Supplementary Data

Synthesis of bivalent neogalactolipids *via* modified Staudinger reaction

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General methods

Molecular sieves were activated at 180°C under diminished pressure for 2 h. DCM and all amines were purified and dried by distillation from CaH_2 immediately before the experiment. All solvents for a column chromatography were distilled before using. Thin layer chromatography was performed using pre-coated aluminum plates (Kieselgel 60 F_{254}, Merck), which were visualized with the phosphomolybdic acid – ceric sulfate reagent. Flash column chromatography (FC) was performed on Kieselgel 60 (40–63μm, Merck). ^1H and ^{13}C NMR spectra were recorded at 24°C in Bruker DPX-300, and Bruker Avance-600 spectrometers in $CDCl_3$ as a solvent unless otherwise stated. The signals of $SiMe_4$ (δ = 0.00 ppm) and $CDCl_3$ (δ = 77.16 ppm) were used as internal references**.** *J* values are given in Hz. Signals were assigned by 2D proton–proton (COSY) shift correlation spectra. Mass spectra were recorded in a Bruker Ultraflex time-of-flight mass spectrometer using 2,5-dihydroxybenzoic acid as a matrix.

[*rac*-2,3-Bis(tetradecyloxy)propyl] *N*-(7-aza-10-carboxy-8-oxodecyl)carbamate (3a)
Succinic anhydride (0.018 g, 0.195 mmol) was added to a solution of [*rac*-2,3-bis(tetradecyloxy)propyl] *N*-(6-aminohexyl)carbamate (2a) (0.111 g, 0.177 mmol) and Et_3N (0.062 mL, 0.444 mmol) in dry DCM (10 mL). The mixture was refluxed for 2 h, cooled to room temperature and washed with 3% aq. HCl (6 × 5 mL), water to pH 7.0. The organic extract was dried (Na_2SO_4), filtered, and concentrated under diminished pressure. The residue was purified by column chromatography on a silica gel (chloroform – methanol, 25:1) to give compound **3a** (0.087 g, 68%) as a colorless amorphous solid. δ_H (300 MHz; $CDCl_3$; Me_4Si): 0.87 (6 H, t, *J* 6.7, 2 $(CH_2)_{11}CH_3$), 1.11–1.39 (48 H, m, 2 $(CH_2)_{11}$, $(CH_2)_2$), 1.40-1.64 (8 H, m, 2 $NHCH_2CH_2$, 2 OCH_2CH_2), 2.40–2.61 (4 H, m, $C(O)(CH_2)_2C(O)$), 3.04–3.30 (4 H, m, 2 CH_2NH), 3.40-3.54 (6 H, m, 2 OCH_2, $CHCH_2O$), 3.57-3.63 (m, 1H, $CHCH_2O$), 4.07 (1 H, dd, *J* 5.3, 11.3) and 4.16 (1 H, dd, *J* 3.8, 11.3, $CH_2OC(O)$), 4.92-5.1 (1 H, m, NH), δ_c (75 MHz; $CDCl_3$): 14.21, 22.80, 26.18, 26.23, 29.29, 29.48, 29.63, 29.76, 29.78, 29.82, 30.14, 32.05, 39.50, 40.80, 47.37, 51.92, 64.65, 66.72, 70.59, 70.74, 71.93, 76.74, 77.03, 77.16, 77.58, 156.82. *m/z*: 749.540 (M^++Na, 100%).

(Cholest-5-en-3β-yl) *N*-(7-aza-10-carboxy-8-oxodecyl)carbamate (**3b**) was prepared at the same way as compound **3a** from compound **2b** (0.500 g, 0.945 mmol), succinic anhydride (0.189 g, 1.889 mmol) and Et_3N (0.329 mL, 2.364 mmol). After work-up the residue, was purified by column chromatography on a silica gel (chloroform – methanol, 10:1) to give compound **3b** (0.393 g, 66%) as a colorless amorphous solid. δ_H (300 MHz; $CDCl_3$; Me_4Si): 0.66 (3 H, s, C(13)Me Chol), 0.85 (3 H, d, *J* 6.5, C(25)Me Chol), 0.86 (3 H, d, *J* 6.5, C(25)Me Chol), 0.89 (3 H, d, *J* 6.5, C(20)Me Chol), 0.99 (3 H, s, C(10)Me Chol), 1.03-1.67 (28 H, m, Chol, $NHCH_2(CH_2)_4$), 1.72-2.06 (6 H, m, Chol), 2.17-2.38 (2 H, m, $C(4)H_2$ Chol), 2.39-2.53 (2 H, m,

