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The Interaction of Per-O-Acetylated Acyclic 1-(1-Butylindol-3-yl)-1-deoxy-ketoses with Silylated Uracil[†]

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ABSTRACT

The per-O-acetylated open chain derivatives of 1-(1-butylindol-3-yl)-1-deoxy-1-L-sorbose and 1-(1-butylindol-3-yl)-1-deoxy-L-tagatose, which are readily available by alkaline degradation of 1-butylascorbigen followed by acetylation, were used in a nucleoside-type synthesis. The interaction of these ketoses derivatives with bis-(trimethylsilyl)-uracil yielded in each case a mixture of (E)-2,4,5,6-tetra-O-acetyl-1-(1-butylindol-3-yl)-1,3-dideoxy-3-(uracil-1-yl)-L-xylo-hexa-1-enitol and (E)-2,4,5,6-tetra-O-acetyl-1-(1-butylindol-3-yl)-1,3-dideoxy-3-(uracil-1-yl)-L-lyxo-hexa-1-enitol, which were separated by preparative HPLC. The deacetylation of each of these compounds by MeONa in MeOH produced a mixture of 1-(1-butylindol-3-yl)-1,3-dideoxy-4-O-methyl-3-(uracil-1-yl)- α -L-sorbopyranose and 1-(1-butylindol-3-yl)-1,3-dideoxy-4-O-methyl-3-(uracil-1-yl)- β -D-fructopyranose, which were also separated by HPLC, the structures were confirmed by NMR.

Key Words: 1-Deoxy-1-(indol-3-yl)ketoses; Uracil; C-ketosides.

[†]In honor and celebration of the 70th birthday of Professor Leroy B. Townsend.

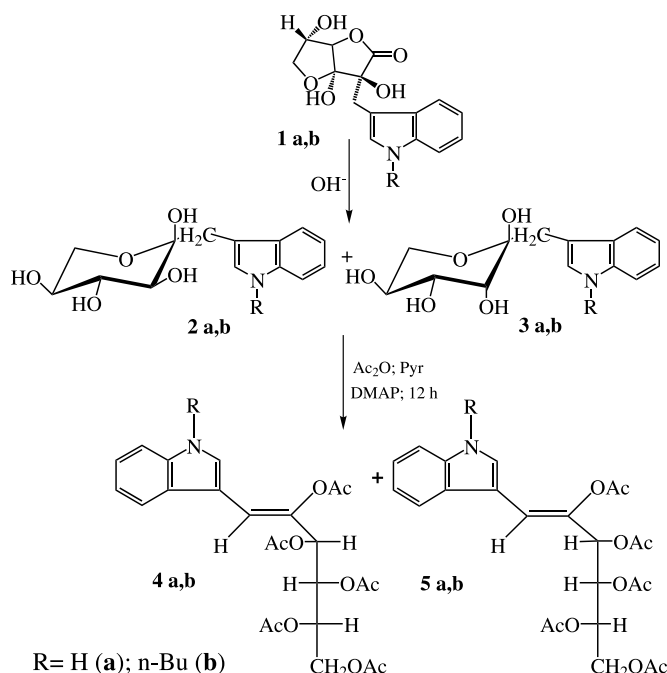
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INTRODUCTION

Whereas heteroaryl-containing aldoses and ketoses are rather rare compounds, a mixture of 1-deoxy-1-(indol-3-yl)-L-sorbose (**2a**) and 1-deoxy-1-(indol-3-yl)-L-tagatose (**3a**) can be readily obtained by the alkaline degradation of ascorbigen which is 2-C-[(indol-3-yl)methyl]- α -L-xylo-hex-3-ulofuranosono-4-lactone (**1a**). N-Substituted ascorbigens represent a good source of the corresponding N-substituted indolylketoses (Scheme 1).^[1] Recently we have shown that the mixture of diastereomeric ketoses underwent acetylation and pyranose ring opening under the action of acetic anhydride in pyridine in the presence of DMAP with the formation of a mixture of (*E*)-2,3,4,5,6-penta-O-acetyl-1-deoxy-1-(indol-3-yl)-L-xylo-hex-1-enitol (**4a**) and (*E*)-1-deoxy-1-(indol-3-yl)-L-lyxo-hex-1-enitol (**5a**), which were separated chromatographically.^[2] The goal of this work was to study a possibility of usage of these compounds in the synthesis of nucleoside analogs.

