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Glucosaminides Proceeding from Levoglycosenone

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Abstract—The possibility was studied of using levoglycosenone in the synthesis of *N*-(glucosen-2-yl) aminides. New glucosaminides were obtained containing the residues of *L*-a, isopinocampheol, borneol, , and (–)-(1*S*,2*R*,3*S*,7*S*,8*R*)-4,4-dimethyl-3-nitro-9,11-dioxatricyclo[6.2.1.$0^{2,6}$]-undec-5-en-7-ol.

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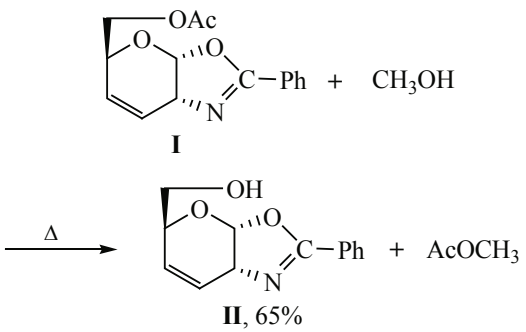
The application of unsaturated carbohydrates in the glucosylation reactions is attractive for it provides a possibility to avoid the superfluous operations with protective groups. Besides the lack of hydroxy groups permits a hope of increasing the efficiency of the process due to reduced sterical hindrances and to the increased solubility of the glycoside in organic solvents which is important for its possible further modification.

In extension of study [1] we investigated the glucosylation opportunities of oxazoline **I** obtained from the levoglycosenone; compounds of this class were known to easily convert at treating with alcohols into 1,2-*trans*-glucosaminides [2].

The attempt to convert oxazoline **I** into methylacetal by boiling in methanol failed and resulted in the methanolysis of the acetoxy group (Scheme 1).

Scheme 1.



Yet in the presence of the catalytic quantity of 50% water solution of HBr in dichlororethane the isopinocampheol reacted with oxazoline **I** (Scheme 2).

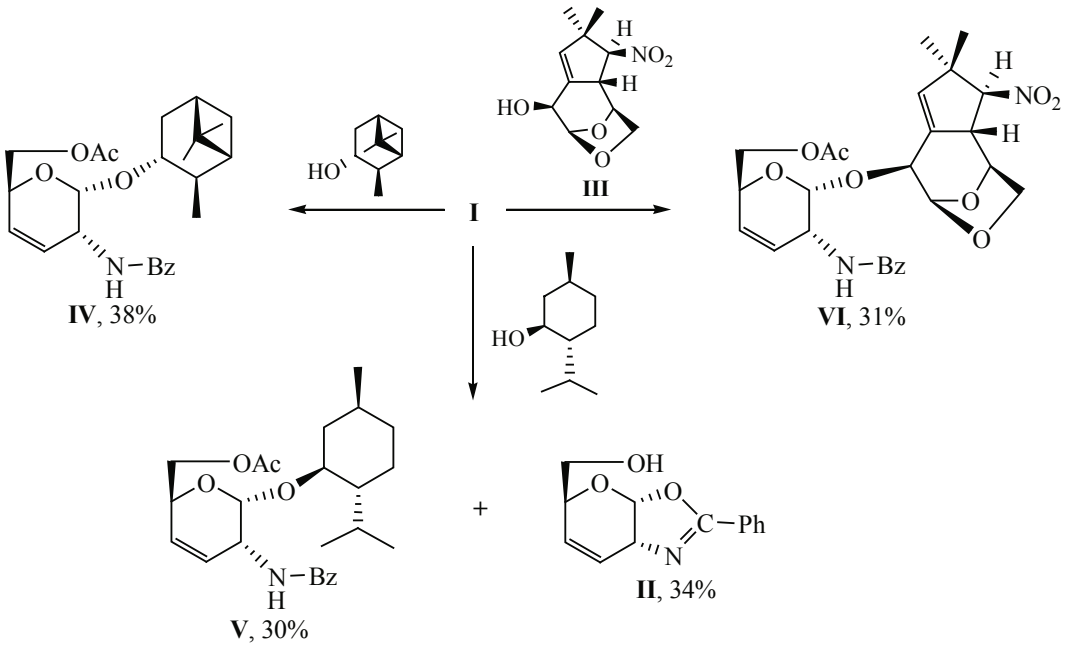
In the ^{1}H NMR spectrum of compound **IV** the proton H^1 gave rise to a singlet at 5.45 ppm in contrast to the analogous proton in the initial oxazoline **I** that appeared as a doubpet at 6.36 ppm, *J* 7.1 Hz. These findings indicate the *cis*-position of the substituents at the C^1 and C^2 atoms. Apparently at the action of HBr 1,2-*trans*-glycosyl bromide was formed accompanied with the first inversion of the configuration at C^1. Then the bromine atom is replaced by the alkoxide anion with the second inversion in the acetal center resulting in 1,2-*cis*-glucosaminide **IV**.

