

On a Practical Synthesis of β -Hydroxy Fatty Acid Derivatives

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Abstract: An efficient synthesis of three homochiral β -hydroxy fatty acid derivatives, which have been utilized in our total synthesis of lipid A, is reported. The synthesis features Sharpless asymmetric dihydroxylation of an unsaturated ester, regioselective conversion of a diol into acyloxy chlorides, and a reductive removal of the chloro group.

Lipid A is one of the important lipophilic components of lipopolysaccharides in the cell surface of Gram-negative bacteria.¹ Lipid A contains several types of tetradecanoyl esters such as β -hydroxy-, β -dodecanoyloxy-, β -tetradecanoyloxy-, and β -hexadecanoyloxy-derivatives.^{1,2} Toward the synthesis of lipid A, there have been some reports concerning the preparation of these fatty acid derivatives: our^{3a} and Noyori's^{3b} asymmetric reductions, optical resolution,^{3c} Brown's asymmetric allylboration,^{3d} and the enzymatic approach.^{3e} A more practical preparative method for these β -substituted fatty acid derivatives was, however, strongly desired for further synthetic study of lipid A and related compounds. In this article, we describe highly enantioselective syntheses of three tetradecanoic acid derivatives **2**, **3**, and **4** via a Sharpless asymmetric dihydroxylation. As shown in Fig. 1, these components have been utilized in our previous total synthesis of lipid A **1** from *Escherichia coli*.⁴

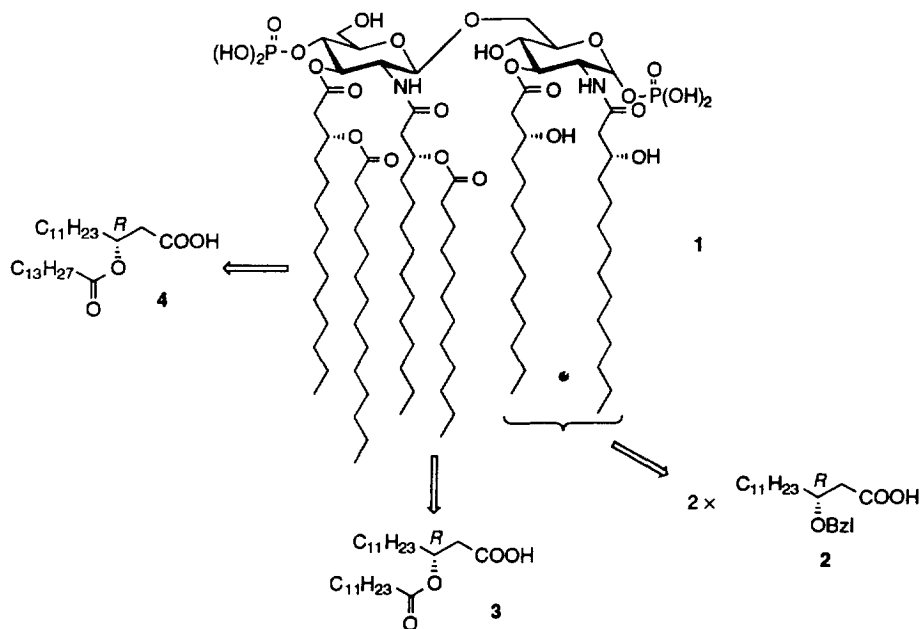
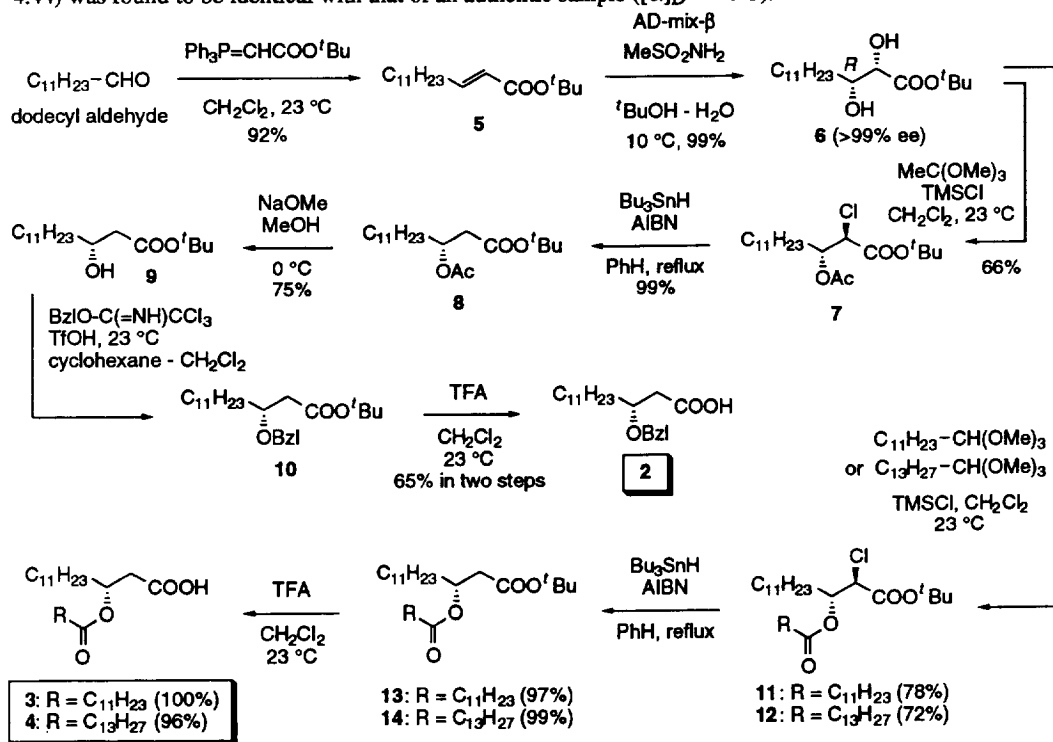


