

Supporting Information

for

Solid-Phase Synthesis of 89 Polyamine-Based Cationic Lipids for DNA Delivery to Mammalian Cells

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Compound Library 1.

***N*¹-(Carbamimidoyl)-*N*⁹-(pentanoly)-norspermidine (1.1).**

Yield : (resin: 0.64 mmol/g, 50 mg) 17 mg, 75 %.

*t*_R = 2.1 min (RP-HPLC, 79 % pure); IR (Film): ν = 1663, 1625 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.93 (t, ³*J*(HH) = 7 Hz, 3H; CH₂CH₂CH₃), 1.37 (m, 2H; CH₂CH₂CH₃), 1.61 (quin, ³*J*(HH) = 7 Hz, 2H; CH₂CH₂CH₂CH₃), 1.90 (tt, ³*J*(HH) = 7, 7 Hz, 2H; NHCH₂CH₂CH₂NHCO), 2.01 (tt, ³*J*(HH) = 7, 7 Hz, 2H; NHC(NH₂)HNCH₂CH₂CH₂), 2.24 (t, ³*J*(HH) = 7 Hz, 2H; COCH₂CH₂), 3.03 (t, ³*J*(HH) = 7 Hz, 2H; NHCH₂CH₂CH₂NHCO), 3.08 (t, ³*J*(HH) = 7 Hz, 2H; NHC(NH₂)HNCH₂CH₂CH₂), 3.32 (m, 4H; *m*, CH₂CH₂CH₂NHCH₂CH₂CH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 14.4 (CH₂CH₂CH₃), 23.8 (CH₂CH₂CH₃), 27.2 (NHC(NH₂)HNCH₂CH₂), 28.1 (CH₂CH₂NHCO), 29.5

(CH₂CH₂CH₂CH₃), 37.1 (CH₂NHCO), 37.2 (COCH₂CH₂), 39.9 (NHC(NH₂)HNCH₂), 46.6 (NHC(NH₂)HNCH₂CH₂CH₂), 46.8 (CH₂CH₂CH₂NHCO), 159.2 (NHC(NH₂)NH); 178.0 (C=O amide); MS (ES⁺): m/z (%): 258.1 (33) (M+H)⁺; HRMS (ES⁺): m/z: calcd for C₁₂H₂₈N₅O [M+H]⁺: 258.2289; found: 258.2291.

***N*¹-(Carbamimidoyl)-*N*⁹-(3-methylbutanoyl)-norspermidine (1.2).**

Yield : (resin: 0.64 mmol/g, 50 mg) 20 mg, 89 %.

*t*_R = 2.2 min (RP-HPLC, 99% pure); IR (Film): ν = 1665, 1630 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.82 (d, ³*J*(HH) = 7 Hz, 6H; CH(CH₃)₂), 1.76 (tt, ³*J*(HH) = 7, 7 Hz, 2H; NHCH₂CH₂CH₂NHCO), 1.86 (tt, ³*J*(HH) = 8, 8 Hz, 2H; NHC(NH₂)NHCH₂CH₂CH₂), 1.96 (m, 3H; COCH₂CH), 2.89 (t, ³*J*(HH) = 7 Hz, 2H; NHCH₂CH₂CH₂NHCO), 2.94 (t, ³*J*(HH) = 8 Hz, 2H; NHC(NH₂)NHCH₂CH₂CH₂), 3.18 (m, 4H; CH₂CH₂CH₂NHCH₂CH₂CH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 23.1 (CH(CH₃)₂), 27.2 (NHC(NH₂)NHCH₂CH₂), 27.7 ((CH(CH₃)₂), 28.1 (CH₂CH₂NHCO), 37.2 (CH₂NHCO), 39.9 (NHC(NH₂)NHCH₂), 46.5 (NHC(NH₂)NHCH₂CH₂CH₂), 46.6 (COCH₂CH), 46.9 (CH₂CH₂CH₂NHCO), 159.2 (NHC(NH₂)NH), 177.2 (C=O amide); MS (ES⁺): m/z (%): 258.2 (95) (M+H)⁺; HRMS (ES⁺): m/z: calcd for C₁₂H₂₈N₅O [M+H]⁺: 258.2289; found: 258.2291.

***N*¹-(Carbamimidoyl)-*N*⁹-(2,2-dimethylpropanoyl)-norspermidine (1.3).**

Yield : (resin: 0.64 mmol/g, 50 mg) 19 mg, 84 %.

*t*_R = 2.1 min (RP-HPLC, 96 % pure); IR (Film): ν = 1667, 1624 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 1.20 (s, 9H; C(CH₃)₃), 1.90 (tt, ³*J*(HH) = 7, 7 Hz, 2H; NHCH₂CH₂CH₂NHCO), 2.01 (tt, ³*J*(HH) = 8, 8 Hz, 2H; NHC(NH₂)NHCH₂CH₂CH₂), 2.99 (t, ³*J*(HH) = 7 Hz, 2H; NHCH₂CH₂CH₂NHCO), 3.08 (t, ³*J*(HH) = 8 Hz, 2H; NHC(NH₂)NHCH₂CH₂CH₂), 3.33 (m, 4H; CH₂CH₂CH₂NHCH₂CH₂CH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 25.1 (NHC(NH₂)NHCH₂CH₂), 26.7 (CH₂CH₂NHCO, C(CH₃)₃), 35.7 (CH₂NHCO), 38.4 (NHC(NH₂)NHCH₂), 38.6 (C(CH₃)₃), 45.1 (NHC(NH₂)NHCH₂CH₂CH₂), 45.2 (CH₂CH₂CH₂NHCO), 157.7 (NHC(NH₂)NH), 181.8 (C=O amide); MS (ES⁺): m/z (%): 258.1 (17) (M+H)⁺; HRMS (ES⁺): m/z: calcd for C₁₂H₂₈N₅O [M+H]⁺: 258.2289; found: 258.2291.

***N*¹-(Carbamimidoyl)-*N*⁹-(hexanoyl)-norspermidine (1.4).**

Yield : (resin: 0.64 mmol/g, 50 mg) 17.7 mg, 76 %.

t_R = 2.1 min (RP-HPLC, 98 % pure); IR (Film): ν = 1666, 1630 cm^{-1} (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.79 (t, ³*J*(HH) = 7 Hz, 3H; CH₂CH₂CH₃), 1.20 (m, 4H; CH₂CH₂CH₃), 1.49 (quin, ³*J*(HH) = 8 Hz, 2H; COCH₂CH₂CH₂), 1.75 (tt, ³*J*(HH) = 7 Hz, 2H; NHCH₂CH₂CH₂NHCO), 1.86 (tt, ³*J*(HH) = 7, 7 Hz, 2H; NHC(NH₂)HNCH₂CH₂CH₂), 2.09 (t, ³*J*(HH) = 8 Hz, 2H; COCH₂CH₂), 2.89 (t, ³*J*(HH) = 7 Hz, 2H; NHCH₂CH₂CH₂NHCO), 2.94 (t, ³*J*(HH) = 8 Hz, 2H; NHC(NH₂)HNCH₂CH₂CH₂), 3.17 (m, 4H; CH₂CH₂CH₂NHCH₂CH₂CH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 14.6 (CH₂CH₂CH₃), 23.8 (CH₂CH₂CH₃), 27.0 (NHC(NH₂)HNCH₂CH₂), 27.2 (COCH₂CH₂), 28.1 (CH₂CH₂NHCO), 32.9 (CH₂CH₂CH₂CH₃), 37.1 (CH₂NHCO), 37.3 (COCH₂CH₂), 39.9 (NHC(NH₂)HNCH₂), 46.6 (NHC(NH₂)HNCH₂CH₂CH₂), 46.8 (CH₂CH₂CH₂NHCO), 159.2 (HNC(NH₂)NH), 178.0 (C=O amide); MS (ES⁺): *m/z* (%): 272.2 (22) (M+H)⁺; HRMS (ES⁺): *m/z*: calcd for C₁₃H₃₀N₅O [M+H]⁺: 272.2445; found: 272.2444.

***N*¹-(Carbamimidoyl)-*N*⁹-(2-methylpentanoyl)-norspermidine (1.5).**

Yield : (resin: 0.64 mmol/g, 50 mg) 20 mg, 85 %.

t_R = 2.3 min (RP-HPLC, 100 % pure); IR (Film): ν = 1663, 1638 cm^{-1} (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.93 (t, ³*J*(HH) = 7 Hz, 3H; CH₂CH₃), 1.13 (d, ³*J*(HH) = 7 Hz, 3H; COCH(CH₃)CH₂), 1.33, 1.60 (m, 4H; CH₂CH₂CH₃), 1.90 (tt, ³*J*(HH) = 7, 7 Hz, 2H; NHCH₂CH₂CH₂NHCO), 2.01 (tt, ³*J*(HH) = 8, 8 Hz, 2H; NHC(NH₂)NHCH₂CH₂CH₂), 2.35 (m, 1H; COCH(CH₃)CH₂), 3.02 (t, ³*J*(HH) = 7 Hz, 2H; NHCH₂CH₂CH₂NHCO), 3.08 (t, ³*J*(HH) = 8 Hz, 2H; NHC(NH₂)NHCH₂CH₂CH₂), 3.32 (m, 4H; CH₂CH₂CH₂NHCH₂CH₂CH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 14.7 (CH₂CH₂CH₃), 18.7 (COCH(CH₃)CH₂), 22.1 (CH₂CH₂CH₃), 27.2 (NHC(NH₂)NHCH₂CH₂), 28.1 (CH₂CH₂NHCO), 37.2 (CH₂NHCO), 37.9 (COCH(CH₃)CH₂), 39.9 (NHC(NH₂)NHCH₂), 42.2 (COCH(CH₃)CH₂), 46.6 (NHC(NH₂)NHCH₂CH₂CH₂), 46.8 (CH₂CH₂CH₂NHCO), 159.2 (NHC(NH₂)NH), 181.4 (C=O amide); MS (ES⁺): *m/z* (%): 272.1 (60) (M+H)⁺; HRMS (ES⁺): *m/z*: calcd for C₁₃H₃₀N₅O [M+H]⁺: 272.2445; found: 272.2444.

***N*¹-(Carbamimidoyl)-*N*⁹-(3-methylpentanoyl)-norspermidine (1.6).**

Yield : (resin: 0.64 mmol/g, 50 mg) 17 mg, 73 %.

$t_R = 2.3$ min (RP-HPLC, 93 % pure); IR (Film): $\nu = 1663, 1630$ cm^{-1} (C=O); ¹H NMR (400 MHz, CD₃OD) $\delta = 0.79$ (m, 6H; CH(CH₃)CH₂CH₃), 1.10, 1.24 (m, 2H; CH(CH₃)CH₂CH₃), 1.76 (m, 3H; *m*, CH(CH₃)CH₂CH₃, NHCH₂CH₂CH₂NHCO), 1.86 (m, 3H; COCHH, NHC(NH₂)NHCH₂CH₂CH₂), 2.10 (m, 1H; COCHH), 2.90 (m, 4H; CH₂NHCH₂), 3.17 (m, 4H; CH₂CH₂CH₂NHCH₂CH₂CH₂); ¹³C NMR (100 MHz, CD₃OD) $\delta = 12.0$ (CH₂CH₃), 19.8 (CH(CH₃)CH₂CH₃), 27.2 (NHC(NH₂)NHCH₂CH₂), 28.1 (CH₂CH₂NHCO), 30.9 (CH₂CH₃), 34.0 (CH(CH₃)CH₂), 37.2 (CH₂NHCO), 39.9 (NHC(NH₂)NHCH₂), 44.6 (COCH₂), 46.6 (NHC(NH₂)NHCH₂CH₂CH₂), 46.9 (CH₂CH₂CH₂NHCO), 159.2 (NHC(NH₂)NH), 177.4 (C=O amide); MS (ES⁺): *m/z* (%): 272.2 (54) (M+H)⁺; HRMS (ES⁺): *m/z*: calcd for C₁₃H₃₀N₅O [M+H]⁺: 272.2445; found: 272.2444.

***N*¹-(Carbamimidoyl)-*N*⁹-(4-methylpentanoyl)-norspermidine (1.7).**

Yield : (resin: 0.64 mmol/g, 50 mg) 17 mg, 73 %.

$t_R = 2.3$ min (RP-HPLC, 94 % pure); IR (Film): $\nu = 1667, 1630$ cm^{-1} (C=O); ¹H NMR (400 MHz, CD₃OD) $\delta = 0.94$ (m, 6H; CH(CH₃)₂), 1.54 (m, 3H; CH₂CH(CH₃)₂), 1.90 (tt, ³*J*(HH) = 7, 7 Hz, 2H; NHCH₂CH₂CH₂NHCO), 2.01 (tt, ³*J*(HH) = 8, 8 Hz, 2H; NHC(NH₂)NHCH₂CH₂CH₂), 2.25 (t, ³*J*(HH) = 8 Hz, 2H; COCH₂), 3.03 (t, ³*J*(HH) = 7 Hz, 2H; NHCH₂CH₂CH₂NHCO), 3.08 (t, ³*J*(HH) = 8 Hz, 2H; NHC(NH₂)NHCH₂CH₂CH₂), 3.17 (m, 4H; CH₂CH₂CH₂NHCH₂CH₂CH₂); ¹³C NMR (100 MHz, CD₃OD) $\delta = 23.0$ (CH(CH₃)₂), 27.2 (NHC(NH₂)NHCH₂CH₂), 28.1 (CH₂CH₂NHCO), 29.4 (CH(CH₃)₂), 35.4 (COCH₂), 36.4 (CH₂NHCO), 37.2 (COCH₂CH₂), 39.9 (NHC(NH₂)NHCH₂), 46.6 (NHC(NH₂)NHCH₂CH₂CH₂), 46.8 (CH₂CH₂CH₂NHCO), 159.2 (NHC(NH₂)NH), 178.2 (C=O amide); MS (ES⁺): *m/z* (%): 272.2 (87) (M+H)⁺; HRMS (ES⁺): *m/z*: calcd for C₁₃H₃₀N₅O [M+H]⁺: 272.2445; found: 272.2445.

***N*¹-(Carbamimidoyl)-*N*⁹-(2-ethylbutanoyl)-norspermidine (1.8).**

Yield : (resin: 0.64 mmol/g, 50 mg) 19 mg, 82 %.

*t*_R = 2.3 min (RP-HPLC, 96 % pure); IR (Film): ν = 1667, 1636 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.75 (t, ³*J*(HH) = 7 Hz, 6H; CH(CH₂CH₃)₂), 1.39 (m, 4H; CH(CH₂CH₃)₂), 1.76 (tt, ³*J*(HH) = 7, 7 Hz, 2H; NHCH₂CH₂CH₂NHCO), 1.85 (m, 3H; COCH, NHC(NH₂)NHCH₂CH₂CH₂), 2.28 (m, 2H; NHCH₂CH₂CH₂NHCO), 2.93 (m, 2H; NHC(NH₂)NHCH₂CH₂CH₂), 3.17 (m, 4H; CH₂CH₂CH₂NHCH₂CH₂CH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 12.8 (CH(CH₂CH₃), 27.1 (NHC(NH₂)NHCH₂CH₂, CH(CH₂CH₃), 28.2 (CH₂CH₂NHCO), 37.1 (CH₂NHCO), 39.9 (NHC(NH₂)NHCH₂), 46.6 (NHC(NH₂)NHCH₂CH₂CH₂), 47.0 (CH₂CH₂CH₂NHCO), 52.2 (COCH), 159.2 (NHC(NH₂)NH), 180.3 (C=O amide); MS (ES⁺): *m/z* (%): 272.1 (82) (M+H)⁺; HRMS (ES⁺): *m/z*: calcd for C₁₃H₃₀N₅O [M+H]⁺: 272.2445; found: 272.2447.

***N*¹-(Carbamimidoyl)-*N*⁹-(3,3-dimethylbutanoyl)-norspermidine (1.9).**

Yield : (resin: 0.64 mmol/g, 50 mg) 19 mg, 82 %.

*t*_R = 2.3 min (RP-HPLC, 96 % pure); IR (Film): ν = 1663, 1637 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 1.03 (s, 9H; C(CH₃)₃), 1.90 (tt, ³*J*(HH) = 8, 8 Hz, 2H; CH₂CH₂CH₂NHCO), 2.01 (tt, ³*J*(HH) = 8, 8 Hz, 2H; NHC(NH₂)HNCH₂CH₂CH₂), 2.12 (s, 2H; COCH₂), 3.04 (m, 2H; NHCH₂CH₂CH₂NHCO), 3.09 (m, 2H; NHC(NH₂)NHCH₂CH₂CH₂), 3.32 (m, 4H; CH₂CH₂CH₂NHCH₂CH₂CH₂); ¹³C NMR (100 MHz, CD₃OD); δ = 27.2 (NHC(NH₂)NHCH₂CH₂), 28.1 (CH₂CH₂NHCO), 30.6 (C(CH₃)₃), 32.1 (C(CH₃)₃), 37.1 (CH₂NHCO), 39.9 (NHC(NH₂)NHCH₂), 46.6 (NHC(NH₂)NHCH₂CH₂CH₂), 46.9 (CH₂CH₂CH₂NHCO), 50.9 (COCH₂), 159.2 (NHC(NH₂)NH); 176.3 (C=O amide); MS (ES⁺): *m/z* (%): 272.2 (42) (M+H)⁺; HRMS (ES⁺): *m/z*: calcd for C₁₃H₃₀N₅O [M+H]⁺: 272.2445; found: 272.2444.

***N*¹-(Carbamimidoyl)-*N*⁹-(heptanoyl)-norspermidine (1.10).**

Yield : (resin: 0.64 mmol/g, 50 mg) 18 mg, 78 %.

$t_R = 6.1$ min (RP-HPLC, 89 % pure); IR (Film): $\nu = 1665, 1630$ cm^{-1} (C=O); ¹H NMR (400 MHz, CD₃OD) $\delta = 0.92$ (m, 3H; CH₂CH₃), 1.33 (br s, 6H; CH₂CH₂CH₂CH₃), 1.63 (m, 2H; COCH₂CH₂), 1.90 (tt, ³*J*(HH) = 7, 7 Hz, 2H; CH₂CH₂NHCO), 2.01 (m, 2H; NHC(NH₂)NHCH₂CH₂), 2.24 (t, ³*J*(HH) = 8 Hz, 2H; COCH₂CH₂), 3.03 (m, 2H; CH₂CH₂CH₂NHCO), 3.08 (m, 2H; NHC(NH₂)NHCH₂CH₂CH₂), 3.32 (m, 4H; CH₂CH₂CH₂NHCH₂CH₂CH₂); ¹³C NMR (100 MHz, CD₃OD) $\delta = 14.7$ (CH₂CH₃), 23.9 (CH₂CH₂CH₃), 27.2 (NHC(NH₂)NHCH₂CH₂), 27.3 (COCH₂CH₂), 28.1 (CH₂CH₂NHCO), 30.4 (CH₂CH₂CH₃), 33.0 (COCH₂CH₂CH₂), 37.1 (CH₂NHCO), 37.4 (COCH₂), 39.9 (NHC(NH₂)NHCH₂), 46.6 (NHC(NH₂)NHCH₂CH₂CH₂), 46.8 (CH₂CH₂CH₂NHCO), 159.2 (NHC(NH₂)NH); 178.0 (C=O amide); MS (ES⁺): *m/z* (%): 286.2 (52) (M+H)⁺; HRMS (ES⁺): *m/z*: calcd for C₁₄H₃₂N₅O [M+H]⁺: 286.2602; found: 286.2602.

***N*¹-(Carbamimidoyl)-*N*⁹-(2-methylhexanoyl)-norspermidine (1.11).**

Yield : (resin: 0.64 mmol/g, 50 mg) 16 mg, 68 %.

$t_R = 5.7$ min (RP-HPLC, 76 % pure); IR (Film): $\nu = 1663, 1638$ cm^{-1} (C=O); ¹H NMR (400 MHz, CD₃OD) $\delta = 0.92$ (t, ³*J*(HH) = 7 Hz, 3H; CH₂CH₃), 1.13 (d, ³*J*(HH) = 7 Hz, 3H; CH(CH₃)CH₂), 1.34 (m, 5H; *m*, CH(H)CH₂CH₂CH₃), 1.61 (m, 1H; CH(H)CH₂CH₂CH₃), 1.91 (tt, ³*J*(HH) = 7, 7 Hz, 2H; CH₂CH₂CH₂NHCO), 2.01 (tt, ³*J*(HH) = 8, 8 Hz, 2H; NHC(NH₂)NHCH₂CH₂CH₂), 2.33 (m, 1H; COCH), 3.02 (m, 2H; CH₂CH₂CH₂NHCO), 3.08 (t, ³*J*(HH) = 8 Hz, 2H; NHC(NH₂)NHCH₂CH₂CH₂), 3.33 (m, 4H; CH₂CH₂CH₂NHCH₂CH₂CH₂); ¹³C NMR (100 MHz, CD₃OD) $\delta = 14.7$ (CH₂CH₃), 18.8 (CH(CH₃)CH₂), 24.0 (CH₂CH₃), 27.2 (NHC(NH₂)NHCH₂CH₂), 28.1 (CH₂CH₂NHCO), 31.2 (CH₂CH₂CH₃), 35.4 (CH(CH₃)CH₂), 37.1 (CH₂NHCO), 39.9 (NHC(NH₂)NHCH₂), 42.5 (CH(CH₃)CH₂), 46.6 (NHC(NH₂)NHCH₂CH₂CH₂), 46.8 (CH₂CH₂CH₂NHCO), 159.2 (NHC(NH₂)NH), 181.4 (C=O amide); MS (ES⁺): *m/z* (%): 286.2 (100) (M+H)⁺; HRMS (ES⁺): *m/z*: calcd for C₁₄H₃₂N₅O [M+H]⁺: 286.2602; found: 286.2600.

***N*¹-(Carbamimidoyl)-*N*⁹-(5-methylhexanoyl)-norspermidine (1.12).**

Yield : (resin: 0.64 mmol/g, 50 mg) 17 mg, 72 %.

*t*_R = 5.8 min (RP-HPLC, 84 % pure); IR (Film): ν = 1663, 1638 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.92 (d, ³*J*(HH) = 7 Hz, 6H; CH(CH₃)₂), 1.22 (m, 2H; CH₂CH(CH₃)₂), 1.59 (m, 1H; CH₂CH(CH₃)₂), 1.64 (m, 2H; COCH₂CH₂), 1.90 (tt, ³*J*(HH) = 7, 7 Hz, 2H; CH₂CH₂CH₂NHCO), 2.01 (tt, ³*J*(HH) = 8, 8 Hz, 2H; NHC(NH₂)NHCH₂CH₂CH₂), 2.22 (t, ³*J*(HH) = 8 Hz, 2H; COCH₂CH₂), 3.03 (t, ³*J*(HH) = 7 Hz, 2H; CH₂CH₂CH₂NHCO), 3.09 (t, ³*J*(HH) = 8 Hz, 2H; NHC(NH₂)NHCH₂CH₂CH₂), 3.32 (m, 4H; CH₂CH₂CH₂NHCH₂CH₂CH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 23.3 (CH(CH₃)₂), 25.2 (COCH₂CH₂), 27.2 (NHC(NH₂)NHCH₂CH₂), 28.1 (CH₂CH₂NHCO), 29.3 (CH₂CH(CH₃)₂), 37.2 (CH₂NHCO), 37.6 (COCH₂), 39.9 (NHC(NH₂)NHCH₂), 40.0 (CH₂CH(CH₃)₂), 46.6 (NHC(NH₂)NHCH₂CH₂CH₂), 46.8 (CH₂CH₂CH₂NHCO), 159.2 (NHC(NH₂)NH), 178.0 (C=O amide); MS (ES⁺): *m/z* (%): 286.2 (37) (M+H)⁺; HRMS (ES⁺): *m/z*: calcd for C₁₄H₃₂N₅O [M+H]⁺: 286.2602; found: 286.2602.

***N*¹-(Carbamimidoyl)-*N*⁹-(octanoyl)-norspermidine (1.13).**

Yield : (resin: 0.64 mmol/g, 200 mg) 61 mg, 63 %.

*t*_R = 6.8 min (RP-HPLC, 95 % pure); IR (Film): ν = 1661, 1616 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.92 (t, ³*J*(HH) = 7 Hz, 3H; CH₂CH₃), 1.33 (m, 8H; CH₂(CH₂)₄CH₃), 1.63 (m, 2H; COCH₂CH₂), 1.90 (tt, ³*J*(HH) = 7, 7 Hz, 2H; CH₂CH₂CH₂NHCO), 2.01 (tt, ³*J*(HH) = 8, 8 Hz, 2H; NHC(NH₂)NHCH₂CH₂CH₂), 2.24 (t, ³*J*(HH) = 8 Hz, 2H; COCH₂CH₂), 3.03 (t, ³*J*(HH) = 7 Hz, 2H; CH₂CH₂CH₂NHCO), 3.09 (t, ³*J*(HH) = 8 Hz, 2H; NHC(NH₂)NHCH₂CH₂CH₂), 3.32 (m, 4H; CH₂CH₂CH₂NHCH₂CH₂CH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 14.8 (CH₂CH₃), 24.0 (CH₂CH₃), 27.2 (NHC(NH₂)NHCH₂CH₂), 27.4 (COCH₂CH₂), 28.1 (CH₂CH₂NHCO), 30.5 (CH₂CH₂CH₂CH₃), 30.7 (COCH₂CH₂CH₂), 33.2 (CH₂CH₂CH₃), 37.2 (CH₂NHCO), 37.4 (COCH₂), 39.9 (NHC(NH₂)NHCH₂), 46.6 (NHC(NH₂)NHCH₂CH₂CH₂), 46.8 (CH₂CH₂CH₂NHCO), 159.2 (NHC(NH₂)NH), 178.0 (C=O amide); MS (ES⁺): *m/z* (%): 300.3 (70) (M+H)⁺; HRMS (ES⁺): *m/z*: calcd for C₁₅H₃₄N₅O [M+H]⁺: 300.2758; found: 300.2761.

***N*¹-(Carbamimidoyl)-*N*⁹-(2-methylheptanoyl)-norspermidine (1.14).**

Yield : (resin: 0.64 mmol/g, 200 mg) 59 mg, 62 %.

*t*_R = 6.4 min (RP-HPLC, 93 % pure); IR (Film): ν = 1665, 1630 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.92 (t, ³*J*(HH) = 7 Hz, 3H; CH₂CH₃), 1.13 (d, ³*J*(HH) = 7 Hz, 3H; COCH(CH₃)CH₂), 1.32 (m, 7H; CH(CH₃)CH(H)CH₂CH₂CH₂), 1.62 (m, 1H; CH(CH₃)CH(H)CH₂), 1.91 (tt, ³*J*(HH) = 7, 7 Hz, 2H; CH₂CH₂CH₂NHCO), 2.01 (tt, ³*J*(HH) = 8, 8 Hz, 2H; NHC(NH₂)NHCH₂CH₂CH₂), 2.33 (m, 1H; COCH), 3.03 (t, ³*J*(HH) = 7 Hz, 2H; CH₂CH₂CH₂NHCO), 3.09 (t, ³*J*(HH) = 8 Hz, 2H; NHC(NH₂)NHCH₂CH₂CH₂), 3.32 (m, 4H; CH₂CH₂CH₂NHCH₂CH₂CH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 14.7 (CH₂CH₃), 18.8 (CH(CH₃)CH₂), 24.0 (CH₂CH₃), 27.2 (NHC(NH₂)NHCH₂CH₂), 28.1 (CH₂CH₂NHCO), 28.7 (CH₂CH₂CH₂CH₃), 33.3 (CH₂CH₂CH₃), 35.6 (CH(CH₃)CH₂), 37.1 (CH₂NHCO), 39.9 (NHC(NH₂)NHCH₂), 42.5 (COCH), 46.6 (NHC(NH₂)NHCH₂CH₂CH₂), 46.9 (CH₂CH₂CH₂NHCO), 159.2 (NHC(NH₂)NH), 181.3 (C=O amide); MS (ES⁺): *m/z* (%): 300.3 (40) (M+H)⁺; HRMS (ES⁺): *m/z*: calcd for C₁₅H₃₄N₅O [M+H]⁺: 300.2758; found: 300.2760.

***N*¹-(Carbamimidoyl)-*N*⁹-(2-ethylhexanoyl)-norspermidine (1.15).**

Yield : (resin: 0.64 mmol/g, 100 mg) 44 mg, 92 %.

