

Carbocyclization of Unsaturated Thioesters Under Palladium Catalysis

Supplementary Information
(26 pages)

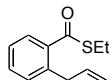
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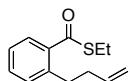
General. All reagents were obtained commercially unless otherwise noted. Copper(I)-thiophene-2-carboxylate (CuTC) was prepared according to the procedure described by Liebeskind.¹ Thioesters were prepared from the corresponding carboxylic acids under standard conditions.² Reactions were performed using glassware that was flame-dried under vacuum (~1 Torr). Air- and moisture-sensitive liquids and solutions were transferred via syringe or stainless steel cannula. Organic solutions were concentrated under reduced pressure (~15 Torr) by rotary evaporation. Solvents were purified by passage under 12 psi N₂ through activated alumina columns. Chromatography was performed on Silicycle Silia-P Silica Gel (40-63 μ m). Compounds purified by chromatography were typically applied to the adsorbent bed using the indicated solvent conditions with a minimum amount of additional benzene as needed for solubility. Thin layer chromatography was performed on Whatman Partisil K6F Silica Gel 60 \AA plates (250 μ m). Visualization of the developed chromatogram was accomplished by fluorescence quenching and by staining with aqueous potassium permanganate.

Nuclear magnetic resonance (NMR) spectra were acquired on a Varian Inova-300 operating at 300 and 75 MHz, a Varian Mercury-400 operating at 400 and 100 MHz, or a Varian Inova-500 operating at 500 and 125 MHz for ¹H and ¹³C, respectively, and are referenced internally according to residual solvent signals. Data for ¹H NMR are recorded as follows: chemical shift (δ , ppm), multiplicity (s, singlet; d, doublet; t, triplet; q, quartet; quin, quintet; m, multiplet), integration, coupling constant (Hz). Data for ¹³C NMR are reported in terms of chemical shift (δ , ppm). Infrared spectra were recorded on a ThermoNicolet IR300 spectrometer as thin films using NaCl salt plates and are reported in frequency of absorption. High-resolution mass spectra were obtained from the Vincent Coates Foundation Mass Spectrometry Laboratory at Stanford University.

General procedure for substrate preparation. Ethanethiol (0.45 mL, 6.20 mmol, 5.0 equiv) was added to a solution of carboxylic acid (1.24 mmol), *N*-(3-dimethylaminopropyl)-*N'*-ethylcarbodiimide hydrochloride (284 mg, 1.49 mmol, 1.20 equiv), 4-(dimethylamino)pyridine (15 mg, 0.12 mmol, 10 mol%) in 12.4 mL of CH₂Cl₂. The mixture was stirred until the carboxylic acid was completely consumed, as determined by TLC (1–2 h). The reaction was then quenched with 15 mL of saturated aqueous NaHCO₃. The mixture was transferred to a separatory funnel and the aqueous layer was extracted 3 x 20 mL CH₂Cl₂. The combined organic layers were washed with 25 mL of saturated aqueous sodium chloride, dried over MgSO₄, filtered, and concentrated under reduced pressure. The desired product was isolated following purification by chromatography on silica gel (conditions given below).



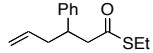
Purified by chromatography on silica gel (9:1 hexanes/EtOAc); yellow oil (80%); TLC R_f = 0.81 (3:1 hexanes/EtOAc); ¹H NMR (CDCl₃, 500 MHz) δ 7.73 (dd, 1H, J = 8.5, 1.5 Hz), 7.42 (dt, 1H, J = 7.5, 1.5 Hz), 7.29–7.25 (m, 2H), 5.97 (ddt, 1H, J = 17.0, 10.0, 6.5 Hz), 5.06–4.99 (m, 2H), 3.60 (d, 2H, J = 6.5 Hz), 3.03 (q, 2H, J = 7.5 Hz), 1.35 (t, 3H, J = 7.5 Hz) ppm; ¹³C NMR (CDCl₃, 100 MHz) δ 194.8, 138.3, 137.9, 137.0, 131.7, 130.8, 128.5, 126.3, 116.0, 37.6, 24.2, 14.8 ppm; IR (thin film) ν 2973, 2930, 1668, 1638, 1447, 1210, 914 cm⁻¹; HRMS (ES+) calcd for C₁₂H₁₅OS 207.0836 found 207.0838 (M+H⁺).



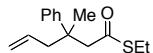
Purified by chromatography on silica gel (9:1 hexanes/EtOAc); yellow oil (91%); TLC R_f = 0.58 (9:1 hexanes/EtOAc); ¹H NMR (CDCl₃, 400 MHz) δ 7.71 (dd, 1H, J = 8.0, 1.2 Hz), 7.40 (dt, 1H, J = 7.6, 1.2 Hz), 7.28–7.22 (m, 2H), 5.85 (ddt, 1H, J = 16.8, 10.0, 6.8 Hz), 5.07–4.94 (m, 2H), 3.04 (q, 2H, J = 7.2 Hz), 2.90 (t, 2H, J = 8.0 Hz), 2.39–2.32 (m, 2H), 1.36 (t, 3H, J = 7.2 Hz) ppm; ¹³C NMR (CDCl₃, 100 MHz) δ 194.9, 140.3, 138.1, 138.0, 131.4, 130.8, 128.5, 126.0, 115.0, 35.6, 33.0, 24.2, 14.8 ppm; IR (thin film) ν 2970, 2930, 1668, 1641, 1453, 1265, 1207, 909 cm⁻¹; HRMS (ES⁺) calcd for C₁₃H₁₇OS 221.0992 found 221.0995 (M+H⁺).

(1) Allred, G. D.; Liebeskind, L. S. *J. Am. Chem. Soc.* **1996**, *118*, 2748–2749.

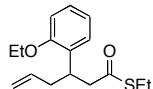
(2) Mo, S. J., et. al. *J. Am. Chem. Soc.* **2011**, *133*, 976–985.



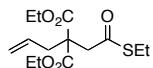
Purified by chromatography on silica gel (9:1 hexanes/EtOAc); yellow oil (87%); TLC R_f = 0.67 (3:1 hexanes/EtOAc); ^1H NMR (CDCl_3 , 400 MHz) δ 7.31-7.26 (m, 2H), 7.23-7.15 (m, 3H), 5.64 (ddt, 1H, J = 17.6, 10.4, 7.2 Hz), 5.04-4.94 (m, 2H), 3.28 (tt, 1H, J = 14.8, 7.2 Hz), 2.90 (dd, 1H, J = 14.8, 6.8 Hz), 2.84-2.76 (m, 3H), 2.43-2.37 (m, 2H), 1.16 (t, 3H, J = 7.2 Hz) ppm; ^{13}C NMR (CDCl_3 , 100 MHz) δ 198.4, 143.2, 135.9, 128.5, 127.6, 126.7, 117.1, 49.5, 42.3, 40.4, 23.4, 14.8 ppm; IR (thin film) ν 2973, 2930, 1688, 1641, 1453, 1265, 1054 cm^{-1} ; HRMS (ES $^+$) calcd for $\text{C}_{14}\text{H}_{19}\text{OS}$ 235.1150 found 235.1151 ($\text{M}+\text{H}^+$).



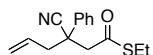
Purified by chromatography on silica gel (99:1 hexanes/EtOAc); yellow oil (94%); TLC R_f = 0.26 (19:1 hexanes/EtOAc); ^1H NMR (CDCl_3 , 400 MHz) δ 7.33-7.31 (m, 4H), 7.23-7.16 (m, 1H), 5.56-5.45 (m, 1H), 5.05-4.96 (m, 2H), 2.95 (d, 1H, J = 14.4 Hz), 2.82 (d, 1H, J = 14.4 Hz), 2.74 (q, 2H, J = 7.6 Hz), 2.60 (dd, 1H, J = 14.0, 6.8 Hz), 2.46 (dd, 1H, J = 14.0, 8.0 Hz), 1.47 (s, 3H), 1.13 (t, 3H, J = 7.6 Hz) ppm; ^{13}C NMR (CDCl_3 , 100 MHz) δ 197.4, 146.0, 134.4, 128.2, 126.20, 126.16, 118.2, 55.4, 46.9, 41.1, 24.6, 23.5, 14.8 ppm; IR (thin film) ν 2970, 2930, 1688, 1639, 1604, 1446, 1374, 1012 cm^{-1} ; HRMS (ES $^+$) calcd for $\text{C}_{15}\text{H}_{21}\text{OS}$ 249.1307 found 249.1308 ($\text{M}+\text{H}^+$).



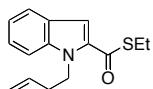
Purified by chromatography on silica gel (49:1 hexanes/EtOAc); yellow oil (90%); TLC R_f = 0.15 (49:1 hexanes/EtOAc); ^1H NMR (CDCl_3 , 400 MHz) δ 7.16 (dt, 1H, J = 7.6, 1.6 Hz), 7.09 (dd, 1H, J = 7.2, 1.6 Hz), 6.87 (dt, 1H, J = 7.4, 1.2 Hz), 6.83 (dd, 1H, J = 8.0, 0.8 Hz), 5.66 (ddt, 1H, J = 17.4, 10.0, 6.4 Hz), 5.03-4.91 (m, 2H), 4.04 (q, 2H, J = 6.8 Hz), 3.64 (dt, 1H, J = 14.4, 7.2 Hz), 2.96 (dd, 1H, J = 15.2, 7.2 Hz), 2.89 (dd, 1H, J = 15.2, 7.6 Hz), 2.80 (q, 2H, J = 7.2 Hz), 2.52-2.39 (m, 2H), 1.49 (t, 3H, J = 6.8 Hz), 1.16 (t, 3H, J = 7.6 Hz) ppm; ^{13}C NMR (CDCl_3 , 100 MHz) δ 198.8, 156.7, 136.6, 131.0, 128.5, 127.5, 120.3, 116.4, 111.6, 63.6, 48.3, 38.4, 37.0, 23.3, 15.0, 14.8 ppm; IR (thin film) ν 2978, 2930, 1689, 1494, 146, 1290, 1241, 1125, 1049, 997, 916, 752 cm^{-1} ; HRMS (ES $^+$) calcd for $\text{C}_{16}\text{H}_{23}\text{O}_2\text{S}$ 279.1412 found 279.1413 ($\text{M}+\text{H}^+$).



Purified by chromatography on silica gel (19:1 hexanes/EtOAc); yellow oil (21%); TLC R_f = 0.42 (9:1 hexanes/EtOAc); ^1H NMR (CDCl_3 , 400 MHz) δ 5.72-5.59 (m, 1H), 5.16-5.08 (m, 2H), 4.20 (q, 4H, J = 6.8 Hz), 3.20 (s, 2H), 2.86 (q, 2H, J = 7.2 Hz), 2.74 (d, 2H, 7.6 Hz), 1.28-1.20 (m, 9H) ppm; ^{13}C NMR (CDCl_3 , 100 MHz) δ 196.3, 169.9, 132.2, 120.1, 61.9, 55.9, 45.8, 37.4, 23.6, 14.8, 14.1 ppm; IR (thin film) ν 2982, 1737, 1690, 1287, 1214, 1192 cm^{-1} ; HRMS (ES $^+$) calcd for $\text{C}_{14}\text{H}_{22}\text{O}_5\text{SNa}^+$ 325.1079 found 325.1080 (MNa^+).

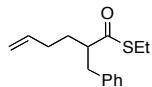


Purified by chromatography on silica gel (19:1 hexanes/EtOAc); yellow oil (17%); TLC R_f = 0.27 (9:1 hexanes/EtOAc); ^1H NMR (CDCl_3 , 400 MHz) δ 7.47-7.29 (m, 5H), 5.73-5.60 (m, 1H), 5.23-5.16 (m, 2H), 3.23 (d, 1H, J = 15.6 Hz), 3.17 (d, 1H, J = 15.6 Hz), 2.86-2.71 (m, 4H), 1.15 (t, 3H, J = 7.6 Hz) ppm; ^{13}C NMR (CDCl_3 , 100 MHz) δ 194.2, 136.9, 131.1, 129.0, 128.3, 126.1, 121.1, 94.5, 51.4, 44.9, 44.6, 23.4, 14.6 ppm; IR (thin film) ν 2929, 2359, 1688, 1495, 1449, 1265, 993 cm^{-1} ; HRMS (ES $^+$) calcd for $\text{C}_{15}\text{H}_{18}\text{ONs}$ 260.1109 found 260.1104 ($\text{M}+\text{H}^+$).

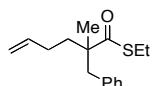


Purified by chromatography on silica gel (19:1 hexanes/EtOAc); yellow oil (12%); TLC R_f = 0.46 (9:1 hexanes/EtOAc); ^1H NMR (CDCl_3 , 400 MHz) δ 7.68 (d, 1H, J = 8.0 Hz), 7.45-7.32 (m, 3H), 7.16 (t, 1H, J = 7.4

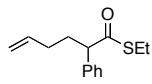
Hz), 5.82 (ddt, 1H, J = 16.8, 10.4, 6.8 Hz), 5.11-4.99 (m, 2H), 4.57 (t, 2H, J = 6.8 Hz), 3.07 (q, 2H, J = 7.2 Hz), 2.54 (dt, 2H, J = 7.6, 6.8 Hz), 1.37 (t, 3H, J = 6.8 Hz) ppm; ^{13}C NMR (CDCl_3 , 100 MHz) δ 185.0, 139.2, 134.7, 133.9, 126.1, 125.6, 122.9, 120.9, 117.3, 110.6, 110.5, 44.5, 34.9, 23.4, 15.0 ppm; IR (thin film) ν 2929, 1649, 1614, 1511, 1461, 1476, 1354, 1154, 1132, 942 cm^{-1} ; HRMS (ES $^+$) calcd for $\text{C}_{15}\text{H}_{18}\text{ONS}$ 260.1109 found 260.1104 ($\text{M}+\text{H}^+$).



