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Supplementary Material

Experimental Section

Reagents. Tetrahydrofuran (THF) was distilled immediately prior to use from benzophenone ketyl under Ar. Samarium metal was purchased from Cerac Inc., Milwaukee, WI, and was stored under an inert atmosphere. CH_2I_2 was purchased from Aldrich Chemicals and was distilled prior to use and stored under argon over copper turnings. Standard benchtop techniques were employed for handling air sensitive reagents,[1] and all reactions were carried out under argon.

(*tert*-Butoxycarbonylmethylene)triphenylphosphorane. General Procedure for the Preparation of Wittig Reagents. Triphenylphosphine (7.34 g, 28.0 mmol) and *tert*-butyl bromoacetate (5.85 g, 30.0 mmol) were heated together at reflux in 40 mL of THF for 24 h. After this period of time, the reaction mixture was cooled to rt and the phosphonium salt was isolated by vacuum filtration, washed with 3-20 mL portions of ether, and then dried under high vacuum to afford the desired intermediate (12.4 g, 27.1 mmol) in 90% yield. The resultant phosphonium salt was taken up in 20 mL of H_2O and then NaOH (2.0 M in H_2O) was added in small portions to the stirred solution until the reaction mixture was basic (pH = 8-9) at which time a white precipitate had formed. The white solid Wittig reagent was isolated by vacuum filtration, washed with H_2O (3x 20 mL), and then dried under high vacuum to afford the desired ylide (7.32 g, 19.5 mmol) in 72% yield; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.65-7.43 (m, 16H), 1.19 (s, 9H).

***N*, *N*-Diethyl 2-(Triphenylphosphoranylidene)acetamide.** Prepared from 2-chloro-*N*, *N*-diethylacetamide and triphenylphosphine according to the general procedure outlined for the preparation of (*tert*-butoxycarbonylmethylene)triphenyl-phosphorane to afford the desired ylide in 52% yield (2 steps); ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.68-7.61 (m, 6H), 7.47-7.36 (m, 10H), 3.32 (q, J = 7.08 Hz, 4H), 1.12 (t, J = 7.08 Hz, 6H).

5-Bromopentanal (2a). General Procedure for Ozonolysis of Olefins. Ozone was bubbled through a -78 °C cooled solution of 6-bromo-1-hexene (5.0 g, 30.7 mmol) in a 5:1 mixture of CH_2Cl_2/EtOH (20mL) with a catalytic amount of $NaHCO_3$ until a blue color persisted. Then the reaction mixture was purged with argon for 5 min. Dimethylsulfide (DMS) (25 mL) was added in one portion to

the -78 °C cooled solution, and the reaction mixture was warmed to rt and allowed to stir for 12 h at ambient temperature. After this period of stirring, the reaction mixture was concentrated *in vacuo*, flashed through a short plug of silica gel (20% EtOAc/hexanes) to remove the DMSO, and subjected to Kugelrohr distillation (ot 70-80 °C) to afford the desired aldehyde (**2a**) in 84% yield (4.3 g, 25.8 mmol); ^{1}H NMR (300 MHz, $CDCl_3$): δ 9.76 (s, 1H), 3.39 (t, J = 6.35 Hz, 2H), 2.47 (t, J = 7.08 Hz, 2H), 1.87 (m, 2H), 1.79 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 220.46, 41.57, 31.74, 30.62, 19.34.

6-Bromo-4-methylhexanal (2d). Prepared by ozonolysis and reductive workup of citronellyl bromide according to the general procedure outlined for the preparation of **2a** to afford aldehyde **2d** in 88% yield after flash chromatography with 20% EtOAc/hexanes and Kugelrohr distillation (ot 140 °C @ 5 mm Hg); ^{1}H NMR (300 MHz, $CDCl_3$): δ 9.77 (s, 1H), 3.42 (m, 2H), 2.45 (m, 2H), 1.85 (m, 1H), 1.67 (m, 3H), 1.44 (m, 1H), 0.89 (d, J = 6.10 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 202.25, 41.46, 39.56, 31.56, 31.11, 28.22, 18.57.

Ethyl 7-Bromo-2-heptenoate (3a). Prepared from **2a** and (carbethoxymethylene)triphenylphosphorane according to the general procedure outlined for the preparation of **3b** to afford **3a** in 96% yield after flash chromatography with 5% EtOAc/hexanes; (E isomer) ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.92 (dt, J = 6.94, 15.20 Hz, 1H), 5.81 (d, J = 15.20 Hz, 1H), 4.15 (q, J = 7.16 Hz, 2H), 3.39 (t, J = 6.09 Hz, 2H), 2.21 (m, 2H), 1.85 (m, 2H), 1.61 (m, 2H), 1.26 (t, J = 7.19 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 178.29, 148.17, 121.91, 60.27, 33.30, 32.03, 31.22, 26.52, 14.29.

***tert*-Butyl 7-Bromo-2-heptenoate (3b). General Procedure for Wittig Olefination of Aldehydes.** 5-Bromopentanal (**2a**, 1.0 g, 6.10 mmol) and (*tert*-butoxycarbonylmethylene)triphenylphosphorane (3.2 g, 9.20 mmol) were heated together at reflux in 10 mL of benzene for 18-20 h. After this period of time, the reaction mixture was cooled to rt and concentrated *in vacuo*. The resultant slurry was subject to flash chromatography (5% EtOAc/hexanes) to afford the desired olefin **3b** (20:1 mixture of E:Z isomers, 1.35 g, 4.1 mmol) in 96% combined yield; (E isomer) ^{1}H NMR (300 MHz, $CDCl_3$): δ 6.81 (dt, J = 7.08, 15.63 Hz, 1H), 5.73 (dt, J = 1.71, 15.63

Hz, 1H), 3.39 (t, J = 6.59 Hz, 2H), 2.18 (dq, J = 1.47, 7.32 Hz, 2H), 1.86 (m, 2H), 1.58 (m, 2H), 1.46 (s, 9H).

***N*, *N*-Diethyl 7-Bromo-2-heptenamide (3c).** Prepared from *N*, *N*-diethyl 2-(triphenylphosphoranylidene)acetamide and 5-bromopentanal (**2a**) according to the general procedure outlined for the preparation of **3b** to afford **3c** (as a 20:1 mixture of E/Z isomers) in 67% yield after flash chromatography with 50% EtOAc/hexanes; (*E* -isomer) ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.86 (m, 1H), 6.18 (m, 1H), 3.39 (t, J = 7.09 Hz, 4H), 3.35 (m, 2H), 2.22 (m, 2H), 1.88 (m, 2H), 1.60 (m, 2H), 1.17 (t, J = 7.09 Hz, 3H), 1.12 (t, J = 7.09 Hz, 3H); (*Z*-isomer) ^{1}H NMR (300 MHz, $CDCl_3$): δ 6.01 (dt, J = 1.46, 11.72 Hz, 1H), 5.86 (dt, J = 7.33, 11.72 Hz, 1H), 3.40 (t, J = 6.84 Hz, 4H), 3.34 (m, 2H), 2.41 (dq, J = 1.46, 7.33 Hz, 2H), 1.87 (m, 2H), 1.57 (m, 2H), 1.13 (t, J = 7.08 Hz, 6H).

Ethyl 8-Bromo-6-methyl-2-octenoate (3d). Prepared from **2d** and (carbethoxymethylene)triphenylphosphorane according to the general procedure outlined for the preparation of **3b** to afford **3d** in 84% yield after flash chromatography with 3-5% EtOAc/hexanes; (E Isomer) ^{1}H NMR (300 MHz, $CDCl_3$): δ 6.93 (dt, J = 6.84, 15.63 Hz, 1H), 5.80 (dt, J = 1.71, 15.63 Hz, 1H), 4.15 (q, J = 7.08 Hz, 2H), 3.41 (m, 2H), 2.20 (m, 2H), 1.85 (m, 1H), 1.67 (m, 2H), 1.45 (m, 1H), 1.32 (m, 1H), 1.27 (t, J = 7.08 Hz, 3H), 0.89 (d, J = 6.35 Hz, 3H).

***N*, *N*-Diethyl 8-Bromo-6-methyl-2-octenamide (3e).** Prepared from **2d** and *N*, *N*-diethyl 2-(triphenylphosphoranylidene)acetamide according to the general procedure outlined for the preparation of **3b** to afford **3e** in 76% yield after flash chromatography with 50% EtOAc/hexanes: ^{1}H NMR (300 MHz, $CDCl_3$): δ 6.86 (m, 1H), 6.18 (m, 1H), 3.89 (m, 6H), 2.21 (m, 2H), 1.88 (m, 1H), 1.65 (m, 2H), 1.44 (m, 1H), 1.28 (m, 1H), 1.15 (m, 6H), 0.89 (d, J = 6.35 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 165.73, 145.58, 120.59, 42.06, 40.72, 39.71, 35.06, 31.80, 31.14, 29.79, 18.64, 14.85, 13.13.

Ethyl 7-Iodo-2-heptenoate (4a). Prepared from **3a** according to the general Finkelstein procedure outlined for the preparation of **4b** to afford **4a** in 100% yield after flash chromatography with 5% EtOAc/hexanes; ^{1}H NMR (300 MHz, $CDCl_3$): δ 6.92 (dt, J = 7.08, 15.63 Hz, 1H), 5.80 (dt, J = 1.71, 15.63 Hz, 1H), 4.16 (q, J = 7.08 Hz, 2H), 3.17 (t, J = 6.84 Hz, 2H), 2.20 (dq, J = 1.71, 7.32

Hz, 2H), 1.82 (m, 2H), 1.57 (m, 2H), 1.27 (t, J = 7.08 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.51, 148.09, 121.87, 60.21, 32.69, 31.00, 28.82, 14.25, 6.14.

***tert*-Butyl 7-Iodo-2-heptenoate (4b). General Procedure for the Preparation of Iodoesters.** *tert*-Butyl 7-bromo-2-heptenoate (**3b**, 1.10 g, 4.2 mmol) and NaI (7.5 g, 50 mmol) were heated together in acetone (20 mL) at reflux for 16-18 h. After this period of time, the reaction mixture was cooled to rt and subjected to an aqueous workup after removal of acetone solvent *in vacuo*. Flash chromatography of the crude product (5% EtOAc/hexanes) afforded **4b** (0.91 g, 2.94 mmol) in 70 % yield; ^1H NMR (300 MHz, $CDCl_3$): δ 6.81 (dt, J = 6.84, 15.63 Hz, 1H), 5.73 (dt, J = 1.71, 15.63 Hz, 1H), 3.16 (t, J = 6.84 Hz, 2H), 2.18 (dq, J = 1.46, 7.32 Hz, 2H), 1.82 (m, 2H), 1.55 (m, 2H), 1.46 (s, 9H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 165.89, 146.77, 123.61, 32.76, 30.83, 28.91, 28.29, 28.15, 6.12.

***N*, *N*-Diethyl 7-Iodo-2-heptenamide (4c).** Prepared from **3c** according to the general procedure outlined for the preparation of **4b** to afford **4c** in 77% yield after flash chromatography with 50% EtOAc/hexanes; (Z Isomer) ^1H NMR (400 MHz, $CDCl_3$): δ 6.00 (m, 1H), 5.85 (dt, J = 11.53, 7.33 Hz, 1H), 3.36 (m, 4H), 3.18 (m, 2H), 2.40 (dq, J = 1.39, 7.48 Hz, 2H), 1.84 (m, 2H), 1.54 (m, 2H), 1.13 (m, 6H); (E Isomer) ^1H NMR (300 MHz, $CDCl_3$): δ 6.86 (dt, J = 7.08, 14.89 Hz, 1H), 6.19 (dt, J = 1.47, 14.89 Hz, 1H), 3.37 (m, 4H), 3.17 (t, J = 6.84 Hz, 2H), 2.20 (m, 2H), 1.83 (m, 2H), 1.56 (m, 2H), 1.15 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 165.61, 114.89, 121.02, 42.09, 40.76, 32.78, 31.23, 29.29, 14.87, 13.13, 6.36; HRMS calcd for $C_{11}H_{20}NOI$: 309.0561, found 309.0590; LRMS (EI^+): *m/z* 309 (31), 237 (100), 182 (98), 126 (91), 100 (9), 81 (32), 72 (18), 55 (63), 41 (30), 29 (28).

Ethyl 8-Iodo-6-methyl-2-octenoate (4d). Prepared from **3d** according to the general procedure outlined for the preparation of **4b** to afford **4d** in 92% yield after flash chromatography with 3% EtOAc/hexanes; (E Isomer) ^1H NMR (300 MHz, $CDCl_3$): δ 6.93 (dt, J = 7.08, 15.63 Hz, 1H), 5.80 (dt, J = 1.47, 15.63 Hz, 1H), 4.16 (q, J = 7.08 Hz, 2H), 3.24-3.09 (m, 2H), 2.20 (m, 2H), 1.84 (m, 1H), 1.70-1.40 (m, 3H), 1.32 (m, 1H), 1.26 (t, J = 7.08 Hz, 3H), 0.87 (d, J = 6.35 Hz, 3H); ^{13}C

NMR (100 MHz, $CDCl_3$): δ 166.62, 148.86, 121.42, 60.15, 40.56, 34.39, 33.38, 29.49, 18.40, 14.26, 4.60.

***N*, *N*-Diethyl 8-Iodo-6-methyl-2-octenamide (4e).** Prepared from **3e** according to the general procedure outlined for the preparation of **4b** to afford **4e** in 97% yield after flash chromatography with 50% EtOAc/hexanes: ^{1}H NMR (300 MHz, $CDCl_3$): δ 6.85 (dt, J = 7.08, 15.14 Hz, 1H), 6.15 (d, J = 15.14 Hz, 1H), 3.30 (m, 4H), 3.23-3.08 (m, 2H), 2.19 (m, 2H), 1.85 (m, 1H), 1.68-1.39 (m, 3H), 1.33-1.24 (m, 1H), 1.13 (m, 6H), 0.86 (d, J = 6.35 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 165.73, 145.60, 120.59, 40.61, 34.84, 33.39, 29.78, 18.43, 15.25, 13.13, 4.77; LRMS (EI$^+$): *m/z* 338 (63), 265 (98), 210 (100), 154 (80), 126 (81), 109 (34), 81 (38), 58 (31), 41 (32), 29 (32).

Tetrahydro-3-(3-chloropropyl)-(2H)-pyran-2-one (6b). General Procedure for Alkylation of Lactones. Prepared from δ-valerolactone according to the following general procedure. A 1.0 M solution of δ-valerolactone (2.0 g, 20.0 mmol) in THF was added dropwise *via* cannula over 1 h to a stirred solution of 22.0 mmol of LDA at -78 °C. After the addition of the substrate was complete, the reaction mixture was stirred an additional 0.5 h at -78 °C. After this period of stirring, 1-chloro-3-iodopropane (8.2 g, 40.0 mmol) in 10 mL of HMPA was added dropwise *via* cannula. After the addition of the halide was complete, the reaction mixture was warmed to -30 °C and stirred at reduced temperature for 6 h. After this period of time, the reaction mixture was quenched at -30 °C with saturated aqueous NH_4Cl. An aqueous workup followed by flash column chromatography with 15% EtOAc/hexanes afforded the desired alkylated product, **6b** (1.18g, 6.77 mmol), in 34% yield; ^{1}H NMR (300 MHz, $CDCl_3$): δ 4.29 (t, J = 5.62 Hz, 2H), 3.55 (dt, J = 6.10, 2.20 Hz, 2H), 2.46 (m, 1H), 2.09 (m, 1H), 1.95-1.80 (m, 5H), 1.70-1.51 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 174.15, 68.21, 44.83, 38.95, 30.04, 28.72, 24.78, 21.94.

