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The β-lactone route to α,β-unsaturated δ-lactones. Total syntheses of (±)-goniothalamin and (−)-massoialactone

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Abstract—The HF-induced translactonization of 2′-silyloxy-3-trimethylsilyl-2-oxetanones, obtained through Lewis acid-promoted [2+2] cycloaddition between β-silyloxyaldehydes and trimethylsilylsilylketene, into α,β-unsaturated δ-lactones is applied to the syntheses of (±)-goniothalamin and (−)-massoialactone.

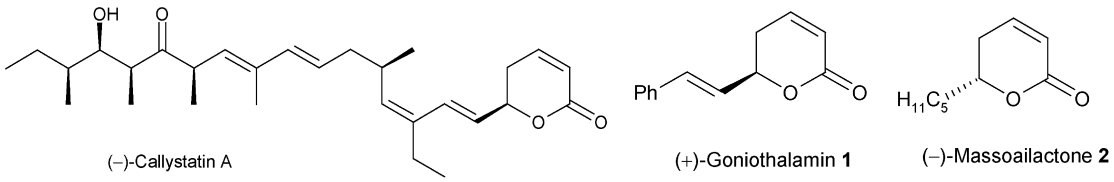
1. Introduction

The α,β-unsaturated δ-lactone (or 5,6-dihydro-2-(*2H*)-pyranone) moiety is present in a number of bioactive natural products such as (−)-callystatin A,[1] (+)-goniothalamin **1**[2] and (−)-massoialactone **2**[3] (Scheme 1). Given its activity, (−)-callystatin A has been the object of wide interest over the past decade and several syntheses or approaches of this molecule have been reported.[4–11]

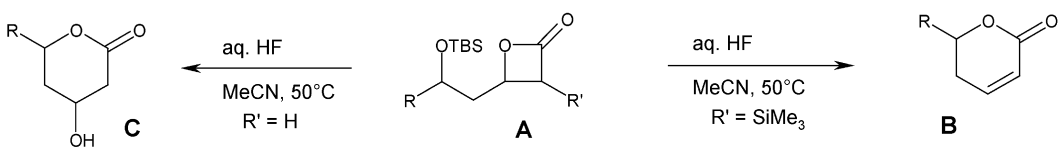
As part of a program directed towards the total synthesis of callystatin A, we recently reported a new route to the α,β-unsaturated δ-lactone moiety.[12] This approach (Scheme 2) is based on a HF-induced translactonization reaction leading, from a β-silyloxy silyl β-lactone **A**, to an α,β-unsaturated δ-lactone **B**. Provided, the β-lactone is desilylated, the reaction can selectively lead to a β-hydroxy δ-lactone **C**. Syntheses of (±)-massoialactone and (±)-prelactone B exemplify the synthetic potential of this new route to the α,β-unsaturated δ-lactone and β-hydroxy δ-lactone moieties.[12] In the present paper, we report full experimental details on the synthesis of a (±)-goniothalamin (**1**) as well as a synthesis of (−)-massoialactone **2**.

2. Synthesis of (±)-goniothalamin (1)

Originally isolated from *Cryptocarya caloneura*,[2] (+)-goniothalamin (**1**), which can also be found in various other sources,[13] was first assigned the (*S*) configuration.



Scheme 1.



Scheme 2.

