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Original article

Synthesis of amphiphilic galactopyranosyl diamines and amino alcohols as antitubercular agents

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Abstract

Mono- and diacylated derivatives of galactopyranosyl amines were obtained from D-galactose, via aminated intermediates prepared by reaction of 6-deoxy-6-iodo-1,2:3,4-di-*O*-isopropylidene-α-D-galactopyranose with 1,3-propanediamine, 1,2-ethanediamine or ethanolamine. Monoacylated derivatives displayed antitubercular activity.

Keywords: D-Galactose; Surfactant; *N*-Alkyl-diamine; *N*-Alkyl-ethanolamine; Tuberculosis

1. Introduction

Carbohydrate-derived surfactants are non-ionic amphiphilic compounds in which the carbohydrate moiety is linked to a long alkyl chain. In recent years, this class of compounds has been extensively studied [1–10] due to several interesting properties: they are non-toxic, biodegradable, and they can be used in several areas such as food industry, biology, glycobiology, or as detergents [11–17]. Another class of surfactants are *N*-acylated amines derived from fatty acids, which have been shown to have antimicrobial activity [18]. Furthermore, glycosyl amines possess anti-infective, immunomodulatory, anti-cancer, anti-inflammatory, antiviral and antifungal activities [19–23]. Tripathi and co-workers [24–27] synthesized glycosyl amino alcohols (Fig. 1, compounds **1a** and **1b**) having alkyl chains of varying lengths, as a new class of antitubercular and antifungal agents. Compounds **1a** [26] were found to be, in vitro, superior to the amino alcohol ethambutol, a clinically used antitubercular drug. Tuberculosis is caused by an intracellular pathogen, *M. tuberculosis*, whose cell wall possesses a series of complex polysaccharides containing several unique monosaccharides in defined linkages [28,29]. Galactose and arabinose are predominant in these superstructures in the form of lipoarabinomannan and arabinogalactan. These carbohydrates are critical for cell wall integrity, and any alteration could lead to a disturbance in cell wall biosynthesis. The enzymes responsible for the synthesis of the extracellular polysaccharides are glycosyltransferases, inhibited by clinical antitubercular drugs such as ethambutol [30].

We report in this work the preparation and biological evaluation of some aminoacylated derivatives of galactopyranose.

2. Results and discussion

D-Galactose **2** was converted into 6-deoxy-6-iodo-1,2:3,4-di-*O*-isopropylidene-α-D-galactopyranose **3** according to the literature procedure [31]. The reactive halogen of compound **3** may be readily displaced by an amino group in a nucleophilic substitution reaction. Thus treatment of **3** with 1,3-propanediamine, 1,2-ethanediamine or ethanolamine, in ethanol for 72 h

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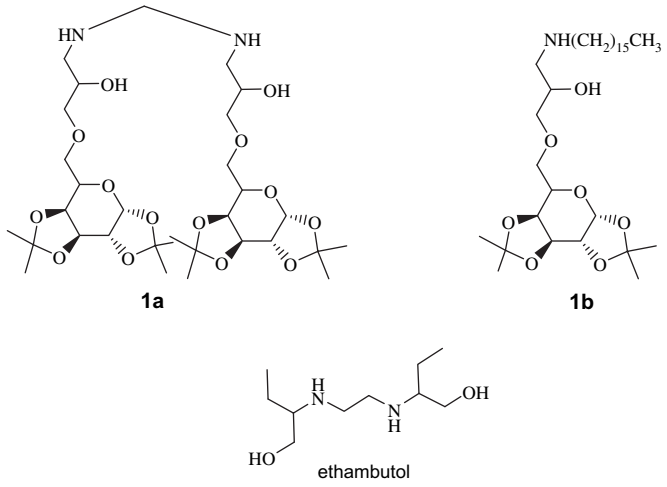
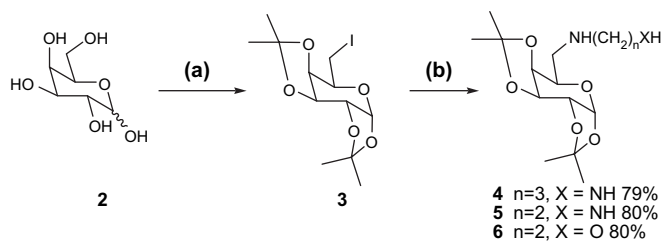


Fig. 1. Structures of compounds **1a, 1b** and ethambutol.

under reflux, gave the corresponding amines **4**, **5** and **6** in 79–80% yield (Scheme 1).

The three aminated compounds were treated with 2 equiv./mol of palmitoyl chloride or stearoyl chloride, in pyridine at 0 °C, leading to diacylated compounds **7**–**12** in 67–75% yield (Scheme 2). COSY, NOESY and HSQC NMR experiments were used to characterize these compounds. The ^{1}H NMR analysis revealed a duplicity of the signals, which was attributed to the presence of rotamers, often observed in trisubstituted nitrogen compounds [32,33]. This was confirmed by acquiring the NMR spectra of compound **6** by varying the temperature between 300 and 337 K, at which temperature the coalescence of the signals was observed. Regioselective monoacylation of the aminated compounds **4** and **5** was achieved using 1.04 equiv./mol of stearoyl chloride (Scheme 2). The acylation occurred mainly at the sterically less hindered primary amino group, giving monoamides **13** and **14** in 50% yield, along with 15% of diacylated compounds and unreacted material. Protected galactopyranosyl amino alcohol **6** was mainly converted to amide **15** (50% yield), along with 20% of the diacylated compound. The site of the acylation was established by ^{1}H NMR experiments, showing two different signals for NH protons for compounds **13** and **14**, one of them attributed to the amide at δ 6.83 and 6.60, respectively. The ^{1}H NMR spectra of compound **15** did not show any signal for NH, and there is no ester band in its infrared spectra, confirming the monoacylation of the amino group.

Deprotection of the isopropylidene groups of all acylated compounds was achieved using BF_3–OMe_2 in a mixture of dichloromethane/acetonitrile at room temperature (Scheme 3), leading to compounds **16**–**24** in 86–91% yield.



(a) i- $ZnCl_2$, H_2SO_4, acetone, 75%; ii- I_2, PPh_3, imidazole, toluene, reflux, 78%;
(b) $H_2N(CH_2)_nXH$, EtOH, reflux.

Scheme 1. Synthesis of galactopyranosyl diamines and amino alcohol **4, 5** and **6**.

2.1. Antitubercular activity

The activity of the compounds against *M. tuberculosis* virulent strain H37Rv was determined in vitro using rifampicin as a reference for activity. The minimum inhibitory concentration (MIC), concentration that inhibits the colony forming ability of *M. tuberculosis*, was determined by incorporating decreasing concentrations of the test compound in Middlebrook 7H9 agar medium. The results are reported in Table 1. *N*-Acylated compounds **13** and **15** inhibited growth at concentrations of 12.5 and 25 g/mL, respectively, whereas compound **14** inhibited growth at a concentration of 3.12 g/mL. Diacylated compounds **7**, **9**, **12**, **17** and **19** did not show antitubercular activity in the concentration range studied.