RESULTS AND DISCUSSION

The starting ketoses **2b** and **3b** were obtained by the alkaline degradation of N-butylascorbigen as previously described.^[1] The action of Ac₂O in pyridine in the presence of DMAP produced (*E*)-2,3,4,5,6-penta-O-acetyl-1-(1-butylindol-3-yl)-1-deoxy-L-xylo-hex-1-enitol (**4b**) and (*E*)-2,3,4,5,6-penta-O-acetyl-1-(1-butylindol-3-yl)-1-deoxy-L-lyxo-hex-1-enitol (**5b**) as previously described for the ketoses obtained from



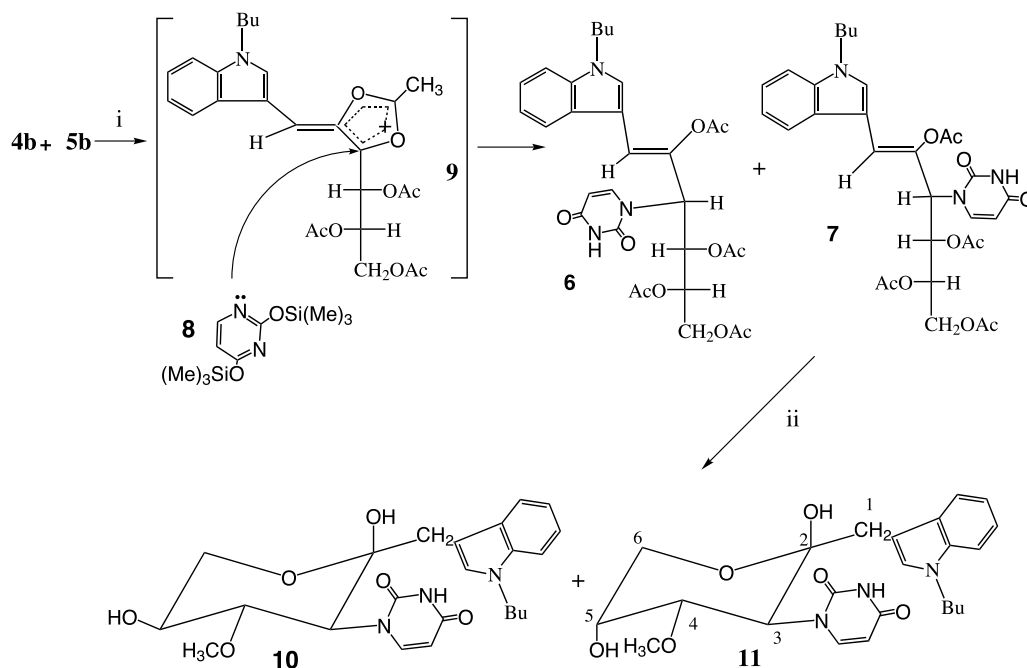
Scheme 1.



