MITSUNOBU TRANSFORMATIONS OF 1,2-*O*-ISOPROPYLIDENE-α-D-PENTOFURANOSES MEDIATED BY ZINC SALTS

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A one-pot regioselective heterofunctionalization of 1,2-*O*-isopropylidene-α-D-xylofuranose (**1**) and 1,2-*O*-isopropylidene-α-D-ribofuranose (**2**) with zinc azide, zinc thiocyanate or zinc *N*,*N*-dimethyldithiocarbamate *via* the Mitsunobu reaction has been performed. With **2**, the reaction gave selectively the desired products substituted at C-5 in good isolated yields (60–65%). However, application of the same reaction conditions to **1** led to the predominant formation of a cyclic 3,5-anhydro derivative. In contrast, the reaction of hydrazoic acid with **1** afforded 5-azido-5-deoxy-1,2-*O*-isopropylidene-α-D-xylofuranose besides formerly unknown 5-azido-3,5-dideoxy-1,2-*O*-isopropylidene-α-D-*glycero*-pent-3-enofuranose and 3,5-diazido-3,5-dideoxy-1,2-*O*-isopropylidene-α-D-ribofuranose; the yields depended on the reaction time and the molar ratio of reagents.

Key words: Carbohydrates; Pentofuranoses; Mitsunobu reaction; Nucleophilic displacement; Zinc salts; Azidosugars; Anhydrosugars; Azides; Thiocyanates; Dithiocarbamates.

The Mitsunobu reaction, originally employing triphenylphosphine (TPP), diethyl azodicarboxylate (DEAD) and an acid-type reagent[1], has become a powerful tool for the selective transformation of polyhydroxy compounds. Exceptionally mild reaction conditions, as well as the stereoselectivity and regioselectivity associated with reasonably high yields are the main advantages of the reaction. However, the method has some limitations as well. Due to the complexity of intermediates involved, undesired products can be formed and the outcome of the Mitsunobu reaction can also be affected in some cases by the order of addition of the reagents involved. The tedious removal of side-products – in particular triphenylphosphine oxide and dialkyl hydrazine-1,2-dicarboxylate – is a serious drawback commonly associated with the method.

In carbohydrate or nucleoside chemistry, the title reaction is an attractive one-step procedure as compared with a classic route requiring the preliminary activation of a hydroxy function in the form of a good leaving group. In such a way, 5′-(alkylsulfanyl)nucleosides[2] or 5′-(acetylsulfanyl)adenosine[3] were prepared directly in high yields. The Mitsunobu conditions were successfully applied to the synthesis of 5′-deoxy-5′-(hydroxyamino)nucleosides[4], reversed azole nucleosides[5] or dideoxyepimino derivatives of 1,6-anhydrohexopyranoses[6]. Miscellaneous nucleophiles – including metallic salts – have been introduced in the Mitsunobu methodology: zinc tosylate[7], zinc halides[8] or zinc *N*,*N*-dimethyldithiocarbamate[9] are well-known examples. Moreover, zinc azide[10] has become the reagent of choice for azidation, competing favourably with inconvenient hydrazoic acid[11,12] or azidotrimethylsilane[13,14].

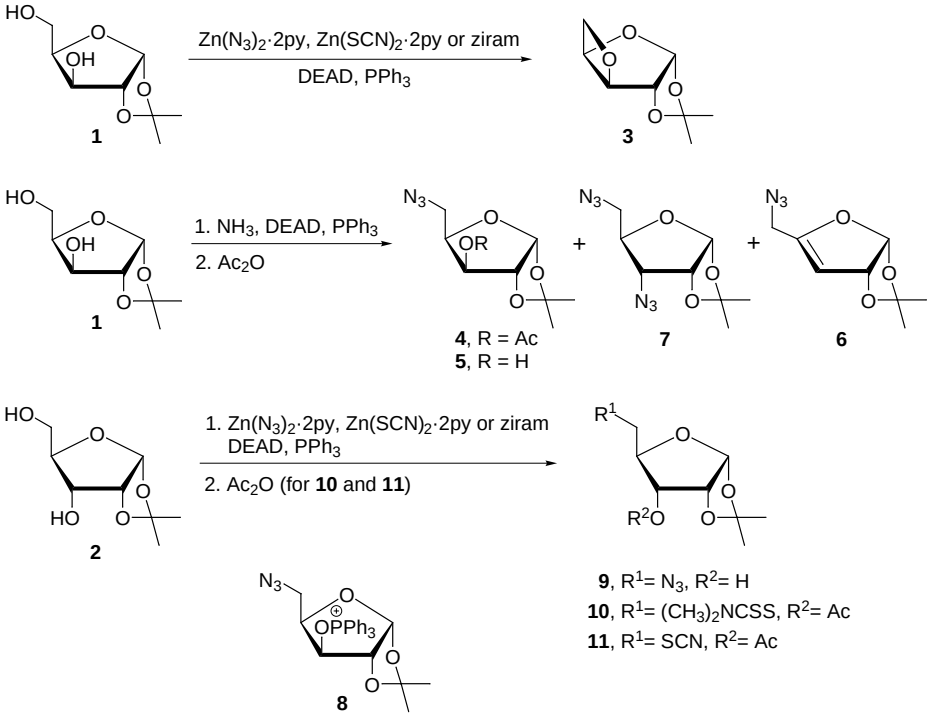
In previous studies[15,16], we have described regioselective thio functionalizations of partially protected pentofuranoses. We now extend the use of zinc salts as nucleophiles in the Mitsunobu substitution of 1,2-*O*-isopropylidene-α-D-pentofuranoses.

