A Domino Copper-Catalyzed C-O Coupling-Claisen Rearrangement

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Supporting Information

General Considerations: All reactions were carried out in test tubes (10 mL) sealed with screw caps. Copper (I) iodide was purchased from Fisher Scientific and Strem. Toluene and *o*-xylene were purchased from Aldrich. Cesium carbonate was purchased from different sources (Strem, Alfa Aesar, Chemetall, Aldrich) and stored in a dry box. [If it was not a fine powder, it was ground in the air immediately prior to storage]. 1,10-phenanthroline and tetramethyl-1,10-phenanthroline were purchased from Alfa Aesar and used without further purification. All other reagents were commercially available and used without further purification; all materials were weighed in the air. Flash column chromatography was performed with Silica gel 60 (0.040-0.063 mm, 230-400 mesh) purchased from EM science. IR spectra were recorded on a Perkin-Elmer FT-IR 1600 instrument. Elemental analyses were performed by Atlantic Microlabs, Inc., Norcross, GA. ¹H NMR and ¹³C NMR spectra were recorded on a Varian 300 MHz or 500 MHz instrument with chemical shifts reported relative to residual deuterated solvent peaks. Gas chromatographic analyses were performed on a Hewlett Packard 6890 instrument with an FID detector and a Hewlett Packard 10 m x 0.2 mm i.d. HP-1 capillary column. Mass spectra (GC/MS) were recorded on a Hewlett Packard model GCD. The microwave experiment was performed in a single mode cavity, producing controlled irradiation at 2450 MHz using a Personal Chemistry/Smith Synthesizer. Reaction temperature and pressure were determined using the built-in, on-line IR and pressure sensors. Microwave-mediated reactions were

performed in sealed process vials under air with magnetic stirring. All yields reported in Tables 1-3 refer to isolated yields (average of two runs) of compounds estimated to be >95% pure as determined by ^1H NMR and GC. All new compounds were further characterized by elemental analysis or high resolution mass spectroscopy. Diastereomeric ratios were determined by ^1H NMR or GC analysis of the crude reaction mixtures. *E/Z*-ratios of vinyl compounds were determined by ^1H NMR or GC analysis. The procedures in this section are representative, and thus the yields may differ from those in Tables 1-3. Compounds described in the literature were characterized by comparing their ^1H NMR spectra to the previously reported data.

Vinyl iodide 11. ¹E : Z = 88 : 12 (GC). ¹H NMR (500 MHz, CDCl₃): δ = 2.50 (t, *J* = 7.9 Hz, 2H), 2.18 (m, 2H), 1.92 (t, *J* = 0.6 Hz, 3H), 1.55 (mc, 2H), 1.45 (mc, 2H), 0.91 (t, *J* = 7.6 Hz, 3H), 0.90 (t, *J* = 7.6 Hz, 3H). ¹³C NMR (125 MHz, CDCl₃): δ = 140.39, 105.08, 43.23, 35.98, 30.05, 23.30, 21.82, 14.13, 13.25. IR (neat, cm⁻¹): 2960, 2931, 2871, 1461, 1378, 1237, 1100, 739. Anal. Calcd. for C₉H₁₇I: C, 42.87; H, 6.80. Found: C, 43.35; H 7.05.

General Procedures

General procedure A. A screw cap test tube was charged with the alcohol (2.0 mmol), dodecane (as internal standard) (227 μ L, 1.00 mmol), CuI (19.0 mg, 100 μ mol), tetramethyl-1,10-phenanthroline (47.3 mg, 200 μ mol), *E*-1-iododecene (266 mg, 1.00 mmol), Cs₂CO₃ (489 mg, 1.50 mmol) and toluene (500 μ L). The test tube was sealed with a screw cap and stirred vigorously at 80 °C for the indicated time. The resulting suspension was cooled to room temperature and filtered through a 1 x 2 cm pad of silica gel eluting with diethyl ether (100 mL). The filtrate was concentrated and the residue was purified by flash chromatography to afford pure product.

