Supporting Information

For

Regioselective Formation of 1, 1-Disubstituted Allenylsilanes via Cross-Coupling Reactions of 3-Tri-*n*-butylstannyl-1-trimethylsilyl-1-propyne

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General Information

Unless otherwise noted, all reactions were performed in flame-dried or oven-dried glassware under argon atmosphere. Non-aqueous reagents were transferred using syringe techniques under argon atmosphere. Bulk grade hexanes, pentane and ethyl acetate (EtOAc) for chromatography were distilled prior to use. Tetrahydrofuran (THF), dimethylformamide (DMF), dichloromethane (DCM), acetonitrile (MeCN) and diethylether (Et_2O) were obtained anhydrous by degassing with argon and then passing through activated alumina columns to remove water and oxygen.[1] Triethylamine (Et_3N) and pyridine (C_5H_5N) were distilled from CaH_2 under argon immediately before use. Titanium isopropoxide ($Ti(O^iPr)_4$) and ethyl chloroformate (EtOCOCl) were distilled using a fractionating column at atmospheric pressure under argon immediately before use.

Reactions were monitored by standard thin-layer chromatography (TLC) techniques using EMD silica gel 60 F_{254} pre-coated plates (0.25 mm thickness). Developed TLC plates were visualized under UV light and/or by appropriate stains (*p*-anisaldehyde or cerric ammonium nitrate or potassium permanganate). Preparative TLC separations were performed using Merck silica gel 60 F_{254} pre-coated plates (0.50 mm thick). Flash column chromatography was performed with Silica-P Flash Silica Gel (ultra-pure 40-63 μm) from Silicycle Chemical Division (Quebec QC, Canada).

Proton nuclear magnetic resonance (^{1}H NMR) spectra were recorded on Varian VXR 400 (400 MHz), Varian INOVA 400 (400 MHz) or Varian Gemini 2000 (300 MHz) instruments. Carbon nuclear magnetic resonance (^{13}C NMR) spectra were measured using Varian VXR 400 (101 MHz), Varian INOVA 400 (101 MHz) or Varian Gemini 2000 (75.5 MHz) instruments.

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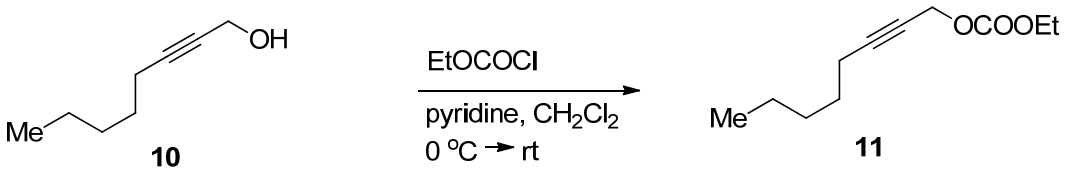
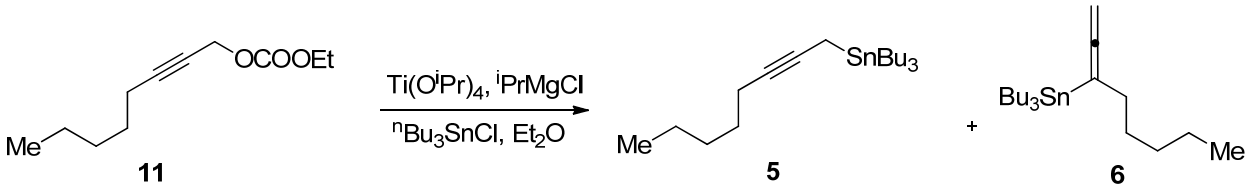
[1] Pangborn, A. B.; Giardellow, M. A.; Grubbs, R. H.; Rosen, R. K.; Timmers, F. J. *Organometallics*, 1996, **15**, 1518-1520.

NMR coupling constants and signal patterns are reported as *J* values in Hz and δ values in parts per million (ppm). ^{1}H NMR Chemical shifts (δ) are reported in ppm relative to $CDCl_3$ (δ 7.26) or acetone-*d6* (δ 2.05) or benzene-*d6* (δ 7.16). ^{13}C NMR Chemical shifts (δ) are reported in ppm relative to $CDCl_3$ (δ 77.23) or acetone-*d6* (δ 29.84) or benzene-*d6* (δ 128.06). The following abbreviations were used to indicate the multiplicities: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet. High resolution mass measurements (HRMS) were obtained on Kratos MS-80 RFA mass spectrometer by use of chemical ionization (CI) or electron impact (EI).

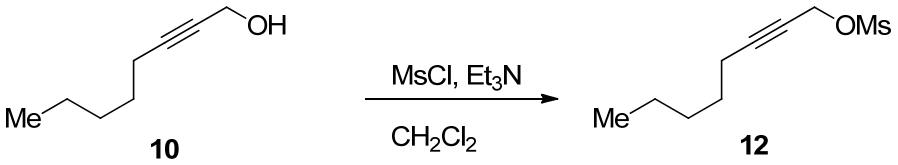
Experimental Procedures:



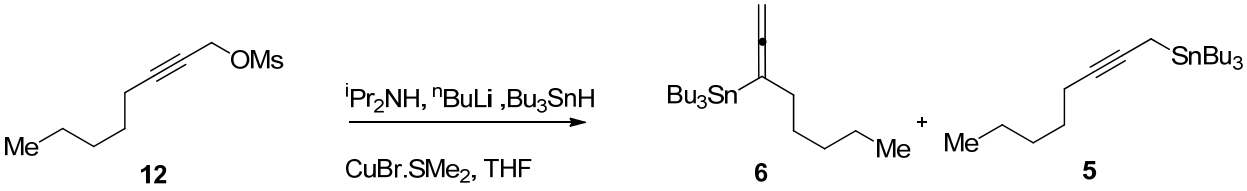
Ethyl oct-2-ynyl carbonate (11): To a solution of propargylic alcohol **10** (3.00 g, 23.77 mmol) and pyridine (5.78 mL) in dichloromethane (60 mL) was added ethyl chloroformate (2.94 mL, 3.35 g, 30.90 mmol) dropwise at 0 °C. After stirring for 1 h at 0 °C, the reaction was warmed to rt and quenched with saturated aqueous NH_4Cl and extracted with dichloromethane (2 × 50 mL). The combined organic layers were washed with water, dried over anhydrous Na_2SO_4 and evaporated *in vacuo*. The residue was purified by flash column chromatography on silica gel (100% pentane) to give **11** (4.50 g, 96% yield) as a clear oil. R_f = 0.13 (SiO_2, 100% pentane); IR (film) ν_{max} 2934, 2860, 2236, 1752, 1379, 1255, 1151, 996 cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 4.67(2H, s), 4.17(2H, q, *J* = 6.7 Hz), 2.16(2H, t, *J* = 5.8 Hz), 1.47(2H, t, *J* = 6.8 Hz), 1.29-1.25(7H, m), 0.85(3H, t, *J* = 6.4 Hz); ^{13}C NMR ($CDCl_3$, 75.5 MHz) δ 154.9, 88.7, 73.6, 64.5, 56.3, 31.2, 28.2, 22.4, 18.9, 14.4, 14.1; HRMS *m/e* calcd. for $C_{11}H_{18}O_3$ $(M+H)^+$ 199.1329 found 199.1324.



1-Tri-n-butylstannyl-2-octyne (5): To a solution of **11** (250 mg, 1.26 mmol) and $Ti(O^iPr)_4$ (0.56 mL, 1.89 mmol) in diethyl ether (4.2mL) was added ^{i}PrMgCl (2.25mL, 3.78 mmol) dropwise at -78 °C. The resulting yellow solution was stirred for 2 h at -50 °C to -40 °C. During this period, the solution turned to yellow-brown in color. $^n Bu_3SnCl$ (0.27 mL, 1.01mmol) was then added to the mixture at -78 °C. The solution was warmed to -30 °C over 90 min. and was quenched with saturated aqueous $NaHCO_3$. The solid residue was filtered out and filtrate was diluted with hexanes. To the combined organic layer was added saturated KF (2 × 20 mL) and the mixture was stirred for 10 min. The organic layers were then separated, dried over anhydrous Na_2SO_4 and evaporated *in vacuo.* Flash silica gel Column chromatography (100 % hexanes containing 1% Et_3N) was performed to obtain 1-tri-n-butylstannyl-2-octyne (**5**) and tributyl(octa-1,2-dien-3-yl)stannane (**6**) in a 95:5 ratio (322.5 mg, 64% yield) as a clear oil. R_f = 0.55 (SiO_2, 100% hexanes); IR (film) ν_{max} 2956, 2854, 2216, 1463, 1377, 1072 cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.17-2.11(2H, m), 1.61-1.27(23H, m), 0.95(6H, t, *J* = 8.0 Hz), 0.89(9H, t, *J* = 7.4 Hz); ^{13}C NMR ($CDCl_3$, 101 MHz) δ 80.2, 78.1, 31.4, 29.5, 29.2, 27.5, 22.5, 19.4, 14.3, 13.9, 9.9, -4.1; HRMS *m/e* calcd. for $C_{16}H_{31}Sn$ $(M\text{-}C_4H_9)^+$ 343.1442 found 343.1455.

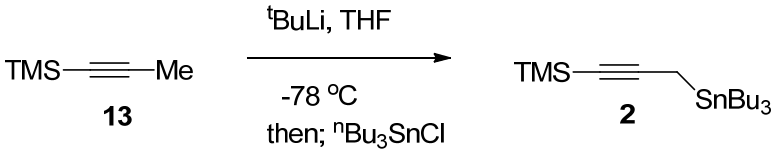


Oct-2-ynyl methanesulfonate (12): To a solution of propargylic alcohol **10** (2.00 g, 15.85 mmol) in CH_2Cl_2 (30 mL) were added Et_3N (5.52 mL, 39.63 mmol) and MsCl (2.45 mL, 31.70 mmol) at -50 °C. The solution was stirred for 4 h at -50 °C to -40 °C. The reaction was then warmed to 0 °C, quenched with saturated aqueous $NaHCO_3$ and extracted in diethylether (3 × 40 mL). The combined organic layers were dried over anhydrous Na_2SO_4 and evaporated *in vacuo.* The residue was purified by flash column chromatography on silica gel (10% ethyl acetate in hexanes) to afford **12** (2.57 g, 80 % yield) as a clear oil. R_f = 0.53 (SiO_2, 30% ethyl acetate in hexanes); IR (film) ν_{max} 2935, 2862, 2235, 1364, 1176, 974, 941, 806 cm^{-1}; ^1H NMR ($CDCl_3$, 300 MHz) δ 4.85(2H, t, *J* = 2.1 Hz), 3.10(3H, s), 2.24(2H, dt, *J* = 6.9, 2.1 Hz), 1.55-1.48(2H, m), 1.37-1.25(4H, m), 0.89(3H, t, *J* = 6.9 Hz); ^{13}C NMR ($CDCl_3$, 75.5 MHz) δ 91.2, 72.4, 58.8, 39.1, 31.1, 27.9, 22.2, 18.8, 14.0; HRMS *m/e* calcd. for $C_9H_{16}O_3S$ $(M+H)^+$ 205.0893 found 205.0887.



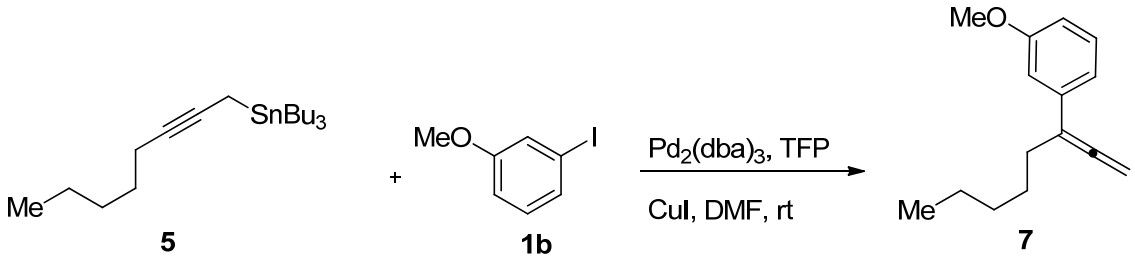
Tributyl(octa-1,2-dien-3-yl)stannane (6): To a solution of anhydrous $^iPr_2NH$ (0.2 mL, 1.36 mmol) in THF (3 mL) was added nBuLi (2.30M hexane solution, 0.56 mL, 1.30 mmol) at 0 °C. After 45 min, $^nBu_3SnH$ (0.34 mL, 1.25 mmol) was added. After 30 min, the resulting clear yellow solution was cooled to -78 °C, and the mixture was then added to a pre-cooled (-78 °C) solution of $CuBr.SMe_2$ (465 mg, 2.26 mmol) in THF (1 mL). After 30 min, a solution of

mesylate **12** (200 mg, 0.98 mmol) in THF (1 mL) was added at -78 °C. The reaction mixture was warmed to -10 °C over a period of 3 h and quenched with saturated aqueous $NaHCO_3$. The aqueous layer was extracted in diethylether (3 × 20 mL). The combined organic layers were then dried over anhydrous Na_2SO_4 and evaporated *in vacuo.* The residue was purified by flash column chromatography on silica gel (100% pentane) to afford the mixture of allenyl stannane **6** and propargyl stannane **5** in a 60:40 ratio (38% yield) as a clear oil. R_f = 0.55 (SiO_2, 100% hexanes); ^{1}H NMR for **6** ($CDCl_3$, 400 MHz) δ 4.13(2H, t, *J* = 3.0 Hz), 2.09-2.04(2H, m), 1.57-1.04(21H, m), 1.02-0.879(15H, m). ^{13}C NMR for the mixture ($CDCl_3$, 75.5 MHz) δ 204.6, 91.5, 80.2, 78.1, 65.0, 32.5, 31.8, 31.4, 30.9, 29.8, 29.5, 29.2, 27.8, 27.7, 27.5, 22.7, 22.5, 19.3, 14.3, 14.2, 13.9, 10.3, 9.9, -4.1.