$C\underline{H}_2COOH$), 2.55-2.73 (2 H, m, $C\underline{H}_2CH_2COOH$), 3.01-3.17 (2 H, m, $C\underline{H}_2NH$), 3.18-3.30 (2 H, m, $C\underline{H}_2NH$), 4.33-4.58 (1 H, m, 3-H Chol), 4.80-4.98 (1 H, m, N<u>H</u>), 5.27-5.41 (1 H, m, 6-H Chol), 6.24-6.60 (1 H, m, N<u>H</u>). δ_c (75 MHz; $CDCl_3$): 176.05, 172.76, 162.97, 156.67, 139.89, 122.62, 74.56, 56.78, 56.31, 50.11, 42.41, 40.57, 39.83, 39.62, 39.41, 38.65, 37.08, 36.65, 36.29, 35.90, 31.99, 31.10, 30.22, 29.77, 29.25, 29.10, 28.33, 28.25, 28.10, 26.04, 24.39, 23.95, 22.92, 22.66, 21.14, 19.43, 18.82, 11.96. *m/z*: 651.292 (M^++Na, 100%).

8-chloro-1-(2,3,4,6-tetra-*O*-acetyl-β-D-galactopyranosyloxy)-3,6-dioxaoctane (5)

$HgCN_2$ (2.247 g, 8.896 mmol) and 4 Å crushed molecular sieves were added to a solution of compound **4** (1.00g, 5.931 mmol) in dry DCM (15 mL) at 20°C under stirring. After 15 min, the solution of 2,3,4,6-tetra-*O*-acetyl-*α*-D-galactopyranosyl bromide (3.658 g, 8.896 mmol) in dry DCM (15 mL) was added dropwise within 1 h. After 4 h at 40 °C, the reaction mixture was cooled to ambient temperature, filtered through Celite 545® pad, and washed with 20% aq. KI (4 × 50 mL), water (3 × 30 mL). The organic layer was dried (Na_2SO_4), filtered, and concentrated under diminished pressure. The residue was purified by column chromatography on a silica gel (toluene – ethyl acetate, 2:1) to give compound **5** (2.078 g, 75%) as a yellowish oil. δ_H (300 MHz; $CDCl_3$; Me_4Si): 1.91 (3H, s), 1.98 (3H, s), 2.99 (3H, s), 2.08 (3H, s, 4 OCOMe), 3.50-3.64 (8 H, m, $C\underline{H}_2O(C\underline{H}_2)_2OC\underline{H}_2$), 3.65-3.75 (3 H, m, $C\underline{H}_2Cl$, $OC\underline{H}_aH$), 3.81-3.93 (2 H, m, 5-H Gal, $OC\underline{H}_bH$), 4.06 (1 H, dd, *J* 6.5, 11.1, 6-H_a Gal), 4.07 (1 H, dd, *J* 6.5, 11.1, 6-H_b Gal), 4.52 (1 H, d, *J* 8.0, 1-H Gal), 4.95 (1 H, dd, *J* 3.4, 10.5, 3-H Gal), 5.14 (1 H, dd, *J* 8.0, 10.5, 2-H Gal), 5.32 (1 H, dd, *J* 1.0, 3.4, 4-H Gal). δ_c (75 MHz; $CDCl_3$): 20.56, 20.64, 20.66, 20.76, 42.78, 61.25, 67.01, 68.73, 69.02, 70.33, 70.55, 70.58, 70.61, 70.83, 101.25, 169.46, 170.12, 170.24, 170.36. *m/z*: 521.893 (M^++Na, 100%).