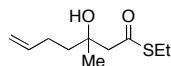
Purified by chromatography on silica gel (9:1 hexanes/EtOAc); yellow oil (86%); TLC R_f = 0.86 (3:1 hexanes/EtOAc); ^1H NMR (CDCl_3 , 400 MHz) δ 7.30-7.12 (m, 5H), 5.73 (ddt, 1H, J = 16.8, 10.4, 6.4 Hz), 5.05-4.94 (m, 2H), 2.98 (dd, 1H, J = 13.2, 7.6 Hz), 2.90-2.77 (m, 3H), 2.72 (dd, 1H, J = 13.2, 6.8 Hz), 2.17-1.97 (m, 2H), 1.87-1.75 (m, 1H), 1.61-1.50 (m, 1H), 1.18 (t, 3H, J = 7.2 Hz) ppm; ^{13}C NMR (CDCl_3 , 100 MHz) δ 202.8, 138.9, 137.7, 129.1, 128.4, 126.4, 115.4, 55.5, 39.0, 31.4, 31.3, 23.2, 14.8 ppm; IR (thin film) ν 3028, 2929, 2856, 1683, 1641, 1453, 1265, 946, 915 cm^{-1} ;



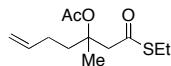
Purified by chromatography on silica gel (19:1 hexanes/EtOAc); yellow oil (53%); TLC R_f = 0.49 (9:1 hexanes/EtOAc); ^1H NMR (CDCl_3 , 400 MHz) δ 7.29-7.20 (m, 3H), 7.15-7.11 (m, 2H), 5.80 (ddt, 1H, J = 17.2, 10.0, 6.4 Hz), 5.07-4.94 (m, 2H), 3.08 (d, 1H, J = 13.2 Hz), 2.88 (q, 2H, J = 7.2 Hz), 2.76 (d, 1H, J = 13.6 Hz), 2.13-1.98 (m, 2H), 1.91 (ddd, 1H, J = 13.6, 11.2, 6.8 Hz), 1.53 (ddd, 1H, J = 13.6, 10.8, 6.0 Hz), 1.26 (t, 3H, J = 7.2 Hz), 1.20 (s, 3H) ppm; ^{13}C NMR (CDCl_3 , 100 MHz) δ 205.8, 128.3, 137.1, 130.5, 128.0, 126.5, 114.7, 53.8, 45.8, 38.9, 28.8, 23.2, 20.6, 14.7 ppm; IR (thin film) ν 2973, 2930, 1671, 1641, 1454, 954, 913, 704 cm^{-1} ; HRMS (ES $^+$) calcd for $\text{C}_{16}\text{H}_{23}\text{OS}$ 263.1469 found 263.1464 ($\text{M}+\text{H}^+$).



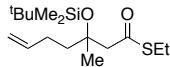
Purified by chromatography on silica gel (3:1 hexanes/EtOAc); clear oil (59%); TLC R_f = 0.74 (3:1 hexanes/EtOAc); ^1H NMR (CDCl_3 , 400 MHz) δ 7.31-7.19 (m, 5H), 5.72 (ddt, 1H, J = 16.8, 10.4, 6.4 Hz), 4.99-4.92 (m, 2H), 3.70 (t, 1H, J = 7.2 Hz), 2.86-2.70 (m, 2H), 2.24-2.14 (m, 1H), 1.96 (m, 2H), 1.91-1.81 (m, 1H), 1.15 (t, 3H, J = 7.2 Hz) ppm; ^{13}C NMR (CDCl_3 , 100 MHz) δ 200.5, 138.4, 137.5, 128.7, 128.3, 127.5, 115.6, 59.5, 32.3, 31.4, 23.6, 14.6 ppm; IR (thin film) ν 2930, 1686, 1642, 1453, 1265, 996 cm^{-1} ; HRMS (ES $^+$) calcd for $\text{C}_{14}\text{H}_{19}\text{OS}$ 235.1156 found 235.1151 ($\text{M}+\text{H}^+$).



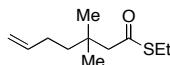
Purified by chromatography on silica gel (19:1 hexanes/EtOAc); yellow oil (25%); TLC R_f = 0.56 (3:1 hexanes/EtOAc); ^1H NMR (CDCl_3 , 400 MHz) δ 5.82 (ddt, 1H, J = 16.8, 10.0, 6.8 Hz), 5.07-5.00 (m, 1H), 4.97-4.92 (m, 1H), 3.35 (s, 1H), 2.91 (q, 2H, J = 7.6 Hz), 2.72 (q, 2H, J = 12.4 Hz), 2.19-2.11 (m, 2H), 1.63-1.56 (m, 2H), 1.27 (t, 3H, J = 7.6 Hz), 1.24 (s, 3H) ppm; ^{13}C NMR (CDCl_3 , 100 MHz) δ 200.5, 138.5, 114.6, 72.2, 53.8, 41.0, 28.3, 26.6, 23.6, 14.7 ppm; IR (thin film) ν 3519, 2972, 2931, 1671, 1642, 1454, 1377, 1126, 1013, 912 cm^{-1} .



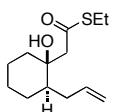
Purified by chromatography on silica gel (9:1 hexanes/EtOAc); yellow oil (83%); TLC R_f = 0.81 (3:1 hexanes/EtOAc); ^1H NMR (CDCl_3 , 400 MHz) δ 5.84-5.71 (m, 1H), 5.06-4.97 (m, 1H), 4.97-4.91 (m, 1H), 3.2 (d, 1H, J = 14.0 Hz), 3.06 (d, 1H, J = 14.4 Hz), 2.84 (q, 2H, J = 7.6 Hz), 2.13-2.04 (m, 3H), 1.99 (s, 3H), 1.85-1.75 (m, 1H), 1.49 (s, 3H), 1.22 (t, 3H, J = 7.6 Hz) ppm; ^{13}C NMR (CDCl_3 , 100 MHz) δ 195.9, 170.6, 137.9, 114.9, 81.6, 50.9, 38.0, 27.9, 24.1, 23.6, 22.4, 14.7 ppm; IR (thin film) ν 3078, 2977, 2933, 1737, 1689, 1642, 1452, 1368, 1245, 1131, 1016, 913 cm^{-1} .



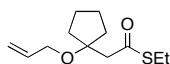
Purified by chromatography on silica gel (gradient elution: 99:1 → 19:1 hexanes/EtOAc); yellow oil (47%); TLC R_f = 0.24 (19:1 hexanes/EtOAc); ^1H NMR (CDCl_3 , 400 MHz) δ 5.81 (ddt, 1H, J = 16.8, 10.4, 6.8 Hz), 5.01 (dq, 1H, J = 16.8, 1.6 Hz), 4.93 (dq, 1H, J = 10.0 Hz, 1.2 Hz), 2.84 (q, 2H, J = 7.6 Hz), 2.70 (s, 2H), 2.24-2.06 (m, 2H), 1.69-1.61 (m, 2H), 1.36 (s, 3H), 1.23 (t, 3H, J = 7.6 Hz), 0.86 (s, 9H), 0.09 (s, 6H) ppm; ^{13}C NMR (CDCl_3 , 100 MHz) δ 197.0, 138.8, 114.3, 74.9, 55.5, 42.0, 28.6, 28.2, 25.9, 23.6, 18.3, 14.7, -1.9 ppm; IR (thin film) ν 2931, 2956, 2857, 1691, 1642, 1461, 1377, 1254, 1082, 836 cm^{-1} .



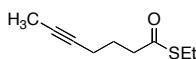
Purified by chromatography on silica gel (9:1 hexanes/EtOAc); yellow oil (82%); TLC R_f = 0.81 (3:1 hexanes/EtOAc); ^1H NMR (CDCl_3 , 400 MHz) δ 5.78 (ddt, 1H, J = 16.8, 10.4, 6.8 Hz), 5.03-4.88 (m, 2H), 2.83 (q, 2H, J = 7.2 Hz), 2.42 (s, 2H), 2.07-1.81 (m, 2H), 1.42-1.36 (m, 2H), 1.22 (t, 3H, J = 6.8 Hz), 0.99 (s, 6H) ppm; ^{13}C NMR (CDCl_3 , 100 MHz) δ 198.2, 139.1, 114.2, 54.8, 41.4, 34.2, 28.6, 27.4, 23.6, 14.8 ppm; IR (thin film) 2980, 1688, 1641, 1600, 1454, 1012 cm^{-1} .



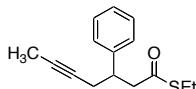
Purified by chromatography on silica gel (19:1 hexanes/EtOAc); yellow oil (29%); TLC R_f = 0.32 (9:1 hexanes/EtOAc); ^1H NMR (CDCl_3 , 400 MHz) δ 5.81-5.69 (m, 1H), 5.05-4.95 (m, 2H), 2.98-2.92 (m, 2H), 2.88 (q, 2H, J = 7.6 Hz), 2.63 (d, 1H, 14.8 Hz), 2.40-2.32 (m, 1H), 2.02-1.90 (m, 1H), 1.75-1.53 (m, 4H), 1.50-1.32 (m, 4H), 1.25 (t, 3H, 7.2 Hz) ppm; ^{13}C NMR (CDCl_3 , 100 MHz) δ 200.8, 138.0, 115.9, 73.5, 53.2, 44.5, 36.9, 34.0, 26.8, 25.0, 23.6, 21.7, 14.7 ppm; IR (thin film) ν 3524, 2932, 2860, 1668, 1446, 1391, 1302, 1078, 1014, 910 cm^{-1} .



Purified by chromatography on silica gel (9:1 hexanes/EtOAc); yellow oil (31%); TLC R_f = 0.80 (3:1 hexanes/EtOAc); ^1H NMR (CDCl_3 , 400 MHz) δ 5.96-5.85 (m, 1H), 5.27 (dq, 1H, J = 17.2, 1.6 Hz), 5.11 (dq, 1H, J = 10.4, 1.6 Hz), 3.94 (t, 1H, J = 1.4 Hz), 3.92 (t, 1H, J = 1.4 Hz), 2.90-2.83 (m, 4H), 1.96-1.55 (m, 8H), 1.24 (t, 3H, J = 7.6 Hz) ppm; ^{13}C NMR (CDCl_3 , 100 MHz) δ 196.7, 135.5, 116.2, 85.6, 63.9, 50.0, 36.4, 23.7, 23.4, 14.7 ppm; IR (thin film) ν 2961, 2872, 1690, 1452, 1377, 1254, 1082, 836 cm^{-1} .

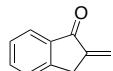


Purified by chromatography on silica gel (9:1 hexanes/EtOAc); yellow oil (67%); TLC R_f = 0.79 (3:1 hexanes/EtOAc); ^1H NMR (CDCl_3 , 400 MHz) δ 2.87 (dq, 2H, J = 9.6, 2.0 Hz), 2.65 (dt, 2H, 10.0, 1.6 Hz), 2.23-2.13 (m, 2H), 1.88-1.74 (m, 5H), 1.24 (dt, 3H, J = 9.6, 1.6 Hz) ppm; ^{13}C NMR (CDCl_3 , 75 MHz) δ 199.2, 77.8, 76.6, 42.9, 24.8, 23.3, 18.1, 14.8, 3.5 ppm; IR (thin film) ν 2962, 1931, 1689, 1452, 1414, 1266, 1074 cm^{-1} ; HRMS (ES⁺) calcd for $\text{C}_{15}\text{H}_{18}\text{OS}$ 171.0843 found 171.0838 ($\text{M}+\text{H}^+$).

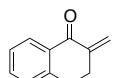


Purified by chromatography on silica gel (7:3 hexanes/ CH_2Cl_2); yellow oil (74%); TLC R_f = 0.79 (7:3 hexanes/ CH_2Cl_2); ^1H NMR (CDCl_3 , 400 MHz) δ 7.33-7.27 (m, 2H), 7.24-7.19 (m, 3H), 3.38 (tt, 1H, J = 14.0, 6.4 Hz), 3.14 (dd, 1H, J = 15.2, 6.4 Hz), 2.89 (dd, 1H, J = 15.2, 8.4 Hz), 2.80 (q, 2H, J = 7.2 Hz), 2.54-2.37 (m, 2H), 1.76 (t, 3H, J = 2.6 Hz), 1.16 (t, 3H, J = 7.2 Hz) ppm; ^{13}C NMR (CDCl_3 , 125 MHz) δ 198.2, 142.7, 128.5, 127.4, 126.9, 78.1, 76.5, 48.9, 41.6, 26.1, 23.4, 14.8, 3.6 ppm; IR (thin film) ν 2968, 2930, 1688, 1454, 1430, 1265, 1063 cm^{-1} ; HRMS (ES⁺) calcd for $\text{C}_{15}\text{H}_{19}\text{OS}$ 247.1156 found 247.1151 ($\text{M}+\text{H}^+$).

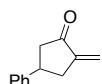
General procedure for carbocyclization of thioesters. Thioester (0.20 mmol), $\text{Pd}(\text{OAc})_2$ (2.2 mg, 0.01 mmol, 5 mol%), Cu(thiophene-2-carboxylate) (61.0 mg, 0.32 mmol, 1.6 equiv), and $\text{Zn}(\text{O}_2\text{CH})_2$ (31.1 mg, 0.20 mmol, 1.0 equiv) were added sequentially to a flame-dried 10 mL round bottom flask. The flask was placed under vacuum and back-filled with N_2 . THF (1.7 mL), which had been degassed by a freeze-pump-thaw cycle (3x), was added, followed immediately by $\text{P}(\text{OMe})_3$ (24 μL , 0.20 mmol, 1.0 equiv). The brown suspension was stirred at 60 °C for 18 h, after which time the reaction was quenched with 6 mL of saturated aqueous NaHCO_3 . The mixture was transferred to a separatory funnel and extracted with 3 x 15 mL of EtOAc. The combined organic layers were washed with 1 x 20 mL of saturated aqueous sodium chloride, dried over MgSO_4 , filtered through a pad of Celite, and concentrated under reduced pressure to a brown residue. The desired product was isolated following purification by chromatography on silica gel (conditions given below).



