Stereoselective Total Synthesis of *iso*-Cladospolide B

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Abstract: A simple and efficient stereoselective total synthesis of *iso*-cladospolide B and a formal total synthesis of cladospolide B, using Jacobsen's hydrolytic kinetic resolution, is described.

Key words: phytotoxic, Wadsworth–Emmons, oxidation, kinetic resolution, Mitsunobu

Marine fungi are emerging as a potential source of new pharmaceuticals and pharmaceutical leads.[1] Recently, *iso*-cladospolide B (**1**), obtained from the fermentation of fungal isolate I962S215, was also isolated from ethyl acetate extracts of cladosporium species and from red sea sponge *Niphates rowi*.[2] It closely resembles the natural product cladospolide B (**2**), a highly phytotoxic compound[3] produced from *Cladosporium tenuissimum*. Though there have been two earlier synthesis reported for *iso*-cladospolide B,[4] there is still need for a simple, short and efficient synthetic route for further biological studies. Our group has recently initiated an academic programme focused on the synthesis of several lactone-containing natural products[5] and their analogues for further evaluation of their biological properties. As part of this programme, herein we report the stereoselective total synthesis of *iso*-cladospolide B and the formal total synthesis of cladospolide B, with Jacobsen's kinetic resolution and a modified Wadsworth–Emmons reaction being the key steps.

Our retrosynthetic analysis for both *iso*-cladospolide B and cladospolide B (Figure 1) revealed a common key intermediate **3**, which could be prepared by a Wittig reaction of the ylide generated from intermediate **4** and aldehyde **5**. The intermediate **4** could be prepared from the chiral epoxide obtained from resolution of the racemic epoxide **6**.

The synthesis of intermediate **4** started with commercially available 5-hexen-1-ol (Scheme 1). Accordingly, 5-hexen-1-ol was protected as its benzyl ether **7** with sodium hydride and benzyl bromide, then epoxidized with *m*-chloroperoxybenzoic acid (MCPBA) to give the racemic oxirane **6**. The oxirane **6** was hydrolyzed employing (*S*,*S*)-Salen-Co-(OAc) catalyst[6] to give the chiral epoxide **8**. The epoxide was reduced with lithium aluminum hydride to generate the secondary alcohol **9**, which was then protected as its *tert*-butyldiphenylsilyl ether **10**. Debenzylation of **10** with carbon-supported palladium afforded

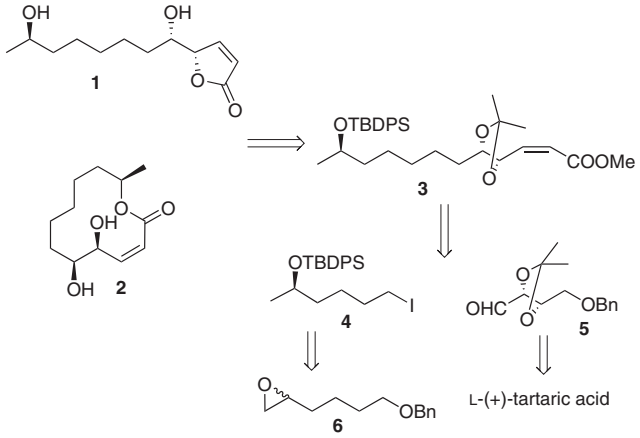

Figure 1

alcohol **11**. The alcohol was coupled with mercaptobenzothiazole under Mitsunobu conditions[7] to afford the thioether **12** which, on oxidation with MCPBA, resulted in sulphone **13**. Unfortunately, attempts at reacting **13** with the known aldehyde **5** under Julia conditions[8a] failed to give the expected alkene **16** (Scheme 2).[8b] We therefore converted the alcohol **11** into the corresponding phosphorane salt **15**, with the intention of using this as one of the components in a Wittig reaction with **5**. The conversion of **11** to **15** was accomplished using I_2, triphenylphosphine and imidazole,[9] followed by treatment of the resulting iodide **14** with triphenyl phosphine.

The other key fragment, aldehyde **5**, was prepared from L-(+)-diethyltartaric acid according to a known procedure.[10] The Wittig salt **15** was treated with *n*-butyllithium and reacted with aldehyde **5** to afford **16a**, which was then hydrogenated to yield the saturated alcohol **17** (Scheme 3). Swern oxidation of **17** gave the aldehyde **18** which was subjected to a modified Wadsworth–Emmons reaction, in the presence of sodium hydride in tetrahydrofuran, to provide the intermediate **3** exclusively.