In a similar way the reactions of *L*-menthol and compound **III** [3] with oxazoline **I** afforded 1,2-*cis*-glucosaminides **V** and **VI**.

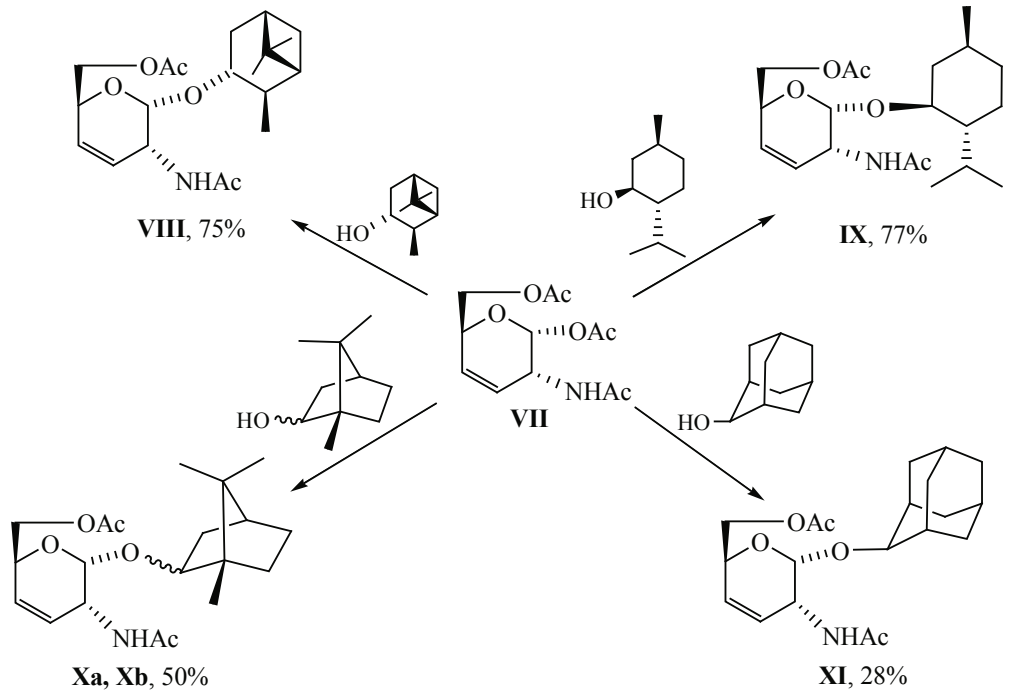
The low yields of glycosides prompted us to estimate the efficiency of the alternative glucosylation procedure with the use of triacetate **VII** obtained by the treatment of 1,6-anhydro-5-amino-2,3,4-trideoxy-β-D-glucopyranose [4] with acetic anhydride in the presence of sulfuric acid [1].

The reactions of *L*-menthol and isopinocampheol with triacetate **VII** in dichloromethane in the presence of a stoichiometric amount of $SnCl_4$ furnished in good yields 1,2-*cis*-glucosaminides **VIII** and **IX**. The yield in the reaction with borneol was somewhat lower, and it

Scheme 2.



Scheme 3.



was still worse with adamantan-2-ol. In the latter case it is likely to occur due to the low solubility of the aglycone in the dichloromethane (Scheme 3).

The assignement of the signals in the ^{1}H and ^{13}C NMR spectra of glycosides **VIII** –**XI** was carried out using the spectra HHCOSY and HETCOR.

