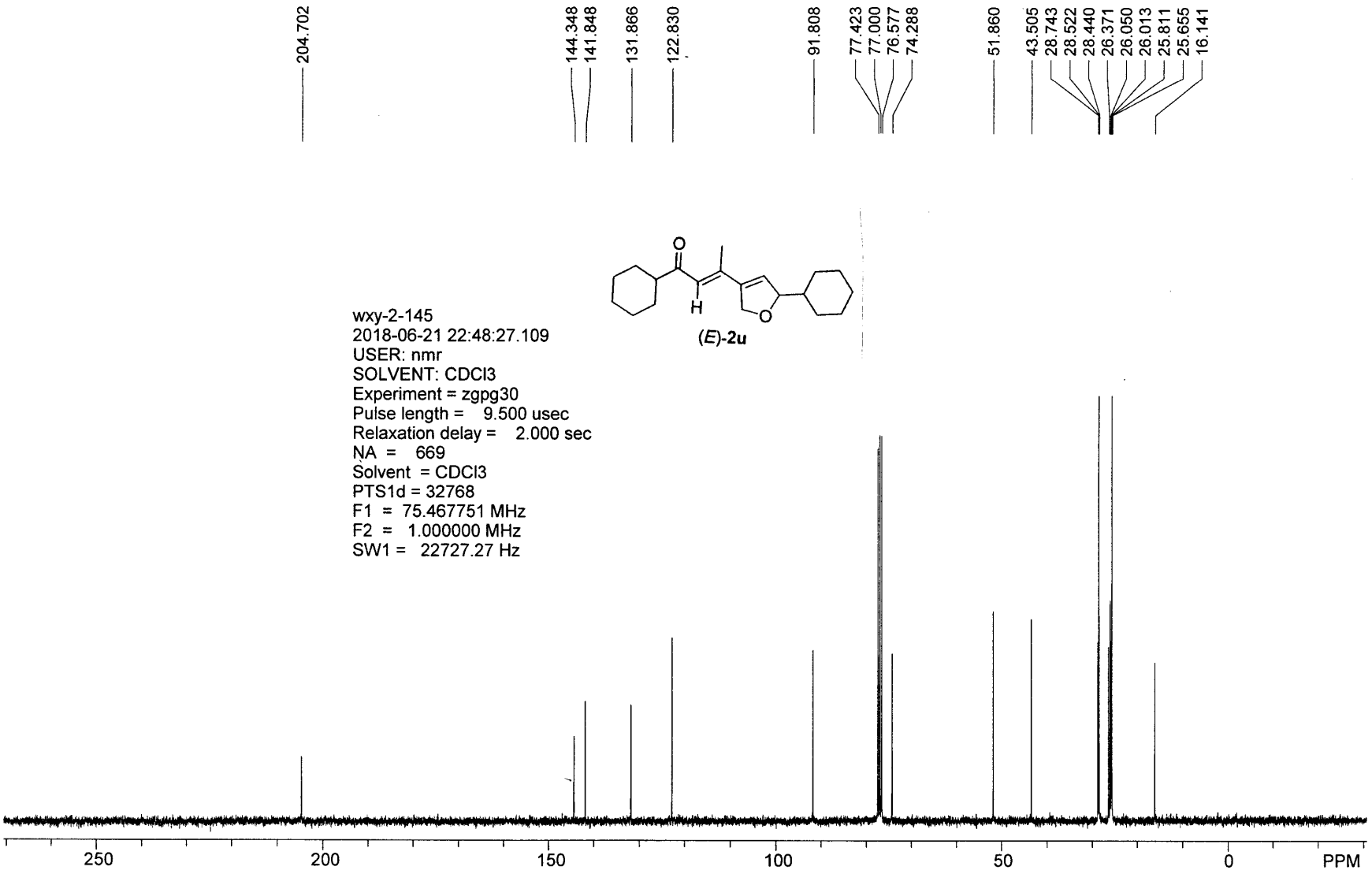

7.297
6.245
6.240
5.804
4.982
4.966
4.950
4.847
4.830
4.809
4.792
4.770
4.764
4.759
4.727
3.565
3.542
3.522
2.532
2.508
2.484
2.327
1.841
1.818
1.795
1.769
1.747
1.684
1.659
1.634
1.609
1.584
1.503
1.486
1.476
1.464
1.453
1.425
1.404
-0.000
Cl
O
H
O
Cl
(E)-2s
wxy-2-018
2018-05-12 21:24:00.125
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
0.99
0.98
2.00
1.00
4.10
2.00
3.05
4.18
4.09
6.06
10
8
6
4
2
0
PPM
* impurity

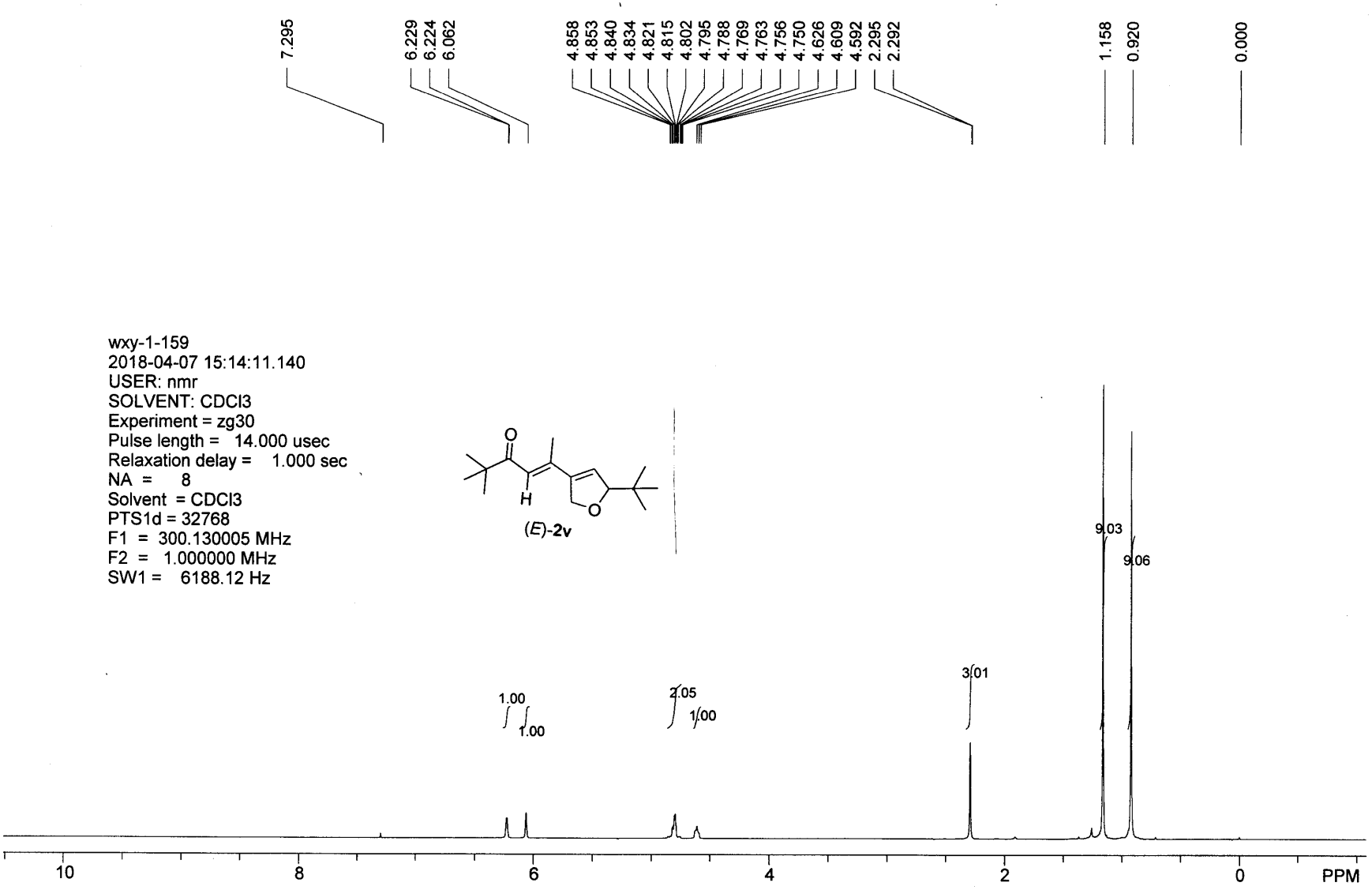

wxy-2-018
2018-05-11 22:42:04.406
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 131
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
(E)-2s
Cl
O
H
O
Cl
200.952
143.880
141.196
133.383
123.327
86.900
77.423
77.000
76.577
73.820
44.801
44.663
44.213
35.288
32.291
32.190
26.666
26.206
24.358
23.081
15.939
* impurity
PPM
250
200
150
100
50
0

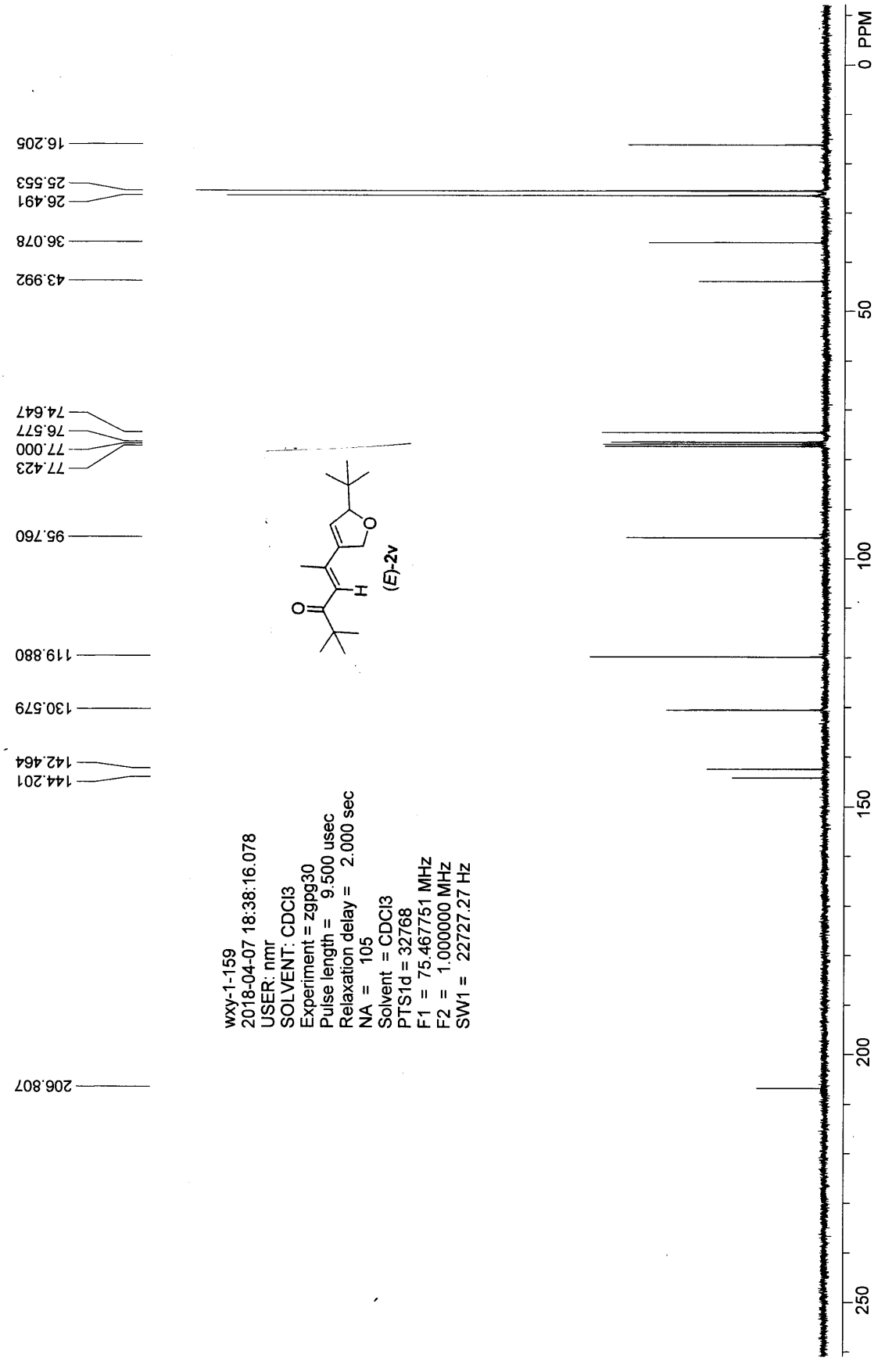

fjj-1-121
2019-04-12 22:14:18.281
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
(E)-2t
7.316
6.250
5.805
4.841
4.962
4.823
4.800
4.787
4.758
4.721
3.652
3.631
3.611
2.526
2.501
2.477
2.317
1.650
1.625
1.601
1.580
1.557
1.536
1.419
1.404
1.396
1.372
-0.000
0.99
0.98
1.00
1.97
4.05
4.26
3.12
6.44
6.48
0 PPM

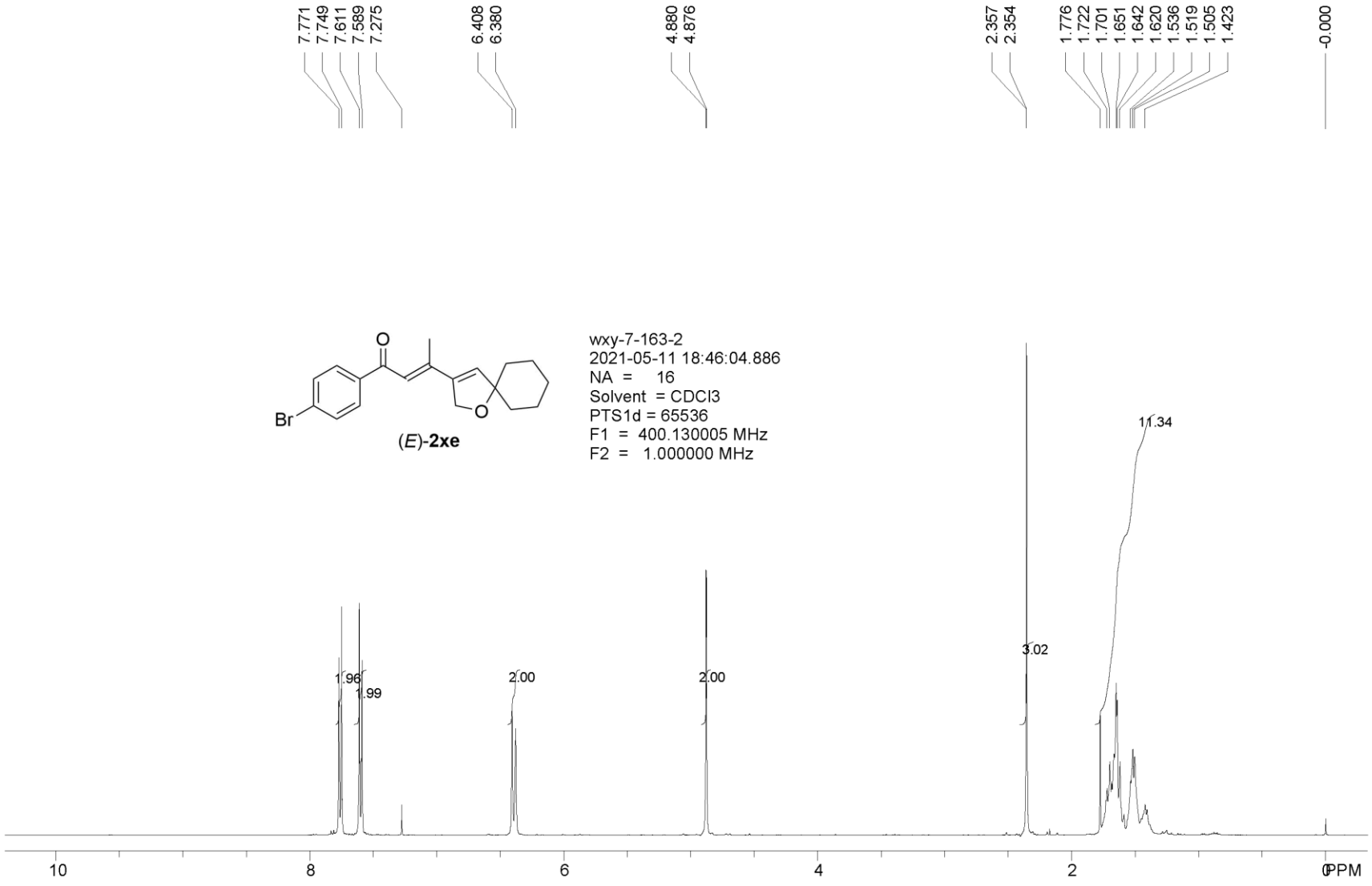

201.687
144.063
141.122
133.548
123.428
87.129
77.423
77.000
76.568
73.856
62.449
62.266
44.507
35.499
32.447
32.273
25.636
25.177
24.928
23.642
16.031
fjj-1-121
2019-04-12 22:21:02.921
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 95
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
HO
O
H
O
OH
(E)-2t
250
200
150
100
50
PPM

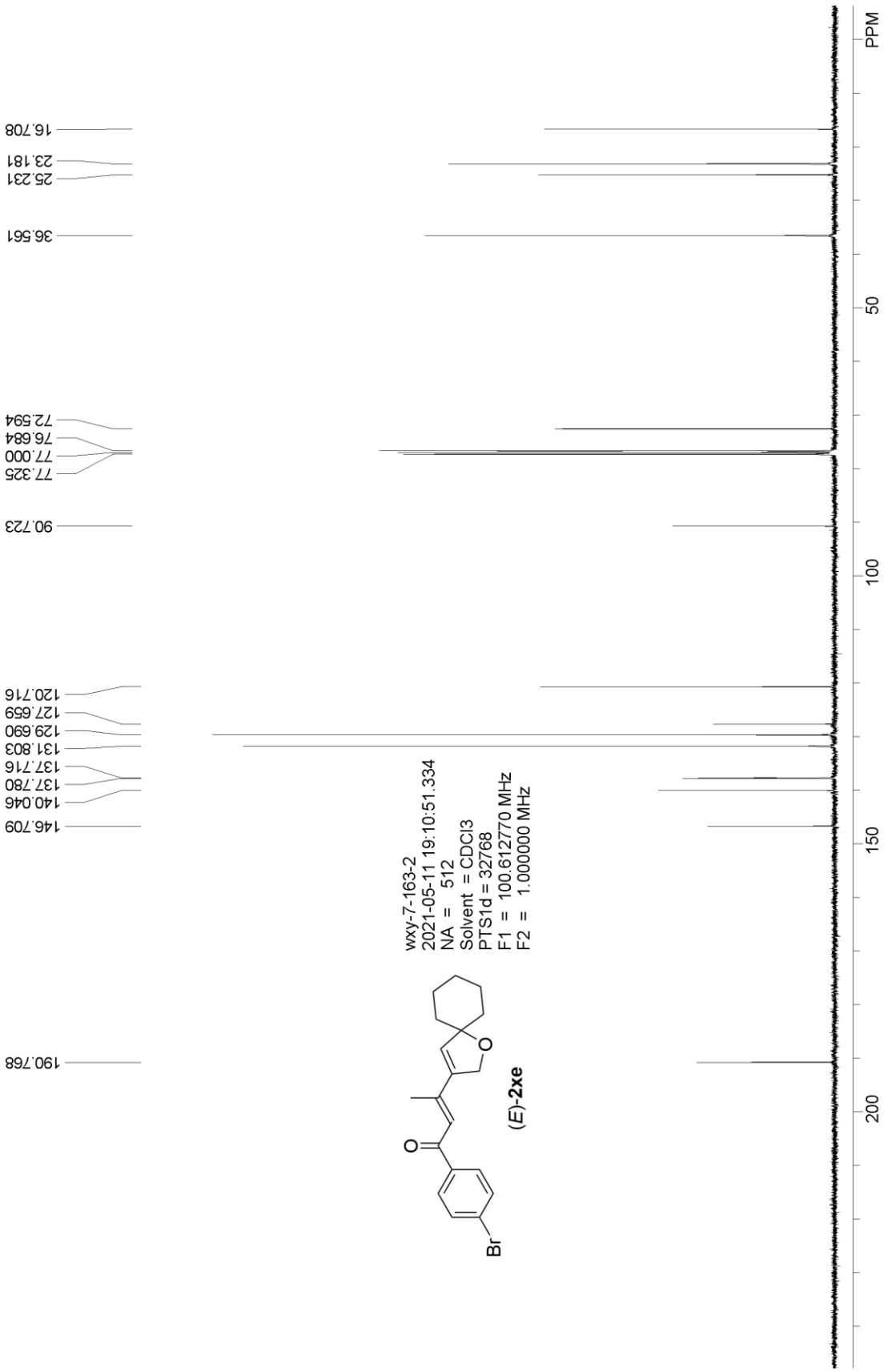

wxy-2-145
2018-06-21 22:08:16.171
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
(E)-2u
PPM
0
2
4
6
8
10
8.00
4.11
10.92
10.96
1.36
0.96
0.96
0.96
7.290
6.243
5.841
4.829
4.793
4.767
4.736
2.372
2.316
1.877
1.804
1.696
1.664
1.581
1.528
1.481
1.393
1.266
1.042
1.001
0.000

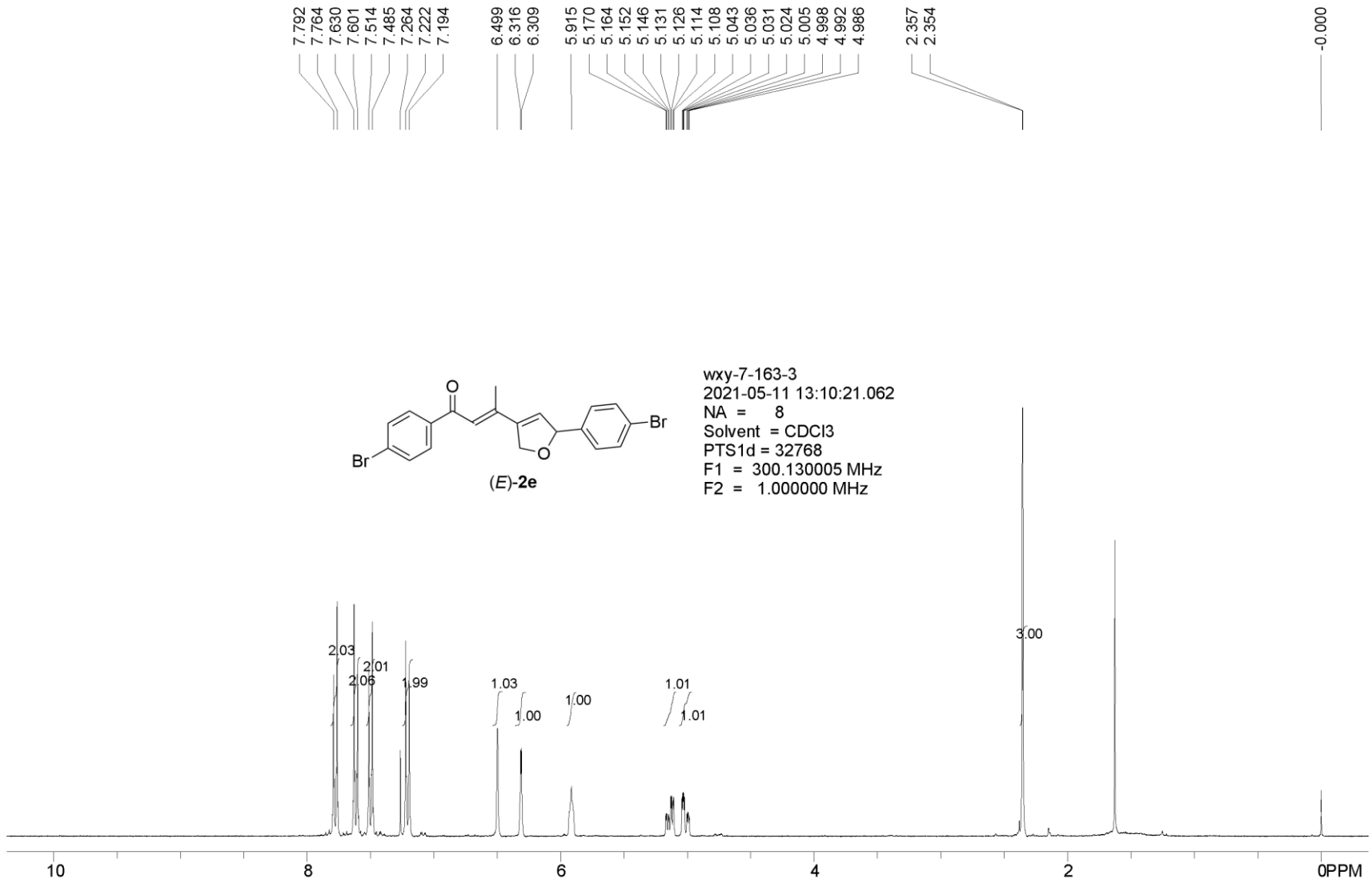

204.702
144.348
141.848
131.866
122.830
91.808
77.423
77.000
76.577
74.288
51.860
43.505
28.743
28.522
28.440
26.371
26.050
26.013
25.811
25.655
16.141
O
H
O
(E)-2u
wxy-2-145
2018-06-21 22:48:27.109
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 669
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
250
200
150
100
50
0
PPM

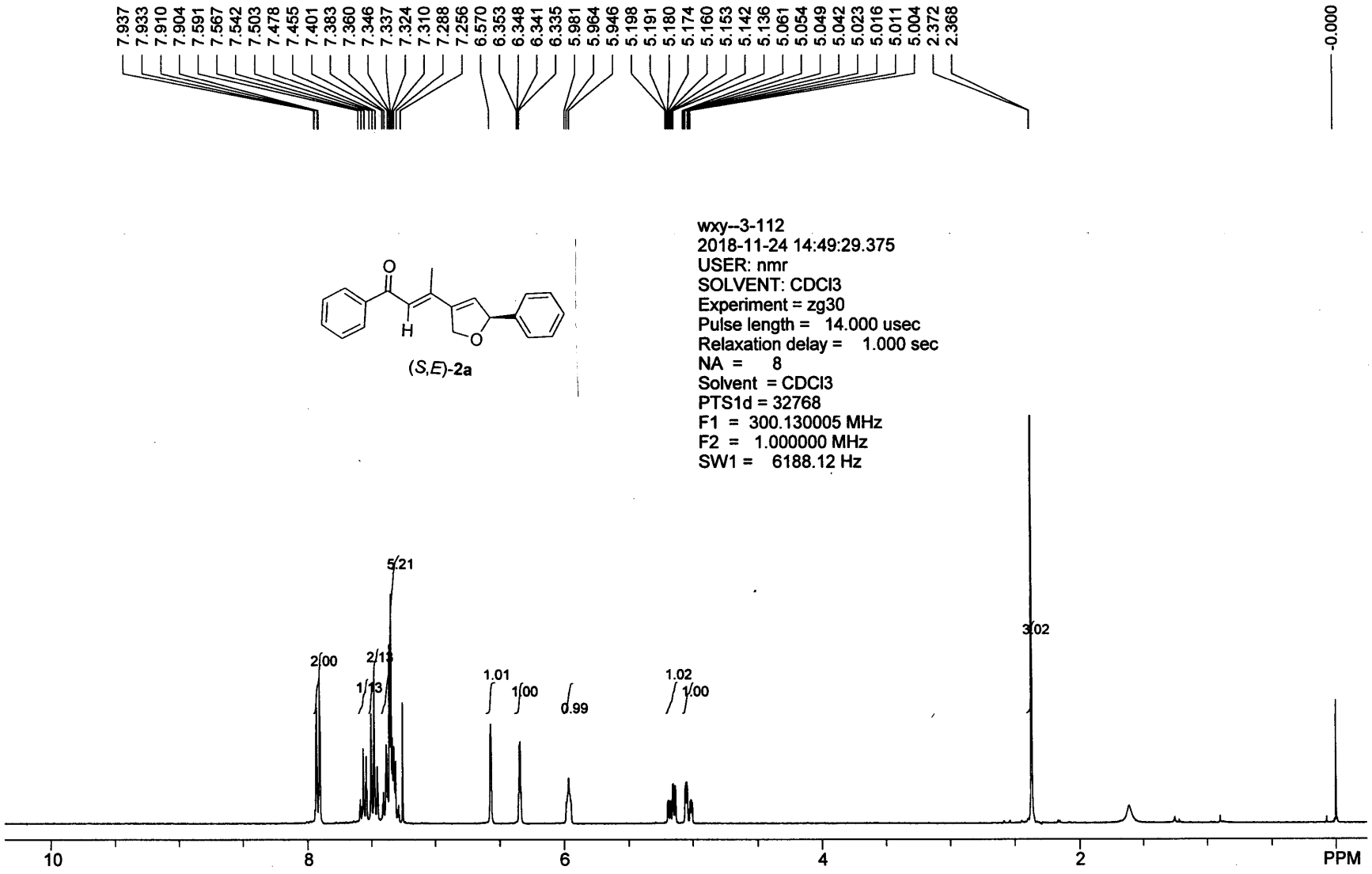

7.295
6.229
6.224
6.062
4.858
4.853
4.840
4.834
4.821
4.815
4.802
4.795
4.788
4.769
4.763
4.756
4.750
4.626
4.609
4.592
2.295
2.292
1.158
0.920
0.000
wxy-1-159
2018-04-07 15:14:11.140
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
O
H
O
(E)-2v
1.00
1.00
2.05
1.00
3.01
9.03
9.06
10
8
6
4
2
0
PPM

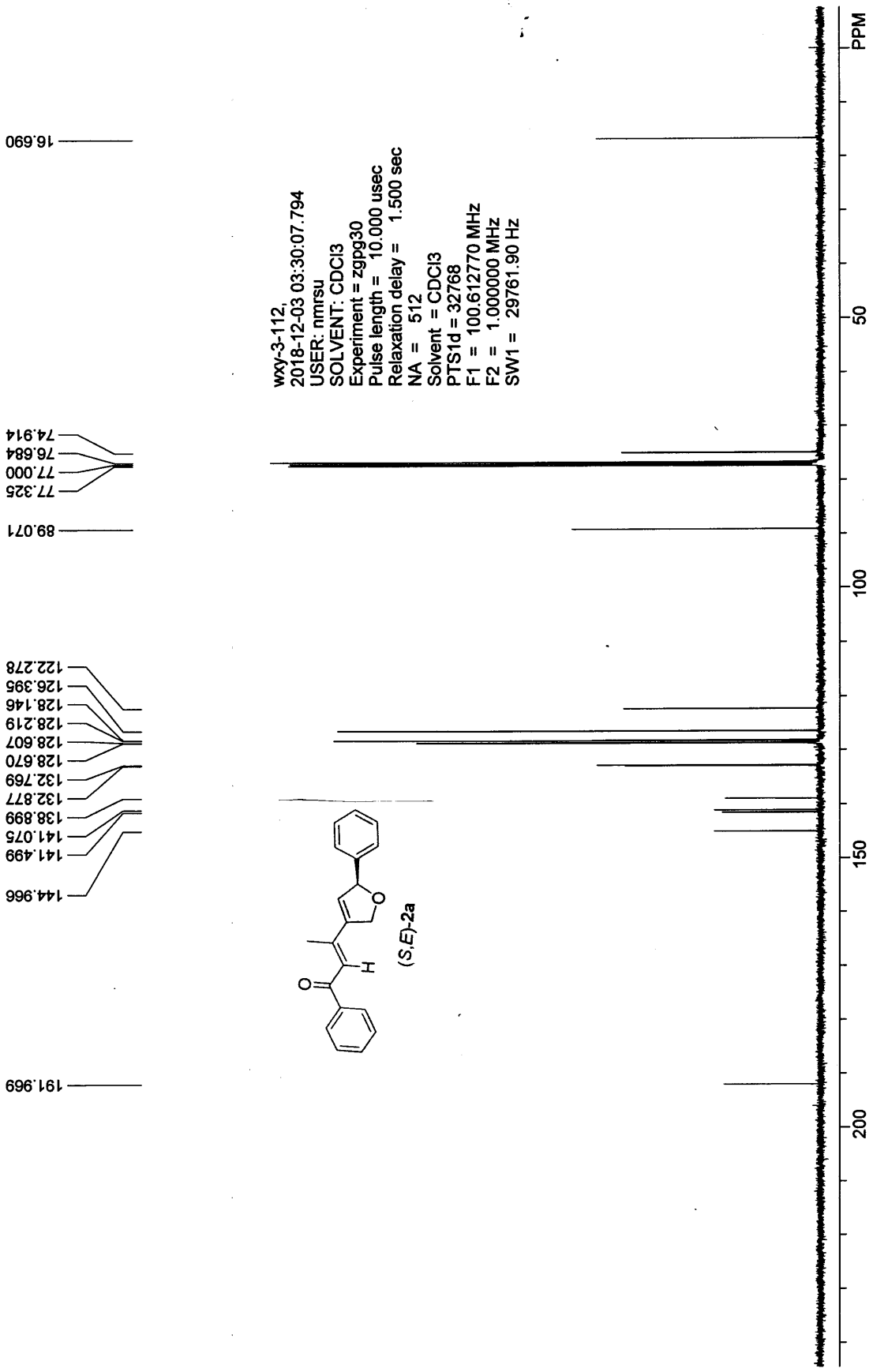

wxy-1-159
2018-04-07 18:38:16.078
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 105
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
(E)-2v
206.807
144.201
142.464
130.579
119.880
95.760
77.423
77.000
76.577
74.647
43.992
36.078
26.491
25.553
16.205
250
200
150
100
50
0 PPM


7.771
7.749
7.611
7.589
7.275
6.408
6.380
4.880
4.876
2.357
2.354
1.776
1.722
1.701
1.651
1.642
1.620
1.536
1.519
1.505
1.423
-0.000
O
Br
O
(E)-2xe
wxy-7-163-2
2021-05-11 18:46:04.886
NA = 16
Solvent = CDCl3
PTS1d = 65536
F1 = 400.130005 MHz
F2 = 1.000000 MHz
1.96
1.99
2.00
2.00
3.02
11.34
10
8
6
4
2
0
PPM


16.708
23.181
25.231
36.561
72.594
76.684
77.000
77.325
90.723
120.716
127.659
129.690
131.803
137.716
137.780
140.046
146.709
190.768
wxy-7-163-2
2021-05-11 19:10:51.334
NA = 512
Solvent = CDCl3
PTS1d = 32768
F1 = 100.612770 MHz
F2 = 1.000000 MHz
(E)-2xe
O
O
Br
PPM
50
100
150
200


7.792
7.764
7.630
7.601
7.514
7.485
7.264
7.222
7.194
6.499
6.316
6.309
5.915
5.170
5.164
5.152
5.146
5.131
5.126
5.114
5.108
5.043
5.036
5.031
5.024
5.005
4.998
4.992
4.986
2.357
2.354
-0.000
O
Br
O
Br
(E)-2e
wxy-7-163-3
2021-05-11 13:10:21.062
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
2.03
2.06
2.01
1.99
1.03
1.00
1.00
1.01
1.01
3.00
10
8
6
4
2
0PPM


7.937
7.933
7.910
7.904
7.591
7.567
7.542
7.503
7.478
7.455
7.401
7.383
7.360
7.346
7.337
7.324
7.310
7.288
7.256
6.570
6.353
6.348
6.341
6.335
5.981
5.964
5.946
5.198
5.191
5.180
5.174
5.160
5.153
5.142
5.136
5.061
5.054
5.049
5.042
5.023
5.016
5.011
5.004
2.372
2.368
-0.000
O
H
O
(S,E)-2a
wxy--3-112
2018-11-24 14:49:29.375
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
5.21
2.00
2.13
1.13
1.01
1.00
0.99
1.02
1.00
3.02
10
8
6
4
2
PPM


wxy-3-112,
2018-12-03 03:30:07.794
USER: nmrsu
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 10.000 usec
Relaxation delay = 1.500 sec
NA = 512
Solvent = CDCl3
PTS1d = 32768
F1 = 100.612770 MHz
F2 = 1.000000 MHz
SW1 = 29761.90 Hz
PPM
50
100
150
200
16.690
74.914
76.684
77.000
77.325
89.071
122.278
126.395
128.146
128.219
128.607
128.670
132.769
132.877
138.899
141.075
141.499
144.966
191.969
O
H
O
(S,E)-2a

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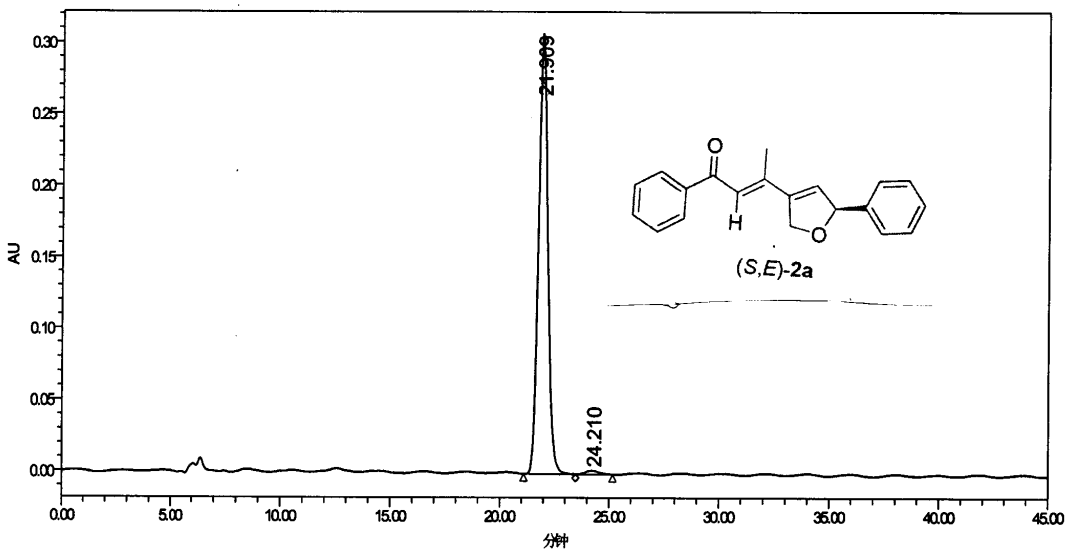
Project Name: defaults for copy

Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	wxy-3-112-io-80-10-1-214	Acquired By:	Breeze
Sample Type:	未知	Date Acquired:	2018/12/7 9:48:07 CST
Vial:	999	Acq. Method:	zgj90
Injection #:	77	Date Processed:	2018/12/7 18:05:33 CST
Injection Volume:	10.00 ul	Channel Name:	W2489 ChA
Run Time:	45.00 Minutes	Channel Desc.:	W2489 ChA 210nm
Column Type:		Sample Set Name:	



	RT (min)	Area (微sec)	%Area	Height (微)	% Height
1	21.909	9180817	98.72	308271	99.12
2	24.210	118640	1.28	2743	0.88

Report Method: Individual Report ASC

Page: 1 (共计 1)

Printed: 2018/12/7 18:09:00 PRC

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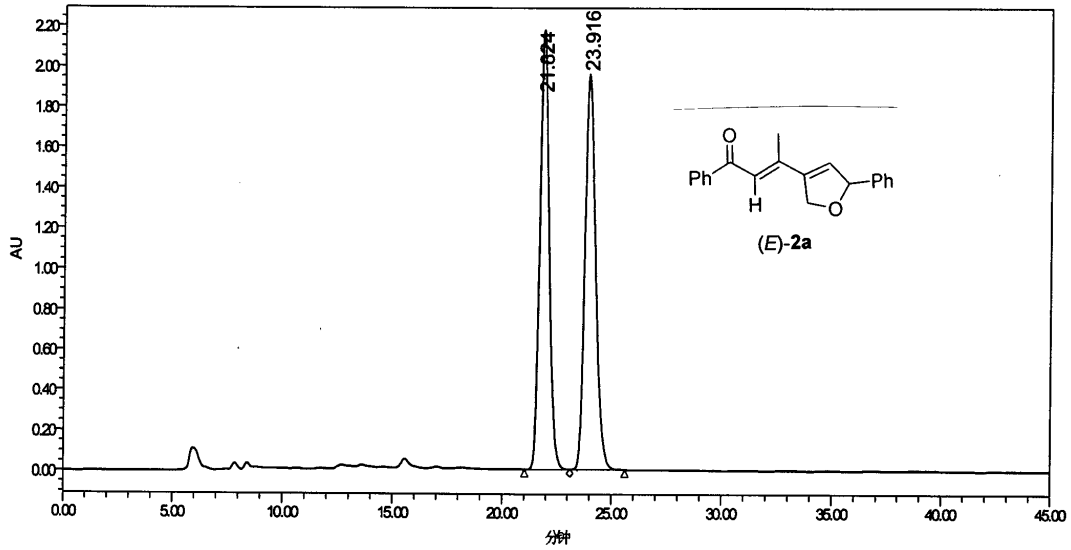
Project Name: defaults for copy

Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	hx-15-188-io-90-10-1-214	Acquired By:	Breeze
Sample Type:	未知	Date Acquired:	2018/12/7 8:46:28 CST
Vial:	999	Acq. Method:	zgj90
Injection #:	76	Date Processed:	2018/12/7 18:05:08 CST
Injection Volume:	10.00 ul	Channel Name:	W2489 ChA
Run Time:	45.00 Minutes	Channel Desc.:	W2489 ChA 210nm
Column Type:		Sample Set Name:	



	RT (min)	Area (碘sec)	%Area	Height (碘	% Height
1	21.824	66382232	49.83	2174727	52.65
2	23.916	66838397	50.17	1955548	47.35

Report Method: Individual Report ASC

Page: 1 (共计 1)

Printed: 2018/12/7 18:08:03 PRC

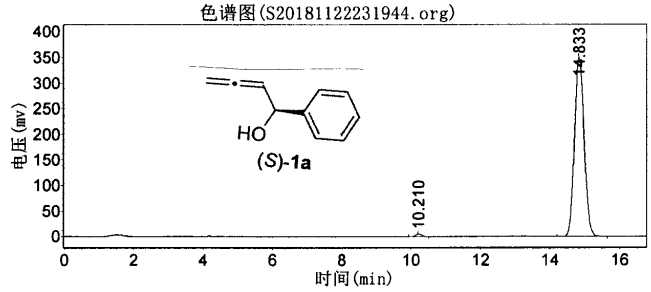
fjj-1-018-Chiral

实验时间：2018-11-22,23:19:43
谱图文件:D:\浙大智达\N2000\样品\S20181122231944.org
方法文件:D:\浙大智达\N2000\djx.mtd

实验者：fjj
报告时间：2018-11-22,23:39:13
积分方法:面积归一法

实验内容简介：
od-H,n-hexane/i-PrOH = 95/5,1.0, 220

色谱图(S20181122231944.org)



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		10.210	5387.825	65427.152	0.9580
2		14.833	349620.563	6763982.000	99.0420
总计			355008.387	6829409.152	100.0000

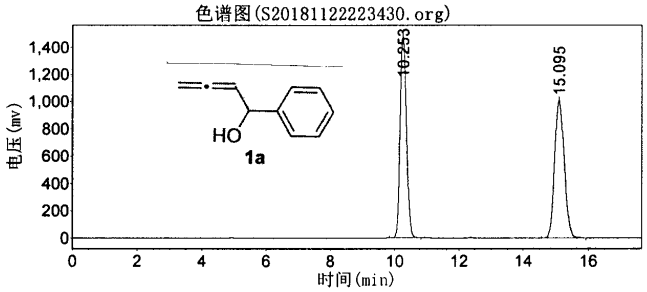
fjj-1-018-racemic

实验时间：2018-11-22,22:34:30
谱图文件:D:\浙大智达\N2000\样品\S20181122223430.org
方法文件:D:\浙大智达\N2000\djx.mtd

实验者：ssh
报告时间：2018-11-22,22:55:01
积分方法:面积归一法

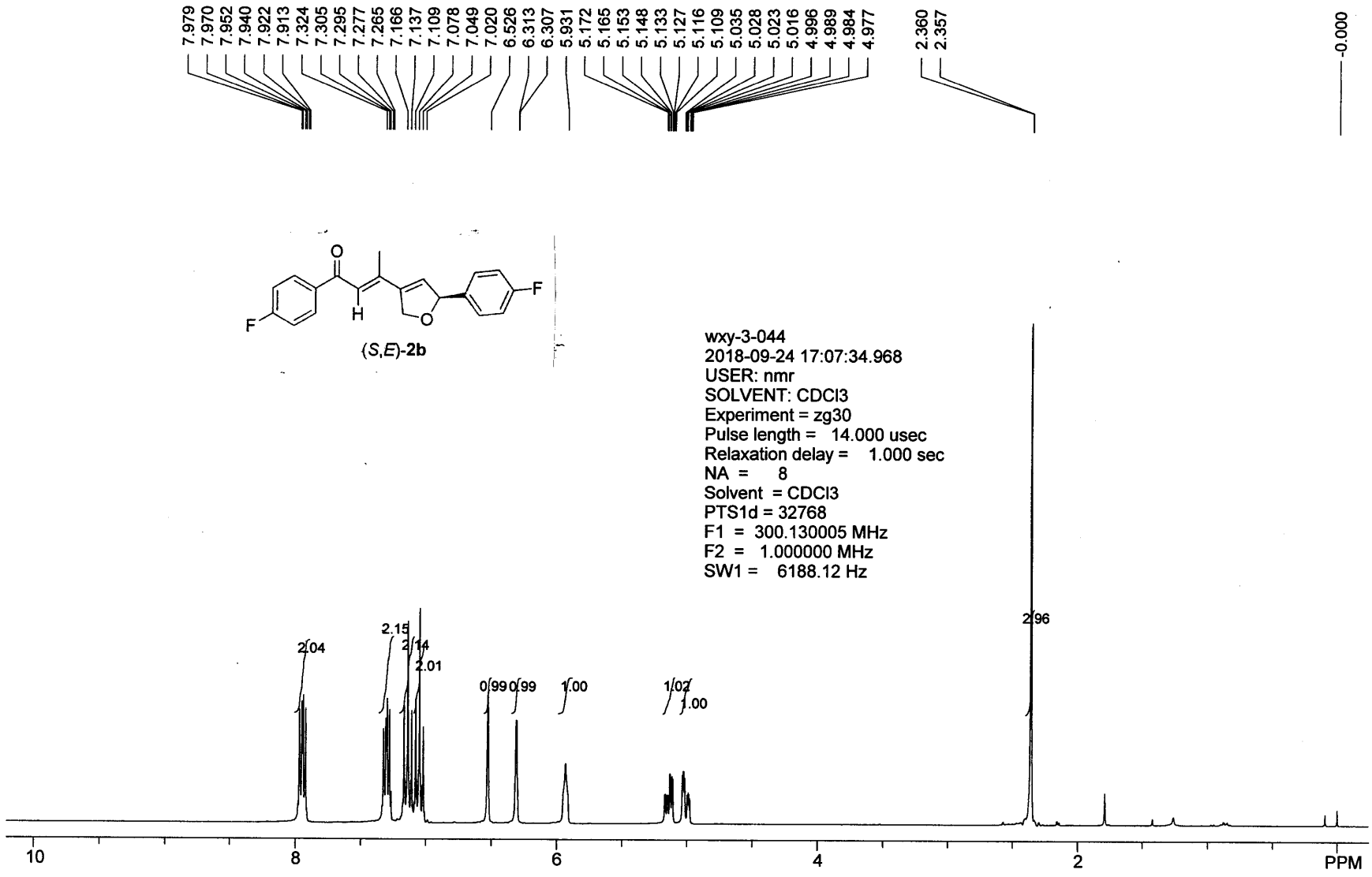
实验内容简介：
od-H,n-hexane/i-PrOH = 95/5,1.0,220

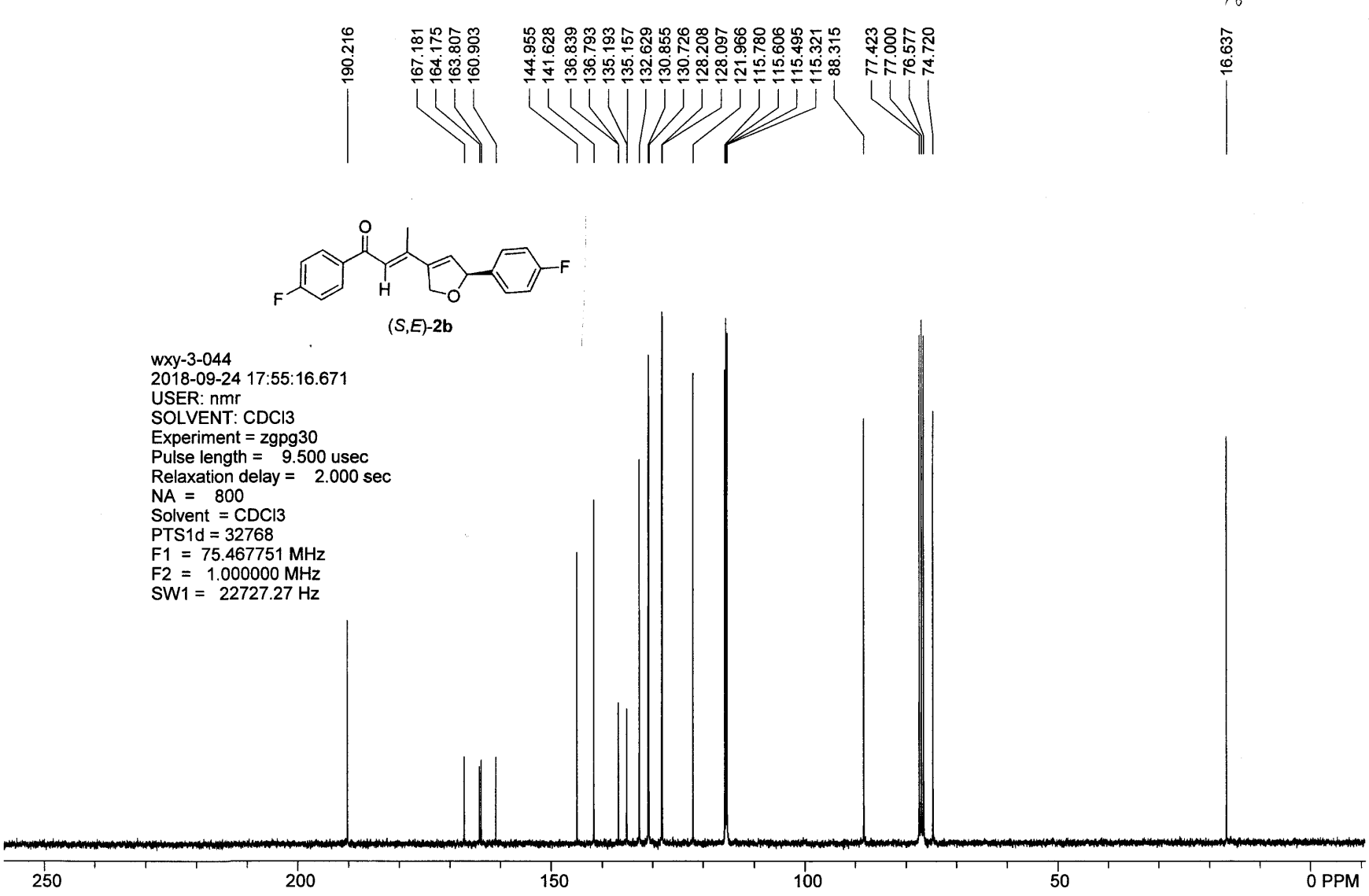
色谱图(S20181122223430.org)

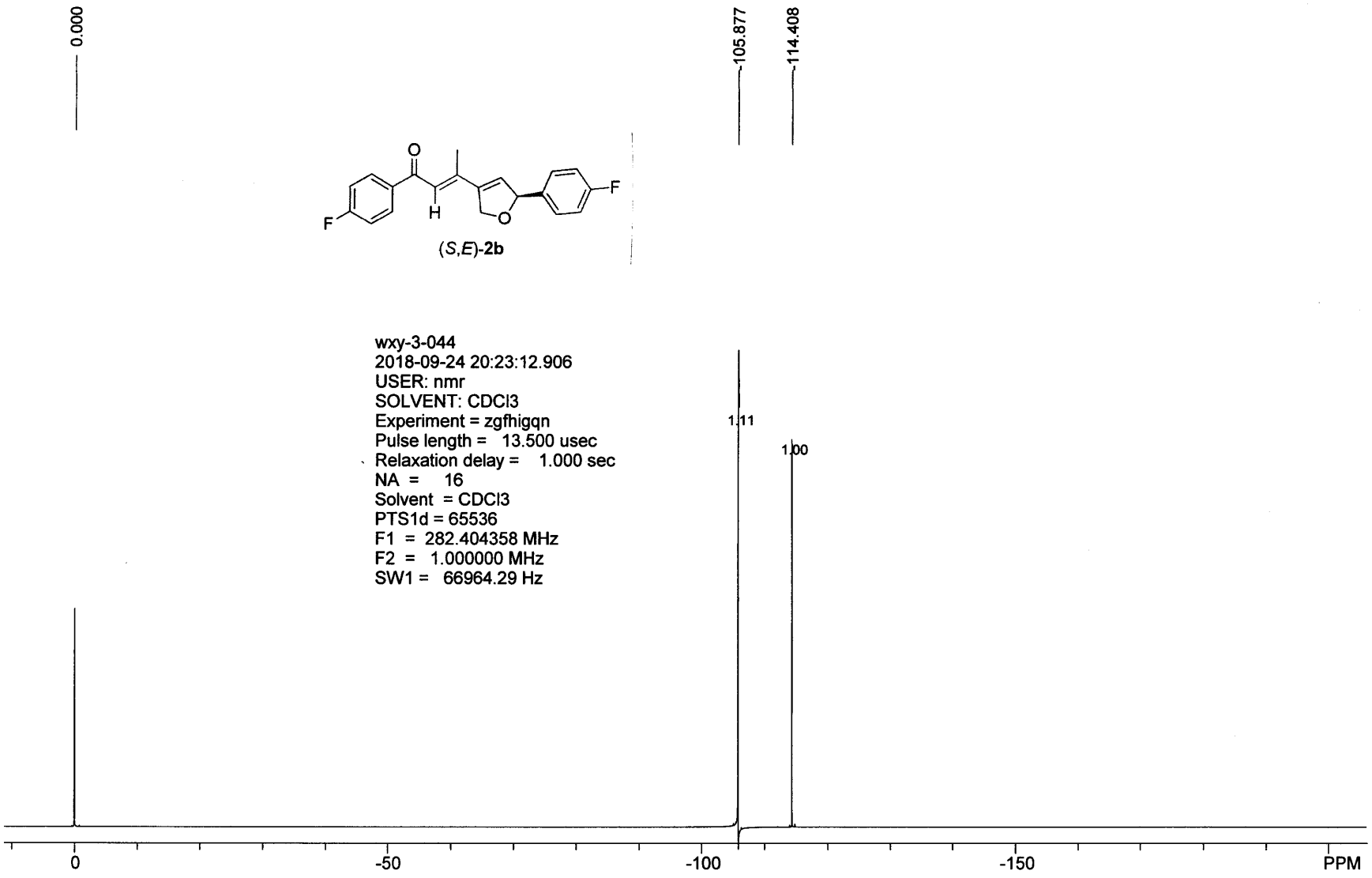


分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		10.253	1485339.625	20370190.000	49.4233
2		15.095	1007126.188	20845612.000	50.5767
总计			2492465.813	41215802.000	100.0000


7.979
7.970
7.952
7.940
7.922
7.913
7.324
7.305
7.295
7.277
7.265
7.166
7.137
7.109
7.078
7.049
7.020
6.526
6.313
6.307
5.931
5.172
5.165
5.153
5.148
5.133
5.127
5.116
5.109
5.035
5.028
5.023
5.016
4.996
4.989
4.984
4.977
2.360
2.357
-0.000
O
H
O
F
F
(S,E)-2b
wxy-3-044
2018-09-24 17:07:34.968
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
2.04
2.15
2.14
2.01
0.99
0.99
1.00
1.02
1.00
2.96
10
8
6
4
2
PPM

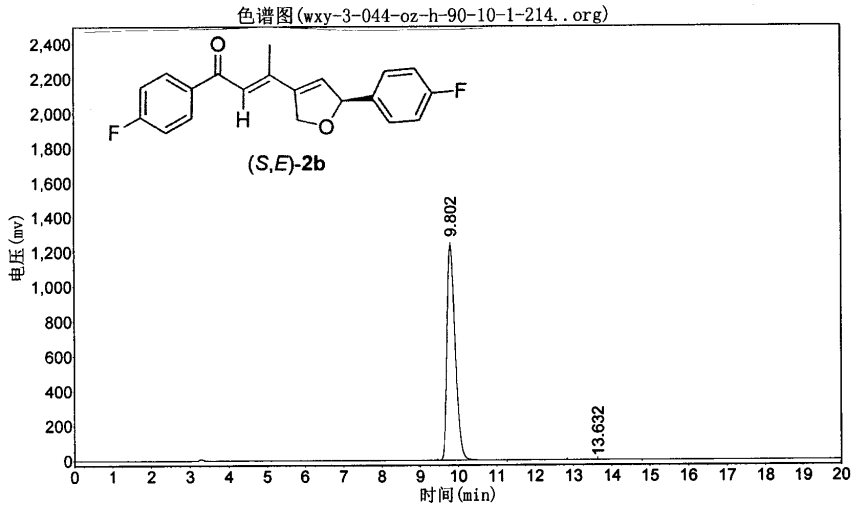

190.216
167.181
164.175
163.807
160.903
144.955
141.628
136.839
136.793
135.193
135.157
132.629
130.855
130.726
128.208
128.097
121.966
115.780
115.606
115.495
115.321
88.315
77.423
77.000
76.577
74.720
16.637
O
H
O
F
F
(S,E)-2b
wxy-3-044
2018-09-24 17:55:16.671
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 800
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
250
200
150
100
50
0 PPM


0.000
-105.877
-114.408
O
H
O
F
F
(S,E)-2b
wxy-3-044
2018-09-24 20:23:12.906
USER: nmr
SOLVENT: CDCl3
Experiment = zgfhigqn
Pulse length = 13.500 usec
Relaxation delay = 1.000 sec
NA = 16
Solvent = CDCl3
PTS1d = 65536
F1 = 282.404358 MHz
F2 = 1.000000 MHz
SW1 = 66964.29 Hz
1.11
1.00
0
-50
-100
-150
PPM

wxy-3-044-oz-h-90-10-1-214

实验时间：2018-09-29,12:33:40　　报告时间：2018-09-29,18:19:29
谱图文件:D:\zhuguangjiong\wxy\20190929\wxy-3-044-oz-h-90-10-1-214..org

实验内容简介：



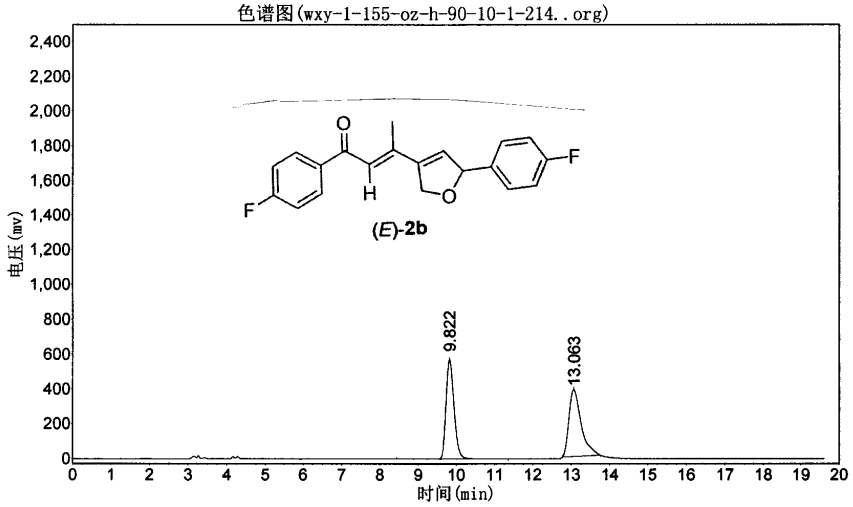
分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		9.802	1238822.125	18647398.000	99.7822
2		13.632	873.859	40696.656	0.2178
总计			1239695.984	18688094.656	100.0000

wxy-1-155-oz-h-90-10-1-214

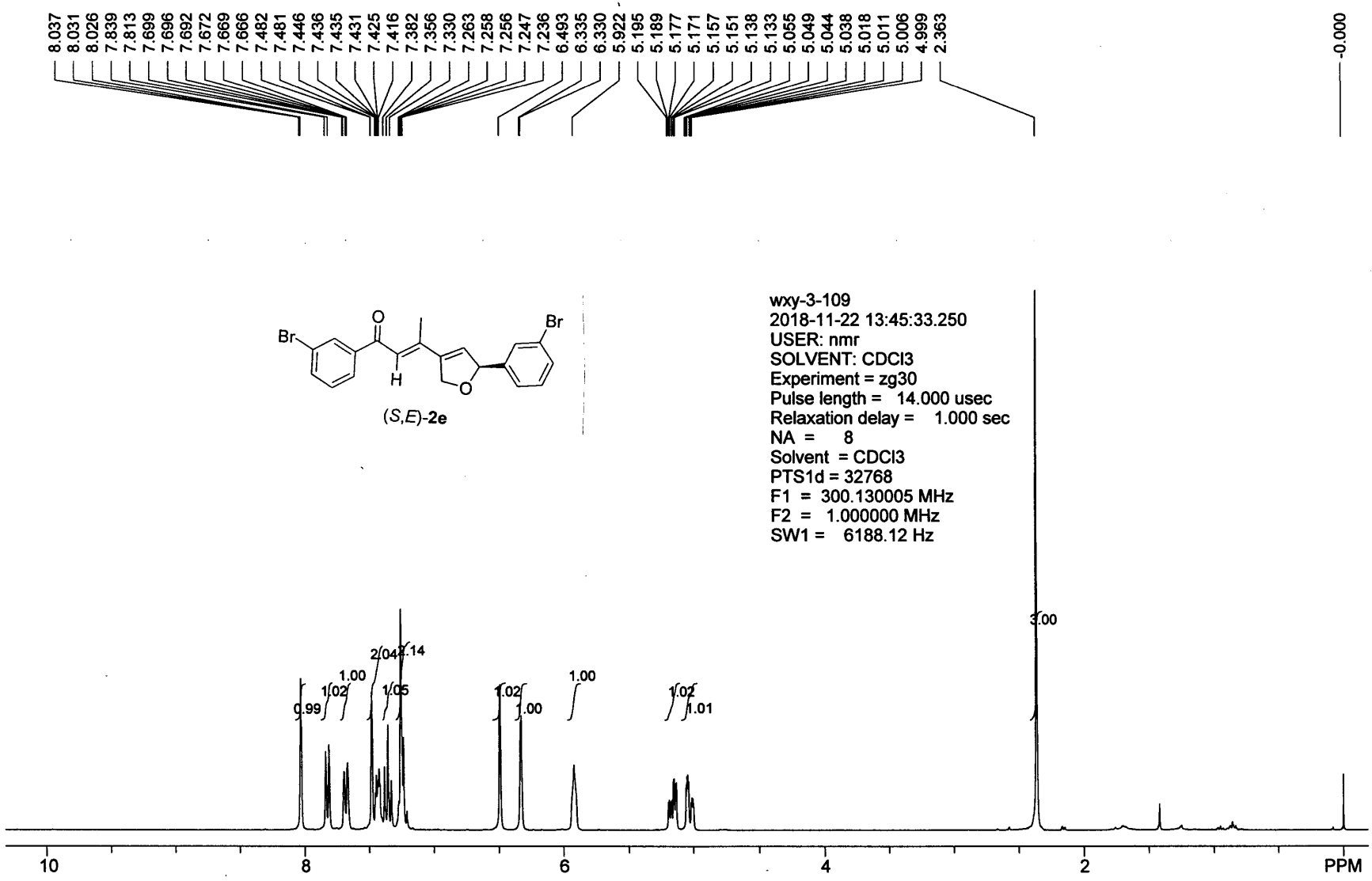
实验时间：2018-09-29,12:09:54　　报告时间：2018-09-29,18:18:35
谱图文件:D:\zhuguangjiong\wxy\20190929\wxy-1-155-oz-h-90-10-1-214..org

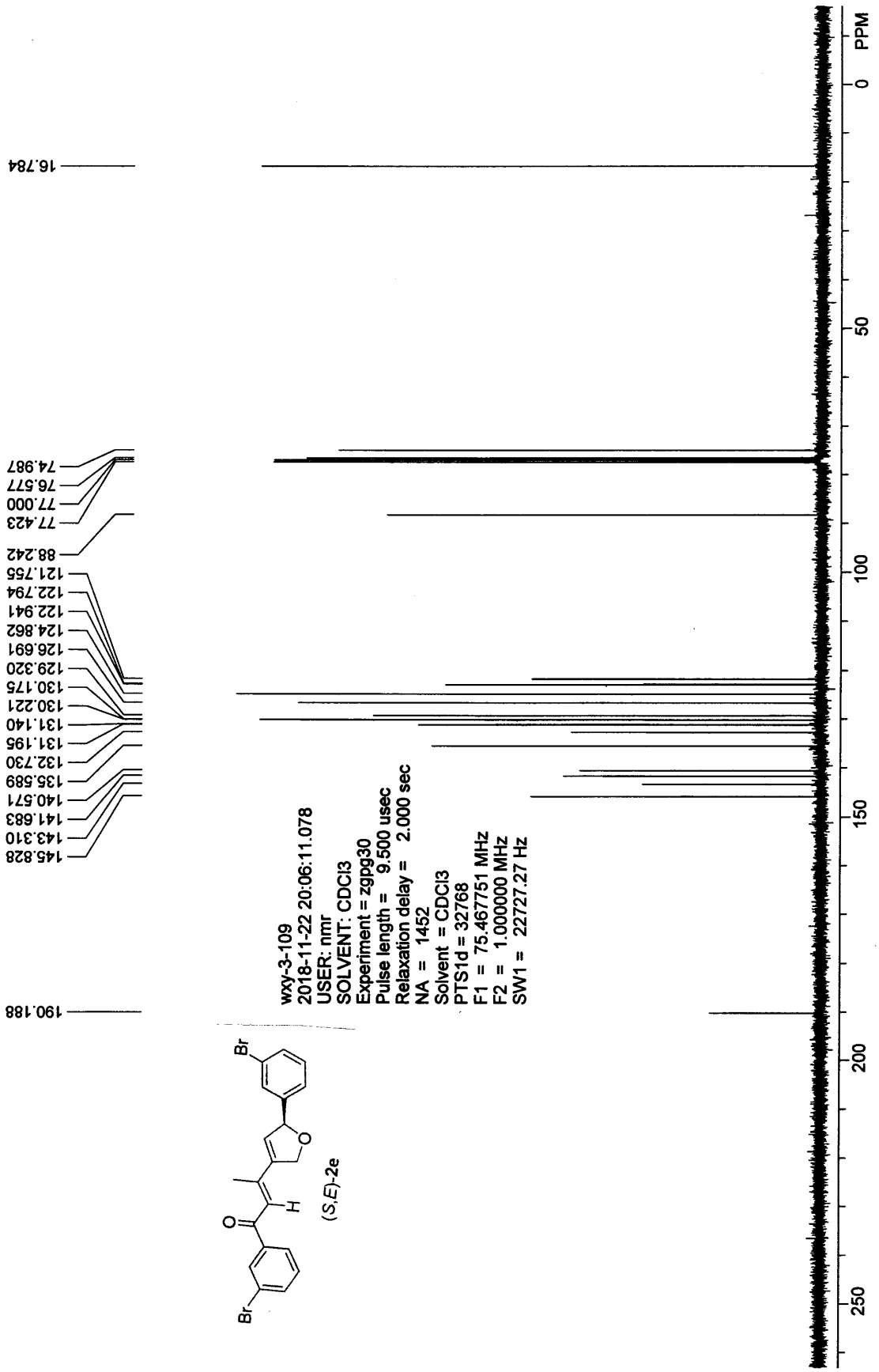
实验内容简介：



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		9.822	572089.688	8352689.000	49.6749
2		13.063	385279.875	8462007.000	50.3251
总计			957369.563	16814696.000	100.0000


8.037
8.031
8.026
7.839
7.813
7.699
7.696
7.692
7.672
7.669
7.666
7.482
7.481
7.446
7.436
7.435
7.431
7.425
7.416
7.382
7.356
7.330
7.263
7.258
7.256
7.247
7.236
6.493
6.335
6.330
5.922
5.195
5.189
5.177
5.171
5.157
5.151
5.138
5.133
5.055
5.049
5.044
5.038
5.018
5.011
5.006
4.999
2.363
-0.000
Br
O
H
O
Br
(S,E)-2e
wxy-3-109
2018-11-22 13:45:33.250
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
0.99
1.02
1.00
2.04
2.14
1.05
1.02
1.00
1.00
1.02
1.01
3.00
10
8
6
4
2
PPM


16.784
74.987
76.577
77.000
77.423
88.242
121.755
122.794
122.941
124.862
126.691
129.320
130.175
130.221
131.140
131.195
132.730
135.589
140.571
141.683
143.310
145.828
190.188
wxy-3-109
2018-11-22 20:06:11.078
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 1452
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
Br
O
H
O
Br
(S,E)-2e
250
200
150
100
50
0
PPM

中国科学院上海有机化学研究所

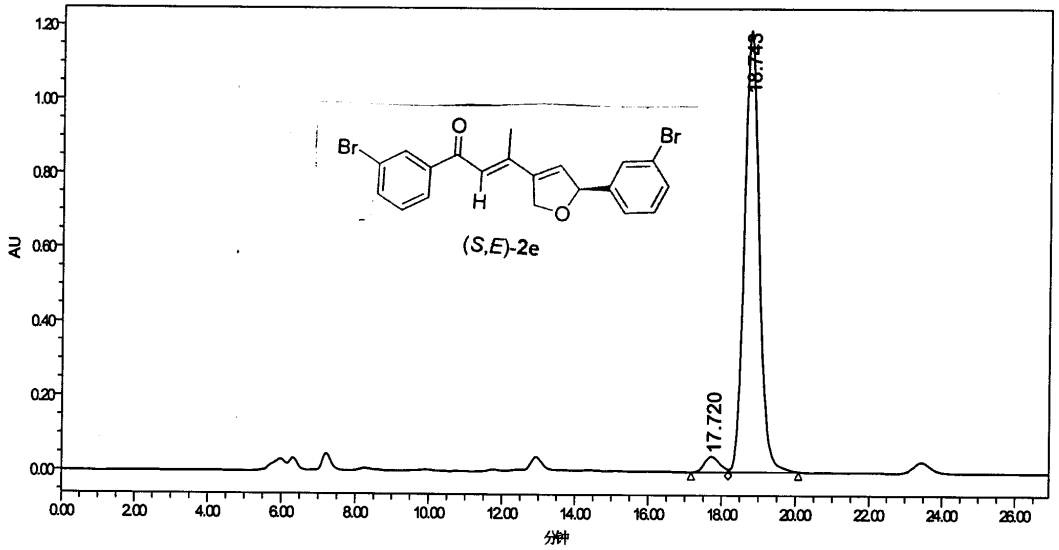
Project Name: defaults for copy

Reported by User: Breeze user (Breeze)

Breeze 2 HPLC System

SAMPLE INFORMATION

Sample Name:	wxy-3-109-io-90-10-1-214	Acquired By:	Breeze
Sample Type:	未知	Date Acquired:	2018/12/7 12:33:21 CST
Vial:	999	Acq. Method:	zgj90
Injection #:	81	Date Processed:	2018/12/7 18:10:28 CST
Injection Volume:	10.00 ul	Channel Name:	W2489 ChA
Run Time:	30.00 Minutes	Channel Desc.:	W2489 ChA 210nm
Column Type:		Sample Set Name:	



	RT (min)	Area (磅sec)	%Area	Height (磅)	% Height
1	17.720	1221580	3.55	41934	3.41
2	18.743	33232016	96.45	1187117	96.59

Report Method: Individual Report ASC

Page: 1 (共计 1)

Printed: 2018/12/7 18:10:55 PRC

中国科学院上海有机化学研究所

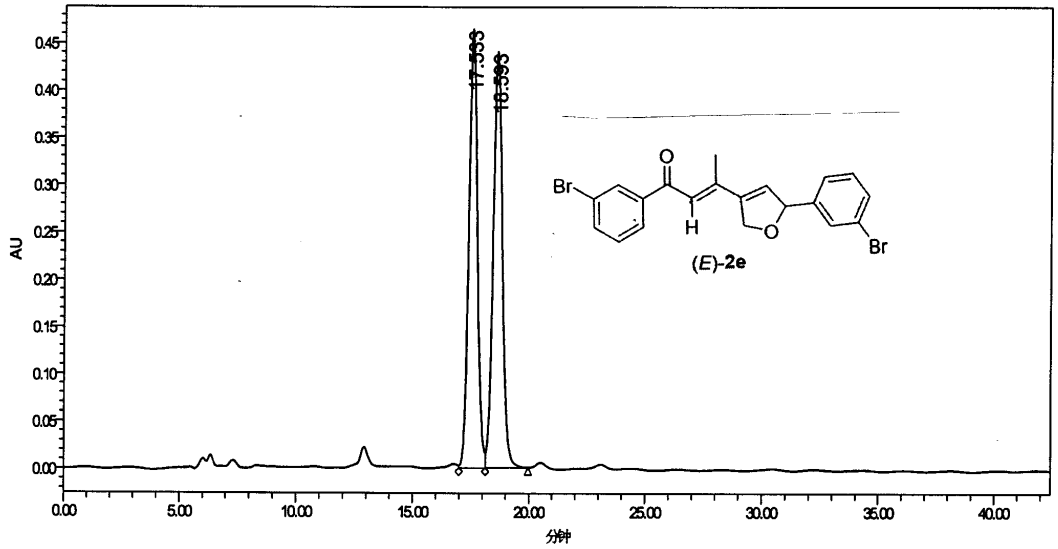
Project Name: defaults for copy

Reported by User: Breeze user (Breeze)

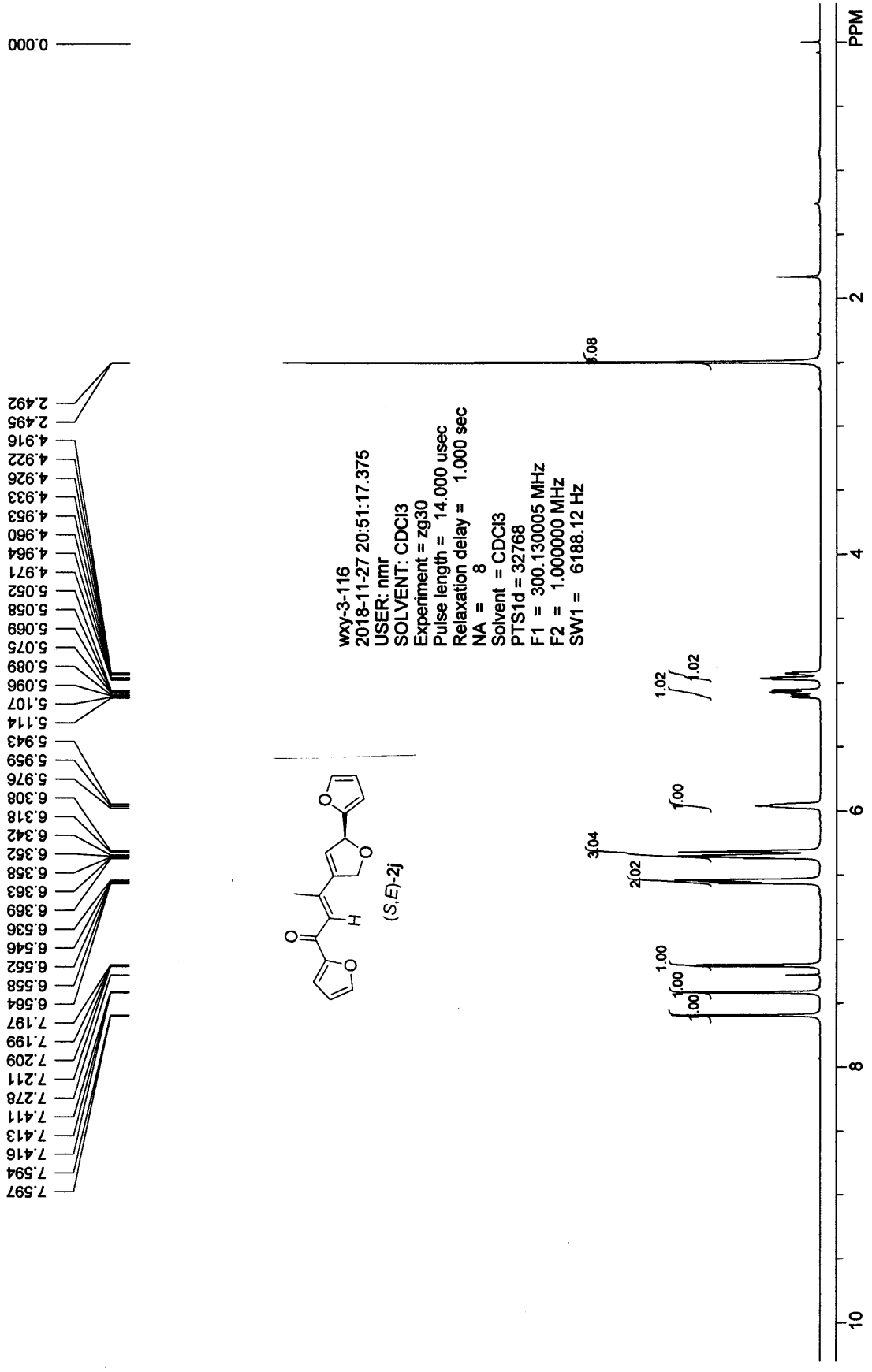
Breeze 2 HPLC System

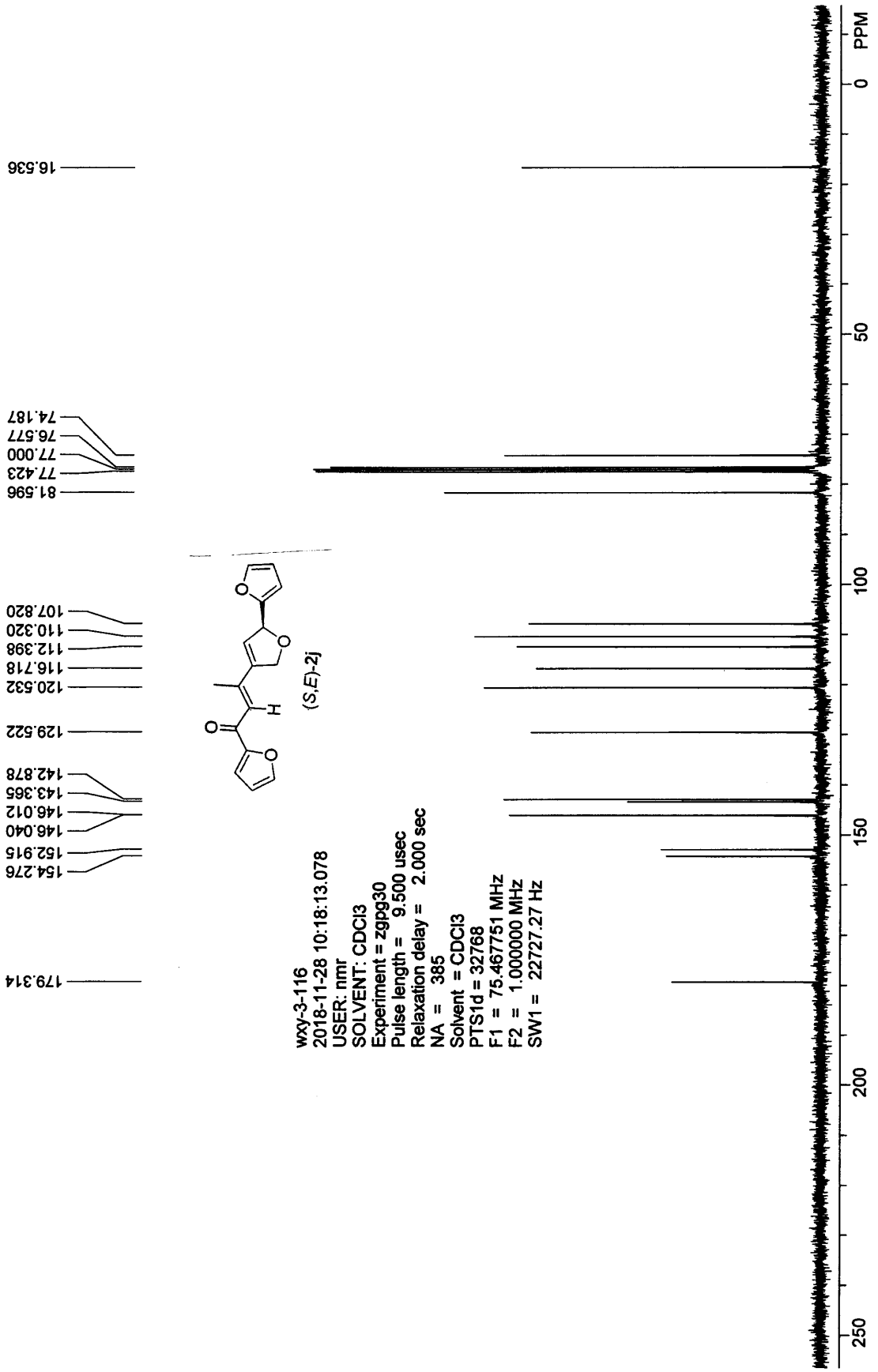
SAMPLE INFORMATION

Sample Name:	wxy-1-192-io-90-10-1-214	Acquired By:	Breeze
Sample Type:	未知	Date Acquired:	2018/12/7 10:36:57 CST
Vial:	999	Acq. Method:	zgj90
Injection #:	78	Date Processed:	2018/12/7 18:06:22 CST
Injection Volume:	10.00 ul	Channel Name:	W2489 ChA
Run Time:	45.00 Minutes	Channel Desc.:	W2489 ChA 210nm
Column Type:		Sample Set Name:	



