Stereoselective Dichlorination of Allylic Alcohol Derivatives to Access Key Stereochemical Arrays of the Chlorosulfolipids

Grant M. Shibuya, Jacob S. Kanady, and Christopher D. Vanderwal

1102 Natural Sciences II, Department of Chemistry, The University of California, Irvine, California 92697-2025

SUPPORTING INFORMATION

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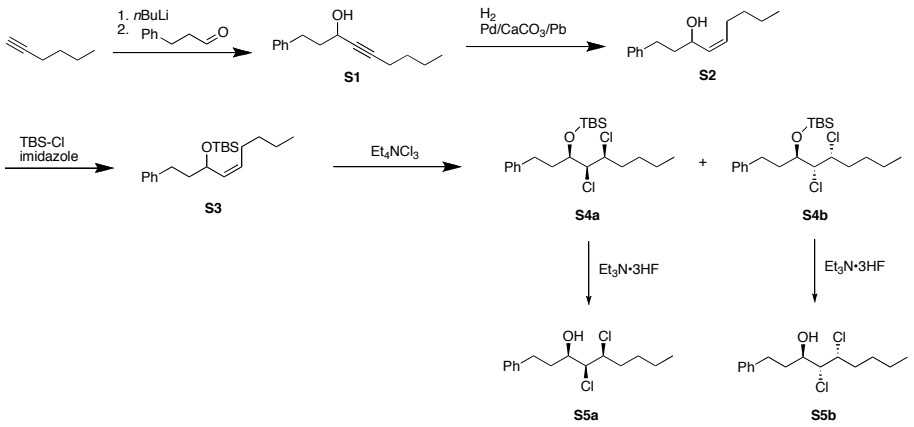
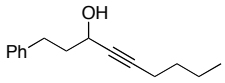
I. General

^{1}H NMR and ^{13}C NMR spectra were recorded at ambient temperature using Bruker DRX spectrometers at either 500 or 600 MHz (^{1}H) and 125 MHz (^{13}C). The chemical shifts are reported in parts per million (ppm) using the deuterated solvent as the internal reference. Coupling constants are reported in Hertz (Hz) and the peak multiplicity is listed as follows: s, singlet; d, doublet; t, triplet; q, quartet, m, multiplet. Infrared (IR) spectra were obtained using a Perkin-Elmer Spectrum RXI FT-IR instrument. High-resolution mass spectra were obtained with a Waters LCT Premier mass spectrometer. All column chromatography was performed using Dynamic Adsorbents 230-400 mesh silica gel (SiO_2) with the indicated solvent system, unless otherwise noted. All reactions were carried under a nitrogen atmosphere using flame-dried or oven-dried glassware. All amine bases were distilled over drying agents (CaH_2) prior to use. All solvents were dried by passing through activated alumina columns.

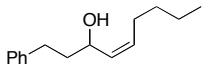
II. Evaluation of Optimal *O*-substituent (representative sequence)





1-Phenylnon-4-yn-3-ol (S1):

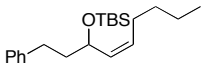
To a cooled (0 °C) solution of 1-hexyne (2.8 g, 34 mmol) in THF (76 mL) was added *n*-butyllithium (13.6 mL, 29 mmol, 2.1 M). After stirring for 15 min, hydrocinnamaldehyde (3.3 mL, 23 mmol, 90%) was added over 10 min. The reaction mixture was warmed to ambient temperature over 10 min and stirred for 30 min. The reaction was quenched with saturated aqueous NH_4Cl (50 mL) and extracted with EtOAc (2 x 20 mL). The combined organic layers were washed with saturated aqueous $NaHCO_3$ (20 mL) and brine (20 mL), dried over anhydrous $MgSO_4$, and concentrated *in vacuo* to afford **S1** as a yellow oil (5.7 g, quant.): ^{1}H NMR: (500 MHz, $CDCl_3$) δ 7.31–7.28 (m, 2H), 7.23–7.19 (m, 3H), 4.37 (app. q, J = 4.9, 1H), 2.80 (t, J = 7.9, 2H), 2.24 (ddd, J = 7.1, 6.9, 2.0, 2H), 2.0 (m, 2H), 1.7 (d, J = 5.2, 1H), 1.55–1.46 (m, 2H), 1.45–1.41 (m, 2H), 0.93 (t, J = 7.2, 3H). The spectroscopic data were in agreement with those previously reported.[a]



(*Z*)-1-Phenylnon-4-en-3-ol (S2):

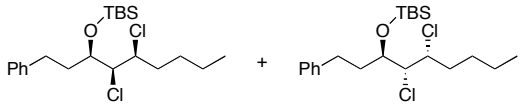
To a solution of 1-phenylnon-4-yn-3-ol (**S1**) (2.1 g, 9.5 mmol) in MeOH (48 mL) were added quinoline (1.1 mL, 9.5 mmol) and Lindlar's catalyst (0.048 g). The reaction mixture was vigorously stirred under an atmosphere of hydrogen for 2 h. The reaction mixture was filtered through Celite while rinsing with EtOAc. The solvent was removed *in vacuo* to afford **S2** as a light yellow oil. The crude product was

purified by flash chromatography (14:86 EtOAc:hexanes) to give a light yellow oil (1.7 g, 81%): ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.33–7.28 (m, 2H), 7.22–7.18 (m, 3H), 5.53 (ddt, *J* = 10.9, 7.4, 0.9, 1H), 5.44 (ddt, *J* = 11.2, 8.6, 1.4, 1H), 4.47 (app. q, *J* = 14.2, 1H), 2.76–2.65 (m, 2H), 2.08–2.01 (m, 2H), 1.98–1.91 (m, 1H), 1.80–1.73 (m, 1H), 1.37–1.28 (m, 4H), 0.91 (t, *J* = 7.2, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 142.2, 132.9, 132.5, 128.59, 128.57, 126.0, 67.4, 39.2, 32.0, 31.9, 27.6, 22.5, 14.1; IR (thin film): 3338, 2928, 1603, 1496, 1454, 1050, 699 cm^{-1}; HRMS (CI/dichloromethane) *m* / *z* calcd for $C_{15}H_{22}O$ $(M + Na)^+$ 241.1568, found 241.1566.



(*Z*)-3-(*tert*-Butyldimethylsilyloxy)-1-phenylnon-4-ene (S3):

To a solution of (*Z*)-1-phenylnon-4-en-3-ol (**S2**) (0.74 g, 3.4 mmol) in CH_2Cl_2 (13.6 mL) was added imidazole (0.28 g, 4.1 mmol). The mixture was stirred for 5 min until dissolution of the imidazole and then *tert*-butyldimethylsilylchloride (0.56 g, 3.7 mmol) was added. The reaction mixture was stirred at room temperature for 3 h. The reaction mixture was diluted with hexanes (50 mL) and rinsed with saturated aqueous $NaHCO_3$ (30 mL), brine (30 mL), dried over anhydrous $MgSO_4$, and concentrated *in vacuo*. The solution was filtered through a plug of silica gel with hexanes to afford **S3** as a colorless oil (0.86 g, 76%): ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.30–7.26 (m, 2H), 7.21–7.16 (m, 3H), 5.42–5.32 (m, 2H), 4.47 (ddd, *J* = 7.4, 5.3, 2.5, 1H), 2.72 (ddd, *J* = 13.7, 10.8, 5.4, 1H), 2.61 (ddd, *J* = 13.8, 10.6, 5.8, 1H), 2.06–1.96 (m, 2H), 1.90–1.82 (m, 1H), 1.74–1.67 (m, 1H), 1.38–1.28 (m, 4H), 0.95–0.87 (m, 12H), 0.06 (s, 3H), 0.04 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 142.6, 133.8, 129.5, 128.4, 128.3, 125.7, 68.5, 40.4, 31.9, 31.8, 27.6, 26.0, 22.5, 18.3, 14.1, –4.1, –4.7; IR (thin film) 3028, 2956, 2929, 2858, 1472, 1456, 1253, 1086, 836, 776, 698 cm^{-1}; HRMS (EI/dichloromethane) *m* / *z* calcd for $C_{21}H_{36}OSi$ $(M + Na)^+$ 355.2433, found 355.2428.



S4a **S4b**

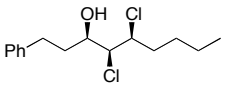
3-(*tert*-Butyldimethylsilyloxy)-4,5-dichloro-1-phenylnonane (S4a and S4b):

To a cooled (–78 °C) solution of **S3** (0.47 g, 1.4 mmol) in CH_2Cl_2 (4.7 mL, 0.3 M) was added tetraethylammonium trichloride (0.49 g, 2.1 mmol). The reaction mixture was stirred for 2 h and quenched with a 1:1 solution of saturated aqueous $NaHCO_3$/10% aqueous $Na_2S_2O_3$ (15 mL). The aqueous layer was extracted with CH_2Cl_2 (10 mL) and the combined organic layers were washed with brine (3 mL), dried over anhydrous $MgSO_4$, and concentrated *in vacuo*. ^{1}H NMR spectroscopic analysis revealed a 1.8:1 ratio of diastereomers. The diastereomers were separated by flash chromatography (hexanes) to yield **S4a** (0.25 g, 44%, ca. 90% purity) and **S4b** (0.12 g, 21%) as colorless oils (65%).

(3*R,4*S**,5*S**)-(*tert*-Butyldimethylsilyloxy)-4,5-dichloro-1-phenylnonane (S4a):** ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.32–7.29 (m, 2H), 7.23–7.20 (m, 3H), 4.12 (ddd, 8.3, 5.4, 3.2, 1H), 4.06 (ddd, *J* = 6.5, 4.9, 4.9, 1H), 4.00 (dd, *J* = 6.5, 3.2, 1H), 2.76 (ddd, *J* = 13.6, 11.0, 5.1, 1H) 2.62 (ddd, *J* = 13.6, 11.3, 6.0, 1H), 2.11–2.06 (m, 1H), 1.99–1.93 (m, 1H), 1.88–1.82 (m, 2H), 1.48–1.40 (m, 1H), 1.39–1.28 (m, 3H), 0.96 (s, 9H), 0.93 (t, *J* = 7.2, 3H), 0.15 (s, 3H), 0.14 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 141.9, 128.8, 128.5, 126.3, 74.4, 68.9, 36.6, 35.5, 30.7, 28.8, 26.1, 26.0, 22.4, 18.4, 14.1, –3.9, –4.0; IR (thin film) 3028, 2930, 2858, 1463, 1255, 1101, 836, 777 cm^{-1}; HRMS (CI/methanol) *m* / *z* calcd for $C_{21}H_{36}Cl_2OSi$ $(M + Na)^+$ 425.1810, found 425.1796.

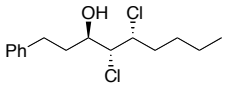
(3*R,4*R**,5*R**)-(*tert*-Butyldimethylsilyloxy)-4,5-dichloro-1-phenylnonane (S4b):** ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.31–7.28 (m, 2H), 7.22–7.19 (m, 3H), 4.45 (ddd, *J* = 9.0, 5.3, 1.5, 1H), 4.13 (ddd, *J* = 6.8, 6.2, 3.1, 1H), 3.93 (dd, *J* = 9.3, 1.5 Hz, 1H), 2.77 (ddd, *J* = 12.9, 12. 8, 4.2, 1H), 2.66 (ddd, *J* = 13.0, 12.7, 5.3, 1H), 2.30–2.23 (m, 1H), 2.03–1.97 (m, 1H), 1.89 (ddt, *J* = 18.6, 16.6, 4.1, 1H), 1.84–1.78 (m, 1H), 1.56–1.51 (m, 1H), 1.46–1.34 (m, 3H), 0.99–0.91 (m, 12H), 0.17 (d, *J* = 2.9, 6H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 142.4, 128.67, 128.60, 126.1, 72.2, 65.6, 62.3, 36.4, 35.8, 28.9, 28.7, 26.1,

22.3, 18.4, 14.1, –4.0, –4.7; IR (thin film) 2957, 2859, 1604, 1463, 1362, 1259, 1090, 1066, 837 cm^{-1}; HRMS (CI/methanol) *m* / *z* calcd for $C_{21}H_{36}Cl_2OSi$ $(M + Na)^+$ 425.1810, found 425.1802.



(3*R,4*S**,5*S**)-4,5-Dichloro-1-phenylnonan-3-ol (S5a):**

To a solution of TBS-protected chlorohydrin **S4a** (0.033 g, 0.11 mmol) in CH_2Cl_2 (1.1 mL) was added $Et_3N{\cdot}3HF$ (2 mL). The reaction mixture was stirred for 48 hours and monitored by thin-layer chromatography (5:1 hexanes/EtOAc). The reaction was quenched with saturated aqueous $NaHCO_3$ (5 mL) and stirred for 10 min. The reaction mixture was diluted with CH_2Cl_2 (3 mL) and the phases were separated. The aqueous layer was extracted with CH_2Cl_2 (5 mL). The combined organic layers were washed with saturated aqueous NH_4Cl (5 mL), brine (10 mL), dried over anhydrous $MgSO_4$, and concentrated *in vacuo*. The crude product was purified using preparative TLC (10:90 EtOAc:hexanes) to afford **S5a** as a colorless oil (0.007 g, 21%): ^1H NMR (500 MHz, $CDCl_3$) δ 7.32–7.29 (m, 2H), 7.24–7.20 (m, 3H), 4.12 (ddd, *J* = 9.0, 8.0, 4.6, 1H), 4.01 (dd, *J* = 5.6, 3.4, 1H), 3.97 (dddd, *J* = 8.9, 5.6, 5.3, 3.7, 1H), 2.89 (ddd, *J* = 13.8, 9.7, 5.5, 1H), 2.74 (ddd, *J* = 13.8, 9.6, 6.8, 1H), 2.23 (d, *J* = 5.3, 1H), 1.97–1.91 (m, 1H), 1.90–1.81 (m, 3H), 1.49–1.44 (m, 1H), 1.39–1.29 (m, 3H), 0.90 (t, *J* = 7.2, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 141.7, 128.7, 128.6, 126.3, 72.8, 67.6, 62.3, 36.1, 35.7, 31.9, 28.81, 22.4, 14.1, IR (thin film) 3410, 3027, 2928, 2860, 1497, 1455, 1066, 748, 700 cm^{-1}; HRMS (CI/methanol) *m* / *z* calcd for $C_{15}H_{22}Cl_2O$ $(M + Na)^+$ 311.0945, found 311.0940.



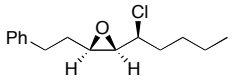
(3*R,4*R**,5*R**)-4,5-Dichloro-1-phenylnonan-3-ol (S5b):**

To a solution of TBS-protected chlorohydrin **S4b** (0.064 g, 0.16 mmol) in CH_2Cl_2 (1.6 mL) was added $Et_3N{\cdot}3HF$ (3 mL). The reaction mixture was stirred for 48 hours and monitored by thin-layer chromatography (5:1 hexanes/EtOAc). The reaction was quenched with saturated aqueous $NaHCO_3$ (10 mL) and stirred for 10 minutes. The reaction mixture was diluted with CH_2Cl_2 (6 mL) and the phases were separated. The aqueous layer was extracted with CH_2Cl_2 (6 mL). The combined organic layers were washed with saturated aqueous NH_4Cl (5 mL), brine (10 mL), dried over anhydrous $MgSO_4$, and

concentrated *in vacuo*. The crude product was purified by flash chromatography (10:90 EtOAc:hexanes) to give **S5b** colorless oil (0.015 g, 33%): ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.33–7.30 (m, 2H), 7.25–7.20 (m, 3H), 4.52 (ddd, *J* = 8.8, 5.3, 2.1, 1H), 3.95 (ddd, *J* = 8.8, 5.8, 2.3, 1H), 3.84 (dd, *J* = 8.8, 2.1, 1H), 2.91 (ddd, *J* = 13.7, 10.3, 5.2, 1H), 2.74 (ddd, *J* = 13.7, 9.8, 6.6, 1H), 2.34–2.28 (m, 1H), 2.23 (d, *J* = 5.7, 1H), 2.00–1.93 (m, 1H), 1.85–1.76 (m, 2H), 1.55–1.46 (m, 1H), 1.43–1.32 (m, 3H), 0.95 (t, *J* = 8.9, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 141.7, 128.7, 128.6, 126.3, 72.7, 67.5, 62.4, 36.1, 35.7, 31.9, 28.8, 22.4, 14.1; IR (thin film) 3430, 3028, 2958, 2929, 1641, 1496, 1455, 1069, 746, 700 cm^{-1}; HRMS (CI/methanol) *m* / *z* calcd for $C_{15}H_{22}Cl_2O$ $(M + Na)^+$ 311.0945, found 311.0938.

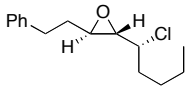
Alcohols S5a and S5b, whose relative stereochemistry was secured as discussed below, could be O-functionalized for correlation with the other products in Table 2.

III. Relative Stereochemistry Determination by Epoxide Formation



(3*R,4*R**,5*S**)-5-Chloro-3,4-epoxy-1-phenylnonane (S6a):**

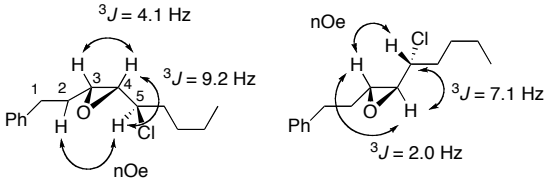
To a solution of chlorohydrin **S5a** (0.02 g, 0.07 mmol, contaminated with a trace of **S5b**) in THF (0.7 mL) was added NaH (0.004 g, 0.1 mmol, 60%). The solution was stirred for 2 h, then diluted with EtOAc (20 mL) and washed with water (10 mL), brine (10 mL), dried over anhydrous $MgSO_4$, filtered, and concentrated *in vacuo*. The crude product was purified by flash chromatography (9:1 hexanes:EtOAc) to afford **S6a** as a colorless oil (0.002 g, 11%, ca. 80% purity, contaminated with **S6b**): ^{1}H NMR (500 MHz, DMSO) δ 7.30–7.22 (m, 4H), 7.21–7.18 (m, 1H), 3.84 (ddd, *J* = 9.2, 8.6, 4.8, 1H), 3.13 (dd, *J* = 9.4, 4.1, 1H), 3.06 (ddd, *J* = 6.9, 5.5, 4.1, 1H), 2.76 (t, *J* = 8.0, 2H), 1.94–1.77 (m, 4H), 1.50–1.40 (m, 2H), 1.38–1.28 (m, 2H), 0.88 (t, *J* = 7.3, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 141.2, 128.7, 128.6, 126.4, 59.9, 59.0, 57.3, 36.7, 32.9, 29.8, 28.3, 22.5, 14.1; IR (thin film) 2958, 2931, 2862, 1496, 1454, 1266, 1030, 738, 699 cm^{-1}; HRMS (EI/dichloromethane) *m* / z calcd for $C_{15}H_{21}ClO$ $(M + Na)^+$ 275.1179, found 275.1169.