Fig. 1. Lipid A 1 in *Escherichia coli* lipopolysaccharide and the tetradecanoic acid components.⁴

The present syntheses were performed using a common intermediate, diol **6** (Scheme 1). Starting with dodecyl aldehyde, α,β -unsaturated ester **5** was obtained by a Wittig reaction in 92% yield ($\text{Ph}_3\text{P}=\text{CHCO}_2^t\text{Bu}$, CH_2Cl_2). Asymmetric dihydroxylation⁵ employing AD-mix- β and methanesulfonamide at 10 °C afforded diol **6** in 99% yield. By AD-mix- α and methanesulfonamide at 10 °C, *ent*-**6** (structure not shown) was provided in 73% yield. The enantiomeric excesses of diols **6** and *ent*-**6** were determined to be >99% ee and 97.8% ee, respectively, as judged by ¹H-NMR spectra of the corresponding Mosher's (*S*)- α -methoxy- α -(trifluoromethyl)phenylacetic acid (MTPA) esters.⁶ For the synthesis of benzyloxytetradecanoic acid **2**, diol **6** was subsequently treated with trimethyl orthoacetate in the presence of chlorotrimethylsilane (TMSCl).⁷ In this reaction, completely regioselective $\text{S}_{\text{N}}2$ -type substitution was realized at the C₂ position, which was electronically activated by the carbonyl functionality as reported by Sharpless *et al.*,⁸ to furnish **7** in 66% yield, while 23% of unreacted **6** was recovered. Reductive removal of the chloro group in **7** was smoothly achieved by tri-*n*-butyltin hydride in the presence of a catalytic amount of α,α' -azobisisobutyronitrile (AIBN) in 99% yield, meanwhile hydrogenolysis conditions (H_2 , Pd-C, NaOAc)⁸ provided only an over-reduction product, *tert*-butyl tetradecanoate, in quantitative yield. The acetyl group was then deprotected by sodium methoxide in methanol giving rise to alcohol **9** in 75% yield. Because transesterification of **9** gradually competed as deacetylation proceeded, the reaction had to be quenched before completion, 17% of the starting material **8** being recovered. Next, benzylation of **9** under acidic conditions (benzyl 2,2,2-trichloroacetimidate, trifluoromethanesulfonic acid)⁹ followed by deprotection of the *tert*-butyl ester (trifluoroacetic acid, CH_2Cl_2) gave (*R*)-3-benzyloxytetradecanoic acid **2** in 65% overall yield from **9**. The specific rotation of **2** ($[\alpha]_{\text{D}} = -4.44$) was found to be identical with that of an authentic sample ($[\alpha]_{\text{D}} = -4.43$).¹⁰



