Supporting Information

for

A new phenylethyl alkyl amide from the *Ambrostoma quadriimpressum* Motschulsky

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**Detailed experimental procedures for the synthesis of compound 1**

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Experimental details

General Methods: Commercial spectral grade solvents were used for experiments unless otherwise stated. Infrared spectra were recorded on a Perkin–Elmer 1710 Fourier transform spectrometer. Low-resolution mass spectra were obtained from Agilent 7890A-5975C GC–MS by means of electron impact (EI) ionization at 70 eV. \(^1\)H NMR spectra were obtained at 400 MHz on Bruker AV-400 instrument. \(^{13}\)C NMR spectra were recorded at 100 MHz. High-resolution mass spectra were recorded on an Agilent 1200-6520 Q-TOF electrospray mass spectrometer.

Synthesis of (S)-5-benzyl 1-methyl 2-hydroxypentanedioate (5)

\[
\begin{align*}
\text{NH}_2 \quad \text{COOH} & \quad 1. \text{BnOH, } H_2SO_4 \\
& \quad 2. \text{NaNO}_2, \text{AcOH} \\
& \quad 3. \text{CH}_2N_2 \text{ work up} \\
\text{BnOOC} & \quad \text{COOMe} \\
\text{OH} & \quad 5
\end{align*}
\]

The \(\gamma\)-benzyl ester of glutamic acid (5) was prepared according to Rapoport et al. [1-3]. Benzyl alcohol (200 mL, 1.9 mol) was added slowly to a solution of conc. \(H_2SO_4\) (20 mL, 375 mmol) in dry \(Et_2O\) (200 mL). The solution was concentrated in vacuo, and glutamic acid (29.6 g, 201 mmol) was added sequentially. The reaction mixture was stirred overnight at r.t., then 95% EtOH (400 mL) and pyridine (100 mL) were added. The mixture was stirred for 1 h in an ice bath. The white precipitate \(\gamma\)-benzylglutamic acid was obtained by filtration, washed with \(Et_2O\), dried at 60 °C for 24 h in vacuo, and was used for next step without further purification (yield 28.6 g, 60%). To a solution of \(\gamma\)-benzylglutamic acid (23.8 g, 100 mmol) in \(H_2O\) (150 mL) and AcOH (50 mL), a solution of \(NaNO_2\) (10.3 g, 149 mmol) in \(H_2O\) (100 mL) was added slowly over 4 h. After stirring for 2 h at rt, the reaction mixture was extracted with CHCl₃ and isopropanol (v/v = 3:1). The extracts were washed with brine, dried with \(Na_2SO_4\), and concentrated in vacuo to give a colorless oil. The crude product was immediately
esterified with CH₂N₂ in Et₂O, and then concentrated in vacuo. Column chromatography (SiO₂, AcOEt/PE 1:1) of the residue afforded compound 5 (12.4 g, 70%) as a colorless oil. [α]D²⁰ −13.8 (c 0.52, CHCl₃); IR (film, KBr) νₘₐₓ: 3487, 2944, 1738, 1455, 1162 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.38–7.31 (m, 5H), 5.12 (s, 2H), 4.27–4.22 (m, 1H), 3.77 (s, 3H), 2.98 (t, J = 6.4 Hz, 1H), 2.59–2.47 (m, 2H), 2.23–2.16 (m, 1H), 1.99–1.93 (m, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 175.0, 173.0, 135.9, 128.6, 128.3, 128.2, 69.5, 66.4, 52.7, 29.7, 29.3; EIMS (m/z): 252, 234, 145, 108, 91(base), 85; HRMS–ESI: Calcd. for C₁₃H₁₆O₅: 253.1076 [M + H]+. Found: 253.1080.