Tetrahydro-3-ethyl-2(2H)-pyranone (6c). Prepared from δ-valerolactone by alkylation with ethyl iodide according to the general procedure outlined for the preparation of tetrahydro-3-(3-chloropropyl)-(2H)-pyran-2-one, **6b**, to afford the desired lactone in 61% yield after flash column chromatography with 20% EtOAc/hexanes; ^{1}H NMR (500 MHz, $CDCl_3$): δ 4.28 (m, 2H), 2.37 (m,

1H), 2.06 (m, 1H), 1.92-1.85 (m, 3H), 1.56-1.50 (m, 2H), 0.96 (t, J = 7.44 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 174.44, 68.30, 40.91, 24.19, 24.06, 21.96, 11.25; IR (neat) 1731.6 cm^{-1}.

Tetrahydro-3-(2-propenyl)-(2H)-pyran-2-ol (7a). General DIBAL-H Reduction Procedure for Lactones to Lactols. Prepared from 3-(2-propenyl)tetrahydro-(4H)-pyran-2-one by reduction with DIBAL-H according to the following general procedure. DIBAL-H (20 mL of a 1.0 M solution in hexanes, 20 mmol) was added rapidly dropwise to a -78 °C cooled solution of **6a** (2.50 g, 17.8 mmol) in 20 mL of dry THF. The reaction mixture was maintained at -78 °C for 30 min and then the reaction was quenched by pouring into a vigorously stirred solution of 20 mL of MeOH. The reaction mixture was concentrated in vacuo after stirring with the MeOH for 0.5 h. Flash column chromatography with 12% EtOAc/hexanes afforded the desired lactol, **7a** (2.15 g, 15.1 mmol), in 85% yield; ^1H NMR (400 MHz, $CDCl_3$): δ 5.75 (m, 1H), 5.05-4.97 (m, 2H), 4.44 (m, 0.5H), 3.96 (m, 1H), 3.56-3.43 (m, 1H), 3.14 (d, J = 5.62 Hz, 0.5H), 2.72 (m, 0.5H), 2.39 (m, 0.5H), 2.15 (m, 0.5H), 1.96-1.86 (m, 2.5H), 1.73 (m, 1H), 1.59-1.44 (m, 2.5H), 1.22 (m, 0.5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 136.51, 136.18, 116.37, 116.10, 98.95, 93.70, 65.27, 59.89, 41.38, 39.61, 35.92, 35.53, 26.73, 25.21, 24.63, 23.38.

Tetrahydro-3-(3-chloropropyl)-(2H)-pyran-2-ol (7b). Prepared from **6b** by DIBAL-H reduction of **6b** according to the general procedure outlined for the preparation of **7a** to afford **7b** in 94% yield after flash column chromatography with 15% EtOAc/hexanes; ^1H NMR (300 MHz, $CDCl_3$): δ 5.05 (m, 0.5H), 4.42 (m, 0.5H), 3.96 (m, 1H), 3.58-3.43 (m, 3H), 2.73 (m, 0.5H), 2.34 (m, 0.5H), 1.90-1.47 (m, 7H), 1.38-1.23 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 99.47, 93.74, 65.34, 59.86, 45.24, 45.16, 41.17, 39.18, 29.99, 29.91, 28.87, 28.64, 27.29, 25.22, 24.69, 23.61.

Tetrahydro 3-ethylpyran-2(2H)-ol (7c). Prepared from tetrahydro-3-ethyl-2(2H)-pyranone by reduction with DIBAL-H according to the general procedure outlined for the preparation of **7a** to afford the desired lactol/hydroxy aldehyde in 85% yield after Kugelrohr distillation (ot 90-100 °C @ 0.05 mm Hg); ^1H NMR (400 MHz, $CDCl_3$): δ 8.24 (s, 0.1H), 5.07 (s, 0.4H), 4.41 (d, J = 6.96 Hz, 0.4H), 3.96 (m, 0.8H), 3.76 (d, J = 6.69 Hz, 0.2H), 3.55 (m, 0.5H), 3.47 (m, 0.5H), 3.10 (m, 0.4H), 2.67 (m, 0.4H), 1.92 (m, 0.7H), 1.67 (m, 1H), 1.55 (m, 3H), 1.39 (m, 0.6H), 1.27-1.12 (m, 2H), 0.89 (m, 3H);

^{13}C NMR (100 MHz, $CDCl_3$): δ 99.42, 93.83, 83.47, 65.26, 59.76, 43.21, 41.50, 26.48, 25.39, 24.71, 24.38, 23.68, 23.28, 19.17, 11.25, 10.99; IR (neat) 3388.9, 1729.8 cm^{-1}; HRMS calcd for $C_7H_{14}O_2$: 130.0994; found 130.0994; LRMS (EI^+): *m/z* 130 (10), 84 (62), 69 (38), 56 (100), 41 (82), 27 (32).

6-Hydroxyhexanal. Prepared from ε-caprolactone by reduction with DIBAL-H according to the general reaction conditions described for the preparation of **7a** to afford the desired hydroxy aldehyde in 74% yield after flash column chromatography with 30% EtOAc/hexanes; ^1H NMR (500 MHz, $CDCl_3$): δ 9.74 (s, 1H), 3.63 (t, *J* = 6.55 Hz, 2H), 2.43 (t, *J* = 7.25 Hz, 2H), 1.64 (m, 2H), 1.56 (m, 3H), 1.38 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 202.60, 62.54, 43.80, 32.35, 25.31, 21.75.

Ethyl (2*E*)-7-Hydroxy-4-(2-propenyl)-2-heptenoate (8a). Prepared from **7a** and (carbethoxymethylene)triphenylphosphorane according to the general Wittig olefination procedure outlined for the preparation of **3b** to afford **8a** in 83% yield after flash column chromatography with 30% EtOAc/hexanes; ^1H NMR (400 MHz, $CDCl_3$): δ 6.74 (dd, *J* = 8.84, 15.53 Hz, 1H), 5.77 (d, *J* = 15.53 Hz, 1H), 5.68 (m, 1H), 5.02 (m, 2H), 4.16 (q, *J* = 7.23 Hz, 2H), 3.60 (m, 2H), 2.24 (m, 1H), 2.18 (m, 1H), 1.62-1.36 (m, 5H), 1.27 (t, *J* = 7.23 Hz, 3H), 1.24 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.54, 152.11, 135.63, 121.54, 116.79, 62.75, 60.25, 42.06, 38.70, 30.24, 29.76, 14.24.

Ethyl (2*E*)-4-(3-Chloropropyl)-7-hydroxy-2-heptenoate (8b). Prepared from **7b** by reaction of **7b** with (carbethoxymethylene)triphenylphosphorane according to the general procedure outlined for the preparation of **3b** to afford **8b** in 62% yield after flash column chromatography with 30% EtOAc/hexanes; ^1H NMR (300 MHz, $CDCl_3$): δ 6.68 (dd, *J* = 15.63, 9.52 Hz, 1H), 5.78 (d, *J* = 15.63 Hz, 1H), 4.16 (q, *J* = 7.08 Hz, 2H), 3.61 (m, 2H), 3.49 (t, *J* = 6.59 Hz, 2H), 2.18 (m, 1H), 1.76-1.54 (m, 3H), 1.53-1.34 (m, 6H), 1.25 (t, *J* = 7.08 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.39, 152.08, 121.98, 62.67, 60.33, 44.86, 41.91, 31.61, 30.55, 30.23, 30.17, 14.23.

Ethyl (2*E*)-2-Ethyl-7-hydroxy-2-heptenoate (8c). Prepared from **7c** and (carbethoxymethylidene)triphenylphosphorane according to the general Wittig olefination procedure outlined for the preparation of **3b** to afford the desired α, β-unsaturated ester in 52% yield after flash column chromatography with 30% EtOAc/hexanes; ^1H NMR (500 MHz, $CDCl_3$): δ 6.70 (dd, *J* = 9.33,

15.68 Hz, 1H), 5.76 (d, J = 15.68 Hz, 1H), 4.16 (q, J = 7.15 Hz, 2H), 3.60 (t, J = 6.35 Hz, 2H), 2.04 (m, 1H), 1.55-1.44 (m, 4H), 1.39-1.30 (m, 3H), 1.27 (t, J = 7.15 Hz, 3H), 0.84 (t, J = 7.44 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 166.62, 152.91, 121.51, 62.81, 60.19, 44.10, 30.38, 30.14, 27.24, 14.24, 11.60; IR (neat) 3437.7, 1714.0 cm^{-1}; HRMS calcd for $C_{11}H_{20}O_3$: 200.1412; found 200.1395; LRMS (EI^+): *m/z* 200 (2), 155 (100), 125 (83), 109 (42), 99 (100), 81 (51), 67 (49), 55 (40), 41 (50), 29 (73).

***N*-Methoxy-*N*-methyl (*E*)-7-Hydroxy-4-(2-propenyl)-2-heptenamide (8d).** Prepared from tetrahydro-3-(2-propenyl)-(2H)-pyran-2-ol, **7a**, and *N*-methoxy-*N*-methyl-2-(triphenylphosphoranylidene)acetamide according to the general procedure outlined for the preparation of **3b** (except three drops of concentrated AcOH were added to the reaction mixture and $CHCl_3$ solvent replaced PhH) to afford the desired α, β-unsaturated amide in 100% yield after flash column chromatography with 20% EtOAc/hexanes; ^1H NMR (400 MHz, $CDCl_3$): δ 6.75 (dd, J = 15.38, 8.79 Hz, 1H), 6.34 (d, J = 15.38 Hz, 1H), 5.70 (m, 1H), 4.99 (m, 2H), 3.67 (s, 3H), 3.59 (t, J = 6.10 Hz, 2H), 3.22 (s, 3H), 2.27 (m, 1H), 2.17 (m, 2H), 2.02 (s, 1H), 1.59-1.34 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.66, 135.92, 150.67, 118.83, 116.54, 62.64, 61.63, 42.33, 38.86, 32.28, 30.29, 29.94; IR (neat) 3416.1, 1732.8, 1651.9 cm^{-1}.

Ethyl (2*E*)-7-Iodo-4-(2-propenyl)-2-hepteneoate (9a). General Procedure for the Preparation of Iodides from Alcohols. Prepared from **8a** according to the following general procedure. Triphenylphosphine (1.15 g, 4.4 mmol) and imidazole (0.30 g, 4.4 mmol) were dissolved in 8 mL of a 1:1 mixture of CH_3CN/Et_2O and the resultant clear solution was cooled to 0 °C in an ice bath. Then I_2 (1.12 g, 4.4 mmol) was added in small portions to the 0 °C cooled solution and the resultant yellow slurry was warmed to rt and stirred for 0.5 h. After this period of stirring, the reaction mixture was cooled to 0 °C and the alcohol **8a** (0.85 g, 4.0 mmol) was added in 5 mL of dry CH_3CN. The resultant slurry was maintained at 0 °C for 15 min and then warmed to rt and allowed to stir at ambient temperature for 18 h. The reaction mixture was quenched with saturated aqueous sodium thiosulfate. An aqueous workup followed by flash column chromatography with 5% EtOAc/hexanes afforded the desired iodide, **9a**, (1.08 g, 3.36 mmol) in 84% yield; ^1H NMR (400 MHz, $CDCl_3$): δ 6.72 (dd, J = 15.80,

8.84 Hz, 1H), 5.76 (dd, J = 15.80, 0.80 Hz, 1H), 5.72-5.65 (m, 1H), 5.04-5.00 (m, 2H), 4.17 (q, J = 7.23 Hz, 2H), 3.13 (dt, J = 6.69, 1.07 Hz, 2H), 2.24 (m, 1H), 2.14 (m, 2H), 1.84-1.72 (m, 2H), 1.60 (m, 1H), 1.42 (m, 1H), 1.27 (t, J = 7.23 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.41, 151.58, 135.34, 121.75, 117.04, 60.31, 41.45, 38.62, 34.45, 30.98, 14.29, 6.34.

Ethyl (2*E*)-4-(3-Chloropropyl)-7-iodo-2-heptenoate (9b). Prepared from **8b** according to the general iodination procedure outlined for the preparation of **9a** to afford **9b** in 84% yield after flash column chromatography with 5% EtOAc/hexanes; ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.67 (dd, J = 9.37, 15.53 Hz, 1H), 5.78 (d, J = 15.53 Hz, 1H), 4.17 (q, J = 7.23 Hz, 2H), 3.50 (t, J = 6.43 Hz, 2H), 3.13 (t, J = 6.96 Hz, 2H), 2.18 (m, 1H), 1.79-1.54 (m, 6H), 1.47-1.42 (m, 2H), 1.28 (t, J = 7.23 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.24, 151.51, 122.21, 60.38, 44.79, 41.29, 35.21, 31.55, 30.89, 30.10, 14.22, 6.39; LRMS (EI$^+$): *m/z* 358 (63), 323 (50), 313 (82), 295 (9), 277 (13), 249 (10), 231 (100), 185 (48), 157 (98), 121 (76), 107 (86), 93 (48), 81 (97), 67 (54), 55 (43), 41 (54), 29 (45).

Ethyl (2*E*)-2-Ethyl-7-iodo-2-heptenoate (9c). Prepared from ethyl (2*E*)-2-ethyl-7-hydroxy-2-heptenoate according to the general iodination procedure outlined for the preparation of **9a** to afford the desired iodide in 53% yield after flash column chromatography with 2% EtOAc/hexanes; ^{1}H NMR (500 MHz, $CDCl_3$): δ 6.68 (dd, J = 9.33, 15.58 Hz, 1H), 5.77 (d, J = 15.58 Hz, 1H), 4.17 (t, J = 7.15 Hz, 2H), 3.13 (t, J = 6.95 Hz, 2H), 2.06 (m, 1H), 1.81-1.67 (m, 2H), 1.59-1.54 (m, 1H), 1.48 (m, 1H), 1.46-1.33 (m, 2H), 1.28 (t, J = 7.15 Hz, 3H), 0.85 (t, J = 7.44 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 166.48, 152.33, 121.74, 60.26, 43.47, 34.85, 31.10, 27.19, 14.26, 11.58, 6.56; IR (neat) 1715.0, 1651.3, 1455.1 cm^{-1}.

***N*-Methoxy-*N*-methyl (2*E*)-7-Iodo-4-(2-propenyl)-2-heptenamide (9d).** Prepared from *N*-methoxy-*N*-methyl (2*E*)-7-hydroxy-4-(2-propenyl)-2-heptenamide, **8d**, according to the general procedure outlined for the preparation of **9a** to afford the desired iodide in 62% yield after flash column chromatography with 30% EtOAc/hexanes; ^{1}H NMR (500 MHz, $CDCl_3$): δ 6.73 (dd, J = 15.39, 9.03 Hz, 1H), 6.34 (d, J = 15.39 Hz, 1H), 5.79 (m, 1H), 5.07-4.99 (m, 2H), 3.68 (s, 3H), 3.22 (s, 3H), 3.12 (dt, J = 6.95, 1.79 Hz, 2H), 2.92 (m, 1H), 2.17 (m, 2H), 1.84-1.70 (m, 2H), 1.65-1.58 (m, 1H), 1.45-1.39 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.45, 150.02, 135.62, 119.08, 116.81, 61.70,

41.66, 38.82, 34.60, 32.27, 31.04, 6.44; IR (neat) 1667.2, 1633.8, 1416.7 cm^{-1}; HRMS calcd for $C_{12}H_{20}NIO_2$: 337.0539; found 337.0546; LRMS (EI^+): *m/z* 337 (100), 306 (10), 277 (98), 155 (11), 81 (21), 67 (19), 55 (58), 41 (16).