*t*_R = 6.3 min (RP-HPLC, 98 % pure); IR (Film): ν = 1665, 1637 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.91 (m, 6H; CH(CH₂CH₃) + CH₂CH₂CH₃), 1.30 (m, 3H; CH(H)CH₂CH₃), 1.45, 1.57 (m, 5H; *m*, CH(H)CH₂CH₃ + CH(CH₂CH₃)), 1.92 (tt, ³*J*(HH) = 7, 7 Hz, 2H; CH₂CH₂CH₂NHCO), 2.01 (m, 2H; NHC(NH₂)NHCH₂CH₂CH₂), 2.13 (m, 1H; COCH), 3.04 (t, ³*J*(HH) = 7 Hz, 2H; CH₂CH₂CH₂NHCO), 3.09 (t, ³*J*(HH) = 8 Hz, 2H; NHC(NH₂)NHCH₂CH₂CH₂), 3.33 (m, 4H; CH₂CH₂CH₂NHCH₂CH₂CH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 12.8 (CH₂CH₂CH₃), 14.7 (CH(CH₂CH₃)), 24.1 (CH₂CH₂CH₃), 27.2 (NHC(NH₂)NHCH₂CH₂), 27.5 (CH₂CH₂CH₃), 28.1 (CH₂CH₂NHCO), 31.4 (CH₂CH₂CH₂CH₃), 33.9 (CH(CH₂CH₃)), 37.2 (CH₂NHCO), 39.9 (NHC(NH₂)NHCH₂), 46.6 (NHC(NH₂)NHCH₂CH₂CH₂), 47.0 (CH₂CH₂CH₂NHCO), 50.4 (COCH), 159.2 (NHC(NH₂)NH), 180.5 (C=O amide); MS (ES⁺): *m/z* (%): 300.3 (15) (M+H)⁺; HRMS (ES⁺): *m/z*: calcd for C₁₅H₃₄N₅O [M+H]⁺: 300.2758; found: 300.2757.

***N*¹-(Carbamimidoyl)-*N*⁹-(2-propylpentanoyl)-norspermidine (1.16).**

Yield : (resin: 0.64 mmol/g, 200 mg) 50 mg, 52 %.

t_R = 5.8 min (RP-HPLC, 88 % pure); IR (Film): ν = 1670, 1629 cm^{-1} (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.93 (t, ³*J*(HH) = 7 Hz, 6H; CH(CH₂CH₂CH₃)₂), 1.31 (m, 4H; CH(CH₂CH₂CH₃)₂), 1.40 (m, 2H; CH(CH(H)CH₂CH₃)₂), 1.57 (m, 2H; CH(CH(H)CH₂CH₃)₂), 1.91 (tt, ³*J*(HH) = 7, 7 Hz, 2H; CH₂CH₂CH₂NHCO), 2.01 (tt, ³*J*(HH) = 8, 8 Hz, 2H; NHC(NH₂)NHCH₂CH₂CH₂), 2.25 (m, 1H; COCH), 3.03 (t, ³*J*(HH) = 7 Hz, 2H; CH₂CH₂CH₂NHCO), 3.09 (t, ³*J*(HH) = 8 Hz, 2H; NHC(NH₂)NHCH₂CH₂CH₂), 3.32 (m, 4H; CH₂CH₂CH₂NHCH₂CH₂CH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 14.8 (CH(CH₂CH₂CH₃)₂), 22.2 (CH(CH₂CH₂CH₃)₂), 27.2 (NHC(NH₂)NHCH₂CH₂), 28.1 (CH₂CH₂NHCO), 36.7 (CH(CH₂CH₂CH₃) + CH₂NHCO), 37.2 (CH(CH₂CH₂CH₃)), 39.9 (NHC(NH₂)NHCH₂), 46.6 (NHC(NH₂)NHCH₂CH₂CH₂), 47.0 (CH₂CH₂CH₂NHCO), 48.2 (COCH), 159.2 (NHC(NH₂)NH), 180.5 (C=O amide); MS (ES⁺): *m/z* (%): 300.2 (100) (M+H)⁺; HRMS (ES⁺): *m/z*: calcd for C₁₅H₃₄N₅O [M+H]⁺: 300.2758; found: 300.2763.

***N*¹-(Carbamimidoyl)-*N*⁹-(nonanoyl)-norspermidine (1.17).**

Yield : (resin: 0.64 mmol/g, 200 mg) 34 mg, 35 %.

t_R = 7.3 min (RP-HPLC, 90 % pure); IR (Film): ν = 1665, 1629 cm^{-1} (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.80 (t, ³*J*(HH) = 7 Hz, 3H; CH₂CH₂CH₃), 1.21 (br s, 10H; CH₂(CH₂)₅CH₃), 1.51 (m, 2H; CH₂(CH₂)₅CH₃), 1.78 (tt, ³*J*(HH) = 7, 7 Hz, 2H; CH₂CH₂CH₂NHCO), 1.89 (m, 2H; NHC(NH₂)NHCH₂CH₂CH₂), 2.12 (t, ³*J*(HH) = 7 Hz, COCH₂), 2.91 (t, ³*J*(HH) = 7 Hz, 2H; CH₂CH₂CH₂NHCO), 2.97 (m, 2H; NHC(NH₂)NHCH₂CH₂CH₂), 3.21 (m, 4H; CH₂CH₂CH₂NHCH₂CH₂CH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 14.8 (CH₂CH₃), 24.1 (CH₂CH₃), 27.2 (COCH₂CH₂), 27.4 (NHC(NH₂)NHCH₂CH₂), 28.2 (CH₂CH₂NHCO), 30.7, 30.8, (COCH₂CH₂(CH₂)₃CH₂), 33.4 (CH₂CH₂CH₃), 37.1 (CH₂NHCO), 37.4 (COCH₂), 39.9 (NHC(NH₂)NHCH₂), 46.4 (NHC(NH₂)NHCH₂CH₂CH₂), 46.8 (CH₂CH₂CH₂NHCO), 159.2 (NHC(NH₂)NH), 178.0 (C=O amide); MS (ES⁺): *m/z* (%): 314.2 (27) (M+H)⁺; HRMS (ES⁺): *m/z*: calcd for C₁₆H₃₆N₅O [M+H]⁺: 314.2915; found: 314.2908.

***N*¹-(Carbamimidoyl)-*N*⁹-(4-methyloctanoyl)-norspermidine (1.18).**

Yield : (resin: 0.64 mmol/g, 200 mg) 44 mg, 45 %.

$t_R = 7.1$ min (RP-HPLC, 93 % pure); IR (Film): $\nu = 1665, 1629$ cm^{-1} (C=O); ¹H NMR (400 MHz, CD₃OD) $\delta = 0.81$ (m, 6H; CH₂CH₃ + CHCH₃), 1.21 (m, 6H; CH(CH₃)CH₂CH₂CH₂CH₃), 1.32 (m, 2H; CH(H)CH(CH₃)CH₂), 1.55 (m, 1H, CH(H)CH(CH₃)CH₂), 1.78 (tt, ³J(HH) = 7, 7 Hz, 2H; CH₂CH₂CH₂NHCO), 1.89 (m, 2H; NHC(NH₂)NHCH₂CH₂CH₂), 2.13 (m, 2H; COCH₂), 2.91 (t, ³J(HH) = 7 Hz, 2H; CH₂CH₂CH₂NHCO), 2.97 (t, ³J(HH) = 8 Hz, 2H; NHC(NH₂)NHCH₂CH₂CH₂), 3.21 (m, 4H; CH₂CH₂CH₂NHCH₂CH₂CH₂); ¹³C NMR (100 MHz, CD₃OD) $\delta = 14.8$ (CH₂CH₃), 20.1 (CHCH₃), 24.4 (CH₂CH₃), 27.2 (NHC(NH₂)NHCH₂CH₂), 28.2 (CH₂CH₂NHCO), 30.7, 34.2, 34.5, 35.2 (CH₂CH(CH₃)CH₂CH₂C₂H₅), 37.1 (CH₂NHCO), 37.9 (COCH₂), 39.9 (NHC(NH₂)NHCH₂), 46.6 (NHC(NH₂)NHCH₂CH₂CH₂), 46.8 (CH₂CH₂CH₂NHCO), 159.2 (NHC(NH₂)NH), 178.5 (C=O amide); MS (ES⁺): m/z (%): 314.2 (45) (M+H)⁺; HRMS (ES⁺): m/z: calcd for C₁₆H₃₆N₅O [M+H]⁺: 314.2915; found: 314.2917.

***N*¹-(Carbamimidoyl)-*N*⁹-(decanoyl)-norspermidine (1.19).**

Yield : (resin: 0.64 mmol/g, 50 mg) 20 mg, 80 %.

$t_R = 7.7$ min (RP-HPLC, 99 % pure); IR (Film): $\nu = 1663, 1637$ cm^{-1} (C=O); ¹H NMR (400 MHz, CD₃OD) $\delta = 0.91$ (t, ³J(HH) = 7 Hz, 3H; CH₂CH₃), 1.32 (s, 12H; CH₂(CH₂)₆CH₃), 1.62 (tt, ³J(HH) = 7, 7 Hz, 2H; COCH₂CH₂), 1.90 (tt, ³J(HH) = 7, 7 Hz, 2H; CH₂CH₂CH₂NHCO), 2.00 (tt, ³J(HH) = 8, 8 Hz, 2H; NHC(NH₂)NHCH₂CH₂CH₂), 2.24 (t, ³J(HH) = 7 Hz, 2H; COCH₂), 3.03 (t, ³J(HH) = 7 Hz, 2H; CH₂CH₂CH₂NHCO), 3.08 (t, ³J(HH) = 8 Hz, 2H; NHC(NH₂)NHCH₂CH₂CH₂), 3.32 (m, 4H; CH₂CH₂CH₂NHCH₂CH₂CH₂); ¹³C NMR (100 MHz, CD₃OD) $\delta = 14.8$ (CH₂CH₃), 24.1 (CH₂CH₃), 27.2 (NHC(NH₂)NHCH₂CH₂), 27.4 (COCH₂CH₂), 28.1 (CH₂CH₂NHCO), 30.7, 30.8, 31.0 (CH₂(CH₂)₄CH₂CH₂CH₃), 33.4 (CH₂CH₂CH₃), 37.1 (CH₂NHCO), 37.4 (COCH₂), 39.9 (NHC(NH₂)NHCH₂), 46.6 (NHC(NH₂)NHCH₂CH₂CH₂), 46.8 (CH₂CH₂CH₂NHCO), 159.2 (NHC(NH₂)NH), 178.0 (C=O amide); MS (ES⁺): m/z (%): 328.4 (33) (M+H)⁺; HRMS (ES⁺): m/z: calcd for C₁₇H₃₈N₅O [M+H]⁺: 328.3071; found: 328.3063.

***N*¹-(Carbamimidoyl)-*N*⁹-(4-methylnonanoyl)-norspermidine (1.20).**

Yield : (resin: 0.64 mmol/g, 200 mg) 31 mg, 31 %.

$t_R = 7.6$ min (RP-HPLC, 93 % pure); IR (Film): $\nu = 1665, 1629$ cm^{-1} (C=O); ¹H NMR (400 MHz, CD₃OD) $\delta = 0.92$ (m, 6H; CH₂CH₃ + CHCH₃), 1.16, 1.32 (m, 8H; CH₂(CH₂)₃CH₃), 1.43 (m, 2H; CH(H)CH(CH₃)), 1.66 (m, 1H; CH(H)CH(CH₃)), 1.90 (tt, ³J(HH) = 7, 7 Hz, 2H; CH₂CH₂CH₂NHCO), 2.01 (m, 2H; NHC(NH₂)NHCH₂CH₂), 2.25 (m, 2H; COCH₂), 3.03 (m, 2H; CH₂CH₂CH₂NHCO), 3.09 (m, 2H; NHC(NH₂)NHCH₂CH₂CH₂), 3.32 (m, 4H; CH₂CH₂CH₂NHCH₂CH₂CH₂); ¹³C NMR (100 MHz, CD₃OD) $\delta = 14.8$ (CH₂CH₃), 20.1 (CHCH₃), 24.1 (COCH₂CH₂), 27.1 (NHC(NH₂)NHCH₂CH₂), 28.1 (NHC(NH₂)NHCH₂CH₂CH₂), 33.7, 34.2, 34.5, 35.2 (CH(CH₃)CH₂(CH₂)₃CH₃), 37.2 (COCH₂), 38.2 (CH₂NHCO), 39.9 (NHC(NH₂)NHCH₂), 46.6 (NHC(NH₂)NHCH₂CH₂CH₂), 46.7 (CH₂CH₂CH₂NHCO), 159.2 (NHC(NH₂)NH), 178.2 (C=O amide); MS (ES⁺): m/z (%): 328.3 (46) (M+H)⁺; HRMS (ES⁺): m/z: calcd for C₁₇H₃₈N₅O [M+H]⁺: 328.3071; found: 328.3068.

***N*¹-(Carbamimidoyl)-*N*⁹-(4-ethyloctanoyl)-norspermidine (1.21).**

Yield : (resin: 0.64 mmol/g, 200 mg) 39 mg, 39 %.

$t_R = 7.5$ min (RP-HPLC, 96 % pure); IR (Film): $\nu = 1665, 1629$ cm^{-1} (C=O); ¹H NMR (400 MHz, CD₃OD) $\delta = 0.91$ (m, 6H; CH₂CH₂CH₃ + CHCH₂CH₃), 1.32 (m, 9H; CH(CH₂CH₃)CH₂(CH₂)₂CH₃), 1.59 (m, 2H; COCH₂CH₂), 1.90 (tt, ³J(HH) = 7, 7 Hz, 2H; CH₂CH₂CH₂NHCO), 2.01 (tt, ³J(HH) = 8, 8 Hz, 2H; NHC(NH₂)NHCH₂CH₂CH₂), 2.23 (t, ³J(HH) = 8 Hz, 2H; COCH₂), 3.03 (t, ³J(HH) = 7 Hz, 2H; CH₂CH₂CH₂NHCO), 3.08 (t, ³J(HH) = 8 Hz, 2H; NHC(NH₂)NHCH₂CH₂CH₂), 3.32 (m, 4H; CH₂CH₂CH₂NHCH₂CH₂CH₂); ¹³C NMR (100 MHz, CD₃OD) $\delta = 11.5$ (CH₂CH₂CH₃), 14.8 (CHCH₂CH₃), 24.5 (CH₂CH₂CH₃), 27.0 (NHC(NH₂)NHCH₂CH₂), 27.2 (CH₂CH₂CH₃), 28.1 (CH₂CH₂NHCO), 30.4, (CH₂CH₂CH₂CH₃), 30.8 (COCH₂CH₂), 34.0 (CHCH₂CH₃), 34.9 (COCH₂), 37.2 (NHC(NH₂)NHCH₂), 39.9 (CH₂NHCO), 40.4 (CHCH₂CH₃), 46.6 (CH₂CH₂CH₂NHCO), 46.9 (NHC(NH₂)NHCH₂CH₂CH₂), 159.2 (NHC(NH₂)NH), 178.2 (C=O amide); MS (ES⁺): m/z (%): 328.3 (40) (M+H)⁺; HRMS (ES⁺): m/z: calcd for C₁₇H₃₈N₅O [M+H]⁺: 328.3071; found: 328.3069.

***N*¹-(Carbamimidoyl)-*N*⁹-(undecanoyl)-norspermidine (1.22).**

Yield : (resin: 0.64 mmol/g, 200 mg) 59 mg, 58 %.

t_R = 8.2 min (RP-HPLC, 97 % pure); IR (Film): ν = 1665, 1629 cm^{-1} (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.91 (t, ³*J*(HH) = 7 Hz, 3H; CH₂CH₃), 1.32 (m, 14H; CH₂(CH₂)₆CH₃), 1.63 (m, 2H; COCH₂CH₂), 1.90 (tt, ³*J*(HH) = 7, 7 Hz, 2H; CH₂CH₂CH₂NHCO), 2.01 (m, 2H; NHC(NH₂)NHCH₂CH₂), 2.24 (t, ³*J*(HH) = 8 Hz, 2H; COCH₂), 3.02 (m, 2H; CH₂CH₂CH₂NHCO), 3.08 (m, 2H; NHC(NH₂)NHCH₂CH₂CH₂), 3.32 (m, 4H; CH₂CH₂CH₂NHCH₂CH₂CH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 14.8 (CH₂CH₃), 24.1 (CH₂CH₃), 27.2 (COCH₂CH₂), 27.4 (NHC(NH₂)NHCH₂CH₂), 28.1 (CH₂CH₂NHCO), 30.7, 30.8, 31.0, 31.1, (COCH₂CH₂(CH₂)₅CH₂), 33.4 (CH₂CH₂CH₃), 37.1 (CH₂NHCO), 37.4 (COCH₂), 39.9 (NHC(NH₂)NHCH₂), 46.6 (NHC(NH₂)NHCH₂CH₂CH₂), 46.8 (CH₂CH₂CH₂NHCO), 159.2 (NHC(NH₂)NH), 178.0 (C=O amide); MS (ES⁺): *m/z* (%): 342.3 (38) (M+H)⁺; HRMS (ES⁺): *m/z*: calcd for C₁₈H₄₀N₅O [M+H]⁺: 342.3228; found: 342.3229.

***N*¹-(Carbamimidoyl)-*N*⁹-(dodecanoyl)-norspermidine (1.23).**

Yield : (resin: 0.64 mmol/g, 50 mg) 15 mg, 57 %.

t_R = 8.6 min (RP-HPLC, 100 % pure); IR (Film): ν = 1659, 1633 cm^{-1} (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.91 (t, ³*J*(HH) = 7 Hz, 3H; CH₂CH₃), 1.32 (br s, 16H; CH₂(CH₂)₈CH₃), 1.63 (m, 2H; COCH₂CH₂), 1.90 (tt, ³*J*(HH) = 7, 7 Hz, 2H; CH₂CH₂CH₂NHCO), 2.01 (tt, ³*J*(HH) = 8, 8 Hz, 2H; NHC(NH₂)NHCH₂CH₂CH₂), 2.24 (t, ³*J*(HH) = 8 Hz, 2H; COCH₂CH₂), 3.03 (t, ³*J*(HH) = 7 Hz, 2H; CH₂CH₂CH₂NHCO), 3.08 (t, ³*J*(HH) = 8 Hz, 2H; NHC(NH₂)NHCH₂CH₂CH₂), 3.32 (m, 4H; CH₂CH₂CH₂NHCH₂CH₂CH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 14.8 (CH₂CH₃), 24.1 (CH₂CH₃), 27.2 (COCH₂CH₂), 27.4 (NHC(NH₂)NHCH₂CH₂), 28.1 (CH₂CH₂NHCO), 30.7, 30.8, 31.0, 31.1, (CH₂(CH₂)₆CH₂CH₂CH₃), 33.4 (CH₂CH₂CH₃), 37.1 (CH₂NHCO), 37.4 (COCH₂), 39.9 (NHC(NH₂)NHCH₂), 46.6 (NHC(NH₂)NHCH₂CH₂CH₂), 46.8 (CH₂CH₂CH₂NHCO), 159.2 (NHC(NH₂)NH), 178.0 (C=O amide); MS (ES⁺): *m/z* (%): 356.4 (45) (M+H)⁺; HRMS (ES⁺): *m/z*: calcd for C₁₉H₄₂N₅O [M+H]⁺: 356.3384; found: 356.3382.

***N*¹-(Carbamimidoyl)-*N*⁹-(tetradecanoyl)-norspermidine (1.24).**

Yield : (resin: 0.64 mmol/g, 200 mg) 51 mg, 48 %.

*t*_R = 9.4 min (RP-HPLC, 100 % pure); IR (Film): ν = 1657, 1627 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.80 (t, ³*J*(HH) = 7 Hz, 3H; CH₂CH₃), 1.20 (br s, 20H; CH₂(CH₂)₁₀CH₃), 1.51 (m, 2H; COCH₂CH₂), 1.78 (tt, ³*J*(HH) = 7, 7 Hz, 2H; CH₂CH₂CH₂NHCO), 1.89 (tt, ³*J*(HH) = 8, 8 Hz, 2H; NHC(NH₂)NHCH₂CH₂CH₂), 2.12 (t, ³*J*(HH) = 8 Hz, 2H; COCH₂), 2.91 (t, ³*J*(HH) = 7 Hz, 2H, CH₂CH₂CH₂NHCO), 2.97 (t, ³*J*(HH) = 8 Hz, 2H; NHC(NH₂)NHCH₂CH₂CH₂), 3.21 (m, 4H; CH₂CH₂CH₂NHCH₂CH₂CH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 14.8 (CH₂CH₃), 24.1 (CH₂CH₃), 27.2 (COCH₂CH₂), 27.4 (NHC(NH₂)NHCH₂CH₂), 28.1 (CH₂CH₂NHCO), 30.7, 30.8, 31.0, 31.1, 31.2, (CH₂(CH₂)₈CH₂CH₂CH₃), 33.4 (CH₂CH₂CH₃), 37.1 (CH₂NHCO), 37.4 (COCH₂), 39.9 (NHC(NH₂)NHCH₂), 46.6 (NHC(NH₂)NHCH₂CH₂CH₂), 46.8 (CH₂CH₂CH₂NHCO), 159.2 (NHC(NH₂)NH), 178.0 (C=O amide); MS (ES⁺): *m/z* (%): 384.4 (42) (M+H)⁺; HRMS (ES⁺): *m/z*: calcd for C₂₁H₄₆N₅O [M+H]⁺: 384.3697; found: 384.3692.

***N*¹-(Carbamimidoyl)-*N*⁹-(pentadecanoyl)-norspermidine (1.25).**

Yield : (resin: 0.64 mmol/g, 200 mg) 60 mg, 55 %.

*t*_R = 9.8 min (RP-HPLC, 100 % pure); IR (Film): ν = 1659, 1630 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.91 (m, 3H; CH₂CH₃), 1.30 (br s, 22H; CH₂(CH₂)₁₁CH₃), 1.63 (m, 2H; COCH₂CH₂), 1.90 (tt, ³*J*(HH) = 7, 7 Hz, 2H; CH₂CH₂CH₂NHCO), 2.01 (tt, ³*J*(HH) = 8, 8 Hz, 2H; NHC(NH₂)NHCH₂CH₂CH₂), 2.24 (t, ³*J*(HH) = 8 Hz, 2H; COCH₂CH₂), 3.03 (t, ³*J*(HH) = 7 Hz, 2H; CH₂CH₂CH₂NHCO), 3.08 (t, ³*J*(HH) = 8 Hz, 2H; NHC(NH₂)NHCH₂CH₂CH₂), 3.32 (m, 4H; CH₂CH₂CH₂NHCH₂CH₂CH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 14.8 (CH₂CH₃), 24.1 (CH₂CH₃), 27.2 (COCH₂CH₂), 27.4 (NHC(NH₂)NHCH₂CH₂), 28.1 (CH₂CH₂NHCO), 30.7, 30.8, 30.8, 31.0, 31.1, (CH₂(CH₂)₉CH₂CH₂CH₃), 33.1 (CH₂CH₂CH₃), 37.1 (CH₂NHCO), 37.4 (COCH₂), 39.9 (NHC(NH₂)NHCH₂), 46.6 (NHC(NH₂)NHCH₂CH₂CH₂), 46.8 (CH₂CH₂CH₂NHCO), 159.2 (NHC(NH₂)NH), 178.0 (C=O amide); MS (ES⁺): *m/z* (%): 398.2 (38) (M+H)⁺; HRMS (ES⁺): *m/z*: calcd for C₂₂H₄₈N₅O [M+H]⁺: 398.3854; found: 398.3845.

***N*¹-(Carbamimidoyl)-*N*⁹-(pamitoyl)-norspermidine (1.26).**

Yield : (resin: 0.64 mmol/g, 50 mg) 21 mg, 77 %.

*t*_R = 10.2 min (RP-HPLC, 100 % pure); IR (Film): ν = 1659, 1636 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.91 (m, 3H; CH₂CH₃), 1.30 (br s, 24H; CH₂(CH₂)₁₂CH₃), 1.63 (m, 2H; COCH₂CH₂), 1.90 (tt, ³*J*(HH) = 7, 7 Hz, 2H; CH₂CH₂CH₂NHCO), 2.01 (tt, ³*J*(HH) = 8, 8 Hz, 2H; NHC(NH₂)NHCH₂CH₂CH₂), 2.24 (t, ³*J*(HH) = 7 Hz, 2H; COCH₂CH₂), 3.03 (m, 2H; CH₂CH₂CH₂NHCO), 3.09 (m, 2H; NHC(NH₂)NHCH₂CH₂CH₂), 3.32 (m, 4H; CH₂CH₂CH₂NHCH₂CH₂CH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 12.5 (CH₂CH₃), 21.8 (CH₂CH₃), 24.9 (NHC(NH₂)NHCH₂CH₂), 25.0 (COCH₂CH₂), 25.8 (CH₂CH₂NHCO), 28.4, 28.5, 28.7, 28.8, 31.1 (CH₂(CH₂)₁₁CH₂CH₃), 34.8 (CH₂NHCO), 35.0 (COCH₂), 37.6 (NHC(NH₂)NHCH₂), 44.3 (NHC(NH₂)NHCH₂CH₂CH₂), 44.5 (CH₂CH₂CH₂NHCO), 156.9 (NHC(NH₂)NH), 175.7 (C=O amide); MS (ES⁺): *m/z* (%): 412.4 (75) (M+H)⁺; HRMS (ES⁺): *m/z*: calcd for C₂₃H₅₀N₅O [M+H]⁺: 412.4010; found: 412.4005.

***N*¹-(Carbamimidoyl)-*N*⁹-(oleoyl)-norspermidine (1.27).**

Yield : (resin: 0.64 mmol/g, 50 mg) 28 mg, 99 %.

*t*_R = 10.4 min (RP-HPLC, 100 % pure); IR (Film): ν = 1670, 1638 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.80 (m, 3H; CH₂CH₃), 1.21 (m, 20H; COCH₂CH₂(CH₂)₄CH₂ + CH₂(CH₂)₆CH₃), 1.51 (m, 2H; COCH₂CH₂), 1.78 (tt, ³*J*(HH) = 7, 7 Hz, 2H; CH₂CH₂CH₂NHCO), 1.90 (m, 6H; NHC(NH₂)NHCH₂CH₂ + CH₂CHCHCH₂), 2.12 (t, ³*J*(HH) = 8 Hz, 2H; COCH₂), 2.92 (m, 2H; CH₂CH₂CH₂NHCO), 2.97 (m, 2H; NHC(NH₂)NHCH₂CH₂CH₂), 3.21 (m, 4H; CH₂CH₂CH₂NHCH₂CH₂CH₂), 5.24 (m, 2H; CH₂CHCHCH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 14.2 (CH₂CH₃), 23.5 (CH₂CH₃), 26.6 (CH₂CHCHCH₂), 26.8 (COCH₂CH₂), 27.5 (CH₂CH₂NHCO), 27.9 (NHC(NH₂)NHCH₂CH₂), 30.0, 30.1, 30.2, 30.4, 30.6, (COCH₂CH₂(CH₂)₄CH₂ + CH₂(CH₂)₄CH₂CH₂CH₃), 32.8 (CH₂CH₂CH₃), 36.5 (CH₂NHCO), 36.8 (COCH₂), 39.3 (NHC(NH₂)NHCH₂), 46.0 (NHC(NH₂)NHCH₂CH₂CH₂), 46.2 (CH₂CH₂CH₂NHCO), 130.5, 130.7 (CH₂CHCHCH₂), 158.6 (NHC(NH₂)NH), 177.3 (C=O amide); MS (ES⁺): *m/z* (%): 438.4 (81) (M+H)⁺; HRMS (ES⁺): *m/z*: calcd for C₂₅H₅₂N₅O [M+H]⁺: 438.4167; found: 438.4168.

***N*¹-(Carbamimidoyl)-*N*⁹-(3a,7a,12a-trihydroxyl-5b-cholan-24-carbonyl)-norspermidine (1.28).**

Yield : (resin: 0.64 mmol/g, 100 mg) 44 mg, 93 %.

*t*_R = 7.4 min (RP-HPLC, 81 % pure); IR (Film): ν = 3347 (OH), 1671 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD, selected data) δ = 0.72 (s, 3H; 18'-CH₃), 0.92 (s, 3H; 19'-CH₃), 1.04 (d, ³*J*(HH) = 6 Hz, 3H; 21'-CH₃), 2.16 (m, 1H; 23'-CHH), 2.29 (m, 1H; 23'-CHH), 3.03 (m, 2H; CONHCH₂CH₂CH₂), 3.08 (m, 2H; CH₂CH₂CH₂NHC(NH₂)NH), 3.32 (m, 4H; CONHCH₂ + CH₂NHC(NH₂)NH), 3.40 (m, 1H; 3'-CH), 3.81 (m, 1H; 7'-CH), 3.97 (m, 1H; 12'-CH); ¹³C NMR (100 MHz, CD₃OD, selected data) δ = 13.3 (18'-CH₃), 18.1 (21'-CH₃), 23.5 (19'-CH₃), 27.2 (CONHCH₂CH₂), 28.3 (CH₂CH₂NHC(NH₂)NH), 37.4 (CH₂NHC(NH₂)NH), 39.9 (CONHCH₂), 46.6 (CONHCH₂CH₂CH₂), 46.8 (CH₂CH₂CH₂NHC(NH₂)NH), 69.5 (7'-CH), 73.3 (3'-CH), 74.4 (12'-CH), 159.2 (NHC(NH₂)NH), 178.6 (24'-CO). MS (ES⁺): *m/z* (%): 564.2 (50) (M+H)⁺; HRMS (ES⁺): *m/z*: calcd for C₃₁H₅₈N₅O₄ [M+H]⁺: 564.4484; found: 564.4477.