The intermediate **3** upon one-pot desilylation, deacetonization and lactonization using 3% HCl in methanol afforded *iso*-cladospolide B $[\alpha]_D = -106.0$ (*c* 1.35, MeOH) {lit.[4b] $[\alpha]_D = -105.0$ (c 0.23, MeOH)}.[11]

The intermediate **3**, on hydrolysis with lithium hydroxide, was converted into acid **19** which has already been utilized for the synthesis of cladospolide B (Scheme 4).[4a]

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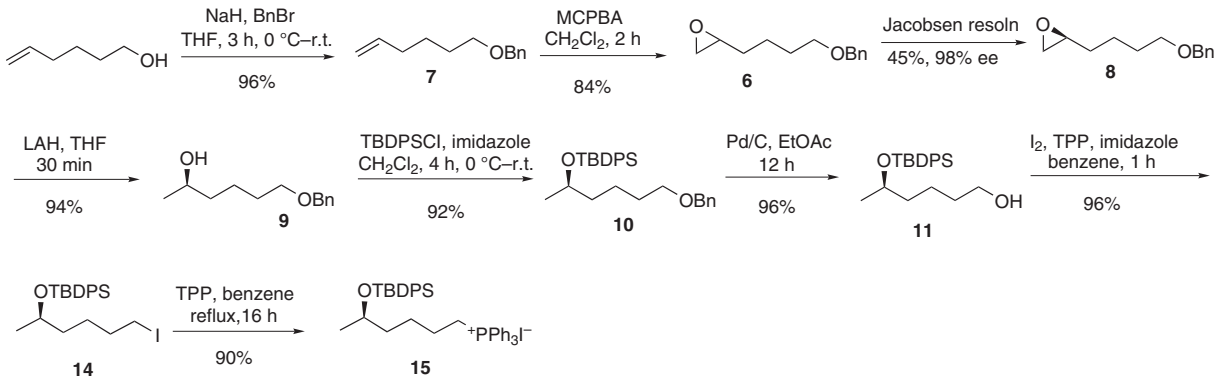
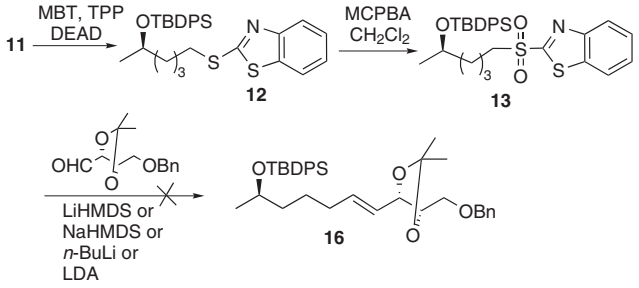
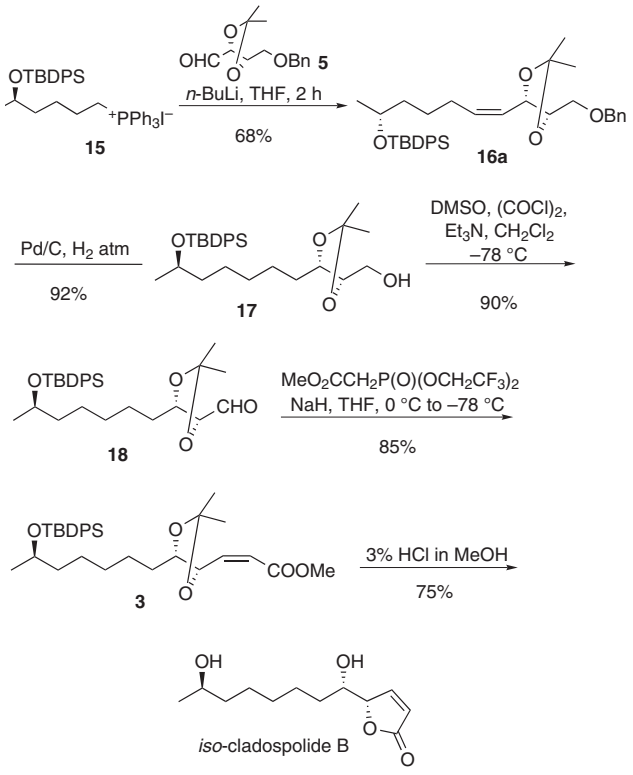