	RT (min)	Area (毫sec)	% Area	Height (毫	% Height
1	17.533	11893795	49.62	464008	51.31
2	18.589	12077161	50.38	440304	48.69


wxy-3-116
2018-11-27 20:51:17.375
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
(S,E)-2j
PPM
0.000
2.492
2.495
4.916
4.922
4.926
4.933
4.953
4.960
4.964
4.971
5.052
5.058
5.069
5.075
5.089
5.096
5.107
5.114
5.943
5.959
5.976
6.308
6.318
6.342
6.352
6.358
6.363
6.369
6.536
6.546
6.552
6.558
6.564
7.197
7.199
7.209
7.211
7.278
7.411
7.413
7.416
7.594
7.597


16.536
74.187
76.577
77.000
77.423
81.596
107.820
110.320
112.398
116.718
120.532
129.522
142.878
143.365
146.012
146.040
152.915
154.276
179.314
(S,E)-2j
wxy-3-116
2018-11-28 10:18:13.078
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 385
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
250
200
150
100
50
0
PPM

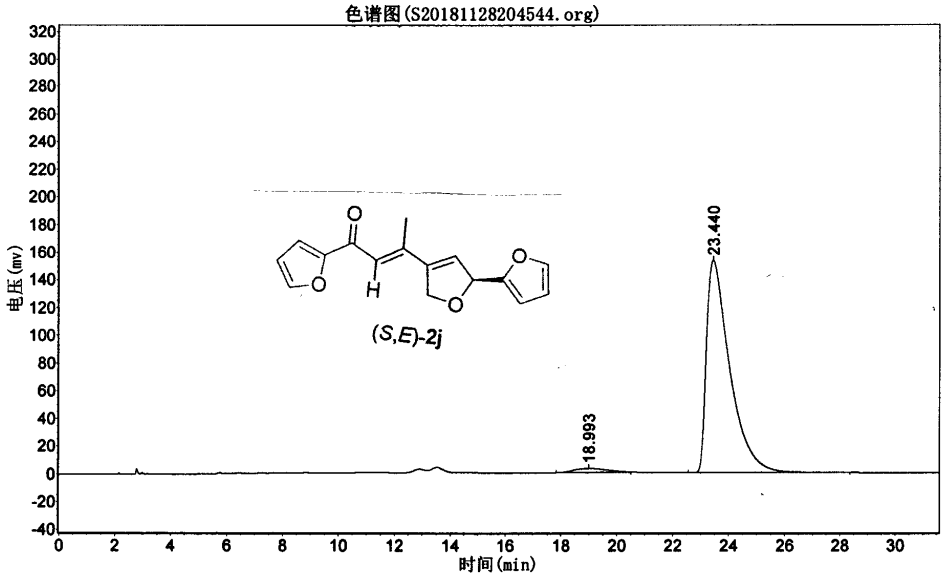
wxy-3-116-Chiral

实验时间：2018-11-28,20:45:44
谱图文件:D:\浙大智达\N2000\样品\S20181128204544.org
方法文件:D:\浙大智达\N2000\djx.mtd

实验者：wxy
报告时间：2018-12-03,15:55:04
积分方法:面积归一法

实验内容简介：
od-H,n-hexane/i-PrOH = 85/15,1.0,220

色谱图(S20181128204544.org)



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		18.993	2841.981	232215.250	2.5592
2		23.440	153313.500	8841646.000	97.4408
总计			156155.481	9073861.250	100.0000

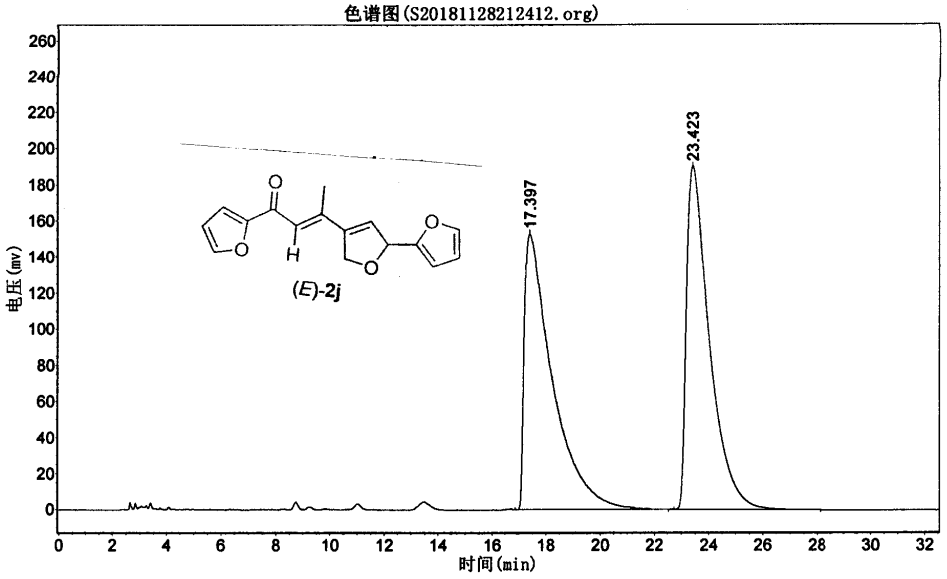
wxy-3-116-racemic

实验时间：2018-11-28,21:24:12
谱图文件:D:\浙大智达\N2000\样品\S20181128212412.org
方法文件:D:\浙大智达\N2000\djx.mtd

实验者：wxy
报告时间：2018-11-28,22:46:15
积分方法:面积归一法

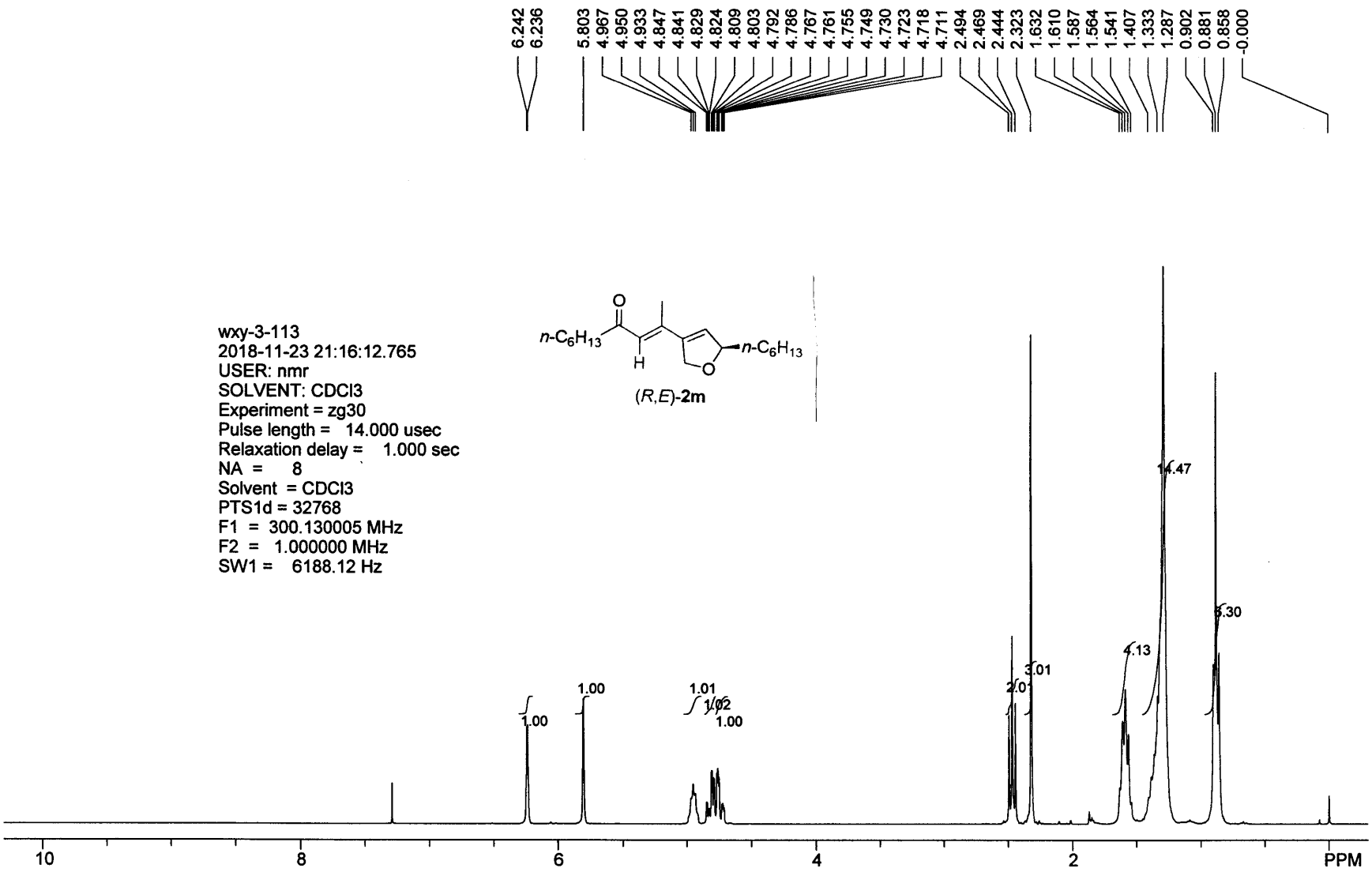
实验内容简介：
od-H,n-hexane/i-PrOH = 85/15,1.0,220

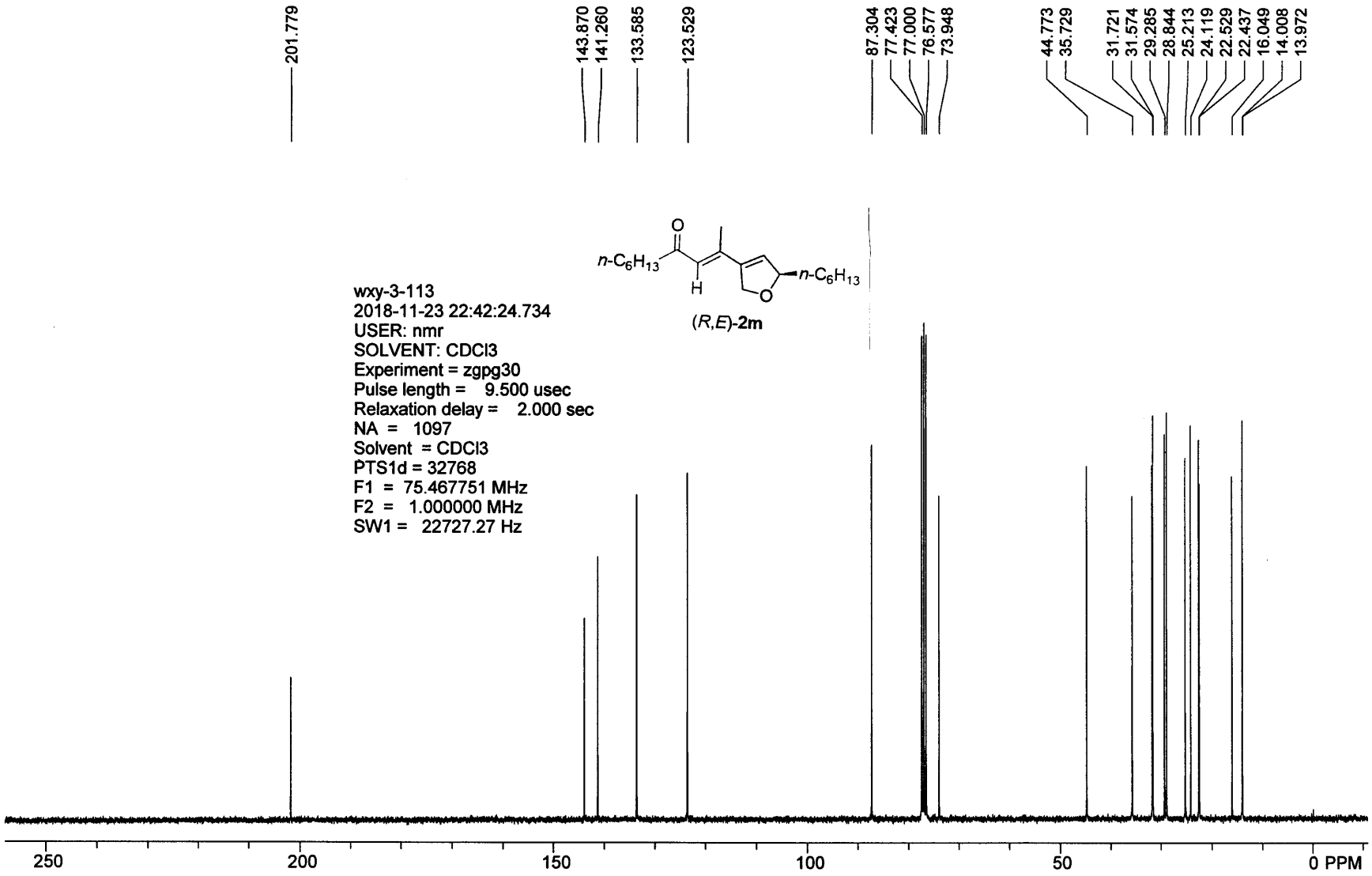
色谱图(S20181128212412.org)



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		17.397	152830.547	10991313.000	49.6236
2		23.423	190313.594	11158053.000	50.3764
总计			343144.141	22149366.000	100.0000


6.242
6.236
5.803
4.967
4.950
4.933
4.847
4.841
4.829
4.824
4.809
4.803
4.792
4.786
4.767
4.761
4.755
4.749
4.730
4.723
4.718
4.711
2.494
2.469
2.444
2.323
1.632
1.610
1.587
1.564
1.541
1.407
1.333
1.287
0.902
0.881
0.858
-0.000
wxy-3-113
2018-11-23 21:16:12.765
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
n-C6H13
n-C6H13
(R,E)-2m
1.00
1.00
1.01
1.00
2.01
3.01
4.13
14.47
6.30
10
8
6
4
2
PPM


201.779
143.870
141.260
133.585
123.529
87.304
77.423
77.000
76.577
73.948
44.773
35.729
31.721
31.574
29.285
28.844
25.213
24.119
22.529
22.437
16.049
14.008
13.972
n-C6H13
n-C6H13
O
H
O
(R,E)-2m
wxy-3-113
2018-11-23 22:42:24.734
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 1097
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
250
200
150
100
50
0 PPM

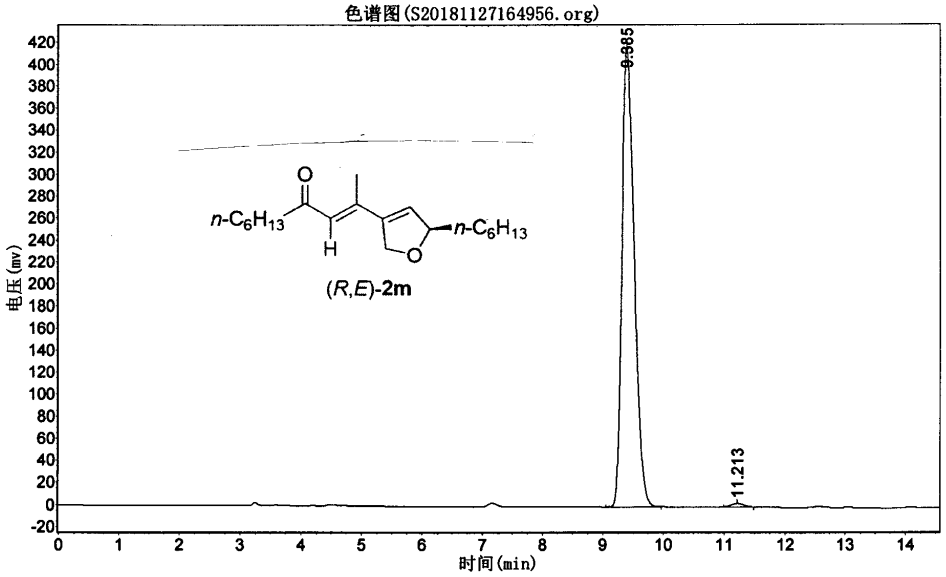
wxy-3-113-chiral

实验时间：2018-11-27,16:49:56
谱图文件:D:\浙大智达\N2000\样品\S20181127164956.org
方法文件:D:\浙大智达\N2000\djx.mtd

实验者：wxy
报告时间：2018-11-27,21:42:53
积分方法:面积归一法

实验内容简介：
IC,n-hexane/i-PrOH = 99/1,1.0, 220

色谱图(S20181127164956.org)



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		9.385	417135.250	5857646.000	99.1850
2		11.213	3373.337	48129.504	0.8150
总计			420508.587	5905775.504	100.0000

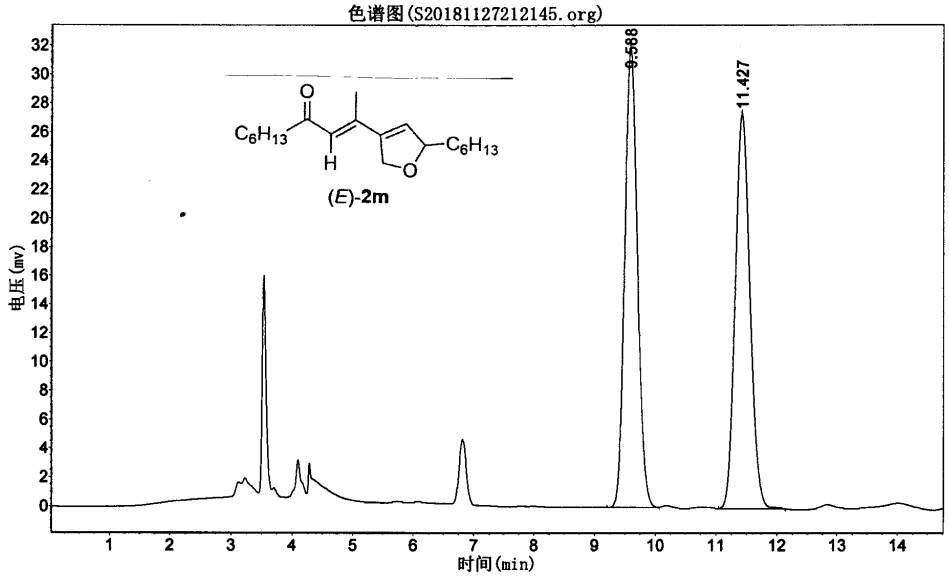
wxy-3-113-racemic

实验时间：2018-11-27,21:21:45
谱图文件:D:\浙大智达\N2000\样品\S20181127212145.org
方法文件:D:\浙大智达\N2000\djx.mtd

实验者：wxy
报告时间：2018-11-27,21:40:13
积分方法:面积归一法

实验内容简介：
IC,n-hexane/i-PrOH = 99/1,1.0,220

色谱图(S20181127212145.org)



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		9.588	31870.615	462511.313	50.1733
2		11.427	27356.883	459315.875	49.8267
总计			59227.498	921827.188	100.0000

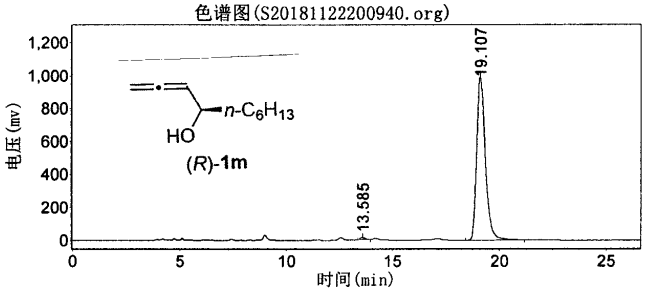
fjj-1-016-chiral

实验时间：2018-11-22,20:09:40
谱图文件:D:\浙大智达\N2000\样品\S20181122200940.org
方法文件:D:\浙大智达\N2000\djx.mtd

实验者：wxy
报告时间：2018-11-22,23:03:32
积分方法:面积归一法

实验内容简介：
od-H,n-hexane/i-PrOH = 90/10,0.7,220

色谱图(S20181122200940.org)



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		13.585	14308.385	224406.656	0.7979
2		19.107	993045.313	27898604.000	99.2021
总计			1007353.697	28123010.656	100.0000

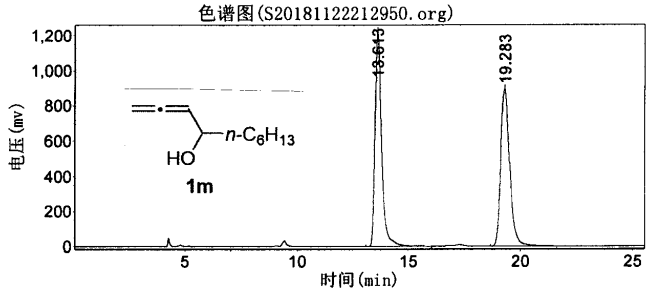
fjj-1-016-racemic

实验时间：2018-11-22,21:29:50
谱图文件:D:\浙大智达\N2000\样品\S20181122212950.org
方法文件:D:\浙大智达\N2000\djx.mtd

实验者：fjj
报告时间：2018-11-22,22:58:33
积分方法:面积归一法

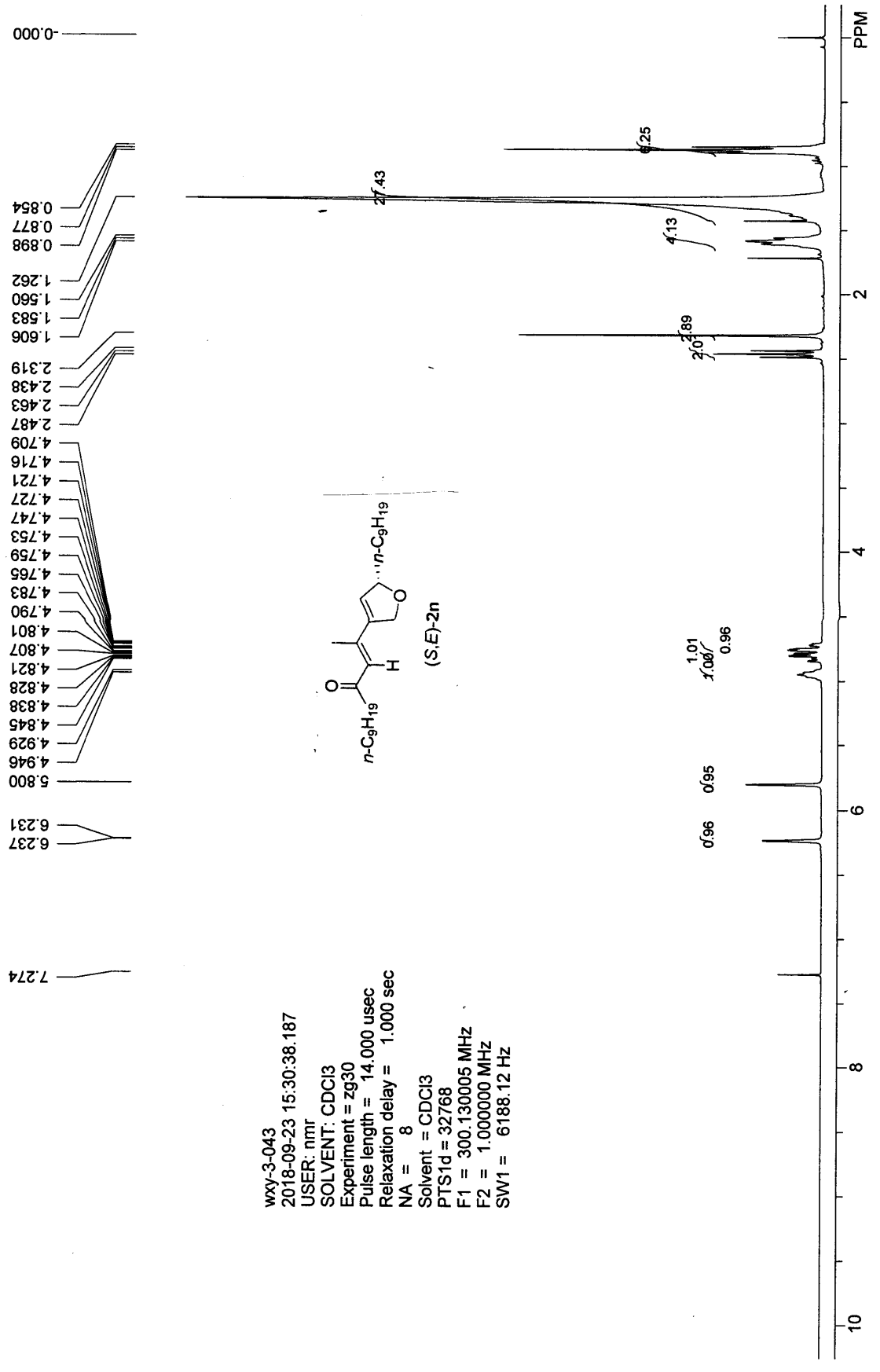
实验内容简介：
od-H,n-hexane/i-PrOH = 90/10,0.7,220

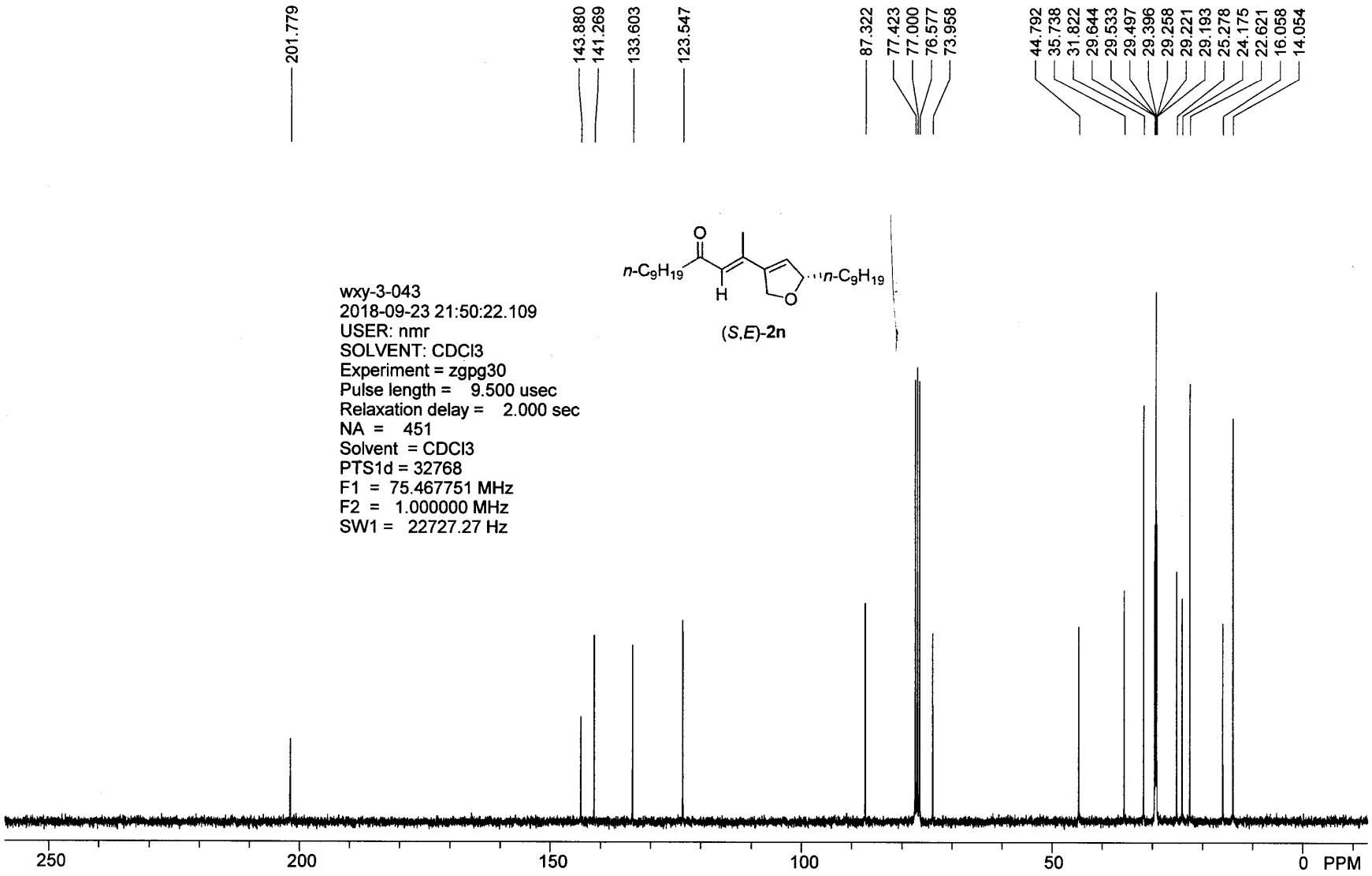
色谱图(S20181122212950.org)



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		13.613	1225907.500	25309176.000	49.9859
2		19.283	895078.813	25323422.000	50.0141
总计			2120986.313	50632598.000	100.0000


wxy-3-043
2018-09-23 15:30:38.187
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
(S,E)-2n
PPM
0.000
0.854
0.877
0.898
1.262
1.560
1.583
1.606
2.319
2.438
2.463
2.487
4.709
4.716
4.721
4.727
4.747
4.753
4.759
4.765
4.783
4.790
4.801
4.807
4.821
4.828
4.838
4.845
4.929
4.946
5.800
6.231
6.237
7.274
0.96
0.95
1.01
1.00
0.96
2.03
2.89
4.13
6.25
2.43


201.779
143.880
141.269
133.603
123.547
87.322
77.423
77.000
76.577
73.958
44.792
35.738
31.822
29.644
29.533
29.497
29.396
29.258
29.221
29.193
25.278
24.175
22.621
16.058
14.054
n-C9H19
n-C9H19
(S,E)-2n
wxy-3-043
2018-09-23 21:50:22.109
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 451
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
250
200
150
100
50
0 PPM

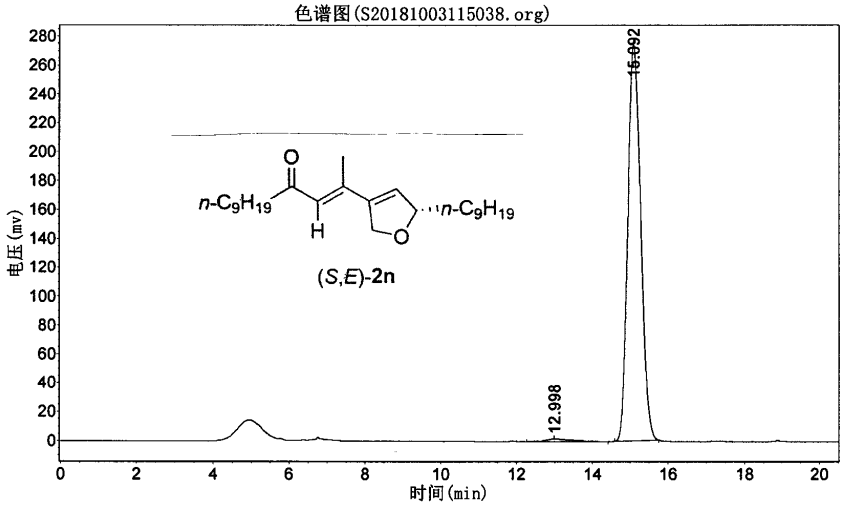
wxy-3-043-chiral

实验时间：2018-10-03,11:50:38
谱图文件:D:\浙大智达\N2000\样品\S20181003115038.org
方法文件:D:\浙大智达\N2000\djx.mtd

实验者：wxy
报告时间：2018-10-19,19:56:54
积分方法:面积归一法

实验内容简介：
IC,n-hexane/i-PrOH = 99/1,0.7,254

色谱图(S20181003115038.org)



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		12.998	1624.292	73424.297	1.1237
2		15.092	274093.281	6460805.500	98.8763
总计			275717.573	6534229.797	100.0000

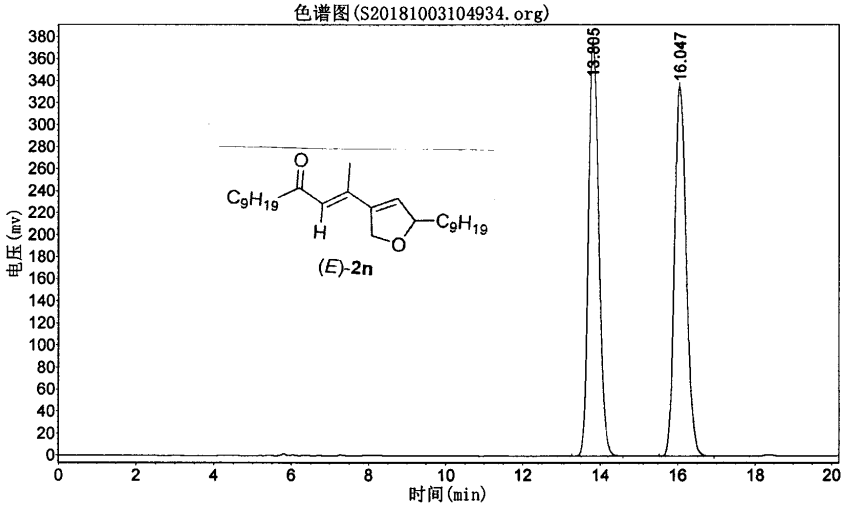
wxy-3-041-racemic

实验时间：2018-10-03,10:49:34
谱图文件:D:\浙大智达\N2000\样品\S20181003104934.org
方法文件:D:\浙大智达\N2000\djx.mtd

实验者：wxy
报告时间：2018-10-03,11:32:28
积分方法:面积归一法

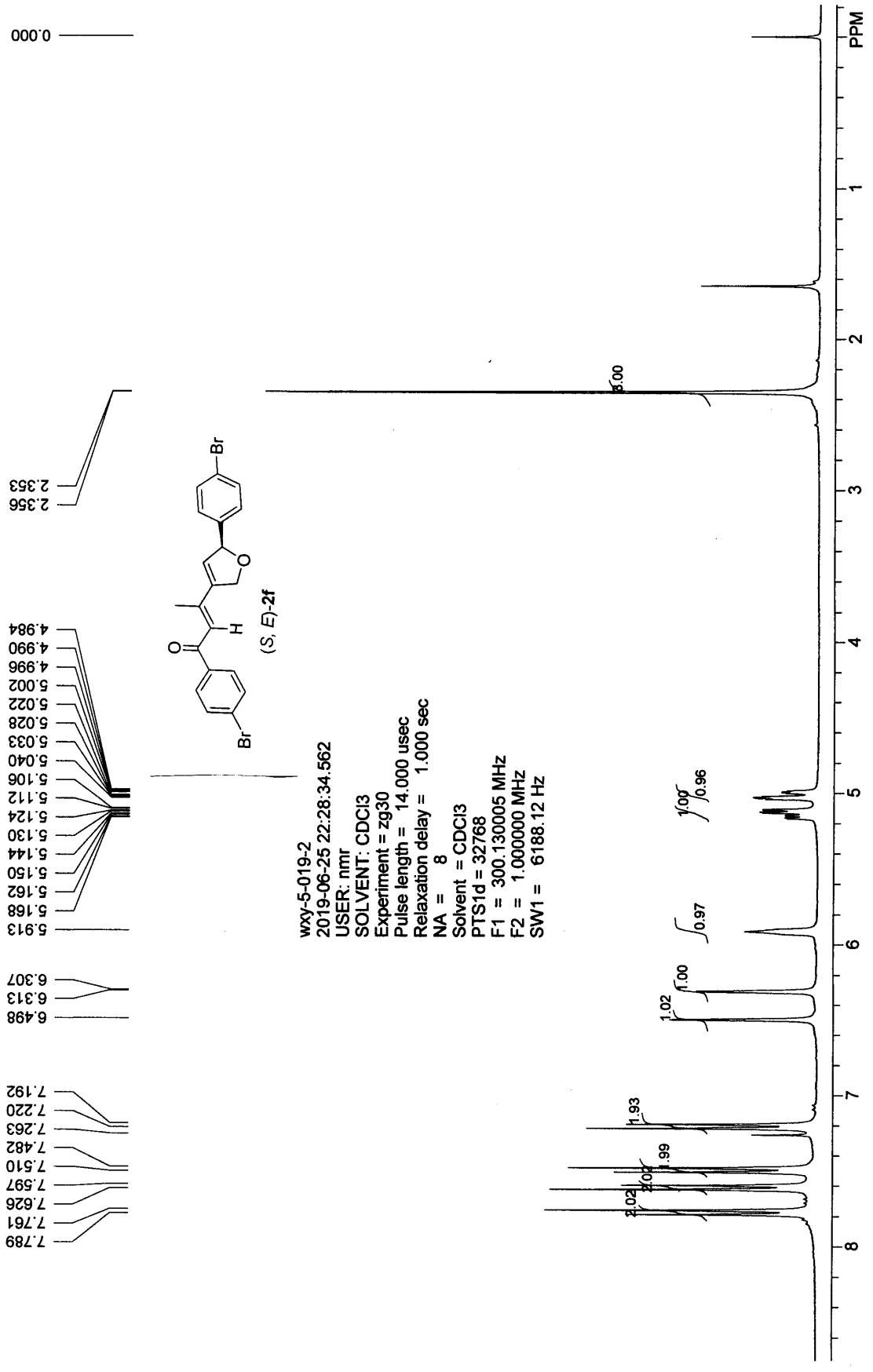
实验内容简介：
IC,n-hexane/i-PrOH = 99/1,0.7,254

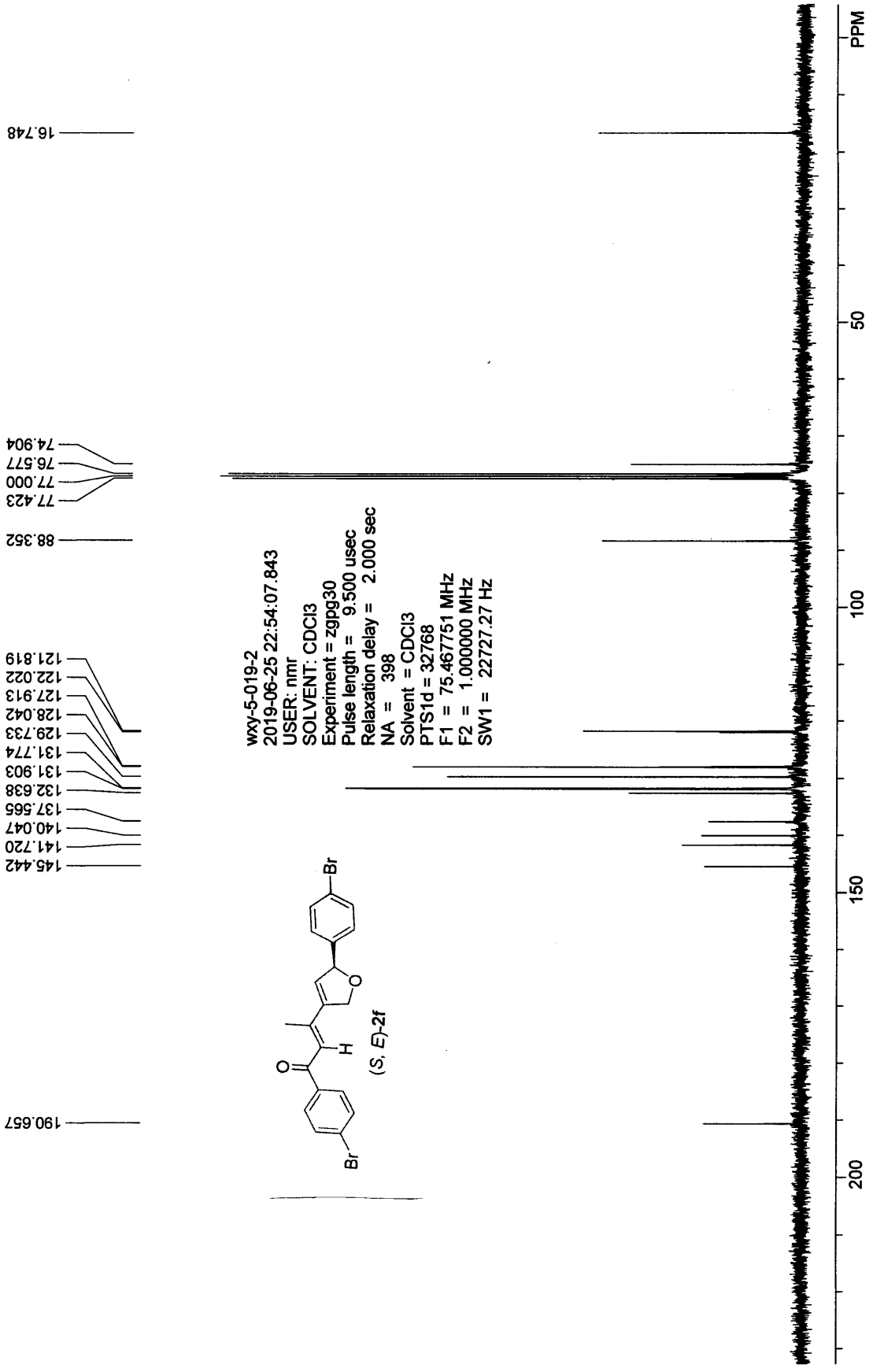
色谱图(S20181003104934.org)