RESULTS AND DISCUSSION

Zinc *N*,*N*-dimethyldithiocarbamate (ziram), zinc azide and zinc thiocyanate were chosen as the nucleophilic partners for Mitsunobu transformations of 1,2-*O*-isopropylidene-α-D-xylofuranose (**1**) and 1,2-*O*-isopropylidene-α-D-ribofuranose (**2**) (Scheme 1). Zinc azide was used in the form of its more stable bis-pyridine complex prepared according to Agrell[17]. Analogously, the bis-pyridine complex of zinc thiocyanate was synthesized for the first time and characterized by microanalysis. Throughout the study, the same experimental protocol for the Mitsunobu reaction was applied: diethyl azodicarboxylate (DEAD) was added to a solution of **1** or **2** containing triphenylphosphine (TPP), the mixture was left standing for 5 min and then the nucleophilic species was introduced in one portion. The reaction was performed in pyridine, due to the enhanced solubility of zinc salts.

The reaction of **1** with either ziram or zinc azide or zinc thiocyanate was complete within 30 min giving 3,5-anhydro-1,2-*O*-isopropylidene-α-D-xylofuranose (**3**) as the major product. The oxetane **3** was identified by comparison of its ^{1}H NMR data and optical rotation with those reported previously[18]. Compound **1** has been known[15] to produce oxetane **3** even when treated only with TPP and DEAD. Evidently, the suitably oriented 3-OH group in **1** competes with an external nucleophile when their nucleophilicities are comparable. In order to test this hypothesis, com-

pound **1** was subjected to the attack of hydrazoic acid, which is a stronger nucleophile. After 15 min, **1** was consumed and after acetylation, the desired 3-*O*-acetyl-5-azido-5-deoxy-1,2-*O*-isopropylidene-α-D-xylofuranose (**4**) was isolated in 60% yield. Zemplen's deprotection of **4** afforded 5-azido-5-deoxy-1,2-*O*-isopropylidene-α-D-xylofuranose (**5**) in 55% overall yield based on **1**. Thus the yield of the one-pot Mitsunobu azidation of the



SCHEME 1

xylofurano derivative **1** compares favourably with the results reported[19–21] for the classic displacement of the 5-*O*-tosylate of **1** or the ring-opening[18] of oxetane **3**. At the final stage of the reaction, formation of two minor less polar side products was observed. An experiment in which compound **1** was treated with an excess of hydrazoic acid for 90 min gave only 27% of azide **5** besides two side products, **6** and **7**. The syrupy compound **6** with the highest R_F exhibited a strong IR band at 2 101 cm^{-1} and [α] –11, and its structure was established from ^1H and ^{13}C NMR spectra (Table I) as 5-azido-3,5-dideoxy-1,2-*O*-isopropylidene-α-D-*glycero*-pent-3-enofuranose. In the ^1H NMR spectrum, the singlet at δ 3.84 ppm, due to the equivalent

