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Original article

Synthesis and antimycobacterial activities of glycosylated amino alcohols and amines

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Abstract

Reduction of glycosyl β-amino esters (**6**–**14** and **25**–**30**) with lithium aluminum hydride resulted in glycosyl amino alcohols (**15**–**23** and **31**–**36**) in good yields. However, reductive amination of glycosyl aldehydes (**1**–**3**) with different amines in presence of sodium borohydride resulted in good to moderate yields of glycosyl amines (**37**–**41**). All the compounds were evaluated for antitubercular activity against *Mycobacterium tuberculosis* $H_{37}Ra$ and $H_{37}Rv$. Compounds **18**, **21**, **35** and **36** exhibited antitubercular activities with MIC ranging from 6.25 to 3.12 μg ml^{-1}.

Keywords: Tuberculosis; Glycosyl amines; Glycosyl amino alcohols; Reductive amination; MDR tuberculosis

1. Introduction

Mycobacterium tuberculosis is a single infectious agent that causes tuberculosis and kills roughly two million people annually throughout the world. A WHO report indicates that one third of the world population is infected with this bacterium [1–3]. The synergy of this disease with AIDS and resistance to almost every drug in clinics has further worsened the problem [4], as this deadly pathogen has rendered treatments difficult and in many cases, totally ineffective [5]. These problems can be solved by developing new drugs with novel modes of action with additional antiviral and immunopotentiatory activities. The mycobacterial cell wall is a unique target as it is absent in human host and many crucial enzymes involved in the biosynthesis of different macromolecules as constituent of the cell wall are targets to develop new drugs [6]. The long treatment of tuberculosis with many antibiotics [7–9] is mainly due to the incredible thickness and impermeability of the cell wall complex, which prevents the effective passage of the drugs into the organism [6,10]. Carbohydrates present mostly as arabinogalactan and arabinomannan are the integral part of mycolylated arabinogalactan peptidoglycan (mAGP) complex of mycobacterial cell wall [6]. The sugars present are not the only targets for new drug development but they are involved in pathogenesis of the disease also [11]. The glycosyl transferases and many other enzymes including D-alanine racemase [11,12] and mycolyl transferase [13] are involved in the biosynthesis of the macromolecules of mAGP complex and many inhibitors of these enzymes are known to be good antitubercular compounds. Based upon the structures of known inhibitors of the above enzymes others and we have synthesized certain sugar based amino compounds possessing antimycobacterial activity [14,15]. In our preliminary communications we have shown that glycosyl amino esters [15a] and galactopyranosyl amino alcohols [15c] possess good antitubercular activity in vitro (against MDR strain also) and in vivo (marginal, in mice model). In continuation of the above study we have reported herein the synthesis of glycosylated amino alcohols and amines and their evaluation for antitubercular activity in order to gain an insight into the SAR of this class of compound.

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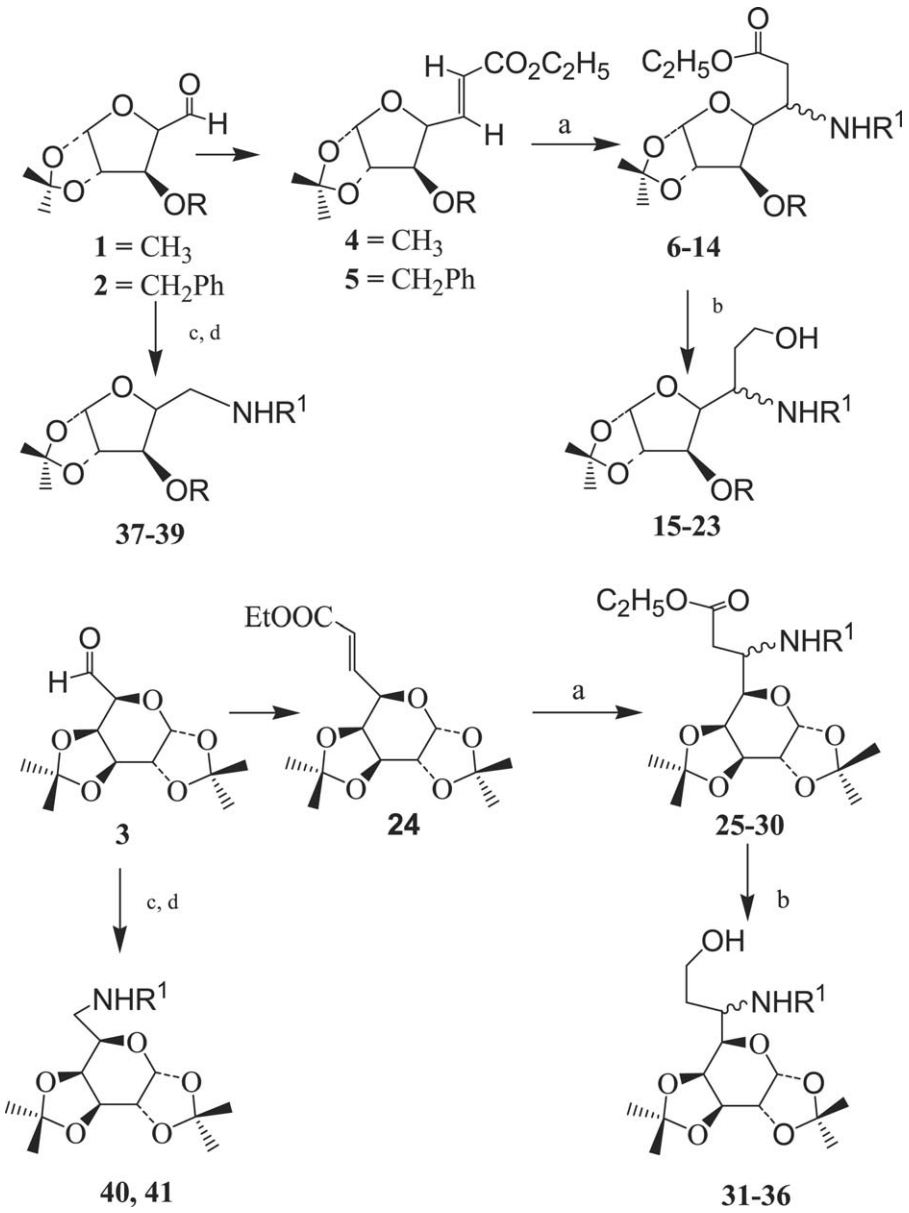
2. Chemistry

The synthetic strategy is shown in Scheme 1. Glycosyl amino esters (**6–14**) [15a] obtained earlier on reduction with lithium aluminum hydride at ambient temperature resulted in corresponding glycosyl amino alcohols (**15–23**), respectively, in very good yields. The reduction of the glycosyl amino esters to the respective amino alcohols could be proved by their spectroscopic data and analysis. The disappearance of signals corresponding to carbethoxy protons in ^{1}H NMR spectra at around δ 4.2 (OCH_2) and 1.25 (OCH$_2$CH_3) and carbon signals of the same in ^{13}C NMR spectra of these glycosyl amino alcohols at around δ 170 (OC=O), 60 (OCH_2) and 14.5 (OCH$_2$CH_3) indicated the conversion of ester into alcohol. As the C-5 stereocenter is not involved in the reduction process, the stereochemistry at this center would be same as in the glycosyl amino esters already established [15a].

Similarly, 1,2:3,4-di-*O*-isopropylidene-α-D-galactopyranosyl amino alcohols (**31–36**) were prepared by reduction of pure diastereoisomeric galactopyranosylated amino esters (**25–30**) [16] having '*S*' stereochemistry at C-6. However, some of the compounds **17**[a], **18**[a], **21**[a], **23**[a] and **35**[a] were synthesized by reducing the minor isomers of the respective glycosyl amino esters having '*R*' stereochemistry at C-5 or C-6. All the compounds synthesized were characterized using ^{1}H NMR, ^{13}C NMR, FAB MS and C, H, N analysis prior to evaluation for antimycobacterial activity and evaluated individually for their efficacy against *M. tuberculosis*.

To see the effect of substituents at C-5 in furanose and C-6 in pyranose and the subsequent structure activity rela-



Scheme 1. Synthesis of glycosyl amino alcohols (**15–23** and **31–36**) and amines (**37–41**): reagents and conditions: (a) R^{1}NH$_2$, C$_2$H$_5$OH, 16–24 h, 25 °C, (b) LiAlH$_4$, THF, 0–25 °C, (c) R^{1}NH$_2$, dry DCM, 4 Å MS, 0 °C, (d) MeOH, NaBH$_4$ or NaCNBH$_3$, NH$_4$Cl, 0–25 °C.

tionship, we thought to synthesize glycosylated amines by reductive amination of glycosyl aldehydes **1** [17], **2** [18] and **3** [16]. The reaction of glycosyl aldehydes (**1**–**3**) with different amines separately resulted in the formation of respective intermediate imines (unisolated), which on in situ reaction with sodium borohydride gave the respective glycosyl amines (**37**–**41**). The structures of the compounds (**37**–**41**) were determined on the basis of their spectroscopic data and microanalysis. Conversion of glycosyl aldehyde into imine as intermediate could be proved by isolation and characterization of one of the intermediates as its ^{1}H NMR spectrum showed disappearance of the aldehydic proton at δ 9.5 and appearance of a doublet at δ 7.79 (imine proton) having J value of 4.1 Hz. The glycosylated amines (**37**–**41**) exhibited the usual signals for the sugar ring protons along with multiplet at around δ 2.8 for NCH_2 in ^{1}H NMR spectra of compounds.

