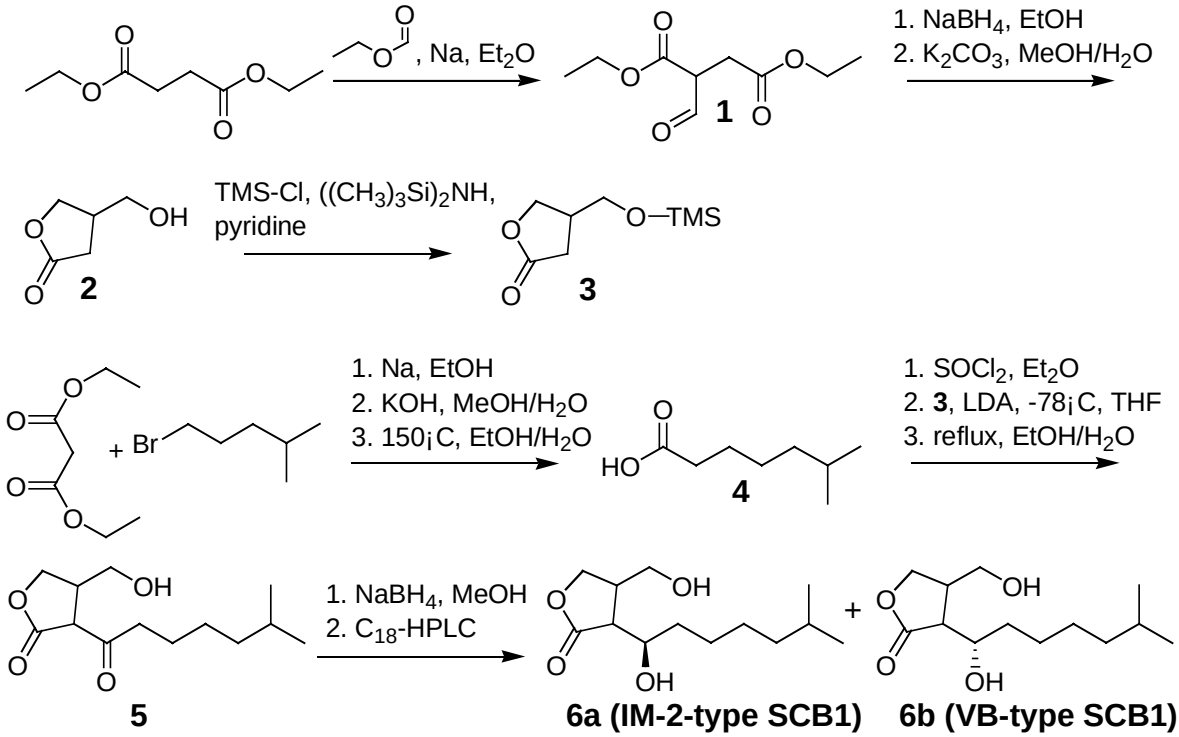
Supplementary Information

Synthesis of SCB1



Synthesis of racemic SCB1

General

All reagents and solvents were obtained from commercial sources and used without further purification. Reactions were monitored by thin layer chromatography on Fluka (Buchs, Switzerland) precoated silica gel 60 F-254 (0.25 mm). Spots were visualized with UV light or potassium permanganate. ^{1}H NMR (300.1 MHz) and ^{13}C NMR (75.5 MHz) spectra were recorded on a Bruker Avance 300 spectrometer. Mass spectra were obtained by atmospheric pressure chemical ionization at 40 eV/60 eV using a Hewlett Packard 1100 LC-MS apparatus.

Diethyl formylsuccinate (1)

200 ml diethyl ether, 9.0 g sodium (391 mmol), 46.2 ml diethyl succinate (267 mmol) and 33.7 ml ethyl formate (419 mmol) were added into a cooler-equipped three-neck flask. When the reaction mixture was warmed using a waterbath, the reaction mixture turned into a brown viscous mass due to the formation of the sodium salt of the formylsuccinate ester. After 1 h at about 50°C, the mixture was stirred for another 3 h at room temperature. Then ice was added until the sodium salts dissolved. After all the remaining sodium metal had reacted, the aqueous phase was acidified to pH 3 using 2 M HCl. The ether phase was extracted three times with 200 ml 1 M NaOH, these aqueous solutions were also acidified with 2 M HCl. All the acidic solutions were combined and thoroughly extracted with diethyl ether, the organic solutions were washed with 5% $NaHCO_3$ and brine, dried over Na_2SO_4, filtered and concentrated *in vacuo*. The remaining liquid was distilled under vacuum (bp 76°C/6mbar) resulting in 40 g (199 mmol) of colorless oil.