It would appear that the antitubercular activity of these compounds depends on the availability of either an amino group or a hydroxyl group, which can interact with acidic residues in the catalytic active site, as previously suggested [34]. The best result was obtained with the aminoethyl derivative **14** (MIC 3.12 μg/mL). None of the unprotected compounds tested showed antitubercular activity, suggesting that hydrophobicity may play an important role for glycosyltransferase inhibition. Other studies are currently underway in our laboratories to better elucidate the structure–activity relationship of these galactopyranosyl derivatives against *M. tuberculosis*.

3. Experimental

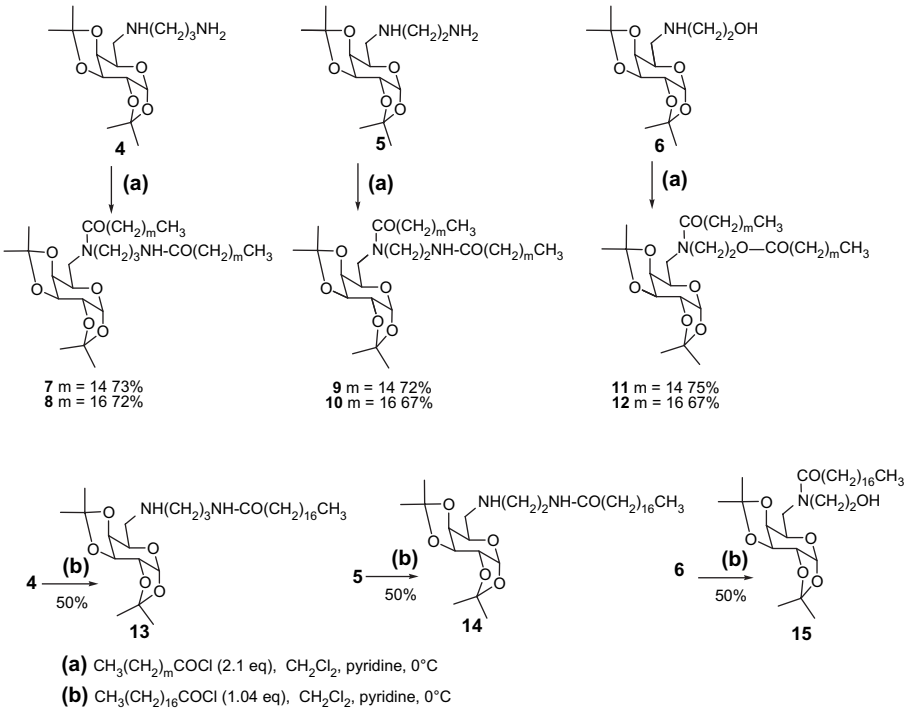
3.1. General methods

Melting points were determined on a Microquímica MQAPF apparatus and are uncorrected. IR spectra were recorded using a BOMEM-FTIR MB102 spectrometer. Optical rotations were measured with a Perkin Elmer 341 polarimeter, using a sodium lamp ($\lambda = 589$ nm) at 20 °C. ^{1}H and ^{13}C NMR spectra were recorded on Bruker Advance DRX300 and DRX400 spectrometers. Elemental analyses were performed at the Central Analítica of Instituto de Química of the Universidade de São Paulo, Brazil. Thin-layer chromatography (TLC) was performed on glass plates and silica gel sheets (Silica Gel F254, Merck) and visualized with iodine vapor and/or revealed with ethanolic H_2SO_4 solution. Column chromatography was carried out on silica gel (E. Merck 230–400 mesh). Solvents were purchased from Vetec Química and were distilled prior to use. Reagents were purchased from Aldrich and used without further purification.

* Indicates interchangeable assignment of NMR.

3.2. General procedure for the amination of protected iodogalactose (*3*)

A solution of the iodinated compound **3** (10 mmol) in THF (10 mL) was slowly added to an ethanolic solution of the amine (50 mmol; 1,3-propanediamine, 1,2-ethanediamine or

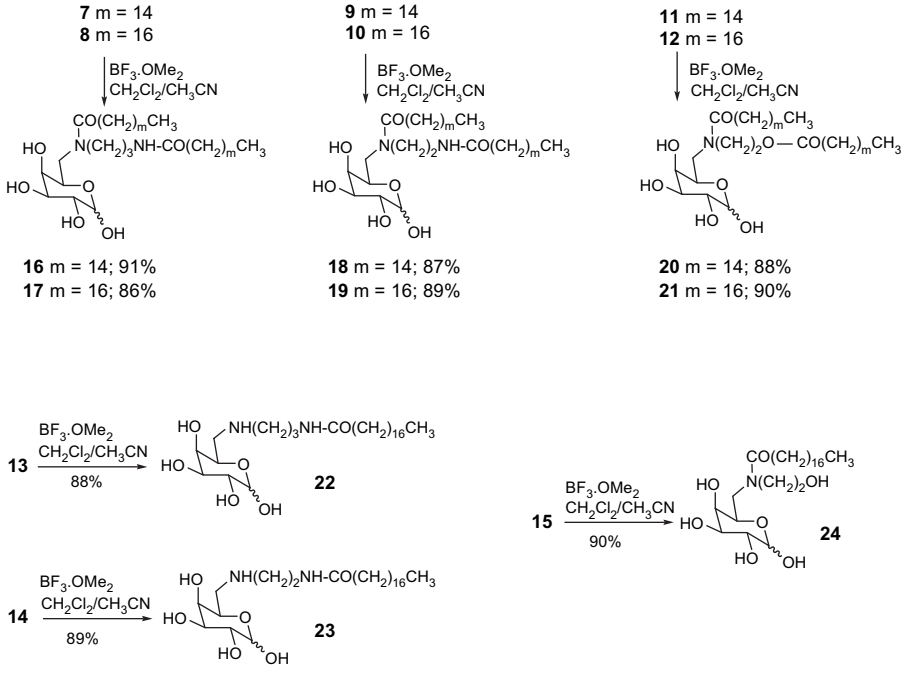


Scheme 2. Preparation of mono- and diacylated compounds.

ethanolamine). The mixture was stirred under reflux for 72 h and the solution was concentrated under reduced pressure. The crude product was dissolved in methylene chloride and extracted three times with water. After drying with sodium sulphate the organic phase was concentrated under reduced pressure. The residue was chromatographed on silica gel (methylene chloride/methanol) to furnish the desired compounds **4**, **5** and **6**, respectively.

*3.2.1. 6-(3-Aminopropylamino)-6-deoxy-1,2:3,4-di-O-isopropylidene-α-D-galactopyranose (**4**)*

From compound **3** (3.7 g), oil (2.5 g, 79%). $[\alpha]_D$: −53.9 (*c* 0.63, CH_2Cl_2). ^1H NMR ($CDCl_3$, 300 MHz): δ 5.40 (d, 1H, $J_{1,2}$ 5.1, H_1), 4.45 (dd, 1H, $J_{3,4}$ 8.0, $J_{3,2}$ 2.3, H_3), 4.17 (dd, 1H, $J_{2,1}$ 5.1, $J_{2,3}$ 2.3, H_2), 4.05 (dd, 1H, $J_{4,3}$ 8.0, $J_{4,5}$ 1.8, H_4), 3.77 (m, 1H, H_5), 2.75 (dd, 1H, $J_{6',6}$ 12.0, $J_{6,5}$ or $J_{6',5}$ 8.9, H_6 or $H_{6'}$), 2.56 (m, 8H, H_6 or $H_{6'}$, H_7, H_9, NH, NH_2), 1.51 (m, 2H,



Scheme 3. Synthesis of compounds **16**–**24** by cleavage of the isopropylidene groups.