(3*R,4*S**,5*R**)-5-Chloro-3,4-epoxy-1-phenylnonane (S6b):**

To a solution of chlorohydrin **S5b** (0.008 g, 0.03 mmol) in THF (0.3 mL) was added NaH (0.002 g, 0.04 mmol, 60%). The solution was stirred for 160 min and diluted with EtOAc (10 mL). The solution was washed with water (5 mL), brine (5 mL), dried over anhydrous $MgSO_4$, filtered, and concentrated *in vacuo*. The crude product was purified by flash chromatography (9:1 hexanes:EtOAc) to afford **S6b** as a colorless oil (0.002 g, 21 %): ^{1}H NMR (500 MHz, DMSO) δ 7.30–7.27 (m, 2H), 7.24–7.22 (m, 2H), 7.20–7.17 (m, 1H), 3.75 (ddd, *J* = 8.7, 7.1, 4.5, 1H), 3.00 (dd, *J* = 7.1, 2.0, 1H), 2.96 (td, *J* = 5.7, 2.0, 1H), 2.69 (ddd, *J* = 8.9, 7.6, 4.0, 2H), 1.84–1.77 (m, 3H), 1.70–1.63 (m, 1H), 1.50–1.39 (m, 1H), 1.38–1.23 (m, 3H), 0.87 (t, *J* = 7.3, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 141.0, 128.6, 128.5, 126.2, 62.2, 61.1, 58.5, 35.6, 33.4, 32.2, 28.1, 22.3, 14.0; IR (thin film) 3029, 2929, 2861, 1604, 1496, 1456, 904, 751, 699 cm^{-1}; HRMS (EI/dichloromethane) *m* / *z* calcd for $C_{15}H_{21}ClO$ $(M + Na)^+$ 275.1179, found 275.1180.

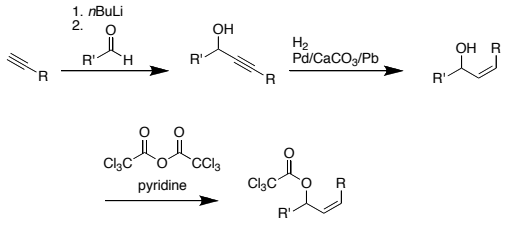
The relative stereochemistry at C3 and C4 were secured by comparison of the values of $^3J_{H3–H4}$ for S6a and S6b. Cis-epoxides are well known to exhibit larger $^3J_{H-H}$ values than the corresponding trans-epoxides. nOe experiments further supported our assignments.



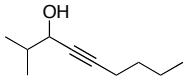
S6a **S6b**

IV. Scope of Diastereoselective Dichlorination

General Scheme for Substrate Preparation

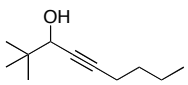


IVa. Propargylic Alcohol Syntheses (unoptimized):



2-Methylnon-4-yn-3-ol (S7):

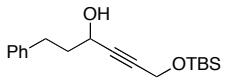
To a cooled solution (0 °C) of 1-hexyne (7.7 mL, 65 mmol, 98%) in THF (110 mL) was added *n*-butyllithium (22 mL, 2.5 M), and the resulting mixture was stirred for 15 min. Isobutyraldehyde (3 mL, 32 mmol) was added dropwise. The solution was warmed to ambient temperature over 15 min and stirred for 30 min. Excess reagent was quenched with saturated aqueous NH_4Cl (50 mL). The aqueous phase was extracted with EtOAc (2 x 20 mL). The combined organic extracts were washed with saturated aqueous $NaHCO_3$ (20 mL), brine (20 mL), dried over anhydrous $MgSO_4$, and concentrated *in vacuo*. The crude product was purified by flash chromatography (90:10 hexanes:EtOAc) to afford **S7** as a colorless oil (5.0 g, 91%): ^{1}H NMR (500 MHz, $CDCl_3$) δ 4.16 (s, 1H), 2.22 (td, *J* = 7.0, 1.9, 2H), 1.84 (d, *J* = 6.7, 1H), 1.68 (bs, 1H), 1.53-1.47 (m, 2H), 1.45–1.38 (m, 2H), 0.99 (d, *J* = 6.7, 3H), 0.98 (d, *J* = 6.7, 3H), 0.92 (t, *J* = 7.3, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 133.5, 130.9, 72.9, 34.4, 32.1, 27.7, 22,6, 18.5, 18.3, 14.2; IR (thin film) 3401, 2957, 2871, 1464, 1364, 1137, 1038, 1005 cm^{-1}; HRMS (CI/Methanol) *m* / *z* calcd for $C_{10}H_{18}O$ $(M + Na)^+$ 177.1255, found 177.1256.



2,2-Dimethylnon-4-yn-3-ol (S8):

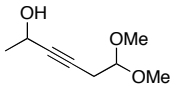
To a cooled (0 °C) solution of 1-hexyne (6.5 mL, 55 mmol) in THF (92 mL) was added dropwise *n*-butyllithium (18 mL, 2.3 M). The solution was stirred for 15 min before adding pivaldehyde (3.1 mL,

28 mmol) dropwise. The solution was warmed to ambient temperature over 15 min and stirred for 30 min. Excess reagent was quenched with saturated aqueous NH_4Cl (50 mL). The aqueous phase was extracted with EtOAc (2 x 20 mL). The combined organic extracts were washed with saturated aqueous $NaHCO_3$ (20 mL), brine (20 mL), dried over anhydrous $MgSO_4$, and concentrated *in vacuo* to afford **S8** as a colorless oil of sufficient purity for subsequent reactions (3.72 g, 80%): ^{1}H NMR (500 MHz, $CDCl_3$) δ 4.00, (app. td, *J* = 1.9, 5.8, 1H), 2.22 (td, *J* = 2.0, 6.9, 2H), 1.64 (d, *J* = 6.0, 1H), 1.53–1.48 (m, 2H), 1.46–1.38 (m, 2H), 0.99 (s, 9H), 0.92 (t, *J* = 7.3, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 86.5, 80.0, 71.9, 36.0, 31.0, 25.5, 22.2, 18.6, 13.8; IR (thin film) 3370, 2960, 2874, 1469, 1381, 1024 cm^{-1}; HRMS (EI/methanol) *m* / *z* calcd for $C_{11}H_{20}O$ $(M + Na)^+$ 191.1412, found 191.1414.



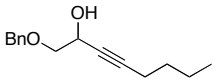
6-(*tert*-Butyldimethylsilyloxy)-1-phenylhex-4-en-3-ol (S9):

To a cold (–78 ºC) solution of 1-*tert*-butyldimethylsilyloxy-2-propyne[b] (0.35 g, 2.01 mmol) in THF (6.9 mL) was added *n*-butyllithium (0.77 mL, 1.93 mmol, 2.52 M), and the resulting mixture was stirred for 15 min. Hydrocinnamaldehyde (0.20 mL, 1.38 mmol) was added dropwise and the solution was warmed to ambient temperature over 10 min and stirred for 15 min. The reaction mixture was quenched with saturated aqueous NH_4Cl (5 mL) and diluted with hexanes (10 mL). The aqueous phase was extracted with hexanes (5 mL) and the combined organic extracts were washed with saturated aqueous $NaHCO_3$ (5 mL), brine (5 mL), dried over anhydrous $MgSO_4$, filtered, and concentrated *in vacuo* to afford a crude yellow oil that was purified by flash chromatography (5:1 hexanes:EtOAc) to afford **S9** as a light yellow oil (0.243 g, 58%): ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.31–7.28 (m, 2H), 7.23–7.19 (m, 3H), 4.44–4.40 (m, 1H), 4.37 (d, *J* = 1.4, 2H), 2.80 (t, *J* = 6.5, 2H), 2.07–1.99 (m, 2H), 1.75 (d, *J* = 4.5, 1H), 0.93 (s, 9H), 0.14 (s, 6H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 141.5, 128.71, 128.65, 126.2, 85.6, 84.2, 62.0, 51.9, 39.3, 31.6, 26.0, 18.5, –4.9; IR (thin film) 3369, 2954, 2930, 2858, 1255, 1087, 837 cm^{-1}; HRMS (CI/Dichloromethane) *m* / *z* calcd for $C_{18}H_{28}O_2Si$ $(M + Na)^+$ 327.1756, found 327.1753.



6,6-Dimethoxyhex-3-yn-2-ol (S10):

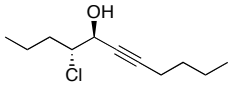
To a cold (–78 ºC) solution of 1,1-dimethoxy-3-butyne[c] (0.550 g, 4.25 mmol, 88% purity) in ether (14.2 mL) was added *n*-butyllithium (1.66 mL, 4.25 mmol, 2.56 M), and the resulting mixture was stirred for 15 min. Acetaldehyde (0.72 mL, 12.7 mmol) was added dropwise and the reaction mixture was stirred at –78 ºC for 15 min before warming to ambient temperature over 10 min. The reaction mixture was quenched with saturated aqueous NH_4Cl (5 mL) and the phases were separated. The aqueous phase was extracted with ether (5 mL) and the combined organic extracts were washed with brine (5 mL), dried over anhydrous $MgSO_4$, and concentrated *in vacuo*. The crude product was purified by flash chromatography (3:4 pentane:ether) to afford **S10** as a colorless and volatile oil (0.17 g, 25%): ^{1}H NMR (500 MHz, $CDCl_3$) δ 4.55–4.51 (m, 2H), 3.38 (s, 6H), 2.55 (dd, J = 1.9, 5.6, 2H), 1.76 (bs, 1H), 1.44 (d, J = 6.6, 3H) ^{13}C NMR (125 MHz) δ 102.6, 84.2, 79.4, 58.7, 53.6, 24.7, 24.1; IR (thin film) 3402, 2982, 2936, 1368, 1122, 1066 cm^{-1}; HRMS (CI/Methanol) m / z calcd for $C_8H_{14}O_3$ $(M + Na)^+$ 181.0841, found 181.0844.



1-Benzyloxyoct-3-yn-2-ol (S11):

To a cold (–78 ºC) solution of 1-hexyne (0.56 mL, 4.8 mmol) in THF (8.0 mL) was added *n*-butyllithium (1.6 mL, 3.6 mmol, 2.30 M), and the resulting mixture was stirred for 15 min. Benzyloxyacetaldehyde (0.35 mL, 2.4 mmol) was added dropwise and the reaction mixture was warmed to ambient temperature over 10 min and stirred for 15 min. The reaction mixture was quenched with saturated aqueous NH_4Cl (5 mL) and diluted with hexanes (10 mL). The organic phase was washed with $NaHCO_3$ (5 mL), brine (5 mL), dried over anhydrous $MgSO_4$, and concentrated *in vacuo*. The crude product was purified by flash chromatography (5:1 hexanes:EtOAc) to afford **S11** as a colorless oil (0.180 g, 32%): ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38–7.35 (m, 4H), 7.34–7.30 (m, 1H), 4.62 (dd, J = 10.0, 15.7, 2H), 4.57 (m, 1H), 3.63 (dd, J = 2.9, 8.1, 1H), 3.54 (dd, J = 6.5, 8.1, 1H), 2.40 (d, J = 3.7, 1H), 2.23–2.20 (m, 2H), 1.52–1.47 (m, 2H), 1.43–1.37 (m, 2H), 0.91 (t, J = 6.1, 3H); ^{13}C NMR (125

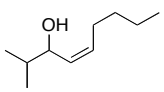
MHz, $CDCl_3$) δ 137.9, 128.7, 128.1, 128.0, 86.8, 77.8, 74.2, 73.6, 62.1, 30.8, 22.1, 18.6, 13.8; IR (neat) 3418, 3032, 2933, 2872, 2212, 1694, 1455, 1113, 1028; HRMS (CI/Dichloromethane) *m* / *z* calcd for $C_{15}H_{20}O_2$ $(M + Na)^+$ 255.1361, found 255.1363.



(4*R,5*S**)-4-Chloroundec-6-yn-5-ol (S12):**

To a cold (–78 ºC) solution of 1-hexyne (0.20 mL, 1.7 mmol) in THF (17 mL) was added *n*-butyllithium (0.68 mL, 1.7 mmol, 2.55 M). The reaction mixture was stirred for 1 min before adding a solution of 2-chloropentanal[d,e] (0.23 g, 1.9 mmol) in THF (1.7 mL, 1.1 M) dropwise. The reaction mixture was stirred at –78 ºC for 5 min and quenched with saturated NH_4Cl (5 mL), then warmed to ambient temperature. The reaction mixture was diluted with ether (8 mL) and the phases were separated. The aqueous phase was extracted with ether (5 mL). The combined organic extracts were rinsed with brine (3 x 5 mL), dried over anhydrous $MgSO_4$, and concentrated *in vacuo* to afford a yellow oil. The crude product was purified by flash chromatography (10:90 EtOAc:Hexanes eluent) to afford **S12** as a light yellow oil (0.44 g, 70%): ^{1}H NMR (500 MHz, $CDCl_3$) δ 4.51 (ddt, *J* = 2.0, 3.4, 8.4, 1H) 4.04 (ddd, *J* = 3.9, 5.0, 8.5, 1H), 2.35 (m, 1H), 2.24 (td, *J* = 2.0, 7.0, 2H), 1.84–1.79 (m, 2H), 1.66–1.59 (m, 1H), 1.54–1.48 (m, 2H), 1.47–1.39 (m, 3H), 0.96 (t, *J* = 7.4, 3H), 0.92 (t, *J* = 7.2, 3H); ^{13}C NMR (125 MHz, C_6D_6) δ 87.6, 78.5, 68.0, 67.0, 36.5, 31.2, 22.5, 20.3, 18.9, 14.0, 13.9; IR (neat) 3390, 2960, 2934, 2874, 2233, 1465, 1381, 1038 cm^{-1}; HRMS (CI/Dichloromethane) *m* / *z* calcd for $C_{11}H_{19}ClO$ $(M + Na)^+$ 225.1022, found 225.1021.

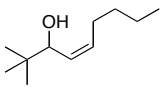
IVb. *Z*-allylic Alcohol Syntheses (unoptimized):



(*Z*)-2-Methyl-non-4-en-3-ol (S13):

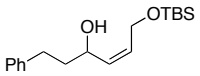
To a solution of 2-methylnon-4-yn-3-ol (**S7**) (0.39 g, 2.5 mmol) dissolved in MeOH (13 mL) was added Lindlar's catalyst (25 mg). The reaction mixture was stirred vigorously under an atmosphere of hydrogen for 3 h. The reaction mixture was filtered through Celite and washed with EtOAc (50 mL). The crude product was purified by flash chromatography (90:10 hexanes:EtOAc) to afford **S13** as a

colorless oil (0.18 g, 46 %): ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.55 (td, *J* = 11.1, 7.5, 1H), 5.39 (ddt, *J* = 10.9, 9.2, 1.5, 1H), 4.14 (dd, *J* = 8.8, 7.0, 1H), 2.16-2.05 (m, 2H), 1.70 (app. o, *J* = 6.8, 1H), 1.39-1.30 (m, 4H), 0.97 (d, *J* = 6.7, 3H), 0.91 (t, *J* = 7.0, 3H), 0.88 (d, *J* = 6.8, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 85.5, 80.0, 68.4, 34.9, 31.0, 22.1, 18.6, 18.3, 17.6, 13.8; IR (thin film) 3401, 2958, 1652, 1468, 1017 cm^{-1}; HRMS (CI/methanol) calcd for *m/z* $C_{10}H_{20}O$ $(M + Na)^+$ 179.1412, found 179.1411.



(*Z*)-2,2-Dimethylnon-4-en-3-ol (S14):

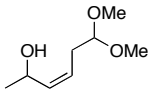
To a solution of 2,2-dimethylnon-4-yn-3-ol (**S8**) (1.0 g, 6 mmol) in MeOH (30 mL) was added Lindlar's catalyst (0.09 g). The reaction mixture was stirred vigorously under an atmosphere of hydrogen for 80 min. The reaction mixture was filtered through Celite and washed with EtOAc (100 mL). The crude product was purified by flash chromatography (9:1 hexanes:EtOAc) to afford **S14** as a colorless oil (0.64 g, 63%): ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.56 (td, *J* = 11.1, 7.0, 1H), 5.43 (dd, *J* = 11.1, 9.4, 1H), 4.09 (dd, *J* = 9.4, 3.3, 1H), 2.20–2.12 (m, 1H), 2.10–2.03 (m, 1H), 1.39–1.30 (m, 4H), 0.93–0.90 (m, 12H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 133.9, 129.3, 75.1, 35.2, 21.0, 27.8, 25.7, 22.6, 14.2; IR (thin film) 3391, 2956, 1872, 1465, 1363, 1038, 999 cm^{-1}; HRMS (CI/Methanol) *m* / *z* calcd for $C_{11}H_{22}O$ $(M + Na)^+$ 193.1568, found 193.1561.