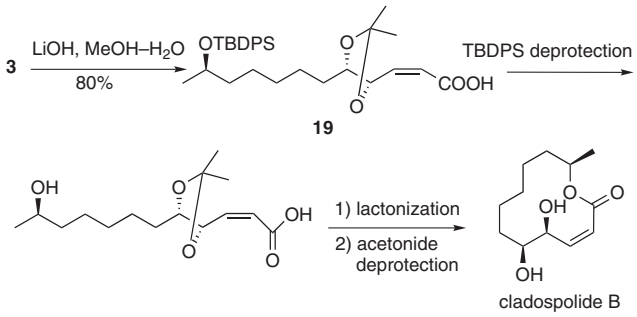
Scheme 1



Scheme 2



Scheme 3



Scheme 4

In conclusion, we have developed a simple and efficient route for total synthesis of *iso*-cladospolide B, and synthesized a common intermediate that can be further used for the total synthesis of cladospolide B. Furthermore, by altering Jacobsen's salen reagent, the other isomer can also be easily prepared. Further applications of this class of compounds for analogue synthesis is underway and will be published in future.

Column chromatography was performed using silica gel 60–120 mesh. All solvents were dried and distilled prior to use. IR spectra were recorded on a Perkin-Elmer Infrared spectrophotometer as KBr wafers, neat or in $CHCl_3$, as a thin film. ^1H and ^{13}C NMR were recorded on a Varian Gemini 200, Bruker Avance 300 or Varian Unity 400 instrument (^1H operating frequencies of 300 MHz and 400 MHz, respectively) using TMS as an internal standard. Mass spectra were recorded on Micro mass VG 7070H mass spectrometer for EI, VG Autospec mass spectrometer for FABMS and micromass Quatro LC triple quadrupole mass spectrometer for ESI analysis. The optical rotations were recorded on a JASCO DIP-360 digital polarimeter at 25 °C. Enantiomeric excess was analyzed using a chiralcel OB (H) 250 × 4.6 mm Daicel column; flow rate 1.0 mL/min; mobile phase 10% IPA in *n*-hexane; Machine Shimadzu LC 10 AT pump, and SPD-10A UV detection at 254 nm.

[(Hex-5-enyloxy)methyl] Benzene (7)

To a well-stirred suspension of freshly activated NaH (2.88 g, 120 mmol, 60% w/v dispersion in mineral oil) in anhydrous THF (50 mL), a solution of 5-hexen-1-ol (10 g, 100 mmol) in dry THF (60 mL) was added dropwise at 0 °C. After 30 min, benzyl bromide (20.52 g, 120 mmol) was added and the reaction mixture was brought to r.t. and stirred for 3 h. The reaction was quenched with

ice pieces and product was extracted with Et_2O (3 × 100 mL). The combined organic layer was washed with H_2O (100 mL), brine (2 × 50 mL) and dried over anhydrous Na_2SO_4. After filtering and removing the volatiles under reduced pressure, the crude benzyl ether was purified by column chromatography (EtOAc–hexane, 5%) to afford the pure product **7**.

Yield: 18.3 g (96%); colorless liquid.

IR (neat): 3069, 3031, 1639.7, 1495.4, 1453.8, 1361.9 1204.6, 1027.5, 994.8, 910.4, 735.8, 697.6, 612.5 cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$): δ = 7.34–7.20 (m, 5 H), 5.83–5.71 (m, 1 H), 5.0–4.87 (m, 2 H), 4.47 (s, 2 H), 3.43 (t, *J* = 6.2 Hz, 2 H), 2.06 (q, *J* = 7.2 Hz, 2 H), 1.67–1.55 (m, 2 H), 1.54–1.41 (m, 2 H).

^{13}C NMR (50 MHz, $CDCl_3$): δ = 138.5, 128.1, 127.4, 127.3, 72.7, 69.9, 51.9, 46.7, 32.0, 29.3, 22.5.

ESI-MS: *m*/*z* calcd for $C_{13}H_{18}O$ + H: 191; found: 191.