Table 1
In vitro antitubercular activities

Compound	MIC (μg/mL)
4	>50
6	>50
7	>50
9	>50
12	>50
13	12.5
14	<3.12
15	25
17	>50
19	>50
Rifampicin	1.0

$J_{8,7}$ or $J_{8,9}$ 6.9, H_8), 1.41–1.20 (3 s, 12H, 4 $CH_{3i\text{-}Pr}$); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 109.1 and 108.4 ($C_{i\text{-}Pr}$), 96.3 (C-1), 71.9 (C-4)*, 70.7 (C-3)*, 70.5 (C-2)*, 66.5 (C-5), 49.4 (C-6), 47.3 (C-7), 40.1 (C-9), 32.4 (C-8), 25.9, 25.8, 24.8 and 24.2 ($CH_{3i\text{-}Pr}$). IR (ν, cm^{-1}, KRS-5): 3367, 2987, 2933 and 1068. Anal. Calcd. for $C_{15}H_{28}N_2O_5$: C, 56.94; H, 8.92; N, 8.85. Found: C, 56.55; H, 8.72; N, 8.47.

3.2.2. 6-(2-Aminoethylamino)-6-deoxy-1,2:3,4-di-O-isopropylidene-α-D-galactopyranose (*5*) [35]

From compound **3** (3.7 g), oil, (2.4 g, 80%). $[\alpha]_D$: −46.0 (*c* 1.0, DMSO) ^1H NMR (C_5D_5N, 400 MHz): δ 5.74 (d, 1H, $J_{1,2}$ 5.2, H_1), 4.74 (dd, 1H, $J_{3,4}$ 8.0, $J_{3,2}$ 2.4, H_3), 4.47 (dd, 1H, $J_{2,1}$ 5.2, $J_{2,3}$ 2.4, H_2), 4.36 (dd, 1H, $J_{4,3}$ 8.0, $J_{4,5}$ 2.0, H_4), 4.17 (dt, 1H, $J_{5,4}$ 2.0, $J_{5,6}$ or $J_{5,6'}$ 5.6, H_5), 3.10 (m, 5H, H_6 or $H_{6'}$, H_7, NH_2), 2.99 (dd, 1H, $J_{6',6}$ 12.4, $J_{6,5}$ or $J_{6',5}$ 5.6, H_6 or $H_{6'}$), 2.75 (m, 3H, H_8, NH), 1.54–1.30, (4s, 12H, $CH_{3i\text{-}Pr}$); ^{13}C NMR (C_5D_5N, 100 MHz): δ 109.0 and 108.4 ($C_{i\text{-}Pr}$), 96.9 (C-1), 72.4, (C-4)*, 71.3 (C-3)*, 71.2 (C-2)*, 67.6 (C-5), 52.9 (C-6), 50.0 (C-7), 42.3 (C-8), 26.3, 26.2, 25.0 and 24.5 ($CH_{3i\text{-}Pr}$). IR (ν, cm^{-1}, KRS-5): 3364, 2987, 2935 and 1068. Anal. Calcd. for $C_{14}H_{26}N_2O_5$: C, 55.61; H, 8.67; N, 9.26. Found: C, 55.36; H, 8.50; N, 9.02.

3.2.3. 6-(2-Hydroxyethylamino)-6-deoxy-1,2:3,4-di-O-isopropylidene-α-D-galactopyranose (*6*)

From compound **3** (3.7 g), solid (2.4 g, 80%), mp: 85.5–87.5 °C. $[\alpha]_D$: −54.5 (*c* 0.46, CH_2Cl_2). ^1H NMR ($CDCl_3$, 300 MHz): δ 5.80 (d, 1H, $J_{1,2}$ 4.9, H_1), 4.86 (dd, 1H, $J_{3,4}$ 7.9, $J_{3,2}$ 2.0, H_3), 4.59 (dd, 1H, $J_{2,1}$ 4.9, $J_{2,3}$ 2.0, H_2), 4.46 (d, 1H, $J_{4,3}$ 7.9, H_4), 4.16 (m, 1H, H_5), 3.89 (t, 2H, $J_{8,7}$ 5.1, H_8), 3.18 (dd, 1H, $J_{6',6}$ 12.4, $J_{6,5}$ or $J_{6',5}$, 8.6, H_6 or $H_{6'}$), 3.06 (m, 3H, H_6 or $H_{6'}$, H_7), 2.50 (s, 2H, NH, OH), 1.80–1.60 (3 s, 12H, $CH_{3i\text{-}Pr}$); ^{13}C NMR ($CDCl_3$, 75 MHz): δ 109.4 and 108.7 ($C_{i\text{-}Pr}$), 96.5 (C-1), 72.0 (C-4)*, 71.0 (C-3)*, 70.6 (C-2)*, 66.9 (C-5), 51.1 (C-6), 60,8 (C-8), 49.2 (C-7), 26.2, 26.1, 25.0 and 24.5 ($CH_{3i\text{-}Pr}$). IR (ν, cm^{-1}, KBr): 3400, 3312, 2938, 2867 and 1071. Anal. Calcd. for $C_{14}H_{25}NO_6$: C, 55.43; H, 8.31; N, 4.62. Found: C, 55.79; H, 8.09; N, 4.34.

3.3. General procedure for the acylation of amines *4*, *5* and *6*

3.3.1. Diacylation

To a cold solution of the amine **4**, **5** or **6** in pyridine (40 mL), stirred in an ice bath, was added a solution of acid chloride (2.1 equiv.) in methylene chloride (10 mL). The mixture was stirred for 12 h at 0 °C and the solvent was concentrated under reduced pressure. The crude product was dissolved in methylene chloride and washed twice with a saturated aqueous solution of sodium bicarbonate. The organic phase was dried with magnesium sulphate and concentrated under reduced pressure. The residue was chromatographed on silica gel (hexane/ethyl acetate) to furnish the diacylated derivatives **7**–**12**.

3.3.2. Monoacylation

The procedure was the same used for the diacyl derivatives, using only 1.04 equiv. of stearoyl chloride, furnishing the monoacylated compounds **13**, **14** and **15**.