General procedure B. A screw cap test tube was charged with the alcohol (2.0 mmol), dodecane (as internal standard) (227 μ L, 1.00 mmol), CuI (19.0 mg, 100 μ mol), tetramethyl-1,10-phenanthroline (47.3 mg, 200 μ mol), the vinyl iodide (1.00 mmol), Cs₂CO₃ (978 mg, 3.00 mmol) and *o*-xylene (500 μ L). The test tube was then evacuated and backfilled with argon (flushing for 10 min). The test tube was sealed with a screw cap and stirred vigorously at 120 °C for 48 h. The resulting suspension was cooled to room temperature and filtered through a 1 x 2 cm pad of silica gel eluting with diethyl ether (100 mL). The filtrate was concentrated and the residue was purified by flash chromatography to afford pure product.

Vinylation of Alcohols

Allyl vinyl ether 3. Using general procedure A, *E*-2-undecene-1-ol (401 μ L, 2.00 mmol) and *E*-1-iododecene (266 mg, 1.00 mmol) were subjected to the indicated reaction conditions for 24 h. The crude product was purified by flash chromatography on silica gel (2x20 cm; 200 mL pentane followed by pentane/dichloromethane = 100:1; 10 mL fractions) providing the product as a colorless oil (199 mg, 65 % yield). *E* : *Z* > 95 : 5 (GC). ^1H NMR (500 MHz, CDCl_3): δ = 6.21 (d, J = 12.5 Hz, 1H), 5.73 (mc, 1H), 5.57 (mc, 1H), 4.80 (td, J = 12.5, 7.3 Hz, 1H), 4.11 (d, J = 6.1 Hz, 2H), 2.05 (quart, J = 7.0 Hz, 2H), 1.89 (quart, J = 7.0 Hz, 2H), 1.42-1.18 (m, 24H), 0.88 (t, J = 7.0 Hz, 6H). ^{13}C NMR (125 MHz, CDCl_3): δ = 146.23, 136.22, 125.79, 105.57, 70.74, 33.02, 32.61, 32.60, 31.41, 30.17, 30.16, 30.04, 29.98, 29.90, 29.73, 29.71, 28.46, 23.40, 23.39, 14.83. IR (neat, cm^{-1}): 2923, 2854, 1671, 1652, 1465, 1158, 969, 920, 721. Anal. Calcd. for $\text{C}_{21}\text{H}_{40}\text{O}$: C, 81.75; H, 13.07. Found: C, 81.77; H 13.15.

1-Heptyloxy-*E*-dec-1-ene. Using general procedure A, *n*-heptanol (283 μ L, 2.00 mmol) and *E*-1-iododecene (266 mg, 1.00 mmol) were subjected to the indicated reaction conditions for 36 h. The crude product was purified by flash chromatography on silica gel (2x20 cm; 200 mL pentane followed by pentane/dichloromethane = 5:1; 10 mL fractions) providing the product as a colorless oil (203 mg, 80 % yield). *E* : *Z* > 95 : 5 (GC). ^1H NMR (500 MHz, CDCl_3): δ = 6.21 (d, J = 12.7 Hz, 1H), 4.76 (dt, J = 12.5, 7.3 Hz, 1H), 3.62 (t, J = 6.7 Hz, 2H) 1.89 (quart, J = 6.9 Hz,

2 H), 1.62 (quint, $J = 7.3$ Hz, 2H), 1.40-1.22 (m, 20H), 0.88 (t, $J = 6.3$ Hz, 6H). ^{13}C NMR (125 MHz, CDCl_3): $\delta = 146.79, 104.89, 69.92, 32.61, 32.49, 31.49, 30.18, 30.06, 30.03, 29.79, 29.75, 28.50, 26.68, 23.40, 23.31, 14.83, 14.80$. IR (neat, cm^{-1}): 2956, 2923, 2854, 1654, 1466, 1129, 932, 724. Anal. Calcd. for $\text{C}_{17}\text{H}_{34}\text{O}$: C, 80.24; H, 13.47. Found: C, 80.46; H 13.70.