(*Z*)-6-(*tert*-Butyldimethylsilyloxy)-1-phenylhex-4-en-3-ol (S15):

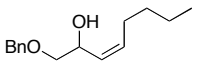
To a solution of 6-(*tert*-butyldimethylsilyloxy)-1-phenylhex-4-en-3-ol (**S9**) (0.141 g, 0.46 mmol) in MeOH (1.5 mL) was added quinoline (0.027 mL, 0.23 mmol) and Lindlar's Catalyst (4.6 mg). The reaction mixture was vigorously stirred under an atmosphere of hydrogen for 90 min. The reaction mixture was filtered through Celite and washed with MeOH (5 mL). The crude product was purified by flash chromatography (5:1 hexanes:EtOAc) to afford **S15** as a light yellow oil (0.108 g, 76%): ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.32–7.28 (m, 2H), 7.22–7.18 (m, 3H), 5.66 (tdd, *J* = 1.0, 5.3, 11.3, 1H), 5.57 (ddt, 1.3, 7.9, 11.3, 1H), 4.46–4.41 (m, 1H), 4.26 (ddd, *J* = 1.5, 6.2, 13.4, 1H), 4.18 (ddd, *J* = 1.3, 5.5, 13.4, 1H), 2.77–2.66 (m, 2H), 2.18 (d, *J* = 3.5, 1H), 1.98–1.90 (m, 1H), 1.82–1.75 (m, 1H), 0.91 (s, 9H), 0.08

(s, 6H); ^{13}C NMR (125 MHz) 142.1, 134.3, 131.2, 128.66, 128.59, 126.1, 67.4, 59.8, 39.0, 31.9, 26.1, 18.5, –5.0 IR (thin film) 3368, 2929, 2857, 1456, 1255, 1082, 837, 777 cm^{-1}; HRMS (CI/Dichloromethane) *m* / *z* calcd for $C_{18}H_{30}O_2Si$ (M + Na)$^+$ 329.1913, found 329.1909.



(*Z*)-6,6-Dimethoxyhex-3-en-2-ol (S16):

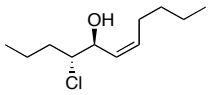
To a solution of 6,6-dimethoxyhex-3-yn-2-ol (**S10**) (0.111 g, 0.70 mmol) in MeOH (2.3 mL, 0.3 M) was added Lindlar's catalyst (7 mg). The reaction was vigorously stirred under an atmosphere of hydrogen gas and monitored by NMR. The reaction mixture was filtered through Celite and washed with MeOH (2 x 5 mL). The crude product was purified by flash chromatography (3:4 pentane:ether) to afford **S16** as a colorless oil (0.080 g, 71%): ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.63 (ddt, *J* = 1.4, 8.2, 10.9, 1H), 5.48 (dddd, *J* = 1.1, 6.9, 8.7, 10.9, 1H), 4.62–4.56 (m, 1H), 4.37 (dd, *J* = 4.5, 6.3, 1H), 3.37 (s, 3H), 3.36 (s, 3H), 2.57 (dddd, *J* = 1.4, 6.3, 8.7, 14.3, 1H), 2.36 (dddd, *J* = 1.4, 4.5, 6.9, 14.3, 1H), 2.24 (s, 1H), 1.26 (d, *J* = 6.3, 3H); ^{13}C NMR (125 MHz) δ 137.5, 125.2, 104.0, 63.3, 54.4, 53.2, 31.8, 23.1; IR (thin film) 3422, 2970, 2932, 2833, 1736, 1366, 1192, 1123, 1062 cm^{-1}; HRMS (CI/Methanol) *m* / *z* calcd for $C_8H_{16}O_3$ (M + Na)$^+$ 183.0997, found 183.0992.



(*Z*)-1-Benzyloxyoct-3-en-2-ol (S17):

To a solution of 1-benzyloxyoct-3-yn-2-ol (**S11**) (0.120 g, 0.52 mmol) in MeOH (1.7 mL) was added quinoline (0.06 mL, 0.52 mmol) and Lindlar's catalyst (0.0052 g). The reaction mixture was vigorously stirred under an atmosphere of hydrogen for 30 min. The reaction mixture was filtered through Celite and washed with MeOH (10 mL). The crude product was purified by flash chromatography (5:1 hexanes:EtOAc) to afford **S17** as a light yellow oil (0.101 g, 83 %): ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.39–7.34 (m, 4H), 7.33–7.30 (m, 1H), 5.58 (tdd, *J* = 1.0, 7.5, 11.0, 1H), 3.37 (ddt, *J* = 1.5, 8.5, 11.0, 1H), 4.68 (td, *J* = 2.7, 8.4, 1H), 4.59 (s, 2H), 3.47 (dd, *J* = 3.3, 9.7, 1H), 3.38 (dd, *J* = 8.5, 9.6, 1H), 2.47 (s, 1H), 2.14–2.04 (m, 2H), 1.38–1.28 (m, 4H), 0.90 (t, *J* = 7.1, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 138.1, 134.4, 128.7, 127.99, 127.96, 127.8, 74.22, 73.5, 67.1, 31.9, 27.8, 22.5, 14.1; IR (neat) 3422,

2957, 2928, 2859, 1454, 1106, 1075, 736, 698 cm^{-1}; HRMS (CI/Dichloromethane) *m* / *z* calcd for $C_{15}H_{22}O_2$ $(M + Na)^+$ 257.1518, found 257.1516.

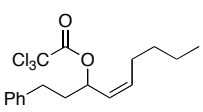


(*Z*)-(4*R,5*S**)-4-Chloroundec-6-en-5-ol (S18):**

To a solution of (4*R**,5*S**)-4-chloroundec-6-yn-5-ol (**S12**) (0.248 g, 1.23 mmol) in MeOH (4.0 mL) was added Lindlar's catalyst (0.121 g). The reaction mixture was stirred vigorously under an atmosphere of hydrogen and monitored by ^{1}H NMR. After 90 min, additional Lindlar's catalyst (0.121 g) was added, and the reaction was further monitored. Upon completion, the reaction mixture was filtered through Celite and washed with MeOH (2 x 10 mL). The crude product was filtered through a plug of silica gel (9:1 hexanes:EtOAc) to afford **S18** as a light yellow oil (0.20 g, 80%): ^{1}H NMR (500 MHz) δ 5.66 (tdd, *J* = 0.9, 7.6, 10.2, 1H), 5.52 (ddt, *J* = 1.6, 8.5, 10.9, 1H), 4.56 (dddd, *J* = 1.0, 4.1, 6.2, 8.3, 1H), 4.06 (ddd, *J* = 3.8, 5.3, 8.8, 1H), 2.19–2.06 (m, 2H), 2.00 (d, *J* = 6.2, 1H), 1.72–1.58 (m, 3H), 1.46–1.31 (m, 5H), 0.94 (t, *J* = 7.1, 3H), 0.92 (*J* = 7.0, 3H); ^{13}C NMR (125 MHz) δ 135.6, 127.2, 70.5, 68.4, 35.3, 31.9, 28.1, 22.6, 20.1, 14.2, 13.7; IR (thin film) 3392, 2960, 2932, 2874, 1466, 1031 cm^{-1}; HRMS (CI/Dichloromethane) *m* / *z* calcd for $C_{11}H_{21}ClO$ $(M + Na)^+$ 227.1179, found 227.1175.

IVc. General Procedure for Trichloroacetylation (unoptimized):

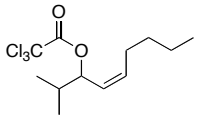
To a cooled (0 ºC) solution of the substrate (1 equiv.) in THF (0.05 M) was added pyridine (2 equiv.) followed by trichloroacetic anhydride (1.2 equiv.). The solution was warmed to ambient temperature and stirred for 30 min before diluting with hexanes (10 mL). The reaction mixture was rinsed with water (4 x 3 mL), saturated aqueous $NaHCO_3$ (3 mL), brine (3 mL), dried over anhydrous Na_2SO_4, and concentrated *in vacuo*.



(*Z*)-3-Trichloroacetoxy-1-phenylnon-4-ene (10a):

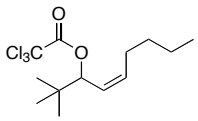
The general procedure for trichloroacetylation was used with (*Z*)-1-phenylnon-4-en-3-ol (**S2**) (0.13 g, 0.60 mmol) as the substrate. The crude product was immediately purified by flash

chromatography (98:2 hexanes:EtOAc) to afford **10a** as a colorless oil (0.13 g, 60%): ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.32–7.29 (m, 2H), 7.23–7.18 (m, 3H), 5.71 (tdd, *J* = 7.6, 10.9, 0.7, 1H), 5.67–5.62 (m, 1H), 5.43 (ddt, *J* = 10.9, 9.3, 1.6, 1H) 2.78–2.66 (m, 2H), 2.23–2.10 (m, 3H), 2.00–1.92 (m, 1H), 1.38-1.27 (m, 4H), 0.89 (t, *J* = 6.9, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 161.4, 140.9, 137.2, 128.8, 128.5, 126.4, 125.6, 90.5, 76.15, 36.3, 31.7, 31.4, 27.9, 22.5, 14.1; IR (thin film) 3027, 2958, 2929, 2859, 1764, 1455, 1242, 971, 826, 681 cm^{-1}; HRMS (CI/dichloromethane) *m* / *z* calcd for $C_{17}H_{21}Cl_3O_2$ $(M + Na)^+$ 385.0505, found 385.0504.



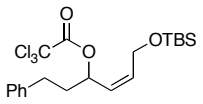
(*Z*)-3-(Trichloroacetate)-2-methylnon-4-ene (10b):

The general procedure for trichloroacetylation was used with (*Z*)-2-methyl-non-4-en-3-ol (**S13**) (0.048 g, 0.31 mmol) to afford **10b** as a colorless oil that was used without purification (0.075 g, 80%): ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.73 (td, *J* = 7.6, 10.0, 1H), 5.43-5.35 (m, 2H), 2.27-2.21 (m, 1H), 2.19-2.14 (m, 1H), 2.04-1.97 (m, 1H), 1.41-1.32 (m, 4H), 1.00 (d, *J* = 6.8, 3H), 0.96 (d, *J* = 6.9, 3H), 0.92 (m, *J* = 7.0, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 161.2, 137.5, 124.0, 90.5, 81.1, 32.4, 31.6, 27.8, 22.4, 18.1, 18.0, 14.0; IR (thin film) 2963, 2931, 2874, 1763, 1468, 1244, 975, 908, 838, 828, 681 cm^{-1}; Multiple attempts to find parent peak in MS failed due to fragmentation.



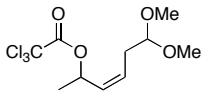
(*Z*)-3-(Trichloroacetoxy)-2,2-dimethylnon-4-ene (10c):

The general procedure for trichloroacetylation was used with (*Z*)-2,2-dimethylnon-4-en-3-ol (**S14**) (0.10 g, 0.60 mmol) to afford **10c** as a colorless oil that was used without purification (0.17 g, 95 %): ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.78 (td, *J* = 7.5, 9.2, 1H), 5.43–5.34 (m, 2H), 2.30–2.24 (m, 1H), 2.20–2.12 (m, 1H), 1.44–1.33 (m, 4H), 1.00 (s, 9H), 0.94 (t, *J* = 6.6, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 161.3, 138.2, 122.6, 90.7, 83.2, 35.4, 31.7, 28.0, 25.7, 22.6, 14.2; IR (thin film) 2961, 2933, 2874, 1766, 1467, 1368, 1247, 977, 917, 841, 826, 681 cm^{-1}; Multiple attempts to find parent peak in MS failed due to fragmentation.



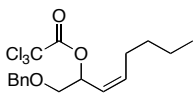
(*Z*)-6-(*tert*-Butyldimethylsilyloxy)-3-trichloroacetoxy-1-phenylhex-4-ene (10d):

The general procedure for trichloroacetylation was used with (*Z*)-6-(*tert*-butyldimethylsilyloxy)-1-phenylhex-4-en-3-ol (**S15**) (0.108 g, 0.35 mmol). The crude product was purified using a short column of silica (9:1 hexanes:EtOAc) to afford **10d** as a light yellow oil (0.143 g, 90%): ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.32–7.29 (m, 2H), 7.23–7.18 (m, 3H), 5.82 (td, *J* = 5.8, 11.2, 1H), 5.64 (td, *J* = 8.7, 5.7, 1H), 5.48 (ddt, *J* = 1.6, 11.0, 12.7, 1H), 4.30 (dd, *J* = 1.7, 5.9, 2H), 2.79–2.67 (m, 2H), 2.23–2.15 (m, 1H), 2.02–1.95 (m, 1H), 0.91 (s, 9H), 0.08 (s, 3H), 0.07 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 161.4, 140.7, 136.0, 128.8, 128.5, 126.5, 126.0, 90.4, 76.0, 59.8, 36.2, 31.3, 26.1, 18.5, –5.0, –5.1; IR (thin film) 3055, 2998, 2858, 1765, 1242, 1091, 837 cm^{-1}; HRMS (CI/Dichloromethane) *m* / *z* calcd for $C_{20}H_{29}Cl_3O_3Si$ $(M + Na)^+$ 473.0849, found 473.0838.



(*Z*)-6,6-Dimethoxy-2-trichloroacetoxy-3-hexene (10e):

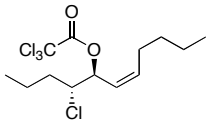
The general procedure for trichloroacetylation was used with (*Z*)-6,6-dimethoxyhex-3-en-2-ol (**S16**) (0.060 g, 0.37 mmol). The crude product was purified by flash chromatography (9:1 hexanes:EtOAc) to afford a light yellow oil (0.094 g, 83%): ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.74 (ddd, *J* = 6.4, 8.9, 12.8, 1H), 5.68 (td, *J* = 7.5, 11.0, 1H), 5.57 (ddt, *J* = 1.3, 9.2, 10.8, 1H), 4.40 (t, *J* = 5.6, 1H), 3.36 (s, 6H), 2.53 (ddt, *J* = 1.3, 5.8, 6.8, 2H), 1.46 (d, *J* = 6.4, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 151.4, 129.7, 129.4, 103.9, 90.4, 73.4, 53.8, 53.2, 31.9, 20.3; IR (thin film) 2988, 2936, 1761, 1246, 1122, 1075 cm^{-1}; HRMS (CI/Dichloromethane) *m* / *z* calcd for $C_{10}H_{15}Cl_3O_4$ $(M + Na)^+$ 326.9933, found 326.9934.



(*Z*)-1-Benzyloxy-2-trichloroacetoxy-3-octene (10f):

The general procedure for trichloroacetylation was used with (*Z*)-1-benzyloxyoct-3-en-2-ol (**S17**) (0.055 g, 0.24 mmol). The crude product was purified by flash chromatography (96:4 hexanes:EtOAc) to afford a yellow oil (0.025 g, 27%): ^{1}H NMR (500 MHz); 7.37–7.33 (m, 4H), 7.32–7.28 (m, 1H), 5.89 (dddd, *J* = 0.94, 3.4, 7.9, 9.0, 1H), 5.76 (tdd, *J* = 0.87, 7.6, 10.9, 1H), 5.40 (ddt, 1.6, 9.3, 10.9, 1H), 4.61 (dd, *J* = 4.6, 12.2, 2H), 3.72 (dd, *J* = 7.8, 11.2, 1H), 3.60 (dd, *J* = 3.5, 11.2, 1H),

2.26–2.15 (m, 2H), 1.42–1.29 (m, 4H), 0.91 (t, J = 6.9, 3H); ^{13}C NMR (125 MHz) δ 161.4, 138.7, 137.9, 128.7, 128.0, 127.8, 122.1, 90.4, 75.4, 73.5, 71.2, 31.7, 28.1, 22.5, 14.1; IR (thin film) 2959, 2930, 2861, 1764, 1242 cm^{-1}; HRMS (CI/Dichloromethane) m / z calcd for $C_{17}H_{21}Cl_3O_3$ $(M + Na)^+$ 401.0454, found 401.0448.



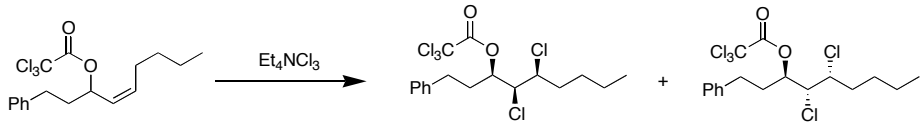
(*Z*)-(4*R,5*S**)-4-Chloro-5-trichloroacetoxyundec-6-ene (14):**

The general procedure for trichloroacetylation was used with (*Z*)-(4*R**,5*S**)-4-chloroundec-6-en-5-ol (**S18**) (0.083 g, 0.40 mmol). The crude product was purified by flash chromatography (98:2 hexanes:EtOAc) to afford **14** as a light yellow oil (0.107 g, 76%): ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.86 (td, J = 7.9, 11.0, 1H), 5.76 (ddd, J = 0.74, 4.8, 9.3, 1H), 5.53 (ddt, J = 1.6, 10.9, 12.6, 1H), 4.10 (td, J = 4.2, 9.4, 1H), 2.28–2.15 (m, 2H), 1.81–1.71 (m, 2H), 1.69–1.62 (m, 1H), 1.50–1.32 (m, 5H), 0.96 (t, J = 7.4, 3H), 0.93 (t, J = 7.1, 3H); ^{13}C NMR (125 MHz, C_6D_6) δ 161.1, 139.8, 121.8, 90.6, 77.3, 62.6, 57.6, 31.6, 28.3, 22.6, 19.7, 14.1, 13.5; IR (thin film) 2958, 2926, 2875, 1768, 1462, 1239, 974, 839 cm^{-1}; Multiple attempts to find parent peak in MS failed due to fragmentation.

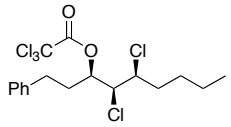
IVd. General Procedure for Dichlorination

To a cooled (–90 ºC) solution of the trichloroacetylated allylic alcohol in CH_2Cl_2 (0.3 M) was added a solution of tetraethylammonium trichloride in CH_2Cl_2 (10 equiv., 2.0 M) over 30 min. The reaction mixture was stirred for an additional 90 min at –90 ºC, then warmed to –78 ºC and stirred for 2 h before warming to 0 ºC over 15 minutes and quenching with a 1:1 solution of saturated aqueous $NaHCO_3$/saturated aqueous $Na_2S_2O_3$ (2 mL per mmol of tetraethylammonium trichloride). The solution was diluted with CH_2Cl_2 (2 mL) and water (2 mL). The phases were separated and the aqueous phase was extracted with CH_2Cl_2 (4 x 2 mL) and ether (2 mL). The combined organic extracts were washed with brine (2 mL), dried over anhydrous Na_2SO_4, and concentrated *in vacuo*. The crude reaction products were analyzed by ^{1}H NMR to determine the diastereomeric ratio.