8-azido-1-(2,3,4,6-tetra-*O*-acetyl-β-D-galactopyranosyloxy)-3,6-dioxaoctane (6)

Sodium azide (0.542 g, 8.329 mmol) was added to a solution of compound **5** (2.078 g, 4.164 mmol) in dry DMF (100 mL) and stirred for 40 h at 100º C. The solvent was removed under diminished pressure, the residue was dissolved in DCM (70 mL) and washed by 3% aq. HCl (4 × 20 mL) and water (3 × 20 mL). The organic layer was dried (Na_2SO_4), filtered, the solvent was removed under diminished pressure. Column chromatography on silica gel (toluene – ethylacetate, 1:2) gave compound **6** (1,747 g, 83%) as a yellowish oil. δ_H (300 MHz; $CDCl_3$; Me_4Si): 1.92 (3H, s), 1.98 (3H, s), 2.00 (3H, s), 2.08 (3H, s, 4 OCOMe), 3.35 (2 H, t, *J* 5.0, CH_2N_3), 3.40-3.64 (8 H, m, $C\underline{H}_2O(C\underline{H}_2)_2OC\underline{H}_2$), 3.65-3.75 (1 H, m, $OC\underline{H}_aH$), 3.81-3.93 (2 H, m, 5-H Gal, $OC\underline{H}_bH$), 4.06 (1 H, dd, *J* 6.6, 11.1, 6-H_a Gal), 4.11 (1 H, dd, *J* 6.5, 11.1, 6-H_b Gal), 4.51 (1 H, d, *J* 8.0, 1-H Gal), 4.95 (1 H, dd, *J* 3.4, 10.5, 3-H Gal), 5.15 (1 H, dd, *J* 8,0, 10.5, 2-H Gal), 5.32 (1 H, dd, *J* 1.0, 3.4, 4-H Gal). δ_c (75 MHz; $CDCl_3$): 20.72, 20.81, 20.90, 50.77, 61.39, 67.14, 68.89, 69.17, 70.12, 70.51, 70.73, 70.78, 70.82, 71.00, 101.45, 169.63, 170.31, 170.41. *m/z*: 528.031 (M^++Na, 100%).

8-amino-1-(2,3,4,6-tetra-*O*-acetyl-β-D-galactopyranosyloxy)-3,6-dioxaoctane (7)

A catalytic amount of 10% Pd/C was added to a solution of compound **6** (0.721 g, 1.978 mmol) and ammonium formate (0.500 g, 7.913 mmol) in methanol (10 mL) heated to 60 °C. After 15 min the catalyst was filtered off and methanol was removed under diminished pressure. Column chromatography on a silica gel (DCM – methanol, 10:1) gave compound **7** (0.308 g, 45%) as a colorless oil. δ_H (300 MHz; $CDCl_3$; Me_4Si): 1.98 (3H, s), 2.05 (3H, s), 2.08 (3H, s), 2.18 (3H, s, 4 OCOMe), 3.15-3.26 (2 H, m, $C\underline{H}_2NH_2$), 3.49-3.70 (8 H, m, $C\underline{H}_2O(C\underline{H}_2)_2OC\underline{H}_2$), 3.73-4.04 (3 H, m, $OC\underline{H}_2$, 5-H- Gal), 4.05-4.22 (2 H, m, 6-H Gal), 4.55 (1 H, d, *J* 7.9, 1-H Gal), 5.03 (1 H, dd, *J* 3.3, 10.5, 3-H Gal), 5.16 (1 H, dd, *J* 7.9, 10.5, 2-H Gal), 5.38 (1 H, dd, *J* 0.8, 3.3, 4-H Gal), 5.90-6.70 (2 H, m, $N\underline{H}_2$). δ_c (75 MHz; $CDCl_3$): 20.74, 20.84, 20.86, 21.03, 39.95, 61.33, 66.83, 67.11, 68.95, 69.19, 70.10, 70.21, 70.47, 70.81, 70.83, 101.38, 170.14, 170.30, 170.42, 170.63. *m/z*: 480.400 (M^+ + H, 100%)