1-Hept-2-ynyloxy-*E*-dec-1-ene. Using general procedure A, 2-heptyn-1-ol (254 μL , 2.00 mmol) and *E*-1-iododecene (266 mg, 1.00 mmol) were subjected to the indicated reaction conditions for 24 h. The crude product was purified by flash chromatography on silica gel (2x20 cm; 200 mL pentane followed by pentane/dichloromethane = 5:1; 10 mL fractions) providing the product as a colorless oil (174 mg, 70 % yield). *E* : *Z* > 95 : 5 (GC). ^1H NMR (500 MHz, CDCl_3): $\delta = 6.22$ (d, $J = 12.5$ Hz, 1H), 4.87 (dt, $J = 12.5, 7.3$ Hz, 1H), 4.31 (t, $J = 2.1$ Hz, 2H), 2.23 (tt, $J = 7.0, 2.1$ Hz, 2H), 1.92 (quart, $J = 6.4$ Hz, 2H), 1.54-1.47 (m, 2H), 1.46-1.37 (m, 2H), 1.36-1.22 (m, 12H), 0.91 (t, $J = 7.3$ Hz, 3H), 0.88 (t, $J = 7.0$ Hz, 3H). ^{13}C NMR (125 MHz, CDCl_3): $\delta = 144.80, 106.32, 87.99, 75.27, 57.63, 32.15, 30.80, 30.76, 29.70, 29.57, 29.25, 27.93, 22.93, 22.15, 18.69, 14.37, 13.83$. IR (neat, cm^{-1}): 2958, 2923, 2854, 1673, 1656, 1466, 1364, 1160, 930, 724. Anal. Calcd. for $\text{C}_{17}\text{H}_{30}\text{O}$: C, 81.54; H, 12.08. Found: C, 81.45; H 12.04.

***E*-Dec-1-enyloxymethyl-benzene.** Using general procedure A, benzyl alcohol (207 μL , 2.00 mmol) and *E*-1-iododecene (266 mg, 1.00 mmol) were subjected to the indicated reaction conditions for 12 h. The crude product was purified by flash chromatography on silica gel (2x20

cm; 200 mL pentane followed by pentane/dichloromethane = 5:1; 10 mL fractions) providing the product as a colorless oil (188 mg, 76 % yield). *E* : *Z* > 95 : 5 (GC). ¹H NMR (500 MHz, CDCl₃): δ = 7.40-7.29 (m, 5H), 6.33 (d, *J* = 12.5 Hz, 1H), 4.86 (dt, *J* = 12.5, 7.3 Hz, 1H), 4.67 (s, 2H), 1.93 (quart, *J* = 7.2 Hz, 2H), 1.38-1.24 (m, 12H), 0.90 (t, *J* = 7.0 Hz, 3H). ¹³C NMR (125 MHz, CDCl₃): δ = 145.98, 137.61, 128.70, 128.04, 127.80, 105.53, 71.30, 32.15, 30.91, 29.71, 29.57, 29.27, 28.00, 22.94, 14.39. IR (neat, cm⁻¹): 2956, 2923, 2854, 1671, 1652, 1455, 1131, 930, 733, 697. Anal. Calcd. for C₁₇H₂₆O: C, 82.87; H, 10.64. Found: C, 82.73; H, 10.74.

(2-Methyl-propenyloxymethyl)-benzene.² Using general procedure A, benzyl alcohol (207 μL, 2.00 mmol) and 1-bromo-2-methylpropene (103 μL, 1.00 mmol) were subjected to the indicated reaction conditions for 48 h. The crude product was purified by flash chromatography on silica gel (2x20 cm; 200 mL pentane followed by pentane/dichloromethane = 5:1; 10 mL fractions) providing the product as a colorless liquid (104 mg, 64 % yield, compound volatile under reduced pressure). *E* : *Z* > 95 : 5 (GC).