3-Methyloxepan-2-one. Prepared from ε-caprolactone by alkylation with methyl iodide according to the general alkylation procedure of **6b** to afford the desired lactone in 37% yield after flash column chromatography with 15% EtOAc/hexanes; ^1H NMR (400 MHz, $CDCl_3$): δ 4.24 (m, 2H), 2.68 (m, 1H), 1.91 (m, 2H), 1.75-1.58 (m, 3H), 1.48 (m, 1H), 1.17 (d, J = 6.69 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 178.09, 68.37, 37.39, 31.95, 28.88, 28.43, 18.48.

6-Hydroxy-2-methylhexanal. Prepared from 2-methyloxepan-2-one by reduction with DIBAL-H according to the general procedure outlined for the preparation of **7a** to afford the desired hydroxy aldehyde in 78% yield after flash column chromatography with 30% EtOAc/hexanes; ^1H NMR (400 MHz, $CDCl_3$): δ 9.56 (s, 1H), 3.63 (t, J = 6.69 Hz, 2H), 2.31 (m, 1H), 1.70 (m, 1H), 1.58-1.53 (m, 2H), 1.42-1.35 (m, 4H), 1.07 (d, J = 6.96 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 205.16, 62.54, 46.26, 32.60, 30.17, 23.18, 13.30.

Ethyl (2Z)-8-Hydroxy-4-methyl-2-octenoate. Prepared from 6-hydroxy-2-methylhexanal (*vide supra*) according to the following procedure. A solution of triethyl phosphonoacetate (1.35 g, 6.0 mmol) and 18-crown-6 (5.2 g, 19.7 mmol) in 50 mL of dry THF was cooled to -78 °C, and then $KN(TMS)_2$ (12 mL of a 0.5 M solution in toluene, 6.0 mmol) was added dropwise *via* syringe over 5 min. The reaction mixture was stirred at -78 °C for 30 min and then 6-hydroxy-2-methylhexanal (0.52 g, 4.0 mmol) was added dropwise in 10 mL of dry THF. The reaction mixture was stirred at -78 °C for 30 min and then allowed to warm to rt. The reaction mixture was quenched with saturated aqueous NH_4Cl and subjected to an aqueous workup. Flash column chromatography with 30% EtOAc/hexanes afforded a 9:1 mixture of Z and E isomers, respectively, in 72% combined yield; (major diastereomer, Z isomer) ^1H NMR (500 MHz, $CDCl_3$): δ 5.93 (dd, J = 10.24, 11.51 Hz, 1H), 5.69 (dd, J = 0.79, 11.51 Hz, 1H), 4.13 (q, J = 7.15 Hz, 2H), 3.61 (t, J = 6.55 Hz, 2H), 3.47 (m, 1H), 1.54 (m, 2H), 1.37-1.29 (m, 5H), 1.26 (t, J = 7.15 Hz, 3H), 0.98 (d, J = 6.65 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 166.46, 155.83, 118.48, 62.88, 59.80, 36.69, 32.77, 32.56, 23.47, 20.30, 14.24.

Ethyl (2*Z*)-8-Iodo-4-methyl-2-octenoate (12). Prepared from ethyl (2*Z*)-8-hydroxy-4-methyl-2-octenoate according to the general iodination procedure outlined for the preparation of **9a** to afford the desired iodide in 70% yield after flash column chromatography with 3% EtOAc/hexanes; ^{1}H NMR (400 MHz, $CDCl_3$): δ 5.92 (dd, J = 10.17, 11.25 Hz, 1H), 5.70 (d, J = 11.25 Hz, 1H), 4.14 (q, J = 6.96 Hz, 2H), 3.49 (m, 1H), 3.15 (t, J = 6.96 Hz, 2H), 1.78 (m, 3H), 1.39-1.34 (m, 3H), 1.30 (t, J = 6.96 Hz, 3H), 0.98 (d, J = 6.69 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.37, 155.49, 118.65, 59.83, 35.68, 33.67, 32.35, 28.16, 20.27, 14.27, 7.02; IR (neat) 1644.0, 1455.2, 1015.7 cm^{-1}.

Tetrahydro-(2H)-pyran-2-ol. Prepared according to the following general procedure. 3,4-Dihydro-(2*H*)-pyran (0.84 g, 10.0 mmol) in a 1:1 mixture of THF:H_2O was stirred for 2 h with catalytic *p*-TsOH. After this period of time, the reaction was quenched with saturated aqueous $NaHCO_3$ and subjected to an aqueous workup. Flash column chromatography of the crude product afforded the desired lactol (0.74 g, 8.6 mmol) in 86% yield in equilibrium with its corresponding hydroxy aldehyde; ^{1}H NMR (400 MHz, $CDCl_3$): δ 5.35 (m, 1H), 4.47 (m, 1H), 4.00 (m, 1H), 3.56 (m, 1H), 2.32-2.52 (m, 2H), 2.00 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 94.64, 62.93, 30.66, 25.41, 19.73.

Ethyl (2*E*)-7-Hydroxy-2-methyl-2-heptenoate (14a).[2] Prepared from tetrahydro-(2H)-pyran-2-ol, *vide supra*, and (carbethoxyethylidene)triphenylphosphorane according to the general procedure outlined for the preparation of **3b** to afford **14a** in 43% yield after flash column chromatography with 30% EtOAc/hexanes; ^{1}H NMR (500 MHz, $CDCl_3$): δ 6.73 (m, 1H), 4.16 (q, J = 7.08 Hz, 2H), 3.64 (t, J = 6.24 Hz, 2H), 2.18 (m, 2H), 1.81 (s, 3H), 1.60-1.49 (m, 4H), 1.38 (s, 1H), 1.27 (t, J = 7.08 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 168.22, 141.75, 128.03, 62.59, 60.40, 32.30, 28.32, 24.78, 14.25, 12.34.

Tetrahydro-3-methyl-(2H)-pyran-2-one. Prepared from δ-valerolactone by alkylation with MeI according to the general alkylation procedure of **6b** to afford the desired methylated product in 50% yield after flash column chromatography with 15% EtOAc/hexanes; ^{1}H NMR (400 MHz, $CDCl_3$): δ 4.33 (m, 2H), 2.58 (m, 1H), 2.10 (m, 1H), 1.91 (m, 2H), 1.55 (m, 1H), 1.26 (d, J = 7.1 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 175.22, 68.51, 34.61, 27.08, 22.01, 16.66.

Tetrahydro-3-methyl-(2H)-pyran-2-ol. Prepared from tetrahydro-3-methyl-(2H)-pyran-2-one by reduction with DIBAL-H according to the general procedure for **7a** to afford the desired lactol in 71% yield after flash column chromatography with 20% EtOAc/hexanes; ^{1}H NMR (500 MHz, $CDCl_3$): δ 4.96 (s, 0.33H), 4.31 (m, 0.67H), 3.98 (m, 0.67H), 3.92 (m, 0.33H), 3.54-3.45 (m, 1H), 3.25 (m, 1H), 1.83-1.79 (m, 1H), 1.59-1.41 (m, 3H), 1.23-1.15 (m, 1H), 0.94 (d, J = 6.85 Hz, 2H), 0.90 (d, J = 6.65 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): (major) δ 100.81, 65.93, 37.14, 30.30, 35.09, 16.82; (minor) δ 95.08, 60.12, 34.55, 25.75, 24.89, 16.13.

Ethyl (2*E*)-2,6-Dimethyl-7-hydroxy-2-heptenoate (14b). Prepared from tetrahydro-3-methyl-(2H)-pyran-2-ol and (carbethoxyethylidene)triphenyl-phosphorane according to the general procedure for the preparation of **3b** to afford **14b** in 64% yield after flash column chromatography with 30% EtOAc/hexanes; ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.49 (dd, J = 10.17, 1.34 Hz, 1H), 4.15 (q, J = 7.23 Hz, 2H), 3.58 (t, J = 6.16 Hz, 2H), 2.48 (m, 1H), 1.80 (s, 3H), 1.56-1.42 (m, 5H), 1.24 (t, J = 7.23 Hz, 3H), 0.96 (d, J = 6.69 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 168.40, 147.44, 126.45, 62.85, 60.44, 33.04, 32.89, 30.61, 20.04, 14.24, 12.51.

Ethyl (2*E*)-8-Hydroxy-2-methyl-2-octenoate (14c). Prepared from 6-hydroxyhexanal (*vide supra*) and (carbethoxyethylidene)triphenylphosphorane according to the general procedure outlined for the preparation of **3b** to afford the desired α,β-unsaturated ester in 93% yield after flash column chromatography with 30% EtOAc/hexanes; ^{1}H NMR (300 MHz, $CDCl_3$): δ 6.73 (dt, J = 1.46, 6.10 Hz, 1H), 4.16 (q, J = 7.08 Hz, 2H), 3.63 (t, J = 6.35 Hz, 2H), 2.16 (m, 2H), 1.80 (s, 3H), 1.54 (m, 2H), 1.52-1.34 (m, 5H), 1.27 (t, J = 7.08 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 168.26, 141.99, 127.86, 62.79, 60.38, 32.54, 28.58, 28.37, 25.51, 14.26, 12.32; IR (neat) 3429.9, 1711.1, 1650.0, 1462.2 cm^{-1}.

Ethyl (2*E*)-7-Iodo-2-methyl-2-heptenoate (15a). Prepared from **14a** according to the general iodination procedure of **9a** to afford **15a** in 69% yield after flash column chromatography with 5% EtOAc/hexanes; ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.71 (m, 1H), 4.16 (q, J = 7.23 Hz, 2H), 3.17 (t, J = 6.96 Hz, 2H), 2.19 (m, 2H), 1.86 (m, 2H), 1.81 (s, 3H), 1.58-1.51 (m, 2H), 1.27 (t, J = 7.23 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 168.05, 141.03, 128.37, 60.43, 32.94, 29.40, 27.49, 14.26, 12.37,

6.32; LRMS (EI^+): *m/z* 296 (100), 268 (9), 251 (43), 169 (21), 155 (13), 141 (22), 123 (12), 113 (21), 95 (100), 81 (14), 67 (31), 55 (49), 41 (34), 29 (24).

Ethyl (2*E*)-2,6-Dimethyl-7-iodo-2-heptenoate (15b). Prepared from **14b** according to the general iodination procedure of **9a** to afford **15b** in 55% yield after flash column chromatography with 5% EtOAc/hexanes; ^1H NMR (400 MHz, $CDCl_3$): δ 6.48 (d, *J* = 10.17 Hz, 1H), 4.17 (q, *J* = 7.23 Hz, 2H), 3.13 (t, *J* = 6.69 Hz, 2H), 2.50 (m, 1H), 1.81 (s, 3H), 1.77 (m, 2H), 1.52 (m, 1H), 1.36 (m, 1H), 1.28 (t, *J* = 7.23 Hz, 3H), 0.99 (d, *J* = 6.69 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 168.26, 146.83, 126.98, 60.51, 37.58, 32.49, 31.42, 19.98, 14.28, 12.55, 6.60; LRMS (EI^+): *m/z* 310 (100), 265 (91), 183 (61), 155 (23), 137 (42), 109 (97), 95 (23), 81 (22), 67 (40), 55 (22), 41 (56), 29 (34).

Ethyl (2*E*)-8-Iodo-2-methyl-2-octenoate (15c). Prepared from ethyl (2*E*)-8-hydroxy-2-methyl-2-octenoate according to the general iodination procedure outlined for the preparation of **9a** to afford the desired iodide in 70% isolated yield after flash column chromatography with 2% EtOAc/hexanes; ^1H NMR (400 MHz, $CDCl_3$): δ 6.72 (dt, *J* = 7.50, 1.34 Hz, 1H), 4.16 (q, *J* = 7.32 Hz, 2H), 3.17 (t, *J* = 6.96 Hz, 2H), 2.16 (m, 2H), 1.81 (m, 5H), 1.44 (m, 4H), 1.27 (t, *J* = 7.23 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 168.18, 141.63, 128.04, 60.41, 33.25, 30.20, 28.39, 27.49, 14.27, 12.37, 6.73; IR (neat) 1714.3, 1651.2, 1455.5 cm^{-1}; HRMS calcd for $C_{11}H_{19}IO_2$: 310.0430; found 310.0440; LRMS (EI^+): *m/z* 310 (100), 265 (41), 109 (98), 99 (40), 81 (60), 67 (41), 55 (80), 39 (40), 29 (71).

6-*n*-Pentylpyran-2-ol (18a). Prepared from δ-decanolactone by DIBAL-H reduction according to the general procedure outlined for the preparation of **7a** to afford the desired lactol in 75% yield after flash column chromatography with 15% EtOAc/hexanes; ^1H NMR (400 MHz, $CDCl_3$): δ 5.28 (m, 0.33H), 4.67 (m, 0.67H), 3.89 (m, 0.67H), 3.39 (m, 0.67H), 3.01 (d, *J* = 6.16 Hz, 0.33H), 2.49 (m, 0.33H), 1.83 (m, 0.33H), 1.71-1.09 (m, 11H), 0.86 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 96.45, 91.83, 76.52, 68.77, 36.09, 35.93, 32.85, 31.90, 31.84, 31.09, 30.35, 29.84, 25.16, 25.05, 22.60, 22.58, 22.10, 17.42, 14.02; IR (neat) 3395.2, 1742.3, 1461.2 cm^{-1}.

7-Methyloxepan-2-one (18c). Prepared from 2-methylcyclohexanone according to the following general procedure. Trifluoroacetic anhydride (4.20 g, 20.0 mmol) was added slowly dropwise

to a 0 °C cooled solution of 2-methylcyclohexanone (1.56 g, 13.9 mmol) and urea hydrogen peroxide addition compound (7.52 g, 80.0 mmol) in 50 mL of dry CH_2Cl_2. TLC analysis after 1.5 h revealed the complete consumption of the starting ketone and formation of a single, lower R_f product. The reaction mixture was quenched after this period of time by *careful* addition of saturated aqueous $NaHCO_3$ followed by *careful* addition of saturated aqueous sodium bisulfite. The reaction mixture was subjected to an aqueous workup and concentrated *in vacuo* after verifying the absence of residual peroxides (KI/starch paper). Flash column chromatography with 20% EtOAc/hexanes afforded the desired lactone (1.26 g, 9.87 mmol) in 71% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 4.42 (m, 1H), 2.61 (m, 2H), 1.92-1.84 (m, 3H), 1.67-1.53 (m, 3H), 1.32 (d, J = 6.43 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 175.55, 76.78, 36.22, 34.98, 28.25, 22.88, 22.54.

Ethyl (2*E*)-7-Hydroxy-2-dodecenoate (19a). Prepared from 6-*n*-pentylpyran-2-ol and (carbethoxymethylidene)triphenylphosphorane according to the general procedure outlined for the preparation of **3b** to afford the desired α, β-unsaturated ester in 94% yield after flash column chromatography with 20% EtOAc/hexanes; ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.94 (dt, J = 15.63, 6.84 Hz, 1H), 5.79 (d, J = 15.63 Hz, 1H), 4.15 (q, J = 7.08 Hz, 2H), 3.57 (m, 1H), 2.20 (m, 1H), 1.01 (m, 1H), 1.54-1.31 (m, 9H), 1.30-1.21 (m, 7H), 0.87 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.69, 148.94, 121.49, 71.63, 60.14, 37.50, 36.76, 32.11, 31.93, 25.27, 24.10, 22.60, 14.24, 14.00; IR (neat) 3418.0, 1716.2, 1652.4 cm^{-1}.

