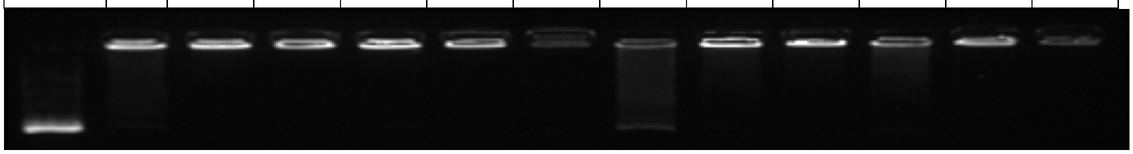
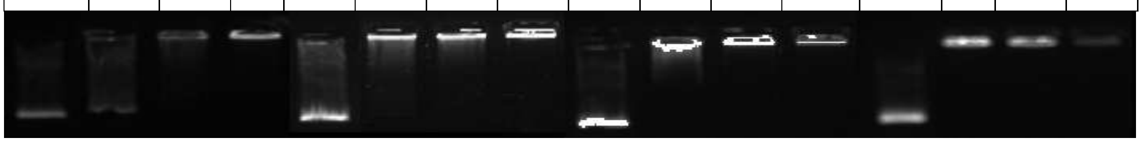
Electrophoretic Mobility Assay (Supporting Information)

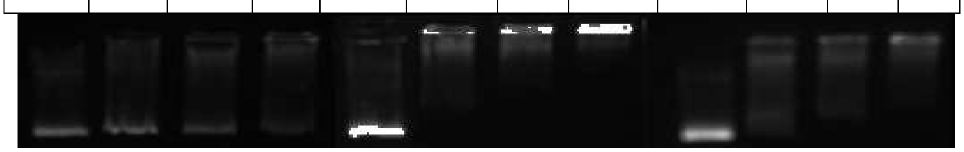
DNA	69			70			72			71		
w/w	6	12	18	6	12	18	6	12	18	6	12	18



DNA	76			DNA	49			DNA	80			DNA	84		
w/w	6	12	18		6	12	18		6	12	18		6	12	18



DNA	74			DNA	78			DNA	82		
w/w	6	12	18		6	12	18		6	12	18



Analytical Data

1H NMR spectra were recorded using a Jeol JNMR-GX 400 (400 MHz) or JNMR-GX 500 (500 MHz) unit produced by Jeol. All spectra were recorded without TMS as internal standard and therefore all signals were calibrated to the residual proton signal of the solvent. The coupling constant had an accuracy of 0.3 Hz. Chemical shifts are reported in ppm and refer to the solvent as internal standard (D_2O at 4.80 or methanol D_4 at 3.3 and 4.87). Data are reported as s = singlet, d = doublet, t = triplet, m = multiplet; integration was performed manually. The spectra were analyzed using MestreNova (Ver. 5.2.5-4119 by MestReLab Research).

Sequence: HO-K-Stp$_1$-K #: **1**
Mass calculated: 545.7 Found: 546.9
NMR: ^{1}H-NMR (400 MHz, D_2O, 17.0 °C): δ = 1.29 - 1.48 (m, 4H, γ-C<u>H</u>$_2$-, Lys), 1.54 - 1.68 (m, 4H, δ-C<u>H</u>$_2$-, Lys),1.69 - 1.93 (m, 4H, β-C<u>H</u>$_2$-, Lys), 2.42 - 2.52 (m, 2H, O=C-C<u>H</u>$_2$-, Stp), 2.52 - 2.62 (m, 2H, O=C-C<u>H</u>$_2$-, Stp), 2.87 – 2.94 (t, *J*=7.6 Hz, 4H, ε–C<u>H</u>$_2$-, Lys), 3.15 – 3.32 (m, 4H, N-C<u>H</u>$_2$-, Stp), 3.35 – 3.55 (m, 12H, N-C<u>H</u>$_2$-, Stp), 3.89 – 3.99 (t, *J*=6.64 Hz, 1H, O=C-C(R)<u>H</u>-NH-, Lys), 4.20 – 4.25 (dd, *J*=5.3, 8.8, 1H, O=C-C(R)<u>H</u>-NH-, Lys)