^{1}H NMR: ($CDCl_3$) δ 1.31 (t, 6H, $CH_2C\underline{H}_3$), 2.91 (q, 2H, $C\underline{H}_2$), 4.26 (m, 4H, $C\underline{H}_2CH_3$), 7.09 (d, 1H, C<u>H</u>), 11.52 (d, 1H, C<u>H</u>O)

^{13}C NMR: ($CDCl_3$) δ 14.1 ($CH_2\underline{C}H3$), 29.2 ($\underline{C}H_2$), 54.1 (<u>C</u>H), 60.7 ($\underline{C}H_2CH_3$), 171.7 (<u>C</u>OO), 195.8 (<u>C</u>HO)

3-(hydroxymethyl)-butanolide (2)

NaBH4 (8.3 g, 220 mmol) was added stepwise to a solution of 22.2 g (110 mmol) diethyl formylsuccinate in 100 ml ethanol under stirring and ice cooling. After $NaBH_4$ addition, the reaction mixture was stirred overnight at room temperature. A 4 M aqueous HCl solution was added and after stirring for 1 h, the precipitate formed was filtered off. The filtrate was concentrated *in vacuo* and the residue was extracted with ethyl acetate. The organic solution was washed with brine, dried over Na_2SO_4 and concentrated to give 6.4 g of yellowish oil. The oil was dissolved in 60 ml methanol and 20 ml

water, then 7.5 g K_2CO_3 was added portionwise. After refluxing for 3 h, the reaction mixture was acidified with 4 M HCl and concentrated *in vacuo*. The residue was extracted with ethyl acetate and the organic solution was washed with brine, dried over Na_2SO_4 and concentrated yielding 6.4 g of the crude product. The product was purified by column chromatography (silica, ethyl acetate/ethanol 95:5 v/v) resulting in 1.5 g (9.3 mmol) lactone.

R*f* = 0.53 (ethyl acetate/ethanol 95:5 v/v)

^{1}H-NMR: ($CDCl_3$) δ 2.41 (dd, 1H, OCOC$\underline{H}_2$), 2.63 (dd, 1H, OCOC$\underline{H}_2$), 2.78 (m, 1H, C$\underline{H}$), 2.80 (br s, 1H, O$\underline{H}$), 3.69 (m, 2H, C$\underline{H}_2$OH), 4.23 (dd, 1H, COOC$\underline{H}_2$), 4.43 (dd, 1H, COOC$\underline{H}_2$)

^{13}C-NMR: ($CDCl_3$) δ 30.9 (OCO$\underline{C}H_2$), 37.1 ($\underline{C}$H), 63.0 ($\underline{C}H_2$OH), 70.8 (COO$\underline{C}H_2$), 177.6 ($\underline{C}$(O)O)

3-(trimethylsilyloxymethyl)-butanolide (3)

5 ml hexamethyldisilazane (24 mmol) and 5 ml trimethylsilyl chloride (40 mmol) were added to an ice-cooled solution of 5.2 g 3-(hydroxymethyl)-butanolide (**2**) (45 mmol) in 5 ml pyridine. The reaction mixture was stirred for 2 h at room temperature under an argon atmosphere. Quickly after the addition a white precipitate was formed. Then, 10 ml of benzene/hexane (1:1 v/v) was added to the reaction mixture and the precipitate was removed by filtration over Hyflow. The filtrate was concentrated *in vacuo* and the product was obtained after column chromatography (silica, ethyl acetate/hexane (1:2 v/v) as a colorless oil (4.5 g, 1.47 mmol).