4,5-Dichloro-3-trichloroacetoxy-1-phenylnonane (11a):

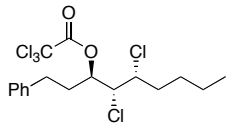


The general procedure was followed using (*Z*)-3-trichloroacetoxy-1-phenylnon-4-ene (**10a**) (0.025 g, 0.07 mmol). The crude reaction mixture consisted of a 8.3:1 ratio of diastereomers. The crude product was purified by flash chromatography (98:2 hexanes:EtOAc) to afford a colorless oil (0.024 g total, 82%, 9.2:1.0 *d.r.*). Diastereomers from a previous reaction were separated by flash chromatography (98:2 hexanes:EtOAc) using high-performance silica gel (40-63 μm, EM Science) and characterized:



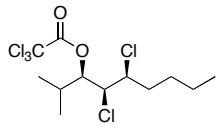
(3*R,4*S**,5*S**)-4,5-Dichloro-3-trichloroacetoxy-1-phenylnonane (11a) (major):**

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.32 (m, 2H), 7.24 (m, 1H), 7.20 (m, 2H), 5.46 (ddd, *J* = 8.4, 7.2, 3.9, 1H), 4.21 (dd, *J* = 7.1, 2.9, 1H), 4.07 (td, *J* = 7.0, 2.9, 1H), 2.78–2.71 (m, 2H), 2.27–2.20 (m, 1H), 2.16–2.08 (m, 1H), 1.86 (appar. q, *J* = 7.1, 2H), 1.50–1.43 (m, 1H), 1.40–1.29 (m, 3H), 0.92 (t, *J* = 7.0, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 161.2, 140.2, 128.9, 128.5, 126.8, 90.1, 80.1, 65.5, 61.7, 35.8, 33.2, 31.1, 29.9, 28.7, 22.3, 14.1; IR (thin film); HRMS (CI/methanol) *m* / *z* calcd for $C_{17}H_{21}Cl_5O_2$ $(M + Na)^+$ 454.9882, found 454.9891.



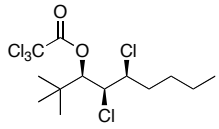
(3*S,4*S**,5*S**)-4,5-Dichloro-3-trichloroacetoxy-1-phenylnonane (minor) (ca. 90% purity):**

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.33–7.30 (m, 2H), 7.24–7.19 (m, 3H), 5.38 (ddd, *J* = 3.1, 7.1, 9.5, 1H), 4.27 (ddd, *J* = 2.0, 5.2, 7.6, 1H), 4.20 (dd, *J* = 2.1, 9.2, 1H), 2.79–2.72 (m, 2H), 2.44–2.37 (m, 1H), 2.28–2.20 (m, 1H), 2.00–1.93 (m, 1H), 1.84–1.77 (m, 1H), 1.53–1.44 (m, 1H), 1.42–1.30 (m, 3H), 0.93 (t, *J* = 7.2, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 160.6, 140.6, 128.9, 128.5, 126.6, 90.0, 79.0, 63.8, 61.0, 35.9, 33.2, 30.6, 28.7, 22.2, 14.1; IR (thin film) 2959, 2916, 2864, 1774, 1231, 977 cm^{-1}.



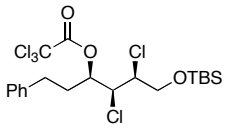
(3*R,4*S**,5*S**)-4,5-Dichloro-3-trichloroacetoxy-2-dimethylnonane (11b):**

The general dichlorination procedure was followed using (*Z*)-3-(trichloroacetoxy)-2-methylnon-4-ene (**10b**) (0.035 g, 0.012 mmol). The crude product consisted of a 7.0:1.0 mixture of diastereomers. The crude product was purified by flash chromatography (96:4 hexanes:EtOAc) to afford **11b** as a colorless oil (7.5:1.0 *d.r.*, 0.034 g, 77%). Data for major isomer: ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.35 (dd, *J* = 3.3, 7.9, 1H), 4.27 (dd, *J* = 2.7, 7.9, 1H), 4.08 (td, *J* = 2.6, 6.9, 1H), 2.29-2.24 (m, 1H), 1.92 (app. q, *J* = 7.4, 2H), 1.52-1.48 (m, 1H), 1.43-1.34 (m, 3H), 1.06 (d, *J* = 6.9, 3H), 1.00 (d, *J* = 6.9, 3H), 0.94 (t, *J* = 7.1, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 161.5, 90.3, 84.0, 64.7, 61.5, 36.0, 29.7, 28.7, 22.4, 19.9, 15.5, 14.1; IR (thin film) 2964, 2876, 1770, 1468, 1241, 994, 825, 679 cm^{-1}; Multiple attempts to find parent peak in MS failed due to fragmentation.



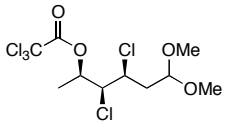
(3*R,4*S**,5*S**)-4,5-Dichloro-3-trichloroacetatoxy-2,2-dimethylnonane (11c):**

The general dichlorination procedure was followed using (*Z*)-3-(trichloroacetoxy)-2,2-dimethylnon-4-ene (**10c**) (0.036 g, 0.12 mmol). The crude product consisted of an 8.6:1 mixture of diastereomers. The crude product was purified by flash chromatography (48:2 hexanes:EtOAc) to afford **11c** as a colorless oil (0.036 g, 77%, 8.6:1.0 *d.r.*). Characterized as a 8.7:1.0 mixture of diastereomers. Data for major isomer: ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.26 (d, *J* = 1.5, 1H), 4.42 (dd, *J* = 1.5, 3.5, 1H), 4.05 (td, *J* = 3.3, 10.5, 1H), 2.08–1.96 (m, 1H), 1.61–1.50 (m, 2H), 1.39–1.29 (m, 3H), 1.09 (s, 9H), 0.91 (t, *J* = 7.0, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) 160.9, 90.2, 80.7, 64.9, 60.4, 36.7, 32.2, 29.0, 26.3, 22.0, 13.8; IR (thin film) 2962, 2974, 1769, 1469, 1372, 1246, 984, 845, 826, 697 cm^{-1}; Multiple attempts to find parent peak in MS failed due to fragmentation.



(3*R,4*S**,5*S**)-6-(*tert*-Butyldimethylsilyloxy)-4,5-dichloro-3-trichloroacetoxy-1-phenylhexane (11d):**

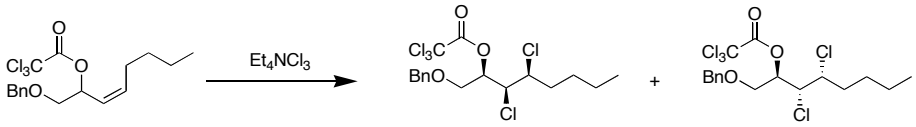
The general dichlorination procedure was followed using (*Z*)-6-(*tert*-butyldimethylsilyloxy)-3-trichloroacetoxy-1-phenylhex-4-ene (**10d**) (0.053 g, 0.12 mmol). The crude product consisted of a 10.2:1.0 mixture of diastereomers. The crude product was purified by flash chromatography (98:2 hexanes:EtOAc) to afford **11d** as a glass [0.005 g major (12%), 0.022 g mixture (57%), 70% total, combined 10.9:1.0 *d.r.*]. Data for major isomer: ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.33–7.30 (m, 2H), 7.25–7.22 (m, 1H), 7.20–7.19 (m, 2H), 5.43 (ddd, *J* = 3.8, 7.4, 8.6, 1H), 4.56 (dd, *J* = 2.4, 7.3, 1H), 4.14 (ddd, *J* = 2.4, 5.2, 7.8, 1H), 3.86 (dd, *J* = 8.6, 10.1, 1H), 2.80–2.69 (m, 2H), 2.25–2.18 (m, 2H), 2.17–2.10 (m, 1H), 0.90 (s, 9H), 0.11 (s, 3H), 0.10 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 161.4, 140.2, 128.9, 128.5, 126.7, 90.1, 80.2, 64.4, 61.5, 59.8, 33.2, 31.1, 27.0, 18.4, –5.2, –5.3; IR (thin film) 2955, 2930, 2858, 1772, 1472, 1236, 1101, 839; HRMS (CI/Dichloromethane) *m* / *z* calcd for $C_{20}H_{29}Cl_5O_3Si$ $(M + Na)^+$ 543.0226, found 543.0226.



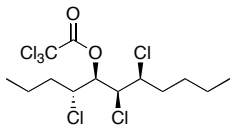
(2*S,3*R**,4*R**)-3,4-dichloro-6,6-dimethoxy-2-trichloroacetoxyhexane (11e):**

The general dichlorination procedure was used with (*Z*)-6,6-dimethoxy-2-trichloroacetoxy-3-hexene (**10e**) (0.019 g, 0.063 mmol). The crude product consisted of an 8.3:1.0 mixture of diastereomers. The crude product was immediately purified by flash chromatography (6:1 pentane:ether) to afford **11e** as a glass (single diastereomer, 0.019 g, 78%): ^{1}H NMR (500 MHz) δ 5.42–5.36 (m, 1H), 4.59 (dd, *J* = 4.1, 7.0, 1H), 4.32 (ddd, *J* = 2.7, 4.9, 7.7, 1H), 4.18 (dd, *J* = 2.7, 7.5, 1H), 3.41 (s, 3H), 3.38 (s, 3H), 2.25–2.16 (m, 2H), 1.50 (d, *J* = 6.4, 3H); ^{13}C NMR (125 MHz) δ 161.0, 102.2, 89.9, 77.7, 66.6, 57.6, 54.7, 53.8, 39.4, 17.1; IR (thin film) 2938, 2836, 1769, 1246, 1127, 1061 cm^{-1}; HRMS (CI/Dichloromethane) *m* / *z* calcd for $C_{10}H_{15}Cl_5O_4$ $(M + Na)^+$ 396.9311, found 396.9309.

(3*R,4*S**,5*S**)-1-benzyloxy-3,4-dichloro-2-trichloroacetoxyoctane (11f):**



The general dichlorination procedure was followed using (*Z*)-1-benzyloxy-2-trichloroacetoxy-3-octene (**10f**) (0.019 g, 0.049 mmol). The crude product consisted of a 4.4:1.0 mixture of diastereomers. The crude product was purified by flash chromatography (98:2 hexanes:EtOAc) to afford **11f** as a light yellow oil (4.4:1.0 *d.r.*, 0.015 g, 67%). Characterized as a 4.4:1.0 mixture of diastereomers: ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38–7.31 (m, 6.4H), 5.43 (td, *J* = 2.6, 6.5, 1H), 5.30 (ddd, *J* = 1.7, 2.6, 8.2, 0.23H), 4.62–4.54 (m, 3.8H), 4.32 (ddd, *J* = 1.6, 4.2, 5.9, 0.23H), 4.14 (td, *J* = 2.0, 5.8, 1H), 3.99 (dd, *J* = 2.7, 9.8, 0.22H), 3.87 (dd, *J* = 3.0, 9.8, 1.26H, contains unresolvable minor diastereomer peak), 3.76 (dd, *J* = 2.4, 9.8, 1H), 2.01–1.97 (m, 0.32H), 1.91 (app. q, *J* = 6.0, 2H), 1.87–1.82 (m, 0.21H), 1.53–1.43 (m, 1.4H), 1.42–1.31 (m, 4.3H), 0.93 (t, *J* = 6.1, 4.1H); ^{13}C NMR (125 MHz, $CDCl_3$) (major diastereomer) δ 161.1, 137.0, 128.6, 128.2, 127.9, 89.7, 79.8, 73.8, 67.4, 62.8, 61.1, 35.8, 28.4, 22.1, 13.9; IR (thin film) 2959, 2930, 2872, 1770, 1455, 1236; HRMS (CI/Dichloromethane) *m* / *z* calcd for $C_{17}H_{21}Cl_5O_3$ $(M + Na)^+$ 470.9831, found 470.9820.

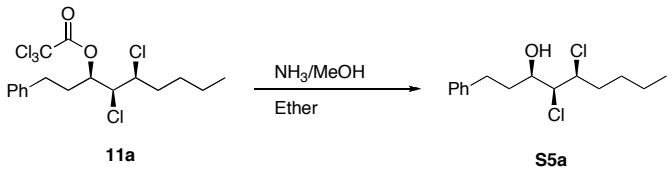


(4*R,5*S**,6*R**,7*R**)-5-trichloroacetoxy-4,6,7-trichloroundecane (15):**

The general dichlorination procedure was followed using **14** (0.028 g, 0.08 mmol). The crude product consisted of an 8.6:1.0 mixture of diastereomers. The crude product was purified by flash chromatography (HP silica, hexanes) to afford **15** as a glass (0.025 g, 76%, 10.5:1.0 *d.r.*). Characterized as a 10.5:1.0 mixture of diastereomers. Data for major isomer: ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.54 (dd, *J* = 4.1, 6.8, 1H), 4.66 (t, *J* = 4.1, 1H), 4.28 (td, *J* = 6.4, 6.7, 1H), 4.07 (td, *J* = 4.1, 9.2, 1H), 2.02–1.95 (m, 1H), 1.82–1.77 (m, 3H), 1.76–1.63 (m, 1H), 1.60–1.44 (m, 2H), 1.42–1.31 (m, 3H), 0.97–0.93 (m, 6H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 161.0, 89.7, 80.0, 63.2, 62.7, 59.9, 35.1, 34.6,

28.7, 22.2, 19.5, 14.1, 13.6; IR (thin film) 2963, 2935, 2875, 1775, 1467, 1227; HRMS (CI/Dichloromethane) *m* / *z* calcd for $C_{13}H_{20}Cl_6O_2$ $(M + Na)^+$ 440.9492, found 440.9500.

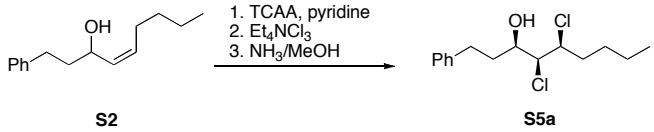
V. Trichloroacetate Ester Cleavage



11a **S5a**

To a solution of (3*R**,4*S**,5*S**)-4,5-dichloro-3-trichloroacetoxy-1-phenylnonane (**11a**) (0.013 g, 0.03 mmol) in ether (0.2 mL, 0.3 M) was added a solution of ammonia (0.03 mL, 2 M in MeOH). The resulting mixture was stirred for 60 min and diluted with ether (3 mL) and washed with water (3 mL). The aqueous phases were extracted with ether (2 mL) and the combined organic extracts were washed with 0.1 M aqueous citric acid (3 mL), saturated aqueous $NaHCO_3$ (3 mL), brine (2 mL), dried over anhydrous Na_2SO_4, filtered, and concentrated *in vacuo* to afford essentially pure **S5a** (9.4 mg, quant.).

VI. Representative 3-Step Sequence of Trichloroacetylation/Dichlorination/Detrichloroacetylation without Isolation of Intermediates



S2 **S5a**

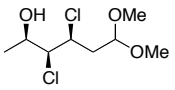
To a cooled (0 ºC) solution of (*Z*)-1-phenylnon-4-en-3-ol (**S2**) (0.55 g, 0.25 mmol) and pyridine (0.04 mL, 0.50 mmol) in THF (5.0 mL, 0.05 M) was added trichloroacetic anhydride (0.06 mL, 0.30 mmol). The resulting mixture was stirred for 20 minutes before diluting with hexanes (5 mL). The reaction mixture was washed with water (4 x 5 mL), $NaHCO_3$ (5 mL), brine (5 mL), dried over anhydrous Na_2SO_4, and concentrated *in vacuo*.

The product was dissolved in CH_2Cl_2 (0.8 mL, 0.3 M), cooled (–90 ºC), and a solution of Et_4NCl_3 (2.5 mmol, 2 M in CH_2Cl_2) was added over 30 min. The reaction mixture was stirred at –90 ºC for an

additional 90 min, then warmed to –78 ºC and stirred for 2 h. The reaction mixture was warmed to 0 ºC and quenched with a solution of saturated aqueous $NaHCO_3$ and saturated aqueous $Na_2S_2O_3$ (1:1, 4 mL). The reaction mixture was diluted with CH_2Cl_2 (5 mL) and water (2 mL). The phases were separated and the aqueous phase was extracted with CH_2Cl_2 (4 x 3 mL) and ether (3 mL). The combined organic extracts were washed with brine (3 mL), dried over anhydrous Na_2SO_4, and concentrated *in vacuo*.

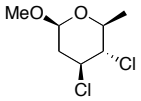
To a solution of the product in ether (0.8 mL, 0.3 M) was added NH_3 (0.5 mL, 1.0 mmol, 2 M in methanol), and the resulting mixture was stirred for 20 min. The solution was diluted with ether (5 mL) and water (2 mL). The aqueous phase was extracted with ether (2 x 2 mL) and the combined organic layer was rinsed with NH_4Cl (3 mL), brine (3 mL), dried over anhydrous Na_2SO_4, and concentrated *in vacuo*. The crude product showed an 8.9:1.0 mixture of diastereomers. The crude product was purified by flash chromatography (90:10 hexanes:EtOAc) to afford a colorless oil (0.041 g, 57 % over 3 steps, 9.3:1.0 *d.r.*).