Scheme 1

Only five steps were totally needed for the synthesis of **3** and **4** using a pathway similar to that described for **2**. Treatment of diol **6** with trimethyl orthododecanoate¹² or trimethyl orthotetradecanoate¹² in the presence of TMSCl afforded **11** or **12** in 78% and 72% yield, respectively. These reactions were also highly regioselective as that for **7**. After **11** and **12** were converted to their dechlorinated products **13** and **14** individually, *tert*-butyl groups for carboxyl protections were removed by TFA in CH₂Cl₂ providing **3** ($[\alpha]_D = -1.03$) and **4** ($[\alpha]_D = -1.06$) in 97% and 95% overall yield, respectively. The specific rotation of **4** was in good accord with that of an authentic sample ($[\alpha]_D = -1.04$).¹⁰

As presented herein, the sequence of highly enantioselective dihydroxylation, regioselective conversion of the resulting diol into acyloxy chloride, and a reductive removal of the chloro group are quite practical for the synthesis of homochiral β -hydroxy fatty acid derivatives. This pathway will apply not only for the syntheses of the components of lipid A analogues but also for the synthesis of more complicated bioactive natural products.

EXPERIMENTAL

General Procedures. Melting points were determined with a Yamato Melting Point Apparatus Model MP-21 and are uncorrected. Optical rotations were measured with a Perkin-Elmer 241 polarimeter. ¹H-NMR spectra were measured in CDCl₃ at 270 MHz with a JEOL EX-270 spectrometer. Chemical shifts were referenced to residual CHCl₃ (7.26 ppm). Low and high resolution mass spectra were obtained on a JEOL JMS-SX102 mass spectrometer. Elemental analyses were performed by the staffs of our faculty. Silica-gel flash chromatography was performed on Kieselgel 60 Art.9385 (Merck) or Silica Gel 60 (nacalai tesque) using the indicated solvent system. Unless otherwise noted, nonaqueous reactions were carried out under nitrogen atmosphere. All commercially obtained materials were used as received.

***tert*-Butyl (*E*)-2-Tetradecenoate (**5**).** A solution of 24.6 g (0.113 mol) of 85% dodecyl aldehyde and 56.5 g (0.150 mol) of *tert*-butyl (triphenylphosphoranylidene)acetate in 750 mL of CH₂Cl₂ was stirred at ambient temperature for 12 h. The mixture was concentrated *in vacuo*, and to the residue was added 150 mL of Et₂O/hexane (1:1) to remove insoluble material by filtration. After the filtrate was concentrated, the residue was purified by silica-gel flash chromatography (Et₂O/hexane, 8:92 \rightarrow 15:85) to give 2.59 g (8%) of *tert*-butyl (*Z*)-2-tetradecenoate and 29.3 g (92%) of **5** as colorless oils. Data for **5**: EI-MS *m/z* 282 (M⁺), 209 (M⁺-^tBuO); ¹H-NMR δ 6.86 (dt, *J* = 15.5, 6.9 Hz, 1H, C₃-H), 5.73 (dt, *J* = 15.5, 1.7 Hz, 1H, C₂-H), 2.16 (dq, *J* = 1.7, 6.9 Hz, 2H, C₄-H₂), 1.54-1.37 (m, 2H, C₅-H₂), 1.48 (s, 9H, C(CH₃)₃), 1.37-1.20 (m, 16H, C₆-C₁₃-H₂), 0.88 (t, *J* = 6.6 Hz, 3H, C₁₄-H₃); Anal. calcd for C₁₈H₃₄O₂: C, 76.54; H, 12.13 found: C, 76.32; H, 12.22.

***tert*-Butyl (2*S*,3*R*)-2,3-Dihydroxytetradecanoate (**6**).** Potassium ferricyanide(III) (44.5 g, 0.135 mol), 18.7 g (0.135 mol) of K₂CO₃, 351 mg (0.451 mmol) of hydroquinidine 1,4-phthalazinediyl diether [(DHQD)₂PHAL], and 0.64 mL (0.090 mmol) of 0.14 M aqueous osmium tetroxide were stirred in 225 mL of 2-methyl-2-propanol/water (1:1) at ambient temperature for 15 min. To this was added 4.29 g (45.1 mmol) of methanesulfonamide and the mixture was cooled to 10 °C. Then 12.7 g (45.1 mmol) of **5** in 5 mL of 2-methyl-2-propanol/water (1:1) was introduced and the mixture was stirred at 10 °C for 14 h. The reaction was quenched by the addition of 67.6 g (0.536 mol) of Na₂SO₃ followed by stirring at ambient temperature for another 2 h. The mixture was subsequently extracted with EtOAc (350 mL, then 3 \times 100 mL). The combined organic extracts were washed with 100 mL of 2 M aqueous KOH, dried over anhydrous Na₂SO₄, filtered, and