Sequence: HO-K-Stp$_2$-K #: **6**
Mass calculated: 817.1 Found: 817.5
NMR: ^{1}H-NMR (400 MHz, D_2O, 16.8 °C): δ = 1.29 - 1.46 (m, 4H, γ-C<u>H</u>$_2$-, Lys), 1.56 - 1.76 (m, 4H, δ-C<u>H</u>$_2$-, Lys),1.76 - 1.92 (m, 4H, β-C<u>H</u>$_2$-, Lys), 2.43 - 2.51 (m, 6H, O=C-C<u>H</u>$_2$-, Stp), 2.52 - 2.62 (m, 2H, O=C-C<u>H</u>$_2$-, Stp), 2.87 – 2.95 (t, *J*=7.6 Hz, 4H, ε–C<u>H</u>$_2$-, Lys), 3.13 – 3.29 (m, 8H, N-C<u>H</u>$_2$-, Stp), 3.35 – 3.55 (m, 24H, N-C<u>H</u>$_2$-, Stp), 3.89 – 3.99 (t, *J*=6.64 Hz, 1H, O=C-C(R)<u>H</u>-NH-, Lys), 4.20 – 4.25 (dd, *J*=5.2, 8.8, 1H, O=C-C(R)<u>H</u>-NH-, Lys)

Sequence: HO-K-Stp$_5$-K #: **23**
Mass calculated: 1631.2 Found: 1634.0
NMR: ^{1}H-NMR (400 MHz, D_2O, 17.1 °C): δ = 1.28 - 1.49 (m, 4H, γ-C<u>H</u>$_2$-, Lys), 1.49 - 1.88 (m, 8H, δ-C<u>H</u>$_2$-/β-C<u>H</u>$_2$-, Lys), 2.40 - 2.52 (m, 18H, O=C-C<u>H</u>$_2$-, Stp), 2.52 - 2.60 (m, 2H, O=C-C<u>H</u>$_2$-, Stp), 2.87 – 2.95 (t, *J*=7.6 Hz, 4H, ε–C<u>H</u>$_2$, Lys), 3.13 – 3.26 (m, 20H, N-C<u>H</u>$_2$-, Stp), 3.35 – 3.55 (m, 60H, N-C<u>H</u>$_2$-, Stp), 3.89 – 3.99 (t, *J*=6.64 Hz, 1H, O=C-C(R)<u>H</u>-NH-, Lys), 4.20 – 4.25 (dd, *J*=5.2, 8.8, 1H, O=C-C(R)<u>H</u>-NH-, Lys)

Sequence: HO-K-Stp$_2$-ButA$_1$ #: **34**
Mass calculated: 759.0 Found: 759.8
NMR: ^{1}H-NMR (400 MHz, D_2O, 17.3 °C): δ = 0.79 – 0.86 (t, *J*=7.4 Hz, 3H, -C<u>H</u>$_3$, ButA)**,** 1.34 - 1.45 (m, 2H, γ-C<u>H</u>$_2$, Lys), 1.46 - 1.56 (m, 2H,-C<u>H</u>$_2$-, ButA), 1.56 - 1.87 (m, 4H, δ-C<u>H</u>$_2$/β-C<u>H</u>$_2$, Lys), 2.14 – 2.23 (t, *J*=7.4 Hz, 2H, O=C-C<u>H</u>$_2$, ButA), 2.44 - 2.51 (m, 6H, O=C-C<u>H</u>$_2$-, Stp), 2.51 - 2.60

(m, 2H, O=C-C$\underline{H}_2$-, Stp), 2.87 – 2.96 (t, *J*=7.8 Hz, 2H, ε–C$\underline{H}_2$, Lys), 3.15 – 3.26 (m, 8H, N-C$\underline{H}_2$-, Stp), 3.37 – 3.54 (m, 12H, N-C$\underline{H}_2$-, Stp), 4.18 – 4.27 (m, 1H, O=C-C(R)$\underline{H}$-NH-, Lys)

Sequence: HO-K-Stp_2-$CapA_1$ #: **35**
Mass calculated: 815.1 Found: 815.6
NMR: ^{1}H-NMR (400 MHz, D_2O, 16.7 °C): δ = 0.73 – 0.82 (m, 3H, -C$\underline{H}_3$, CapA), 1.15 – 1.26 (m, 8H, -C$\underline{H}_2$-, CapA), 1.34 - 1.44 (m, 2H, γ-C$\underline{H}_2$, Lys), 1.45 - 1.56 (m, 2H, δ-C$\underline{H}_2$, Lys) 1.57 - 1.66 (m, 2H,O=C-CH_2-C$\underline{H}_2$-, CapA), 1.67 - 1.82 (m, 2H, β-C$\underline{H}_2$, Lys), 2.15 – 2.24 (t, *J*=7.4 Hz, 2H, O=C-C$\underline{H}_2$, CapA), 2.44 - 2.52 (m, 6H, O=C-C$\underline{H}_2$-, Stp), 2.52 - 2.60 (m, 2H, O=C-C$\underline{H}_2$-, Stp), 2.88 – 2.96 (t, *J*=7.8 Hz 2H, ε–C$\underline{H}_2$, Lys), 3.16 – 3.25 (m, 8H, N-C$\underline{H}_2$-, Stp), 3.37 – 3.51 (m, 24H, N-C$\underline{H}_2$-, Stp), 4.16 – 4.25 (m, 1H, O=C-C(R)$\underline{H}$-NH-, Lys)