6-Hydroxyheptanal (19c). Prepared from 7-methyloxepan-2-one, **18c**, according to the general procedure outlined for the preparation of **7a** to afford the desired hydroxy aldehyde in 76% yield after flash column chromatography with 50% EtOAc/hexanes; ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.76 (s, 1H), 3.78 (m, 1H), 2.44 (dt, J = 1.61, 7.23 Hz, 2H), 1.63 (m, 3H), 1.66-1.24 (m, 4H), 1.17 (d, J = 6.16 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 202.55, 67.77, 43.82, 38.89, 25.28, 23.56, 21.99.

Ethyl (2*E*)-7-Hydroxy-2-methyl-2-dodecenoate (20a). Prepared from 6-*n*-pentylpyran-2-ol and (carbethoxyethylidene)triphenylphosphorane according to the general procedure outlined for the preparation of **3b** to afford the desired α, β-unsaturated ester in 91% yield after flash column chromatography with 20% EtOAc/hexanes; ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.73 (m, 1H), 4.16 (q, J =

6.96 Hz, 2H), 3.57 (m, 1H), 2.17 (m, 2H), 1.80 (s, 3H), 1.58 (m, 1H), 1.54-1.33 (m, 7H), 1.30-1.22 (m, 8H), 0.87 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 168.23, 141.89, 127.96, 71.71, 60.39, 37.49, 37.01, 31.84, 28.60, 25.28, 24.69, 22.61, 14.27, 14.01, 12.36; IR (neat) 3409.9, 1697.9, 1651.5 cm^{-1}; HRMS calcd for $C_{15}H_{28}O_3$: 256.2038; found: 256.2054; LRMS (EI^+): *m/z* 238 (48), 210 (100), 140 (74), 128 (42), 112 (99), 95 (51), 83 (50), 69 (45), 55 (97), 41 (81), 29 (60).

Ethyl (2*E*)-8-Hydroxy-2-nonenoate (20c). Prepared from 6-hydroxyheptanal, **19c**, and (carbethoxymethylidene)triphenylphosphorane according to the general procedure outlined for the preparation of **3b** to afford **20c** in 86% yield after flash column chromatography with 30% EtOAc/hexanes; ^1H NMR (400 MHz, $CDCl_3$): δ 7.41 (dt, *J* = 6.69, 15.53 Hz, 1H), 6.27 (d, *J* = 15.53 Hz, 1H), 4.64 (q, *J* = 7.23 Hz, 2H), 4.25 (m, 1H), 2.67 (m, 2H), 1.95-1.77 (m, 7H), 1.75 (t, *J* = 7.23 Hz, 3H), 1.65 (d, *J* = 6.16 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.69, 149.01, 121.43, 67.91, 60.11, 39.01, 32.10, 28.02, 25.28, 23.55, 14.24.

***N*-Methoxy-*N*-methyl (2*E*)-7-Hydroxy-2-dodecenamide (20d).** Prepared from 6-*n*-pentylpyran-2-ol and *N*-methoxy-*N*-methyl-2-(triphenylphosphor-anylidene)acetamide according to the general procedure outlined for the preparation of **3b** (except 3 drops of AcOH was added) to afford the desired α, β-unsaturated hydroxy amide in 77% yield after flash column chromatography with 65% EtOAc/hexanes; ^1H NMR (500 MHz, $CDCl_3$): δ 6.95 (dt, *J* = 6.95, 15.38 Hz, 1H), 6.38 (d, *J* = 15.38 Hz, 1H), 3.68 (s, 3H), 3.58 (m, 1H), (3.22, s, 3H), 2.24 (m, 2H), 1.62 (m, 1H), 1.52-1.37 (m, 7H), 1.31-1.22 (m, 5H), 0.87 (t, *J* = 6.95 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.94, 147.51, 118.81, 71.62, 61.64, 37.51, 36.84, 32.41, 32.29, 31.83, 25.28, 24.35, 22.59, 14.01; IR (neat) 1721.9, 1691.4 cm^{-1}; HRMS calcd for $C_{14}H_{27}NO_3$: 257.1991; found, 257.1968; LRMS (EI^+): *m/z* 258 (65), 257 (51), 240 (100), 226 (35), 208 (10), 197 (11), 186 (19), 151 (23), 129 (6), 109 (20), 95 (60), 81 (100), 67 (28), 55 (33), 41 (34), 29 (18).

Ethyl (2*E*)-7-Iodo-2-dodecenoate (21a). Prepared from ethyl (2*E*)-7-hydroxy-2-dodecenoate according to the general procedure outlined for the preparation of **9a** to afford the desired iodide in 84% yield after flash column chromatography with 2% EtOAc/hexanes; ^1H NMR (400 MHz, $CDCl_3$): δ 6.93 (dt, *J* = 6.96, 15.53 Hz, 1H), 5.81 (dt, *J* = 1.61, 15.53 Hz, 1H), 4.16 (q, *J* = 6.96 Hz,

2H), 4.07 (m, 1H), 2.21 (m, 2H), 1.83 (m, 2H), 1.77-1.61 (m, 3H), 1.60-1.44 (m, 2H), 1.41-1.21 (m, 5H), 1.27 (t, J = 6.96 Hz, 3H), 0.87 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.56, 148.29, 121.81, 60.20, 40.61, 39.87, 39.34, 31.29, 30.98, 29.16, 28.02, 22.48, 14.26, 13.99; IR (neat) 1654.2, 1456.6 cm^{-1}; HRMS calcd for $C_{14}H_{25}IO_2$: 353.0977; found 353.0986; LRMS (EI$^+$): 353 (13), 307 (100), 225 (38), 179 (12), 151 (81), 109 (50), 95 (99), 81 (96), 67 (57), 55 (76), 41 (68), 29 (71); Analysis calcd for $C_{14}H_{25}IO_2$: C, 47.74; H, 7.15; found: C, 47.34; H, 7.15.

Ethyl (2*E*)-7-Iodo-2-methyl-2-dodecenoate (21b). Prepared from ethyl (2*E*)-7-hydroxy-2-methyl-2-dodecenoate, **20a**, according to the general procedure outlined for the preparation of **9a** to afford the desired iodide in 98% yield after flash column chromatography with 2% EtOAc/hexanes; ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.72 (dt, J = 1.61, 7.50 Hz, 1H), 4.16 (q, J = 7.23 Hz, 2H), 4.07 (m, 1H), 2.18 (m, 2H), 1.85 (m, 2H), 1.81 (s, 3H), 1.71-1.60 (m, 3H), 1.58-1.44 (m, 2H), 1.42-1.24 (m, 5H), 1.28 (t, J = 7.23 Hz, 3H), 0.87 (m, 3H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 168.11, 141.28, 128.27, 60.44, 40.58, 40.09, 39.65, 30.96, 29.15, 28.58, 27.79, 22.47, 14.27, 13.99, 12.40; IR (neat) 1714.0, 1651.2, 1455.5 cm^{-1}; HRMS calcd for $C_{15}H_{27}IO_2$: 366.1056; found: 366.1064; LRMS (EI$^+$): 321 (18), 239 (77), 193 (33), 105 (95), 137 (83), 95 (95), 69 (87), 41 (82).

Ethyl (2*E*)-8-Iodo-2-nonenoate (21c). Prepared from ethyl (2*E*)-8-hydroxy-2-nonenoate according to the general procedure outlined for the preparation of **9a** to afford the desired secondary iodide in 90% yield after flash column chromatography with 5% EtOAc/hexanes: ^{1}H NMR (500 MHz, $CDCl_3$): δ 6.93 (dt, J = 15.68, 6.95 Hz, 1H), 5.80 (dt, J = 15.68, 1.49 Hz, 1H), 4.19-4.13 (m, 3H), 2.20 (m, 2H), 1.89 (d, J = 6.85 Hz, 3H), 1.79 (m, 1H), 1.60 (m, 1H), 1.54-1.36 (m, 4H), 1.27 (t, J = 7.15 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 166.62, 148.69, 121.55, 60.15, 42.55, 31.95, 29.97, 29.24, 28.90, 27.13, 14.26; HRMS calcd for $C_{11}H_{19}IO_2$: 310.0430; found 310.0425; LRMS (EI$^+$): *m/z* 310 (5), 265 (100), 183 (31), 137 (24), 109 (98), 95 (41), 81 (25), 67 (53), 55 (51), 29 (60).

***N*-Methoxy-*N*-methyl (2*E*)-7-Iodo-2-dodecenamide (21d).** Prepared from *N*-methoxy-*N*-methyl (2*E*)-7-hydroxy-2-dodecenamide (**20d**) according to the general procedure outlined for the preparation of **9a** to afford the desired iodide in 74% yield after flash column chromatography with 20% EtOAc/hexanes; ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.94 (dt, J = 6.96, 15.53 Hz, 1H), 6.39 (d, J = 5.53

Hz, 1H), 4.08 (m, 1H), 3.68 (s, 3H), 3.22 (s, 3H), 2.24 (m, 2H), 1.83 (m, 2H), 1.73-1.43 (m, 5H), 1.42-1.18 (m, 5H), 0.87 (t, J = 6.96 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.79, 146.81, 119.13, 61.68, 40.57, 35.88, 39.60, 32.29, 31.50, 30.94, 29.13, 28.20, 22.46, 13.99; IR (neat) 1721.9, 1690.2 cm^{-1}; HRMS calcd for $C_{14}H_{26}NO_2I$: 367.1008, found 367.1026; LRMS (EI^+): *m/z* 367 (2), 307 (37), 240 (82), 179 (12), 151 (10), 109 (27), 95 (67), 81 (87), 55 (100).

Tetrahydro-6,6-dimethylpyran-2(2H)-one. Prepared from 2,2-dimethylcyclopentanone according to the general oxidation procedure outlined for the preparation of **18c** to afford the desired bicyclic lactone in 61% isolated yield after flash column chromatography with 15% EtOAc/hexanes; ^1H NMR (500 MHz, $CDCl_3$): δ 2.50 (t, J = 6.95 Hz, 2H), 1.90 (m, 2H), 1.77 (m, 2H), 1.43 (s, 6H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 171.34, 82.19, 33.84, 29.03, 28.65 (2), 16.75.

Dihydro-6,6-dimethylpyran-2(2H)-ol. Prepared from dihydro-6,6-dimethylpyran-2(2H)-one by reduction with DIBAL-H according to the general procedure outlined for the preparation of **7a** to afford the desired lactol in 66% yield after Kugelrohr distillation (ot 100-120 °C at 0.1 mm Hg); ^1H NMR (500 MHz, $CDCl_3$): δ 4.97 (m, 0.7H), 4.60 (m, 0.3H), 3.77 (m, 0.3H), 2.76 (s, 0.7H), 1.97 (m, 0.3H), 1.81 (m, 1H), 1.73 (m, 1H), 1.72-1.57 (m, 1.7H), 1.41 (m, 2H), 1.27 (s, 3H), 1.20 (s, 3H); IR (neat) 3387.2, 1724.9, 1651.7, 1557.6, 1461.9 cm^{-1}; HRMS calcd for $C_7H_{13}O_2$ ($M-H^+$): 129.0922; found 129.0916; LRMS (EI^+): *m/z* 129 (100), 115 (12), 102 (22), 71 (30), 59 (98), 43 (37).

Ethyl (2*E*)-7-Hydroxy-7-methyl-2-octenoate. Prepared from dihydro-6,6-dimethylpyran-2(2H)-ol and (carbethoxymethylidene)triphenylphosphorane according to the general procedure outlined for the preparation of **3b** to afford the desired α, β-unsaturated ester in 94% yield after flash column chromatography with 25% EtOAc/hexanes; ^1H NMR (400 MHz, $CDCl_3$): δ 6.94 (dt, J = 15.80, 6.96 Hz, 1H), 5.80 (d, J = 15.80 Hz, 1H), 4.16 (q, J = 7.23 Hz, 2H), 2.21 (m, 2H), 1.55-1.43 (m, 5H), 1.27 (t, J = 7.23 Hz, 3H), 1.19 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.67, 148.91, 121.50, 70.74, 60.14, 43.19, 32.49, 29.22 (2), 22.78, 14.23; IR (neat) 3451.0, 1722.0, 1650.3 cm^{-1}; HRMS calcd for $C_{11}H_{18}O_3$ ($M-H_2O$): 182.1307; found 182.1295; LRMS (EI^+): *m/z* 182 (100), 155 (62), 139 (50), 127 (51), 114 (72), 81 (75), 59 (86), 43 (95), 29 (68).

Ethyl (2*E*)-7-Iodo-7-methyl-2-octenoate (23). Prepared from ethyl (2*E*)-7-hydroxy-7-methyl-2-octenoate according to the following general procedure.[3] TMSI (0.30 g, 1.50 mmol) was added to the tertiary alcohol (0.20 g, 1.0 mmol) in 5 mL of dry CH_2Cl_2 and the resultant reaction mixture was stirred for 24 h at rt. After this period of time, the reaction was quenched with saturated aqueous sodium bisulfite and subjected to an aqueous workup. Flash column chromatography with 2% EtOAc/hexanes afforded the desired halide (0.26 g, 0.83 mmol) in 83% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.94 (dt, J = 6.96, 15.80 Hz, 1H), 5.83 (dt, J = 1.61, 15.80 Hz, 1H), 4.17 (q, J = 7.23 Hz, 2H), 2.23 (dq, J = 1.61, 7.23 Hz, 2H), 1.90 (s, 6H), 1.69 (m, 2H), 1.59 (m, 2H), 1.27 (t, J = 7.23 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.56, 148.35, 121.79, 60.20, 51.35, 49.71, 37.99 (2), 31.72, 27.04, 14.25; IR (neat) 1718.6, 1654.2 cm^{-1}; HRMS calcd for $C_{11}H_{19}IO_2$: 311.0508; found 311.0478; LRMS (EI$^+$): *m/z* 311 (100), 265 (49), 183 (32), 137 (42), 109 (98), 95 (64), 81 (31), 69 (91), 55 (50), 39 (39), 29 (74).

2-Oxabicyclo[3.2.1]octan-3-one (25). Prepared from norcamphor by a Baeyer-Villiger oxidation according to the general procedure outlined for the preparation of 7-methyloxepan-2-one, **18c**, to afford the desired bicyclic lactone in 74% isolated yield after flash column chromatography with 20% EtOAc/hexanes; ^{1}H NMR (500 MHz, $CDCl_3$): δ 4.82 (m, 1H), 2.68 (ddd, J = 18.56, 5.06, 2.28 Hz, 1H), 2.52 (m, 1H), 2.43 (m, 1H), 2.11 (m, 1H), 2.00-1.88 (m, 3H), 1.70-1.60 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 170.67, 80.97, 40.61, 35.75, 32.42, 31.75, 29.20.

2-Oxabicyclo[3.2.1]octan-3-ol (26). Prepared from 2-oxabicyclo[3.2.1]octan-3-one, **25**, by reduction with DIBAL-H according to the general procedure outlined for the preparation of **7a** to afford the desired lactol/hydroxy aldehyde in 100% yield after flash column chromatography with 25% EtOAc/hexanes; ^{1}H NMR (400 MHz, $CDCl_3$): δ 9.74 (s, 0.5H), 5.21 (t, J = 5.62 Hz, 0.17H), 4.31 (m, 0.58H), 3.78 (m, 0.25H), 2.57 (m, 0.5H), 2.41-2.10 (m, 2H), 2.04-1.54 (m, 5H), 1.44 (m, 1H), 1.25 (m, 1H), 0.90 (d, J = 6.09 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 202.42, 99.37, 81.50, 73.44, 73.40, 60.37, 50.55, 42.27, 41.69, 40.64, 39.56, 35.38, 35.31, 35.27, 34.14, 32.44, 32.18, 30.36, 30.31, 30.08, 26.98, 19.29, 14.14; IR (neat) 3380.3, 1715.0 cm^{-1}; HRMS calcd for $C_7H_{11}O_2$ (M-H$^+$): 127.0759; found 127.0751; LRMS (EI$^+$): *m/z* 127 (8), 111 (98), 82 (22), 67 (97), 55 (30), 41 (37).