***N*¹-(Carbamimidoyl)-*N*⁹-(3a,12a-dihydroxyl-5b-cholan-24-carbonyl)-norspermidine (1.29).**

Yield : (resin: 0.64 mmol/g, 100 mg) 47 mg, 94 %.

*t*_R = 8.1 min (RP-HPLC, 100 % pure); IR (Film): ν = 3332 (OH), 1672 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD, selected data) δ = 0.73 (s, 3H; 18'-CH₃), 0.99 (s, 3H; 19'-CH₃), 1.04 (d, ³*J*(HH) = 6 Hz, 3H; 21'-CH₃), 1.94 (m, 2H; CONHCH₂CH₂), 2.01 (m, 2H; CH₂CH₂NHC(NH₂)NH), 2.17 (m, 1H; 23'-CHH), 2.31 (m, 1H; 23'-CHH), 3.02 (m, 2H; CONHCH₂CH₂CH₂), 3.08 m, 2H; CH₂CH₂CH₂NHC(NH₂)NH), 3.32 (m, 4H; CONHCH₂ + CH₂NHC(NH₂)NH), 3.55 (m, 1H; 3'-CH), 3.98 (m, 1H; 12'-CH); ¹³C NMR (100 MHz, CD₃OD, selected data) δ = 14.3 (18'-CH₃), 18.8 (21'-CH₃), 24.5 (19'-CH₃), 28.0 (CH₂CH₂NHC(NH₂)NH), 28.9 (CONHCH₂CH₂), 38.1 (CONHCH₂), 40.7 (CH₂NHC(NH₂)NH), 47.4 (CH₂CH₂CH₂NHC(NH₂)NH), 47.6 (CONHCH₂CH₂CH₂), 73.7 (3'-CH), 75.2 (12'-CH), 160.0 (NHC(NH₂)NH), 179.3 (24'-CO); MS (ES⁺): *m/z* (%): 548.3 (25) (M+H)⁺; HRMS (ES⁺): *m/z*: calcd for C₃₁H₅₈N₅O₃ [M+H]⁺: 548.4534; found: 548.4533.

***N*¹-(Carbamimidoyl)-*N*⁹-(3,7,12-trioxo-5b-cholan-24-carbonyl)-norspermidine (1.30).**

Yield : (resin: 0.64 mmol/g, 100 mg) 30 mg, 59 %.

*t*_R = 6.9 min (RP-HPLC, 100 % pure); IR (Film): ν = 1671 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD, selected data) δ = 0.87 (d, ³*J*(HH) = 7 Hz, 3H; 21'-CH₃); 1.10 (s, 3H; 18'-CH₃), 1.36 (s, 3H; 19'-CH₃), 1.96-2.08 (m, 4H; CH₂CH₂NHCH₂CH₂), 2.99-3.12 (m, 4H; CH₂NHCH₂), 3.28-3.37 (m, 4H, CONHCH₂ + CH₂NHC(NH₂)NH); ¹³C NMR (100 MHz, CD₃OD) δ = 14.4, 18.6, 22.1, 25.4, 26.1, 26.6, 27.1, 27.2, 28.1, 32.1, 32.3, 33.5, 35.0, 36.1, 36.4, 36.5, 38.8, 39.0, 44.2, 45.3, 45.6, 45.8, 46.2, 46.6, 49.3, 53.2, 57.6, 158.2 (C-Guanidine); 177.3 (C=O), 212.0, 215.1 (C=O). MS (ES⁺): *m/z* (%): 558.2 (18) (M+H)⁺; HRMS (ES⁺): *m/z*: calcd for C₃₁H₅₂N₅O₄ [M+H]⁺: 558.4014; found: 558.4066.

***N*¹-(Carbamimidoyl)-*N*⁹-(3a,7a,12a-trihydroxyl-5b-cholan-24-carbonyl)-norspermidine (1.31).**

Yield : (resin: 0.64 mmol/g, 100 mg) 39 mg, 79 %.

*t*_R = 8.0 min (RP-HPLC, 79 % pure); IR (Film): ν = 3333 (OH), 1671 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD, selected data) δ = 0.69 (s, 3H; 18'-CH₃), 0.96 (s, 3H; 19'-CH₃), 0.97 (d, ³*J*(HH) = 7 Hz, 3H; 21'-CH₃), 1.87 (m, 2H; CONHCH₂CH₂), 1.98 (m, 2H; CH₂CH₂NHC(NH₂)NH), 2.13 (m, 1H; 23'-CHH), 2.27 (m, 1H; 23'-CHH), 2.96-3.09 (m, 4H; CH₂NHCH₂), 3.24-3.35 (m, 4H; CONHCH₂ + CH₂NHC(NH₂)NH), 3.39 (m, 1H; 3'-CH), 3.80 (m, 1H; 7'-CH); ¹³C NMR (100 MHz, CD₃OD, selected data) δ = 12.6 (18'-CH₃), 19.3 (21'-CH₃), 22.2 (19'-CH₃), 27.2 (CH₂CH₂NHC(NH₂)NH), 28.1 (CONHCH₂CH₂), 36.6 (CONHCH₂), 39.9 (CH₂NHC(NH₂)NH), 46.6 (CH₂CH₂CH₂NHC(NH₂)NH), 46.8 (CONHCH₂CH₂CH₂), 73.2 (7'-CH), 81.4 (3'-CH), 159.2 (NHC(NH₂)NH), 178.5 (24'-CO); MS (ES⁺): *m/z* (%): 548.1 (27) (M+H)⁺; HRMS (ES⁺): *m/z*: calcd for C₃₁H₅₈N₅O₃ [M+H]⁺: 548.4534; found: 548.4536.

Compound Library 2.

***N*¹-(3-(*N',N'*-bis-(3'(Guanidinopropyl)carbamoyl)propionyl)-*N*⁹-(pentanoyl)-norspermidine (2.1).**

Yield : (resin: 0.64 mmol/g, 45.5 mg) 20 mg, 53 %.

$t_R = 2.2$ min (RP-HPLC, 95 % pure); IR (Film): $\nu = 1655, 1621$ cm^{-1} (C=O); ¹H NMR (400 MHz, CD₃OD) $\delta = 0.95$ (t, ³*J*(HH) = 8 Hz, 3H; CH₂CH₃), 1.37 (m, 2H; CH₂CH₃), 1.41 (m, 2H; COCH₂CH₂), 1.89 (m, 8H; CH₂CH₂NHCOC₄H₉ + CONHCH₂CH₂ + CH₂CH₂NCH₂CH₂), 2.24 (t, ³*J*(HH) = 8 Hz, 2H; COCH₂CH₂), 2.57 (t, ³*J*(HH) = 6 Hz, 2H; COCH₂CH₂CONH), 2.71 (t, ³*J*(HH) = 6 Hz, 2H; COCH₂CH₂CONH), 3.01 (m, 4H; CH₂NHCH₂), 3.19 (t, ³*J*(HH) = 7 Hz, 2H; NHC(NH₂)NHCH₂), 3.30 (m, 6H; CH₂NHCOC₄H₉ + CONHCH₂CH₂CH₂NH + NHC(NH₂)NHCH₂), 3.43 (m, 4H; CH₂NCH₂); ¹³C NMR (100 MHz, CD₃OD) $\delta = 16.2$ (CH₂CH₃), 25.6 (CH₂CH₃), 29.8 (CONHCH₂CH₂), 29.9 (CH₂CH₂NHCOC₄H₉), 30.3 (NHC(NH₂)NHCH₂), 31.0 (NCOCH₂), 31.1 (NHC(NH₂)NHCH₂), 31.3 (NHCOCH₂CH₂), 33.5 (CH₂CONH), 38.9 (COCH₂C₃H₇), 39.1 (CH₂NHCOC₄H₉ + CONHCH₂), 42.1, 42.3 (NHC(NH₂)NHCH₂), 46.4, 48.4 (CH₂NCH₂), 48.6 (CONHCH₂CH₂CH₂NH), 48.7 (CH₂CH₂CH₂NHCOC₄H₉), 160.9 (NHC(NH₂)NH), 176.6 (NCOCH₂CH₂CONH), 178.1 (NCOCH₂CH₂CONH), 179.5 (NHCOC₄H₉); MS (ES⁺): *m/z* (%): 257.4 (100) (M+2H)²⁺, 513.6 (8) (M+H)⁺; HRMS (ES⁺): *m/z*: calcd for C₂₃H₄₉N₁₀O₃ [M+H]⁺: 513.3984; found: 513.3981.

***N*¹-(3-(*N',N'*-bis-(3'(Guanidinepropyl)carbamoyl)propionyl)-*N*⁹-(3-methylbutanoyl)-norspermidine (2.2).**

Yield : (resin: 0.64 mmol/g, 39.0 mg) 12 mg, 36 %.

$t_R = 2.2$ min (RP-HPLC, 95 % pure); IR (Film): $\nu = 1654, 1635$ cm^{-1} (C=O); ¹H NMR (400 MHz, CD₃OD) $\delta = 0.97$ (d, ³*J*(HH) = 7 Hz, 6H; CH(CH₃)₂), 1.82 (m, 2H; NHC(NH₂)NHCH₂CH₂), 1.92 (m, 6H; CH₂CH₂NHCOC₄H₉ + CONHCH₂CH₂ + NHC(NH₂)NHCH₂CH₂), 2.10 (m, 3H; COCH₂CH(CH₃)₂), 2.57 (t, ³*J*(HH) = 6 Hz, 2H; NCOCH₂CH₂CONH), 2.72 (t, ³*J*(HH) = 6 Hz, 2H; NCOCH₂CH₂CONH), 3.00 (m, 2H; CH₂CH₂CH₂NHCOC₄H₉), 3.04 (m, 2H; CONHCH₂CH₂CH₂), 3.19 (t, ³*J*(HH) = 7 Hz, 2H; NHC(NH₂)NHCH₂), 3.31 (m, 6H; CH₂NHCOC₄H₉ + CONHCH₂ + NHC(NH₂)NHCH₂), 3.44 (m, 4H; CH₂NCH₂); ¹³C NMR (100 MHz, CD₃OD) $\delta = 23.1$ (CH(CH₃)₂), 27.7 (CH(CH₃)₂),

28.0 (CONHCH₂CH₂), 28.1 (CH₂CH₂NHCOC₄H₉), 28.6 (NHC(NH₂)NHCH₂CH₂), 29.2 (NCOCH₂CH₂CONH), 29.3 (NHC(NH₂)NHCH₂CH₂), 31.7 (NCOCH₂CH₂CONH), 37.3 (CONHCH₂ + CH₂NHCOC₄H₉), 40.3, 40.5 (NHC(NH₂)NHCH₂), 44.6 (NHC(NH₂)NHCH₂CH₂CH₂), 46.6 (NHC(NH₂)NHCH₂CH₂CH₂ + COCH₂CH(CH₃)₂), 46.8 (CONHCH₂CH₂CH₂), 46.9 (CH₂CH₂CH₂NHCOC₄H₉), 159.3 (NHC(NH₂)NH), 174.8 (NCOCH₂CH₂CONH), 176.4 (NCOCH₂CH₂CONH), 176.9 (NHCOC₄H₉); MS (ES⁺): m/z (%): 257.4 (100) (M+2H)²⁺, 513.6 (8) (M+H)⁺; HRMS (ES⁺): m/z: calcd for C₂₃H₄₉N₁₀O₃ [M+H]⁺: 513.3984; found: 513.3982.

***N*¹-(3-(*N',N'*-bis-(3'(Guanidinopropyl)carbamoyl)propionyl)-*N*⁹-(2,2-dimethylpropionyl)-norspermidine (2.3).**

Yield : (resin: 0.64 mmol/g, 45.1 mg) 18 mg, 47 %.

*t*_R = 2.2 min (RP-HPLC, 98 % pure); IR (Film): ν = 1661, 1618 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 1.21 (s, 9H; C(CH₃)₃), 1.82 (m, 2H; NHC(NH₂)NHCH₂CH₂), 1.91 (m, 6H; NHC(NH₂)NHCH₂CH₂ + CONHCH₂CH₂ + CH₂CH₂NHCOC₄H₉), 2.57 (t, ³J(HH) = 6 Hz, 2H; NCOCH₂CH₂CONH), 2.71 (t, ³J(HH) = 6 Hz, 2H; NCOCH₂CH₂CONH), 2.96 (m, 2H; CH₂CH₂CH₂NHCOC₄H₉), 3.03 (m, 2H; CONHCH₂CH₂CH₂), 3.19 (t, ³J(HH) = 7 Hz, 2H; NHC(NH₂)NHCH₂), 3.31 (m, 6H; NHC(NH₂)NHCH₂ + CONHCH₂ + CH₂NHCOC₄H₉), 3.44 (m, 4H; CH₂NCH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 28.0 (CONHCH₂CH₂CH₂), 28.1 (CH₂CH₂NHCOC₄H₉), 28.2 (C(CH₃)₃), 28.5 (NHC(NH₂)NHCH₂), 29.2 (NCOCH₂CH₂CONH), 29.3 (NHC(NH₂)NHCH₂), 31.7 (NCOCH₂CH₂CONH), 37.4 (CH₂NHCOC₄H₉ + CONHCH₂), 40.1 (C(CH₃)₃), 40.3, 40.5 (NHC(NH₂)NHCH₂), 44.6, 46.6 (CH₂NCH₂), 46.8 (CH₂NHCH₂), 159.1, 159.2 (NHC(NH₂)NH), 174.8 (NCOCH₂CH₂CONH), 176.3 (NCOCH₂CH₂CONH), 183.0 (COC(CH₃)₃); MS (ES⁺): m/z (%): 257.4 (100) (M+2H)²⁺, 513.6 (8) (M+H)⁺; HRMS (ES⁺): m/z: calcd for C₂₃H₅₀N₁₀O₃ [M+2H]²⁺: 257.2028; found: 257.2028.

***N*¹-(3-(*N',N'*-bis-(3'(Guanidinopropyl)carbamoyl)propionyl)-*N*⁹-(hexanoyl)-norspermidine (2.4).**

Yield : (resin: 0.64 mmol/g, 41.3 mg) 16 mg, 46 %.

$t_R = 2.2$ min (RP-HPLC, 95 % pure); IR (Film): $\nu = 1655, 1621$ cm^{-1} (C=O); ¹H NMR (400 MHz, CD₃OD) $\delta = 0.93$ (t, ³*J*(HH) = 7 Hz, 3H; CH₂CH₃), 1.34 (m, 4H; CH₂CH₂CH₃), 1.63 (m, 2H; COCH₂CH₂C₃H₇), 1.82 (m, 2H; NHC(NH₂)NHCH₂CH₂), 1.91 (m, 6H; NHC(NH₂)NHCH₂CH₂ + CONHCH₂CH₂ + CH₂CH₂NHCOC₅H₁₁), 2.23 (t, ³*J*(HH) = 8 Hz, 2H; COCH₂CH₂C₃H₇), 2.57 (t, ³*J*(HH) = 6 Hz, 2H; NCOCH₂CH₂CONH), 2.71 (t, ³*J*(HH) = 6 Hz, 2H; NCOCH₂CH₂CONH), 3.01 (m, 4H; CH₂NHCH₂), 3.19 (t, ³*J*(HH) = 7 Hz, 2H; NHC(NH₂)NHCH₂), 3.29 (m, 6H; NHC(NH₂)NHCH₂ + CONHCH₂ + CH₂NHCOC₅H₁₁), 3.44 (m, 4H; CH₂NCH₂); ¹³C NMR (100 MHz, CD₃OD) $\delta = 14.6$ (CH₂CH₃), 23.8 (CH₂CH₃), 27.0 (COCH₂CH₂C₃H₇), 28.0 (CONHCH₂CH₂), 28.1 (CH₂CH₂NHCOC₅H₁₁), 28.6 (NHC(NH₂)NHCH₂CH₂), 29.2 (NCOCH₂CH₂CONH), 29.3 (NHC(NH₂)NHCH₂CH₂), 31.7 (NCOCH₂CH₂CONH), 32.9 (CH₂CH₂CH₃), 37.3 (CONHCH₂ + CH₂NHCOC₅H₁₁ + COCH₂C₄H₉), 40.3, 40.5 (NHC(NH₂)NHCH₂), 44.6, 46.6 (CH₂NCH₂), 46.8 (CONHCH₂CH₂CH₂), 46.9 (CH₂CH₂CH₂NHCOC₅H₁₁), 159.1, 159.2 (NHC(NH₂)NH), 174.8 (NCOCH₂CH₂CONH), 176.3 (NCOCH₂CH₂CONH), 177.7 (NHCOC₅H₁₁); MS (ES⁺): *m/z* (%): 264.3 (58) (M+2H)²⁺.

***N*¹-(3-(*N',N'*-bis-(3'(Guanidinopropyl)carbamoyl)propionyl)-*N*⁹-(2-methylpentanoyl)-norspermidine (2.5).**

Yield : (resin: 0.64 mmol/g, 44.6 mg) 13 mg, 34 %.

$t_R = 2.2$ min (RP-HPLC, 88 % pure); IR (Film): $\nu = 1661, 1621$ cm^{-1} (C=O); ¹H NMR (400 MHz, CD₃OD) $\delta = 0.93$ (t, ³*J*(HH) = 7 Hz, 3H; CH₂CH₃), 1.13 (d, ³*J*(HH) = 7 Hz, 3H; CHCH₃), 1.33 (m, 3H; CH(H)CH₂CH₃), 1.60 (m, 1H; CH(H)CH₂CH₃), 1.82 (m, 2H; NHC(NH₂)NHCH₂CH₂), 1.92 (m, 6H; NHC(NH₂)NHCH₂CH₂ + CONHCH₂CH₂ + CH₂CH₂NHCOC₅H₁₁), 2.35 (m, 1H; COCH), 2.57 (t, ³*J*(HH) = 6 Hz, 2H; NCOCH₂CH₂CONH), 2.71 (t, ³*J*(HH) = 6 Hz, 2H; NCOCH₂CH₂CONH), 2.98 (m, 2H; CH₂CH₂CH₂NHCOC₅H₁₁), 3.04 (m, 2H; CONHCH₂CH₂CH₂), 3.19 (t, ³*J*(HH) = 7 Hz, 2H; NHC(NH₂)NHCH₂), 3.31 (m, 6H; NHC(NH₂)NHCH₂ + CONHCH₂ + CH₂NHCOC₅H₁₁), 3.44 (m, 4H; CH₂NCH₂); ¹³C NMR (100 MHz, CD₃OD) $\delta = 14.7$ (CH₂CH₃), 18.8 (CHCH₃),

22.1 (CH₂CH₃), 28.0 (CONHCH₂CH₂), 28.1 (CH₂CH₂NHCOC₅H₁₁), 28.5 (NHC(NH₂)NHCH₂CH₂), 29.2 (NCOCH₂CH₂CONH), 29.3 (NHC(NH₂)NHCH₂CH₂), 31.7 (NCOCH₂CH₂CONH), 37.2 (CONHCH₂ + CH₂NHCOC₅H₁₁), 37.9 (CH₂CH₂CH₃), 40.3, 40.5 (NHC(NH₂)NHCH₂), 42.2 (NHCOCH), 44.6, 46.6 (CH₂NCH₂), 46.8 (CONHCH₂CH₂CH₂), 46.9 (CH₂CH₂CH₂NHCOC₅H₁₁), 159.2 (NHC(NH₂)NH), 174.8 (NCOCH₂CH₂CONH), 176.3 (NCOCH₂CH₂CONH), 181.1 (NHCOC₅H₁₁); MS (ES⁺): m/z (%): 264.3 (100) (M+2H)²⁺, 527.4 (8) (M+H)⁺; HRMS (ES⁺): m/z: calcd for C₂₄H₅₁N₁₀O₃ [M+H]⁺: 527.4140; found: 527.4146.

***N*¹-(3-(*N',N'*-bis-(3'(Guanidinopropyl)carbamoyl)propionyl)-*N*⁹-(3-methylpentanoyl)-norspermidine (2.6).**

Yield : (resin: 0.64 mmol/g, 43.7 mg) 19 mg, 51 %.

*t*_R = 2.2 min (RP-HPLC, 80 % pure); IR (Film): ν = 1664, 1621 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.93 (m, 6H, *m*; CH(CH₃)CH₂CH₃), 1.25, 1.39 (m, 2H; CH₂CH₃), 1.39, 1.89 (m, 9H; 2xNHC(NH₂)NHCH₂CH₂ + CONHCH₂CH₂ + CH₂CH₂NHCOC₅H₁₁ + CH(CH₃)CH₂CH₃), 2.01 (dd, ³*J*(HH) = 6, 14 Hz, 1H; COCH(H)CH(CH₃)C₂H₅), 2.22 (dd, ³*J*(HH) = 6, 14 Hz, 1H; COCH(H)CH(CH₃)C₂H₅), 2.57 (t, ³*J*(HH) = 6 Hz, 2H; NCOCH₂CH₂CONH), 2.71 (t, ³*J*(HH) = 6 Hz, 2H; NCOCH₂CH₂CONH), 3.02 (m, 4H; CH₂NHCH₂), 3.19 (t, ³*J*(HH) = 7 Hz, 2H; NHC(NH₂)NHCH₂CH₂), 3.30 (m, 6H; NHC(NH₂)NHCH₂ + CONHCH₂ + CH₂NHCOC₅H₁₁), 3.44 (m, 4H; CH₂NCH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 11.5 (CH₂CH₃), 19.4 (CHCH₃), 27.5 (CONHCH₂CH₂), 27.6 (CH₂CH₂NHCOC₅H₁₁), 28.1 (NHC(NH₂)NHCH₂CH₂), 28.7 (NCOCH₂CH₂CONH), 28.8 (NHC(NH₂)NHCH₂CH₂), 30.4 (CH₂CH₃), 31.2 (NCOCH₂CH₂CONH), 33.5 (CHCH₃), 36.9 (CONHCH₂ + CH₂NHCOC₅H₁₁), 39.9, 40.0 (NHC(NH₂)NHCH₂), 42.1 (COCH₂C₄H₉), 44.2, 46.2 (CH₂NCH₂), 46.3 (CONHCH₂CH₂CH₂), 46.5 (CH₂CH₂CH₂NHCOC₅H₁₁), 158.7 (NHC(NH₂)NH), 174.4 (NCOCH₂CH₂CONH), 176.7 (NCOCH₂CH₂CONH + NHCOC₅H₁₁); MS (ES⁺): m/z (%): 264.3 (100) (M+2H)²⁺, 527.3 (5) (M+H)⁺; HRMS (ES⁺): m/z: calcd for C₂₄H₅₁N₁₀O₃ [M+H]⁺: 527.4140; found: 527.4143.

***N*¹-(3-(*N,N'*-bis-(3'(Guanidinopropyl)carbamoyl)propionyl)-*N*⁹-(4-methylpentanoyl)-norspermidine (2.7).**

Yield : (resin: 0.64 mmol/g, 44.5 mg) 19 mg, 50 %.

*t*_R = 2.2 min (RP-HPLC, 81 % pure); IR (Film): ν = 1656, 1616 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.93 (d, ³*J*(HH) = 7 Hz, 6H; CH(CH₃)₂), 1.52 (m, 2H; CH₂CH(CH₃)₂), 1.58 (m, 1H; CH(CH₃)₂), 1.81 (m, 2H; NHC(NH₂)NHCH₂CH₂), 1.92 (m, 6H; NHC(NH₂)NHCH₂CH₂ + CONHCH₂CH₂ + CH₂CH₂NHCOC₅H₁₁), 2.24 (t, ³*J*(HH) = 8 Hz, 2H; NHCOCH₂C₄H₉), 2.56 (t, ³*J*(HH) = 6 Hz, 2H; NCOCH₂CH₂CONH), 2.71 (t, ³*J*(HH) = 6 Hz, 2H; NCOCH₂CH₂CONH), 2.98 (m, 2H; CH₂CH₂CH₂NHCOC₅H₁₁), 3.03 (m, 2H; CONHCH₂CH₂CH₂), 3.18 (t, ³*J*(HH) = 7 Hz, 2H; NHC(NH₂)NHCH₂), 3.30 (m, 6H; NHC(NH₂)NHCH₂ + CONHCH₂ + CH₂NHCOC₅H₁₁), 3.43 (m, 4H; CH₂NCH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 23.0 (CH(CH₃)₂), 28.1 (CONHCH₂CH₂ + CH₂CH₂NHCOC₅H₁₁), 28.5 (NHC(NH₂)NHCH₂CH₂), 29.2 (NCOCH₂CH₂CONH), 29.3 (NHC(NH₂)NHCH₂CH₂), 29.4 (CH(CH₃)₂), 31.7 (NCOCH₂CH₂CONH), 35.5 (NHCOCH₂C₄H₉), 37.3 (CONHCH₂ + CH₂NHCOC₅H₁₁), 40.3, 40.5 (NHC(NH₂)NHCH₂), 44.6, 46.6 (CH₂NCH₂), 46.8 (CONHCH₂CH₂CH₂), 46.9 (CH₂CH₂CH₂NHCOC₅H₁₁), 159.2 (NHC(NH₂)NH), 174.8 (NCOCH₂CH₂CONH), 175.9 (NCOCH₂CH₂CONH), 176.4 (NHCOC₅H₁₁); MS (ES⁺): *m/z* (%): 264.3 (100) (M+2H)²⁺, 527.4 (3) (M+H)⁺; HRMS (ES⁺): *m/z*: calcd for C₂₄H₅₁N₁₀O₃ [M+H]⁺: 527.4140; found: 527.4145.

***N*¹-(3-(*N,N'*-bis-(3'(Guanidinopropyl)carbamoyl)propionyl)-*N*⁹-(2-ethylbutanoyl)-norspermidine (2.8).**

Yield : (resin: 0.64 mmol/g, 38.0 mg) 16 mg, 50 %.

*t*_R = 2.2 min (RP-HPLC, 83 % pure); IR (Film): ν = 1656, 1621 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.66 (t, ³*J*(HH) = 8 Hz, 6H; CH(CH₂CH₃)₂), 1.25, 1.34 (m, 4H; CH(CH₂CH₃)₂), 1.58 (m, 2H; NHC(NH₂)NHCH₂CH₂), 1.67 (m, 6H; NHC(NH₂)NHCH₂CH₂ + CONHCH₂CH₂ + CH₂CH₂NHCOC₅H₁₁), 1.81 (m, 1H; COCH(C₄H₁₀), 2.33 (t, ³*J*(HH) = 6 Hz, 2H; NCOCH₂CH₂CONH), 2.47 (t, ³*J*(HH) = 6 Hz, 2H; NCOCH₂CH₂CONH), 2.71 (m, 4H; CH₂NHCH₂), 2.95 (t, ³*J*(HH) = 7 Hz, 2H; NHC(NH₂)NHCH₂CH₂), 3.06 (m, 6H; *m*, NHC(NH₂)NHCH₂ + CONHCH₂ + CH₂NHCOC₅H₁₁), 3.20 (m, 4H; CH₂NCH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 12.8 (CH(CH₂CH₃)₂), 27.1 (CH(CH₂CH₃)₂), 28.0 (CONHCH₂CH₂),

28.2 (CH₂CH₂NHCOC₅H₁₁), 28.6 (NHC(NH₂)NHCH₂CH₂), 29.3 (NHC(NH₂)NHCH₂CH₂ + NCOCH₂CH₂CONH), 31.7 (NCOCH₂CH₂CONH), 37.3 (CONHCH₂ + CH₂NHCOC₅H₁₁), 40.3, 40.5 (NHC(NH₂)NHCH₂), 44.6, 46.6 (CH₂NCH₂), 46.8 (CONHCH₂CH₂CH₂), 47.0 (CH₂CH₂CH₂NHCOC₅H₁₁), 52.2 (NHCOCH), 159.3 (NHC(NH₂)NH), 174.8 (NCOCH₂CH₂CONH), 176.3 (NCOCH₂CH₂CONH), 180.2 (NHCOC₅H₁₁); MS (ES⁺): m/z (%): 264.2 (56) (M+2H)²⁺, 527.4 (8) (M+H)⁺; HRMS (ES⁺): m/z: calcd for C₂₄H₅₁N₁₀O₃ [M+H]⁺: 527.4140; found: 527.4144.

***N*¹-(3-(*N,N'*-bis-(3'(Guanidinopropyl)carbamoyl)propionyl)-*N*⁹-(3,3-dimethylbutanoyl)-norspermidine (2.9).**

Yield : (resin: 0.64 mmol/g, 38.7 mg) 14 mg, 42 %.