ascorbigen and 1-methylascorbigen.^[2] The interaction of the individual **4b** or **5b** with bis-(trimethylsilyl)-uracil (**8**) in the presence of SnCl_4 or $\text{Me}_3\text{SiSO}_3\text{CF}_3$ in acetonitrile gave a mixture of (*E*)-2,4,5,6-tetra-*O*-acetyl-1-(1-butylindol-3-yl)-1,3-dideoxy-3-(uracil-1-yl)-*L*-xylo-hexa-1-enitol (**6**) and (*E*)-2,4,5,6-tetra-*O*-acetyl-1-(1-butylindol-3-yl)-1,3-dideoxy-3-(uracil-1-yl)-*L*-lyxo-hexa-1-enitol (**7**) in $\sim 80\%$ yield in both cases. The mixture of the acetylated ketoses **4b** and **5b** also gave a mixture of **6** and **7**. We failed to separate **6** and **7** by TLC or column chromatography methods. NMR spectra demonstrated two sets of signals typical for the two diastereomers. The compounds were separated by preparative HPLC. Mass-spectra of both compounds were similar and had the peaks of molecular ions (m/z 597), corresponding to the molecular weight of **4b** or **5b** and also the peaks corresponding to [N-Bu-indole- CH_2] fragment, uracil fragment and to the products of the consequent splitting of acetic acid or/and acetyl groups. As each of compounds **4b** and **5b** produced one and the same mixture of the two compounds **6** and **7**, it suggests that the attack of the silylated uracil is directed to the position 3 of the ketoses chains.

The interaction of uracil with compounds **4b** or **5b** supposedly proceeds *via* intermediate acyloxonium ion **9** which is attacked by bis-(trimethylsilyl)-uracil **8** on the 3-C atom to give a mixture of diastereomers (Scheme 2).

Deacetylation of compounds **6** and **7** was performed by NaOMe in methanol. Mixtures of two compounds **10** and **11** were isolated in $\sim 35\%$ yield from the



i) SnCl_4 or $\text{Me}_3\text{SiSO}_3\text{CF}_3$; MeCN ii) NaOMe/MeOH

Scheme 2.



Compound/ Solvent	Carbohydrate moiety					Others		
	H-1	H-3 ($^3J_{3,4}$)	H-4 ($^3J_{4,3}$) [$^3J_{4,5}$]	H-5 ($^2J_{6a,6b}$) [$^3J_{6a,5}$]	H-6 _b ($^2J_{6b,6a}$) [$^3J_{6b,5}$]	Ind N-CH ₂	-OAc; OH and OCH ₃	Uracil
4b	6.60 s	5.69 d (8.2)	5.59 dd (8.2) [3.2]	5.45 m 4.31 dd (11.7) [5.3]	4.05 dd (11.7) [6.5]	4.09 t	2.26; 2.12; 2.11; 2.05; 2.04 sssss; s (OAc)	–
5b	6.68 s	5.58 s		5.52 m 4.35 dd (11.7) [4.9]	3.97 dd (11.7) [7.6]	4.08 t	2.23; 2.14; 2.06; 2.01; 2.00 sssss; s (OAc)	–
6	6.58 s	5.83 d (8.9)	5.78 dd (8.9) [2.9]	5.49 m 4.24 dd (11.6) [6.0]	4.05 m		2.15; 2.10; 2.03; 2.02 ssssis (OAc)	6-H; 5-H 7.61; 5.64 d; d; $^3J_{5,6} = 8$; NH; s, 8.40
7	6.85 s	5.58 d (8.2)	5.75 dd (8.2) [4.4]	5.35 m 4.29 dd (12.1) [4.4]	4.06 m		2.14; 2.05; 2.03; 2.02 ssssis (OAc)	6-H; 5-H 7.65; 5.68 d; d; $^3J_{5,6} = 8$ NH; s, 8.19
10	2.80; 3.11 d; d $^2J = 14.4$	4.84 dd (10.8) $^4J_{3H,2OH} = 1.8$	3.68 dd (10.8) [8.5]	3.95 m 3.72 dd (11.2) [6.0]	3.59 t $J = 11.2$	4.07 t	2-OH; 2.75, d, $^4J_{2-OH, 3H} = 1.8$; 4-OCH ₃ ; 3.45, s; 5-OH; 2.20; d, $^3J_{5-OH, 5H} = 3.9$	6-H; 5-H 7.82; 5.78 d; d; $^3J_{5,6} = 8$ NH; s, 8.40
11	2.84; 3.23 d; d $^2J = 14.6$	5.22 dd (11.2) $^4J_{3H,2OH} = 1.8$	3.75 dd (11.2) [3.4]	4.21 m 3.85 narrow m		4.07 t	2-OH; 2.71, d, $^4J_{2-OH, 3H} = 1.8$; 4-OCH ₃ ; 3.36, s; 5-OH; 2.57, bs	6-H; 5-H 7.77; 5.76 d; d; $^3J_{5,6} = 8$ NH; s, 8.18