Ethyl (*E*)-4-[(1*R, 3*R**)-3-Hydroxycyclopentyl]-2-butenoate (27a).** Prepared from 2-oxabicyclo[3.2.1]octan-3-ol, **26**, and (carbethoxymethylidene)triphenylphosphorane according to the general Wittig olefination procedure outlined for the preparation of **3b** to afford the desired α, β-unsaturated ester in 76% yield after flash column chromatography with 20% EtOAc/hexanes; ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.91 (dt, *J* = 15.63, 7.08 Hz, 1H), 5.80 (d, *J* = 15.63 Hz, 1H), 4.30 (m, 1H), 4.16 (q, *J* = 7.08 Hz, 2H), 2.82 (t, *J* = 7.33 Hz, 2H), 2.13 (m, 1H), 1.97 (m, 2H), 1.77 (m, 2H), 1.63 (m, 1H), 1.43 (m, 2H), 1.26 (t, *J* = 7.08 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.71, 148.18, 121.88, 73.48, 60.15, 41.78, 39.10, 37.14, 35.33, 29.96, 14.23; IR (neat) 3435.6, 1776.8, 1692.5 cm^{-1}; HRMS calcd for $C_{11}H_{18}O_3$: 198.1256; found 198.1243; LRMS (EI^+): *m/z* 180 (42), 152 (81), 135 (100), 125 (34), 114 (52), 107 (62), 86 (80), 67 (98), 55 (31), 41 (72), 29 (72).

***N*-Methoxy-*N*-methyl (2*E*)-[(1*R**, 3*S**)-3-Hydroxycyclopentyl]-2-butenamide (27b).** Prepared from 2-oxabicyclo[3.2.1]octan-3-ol, **26**, and *N*-methoxy-*N*-methyl-2-(triphenylsphosporanylidene)acetamide according to the general procedure outlined for the preparation of **3b** (except three drops of concentrated AcOH were added to the reaction mixture) to afford the desired α, β-unsaturated hydroxy amide in 81% yield after flash column chromatography with 25% EtOAc/hexanes; ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.92 (dt, *J* = 15.56, 7.50 Hz, 1H), 6.38 (d, *J* = 15.56 Hz, 1H), 4.30 (m, 1H), 3.67 (s, 3H), 3.22 (s, 3H), 2.32 (t, *J* = 7.23 Hz, 2H), 1.14 (m, 1H), 1.99 (m, 1H), 1.77 (m, 2H), 1.63-1.38 (m, 3H), 1.23 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.93, 146.67, 119.28, 73.50, 61.62, 41.85, 39.50, 37.63, 35.35, 32.30, 29.99; IR (neat) 3415.9, 1735.0, 1653.0, 1436.1 cm^{-1}.

Ethyl (*E*)-4-[(1*R, 3*R**/*S**)-3-Iodocyclopentyl]-2-butenoate (28a).** Prepared from ethyl (*E*)-4-[(1*R**, 3*R**)-3-hydroxycyclopentyl]-2-butenoate according to the general iodination procedure outlined for the preparation of **9a** to afford the desired iodide as a 1:1 mixture of diastereomeric products epimeric at C-3 in 68% yield after flash column chromatography with 2% EtOAc/hexanes; ^{1}H NMR (500 MHz, $CDCl_3$): δ 6.88 (m, 1H), 5.80 (m, 1H), 4.16 (m, 3H), 2.48 (m, 1H), 2.33 (m, 1H), 2.26-2.01 (m, 4H), 1.84-1.65 (m, 2H), 1.48 (m, 1H), 1.26 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 166.48, 166.45, 147.29, 147.24, 122.37, 122.30, 60.22, 45.99, 45.75, 39.44, 39.27, 38.60, 38.15, 37.94,

36.76, 31.13, 30.05, 27.34, 23.43, 14.24; IR (neat) 1722.6, 1658.0 cm^{-1}; HRMS calcd for $C_{11}H_{17}IO_2$: 308.0273; found 308.0256; LRMS (EI^+): *m/z* 308 (8), 263 (100), 181 (33), 135 (40), 107 (47), 81 (30), 67 (98), 55 (17), 41 (36), 28 (28), 18 (40).

***N*-Methoxy-*N*-methyl (2*E*)-[(1*R**, 3*R**/*S**)-3-Iodocyclopentyl]-2-butenamide (28b).** Prepared from *N*-methoxy-*N*-methyl (2*E*)-[(1*R**, 3*S**)-3-hydroxycyclopentyl]-2-butenamide (**27b**) according to the general procedure outlined for the preparation of **9a** to afford the desired iodide as a 1:1 mixture of diastereomeric iodides epimeric at C-3 in 43% yield after flash column chromatography with 20% EtOAc/hexanes; ^1H NMR (500 MHz, $CDCl_3$): δ 6.90 (m, 1H), 6.38 (m, 1H), 4.39 (m, 0.5H), 4.13 (m, 0.5H), 3.68 (s, 3H), 3.22 (s, 3H), 2.52-2.44 (m, 1H), 2.37 (m, 1H), 2.28 (m, 1H), 2.22-2.01 (m, 3H), 1.81 (m, 1H), 1.72 (m, 1H), 1.49 (m, 0.5H), 1.26 (m, 0.5H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.72, 145.80, 145.71, 119.75, 119.67, 61.67, 46.02, 45.72, 39.46, 39.29, 38.94, 38.36, 38.18, 36.95, 32.28, 31.11, 30.63, 27.46, 23.63; IR (neat) 1666.2, 1633.4, 1462.3 cm^{-1}; HRMS calcd for $C_{11}H_{18}NO_2I$: 323.0344; found 323.0382; LRMS (EI^+): *m/z* 323 (9), 263 (100), 196 (32), 135 (41), 79 (31), 67 (91), 55 (98), 41 (42).

Ethyl (1*R/*S**, 3*R**/*S**)-2-Oxo-3-(2-propenyl)cyclopentanecarboxylate (31a).** Prepared according to the following general procedure. Ethyl 2-oxo-1-(2-propenyl)cyclopentanecarboxylate, **30a**, (0.98 g, 5.0 mmol) was added dropwise *via* cannula to a stirred solution of KH (0.28 g, 7.0 mmol) and 18-Crown-6 (6.61 g, 25.0 mmol) in 10 mL of dry THF at ambient temperature. The resultant reaction mixture was stirred for 1 h at ambient temperature and then quenched by the careful addition of 10 mL of saturated aqueous NH_4Cl. The crude reaction mixture was subjected to an aqueous workup followed by Kugelrohr distillation (ot 100-110 °C @ 10 mm Hg) to afford the desired rearranged product in 99% yield; ^1H NMR (300 MHz, $CDCl_3$): δ 5.72 (m, 1H), 5.03 (m, 2H), 4.17 (m, 2H), 3.23 (m, 0.25H), 3.08 (m, 0.5H), 2.81 (m, 0.25H), 2.56-1.80 (m, 6H), 1.52 (m, 1H), 1.26 (m, 3H); ^{13}C NMR (300 MHz, $CDCl_3$): δ 212.38, 169.42, 169.26, 135.38, 175.18, 133.00, 116.89, 61.33, 55.03, 54.19, 48.79, 48.27, 38.05, 37.80, 34.11, 33.70, 26.88, 26.69, 25.02, 24.98, 19.48, 14.14.

Ethyl (1*R, 2*S**/*R**)-2-Oxo-3-(3-butenyl)cyclopentanecarboxylate (31b).** Prepared from ethyl 2-oxo-1-(3-butenyl)cyclopentanecarboxylate, **30b**, according to the general procedure outlined for the preparation of ethyl (1*R**/*S**, 3*R**/*S**)-2-oxo-3-(2-propenyl)cyclopentanecarboxylate, **31a**, to afford the desired product in 84% yield after flash column chromatography with 3% EtOAc/hexanes; ^{1}H NMR (500 MHz, $CDCl_3$): δ 5.74 (m, 1H), 4.96 (m, 2H), 4.13 (m, 1H), 2.52 (m, 1H), 2.41 (m, 1H), 2.21 (m, 1H), 2.10-1.86 (m, 7H), 1.63 (m, 1H), 1.23 (t, J = 7.23 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 214.54, 170.80, 137.61, 114.96, 61.28, 60.12, 37.84, 32.93, 32.76, 29.05, 19.53, 14.02.

Ethyl 2-Hydroxy-3-(2-propenyl)cyclopentanecarboxylate. Ethyl (1*R**/*S**, 3*R**/*S**)-2-oxo-3-(2-propenyl)cyclopentanecarboxylate (2.90 g, 14.8 mmol) was added slowly dropwise in 10 mL of dry ethanol to a 0 °C cooled solution of $NaBH_4$ (0.56 g, 14.8 mmol) in 20 mL of dry ethanol and the resultant reaction mixture was stirred for 0.5 h at 0 °C and then quenched with saturated aqueous Na_2SO_4. The crude reaction mixture was subjected to an aqueous workup followed by Kugelrohr distillation to afford the desired alcohol (2.42 g, 12.2 mmol) in 83% yield after Kugelrohr distillation (ot 110-120 °C @ 0.1 mm Hg); ^{1}H NMR (400 MHz, $CDCl_3$): δ 5.79 (m, 1H), 5.03 (m, 2H), 4.32 (m, 0.33H), 4.26 (m, 0.33H), 4.01 (m, 0.34H), 2.76 (m, 1H), 2.39-2.08 (m, 3H), 2.03-1.77 (m, 7H), 1.24 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 175.03, 174.98, 174.77, 137.68, 136.98, 136.80, 115.93, 115.47, 80.18, 77.86, 76.80, 74.81, 60.61, 60.56, 52.24, 51.59, 49.31, 47.89, 46.21, 45.88, 45.30, 43.64, 38.00, 37.35, 33.55, 33.02, 31.89, 29.66, 29.32, 28.66, 28.53, 28.00, 27.35, 26.25, 26.22, 25.24, 24.45, 14.19; IR (neat) 3454.3, 1726.0, 1678.7 cm^{-1}; HRMS calcd for $C_{11}H_{18}O_3$: 198.1256; found, 198.1267; LRMS (EI$^+$): *m/z* 198 (3), 180 (19), 156 (87), 134 (40), 107 (100), 101 (40), 95 (42), 83 (61), 73 (60), 55 (54), 41 (68), 29 (52).

Ethyl 3-(2-Propenyl)-1-cyclopentenecarboxylate (32a). Prepared from ethyl 2-hydroxy-3-(2-propenyl)cyclopentanecarboxylate according to the following procedure. Triethylamine (1.34 g, 13.3 mmol) was added in portions to a 0 °C cooled solution of methanesulfonyl chloride (1.53 g, 13.3 mmol), ethyl 2-hydroxy-3-(2-propenyl)cyclopentanecarboxylate (2.40 g, 12.1 mmol), and catalytic DMAP. Then the reaction was stirred for 1 h at 0 °C. After this period of time, the reaction mixture was quenched with H_2O and then subjected to an aqueous workup and concentrated *in vacuo* to afford the

crude mesylate. The crude reaction mixture was taken up in THF, and then DBU (2.31 g, 15.0 mmol) was added in portions *via* syringe and the reaction mixture was stirred for 2 h at ambient temperature. The reaction mixture was quenched with H_2O and then subjected to an aqueous workup. The crude reaction mixture was purified by Kugelrohr distillation to afford the desired diene (1.89 g, 10.5 mmol) in 86% yield for the two steps; ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.66 (m, 1H), 5.76 (m, 1H), 5.01 (m, 2H), 4.16 (dq, J = 1.61, 6.96 Hz, 2H), 2.88 (m, 1H), 2.62-2.44 (m, 2H), 2.21-2.06 (m, 3H), 1.56 (m, 1H), 1.27 (t, J = 6.96 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 165.45, 146.36, 136.40, 136.31, 116.11, 60.10, 45.78, 38.93, 30.83, 29.31, 14.25; IR (neat) 1715.5, 1634.5, 1458.3 cm^{-1}; HRMS calcd for $C_{11}H_{16}O_2$: 180.1150; found, 180.1140; LRMS (EI$^+$): *m/z* 180 (100), 139 (100), 111 (42), 93 (19), 67 (61), 41 (21), 29 (52).

Ethyl 3-(3-Butenyl)-2-hydroxycyclopentanecarboxylate. Prepared from ethyl (1*R**, 2*S**/*R**)-2-oxo-3-(3-butenyl)cyclopentanecarboxylate according to the general procedure outlined for the preparation of ethyl 3-(2-propenyl)-1-cyclopentenecarboxylate to afford the desired alcohol in 89% yield after Kugelrohr distillation (ot 110-120 °C @ 0.1 mm Hg); ^{1}H NMR (500 MHz, $CDCl_3$): δ 5.78 (m, 1H), 5.02-4.92 (m, 2H), 4.28 (m, 0.4H), 4.19-4.11 (m, 1.6H), 3.99 (m, 1H), 2.77 (m, 1H), 2.11-1.84 (m, 5H), 1.84-1.54 (m, 4H), 1.26 (m, 1H), 1.24 (m, 3H); IR (neat) 3443.5, 1730.0, 1640.7 cm^{-1}; HRMS calcd for $C_{12}H_{20}O_3$: 212.1420; found 212.1412; LRMS (EI$^+$): *m/z* 212 (83), 194 (100), 171 (51), 140 (34), 125 (96), 111 (32), 97 (72), 81 (51), 67 (72), 55 (78), 41 (91).

Ethyl 3-(3-Butenyl)-1-cyclopentenecarboxylate (32b). Prepared from ethyl 3-(3-butenyl)-2-hydroxycyclopentanecarboxylate according to the general procedure outlined for the preparation of ethyl 3-(2-propenyl)-1-cyclopentenecarboxylate (**32a**) to afford the desired α, β-unsaturated ester (**32b**) in 64% yield (for two steps) after flash column chromatography with 2% EtOAc/hexanes and Kugelrohr distillation (ot 110-120 °C @ 10 mm Hg); ^{1}H NMR (500 MHz, $CDCl_3$): δ 6.68 (m, 1H), 5.78 (m, 1H), 5.03-4.93 (m, 2H), 4.16 (q, J = 7.15 Hz, 2H), 2.79 (m, 1H), 2.56 (m, 1H), 2.49 (m, 1H), 2.16-2.06 (m, 3H), 1.57-1.48 (m, 2H), 1.41 (m, 1H), 1.27 (t, J = 7.15 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 165.52, 146.86, 138.35, 136.04, 114.70, 60.09, 45.72, 34.05, 31.83, 30.80, 29.87, 14.27;

IR (neat) 1715.7, 1637.7, 1557.8 cm^{-1}; HRMS calcd for $C_{12}H_{18}O_2$: 194.1307; found 194.1307; LRMS (EI^+): *m/z* 194 (100), 165 (97), 152 (98), 121 (62), 93 (42), 79 (82), 67 (80), 55 (32), 41 (40).

