Supporting Materials and Methods

General. Organic solvents were dried by standard methods. Other commercially obtained reagents were used without additional purification. Flash chromatography was performed on 230–400 mesh silica gel. Thin layer chromatography (TLC) was performed on glass plates coated with a 0.02-mm layer of silica gel 60 F-254. ^{1}H and ^{13}C NMR spectra were recorded at 600 or 400 and 150 or 100 MHz, respectively, and the chemical shifts are reported in parts per million on a δ scale with $CDCl_3$ as reference unless otherwise noted. Wherever necessary, ^{1}H NMR assignments were supported by appropriate homonuclear correlation experiments (COSY). Quantitative ^{13}C NMR measurements were performed by using the inverse-gated decoupling pulse sequence, with an ≈30° pulse and 10-sec relaxation delays. Optical rotations were measured at 25°C at the sodium line. Mass spectra and high-resolution mass spectra (HRMS) were recorded by using electron ionization or by the fast atom bombardment (FAB) technique using *p*-nitrobenzyl alcohol as solvent.

(3*R*,4*R*)-4-Benzyloxymethoxy-5-(*tert*-butyl-diphenyl-silanyloxy)-3-methyl-pentanal (4b) (ref. 1). To a solution of $(COCl)_2$ (1.48 ml, 8.58 mmol) in CH_2Cl_2 (20 ml) was added DMSO (1.2 ml, 17.16 mmol) at –78°C. The resulting solution was stirred at that temperature for 15 min, and alcohol **4a** (1.41 g, 2.86 mmol) in CH_2Cl_2 (10 ml) was added slowly via cannula. The solution was stirred at –78°C for 30 min, and NEt_3 (3.98 ml, 28.6 mmol) was added. The resulting slurry was stirred at –70°C for another 20 min and then quenched with saturated NH_4Cl (15 ml). The aqueous phase was extracted with CH_2Cl_2 (3 × 50 ml), and the organic phase was successively washed with 2% HCl (50 ml) and brine (50 ml) and dried over Na_2SO_4. After evaporation, the residue was purified by flash chromatography (8:2 hexanes/EtOAc) to give aldehyde **4b** (1.34 g, 96%) as a colorless oil. $[\alpha]_D$ +23.9° (*c* 1.7, $CHCl_3$); IR ($CHCl_3$) 2,932, 2,858, 1,725, 1,428, 1,112, 1,040 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 9.78 (s, 1H), 7.81–7.68 (m, 4H), 7.55–7.25 (m, 11H), 4.88 (d, 1H, *J* = 6.9 Hz), 4.78 (d, 1H, *J* = 6.9 Hz), 4.66 (d, 1H, *J* = 11.9 Hz), 4.56 (d, 1H, *J* = 11.9 Hz), 3.93–3.72 (m, 2H), 3.62 (m, 1H), 2.73–2.50 (m, 2H), 2.30 (m, 1H), 1.15 (s, 9H), 1.07 (d, 3H, *J* = 6.8 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 202.6, 138.3, 136.1, 136.0, 133.7, 130.3, 128.8, 128.2, 128.1, 128.0, 95.0, 82.2, 70.3, 64.3, 47.4, 30.3, 27.3, 19.7, 17.6; MS [electrospray (ES+)] *m*/*z* (%) 513.3 ($M + Na^+$, 100), 282.0 (85).

(5*R*,6*R*)-6-Benzyloxymethoxy-7-(*tert*-butyl-diphenyl-silanyloxy)-5-methyl-hept-2-enoic Acid 1-Methyl-cyclopentyl Ester (6). To a stirred solution of aldehyde **4b** (1.34 g, 2.73 mmol) in CH_2Cl_2 (25 ml) was added PPh_3CHCO_2MCP (1.54 g, 4.09 mmol) at room temperature (rt). The resulting solution was stirred for 13 h and evaporated to dryness. The residue was triturated with hexanes/Et_2O (3:1) (100 ml), and the suspension was filtered through a short silica pad to remove triphenylphosphine oxide. After evaporation of the filtrate, the residue was purified by flash chromatography (95:5 hexanes/EtOAc) to give ester **6** (1.46 g, 91%) as a colorless oil. $[\alpha]_D$ +11.6 (*c* 0.47, $CHCl_3$); IR ($CHCl_3$) 2,960, 1,713, 1,653, 1,472, 1,428, 1,168, 1,112, 1,040 cm^{-1}; ^{1}H NMR (400 MHz, C_6D_6) δ (ppm) 7.94–7.80 (m, 4H), 7.42–7.22 (m, 11H), 6.00 (d, 1H, *J* = 15.5 Hz), 4.90 (d, 1H, *J* = 6.8 Hz), 4.74 (d, 1H, *J* = 6.8 Hz), 4.63 (d, 1H, *J* = 12.1 Hz), 4.55 (d, 1H, *J* = 12.1 Hz),

3.89 (dd, 1H, J = 6.6 and 10.8 Hz), 3.81 (dd, 1H, J = 6.6 and 10.8 Hz), 3.63 (q, 1H, J = 4.3 and 10.6 Hz), 2.34 (m, 3H), 2.04 (m, 2H), 1.70 (m, 7H), 1.50 (m, 2H), 1.24 (s, 9H), 0.88 (d, 3H, J = 6.4 Hz); ^{13}C NMR (125 MHz, CD_2Cl_2) δ (ppm) 172.7, 166.2, 147.2, 147.0, 138.6, 136.4, 136.0, 135.9, 133.8, 130.1, 128.6, 128.1, 128.0, 127.8, 124.5, 95.3, 89.6, 82.2, 70.1, 64.5, 39.5, 35.1, 34.5, 30.9, 27.0, 26.8, 26.6, 24.6, 24.1, 19.5, 16.2; MS (FAB+) m/z (%) 613.3 (M – H^+, 10).