2-[4-(Benzyloxy)butyl]oxirane (6)

To a solution of olefin **7** (18.0 g, 94.0 mmol) dissolved in dry CH_2Cl_2 (150 mL) was added $NaHCO_3$ (23.78 g, 282 mmol), followed by MCPBA (24.39 g, 141.36 mmol) and stirred at r.t. for 2 h. The reaction mixture was diluted with H_2O (150 mL) and extracted with CH_2Cl_2 (3 × 100 mL). The combined organic layer was washed with brine (2 × 100 mL) and dried over anhydrous Na_2SO_4. The solvent was removed under reduced pressure and the crude product was purified by column chromatography (EtOAc–hexane, 20%) to afford product **6** (16.38 g, 84%) as a colorless oil.

(*S*)-2-[4-(Benzyloxy)butyl]oxirane (8)

A mixture of (*S*,*S*)-(–)-*N*,*N*′-bis(3,5-di-*tert*-butylsalicylidene)-1,2-cyclohexanediaminocobalt (143.0 mg, 0.24 mmol), toluene (1 mL) and AcOH (0.027 mL, 0.006 mmol) was stirred while open to the air for 1 h at r.t. The solvent was removed by rotary evaporator under reduced pressure and the brown residue was dried under high vacuum. The oxirane **6** (16.38 g, 79.5 mmol) was added in one portion, and the mixture was cooled in an ice–water bath. H_2O (0.78 mL, 43.7 mmol) was slowly added over 1 h, whilst keeping the temperature of the reaction mixture below 20 °C. The ice bath was removed and the reaction was stirred for 24 h at r.t.. The product **8** was isolated by column chromatography (EtOAc–hexane, 20%).

Yield: 7.4 g (45%); colorless liquid; $[\alpha]_D^{20}$ –5.12 (*c* 2.3, $CHCl_3$); 99% ee.

IR (neat): 2934, 2847.7, 1637.6, 1355.5, 1218.8, 1102.5, 771.9 cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$): δ = 7.33–7.19 (m, 5 H), 4.46 (s, 2 H), 3.44 (t, 6.0 Hz, 2 H), 2.87–2.79 (m, 1 H), 2.67 (dd, *J* = 3.8, 9.1 Hz, 1 H), 2.39 (dd, *J* = 2.2, 7.5 Hz, 1 H), 1.71–1.48 (m, 6 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 138.5, 128.1, 127.4, 127.3, 72.7, 69.9, 51.9, 46.7, 32.1, 29.4, 22.5.

ESI-MS: *m*/*z* calcd for $C_{13}H_{18}O_2$ + H: 207.3; found: 207.

(*R*)-6-(Benzyloxy)hexan-2-ol (9)

To a well stirred suspension of $LiAlH_4$ (930 mg, 26.57 mmol) in dry THF (15 mL), a solution of epoxide **8** (5.0 g, 24.15 mmol) in THF (30 mL) was added dropwise at 0 °C. The reaction mixture was brought to r.t. and stirred for 30 min. The reaction mixture was quenched with sat aq NH_4Cl (10 mL) at 0 °C. The reaction mixture was filtered and dried over anhydrous Na_2SO_4, filtered and concentrated. Purification by column chromatography (EtOAc–hexane, 20%) gave the product **9**.

Yield: 4.08 g (94%); colorless oil; $[\alpha]_D^{20}$ +9.5 (*c* 1.8, $CHCl_3$).

IR (neat): 3417.0, 2931.2, 2847.7, 1618., 1354.0, 1219.0, 1108.0, 772.2, 702.0, 612.0 cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$): δ = 7.34–7.20 (m, 5 H), 4.46 (s, 2 H), 3.82–3.70 (m, 1 H), 3.44 (t, *J* = 6.0 Hz, 2 H), 1.68–1.30 (m, 6 H), 1.16 (d, *J* = 6.0 Hz, 3 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 138.6, 128.3, 127.6, 127.5, 72.8, 70.3, 67.8, 39.0, 29.6, 23.4, 22.4.

ESI-MS: *m*/*z* calcd for $C_{12}H_{20}O_2$ + H: 209.30; found: 209.

(*R*)-[6-(Benzyloxy)hexan-2-yloxy]-*tert*-butyldiphenylsilane (10)

To a stirred solution of alcohol **9** (3.80 g, 18.18 mmol) in dry CH_2Cl_2 (50 mL) and imidazole (1.85 g, 27.27 mmol) at 0 °C was added TBDPSCl (5.99 g, 21.81 mmol), dropwise and the reaction was stirred at r.t. for 4 h. The reaction mixture was quenched with H_2O (50 mL) and extracted with CH_2Cl_2 (3 × 40 mL). The combined organic layer was washed with brine (2 × 40 mL) and dried over anhydrous Na_2SO_4. The solvent was concentrated under reduced pressure and the residue was purified by column chromatography (EtOAc–hexane, 10%) to give **10**.

