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Concise Total Synthesis of (–)-*cis*-Aerangis Lactone and (–)-*cis*-Cognac Lactone

Jhillu S. Yadav,*[a] Ragam Nageshwar Rao,[a] Begari Prem Kumar,[a] Ragam Somaiah,[a] Kontham Ravindar,[a] Basi V. Subba Reddy,[a] Ahamad Al Khazim Al Ghamdi[b]

[a] Organic Chemistry Division – I, Indian Institute of Chemical Technology, Hyderabad – 500 007, India
Fax +91(40)27160512; E-mail: yadavpub@iict.res.in

[b] Engineer Abdullah Bagshan for Bee Research, King Saudi University, Saudi Arabia

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Abstract: An efficient and concise stereoselective total synthesis of naturally occurring (–)-*cis*-aerangis lactone and (–)-*cis*-cognac lactone is described. The Sharpless asymmetric epoxidation of a primary allylic alcohol and TBSOTf-mediated intramolecular hydride transfer of a chiral epoxy alcohol have been successfully utilized for the construction of a key precursor with *syn*-aldol stereochemistry using a non-aldol pathway.

Key words: lactones, Wittig olefination, Sharpless asymmetric epoxidation, intramolecular hydride transfer, *syn*-aldol

δ-Lactones and γ-butyrolactones are important structural motifs in many biologically potent natural products.[1] They are versatile building blocks for the synthesis of various biologically interesting natural products. These lactone-derived molecules are present in Nature in particular as pheromones and aroma compounds of many fruits, flowers, and other natural products.[2] (–)-*cis*-Aerangis lactone [(4*S*,5*S*)-4-methyl-5-decanolide, **1**] was discovered by Kaiser in 1993 as the main odor component of the African moth orchids *Aerangis confusa* and *Aerangis kirkii* as a 1:1 mixture with its *trans*-diastereomer **2**.[3] Later, (–)-*cis*-aerangis lactone was found to be the sole stereoisomer in the scent of living white flowering orchids (*Aerangis confusa*). The fragrance of this natural product was typical for the lactonic odor of *A. confusa* and *A. kirkii* and was identical to the olfactory qualities of natural aerangis lactone. Its enantiomer (+)-*cis*-aerangis lactone was found to be reminiscent of δ-decalactone and its fragrance intensity was much lower than (–)-*cis*-aerangis lactone (**1**).[4]

(–)-*cis*-Cognac lactone [(4*S*,5*S*)-*cis*-4-methyl-5-pentyldihydrofuran-2(3*H*)-one, **3**] is one of the *Quercus* lactones which are known to present in various types of wood. These *Quercus* lactones are responsible for the sensory characteristics of wine and other alcoholic beverages such as whisky, brandy, and cognac; they are extracted during their ageing in oak barrels.[5] (–)-*cis*-Cognac lactone was found in both diastereomeric forms **3** and **4** in the literature.[6] Consequently, many research groups were actively involved in the synthesis of racemic and enantiomerically pure *cis*- and *trans*-aerangis lactones[7] and also *cis*- and *trans*-cognac lactones.[8] Most of these methods involve the use of either a chiral auxiliary or enzymatic hydrolysis, mainly Baker's yeast reduction of 3-methyl-4-oxononanoic acid or bioreduction of 3-methyl-4-oxononanoic acid to introduce the chirality (Figure 1).[9–11]

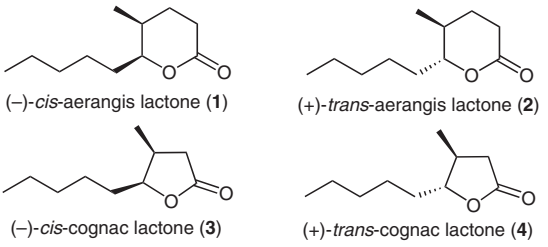
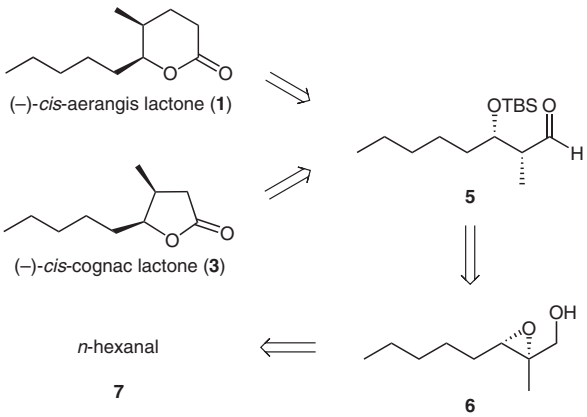