(3*S*,5*R*,6*R*)-6-Benzyloxymethoxy-7-(*tert*-butyl-diphenyl-silanyloxy)-3,5-dimethyl-heptanoic Acid *tert*-Butyl Ester (7 and Its Epimer 7'). To a suspension of CuI (1.67 g, 8.78 mmol) in tetrahydrofuran (THF) (27 ml) was added MeLi•LiBr (1.5 M in Et_2O, 11.7 ml, 17.56 mmol) at –20°C, and the mixture was allowed to warm up to 0°C over 30 min and then cooled to –78°C. To the resulting mixture was added chlorotrimethylsilane (TMSCl) (3.33 ml, 26.28 mmol) followed by a solution of **6** (0.9 g, 1.46 mmol) in THF (9 ml). After stirring for 3 h at –78°C, the mixture was quenched with a NH_4OH/NH_4Cl pH 8 buffer solution (50 ml) and stirred at rt for 30 min. The mixture was diluted with Et_2O (50 ml), and the aqueous layer was extracted with Et_2O (3 × 50 ml), the organic extracts were washed with NH_4OH/NH_4Cl (1:1) (50 ml), saturated NH_4Cl (50 ml), and brine (50 ml) and then dried over Na_2SO_4. The residue was purified by flash chromatography (95:5 hexanes/EtOAc) to give ester **7** and **7'** as an 8:1 mixture (804 mg, 87%). $[\alpha]_D$ +20.4 (c 0.23, $CHCl_3$); IR ($CHCl_3$) 2,932, 1,730, 1,428, 1,112, 1,040 cm^{-1}; ^{1}H NMR (400 MHz, C_6D_6) δ (ppm) 8.00–7.78 (m, 4H), 7.48–7.08 (m, 11H), 5.04 (d, 1H, J = 6.8 Hz), 4.84 (d, 1H, J = 6.8 Hz), 4.72 (d, 1H, J = 12.1 Hz), 4.59 (d, 1H, J = 12.1 Hz), 4.03 (dd, 1H, J = 6.6 and 10.8 Hz), 3.94 (dd, 1H, J = 6.6 and 10.8 Hz), 3.682 (q, 1H, J = 4.3 and 10.6 Hz), 2.39 (dd, 1H, J = 5.1 and 14.3 Hz), 2.25 (m, 3H), 2.09 (m, 1H), 1.98 (dd, 1H, J = 8.4 and 14.2 Hz), 1.75 (m, 2H), 1.66 (m, 5H), 1.07 (m, 3H), 1.27 (m, 9H), 1.14 (m, 1H), 1.07 (d, 3H, J = 6.6 Hz), 1.01 (d, 3H, J = 6.9 Hz) ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 173.3, 173.0 (min), 138.5, 136.1, 136.0, 133.9, 130.1, 130.0, 128.8, 128.2, 128.1, 127.9, 95.3, 89.9, 83.3 (min), 82.8, 70.0, 64.6, 44.6 (min), 42.4, 39.8, 39.6, 39.4, 32.7, 32.4 (min), 28.6, 28.4 (min), 27.3, 24.8, 24.2, 21.2, 19.6, 19.2 (min), 16.4, 15.9 (min); MS (FAB+) m/z (%) 629.6 (M – H^+, 10).

(3*S*,5*R*,6*R*)-6-Benzyloxymethoxy-7-(*tert*-butyl-diphenyl-silanyloxy)-3,5-dimethyl-heptan-1-ol (7a). To a stirred solution of **7** and **7'** (1.0 g, 1.58 mmol) in CH_2Cl_2 (20 ml) at –78°C was slowly added diisobutylaluminum hydride (DIBAL-H) (1.5 M in PhMe, 7 ml, 10.38 mmol), the resulting solution was stirred at –78°C for 6 h, and the mixture was quenched with 7 ml of MeOH at –78°C. The solution was diluted with 20 ml of EtOAc, stirred at rt for 30 min, filtered through a pad of Celite, and then washed with hot EtOAc. The combined organic layers were concentrated *in vacuo*, and the residue was purified by flash chromatography (8:2 hexanes/EtOAc) to give alcohol **7a** (550 mg, 65%), a (1:1) mixture of diastereomers (100 mg, 12%), and the **7a'** *R* isomer (25 mg, 3%) as colorless oils. Data for **7a** (80%): $[\alpha]_D$ +23.4 (c 0.81, $CHCl_3$); IR ($CHCl_3$) 3,430, 3,071, 2,957, 2,868, 1,960, 1,890, 1,584, 1,472, 1,428, 1,112, 1,041 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.74–7.65 (m, 4H), 7.52–7.23 (m, 11H), 4.97 (d, 1H, J = 6.9 Hz), 4.86 (d, 1H, J = 6.9 Hz), 4.72 (d, 1H, J = 11.8 Hz), 4.54 (d, 1H, J = 11.8 Hz), 3.86–3.51 (m, 5H), 1.98 (bs, 1H), 1.63 (m, 2H), 1.56–1.32 (m, 2H), 1.27 (m, 1H), 1.09 (s, 9H), 1.02 (m, 1H), 0.96–0.84 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 138.4, 136.1, 136.0, 133.9,

133.8, 130.2, 130.1, 128.8, 128.2, 128.1, 128.0, 95.2, 82.8, 69.4, 64.6, 61.4, 40.4, 34.3, 32.7, 27.6, 27.3, 21.1, 19.6, 16.5; MS (ES+) *m*/*z* (%) 557.4($M + Na^+$, 100), 282.2 (54).