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		13.805	376373.594	7049943.500	50.0617
2		16.047	335684.969	7032569.500	49.9383
总计			712058.563	14082513.000	100.0000


wxy-5-019-2
2019-06-25 22:28:34.562
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
(S, E)-2f
Br
O
H
O
Br
PPM
0.000
2.353
2.356
4.984
4.990
4.996
5.002
5.022
5.028
5.033
5.040
5.106
5.112
5.124
5.130
5.144
5.150
5.162
5.168
5.913
6.307
6.313
6.498
7.192
7.220
7.263
7.482
7.510
7.597
7.626
7.761
7.789
6.00
1.02
1.00
0.97
1.00
0.96
1.93
1.99
2.08
2.02


wxy-5-019-2
2019-06-25 22:54:07.843
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 398
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
(S, E)-2f
PPM
16.748
74.904
76.577
77.000
77.423
88.352
121.819
122.022
127.913
128.042
129.733
131.774
131.903
132.638
137.565
140.047
141.720
145.442
190.657

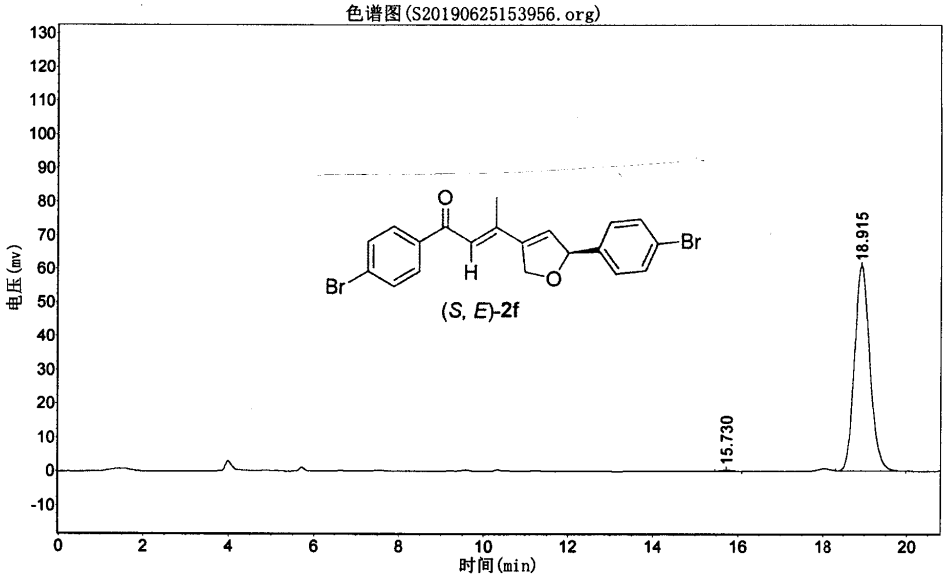
wxy-5-019(Chiral)

实验时间：2019-06-25,15:39:56
谱图文件:D:\浙大智达\N2000\样品\S20190625153956.org
方法文件:D:\浙大智达\N2000\djx.mtd

实验者：wxy
报告时间：2019-06-25,16:09:57
积分方法:面积归一法

实验内容简介：
IC,n-hexane/i-PrOH = 90/10,0.8,254

色谱图(S20190625153956.org)



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		15.730	283.355	5224.500	0.3407
2		18.915	60557.109	1528178.500	99.6593
总计			60840.465	1533403.000	100.0000

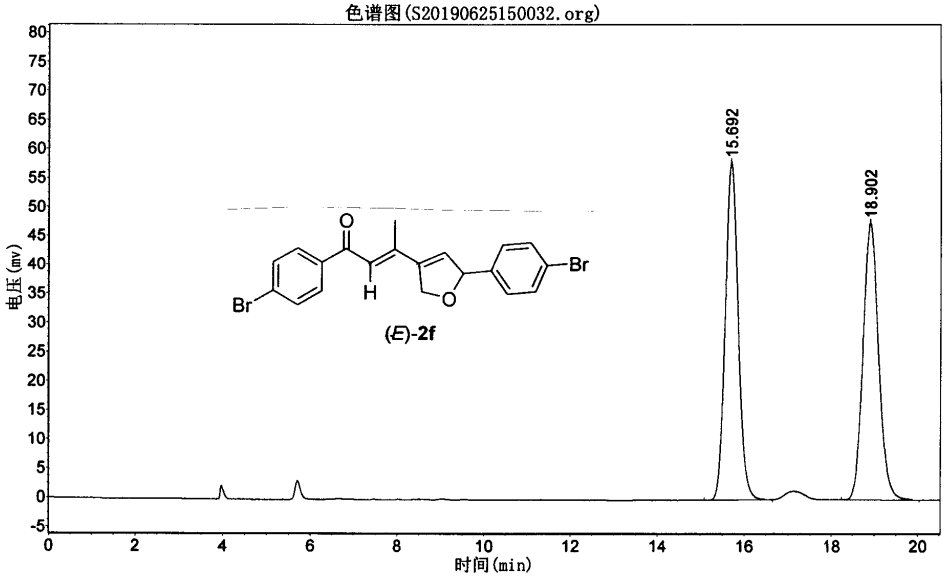
WXY-5-012(racemic)

实验时间：2019-06-25,15:00:32
谱图文件:D:\浙大智达\N2000\样品\S20190625150032.org
方法文件:D:\浙大智达\N2000\djx.mtd

实验者：wxy
报告时间：2019-06-25,16:07:08
积分方法:面积归一法

实验内容简介：
IC,n-hexane/i-PrOH = 90/10,0.8,254

色谱图(S20190625150032.org)



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		15.692	58021.137	1190815.125	50.0806
2		18.902	47523.688	1186981.500	49.9194
总计			105544.824	2377796.625	100.0000

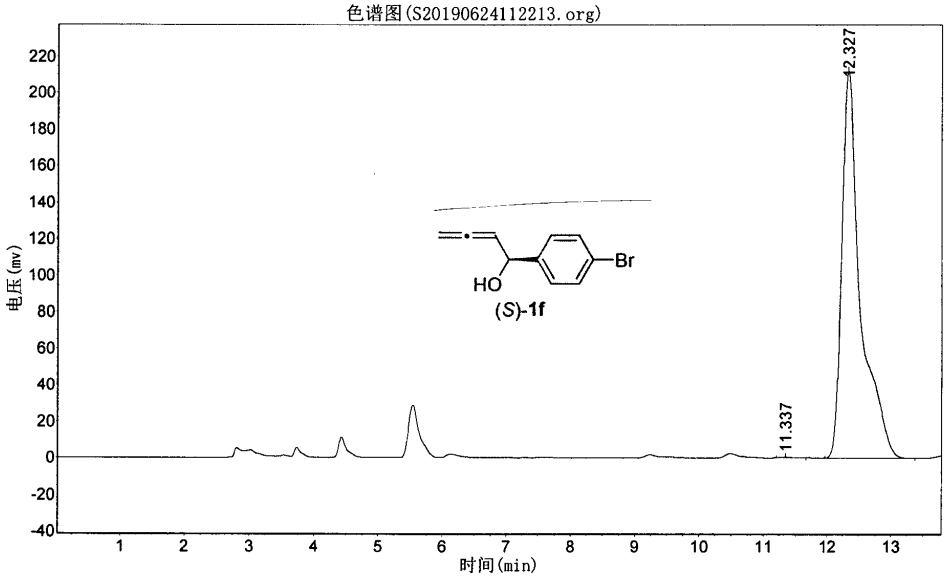
wxy-5-015-Chiral

实验时间：2019-06-24,11:22:13
谱图文件:D:\浙大智达\N2000\样品\S20190624112213.org
方法文件:D:\浙大智达\N2000\djx.mtd

实验者：wxy
报告时间：2019-06-24,12:13:37
积分方法:面积归一法

实验内容简介：
AD-H,n-hexane/i-PrOH = 95/5,1.0,220

色谱图(S20190624112213.org)



分析结果表

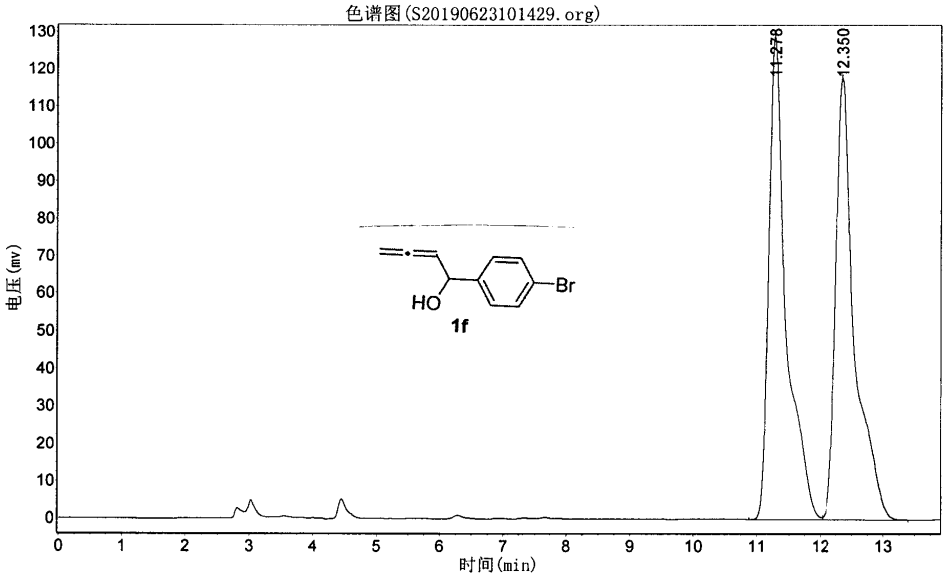
峰号	峰名	保留时间	峰高	峰面积	含量
1		11.337	355.909	4290.550	0.1000
2		12.327	212233.563	4288239.500	99.9000
总计			212589.471	4292530.050	100.0000

WXY-5-015-racemic

实验时间：2019-06-23,10:14:29
谱图文件:D:\浙大智达\N2000\样品\S20190623101429.org
方法文件:D:\浙大智达\N2000\djx.mtd

实验者：wxy
报告时间：2019-06-23,22:41:58
积分方法:面积归一法

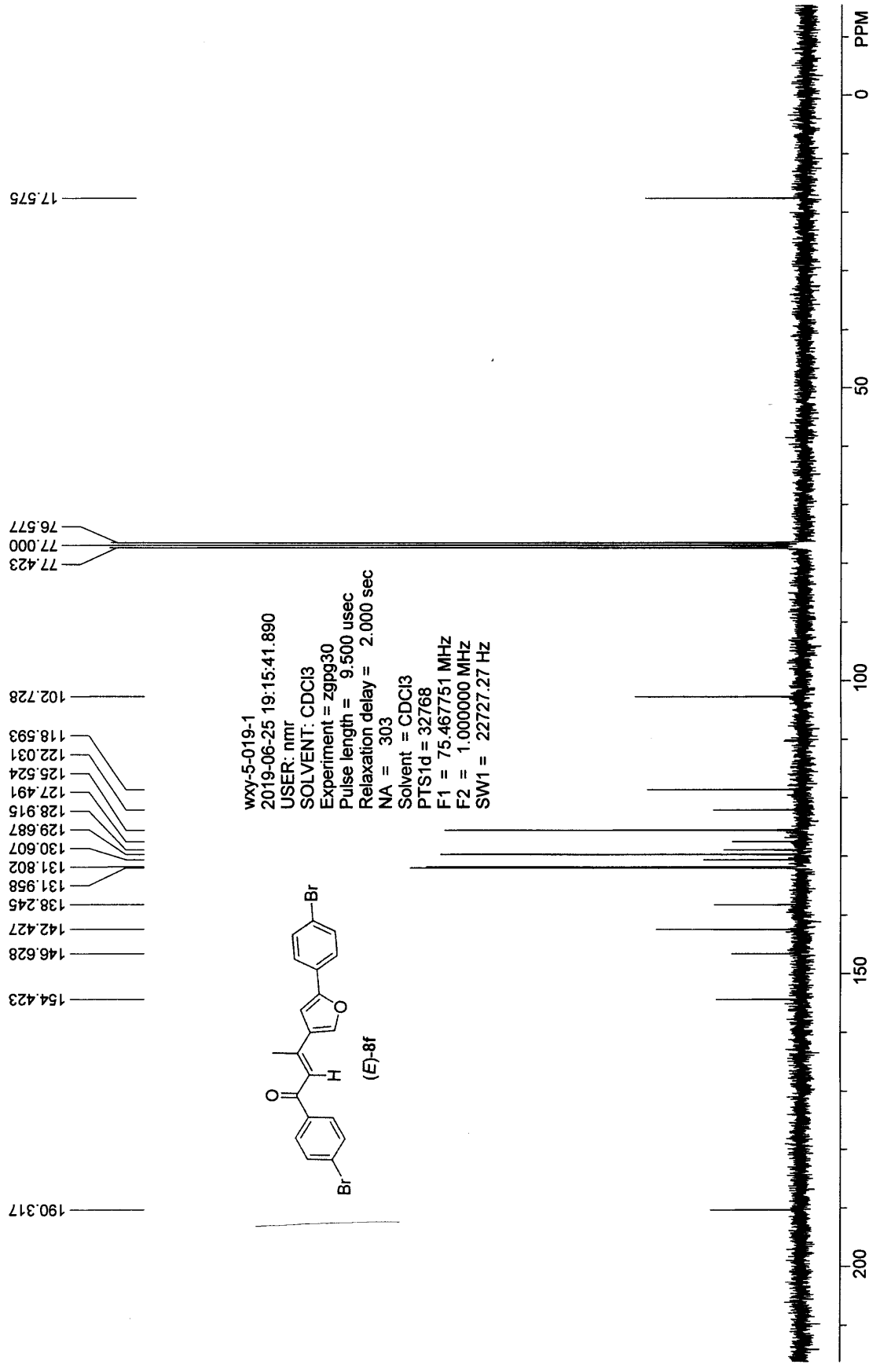
实验内容简介：
AD-H,n-hexane/i-PrOH = 95/5,1.0,220

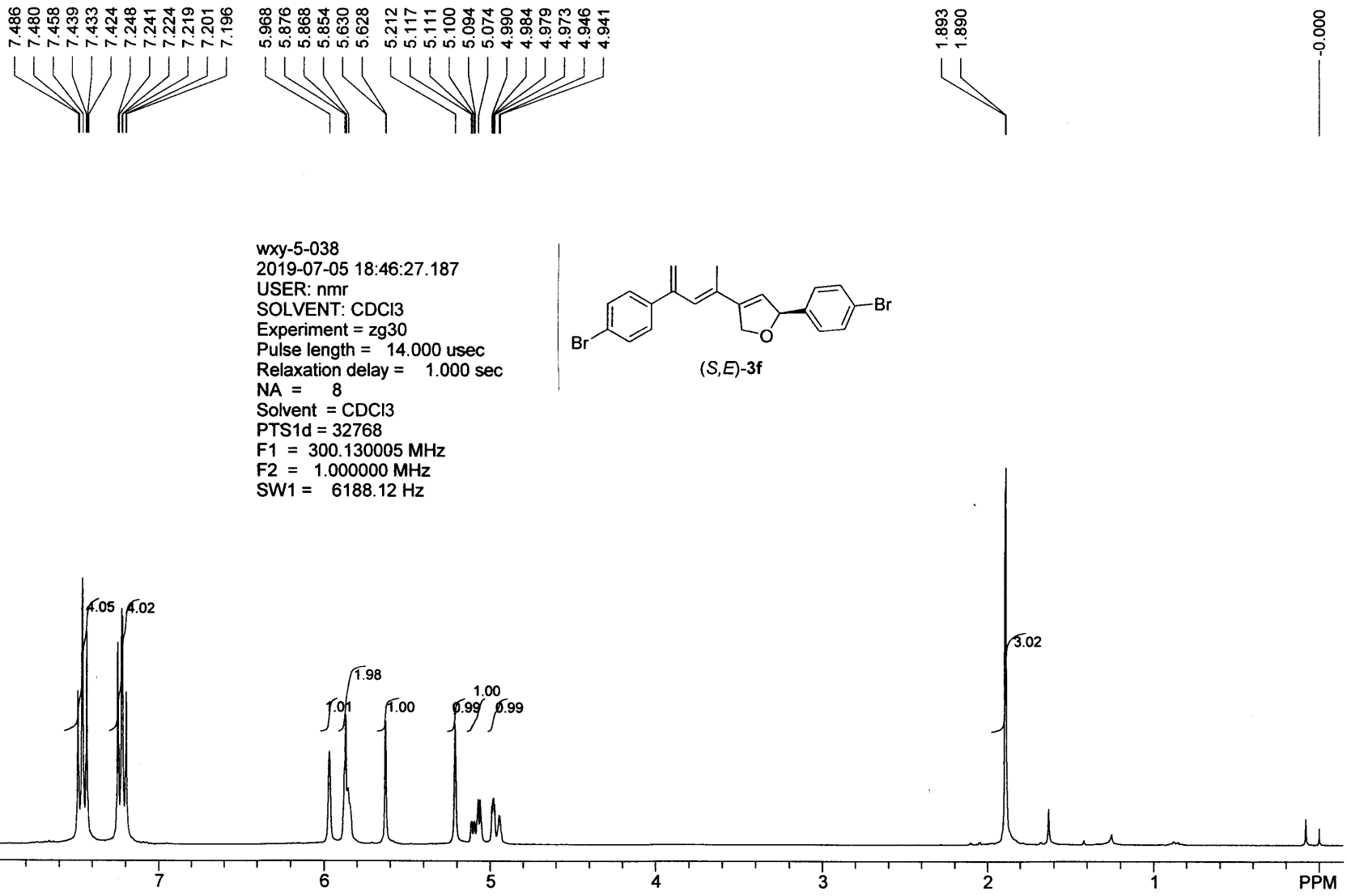


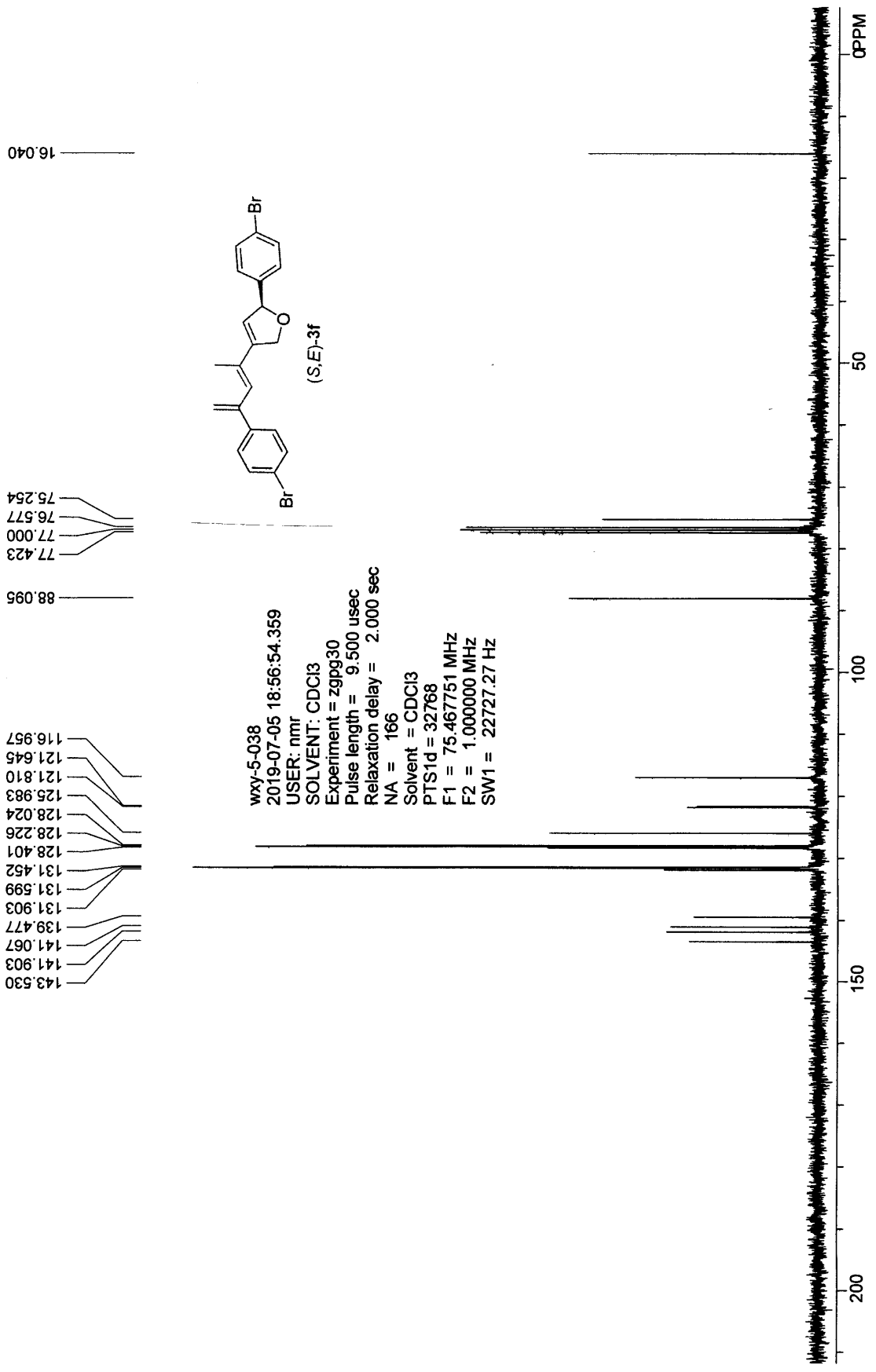
分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		11.278	128941.070	2447323.250	50.0950
2		12.350	117785.195	2438037.750	49.9050
总计			246726.266	4885361.000	100.0000


wxy-5-019-1
2019-06-25 18:56:26.187
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
(E)-8f
PPM
0.000
2.520
6.98
5.98
2.04
1.04
1.00
0.98
6.918
7.098
7.261
7.518
7.547
7.556
7.577
7.586
7.602
7.630
7.773
7.828
7.857


wxy-5-019-1
2019-06-25 19:15:41.890
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 303
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
(E)-8f
Br
O
H
O
Br
190.317
154.423
146.628
142.427
138.245
131.958
131.802
130.607
129.687
128.915
127.491
125.524
122.031
118.593
102.728
77.423
77.000
76.577
17.575
PPM
200
150
100
50
0


7.486
7.480
7.458
7.439
7.433
7.424
7.248
7.241
7.224
7.219
7.201
7.196
5.968
5.876
5.868
5.854
5.630
5.628
5.212
5.117
5.111
5.100
5.094
5.074
4.990
4.984
4.979
4.973
4.946
4.941
1.893
1.890
-0.000
wxy-5-038
2019-07-05 18:46:27.187
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
Br
Br
O
(S,E)-3f
4.05
4.02
1.98
1.01
1.00
1.00
0.99
0.99
3.02
8
7
6
5
4
3
2
1
PPM


16.040
75.254
76.577
77.000
77.423
88.095
116.957
121.645
121.810
125.983
128.024
128.226
128.401
131.452
131.599
131.903
139.477
141.067
141.903
143.530
wxy-5-038
2019-07-05 18:56:54.359
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
Br
Br
O
(S,E)-3f
200
150
100
50
0PPM

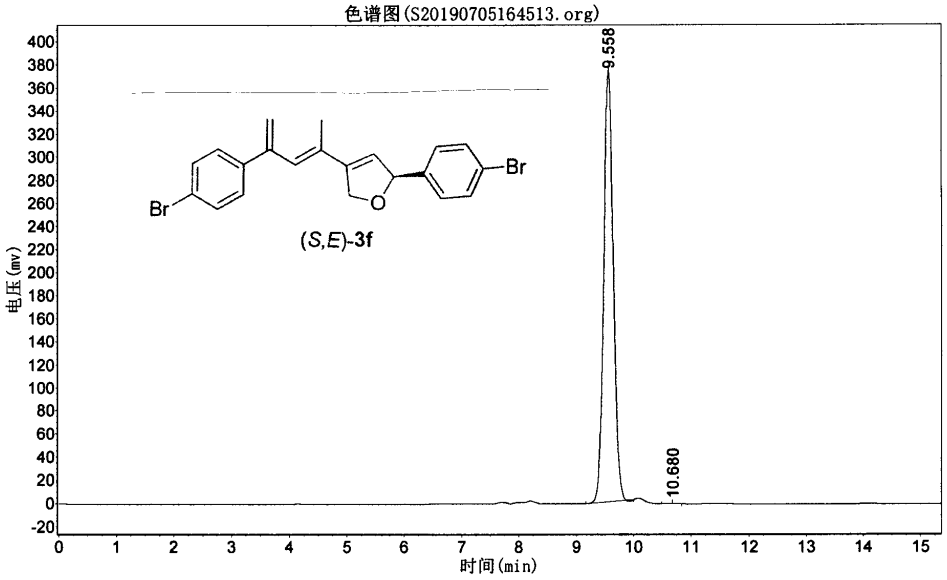
WXY-5-038(Chiral)

实验时间：2019-07-05,16:45:13
谱图文件:D:\浙大智达\N2000\样品\S20190705164513.org
方法文件:D:\浙大智达\N2000\djx.mtd

实验者：wxy
报告时间：2019-07-05,17:09:05
积分方法:面积归一法

实验内容简介：
IA,n-hexane/i-PrOH = 80/20,0.7,254

色谱图(S20190705164513.org)



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		9.558	372756.344	4288745.000	99.9219
2		10.680	310.067	3353.850	0.0781
总计			373066.411	4292098.850	100.0000

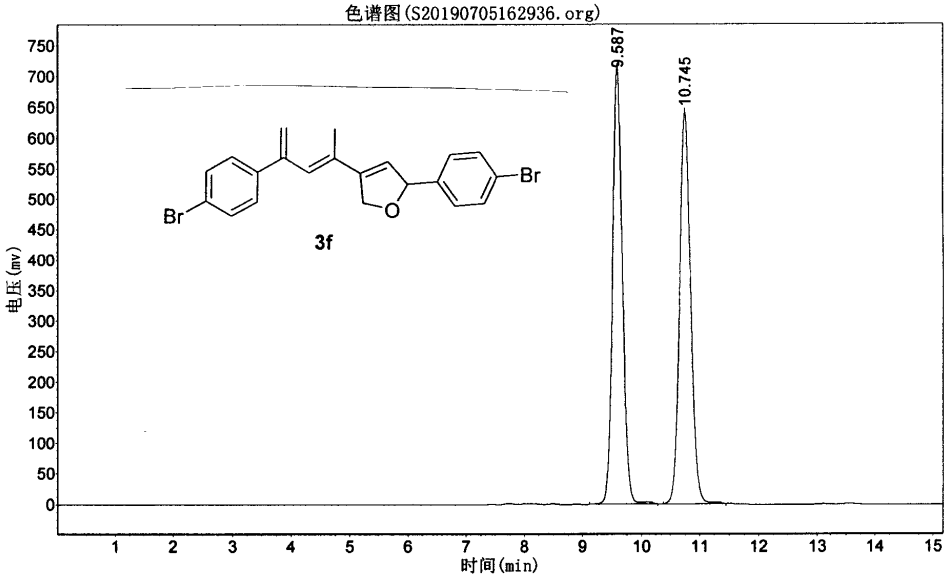
wxy-5-037(racemic)

实验时间：2019-07-05,16:29:36
谱图文件:D:\浙大智达\N2000\样品\S20190705162936.org
方法文件:D:\浙大智达\N2000\djx.mtd

实验者：wxy
报告时间：2019-07-05,16:47:41
积分方法:面积归一法

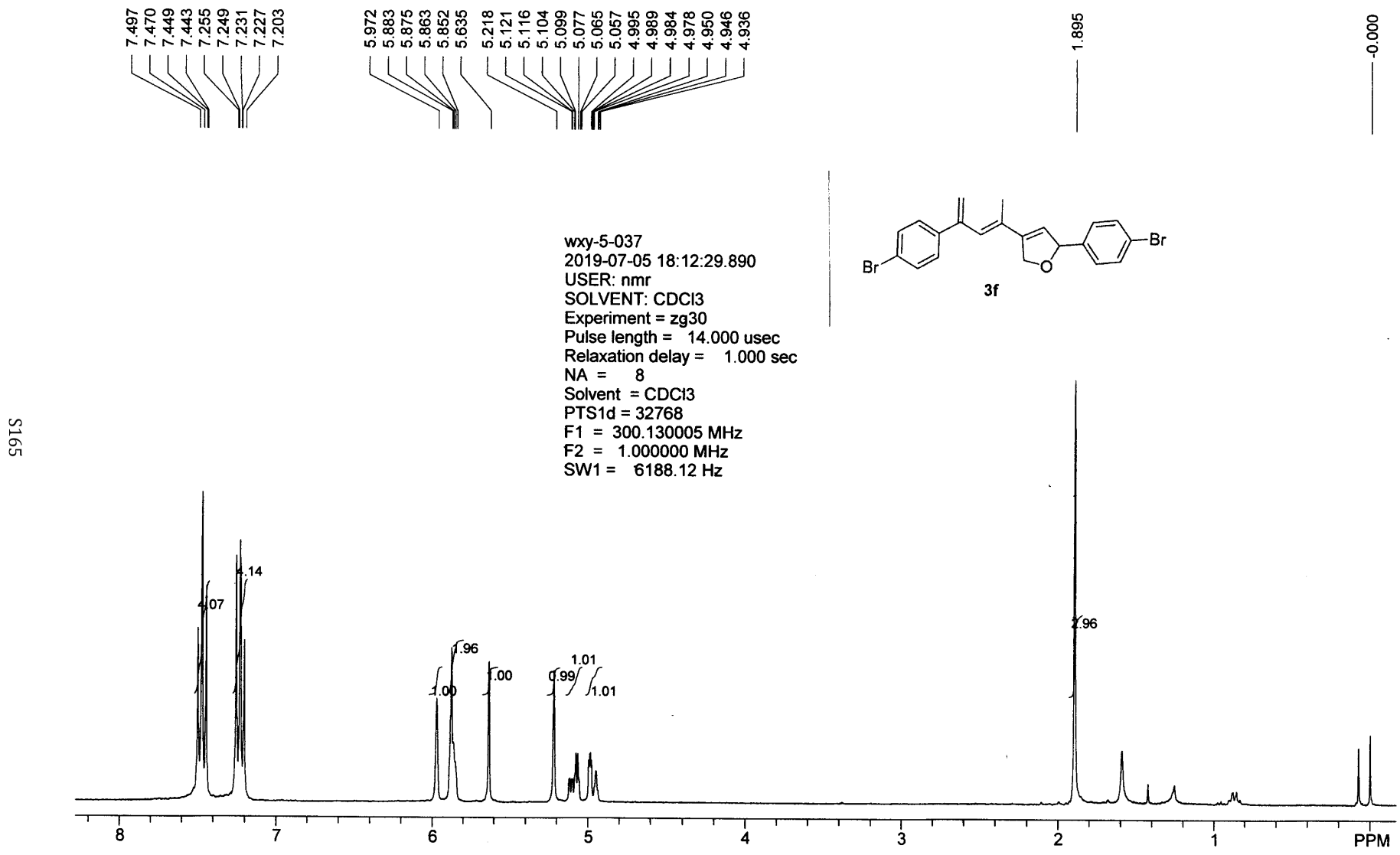
实验内容简介：
IA,n-hexane/i-PrOH = 90/10,0.7,254

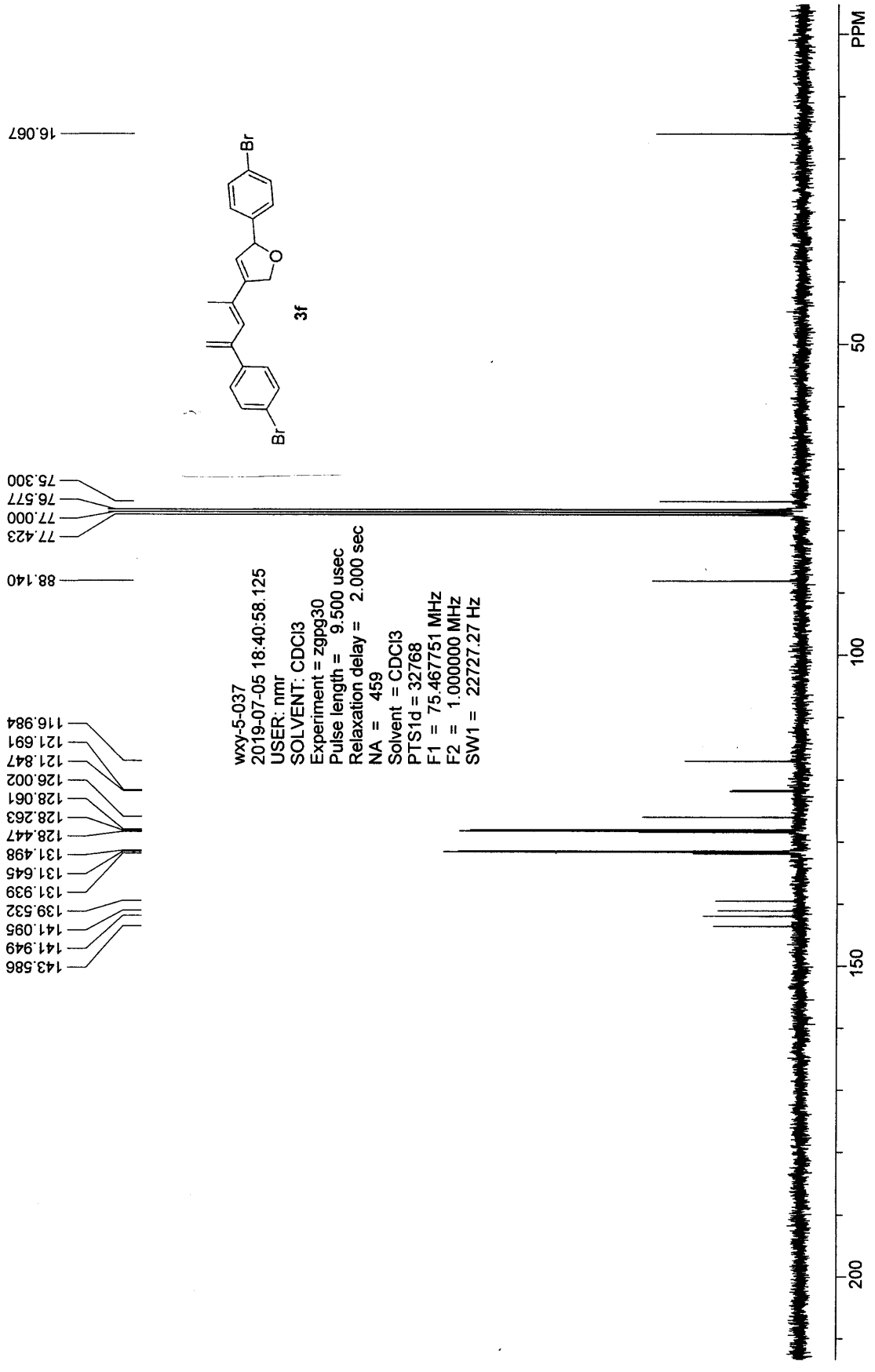
色谱图(S20190705162936.org)

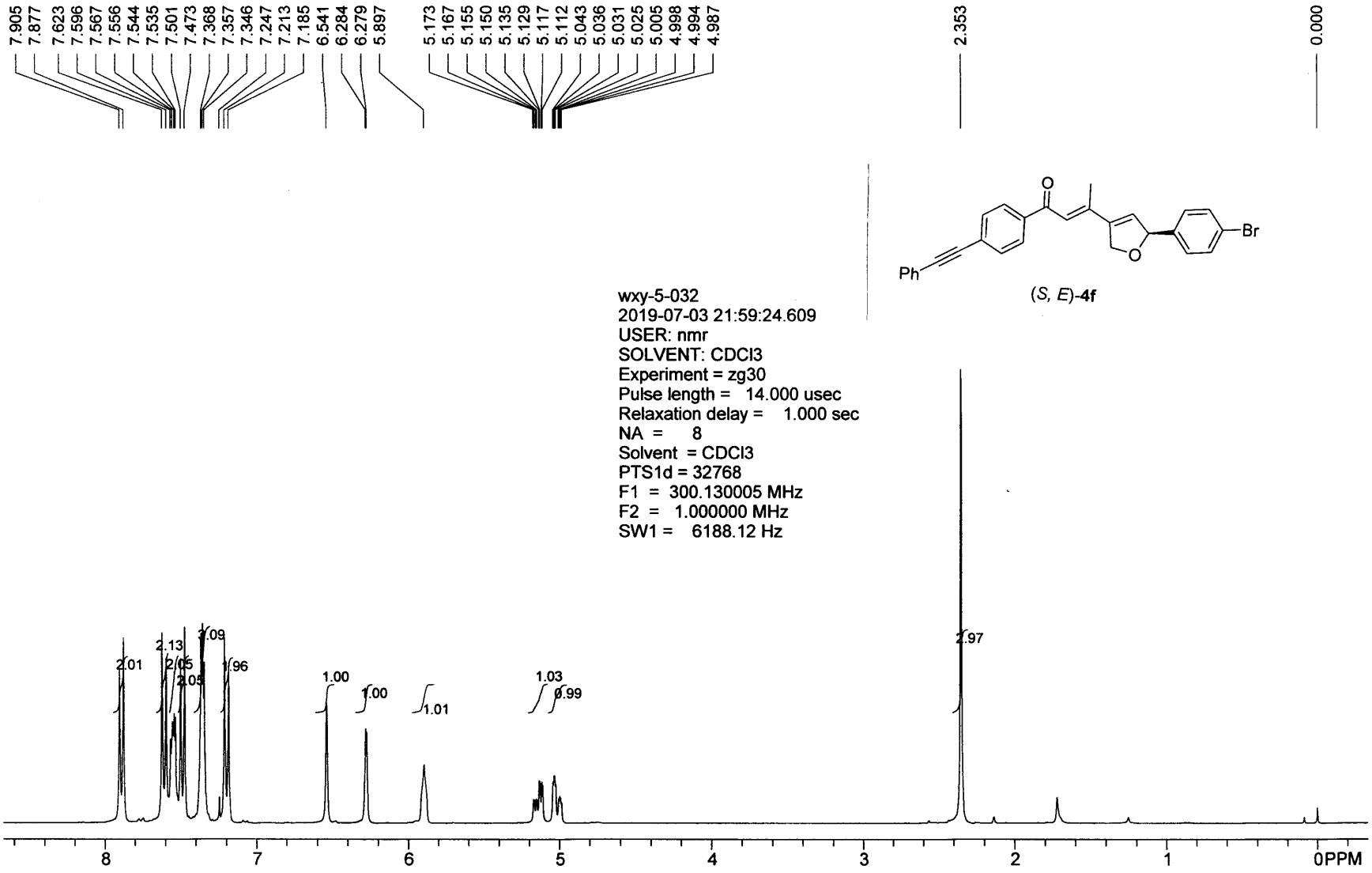


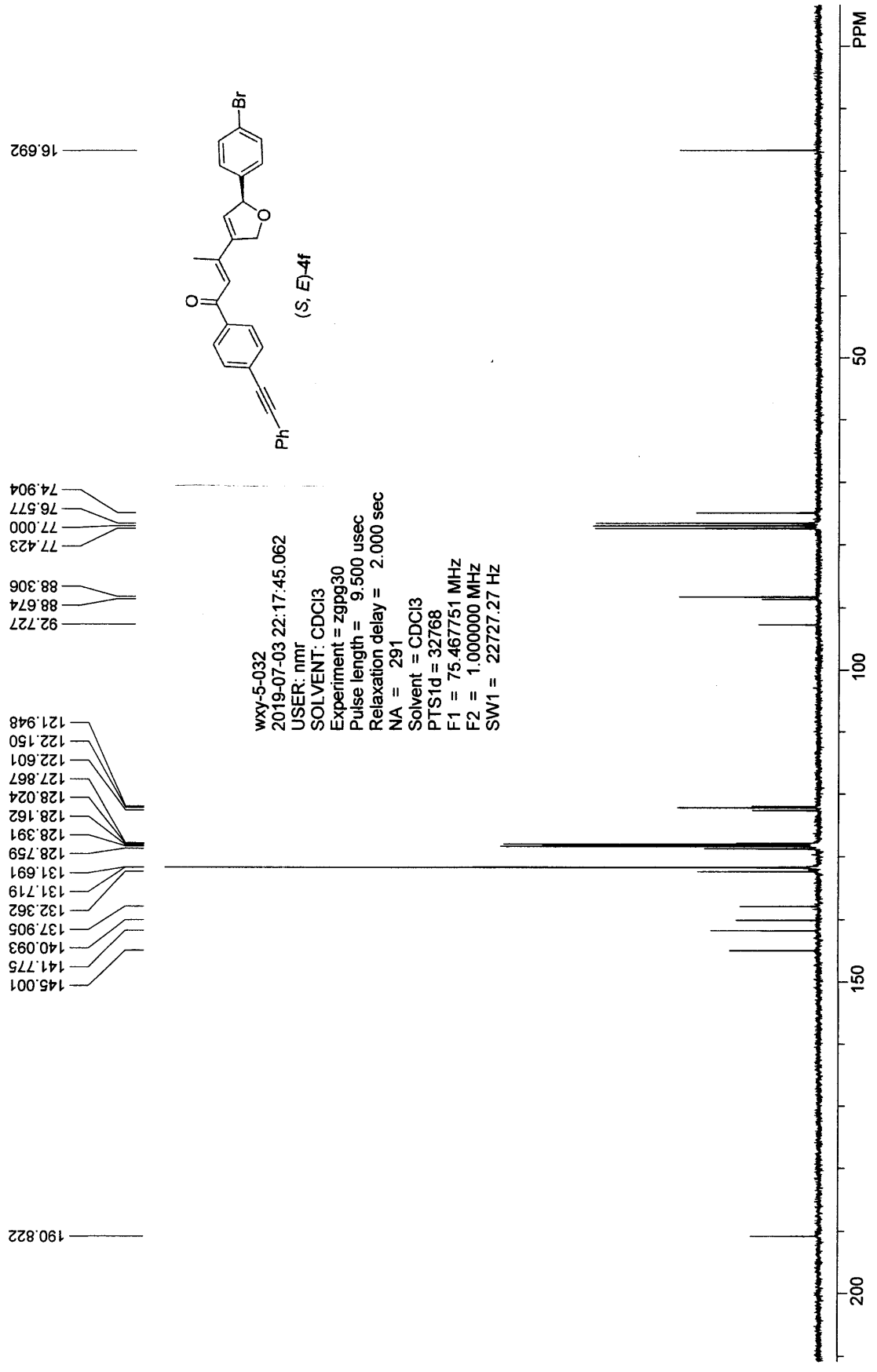
分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		9.587	713513.500	8305324.500	49.8856
2		10.745	640357.125	8343432.000	50.1144
总计			1353870.625	16648756.500	100.0000