Figure 1 Aerangis and cognac lactones **1**–**4**

In continuation of our interest in the total synthesis of optically active lactones and lactone-containing natural products,[12] herein we wish to report a facile synthesis of (–)-*cis*-aerangis lactone (**1**) and (–)-*cis*-cognac lactone (**3**).



Scheme 1 Retrosynthetic analysis of (–)-*cis*-aerangis lactone and (–)-*cis*-cognac lactone

In our approach, we utilized a common chiral aldehyde precursor **5** for the construction of both the natural products (–)-*cis*-aerangis lactone (**1**) and (–)-*cis*-cognac lactone (**3**). The stereoselective construction of aldehyde **5** was achieved by silyl triflate mediated regioselective opening of epoxy alcohol **6**, which in turn can be easily accessed from commercially available and cost-effective *n*-hexanal (**7**). This synthetic strategy was successfully

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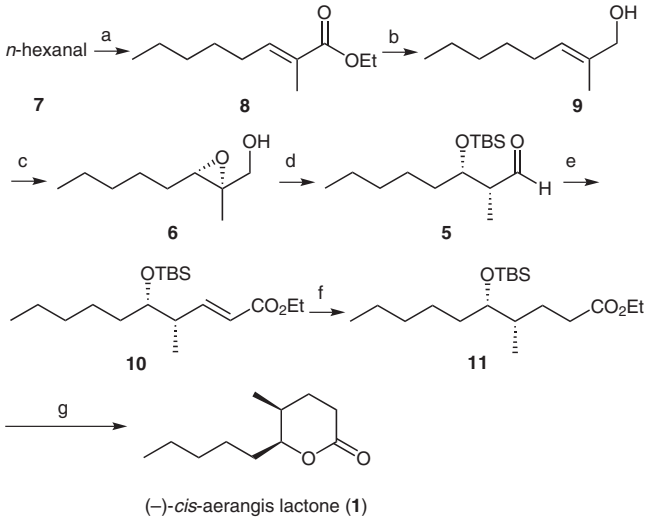
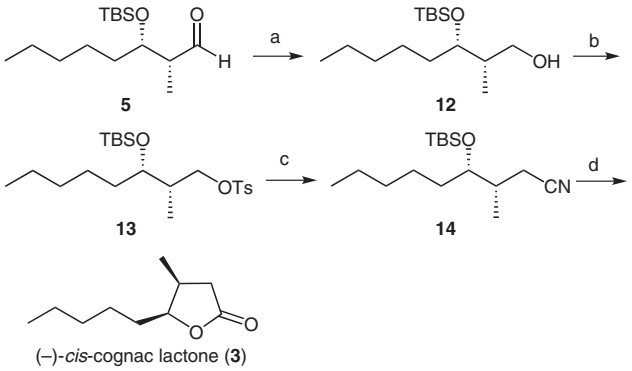
utilized by our group for the total synthesis of (–)-maurenone[13] and 5-*epi*-prelactone B (Scheme 1).[14]



Scheme 2 *Reagents and conditions*: (a) $Ph_3P{=}C(CH_3)CO_2Et$, CH_2Cl_2, 0–25 °C, 2 h, 85%; (b) DIBAL-H, CH_2Cl_2, 0 °C, 2 h, 90%; (c) *t*-BuOOH, (+)-DET, Ti(O*i*-Pr)$_4$, CH_2Cl_2, MS 4 Å, –25 °C, 90%; (d) TBSOTf, *i*-Pr_2NEt, CH_2Cl_2, MS 4 Å, –42 °C, 1 h, 85%; (e) $Ph_3P{=}CHCO_2Et$, CH_2Cl_2, r.t., 12 h, 80%; (f) H_2, Pd/C, EtOAc, 6 h, 95%; (g) AcOH, 1 M HCl, THF, 65 °C, 4 h, 65%.