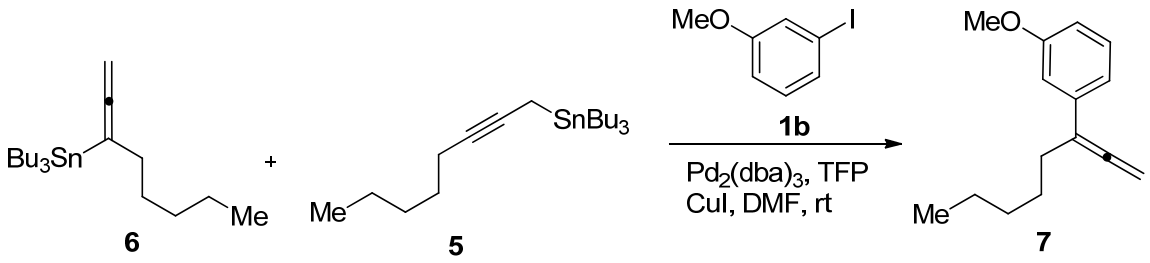


3-Tri-n-butylstannyl-1-trimethysilyl-1-propyne (2): To a solution of **13** (1 gm, 8.90 mmol) in THF (22 mL) was added ^{t}BuLi (1.70 M hexane solution, 9.42 mL, 16.02 mmol) at -78 °C and stirred for 2 h at -78 °C. To the resulting clear yellow solution was added ^{n}Bu_3SnCl (3.12 mL, 11.57 mmol) quickly at -78 °C and stirring was continued for 45 min at -78 °C. The solution was then quenched with saturated aqueous $NaHCO_3$ at -78 °C and warmed to rt. The aqueous layer was extracted with diethylether (3 × 15 mL). The combined organic layers were dried over anhydrous Na_2SO_4 and evaporated *in vacuo.* The residue was then purified by flash column chromatography on silica gel (100% hexanes) to afford **2** in quantitative yield as a clear oil. R_f = 0.65 (SiO_2, 100% pentane); IR (film) ν_{max} 2957, 2926, 2164, 1464, 1248, 1044, 841 cm^{-1}; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.61(2H, s), 1.58-1.26(12H, m), 0.98(6H, t, *J* = 8.0 Hz), 0.90(9H, t,

J = 7.2 Hz), 0.12(9H, s); ^{13}C NMR ($CDCl_3$, 75.5 MHz) δ 109.1, 82.1, 29.1, 27.5, 13.9, 10.2, 0.6, -2.2; HRMS *m/e* calcd. for $C_{17}H_{35}SiSn$ $(M\text{-}CH_3)^+$ 387.1525 found 387.1512.

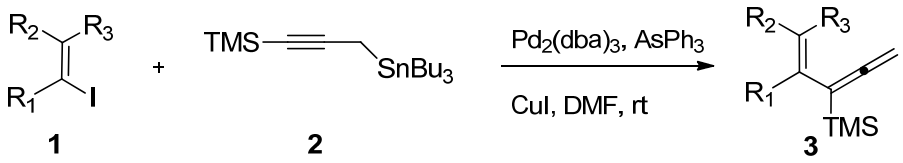


1-Methoxy-3-(octa-1,2-dien-3-yl)benzene (7): To a solution of **5 (**35 mg, 0.09 mmol) and **1b** (8 μL, 0.07 mmol) in DMF (1 mL) was added Tri-(2-furyl)phosphine (TFP) (3.48 mg, 20 mol%) followed by $Pd_2(dba)_3$ (3.30 mg, 5 mol%) and CuI (2.86 mg, 20 mol%) at rt. The reaction was then stirred for 9 hr at rt and was quenched with saturated aqueous $NaHCO_3$. The aqueous layer was extracted in pentane (4 × 25 mL). The combined organic layers were then dried over anhydrous Na_2SO_4 and evaporated *in vacuo.* The residue was then purified by flash column chromatography on silica gel (100% pentane then gradient to 12% diethylether in pentane containing 1% Et_3N) to afford **7** in quantitative yield as a clear oil. R_f = 0.13 (SiO_2, 100% pentane); IR (film) ν_{max} 3054, 2955, 2926, 1939, 1598, 1581, 1487, 1454, 1052, 849 cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.24(1H, t, *J* = 7.6 Hz), 7.01(1H, d, *J* = 7.6 Hz), 6.96(1H, s), 6.75(1H, dd, *J* = 8.4, 2.4 Hz), 5.06(2H, t, *J* = 3.2 Hz), 3.81(3H, s), 2.39(2H, m), 1.57-1.32(6H, m), 0.90(3H, t, *J* = 6.8 Hz); ^{13}C NMR ($CDCl_3$, 101 MHz) δ 208.9, 159.9, 138.4, 129.4, 118.8, 112.13, 112.09, 105.2, 78.3, 55.4, 31.9, 29.7, 27.8, 22.8, 14.3; HRMS *m/e* calcd. for $C_{15}H_{20}O$ $(M+H)^+$ 217.1548 found 217.1569.

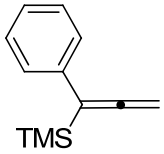


A 60:40 ratio of **6** and **5** (15 mg, 0.04mmol) was dissolved in DMF (1.5mL). To this solution was added **1b** (19 μL, 0.16 mmol) followed by Tri-(2-furyl)phosphine (TFP) (7.43 mg, 20 mol%), $Pd_2(dba)_3$ (3.30 mg, 5 mol%) and CuI (2.86 mg, 20 mol%) at rt. The mixture was stirred for 9 hr at rt and was quenched with saturated aqueous $NaHCO_3$. The aqueous layer was extracted in pentane (4 × 15 mL). The combined organic layers were then dried over anhydrous Na_2SO_4 and evaporated *in vacuo.* The residue was then purified by flash column chromatography on silica gel (100% pentane containing 1% Et_3N) to afford **7** (8 mg, 72% yield) as a clear oil. Spectroscopy data is as described above for **7**.

General Procedure for Stille cross-coupling reactions:

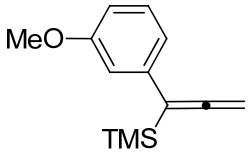


To a solution of propargyl stannane **2 (**1.3 equiv) and iodide **1** (1 equiv.) in DMF (~0.2 M) was added $AsPh_3$ (0.8 equiv) followed by $Pd_2(dba)_3$ (20 mol%) and CuI (0.8 equiv) at rt. The reaction was stirred at 22 °C unless otherwise mentioned for 8-15 hrs. All reactions were monitored by TLC for completion. The reaction mixture then was quenched with saturated aqueous $NaHCO_3$. The aqueous layer was extracted in hexane or diethylether (4 × 15 mL). The combined organic layers were dried over anhydrous Na_2SO_4 and evaporated *in vacuo.* The residue was then purified by flash silica gel column chromatography to afford the cross-coupling product **3**.



3a

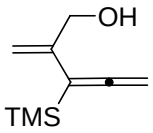
Trimethyl(1-phenylpropa-1,2-dien-1-yl)silane (3a): Following the general procedure, **3a** was prepared using iodobenzene (**1a**) (0.11 mL, 0.98 mmol). The crude product was purified by flash column chromatography on silica gel (100% hexanes the gradient to 1% diethylether in pentane) to afford the desired product (70 mg, 76%) as a clear oil. Analytical data was compared with literature data and found to be identical.[2]



3b

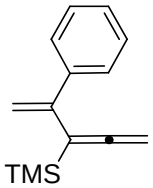
1-Methoxy-3-(octa-1,2-dien-3-yl)benzene (3b): Following the general procedure, **3b** was prepared using 3-iodoanisole (**1b**) (300 mg, 1.28 mmol). The reaction was stirred at 45°C. The crude product was purified by flash column chromatography on silica gel (100% hexanes then 1% ethyl acetate in hexane) to afford the desired product (115 mg, 82%) as a clear oil. R_f = 0.44 (SiO_2, 8% ethyl acetate in hexane); IR (film) ν_{max} 3068, 3052, 2956, 1915,1595, 1578, 1250, 1049, 841,737, 696 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz) δ 7.21(1H, t, *J* = 8.0 Hz), 6.90(1H, d, *J* = 8.0 Hz), 6.87(1H, t, *J* = 2.0 Hz), 6.74(1H, dd, *J* = 8.8, 2.0 Hz), 4.67(2H, s), 3.80(3H, s), 0.24(9H, s); ^{13}C NMR ($CDCl_3$, 101 MHz) δ 211.3, 159.9, 138.8, 129.5, 120.4, 113.5, 111.9, 98.8, 70.8, 55.4, -0.3; HRMS *m/e* calcd. for $C_{13}H_{18}OSi$ $(M)^+$ 218.1121 found 218.1117.

[2] J. Kjellgren, H. Sunden, K. J. Szabo, *J. Am. Chem. Soc*., 2005, **127**, 1787.



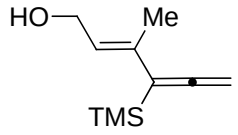
3c

2-Methylene-3-(trimethylsilyl)penta-3,4-dien-1-ol (3c): Following the general procedure, **3c** was prepared using **1c** (10 mg, 0.05 mmol). Tri-(2-furyl)phosphine (TFP) (10.22 mg, 0.44 mmol) was used instead in place of $AsPh_3$. The crude product was purified by flash column chromatography on silica gel (100% pentane then gradient to 20% diethylether in pentane) to afford **3c** (5.5 mg, 61%) as a clear oil. R_f = 0.30 (SiO_2, 20% diethylether in pentane); IR (film) ν_{max} 3357 (br), 2956, 2923, 1910, 1249, 1063, 839 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz) δ 5.19(1H, s), 5.05(1H, s), 4.59(2H, s), 4.23(2H, d, *J* = 6.2 Hz), 1.62(1H, t, *J* = 6.5 Hz), 0.20(9H, s); ^{13}C NMR ($CDCl_3$, 101 MHz) δ 210.4, 143.4, 112.1, 96.8, 71.1, 65.9, -0.4; HRMS *m/e* calcd. for $C_9H_{16}OSi$ $(M)^+$ 168.0965 found 168.0966.



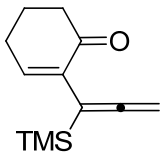
3d

Trimethyl(4-phenylpenta-1,2,4-trien-3-yl)silane (3d): Following the general procedure, **3d** was prepared using **1d** (20 mg, 0.087 mmol). The crude product was purified by flash column chromatography on silica gel (100% pentane) to afford the desired product (8.5 mg, 46% yield) as a clear oil. R_f = 0.63 (SiO_2, 100% pentane); IR (film) ν_{max} 3080, 3057, 3023, 2957, 1922, 1606, 1492, 1249, 841, 813 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz) δ 7.40-7.28(5H, m), 5.30(1H, s), 5.11(1H, s), 4.46(2H, s), 0.11(9H, s); ^{13}C NMR ($CDCl_3$, 101 MHz) δ 211.2, 145.8, 141.4, 128.1, 127.7, 127.6, 113.5, 98.9, 69.4, -0.7; HRMS *m/e* calcd. for $C_{14}H_{18}Si$ $(M)^+$ 214.1172 found 214.1169.



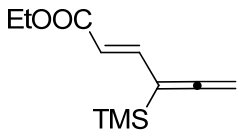
3e

(E)-3-Methyl-4-(trimethylsilyl)hexa-2,4,5-trien-1-ol (3e): Following the general procedure, **3e** was prepared using **1e** (9 mg, 0.045 mmol). The crude product was purified by flash column chromatography on silica gel (100% pentane then gradient to 8% diethylether in pentane) to afford the desired product (5.2 mg, 64% yield) as a clear oil. R_f = 0.17 (SiO_2, 20% diethylether in pentane); IR (film) ν_{max} 3317 (br), 2956, 1909, 1636, 1417, 1250, 1000, 840 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz) δ 5.58(1H, t, *J* = 6.5 Hz), 4.58(1H, s), 4.25(2H, t, *J* = 5.9 Hz), 1.83(3H, s), 1.19(1H, t, *J* = 5.5 Hz), 0.19(9H, s); ^{13}C NMR ($CDCl_3$, 101 MHz) δ 211.8, 134.8, 126.5, 101.6, 71.3, 60.2, 16.6, -0.03; HRMS *m/e* calcd. for $C_{10}H_{18}OSi$ $(M)^+$ 182.1121 found 182.1118.



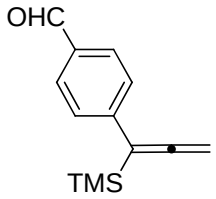
3f

2-(1-(Trimethylsilyl)propa-1,2-dienyl)cyclohex-2-enone (3f): Following the general procedure, **3f** was prepared using **1f** (150 mg, 0.68 mmol). The crude product was purified by flash column chromatography on silica gel (100% pentane then gradient to 5% diethylether in pentane) to afford the desired product (72 mg, 51% yield) as a clear oil. R_f = 0.66 (SiO_2, 20% diethylether in pentane); IR (film) ν_{max} 2953, 1925, 1679, 1358, 1246, 1167, 1129, 842 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz) δ 6.79(1H, t, *J* = 4.2 Hz), 4.49(2H, s), 2.48-2.39(4H, m), 2.03-1.97(2H, m), 0.12(9H, s); ^{13}C NMR ($CDCl_3$, 101 MHz) δ 210.5, 198.2, 146.2, 137.3, 94.7, 69.0, 38.9, 26.7, 23.2, -0.5; HRMS *m/e* calcd. for $C_{12}H_{19}OSi$ $(M+H)^+$ 207.1200 found 207.1190.