Synthesis of (S)-5-benzyl 1-methyl 2-(tert-butyldiphenylsilyloxy)-pentanedioate (6)

\[
\text{BnOOC} \text{COOMe} \xrightarrow{TBDPSCI, Imida.} \text{BnOOC} \text{COOMe} \xrightarrow{\text{OH}} \text{OTBDPS}
\]

Imidazole (1.36 g, 20 mm) was added sequentially to a solution of 5 (2.52 g, 10 mmol) in dry CH₂Cl₂ (150 mL) at 0 °C. The mixture was stirred for 15 min, and then TBDPSCI (4.12 g, 15 mmol) was added and stirred at rt for 3 h. The reaction mixture was quenched with H₂O and extracted with CH₂Cl₂. The combined extracts were washed with brine, dried (Na₂SO₄) and concentrated in vacuo. Column chromatography (SiO₂, 10% AcOEt in PE) of the residue afforded TBDPS ether (4.9 g, 100%) as a colorless oil. [α]D²⁰ −12.8 (c 1.0, CHCl₃); IR (film, KBr) νₘₐₓ: 2945, 2831, 1740, 1455, 1109, 751, 699 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.76–7.71 (m, 4H), 7.69–7.58 (m, 11H), 5.09 (s, 2H), 4.32 (t, J = 5.2 Hz, 1H), 3.77 (s, 3H), 2.58 (m, 1H), 2.46 (m, 1H), 2.08 (m, 2H), 1.09 (s, 9H); ¹³C NMR (100 MHz, CDCl₃): δ 172.9, 172.9, 136.0, 135.8, 135.2, 133.1, 132.9, 129.9, 129.8, 128.6, 127.7, 127.6, 71.4, 66.3, 51.5, 30.0, 29.2, 26.9, 19.4.
EI MS (m/z): 433, 373, 289, 265, 237, 213, 183, 153, 135, 91 (base). HRMS (ESI): Calcd. for C_{29}H_{34}O_{5}Si: 508.2519 [M + NH_{4}]^+. Found: 508.2530.

Synthesis of (S)-methyl 2-(t er t -butyldiphenylsilyloxy)-5-hydroxy pentanoate (7)

To a solution of 6 (4.9 g, 10 mmol) in MeOH (30 mL), 10% Pd/C (447 mg) was added and the mixture was hydrogenated for 12 h. It was then filtered through a short pad of Celite and the filter cake was washed with MeOH. The filtrate was concentrated in vacuo to give the acid as a colorless oil, which was used without further purification.

To a solution of the above acid in dried THF (30 mL) at −5 °C, BH_{3}·THF (1.0 M in THF, 11 mL) was added over 10 min. After addition, the reaction mixture was allowed to warm to rt for 3 h. Then it was carefully quenched with MeOH at 0 °C and concentrated in vacuo. Purification of the residue by flash chromatography (SiO_{2}, AcOEt/PE 1:1) provided the alcohol 7 (3.67 g, 95%) as a colorless oil. \([ \alpha ]_{20}^D −33.2 \) (c 0.93, CHCl_{3}); IR (film, KBr) \( \nu_{\text{max}} \): 3434, 2952, 1755, 1427, 1111 cm\(^{-1}\); \(^1\)H NMR (400 MHz, CDCl_{3}): \( \delta \) 7.68–7.63 (m, 4H), 7.44–7.34 (m, 6H), 4.29 (t, \( J = 5.6 \) Hz, 1H), 3.59 (t, \( J = 6.4 \) Hz, 2H), 3.49 (s, 3H), 1.84–1.77 (m, 2H), 1.66–1.59 (m, 2H), 1.10 (s, 9H); \(^{13}\)C NMR (100 MHz, CDCl_{3}): \( \delta \) 173.4, 136.0, 13.8, 133.3, 133.0, 129.9, 129.8, 127.7, 127.5, 72.3, 62.5, 51.5, 31.5, 27.8, 26.9, 19.4; EIMS (m/z): 297 (base), 277, 253, 227, 199, 183, 165, 135, 105, 77. HRMS–ESI: Calcd for C_{22}H_{30}O_{4}Si: 387.1992 [M + H]^+. Found: 387.1991.
Synthesis of (S)-methyl 5-(tert-butyldimethylsilyloxy)-2-(tert-butyldiphenylsilyloxy)pentanoate (8)