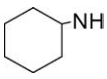
3. Biology

All the newly synthesized compounds were evaluated for their antimycobacterial activity against *M. tuberculosis* H_{37}Rv and H_{37}Ra. The activities of a few of the glycosyl amino esters have been published as preliminary communication [15a]. All the compounds including glycosyl amino alcohols and glycosyl amines were screened for their antitubercular efficacy using microalamar blue (MABA) method against *M. tuberculosis* H_{37}Ra, while agar microdilution method was used against *M. tuberculosis* H_{37}Rv and the results are depicted in Table 1.

4. Results and discussion

As evident from Table 1 compounds **17**[a], **18**, **18**[a], **21**, **21**[a], **22**, **23**, **32**, **34**–**38**, **40** and **41** exhibited good in vitro activity against both the strains with MIC values ranging from 12.5 to 3.125 μg ml^{-1} while all other compounds have MIC values more than 12.5 μg ml^{-1}. It has been established that 1, 2-*O*-isopropylidene skeleton is crucial for displaying biological activity as it offers lipophilicity in all the molecules and is expected to be hydrolyzed in the cells [19], therefore it is kept intact in all the compounds. The effect of substituent at the nitrogen attached to C-5 in case of furanoses or C-6 in case of pyranoses and *3-O*-substituent in the furanoses on the antitubercular activity potential has been studied in the present work. Glycofuranosylated β-amino alcohols (**15**–**23**) in general exhibited better antitubercular activities than the corre-

Table 1
Antimycobacterial activities of glycosylated amino alcohols (**15**–**23** and **31**–**36**) and amines (**37**–**41**)

Compounds	R	NHR[1]	MIC (μg ml^{-1}) H_{37}Ra	MIC (μg ml^{-1}) H_{37}Rv	Yield (%)
15	CH_3		50	50	90
16	CH_3	$CH_3(CH_2)_6NH$	50	50	90
17	CH_3	$NH(CH_2)_{11}CH_3$	25	25	92
17 [a]	CH_3	$NH(CH_2)_{11}CH_3$	12.5	12.5	90
18	CH_3	$NH(CH_2)_{15}CH_3$	6.25	6.25	92
18 [a]	CH_3	$NH(CH_2)_{15}CH_3$	12.5	12.5	90
19	CH_3	Oleyl amine	25	25	90
20	$CH_2C_6H_5$		50	50	90
21	$CH_2C_6H_5$	$NH(CH_2)_{11}CH_3$	3.12	3.12	92
21 [a]	$CH_2C_6H_5$	$NH(CH_2)_{11}CH_3$	6.25	12.5	90
22	$CH_2C_6H_5$	$NH(CH_2)_{15}CH_3$	12.5	12.5	90
23	$CH_2C_6H_5$	Oleyl amine	12.5	12.5	92
23 [a]	$CH_2C_6H_5$	Oleyl amine	25	25	90
31	–		>100	50	88
32	–		>100	12.5	90
33	–	$CH_3(CH_2)_6NH$	>100	>100	74
34	–	$CH_3(CH_2)_{11}NH$	12.5	<12.5	88
35	–	$CH_3(CH_2)_{15}NH$	25	3.125	79
35 [a]	–	$CH_3(CH_2)_{15}NH$	12.5	<25	76
36	–	Oleyl amine	3.12	6.25	89
37	CH_3	Hexadecyl-NH	12.5	n.d.	91
38	$CH_2C_6H_5$	Dodecyl-NH	12.5	<12.5	50
39	$CH_2C_6H_5$	Oleyl-NH	25	<12.5	55
40	–	Dodecyl-NH	12.5	6.25	58.3
41	–	Hexadecyl-NH	25	<12.5	81

[a] Minor isomer; MIC = minimum inhibitory concentration; n.d. = not determined; MIC of the compounds used as control, ethambutol 1.5–5.0 μg ml^{-1}, rifampicin 0.75 μg ml^{-1}, INH 0.65 μg ml^{-1}.

sponding amino esters (**6–14**; the antitubercular activities of glycosyl amino esters have already been published as preliminary communication in Eur. J. Med. Chem. [15a]) and glycosyl amines (**37–39**). Among amino alcohols, compounds (**18** and **21**) with long alkyl chain (hexadecyl and dodecyl) at C-5 nitrogen showed better inhibition of *M. tuberculosis* $H_{37}Rv$ than those of shorter alkyl chain. Further, on careful analysis of the results it is clear that compound **18** with hexadecyl substituent at *N*- and 3-*O*-methyl substituent in sugar and compound **21** with dodecyl *N*-substituent and 3-*O*-benzyl substituent in sugar; exhibited better antitubercular activity than rest of the compounds in the series. This indicates the role of hydrophobicity in displaying good antitubercular activity. Further shortening (**16**) or increasing the length of aminoalkyl chain at C-5 nitrogen or introduction of unsaturation (**19** and **23**) in it lowers the activity potential. Substitution of nitrogen by cyclopropyl ring (**15** and **20**) did not play any significant role in displaying antitubercular activity. In short the most optimum cumulative number of carbon atoms in substituents at 3-*O*- in furanose ring and amine ranges from 17 to 19.

Among galactopyranosylated series all the amino esters (**25–30**) were inactive up to 12.5 g ml^{-1} (not given in the table). The galatopyranosylated amino alcohols with *N*-dodecyl, hexadecyl and oleyl substituents (**34–36**, respectively) at C-6 here too were the most active compounds having MIC values of <12.5, 3.12 and 6.25 μg ml^{-1}, respectively, against *M. tuberculosis* $H_{37}Rv$. The most active compound in this series was found to be the major diastereoisomer **35** (MIC value of 3.12 μg ml^{-1}) with hexadecyl substituent at C-6 nitrogen while the minor diastereoisomer **35[a]** is less active (MIC value of <25 μg ml^{-1}) against $H_{37}R_V$ strain. Compound **36** with oleyl substituent at C-6 nitrogen is the most active compound against *M. tuberculosis* $H_{37}Ra$ (MIC 3.12 μg ml^{-1}) while against the virulent strain the MIC value was found to be 6.25 μg ml^{-1} only. Here too substitution of nitrogen with cyclopropyl (**31**) and heptyl groups (**33**) led to inactivity against both avirulent and virulent strains. However, compound **32** with *N*-cyclohexyl substituent showed marginal activity against *M. tuberculosis* $H_{37}Rv$ with MIC 12.5 μg ml^{-1}.

To get an insight into the role of hydroxyalkyl substituent in glycosyl amino alcohols we have synthesized glycosyl amines lacking hydroxyethyl substituent at C-5 in furanose and C-6 in pyranose series, respectively. As evident from Table 1 in glycosyl amine sereis compound (**40**) with dodecyl *N*-substituent showed better activity against $H_{37}Rv$ while other compounds (**37–39** and **41**) showed only marginal activity against both $H_{37}Ra$ and $H_{37}Rv$ strains. It is interesting to note that amines as such did not display any significant activity.

5. Conclusion

Glycosyated amino alcohols and amines could be synthesized in good yields starting from simple glycosyluloses in diastereomerically pure forms. Further, glycosyl amino alcohols having C_{12} to C_{18} *N*-alkyl substituents although not screened for enzyme inhibitory activity showed very good in vitro antitubercular activity against *M. tuberculosis* $H_{37}Rv$. This study offers a lead for further optimization in order to get compounds with still lower MIC values.

6. Experimental

6.1. Biology

6.1.1. Activity against M. tuberculosis $H_{37}Ra$ Strain

All the glycosyl amino alcohols synthesized were evaluated for their efficacy against *M. tuberculosis* $H_{37}Ra$ at concentration ranging from 50 μg ml^{-1} to 1.56 μg ml^{-1} using twofold dilutions in the initial screen. Log phase culture of *M. tuberculosis* H_{37} Ra is diluted so as to give final $OD_{550\ nm}$ of 0.05 in Sauton's medium. In 96 well white plate 190 μl of culture is dispensed in each well. A dimethyl sulfoxide (DMSO) solution of test compounds is dispensed to 96 well plates so as to make final test concentration 25 μg ml^{-1} (5 μg test compound is dispensed in 10 μl of DMSO). Then the plate is incubated at 37 °C/5% CO_2 for 5 days. On 5th day 15 μl Alamar blue solution is added to the each well of plate. The plate is again incubated overnight at 37 °C/5% CO_2 incubator. The fluorescence is read on BMG polar star with excitation frequency at 544 nm and emission frequency at 590 nm. The compounds, which were found active (>90% inhibition as compared with control) at this concentration are then tested at 6 serial dilutions starting from 50 to 3.12 μg ml^{-1} [20].

6.1.2. Activity against M. tuberculosis $H_{37}Rv$ strain

Drug susceptibility and determination of MIC of the test compounds/drugs against *M. tuberculosis* $H_{37}Rv$ was performed by agar microdilution method [21] where twofold dilutions of each test compound were added into 7H10 agar supplemented with OADC and organism. A culture of *M. tuberculosis* $H_{37}Rv$ growing on L-J medium was harvested in 0.85% saline with 0.05% Tween-80. A suspension of 1 μg ml^{-1} concentration of extracts/compounds was prepared in DMSO. This suspension was added to (in tubes) 7H10 middle brook's medium (containing 1.7 ml medium and 0.2 ml OADC supplement) at different concentration of compound keeping the volume constant i.e. 0.1 ml. Medium was allowed to cool keeping the tubes in slanting position. These tubes were then incubated at 37 °C for 24 h followed by streaking of *M. tuberculosis* $H_{37}Rv$ (5×10^4 bacilli per tube). These tubes were then incubated at 37 °C. Growth of bacilli was seen after 30 days of incubation. Tubes having the compounds were compared with control tubes where medium alone was incubated with $H_{37}Rv$. The concentration at which complete inhibition of colonies occurred was taken as active concentration of test compound.