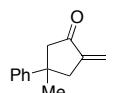
Characterization data matched that reported in literature.³



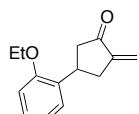
Characterization data matches that reported in literature.⁴



Characterization data matches that reported in literature.⁵



Purified by chromatography on silica gel (19:1 hexanes/EtOAc); yellow oil (83%); TLC R_f = 0.34 (9:1 hexanes/EtOAc); ^1H NMR (CDCl_3 , 400 MHz) δ 7.39-7.21 (m, 5H), 6.12-6.08 (m, 1H), 5.44-5.39 (m, 1H), 3.05-2.92 (m, 2H), 2.76 (d, 1H, J = 17.6 Hz), 2.64 (dd, 1H, J = 17.2, 1.6 Hz), 1.34 (d, 3H, J = 1.3 Hz) ppm; ^{13}C NMR (CDCl_3 , 100 MHz) δ 205.4, 148.2, 144.2, 128.7, 126.5, 125.5, 118.4, 52.2, 43.7, 41.1, 30.1 ppm; IR (thin film) ν 2956, 2923, 1729, 1645, 14967, 1445, 1408, 1268 cm^{-1} ; HRMS (ES $^+$) calcd for $\text{C}_{13}\text{H}_{15}\text{O}$ 187.1123 found 187.1117 ($\text{M}+\text{H}^+$).

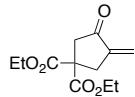


Purified by chromatography on silica gel (99:1 hexanes/EtOAc); yellow oil (80%); TLC R_f = 0.16 (19:1 hexanes/EtOAc); ^1H NMR (CDCl_3 , 400 MHz) δ 7.25-7.13 (m, 2H), 6.96-6.84 (m, 2H), 6.09-6.02 (m, 1H), 5.39-5.33 (m, 1H), 4.05 (q, 2H, J = 6.8 Hz), 3.74-3.62 (m, 1H), 3.07 (dd, 1H, J = 16.2, 7.6 Hz), 2.86-2.79 (m, 1H), 2.76 (dd, 1H, J = 18.0, 8.0 Hz), 2.59 (dd, 1H, J = 18.0, 9.6 Hz), 1.41 (t, 3H, J = 7.2 Hz) ppm; ^{13}C NMR (CDCl_3 , 100 MHz) δ 206.7, 156.8, 145.2, 131.4, 127.8, 127.0, 120.4, 117.0, 111.4, 63.4, 44.6, 36.1, 34.0, 14.9 ppm; IR (thin film) ν 2979, 1727, 1642, 1494, 1242, 1118, 1046, 752 cm^{-1} ; HRMS (ES $^+$) calcd for $\text{C}_{14}\text{H}_{17}\text{O}_2$ 217.1228 found 217.1223 ($\text{M}+\text{H}^+$).

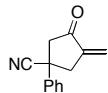
(3) Crich, D.; Chen, C.; Hwang, J-T.; Yuan, H.; Papadatos, A.; Walter, R. I. *J. Am. Chem. Soc.* **1994**, *116*, 8937-8951.

(4) Du, Y.; Lu, X.; Yu, Y. *J. Org. Chem.* **2002**, *67*, 8901-8905.

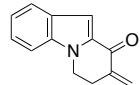
(5) Barbero, A.; García, C.; Pulido, F. J. *Tetrahedron*, **2000**, *56*, 2739-2751.



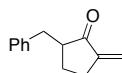
Purified by chromatography on silica gel (9:1 hexanes/EtOAc); yellow oil (89%); TLC R_f = 0.29 (3:1 hexanes/EtOAc); ¹H NMR (CDCl₃, 400 MHz) δ 6.06 (s, 1H), 5.41 (s, 1H), 4.22 (q, 4H, J = 7.2 Hz), 3.23 (s, 2H), 2.91 (s, 2H), 1.25 (t, 6H, J = 7.2 Hz) ppm; ¹³C NMR (CDCl₃, 100 MHz) δ 201.5, 170.7, 141.6, 119.0, 62.2, 54.2, 45.2, 37.0, 14.0 ppm; IR (thin film) ν 2939, 1733, 1645, 1284, 1246, 1189 cm⁻¹; HRMS (ES⁺) calcd for C₁₂H₁₇O₅ 241.1076 found 241.1071 (M+H⁺).



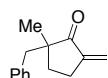
Purified by chromatography on silica gel (9:1 hexanes/EtOAc); yellow oil (94%); TLC R_f = 0.13 (9:1 hexanes/EtOAc); ¹H NMR (CDCl₃, 400 MHz) δ 7.53-7.30 (m, 5H), 6.24 (t, 1H, J = 2.4 Hz), 5.57 (t, 1H, J = 2.8 Hz), 3.53 (dt, 1H, J = 16.0, 2.8 Hz), 3.26-3.15 (m, 2H), 2.98 (d, 1H, J = 17.6 Hz) ppm; ¹³C NMR (CDCl₃, 100 MHz) δ 200.0, 140.4, 137.9, 129.5, 129.2, 129.0, 125.7, 125.2, 121.1, 50.1 43.4 ppm; IR (thin film) ν 2924, 2854, 2238, 1734, 1645, 1496, 1449, 1404, 1266, 1125 cm⁻¹; HRMS (ES⁺) calcd for C₁₃H₁₂ON 217.1228 found 396.1761, dimer of product (M+H⁺).



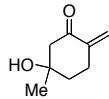
Purified by chromatography on silica gel (9:1 hexanes/EtOAc); off-white foam (30%); TLC R_f = 0.11 (9:1 hexanes/EtOAc); ¹H NMR (CDCl₃, 400 MHz) δ 7.74 (td, 1H, J = 8.0, 1.2 Hz), 7.42-7.37 (m, 3H), 7.19-7.15 (qd, 1H, J = 6.0, 2.0 Hz), 6.36-6.35 (m, 1H), 5.58-5.59 (m, 1H), 4.31 (t, 2H, J = 6.0 Hz), 3.13 (td, 1H, J = 6.4, 1.2 Hz), 3.11 (td, 1H, J = 6.4, 1.2 Hz) ppm; ¹³C NMR (CDCl₃, 100 MHz) δ 180.3, 140.4, 137.6, 134.5, 127.3, 125.9, 123.5, 123.3, 121.3, 110.4, 107.3, 41.3, 31.5 ppm; IR (thin film) ν 2922, 1671, 1621, 1524, 1477, 1326, 1162 cm⁻¹; HRMS (ES⁺) calcd for C₁₃H₁₂ON 198.0919 found 198.0913 (M+H⁺).



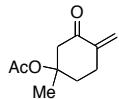
Purified by chromatography on silica gel (49:1 hexanes/EtOAc); yellow oil (50%); TLC R_f = 0.43 (9:1 hexanes/EtOAc); ¹H NMR (CDCl₃, 400 MHz) δ 7.31-7.25 (m, 3H), 7.23-7.16 (m, 2H), 6.05-6.02 (m, 1H), 5.35-5.32 (m, 1H), 3.30-3.21 (m, 1H), 2.62-2.42 (m, 4H), 2.11-2.01 (m, 1H), 1.58-1.47 (m, 1H) ppm; ¹³C NMR (100 MHz, CDCl₃) δ 207.2, 147.8, 140.0, 129.0, 128.5, 126.3, 117.7, 51.0, 35.9, 27.8, 26.4 ppm; IR (thin film) ν 2925, 2854, 1725, 1640, 1604, 1495, 1454, 1261, 1065, 941 cm⁻¹; HRMS (ES⁺) calcd for C₁₃H₁₅O 187.1123 found 187.1117 (M+H⁺).



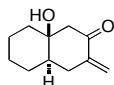
Purified by chromatography on silica gel (99:1 hexanes/EtOAc); yellow oil (33%); TLC R_f = 0.43 (9:1 hexanes/EtOAc); ¹H NMR (CDCl₃, 400 MHz) δ 7.33-7.18 (m, 3H), 7.16-7.07 (m, 2H), 6.08-6.03 (m, 1H), 5.37-5.31 (m, 1H), 2.85 (d, 1H, J = 13.6 Hz), 2.69 (d, 1H, J = 13.2 Hz), 2.60-2.39 (m, 2H), 1.64-1.53 (m, 2H), 1.06 (s, 3H) ppm; ¹³C NMR (CDCl₃, 100 MHz) δ 210.3, 144.5, 137.9, 130.4, 128.2, 126.5, 118.5, 50.1, 42.6, 31.9, 25.8, 22.8 ppm; IR (thin film) ν 2927, 1723, 1672, 1638, 1454, 1262, 1024 cm⁻¹; HRMS (ES⁺) calcd for C₁₄H₁₇O 201.1279 found 201.1274 (M+H⁺).



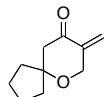
Purified by chromatography on silica gel (9:1 hexanes/EtOAc); yellow oil (73%); TLC R_f = 0.05 (9:1 hexanes/EtOAc); ^1H NMR (CDCl_3 , 400 MHz) δ 5.94-5.91 (m, 1H), 5.37 (d, 1H, J = 6.0 Hz), 4.92 (d, 1H, J = 6.0 Hz), 3.96 (dd, 1H, J = 11.6, 1.3 Hz), 3.90 (d, 1H, J = 11.6 Hz), 2.67 (ddd, 1H, J = 13.6, 4.8, 2.6 Hz), 2.62-2.50 (m, 1H), 2.42 (ddd, 1H, J = 19.6, 5.2, 2.4 Hz), 2.25-2.14 (m, 1H), 2.04 (s, 3H) ppm; ^{13}C NMR (CDCl_3 , 100 MHz) δ 200.5, 166.7, 125.9, 87.4, 65.9, 60.5, 28.0, 24.6 ppm; IR (thin film) ν 3179, 2925, 1730, 1638, 1430, 1383, 1310, 1228, 1162, 1113, 1042 cm^{-1} .



Purified by chromatography on silica gel (9:1 hexanes/EtOAc); yellow oil (37%); TLC R_f = 0.1 (9:1 hexanes/EtOAc); ^1H NMR (CDCl_3 , 400 MHz) δ 5.42 (d, 1H, J = 6.2 Hz), 4.94 (d, 1H, J = 6.2 Hz), 3.98 (dd, 1H, J = 11.6, 1.3 Hz), 3.92 (d, 1H, J = 11.6 Hz), 2.71 (ddd, 1H, J = 13.6, 4.8, 2.6 Hz), 2.71 (s, 3H) 2.62-2.52 (m, 1H), 2.58 (ddd, 1H, J = 19.6, 5.2, 2.4 Hz), 2.27-2.16 (m, 1H), 2.09 (s, 3H) ppm; ^{13}C NMR (CDCl_3 , 100 MHz) δ 200.6, 166.9, 126.1, 87.4, 65.9, 60.4, 31.1, 28.0, 25.2, 24.7 ppm; IR (thin film) ν 2972, 2928, 1739, 1733, 1638, 1444, 1380, 1312, 1228, 1170, 1040 cm^{-1} .

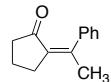


Purified by chromatography on silica gel (19:1 hexanes/EtOAc); yellow oil (81%); TLC R_f = 0.14 (9:1 hexanes/EtOAc); ^1H NMR (CDCl_3 , 400 MHz) δ 5.89-5.86 (m, 1H), 5.22-5.19 (m, 1H), 3.84-3.76 (m, 2H), 2.81 (dd, 1H, J = 13.2, 4.0 Hz), 2.52-2.46 (m, 1H), 2.02-1.22 (m, 9H) ppm; ^{13}C NMR (CDCl_3 , 100 MHz) δ 200.4, 168.1, 126.1, 82.4, 60.1, 36.9, 35.6, 33.9, 30.0, 27.2, 25.6 ppm; IR (thin film) ν 3520, 2928, 2855, 1725, 1640, 1388, 1345, 1289, 1100, 1010, 899 cm^{-1} .

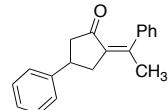


Purified by chromatography on silica gel (9:1 hexanes/EtOAc); yellow oil (61%); TLC R_f = 0.19 (9:1 hexanes/EtOAc); ^1H NMR (CDCl_3 , 400 MHz) δ 6.06-6.05 (m, 1H), 5.25-5.23 (m, 1H), 4.73 (t, 2H, J = 1.6 Hz), 2.59 (s, 2H), 1.97-1.44 (m, 8H) ppm; ^{13}C NMR (CDCl_3 , 100 MHz) δ 198.9, 151.3, 126.3, 79.1, 68.9, 60.1, 43.1, 24.7 ppm; IR (thin film) ν 2901, 1734, 1646, 1604, 1499, 1309, 1159, 1034 cm^{-1} .

General procedure for carbocyclization of alkyne-derived thioesters. Thioester (0.44 mmol), $\text{Pd}(\text{OAc})_2$ (9.9 mg, 0.04 mmol, 10 mol%), $\text{Cu}(\text{thiophene-2-carboxylate})$ (269.0 mg, 1.41 mmol, 3.2 equiv), tri(2-furyl)phosphine (20.5 mg, 0.09 mmol, 20 mol%), and phenylboronic acid (86.0 mg, 0.71 mmol, 1.6 equiv) were added sequentially to a flame-dried 10 mL round bottom flask. The flask was placed under vacuum and back-filled with N_2 . THF (3.7 mL), which had been degassed by a freeze-pump-thaw cycle (3x), was added. The brown suspension was stirred at 60 $^{\circ}\text{C}$ for 18 h, after which time the reaction was quenched with 10 mL of saturated aqueous NaHCO_3 . The mixture was transferred to a separatory funnel and extracted with 3 x 15 mL of EtOAc. The combined organic layers were washed with 1 x 20 mL of saturated aqueous sodium chloride, dried over MgSO_4 , filtered through a pad of Celite, and concentrated under reduced pressure to a brown residue. The desired product was isolated following purification by chromatography on silica gel (conditions given below).