*t*_R = 2.2 min (RP-HPLC, 81 % pure); IR (Film): ν = 1656, 1621 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 1.06 (s, 9H; C(CH₃)₃), 1.82 (m, 2H; NHC(NH₂)NHCH₂CH₂), 1.91 (m, 6H; *m*, NHC(NH₂)NHCH₂CH₂ + CONHCH₂CH₂ + CH₂CH₂NHCOC₅H₁₁), 2.12 (s, 2H; COCH₂C(CH₃)₃), 2.57 (t, ³*J*(HH) = 6 Hz, 2H; NCOCH₂CH₂CONH), 2.71 (³*J*(HH) = 6 Hz, 2H; NCOCH₂CH₂CONH), 3.02 (m, 4H; CH₂NHCH₂), 3.19 (³*J*(HH) = 7 Hz, 2H; NHC(NH₂)NHCH₂CH₂), 3.30 (m, 6H; NHC(NH₂)NHCH₂CH₂ + CONHCH₂ + CH₂NHCOC₅H₁₁), 3.44 (m, 4H; CH₂NCH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 28.0 (CONHCH₂CH₂), 28.1 (CH₂CH₂NHCOC₅H₁₁), 28.5 (NHC(NH₂)NHCH₂CH₂), 29.2 (NCOCH₂CH₂CONH), 29.3 (NHC(NH₂)NHCH₂CH₂), 30.6 (C(CH₃)₃), 31.7 (NCOCH₂CH₂CONH), 37.3 (CONHCH₂ + CH₂NHCOC₅H₁₁), 40.3, 40.5 (NHC(NH₂)NHCH₂), 44.6, 46.6 (CH₂NCH₂), 46.8 (CONHCH₂CH₂CH₂), 47.0 (CH₂CH₂CH₂NHCOC₅H₁₁), 159.2 (NHC(NH₂)NH), 174.8 (NCOCH₂CH₂CONH), 176.1 (NCOCH₂CH₂CONH), 176.3 (NHCOC₅H₁₁); MS (ES⁺): m/z (%): 264.2 (38) (M+2H)²⁺, 527.4 (6) (M+H)⁺; HRMS (ES⁺): m/z: calcd for C₂₄H₅₁N₁₀O₃ [M+H]⁺: 527.4140; found: 527.4143.

***N*¹-(3-(*N',N'*-bis-(3'(Guanidinopropyl)carbamoyl)propionyl)-*N*⁹-(heptanoyl)-norspermidine (2.10).**

Yield : (resin: 0.64 mmol/g, 40.2 mg) 19 mg, 56 %.

*t*_R = 5.9 min (RP-HPLC, 63 % pure); IR (Film): ν = 1655, 1619 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.92 (t, ³*J*(HH) = 7 Hz, 3H; CH₂CH₃), 1.33 (br s, 6H; CH₂(CH₂)₃CH₃), 1.62 (m, 2H; NHCOCH₂CH₂C₄H₉), 1.82 (m, 2H; NHC(NH₂)NHCH₂CH₂), 1.91 (m, 6H, NHC(NH₂)NHCH₂CH₂ + CONHCH₂CH₂ + CH₂CH₂NHCOC₆H₁₃), 2.23 (t, ³*J*(HH) = 8 Hz, 2H; NHCOCH₂CH₂C₄H₉), 2.57 (t, ³*J*(HH) = 6 Hz, 2H; NCOCH₂CH₂CONH), 2.71 (t, ³*J*(HH) = 6 Hz, 2H; NCOCH₂CH₂CONH), 3.01 (m, 4H; CH₂NHCH₂), 3.19 (t, ³*J*(HH) = 7 Hz, 2H; NHC(NH₂)NHCH₂), 3.30 (m, 6H; NHC(NH₂)NHCH₂ + CONHCH₂ + CH₂NHCOC₆H₁₃), 3.44 (m, 4H; CH₂NCH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 14.7 (CH₂CH₃), 23.9 (CH₂CH₃), 27.3 (COCH₂CH₂C₄H₉), 28.1 (CONHCH₂CH₂ + CH₂CH₂NHCOC₆H₁₃), 28.6 (NHC(NH₂)NHCH₂CH₂), 29.3 (NHC(NH₂)NHCH₂CH₂ + NCOCH₂CH₂CONH), 30.4 (CH₂CH₂C₃H₇), 31.7 (NCOCH₂CH₂CONH), 33.0 (CH₂CH₂CH₂CH₃), 37.3 (CONHCH₂ + CH₂NHCOC₆H₁₃), 37.4 (NHCOCH₂C₅H₁₁), 40.3, 40.5 (NHC(NH₂)NHCH₂), 44.6, 46.6 (CH₂NCH₂), 46.8 (CONHCH₂CH₂CH₂), 46.9 (CH₂CH₂CH₂NHCOC₆H₁₃), 159.2 (NHC(NH₂)NH), 174.8 (NCOCH₂CH₂CONH), 176.3 (NCOCH₂CH₂CONH), 177.8 (NHCOC₆H₁₃); MS (ES⁺): *m/z* (%): 271.2 (50) (M+2H)²⁺, 541.5 (8) (M+H)⁺; HRMS (ES⁺): *m/z*: calcd for C₂₅H₅₃N₁₀O₃ [M+H]⁺: 541.4297; found: 541.4305.

***N*¹-(3-(*N',N'*-bis-(3'(Guanidinopropyl)carbamoyl)propionyl)-*N*⁹-(2-methylhexanoyl)-norspermidine (2.11).**

Yield : (resin: 0.64 mmol/g, 42.1 mg) 14 mg, 39 %.

*t*_R = 5.6 min (RP-HPLC, 63 % pure); IR (Film): ν = 1654, 1621 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.92 (t, ³*J*(HH) = 7 Hz, 3H; CH₂CH₃), 1.13 (d, ³*J*(HH) = 7 Hz, 3H; CHCH₃), 1.34 (m, 5H; CH(H)CH₂CH₂CH₃), 1.61 (m, 1H; CH(H)CH₂CH₂CH₃), 1.82 (m, 2H; NHC(NH₂)NHCH₂CH₂), 1.91 (m, 6H; NHC(NH₂)NHCH₂CH₂ + CONHCH₂CH₂ + CH₂CH₂NHCOC₆H₁₃), 2.32 (m, 1H; COCH), 2.57 (t, ³*J*(HH) = 6 Hz, 2H; NCOCH₂CH₂CONH), 2.71 (t, ³*J*(HH) = 6 Hz, 2H; NCOCH₂CH₂CONH), 3.01 (m, 4H; CH₂NHCH₂), 3.19 (t, ³*J*(HH) = 2 Hz, 2H; NHC(NH₂)NHCH₂CH₂), 3.30 (m, 6H; NHC(NH₂)NHCH₂ + CONHCH₂ + CH₂NHCOC₆H₁₃), 3.44 (m, 4H; CH₂NCH₂); ¹³C NMR

(100 MHz, CD₃OD) δ = 15.3 (CH₂CH₃), 19.4 (CHCH₃), 24.6 (CH₂CH₃), 28.6 (CONHCH₂CH₂), 28.7 (CH₂CH₂NHCOC₆H₁₃), 29.1 (NHC(NH₂)NHCH₂CH₂), 29.8 (NCOCH₂CH₂CONH), 29.9 (NHC(NH₂)NHCH₂CH₂), 31.8 (CH₂CH₂CH₃), 32.3 (NCOCH₂CH₂CONH), 36.0 (COCH(CH₃)CH₂), 37.8 (CONHCH₂), 37.9 (CH₂NHCOC₆H₁₁), 40.9, 41.1 (NHC(NH₂)NHCH₂), 43.1 (COCH), 45.2, 47.2 (CH₂NCH₂), 47.4 (CONHCH₂CH₂CH₂), 47.5 (CH₂CH₂CH₂NHCOC₆H₁₃), 159.7, 159.8 (NHC(NH₂)NH), 75.4 (NCOCH₂CH₂CONH), 176.9 (NCOCH₂CH₂CONH), 177.8 (NHCOC₆H₁₃); MS (ES⁺): m/z (%): 271.4 (100) (M+2H)²⁺, 541.4 (3) (M+H)⁺; HRMS (ES⁺): m/z: calcd for C₂₅H₅₃N₁₀O₃ [M+H]⁺: 541.4297; found: 541.5307.

***N*¹-(3-(*N',N'*-bis-(3'(Guanidinopropyl)carbamoyl)propionyl)-*N*⁹-(5-methylhexanoyl)-norspermidine (2.12).**

Yield : (resin: 0.64 mmol/g, 41.9 mg) 21 mg, 58 %.

t_R = 5.7 min (RP-HPLC, 54 % pure); IR (Film): ν = 1655, 1621 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.91 (d, ³J(HH) = 7 Hz, 6H; CH(CH₃)₂), 1.21 (m, 2H; CH₂CH(CH₃)₂), 1.61 (m, 3H; CH₂CH₂CH(CH₃)₂), 1.82 (m, 2H; NHC(NH₂)NHCH₂CH₂), 1.91 (m, 6H; NHC(NH₂)NHCH₂CH₂ + CONHCH₂CH₂ + CH₂CH₂NHCOC₆H₁₃), 2.22 (t, ³J(HH) = 8 Hz, 2H; NHCOC₆H₁₃), 2.57 (m, 2H; NCOCH₂CH₂CONH), 2.71 (m, 2H; NCOCH₂CH₂CONH), 3.01 (m, 4H; CH₂NHCH₂), 3.19 (t, ³J(HH) = 7 Hz, 2H; NHC(NH₂)NHCH₂CH₂), 3.31 (m, 6H; NHC(NH₂)NHCH₂ + CONHCH₂ + CH₂NHCOC₆H₁₃), 3.45 (m, 4H; CH₂NCH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 23.3 (CH(CH₃)₂), 25.2 (COCH₂CH₂C₄H₉), 28.1 (CONHCH₂CH₂ + CH₂CH₂NHCOC₆H₁₃), 28.6 (NHC(NH₂)NHCH₂CH₂), 29.2 (NHC(NH₂)NHCH₂CH₂ + NCOCH₂CH₂CONH), 29.3 (CH₂CH(CH₃)₂), 31.7 (NCOCH₂CH₂CONH), 37.3 (CONHCH₂ + CH₂NHCOC₆H₁₃), 37.6 (NHCOC₆H₁₃), 40.0 (CH₂CH(CH₃)₂), 40.3, 40.5 (NHC(NH₂)NHCH₂), 44.6, 46.6 (CH₂NCH₂), 46.8 (CONHCH₂CH₂CH₂), 46.9 (CH₂CH₂CH₂NHCOC₆H₁₃), 159.2 (NHC(NH₂)NH), 174.9 (NCOCH₂CH₂CONH), 176.3 (NCOCH₂CH₂CONH), 177.8 (NHCOC₆H₁₃); MS (ES⁺): m/z (%): 271.3 (52) (M+2H)²⁺, 541.5 (6) (M+H)⁺; HRMS (ES⁺): m/z: calcd for C₂₅H₅₃N₁₀O₃ [M+H]⁺: 541.4297; found: 541.4299.

***N*¹-(3-(*N',N'*-bis-(3'(Guanidinopropyl)carbamoyl)propionyl)-*N*⁹-(octanoyl)-norspermidine (2.13).**

Yield : (resin: 0.64 mmol/g, 43.8 mg) 16 mg, 42 %.

*t*_R = 6.4 min (RP-HPLC, 78 % pure); IR (Film): $\nu = 1655, 1621 \text{ cm}^{-1}$ (C=O); ¹H NMR (400 MHz, CD₃OD) $\delta = 0.92$ (t, ³*J*(HH) = 7 Hz, 3H; CH₂CH₃), 1.33 (br s, 8H; CH₂(CH₂)₄CH₃), 1.62 (m, 2H; COCH₂CH₂C₅H₁₁), 1.82 (m, 2H; NHC(NH₂)NHCH₂CH₂), 1.91 (m, 6H; NHC(NH₂)NHCH₂CH₂ + CONHCH₂CH₂ + CH₂CH₂NHCOC₇H₁₅), 2.23 (t, ³*J*(HH) = 8 Hz, 2H; NHCOCH₂CH₂C₅H₁₁), 2.57 (t, ³*J*(HH) = 6 Hz, 2H; NCOCH₂CH₂CONH), 2.71 (t, ³*J*(HH) = 6 Hz, 2H; NCOCH₂CH₂CONH), 3.01 (m, 4H; CH₂NHCH₂), 3.19 (t, ³*J*(HH) = 7 Hz, 2H; NHC(NH₂)NHCH₂CH₂), 3.31 (m, 6H; NHC(NH₂)NHCH₂ + CONHCH₂ + CH₂NHCOC₇H₁₅), 3.44 (m, 4H; CH₂NCH₂); ¹³C NMR (100 MHz, CD₃OD) $\delta = 14.8$ (CH₂CH₃), 24.0 (CH₂CH₃), 27.4 (NHCOCH₂CH₂C₅H₁₁), 28.0 (CONHCH₂CH₂), 28.1 (CH₂CH₂CONHC₇H₁₅), 28.6 (NHC(NH₂)NHCH₂CH₂), 29.2 (NCOCH₂CH₂CONH), 29.3 (NHC(NH₂)NHCH₂CH₂), 30.5 (CH₂CH₂C₃H₇), 30.7 (CH₂CH₂C₃H₇), 31.7 (NCOCH₂CH₂CONH), 33.2 (CH₂CH₂CH₃), 37.3 (CONHCH₂ + CH₂NHCOC₇H₁₅), 37.4 (NHCOCH₂C₆H₁₃), 40.3, 40.5 (NHC(NH₂)NHCH₂), 44.6, 46.6 (CH₂NCH₂), 46.8 (CONHCH₂CH₂CH₂), 46.9 (CH₂CH₂CH₂NHCOC₇H₁₅), 159.1 (NHC(NH₂)NH), 174.8 (NCOCH₂CH₂CONH), 176.4 (NCOCH₂CH₂CONH), 177.8 (NHCOC₇H₁₅); MS (ES⁺): *m/z* (%): 278.3 (70) (M+2H)²⁺, 555.5 (8) (M+H)⁺; HRMS (ES⁺): *m/z*: calcd for C₂₆H₅₅N₁₀O₃ [M+H]⁺: 555.4453; found: 555.4456.

***N*¹-(3-(*N',N'*-bis-(3'(Guanidinopropyl)carbamoyl)propionyl)-*N*⁹-(2-methylheptanoyl)-norspermidine (2.14).**

Yield : (resin: 0.64 mmol/g, 42.5 mg) 18 mg, 49 %.

*t*_R = 6.2 min (RP-HPLC, 69 % pure); IR (Film): $\nu = 1654, 1621 \text{ cm}^{-1}$ (C=O); ¹H NMR (400 MHz, CD₃OD) $\delta = 0.91$ (t, ³*J*(HH) = 7 Hz, 3H; CH₂CH₃), 1.12 (d, ³*J*(HH) = 7 Hz, 3H; CHCH₃), 1.32 (m, 7H; CH(H)(CH₂)₃CH₃), 1.61 (m, 1H; CH(H)(CH₂)₃CH₃), 1.82 (m, 2H; NHC(NH₂)NHCH₂CH₂), 1.92 (m, 6H; NHC(NH₂)NHCH₂CH₂ + CONHCH₂CH₂ + CH₂CH₂NHCOC₇H₁₅), 2.32 (m, 1H; NHCOCH(CH₃)), 2.57 (t, ³*J*(HH) = 6 Hz, 2H; NCOCH₂CH₂CONH), 2.71 (t, ³*J*(HH) = 6 Hz, 2H; NCOCH₂CH₂CONH), 3.01 (m, 4H; CH₂NHCH₂), 3.19 (t, ³*J*(HH) = 7 Hz, 2H; NHC(NH₂)NHCH₂CH₂), 3.31 (m, 6H;

NHC(NH₂)NHCH₂ + CONHCH₂ + CH₂NHCOC₇H₁₅), 3.44 (m, 4H; CH₂NCH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 14.7 (CH₂CH₃), 18.8 (CHCH₃), 24.0 (CH₂CH₃), 28.0 (CONHCH₂CH₂), 28.1 (CH₂CH₂NHCOC₇H₁₅), 28.6 (NHC(NH₂)NHCH₂CH₂), 28.7 (CH₂CH₂CH₂CH₃), 29.2 (NCOCH₂CH₂CONH), 29.3 (NHC(NH₂)NHCH₂CH₂), 31.8 (NCOCH₂CH₂CONH), 33.3 (CH₂CH₂CH₃), 35.6 (COCH(CH₃)CH₂C₄H₉), 37.2 (CONHCH₂), 37.3 (CH₂NHCOC₇H₁₅), 40.3, 40.5 (NHC(NH₂)NHCH₂), 42.5 (NHCOCH(CH₃)C₅H₁₁), 44.6, 46.6 (CH₂NCH₂), 46.8 (CONHCH₂CH₂CH₂), 46.9 (CH₂CH₂CH₂NHCOC₇H₁₅), 159.1 (NHC(NH₂)NH), 174.8 (NCOCH₂CH₂CONH), 176.4 (NCOCH₂CH₂CONH), 181.1 (NHCOC₇H₁₅); MS (ES⁺): m/z (%): 278.4 (95) (M+2H)²⁺, 555.5 (8) (M+H)⁺; HRMS (ES⁺): m/z: calcd for C₂₆H₅₅N₁₀O₃ [M+H]⁺: 555.4453; found: 555.4450.

***N*¹-*(3-(N',N'*-bis-(3'(Guanidinopropyl)carbamoyl)propionyl)-N⁹-(2-ethylhexanoyl)-norspermidine (2.15).**

Yield : (resin: 0.64 mmol/g, 47.7 mg) 15 mg, 37 %.

*t*_R = 5.9 min (RP-HPLC, 64 % pure); IR (Film): ν = 1654, 1621 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.79 (m, 6H; 2xCH₂CH₃), 1.18 (m, 4H; CH₂CH₂CH₃), 1.40 (m, 4H; COCH(CH₂)₂), 1.71 (m, 2H; NHC(NH₂)NHCH₂CH₂), 1.80 (m, 6H; NHC(NH₂)NHCH₂CH₂ + CONHCH₂CH₂ + CH₂CH₂NHCOC₇H₁₅), 2.01 (m, 1H; COCH), 2.45 (m, 2H; NCOCH₂CH₂CONH), 2.60 (m, 2H; NCOCH₂CH₂CONH), 2.90 (m, 4H; CH₂NHCH₂), 3.07 (t, ³J(HH) = 7 Hz, 2H; NHC(NH₂)NHCH₂CH₂), 3.20 (m, 6H; NHC(NH₂)NHCH₂ + CONHCH₂ + CH₂NHCOC₇H₁₅), 3.33 (m, 4H; CH₂NCH₂); ¹³C NMR (100MHz, CD₃OD) δ = 12.8 (CH₂CH₃), 14.7 (CHCH₂CH₃), 24.1 (CH₂CH₃), 27.5 (CHCH₂CH₃), 28.0 (CONHCH₂CH₂), 28.2 (CH₂CH₂NHCOC₇H₁₅), 28.6 (NHC(NH₂)NHCH₂CH₂), 29.2 (NCOCH₂CH₂CONH), 29.3 (NHC(NH₂)NHCH₂CH₂), 31.4 (NCOCH₂CH₂CONH), 31.7 (CH₂CH₂CH₃), 33.9 (COCH(C₂H₅)CH₂), 37.3 (CONHCH₂ + CH₂NHCOC₇H₁₅), 40.3, 40.5 (NHC(NH₂)NHCH₂), 44.6, 46.6 (CH₂NCH₂), 46.8 (CONHCH₂CH₂CH₂), 47.0 (CH₂CH₂CH₂NHCOC₇H₁₅), 50.4 (NHCOCH), 159.2 (NHC(NH₂)NH), 174.8 (NCOCH₂CH₂CONH), 176.3 (NCOCH₂CH₂CONH), 180.2 (NHCOC₇H₁₅); MS (ES⁺): m/z (%): 278.4 (50) (M+2H)²⁺, 555.5 (16) (M+H)⁺; HRMS (ES⁺): m/z: calcd for C₂₆H₅₅N₁₀O₃ [M+H]⁺: 555.4453; found: 555.4462.

***N*¹-(3-(*N,N'*-bis-(3'(Guanidinopropyl)carbamoyl)propionyl)-*N*⁹-(2-propylpentanoyl)-norspermidine (2.16).**

Yield : (resin: 0.64 mmol/g, 41.7 mg) 19 mg, 53 %.

*t*_R = 5.9 min (RP-HPLC, 69 % pure); IR (Film): ν = 1655, 1621 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.93 (t, ³*J*(HH) = 7 Hz, 6H; (CH₂CH₃)₂), 1.30 (m, 4H; (CH₂CH₃)₂), 1.39, 1.56 (m, 4H; CH(CH₂CH₂CH₃)₂), 1.81 (m, 2H; NHC(NH₂)NHCH₂CH₂), 1.91 (m, 6H; NHC(NH₂)NHCH₂CH₂ + CONHCH₂CH₂ + CH₂CH₂NHCOC₇H₁₅), 2.24 (m, 1H; NHCOCH), 2.56 (t, ³*J*(HH) = 6 Hz, 2H; NCOCH₂CH₂CONH), 2.71 (t, ³*J*(HH) = 6 Hz, 2H; NCOCH₂CH₂CONH), 3.01 (m, 4H; CH₂NHCH₂), 3.18 (t, ³*J*(HH) = 7 Hz, 2H; NHC(NH₂)NHCH₂), 3.30 (m, 6H; NHC(NH₂)NHCH₂ + CONHCH₂ + CH₂NHCOC₇H₁₅), 3.44 (m, 4H; CH₂NCH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 14.8 ((CH₂CH₃)₂), 22.2 ((CH₂CH₃)₂), 28.0 (CONHCH₂CH₂), 28.1 (CH₂CH₂NHCOC₇H₁₅), 28.6 (NHC(NH₂)NHCH₂CH₂), 29.2 (NCOCH₂CH₂CONH), 29.3 (NHC(NH₂)NHCH₂CH₂), 31.7 (NCOCH₂CH₂CONH), 36.7 (CH(CH₂CH₂CH₃)₂), 37.3 (CONHCH₂ + CH₂NHCOC₇H₁₅), 40.3, 40.5 (NHC(NH₂)NHCH₂), 44.6, 46.6 (CH₂NCH₂), 46.8 (CONHCH₂CH₂CH₂) 47.0 (CH₂CH₂CH₂NHCOC₇H₁₅), 48.2 (NHCOCH), 159.2 (NHC(NH₂)NH), 174.8 (NCOCH₂CH₂CONH), 176.3 (NCOCH₂CH₂CONH), 180.4 (NHCOC₇H₁₅); MS (ES⁺): *m/z* (%): 278.4 (67) (M+2H)²⁺, 555.5 (5) (M+H)⁺; HRMS (ES⁺): *m/z*: calcd for C₂₆H₅₅N₁₀O₃ [M+H]⁺: 555.4453; found: 555.4454.

***N*¹-(3-(*N,N'*-bis-(3'(Guanidinopropyl)carbamoyl)propionyl)-*N*⁹-(nonanoyl)-norspermidine (2.17).**

Yield : (resin: 0.64 mmol/g, 50.0 mg) 16 mg, 36 %.

*t*_R = 7.1 min (RP-HPLC, 73 % pure); IR (Film): ν = 1667, 1628 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.80 (t, ³*J*(HH) = 7 Hz, 3H; CH₂CH₃), 1.22 (br s, 10H; CH₂(CH₂)₅CH₃), 1.51 (m, 2H; NHCOCH₂CH₂C₆H₁₃), 1.70 (m, 2H; NHC(NH₂)NHCH₂CH₂), 1.80 (m, 6H; NHC(NH₂)NHCH₂CH₂ + CONHCH₂CH₂ + CH₂CH₂NHCOC₈H₁₇), 2.12 (t, ³*J*(HH) = 8 Hz, 2H; NHCOCH₂CH₂C₆H₁₃), 2.45 (t, ³*J*(HH) = 6 Hz, 2H; NCOCH₂CH₂CONH), 2.60 (t, ³*J*(HH) = 6 Hz, 2H; NCOCH₂CH₂CONH), 2.90 (m, 4H; CH₂NHCH₂), 3.07 (t, ³*J*(HH) = 7 Hz, 2H; NHC(NH₂)NHCH₂), 3.20 (m, 6H; NHC(NH₂)NHCH₂ + CONHCH₂ + CH₂NHCOC₈H₁₇), 3.34 (m, 4H; CH₂NCH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 14.8

(CH₂CH₃), 24.1 (CH₂CH₃), 27.4 (COCH₂CH₂C₆H₁₃), 28.0 (CONHCH₂CH₂), 28.1 (CH₂CH₂NHCOC₈H₁₇), 28.6 (NHC(NH₂)NHCH₂CH₂), 29.2 (NCOCH₂CH₂CONH), 29.3 (NHC(NH₂)NHCH₂CH₂), 30.7, 30.8 (CH₂(CH₂)₃C₃H₇), 31.7 (NCOCH₂CH₂CONH), 33.4 (CH₂CH₂CH₃), 37.3 (CONHCH₂ + CH₂NHCOC₈H₁₇), 37.4 (NHCOCH₂C₇H₁₅), 40.3, 40.5 (NHC(NH₂)NHCH₂), 44.6, 46.6 (CH₂NCH₂), 46.8 (CONHCH₂CH₂CH₂), 46.9 (CH₂CH₂CH₂NHCOC₈H₁₇), 159.2 (NHC(NH₂)NH), 174.8 (NCOCH₂CH₂CONH), 176.3 (NCOCH₂CH₂CONH), 177.8 (NHCOC₈H₁₇); MS (ES⁺): m/z (%): 638.4 (5) (M+H+TFA)⁺; HRMS (ES⁺): m/z: calcd for C₂₇H₅₇N₁₀O₃ [M+H]⁺: 569.4610; found: 569.4628.

***N*¹-(3-(*N*'₂*N*'-bis-(3'(Guanidinopropyl)carbamoyl)propionyl)-*N*⁹-(4-methyloctanoyl)-norspermidine (2.18).**

Yield : (resin: 0.64 mmol/g, 41.2 mg) 19 mg, 53 %.

*t*_R = 6.7 min (RP-HPLC, 80 % pure); IR (Film): ν = 1650, 1621 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.81 (m, 6H; CHCH₃ + CH₂CH₃), 1.06, 1.21 (m, 6H; CH₂CH₂CH₂CH₃), 1.31, 1.54 (m, 3H; CH₂CH(CH₃)C₄H₉), 1.70 (m, 2H; NHC(NH₂)NHCH₂CH₂), 1.79 (m, 6H; NHC(NH₂)NHCH₂CH₂ + CONHCH₂CH₂ + CH₂CH₂NHCOC₈H₁₇), 2.12 (m, 2H; NHCOCH₂C₇H₁₅), 2.54 (t, ³J(HH) = 6 Hz, 2H; NCOCH₂CH₂CONH), 2.60 (t, ³J(HH) = 6 Hz, 2H; NCOCH₂CH₂CONH), 2.90 (m, 4H; CH₂NHCH₂), 3.07 (t, ³J(HH) = 7 Hz, 2H; NHC(NH₂)NHCH₂CH₂), 3.19 (m, 6H; NHC(NH₂)NHCH₂ + CONHCH₂ + CH₂NHCOC₈H₁₇), 3.34 (m, 4H; CH₂NCH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 14.8 (CH₂CH₃), 20.1 (CHCH₃), 24.4 (CH₂CH₃), 28.0 (CONHCH₂CH₂), 28.1 (CH₂CH₂NHCOC₈H₁₇), 28.6 (NHC(NH₂)NHCH₂CH₂), 29.2 (NCOCH₂CH₂CONH), 29.3 (NHC(NH₂)NHCH₂CH₂), 30.7 (CH₂CH₂CH₃), 31.7 (NCOCH₂CH₂CONH), 34.2 (CHCH₃), 34.5 (COCH₂CH₂C₆H₁₃), 35.2 (COCH₂C₇H₁₅), 37.3 (CONHCH₂ + CH₂NHCOC₈H₁₇), 37.9 (CH₂C₃H₇), 40.3, 40.5 (NHC(NH₂)NHCH₂), 44.6, 46.6 (CH₂NCH₂), 46.8, 46.9 (CH₂NHCH₂), 159.2 (NHC(NH₂)NH), 174.8 (NCOCH₂CH₂CONH), 176.3 (NCOCH₂CH₂CONH), 178.0 (NHCOC₈H₁₇); MS (ES⁺): m/z (%): 285.4 (70) (M+2H)²⁺, 569.6 (5) (M+H)⁺; HRMS (ES⁺): m/z: calcd for C₂₇H₅₇N₁₀O₃ [M+H]⁺: 569.4610; found: 569.4610.

***N*¹-(3-(*N',N'*-bis-(3'(Guanidinopropyl)carbamoyl)propionyl)-*N*⁹-(decanoyl)-norspermidine (2.19).**

Yield : (resin: 0.64 mmol/g, 38.3 mg) 16 mg, 47 %.