2) Of N-butyl group (except for triplet N-CH₂ given in the table): 3 multiplets in the area ~ 0.8–1.8 ppm.

individual **6** or **7** after NaOMe/MeOH treatment and consequent column chromatography as shown by NMR and HPLC. Compounds **10** and **11** were separated by preparative HPLC. A mixture of **6** + **7** similarly gave the same mixture of compounds **10** and **11**. Unexpectedly to get totally deacetylated compounds it was necessary to use about 10 equivalents of NaOMe. The analyses of mass-spectra of these compounds revealed that each of them contains a methyl group in the structure.

^1H NMR data for the compounds obtained are presented in Table 1. NMR methods assigned these compounds as epimeric at C-5 1-(1-butylindol-3-yl)-1,3-dideoxy-4-*O*-methyl-3-(uracil-1-yl)- α -L-sorboxypyrano (**10**) and 1-(1-butylindol-3-yl)-1,3-dideoxy-4-*O*-methyl-3-(uracil-1-yl)- β -D-fructopyranose (**11**). Structural assignments of **10** and **11** was based on the analysis of two-dimensional COSY spectra, spin decoupling and on deuterium exchange experiments. Spin-spin coupling constants in ^1H -NMR spectrum of compound **10** ($^3J_{3,4} = 10.8$ Hz; $^3J_{4,5} = 8.5$ Hz) demonstrated that hydrogen atoms of carbohydrate skeleton at C-3 (d, 4.84 ppm), C-4 (q., 3.68 ppm.) and C-5 (m, 3.95 ppm) are *axial* atoms of a hexapyranose ring. A narrow doublet at 2.75 ppm in the ^1H NMR spectrum of **10**, which disappeared after the addition of two drops of CD_3OD to CDCl_3 solution as a result of deuterium exchange was identified as 2-OH proton, which participated in long range spin-spin interaction with a hydrogen atom at 4.84 ppm (q, 3- H_{ax}) (through 4 bonds in W-type disposition, $^4J_{\text{C2OH}, \text{C3H}_{\text{ax}}} = 1.8$ Hz). A broad doublet at 2.20 ppm was attributed as the 5-OH hydrogen atom, which has vicinal spin-spin interaction with 5- H_{ax} (m, 3.95 ppm, $^3J_{\text{C5OH}, \text{C5 H}_{\text{ax}}} = 3.9$ Hz). Signals in ^1H NMR spectrum of **11** were identified by similar way.

As it follows from the vicinal spin-spin constants values 3-H and 4-H in compound **11** are *axial* similarly to these atoms in compound **10**, but 5-H atom in compound **11** is *equatorial* and 5-OH atom is *axial*. It follows from the values of spin-spin coupling constants for 4-H ($^3J_{3\text{H}, 4\text{H}} = 11.2$ Hz; $^3J_{4\text{H}, 5\text{H}} = 3.4$ Hz). In this compound 2-OH hydrogen atom also interacts through four bonds with 3- H_{ax} ($^4J_{2\text{-OH}, 3\text{-H}_{\text{ax}}} = 1.8$ Hz). It assumes that 2-OH is *axial*.

NOEDIF experiments supported these structures. Figure 1 presents data of NOEDIF experiments showing the disposition of the substituents in compounds **10** and **11**. These data support 2-OH *axial* configuration in **10** and **11**, 5-H *axial* and 5-OH *equatorial* positions in **10** and 5-H *equatorial* and 5-OH *axial* positions in **11**.