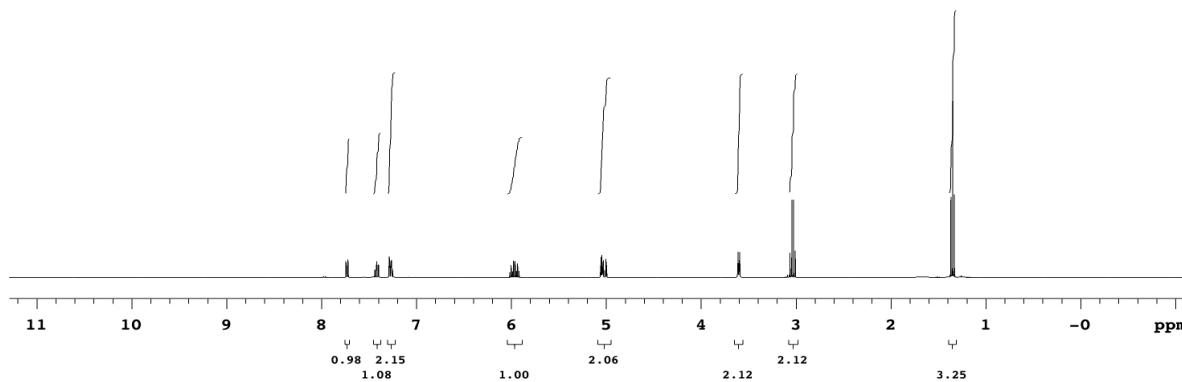
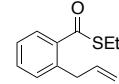


Purified by chromatography on silica gel (9:1 hexanes/EtOAc); yellow oil (56%); TLC R_f = 0.20 (9:1 hexanes/EtOAc); ^1H NMR (CDCl_3 , 400 MHz) δ 7.37-7.29 (m, 3H), 7.18-7.14 (m, 2H), 2.77 (tq, 2H, J = 7.2, 1.2 Hz), 2.32 (t, 2H, J = 8.0 Hz), 2.12 (t, 3H, J = 1.6 Hz), 1.97 (tt, 2H, J = 7.6, 7.2 Hz) ppm; ^{13}C NMR (CDCl_3 , 100 MHz) δ 205.0, 146.5, 141.7, 132.2, 127.9, 127.5, 127.3, 40.3, 30.0, 24.9, 19.3 ppm; IR (thin film) ν 2962, 1772, 1712, 1624, 1440, 1411, 1191, 1125 cm^{-1} . HRMS (ES $^+$) calcd for $\text{C}_{13}\text{H}_{15}\text{O}$ 187.1123 found 187.1117 ($\text{M}+\text{H}^+$).



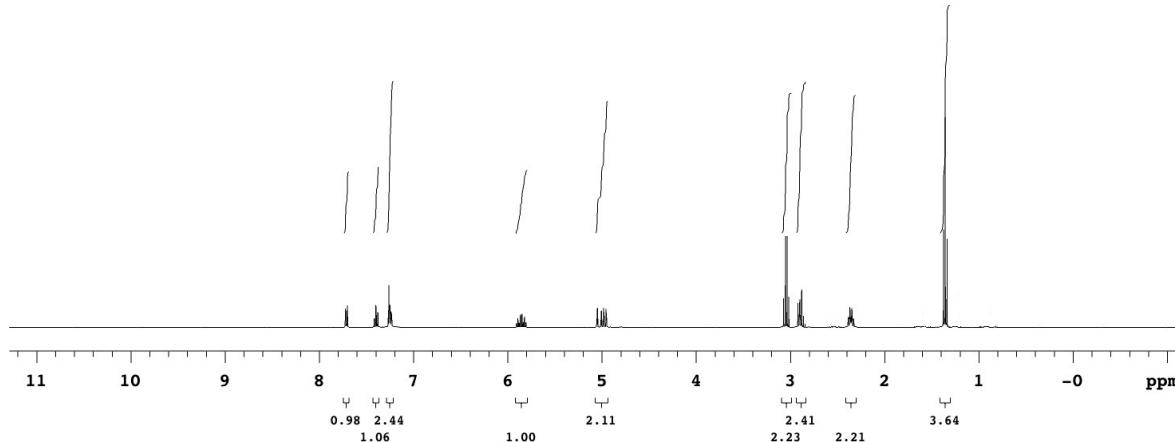
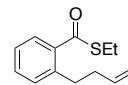
Purified by chromatography on silica gel (gradient elution: 7:3 hexanes/ CH_2Cl_2 \rightarrow 8:1 hexanes/EtOAc); white solid (67%); TLC R_f = 0.11 (7:3 hexanes/ CH_2Cl_2); ^1H NMR (CDCl_3 , 400 MHz) δ 7.41-7.17 (m, 10H), 3.52-3.40 (m, 1H), 3.26 (dd, 1H, J = 16.2, 7.8 Hz), 2.86-2.78 (m, 1H), 2.74 (dd, 1H, J = 17.6, 7.8 Hz), 2.54 (dd, 1H, J = 17.6, 10.8 Hz), 2.14 (s, 3H); ^{13}C NMR (CDCl_3 , 100 MHz) δ 203.2, 147.2, 144.0, 141.5, 132.2, 128.8, 128.0, 127.7, 127.4, 126.8, 126.8, 47.6, 38.53, 38.46, 24.9 ppm; IR (thin film) ν 3206, 2891, 1706, 1621, 1596, 1491, 1440, 1193 cm^{-1} ; HRMS (ES $^+$) calcd for $\text{C}_{19}\text{H}_{19}\text{O}$ 263.1436 found 263.1430 ($\text{M}+\text{H}^+$).

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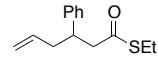
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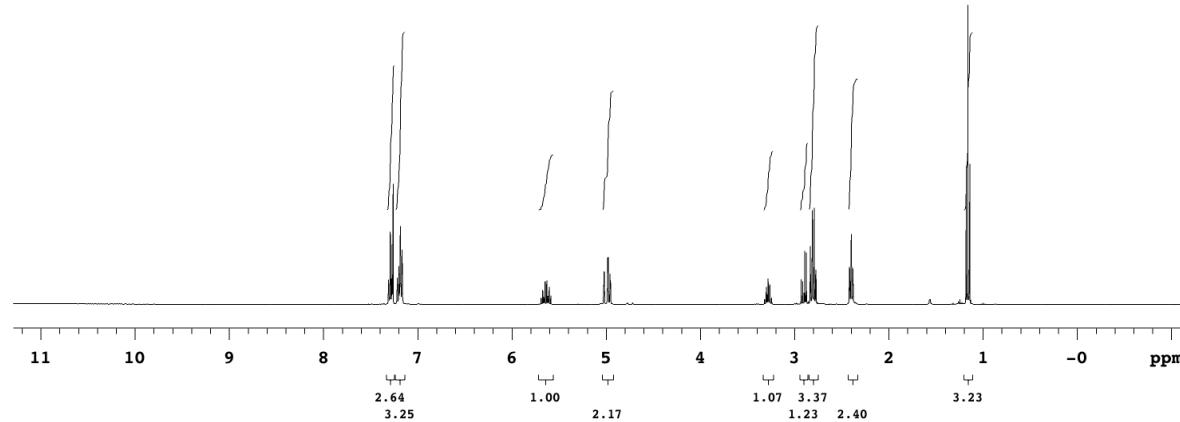
STANDARD 1H OBSERVE

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/export/home/arun/vnmrsys/data
Sample directory:



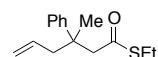
Pulse Sequence: s2pul
Solvent: CDCl3

Relax. delay 0.500 sec
Pulse 59.0 degrees
Acq. time 4.002 sec
Width 4997.5 Hz
16 repetitions
OBSERVE H1, 400.1115367 MHz
DATA PROCESSING
FT size 65536
Total time 1 min

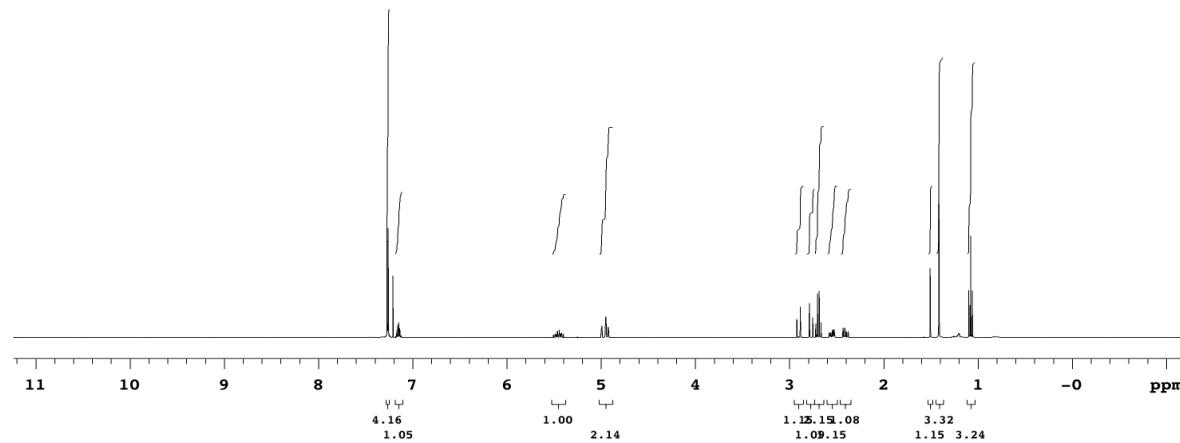


Archive directory:
/export/home/arun/vnmrsys/data
Sample directory:

File: apt3-133-fracl0-forcharac
Pulse Sequence: s2pul
Solvent: CDCl3



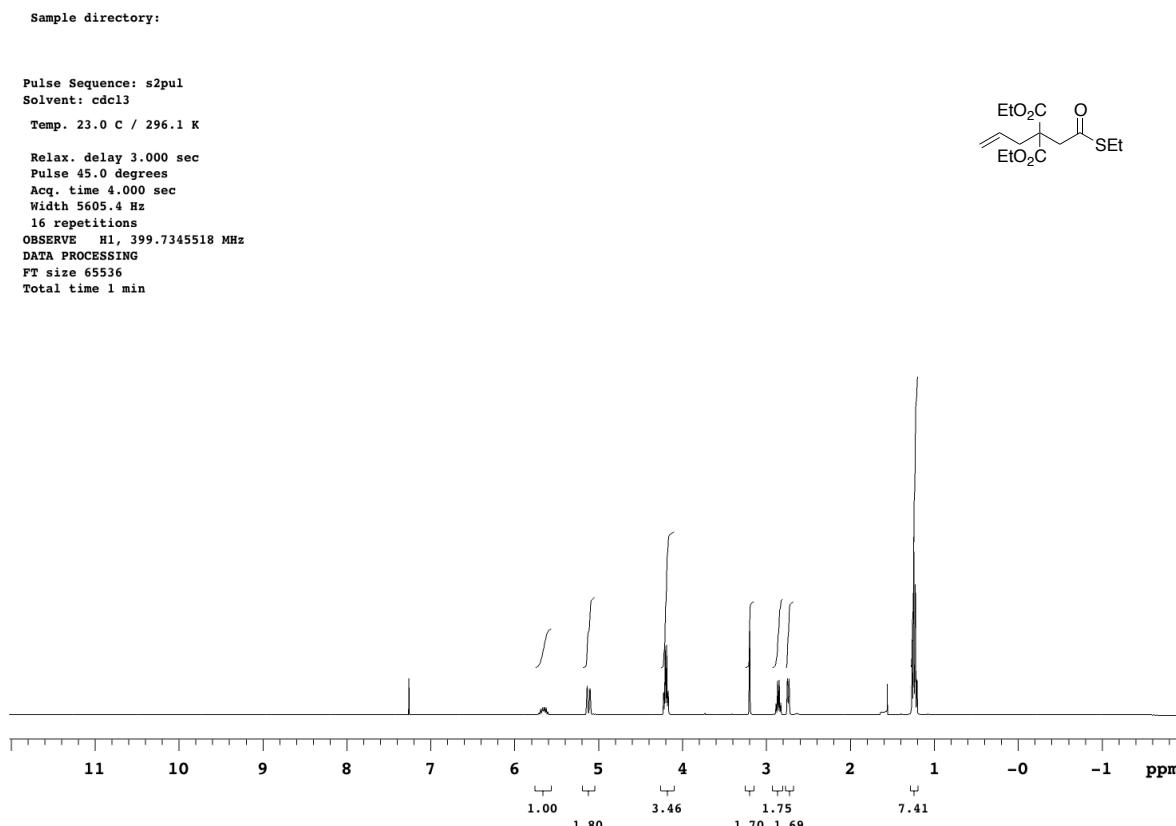
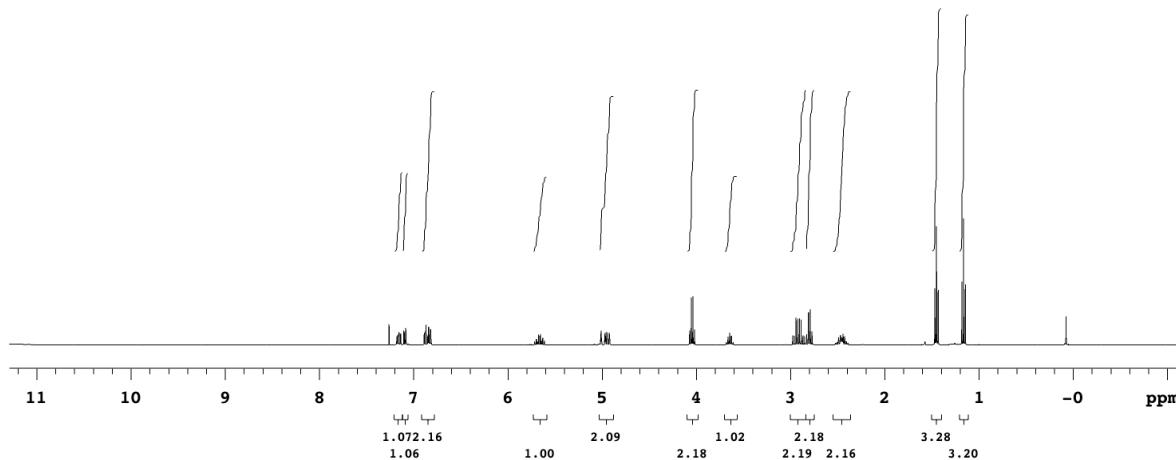
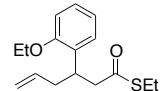
Relax. delay 3.000 sec
Pulse 52.1 degrees
Acq. time 4.002 sec
Width 4997.5 Hz
64 repetitions
OBSERVE H1, 400.1115580 MHz
DATA PROCESSING
FT size 65536
Total time 7 min



Archive directory:
/export/home/arun/vnmrsys/data
Sample directory:

Pulse Sequence: s2pul
Solvent: CDCl₃

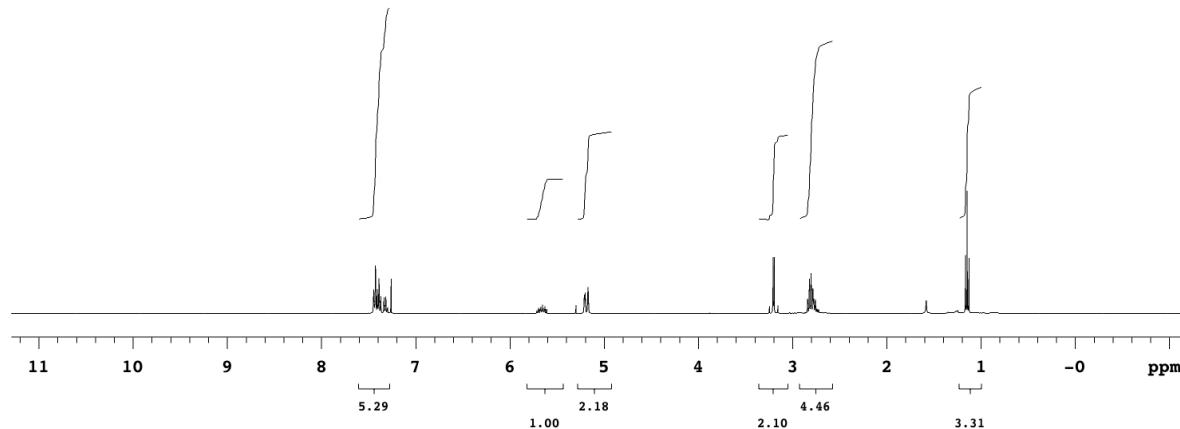
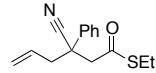
Relax. delay 3.000 sec
Pulse 52.1 degrees
Acq. time 4.002 sec
Width 4997.5 Hz
16 repetitions
OBSERVE H1, 400.1115369 MHz
DATA PROCESSING
FT size 65536
Total time 1 min



Archive directory:
/export/home/arun/vnmrsys/data
Sample directory:

Pulse Sequence: s2pul
Solvent: CDCl₃

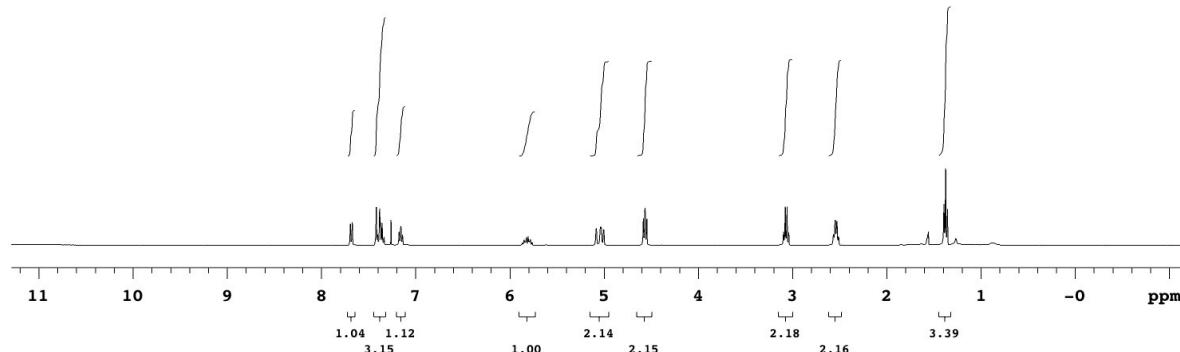
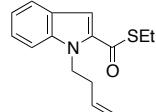
Relax. delay 3.000 sec
Pulse 52.1 degrees
Acq. time 4.002 sec
Width 4997.5 Hz
16 repetitions
OBSERVE H1, 400.1115366 MHz
DATA PROCESSING
FT size 65536
Total time 1 min



Archive directory:
/export/home/arun/vnmrsys/data
Sample directory:

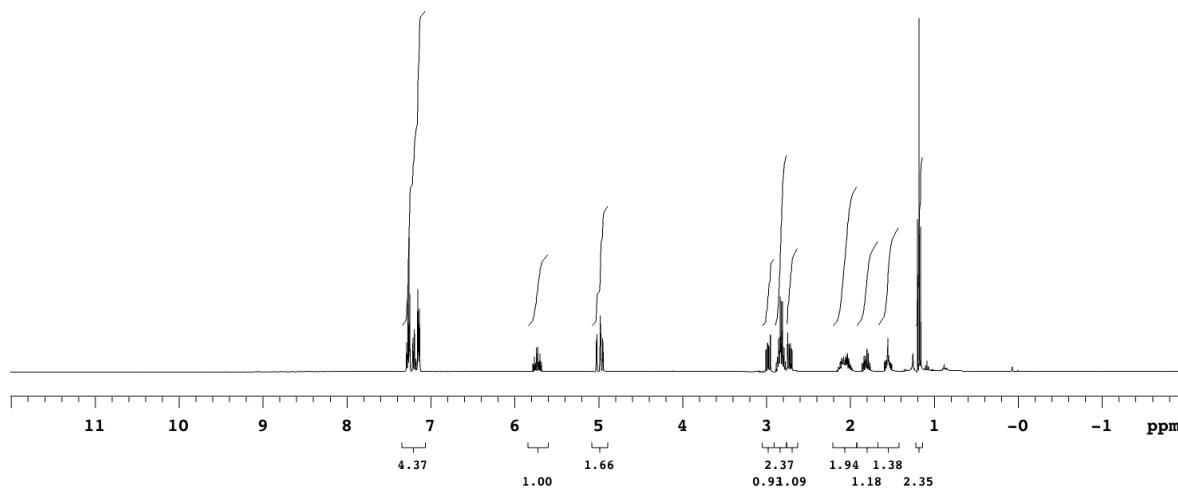
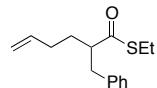
Pulse Sequence: s2pul
Solvent: CDCl₃

Relax. delay 3.000 sec
Pulse 52.1 degrees
Acq. time 4.002 sec
Width 4997.5 Hz
16 repetitions
OBSERVE H1, 400.1115369 MHz
DATA PROCESSING
FT size 65536
Total time 1 min

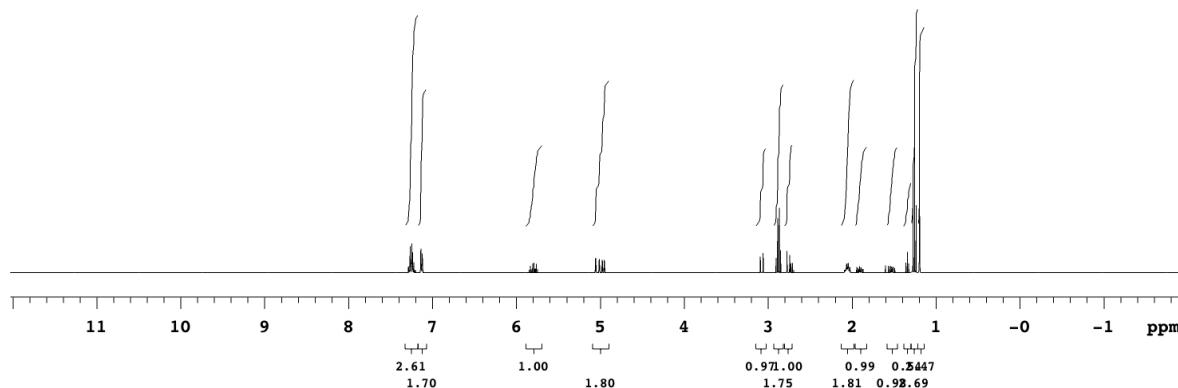
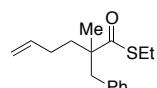


Archive directory:
Sample directory:

Pulse Sequence: s2pul
Solvent: *cdcl*3
Temp. 22.0 C / 295.1 K
Relax. delay 3.000 sec
Pulse 45.0 degrees
Acq. time 4.000 sec
Width 5605.4 Hz
16 repetitions
OBSERVE H1, 399.7291453 MHz
DATA PROCESSING
FT size 65536
Total time 1 min

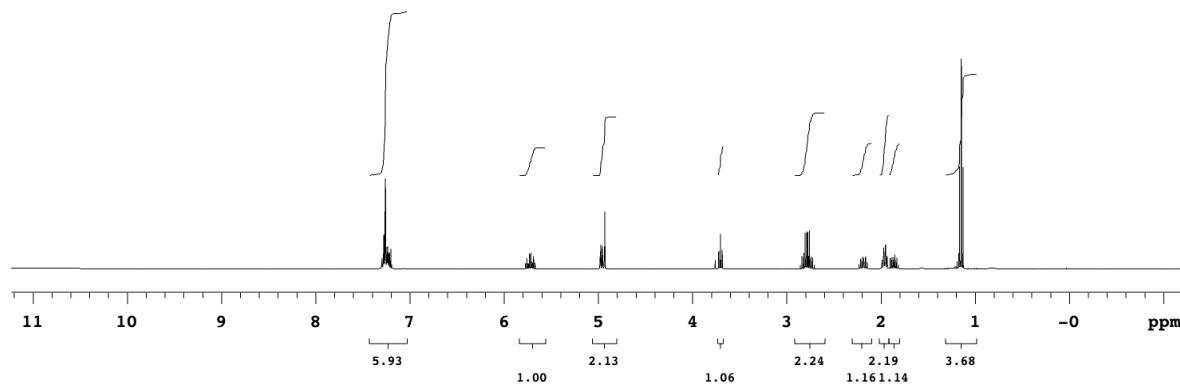
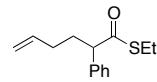


Pulse Sequence: s2pul
Solvent: *cdcl*3
Temp. 23.0 C / 296.1 K
Relax. delay 3.000 sec
Pulse 45.0 degrees
Acq. time 4.000 sec
Width 5605.4 Hz
16 repetitions
OBSERVE H1, 399.7345483 MHz
DATA PROCESSING
FT size 65536
Total time 1 min

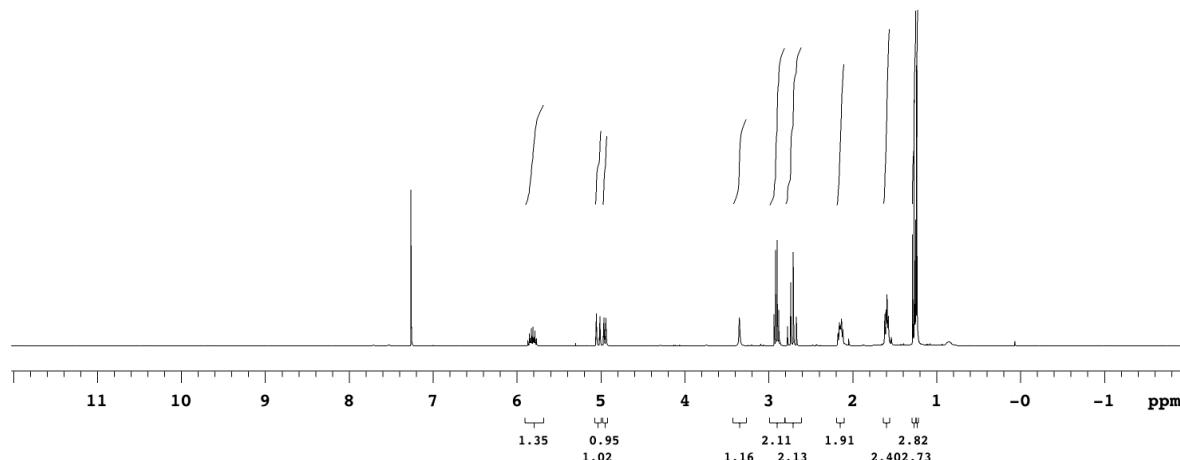
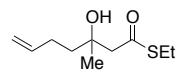


Archive directory:
/export/home/arun/vnmrsys/data
Sample directory:

Pulse Sequence: s2pul
Solvent: CDCl₃
Relax. delay 3.000 sec
Pulse 52.1 degrees
Acq. time 4.002 sec
Width 4997.5 Hz
16 repetitions
OBSERVE H1, 400.1115613 MHz
DATA PROCESSING
FT size 65536
Total time 1 min



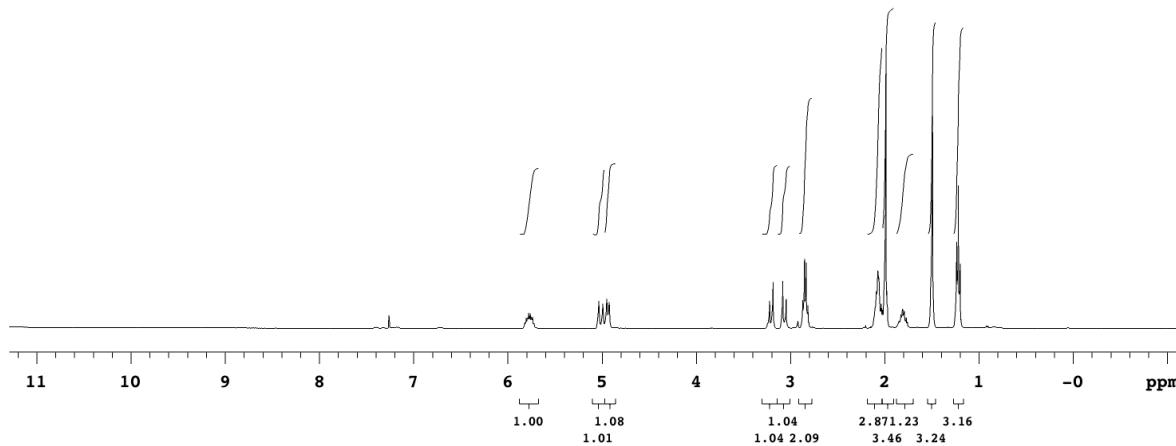
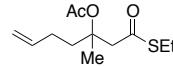
Pulse Sequence: s2pul
Solvent: cdcl₃
Temp. 20.0 C / 293.1 K
Relax. delay 3.000 sec
Pulse 45.0 degrees
Acq. time 4.000 sec
Width 5605.4 Hz
16 repetitions
OBSERVE H1, 399.7345501 MHz
DATA PROCESSING
FT size 65536
Total time 1 min



Archive directory:
/export/home/arun/vnmrsys/data
Sample directory:

Pulse Sequence: s2pul
Solvent: CDCl₃

Relax. delay 3.000 sec
Pulse 51.2 degrees
Acq. time 4.002 sec
Width 4997.5 Hz
16 repetitions
OBSERVE H1, 400.1115371 MHz
DATA PROCESSING
FT size 65536
Total time 1 min