$t_R = 7.4$ min (RP-HPLC, 91 % pure); IR (Film): $\nu = 1657, 1627$ cm^{-1} (C=O); ¹H NMR (400 MHz, CD₃OD) $\delta = 0.91$ (t, ³*J*(HH) = 7 Hz, 3H; CH₂CH₃), 1.31 (br s, 12H; CH₂(CH₂)₆CH₃), 1.62 (m, 2H; COCH₂CH₂C₇H₁₅), 1.81 (m, 2H; NHC(NH₂)NHCH₂CH₂), 1.91 (m, 6H; NHC(NH₂)NHCH₂CH₂ + CONHCH₂CH₂ + CH₂CH₂NHCOC₉H₁₉), 2.22 (t, ³*J*(HH) = 8 Hz, 2H; NHCOCH₂C₈H₁₇), 2.56 (t, ³*J*(HH) = 6 Hz, 2H; NCOCH₂CH₂CONH), 2.71 (t, ³*J*(HH) = 6 Hz, 2H; NCOCH₂CH₂CONH), 2.99, 3.03 (m, 4H; CH₂NHCH₂), 3.18 (t, ³*J*(HH) = 7 Hz, 2H; NHC(NH₂)NHCH₂CH₂), 3.29 (m, 6H; NHC(NH₂)NHCH₂ + NHCOCH₂ + CH₂NHCOC₉H₁₉), 3.43 (m, 4H; CH₂NCH₂); ¹³C NMR (100 MHz, CD₃OD) $\delta = 14.8$ (CH₂CH₃), 24.1 (CH₂CH₃), 27.4 (COCH₂CH₂C₇H₁₅), 28.0 (NHCOCH₂CH₂), 28.1 (CH₂CH₂NHCOC₉H₁₉), 28.5 (NHC(NH₂)NHCH₂CH₂), 29.2 (NCOCH₂CH₂CONH), 29.3 (NHC(NH₂)NHCH₂CH₂), 30.7, 30.8, 30.9 (CH₂(CH₂)₄C₃H₇), 31.7 (NCOCH₂CH₂CONH), 33.4 (CH₂CH₂CH₃), 37.3 (CONHCH₂ + CH₂NHCOC₉H₁₉), 37.4 (NHCOCH₂C₈H₁₇), 40.3, 40.5 (NHC(NH₂)NHCH₂), 44.6, 46.6 (CH₂NCH₂), 46.8, 46.9 (CH₂NHCH₂), 159.2 (NHC(NH₂)NH), 174.8 (NCOCH₂CH₂CONH), 176.3 (NCOCH₂CH₂CONH), 177.8 (NHCOC₉H₁₉); MS (ES⁺): *m/z* (%): 292.3 (100) (M+2H)²⁺, 697.3 (13) (M+TFA)⁺; HRMS (ES⁺): *m/z*: calcd for C₂₈H₅₉N₁₀O₃ [M+H]⁺: 583.4766; found: 583.4759.

***N*¹-(3-(*N',N'*-bis-(3'(Guanidinopropyl)carbamoyl)propionyl)-*N*⁹-(4-methylnonanoyl)-norspermidine (2.20).**

Yield : (resin: 0.64 mmol/g, 38.8 mg) 20 mg, 59 %.

$t_R = 7.2$ min (RP-HPLC, 82 % pure); IR (Film): $\nu = 1662, 1623$ cm^{-1} (C=O); ¹H NMR (400 MHz, CD₃OD) $\delta = 0.92$ (m, 6H; CHCH₃ + CH₂CH₃), 1.17, 1.32 (m, 8H; CH(CH₂)₄CH₃), 1.43 (m, 2H; COCH₂CHHCH), 1.65 (m, 1H; COCH₂CHHCH), 1.82 (m, 2H; NHC(NH₂)NHCH₂CH₂), 1.91 (m, 6H; NHC(NH₂)NHCH₂CH₂ + CONHCH₂CH₂ + CH₂CH₂NHCOC₉H₁₉), 2.24 (m, 2H; COCH₂C₈H₁₇), 2.57 (t, ³*J*(HH) = 6 Hz, 2H; NCOCH₂CH₂CONH), 2.71 (t, ³*J*(HH) = 6 Hz, 2H; NCOCH₂CH₂CONH), 2.99 (m, 2H; CH₂CH₂CH₂NHCOC₉H₁₉), 3.03 (m, 2H; CONHCH₂CH₂CH₂), 3.19 (t, ³*J*(HH) = 7 Hz, 2H; NHC(NH₂)NHCH₂CH₂), 3.31 (m, 6H; NHC(NH₂)NHCH₂ + CONHCH₂ + CH₂NHCOC₉H₁₉),

3.43 (m, 4H; CH_2NCH_2); ^{13}C NMR (100 MHz, CD_3OD) δ = 14.8 ($CHCH_3$), 20.1 (CH_2CH_3), 24.1 (CH_2CH_3), 28.0 ($CONHCH_2CH_2$), 28.1 ($CH_2CH_2NHCOC_9H_{19}$ + $CH_2CH_2CH_2CH_3$), 28.6 ($NHC(NH_2)NHCH_2CH_2$), 29.2 ($NCOCH_2CH_2CONH$), 29.3 ($NHC(NH_2)NHCH_2CH_2$), 31.7 ($NCOCH_2CH_2CONH$), 33.7 ($CHCH_3$), 34.2 ($CH_2CH_2CH_3$), 34.5 ($COCH_2CH_2C_7H_{15}$), 35.2 ($COCH_2C_8H_{17}$), 37.3 ($CONHCH_2$ + $CH_2NHCOC_9H_{19}$), 38.2 ($CH_2C_4H_9$), 40.3, 40.5 ($NHC(NH_2)NHCH_2$), 44.6, 46.6 (CH_2NCH_2), 46.8, 46.9 (CH_2NHCH_2), 159.1 ($NHC(NH_2)NH$), 174.8 ($NCOCH_2CH_2CONH$), 176.3 ($NCOCH_2CH_2CONH$), 178.0 ($NHCOC_9H_{19}$); MS (ES^+): m/z (%):292.3 (100) ($M+2H$) $^{2+}$, 697.3 (12) ($M+TFA$) $^+$; HRMS (ES^+): m/z: calcd for $C_{28}H_{59}N_{10}O_3$ [$M+H$] $^+$: 583.4766; found: 583.4757.

***N*¹-(3-(*N',N'*-bis-(3'(Guanidinopropyl)carbamoyl)propionyl)-*N*⁹-(4-ethyloctanoyl)-norspermidine (2.21).**

Yield : (resin: 0.64 mmol/g, 39.6 mg) 24 mg, 69 %.

t_R = 7.1 min (RP-HPLC, 83 % pure); IR (Film): ν = 1666, 1627 cm^{-1} (C=O); 1H NMR (400 MHz, CD_3OD) δ = 0.91 (m, 6H; $CHCH_2CH_3$ + $CH_2CH_2CH_3$), 1.31 (m, 9H; $CH(CH_2CH_3)(CH_2)_3CH_3$), 1.59 (m, 2H; $COCH_2CH_2C_7H_{15}$), 1.82 (m, 2H; $NHC(NH_2)NHCH_2CH_2$), 1.91 (m, 6H; $NHC(NH_2)NHCH_2CH_2$ + $CONHCH_2CH_2$ + $CH_2CH_2NHCOC_9H_{19}$), 2.22 (t, $^3J(HH)$ = 8 Hz, 2H; $CHCOCH_2C_8H_{17}$), 2.57 (t, $^3J(HH)$ = 6 Hz, 2H; $NCOCH_2CH_2CONH$), 2.71 (t, $^3J(HH)$ = 6 Hz, 2H; $NCOCH_2CH_2CONH$), 2.99, 3.03 (m, 4H; CH_2NHCH_2), 3.19 (t, $^3J(HH)$ = 7 Hz, 2H; $NHC(NH_2)NHCH_2CH_2$), 3.30 (m, 6H; $NHC(NH_2)NHCH_2$ + $CONHCH_2$ + $CH_2NHCOC_9H_{19}$), 3.43 (m, 4H; CH_2NCH_2); ^{13}C NMR (100 MHz, CD_3OD) δ = 11.5 ($CH_2CH_2CH_3$), 14.8 ($CHCH_2CH_3$), 24.5 ($CHCH_2CH_3$), 27.0 ($CH_2CH_2CH_3$), 28.0 ($CONHCH_2CH_2$), 28.1 ($CH_2CH_2NHCOC_9H_{19}$), 28.6 ($NHC(NH_2)NHCH_2CH_2$), 29.2 ($NCOCH_2CH_2CONH$), 29.3 ($NHC(NH_2)NHCH_2CH_2$), 30.4 ($CH_2CH_2CH_3$), 30.8 ($COCH_2CH_2C_7H_{15}$), 31.7 ($NCOCH_2CH_2CONH$), 34.0 ($CH_2C_3H_7$), 34.9 ($COCH_2C_8H_{17}$), 37.4 ($CONHCH_2$ + $CH_2NHCOC_9H_{19}$), 40.4 ($CHCH_2CH_2$ + $NHC(NH_2)NHCH_2$), 40.5 ($NHC(NH_2)NHCH_2$), 44.6, 46.6 (CH_2NCH_2), 46.8, 46.9 (CH_2NHCH_2), 159.1, 159.2 ($NHC(NH_2)NH$), 174.8 ($NCOCH_2CH_2CONH$), 176.3 ($NCOCH_2CH_2CONH$), 178.0 ($NHCOC_9H_{19}$); MS (ES^+): m/z (%):292.3 (100) ($M+2H$) $^{2+}$, 697.3 (12) ($M+TFA$) $^+$; HRMS (ES^+): m/z: calcd for $C_{28}H_{59}N_{10}O_3$ [$M+H$] $^+$: 583.4766; found: 583.4762.

***N*¹-(3-(*N,N'*-bis-(3'(Guanidinopropyl)carbamoyl)propionyl)-*N*⁹-(undecanoyl)-norspermidine (2.22).**

Yield : (resin: 0.64 mmol/g, 38.4 mg) 24 mg, 71 %.

*t*_R = 7.7 min (RP-HPLC, 85 % pure); IR (Film): ν = 1666, 1626 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.91 (t, ³*J*(HH) = 7 Hz, 3H; CH₂CH₃), 1.30 (br s, 14H; CH₂(CH₂)₇CH₃), 1.62 (m, 2H; COCH₂CH₂C₈H₁₇), 1.82 (m, 2H; NHC(NH₂)NHCH₂CH₂), 1.91 (m, 6H; NHC(NH₂)NHCH₂CH₂ + CONHCH₂CH₂ + CH₂CH₂NHCOC₁₀H₂₁), 2.23 (t, ³*J*(HH) = 8 Hz, 2H; NHCOC₂C₉H₁₉), 2.57 (t, ³*J*(HH) = 6 Hz, 2H; NCOCH₂CH₂CONH), 2.71 (t, ³*J*(HH) = 6 Hz, 2H; NCOCH₂CH₂CONH), 2.99, 3.03 (m, 4H; CH₂NHCH₂), 3.19 (t, ³*J*(HH) = 7 Hz, 2H; NHC(NH₂)NHCH₂), 3.31 (m, 6H; NHC(NH₂)NHCH₂ + CONHCH₂ + CH₂NHCOC₁₀H₂₁), 3.44 (m, 4H; CH₂NCH₂); ¹³C NMR (100 MHz, CD₃OD), δ = 14.8 (CH₂CH₃), 24.1 (CH₂CH₃), 27.4 (COCH₂CH₂C₈H₁₇), 28.0 (CONHCH₂CH₂), 28.1 (CH₂CH₂NHCOC₁₀H₂₁), 28.6 (NHC(NH₂)NHCH₂CH₂), 29.2 (NCOCH₂CH₂CONH), 29.3 (NHC(NH₂)NHCH₂CH₂), 30.7, 30.8, 31.0, 31.1 (CH₂(CH₂)₅C₃H₇), 31.7 (NCOCH₂CH₂CONH), 33.4 (CH₂CH₂CH₃), 37.3 (CONHCH₂ + CH₂NHCOC₁₀H₂₁), 37.4 (COCH₂C₉H₁₉), 40.3, 40.5 (NHC(NH₂)NHCH₂), 44.6, 46.6 (CH₂NCH₂), 46.8, 46.9 (CH₂NHCH₂), 159.1, 159.2 (NHC(NH₂)NH), 174.8 (NCOCH₂CH₂CONH), 176.3 (NCOCH₂CH₂CONH), 177.7 (NHCOC₁₀H₂₁); MS (ES⁺): *m/z* (%): 299.4 (100) (M+2H)²⁺, 711.4 (12) (M+TFA)⁺; HRMS (ES⁺): *m/z*: calcd for C₂₉H₆₁N₁₀O₃ [M+H]⁺: 597.4923; found: 597.4524.

***N*¹-(3-(*N,N'*-bis-(3'(Guanidinopropyl)carbamoyl)propionyl)-*N*⁹-(dodecanoyl)-norspermidine (2.23).**

Yield : (resin: 0.64 mmol/g, 41.1 mg) 26 mg, 70 %.

*t*_R = 8.0 min (RP-HPLC, 90 % pure); IR (Film): ν = 1657, 1627 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.67 (t, ³*J*(HH) = 7 Hz, 3H; CH₂CH₃), 1.06 (br s, 16H; CH₂(CH₂)₈CH₃), 1.38 (m, 2H; COCH₂CH₂C₉H₁₉), 1.58 (m, 2H; NHC(NH₂)NHCH₂CH₂), 1.67 (m, 6H; NHC(NH₂)NHCH₂CH₂ + CONHCH₂CH₂ + CH₂CH₂NHCOC₁₁H₂₃), 1.99 (t, ³*J*(HH) = 8 Hz, 2H; COCH₂CH₂C₉H₁₉), 2.32 (t, ³*J*(HH) = 6 Hz, 2H; NCOCH₂CH₂CONH), 2.47 (t, ³*J*(HH) = 6 Hz, 2H; NCOCH₂CH₂CONH), 2.75, 2.79 (m, 4H; CH₂NHCH₂), 2.94 (t, ³*J*(HH) = 7 Hz, 2H; NHC(NH₂)NHCH₂), 3.06 (m, 6H; NHC(NH₂)NHCH₂ + CONHCH₂ + CH₂NHCOC₁₁H₂₃), 3.20 (m, 4H; CH₂NCH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 14.8

(CH₂CH₃), 24.1 (CH₂CH₃), 27.4 (COCH₂CH₂C₉H₁₉), 28.0 (CONHCH₂CH₂), 28.1 (CH₂CH₂NHCOC₁₁H₂₃), 28.6 (NHC(NH₂)NHCH₂CH₂), 29.2 (NCOCH₂CH₂CONH), 29.3 (NHC(NH₂)NHCH₂CH₂), 30.7, 30.8, 31.0, 31.1 (CH₂(CH₂)₆C₃H₇), 31.7 (NCOCH₂CH₂CONH), 33.4 (CH₂CH₂CH₃), 37.3 (CONHCH₂ + CH₂NHCOC₁₁H₂₃), 37.4 (NHCOCH₂C₁₀H₂₁), 40.3, 40.5 (NHC(NH₂)NHCH₂), 44.6, 46.6 (CH₂NCH₂), 46.8, 46.9 (CH₂NHCH₂), 159.2 (NHC(NH₂)NH), 174.8 (NCOCH₂CH₂CONH), 176.3 (NCOCH₂CH₂CONH), 177.7 (NHCOC₁₁H₂₃); MS (ES⁺): m/z (%): 306.4 (100) (M+2H)²⁺, 725.5 (10) (M+TFA)⁺; HRMS (ES⁺): m/z: calcd for C₃₀H₆₃N₁₀O₃ [M+H]⁺: 611.5077; found: 611.5079.

***N*¹-(3-(*N,N'*-bis-(3'(Guanidinopropyl)carbamoyl)propionyl)-*N*⁹-(tetradecanoyl)-norspermidine (2.24).**

Yield : (resin: 0.64 mmol/g, 38.1 mg) 27 mg, 77 %.

*t*_R = 8.7 min (RP-HPLC, 77 % pure); IR (Film): ν = 1657, 1627 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.91 (t, ³J(HH) = 7 Hz, 3H; CH₂CH₃), 1.30 (br s, 20H; CH₂(CH₂)₁₀CH₃), 1.62 (m, 2H; NHCOCH₂CH₂C₁₁H₂₃), 1.82 (m, 2H; NHC(NH₂)NHCH₂CH₂), 1.91 (m, 6H; NHC(NH₂)NHCH₂CH₂ + CONHCH₂CH₂ + CH₂CH₂NHCOC₁₃H₂₇), 2.23 (t, ³J(HH) = 8 Hz, 2H; NHCOCH₂CH₂C₁₁H₂₃), 2.57 (t, ³J(HH) = 6 Hz, 2H; NCOCH₂CH₂CONH), 2.71 (t, ³J(HH) = 6 Hz, 2H; NCOCH₂CH₂CONH), 2.99, 3.03 (m, 4H, CH₂NHCH₂), 3.19 (t, ³J(HH) = 7 Hz, 2H; NHC(NH₂)NHCH₂CH₂), 3.30 (m, 6H; NHC(NH₂)NHCH₂ + CONHCH₂ + CH₂NHCOC₁₃H₂₇), 3.44 (m, 4H, CH₂NCH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 14.8 (CH₂CH₃), 24.1 (CH₂CH₃), 27.4 (COCH₂CH₂C₁₁H₂₃), 28.0 (NHCOCH₂CH₂), 28.1 (CH₂CH₂NHCOC₁₃H₂₇), 28.6 (NHC(NH₂)NHCH₂CH₂), 29.2 (NCOCH₂CH₂CONH), 29.3 (NHC(NH₂)NHCH₂CH₂), 30.7, 30.8, 31.0, 31.1 (CH₂(CH₂)₈C₃H₇), 31.7 (NCOCH₂CH₂CONH), 33.4 (CH₂CH₂CH₃), 37.3 (CONHCH₂ + CH₂NHCOC₁₃H₂₇), 37.4 (NHCOCH₂C₁₂H₂₅), 40.3, 40.5 (NHC(NH₂)NHCH₂), 44.6, 46.6 (CH₂NCH₂), 46.8, 46.9 (CH₂NHCH₂), 159.2 (NHC(NH₂)NH), 174.8 (NCOCH₂CH₂CONH), 176.3 (NCOCH₂CH₂CONH), 177.8 (NHCOC₁₃H₂₇); MS (ES⁺): m/z (%): 320.4 (100) (M+2H)²⁺, 753.5 (10) (M+TFA)⁺; HRMS (ES⁺): m/z: calcd for C₃₂H₆₇N₁₀O₃ [M+H]⁺: 639.5392; found: 639.5373.

***N*¹-(3-(*N',N'*-bis-(3'(Guanidinopropyl)carbamoyl)propionyl)-*N*⁹-(pentadecanoyl)-norspermidine (2.25).**

Yield : (resin: 0.64 mmol/g, 42.4 mg) 23 mg, 59 %.

*t*_R = 8.8 min (RP-HPLC, 86 % pure); IR (Film): ν = 1655, 1626 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.91 (t, ³*J*(HH) = 7 Hz, 3H; CH₂CH₃), 1.30 (br s, 22H; CH₂(CH₂)₁₁CH₃), 1.62 (m, 2H; COCH₂CH₂C₁₂H₂₅), 1.82 (m, 2H; NHC(NH₂)NHCH₂CH₂), 1.91 (m, 6H; NHC(NH₂)NHCH₂CH₂ + CH₂CH₂NHCH₂CH₂), 2.23 (t, ³*J*(HH) = 8 Hz, 2H; COCH₂CH₂C₁₁H₂₃), 2.57 (m, 2H; NCOCH₂CH₂CONH), 2.71 (m, 2H; NCOCH₂CH₂CONH), 3.01 (m, 4H; CH₂NHCH₂), 3.19 (t, ³*J*(HH) = 7 Hz, 2H; NHC(NH₂)NHCH₂CH₂), 3.30 (m, 6H; NHC(NH₂)NHCH₂ + CONHCH₂ + CH₂NHCOC₁₄H₂₉), 3.44 (m, 4H; CH₂NCH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 14.8 (CH₂CH₃), 24.1 (CH₂CH₃), 27.4 (COCH₂CH₂C₁₂H₂₅), 28.0, 28.1 (CH₂CH₂NHCH₂CH₂), 28.6 (NHC(NH₂)NHCH₂CH₂), 29.2 (NCOCH₂CH₂CONH), 29.3 (NHC(NH₂)NHCH₂CH₂), 30.8, 31.0, 31.1 (CH₂(CH₂)₉C₃H₇), 31.7 (NCOCH₂CH₂CONH), 33.4 (CH₂CH₂CH₃), 37.3 (CONHCH₂ + CH₂NHCOC₁₄H₂₉), 37.4 (NHCOCH₂C₁₃H₂₇), 40.3, 40.5 (NHC(NH₂)NHCH₂), 44.6, 46.6 (CH₂NCH₂), 46.8, 46.9 (CH₂NHCH₂), 159.1 (NHC(NH₂)NH), 174.8 (NCOCH₂CH₂CONH), 176.3 (NCOCH₂CH₂CONH), 177.8 (NHCOC₁₄H₂₉); MS (ES⁺): *m/z* (%): 327.5 (60) (M+2H)²⁺, 653.8 (5) (M+H)⁺; HRMS (ES⁺): *m/z*: calcd for C₃₃H₆₉N₁₀O₃ [M+H]⁺: 653.5549; found: 653.5565.

***N*¹-(3-(*N',N'*-bis-(3'(Guanidinopropyl)carbamoyl)propionyl)-*N*⁹-(pamitoyl)-norspermidine (2.26).**

Yield : (resin: 0.64 mmol/g, 41.2 mg) 23 mg, 59 %.

*t*_R = 9.1 min (RP-HPLC, 88 % pure); IR (Film): ν = 1655, 1636 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.91 (t, ³*J*(HH) = 7 Hz, 3H; CH₂CH₃), 1.30 (br s, 24H; CH₂(CH₂)₁₂CH₃), 1.62 (m, 2H; COCH₂CH₂C₁₂H₂₅), 1.81 (m, 2H; NHC(NH₂)NHCH₂CH₂), 1.90 (m, 6H; NHC(NH₂)NHCH₂CH₂ + CH₂CH₂NHCH₂CH₂), 2.22 (t, ³*J*(HH) = 8 Hz, 2H; COCH₂CH₂C₁₂H₂₅), 2.56 (t, ³*J*(HH) = 6 Hz, 2H; NCOCH₂CH₂CONH), 2.71 (t, ³*J*(HH) = 6 Hz, 2H; NCOCH₂CH₂CONH), 2.99, 3.03 (m, 4H; CH₂NHCH₂), 3.18 (t, ³*J*(HH) = 7 Hz, 2H; NHC(NH₂)NHCH₂CH₂), 3.30 (m, 6H; NHC(NH₂)NHCH₂ + CONHCH₂ + CH₂NHCOC₁₅H₃₁), 3.44 (m, 4H; CH₂NCH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 14.8

(CH₂CH₃), 24.1 (CH₂CH₃), 27.4 (COCH₂CH₂C₁₃H₂₇), 28.0, 28.1 (CH₂CH₂NHCH₂CH₂), 28.6 (NHC(NH₂)NHCH₂CH₂), 29.2 (NCOCH₂CH₂CONH), 29.3 (NHC(NH₂)NHCH₂CH₂), 30.7, 30.8, 31.0, 31.1 (CH₂(CH₂)₁₀C₃H₇), 31.7 (NCOCH₂CH₂CONH), 30.4 (CH₂CH₂CH₃), 37.3 (CONHCH₂ + CH₂NHCOC₁₅H₃₁), 37.4 (COCH₂C₁₄H₂₉), 40.3, 40.5 (NHC(NH₂)NHCH₂), 44.6, 46.6 (CH₂NCH₂), 46.8, 46.9 (CH₂NHCH₂), 159.2 (NHC(NH₂)NH) 174.8 (NCOCH₂CH₂CONH), 176.3 (NCOCH₂CH₂CONH), 177.7 (NHCOC₁₅H₃₁); MS (ES⁺): m/z (%): 334.5 (100) (M+2H)²⁺, 781.4 (12) (M+TFA)⁺; HRMS (ES⁺): m/z: calcd for C₃₄H₇₁N₁₀O₃ [M+H]⁺: 667.5705; found: 667.5707.

***N*¹-(3-(*N',N'*-bis-(3'(Guanidinopropyl)carbamoyl)propionyl)-*N*⁹-(oleoyl)-norspermidine (2.27).**

Yield : (resin: 0.64 mmol/g, 41.2 mg) 26 mg, 67 %.

*t*_R = 9.4 min (RP-HPLC, 68 % pure); IR (Film): ν = 1655, 1621 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.91 (t, ³J(HH) = 7 Hz, 3H; CH₂CH₃), 1.32 (br s, 20H; CH₂(CH₂)₄CH₂CHCHCH₂(CH₂)₆CH₃), 1.62 (m, 2H; COCH₂CH₂C₁₅H₂₉), 1.82 (m, 2H; NHC(NH₂)NHCH₂CH₂), 1.90 (m, 6H; NHC(NH₂)NHCH₂CH₂ + CH₂CH₂NHCH₂CH₂), 2.04 (m, 4H; CH₂CHCHCH₂), 2.23 (t, ³J(HH) = 8 Hz, 2H; COCH₂CH₂C₁₅H₂₉), 2.57 (t, ³J(HH) = 6 Hz, 2H; NCOCH₂CH₂CONH), 2.71 (t, ³J(HH) = 6 Hz, 2H; NCOCH₂CH₂CONH), 3.01 (m, 4H; CH₂NHCH₂), 3.19 (t, ³J(HH) = 7 Hz, 2H; NHC(NH₂)NHCH₂CH₂), 3.31 (m, 6H; NHC(NH₂)NHCH₂ + CONHCH₂ + CH₂NHCOC₁₇H₃₃), 3.44 (m, 4H; CH₂NCH₂), 5.36 (m, 2H; CH₂CHCHCH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 14.8 (CH₂CH₃), 24.1 (CH₂CH₃), 27.4 (COCH₂CH₂C₁₅H₂₉), 28.0, 28.1 (CH₂CH₂NHCH₂CH₂), 28.5 (CH₂CHCHCH₂), 28.6 (NHC(NH₂)NHCH₂CH₂), 29.2 (NCOCH₂CH₂CONH), 29.3 (NHC(NH₂)NHCH₂CH₂), 30.6, 30.7, 30.8, 31.0, 31.2 (CH₂(CH₂)₄CH₂CHCHCH₂(CH₂)₄C₃H₇), 31.7 (NCOCH₂CH₂CONH), 33.4 (CH₂CH₂CH₃), 37.3 (CONHCH₂ + CH₂NHCOC₁₇H₃₃), 37.4 (COCH₂C₁₆H₃₁), 40.3, 40.5 (NHC(NH₂)NHCH₂), 44.6, 46.6 (CH₂NCH₂), 46.8, 46.9 (CH₂NHCH₂), 131.1, 131.3 (CH₂CHCHCH₂), 159.2 (NHC(NH₂)NH), 174.8 (NCOCH₂CH₂CONH), 176.3 (NCOCH₂CH₂CONH), 177.7 (NHCOC₁₇H₃₃); MS (ES⁺): m/z (%): 347.6 (100) (M+2H)²⁺, 694.8 (4) (M+H)⁺; HRMS (ES⁺): m/z: calcd for C₃₆H₇₃N₁₀O₃ [M+H]⁺: 693.5862; found: 693.5869.

***N*¹-(3-(*N',N'*-bis-(3'(Guanidinopropyl)carbamoyl)propionyl)-*N*⁹-(3a,7a,12a-trihydroxyl-5b-cholan-24-carbonyl)-norspermidine (2.28).**

Yield : (resin: 0.64 mmol/g, 42.9 mg) 32 mg, 73 %.

