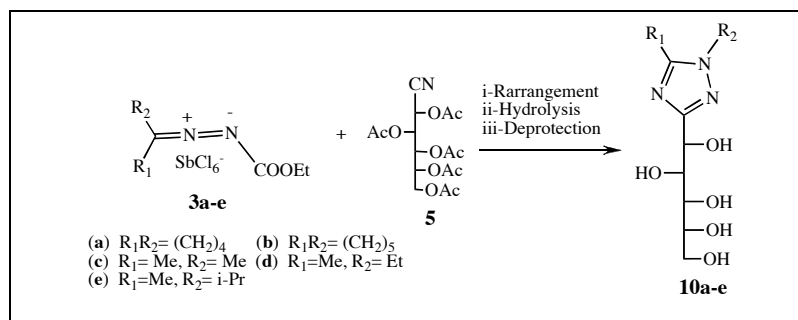


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Reported are preparations of acyclic derivatives of 1,2,4-triazole-5-glycosidies **9** by cycloadditions of 1-aza-2-azonia-allene salts **3** to the nitrile group of D-glucononitrile-2,3,4,5,6-pentaacetate **5** affording triazolium salts **8**, which with aqueous sodium hydrogencarbonate are hydrolyzed to **9**. Deacetylation of compounds **9** produced the C-glycosides **10**.

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INTRODUCTION

The biological importance of C-nucleosides [1-4], N-nucleosides [5-9] and acyclonucleosides [10-12] attracted our attention to syntheses of acyclic C-nucleosides of potential therapeutical effectiveness. Some 1,2,4-triazoles had been tested for potential biological activities as antiviral herbicides, fungicides and insecticides [13,14].

Al-Masoudi and we have reported preparations of C- and N-glycosides by cycloadditions of 1-aza-2-azoniaallene salts **3** to glycosyl nitriles, to a glycosyl alkyne, and to a glucopyranosyl isothiocyanate [16-20].

Here, I report extensions of my experiments of reactions of cumulenes **3** with D-glucononitrile-2,3,4,5,6-pentaacetate **5** leading, after treatment with aqueous sodium hydrogencarbonate, to compounds **9a-e**, and after deacetylation to glycosides **10a-e** (Scheme 1). Recently, similar compounds **A** and **B** (Figure 1) have been prepared by El Ashry *et al* [21,22]. Potent glucosidase inhibitors, which are analogues of nojirimycin (5-amino-5-deoxy-D-glucopyranose) **C** (Figure 1), were investigated by Vasella *et al*. [23].

RESULTS AND DISCUSSION

1-Aza-2-azoniaallene cations **3** are efficient 4-electron components for cycloadditions to many types of multiple bonds [15,16,24-31]. Cycloadditions of 1-aza-2-azoniaallenes **3** suffer from the disadvantage that one ends up with salts. For applications electronically neutral heterocycles would be more desirable. In order to obtain electronically neutral acyclic C-glycosides, heterocumu-

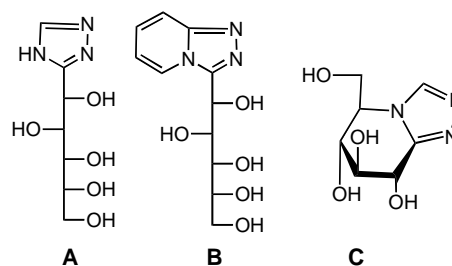
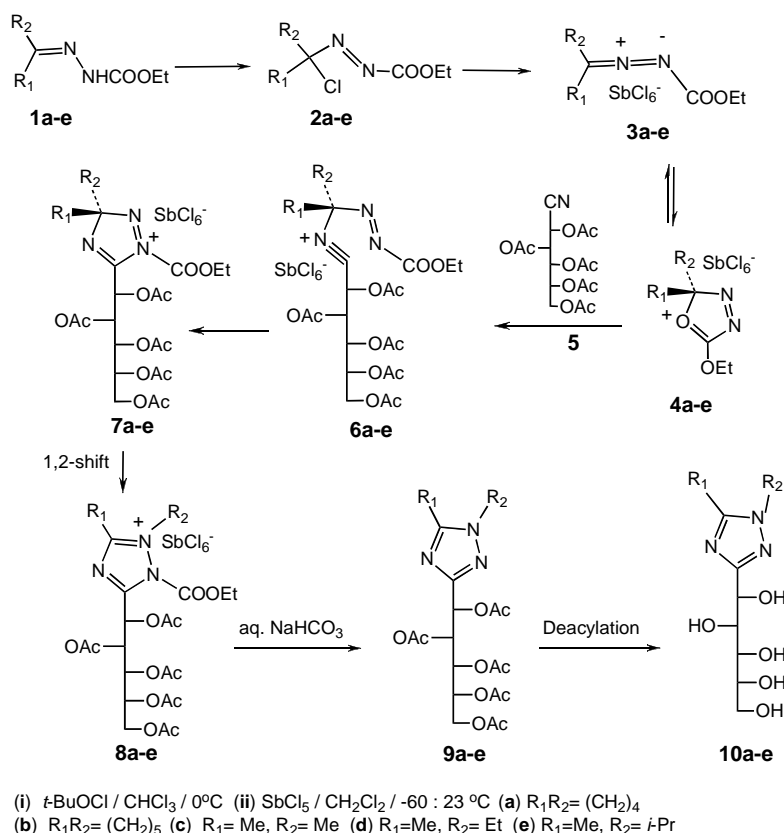