6.2. Chemistry

6.2.1. General methods

Thin-layer chromatography was carried out on silica gel (Kiesel 60-F254, Merck) and spots were developed in iodine vapors and also by spraying with 5% sulfuric acid in alcohol followed by heating at 100 °C. Column chromatography was carried out on flash silica gel (230–400 mesh, Merck) using the indicated eluent. IR spectra were recorded as thin films on KBr plates with a Perkin–Elmer 881 spectrophotometer. NMR spectra were recorded on Bruker spectrometers 200 and 300 MHz and reference used was $CDCl_3$. Chemical shifts were given as δ ppm values and '*J*' values were given in Hertz (Hz). Elemental analyses were performed on a Perkin–Elmer 2400 II elemental analyzer. The optical rotations were measured in a 1.0 diameter tube with Jasco dip-140 polarimeter in chloroform. The excess of the reagents or solvents were evaporated under reduced pressure at a bath temperature between the ranges 55 and 60 °C.

*6.2.2. 5-Cyclopropylamino-5,6-dideoxy-6-hydroxymethyl-1,2-*O*-isopropylidene-3-*O*-methyl-β-L-*ido*furanose (**15**)*

To a magnetically stirred slurry of $LiAlH_4$ (0.058 g, 1.52 mmol) in anhydrous THF (5.0 ml), a solution of glycosyl amino ester **6** (0.5 g, 1.52 mmol) in anhydrous THF (5.0 ml) was added drop-wise at 0 °C under inert atmosphere and stirring continued for 30 min at 0 °C. The reaction mixture was further stirred magnetically for 2.5 h at ambient temperature. Excess $LiAlH_4$ was quenched by adding saturated aqueous sodium sulfate solution and the reaction mixture was filtered. The solid cake was washed with THF and the filtrate concentrated under reduced pressure. The later was extracted with chloroform (2 × 25 ml) and water (12.5 ml) and dried (Na_2SO_4), organic layer was concentrated under reduced pressure to give a crude mass, which was chromatographed over SiO_2 column using chloroform/methanol (98:2) as eluent to give the glycosyl amino alcohol **15** as colorless oil. Yield: 90%; $[\alpha]_D = -58.4°$ ($c = 0.625$, $CHCl_3$); IR (KBr): ν_{max} cm^{-1} 3350, 2970 and 2910; MS (FAB): 288 $(M + H)^+$; ^1H NMR (200 MHz, $CDCl_3$): δ 5.93 (d, $J = 3.9$ Hz, 1H, H-1), 4.60 (d, $J = 3.9$ Hz, 1H, H-2), 4.16 (dd, $J = 9.6$ and 3.0 Hz, 1H, H-4), 3.84–3.77 (m, 3H, H-3 and H-7), 3.41 (m, 1H, H-5), 3.39 (s, 3H, $-OCH_3$), 2.65 (m, 1H, NCH), 1.71 (m, 2H, H-6), 1.50 and 1.31 [s, each 3H, $>C(CH_3)_2$], 0.47–0.40 (m, 4H, CH_2); ^{13}C NMR (50 MHz, $CDCl_3$): δ 111.9 [$>C(CH_3)_2$], 105.1 (C-1), 84.4 (C-2), 83.2 (C-4), 81.5 (C-3), 62.7 (C-7), 58.1 (C-5), 57.9 (OCH_3), 31.2 (C-6), 30.7 (NCH), 27.1 and 26.5 [$>C(CH_3)_2$], 7.0 and 6.7 (cyclopropyl ring CH_2,); Anal. Calc. for $C_{14}H_{25}O_5N$: C, 58.54; H, 8.71; N, 4.88. Found: C, 58.92; H, 8.96; N, 5.06.

*6.2.3. 5,6-Dideoxy-5-heptylamino-6-hydroxymethyl-1,2-*O*-isopropylidene-3-*O*-methyl-β-L-*ido*-furanose (**16**)*

Reduction of **7** (0.40 g, 1.03 mmol) with $LiAlH_4$ (0.039 g, 1.03 mmol) and work up as described above gave glycosyl amino alcohol **16** as colorless oil. Yield: 90%; $[\alpha]_D = -34.2°$ ($c = 0.13$, CH_3OH); IR (KBr): ν_{max} cm^{-1} 3332, 2929 and 2858; MS (FAB): 346 $(M + H)^+$; ^1H NMR (200 MHz, $CDCl_3$): δ 5.89 (d, $J = 3.8$ Hz, 1H, H-1), 4.59 (d, $J = 3.8$ Hz, 1H, H-2), 4.22 (dd, $J = 9.6$ and 3.1 Hz, 1H, H-4), 3.85 (m, 2H, H-7), 3.61 (d, $J = 3.1$ Hz, 1H, H-3), 3.37 (s, 3H, $-OCH_3$), 3.18 (m, 1H, H-5), 2.70 (m, 2H, NCH_2), 1.70 (m, 2H, H-6), 1.50–1.32 [m, 16H, $>C(CH_3)_2$ and 5 × CH_2], 0.87 (t, $J = 7.1$ Hz, 3H, CH_3); ^{13}C NMR (50 MHz, $CDCl_3$): δ 111.9 [$>C(CH_3)_2$], 104.9 (C-1), 84.2 (C-2), 81.7 (C-4), 81.6 (C-3), 62.4 (C-7), 57.8 (OCH_3), 57.4 (C-5), 46.9 (NCH_2), 32.1, 30.6, 29.6, 29.5, 27.5 and 22.9 (CH_2's), 27.0 and 26.5 [$>C(CH_3)_2$], 14.3 (CH_2CH_3); Anal. Calc. for $C_{18}H_{35}O_5N$: C, 62.61; H, 10.14; N, 4.06. Found: C, 62.81; H, 9.94; N, 4.16.

*6.2.4. 5,6-Dideoxy-5-dodecylamino-6-hydroxymethyl-1,2-*O*-isopropylidene-3-*O*-methyl-β-L-*ido*-furanose (**17**)*

Reduction of **8** (0.35 g, 0.77 mmol) with $LiAlH_4$ (0.029 g, 0.77 mmol) and work up as described above gave glycosyl amino alcohol **17** as colorless oil. Yield: 92%; $[\alpha]_D = -41.85°$ ($c = 0.16$, CH_3OH); IR (KBr): ν_{max} cm^{-1} 3335, 2943 and 2875; MS (FAB): 416 $(M + H)^+$; ^1H NMR (200 MHz, $CDCl_3$): δ 5.90 (d, $J = 3.8$ Hz, 1H, H-1), 4.59 (d, $J = 3.8$ Hz, 1H, H-2), 4.21 (dd, $J = 9.6$ and 3.0 Hz, 1H, H-4), 3.89 (m, 2H, H-7), 3.61 (d, $J = 3.0$ Hz, 1H, H-3), 3.37 (s, 3H, $-OCH_3$), 3.19 (m, 1H, H-5), 2.69 (m, 2H, NCH_2), 1.50–1.32 [m, 26H, $>C(CH_3)_2$, H-6 and 10 × CH_2], 0.90 (t, $J = 7.1$ Hz, 3H, CH_3); ^{13}C NMR (50 MHz, $CDCl_3$): δ 111.9 [$>C(CH_3)_2$], 104.9 (C-1), 84.2 (C-2), 81.7 (C-4), 81.6 (C-3), 62.4 (C-7), 57.8 (OCH_3), 57.5 (C-5), 46.9 (NCH_2), 32.2, 30.7, 30.6, 29.9, 28.9, 29.7 and 27.5 (CH_2's), 27.0 and 26.5 [$>C(CH_3)_2$], 14.4 (CH_2CH_3); Anal. Calc. for $C_{23}H_{45}O_5N$: C, 66.51; H, 10.84; N, 3.37. Found: C, 66.81; H, 11.14; N, 3.73.

*6.2.5. 5,6-Dideoxy-5-dodecylamino-6-hydroxymethyl-1,2-*O*-isopropylidene-3-*O*-methyl-α-D-glucofuranose (**17^a**)*

Reduction of **8^a** (0.30 g, 0.66 mmol) with $LiAlH_4$ (0.025 g, 0.66 mmol) and work up as described above gave glycosyl amino alcohol **17^a** as colorless oil. IR (KBr): Yield: 90%; $[\alpha]_D = -38.0°$ ($c = 0.15$, CH_3OH); IR (Neat): ν_{max} cm^{-1} 3346, 2928 and 2858; MS (FAB): 416 $(M + H)^+$; ^1H NMR (200 MHz, $CDCl_3$): δ 5.89 (d, $J = 3.8$ Hz, 1H, H-1), 4.58 (d, $J = 3.8$ Hz, 1H, H-2), 4.07 (dd, $J = 7.1$ and 3.0 Hz, 1H, H-4), 3.86 (m, 2H, H-7), 3.75 (d, $J = 3.0$ Hz, 1H, H-3), 3.42 (s, 3H, $-OCH_3$), 3.25 (m, 1H, H-5), 2.82 and 2.65 (each m, 2H, NCH_2), 1.75 (m, 2H, H-6), 1.49-1.25 [m, 26H, $>C(CH_3)_2$ and 10 × CH_2's], 0.88 (t, $J = 6.9$ Hz, 3H, CH_3); ^{13}C NMR (50 MHz, $CDCl_3$): δ 112.7 [$>C(CH_3)_2$], 105.2 (C-1), 83.5 (C-2), 81.3 (C-4), 80.7 (C-3), 61.9 (C-7), 57.6 (OCH_3), 57.1 (C-5), 46.1 (NCH_2), 31.9, 30.6, 30.6, 29.7, 28.7 and 27.5 (CH_2's), 27.0 and 26.8 [$>C(CH_3)_2$], 14.1 (CH_2CH_3); Anal. Calc. for $C_{23}H_{45}O_5N$: C, 66.51; H, 10.84; N, 3.37. Found: C, 66.31; H, 10.54; N, 3.77.