R*f* = 0.48 (ethyl acetate/hexane 1:2 v/v)

^{1}H-NMR: ($CDCl_3$) δ 0.12 (s, 9H, Si(C$\underline{H}_3$)$_3$), 2.36 (dd, 1H, OCOC$\underline{H}_2$), 2.57 (dd, 1H, OCOC$\underline{H}_2$), 2.73 (m, 1H, C$\underline{H}$), 3.59 (m, 2H, C$\underline{H}_2$OTMS), 4.17 (dd, 1H, COOC$\underline{H}_2$), 4.37 (dd, 1H, COOC$\underline{H}_2$)

^{13}C-NMR: ($CDCl_3$) δ -0.7 (Si(<u>C</u>H_3)), 30.7 (OCO<u>C</u>H_2), 37.2 (<u>C</u>H), 62.7 (<u>C</u>H_2OH) , 70.5 (COO<u>C</u>H_2), 177.1 (<u>C</u>OO)

6-methylheptanoic acid (4)

Sodium (0.97 g, 42 mmol) was dissolved in 100 ml ethanol of 55°C. When sodium was no longer visible and the mixture was cooled to <50°C, 6.4 ml (40 mmol) diethyl malonate was added and the mixture was refluxed for about 20 min. Then 5.7 ml (39 mmol) 1-bromo-4-methylpentane were added to the stirred solution and the reaction mixture was refluxed for 3 h. The reaction mixture was concentrated *in vacuo*, acidified with 3 M HCl and extracted with dichloromethane. The dichloromethane solution was dried over Na_2SO_4 and evaporated to yield 9.5 g of yellowish oil. This crude diethyl 2-(4'-methylpentyl)malonate was stirred overnight at room temperature in a mixture of 9.5 g KOH in 30 ml water and 60 ml methanol. The methanol was removed *in vacuo* and the remaining aqueous solution was washed with diethyl ether, acidified with 3 M HCl and extracted with dichloromethane. The dichloromethane solution was dried over Na_2SO_4 and evaporated to yield 4.6 g of white crystals.

7.7 g crude 2-(4'-methylpentyl)malonate was heated at 150°C for 12 h in the presence of boiling stones, then 50 ml of a 5% $NaHCO_3$ solution were added and the mixture was washed with hexane to remove the neutral impurities. The aqueous solution was acidified with 3 M HCl and extracted with dichloromethane. The dichloromethane solution was dried over Na_2SO_4, concentrating *in vacuo* gave 4.4 g of crude product. The pure product (4.0 g, 28 mmol) was obtained after distillation under reduced pressure (bp 87°C/5 mbar).

^{1}H-NMR: ($CDCl_3$) δ 0.87 (d, 6H, C<u>H</u>$_3$), 1.20 (m, 2H, C<u>H</u>$_2$), 1.34 (m, 2H, C<u>H</u>$_2$), 1.54 (m, 1H, C<u>H</u>), 1.62 (m, 2H, C<u>H</u>$_2$), 2.35 (t, 2H, C<u>H</u>$_2$COOH), 10.72 (br s, 1H, COO<u>H</u>)

^{13}C-NMR: ($CDCl_3$) δ 22.6 ($\underline{C}H_3$), 24,9 ($\underline{C}H_2$), 26.9 ($\underline{C}H_2$), 27.8 ($\underline{C}H$), 34.1 ($\underline{C}H_2$), 38.5 ($\underline{C}H_2COOH$), 180.4 ($\underline{C}OOH$)

2-(6-methylheptanoyl)-3-(hydroxymethyl)-butanolide (5)

0.94 g 3-(trimethylsilyloxymethyl)-butanolide (5 mmol) was added to 5 ml of a 2 M lithium diisopropylamine solution in THF at –78°C and the mixture was stirred for 1.5 h at –78°C. Then 0.81 g 6-methylheptanoyl chloride (5 mmol), which was prepared from 6-methylheptanoic acid with 2 eq thionyl chloride, was added and the reaction was stirred for another 1.5 h at –78°C. The reaction mixture was allowed to warm up to 0°C before 50 ml ice-cold water with 7.5 ml acetic acid was added and subsequently extracted three times with 150 ml dichloromethane. The dichloromethane was concentrated *in vacuo* and the residue was dissolved in 200 ml dichloromethane, washed with 100 ml saturated $NaHCO_{3\ (aq)}$ and brine. After removal of the solvents *in vacuo*, the crude trimethylsilyl ether was obtained as yellowish oil. The trimethylsilyl group was removed by refluxing the oil in 40 ml of ethanol and 10 ml of water for 30 min. After concentrating the reaction mixture *in vacuo*, the racemic A-factor (188 mg, 0.8 mmol) was obtained after column chromatography (silica, ethyl acetate /hexane (1:4 to 1:2 v/v) as an oil.