Yield: 7.5 g (92%); colorless liquid; $[\alpha]_D^{20}$ +11.74 (*c*, 2.1, $CHCl_3$).

IR (neat): 2930.7, 1618, 1354.8, 1219.2, 1109.6, 772.4, 701.0, 612.2 cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$): δ = 7.67–7.60 (m, 4 H), 7.41–7.19 (m, 11 H), 4.42 (s, 2 H), 3.87–3.74 (m, 1 H), 3.34 (t, *J* = 6.0 Hz, 2 H), 1.54–1.22 (m, 6 H), 1.07–1.09 (m, 12 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 135.8, 129.3, 128.2, 127.3, 72.7, 70.3, 69.5, 39.2, 29.7, 27.0, 23.1, 21.8, 19.2.

FAB-MS: *m*/*z* calcd for $C_{29}H_{38}O_2Si$ + H: 447.7; found: 448.

(*R*)-5-(*tert*-Butyldiphenylsilyloxy)hexan-1-ol (11)

A solution of benzyl compound **10** (7.0 g, 15.6 mmol) in EtOAc (45 mL) was mixed with Pd/C (10% mol) and stirred for 12 h under 55 psi hydrogen atmosphere. The catalyst was removed by filtration and the solvent was evaporated to give a residue that was purified by column chromatography (EtOAc–hexane, 35%) to afford the pure **11**.

Yield: 5.37 g (96%); light yellow viscous oil; $[\alpha]_D^{20}$ +8.25 (*c* 1.1, $CHCl_3$).

IR (neat): 3417.3, 2930.1, 1618.3, 1354.1, 1219.0, 1109, 772.1, 702.5, 612.1 cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$): δ = 7.67–7.61 (m, 4 H), 7.41–7.22 (m, 6 H), 3.89–3.75 (m, 1 H), 3.49 (d, *J* = 6.2 Hz, 2 H), 1.51–1.22 (m, 6 H), 1.08–1.01 (m, 12 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 129.5, 128.5, 127.4, 126.7, 79.3, 53.0, 52.3, 49.6, 38.5, 37.7, 28.37.

FAB-MS: *m*/*z* calcd for $C_{22}H_{32}O_2Si$ + H: 356.59; found: 357.

(*R*)-*tert*-Butyl(6-iodohexan-2-yloxy)diphenylsilane (14)

To a solution of alcohol **13** (5.0 g, 14.04 mmol) in dry benzene (50 mL) was added imidazole (1.91 g, 28.28 mmol), TPP (4.95 g, 18.9 mmol) and iodine (7.13 g, 28.08 mmol) at 0 °C. After 5 min, the cooling bath was removed and the reaction mixture was stirred for 1 h at r.t., during which time the color changed from brown to bright yellow and the mixture became highly viscous. The reaction was quenched with sat. aq $Na_2S_2O_3$ (30 mL) and extracted with Et_2O (3 × 60 mL). The combined organic layer was washed with sat. $Na_2S_2O_3$ solution (40 mL), brine (2 × 40 mL) and dried over anhydrous Na_2SO_4. The solvent was removed on a rotavapor and the crude product was purified by flash chromatography (EtOAc–hexane, 5%) to give the iodo compond **14** (6.34 g, 96%) as a light yellow oil. This was refluxed with triphenylphosphine (1 equiv) in benzene for 16 h then the solvent was filtered off and the solid residue was washed with petroleum ether. The resulting salt **15** was used without further purification.

{(*R*)-(*Z*)-7-[(4*S*,5*S*)-5-(Benzyloxymethyl)-2,2-dimethyl-1,3-dioxolan-4-yl]hept-6-en-2-yloxy}-9-*tert*-butyldiphenylsilane (16a)

A suspension of salt **15** (7.36 g 12.7 mmol) in dry THF (60 mL) was treated with *n*-BuLi (1.6 M solution in hexane, 7.28 mL, 12.76 mmol) at 0 °C and stirred at r.t. for 1 h. The aldehyde **5** (2.65 g, 10.6 mmol) in THF (30 mL) was added at 0 °C and, after 2 h, the reaction mixture was quenched with sat aq NH_4Cl (5 mL) and extracted with EtOAc (3 × 60 mL). The combined organic layer was washed with brine (2 × 30 mL) and dried over anhydrous $NaSO_4$. Volatiles were removed under reduced pressure and the crude product was purified by column chromatography (EtOAc–hexane, 35%) to furnish the product **16a**.