concentrated *in vacuo*. Purification by silica-gel flash chromatography (EtOAc/hexane, 20:80) gave 14.1 g (99%) of diol **6** as colorless crystals: mp 46-46.5 °C; $[\alpha]_D^{21} = +6.16$ (*c* 1.51, CHCl₃); EI-MS *m/z* 260 (M⁺ - H - ^tBu), 242 (M⁺ - H₂O), 185 (M⁺ - ^tBu - H₂O); ¹H-NMR δ 3.96 (dd, *J* = 5.0, 2.3 Hz, 1H, C₂-H), 3.82 (m, 1H, C₃-H), 3.08 (d, *J* = 5.0 Hz, 1H, C₂-OH), 1.85 (d, *J* = 9.2 Hz, 1H, C₃-OH), 1.64-1.43 (m, 2H, C₄-H₂), 1.51 (s, 9H, C(CH₃)₃), 1.43-1.20 (m, 18H, C₅-C₁₃-H₂), 0.88 (t, *J* = 6.6 Hz, 3H, C₁₄-H₃); Anal. calcd for C₁₈H₃₆O₄: C, 68.31; H, 11.47. found: C, 68.20; H, 11.37.

tert-Butyl (2R,3S)-2,3-Dihydroxytetradecanoate (ent-6). An experimental procedure was followed as described for **6** using hydroquinine 1,4-phthalazinediyl diether [(DHQ)₂PHAL] instead of (DHQD)₂PHAL affording 73% yield of *ent-6* as colorless crystals: mp 46-47 °C; $[\alpha]_D^{19} = -6.14$ (*c* 3.50, CHCl₃).

tert-Butyl (2R,3R)-3-Acetoxy-2-chlorotetradecanoate (7). A solution of 14.1 g (44.5 mmol) of **6**, 8.49 mL (66.7 mmol) of trimethyl orthoacetate, and 8.47 mL (66.7 mmol) of chlorotrimethylsilane in 297 mL of CH₂Cl₂ was stirred at ambient temperature for 3 days. The reaction mixture was concentrated *in vacuo*, and the residue was purified by silica-gel flash chromatography (EtOAc/hexane, 5:95 → 20:80) to give 11.0 g (66%) of chloro acetate **7** and 3.23 g (23% recovery) of **6** as colorless oils. Data for **7**: $[\alpha]_D^{24} = +1.91$ (*c* 5.02, CHCl₃); EI-MS *m/z* 375 (M⁺ - H), 303 (M⁺ - ^tBuO); ¹H-NMR δ 5.27 (q, *J* = 6.2 Hz, 1H, C₃-H), 4.36 (d, *J* = 6.2 Hz, 1H, C₂-H), 2.07 (s, 3H, Ac), 1.71 (m, 2H, C₄-H₂), 1.48 (s, 9H, C(CH₃)₃), 1.37-1.20 (m, 18H, C₅-C₁₃-H₂), 0.88 (t, *J* = 6.5 Hz, 3H, C₁₄-H₃); Anal. calcd for C₂₀H₃₇O₄Cl: C, 63.73; H, 9.89. found: C, 63.55; H, 9.86.

tert-Butyl (R)-3-Acetoxytetradecanoate (8). A solution of 12.5 g (33.0 mmol) of **7**, 11.5 mL (42.8 mmol) of tri-*n*-butyltin hydride, and 271 mg (1.65 mmol) of α,α' -azobisisobutyronitrile in 165 mL of PhH was stirred under reflux for 6 h. The solvent was then removed *in vacuo*, and the residue was purified by silica-gel flash chromatography (EtOAc/hexane, 8:92) to give 11.2 g (99%) of product **8** as a colorless oil: $[\alpha]_D^{24} = +3.16$ (*c* 5.10, CHCl₃); EI-MS *m/z* 341 (M⁺ - H), 269 (M⁺ - ^tBuO); ¹H-NMR δ 5.19 (m, 1H, C₃-H), 2.50 (dd, *J* = 15.2, 7.1 Hz, 1H, C₂-H), 2.44 (dd, *J* = 15.2, 5.6 Hz, 1H, C₂-H), 2.03 (s, 3H, Ac), 1.63-1.20 (m, 20H, C₄-C₁₃-H₂), 1.43 (s, 9H, C(CH₃)₃), 0.88 (t, *J* = 6.6 Hz, 3H, C₁₄-H₃); Anal. calcd for C₂₀H₃₈O₄: C, 70.13; H, 11.18. found: C, 70.05; H, 11.14.