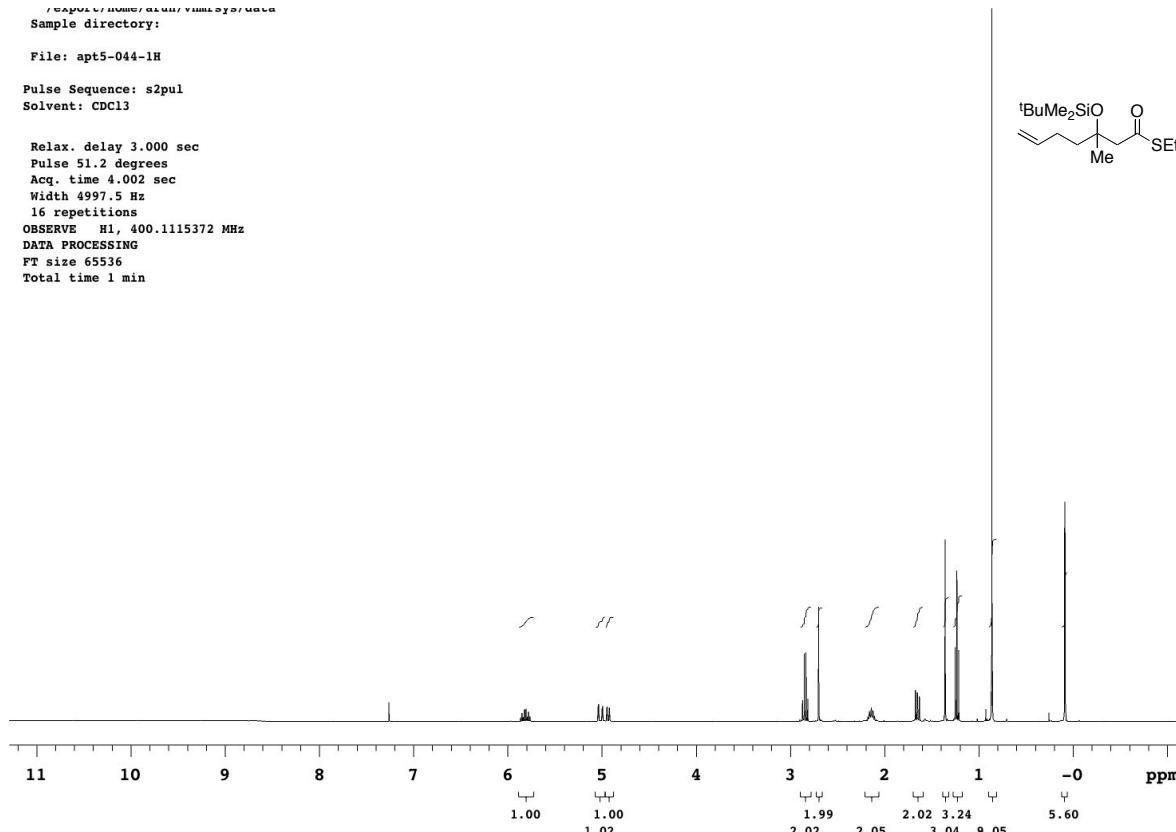
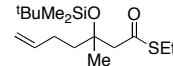


/export/home/arun/vnmrsys/data
Sample directory:

File: apt5-044-1H

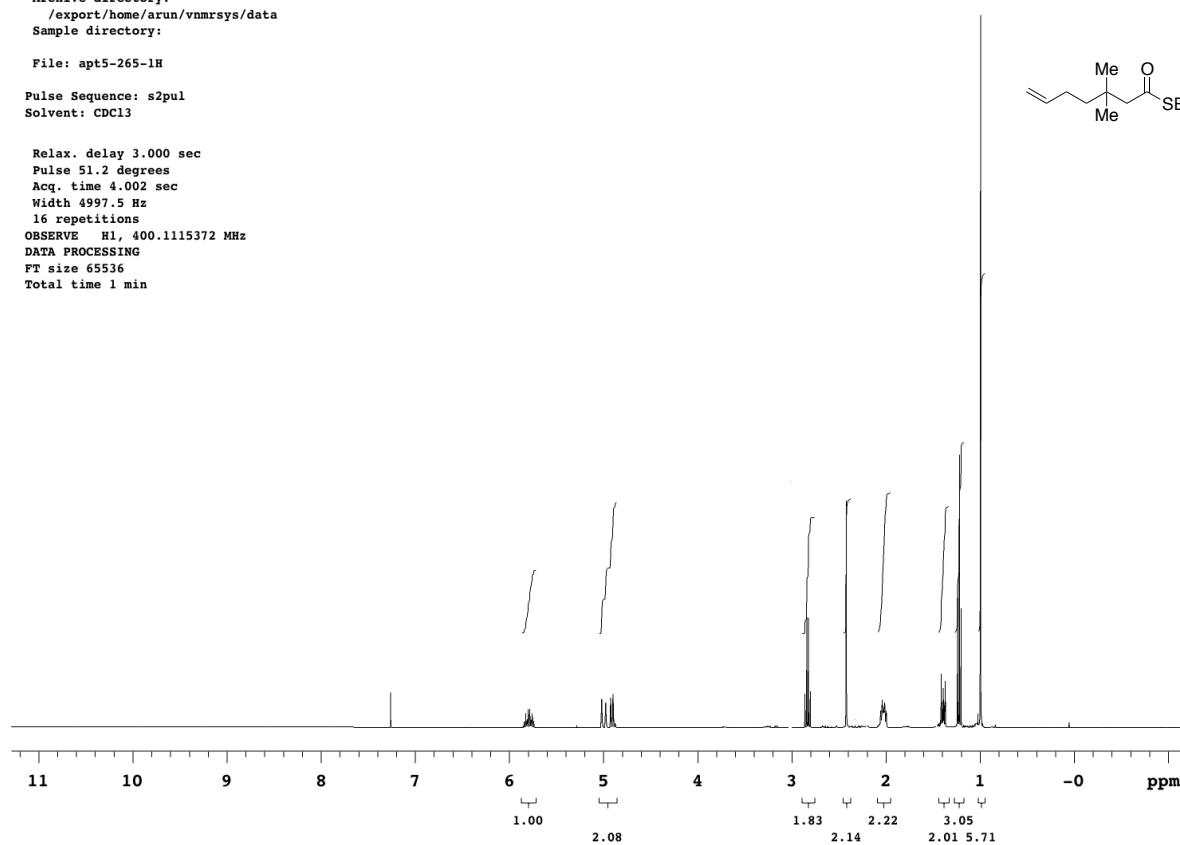
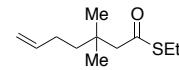
Pulse Sequence: s2pul
Solvent: CDCl₃

Relax. delay 3.000 sec
Pulse 51.2 degrees
Acq. time 4.002 sec
Width 4997.5 Hz
16 repetitions
OBSERVE H1, 400.1115372 MHz
DATA PROCESSING
FT size 65536
Total time 1 min



Archive directory:
/export/home/arun/vnmrjsys/data
Sample directory:
File: apt5-265-1H
Pulse Sequence: s2pul
Solvent: CDCl₃

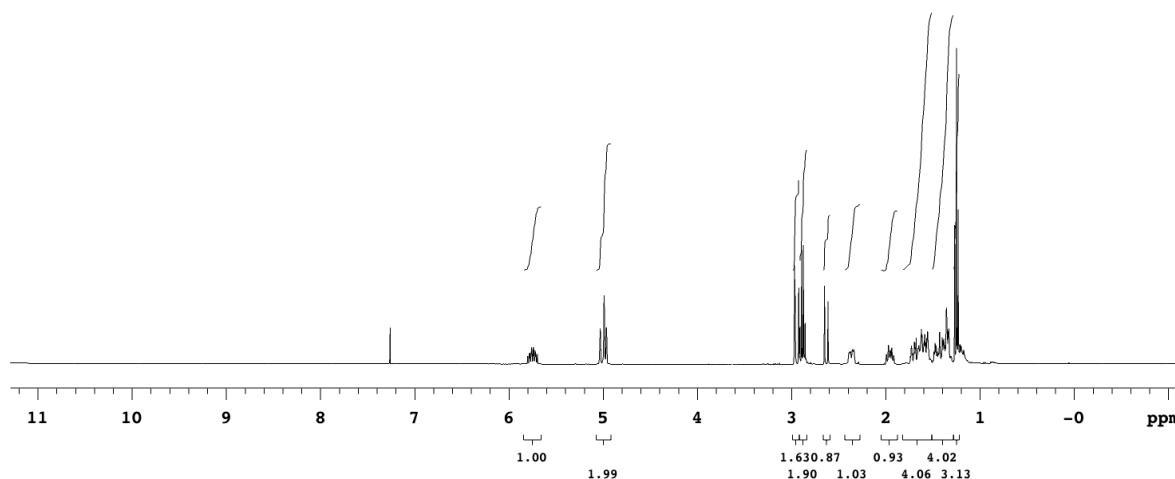
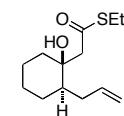
Relax. delay 3.000 sec
Pulse 51.2 degrees
Acq. time 4.002 sec
Width 4997.5 Hz
16 repetitions
OBSERVE H₁, 400.1115372 MHz
DATA PROCESSING
FT size 65536
Total time 1 min



/export/home/arun/vnmrjsys/data
Sample directory:

Pulse Sequence: s2pul
Solvent: CDCl₃

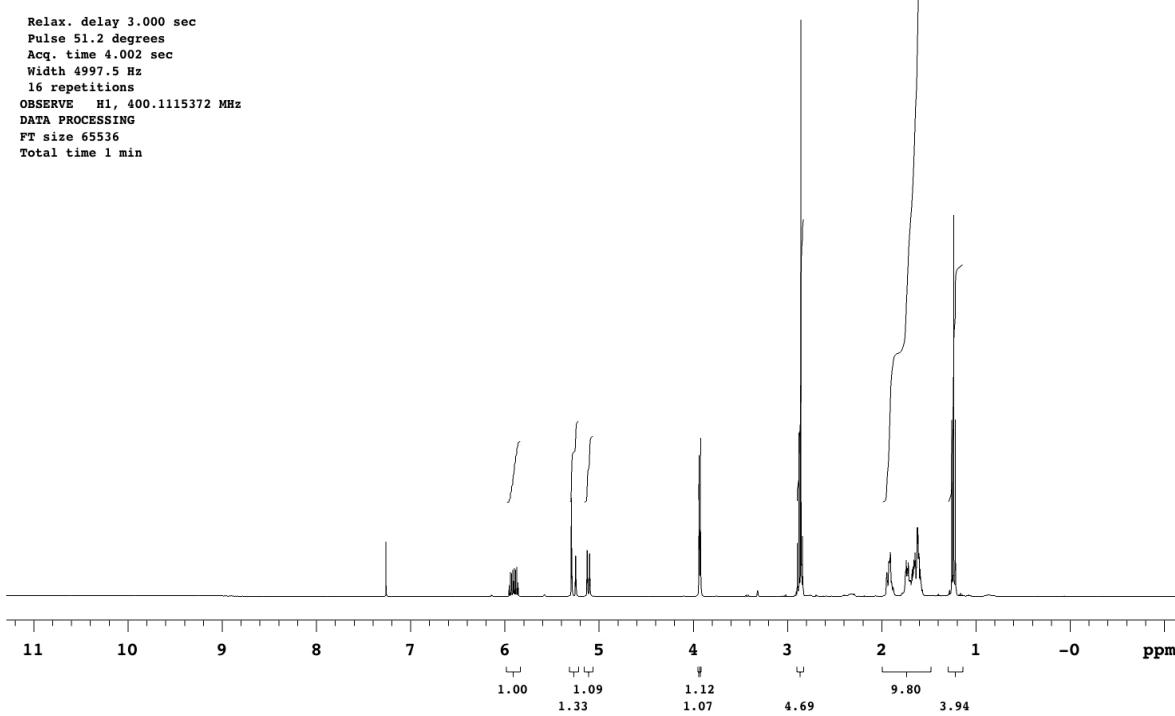
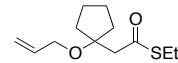
Relax. delay 3.000 sec
Pulse 51.2 degrees
Acq. time 4.002 sec
Width 4997.5 Hz
16 repetitions
OBSERVE H₁, 400.1115366 MHz
DATA PROCESSING
FT size 65536
Total time 1 min



Archive directory:
/export/home/arun/vnmrsys/data
Sample directory:

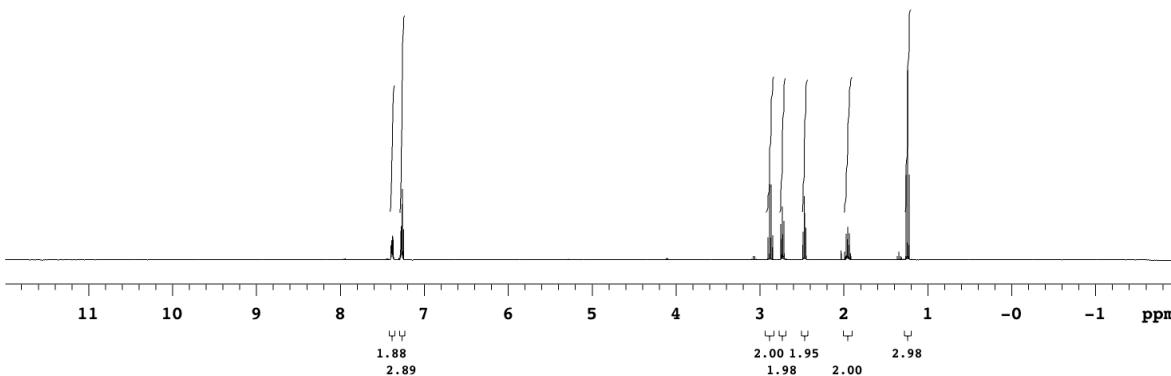
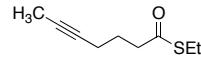
Pulse Sequence: s2pul
Solvent: CDCl₃

Relax. delay 3.000 sec
Pulse 51.2 degrees
Acq. time 4.002 sec
Width 4997.5 Hz
16 repetitions
OBSERVE H1, 400.1115372 MHz
DATA PROCESSING
FT size 65536
Total time 1 min