Accordingly, *n*-hexanal (**7**) was converted into the corresponding α,β-unsaturated ester **8** using a stabilized Wittig ylide [$Ph_3P{=}C(CH_3)CO_2Et$] in 85% yield.[15] Selective reduction of α,β-unsaturated ester **8** using diisobutylaluminum hydride gave the allyl alcohol **9** in 90% yield. Sharpless asymmetric epoxidation of allyl alcohol **9** with *tert*-butyl hydroperoxide and (+)-diethyl tartrate in dichloromethane afforded the chiral epoxy alcohol **6** in 90% yield. Upon treatment of epoxy alcohol **6** with *tert*-butyldimethylsilyl triflate at –42 °C gave the TBS-protected *syn*-aldol product **5** (common aldehyde precursor) in 85% yield.[16] The aldehyde **5** was further treated with $Ph_3P{=}CHCO_2Et$ in dichloromethane to obtain the α,β-unsaturated ester **10** as a mixture of *cis*- and *trans*-isomers in 80% yield. The *cis*/*trans* mixture of ester **10** was then subjected to catalytic hydrogenation over 10% palladium on carbon to give the saturated ester **11** in 95% yield. Finally, acid-catalyzed (AcOH, 1 M HCl, THF) desilylation of **11** followed by concomitant lactonization gave the (–)-*cis*-aerangis lactone (**1**) in 65% yield.[17] The spectroscopic (^{1}H and ^{13}C NMR and IR) data and optical rotation of (–)-*cis*-aerangis lactone (**1**) were in good agreement with the literature (Scheme 2).[7]

The synthesis of (–)-*cis*-cognac lactone (**3**) commenced from the common aldehyde precursor **5**. Accordingly, aldehyde **5** was reduced to alcohol **12** using sodium borohydride in methanol in 92% yield.[18] This primary alcohol **12** was then treated with tosyl chloride in the presence of triethylamine and 4-(dimethylamino)pyridine in dichloromethane to afford the tosylate **13** in 90% yield. Tosylate **13** was converted into corresponding nitrile **14** using sodium cyanide in dimethyl sulfoxide in the presence of a catalytic amount of sodium iodide.[19] The crude nitrile **14** was used as such without further work-up in the next step. The subsequent base-induced hydrolysis of **14** followed by lactonization gave the target molecule (–)-*cis*-cognac lactone (**3**) in 70% yield. The spectroscopic (^{1}H and ^{13}C NMR and IR) data and optical rotation of (–)-*cis*-cognac lactone (**3**) were in good agreement with the literature (Scheme 3).[8]



Scheme 3 *Reagents and conditions*: (a) $NaBH_4$, MeOH, 0 °C, 2 h, 92%; (b) TsCl, Et_3N, DMAP, CH_2Cl_2, 0 °C, 4 h, 90%; (c) NaCN, NaI (cat.), DMSO, 60 °C, 82%; (d) 1. 2 M NaOH, EtOH, 100 °C, 8 h; 2. 10% HCl, THF, 10 °C, 12 h, 70% over 2 steps.

In conclusion, we have demonstrated a highly efficient and concise total synthesis of naturally occurring (–)-*cis*-aerangis lactone and (–)-*cis*-cognac lactone via the stereoselective construction of a key aldehyde precursor with *syn*-aldol stereochemistry using *tert*-butyldimethylsilyl triflate and Hünig's base mediated regioselective opening of epoxy alcohol involving intramolecular hydride transfer reaction. In contrast to previous reports, Sharpless asymmetric epoxidation was used for the introduction of chirality for the synthesis of both natural products. (–)-*cis*-Aerangis lactone was obtained in 29% overall yield in seven steps starting from *n*-hexanal and (–)-*cis*-cognac lactone was obtained in 34% overall yield in eight steps. The synthesis of remaining stereoisomers of aerangis lactone and cognac lactone are in progress.