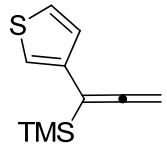
3g

Ethyl 4-(trimethylsilyl)hexa-2,4,5-trienoate (3g): Following the general procedure, **3g** was prepared using ethyl *cis* 3-iodoacrylate (**1g**) (11.33 µL, 0.09 mmol). The crude product was purified by flash column chromatography on silica gel (2% diethylether in pentane) to afford the desired product (9.5 mg, 50%) as a clear oil. R_f = 0.35 (SiO_2, 2% diethylether in pentane); IR (film) ν_{max} 2956, 1910, 1701, 1638; ^{1}H NMR (C_6D_6, 400 MHz) δ 7.66(1H, d, *J* =16.0 Hz), 6.24(1H, d, *J* =16.0 Hz), 4.31(2H, s), 4.06(2H, q, *J* =7.2 Hz), 0.98(3H, t, *J* =7.2 Hz), 0.09(9H, s); ^{13}C NMR (C_6D_6, 101 MHz) δ 216.1, 166.3, 143.5, 120.9, 96.5, 70.3, 60.2, 14.3, -1.2; HRMS *m/e* calcd. for $C_{11}H_{19}O_2Si$ $(M+H)^+$ 211.1149 found 211.1158.



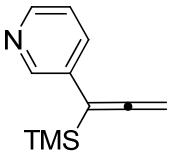
3h

4-(1-(Trimethylsilyl)propa-1,2-dienyl)benzaldehyde (3h): Following the general procedure, **3h** was prepared using **1h** (90 mg, 0.39 mmol). The crude product was purified by flash column chromatography on silica gel (100% pentane then gradient to 5% diethylether in pentane) to afford the desired product (65 mg, 77% yield) as a clear oil. R_f = 0.45 (SiO_2, 10% diethylether in pentane); IR ν_{max} 3051, 2958, 2822, 2732, 1915, 1701, 1600, 1388, 1251, 1213, 841 cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 9.97(1H, s), 7.81(2H, d, *J* = 8.1 Hz), 7.46(2H, d, *J* = 8.0 Hz), 4.76(2H, s), 0.26(9H, s); ^{13}C NMR ($CDCl_3$, 101 MHz) δ 212.3, 192.0, 144.6, 134.6, 130.2, 128.4, 98.9, 71.4, -0.3; HRMS *m/e* calcd. for $C_{13}H_{16}OSi$ $(M)^+$ 216.0965 found 216.0969.



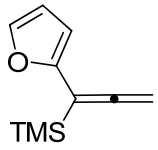
3i

Trimethyl(1-(thiophen-3-yl)propa-1,2-dienyl)silane (3i): Following the general procedure, **3i** was prepared using 3-iodothiophene (**1i**) (12 mg, 0.057 mmol). The crude product was purified by flash column chromatography on silica gel (100% pentane) to afford the desired product (7.20 mg, 65% yield) as a clear oil. R_f = 0.50 (SiO_2, 100% hexanes); IR (film) ν_{max} 3123, 2957, 1925, 1249, 838, 780 cm^{-1}; ^1H NMR (acetone-*d6*, 400 MHz) δ 7.42(1H, dd, *J* = 5.1, 2.8 Hz), 7.21(1H, s), 7.11(1H, dd, *J* = 5.1, 1.2 Hz), 4.71(2H, s), 0.25(9H, s); ^{13}C NMR (acetone-*d6*, 101 MHz) δ 212.3, 137.4, 128.4, 126.2, 121.1, 94.3, 70.8, -0.6; HRMS *m/e* calcd. for $C_{10}H_{14}SSi$ $(M)^+$ 194.0580 found 194.0571.



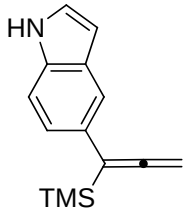
3j

3-(1-(Trimethylsilyl)propa-1,2-dienyl)pyridine (3j): Following the general procedure, **3j** was prepared using 3-iodopyridine (**1j**) (10 mg, 0.049 mmol). The crude product was purified by flash column chromatography on silica gel (100% pentane then gradient to 15% diethylether in pentane) to afford the desired product (5.75 mg, 62% yield) as a clear oil. R_f = 0.17 (SiO_2, 20% diethylether in pentane); IR (film) ν_{max} 3028, 2958, 1918, 1475, 1251, 840 cm^{-1}; ^1H NMR (C_6D_6, 400 MHz) δ 9.03(1H, br s), 8.59(1H, br s), 7.48(1H, d, *J* = 7.7 Hz), 6.84(1H,s), 4.54(2H, s), 0.26(9H, s); ^{13}C NMR (C_6D_6, 101 MHz) δ 212.1, 149.8, 148.4, 134.7, 133.4, 123.7, 96.6, 71.3, -0.4; HRMS *m/e* calcd. for $C_{11}H_{15}NSi$ $(M)^+$ 189.0968 found 189.0966.



3k

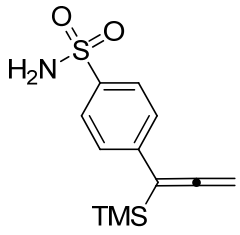
(1-(Furan-2-yl)propa-1,2-dienyl)trimethylsilane (3k): Following the general procedure, **3k** was prepared using 1-iodofuran (**1k**) (10 mg, 0.049 mmol). The crude product was purified by flash column chromatography on silica gel (100% pentane) to afford the desired product (6.65 mg, 72% yield) as a clear oil. R_f = 0.63 (SiO_2, 100% pentane); IR (film) ν_{max} 3115, 2958, 1920, 1486, 1465, 1249, 841, 758 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz) δ 7.36(1H, s), 6.37(1H, dd, *J* = 3.1, 2.0 Hz), 6.20(1H, d, *J* = 3.0 Hz), 4.81(2H, s), 0.25(9H, s); ^{13}C NMR ($CDCl_3$, 101 MHz) δ 210.8, 150.2, 141.8, 111.4, 106.93, 106.89, 72.3, -0.7; HRMS *m/e* calcd. for $C_{10}H_{14}OSi$ $(M)^+$ 178.0808 found 178.0816.



3l

5-(1-(Trimethylsilyl)propa-1,2-dienyl)-1H-indole (3l): Following the general procedure, **3l** was prepared using 5-iodoindole (**1l**) (10 mg, 0.041 mmol). The reaction mixture was stirred at 70°C. The crude product was purified by flash column chromatography on silica gel (100% pentane then gradient to 30% diethylether in pentane) to afford the desired product (5.10 mg, 54% yield) as a white crystals. R_f = 0.23 (SiO_2, 30% diethylether in pentane); IR (film) ν_{max} 3375, 3098, 3047, 2958, 1914, 1466, 1250, 839, 806 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz) δ 8.19(1H, br s), 7.55(1H, s), 7.34-7.32(1H, m), 7.22-7.17(2H, m), 6.51(1H, s), 4.67(2H, s),

0.28(9H, s); ^{13}C NMR ($CDCl_3$, 101 MHz) δ 211.1, 134.8, 128.6, 128.4, 124.6, 122.7, 119.6, 111.2, 102.9, 99.3, 70.3, -0.1; HRMS *m/e* calcd. for $C_{14}H_{17}NSi$ $(M)^+$ 227.1125 found 227.1121.



3m

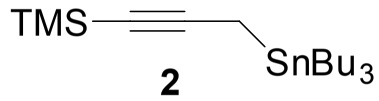
4-(1-(Trimethylsilyl)propa-1,2-dienyl)benzenesulfonamide (3m): Following the general procedure, **3m** was prepared using 4-iodobenzenesulfonamide (**1m**) (15 mg, 0.053 mmol). The crude product was purified by flash column chromatography on silica gel (100% hexanes then gradient to 20% EtOAc in hexanes) to afford the desired product (14.16 mg, 71% yield) as a clear oil. R_f = 0.24 (SiO_2, 30% EtOAc in hexanes); IR (film) ν_{max} 3319, 3250, 2956, 1918, 1590, 1328, 1166, 1140, 842, 809 cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.85(2H, d, *J* = 8.6 Hz), 7.43(2H, d, *J* = 8.6 Hz), 4.79(2H,br s), 4.76(2H, s), 0.25(9H, s); ^{13}C NMR ($CDCl_3$, 101 MHz) δ 212.2, 143.0, 139.4, 128.4, 126.9, 98.4, 71.5, -0.4; HRMS *m/e* calcd. for $C_{12}H_{18}O_2NSSi$ $(M+H)^+$ 268.0822 found 268.0823.

AAS.1.106.1H

Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / K298.1
File: AAS_1_106_1H
GEMINI-300 "nmrsun4"

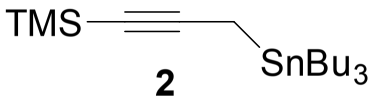
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.731 sec
Width 4500.0 Hz
16 repetitions
OBSERVE H1, 300.0673506 MH
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 0 min, 59 sec



2

AAS.1.106.13C

Archive directory:
Sample directory:

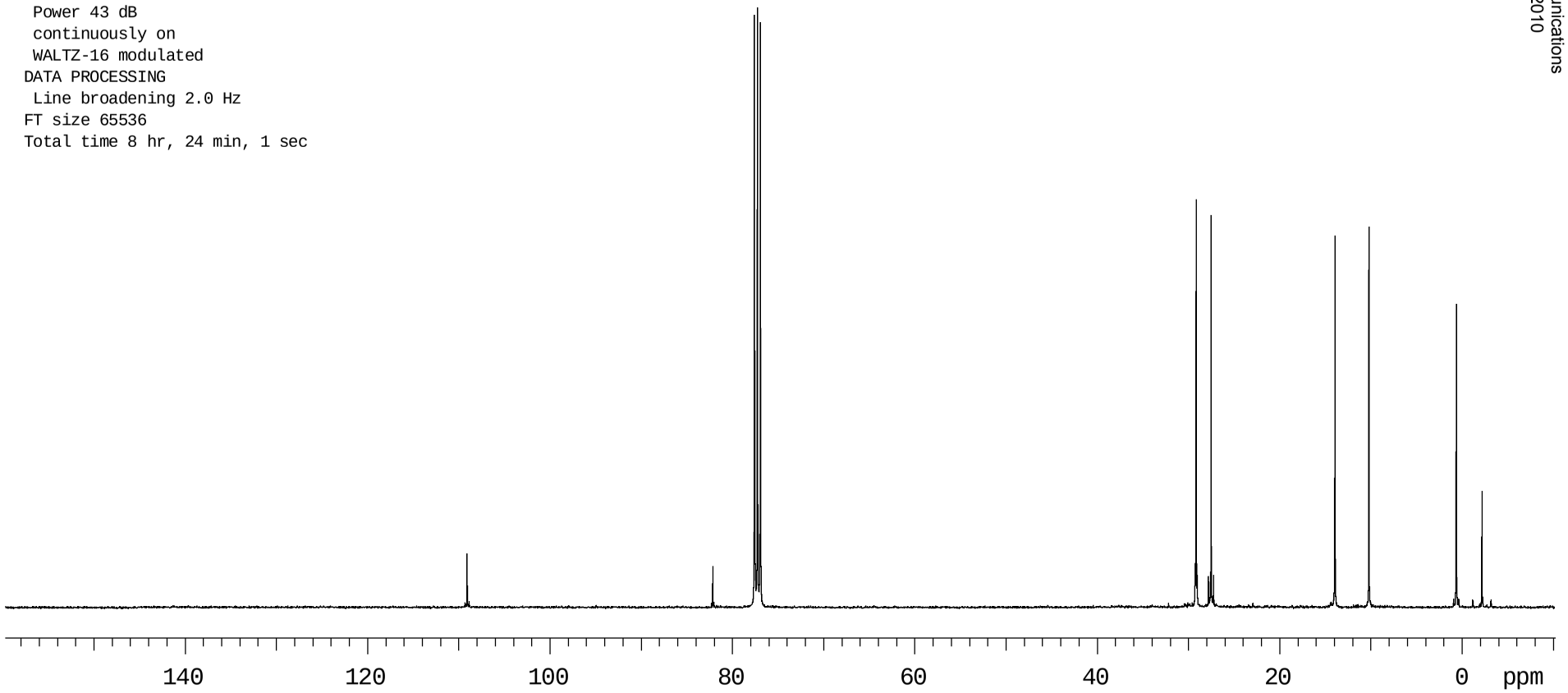


2

Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
File: AAS_1_106_13C
INOVA-400 "nmrsun4"

Relax. delay 1.500 sec
Pulse 29.7 degrees
Acq. time 0.651 sec
Width 25157.2 Hz
14000 repetitions
OBSERVE C13, 100.6073042 MHz
DECOUPLE H1, 400.1103789 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 8 hr, 24 min, 1 sec



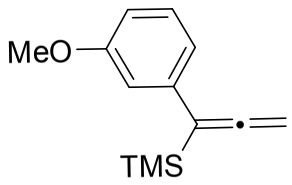
AAS.1.ONE.1H

Archive directory:
Sample directory:

Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / K298.1
File: AAS.1.one.1H
VXR-400 "nmrsun4"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.560 sec
Width 6399.0 Hz
16 repetitions
OBSERVE H1, 399.9252124 MH
DATA PROCESSING
Line broadening 0.3 Hz
FT size 32768
Total time 0 min, 57 sec