1,5-Bis{*N*-[8-(2,3,4,6-tetra-*O*-acetyl-β-D-galactopyranosyloxy)-3,6-dioxaoctyl]}-*N*-*tert*-butyloxycarbonyl-L-glutaminamid (8)

A solution of Boc-L-glutamic acid (0.074 g, 0.302 mmol) and HOBt (0.090 g, 0.664 mmol) in anhydrous THF (5 mL) was added under the argon atmosphere to a solution of azide **6** (0.335 g, 0.664 mmol) in anhydrous THF (15mL). After stirring for 15 min the solution was cooled to 0 C and DIC (0,104 mL, 0.664 mmol) was added and the mixture was additionally stirred for 15 min, then tributylphosphine (0.302 mL, 1.208 mmol) was added. After 1 h the reaction mixture was heated to 22°C and stirred for 48 h. The reaction was quenched with water (30 mL) and after stirring for 20 min the products were extracted with ethyl acetate (4 × 70 mL), the obtained organic extract was dried (Na_2SO_4), filtered and the solvent was removed under diminished pressure. Column chromatography on silica gel ($CHCl_3$ – MeOH, 60:1) gave compound **8** (0.304 g, 86%) as a colorless crystallizing oil. δ_H (300 MHz; $CDCl_3$; Me_4Si): 1.36 (9 H, s, CMe_3), 1.85-2.00 (22 H, m, 6 OCOMe, $(C\underline{H}_2)_2$ Glu), 2.08 (6 H, s, 2 OCOMe), 3.25-3.79 (24 H, m, 2 $C\underline{H}_2NH$, 2 $C\underline{H}_2O(C\underline{H}_2)_2OC\underline{H}_2$, 2 $OC\underline{H}_2$), 3.82-3.96 (2 H, m, 2 × 5-H Gal), 4.02-4.16 (5 H, m, 2 × 6-H Gal, $C\underline{H}NH$ Glu), 4.54 (2 H, d, *J* 7.9, 2 × 1-H Gal), 4.95 (2 H, dd, *J* 3.4, 10.5, 2 × 3-H Gal), 5.15 (2 H, dd, *J* 7.9, 10.5, 2 × 2-H Gal), 5.33 (2 H, dd, *J* 0.8, 3.4, 2 × 4-H Gal), 5.53-5.67 (1 H, m, $N\underline{H}$), 6.50-6.80 (1 H, m, $N\underline{H}$), 6.95-7.16 (1 H, m, $N\underline{H}$). δ_c (75 MHz; $CDCl_3$): 20.74, 20.82, 20.93, 28.44, 36.63, 37.85, 38.73, 39.39, 53.92, 61.36, 67.07, 68.88, 69.72, 69.33, 69.73, 70.23, 70.36, 70.63, 70.69, 70.74, 70.79, 70,90, 70.94, 101.45, 161.58, 170.32. *m/z*: 1192.251 (M^++Na, 100%)

1,5-Bis{*N*-[8-(2,3,4,6-tetra-*O*-acetyl-β-D-galactopyranosyloxy)-3,6-dioxaoctyl]}-L-glutaminamid trifluoroacetate(9)