(3*S*,5*R*,6*R*)-6-Benzyloxymethoxy-7-(*tert*-butyl-diphenyl-silanyloxy)-3,5-dimethyl-heptanal (7b). To a solution of $(COCl)_2$ (4.2 ml, 24.5 mmol) in CH_2Cl_2 (40 ml) was added DMSO (3.42 ml, 49.0 mmol) at –78°C. The resulting solution was stirred at that temperature for 15 min, and alcohol **7a** (5.35 g, 10.0 mmol) in CH_2Cl_2 (10 ml) was added slowly via cannula. The solution was stirred at –78°C for 30 min, NEt_3 (7.0 ml, 50.3 mmol) was added, and the resulting slurry was stirred at –70°C for another 20 min and then quenched with saturated NH_4Cl (50 ml). The aqueous phase was extracted with CH_2Cl_2 (3 × 50 ml). The organic phase were successively washed with 2% HCl (50 ml) and brine (50 ml) and dried over Na_2SO_4. After evaporation, the residue was purified by flash chromatography (8:2 hexanes/EtOAc) to give aldehyde **7b** (4.5 g, 88%) as a colorless oil. $[\alpha]_D$ +20.4° (*c* 0.80, $CHCl_3$); IR ($CHCl_3$) 2,931, 1,725, 1,472, 1,428, 1,112, 1,040 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ (ppm) 9.72 (s, 1H), 7.82–7.66 (m, 4H), 7.51–7.25 (m, 11H), 4.95 (d, 1H, *J* = 6.9 Hz), 4.84 (d, 1H, *J* = 6.9 Hz), 4.69 (d, 1H, *J* = 11.8 Hz), 4.59 (d, 1H, *J* = 11.8 Hz), 3.87–3.70 (m, 2H), 3.66 (m, 1H), 2.38 (m, 1H), 2.21–2.04 (m, 2H), 2.02–1.87 (m, 1H), 1.41 (m, 1H), 1.16 (s, 11H), 1.04–0.88 (m, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 203.2, 136.1, 136.0, 133.9, 130.2, 130.1, 128.8, 128.2, 128.0, 95.3, 82.6, 70.0, 64.4, 50.6, 39.9, 32.7, 27.3, 26.3, 21.5, 19.6, 16.5; MS (ES+) *m*/*z* (%) 555.3 ($M + Na^+$, 100), 282.2 (41).

(3*R*,5*R*,6*R*)-8-Benzyloxymethoxy-9-(*tert*-butyl-diphenyl-silanyloxy)-5,7-dimethyl-non-2-enoic Acid *tert*-Butyl Ester (8). To a stirred solution of aldehyde **7b** (0.3 g, 0.56 mmol) in CH_2Cl_2 (5 ml) was added PPh_3CHCO_2MCP (0.45 g, 1.12 mmol) at rt. The resulting solution was stirred for 12 h and then evaporated to dryness. The residue was triturated with hexanes/Et_2O (3:1) (15 ml), and the suspension was filtered through a silica pad to remove triphenylphosphine oxide. After evaporation of the filtrate, the residue was purified by flash chromatography (95:5 hexanes/EtOAc) to give ester **8** (0.3 g, 81%) as a colorless oil. $[\alpha]_D$ +17.5 (*c* 0.29, $CHCl_3$); IR ($CHCl_3$) 2,957, 1,714, 1,653, 1,428, 1,170, 1,113, 1,040; ^1H NMR (400 MHz, C_6D_6) δ (ppm) 7.90 (m, 4H), 7.47–7.01 (m, 12H), 5.98 (d, 1H, *J* = 15.5 Hz), 5.05 (d, 1H, *J* = 6.8 Hz), 4.86 (d, 1H, *J* = 6.8 Hz), 4.75 (d, 1H, *J* = 12.1 Hz), 4.61 (d, 1H, *J* = 12.1 Hz), 4.01 (dd, 1H, *J* = 6.6, 10.8 Hz), 3.90 (dd, 1H, *J* = 4.3, 10.8 Hz), 3.77 (m, 1H), 2.42–2.28 (m, 2H), 2.12 (m, 1H), 2.02 (m, 1H), 1.70 (m, 8H), 1.59 (m,1H), 1.51 (m, 2H), 1.41 (m, 1H), 1.26 (s, 9H), 1.01 (m, 1H), 0.93 (d, 3H, *J* = 6.9 Hz), 0.83 (d, 3H, *J* = 7.2 Hz); ^{13}C NMR (100 MHz, CD_2Cl_2) δ (ppm) 166.2, 147.0, 138.8, 136.04, 136.02, 133.9, 130.1, 130.0, 128.6, 128.1, 128.0, 127.8, 124.5, 95.3, 89.6, 82.6, 69.8, 64.5, 40.0, 39.5, 38.9, 32.6, 30.5, 27.0, 26.6, 24.6, 20.5, 19.4, 16.2; MS (FAB+) *m*/*z* (%) 657.2 ($M + H^+$, 100).

(3*S*,5*R*,7*R*,8*R*)-8-Benzyloxymethoxy-9-(*tert*-butyl-diphenyl-silanyloxy)-3,5,7-trimethyl-nonanoic Acid 1-Methyl-cyclopentyl Ester (9). To a suspension of CuI (266 mg, 1.39 mmol) in THF (4.3 ml) was added MeLi•LiBr (1.5 M in Et_2O, 1.86 ml, 2.79 mmol) at –15°C, and the mixture was allowed to warm up to 0°C over 30 min and then cooled to –78°C. To the resulting mixture was added TMSCl (0.531 ml, 4.19 mmol) followed by a solution of **8** (153 mg, 0.233 mmol) in THF (1.4 ml). The mixture was