Keywords: Silylketenes; Lactone; HF; [2+2] Cycloaddition; Translactonization; (−)-Dimethylmalate; Enantioselective.
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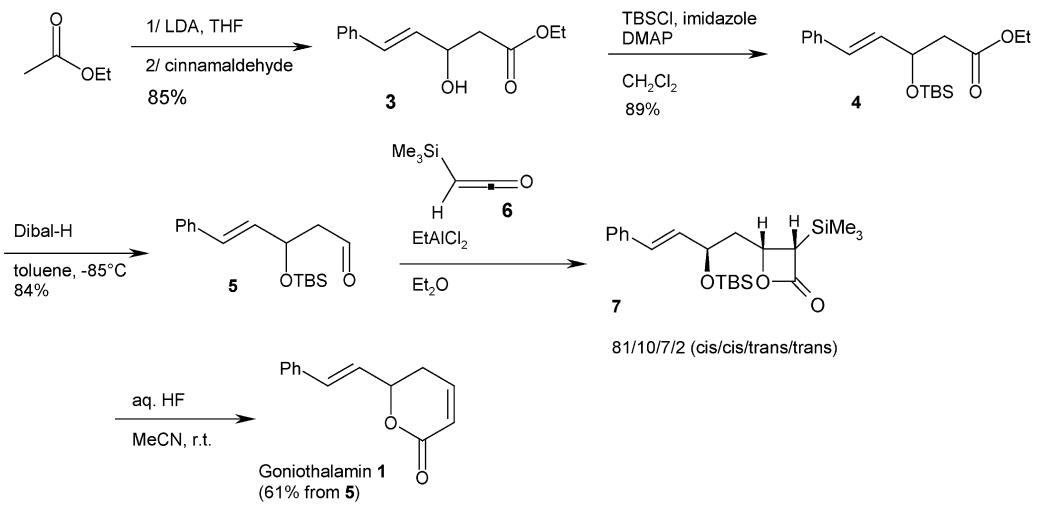


Scheme 3.

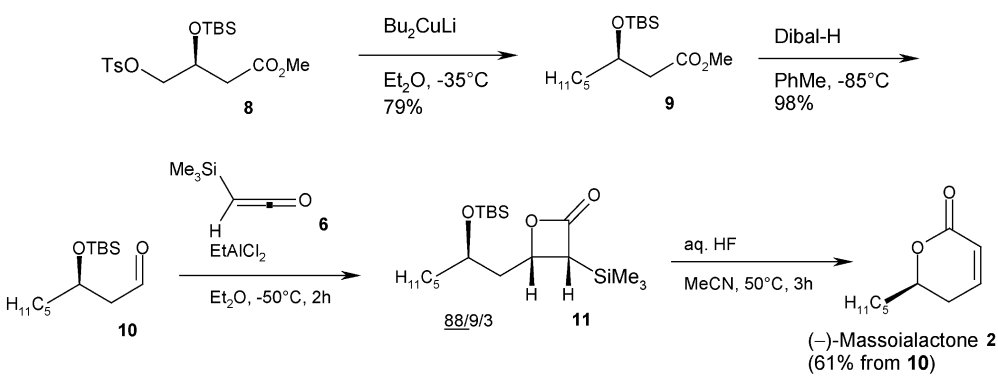
This was later corrected to the (*R*) configuration on the basis of two synthetic studies.[14,15] Interest in the synthesis of goniothalamin was stimulated by its potential as a key intermediate in the synthesis of mevinolic acid analogues. Among the many approaches to goniothalamin,[16,17] several deploy the Wittig reaction for the construction of the exocyclic double bond,[15,18,19] one uses a ring closing metathesis approach for the formation of the endocyclic double bond[20] and one the oxidation of a dihydropyran.[21]

In our synthesis (Scheme 3) the lithium enolate of ethyl acetate added to cinnamaldehyde to yield the corresponding β-hydroxy ester **3** which was protected as its *tert*-butyldimethylsilyl ether without further purification leading to unsaturated ester **4** (76% over the two steps). Ester **4** was then reduced, with Dibal-H at low temperature, to aldehyde **5** (84% yield). In the key step, aldehyde **5** underwent [2+2]-cycloaddition with trimethylsilylketene **6**[22,23] in the presence of $EtAlCl_2$ leading to β-lactones **7** as a mixture of four diastereoisomers (89/10/7/2), which were not purified. In the last step, β-lactones **7** translactonized in the presence of aq. HF in MeCN at room temperature to yield (±)-goniothalamin (**1**) in 61% overall yield for the [2+2] cycloaddition–translactonization sequence. The target molecule was thus obtained in 5 steps and 39% overall yield from cinnamaldehyde.