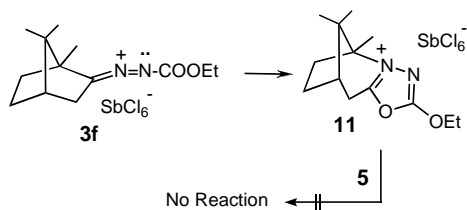
Figure 1

lenes **3a-e** were prepared, N-substituted with a leaving group COOEt, which can be removed from the cycloadducts **8a-e** resulting in the formation of the triazoles **9a-e**. The hydrazones **1**, prepared by condensation of ethylcarbrazate with ketones in boiling ethanol containing a few drops of acetic acid, were oxidized with *tert*-butylhypochlorite resulting in the formation of the (chloroalkyl)azo compounds **2a-e** [25,27,28,32]. These products reacted with antimony pentachloride at -60 °C in dry dichloromethane to afford the 1-aza-2-azoniaallene salts **3**, which cannot be isolated. However, at temperatures between -60 °C and room temperature the colours of solutions of mixtures of compounds **3** and penta-O-acetyl-D-glucononitrile **5** [33] changed indicating reactions. Work up led to the isolation of the triazolium salts **8a-e**, hydrolysis of which with aqueous sodium hydrogencarbonate afforded residues, which were purified by silica gel column chromatography to give the glycosides **9a-e**. Deacetylation of **9a-e** resulted



Scheme 1

in the formation of the C-glycosides **10a-e**. To rationalize the formation of compounds **10a-e**, I propose, in conformity with results of Wang, [15,31] that the reaction of a cation **3** with penta-*O*-acetyl-D-glucononitrile **5** results in the formation of a nitrilium salt **6**. These nitrilium salts, with an azo group in the α -position to the nitrilium nitrogen atom, cyclize spontaneously to furnish the triazolium salts **7**. At temperatures above -30°C the primarily formed product **7** rearranges to the final product **8** by [1,2] migration of an alkyl group from C-3 to N-1. If R^1 and R^2 are parts of a cyclus as in the cases of **1a,b**, the sequence **7a,b** \rightarrow **8a,b** constitutes a ring enlargement reminiscent of a Beckmann rearrangement [34]. In the case of the 1-aza-2-azoniaallene salt **3f** the cyclization to the oxadiazolium salt **11** (yield 90%) was faster than the reaction with penta-*O*-acetyl-D-glucononitrile (Scheme 2) [25]. Obviously, the salt **11** doesn't react with nitriles.



Scheme 2

The structures of the new products were established by their microanalytical and spectroscopic data (IR, ^1H NMR, ^{13}C NMR, and mass spectroscopy (*cf.* Experimental).

EXPERIMENTAL

All melting points are uncorrected and measured using Electro-thermal IA 9100 apparatus. IR spectra were recorded as potassium bromide pellets on a Nexus 670 spectrophotometer. ^1H NMR spectra were recorded on Bruker 300 spectrometers at 300 MHz and ^{13}C NMR spectra were recorded on Bruker 300 spectrometers at 75 MHz. ^1H NMR and ^{13}C NMR spectra were measured at Brock University (Canada). Chemical shifts were expressed as part per million; ppm (δ values) against TMS as internal reference. Microanalytical data were performed by Vario El Elemental apparatus. The chemicals were purchased from Aldrich.