Ethyl 3-(3-Iodopropyl)-1-cyclopentenecarboxylate (33a). Prepared from ethyl 3-(2-propenyl)-1-cyclopentenecarboxylate (**32a**) according to the following general procedure. Borane dimethylsulfide (0.22 mL, 2.2 mmol, 10.0 M in DMS) was added to a 0 °C cooled solution of cyclohexene (0.40 g, 4.84 mmol) in 5 mL of dry THF. The reaction mixture was stirred for 1 h at 0 °C and then for 2 h at rt. After this period of time, the reaction mixture was cooled to 0 °C and ethyl 3-(2-propenyl)-1-cyclopentenecarboxylate (0.36 g, 2.0 mmol) was added in 3 mL of dry THF. The resultant reaction mixture was stirred for 1 h at 0 °C and then for 2 h at ambient temperature. After this period of time, NaOAc (0.40 g, 4.84 mmol) and ICl (0.36 g, 2.2 mmol) were added successively as 1 M solutions in MeOH. The reaction mixture was stirred for 15 min and then quenched with saturated aqueous $NaHSO_3$. Then the reaction mixture was subjected to an aqueous workup and then subjected to flash column chromatography with 1% EtOAc/hexanes followed by Kugelrohr distillation (ot 100-110 °C @ 0.1 mm Hg) to afford the desired iodide (0.33 g, 1.46 mmol) in 73% yield; ^1H NMR (400 MHz, $CDCl_3$): δ 6.64 (m, 1H), 4.16 (q, J = 7.08 Hz, 2H), 3.17 (t, J = 6.84 Hz, 2H), 2.80 (m, 1H), 2.64-2.43 (m, 2H), 2.14 (m, 1H), 1.85 (m, 2H), 1.62-1.38 (m, 3H), 1.27 (t, J = 7.08 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 165.39, 146.15, 136.46, 60.18, 45.36, 35.71, 31.66, 30.83, 29.83, 14.28, 6.59; IR (neat) 1711.6, 1631.1, 1547.4, 1512.2 cm^{-1}; HRMS calcd for $C_{11}H_{17}IO_2$: 308.0273; found 308.0250; LRMS (EI^+): *m/z* 308 (13), 263 (31), 235 (100), 181 (21), 139 (42), 107 (79), 79 (42), 67 (99), 55 (18), 41 (38), 29 (80).

Ethyl 3-(4-Iodobutyl)-1-cyclopentenecarboxylate (33b). Prepared from ethyl 3-(3-butenyl)-1-cyclopentenecarboxylate, **32b**, according to the general procedure outlined for the preparation of ethyl 3-(3-iodopropyl)-1-cyclopentenecarboxylate, **33a**, to afford the desired iodide in 42% yield after flash column chromatography with 5% EtOAc/hexanes; ^1H NMR (400 MHz, $CDCl_3$): δ 6.66 (m, 1H), 4.16 (q, J = 6.59 Hz, 2H), 3.17 (t, J = 6.84 Hz, 2H), 2.75 (m, 1H), 2.64-2.41 (m, 2H), 2.19-2.08 (m, 1H), 1.81 (m, 2H), 1.57-1.31 (m, 5H), 1.27 (t, J = 6.59 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 165.49, 146.67, 136.15, 60.12, 46.06, 33.76, 33.44, 30.80, 29.90, 28.66, 14.28, 6.83; IR (neat)

1713.4, 1631.6, 1454.3 cm^{-1}; HRMS calcd for $C_{12}H_{19}IO_2$: 322.0430; found 322.0406; LRMS (EI^+): *m/z* 322 (29), 277 (28), 249 (78), 183 (52), 139 (63), 121 (86), 93 (43), 79 (50), 67 (100), 55 (83).

Tetrahydro-3-phenylselenyl-6-(2-propenyl)pyran-2(2H)-one. Prepared from tetrahydro-6-(2-propenyl)pyran-2(2H)-one according to the following general procedure. Tetrahydro-6-(2-propenyl)pyran-2(2H)-one (1.98 g, 14.1 mmol) in 10 mL of dry THF was added dropwise *via* cannula to a 1 M solution of LDA (15.5 mmol) at -78 °C. The resultant reaction mixture was stirred for 45 min at -78 °C and then phenylselenyl chloride (2.97 g, 15.5 mmol) was added *via* cannula in 5 mL of dry THF. The reaction mixture was stirred for 15 min at reduced temperature and then quenched with saturated aqueous NH_4Cl to afford a 1.4:1 mixture of diastereomers (2.50 g, 8.46 mmol) in 60% yield after flash column chromatography with 15% EtOAc/hexanes; (Higher R_f) ^1H NMR (400 MHz, $CDCl_3$): δ 7.63 (m, 2H), 7.32 (m, 3H), 5.78 (m, 1H), 5.12 (m, 2H), 4.42 (m, 1H), 3.93 (m, 1H), 2.46-2.92 (m, 3H), 2.08-1.87 (m, 2H), 1.56 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.42, 135.66 (2), 132.32, 129.26 (2), 128.85, 127.59, 118.65, 78.94, 39.85, 37.77, 26.97, 26.44; IR (neat) 3072.5, 1714.3, 1643.6, 1574.3 cm^{-1}; HRMS calcd for $C_{14}H_{16}SeO_2$: 296.0316; found 296.0308; LRMS (EI^+): *m/z* 296 (100), 227 (21), 184 (50), 157 (64), 139 (29), 104 (23), 93 (31), 77 (54), 67(42), 51(34), 41 (99), 27 (3); (Lower R_f) ^1H NMR (400 MHz, $CDCl_3$): δ 7.66 (m, 2H), 7.36-7.28 (m, 3H), 5.78 (m, 1H), 5.13 (m, 2H), 4.35 (m, 1H), 4.01 (m, 1H), 2.44 (m, 1H), 2.37 (m, 1H), 2.24 (m, 1H), 2.12 (m, 1H), 1.81 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.56, 135.55 (2), 132.29, 129.24 (2), 127.98, 128.72, 118.66, 80.29, 39.94, 39.51, 26.38, 25.13; IR (neat) 3072.6, 1729.9, 1643.3, 1574.3 cm^{-1}; HRMS calcd for $C_{14}H_{16}SeO_2$: 296.0316; found 296.0314; LRMS (EI^+): *m/z* 296 (100), 227 (23), 184 (30), 157 (40), 139 (30), 104 (20), 93 (38), 77 (43), 64.48, 55 (28), 41 (99), 27 (38).

Tetrahydro-6-(2-propenyl)pyridin-2-one. Prepared according to the following general procedure. A 50 mL round bottom flask equipped with a reflux condenser and addition funnel was charged with *N*-hydroxylamine-*O*-sulfonic acid (1.70 g, 15.0 mmol) and 9 mL of 95-97% formic acid. The reaction mixture was cooled to 0 °C in an ice bath, and then 2-(2-propenyl)cyclopentanone (1.24 g, 10.0 mmol) was added with stirring over 1 min. The ice bath was removed and the reaction mixture was heated at reflux for 3 h. After this period, the reaction mixture was cooled to rt and quenched by pouring

into ice/H_2O. The reaction mixture was subjected to an aqueous workup (extracting with $CHCl_3$) after neutralization with 10% NaOH (to pH 7). Flash column chromatography with EtOAc afforded the desired lactam (0.56 g, 4.0 mmol) in 40% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 5.80 (m, 1H), 5.72 (m, 1H), 5.13 (m, 2H), 3.37 (m, 1H), 2.37-2.20 (m, 3H), 2.09 (m, 1H), 1.89 (m, 2H), 1.67 (m, 1H), 1.34 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 172.12, 133.28, 119.15, 52.04, 4.28, 31.25, 28.50, 19.82; IR ($CHCl_3$) 3392.0, 3082.6, 1641.4 cm^{-1}; HRMS calcd for $C_8H_{13}NO$: 139.0997; found 139.1001; LRMS (EI$^+$): *m/z* 140 (100), 139 (42), 124 (27), 110 (21), 98 (81), 70 (27), 55 (100), 41 (33), 27 (22).

***N*-Methyl Tetrahydro-6-(2-propenyl)pyridin-2-one (36a).** Prepared from tetrahydro-6-(2-propenyl)pyridin-2-one according to the following general procedure. Tetrahydro-6-(2-propenyl)pyridin-2-one (0.53 g, 3.8 mmol) in 10 mL of dry THF was added dropwise *via* cannula to a stirred slurry of NaH (0.17 g, 4.2 mmol, 60% dispersion in mineral oil) cooled to 0 °C in an ice bath. The reaction mixture was then warmed to rt and stirred for 1 h before cooling to 0 °C and adding MeI (1.08 g, 7.6 mmol) neat. The reaction mixture was then warmed to rt and stirred for 12 h. After this period of time, the reaction mixture was quenched with saturated aqueous NH_4Cl and subjected to an aqueous workup. Kugelrohr distillation of the crude reaction mixture afforded the desired *N*-methyl lactam (0.58 g, 3.8 mmol) in 100% yield; ^{1}H NMR (500 MHz, $CDCl_3$): δ 5.67 (m, 1H), 5.11 (m, 2H), 3.33 (m, 1H), 2.93 (s, 3H), 2.43 (m, 1H), 2.34 (m, 2H), 2.24 (m, 1H), 1.85-1.66 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 170.38, 133.84, 118.26, 58.38, 37.14, 33.36, 32.00, 26.24, 17.49; IR (neat) 3075.4, 1643.2 cm^{-1}; HRMS calcd for $C_9H_{15}NO$: 153.1154; found 153.1150; LRMS (EI$^+$): *m/z* 154 (39), 153 (100), 152 (47), 138 (28), 124 (48), 112.98, 84 (33), 55 (91), 4253, 28 (23).

***N*-Methyl (*cis/trans*)-Tetrahydro-3-phenylselenyl-6-(2-propenyl)-pyridin-2-one.** Prepared from *N*-methyl tetrahydro-6-(2-propenyl)pyridin-2-one according to the general procedure outlined for the preparation of tetrahydro-3-phenylselenyl-6-(2-propenyl)pyran-2(2H)-one to afford the desired lactam in 62% yield as a *ca.* 1:1 mixture of cis and trans diastereomers after flash column chromatography with 30% EtOAc/hexanes; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.36-7.26, (m, 3H), 7.71 (m, 1H), 7.66, (m, 1H), 5.63 (m, 0.5H), 5.40 (m, 0.5H), 5.08 (m, 1H), 4.91 (m, 0.5H), 4.77 (m,

0.5H), 4.01 (m, 0.5H), 3.34 (m, 0.5H), 3.11 (m, 0.5H), 2.93 (s, 1.5H), 2.88 (s, 1.5H), 2.43 (m, 0.5H), 2.23-2.09 (m, 2H), 2.02-1.77 (m, 2H), 1.68 (m, 1H), 1.39 (m, 1H); IR (neat) 3070.3, 1630.4, 1578.0 cm^{-1}; HRMS calcd for $C_{15}H_{19}NOSe$: 309.0632; found 309.0648; LRMS (EI^+): *m/z* 309 (19), 268 (58), 228 (31), 190 (11), 152 (40), 110 (100), 82 (33), 67 (32), 55 (33), 41 (49).

Dihydro *N*-Methyl-6-(2-propenyl)pyridin-2-one (37a). Prepared from tetrahydro *N*-methyl-3-phenylselenyl-6-(2-propenyl)pyridin-2-one according to the general procedure outlined for the preparation of dihydro-6-(2-propenyl)pyran-2-one to afford the desired lactam in 60% yield after flash column chromatography with 80% EtOAc/hexanes; ^1H NMR (500 MHz, $CDCl_3$): δ 6.36 (m, 1H), 5.91 (dd, J = 2.98, 9.73 Hz, 1H), 5.69 (m, 1H), 5.08 (m, 2H), 3.43 (m, 1H), 2.99 (s, 3H), 2.59 (ddt, J = 18.16, 7.24, 2.78 Hz, 1H), 2.40-2.26 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 163.78, 136.76, 134.24, 125.13, 118.44, 57.48, 35.70, 33.48, 26.82; IR (neat) 3076.1, 1666.2, 1613.6 cm^{-1}; HRMS calcd for $C_9H_{13}NO$: 151.0997; found 151.0986; LRMS (EI^+): *m/z* 151 (100), 134 (12), 128 (29), 110 (99), 42 (42).

Dihydro-6-(2-propenyl)pyran-2-one (37b). Prepared from tetrahydro-3-phenylselenyl-6-(2-propenyl)pyran-2(2H)-one according to the following general procedure. A 30% solution of H_2O_2 (3.0 mL, 26.2 mmol, 0.89 g) was added dropwise *via* addition funnel to a 0 °C cooled solution of tetrahydro-3-phenylselenyl-6-(2-propenyl)pyran-2(2H)-one (2.21 g, 7.51 mmol) in 30 mL of dry CH_2Cl_2. The reaction mixture was stirred at 0 °C for 30 min and then warmed slowly to rt and quenched by the careful addition of saturated aqueous $NaHCO_3$ followed by saturated aqueous $NaHSO_3$. Flash column chromatography with 15% EtOAc/hexanes followed by Kugelrohr distillation (ot 90-100 °C @ 10 mm Hg) afforded the desired α, β-unsaturated lactone (0.54 g, 3.90 mmol) in 52% yield; ^1H NMR (400 MHz, $CDCl_3$): δ 6.85 (dt, J = 4.28, 9.91 Hz, 1H), 6.00 (dt, J = 1.87, 9.91 Hz, 1H), 5.81 (m, 1H), 5.18-5.12 (m, 2H), 4.47 (m, 1H), 2.57-2.41 (m, 2H), 2.34-2.31 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 164.20, 144.92, 132.22, 121.32, 118.79, 77.08, 39.01, 28.63; IR (neat) 1731.6, 1643.5, 1422.3, 1386.0 cm^{-1}; HRMS calcd for $C_8H_{11}O_2$ ($M+H^+$): 139.0759; found, 139.0750; LRMS (EI^+): *m/z* 139 (19), 110 (100), 97 (96), 69 (37), 41 (51).

Dihydro *N*-Methyl-6-(3-iodopropyl)pyridin-2-one (38a). Prepared from dihydro *N*-methyl-6-(2-propenyl)pyridin-2-one according to the general procedure outlined for the preparation of ethyl 3-(3-iodopropyl)-1-cyclopentenecarboxylate to afford the desired iodide in 71% yield after flash column chromatography with 80% EtOAc/hexanes; ^{1}H NMR (500 MHz, $CDCl_3$): δ 6.37 (m, 1H), 5.90 (dd, J = 2.98, 9.73 Hz, 1H), 3.40 (m, 1H), 3.18-3.10 (m, 2H), 2.98 (s, 3H), 2.67 (ddt, J = 2.78, 12.61, 25.41 Hz, 1H), 2.20 (ddd, J = 1.49, 6.15, 18.06 Hz, 1H), 1.84-1.69 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 163.69, 136.59, 125.20, 57.01, 33.65, 32.48, 30.14, 27.46, 5.60; HRMS calcd for $C_9H_{14}ION$: 279.0120; found 279.0106; LRMS (EI$^+$): *m/z* 279 (31), 251 (23), 212 (100), 127 (51), 110 (98), 42 (41).

Dihydro-6-(3-iodopropyl)pyran-2-one (38b). Prepared from dihydro-6-(2-propenyl)pyran-2-one according to the general procedure outlined for the preparation of ethyl 3-(3-iodopropyl)-1-cyclopentenecarboxylate to afford the desired iodide in 73% yield after flash column chromatography with 15% EtOAc/hexanes; ^{1}H NMR (500 MHz, $CDCl_3$): δ 6.86 (dt, J = 4.37, 9.53 Hz, 1H), 6.00 (m, 1H), 4.43 (m, 1H), 3.21 (m, 2H), 2.34 (m, 2H), 2.09 (m, 1H), 1.93 (m, 1H), 1.82 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 164.04, 144.80, 121.38, 35.57, 29.32, 28.55, 6.01; IR (neat) 1725.9, 1641.2 cm^{-1}; HRMS calcd for $C_8H_{11}IO_2$: 265.9804; found 265.9810; LRMS (EI$^+$): *m/z* 266 (9), 197 (32), 169 (39), 139 (100), 97 (78), 71 (90), 43 (92).

