Brønsted Acid-Mediated Nazarov Cyclization of Vinyl Allenes

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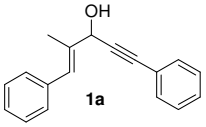
Supporting Information: Experimental procedures, characterization data for all compounds and synthetic intermediates.

Contents:

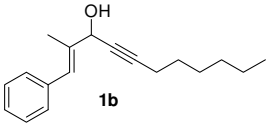
General Information. Reactions were carried out in flame-dried glassware under a positive argon atmosphere unless otherwise stated. Transfer of anhydrous solvents and reagents was accomplished with oven-dried syringes or cannulae. Solvents were distilled before use: methylene chloride from calcium hydride, tetrahydrofuran, diethylether and benzene from sodium/benzophenone ketyl, toluene from sodium metal. All other solvents and commercially available reagents were either purified by standard procedures or used without further purification. Thin layer chromatography was performed on glass plates precoated with 0.25 mm silica gel; the stains for TLC analysis were conducted with 2.5 % *p*-anisaldehyde in AcOH-H_2SO_4-EtOH (1:3:85) and further heating until development of color. Flash chromatography was performed on 230-400 mesh silica gel with the indicated eluents. Nuclear magnetic resonance (NMR) spectra were recorded in indicated deturated solvents and are reported in ppm in the presence of TMS as internal standard and coupling constants (*J*) are reported in Hertz (Hz). Infrared (IR) spectra were recorded neat and reported in cm^{-1}. Mass spectra were recorded by using EI or ESI as specified in each case.

(*E*)-2-Methyl-1,5-diphenylpent-1-en-4-yn-3-ol (**1a**).



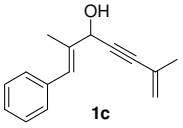
To a flame-dried round bottom flask containing a magnetic stirring bar was added phenylacetylene (3.98 mmol, 406 mg) and ether (8 mL) under Ar. The temperature of the solution was dropped to -78 ^{o}C. *n*-BuLi (1.60 M solution in hexane, 3.98 mmol, 2.48 mL) was added dropwise and the reaction mixture was stirred at the same temperature for 30 min. The α-methyl-*trans*-cinnamaldehyde (3.98 mmol, 581 mg) was added and the resulting solution was allowed to warm to room temperature. The reaction was quenched with saturated aqueous NH_4Cl and diluted with ether. The separated organic layer was washed with brine, dried over anhydrous $MgSO_4$, filtered and concentrated to provide pure compound **1a** (850 mg, yield 86%): R_f 0.28 (hexane:EtOAc 4:1); IR (film) 3341 (br), 3081, 3056, 3024, 2981, 2917, 2859, 2201, 1664, 1598, 1573, 1489, 1442, 1381, 1361, 1281, 1070, 1008, 998, 756, 691 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52-7.50 (m, 2H), 7.39-7.30 (m, 7H), 7.29-7.27 (m, 1H), 6.81 (s, 1H), 5.20 (s, 1H), 2.31 (br s, 1H), 2.11 (d, *J* = 1.2 Hz, 3H); ^{13}C NMR (100 MHZ, $CDCl_3$) δ 137.0, 136.7, 131.7, 129.0, 128.5, 128.2, 128.1, 127.2, 126.7, 122.4, 88.0, 86.3, 68.7, 14.1; HRMS (EI, M^+) for $C_{18}H_{16}O$ calcd 248.1201, found: m/z 248.1196.

(*E*)-2-Methyl-1-phenylundec-1-en-4-yn-3-ol (**1b**).



Synthesized according to the previous procedure using α-methyl-*trans*-cinnamaldehyde (5.61 mmol, 819 mg), 1-octyne (5.61 mmol, 619 mg), and *n*-BuLi (5.61 mmol, 3.50 mL) to afford **1b** (1.187g, yield 83%) as a pale yellow oil: R_f 0.50 (hexane:EtOAc 4:1); IR (film) 3354 (br), 3082, 3057, 3025, 2955, 2930, 2858, 2274, 2223, 1682, 1621, 1600, 1492, 1446, 1378, 1304, 1131, 1011, 751, 699 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36-7.27 (m, 4H), 7.25-7.21 (m, 1H), 6.68 (s, 1H), 4.91 (s, 1H), 2.26 (td, *J* = 7.2, 2.0 Hz, 2H), 1.99 (d, *J* = 1.6 Hz, 3H), 1.92 (br s, 1H), 1.57-1.50 (m, 2H), 1.44-1.36 (m, 2H) 1.34-1.24 (m, 4H), 0.89 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.6, 137.5, 129.2, 128.3, 126.9, 126.9, 87.6, 79.4, 68.8, 31.5, 28.8, 22.7, 19.0, 14.2 [Two aliphatic carbon signals are missing due to peak overlap.]; HRMS (EI, M^+) for $C_{18}H_{24}O$ calcd 256.1827, found: m/z 256.1826.

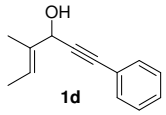
(*E*)-2,6-Dimethyl-1-phenylhepta-1,6-dien-4-yn-3-ol (**1c**).



Synthesized according to the previous procedure using α-methyl-*trans*-cinnamaldehyde (4.12 mmol, 601 mg), 2-methylbut-1-en-3-yne (4.12 mmol, 272 mg), and *n*-BuLi (4.12 mmol, 2.57 mL) to afford

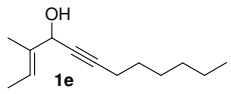
1c (786 mg, yield 90%) as a pale yellow oil: R_f 0.61 (hexane: EtOAC 4:1); IR (film) 3372 (br), 3057, 3025, 2952, 2921, 2856, 2199, 1615, 1446, 1373, 1291, 1073, 1010, 899, 753, 699 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.36-7.29 (m, 4H), 7.25-7.19 (m, 1H), 6.70 (s, 1H), 5.34 (m, 1H), 5.27 (app. quintet, *J* = 1.6 Hz, 1H), 5.04 (s, 1H), 2.01 (d, *J* = 1.2 Hz, 3H), 1.95 (br s, 1H), 1.94 (dd, *J* = 1.4, 1.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.3, 137.0, 129.2, 128.3, 127.4, 127.0, 126.4, 122.8, 87.8, 87.2, 68.9, 23.5, 14.3. HRMS (EI, M^+) for $C_{15}H_{16}O$ calcd 212.1201, found: m/z 212.1193.

(*E*)-4-Methyl-1-phenylhex-4-en-1-yn-3-ol (**1d**).



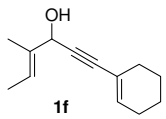
Synthesized according to the previous procedure using (*E*)-2-methylbut-2-enal (2.69 mmol, 226 mg), phenylacetylene (2.69 mmol, 275 mg), and *n*-BuLi (2.69 mmol, 1.68 mL) to afford **1d** (455 mg, yield 91%) as a pale yellow oil: R_f 0.44 (hexane:EtOAc 4:1); IR (microsope) 3353 (br), 3080, 3057, 3033, 2978, 2919, 2860, 2198, 1673, 1598, 1489, 1443, 1380, 1069, 1006, 996, 756, 691 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.49-7.45 (m, 2H), 7.34-7.30 (m, 3H), 5.79 (qq, *J* = 6.6, 0.8 Hz, 1H), 5.01 (s, 1H), 2.56 (br s, 1H), 1.85 (d, *J* = 1.2 Hz, 3H), 1.70 (d, *J* = 6.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 135.3, 131.9, 128.6, 128.5, 122.9, 122.9, 88.9, 86.1, 68.7, 13.5, 12.3; HRMS (EI, M^+) for $C_{13}H_{14}O$ calcd 186.1045, found: m/z 186.1045.

(*E*)-3-Methyldodec-2-en-5-yn-4-ol (**1e**).



Synthesized according to the previous procedure using (*E*)-2-methylbut-2-enal (4.29 mmol, 360 mg), 1-octyne (4.29 mmol, 473 mg), and *n*-BuLi (4.29 mmol, 2.68 mL) to afford **1e** (779 mg, 94%) as a pale yellow oil: R_f 0.56 (hexane: EtOAc 4:1); IR (microscope) 3347 (br), 2956, 2930, 2859, 2229, 1674, 1456, 1379, 1135, 995 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 5.68 (qq, *J* = 6.8, 1.2 Hz, 1H), 4.73 (br s, 1H), 2.25 (td, *J* = 7.2, 2.0 Hz, 2H), 1.74 (app t, *J* = 1.2 Hz, 3H), 1.72 (br s, 1H), 1.64 (d, *J* = 6.8 Hz, 3H), 1.54-1.47 (m, 2H), 1.41-1.25 (m, 6H), 0.88 (t, *J* = 7.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 135.5, 122.1, 86.8, 79.5, 68.3, 31.3, 28.5, 28.5, 22.5, 18.7, 14.0, 13.2, 11.8; HRMS (EI, M^+) for $C_{13}H_{22}O$ calcd 194.1671, found: m/z 194.1672.

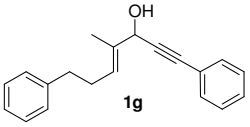
(4*E*)-1-cyclohexenyl-4-methylhex-4-en-1-yn-3-ol (**1f**).



Synthesized according to the previous procedure using (*E*)-2-methylbut-2-enal (4.31 mmol, 362 mg),

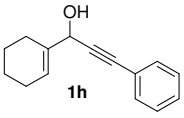
1-ethynylcyclohex-1-ene (4.31 mmol, 458 mg), and *n*-BuLi (4.31 mmol, 2.69 mL) to afford **1f** (778 mg, yield 95%) as a pale yellow oil: R_f 0.60 (hexane:EtOAc 4:1); IR (microscope) 3356 (br), 3027, 2930, 2859, 2218, 2184, 1670, 1631, 1436, 1379, 1199, 1003, 918, 841 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 6.12 (app. quintet, *J* = 2.0 Hz, 1H), 5.69 (qqd, *J* = 6.8, 1.2, 1.2 Hz, 1H), 4.85 (s, 1H), 2.13-2.06 (m, 4H), 1.75 (app t, *J* = 1.0 Hz, 3H), 1.64 (d, *J* = 7.2 Hz, 3H), 1.61-1.54 (m, 4H) [OH peak is not observed.]; ^{13}C NMR (100 MHz, $CDCl_3$) δ 135.3, 135.2, 122.4, 120.1, 87.8, 85.7, 68.5, 29.1, 25.6, 22.2, 21.4, 13.2, 11.9; HRMS (EI, M^+) for $C_{13}H_{18}O$ calcd 190.1358, found: m/z 190.1354.

(*E*)-4-Methyl-1,7-diphenylhept-4-en-1-yn-3-ol (**1g**).



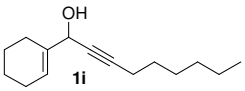
Synthesized according to the previous procedure using (*E*)-2-methyl-5-phenylpent-2-enal (3.18 mmol, 554 mg), phenylacetylene (3.18 mmol, 324 mg), and *n*-BuLi (3.18 mmol, 1.98 mL) to afford **1g** (792 mg, yield 91%) as a pale yellow oil: R_f 0.55 (hexane:EtOAc 4:1); IR (film) 3370 (br), 3083, 3061, 3026, 2925, 2857, 2225, 2203, 1671, 1599, 1489, 1453, 1443, 1302, 1029, 1007, 996, 756, 691 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.47-7.44 (m, 2H), 7.37-7.29 (m, 5H), 7.23-7.19 (m, 3H), 5.77 (tq, *J* = 7.2, 1.2 Hz, 1H), 4.99 (br s, 1H), 2.73 (t, *J* = 6.8 Hz, 2H), 2.43 (app q, *J* = 7.2 Hz, 2H), 1.88 (br s, 1H), 1.78 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.0, 135.2, 131.9, 128.7, 128.6, 128.5, 128.5, 127.6, 126.1, 122.8, 88.5, 86.2, 68.7, 35.7, 29.9, 12.6; HRMS (EI, M^+) for $C_{20}H_{20}O$ calcd 276.1514, found: m/z 276.1509.

1-Cyclohexenyl-3-phenylprop-2-yn-1-ol (**1h**).



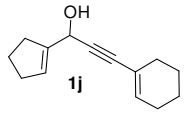
Synthesized according to the previous procedure using cyclohex-1-enecarbaldehyde (2.61 mmol, 287 mg), phenylacetylene (2.61 mmol, 267 mg), and *n*-BuLi (2.61 mmol, 1.63 mL) to afford **1h** (454 mg, yield 82%) as a pale yellow oil: R_f 0.48 (hexane:EtOAc 4:1); IR (microscope) 3406 (br), 3056, 3033, 2930, 2858, 2197, 1714, 1669, 1627, 1489, 1443, 1305, 1029, 1010, 916, 756, 690 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.48-7.46 (m, 2H), 7.34-7.31 (m, 3H), 6.00 (br s, 1H), 4.98 (s, 1H), 2.33-2.26 (m, 1H), 2.22-2.17 (m, 1H), 1.90 (br s, 1H), 2.13-2.09 (m, 2H), 1.75-1.70 (m, 2H), 1.68-1.59 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.2, 131.9, 128.6, 128.5, 125.3, 122.9, 88.6, 86.1, 67.5, 25.3, 24.4, 22.7, 22.4; HRMS (EI, M^+) for $C_{15}H_{16}O$ calcd 212.1201, found: m/z 212.1199.

1-Cyclohexenylnon-2-yn-1-ol (**1i**).



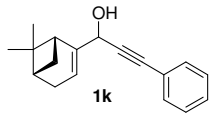
Synthesized according to the previous procedure using cyclohex-1-enecarbaldehyde (3.25 mmol, 357 mg), 1-octyne (3.25 mmol, 357 mg), and *n*-BuLi (3.25 mmol, 2.03 mL) to afford **1i** (652 mg, yield 92%) as a pale yellow oil: R_f 0.58 (hexane:EtOAc 4:1); IR (microscope) 3542 (br), 2930, 2858, 2222, 1636, 1457, 1378, 1136, 992, 847 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 5.90 (br s, 1H), 4.73 (s, 1H), 2.23 (td, *J* = 7.2, 2.0 Hz, 2H), 2.21-2.18 (m, 1H), 2.16-2.06 (m, 3H), 1.73-1.58 (m, 5H), 1.55-1.49 (m, 2H), 1.44-1.29 (m, 6H), 0.91 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.8, 124.6, 86.9, 79.7, 67.3, 31.5, 28.8, 28.7, 25.2, 24.3, 22.7, 22.7, 22.5, 19.0, 14.2; HRMS (EI, M^+) for $C_{15}H_{24}O$ calcd 220.1827, found: m/z 220.1827.

3-Cyclohexenyl-1-cyclopentenylprop-2-yn-1-ol (**1j**).



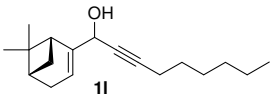
Synthesized according to the previous procedure using cyclopent-1-enecarbaldehyde (4.69 mmol, 450 mg), 1-ethynylcyclohex-1-ene (4.69 mmol, 497 mg), and *n*-BuLi (4.69 mmol, 2.93 mL) to afford **1j** (923 mg, yield 98%) as a pale yellow oil: R_f 0.60 (hexane:EtOAc 4:1); IR (microscope) 3355 (br s), 3026, 2931, 2855, 2217, 2185, 1713, 1629, 1436, 1298, 1040, 950, 918 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 6.12 (tt, *J* = 4.0, 2.0 Hz, 1H), 5.80 (m, 1H), 5.07 (s, 1H), 2.52-2.28 (m, 5H), 2.13-2.06 (m, 4H), 1.94 (quintet, *J* = 7.2 Hz, 2H), 1.66-1.56 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.6, 135.4, 127.5, 120.1, 87.1, 85.5, 62.0, 32.2, 31.4, 29.1, 25.5, 23.3, 22.2, 21.4; HRMS (EI, M^+) for $C_{14}H_{18}O$ calcd 202.1358, found: m/z 202.1353.

1-((1*R*,5*S*)-6,6-dimethylbicyclo[3.1.1]hept-2-en-2-yl)-3-phenylprop-2-yn-1-ol (**1k**).



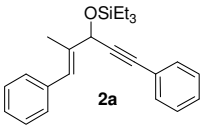
Synthesized according to the previous procedure using (-)-myrtenal (3.59 mmol, 539 mg), phenylacetylene (3.59 mmol, 366 mg), and *n*-BuLi (3.59 mmol, 2.24 mL) to afford **1k** (904 mg, yield 99%) as an inseparable mixture of two diastereomers (dr = 1:1): R_f 0.50 (hexane:EtOAc 4:1); Spectral data for the mixture of isomers: IR (microscope) 3341 (br s), 3081, 3054, 3033, 2985, 2915, 2885, 2830, 2226, 1598, 1490, 1443, 1381, 1365, 1264, 1030, 1014, 1030, 957, 755, 690 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.47-7.44 (m, 4H), 7.34-7.30 (m, 6H), 5.77 (td, *J* = 3.0, 1.6 Hz, 1H), 5.75-5.74 (m, 1H), 5.02 (d, *J* = 0.8 Hz, 1H), 4.99 (d, *J* = 1.2 Hz, 1H), 2.53-2.45 (m, 4H), 2.43-2.31 (m, 4H), 2.30-2.20 (m, 2H), 2.17-2.14 (m, 2H), 1.36 (s, 3H), 1.35 (s, 3H), 1.28 (dd, *J* = 8.2, 3.0 Hz, 2H; 2 overlapping 1H signals), 0.92 (s, 3H), 0.91 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.9, 146.9, 131.9, 131.9, 128.6, 128.5, 128.5, 122.9, 120.3, 119.4, 88.31, 85.8, 85.6, 65.9, 65.5, 43.2, 42.9, 41.0, 41.0, 38.3, 32.0, 31.9, 31.4, 31.4, 26.3, 21.5, 21.4 [Some carbon signals are missing due to peak overlap.]; HRMS (EI, M^+) for $C_{18}H_{20}O$ calcd 252.1514, found: m/z 252.1513.

1-((1*R*,5*S*)-6,6-dimethylbicyclo[3.1.1]hept-2-en-2-yl)non-2-yn-1-ol (**1l**).



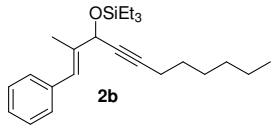
Synthesized according to the previous procedure using (-)-myrtenal (3.14 mmol, 471 mg), 1-octyne (3.14 mmol, 346 mg), and *n*-BuLi (3.14 mmol, 1.96 mL) to afford **1l** (711 mg, yield 87%) as an inseparable mixture of two diastereomers (dr = 1:1): R_f 0.56 (hexane:EtOAc 4:1); Spectral data for the mixture of isomers: IR (microscope) 3365 (br s), 2984, 2930, 2871, 2832, 2222, 1614, 1467, 1381, 1365, 1001, 965 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 5.65-5.63 (m, 2H), 4.74 (d, *J* = 1.2 Hz, 1H), 4.72 (d, *J* = 1.2 Hz, 1H), 2.47-2.36 (m, 4H), 2.33-2.26 (m, 4H), 2.22 (td, *J* = 7.0, 1.5 Hz, 4H), 2.12-2.10 (m, 2H), 1.51 (m, 4H), 1.41-1.33 (m, 4H), 1.32-1.24 (m, 8H), 1.32 (s, 6H), 0.90 (t, *J* = 6.8 Hz, 6H), 0.85 (s, 3H), 0.84 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.5, 119.6, 118.9, 86.6, 86.4, 79.2, 79.1, 65.7, 65.3, 43.0, 42.8, 41.0, 41.0, 38.1, 38.1, 32.0, 31.9, 31.5, 31.3, 31.3, 28.8, 28.8, 28.7, 28.7, 26.3, 22.7, 21.4, 21.2, 18.9, 14.2 [Some carbon signals are missing due to peak overlap.]; HRMS (EI, M^+) for $C_{18}H_{28}O$ calcd 260.2140, found: m/z 260.2138.

((*E*)-2-Methyl-1,5-diphenylpent-1-en-4-yn-3-yloxy)triethylsilane (**2a**).



To a flame-dried round bottom flask containing a magnetic stirring bar was sequentially added hydroxyenyne **1a** (1.06 mmol, 264 mg), CH_2Cl_2 (5 mL), and 2,6-lutidine (3.18 mmol, 0.37 mL) under Ar. The temperature of the solution was dropped to -78 ^oC. Triethylsilyl trifluoromethanesulfonate (1.59 mmol, 0.36 mL) was added dropwise and the resulting solution was stirred at the same temperature for 30 min. The reaction mixture was quenched with H_2O and diluted with CH_2Cl_2. The separated organic layer was washed with brine, dried over anhydrous $MgSO_4$, filtered and concentrated. The crude mixture thus obtained was purified by flash column chromatography (silica gel, 2% EtOAc/hexane) to give pure siloxyenyne **2a** (369 mg, yield 96%): R_f 0.80 (hexane:EtOAc 4:1); IR (film) 3081, 3060, 3026, 2956, 2912, 2876, 2203, 1691, 1665, 1490, 1450, 1238, 1060, 1003, 754, 690 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.53-7.50 (m, 2H), 7.43-7.35 (m, 7H), 7.29-7.27 (m, 1H), 6.80 (br s, 1H), 5.21 (s, 1H), 2.11 (d, *J* = 1.2 Hz, 3H), 1.16-1.06 (m, 9H), 0.86-0.80 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.5, 137.5, 131.6, 129.0, 128.2, 128.0, 126.5, 125.7, 123.0, 89.2, 85.0, 68.7, 14.1, 6.8, 4.9 [One sp^2 carbon signal is missing due to peak overlap.]; HRMS (EI, M^+) for $C_{24}H_{30}OSi$ calcd 362.2066, found: m/z 362.2063.

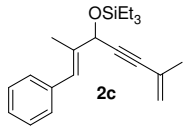
((*E*)-2-Methyl-1-phenylundec-1-en-4-yn-3-yloxy)triethylsilane (**2b**).



2b

Prepared according to the previous procedure using **1b** (1.95 mmol, 500 mg), 2,6-lutidine (5.85 mmol, 0.68 mL), triethylsilyl trifluoromethanesulfonate (2.93 mmol, 0.66 mL) to afford **2b** (721 mg, yield 99%) as a pale yellow oil: R_f 0.85 (hexane:EtOAc 4:1); IR (microsope) 3062, 3029, 2956, 2932, 2875, 2860, 2211, 1712, 1671, 1601, 1455, 1413, 1379, 1239, 1069, 1005, 745, 700 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.34-7.27 (m, 4H), 7.23-7.18 (m, 1H), 6.62 (s, 1H), 4.89 (br s, 1H), 2.23 (td *J* = 7.2, 2.0 Hz, 2H), 1.95 (d, *J* = 1.2 Hz, 3H), 1.56-1.48 (m, 2H), 1.44-1.36 (m, 2H), 1.35-1.24 (m, 4H), 1.00 (t, *J* = 8.0 Hz, 9H), 0.88 (t, *J* = 7.0 Hz, 3H), 0.73-0.66 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.5, 137.9, 129.2, 128.2, 126.6, 125.4, 86.0, 80.2, 68.7, 31.5, 28.8, 28.8, 22.8, 19.1, 14.2, 7.0, 5.1 [One aliphatic carbon signal is missing due to peak overlap.]; HRMS (EI, M^+) for $C_{24}H_{38}OSi$ calcd 370.2692, found: m/z 370.2684.

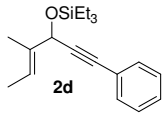
((*E*)-2,6-Dimethyl-1-phenylhepta-1,6-dien-4-yn-3-yloxy)triethylsilane (**2c**).



2c

Prepared according to the previous procedure using **1c** (0.92 mmol, 193 mg), 2,6-lutidine (2.76 mmol, 0.32 mL), triethylsilyl trifluoromethanesulfonate (1.38 mmol, 0.31 mL) to afford **2c** (283 mg, yield 95%) as a pale yellow oil: R_f 0.78 (hexane:EtOAc 4:1); IR (film) 3061, 3027, 2956, 2913, 2877, 2204, 1721, 1679, 1616, 1454, 1413, 1239, 1072, 1005, 746, 699 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.35-7.27 (m, 4H), 7.24-7.19 (m, 1H), 6.65 (s, 1H), 5.29 (m, 1H), 5.22 (app. quintet, *J* = 1.6 Hz, 1H), 5.02 (br s, 1H), 1.97 (d, *J* = 1.2 Hz, 3H), 1.90 (dd, *J* = 1.4, 1.0 Hz, 3H), 1.01 (t, *J* = 7.8 Hz, 9H), 0.71 (m, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.8, 137.8, 129.2, 128.3, 126.8, 126.7, 125.8, 122.0, 88.4, 86.5, 68.8, 23.5, 14.3, 7.0, 5.12; HRMS (EI, M^+) for $C_{21}H_{30}OSi$ calcd 326.2066, found: m/z 326.2063.

((*E*)-4-Methyl-1-phenylhex-4-en-1-yn-3-yloxy)triethylsilane (**2d**).

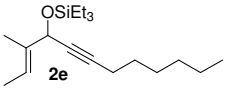


2d

Prepared according to the previous procedure using **1d** (1.38 mmol, 258 mg), 2,6-lutidine (4.14 mmol, 0.48 ml), triethylsilyl trifluoromethanesulfonate (2.07 mmol, 0.47 ml) to afford **2d** (296 mg, yield 72%) as a pale yellow oil: R_f 0.80 (hexane:EtOAc 4:1); IR (film) 3081, 3033, 2955, 2937, 2913, 2876, 2202, 1689,1672, 1632, 1598, 1490, 1458, 1317, 1239, 1055, 1005, 754, 690 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.48-7.44 (m, 2H), 7.35-7.31 (m, 3H), 5.74 (qqd, *J* = 6.4, 1.2, 1.2 Hz, 1H), 5.01 (br s, 1H), 1.82 (app. t, *J* = 1.0 Hz, 3H), 1.70 (dd, *J* = 6.8, 0.8 Hz, 3H), 1.04 (t, *J* = 7.8 Hz, 9H),

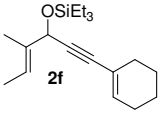
0.77-0.71 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 135.8, 131.8, 128.4, 128.3, 123.4, 121.2, 89.9, 85.0, 68.8, 13.5, 12.3, 7.0, 5.1; HRMS (EI, M^+) for $C_{19}H_{28}OSi$ calcd 300.1910, found: m/z 300.1906.

((*E*)-3-Methyldodec-2-en-5-yn-4-yloxy)triethylsilane (**2e**).



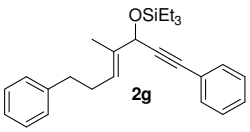
Prepared according to the previous procedure using **1e** (1.46 mmol, 284 mg), 2,6-lutidine (4.38 mmol, 0.50 ml), triethylsilyl trifluoromethanesulfonate (2.19 mmol, 0.49 ml) to afford **2e** (427 mg, 95%) as a pale yellow oil: R_f 0.86 (hexane:EtOAc 4:1); IR (microscope) 2955, 2934, 2876, 2861, 2229, 1458, 1379, 1093, 1045, 1005, 849, 743, 728 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.60 (qqd, *J* = 6.7, 1.4, 1.4 Hz, 1H), 4.72 (br s, 1H), 2.20 (td, *J* = 7.0, 2.0 Hz, 2H), 1.70 (app. t, *J* = 1.0 Hz, 3H), 1.61 (dd, *J* = 7.2, 1.0 Hz, 3H), 1.54-1.47 (m, 2H), 1.42-1.22 (m, 6H), 0.96 (t, *J* = 8.0 Hz, 9H), 0.88 (t, *J* = 7.0 Hz, 3H), 0.68-0.61 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.4, 120.5, 85.7, 80.6, 68.5, 31.5, 28.8, 28.8, 22.8, 19.0, 14.2, 13.4, 12.1, 7.0, 5.0; HRMS (EI, M^+) for $C_{19}H_{36}OSi$ calcd 308.2535, found: m/z 308.2541.

((4*E*)-1-cyclohex-1-enyl-4-methylhex-4-en-1-yn-3-yloxy)triethylsilane (**2f**).



Prepared according to the previous procedure using **1f** (2.42 mmol, 461 mg), 2,6-lutidine (7.26 mmol, 0.84 mL), triethylsilyl trifluoromethanesulfonate (3.63 mmol, 0.82 mL) to afford **2f** (562 mg, yield 77%) as a pale yellow oil: R_f 0.80 (hexane:EtOAc 4:1); IR (microscope) 2953, 2876, 2216, 2188, 1718, 1673, 1457, 1239, 1074, 1006, 842, 742 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.06 (app. quintet, *J* = 2.0 Hz, 1H), 6.62 (qqd, *J* = 6.6, 1.2, 1.2 Hz, 1H), 4.84 (br s, 1H), 2.12-2.04 (m, 4H), 1.71 (app. t, *J* = 1.0 Hz, 3H), 1.62 (dd, *J* = 6.8, 0.8 Hz, 3H), 1.59-1.53 (m, 4H), 0.97 (t, *J* = 7.8 Hz, 9H), 0.69-0.62 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 135.8, 134.4, 120.5, 120.4, 86.7, 86.6, 68.5, 29.0, 25.5, 22.2, 21.5, 13.2, 11.9, 6.80, 4.87; HRMS (EI, M^+) for $C_{19}H_{32}OSi$ calcd 304.2222, found: m/z 304.2220.

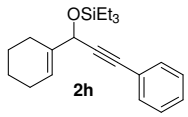
((*E*)-4-Methyl-1,7-diphenylhept-4-en-1-yn-3-yloxy)triethylsilane (**2g**).



Prepared according to the previous procedure using **1g** (2.83 mmol, 784 mg), 2,6-lutidine (8.49 mmol, 0.98 mL), triethylsilyl trifluoromethanesulfonate (4.25 mmol, 0.96 mL) to afford **2g** (999 mg, yield 91%) as a pale yellow oil: R_f 0.80 (hexane:EtOAc 4:1); IR (film) 3084, 3027, 2955, 2912, 2876, 2202, 1714, 1665, 1602, 1490, 1454, 1239, 1070, 1005, 747, 698 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$)

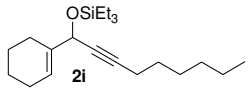
δ 7.45-7.42 (m, 2H), 7.33-7.27 (m, 5H), 7.23-7.19 (m, 3H), 5.69 (t, *J* = 7.2 Hz, 1H), 4.97 (s, 1H), 2.72 (t, *J* = 7.6 Hz, 2H), 2.41 (app. q, *J* = 7.6 Hz, 2H), 1.75 (s, 3H), 1.02 (t, *J* = 8.0 Hz, 9H), 0.74-0.67 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.0, 135.5, 131.5, 128.4, 128.2, 128.1, 128.0, 125.7, 125.5, 123.1, 89.6, 84.7, 68.4, 35.5, 29.6, 12.3, 6.8, 4.9; HRMS (EI, M^+) for $C_{26}H_{34}OSi$ calcd 390.2379, found: m/z 390.2380.

(1-Cyclohexenyl-3-phenylprop-2-ynyloxy)triethylsilane (**2h**).



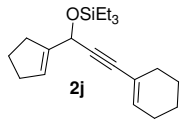
Prepared according to the previous procedure using **1h** (2.25 mmol, 479 mg), 2,6-lutidine (6.75 mmol, 0.785 mL), triethylsilyl trifluoromethanesulfonate (3.38 mmol, 0.76 mL) to afford **2h** (697 mg, yield 95%) as a pale yellow oil: R_f 0.83 (hexane:EtOAc 4:1); IR (microscope) 3060, 2939, 2875, 2201, 1717, 1663, 1598, 1449, 1289, 1115, 1070, 1003, 757, 690 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48-7.45 (m, 2H), 7.34-7.31 (m, 3H), 5.97 (br s, 1H), 5.00 (br s, 1H), 2.26-2.20 (m, 2H), 2.13-2.10 (m, 2H), 1.76-1.69 (m, 2H), 1.67-1.61 (m, 2H), 1.06 (t, *J* = 8.0 Hz, 9H), 0.78-0.72 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.7, 131.8, 128.4, 128.3, 123.6, 123.4, 89.8, 85.1, 67.6, 25.3, 24.5, 22.8, 22.6, 7.1, 5.1; HRMS (EI, M^+) for $C_{21}H_{30}OSi$ calcd 326.2066, found: m/z 326.2063.

(1-Cyclohexenylnon-2-ynyloxy)triethylsilane (**2i**).



Prepared according to the previous procedure using **1i** (2.95 mmol, 651 mg), 2,6-lutidine (8.85 mmol, 1.0 mL), triethylsilyl trifluoromethanesulfonate (4.43 mmol, 1.0 mL) to afford **2i** (927 mg, yield 94%) as a pale yellow oil: R_f 0.84 (hexane:EtOAc 4:1); IR (microscope) 2954, 2933, 2875, 2859, 2838, 2225, 1644, 1458, 1413, 1326, 1239, 1127, 1057, 1035, 1005, 742, 728 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.83 (br s, 1H), 4.73 (br s, 1H), 2.22 (td, *J* = 6.8, 2.0 Hz, 2H), 2.14-2.11 (m, 2H), 2.07-2.04 (m, 2H), 1.68-1.63 (m, 2H), 1.61-1.55 (m, 2H), 1.53-1.48 (m, 2H), 1.44-1.35 (m, 2H), 1.35-1.26 (m, 4H), 0.99 (t, *J* = 7.8 Hz, 9H), 0.90 (t, *J* = 6.8 Hz, 3H), 0.70-0.63 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.3, 122.9, 85.6, 80.4, 67.3, 31.5, 28.8, 28.7, 25.2, 24.3, 22.8, 22.7, 22.6, 19.0, 14.2, 7.0, 5.1; HRMS (EI, M^+) for $C_{21}H_{38}OSi$ calcd 334.2692, found: m/z 334.2688.

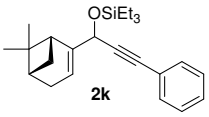
(3-Cyclohexenyl-1-cyclopentenylprop-2-ynyloxy)triethylsilane (**2j**).



Prepared according to the previous procedure using **1j** (1.81 mmol, 366 mg), 2,6-lutidine (5.43 mmol, 0.63 mL), triethylsilyl trifluoromethanesulfonate (2.72 mmol, 0.61 mL) to afford **2j** (413 mg, yield

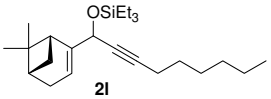
73%) as a pale yellow oil: R_f 0.78 (hexane:EtOAc 4:1); IR (microscope) 2952, 2876, 2214, 2188, 1717, 1666, 1436, 1239, 1073, 1005, 742, 728 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 6.09 (t, *J* = 2.0 Hz, 1H), 5.76 (br s, 1H), 5.10 (br s, 1H), 2.48-2.33 (m, 4H), 2.14-2.07 (m, 4H), 1.93 (app quintet, *J* = 7.6 Hz, 2H), 1.67-1.56 (m, 4H), 1.00 (t, *J* = 7.8 Hz, 9H), 0.72-0.65 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.5, 134.7, 126.6, 120.6, 86.5, 86.5, 62.4, 32.4, 31.8, 29.3, 25.8, 23.6, 22.5, 21.7, 7.0, 5.1 ; HRMS (EI, M^+) for $C_{20}H_{32}OSi$ calcd 316.2222, found: m/z 316.2219.

(1-((1*R*,5*S*)-6,6-dimethylbicyclo[3.1.1]hept-2-en-2-yl)-3-phenylprop-2-ynyloxy)triethylsilane (2k).



Prepared according to the previous procedure using **1k** (1.99 mmol, 503 mg), 2,6-lutidine (5.97 mmol, 0.69 mL), triethylsilyl trifluoromethanesulfonate (2.99 mmol, 0.67 mL) to afford **2l** (686 mg, yield 94%) as an inseparable mixture of two diastereomers (dr = 1:1): R_f 0.80 (hexane:EtOAc 4:1); Spectral data for the mixture of isomers: IR (microscope) 3081, 3055, 3033, 2986, 2952, 2913, 2876, 2831, 2224, 1598, 1490, 1465, 1381, 1314, 1238, 1125, 1083, 1064, 1051, 1002, 977, 850, 754, 690 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ7.43-7.40 (m, 4H), 7.33-7.29 (m, 6H), 5.68 (td, *J* = 3.0, 1.4 Hz, 1H), 5.64 (td, *J* = 2.8, 1.2 Hz, 1H), 5.00 (d, *J* = 1.2 Hz, 1H), 4.97 (d, *J* = 1.6 Hz, 1H), 2.48-2.43 (m, 3H), 2.40-2.35 (m, 3H), 2.32-2.25 (m, 2H), 2.14-2.11 (m, 2H), 1.33 (s, 6H), 1.28-1.24 (m, 2H), 1.03 (t, *J* = 7.8 Hz, 9H), 1.02 (t, *J* = 7.8 Hz, 9H), 0.91 (s, 3H), 0.90 (s, 3H), 0.75-0.67 (m, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.4, 137.2, 131.8, 131.8, 128.4, 128.4, 128.2, 128.2, 123.5, 123.4, 89.3, 89.3, 84.5, 84.5, 65.9, 65.4, 43.2, 42.9, 41.2, 41.1, 38.3, 38.2, 32.0, 31.8, 31.3, 31.3, 26.5, 26.4, 21.4, 21.3, 7.0, 7.0, 5.2, 5.1 [Some carbon signals are missing due to peak overlap.]; HRMS (EI, M^+) for $C_{24}H_{34}OSi$ calcd 366.2379, found: m/z 366.2377.

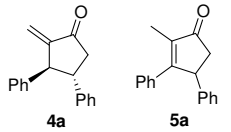
(1-((1*R*,5*S*)-6,6-dimethylbicyclo[3.1.1]hept-2-en-2-yl)non-2-ynyloxy)triethylsilane (**2l**).



Prepared according to the previous procedure using **1l** (1.53 mmol, 399.1 mg), 2,6-lutidine (4.59 mmol, 0.54 mL), triethylsilyl trifluoromethanesulfonate (2.30 mmol, 0.52 mL) to afford **2l** (500 mg, yield 87%) as an inseparable mixture of two diastereomers (dr = 1:1): R_f 0.90 (hexane:EtOAc 4:1); Spectral data for the mixture of isomers: IR (microscope) 2986, 2953, 2934, 1916, 2876, 2225, 1607, 1466, 1431, 1414, 1238, 1121, 1062, 1050, 1005, 852, 743 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 5.58-5.54 (m, 2H), 4.75-4.73 (m, 2H), 2.44-2.39 (m, 2H), 2.37-2.33 (m, 2H), 2.32-2.30 (m, 2H), 2.28-2.25 (m, 2H), 2.20 (td, *J* = 7.0, 2.0 Hz, 4H), 2.13-2.08 (m, 2H), 1.53-1.48 (m, 4H), 1.43-1.34 (m, 6H), 1.34-1.25 (m, 6H), 1.31 (s, 6H), 1.20 (t, *J* = 8.4 Hz, 2H), 1.00 (t, *J* = 8.0 Hz, 9H), 0.99 (t, *J* =

8.0 Hz, 9H), 0.91 (t, J = 6.8 Hz, 6H), 0.85 (s, 3H), 0.84 (s, 3H), 0.70-0.64 (m, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.9, 117.4, 117.1, 85.1, 79.9, 65.5, 65.1, 43.1, 42.9, 41.2, 41.1, 38.2, 38.1, 32.0, 31.8, 31.6, 31.2, 31.2, 28.8, 28.8, 28.7, 26.4, 26.4, 22.8, 21.4, 21.3, 19.0, 19.0, 14.2, 7.1, 7.0, 5.2, 5.1 [Some carbon signals are missing due to peak overlap.]; HRMS (EI, M^+) for $C_{24}H_{42}OSi$ calcd 374.3005, found: m/z 374.3008.

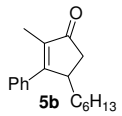
2-Methylene-3,4-diphenylcyclopentanone (**4a**) **and 2-methyl-3,4-diphenylcyclopent-2-enone** (**5a**).



4a **5a**

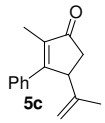
To a flame-dried round bottom flask containing a magnetic stirring bar was added siloxyenyne **2a** (0.044 mmol, 16 mg) and ether (4 mL) under Ar. The temperature of the solution was dropped to -78 ^{o}C (acetone/dry ice bath). KO*t*-Bu (0.07 mmol, 7 mg) was added in one portion and the resulting suspension was stirred vigorously followed by the immediate removal of the cooling bath. Upon consumption of **2a** as determined by thin layer chromatography, the reaction was quenched with 15% aqueous NH_4Cl and diluted with ether. The separated organic layer was washed with brine, dried over anhydrous $MgSO_4$, filtered and concentrated to give the crude siloxyallene **3a** which was used for the next step without further purification. The unpurified **3a** thus obtained was dissolved in CH_2Cl_2 (2 mL) and treated with trifluoroacetic acid (3.0 equiv based on **2a**, 0.13 mmol, 10 μL) at room temperature. After stirring for 4 h, the reaction was quenched with saturated aqueous $NaHCO_3$ and diluted with CH_2Cl_2. The separated organic layer was washed with brine, dried over anhydrous $MgSO_4$, filtered and concentrated. The crude mixture was purified by flash column chromatography (silica gel, 10% EtOAc/hexane) to provide the desired products **4a** (5.6 mg, yield 51%) and **5a** (5.1 mg, yield 47%). **4a**: R_f 0.61 (hexane:EtOAc 4:1); IR (film) 3085, 3061, 3028, 2925, 2854, 1718, 1601, 1495, 1453, 1119, 1075, 785, 698 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29-7.17 (m, 6H), 7.12-7.07 (m, 4H), 6.22 (dd, J = 3.3, 0.9 Hz, 1H), 5.04 (dd, J = 3.0, 0.8 Hz, 1H), 3.96 (ddd, J = 10.6, 3.0, 3.0 Hz, 1H), 3.41 (ddd, J = 11.2, 11.2, 7.2 Hz, 1H), 2.91 (ddd, J = 18.2, 7.6, 0.8 Hz, 1H), 2.71 (dd, J = 18.0, 12.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.7, 149.6, 141.1, 140.9, 128.9, 128.8, 128.8, 127.4, 127.3, 127.2, 120.0, 56.7, 48.9, 45.8; HRMS (EI, M^+) for $C_{18}H_{16}O$ calcd 248.1201, found: m/z 248.1201. **5a**: R_f 0.50 (hexane:EtOAc 4:1); IR (film) 3060, 3027, 2923, 2853, 1698, 1624,1495, 1454, 1443, 1378, 1342, 1076, 761, 698 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27-7.02 (m, 10H), 4.43 (ddq, J = 7.1, 2.0, 2.0 Hz, 1H), 3.05 (dd, J = 19.0, 7.2 Hz, 1H), 2.44 (dd, J = 19.0, 2.2 Hz, 1H), 1.97 (d, J = 2.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 208.9, 169.1, 142.4, 137.7, 135.3, 128.9, 128.7, 128.3, 128.0, 127.3, 126.7, 47.1, 45.1, 9.9; HRMS (EI, M^+) for $C_{18}H_{16}O$ calcd 248.1201, found: m/z 248.1198.

4-Hexyl-2-methyl-3-phenylcyclopent-2-enone (**5b**).



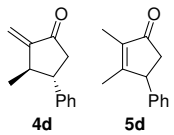
As described for the synthesis of siloxyallene **3a**, compound **2b** (0.18 mmol, 69 mg) in 3 mL ether was reacted with KO*t*-Bu (0.27 mmol, 30 mg) followed by workup as described above to give crude **3b**. The desired Nazarov product was then prepared according to the previous procedure using trifluoroacetic acid (0.54 mmol, 40 μL) to afford **5b** (38 mg, yield 82%) as a pale yellow oil: R_f 0.65 (hexane:EtOAc 4:1); IR (microsope) 3058, 3030, 2955, 2927, 2855, 1702, 1629, 1454, 1379, 1342, 1075, 777, 698 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.48-7.44 (m, 2H), 7.42-7.25 (m, 3H), 3.26 (m, 1H), 2.69 (dd, *J* = 18.8, 6.4 Hz, 1H), 2.22 (dd, *J* = 18.8, 2.0 Hz, 1H), 1.84 (d, *J* = 1.6 Hz, 3H), 1.58-1.51 (m, 2H), 1.28-1.10 (m, 8H), 0.833 (t, *J* = 6.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 209.3, 172.0, 136.9, 135.9, 129.1, 128.8, 127.8, 41.2, 40.8, 33.9, 31.8, 29.4, 27.2, 22.7, 14.2, 9.6; HRMS (EI, M^+) for $C_{18}H_{24}O$ calcd 256.1827, found: m/z 256.1826.

2-Methyl-3-phenyl-4-(prop-1-en-2-yl)cyclopent-2-enone (**5c**).



As described for the synthesis of siloxyallene **3a**, compound **2c** (0.57 mmol, 188 mg) in 4 mL ether was reacted with KO*t*-Bu (0.86 mmol, 96 mg) followed by workup as described above to give crude **3c**. The desired Nazarov product was then prepared according to the previous procedure using trifluoroacetic acid (1.71 mmol, 127 μL) to afford **5c** (43 mg, yield 35%) as a pale yellow oil: R_f 0.51 (hexane:EtOAc 4:1); IR (microsope) 3074, 3024, 2924, 2856, 1699, 1648, 1624, 1442, 1377, 1340, 1277, 894, 733, 696 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.43-7.38 (m, 5H),4.83-4.82 (m, 1H), 4.74 (app. quintet, *J* = 1.6 Hz, 1H), 4.01 (dt, *J* = 7.2, 2.0 Hz, 1H), 2.78 (dd, *J* = 19.0, 7.0 Hz, 1H), 2.35 (dd, *J* = 18.8, 2.0 Hz, 1H), 1.94 (d, *J* = 2.0 Hz, 3H), 1.25 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 208.7, 168.7, 144.8, 138.2, 135.6, 129.4, 128.6, 128.2, 114.2, 48.9, 40.9, 18.3, 10.1; HRMS (EI, M^+) for $C_{15}H_{16}O$ calcd 212.1201, found: m/z 212.1199.

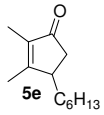
3-Methyl-2-methylene-4-phenylcyclopentanone (**4d**) **and 2,3-Dimethyl-4-phenylcyclopent-2-enone** (**5d**).



As described for the synthesis of siloxyallene **3a**, compound **2d** (0.56 mmol, 170 mg) in 3 mL ether was reacted with KO*t*-Bu (0.84 mmol, 94 mg) followed by workup as described above to give crude **3d**. The desired Nazarov product was then prepared according to the previous procedure using trifluoroacetic acid (1.68 mmol, 125 μL) to afford **4d** (10 mg, yield 10%) and **5d** (32 mg, yield 30%).

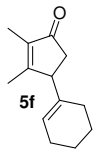
4d: R_f 0.64 (hexane:EtOAc 4:1); IR (film) 3061, 3029, 2691, 2926, 2874, 2853, 1726, 1641, 1602, 1495, 1454, 1376, 1221, 1169, 1123, 1074, 759, 700 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.40-7.36 (m, 2H), 7.31-7.27 (m, 3H), 6.13 (dd, *J* = 2.8, 0.8 Hz, 1H), 5.29 (d, *J* = 2.4 Hz, 1H), 2.84-2.74 (m, 3H), 2.59-2.51 (m, 1H), 1.18 (d, *J* = 6.0 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.5, 150.4, 141.8, 128.9, 127.5, 127.2, 116.6, 48.6, 46.3, 44.2, 16.3; HRMS (EI, M^+) for $C_{13}H_{14}O$ calcd 186.1045, found: m/z 186.1041. **5d**: R_f 0.51 (hexane:EtOAc 4:1); IR (microscope) 3062, 3027, 2961, 2923, 1701, 1648, 1601, 1494, 1454, 1384, 1323, 1073, 764, 702 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.36-7.32 (m, 2H), 7.29-7.25 (m, 1H), 7.12-7.09 (m, 2H), 3.83 (br d, *J* = 6.4 Hz, 1H), 2.92 (dd, *J* = 19.0, 7.0 Hz, 1H), 2.38 (dd, *J* = 18.8, 2.4 Hz, 1H), 1.84 (q, *J* = 0.8 Hz, 3H), 1.81 (q, *J* = 0.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 209.2, 171.7, 142.3, 129.1, 129.1, 127.5, 127.2, 49.4, 44.7, 15.7, 8.4; HRMS (EI, M^+) for $C_{13}H_{14}O$ calcd 186.1045, found: m/z 186.1042.

4-Hexyl-2,3-dimethylcyclopent-2-enone (**5e**).



As described for the synthesis of siloxyallene **3a**, compound **2e** (0.58 mmol, 180 mg) in 5 mL ether was reacted with KO*t*-Bu (0.87 mmol, 98 mg) followed by workup as described above to give crude **3e**. The desired Nazarov product was then prepared according to the previous procedure using trifluoroacetic acid (1.74 mmol, 129 μL) to afford **5e** (51 mg, 45%) as a pale yellow oil: R_f 0.58 (hexane:EtOAc 4:1); IR (microscope) 2956, 2927, 2856, 1702, 1648, 1457, 1410, 1384, 1325, 1072 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 2.64-2.60 (m, 1H), 2.48 (dd, *J* = 18.4, 6.4 Hz, 1H), 2.67 (dd, *J* = 18.6, 1.8 Hz, 1H), 1.98 (s, 3H), 1.78-1.73 (m, 2H), 1.67 (q, *J* = 0.8 Hz, 3H), 1.35-1.21 (m, 8H), 0.88 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 209.0, 172.9, 136.2, 42.7, 40.5, 32.8, 31.7, 9.3, 27.0, 22.5, 15.1, 14.0, 7.9; HRMS (EI, M^+) for $C_{13}H_{22}O$ calcd 194.1671, found: m/z 194.1670.

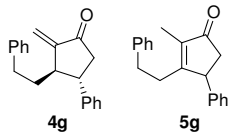
4-Cyclohexenyl-2,3-dimethylcyclopent-2-enone (**5f**).



As described for the synthesis of siloxyallene **3a**, compound **2f** (0.48 mmol, 147 mg) in 3 mL ether was reacted with KO*t*-Bu (0.72 mmol, 82 mg) followed by workup as described above to give crude **3f**. The desired Nazarov product was then prepared according to the previous procedure using trifluoroacetic acid (1.44 mmol, 107 μL) to afford **5f** (41 mg, yield 45%) as a pale yellow oil: R_f 0.60 (hexane:EtOAc 4:1); IR (microscope) 2927, 2858, 2837, 1701, 1649, 1573, 1437, 1383, 1072 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 5.57 (m, 1H), 3.23 (br d, *J* = 6.8 Hz, 1H), 2.51 (dd, *J* = 19.0, 7.0 Hz, 1H), 2.18 (dd, *J* = 19.0, 1.8 Hz, 1H), 2.04-2.02 (m, 2H), 1.89 (s, 3H), 1.70 (q, *J* = 1.2 Hz, 3H),

1.61-1.54 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 209.0, 171.5, 137.2, 136.6, 125.0, 51.2, 40.0, 25.3, 23.7, 22.7, 22.4, 15.1, 8.1; HRMS (EI, M^+) for $C_{13}H_{18}O$ calcd 190.1358, found: m/z 190.1358.

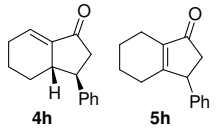
2-Methylene-3-phenethyl-4-phenylcyclopentanone (**4g**) **and 2-methyl-3-phenethyl-4-phenylcyclopent-2-enone** (**5g**).