Column chromatography was performed using silica gel 60–120 mesh. All the solvents were dried and distilled prior to use. IR spectra were recorded on a Perkin-Elmer Infrared spectrophotometer either neat or in $CHCl_3$ as a thin film. ^{1}H and ^{13}C NMR were recorded on a Varian Gemini 200MHZ and Bruker Avance 300 MHZ instruments 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 Jasco DIP-360 digital polarimeter.

Ethyl (*E*)-2-Methyloct-2-enoate (8)

To a soln of ethyl 2-(triphenylphosphoranylidene)propanoate (72.0 g, 200 mmol) in CH_2Cl_2 (300 mL) was added *n*-hexanal (**7**; 10.0 g, 100 mmol) at 0 °C and stirred at r.t. for 2 h. The solvent was removed under reduced pressure and the residue was purified by column chromatography (silica gel, 8% EtOAc–hexane) to afford **8** (15.64 g, 85%) as a colorless liquid.

IR (neat): 2931, 2864, 1721, 1244, 1094 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 6.70 (td, *J* = 7.3, 5.8 Hz, 1 H), 4.17 (q, *J* = 7.3 Hz, 2 H), 2.16 (q, *J* = 6.5 Hz, 2 H), 1.81 (s, 3 H), 1.53–1.23 (m, 9 H), 0.91 (t, *J* = 5.8 Hz, 3 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 168.1, 142.2, 127.5, 60.1, 31.6, 29.5, 29.4, 22.3, 14.1, 13.8, 12.1.

MS (EI): *m*/*z* = 185 $[M + H]^+$.

(*E*)-2-Methyloct-2-en-1-ol (9)

The ester **8** (15.64 g, 85 mmol) was dissolved in anhyd CH_2Cl_2 (200 mL) and then treated with 25% DIBAL-H in toluene (w/v) (96.52 mL, 170 mmol) in a dropwise fashion at 0 °C under an N_2 atmosphere. After stirring for 2 h at 0 °C, sat. aq potassium sodium tartrate (30 mL) was added and the mixture was extracted with Et_2O (3 × 150 mL). The combined organic layers were washed with brine, dried (anhyd Na_2SO_4), and concentrated under reduced pressure. The crude product was purified by column chromatography (silica gel, 10% EtOAc–hexane) to afford the **9** (10.86 g, 90%) as a colorless oil.

IR (neat): 3337, 2925, 2857, 1460, 1379, 1012 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 5.34 (td, *J* = 5.8, 7.3 Hz, 1 H), 3.92 (s, 2 H), 1.98 (m, 2 H), 1.62 (s, 3 H), 1.54–1.15 (m, 6 H), 0.87 (t, *J* = 5.1 Hz, 3 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 128.8, 128.3, 68.1, 31.7, 28.5, 25.3, 22.5, 16.4, 13.9.

MS (EI): *m*/*z* = 165 $[M + Na]^+$.

[(2*S*,3*S*)-2-Methyl-3-pentyloxiran-2-yl]methanol (6)

A mixture of (+)-DET (2.36 g, 11.4 mmol) and Ti(O*i*-Pr)$_4$ (2.72 mL, 9.1 mmol) in CH_2Cl_2 (20 mL) containing 4Å molecular sieves was stirred for 15 min at –25 °C under an N_2 atmosphere. After 15 min, 4 M *t*-BuOOH in toluene (42 mL, 168 mmol) was added over a period of 10 min and stirring was continued for 30 min. Then a soln of allyl alcohol **9** (10.86 g, 76.4 mmol) in anhyd CH_2Cl_2 (100 mL) was slowly added at –25 °C and the mixture was allowed to stir for 1.5 h at –25 °C and then 20% NaOH (9 mL) was added followed by H_2O (50 mL) at 0 °C. The resulting mixture was warmed up to r.t. for 1 h and diluted with CH_2Cl_2. The mixture was filtered through Celite and extracted with CH_2Cl_2 (2 × 250 mL). The combined organic layers were washed with H_2O and brine soln and concentrated under reduced pressure. Removal of the solvent followed by purification by column chromatography (silica gel, 15% EtOAc–hexane) afforded **6** (10.87 g, 90%) as a colorless oil.