5-Hexyn-1-ol, *tert*-Butyldimethylsilyl Ether (40a). Prepared from 5-hexyn-1-ol according to the following general procedure. A solution of 5-hexyn-1-ol (9.82 g, 100 mmol) in 100 mL of CH_2Cl_2, cooled to 0 °C in an ice bath, was treated successively with Et_3N (11.1 g, 110 mmol) and TBDMSCl (16.6 g, 110 mmol). After the addition of Et_3N and TBDMSCl were complete, the reaction mixture was warmed to rt and stirred at ambient temperature overnight. After this period of stirring, the reaction mixture was diluted with pentane and filtered to remove the precipitated salts. Flash chromatography through a short plug of silica gel followed by Kugelrohr distillation afforded the desired TBS ether **40a**, (21.2g, 100 mmol), in quantitative yield: ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.61 (t, J = 5.86 Hz, 2H), 2.18 (m, 2H), 1.92 (t, J = 2.69 Hz, 1H), 1.58 (m, 4H), 0.87 (s, 9H), 0.03 (s, 6H).

Methyl 7-(*tert*-Butyldimethylsilyloxy)-2-heptynoate (41a). Prepared from **40a** according to the following general procedure. The alkyne (**40a** , 6.36 g, 30.0 mmol) in 40 mL of dry THF was cooled to 0 °C in an ice bath and treated with EtMgBr (16.5 mL of a 2.0 M solution in Et_2O, 33.0 mmol). The reaction mixture was maintained at 0 °C for 2 h and then methyl chloroformate (3.12 g, 33.0 mmol) was added rapidly. The resultant reaction mixture was stirred at 0 °C for 30 min and then warmed to rt. The reaction mixture was quenched with saturated aqueous $NaHCO_3$ upon reaching rt. An aqueous workup followed by flash column chromatography with 3% EtOAc/hexanes afforded **41a** (6.11 g, 22.8 mmol) in 76% yield: ^1H NMR (400 MHz, $CDCl_3$): δ 3.74 (s, 3H), 3.61 (t, J = 5.89 Hz, 2H), 2.35 (t, J = 6.96 Hz, 2H), 1.62 (m, 4H), 0.87 (s, 9H), 0.02 (s, 6H).

***N,N*-Dimethyl 7-(*tert*-Butyldimethylsilyloxy)-2-heptynamide (41b).** Prepared from **40a** and dimethylcarbamoyl chloride according to the general procedure outlined for the preparation of **41a** to afford **41b** in 44% yield after flash chromatography with 35% EtOAc/hexanes; ^1H NMR (300 MHz, $CDCl_3$): δ 3.60 (m, 2H), 3.17 (s, 3H), 2.94 (s, 3H), 2.37 (m, 2H), 1.61 (m, 4H), 0.86 (s, 9H), 0.02 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 154.75, 92.89, 74.11, 62.37, 38.31, 33.98, 31.86, 25.89 (3), 24.40, 18.73, 18.27, -5.37 (2).

Methyl 7-Hydroxy-2-heptynoate (42a). Prepared from **41a** according to the following general procedure. The TBS ether, **41a** (4.05 g, 15.0 mmol) in 20 mL of MeOH was stirred overnight with Amberlyst-15. After this period of stirring, the reaction mixture was filtered through a short plug of Celite to remove the Amberlyst-15, concentrated *in vacuo*, and then subjected to flash column chromatography with 30% EtOAc/hexanes to afford **42a** (2.21 g, 14.1 mmol) in 94% yield: ^1H NMR (300 MHz, $CDCl_3$): δ 3.74 (s, 3H), 3.66 (m, 2H), 2.37 (m, 2H), 1.67 (m, 4H), 1.30 (m, 1H); IR (neat) 3390.3, 2236.3, 1713.8 cm^{-1}.

***N,N*-Dimethyl 7-Hydroxy-2-heptynamide (42b).** Prepared from **41b** according to the general procedure outlined for the preparation of **42a** to afford **42b** in 85% yield after flash column chromatography with EtOAc; ^1H NMR (400 MHz, $CDCl_3$): δ 6.67 (m, 2H), 3.17 (s, 3H), 2.94 (s, 3H), 2.39 (m, 2H), 1.67 (m, 4H); IR (neat) 3370.0, 2358.8, 1736.5, 1631.9 cm^{-1}.

Methyl 7-Iodo-2-heptynoate (43a). Prepared from **42a** according to the following general procedure outlined for the preparation of **9a** to afford **43a** in 66% yield after flash column chromatography with 2% EtOAc/hexanes; ^{1}H NMR (300 MHz, $CDCl_3$): δ 3.74 (s, 3H), 3.18 (t, J = 6.84 Hz, 2H), 2.36 (t, J = 7.08 Hz, 2H), 1.92 (m, 2H), 1.68 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 154.05, 88.53, 73.34, 52.61, 32.09, 28.18, 17.63, 5.44; LRMS (EI$^+$): *m/z* 266 (99), 251 (54), 235 (100), 155 (16), 139 (21), 127 (19), 107 (37), 79 (100), 66 (31), 59 (54), 53 (38), 41 (51), 27 (28).

***N,N*-Dimethyl 7-Iodo-2-heptynamide (43b).** Prepared from **42b** according to the general procedure outlined for the preparation of **43a** to afford **43b** in 64% yield after flash chromatography with 50% EtOAc/hexanes; ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.17 (s, 3H), 3.16 (m, 2H), 2.94 (s, 3H), 2.38 (t, J = 6.96 Hz, 2H), 1.93 (m, 2H), 1.72-1.63 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 154.50, 91.77, 74.51, 38.31, 33.97, 32.21, 28.44, 17.87, 5.65; LRMS (EI$^+$): *m/z* 280 (14), 235 (26), 152 (54), 109 (100), 95 (88), 79 (83), 66 (58), 42 (41).

Ethyl 8-Iodo-2-octynoate (43c). Ethyl propiolate (0.98 g, 10.0 mmol) in 10 mL of dry THF was added dropwise *via* cannula to a stirred solution of 11.0 mmol LDA (1 M in THF) at -78 °C in a Dry Ice/acetone bath. After the addition of the substrate was complete, the reaction mixture was stirred for 15 min at -78 °C and then 1,5-diiodopentane in 10 mL of HMPA was added rapidly dropwise *via* cannula. The reaction mixture was quenched with saturated aqueous NH_4Cl after stirring for 1 h at -78 °C. An aqueous workup followed by flash column chromatography with hexanes afforded the desired halide (1.79 g, 6.1 mmol) in 61% yield after flash column chromatography with hexanes; ^{1}H NMR (500 MHz, $CDCl_3$): δ 4.19 (q, J = 7.15 Hz, 2H), 3.16 (t, J = 6.95 Hz, 2H), 2.33 (t, J = 6.85 Hz, 2H), 1.82 (m, 2H), 1.59 (m, 2H), 1.28 (t, J = 7.15 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 153.71, 88.61, 73.43, 61.78, 32.80, 29.64, 26.43, 18.50, 14.01, 6.20; IR (neat) 2234.4, 1713.2 cm^{-1}.

(1*E*/1*Z*)-1-Cyano-6-iodo-1-hexene (47a).[4] Prepared from 6-bromo-4-methylhexanal according to the general procedure outlined for the preparation of (1*E*/1*Z*)-1-cyano-5-methyl-7-iodo-1-heptene to afford the desired α, β-unsaturated nitrile in 61% yield for the two steps after flash column chromatography with 2% EtOAc/hexanes; ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.68 (dt, J = 6.96, 16.33 Hz, 0.5H), 6.45 (dt, J = 7.76, 10.65 Hz, 0.5H), 5.36-5.31 (m, 1H), 3.17 (m, 2H), 2.44 (dq, J = 1.07, 7.50

Hz, 1H), 2.24 (dq, J = 1.87, 7.50 Hz, 1H), 1.84 (m, 2H), 1.58 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 154.86, 154.01, 117.26, 115.79, 100.34, 100.22, 32.46, 32.42, 32.10, 30.67, 28.93, 28.38, 5.88, 5.69; IR (neat) 2358.5, 1633.2 cm^{-1}; HRMS calcd for $C_7H_{10}INO$: 234.9858; found 234.9839; LRMS (EI^+): *m/z* 235 (100), 155 (64), 141 (36), 127 (70), 108 (98), 81 (51), 66 (31), 53 (28), 41 (42).

(1*E*/1*Z*)-1-Cyano-5-methyl-7-iodo-1-heptene (47b). Prepared from 6-bromo-4-methylhexanal according to the following general procedure. *n*-Butyllithium (3.4 mL of a 1.6 M solution in hexanes, 5.5 mmol) was added dropwise *via* syringe to a stirred solution of diethyl cyanomethylphosphonate (0.97 g, 5.5 mmol) in 10 mL of dry THF at 0 °C. The reaction mixture was stirred for 10 min at reduced temperature and then 6-bromo-4-methylhexanal (0.97 g, 5.0 mmol) in 5 mL of dry THF was added dropwise via cannula. The reaction mixture was then warmed to rt, quenched with saturated aqueous NH_4Cl, and then subjected to an aqueous workup. The crude reaction mixture was concentrated *in vacuo*, taken up in 20 mL of acetone, and heated at reflux for 18 h with NaI (3.75 g, 25.0 mmol). Then, reaction mixture was cooled to rt and subjected to an aqueous workup. Flash column chromatography with 2% EtOAc/hexanes afford the desired α, β-unsaturated nitrile (0.82 g, 3.12 mmol) as a 1:1 mixture of E and Z olefin isomers in 62% yield (2 steps); ^1H NMR (500 MHz, $CDCl_3$): δ 6.70 (dt, J = 16.28, 6.95 Hz, 0.5H), 6.45 (dt, J = 10.92, 7.64 Hz, 0.5H), 5.33 (dt, J = 16.28, 1.59 Hz, 0.5H), 5.31 (dt, J = 10.92, 1.29 Hz, 0.5H), 3.23 (m, 1H), 3.14 (m, 1H), 2.42 (m, 1H), 2.23 (m, 1H), 1.85 (m, 1H), 1.68-1.58 (m, 2H), 1.45 (m, 1H), 1.28 (m, 1H), 0.99 (d, J = 6.45 Hz, 1.5H), 0.88 (d, J = 6.45 Hz, 1.5H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 155.57, 154.70, 117.38, 115.87, 99.91, 99.73, 40.30, 40.27, 34.47, 33.95, 33.26, 33.18, 30.69, 29.20, 18.31, 18.26, 4.53, 4.41; IR (neat) 2221.0 cm^{-1}; HRMS calcd for $C_9H_{14}BrN$: 263.0171; found 263.0125; LRMS (EI^+): *m/z* 263 (100), 164 (84), 136 (98), 109 (19), 94 (29), 80 (32), 67 (32), 55 (31), 41 (74), 27 (41).

Dihydro-5-(iodomethyl)furan-2(3H)-one (50a). Prepared according to the following general procedure. 4-Pentenoic acid, **49a**, (0.50 g, 5.0 mmol), iodine (2.53 g, 10.0 mmol), and $NaHCO_3$ (0.84 g, 10.0 mmol) were stirred together in 10 mL of CH_3CN for 18 h. After this period of stirring, the reaction mixture was quenched by the **careful** addition of sodium bisulfite and then subjected to an aqueous workup. Flash column chromatography with 30% EtOAc/hexanes afforded the desired

halolactone (1.12 g, 4.96 mmol) in 99% yield; ^{1}H NMR (400 MHz, $CDCl_3$): δ 4.53 (dq, J = 4.28, 7.23 Hz, 1H), 3.40 (dd, J = 4.28, 10.44 Hz, 1H), 3.28 (dd, J = 7.23, 10.44 Hz, 1H), 2.67-2.44 (m, 3H), 2.02-1.95 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 176.13, 78.36, 28.78, 28.07, 7.38; IR (neat) 1773.9 cm^{-1}; HRMS calcd for $C_5H_7IO_2$: 225.9491; found 225.9477; LRMS (EI$^+$): *m/z* 226 (100), 169 (9), 141 (12), 127 (16), 99 (98), 85 (83), 71 (12), 55 (29), 43 (32), 29 (48).

Tetrahydro-6-(iodomethyl)pyran-2-one (50b). Prepared from 5-hexenoic acid according to the general procedure outlined for the preparation of **50a** to afford the desired halolactone in 85% yield after flash column chromatography with 30% EtOAc/hexanes; ^{1}H NMR (400 MHz, $CDCl_3$): δ 4.27 (m, 1H), 3.32 (dq, J = 4.55, 10.44 Hz, 2H), 2.63-2.55 (m, 1H), 2.49-2.39 (m, 1H), 2.20-2.14 (m, 1H), 2.01-1.83 (m, 2H), 1.63 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.47, 78.73, 29.19, 27.96, 18.11, 7.32.

Tetrahydro-5-(iodomethyl)furan-2-ol (51a). Prepared from dihydro-5-(iodomethyl)furan-2(2H)-one by reduction with DIBAL-H according to the general procedure outlined for the preparation of **7a** to afford the desired lactol in 63% yield after Kugelrohr distillation; ^{1}H NMR (400 MHz, $CDCl_3$): δ 5.62 (m, 0.5H), 5.59 (m, 0.5H), 4.29 (m, 0.5H), 4.20 (m, 0.5H), 3.36 (dd, J = 9.83, 5.66 Hz, 0.5H), 3.25 (dd, J = 7.15, 9.83 Hz, 0.5H), 3.22 (d, J = 5.56 Hz, 1H), 2.63 (m, 0.5H), 2.58 (m, 0.5H), 2.26-1.85 (m, 3H), 1.66 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 99.18, 98.97, 80.22, 77.49, 34.02, 32.72, 29.75, 29.35, 11.22, 10.05; IR (neat) 3419.6, 1769.2, 1455.6 cm^{-1}; HRMS calcd for $C_5H_9IO_2$: 227.9647; found 227.9646; LRMS (EI$^+$): *m/z* 228 (8), 211 (7), 171 (11), 127 (9), 101 (40), 87 (100), 69 (29), 55 (76), 43 (56), 29 (38).

Ethyl (2*E*)-7-Iodo-6-hydroxy-2-heptenoate (52a). Prepared from dihydro 5-(iodomethyl)furan-2(2H)-ol and (carbethoxymethylidene)triphenylphosphorane according to the general procedure outlined for the preparation of **3b** to afford the desired α, β-unsaturated ester in 65% yield after flash column chromatography with 30% EtOAc/hexanes; ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.93 (dt, J = 7.08, 15.63 Hz, 1H), 5.83 (dt, J = 1.71, 15.63 Hz, 1H), 4.16 (q, J = 7.08 Hz, 2H), 4.13 (m, 1H), 3.53 (m, 1H), 3.35 (dd, J = 3.42, 10.01 Hz, 1H), 3.20 (dd, J = 6.84, 10.25 Hz, 1H), 2.34 (m, 2H), 2.01 (m, 1H), 1.69 (m, 1H), 1.26 (t, J = 7.08 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.51, 147.76,

121.99, 70.05, 60.28, 34.65, 28.28, 15.87, 14.22; IR (neat) 3447.7, 1700.1, 1653.9 cm^{-1}; HRMS calcd for $C_9H_{14}IO_2$ ($M-H^+$): 296.9988; found 296.9977; LRMS (EI^+): *m/z* 297 (9), 252 (100), 205 (63), 125 (23), 99 (24), 81 (19), 55 (23), 43 (100), 29 (61).