3b

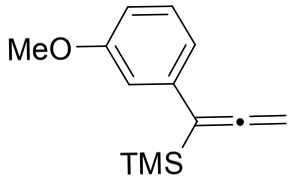
AAS.1.ONE.13C

Archive directory:
Sample directory:

Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
User: 1-14-87
File: AAS.1.1one.13c
VXR-400 "nmrsun4"

Relax. delay 3.000 sec
Pulse 45.0 degrees
Acq. time 1.303 sec
Width 25141.4 Hz
9000 repetitions
OBSERVE C13, 100.5612523 MH
DECOUPLE H1, 399.9272260 MH
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 10 hr, 46 min, 50 sec



3b

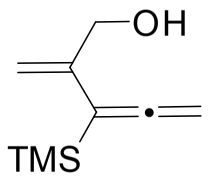
AAS.1.294.1H

Archive directory:
Sample directory:

Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
File: AAS_1_294_b
VXR-400 "nmrsun4"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.730 sec
Width 6000.6 Hz
16 repetitions
OBSERVE H1, 400.1082741 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 0 min, 59 sec

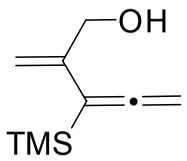


3c

AAS.1.294.13C

Archive directory:
Sample directory:

Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
User: 1-14-87
File: AAS.1.294.b.13C
VXR-400 "nmrsun4"



3c

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.303 sec
Width 25141.4 Hz
10000 repetitions
OBSERVE C13, 100.5612523 MH
DECOUPLE H1, 399.9272260 MH
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 9 hr, 12 min, 2 sec

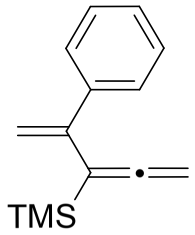
AAS.2.132

Archive directory:
Sample directory:

Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
File: AAS.2.132
VXR-400 "nmrsun4"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.560 sec
Width 6399.0 Hz
32 repetitions
OBSERVE H1, 399.9252124 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 32768
Total time 1 min, 54 sec



3d

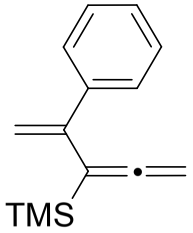
AAS.2.132.13C

Archive directory:
Sample directory:

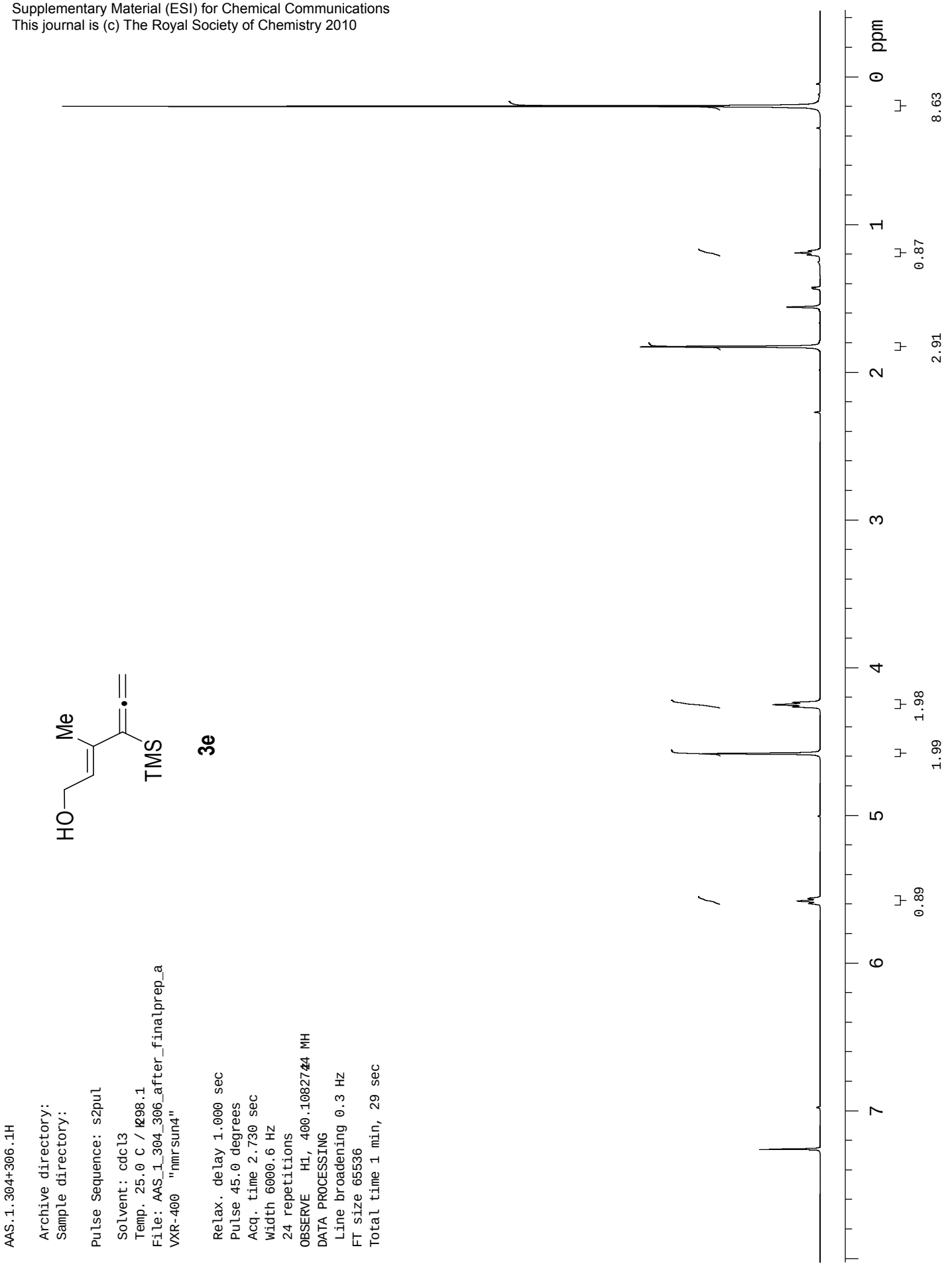
Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
User: 1-14-87
File: AAS.2.132.13C
VXR-400 "nmrsun4"

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.303 sec
Width 25141.4 Hz
9936 repetitions
OBSERVE C13, 100.5612523 MH
DECOUPLE H1, 399.9272260 MH
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 9 hr, 12 min, 2 sec



3d


AAS.1.304+306.1H
Archive directory:
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
File: AAS_1_304_306_after_finalprep_a
VXR-400 "nmrsun4"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.730 sec
Width 6000.6 Hz
24 repetitions
OBSERVE H1, 400.1082744 MH
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 1 min, 29 sec
HO
Me
TMS
3e
ppm
0
1
2
3
4
5
6
7
8.63
2.91
0.87
1.98
1.99
0.89

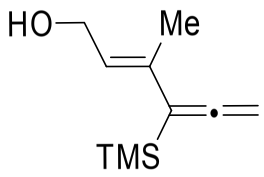
AAs.1.304+306.13C

Archive directory:
Sample directory:

Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
File: AAS_1_304_306_13C
INOVA-400 "nmrsun4"

Relax. delay 1.500 sec
Pulse 29.7 degrees
Acq. time 0.651 sec
Width 25157.2 Hz
9000 repetitions
OBSERVE C13, 100.6072819 MHz
DECOUPLE H1, 400.1102746 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 5 hr, 24 min, 0 sec



3e

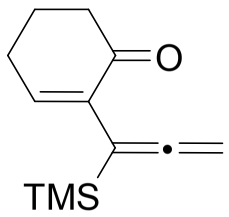
AAs.1.296.1H

Archive directory:
Sample directory:

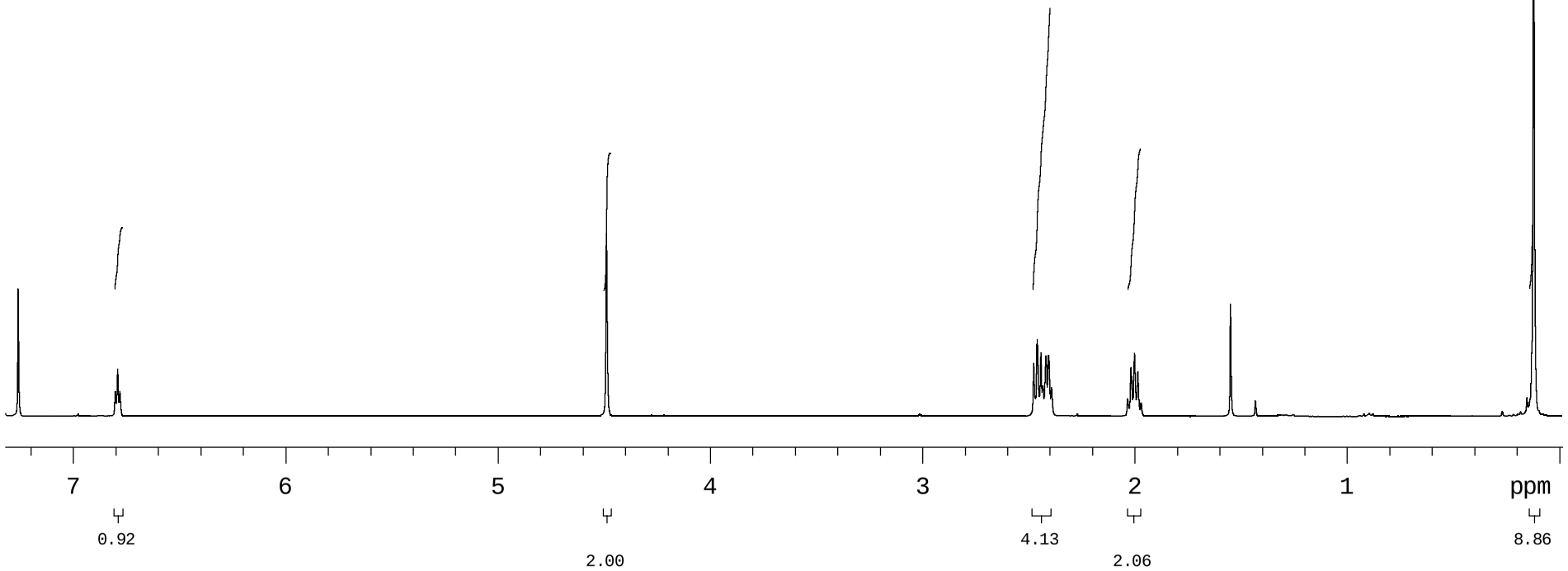
Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
File: AAS_1_296_1H
VXR-400 "nmrsun4"

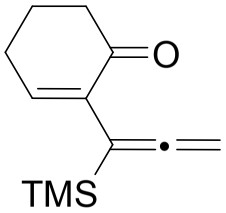
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.730 sec
Width 6000.6 Hz
24 repetitions
OBSERVE H1, 400.1082739 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 1 min, 29 sec



3f



AAS.1.296.13C



3f

Archive directory:
Sample directory:

Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
File: AAS.1.296.13C
INOVA-400 "nmrsun4"

Relax. delay 1.500 sec
Pulse 29.7 degrees
Acq. time 0.651 sec
Width 25157.2 Hz
4000 repetitions
OBSERVE C13, 100.6072819 MH
DECOUPLE H1, 400.1102746 MH
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 2 hr, 24 min, 0 sec

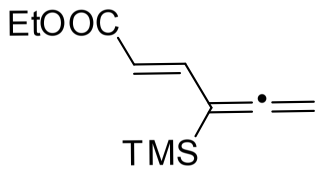
AAS-2-272-1H

Archive directory:
Sample directory:

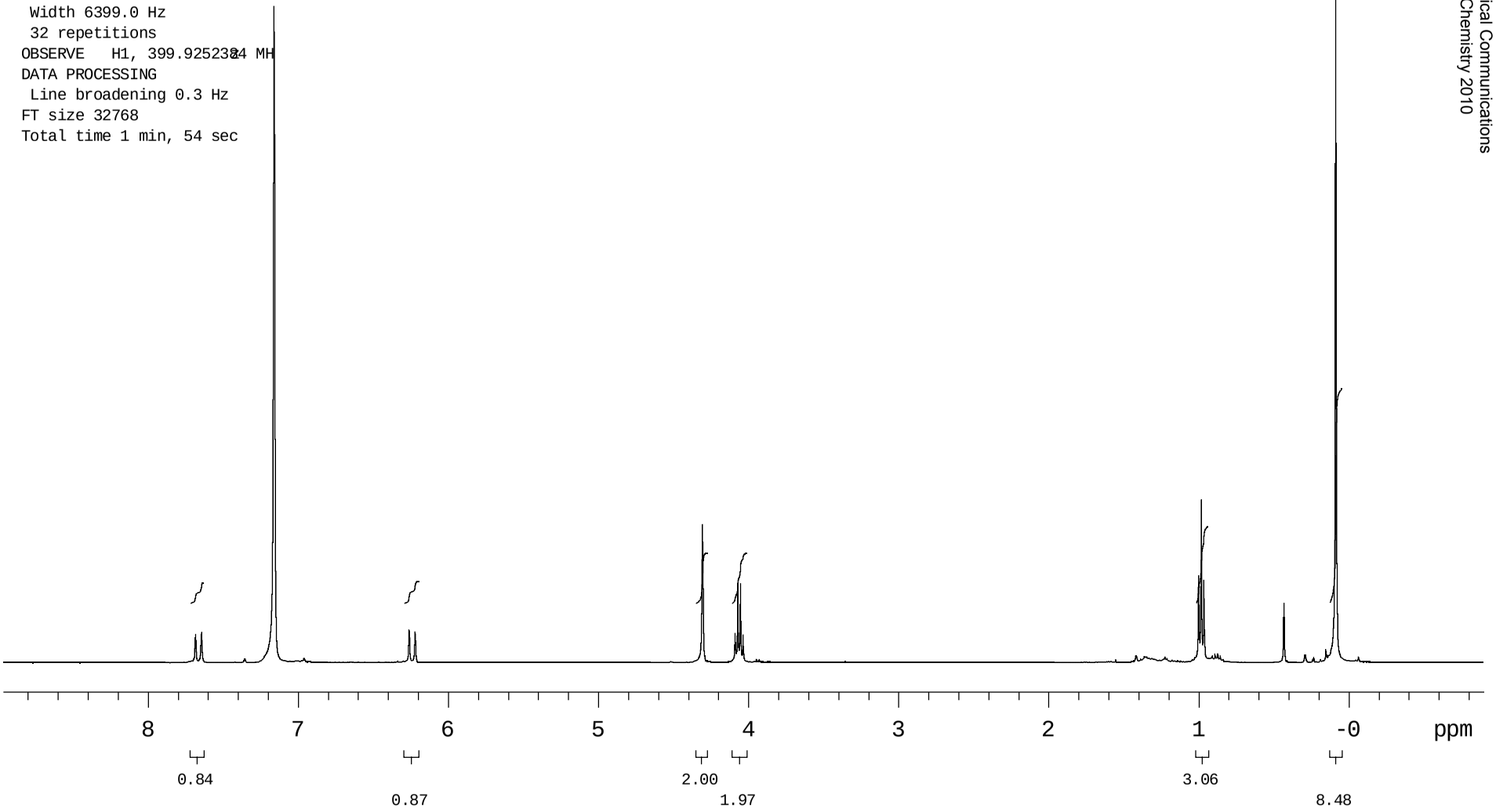
Pulse Sequence: s2pul

Solvent: c6d6
Temp. 25.0 C / 298.1 K
File: AAS-2-272-pure-1H
VXR-400 "nmrsun4"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.560 sec
Width 6399.0 Hz
32 repetitions
OBSERVE H1, 399.9252384 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 32768
Total time 1 min, 54 sec



3g



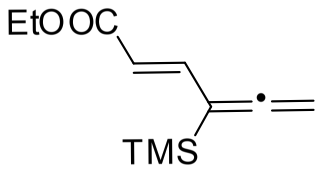
AAS-2-272-13C

Archive directory:
Sample directory:

Pulse Sequence: s2pul

Solvent: c6d6
Temp. 25.0 C / K298.1
User: 1-14-87
File: AAS-2-272-13c
VXR-400 "nmrsun4"

Relax. delay 2.000 sec
Pulse 30.0 degrees
Acq. time 1.303 sec
Width 25141.4 Hz
13000 repetitions
OBSERVE C13, 100.5612451 MH
DECOUPLE H1, 399.9272626 MH
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 11 hr, 57 min, 39 sec



3g

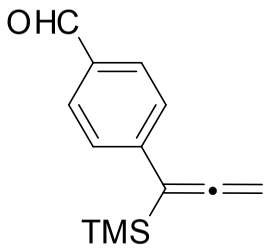
AAS.2.84.1H

Archive directory:
Sample directory:

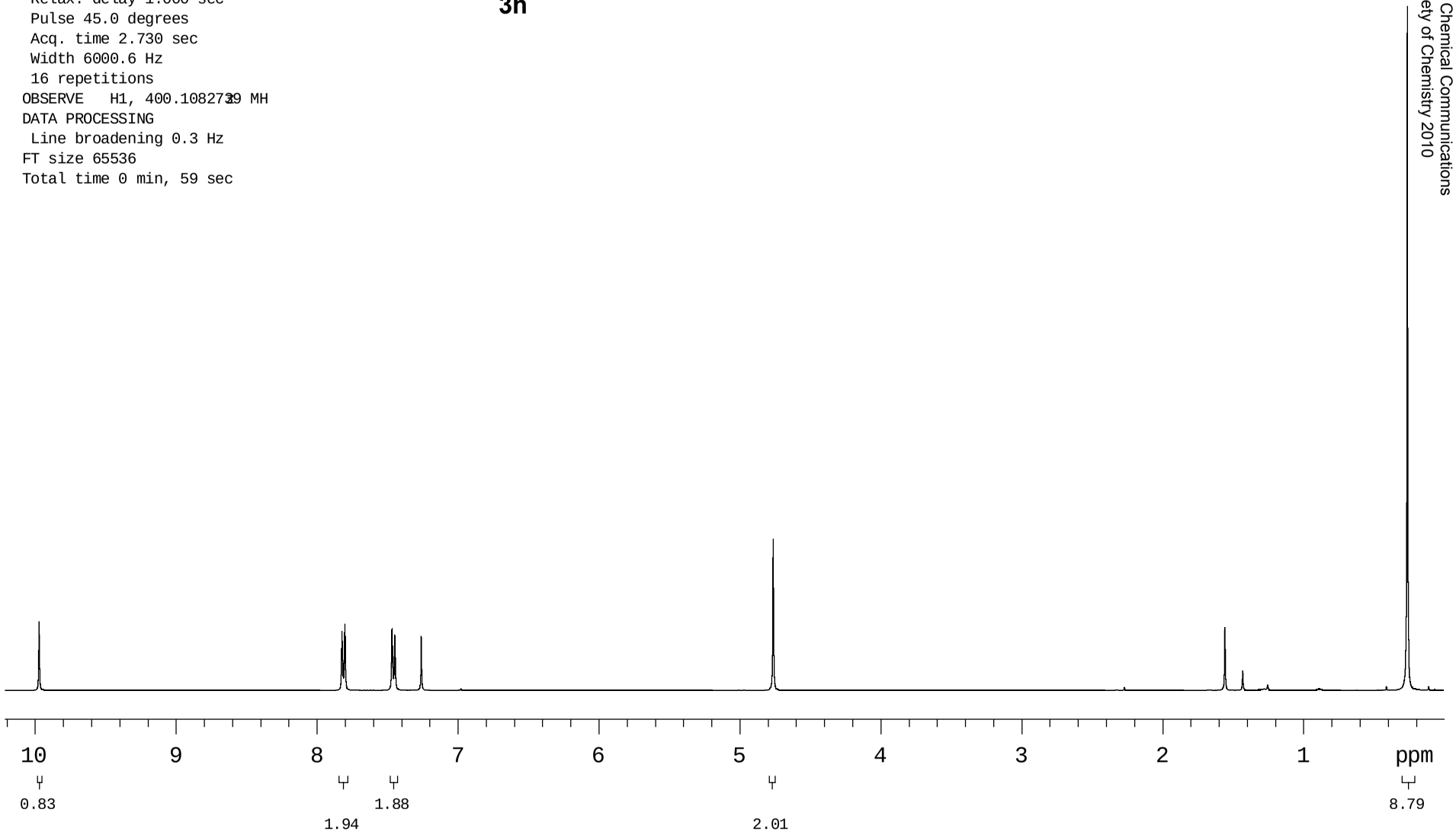
Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 0.0 C / 273.1 K
File: AAS_2_84_1H
VXR-400 "nmrsun4"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.730 sec
Width 6000.6 Hz
16 repetitions
OBSERVE H1, 400.1082739 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 0 min, 59 sec



3h



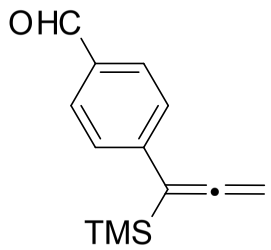
AAS.2.84.13C

Archive directory:
Sample directory:

Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
User: 1-14-87
File: AAS.2.84.13C
VXR-400 "nmrsun4"

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.303 sec
Width 25141.4 Hz
9000 repetitions
OBSERVE C13, 100.5612531 MH
DECOUPLE H1, 399.9272260 MH
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 8 hr, 16 min, 50 sec



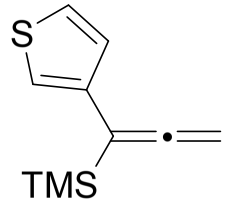
3h

AAs.2.162.1H

Archive directory:
Sample directory:

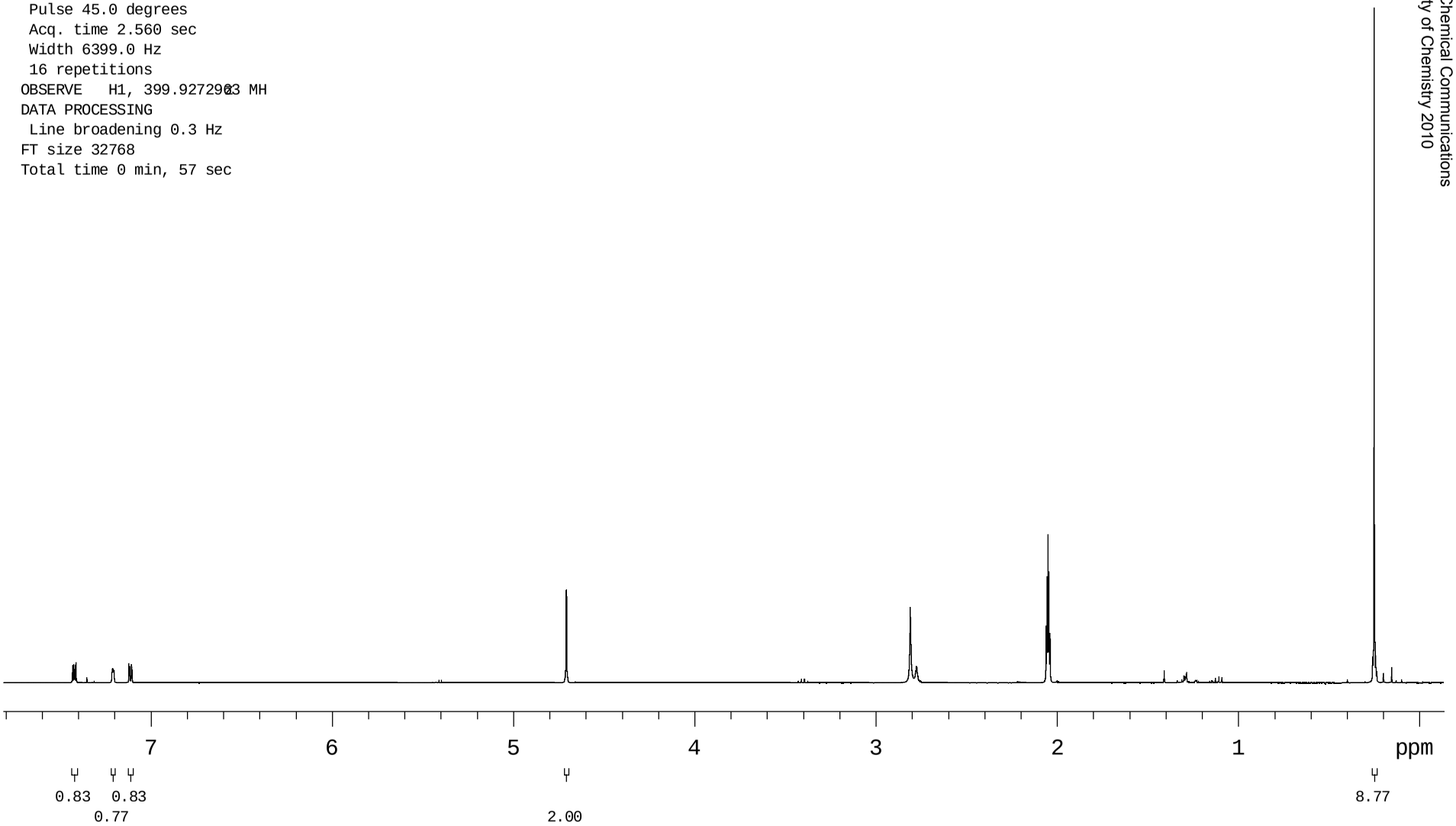
Pulse Sequence: s2pul

Solvent: acetone
Temp. 25.0 C / 298.1 K
File: AAS.2.162.1H
VXR-400 "nmrsun4"



3i

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.560 sec
Width 6399.0 Hz
16 repetitions
OBSERVE H1, 399.9272903 MH
DATA PROCESSING
Line broadening 0.3 Hz
FT size 32768
Total time 0 min, 57 sec



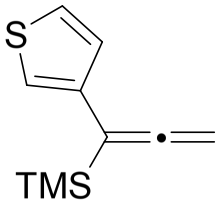
AAS.2.162.13C

Archive directory:
Sample directory:

Pulse Sequence: s2pul

Solvent: acetone
Temp. 25.0 C / 298.1 K
User: 1-14-87
File: AAS.2.162.13C
VXR-400 "nmrsun4"

Relax. delay 3.000 sec
Pulse 45.0 degrees
Acq. time 1.303 sec
Width 25141.4 Hz
9000 repetitions
OBSERVE C13, 100.5617061 MH
DECOUPLE H1, 399.9293022 MH
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 10 hr, 46 min, 50 sec



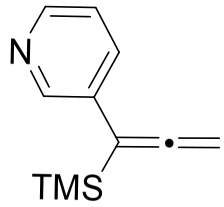
3i

AAS.2.134.1H

Archive directory:
Sample directory:

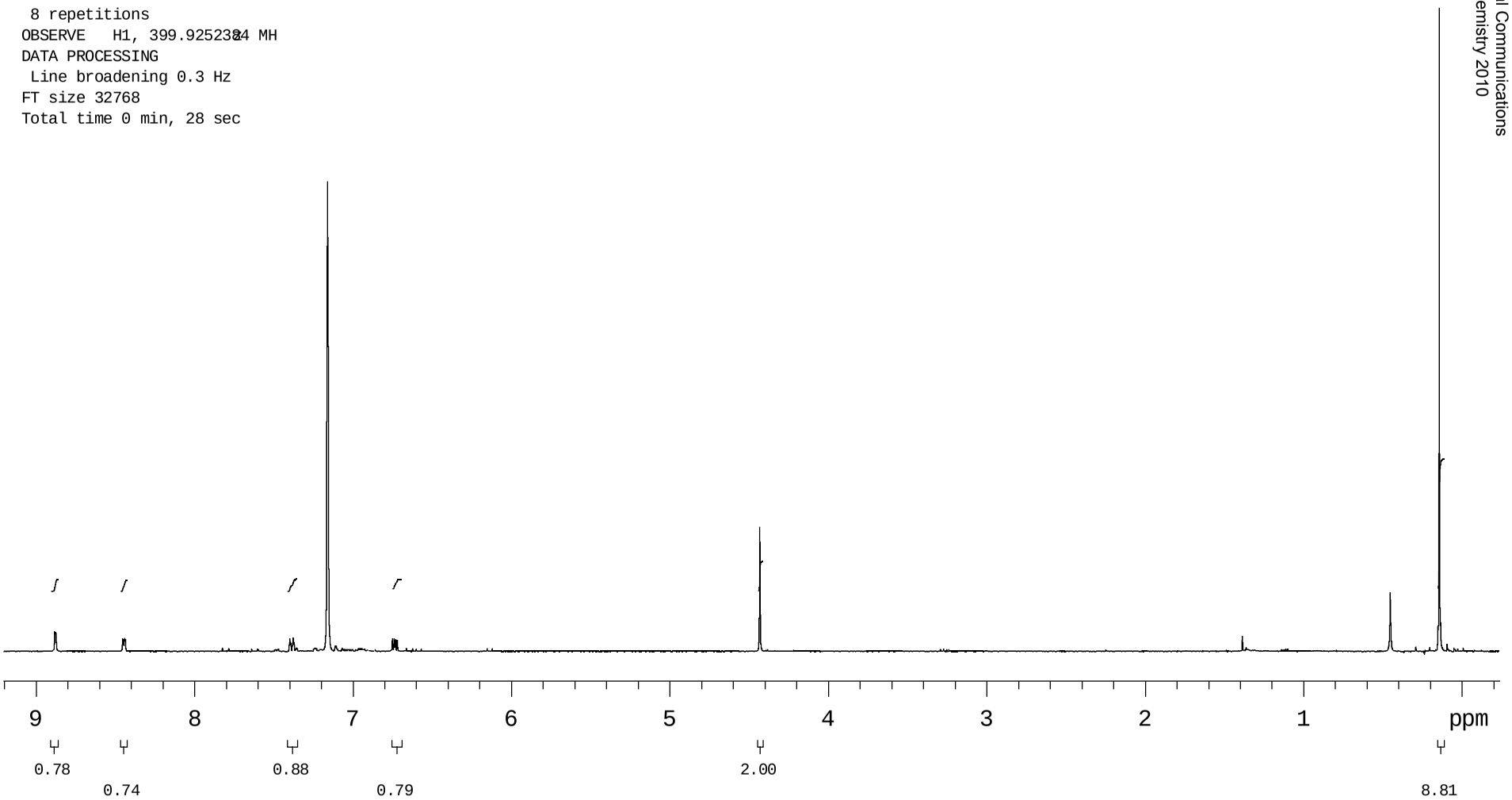
Pulse Sequence: s2pul

Solvent: c6d6
Temp. 25.0 C / 298.1 K
File: AAS.2.134.again
VXR-400 "nmrsun4"



3j

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.560 sec
Width 6399.0 Hz
8 repetitions
OBSERVE H1, 399.9252384 MH
DATA PROCESSING
Line broadening 0.3 Hz
FT size 32768
Total time 0 min, 28 sec



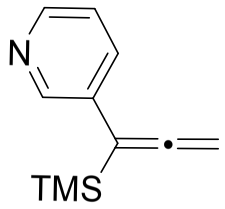
AAS.2.134.13C

Archive directory:
Sample directory:

Pulse Sequence: s2pul

Solvent: c6d6
Temp. 25.0 C / K298.1
User: 1-14-87
File: AAS.2.134.13C
VXR-400 "nmrsun4"

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.303 sec
Width 25141.4 Hz
12000 repetitions
OBSERVE C13, 100.5612451 MH
DECOUPLE H1, 399.9272626 MH
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 11 hr, 2 min, 27 sec



3j

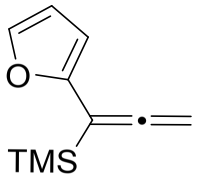
AAS.2.124

Archive directory:
Sample directory:

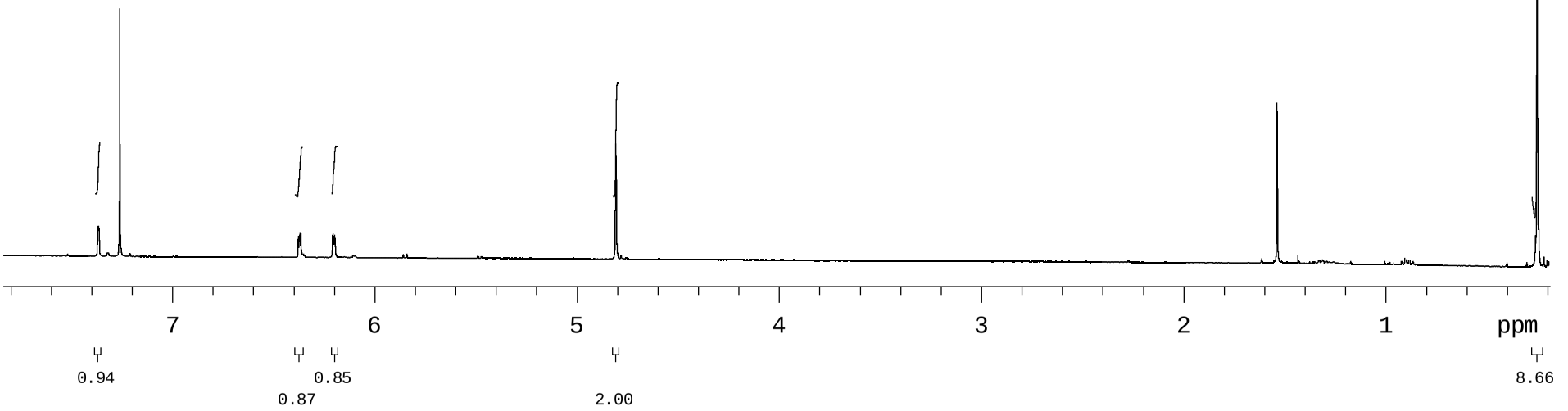
Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
File: AAS.2.124.1H
VXR-400 "nmrsun4"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.560 sec
Width 6399.0 Hz
16 repetitions
OBSERVE H1, 399.9252192 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 32768
Total time 0 min, 57 sec



3k

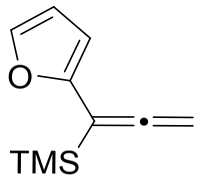


AAS.2.124.13C

Archive directory:
Sample directory:

Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
User: 1-14-87
File: AAS.2.124.13C
VXR-400 "nmrsun4"



3k

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.303 sec
Width 25141.4 Hz
10000 repetitions
OBSERVE C13, 100.5612528 MH
DECOUPLE H1, 399.9272260 MH
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 9 hr, 12 min, 2 sec

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

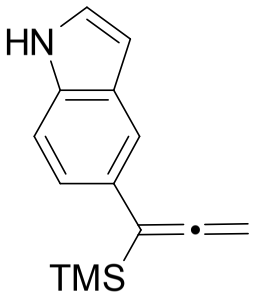
AAs.2.156.1H

Archive directory:
Sample directory:

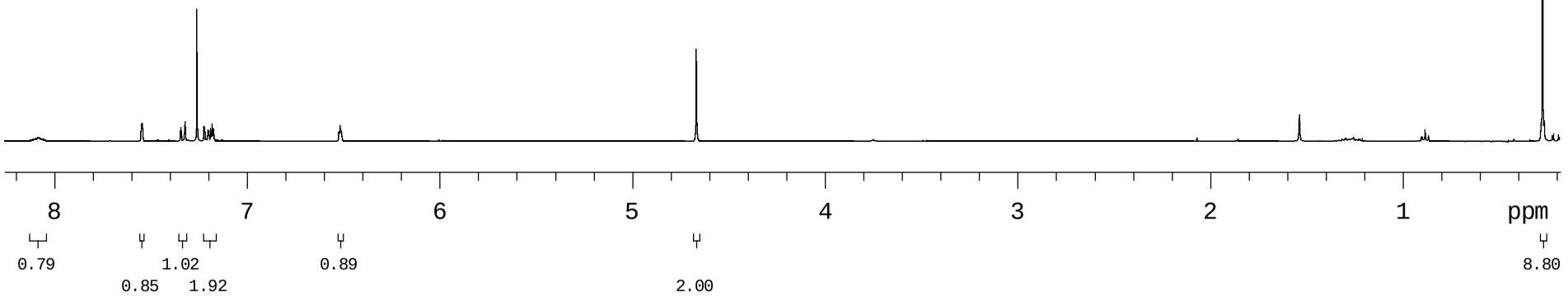
Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
File: AAS.2.156.1H
VXR-400 "nmrsun4"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.560 sec
Width 6399.0 Hz
16 repetitions
OBSERVE H1, 399.9252124 MH
DATA PROCESSING
Line broadening 0.3 Hz
FT size 32768
Total time 0 min, 57 sec



3l



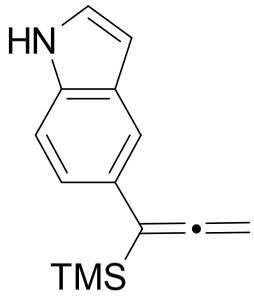
AAS.2.156.13C.again

Archive directory:
Sample directory:

Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
User: 1-14-87
File: AAS.2.156.13C.again
VXR-400 "nmrsun4"

Relax. delay 3.000 sec
Pulse 45.0 degrees
Acq. time 1.303 sec
Width 25141.4 Hz
9000 repetitions
OBSERVE C13, 100.5612531 MH
DECOUPLE H1, 399.9272260 MH
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 10 hr, 46 min, 50 sec



3l

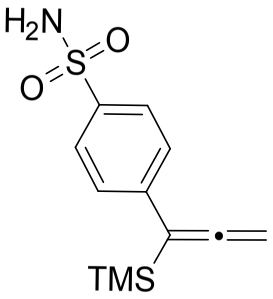
AAS.2.146.1H

Archive directory:
Sample directory:

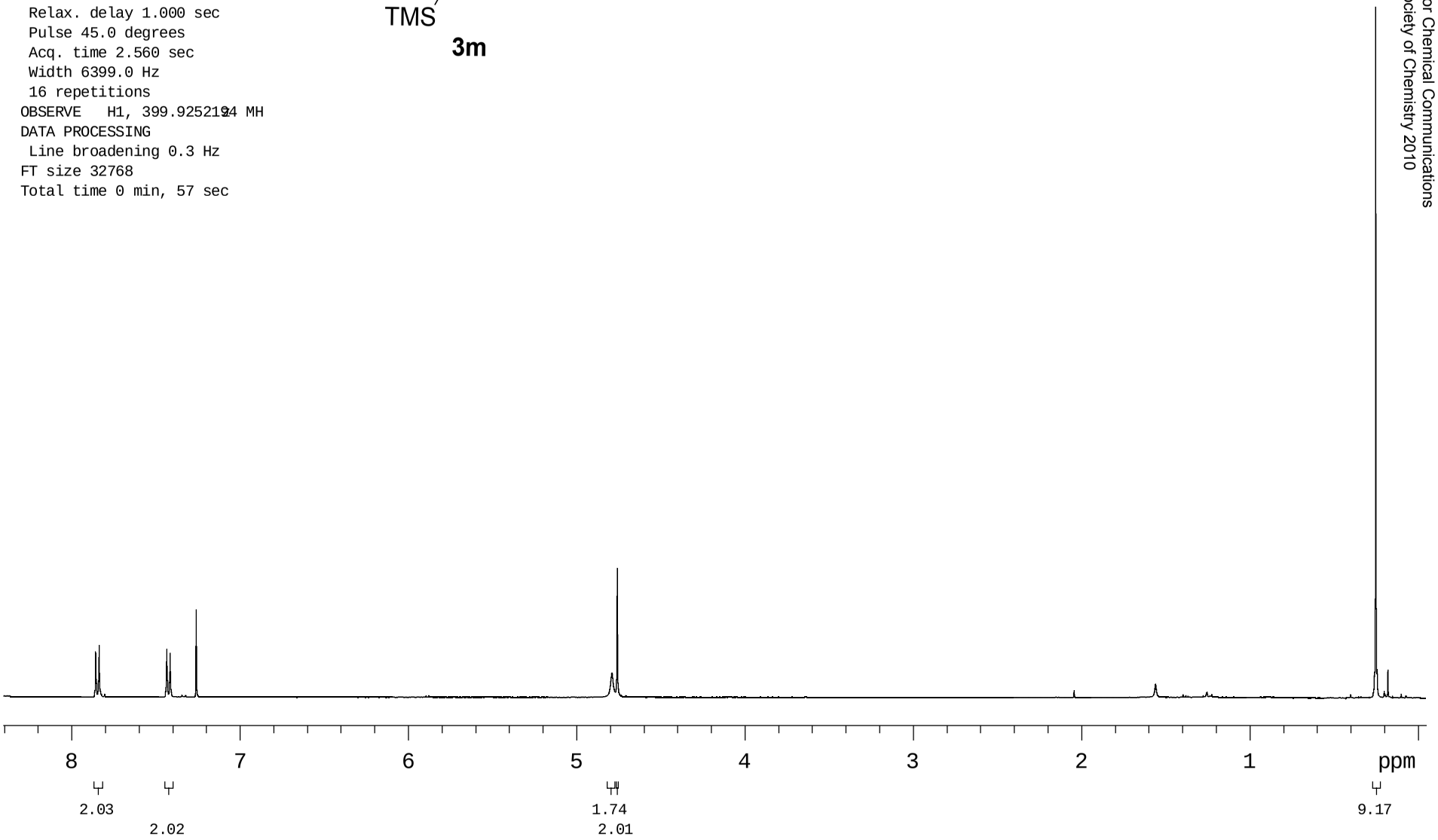
Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
File: AAS.2.146
VXR-400 "nmrsun4"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.560 sec
Width 6399.0 Hz
16 repetitions
OBSERVE H1, 399.9252192 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 32768
Total time 0 min, 57 sec