Sequence: HO-K-Stp_2-$MyrA_1$ #: **36**
Mass calculated: 899.3 Found: 899.7
NMR: ^{1}H-NMR (400 MHz, D_2O, 17.6 °C): δ = 0.74 – 0.81 (m, 3H, -C$\underline{H}_3$, MyrA), 1.17 – 1.24 (m, 20H, -C$\underline{H}_2$-, MyrA), 1.34 - 1.57 (m, 4H, δ-C$\underline{H}_2$/γ-C$\underline{H}_2$, Lys), 1.56 - 1.66 (m, 2H,O=C-CH_2-C$\underline{H}_2$-, MyrA), 1.67 - 1.83 (m, 2H, β-C$\underline{H}_2$, Lys), 2.16 – 2.24 (t, *J*=7.4 Hz, 2H, O=C-C$\underline{H}_2$-, MyrA), 2.44 - 2.52 (m, 6H, O=C-C$\underline{H}_2$-, Stp), 2.52 - 2.58 (m, 2H, O=C-C$\underline{H}_2$-, Stp), 2.88 – 2.96 (t, *J*=7.8 Hz, 2H, ε–C$\underline{H}_2$, Lys), 3.15 – 3.25 (m, 8H, N-C$\underline{H}_2$-, Stp), 3.37 – 3.52 (m, 24H, N-C$\underline{H}_2$-, Stp), 4.18 – 4.26 (m, 1H, O=C-C(R)$\underline{H}$-NH-, Lys)

Sequence: HO-K-Stp_2-$OleA_1$ #: **37**
Mass calculated: 953.4 Found: 953.77
NMR: ^{1}H-NMR (400 MHz, D_2O, 16.1 °C): δ = 0.71 – 0.83 (m, 3H, -C$\underline{H}_3$, OleA), 1.16 – 1.29 (m, 18H, -C$\underline{H}_2$-, OleA), 1.31 - 1.82 (m, 12H, β-C$\underline{H}_2$,/δ-C$\underline{H}_2$/γ-C$\underline{H}_2$, Lys; -C$\underline{H}_2$-CH=CH-C$\underline{H}_2$-, O=C-CH_2-C$\underline{H}_2$-, OleA), 2.13 – 2.25 (m, 2H, O=C-C$\underline{H}_2$-, OleA), 2.41 - 2.52 (m, 6H, O=C-C$\underline{H}_2$-, Stp), 2.52 - 2.61 (m, 2H, O=C-C$\underline{H}_2$-, Stp), 2.86 – 2.97 (m, 2H, ε–C$\underline{H}_2$-, Lys), 3.12 – 3.25 (m, 8H, N-C$\underline{H}_2$-, Stp), 3.36 – 3.53 (m, 24H, N-C$\underline{H}_2$-, Stp), 4.16 – 4.24 (m, 1H, O=C-C(R)$\underline{H}$-NH-, Lys)

Sequence: HO-K-Stp_2-K-$CapA_2$ #: **8**
Mass calculated: 1069.5 Found: 1070.5
NMR: ^{1}H-NMR (400 MHz, D_2O, 16.9 °C): δ = 0.72 – 0.81 (m, 6H, -C$\underline{H}_3$, CapA), 1.09 – 1.25 (m, 16H, -C$\underline{H}_2$-, CapA), 1.26 - 1.90 (m, 16H, δ-C$\underline{H}_2$/γ-C$\underline{H}_2$/β-C$\underline{H}_2$, Lys; O=C-CH_2-C$\underline{H}_2$-, CapA), 2.08 – 2.32 (m, 4H, O=C-C$\underline{H}_2$, CapA), 2.42 - 2.50 (m, 6H, O=C-C$\underline{H}_2$-, Stp), 2.52 - 2.59 (m, 2H, O=C-C$\underline{H}_2$-, Stp), 2.87 – 2.95 (t, *J*=7.7 Hz, 2H, ε–C$\underline{H}_2$, Lys), 3.06 – 3.13 (m, 2H, ε–C$\underline{H}_2$, Lys), 3.14 – 3.25 (m, 8H, N-C$\underline{H}_2$-, Stp), 3.33 – 3.56 (m, 24H, N-C$\underline{H}_2$-, Stp), 4.06 – 4.16 (m, 1H, O=C-C(R)$\underline{H}$-NH-, Lys), 4.17 – 4.24 (m, 1H, O=C-C(R)$\underline{H}$-NH-, Lys)