General procedure for the preparation of hydrazones (1) [24]. A mixture of ketone (100 mmol) and ethyl hydrazine-carboxylate (100 mmol) in EtOH (100 ml)/AcOH (1 ml) was boiled under reflux for 4-8 h. Evaporation of the solvent and crystallization of the residue from EtOH afforded the pure hydrazone.

General procedure for the preparation of the α -chloro azo compounds [24] (2). A solution of *tert*-butylhypochlorite (13.02 g, 120 mmol) was added dropwise to a cold (-10°C) solution of the hydrazone (100 mmol) in CHCl_3 (100 ml). After stirring at 0°C for 3 h the solvent was removed under reduced pressure. The oily residue crystallized on cooling or after trituration with cold methanol or used without further purification.

General procedure for the preparation of acylated glycosidies (9). A solution of SbCl_5 (2.99 g, 10 mmol) in CH_2Cl_2 (20 ml) was added dropwise to a cold (-60°C) solution of **2** (10 mmol) and the penta-*O*-acetyl-D-glucononitrile **5** (10 mmol) in CH_2Cl_2 (30 ml). The reaction mixture was stirred at -60°C for 1 h, then between -30 and 0°C for 3 h, and finally between 0 and 25°C for 1 h. The solvent was evaporated and the residue was dissolved in CH_3CN (70 ml). At 0°C an aqueous solution of NaHCO_3 (3.36 g, 40 mmol) in H_2O (40 ml) was added and the mixture was stirred at room temperature for 1 h. The organic layer was separated and the aqueous layer was extracted with ethyl acetate (2 x 50 ml). The combined organic layers were dried over Na_2SO_4 and evaporated after filtration. Purification by column chromatography (SiO_2 ; eluent $\text{CHCl}_3/\text{MeOH}$ 9:1) afforded compounds **9**.

1,2,3,4,5-Penta-*O*-acetyl-1-(5,6,7,8-tetrahydro-[1,2,4]-triazolo[1,5-*a*]pyridin-2-yl)-D-arabinitol (9a). Yield 3.43 g (71%) as an oil. IR (KBr): ν (cm^{-1}) 1757 (C=O), 1603 (C=N). ^1H NMR (CDCl_3): δ = 1.88 (m, 4 CH_2), 1.99 - 2.11 (5 singlets, 5 Ac), 2.66 (m, CH_2), 3.98 (m, CH_2), 4.03 (dd, H-5", $J_{4',5''}$ = 5.76 Hz, $J_{5',5''}$ = 12.35 Hz), 4.59 (dd, H-5', $J_{4',5'}$ = 3.00 Hz), 5.29 (m, H-4'), 5.60 (dd, H-3', $J_{3',4'}$ = 8.15 Hz), 5.38 (dd, H-2', $J_{2',3'}$ = 3.06 Hz), 6.37 (d, H-1', $J_{1',2'}$ = 7.80 Hz). MS (FAB): m/z 484 [M+1]. Calcd. for $\text{C}_{21}\text{H}_{29}\text{N}_3\text{O}_{10}$ (483.48): C, 52.17; H, 6.05; N, 8.69. Found: C, 52.19; H, 6.07; N, 8.69.

1,2,3,4,5-Penta-*O*-acetyl-1-(6,7,8,9-tetrahydro-5H-[1,2,4]-triazolo[1,5-*a*]azepin-2-yl)-D-arabinitol (9b). Yield 3.03 g (61%) as an oily product; IR (KBr) ν (cm^{-1}): 1759 (C=O), 1601 (C=N); ^1H NMR (CDCl_3): δ = 1.45 (m, CH_2), 1.51 (m, CH_2), 1.60 (m, CH_2), 1.98 - 2.11 (5 singlets, 5 Ac), 2.64 (m, CH_2), 3.94 (m, CH_2), 4.01 (dd, H-5", $J_{4',5''}$ = 5.68 Hz, $J_{5',5''}$ = 12.40 Hz), 4.55 (dd, H-5', $J_{4',5'}$ = 2.99 Hz), 5.27 (m, H-4'), 5.58 (dd, H-3', $J_{3',4'}$ = 8.37 Hz), 5.31 (dd, H-2', $J_{2',3'}$ = 3.00 Hz), 6.32 (d, H-1', $J_{1',2'}$ = 7.79 Hz). MS (FAB): m/z 498 [M+1]. Calcd. for $\text{C}_{22}\text{H}_{31}\text{N}_3\text{O}_{10}$ (497.51): C, 53.11; H, 6.28; N, 8.45. Found: C, 53.10; H, 6.30; N, 8.46.