stirred for 3 h at –78°C and quenched with a NH_4OH/NH_4Cl pH 8 buffer solution (5 ml) and stirred at rt for 30 min. The aqueous layer was extracted with Et_2O (3 × 5 ml), the organic extracts were washed with NH_4OH/NH_4Cl (1:1) (5 ml), saturated NH_4Cl (5 ml), and brine (5 ml), and dried over Na_2SO_4. The residue was purified by flash chromatography (95:5 hexanes/EtOAc) to give ester **9** (145 mg, 92%) as a colorless oil (12:1 *syn*/*syn*/*syn*/*anti*). $[\alpha]_D$ +22.0 (*c* 0.05, $CHCl_3$); IR ($CHCl_3$) 2,958, 1,727, 1,113, 1,041 cm^{-1}; ^1H NMR (400 MHz, C_6D_6) δ (ppm) 7.93 (m, 4H), 7.50–7.12 (m, 11H), 5.08 (d, 1H, *J* = 6.8 Hz), 4.90 (d, 1H, *J* = 6.8 Hz), 4.76 (d, 1H, *J* = 12.1 Hz), 4.62 (d, 1H, *J* = 12.1 Hz), 4.04 (dd, 1H, *J* = 6.5 and 10.7 Hz), 3.96 (dd, 1H, *J* = 6.5 and 10.7 Hz), 3.89 (m, 1H), 2.35 (dd, 1H, *J* = 5.1 and 14.3 Hz), 2.32–2.21 (m, 2H), 2.17 (m, 1H), 2.02 (dd, 1H, *J* = 8.4 and 14.3 Hz), 1.74 (m, 2H), 1.67 (m, 6H), 1.51 (m, 3H), 1.41 (m, 1H), 1.27 (s, 9H), 1.07 (d, 3H + 1H, *J* = 6.6 Hz), 1.02 (d, 3H, *J* = 6.9 Hz), 0.97 (d, 3H + 1H, *J* = 6.5 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 173.3, 173.1 (min), 138.5, 136.3, 133.9, 130.1, 128.1, 127.9, 95.3, 89.8, 83.0 (min), 82.7, 69.9, 64.6, 44.9 (min), 44.5, 43.6 (min), 42.7, 40.9 (min), 40.6, 39.6, 39.5, 32.6, 28.4, 28.2, 27.3, 21.4, 21.3, 19.9, 19.6 (min), 16.6, 16.4 (min); MS (FAB+) *m*/*z* (%) 671.1 ($M - H^+$, 10).

(3*S*,5*R*,7*R*,8*R*)-8-Benzyloxymethoxy-9-(*tert*-butyl-diphenyl-silanyloxy)-3,5,7-trimethyl-nonan-1-ol (9a). To a stirred solution of **9** (100 mg, 0.148 mmol) in CH_2Cl_2 (1.5 ml) at –78°C was slowly added DIBAL-H (1.5 M in PhMe, 980 µl, 1.48 mmol), and the resulting solution was stirred at –78°C for 3 h and quenched with 1 ml of MeOH at –78°C. The solution was diluted with EtOAc (5 ml) and stirred at rt for 30 min. The resulting suspension was filtered through a Celite pad and washed with EtOAc (20 ml). After evaporation of the organic phase, the residue was purified by flash chromatography (9:1 hexanes/EtOAc) to give alcohol **9a** (69 mg, 81%) as a colorless oil. $[\alpha]_D$ +24.1 (*c* 1.12, $CHCl_3$); IR ($CHCl_3$) 3,370, 2,930, 1,461, 1,428, 1,379, 1,112, 1,040 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.80–7.67 (m, 4H), 7.51–7.23 (m, 11H), 4.97 (d, 1H, *J* = 6.8 Hz), 4.86 (d, 1H, *J* = 6.8 Hz), 4.72 (d, 1H, *J* = 11.8 Hz), 4.59 (d, 1H, *J* = 11.8 Hz), 3.86–3.54 (m, 5H), 2.00 (bs, 1H), 1.82–1.49 (m, 3H), 1.48–1.17 (m, 4H), 1.08 (s, 9H), 1.02–0.79 (m, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 138.4, 136.1, 136.0, 134.0, 133.9, 130.1, 130.0, 128.8, 128.2, 128.1, 127.9, 95.3, 82.8, 69.9, 64.6, 61.5, 45.2, 40.7, 39.6, 32.7, 28.2, 27.4, 27.3, 21.6, 21.2, 19.6, 16.6; MS (ES+) *m*/*z* (%) 599.3 ($M + Na^+$, 100), 583.0 (55), 236.1 (48).

(2*R*,3*R*,5*R*,7*S*)-(2-Benzyloxymethoxy-3,5,7-trimethyl-non-8-enyloxy)-*tert*-butyl-diphenyl-silane (10). To a stirred solution of **9a** (1.11 g, 1.92 mmol) and 2-nitrophenyl selenocyanate (0.5 g, 1.2 eq) in 15 ml of anhydrous THF was added dropwise $(n\text{-Bu})_3P$ (0.55 ml, 1.2 eq), and the resulting solution was stirred for 1 h. The reaction mixture was concentrated and dried *in vacuo* to give a yellow crude selenoether that was used in the next step without additional purification.

To a solution of the above product in 5 ml of THF was added at 0°C 1.55 ml of a 30% aqueous hydrogen peroxide solution, and the resulting solution was then stirred for 2 h, then quenched by addition of 25 ml of water, and diluted with Et_2O (50 ml). The aqueous phase was separated and the organic phase was washed with saturated $NaHCO_3$ solution (2 × 15 ml). The organic phase was dried over Na_2SO_4 and concentrated *in vacuo*. The

residue was purified by flash chromatography (97:03 hexanes/EtOAc) to give **10** (735 mg, 68%) as a colorless oil. $[\alpha]_D$ +29.8 (*c* 1.45, $CHCl_3$); IR (film) 2,960, 2,930, 1,428, 1,113 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.73–7.71 (m, 4H), 7.46–7.30 (m, 11H), 5.60 (ddd, 1H, *J* = 18.5, 10.2, and 8.3 Hz), 5.00–4.90 (m, 3H), 4.86 (d, 1H, *J* = 6.9 Hz), 4.72 (d, 1H, *J* = 11.7 Hz), 4.58 (d, 1H, *J* = 11.7 Hz), 3.80–3.65 (m, 3H), 2.24 (m, 1H), 1.98 (m, 1H), 1.54 (m, 1H), 1.40–1.20 (m, 2H), 1.08 (s, 9H), 0.99 (d, 3H, *J* = 6.7 Hz), 0.98–0.92 (m, 1H), 0.91 (d, 3H, *J* = 6.9 Hz), 0.88 (d, 3H, *J* = 6.5 Hz), 0.91–0.88 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 144.3, 137.9, 135.5 (2), 135.4 (2), 133.4 (2), 129.5 (2), 128.2 (2), 127.7 (2), 127.6 (4), 127.4, 112.7, 94.8, 82.5, 69.4, 64.0, 43.2, 40.1, 35.6, 31.9, 27.6, 26.7, 21.7, 20.5, 19.1, 15.8; HRMS calcd. for $C_{36}H_{50}O_3SiNa$ (M + Na): 581.34269; found: 581.34427.