Sequence: HO-K-Stp_2-K-$MyrA_2$ #: **9**
Mass calculated: 1237.8 Found: 1237.6

NMR: ^{1}H-NMR (400 MHz, D_2O, 16.8 °C): δ = 0.72 – 0.85 (m, 6H, -C$\underline{H}_3$, MyrA), 1.06 – 1.28 (m, 40H, -C$\underline{H}_2$-, MyrA), 1.26 - 1.90 (m, 16H, γ-C$\underline{H}_2$/δ-C$\underline{H}_2$/β-C$\underline{H}_2$, Lys; O=C-CH_2-C$\underline{H}_2$-, MyrA), 2.08 – 2.32 (m, 4H, O=C-C$\underline{H}_2$, MyrA), 2.42 - 2.53 (m, 6H, O=C-C$\underline{H}_2$-, Stp), 2.53 - 2.63 (m, 2H, O=C-C$\underline{H}_2$-, Stp), 2.87 – 2.98 (t, *J*=7.6 Hz, 2H, ε–C$\underline{H}_2$, Lys), 3.04 – 3.14 (m, 2H, ε–C$\underline{H}_2$, Lys), 3.15 – 3.28 (m, 8H, N-C$\underline{H}_2$-, Stp), 3.33 – 3.58 (m, 24H, N-C$\underline{H}_2$-, Stp), 4.05 – 4.22 (m, 2H, O=C-C(R)$\underline{H}$-NH-, Lys)

Sequence: HO-K-Stp_2-K-$OleA_2$ #: **10**

NMR: ^{1}H-NMR (400 MHz, D_2O, 16.9 °C): δ = 0.69 – 0.82 (m, 6H, -C$\underline{H}_3$, OleA), 0.99 – 1.30 (m, 40H, -C$\underline{H}_2$-, OleA), 1.32 – 1.91 (m, 24H, β-C$\underline{H}_2$,/δ-C$\underline{H}_2$/γ-C$\underline{H}_2$, Lys; -C$\underline{H}_2$-CH=CH-C$\underline{H}_2$, O=C-CH_2-C$\underline{H}_2$-; OleA), 1.92 – 1.99 (m, 4H, O=C-C$\underline{H}_2$, OleA), 2.42 - 2.51 (m, 6H, O=C-C$\underline{H}_2$-, Stp), 2.51 - 2.60 (m, 2H, O=C-C$\underline{H}_2$-, Stp), 2.87 – 2.99 (m, 4H, ε–C$\underline{H}_2$, Lys), 3.14 – 3.26 (m, 8H, N-C$\underline{H}_2$-, Stp), 3.35 – 3.55 (m, 24H, N-C$\underline{H}_2$-, Stp), 4.11 – 4.31 (m, 2H, O=C-C(R)$\underline{H}$-NH-, Lys)

Sequence: HO-K-Stp_4-K-$MyrA_2$ #: **21**

Mass calculated: 1780.5 Found: 1782.7

NMR: ^{1}H-NMR (400 MHz, D_2O, 16.8 °C): δ = 0.72 – 0.85 (m, 6H, -C$\underline{H}_3$, MyrA), 1.07 – 1.29 (m, 40H, -C$\underline{H}_2$-, MyrA), 1.26 - 1.88 (m, 16H, δ-C$\underline{H}_2$/γ-C$\underline{H}_2$/β-C$\underline{H}_2$, Lys; O=C-CH_2-C$\underline{H}_2$-, MyrA), 2.10 – 2.29 (m, 4H, O=C-C$\underline{H}_2$, MyrA), 2.40 - 2.52 (m, 14H, O=C-C$\underline{H}_2$-, Stp), 2.52 - 2.63 (m, 2H, O=C-C$\underline{H}_2$-, Stp), 2.87 – 2.97 (t, *J*=7.8 Hz, 2H, ε–C$\underline{H}_2$, Lys), 3.06 – 3.14 (m, 2H, ε–C$\underline{H}_2$, Lys), 3.13 – 3.28 (m, 16H, N-C$\underline{H}_2$-, Stp), 3.28 – 3.56 (m, 48H, N-C$\underline{H}_2$-, Stp), 4.06 – 4.22 (m, 1H, O=C-C(R)$\underline{H}$-NH-, Lys)