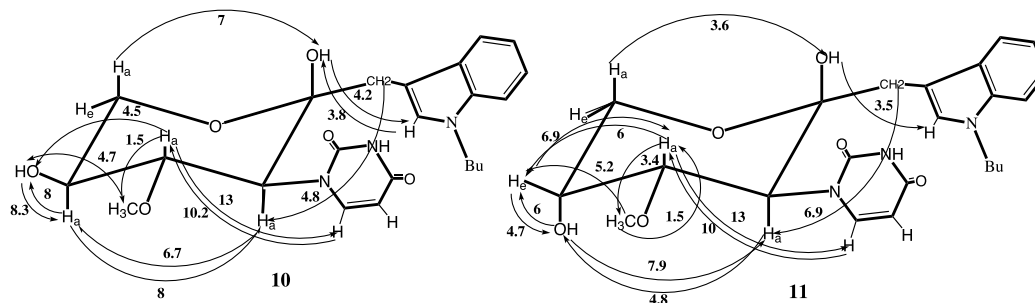
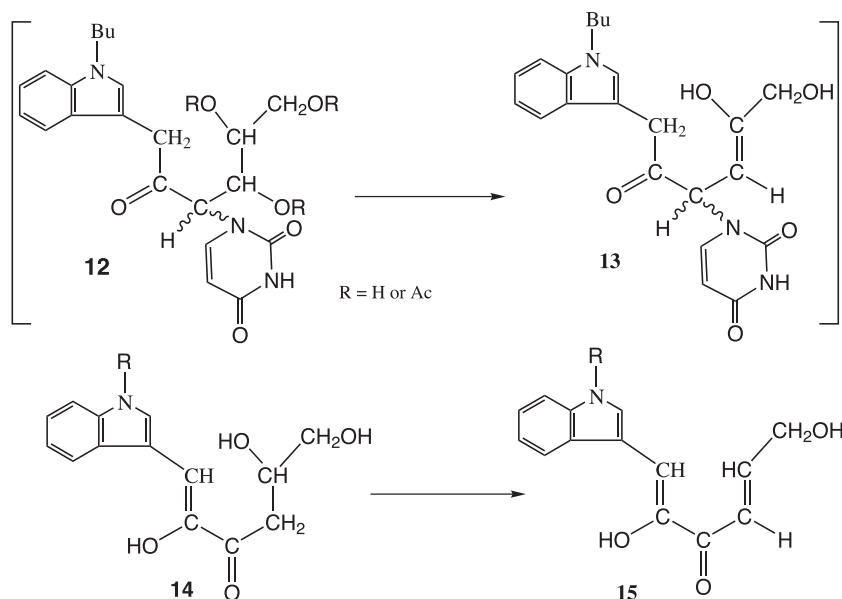


Figure 1. Data of NOEDIF experiments (in %) showing the positions of substituents in compounds **10** and **11**.





Scheme 3.

It is important to emphasize that independently on configuration at 3-C in starting compounds **6** and **7** both deacetylated compounds **10** and **11** have similar (3*S*,4*S*) configurations. Compound **10** preserved 5*S* stereochemistry (as in starting **6** or **7**), whereas compound **11** isomerized into (5*R*) derivative, i.e. turned from L into D-compound. This result supports the hypothesis that the transformation of **6** and **7** under the action of MeONa in MeOH does not proceed as a Zemplen trans-esterification reaction. In the reaction conditions compounds **6** and **7** split of acetic acid, which results in the formation of intermediates **12** and then **13** capable of epimerization at C-3. MeOH is added to the C4=C5 double bond followed by formation of a pyranose cycles with the conformationally preferable disposition of the substituents. Earlier we have demonstrated easy dehydration of compound **14** and formation of dienone **15** which underwent further transformations.^[3,4]

Compounds **10** and **11** belong to a rare class of ketosides containing a nucleoside base moiety. The usage of ascorbigen degradation products in glycosylation reactions can provide a valuable analogs of nucleosides of new types. Biological properties of the compounds of these types remain to be investigated.