tert-Butyl (R)-3-Hydroxytetradecanoate (9). To a solution of 11.0 g (32.1 mmol) of **8** in 100 mL of methanol at 0 °C was added NaOMe in methanol (freshly prepared from 1.28 g (32.0 mmol) of 60% NaH and 60 mL of methanol at 0 °C). The mixture was stirred for 3 h and then concentrated *in vacuo* after addition of 2.76 mL (48.2 mmol) of acetic acid. Purification by silica-gel flash chromatography (Et₂O/hexane, 15:85) gave 1.86 g (17% recovery) of **8** and 7.24 g (75%) of alcohol **9** as colorless oils. Data for **9**: $[\alpha]_D^{23} = -14.6$ (*c* 2.03, CHCl₃); EI-MS *m/z* 300 (M⁺), 243 (M⁺ - ^tBu); ¹H-NMR δ 3.95 (m, 1H, C₃-H), 3.07 (d, *J* = 3.6 Hz, 1H, C₃-OH), 2.43 (dd, *J* = 16.2, 3.3 Hz, 1H, C₂-H), 2.31 (dd, *J* = 16.2, 8.9 Hz, 1H, C₂-H), 1.56-1.20 (m, 20H, C₄-C₁₃-H₂), 1.46 (s, 9H, C(CH₃)₃), 0.88 (t, *J* = 6.6 Hz, 3H, C₁₄-H₃); Anal. calcd for C₁₈H₃₆O₃: C, 71.95; H, 12.08. found: C, 71.64; H, 12.09.

tert-Butyl (R)-3-Benzoyloxytetradecanoate (10) and (R)-3-Benzoyloxytetradecanoic acid (2). To a solution of 1.204 g (4.01 mmol) of **9** and 2.02 g (8.00 mmol) of benzyl 2,2,2-trichloroacetimidate in 6 mL of cyclohexane and 3 mL of CH₂Cl₂ was added 0.06 mL of trifluoromethanesulfonic acid. The mixture was stirred at ambient temperature for 30 min, then quenched by addition of 10 mL of saturated aqueous solution of NaHCO₃ and extracted with Et₂O (3 × 15 mL). The combined organic extracts were dried over anhydrous MgSO₄, filtered, and concentrated *in vacuo*. Purification by silica-gel flash chromatography (Et₂O/hexane, 5:95 → 20:80) gave 1.71 g of crude **10**, 277 mg (16%) of benzyl (R)-3-benzoyloxytetradecanoate, and 219 mg (18% recovery) of **9** as colorless oils. Benzyl ether **10** thus obtained was contaminated by reagent-derived material, and was used without further purification. Selected data for **10**: EI-MS *m/z* 390 (M⁺), 333 (M⁺ -

^tBu); ¹H-NMR δ 7.38-7.25 (m, 5H, Ar-*H*₅), 4.57 (d, *J* = 11.2 Hz, 1H, Ar-*CH*), 4.51 (d, *J* = 11.2 Hz, 1H, Ar-*CH*), 3.86 (m, 1H, C₃-*H*), 2.54 (dd, *J* = 15.1, 7.3 Hz, 1H, C₂-*H*), 2.38 (dd, *J* = 15.1, 5.6 Hz, 1H, C₂-*H*), 1.60-1.20 (m, 20H, C₄-C₁₃-*H*₂), 1.45 (s, 9H, C(CH₃)₃), 0.89 (t, *J* = 6.7 Hz, 3H, C₁₄-*H*₃).