*6.2.6. 5,6-Dideoxy-5-hexadecylamino-6-hydroxymethyl-1,2-*O*-isopropylidene-3-*O*-methyl-β-L-*ido*-furanose (**18**)*

Reduction of glycosyl amino ester **9** (3.50 g, 6.82 mmol) with $LiAlH_4$ (0.259 g, 6.82 mmol) and work up as described

above gave glycosyl amino alcohol **18** as colorless oil. Yield: 92%; $[\alpha]_D = -60.0°$ ($c = 0.05$, $CHCl_3$); IR (KBr): ν_{max} cm^{-1} 3332, 2925 and 2855; MS (FAB): 472 $(M + H)^+$; ^1H NMR (200 MHz, $CDCl_3$): δ 5.91 (d, $J = 3.6$ Hz, 1H, H-1), 4.60 (d, $J = 3.6$ Hz, 1H, H-2), 4.22 (dd, $J = 9.6$ and 2.9 Hz, 1H, H-4), 3.85 (m, 2H, H-7), 3.61 (d, $J = 2.9$ Hz, 1H, H-3), 3.38 (s, 3H, $-OCH_3$), 3.18 (m, 1H, H-5), 2.69 (m, 3H, NH and NCH_2), 1.77 (m, 2H, H-6), 1.49–1.25 [m, 34H, $>C(CH_3)_2$ and 14 × CH_2's], 0.88 (t, $J = 6.3$ Hz, 3H, CH_3); ^{13}C NMR (50 MHz, $CDCl_3$): δ 111.9 [$>C(CH_3)_2$], 104.9 (C-1), 84.2 (C-2), 81.7 (C-4), 81.6 (C-3), 62.5 (C-7), 57.8 (OCH_3), 57.5 (C-5), 46.9 (NCH_2), 32.3, 30.7, 30.0, 30.0, 29.9, 29.7, 29.5, 27.6 and 23.06 (CH_2's), 27.0 and 26.5 [$>C(CH_3)_2$], 14.4 (CH_2CH_3); Anal. Calc. for $C_{27}H_{53}O_5N$: C, 68.79; H, 11.25; N, 2.97. Found: C, 68.99; H, 11.65; N, 2.89.

*6.2.7. 5,6-Dideoxy-5-hexadecylamino-6-hydroxymethyl-1,2-*O*-isopropylidene-3-*O*- methyl-α-D-gluco-furanose (18[a])*

Reduction of **9[a]** (0.33 g, 0.79 mmol) with $LiAlH_4$ (0.03 g, 0.79 mmol) and work up as described above gave glycosyl amino alcohol **18[a]** as colorless oil. Yield: 90%; $[\alpha]_D = -33.5°$ ($c = 0.12$, CH_3OH); IR (KBr): ν_{max} cm^{-1} 3318, 2926 and 2854; MS (FAB): 472 $(M + H)^+$; ^1H NMR (200 MHz, $CDCl_3$): δ 5.89 (d, $J = 3.8$ Hz, 1H, H-1), 4.58 (d, $J = 3.8$ Hz, 1H, H-2), 4.11 (dd, $J = 6.8$ and 3.1 Hz, 1H, H-4), 3.86 (m, 2H, H-7), 3.77 (d, $J = 3.1$ Hz, 1H, H-3), 3.42 (s, 3H, $-OCH_3$), 3.25 (m, 1H, H-5), 2.70 (m, 2H, NCH_2), 1.49–1.25 [m, 34H, H-6, $>C(CH_3)_2$ and 14 × CH_2's], 0.88 (t, $J = 6.5$ Hz, 3H, CH_3); ^{13}C NMR (50 MHz, $CDCl_3$): δ 111.5 [$>C(CH_3)_2$], 104.4 (C-1), 84.5 (C-2), 81.1 (C-4), 80.9 (C-3), 62.6 (C-7), 57.5 (OCH_3), 57.3 (C-5), 47.7 (NCH_2), 31.8, 31.7, 30.2, 29.6, 29.4, 29.3, 27.1 and 22.6 (CH_2's), 27.6 and 26.1 [$>C(CH_3)_2$], 14.0 (CH_2CH_3); Anal. Calc. for $C_{27}H_{53}O_5N$: C, 68.79; H, 11.25; N, 2.97. Found: C, 68.49; H, 11.05; N, 2.57.

*6.2.8. 5,6-Dideoxy-6-hydroxymethyl-1,2-*O*-isopropylidene-3-*O*-methyl-5-oleylamino-β-L-*ido*-furanose (19)*

Reduction of glycosyl amino ester **10** (0.50 g, 0.93 mmol) with $LiAlH_4$ (0.035 g, 0.93 mmol) and work up as described above gave glycosyl amino alcohol **19** as colorless oil. Yield: 90%; $[\alpha]_D = -32.0°$ ($c = 0.125$, CH_3OH); IR (KBr): ν_{max} cm^{-1} 3402, 2997 and 2856; MS (FAB): 498 $(M + H)^+$; ^1H NMR (200 MHz, $CDCl_3$): δ 5.90 (d, $J = 3.8$ Hz, 1H, H-1), 5.36 (m, 2H, HC=CH), 4.59 (d, $J = 3.8$ Hz, 1H, H-2), 4.22 (dd, $J = 9.6$ and 3.0 Hz, 1H, H-4), 3.87 (d, $J = 3.0$ Hz, 1H, H-3), 3.77 (m, 2H, H-7), 3.38 (s, 3H, $-OCH_3$), 3.20 (m, 1H, H-5), 2.71 (m, 2H, NCH_2), 2.01 (m, 4H, $CH_2HC{=}CHCH_2$), 1.50–1.25 [m, 30 H, $>C(CH_3)_2$, H-6, and 12 × CH_2's], 0.88 (t, $J = 6.3$ Hz, 3H, CH_3); ^{13}C NMR (50 MHz, $CDCl_3$): δ 130.2 (C=CH), 111.9 [$>C(CH_3)_2$], 104.9 (C-1), 84.1 (C-2), 81.7 (C-4), 81.6 (C-3), 62.5 (C-7), 57.8 (OCH_3), 57.5 (C-5), 46.9 ($-NCH_2$), 32.9, 32.2, 30.7, 29.9, 29.6, 27.6 and 23.0 (CH_2's), 27.0 and 26.5 [$>C(CH_3)_2$], 14.4 (CH_2CH_3); Anal. Calc. for $C_{29}H_{55}O_5N$: C, 70.02; H, 11.07; N, 2.82. Found: C, 70.07; H, 11.22; N, 3.17.

*6.2.9. 3-*O*-Benzyl-5-cyclopropylamino-5,6-dideoxy-6-hydroxymethyl-1,2-*O*-isopropylidene-β-L-*ido*-furanose (20)*

Reduction of glycosyl amino ester **11** (0.55 g, 1.36 mmol) with $LiAlH_4$ (0.052 g, 1.36 mmol) and work up as described above gave glycosyl amino alcohol **20** as colorless oil. Yield: 90%; $[\alpha]_D = -88.68°$ ($c = 0.437$, $CHCl_3$); IR (KBr): ν_{max} cm^{-1} 3350, 2970 and 2910; MS (FAB): 364 $(M + H)^+$; ^1H NMR (200 MHz, $CDCl_3$): δ 7.34 (m, 5H, Ar-H), 5.95 (d, $J = 3.9$ Hz, 1H, H-1), 4.70 (d, $J = 11.8$ Hz, 1H, $-OCH_APh$), 4.65 (d, $J = 3.9$ Hz, 1H, H-2), 4.40 (d, $J = 11.8$ Hz, 1H, $-OCH_BPh$), 4.16 (dd, $J = 9.6$ and 3.1 Hz, 1H, H-4), 3.82 (d, $J = 3.1$ Hz, 1H, H-3), 3.76 (m, 2H, H-7), 3.30 (m, 1H, H-5), 2.43 (m, 1H, NCH), 1.43–1.33 [m, 8H, $>C(CH_3)_2$ and H-6], 0.47–0.40 (m, 4H, cyclopropyl ring CH_2); ^{13}C NMR (50 MHz, $CDCl_3$): δ 137.3, 128.9, 128.6 and 128.5 (Ar-C), 111.9 [$>C(CH_3)_2$], 105.1 (C-1), 83.1 (C-2), 82.1 (C-4), 81.8 (C-3), 72.1 (OCH_2Ph), 63.0 (C-7), 58.1 (C-5), 30.8 (C-6), 28.9 (CH), 27.0 and 26.5 [$>C(CH_3)_2$], 6.8 (m, 4H, 2 × CH_2); Anal. Calc. for $C_{20}H_{29}O_5N$: C, 66.11; H, 7.99; N, 3.86. Found: C, 66.44; H, 8.40; N, 4.09.