Sequence: HO-K-Stp_4-K-$OleA_2$ #: **22**

Mass calculated: 1888.7 Found: 1888.6

NMR: ^{1}H-NMR (400 MHz, D_2O, 16.9 °C): δ = 0.67 – 0.83 (m, 6H, -C$\underline{H}_3$, OleA), 0.99 – 1.30 (m, 40H, -C$\underline{H}_2$-, OleA), 1.32 – 1.91 (m, 24H, β-C$\underline{H}_2$,/δ-C$\underline{H}_2$/γ-C$\underline{H}_2$, Lys; -C$\underline{H}_2$-CH=CH-C$\underline{H}_2$, O=C-CH_2-C$\underline{H}_2$-; OleA), 1.92 – 1.99 (m, 4H, O=C-C$\underline{H}_2$, OleA), 2.41 - 2.52 (m, 14H, O=C-C$\underline{H}_2$-, Stp), 2.52 - 2.60 (m, 2H, O=C-C$\underline{H}_2$-, Stp), 2.87 – 2.96 (m, 2H, ε–C$\underline{H}_2$, Lys), 2.99 – 3.11 (m, 2H, ε–C$\underline{H}_2$, Lys), 3.11 – 3.26 (m, 16H, N-C$\underline{H}_2$-, Stp), 3.30 – 3.55 (m, 48H, N-C$\underline{H}_2$-, Stp), 4.07 – 4.24 (m, 2H, O=C-C(R)$\underline{H}$-NH-, Lys)

Sequence: HO-C-Stp_2-K-$MyrA_2$ #: **69**

NMR: ^{1}H-NMR (400 MHz, D_2O, 16.7 °C): δ = 0.70 – 0.84 (m, 6H, -C$\underline{H}_3$, MyrA), 1.08 – 1.32 (m, 40H, -C$\underline{H}_2$-, MyrA), 1.32 - 1.97 (m, 10H, δ-C$\underline{H}_2$/γ-C$\underline{H}_2$/β-C$\underline{H}_2$, Lys; O=C-CH_2-C$\underline{H}_2$-, MyrA), 2.11 – 2.32 (m, 4H, O=C-C$\underline{H}_2$, MyrA), 2.41 - 2.55 (m, 6H, O=C-C$\underline{H}_2$-, Stp), 2.55 - 2.67 (m, 2H, O=C-C$\underline{H}_2$-, Stp), 2.83 - 2.97 (m, 2H, β-C$\underline{H}_2$-, Cys), 3.04 – 3.17 (m, 2H, ε–C$\underline{H}_2$, Lys), 3.14 – 3.29 (m, 8H, N-C$\underline{H}_2$-, Stp), 3.29 – 3.60 (m, 24H, N-C$\underline{H}_2$-, Stp), 4.07 – 4.24 (m, 1H, O=C-C(R)$\underline{H}$-NH-, Lys), 4.24 – 4.40 (m, 1H, O=C-C(R)$\underline{H}$-NH-, Cys)

Sequence: HO-C-K-Stp$_2$-K-MyrA$_2$ #: **70**

NMR: ^{1}H-NMR (400 MHz, D$_2$O, 16.7 °C): δ = 0.70 – 0.84 (m, 6H, -C$\underline{H}_3$, MyrA), 1.08 – 1.32 (m, 40H, -C$\underline{H}_2$-, MyrA), 1.32 - 1.97 (m, 10H, δ-C$\underline{H}_2$/γ-C$\underline{H}_2$/β-C$\underline{H}_2$, Lys; O=C-CH$_2$-C$\underline{H}_2$-, MyrA), 2.11 – 2.32 (m, 4H, O=C-C$\underline{H}_2$, MyrA), 2.41 - 2.55 (m, 6H, O=C-C$\underline{H}_2$-, Stp), 2.55 - 2.67 (m, 2H, O=C-C$\underline{H}_2$-, Stp), 2.83 - 2.97 (m, 2H, β-C$\underline{H}_2$-, Cys), 3.04 – 3.17 (m, 2H, ε–C$\underline{H}_2$, Lys), 3.14 – 3.29 (m, 8H, N-C$\underline{H}_2$-, Stp), 3.29 – 3.60 (m, 24H, N-C$\underline{H}_2$-, Stp), 4.07 – 4.24 (m, 1H, O=C-C(R)$\underline{H}$-NH-, Lys), 4.24 – 4.40 (m, 1H, O=C-C(R)$\underline{H}$-NH-, Cys)

Sequence: HO-C-Stp$_2$-K-OleA$_2$ #: **72**

NMR: ^{1}H-NMR (400 MHz, MeOD, 16.8 °C): δ = 0.85 – 0.96 (m, 6H, -C$\underline{H}_3$, OleA), 1.21 – 1.40 (m, 40H, -C$\underline{H}_2$-, OleA), 1.41 - 1.92 (m, 10H, δ-C$\underline{H}_2$/γ-C$\underline{H}_2$/β-C$\underline{H}_2$, Lys; -C$\underline{H}_2$-CH=CH-C$\underline{H}_2$, O=C-CH$_2$-C$\underline{H}_2$-; OleA), 1.93 – 2.07 (m, 4H, O=C-C$\underline{H}_2$, OleA), 2.41 - 2.57 (m, 6H, O=C-C$\underline{H}_2$-, Stp), 2.57 - 2.74 (m, 2H, O=C-C$\underline{H}_2$-, Stp), 2.85 - 3.08 (m, 2H, β-C$\underline{H}_2$-, Cys), 3.08 – 3.19 (m, 2H, ε–C$\underline{H}_2$, Lys), 3.19 – 3.72 (m, 32H, N-C$\underline{H}_2$-, Stp), 4.04 – 4.17 (m, 1H, O=C-C(R)H-NH-, Lys), 4.14 – 4.57 (m, 1H, O=C-C(R)$\underline{H}$-NH-, Cys)