To a solution of 7 in anhydrous CH$_2$Cl$_2$ (40 mL) were added imidazole (1.29 g, 19 mmol) and TBSCl (2.15 g, 14.3 mm). After stirring for 5 h at rt, the reaction mixture was quenched with H$_2$O and extracted with CH$_2$Cl$_2$. The extracts were washed with brine, dried (Na$_2$SO$_4$), and concentrated in vacuo. Column chromatography (5% EtOAc in PE) of the residue afforded the TBS ether 8 (4.75 g, 100 %) as a colorless oil. $\alpha_{D}^{20}$ = −18.3 (c 0.52, CHCl$_3$); IR (film, KBr) $\nu_{\text{max}}$: 2948, 1757, 1473, 1427, 1257, 1192, 1107 cm$^{-1}$; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.70–7.64 (m, 4H), 7.45–7.36 (m, 6H), 4.27 (t, $J = 5.6$ Hz, 2H), 3.60–3.55 (m, 2H), 3.50 (s, 3H), 1.80–1.76 (m, 2H), 1.65–1.56 (m, 2H), 1.12 (s, 9H), 0.90 (s, 9H), 0.04 (s, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 173.6, 136.0, 135.8, 133.4, 133.2, 129.8, 129.7, 127.6, 127.5, 72.5, 62.8, 51.4, 31.6, 27.9, 26.9, 25.9, 19.4, 18.3, −5.3; EIMS ($m/e$): 485, 443, 415, 355, 309, 283, 213, 183, 135, 91, 75. HRMS–ESI: Calcd for C$_{28}$H$_{44}$O$_4$Si$_2$: 501.2856 [M + H]$^+$. Found: 501.2859.

Synthesis of (S)-5-(tert-butyldimethylsilyloxy)-2-(tert-butyldiphenylsilyloxy)pentan-1-ol (9)

To a solution of 8 (1.1 g, 2.1 mmol) in CH$_2$Cl$_2$ at −78 °C Dibal-H (3.0 mL, 1.487 mol/L in toluene, 4.4 mmol) was added. After stirring for 30 min at this temperature, the reaction mixture was then stirred at rt for 2 h. The reaction mixture was then carefully quenched with saturated aqueous NH$_4$Cl, and diluted with AcOEt, filtered, washed...
with brine, dried (Na$_2$SO$_4$) and concentrated in vacuo. Purification by flash chromatography (10% AcOEt in PE) gave the product (854 mg, 86%) as a colorless oil. $[\alpha]_{D}^{20} +26.6$ (c 0.83, CHCl$_3$); IR (film, KBr) $v_{\text{max}}$: 3452, 2931, 1428, 1111, 702 cm$^{-1}$; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.71–7.67 (m, 4H), 7.46–7.37 (m, 6H), 3.82–3.81 (m, 1H), 3.55–3.40 (m, 4H), 1.87 (t, J = 6.4 Hz, 1H), 1.65–1.40 (m, 4H), 1.09 (s, 9H), 0.87 (s, 9H), −0.01 (s, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 135.9, 135.7, 134.0, 133.8, 129.8, 127.8, 127.7, 73.9, 65.9, 63.1, 30.0, 28.3, 27.1, 26.0, 19.4, 18.3, −5.3; EI–MS (m/z): 415, 309, 283, 253, 199, 181, 159, 135. HRMS–ESI: Calcd for C$_{27}$H$_{45}$O$_3$Si$_2$: 473.2907, [M + H]$^+$. Found: 473.2911.

Synthesis of (S)-1-(tert-butyl(1-(tert-butyldimethylsilyloxy)dodec-5-en-4-yloxy)(phenyl)silyl)benzene (11)