*6.2.10. 3-*O*-Benzyl-5,6-dideoxy-5-dodecylamino-6-hydroxymethyl-1,2-*O*-isopropylidene-β-L-*ido*-furanose (21)*

Reduction of glycosyl amino ester **12** (3.80 g, 7.13 mmol) with $LiAlH_4$ (0.271 g, 7.13 mmol) and work up as described above gave glycosyl amino alcohol **21** as colorless solid. Yield: 92%; $[\alpha]_D = -58.66°$ ($c = 0.075$, $CHCl_3$); IR (KBr): ν_{max} cm^{-1} 3330, 2929 and 2857; MS (FAB): 492 $(M + H)^+$; ^1H NMR (200 MHz, $CDCl_3$): δ 7.32 (m, 5H, Ar-H), 5.93 (d, $J = 3.8$ Hz, 1H, H-1), 4.69 (d, $J = 11.7$ Hz, 1H, $-OCH_AAr$), 4.64 (d, $J = 3.8$ Hz, 1H, H-2), 4.39 (d, $J = 11.7$ Hz, 1H, $-OCH_BAr$), 4.19 (dd, $J = 9.6$ and 3.0 Hz, 1H, H-4), 3.82 (d, $J = 3.0$ Hz, 1H, H-3), 3.71 (m, 2H, H-7), 3.24 (m, 1H, H-5), 2.68 (t, $J = 6.3$ Hz, 2H, NCH_2), 1.50–1.25 [m, 28H, $>C(CH_3)_2$, H-6 and 10 × CH_2's], 0.87 (t, $J = 6.7$ Hz, 3H, CH_2CH_3); ^{13}C NMR (50 MHz, $CDCl_3$): δ 137.3, 128.9, 128.5 and 128.4 (Ar-C), 112.0 [$>C(CH_3)_2$], 105.0 (C-1), 82.3 (C-2), 81.7 (C-4), 81.6 (C-3), 72.1 (OCH_2Ar), 62.8 (C-7), 57.5 (C-5), 46.5 (NCH_2), 32.3, 30.8, 30.0, 29.9, 29.7, 29.2, 27.6, 23.0 (CH_2's), 27.1 and 26.6 [$>C(CH_3)_2$], 14.4 (CH_2CH_3); Anal. Calc. for $C_{29}H_{49}O_5N$: C, 70.87; H, 9.98; N, 2.85. Found: C, 70.46; H, 9.78; N, 3.05.

*6.2.11. 3-*O*-Benzyl-5,6-dideoxy-5-dodecylamino-6-hydroxymethyl-1,2-*O*-isopropylidene-α-D-gluco-furanose (21[a])*

Reduction of glycosyl amino ester **12[a]** (0.33 g, 0.62 mmol) with $LiAlH_4$ (0.024 g, 0.62 mmol) and work up as described above gave glycosyl amino alcohol **21[a]** as colorless oil. Yield: 90%; $[\alpha]_D = -29.6°$ ($c = 0.12$, CH_3OH); IR (KBr): ν_{max} cm^{-1} 3335, 2938 and 2851; MS (FAB): 492 $(M + H)^+$; ^1H NMR (200 MHz, $CDCl_3$): δ 7.33 (m, 5H, Ar-H), 5.91 (d, $J = 3.8$ Hz, 1H, H-1), 4.68 (d, $J = 11.8$ Hz, 1H, $-OCH_AAr$), 4.62 (d, $J = 3.8$ Hz, 1H, H-2), 4.41 (d, $J = 11.8$ Hz, 1H, $-OCH_BAr$), 4.19 (dd, $J = 7.0$ and 3.0 Hz, 1H, H-4), 3.82 (d, $J = 3.0$ Hz, 1H, H-3), 3.86 (m, 2H, H-7), 3.25 (m, 1H, H-5), 2.66 (t,

J = 6.4 Hz, 2H, NCH_2), 1.50–1.25 [m, 28H, >C(CH_3)$_2$, H-6 and 10 × CH_2's], 0.87 (t, J = 6.7 Hz, 3H, CH_2CH_3); Anal. Calc. for $C_{29}H_{49}O_5N$: C, 70.87; H, 9.98; N, 2.85. Found: C, 71.02; H, 10.18; N, 3.00.

*6.2.12. 3-*O*-Benzyl-5,6-dideoxy-5-hexadecylamino-6-hydroxymethyl-1,2-*O*-isopropylidene-β-L-*ido*-furanose (**22**)*

Reduction of glycosyl amino ester **13** (0.80 g, 1.36 mmol) with $LiAlH_4$ (0.052 g, 1.36 mmol) and work up as described above gave glycosyl amino alcohol **22** as colorless oil; Yield: 90%; $[\alpha]_D$ = –27.00° (c = 0.50, $CHCl_3$); FAB MS: 548 (M + H)$^+$; IR (KBr): ν_{max} cm^{-1} 3346, 2927 and 2856; ^{1}H NMR (200 MHz, $CDCl_3$): δ 7.30 (m, 5H, Ar-H), 5.94 (d, J = 3.7 Hz, 1H, H-1), 4.69 (d, J = 11.6 Hz, 1H, –OCH_APh), 4.64 (d, J = 3.7 Hz, 1H, H-2), 4.40 (d, J = 11.6 Hz, 1H, –OCH_BPh), 4.25 (dd, J = 9.4 and 2.9 Hz, 1H, H-4), 3.85 (d, J = 2.9 Hz, 1H, H-3), 3.74 (m, 2H, H-7), 3.34 (m, 1H, H-5), 2.79 (m, 2H, NCH_2), 1.53–1.25 [m, 34H, >C(CH_3)$_2$, H-6, 14 × CH_2's], 0.87 (t, J = 6.7 Hz, 3H, –CH_2CH_3); ^{13}C NMR (50 MHz, $CDCl_3$): δ 136.5, 129.1, 128.9 and 128.78 (Ar-C), 113.0 [>C(CH_3)$_2$], 105.4 (C-1), 82.2 (C-2), 81.4 (C-4), 79.72 (C-3), 72.3 (–OCH$_2$Ph), 59.9 (C-7), 58.4 (C-5), 47.3 (NCH_2), 32.2, 30.0, 29.9, 29.8, 29.7, 29.4, 27.44 and 27.2 (CH_2'$_S$), 27.0 [2 × >C(CH_3)$_2$], 14.4 (CH_2CH_3); Anal. Calc. for $C_{33}H_{57}O_5N$: C, 72.39; H, 10.42; N, 2.56. Found: C, 72.19; H, 10.17; N, 2.51.

*6.2.13. 3-*O*-Benzyl-5,6-dideoxy-6-hydroxymethyl-1,2-*O*-isopropylidene-5-oleylamino-β-L-*ido*-furanose (**23**)*

Reduction of glycosyl amino ester **14** (4.3 g, 6.99 mmol) with $LiAlH_4$ (0.270 g, 6.99 mmol) and work up as described above gave glycosyl amino alcohol **23** as colorless solid. Yield: 92%; $[\alpha]_D$ = –35.33° (c = 0.15, CH_3OH); IR (KBr): ν_{max} cm^{-1} 3396, 2926 and 2856; MS (FAB): 574 (M + H)$^+$; ^{1}H NMR (200 MHz, $CDCl_3$): δ 7.32 (m, 5H, Ar-H), 5.93 (d, J = 3.7 Hz, 1H, H-1), 5.34 (m, 2H, HC=CH), 4.69 (d, J = 11.9 Hz, 1H, –OCH_APh), 4.62 (d, J = 3.7 Hz, 1H, H-2), 4.39 (d, J = 11.9 Hz, 1H, –OCH_BPh), 4.20 (dd, J = 9.6 and 2.8 Hz, 1H, H-4), 3.82 (d, J = 2.8 Hz, 1H, H-3), 3.75 (m, 2H, H-7), 3.21 (m, 1H, H-5), 2.69 (m, 2H, NCH_2), 2.00 (m, 4H, CH_2HC=CHCH_2), 1.50–1.26 [m, 32 H, >C(CH_3)$_2$, H-6 and 12 × CH_2'$_S$], 0.88 (t, J = 6.3 Hz, 3H, CH_3); ^{13}C NMR (50 MHz, $CDCl_3$): δ 137.3 (Ar-C), 130.8, 128.9, 128.5 and 128.4 (Ar-CH), 111.9 [>C(CH_3)$_2$], 105.0 (C-1), 82.3 (C-2), 81.7 (C-4), 81.6 (C-3), 72.1 (–OCH$_2$Ph), 62.7 (C-7), 57.4 (C-5), 46.5 (–NCH_2), 32.9, 32.2, 30.8, 30.1, 30.0, 29.9, 29.6, 29.3, 27.6 and 23.0 (CH_2's), 27.1 and 26.6 [>C(CH_3)$_2$], 14.4 (CH_2CH_3); Anal. Calc. for $C_{35}H_{59}O_5N$: C, 73.30; H, 10.30; N, 2.44. Found: C, 72.95; H, 10.05; N, 2.59.