Yield: 4.95 g (68%); colorless oil; $[\alpha]_D^{20}$ +15.1 (*c* 1.3, $CHCl_3$).

IR (neat): 2932.7, 1374.1, 1219.0, 1106.9, 771.5, 702.1, 610.7, 509.3 cm^{-1}.

^1H NMR (200 MHz): δ = 7.67–7.60 (m, 4 H), 7.41–7.16 (m, 11 H), 5.57–5.46 (m, 1 H), 5.32 (t, *J* = 9.5 Hz, 1 H), 4.58–4.46 (m, 2 H), 3.82–3.71 (m, 2 H), 3.55–3.41 (m, 2 H), 2.10–1.79 (m, 2 H), 1.39 (m, 6 H), 1.38–1.23 (m, 4 H), 1.07–0.99 (m, 12 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 135.9, 135.8, 129.4, 129.3, 128.2, 127.5, 127.4, 127.3, 109.3, 80.5, 73.5, 69.3, 38.8, 27.6, 27.1, 27.0, 24.0, 23.1, 19.2.

FAB-MS: *m*/*z* calcd for $C_{36}H_{48}O_4Si$ + H: 572.87; found: 573.

(4*S*,5*S*)-5-[(*R*)-6-(*tert*-Butyldiphenylsilyloxy)heptyl]-2,2-dimethyl-1,3-dioxolan-4-yl}methanol (17)

A solution of benzyl compound **16a** (4.5 g, 7.86 mmol) in EtOAc (40 mL) was mixed with Pd/C (10 mol%) and stirred for 24 h at r.t under H_2 atmosphere. The catalyst was removed by filtration through a celite pad and the solvent was evaporated. The residue was purified by column chromatography (EtOAc–hexane, 35%) to give **17**.

Yield: 3.49 g (92%); colorless liquid.

IR (neat): 3451.4, 1637.3, 1352.9, 1219.6, 1109.9, 772.5 cm^{-1}.

^1H NMR (300 MHz, $CDCl_3$): δ = 7.67–7.60 (m, 4 H), 7.42–7.28 (m, 6 H), 3.86–3.68 (m, 3 H), 3.67–3.58 (m, 1 H), 3.56–3.45 (m, 1 H), 1.56 (br s, 1 H), 1.56–1.13 (m, 16 H), 1.07–1.01 (m, 12 H).

^{13}C NMR (100 MHz, $CDCl_3$): δ = 135.8, 134.8, 134.5, 129.36, 129.31, 127.38, 127.31, 108.5, 81.4, 76.8, 69.4, 61.9, 39.2, 32.9, 29.6, 27.3, 26.9, 25.9, 25.0, 23.2, 19.2.

FAB-MS: *m*/*z* calcd for $C_{29}H_{44}O_4Si$ + H: 485.76; found 486.

(4*R*,5*S*)-5-[(*R*)-6-(*tert*-Butyldiphenyloxy)heptyl)-2,2-dimethyl-1,3-dioxolane-4-carbaldehyde (18)

To the solution of oxalylchloride (1.68 g, 1.16 mL) in dry CH_2Cl_2 (20 mL) at –78 °C, was added DMSO (2.06 g, 1.88 mL) slowly via syringe and the reaction was stirred for 10 min. The alcohol **17** (3.2 g, 6.6 mmol) in dry CH_2Cl_2 (25 mL) was then added dropwise at –78 °C. After 2 h, Et_3N (5.5 mL, 39.6 mmol) was added slowly and the reaction mixture was allowed to come to 0 °C over 30 min. The reaction mixture was diluted with H_2O (50 mL) and extracted with CH_2Cl_2 (3 × 30 mL). The combined organic layer was washed with brine (2 × 30 mL) and dried over anhydrous Na_2SO_4. Evaporation of the solvent gave the crude aldehyde **18** (3.0 g) which was used directly for the next step.