EXPERIMENTAL

NMR spectra were registered on a Varian Unity Plus instrument (400 MHz). Analytical TLC was performed on Kieselgel F₂₅₄ plates (E.Merck), preparative TLC chromatography on plates (20 × 20 cm, 0.5 mm) with Kieselgel 60 F₂₅₄ (E.Merck), and column chromatography on Kieselgel 60 (E.Merck), using the following systems of



solvents: **A** (petroleum ether–EtOAc, 3:2), **B** (petroleum ether–EtOAc, 2:1), **C** (CHCl₃–CH₃OH, 5:1). All solutions were dried over sodium sulfate and evaporated at reduced pressure on a Buchi rotary evaporator. Optical rotations were measured on a Perkin-Elmer 241 polarimeter.

High resolution mass spectra were registered on a MAT 8430 Finnigan instrument (USA) with data operating system SS-300 (EI, 70 eV, direct introduction, temperature of ion source 250°C). Electron impact (EI) mass-spectra were registered on a SSQ 710 Finnigan MAT instrument (USA), (EI: 70 eV, direct introduction). Analytical HPLC analyses were performed on a Millichrom 5 instrument (Russia), using a Diasorb C 16 column (2 × 120 mm and particle size 7 μm), injection volume 5 μl, detection at 280 nm, by isocratic elution, using systems of solvents: #1 (H₂O–CH₃CN, 45:55) and #2 (H₂O–CH₃CN, 60:40). Preparative HPLC were performed on a Shimadzu HPLC series LC 10 using a Diasorb C 16 column (15 × 250 mm and particle size 7 μm), with injection volume 50 μl, detection at 280 nm, using the same systems of solvents. Melting points were determined on a Buchi SMP-20 apparatus and are uncorrected.

(E)-1-Deoxy-1-(1-butylindol-3-yl)-2,3,4,5,6-penta-O-acetyl-L-xylo-hex-1-enitol (4b) and (E)-1-deoxy-1-(1-butylindol-3-yl)-2,3,4,5,6-penta-O-acetyl-L-lyxo-hex-1-enitol (5b). A solution of **2b** and **3b** mixture^[1] (1.3 g, 3.7 mmol) and DMAP (60 mg) in dry pyridine (20 mL) was cooled to –10°C, then Ac₂O (2.52 mL, 26.64 mmol) was added, and the mixture was left at room temperature for 12 h. It was then dissolved in 1N HCl (300 mL), and extracted with ether (3 × 50 mL). The extract was washed by brine, dried over Na₂SO₄ and evaporated *in vacuo* to give a mixture (1.8 g, 81%) of **4b** and **5b** as brown crystals. It was chromatographed on column with silica gel (system A), the fractions were evaporated, and after crystallizing from ether gave individual **4b** (1 g, 40 %) and **5b** (0.4 g, 20%).

4b: White crystals, mp 88–90°C (CHCl₃); [α]_D²⁰ – 8.5° (C 2.5, CHCl₃); R_f 0.46 (B); Anal. Calcd for C₂₈H₃₅NO₁₀: C, 61.63; H, 6.47; N, 2.57. Found: C, 61.56; H, 6.58; N, 2.44.

5b: White crystals, mp 104–106°C (CHCl₃); [α]_D²⁰ + 3° (C 2.5, CHCl₃); R_f 0.54 (B); Anal. Calcd for C₂₈H₃₅NO₁₀: C, 61.63; H, 6.47; N, 2.57. Found: C, 61.79; H, 6.38; N, 2.40.