Sequence: HO-C-K-Stp$_2$-K-OleA$_2$ #: **71**

NMR: ^{1}H-NMR (400 MHz, MeOD, 16.8 °C): δ = 0.78 – 0.88 (m, 6H, -C$\underline{H}_3$, OleA), 1.12 – 1.35 (m, 40H, -C$\underline{H}_2$-, OleA), 1.34 - 1.89 (m, 18H, δ-C$\underline{H}_2$/γ-C$\underline{H}_2$/β-C$\underline{H}_2$, Lys; -C$\underline{H}_2$-CH=CH-C$\underline{H}_2$, O=C-CH$_2$-C$\underline{H}_2$-; OleA), 1.90 – 1.97 (m, 4H, O=C-C$\underline{H}_2$, OleA), 2.40 - 2.55 (m, 10H, O=C-C$\underline{H}_2$-, Stp), 2.54 - 2.65 (m, 2H, O=C-C$\underline{H}_2$-, Stp), 2.80 – 3.10 (m, 6H, ε–C$\underline{H}_2$, Lys + Cys), 3.12 – 3.28 (m, 12H, N-C$\underline{H}_2$-, Stp), 3.29 – 3.61 (m, 36H, N-C$\underline{H}_2$-, Stp), 4.16 – 4.45 (m,3H, O=C-C(R)$\underline{H}$-NH-, Lys + Cys)

Sequence: HO-C-Stp$_3$-C-K #: **51**

Mass calculated: 1166.6 Found: 1167.0

NMR: ^{1}H-NMR (400 MHz, D2O, 17.3 °C): δ = 1.37 - 1.49 (m, 2H, δ-C$\underline{H}$2, Lys), 1.59 - 1.71 (m, 2H, δ-C$\underline{H}_2$, Lys),1.80 - 1.97 (m, 2H, β-C$\underline{H}$2, Lys), 2.44 - 2.55 (m, 10H, O=C-C$\underline{H}$2-, Stp), 2.55 - 2.66 (m, 2H, O=C-C$\underline{H}$2-, Stp), 2.81 – 2.99 (m, 4H, ε–C$\underline{H}_2$, Lys + Cys), 3.13 – 3.29 (m, 12H, N-C$\underline{H}_2$- , Stp), 3.33 – 3.58 (m, 36H, Stp) 3.96 – 4.05 (m, 1H, O=C-C(R)$\underline{H}$-NH-, Lys), 4.33 – 4.46 (m, 2H, O=C-C(R)$\underline{H}$-NH-, Lys)

Sequence: HO-C-Stp$_3$-C-K-MyrA$_2$ #: **45**

Mass calculated: 1587.3 Found: 1589.4

NMR: ^{1}H-NMR (400 MHz, D$_2$O, 17.2 °C): δ = 0.71 – 0.85 (m, 6H, -C$\underline{H}_3$, MyrA), 1.05 – 1.31 (m, 40H, -C$\underline{H}_2$-, MyrA), 1.26 - 1.90 (m, 16H, δ-C$\underline{H}_2$/γ-C$\underline{H}_2$/β-C$\underline{H}_2$, Lys; O=C-CH$_2$-C$\underline{H}_2$-, MyrA), 2.08 – 2.33 (m, 4H, O=C-C$\underline{H}_2$, MyrA), 2.40 - 2.55 (m, 10H, O=C-C$\underline{H}_2$-, Stp), 2.54 - 2.67 (m, 2H, O=C-C$\underline{H}_2$-, Stp), 2.80 – 3.10 (m, 6H, ε–C$\underline{H}_2$, Lys + Cys), 3.12 – 3.28 (m, 12H, N-C$\underline{H}_2$-, Stp), 3.29 – 3.58 (m, 36H, N-C$\underline{H}_2$-, Stp), 4.16 – 4.44 (m, 3H, O=C-C(R)$\underline{H}$-NH-, Lys + Cys)