7.497
7.470
7.449
7.443
7.255
7.249
7.231
7.227
7.203
5.972
5.883
5.875
5.863
5.852
5.635
5.218
5.121
5.116
5.104
5.099
5.077
5.065
5.057
4.995
4.989
4.984
4.978
4.950
4.946
4.936
1.895
-0.000
wxy-5-037
2019-07-05 18:12:29.890
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
Br
Br
O
3f
4.07
4.14
1.00
1.96
1.00
0.99
1.01
1.01
2.96
8
7
6
5
4
3
2
1
PPM


16.067
75.300
76.577
77.000
77.423
88.140
116.984
121.691
121.847
126.002
128.061
128.263
128.447
131.498
131.645
131.939
139.532
141.095
141.949
143.586
wxy-5-037
2019-07-05 18:40:58.125
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 459
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
Br
Br
O
3f
200
150
100
50
PPM


7.905
7.877
7.623
7.596
7.567
7.556
7.544
7.535
7.501
7.473
7.368
7.357
7.346
7.247
7.213
7.185
6.541
6.284
6.279
5.897
5.173
5.167
5.155
5.150
5.135
5.129
5.117
5.112
5.043
5.036
5.031
5.025
5.005
4.998
4.994
4.987
2.353
0.000
O
Br
Ph
O
(S, E)-4f
wxy-5-032
2019-07-03 21:59:24.609
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
2.13
2.05
2.05
2.09
1.96
1.00
1.00
1.01
1.03
0.99
2.97
8
7
6
5
4
3
2
1
0PPM


16.692
74.904
76.577
77.000
77.423
88.306
88.674
92.727
121.948
122.150
122.601
127.867
128.024
128.162
128.391
128.759
131.691
131.719
132.362
137.905
140.093
141.775
145.001
190.822
wxy-5-032
2019-07-03 22:17:45.062
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 291
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
(S, E)-4f
Br
O
O
Ph
PPM
200
150
100
50

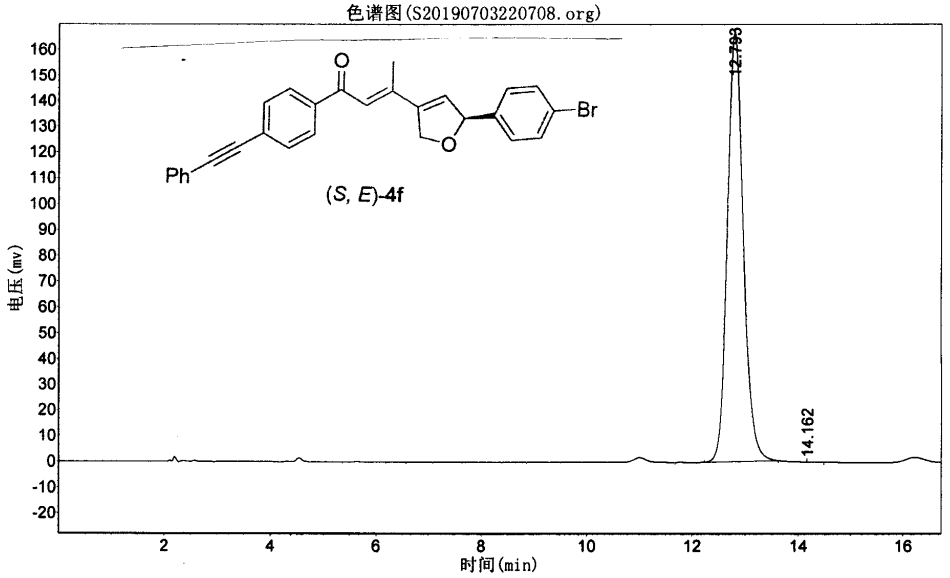
wxy-5-032(Chiral)

实验时间：2019-07-03,22:07:08
谱图文件:D:\浙大智达\N2000\样品\S20190703220708.org
方法文件:D:\浙大智达\N2000\djx.mtd

实验者：wxy
报告时间：2019-07-04,9:57:10
积分方法:面积归一法

实验内容简介：
IA,n-hexane/i-PrOH = 90/10,1.3,254

色谱图(S20190703220708.org)



分析结果表

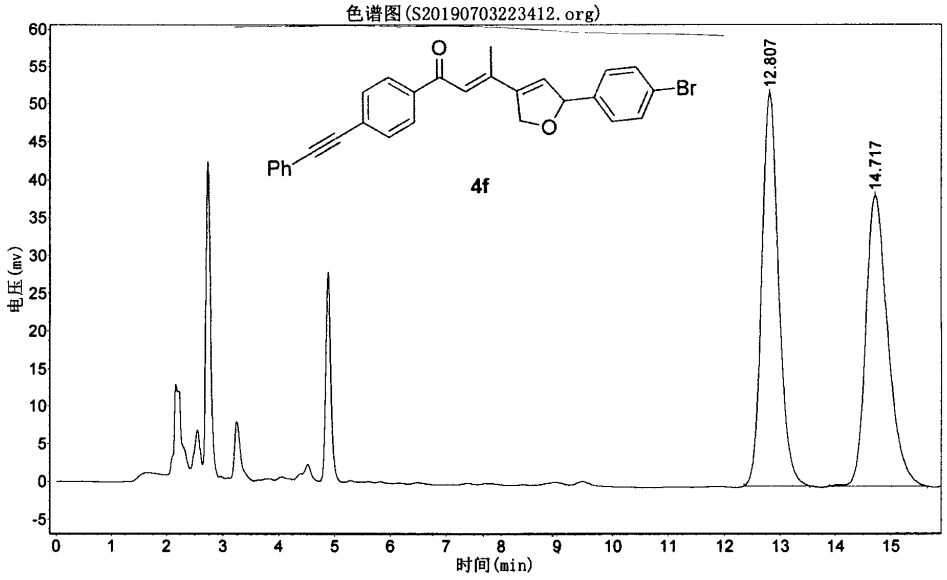
峰号	峰名	保留时间	峰高	峰面积	含量
1		12.793	167018.141	3491009.250	100.0034
2		14.162	2.522	-119.200	-0.0034
总计			167020.663	3490890.050	100.0000

wxy-5-026(racemic)

实验时间：2019-07-03,22:34:12
谱图文件:D:\浙大智达\N2000\样品\S20190703223412.org
方法文件:D:\浙大智达\N2000\djx.mtd

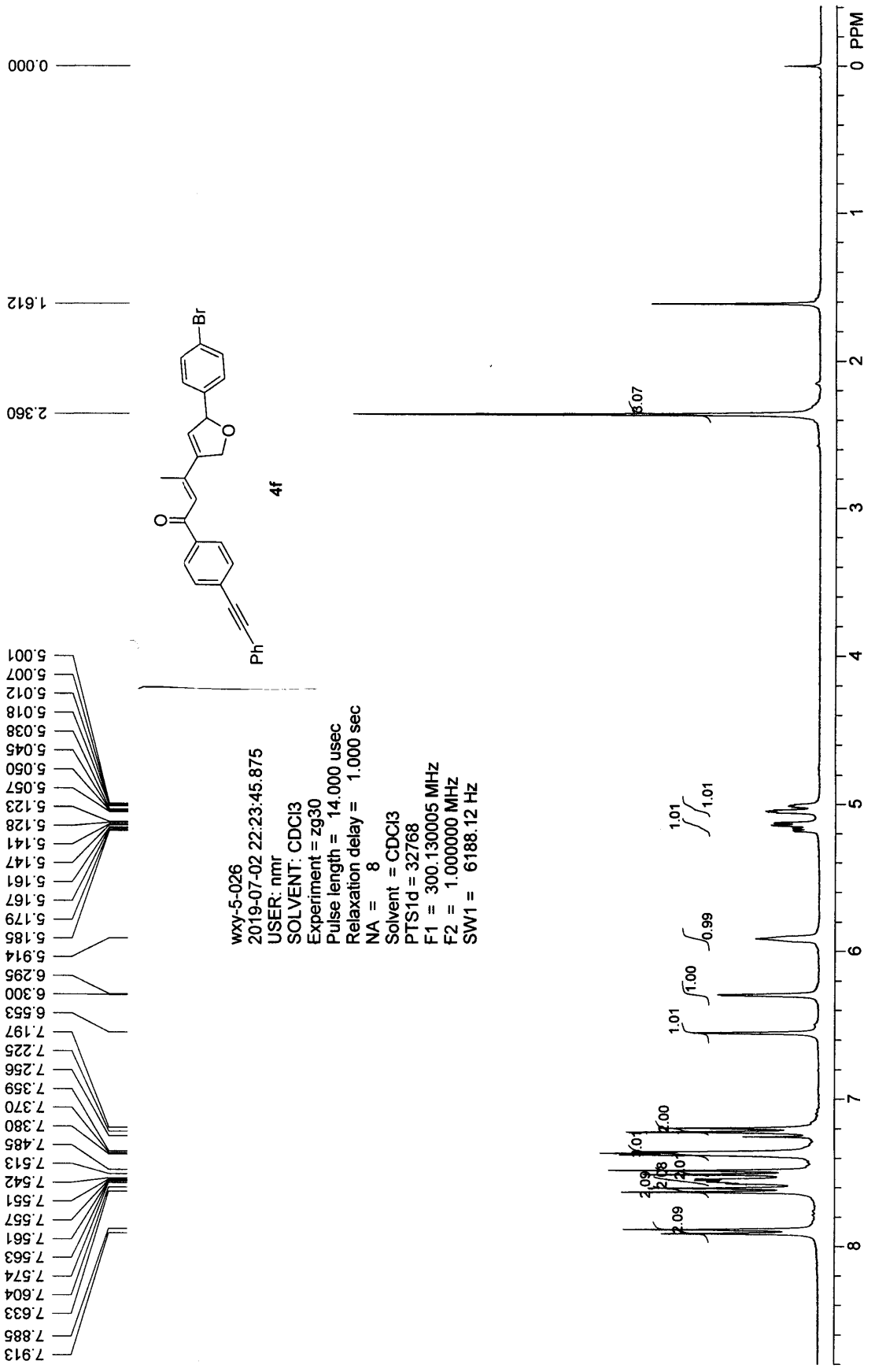
实验者：wxy
报告时间：2019-07-04,9:51:31
积分方法:面积归一法

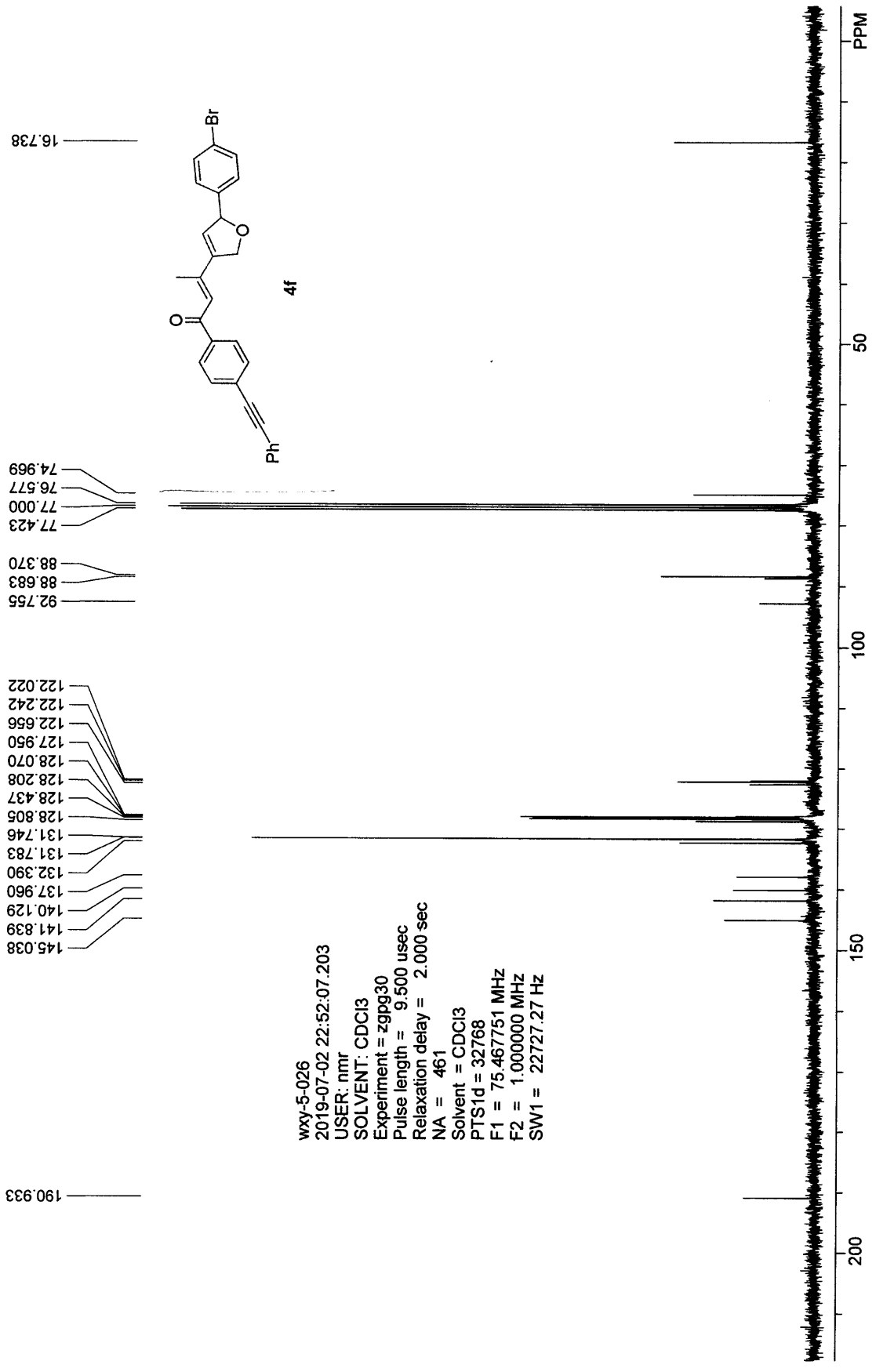
实验内容简介：
IA,n-hexane/i-PrOH = 90/10,1.3,254

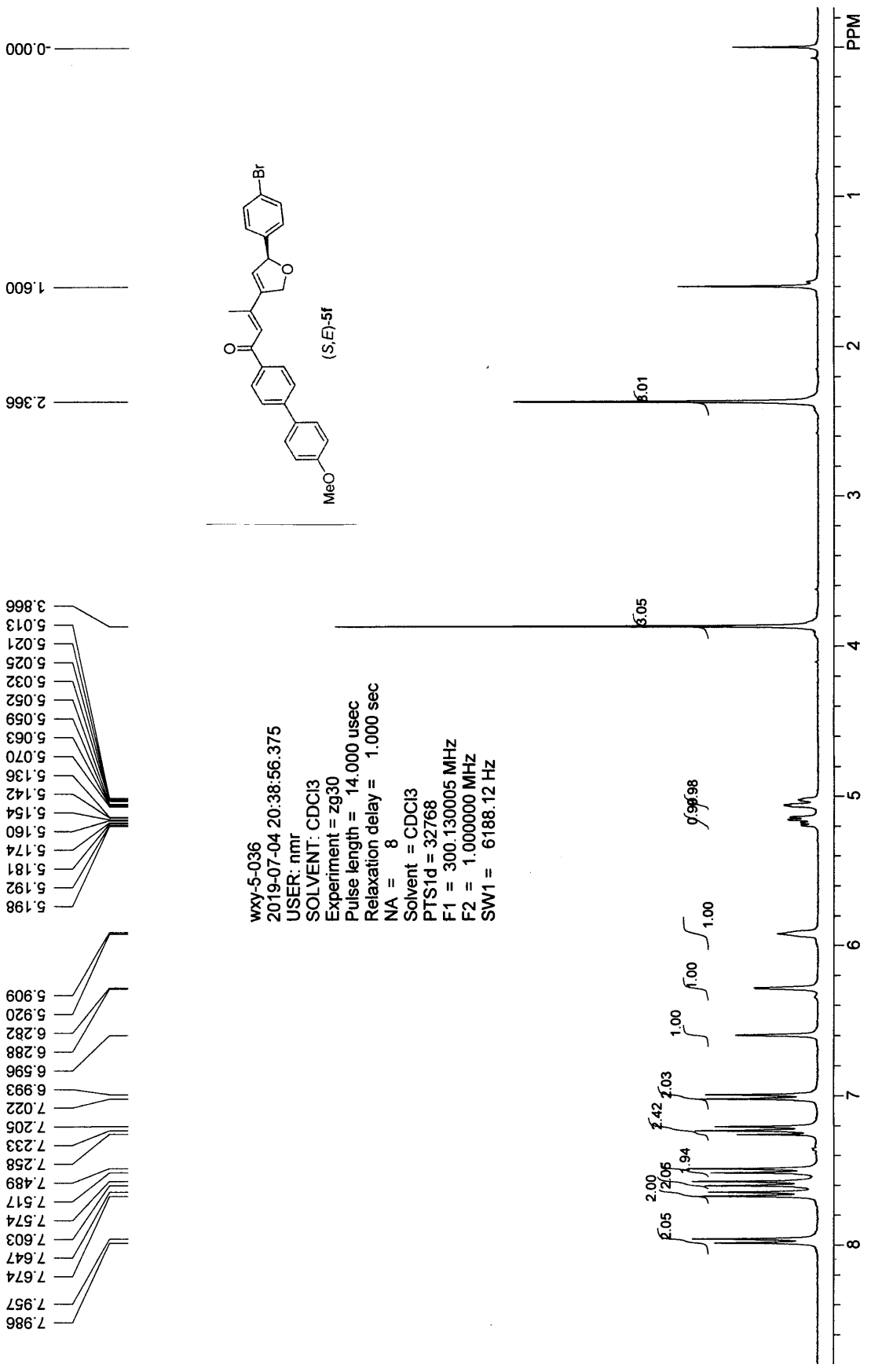


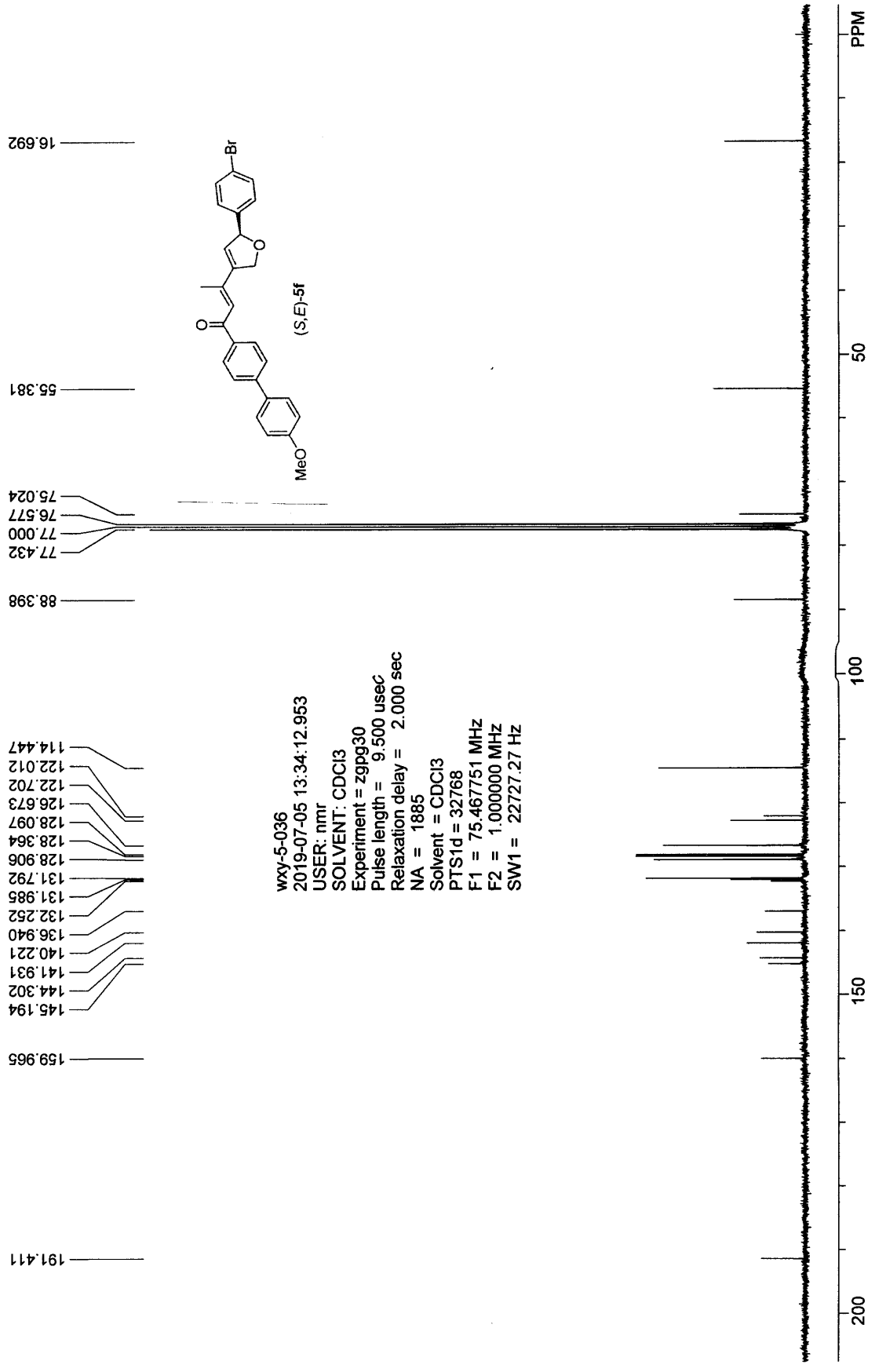
分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		12.807	52011.453	1089278.625	50.4702
2		14.717	38594.563	1068981.625	49.5298
总计			90606.016	2158260.250	100.0000


wxy-5-026
2019-07-02 22:23:45.875
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
4f
Br
Ph
O
O
0.000
1.612
2.360
3.07
1.01
1.00
0.99
1.01
1.01
2.00
1.01
2.09
2.08
2.10
2.09
0 PPM
1
2
3
4
5
6
7
8
5.001
5.007
5.012
5.018
5.038
5.045
5.050
5.057
5.123
5.128
5.141
5.147
5.161
5.167
5.179
5.185
5.914
6.295
6.300
6.553
7.197
7.225
7.256
7.359
7.370
7.380
7.485
7.513
7.542
7.551
7.557
7.561
7.563
7.574
7.604
7.633
7.885
7.913


wxy-5-026
2019-07-02 22:52:07.203
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 461
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
4f
Br
O
O
Ph
16.738
74.969
76.577
77.000
77.423
88.370
88.683
92.755
122.022
122.242
122.656
127.960
128.070
128.208
128.437
128.805
131.746
131.783
132.390
137.960
140.129
141.839
145.038
190.933
PPM
200
150
100
50


wxy-5-036
2019-07-04 20:38:56.375
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
(S,E)-5f
Br
MeO
O
PPM


wxy-5-036
2019-07-05 13:34:12.953
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 1885
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
(S,E)-5f
Br
MeO
O
191.411
159.965
145.194
144.302
141.931
140.221
136.940
132.252
131.985
131.792
128.906
128.364
128.097
126.673
122.702
122.012
114.447
88.398
77.432
77.000
76.577
75.024
55.381
16.692
PPM
200
150
100
50

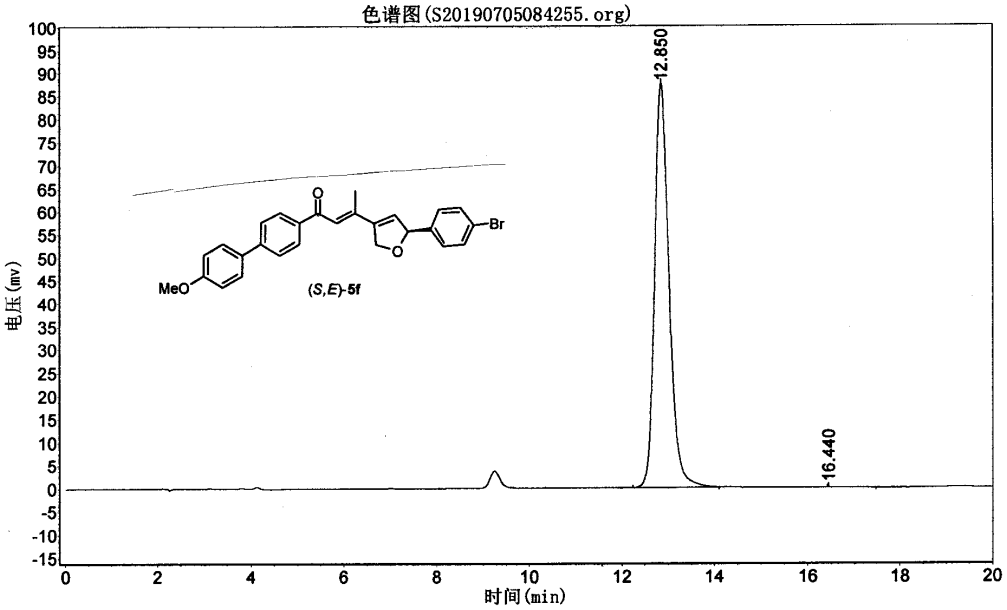
WXY-5-029(Chiral)

实验时间：2019-07-05,8:42:55
谱图文件:D:\浙大智达\N2000\样品\S20190705084255.org
方法文件:D:\浙大智达\N2000\djx.mtd

实验者：WXY
报告时间：2019-07-05,9:35:50
积分方法:面积归一法

实验内容简介：
IA,n-hexane/i-PrOH = 80/20,1.3,254

色谱图(S20190705084255.org)



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		12.850	87458.180	1886878.750	100.0077
2		16.440	8.189	-144.900	-0.0077
总计			87466.369	1886733.850	100.0000

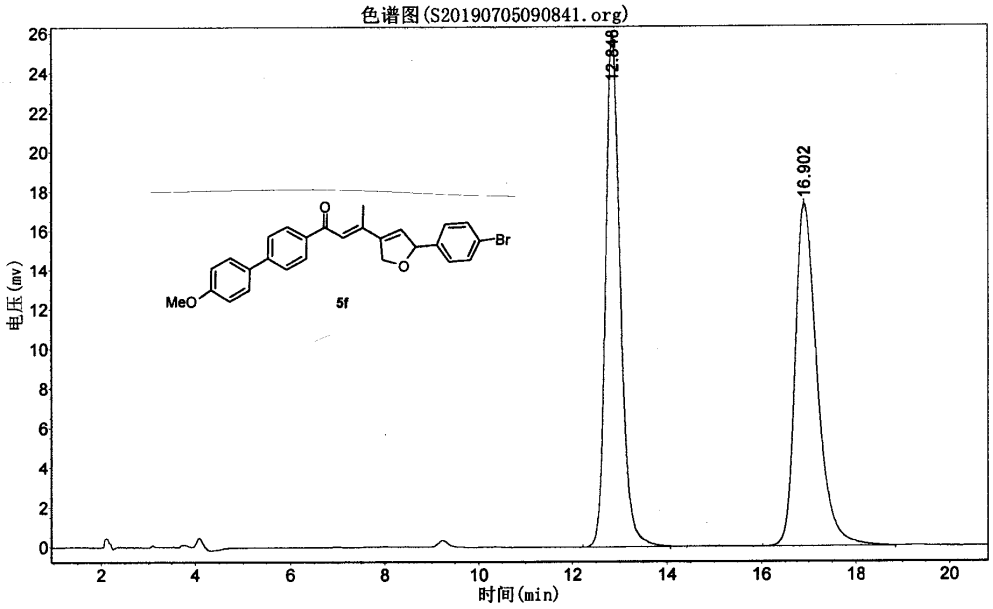
wxy-5-036(Racemic)

实验时间：2019-07-05,9:08:41
谱图文件:D:\浙大智达\N2000\样品\S20190705090841.org
方法文件:D:\浙大智达\N2000\djx.mtd

实验者：wxy
报告时间：2019-07-05,9:31:39
积分方法:面积归一法

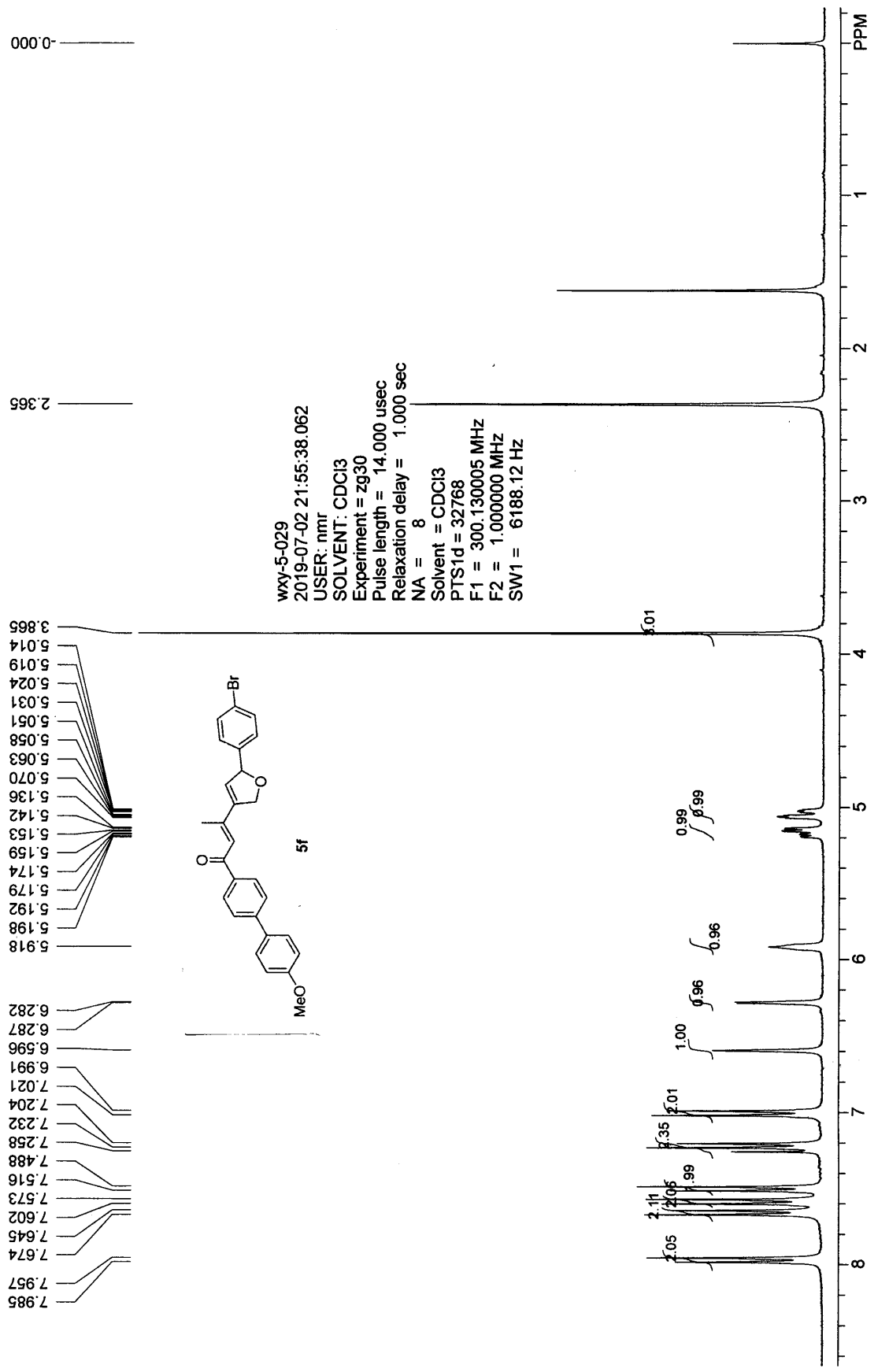
实验内容简介：
IA,n-hexane/i-PrOH = 80/20,1.3,254

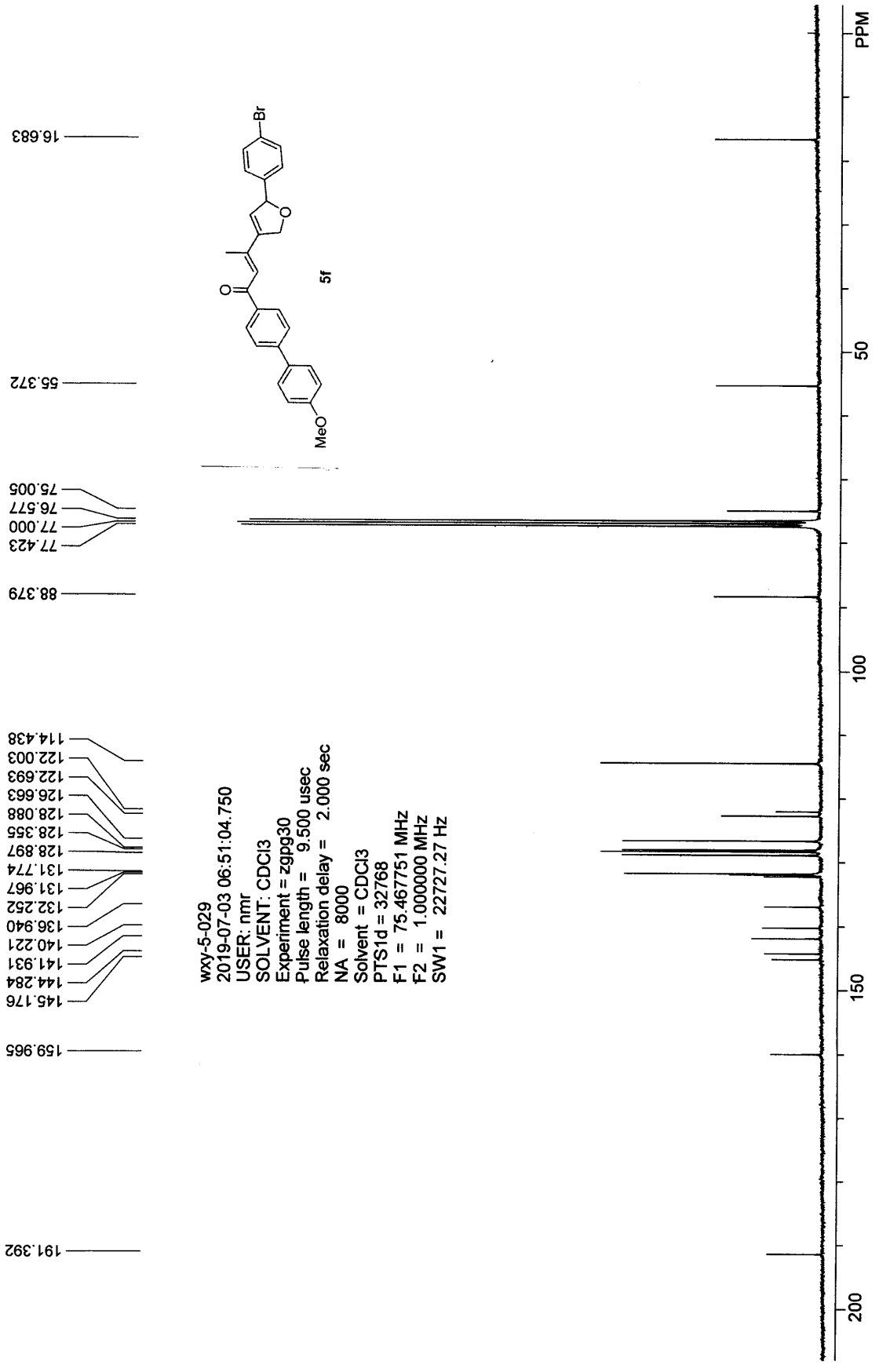
色谱图(S20190705090841.org)