Anhydrous TFA (1.05 mL, 12.768 mmol) was added to a solution of compound **8** (0.304 g, 0.319 mmol) in DCM (5 mL). The reaction mixture was stirred for 2 h at 24 °C, and then DCM was removed under diminished pressure. The residue was purified by column chromatography on silica gel ($CHCl_3$ – MeOH – 1% aq. CH_3COOH, 14:1:0.02) to give compound **9** (0.212 g, 72%) as a colorless crystallizing oil. δ_H (300 MHz; $CDCl_3$; Me_4Si): 1.85-2.08 (28 H, m, 8 OCOMe, $(C\underline{H}_2)_2$ Glu), 3.25-3.79 (22 H, m, 2 $C\underline{H}_2NH$, 2 $C\underline{H}_2O(C\underline{H}_2)_2OC\underline{H}_2$, 2 $OC\underline{H}_aH$), 3.77-3.96 (4 H, m, 2 × 5-H Gal, 2 $OC\underline{H}_bH$), 4.00-4.15 (5 H, m, 2 × 6-H Gal, $C\underline{H}NH_2$), 4.50 (2 H, d, *J* 7.9, 2 × 1-H Gal), 4.93 (2 H, dd, *J* 3.4, 10.5, 2 × 3-H Gal), 5.06 (2 H, dd, *J* 7.9, 10.5, 2 × 2-H Gal), 5.28 (2 H, dd, *J* 0.8, 3.4, 2 × 4-H Gal). δ_C (75 MHz; $CDCl_3$): 12.40, 17.58, 20.42, 20.46, 20.51, 20.61, 25.04, 29.61, 31.57, 38.96, 42.86, 51.87, 54.79, 61.36, 67.23, 67.61, 68.93, 68.97, 69.92, 70.23, 70.50, 70.56, 70.95, 101.17, 169.94, 170.44, 170.61, 170.80, 172.04, 174.16, 174.56. m/z: 1070.380 (M^++Na, 100%)

1,5-Bis{*N*-[8-(2,3,4,6-tetra-O-acetyl-β-D-galactopyranosyloxy)-3,6-dioxaoctyl]-*N*-[5,12-diaza-1,4,13-trioxo-13-(*rac*-2,3-di(tetradecyloxy)prop-1-yloxy)tridecyl]-L-glutaminamid (10a)

A solution of compound **9** (0.138 g, 0.109 mmol) and DIPEA (56 µL, 0.326 mmol) in dry DMF (4 mL) was stirred at 0 °C for 15 min, then a solution of compound **3a** (0.119 g, 0.163 mmol) and HBTU (0.124 g, 0.326 mmol) in dry DMF (4 mL) was added dropwise within 10 min. After 72 h at 24 °C, DMF was removed under diminished pressure, the residue was dissolved in chloroform (25 mL), washed with 3% aq. HCl (2 × 10 mL), water to pH 7, dried (Na_2SO_4), filtered and the solvent was removed under diminished pressure. Column chromatography on silica gel ($CHCl_3$ – MeOH, 40:1) gave compound **10a** (0.155 g, 80%) as a colorless crystallizing oil. δ_H (300 MHz; $CDCl_3$; Me_4Si): 0.86 (6 H, t, *J* 6.7, 2 $(CH_2)_{11}C\underline{H}_3$), 1.14-1.40 (44 H, m, 2 $(C\underline{H}_2)_{11}$, $C\underline{H}_2C\underline{H}_2$), 1.42-1.62 (8 H, m, $C\underline{H}_2(CH_2)_{11}$, 2 $NHCH_2C\underline{H}_2$), 1.92-2.19 (26 H, m, 8 OCOMe, $C\underline{H}_2$ Glu), 2.20-2.40 (2 H, m, $C\underline{H}_2$ Glu), 2.45-2.63 (4 H, m, $C(O)(C\underline{H}_2)_2C(O)$), 3.10-3.26 (4 H, m, 2 $NHC\underline{H}_2$), 3.33-3.49 (8 H, m, 2 $C\underline{H}_2NH$, $OC\underline{H}_2$, $CHC\underline{H}_2O$), 3.50-3.78 (22 H, m, 2 $C\underline{H}_2O(C\underline{H}_2)_2OC\underline{H}_2$, $OC\underline{H}_2CH_2(CH_2)_{11}CH_3$, $C\underline{H}$), 3.88-4.00 (2 H, m, 2 × 5-H Gal), 4.01-4.21 (6 H, m, 2 × 6-H Gal, $CHC\underline{H}_2OC(O)$), 4.30-4.43 (1 H, m, $N\underline{H}$), 4.54 (1 H, d, *J* 7.9, 1-H Gal), 4.57 (1 H, d, *J* 7.9, 1-H Gal), 4.81-4.94 (1 H, m, $N\underline{H}$), 5.02 (2 H, dd, *J* 3.4, 10.5, 2 × 3-H Gal), 5.17 (2