3. Synthesis of (−)-massoialactone

(−)-Massoialactone (**2**) is the major constituent of the bark oil of *Crytocaria massoia* and was first isolated by Abe in 1937.[3] Over the years, this molecule has received considerable interest and many racemic[12,24–28] and enantioselective[29–39] syntheses have been reported. The shortest and most efficient synthesis of (−)-massoialactone reported to date is by Ramachandran and co-workers who exploited an enantioselective allylboration of hexanal with (+)-*B*-allyldiisopinocampheylborane followed by ring closing metathesis to give the target molecule in 3 steps with 49% yield and 97% ee.[20]

Our synthesis of (−)-massoialactone (Scheme 4) began with the cheap chiral pool molecule (−)-dimethyl malate, which was transformed into ester **8** through BH_3-DMS induced monoreduction, monotosylation and *tert*-butyldimethylsilyl protection to the tosylate **8** as described in the literature (67% over the three steps).[40,41] Treatment of tosylate **8** with the lithium dialkylcuprate generated from *n*-BuLi and CuI led in 79% yield to ester **9** which was reduced with Dibal-H at low temperature to the corresponding aldehyde **10** in 98% yield. The [2+2]-cycloaddition of aldehyde **10** with trimethylsilylketene **6** gave a diastereoisomeric mixture of four β-lactones **11** which were then treated with aq. HF in



Scheme 4.

acetonitrile at 50 °C to give (−)-massoialactone **2** in 61% yield. The target molecule was obtained in 32% overall yield (99% ee) from (−)-dimethyl malate.

4. Conclusion

We have shown that the diastereoselective [2+2]-cycloaddition of β-silyloxy aldehydes with trimethylsilylketene followed by HF-induced translactonization is a useful method for the synthesis of α,β-unsaturated-δ-lactones. The method has delivered short and efficient syntheses of goniothalamin and (−)-massoialactone. Efforts are currently underway to study the mechanism of the formal elimination of Me_3SiOH and to apply this approach to a total synthesis of (−)-callystatin A.

5. Experimental

5.1. General

Reactions requiring anhydrous conditions were conducted in flame-dried apparatus under a static atmosphere of dry argon. Organic extracts were dried over $MgSO_4$ unless otherwise specified and evaporated using a rotary evaporator. Where appropriate, solvents and reagents were dried by standard methods, i.e. by distillation from the usual drying agent prior to use. All reactions were magnetically stirred and were monitored by TLC using precoated aluminium foil sheets. Flash chromatography was performed on 230–400 mesh silica gel. Optical rotations were recorded on a Perkin–Elmer 341 Polarimeter at approximatively 20 °C. Enantiomeric excesses was determined by HPLC on a Chiracel-OB-H 0.46×25 cm column (hexane/isopropanol: 80/20, 1 mL/min). IR spectra were recorded on a Perkin–Elmer 1600 series FTIR spectrometer as thin films supported on NaCl plates (absorptions are reported as values in cm^{-1}). ^1H and ^{13}C NMR spectra were recorded on Brucker AC 200, AC 300 and AC 500 spectrometers in $CDCl_3$. Chemical shifts are reported in ppm relative to residual $CHCl_3$ for ^1H NMR (δ=7.27) and $CDCl_3$ for ^{13}C NMR (δ=77.0).