$[\alpha]_D^{28}$ –13.5 (*c* 1.6, $CHCl_3$).

IR (neat): 3427, 2956, 2927, 2862, 1461, 1382, 1040 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 3.71–3.39 (m, 2 H), 1.98 (m, 2 H), 2.96 (t, *J* = 5.1 Hz, 1 H), 1.61–1.20 (m, 9 H), 0.90 (t, *J* = 6.6 Hz, 3 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 65.7, 61.2, 60.4, 31.5, 28.0, 26.0, 22.4, 14.0, 13.8.

HRMS (ESI): *m*/*z* $[M + H]^+$ calcd for $C_9H_{19}O_2$: 159.1385; found: 159.1390.

(2*R*,3*S*)-3-(*tert*-Butyldimethylsiloxy)-2-methyloctanal (5)

To a cooled (–42 °C) suspension of 4 Å MS (3.0 g) in anhyd CH_2Cl_2 (80 mL) was added a soln of epoxy alcohol **6** (10 g, 63.2mmol) and *i*-Pr_2NEt (11.43 g, 88.6 mmol) in CH_2Cl_2. After 20 min, TBSOTf (21.72 g, 82.2 mmol) was added dropwise over 15 min. The resulting soln was stirred for 1 h at –42 °C, then quenched by addition of a buffer soln (25 mL) at pH 7.0 and was allowed to warm to r.t. The resulting mixture was diluted with hexane and the phases were separated. The combined organic layers were washed sat. $CuSO_4$ (2 ×) followed by brine and then dried (anhyd Na_2SO_4), filtered through Celite, and concentrated under reduced pressure to afford the crude **5** (14.63 g, 85%) as a colorless oil. This crude aldehyde was used as such in the next step without further purification.

Ethyl (4*S*,5*S*,*E*)-5-(*tert*-Butyldimethylsiloxy)-4-methyldec-2-enoate (10)

A soln of aldehyde **5** (2 g, 7.3 mmol) in anhyd CH_2Cl_2 (50 mL) was added $Ph_3P{=}CHCO_2Et$ (3.83 g, 11.0 mmol). The resulting mixture was stirred for 12 h at r.t. and then extracted with Et_2O (2 × 100 mL). The combined organic extracts were dried (anhyd Na_2SO_4) and concentrated under reduced pressure to give 2.01 g (80%) of the crude **10** (as a mixture of *cis*/*trans* isomers), which was used as such in the next step.

Ethyl (4*S*,5*S*)-5-(*tert*-Butyldimethylsiloxy)-4-methyldecanoate (11)

A soln of ester **10** (2 g, 5.8 mmol) was dissolved in anhyd EtOAc and then Pd/C (140 mg, 1.10 mmol) was added. The mixture was stirred for 6 h at r.t. under H_2 atmosphere. After completion, the catalyst was filtered through a Celite pad and concentrated under reduced pressure. The resulting crude residue was purified by column chromatography (silica gel) to give **11** (1.91 g, 95%).

$[\alpha]_D^{25}$ –5.2 (*c* 2.15, $CHCl_3$).

IR (neat): 3450, 2950, 2857, 2246, 1740, 1637, 1464, 1383, 1254, 1088, 836, 774, 667 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 4.08 (q, *J* = 7.3, 14.6 Hz, 2 H), 3.52–3.45 (m, 1 H), 2.26 (dd, *J* = 6.5, 14.6 Hz, 2 H), 1.44–1.18 (m, 18 H), 0.93–0.78 (m, 11 H), 0.01 (s, 6 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 174.1, 76.7, 60.2, 37.4, 33.7, 32.9, 32.6, 29.7, 26.1, 25.7, 22.8, 18.3, 14.7, –0.1.

HRMS (ESI): *m*/*z* $[M + Na]^+$ calcd for $C_{19}H_{40}O_3NaSi$: 367.2644; found: 367.2653.