*6.2.14. 3-*O*-Benzyl-5,6-dideoxy-6-hydroxymethyl-1,2-*O*-isopropylidene-5-oleylamino-α-D-gluco-furanose (**23**^{a})*

Reduction of glycosyl amino ester **14**^{a} (1.0 g, 1.63 mmol) with $LiAlH_4$ (0.062 g, 1.63 mmol) and work up as described above gave glycosyl amino alcohol **23**^{a} as colorless oil. Yield: 90%; $[\alpha]_D$ = –34.4° (c = 0.125, CH_3OH); IR (KBr): ν_{max} cm^{-1} 3347, 2930 and 2957; MS (FAB): 574 (M + H)$^+$; ^{1}H NMR (200 MHz, $CDCl_3$): δ 7.34 (m, 5H, Ar-H), 5.92 (d, J = 3.9 Hz, 1H, H-1), 5.34 (m, 2H, HC=CH), 4.71 (d, J = 11.6 Hz, 1H, –OCH_APh), 4.64 (d, J = 3.9 Hz, 1H, H-2), 4.47 (d, J = 11.6 Hz, 1H, –OCH_BPh), 4.14 (dd, J = 7.3 and 3.2 Hz, 1H, H-4), 3.98 (d, J = 3.2 Hz, 1H, H-3), 3.85 (m, 2H, H-7), 3.30 (m, 1H, H-5), 2.68–2.20 (m, 6H, NCH_2 and H_2CHC=CHCH_2), 1.49–1.25 [m, 32 H, >C(CH_3)$_2$, H-6 and 12 × CH_2'$_S$], 0.88 (t, J = 6.6 Hz, 3H, CH_3); ^{13}C NMR (50 MHz, $CDCl_3$): δ 137.0 (Ar-C), 130.6, 128.6, 128.2 and 128.1 (Ar-CH), 112.0 [>C(CH_3)$_2$], 104.9 (C-1), 82.2 (C-2), 81.6 (C-4), 81.4 (C-3), 72.2 (–OCH$_2$Ph), 62.5 (C-7), 57.5 (C-5), 46.4 (–NCH_2), 32.4, 32.1, 30.7, 30.2, 30.0, 29.7, 29.4, 29.2, 27.3 and 23.0 (CH_2's), 27.1 and 26.5 [2 × >C(CH_3)$_2$], 14.3 (CH_2CH_3); Anal. Calc. for $C_{35}H_{59}O_5N$: C, 73.30; H, 10.30; N, 2.44. Found: C, 73.68; H, 10.56; N, 2.67.

*6.2.15. 6-Cyclopropylamino-6-deoxy-1,2:3,4-di-*O*-isopropylidene-7-hydroxymethyl-β-L-glycero-D-galacto-heptopyranose (**31**)*

Reduction of glycosyl amino ester **25** (0.50 g, 1.29 mmol) with $LiAlH_4$ (0.049 g, 1.29 mmol) and work up as described above gave glycosyl amino alcohol **31** as colorless oil. Yield: 88%; $[\alpha]_D^{20}$ = –67.4° (c = 0.19, $CHCl_3$); MS (FAB) = m/z 344 (M + H)$^+$; IR (Neat): ν_{max} cm^{-1} 3329, 3086, 2986, 2935 and 1378; ^{1}H NMR (200 MHz, $CDCl_3$) δ 5.57 (d, J = 5.0 Hz, 1H, H-1), 4.60 (dd, J = 7.9 and 2.3 Hz, 1H, H-3), 4.34 (dd, J = 5.0 and 2.3 Hz, 1H, H-2), 4.25 (d, J = 7.9 Hz, 1H, H-4), 3.90–375 (m, 3H, H-5 and H-8), 3.55 (bs, 1H, OH), 3.27–3.17 (m, 1H, H-6), 2.36–2.27 (m, 1H, NCH), 2.10–1.95 (m, 1H, H-7$_A$), 1.74–1.62 (m, 1H, H-7$_B$), 1.52, 1.46 and 1.33 [s, 3H, 3H, 6H, 2 × C(CH_3)$_2$], 0.52–0.46 (m, 4H, cyclopropyl ring CH_2's); ^{13}C NMR (50 MHz, $CDCl_3$) δ 109.7 and 109.1 [2 × C(CH_3)$_2$], 97.0 (C-1), 71.5 (C-3), 71.3 (C-2), 70.9 (C-4), 69.2(C-5), 62.5 (C-8), 58.8 (C-6), 29.4 (C-7), 29.0 (NCH), 26.3, 25.3 and 24.8 [2 × C(CH_3)$_2$], 6.9 and 6.0 (cyclopropyl ring CH_2's); Anal. Calc. for $C_{17}H_{29}NO_6$: C, 59.47; H, 8.45; N, 4.08. Found: C, 59.50; H, 8.47; N, 4.09.

*6.2.16. 6-Cyclohexylamino-6-deoxy-1,2:3,4-di-*O*-isopropylidene-7-hydroxymethyl-β-L-glycero-D-galacto-heptopyranose (**32**)*

Reduction of glycosyl amino ester **26** (0.70 g, 1.64 mmol) with $LiAlH_4$ (0.063 g, 1.64 mmol) and work up as described above gave glycosyl amino alcohol **32** as colorless oil. Yield: 90%; $[\alpha]_D^{20}$ = –76.0° (c = 0.1, $CHCl_3$); MS (FAB) = m/z 386 (M + H)$^+$; IR (Neat): ν_{max} cm^{-1} 3470, 2992, 2932 and 1379; ^{1}H NMR (200 MHz, $CDCl_3$): δ 5.54 (d, J = 5.0 Hz, 1H, H-1), 4.61 (dd, J = 7.8 and 2.4 Hz, 1H, H-3), 4.33 (dd, J = 5.0 and 2.4 Hz, 1H, H-2), 4.24 (dd, J = 7.8 and 1.5 Hz, 1H, H-4), 3.96-3.69 (m, 3H, H-5 and H-8), 3.28-3.19 (m, 1H, H-6), 2.67–2.57 (m, 1H, NCH), 2.1–1.68 (m, 6H, H-7 and 2 × NCHCH_2), 1.54, 1.44 and 1.33 [s, 3H, 3H, 6H, 2 × C(CH_3)$_2$], 1.25–1.05 (m, 6H, cyclohexyl ring protons); ^{13}C NMR (50 MHz, $CDCl_3$): δ 109.8 and 109.2 [2 × C(CH_3)$_2$], 97.1 (C-1), 71.5 (C-3), 71.4 (C-2), 70.9 (C-4), 69.5 (C-5), 62.1

(C-8), 54.8 (C-6), 34.7, 33.4, 29.9, 25.6 and 25.5 (CH_2's), 30.7 (NCH), 26.4, 25.4 and 24.9 [2 × C(CH_3)$_2$]; Anal. Calc. for $C_{20}H_{35}NO_6$: C, 62.34; H, 9.09; N, 3.64. Found: C, 62.28; H, 8.98; N, 3.62.

*6.2.17. 6-Deoxy-1,2:3,4-di-*O*-isopropylidene-6-heptylamino-7-hydroxymethyl-β-L-glycero-D-galacto-heptopyranose (**33**)*

Reduction of glycosyl amino ester **27** (0.90 g, 2.03 mmol) with $LiAlH_4$ (0.077 g, 2.03 mmol) and work up as described above gave glycosyl amino alcohol **33** as colorless oil. Yield: 74%; $[\alpha]_D^{20}$ = –68.2° (*c* = 0.09, $CHCl_3$); MS (FAB) = *m/z* 402 (M + H)$^+$; IR (Neat): ν_{max} cm^{-1} 3330, 2928, 2859 and 1378; ^{1}H NMR (200 MHz, $CDCl_3$): δ 5.55 (d, *J* = 5.0 Hz, 1H, H-1), 4.61 (dd, *J* = 8.0 and 2.0 Hz, 1H, H-3), 4.34 (dd, *J* = 5.0 and 2.0 Hz, 1H, H-2), 4.22 (d, *J* = 8.0 Hz, 1H, H-4), 3.86–3.82 (m, 3H, H-5 and H-8), 3.12 (m, 1H, H-6), 2.8–2.5 (m, 2H, NHCH_2), 2.0-1.61 (m, 2H, H-7), 1.54 and 1.44 [s, each 3H, C(CH_3)$_2$], 1.32–1.25 (m, 16H, C(CH_3)$_2$ and 5 × CH_2), 0.85 (t, *J* = 6.5 Hz, 3H, CH_2CH_3); ^{13}C NMR (50 MHz, $CDCl_3$): δ 109.3 and 108.7 [2 × *C*(CH_3)$_2$], 96.6 (C-1), 71.0 (C-3), 70.9 (C-2), 70.6 (C-4), 67.9 (C-5), 62.0 (C-8), 57.5 (C-6), 45.5 (NCH_2), 31.8, 30.1, 29.2, 27.6, 27.2 and 22.6 (CH_2's), 25.9, 25.0 and 24.4 [2 × C(*C*H_3)$_2$], 14.1 (CH_2CH_3); Anal. Calc. for $C_{21}H_{39}NO_6$: C, 62.80; H, 9.73; N, 3.49. Found: C, 62.82; H, 9.73; N, 3.50.

*6.2.18. 6-Deoxy-6-dodecylamino-1,2:3,4-di-O-isopropylidene-7-hydroxymethyl-β-L-glycero-D-galacto-heptopyranose (**34**)*

Reduction of glycosyl amino ester **28** (1.5 g, 2.92 mmol) with $LiAlH_4$ (0.11 g, 2.92 mmol) and work up as described above gave glycosyl amino alcohol **34** as colorless oil. Yield: 88%; $[\alpha]_D^{20}$ = –36.8° (*c* = 0.13, $CHCl_3$); MS (FAB) = *m/z* 472 (M + H)$^+$; IR (Neat): ν_{max} cm^{-1} 3410, 2925, 2854 and 1377; ^{1}H NMR (200 MHz, $CDCl_3$): δ 5.54 (d, *J* = 4.9 Hz, 1H, H-1), 4.61 (dd, *J* = 7.7 and 2.0 Hz, 1H, H-3), 4.33 (dd, *J* = 4.9 and 2.0 Hz, 1H, H-2), 4.23 (d, *J* = 7.9 Hz, 1H, H-4), 3.86–3.82 (m, 3H, H-5 and H-8), 3.13–3.05 (m, 1H, H-6), 2.77–2.50 (m, 2H, NHCH_2), 1.98–1.61 (m, 2H, H-7), 1.54 and 1.44 [s, each 3H, C(CH_3)$_2$], 1.33–1.25 (m, 26H, C(CH_3)$_2$ and 10 × CH_2), 0.86 (t, *J* = 6.6 Hz, 3H, CH_2CH_3); ^{13}C NMR (50 MHz, $CDCl_3$): δ 109.8 and 109.2 [2 × *C*(CH_3)$_2$], 97.0 (C-1), 71.4 (C-3), 71.3 (C-2), 70.9 (C-4), 68.3 (C-5), 62.4 (C-8), 58.0 (C-6), 46.0 , 32.3, 30.5, 30.0, 29.9, 29.7, 28.0, 27.6, 23.0 (CH_2's), 26.3, 25.4 and 24.8 [2 × C(*C*H_3)$_2$], 14.5 (CH_2CH_3); Anal. Calc. for $C_{26}H_{49}NO_6$: C, 66.24; H, 10.40; N, 2.97. Found: C, 66.18; H, 10.36; N, 3.0.