1,2,3,4,5-Penta-*O*-acetyl-1-(1,5-dimethyl-1H-1,2,4-triazol-3-yl)-D-arabinitol (9c). Yield 3.24 g (71%); m.p. $80-82^\circ\text{C}$; IR (KBr) ν (cm^{-1}): 1757 (C=O), 1601 (C=N); ^1H NMR (CDCl_3): δ = 1.99, 2.05, 2.06, 2.09, 2.10 (5 singlets, 5 Ac), 2.17 (s, CH_3), 3.51 (s, CH_3), 4.00 (dd, H-5", $J_{4',5''}$ = 5.78 Hz, $J_{5',5''}$ = 12.40 Hz), 4.57 (dd, H-5', $J_{4',5'}$ = 3.19 Hz), 5.25 (m, H-4'), 5.61 (dd, H-3', $J_{3',4'}$ = 8.07 Hz), 5.85 (dd, 1H, $J_{2',3'}$ = 3.00 Hz, H-2'), 6.30 (d, 1H, $J_{1',2'}$ = 7.81 Hz, H-1'); ^{13}C NMR (CDCl_3): δ = 20.53, 20.64, 20.82, (Ac), 11.18, 34.80 (CH_3), 67.79 (C-1'), 69.97 (C-2'), 68.70 (C-3'), 68.29 (C-4'), 61.98 (C-5'), 145.95 (C-5), 157.88 (C-3), 169.61, 169.78, 169.99, 170.80 (CO). MS (FAB): m/z 458 [M+1]. Calcd. for $\text{C}_{19}\text{H}_{27}\text{N}_3\text{O}_{10}$ (457.44): C, 49.89; H, 5.95; N, 9.19. Found: C, 49.91; H, 5.98; N, 9.20.

1,2,3,4,5-Penta-*O*-acetyl-1-(1-ethyl-5-methyl-1H-1,2,4-triazol-3-yl)-D-arabinitol (9d). Yield 3.11 g (66%); m.p. $79-81^\circ\text{C}$; IR (KBr) ν (cm^{-1}): 1756 (C=O), 1602 (C=N); ^1H NMR (CDCl_3): δ = 1.07 (t, 3H, ethyl- CH_3), 1.98, 2.01, 2.06, 2.08, 2.09 (5 singlets, 5 Ac), 2.19 (s, CH_3), 3.80 (q, J = 7.0 Hz, ethyl- CH_2), 4.03 (dd, H-5", $J_{4',5''}$ = 5.78 Hz, $J_{5',5''}$ = 12.55 Hz), 4.53 (dd, H-5', $J_{4',5'}$ = 3.12 Hz), 5.28 (m, H-4'), 5.60 (dd, H-3', $J_{3',4'}$ = 8.09 Hz), 5.75 (dd, H-2', $J_{2',3'}$ = 3.01 Hz), 6.29 (d, H-1', $J_{1',2'}$ = 7.79 Hz). MS (FAB): m/z 472 [M+1]. Calcd. for $\text{C}_{20}\text{H}_{29}\text{N}_3\text{O}_{10}$ (471.4): C, 50.95; H, 6.20; N, 8.91. Found: C, 50.95; H, 6.22; N, 8.92.

1,2,3,4,5-Penta-*O*-acetyl-1-(1-isopropyl-5-methyl-1H-1,2,4-triazol-3-yl)-D-arabinitol (9e). Yield 3.79 g (78%); m.p. $65-67^\circ\text{C}$; IR (KBr) ν (cm^{-1}): 1755 (C=O), 1601 (C=N); ^1H NMR

(CDCl_3): δ = 1.15 (d, J = 6.9 Hz, 6H, isopropyl- CH_3), 1.97, 2.03, 2.05, 2.09, 2.10 (5 singlets, 5 Ac), 2.24 (s, CH_3), 4.06 (dd, H-5", $J_{4',5''}$ = 5.69 Hz, $J_{5',5''}$ = 12.48 Hz), 4.49 (sept., isopropyl-CH), 4.59 (dd, H-5', $J_{4',5'}$ = 3.00 Hz), 5.30 (m, H-4'), 5.55 (dd, H-3', $J_{3',4'}$ = 8.09 Hz), 5.93 (dd, H-2', $J_{2',3'}$ = 3.01 Hz), 6.17 (d, H-1', $J_{1',2'}$ = 7.78 Hz). MS (FAB): m/z 486 [M+1]. Calcd. for $\text{C}_{21}\text{H}_{31}\text{N}_3\text{O}_{10}$ (485.50): C, 51.95; H, 6.44; N, 8.66. Found: C, 51.96; H, 6.22; N, 8.93.