3.3.3. 6-[(N,N′-Dihexadecanoyl)-3-aminopropylamino]-6-deoxy-1,2:3,4-di-O-isopropylidene-α-D-galactopyranose (*7*)

From compound **4** (1.11 g), solid (2.03 g, 73%), mp: 51.0–54.0 °C. $[\alpha]_D$: −12.3 (*c* 0.52, CH_2Cl_2). ^1H NMR (DMSO-d_6, 400 MHz): δ 7.77–7.62 (s, 1H, NH), 5.44 and 5.40 (2d, 1H, $J_{1,2}$ 4,8, H_1), 4.58 (2dd, 1H, $J_{3,4}$ 8.0, H_3), 4.30 (2dd, 1H, $J_{2,1}$ 4.8, H_2), 4.16 (2d, 1H, $J_{4,3}$ 8.0, H_4), 3.98 and 3.81 (2d, 1H, $J_{5,6}$ or $J_{5,6}$ 7.6, H_5), 3.57–3.04 (m, 6H, H_6, H_7, H_9), 2.25–2.05 (m, 4H, H_{10}, $H_{10'}$), 1.63 (m, 2H, $J_{8,7}$ or $J_{8,9}$ 6.4, H_8), 1.49 (m, 4H, H_{11}, $H_{11'}$), 1.42, 1.39, 1.38 and 1.35 (4 s, 6H, $CH_{3i\text{-}Pr}$), 1.31–1.24 (m, 54H, $CH_{3i\text{-}Pr}$, CH_{2aliph}), 0.86 (t, 6H, *J* 6.8, CH_3); ^{13}C NMR ($CDCl_3$, 75.0 MHz): δ 175.3 and 173.6 (CO), 109.6–108.4 ($C_{i\text{-}Pr}$), 96.4 (C-1), 72.1 and 71.6 (C-4), 71.0 and 70.9 (C-3), 70.6 and 70.3 (C-2), 67.3 and 66.2 (C-5), 48.0 (C-6), 42.8 (C-7), 37.1 and 37.0 (C-9), 36.9 and 35.6 (C-10′)*, 33.4 and 33.3 (C-10)*, 32.0–22.7 ($CH_{3i\text{-}Pr}$, CH_{2aliph}), 29.8 (C-8), 14.2 (CH_{3aliph}). IR (ν, cm^{-1}, KBr): 3282, 2918, 2850, 1640 and 1067. Anal. Calcd. for $C_{47}H_{88}N_2O_7$: C, 71.17; H, 11.18; N, 3.53. Found: C, 70.89; H, 10.85; N, 3.73.

3.3.4. 6-[(N,N′-Dioctadecanoyl)-3-aminopropylamino]-6-deoxy-1,2:3,4-di-O-isopropylidene-α-D-galactopyranose (*8*)

From compound **4** (1.11 g), oil (2.15 g, 72%). $[\alpha]_D$: −8.2 (*c* 0.46, CH_2Cl_2). ^1H NMR ($CDCl_3$, 300 MHz): δ 6.97 (s, 1H, NH), 5.49 (d, 1H, $J_{1,2}$ 4.9, H_1), 4.60 (2dd, 1H, $J_{3,4}$ 8.0, H_3), 4.30 (2dd, 1H, $J_{2,1}$ 4.9, H_2), 4.12 (2d, 1H, $J_{4,3}$ 8.0, H_4), 3.90 (m, 1H, H_5), 3.71–3.34 (m, 6H, H_6, H_7, H_9), 2.40–2.05 (m, 4H, H_{10}, $H_{10'}$), 1.61 (m, 6H, H_8, H_{11}, $H_{11'}$), 1.42–1.38 (3 s, 9H, $CH_{3i\text{-}Pr}$), 1.35–1.24 (m, 59H, $CH_{3i\text{-}Pr}$, CH_{2aliph}), 0.87 (t, 6H, *J* 6.9, CH_{3aliph}); ^{13}C NMR ($CDCl_3$, 75.0 MHz): δ 178.0 and 174.0 (CO), 109.6–108.7 ($C_{i\text{-}Pr}$), 96.4 (C-1), 72.1, 71.6 (C-4)*, 71.0, 70.9 (C-3)*, 70.6, 70.4 (C-2)*, 67.3, 66.1 (C-5), 48.0 (C-6), 42.9 (C-7), 37.0 (C-9), 36.9, 35.8 (C-10′)*, 34.3, 33.4 (C-10)*, 32.0–22.8 ($CH_{3i\text{-}Pr}$, CH_{2aliph}), 29,8 (C-8), 14.2 (CH_{3aliph}). IR (ν, cm^{-1}, KRS-5): 3307, 2918, 2851,

1644 and 1071. Anal. Calcd. for $C_{51}H_{96}N_2O_7$: C, 72.12; H, 11.39; N, 3.30. Found: C, 71.93; H, 11.07; N, 2.96.

3.3.5. 6-[(N,N′-Dihexadecanoyl)-2-aminoethylamino]-6-deoxy-1,2:3,4-di-O-isopropylidene-α-D-galactopyranose (**9**)

From compound **5** (1.06 g), solid (1.97 g, 72%), mp: 39.0–42.0 °C. $[\alpha]_D$: +34.2 (*c* 0.54, CH_2Cl_2). ^{1}H NMR ($CDCl_3$, 300 MHz): δ 6.63 (s, 1H, NH), 5.51 (d, 1H, $J_{1,2}$ 4.8, H_1), 4.59 (dd, 1H, $J_{3,4}$ 8.0, H_3), 4.30 (m, 2H, H_2, H_4), 4.18 (m, 1H, H_5), 3.90 (m, 1H, H_6 or $H_{6'}$), 3.62 (m, 1H, H_6 or $H_{6'}$), 2.96–2.78 (m, 4H, H_7, H_8), 2.12–2.04 (m, 4H, H_9, $H_{9'}$), 1.56 (m, 4H, H_{10}, $H_{10'}$), 1.46–1.44 (3 s, 6H, $CH_{3i\text{-Pr}}$); 1.36–1.24 (m, 54H, $CH_{3i\text{-Pr}}$, CH_{2aliph}), 0.87 (t, 6H, *J* 6.7, CH_{3aliph}); ^{13}C NMR ($CDCl_3$, 75.0 MHz): δ 174.6 and 174.0 (CO), 109.6–108.7 ($C_{i\text{-Pr}}$), 96.7 and 96.5 (C-1), 72.2 and 71.6 (C-4)*, 71.0 (C-3)*, 70.7 and 70.5 (C-2)*, 66.6 (C-5), 49.5 (C-6), 45.7 (C-7), 39.5 and 37.5 (C-8), 37.0 and 36.9 (C-9′)*, 33.5 (C-9)*, 32.1–22.9 ($CH_{3i\text{-Pr}}$, CH_{2aliph}), 14.3 (CH_{3aliph}). IR (ν, cm^{-1}, KBr): 3307, 2919, 2851, 1635 and 1071. Anal. Calcd. for $C_{46}H_{86}N_2O_7$: C, 70.91; H, 11.12; N, 3.60. Found: C, 70.56; H, 10.72; N, 3.73.