3m



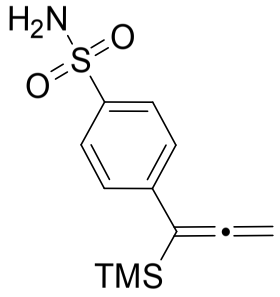
AAS.2.146.13C

Archive directory:
Sample directory:

Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
User: 1-14-87
File: AAS.2.146.13C
VXR-400 "nmrsun4"

Relax. delay 2.000 sec
Pulse 45.0 degrees
Acq. time 1.303 sec
Width 25141.4 Hz
10000 repetitions
OBSERVE C13, 100.5612531 MH
DECOUPLE H1, 399.9272260 MH
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 9 hr, 12 min, 2 sec



3m

AAS.1.104.1H

Archive directory:
Sample directory:

Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
File: AAS_1_104_1H
VXR-400 "nmrsun4"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.730 sec
Width 6000.6 Hz
16 repetitions
OBSERVE H1, 400.1083670 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 0 min, 59 sec

5

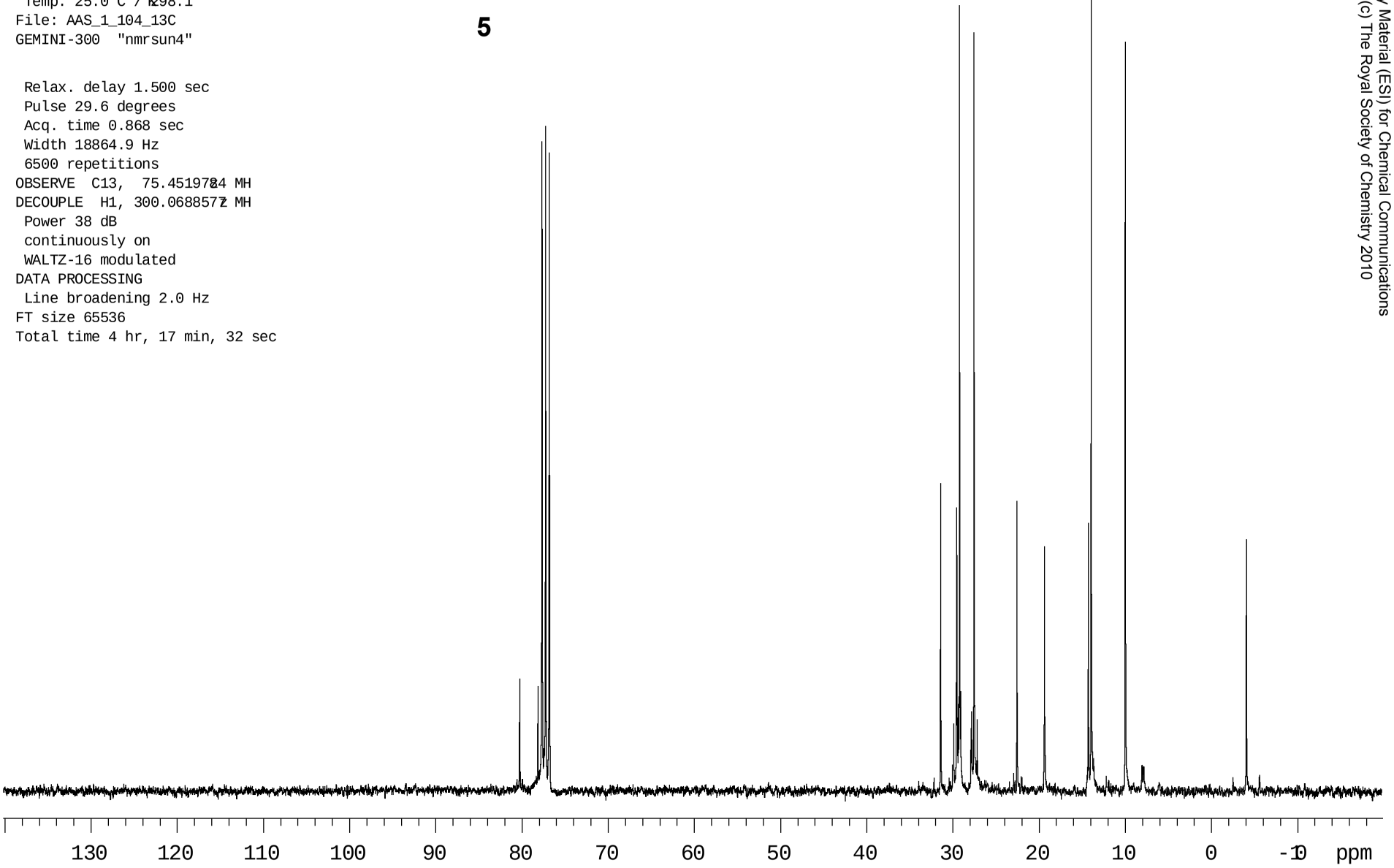
AAS.1.104.13C

Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
File: AAS_1_104_13C
GEMINI-300 "nmrsun4"

Relax. delay 1.500 sec
Pulse 29.6 degrees
Acq. time 0.868 sec
Width 18864.9 Hz
6500 repetitions
OBSERVE C13, 75.4519784 MHz
DECOUPLE H1, 300.0688577 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 4 hr, 17 min, 32 sec

5



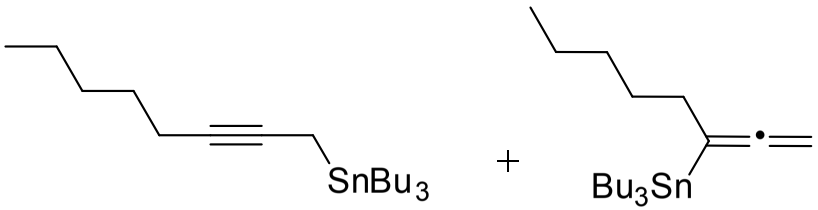
AAS.1.146.1H

Archive directory:
Sample directory:

Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / K298.1
File: AAS_1_146_1H
VXR-400 "nmrsun4"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.730 sec
Width 6000.6 Hz
16 repetitions
OBSERVE H1, 400.10836[illegible]3 MH
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 0 min, 59 sec



5

+

6

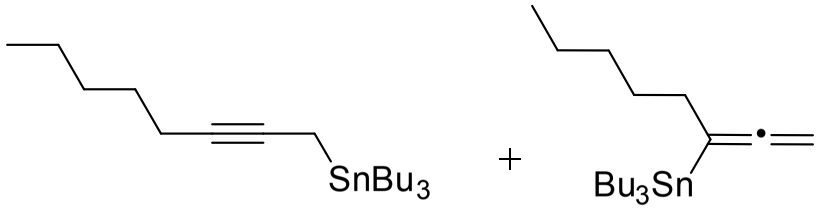
AAS.2.184.13C

Archive directory:
Sample directory:

Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
User: 1-14-87
File: AAS-2-184-13C
VXR-400 "nmrsun4"

Relax. delay 3.000 sec
Pulse 30.0 degrees
Acq. time 1.303 sec
Width 25141.4 Hz
10000 repetitions
OBSERVE C13, 100.5612515 MH
DECOUPLE H1, 399.9272266 MH
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 11 hr, 58 min, 42 sec



5

+

6

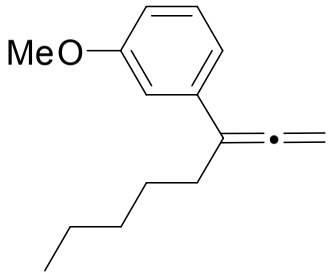
AAS.1.150.1H

Archive directory:
Sample directory:

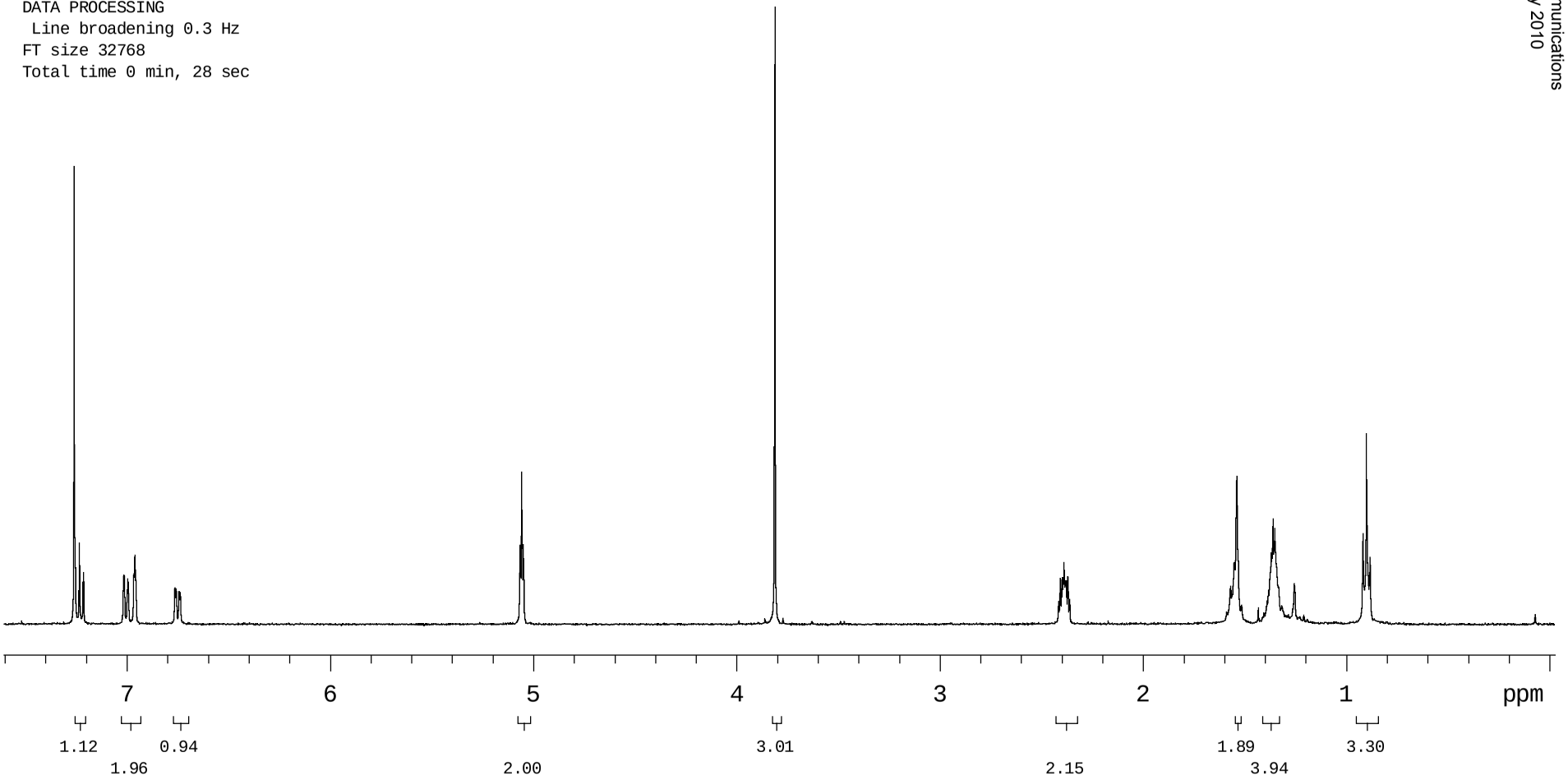
Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
File: AAS.1.150.1H
VXR-400 "nmrsun4"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.560 sec
Width 6399.0 Hz
8 repetitions
OBSERVE H1, 399.9252194 MH
DATA PROCESSING
Line broadening 0.3 Hz
FT size 32768
Total time 0 min, 28 sec



7

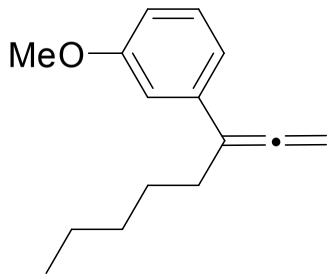


AAS.1.150.13C

Archive directory:
Sample directory:

Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
User: 1-14-87
File: AAS.1.150.13C
VXR-400 "nmrsun4"



7

Relax. delay 3.000 sec
Pulse 45.0 degrees
Acq. time 1.303 sec
Width 25141.4 Hz
9400 repetitions
OBSERVE C13, 100.5612523 MH
DECOUPLE H1, 399.9272260 MH
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 11 hr, 15 min, 35 sec

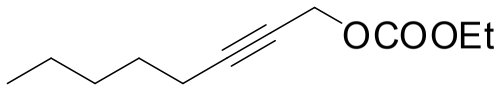
AAS.1.92.1H.