5.1.1. 3-Hydroxy-5-phenylpent-4-enoic acid ethyl ester (3). A solution of *n*-butyllithium in hexane (1.6 M, 5.2 mL, 8.25 mmol) was added dropwise at −20 °C to a stirred solution of dry diisopropylamine (0.84 g, 8.25 mmol) in dry tetrahydrofuran (10 mL) under argon. After being stirred for 30 min at 0 °C, the reaction mixture was cooled to −70 °C and dry ethyl acetate (0.83 mL, 8.25 mmol) was added dropwise. The resulting solution was stirred for 1 h at −70 °C, becoming very pale yellow. Dry cinnamaldehyde (0.95 mL, 7.5 mmol) was added dropwise to it, and the reaction mixture was stirred at −70 °C for 2 h. The reaction was quenched at −70 °C by addition of glacial acetic acid (0.72 mL, 12.4 mmol) and the resulting gel was diluted with saturated aqueous sodium hydrogen carbonate (8.3 mL) and warmed to room temperature. The resulting suspension was filtered through celite, and the filtrate was washed with ether (15 mL). The aqueous and organic phases were separated, and the aqueous layer was saturated with sodium chloride, prior to extraction with ether (3×10 mL). The combined organic layers were dried ($MgSO_4$), and concentrated in vacuo. The yellow residue was purified by flash chromatography (petrol/ether: 70/30) to give alcohol **3** (1.406 g, 6.4 mmol, 85%) as a white solid: mp 39 °C. Anal. Calcd for $C_{13}H_{16}O_3$: C, 70.89; H, 7.32. Found: C, 70.93; H, 7.32. IR=3600–3200, 3024, 1733, 1495, 1400, 1275, 750 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$): δ=7.45–7.23 (5H, m), 6.67 (1H, d, *J*=15.8 Hz), 6.23 (1H, dd, *J*=16.0; 6.1 Hz), 4.74 (1H, q, *J*=5.7 Hz), 4.20 (2H, q, *J*=7.1 Hz), 3.12 (1H, s, OH), 2.66 (1H, 1/2 ABX, J_{AB}=16.2 Hz, J_{AX}=4.7 Hz), 2.64 (1H, 1/2 ABX, J_{AB}=16.2 Hz, J_{BX}=7.5 Hz), 1.29 (3H, t, *J*=7.2 Hz). ^{13}C NMR (75.4 MHz, $CDCl_3$): δ=172.2 (s), 136.4 (s), 130.7 (d), 129.9 (d), 128.6 (d, 2C), 127.8 (d), 126.5 (d, 2C), 68.9 (d), 60.8 (t), 41.5 (t), 14.2 (q).

5.1.2. 3-(*tert*-Butyldimethylsilyloxy)-5-phenylpent-4-enoic acid ethyl ester (4). A solution of *tert*-butyldimethylsilyl chloride (166 mg, 1.1 mmol) in CH_2Cl_2 (2 mL) was added to a stirred solution of imidazole (241 mg, 3.6 mmol), ester **3** (200 mg, 0.9 mmol) and 4-dimethylaminopyridine (6 mg, 0.045 mmol) in CH_2Cl_2 (3 mL) under argon and the resulting mixture was stirred at room temperature overnight. The reaction was then quenched with water (2 mL) and extracted with ether (2×5 mL). The combined organic layers were dried, concentrated in vacuo and the residue purified by flash column chromatography (petrol/ether: 90/10) to yield ester **4** (273 mg, 0.8 mmol, 89%) as a colourless oil: IR (film): ν=1736, 1464, 1368, 1252, 1164, 1076, 961, 834, 777 cm^{-1}. ^1H NMR (200 MHz, $CDCl_3$): δ=7.40–7.20 (5H, m, H(Ar)), 6.58 (1H, d, *J*=15.9 Hz), 6.20 (1H, dd, *J*=15.9, 6.7 Hz), 4.77 (1H, q, *J*=6.6 Hz), 4.13 (2H, qd, *J*=7.2, 2.0 Hz), 2.64 (1H, 1/2 ABX, J_{AB}=14.5 Hz, J_{AX}=7.8 Hz), 2.52 (1H, 1/2 ABX, J_{AB}=14.5 Hz, J_{BX}=5.5 Hz), 1.27 (3H, t, *J*=7.2 Hz), 0.90 (9H, s), 0.09 (3H, s), 0.07 (3H, s). ^{13}C NMR (75.4 MHz, $CDCl_3$): δ=171.0 (s), 136.6 (s), 131.6 (d), 129.9 (d), 128.5 (d, 2C), 126.4 (2C, d), 127.6 (d), 70.7 (d), 60.4 (t), 44.0 (t), 25.7 (3C, q), 18.1 (s), 14.2 (q), −4.3 (q), −5.1 (q).