The comparative analysis of the parameters of ^{1}H and ^{13}C NMR spectra of glycoside **VIII** and triacetate **VII** showed that at the formation of compound **VIII** the signal of proton H^1 suffered an upfield shift (δ 4.77 ppm), and the signal of atom C^1 shifted downfield (δ 98.15 ppm). In the ^{1}H NMR spectrum of compound **VIII** the proton H^1

gives rise to a doublet at 4.77 ppm, $^3J_{1-2}$ 4.7 Hz indicating the 1,2-*cis*-position of the substituents. Consequently, the asymmetrical center C^1 possesses the *R*-configuration. The established spectral regularities are also observed in the spectra of glucosaminides **IX–XI**.

Thus the second way proved to be more efficient for the preparation of *N*-(glucosen-2-yl)amides containing a double bond in the carbohydrate fragment. Amidoglycosides **IV, V** exhibited a positive cytotoxic effect in the test on the cells of a number of human tumors.

EXPERIMENTAL

^{1}H and ^{13}C NMR spectra were registered on a spectrometer Bruker AM-300 at operating frequencies 300.13 and 75.47 MHz respectively, internal reference TMS, solvent $CDCl_3$. The optical rotation angles were measured on a polarimeter Perkin Elmer-141. Melting pints were measured on a Koeffler heating block S 30A/G (DDR). The analytical TLC was performed on plates Sorbfil PTSKh-AF-A, development with anise aldehyde. The products of syntheses were isolated by column chromatography on silica gel L-40/100, 30–100 g of sorbent per 1 g of compound.

(–)-(3a*R*,5S,7aR)-(2-Phenyl-6-O-acetyl-5,7a-dihydro-3a*H*-pyrano[3,2-*d*][1,3]oxazol-5-yl)-methyl acetate (**I**) and (–)-(2*S*,5*R*,6*R*)-[5-(acetylamino)-6-(acetoxy)-5,6-dihydro-2*H*-pyran)-2-yl]methyl avetate (**VII**) were obtained by procedure [1], (–)-(1*S*,2*R*,3*S*,7*S*,8*R*)-4,4-dimethyl-3-nitro-9,11-dioxatricyclo[6.2.1.0^{2,6}]-undecen-7-ol (**III**), by method [3].

(–)-(3a*R*,5*S*,7a*R*)-(2-Phenyl-5,7a-dihydro-3a*H*-pyrano[3,2-*d*][1,3]oxazol-5-yl)methanol (II). A solution of 0.035 g (0.12 mmol) of oxazoline **I** in 4 ml of anhydeous MeOH was boiled for 2 h. Than the solvent was evaporated, the residue was subjected to chromatography. Yield 0.02 g (65%). Oily substance, R_f 0.3 (ethyl acetate–hexane, 2 : 1), $[\alpha]_D^{20}$ –106.2° (*c* 1.0, CH_2Cl_2). ^{1}H NMR spectrum, δ, ppm (the numeration of atoms of carbohydrate moiety is conserved): 3.70 d.d (1H, H^{6A}, *J* 11.9, *J* 5.5 Hz), 3.80 d.d (1H, H^{6B}, *J* 11.9, *J* 2.8 Hz), 4.35 m (1H, H^2), 4.62 d.d (1H, H^5, *J* 5.5, *J* 2.8 Hz), 6.00 m (1H, H^3), 6.20 m (1H, H^4), 6.36 d (1H, H^1, *J* 7.1 Hz), 7.48 m (3H, Ph), 8.00 m (2H, Ph). ^{13}C NMR spectrum, δ, ppm: 59.6 (C^2), 64.7 (C^6), 68.3 (C^5), 101.8 (C^1), 125.9 (C^3), 128.2, 128.4, 128.6, 129.6 (Ph), 131.7 (C^4), 164.0 (N=<u>C</u>OPh).

Glucosylation of alcohols with oxazoline I. To a solution of 1 mmol of oxazoline **I** and 1 mmol of an appropriate alcohol in 5 ml of 1,2-dichloroethane was added at stiring a catalytic quantity of 50% HBr solution. The mixture was stirred at 15°C till complete conversion of initial oxazoline (TLC monitoring). The solvent was evaporated, the residue was subjected to chromatography.