TABLE I
NMR data for compounds **4**, **6**, **7**, **10** and **11**

Parameter	**4**	**6**	**7**	**10**[a]	**11**[b]
			Chemical shifts (δ, ppm)		
H-1	5.94	6.11	5.84	5.80	5.85
H-2	4.55	5.31[c]	4.78	4.80	4.86
H-3	5.21	5.25[c]	3.52	4.61	4.74
H-4	4.41	–	4.21	4.43	4.42
H-5	3.52	3.82	3.76	3.83	3.44
H-5′	3.45	3.82	3.45	3.68	3.12
CH_3	1.32; 1.50		1.29; 1.48	1.32; 1.54	1.35; 1.55
CH_3CO	2.11	–	–	2.12	2.15
$(CH_3)_2)C$	112.27	112.50	113.5	113.02	113.56
C-1	101.70	101.0	104.2	104.0	103.95
C-2	83.35	83.2	80.0	77.32	77.46
C-3	76.23	106.8	60.5	74.38	74.21
C-4	77.52	156.3	76.6	76.03	75.38
C-5	49.13	47.5	50.3	39.15	35.48
CH_3	26.13; 26.61	27.8; 28.0	26.4	26.52	26.41; 26.51
CH_3CO	20.62	–	–	20.69	20.50
CH_3CO	169.58	–	–	170.28	170.14
			Vicinal and geminal coupling constants (*J*(H,H), Hz)		
J(1,2)	3.7	5.3	3.6	3.7	3.7
J(2,3)	0	0	4.6	4.8	4.7
J(3,4)	3.0	2.1	9.6	9.1	9.0
J(4,5)	6.9	0	2.8	3.1	3.6
J(4,5′)	5.7	0	3.8	6.2	5.8
J(5,5′)	12.8	0	13.7	14.3	13.9

[a] Additional signals: 3.40 and 3.55 (2 × s, each 3 H, N-CH_3); 41.33 and 45.67 (N-CH_3); 196.20 (C=S). [b] Additional signals: 111.80 (CN). [c] Long-range coupling.

two C-5 protons, and multiplet at δ 5.25 ppm of H-3 olefinic proton, with coupling constant $J(2,3)$ = 2.1 Hz and two long-range allylic coupling constants $^4J(3,5)$ = 1 Hz, were observed. The ^{13}C NMR signals at δ 156.3 and 106.8 ppm could be assigned to C-4 and C-3 atoms, respectively. Finally, the elemental analysis for **6** was in accord with the proposed structure. Compound **7** was isolated also as a syrup, [α] +192, with an IR spectrum showing a strong band at 2 107 cm^{-1}. Elemental analysis and examination of ^{1}H and ^{13}C NMR spectra (Table I) suggested the structure of 3,5-diazido-3,5-dideoxy-1,2-*O*-isopropylidene-α-D-ribofuranose for **7**. The yields of compounds **6** and **7** were 11 and 29%, respectively. The formation of related compounds has been previously observed in the reaction of **1** with 2-sulfanylbenzothiazole[15]. It can be hypothesized that the alkoxyphosphonium species **8** takes part in the Mitsunobu azidation under the conditions used and that products **6** and **7** arise by its subsequent E2 and SN2 reactions.

1,2-*O*-Isopropylidene-α-D-ribofuranose (**2**) reacted smoothly with all three zinc salts producing the desired products. Thus, 5-azido-5-deoxy-1,2-*O*-isopropylidene-α-D-ribofuranose (**9**) was obtained in 65% yield and identified by comparison of both ^{1}H NMR data and optical rotation with those reported[21]. 5-Deoxy-5-(*N*,*N*-dimethyldithiocarbamoyl)-1,2-*O*-isopropylidene-α-D-ribofuranose was isolated as its 3-*O*-acetyl derivative **10** in 65% yield and the structure was ascertained from ^{1}H and ^{13}C NMR data (Table I). In the ^{13}C NMR spectrum, the signals for C-5 (δ 39.15 ppm) and the thiocarbonyl moiety (δ 196.2 ppm) were the most significant. Additional support also came from the signals of the dimethylamino group exhibiting two resolved singlets at 3.40 and 3.55 ppm in the ^{1}H NMR spectrum. The reaction of ribofuranose **2** with zinc thiocyanate followed by acetylation afforded 3-*O*-acetyl-5-deoxy-1,2-*O*-isopropylidene-5-thiocyanato-α-D-ribofuranose (**11**) in 60% yield. Its identification was unambiguously confirmed by both IR and ^{13}C NMR data. Compound **11** gave medium-strong IR band at 2 160 cm^{-1}, falling into the range of 2 175–2 100 cm^{-1} reported for thiocyanates[22,23]: typically, 2 140 cm^{-1} was described for 5-deoxy-1,2-*O*-isopropylidene-5-thiocyanato-α-D-xylofuranose[24]. In contrast, isothiocyanates are distinguishable[22] by a broad double band ranging from 2 020 to 1 990 cm^{-1}. In the ^{13}C NMR spectrum of **11** (Table I), the resonance at δ 111.8 ppm can be assigned to the thiocyanato carbon[23] whilst the signal of the corresponding isothiocyanato carbon[22] at about 142–144 ppm was absent. In addition, the resonance of C-5 at δ 35.48 ppm fits well with the proposed structure.