R*f* = 0.30 (ethyl acetate/hexane 1:2 v/v)

LC-MS: m/z 243 (M+H)

^{1}H-NMR: ($CDCl_3$) δ 0.86 and 0.87 (d, 6H, C$\underline{H}_3$), 1.19 (m, 2H, C$\underline{H}_2$), 1.34 (m, 3H, C$\underline{H}_2$ + C$\underline{H}$), 1.55 (m, 4H, C$\underline{H}_2$ + C$\underline{H}_2$), 2.31 (br s, 1H, O$\underline{H}$), 2.64 and 2.97 (dt, 1H, COC$\underline{H}$), 3.23 (m, 1H, CH_2C$\underline{H}$), 3.69 (m, 2H, C$\underline{H}_2$OH), 4.15 (m, 1H, OC$\underline{H}_2$CH), 4.45 (m, 1H, OC$\underline{H}_2$CH)

^{13}C-NMR: ($CDCl_3$) δ 22.6 ($\underline{C}H_3$), 23.5 ($\underline{C}H_2$), 26.8 ($\underline{C}H_2$), 27.8 ($\underline{C}H$), 38.7 (CO$\underline{C}H_2$), 39.2 ($CH_2\underline{C}H$), 42.5 (CO$\underline{C}H_2$), 55.0 (CO$\underline{C}H$), 61.8 (O$\underline{C}H_2$CH), 69.1 ($\underline{C}H_2$OH), 172.5 ($\underline{C}$OO), 203.0 ($\underline{C}$O)

2-(6'-methyl-1'-hydroxyheptyl)-3-(hydroxymethyl)-butanolide (6)

The racemic A-factor (150 mg, 0.6 mmol) was dissolved in 20 ml methanol and 1.5 eq sodium borohydride was added at 0°C. After stirring, the reaction mixture for 1 h at 0°C, the reaction was stopped by adding 3 M HCl. The formed precipitate was filtered off and the methanol was removed from the filtrate *in vacuo*. The aqueous solution was extracted three times with 100 ml ethyl acetate. The ethyl acetate solution was dried over Na_2SO_4 and concentrated *in vacuo*.

The two diastereomers were purified and separated using preparative HPLC (Biocad 700E, Perseptive Biosystems) with a reversed phase C18 column (Econosphere, Alltech) using UV detection at 210 nm. The used eluent system consisted of 35% acetonitrile and 65% 0.1% TFA in water at a flow rate of 10 ml/min. Racemic **6a (IM-2-type SCB1)** had a retention time of 23.5 min and was obtained in a 52 % yield (76 mg).

LC-MS: m/z 245 (M+H)

^{1}H-NMR: ($CDCl_3$) δ 0.87 (d, 6H. C<u>H</u>$_3$), 1.19 (m, 2H, C<u>H</u>$_2$), 1.33 (m, 3H, C<u>H</u>$_2$ + C<u>H</u>), 1.53 (m, 4H, C<u>H</u>$_2$ + C<u>H</u>$_2$), 2.65 (dd, 1H, COC<u>H</u>), 2.77 (m, 1H, CH$_2$C<u>H</u>), 3.29 (s, 2H, O<u>H</u>), 3.70 (ddd, 2H, C<u>H</u>$_2$OH), 3.99 (m, 2H, OC<u>H</u>$_2$CH + C<u>H</u>OH), 4.41 (t, 1H, OC<u>H</u>$_2$CH)

^{13}C-NMR: ($CDCl_3$) δ 22.6 (<u>C</u>H$_3$), 26.2 (<u>C</u>H$_2$), 27.2 (<u>C</u>H$_2$), 27.9 (CH), 34.0 (<u>C</u>H$_2$), 38.9 (<u>C</u>H$_2$), 40.1 (CH$_2$<u>C</u>H), 49.3 (CO<u>C</u>H), 62.9 (O<u>C</u>H$_2$CH), 68.6 (<u>C</u>H$_2$OH), 70.8 (<u>C</u>HOH), 177.5 (<u>C</u>OO)

The other stereoisomer racemic **6b (VB-type SCB1)** had a retention time of 21.8 min and was obtained in a 22 % yield (32 mg).