3.3.6. 6-[(N,N′-Dioctadecanoyl)-2-aminoethylamino]-6-deoxy-1,2:3,4-di-O-isopropylidene-α-D-galactopyranose (**10**)

From compound **5** (1.06 g), oil (1.96 g, 67%). $[\alpha]_D$: +32.1 (*c* 0.80, CH_2Cl_2). ^{1}H NMR ($CDCl_3$, 300 MHz): δ 6.68 (s, 1H, NH), 5.48 (d,1H, $J_{1,2}$ 4.8, H_1), 4.57 (dd, 1H, $J_{3,4}$ 8.0, H_3), 4.30 (m, 1H, H_2), 4.17 (dd, 1H, $J_{4,3}$ 8.0, H_4), 4.00 (m, 1H, H_5), 3.90–3.30 (m, 4H, H_7, H_8), 3.62 (m, 1H, H_6 or $H_{6'}$), 2.85 (m, 1H, H_6 or $H_{6'}$), 2.31–2.04 (m, 4H, H_9, $H_{9'}$), 1.57 (m, 4H, H_{10}, $H_{10'}$), 1.45–1.43 (4 s, 6H, $CH_{3i\text{-Pr}}$), 1.36–1.24 (m, 62H, $CH_{3i\text{-Pr}}$ and CH_{2aliph}), 0.85 (t, 6H, CH_{3aliph}, *J* 7.1); ^{13}C NMR ($CDCl_3$, 75.0 MHz): 178.0, 174.0 (CO), 109.6–108.7 ($C_{i\text{-Pr}}$), 96.7, 96.5 (C-1), 72.1, 71.5 (C-4)*, 71.0 (C-3)*, 70.7, 70.4 (C-2)*, 66.6, 66.1 (C-5), 49.4, 49.1 (C-6), 48.4, 45.4 (C-7), 39.3, 37.5 (C-8), 36.9, 36.8 (C-9′)*, 34.2, 33.5 (C-9)*, 32.1–22.9 ($CH_{3i\text{-Pr}}$, CH_{2aliph}), 14.3 (CH_{3aliph}). IR (ν, cm^{-1}, KRS-5): 3316, 2918, 2851, 1629 and 1071. Anal. Calcd. for $C_{50}H_{94}N_2O_7$: C, 71.90; H, 11.34; N, 3.35. Found: C, 71.55; H, 10.96; N, 3.15.

3.3.7. 6-[(N,O-Dihexadecanoyl)-2-hydroxyethylamino]-6-deoxy-1,2:3,4-di-O-isopropylidene-α-D-galactopyranose (**11**)

From compound **6** (1.06 g), oil (2.04 g, 75%) $[\alpha]_D$: −9.0 (*c* 0.73, CH_2Cl_2). ^{1}H NMR ($CDCl_3$, 300 MHz): δ 5.47 (2d, 1H, $J_{1,2}$ 4.6, H_1), 4.55 (2dd, 1H, $J_{3,4}$ 8.0, H_3), 4.24 (m, 4H, H_2, H_4, H_8), 3.92 (m, 1H, H_5), 3.67–3.00 (m, 4H, H_6, H_7), 2.40–2.27 (m, 4H, H_9, $H_{9'}$), 1.60 (m, 4H, H_{10}, $H_{10'}$), 1.48–1.44 (4 s, 6H, $CH_{3i\text{-Pr}}$), 1.35–1.26 (m, 54H, $CH_{3i\text{-Pr}}$, CH_{2aliph}), 0.88 (t, 6.7, 6H, CH_{3aliph}); ^{13}C NMR ($CDCl_3$, 75.0 MHz): δ 176.0 and 72.0 (CO), 109.7 and 108.8 ($C_{i\text{-Pr}}$), 96.6 and 96.5 (C-1), 72.0 and 71.4 (C-4)*, 71.1 and 70.7 (C-3)*, 70.6 and 70.0 (C-2)*, 66.3 and 66.2 (C-5), 62.3 and 62.1 (C-8), 49.0 and 48.4 (C-6), 48.0 and 45.3 (C-7), 35.6 and 34.5 (C-9′)*, 34.4 and 34.2 (C-9)*, 32.1–22.9 ($CH_{3i\text{-Pr}}$, CH_{2aliph}), 14.3 (CH_{3aliph}). IR (ν, cm^{-1}, KRS-5): 2924, 2854, 1741, 1646 and 1071. Anal. Calcd. for $C_{46}H_{85}NO_8$: C, 70.82; H, 10.98; N, 1.80. Found: C, 70.43; H, 10.69; N, 2.12.

3.3.8. 6-[(N,O-Dioctadecanoyl)-2-hydroxyethylamino]-6-deoxy-1,2:3,4-di-O-isopropylidene-α-D-galactopyranose (**12**)

From compound **6** (1.06 g), oil (2.13 g, 67%). $[\alpha]_D$: −9.0 (*c* 0.54, CH_2Cl_2). ^{1}H NMR ($CDCl_3$, 300 MHz): δ 5.47 (2d, 1H, H_1, $J_{1,2}$ 5.1), 4.55 (2dd, 1H, H_3, $J_{3,4}$ 8.0), 4.24 (m, 4H, H_2, H_4, H_8), 3.92 (m, 1H, H_5), 3.67, 3.00 (m, 4H, H_6, H_7), 2.39–2.26 (m, 4H, H_9, $H_{9'}$), 1.61 (m, 4H, H_{10}, $H_{10'}$), 1.47, 1.46, 1.44 (s, 9H, $CH_{3i\text{-Pr}}$), 1.35–1.26 (m, 59H, $CH_{3i\text{-Pr}}$, CH_{2aliph}), 0.87 (t, 6H, *J* 6.9); ^{13}C NMR ($CDCl_3$, 75.0 MHz): δ 175.0–173.0 (CO), 109.7–108.8 ($C_{i\text{-Pr}}$), 96.5 (C-1), 72.0, 71.4 (C-4)*, 71.0, 70.7 (C-3)*, 70.6, 70.0 (C-2)*, 66.3 (C-5), 62.3, 62.1 (C-8), 48.8, 48.3 (C-6), 48.0, 45.3 (C-7), 34.4 (C-9′)*, 34.3 (C-9)*, 32.1–22.9 ($CH_{3i\text{-Pr}}$, CH_{2aliph}), 14.3 (CH_{3aliph}). IR (ν, cm^{-1}, KRS-5): 2919, 2851, 1739, 1648 and 1071. Anal. Calcd. for $C_{50}H_{93}NO_8$: C, 71.81; H, 11.21; N, 1.67. Found: C, 71.46; H, 10.93; N, 1.61.

3.3.9. 6-[(3-N-Octadecanoyl)-3-aminopropylamino]-6-deoxy-1,2:3,4-di-O-isopropylidene-α-D-galactopyranose (**13**)