Ethyl (2*E*)-8-Iodo-7-hydroxy-2-octenoate (52b). Prepared from **50b** according to the general procedure outlined for the preparation of **51a** and **52a** to afford the desired hydroxy olefin in 76% yield after flash column chromatography with 30% EtOAc/hexanes; ^1H NMR (400 MHz, $CDCl_3$): δ 6.92 (dt, J = 15.53, 6.96 Hz, 1H), 5.81 (dt, J = 15.53, 1.61 Hz, 1H), 4.14 (q, J = 7.23 Hz, 2H), 3.52 (m, 1H), 3.35 (dd, J = 3.75, 10.17 Hz, 1H), 3.20 (dd, J = 6.97, 10.17 Hz, 1H), 2.22 (m, 2H), 1.98 (m, 1H), 1.66-1.51 (m, 4H), 1.26 (t, J = 7.23 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.59, 148.34, 121.78, 70.67, 60.21, 35.82, 31.80, 24.12, 16.28, 14.24; IR (neat) 3439.1, 1709.5, 1651.7 cm^{-1}; HRMS calcd for $C_{10}H_{17}IO_3$ $(M+H)^+$: 313.0300; found 313.0288; LRMS (EI^+): *m/z* 269 (21), 171 (100), 93 (69), 81 (97), 67 (63), 55 (80), 43 (98), 29 (98).

Ethyl (2*E*)-6-(*tert*-Butyldimethylsilyloxy)-7-iodo-2-heptenoate (53a). Prepared from ethyl (2*E*)-7-iodo-6-hydroxy-2-heptenoate according to the following general procedure. Ethyl (2*E*)-6-acetoxy-7-iodo-2-heptenoate (1.07 g, 3.60 mmol), Et_3N (0.44 g, 4.32 mmol), and TBSCl (0.65 g, 4.32 mmol) in 5 mL of dry CH_2Cl_2 with catalytic DMAP were stirred together for 18 h at ambient temperature. After this period of time, the reaction mixture was quenched with 1N HCl and subjected to an aqueous workup. Flash column chromatography with 2% EtOAc/hexanes afforded the desired TBS ether in 100% yield; ^1H NMR (500 MHz, $CDCl_3$): δ 6.95 (dt, J = 6.85, 15.58 Hz, 1H), 5.82 (d, J = 15.58 Hz, 1H), 4.16 (q, J = 7.15 Hz, 2H), 3.57 (m, 1H), 3.15 (m, 2H), 2.22 (m, 2H), 1.79-1.69 (m, 2H), 1.27 (t, J = 7.15 Hz, 3H), 0.87 (s, 9H), 0.07 (s, 3H), 0.05 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 166.53, 148.28, 121.66, 70.63, 60.21, 35.06, 27.52, 25.76 (3), 18.02, 14.27, 12.82, -4.34, -4.64; IR (neat) 1722.2, 1654.8, 1471.9 cm^{-1}; HRMS calcd for $C_{15}H_{28}SiIO_3$ ($M-H^+$): 411.0853; found 411.0852; LRMS (EI^+): *m/z* 367 (33), 355 (100), 309 (81), 263 (46), 235 (49), 185 (82), 153 (20), 107 (92), 93 (47), 75 (98), 59 (70), 43 (72), 29 (73).

Ethyl (2*E*)-6-Acetoxy-7-iodo-2-heptenoate (53b). Prepared from ethyl (2*E*)-7-iodo-6-hydroxy-2-heptenoate according to the following general procedure. Ethyl (2*E*)-6-acetoxy-7-iodo-2-

heptenoate (0.38 g, 1.29 mmol), Ac_2O (0.16 g, 1.54 mmol), and 1 mL of pyridine with catalytic DMAP were stirred together at ambient temperature for 18 h in 2 mL of dry CH_2Cl_2. After this period of time, the reaction mixture was quenched with 10% HCl and subjected to an aqueous workup. Flash column chromatography with 20% EtOAc/hexanes afforded the desired acetate (0.35 g, 1.03 mmol) in 78% yield; ^1H NMR (500 MHz, $CDCl_3$): δ 6.90 (dt, J = 15.68, 6.75 Hz, 1H), 5.82 (d, J = 15.68 Hz, 1H), 4.69 (m, 1H), 4.16 (q, J = 7.15 Hz, 2H), 3.30 (dd, J = 5.06, 10.62 Hz, 1H), 3.24 (dd, J = 5.06, 10.62, 1H), 2.23 (m, 2H), 2.07 (s, 3H), 1.83 (m, 2H), 1.26 (t, J = 7.15 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 170.17, 166.26, 146.93, 122.13, 71.51, 60.24, 32.45, 27.78, 20.96, 14.21, 7.46; IR (neat) 1714.8, 1655.6, 1445.4 cm^{-1}; HRMS calcd for $C_{11}H_{18}IO_4(M+H^+)$: 341.0250; found 341.0255; LRMS (EI^+): *m/z* 298 (11), 280 (9), 253 (100), 235 (19), 213 (96), 153 (98), 125 (98), 107 (23), 79 (80), 43 (43).

Ethyl (2*E*)-7-(*tert*-Butyldimethylsilyloxy)-8-iodo-2-octenoate (53c). Prepared from **52b** according to the general procedure outlined for the preparation of **53a** to afford the desired TBS ether in 94% yield after flash column chromatography with 4% EtOAc/hexanes: ^1H NMR (500 MHz, $CDCl_3$): δ 6.93 (dt, J = 6.65, 15.58 Hz, 1H), 5.81 (dd, J = 0.79, 15.58 Hz, 1H), 4.17 (q, J = 7.05 Hz, 2H), 3.55 (m, 1H), 3.16 (m, 2H), 2.20 (m, 2H), 1.63 (m, 1H), 1.56 (m, 2H), 1.42 (m, 1H), 1.28 (t, J = 7.05 Hz, 3H), 0.88 (s, 9H), 0.08 (s, 3H), 0.05 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 166.62, 148.58, 121.64, 71.10, 60.17, 36.22, 32.02, 25.80 (2), 23.35, 18.03, 14.27, 13.22, -4.37, -4.60; IR (neat) 1721.9, 1652.7 cm^{-1}.

(*E*/*Z*)-6-Iodo-1-nitrohexene. Prepared according to the following general procedure.[5] A cold solution of 10 M NaOH, (0.21 mL, mmol) was added to a 0 °C cooled solution of CH_3NO_2 (0.12 g, 2.0 mmol) and 5-bromopentanal (0.3 g, 2.0 mmol) in 1 mL of dry methanol. The reaction was stirred at 0 °C for 15 min after which time TLC analysis revealed the complete consumption of the starting aldehyde. Then, the reaction was quenched with 2 mL of a 3% aqueous HCl solution and stirred at 0 °C for 20 min and then subjected to an aqueous workup. The crude product was then taken up in acetone and heated at reflux for 18 h with NaI (3.5 g, 23.0 mmol). After this period of stirring, the reaction mixture was cooled to rt and subjected to an aqueous workup. Flash column chromatography with 8% EtOAc/hexanes afforded the desired α,β-unsaturated nitro compound (0.13 g, 0.50 mmol) in 25% yield; ^1H NMR (400

MHz, $CDCl_3$): δ 7.29-7.20 (m, 1.5H), 7.07-6.95 (m, 0.5H), 3.18 (m, 2H), 2.30 (m, 2H), 1.85 (m, 2H), 1.65 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 141.58, 139.89, 32.48, 28.51, 27.31, 5.48; IR (neat) 1518.5, 1351.6 cm^{-1}.

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INDEX	FREQUENCY	PPM	HEIGHT
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7	5285.687	42.006	0.7
8	5010.976	39.823	0.9
9	4073.780	32.375	0.8
10	3960.716	31.476	0.9
11	3433.377	27.286	0.7
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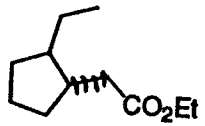

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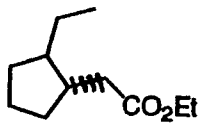

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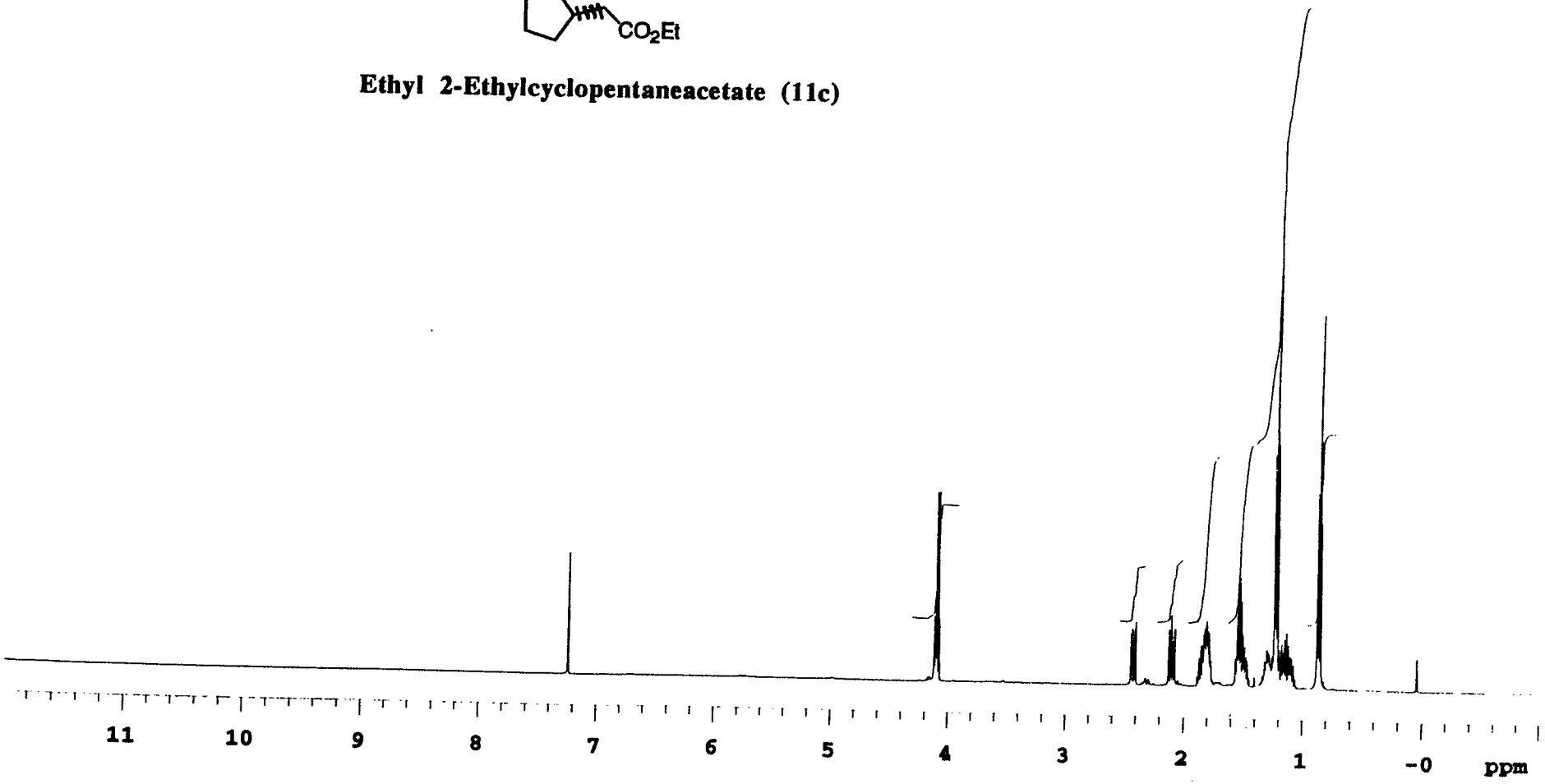

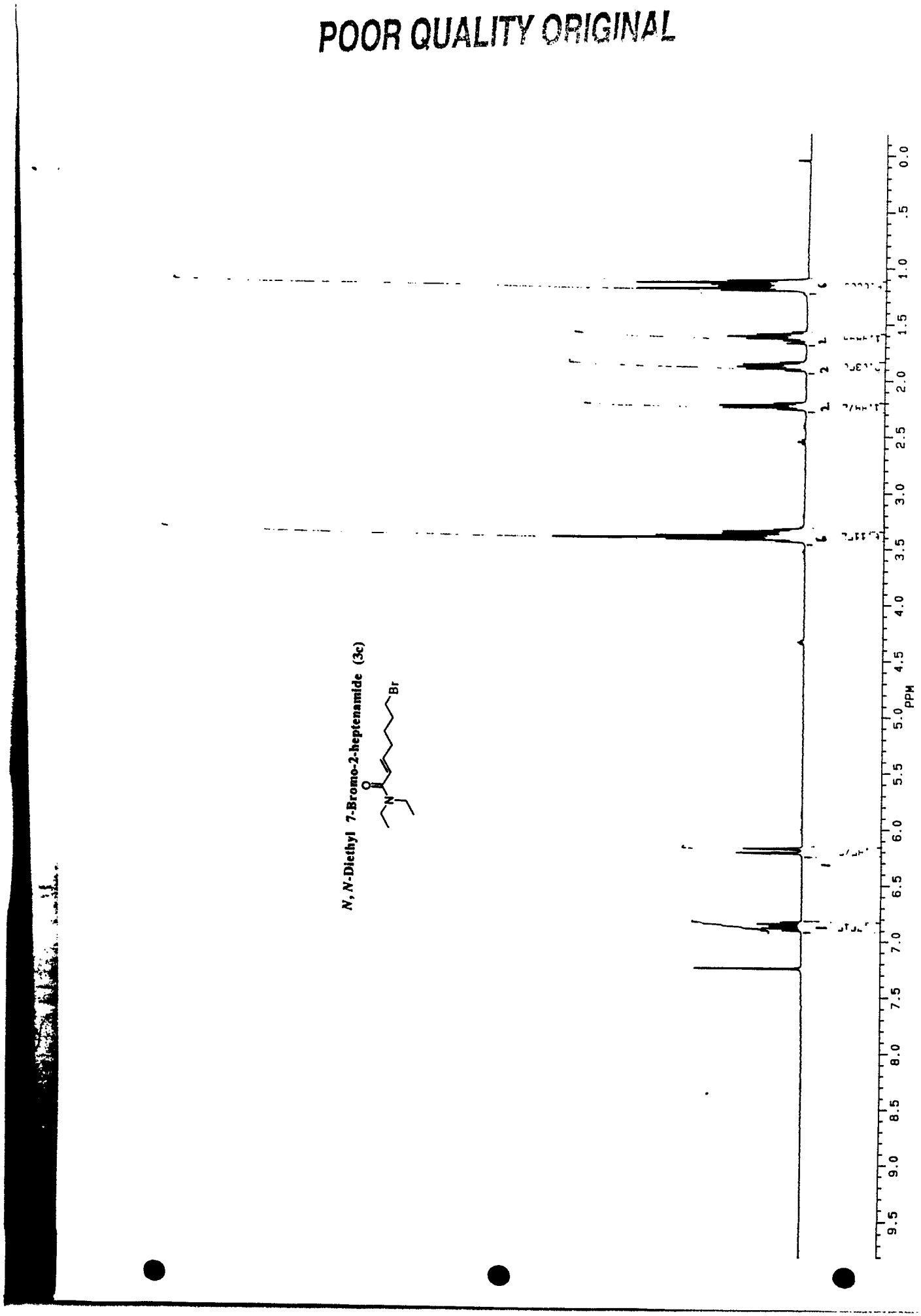

Ethyl 8-Bromo-6-methyl-2-octenoate (3d)