LC-MS: m/z 245 (M+H)

^{1}H-NMR: ($CDCl_3$) δ 0.87 (d, 6H. C<u>H</u>$_3$), 1.19 (m, 2H, C<u>H</u>$_2$), 1.32 (m, 3H, C<u>H</u>$_2$ + C<u>H</u>), 1.55 (m, 4H, C<u>H</u>$_2$ + C<u>H</u>$_2$), 2.55 (dd, 1H, COC<u>H</u>), 2.73 (s, 2H, O<u>H</u>),

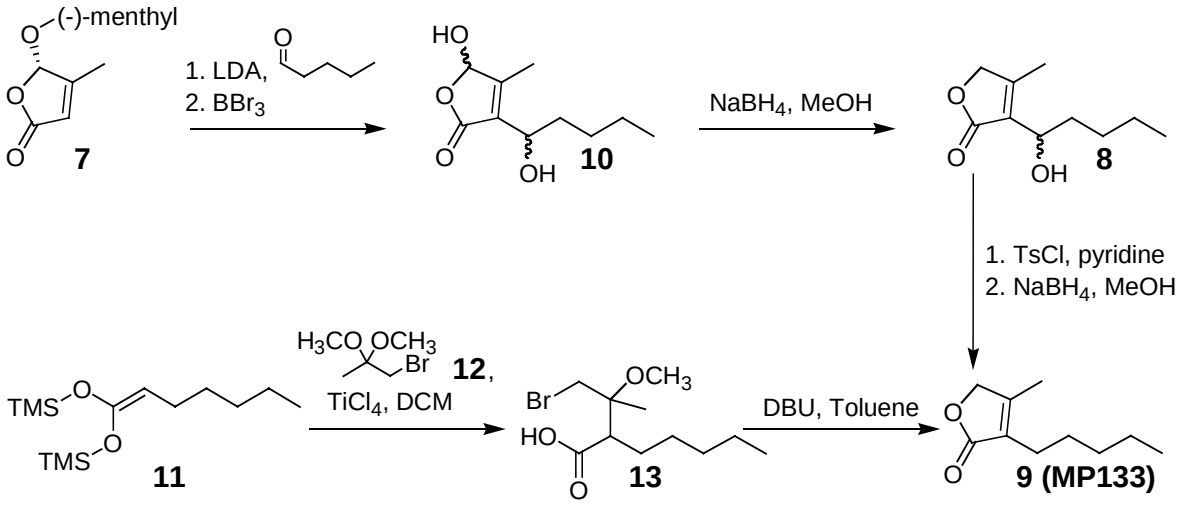
2.84 (m, 1H, $CH_2C\underline{H}$), 3.70 (m, 2H, $C\underline{H_2}OH$), 4.09 (m, 2H, $OC\underline{H_2}CH$ + $C\underline{H}OH$), 4.41 (t, 1H, $OC\underline{H_2}CH$)

^{13}C-NMR: ($CDCl_3$) δ 22.6 ($\underline{C}H_3$), 26.1 ($\underline{C}H_2$), 27.2 ($\underline{C}H_2$), 27.9 (CH), 34.9 ($\underline{C}H_2$), 38.1 ($\underline{C}H_2$), 38.9 ($CH_2\underline{C}H$), 48.3 ($CO\underline{C}H$), 63.3 ($O\underline{C}H_2CH$), 69.8 ($\underline{C}H_2OH$), 70.8 ($\underline{C}HOH$), 179.1 ($\underline{C}OO$)

Synthesis of MP133

Synthesis of MP133 using Grossmann's method

Following Grossmann's procedure (1), the readily available menthylated furanone **7** (2) was condensed with pentanal. Subsequent deprotection with BBr_3 gave **10** as a mixture of four stereoisomers. Borohydride reduction then removed the OH group at the furanone ring. The resulting compound **8** was tosylated at the side chain hydroxy group, and the tosylate obtained reduced with $NaBH_4$ to give MP133 (**9**) (3).



5-Hydroxy-3-(1-hydroxypentyl)-4-methylfuran-2(5*H*)-one (**10**, mixture of two pairs of enantiomers). Following the general procedure (1), 5-(–)-menthyloxy-4-methyl-2(5*H*)-furanone (**7**, 15 g, 59 mmol) was deprotonated at –110°C with lithium diisopropylamine (prepared from 11 ml (77 mmol) of diisopropylamine) and condensed with pentanal (8 ml, 75 mmol) to give 3-(1-hydroxypentyl)-5-[(–)-menthyloxy]-4-methyl-2-(5*H*)-furanone (5 g, 45%). Without further purification, this compound (7.3 g, 21 mmol) was deprotected (1) with BBr_3 (6 ml, 62 mmol) to give 5-hydroxy-3-(1-hydroxypentyl)-4-methyl-2-(5*H*)-furanone (**10**, 2.6 g, 60 %).