To our knowledge, this is the first example of the utilization of zinc thiocyanate as a nucleophile in the Mitsunobu reaction. The yield of **11** can

compete with that observed in the conversion of silyl ethers into the corresponding thiocyanates by *in situ* generation of (dithiocyanato)triphenyl phosphoranes[23].

In conclusion, this paper once again demonstrates the ability of zinc salts to act as nucleophiles in the regioselective Mitsunobu transformation of 1,2-*O*-isopropylidene-α-D-ribofuranose (**2**). In particular, zinc thiocyanate is introduced as a new and efficient agent for the preparation of thiocyanato derivatives. In the case of 1,2-*O*-isopropylidene-α-D-xylofuranose (**1**), the competition between external and internal nucleophiles occurred under the conditions used.

EXPERIMENTAL

Optical rotations were measured on a JASCO Model DIP-370 polarimeter and are given in 10^{-1} deg cm^2 g^{-1}. Melting points were determined with a Kofler hot block and are uncorrected. NMR data were extracted from spectra measured in $CDCl_3$ solutions (tetramethylsilane as an internal standard) at 25 °C with a Bruker AM 400 spectrometer (^{1}H, 400 MHz; ^{13}C, 100.62 MHz). Chemical shifts are given in ppm (δ-scale) and coupling constants (*J*) in Hz. Assignment of ^{13}C and ^{1}H signals are based on APT, HETCOR and COSY experiments. IR spectra (wavenumbers in cm^{-1}) were measured on a FT IR Nicolet 740 spectrometer in CCl_4 or $CHCl_3$ (compounds **10** and **11**) solutions. Column chromatography was performed on silica gel 100–160 μm (Lachema, Czech Republic), and TLC on silica gel according to Stahl (10–40 μm, Merck, Germany) with detection by spraying with 1% $Ce(SO_4)_2$ in 10% H_2SO_4 and subsequent mineralization. Solutions were concentrated under reduced pressure with a bath temperature below 40 °C.

DEAD, TPP and ziram were purchased from Sigma–Aldrich. 1,2-*O*-Isopropylidene-α-D-xylofuranose (**1**) was prepared by the known procedure[25]. 1,2-*O*-Isopropylidene-α-D-ribofuranose (**2**) was obtained from **1** by benzoylation, oxidation and reduction[26]. Zinc azide bis-pyridine complex was prepared from zinc nitrate and sodium azide in pyridine by the described procedure[17] and crystallized from ethanol. For $C_{10}H_{10}N_8Zn$ (307.6) calculated: 39.04% C, 3.28% H, 36.43% N; found: 38.85% C, 3.14% H, 36.62% N. Zinc thiocyanate bis-pyridine complex was synthesized analogously from zinc nitrate and sodium thiocyanate. For $C_{12}H_{10}N_4S_2Zn$ (339.7) calculated: 42.42% C, 2.97% H, 16.49% N, 18.87% S; found: 42.44% C, 3.01% H, 16.54% N, 18.35% S. Hydrazoic acid[27] was used in benzene solution and its concentration was determined by NaOH titration of water–benzene emulsion.

Zinc Salt-Mediated Mitsunobu Transformation. General Procedure

DEAD (2 equivalents) was introduced dropwise at room temperature to a stirred dry pyridine solution containing TPP (2 equivalents) and furanose **1** or **2** (1 equivalent) under nitrogen. After 5 min, the zinc salt (1.1 equivalent) was added in one portion and the reaction mixture was maintained at 80 °C until the furanose was consumed (TLC). In procedure A, the solvent was evaporated and the residue was separated by silica gel chromatography using petroleum ether–ethyl acetate (10 : 1) with the gradually increasing ethyl acetate content. In procedure B, the product was directly acetylated with 10 molar excess of acetic anhydride

overnight; then the mixture was decomposed with water, concentrated to dryness and separated by flash chromatography on silica gel with toluene–ethanol (9 : 1) or petroleum ether–ethyl acetate (10 : 1) solvent systems.