(–)-(1'*S*,2'*S*,3'*S*,5'*S*)-(2,6,6-Trimethylbicyclo-[3.1.1]hept-3-yl) 6-O-acetyl-2-benzamido-2,3,4-trideoxy-α-D-*erythro*-hex-3-enopyranoside (IV). Yield 0.013 g (38%). Colorless crystals, mp 50°C (from ethyl acetate), R_f 0.47 (hexane– ethyl acetate, 1 : 1), $[\alpha]_D^{20}$ –71.8° (*c* 1.0, $CHCl_3$). ^{1}H NMR spectrum, δ, ppm (CD_3COCD_3): 0.75 s (3H, CH_3), 1.00 m (3H, CH_3), 1.35 s (3H, CH_3), 2.00 m (5H, CH_3, $H^{7'}$), 2.30 m (2H, $H^{4'}$), 2.91 m (1H, $H^{2'}$), 2.75 m (2H, $H^{5'}$, $H^{1'}$), 3.60 d.d (1H, $H^{6'A}$, *J* 6.3, *J* 4.1 Hz), 3.80 d (1H, $H^{6'B}$, *J* 6.3 Hz), 4.02 m (1H, $H^{3'}$), 4.35 m (1H, H^2), 4.75 t (1H, H^5, *J* 4.1 Hz), 5.45 s (1H, H^1), 5.54 m (1H, H^3), 6.30 m (1H, H^4), 7.45 m (3H, Ph), 8.00 m (2H, Ph). ^{13}C NMR spectrum, δ, ppm: 16.23 (CH_3), 16.23 (CH_3), 20.71 (CH_3), 26.73 (CH_3), 22.59 ($C^{7'}$), 34.06 ($C^{4'}$), 37.61 ($C^{6'}$), 44.3 ($C^{2'}$), 44.60 ($C^{1'}$), 46.30 ($C^{5'}$), 47.2 (C^2), 53.29 ($C^{3'}$), 69.9 (C^6), 70.1 (C^5), 101.4 (C^1), 123.6 (Ph), 126.86 (C^3), 127.0 (C^4), 127.18 (Ph), 127.81 (Ph), 131.02 (Ph), 131.25 (Ph), 133.99 (Ph), 165.68 (C=O), 172.73 (C=O).

(–)-(1'*R*,5'*R*,2'*S*)-(2-Isopropyl-5-methylcyclohexyl) 6-O-acetyl-2-benzamido--2,3,4-trideoxy-α-D-*erythro*-hex-3-enopyranoside (V). We obtained 0.014 g (30%) of glycoside **V** and 0.016 g (60%) of alcohol **II**. Colorless crystals, mp 119°C (from ethyl acetate), R_f 0.5 (hexane–ethyl acetate, 1 : 1), $[\alpha]_D^{20}$ –58.8° (*c* 1.0, $CHCl_3$). ^{1}H NMR spectrum, δ, ppm: 0.45 d [3H, $CH(C\underline{H}_3)_2$, *J* 7.0 Hz], 0.65 d [3H, $CH(C\underline{H}_3)_2$, *J* 7.0 Hz], 0.82 m (1H, $H^{5'A}$), 0.85 m (1H, $H^{4'A}$), 0.89 d (3H, CH_3), 1.00 m (1H, $H^{2'A}$), 1.22 m (1H, $H^{6'}$), 1.30 m (1H, $H^{3'}$), 1.52 m (1H, $H^{5'B}$), 1.55 m (1H, $H^{4'B}$), 1.95 s (3H, $COCH_3$), 2.00 m (1H, $H^{2'B}$), 2.08 a (3H, $NHCH_3$), 2.23 m [1H, $C\underline{H}(CH_3)_2$], 3.27 d.t (1H, $H^{1'}$, *J* 4.3, *J* 10.6 Hz), 4.00 d.d (2H, H^{6A}, H^{6B}, *J* 6.2, *J* 3.6 Hz), 4.38 d (1H, H^2, *J* 3.6 Hz), 4.90 m (1H, H^5), 5.00 d (1H, H^1, *J* 4.9 Hz), 5.53 m (1H, $N\underline{H}CH_3$), 5.62 m (2H, H^3, H^4), 6.25 d (1H, *J* 6.5 Hz), 7.30 m (3H, Ph), 7.70 m (2H, Ph). ^{13}C NMR spectrum, δ, ppm: 15.68 (CH_3), 20.73 ($CO\underline{C}H_3$), 21.44 [$CH(\underline{C}H_3)_2$], 22.73 [$CH(\underline{C}H_3)_2$], 23.15 ($C^{5'}$), 26.02 ($C^{3'}$), 32.01 [$\underline{C}H(CH_3)_2$], 34.64 ($C^{4'}$), 43.43 ($C^{2'}$), 46.59 ($C^{6'}$), 49.10 (C^2), 65.79 (C^6), 67.01 (C^5), 81.54 ($C^{1'}$), 97.95 (C^1), 126.65 (Ph), 127.37 (C^4), 127.67 (C^3), 128.32 (Ph), 128.46 (Ph), 131.35 (Ph), 165.42 (C=O), 169.22 (C=O).