To a solution of above product **10** in 27 mL of CH₂Cl₂ at ambient temperature was added 3 mL of trifluoroacetic acid. After stirring for 22 h, the mixture was concentrated *in vacuo* and the residue was purified by silica-gel flash chromatography (Et₂O/PhH, 10:90) to give 872 mg (65%) of carboxylic acid **2** as a colorless oil: $[\alpha]_D^{26} = -4.44$ (*c* 1.62, CHCl₃); EI-MS *m/z* 334 (M⁺); EI-HR-MS calcd for C₂₁H₃₄O₃ (M⁺) *m/z* 334.2508, found 334.2487; ¹H-NMR (270MHz, CDCl₃) δ 7.40-7.25 (m, 5H, Ar-*H*₅), 4.60 (d, *J* = 11.9 Hz, 1H, Ar-*CH*), 4.56 (d, *J* = 11.9 Hz, 1H, Ar-*CH*), 3.88 (m, 1H, C₃-*H*), 2.65 (dd, *J* = 15.5, 6.7 Hz, 1H, C₂-*H*), 2.58 (dd, *J* = 15.5, 5.3 Hz, 1H, C₂-*H*), 1.75-1.49 (m, 2H, C₄-*H*₂), 1.42-1.17 (m, 18H, C₅-C₁₃-*H*₂), 0.88 (t, *J* = 6.7 Hz, 3H, C₁₄-*H*₃).

tert-Butyl (2R,3R)-2-Chloro-3-dodecanoyloxytetradecanoate (11). An experimental procedure was followed as described for **7** using trimethyl orthododecanoate¹² instead of trimethyl orthoacetate affording 78% yield of chloro dodecanoate **11** and 18% of **6** as colorless oils. Data for **11**: $[\alpha]_D^{21} = +3.88$ (*c* 1.03, CHCl₃); ¹H-NMR δ 5.27 (ddd, *J* = 8.3, 5.9, 3.6 Hz, 1H, C₃-*H*), 4.38 (d, *J* = 5.9 Hz, 1H, C₂-*H*), 2.31 (t, *J* = 7.5 Hz, 2H, C₂-*H*₂), 1.78-1.50 (m, 4H, C₄-*H*₂ and C₃-*H*₂), 1.48 (s, 9H, C(CH₃)₃), 1.40-1.18 (m, 34H, C₅-C₁₃-*H*₂ and C₄-C₁₁-*H*₂), 0.88 (t, *J* = 6.6 Hz, 6H, C₁₄-*H*₃ and C₁₂-*H*₃); Anal. calcd for C₃₀H₅₇O₄Cl: C, 69.67 H, 11.11. found: C, 69.52; H, 11.06.

tert-Butyl (2R,3R)-2-Chloro-3-tetradecanoyloxytetradecanoate (12). An experimental procedure was followed as described for **7** using trimethyl orthotetradecanoate¹² instead of trimethyl orthoacetate affording 72% yield of chloro tetradecanoate **12** and 22% of **6** as colorless oils. Data for **12**: $[\alpha]_D^{22} = +3.80$ (*c* 2.42, CHCl₃); EI-MS *m/z* 544 (M⁺), 471 (M⁺-^tBuO); ¹H-NMR δ 5.27 (ddd, *J* = 8.3, 6.3, 4.0 Hz, 1H, C₃-*H*), 4.38 (d, *J* = 6.3 Hz, 1H, C₂-*H*), 2.31 (t, *J* = 7.6 Hz, 2H, C₂-*H*₂), 1.80-1.52 (m, 4H, C₄-*H*₂ and C₃-*H*₂), 1.48 (s, 9H, C(CH₃)₃), 1.38-1.15 (m, 38H, C₅-C₁₃-*H*₂ and C₄-C₁₃-*H*₂), 0.88 (t, *J* = 6.6 Hz, 6H, C₁₄-*H*₃ and C₁₄-*H*₃); Anal. calcd for C₃₂H₆₁O₄Cl: C, 70.49 H, 11.28. found: C, 70.43; H, 11.36.