Sequence: HO-C-Stp_3-C-K-$OleA_2$ #: **46**

Mass calculated: 1695.4 Found: 1699.8

NMR: ^{1}H-NMR (400 MHz, D_2O, 17.3 °C): δ = 0.63 – 0.82 (m, 6H, -C$\underline{H}_3$, OleA), 1.01 – 1.33 (m, 40H, -C$\underline{H}_2$-, OleA), 1.34 - 1.89 (m, 18H, δ-C$\underline{H}_2$/γ-C$\underline{H}_2$/β-C$\underline{H}_2$, Lys; -C$\underline{H}_2$-CH=CH-C$\underline{H}_2$, O=C-CH_2-C$\underline{H}_2$-; OleA), 1.90 – 1.97 (m, 4H, O=C-C$\underline{H}_2$, OleA), 2.40 - 2.55 (m, 10H, O=C-C$\underline{H}_2$-, Stp), 2.54 - 2.65 (m, 2H, O=C-C$\underline{H}_2$-, Stp), 2.80 – 3.10 (m, 6H, ε–C$\underline{H}_2$, Lys + Cys), 3.12 – 3.28 (m, 12H, N-C$\underline{H}_2$-, Stp), 3.29 – 3.61 (m, 36H, N-C$\underline{H}_2$-, Stp), 4.16 – 4.45 (m, 3H, O=C-C(R)$\underline{H}$-NH-, Lys + Cys)

Sequence: HO-C-Stp_1-K(K-$OleA_2$)-Stp_1-C #: **76**

Mass calculated: 1552.3 Found: 1552.4

NMR: ^{1}H-NMR (400 MHz, D_2O, 16.7 °C): δ = 0.86 – 0.95 (m, 6H, -C$\underline{H}_3$, OleA), 1.01 – 1.40 (m, 40H, -C$\underline{H}_2$-, OleA), 1.41 - 1.92 (m, 24H, δ-C$\underline{H}_2$/γ-C$\underline{H}_2$/β-C$\underline{H}_2$, Lys; -C$\underline{H}_2$-CH=CH-C$\underline{H}_2$, O=C-CH_2-C$\underline{H}_2$-; OleA), 2.12 – 2.32 (m, 4H, O=C-C$\underline{H}_2$-, OleA), 2.44 - 2.73 (m, 16H, O=C-C$\underline{H}$2-, Stp), 2.81 – 3.22 (m, 8H, ε–C$\underline{H}_2$, Lys + Cys), 3.22 – 3.63 (m, 32H, N-C$\underline{H}_2$- , Stp), 3.99 – 4.25 (m, 2H, O=C-C(R)$\underline{H}$-NH-, Cys), 4.45 – 4.60 (m, 2H, O=C-C(R)$\underline{H}$-NH-, Lys), 7.05 – 7.33 (m, 4H, -C$\underline{H}$=C$\underline{H}$-, OleA)

Sequence: HO-C-Stp_2-K(K-$OleA_2$)-Stp_2-C #: **49**

Mass calculated: 2095.0 Found: 2094.4

NMR: ^{1}H-NMR (400 MHz, D_2O, 16.7 °C): δ = 0.71 – 0.88 (m, 6H, -C$\underline{H}_3$, OleA), 1.06 – 1.31 (m, 40H, -C$\underline{H}_2$-, OleA), 1.31 - 1.92 (m, 10H, δ-C$\underline{H}_2$/γ-C$\underline{H}_2$/β-C$\underline{H}_2$, Lys; -C$\underline{H}_2$-CH=CH-C$\underline{H}_2$, O=C-CH_2-C$\underline{H}_2$-; OleA), 2.05 – 2.27 (m, 2H, O=C-C$\underline{H}_2$-, OleA), 2.40 - 2.69 (m, 16H, O=C-C$\underline{H}$2-, Stp), 2.81 – 3.14 (m, 8H, ε–C$\underline{H}_2$, Lys + Cys), 3.14 – 3.30 (m, 16H, N-C$\underline{H}_2$- , Stp), 3.30 – 3.68 (m, 48H, N-C$\underline{H}_2$-Stp), 4.04 – 4.21 (m, 2H, O=C-C(R)$\underline{H}$-NH-, Cys), 4.25 – 4.38 (m, 2H, O=C-C(R)$\underline{H}$-NH-, Lys)

Sequence: HO-C-Stp_3-K(K-$OleA_2$)-Stp_3-C #: **80**

Mass calculated: 2637.7 Found: 2638.3

NMR: ^{1}H-NMR (400 MHz, D_2O, 17.3 °C): δ = 0.74 – 0.86 (m, 6H, -C$\underline{H}_3$, OleA), 1.06 – 1.33 (m, 40H, -C$\underline{H}_2$-, OleA), 1.33 - 1.99 (m, 24H, δ-C$\underline{H}_2$/γ-C$\underline{H}_2$/β-C$\underline{H}_2$, Lys; -C$\underline{H}_2$-CH=CH-C$\underline{H}_2$, O=C-CH_2-C$\underline{H}_2$-; OleA), 2.07 – 2.30 (m, 4H, O=C-C$\underline{H}_2$-, OleA), 2.40 - 2.66 (m, 24H, O=C-C$\underline{H}_2$-, Stp), 2.83 – 3.14 (m, 8H, ε–C$\underline{H}_2$, Lys + Cys), 3.14 – 3.30 (m, 24H, N-C$\underline{H}_2$- , Stp), 3.30 – 3.65 (m, 72H, N-C$\underline{H}_2$-, Stp) 4.04 – 4.14 (m, 2H, O=C-C(R)$\underline{H}$-NH-, Cys), 4.29 – 4.41 (m, 2H, O=C-C(R)$\underline{H}$-NH-, Lys)