5.1.3. 3-(*tert*-Butyldimethylsilyloxy)-5-phenylpent-4-enal (5). A solution of diisobutylaluminium hydride in toluene (1.5 M, 0.93 mL, 1.4 mmol) was added dropwise at −85 °C to a stirred solution of the protected ester **4** (265 mg, 0.79 mmol) in dry toluene (7 mL) under argon. The solution was stirred at −90 °C for 1 h, then quenched by dropwise addition of saturated aqueous ammonium chloride (0.5 mL) ($T \leq -75$ °C). The reaction mixture was allowed to warm to room temperature, and saturated aqueous Rochelle salt (potassium sodium tartrate) solution (1.6 mL) was added. The solution was poured into brine (3.2 mL) and then ethyl acetate (4.8 mL) was added. Agitation of the mixture led to formation of a gel. Further Rochelle salt solution (1.6 mL) and ethyl acetate (1.6 mL) were added to the gel, which was left overnight to break down. The resulting two liquid phases were separated, and the aqueous layer was saturated with sodium chloride prior to extraction with ethyl acetate (3×15 mL). The combined organic layers were dried ($MgSO_4$) and then solvent was removed by evaporation. The yellow residue was purified by flash chromatography (petrol/ether: 95/5) to give aldehyde **5** (193 mg, 0.66 mmol, 84%) as a colourless oil: Anal. Calcd for $C_{17}H_{26}O_2Si$: C, 70.29; H, 9.02. Found: C, 69.80; H, 9.19. IR (film): ν=1724, 1463, 1361, 1253, 1074, 968, 834, 778 cm^{-1}. ^1H NMR

(200 MHz, $CDCl_3$): δ=9.83 (1H, t, J=2.4 Hz), 7.40–7.15 (5H, m, H(Ar)), 6.60 (1H, d, J=15.9 Hz), 6.23 (1H, dd, J=15.9, 6.3 Hz), 4.84 (1H, q, J=6.4 Hz), 2.72 (1H, 1/2 ABMX, J_{AB}=15.8 Hz, J_{AM}=6.8 Hz, J_{AX}=2.6 Hz), 2.63 (1H, 1/2 ABMX, J_{AB}=15.8 Hz, J_{BM}=5.1 Hz, J_{BX}=2.2 Hz), 0.91 (9H, s), 0.11 (3H, s), 0.08 (3H, s). ^{13}C NMR (75.4 MHz, $CDCl_3$): δ=201.4 (d), 136.4 (s), 131.3 (d), 130.1 (d), 128.6 (d, 2C), 126.5 (2C, d), 127.8 (d), 69.3 (d), 51.6 (t), 25.8 (3C, q), 18.1 (s), −4.2 (q), −5.0 (q).

5.1.4. (2′*E*)-6-(2′-Phenylvinyl)-5,6-dihydro-2*H*-pyran-2-one 1 [(±)-goniothalamin 1]. To a solution of aldehyde **5** (653 mg, 2.25 mmol) in ether (16 mL) at −50 °C under argon, was added trimethylsilylketene **6** (308 mg, 2.7 mmol) in ether (6 mL). A solution of ethyl aluminium dichloride in hexane (1 M, 2.7 mL, 2.7 mmol) was then added dropwise at that temperature. The reaction was stirred for 2 h between −45 and −30 °C. The mixture was then quenched with water (7 mL) and the aqueous layer extracted with ether (2×12 mL). The organic phases were dried and concentrated to yield the corresponding β-lactone **7** as a 81/10/7/2 (*cis*/*cis*/*trans*/*trans*) mixture of diastereomers. The major *cis* diastereoisomer gave the following ^{1}H NMR spectroscopic data (300 MHz, $CDCl_3$) recorded on the mixture δ=7.50–7.24 (5H, m), 6.57 (1H, d, J=15.9 Hz), 6.17 (1H, dd, J=15.9; 6.8 Hz), 4.88 (1H, m), 4.48 (1H, m), 3.41 (1H, d, J=6.3 Hz), 1.95 (2H, m), 0.93 (9H, s), 0.23 (9H, m), 0.12 (6H, s).