To a solution of PCC (630 mg, 3 mmol) in dry CH$_2$Cl$_2$ (30 mL), 9 (709 mg, 1.5 mmol) in CH$_2$Cl$_2$ (10 mL) was added slowly and stirred at rt for 2 h. Purification by FC (1% AcOEt in PE) afforded the aldehyde (664 mg, 94%), which was directly used for the next reaction step. To a solution of $n$-heptylideneriphenylphosphonium bromide (950 mg, 2.1 mmol) in THF (20 mL) at −40 °C, $n$-BuLi (1.0 mL, 2.2 M in hexane, 2.2 mm) was slowly added. The orange colored solution was stirred for 1 h, then aldehyde 10 in THF (5 mL) was added. The mixture was allowed to stir at rt for 12 h and quenched with aqueous NH$_4$Cl. Ether was added, and the mixture was washed with brine, dried with Na$_2$SO$_4$ and concentrated in vacuo. Chromatography of the residue (1% AcOEt in PE) afforded 11 (585 mg, 75%) as a colorless oil. $[\alpha]_{D}^{20} +10.8$ (c 1.1, CHCl$_3$); IR (film, KBr) $v_{\text{max}}$: 2952, 1461, 1427, 1253, 1105, 836 cm$^{-1}$; $^1$H NMR (400 MHz, CDCl$_3$):
$\delta$ 7.68–7.64 (m, 4H), 7.41–7.32 (m, 6H), 5.42–5.34 (m, 1H), 5.21–5.18 (m, 1H), 4.43–4.41 (m, 1H), 3.52–3.48 (m, 2H), 2.04–2.00 (m, 2H), 1.58–1.05 (m, 12H), 1.04 (s, 9H), 0.87 (s, 9H), 0.85 (t, $J = 7.2$ Hz, 3H), 0.05 (s, 6H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 136.0, 135.9, 134.6, 134.5, 132.7, 129.9, 129.4, 129.3, 127.4, 127.3, 69.4, 63.3, 34.7, 31.8, 29.8, 29.0, 28.3, 27.2, 27.02, 25.98, 22.7, 19.3, 18.3, 14.0, −5.3; EIMS (m/z): 441, 415, 309, 283, 253, 235, 199, 181, 159, 135, 115, 91, 73; HRMS–ESI: Calcd for C$_{34}$H$_{56}$O$_2$Si$_2$: 575.3717 [M + Na]$^+$. Found: 575.3717.

Synthesis of (S)-4-(tert-butyldiphenylsilyloxy)dodec-5-en-1-ol (12)

A solution of 11 (420 mg, 0.76 mmol), pyridinium $p$-toluenesulfonate (95 mg, 0.38 mmol) in absolute EtOH (10 mL) was stirred at rt for 24 h. Purification by flash chromatography (CH$_2$Cl$_2$) of the residue obtained after removal of the solvent afforded 12 (326 mg, 98%) as a colorless oil. $\left[\alpha\right]_{D}^{20} +10.5$ (c 0.55, CHCl$_3$); IR (film, KBr) $\nu_{\text{max}}$: 3347, 2933, 1471, 1427, 1107, 1056, 821 cm$^{-1}$; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.67 (t, $J = 8.4$ Hz, 4H), 7.44–7.32 (m, 6H), 5.46–5.41 (m, 1H), 5.37–5.18 (m, 1H), 4.49–4.45 (m, 1H), 3.55–3.51 (m, 2H), 1.87–1.49 (m, 6H), 1.25–1.05 (m, 8H), 1.04 (s, 9H), 0.85 (t, $J = 7.2$ Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 136.1, 136.0, 134.4, 134.1, 132.3, 130.2, 129.6, 129.5, 127.5, 127.3, 69.3, 63.0, 34.7, 31.7, 29.4, 29.0, 28.1, 27.6, 27.0, 22.6, 19.3, 14.0; EIMS (m/z): 381, 339, 303, 269, 254, 199, 181, 165, 139, 123, 81; HRMS–ESI: Calcd for C$_{28}$H$_{42}$O$_2$Si: 461.2852 [M + Na]$^+$. Found: 461.2853.
Synthesis of (5-carboxypentyl)triphenylphosphonium bromide (15)

A solution of ε-caprolactone (1.14 g, 10 mmol) in 5% NaOH (50 mL) was stirred at rt for 12 h. The solution was cooled in an ice bath and acidified with 10% HCl. The aqueous phase was extracted with Et₂O three times. The extracts were washed with brine, dried (Na₂SO₄), concentrated in vacuo to give crude product 13 (1.34 g, 100%), which was directly used for next step. To a solution of 13 (1.34 g, 10 mmol) in CH₂Cl₂ (30 mL), PBr₃ (0.8 mL, 8.4 mmol) in CH₂Cl₂ (10 mL) was added dropwise at 0 °C. The reaction mixture was warmed to 40 °C and stirred for 5 h. The mixture was cooled to rt, then poured into ice water and extracted with CH₂Cl₂ (50 mL). The extracts were washed (brine), dried and concentrated in vacuo to afford crude compound 14 (1.6 g, 82%). A solution of 14 and PPh₃ in CH₃CN was refluxed under N₂ for 12 h. After removal of the solvent, the residue was washed with Et₂O to give 15 as a white solid (3.6 g, 98%) [4,5]. ¹H NMR (400 MHz, D₂O): δ 7.71–7.53 (m, 15H), 3.18–3.10 (m, 2H), 2.17 (t, J = 7.2 Hz, 3H), 1.57–1.36 (m, 6H); ¹³C NMR (100 MHz, D₂O): δ 178.5, 134.9, 133.5, 133.4, 130.0, 128.9, 118.5, 117.7, 33.3, 29.2, 29.0, 23.4, 21.4, 21.3, 20.9.