Preparations of free C-glycosidic compounds (10). Dry gaseous ammonia was passed at 0°C for about 1 h into a solution of a nucleoside **9** (10 mmol) in dry MeOH (20 ml). Then, the mixture was stirred at 23°C until the reaction was judged to be complete by TLC. Evaporation at 40°C under reduced pressure afforded the free C-glycosides **10**.

1-(5,6,7,8-Tetrahydro-[1,2,4]triazolo[1,5-*a*]pyridin-2-yl)-D-arabinitol (10a). Yield 1.99 g (73%) of a foam. IR (KBr): ν (cm^{-1}) 3222 (OH), 1608 (C=N); ^1H NMR ($\text{DMSO}-d_6$): δ = 1.91 (m, 2 CH_2), 2.79 (m, 2 CH_2), 3.45 (dd, H-5", $J_{4',5''}$ = 6.15 Hz, $J_{5',5''}$ = 11.86 Hz), 3.60 (t, H-5'), 3.47 (t, H-4', $J_{4',5'}$ = 2.80 Hz), 3.77 (t, H-3', $J_{3',4'}$ = 8.00 Hz), 4.07 (dd, H-2', $J_{2',3'}$ = 1.58 Hz), 4.09 (m, CH_2), 4.20 (d, OH-3', $J_{3',\text{OH}}$ = 7.40 Hz), 4.23 (dd, H-1', $J_{1',2'}$ = 8.40 Hz), 4.31 (t, OH-5', $J_{5',\text{OH}}$ = 5.58 Hz), 4.32 (d, OH-2', $J_{2',\text{OH}}$ = 6.1 Hz), 4.34 (d, OH-4', $J_{4',\text{OH}}$ = 5.60), 5.19 (d, OH-1', $J_{1',\text{OH}}$ = 5.50 Hz); ^1H NMR ($\text{DMSO}-d_6 + \text{D}_2\text{O}$): δ = 1.93 (m, 2 CH_2), 2.81 (m, CH_2), 3.45 (dd, H-5", $J_{4',5''}$ = 6.15 Hz, $J_{5',5''}$ = 11.86 Hz), 3.60 (t, H-5'), 3.47 (t, H-4', $J_{4',5'}$ = 2.80 Hz), 3.77 (t, H-3', $J_{3',4'}$ = 8.00 Hz), 4.10 (m, CH_2), 4.01 (dd, H-2', $J_{2',3'}$ = 1.58 Hz), 4.23 (dd, H-1', $J_{1',2'}$ = 8.40 Hz); ^{13}C NMR ($\text{DMSO}-d_6$): δ = 19.9, 22.3, 23.0, 47.2 (CH_2), 66.1 (C-1'), 70.9 (C-2'), 70.5 (C-3'), 71.4 (C-4'), 63.7 (C-5'), 158.7 (C-5), 160.1 (C-3). MS (FAB): m/z 274 [M+1]. Calcd. for $\text{C}_{11}\text{H}_{19}\text{N}_3\text{O}_5$ (273.29): C, 48.35; H, 7.01; N, 15.38. Found: C, 48.38; H, 7.05; N, 15.39.