(1R,2*R*,4*R*,6*S*)-(1-Iodomethyl-2,4,6-trimethyl-oct-7-enyloxymethoxymethyl)-benzene (11). To a solution of **10** (770 mg, 1.38 mmol) in THF (5 ml) was added tetrabutylammonium fluoride (TBAF) (1 M in THF, 2.7 ml, 2 eq) at 0°C. The reaction mixture was stirred for 3 h and quenched with saturated NH_4Cl (3 ml). The solution was diluted with Et_2O (20 ml), the aqueous phase was separated, and the organic phase was washed with water (2 × 10 ml).The organic phase was dried over Na_2SO_4 and concentrated and dried *in vacuo* to give a crude alcohol that was used in the next step without additional purification.

To a stirred solution of the above alcohol were added dry imidazole (188 mg, 2 eq) and triphenylphosphine (723 mg, 2 eq) in anhydrous toluene (14 ml) at 0°C followed by iodine (700 mg, 2 eq). The resulting mixture was stirred for 5 min at 0°C, and then the temperature was raised to 25°C over 1 h. The reaction mixture was quenched by addition of saturated $Na_2S_2O_3$, and the organic phase was dried over Na_2SO_4. Concentration and purification by flash chromatography (95:05 hexanes/EtOAc) gave **11** as a colorless oil (553 mg, 93%). $[\alpha]_D$ +23.5 (*c* 1.15, $CHCl_3$); IR (film) 2,960, 2,926, 1,455, 1,040 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.40–7.28 (m, 5H), 5.60 (ddd, 1H, *J* = 18.5, 10.2, and 8.3 Hz), 5.02–4.92 (m, 2H), 4.89 (d, 1H, *J* = 7.2 Hz), 4.85 (d, 1H, *J* = 7.2 Hz), 4.76 (d, 1H, *J* = 11.7 Hz), 4.68 (d, 1H, *J* = 11.7 Hz), 3.45–3.35 (m, 2H), 3.32–3.28 (m, 1H), 2.26 (m, 1H), 2.00 (m, 1H), 1.54 (m, 1H), 1.42–1.28 (m, 2H), 1.07 (m, 1H), 1.00 (d, 3H, *J* = 6.7 Hz), 0.98–0.92 (m, 1H), 0.90 (d, 3H, *J* = 6.3 Hz), 0.88 (d, 3H, *J* = 6.7 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 144.3, 137.6, 128.3 (2), 127.8 (2), 127.6, 112.8, 94.5, 81.9, 70.0, 43.2, 40.3, 35.6, 34.1, 27.6, 21.6, 20.5, 14.8, 8.3.

(2*S*,3*R*,5*R*,7*S*)-2-(2-Benzyloxymethoxy-3,5,7-trimethyl-non-8-enyl)-2-isopropyl-[1,3]dithiane (12). To a solution of 2-isopropyl-[1,3]dithiane (432 mg, 2.2 eq) in anhydrous THF (13 ml) at –78°C were added hexamethylphosphoramide (460 μl, 2.2 eq/**11**) and *tert*-butyl lithium (1.7 M in pentane) until yellow color remained, and then more *tert*-butyl lithium (1.7 M in pentane, 1.9 ml, 1.2 eq/dithiane) was added. The resulting mixture was stirred for 1 h at –78°C before a solution of **11** (521 mg, 1.21 mmol) in anhydrous THF (12 ml) was added. Stirring was continued for 6 h at –78°C, then the mixture was quenched with saturated NH_4Cl and extracted with EtOAc (3 × 50 ml), dried over Na_2SO_4, and concentrated, and the residue was purified by flash

chromatography (97.5:2.5 hexanes/EtOAc) to give **12** as a colorless oil (351 mg, 75%). $[\alpha]_D$ +22.3 (*c* 0.7, $CHCl_3$); IR (film) 2,960, 1,455, 1,043 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.36–7.27 (m, 5H), 5.54 (ddd, 1H, *J* = 18.4, 10.2, and 8.2 Hz), 4.97 (d, 1H, *J* = 7.0 Hz), 4.95–4.85 (m, 2H), 4.84 (d, 1H, *J* = 7.0 Hz), 4.75 (d, 1H, *J* = 11.8 Hz), 4.56 (d, 1H, *J* = 11.8 Hz), 3.90 (m, 1H), 2.90–2.77 (m, 3H), 2.75–2.65 (m, 1H), 2.42 (qt-like, 1H, *J* = 6.8 Hz), 2.22 (m, 1H), 2.15 (m, 1H), 2.00–1.90 (m, 4H), 1.57 (m, 1H), 1.31–1.16 (m, 3H), 1.15 (d, 3H, *J* = 6.0 Hz), 1.13 (d, 3H, *J* = 5.7 Hz), 1.00–0.96 (m, 1H), 0.95 (d, 3H, *J* = 6.6 Hz), 0.94 (d, 3H, *J* = 6.8 Hz), 0.87 (d, 3H, *J* = 6.5 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 144.9, 138.6, 128.8 (2), 128.1 (2), 127.9, 113.2, 95.9, 81.0, 70.3, 58.9, 44.5, 42.1, 36.1, 35.2 (2), 34.0, 28.2, 26.3, 26.2, 25.6, 22.2, 20.5, 18.7, 18.3, 14.6; HRMS calcd. for $C_{27}H_{45}O_2S_2$ (M + 1): 465.28610; found: 465.28935.