*Reaction of **1** with $Zn(N_3)_2 \cdot 2py$*. The reaction of **1** (510 mg, 2.7 mmol) with TPP (1.44 g, 5.4 mmol), DEAD (0.83 ml, 5.4 mmol) and $Zn(N_3)_2 \cdot 2py$ (920 mg, 3.0 mmol) in pyridine (15 ml) was complete after 30 min. The major product (420 mg, R_F 0.77, benzene–acetone, 4 : 1) was identified by ^{1}H NMR (ref.[18]) as 3,5-anhydro-1,2-*O*-isopropylidene-α-D-xylofuranose (**3**), yield 90%, $[\alpha]_D^{20}$ +12 (*c* 1.0, $CHCl_3$), ref.[18] $[\alpha]_D^{20}$ 11.6 (*c* 0.75, $CHCl_3$). A minor product (R_F 0.60, benzene–acetone, 4 : 1) could not be isolated in pure form.

*Reaction of **1** with ziram*. The reaction of **1** (510 mg, 2.7 mmol) with TPP (1.44 g, 5.4 mmol), DEAD (0.83 ml, 5.4 mmol) and ziram (920 mg, 3.0 mmol) in pyridine (15 ml) was complete after 30 min. TLC analysis revealed the presence of predominant oxetane **3** (R_F 0.77, benzene–acetone, 4 : 1) besides two minor compounds with R_F 0.81 and 0.56 (benzene–acetone, 4 : 1), respectively. Repeated chromatography gave oxetane **3** (310 mg, 66%) but minor compounds could not be isolated in pure form.

*Reaction of **1** with $Zn(SCN)_2 \cdot 2py$*. The reaction of **1** (50 mg, 0.26 mmol) with TPP (140 mg, 0.52 mmol), DEAD (0.083 ml, 0.54 mmol) and $Zn(SCN)_2 \cdot 2py$ (98 mg, 0.29 mmol) in pyridine (1.5 ml) was completed after 30 min. The major product showed the same R_F values in three solvent systems (benzene–acetone, 4 : 1; petroleum ether–ethyl acetate, 8 : 3; toluene–ethanol, 10 : 1) as oxetane **3**. The formation of a minor product (R_F 0.62, benzene–acetone, 4 : 1) was also observed.

*Reaction of **2** with $Zn(N_3)_2 \cdot 2py$*. The reaction of **2** (500 mg, 2.6 mmol) with TPP (1.38 g, 5.2 mmol), DEAD (0.80 ml, 5.2 mmol) and $Zn(N_3)_2 \cdot 2py$ (880 mg, 2.7 mmol) in pyridine (15 ml) was complete after 15 min. 5-Azido-5-deoxy-1,2-*O*-isopropylidene-α-D-ribofuranose (**9**; 370 mg, 65%) was isolated by procedure A, m.p. 50–51°C, ref.[21] m.p. 50.5–51.5 °C, $[\alpha]_D^{20}$ +61 (*c* 1.0, $CHCl_3$), ref.[21] $[\alpha]_D^{20}$ +65.5 (*c* 0.5, $CHCl_3$). ^{1}H NMR spectrum corresponded with this already described[21].

*Reaction of **2** with ziram*. The reaction of **2** (500 mg, 2.6 mmol) with TPP (1.40 g, 5.3 mmol), DEAD (0.80 ml, 5.3 mmol) and ziram (885 mg, 2.9 mmol) in pyridine (15 ml) was complete after 10 min. TLC analysis revealed the presence of a major product (R_F 0.64, benzene–acetone, 4 : 1) which was isolated after acetylation (procedure B). Repeated chromatography gave 3-*O*-acetyl-5-deoxy-5-(*N*,*N*-dimethyldithiocarbamoyl)-1,2-*O*-isopropylidene-α-D-ribofuranose (**10**) (545 mg, 65%), $[\alpha]_D^{21}$ +54.5 (*c* 1.2, $CHCl_3$). IR: 1 240, 1 376, 1 711, 1 742, 2 937–3 022. ^{1}H and ^{13}C NMR spectra are summarized in Table I. For $C_{13}H_{21}NO_5S_2$ (335.4) calculated: 46.55% C, 6.31% H, 4.18% N, 19.12% S; found: 47.01% C, 6.62% H, 4.23% N, 20.15% S.