Claisen Rearrangement

Aldehyde 5 (conventional heating). A test tube was charged with *E*-1-dec-1-enyl-*E*-undec-2-ene (160 mg, 519 μmol) and DMF (500 μL), evacuated and backfilled with argon (flushing for 10 min). This was then sealed and heated at 120 $^{\circ}\text{C}$ for 24 h. After cooling to room temperature, Et_2O (20 mL) was added and the solution was extracted with water (3 x 25 mL). The combined organic layers were dried over MgSO_4 and concentrated under reduced pressure. The product was obtained as a yellow oil without further purification (144 mg, 90% yield). $\text{dr} = 94 : 6$ (^1H NMR).

^1H NMR (500 MHz, CDCl_3): $\delta = 9.59$ (d, $J = 3.7$ Hz, 1H), 5.61 (td, $J = 17.1, 9.2$ Hz, 1H), 5.09 (dd, $J = 10.1, 1.8$ Hz, 1H), 5.04 (dd, $J = 17.8$, Hz, 1H), 2.35-2.26 (m, 1H), 2.25-2.19 (m, 1H), 1.70-1.58 (m, 2H), 1.50-1.43 (m, 2H), 1.37-1.16 (m, 24H), 0.88 (t, $J = 7.3$ Hz, 6H). ^{13}C NMR (125 MHz, CDCl_3): $\delta = 205.98, 139.44, 116.97, 56.16, 45.27, 32.13, 32.11, 32.09, 29.92, 29.76, 29.64, 29.53, 29.47, 27.71, 27.47, 27.11, 22.90, 14.36$. IR (neat, cm^{-1}): 2956, 2923, 2854, 1725, 1466, 996, 917, 722.

Aldehyde 5 (microwave heating). A microwave vial was charged with *E*-1-dec-1-enyl-*E*-undec-2-ene (76 mg, 247 μ mol) and DMF (1 mL). This was then sealed and heated at 250 $^{\circ}$ C for 5 min in a microwave synthesizer. Et₂O was added (20 mL) and extracted with water (3 x 25 mL). The combined organic layers were dried over MgSO₄ and concentrated under reduced pressure. The product was obtained as a yellow oil without further purification (61 mg, 80% yield). dr = 91 : 9 (¹H NMR).

Domino Reactions

Aldehyde 4. A screw cap test tube was charged with *E*-2-undecene-1-ol (401 μ L, 2.0 mmol), dodecane (227 μ L, 1.00 mmol), CuI (19.0 mg, 100 μ mol), 1,10-phenanthroline (36.0 mg, 200 μ mol), *E*-1-iododecene (266 mg, 1.00 mmol), Cs₂CO₃ (489 mg, 1.50 mmol) and *o*-xylene (500 μ L). The test tube was sealed with a screw cap and stirred vigorously at 120 $^{\circ}$ C for 24 h. The resulting suspension was cooled to room temperature and filtered through a 1x2 cm pad of silica gel eluting with diethyl ether (100 ml). After concentration of the combined organic layers under reduced pressure, the crude product was purified by flash chromatography on silica gel (2x20 cm; 200 mL pentane followed by pentane/dichloromethane = 3:1; 10 mL fractions) providing the product 2,3-dioctyl-pent-4-enal as a yellow oil (188 mg, 61 % yield) and *E*-1-dec-1-enyloxy-*E*-undec-2-ene (4.0 mg, 1 % yield) as a colorless oil. 2,3-dioctyl-pent-4-enal (1:1 mixture of diastereoisomers): ¹H NMR (500 MHz, CDCl₃): δ = 9.56 (d, *J* = 3.6 Hz, 1H), 9.52 (d, *J* = 3.9 Hz, 1H), 5.61 (td, *J* = 16.8, 7.4 Hz, 1H), 5.49 (td, *J* = 17.1, 6.9 Hz, 1H), 5.11-4.97 (m, 2H), 2.40-2.10 (m, 2H), 1.77-1.00 (m, 28H), 0.88 (t, *J* = 7.2 Hz, 6H). ¹³C NMR (125 MHz, CDCl₃): δ = 182.33, 181.76, 140.31, 140.10, 117.58, 117.06, 51.01, 50.82, 47.89, 47.24, 33.36, 32.66, 32.58, 32.55, 30.65, 30.54, 30.28, 30.25, 30.22, 30.18, 30.12, 30.00, 29.95, 28.35, 28.29, 27.88, 27.83, 23.38, 14.82. HRMS (ESI) Calcd. for C₂₁H₄₀O [M + Na] 347.2921; Found 347.2915.