From compound **4** (0.5 g), oil (0.46 g, 50%). $[\alpha]_D$: −30.5 (*c* 0.59, CH_2Cl_2). ^{1}H NMR ($CDCl_3$, 300 MHz): δ 6.83 (s, 1H, NH), 5.53 (d, 1H, $J_{1,2}$ 5.1, H_1), 4.60 (dd, 1H, $J_{3,4}$ 8.0, $J_{3,2}$ 2.6, H_3), 4.32 (dd, 1H, $J_{2,1}$ 5.1, $J_{2,3}$ 2.6, H_2), 4.17 (dd, 1H, $J_{4,3}$ 8.0, $J_{4,5}$ 2.0, H_4), 3.96 (m, 1H, H_5), 3.33 (t, 2H, $J_{7,8}$ or $J_{9,8}$ 6.2, H_7 or H_9), 2.95–2.75 (m, 5H, H_7 or H_9, H_6, NH), 2.14 (t, 2H, $J_{10,11}$ 6.9, H_{10}), 1.72 (m, 2H, H_8), 1.60 (m, 2H, H_{11}), 1.53, 1.44 (s, 6H, $CH_{3i\text{-Pr}}$), 1.32–1.24 (m, 34H, $CH_{3i\text{-Pr}}$, CH_{2aliph}), 0.87 (t, 3H, *J* 6.9, CH_{3aliph}); ^{13}C NMR ($CDCl_3$, 75.0 MHz): δ 173.4 (CO), 109.3, 108.6 ($C_{i\text{-Pr}}$), 96.3 (C-1), 71.8 (C-4)*, 70.8 (C-3)*, 70.5 (C-2)*, 66.2 (C-5), 49.2 (C-6), 47.6 (C-7), 38.3 (C-9), 36.8 (C-10), 31.8–22.6 ($CH_{3i\text{-Pr}}$, CH_{2aliph}), 29.7 (C-8), 14.1 ($CH_{3i\text{-Pr}}$). IR (ν, cm^{-1}, KRS-5): 3229, 2918, 2850, 1642 and 1070. Anal. Calcd. for $C_{33}H_{62}N_2O_6$: C, 68.00; H, 10.72; N, 4.81. Found: C, 67.68; H, 10.38; N, 4.44.

3.3.10. 6-[(2-N-Octadecanoyl)-2-aminoethylamino]-6-deoxy-1,2:3,4-di-O-isopropylidene-α-D-galactopyranose (**14**)

From compound **5** (0.5 g), oil (0.47 g, yield 50%). $[\alpha]_D$: −29.5 (*c* 0.63, CH_2Cl_2). ^{1}H NMR ($CDCl_3$, 300 MHz): δ 6.60 (s, 1H, NH), 5.52 (d, 1H, $J_{1,2}$ 5.1, H_1), 4.59 (dd, 1H, $J_{3,4}$ 8.0, $J_{3,2}$ 2.3, H_3), 4.31 (dd, 1H, $J_{2,1}$ 5.1, $J_{2,3}$ 2.3, H_2), 4.16 (dd, 1H, $J_{4,3}$ 8.0, $J_{4,5}$ 1.8, H_4), 3.92 (m, 1H, H_5), 3.38 (m, 3H, NH, H_8), 2.97–2.82 (m, 4H, H_7, H_6), 2.16 (t, 2H, H_9, $J_{9,10}$ 7.4), 1.60 (m, 2H, H_{10}), 1.53, 1.42 (s, 1H, $CH_{3i\text{-Pr}}$), 1.32–1.24 (m, 34H, $CH_{3i\text{-Pr}}$, CH_{2aliph}), 0.86 (t, 3H, *J* 6.4, CH_{3aliph}); ^{13}C NMR ($CDCl_3$, 75.0 MHz): δ 173.6 (CO), 109.4, 108.7 ($C_{i\text{-Pr}}$), 96.5 (C-1), 71.8 (C-4), 70.8 (C-3), 70.4 (C-2), 66.1 (C-5), 48.7 (C-6), 48.1 (C-7), 38.2 (C-8), 36.7 (C-9), 31.9–22.6 ($CH_{3i\text{-Pr}}$, CH_{2aliph}), 14.1 (CH_{3aliph}). IR (ν, cm^{-1}, KRS-5): 3300, 2919, 2851, 1651 and 1071. Anal. Calcd. for $C_{32}H_{60}N_2O_6$: C, 67.57; H, 10.63; N, 4.92. Found. C, 67.19; H 10.29; N, 4.63.

*3.3.11. 6-[(N-Octadecanoyl)-2-hydroxyethylamino]-6-deoxy-1,2:3,4-di-O-isopropylidene-α-D-galactopyranose (**15**)*

From compound **6** (0.50 g), oil (0.47 g, 50%). $[\alpha]_D$: −6.45 (*c* 0.83, CH_2Cl_2). ^1H NMR ($CDCl_3$, 300 MHz): δ 5.50 (2d, 1H, H_1, $J_{1,2}$ 5.6), 4.58 (2dd, 1H, H_3, $J_{3,4}$ 7.7), 4.26 (m, 2H, H_8), 4.16–4.00 (m, 2H, H_2, H_4), 3.97 (m, 1H, H_5), 3.88–2.87 (m, 4H, H_6, H_7), 2.30 (m, 2H, H_9), 1.59 (m, 4H, H_{10}), 1.48, 1.45 (s, 6H, $CH_{3i\text{-Pr}}$), 1.44–1.24 (m, 36H, $CH_{3i\text{-Pr}}$, CH_{2aliph}), 0.87 (t, 3H, CH_{3aliph}, *J* 6.7); ^{13}C NMR ($CDCl_3$, 75.0 MHz): δ 174.8 (CO), 109.7–109.5 ($C_{i\text{-Pr}}$), 96.5 (C-1), 72.1, 71.4 (C-4)*, 71.1, 71.0 (C-3)*, 70.8, 70.5 (C-2)*, 66.6, 66.3 (C-5), 62.7, 60.2 (C-8), 52.5, 51.3 (C-6), 49.9, 49.1 (C-7), 33.8, 33.5 (C-9), 32.1–22.9 ($CH_{3i\text{-Pr}}$, CH_{2aliph}), 14.3 ($CH_{3i\text{-Pr}}$). IR (ν, cm^{-1}, KRS-5): 3403, 2925, 2854, 1625 and 1071. Anal. Calcd. for $C_{32}H_{59}NO_7$: C, 67.45; H, 10.44; N, 2.46. Found: C, 67.15; H, 10.17; N, 2.77.

3.4. General procedure for the deprotection of isopropylidene derivatives

Boron trifluoride dimethyl etherate (2 mL) was slowly added to a solution of the protected compound (100 mg) in 10 mL of a mixture of methylene chloride and acetonitrile (7:3). The solution was stirred at room temperature for 72 h (TLC hexane:ethyl acetate 3:7). The solvent was then evaporated under reduced pressure and the residue was chromatographed on silica gel (methylene chloride/methanol) to furnish the deprotected derivatives **16–24**.

*3.4.1. 6-[(N,N′-Dihexadecanoyl)-3-aminopropylamino]-6-deoxy-D-galactopyranose (**16**)*

From compound **7** (100 mg), solid (82 mg, 91%), mp: 98.0–102.0 °C. $[\alpha]_D$: +28.4 (*c* 0.63, C_5H_5N). ^1H NMR (C_5D_5N, 300 MHz): δ 5.21 (d, 1H, H_1 β, $J_{1,2}$ 7.7), 4.80–3.52 (m, 11H, H_2, H_3, H_4, H_5, H_6, H_7, H_9, $H_{9'}$, $H_{1\alpha}$), 2.82–2.70 (m, 4H, H_{10}, $H_{10'}$), 2.02 (m, 2H, H_8), 1.82 (m, 4H, H_{11}, $H_{11'}$), 1.27 (m, 48H, CH_{2aliph}), 0.88 (t, 6H, *J* 7.0, CH_{3aliph}); ^{13}C NMR (C_5D_5N, 75.0 MHz): δ 174.6, 173.9, 173.8, 173.5 (CO), 99.8 (C-1 β), 94.8 (C-1 α), 75.7 (C-5 α)*, 74.4 (C-3 α)*, 74.1(C-2 β)*, 73.9 (C-5 β)*, 72.0 (C-2 α)*, 71.0 (C-4 α)*, 69.7 (C-4 β)*, 69.2 (C-3 β)*, 49.9, 48.6 (C-6), 48.0, 44.0 (C-7), 37.5–37.1 (C-9), 34.0–23.3 (C-8, C-10, C-10′, CH_{2aliph}), 14.6 (CH_{3aliph}). IR (ν, cm^{-1}, KBr): 3317, 2919, 2852, 1638 and 1081. Anal. Calcd. for $C_{41}H_{80}N_2O_7$: C, 69.06; H, 11.31; N, 3.93. Found: C, 68.81; H, 10.95, N, 3.73.