(*Z*)-Methyl-3-{(4*S*,5*S*)-5-[(*R*)-6-(*tert*-butyldiphenylsilyloxy)hept-1-enyl]-2,2-dimethyl-1,3-dioxolan-4-yl}acrylate (3)

To solution of bis(2,2,2-trifluoroethyl)(methoxycarbonylmethyl)phosphonate (1.45 mL, 6.84 mmol) in dry THF (10 mL) was added NaH (74 mg, 60% dispersion in mineral oil) at 0 °C, vigorous gas evolution was observed. To the resulting clear solution, after 45 min, was added aldehyde **5** (3.0 g, 6.22 mmol) in dry THF (25 mL) dropwise at –78 °C. After 30 min, the reaction mixture was diluted with H_2O (30 mL) and extracted with EtOAc (3 × 30 mL). The combined organic layer was washed with H_2O (2 × 30 mL), brine (2 × 30 mL) and dried over anhydrous Na_2SO_4. Solvent was removed and the residue was chromatographed (EtOAc–hexane, 50%) to furnish the product **3**.

Yield: 2.53 g (84.5%); clear, colorless liquid; $[\alpha]_D^{20}$ +35.18 (*c* 1.5, $CHCl_3$).

IR (neat): 3416.2, 2932.5, 1729.0, 1618.2, 1376.1, 1243.1, 1049.8, 912.8, 794.7, 612.1 cm^{-1}.

^1H NMR (200 MHz, $CDCl_3$): δ = 7.72–7.56 (m, 4 H), 7.43–7.26 (m, 6 H), 6.11 (dd, *J* = 11.7, 8.6 Hz, 1 H), 5.89 (d, *J* = 12.5 Hz, 1 H), 5.20 (t, *J* = 8.6 Hz, 1 H), 3.88–3.73 (m, 1 H), 3.70 (s, 3 H), 3.67–3.55 (m, 1 H), 1.63–1.11 (m, 16 H), 1.08–0.98 (m,12 H).

^{13}C NMR (50 MHz, $CDCl_3$): δ = 165.7, 135.8, 134.6, 129.3, 127.3, 122.5, 109.2, 81.0, 76.1, 69.5, 51.4, 39.4, 31.9, 29.6, 27.3, 27.0, 25.9, 25.0, 23.1, 19.2.

FAB-MS: *m*/*z* calcd for $C_{32}H_{46}O_5Si$ + H: 539.77; found: 540.

(*Z*)-3-{(4*S*,5*S*)-5-[(*R*)-6-(*tert*-Butyldiphenylsilyloxy)heptyl]-2,2-dimethyl[1,3]dioxolan-4-yl}acrylic Acid (19)

To the ester **3** (2.3 g, 4.27 mmol) dissolved in MeOH (15 mL) and H_2O (10 mL) was added $LiOH{\cdot}H_2O$ (0.51 g, 21.3 mmol) and stirred at r.t. for 24 h. The reaction mixture was further diluted with H_2O (10 mL) and stirred for 30 min then concentrated by rotary evaporator to quarter of its volume. The mixture was acidified with 1 M HCl (pH 3) and the reaction mixture was extracted with EtOAc (3 × 30 mL). The combined organic layer was washed with brine (2 × 30 mL) and dried over anhydrous Na_2SO_4, filtered, evaporated and the crude product was purified by column chromatography (EtOAc–hexane, 80%) to yield pure **19**.

Yield: 2.04 g (91%); colorless liquid; $[\alpha]_D^{20}$ +40.14 (*c* 1.12, $CHCl_3$).

IR (neat): 3416.6, 1771.0, 1618.1, 1380.4, 1245.8, 1054.4, 913.4, 795.0, 744.1, 613.3 cm^{-1}.

^1H NMR (200 MHz, $CDCl_3$): δ = 7.69–7.58 (m, 4 H), 7.39–7.24 (m, 6 H), 6.22 (dd, *J* = 11.7, 8.6 Hz, 1 H), 5.89 (d, *J* = 11.7 Hz, 1 H), 5.17 (t, *J* = 8.6 Hz, 1 H), 3.86–3.73 (m, 1 H), 3.70–3.55(m, 1 H), 1.61–1.10 (m, 16 H), 1.09–0.96 (m, 12 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 169.9, 148.1, 135.8, 129.4, 129.3, 127.4, 127.3, 122.1, 109.4, 81.0, 76.1, 69.1, 39.3, 31.8, 29.6, 27.3, 27.0, 25.8, 25.1, 23.1, 19.2, 14.1.

FAB-MS: *m*/*z* calcd for $C_{31}H_{44}O_5Si$ + H: 525.78; found: 526.

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