***cis*-(4*S*,5*S*)-4-Methyl-5-decanolide (1)**

A soln of ester **11** (1 g, 2.9 mmol) in AcOH (10 mL), 1 M HCl (10 mL), and THF (10 mL) was stirred at 65 °C for 4 h. After completion, the mixture was quenched with sat. $NaHCO_3$ soln (100 mL) and extracted with EtOAc (3 × 150 mL). The combined organic layers were washed with brine, dried (anhyd Na_2SO_4), and concentrated under reduced pressure. The crude product was purified by flash chromatography (silica gel, 50% EtOAc–hexane) to afford the pure **1** (0.34 g, 65%) as a colorless viscous oil.

$[\alpha]_D^{25}$ –33 (*c* 1.25, $CHCl_3$).

IR (neat): 3452, 2930, 2863, 1734, 1460, 1378, 1246, 1122, 994, 909 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 4.33–4.21 (m, 1 H), 2.51 (t, *J* = 7.5 Hz, 2 H), 2.10–1.96 (m, 2 H), 1.74–1.24 (m, 9 H), 0.98 (d, *J* = 7.5 Hz, 3 H), 0.90 (t, *J* = 6.7 Hz, 3 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 172.1, 83.1, 35.8, 32.2, 32.1, 31.0, 28.4, 26.8, 26.1, 25.3, 14.6.

HRMS (ESI): *m*/*z* $[M + Na]^+$ calcd for $C_{11}H_{20}O_2Na$: 207.1360; found: 207.1366.

(2*S*,3*S*)-3-(*tert*-Butyldimethylsiloxy)-2-methyloctan-1-ol (12)

A soln of aldehyde **5** (2 g, 7.3 mmol) in MeOH (30 mL) was treated with $NaBH_4$ (558 mg, 5 mmol) slowly at 0 °C. The mixture stirred at 0 °C for 2 h. After completion, the solvent was removed under reduced pressure and then extracted with Et_2O (3 × 50 mL). The combined organic layers were washed with H_2O, brine, dried (anhyd Na_2SO_4) and concentrated under reduced pressure. The crude product was purified by column chromatography (silica gel, 14% EtOAc–hexane) to afford **12** (1.85 g, 92%) as a colorless oil.

$[\alpha]_D^{27}$ –4.0 (*c* 1.0, $CHCl_3$).

IR (neat): 3452, 2920, 2852, 1464, 1360, 1174 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 3.80–3.72 (m, 1 H), 3.65 (dd, *J* = 10.5, 2.0 Hz, 1 H), 3.49 (dd, *J* = 10.5, 5.0 Hz, 1 H), 1.98–1.87 (m, 1 H), 1.52–1.13 (m, 10 H), 0.92 (s, 12 H), 0.82 (d, *J* = 6.9 Hz, 2 H), 0.10 (d, *J* = 6.7 Hz, 6 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 68.1, 66.5, 38.7, 34.4, 31.8, 25.7, 22.5, 18.1, 14.3, 10.5, 0.1,

HRMS (ESI): *m*/*z* [M + Na]$^+$ calcd for $C_{15}H_{34}O_2NaSi$: 297.2225; found: 297.2224.

(2*S*,3*S*)-3-(*tert*-Butyldimethylsiloxy)-2-methyloctyl 4-Methylbenzenesulfonate (13)

To a soln of alcohol **12** (1.85 g, 6.7 mmol) in anhyd CH_2Cl_2 (20 mL) at 0 °C, Et_3N (1.87 mL, 13.5 mmol), TsCl (1.53 g, 8.1 mmol), and DMAP (164 mg, 1.35 mmol) were added under an N_2 atmosphere and the stirring continued for a further 4 h. After completion, the mixture was quenched with sat. aq NH_4Cl soln and then diluted with EtOAc (2 × 50 mL), washed with H_2O and brine, and dried (Na_2SO_4). Removal of the solvent followed by purification by column chromatography (silica gel, 10% EtOAc–hexane) afforded **13** (2.6 g, 90%) as a colorless oil.

$[\alpha]_D^{27}$ +7.6 (*c* 1.1, $CHCl_3$).