Archive directory:
Sample directory:

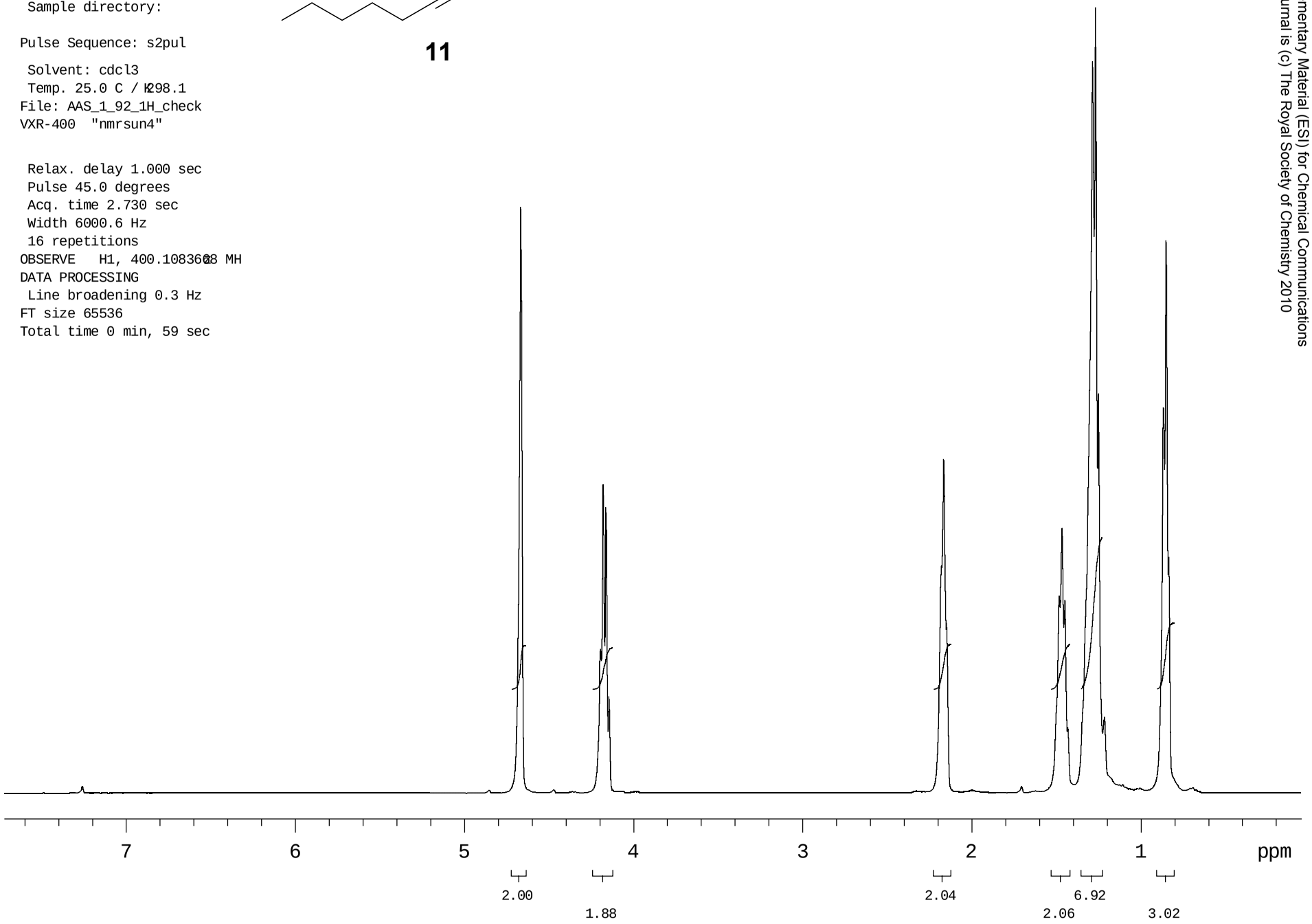
Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
File: AAS_1_92_1H_check
VXR-400 "nmrsun4"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.730 sec
Width 6000.6 Hz
16 repetitions
OBSERVE H1, 400.1083608 MHz
DATA PROCESSING
Line broadening 0.3 Hz
FT size 65536
Total time 0 min, 59 sec



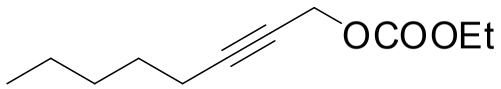
11



AAS.1.92.13C

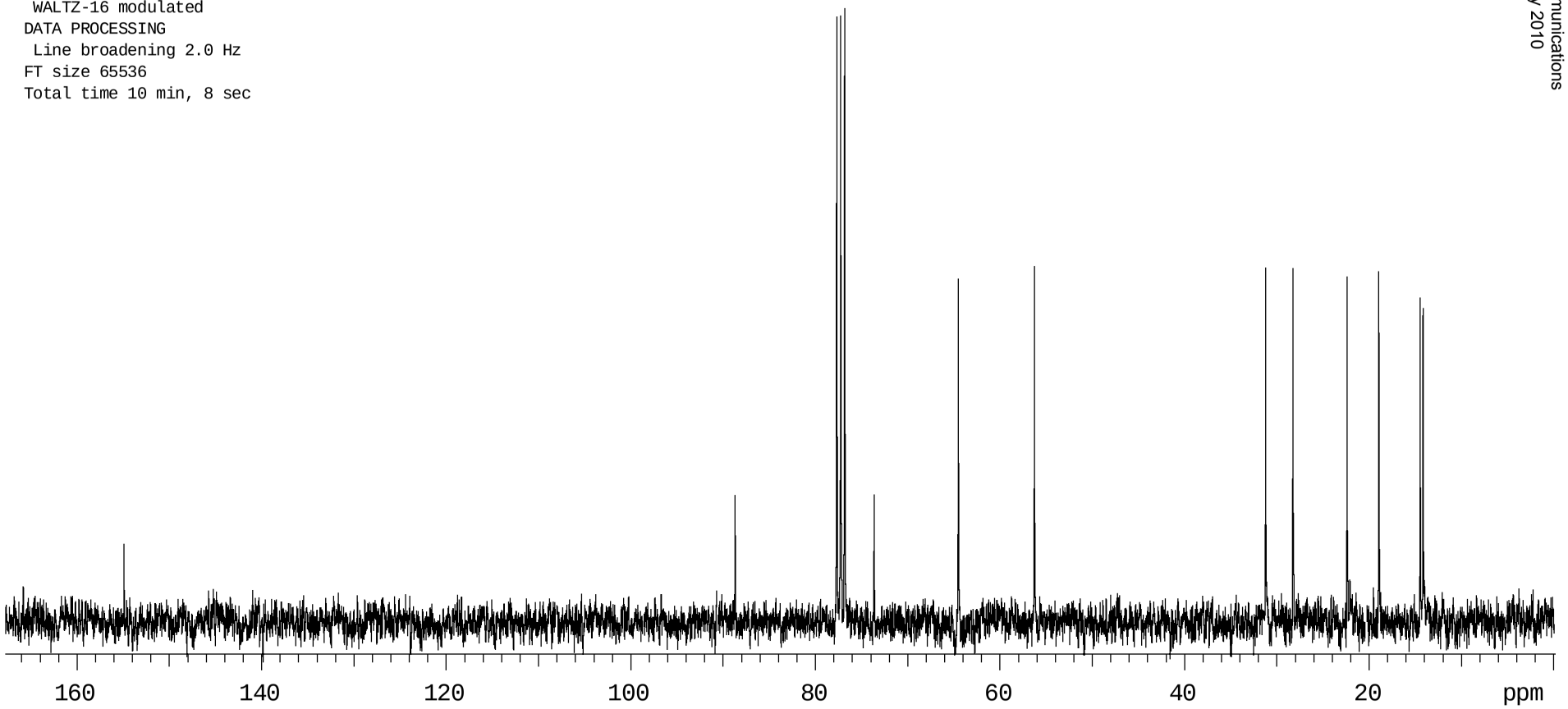
Pulse Sequence: s2pul

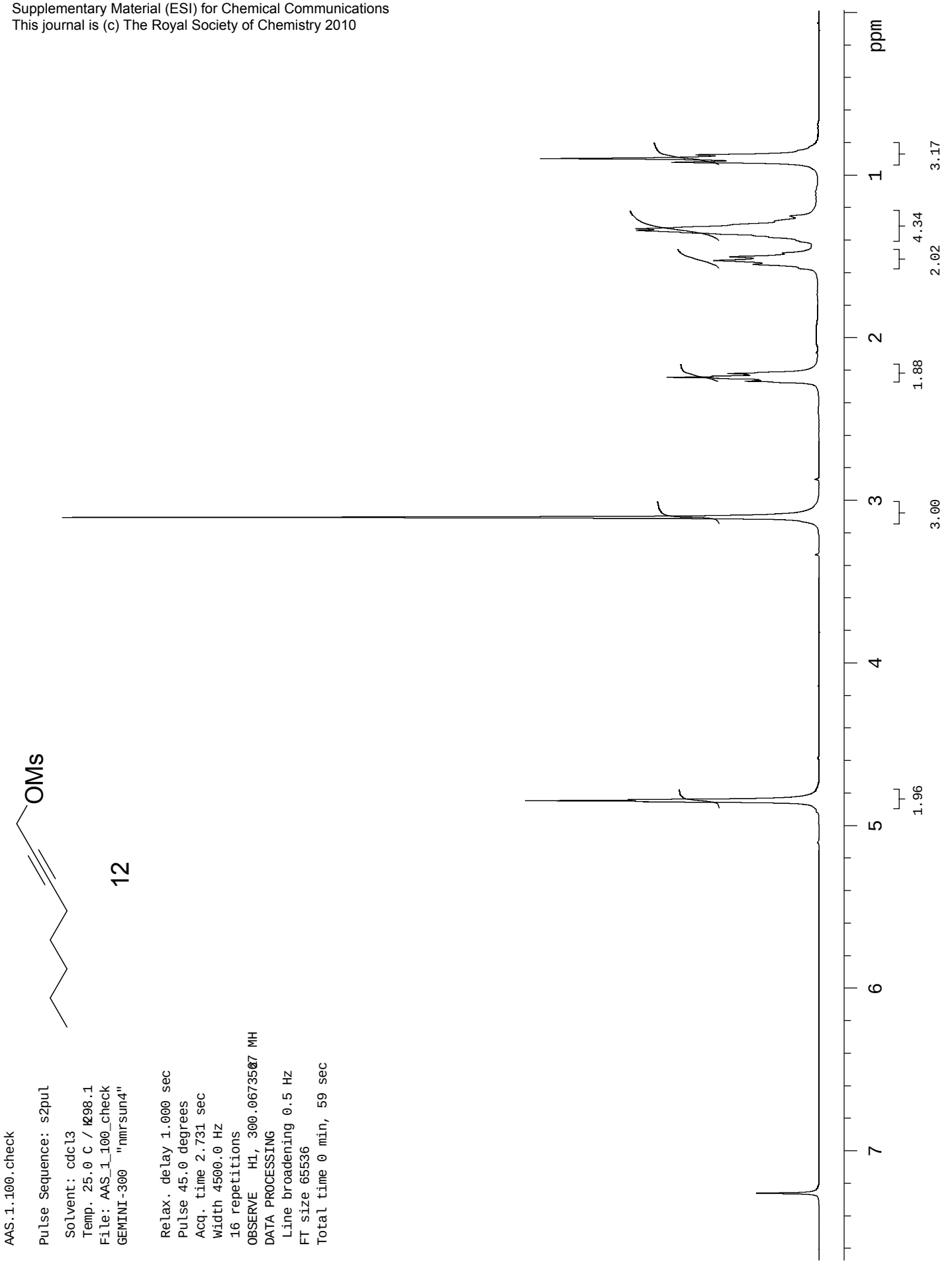
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
File: AAS_1_92_13C
GEMINI-300 "nmrsun4"



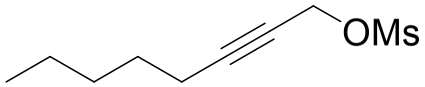
11

Relax. delay 1.500 sec
Pulse 29.6 degrees
Acq. time 0.868 sec
Width 18864.9 Hz
226 repetitions
OBSERVE C13, 75.4519729 MHz
DECOUPLE H1, 300.0688577 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 10 min, 8 sec




AAS.1.100.check
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 25.0 C / 298.1
File: AAS_1_100_check
GEMINI-300 "nmrsun4"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 2.731 sec
Width 4500.0 Hz
16 repetitions
OBSERVE H1, 300.0673587 MH
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 0 min, 59 sec
OMs
12
ppm
1.96
3.00
1.88
4.34
2.02
3.17

AAS.1.100.13C



12

Pulse Sequence: s2pul

Solvent: cdcl3
Temp. 25.0 C / 298.1 K
File: AAS_1_100_again_13C
GEMINI-300 "nmrsun4"

Relax. delay 1.500 sec
Pulse 29.6 degrees
Acq. time 0.868 sec
Width 18864.9 Hz
60 repetitions
OBSERVE C13, 75.4519803 MHz
DECOUPLE H1, 300.0688577 MHz
Power 38 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 2 min, 22 sec