To a solution of this crude mixture of β-lactones in acetonitrile (22 mL) was added aqueous HF (5.4 mL). After 15 min stirring at room temperature, the reaction was extracted with ethyl acetate (4×6 mL). The organic phases were dried over $MgSO_4$ and concentrated in vacuo. The residue was purified by flash chromatography (petrol/ethyl acetate: 75/25) to give (±)-goniothalamin **1** (276 mg, 1.38 mmol, 61%) as a white solid: mp 80 °C (lit. mp[18] 82 °C) IR (film): ν=1720, 1702, 765, 700 cm^{-1}. ^{1}H NMR (200 MHz, $CDCl_3$): δ=7.45–7.30 (5H, m), 6.93 (1H, td, J=9.9, 4.0 Hz), 6.74 (1H, d, J=16.3 Hz), 6.28 (1H, dd, J=16.0, 6.4 Hz), 6.11 (1H, dt, J=9.9, 1.8 Hz), 5.13 (1H, q, J=7.3 Hz), 2.56 (2H, m). ^{13}C NMR (75.4 MHz, $CDCl_3$): δ=163.9 (s), 144.6 (d), 135.8 (s), 133.1 (d), 128.7 (2C)(d), 128.3 (d), 126.7 (2C)(d), 125.6 (d), 121.7 (d), 77.9 (d), 29.9 (t).

5.1.5. (*R*)-(−)-3-(*tert*-Butyldimethylsilyloxy)octanoic acid methyl ester (9). A three-necked round bottom flask with a nitrogen inlet was charged with CuI (1.333 g, 7 mmol) and Et_2O (10 mL). The solution was stirred and cooled at −35 °C before BuLi (1.6 M in hexane, 8.8 mL, 14 mmol) was added. After 1 h, tosylate **8** (486 mg, 1.2 mmol) dissolved in Et_2O (7 mL) was added. The reaction was stopped after 1.5 h (TLC monitoring) by diluting with Et_2O and then adding saturated aqueous NH_4Cl (3.6 mL). The mixture was warmed to room temperature with stirring before the organic layer was separated. The aqueous layer was extracted with EtOAc (3×7 mL), and the combined organic layers were washed with saturated aqueous $NaHCO_3$ (12 mL) and brine (12 mL). The organic phase was then dried over Na_2SO_4 and concentrated in vacuo. The residue was purified by flash chromatography (petrol/ether: 90/10) to give ester **9** (274 mg, 0.95 mmol, 79%) as a colourless oil: $[\alpha]_D$=−20.7 (c=1, $CHCl_3$) (lit.[42] $[\alpha]_D$=−20.77 (c=1.32, $CHCl_3$)).^{1}H NMR (300 MHz, $CDCl_3$): δ=4.13 (1H, quint, J=6.2 Hz), 3.67 (3H, s), 2.44 (2H, d, J=6.2 Hz), 1.50–1.25 (8H, m), 0.94 (3H, t, J=5.7 Hz), 0.92 (9H, s), 0.07 (3H, s), 0.04 (3H, s). ^{13}C NMR (62.9 MHz, $CDCl_3$, lit.[42]): δ=172.2 (s), 69.6 (d), 51.2 (q), 42.6 (t), 37.6 (t), 31.9 (t), 25.7 (3C) (q), 24.6 (t), 22.5 (t), 17.9 (s), 13.8 (q), −4.6 (q), −4.9 (q).