tert-Butyl (R)-3-Dodecanoyloxytetradecanoate (13). An experimental procedure was followed as described for **8** affording 97% yield of product **13** as a colorless oil: $[\alpha]_D^{23} = +3.04$ (*c* 1.02, CHCl₃); EI-MS *m/z* 482 (M⁺), 409 (M⁺-^tBuO); ¹H-NMR δ 5.20 (m, 1H, C₃-*H*), 2.50 (dd, *J* = 15.2, 7.3 Hz, 1H, C₂-*H*), 2.43 (dd, *J* = 15.2, 5.9 Hz, 1H, C₂-*H*), 2.26 (t, *J* = 7.6 Hz, 2H, C₂-*H*₂), 1.66-1.48 (m, 4H, C₄-*H*₂ and C₃-*H*₂), 1.43 (s, 9H, C(CH₃)₃), 1.34-1.17 (m, 34H, C₅-C₁₃-*H*₂ and C₄-C₁₁-*H*₂), 0.88 (t, *J* = 6.6 Hz, 6H, C₁₄-*H*₃ and C₁₂-*H*₃); Anal. calcd for C₃₀H₅₈O₄: C, 74.64; H, 12.11. found: C, 74.57; H, 12.09.

tert-Butyl (R)-3-Tetradecanoyloxytetradecanoate (14). An experimental procedure was followed as described for **8** affording 99% yield of product **14** as a colorless oil: $[\alpha]_D^{21} = +2.93$ (*c* 2.08, CHCl₃); EI-MS *m/z* 510 (M⁺), 437 (M⁺-^tBuO); ¹H-NMR δ 5.20 (m, 1H, C₃-*H*), 2.50 (dd, *J* = 15.2, 7.3 Hz, 1H, C₂-*H*), 2.43 (dd, *J* = 15.2, 5.9 Hz, 1H, C₂-*H*), 2.26 (t, *J* = 7.6 Hz, 2H, C₂-*H*₂), 1.68-1.47 (m, 4H, C₄-*H*₂ and C₃-*H*₂), 1.43 (s, 9H, C(CH₃)₃), 1.36-1.15 (m, 38H, C₅-C₁₃-*H*₂ and C₄-C₁₃-*H*₂), 0.88 (t, *J* = 6.8 Hz, 6H, C₁₄-*H*₃ and C₁₄-*H*₃); Anal. calcd for C₃₂H₆₂O₄: C, 75.24; H, 12.23. found: C, 75.09; H, 12.24.

(R)-3-Dodecanoyloxytetradecanoic acid (3). An experimental procedure was followed as described for **2** affording 100% yield of carboxylic acid **3** as a colorless oil: $[\alpha]_D^{16} = -1.03$ (*c* 1.17, CHCl₃); EI-MS *m/z* 426 (M⁺), 369 (M⁺-^tBu); ¹H-NMR δ 5.21 (m, 1H, C₃-*H*), 2.64 (dd, *J* = 15.8, 6.9 Hz, 1H, C₂-*H*), 2.57 (dd, *J* = 15.8, 5.9 Hz, 1H, C₂-*H*), 2.28 (t, *J* = 7.4 Hz, 2H, C₂-*H*₂), 1.68-1.54 (m, 4H, C₄-*H*₂ and C₃-*H*₂), 1.35-1.19 (m, 34H, C₅-C₁₃-*H*₂ and C₄-C₁₁-*H*₂), 0.88 (t, *J* = 6.6 Hz, 6H, C₁₄-*H*₃ and C₁₂-*H*₃); Anal. calcd for C₂₆H₅₀O₄: C, 73.19; H, 11.81. found: C, 72.99; H, 11.83.

(R)-3-Tetradecanoyloxytetradecanoic acid (4). An experimental procedure was followed as described for **2** affording 96% yield of carboxylic acid **4** as a white solid: mp 28-29 °C; $[\alpha]_D^{24} = -1.06$ (c 3.12, CHCl₃); EI-MS *m/z* 454 (M⁺), 426 (M⁺- CO), 410 (M⁺- CO₂); ¹H-NMR δ 6.56 (br, 1H, C₁-OH), 5.21 (m, 1H, C₃-H), 2.65 (dd, *J* = 15.8, 7.3 Hz, 1H, C₂-H), 2.57 (dd, *J* = 15.8, 5.6 Hz, 1H, C₂-H), 2.28 (t, *J* = 7.4 Hz, 2H, C₂-H₂), 1.68-1.52 (m, 4H, C₄-H₂ and C₃-H₂), 1.40-1.17 (m, 38H, C₅-C₁₃-H₂ and C₄-C₁₃-H₂), 0.88 (t, *J* = 6.6 Hz, 6H, C₁₄-H₃ and C₁₄-H₃); Anal. calcd for C₂₈H₅₄O₄: C, 73.96; H, 11.97. found: C, 73.62 H, 11.92.

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References and Notes

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