*3.4.2. 6-[(N,N′-Dioctadecanoyl)-3-aminopropylamino]-6-deoxy-D-galactopyranose (**17**)*

From compound **8** (100 mg), solid (78 mg, 86%), mp: 86.0–89.0 °C. $[\alpha]_D$: +21.6 (*c* 0.56, C_5H_5N). ^1H NMR (C_5D_5N, 300 MHz): δ 5.20 (d, 1H, $J_{1,2}$ 7.7, $H_{1\beta}$), 4.72–3.52 (m, 11H, H_2, H_3, H_4, H_5, H_6, H_7, H_9, $H_{9'}$, $H_{1\alpha}$), 2.42 (m, 4H, H_{10}, $H_{10'}$), 2.02 (m, 2H, H_8), 1.81 (m, 4H, H_{11}, $H_{11'}$), 1.27 (m, 56H, CH_{2aliph}), 0.88 (t, 6H, *J* 6.8, CH_{3aliph}); ^{13}C NMR (C_5D_5N, 75.0 MHz): δ 175.3, 175.2, 174.8, 174.6 (CO), 99.8 (C-1 β), 94.8 (C-1 α), 75.8 (C-5 α)*, 74.5 (C-3 α)*, 74.2 (C-2 β)*, 72.8 (C-5 β)*, 71.8 (C-2 α)*, 71.1 (C-4 α)*, 69.6 (C-4 β)*, 68.9 (C-3 β)*, 50.3 (C-6), 48.2, 44.6 (C-7), 39.7–37.5 (C-9), 34.2–23.6 (C-8, C-10, C-10′, CH_{2aliph}), 14.9 (CH_{3aliph}). IR (ν, cm^{-1}, KBr): 3312, 2917, 2851, 1638 and 1074. Anal. Calcd. for $C_{45}H_{88}N_2O_7$: C, 70.27; H, 11.53; N, 3.64. Found: C, 69.88; H, 11.12; N, 3.94 .

*3.4.3. 6-[(N,N′-Dihexadecanoyl)-2-aminoethylamino]-6-deoxy-D-galactopyranose (**18**)*

From compound **9** (100 mg), solid (78 mg, 87%), mp: 83.0–86.0 °C. $[\alpha]_D$: +31.7 (*c* 0.54, C_5H_5N). ^1H NMR (C_5D_5N, 300 MHz): δ 5.30–3.50 (m, 11H, H_1, H_2, H_3, H_4, H_5, H_6, H_7, H_8), 2.41 (m, 4H, H_9, $H_{9'}$), 2.00 (m, 4H, H_{10}, $H_{10'}$), 1.27 (m, 48H, CH_{2aliph}), 0.88 (t, 6H, *J* 6.8, CH_{3aliph}); ^{13}C NMR (DMSO-d_6, 75.0 MHz): δ 174.5–170.4 (CO), 94.7 (C-1 β), 89.5 (C-1 α), 73.6 (C-5 α)*, 72.1 (C-3 α)*, 71.7 (C-2 β)*, 71.0 (C-5 β)*, 69.8 (C-2 α)*, 69.0 (C-4 α)*, 67.6 (C-4 β)*, 66.3 (C-3 β)*, 55.0 (C-6), 52.5 (C-7), 35.5, 35.3 (C-8, C-9)*, 33.6–22.1 (C-9′*, CH_{2aliph}), 13.9 (CH_{3aliph}). IR (ν, cm^{-1}, KBr): 3316, 2917, 2850, 1646 and 1078. Anal. Calcd. for $C_{40}H_{78}N_2O_7$: C, 68.73; H, 11.25; N, 4.01. Found: C, 68.57; H, 10.93; N 3.92.

*3.4.4. 6-[(N,N′-Dioctadecanoyl)-2-aminoethylamino]-6-deoxy-D-galactopyranose (**19**)*

From compound **10** (100 mg), solid (81 mg, 89%), mp: 85.0–87.0 °C. $[\alpha]_D$: +14.3 (*c* 0.51, C_5H_5N). ^1H NMR (C_5D_5N, 300 MHz): δ 5.22–3.60 (m, 11H, H_1, H_2, H_3, H_4, H_5, H_6, H_7, H_8), 2.42 (m, 4H, H_9, $H_{9'}$), 1.80 (m, 4H, H_{10}, $H_{10'}$), 1.29 (m, 56H, CH_{2aliph}), 0.88 (t, 6H, *J* 6.6, CH_{3aliph}); ^{13}C NMR (C_5D_5N, 75.0 MHz): δ 175.3–174.6 (CO), 99.4 (C-1 β), 94.4 (C-1 α), 75.4 (C-5 α)*, 74.1 (C-3 α)*, 73.9 (C-2 β)*, 71.7 (C-5 β)*, 71.3 (C-2 α)*, 71.0 (C-4 α)*, 70.5 (C-4 β)*, 69.3 (C-3 β)*, 50.6 (C-6), 49.1 (C-7), 38.6–36.9 (C-8), 33.8–23.2 (C-9, C-9, CH_{2aliph}), 14.5 (CH_{3aliph}). IR (ν, cm^{-1}, KBr): 3413, 2917, 2850, 1647 and 1081. Anal. Calcd. for $C_{44}H_{86}N_2O_7$: C, 69.98; H, 11.48; N, 3.71. Found: C, 69.59; H, 11.13; N, 3.93.

*3.4.5. 6-[(N,O-Dihexadecanoyl)-2-hydroxyethylamino]-6-deoxy-D-galactopyranose (**20**)*

From compound **11** (100 mg), oil (79 mg, 88%). $[\alpha]_D$: +7,44 (*c* 1.1, C_5H_5N). ^1H NMR (C_5D_5N, 300 MHz): 5.30–3.50 (m, 11H, H_1, H_2, H_3, H_4, H_5, H_6, H_7, H_8), 2.36 (m, 4H, H_9, $H_{9'}$), 1.80 (m, 4H, H_{10}, $H_{10'}$), 1.27 (m, 48H, CH_{2aliph}), 0.87 (t, 6H, *J* 6.8 Hz, CH_{3aliph}); ^{13}C NMR (C_5D_5N, 75.0 MHz): 175.0–173.0 (CO), 99.8 (C-1 β), 94.8 (C-1 α), 75.7 (C-5 α)*, 74.5 (C-3 α)*, 74.2 (C-2 β)*, 72.1 (C-5 β)*, 71.8 (C-2 α)*, 71.5 (C-4 α)*, 71.1 (C-4 β)*, 69.4 (C-3 β)*, 62.5 (C-8), 50.1 (C-6), 48.0 (C-7); 34.7–23.2 (C-9, C-9′, CH_{2aliph}); 14.6 (CH_{3aliph}). IR (ν, cm^{-1}, KRS-5): 3419, 2919, 2851, 1740, 1621 and 1084. Anal. Calcd. for $C_{40}H_{77}NO_8$: C, 68.63; H, 11.09; N, 2.00. Found: C, 68.30; H, 10.82; N, 1.89.