5.1.6. (*R*)-(−)-3-(*tert*-Butyldimethylsilyloxy)octanal (10). A solution of diisobutylaluminium hydride in toluene (1.5 M, 1.3 mL, 1.94 mmol) was added dropwise at −85 °C to a stirred solution of the protected ester **9** (507 mg, 1.76 mmol) in dry dichloromethane (9 mL) under argon. The solution was stirred at −90 °C for 1 h, then quenched by dropwise addition of saturated aqueous ammonium chloride (0.5 mL) ($T\leq$−75 °C). The biphasic mixture was allowed to warm to room temperature, filtrated through a short path of silica gel and the filtrate was washed with dichloromethane. Purification of the crude product by flash chromatography (petrol/ether: 90/10) yielded aldehyde **10** (447 mg, 1.73 mmol, 98%) as a colourless oil: $[\alpha]_D$=−5.3 (c=1.0, $CHCl_3$) (lit.[43] $[\alpha]_D$=−5.3 (c=1.0, $CHCl_3$)) IR (film): ν=1725, 1255, 1101, 836 cm^{-1}. ^{1}H NMR (300 MHz, $CDCl_3$): δ=9.81 (1H, t, J=2.4 Hz), 4.20 (1H, quint, J=5.8 Hz), 2.52 (2H, dd, J=5.8, 2.6 Hz), 1.60–1.33 (8H, m), 0.89 (3H, t, J=6.0 Hz), 0.86 (9H, s), 0.08 (3H, s), 0.06 (3H, s). ^{13}C NMR (75.4 MHz, $CDCl_3$): δ=202.0 (d), 68.2 (d), 50.8 (t), 37.8 (t), 31.7 (t), 25.7 (q, 3C), 24.7 (t), 22.5 (t), 17.9 (s), 13.9 (q), −4.5 (q), −4.6 (q).

5.1.7. (*R*)-(−)-6-Pentyl-5,6-dihydro-2*H*-pyran-2-one 2 [massoialactone (2)]. To a solution of aldehyde **10** (447 mg, 1.73 mmol) in ether (12 mL) at −50 °C under argon, was added trimethylsilylketene **6** (237 mg, 2.08 mmol) in ether (3 mL). A solution of ethyl aluminium dichloride in hexane (1 M, 2.1 mL, 2.08 mmol) was then added dropwise at that temperature. The reaction was stirred for 2 h between −45 and −30 °C. The mixture was quenched with water (5 mL) and the aqueous layer extracted with ether (2×15 mL). The organic phases were dried and concentrated in vacuo to yield oxetanones **11** as a mixture of 3 diastereoisomers (*cis*/*trans*/*trans*: 88/9/3). The major cis diastereoisomer gave the following ^{1}H NMR spectroscopic data (300 MHz, $CDCl_3$) recorded on the mixture δ=4.80 (1H, ddd, J=10.6, 6.2, 2.7 Hz), 3.85 (1H, m), 3.38 (1H, d, J=6.2 Hz), 1.90–1.20 (10H, m), 0.92 (9H, s), 0.95–0.80 (3H, m), 0.24 (9H, br s), 0.08 (6H, br s).

To a solution of the crude mixture of oxetanones in acetonitrile (17 mL) was added aqueous HF (4.1 mL). After 4 h stirring at 50 °C, the reaction was extracted with ether (4×10 mL). The organic phases were dried over $MgSO_4$ and concentrated in vacuo. The residue was purified by flash chromatography (petrol/ethyl acetate: 75/25) to give (−)-massoialactone **2** (178 mg, 1.06 mmol, 61%, with 99% ee): $[\alpha]_D$=−110.7 (c=1, $CHCl_3$). [Lit.[31] $[\alpha]_D$=−110.7 (c=1, $CHCl_3$)]. IR (film): ν=1725, 1630 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$): δ=6.85 (1H, ddd, J=9.7, 5.4, 3.1 Hz), 6.00 (1H, ddd, J=9.7, 2.3, 1.3 Hz), 4.39 (1H, ddt, J=10.5, 7.4, 5.3 Hz), 2.31 (2H, m), 1.80–1.45 (4H, m), 1.42–1.25 (4H, m), 0.90 (3H, t, J=6.9 Hz). ^{13}C NMR (50.3 MHz, $CDCl_3$): δ=164.6 (s), 145.0 (d), 121.4 (d), 78.0 (d), 34.8 (t), 31.5 (t), 29.3 (t), 24.4 (t), 22.4 (t), 13.9 (q).

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