4g **5g**

As described for the synthesis of siloxyallene **3a**, compound **2g** (0.31 mmol, 123 mg) in 5 mL ether was reacted with KO*t*-Bu (0.46 mmol, 52 mg) followed by workup as described above to give crude **3g**. The desired Nazarov product was then prepared according to the previous procedure using trifluoroacetic acid (0.93 mmol, 69 μL) to afford **4g** (24 mg, yield 28%) and **5g** (33 mg, 39%). **4g**: R_f 0.53 (hexane:EtOAc 4:1); IR (microscope) 3084, 3061, 3027, 3003, 2927, 2860, 1725, 1637, 1602, 1495, 1453, 1370, 1222, 1171, 1158, 1030, 753, 700 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37-7.17 (m, 8H), 7.09-7.06 (m, 2H), 6.21 (d, *J* = 2.4 Hz, 1H), 5.40 (d, *J* = 2.4 Hz, 1H), 3.22 (app. q, *J* = 8.7 Hz, 1H), 3.00-2.94 (m, 1H), 2.84 (dd, *J* = 18.4, 7.9 Hz, 1H), 2.64-2.60 (m, 2H), 2.52 (dd, *J* = 18.4, 9.8 Hz, 1H), 2.01-1.94 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.41, 148.4, 142.9, 141.6, 128.8, 128.4, 128.2, 127.1, 126.9, 125.9, 117.4, 48.1, 46.2, 45.5, 34.2, 32.7; HRMS (EI, M^+) for $C_{20}H_{20}O$ calcd 276.1514, found: m/z 276.1514. **5g**: R_f 0.47 (hexane:EtOAc 4:1); IR (microscope) 3084, 3026, 3002, 2924, 2860, 1700, 1643, 1601, 1494, 1454, 1325, 1076, 756, 701 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37-7.32 (m, 2H), 7.30-7.25 (m, 3H), 7.23-7.19 (m, 1H), 7.11-7.06 (m, 4H), 3.85 (dd, *J* = 6.8, 1.6 Hz, 1H), 2.89 (dd, *J* = 18.8, 7.0 Hz, 1H), 2.80-2.68 (m, 2H), 2.64-2.56 (m, 1H), 2.39 (dd, *J* = 18.9, 2.3 Hz, 1H), 2.39-2.31 (m, 1H), 1.73 (d, *J* = 1.9 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 209.1, 173.8, 141.8, 140.6, 137.5, 128.9, 128.4, 128.1, 127.4, 127.1, 126.3, 47.2, 44.5, 33.2, 31.0, 8.1; HRMS (EI, M^+) for $C_{20}H_{20}O$ calcd 276.1514, found: m/z 276.1514.

2,3,3a,4,5,6-Hexahydro-3-phenylinden-1-one (**4h**) **and 2,3,4,5,6,7-hexahydro-3-phenylinden-1-one** (**5h**).

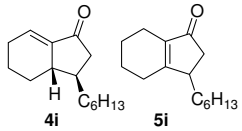


4h **5h**

As described for the synthesis of siloxyallene **3a**, compound **2h** (0.06 mmol, 19 mg) in 3 mL ether was reacted with KO*t*-Bu (0.09 mmol, 10 mg) followed by workup as described above to give crude **3h**. The desired Nazarov product was then prepared according to the previous procedure using trifluoroacetic acid (0.18 mmol, 13 μL) to afford **4h** (8 mg, yield 65%) and **5h** (2 mg, 14%). **4h**: R_f 0.52 (hexane:EtOAc 4:1); IR (microscope) 3061, 3028, 2931, 2858, 1719, 1652, 1601, 1453, 1247, 1223, 1187, 930, 749, 700 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40-7.35 (m, 2H), 7.32-7.27 (m, 3H),

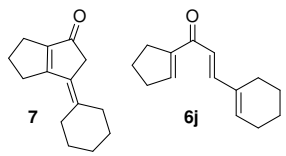
6.81 (app. q, *J* = 2.4 Hz, 1H), 2.83 (ddd, *J* = 12.6, 10.6, 7.0 Hz, 1H), 2.71-2.63 (m, 1H), 2.68 (dd, *J* = 17.8, 7.4 Hz, 1H), 2.55 (dd, *J* = 17.8, 12.6 Hz, 1H), 2.40-.32 (m, 1H), 2.86-2.17 (m, 1H), 2.04 (dq, *J* = 12.0, 3.2 Hz, 1H), 1.92-1.86 (m, 1H), 1.55-1.43 (m, 1H), 1.19-1.09 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.7, 141.8, 141.3, 133.2, 128.9, 127.3, 127.1, 48.4, 46.3, 46.0, 27.3, 25.7, 21.8; HRMS (EI, M^+) for $C_{15}H_{16}O$ calcd 212.1201, found: m/z 212.1203. **5h**: R_f 0.48 ((hexane:EtOAc 4:1); IR (microscope) 3061, 3027, 2930, 2858, 1698, 1645, 1453, 1390, 1277, 1245, 766, 701 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37-7.31 (m, 2H), 7.28-7.24 (m, 1H), 7.12-7.10 (m, 2H), 3.85 (br d, *J* = 6.8 Hz, 1H), 2.93 (dd, *J* = 18.8, 7.2 Hz, 1H), 2.38 (dd, *J* = 18.8, 2.4 Hz, 1H), 2.27-2.25 (m, 2H), 2.10-2.07 (m, 2H), 1.72-1.66 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 208.4, 175.4, 142.1, 139.5, 129.1, 127.5, 127.2, 48.2, 45.1, 26.7, 22.4, 21.8, 20.3; HRMS (EI, M^+) for $C_{15}H_{16}O$ calcd 212.1201, found: m/z 212.1197.

3-Hexyl-2,3,3a,4,5,6-hexahydroinden-1-one (**4i**) **and 3-hexyl-2,3,4,5,6,7-hexahydroinden-1-one** (**5i**).



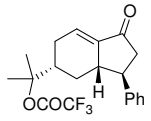
As described for the synthesis of siloxyallene **3a**, compound **2i** (0.28 mmol, 96 mg) in 4 mL ether was reacted with KO*t*-Bu (0.42 mmol, 48 mg) followed by workup as described above to give crude **3i**. The desired Nazarov product was then prepared according to the previous procedure using trifluoroacetic acid (0.84 mmol, 64 μL) to afford **4i** (21 mg, yield 33%) and **5i** (12 mg, yield 20%). **4i**: R_f 0.61 (hexane:EtOAc 4:1); IR (microscope) 2927, 2856, 1720, 1655, 1456, 1248, 1170, 1139 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.67 (br s, 1H), 2.47 (dd, *J* = 17.8, 7.0 Hz, 1H), 2.44-2.38 (m, 1H), 2.33-2.23 (m, 1H), 2.22-2.14 (m, 3H), 1.74-1.44 (m, 6H), 1.36-1.27 (m, 8H), 0.911 (t, *J* = 6.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 206.1, 142.0, 132.0, 45.0, 44.7, 42.5, 34.4, 32.0, 29.7, 28.2, 27.8, 25.6, 22.8, 21.9, 14.3; HRMS (ESI, $[M+H]^+$) for $C_{15}H_{25}O$ calcd 221.1900, found: m/z 221.1901. **5i**: R_f 0.57 (hexane:EtOAc 4:1); IR (microscope) 2927, 2856, 1698, 1647, 1457, 1437, 1277, 1169 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 2.68 (m, 1H), 2.53 (dd, *J* = 18.6, 6.6 Hz, 1H), 2.44-2.36 (m, 1H), 2.22-2.12 (m, 3H), 2.07 (dd, *J* = 18.2, 2.2 Hz, 1H), 1.78-1.59 (m, 6H), 1.34-1.30 (m, 8H), 0.90 (t, *J* = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 208.6, 176.7, 138.6, 42.1, 41.3, 33.0, 31.9, 29.6, 27.3, 26.6, 22.8, 22.4, 21.9, 20.2, 14.2; HRMS (ESI, $[M+H]^+$) for $C_{15}H_{25}O$ calcd 221.1900, found 221.1898.

3-Cyclohexylidene-2,3,5,6-tetrahydropentalen-1(4*H*)-one (7) and **(2*E*)-3-cyclohexenyl-1-cyclopentenylprop-2-en-1-one** (**6j**).



As described for the synthesis of siloxyallene **3a**, compound **2j** (0.63 mmol, 202 mg) in 5 mL ether was reacted with KO*t*-Bu (0.95 mmol, 105 mg) followed by workup as described above to give crude **3j**. The desired Nazarov product was then prepared according to the previous procedure using trifluoroacetic acid (1.89 mmol, 140 μL) to afford **7** (35 mg, yield 28%) and **6j** (2 mg, yield 2%). **7**: R_f 0.65 (hexane:EtOAc 4:1); IR (microscope) 3346, 2929, 2878, 2861, 2818, 1691, 1659, 1635, 1450, 1394, 1277, 1229, 1162, 1098, 1058, 750 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 2.93-2.88 (m, 4H), 2.38 (t, *J* = 7.0 Hz, 2H), 2.35 (t, *J* = 5.8 Hz, 2H), 2.22-2.19 (m, 2H), 1.80-1.57 (m, 8H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.3, 163.2, 154.8, 141.3, 126.1, 35.8, 33.5, 31.7, 27.6, 26.8, 25.2, 22.3, 21.8, 20.2; HRMS (EI, H^+) for $C_{14}H_{18}O$ calcd 202.1358, found: m/z 202.1355. **6j**: R_f 0.70 (hexane:EtOAc 4:1); IR (microscope) 2928, 2856, 1729, 1650, 1620, 1610, 1588, 1449, 1365, 1297, 1168, 1001, 981 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.28 (d, *J* = 16.0 Hz, 1H), 6.78-6.76 (m, 1H), 6.60 (d, *J* = 15.6 Hz, 1H), 6.24 (s, 1H), 2.67-2.57 (m, 4H), 2.24-2.20 (m, 4H), 1.96 (quintet, *J* = 7.6 Hz, 2H), 1.78-1.62 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 188.7, 146.9, 145.7, 142.5, 139.4, 135.3, 119.2, 34.0, 31.1, 29.6, 26.6, 24.3, 22.8, 22.1; HRMS (EI, M^+) for $C_{14}H_{18}O$ calcd 202.1358, found: m/z 202.1348.

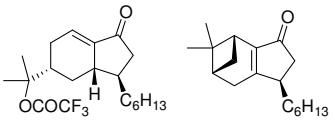
2-((3*R*,3a*S*,5*R*)-2,3,3a,4,5,6-Hexahydro-1-oxo-3-phenyl-1H-inden-5-yl)propan-2-yl 2,2,2-trifluoroacetate (9k).



(+)-**9k**

As described for the synthesis of siloxyallene **3a**, compound **2k** (1.41 mmol, 516 mg) in 5 mL ether was reacted with KO*t*-Bu (2.12 mmol, 237 mg) followed by workup as described above to give crude **3k**. The desired Nazarov product was then prepared according to the previous procedure using trifluoroacetic acid (4.23 mmol, 315 μL) to afford **9k** (193 mg, yield 45%): R_f 0.36 (hexane:EtOAc 4:1); IR (microscope) 3062, 3030, 2924, 1776, 1721, 1660, 1602, 1454, 1422, 1392, 1372, 1218, 1166, 1134, 776, 700 cm^{-1}; ^1H NMR (400 MHz, CDCl3) δ 7.40-7.37 (m, 2H), 7.31-7.29 (m, 3H), 6.78 (app. q, *J* = 2.0 Hz, 1H), 2.85 (ddd, *J* = 12.6, 10.5, 7.0 Hz, 1H), 2.80-2.77 (m, 1H), 2.72 (dd, *J* = 17.9, 7.0 Hz, 1H), 2.57 (dd, *J* = 18.2, 12.6 Hz, 1H), 2.43 (app. dq, *J* = 19.6, 4.2 Hz, 1H), 2.25 (app. tdd, *J* = 11.9, 4.9, 2.1 Hz, 1H), 2.21-2.15 (m, 1H), 2.03 (ddd, *J* = 11.9, 4.9, 2.1 Hz, 1H), 1.59 (s, 3H), 1.55 (s, 3H), 1.07 (app. td, *J* = 12.6, 11.2 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.4, 156.0 (q, *J* = 40.9 Hz), 141.0, 140.0, 131.2, 128.8, 127.0, 127.0, 114.3 (q, *J* = 285.4 Hz), 90.5, 47.8, 46.5, 45.9, 42.7, 28.1, 26.7, 23.3, 22.7; ^{19}F NMR (376 MHz, $CDCl_3$) δ -76.0; HRMS (EI, $[M-CF_3CO_2H]^+$) for $C_{18}H_{20}O$ calcd 252.1514, found: m/z 252.1512; $[\alpha]_D^{25}$: +67.16° (c 2.87, $CHCl_3$).

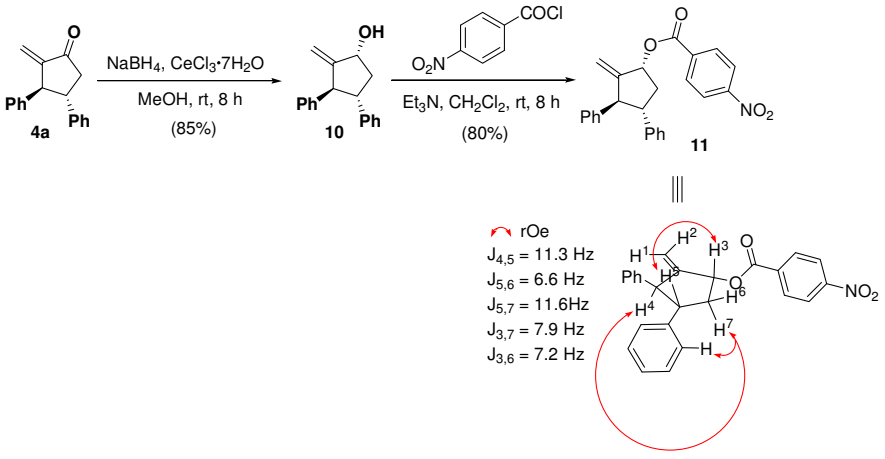
2-((1*R*,6*R*,7a*S*)-1-Hexyl-2,3,5,6,7,7a-hexahydro-3-oxo-1H-inden-6-yl)propan-2-yl 2,2,2-trifluoroacetate (**9l**) and **5l**.



(-)-**9l** (-)-**5l**

As described for the synthesis of siloxyallene **3a**, compound **2l** (0.14 mmol, 52 mg) in 3 mL ether was reacted with KO*t*-Bu (0.21 mmol, 23 mg) followed by workup as described above to give crude **3l**. The desired Nazarov product was then prepared according to the previous procedure using trifluoroacetic acid (0.42 mmol, 31 μL) to afford **9l** (7 mg, yield 13%) and **5l** (9 mg, yield 25%). **9l**: R_f 0.41 (hexane:EtOAc 4:1); IR (film) 2955, 2926, 2856, 1778, 1722, 1660, 1466, 1372, 1219, 1167, 1125, 1167, 1125, 777 cm^{-1}; ^1H NMR (400 MHz, CDCl3) δ 6.63 (dt, *J* = 4.0, 3.0 Hz, 1H), 2.50 (dd, *J* = 18.0, 6.9 Hz, 1H), 2.39-2.30 (m, 1H), 2.27-2.17 (m, 3H), 2.14-2.08 (m, 1H), 1.94 (dd, *J* = 18.0, 12.0 Hz, 1H), 1.71-1.62 (m, 2H), 1.61 (s, 3H), 1.59 (s, 3H), 1.39-1.25 (m, 9H), 1.04-0.94 (m, 1H), 0.89-0.87 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.8, 156.1 (q, *J* = 41.2 Hz), 141.8, 130.1, 114.4 (q, *J* = 285.0 Hz), 90.8, 45.0, 44.9, 42.8, 42.0, 34.1, 31.7, 29.4, 28.6, 28.0, 26.8, 22.9, 22.8, 22.5, 14.0; ^{19}F NMR (376 MHz, $CDCl_3$) δ -76.0; HRMS (EI, $[M\text{-}CF_3CO_2H]^+$) for $C_{18}H_{28}O$ calcd 260.2140, found: m/z 260.2136; $[\alpha]_D^{25}$: -44.56° (c 0.35, $CHCl_3$). **5l**: R_f 0.50 (hexane:EtOAc 4:1); IR (film) 2954, 2927, 2857, 1695, 1625, 1466, 1416, 1385, 1239, 1168 cm^{-1}; ^1H NMR (400 MHz, CDCl3) δ 2.76-2.68 (m, 1H), 2.68-2.61 (m, 2H), 2.54 (app. dt, *J* = 9.0, 2.7 Hz, 1H), 2.47 (br d, *J* = 18.8 Hz, 1H), 2.25 (app. tt, *J* = 5.6, 2.8 Hz, 1H), 2.17 (dd, *J* = 19.2, 2.0 Hz, 1H), 1.82-1.73 (m, 2H), 1.69-1.57 (m, 4H), 1.36 (s, 3H), 1.36-1.26 (m, 5H), 1.05 (d, *J* = 9.0 Hz, 1H), 0.93-0.87 (m, 3H), 0.71 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 205.4, 175.9, 148.8, 42.8, 41.0, 40.6, 37.1, 33.7, 32.4, 31.9, 31.7, 29.3, 28.3, 25.8, 22.5, 20.9, 14.0 [One aliphatic carbon signal is missing due to peak overlap.]; HRMS (EI, M^+) for $C_{18}H_{28}O$ calcd 260.2140, found: m/z 260.2143; $[\alpha]_D^{25}$: -8.62° (c 0.21, $CHCl_3$).

Preparation of ester **11** from enone **4a** for 2D TROESY study.



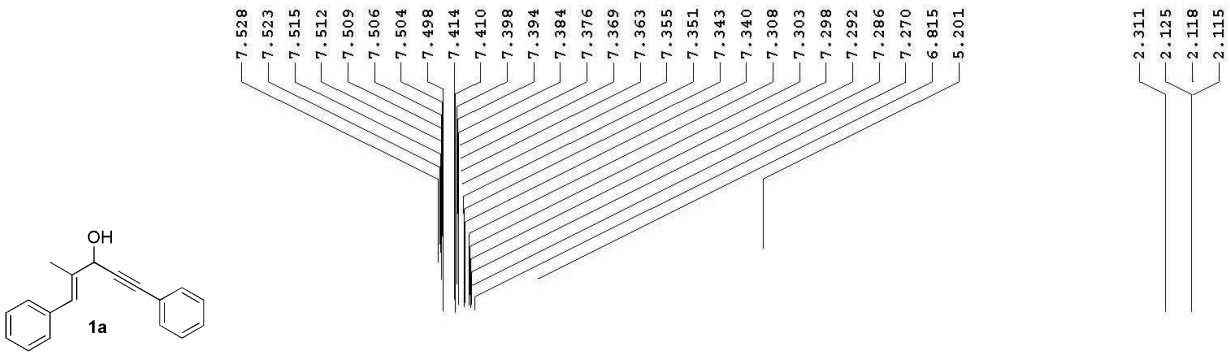
Alcohol 10: To a solution of **4a** (0.06 mmol, 15 mg) and cerium (III) chloride heptahydrate (0.06

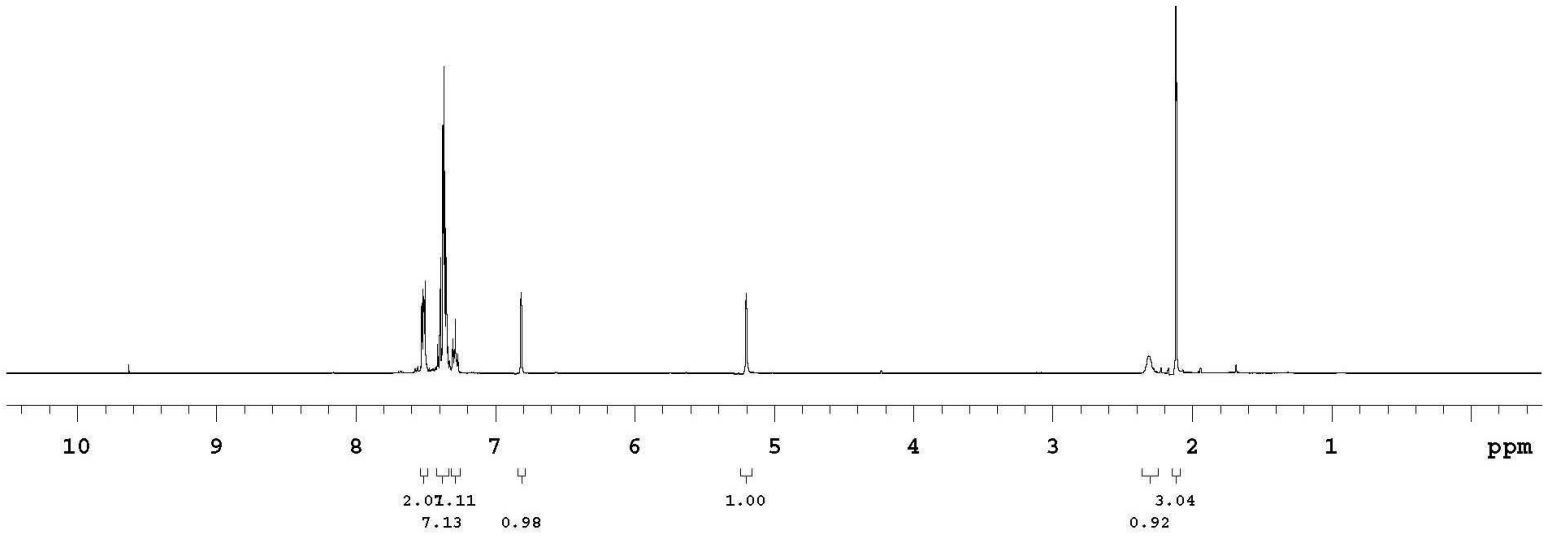
mmol, 22 mg) in 3 mL MeOH was added $NaBH_4$ (0.06 mmol, 3 mg) at room temperature. The mixture was stirred for 8 h then quenched by adding saturated NH_4Cl solution. The organic layer was separated and washed with brine, dried over anhydrous $MgSO_4$, filtered and concentrated under reduced pressure. The crude mixture was purified by flash column chromatography (hexane:EtOAc 2:1) to provide the alcohol as a single diastereomer (13 mg, yield 85%): R_f 0.25 (hexane:EtOAc 4:1); IR (microscope) 3411 (br s), 3063, 3028, 2959, 2923, 2857, 1661, 1602, 1469, 1452, 1328, 1211, 1087, 1076, 914, 905, 761, 699 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.26-7.21 (m, 3H), 7.20-7.15 (m, 3H), 7.11-7.08 (m, 2H), 7.05-7.02 (m, 2H), 5.33 (app. t, *J* = 2.4 Hz, 1H), 4.86-4.81 (m, 2H), 3.81 (app. dq, *J* = 10.8, 2.6 Hz, 1H), 3.09 (ddd, *J* = 12.4, 11.2, 6.0 Hz, 1H), 2.61 (ddd, *J* = 12.4, 6.4, 6.4 Hz, 1H), 1.95 (ddd, *J* = 12.4, 12.4, 9.6 Hz, 1H) 1.82 (br s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 158.0, 142.6, 141.9, 128.4, 128.3, 128.2, 127.3, 126.5, 126.3, 110.1, 74.7, 57.2, 50.0, 42.7; HRMS (EI, M^+) for $C_{18}H_{18}O$ calcd 250.1358, found: m/z 250.1358.

***p*-Nitrobenzoate 11**: To a solution of **10** (0.032 mmol, 8 mg) and triethylamine (0.096 mmol, 13 μL) in 3 mL CH_2Cl_2 was added 4-nitrobenzoyl chloride (0.035 mmol, 7 mg) at room temperature. The mixture was stirred for 8 h then quenched by adding 5 mL water and CH_2Cl_2 (10 mL). The organic layer was separated and washed with brine, dried over anhydrous $MgSO_4$, filtered and concentrated under reduced pressure. The crude mixture was purified by flash column chromatography (hexane:EtOAc 5:1) to provide **11** (10 mg, 80%): R_f 0.60 (hexane:EtOAc 4:1); IR (microscope) 3109, 3085, 3061, 3029, 1724, 1605, 1528, 1347, 1273, 1119, 1103 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 8.36–8.33 (m, 2H), 8.32-8.29 (m, 2H), 7.30-7.26 (m, 4H), 7.24-7.20 (m, 2H), 7.17-7.11 (m, 4H), 6.11 (app. td, *J* = 7.2, 2.2 Hz, 1H), 5.41 (dd, *J* = 3.0, 2.2 Hz, 1H), 4.92 (t, *J* = 2.6 Hz, 1H), 3.92 (app. dq, *J* = 11.0, 2.6 Hz, 1H), 3.28 (app. td, *J* = 11.6, 6.6 Hz, 1H), 2.88 (app. dt, *J* = 12.8, 7.2 Hz, 1H), 2.21 (app. td, *J* = 12.6, 8.6 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.8, 153.0, 150.8, 141.6, 141.4, 135.9, 131.0, 128.8, 128.7 128.6, 127.5, 127.0, 126.9, 123.8, 113.5, 77.5, 57.5, 50.7, 39.5; HRMS (EI, M^+) for $C_{25}H_{21}NO_4$ calcd 399.1471, found: m/z 399.1466.

Yen-Ku, YKW-1-43
400.393 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe

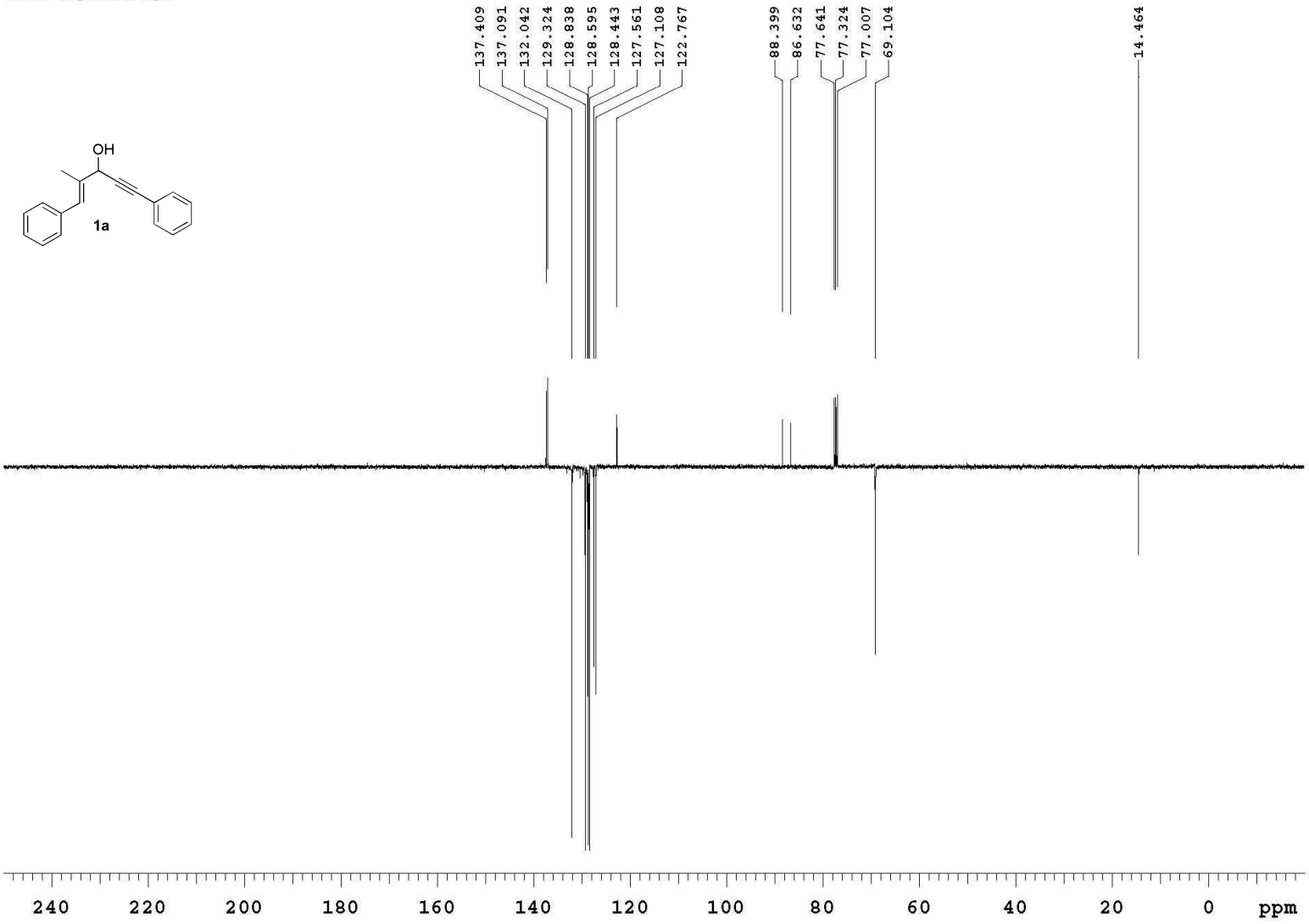
Pulse Sequence: s2pul





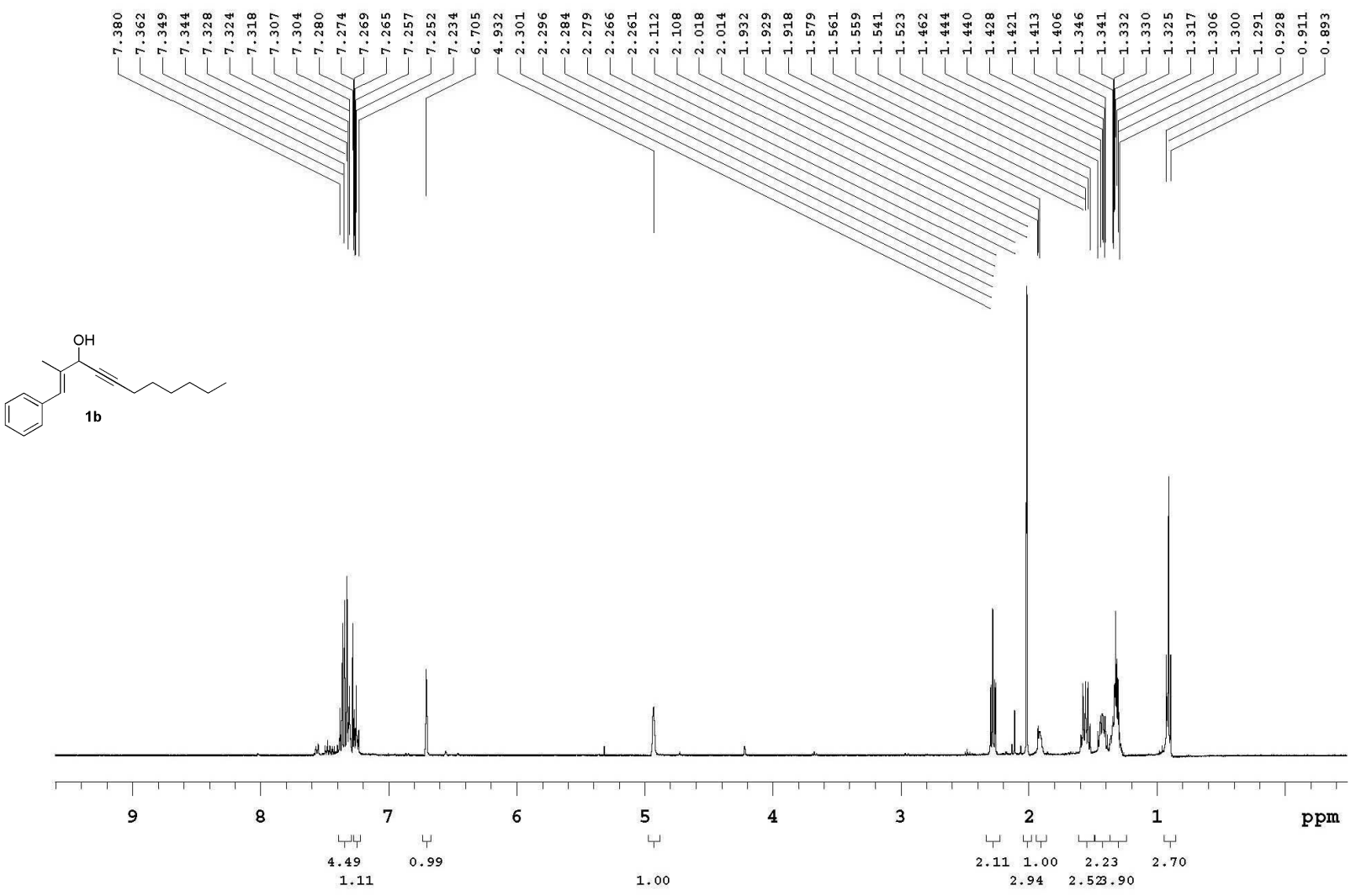
100.690 MHz C13[H1] apt in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe
date:May 9, 2019 sweep width: 27174Hz acq.time: 2.0s relax.time: 0.1s # scans: 1024 dig.res.: 0.4 Hz/pt hz/mm:113.2
spectrometer:d601 file:/mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/2009.05/2009.05.9.m4_YKW-1-43P_20:04_C13_apt.fid

Pulse Sequence: apt



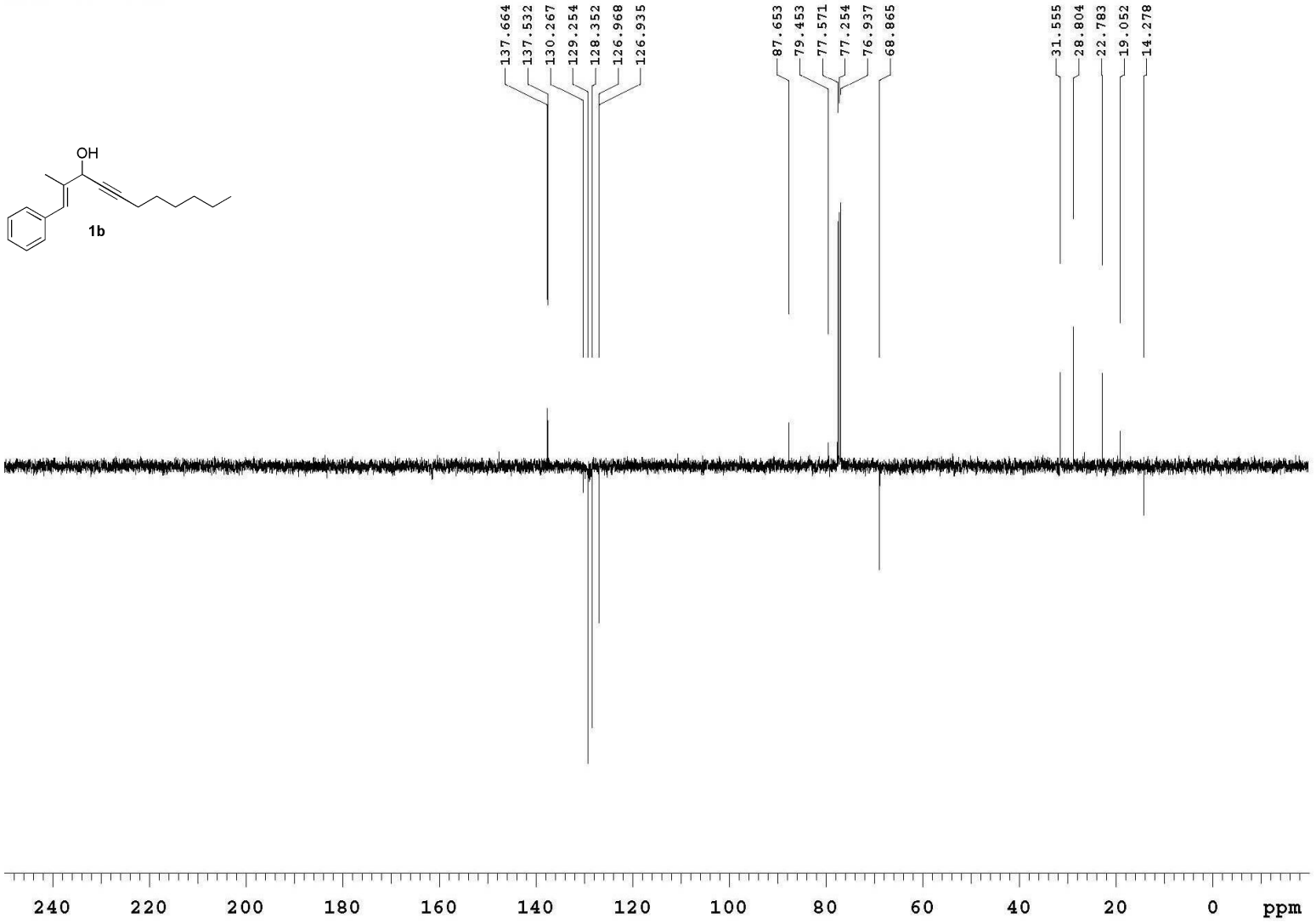
Yen-Ku, YKW-2-29-APT
400.393 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe

Pulse Sequence: s2pul



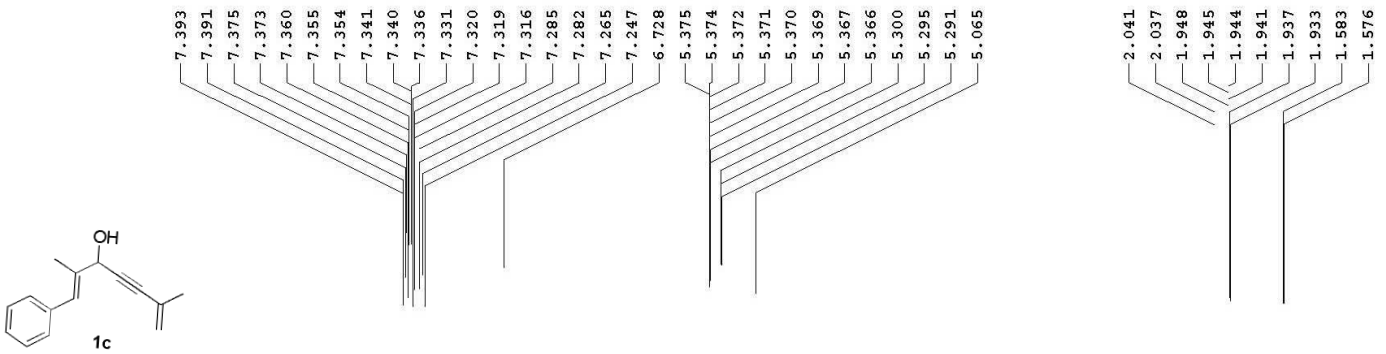
100.690 MHz C13[H1] apt in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe
date: Jul 8 2009 sweep width: 27164Hz acq.time: 2.0s relax.time: 0.1s # scans: 1024 dig.res.: 0.4 Hz/pt hz/mm:113.2
spectrometer:d601 file:/mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/2009.07/2009.07.9.m4_YKW-2-29-APT_06:29_C13_apt.fid

Pulse Sequence: apt

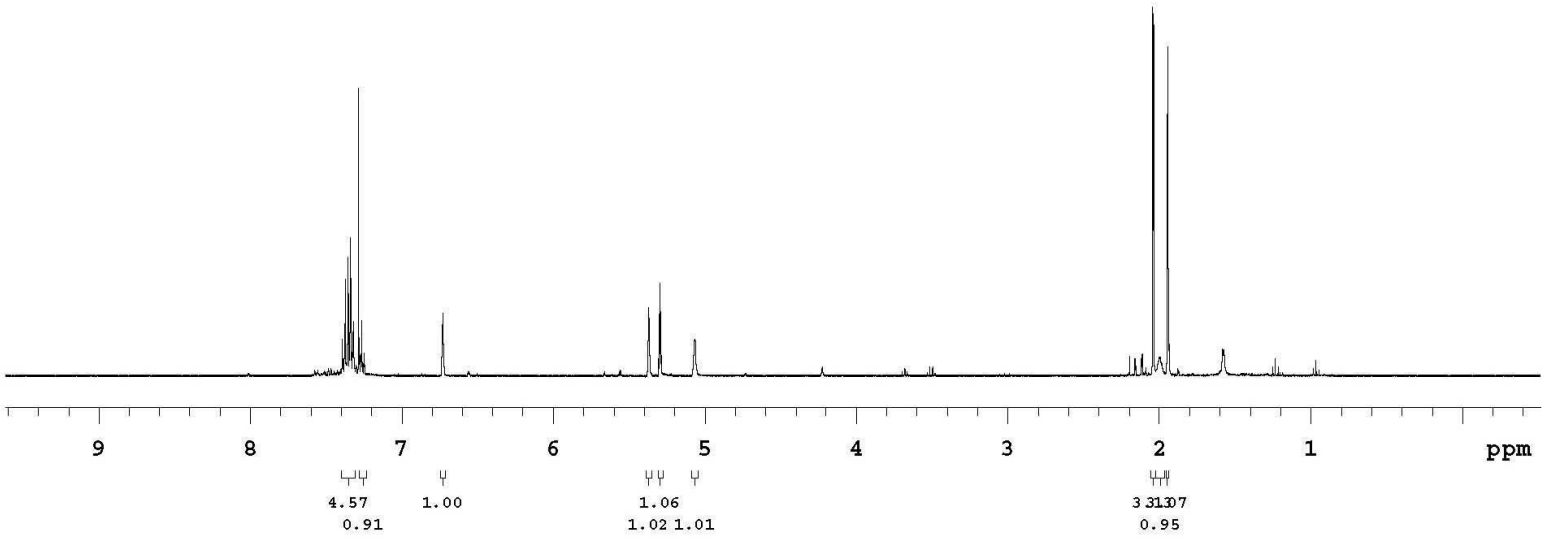


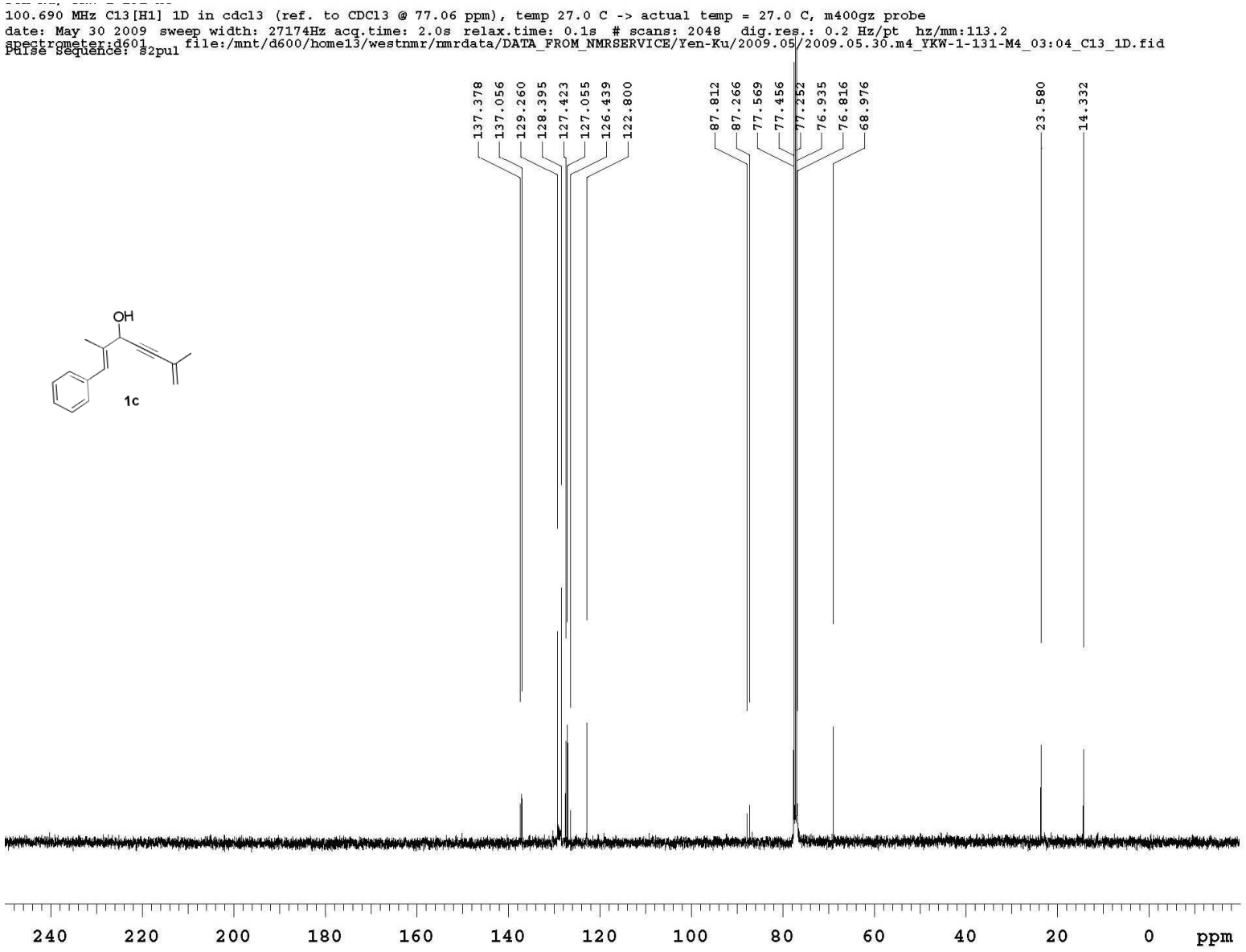
Yen-Ku, YKW-1-131-M4
400.393 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe

Pulse Sequence: s2pul



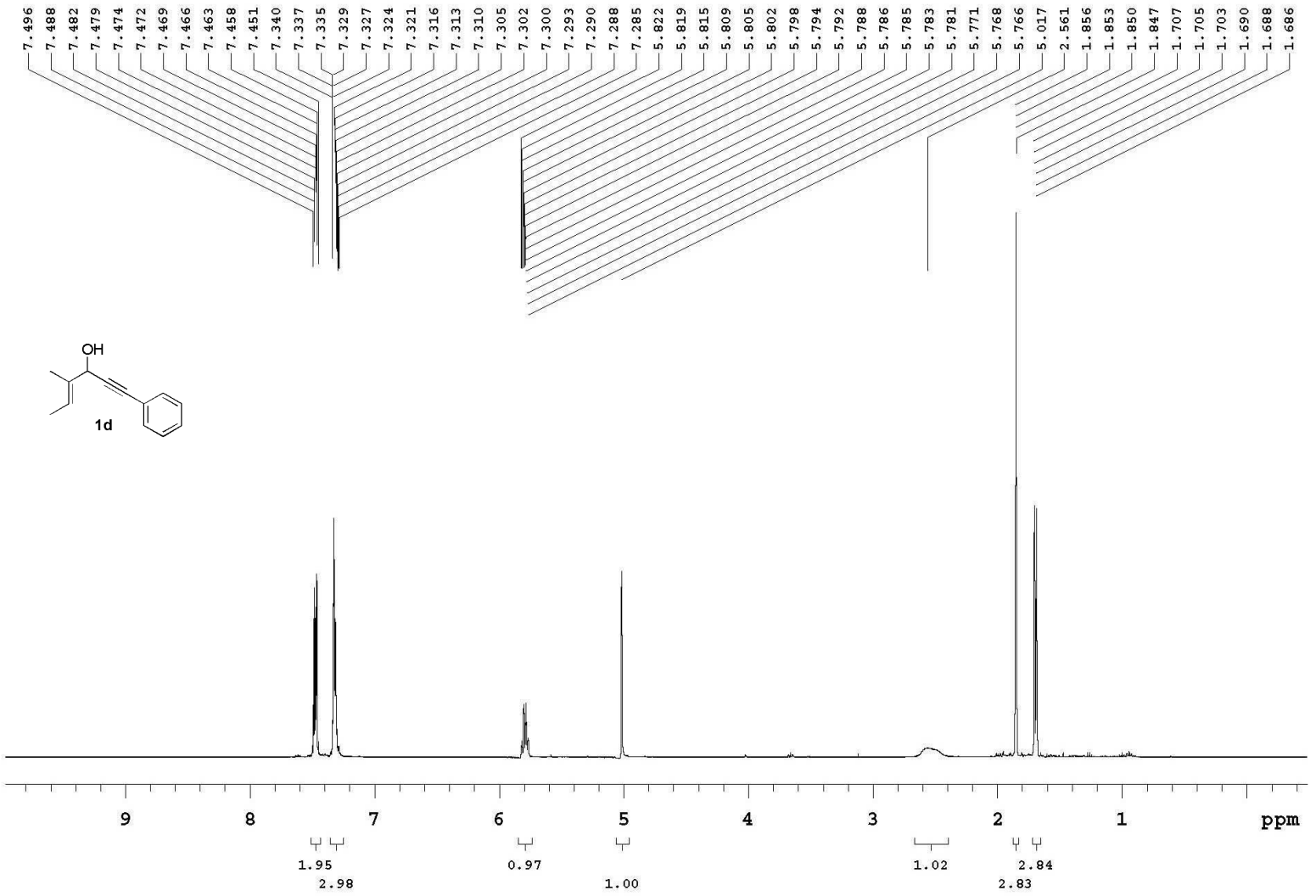
1c




100.690 MHz C13[H1] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe
date: May 30 2009 sweep width: 27174Hz acq.time: 2.0s relax.time: 0.1s # scans: 2048 dig.res.: 0.2 Hz/pt hz/mm:113.2
spectrometer:d601 file:/mnt/d600/home13/westnmr/rmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/2009.05/2009.05.30.m4_YKW-1-131-M4_03:04_C13_1D.fid
Pulse sequence: s2pul
137.378
137.056
129.260
128.395
127.423
127.055
126.439
122.800
87.812
87.266
77.569
77.456
77.252
76.935
76.816
68.976
23.580
14.332
OH
1c
240
220
200
180
160
140
120
100
80
60
40
20
0
ppm

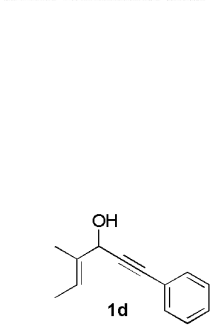
Yen-Ku, YKW-1-63-C
400.393 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe

Pulse Sequence: s2pul



100.690 MHz C13[H1] apt in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe
date:May 14 2009 sweep width: 27154Hz acq.time: 2.0s relax.time: 0.1s # scans: 600 dig.res.: 0.4 Hz/pt hz/mm:113.2
spectrometer:d601 file:/mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/2009.05/2009.05.14.m4_YKW-1-63-P_14:17_C13_apt.fid