H, dd, *J* 7.9, 10.5, 2 × 2-H Gal), 5.37 (2 H, dd, *J* 0.8, 3.4, 2 × 4-H Gal), 6.05-6.16 (1 H, m, NH), 6.67-6.78 (1 H, m, NH). δ_H (75 MHz; $CDCl_3$): 14.22, 20.70, 20.76, 20.79, 20.90, 22.79, 26.15, 26.21, 26.34, 26.47, 29.50, 29.61, 29.73, 29.76, 29.80, 29.93, 30.13, 31.46, 31.50, 31.58, 31.62, 31.63, 31.66, 32.02, 32.55, 39.23, 39.35, 39.46, 40.91, 52.95, 53.02, 53.04, 61.36, 61.39, 64.32, 67.17, 67.20, 68.97, 69.24, 69.25, 69.65, 69.81, 70.25, 70.29, 70.32, 70.57, 70.65, 70.71, 70.75, 70.78, 70.80, 70.97, 70.98, 71.89, 77.36, 101.45, 101.46, 125.40, 128.33, 129.14, 156.61, 169.71, 169.72, 170.27, 170.28, 170.35, 170.37, 170.55, 170.58, 171.48, 171.54, 171.56, 172.15, 172.46, 172.53, 173.21. m/z: 1801.946 (M^++Na, 100%)

1,5-Bis{*N*-[8-(2,3,4,6-tetra-*O*-acetyl-β-D-galactopyranosyloxy)-3,6-dioxaoctyl]-*N*-[5,12-diaza-13-(cholest-5-ene-3β-yloxy)-1,4,13-trioxotridecyl]-L-glutaminamid (10b) was prepared at the same way as compound **10a** from compound **9** (0.100 g, 0.088 mmol), DIPEA (46 μL, 0.266 mmol), compound **3b** (0.084 g, 0.133 mmol) and HBTU (0.101 g, 0.266 mmol). Column chromatography on silica gel ($CHCl_3$ – MeOH, 30:1) gave compound **10a** (0.101 g, 72%) as a colorless crystallizing oil. δ_H (300 MHz; $CDCl_3$; Me_4Si): 0.61 (3 H, s, C(13)Me Chol), 0.78 (3 H, d, *J* 6.6, C(25)Me Chol), 0.81 (3 H, d, *J* 6.6, C(25)Me Chol), 0.84 (3 H, d, *J* 6.5, C(20)Me Chol), 0.89-1.60 (35 H, m, 27 H Chol, $(CH_2)_4$), 1.62-1.84 (2 H, m, CH_2 Glu), 1.89-2.08 (24 H, m, 8 OCOMe), 2.12-2.36 (5 H, m, CH_2 Glu, Chol), 2.40-2.48 (4 H, m, $C(O)(CH_2)_2C(O)$), 2.97-3.23 (4 H, m, 2 CH_2NH), 3.25-3.43 (4 H, m, 2 CH_2NH), 3.43-3.61 (16 H, m, 2 $CH_2O(CH_2)_2OCH_2$), 3.62-3.74 (2 H, m, 2 OCH_aH), 3.80-3.96 (4 H, m, 2 × 5-H Gal, 2 OCH_bH), 3.99-4.18 (5 H, m, 2 × 6-H Gal, CHNH Glu), 4.24-4.45 (2 H, m, 3-H Chol, NH), 4.48 (1 H, d, *J* 7.9, 1-H Gal), 4.52 (1 H, d, *J* 7.9, 1-H Gal), 4.68-4.81 (1 H, m, NH), 4.97 (2 H, dd, *J* 3.4, 10.5, 2 × 3-H Gal), 5.12 (2 H, dd, *J* 7.9, 10.5, 2 × 2-H Gal), 5.32 (3 H, m, 2 × 4-H Gal, 6-H Chol), 6.05-6.15 (1 H, m, NH), 6.60-6.70 (1 H, m, NH). δ_C (75 MHz; $CDCl_3$): 11.87, 18.72, 19.34, 20.61, 20.68, 20.70, 20.81, 21.05, 22.57, 22.83, 23.84, 24.30, 26.21, 26.34, 28.01, 28.23, 29.39, 29.70, 29.87, 31.43, 31.59, 31.89, 32.48, 35.80, 36.19, 36.58, 37.01, 38.61, 39.34, 39.52, 39.75, 40.63, 42.32, 50.02, 52.95, 56.15, 56.70, 61.30, 67.11, 68.87, 69.15, 69.53, 69.70, 70.16, 70.24, 70.68, 70.88, 74.23, 77.27, 101.36, 122.48, 139.87, 169.60, 170.17, 170.27, 170.44, 171.44, 172.07, 172.43, 173.08. m/z: 1702.798 (M^++Na, 100%)