(–)-(1'*S*,4'*R*,8'*S*,5'*S*,8'*S*,8a'*R*)-(7,7-Dimethyl-8-nitro-1,4,5,7,8,8a-hexahydro-2*H*-1,4-epoxycyclo-

penta[*d*]oxepin-5-yl) 6-*O*-acetyl-2-benz-amido-2,3,4-trideoxyhex-3-enopyranoside (VI). Yield 0.013 g (38%), R_f 0.47 (hexane–ethyl acetate, 1 : 1), $[\alpha]_D^{20}$ –71.8° (*c* 1.0, $CHCl_3$). ^{1}H NMR spectrum, δ, ppm: 1.01 s (3H, CH_3), 1.40 s (3H, CH_3), 1.42 s (3H, CH_3), 3.46 d.t (1H, H$^{8'A}$, *J* 9.4, *J* 3.9 Hz), 3.50 m (1H, H^{6A}), 3.51 m (1H, H$^{2'A}$), 3.85 d.d (1H, H$^{2'B}$, *J* 6.5, *J* 3.4 Hz), 3.94 d (1H, H^{6B}, *J* 7.6 Hz), 4.16 m (1H, H$^{5'}$), 4.21 m (1H, H^{2}), 4.45 m (1H, H$^{1'}$), 4.70 m (1H, H^{5}), 4.72 d (1H, H$^{8'}$), 5.31 d (1H, H^{1}, *J* 5.0 Hz), 5.53 s (1H, H$^{4'}$), 5.72 d (1H, H$^{6'}$, *J* 2.4 Hz), 5.75 m (2H, H^{3}, H^{4}), 7.50 m (3H, Ph), 7.78 m (2H, Ph). ^{13}C NMR spectrum, δ, ppm: 20.78 (CH_3), 26.77 (CH_3), 26.91 (CH_3), 47.31 (C$^{7'}$), 47.63 (C^{8A}), 66.16 (C^{2}), 70.02 (C^{6}), 70.33 (C$^{2'}$), 70.57 (C^{5}), 71.33 (C$^{5'}$), 74.22 (C$^{1'}$), 97.41 (C$^{8'}$), 101.73 (C^{1}), 104.05 (C$^{4'}$), 124.26 (C^{3}), 127.03, 128.53, 131.28, 131.78 (Ph), 131.71 (C^{4}), 134.89 (C$^{5'A}$), 135.16 (C$^{6'}$), 166.58 (C=O), 167.05 (C=O).

Glucosylation of alcohols with triaxetate VII. To a solution of 1 mmol of triacetate **VII** and 1 mmol of an appropriate alcohol in 7 ml of CH_2Cl_2 was added at stirring 1 mmol of $SnCl_4$. The mixture was stirred at 15°C till complete conversion of initial compounds (TLC monitoring). The solvent was evaporated, the residue was subjected to chromatography.