*3.4.6. 6-[(N,O-Dioctadecanoyl)-2-hydroxyethylamino]-6-deoxy-D-galactopyranose (**21**)*

From compound **12** (100 mg), solid (82 mg, 90%), mp: 120–123 °C. $[\alpha]_D$: +22.4 (*c* 0.6, C_5H_5N). ^1H NMR (C_5D_5N, 300 MHz): δ 5.28 (d, 1H, $J_{1,2}$ 7.7, $H_{1\beta}$), 5.21 (d, 1H, $J_{1,2}$ 7.7, $H_{1\beta}$), 4.92–3.62 (m, 11H, $H_{1\alpha}$, H_2, H_3, H_4, H_5, H_6, H_7, H_8), 2.40 (m, 4H, H_9, $H_{9'}$), 1.82 (m, 4H, H_{10}, $H_{10'}$), 1.35–1.25 (m, 56H, CH_{2aliph}), 0.89 (t, 6H, *J* 6.8, CH_{3aliph}); ^{13}C NMR (C_5D_5N, 75.0 MHz): δ 175.0–173.0 (CO), 97.4 (C-1 β), 91.8 (C-1 α), 76.4 (C-5 α)*, 74.3 (C-3 α)*, 74.2 (C-2 β)*, 73.7 (C-5 β)*, 72.2 (C-2 α)*, 71.6 (C-4 α)*, 71.3 (C-4 β)*, 69.4 (C-3 β)*, 62.7 (C-8), 50.7 (C-6), 46.0 (C-7), 34.9–21.6 (C-9, C-9′, CH_{2aliph}), 14.8 (CH_{3aliph}). IR (ν, cm^{-1}, KBr): 3404, 2918, 2851, 1737, 1622 and 1066. Anal. Calcd. for $C_{44}H_{85}NO_8$: C, 69.89; H, 11.33; N, 1.85. Found: C, 69.58; H, 10.99; N, 1.83.

*3.4.7. 6-[(3-N-Octadecanoyl)-3-aminopropylamino]-6-deoxy-D-galactopyranose (**22**)*

From compound **13** (100 mg), oil (76 mg, 88%). ^1H NMR (C_5D_5N, 300 MHz): δ 5.28 (d, 1H, $J_{1,2}$ 7.7, H_1 β), 4.77–3.39 (m, 11H, $H_{1\alpha}$, H_2, H_3, H_4, H_5, H_6, H_7, H_9, $H_{9'}$), 2.45 (t, 2H, $J_{10,11}$ 7.5, H_{10}), 2.26 (m, 2H, H_8), 1.91 (m, 2H, H_{11}), 1.27 (m, 28H, CH_{2aliph}), 0.88 (t, 3H, *J* 6.9, CH_{3aliph}); ^{13}C NMR (C_5D_5N, 75.0 MHz): δ 175.1–174.0 (CO), 99.6 (C-1 β), 94.5 (C-1 α), 76.2 (C-5 α)*, 74.7 (C-3 α)*, 73.7 (C-2 β)*, 71.8 (C-5 β)*, 71.7 (C-2 α)*, 70.9 (C-4 α)*, 68.3 (C-4 β)*, 66.9 (C-3 β)*, 52.2 (C-6), 49.4 (C-7), 37.2–36.6 (C-9, C-10), 32.3–23.2 (C-8, CH_{2aliph}), 14.5 (CH_{3aliph}). IR (ν, cm^{-1}, KRS-5): 3416, 2918, 2850, 1639 and 1084. Anal. Calcd. for $C_{27}H_{54}N_2O_6$: C, 64.51; H, 10.83; N, 5.57. Found: C, 64.12; H, 10.43; N, 5.82.

*3.4.8. 6-[(2-N-Octadecanoyl)-2-aminoethylamino]-6-deoxy-D-galactopyranose (**23**)*

From compound **14** (100 mg), oil (77 mg, 89%). ^1H NMR (C_5D_5N, 300 MHz): δ 5.26 (d, 1H, $J_{1,2}$ 7.7, H_1 β), 4.60–3.58 (m, 11H, H_1 α, H_2, H_3, H_4, H_5, H_6, H_7, H), 2.35 (t, 2H, $J_{9,10}$ 6.9, H_9), 1.71 (m, 2H, H_{10}), 1.25 (m, 28H, CH_{2aliph}), 0.85 (t, 3H, *J* 6.9, CH_{3aliph}); ^{13}C NMR (C_5D_5N, 75.0 MHz): δ 175.5–174.0 (CO), 99.9 (C-1 β), 94.9 (C-1 α), 76.8 (C-5 α)*, 75.3 (C-3 α)*, 74.0 (C-2 β)*, 72.2 (C-5 β)*, 71.5 (C-2 α)*, 70.7 (C-4 α)*, 68.8 (C-4 β)*, 67.0 (C-3 β)*, 52.1 (C-6), 49.8 (C-7), 37.3–36.8 (C-8, C-9), 32.6–23.4 (CH_{2aliph}), 14,8 (CH_{3aliph}). IR (ν, cm^{-1}, KRS-5): 3387, 2918, 2850, 1635 and 1084. Anal. Calcd. for $C_{26}H_{52}N_2O_6$: C, 63.90; H, 10.72; N, 5.73. Found: C, 63.65; H, 10.55; N, 5.54.

*3.4.9. 6-[(N-Octadecanoyl)-2-hydroxyethylamino]-6-deoxy-D-galactopyranose (**24**)*

From compound **15** (100 mg), oil (77 mg, 90%). $[\alpha]_D$: +14,5 (*c* 0.67, C_5H_5N). ^1H NMR (C_5D_5N, 300 MHz): δ 5.20–3.35 (m, 11H, H_1, H_2, H_3, H_4, H_5, H_6, H_7, H_8), 2.36 (t, 2H, $J_{9,10}$ 7.5, H_9), 1.66 (m, 2H, H_{10}), 1.28 (m, 28H, CH_{2aliph}), 0.89 (t, *J*7.1, 3H, CH_{3aliph}); ^{13}C NMR (C_5D_5N, 75.0 MHz): δ 173.7–173.2 (CO), 103.0 (C-1 β), 83.0 (C-1 α), 76.7–65.9 (C-2, C-3, C-4, C-5), 62.3 (C-8), 55.3 (C-6), 52.1 (C-7), 35.0–34.0 (C-9), 32.20–23.0 (CH_{2aliph}), 14.4 CH_{3aliph}). IR (ν, cm^{-1}, KRS-5): 3341, 2922, 2852, 1657 and 1036. Anal. Calcd. for $C_{26}H_{51}NO_7$: C, 63.77; H, 10.50; N, 2.86. Found: C, 63.40; H, 10.12; N, 2.72.

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