Sequence: HO-C-Stp_4-K(K-$OleA_2$)-Stp_4-C #: **84**

NMR: ^{1}H-NMR (400 MHz, D_2O, 17.3 °C): δ = 0.73 – 0.87 (m, 6H, -C$\underline{H}_3$, OleA), 1.10 – 1.36 (m, 40H, -C$\underline{H}_2$-, OleA), 1.36 - 1.99 (m, 24H, δ-C$\underline{H}_2$/γ-C$\underline{H}_2$/β-C$\underline{H}_2$, Lys; -C$\underline{H}_2$-CH=CH-C$\underline{H}_2$, O=C-CH_2-C$\underline{H}_2$-; OleA), 2.07 – 2.29 (m, 4H, O=C-C$\underline{H}_2$-, OleA), 2.40 - 2.70 (m, 32H, O=C-C$\underline{H}_2$-, Stp), 2.72 – 3.13 (m, 8H, ε–C$\underline{H}_2$, Lys + Cys), 3.14 – 3.30 (m, 32H, N-C$\underline{H}_2$- , Stp), 3.30 – 3.65 (m, 96H, N-

C$\underline{H}_2$-, Stp) 4.01 – 4.21 (m, 2H, O=C-C(R)$\underline{H}$-NH-, Cys), 4.21 – 4.40 (m, 2H, O=C-C(R)$\underline{H}$-NH-, Lys)

Sequence: HO-C-Stp$_1$-K(K)-Stp$_1$-C #: **74**
Mass calculated: 1023.4 Found: 1024.5
NMR: ^{1}H-NMR (400 MHz, D_2O, 17.0 °C): δ = 1.37 - 1.97 (m, 12H, δ-C$\underline{H}_2$/γ-C$\underline{H}_2$/β-C$\underline{H}_2$, Lys), 2.41 - 2.64 (m, 8H, O=C-C$\underline{H}$2-, Stp), 2.84 – 3.11 (m, 8H, ε–C$\underline{H}_2$, Lys + Cys), 3.17 – 3.29 (m, 8H, N-C$\underline{H}_2$-, Stp), 3.35 – 3.70 (m, 24H, Stp) 4.10 – 4.17 (m, 2H, O=C-C(R)$\underline{H}$-NH-, Cys), 4.38 – 4.48 (m, 2H, O=C-C(R)$\underline{H}$-NH-, Lys)

Sequence: HO-C-Stp$_3$-K(K)-Stp$_3$-C #: **78**
Mass calculated: 2108.8 Found: 2108.4
NMR: ^{1}H-NMR (400 MHz, D_2O, 17.0 °C): δ = 1.28 - 1.92 (m, 12H, δ-C$\underline{H}_2$/γ-C$\underline{H}_2$/β-C$\underline{H}_2$, Lys), 2.41 - 2.66 (m, 24H, O=C-C$\underline{H}$2-, Stp), 2.84 – 3.11 (m, 8H, ε–C$\underline{H}_2$, Lys + Cys), 3.17 – 3.29 (m, 24H, N-C$\underline{H}_2$-, Stp), 3.35 – 3.70 (m, 72H, Stp) 4.10 – 4.20 (m, 2H, O=C-C(R)$\underline{H}$-NH-, Cys), 4.38 – 4.48 (m, 2H, O=C-C(R)$\underline{H}$-NH-, Lys)

Sequence: HO-C-Stp$_4$-K(K)-Stp$_4$-C #: **82**
NMR: ^{1}H-NMR (400 MHz, D_2O, 17.0 °C): δ = 1.27 - 1.90 (m, 12H, δ-C$\underline{H}_2$/γ-C$\underline{H}_2$/β-C$\underline{H}_2$, Lys), 2.40 - 2.67 (m, 24H, O=C-C$\underline{H}$2-, Stp), 2.84 – 3.04 (m, 8H, ε–C$\underline{H}_2$, Lys + Cys), 3.10 – 3.27 (m, 32H, N-C$\underline{H}_2$-, Stp), 3.35 – 3.70 (m, 96H, Stp) 4.07 – 4.19 (m, 2H, O=C-C(R)$\underline{H}$-NH-, Cys), 4.28 – 4.38 (m, 2H, O=C-C(R)$\underline{H}$-NH-, Lys)