

Dimer 27 and Acid 28. According to the general procedure, **27** (5.3 mg, 68 %) and **28** (1.2 mg, 14 %) were obtained as white solids after lyophilization. **27**: R_f 0.18 (*i*-PrOH/1 M NH₄OAc, 2:1); ¹H NMR (400 MHz, D₂O) δ 5.08 (d, 2H, J = 3.9 Hz), 4.51 (d, 2H, J = 7.8 Hz), 4.50 (d, 2H, J = 8.1 Hz), 4.07 (dd, 2H, J = 3.1, 9.9 Hz), 4.05 (s, 4H), 3.99 (m, 2H), 3.94–3.79 (m, 20H), 3.76 (d, 2H, J = 3.2 Hz), 3.74–3.48 (m, 34H), 3.38–3.31 (m, 8H), 2.74 (dd, 2H, J = 4.5, 12.3 Hz), 2.21 (t, 4H, J = 7.4 Hz), 2.01 (s, 6H), 2.00 (s, 6H), 1.78 (t, 2H, J = 12.2 Hz), 1.59–1.50 (m, 8H), 1.32–1.24 (m, 4H), 1.15 (d, 6H, J = 6.7 Hz); ¹³C NMR (100 MHz, D₂O) δ 179.6, 177.5, 176.5, 176.4, 175.2, 104.0, 103.4, 102.1, 101.1, 78.1, 77.7, 77.34, 77.30, 75.8, 75.3, 74.34, 74.30, 72.8, 72.7, 72.1, 72.0, 71.9, 71.7, 71.6, 70.8, 70.5, 70.1, 69.7, 69.1, 65.0, 63.9, 62.1, 58.3, 54.1, 42.2, 40.84, 40.80, 38.2, 30.7, 27.4, 27.1, 24.7, 24.5, 17.7; ESIMS (H₂O, pos.) calcd for C₈₈H₁₅₀N₈O₅₄Na (M + Na⁺) *m/e* 2206, found 2206. **28**: R_f 0.30 (*i*-PrOH/1 M NH₄OAc, 2:1); ¹H NMR (400 MHz, D₂O) δ 5.08 (d, 1H, J = 3.9 Hz), 4.51 (d, 1H, J = 7.8 Hz), 4.50 (d, 1H, J = 8.0 Hz), 4.09–4.05 (m, 1H), 4.07 (s, 2H), 4.05 (s, 2H), 3.99 (m, 1H), 3.95–3.79 (m, 10H), 3.77–3.48 (m, 24H), 3.39–3.31 (m, 4H), 2.75 (dd, 1H, J = 4.6, 12.4 Hz), 2.21 (t, 2H, J = 7.5 Hz), 2.01 (s, 3H), 2.00 (s, 3H), 1.78 (t, 1H, J = 12.2 Hz), 1.60–1.50 (m, 4H), 1.32–1.24 (m, 2H), 1.15 (d, 3H, J = 6.7 Hz); ESIMS (H₂O, neg.) calcd for C₄₉H₈₃N₄O₃₁ (M – H⁺) *m/e* 1223, found 1223.

Dimer 29 and Acid 30. According to the general procedure, **29** (4.5 mg, 56 %) and **30** (1.8 mg, 20 %) were obtained as white solids after lyophilization. **29**: R_f 0.15 (*i*-PrOH/1 M NH₄OAc, 2:1); ¹H NMR (400 MHz, D₂O) δ 5.08 (d, 2H, J = 3.8 Hz), 4.51 (d, 2H, J = 7.8 Hz), 4.50 (d, 2H, J = 8.0 Hz), 4.07 (dd, 2H, J = 3.2, 9.9 Hz), 4.05 (s, 4H), 3.99 (m,