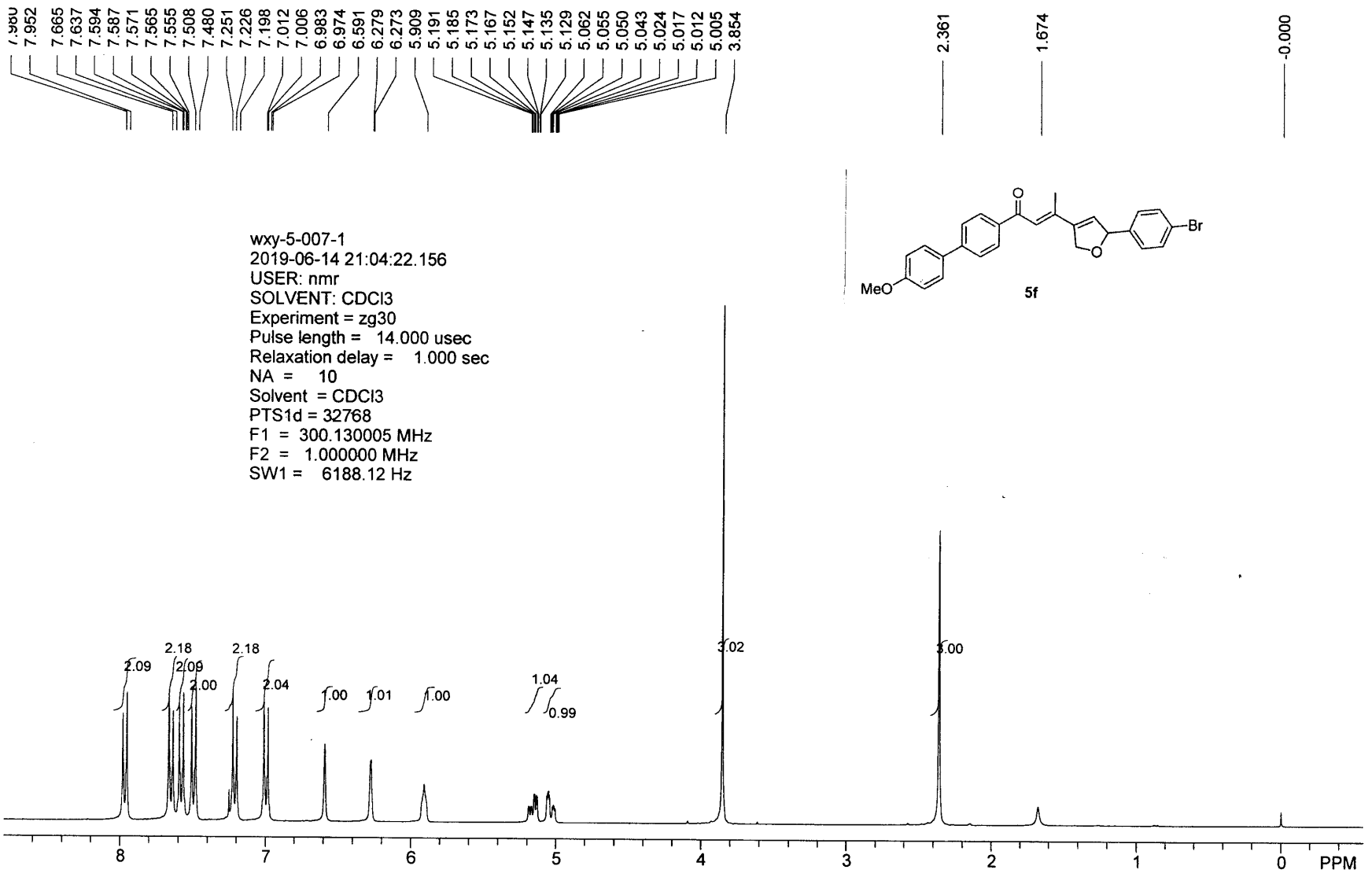


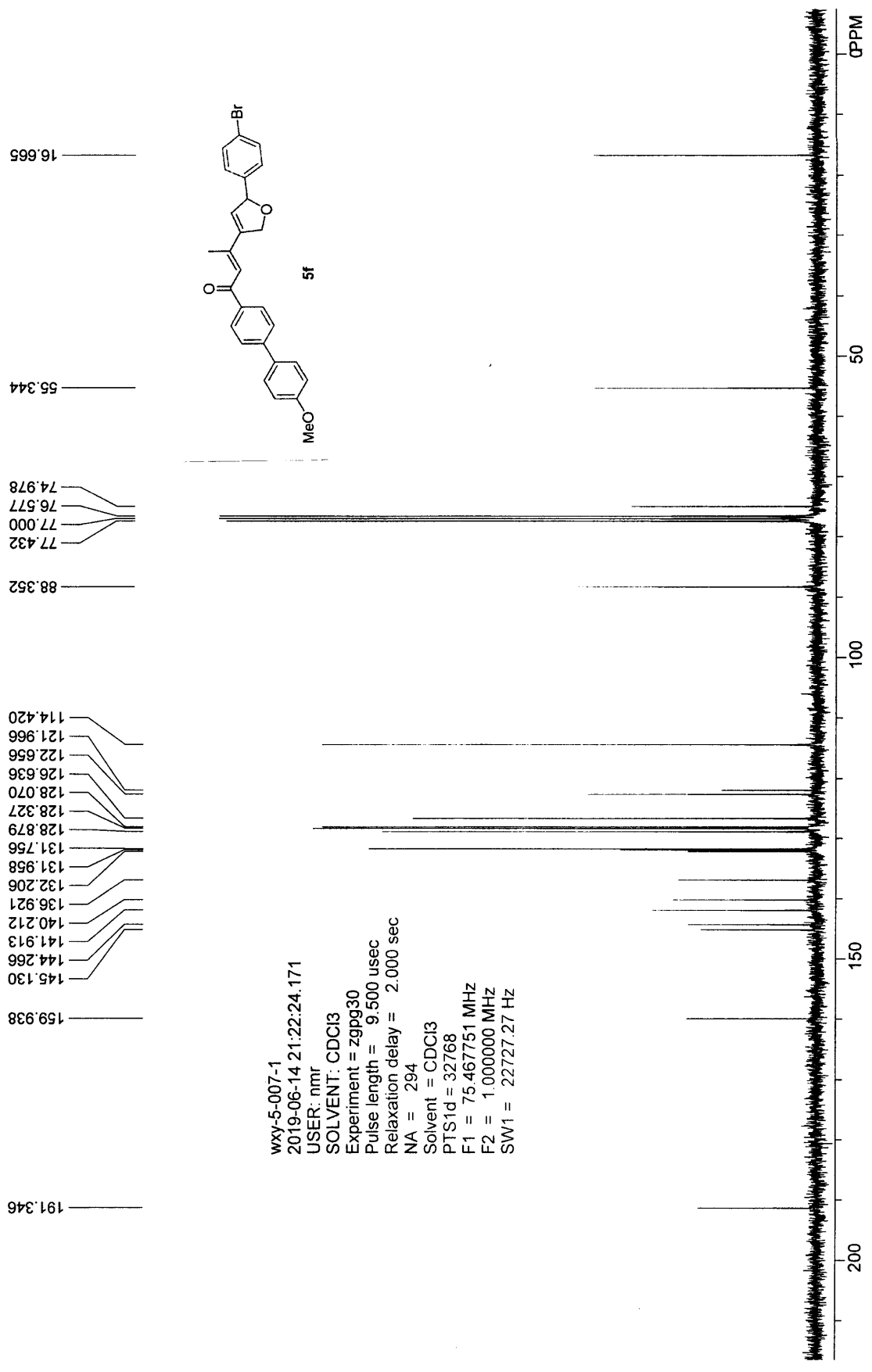
分析结果表

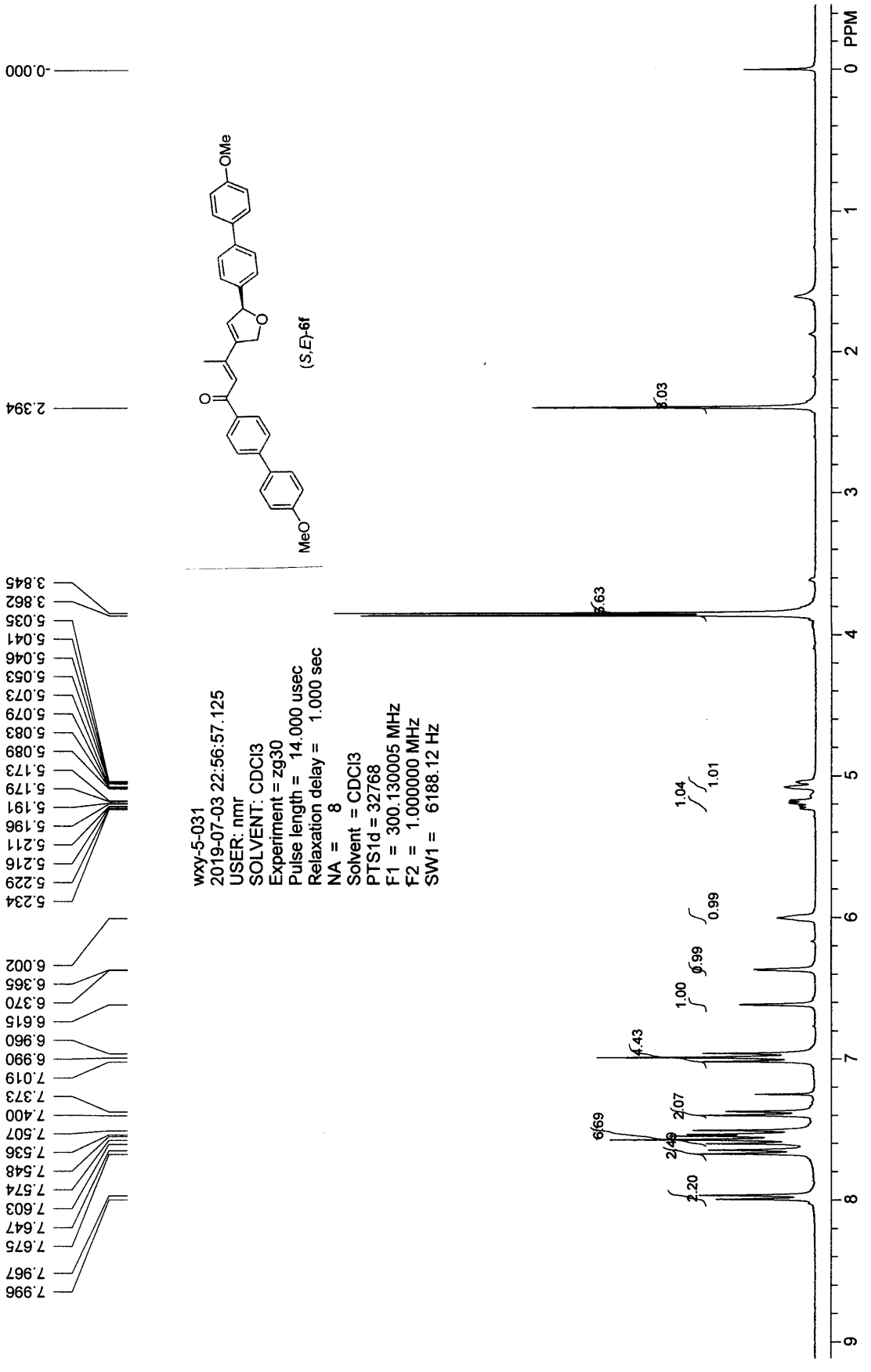
峰号	峰名	保留时间	峰高	峰面积	含量
1		12.848	25912.004	561805.438	50.1787
2		16.902	17321.164	557803.063	49.8213
总计			43233.168	1119608.500	100.0000

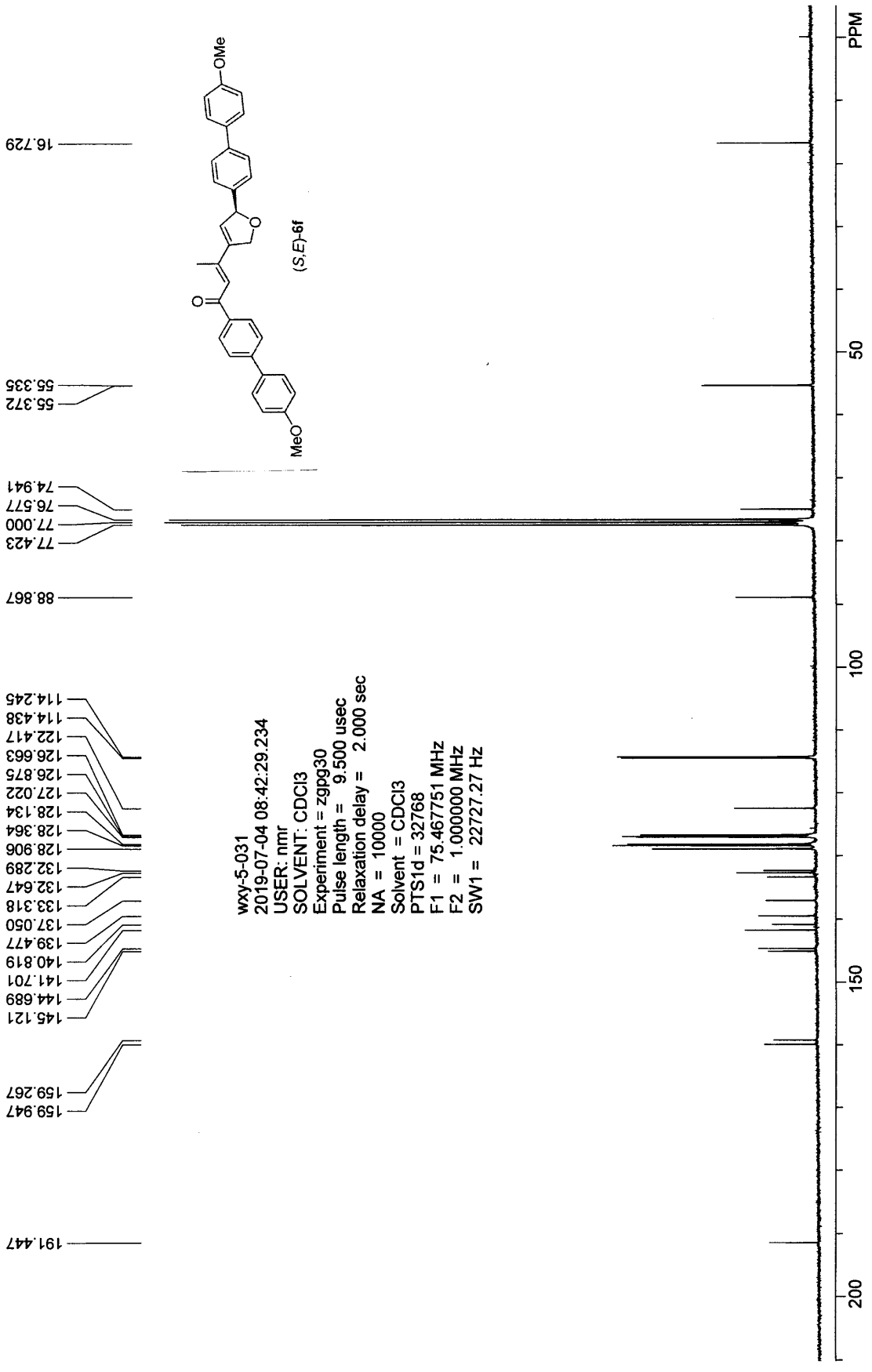

wxy-5-029
2019-07-02 21:55:38.062
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
5f
Br
MeO
O
PPM

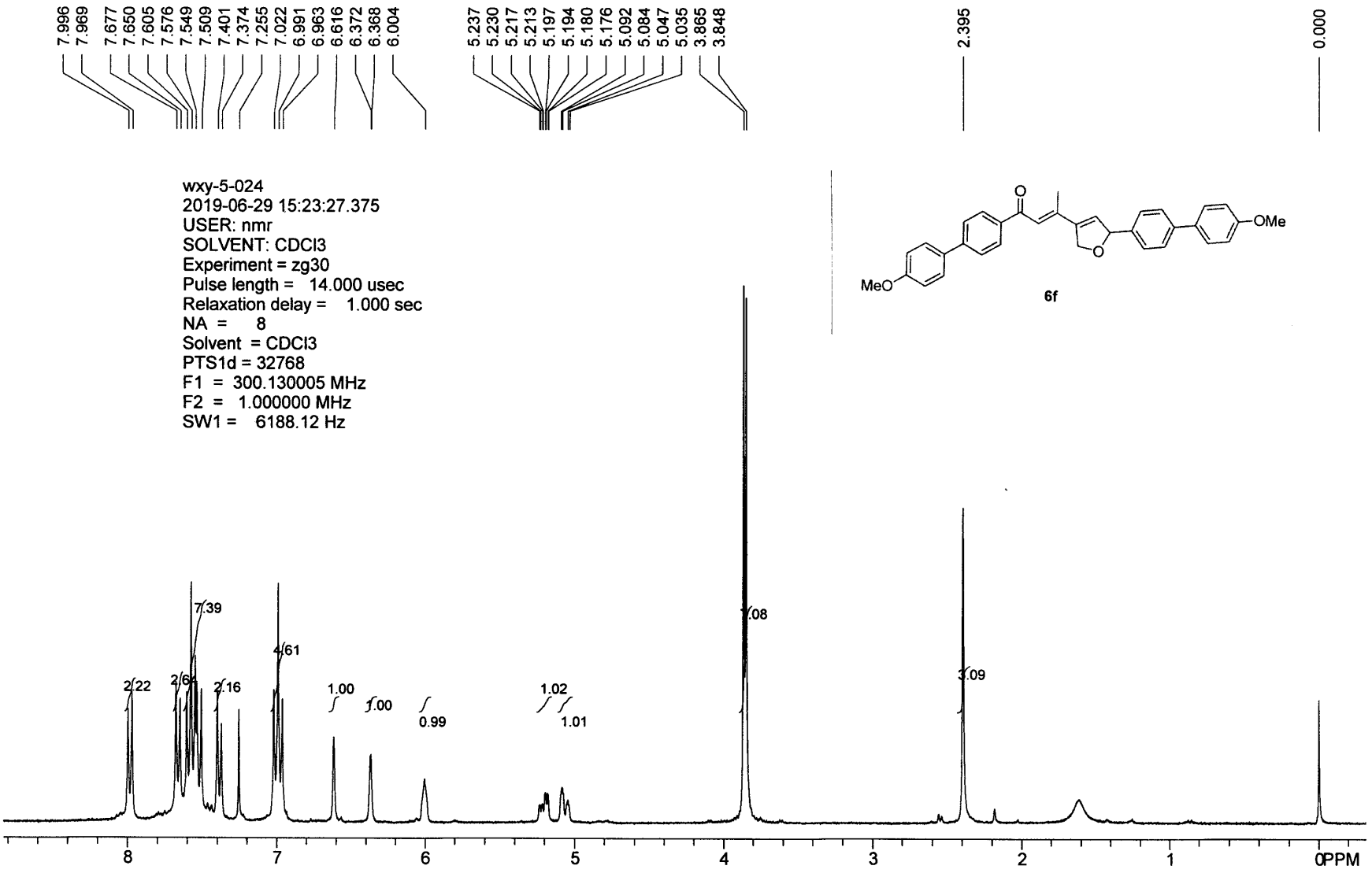

16.683
55.372
75.005
76.577
77.000
77.423
88.379
114.438
122.003
122.693
126.663
128.088
128.355
128.897
131.774
131.967
132.252
136.940
140.221
141.931
144.284
145.176
159.965
191.392
wxy-5-029
2019-07-03 06:51:04.750
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 8000
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
Br
O
O
MeO
5f
PPM
200
150
100
50

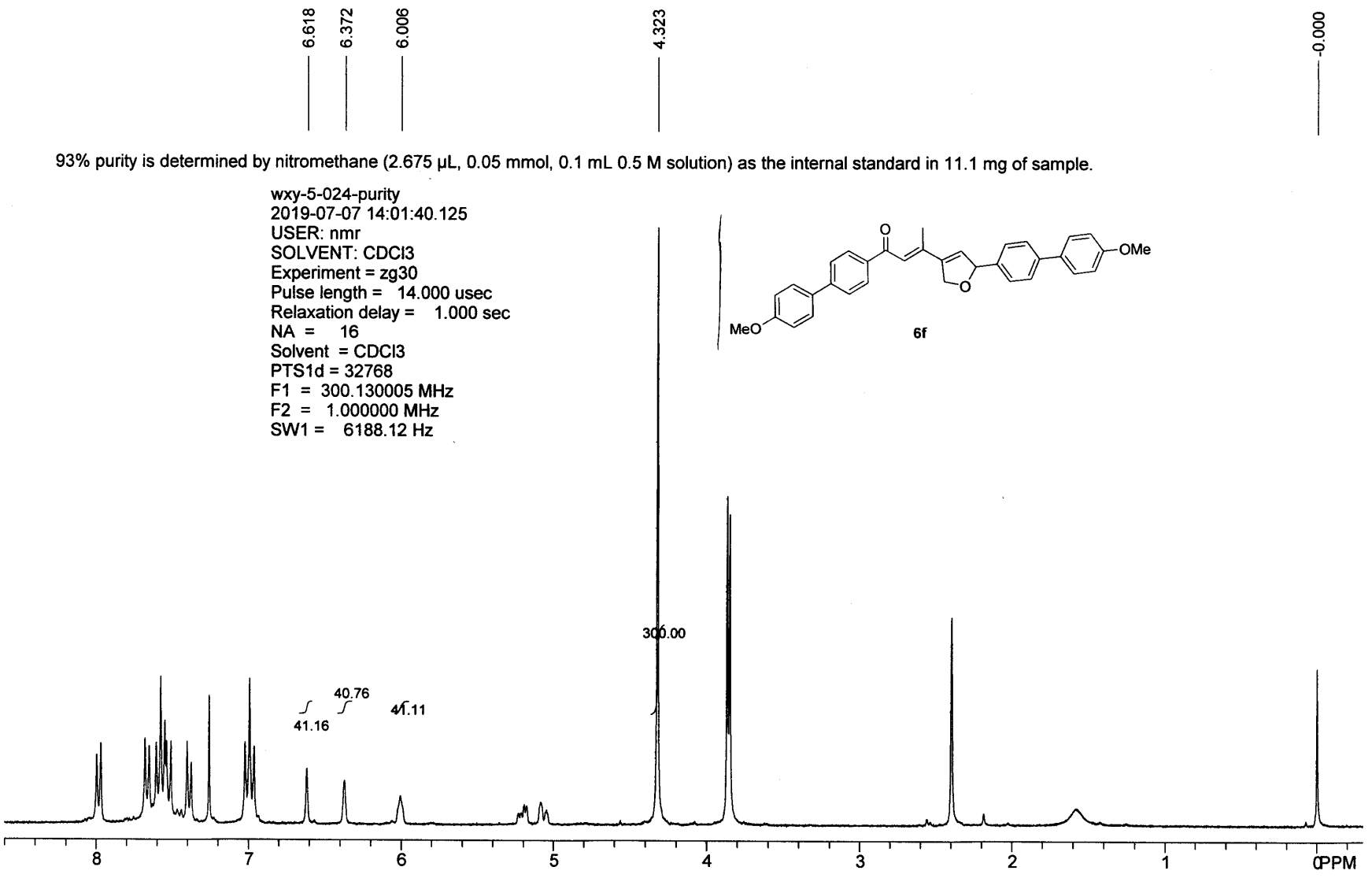

7.960
7.952
7.665
7.637
7.594
7.587
7.571
7.565
7.555
7.508
7.480
7.251
7.226
7.198
7.012
7.006
6.983
6.974
6.591
6.279
6.273
5.909
5.191
5.185
5.173
5.167
5.152
5.147
5.135
5.129
5.062
5.055
5.050
5.043
5.024
5.017
5.012
5.005
3.854
2.361
1.674
-0.000
wxy-5-007-1
2019-06-14 21:04:22.156
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 10
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
MeO
Br
5f
2.09
2.18
2.09
2.00
2.18
2.04
1.00
1.01
1.00
1.04
0.99
3.02
3.00
8
7
6
5
4
3
2
1
0
PPM

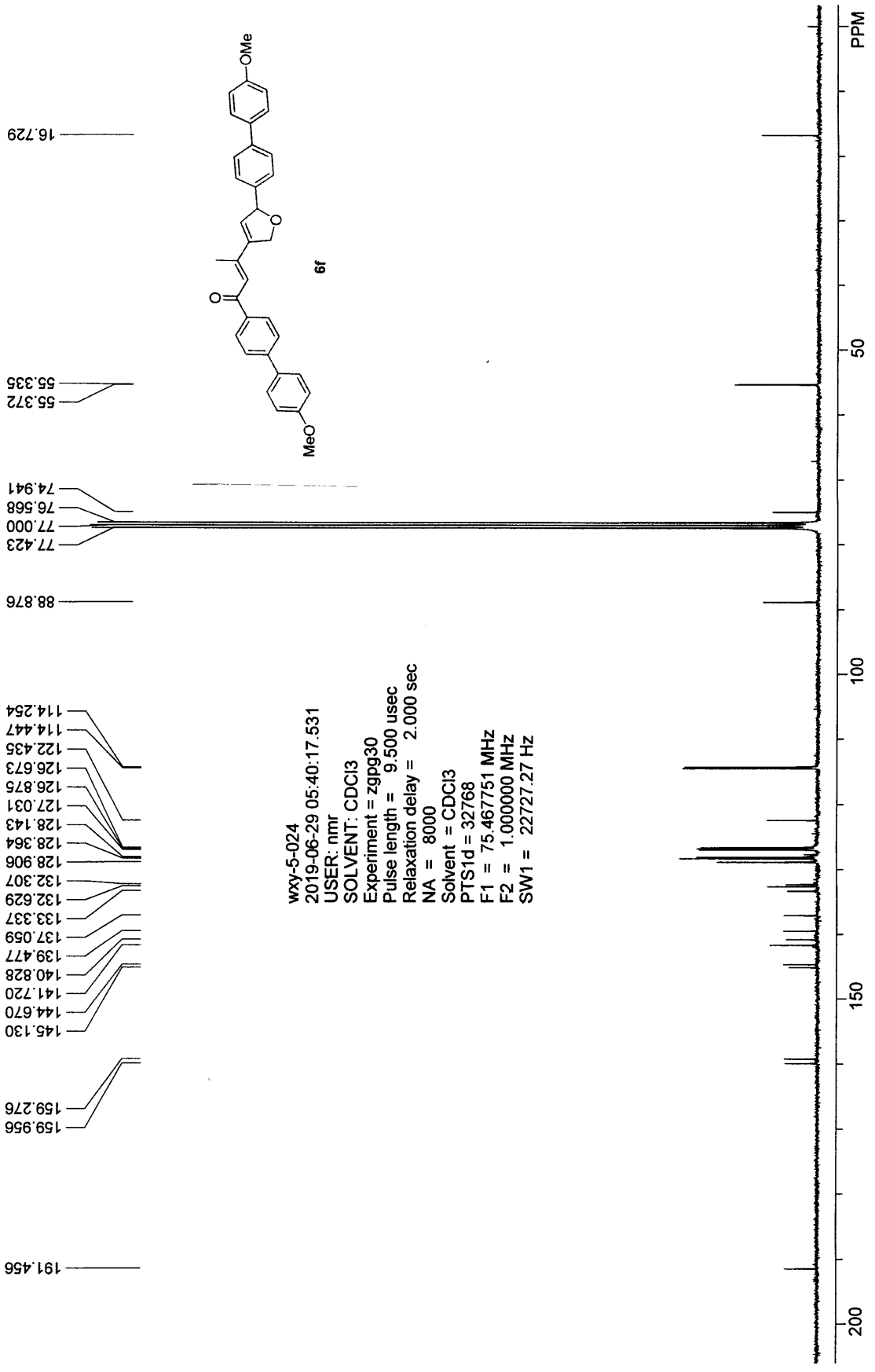

wxy-5-007-1
2019-06-14 21:22:24.171
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 294
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
191.346
159.938
145.130
144.266
141.913
140.212
136.921
132.206
131.958
131.756
128.879
128.327
128.070
126.636
122.656
121.966
114.420
88.352
77.432
77.000
76.577
74.978
55.344
16.665
Br
O
MeO
5f
200
150
100
50
0PPM

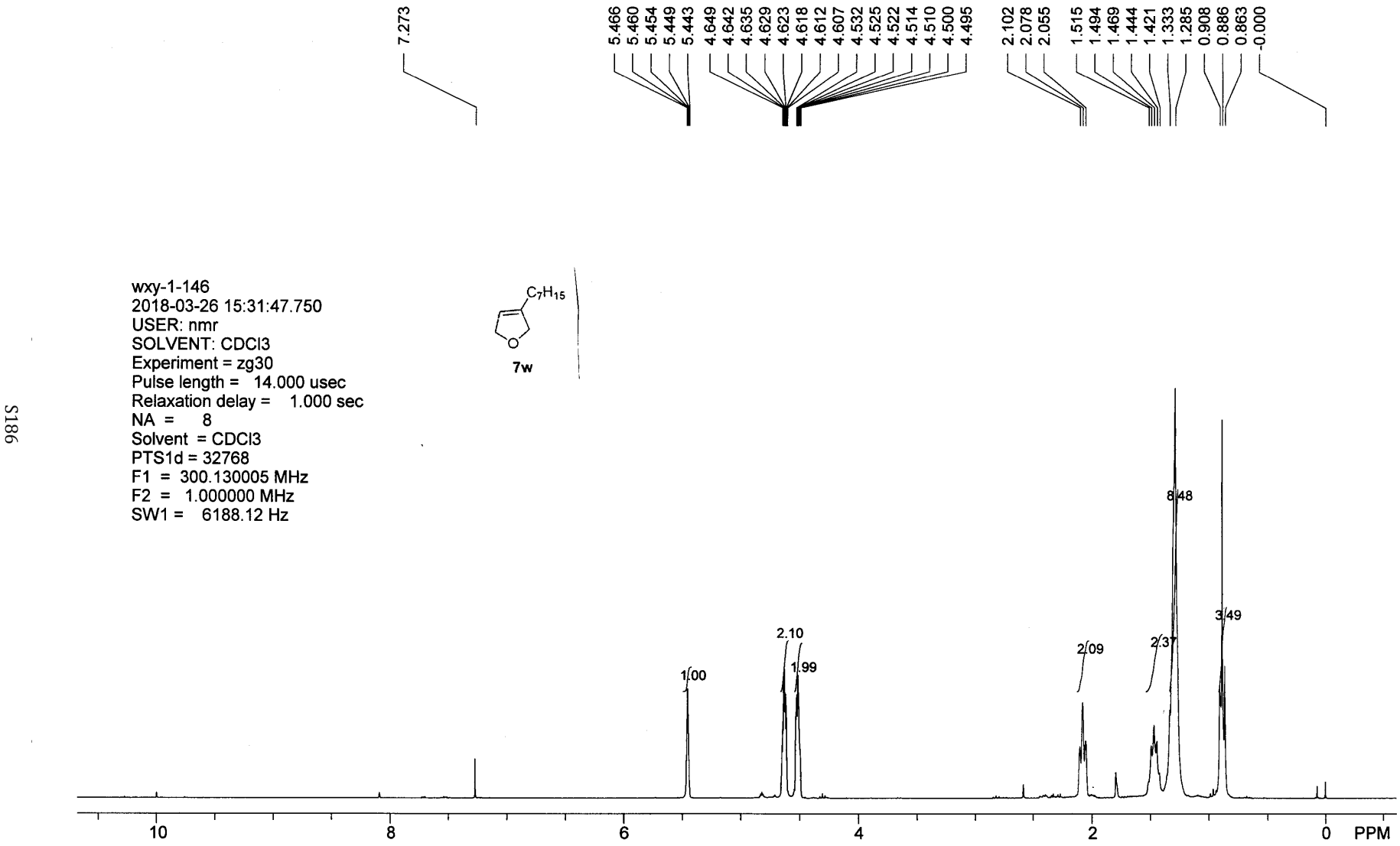

wxy-5-031
2019-07-03 22:56:57.125
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
f2 = 1.000000 MHz
SW1 = 6188.12 Hz
(S,E)-6f
OMe
MeO
PPM

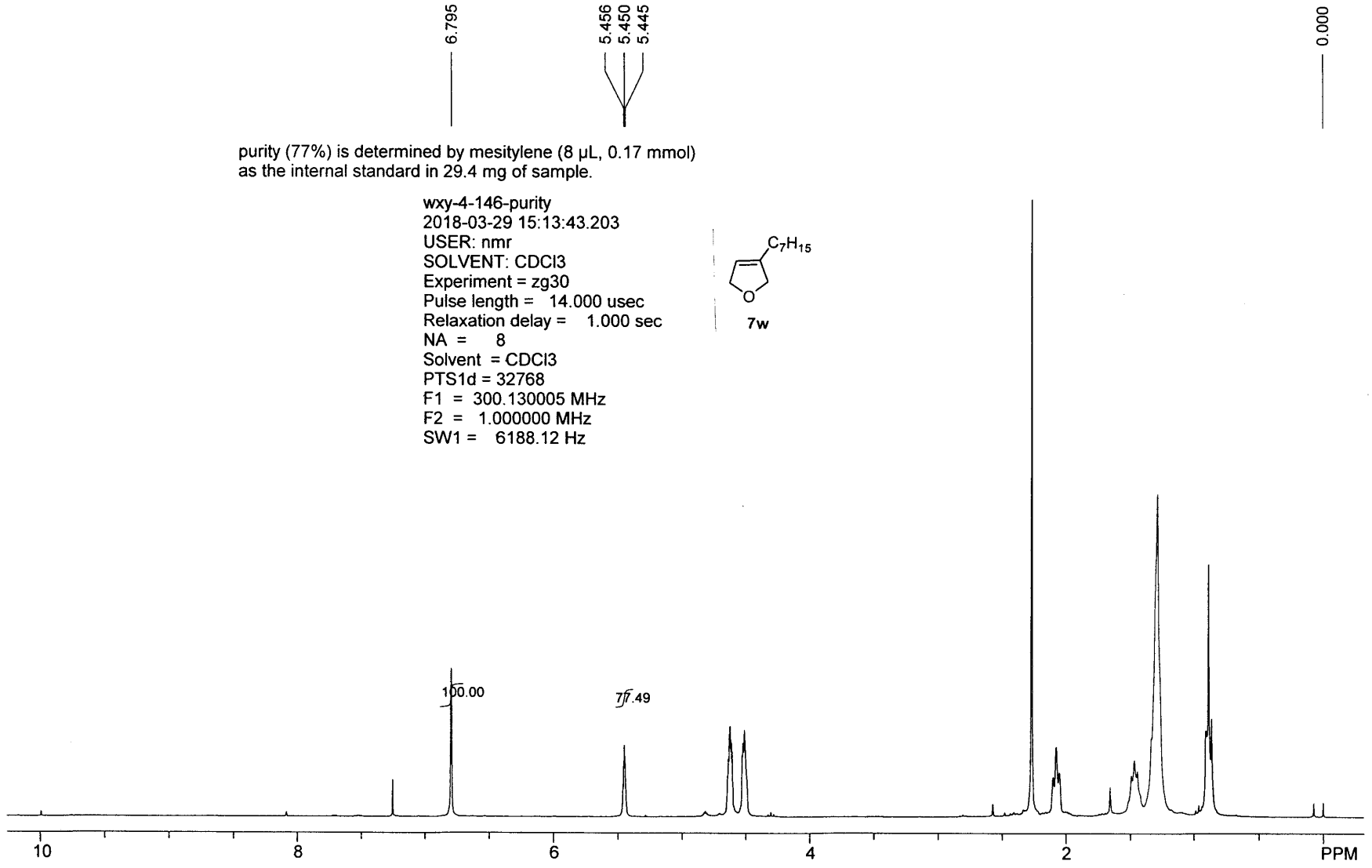

wxy-5-031
2019-07-04 08:42:29.234
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 10000
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
(S,E)-6f
OMe
MeO
O
O
16.729
55.335
55.372
74.941
76.577
77.000
77.423
88.867
114.245
114.438
122.417
126.663
126.875
127.022
128.134
128.364
128.906
132.289
132.647
133.318
137.050
139.477
140.819
141.701
144.689
145.121
159.267
159.947
191.447
PPM
200
150
100
50

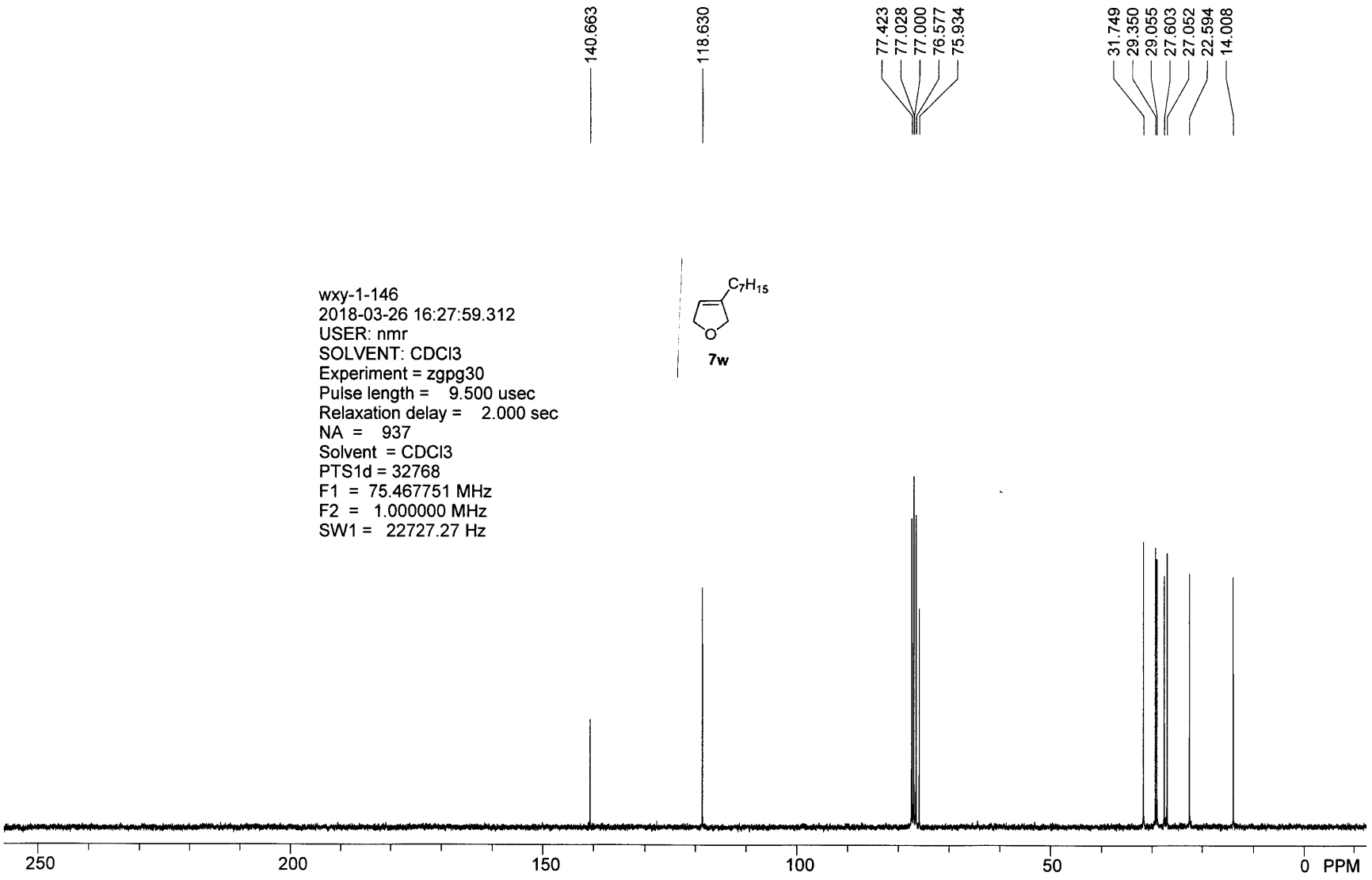

7.996
7.969
7.677
7.650
7.605
7.576
7.549
7.509
7.401
7.374
7.255
7.022
6.991
6.963
6.616
6.372
6.368
6.004
5.237
5.230
5.217
5.213
5.197
5.194
5.180
5.176
5.092
5.084
5.047
5.035
3.865
3.848
2.395
0.000
wxy-5-024
2019-06-29 15:23:27.375
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
MeO
OMe
6f
2.22
2.64
7.39
2.16
4.61
1.00
1.00
0.99
1.02
1.01
7.08
3.09
8
7
6
5
4
3
2
1
0PPM