VII. Pyranose Synthesis and ^{1}H NMR Analysis



(2*S*,3R*,4R)-3,4-Dichloro-6,6,-dimethoxy-2-hexanol (S19):**

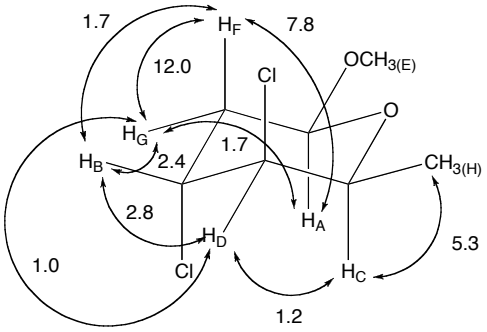
To a solution of **11e** (0.019 g, 0.049 mmol) in ether (0.32 mL, 0.15 M) was added a solution of dimethylamine (0.025 mL, 0.20 mmol, 40% by wt. in water). The solution was stirred for 40 min and diluted with ether (3 mL). The organic layer was washed with saturated aqueous NH_4Cl (1 mL), water (1 mL), brine (1 mL), dried over anhydrous Na_2SO_4, filtered, and concentrated *in vacuo*. The crude product was purified by flash chromatography (4:1 pentane:EtOAc) to afford **S19** as a colorless oil (0.0077 g, 68%): ^{1}H NMR (500 MHz, $CDCl_3$) δ 4.60 (dd, *J* = 4.0, 7.3, 1H), 4.30 (ddd, *J* = 3.0, 4.4, 7.5, 1H), 4.17–4.11 (m, 1H), 3.94 (dd, *J* = 2.9, 6.4, 1H), 3.40 (s, 3H), 3.70 (s, 3H), 2.28 (d, *J* = 4.39, 1H), 2.25–2.15 (m, 2H), 1.32 (d, *J* = 6.3, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 102.2, 72.7, 69.3, 58.8, 54.3, 53.7, 39.4, 19.9; IR (thin film) 3445, 2937, 2833, 1126, 1057 cm^{-1}; HRMS (CI/Dichloromethane) *m* / *z* calcd for $C_8H_{16}Cl_2O_3$ $(M + Na)^+$ 253.0374, found 253.0382.



(4*S*,5R*,6R)-4,5-dichloro-2-methoxy-6-methylpyran (16)**

To a solution of **S19** (0.0051 g, 0.022 mmol) in CH_2Cl_2 (0.22 mL, 0.1 M) was added a solution of TFA (0.05 mL, 40 μM in CH_2Cl_2 prepared from 0.004 mL TFA dissolved in 0.1 mL CH_2Cl_2). The solution was stirred for 4 h, adding additional TFA solution in 0.02 mL increments every hour. The reaction mixture was neutralized with saturated aqueous $NaHCO_3$ (1 mL) and diluted with CH_2Cl_2. The organic layer was washed with water (1 mL), brine (1 mL), dried over anhydrous Na_2SO_4, filtered, and concentrated *in vacuo*. The crude product consisted of a 2.8:1.0 mixture of anomers. The crude product was purified by flash chromatography (20:1 pentane:ether) to afford **16** as separate anomers (β: 0.6 mg (14%), mixture: 0.9 mg (21%), α: 0.5 mg (11%), total: 2 mg, 46%).

In order to resolve all signals and their associated coupling constants, ^{1}H NMR spectra were recorded on the 2.8:1.0 mixture of anomers in multiple solvents at 600 MHz. Representative data of the β (major) anomer is shown in the table:



Proton(s)	Shift	Multiplicity	Couplings	Solvent
A	4.72	dd	1.7, 7.8	THF-d_8 (600 MHz)
B	4.58	td	2.4, 2.7	THF-d_8 (600 MHz)
C	4.31	dq	1.2, 5.3	THF-d_8 (600 MHz)
D	4.13	td	1.3, 2.8	acetone-d_6 (600 MHz)
E	3.40	s	none	THF-d_8 (600 MHz)
F	2.27	ddd	3.0, 7.6, 11.9	benzene-d_6 (600 MHz)
G	1.72	dddd	1.0, 2.0, 2.4, 12.0	benzene-d_6 (600 MHz)
H	1.27	d	5.2	THF-d_8 (600 MHz)

VIII. References

[a]Harada, T.; Kutsuwa, E. *J. Org. Chem.* **2005**, *68*, 6716.

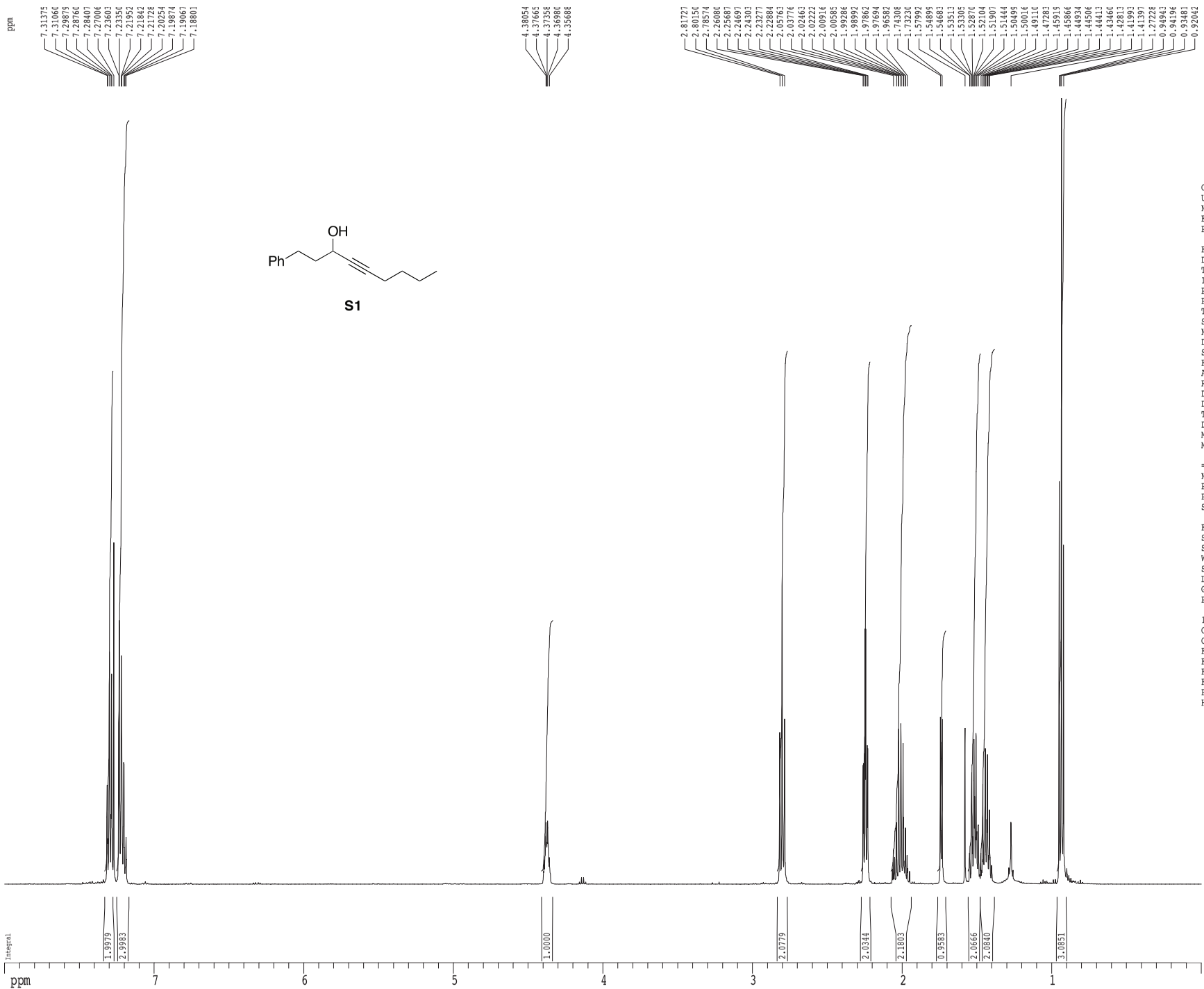
[b]Wong, U.; Cox, R. J. *Angew. Chem. Int. Ed.* **2007**, *46*, 4926.

[c]Shang, S.; Iwadare, H.; Macks, D. E.; Ambrosini, L. M.; Tan, D. S. *Org. Lett.* **2007**, *9*, 1895.

[d]Kang, D.; Britton, R. *Org. Lett.* **2007**, *9*, 5083.

Appendix. Proton NMR Spectra

1H spectrum

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FIDRES             0.098043 Hz
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RG                      128
DW                   62.400 usec
DE                    6.00 usec
TE                   298.0 K
D1                0.10000000 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

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F2 - Processing parameters
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GB                        0
PC                     0.50

1D NMR plot parameters
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CY                    15.00 cm
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F1                  4001.76 Hz
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F2                     0.00 Hz
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HZCM             175.51581 Hz/cm
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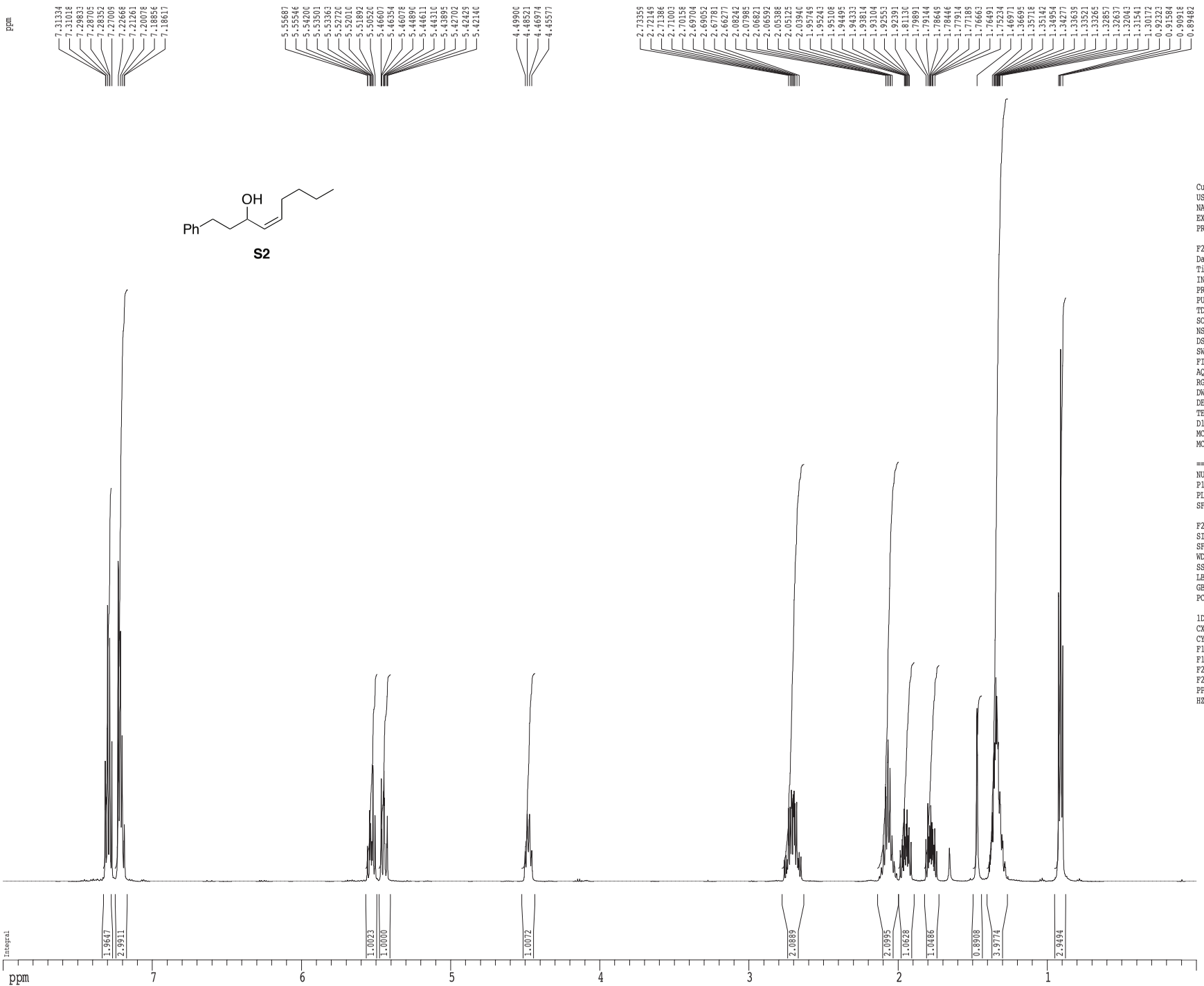
1H spectrum



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PROCNO 1

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FIDRES 0.098043 Hz
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RG 45.3
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PL1 -5.00 dB
SFO1 500.2235015 MHz

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SSB 0
LB 0.30 Hz
GB 0
PC 4.00

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F2P 0.000 ppm
F2 0.00 Hz
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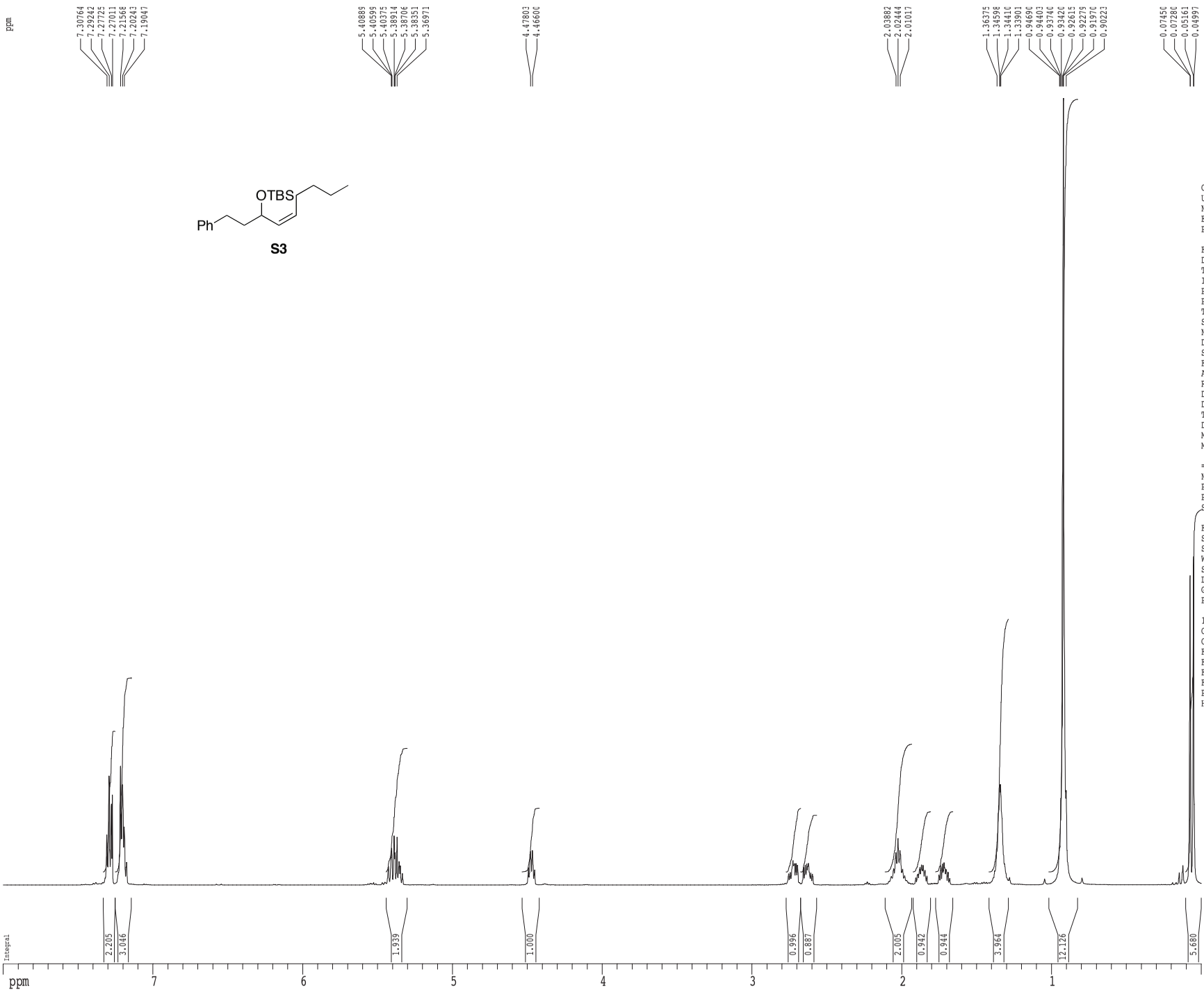
HZCM 175.51581 Hz/cm

1H spectrum



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PROCNO 1

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SOLVENT CDCl3
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FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 4
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======= CHANNEL f1 =======
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P1 8.00 usec
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F2 - Processing parameters
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SF 500.2200263 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
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CY 15.00 cm
F1P 8.000 ppm
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F2P 0.000 ppm
F2 0.00 Hz
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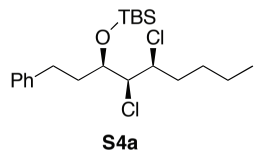
HZCM 175.51581 Hz/cm

1H spectrum



S4a

(ca 90% purity)

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EXPNO 1
PROCNO 1

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DS 2
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RG 5.7
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
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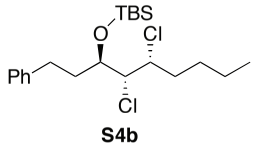
1H spectrum



S4b

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PROCNO 1

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SOLVENT CDCl3
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AQ 5.0998774 sec
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DE 6.00 usec
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CY 100.21 cm
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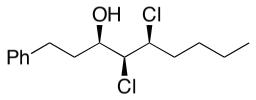
1H spectrum



S5a

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AQ 5.0998774 sec
RG 4.5
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
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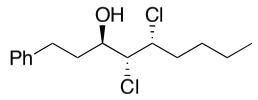
1H spectrum



S5b

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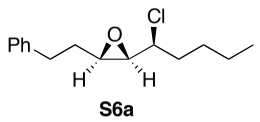
1H spectrum



S6a

(contaminated with **S6b**)

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FIDRES 0.098043 Hz
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RG 574.7
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
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MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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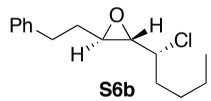
F2 0.00 Hz
PPMCM 0.35088 ppm/cm
HZCM 175.41405 Hz/cm

1H spectrum



S6b

Current Data Parameters
USER grants
NAME gs_p214_1H_DMSO
EXPNO 1
PROCNO 1

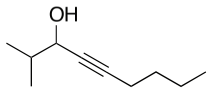
F2 - Acquisition Parameters
Date_ 20060812
Time 16.13
INSTRUM gn500
PROBHD 5 mm broadband
PULPROG zg30
TD 81728
SOLVENT DMSO
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 322.5
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 -3.00 dB
SFO1 499.9334995 MHz