*Reaction of **2** with $Zn(SCN)_2 \cdot 2py$*. The reaction of **2** (480 mg, 2.52 mmol) with TPP (1.32 mg, 5.0 mmol), DEAD (0.82 ml, 5.0 mmol) and $Zn(SCN)_2 \cdot 2py$ (941 mg, 2.8 mmol) in pyridine (15 ml) was completed after 15 min. TLC analysis revealed the presence of a major product (R_F 0.57, benzene–acetone, 4 : 1) which was isolated after acetylation (procedure B). Chromatography afforded a mixture of at least three minor compounds (145 mg) with $R_F > 0.8$ (petroleum ether–ethyl acetate, 8 : 3). Further elution gave 3-*O*-acetyl-5-deoxy-1,2-*O*-isopropylidene-5-thiocyanato-α-D-ribofuranose (**11**) (415 mg, 60%), $[\alpha]_D^{21}$ +76 (*c* 1.3, $CHCl_3$). IR: 1 240, 1 376, 1 711, 1 745, 2 160, 3 160. ^{1}H and ^{13}C NMR spectra are summarized in Table I. For $C_{11}H_{15}NO_5S$ (273.3) calculated: 48.34% C, 5.53% H, 5.12% N, 11.73% S; found: 48.39% C, 5.48% H, 5.21% N, 11.17% S.

3-*O*-Acetyl-5-azido-5-deoxy-1,2-*O*-isopropylidene-α-D-xylofuranose (**4**)

To a solution of xylofuranose **1** (210 mg, 1.1 mmol) and TPP (590 mg, 2.2 mmol) in pyridine (4 ml), DEAD (0.36 ml, 2.2 mmol) was introduced dropwise. After 5 min, 0.47 mM benzene solution of hydrazoic acid (2.6 ml) was added under stirring. The mixture was heated under reflux for 15 min. TLC indicated the presence of a major product (R_F 0.60, benzene–acetone, 4 : 1) and two minor products with R_F 0.84 and 0.88 (benzene–acetone, 4 : 1) were also detected. Procedure B afforded 3-*O*-acetyl-5-azido-5-deoxy-1,2-*O*-isopropylidene-α-D-xylofuranose (**4**; 140 mg, 59%), $[\alpha]_D^{24}$ –20 (*c* 1.0, $CHCl_3$). IR: 1 378, 1 384, 1 756, 2 103. ^1H and ^{13}C NMR spectra are summarized in Table I. For $C_{10}H_{15}N_3O_5$ (257.2) calculated: 46.69% C, 5.88% H, 16.33% N; found: 46.51% C, 5.75% H, 16.35% N.

5-Azido-3,5-dideoxy-1,2-*O*-isopropylidene-α-D-*glycero*-pent-3-enofuranose (**6**) and 3,5-Diazido-3,5-dideoxy-1,2-*O*-isopropylidene-α-D-ribofuranose (**7**)

DEAD (1.7 ml, 10.3 mmol) was introduced dropwise into a solution of xylofuranose **1** (1.0 g, 5.3 mmol) and TPP (2.8 g, 10.3 mmol) in pyridine (20 ml). After 5 min, 1.15 mM benzene solution of hydrazoic acid (10 ml) was added under stirring. The mixture was heated under reflux for 30 min. Another portion of 1.15 mM hydrazoic acid (5 ml) was then added and stirring was continued for 60 min. Procedure A gave a mixture of **6** and **7** (485 mg) followed by 5-azido-5-deoxy-1,2-*O*-isopropylidene-α-D-xylofuranose (350 mg, 27%), which was acetylated (procedure B) to give **4**. Rechromatography of the **6** and **7** mixture (petroleum ether–ethyl acetate, 10 : 1) afforded **6** (115 mg, 11%), $[\alpha]_D^{24}$ –11 (*c* 1.0, $CHCl_3$). IR: 1 050, 1 248, 1 664, 2 101. ^1H and ^{13}C NMR spectra are summarized in Table I. For $C_8H_{11}N_3O_3$ (197.2) calculated: 48.72% C, 5.62% H, 21.31% N; found: 48.20% C, 5.41% H, 21.03% N. Further elution gave **7** (360 mg, 29%), $[\alpha]_D^{23}$ +192 (*c* 1.2, $CHCl_3$). IR: 2 106. ^1H and ^{13}C NMR spectra are summarized in Table I. For $C_8H_{12}N_6O_3$ (240.2) calculated: 40.00% C, 5.04% H, 34.99% N; found: 40.20% C, 5.20% H, 34.25% N.

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