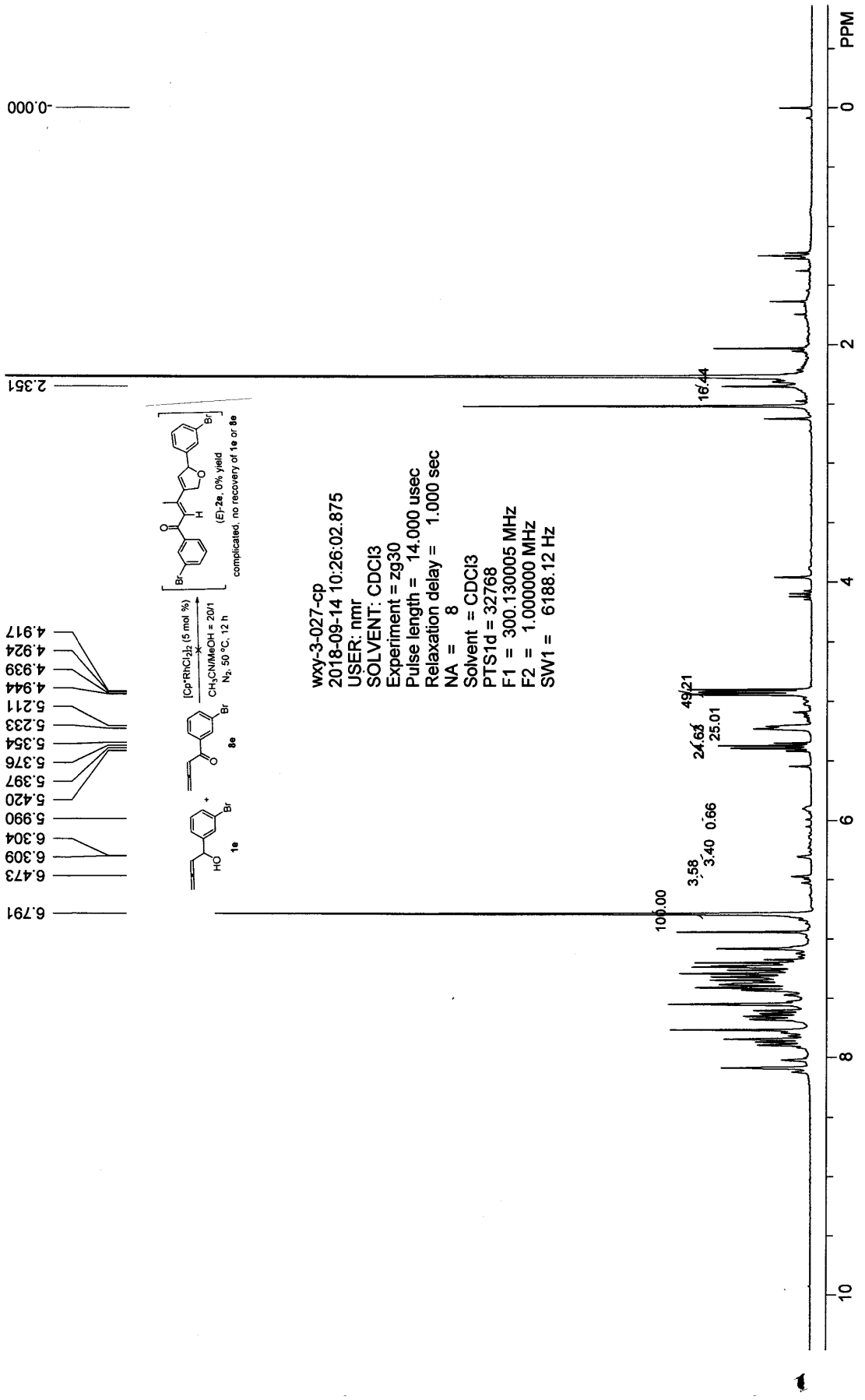

6.618
6.372
6.006
4.323
-0.000
93% purity is determined by nitromethane (2.675 µL, 0.05 mmol, 0.1 mL 0.5 M solution) as the internal standard in 11.1 mg of sample.
wxy-5-024-purity
2019-07-07 14:01:40.125
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 16
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
O
OMe
MeO
O
6f
300.00
40.76
41.16
41.11
8
7
6
5
4
3
2
1
0
PPM

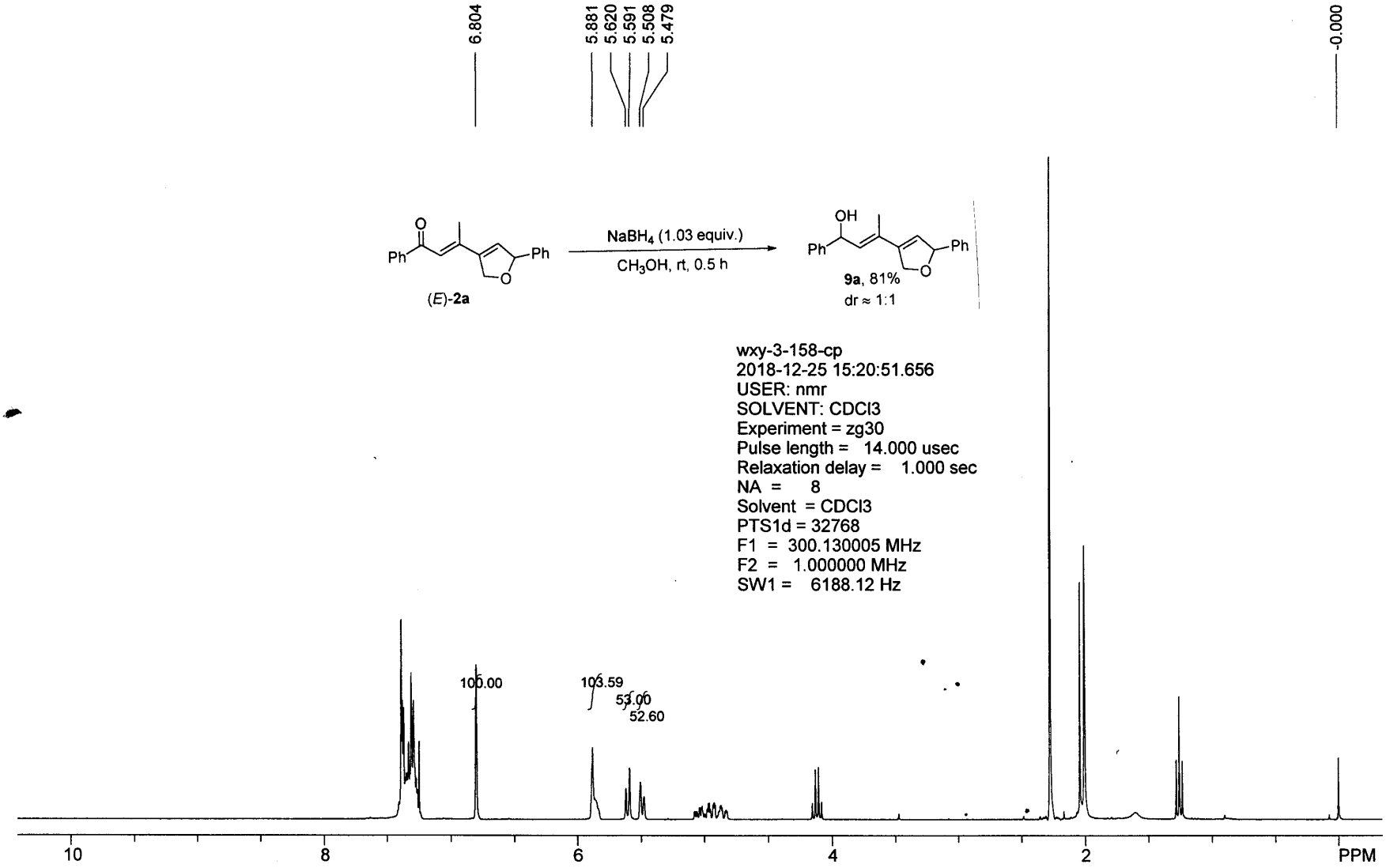

wxy-5-024
2019-06-29 05:40:17.531
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 8000
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
6f
OMe
MeO
O
O
16.729
55.335
55.372
74.941
76.568
77.000
77.423
88.876
114.254
114.447
122.435
126.673
126.875
127.031
128.143
128.364
128.906
132.307
132.629
133.337
137.059
139.477
140.828
141.720
144.670
145.130
159.276
159.956
191.456
PPM
200
150
100
50

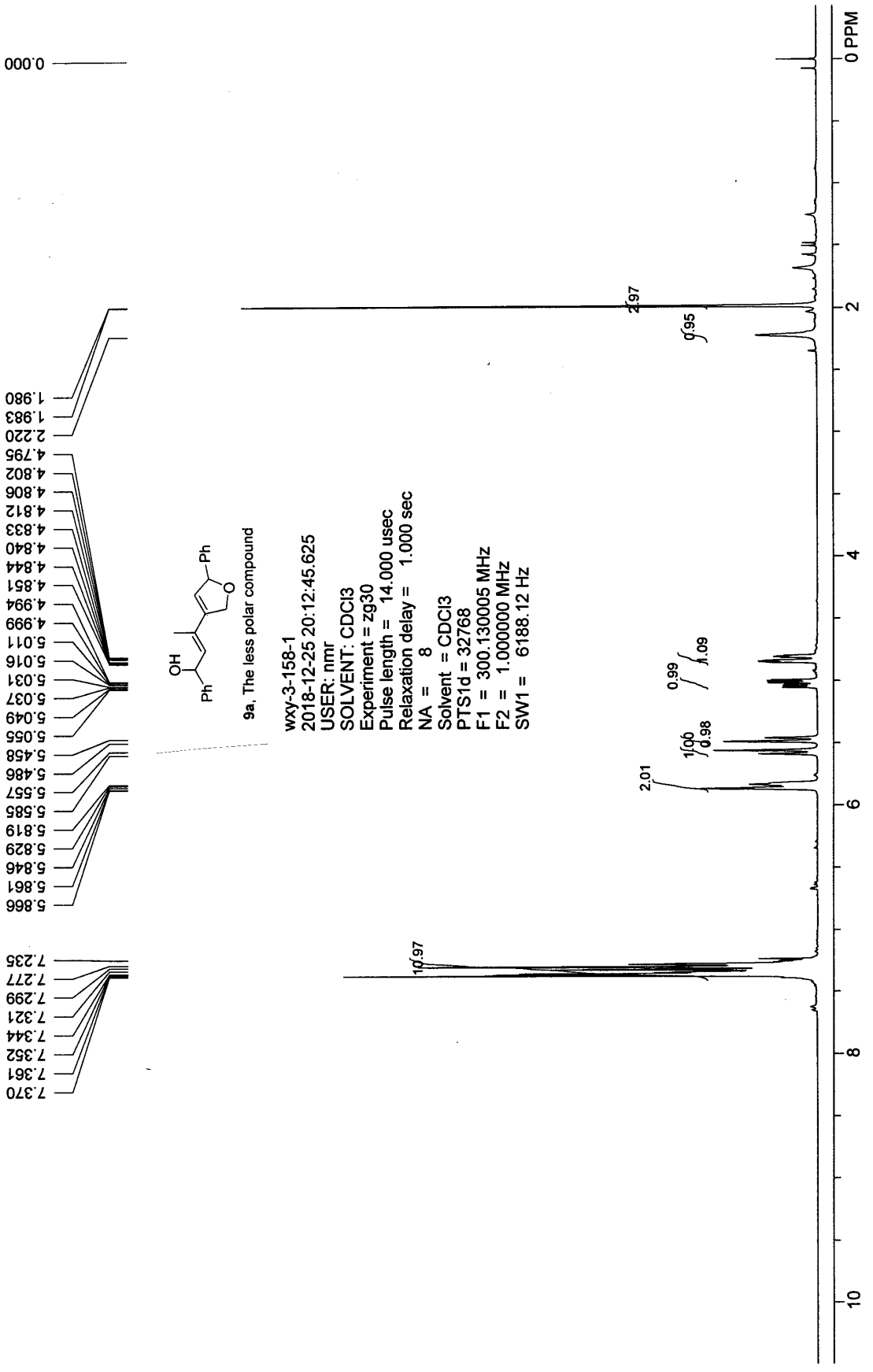

7.273
5.466
5.460
5.454
5.449
5.443
4.649
4.642
4.635
4.629
4.623
4.618
4.612
4.607
4.532
4.525
4.522
4.514
4.510
4.500
4.495
2.102
2.078
2.055
1.515
1.494
1.469
1.444
1.421
1.333
1.285
0.908
0.886
0.863
-0.000
wxy-1-146
2018-03-26 15:31:47.750
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
C7H15
7w
1.00
2.10
1.99
2.09
2.37
8.48
3.49
10
8
6
4
2
0
PPM

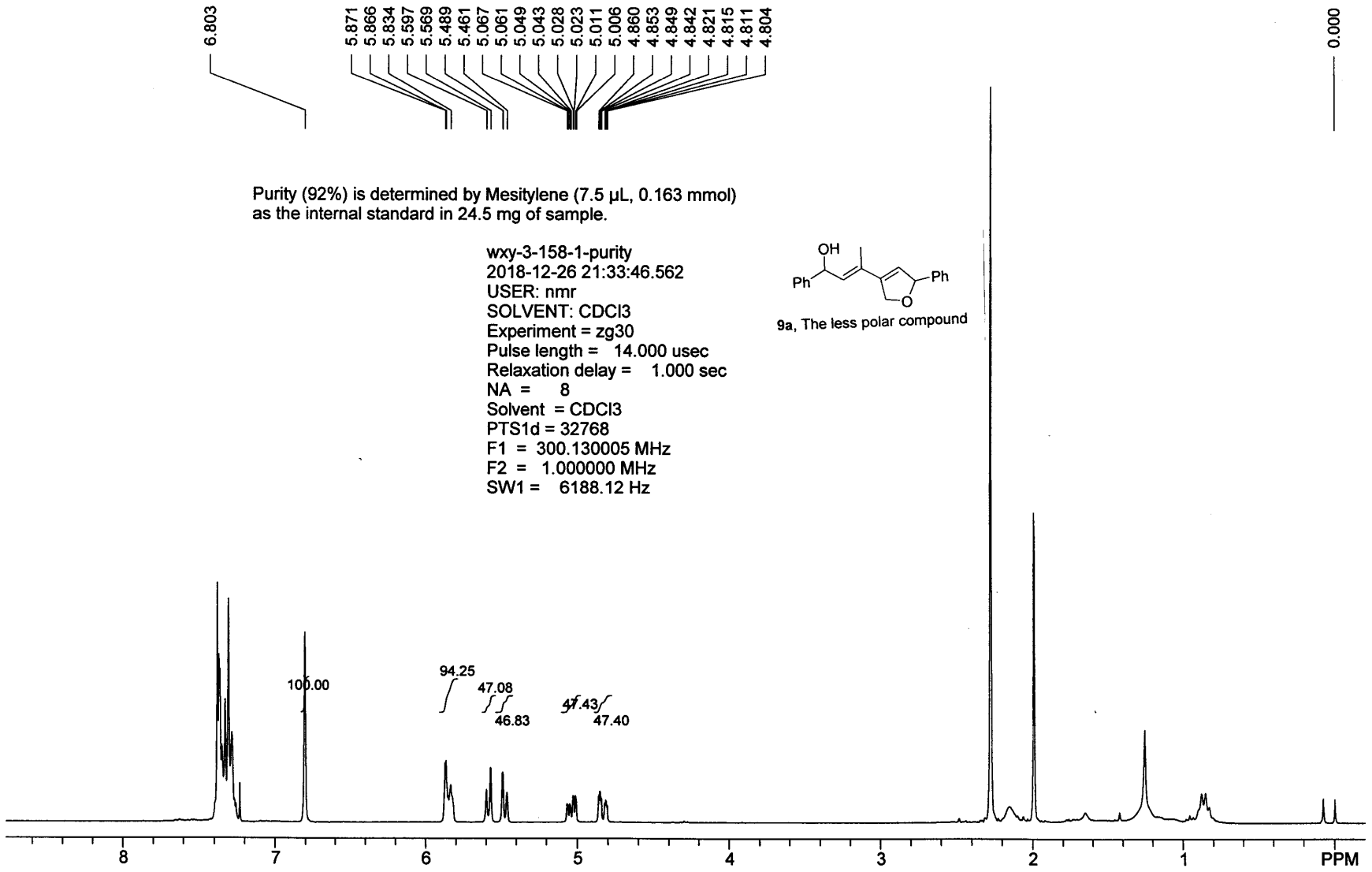

6.795
5.456
5.450
5.445
0.000
purity (77%) is determined by mesitylene (8 μL, 0.17 mmol)
as the internal standard in 29.4 mg of sample.
wxy-4-146-purity
2018-03-29 15:13:43.203
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
C7H15
O
7w
100.00
7.49
10
8
6
4
2
PPM

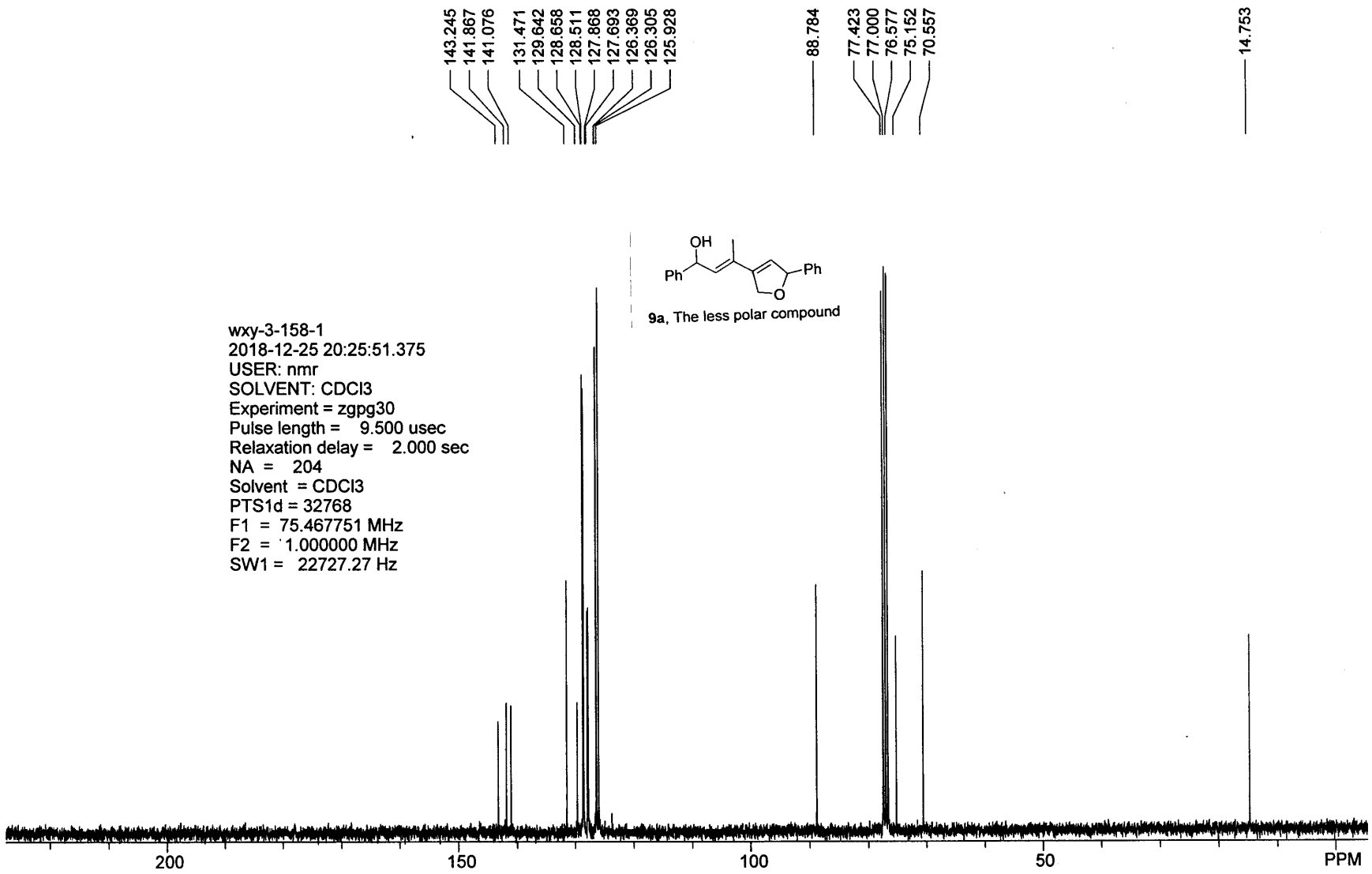

140.663
118.630
77.423
77.028
77.000
76.577
75.934
31.749
29.350
29.055
27.603
27.052
22.594
14.008
wxy-1-146
2018-03-26 16:27:59.312
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 937
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
C7H15
O
7w
250
200
150
100
50
0 PPM

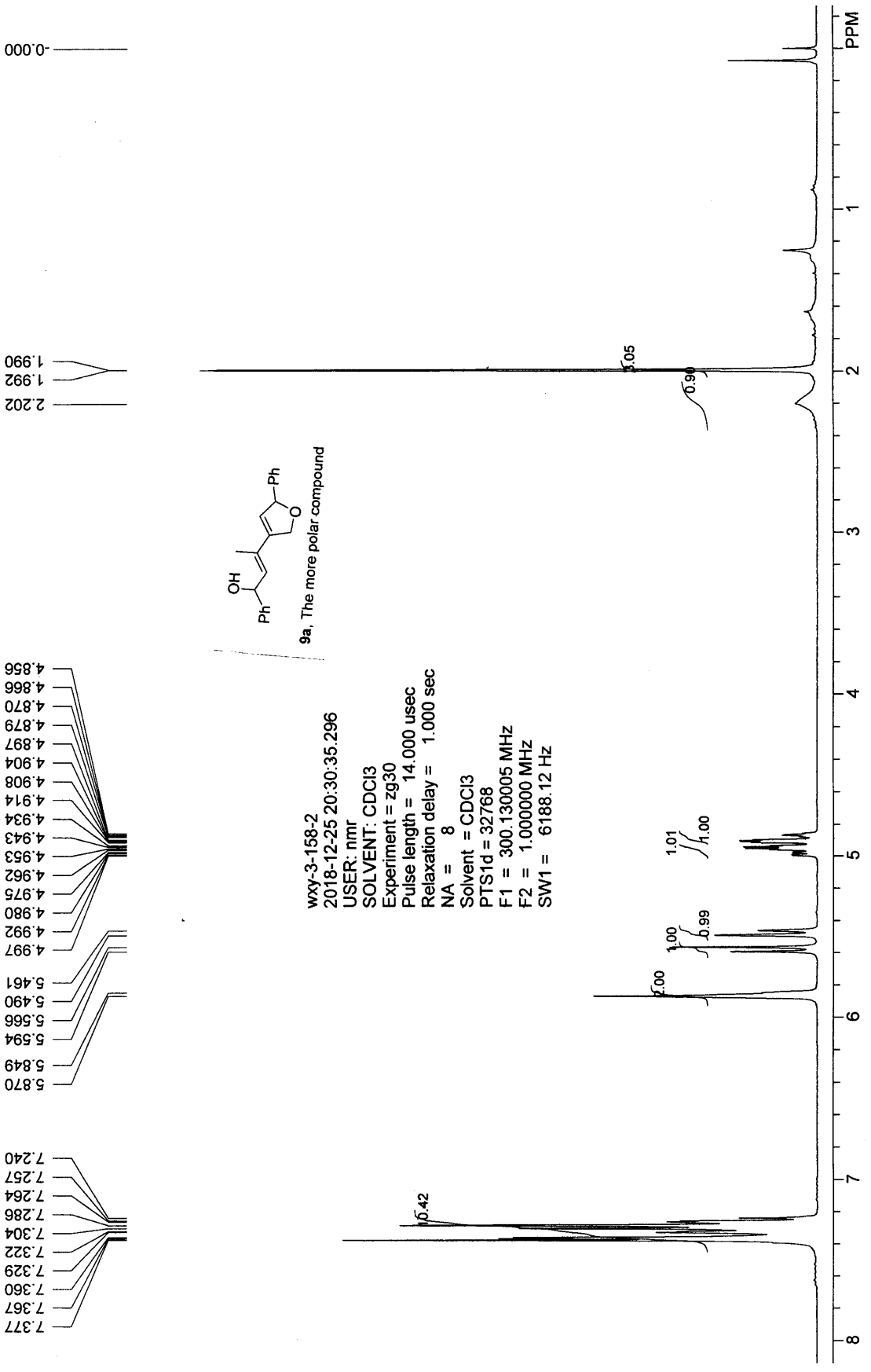

wxy-3-027-cp
2018-09-14 10:26:02.875
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
[Cp*RhCl2]2 (5 mol %)
CH3CN/MeOH = 20/1
N2, 50 °C, 12 h
1e
8e
(E)-2e, 0% yield
complicated, no recovery of 1e or 8e
PPM

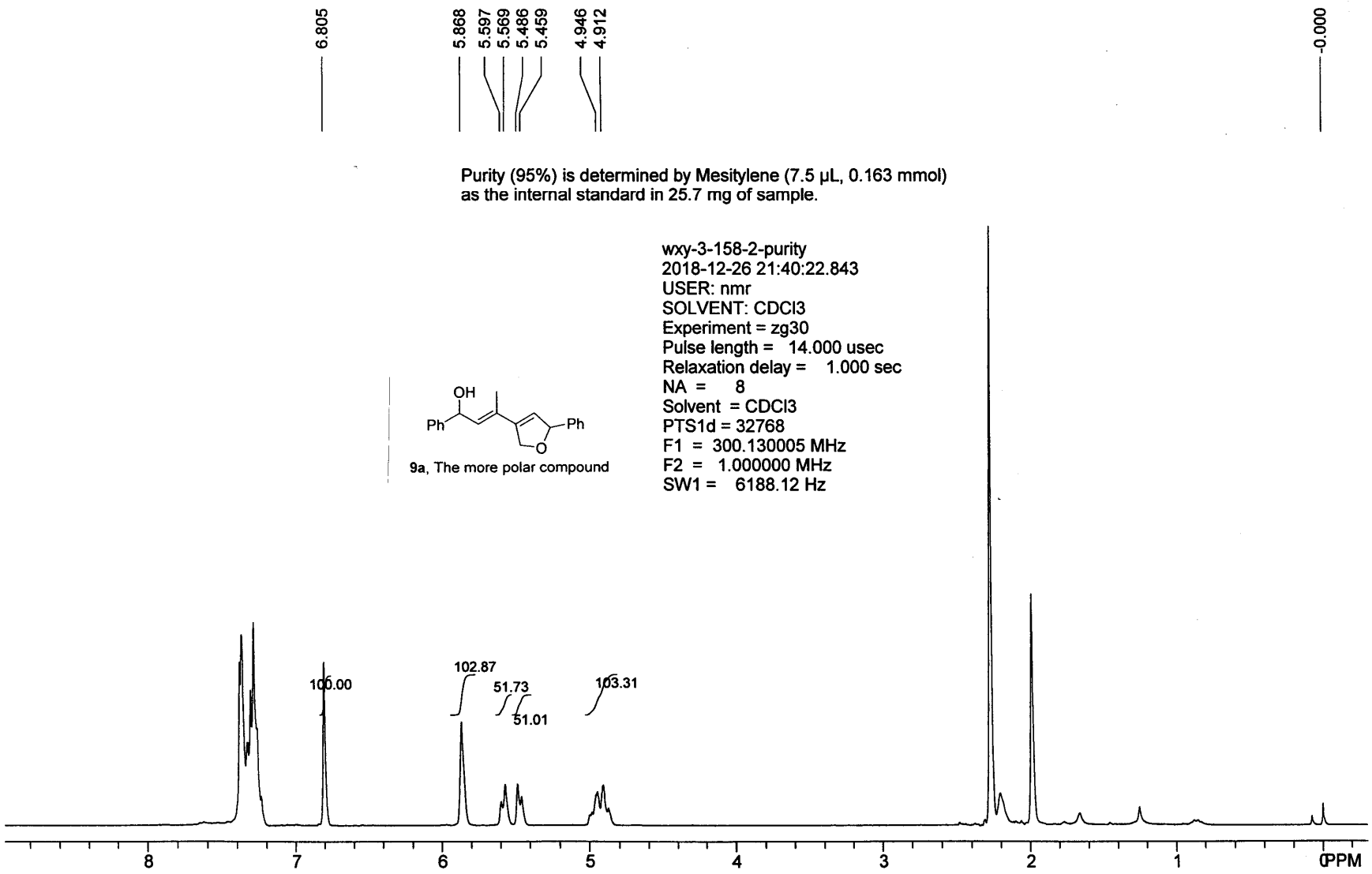

6.804
5.881
5.620
5.591
5.508
5.479
-0.000
(E)-2a
NaBH4 (1.03 equiv.)
CH3OH, rt, 0.5 h
9a, 81%
dr ≈ 1:1
wxy-3-158-cp
2018-12-25 15:20:51.656
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
100.00
103.59
53.00
52.60
10
8
6
4
2
PPM

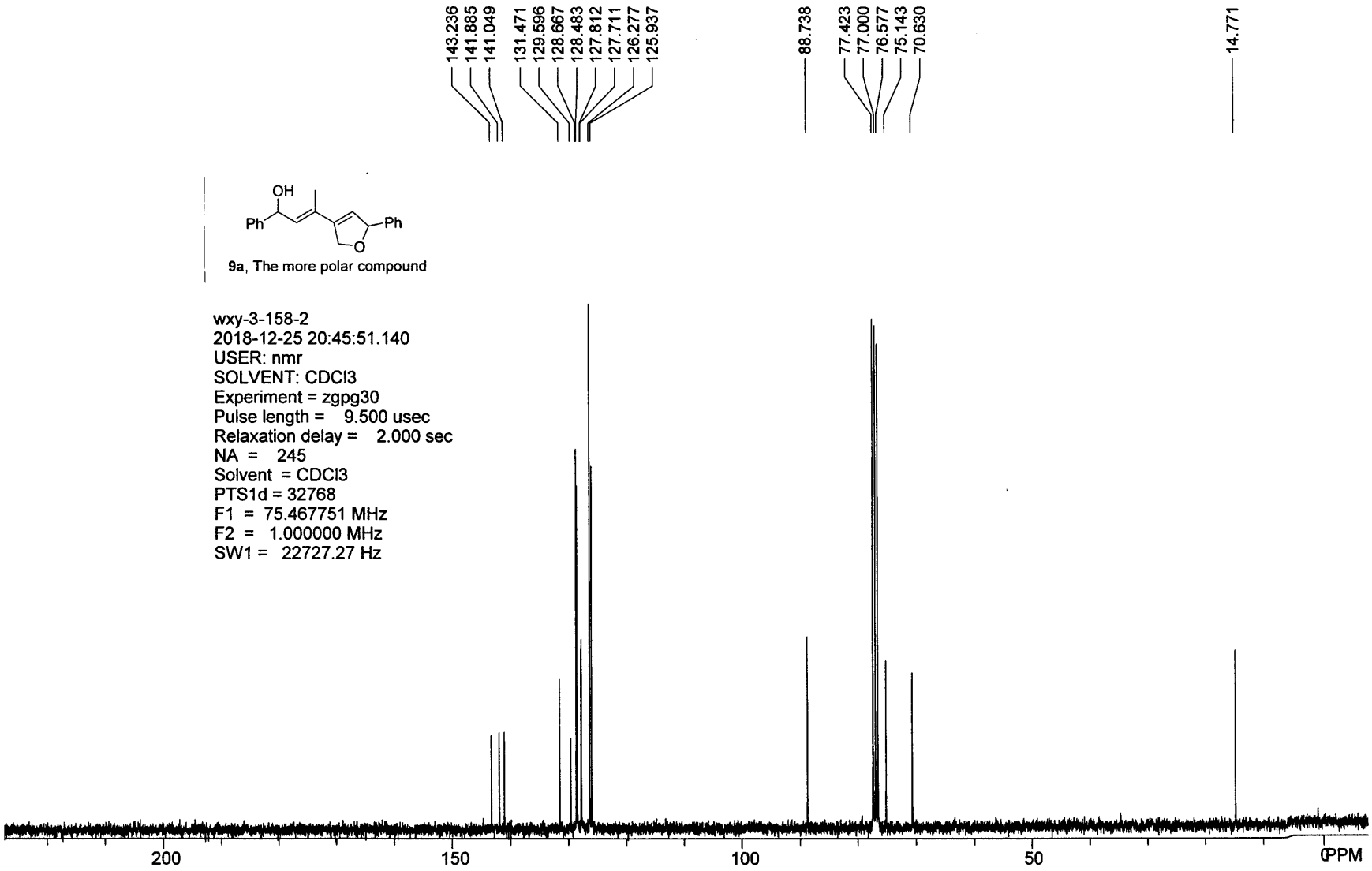

9a, The less polar compound
wxy-3-158-1
2018-12-25 20:12:45.625
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
OH
Ph
Ph
O
0.000
1.980
1.983
2.220
4.795
4.802
4.806
4.812
4.833
4.840
4.844
4.851
4.994
4.999
5.011
5.016
5.031
5.037
5.049
5.055
5.458
5.486
5.557
5.585
5.819
5.829
5.846
5.861
5.866
7.235
7.277
7.299
7.321
7.344
7.352
7.361
7.370
10.97
2.01
1.00
0.98
0.99
1.09
2.97
0.95
10
8
6
4
2
0 PPM

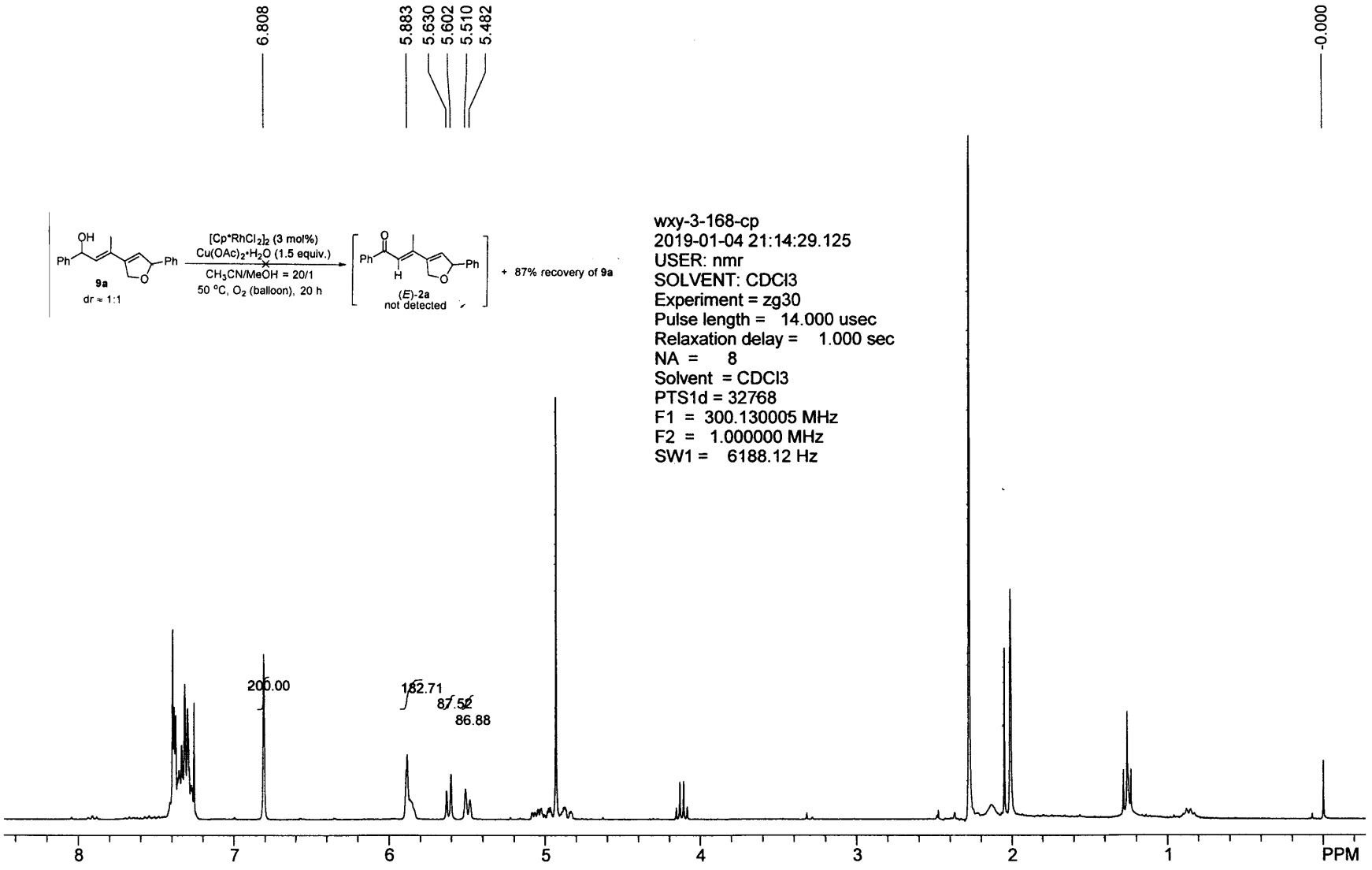

6.803
5.871
5.866
5.834
5.597
5.569
5.489
5.461
5.067
5.061
5.049
5.043
5.028
5.023
5.011
5.006
4.860
4.853
4.849
4.842
4.821
4.815
4.811
4.804
0.000
Purity (92%) is determined by Mesitylene (7.5 µL, 0.163 mmol)
as the internal standard in 24.5 mg of sample.
wxy-3-158-1-purity
2018-12-26 21:33:46.562
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
OH
Ph
Ph
O
9a, The less polar compound
100.00
94.25
47.08
46.83
47.43
47.40
8
7
6
5
4
3
2
1
PPM

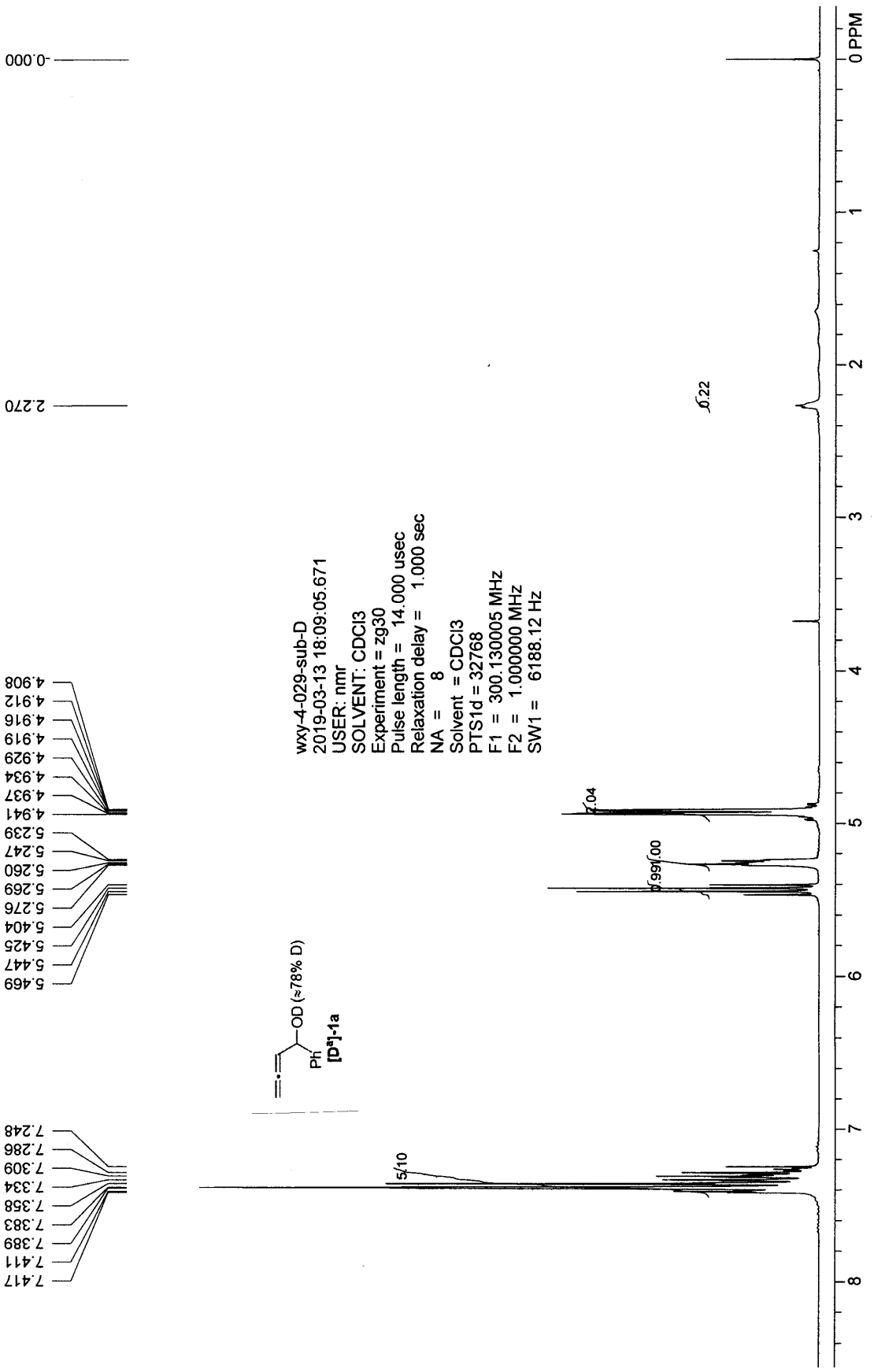

143.245
141.867
141.076
131.471
129.642
128.658
128.511
127.868
127.693
126.369
126.305
125.928
88.784
77.423
77.000
76.577
75.152
70.557
14.753
OH
Ph
Ph
O
9a, The less polar compound
wxy-3-158-1
2018-12-25 20:25:51.375
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 204
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
200
150
100
50
PPM