2H), 3.95–3.79 (m, 20H), 3.76 (d, 2H, $J = 3.0$ Hz), 3.72–3.48 (m, 38H), 3.38–3.31 (m, 8H), 2.75 (dd, 2H, $J = 4.7, 12.5$ Hz), 2.21 (t, 4H, $J = 7.3$ Hz), 2.02 (s, 6H), 2.00 (s, 6H), 1.78 (t, 2H, $J = 12.2$ Hz), 1.60–1.50 (m, 8H), 1.32–1.24 (m, 4H), 1.15 (d, 6H, $J = 6.6$ Hz); ^{13}C NMR (100 MHz, D_2O) δ 179.6, 177.5, 176.6, 176.4, 175.2, 104.1, 103.4, 102.1, 101.1, 78.1, 77.7, 77.35, 77.30, 75.8, 75.3, 74.34, 74.30, 72.8, 72.7, 72.1, 72.0, 71.9, 71.7, 71.6, 70.8, 70.5, 70.1, 69.7, 69.1, 65.0, 63.9, 62.1, 58.3, 54.1, 42.2, 40.9, 40.8, 38.2, 30.7, 27.4, 27.1, 24.7, 24.5, 17.7; ESIMS (H_2O , pos.) calcd for $^{12}\text{C}_{89}^{13}\text{C}_1\text{H}_{154}\text{N}_8\text{O}_{55}\text{Na}$ ($\text{M} + \text{Na}^+$) m/e 2251, found 2251. **30**: R_f 0.25 (*i*-PrOH/1 M NH_4OAc , 2:1); ^1H NMR (400 MHz, D_2O) δ 5.09 (d, 1H, $J = 3.9$ Hz), 4.51 (d, 1H, $J = 7.9$ Hz), 4.50 (d, 1H, $J = 8.0$ Hz), 4.09–4.05 (m, 1H), 4.07 (s, 2H), 4.05 (s, 2H), 3.99 (m, 1H), 3.95–3.80 (m, 10H), 3.77–3.49 (m, 28H), 3.39–3.31 (m, 4H), 2.75 (dd, 1H, $J = 4.7, 12.5$ Hz), 2.21 (t, 2H, $J = 7.6$ Hz), 2.02 (s, 3H), 2.00 (s, 3H), 1.78 (t, 1H, $J = 12.4$ Hz), 1.60–1.50 (m, 4H), 1.32–1.24 (m, 2H), 1.15 (d, 3H, $J = 6.6$ Hz); ESIMS (H_2O , neg.) calcd for $\text{C}_{51}\text{H}_{87}\text{N}_4\text{O}_{32}$ ($\text{M} - \text{H}^+$) m/e 1267, found 1267.

Dimer 31 and Acid 32. According to the general procedure, **31** (4.4 mg, 54 %) and **32** (1.9 mg, 20 %) were obtained as white solids after lyophilization. **31**: R_f 0.16 (*i*-PrOH/1 M NH_4OAc , 2:1); ^1H NMR (400 MHz, D_2O) δ 5.09 (d, 2H, $J = 4.0$ Hz), 4.51 (d, 2H, $J = 7.8$ Hz), 4.50 (d, 2H, $J = 8.2$ Hz), 4.07 (dd, 2H, $J = 3.1, 9.8$ Hz), 4.05 (s, 4H), 3.99 (dd, 2H, $J = 2.5, 12.5$ Hz), 3.95–3.79 (m, 20H), 3.76 (d, 2H, $J = 3.5$ Hz), 3.72–3.48 (m, 42H), 3.39–3.31 (m, 8H), 2.75 (dd, 2H, $J = 4.6, 12.4$ Hz), 2.21 (t, 4H, $J = 7.5$ Hz), 2.02 (s, 6H), 2.00 (s, 6H), 1.78 (t, 2H, $J = 12.2$ Hz), 1.60–1.50 (m, 8H), 1.33–1.25 (m, 4H), 1.15 (d, 6H, $J = 6.6$ Hz); ^{13}C NMR (100 MHz, D_2O) δ 179.6, 177.5, 176.6, 176.4, 175.2, 104.1, 103.4, 102.1, 101.1, 78.1, 77.7, 77.35, 77.31, 75.8, 75.4, 74.34, 74.30, 72.8, 72.7, 72.1,