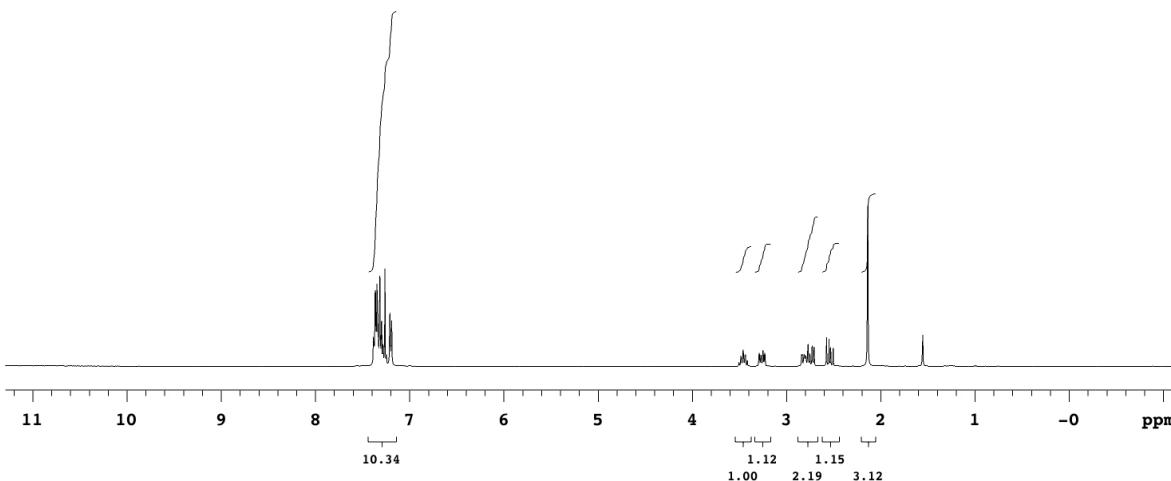
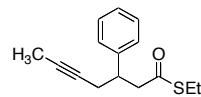
Archive directory:
Sample directory:

Pulse Sequence: s2pul
Solvent: cdc13
Temp. 23.0 C / 296.1 K
Relax. delay 3.000 sec
Pulse 45.0 degrees
Acq. time 4.000 sec
Width 5605.4 Hz
16 repetitions
OBSERVE H1, 399.7291529 MHz
DATA PROCESSING
FT size 65536
Total time 1 min



Archive directory:
/export/home/arun/vnmrsys/data
Sample directory:

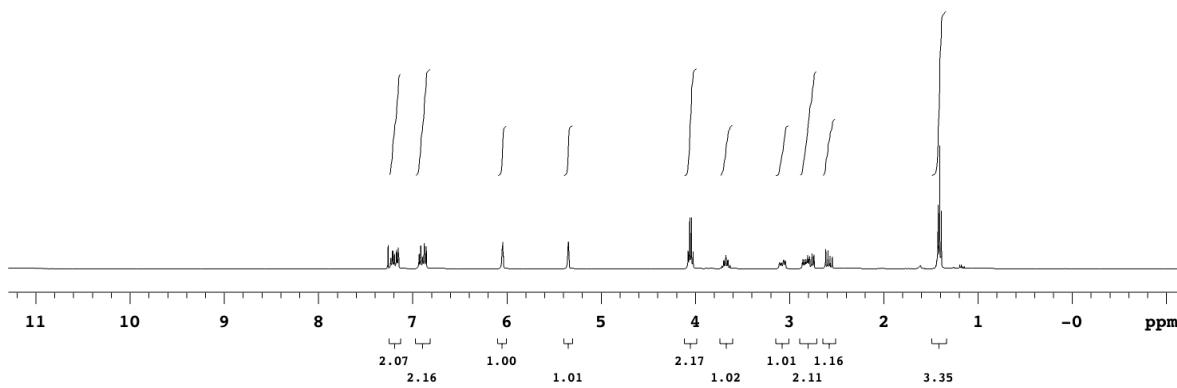
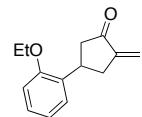
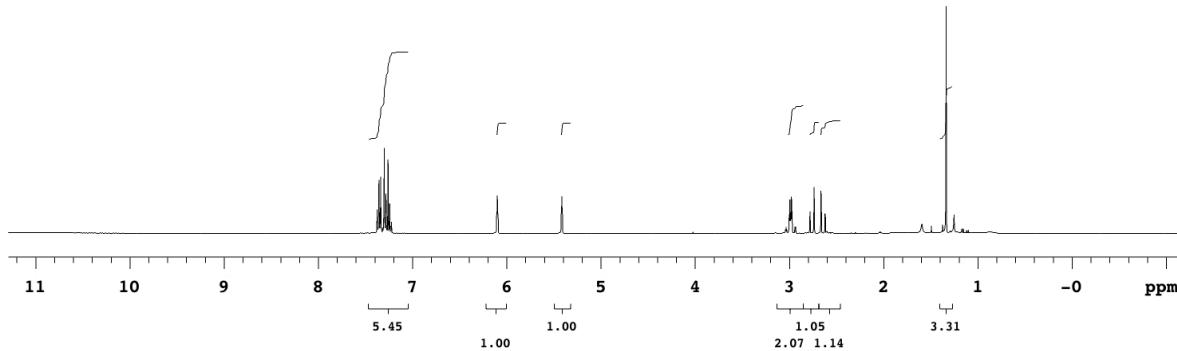
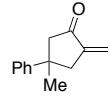
Pulse Sequence: s2pul
Solvent: CDCl3
Relax. delay 0.500 sec
Pulse 60.0 degrees
Acq. time 4.002 sec
Width 4997.5 Hz
32 repetitions
OBSERVE H1, 400.1115371 MHz
DATA PROCESSING
FT size 65536
Total time 2 min



Archive directory:
/export/home/arun/vnmrsys/data
Sample directory:

Pulse Sequence: s2pul
Solvent: CDCl₃

Relax. delay 3.000 sec
Pulse 52.1 degrees
Acq. time 4.002 sec
Width 4997.5 Hz
16 repetitions
OBSERVE H₁, 400.1115375 MHz
DATA PROCESSING
FT size 65536
Total time 1 min



Archive directory:

File: apt3-122flashed

Pulse Sequence: s2pul

Solvent: CDCl₃

Relax. delay 3.000 sec

Pulse 52.1 degrees

Acq. time 4.002 sec

Width 4997.5 Hz

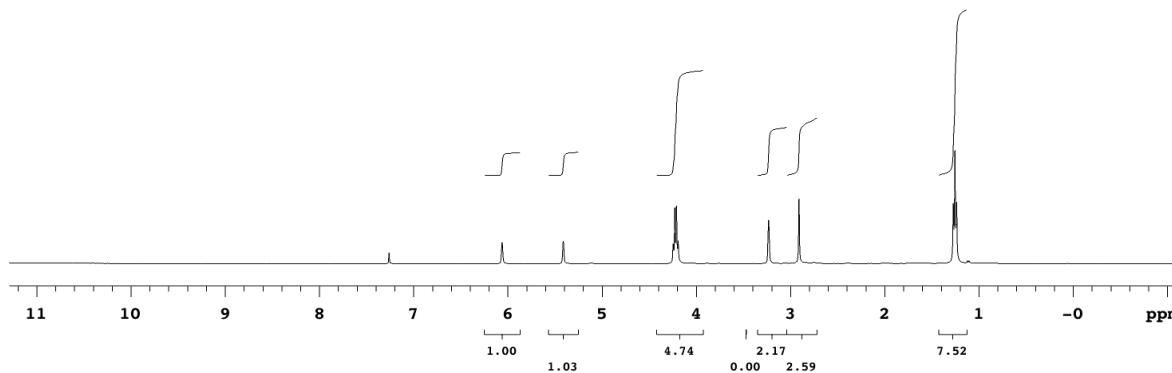
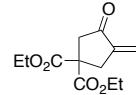
16 repetitions

OBSERVE H1, 400.1115371 MHz

DATA PROCESSING

FT size 65536

Total time 1 min



/export/home/arun/vnmrsys/data
Sample directory:

Pulse Sequence: s2pul

Solvent: CDCl₃

Relax. delay 3.000 sec

Pulse 52.1 degrees

Acq. time 4.002 sec

Width 4997.5 Hz

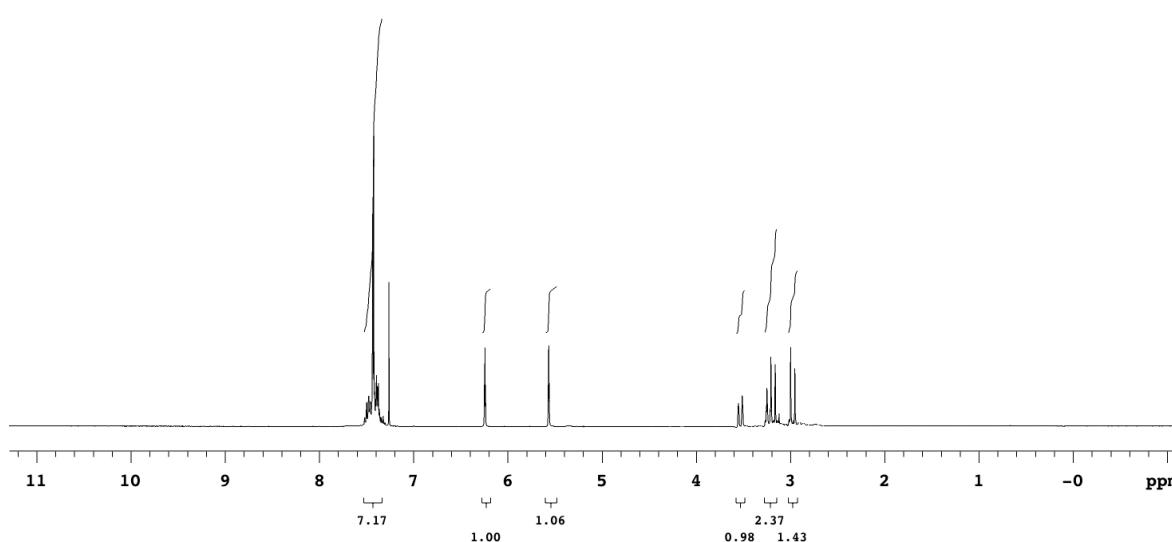
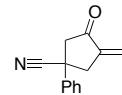
12 repetitions

OBSERVE H1, 400.1115371 MHz

DATA PROCESSING

FT size 65536

Total time 1 min



Archive directory:

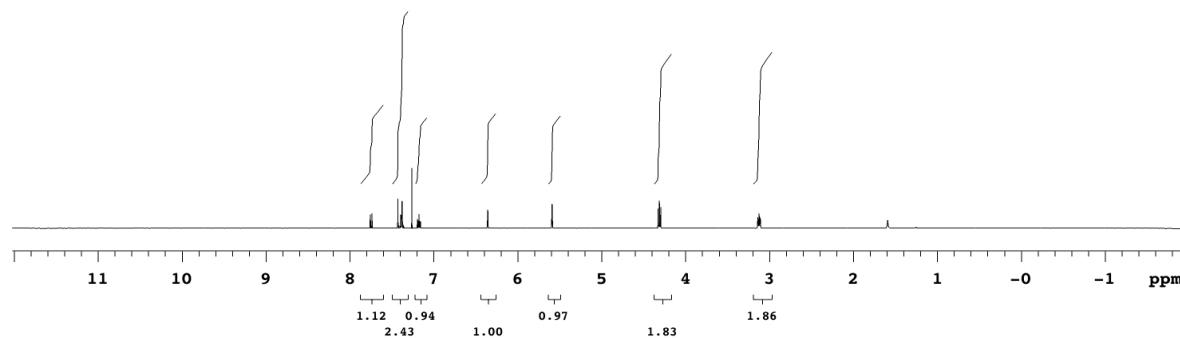
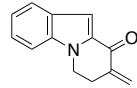
```

Pulse sequence: szpui
Solvent: cdc13

Temp. 22.0 C / 295.1 K

Relax. delay 3.000 sec
Pulse 45.0 degrees
Acq. time 4.000 sec
Width 5605.4 Hz
16 repetitions
OBSERVE H1, 399.7345513 MHz
DATA PROCESSING
F1 size 65536
Total time 1 min

```



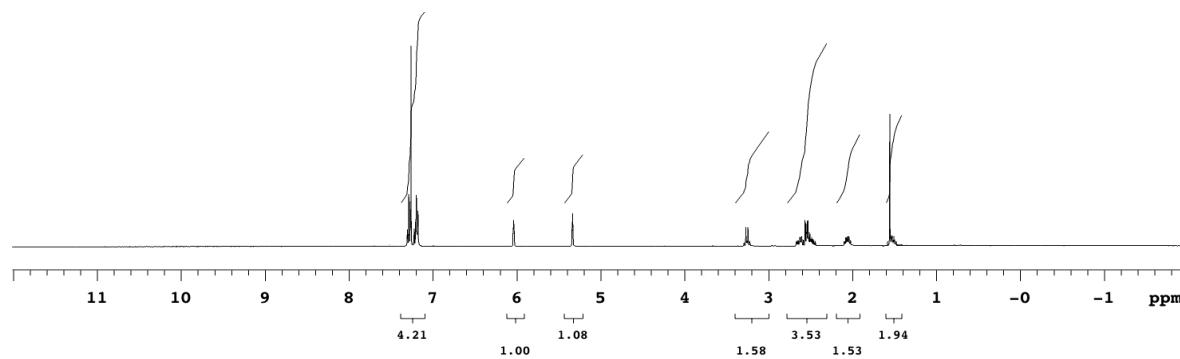
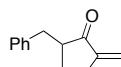
Archive directory:

```

Temp. 22.0 C / 295.1 K

Relax. delay 3.000 sec
Pulse 45.0 degrees
Acq. time 4.000 sec
Width 5605.4 Hz
16 repetitions
OBSERVE H1, 399.7291446 MHz
DATA PROCESSING
FT size 65536
Total time 1 min