^{1}H-NMR (500 MHz, $CDCl_3$) δ 5.97 (br. s, 1H, HO-C(5)), 5.88 and 5.85 (2s, 1H, H-C(5)), 4.50-4.45 (m, 1H, H-C(1')), 3.43 (br. s, 1H, HO-C(1')), 2.08 and 2.06 (2s, 3H, CH_3-C(4)), 1.87-1.63 (2m, 2H, H_2-C(2')), 1.41-1.19 (m, 2H, H_2-C(3') and H_2-C(4')), 0.90 (t, 3H, J = 7 Hz, H_3-C(5')).

^{13}C-NMR (125 MHz, CDCl3) δ 172.2 and 171.8 (C(2)), 158.3 and 158.2 (C(4)), 130.2 and 129.8 (C(3)), 98.9 and 98.8 (C(5)), 66.6 and 66.4 (C(1')), 35.7 and 35.2 (C(2')), 27.6 and 27.5 (C(3')), 22.4 and 22.3 (C(4')), 13.9 (C(5')), 11.6 and 11.4 (CH_3-C(4)).

EI-MS (70 eV, 250°C): 182 (8), 144 (12), 143 (100), 139 (28),127 (13), 126 (38), 125 (66), 115 (19), 98 (16), 97 (72), 85 (17), 71 (11), 69 (45), 67 (11), 57 (27), 55 (11), 43 (20), 41 (55), 39 (31).

FAB-MS (NBA): 202 (11), 201 (100), 183 (81), 165 (16), 137 (23), 77 (11), 41 (14), 39 (13).

Elemental analysis for $C_{10}H_{16}O_4$ (200.24): calc.: C 59.98, H 8.05; found: C 60.05, H 8.14.

3-(1-Hydroxypentyl)-4-methyl-2-(5*H*)-furanone (**8**). Reduction of the mixture (1) **10** (2.8 g, 14 mmol) with $NaBH_4$ (2.8 g, 74 mmol) yielded after removal of the solvent and without further purification 3-(1-hydroxypentyl)-4-methyl-2-(5*H*)-furanone (**8**, 1.9 g, 74 %).

^{1}H-NMR (500 MHz, $CDCl_3$) δ 4.66 (s, 2H, H_2-C(5)), 4.05 (t, 1H, J = 7 Hz, H-C(1')), 3.23 (m, 1H, HO-C(1')), 2.10 (s, 3H, CH_3-C(4)), 1.87-1.64 (m, 2H, H_2-C(2')), 1.43-1.22 (m, 4H, H_2-C(3') and H_2-C(4')), 0.90 (t, 3H, J = 7 Hz, H_3-C(5')).

^{13}C-NMR (125 MHz, $CDCl_3$) δ 174.0 (C(2)), 157.8 (C(4)), 128.0 (C(3)), 72.6 (C(5)), 66.6 (C(1')), 35.91 (C(2')), 27.5 (C(3')), 22.3 (C(4')), 13.8 (C(5')), 12.1 (CH_3-C(4)).

EI-MS (70 eV, 150°C): 166 (4), 127 (100), 99 (22), 82 (5), 71 (5), 53 (7), 43 (10), 41 (10).

FAB-MS (NBA): 185 (81), 167 (100), 77 (11), 51 (11), 41 (14), 39 (16).

Elemental analysis for $C_{10}H_{16}O_3$ (184.24): calc.: C 65.19, H 8.75, O 26.05; found: C 64.99, H 8.63, O 26.32.

4-Methyl-3-pentyl-2-(5*H*)-furanone (MP133, **9**). 3-(1-Hydroxypentyl)-4-methyl-2-(5*H*)-furanone (**8**, 708 mg, 3.8 mmol) was added to a solution of tosyl chloride (817 mg, 4.3 mmol) in pyridine (4 ml) at 0°C. The reaction mixture was stirred overnight at 4°C. After dilution with MeOH (20 ml), $NaBH_4$ (700 mg, 19 mmol) was added and the reaction mixture was stirred for 24 hrs at room temperature, after which it was poured into aqueous 1 M HCl at 0°C. The mixture was extracted with CH_2Cl_2 (3 x 50 ml), and the combined organic layers were washed with saturated $NaHCO_3$ (3 x 50 ml) and with saturated NaCl solution (3 x 50 ml), then dried ($MgSO_4$) and evaporated under reduced pressure. The residue was chromatographed on silica gel (gradient of ethyl acetate in hexane) to give 4-methyl-3-pentyl-2-(5*H*)-furanone (MP133, **9**, 280 mg, 43 %).