(–)-(1'*S*,2'*S*,3'*S*,5'*S*)(2,6,6-Trimethylbicyclo-[3.1.1]hept-3-yl) 6-O-acetyl-2-acetamido-2,3,4-trideoxy-α-D-*erythro*-hex-3-enopyranoside (VIII). Yield 0.35 g (47%), R_f 0.5 (hexane– ethyl acetate, 1:1), $[\alpha]_D^{20}$ +15.7° (*c* 1.0, $CHCl_3$). ^{1}H NMR spectrum, δ, ppm: 0.90 s (3H, CH_3), 1.10 d (3H, CH_3, *J* 7.4 Hz), 1.11 m (1H, H$^{7'A}$), 1.21 s (3H, CH_3), 1.78 m (1H, H$^{1'}$), 1.81 m (1H, H$^{4'A}$), 1.91 m (1H, H$^{5'}$), 2.00 s (3H, $COCH_3$), 2.08 s (3H, $COCH_3$), 2.09 m (1H, H$^{2'}$), 2.33 m (1H, H$^{7'B}$), 2.45 m (1H, H$^{4'B}$), 4.02 m (1H, H$^{3'}$), 4.18 m (2H, H^{6A,6B}), 4.29 m (1H, H^{2}), 4.46 m (1H, H^{5}), 4.77 d (1H, H^{1}, *J* 4.7 Hz), 5.70 d (1H, NH, *J* 6.5 Hz), 5.84 m (2H, H^{3,4}). ^{13}C NMR spectrum, δ, ppm: 20.31 (CH_3), 20.88 ($CO\underline{C}H_3$), 23.33 ($CO\underline{C}H_3$), 23.78 (CH_3), 27.42 (CH_3), 33.16 (C$^{7'}$), 35.65 (C$^{4'}$), 38.29 (C$^{6'}$), 41.28 (C$^{1'}$), 41.4 (C$^{5'}$), 44.3 (C$^{2'}$), 47.7 (C^{2}), 66.4 (C^{6}), 71.3 (C^{5}), 77.94 (C$^{3'}$), 98.15 (C^{1}), 126.30 (C^{3}), 127.35 (C^{4}), 169.81 (C=O), 170.82 (C=O).

(–)-(1'*R*,3'*R*,6'*S*)(6-Isopropyl-1-methylcyclohex-3-yl) 6-O-acetyl-2-acetamido-2,3,4-trideoxy-α-D-*erythro*-hex-3-enopyranoside (IX). Yield 0.03 g (55%), R_f 0.3 (hexane–ethyl acetate, 1:1). $[\alpha]_D^{20}$ –155.7° (*c* 1.0, $CHCl_3$). ^{1}H NMR spectrum, δ, ppm: 0.78 d (3H, CH_3, *J* 6.7 Hz), 0.81 m (1H, H$^{4'A}$), 0.89 d [3H, $CH(C\underline{H}_3)_2$, *J* 6.7 Hz], 0.90 d [3H, $CH(C\underline{H}_3)_2$, *J* 6.7 Hz], 0.92 m (1H, H$^{2'A}$), 0.96 m (1H, H$^{5'A}$), 1.21 m (1H, H$^{6'}$), 1.37 m (1H, H$^{3'}$), 1.61 m (1H, H$^{5'B}$), 1.68 m (1H, H$^{4'B}$), 1.95 m (1H, H$^{2'B}$), 2.00 s (3H, $NHCOC\underline{H}_3$), 2.08 s (3H, $OCOCH_3$), 2.22 m [1H, $C\underline{H}(CH_3)_2$], 3.45 m (1H, H$^{1'}$), 4.13 m (2H, H^{6A,6B}), 4.20 m (1H, H^{2}), 4.45 m (1H, H^{5}), 4.73 d (1H, H^{1}, *J* 6.6 Hz), 5.53 m (1H, NH), 5.72 d.d (1H, H^{3}, *J* 10.1 Hz), 5.83 d.d (1H, H^{4}, *J* 10.1 Hz). ^{13}C NMR spectrum, δ, ppm: 15.59 (CH_3), 20.78 ($CO\underline{C}H_3$), 21.02 [$CH(\underline{C}H_3)_2$], 22.31 [$CH(\underline{C}H_3)_2$], 23.07 (C$^{5'}$), 23.43 ($NH\underline{C}OCH_3$), 25.13 [$\underline{C}H(CH_3)_2$], 31.56 (C$^{3'}$), 34.39 (C$^{4'}$), 40.89 (C$^{2'}$), 47.78 (C$^{6'}$), 49.61 (C^{2}), 66.05 (C^{6}), 72.38 (C^{5}), 77.41 (C$^{1'}$), 97.46 (C^{1}), 126.65 (C^{3}), 128.18 (C^{4}), 169.97 (C=O), 170.81 (C=O).