F2 - Processing parameters
SI 65536
SF 499.9300117 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 8.000 ppm
F1 3999.44 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.35088 ppm/cm
HZCM 175.41405 Hz/cm

1H spectrum



S7

Current Data Parameters
USER grants
NAME GS2_004_p
EXPNO 1
PROCNO 1

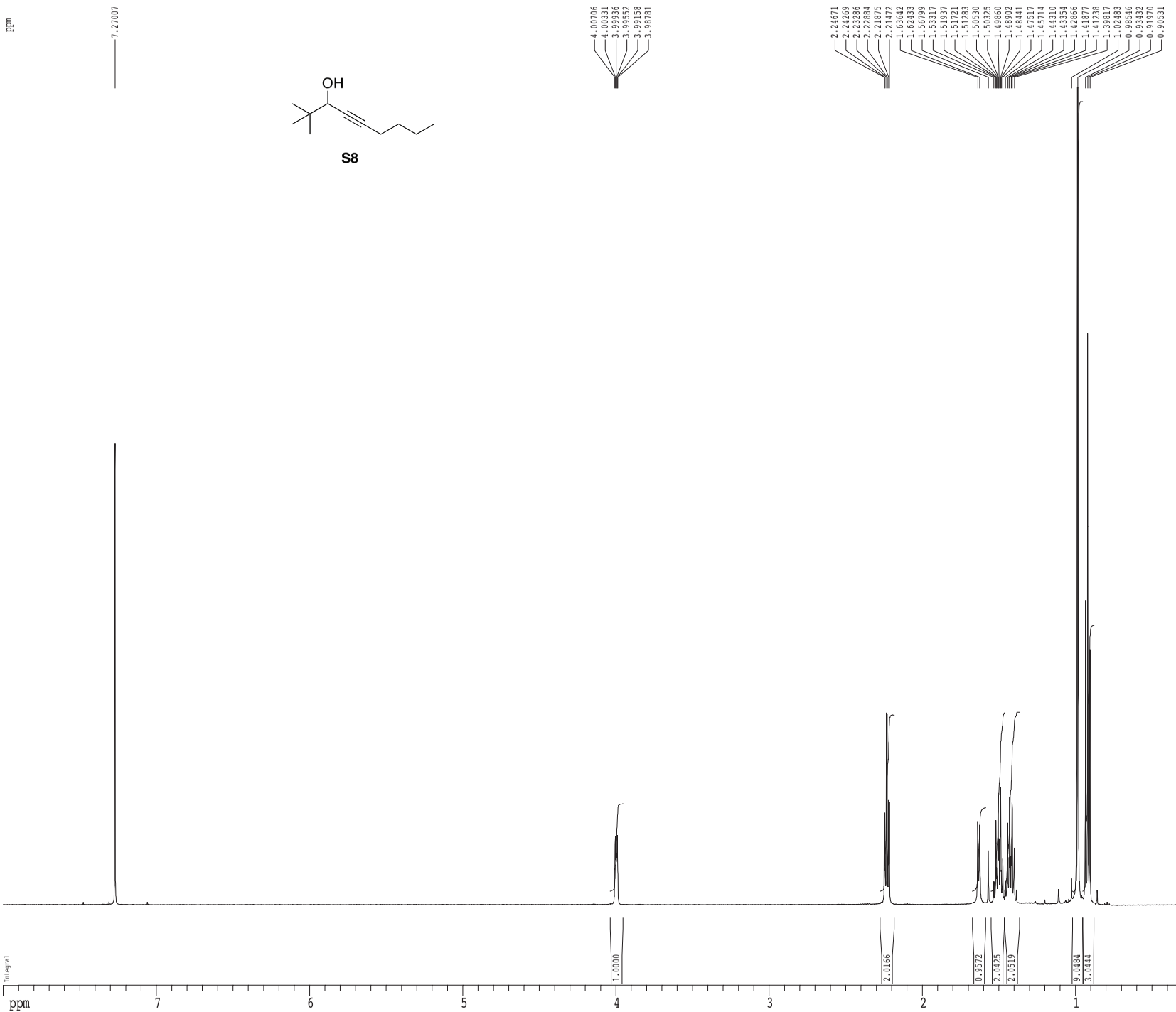
F2 - Acquisition Parameters
Date_ 20060919
Time 16.20
INSTRUM gn500
PROBHD 5 mm broadband
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 256
DW 62.400 usec
DE 6.00 usec
TE 292.8 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 12.30 usec
PL1 -3.00 dB
SFO1 499.9334995 MHz

F2 - Processing parameters
SI 65536
SF 499.9300253 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 22.80 cm
CY 10.24 cm
F1P 8.000 ppm
F1 3999.44 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.35088 ppm/cm
HZCM 175.41405 Hz/cm

1H spectrum



Current Data Parameters

USER	grants
NAME	GS2_003_p
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20060919
Time	16.13
INSTRUM	gn500
PROBHD	5 mm broadband
PULPROG	zg30
TD	81728
SOLVENT	CDCl3
NS	16
DS	2
SWH	8012.820 Hz
FIDRES	0.098043 Hz
AQ	5.0998774 sec
RG	456.1
DW	62.400 usec
DE	6.00 usec
TE	292.8 K
D1	0.10000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

======= CHANNEL f1 =======

NUC1	1H
P1	12.30 usec
PL1	-3.00 dB
SFO1	499.9334995 MHz

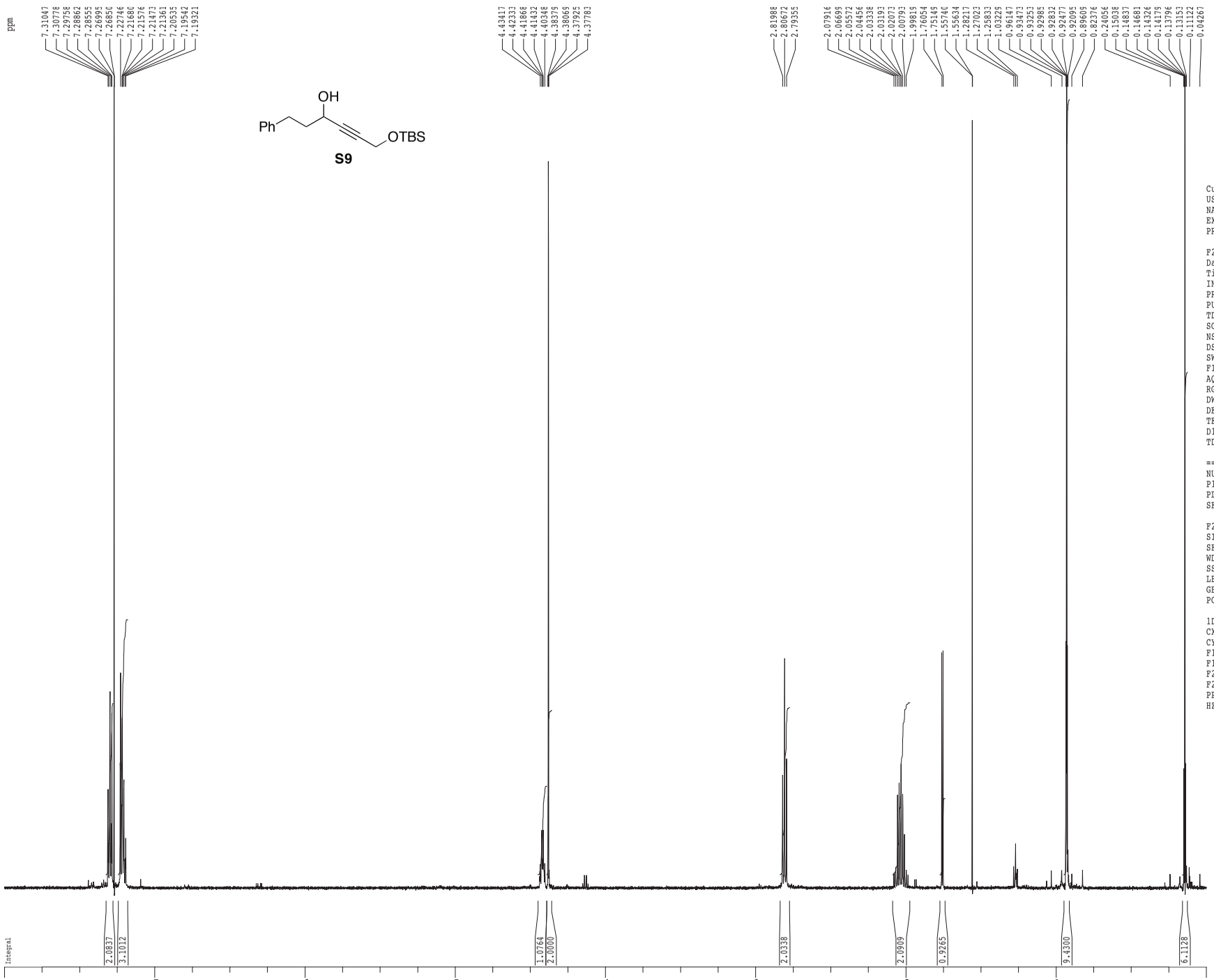
F2 - Processing parameters

SI	65536
SF	499.9300251 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	4.00

1D NMR plot parameters

CX	22.80 cm
CY	68.59 cm
F1P	8.000 ppm
F1	3999.44 Hz
F2P	0.000 ppm
F2	0.00 Hz
PPMCM	0.35088 ppm/cm
HZCM	175.41405 Hz/cm

1H spectrum



Current Data Parameters
USER grants
NAME GS3_242_p
EXPNO 2
PROCNO 1

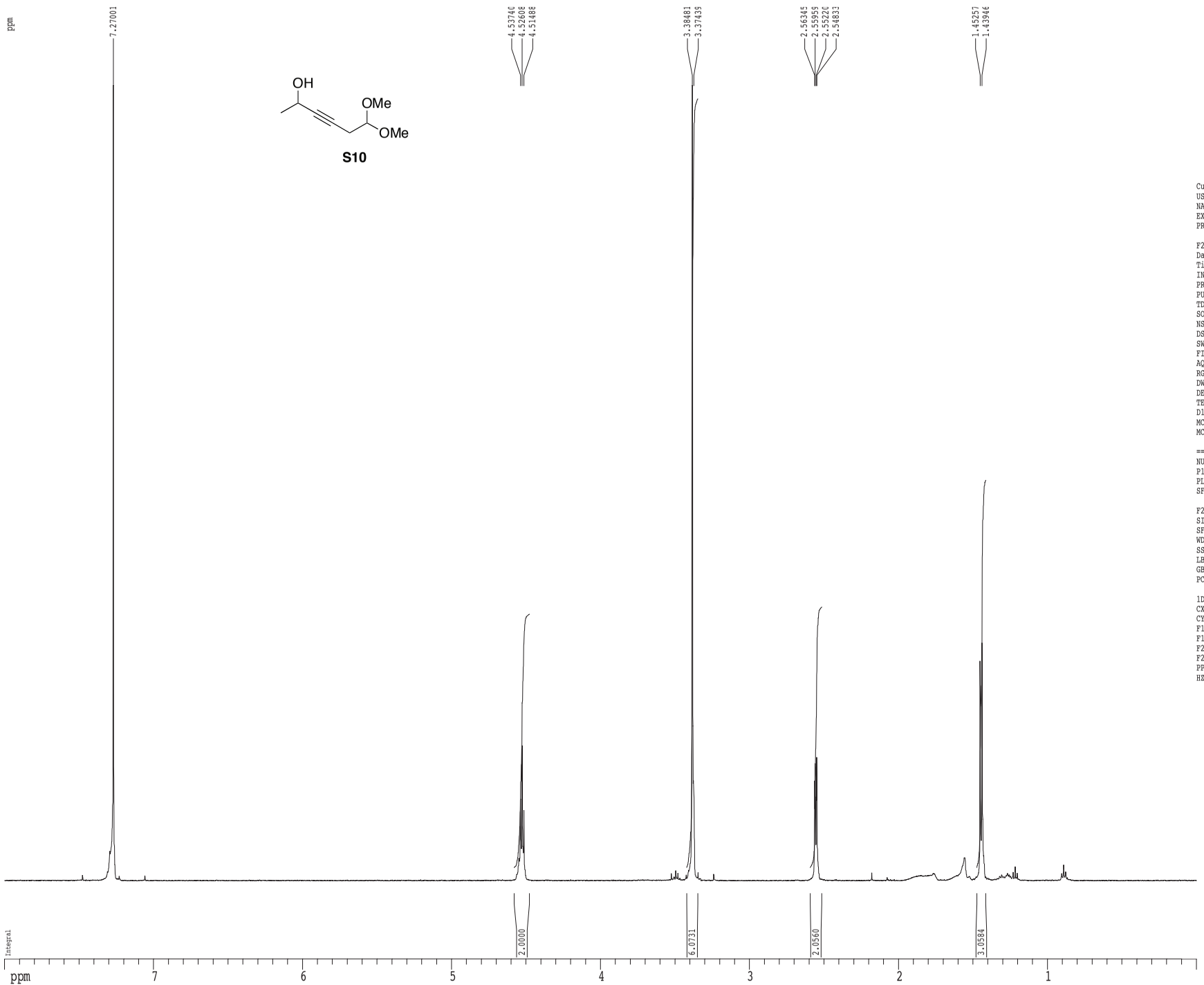
F2 - Acquisition Parameters
Date_ 20080221
Time 11.51
INSTRUM av600
PROBHD 5 mm TBI 1H/13
PULPROG zg30
TD 97938
SOLVENT CDCl3
NS 8
DS 2
SWH 9615.385 Hz
FIDRES 0.098178 Hz
AQ 5.0928259 sec
RG 362
DW 52.000 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
TD0 1

======= CHANNEL f1 =======
NUC1 1H
P1 8.00 usec
PL1 -1.00 dB
SFO1 600.1342009 MHz

F2 - Processing parameters
SI 65536
SF 600.1300294 MHz
WDW GM
SSB 0
LB -0.30 Hz
GB 0.6005
PC 4.00

1D NMR plot parameters
CX 22.80 cm
CY 120.28 cm
F1P 8.000 ppm
F1 4801.04 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.35088 ppm/cm
HZCM 210.57195 Hz/cm

1H spectrum



Current Data Parameters
USER grants
NAME GS3_265_repurified
EXPNO 1
PROCNO 1

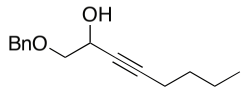
F2 - Acquisition Parameters
Date_ 20080313
Time 16.56
INSTRUM gn500
PROBHD 5 mm broadband
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 2048
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -3.00 dB
SFO1 499.8284988 MHz

F2 - Processing parameters
SI 65536
SF 499.8250256 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 22.80 cm
CY 23.46 cm
F1P 8.000 ppm
F1 3998.60 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.35088 ppm/cm
HZCM 175.37720 Hz/cm

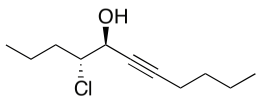
1H spectrum



S11


```
Current Data Parameters
USER                grants
NAME          GS2_163_C13
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20070328
Time                20.38
INSTRUM           cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG              zg30
TD                  81728
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8012.820 Hz
FIDRES           0.098043 Hz
AQ               5.0998774 sec
RG                      5
DW                 62.400 usec
DE                  6.00 usec
TE                298.0 K
D1              0.10000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                  8.00 usec
PL1                 1.60 dB
SFO1          500.2235015 MHz

F2 - Processing parameters
SI                  65536
SF            500.2200267 MHz
WDW                    EM
SSB                     0
LB                  0.30 Hz
GB                      0
PC                  4.00

1D NMR plot parameters
CX                 22.80 cm
CY                 15.00 cm
F1P                8.000 ppm
F1               4001.76 Hz
F2P                0.000 ppm
F2                 0.00 Hz
PPMCM           0.35088 ppm/cm
HZCM          175.51581 Hz/cm
```

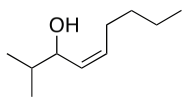
1H spectrum



S12


```
Current Data Parameters
USER                grants
NAME            GS3_213_p
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_             20080201
Time                 9.34
INSTRUM           cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG               zg30
TD                   81728
SOLVENT              CDCl3
NS                       8
DS                       2
SWH               8012.820 Hz
FIDRES               0.098043 Hz
AQ                5.0998774 sec
RG                     5.7
DW                  62.400 usec
DE                   6.00 usec
TE                  298.0 K
D1              0.10000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                    1H
P1                   8.00 usec
PL1                  1.60 dB
SFO1           500.2235015 MHz

F2 - Processing parameters
SI                   65536
SF             500.2200261 MHz
WDW                     EM
SSB                      0
LB                   0.30 Hz
GB                       0
PC                   1.00

1D NMR plot parameters
CX                  22.80 cm
CY                  15.00 cm
F1P                 8.000 ppm
F1                4001.76 Hz
F2P                 0.000 ppm
F2                  0.00 Hz
PPMCM             0.35088 ppm/cm
HZCM            175.51581 Hz/cm
```

1H spectrum



S13

Current Data Parameters
USER grants
NAME GS2_029_p
EXPNO 1
PROCNO 1

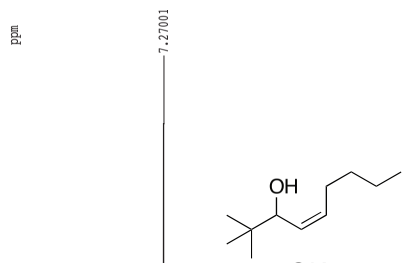
F2 - Acquisition Parameters
Date_ 20061017
Time 13.07
INSTRUM gn500
PROBHD 5 mm broadband
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 322.5
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======= CHANNEL f1 =======
NUC1 1H
P1 12.30 usec
PL1 -3.00 dB
SFO1 499.9334995 MHz

F2 - Processing parameters
SI 65536
SF 499.9300251 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 8.000 ppm
F1 3999.44 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.35088 ppm/cm
HZCM 175.41405 Hz/cm

1H spectrum



S14

Current Data Parameters

USER	grants
NAME	GS2_059_C13
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20061128
Time	20.21
INSTRUM	cryo500
PROBHD	5 mm CPTCI 1H-
PULPROG	zg30
TD	81728
SOLVENT	CDCl3
NS	16
DS	2
SWH	8012.820 Hz
FIDRES	0.098043 Hz
AQ	5.0998774 sec
RG	5
DW	62.400 usec
DE	6.00 usec
TE	298.0 K
D1	0.10000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