(E)-1,3-Dideoxy-1-(1-butylindol-3-yl)-3-(uracil-1-yl)-2,4,5,6-tetra-O-acetyl-L-xylo-hex-1-enitol (6) and (E)-1-dideoxy-1-(1-butylindol-3-yl)-3-(uracil-1-yl)-2,4,5,6-tetra-O-acetyl-L-lyxo-hex-1-enitol (7). To a solution of **4b** (or **5b** or a mixture **4b** + **5b**) in dry acetonitrile (10 mL) was added bis-(trimethylsilyl)-uracil **8** (775 mg, 3 mmol) and then SnCl₄ (0.24 mL, 2 mmol); after 30 min the reaction mixture was diluted with water (100 mL) and the products were extracted with ether (3 × 10 mL). The extract was dried with Na₂SO₄ and evaporated to give a mixture of **6** and **7** (510 mg, 85 %) with R_f 0.65 (C) as yellow oil.

The individual **6** and **7** were obtained with the use of preparative HPLC using 20 mg/mL solution in CH₃CN in system # 1, flow rate 3.0 mL/min, R_t 35.4 and 40.1 min respectively.

6: Yellow amorphous powder. Analytical HPLC: R_t 11.5 min (system # 1). EI-MS, *m/z* (%): 597 (M⁺, 30%); 555 (M–Ac, 100%); 495 (M–Ac–AcOH, 25%); 435 (M–Ac–2AcOH, 15%); 375 (M–Ac–3AcOH, 55%); 186 (N–BuInd–CH₂, 70%); 112 (uracil, 10%).

HR-MS calc. for C₃₀H₃₅N₃O₁₀ 597.2323 found 597.2312.



7: Yellow amorphous powder. Analytical HPLC: R_t 13.0 min (system #1). EI-MS, m/z (%): 597 (M^+ , 20%); 555 (M-Ac, 100%); 495 (M-Ac-AcOH, 15%); 435 (M-Ac-2AcOH, 15%); 375 (M-Ac-3AcOH, 50%); 186 (N-BuInd-CH₂, 65%); 112 (uracil 15%). HR-MS calc. for C₃₀H₃₅N₃O₁₀ 597.2323 found 597.2310.

1,3-Dideoxy-1-(1-butylindol-3-yl)-3-(uracil-1-yl)-4-O-methyl- α -L-sorbopyranose (10) and 1,3-dideoxy-1-(1-butylindol-3-yl)-3-(uracil-1-yl)-4-O-methyl- β -D-fructopyranose (11). To a solution of a mixture of **6** and **7** (500 mg, 0.84 mmol) in methanol (10 mL) was added 2N solution of MeONa (~10 mmol) under argon. Then the reaction mixture was diluted with 5% NaHSO₄ solution and the products were extracted with ethyl acetate. The extract was dried over Na₂SO₄, evaporated and the products were purified by column chromatography (a system) to produce a mixture of **10** and **11** (130 mg, 35%) as amorphous yellowish powder, R_f 0.28 (C). The individual **10** and **11** were obtained by HPLC (sample concentration 15 mg/mL in CH₃CN, system #2, flow rate 3.0 mL/min, R_t 34.4 and 36.5 min respectively).

10: Colorless amorphous powder; $[\alpha]_D^{20} + 7.5^\circ$ (C 1.5, MeOH); analytical HPLC R_t 8.4 min (system # 2); EI-MS, m/z (%): 443 (M^+ , 50%); 425 (M-H₂O, 40%); 407 (M-2H₂O, 40%); 393 (M-H₂O-MeO, 100%); 295 (M-2H₂O-uracil, 65%); 186 (N-BuInd-CH₂, 80%).

HR-MS calc. for C₂₃H₂₉N₃O₆ 443.2056 found 443.2036.

11: Colorless amorphous powder; $[\alpha]_D^{20} - 4^\circ$ (C 2.2, MeOH); analytical. HPLC R_t 8.9 min (system # 2); EI-MS, m/z (%): 443 (M^+ , 40%); 425 (M-H₂O, 40%); 407 (M-2H₂O, 20%); 393 (M-H₂O-MeO, 100%); 295 (M-2H₂O-uracil, 60%); 186 (N-BuInd-CH₂, 90%).

HR-MS calc. for C₂₃H₂₉N₃O₆ 443.2056 found 443.2038.

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