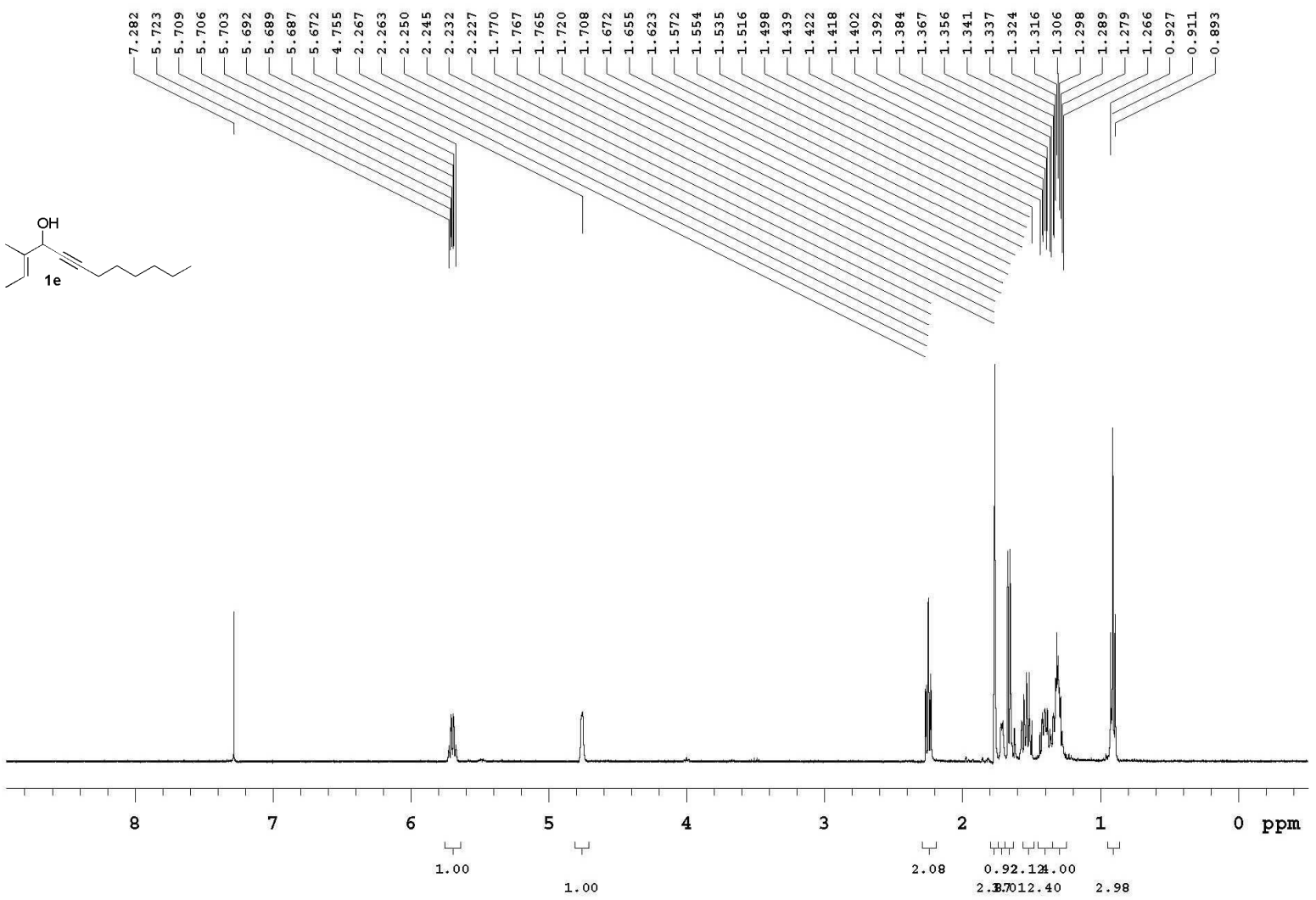
Pulse Sequence: apt



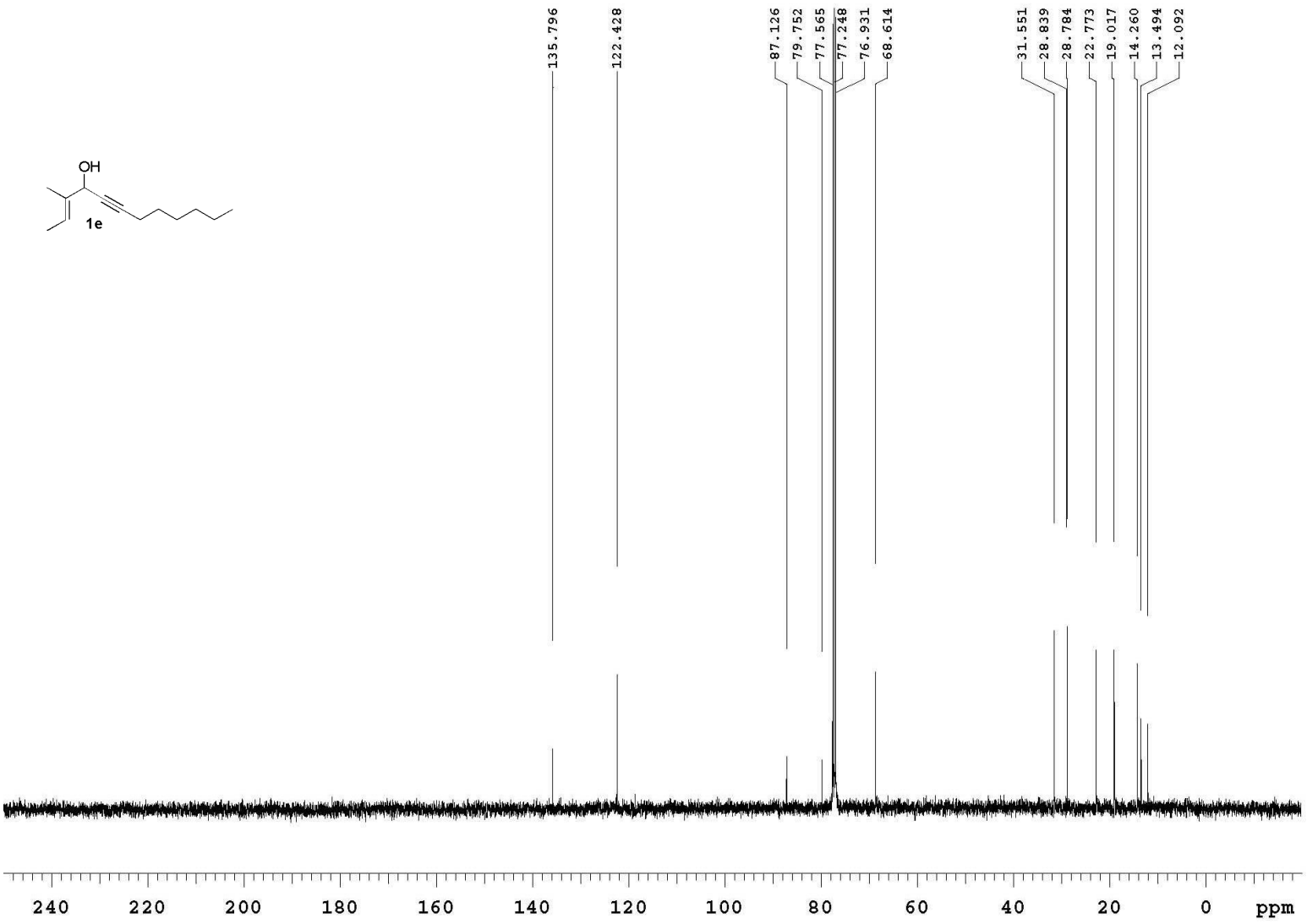
1d

Yen-Ku, YKW-2-119-m4
400.393 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe

Pulse Sequence: s2pul

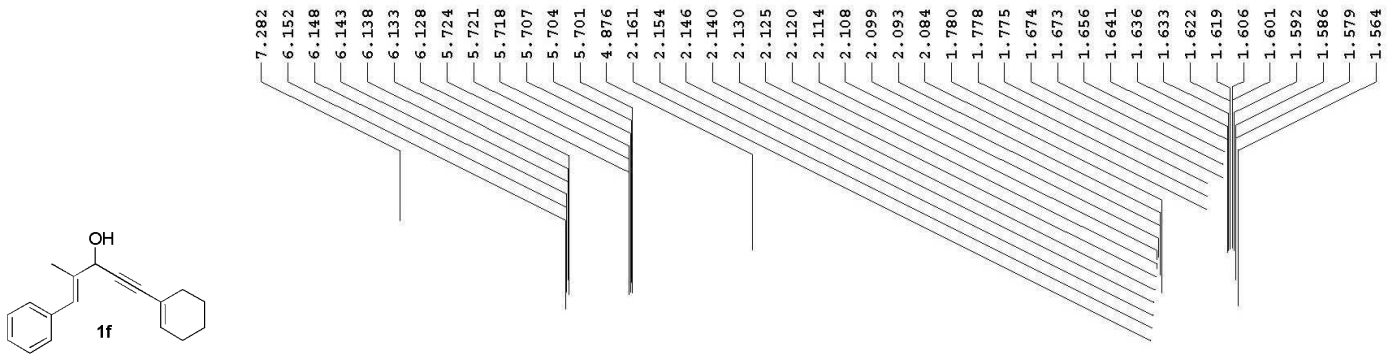


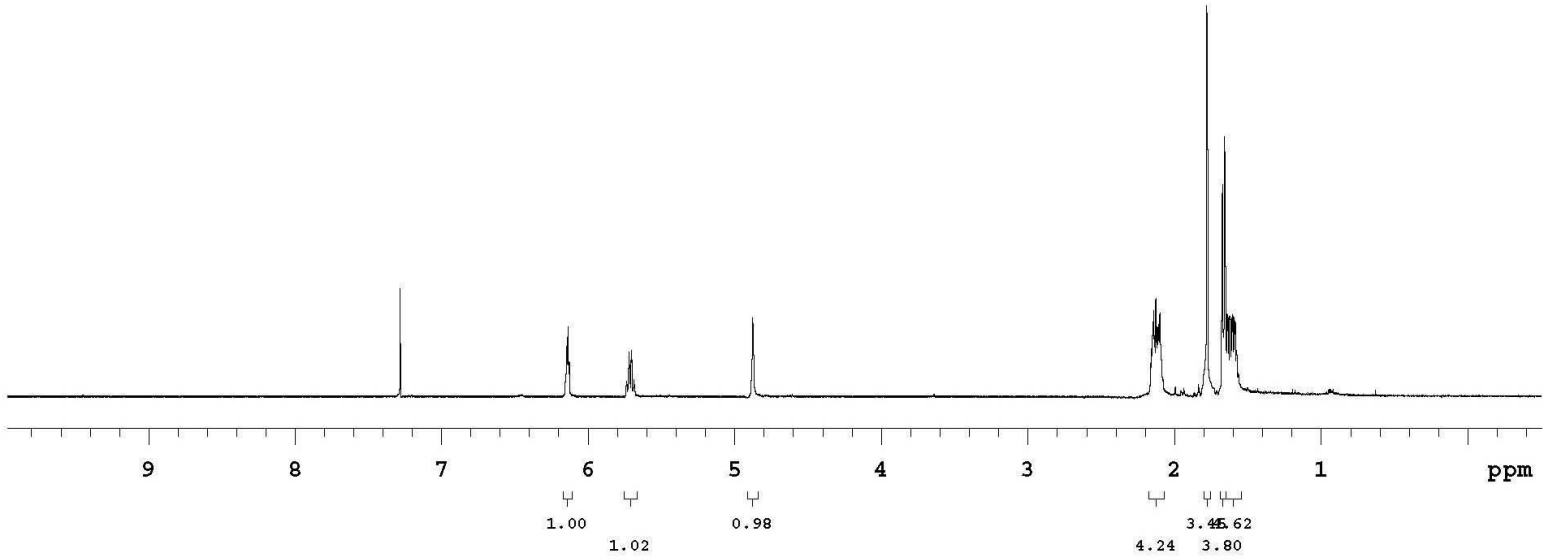
100.690 MHz C13[H1] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe
date: Aug 11 2009 sweep width: 27174Hz acq.time: 2.0s relax.time: 0.1s # scans: 1024 dig.res.: 0.2 Hz/pt hz/mm:113.2
spectrometer:d601 file:/mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/2009.08/2009.08.11.m4_YKW-2-119-m4_13:07_C13_1D.fid
Pulse sequence: s2pul



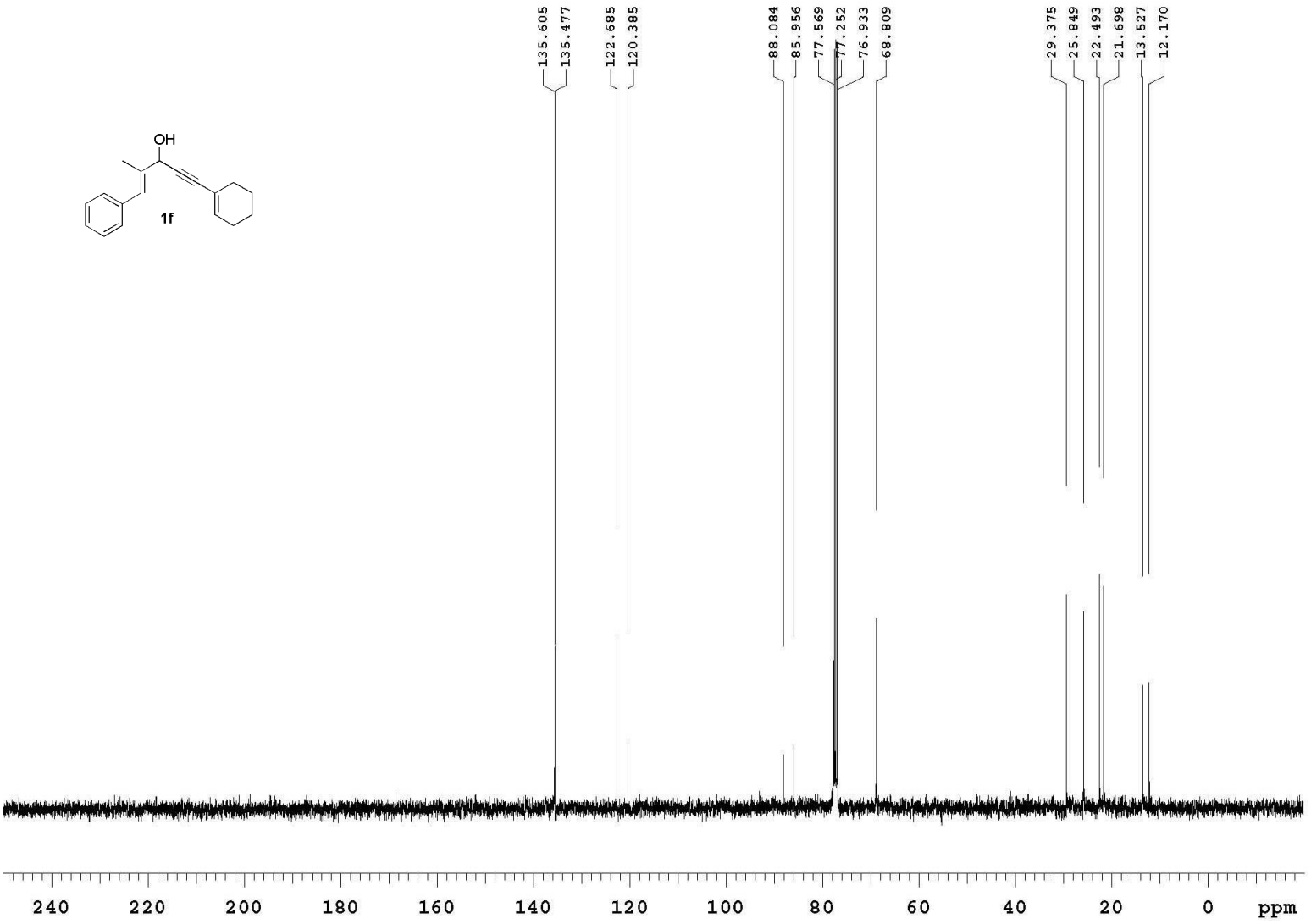
Yen-Ku, YKW-2-121-m4
400.393 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe

Pulse Sequence: s2pul



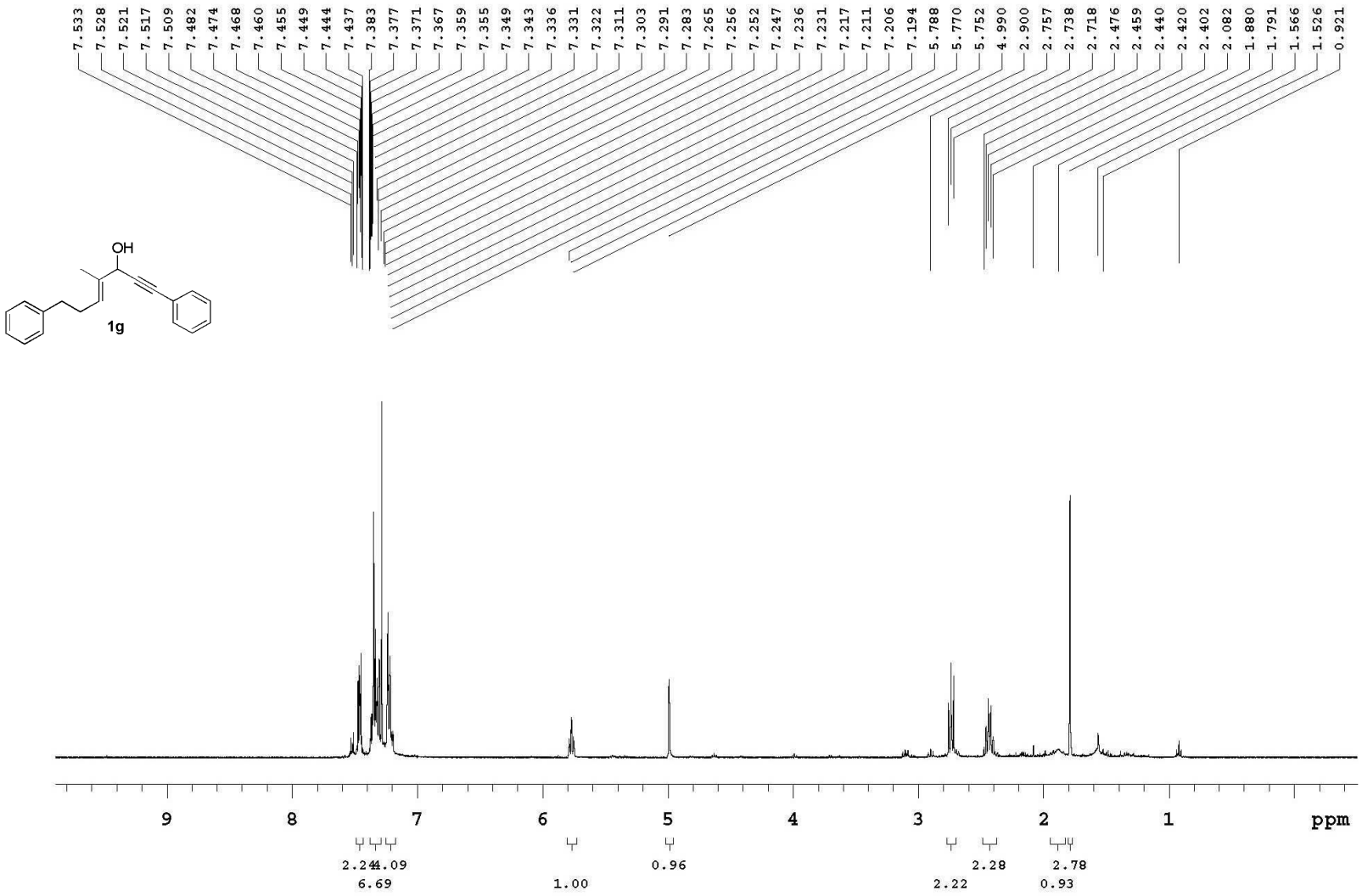


100.690 MHz C13[H1] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe
date: Aug 11 2009 sweep width: 27174Hz acq.time: 2.0s relax.time: 0.1s # scans: 1024 dig.res.: 0.2 Hz/pt hz/mm:113.2
spectrometer:d601 file:/mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/2009.08/2009.08.11.m4_YKW-2-121-m4_15:26_C13_1D.fid
Pulse Sequence: s2pul

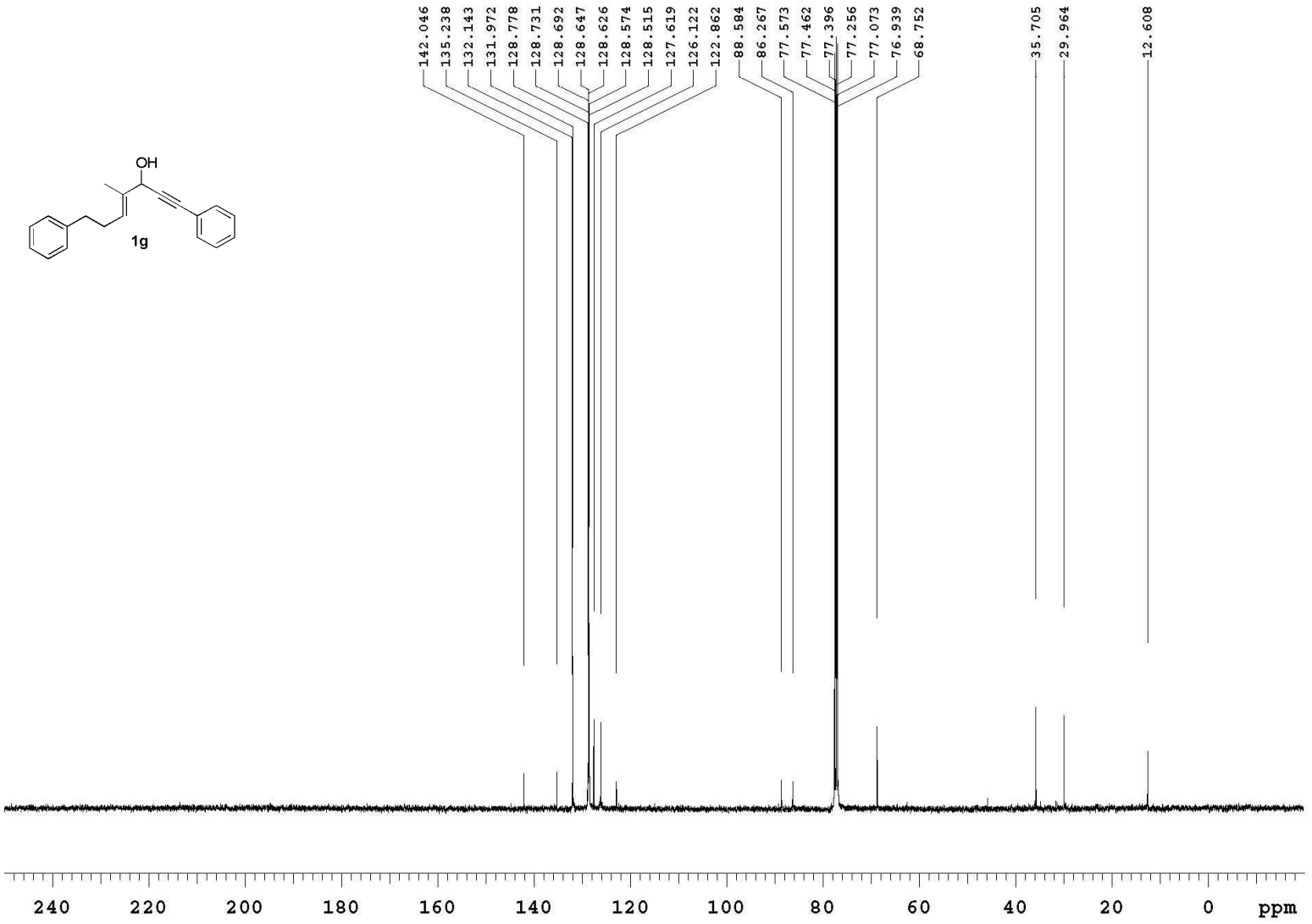


Yen-Ku, YKW-2-149-H1
400.393 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe

Pulse Sequence: s2pul

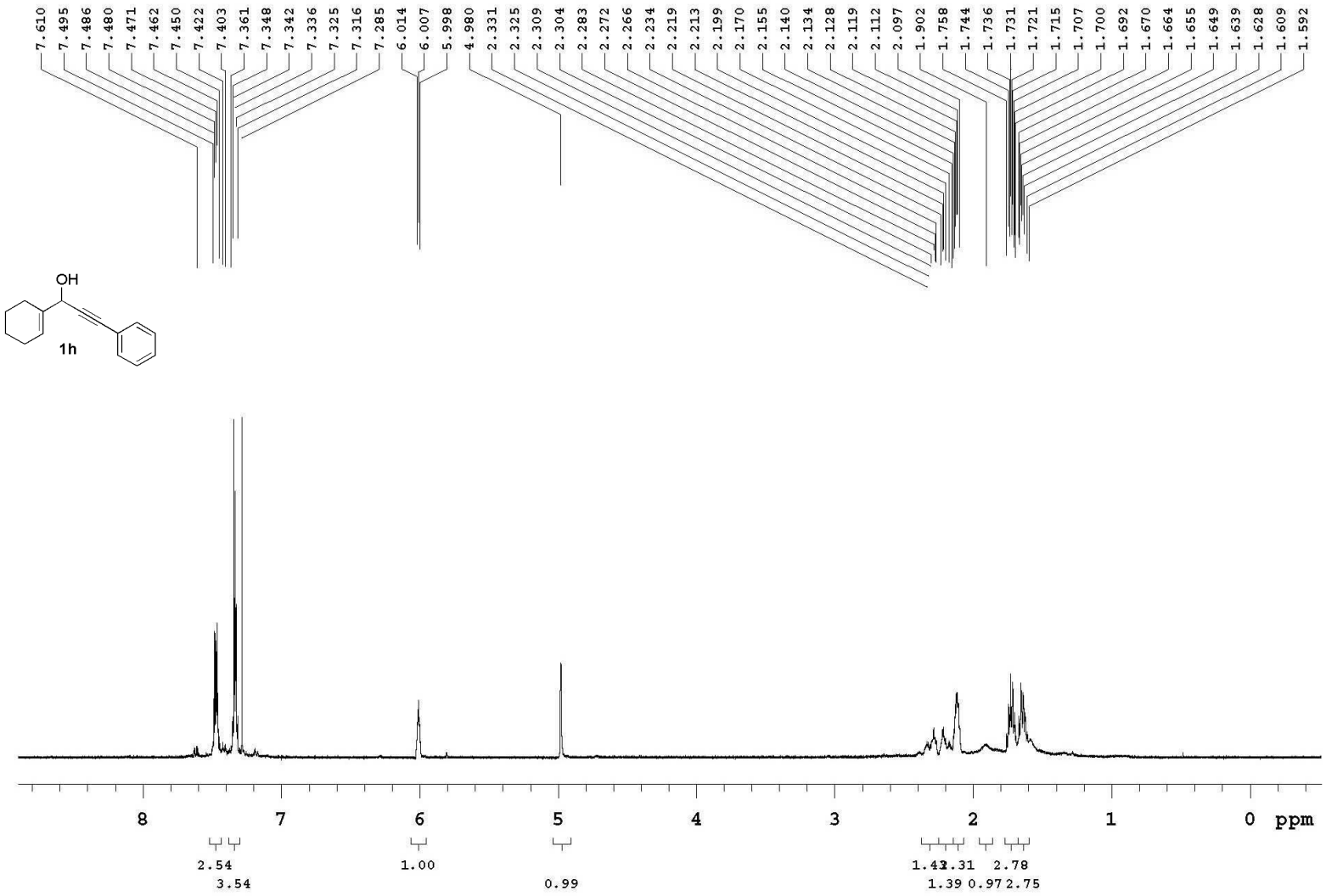


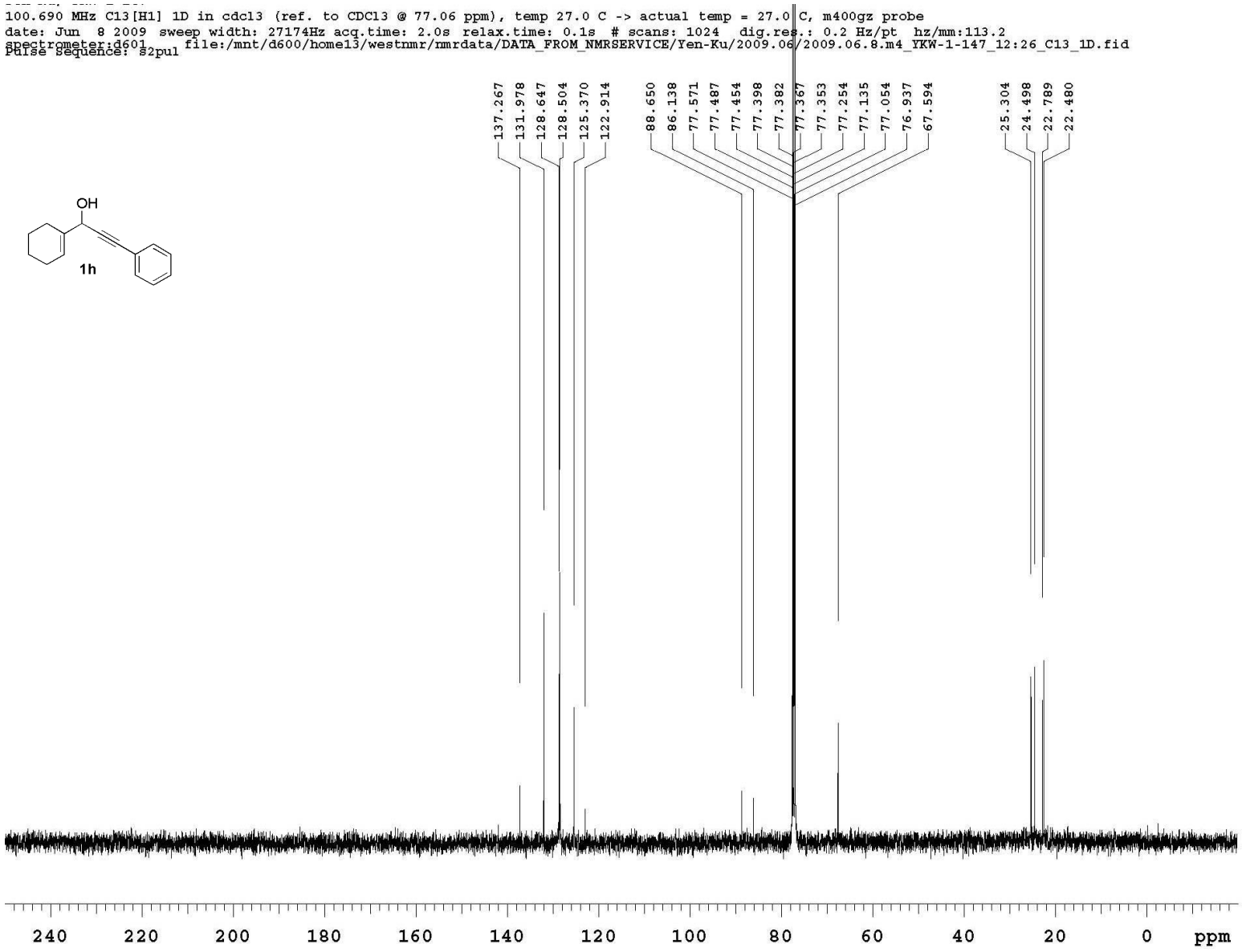
100.690 MHz C13[H1] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe
date: Aug 24 2009 sweep width: 27174Hz acq.time: 2.0s relax.time: 0.1s # scans: 6000 dig.res.: 0.2 Hz/pt hz/mm:113.2
spectrometer:d601 file:/mnt/d600/home13/westnmr/rmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/2009.08/2009.08.24.m4_YKW-2-149-CC_20:05_C13_1D.fid
Pulse sequence: s2pul



Yen-Ku, YKW-1-147
400.393 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe

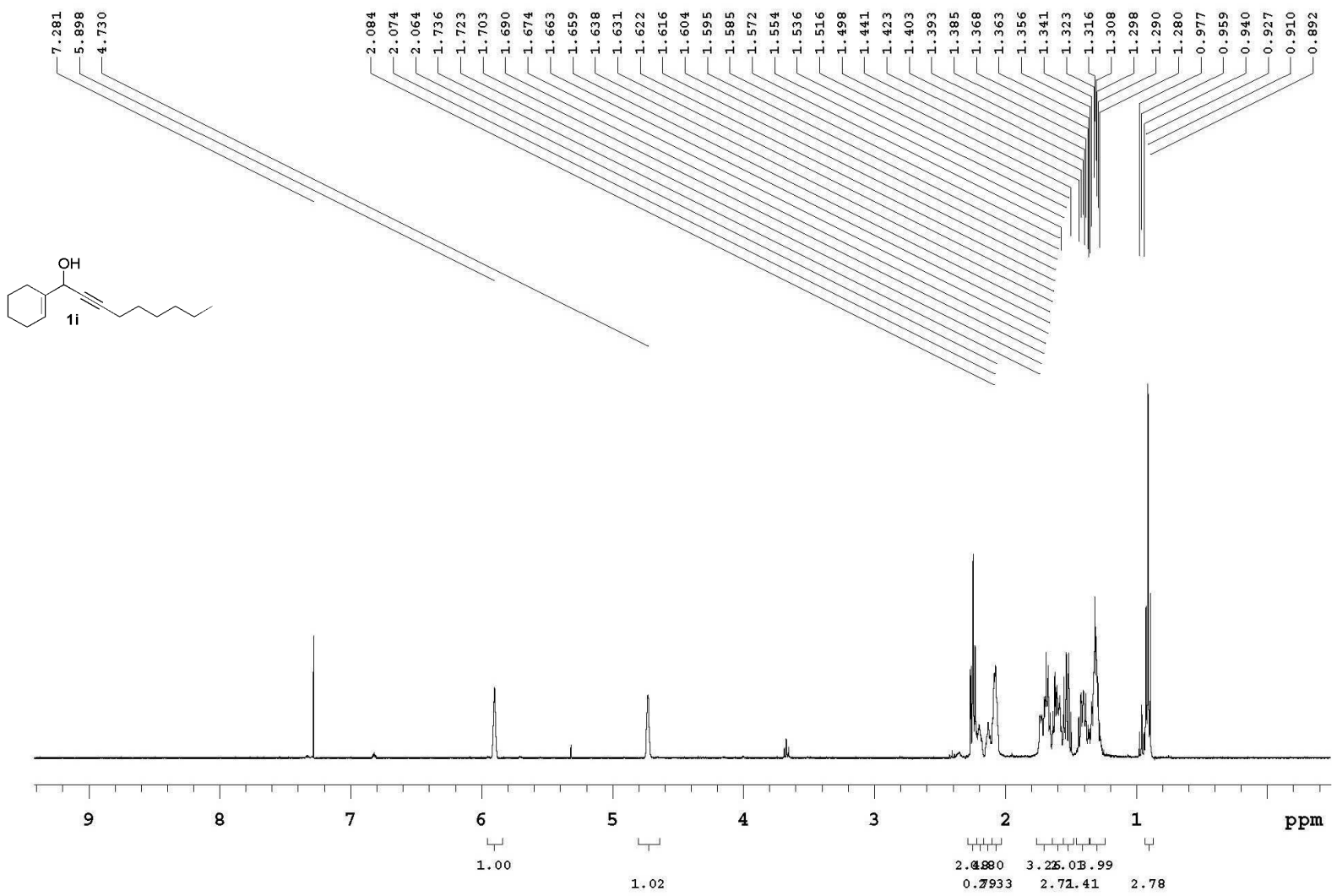
Pulse Sequence: s2pul



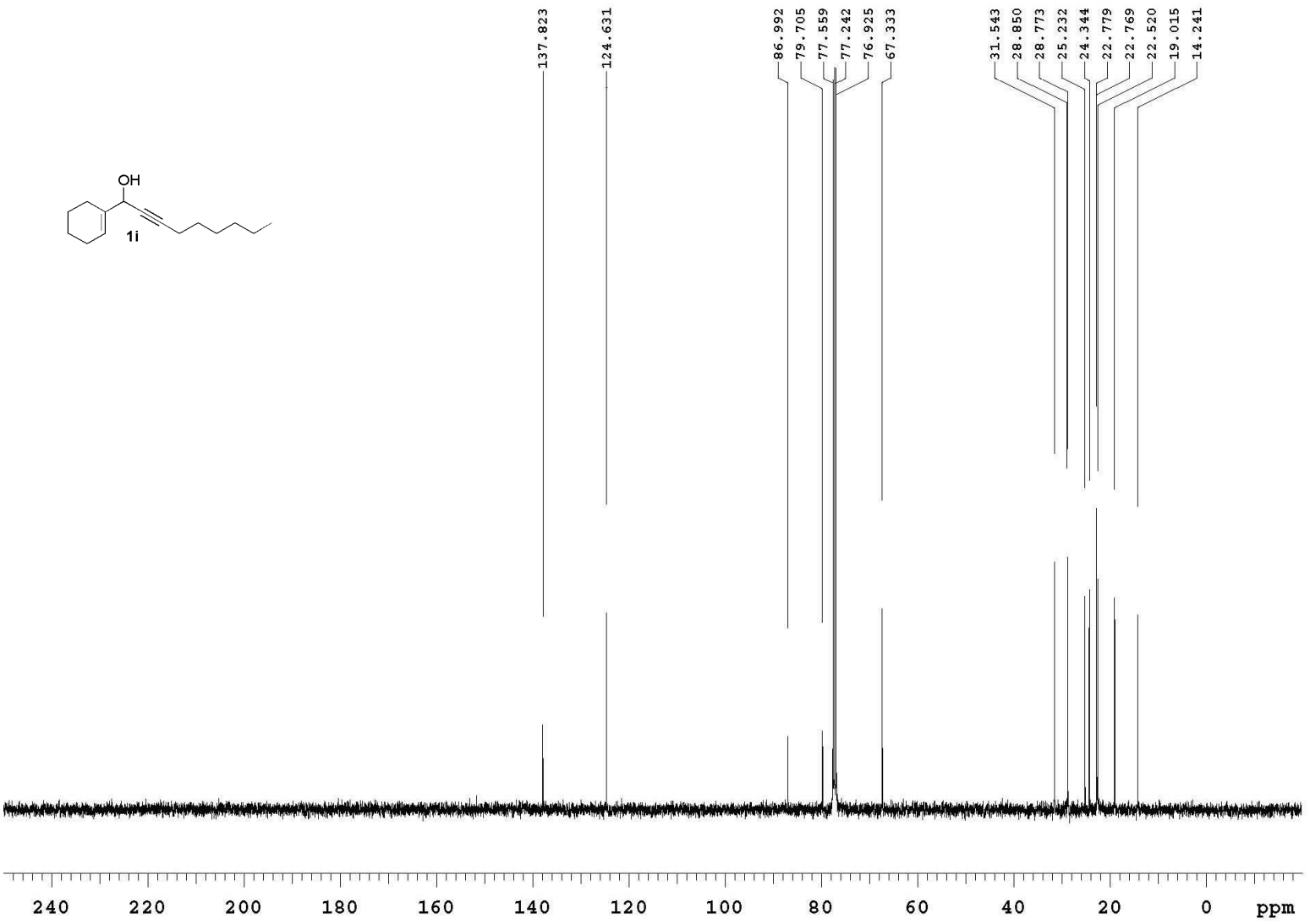

100.690 MHz C13[H1] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe
date: Jun 8 2009 sweep width: 27174Hz acq.time: 2.0s relax.time: 0.1s # scans: 1024 dig.res.: 0.2 Hz/pt hz/mm:113.2
spectrometer:d601 file:/mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/2009.06/2009.06.8.m4_YKW-1-147_12:26_C13_1D.fid
Pulse Sequence: s2pul
137.267
131.978
128.647
128.504
125.370
122.914
88.650
86.138
77.571
77.487
77.454
77.398
77.382
77.367
77.353
77.254
77.135
77.054
76.937
67.594
25.304
24.498
22.789
22.480
OH
1h
240
220
200
180
160
140
120
100
80
60
40
20
0
ppm

Yen-Ku, YKW-1-187-re
400.393 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe

Pulse Sequence: s2pul

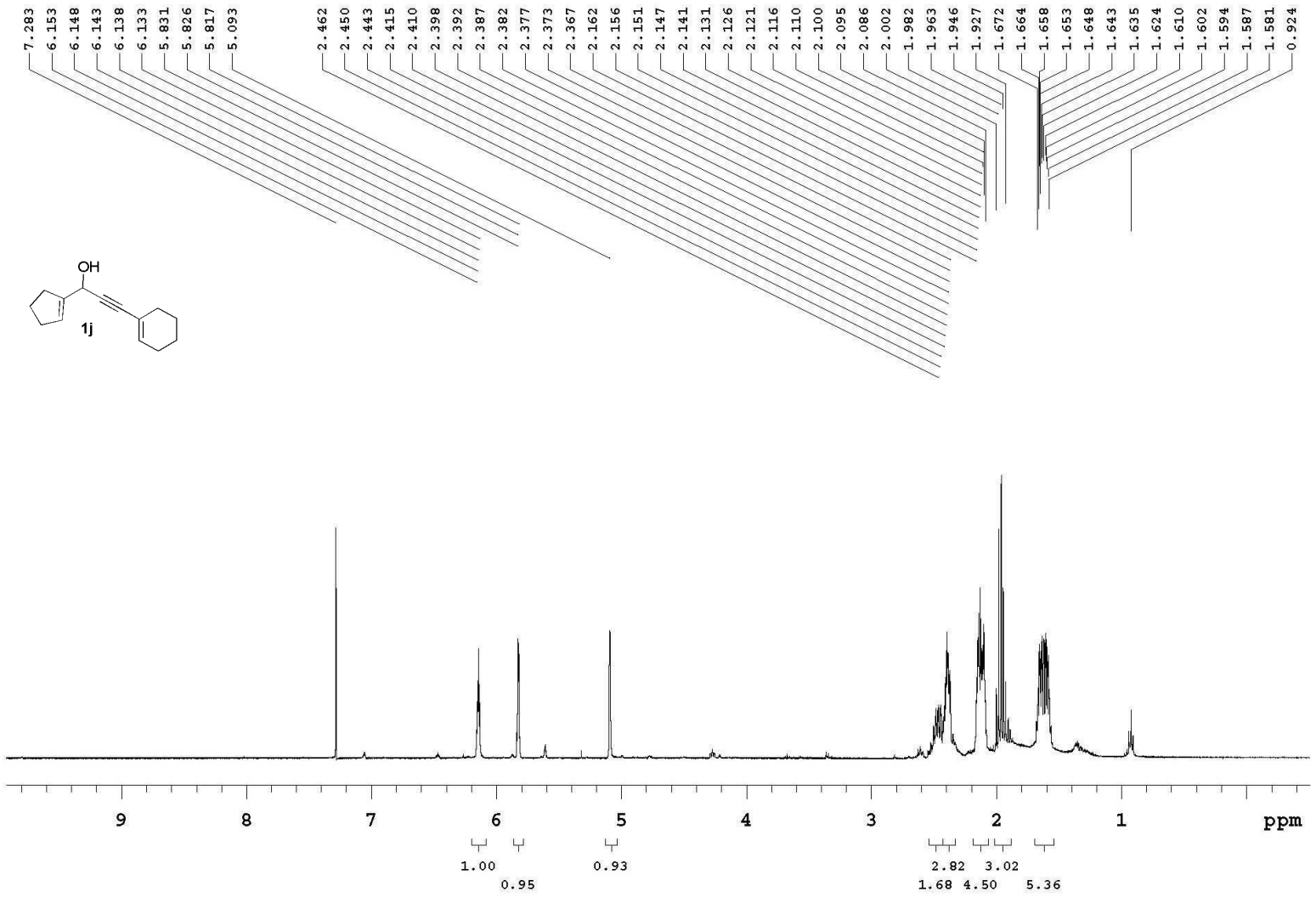


100.690 MHz C13[H1] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe
date: Jun 16 2009 sweep width: 27174Hz acq.time: 2.0s relax.time: 0.1s # scans: 1024 dig.res.: 0.2 Hz/pt hz/mm:113.2
spectrometer:d601 file:/mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/2009.06/2009.06.16.m4_YKW-1-187-re_17:42_C13_1D.fid
Pulse Sequence: s2pul

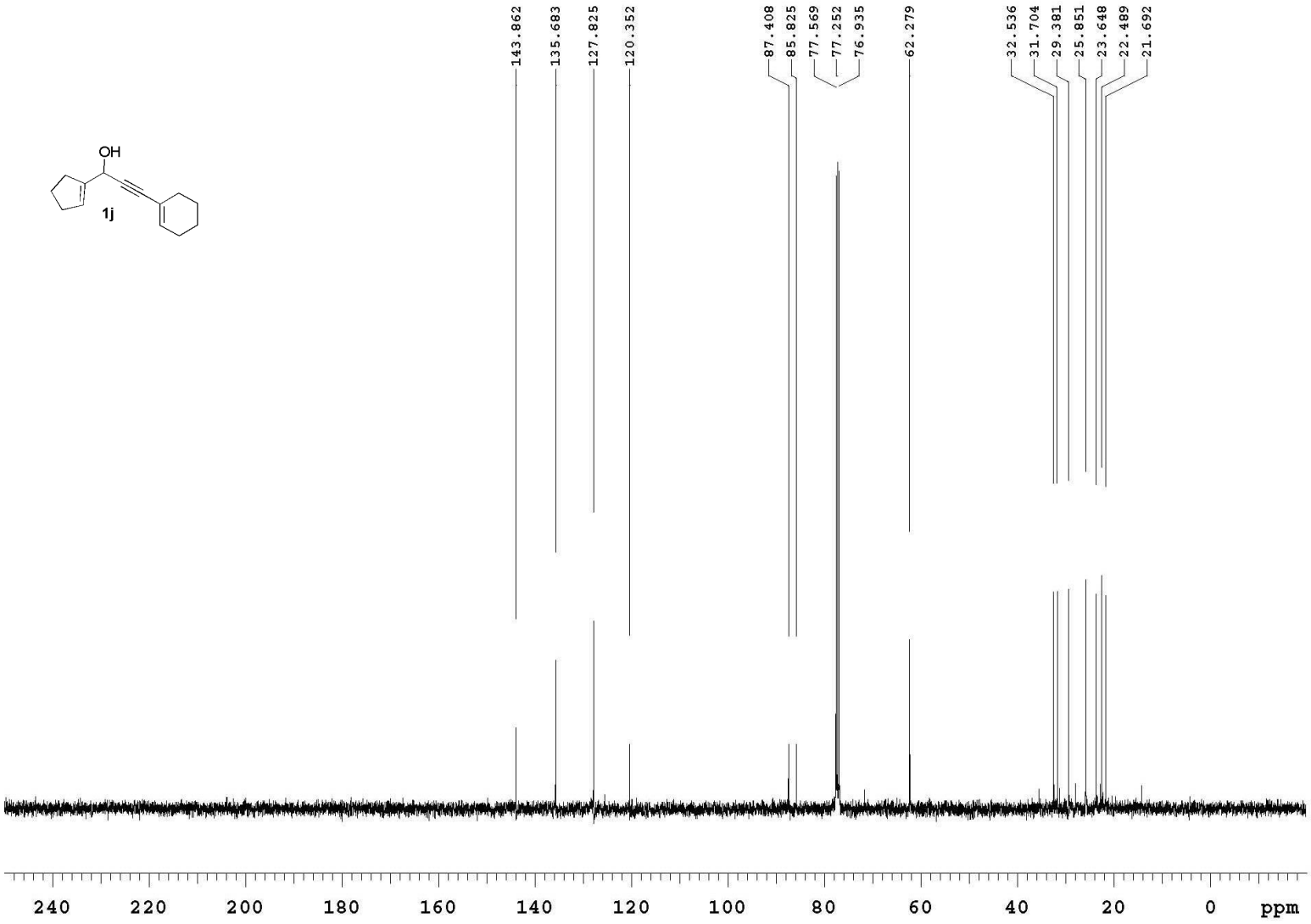


Yen-Ku, YKW-2-181-Re
400.393 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe

Pulse Sequence: s2pul

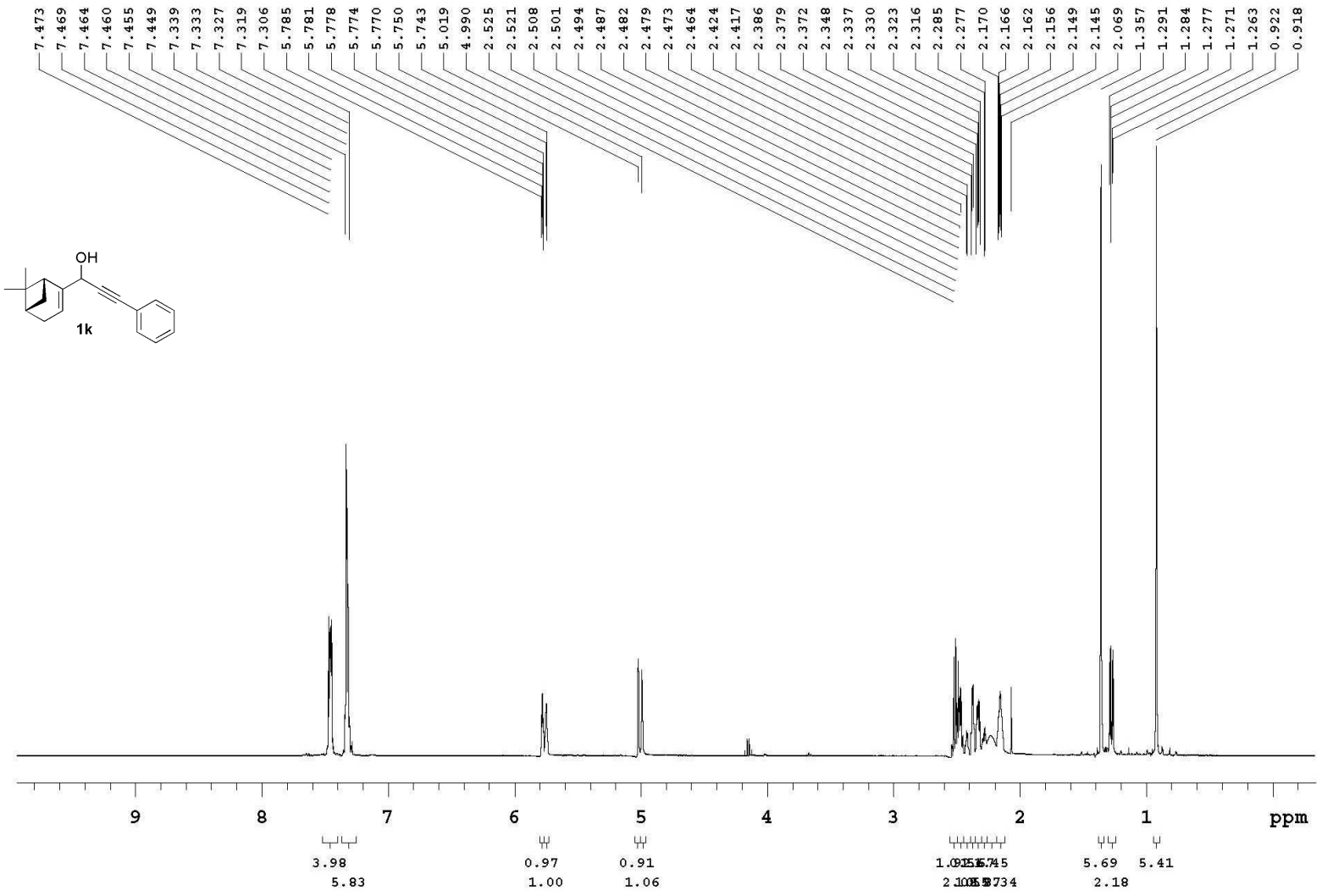


100.690 MHz C13[H1] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe
date: Sep 13 2009 sweep width: 27174Hz acq.time: 2.0s relax.time: 0.1s # scans: 1024 dig.res.: 0.2 Hz/pt hz/mm:113.2
spectrometer:d601 file:/mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/2009.09/2009.09.13.m4_YKW-2-181-Re_02:53_C13_1D.fid
Pulse Sequence: s2pul



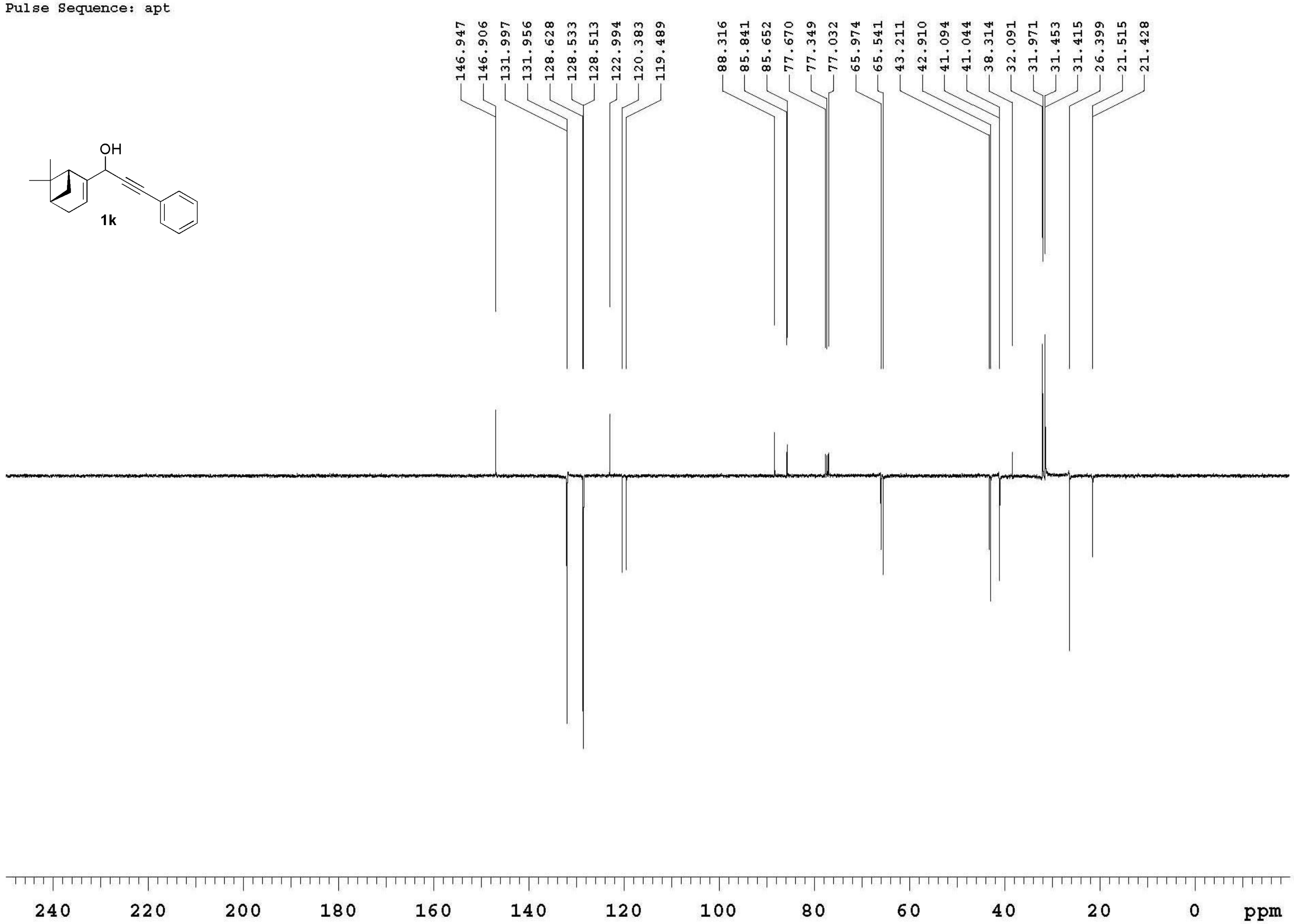
Yen-Ku, YKW-2-3-m4
400.393 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe

Pulse Sequence: s2pul



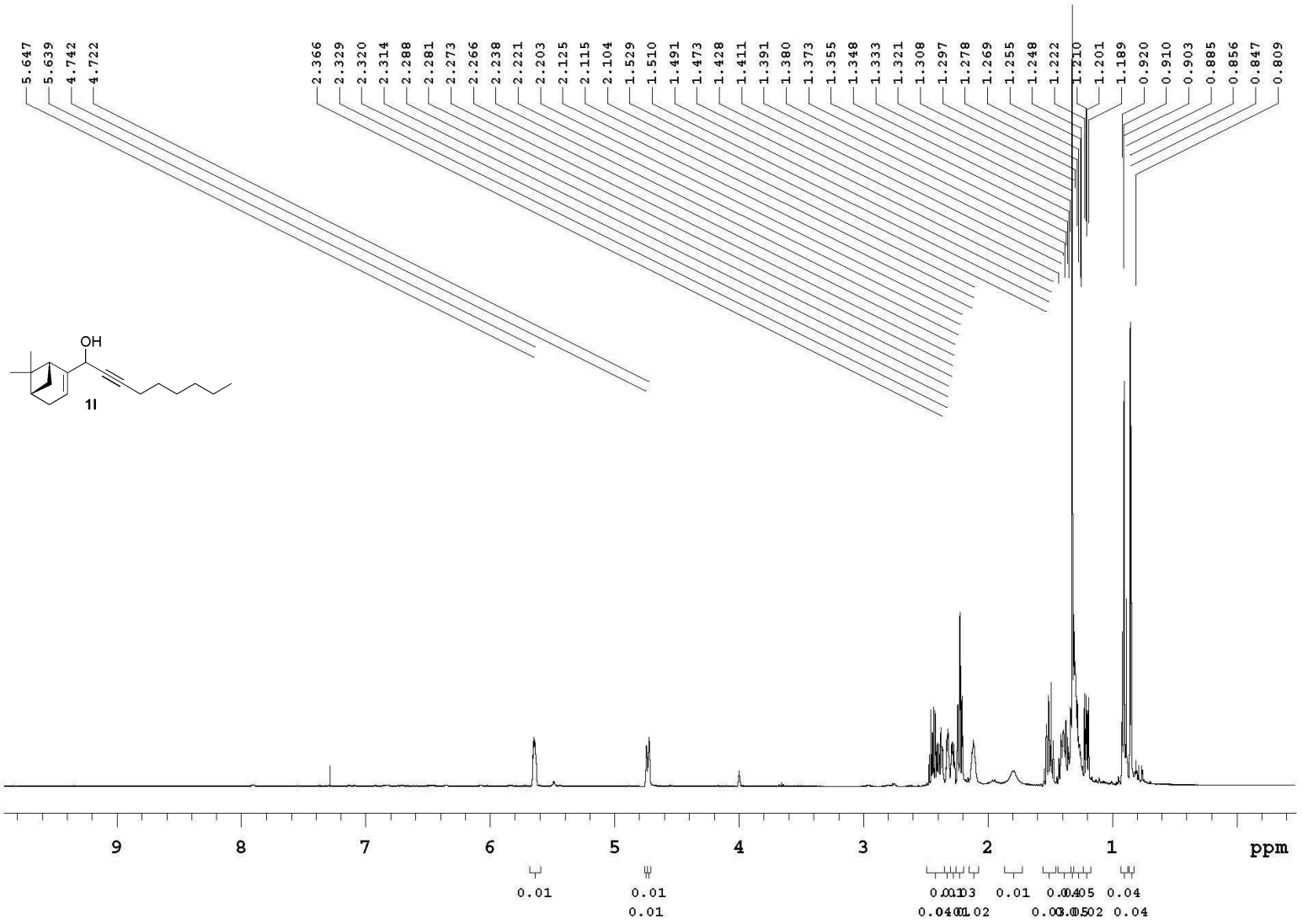
Yen-Ku, YKW-2-3-m4
100.690 MHz C13[H1] apt in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe
date: Jun 23 2009 sweep width: 27174Hz acq.time: 2.0s relax.time: 0.1s # scans: 512 dig.res.: 0.4 Hz/pt hz/mm:113.2
C & CH2 same, CH & CH3 opposite side of solvent signal
spectrometer:d601 file:/mnt/d600/home13/westmmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/2009.06/2009.06.23.m4_YKW-2-3-m4_12:26_C13_apt.fid

Pulse Sequence: apt

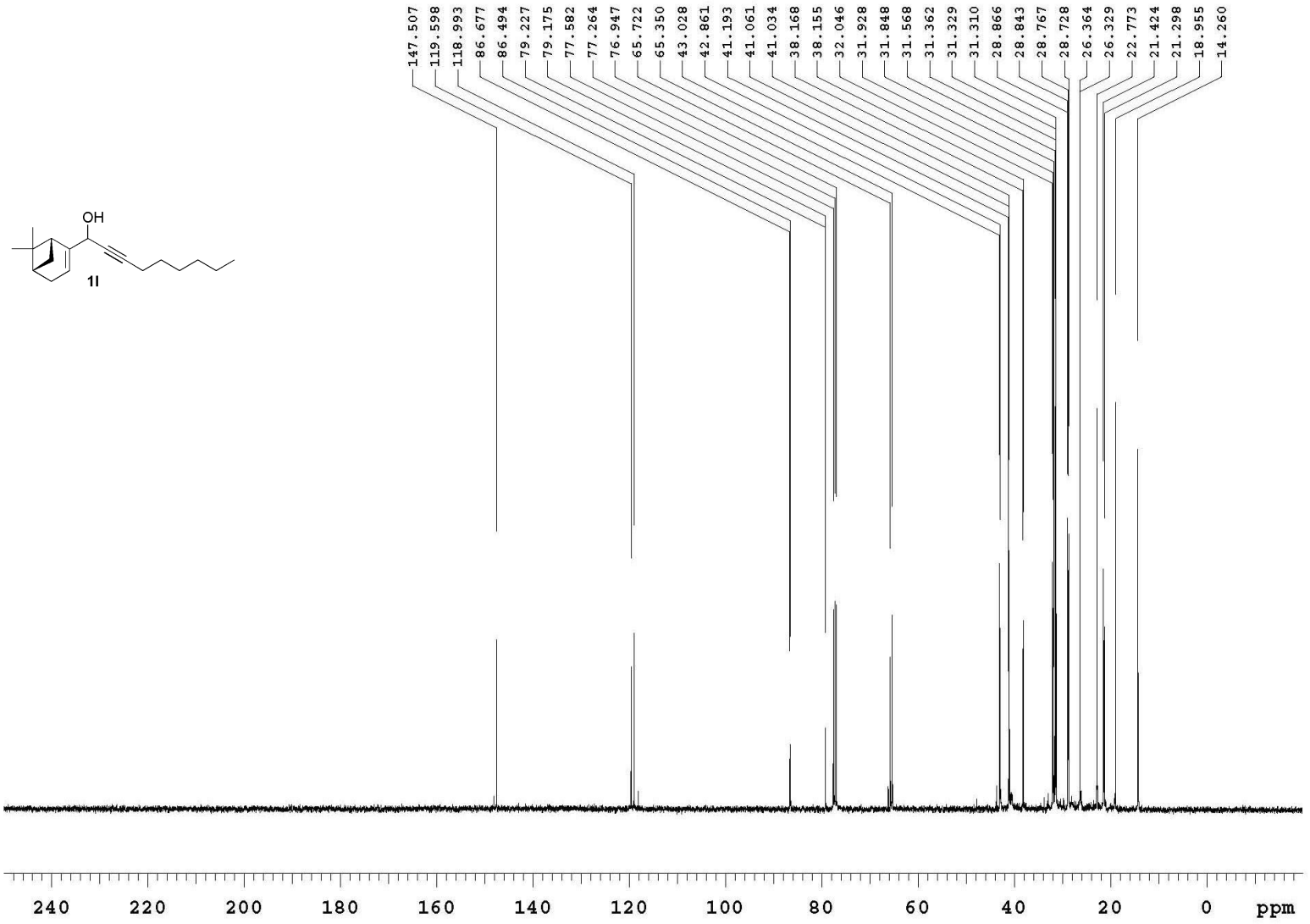


Yen-Ku, YKW-2-11
400.393 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe

Pulse Sequence: s2pul

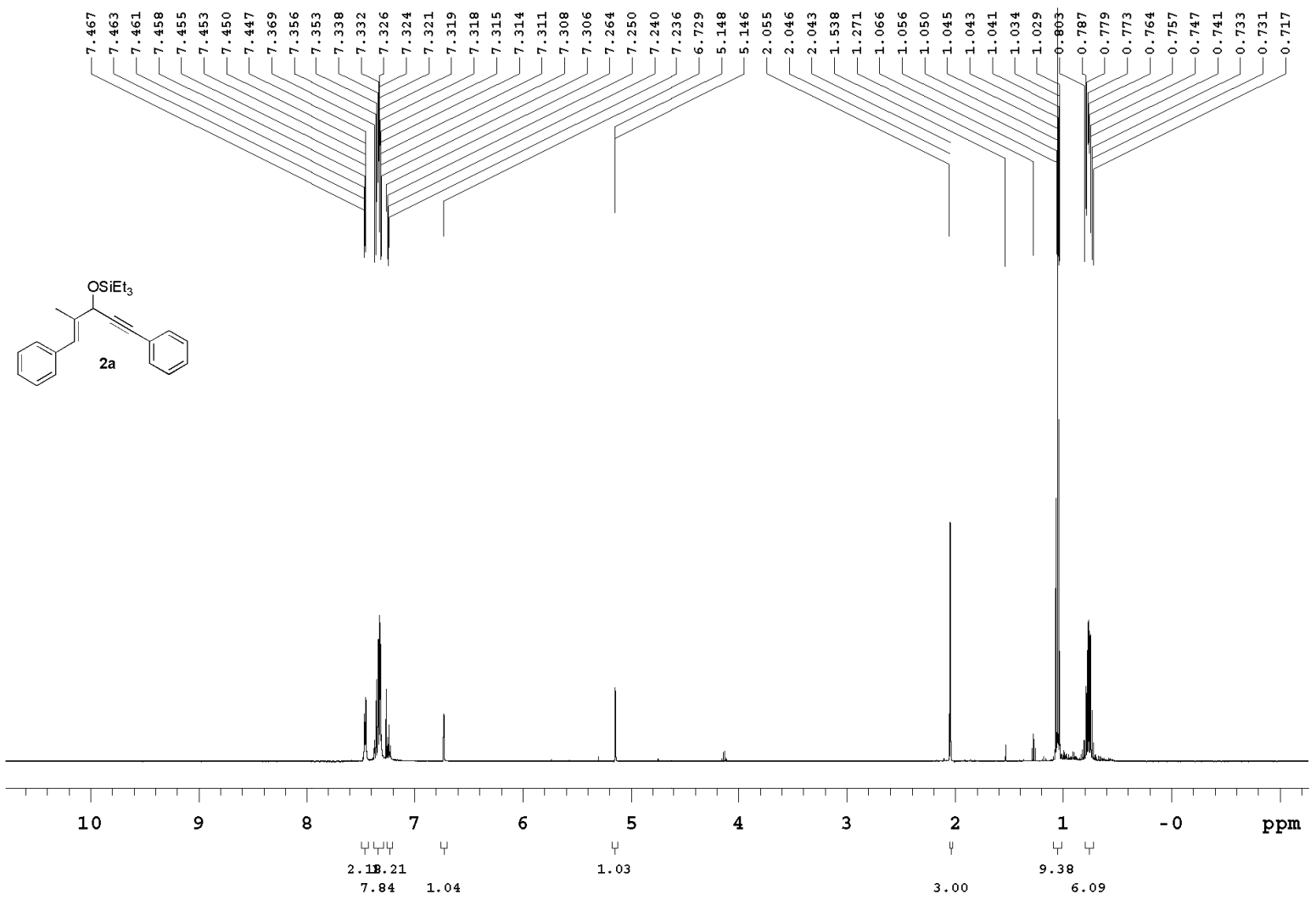


Yen-Ku, YKW-2-11-C13
100.690 MHz C13[H1] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe
date: Jun 25 2009 sweep width: 27174Hz acq.time: 2.0s relax.time: 0.1s # scans: 1024 dig.res.: 0.2 Hz/pt hz/mm:113.2
spectrometer:d601 file:/mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/2009.06/2009.06.25.m4_YKW-2-11-C13_18:35_C13_1D.fid
Pulse sequence: s2pul

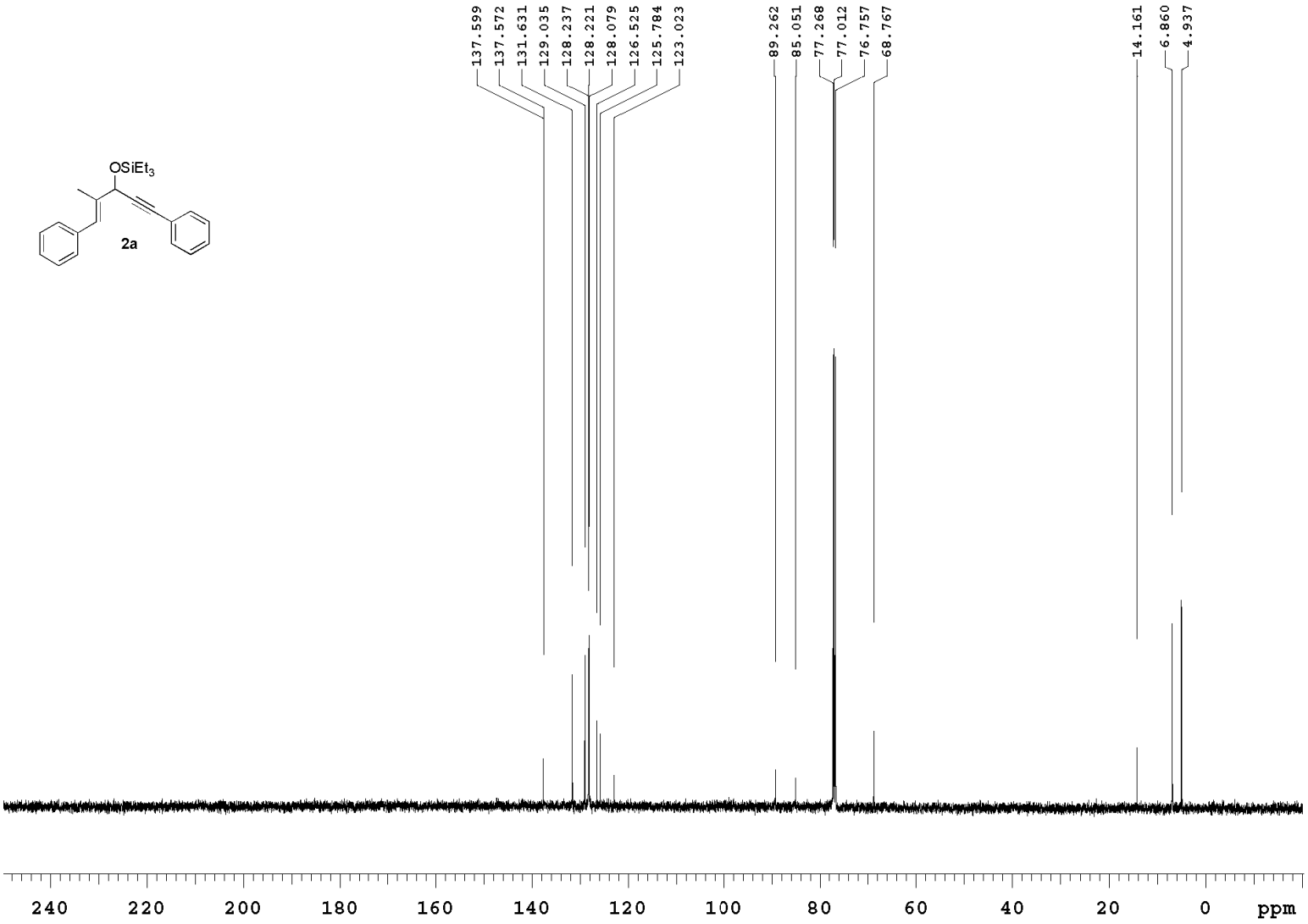


YKW-4-201
498.122 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.2 C -> actual temp = 27.0 C, autoxdb probe

Pulse Sequence: s2pul

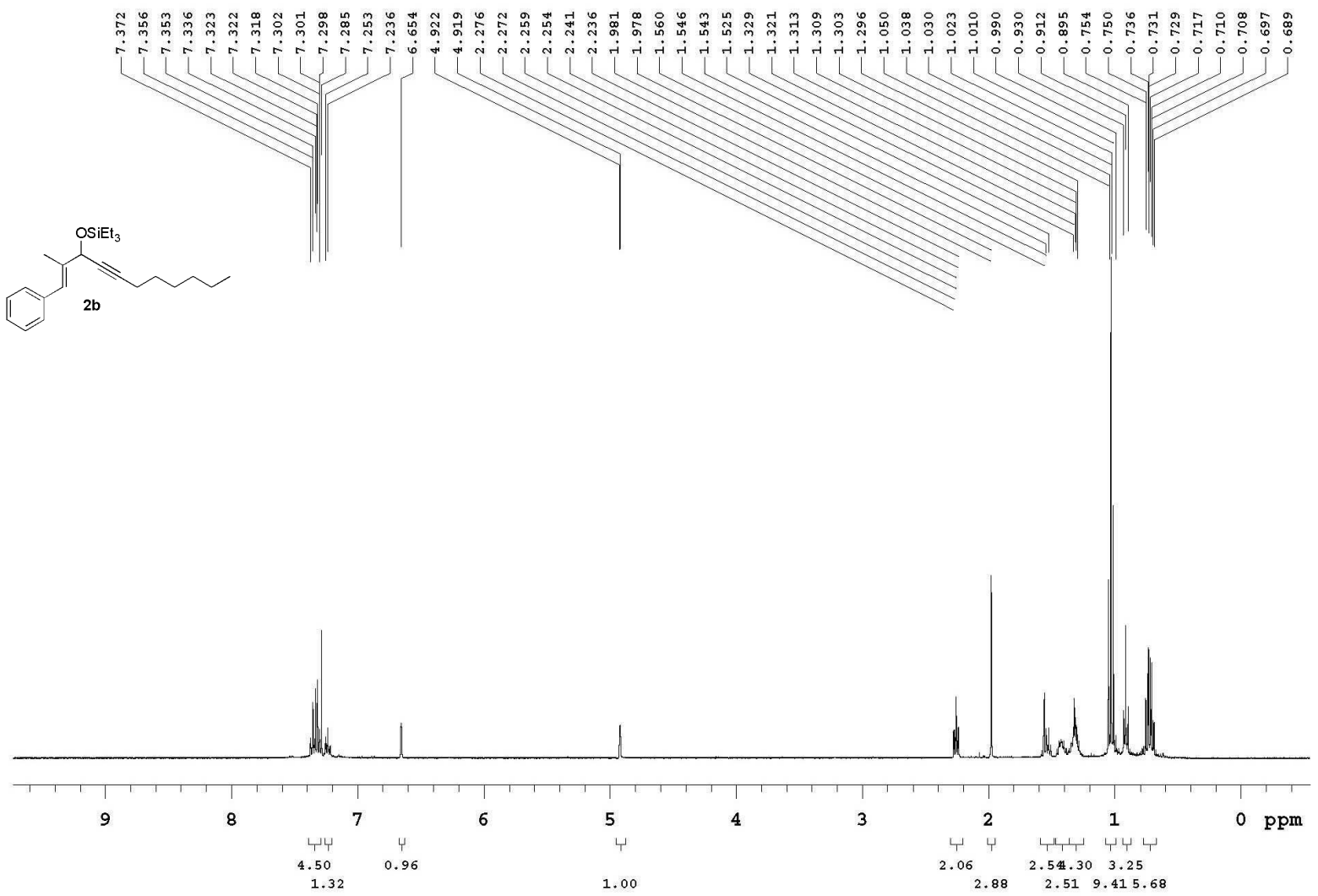


125.264 MHz C13[H1] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.2 C -> actual temp = 27.0 C, autoxdb probe
date: Apr 14 2010 sweep width: 33827Hz acq.time: 2.5s relax.time: 0.1s # scans: 112 dig.res.: 0.3 Hz/pt hz/mm:140.9
spectrometer:ibdw file:/mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/2010.04/2010.04.14.15_YKW-4-201_C13_1D.fid
Pulse sequence: s2pul

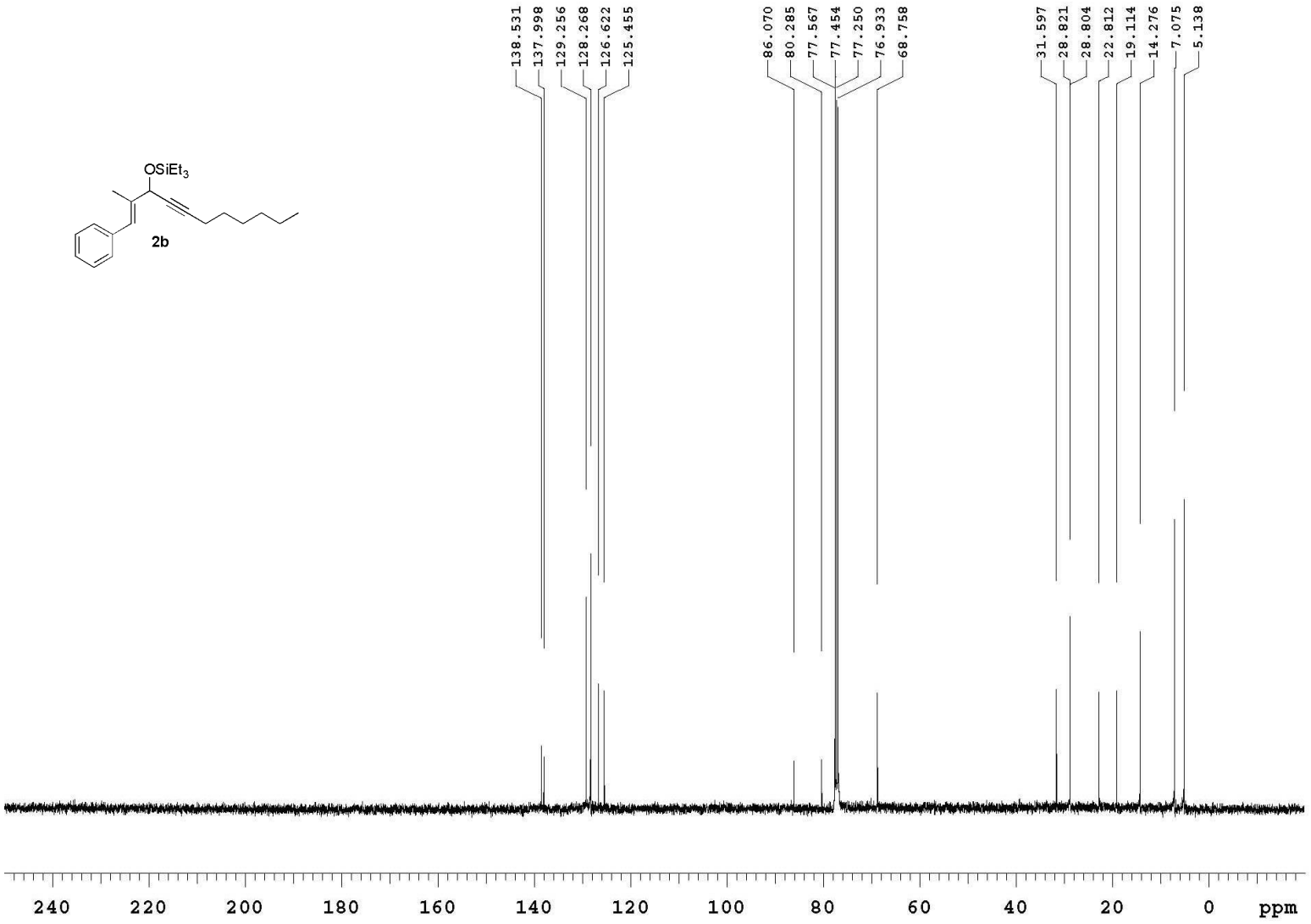


Yen-Ku, YKW-1-129-H
400.393 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe

Pulse Sequence: s2pul

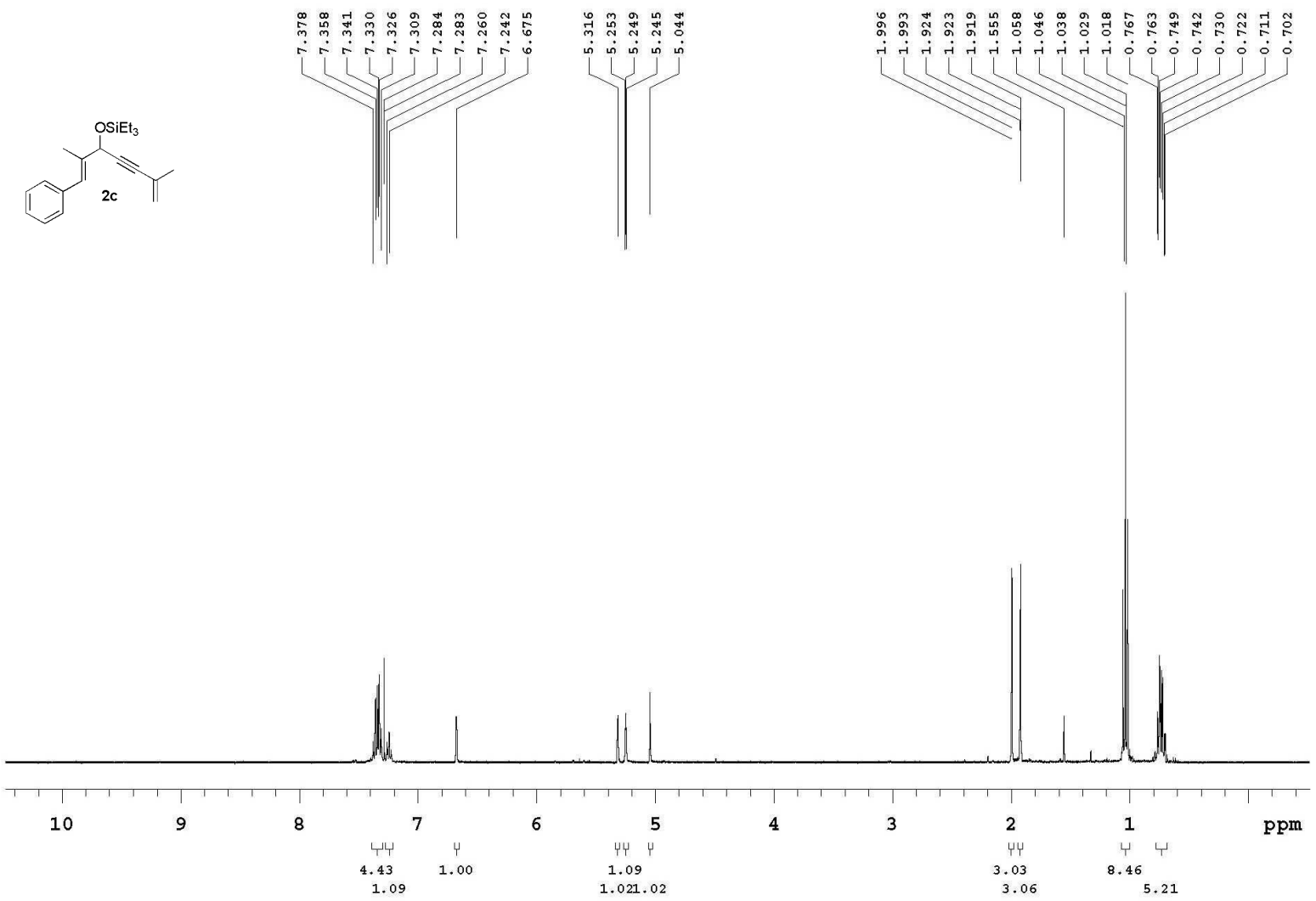


100.690 MHz C13[H1] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe
date: May 30 2009 sweep width: 27174Hz acq.time: 2.0s relax.time: 0.1s # scans: 2048 dig.res.: 0.2 Hz/pt hz/mm:113.2
spectrometer:d601 file:/mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/2009.05/2009.05.30.m4_YKW-1-129-13C_01:48_C13_1D.fid
Pulse sequence: s2pul

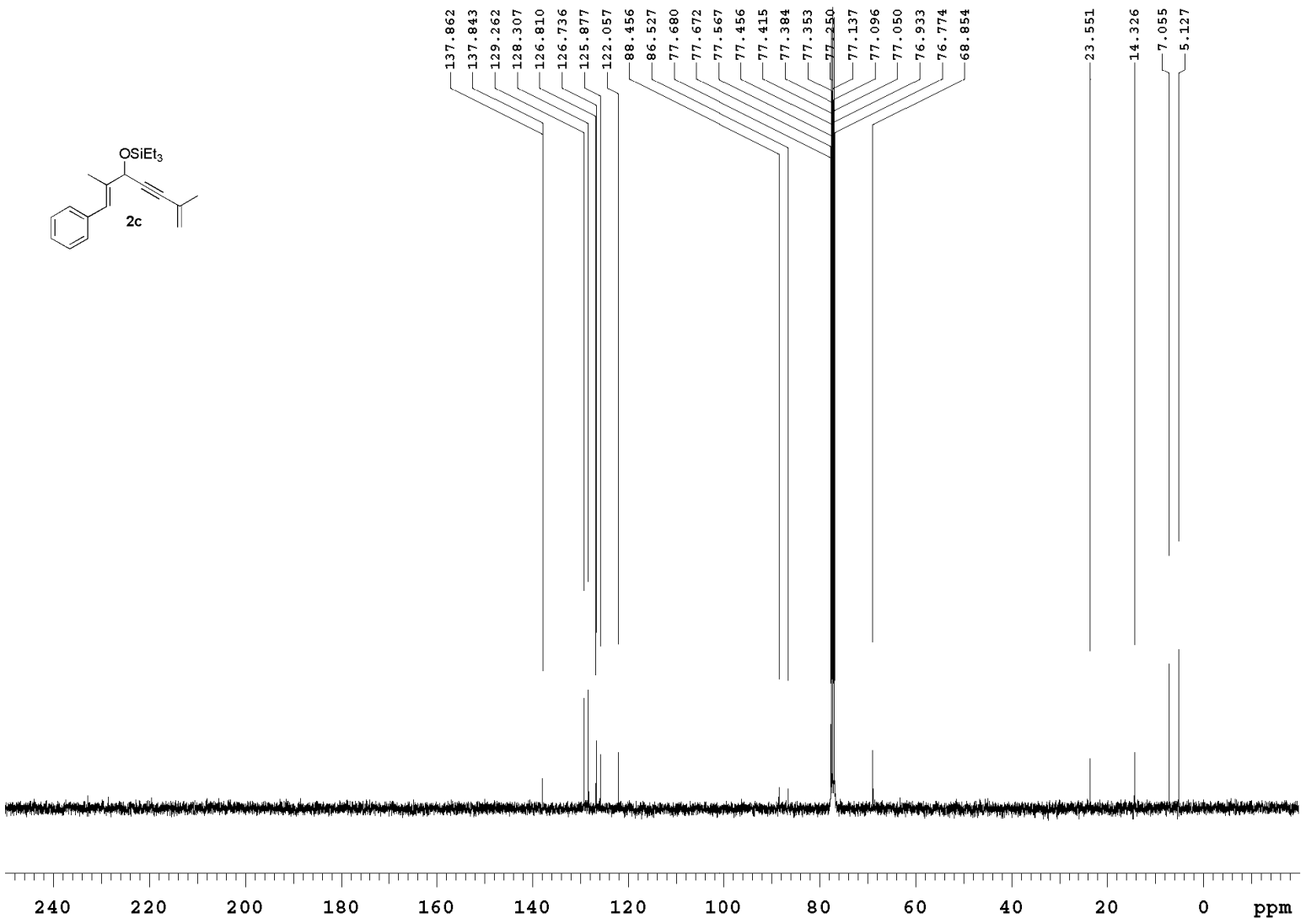


Yen-Ku, YKW-3-115m4
400.393 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe

Pulse Sequence: s2pul

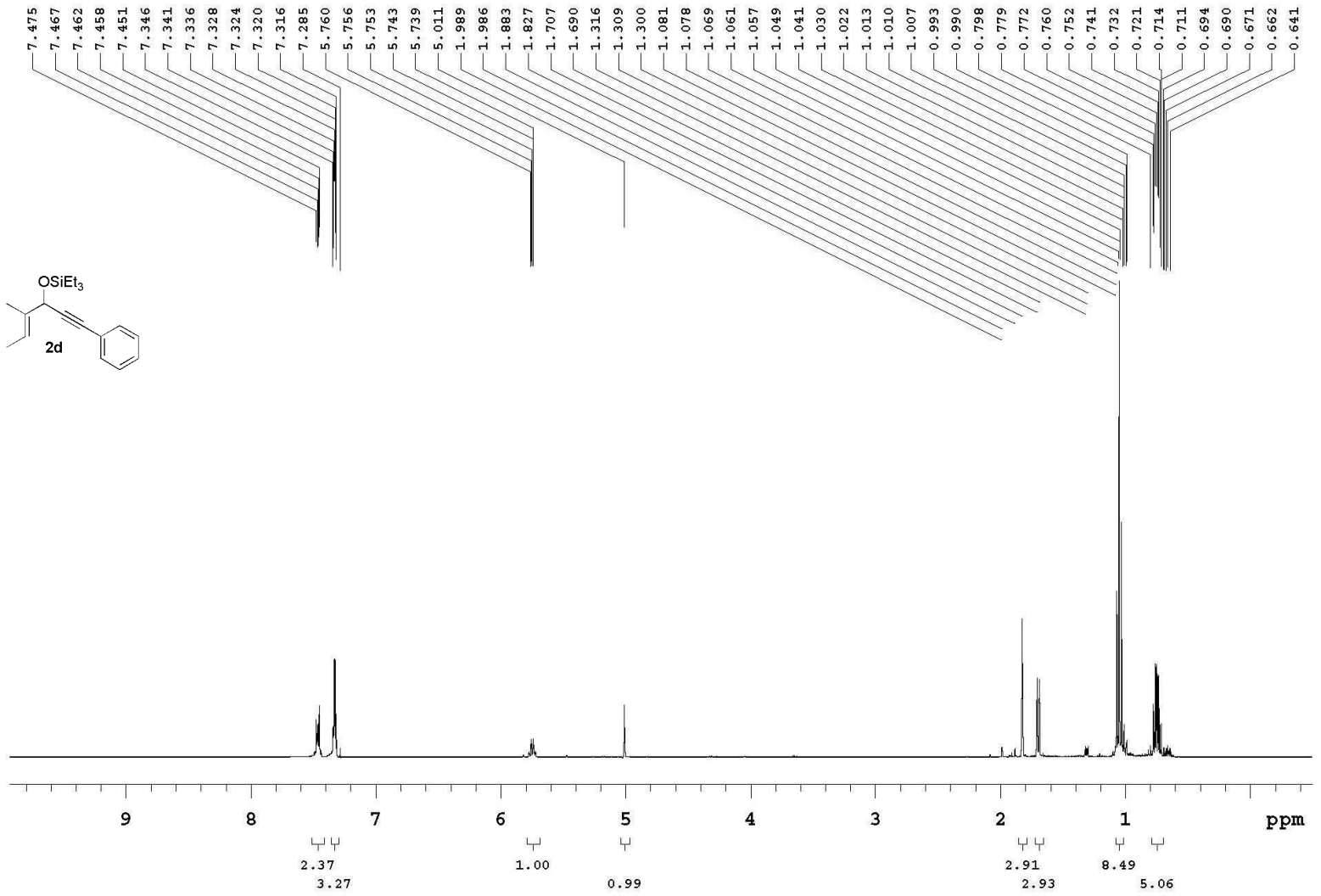


100.690 MHz C13[H1] 1D in cdcl3 (ref. to CDC13 @ 77.06 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe
date: Nov 13 2009 sweep width: 27174Hz acq.time: 2.0s relax.time: 0.1s # scans: 1024 dig.res.: 0.2 Hz/pt hz/mm:113.2
spectrometer:ibdw file:/mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/2009.11/2009.11.13.m4_YKW-3-115m4_11.32_C13_1D.fid
Pulse Sequence: s2pul



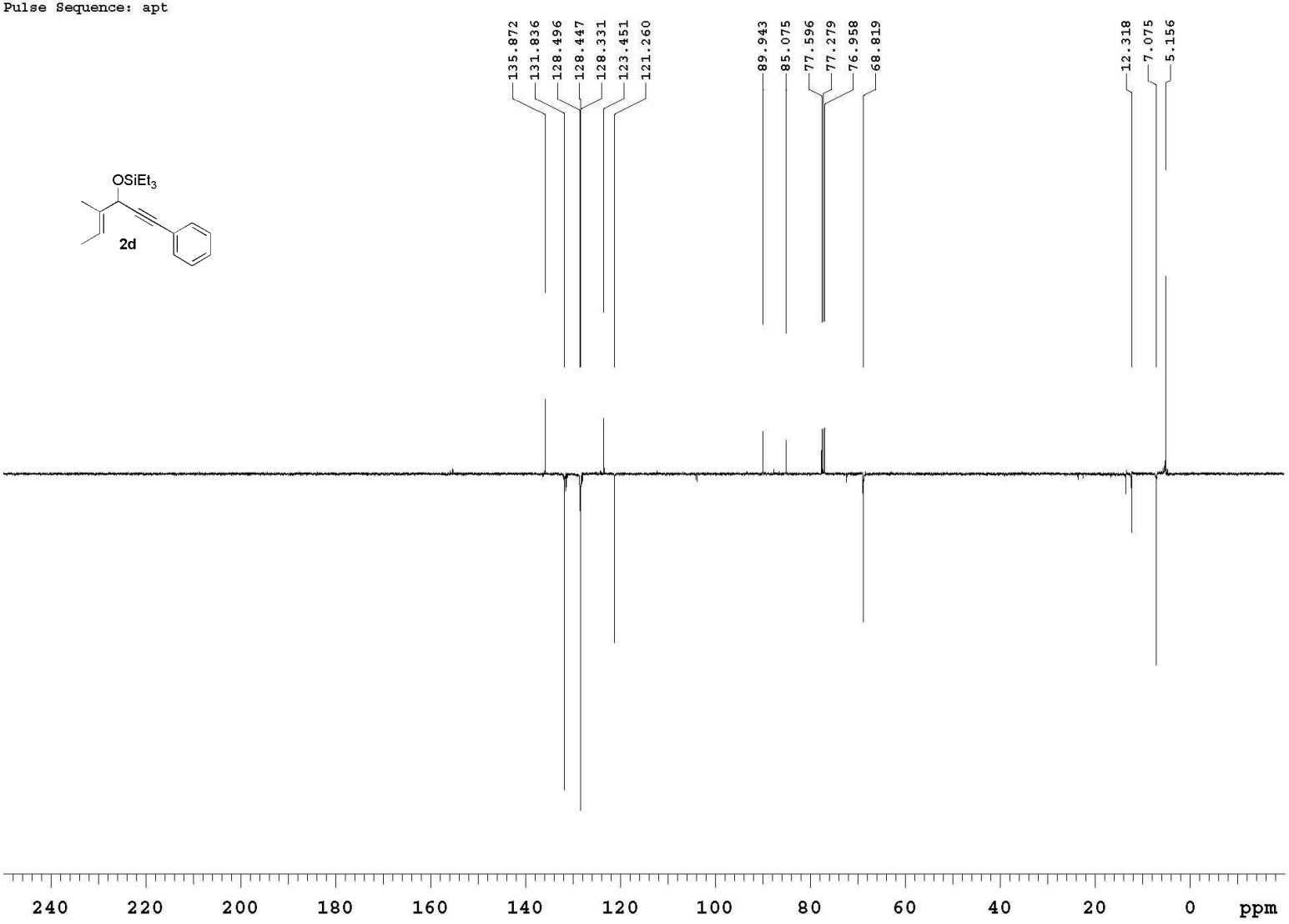
Yen-Ku, YKW-1-69_PP_
400.393 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe

Pulse Sequence: s2pul



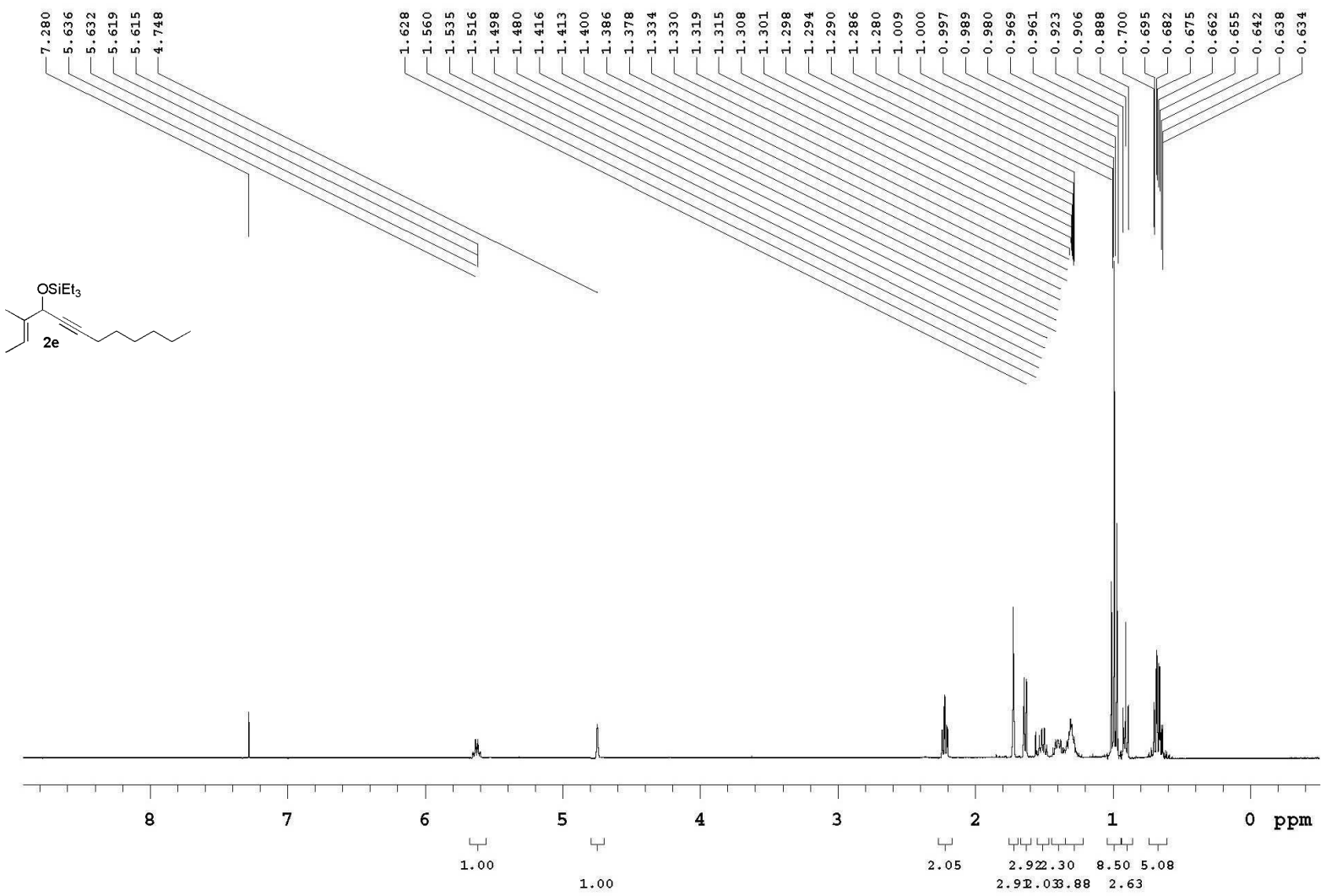
100.690 MHz C13[H1] apt in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe
date:May 14 2019 sw=[illegible] relax.time: 0.1s # scans: 2048 dig.res.: 0.4 Hz/pt hz/mm:113.2
spectrometer:d601 file:/mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/2009.05/2009.05.14.m4_YKW-1-69_PP__20:04_C13_apt.fid

Pulse Sequence: apt

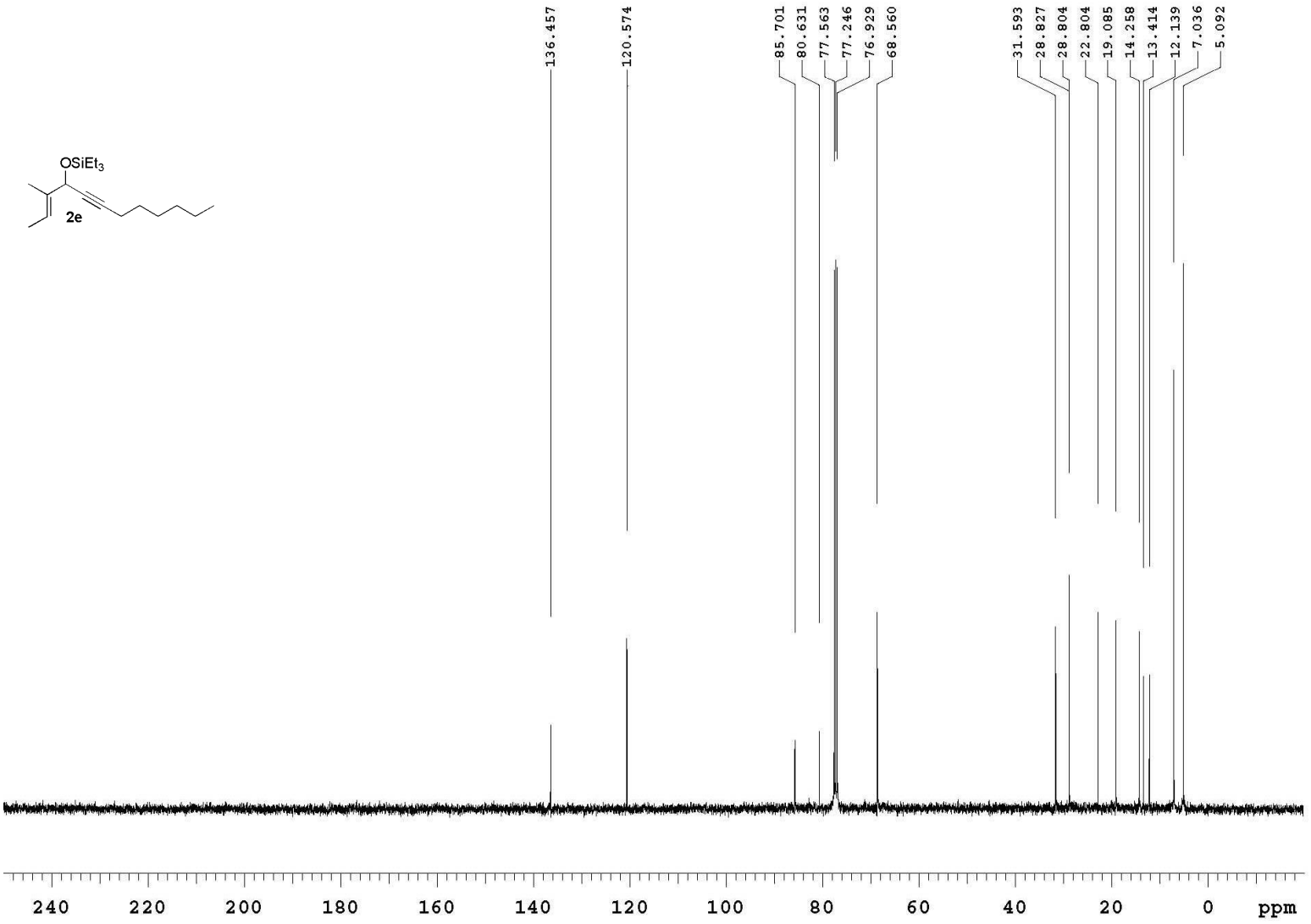


Yen-Ku, YKW-2-123
400.393 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe

Pulse Sequence: s2pul

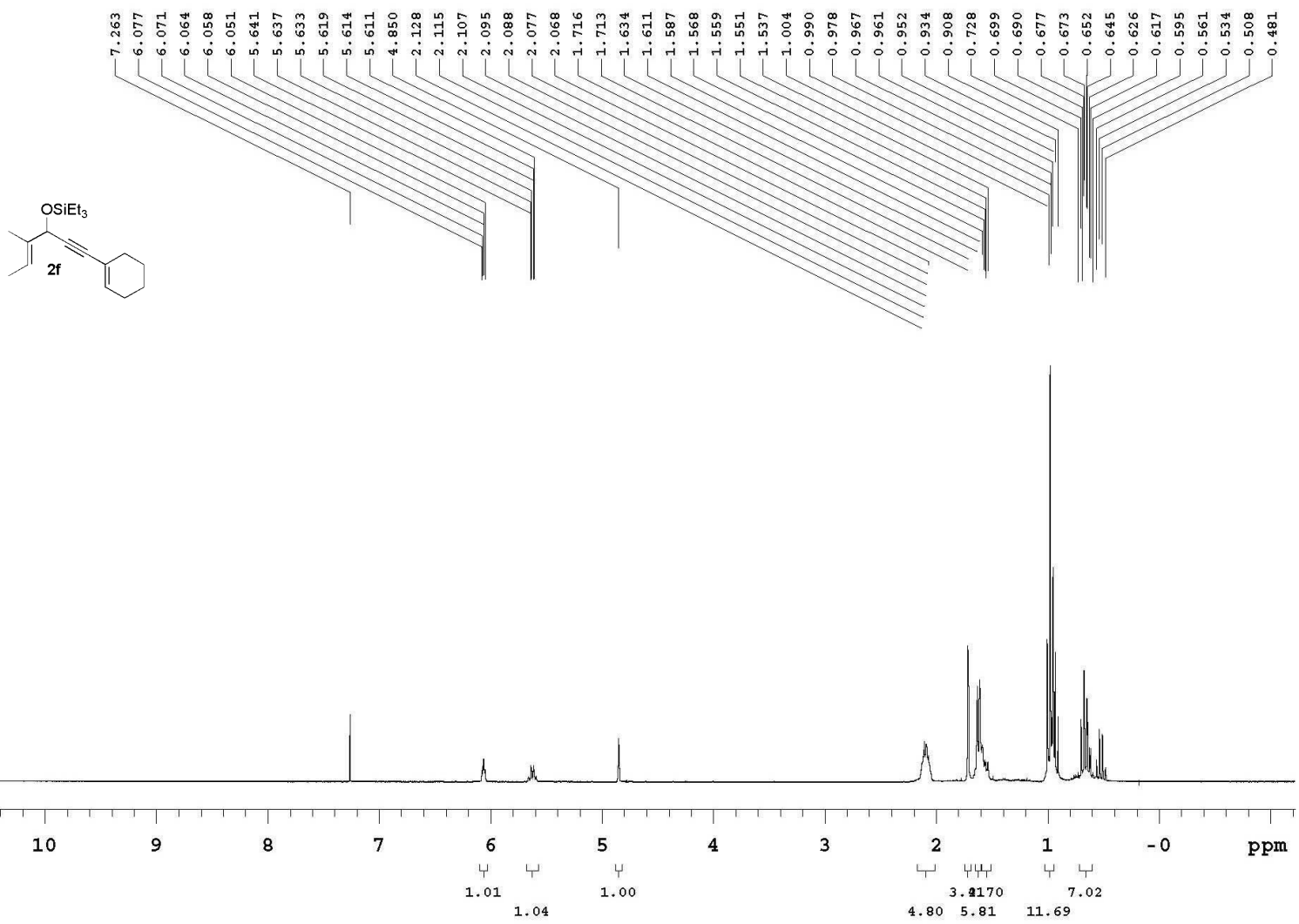


100.690 MHz C13[H1] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe
date: Aug 12 2009 sweep width: 27174Hz acq.time: 2.0s relax.time: 0.1s # scans: 1024 dig.res.: 0.2 Hz/pt hz/mm:113.2
spectrometer:d601 file:/mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/2009.08/2009.08.12.m4_YKW-2-123_13:48_C13_1D.fid
Pulse sequence: s2pul