1,5-Bis-[*N*-[8-(β-D-galactopiranosyloxy)-3,6-dioxaoctyl]-*N*-[5,12-diaza-1,4,13-trioxo-13-(*rac*-2,3-di(tetradecyloxy)prop-1-yloxy)tridecyl]-L-glutaminamid (1a)
A 0.04 M solution of MeONa in MeOH (0.5 mL) was added to a solution of compound **10a** (99 mg, 0.056 mmol) in MeOH (5 mL). After 1 h the reaction mixture was neutralized with 3% aq. HCL (40μl) and the solvent was removed under diminished pressure. Column chromatography on silica gel ($CHCl_3$ – MeOH – 1% aq. CH_3COOH, 3:1:0.08) gave compound **1a** (73 mg, 92%) as a white amorphous solid. δ_H (600 MHz; $CDCl_3$:CD_3OD 1:1; Me_4Si): 0.80 (6 H, t, *J* 7.1, 2 $(CH_2)_{11}CH_3$), 1.12-1.32 (48 H, m, 2 $(CH_2)_{11}$, CH_2CH_2), 1.36-1.51 (8 H, m, 2 $CH_2(CH_2)_{11}$, 2 $NHCH_2CH_2$), 1.80-1.88 (1 H, m) and 2.00-2.08 (1 H, m, CH_2 Glu), 2.23 (2 H, t, *J* 7.6, CH_2 Glu), 2.39-2.51 (4 H, m, $C(O)(CH_2)_2C(O)$), 3.01 (2 H, t, *J* 7.2, CH_2NHCO), 3.07 (2 H, t, *J* 7.2, CH_2NHCO), 3.29-3.75 (39 H, m, 2 $CH_2O(CH_2)_2OCH_2$, 2 × 2-H Gal, 2 × 3-H Gal, 2 × 5-H Gal, 2 × 6-H Gal, 2 OCH_aH, 2 OCH_2CH_2NH, 2 CH_2O, CH_2CH Gro), 3.80-3.85 (2 H, m, 2 × 4-H Gal), 3.94-4.00 (3 H, m, 2 OCH_bH, OCH_aH Gro), 4.03 (1 H, dd, *J* 11.4, *J* 4.5, OCH_bH Gro), 4.22 (1 H, d, *J* 7.7, 1-H Gal), 4.23 (1 H, d, *J* 7.7, 1-H Gal), 4.25 (1 H, dd, *J* 9.2, *J* 5.0, CHNH Glu). δ_c (75 MHz; $CDCl_3$:CD_3OD 1:1): 14.22, 18.05, 23.16, 23.78, 26.55, 26.62, 26.94, 27.10, 28.47, 29.64, 29.71, 29.78, 29.87, 29.99, 30.16, 30.19, 30.37, 30.48, 31.45, 31.62, 31.73, 32.45, 39.77, 39.91, 41.22, 53.81, 61.84, 64.51, 68.82, 69.64, 69.67, 70.33, 70.42, 70.56, 70.60, 70.66, 70.93, 71.14, 71.78, 71.85, 72.26, 74.07, 75.87, 75.89, 78.71, 103.85, 103.91, 157.97, 173.73, 173.76, 174.41, 174.71, 179.89. m/z: 1465.085 (M^++Na, 100%).