Synthesis of (S)-10-(tert-butyldiphenylsilyloxy)octadeca-6,11-dienoic acid (17)

To a solution of PCC (210 mg, 1 mmol) in dry CH₂Cl₂ (10 mL), 12 (230 mg, 0.52 mmol) in CH₂Cl₂ (2 mL) was slowly added and stirred at rt for 4 h. Purification by
flash chromatography (5% AcOEt in PE) afforded the aldehyde (220 mg, 96%), which was used for the next reaction step without further purification.

A stirred solution of diisopropylamine (0.29 mL, 2 mmol) in dry THF (2 mL) was treated under N₂ at −30 °C with n-butyllithium (0.91 mL, 2.2 M in hexane). The solution was stirred at −30 °C for 30 min and was added to a suspension of 15 (456 mg, 1 mmol) in dry THF (20 mL). The blood-red solution was stirred for 30 min at rt and treated dropwise with a solution of the above aldehyde in dry THF (2 mL). The mixture was stirred for 3 h. An ice-cold 10% solution of NaHSO₄ (20 mL) was added, and the aqueous phase was extracted with AcOEt. The extracts were washed with brine, dried (Na₂SO₄) and concentrated in vacuo. Column chromatography (50% AcOEt in PE) of the residue afforded acid 17 (192 mg, 72%) as a colorless oil. [α]D²⁰ +8.7 (c 0.69, CHCl₃); IR (film, KBr) νmax: 3005, 2925, 2853, 1711, 1461, 1427, 1289, 1069, 1109, 823 cm⁻¹; ¹H NMR (400 MHz, CDCl₃): δ 7.67 (t, J = 8.0 Hz, 4H), 7.44–7.32 (m, 6H), 5.42–5.36 (m, 1H), 5.32–5.17 (m, 3H), 4.47–4.43 (m, 1H), 2.33 (t, J = 7.6 Hz, 2H), 1.97–1.92 (m, 4H), 1.62–1.05 (m, 16H), 1.03 (s, 9H), 0.85 (t, J = 7.2 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 178.6, 136.1, 136.0, 134.6, 134.4, 132.6, 130.2, 130.1, 129.5, 129.5, 129.4, 127.4, 127.3, 69.4, 38.5, 33.7, 31.7, 29.4, 29.0, 29.0, 27.6, 27.1, 27.0, 24.3, 22.9, 22.6, 19.3, 14.1; HRMS–ESI: Calcd for C₃₄H₆₀O₃Si: 533.3451 [M - H]⁻. Found: 533.3453.
Synthesis of (S)-10-(tert-butyldiphenylsilyloxy)-N-phenethyloctadeca-6,11-dienamide (18)

A solution of 17 (150 mg, 0.28 mmol), 2-phenylethylamine (34 mg, 0.28 mmol), DCC (58 mg, 0.28 mmol) and DMAP (20 mg) in CH₂Cl₂ (10mL) was stirred for 24 h. The precipitated solid was removed by filtration, the filtrate washed with aqueous HCl (2 N), water and brine, and dried (Na₂SO₄). Solvent removal followed by column chromatography of the residue (10% AcOEt in PE) gave the amide (143 mg, 80%) as a colorless oil. [α]²⁰D +9.9 (c 0.39, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ 7.67 (t, J = 6.4 Hz, 4H), 7.40–7.17 (m, 11H), 5.45–5.37 (m, 2H), 5.26–5.21 (m, 3H), 4.43 (m, 1H), 3.51 (q, J = 6.4 Hz, 2H), 2.80 (t, J = 7.2 Hz, 2H), 2.09 (t, J = 7.6 Hz, 2H), 1.97–1.92 (m, 4H), 1.61–1.05 (m, 16H), 1.03 (s, 9H), 0.85 (t, J = 7.2 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 172.9, 138.9, 136.0, 135.9, 134.6, 134.4, 132.6, 130.2, 129.9, 129.5, 129.4, 129.3, 128.8, 128.7, 127.5, 127.3, 126.5, 69.4, 40.5, 38.6, 36.7, 35.8, 31.7, 29.4, 29.3, 29.0, 27.6, 27.1, 27.0, 26.9, 25.4, 22.9, 22.6, 19.3, 14.1; HRMS–ESI: Calcd for C₄₂H₅₉NO₂Si: 636.4237 [M - H]⁻. Found: 636.4208.
Synthesis of \((R)-10-\text{(tert-butyldiphenylsilyloxy)}-N\text{-phenethyloctadecanamide} \) (19)

\[
\begin{align*}
\text{18} & \quad \text{Pd/C, H}_2 \quad \text{19}
\end{align*}
\]