Aldehyde 9. Using the general procedure B, *E*-2-hexene-1-ol (236 μ L, 2.00 mmol) was reacted with *E*-1-iodo-2-methyl-hept-1-ene (238 mg, 1.00 mmol). The crude product was purified by flash chromatography on silica gel (2x20 cm; 200 mL pentane followed by pentane/dichloromethane = 5:1; 10 mL fractions) providing the product as a yellow oil (116 mg, 55 % yield). dr = 92 : 8 (^1H NMR). ^1H NMR (500 MHz, CDCl_3): δ = 9.45 (s, 1H), 5.53 (td, J = 17.1, 10.1 Hz, 1H), 5.09 (dd, J = 10.1, 1.8 Hz, 1H), 5.01 (ddd, J = 16.8, 2.1, 0.6 Hz, 1H), 2.17 (t, J = 9.9 Hz, 1H), 1.61-1.02 (m, 12H), 0.99 (s, 3H), 0.91-0.85 (m, 6H). ^{13}C NMR (125 MHz,

CDCl_3): δ = 207.32, 138.21, 117.94, 51.79, 49.72, 34.17, 32.81, 30.46, 23.92, 22.73, 21.01, 15.51, 14.26, 14.16. IR (neat, cm^{-1}): 2954, 2929, 2873, 1723, 1466, 1380, 1191, 917.

Aldehyde 10. Using the general procedure B, Z-2-hexene-1-ol (237 μL , 2.00 mmol) was reacted with *E*-1-iodo-2-methyl-hept-1-ene (238 mg, 1.00 mmol). The crude product was purified by flash chromatography on silica gel (2x20 cm; 200 mL pentane followed by pentane/dichloromethane = 5:1; 10 mL fractions) providing the product as a yellow oil (142 mg, 68 % yield). dr = 5 : 95 (^1H NMR).

^1H NMR (500 MHz, CDCl_3): δ = 9.42 (s, 1H), 5.49 (td, J = 17.1, 9.8 Hz, 1H), 5.13 (dd, J = 10.2, 2.0 Hz, 1H), 5.04 (dd, J = 16.9, 2.0 Hz, 1H), 2.19 (dt, J = 9.7, 2.3 Hz, 1H), 1.58-1.00 (m, 12H), 0.96 (s, 3H), 0.87-0.82 (m, 6H). ^{13}C NMR (125 MHz, CDCl_3): δ = 207.99, 137.85, 118.67, 52.62, 49.42, 36.06, 33.19, 32.61, 24.18, 23.17, 21.53, 14.70, 14.60, 14.54. IR (neat, cm^{-1}): 2956, 2931, 2861, 1725, 1466, 1380, 1162, 915. Anal. Calcd. for $\text{C}_{14}\text{H}_{26}\text{O}$: C, 79.94; H, 12.46. Found: C, 79.64; H, 12.52.