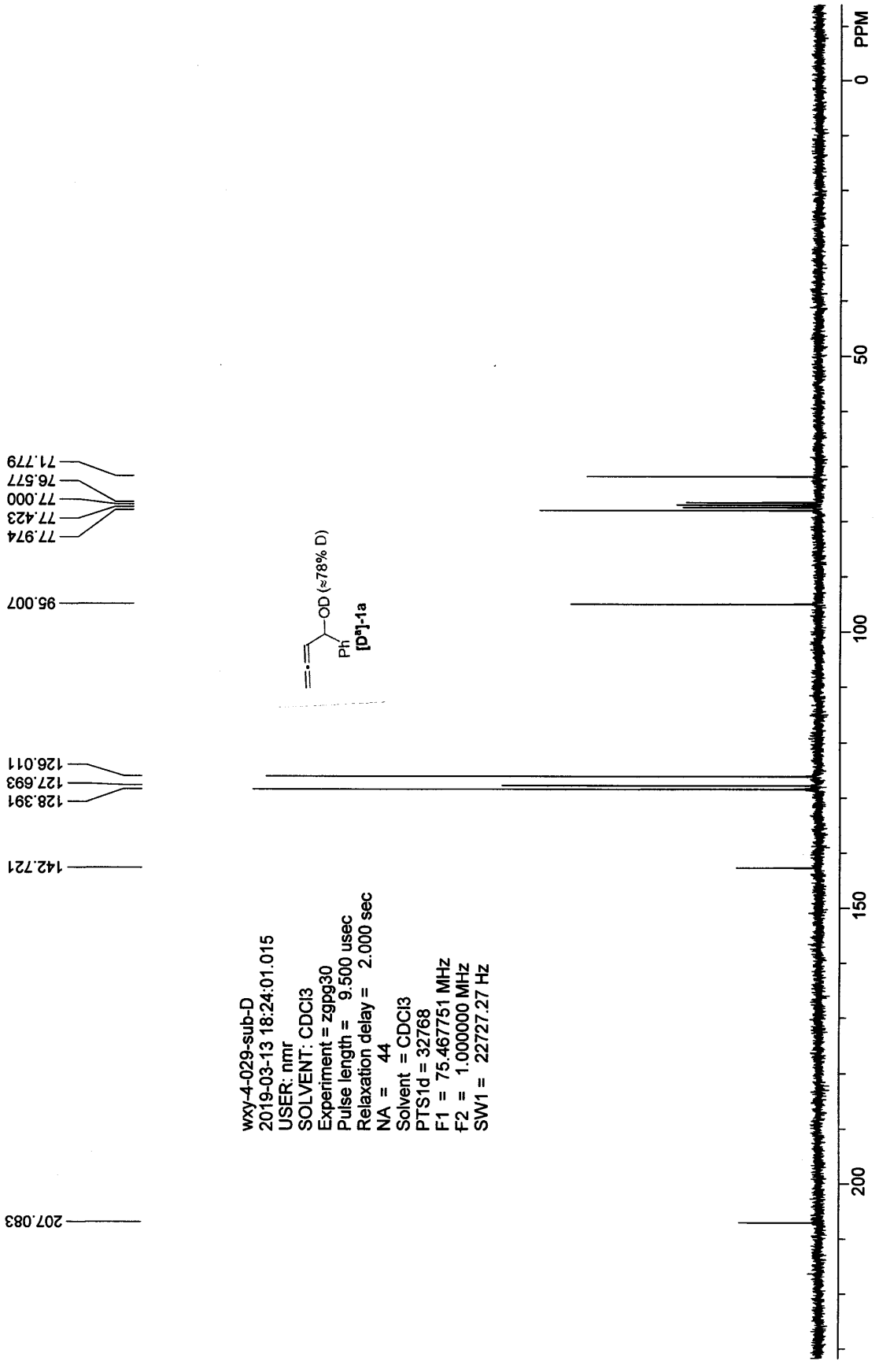

wxy-3-158-2
2018-12-25 20:30:35.296
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
9a, The more polar compound
OH
Ph
Ph
O
PPM
0.000
1.990
1.992
2.202
4.856
4.866
4.870
4.879
4.897
4.904
4.908
4.914
4.934
4.943
4.953
4.962
4.975
4.980
4.992
4.997
5.461
5.490
5.566
5.594
5.849
5.870
7.240
7.257
7.264
7.286
7.304
7.322
7.329
7.360
7.367
7.377

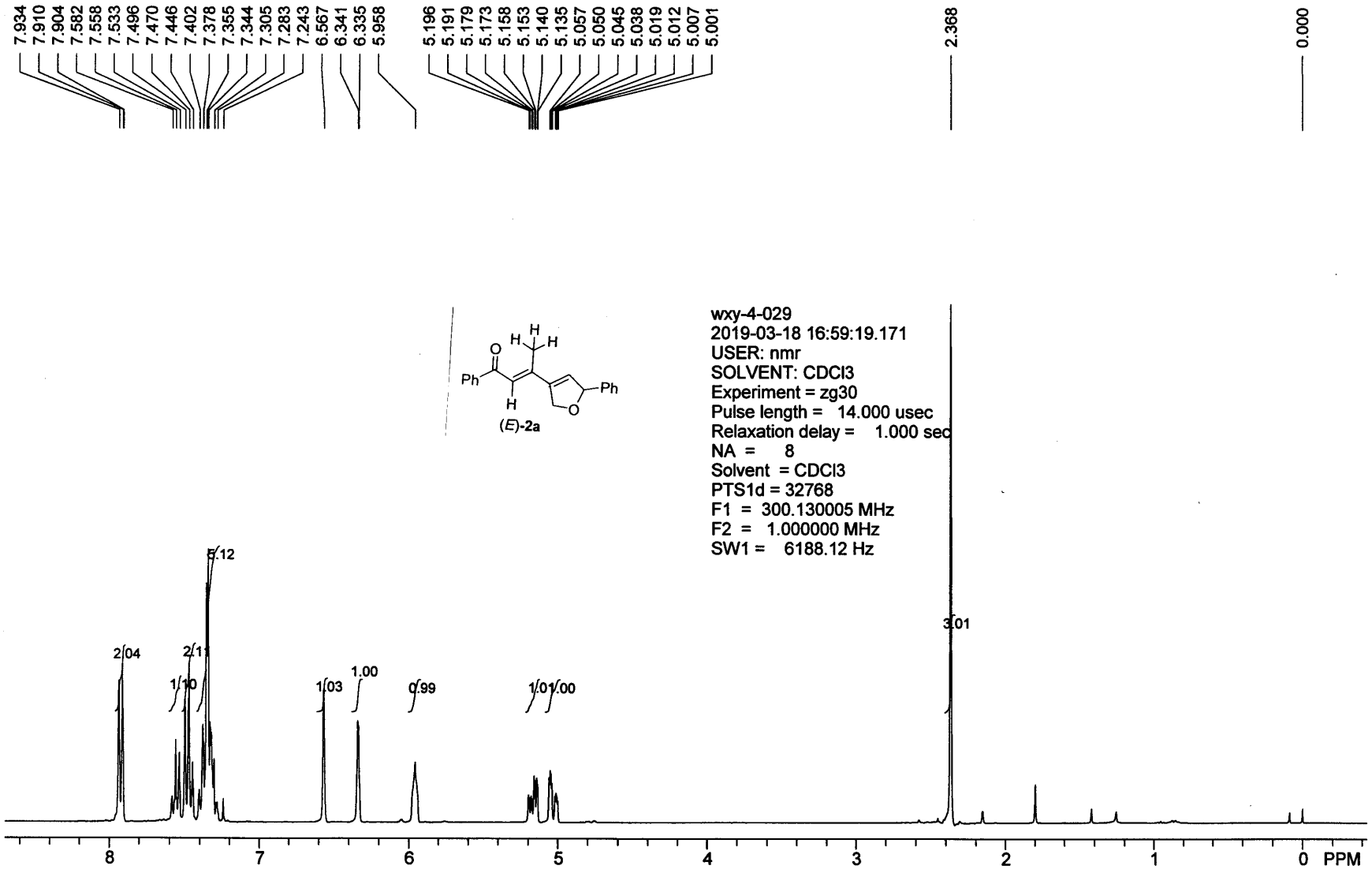

6.805
5.868
5.597
5.569
5.486
5.459
4.946
4.912
0.000
Purity (95%) is determined by Mesitylene (7.5 μL, 0.163 mmol)
as the internal standard in 25.7 mg of sample.
wxy-3-158-2-purity
2018-12-26 21:40:22.843
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
OH
Ph
Ph
O
9a, The more polar compound
100.00
102.87
51.73
51.01
103.31
8
7
6
5
4
3
2
1
0
PPM

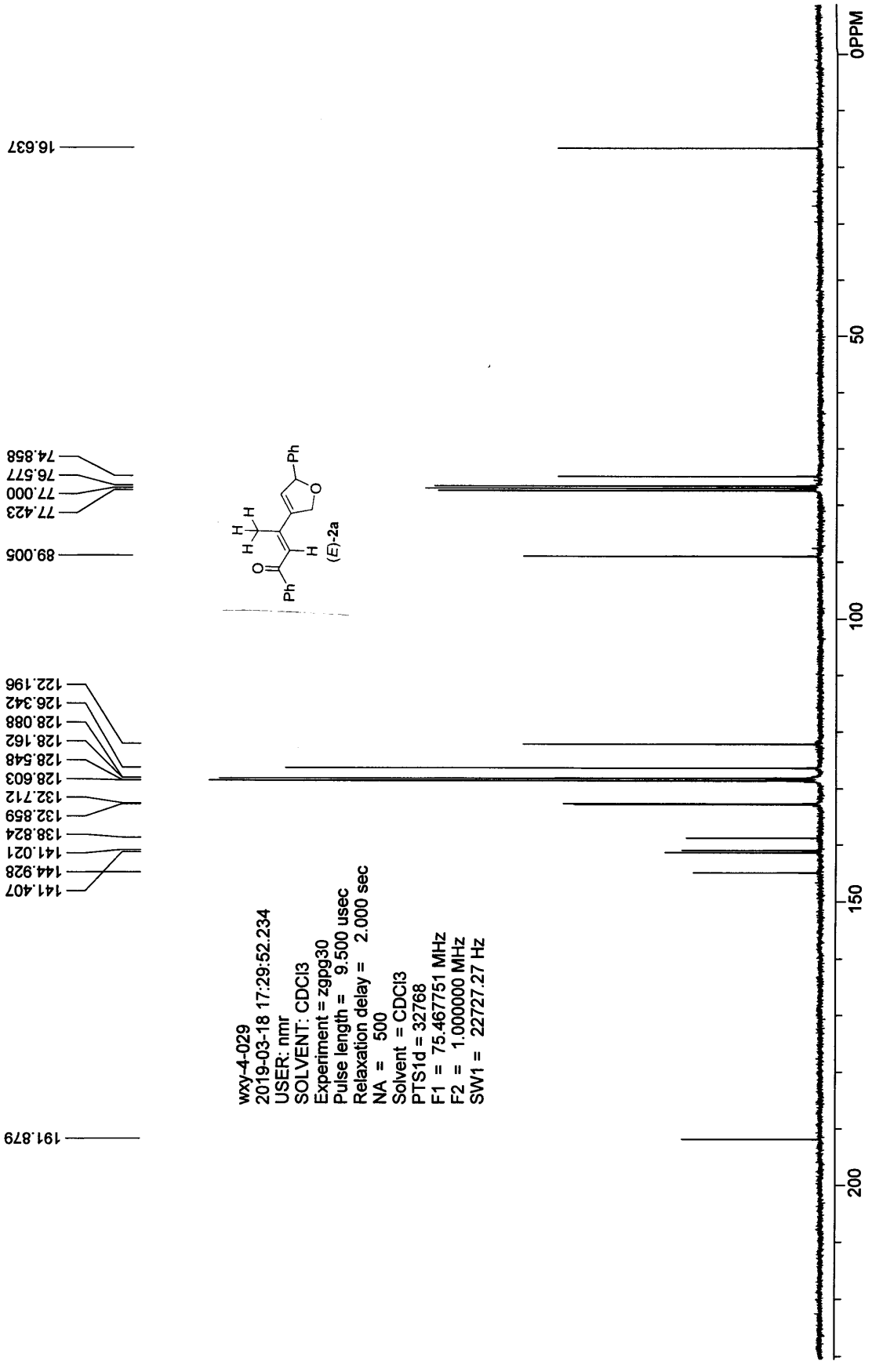

143.236
141.885
141.049
131.471
129.596
128.667
128.483
127.812
127.711
126.277
125.937
88.738
77.423
77.000
76.577
75.143
70.630
14.771
OH
Ph
Ph
O
9a, The more polar compound
wxy-3-158-2
2018-12-25 20:45:51.140
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 245
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
200
150
100
50
0
PPM

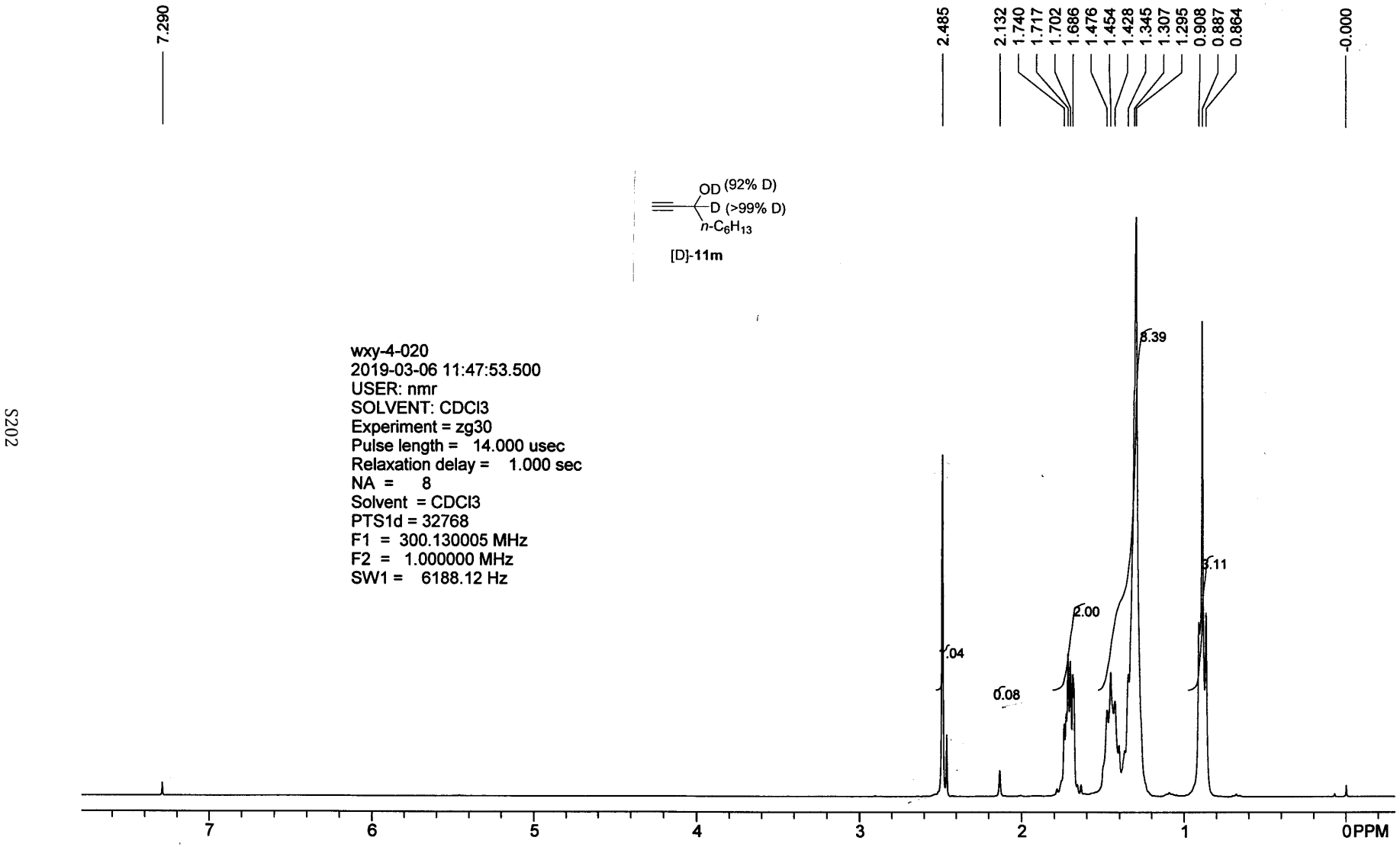

6.808
5.883
5.630
5.602
5.510
5.482
-0.000
9a
dr = 1:1
[Cp*RhCl2]2 (3 mol%)
Cu(OAc)2·H2O (1.5 equiv.)
CH3CN/MeOH = 20/1
50 °C, O2 (balloon), 20 h
(E)-2a
not detected
+ 87% recovery of 9a
wxy-3-168-cp
2019-01-04 21:14:29.125
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
200.00
182.71
87.52
86.88
8
7
6
5
4
3
2
1
PPM

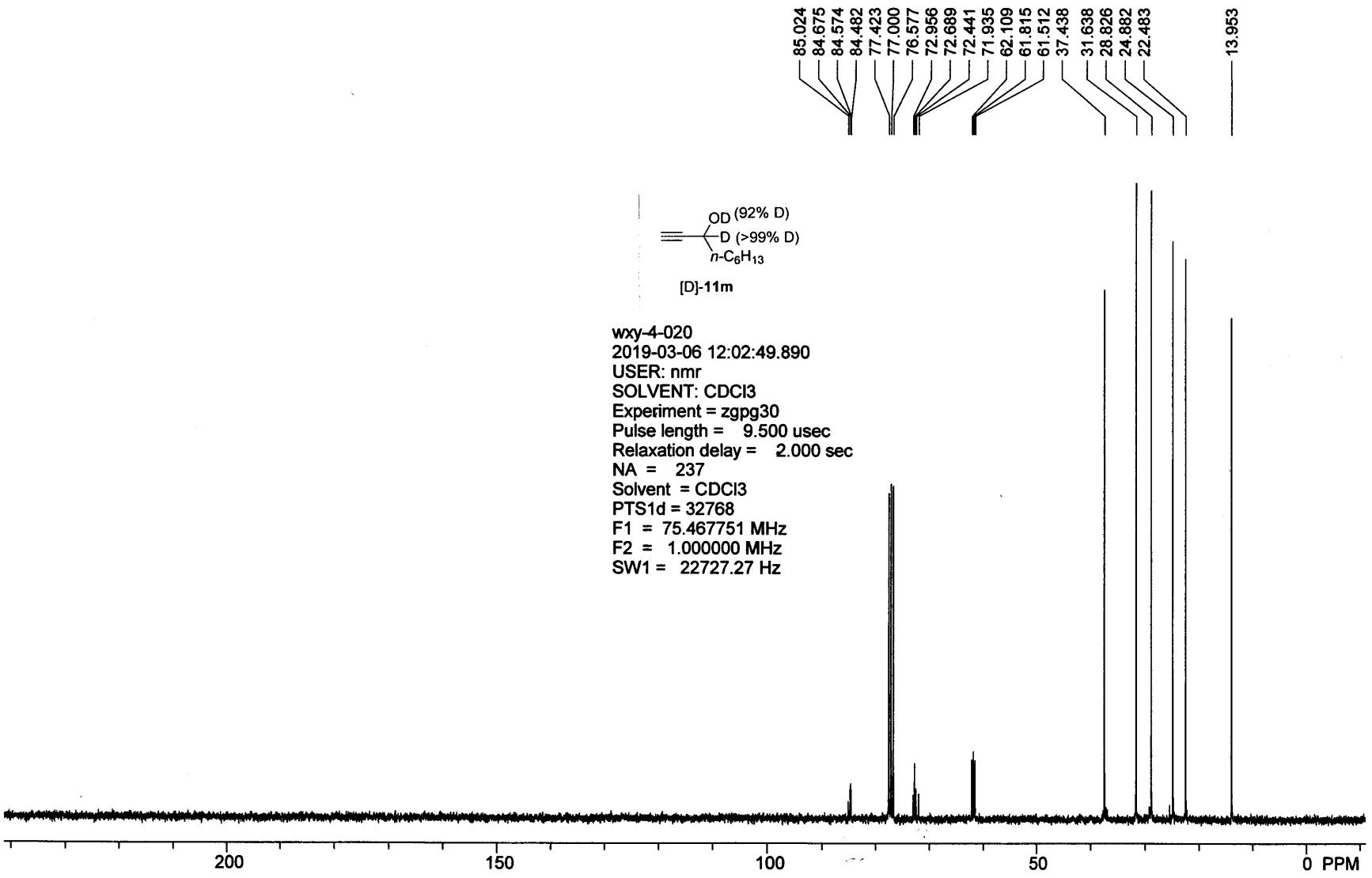

wxy-4-029-sub-D
2019-03-13 18:09:05.671
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
OD (≈78% D)
Ph
[D⁸]-1a
5.10
2.04
0.99
0.66
2.22
0 PPM
-0.000
2.270
4.908
4.912
4.916
4.919
4.929
4.934
4.937
4.941
5.239
5.247
5.260
5.269
5.276
5.404
5.425
5.447
5.469
7.248
7.286
7.309
7.334
7.358
7.383
7.389
7.411
7.417

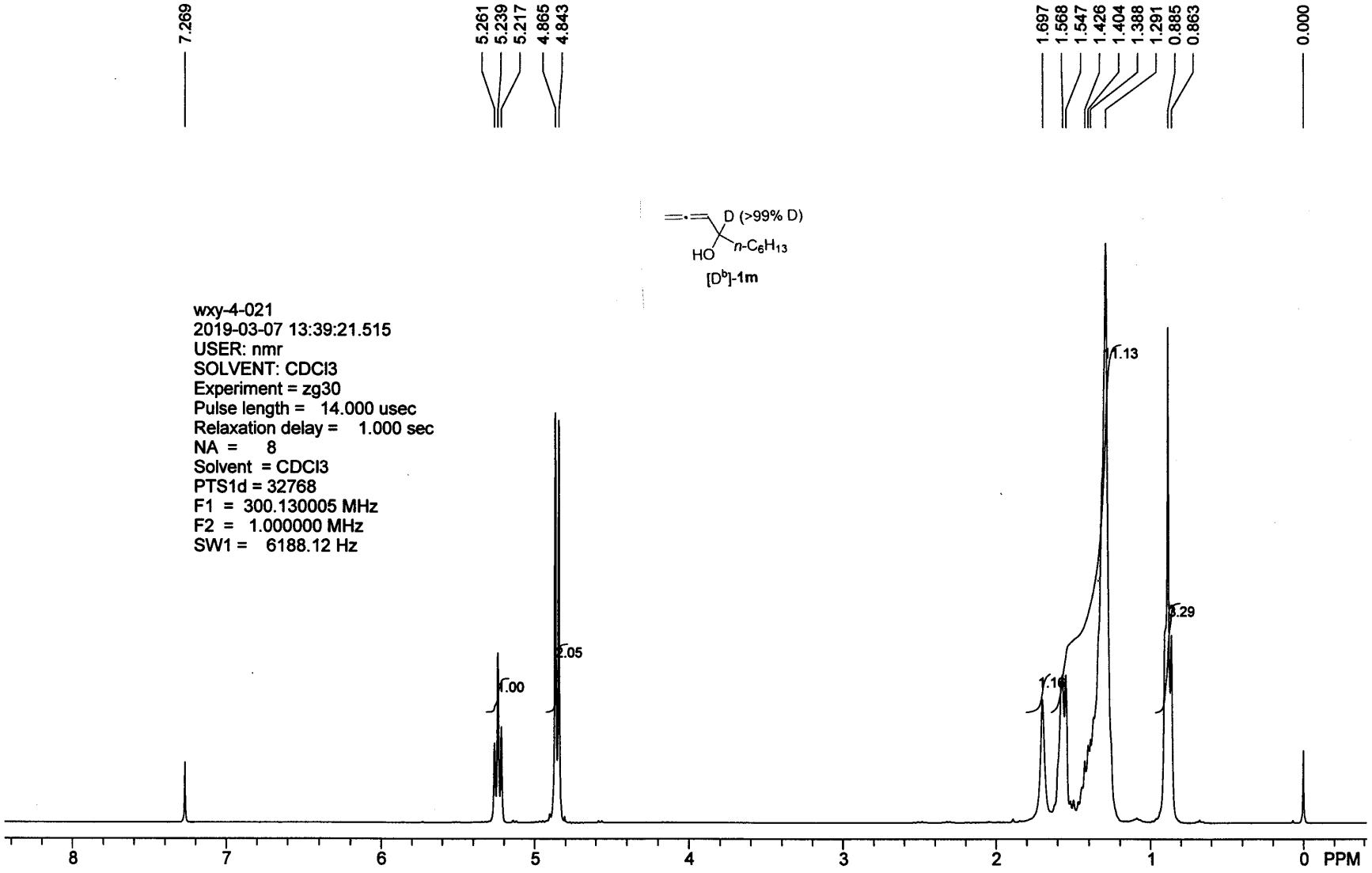

wxy-4-029-sub-D
2019-03-13 18:24:01.015
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 44
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
OD (≈78% D)
Ph
[D³]-1a
207.083
142.721
128.391
127.693
126.011
96.007
77.974
77.423
77.000
76.577
71.779
200
150
100
50
0
PPM

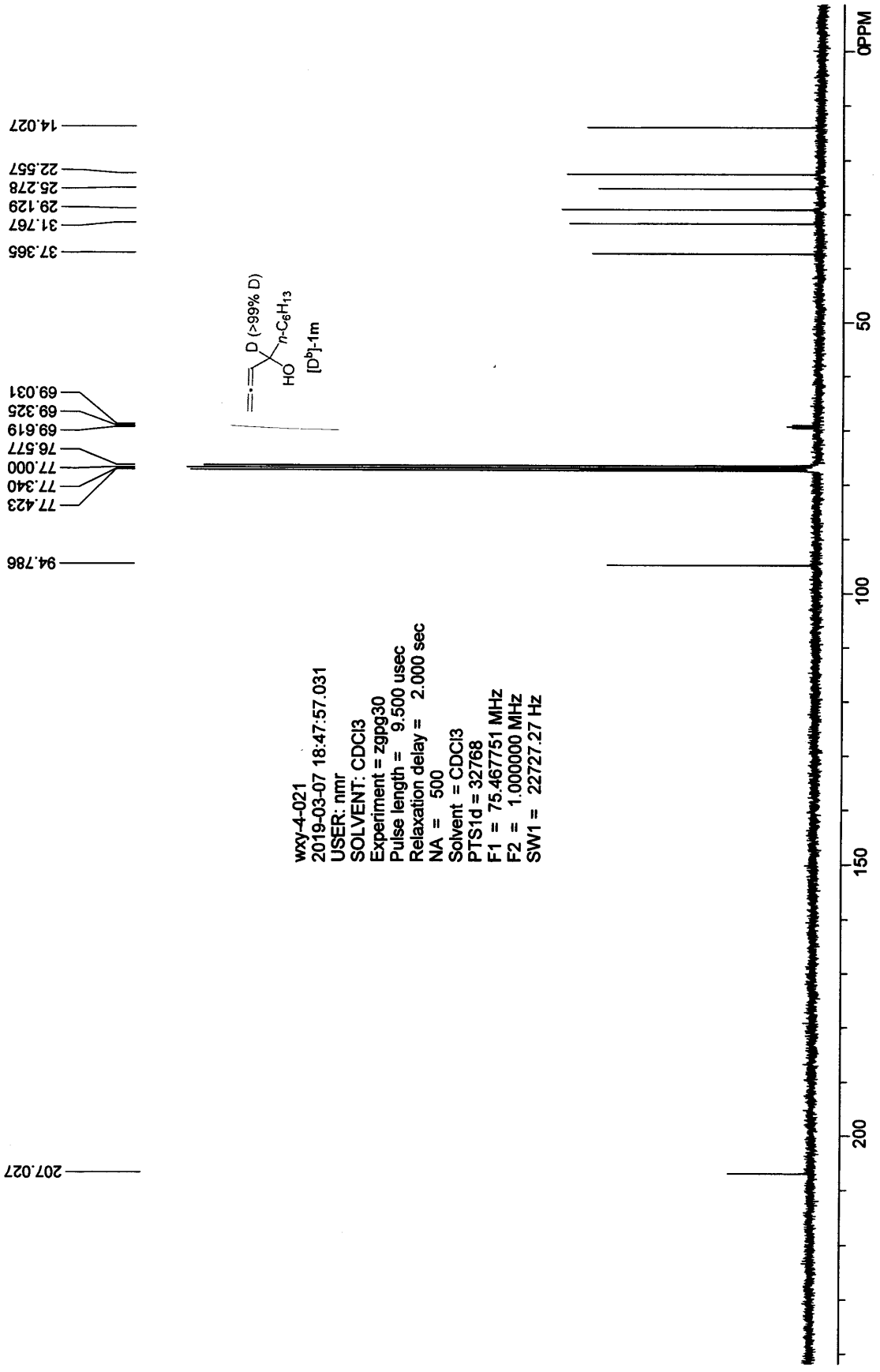

7.934
7.910
7.904
7.582
7.558
7.533
7.496
7.470
7.446
7.402
7.378
7.355
7.344
7.305
7.283
7.243
6.567
6.341
6.335
5.958
5.196
5.191
5.179
5.173
5.158
5.153
5.140
5.135
5.057
5.050
5.045
5.038
5.019
5.012
5.007
5.001
2.368
0.000
(E)-2a
wxy-4-029
2019-03-18 16:59:19.171
USER: nmr
SOLVENT: CDCl3
Experiment = 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
5.12
2.04
2.11
1.10
1.03
1.00
0.99
1.01
1.00
3.01
8
7
6
5
4
3
2
1
0
PPM

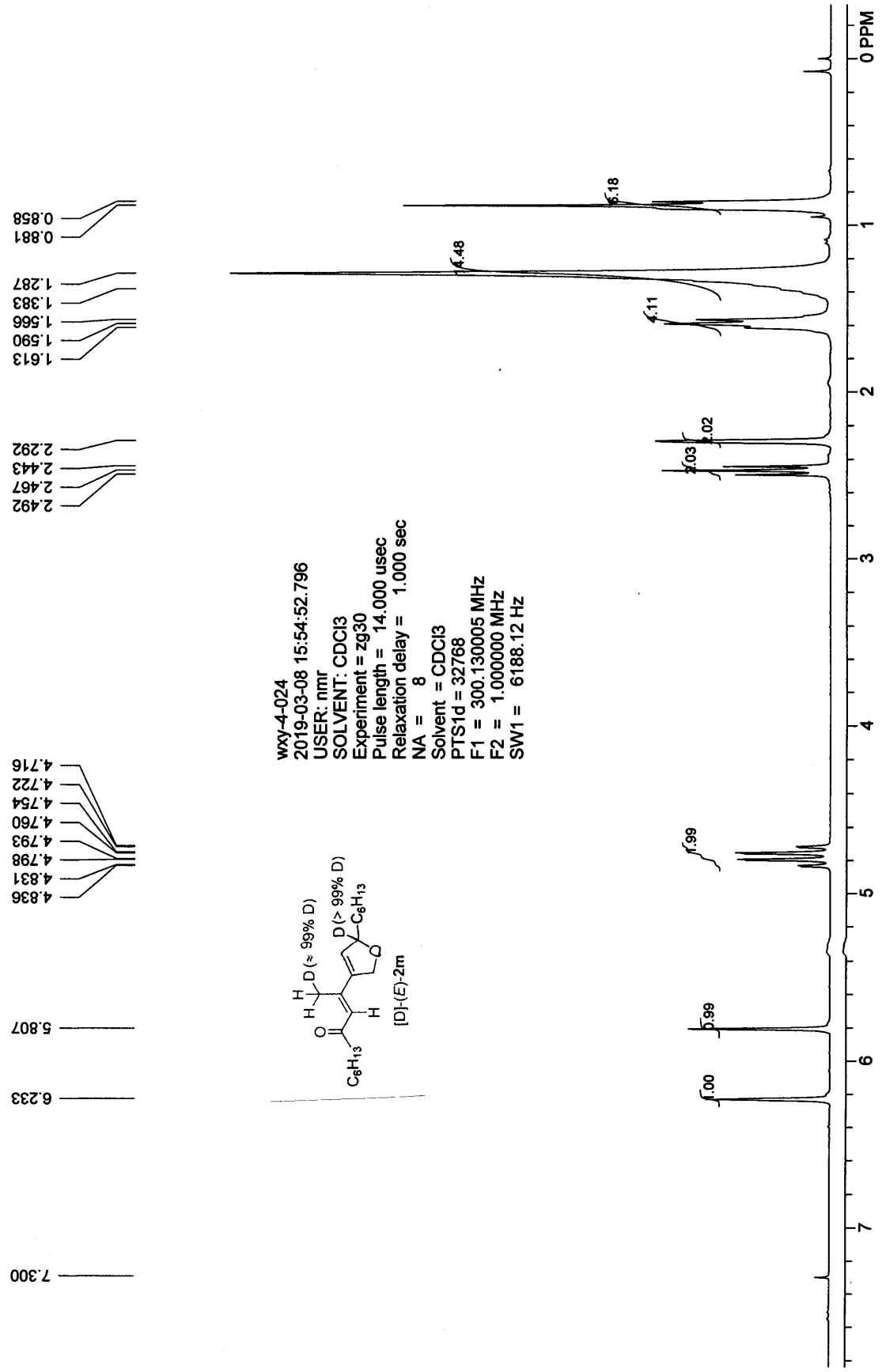

16.637
74.858
76.577
77.000
77.423
89.005
122.196
126.342
128.088
128.162
128.548
128.603
132.712
132.859
138.824
141.021
144.928
141.407
191.879
wxy-4-029
2019-03-18 17:29:52.234
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 500
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
(E)-2a
0PPM
50
100
150
200

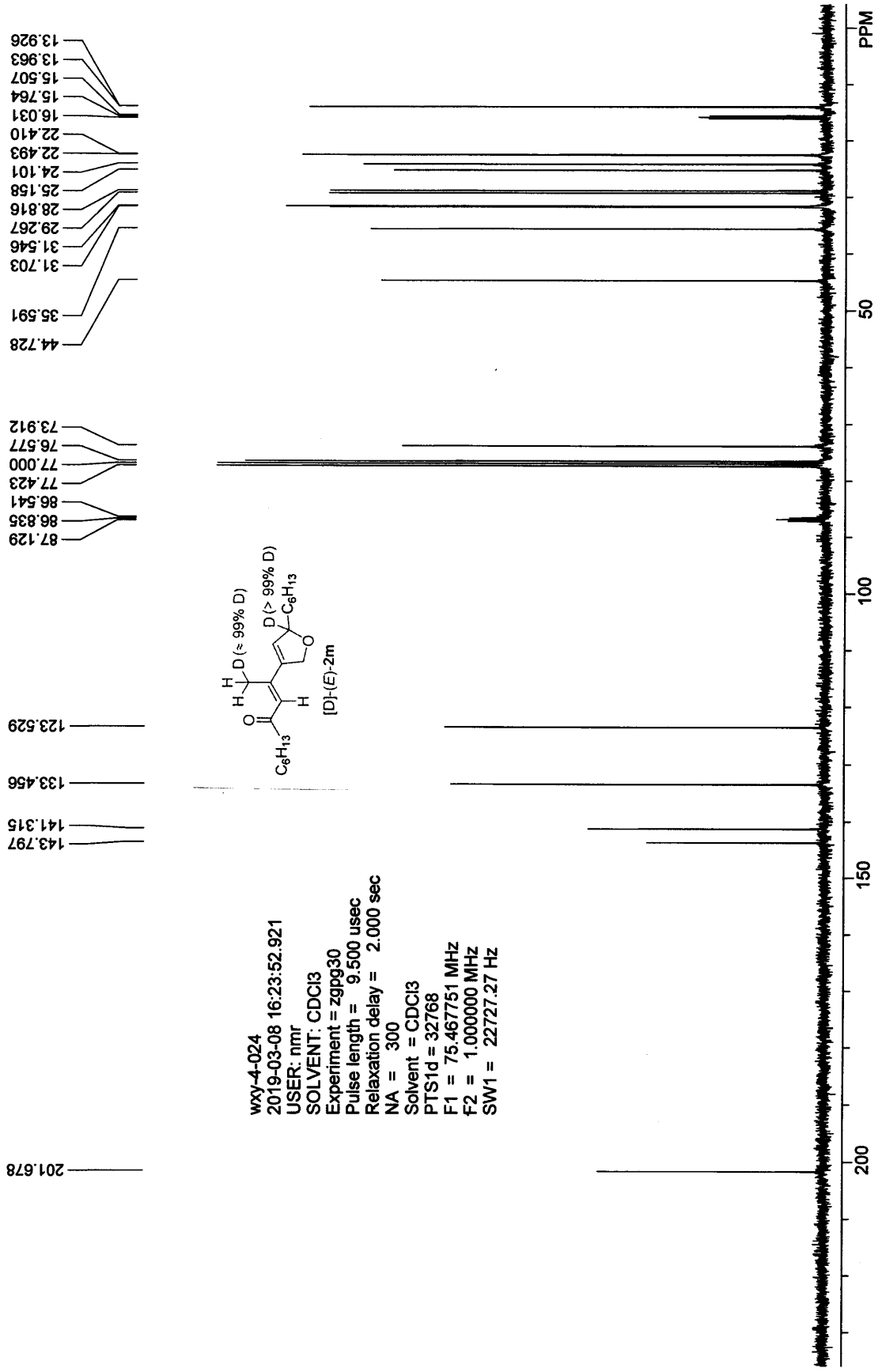

7.290
2.485
2.132
1.740
1.717
1.702
1.686
1.476
1.454
1.428
1.345
1.307
1.295
0.908
0.887
0.864
-0.000
OD (92% D)
D (>99% D)
n-C6H13
[D]-11m
wxy-4-020
2019-03-06 11:47:53.500
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
8.39
3.11
2.00
1.04
0.08
7
6
5
4
3
2
1
0PPM


85.024
84.675
84.574
84.482
77.423
77.000
76.577
72.956
72.689
72.441
71.935
62.109
61.815
61.512
37.438
31.638
28.826
24.882
22.483
13.953
OD (92% D)
D (>99% D)
n-C6H13
[D]-11m
wxy-4-020
2019-03-06 12:02:49.890
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 237
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
200
150
100
50
0 PPM


7.269
5.261
5.239
5.217
4.865
4.843
1.697
1.568
1.547
1.426
1.404
1.388
1.291
0.885
0.863
0.000
D (>99% D)
HO
n-C6H13
[Db]-1m
wxy-4-021
2019-03-07 13:39:21.515
USER: nmr
SOLVENT: CDCl3
Experiment = zg30
Pulse length = 14.000 usec
Relaxation delay = 1.000 sec
NA = 8
Solvent = CDCl3
PTS1d = 32768
F1 = 300.130005 MHz
F2 = 1.000000 MHz
SW1 = 6188.12 Hz
1.00
2.05
1.16
1.13
3.29
8
7
6
5
4
3
2
1
0 PPM


14.027
22.557
25.278
29.129
31.767
37.365
69.031
69.325
69.619
76.577
77.000
77.340
77.423
94.786
207.027
D (>99% D)
n-C6H13
HO
[D1]-1m
wxy-4-021
2019-03-07 18:47:57.031
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 500
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
200
150
100
50
0PPM


wxy-4-024
2019-03-08 15:54:52.796
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
[D]-(E)-2m
D (≈ 99% D)
D (> 99% D)
C6H13
C6H13
0 PPM
1
2
3
4
5
6
7
6.18
4.48
4.11
2.03
2.02
1.99
1.00
0.99
0.858
0.881
1.287
1.383
1.566
1.590
1.613
2.292
2.443
2.467
2.492
4.716
4.722
4.754
4.760
4.793
4.798
4.831
4.836
5.807
6.233
7.300


wxy-4-024
2019-03-08 16:23:52.921
USER: nmr
SOLVENT: CDCl3
Experiment = zgpg30
Pulse length = 9.500 usec
Relaxation delay = 2.000 sec
NA = 300
Solvent = CDCl3
PTS1d = 32768
F1 = 75.467751 MHz
F2 = 1.000000 MHz
SW1 = 22727.27 Hz
[D]-(E)-2m
PPM
13.926
13.963
15.507
15.764
16.031
22.410
22.493
24.101
25.158
28.816
29.267
31.546
31.703
35.591
44.728
73.912
76.577
77.000
77.423
86.541
86.835
87.129
123.529
133.456
141.315
143.797
201.678