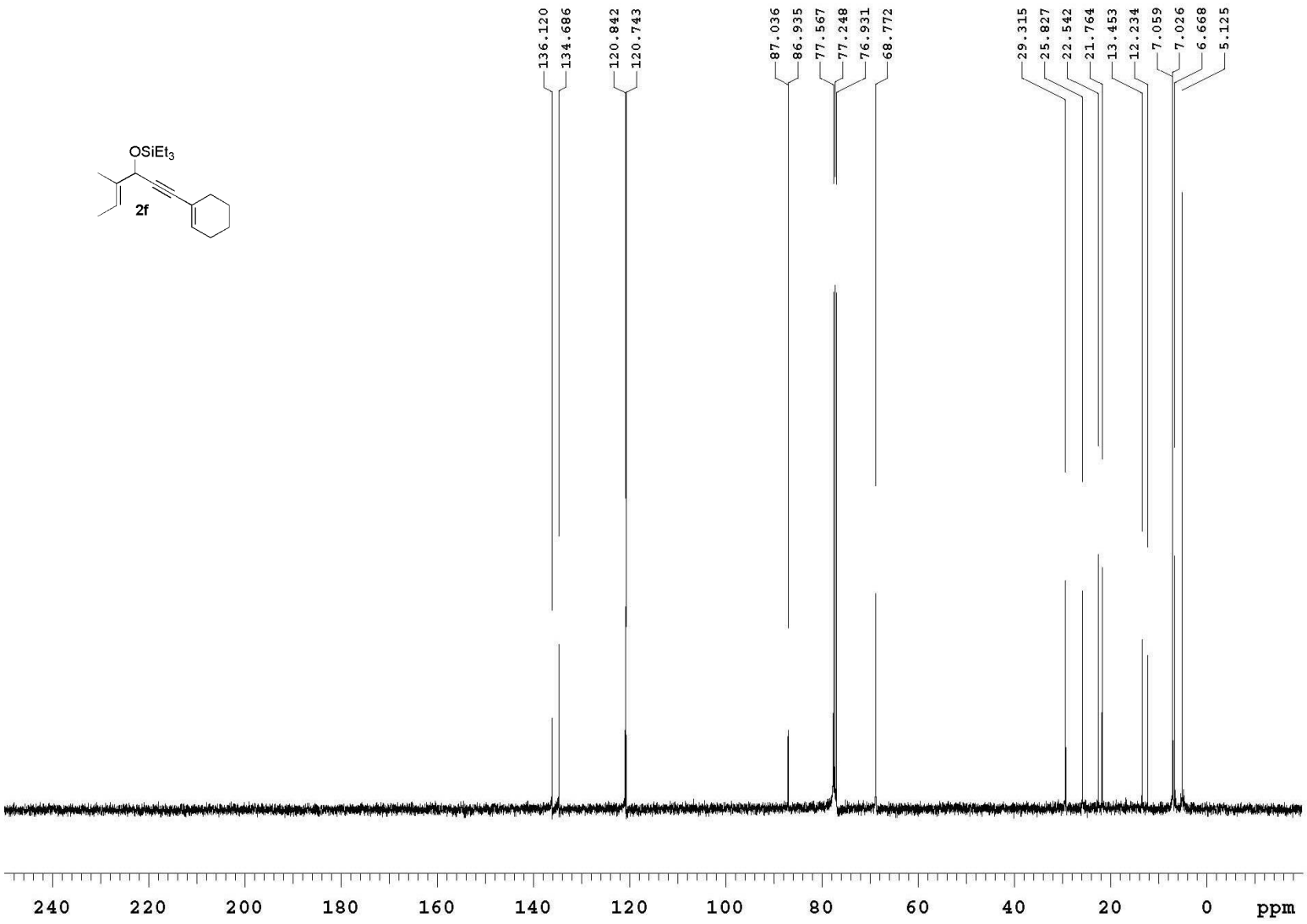


YKW-2-125
299.971 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.5 C -> actual temp = 27.0 C, id300 probe

Pulse Sequence: s2pul

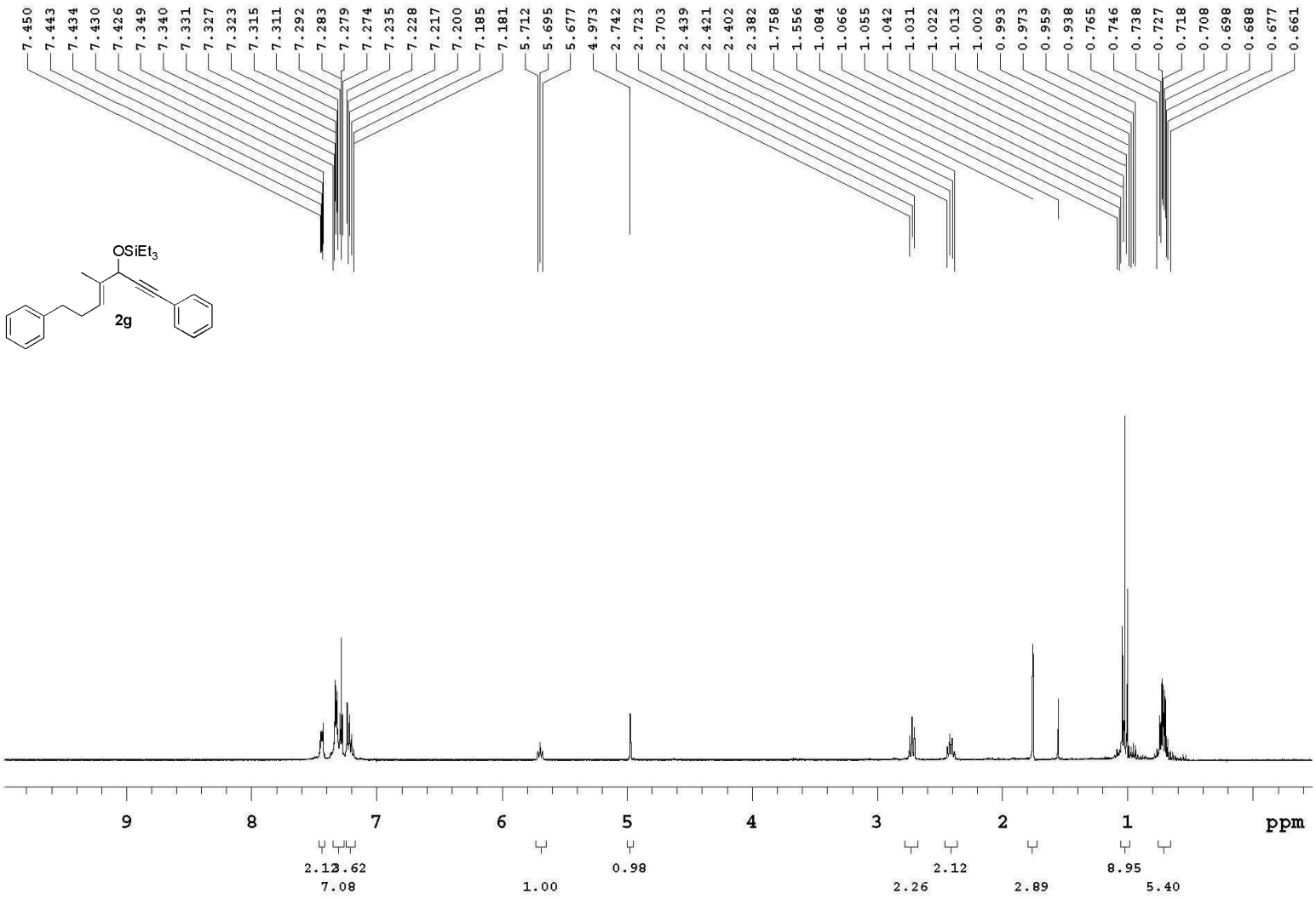


100.690 MHz C13[H1] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe
date: Aug 12 2009 sweep width: 27174Hz acq.time: 2.0s relax.time: 0.1s # scans: 1024 dig.res.: 0.2 Hz/pt hz/mm:113.2
spectrometer:d601 file:/mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/2009.08/2009.08.12.m4_YKW-2-125_17:42_C13_1D.fid
Pulse Sequence: s2pul

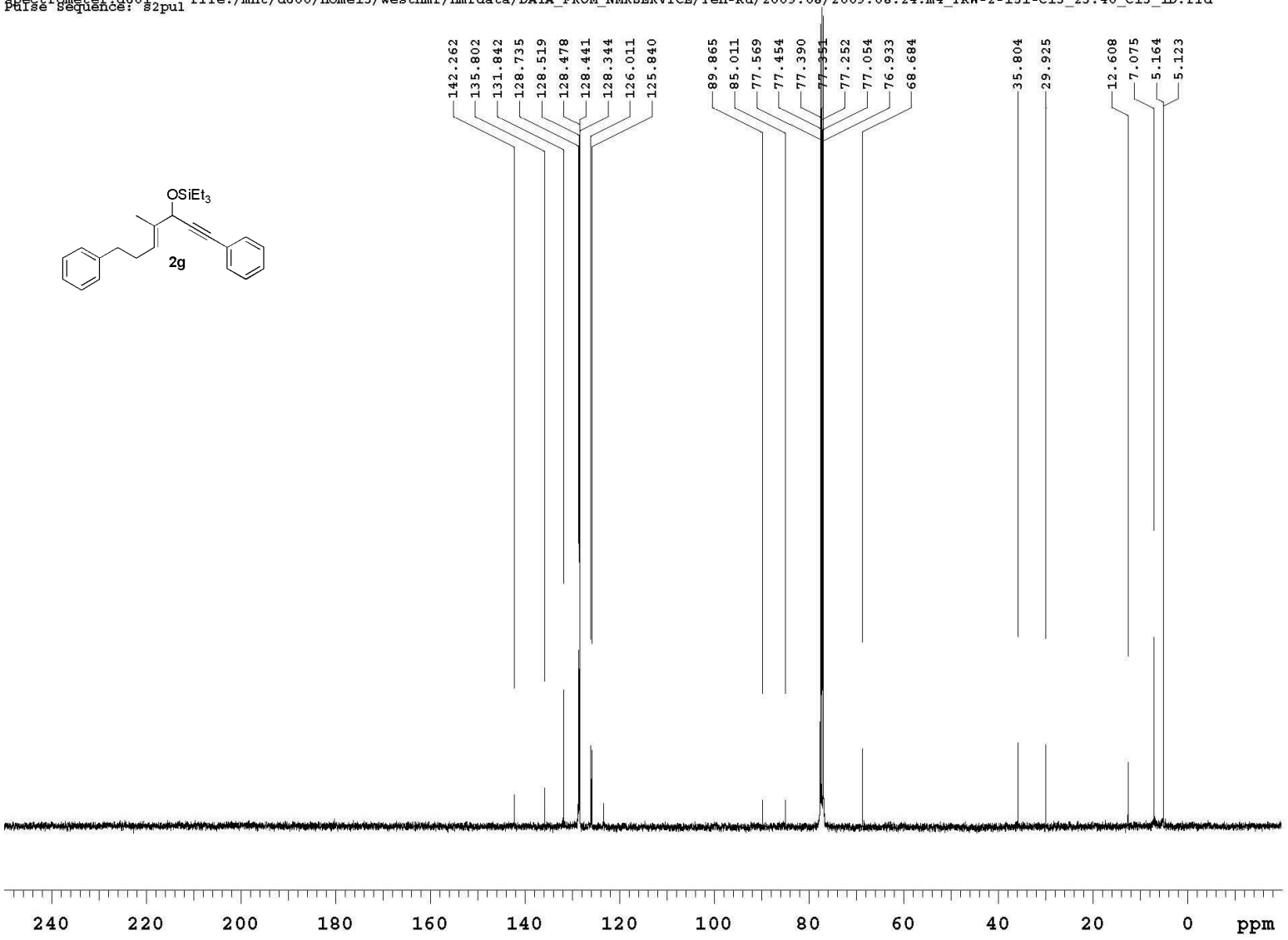


Yen-Ku, YKW-2-151
400.393 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe

Pulse Sequence: s2pul

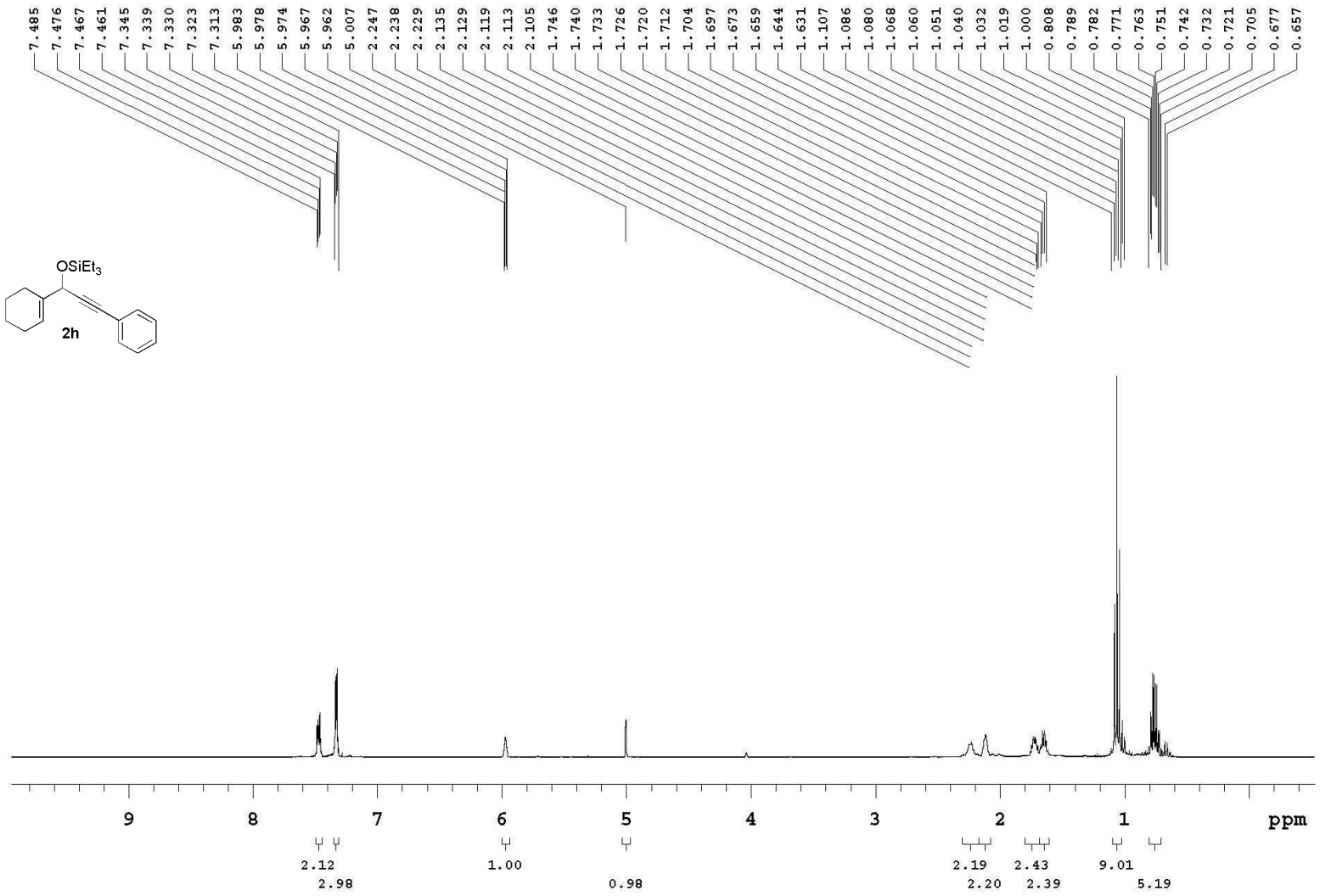


100.690 MHz C13[H1] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe
date: Aug 24 2009 sweep width: 27174Hz acq.time: 2.0s relax.time: 0.1s # scans: 4000 dig.res.: 0.2 Hz/pt hz/mm:113.2
spectrometer:d601 file:/mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/2009.08/2009.08.24.m4_YKW-2-151-C13_23:40_C13_1D.fid
Pulse sequence: s2pul



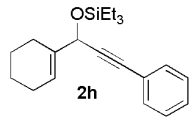
Yen-Ku, YKW-1-151
400.393 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe

Pulse Sequence: s2pul



100.690 MHz C13[H1] apt in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe
date Jun 9 2009 sw=27154Hz acq.time=2.0s relax.time: 0.1s # scans: 512 dig.res.: 0.4 Hz/pt hz/mm:113.2
spectrometer:d601 file:/mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/2009.06/2009.06.9.m4_YKW-1-151-C13_09:49_C13_apt.fid

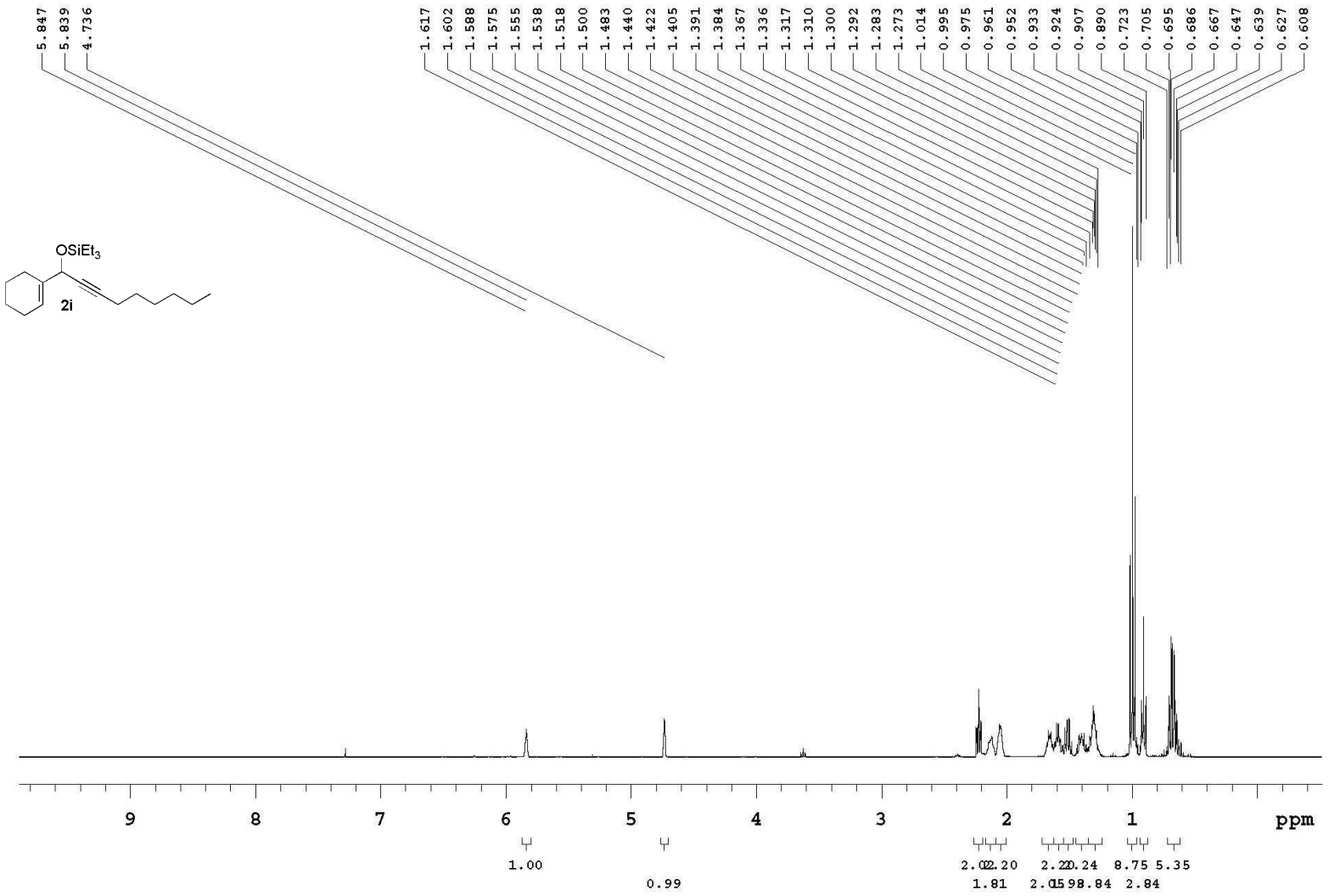
Pulse Sequence: apt



2h

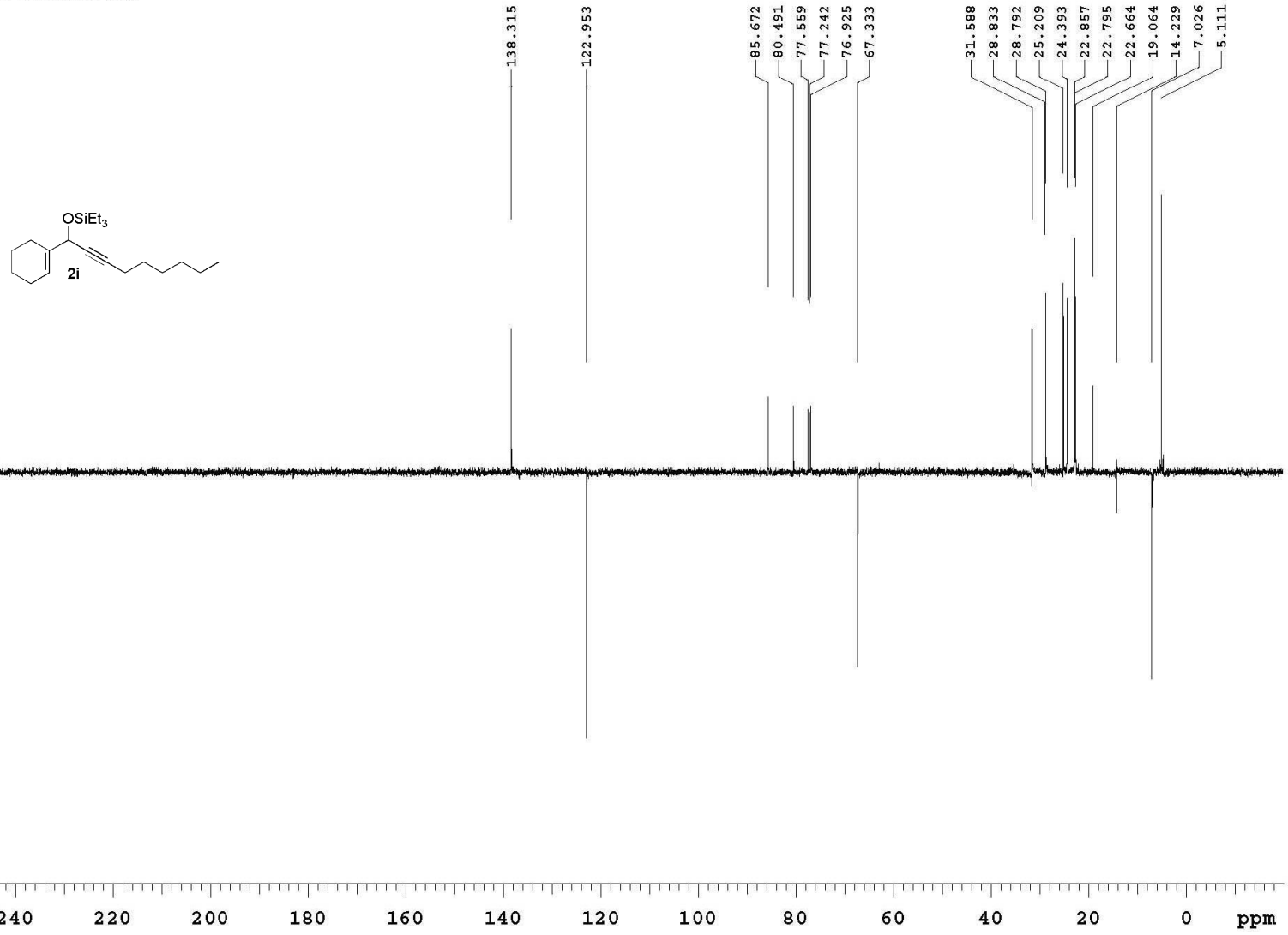
Yen-Ku, YKW-1-189-M4
400.393 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe

Pulse Sequence: s2pul



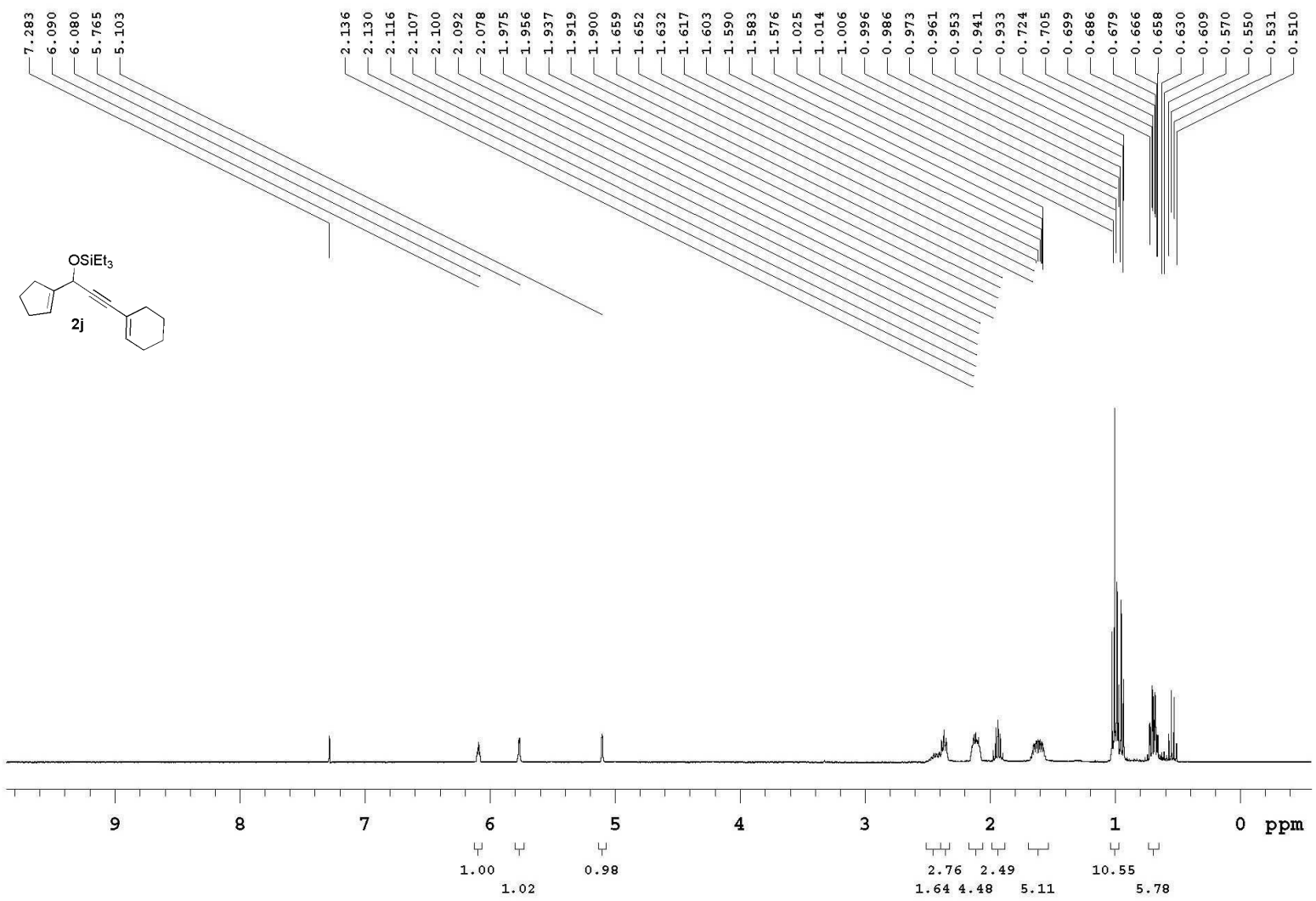
100.690 MHz C13[H1] apt in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe
date:Jun 16, 2019 sweep width: 27144Hz acq.time: 2.0s relax.time: 0.1s # scans: 512 dig.res.: 0.4 Hz/pt hz/mm:113.2
spectrometer:d601 file:/mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/2009.06/2009.06.16.m4_YKW-1-189-M4_17:19_C13_apt.fid

Pulse Sequence: apt

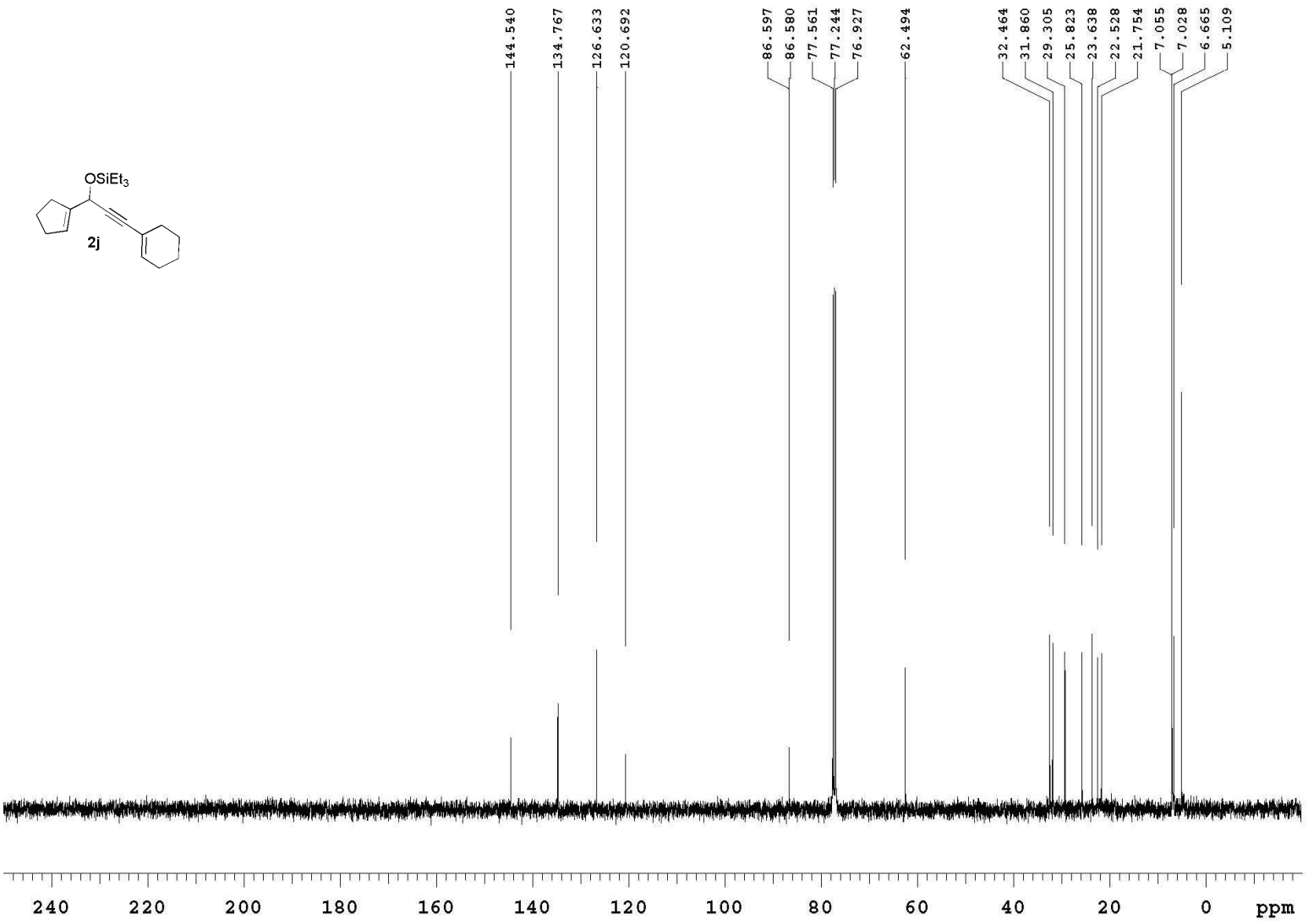


Yen-Ku, YKW-2-185
400.393 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe

Pulse Sequence: s2pul

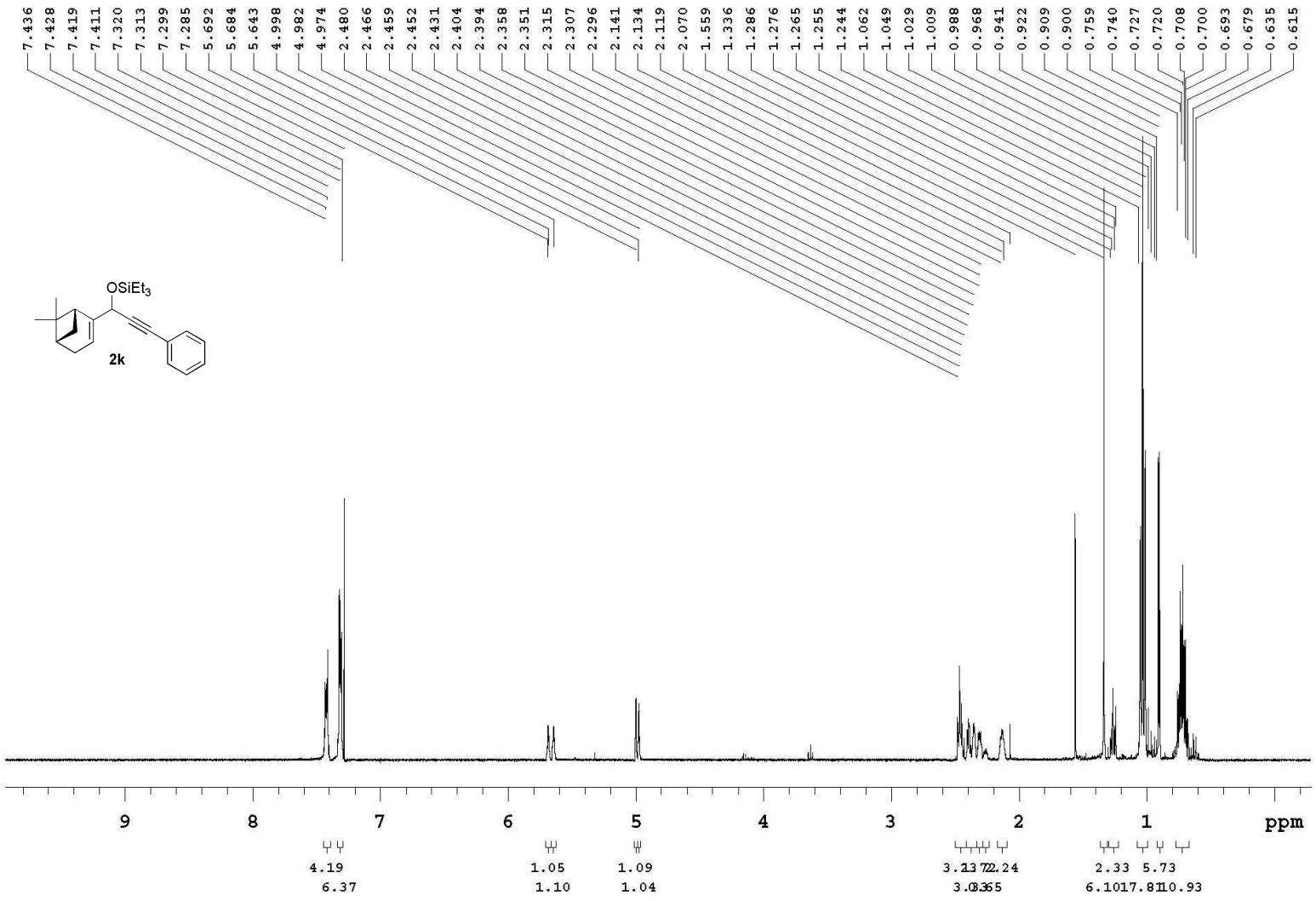


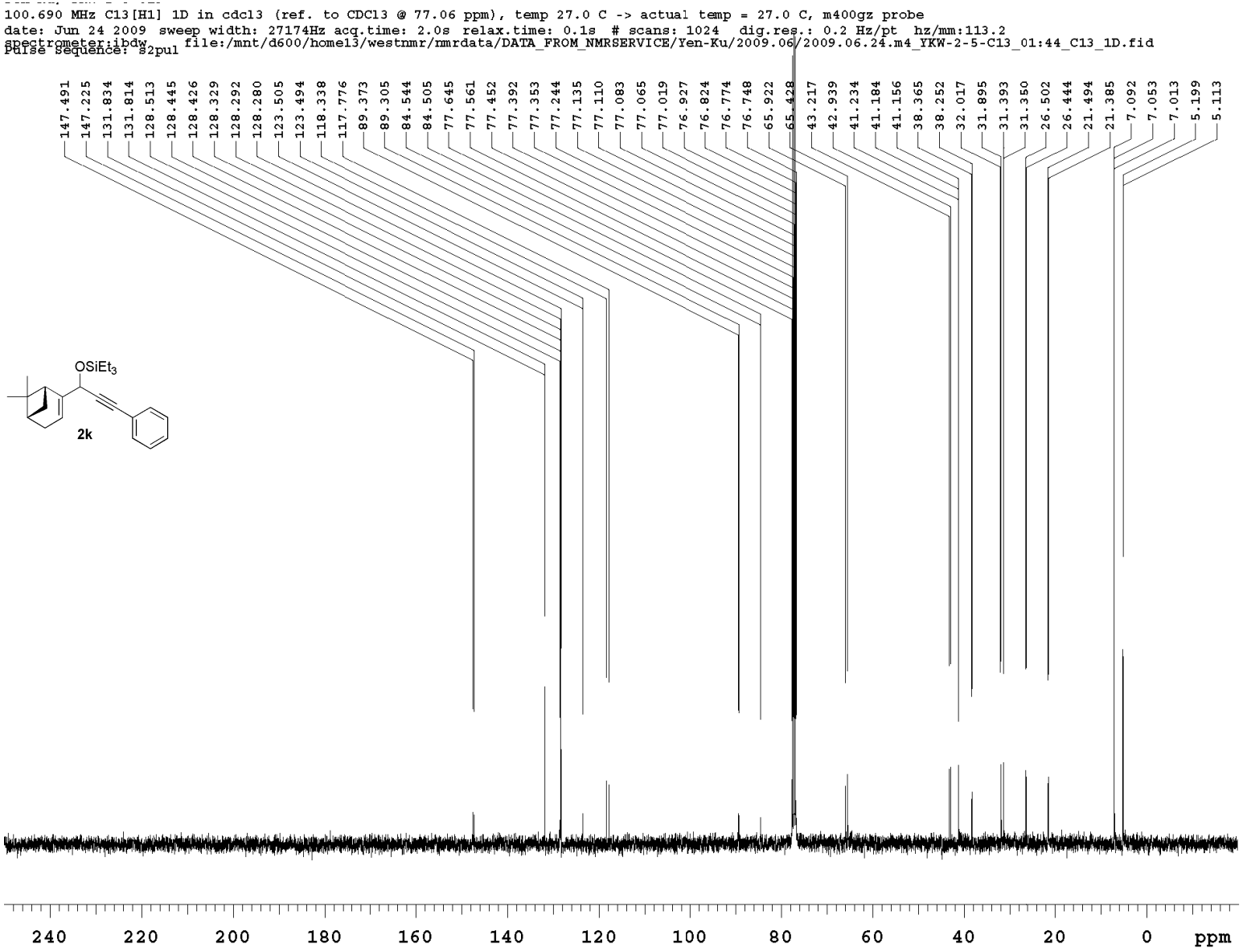
100.690 MHz C13[H1] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe
date: Sep 12 2009 sweep width: 27174Hz acq.time: 2.0s relax.time: 0.1s # scans: 512 dig.res.: 0.2 Hz/pt hz/mm:113.2
spectrometer:d601 file:/mnt/d600/home13/westnmr/rmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/2009.09/2009.09.12.m4_YKW-2-185_19:20_C13_1D.fid
Pulse sequence: s2pul



Yen-Ku, YKW-2-5-m4
400.393 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe

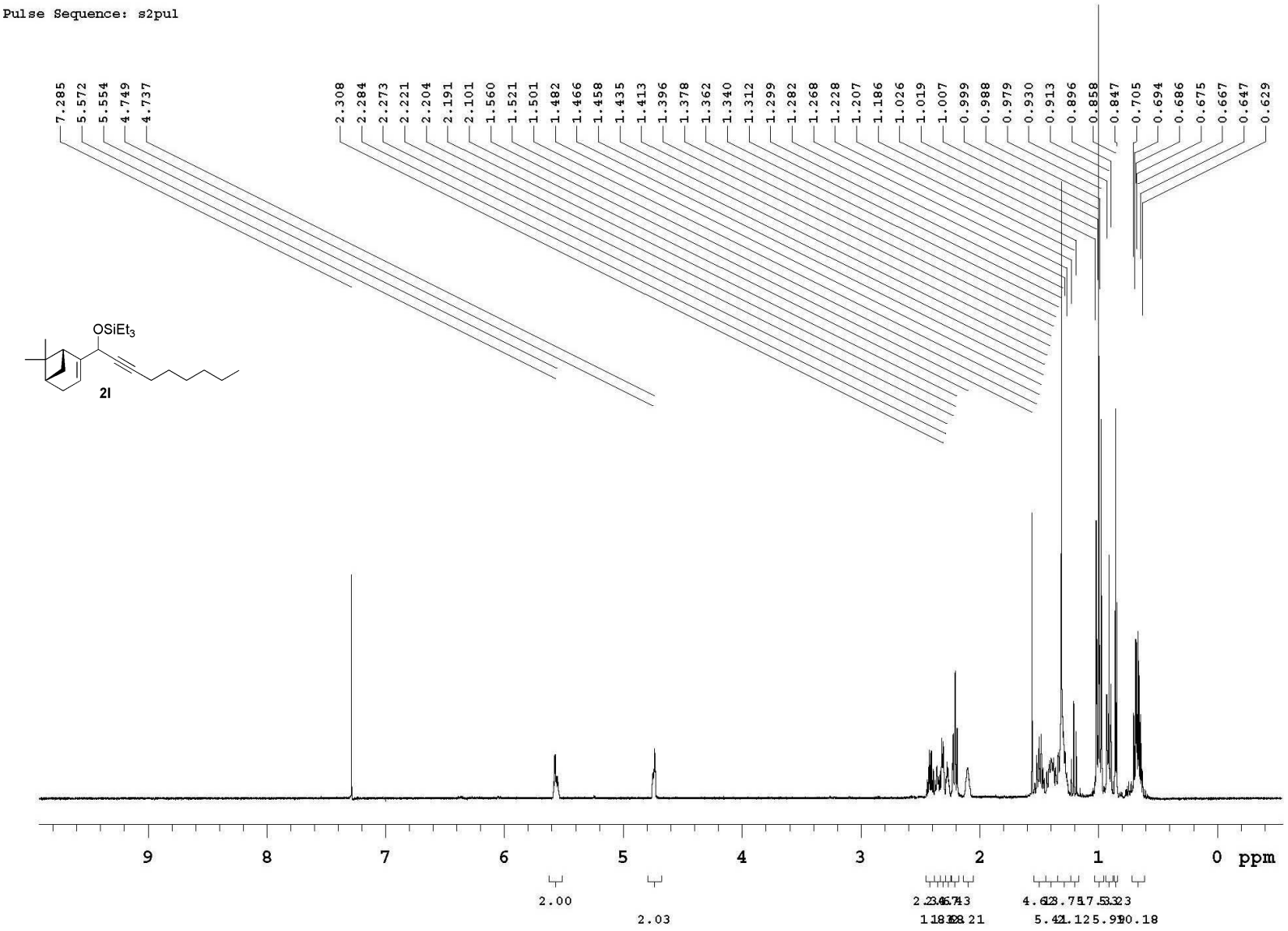
Pulse Sequence: s2pul



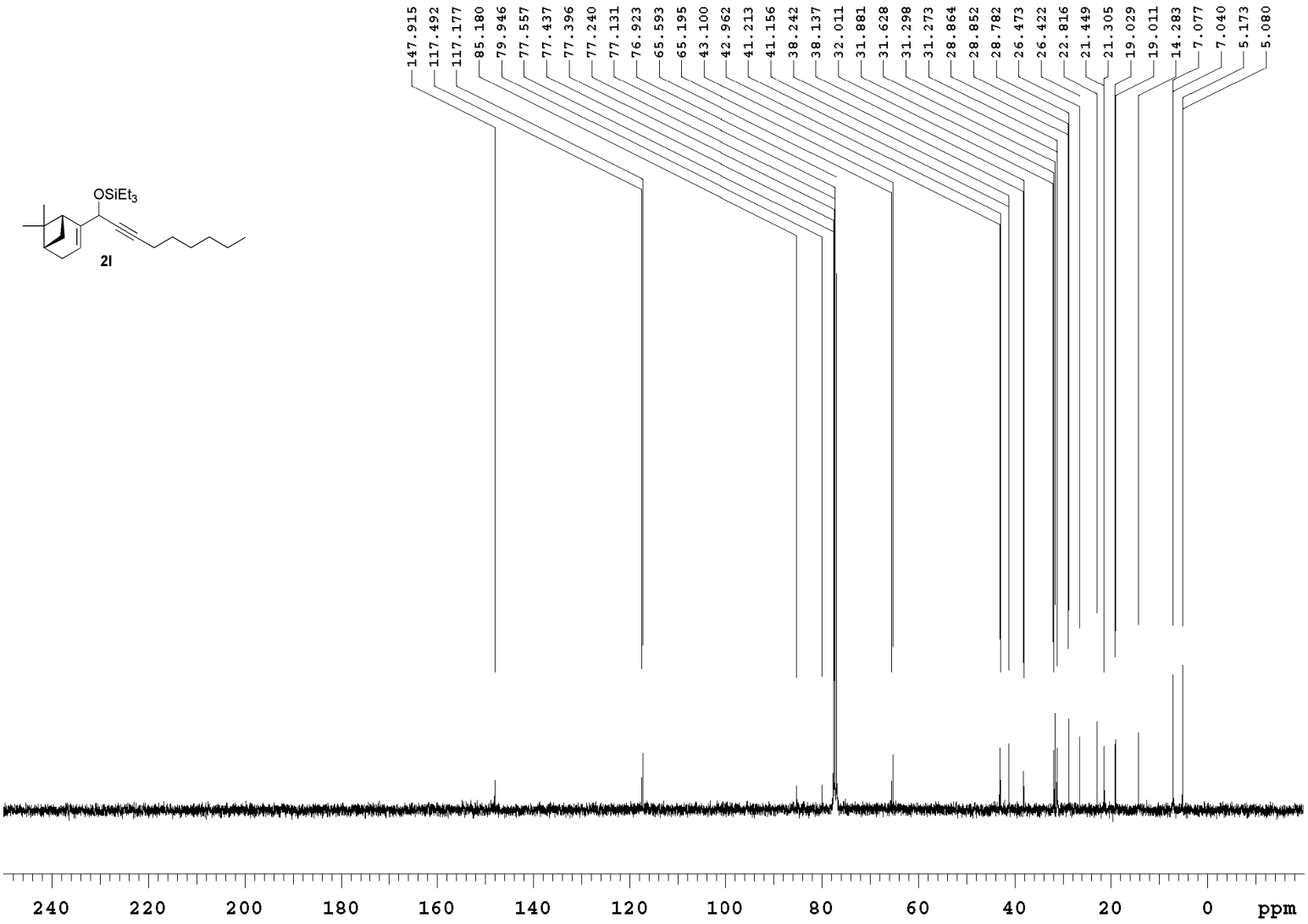

100.690 MHz C13[H1] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe
date: Jun 24 2009 sweep width: 27174Hz acq.time: 2.0s relax.time: 0.1s # scans: 1024 dig.res.: 0.2 Hz/pt hz/mm:113.2
spectrometer:ibdw file:/mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/2009.06/2009.06.24.m4_YKW-2-5-C13_01:44_C13_1D.fid
Pulse Sequence: s2pul
147.491
147.225
131.834
131.814
128.513
128.445
128.426
128.329
128.292
128.280
123.505
123.494
118.338
117.776
89.373
89.305
84.544
84.505
77.645
77.561
77.452
77.392
77.353
77.244
77.135
77.110
77.083
77.065
77.019
76.927
76.824
76.774
76.748
65.922
65.428
43.217
42.939
41.234
41.184
41.156
38.365
38.252
32.017
31.895
31.393
31.350
26.502
26.444
21.494
21.385
7.092
7.053
7.013
5.199
5.113
OSiEt3
2k
240
220
200
180
160
140
120
100
80
60
40
20
0
ppm

Yen-Ku, YKW-2-13-m4
400.393 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe

Pulse Sequence: s2pul

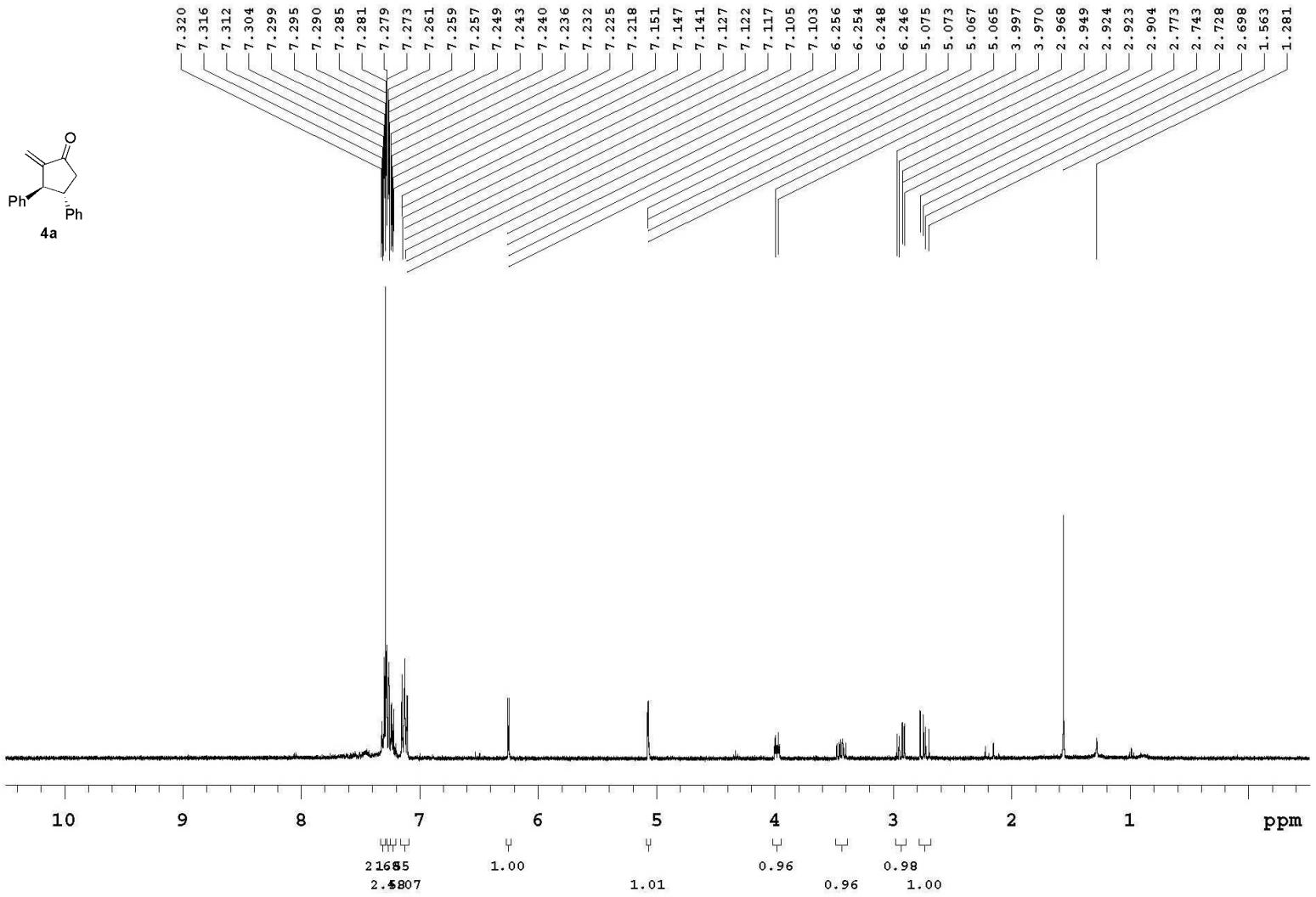


100.690 MHz C13[H1] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe
date: Jun 26 2009 sweep width: 27174Hz acq.time: 2.0s relax.time: 0.1s # scans: 800 dig.res.: 0.2 Hz/pt hz/mm:113.2
spectrometer:ibdw file:/mnt/d600/home13/westnmr/rmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/2009.06/2009.06.26.m4_YKW-2-13-m4_06:05_C13_1D.fid
Pulse sequence: s2pul