1-(6,7,8,9-Tetrahydro-5H-[1,2,4]triazolo[1,5-*a*]azepin-2-yl)-D-arabinitol (10b). Yield 1.75 g (61%); mp $79-81^\circ\text{C}$; IR (KBr) ν (cm^{-1}): 3230 (OH), 1603 (C=N); ^1H NMR (D_2O): δ = 1.58 (m, CH_2), 1.66 (m, CH_2), 1.80 (t, CH_2), 2.82 (t, CH_2), 3.49 (dd, H-5", $J_{4',5''}$ = 6.10 Hz, $J_{5',5''}$ = 11.80 Hz), 3.59 (t, H-5'), 3.46 (t, H-4', $J_{4',5'}$ = 2.80 Hz), 3.77 (dd, H-3', $J_{3',4'}$ = 8.01 Hz), 4.00 (dd, H-2', $J_{2',3'}$ = 1.60 Hz), 4.06 (t, CH_2), 4.36 (dd, H-1', $J_{1',2'}$ = 8.38 Hz). MS (FAB): m/z 288 [M+1]. Calcd. for $\text{C}_{12}\text{H}_{21}\text{N}_3\text{O}_5$ (287.32): C, 50.17; H, 7.37; N, 14.63. Found: C, 49.99; H, 7.30; N, 14.65.

1-(1,5-Dimethyl-1H-1,2,4-triazol-3-yl)-D-arabinitol (10c). Yield: 1.38 g (56%); mp $140-142^\circ\text{C}$; IR (KBr) ν (cm^{-1}): 3210 (OH), 1601 (C=N); ^1H NMR (D_2O): δ = 2.33 (s, CH_3), 3.60 (dd, H-5", $J_{4',5''}$ = 6.2 Hz, $J_{5',5''}$ = 12.2 Hz), 3.69 (t, H-4', $J_{4',5'}$ = 2.80 Hz), 3.65 (t, H-5'), 3.81 (dd, H-3', $J_{3',4'}$ = 8.3 Hz), 3.69 (s, N- CH_3), 4.03 (dd, H-2', $J_{2',3'}$ = 1.60 Hz), 4.06 (d, H-1', $J_{1',2'}$ = 8.40 Hz). MS (FAB): m/z 248 [M+1]. Calcd. for $\text{C}_9\text{H}_{17}\text{N}_3\text{O}_5$ (247.25): C, 43.72; H, 6.93; N, 16.99. Found: C, 43.52; H, 6.84; N, 16.86.

1-(1-Ethyl-5-methyl-1H-1,2,4-triazol-3-yl)-D-arabinitol (10d). Yield: 1.52 g (58%); m.p. $114-116^\circ\text{C}$; IR (KBr) ν (cm^{-1}): 3210 (OH), 1601 (C=N); ^1H NMR (D_2O): δ = 1.37 (t, ethyl- CH_3), 2.60 (s, CH_3), 3.60 (dd, H-5", $J_{4',5''}$ = 6.40 Hz, $J_{5',5''}$ = 12.00 Hz), 3.68 (t, H-4', $J_{4',5'}$ = 2.80 Hz), 3.71 (t, H-5'), 3.78 (dd, H-3', $J_{3',4'}$ = 8.20 Hz), 4.02 (dd, H-2', $J_{2',3'}$ = 1.60 Hz), 4.20 (q, N- CH_2 , J = 7.0), 4.81 (d, H-1', $J_{1',2'}$ = 8.20 Hz). MS (FAB): m/z 262 [M+1]. Calcd. for $\text{C}_{10}\text{H}_{19}\text{N}_3\text{O}_5$ (261.32): C, 45.97; H, 7.33; N, 16.08. Found: C, 45.72; H, 7.19; N, 16.19.

1-(1-Isopropyl-5-methyl-1H-1,2,4-triazol-3-yl)-D-arabinitol (10e). Yield: 1.82 g (66%); foam; IR (KBr) ν (cm^{-1}): 3210 (OH), 1601 (C=N); ^1H NMR (D_2O): δ = (D_2O): 1.10 (d, 6H, isopropyl- CH_3), 2.19 (s, CH_3), 3.57 (dd, H-5", $J_{4',5''}$ = 6.79 Hz, $J_{5',5''}$ = 11.80

Hz), 3.70 (t, H-4', $J_{4',5'} = 2.87$ Hz), 3.78 (t, H-5'), 3.79 (dd, H-3', $J_{3',4'} = 8.20$ Hz), 4.08 (dd, H-2', $J_{2',3'} = 1.65$ Hz), 4.36 (sept, isopropyl-CH, $J = 6.6$ Hz), 4.71 (d, H-1', $J_{1',2'} = 8.19$ Hz). MS (FAB): m/z 276 [M+1]. Calcd for $C_{11}H_{21}N_3O_5$ (275.35): C, 47.99; H, 7.69; N, 15.26. Found: C, 47.84; H, 7.67; N, 15.28.

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