Ketone 12. Using the general procedure B, *E*-2-hexen-1-ol (236 μL , 2.00 mmol) was reacted with *Z*-4-iodo-5-methyl-oct-4-ene (238 mg, 1.00 mmol). The crude product was purified by flash chromatography on silica gel (2x20 cm; 200 mL pentane followed by pentane/dichloromethane = 3:1; 10 mL fractions) providing the product as a yellow oil (153 mg, 68 % yield). dr = 88 : 12 (GC). ^1H NMR (500 MHz, CDCl_3): δ = 5.49 (td, J = 17.1, 9.6 Hz, 1H), 5.01 (dd, J = 10.2, 2.2 Hz, 1H), 4.92 (ddd, J = 16.8, 2.2, 0.6 Hz, 1H), 2.42-2.23 (m, 2H), 2.15 (t, J = 9.9 Hz, 1H), 1.70-0.80 (m, 19H), 1.04 (s, 3H). ^{13}C NMR (125 MHz, CDCl_3): δ = 215.74, 139.03, 117.70, 54.96, 52.23, 41.11, 39.58, 31.14, 21.51, 18.54, 17.62, 17.41, 15.60, 14.71, 14.63. IR (neat, cm^{-1}): 2958, 2935, 2873, 1702, 1466, 1380, 1117, 1000, 915, 741. HRMS (ESI) Calcd. for $\text{C}_{15}\text{H}_{28}\text{O}$ [$\text{M} + \text{Na}$] 247.2032; Found 247.2040.

Aldehyde 16. Using the general procedure B, geraniol (347 μ L, 2.00 mmol) was reacted with *E*-1-iodo-2-methyl-hept-1-ene (238 mg, 1.00 mmol). The crude product was purified by flash chromatography on silica gel (2x20 cm; 200 mL pentane followed by pentane/dichloromethane = 3:1; 10 mL fractions) providing the product as a yellow oil (203 mg, 77 % yield). dr = 92 : 8 (^1H NMR). ^1H NMR (500 MHz, CDCl_3): δ = 9.61 (s, 1H), 5.81 (dd, J = 17.5, 11.0 Hz, 1H), 5.19 (dd, J = 11.0, 0.9 Hz, 1H), 5.05 (t, J = 7.2 Hz, 1H), 4.99 (dd, J = 17.4, 0.9 Hz, 1H), 1.90-1.70 (m, 4H), 1.66 (s, 3H), 1.56 (s, 3H), 1.52-1.08 (m, 8H), 1.02 (s, 3H), 0.99 (s, 3H), 0.85 (t, J = 7.0 Hz, 3H). ^{13}C NMR (125 MHz, CDCl_3): δ = 208.59, 142.63, 132.21, 125.19, 115.94, 54.84, 45.88,

35.94, 33.40, 31.66, 26.38, 25.08, 23.50, 23.25, 18.30, 17.66, 14.73, 14.34. IR (neat, cm^{-1}): 2956, 2929, 2860, 2713, 1721, 1457, 1378, 916. Anal. Calcd. for $\text{C}_{18}\text{H}_{32}\text{O}$: C, 81.75; H, 12.20. Found: C, 81.62; H, 12.23.

Aldehyde 17. Using the general procedure B, nerol (351 μL , 2.00 mmol) was reacted with *E*-1-iodo-2-methyl-hept-1-ene (238 mg, 1.00 mmol). The crude product was purified by flash chromatography on silica gel (2x20 cm; 200 mL pentane followed by pentane/dichloromethane = 3:1; 10 mL fractions) providing the product as a yellow oil (204 mg, 77 % yield). dr = 6 : 94 (^1H NMR). ^1H NMR (500 MHz, CDCl_3): δ = 9.60 (s, 1H), 5.81 (dd, J = 17.7, 11.0 Hz, 1H), 5.21 (dd, J = 11.0, 1.2 Hz, 1H), 5.07 (tquint, J = 7.3, 5.1 Hz, 1H), 4.98 (dd, J = 17.7, 1.5 Hz, 1H), 1.90-1.71 (m, 4H), 1.67 (s, 3H), 1.56 (s, 3H), 1.54-1.10 (m, 8H), 1.02 (s, 3H), 0.97 (s, 3H), 0.86 (t, J = 7.0 Hz, 3H). ^{13}C NMR (125 MHz, CDCl_3): δ = 208.73, 142.37, 131.79, 124.76, 115.61, 54.34, 45.08, 35.31, 32.94, 30.94, 25.94, 24.59, 22.88, 22.78, 17.85, 17.16, 14.26, 13.50. IR (neat, cm^{-1}): 2956, 2929, 2860, 2715, 1719, 1459, 1378, 914. Anal. Calcd. for $\text{C}_{18}\text{H}_{32}\text{O}$: C, 81.75; H, 12.20. Found: C, 81.79; H 12.29.