*6.2.19. 6-Deoxy-1,2:3,4-di-*O*-isopropylidene-6-hexadecylamino-7-hydroxymethyl-β-L-glycero-D-galacto-heptopyranose (**35**)*

Reduction of glycosyl amino ester **29** (1.5 g, 2.63 mmol) with $LiAlH_4$ (0.10 g, 2.63 mmol) and work up as described above gave glycosyl amino alcohol **35** as colorless oil. Yield: 79%; $[\alpha]_D^{20}$ = –62.1° (*c* = 0.18, $CHCl_3$); MS (FAB) = *m/z* 528 (M + H)$^+$; IR (Neat): ν_{max} cm^{-1} 3326, 2926, 2855 and 1378; ^{1}H NMR (200 MHz, $CDCl_3$): δ 5.55 (d, *J* = 5.0 Hz, 1H, H-1), 4.61 (dd, *J* = 7.8 and 2.3 Hz, 1H, H-3), 4.33 (dd, *J* = 5.0 and 2.3 Hz, 1H, H-2), 4.22 (d, *J* = 7.9 Hz, 1H, H-4), 3.89–3.81 (m, 3H, H-5 and H-8), 3.13–3.09 (m, 1H, H-6), 2.78–2.52 (m, 2H, NHCH_2), 2.0–1.61 (m, 2H, H-7), 1.54 and 1.44 [s, each 3H, C(CH_3)$_2$], 1.32–1.25 (m, 34H, C(CH_3)$_2$, 14 × CH_2), 0.88 (t, *J* = 6.5 Hz, 3H, –CH_2CH_3); ^{13}C NMR (50 MHz, $CDCl_3$): δ 109.8 and 109.3 [2 × *C*(CH_3)$_2$], 96.9 (C-1), 71.5 (C-3), 71.4 (C-2), 70.9 (C-4), 68.2 (C-5), 62.0 (C-8), 57.8 (C-6), 46.8 (NCH_2), 32.3, 30.1, 29.9, 29.8, 29.7, 28.2, 27.6, 23.1 (CH_2's), 26.4, 25.3 and 24.7 [2 × C(*C*H_3)$_2$], 14.5 (CH_2CH_3); Anal. Calc. for $C_{30}H_{57}NO_6$: C, 68.31; H, 10.81; N, 2.65. Found: C, 68.28; H, 10.79; N, 2.59.

*6.2.20. 6-Deoxy-1,2:3,4-di-*O*-isopropylidene-6-hexadecylamino-7-hydroxymethyl-α-L-glycero-D-galacto-heptopyranose (**35**^{a})*

Reduction of glycosyl amino ester **29**^{a} (0.50 g, 0.87 mmol) with $LiAlH_4$ (0.033 g, 0.87 mmol) and work up as described above gave glycosyl amino alcohol **35**^{a} as colorless oil. Yield: 76%; $[\alpha]_D^{20}$ = –60.0° (*c* = 0.063, $CHCl_3$); MS (FAB) = *m/z* 528 (M + H)$^+$; IR (Neat): ν_{max} cm^{-1} 3404, 2925, 2855 and 1378; ^{1}H NMR (200 MHz, $CDCl_3$): δ 5.54 (d, *J* = 5.0 Hz, 1H, H-1), 4.63 (dd, *J* = 7.9 and 2.0 Hz, 1H, H-3), 4.50 (d, *J* = 7.9 Hz, 1H, H-4), 4.33 (dd, *J* = 5.0 and 2.0 Hz, 1H, H-2), 3.85–3.69 (m, 3H, H-5 and H-8), 3.11 (m, 1H, H-6), 2.41–1.97 (m, 2H, NHCH_2), 1.71 (m, 2H, H-7), 1.52 and 1.46 [s, each 3H, C(CH_3)$_2$], 1.34–1.25 [m, 34H, C(CH_3)$_2$, 14 × CH_2], 0.88 (t, *J* = 6.5 Hz, 3H, CH_2CH_3); ^{13}C NMR (50 MHz, $CDCl_3$): δ 110.0 and 109.5 [2 × *C*(CH_3)$_2$], 97.0 (C-1), 71.9 (C-3), 71.3 (C-4), 70.6 (C-2), 67.8 (C-5), 62.8 (C-8), 59.9 (C-6), 45.6 (NCH_2), 32.3, 30.1, 29.7, 29.5, 28.5, 27.4, 27.1, 24.3, 23.0 (CH_2's), 26.4, 25.2 and 24.7 [2 × C(*C*H_3)$_2$], 14.5 (CH_2CH_3); Anal. Calc. for $C_{30}H_{57}NO_6$: C, 68.31; H, 10.81; N, 2.65. Found: C, 67.97; H, 10.29; N, 2.46.

*6.2.21. 6-Deoxy-1,2:3,4-di-*O*-isopropylidene-7-hydroxymethyl-6-oleylamino-β-L-glycero-D-galacto-heptopyranose (**36**)*

Reduction of glycosyl amino ester **30** (1.0 g, 1.68 mmol) with $LiAlH_4$ (0.064 g, 1.68 mmol) and work up as described above gave glycosyl amino alcohol **36** as colorless oil. Yield: 89%; $[\alpha]_D^{20}$ = –35.2° (*c* = 0.13, $CHCl_3$); MS (FAB) = *m/z* 554 (M + H)$^+$; IR (Neat): ν_{max} cm^{-1} 3400, 2928, 2857 and 1381; ^{1}H NMR (200 MHz, $CDCl_3$): δ 5.55 (d, *J* = 5.0 Hz, 1H, H-1), 5.37–5.32 (m, 2H, CH=CH), 4.61 (dd, *J* = 7.9 and 2.3 Hz, 1H, H-3), 4.34 (dd, *J* = 5.0 and 2.3 Hz, 1H, H-2), 4.22 (d, *J* = 7.9 Hz, 1H, H-4), 3.92–3.74 (m, 3H, H-5 and H-8), 3.14–3.05 (m, 1H, H-6), 2.80–2.68 and 2.59–2.47 (m, each 1H, NHCH_A and NHCH_B), 2.0–1.87 (m, 5H, CH_2CH=CHCH_2 and H-7$_A$), 1.71–1.60 (m, 1H, H-7$_B$) 1.54 and 1.44 [s, each 3H, C(CH_3)$_2$], 1.32–1.25 (m, 30H, C(CH_3)$_2$ and 12 × CH_2), 0.88 (t, *J* = 6.7 Hz, 3H, CH_2CH_3); ^{13}C NMR (50 MHz, $CDCl_3$): δ 130.3 (CH=CH), 109.9 and 109.2 [2 × *C*(CH_3)$_2$], 97.0 (C-1), 71.4 (C-3), 71.3(C-2), 70.9

(C-4), 68.4 (C-5), 62.6 (C-8), 57.9 (C-6), 46.0 (NCH_2), 32.3, 30.6, 30.2, 30.1, 29.9, 29.7, 27.9, 27.7, 27.6 and 23.0 (CH_2's), 26.4, 25.4 and 24.8 [2 × C(CH_3)$_2$], 14.5 (CH_2CH_3); Anal. Calc. for $C_{32}H_{59}NO_6$: C, 69.44; H, 10.67; N, 2.53. Found: C, 69.38; H, 10.59; N, 2.50.