(5*S*,6*R*,8*R*,10*S*)-5-Benzyloxymethoxy-2,6,8,10-tetramethyl-dodec-11-en-3-one (13). To a stirred solution of **12** (376 mg, 809 μmol) in 10 ml of acetone were successively added at 0°C aqueous $AgNO_3$ (8.9 ml of a 1 M solution) and *N*-chlorosuccinimide (216 mg, 2 eq). The resulting solution was stirred at 0°C for 10 min, quenched with saturated $Na_2S_2O_3$ (5 ml), and extracted with Et_2O (3 × 50 ml). The organic phase was dried over Na_2SO_4 and concentrated, and the residue was purified by flash chromatography (95:5 hexanes/EtOAc) to give **13** as a colorless oil (240 mg, 79%). $[\alpha]_D$ +21.2 (*c* 0.5, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.36–7.27 (m, 5H), 5.54 (ddd, 1H, *J* = 17.2, 8.3, and 7.0 Hz), 4.97–4.89 (m, 2H), 4.79 (d, 1H, *J* = 6.9 Hz), 4.76 (d, 1H, *J* = 6.9 Hz), 4.61 (d, 1H, *J* = 11.7 Hz), 4.54 (d, 1H, *J* = 11.7 Hz), 4.11 (m, 1H), 2.73 (dd, 1H, *J* = 16.4 and 9.0 Hz), 2.62 (qt-like, 1H, *J* = 6.9 Hz), 2.38 (dd, 1H, *J* = 16.4 and 3.1 Hz), 2.20 (m, 1H), 1.99 (m, 1H), 1.73 (m, 1H), 1.53 (m, 1H), 1.26–1.12 (m, 2H), 1.10 (d, 3H, *J* = 6.9 Hz), 1.09 (d, 3H, *J* = 6.9 Hz), 0.95 (d, 3H, *J* = 6.7 Hz), 0.91 (d, 3H, *J* = 6.8 Hz), 0.87 (d, 3H, *J* = 6.5 Hz), 0.95–0.87 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 213.6, 144.9, 138.4, 128.8 (2), 128.3 (2), 128.0, 113.2, 95.0, 78.3, 70.0, 44.1, 42.0, 41.6, 41.3, 36.1, 33.7, 28.0, 22.0, 20.8, 18.5, 18.3, 15.1; HRMS calcd. for $C_{24}H_{39}O_3$ (M + 1): 375.28993; found: 375.29167.

(3*S*,5*S*,6*R*,8*R*,10*S*)-5-Benzyloxymethoxy-2,6,8,10-tetramethyl-dodec-11-en-3-ol (14) and (3*R*,5*S*,6*R*,8*R*,10*S*)-5-Benzyloxymethoxy-2,6,8,10-tetramethyl-dodec-11-en-3-ol (14a). To a stirred solution of **13** (118 mg, 315 μmol) in anhydrous toluene (4.5 ml) at –40°C was added DIBAL-H (1.5 M in PhMe, 630 μl, 2 eq), and the resulting solution was stirred at this temperature for 2 h and then quenched with 5 ml of 1 M HCl at –40°C. The solution was diluted with 30 ml of EtOAc/hexanes (1:1), stirred at rt for 30 min, and extracted with EtOAc/hexanes (1:1, 2 × 30 ml). The combined organic layers were washed with $NaHCO_3$ solution, dried over Na_2SO_4, and concentrated *in vacuo*. The residue was purified by flash chromatography (85:15 hexanes/EtOAc) to give **14** (58 mg, 49%) and **14a** (55 mg, 47%) as colorless oils. For **14**: $[\alpha]_D$ –15 (*c* 0.1, $CHCl_3$); IR (film) 3,200 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.40–7.27 (m, 5H), 5.59 (ddd, 1H, *J* = 18.2, 10.0, and 8.4 Hz), 5.00–4.90 (m, 2H), 4.82 (s, 2H), 4.71 (d, 1H, *J* = 11.8 Hz), 4.63 (d, 1H, *J* = 11.8 Hz), 3.76 (m, 1H), 3.60 (m, 1H), 2.48 (brs, 1H), 2.22 (m, 1H), 1.93 (m, 1H), 1.68–1.48 (m, 3H), 1.42 (td, 1H, *J* = 9.7 and 2.3 Hz), 1.28–1.20 (m, 2H), 1.00–0.86 (m, 17H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 145.0, 138.0, 128.9 (2), 128.2 (3), 113.2,

95.2, 80.3, 73.1, 70.6, 44.1, 41.7, 36.1, 34.3, 34.0, 33.8, 28.0, 22.0, 20.9, 19.2, 18.3, 15.1; HRMS calcd. for $C_{24}H_{40}O_3Na$ (M + Na): 399.28752; found: 399.28591. For **14a**: $[\alpha]_D$ –11.7 (*c* 0.12, $CHCl_3$); IR (film) 3,200 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.37–7.27 (m, 5H), 5.57 (ddd, 1H, *J* = 17.5, 10.2, and 8.3 Hz), 5.00–4.88 (m, 2H), 4.89 (d, 1H, *J* = 6.9 Hz), 4.80 (d, 1H, *J* = 6.9 Hz), 4.70 (d, 1H, *J* = 11.7 Hz), 4.64 (d, 1H, *J* = 11.7 Hz), 3.79 (m, 1H), 3.56 (m, 1H), 3.37 (brs, 1H), 2.21 (m, 1H), 2.05 (m, 1H), 1.69 (m, 1H), 1.57–1.40 (m, 3H), 1.24 (m, 1H), 1.11 (m, 1H), 1.00 (m, 1H), 0.99–0.86 (m, 16H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 144.9, 138.4, 128.9 (2), 128.3 (2), 128.2, 113.2, 93.6, 82.8, 77.0, 70.5, 44.5, 41.8, 36.0, 34.1, 32.5, 32.4, 28.0, 21.9, 20.6, 18.8, 17.8, 14.5; HRMS calcd. for $C_{24}H_{40}O_3Na$ (M + Na): 399.28752; found: 399.28478.