To a solution of the amide 18 (50 mg, 0.078 mmol) in MeOH (20 mL), 10% Pd/C (10 mg) was added and the mixture was hydrogenated for 24 h. It was then filtered through a short pad of Celite and the filter cake was washed with MeOH. The filtrate were concentrated in vacuo to give the product (50 mg, 100%) as a colorless oil. $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.67 (d, $J = 6.8$ Hz, 4H), 7.42–7.18 (m, 11H), 5.46 (br s, 1H), 3.70 (m, 1H), 3.51 (q, $J = 6.4$ Hz, 2H), 2.81 (t, $J = 7.2$ Hz, 2H), 2.10 (t, $J = 7.6$ Hz, 2H), 1.58–1.05 (m, 24H), 1.03 (s, 9H), 0.87 (t, $J = 6.8$ Hz, 3H). $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 173.2, 139.0, 136.0, 134.9, 129.4, 128.8, 128.6, 127.4, 126.5, 73.3, 40.5, 36.9, 36.4, 31.9, 29.7, 29.7, 29.5, 29.4, 29.3, 29.3, 27.1, 25.8, 24.9, 22.7, 19.4, 14.1; HRMS–ESI: Calcd for C$_{42}$H$_{63}$NO$_2$Si: 640.4550 [M - H]$^-$. Found: 640.4527.

Synthesis of \((R)-10\text{-hydroxy-}N\text{-phenethyloctadecanamide} \) (1)

\[
\begin{align*}
\text{19} & \quad \text{TBAF} \quad \text{1}
\end{align*}
\]

A solution of 19 (50 mg, 0.078 mmol) and TBAF (42 mg, 0.16 mmol) in THF (10 mL) was stirred for 24 h. After removal of the solvent, the residue was purified by flash chromatography (30 % EtOAc in PE) to afford \((R)-1 \) (29 mg, 93%) as a white solid. Mp 106–107 °C; $[\alpha]_{D}^{20}$ +37.2 (c 0.85, CHCl$_3$); IR (KBr) $\nu_{\text{max}}$: 3312, 2920, 2846, 1643, 1554 cm$^{-1}$; $^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.32 (t, $J = 7.6$ Hz, 2H), 7.22 (d, $J = 7.6$ Hz, 1H), 7.19 (t, $J = 7.6$ Hz, 2H), 5.39 (br s, 1H), 3.58 (br m, 1H), 3.52 (q, $J = 6.4$ Hz, 2H),
2.82 (t, $J = 6.8$ Hz, 2H), 2.11 (t, $J = 7.2$ Hz, 2H), 1.58–1.05 (m, 24H), 0.88 (t, $J = 6.8$ Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 173.1, 139.0, 128.8, 128.6, 126.5, 72.0, 40.5, 37.5, 37.5, 36.8, 35.7, 31.9, 29.7, 29.6, 29.4, 29.3, 29.2, 25.7, 25.6, 22.7, 14.1; EIMS ($m/z$): 403, 385, 294, 290, 265, 176, 163, 122, 104 (base), 91, 83, 69, 55, 43; HRMS–ESI: Calcd. for C$_{26}$H$_{45}$NO$_2$: 404.3529 [M+H]$^+$. Found: 404.3529.

References

$^1$H NMR and $^{13}$C NMR spectra

BnOOCCOOME

5
$\text{BnOOC} - \text{COOMe}$

5

OH

$\text{BnOOC} - \text{COOMe}$

5

OH
TBSO

COOMe

OTBDPS

8
TBSO\(^{\text{9}}\) OTBDPS
S29
1 (synthesis)
1 (isolation)
1 (isolation)
HMOC of compound 1
COSY of compound 1
HMBC of compound 1