*t*_R = 7.3 min (RP-HPLC, 55 % pure); IR (Film): ν = 1661 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD, selected data) δ = 0.73 (s, 3H, 18'-CH₃), 0.93 (s, 3H, 19'-CH₃), 1.05 (d, ³*J*(HH) = 6 Hz, 3H; 21'-CH₃), 1.89 (m, 8H; CH₂CH₂NHCH₂CH₂ + CH₂CH₂NCH₂CH₂), 2.29 (m, 2H; 23'-CH₂), 2.57 (m, 2H; NHCOCH₂CH₂CON), 2.71 (m, 2H; NHCOCH₂CH₂CON), 2.95-3.08 (m, 4H; CH₂NHCH₂), 3.19 (t, ³*J*(HH) = 7 Hz, 2H; NHC(NH₂)NHCH₂), 3.25-3.34 (m, 6H; NHC(NH₂)NHCH₂ + CONHCH₂ + CH₂NHCO), 3.39-3.50 (m, 5H; 3' β -CH + CH₂NCH₂), 3.81 (s, 1H; 7' β -CH); 3.97 (s, 1H; 12' β -CH). ¹³C NMR (100MHz, CD₃OD, selected data according to HMBC) δ = 13.4 (18'-C), 18.1 (21'-C), 23.5 (19'-C), 28.1 (St-CONHCH₂CH₂), 28.3 (CH₂CH₂NHCO), 28.5 (NHC(NH₂)NHCH₂CH₂), 29.2 (NHCOCH₂CH₂CON), 29.3 (NHC(NH₂)NHCH₂CH₂), 31.7 (NHCOCH₂CH₂CON), 34.4 (23'-C), 36.3 (22'-C), 37.3 (St-CONHCH₂ + CH₂NHCO), 40.3, 40.5 (NHC(NH₂)NHCH₂), 44.6, 46.6 (CH₂NCH₂), 46.8, 46.9 CH₂NHCH₂), 69.5 (7'-C), 73.2 (3'-C), 74.4 (12'-C), 159.1 (NHC(NH₂)NH), 174.8 (NHCOCH₂CH₂CON), 176.3 (NHCOCH₂CH₂CON), 178.3 (24'-C=O); MS (ES⁺): m/z (%): 410.5 (100) (M+2H)²⁺; HRMS (ES⁺): m/z: calcd for C₄₂H₈₀N₁₀O₆ [M+2H]²⁺: 410.3126; found: 410.3129.

***N*¹-(3-(*N',N'*-bis-(3'(Guanidinopropyl)carbamoyl)propionyl)-*N*⁹-(3a,12a-dihydroxyl-5b-cholan-24-carbonyl)-norspermidine (2.29).**

Yield : (resin: 0.64 mmol/g, 40.2 mg) 30 mg, 73 %.

*t*_R = 7.8 min (RP-HPLC, 79 % pure); IR (Film): ν = 1661 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD, selected data) δ = 0.72 (s, 3H, 18'-CH₃), 0.94 (s, 3H, 19'-CH₃), 1.04 (d, ³*J*(HH) = 6 Hz, 3H; 21'-CH₃), 1.89 (m, 8H; CH₂CH₂NHCH₂CH₂ + CH₂CH₂NCH₂CH₂), 2.15, 2.29 (m, 2H; 23'-CH₂), 2.57 (m, 2H; NHCOCH₂CH₂CON), 2.71 (m, 2H; NHCOCH₂CH₂CON), 2.91-3.10 (m, 4H; CH₂NHCH₂), 3.18 (t, ³*J*(HH) = 7 Hz, 2H; NHC(NH₂)NHCH₂), 3.25-3.34 (m, 6H; NHC(NH₂)NHCH₂ + CONHCH₂ + CH₂NHCO), 3.40-3.50 (m, 4H; CH₂NCH₂), 3.55 (m, 1H; 3' β -CH), 3.97 (s, 1H; 12' β -CH). ¹³C NMR (100MHz, CD₃OD, selected data according to HMBC) δ = 13.6 (18'-C), 18.0 (21'-C), 24.1 (19'-C), 28.1 (St-CONHCH₂CH₂), 28.3

(CH₂CH₂NHCO), 28.6 (NHC(NH₂)NHCH₂CH₂), 29.2 (NHCOCH₂CH₂CON), 29.3 (NHC(NH₂)NHCH₂CH₂), 31.7 (NHCOCH₂CH₂CON), 34.4 (23'-C), 37.3 (St-CONHCH₂ + CH₂NHCO), 40.3, 40.5 (NHC(NH₂)NHCH₂), 44.6, 46.6 (CH₂NCH₂), 46.8, 46.9 CH₂NHCH₂), 72.9 (3'-C), 74.4 (12'-C), 159.1, 159.2 (NHC(NH₂)NH), 174.8 (NHCOCH₂CH₂CON), 176.3 (NHCOCH₂CH₂CON), 178.3 (24'-C=O); MS (ES⁺): m/z (%): 402.4 (100) (M+2H)²⁺; HRMS (ES⁺): m/z: calcd for C₄₂H₈₀N₁₀O₆ [M+2H]²⁺: 402.3151; found: 402.3152.

***N*¹-(3-(*N',N'*-bis-(3'(Guanidinopropyl)carbamoyl)propionyl)-*N*⁹-(3,7,12-trioxo-5b-cholan-24-carbonyl)-norspermidine (2.30).**

Yield : (resin: 0.64 mmol/g, 40.8 mg) 25 mg, 60 %.

*t*_R = 6.8 min (RP-HPLC, 83 % pure); IR (Film): ν = 1665 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD, selected data) δ = 0.87 (d, ³*J*(HH) = 7 Hz, 3H; 21'-CH₃), 1.10 (s, 3H; 19'-CH₃), 1.36 (s, 3H; 18'-CH₃), 1.80-1.94 (m, 8H; CH₂CH₂NHCH₂CH₂ + CH₂CH₂NCH₂CH₂), 2.57 (t, ³*J*(HH) = 6 Hz, 2H; NHCOCH₂CH₂CON), 2.71 (t, ³*J*(HH) = 6 Hz, 2H; NHCOCH₂CH₂CON), 2.96-3.09 (m, 4H; CH₂NHCH₂), 3.19 (t, ³*J*(HH) = 7 Hz, 2H; NHC(NH₂)NHCH₂); 3.24-3.35 (m, 6H; NHC(NH₂)NHCH₂ + CONHCH₂ + CH₂NHCO), 3.40-3.51 (m, 4H; CH₂NCH₂). ¹³C NMR (100MHz, CD₃OD) δ = 12.5, 19.6, 23.1, 26.4, 28.0, 28.1, 28.3, 28.6, 29.2, 29.2, 29.3, 31.8, 33.2, 33.3, 34.5, 36.0, 37.3, 37.5, 37.6, 40.1, 40.3, 40.5, 44.6, 45.2, 46.3, 46.6, 46.8, 46.9, 47.3, 47.6, 50.3, 54.3, 58.7, 159.2, 174.8, 176.3, 178.1, 213.1; MS (ES⁺): m/z (%): 407.2 (100) (M+2H)²⁺; HRMS (ES⁺): m/z: calcd for C₄₂H₇₄N₁₀O₆ [M+2H]²⁺: 407.2891; found: 407.2887.

***N*¹-(3-(*N',N'*-bis-(3'(Guanidinopropyl)carbamoyl)propionyl)-*N*⁹-(3a,7a-dihydroxyl-5b-cholan-24-carbonyl)-norspermidine (2.31).**

Yield : (resin: 0.64 mmol/g, 39.8 mg) 30 mg, 73 %.

*t*_R = 7.7 min (RP-HPLC, 61 % pure); IR (Film): ν = 1661 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD, selected data) δ = 0.72 (s, 3H; 18'-CH₃), 0.95 (s, 3H; 19'-CH₃), 1.00 (d, ³*J*(HH) = 6 Hz, 3H; 21'-CH₃), 1.91 (m, 8H; CH₂CH₂NHCH₂CH₂ + CH₂CH₂NCH₂CH₂), 2.15, 2.29 (m, 2H; 23'-CH₂), 2.58 (m, 2H; NHCOCH₂CH₂CON), 2.72 (m, 2H; NHCOCH₂CH₂CON), 3.03 (m, 4H; CH₂NHCH₂), 3.19 (t, ³*J*(HH) = 7Hz, 2H; NHC(NH₂)NHCH₂), 3.25-3.35 (m, 6H;

NHC(NH₂)NHCH₂ + CONHCH₂ + CH₂NHCO), 3.40-3.51 (m, 4H; CH₂NCH₂), 3.57 (m, 1H; 3'-CH), 3.82 (m, 1H; 7'-CH); ¹³C NMR (100 MHz, CD₃OD) δ = 12.6 (18'-CH₃), 19.3 (21'-CH₃), 22.2 (19'-CH₃), 28.1, 28.3 (CH₂CH₂NHCH₂CH₂), 28.6 (NHC(NH₂)NHCH₂CH₂), 29.2 (NHCOCH₂CH₂CON), 29.3 (NHC(NH₂)NHCH₂CH₂), 31.7 (NHCOCH₂CH₂CON), 34.4 (23'-CH₂), 37.3 (CONHCH₂ + CH₂NHCO), 40.3, 40.5 (NHC(NH₂)NHCH₂), 44.6, 46.6 (CH₂NCH₂), 46.8, 46.9 (CH₂NHCH₂), 69.5 (7'-CH), 73.2 (3'-CH), 159.1, 159.2 (NHC(NH₂)NH), 174.8 (NHCOCH₂CH₂CON), 176.3 (NHCOCH₂CH₂CON), 178.3 (24'-CO); MS (ES⁺): m/z (%): 402.2 (100) (M+2H)²⁺; HRMS (ES⁺): m/z: calcd for C₄₂H₈₀N₁₀O₆ [M+2H]²⁺: 402.3151; found: 402.3154.

Compound Library 3.

*N*¹-(3(*N*',*N*'-bis-(3'(Pentanamidopropyl)carbamoyl)propionyl-*N*⁹-(carbamimidoyl)-norspermidine (3.1).

Yield : (resin: 0.64 mmol/g, 38.0 mg) 25 mg, quantitative yield.

*t*_R = 6.5 min (RP-HPLC, 83 % pure); IR (Film): ν = 1670, 1621 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.95 (t, ³J(HH) = 7 Hz, 6H; 2xCH₂CH₃), 1.36 (m, 4H; 2xCH₂CH₃), 1.60 (m, 4H; 2xCH₂CH₂CH₃), 1.72, 1.48 (m, 4H; CH₂CH₂NHCH₂CH₂), 1.89, 2.00 (m, 4H, CH₂CH₂NHCH₂CH₂), 2.21 (m, 4H; 2xCOCH₂C₃H₇), 2.52 (m, 2H; NHCOCH₂CH₂CON), 2.71 (m, 2H; NHCOCH₂CH₂CON), 3.07 (m, 4H; CH₂NHCH₂), 3.15 (t, ³J(HH) = 7 Hz, 2H; CH₂NHCOC₄H₉), 3.23 (t, ³J(HH) = 7 Hz, 2H; CH₂NHCOC₄H₉), 3.34 (m, 8H; 2xNHC(NH₂)NHCH₂ + CH₂NHCOCH₂CH₂CO + CH₂NCH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 14.5 (CH₂CH₃), 23.8 (CH₂CH₃), 27.2, 28.1 (CH₂CH₂NHCH₂CH₂), 29.1 (CH₂CH₂NHCOC₄H₉), 29.6 (CH₂CH₂CH₃ + NHCOCH₂CH₂CON), 30.0 (CH₂CH₂NHCOC₄H₉), 32.0 (NHCOCH₂CH₂CON), 37.0 (CH₂NHCOCH₂CH₂CO), 37.3 (NHCOCH₂C₃H₇), 38.2, 38.4 (CH₂NHCOC₄H₉), 39.9 (NHC(NH₂)NHCH₂), 45.0 (CH₂NCH₂), 46.6 (CH₂NHCH₂), 47.0 (CH₂NCH₂), 159.2 (NHC(NH₂)NH), 174.4 (NHCOCH₂CH₂CON), 176.7 (NHCOC₄H₉), 176.9 (NHCOCH₂CH₂CON); MS (ES⁺): m/z (%): 278.4 (100) (M+2H)²⁺, 555.6 (15) (M+H)⁺; HRMS (ES⁺): m/z: calcd for C₂₇H₅₅N₈O₄ [M+H]⁺: 555.4341; found: 555.4330.

***N*¹-(3(*N*′,*N*′-bis-(3′(3-Methylbutanamidopropyl)carbamoyl)propionyl-*N*⁹-
(carbamimidoyl)-norspermidine (3.2).**

Yield : (resin: 0.64 mmol/g, 37.7 mg) 26 mg, quantitative yield.

*t*_R = 6.3 min (RP-HPLC, 72 % pure); IR (Film): ν = 1670, 1621 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.96 (m, 12H; 2xCH(CH₃)₂), 1.73, 1.84 (m, 4H; CH₂CH₂NCH₂CH₂), 1.90, 2.00 (m, 4H; CH₂CH₂NHCH₂CH₂), 2.07 (m, 6H; 2xCOCH₂CH(CH₃)₂), 2.52 (m, 2H; NHCOCH₂CH₂CON), 2.71 (m, 2H; NHCOCH₂CH₂CON), 3.07 (m, 4H; CH₂NHCH₂), 3.16 (t, ³J(HH) = 7 Hz, 2H; CH₂NHCOC₄H₉), 3.23 (t, ³J(HH) = 7 Hz, 2H; CH₂NHCOC₄H₉), 3.35 (m, 8H; NHC(NH₂)NHCH₂ + CH₂NHCOCH₂CH₂CO + CH₂NCH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 22.1 (CH(CH₃)₂), 26.2 (NHC(NH₂)NHCH₂CH₂), 26.8 (CH(CH₃)₂), 27.1 (CH₂CH₂NHCOCH₂CH₂CO), 28.1 (CH₂CH₂NCH₂CH₂), 28.5 (NHCOCH₂CH₂CON), 29.0 (CH₂CH₂NCH₂CH₂), 30.9 (NHCOCH₂CH₂CON), 36.0 (CH₂NHCOCH₂CH₂CO), 37.1, 37.4 (CH₂NHC₄H₉), 38.9 (NHC(NH₂)NHCH₂), 44.1 (CH₂NCH₂), 45.6 (CH₂NHCH₂), 45.8 (NHCOCH₂C₃H₇), 46.1 (CH₂NCH₂), 158.2 (NHC(NH₂)NH), 173.3 (NHCOCH₂CH₂CON), 175.0 (NHCOC₄H₉), 175.9 (NHCOCH₂CH₂CON); MS (ES⁺): *m/z* (%): 278.4 (100) (M+2H)²⁺, 555.6 (15) (M+H)⁺; HRMS (ES⁺): *m/z*: calcd for C₂₇H₅₅N₈O₄ [M+H]⁺: 555.4341; found: 555.4331.

***N*¹-(3(*N*′,*N*′-bis-(3′(2,2-Dimethylpropionamidopropyl)carbamoyl)propionyl-*N*⁹-
(carbamimidoyl)-norspermidine (3.3).**

Yield : (resin: 0.64 mmol/g, 37.3 mg) 23 mg, quantitative yield.

*t*_R = 6.4 min (RP-HPLC, 70 % pure); IR (Film): ν = 1671, 1619 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 1.19 (m, 18H; 2xCH(CH₃)₃), 1.70 (m, 2H; CH₂CH₂NHCOC₄H₉), 1.87 (m, 4H; CH₂CH₂NHCOC₄H₉ + CH₂CH₂NHCH₂CH₂), 2.01 (m, 2H; CH₂CH₂NHCH₂CH₂), 2.52 (m, 2H; NHCOCH₂CH₂CON), 2.71 (m, 2H; NHCOCH₂CH₂CON), 3.07 (m, 4H; CH₂NHCH₂), 3.14 (t, ³J(HH) = 7 Hz, 2H; CH₂NHCOC₄H₉), 3.23 (t, ³J(HH) = 7 Hz, 2H; CH₂NHCOC₄H₉), 3.33 (m, 8H; NHC(NH₂)NHCH₂ + CH₂NHCOCH₂CH₂CON + CH₂NCH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 27.2, 28.1 (CH₂CH₂NHCH₂CH₂), 28.3 (C(CH₃)₃), 29.0 (CH₂CH₂NHCOC₄H₉), 29.5 (NHCOCH₂CH₂CON), 30.0 (CH₂CH₂NHCOC₄H₉), 32.0 (NHCOCH₂CH₂CON), 37.0 (CH₂NHCOCH₂CH₂CON), 38.3, 38.4 (2xCH₂NHCOC₄H₉), 39.9 (NHC(NH₂)NHCH₂), 44.6 (CH₂NCH₂), 46.6 (CH₂NHCH₂), 46.9 (CH₂NCH₂), 53.9

(COC(CH₃)₃), 159.2 (NHC(NH₂)NH), 174.5 (NHCOCH₂CH₂CON), 181.8 (NHCOC₄H₉), 182.0 (NHCOCH₂CH₂CON); MS (ES⁺): m/z (%): 278.4 (100) (M+2H)²⁺, 555.6 (12) (M+H)⁺; HRMS (ES⁺): m/z: calcd for C₂₇H₅₅N₈O₄ [M+H]⁺: 555.4341; found: 555.4328.

***N*¹-(3(*N*',*N*'-bis-(3'(Hexanamidopropyl)carbamoyl)propionyl-*N*⁹-(carbamimidoyl)-norspermidine (3.4).**

Yield : (resin: 0.64 mmol/g, 36.6 mg) 25 mg, quantitative yield.

*t*_R = 7.3 min (RP-HPLC, 90 % pure); IR (Film): ν = 1670, 1621 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.93 (t, ³*J*(HH) = 7 Hz, 6H; 2xCH₂CH₃), 1.34 (m, 8H; 2xCH₂CH₂CH₃), 1.62 (m, 4H; 2xCOCH₂CH₂C₃H₇), 1.72, 1.83 (m, 4H; CH₂CH₂NCH₂CH₂), 1.89, 2.00 (m, 4H; CH₂CH₂NHCH₂CH₂), 2.20 (m, 4H; 2xCOCH₂C₄H₉), 2.52 (m, 2H; NHCOCH₂CH₂CON), 2.71 (m, 2H; NHCOCH₂CH₂CON), 3.07 (m, 4H; CH₂NHCH₂), 3.15 (t, ³*J*(HH) = 7 Hz, 2H; CH₂NHCOC₅H₁₁), 3.23 (t, ³*J*(HH) = 7 Hz, 2H; CH₂NHCOC₅H₁₁), 3.34 (m, 8H; NHC(NH₂)NHCH₂ + CH₂NHCOCH₂CH₂CON + CH₂NCH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 14.7 (CH₂CH₃), 23.8 (CH₂CH₃), 27.2 (COCH₂CH₂C₃H₇ + NHC(NH₂)NHCH₂CH₂), 28.1 (CH₂CH₂NHCOCH₂CH₂CON), 29.1 (CH₂CH₂NHCOC₅H₁₁), 29.5 (NHCOCH₂CH₂CON), 30.0 (CH₂CH₂NHCOC₅H₁₁), 32.0 (NHCOCH₂CH₂CON), 33.0 (CH₂CH₂CH₃), 37.0 (CH₂NHCOCH₂CH₂CON), 37.5 (NHCOCH₂C₄H₉), 38.2, 38.4 (2xCH₂NHCOC₅H₁₁), 40.0 (NHC(NH₂)NHCH₂), 45.0 (CH₂NCH₂), 46.6 (CH₂NHCH₂), 47.0 (CH₂NCH₂), 159.2 (NHC(NH₂)NH), 174.4 (NHCOCH₂CH₂CON), 176.9 (NHCOCH₂CH₂CON + NHCOC₅H₁₁); MS (ES⁺): m/z (%): 292.4 (100) (M+2H)²⁺, 583.5 (20) (M+H)⁺; HRMS (ES⁺): m/z: calcd for C₂₉H₅₉N₈O₄ [M+H]⁺: 583.4654; found: 583.4653.

***N*¹-(3(*N*',*N*'-bis-(3'(2-Methylpentanamidopropyl)carbamoyl)propionyl-*N*⁹-(carbamimidoyl)-norspermidine (3.5).**

Yield : (resin: 0.64 mmol/g, 36.4 mg) 20 mg, 94 %.

*t*_R = 7.0 min (RP-HPLC, 77 % pure); IR (Film): ν = 1670, 1621 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.92 (m, 6H; 2xCH₂CH₃), 1.10 (m, 6H; 2xCHCH₃), 1.32, 1.59 (m, 8H; 2xCH₂CH₂CH₃), 1.72, 1.84 (m, 4H; CH₂CH₂NCH₂CH₂), 1.89, 2.00 (m, 4H; CH₂CH₂NHCH₂CH₂), 2.32 (m, 2H; COCH(CH₃)C₃H₇), 2.53 (m, 2H; NHCOCH₂CH₂CON), 2.71 (m, 2H; NHCOCH₂CH₂CON), 3.07 (m, 4H; CH₂NHCH₂), 3.14, 3.23 (m, 4H;

2xCH₂NHCOC₅H₁₁), 3.34 (m, 8H; NHC(NH₂)NHCH₂ + CH₂NHCOCH₂CH₂CON + CH₂NCH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 14.8 (CH₂CH₃), 18.8 (CHCH₃), 22.1 (CH₂CH₃), 27.2, 28.1 (CH₂CH₂NHCH₂CH₂), 29.1 (CH₂CH₂NCH₂CH₂), 29.5 (NHCOCH₂CH₂CON), 30.0 (CH₂CH₂NCH₂CH₂), 31.9 (NHCOCH₂CH₂CON), 37.0 (CH₂NHCOCH₂CH₂CON), 38.0 (CH₂NHCOC₅H₁₁), 38.1 (CH₂CH₂CH₃), 38.3 (CH₂NHCOC₅H₁₁), 39.9 (NHC(NH₂)NHCH₂), 42.4 (COCH(CH₃)C₃H₇), 45.0 (CH₂NCH₂), 46.6 (CH₂NHCH₂), 47.1 (CH₂NCH₂), 159.2 (NHC(NH₂)NH), 174.4 (NHCOCH₂CH₂CON), 180.1 (NHCOC₅H₁₁), 180.3 (NHCOCH₂CH₂CON); MS (ES⁺): m/z (%): 292.4 (100) (M+2H)²⁺, 583.5 (18) (M+H)⁺; HRMS (ES⁺): m/z: calcd for C₂₉H₅₉N₈O₄ [M+H]⁺: 583.4654; found: 583.4650.

***N*¹-(3(*N*',*N*'-bis-(3'(3-Methylpentanamidopropyl)carbamoyl)propionyl)-*N*⁹-(carbamimidoyl)-norspermidine (3.6).**

Yield : (resin: 0.64 mmol/g, 37.0 mg) 24 mg, quantitative yield.

*t*_R = 7.0 min (RP-HPLC, 82 % pure); IR (Film): ν = 1671, 1621 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.92 (m, 12H; 2xCH(CH₃)CH₂CH₃), 1.24, 1.38 (m, 4H; 2xCH₂CH₃), 1.72 (m, 2H; CH₂CH₂NHCH₂CH₂), 1.87 (m, 6H; CH₂CH₂NHCH₂CH₂ + CH(CH₃)C₂H₅ + CH₂CH₂NHCH₂CH₂), 2.00 (m, 4H; CH₂CH₂NHCH₂CH₂ + COCH₂C₄H₉), 2.21 (m, 2H; COCH₂C₄H₉), 2.52 (m, 2H; NHCOCH₂CH₂CON), 2.71 (m, 2H; NHCOCH₂CH₂CON), 3.07 (m, 4H; CH₂NHCH₂), 3.16 (t, ³J(HH) = 7 Hz, 2H; CH₂NHCOC₅H₁₁), 3.23 (t, ³J(HH) = 7 Hz, 2H; CH₂NHCOC₅H₁₁), 3.35 (m, 8H; CH₂NHCOCH₂CH₂CON + NHC(NH₂)NHCH₂ + CH₂NCH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 12.0 (CH₂CH₃), 19.9 (CHCH₃), 27.2, 28.1 (CH₂CH₂NHCH₂CH₂), 29.1 (CH₂CH₂NCH₂CH₂), 29.5 (NHCOCH₂CH₂CON), 30.0 (CH₂CH₂NCH₂CH₂), 30.8 (CH₂CH₃), 32.0 (NHCOCH₂CH₂CON), 34.1 (CH(CH₃)C₂H₅), 37.0 (CH₂NHCOCH₂CH₂CON), 38.2 38.4 (2xCH₂NHCOC₅H₁₁), 39.9 (NHC(NH₂)NHCH₂), 44.9 (COCH₂C₄H₉), 45.1 (CH₂NCH₂), 46.6 (CH₂NHCH₂), 47.1 (CH₂NCH₂), 159.2 (NHC(NH₂)NH), 174.4 (NHCOCH₂CH₂CON), 176.2 (NHCOC₅H₁₁), 176.8 (NHCOCH₂CH₂CON); MS (ES⁺): m/z (%): 292.3 (100) (M+2H)²⁺, 583.5 (18) (M+H)⁺; HRMS (ES⁺): m/z: calcd for C₂₉H₅₉N₈O₄ [M+H]⁺: 583.4654; found: 583.4652.

***N*¹-(3(*N*',*N*'-bis-(3'(4-Methylpentanamidopropyl)carbamoyl)propionyl)-*N*⁹-(carbamimidoyl)-norspermidine (3.7).**

Yield : (resin: 0.64 mmol/g, 35.8 mg) 19 mg, 88 %.

*t*_R = 7.2 min (RP-HPLC, 89 % pure); IR (Film): ν = 1670, 1621 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.93 (m, 12H; 2xCH(CH₃)₂), 1.51 (m, 4H; 2xCH₂CH(CH₃)₂), 1.53 (m, 2H; 2xCH(CH₃)₂), 1.72, 1.83 (m, 4H; CH₂CH₂NCH₂CH₂), 1.89, 2.00 (m, 4H; CH₂CH₂NHCH₂CH₂), 2.21 (m, 4H; 2xCOCH₂C₄H₉), 2.52 (m, 2H; NHCOCH₂CH₂CON), 2.71 (m, 2H; NHCOCH₂CH₂CON), 3.07 (m, 4H; CH₂NHCH₂), 3.14 (t, ³J(HH) = 7 Hz, 2H; CH₂NHCOC₅H₁₁), 3.22 (t, ³J(HH) = 7 Hz, 2H; CH₂NHCOC₅H₁₁), 3.34 (m, 8H; NHC(NH₂)NHCH₂ + CH₂NHCOCH₂CH₂CON + CH₂NCH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 23.1 (CH(CH₃)₂), 27.2, 28.1 (CH₂CH₂NHCH₂CH₂), 29.1 (CH₂CH₂NCH₂CH₂), 29.4 (CH(CH₃)₂), 29.5 (NHCOCH₂CH₂CON), 30.0 (CH₂CH₂NCH₂CH₂), 32.0 (NHCOCH₂CH₂CON), 35.6, 35.7 (2xCOCH₂C₄H₉), 36.4, 36.5 (2xCH₂CH(CH₃)₂), 37.0 (CH₂NHCOCH₂CH₂CON), 38.2, 38.5 (2xCH₂NHCOC₅H₁₁), 39.9 (NHC(NH₂)NHCH₂), 45.0 (CH₂NCH₂), 46.6 (CH₂NHCH₂), 47.0 (CH₂NCH₂), 159.2 (NHC(NH₂)NH), 174.0 (NHCOCH₂CH₂CON), 176.9 (NHCOC₅H₁₁ + NHCOCH₂CH₂CON); MS (ES⁺): m/z (%): 292.4 (100) (M+2H)²⁺, 583.6 (6) (M+H)⁺; HRMS (ES⁺): m/z: calcd for C₂₉H₅₉N₈O₄ [M+H]⁺: 583.4654; found: 583.4653.

***N*¹-(3(*N*',*N*'-bis-(3'(2-Ethylbutanamidopropyl)carbamoyl)propionyl)-*N*⁹-(carbamimidoyl)-norspermidine (3.8).**

Yield : (resin: 0.64 mmol/g, 36.5 mg) 19 mg, 87 %.

*t*_R = 6.8 min (RP-HPLC, 39 % pure); IR (Film): ν = 1671, 1621 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.92 (m, 12H; 2xCH(CH₂CH₃)₂), 1.52 (m, 8H; 2xCH(CH₂CH₃)₂), 1.73 (m, 2H; CH₂CH₂NCH₂CH₂), 1.88 (m, 4H; CH₂CH₂NCH₂CH₂ + CH₂CH₂NHCH₂CH₂), 2.01 (m, 4H; CH₂CH₂NHCH₂CH₂ + 2xCH(CH₂CH₃)₂), 2.52 (m, 2H; NHCOCH₂CH₂CON), 2.71 (m, 2H; NHCOCH₂CH₂CON), 3.07 (m, 4H; CH₂NHCH₂), 3.17 (t, ³J(HH) = 7 Hz, 2H; CH₂NHCOC₅H₁₁), 3.25 (t, ³J(HH) = 7 Hz, 2H; CH₂NHCOC₅H₁₁), 3.35 (m, 8H; NHC(NH₂)NHCH₂ + CH₂NHCOCH₂CH₂CON + CH₂NCH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 12.8 (CH(CH₂CH₃)₂), 27.2 (CH(CH₂CH₃)₂ + CH₂CH₂NHCH₂CH₂), 28.1 (CH₂CH₂NHCH₂CH₂), 29.2 (CH₂CH₂NCH₂CH₂), 29.6 (NHCOCH₂CH₂CON), 30.1

(CH₂CH₂NCH₂CH₂), 31.9 (NHCOCH₂CH₂CON), 37.0 (CH₂NHCOCH₂CH₂CON), 38.1, 38.3 (2xCH₂NHCOC₅H₁₁), 39.9 (NHC(NH₂)NHCH₂), 45.1 (CH₂NCH₂), 46.6 (CH₂NHCH₂), 47.2 (CH₂NHCH₂), 52.3 (CH(CH₂CH₃)₂), 159.2 (NHC(NH₂)NH), 174.4 (NHCOCH₂CH₂CON), 176.8 (NHCOC₅H₁₁), 179.2 (NHCOCH₂CH₂CON); MS (ES⁺): m/z (%): 292.4 (90) (M+2H)²⁺, 583.6 (28) (M+H)⁺; HRMS (ES⁺): m/z: calcd for C₂₉H₅₉N₈O₄ [M+H]⁺: 583.4654; found: 583.4651.