```

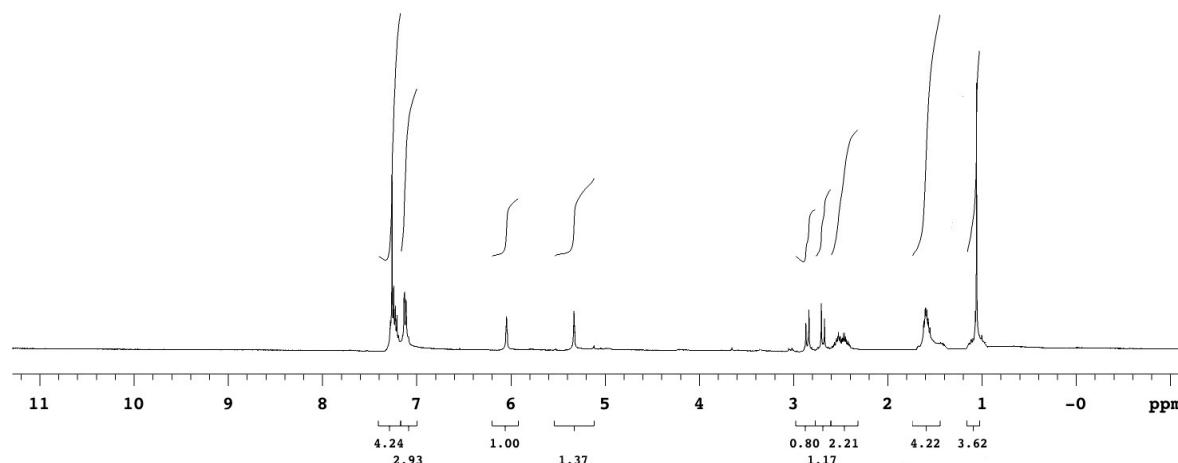
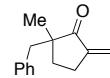


Archive directory:

File: apt3-224-for-charac

Pulse Sequence: s2pul
Solvent: CDCl₃

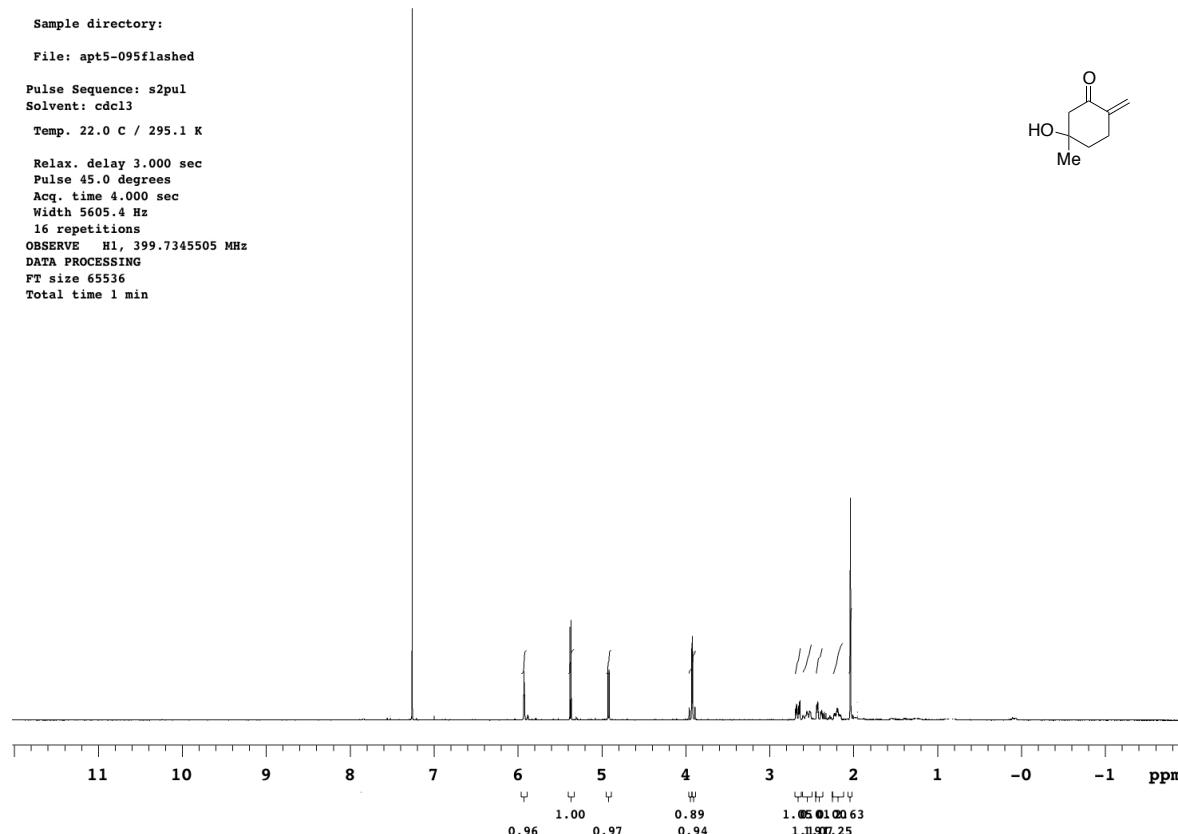
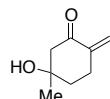
Relax. delay 3.000 sec
Pulse 52.1 degrees
Acq. time 4.002 sec
Width 4997.5 Hz
16 repetitions
OBSERVE H1, 400.1115369 MHz
DATA PROCESSING
FT size 65536
Total time 1 min



Sample directory:

File: apt5-095flashed

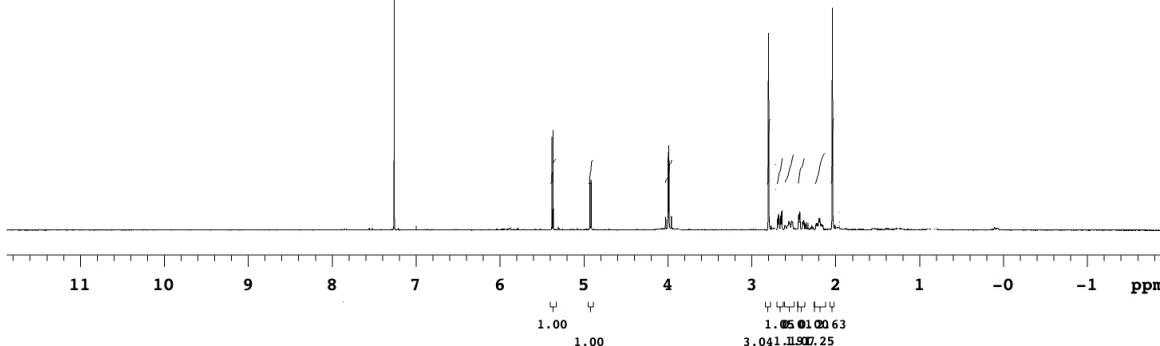
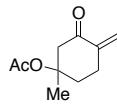
Pulse Sequence: s2pul
Solvent: CDCl₃
Temp. 22.0 C / 295.1 K
Relax. delay 3.000 sec
Pulse 45.0 degrees
Acq. time 4.000 sec
Width 5605.4 Hz
16 repetitions
OBSERVE H1, 399.7345505 MHz
DATA PROCESSING
FT size 65536
Total time 1 min



Archive directory:

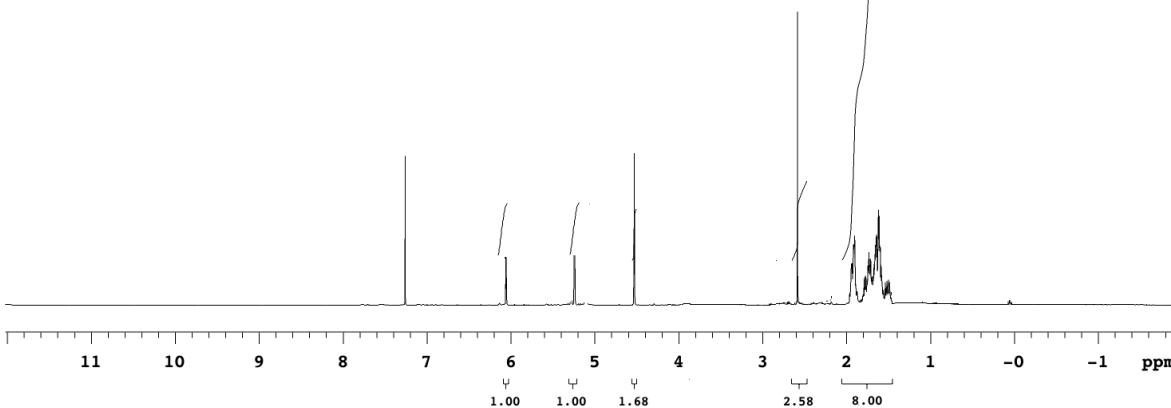
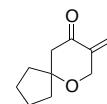
Sample directory:

Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 22.0 C / 295.1 K
Relax. delay 3.000 sec
Pulse 45.0 degrees
Acq. time 4.000 sec
Width 5605.4 Hz
16 repetitions
OBSERVE H1, 399.7345505 MHz
DATA PROCESSING
FT size 65536
Total time 1 min



Sample directory:

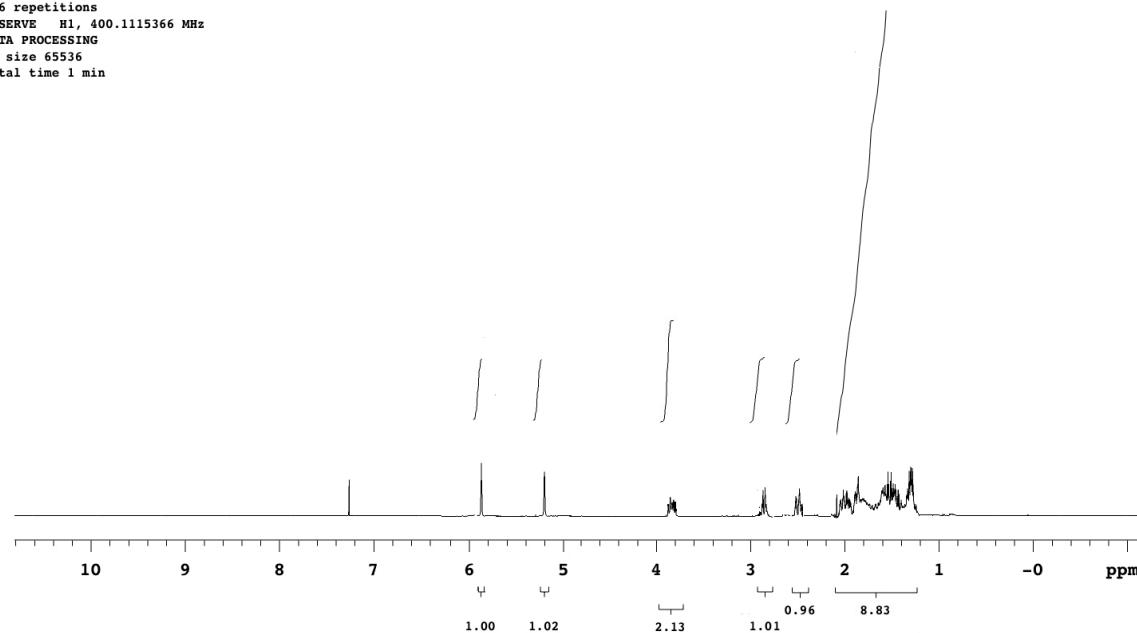
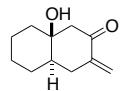
Pulse Sequence: s2pul
Solvent: cdcl3
Temp. 22.0 C / 295.1 K
Relax. delay 3.000 sec
Pulse 45.0 degrees
Acq. time 4.000 sec
Width 5605.4 Hz
16 repetitions
OBSERVE H1, 399.7345506 MHz
DATA PROCESSING
T size 65536
Total time 1 min



Archive directory:
/export/home/arun/vnmrsys/data

Pulse Sequence: s2pul
Solvent: CDCl3

```
Relax. delay 3.000 sec
Pulse 51.2 degrees
Acq. time 4.002 sec
Width 4997.5 Hz
16 repetitions
OBSERVE H1, 400.1115366 MHz
DATA PROCESSING
FT size 65536
Total time 1 min
```



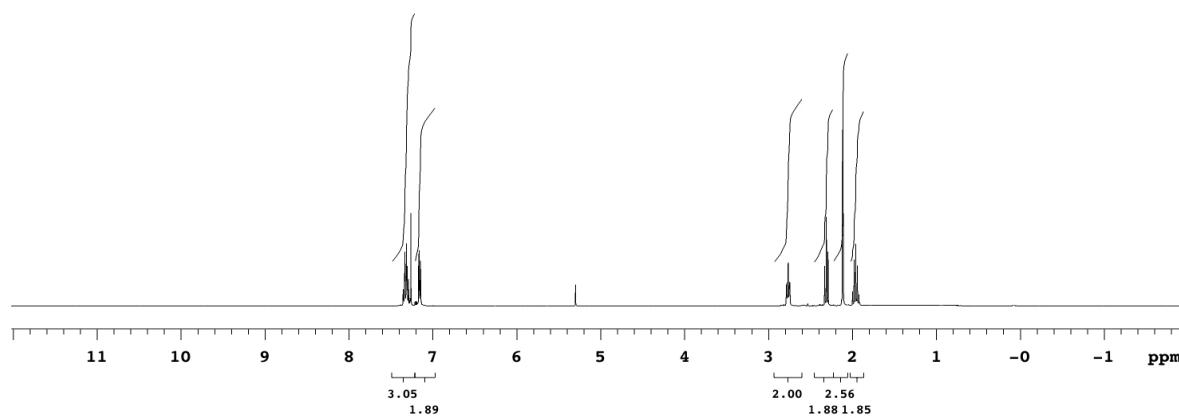
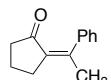
Archive directory:

```

Pulse Sequence: s2pul
Solvent: cdc13
Temp. 22.0 C / 295.1 K

Relax. delay 3.000 sec
Pulse 45.0 degrees
Acq. time 4.000 sec
Width 5605.4 Hz
16 repetitions
OBSERVE H1, 399.73455
DATA PROCESSING
FT size 65536
Total time 1 min

```



STANDARD 1H OBSERVE

Archive directory:
/export/home/arun/vnmrsys/data
Sample directory:

File: TK2-236prod

Pulse Sequence: s2pul
Solvent: CDCl₃

Relax. delay 0.500 sec
Pulse 60.0 degrees
Acq. time 4.002 sec
Width 4997.5 Hz
32 repetitions
OBSERVE H1, 400.1115371 MHz
DATA PROCESSING
FT size 65536
Total time 2 min