IR (neat): 3449, 2954, 2922, 2857, 1708, 1605, 1513, 1464, 1364, 1253, 1180, 1097, 1040, 967, 836, 773, 667, 554 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.73 (d, *J* = 8.0 Hz, 2 H), 7.31 (d, *J* = 8.0 Hz, 2 H), 4.03–3.57 (m, 3 H), 3.45 (s, 3 H), 1.99–1.79 (m, 1 H), 1.50–1.09 (m, 11 H), 0.89 (d, *J* = 3.67 Hz, 3 H), 0.81 (s, 9 H), 0.02 (d, *J* = 9.9 Hz, 6 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 144.5, 133.0, 129.7, 127.8, 73.01, 71.8, 37.2, 33.7, 31.8, 25.7, 25.3, 17.9, 13.3, 10.4, –0.1, –3.6.

MS (ESI): *m*/*z* = 429 [M + H]$^+$.

(3*S*,4*S*)-4-(*tert*-Butyldimethylsiloxy)-3-methylnonanenitrile (14)

To a stirred soln of tosylate **13** (2 g, 4.6 mmol) in anhyd DMSO was added a soln of NaI (700 mg, 4.6 mmol) and NaCN (915 mg, 18.6 mmol) in anhyd DMSO (15 mL). The mixture was stirred at 60 °C for 12 h. After completion, the mixture was diluted with H_2O and extracted with Et_2O (3 × 50 mL). The combined organic layers were washed with brine, dried (anhyd Na_2SO_4), and concentrated under reduced pressure. The crude product was purified by column chromatography (silica gel, 12% EtOAc–hexane) to give pure **14** (1.08 g, 82%) as a colorless oil.

$[\alpha]_D^{27}$ +7.6 (*c* 1.1, $CHCl_3$).

IR (neat): 3450, 2955, 2930, 2857, 2246, 1464, 1383, 1254, 1088, 836, 774 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 3.76–3.65 (m, 1 H), 2.45 (dd, *J* = 16.1, 6.6 Hz, 1 H), 2.20 (dd, *J* = 16.1, 8.0 Hz, 1 H), 2.08–1.95 (m, 1 H), 1.50–1.20 (m, 11 H), 1.03 (d, *J* = 6.6 Hz, 3 H), 0.93 (s, 9 H), 0.11 (s, 6 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 119.0, 96.2, 73.7, 35.4, 33.3, 25.9, 25.4, 22.6, 20.8, 14.0, 13.6, –4.5, –4.1.

MS (ESI): *m*/*z* = 301 [M + H_2O]$^+$.

(4*S*,5*S*)-*cis*-4-Methyl-5-pentyldihydrofuran-2(3*H*)-one (3)

A soln of nitrile **14** (1.0 g, 3.5 mmol) in abs EtOH (50 mL) in the presence of 2 M NaOH in EtOH (10 mL) was heated at 100 °C for 8 h. The solvent was removed under reduced pressure and the resulting residue was dissolved in THF (50 mL) and acidified (until pH 2.0) with aq 10% HCl. The mixture was stirred at 10 °C for 12 h. Then the mixture was diluted with EtOAc (15 mL), washed with $NaHCO_3$, followed by H_2O and brine, and the solvent was removed under reduced pressure. The crude product was purified by column chromatography (silica gel, 40% EtOAc–hexane) to afford pure **3** (420 mg, 70%).

$[\alpha]_D^{25}$ –51 (*c* 1.0, $CHCl_3$).

IR (neat): 2930, 2860, 1778, 1462, 1421, 1338, 1335, 1294, 1077, 933 cm^{-1}.

^{1}H NMR (300 MHz, $CDCl_3$): δ = 4.46–4.31(m, 1 H), 2.60 (t, *J* = 7.3 Hz, 2 H), 2.25–2.0 (m, 1 H), 1.73–1.20 (m, 8 H), 1.0 (d, *J* = 7.3 Hz, 3 H), 0.90 (d, *J* = 6.6 Hz, 3 H).

^{13}C NMR (75 MHz, $CDCl_3$): δ = 176.6, 83.3, 37.2, 32.6, 31.3, 29.5, 25.2, 22.1, 13.6, 13.4,.

HRMS (ESI): *m*/*z* [M + Na]$^+$ calcd for $C_{10}H_{18}O_2Na$: 193.1204; found: 193.1212.

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References and Notes

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