***N*¹-(3(*N*',*N*'-bis-(3'(3,3-Dimethylbutanamidopropyl)carbamoyl)propionyl-*N*⁹-(carbamimidoyl)-norspermidine (3.9).**

Yield : (resin: 0.64 mmol/g, 36.0 mg) 23 mg, quantitative yield.

*t*_R = 6.9 min (RP-HPLC, 70 % pure); IR (Film): ν = 1671, 1621 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 1.03 (br s, 18H; 2xC(CH₃)₃), 1.72, 1.84 (m, 4H; CH₂CH₂NCH₂CH₂), 1.89, 2.00 (m, 4H, CH₂CH₂NHCH₂CH₂), 2.08 (s, 2H; COCH₂C(CH₃)₃), 2.09 (s, 2H; COCH₂C(CH₃)₃), 2.52 (m, 2H; NHCOCH₂CH₂CON), 2.71 (m, 2H; NHCOCH₂CH₂CON), 3.07 (m, 4H; CH₂NHCH₂), 3.14 (t, ³J(HH) = 7 Hz, 2H; CH₂NHCOC₅H₁₁), 3.22 (t, ³J(HH) = 7 Hz, 2H; CH₂NHCOC₅H₁₁), 3.38 (m, 8H; NHC(NH₂)NHCH₂ + CH₂NHCOCH₂CH₂CON + CH₂NCH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 26.1, 27.0 (CH₂CH₂NHCH₂CH₂), 28.0 (CH₂CH₂NCH₂CH₂), 28.5 (NHCOCH₂CH₂CON), 29.0 (CH₂CH₂NCH₂CH₂), 29.6 (C(CH₃)₃), 30.9 (C(CH₃)₃), 31.0 (NHCOCH₂CH₂CON), 36.0 (CH₂NHCOCH₂CH₂CON), 37.1, 37.3 (2xCH₂NHCOC₅H₁₁), 38.9 (NHC(NH₂)NHCH₂), 44.0 (CH₂NCH₂), 45.5 (CH₂NHCH₂), 46.1 (CH₂NCH₂), 50.0, 50.1 (2xCOCH₂C(CH₃)₃), 158.1 (NHC(NH₂)NH), 174.2 (NHCOCH₂CH₂CON), 176.5 (NHCOC₅H₁₁), 177.3 (NHCOCH₂CH₂CON); MS (ES⁺): m/z (%): 292.4 (100) (M+2H)²⁺, 583.6 (37) (M+H)⁺; HRMS (ES⁺): m/z: calcd for C₂₉H₅₉N₈O₄ [M+H]⁺: 583.4654; found: 583.4652.

***N*¹-(3(*N*',*N*'-bis-(3'(Hepatanamidopropyl)carbamoyl)propionyl-*N*⁹-(carbamimidoyl)-norspermidine (3.10).**

Yield : (resin: 0.64 mmol/g, 37.0 mg) 20 mg, 90 %.

*t*_R = 7.9 min (RP-HPLC, 73 % pure); IR (Film): ν = 1670, 1621 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.69 (m, 6H; 2xCH₂CH₃), 1.10 (br s, 12H; 2xCH₂(CH₂)₃CH₃), 1.37 (m, 4H; 2xCH₂(CH₂)₃CH₃), 1.49, 1.61 (m, 4H; CH₂CH₂NCH₂CH₂), 1.67, 1.74 (m, 4H;

$\text{CH}_2\text{CH}_2\text{NHCH}_2\text{CH}_2$), 1.97 (m, 4H; $\text{COCH}_2\text{C}_5\text{H}_{11}$), 2.30 (m, 2H; $\text{NHCOCH}_2\text{CH}_2\text{CON}$), 2.48 (m, 2H; $\text{NHCOCH}_2\text{CH}_2\text{CON}$), 2.84 (m, 4H; CH_2NHCH_2), 2.92, 3.00 (m, 4H; $2\times\text{CH}_2\text{NHCOC}_6\text{H}_{13}$), 3.12 (m, 8H; $\text{NHC}(\text{NH}_2)\text{NHCH}_2 + \text{CH}_2\text{NHCOCH}_2\text{CH}_2\text{CON} + \text{CH}_2\text{NCH}_2$); ^{13}C NMR (100 MHz, CD_3OD) $\delta = 14.8$ (CH_2CH_3), 24.0 (CH_2CH_3), 27.2 ($\text{CH}_2\text{CH}_2\text{NHCH}_2\text{CH}_2$), 27.4 ($\text{COCH}_2\text{CH}_2\text{C}_4\text{H}_9$), 28.1 ($\text{CH}_2\text{CH}_2\text{NHCH}_2\text{CH}_2$), 29.1 ($\text{CH}_2\text{CH}_2\text{NCH}_2\text{CH}_2$), 29.5 ($\text{NHCOCH}_2\text{CH}_2\text{CON}$), 30.0 ($\text{CH}_2\text{CH}_2\text{NCH}_2\text{CH}_2$), 30.4 ($\text{CH}_2\text{C}_3\text{H}_7$), 32.0 ($\text{NHCOCH}_2\text{CH}_2\text{CON}$), 33.1 ($\text{CH}_2\text{CH}_2\text{CH}_3$), 37.0 ($\text{CH}_2\text{NHCOC}_6\text{H}_{13}$), 37.6 ($\text{COCH}_2\text{C}_5\text{H}_{11}$), 38.2, 38.4 ($2\times\text{CH}_2\text{NHCOC}_6\text{H}_{13}$), 39.9 ($\text{NHC}(\text{NH}_2)\text{NHCH}_2$), 45.0 (CH_2NCH_2), 46.6 (CH_2NHCH_2), 47.0 (CH_2NCH_2), 159.2 ($\text{NHC}(\text{NH}_2)\text{NH}$), 174.4 ($\text{NHCOCH}_2\text{CH}_2\text{CON}$), 176.7 ($\text{NHCOC}_6\text{H}_{13} + \text{NHCOCH}_2\text{CH}_2\text{CON}$); MS (ES^+): m/z (%): 306.4 (100) ($\text{M}+2\text{H}$) $^{2+}$, 611.6 (21) ($\text{M}+\text{H}$) $^+$; HRMS (ES^+): m/z: calcd for $\text{C}_{31}\text{H}_{63}\text{N}_8\text{O}_4$ [$\text{M}+\text{H}$] $^+$: 611.4967; found: 611.4951.

***N*¹-(3(*N*′, *N*′-bis-(3′(2-Methylhexanamidopropyl)carbamoyl)propionyl)-*N*⁹-(carbamimidoyl)-norspermidine (3.11).**

Yield : (resin: 0.64 mmol/g, 38.3 mg) 24 mg, quantitative yield.

$t_R = 7.7$ min (RP-HPLC, 91 % pure); IR (Film): $\nu = 1655, 1619$ cm^{-1} (C=O); ^1H NMR (400 MHz, CD_3OD) $\delta = 0.91$ (t, $^3J(\text{HH}) = 7$ Hz, 6H; $2\times\text{CH}_2\text{CH}_3$), 1.12 (m, 6H; $2\times\text{CHCH}_3$), 1.33, 1.60 (m, 12H; $\text{CH}(\text{CH}_3)(\text{CH}_2)_3\text{CH}_3$), 1.72, 1.84 (m, 4H; $2\times\text{CH}_2\text{CH}_2\text{NCH}_2\text{CH}_2$), 1.89, 2.00 (m, 4H; $\text{CH}_2\text{CH}_2\text{NHCH}_2\text{CH}_2$), 2.30 (m, 2H; $2\times\text{CHCH}_3$), 2.52 (m, 2H; $\text{NHCOCH}_2\text{CH}_2\text{CON}$), 2.71 (m, 2H; $\text{NHCOCH}_2\text{CH}_2\text{CON}$), 3.07 (m, 4H; CH_2NHCH_2), 3.15 (t, $^3J(\text{HH}) = 7$ Hz, 2H; $\text{CH}_2\text{NHCOC}_6\text{H}_{13}$), 3.23 (t, $^3J(\text{HH}) = 7$ Hz, 2H; $\text{CH}_2\text{NHCOC}_6\text{H}_{13}$), 3.34 (m, 8H; $\text{NHC}(\text{NH}_2)\text{NHCH}_2 + \text{CH}_2\text{NHCOCH}_2\text{CH}_2\text{CON} + \text{CH}_2\text{NCH}_2$); ^{13}C NMR (100 MHz, CD_3OD) $\delta = 14.7$ (CH_2CH_3), 18.8 (CHCH_3), 24.1 (CH_2CH_3), 27.2, 28.1 ($\text{CH}_2\text{CH}_2\text{NHCH}_2\text{CH}_2$), 29.1 ($\text{CH}_2\text{CH}_2\text{NCH}_2\text{CH}_2$), 29.5 ($\text{NHCOCH}_2\text{CH}_2\text{CON}$), 30.1 ($\text{CH}_2\text{CH}_2\text{NCH}_2\text{CH}_2$), 31.2 ($\text{CH}_2\text{CH}_2\text{CH}_3$), 31.9 ($\text{NHCOCH}_2\text{CH}_2\text{CON}$), 35.4 ($\text{CH}_2\text{C}_3\text{H}_7$), 37.0 ($\text{CH}_2\text{NHCOC}_6\text{H}_{13}$), 38.1, 38.3 ($2\times\text{CH}_2\text{NHCOC}_6\text{H}_{13}$), 39.9 ($\text{NHC}(\text{NH}_2)\text{NHCH}_2$), 42.6 ($\text{COCH}(\text{CH}_3)\text{C}_4\text{H}_9$), 45.0 (CH_2NCH_2), 46.6 (CH_2NHCH_2), 47.1 (CH_2NCH_2), 159.2 ($\text{NHC}(\text{NH}_2)\text{NH}$), 174.4 ($\text{NHCOCH}_2\text{CH}_2\text{CON}$), 176.8 ($\text{NHCOCH}_2\text{CH}_2\text{CON}$), 180.1 ($\text{NHCOC}_6\text{H}_{13}$); MS (ES^+): m/z (%): 306.5 (100) ($\text{M}+2\text{H}$) $^{2+}$, 611.8 (10) ($\text{M}+\text{H}$) $^+$; HRMS (ES^+): m/z: calcd for $\text{C}_{31}\text{H}_{63}\text{N}_8\text{O}_4$ [$\text{M}+\text{H}$] $^+$: 611.4967; found: 611.4957.

***N*¹-(3(*N*',*N*'-bis-(3'(5-Methylhexanamidopropyl)carbamoyl)propionyl-*N*⁹-(carbamimidoyl)-norspermidine (3.12).**

Yield : (resin: 0.64 mmol/g, 37.8 mg) 24 mg, quantitative yield.

*t*_R = 7.8 min (RP-HPLC, 87 % pure); IR (Film): ν = 1670, 1622 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.91 (d, ³*J*(HH) = 7 Hz, 12H; 2xCH(CH₃)₂), 1.22 (m, 4H; 2xCH₂CH(CH₃)₂), 1.62 (m, 6H; 2xCH₂CH₂CH(CH₃)₂), 1.72, 1.84 (m, 4H; CH₂CH₂NCH₂CH₂), 1.89, 2.00 (m, 4H; CH₂CH₂NHCH₂CH₂), 2.19 (m, 4H; 2xCOCH₂C₅H₁₁), 2.52 (m, 2H; NHCOCH₂CH₂CON), 2.71 (m, 2H; NHCOCH₂CH₂CON), 3.07 (m, 4H; CH₂NHCH₂), 3.15 (t, ³*J*(HH) = 7 Hz, 2H; CH₂NHCOC₆H₁₃), 3.22 (t, ³*J*(HH) = 7 Hz, 2H; CH₂NHCOC₆H₁₃), 3.34 (m, 8H; NHC(NH₂)NHCH₂ + CH₂NHCOCH₂CH₂CON + CH₂NCH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 23.3 (CH(CH₃)₂), 25.3 (CH₂CH₂CH(CH₃)₂), 27.2, 28.1 (CH₂CH₂NHCH₂CH₂), 29.3 (CH₂CH₂NCH₂CH₂), 29.4 (NHCOCH₂CH₂CON), 29.5 (CH(CH₃)₂), 30.0 (CH₂CH₂NCH₂CH₂), 32.0 (NHCOCH₂CH₂CON), 37.0 (CH₂NHCOCH₂CH₂CON), 37.8 (NHCOCH₂C₅H₁₁), 38.2, 38.5 (2xCH₂NHCOC₆H₁₃), 39.9 (NHC(NH₂)NHCH₂), 40.0 (CH₂CH(CH₃)₂), 45.0 (CH₂NCH₂), 46.6 (CH₂NHCH₂), 47.0 (CH₂NCH₂), 159.2 (NHC(NH₂)NH), 174.4 (NHCOCH₂CH₂CON), 176.7 (NHCOC₆H₁₃), 176.9 (NHCOCH₂CH₂CON); MS (ES⁺): *m/z* (%): 306.5 (100) (M+2H)²⁺, 611.8 (10) (M+H)⁺; HRMS (ES⁺): *m/z*: calcd for C₃₁H₆₃N₈O₄ [M+H]⁺: 611.4967; found: 611.4977.

***N*¹-(3(*N*',*N*'-bis-(3'(Octanamidopropyl)carbamoyl)propionyl-*N*⁹-(carbamimidoyl)-norspermidine (3.13).**

Yield : (resin: 0.64 mmol/g, 36.5 mg) 16 mg, 71 %.

*t*_R = 8.6 min (RP-HPLC, 87 % pure); IR (Film): ν = 1671, 1622 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.91 (t, ³*J*(HH) = 7 Hz, 6H; 2xCH₂CH₃), 1.33 (br s, 16H; 2xCH₂(CH₂)₄CH₃), 1.61 (m, 4H; 2xCH₂(CH₂)₄CH₃), 1.72, 1.84 (m, 4H; CH₂CH₂NCH₂CH₂), 1.90, 2.00 (m, 4H; CH₂CH₂NHCH₂CH₂), 2.20 (m, 4H; 2xCOCH₂C₆H₁₃), 2.52 (m, 2H; NHCOCH₂CH₂CON), 2.71 (m, 2H; NHCOCH₂CH₂CON), 3.07 (m, 4H; CH₂NHCH₂), 3.15 (t, ³*J*(HH) = 7 Hz, 2H; CH₂NHCOC₇H₁₅), 3.23 (t, ³*J*(HH) = 7 Hz, 2H; CH₂NHCOC₇H₁₅), 3.34 (m, 8H; NHC(NH₂)NHCH₂ + CH₂NHCOCH₂CH₂CON + CH₂NCH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 16.8 (CH₂CH₃), 26.0 (CH₂CH₃), 29.2 (CH₂CH₂NHCH₂CH₂), 29.5 (COCH₂CH₂C₅H₁₁), 30.1 (CH₂CH₂NHCH₂CH₂), 31.1 (CH₂CH₂NCH₂CH₂), 31.5

(NHCOCH₂CH₂CON), 32.0 (CH₂CH₂NCH₂CH₂), 32.5 (CH₂C₃H₇), 32.7 (CH₂C₄H₉), 34.0 (NHCOCH₂CH₂CON), 35.3 (CH₂CH₂CH₃), 39.0 (CH₂NHCOCH₂CH₂CON), 39.6 (COCH₂C₆H₁₃), 40.2, 40.5 (2xCH₂NHCOC₇H₁₅), 41.9 (NHC(NH₂)NHCH₂), 47.0 (CH₂NCH₂), 48.6 (CH₂NHCH₂), 49.0 (CH₂NCH₂), 161.2 (NHC(NH₂)NH), 176.4 (NHCOCH₂CH₂CON), 178.7 (NHCOC₇H₁₅), 178.9 (NHCOCH₂CH₂CON); MS (ES⁺): m/z (%): 320.5 (100) (M+2H)²⁺, 639.7 (10) (M+H)⁺; HRMS (ES⁺): m/z: calcd for C₃₃H₆₇N₈O₄ [M+H]⁺: 639.5280; found: 639.5287.

***N*¹-(3(*N*′, *N*′-bis-(3′(2-Methylheptanamidopropyl)carbamoyl)propionyl-*N*⁹-(carbamimidoyl)-norspermidine (3.14).**

Yield : (resin: 0.64 mmol/g, 37.5 mg) 18 mg, 75 %.

*t*_R = 8.3 min (RP-HPLC, 77 % pure); IR (Film): ν = 1671, 1622 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.80 (m, 6H; 2xCH₂CH₃), 1.00 (m, 6H; 2xCHCH₃), 1.20, 1.49 (m, 16H; 2xCH(CH₃)(CH₂)₄CH₃), 1.61, 1.72 (m, 4H; CH₂CH₂NCH₂CH₂), 1.78, 1.89 (CH₂CH₂NHCH₂CH₂), 2.19 (m, 2H; 2xCH(CH₃), 2.41 (m, 2H; NHCOCH₂CH₂CON), 2.60 (m, 2H; NHCOCH₂CH₂CON), 2.96 (m, 4H; CH₂NHCH₂), 3.04 (t, ³J(HH) = 7 Hz, 2H; CH₂NHCOC₇H₁₅), 3.12 (t, ³J(HH) = 7 Hz, 2H; CH₂NHCOC₇H₁₅), 3.24 (m, 8H; NHC(NH₂)NHCH₂ + CH₂NHCOCH₂CH₂CON + CH₂NCH₂); ¹³C NMR (100 MHz, CD₃OD), δ = 14.8 (CH₂CH₃), 18.9 (CHCH₃), 24.0 (CH₂CH₃), 27.2, 28.1 (CH₂CH₂NHCH₂CH₂), 28.7 (CH₂CH₂CH₃), 29.1 (CH₂CH₂NCH₂CH₂), 29.5 (NHCOCH₂CH₂CON), 30.0 (CH₂CH₂NCH₂CH₂), 31.9 (NHCOCH₂CH₂CON), 33.3 (CH₂C₃H₇), 35.7 (CH₂C₄H₉), 37.0 (CH₂NHCOCH₂CH₂CON), 38.1, 38.3 (2xCH₂NHCOC₇H₁₅), 39.9 (NHC(NH₂)NHCH₂), 42.6 (CHCH₃), 45.0 (CH₂NCH₂), 46.6 (CH₂NHCH₂), 47.1 (CH₂NCH₂), 159.2 (NHC(NH₂)NH), 174.3 (NHCOCH₂CH₂CON), 176.8 (NHCOCH₂CH₂CON), 180.1 (NHCOC₇H₁₅); MS (ES⁺): m/z (%): 320.5 (100) (M+2H)²⁺, 639.7 (14) (M+H)⁺; HRMS (ES⁺): m/z: calcd for C₃₃H₆₇N₈O₄ [M+H]⁺: 639.5280; found: 639.5284.

***N*¹-(3(*N*',*N*'-bis-(3'(2-Ethylhexanamidopropyl)carbamoyl)propionyl-*N*⁹-(carbamimidoyl)-norspermidine (3.15).**

Yield : (resin: 0.64 mmol/g, 36.7 mg) 22 mg, 97 %.

$t_R = 8.3$ min (RP-HPLC, 77 % pure); IR (Film): $\nu = 1671, 1621$ cm^{-1} (C=O); ¹H NMR (400 MHz, CD₃OD) $\delta = 0.79$ (m, 8H; 2xCHCH₂CH₃ + 2xCH₂CH₂CH₃), 1.18 (m, 8H; 2xCH₂CH₂CH₃), 1.33, 1.45 (m, 8H; 2xCH₂C₃H₇ + 2xCHCH₂CH₃), 1.62, 1.74 (m, 4H; CH₂CH₂NCH₂CH₂), 1.78, 1.89 (m, 4H; CH₂CH₂NHCH₂CH₂), 1.99 (m, 2H; 2xCOCHCH₂CH₃), 2.41 (m, 2H; NHCOCH₂CH₂CON), 2.61 (m, 2H; NHCOCH₂CH₂CON), 2.96 (m, 4H; CH₂NHCH₂), 3.06, 3.14 (m, 4H; 2xCH₂NHCOC₇H₁₅), 3.24 (m, 8H; NHC(NH₂)NHCH₂ + CH₂NHCOCH₂CH₂CON + CH₂NCH₂); ¹³C NMR (100 MHz, CD₃OD) $\delta = 12.8$ (CH₂CH₂CH₃), 14.7 (CHCH₂CH₃), 24.1 (CH₂CH₂CH₃), 27.2 (CH₂CH₂NHCH₂CH₂), 27.5 (CHCH₂CH₃), 28.1 (CH₂CH₂NHCH₂CH₂), 29.2 (CH₂CH₂NCH₂CH₂), 29.6 (NHCOCH₂CH₂CON), 30.1 (CH₂CH₂NCH₂CH₂), 31.4 (CH₂CH₂CH₃), 32.0 (NHCOCH₂CH₂CON), 34.0 (CH₂C₃H₇), 37.1 (CH₂NHCOCH₂CH₂CON), 38.1, 38.4 (2xCH₂NHCOC₇H₁₅), 39.9 (NHC(NH₂)NHCH₂), 45.1 (CH₂NCH₂), 46.6 (CH₂NHCH₂), 47.2 (CH₂NCH₂), 50.5 (COCHCH₂CH₃), 159.2 (NHC((NH₂)NH), 174.4 (NHCOCH₂CH₂CON), 176.8 (NHCOCH₂CH₂CON), 179.2 (NHCOC₇H₁₅); MS (ES⁺): m/z (%): 320.5 (100) (M+2H)²⁺, 639.6 (12) (M+H)⁺; HRMS (ES⁺): m/z: calcd for C₃₃H₆₇N₈O₄ [M+H]⁺: 639.5280; found: 639.5294.

***N*¹-(3(*N*',*N*'-bis-(3'(3-Propylhexanamidopropyl)carbamoyl)propionyl-*N*⁹-(carbamimidoyl)-norspermidine (3.16).**

Yield : (resin: 0.64 mmol/g, 37.9 mg) 21 mg, 87 %.

$t_R = 8.1$ min (RP-HPLC, 87 % pure); IR (Film): $\nu = 1671, 1619$ cm^{-1} (C=O); ¹H NMR (400 MHz, CD₃OD) $\delta = 0.92$ (m, 12; 2xCH(CH₂CH₂CH₃)₂), 1.30 (m, 8H; 2xCH(CH₂CH₂CH₃)₂), 1.38, 1.55 (m, 8H; 2xCH(CH₂CH₂CH₃)₂), 1.72, 1.84 (m, 4H; CH₂CH₂NCH₂CH₂), 1.89, 2.00 (m, 2H; CH₂CH₂NHCH₂CH₂), 2.22 (m, 2H; 2xCH(C₃H₇)₂), 2.52 (m, 2H; NHCOCH₂CH₂CON), 2.71 (m, 2H; NHCOCH₂CH₂CON), 3.07 (m, 4H; CH₂NHCH₂), 3.16 (t, ³J(HH) = 7 Hz, CH₂NHCOC₇H₁₅), 3.24 (t, ³J(HH) = 7 Hz, CH₂NHCOC₇H₁₅), 3.35 (m, 8H; NHC(NH₂)NHCH₂ + CH₂NHCOCH₂CH₂CON + CH₂NCH₂); ¹³C NMR (100 MHz, CD₃OD) $\delta = 14.8$ (CH(CH₂CH₂CH₃)₂), 22.2 (CH(CH₂CH₂CH₃)₂), 27.2, 28.1

(CH₂CH₂NHCH₂CH₂), 29.2 (CH₂CH₂NCH₂CH₂), 29.5 (NHCOCH₂CH₂CON), 30.1 (CH₂CH₂NCH₂CH₂), 31.9 (NHCOCH₂CH₂CON), 36.8 (CH(CH₂CH₂CH₃)₂), 37.0 (CH₂NHCOCH₂CH₂CON), 38.1, 38.4 (2xCH₂NHCOC₇H₁₅), 39.9 (NHC(NH₂)NHCH₂), 45.1 (CH₂NCH₂), 46.3 (CH₂NHCH₂), 47.2 (CH₂NCH₂), 48.3 (CO(CH(C₃H₇)₂), 159.2 (NHC(NH₂)NH), 174.4 (NHCOCH₂CH₂CON), 176.8 (NHCOCH₂CH₂CON), 179.3, 179.6 (NHCOC₇H₁₅); MS (ES⁺): m/z (%): 320.4 (100) (M+2H)²⁺, 639.7 (30) (M+H)⁺; HRMS (ES⁺): m/z: calcd for C₃₃H₆₇N₈O₄ [M+H]⁺: 639.5280; found: 639.5283.

***N*¹-(3(*N*',*N*'-bis-(3'(Nonanamidopropyl)carbamoyl)propionyl-*N*⁹-(carbamimidoyl)-norspermidine (3.17).**

Yield : (resin: 0.64 mmol/g, 50.0 mg) 18 mg, 55 %.

*t*_R = 9.2 min (RP-HPLC, 64 % pure); IR (Film): ν = 1666, 1628 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.80 (m, 6H; 2xCH₂CH₃), 1.20 (br s, 20H; 2xCH₂(CH₂)₅CH₃), 1.50 (br s, 4H; 2xCH₂C₆H₁₃), 1.61 (m, 4H; CH₂CH₂NCH₂CH₂), 1.79 (m, 4H, CH₂CH₂NHCH₂CH₂), 2.80 (m, 4H; 2xCOCH₂C₇H₁₅), 2.41 (m, 2H; NHCOCH₂CH₂CON), 2.60 (m, 2H; NHCOCH₂CH₂CON), 2.95 (m, 4H; CH₂NHCH₂), 3.04, 3.11 (m, 4H; 2xCH₂NHCOC₈H₁₇), 3.24 (m, 8H; NHC(NH₂)NHCH₂ + CH₂NHCOCH₂CH₂CON + CH₂NCH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 14.8 (CH₂CH₃), 24.1 (CH₂CH₃), 27.2 (CH₂CH₂NHCH₂CH₂), 27.5 (CH₂C₆H₁₃), 28.5 (CH₂CH₂NHCH₂CH₂), 29.1 (CH₂CH₂NCH₂CH₂), 29.6 (NHCOCH₂CH₂CON), 30.0 (CH₂CH₂NCH₂CH₂), 30.7, 30.8 (CH₂(CH₂)₃C₃H₇), 32.0 (NHCOCH₂CH₂CON), 33.4 (CH₂CH₂CH₃), 37.0 (CH₂NHCOCH₂CH₂CON), 37.3 (COCH₂C₇H₁₅), 38.2, 38.4 (2xCH₂NHCOC₈H₁₇), 39.9 (NHC(NH₂)NHCH₂), 45.0 (CH₂NCH₂), 46.6 (CH₂NHCH₂), 47.0 (CH₂NCH₂), 159.2 (NHC(NH₂)NH), 174.2 (NHCOCH₂CH₂CON), 176.3 (NHCOC₈H₁₇), 176.8 (NHCOCH₂CH₂CON); MS (ES⁺): m/z (%): 334.5 (100) (M+2H)²⁺, 667.6 (12) (M+H)⁺; HRMS (ES⁺): m/z: calcd for C₃₅H₇₁N₈O₄ [M+H]⁺: 667.5593; found: 667.5603.

***N*¹-(3(*N*',*N*'-bis-(3'(4-Methyloctanamidopropyl)carbamoyl)propionyl-*N*⁹-(carbamimidoyl)-norspermidine (3.18).**

Yield : (resin: 0.64 mmol/g, 36.4 mg) 13 mg, 56 %.