======= CHANNEL f1 =======

NUC1	1H
P1	8.00 usec
PL1	1.60 dB
SFO1	500.2235015 MHz

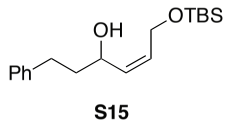
F2 - Processing parameters

SI	65536
SF	500.2200265 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	2.00

1D NMR plot parameters

CX	22.80 cm
CY	122.71 cm
F1P	8.000 ppm
F1	4001.76 Hz
F2P	0.000 ppm
F2	0.00 Hz
PPMCM	0.35088 ppm/cm
HZCM	175.51581 Hz/cm

1H spectrum



S15

Current Data Parameters
USER grants
NAME GS3_243_p_C13
EXPNO 1
PROCNO 1

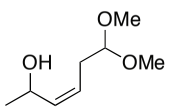
F2 - Acquisition Parameters
Date_ 20080223
Time 11.12
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 5
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======= CHANNEL f1 =======
NUC1 1H
P1 7.38 usec
PL1 1.60 dB
SFO1 500.2235015 MHz

F2 - Processing parameters
SI 65536
SF 500.2200258 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 22.80 cm
CY 101.54 cm
F1P 8.000 ppm
F1 4001.76 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.35088 ppm/cm
HZCM 175.51581 Hz/cm

1H spectrum



S16

Current Data Parameters
USER grants
NAME GS3_295_p
EXPNO 2
PROCNO 1

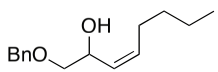
F2 - Acquisition Parameters
Date_ 20080411
Time 14.36
INSTRUM gn500
PROBHD 5 mm broadband
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 812.7
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -3.00 dB
SFO1 499.8284988 MHz

F2 - Processing parameters
SI 65536
SF 499.8250256 MHz
WDW GM
SSB 0
LB -0.30 Hz
GB 0.1505
PC 4.00

1D NMR plot parameters
CX 22.80 cm
CY 100.21 cm
F1P 8.000 ppm
F1 3998.60 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.35088 ppm/cm
HZCM 175.37720 Hz/cm

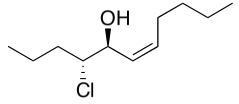
1H spectrum



S17

```
Current Data Parameters
USER                grants
NAME          GS2_171_C13
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20070330
Time                18.40
INSTRUM           cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG              zg30
TD                  81728
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8012.820 Hz
FIDRES           0.098043 Hz
AQ               5.0998774 sec
RG                    3.6
DW                62.400 usec
DE                 6.00 usec
TE                298.0 K
D1             0.10000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                  8.00 usec
PL1                 1.60 dB
SFO1          500.2235015 MHz

F2 - Processing parameters
SI                  65536
SF            500.2200265 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   4.00

1D NMR plot parameters
CX                  22.80 cm
CY                  17.31 cm
F1P                 8.000 ppm
F1                4001.76 Hz
F2P                 0.000 ppm
F2                  0.00 Hz
PPMCM            0.35088 ppm/cm
HZCM           175.51581 Hz/cm
```

1H spectrum



S18

Current Data Parameters
USER grants
NAME GS3_196_p
EXPNO 2
PROCNO 1

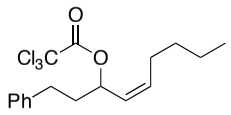
F2 - Acquisition Parameters
Date_ 20080121
Time 14.55
INSTRUM gn500
PROBHD 5 mm broadband
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 812.7
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -3.00 dB
SFO1 499.8284988 MHz

F2 - Processing parameters
SI 65536
SF 499.8250255 MHz
WDW GM
SSB 0
LB -0.35 Hz
GB 0.2505
PC 0.50

1D NMR plot parameters
CX 22.80 cm
CY 45.77 cm
F1P 8.000 ppm
F1 3998.60 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.35088 ppm/cm
HZCM 175.37720 Hz/cm

1H spectrum



10a

Current Data Parameters
USER grants
NAME GS3_243_p_C13
EXPNO 1
PROCNO 1

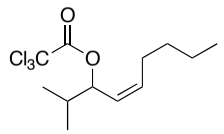
F2 - Acquisition Parameters
Date_ 20080223
Time 11.12
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 5
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======= CHANNEL f1 =======
NUC1 1H
P1 7.38 usec
PL1 1.60 dB
SFO1 500.2235015 MHz

F2 - Processing parameters
SI 65536
SF 500.2200258 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 22.80 cm
CY 101.54 cm
F1P 8.000 ppm
F1 4001.76 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.35088 ppm/cm
HZCM 175.51581 Hz/cm

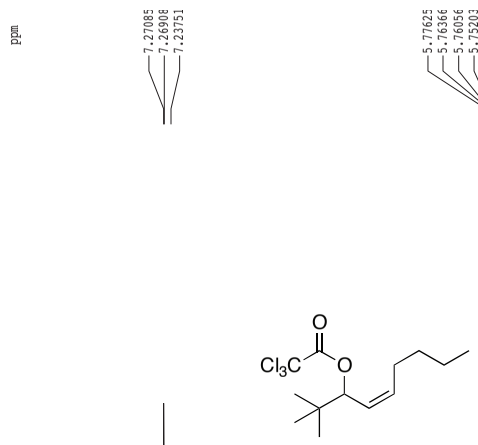
1H spectrum



10b

```
Current Data Parameters
USER              grants
NAME          JK_026_C13
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20070419
Time              15.08
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG             zg30
TD                 81728
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.098043 Hz
AQ              5.0998774 sec
RG                   4.5
DW                62.400 usec
DE                 6.00 usec
TE                298.0 K
D1            0.10000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                 8.00 usec
PL1                1.60 dB
SFO1         500.2235015 MHz

F2 - Processing parameters
SI                 65536
SF           500.2200268 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  4.00

1D NMR plot parameters
CX                 22.80 cm
CY                 15.00 cm
F1P                8.000 ppm
F1               4001.76 Hz
F2P                0.000 ppm
F2                 0.00 Hz
PPMCM           0.35088 ppm/cm
HZCM          175.51581 Hz/cm
```

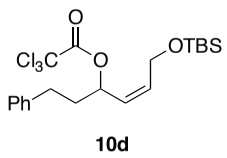
1H spectrum



10c

```
Current Data Parameters
USER              grants
NAME           GS3_092_c
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20071009
Time               11.26
INSTRUM            av600
PROBHD   5 mm TBI 1H/13
PULPROG             zg30
TD                 97938
SOLVENT            CDCl3
NS                     8
DS                     2
SWH             9615.385 Hz
FIDRES          0.098178 Hz
AQ              5.0928259 sec
RG                   287
DW                52.000 usec
DE                 6.00 usec
TE                298.0 K
D1           0.10000000 sec
TD0                    1

======== CHANNEL f1 =======
NUC1                  1H
P1                 8.00 usec
PL1               -1.00 dB
SFO1         600.1342009 MHz

F2 - Processing parameters
SI                 65536
SF           600.1300290 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 22.80 cm
CY                 57.16 cm
F1P                8.000 ppm
F1               4801.04 Hz
F2P                0.000 ppm
F2                 0.00 Hz
PPMCM           0.35088 ppm/cm
HZCM          210.57195 Hz/cm
```

1H spectrum



10d

Current Data Parameters
USER grants
NAME GS3_245_p
EXPNO 2
PROCNO 1

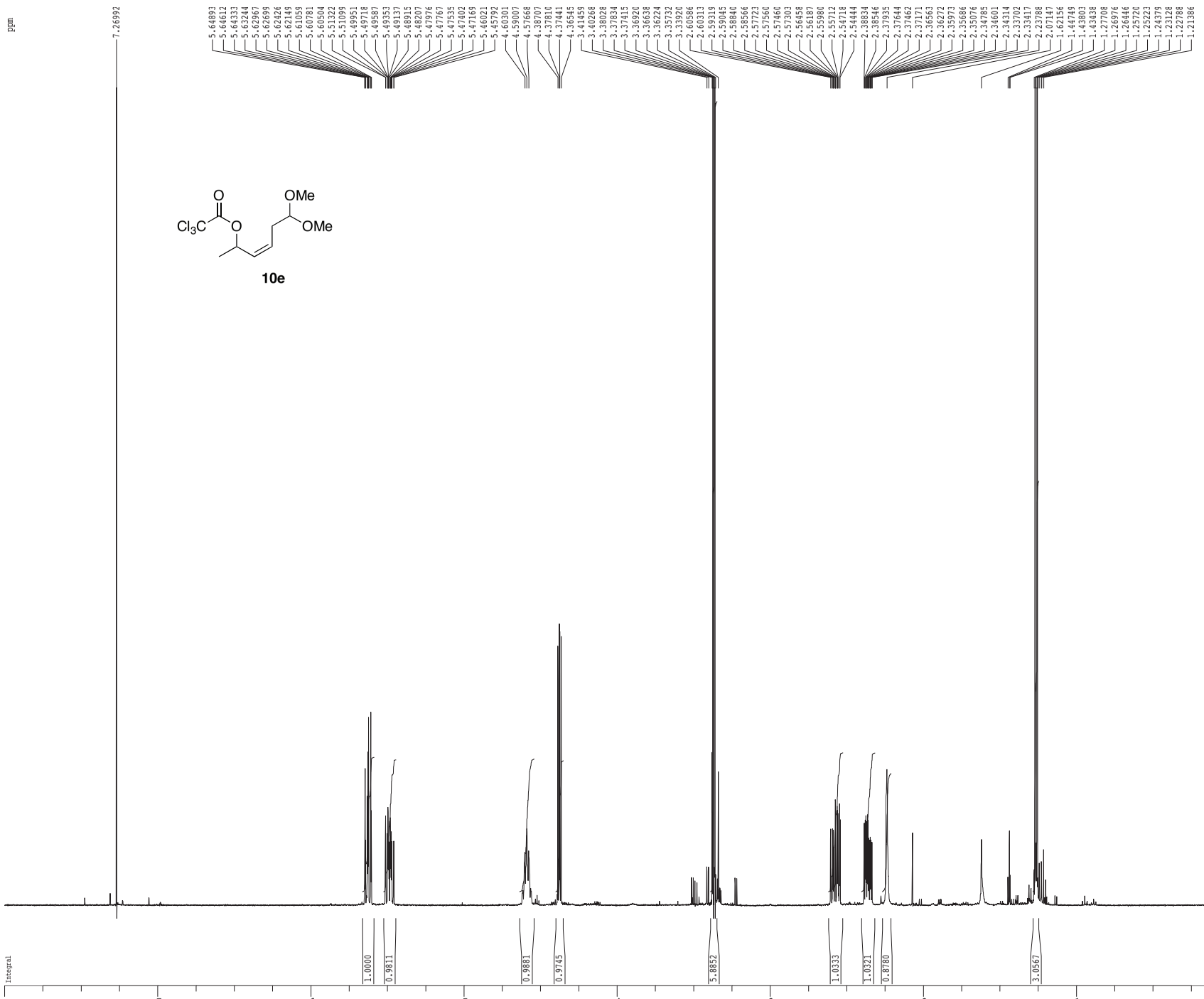
F2 - Acquisition Parameters
Date_ 20080225
Time 18.41
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 5
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 7.38 usec
PL1 1.60 dB
SFO1 500.2235015 MHz

F2 - Processing parameters
SI 65536
SF 500.2200260 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 22.80 cm
CY 54.31 cm
F1P 8.000 ppm
F1 4001.76 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.35088 ppm/cm
HZCM 175.51581 Hz/cm

1H spectrum



10e

Current Data Parameters
USER grants
NAME GS3_295_p
EXPNO 2
PROCNO 1

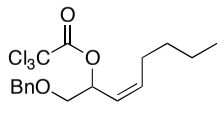
F2 - Acquisition Parameters
Date_ 20080411
Time 14.36
INSTRUM gn500
PROBHD 5 mm broadband
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 812.7
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -3.00 dB
SFO1 499.8284988 MHz

F2 - Processing parameters
SI 65536
SF 499.8250256 MHz
WDW GM
SSB 0
LB -0.30 Hz
GB 0.1505
PC 4.00

1D NMR plot parameters
CX 22.80 cm
CY 100.21 cm
F1P 8.000 ppm
F1 3998.60 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.35088 ppm/cm
HZCM 175.37720 Hz/cm

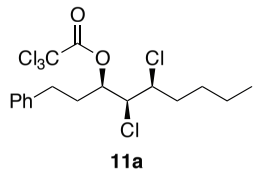
1H spectrum



10f

```
Current Data Parameters
USER                grants
NAME          GS3_233_C13
EXPNO                   4
PROCNO                  1

F2 - Acquisition Parameters
Date_            20080221
Time                13.10
INSTRUM          cryo500
PROBHD    5 mm CPTCI 1H-
PULPROG              zg30
TD                  81728
SOLVENT             CDCl3
NS                      8
DS                      2
SWH              8012.820 Hz
FIDRES           0.098043 Hz
AQ               5.0998774 sec
RG                    6.3
DW                62.400 usec
DE                 6.00 usec
TE                298.0 K
D1             0.10000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                  7.38 usec
PL1                 1.60 dB
SFO1          500.2235015 MHz

F2 - Processing parameters
SI                  65536
SF            500.2200256 MHz
WDW                    GM
SSB                     0
LB                 -0.40 Hz
GB                 0.5005
PC                   4.00

1D NMR plot parameters
CX                  22.80 cm
CY                  22.03 cm
F1P                 8.000 ppm
F1                4001.76 Hz
F2P                 0.000 ppm
F2                  0.00 Hz
PPMCM            0.35088 ppm/cm
HZCM           175.51581 Hz/cm
```

1H spectrum



11a

Current Data Parameters
USER grants
NAME GS3_194_bott_HMQC
EXPNO 1
PROCNO 1

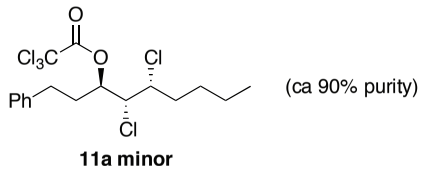
F2 - Acquisition Parameters
Date_ 20080217
Time 8.36
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 5
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 7.38 usec
PL1 1.60 dB
SFO1 500.2235015 MHz

F2 - Processing parameters
SI 65536
SF 500.2200260 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 8.000 ppm
F1 4001.76 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.35088 ppm/cm
HZCM 175.51581 Hz/cm

1H spectrum

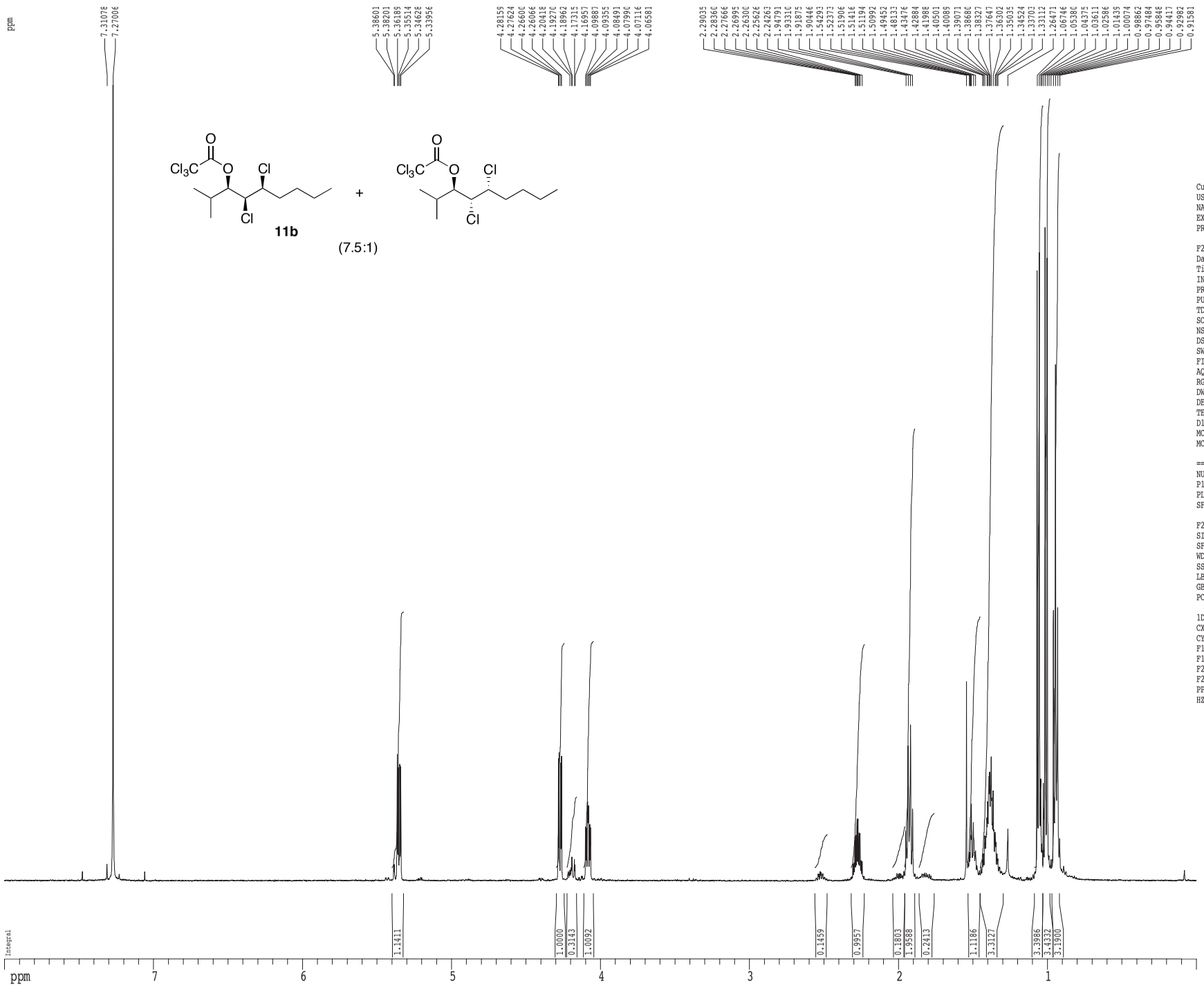


(ca 90% purity)