^{1}H-NMR (500 MHz, $CDCl_3$) δ 4.62 (q, 2H, J = 1 Hz, $\underline{H}_2$-C(5)), 2.25 (t, 2H, J = 7.5 Hz, $\underline{H}_2$-C(1')), 2.03 (s, 3H, C$\underline{H}_3$-C(4)), 1.49 (quint, 2H, J = 8Hz, $\underline{H}_2$-C(2')), 1.36-1.25 (m, 4H, $\underline{H}_2$-C(3') and $\underline{H}_2$-C(4')), 0.89 (t, 3H, J = 7 Hz, $\underline{H}_3$-C(5')).

^{13}C-NMR (125 MHz, $CDCl_3$): 175.0 (C(2)), 156.4 (C(4)), 127.2 (C(3)), 72.3 (C(5)), 31.4 (C(3')), 27.5 (C(2')), 23.2 (C(1')), 22.3 (C(4')), 13.9 (C(5')), 12.1 ($\underline{C}H_3$-C(4)).

EI-MS (70 eV, 100°C) δ 168 (10), 153 (19), 139 (19), 113 (12), 112 (100), 111 (11), 93 (10), 69 (12), 67 (11), 55 (24), 43 (15), 41 (24), 39 (12).

FAB-MS (NBA): 169 (100).

Elemental analysis for $C_{10}H_{16}O_2$ (168.24): calc.: C 71.39, H 9.59; found: C 71.09, H 9.77.

Synthesis of MP133 using Demnitz's method

In analogy to Demnitz's method (4), heptanoic acid was converted into the corresponding ketene bis(trimethylsilyl) acetal **11** (5) which then was condensed with 1-bromo-2,2-dimethoxypropane (**12**) under $TiCl_4$ catalysis. The resulting β-methoxy-γ-bromo carboxylic acid **13** was directly converted to MP133 (**9**) with two equivalents of the base 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU).

4-Methyl-3-pentyl-2(5*H*)-furanone (MP133, **9**). To a solution of 1-bromo-2,2-dimethoxypropane (**12**, 607 µl, 832 mg, 4.50 mmol, Aldrich) in absolute dichloromethane (40 ml), $TiCl_4$ (493 µl, 853 mg, 4.50 mmol) was added in portions at –78°C under Ar. Then, a solution of 1,1-bis(trimethylsilyloxy)hept-1-ene (**11**, 1.372 g, 5.00 mmol) in absolute dichloromethane (4 ml) was added over 15 min. Stirring at –78°C was continued for 90 min. Hydrolysis was performed with a buffer ($KHSO_4$, 710 mg and Na_2HPO_4, 644 mg in 40 ml of water) and the mixture allowed to warm up to room temperature. The organic phase was separated and the aqueous phase washed with dichloromethane (3 x 50 ml). The combined organic phases were dried (Na_2SO_4), filtered, and the solvent removed *in vacuo*. The resulting oil (1.00 g) was disolved in absolute toluene (30 ml). DBU (900 µl, 910 mg, 6.0 mmol) was added and the mixture kept at 60°C for 24 hrs. The mixture was then washed with 1 M HCl and saturated $NaHCO_3$ solution. The organic phase was dried (Na_2SO_4), filtered, and the solvent removed *in vacuo*. The residue was chromatographed (silica, 200g, gradient hexane/dichloromethane 2:8 to hexane/dichloromethane 1:9 to dichloromethane) to give 204 mg (27 %) of MP133 (**9**) as a colorless liquid.

1. Grossmann,G. and Séquin,U. (2001) *Synlett.*, **2**, 278–280.
2. Feringa,B.L., De Lange,B. and De Jong,J.C. (1989) *J. Org. Chem.*, **54**, 2471–2475.
3. Müller,P.M. and Wild,H.J. (1992) In *Eur. Pat. Appl., 91–116738*.
4. Demnitz,F.W.J. (1989) *Tetrahedron Lett.*, **30**, 6109–6112.
5. Ainsworth,C. and Kuo,Y.N. (1972) *J. Organomet. Chem.*, **46**, 73–87.