*6.2.22. 5-Deoxy-5-hexadecylamino-1,2-*O*-isopropylidene-3-*O*-methyl-α-D-xylofuranose (**37**)*

To the magnetically stirred slurry of 4 Å M.S. (1.5 g) in dry chloroform, compound **1** (500 mg, 2.47 mmol) and hexadecylamine (488 mg, 2.47 mmol) was added at 0 °C, stirring continued for 30 min at same temperature followed by 6 h at ambient temperature till the disappearance of aldehyde. Reaction mixture was evaporated under reduced pressure and crude obtained (900 mg) was dissolved in methanol (4.5 ml). Sodium borohydride (94 mg, 2.47 mmol) was added at 0 °C and stirred for 3 h at ambient temperature. Excess of sodium borohydride was quenched by adding saturated ammonium chloride solution and the reaction mixture was filtered. The solid cake was washed with more methanol and the filtrate evaporated and extracted with ethyl acetate (50 ml) and water (12.5 ml). The organic layer dried (Na_2SO_4) and evaporated under reduced pressure to give a crude mass which was chromatographed over SiO_2 column using hexane/ethyl acetate (3:2) as eluant to give **37** as colorless oil. Yield: 91%; $[\alpha]_D$ = –56° (*c* = 0.10, chloroform); MS (FAB) = *m/z* 428 (M + H)$^+$; IR (Neat): ν_{max} cm^{-1} 3369, 1446; ^{1}H NMR (200 MHz, $CDCl_3$): δ 5.92 (d, *J* = 3.8 Hz, 1H, H-1); 4.60 (d, *J* = 3.8 Hz, 1H,H-2,), 4.20 (m, 1H, H-4), 3.90 (d, *J* = 3.1 Hz, 1H, H-3), 3.40 (s, 3H, OCH_3), 2.90 (m, 1H, H-5), 2.60 (m, 2H, NHCH_2), 1.90 (bs, 1H, exchangeable NH), 1.49–1.25 [m, 34 H (CH_3)$_2$C and 14 × CH_2), 0.87 (t, *J* = 6.7 Hz, 3H, CH_3); ^{13}C NMR (50 MHz, $CDCl_3$); δ 111.8 [(CH_3)$_2$*C*], 105.2 (C-1), 83.0 (C-2), 82.0 (C-4), 80.5 (C-3), 58.0 (OCH_3), 50.5 (C-5), 48.3 (NCH_2), 32.3, 30.3, 30.1, 30.0, 29.9, 29.7, 27.6, 23.1 (CH_2's), 27.1 and 26.6 [C(*C*H_3)$_2$], 14.5 (CH_3); Anal. Calc. for $C_{25}H_{25}NO_4$: C, 70.25; H, 11.47; N, 3.27. Found: C, 70.10; H, 11.57; N, 3.17.

*6.2.23. 3-*O*-Benzyl-5-deoxy-5-dodecylamino-1,2-*O*-isopropylidene-α-D-xylofuranose (**38**)*

Reaction of compound **2** (1.0 g, 3.59 mmol), dodecylamine (0.78 g, 3.59 mmol) and $NaBH_4$ (0.14 g, 3.59 mmol) as described above gave compound **38** as colorless oil. Yield: 50%; $[\alpha]_D$ = –40° (*c* = 0.18, chloroform); MS (FAB) = *m/z* 448 (M + H)$^+$; IR (Neat): ν_{max} cm^{-1} 3224, 1457; ^{1}H NMR (200 MHz, $CDCl_3$): δ 7.35–7.26 (m, 5H, Ar-H), 5.93 (d, *J* = 3.8 Hz, 1H, H-1), 4.67–4.51 (m, 3H, CH_2Ph and H-2), 4.30 (m, 1H, H-4), 3.89 (d, *J* = 3.1, 1H, H-3), 2.90 (m, 1H, H-5), 2.60 (m, 2H, NHCH_2), 1.56 (bs, 1H, exchangeable NH), 1.48–1.25 (m, 26 H (CH_3)$_2$C and 10 × CH_2), 0.81 (t, *J* = 6.7 Hz, 3H, CH_3); ^{13}C NMR (50 MHz, $CDCl_3$): δ 137.9 (Ar-C), 128.9, 128.4, 128.1 (Ar-CH), 112.0 [(CH_3)$_2$*C*], 105.3 (C-1), 82.7 (C-2), 82.4 (C-4), 79.8 (C-3), 72.2 (O*C*H_2Ph), 50.5 (C-5), 48.4 (NCH_2), 32.3, 30.2, 30.0, 29.7, 27.6 and 23.1 (CH_2's), 27.1 and 26.7 [C(*C*H_3)$_2$], 14.5 (CH_3); Anal. Calc. for $C_{27}H_{45}O_4N$: C, 72.48; H, 10.06; N, 3.13. Found: C, 71.78; H, 10.10; N, 3.17.

*6.2.24. 3-*O*-Benzyl–5-deoxy-1,2-*O*-isopropylidene-5-oleylamino-α-D-xylofuranose (**39**)*

Reaction of compound **2** (1.0 g, 3.59 mmol), oleylamine (1.0 g, 3.59 mmol) and $NaBH_4$ (0.14 g, 3.59 mmol) as described above gave compound **39** as colorless oil. Yield: 55%; $[\alpha]_D$ = –40° (*c* = 0.18, chloroform); MS (FAB) = *m/z* 530 (M + H)$^+$; IR (Neat): ν_{max} cm^{-1} 3224, 1457; ^{1}H NMR (200 MHz,$CDCl_3$): δ 7.34–7.26 (m, 5H, Ar-H), 5.94 (d, *J* = 3.8 Hz, 1H, H-1), 5.5 (m, 2H, HC=CH), 4.67–4.51 (m, 4H, CH_2Ph and H-2), 4.20 (m, 1H, H-4), 3.91 (d, *J* = 3.2, 1H, H-3), 2.70 (m, 1H, H-5); 2.60 (m, 2H, NCH_2), 2.0 (m, 4H, CH_2CH=CHCH_2), 1.48 and 1.33 [s, each 3H (CH_3)$_2$C], 1.31–1.25 (m, 24H, 12 × CH_2's), 0.81 (t, *J* = 6.5 Hz, 3H, CH_3); ^{13}C NMR (50 MHz, $CDCl_3$): δ 137.7 (CH=CH), 130.3, 129.0, 128.9 (Ar-C); 112.1 [(CH_3)$_2$*C*], 105.4 (C-1), 83.1 (C-2), 82.9 (C-4), 82.6 (C-3), 72.3 (OCH_2Ph), 61.3, 50.1, 47.9, 32.3, 30.1, 27.6 (CH_2's), 27.2 and 26.7 [C(*C*H_3)$_2$], 14.4 (CH_2CH_3); Anal. Calc. for $C_{33}H_{55}O_4N$: C, 74.48; H, 10.39; N, 2.64. Found: C, 74.50; H, 10.30; N, 2.72.

*6.2.25. 6-Deoxy-6-dodecylamino-1,2:3,4-di-*O*-isopropylidene-α-D-galactopyranose (**40**)*

Reaction of compound **3** (2.0 g, 7.75 mmol), dodecylamine (1.43 g, 8.0 mmol) and $NaBH_4$ (0.3 g, 7.75 mmol) as described above gave compound **40** as colorless oil.Yield: 58.3%; $[\alpha]_D$ = –26.4° (*c* = 0.11, chloroform), MS (FAB) = *m/z* 428 (M + H)$^+$; IR (Neat): ν_{max} cm^{-1} 3422, 1451; ^{1}H NMR (200 MHz, $CDCl_3$): δ 5.56 (d, *J* = 5.2 Hz, 1H, H-1), 4.58–3.85 (m, 4H, H-3, H-2, H-4 and H-5), 2.90-2.10 (m, 4H, H-6 and NCH_2), 2.01 (bs, 1H, exchangeable NH), 1.54 and 1.45 [s, each 3H (CH_3)$_2$C], 1.35–1.16 (m, 26H (CH_3)$_2$C and 10 × CH_2's), 0.81 (t, *J* = 7.0 Hz, 3H, CH_3); ^{13}C NMR (50 MHz, $CDCl_3$): δ 109.5 and 108.9 [2 × (CH_3)$_2$*C*], 96.8 (C-1), 72.4 (C-3), 71.3 (C-2), 71.0 (C-4), 66.9 (C-5), 49.9 (C-6), 32.2, 30.0, 29.7, 27.7 and 23.0 (CH_2's), 26.3, 25.3 and 24.7 [2 × C(*C*H_3)$_2$], 14.4 (CH_2CH_3); Anal. Calc. for $C_{24}H_{45}O_5N$: C, 67.44; H, 10.32; N, 3.32. Found: C, 67.63; H, 10.02; N, 3.30.

*6.2.26. 6-Deoxy-1,2:3,4-di-*O*-isopropylidene-6-hexadecylamino-α-D-galactopyranose (**41**)*

Reaction of compound **3** (1.0 g, 3.87 mmol), hexadecylamine (0.94 g, 3.87 mmol) and $NaBH_4$ (0.14 g, 3.87 mmol) as described above gave compound **41** as colorless oil. Yield: 81%; $[\alpha]_D$ = –36.4° (*c* = 0.18, chloroform); MS (FAB) = *m/z* 484 (M + H)$^+$; IR (Neat): ν_{max} cm^{-1} 3403, 1461; ^{1}H NMR (200 MHz, $CDCl_3$): δ 5.55 (d, *J* = 5.1 Hz, 1H, H-1), 4.60 (dd, *J* = 7.9 and 2.0 Hz, 1H, H-3), 4.30 (dd, *J* = 5.1 and 2.0 Hz, 1H, H-2), 4.17 (dd, *J* = 7.9 and 1.5 Hz, H-4), 3.80 (m, 1H, H-5), 2.80–2.41 (m, 4H, H-6 and NCH_2), 2.2 (bs, 1H, exchangeable NH), 1.54 and 1.45 [s, each 3H (CH_3)$_2$C], 1.32–1.25 [m, 34H (CH_3)$_2$C and 14 × CH_2], 0.81 (t, *J* = 6.6 Hz, 3H, CH_3); ^{13}C NMR (50 MHz, $CDCl_3$): δ 109.6 and 108.9 [2 × (CH_3)$_2$*C*], 96.8 (C-1), 72.4 (C-3), 71.3(C-2), 71.0 (C-4), 66.9 (C-5). 49.9 (C-6), 32.3, 30.0, 29.9, 29.7, 27.7 and 23.0 (CH_2's), 26.3, 25.3 and 24.7 [2 × C(*C*H_3)$_2$], 14.4 (CH_2CH_3);

Anal. Calc. for $C_{28}H_{53}O_5N$: C, 69.56; H, 10.97; N, 2.89. Found: C, 69.45; H, 10.77; N, 2.86.

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