Ketone 18. Using the general procedure B, geraniol (347 μL , 2.00 mmol) was reacted with *Z*-4-iodo-5-methyl-oct-4-ene (252 mg, 1.00 mmol). The crude product was purified by flash

chromatography on silica gel (2x20 cm; 200 mL pentane followed by pentane/dichloromethane = 3:1; 10 mL fractions) providing the product as a yellow oil (168 mg, 60 % yield). dr = 87 : 13 (^1H NMR). ^1H NMR (500 MHz, CDCl_3): δ = 5.80 (dd, J = 17.4, 11.0 Hz, 1H), 5.11 (dd, J = 10.9, 1.5 Hz, 1H), 5.05 (mc, 1H), 4.89 (dd, J = 17.6, 1.5 Hz, 1H), 2.45-2.20 (m, 2H), 2.00-1.69 (m, 2H), 1.63 (s, 3H), 1.55 (s, 3H), 1.62-1.02 (m, 8H), 1.15 (s, 3H), 0.96 (s, 3H), 0.94-0.78 (m, 6H). ^{13}C NMR (125 MHz, CDCl_3): δ = 216.14, 143.11, 131.43, 125.10, 114.54, 56.46, 45.49, 44.47, 36.76, 34.87, 25.91, 23.22, 18.75, 17.82, 17.79, 17.38, 17.10, 15.20, 14.08. IR (neat, cm^{-1}): 2964, 2929, 2873, 1696, 1457, 1376, 1111, 1007, 913.

Ketone 19. Using the general procedure B, nerol (351 μL , 2.00 mmol) was reacted with *Z*-4-iodo-5-methyl-oct-4-ene (252 mg, 1.00 mmol). The crude product was purified by flash chromatography on silica gel (2x20 cm; 200 mL pentane followed by pentane-dichloromethane 2:1; 10 mL fractions) providing the product as a yellow oil (211 mg, 76 % yield). $n_D = 1.288$ (1H NMR). ^1H NMR (300 MHz, CDCl_3): δ = 5.66 (dd, J = 17.6, 10.9 Hz, 1H), 5.15 (dd, J = 10.9, 1.5 Hz, 1H), 5.06 (mc, 1H), 4.94 (dd, J = 17.6, 1.5 Hz, 1H), 2.34 (t, J = 7.3 Hz, 2H), 1.97-1.67 (m, 2H), 1.66 (s, 3H), 1.54 (s, 3H), 1.62-1.40 (m, 4H), 1.30-1.15 (m, 4H), 1.13 (s, 3H), 0.96 (s, 3H), 0.93-0.78 (m, 6H). ^{13}C NMR (125 MHz, CDCl_3): δ = 216.64, 143.84, 131.84, 125.64, 115.29, 57.36, 46.47, 44.71, 36.78, 35.60, 26.40, 24.07, 19.32, 18.31, 18.10, 17.97, 17.04, 15.71, 14.60. IR (neat, cm^{-1}): 2962, 2931, 2873, 1696, 1465, 1376, 1111, 1015, 913. Anal. Calcd. for $\text{C}_{19}\text{H}_{34}\text{O}$: C, 81.95; H, 12.31. Found: C, 81.65; H 12.39.

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