1,5-Bis-[*N*-[8-(β-D-galactopiranosyloxy)-3.6-dioxaoctyl]-*N*-[5,12-diaza-13-(cholest-5-ene-3β-yloxy)-1,4,13-trioxotridecyl]-L-glutaminamid (1b) was prepared at the same way as

compound **1a** from 10b (86 mg, 0.051 mmol). Column chromatography on silica gel ($CHCl_3$ – MeOH – $CH3COOH_{aq}$(1%), 1:8:0.08) gave compound **1b** (60 mg, 87%). δ_H (600 MHz; $CDCl_3$:CD_3OD 1:1; Me_4Si): 0.61 (3 H, s, C(13)Me Chol), 0.78 (3 H, d, *J* 6.6, 2 C(25)Me Chol), 0.84 (3 H, d, *J* 6.5, C(20)Me Chol), 0.92 (3 H, s, C(10)Me Chol), 0.85-1.56 (34 H, m, Chol, $(C\underline{H}_2)_4$), 1.69-1.81 (3 H, m, Chol), 1.82-1.92 (1 H, m) and 1.98-2.08 (1 H, m, $C\underline{H}_2$ Glu), 2.15-2.30 (4 H, m, $C\underline{H}_2$ Glu, 4-$C\underline{H}_2$ Chol), 2.37-2.51 (4 H, m, $C(O)(C\underline{H}_2)_2C(O)$), 3.01 (2 H, t, *J* 7.2, $C\underline{H}_2NHCO$) 3.06 (2 H, t, *J* 7.2, $C\underline{H}_2NHCO$), 3.28-3.40 (4 H, m, 2 $OCH_2C\underline{H}_2NH$), 3.41-3.79 (29 H, m, 2 $C\underline{H}_2O(C\underline{H}_2)_2OC\underline{H}_2$, 2 × 2-H Gal, 2 × 3-H Gal, 2 × 5-H Gal, 2 × 6-H Gal, 2 $OC\underline{H}_aH$, 3-H Chol), 3.82-3.87 (2 H, m, 2 × 4-H Gal), 3.97-4.04 (2 H, m, 2 $OC\underline{H}_bH$), 4.22 (1 H, d, *J* 7.6, 1-H Gal), 4.23 (1 H, d, *J* 7.6, 1-H Gal), 4.26 (1 H, dd, *J* 9.2, *J* 5.0, $C\underline{H}NH$ Glu), 5.26-5.32 (1 H, m, 6-H Chol). δ_c (75 MHz; $CDCl_3$:CD_3OD 1:1): 11.49, 18.34, 18.94, 20.79, 22.12, 22.38, 23.47, 23.55, 24.00, 26.09, 26.25, 27.73, 27.93, 28.86, 29.43, 30.79, 31.01, 31.65, 32.01, 35.55, 35.93, 36.32, 36.77, 38.33, 38.94, 38.99, 39.12, 39.26, 39.52, 40.29, 42.07, 47.58, 47.86, 48.15, 48.43, 48.72, 49.00, 49.28, 49.87, 52.99, 55.92, 56.50, 60.96, 61.02, 67.99, 68.72, 68.79, 69.44, 69.55, 69.71, 69.77, 69.81, 69.87, 69.97, 70.90, 71.00, 73.11, 74.17, 102.95, 103.02, 122.22, 139.62, 156.80, 172.85, 163.46, 173.84, 179.18. m/z: 1366.690 (M^++Na, 100%).

Lectin-induced agglutination of galactosylated liposomes

Cationic liposomes (100 µL, 1 mM of phospholipid) were diluted in 1.8 ml of 150 mM NaCl followed by incubation with 100 µL of RCA_{120} (1 mg/mL). After rapid mixing the agglutination of the liposomes was estimated at room temperature by the time dependent increase in turbidity, as measured by the absorbance at 450 nm with Helios Alpha spectrometer (Thermo Spectronic).