(1'*S*,2'*R*,*S*)(1,7,7-Trimethylbicyclo[2.2.1]hept-2-yl) (6-O-acetyl-2-acetamido-2,3,4-trideoxy-α-D-*erythro*-hex-3-enopyranoside (Xa, b). Yield 0.03 g (50%), R_f 0.27 (hexane–ethyl acetate, 1 : 1). ^{1}H NMR spectrum, δ, ppm: 0.80 s (3H, CH_3), 0.88 s (3H, CH_3), 0.92 s (3H, CH_3), 1.00 m (1H, H$^{5'B}$), 1.21 m (1H, H$^{6'B}$), 1.50 m (1H, H$^{5'A}$), 1.62 m (2H, H$^{3'AB}$), 1.87 m (1H, H$^{4'}$), 2.00 s (3H, CH_3), 2.10 s (3H, CH_3), 2.13 m (1H, H$^{6'A}$), 3.59 d.d (1H, H$^{2'}$, *J* 3.6, *J* 8.1 Hz) [4.05 d.d (1H, H$^{2'}$, *J* 1.6, *J* 7.9 Hz)],[1] 4.17 m (2H, H^{6A,B}), 4.40 m (2H, H^{5,2}), 4.70 d (1H, H^{1}, *J* 3.8 Hz) [4.72 d (1H, H^{1}, *J* 3.8 Hz)], 5.49 m (1H, NH), 5.83 m (2H, H^{3}, H^{4}). ^{13}C NMR spectrum, δ, ppm, (**Xa**): 12.04 (CH_3), 20.01 (CH_3), 20.19 (CH_3), 20.88 (CH_3), 23.34 (CH_3), 27.20 (C$^{6'}$), 34.18 (C$^{5'}$), 39.64 (C$^{4'}$), 45.03 (C$^{3'}$), 46.89 (C$^{1'}$), 47.24 (C^{2}), 49.49 (C$^{7'}$), 66.30 (C$^{2'}$), 70.72 (C^{6}), 86.36 (C^{5}), 100.32 (C^{1}), 125.80 (C^{3}), 127.56 (C^{4}), 169.42 (C=O), 170.79 (C=O); (Xb): 13.66 (CH_3), 18.90 (CH_3), 19.83 (CH_3), 20.88 (CH_3), 23.34 (CH_3), 26.63 (C$^{6'}$), 28.38 (C$^{5'}$), 35.87 (C$^{4'}$), 44.93 (C$^{3'}$), 46.63 (C$^{1'}$), 47.88 (C^{2}), 49.03 (C$^{7'}$), 66.53 (C$^{2'}$), 70.72 (C^{6}), 82.19 (C^{5}), 98.36 (C^{1}), 125.69 (C^{3}), 127.66 (C^{4}), 169.54 (C=O), 170.79 (C=O).

(–)-Adamantan-2-yl 6-O-acetyl-2-acetamido-2,3,4-trideoxy-α-D-*erythro*-hex-3- enopyranoside (XI). Yield 0.01 g (28%), R_f 0.3 (hexane– ethyl acetate, 1 : 1), $[\alpha]_D^{20}$ –80° (*c* 1.0, $CHCl_3$). ^{1}H NMR spectrum, δ, ppm: 1.20–2.00 m (14H, Ad), 3.80 m (1H, H$^{2'}$, Ad), 4.08 d.d (1H, H^{6A}, *J* 11.5, *J* 5.4 Hz), 4.12 d (1H, H^{6B}), 4.28 d.d (1H, H^{2}, *J* 3.4, *J* 8.2 Hz), 4.35 t (1H, H^{5}, *J* 5.4, *J* 11.6 Hz), 4.79 d (1H, H^{1}, *J* 3.4 Hz), 5.42 d (1H, NH, *J* 7.8 Hz), 5.80 m (2H, H^{3,4}). ^{13}C NMR spectrum, δ, ppm: 20.85 ($COCH_3$), 23.3 ($COCH_3$), 27.27, 27.34, 30.62, 31.26, 31.53, 33.18,

[1] Signal of isomer **Xb** are given in brackets..

36.26, 36.74, 37.49 (Ad), 47.41 (C^2), 66.42 (C^6), 70.71 (C^5), 79.49 ($C^{2'}$, Ad), 96.59 (C^1), 125.68 (C^4), 127.66 (C^3), 169.60 (C=O), 170.80 (C=O).

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