11a minor

```
Current Data Parameters
USER                grants
NAME      GS3_194_top_HMQC
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20080216
Time                 20.11
INSTRUM            cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG               zg30
TD                   81728
SOLVENT              CDCl3
NS                       8
DS                       2
SWH               8012.820 Hz
FIDRES               0.098043 Hz
AQ                5.0998774 sec
RG                       5
DW                  62.400 usec
DE                   6.00 usec
TE                  298.0 K
D1              0.10000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                     1H
P1                    7.38 usec
PL1                   1.60 dB
SFO1          500.2235015 MHz

F2 - Processing parameters
SI                    65536
SF            500.2200258 MHz
WDW                      EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00

1D NMR plot parameters
CX                   22.80 cm
CY                   23.95 cm
F1P                   8.000 ppm
F1                 4001.76 Hz
F2P                   0.000 ppm
F2                    0.00 Hz
PPMCM             0.35088 ppm/cm
HZCM            175.51581 Hz/cm
```

1H spectrum



Current Data Parameters

USER	grants
NAME	GS4_012_p
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20080421
Time	14.43
INSTRUM	gn500
PROBHD	5 mm broadband
PULPROG	zg30
TD	81728
SOLVENT	CDCl3
NS	8
DS	2
SWH	8012.820 Hz
FIDRES	0.098043 Hz
AQ	5.0998774 sec
RG	1625.5
DW	62.400 usec
DE	6.00 usec
TE	298.0 K
D1	0.10000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

======= CHANNEL f1 =======

NUC1	1H
P1	12.00 usec
PL1	-3.00 dB
SFO1	499.8284988 MHz

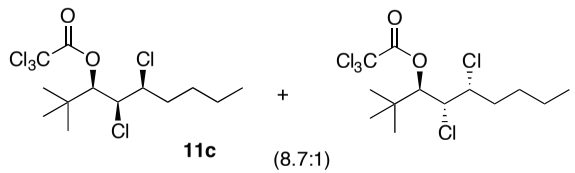
F2 - Processing parameters

SI	65536
SF	499.8250255 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	4.00

1D NMR plot parameters

CX	22.80 cm
CY	32.85 cm
F1P	8.000 ppm
F1	3998.60 Hz
F2P	0.000 ppm
F2	0.00 Hz
PPMCM	0.35088 ppm/cm
HZCM	175.37720 Hz/cm

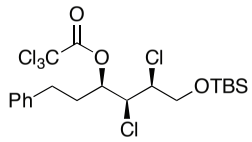
1H spectrum



11c (8.7:1)


```
Current Data Parameters
USER              grants
NAME          GS4_013_p
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20080421
Time              14.50
INSTRUM           gn500
PROBHD   5 mm broadband
PULPROG            zg30
TD                81728
SOLVENT           CDCl3
NS                    8
DS                    2
SWH            8012.820 Hz
FIDRES        0.098043 Hz
AQ            5.0998774 sec
RG               1149.4
DW               62.400 usec
DE                6.00 usec
TE               298.0 K
D1           0.10000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1               12.00 usec
PL1               -3.00 dB
SFO1        499.8284988 MHz

F2 - Processing parameters
SI                65536
SF          499.8250255 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 4.00

1D NMR plot parameters
CX                22.80 cm
CY                68.13 cm
F1P                8.000 ppm
F1              3998.60 Hz
F2P                0.000 ppm
F2                 0.00 Hz
PPMCM          0.35088 ppm/cm
HZCM         175.37720 Hz/cm
```

1H spectrum



11d

Current Data Parameters
USER grants
NAME GS3_282_C13
EXPNO 1
PROCNO 1

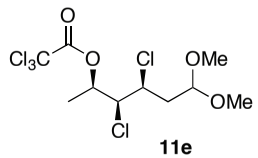
F2 - Acquisition Parameters
Date_ 20080405
Time 20.07
INSTRUM cryo500
PROBHD 5 mm CPTCI 1H-
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 1
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 4.5
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 7.38 usec
PL1 1.60 dB
SFO1 500.2235015 MHz

F2 - Processing parameters
SI 65536
SF 500.2200261 MHz
WDW GM
SSB 0
LB -0.30 Hz
GB 0.2005
PC 4.00

1D NMR plot parameters
CX 22.80 cm
CY 96.63 cm
F1P 8.000 ppm
F1 4001.76 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.35088 ppm/cm
HZCM 175.51581 Hz/cm

1H spectrum



11e

Current Data Parameters
USER grants
NAME GS3_277_p
EXPNO 1
PROCNO 1

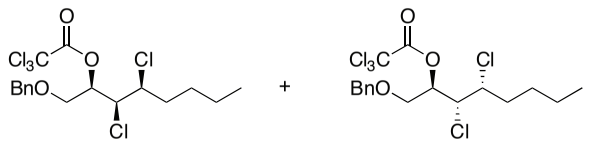
F2 - Acquisition Parameters
Date_ 20080319
Time 17.51
INSTRUM gn500
PROBHD 5 mm broadband
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 2580.3
DW 62.400 usec
DE 6.00 usec
TE 298.2 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======= CHANNEL f1 =======
NUC1 1H
P1 12.00 usec
PL1 -3.00 dB
SFO1 499.8284988 MHz

F2 - Processing parameters
SI 65536
SF 499.8250255 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 22.80 cm
CY 40.83 cm
F1P 8.000 ppm
F1 3998.60 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.35088 ppm/cm
HZCM 175.37720 Hz/cm

1H spectrum

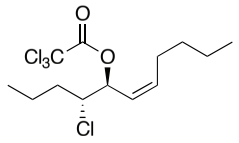


11f

(4.4:1)

```
Current Data Parameters
USER              grants
NAME           GS3_278_p
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20080325
Time               11.08
INSTRUM            av600
PROBHD   5 mm BBO BB-1H
PULPROG            zg30
TD                97938
SOLVENT           CDCl3
NS                    8
DS                    2
SWH            9615.385 Hz
FIDRES        0.098178 Hz
AQ            5.0928259 sec
RG                  575
DW             52.000 usec
DE              6.00 usec
TE              298.0 K
D1           0.10000000 sec
TD0                   1

======== CHANNEL f1 =======
NUC1                 1H
P1                9.00 usec
PL1              -5.30 dB
SFO1        600.1342009 MHz

F2 - Processing parameters
SI                65536
SF          600.1300278 MHz
WDW                  EM
SSB                   0
LB                0.30 Hz
GB                    0
PC                1.00

1D NMR plot parameters
CX               22.80 cm
CY               16.35 cm
F1P               8.000 ppm
F1             4801.04 Hz
F2P               0.000 ppm
F2                0.00 Hz
PPMCM          0.35088 ppm/cm
HZCM         210.57195 Hz/cm
```

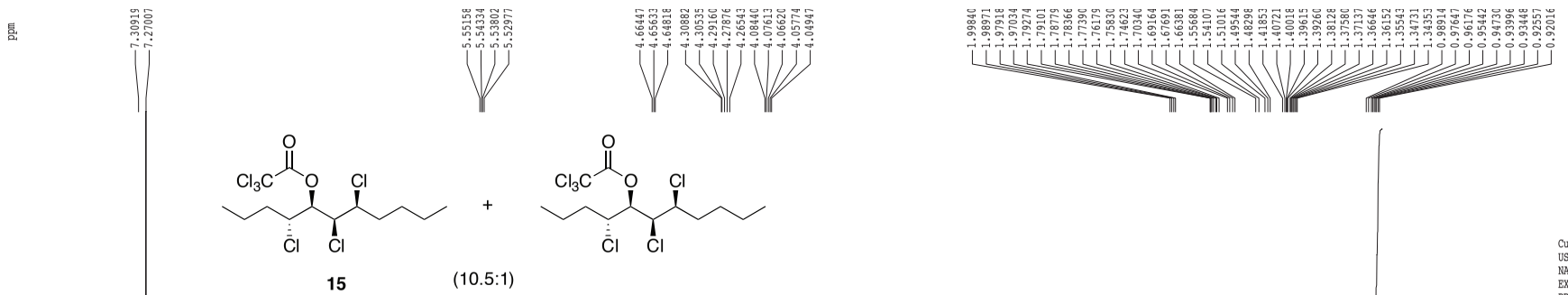
1H spectrum



14

```
Current Data Parameters
USER              grants
NAME           GS3_095_c
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20071013
Time              14.10
INSTRUM           gn500
PROBHD   5 mm broadband
PULPROG            zg30
TD                81728
SOLVENT           CDCl3
NS                    8
DS                    2
SWH            8012.820 Hz
FIDRES         0.098043 Hz
AQ             5.0998774 sec
RG                 1024
DW               62.400 usec
DE                6.00 usec
TE                298.0 K
D1           0.10000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                1H
P1                12.00 usec
PL1               -3.00 dB
SFO1         499.8284988 MHz

F2 - Processing parameters
SI                65536
SF           499.8250258 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 4.00

1D NMR plot parameters
CX                22.80 cm
CY                15.00 cm
F1P                8.000 ppm
F1              3998.60 Hz
F2P                0.000 ppm
F2                 0.00 Hz
PPMCM          0.35088 ppm/cm
HZCM          175.37720 Hz/cm
```

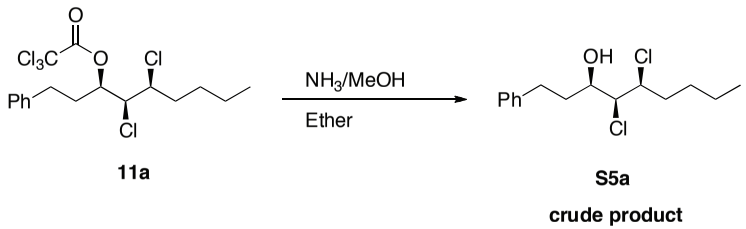
1H spectrum



15 (10.5:1)

```
Current Data Parameters
USER              grants
NAME           GS3_296_p
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20080421
Time              12.06
INSTRUM           gn500
PROBHD   5 mm broadband
PULPROG            zg30
TD                81728
SOLVENT           CDCl3
NS                    8
DS                    2
SWH            8012.820 Hz
FIDRES         0.098043 Hz
AQ             5.0998774 sec
RG                1824.6
DW                62.400 usec
DE                 6.00 usec
TE                298.0 K
D1           0.10000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                1H
P1                12.00 usec
PL1               -3.00 dB
SFO1         499.8284988 MHz

F2 - Processing parameters
SI                 65536
SF           499.8250256 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 2.00

1D NMR plot parameters
CX                22.80 cm
CY                31.80 cm
F1P                8.000 ppm
F1              3998.60 Hz
F2P                0.000 ppm
F2                 0.00 Hz
PPMCM          0.35088 ppm/cm
HZCM          175.37720 Hz/cm
```

1H spectrum



11a

S5a

crude product

Current Data Parameters
USER grants
NAME GS2_200_c
EXPNO 1
PROCNO 1

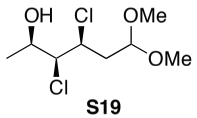
F2 - Acquisition Parameters
Date_ 20070424
Time 13.46
INSTRUM gn500
PROBHD 5 mm broadband
PULPROG zg30
TD 81728
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.098043 Hz
AQ 5.0998774 sec
RG 256
DW 62.400 usec
DE 6.00 usec
TE 298.0 K
D1 0.10000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 12.00 usec
PL1 -3.00 dB
SFO1 499.9334995 MHz

F2 - Processing parameters
SI 65536
SF 499.9300251 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 22.80 cm
CY 15.00 cm
F1P 8.000 ppm
F1 3999.44 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.35088 ppm/cm
HZCM 175.41405 Hz/cm

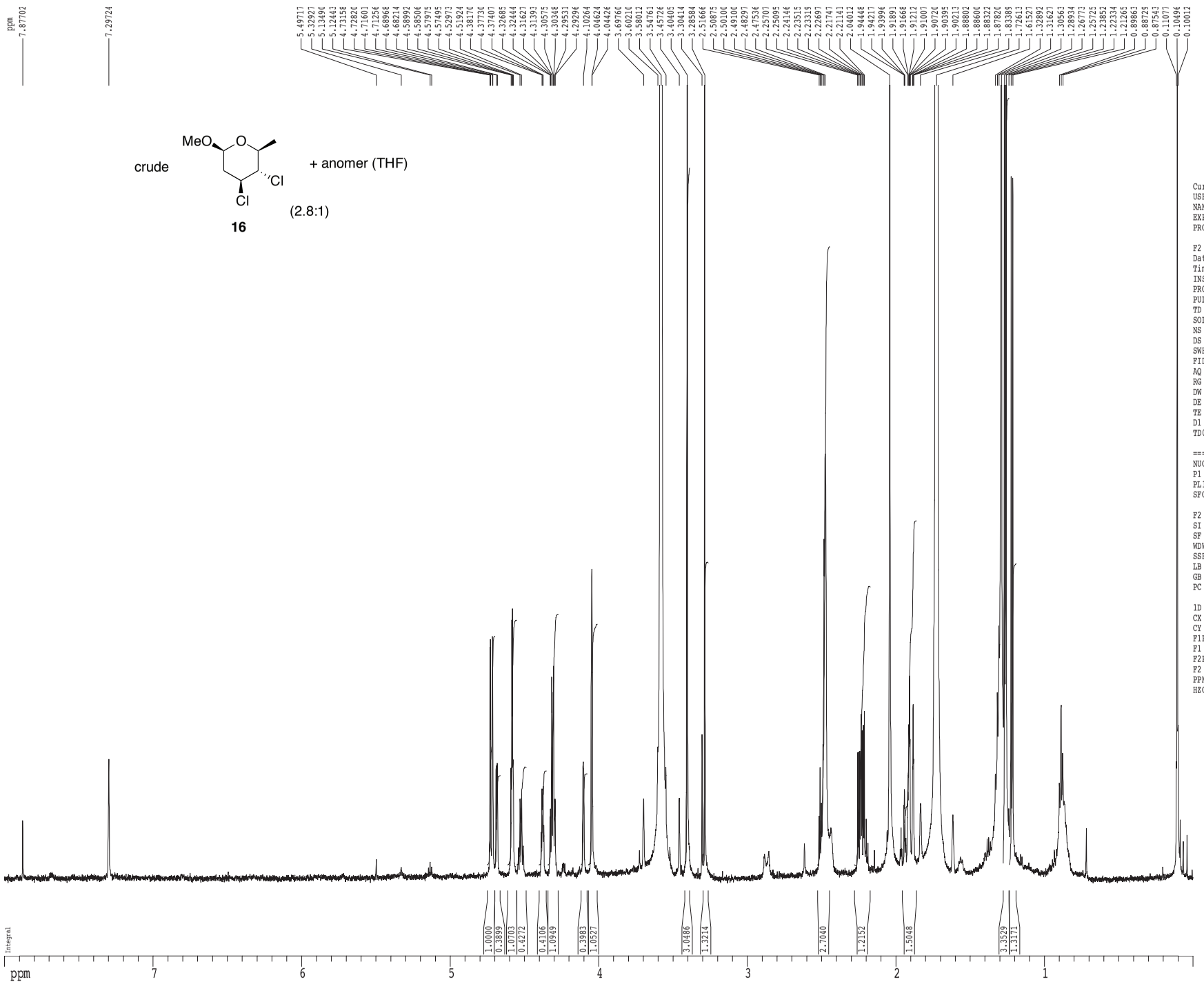
1H spectrum



S19


```
Current Data Parameters
USER                grants
NAME          GS3_290_C13
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20080328
Time                10.09
INSTRUM          cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG              zg30
TD                  81728
SOLVENT             CDCl3
NS                      8
DS                      2
SWH              8012.820 Hz
FIDRES           0.098043 Hz
AQ               5.0998774 sec
RG                      9
DW                 62.400 usec
DE                  6.00 usec
TE                298.0 K
D1              0.10000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                  7.38 usec
PL1                 1.60 dB
SFO1          500.2235015 MHz

F2 - Processing parameters
SI                  65536
SF            500.2200261 MHz
WDW                    EM
SSB                     0
LB                  0.30 Hz
GB                      0
PC                  1.00

1D NMR plot parameters
CX                 22.80 cm
CY                 31.13 cm
F1P                 8.000 ppm
F1                4001.76 Hz
F2P                 0.000 ppm
F2                  0.00 Hz
PPMCM            0.35088 ppm/cm
HZCM           175.51581 Hz/cm
```

1H spectrum



Current Data Parameters
USER grants
NAME GS3_293_c_THF-d8
EXPNO 2
PROCNO 1

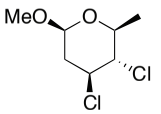
F2 - Acquisition Parameters
Date_ 20080330
Time 11.58
INSTRUM av600
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 97938
SOLVENT THF
NS 16
DS 2
SWH 9615.385 Hz
FIDRES 0.098178 Hz
AQ 5.0928259 sec
RG 575
DW 52.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 9.00 usec
PL1 -5.30 dB
SFO1 600.1342009 MHz

F2 - Processing parameters
SI 65536
SF 600.1311347 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.80 cm
CY 236.41 cm
F1P 8.000 ppm
F1 4801.05 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.35088 ppm/cm
HZCM 210.57234 Hz/cm

1H spectrum



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(d-chloroform)

```
Current Data Parameters
USER              grants
NAME       GS3_293_p_top
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20080416
Time              18.24
INSTRUM         cryo500
PROBHD   5 mm CPTCI 1H-
PULPROG             zg30
TD                81728
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8012.820 Hz
FIDRES         0.098043 Hz
AQ             5.0998774 sec
RG                  4.5
DW                62.400 usec
DE                 6.00 usec
TE                298.0 K
D1           0.10000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                 7.38 usec
PL1                1.60 dB
SFO1        500.2235015 MHz

F2 - Processing parameters
SI                65536
SF          500.2200260 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 4.00

1D NMR plot parameters
CX                22.80 cm
CY               120.95 cm
F1P               8.000 ppm
F1               4001.76 Hz
F2P               0.000 ppm
F2                0.00 Hz
PPMCM          0.35088 ppm/cm
HZCM         175.51581 Hz/cm
```