*t*_R = 9.0 min (RP-HPLC, 73 % pure); IR (Film): ν = 1671, 1621 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.92 (m, 12H; 2xCH₂CH₃ + 2xCHCH₃), 1.17, 1.32 (m, 12H; 2xCH(CH₃)(CH₂)₃CH₃), 1.43, 1.65 (m, 6H; 2xCH₂CH(CH₃), 1.72, 1.83 (m, 4H; CH₂CH₂NCH₂CH₂), 1.89, 2.00 (m, 4H; CH₂CH₂NHCH₂CH₂), 2.21 (m, 4H; 2xCOCH₂C₇H₁₅), 2.52 (m, 2H; NHCOCH₂CH₂CON), 2.71 (m, 2H; NHCOCH₂CH₂CON), 3.07 (m, 4H; CH₂NHCH₂), 3.15 (t, ³J(HH) = 7 Hz, 2H; CH₂NHCOC₈H₁₇), 3.22 (t, ³J(HH) = 7 Hz, 2H; CH₂NHCOC₈H₁₇), 3.35 (m, 8H; NHC(NH₂)NHCH₂ + CH₂NHCOCH₂CH₂CON + CH₂NCH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 14.8 (CH₂CH₃), 20.1 (CHCH₃), 23.4 (CH₂CH₃), 27.2, 28.1 (CH₂CH₂NHCH₂CH₂), 29.1 (CH₂CH₂NCH₂CH₂), 29.5 (NHCOCH₂CH₂CON), 30.0 (CH₂CH₂NCH₂CH₂), 30.7 (CH₂CH₂CH₃), 32.0 (NHCOCH₂CH₂CON), 34.2 (CH₂C₆H₁₃), 34.6 (CHCH₃), 35.4 (COCH₂C₇H₁₅), 37.0 (CH₂NHCOCH₂CH₂CON), 38.0 (CH₂C₃H₇), 38.2, 38.5 (2xCH₂NHCOC₈H₁₇), 39.9 (NHC(NH₂)NHCH₂), 45.0 (CH₂NCH₂), 46.6 (CH₂NHCH₂), 47.0 (CH₂NCH₂), 159.2 (NHC(NH₂)NH), 174.4 (NHCOCH₂CH₂CON), 176.9 (NHCOCH₂CH₂CON + NHCOC₈H₁₇); MS (ES⁺): m/z (%): 334.6 (100) (M+2H)²⁺, 667.9 (20) (M+H)⁺; HRMS (ES⁺): m/z: calcd for C₃₅H₇₁N₈O₄ [M+H]⁺: 667.5593; found: 667.5599.

***N*¹-(3(*N*',*N*'-bis-(3'(Decanamidopropyl)carbamoyl)propionyl-*N*⁹-(carbamimidoyl)-norspermidine (3.19).**

Yield : (resin: 0.64 mmol/g, 35.0 mg) 15 mg, 63 %.

*t*_R = 9.9 min (RP-HPLC, 85 % pure); IR (Film): ν = 1665, 1621 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.91 (t, ³J(HH) = 7 Hz, 6H; 2xCH₂CH₃), 1.31 (br s, 24H; 2xCH₂(CH₂)₆CH₃), 1.61 (br s, 4H; 2xCH₂C₇H₁₅), 1.72, 1.84 (m, 4H; CH₂CH₂NCH₂CH₂), 1.90, 2.00 (m, 4H; CH₂CH₂NHCH₂CH₂), 2.20 (m, 4H; 2xCOCH₂C₈H₁₇), 2.52 (m, 2H; NHCOCH₂CH₂CON), 2.71 (m, 2H; NHCOCH₂CH₂CON), 3.07 (m, 4H; CH₂NHCH₂), 3.15 (t, ³J(HH) = 7 Hz, 2H; CH₂NHCOC₉H₁₉), 3.23 (t, ³J(HH) = 7 Hz, 2H; CH₂NHCOC₉H₁₉), 3.35 (m, 8H; NHC(NH₂)NHCH₂ + CH₂NHCOCH₂CH₂CON + CH₂NCH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 14.8 (CH₂CH₃), 24.1 (CH₂CH₃), 27.2 (CH₂CH₂NHCH₂CH₂), 27.5

(CH₂C₇H₁₅), 28.1 (CH₂CH₂NHCH₂CH₂), 29.1 (CH₂CH₂NCH₂CH₂), 29.5 (NHCOCH₂CH₂CON), 30.0 (CH₂CH₂NCH₂CH₂), 30.8, 30.9, 31.0, (CH₂(CH₂)₄C₃H₇), 32.0 (NHCOCH₂CH₂CON), 33.4 (CH₂CH₂CH₃), 37.0 (CH₂NHCOCH₂CH₂CON), 37.6 (COCH₂C₈H₁₇), 38.2, 38.5 (2xCH₂NHCOC₉H₁₉), 39.9 (NHC(NH₂)NHCH₂), 45.0 (CH₂NCH₂), 46.6 (CH₂NHCH₂), 47.0 (CH₂NCH₂); 159.2 (NHC(NH₂)NH), 173.2 (NHCOCH₂CH₂CON), 176.8 (NHCOCH₂CH₂CON + NHCOC₉H₁₉); MS (ES⁺): m/z (%): 348.6 (100) (M+2H)²⁺, 695.8 (4) (M+H)⁺; HRMS (ES⁺): m/z: calcd for C₃₇H₇₅N₈O₄ [M+H]⁺: 695.5906; found: 695.5924.

***N*¹-(3(*N',N'*-bis-(3'(4-Methylnonanamidopropyl)carbamoyl)propionyl)-*N*⁹-(carbamimidoyl)-norspermidine (3.20).**

Yield : (resin: 0.64 mmol/g, 38.0 mg) 15 mg, 60 %.

*t*_R = 9.7 min (RP-HPLC, 90 % pure); IR (Film): ν = 1675, 1629 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.81 (m, 12H; 2xCHCH₃ + 2xCH₂CH₃), 1.05, 1.21 (m, 16H; 2xCH(CH₃)(CH₂)₄CH₃), 1.31, 1.54 (m, 6H; 2xCH₂CH(CH₃)), 1.61, 1.72 (m, 4H; CH₂CH₂NCH₂CH₂), 1.78, 1.89 (m, 4H; CH₂CH₂NHCH₂CH₂), 2.10 (m, 4H; NHCOCH₂C₈H₁₇), 2.40 (m, 2H, NHCOCH₂CH₂CON), 2.60 (m, 2H; NHCOCH₂CH₂CON), 2.95 (m, 4H; CH₂NHCH₂), 3.03 (t, ³J(HH) = 7 Hz, 2H; CH₂NHCOC₉H₁₉), 3.11 (t, ³J(HH) = 7 Hz, 2H; CH₂NHCOC₉H₁₉), 3.23 (m, 8H; NHC(NH₂)NHCH₂ + CH₂NHCOCH₂CH₂CON + CH₂NCH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 14.8 (CH₂CH₃), 20.1 (CHCH₃), 24.1 (CH₂CH₃), 27.2 (CH₂CH₂NHCH₂CH₂), 28.1 (CH₂C₃H₇), 28.2 (CH₂CH₂NHCH₂CH₂), 29.1 (CH₂CH₂NCH₂CH₂), 29.5 (NHCOCH₂CH₂CON), 30.0 (CH₂CH₂NCH₂CH₂), 31.9 (NHCOCH₂CH₂CON), 33.7 (CHCH₃), 34.2 (CH₂CH₂CH₃), 34.6 (CH₂C₇H₁₅), 35.4 (COCH₂C₈H₁₇), 37.0 (CH₂NHCOCH₂CH₂CON), 38.2 (CH₂C₄H₉), 38.5 (CH₂NHCOC₉H₁₉), 39.9 (NHC(NH₂)NHCH₂), 45.0 (CH₂NCH₂), 46.6 (CH₂NHCH₂), 47.0 (CH₂NCH₂), 159.2 (NHC(NH₂)NH), 174.4 (NHCOCH₂CH₂CON), 176.9 (NHCOCH₂CH₂CON + NHCOC₉H₁₉); MS (ES⁺): m/z (%): 348.6 (100) (M+2H)²⁺, 695.8 (15) (M+H)⁺; HRMS (ES⁺): m/z: calcd for C₃₇H₇₅N₈O₄ [M+H]⁺: 695.5906; found: 695.5925.

***N*¹-(3(*N*',*N*'-bis-(3'(4-Ethyl-octanamidopropyl)carbamoyl)propionyl-*N*⁹-(carbamimidoyl)-norspermidine (3.21).**

Yield : (resin: 0.64 mmol/g, 38.6 mg) 15 mg, 59 %.

*t*_R = 9.6 min (RP-HPLC, 100 % pure); IR (Film): ν = 1671, 1621 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.80 (m, 12H; 2xCHCH₂CH₃ + 2xCH₂CH₂CH₃), 1.20 (m, 18H; 2xCH(CH₂CH₃)(CH₂)₃CH₃), 1.47 (m, 4H; 2xCH₂C₇H₁₅), 1.61, 1.72 (m, 4H; CH₂CH₂NCH₂CH₂), 1.78, 1.89 (m, 4H; CH₂CH₂NCH₂CH₂), 2.08 (m, 4H; COCH₂C₈H₁₇), 2.40 (t, ³*J*(HH) = 6 Hz, 2H; NHCOCH₂CH₂CON), 2.60 (t, ³*J*(HH) = 6 Hz, 2H; NHCOCH₂CH₂CON), 2.96 (m, 4H; CH₂NHCH₂), 3.03 (t, ³*J*(HH) = 7 Hz, 2H; CH₂NHCOC₉H₁₉), 3.11 (t, ³*J*(HH) = 7 Hz, 2H; CH₂NHCOC₉H₁₉), 3.24 (m, 8H; NHC(NH₂)NHCH₂ + CH₂NHCOCH₂CH₂CON + CH₂NCH₂); ¹³C NMR (100 MHz, CD₃OD) δ = 11.5 (CH₂CH₂CH₃), 14.8 (CHCH₂CH₃), 24.5 (CH₂CH₂CH₃), 27.0 (CH₂CH₂CH₃), 27.2, 28.2 (CH₂CH₂NHCH₂CH₂), 29.1 (CH₂CH₂NCH₂CH₂), 29.5 (NHCOCH₂CH₂CON), 30.0 (CH₂CH₂NCH₂CH₂), 30.4 (CH₂C₃H₇), 31.0 (CH₂C₇H₁₅), 32.0 (NHCOCH₂CH₂CON), 34.1 (CHCH₂CH₃), 35.1 (COCH₂C₈H₁₇), 37.0 (CH₂NHCOCH₂CH₂CON), 38.2, 38.5 (2xCH₂NHCOC₉H₁₉), 39.9 (NHC(NH₂)NHCH₂), 40.4 (CHCH₂CH₃), 45.0 (CH₂NCH₂), 46.6 (CH₂NHCH₂), 47.0 (CH₂NCH₂), 159.2 (NHC(NH₂)NH), 174.4 (NHCOCH₂CH₂CON), 176.9 (NHCOCH₂CH₂CON), 177.2 (NHCOC₉H₁₉); MS (ES⁺): *m/z* (%): 348.6 (100) (M+2H)²⁺, 695.8 (15) (M+H)⁺; HRMS (ES⁺): *m/z*: calcd for C₃₇H₇₅N₈O₄ [M+H]⁺: 695.5906; found: 695.5913.

***N*¹-(3(*N*',*N*'-bis-(3'(Undecanamidopropyl)carbamoyl)propionyl-*N*⁹-(carbamimidoyl)-norspermidine (3.22).**

Yield : (resin: 0.64 mmol/g, 36.0 mg) 27 mg, quantitative yield.

*t*_R = 10.6 min (RP-HPLC, 78 % pure); IR (Film): ν = 1670, 1626 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.91 (t, ³*J*(HH) = 7 Hz, 6H; 2xCH₂CH₃), 1.31 (br s, 28H; 2xCH₂(CH₂)₇CH₃), 1.61 (br s, 4H; 2xCH₂C₈H₁₇), 1.72, 1.84 (m, 4H; CH₂CH₂NCH₂CH₂), 1.89, 2.00 (m, 4H; CH₂CH₂NHCH₂CH₂), 2.20 (m, 4H; 2xCOCH₂C₉H₁₉), 2.52 (t, ³*J*(HH) = 6 Hz, 2H; NHCOCH₂CH₂CON), 2.74 (t, ³*J*(HH) = 6 Hz, 2H; NHCOCH₂CH₂CON), 3.07 (m, 4H; CH₂NHCH₂), 3.15 (t, ³*J*(HH) = 7 Hz, 2H; CH₂NHCOC₁₀H₂₁), 3.23 (t, ³*J*(HH) = 7 Hz, 2H; CH₂NHCOC₁₀H₂₁), 3.35 (m, 8H; NHC(NH₂)NHCH₂ + CH₂NHCOCH₂CH₂CON +

CH_2NCH_2); ^{13}C NMR (100 MHz, CD_3OD) δ = 16.3 (CH_2CH_3), 25.6 (CH_2CH_3), 28.7 ($CH_2CH_2NHCH_2CH_2$), 29.0 ($CH_2C_8H_{17}$), 29.7 ($CH_2CH_2NHCH_2CH_2$), 30.6 ($CH_2CH_2NCH_2CH_2$), 31.0 ($NHCOCH_2CH_2CON$), 31.5 ($CH_2CH_2NCH_2CH_2$), 32.0, 32.3, 32.5, 32.6 ($CH_2(CH_2)_5C_3H_7$), 33.4 ($NHCOCH_2CH_2CON$), 34.9 ($CH_2CH_2CH_3$), 38.5 ($CH_2NHCOCH_2CH_2CON$), 39.1 ($COCH_2C_9H_{19}$), 39.7 + 39.9 ($2 \times CH_2NHCOC_{10}H_{21}$), 41.4 ($NHC(NH_2)NHCH_2$), 46.5 (CH_2NCH_2), 48.1 (CH_2NHCH_2), 48.5 (CH_2NCH_2), 160.7 ($NHC(NH_2)NH$), 175.8 ($NHCOCH_2CH_2CON$), 178.4 ($NHCOCH_2CH_2CON$ + $NHCOC_{10}H_{21}$); MS (ES^+): m/z (%): 362.6 (100) ($M+2H$) $^{2+}$, 724.0 (4) ($M+H$) $^+$; HRMS (ES^+): m/z: calcd for $C_{39}H_{79}N_8O_4$ [$M+H$] $^+$: 723.6219; found: 723.6213.

***N*¹-(3(*N*′, *N*′-bis-(3′(Dodecanamidopropyl)carbamoyl)propionyl)-*N*⁹-(carbamiimidoyl)-norspermidine (3.23).**

Yield : (resin: 0.64 mmol/g, 38.6 mg) 25 mg, 93 %.

t_R = 11.5 min (RP-HPLC, 29 % pure); IR (Film): ν = 1672, 1633 cm^{-1} (C=O); 1H NMR (400 MHz, CD_3OD) δ = 0.80 (t, $^3J(HH)$ = 7 Hz, 6H; $2 \times CH_2CH_3$), 1.19 (br s, 32H; $2 \times CH_2(CH_2)_8CH_3$), 1.50 (br s, 4H; $2 \times CH_2C_9H_{19}$), 1.61, 1.72 (m, 4H; $CH_2CH_2NCH_2CH_2$), 1.78, 1.89 (m, 4H; $CH_2CH_2NHCH_2CH_2$), 2.09 (m, 4H; $2 \times COCH_2C_{10}H_{21}$), 2.40 (t, $^3J(HH)$ = 6 Hz, 2H; $NHCOCH_2CH_2CON$), 2.60 (t, $^3J(HH)$ = 6 Hz, 2H; $NHCOCH_2CH_2CON$), 2.96 (m, 4H; CH_2NHCH_2), 3.04 (t, $^3J(HH)$ = 7 Hz, 2H; $CH_2NHCOC_{11}H_{23}$), 3.12 (t, $^3J(HH)$ = 7 Hz, 2H; $CH_2NHCOC_{11}H_{23}$), 3.23 (m, 8H; $NHC(NH_2)NHCH_2$ + $CH_2NHCOCH_2CH_2CON$ + CH_2NCH_2); ^{13}C NMR (100 MHz, CD_3OD) δ = 15.1 (CH_2CH_3), 24.4 (CH_2CH_3), 27.5 ($CH_2CH_2NHCH_2CH_2$), 27.7 ($CH_2C_9H_{19}$), 28.4 ($CH_2CH_2NHCH_2CH_2$), 29.3 ($CH_2CH_2NCH_2CH_2$), 29.8 ($NHCOCH_2CH_2CON$), 30.3 ($CH_2CH_2NCH_2CH_2$), 31.0, 31.1, 31.3, 31.4 ($CH_2(CH_2)_6C_3H_7$), 32.2 ($NHCOCH_2CH_2CON$), 33.7 ($CH_2CH_2CH_3$), 37.3 ($CH_2NHCOCH_2CH_2CON$), 37.8 ($COCH_2C_{10}H_{21}$), 38.5 + 38.7 ($2 \times CH_2NHCOC_{11}H_{23}$), 40.2 ($NHC(NH_2)NHCH_2$), 45.3 (CH_2NCH_2), 46.9 (CH_2NHCH_2), 47.3 (CH_2NCH_2), 159.5 ($NHC(NH_2)NH$), 174.6 ($NHCOCH_2CH_2CON$), 177.0 ($NHCOCH_2CH_2CON$), 177.1 ($NHCOC_{11}H_{23}$); MS (ES^+): m/z (%): 376.5 (100) ($M+2H$) $^{2+}$, 752.0 (4) ($M+H$) $^+$; HRMS (ES^+): m/z: calcd for $C_{41}H_{83}N_8O_4$ [$M+H$] $^+$: 751.6532; found: 751.6536.

***N*¹-(3(*N*',*N*'-bis-(3'(Tetradecanamidopropyl)carbamoyl)propionyl-*N*⁹-(carbamimidoyl)-norspermidine (3.24).**

Yield : (resin: 0.64 mmol/g, 38.0 mg) 24 mg, 82 %.

$t_R = 13.1$ min (RP-HPLC, 80 % pure); IR (Film): $\nu = 1671, 1631$ cm^{-1} (C=O); ¹H NMR (400 MHz, CD₃OD) $\delta = 0.91$ (t, ³*J*(HH) = 7 Hz, 6H; 2xCH₂CH₃), 1.30 (br s, 40H; 2xCH₂(CH₂)₁₀CH₃), 1.61 (br s, 4H; 2xCH₂C₉H₁₉), 1.72, 1.83 (m, 4H; CH₂CH₂NCH₂CH₂), 1.89, 2.00 (m, 4H; CH₂CH₂NHCH₂CH₂), 2.20 (m, 4H; 2xCOCH₂C₁₀H₂₁), 2.52 (m, 2H; NHCOCH₂CH₂CON), 2.71 (m, 2H; NHCOCH₂CH₂CON), 3.07 (m, 4H; CH₂NHCH₂), 3.15 (t, ³*J*(HH) = 7 Hz, 2H; CH₂NHCOC₁₃H₂₇H₂); 3.23 (t, ³*J*(HH) = 7 Hz, 2H; CH₂NHCOC₁₃H₂₇H₂); 3.35 (m, 8H; NHC(NH₂)NHCH₂ + CH₂NHCOCH₂CH₂CON + CH₂NCH₂); ¹³C NMR (100 MHz, CD₃OD) $\delta = 14.8$ (CH₂CH₃), 24.1 (CH₂CH₃), 27.2 (CH₂CH₂NHCH₂CH₂), 27.5 (CH₂C₁₂H₂₅), 28.1 (CH₂CH₂NHCH₂CH₂), 29.1 (CH₂CH₂NCH₂CH₂), 29.5 (NHCOCH₂CH₂CON), 30.0 (CH₂CH₂NCH₂CH₂), 30.7, 30.8, 31.0, 31.1 (CH₂(CH₂)₈C₃H₇), 32.0 (NHCOCH₂CH₂CON), 33.5 (CH₂CH₂CH₃), 37.0 (CH₂NHCOCH₂CH₂CON), 37.6 (COCH₂C₁₂H₂₅), 38.2, 38.4 (2xCH₂NHCOC₁₃H₂₇), 39.9 (NHC(NH₂)NHCH₂), 45.0 (CH₂NCH₂), 46.6 (CH₂NHCH₂), 47.1 (CH₂NCH₂), 159.2 (NHC(NH₂)NH), 174.4 (NHCOCH₂CH₂CON), 176.9 (NHCOCH₂CH₂CON + NHCOC₁₃H₂₇); MS (ES⁺): *m/z* (%): 404.7 (100) (M+2H)²⁺, 807.8 (10) (M+H)⁺; HRMS (ES⁺): *m/z*: calcd for C₄₅H₉₁N₈O₄ [M+H]⁺: 807.7158; found: 807.7140.

***N*¹-(3(*N*',*N*'-bis-(3'(Pentadecanamidopropyl)carbamoyl)propionyl-*N*⁹-(carbamimidoyl)-norspermidine (3.25).**

Yield : (resin: 0.64 mmol/g, 39.4 mg) 33 mg, quantitative yield.

$t_R = 13.9$ min (RP-HPLC, 88 % pure); IR (Film): $\nu = 1671, 1631$ cm^{-1} (C=O); ¹H NMR (400 MHz, CD₃OD) $\delta = 0.89$ (m, 6H; 2xCH₂CH₃), 1.28 (br s, 44H; 2xCH₂(CH₂)₁₁CH₃), 1.60 (br s, 4H; 2xCH₂C₁₂H₂₅), 1.71, 1.82 (m, 4H; CH₂CH₂NCH₂CH₂), 1.89, 1.99 (m, 4H; CH₂CH₂NHCH₂CH₂), 2.18 (m, 4H; 2xCOCH₂C₁₃H₂₇), 2.51 (m, 2H; NHCOCH₂CH₂CON), 2.69 (m, 2H; NHCOCH₂CH₂CON), 3.05 (m, 4H; CH₂NHCH₂), 3.14, 3.22 (m, 4H; 2xCH₂NHCOC₁₄H₂₉), 3.34 (m, 8H; NHC(NH₂)NHCH₂ + CH₂NHCOCH₂CH₂CON + CH₂NCH₂); ¹³C NMR (100 MHz, CD₃OD) $\delta = 15.1$ (CH₂CH₃), 24.2 (CH₂CH₃), 27.2 (CH₂CH₂NHCH₂CH₂), 27.5 (CH₂C₁₂H₂₅), 28.1 (CH₂CH₂NHCH₂CH₂), 29.1

(CH₂CH₂NCH₂CH₂), 29.6 (NHCOCH₂CH₂CON), 30.0 (CH₂CH₂NCH₂CH₂), 30.8, 30.9, 31.0, 31.1, 31.2 (CH₂(CH₂)₉C₃H₇), 32.1 (NHCOCH₂CH₂CON), 33.5 (NHC(NH₂)NHCH₂), 37.0 (CH₂NHCOCH₂CH₂CON), 37.7 (COCH₂C₁₃H₂₇), 38.3, 38.5 (CH₂NHCOC₁₄H₂₉), 39.9 (NHC(NH₂)NHCH₂), 45.1 (CH₂NCH₂), 46.7 (CH₂NHCH₂), 47.1 (CH₂NCH₂), 159.2 (NHC(NH₂)NH), 174.4 (NHCOCH₂CH₂CON), 176.8 (NHCOCH₂CH₂CON + NHCOC₁₄H₂₉); MS (ES⁺): m/z (%): 418.8 (100) (M+2H)²⁺, 863.1 (5) (M+H)⁺; HRMS (ES⁺): m/z: calcd for C₄₇H₉₅N₈O₄ [M+H]⁺: 835.7471; found: 835.7485.

***N*¹-(3(*N*',*N*'-bis-(3'(Pamitionamidopropyl)carbamoyl)propionyl-*N*⁹-(carbamimidoyl)-norspermidine (3.26).**

Yield : (resin: 0.64 mmol/g, 39.3 mg) 34 mg, quantitative yield.

*t*_R = 14.9 min (RP-HPLC, 89 % pure); IR (Film): ν = 1671, 1631 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.70 (t, ³J(HH) = 7 Hz, 6H; 2xCH₂CH₃), 1.08 (br s, 48H; 2xCH₂(CH₂)₁₂CH₃), 1.42 (br s, 4H; 2xNHCOCH₂CH₂C₁₃H₂₇), 1.52, 1.64 (m, 4H; CH₂CH₂NCH₂CH₂), 1.70, 1.81 (m, 4H; (CH₂CH₂NHCH₂CH₂), 2.00 (m, 4H; 2xNHCOCH₂C₁₄H₂₉), 2.32 (m, 2H; NHCOCH₂CH₂CON), 2.50 (m, 2H; NHCOCH₂CH₂CON), 2.85 (m, 4H; CH₂NHCH₂), 2.95 (t, ³J(HH) = 7 Hz, 2H; CH₂NHCOC₁₅H₃₁), 3.03 (t, ³J(HH) = 6 Hz, 2H; CH₂NHCOC₁₅H₃₁), 3.15 (m, 8H; CH₂CH₂CH₂NHCH₂CH₂CH₂ + CH₂NCH₂), ¹³C NMR (100 MHz, CD₃OD) δ = 15.2 (CH₂CH₃), 24.2 (CH₂CH₃), 27.2 (CH₂CH₂NHCH₂CH₂), 27.6 (NHCOCH₂CH₂C₁₃H₂₇), 28.1 (CH₂CH₂NHCH₂CH₂), 29.1 (CH₂CH₂NCH₂CH₂), 29.7 (NHCOCH₂CH₂CON), 30.1 (CH₂CH₂NCH₂CH₂), 30.8, 30.9, 31.1, 31.2, 31.3 (COCH₂CH₂(CH₂)₁₀C₃H₇), 32.1 (NHCOCH₂CH₂CON), 33.5 (CH₂CH₂CH₃), 37.0 (CH₂NHCOCH₂CH₂CON), 37.8, 37.9 (NHCOCH₂C₁₄H₂₉), 38.3, 38.5 (CH₂NHCOC₁₅H₃₁), 39.9 (NHC(NH₂)NHCH₂), 45.1 (CH₂NCH₂), 46.7 (CH₂NHCH₂), 47.2 (CH₂NCH₂), 159.2 (NHC(NH₂)NH), 174.3 (NHCOCH₂CH₂CON), 176.8 (NHCOCH₂CH₂CON + NHCOC₁₅H₃₁); MS (ES⁺): m/z (%): 432.8 (100) (M+2H)²⁺, 864.1 (5) (M+H)⁺; HRMS (ES⁺): m/z: calcd for C₄₉H₉₉N₈O₄ [M+H]⁺: 863.7784; found: 863.7781.

***N*¹-3(*N*',*N*'-bis-(3'(Oleonamidopropyl)carbamoyl)propionyl-*N*⁹-(carbamimidoyl)-norspermidine (3.27).**

Yield : (resin: 0.64 mmol/g, 39.0 mg) 33 mg, quantitative yield.

*t*_R = 15.4 min (RP-HPLC, 68 % pure); IR (Film): ν = 1670, 1626 cm⁻¹ (C=O); ¹H NMR (400 MHz, CD₃OD) δ = 0.91 (t, ³*J*(HH) = 7 Hz, 6H; 2xCH₂CH₃), 1.31 (m, 40H; 2xNHCOCH₂CH₂(CH₂)₄CH₂CHCHCH₂(CH₂)₆CH₃), 1.61 (s, 4H; 2xNHCOCH₂CH₂C₁₅H₂₉), 1.71, 1.83 (m, 4H; CH₂CH₂NCH₂CH₂), 1.89 (m, 2H; CH₂CH₂NHCH₂CH₂), 2.03 (m, 10H; CH₂CH₂NHCH₂CH₂ + 2xCH₂CHCHCH₂), 2.19 (m, 4H; 2xNHCOCH₂C₁₆H₃₁), 2.51 (m, 2H; NHCOCH₂CH₂CON), 2.70 (m, 2H; NHCOCH₂CH₂CON), 3.06 (m, 4H; CH₂NHCH₂), 3.14, 3.22 (m, 4H; 2xCH₂NHCOC₁₇H₃₃), 3.34 (m, 8H; CH₂CH₂CH₂NHCH₂CH₂CH₂ + CH₂NCH₂), ¹³C NMR (100 MHz, CD₃OD) δ = 14.8 (CH₂CH₃), 24.1 (CH₂CH₃), 27.2 (CH₂CH₂NHCH₂CH₂), 27.5 (NHCOCH₂CH₂C₁₅H₂₉), 28.1 (CH₂CH₂NHCH₂CH₂), 28.5 (CH₂CHCHCH₂), 29.0 (CH₂CH₂NCH₂CH₂), 29.5 (NHCOCH₂CH₂CON), 30.0 (CH₂CH₂NCH₂CH₂), 30.6, 30.7, 30.8, 31.0, 31.2 (CH₂(CH₂)₄CH₂CH=CHCH₂(CH₂)₂CH₂CH₂C₃H₇), 32.0 (NHCOCH₂CH₂CON), 33.4 (CH₂C₄H₉), 37.1 (CH₂NHCOCH₂CH₂CON), 37.8, 37.6 (NHCOCH₂C₁₆H₃₁), 38.2, 38.4 (CH₂NHCOC₁₇H₃₃), 39.9 (NHC(NH₂)NHCH₂), 45.0 (CH₂NCH₂), 46.6 (CH₂NHCH₂), 47.1 (CH₂NCH₂), 131.1, 131.3 (CH=CH), 159.2 (NHC(NH₂)NH), 174.3 (NHCOCH₂CH₂CON), 176.7 (NHCOCH₂CH₂CON + NHCOC₁₇H₃₃); MS (ES⁺): m/z (%): 458.8 (100) (M+2H)²⁺, 916.0 (5) (M+H)⁺; HRMS (ES⁺): m/z: calcd for C₅₃H₁₀₃N₈O₄ [M+H]⁺: 915.8097; found: 863.7781.