(2*R*,1'*S*,3'*S*,4'*R*,6'*R*,8'*S*)-2-[(2-*tert*-Butoxycarbonylamino-acetyl)-methyl-amino]-3-(3-iodo-4-triisopropylsilanyloxy-phenyl)-propionic Acid 3'-Benzyloxymethoxy-1'-isopropyl-4',6',8'-trimethyl-dec-9'-enyl Ester (16). To a stirred solution of crude acid **15** (49) (647 mg, 5 eq) and **14** (74 mg, 196 μmol) in CH_2Cl_2 (33 ml) at –20°C were added 1,3-dicyclohexylcarbodiimide (253 mg, 1.2 eq) and dimethylallyl monophosphate (DMAP) (28 mg, 1.15 eq/**14**); the resulting mixture was stirred at –20°C for 2 days and then filtered through a short plug of silica gel and concentrated. The residue was purified by flash chromatography (85:15 hexanes/EtOAc) to give **16** (150 mg, 77%) as a colorless oil. $[\alpha]_D$ –25 (*c* 0.02, $CHCl_3$); IR (film) 2,947, 2,868, 1,744, 1,716, 1,660, 1,488, 1,286, 1,172 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.51 (d, 1H, *J* = 2.1 Hz), 7.36–7.27 (m, 5H), 6.92 (dd, 1H, *J* = 8.2 and 1.9 Hz), 6.72 (d, 1H, *J* = 8.2 Hz), 5.57 (ddd, 1H, *J* = 18.5, 10.1, and 8.3 Hz), 5.45 (m, 1H), 5.12 (m, 1H), 5.00–4.90 (m, 2H), 4.76 (s, AB, 2H), 4.64 (AB, 2H, *J* = 11.7 Hz), 3.90 (dd, 1H, *J* = 17.5 and 3.6 Hz), 3.76 (dd, 1H, *J* = 17.4 and 4.2 Hz), 3.49 (m, 1H), 3.21 (dd, 1H, *J* = 14.2 and 5.3 Hz), 2.92 (m, 1H), 2.71 (s, 3H), 2.23 (m, 1H), 1.90 (m, 2H), 1.68 (t-like, 2H, *J* = 6.0 Hz), 1.44 (s, 9H), 1.45–1.40 (m, 2H), 1.36–1.29 (m, 4H), 1.13 (d, 18H, *J* = 7.4 Hz), 0.97 (d, 3H, *J* = 6.7 Hz), 0.92–0.80 (m, 14H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 169.5, 168.5, 156.0, 154.6, 144.8, 140.1, 138.3, 131.4, 129.8, 128.8 (2), 128.3 (2), 128.1, 118.5, 113.3, 93.8, 79.9, 79.2, 78.9, 70.2, 53.8, 44.2, 42.9, 40.9, 36.1, 33.5, 32.3, 31.9, 31.1, 30.1, 28.8, 28.2, 22.2, 20.8, 19.2, 18.5, 17.0, 15.8, 13.5; (*C*I could not be detected); HRMS calcd. for $C_{50}H_{82}IN_2O_8Si$ (M + 1): 993.48852; found: 993.48791.

(3*R*,9*S*,11*S*,13*R*,14*S*,16*S*)-14-Hydroxy-3-(3-iodo-4-triisopropylsilanyloxy-benzyl)-16-isopropyl-4,9,11,13-tetramethyl-1-oxa-4,7-diaza-cyclohexadecane-2,5,8-trione (17). To a stirred solution of **16** (78 mg, 78.5 μmol) in CH_2Cl_2/MeOH (8 ml, 1/1 vol.) at –78°C was bubbled O_3 for 10 min (until the appearance of a blue coloration). Argon was bubbled in the solution for 15 min, and triphenylphosphine (42 mg, 2 eq) was added at –78°C. The temperature was raised to 0°C over 1 h, and the solution was stirred at this temperature for 1 h. The solution was diluted with CH_2Cl_2 (20 ml) and washed successively with saturated NH_4Cl (5 ml) and brine (2 × 5 ml), and the organic phase was dried over Na_2SO_4 and concentrated *in vacuo*.

To a solution of the above crude aldehyde in MeCN (8.2 ml) was added at 10°C an aqueous solution of $NaH_2PO_4 \cdot H_2O$ (25 mg in 2.75 ml, 2.3 eq) and H_2O_2 (90 μl, 11 eq, 30% solution). Then, an aqueous solution of $NaClO_2$ (100 mg in 1.1 ml of water, 14 eq)

was added dropwise over 1 h at 10°C, and the resulting solution was stirred at this temperature for 1 h. The reaction mixture was quenched by addition of Na_2SO_3 (10 mg), the resulting solution was diluted with Et_2O (50 ml), and the organic phase was washed successively with 10% HCl (5 ml) and brine (5 ml) and dried over Na_2SO_4. Concentration under reduced pressure gave the crude acid that was used in the next step without additional purification.

The above crude acid was dissolved in CH_2Cl_2 (1.6 ml), and trifluoroacetic acid (1.6 ml) was added at 0°C. The temperature was raised to rt over 1 h, the solution was stirred for 2 h and then concentrated, codistilled three times with benzene, and dried under reduced pressure for 1 h. The above crude material was then dissolved in anhydrous CH_2Cl_2 (144 ml), and *N*,*N*-bis(2-oxo-3-oxazolidinyl)-phosphinic reagent (173 mg, 5 eq) and DMAP (86 mg, 9 eq) were added at 0°C. The temperature was allowed to rise to rt overnight, and the reaction mixture was quenched by addition of 1 M HCl (1 ml). The organic phase was washed with brine (20 ml) and dried over Na_2SO_4. Concentration and purification by flash chromatography (70:30 hexanes/EtOAc) gave **17** (30 mg, 49% overall) as a colorless oil. $[\alpha]_D$ –32 (*c* 0.5, $CHCl_3$); IR (film) 3,350, 1,727, 1,654, 1,485, 1,461, 1,284, 1,251, 1,039 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.60 (s, 1H), 7.02 (d, 1H, *J* = 8.3 Hz), 6.77 (d, 1H, *J* = 8.3 Hz), 6.20 (d, 1H, *J* = 8.4 Hz), 5.44 (dd, 1H, *J* = 12.3 and 4.3 Hz), 5.06 (dd, 1H, *J* = 11.8 and 4.8 Hz), 4.79 (dd, 1H, *J* = 16.9 and 8.9 Hz), 3.59 (brd, 1H, *J* = 11 Hz), 3.43 (dd, 1H, *J* = 15.6 and 4.3 Hz), 3.29 (d, 1H, *J* = 16.9 Hz), 2.94 (s, 3H), 2.90 (dd, 1H, *J* = 15.6 and 12.3 Hz), 2.43 (m, 1H), 2.03 (m, 1H), 1.87 (m, 1H), 1.57–1.40 (m, 2H), 1.37–1.20 (m, 5H), 1.20–1.00 (m, 24H, including *J* = 7.4 Hz), 1.00–0.88 (m, 9H), 0.86 (d, 3H, *J* = 6.7 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 178.1, 172.2, 172.0, 155.0, 139.6, 130.9, 129.2, 118.6, 90.8, 77.7, 66.1, 58.6, 45.4, 43.5, 40.1, 39.6, 34.7, 33.2, 32.8, 31.2, 30.8, 27.4, 19.3, 18.7, 18.5 (2), 18.1, 14.8, 13.5; HRMS calcd. for $C_{36}H_{61}IN_2O_6SiNa$ (M + Na): 795.32413; found: 795.32664.