72.0, 71.9, 71.7, 71.6, 70.8, 70.5, 70.2, 69.7, 69.1, 65.0, 63.9, 62.1, 58.3, 54.1, 42.2, 40.9, 40.8, 38.2, 30.7, 27.4, 27.1, 24.7, 24.5, 17.7; ESIMS (H₂O, pos.) calcd for ¹²C₉₁¹³C₁H₁₅₈N₈O₅₆Na (M + Na⁺) *m/e* 2295, found 2295. **32**: *R_f* 0.30 (*i*-PrOH/1 M NH₄OAc, 2:1); ¹H NMR (400 MHz, D₂O) δ 5.09 (d, 1H, *J* = 4.0 Hz), 4.51 (d, 1H, *J* = 7.8 Hz), 4.50 (d, 1H, *J* = 8.1 Hz), 4.07 (dd, 1H, *J* = 3.1, 9.8 Hz), 4.07 (s, 2H), 4.05 (s, 2H), 3.99 (m, 1H), 3.95–3.80 (m, 10H), 3.77–3.49 (m, 32H), 3.39–3.31 (m, 4H), 2.75 (dd, 1H, *J* = 4.6, 12.5 Hz), 2.21 (t, 2H, *J* = 7.5 Hz), 2.02 (s, 3H), 2.00 (s, 3H), 1.78 (t, 1H, *J* = 12.2 Hz), 1.60–1.50 (m, 4H), 1.33–1.24 (m, 2H), 1.15 (d, 3H, *J* = 6.6 Hz); ESIMS (H₂O, neg.) calcd for C₅₃H₉₁N₄O₃₃ (M – H⁺) *m/e* 1311, found 1311.

Dimer 33 and Acid 34. According to the general procedure, **33** (5.3 mg, 64 %) and **34** (2.7 mg, 28 %) were obtained as white solids after lyophilization. **33**: *R_f* 0.19 (*i*-PrOH/1 M NH₄OAc, 2:1); ¹H NMR (400 MHz, D₂O) δ 5.09 (d, 2H, *J* = 3.9 Hz), 4.51 (d, 2H, *J* = 7.8 Hz), 4.50 (d, 2H, *J* = 8.2 Hz), 4.07 (dd, 2H, *J* = 3.2, 9.8 Hz), 4.05 (s, 4H), 3.99 (m, 2H), 3.95–3.79 (m, 20H), 3.76 (d, 2H, *J* = 3.4 Hz), 3.72–3.48 (m, 46H), 3.39–3.31 (m, 8H), 2.75 (dd, 2H, *J* = 4.7, 12.5 Hz), 2.21 (t, 4H, *J* = 7.4 Hz), 2.02 (s, 6H), 2.00 (s, 6H), 1.78 (t, 2H, *J* = 12.2 Hz), 1.60–1.50 (m, 8H), 1.32–1.25 (m, 4H), 1.15 (d, 6H, *J* = 6.6 Hz); ¹³C NMR (100 MHz, D₂O) δ 179.6, 177.4, 176.5, 176.2, 175.2, 104.0, 103.4, 102.1, 101.1, 78.1, 77.7, 77.34, 77.30, 75.8, 75.3, 74.34, 74.30, 72.8, 72.7, 72.0, 71.9, 71.7, 71.6, 70.8, 70.5, 70.1, 69.7, 69.1, 65.0, 63.9, 62.1, 58.3, 54.1, 42.2, 40.9, 40.8, 38.2, 30.7, 27.4, 27.1, 24.7, 24.5, 17.7; ESIMS (H₂O, pos.) calcd for ¹²C₉₃¹³C₁H₁₆₂N₈O₅₇Na (M + Na⁺) *m/e* 2339, found 2339. **34**: *R_f* 0.27 (*i*-PrOH/1 M NH₄OAc, 2:1); ¹H NMR (400 MHz, D₂O) δ 5.08 (d, 1H, *J* = 3.9 Hz), 4.51 (d, 1H, *J* = 7.8 Hz), 4.50 (d, 1H, *J* = 8.0 Hz), 4.09–4.05 (m, 1H), 4.07 (s, 2H), 4.05 (s, 2H), 3.99 (m, 1H), 3.95–3.80 (m, 10H), 3.77–3.49 (m, 36H),

3.39–3.31 (m, 4H), 2.75 (dd, 1H, $J = 4.4, 12.2$ Hz), 2.21 (t, 2H, $J = 7.5$ Hz), 2.01 (s, 3H), 2.00 (s, 3H), 1.78 (t, 1H, $J = 12.2$ Hz), 1.60–1.50 (m, 4H), 1.33–1.24 (m, 2H), 1.15 (d, 3H, $J = 6.6$ Hz); ESIMS (H_2O , neg.) calcd for $\text{C}_{55}\text{H}_{95}\text{N}_4\text{O}_{34}$ ($\text{M} - \text{H}^+$) m/e 1355, found 1355.