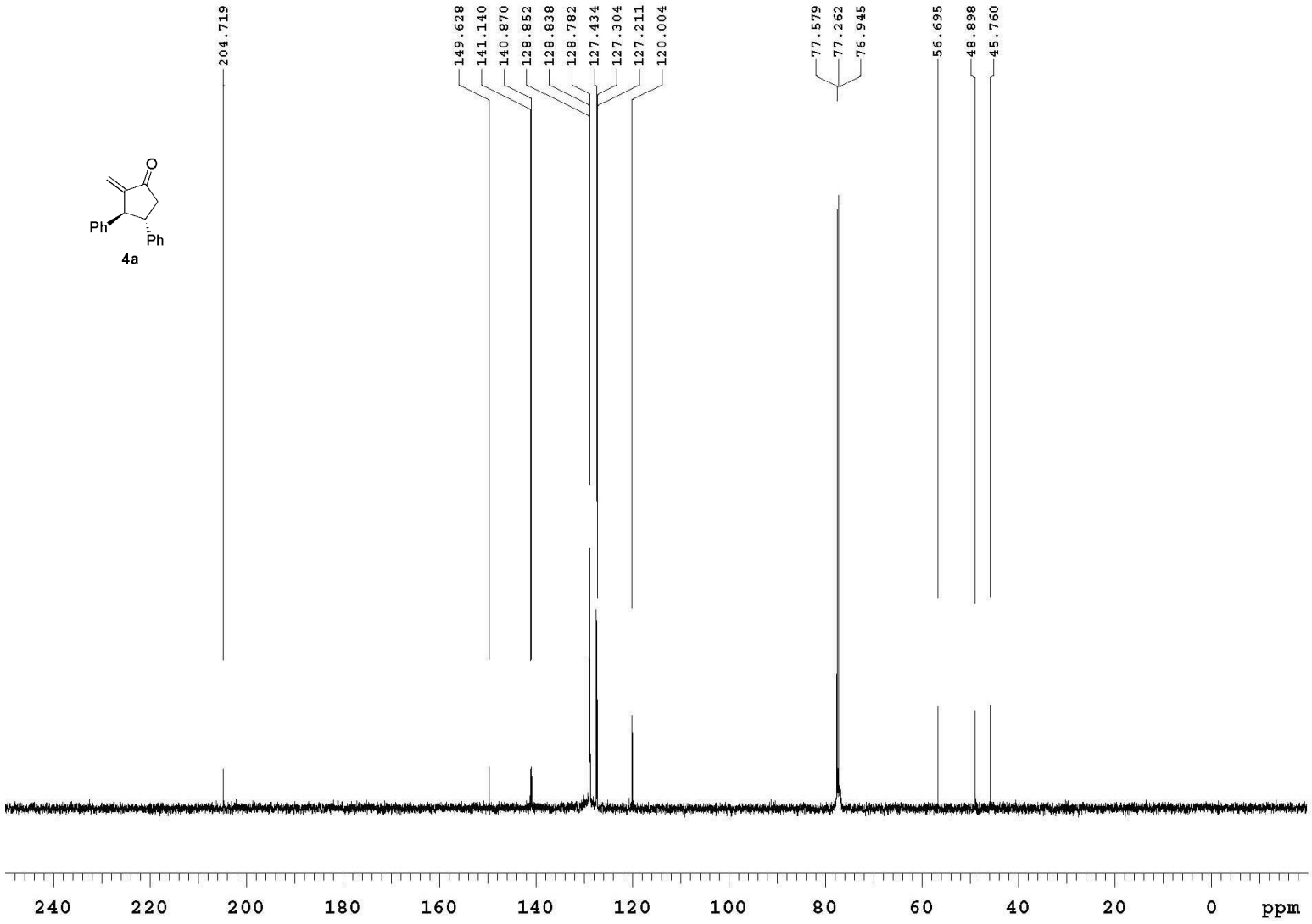


Yen-Ku, YKW-1-65-1
400.393 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe

Pulse Sequence: s2pul

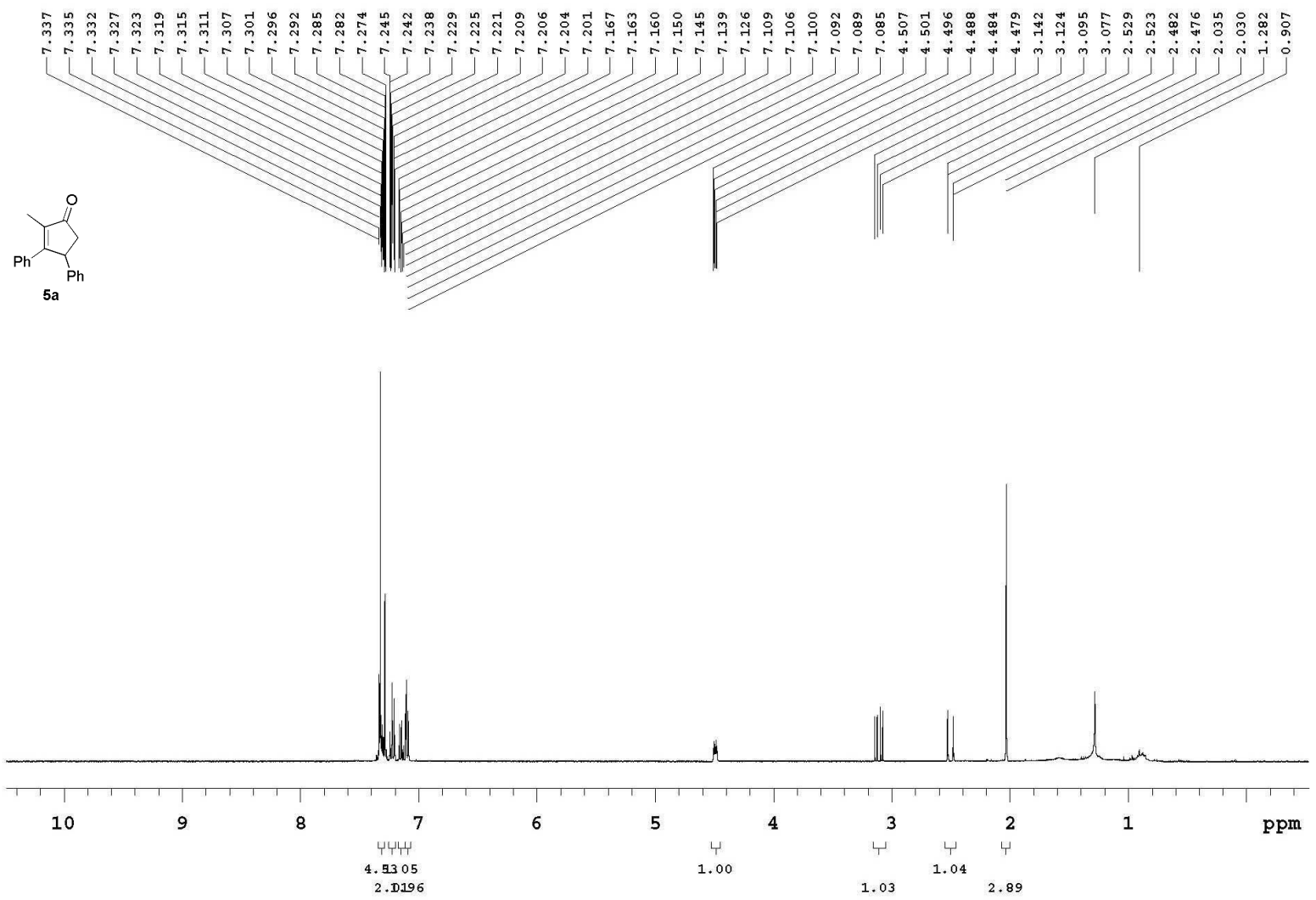


100.690 MHz C13[H1] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe
date: Oct 26 2009 sweep width: 27174Hz acq.time: 2.0s relax.time: 0.1s # scans: 1024 dig.res.: 0.2 Hz/pt hz/mm:113.2
spectrometer:d601 file:/mnt/d600/home13/westnmr/rmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/2009.10/2009.10.26.m4_YKW-3-65-exo_18:39_C13_1D.fid
Pulse sequence: s2pul

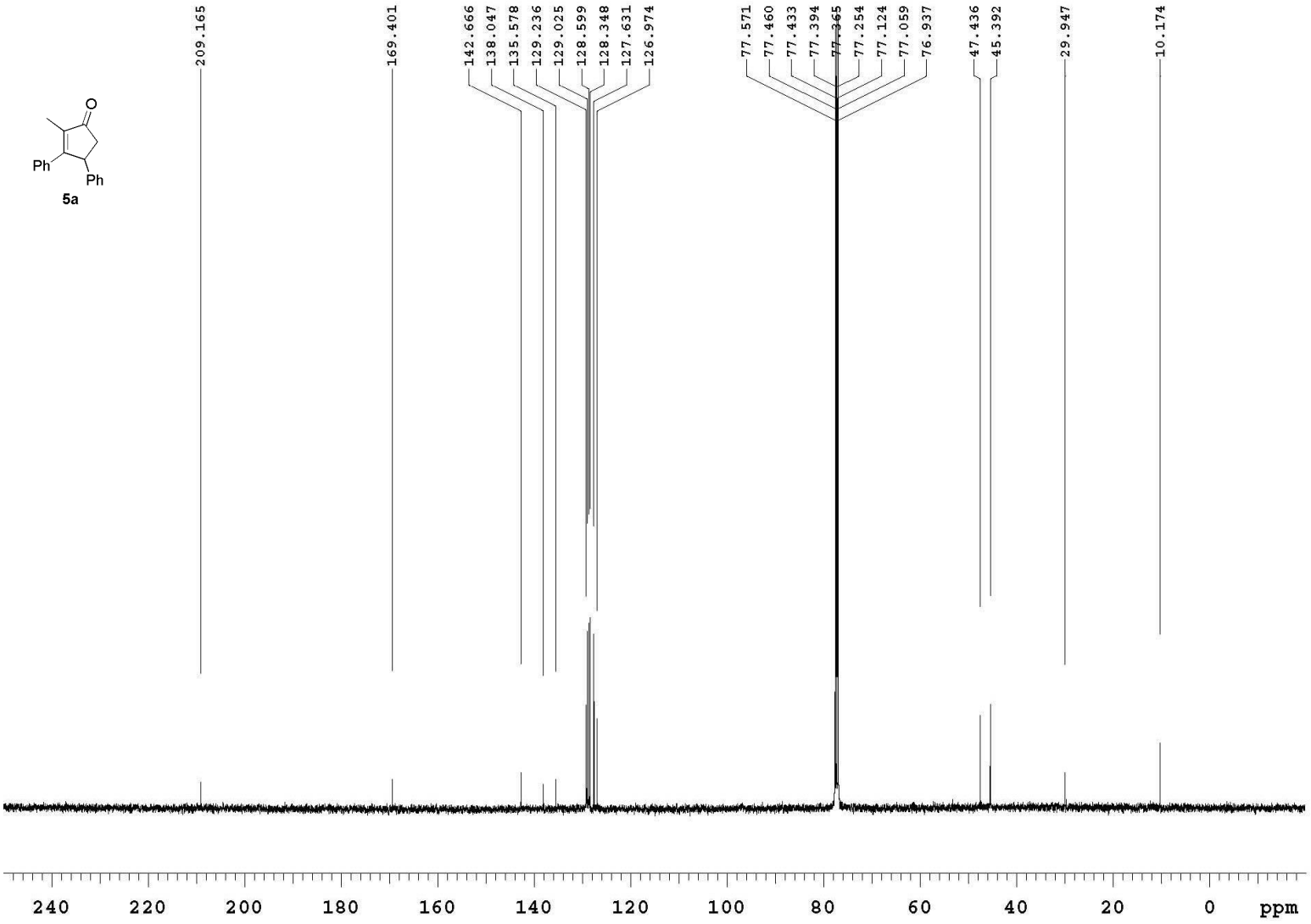


Yen-Ku, YKW-1-65-3-H
400.393 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe

Pulse Sequence: s2pul

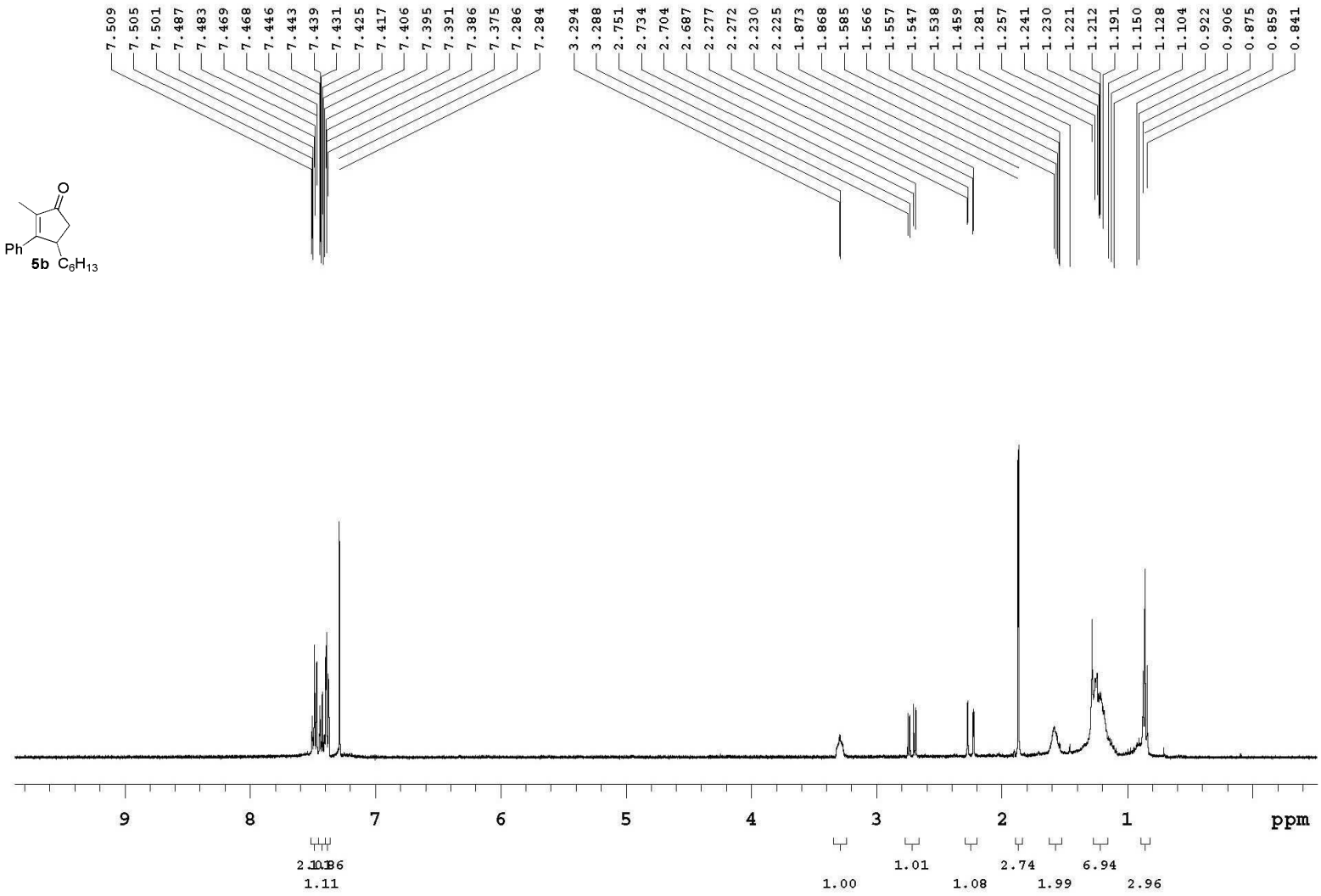


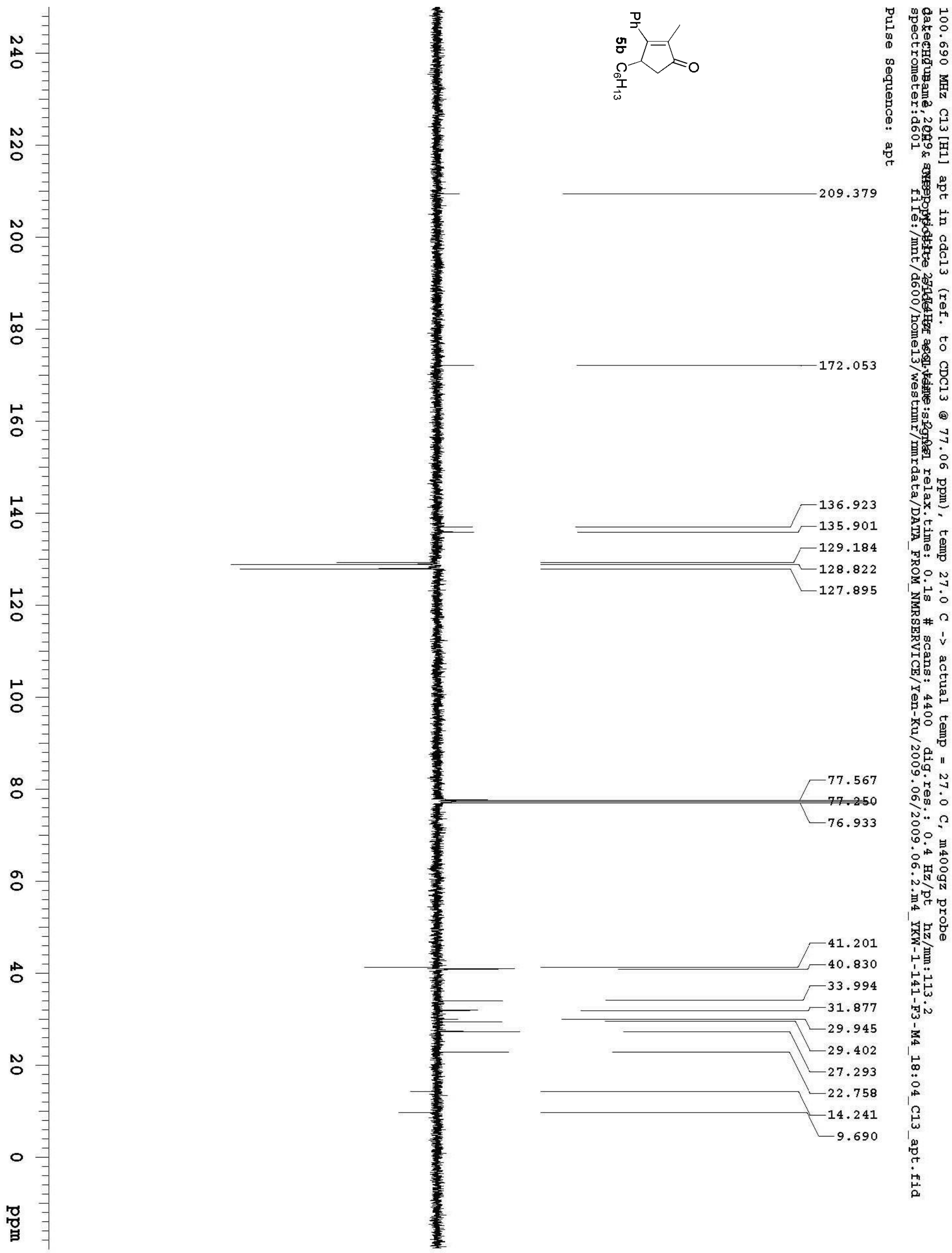
100.690 MHz C13[H1] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe
date: May 16 2009 sweep width: 27174Hz acq.time: 2.0s relax.time: 0.1s # scans: 3072 dig.res.: 0.2 Hz/pt hz/mm:113.2
spectrometer:d601 file:/mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/2009.05/2009.05.16.m4_YKW-1-65-endo_04:30_C13_1D.fid
Pulse Sequence: s2pul



Yen-Ku, YKW-1-141-F3-M4
400.393 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe

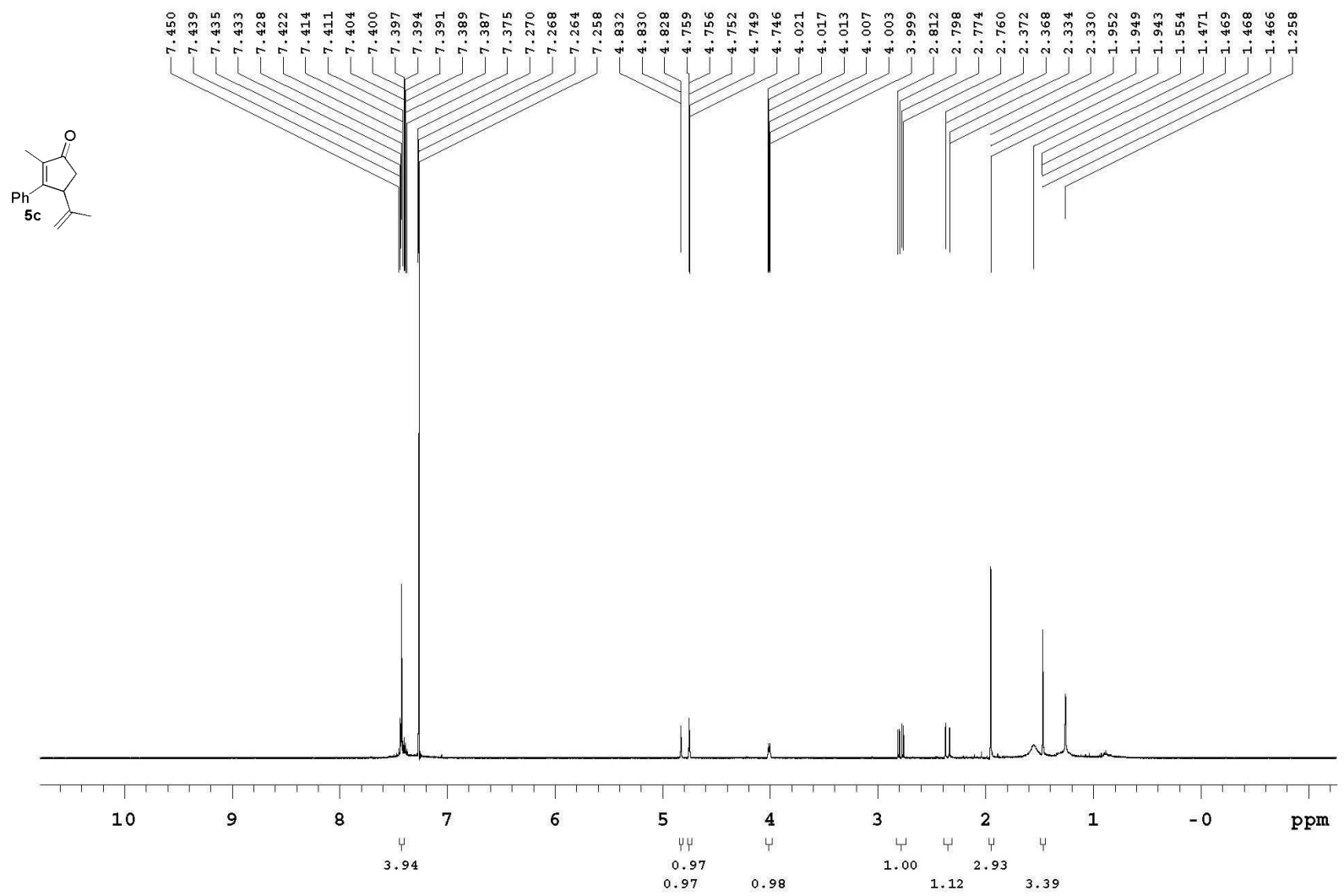
Pulse Sequence: s2pul

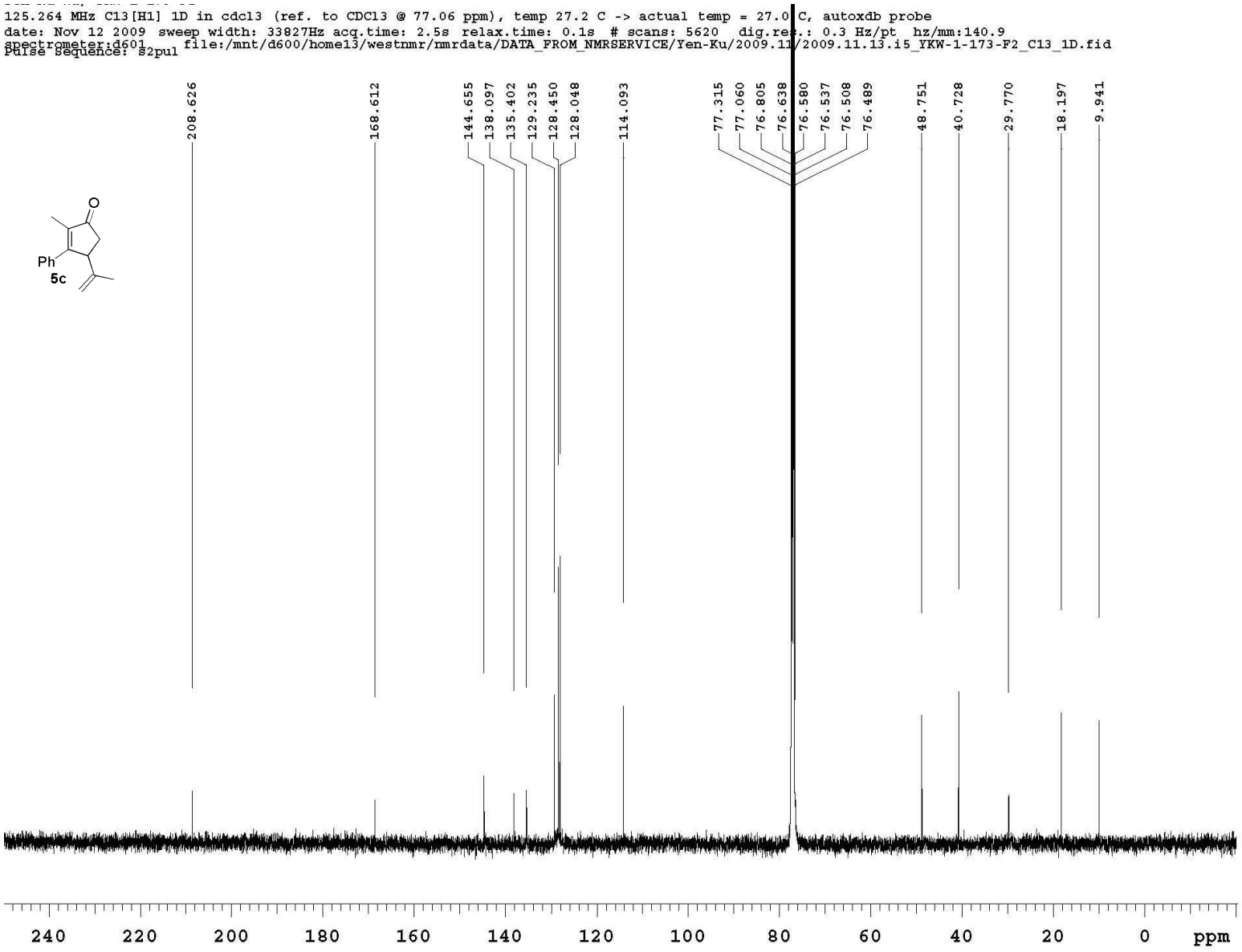



100.690 MHz C13[H1] apt in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe
relax.time: 0.1s # scans: 4400 dig.res.: 0.4 Hz/pt hz/mm:113.2
spectrometer:d601 file:/mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/2009.06/2009.06.2.m4_YKW-1-141-F3-M4_18:04_C13_apt.fid
Pulse Sequence: apt
Ph
5b
C_6H_{13}
O
209.379
172.053
136.923
135.901
129.184
128.822
127.895
77.567
77.250
76.933
41.201
40.830
33.994
31.877
29.945
29.402
27.293
22.758
14.241
9.690
240
220
200
180
160
140
120
100
80
60
40
20
0
ppm

Yen-Ku Wu, YKW-1-173-F2
498.122 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.2 C -> actual temp = 27.0 C, autoxdb probe

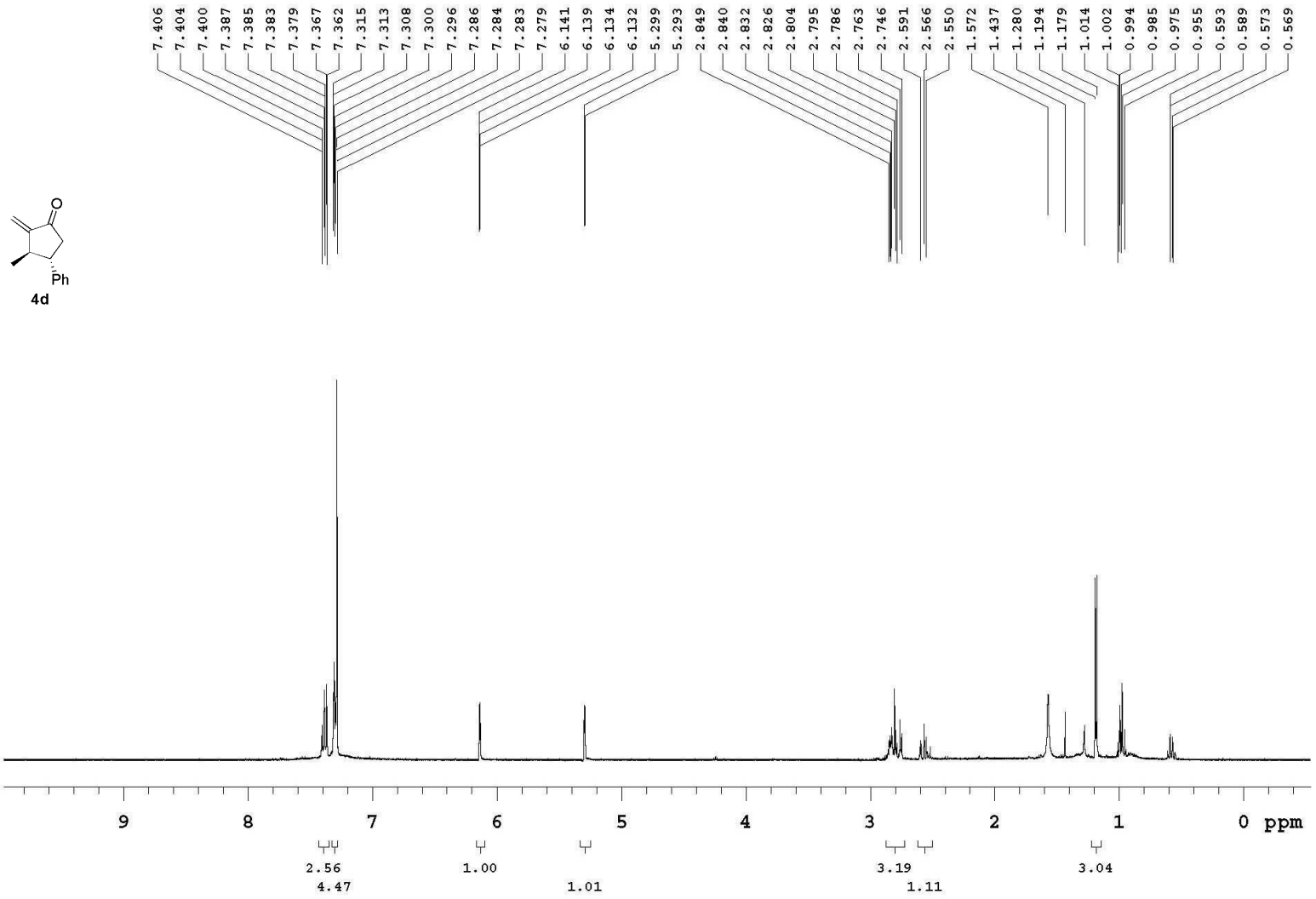
Pulse Sequence: s2pul

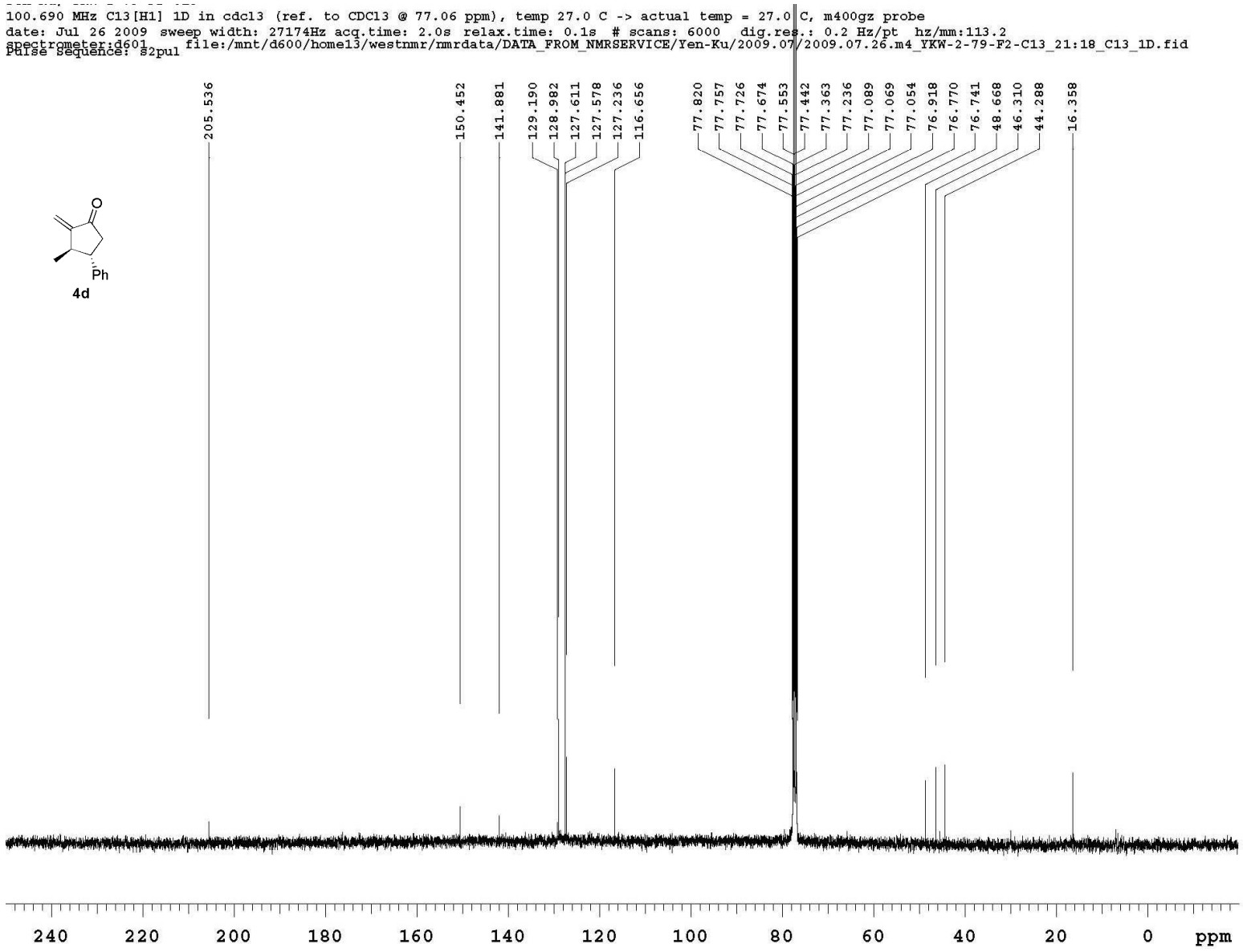



125.264 MHz C13[H1] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.2 C -> actual temp = 27.0 C, autoxdb probe
date: Nov 12 2009 sweep width: 33827Hz acq.time: 2.5s relax.time: 0.1s # scans: 5620 dig.res.: 0.3 Hz/pt hz/mm:140.9
spectrometer:d601 file:/mnt/d600/home13/westnmr/rmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/2009.11/2009.11.13.i5_YKW-1-173-F2_C13_1D.fid
Pulse sequence: s2pul
208.626
168.612
144.655
138.097
135.402
129.235
128.450
128.048
114.093
77.315
77.060
76.805
76.638
76.580
76.537
76.508
76.489
48.751
40.728
29.770
18.197
9.941
O
Ph
5c
240
220
200
180
160
140
120
100
80
60
40
20
0
ppm

Yen-Ku, YKW-2-79-F2-H
400.393 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe

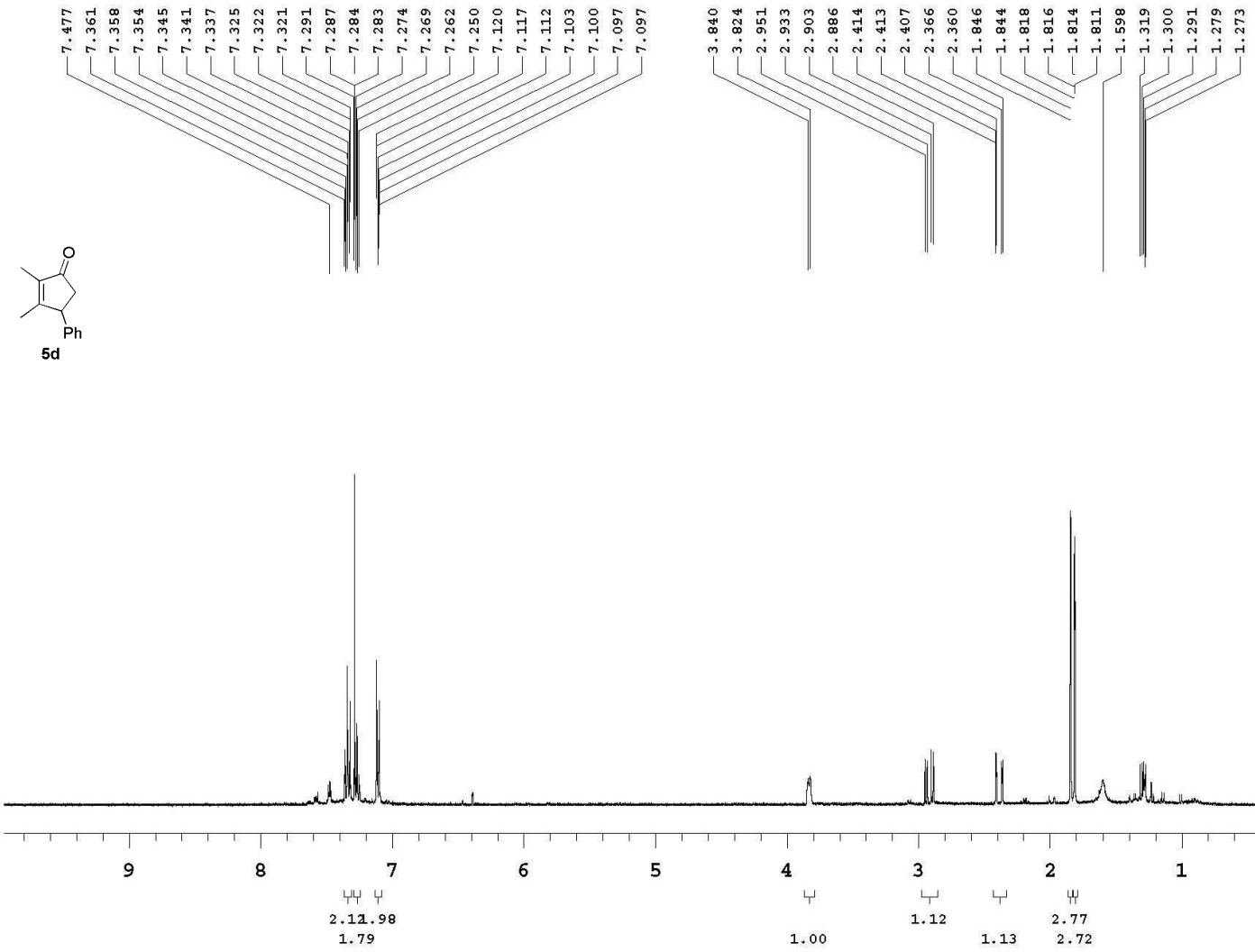
Pulse Sequence: s2pul



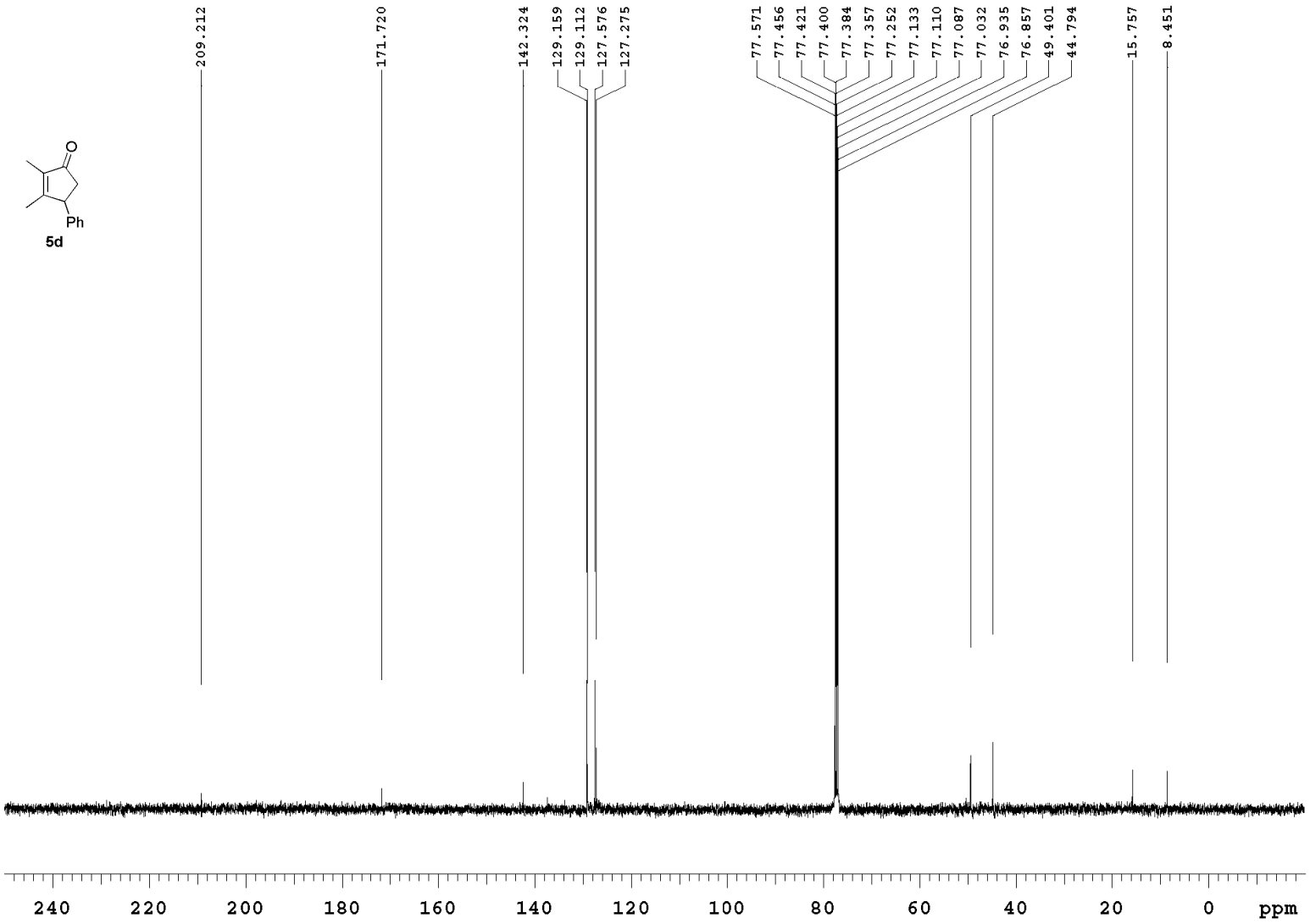

100.690 MHz C13[H1] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe
date: Jul 26 2009 sweep width: 27174Hz acq.time: 2.0s relax.time: 0.1s # scans: 6000 dig.res.: 0.2 Hz/pt hz/mm:113.2
spectrometer:d601 file:/mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/2009.07/2009.07.26.m4_YKW-2-79-F2-C13_21:18_C13_1D.fid
Pulse sequence: s2pul
205.536
150.452
141.881
129.190
128.982
127.611
127.578
127.236
116.656
77.820
77.757
77.726
77.674
77.553
77.442
77.363
77.236
77.089
77.069
77.054
76.918
76.770
76.741
48.668
46.310
44.288
16.358
O
Ph
4d
240
220
200
180
160
140
120
100
80
60
40
20
0
ppm

Yen-Ku, YKW-2-79-F3
400.393 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe

Pulse Sequence: s2pul

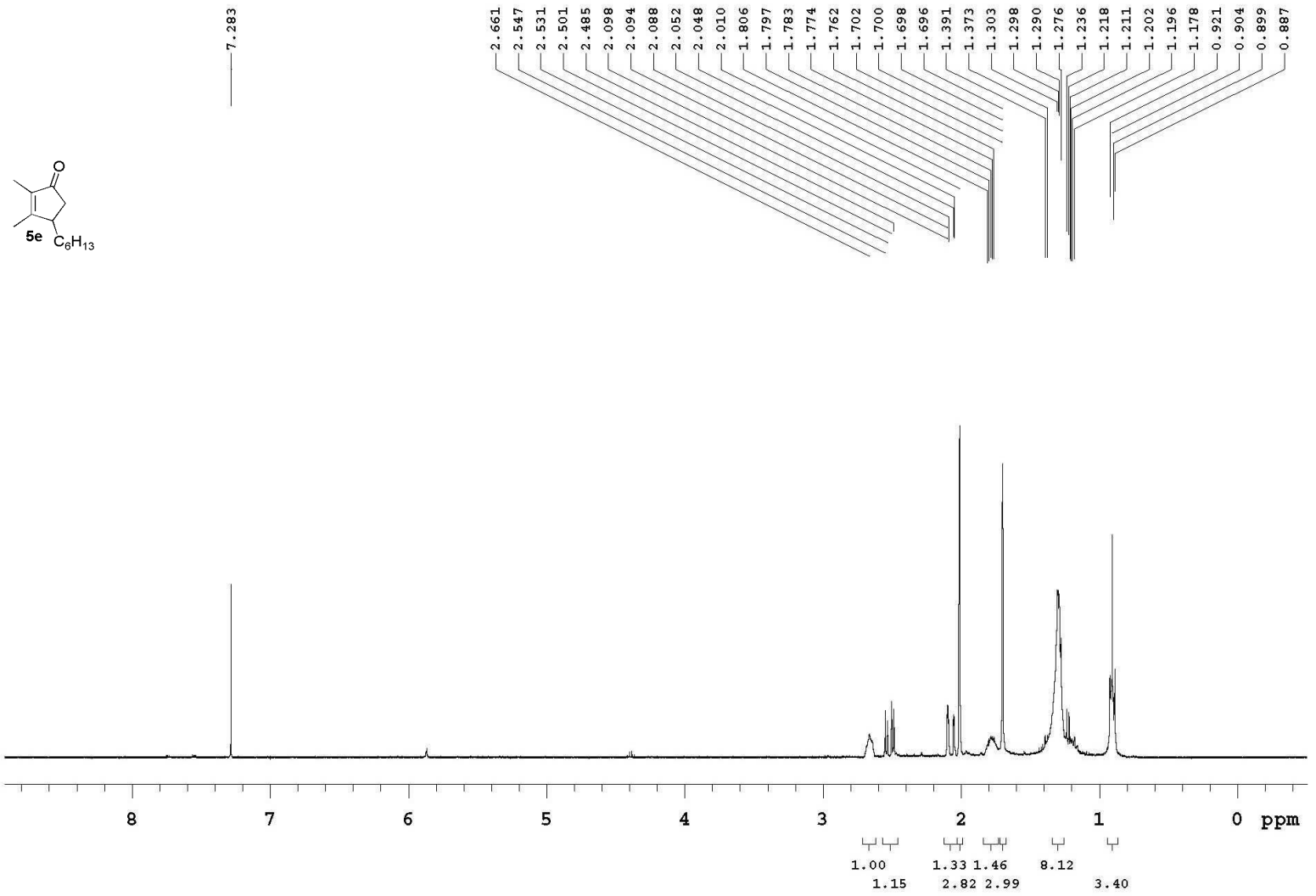


100.690 MHz C13[H1] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe
date: Jul 24 2009 sweep width: 27174Hz acq.time: 2.0s relax.time: 0.1s # scans: 1024 dig.res.: 0.2 Hz/pt hz/mm:113.2
spectrometer:ibdw file:/mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/2009.07/2009.07.24.m4_YKW-2-79-F3_14:45_C13_1D.fid
Pulse sequence: s2pul

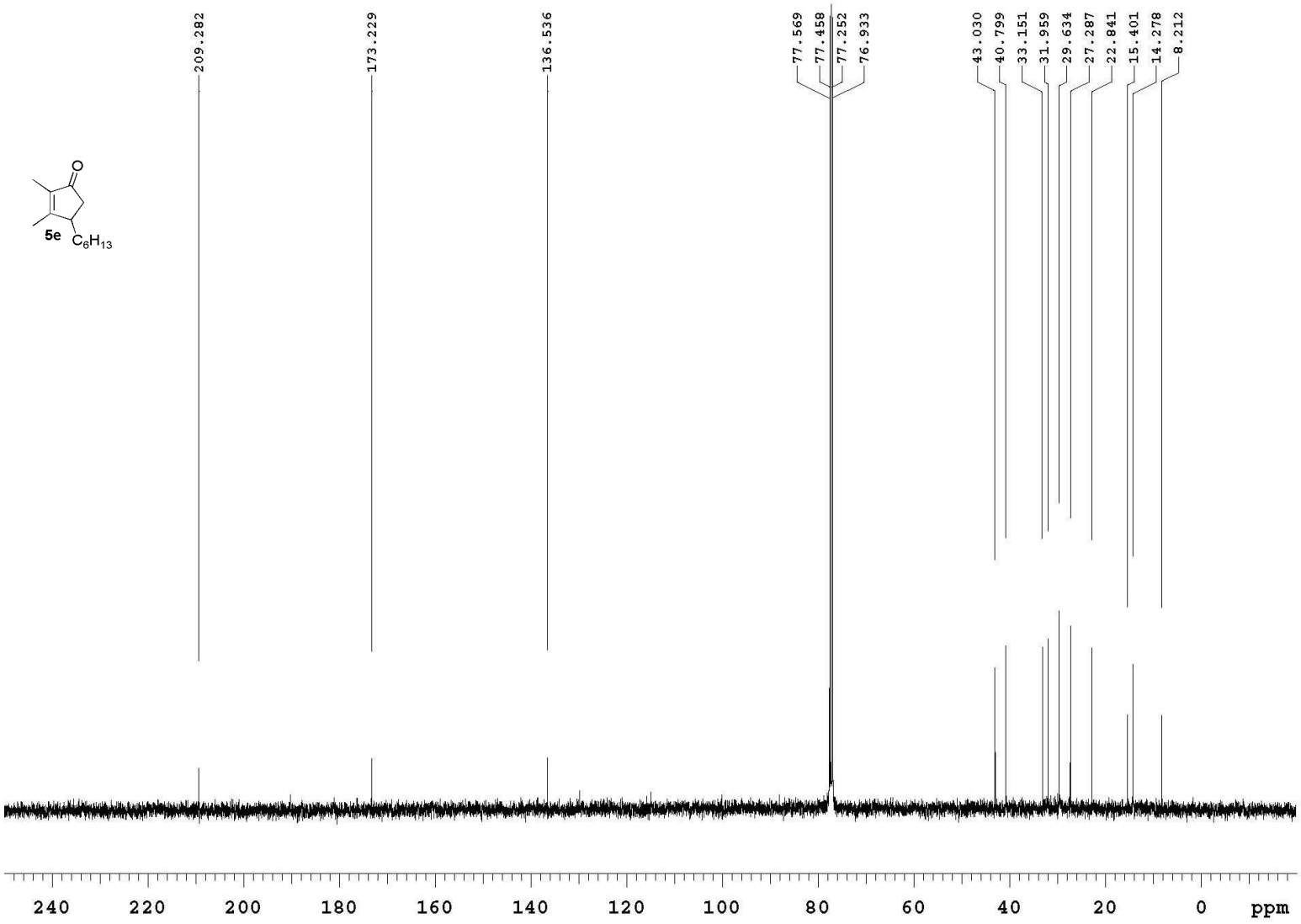


Yen-Ku, YKW-2-127-F3
400.393 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe

Pulse Sequence: s2pul

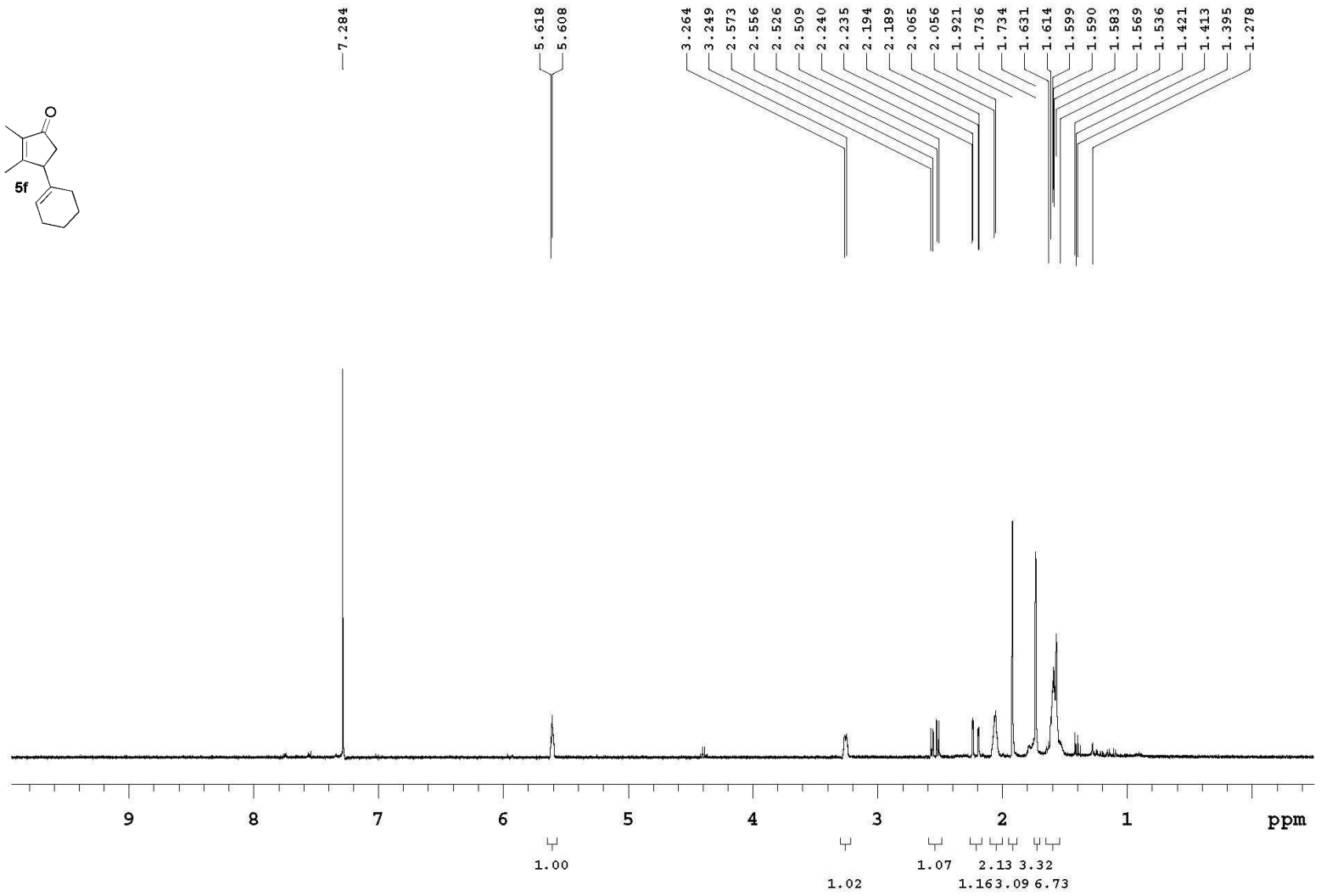


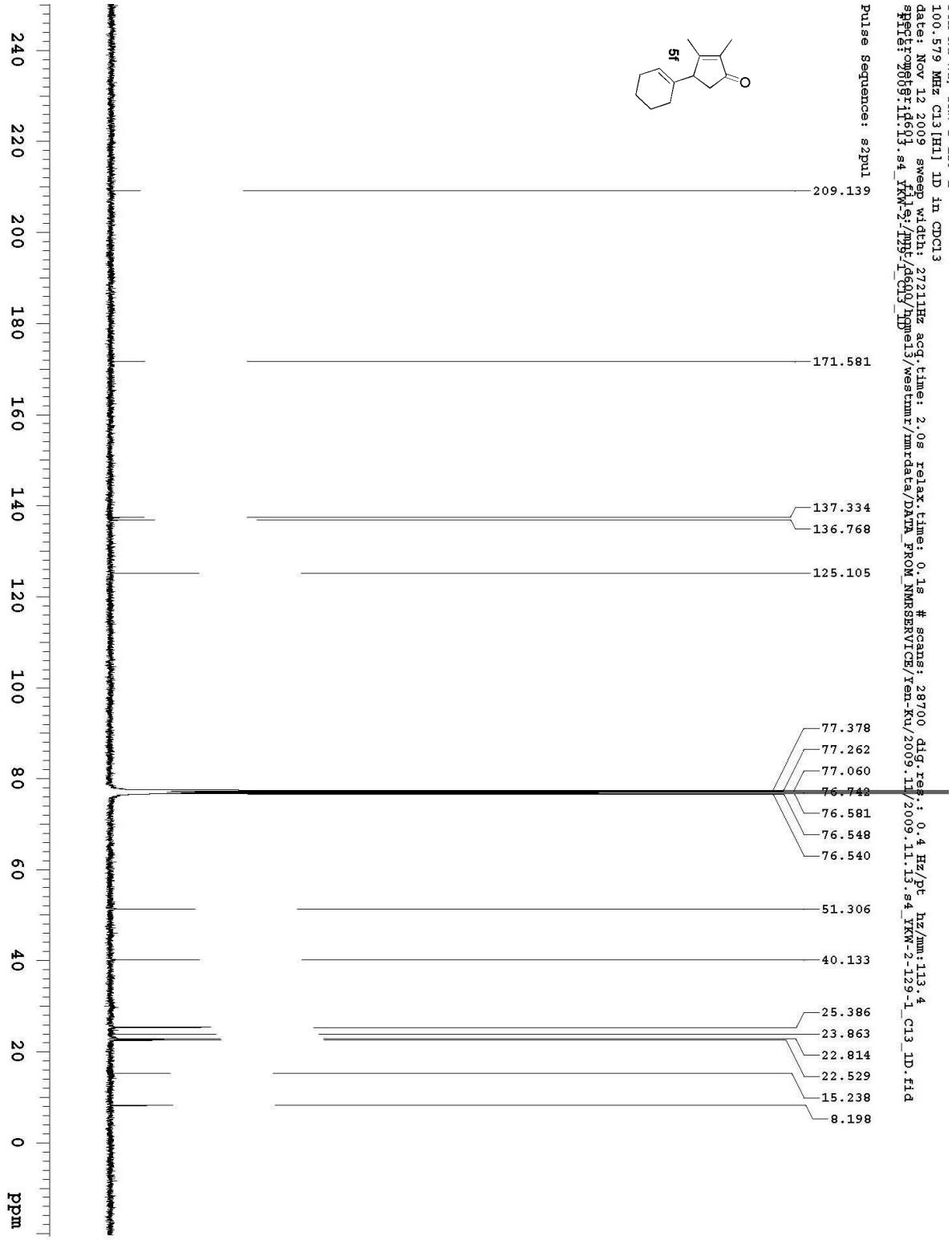
100.690 MHz C13[H1] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe
date: Aug 18 2009 sweep width: 27174Hz acq.time: 2.0s relax.time: 0.1s # scans: 1024 dig.res.: 0.2 Hz/pt hz/mm:113.2
spectrometer:d601 file:/mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/2009.08/2009.08.18.m4_YKW-2-127-F3_17:19_C13_1D.fid
Pulse Sequence: s2pul



Yen-Ku, YKW-2-129-product1
400.393 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe

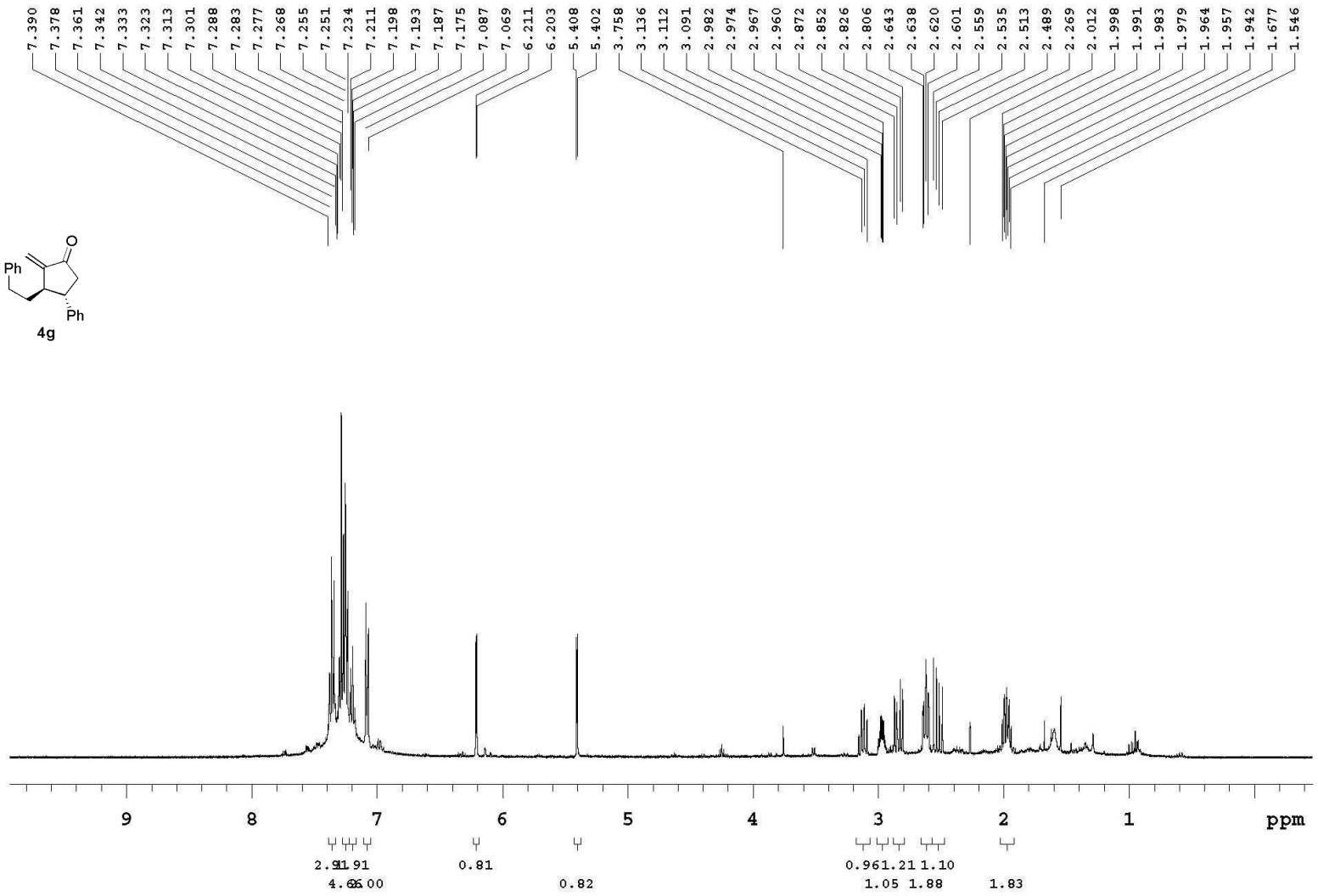
Pulse Sequence: s2pul



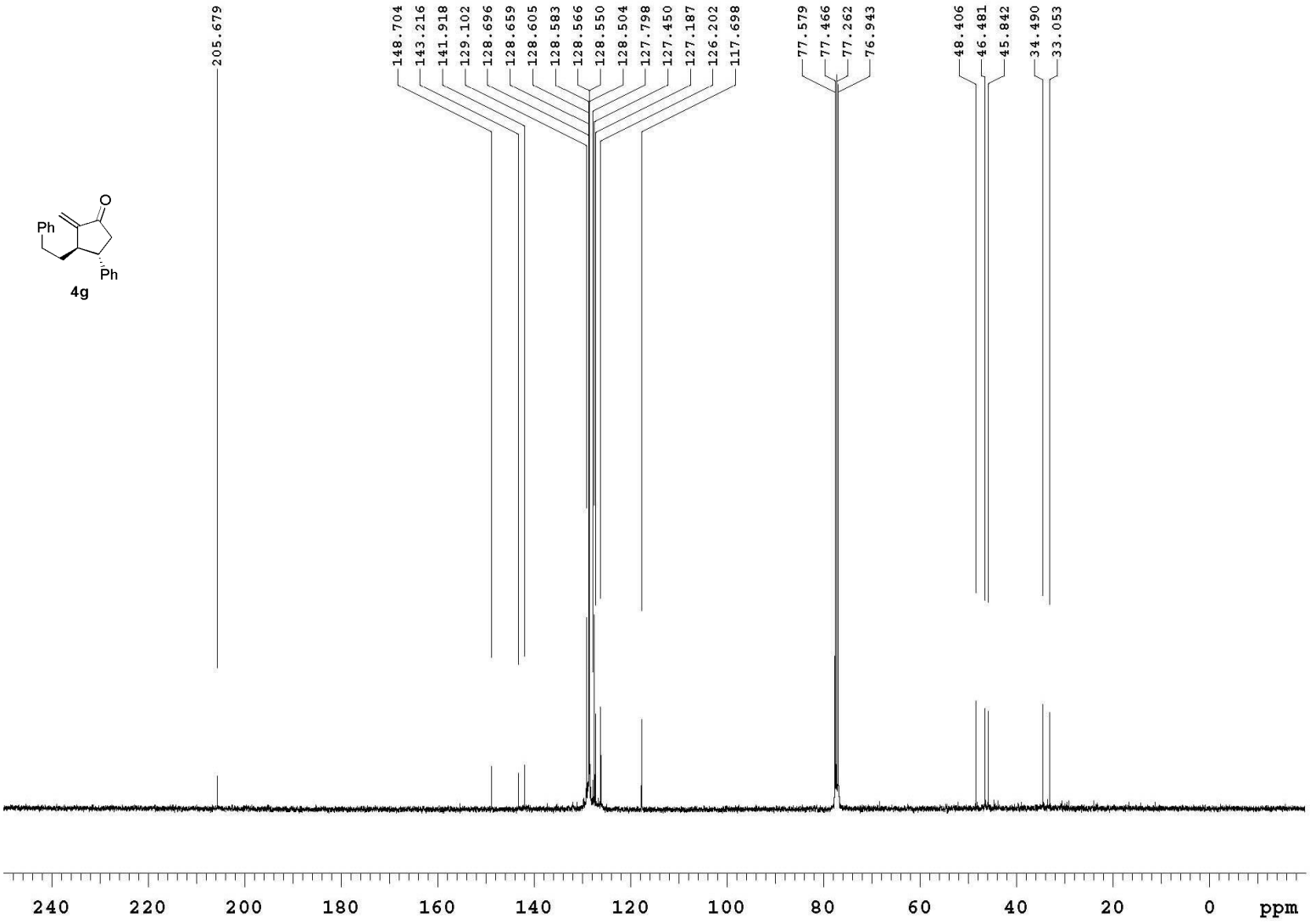

5f
O
209.139
171.581
137.334
136.768
125.105
77.378
77.262
77.060
76.742
76.581
76.548
76.540
51.306
40.133
25.386
23.863
22.814
22.529
15.238
8.198
240
220
200
180
160
140
120
100
80
60
40
20
0
ppm

Yen-Ku, YKW-2-155-F2
400.393 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe

Pulse Sequence: s2pul

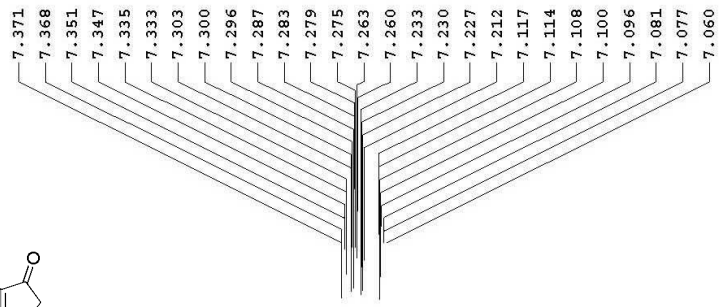


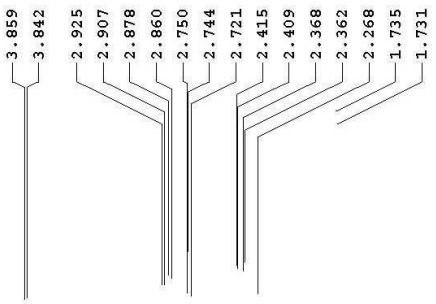
100.690 MHz C13[H1] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe
date: Aug 29 2009 sweep width: 27174Hz acq.time: 2.0s relax.time: 0.1s # scans: 6000 dig.res.: 0.2 Hz/pt hz/mm:113.2
spectrometer:d601 file:/mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/2009.08/2009.08.29.m4_YKW-2-155-F2_21:02_C13_1D.fid
Pulse sequence: s2pul

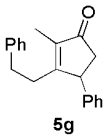


Yen-Ku, YKW-2-155-F3
400.393 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe

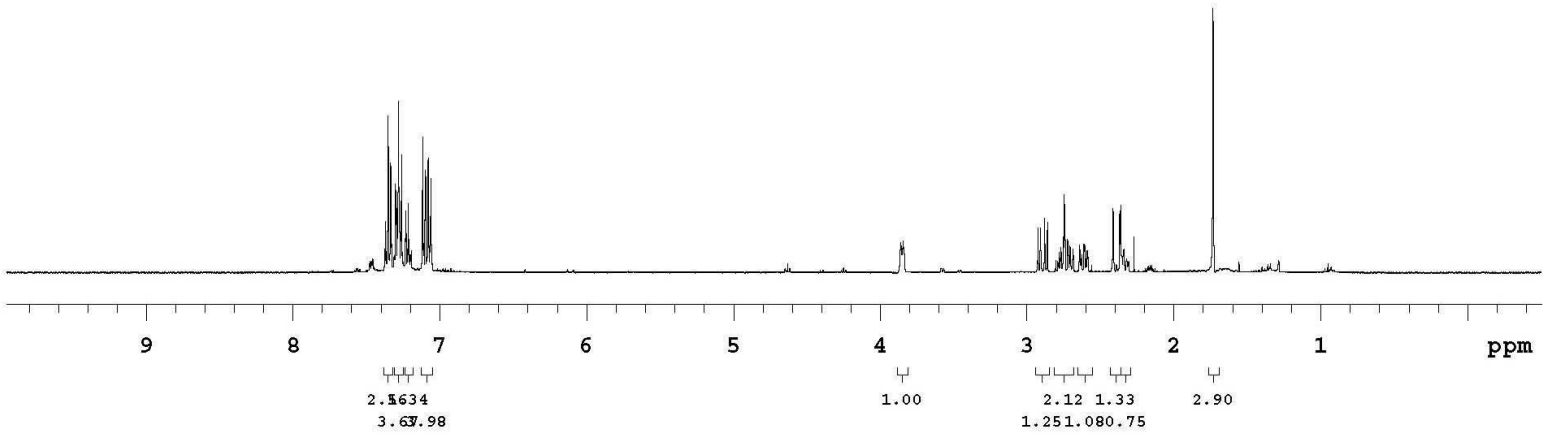
Pulse Sequence: s2pul



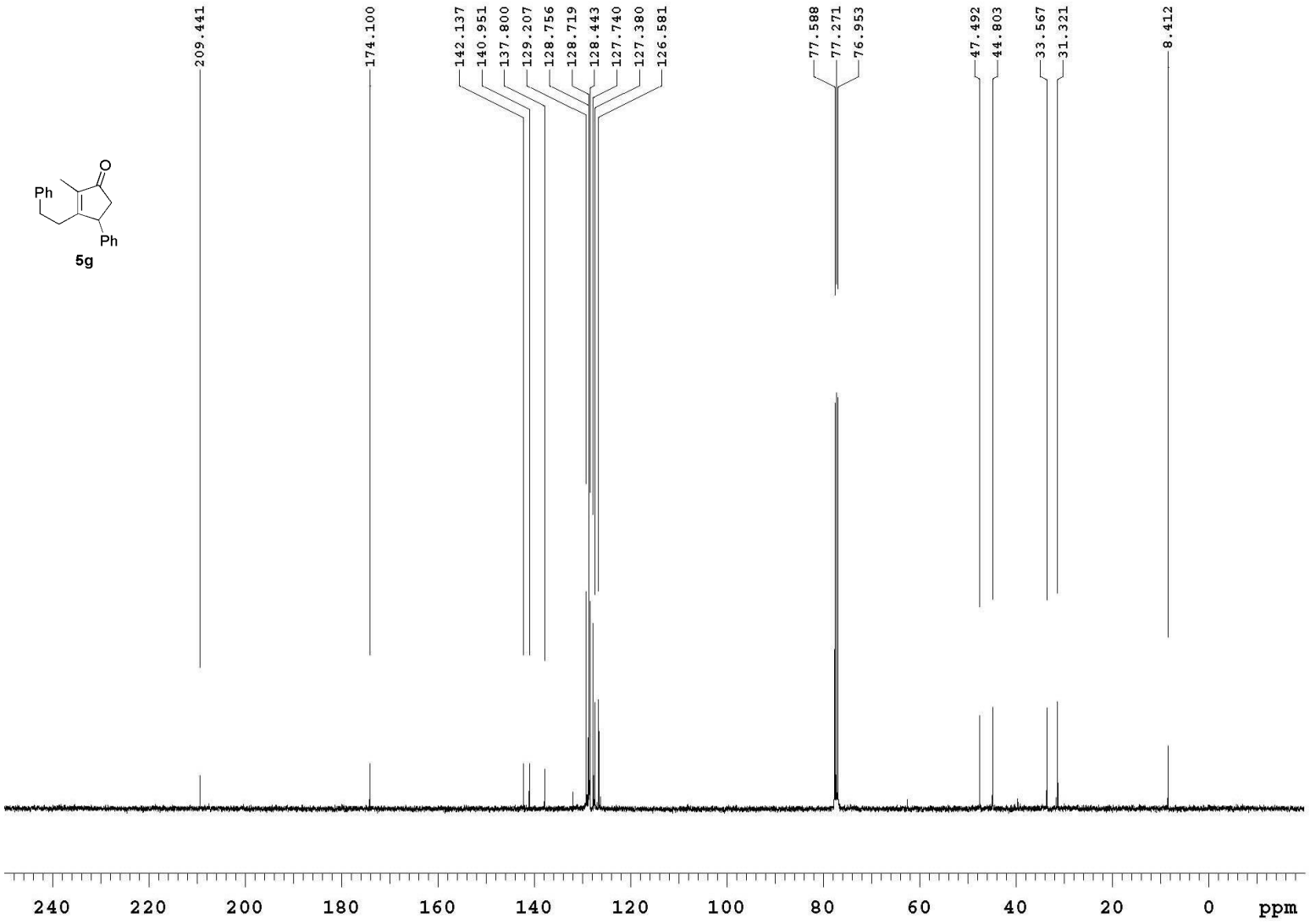




5g

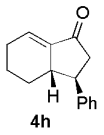


100.690 MHz C13[H1] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe
date: Aug 30 2009 sweep width: 27174Hz acq.time: 2.0s relax.time: 0.1s # scans: 6000 dig.res.: 0.2 Hz/pt hz/mm:113.2
spectrometer:d601 file:/mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/2009.08/2009.08.30.m4_YKW-2-155-F3_00:39_C13_1D.fid
Pulse Sequence: s2pul

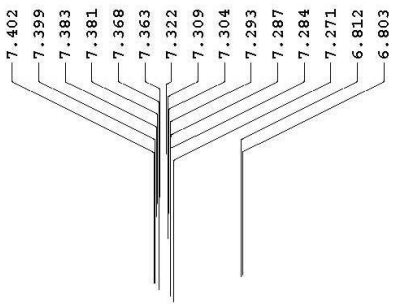


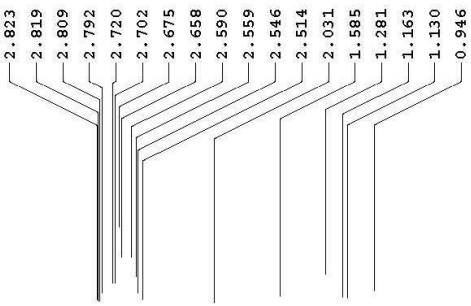
Yen-Ku, YKW-1-155-F2
400.393 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe

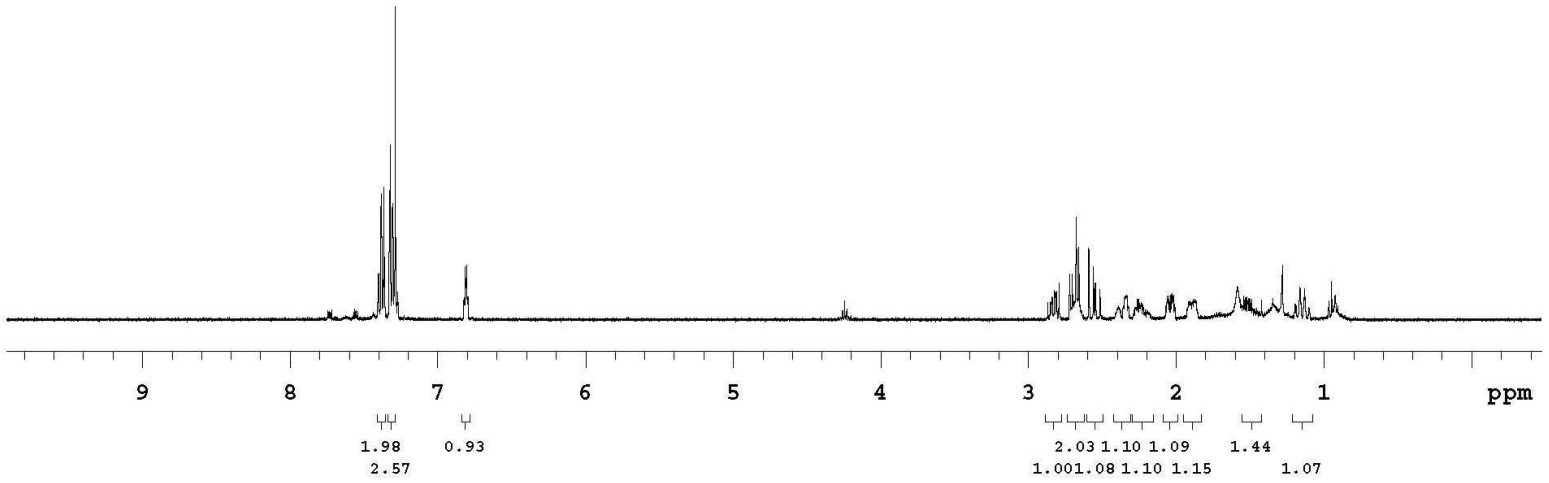
Pulse Sequence: s2pul



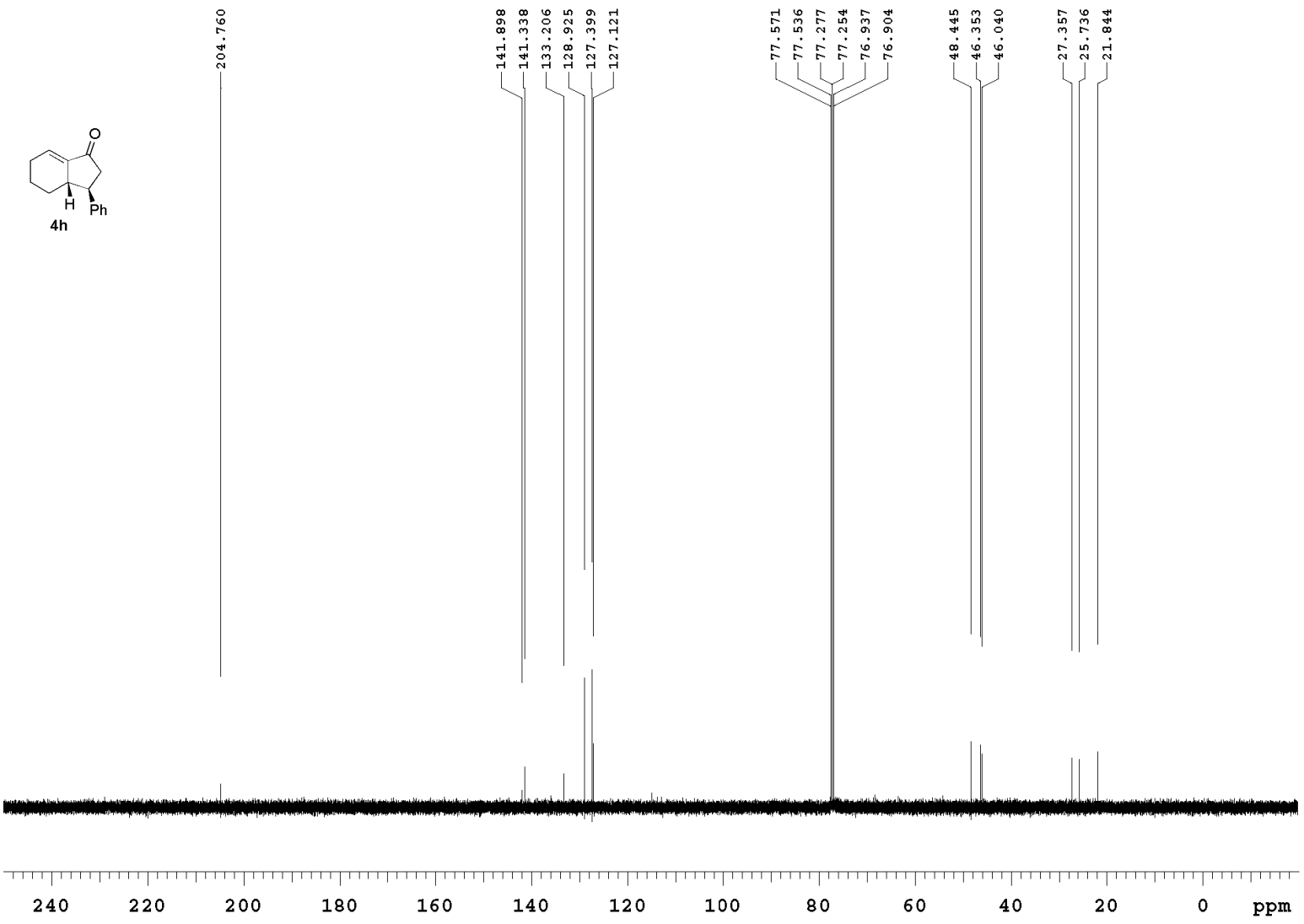
4h





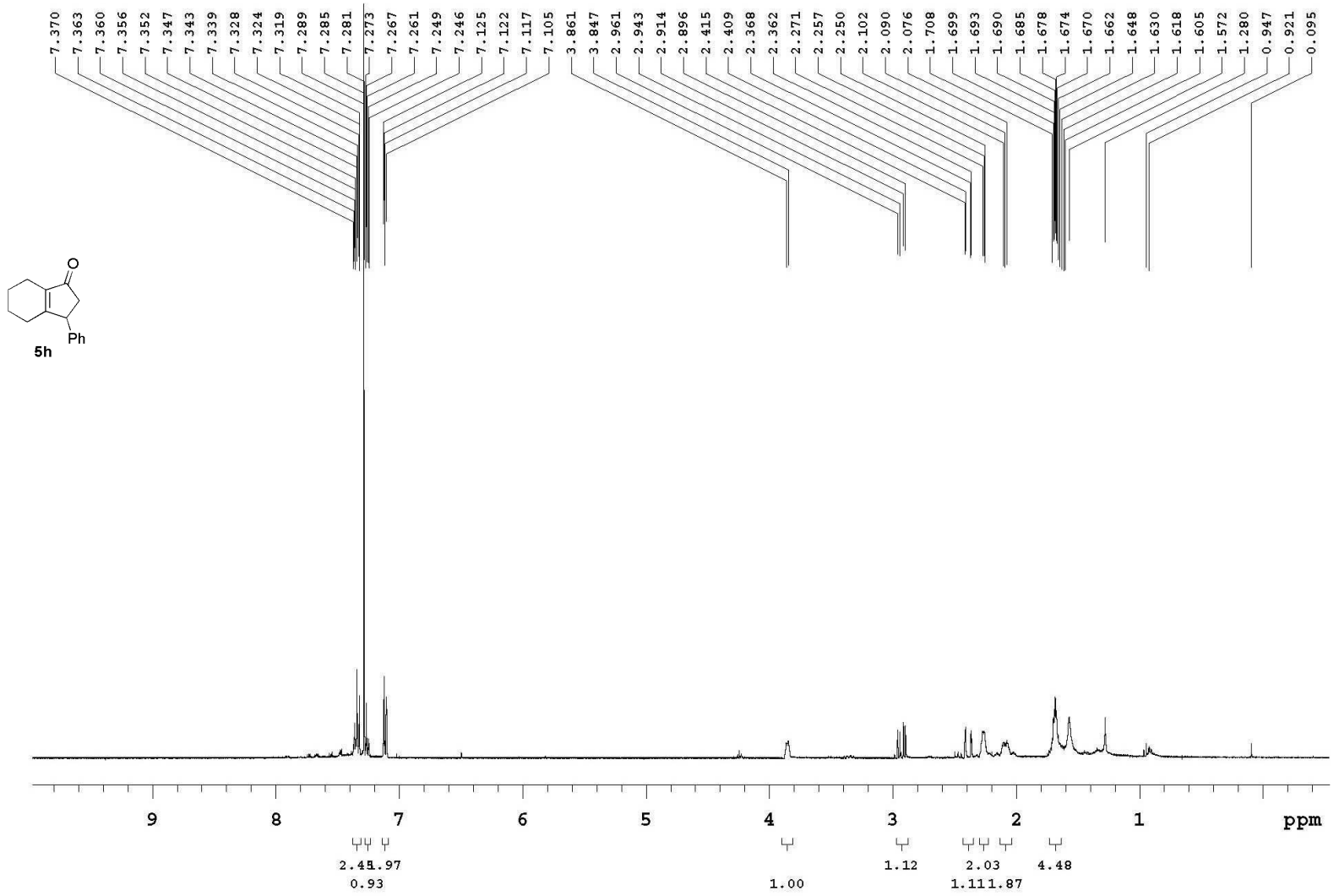


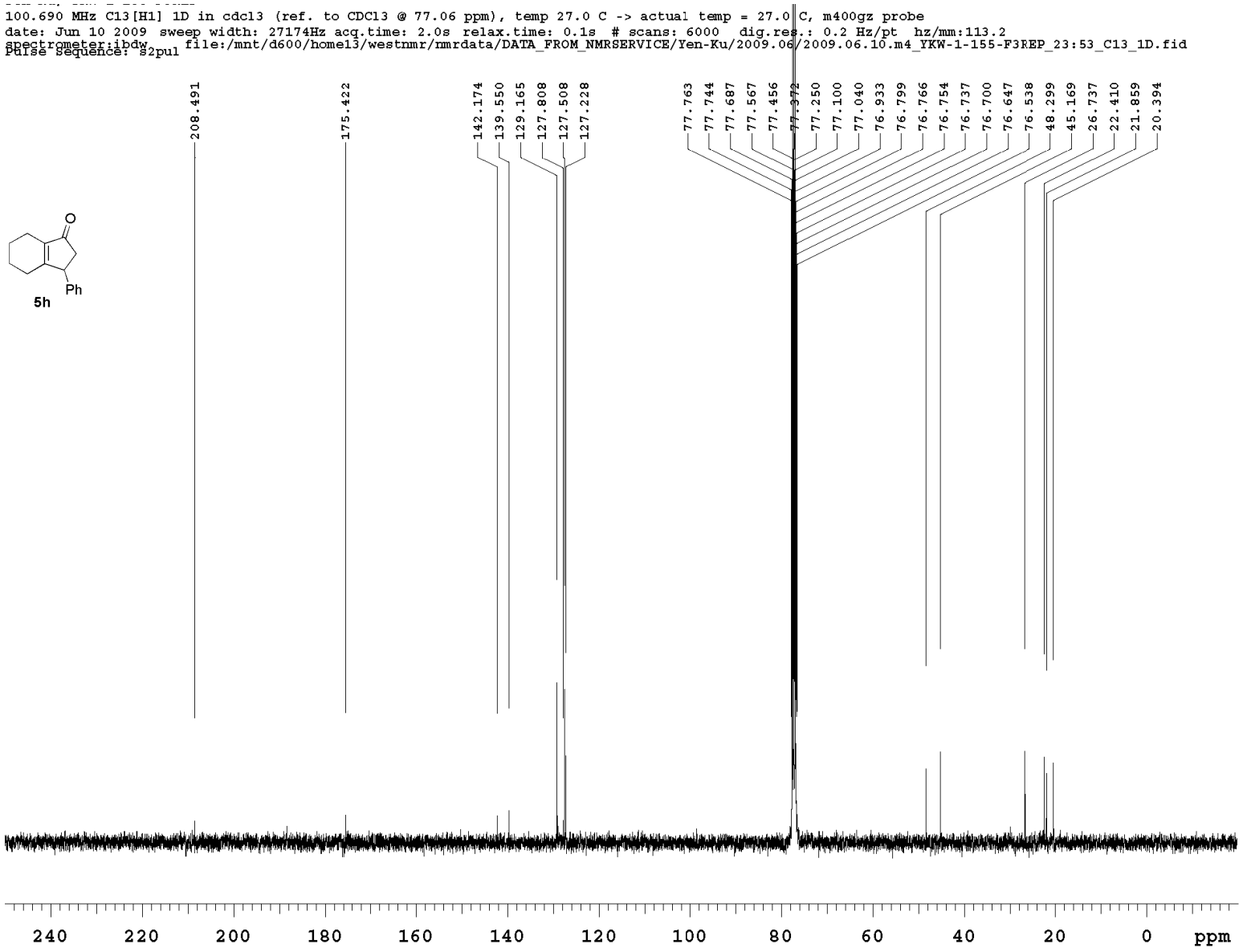
100.690 MHz C13[H1] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe
date: Jun 8 2009 sweep width: 27174Hz acq.time: 2.0s relax.time: 0.1s # scans: 512 dig.res.: 0.2 Hz/pt hz/mm:113.2
spectrometer:ibdw file:/mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/2009.06/2009.06.8.m4_YKW-1-155-F2_13:10_C13_1D.fid
Pulse Sequence: s2pul



Yen-Ku, YKW-1-155-F3reH
400.393 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe

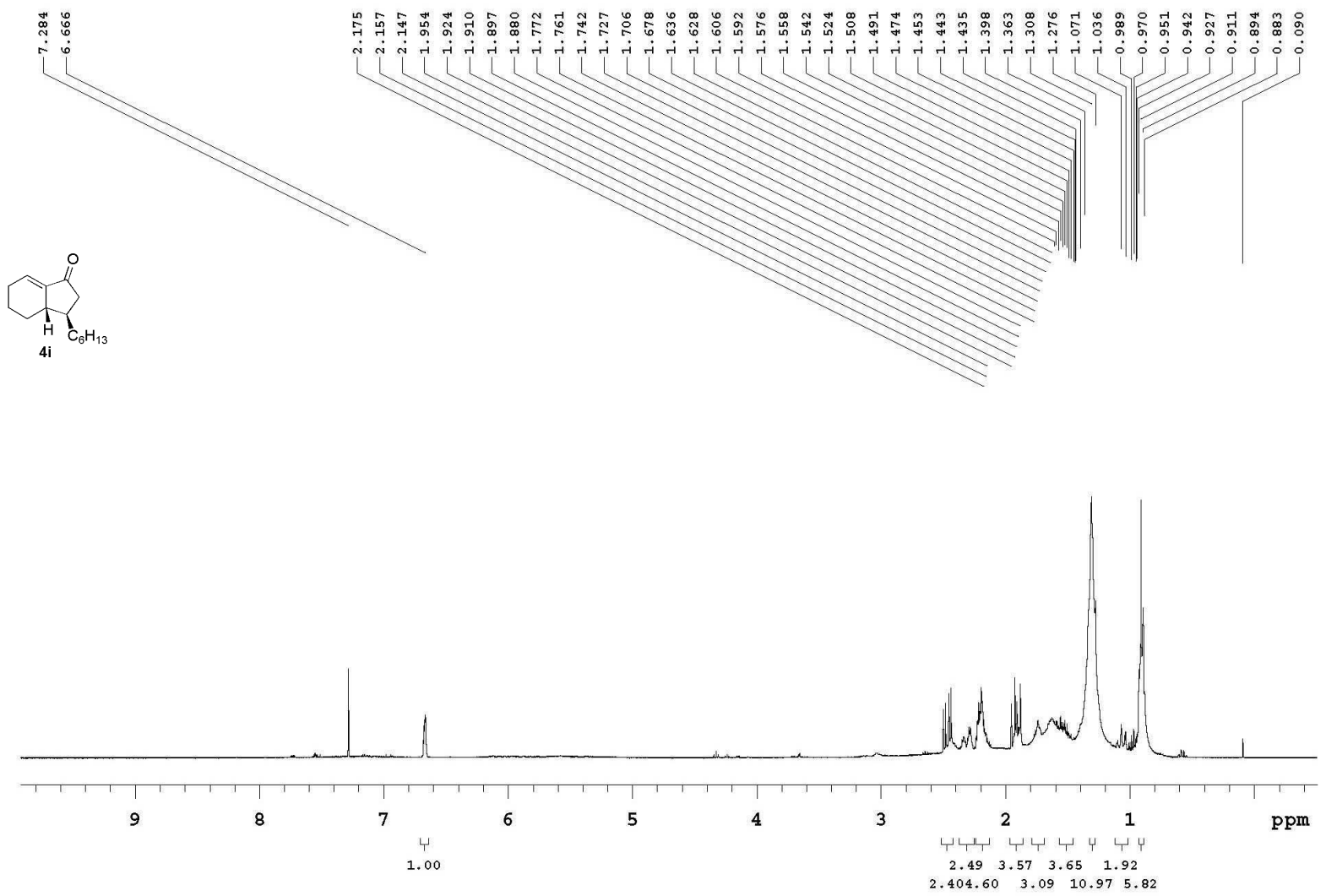
Pulse Sequence: s2pul



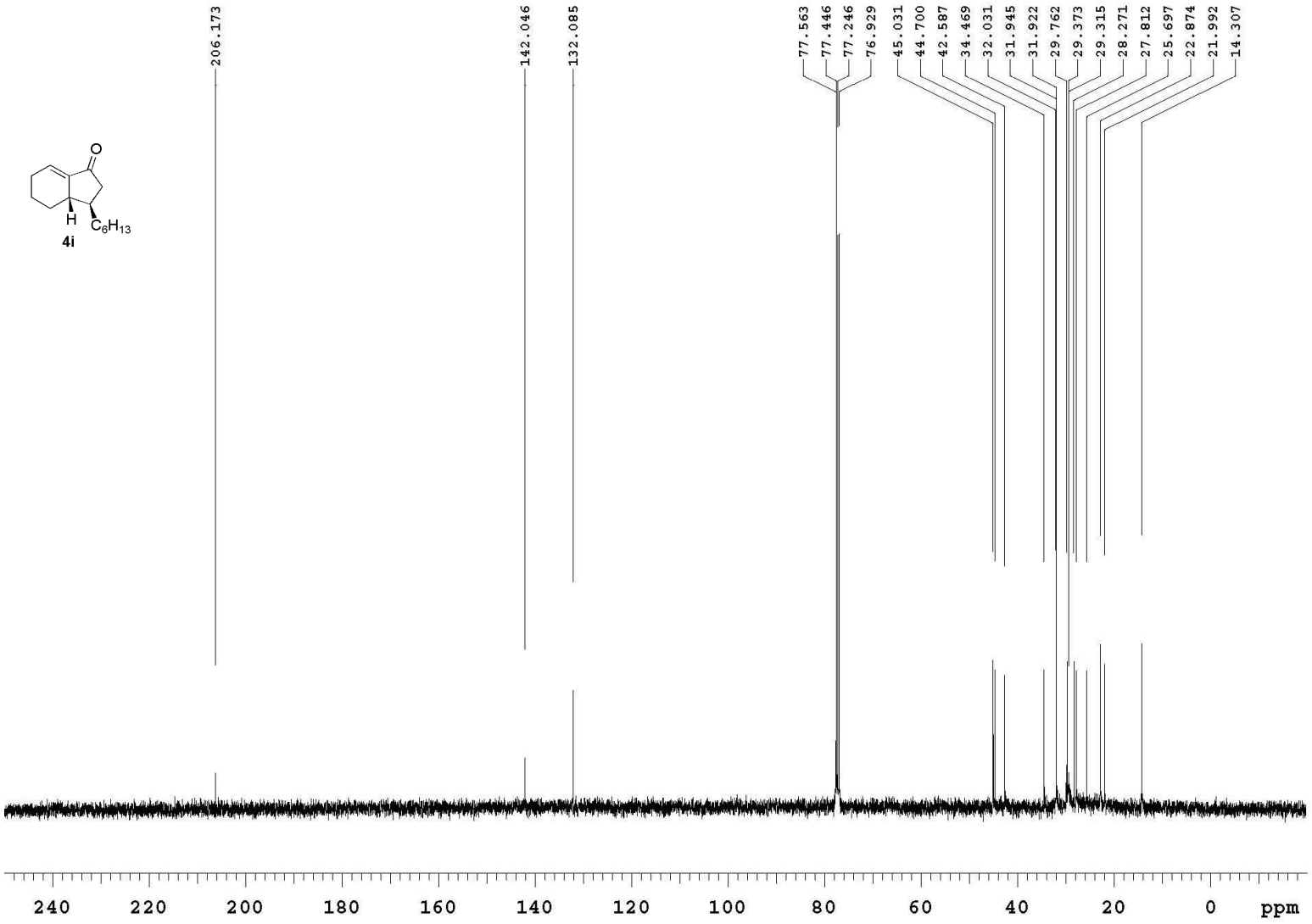

100.690 MHz C13[H1] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe
date: Jun 10 2009 sweep width: 27174Hz acq.time: 2.0s relax.time: 0.1s # scans: 6000 dig.res.: 0.2 Hz/pt hz/mm:113.2
spectrometer:ibdw file:/mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/2009.06/2009.06.10.m4_YKW-1-155-F3REP_23:53_C13_1D.fid
Pulse sequence: s2pul
208.491
175.422
142.174
139.550
129.165
127.808
127.508
127.228
77.763
77.744
77.687
77.567
77.456
77.372
77.250
77.100
77.040
76.933
76.799
76.766
76.754
76.737
76.700
76.647
76.538
48.299
45.169
26.737
22.410
21.859
20.394
O
Ph
5h
240
220
200
180
160
140
120
100
80
60
40
20
0
ppm

Yen-Ku, YKW-1-197-F2-H1
400.393 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe

Pulse Sequence: s2pul

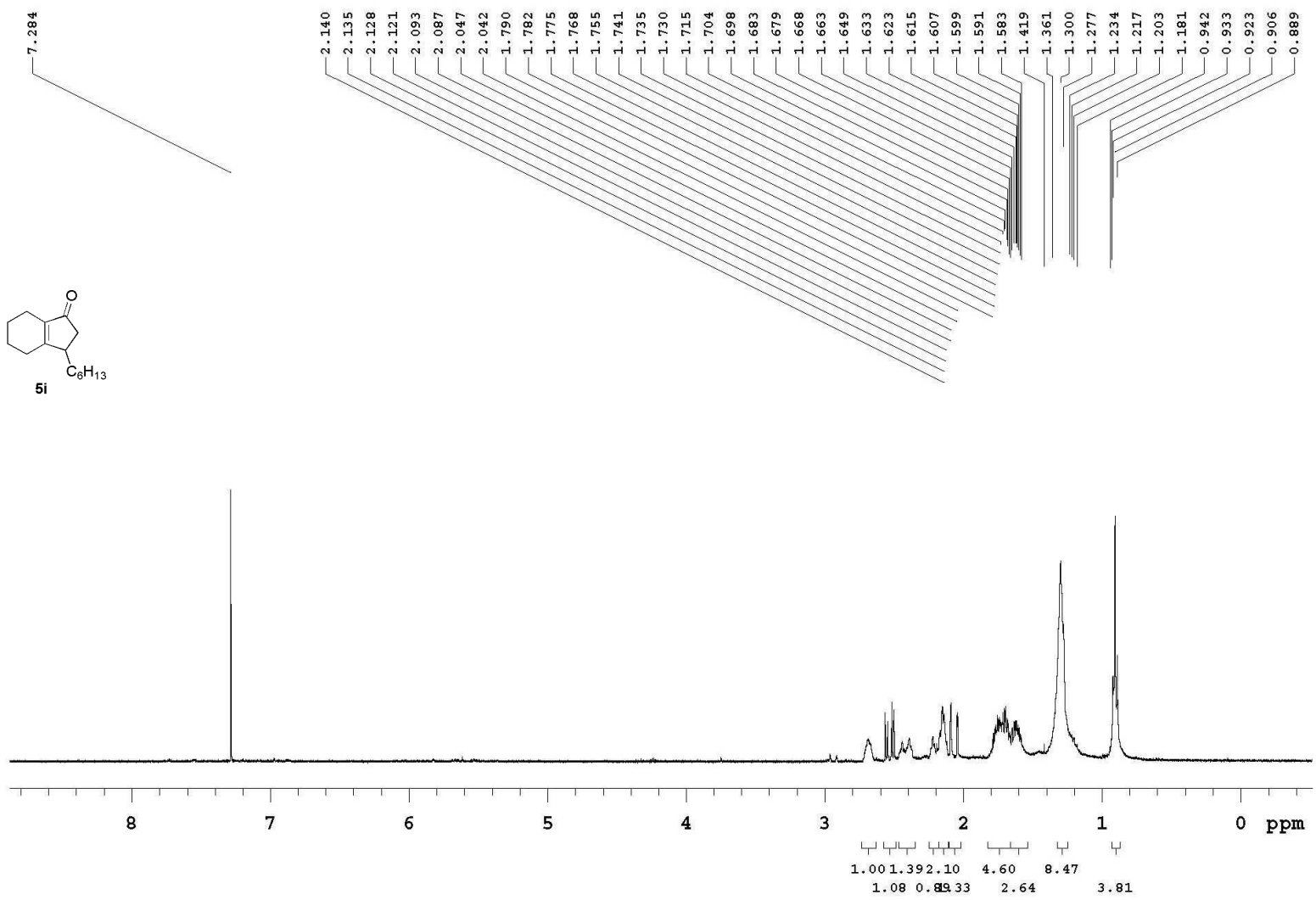


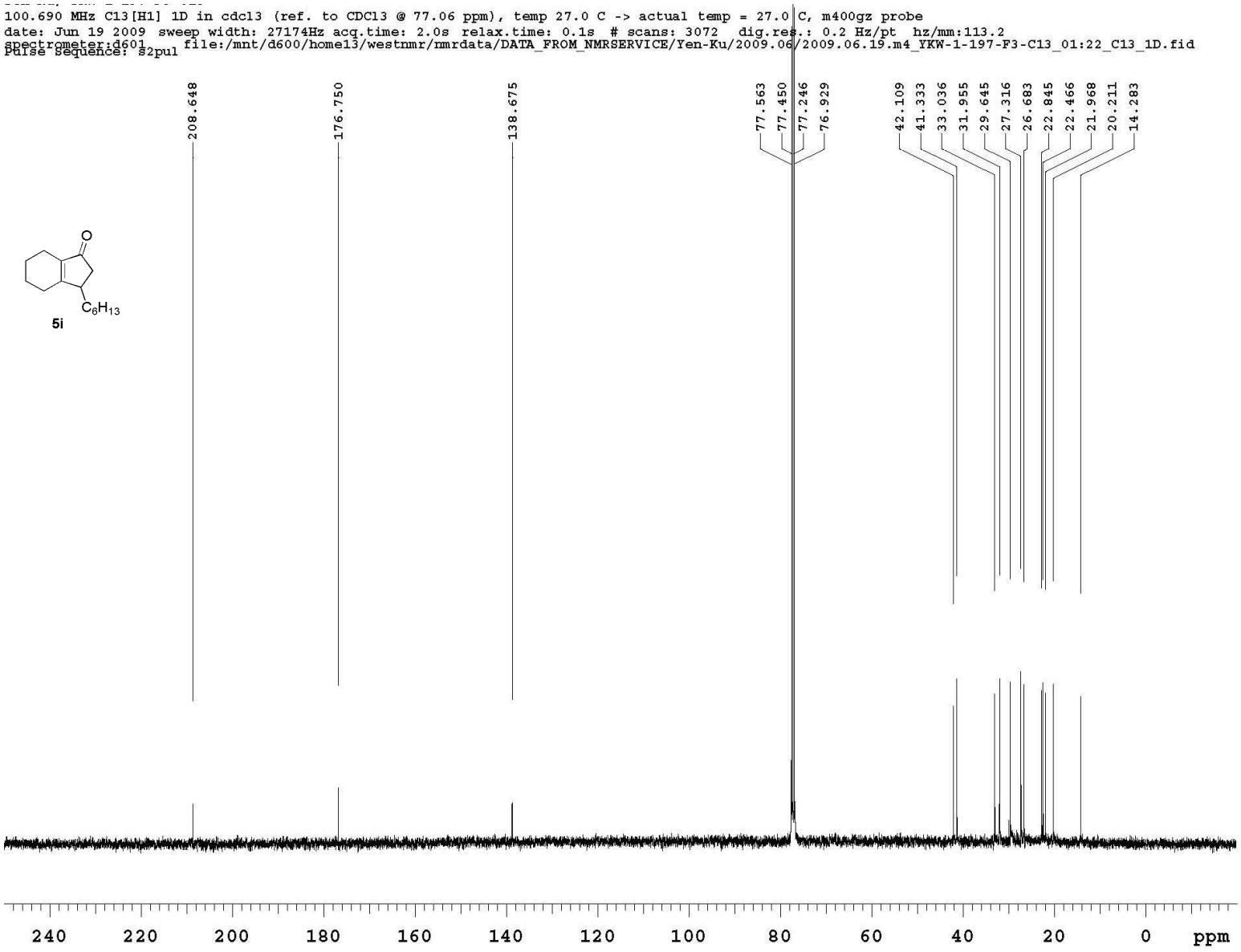
100.690 MHz C13[H1] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe
date: Jun 18 2009 sweep width: 27174Hz acq.time: 2.0s relax.time: 0.1s # scans: 1024 dig.res.: 0.2 Hz/pt hz/mm:113.2
spectrometer:d601 file:/mnt/d600/home13/westnmr/rmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/2009.06/2009.06.18.m4_YKW-1-197-F2-M4_16:00_C13_1D.fid
Pulse sequence: s2pul



Yen-Ku, YKW-1-197-F3
400.393 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe

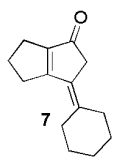
Pulse Sequence: s2pul

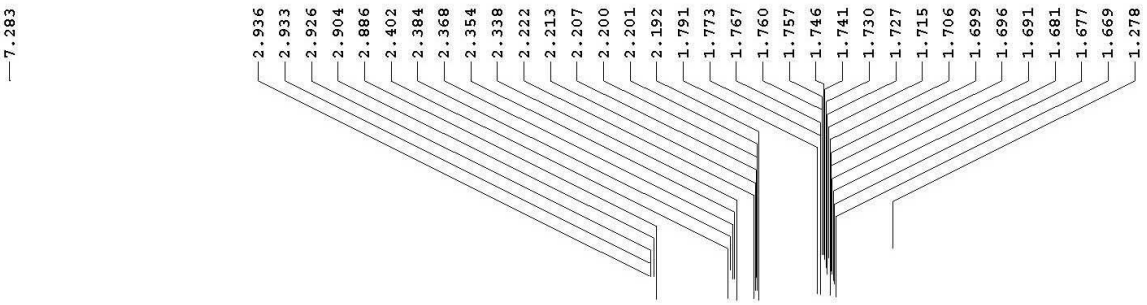


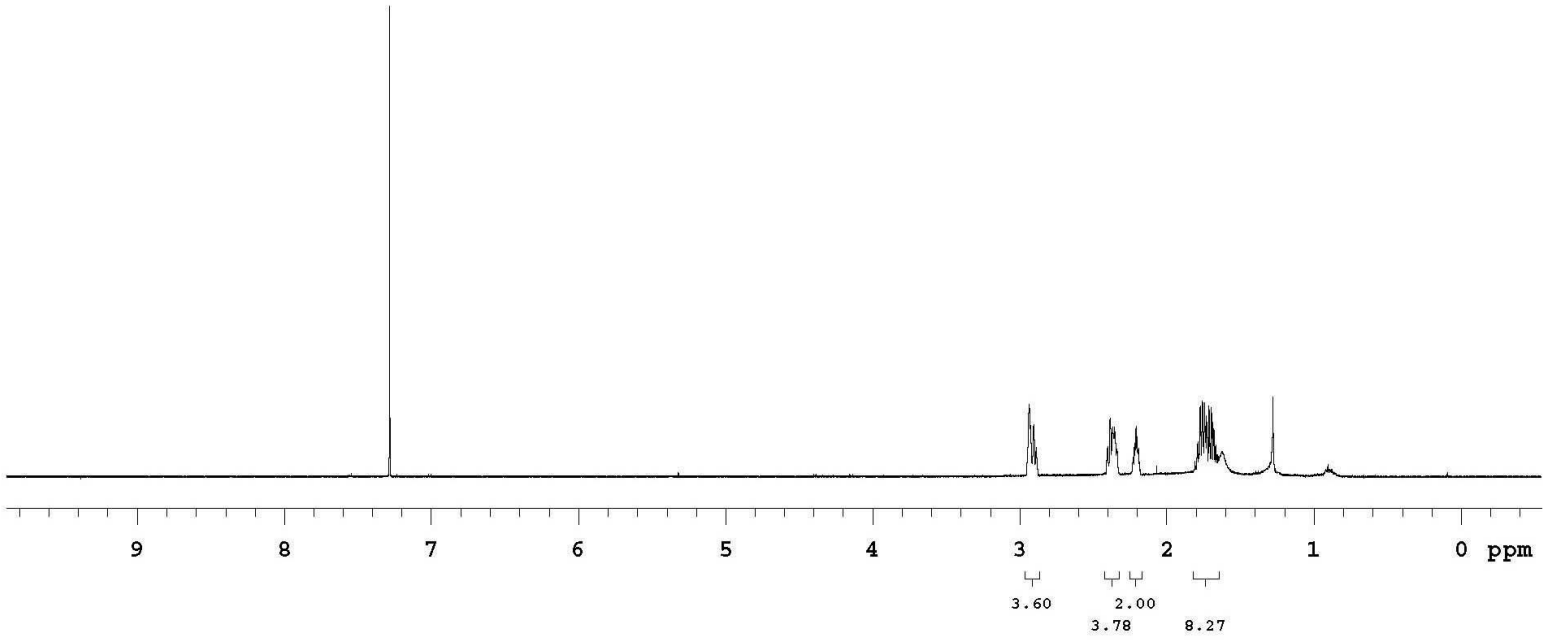

100.690 MHz C13[H1] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe
date: Jun 19 2009 sweep width: 27174Hz acq.time: 2.0s relax.time: 0.1s # scans: 3072 dig.res.: 0.2 Hz/pt hz/mm:113.2
spectrometer:d601 file:/mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/2009.06/2009.06.19.m4_YKW-1-197-F3-C13_01:22_C13_1D.fid
Pulse sequence: s2pul
208.648
176.750
138.675
77.563
77.450
77.246
76.929
42.109
41.333
33.036
31.955
29.645
27.316
26.683
22.845
22.466
21.968
20.211
14.283
O
C6H13
5i
240
220
200
180
160
140
120
100
80
60
40
20
0
ppm

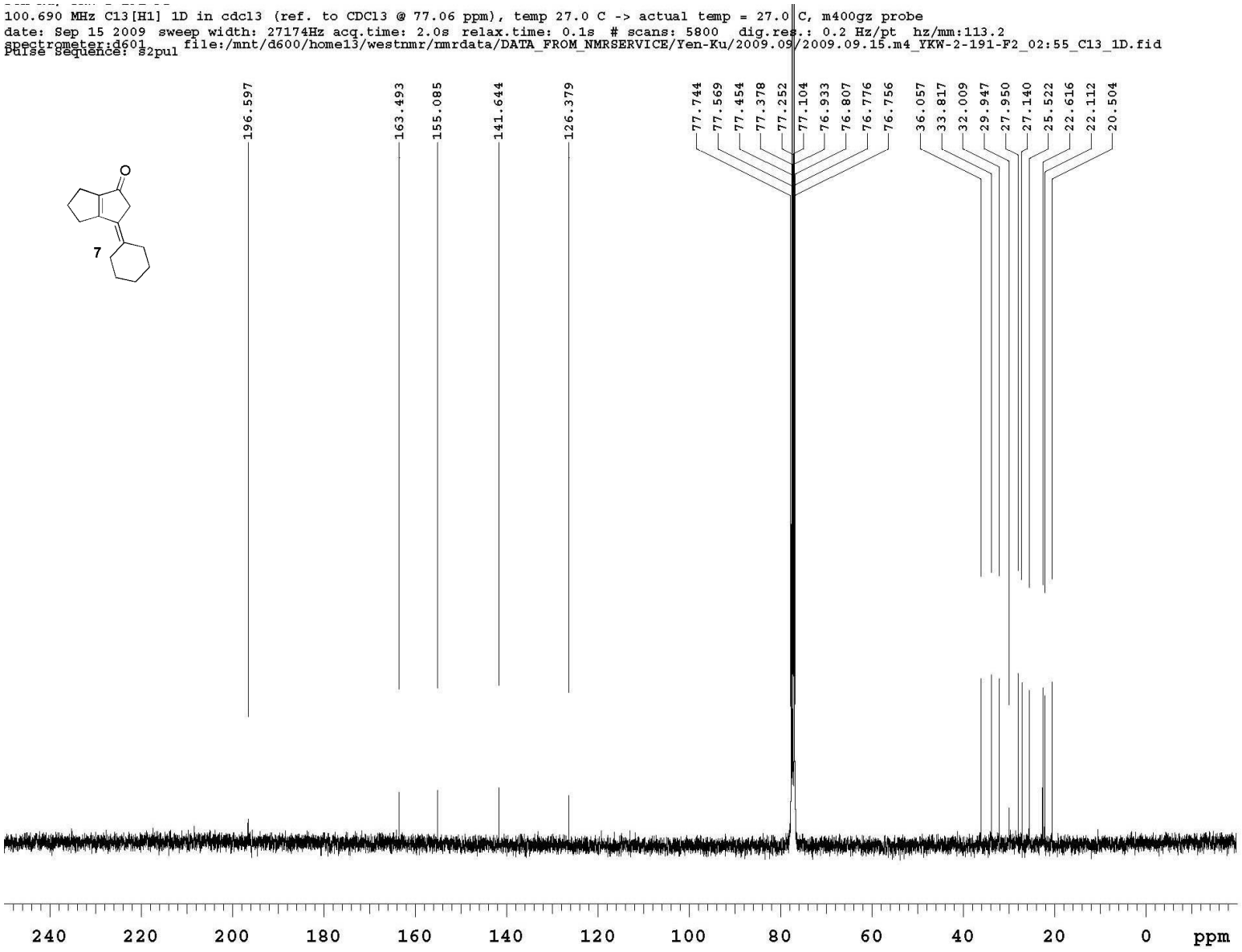
Yen-Ku, YKW-2-191-F2-proton1
400.393 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe

Pulse Sequence: s2pul



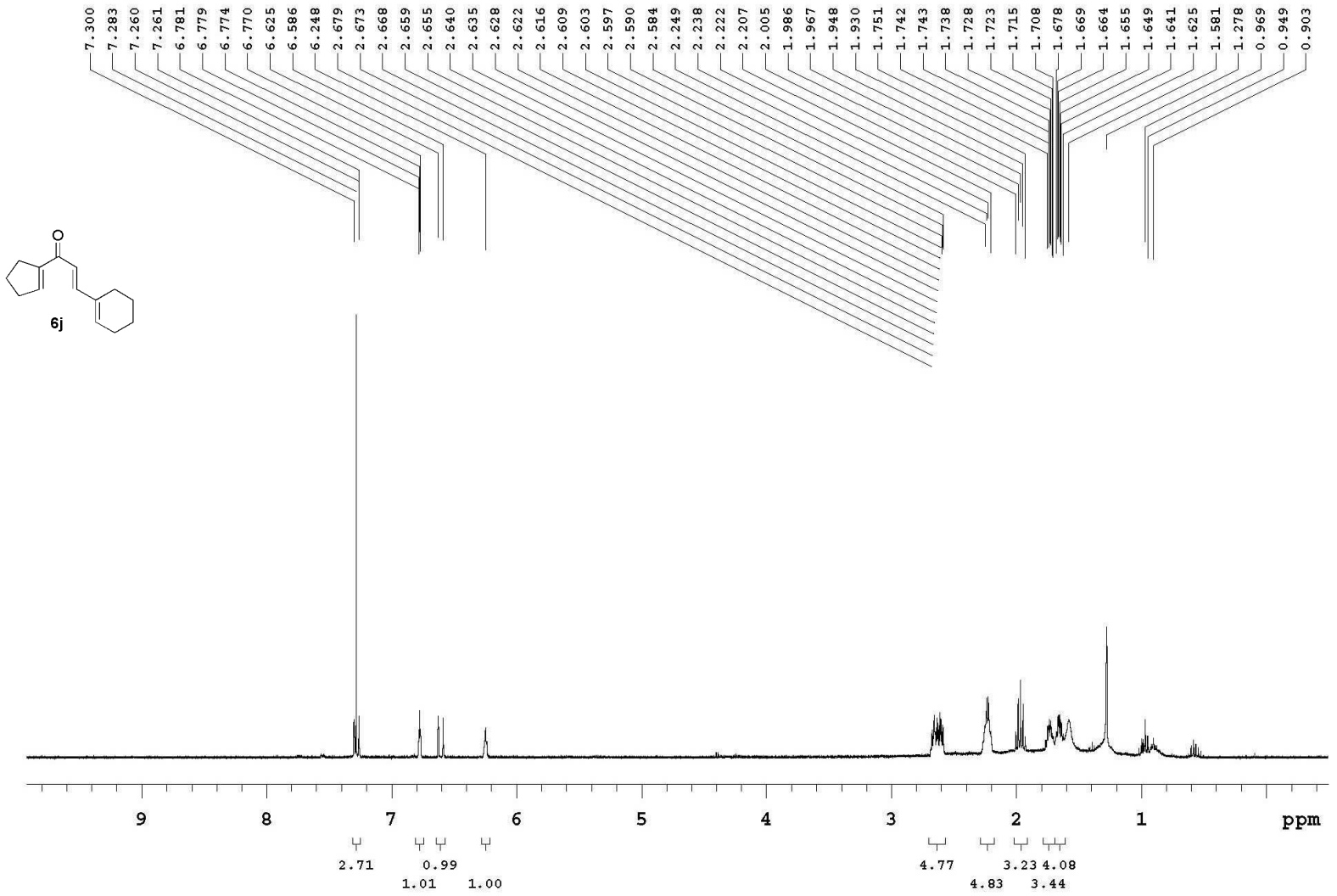




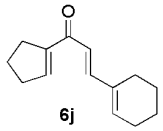

100.690 MHz C13[H1] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe
date: Sep 15 2009 sweep width: 27174Hz acq.time: 2.0s relax.time: 0.1s # scans: 5800 dig.res.: 0.2 Hz/pt hz/mm:113.2
spectrometer:d601 file:/mnt/d600/home13/westnmr/rmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/2009.09/2009.09.15.m4_YKW-2-191-F2_02:55_C13_1D.fid
Pulse sequence: s2pul
196.597
163.493
155.085
141.644
126.379
77.744
77.569
77.454
77.378
77.252
77.104
76.933
76.807
76.776
76.756
36.057
33.817
32.009
29.947
27.950
27.140
25.522
22.616
22.112
20.504
O
7
240
220
200
180
160
140
120
100
80
60
40
20
0
ppm

Yen-Ku, YKW-2-191-F1-H
400.393 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe

Pulse Sequence: s2pul



100.690 MHz C13[H1] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe
date: Sep 15 2009 sweep width: 27174Hz acq.time: 2.0s relax.time: 0.1s # scans: 6000 dig.res.: 0.2 Hz/pt hz/mm:113.2
spectrometer:d601 file:/mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/2009.09/2009.09.15.m4_YKW-2-191-F1-C_18:04_C13_1D.fid
Pulse sequence: s2pul

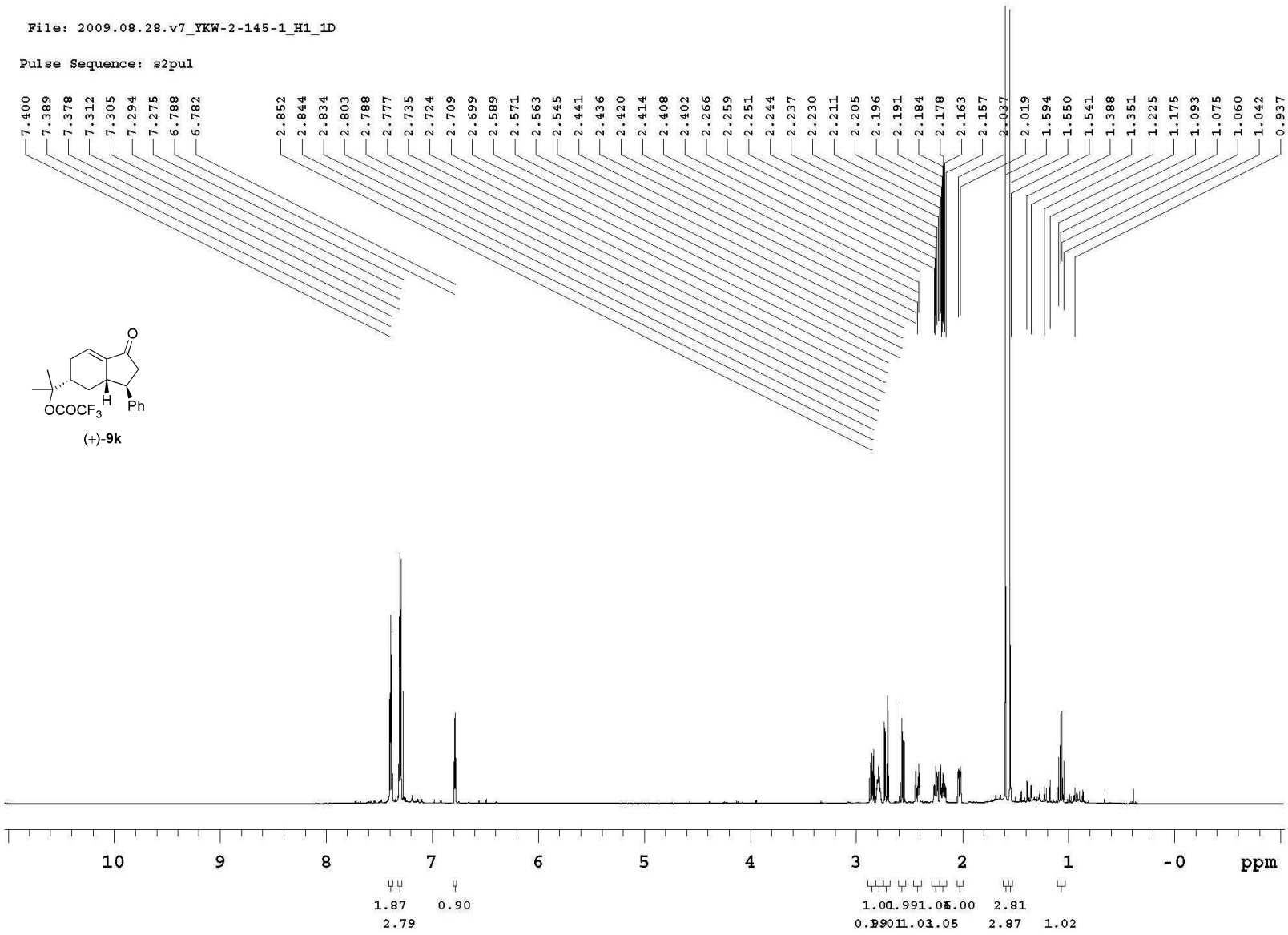


6j

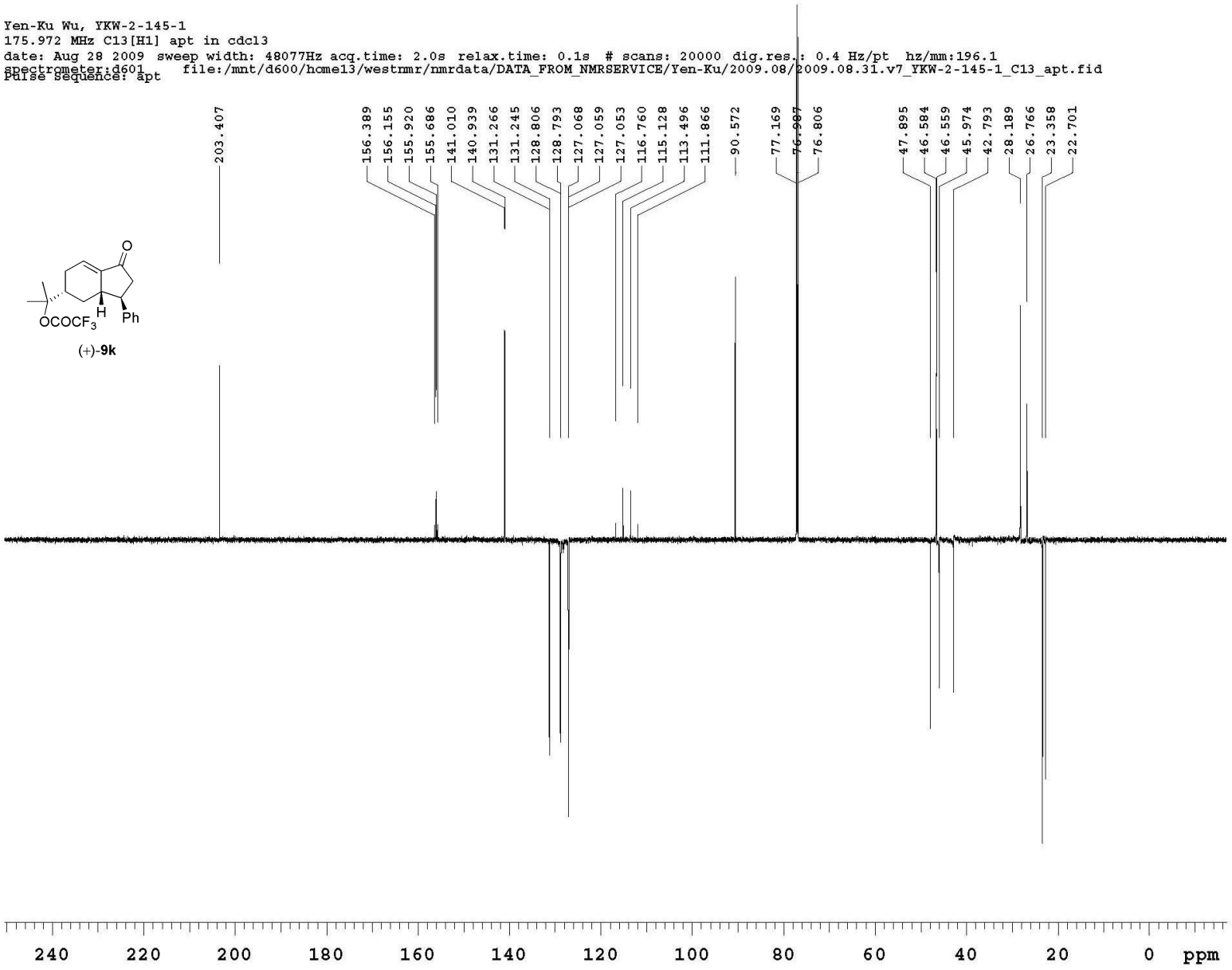
Yen-Ku Wu, YKW-2-145-1
699.752 MHz H1 1D in cdcl3

File: 2009.08.28.v7_YKW-2-145-1_H1_1D

Pulse Sequence: s2pul



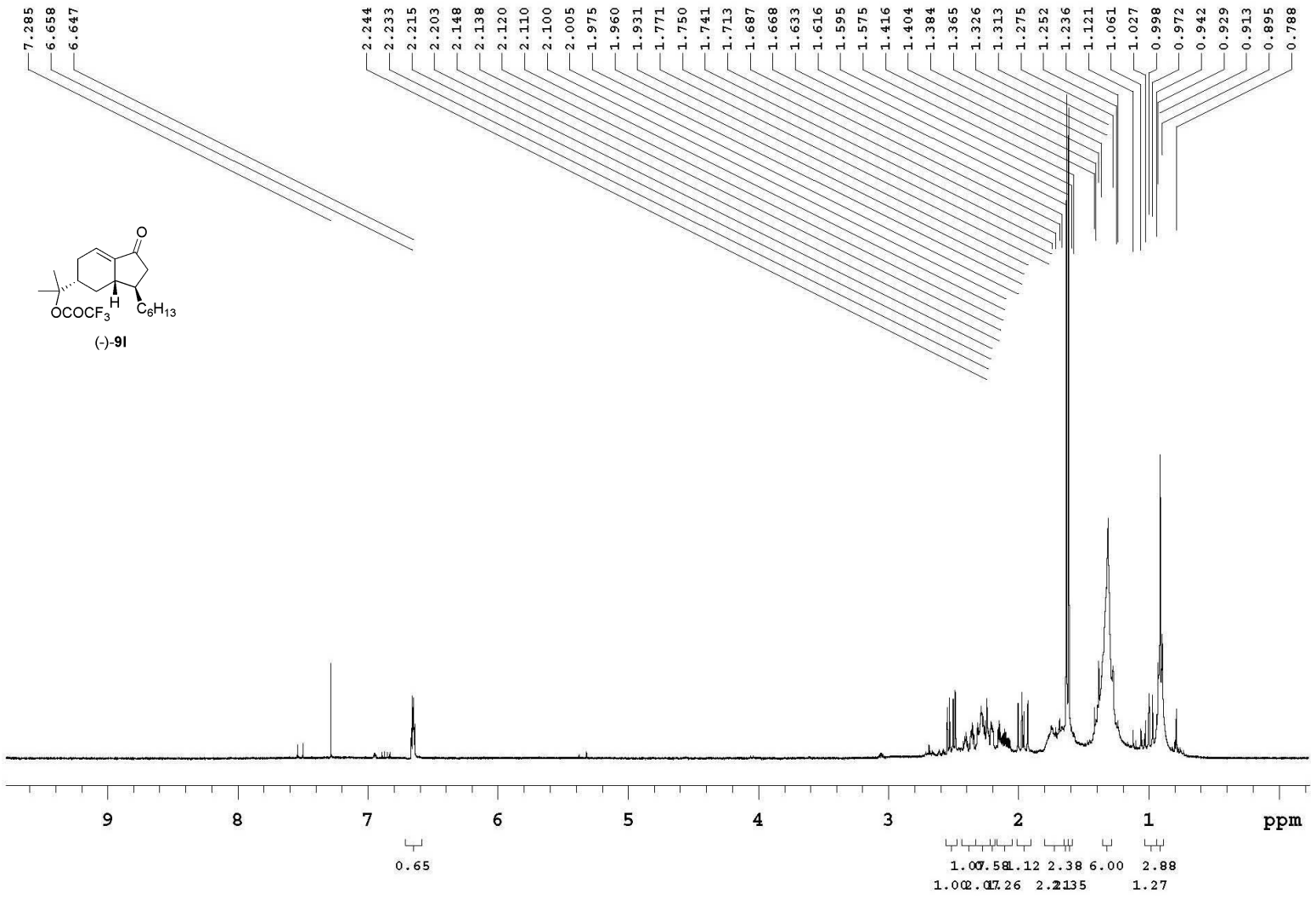
(+)-**9k**


Yen-Ku Wu, YKW-2-145-1
175.972 MHz C13[H1] apt in cdcl3
date: Aug 28 2009 sweep width: 48077Hz acq.time: 2.0s relax.time: 0.1s # scans: 20000 dig.res.: 0.4 Hz/pt hz/mm:196.1
spectrometer:d601 file:/mnt/d600/home13/westrmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/2009.08/2009.08.31.v7_YKW-2-145-1_C13_apt.fid
Pulse sequence: apt
203.407
156.389
156.155
155.920
155.686
141.010
140.939
131.266
131.245
128.806
128.793
127.068
127.059
127.053
116.760
115.128
113.496
111.866
90.572
77.169
76.987
76.806
47.895
46.584
46.559
45.974
42.793
28.189
26.766
23.358
22.701
O
H
Ph
OCOCF3
240
220
200
180
160
140
120
100
80
60
40
20
0
ppm

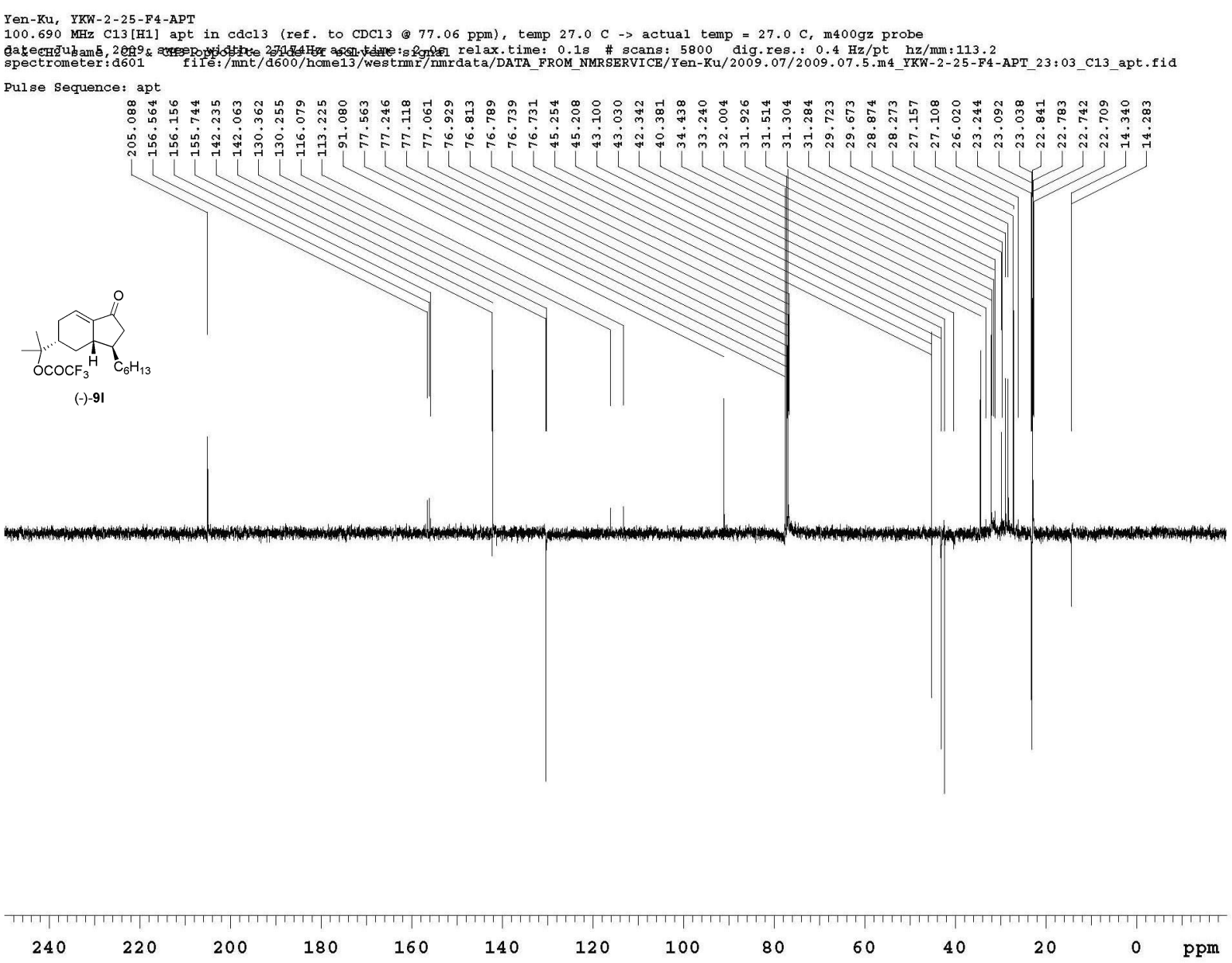
(+)-**9k**

Yen-Ku, YKW-2-25-F4
400.393 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe

Pulse Sequence: s2pul

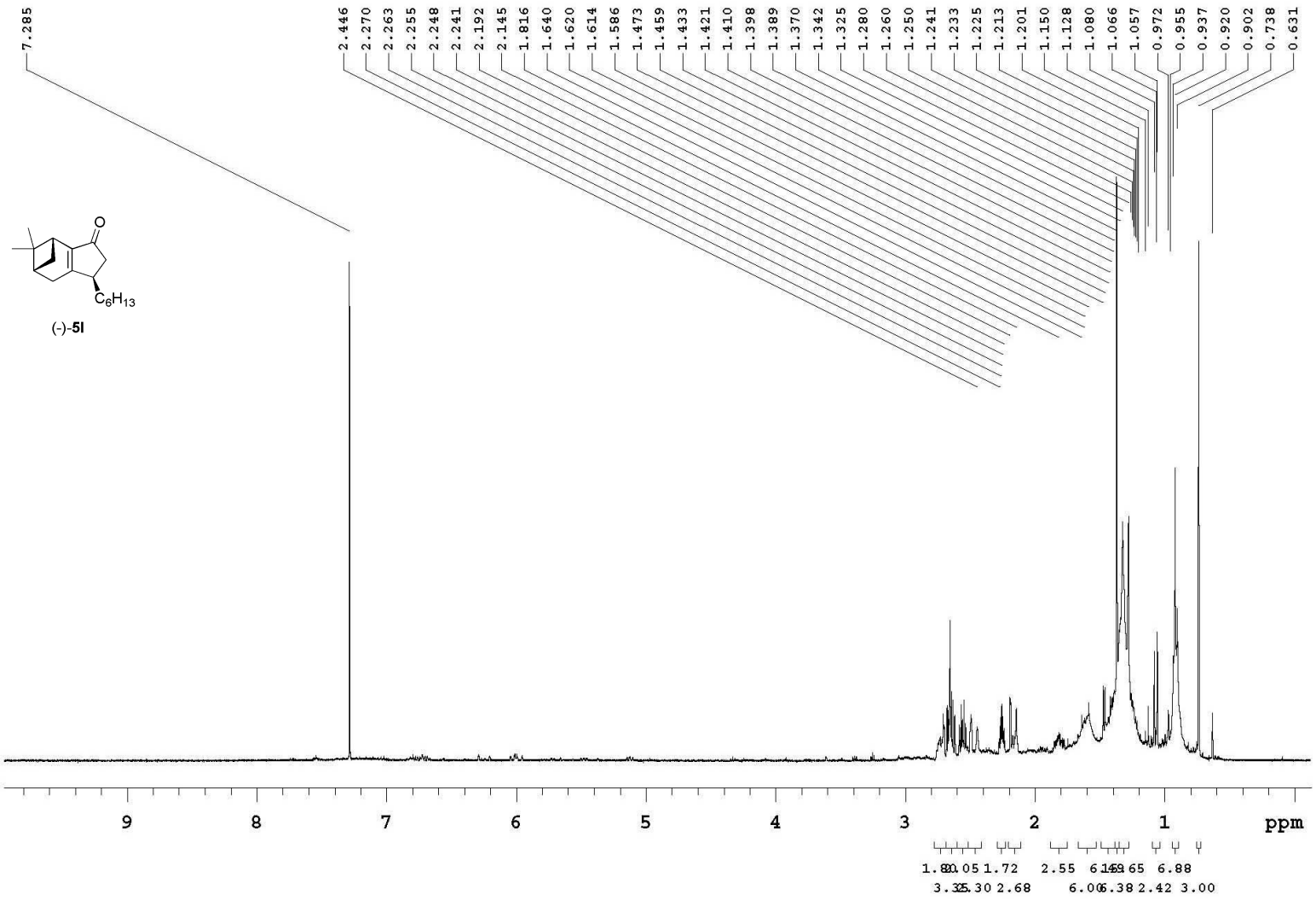


(-)-**9l**


Yen-Ku, YKW-2-25-F4-APT
100.690 MHz C13[H1] apt in cdc13 (ref. to CDC13 @ 77.06 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe
date:Jul 5 2009 sweep width: 27174Hz acq.time: 2.0s relax.time: 0.1s # scans: 5800 dig.res.: 0.4 Hz/pt hz/mm:113.2
spectrometer:d601 file:/mnt/d600/home13/westrmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/2009.07/2009.07.5.m4_YKW-2-25-F4-APT_23:03_C13_apt.fid
Pulse Sequence: apt
205.088
156.564
156.156
155.744
142.235
142.063
130.362
130.255
116.079
113.225
91.080
77.563
77.246
77.118
77.061
76.929
76.813
76.789
76.739
76.731
45.254
45.208
43.100
43.030
42.342
40.381
34.438
33.240
32.004
31.926
31.514
31.304
31.284
29.723
29.673
28.874
28.273
27.157
27.108
26.020
23.244
23.092
23.038
22.841
22.783
22.742
22.709
14.340
14.283
O
H
OCOCF3
C6H13
(-)-9l
240
220
200
180
160
140
120
100
80
60
40
20
0
ppm

Yen-Ku, YKW-2-17-F3-H1
400.393 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe

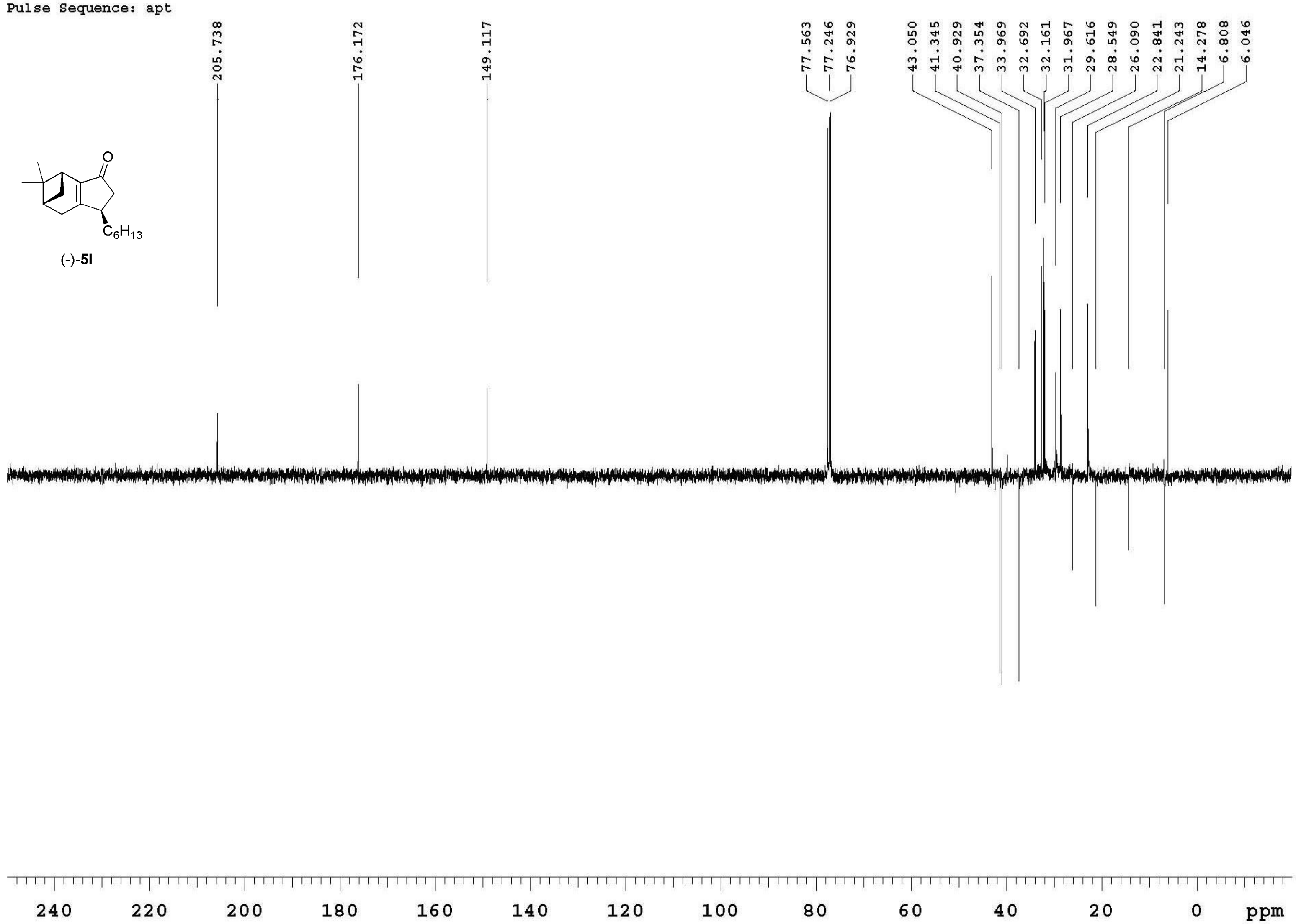
Pulse Sequence: s2pul



(-)-**5l**

Yen-Ku, YKW-2-25-F3-APT
100.690 MHz C13[H1] apt in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe
date: Jul 6 2009 sweep width: 27174Hz acq.time: 2.0s relax.time: 0.1s # scans: 5800 dig.res.: 0.4 Hz/pt hz/mm:113.2
C & CH2 same, CH & CH3 opposite side of solvent signal
spectrometer:d601 file:/mnt/d600/home13/westrmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/2009.07/2009.07.6.m4_YKW-2-25-F3-APT_02:33_C13_apt.fid

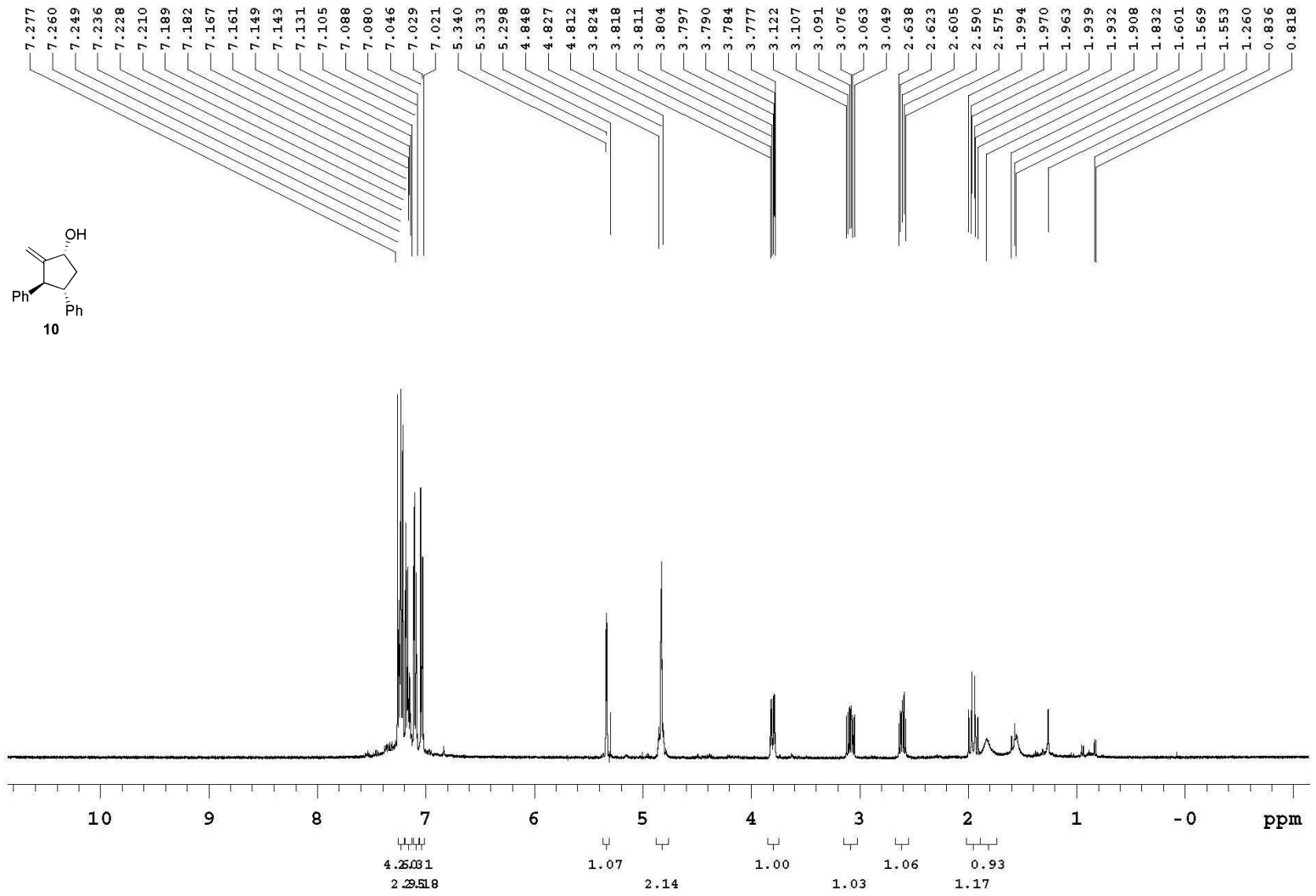
Pulse Sequence: apt



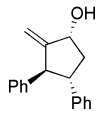
(-)-5l

YKW-1-65-exo-redu
399.794 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 26.5 C -> actual temp = 27.0 C, autoxdb probe

Pulse Sequence: s2pul



YKE-3-31-C13
100.537 MHz C13[H1] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 26.5 C -> actual temp = 27.0 C, autoxdb probe
date: Oct 13 2009 sweep width: 26991Hz acq.time: 2.5s relax.time: 0.1s # scans: 808 dig.res.: 0.2 Hz/pt hz/mm:112.5
spectrometer:d601 file:/mnt/d600/home13/westrmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/2009.10/YKW-3-31-C13.fid
Pulse Sequence: s2pul



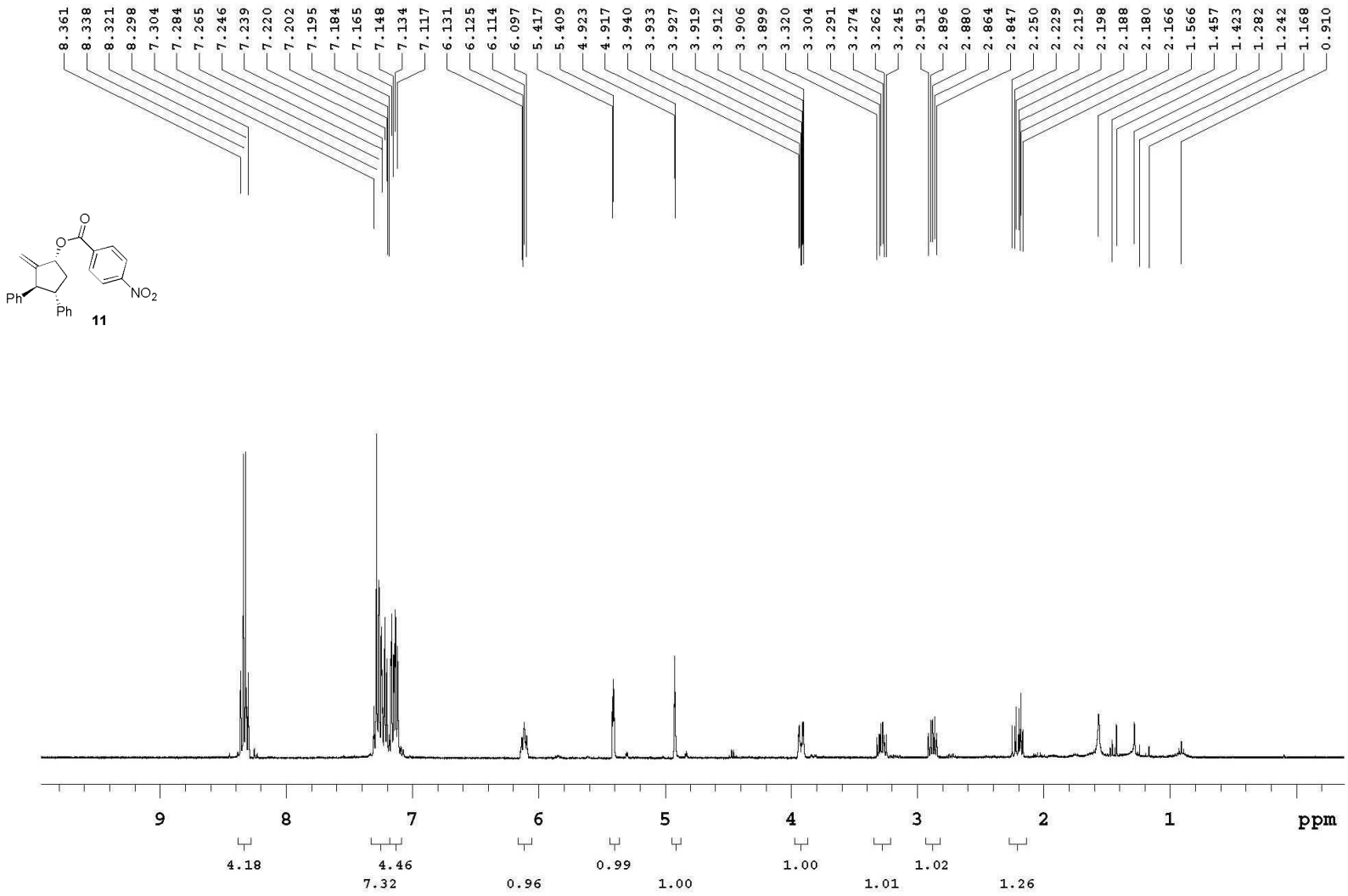
10

158.074
142.633
141.951
128.429
128.343
128.296
127.366
126.500
126.383
110.123
77.426
77.318
77.205
77.000
76.848
76.830
76.799
76.785
76.682
74.769
57.282
50.080
42.702

240 220 200 180 160 140 120 100 80 60 40 20 0 ppm

Yen-Ku, YKW-3-73-m4
400.393 MHz H1 1D in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe

Pulse Sequence: s2pul



100.690 MHz C13[H1] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.0 C -> actual temp = 27.0 C, m400gz probe
date: Oct 27 2009 sweep width: 27174Hz acq.time: 2.0s relax.time: 0.1s # scans: 5800 dig.res.: 0.2 Hz/pt hz/mm:113.2
spectrometer:d601 file:/mnt/d600/home13/westnmr/rmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/2009.10/2009.10.27.m4_YKW-3-73-m4_23:08_C13_1D.fid
Pulse sequence: s2pul