(–)-Doliculide (1). To a solution of **17** (28.5 mg, 37 μmol) in THF (1.3 ml) was added TBAF (60 μl, 1.5 eq, 1 M in THF) at 0°C. The resulting solution was stirred for 10 min at this temperature. The reaction mixture was quenched by addition of saturated NH_4Cl (500 μl) and diluted with EtOAc (10 ml). The aqueous phase was washed with EtOAc (3 × 10 ml), and the organic phase was dried over Na_2SO_4. Concentration and purification by flash chromatography (35:65 hexanes/EtOAc) gave **1** (21.5 mg, 95%); recrystallization from CH_2Cl_2/hexane gave colorless needles. mp 171–174; $[\alpha]_D$ –25.1 (*c* 0.3, MeOH); IR (film) 3,390, 1,731, 1,649, 1,502, 1,460, 1,418, 1,283, 1,039 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ (ppm) 7.50 (d, 1H, *J* = 1.9 Hz), 7.06 (dd, 1H, *J* = 8.3 and 1.9 Hz), 6.85 (d, 1H, *J* = 8.3 Hz), 6.28 (d, 1H, *J* = 8.3 Hz), 5.49 (dd, 1H, *J* = 12.3 and 4.4 Hz), 5.07 (ddd, 1H, *J* = 11.6, 5.0, and 1.8 Hz), 4.80 (dd, 1H, *J* = 16.8 and 8.9 Hz), 3.59 (brd, 1H, *J* = 11.2 Hz), 3.45 (dd, 1H, *J* = 15.4 and 4.4 Hz), 3.25 (dd, 1H, *J* = 16.8 and 1.9 Hz), 2.96 (s, 3H), 2.88 (dd, 1H, *J* = 15.4 and 12.4 Hz), 2.58 (brs, 1H), 2.44 (m, 1H), 2.02 (m, 1H), 1.87 (m, 1H), 1.53 (t, 1H, *J* = 12.3 Hz), 1.44 (ddd, 1H, *J* = 13.9, 11.8, and 1.7 Hz), 1.33 (ddd, 1H, *J* = 13.9, 11.8, and 2.3 Hz), 1.19 (m, 1H), 1.13 (d, 3H, *J* = 6.6 Hz), 1.10–1.04 (m, 3H), 0.97 (d, 3H, *J* = 6.7 Hz), 0.95 (d, 6H, *J* = 7.0 Hz), 0.85 (d, 3H, *J* = 6.8 Hz); ^{13}C NMR (150 MHz, $CDCl_3$) δ (ppm) 177.8, 172.0, 171.5, 154.3, 138.1, 130.2, 129.5, 115.2, 85.4, 77.3, 65.8, 58.1, 44.9, 43.1, 39.7, 39.2, 34.3, 32.8, 32.3, 30.9, 30.1,

27.0, 18.8, 18.4, 18.1, 17.7, 14.4; HRMS calcd. for $C_{27}H_{42}IN_2O_6$ (M + 1): 617.20876; found: 617.20936.

(Triphenyl-λ^5-phosphanylidene)acetic Acid 1-Methylcyclopentyl (MCP) Ester. To a vigorously stirred solution of 1-methylcyclopentanol (ref. 2) (11.6 g, 116 mmol) and pyridine (10.6 ml, 131 mmol) in CH_2Cl_2 (200 ml) at 0°C was slowly added bromoacetyl bromide (11.4 ml, 131 mmol). The resulting solution was stirred at rt for 2 h and quenched by addition of saturated NH_4Cl (100 ml) and diluted with CH_2Cl_2 (100 ml). The aqueous phase was extracted with CH_2Cl_2 (3 × 50 ml), the organic phase was dried over Na_2SO_4 and concentrated to give a residue that was used without additional purification. To a solution of this product in C_6H_6 (200 ml) was added PPh_3 (32.7 g, 125 mmol), and the resulting solution was stirred for at rt 12 h, by which time a white solid precipitated out. The mixture was filtered, and the resulting solid was washed with toluene (100 ml) and EtOAc (100 ml) and dried *in vacuo* to give the phosphonium bromide salt as a white solid (41.2 g, 72%). To a stirred solution of the phosphonium bromide salt (41.2 g, 85.2 mmol) in H_2O (800 ml) was added KOH (5.5 g, 98.0 mmol, dissolved in 200 ml of H_2O) at 0°C. The resulting mixture was stirred at 0°C for 1 h and diluted with CH_2Cl_2 (500 ml). The aqueous phase was extracted with CH_2Cl_2 (3 × 200 ml), and the organic phase was dried over Na_2SO_4 and concentrated. Trituration of the resulting residue with Et_2O/pentane (1:3) gave the title compound (25 g, 70%) as a yellowish solid. mp 150–155; IR (film) 2,960, 1,437, 1,355, 1,182, 1,103 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ (ppm) 7.95–7.40 (m, 15H), 1.80–1.40 (m, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 133.4, 133.4, 133.3, 132.1, 129.0, 128.9, 39.8, 32.2, 30.9, 23.2.

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